One-pot synthesis of 4,6-diarylpyrimidin-2(1*H*)-ones via multi-component reaction promoted by chlorotrimethylsilane

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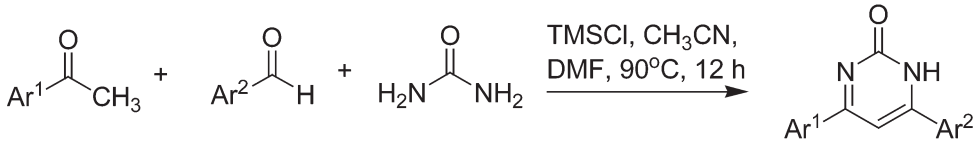
4,6-Diarylpyrimidin-2(1*H*)-ones were effectively synthesised by utilising chlorotrimethylsilane (TMSCl) as an efficient promoter in the cyclisation condensation of arylketones, substituted benzaldehydes and urea by a one-pot, three-component reaction under air in DMF/CH_3CN. The clean, mild reaction conditions, operational simplicity and high yields were attractive features of the reaction which enables a facile preparative procedure for building the pyrimidine ring.

Keywords: 4,6-diarylpyrimidin-2(1*H*)-ones, chlorotrimethylsilane, arylketones, urea, cyclisation reaction

Pyrimidine is a key structural component in bio-molecules, and substituted pyrimidines and their derivatives are a very important class of heterocyclic compound in medicinal chemistry,[1–4] which have been used widely for their antitumour,[5–7] antibacterial,[8–9] antiinflammatory, antimalarial, antihypertensive, antiasthmatic, antiprotozoan, antituberculosis and anticonvulsant activities.[10–14] Furthermore, substituted pyrimidines and their derivatives have played an important part in material science. For example, several pyrimidines are used in polymers as linear oligoheterocycles and in supramolecular chemistry.[15–18] Pyrimidines have found important new applications as the key units in conjugated polymers with many electrical and optical properties and conjugated polymers with a pyrimidine core are prospective candidates for light-emitting devices[19] and molecular wires.[20,21] On the other hand, it is well known that the pyrimidine ring is an effective building block for pyrimidine-based organic chromophores. Therefore, pyrimidine derivatives have been intensively investigated as electroluminescent materials.[22-24] Many methods for the efficient synthesis of substituted pyrimidines and their derivatives have been reported, which can be categorised as follows: the classical reaction of amidines with α,β-unsaturated ketones,[25] the reaction of urea and 1,3-diaryl-1,3-propanedione by using concentrated HCl as a catalyst,[18] the condensation of phenacyldimethylsulfonium salts, aldehydes, and ammonia,[26] the dimerisation–oxidative fragmentation of aryl-β-arylvinylimines,[27] the reaction of alkynes and nitriles promoted by TfOH,[28] the one-pot three-component reaction of aryl halides, terminal propargyl alcohols and amidinium salts based upon a coupling–isomerisation–cyclocondensation sequence,[29] the rearrangement of 2,4,5-trisubstituted-imidazolines,[30] the sequential assembly of aryl groups onto a pyrimidine core (2-methylthiopyrimidine).[31] and the microwave-assisted reaction of amidines and alkynones.[32] Although these synthetic protocols have been proven to be efficient, with the growing realisation that substituted pyrimidines and their derivatives display significant biological activities and powerful applications in materials chemistry, the development of synthetic methods which enable a facile access to the pyrimidine core are desirable. But in all these methods either expensive reagents or toxic solvents are required or the reagents used are toxic and hazardous. Therefore, easy-to-handle and highly efficient methods that the reagents used are nontoxic, inexpensive and safe reagents are still targets of pursuit. Recently multi-component reactions have attracted considerable interest from medicinal and organic chemists because they can be widely employed for the rapid assembly of arrays with high molecular diversity. These processes are performed without need to isolate any intermediate and this reduces time and saves both energy and raw materials. Moreover, many previous studies proved that chlorotrimethylsilane (TMSCl) can be used as a mild and efficient promoter for various organic transformations.[33,34] In the presence of TMSCl Lewis acid catalysts $Bi(TFA)_3$/[nbpy]$FeCl_4$[35], $H_3PMo_{12}O_{40}$[36] and $H_6P_2W_{18}O_{62}$-$18H_2O$[37] have been employed in the one-pot preparation of 4,6-diarylpyrimidin-2(1*H*)-ones. TMSCl has also been reported as a mild, useful and inexpensive Lewis acid promoter for one-pot multicomponent reactions for the preparation of other heterocyclic compounds.[38,39] In previous work,[40–43] TMSCl was employed to carry out several unusual cyclo-condensations including one-pot synthesis of subtituted quinolines, oxazine-3- ones and pyrroles. Continuing our investigations on the TMSCl-promoted synthesis of heterocycles, we were interested to prepare 4,6-diarylpyrimidin-2(1*H*)-ones from arylketones, substituted benzaldehydes and urea via the one-pot three-component reaction under air in DMF/CH_3CN using TMSCl as promoting agent in the absence of other catalysts. The results obtained are reported here (Scheme 1).

Results and discussion

The reaction was initially studied with acetophenone, urea and *p*-methoxybenzaldehyde, which were selected as suitable substrates for reaction development in various solvent in the presence of various promoters (Table 1). At the outset, various promoters and solvents were screened. We were pleased to observe the formation of the desired product **4a** (Table 2) when



Scheme 1 Chlorotrimethylsilane-promoted one-pot synthesis of 4,6-diarylpyrimidin-2(1*H*)-ones.

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Table 1 Various promoters and solvents effect on the reaction[a]

Entry	Catalyst	Solvent	Yields/%[b]
1	5% HCl (0.5mL)	DMF (20 mL)	38
2	PTSA (3 mmol)	DMF (20 mL)	25
3	$ZnCl_2$ (3 mmol)	DMF (20 mL)	59
4	BF_3Et_2O (3 mmol)	DMF (20 mL)	57
5	TMSCl (1.5 mmol)	DMF (20 mL)	62
6	TMSCl (3.3 mmol)	DMF (20 mL)	74
7	TMSCl (3.3 mmol)	DMSO (20 mL)	69
8	TMSCl (3.3 mmol)	dioxane (20 mL)	26
9	TMSCl (3.3 mmol)	DMF/CH_3CN (6/24 mL mL^{-1})	76

[a] Acetophenone (3 mmol), *p*-methoxybenzaldehyde (3 mmol) and urea (4 mmol); 90 °C; 12 h.
[b] Isolated yields.

Table 2 TMSCl-promoted one-pot synthesis of 4,6-diarylpyrimidin-2(1*H*)-ones

Entry	Ketones (Ar^1)	Aldehydes (Ar^2)	Pyrimidines (**4**)	Yield/%
1	*p*-$MeOC_6H_4$	C_6H_5	**4a**	78
2	C_6H_5	*p*-$MeOC_6H_4$	**4a**	76
3	*p*-$MeOC_6H_4$	*p*-$MeOC_6H_4$	**4b**	88
4	C_6H_5	C_6H_5	**4c**	80
5	*p*-MeC_6H_4	*p*-MeC_6H_4	**4d**	85
6	*p*-$MeOC_6H_4$	*3,4,5*-$(MeO)_3C_6H_2$	**4e**	80
7	*o*-BrC_6H_4	*p*-$MeOC_6H_4$	**4f**	76
8	*p*-$O_2NC_6H_4$	*p*-$MeOC_6H_4$	**4g**	85
9	*p*-$MeOC_6H_4$	*p*-$O_2NC_6H_4$	**4g**	83
10	*p*-$O_2NC_6H_4$	*p*-$^iPrC_6H_4$	**4h**	73
11	*p*-$O_2NC_6H_4$	C_6H_5	**4i**	70

the reaction was carried out using *p*-methoxybenzaldehyde (3 mmol), urea (4 mmol), acetophenone (3 mmol), DMF/CH_3CN (6/24 mL mL^{-1}) in the presence of various promoters at 90 °C for 12 h. The result indicated that TMSCl is an efficient promoter (Table 1, entry 5, 62% in yield using 1.5 mmol TMSCl and entry 6, 74% in yield using 3.3 mmol TMSCl). A comparison of the method using TMSCl as a promoter (Table 1, entry 6, 74% in yield), with selected other promoters such as hydrochloric acid, PTSA or another Lewis acid ($ZnCl_2$, $BF_3{\cdot}Et_2O$) (Table 1, entry 1, 38% in yield; entry 2, 25% in yield; entry 3, 59% in yield; entry 4, 57% in yield, respectively) that were examined is collected in Table 1 to demonstrate that the method using TMSCl as a promoter is indeed superior to several of the other protocols. Thus, TMSCl was found to be the better choice for this reaction.

To optimise the reaction conditions, solvent and time were varied. Firstly the solvent in the preparation of 4-(4-methoxyphenyl)-6-phenylpyrimidin-2(1*H*)-one (**4a**) from acetophenone (3 mmol), *p*-methoxybenzaldehyde (3 mmol) and urea (4 mmol) in the presence of TMSCl (3.3 mmol) was varied. Among the solvents tested (Table 1, entries 6-9), DMF and DMSO gave better results. The result showed that DMF and DMSO gave the product **4a** in 74% and 69% yield, respectively. Although the reaction mixture was insoluble in MeCN alone, the solvent mixture DMF/CH_3CN (6/24 mL mL^{-1}) gave the best result of 76% yield. In the second set of experimental work, the model reaction with acetophenone, *p*-methoxybenzaldehyde and urea in DMF/CH_3CN was carried out at various reaction temperatures. After some experimentation, it was found that the model reaction using the reaction temperature 90 °C produced the corresponding compound **4a** in excellent yield. Furthermore, the reaction time could be reduced to 12 h. Thus, with these results in hand, we synthesised nine 4,6-diarylpyrimidin-2(*H*)-ones (Table 2, **4a–i**) in yields varying from 70 to 88% by the cyclo-condensation reaction of 3.0 mmol appropriate acetophenone and 3.0 mmol appropriate aromatic aldehyde in DMF/CH_3CN with 4.0 mmol urea in the presence of 3.3 mmol TMSCl at 90 °C for 12 h (Scheme 1).

In summary, a novel, efficient and one-pot synthesis has been described for the preparation of 4,6-diarylpyrimidin-2(1*H*)-ones in three-component cyclo-condensation reactions of appropriate acetophenones and appropriate aromatic aldehydes and urea, which involves the use of the inexpensive and relatively nontoxic reagent TMSCl as a promoter. The novelty and synthetic utility of this methodology was demonstrated in the efficient synthesis of pyrimidine derivatives.

Experimental

Elemental analytical data were obtained by using a Perkin-Elmer model 240 elemental analyser, IR spectra were measured with a Shimadzu model 408 IR spectrometer, ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a JNM-90Q spectrometer using TMS as an internal standard.

*4-(4-Methoxyphenyl)-6-phenylpyrimidin-2(1*H*)-one* (**4a**):[36] To a mixture of *p*-methoxyacetophenone (450 mg, 3 mmol), benzaldehyde (318 mg, 3 mmol) and urea (240 mg, 4 mmol) in DMF/CH_3CN (6/24 mL mL^{-1}) was added TMSCl (0.4 mL, 3.3 mmol) at room temperature. The resulting mixture was heated at 90 °C for 12 h. After removal of the solvent under reduced pressure, the residue was dissolved in CH_2Cl_2 (30 mL) and was washed with water and brine and dried over Na_2SO_4. The solvent was removed under reduced pressure and recrystallisation of the crude product with MeOH-CH_3CN gave 4-(4-methoxy)phenyl-6-phenylpyrimidin-2(1*H*)-one (651 mg, 78% yield), white solid, m.p. 258–260 °C (CH_2Cl_2/MeOH); IR (KBr) υ 3436, 1609, 1574, 1514, 1262, 1178 cm^{-1}; ^{1}H NMR (300 MHz, CD_3OD, ppm) δ 8.08–8.05 (m, 4H), 8.03–8.01 (m, 3H), 7.30 (s, 1H), 7.08 (d, *J* = 9.0 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (75 MHz, pyridine-d_5, ppm) 166.5, 166.0, 163.3, 137.1, 131.9, 130.3(2C), 129.7(2C), 129.3, 128.5(2C), 126.7, 115.2(2C), 101.7, 55.9; Anal. Calcd for $C_{17}H_{14}N_2O_2$: C, 73.37; H, 5.07; N, 10.07. Found: C, 73.19; H, 5.22; N, 10.02%.

*4,6-Bis(4-methoxyphenyl)pyrimidin-2(1*H*)-one* (**4b**): 88% yield, orange solid, m.p. >300 °C; IR (KBr) υ 3437, 1599, 1513, 1445, 1427, 1255, 1180, 820; ^{1}H NMR (300 MHz, CD_3OD, ppm) δ 8.08 (d, *J* = 9.0 Hz, 2 × 2H), 7.44 (s, 1H), 7.21 (d, *J* = 9.0 Hz, 2 × 2H), 3.95 (s, 2 × 3H); ^{13}C NMR (75 MHz, CD_3OD, ppm) δ 167.0, 165.9 (2C), 162.6, 132.5 (4C), 123.0 (2C), 116.6 (4C), 100.5 (2C), 56.7 (2C); Anal. Calcd for $C_{18}H_{16}N_2O_3$: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.03; H, 5.40; N, 9.04%.

*4,6-Diphenylpyrimidin-2(1*H*)-one* (**4c**):[44] 80% yield, yellow powder, m.p. 292–293 °C (m.p. 292–294 °C[44]).

*4,6-Di(p-tolyl)pyrimidin-2(1*H*)-one* (**4d**):[18] 85% yield, yellow powder, m.p. 274–275 °C (m.p. > 270 °C[18]) : IR (KBr) υ 3432, 1595, 1510, 1445, 1433, 1256, 1200, 826 cm^{-1}; ^{1}H NMR (DMSO-d_6, ppm) δ 8.05 (d, 4H, *J* = 8.1 Hz, ArH), 7.57 (s, 1H, =CH–), 7.45 (d, 4H, *J* = 8.1 Hz, ArH), 4.82 (s, 1H, –OH), 2.40 (s, 6H, $–CH_3$); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 167.2, 160.2 (2C), 142.7 (2C), 135.5 (2C), 130.6 (2C), 130.5 (2C), 129.2 (4C), 96.2, 26.3(2C); Anal. Calcd for $C_{18}H_{16}N_2O$: C, 78.24; H, 5.84; N, 10.14. Found: C, 78.02; H, 5.60; N, 10.30%.

*6-(3,4,5-Trimethoxyphenyl)-4-(4-methoxyphenyl)pyrimidin-2(1*H*)-one* (**4e**): 80% yield, white solid, m.p. 288–290 °C (methanol); IR (KBr) υ 3437, 1630, 1604, 1579, 1505, 1257, 1127; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 8.18 (d, *J* = 8.4 Hz, 2H), 7.47 (s, 1H), 7.42 (s, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 2 × 3H), 3.86 (s, 3H), 3.75 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 165.2, 164.4, 161.7, 158.7 (2C), 152.7, 139.9, 129.2 (2C), 128.2, 127.3, 117.8 (2C), 104.7 (2C), 98.3, 59.8 (2C), 55.8, 55.1; Anal. Calcd for $C_{20}H_{20}N_2O_5$: C, 65.21; H, 5.47; N, 7.60. Found: C, 65.12; H, 5.40; N, 7.66%.

*4-(2-Bromophenyl)-6-(4-methoxyphenyl)pyrimidin-2(1*H*)-one* (**4f**): 76% yield, yellow powder, m.p. 260–262 °C (EtOAc/hexanes); IR (KBr) υ 3437, 1633, 1602, 1580, 1505, 1257, 1129; ^{1}H NMR (300 MHz, CD_3OD, ppm) δ 8.12–8.00 (m, 3H), 7.67–7.51 (m, 3H), 7.30 (s, 1H), 7.07 (d, *J* = 9.0 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 167.1, 163.2, 162.8, 160.1, 147.2, 140.2, 139.4, 138.8 (2C), 136.9, 132.4, 132.0, 130.2, 129.0 (2C), 97.5, 55.8; Anal. Calcd for $C_{17}H_{13}BrN_2O_2$: C, 57.16; H, 3.67; N, 7.84. Found: C, 57.10; H, 3.48; N, 7.68%.

4-(4-Methoxyphenyl)-6-(4-nitrophenyl)pyrimidin-2(1H)-one (**4g**): 85% yield, yellow solid, m.p. >300 °C (DMSO); IR (KBr) υ 3437, 1677, 1605, 1573, 1518, 1349, 1128; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 8.47 (d, *J* = 8.1 Hz, 2H), 8.38 (d, *J* = 8.7 Hz, 2H), 8.20 (d, *J* = 8.4 Hz, 2H), 7.77 (s, 1H), 7.12 (d, *J* = 8.4 Hz, 2H), 4.98 (s, 1H), 3.87 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 167.7, 164.0, 163.2, 158.7, 152.6, 139.9, 129.0 (2C), 128.6 (2C), 127.2, 120.2 (2C), 118.4 (2C), 97.6, 55.8; Anal. Calcd for $C_{17}H_{13}N_3O_4$: C, 63.15; H, 4.05; N, 13.00. Found: C, 63.12; H, 4.20; N, 12.88%.

4-(4-Isopropylphenyl)-6-(4-nitrophenyl)pyrimidin-2(1H)-one (**4h**): 73% yield, white solid, m.p. >300 °C (DMSO); IR (KBr) υ 3436, 1618, 1523, 1449, 1350, 994, 821; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 8.48 (d, *J* = 8.1 Hz, 2H), 8.38 (d, *J* = 8.7 Hz, 2H), 8.10 (d, *J* = 8.4 Hz, 2H), 7.75 (s, 1H), 7.38 (d, *J* = 8.4 Hz, 2H), 4.95 (s, 1H), 3.28 (m, 1H), 1.34 (d, *J* = 10.2 Hz, 6H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 167.7, 164.2, 158.7, 152.6, 152.0, 146.3, 139.2, 129.8 (2C), 128.6 (2C), 128.0 (2C), 124.2 (2C), 96.8, 40.8, 24.8 (2C); Anal. Calcd for $C_{19}H_{17}N_3O_3$: C, 68.05; H, 5.11; N, 12.53. Found: C, 68.24; H, 5.02; N, 12.76%.

4-(4-Nitrophenyl)-6-phenylpyrimidin-2(1H)-one (**4i**): 70% yield, yellow solid, m.p. > 300 °C (DMSO); IR (KBr) υ 3416, 1676, 1613, 1515, 1351, 998, 758; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 8.50 (d, *J* = 8.1 Hz, 2H), 8.29 (d, *J* = 8.7 Hz, 2H), 8.08 (d, *J* = 8.4 Hz, 2H), 7.76 (s, 1H), 7.60–7.26 (m, 3H), 4.95 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 167.8, 164.0, 158.9, 152.4, 142.2, 139.2, 136.2 (2C), 130.2, 129.9 (2C), 128.8 (2C), 125.2 (2C), 96.8; Anal. Calcd for $C_{16}H_{11}N_3O_3$: C, 65.53; H, 3.78; N, 14.33. Found: C, 65.26; H, 4.02; N, 14.16%.

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