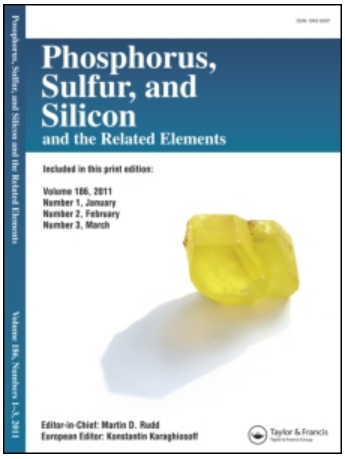

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An Efficient Synthesis of New 3,4-Dihydropyrimidin-2(1H)-ones Incorporating a Phenyl Moiety at C-5 and C-6 Catalyzed by TMSCl and $Co(OAc)_2.4H_2O$

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New 3,4-dihydropyrimidin-2-ones having a phenyl moiety at C-5 and C-6 have been prepared by a microwave-assisted Biginelli–like reaction by a three-component, one-pot condensation of an aromatic aldehyde, deoxybenzoin and urea, or thiourea using TMSCl and $Co(OAc)_2.4H_2O$ as an efficient Lewis acid catalyst.

Keywords Biginelli–like reaction; $Co(OAc)_2.4H_2O$; 3,4-dihydropyrimidin-2-one; microwave-assisted; TMSCl

INTRODUCTION

Many aryl-substituted 3,4-dihydropyrimidin-2-ones (DHPMs) and their derivatives are an important class of compounds in the field of drugs and pharmaceuticals.[1] They are found to exhibit a wide range of biological activities[2] such as antibacterial, antiviral, antitumor, and anti-inflammatory properties. Most of the DHPMs and their derivatives are medicinally important as calcium channel blockers, antihypertensive agents, X_{1a}-antagonists, and HIV agents.[3] The biological activities of some marine alkaloids isolated recently have been attributed to the presence of dihydropyrimidinone moiety.[4] The first reported synthesis of 3,4-dihydropyrimidin-2(1H)-ones using a multicomponent reaction milieu was described by Biginelli in 1893.[5] The Biginelli reaction consists of a three-component, one-pot condensation of β-ketoesters, with aldehydes and urea under strongly acidic conditions to afford 3,4-dihydropyrimidinones having an esters moiety at C-5 and an alkyl substituent at C-6. However, the yields of the products were very low, just 20%–50%. From then on, many new techniques, such as microwave-assisted synthesis techniques,[6] ionic liquids,[7] ultrasound irradiation,[8] and solvent free techniques,[9] and many new catalysts such as

polyphosphonate ester (PPE),[10] montmorillonite,[11] $InCl_3$,[12] lanthanide triflate,[13] cericammonium nitrate,[14] $BiCl_3$,[15] $LiClO_4$,[16] TMSCl,[17–18] and Co(II),[19] etc., were used to improve this transformation.

The first Biginelli–like reaction, reported by Wang et al.,[20] was conducted in CH_3CN by using aldehydes, ketones, and urea, which remarkably broadened the Biginelli reaction. In recent years, several synthetic procedures for preparing DHPMs via a Biginelli–like reaction have been reported. In spite of their potential utility, some of these methods involve long reaction times (12 h), stoichiometric amounts of catalysts, and unsatisfactory yields. Furthermore, the scope of substrates was limited to aromatic aldehydes, acetophenone, and urea.[21–22]

Following our previous work about the Biginelli reaction,[23,24] we would like to report, for the first time, a simple approach to the Biginelli–like reaction via a three-component cyclocondensation of an aromatic aldehyde, ketone or β-ketoester, urea or thiourea as substrates, and $Co(OAc)_2.4H_2O$ and chlorotrimethylsilane (TMSCl) as catalysts under microwave irradiation, which is an efficient preparation of new 3,4-dihydropyrimidin-2(1H)-ones having phenyl moiety at C-5 and C-6.

RESULTS AND DISCUSSION

We first began to study the reaction involving 3-methoxy benzaldehyde (5 mmol), deoxybenzoin (5 mmol), and urea (15 mmol) by examining the type of catalysts (Scheme 1). Many catalysts or promoters, such as $Zn(OAc)_2$, $FeCl_3$, $FeCl_3.6H_2O$, $AlCl_3.6H_2O$, and $Co(OAc)_2.4H_2O$, were used to explore the reaction conditions (Table I). From these results, we found that most of the Lewis acids could promote the reaction, but the yields were not very high. In comparison with other catalysts, the use of 0.25 mmol of $Co(OAc)_2.4H_2O$ could make the yield reach 69% under the microwave power (P) of 300 W and the irradiation time of 7 min. However, we found that the addition of TMSCl accelerated the Biginelli–like reaction and gave a good result. For example, the TMSCl-mediated Biginelli–like reaction of deoxybenzoine, 3-methoxybenzaldehyde, and urea gave the corresponding product in high yield. From the result, we concluded that the mixture of $Co(OAc)_2.4H_2O$ and TMSCl is the best catalyst for this reaction. The amount of $Co(OAc)_2.4H_2O$ and TMSCl was further examined, and the results are summarized in Table I. It could be seen that 0.25 mmol of $Co(OAC)_2.4H_2O$ and 2.5 mmol of TMSCl gave the best result in this reaction.

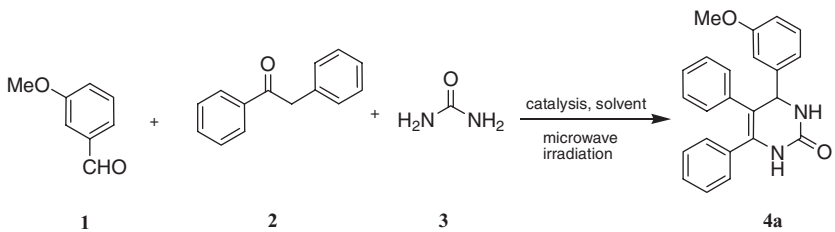
TABLE I Effects of the Type and the Amount of Catalysts on the Formation of 4a*

Entry	Catalysis (mmol)	TMSCI (mmol)	Yield (%)
1	$Zn(OAc)_2$ (0.25)	—	60
2	$Zn(OAc)_2$ (1)	—	73
3	$Zn(OAc)_2$ (1.5)	—	78
4	$Co(OAc)_2.\ 4H_2O$ (0.25)	—	69
5	$Co(OAc)_2.\ 4H_2O$ (1)	—	64
6	$Zn(OAc)_2$ (1)	2.5	69
7	$Zn(OAc)_2$ (2.5)	2.5	54
8	$Co(OAc)_2.\ 4H_2O$ (1)	2.5	82
9	$Co(OAc)_2.\ 4H_2O$ (0.25)	2.5	91
10	$Co(OAc)_2.\ 4H_2O$ (0.25)	5	60
11	$Co(OAc)_2.\ 4H_2O$ (0.25)	7.5	60
12	$Co(OAc)_2.\ 4H_2O$ (.25)	9	58
13	$FeCl_3$ (1)	5	77
14	$FeCl_3.\ 6H_2O$ (1)	5	78
15	$AlCl_3.\ 6H_2O$ (1)	5	74
16	–	2.5	52
17	–	5	57
18	–	7.5	61
19	–	9	45

*Reaction condition: 3-methoxybenzaldehyde 5 mmol, deoxybenzoin 5 mmol, urea 15 mmol, P = 300 W, DMF.

Based on the above optimized result, we further examined the effect of a stoichiometric amount of urea, type of solvent, microwave power, and the irradiation time on the Biginelli–like reaction, involving deoxybenzoin, 3-methoxybenzaldehyde, and urea to afford DHPM **4a**, as shown in Scheme 1.

It could be found that with the increase of the microwave power from 80 to 300 W, the yield of **4a** increased to 91% when the irradiation time was 7 min. However, with the microwave power of 300 W, when



1 2 3 4a

SCHEME 1 Microwave-assisted Biginelli–like reaction by various catalysts.

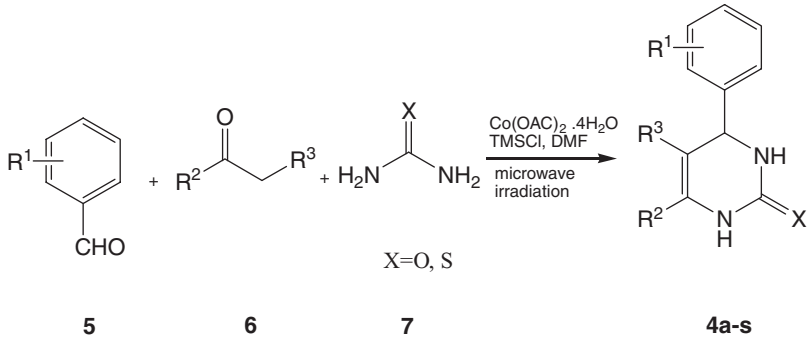
TABLE II Effects of the Microwave Power and the Irradiation Time on the Formation of 4a*

Entry	Time (min)	P(W)	Yield (%)
1	10	80	—
2	15	150	10
3	2	300	60
4	3	300	72
5	4	300	80
6	5	300	80
7	6	300	89
8	7	300	91
9	8	300	88
10	10	300	87
11	14	300	32
12	2	450	45
13	3	450	30
14	4	450	—
15	1	650	15
16	2	650	—

*Reaction condition: 3-methoxybenzaldehyde 5 mmol, deoxybenzoin 5 mmol, urea 15 mmol, $Co(OAC)_2.4H_2O$ 0.25 mmol, and TMSCl 2.5 mmol, DMF.

we increased the microwave irradiation time, the yield of **4a** increased first, then showed a slight decrease when the time was more than 7 min. So optimized microwave power and the irradiation time were 300 W and 7 min, respectively (Table II).

In order to examine the substrate scope of Biginelli–like reaction, we examined the reacation of various ketones, aromatic aldehyde, and urea or thiourea under the above optimized reaction condition (Scheme 2).



SCHEME 2 Microwave-assisted Biginelli–like reaction by various aromatic aldehydes, ketone or β-ketoester, and urea or thiourea.

TABLE III Cyclocondensation of Aromatic Aldehydes, Ketone or β-Ketoester, and Urea or Thiourea*

					Found			Reported		
Entry	R^1	R^2	R^3	X	mp (°C)	t (time)	Yield (%)	mp (°C)	t (time)	Yield (%)
4a	3-OMe	Ph	Ph	O	191–193	7	91	–	–	–
4b	3-Cl	Ph	Ph	O	199–201	7	82	–	–	–
4c	3-NO_2	Ph	Ph	O	222–224	7	96	–	–	–
4d	4-Cl	Ph	Ph	O	256–258	7	91	–	–	–
4e	H	Ph	Ph	O	240–242	7	85	–	–	–
4f	H	Ph	Ph	S	259–261	7	97	–	–	–
4g	4-Me	Ph	Ph	S	241–243	7	98	–	–	–
4h	3-OMe	Ph	Ph	S	239–241	7	95	–	–	–
4i	3-Cl	Ph	Ph	S	263–265	7	95	–	–	–
4j	3-Cl	Me	Me	O	228–230	7	80	–	–	–
4k	H	ph	H	O	218–220	7	90	218–219[21]	3 h	87
4l	4-Cl	ph	H	O	265–266	7	95	267–269[22]	8 min	93
4m	H	Me	COOEt	O	201–202	6	94	200–202[25]	3 h	90
4n	3-NO_2	Me	COOEt	O	224–226	6	97	225–227[25]	3 h	80
4o	3-Cl	Me	COOEt	O	191–192	6	95	192–194[24]	4 h	93
4p	4-OMe	Me	COOEt	O	200–202	6	91	199–201[25]	3 h	84
4q	2-Cl	Me	COOEt	O	217–219	6	90	216–218[25]	3 h	85
4r	H	Me	COOEt	S	204–205	4	96	205–206[25]	3 h	86
4s	3-NO_2	Me	COOEt	S	203–204	4	94	203–205[26]	3 h	53

*Reaction condition: aromatic aldehyde 5 mmol; ketone or β-ketoester 5 mmol; urea15 mmol or thiourea 12 mmol, $Co(OAc)_2.4H_2O$ 0.25 mmol and TMSCl 2.5 mmol, DMF, P = 300 W.

The results are shown in Table III, entries **4a–l**. From the result, we could see that all reactions afforded the corresponding DHPMs in moderate to high yield. Promoted by this success and in order to compare with other methods,[21–26] we extended this reaction of urea or thiourea with various aldehydes and ethylacetoacetate under similar conditions and obtained the respective 3,4-dihydropyrimidin-2(1H)-ones **4m–s** in good to excellent yields. The optimized results are summarized in Table III.

In conclusion, an efficient and high-yield protocol for the synthesis of new 3,4-dihydropyrimidin-2(1H)-ones involving a three-component, one-pot condensation of an aromatic aldehyde, ketones or β-ketoester, and urea or thiourea using $Co(OAc)_2.4H_2O$ and TMSCl as an efficient Lewis acid catalyst under microwave irradiation was developed and compared with other methods. Short reaction time, high yield, simple

product isolation procedure, and the use of low amount of catalyst were the advantages of this protocol.

EXPERIMENTAL

All reagents were purchased from Merck and Fluka and used without further purification. All melting points were measured on an Electrothermal IA 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu FT-IR 8600 instrument. ^{1}H and ^{13}C NMR spectra were recorded in DMSO using TMS as an internal standard on a Bruker DRX-300 Avance spectrometer at 300 MHz. Elemental analyses were carried out on a Thermo Finnigan Flash EA 1112 series instrument. All products are new compounds, which were identified by IR, ^{1}H, and ^{13}C NMR spectral data.

General Procedure

A mixture of an aromatic aldehyde **5** (5 mmol), ketone or β-ketoester **6** (5 mmol), urea (15 mmol) or thiourea **7** (12 mmol), $Co(OAc)_2.4H_2O$ (0.25 mmol), TMSCl (2.5 mmol), and DMF (5 mL) contained in a 100 mL beaker was placed in the microwave oven. The beaker was covered with a stemless funnel and subjected to irradiation conditions as shown in Table III. After the reaction was complete, it was allowed to cool to room temperature. Then distilled water (30 mL) was added into the beaker and stirred for several minutes. The precipitate thus obtained was filtered off. The crude product purified by recrystallization from ethanol and dried to give powder compounds **4a–s**.

3,4-Dihydro-4-(3-methoxyphenyl)-5,6-diphenylpyrimidin-2(1H)-one 4a

White powder; mp 191–193°C; IR (KBr) ν_{max}: 3211, 3076, 2925, 1664, 1595, 1485 cm^{-1}; ^{1}H NMR(DMSO) δ: 3.70 (s, 3H, OMe), 5.12 (s, 1H, CH), 6.81–7.28 (m, 14H, Ar-H), 7.53 (s, 1H, NH), 8.67 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 54.88, 59.18, 109.36, 112.62, 112.84, 119.06, 125.82, 127.70, 128.01, 128.13, 129.25, 129.28, 129.68, 134.79, 134.95, 138.02, 145.29, 153.18, 159.38. Anal. Calcd. For $C_{23}H_{20}N_2O_2$ (356.42): C, 77.51; H, 5.66; N, 7.86. Found: C, 77.40; H, 5.61; N, 7.88.

3,4-Dihydro-4-(3-chlorophenyl)-5,6-diphenylpyrimidin-2(1H)-one 4b

White powder; mp 199–201°C; IR (KBr) ν_{max}: 3220, 3091, 2918, 1689, 1595, 1471 cm^{-1}; ^{1}H NMR(DMSO) δ: 5.19 (s, 1H, CH), 6.78-7.38 (m,

14H, Ar-H), 7.53 (s, 1H, NH), 8.66 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 58.75, 108.94, 125.55, 125.94, 126.86, 127.39, 127.77, 127.99, 128.18, 129.26, 129.28, 130.51, 133.07, 134.75, 135.03, 137.63, 146.20, 152.85. Anal. Calcd. For $C_{22}H_{17}ClN_2O$ (360.84): C, 73.23; H, 4.75; N, 7.76. Found: C, 73.31; H, 4.69; N, 7.85.

3,4-Dihydro-4-(3-nitrophenyl)-5,6-diphenylpyrimidin-2(1H)-one 4c

Yellow powder; mp 222–224°C; IR (KBr) ν_{max}: 3232, 3097, 2928, 1702, 1651, 1530 cm^{-1}; ^{1}H NMR(DMSO) δ: 5.41 (s, 1H, CH), 6.79-8.13 (m, 14H, Ar-H), 8.19 (s, 1H, NH), 8.79 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 58.50, 108.66, 121.65, 122.48, 126.06 127.87, 128.05, 128.29, 129.31, 130.26, 133.69, 134.66, 135.44, 137.38, 145.87, 147.91, 152.80. Anal. Calcd. For $C_{22}H_{17}N_3O_3$ (371.39): C, 71.15; H, 4.61; N, 11.31. Found: C, 71.22; H, 4.57, N, 11.25.

3,4-dihydro-4-(4-chlorophenyl)-5,6-diphenylpyrimidin-2(1H)-one 4d

White powder; mp 256–258°C; IR (KBr) ν_{max}: 3211, 3060, 2906, 1690, 1596, 1490 cm^{-1}; ^{1}H NMR(DMSO) δ: 5.20 (s, 1H, CH), 6.79-7.42 (m, 14H, Ar-H), 7.56 (s, 1H, NH), 8.70 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 59.55, 110.03, 126.83, 128.69, 128.89, 129.08, 129.47, 129.78, 130.2, 132.86, 135.70, 135.74, 138.66, 143.71, 153.82. Anal. Calcd. For $C_{22}H_{17}ClN_2O$ (360.84): C, 73.23; H, 4.75; N, 7.76. Found: C, 73.27; H, 4.71; N, 7.69.

3,4-Dihydro-4,5,6-triphenylpyrimidin-2(1H)-one 4e

White powder; mp 240–242°C; IR (KBr) ν_{max}: 3220, 3082, 2918, 1699, 1595, 1481 Cm^{-1}; ^{1}H NMR(DMSO) δ: 5.15 (s, 1H, CH), 6.78-7.38 (m, 15H, Ar-H), 7.53 (s, 1H, NH), 8.66 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 59.39, 109.53, 125.79, 126.93, 127.40, 127,65, 127,93, 128,06, 128.51, 129.27, 134.61, 134.92, 137.99, 143.81, 153.08. Anal. Calcd. For $C_{22}H_{18}N_2O$ (326.39): C, 80.96; H, 5.56; N, 8.58. Found: C, 80.89; H, 5.54; N, 8.61.

3,4-dihydro-4,5,6-triphenylpyrimidin-2(1H)-thione 4f

White powder; mp 259–261°C; IR (KBr) ν_{max}: 3168, 3100, 2972, 1645, 1568, 1479 cm^{-1}; ^{1}H NMR(DMSO) δ: 5.13 (s, 1H, CH), 6.78–7.39 (m, 15H, Ar-H), 9.33 (s, 1H, NH), 9.95 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 58.82, 111.17, 126.31, 127.09, 127.80, 127.86, 128.00, 128.42, 128.72, 128.97, 129.59, 133.42, 133.81, 137.22, 142.42, 173.56. Anal. Calcd. For

$C_{22}H_{18}N_2S$ (342.46): C, 77.16; H, 5.30; N, 8.18. Found: C, 77.09; H, 5.32; N, 8.21.

3,4-Dihydro-4-(4-metylphenyl)-5,6-diphenylpyrimidin-2(1H)-thione 4g

White powder; mp 241–243°C; IR (KBr) ν_{max}: 3209, 3050, 2943, 1649, 1569, 1479 cm^{-1}; ^1H NMR(DMSO) δ: 2.28 (s, 3H, CH_3), 5.07 (s, 1H, CH), 6.79–7.28 (m, 14H, Ar-H), 9.28 (s, 1H, NH), 9.91 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 20.65, 58.59, 111.23, 126.28, 127.03, 127.79, 128.00, 128.40, 129.27, 129.59, 133.33, 133.85, 137.10, 137.31, 139.54, 173.46. Anal. Calcd. For $C_{23}H_{20}N_2S$ (356.48): C, 77.49; H, 5.65; N, 7.86. Found: C, 77.53; H, 5.62; N, 7.81.

3,4-Dihydro-4-(3-methoxyphenyl)-5,6-diphenylpyrimidin-2(1H)-thione 4h

White powder; mp 239–241°C; IR (KBr) ν_{max}: 3192, 3091, 2954, 2842, 1639, 1558, 1461 cm^{-1}; ^1H NMR(DMSO) δ: 3.71 (s, 3H, OCH_3), 5.10 (s, 1H, CH), 6.80–7.31 (m, 14H, Ar-H), 9.32 (s, 1H, NH), 9.96 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 54.95, 58.68, 111.00, 112.97, 113.05, 119.16, 126.34, 127.84, 128.07, 128.46, 128.96, 129.57, 129.87, 133.56, 133.83, 137.24, 143.84, 159.46, 173.65. Anal. Calcd. For $C_{23}H_{20}N_2OS$ (372.48): C, 74.16; H, 5.41; N, 7.52. Found: C, 74.03; H, 5.44; N, 7.50.

3,4-Dihydro-4-(3-chlorophenyl)-5,6-diphenylpyrimidin-2(1H)-thione 4i

White powder; mp 263–265°C; IR (KBr) ν_{max}: 3161, 3095, 2976, 1649, 1573, 1481 cm^{-1}; ^1H NMR(DMSO) δ: 5.20 (s, 1H, CH), 6.79–7.42 (m, 14H, Ar-H), 9.32 (s, 1H, NH), 9.96 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 58.20, 110.65, 125.69, 126.44, 127.00, 127.85, 127.90, 128.04, 128.51, 128.96, 129.56, 130.71, 133.25, 133.62, 133.78, 136.86, 144.68, 173.76. Anal. Calcd. For $C_{22}H_{17}ClN_2S$ (376.08): C, 70.11; H, 4.55; N, 7.43. Found: C, 70.21; H, 4.53; N, 7.41.

3,4-Dihydro-4-(3-chlorophenyl)-5,6-dimethylpyrimidin-2(1H)-one 4j

White powder; mp 228–230°C; IR (KBr) ν_{max}: 3238, 3085, 2914, 1687, 1587, 1489 cm^{-1}; ^1H NMR(DMSO) δ: 1.79 (s, 3H, Me), 2.12 (s, 3H, Me), 4.79 (s, 1H, CH), 7.34–7.45 (m, 4H, Ar-H), 7.63 (s, 1H, NH), 8.13 (s, 1H, NH); ^{13}C NMR (DMSO) δ: 14.40, 19.04, 59.58, 109.75, 120.20, 127.44, 128.53, 130.48, 133.50, 139.13, 146.31, 152.84. Anal. Calcd. For $C_{12}H_{13}ClN_2O$ (236.70): C, 60.89; H, 5.54; N, 11.84. Found: C, 61.01; H, 5.51; N, 11.86.

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