Synthesis, fluorescence properties and Zn^{2+} recognition of 4-Aryl-6-phenylpyrimidin-2(1*H*)-one

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4-Aryl-6-phenylpyrimidin-2(1*H*)-ones were synthesised via a three-component one-pot cyclocondensation of aromatic aldehyde, acetophenone and urea catalysed by 0.2 ml (0.024 mol%) of concentration HCl in ionic liquid [BMIM][BF_4]. The fluorescence properties of product and partial products' efficient sensing of zinc ion were studied in detail. The structure of 4,6-diphenylpyrimidin-2(1*H*)-one was confirmed by X-ray analysis.

Keywords: 4-aryl-6-phenylpyrimidin-2(1*H*)-ones, fluorescence properties, Zn^{2+} recognition, ionic liquid, synthesis

Zinc is the second abundant transition metal in human body only inferior to iron and plays an important role in various biological processes.[1] Generally, the concentration of Zn^{2+} in the human body is different in various physiological environments, and many other cations usually coexist with Zn^{2+} in these environments. For example, the concentration of intracellular Zn^{2+} in serum is *ca* 12 μM,[2] and that value in the grey matter and brain tissue becomes *ca* 0.1–0.5 mM.[3-4] Therefore, a sensitive and harmless technology to detect Zn^{2+} in living cells, especially in the presence of possible competing cations, is very important. Because Zn^{2+} dose not give any spectroscopic or magnetic signals due to its $3d^{10}4\ s^0$ electronic configuration, the detection of Zn^{2+} in biological systems cannot be measured by the common analytical techniques such as UV-Vis spectroscopy, Mössbauer spectroscopy, NMR and EPR spectroscopy. Therefore, the fluorescence spectroscopy is regarded as a good choice for the real-time and real-space detection of Zn^{2+} in living cells without damaging them.[5]

Pyrimidinone derivatives display various biological[6-7] and pharmacological[8-9] activities such as antitumour action. Experimentally, this skeleton can coordinate with metal ions such as Zn^{2+}, so it may be suitable for recognition of Zn^{2+}. Therefore, the synthesis of these heterocycle is interesting for both organic synthesis and medical chemistry. There are only few methods for the preparation of 4,6-diarylprimidine-2(1*H*)-ones. The same starting materials urea and chalcone have been used by Sedova's[10] and Fathalla's group[11] to prepare 4,6-diarylpyrimidin-2(1*H*)-ones in the presence of HCl. However, the two methods have some drawbacks such as harsh reaction conditions and long reaction time (boiled for 7.5 h[10] or refluxed for 12 h[11]); use of toxic and volatile organic solvents; use of excessive HCl (5 ml), which equires an excessive base as antiacid; low to moderate yields and low selectivity that limit these methods to small-scale synthesis. Khosropour and co-workers developed a three-component one-pot method to synthesise these compounds under microwave irradiation,[12] with costly and unavailable catalysts (TCT(2,4,6-trichloro-1,3,5-triazine)/$Zn(OTf)_2$ or $Bi(OTf)_3$). Use of microwave irradiation limits its industrialisation.

During the past two decades ionic liquids have gained increasing attention for performing all types of reaction with sometimes remarkable results.[13,14] They have many properties which make them of fundamental interest to all chemists. Therefore, there are many good reasons to study ionic liquids as alternative solvents in multi-component reaction. Here we report a simple three-component one-pot cyclocondensation of aromatic aldehyde, acetophenone and urea, catalysed by 0.024 mol% concentrated HCl in ionic liquid [BMIM][BF_4], to afford 4-aryl-6-phenyl pyrimidin-2(1*H*)-one **4** (Scheme 1), the X-ray structure of compound **4a** was determined and is shown in Fig. 1. The results of further experiments indicated that all products showed interesting fluorescent properties. Moreover, products have electron-donating group in *para* position (**4b, 4c, 4d, 4k**) provided an efficient fluorescence response to Zn^2

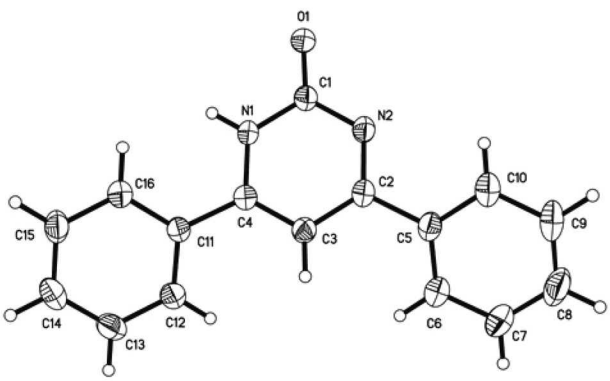
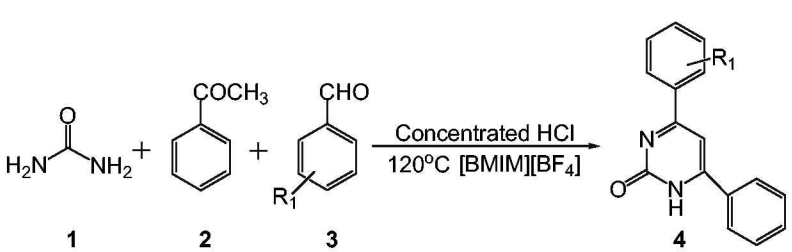


Fig. 1 X-ray structure of compound **4a**.



Scheme 1 Synthesis of 4-aryl-6-phenylpyrimidin-2(1*H*)-one.

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Table 1 Synthesis of 4-phenyl-6-phenylpyrimidin-2(1*H*)-one under different conditions[a]

Entry	Solvent	H_2SO_4		SSA[b]		HCl	
		mol%	yield/%	mol%	yield/%	mol%	Yield/%
1	H_2O	0.024	–	0.024	–	0.024	–
2	EtOH	0.024	–	0.024	–	0.024	–
3	MeOH	0.024	–	0.024	–	0.024	–
4	[BMIM][BF_4]	0.012	10	0.012	–	0.012	50
5	[BMIM][BF_4]	0.024	15	0.024	–	0.024	95
6	[BMIM][BF_4]	0.048	20	0.048	–	0.048	85
7	[BMIM][BF_4]	0.060	15	0.060	–	0.060	70

[a]Reaction conditions: benzaldehyde (2.0 mmol), acetophenone (2.0 mmol) and urea (4.0 mmol) at 120°C. [b]Silica sulfonic acid.

At the onset of this research, an effort was made to establish the optimal conditions for the blank reaction of benzaldehyde, acetophenone and urea, the results are summarised in Table 1. Interestingly, this reaction showed an intriguing solvent effect. Compared with classic organic solvent MeOH, EtOH and H_2O, [BMIM] [BF_4] (1-butyl-3-methylimidazolium tetrafluoroborate) afforded the highest yield of the desired product. [BMIM][BF_4] is not only a solvent but is also a catalyst, so only a little amount of concentrated HCl was needed as catalyst. The effect of different catalysts on yields was also tested and the results indicated that the catalytic activity of concentrated HCl was better than that of H_2SO_4 and SSA (silica sulfonic acid). This may be due to the strong electron withdrawing ability of chloride ion, which reduced the density of the electron cloud around hydrogen ion. So hydrogen ion could accept the electron more easily enabling the reaction to proceed smoothly.

In order to examine the substrate scope of this reaction, aromatic aldehydes with different substitutes were used under the above-optimised reaction conditions (Scheme 1, Table 2). From Table 2, all reactions proceeded smoothly to afforded the corresponding 4-aryl-6-phenylpyrimidin-2(1*H*)-ones in moderate to high yields.

The fluorescence properties of products were shown in Table 2 with notable fluorescence efficiency. From Table 2, **4d** exhibits strong blue-violet fluorescence in EtOH with the biggest fluorescence quantum yield (Φ_F = 0.66, reference to 9,10-diphenylanthracence), which showed its potential application as a fluorescent probe.

4d was chosen as sensor of Zn^{2+} because of its biggest fluorescence quantum yield. The concentration of Zn^{2+} used here is 2.0×10^{-5} mol·l^{-1}, which is similar to the concentration of intracellular Zn^{2+} in serum. It is noteworthy that this sensing process can be readily distinguished by eyes. **4d** was colourless without Zn^{2+}, but became yellow with the addition of Zn^{2+}. A quantitative study on the sensing ability of **4d** for Zn^{2+} was performed by fluorescence titration. As can be seen in Fig. 2, with the stepwise addition of Zn^{2+} to a solution of **4d**, the fluorescence emission at 429 nm gradually increases. To explore the possible coordination mode of **4d** with Zn^{2+}, some further experiments were performed when 4-aryl-6-phenyl-pyrimidin-2(1*H*)-ones without electron-donating groups in *para* position were used, the results showed no appreciable fluorescence sensing abilities for Zn^{2+} under the same conditions, which indicated steric and electronic influence of substitutes. We then deduce that the possible coordination mode of **4d** with Zn^{2+} as shown in Scheme 2. In addition, the Zn^{2+} sensing ability of **4d** at different pH values was also investigated (Fig. 3). The results show that **4d** exhibits poor sensing ability for Zn^{2+} at a pH value below 5.8, which may be due to the protonation of the N atom of **4d** in the acidic environment leading to a weak coordination ability of Zn^{2+} with **4d**, but exhibited satisfactory Zn^{2+} sensing ability at a pH range of 7.4–10.6.

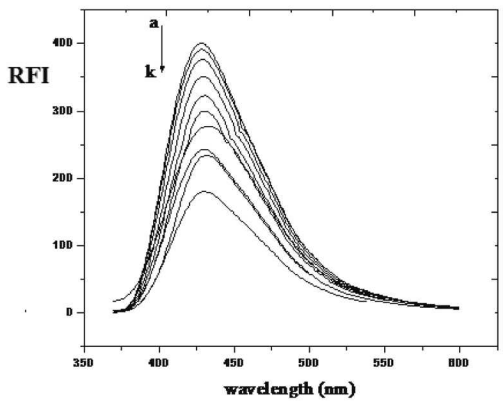
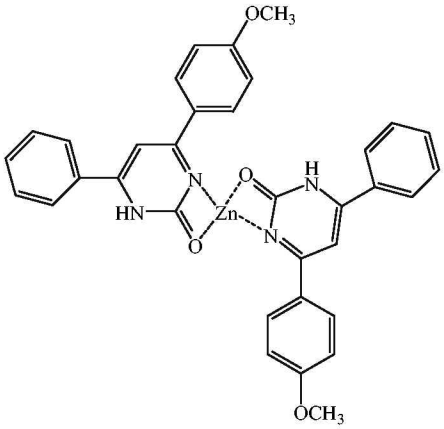


Fig. 2 Fluorescence spectral changes of **4d** (3.4×10^{-5} M) upon addition of Zn^{2+} ([Zn^{2+}] = 1.0×10^{-5} M, 2.0×10^{-5} M, 3.4×10^{-5} M, 4.0×10^{-5} M, 7.0×10^{-5} M, 10×10^{-5} M, 20×10^{-5} M, 50×10^{-5} M, 70×10^{-5} M from k to a) in Tris-HCl buffer solution (pH = 7.40) at 298.15 K.

Table 2 Synthesis and fluorescence properties of **4**

Entry	R_1	Product	Time/h	Yield/%	M.p. (°C)	λ_{ex}/nm	λ_{em}/nm	RFI[a]	Φ_F[b]
1	H	**4a**	3.5	95	227–228	260	370	42	0.10
2	4-Cl	**4b**	5	80	239–228	347	409	867	0.45
3	4-CH_3	**4c**	5	95	>300	346	408	1162	0.50
4	4-OCH_3	**4d**	5	80	244–245	362	405	882	0.66
5	2-OCH_3	**4e**	5	80	219–220	271	454	49	0.10
6	4-OH	**4f**	5	95	264–265	256	394	20	0.09
7	4-Br	**4g**	5	80	267–268	260	361	36	0.10
8	4-CN	**4h**	5	90	278–278	265	420	307	0.16
9	2,3-OCH_3	**4i**	5	80	242–242	307	389	99	0.13
10	3,4-OCH_3	**4j**	5	81	242–243	232	400	42	0.14
11	2,4-Cl	**4k**	5	79	228–229	331	408	227	0.21
12	3,4,5-OCH_3	**4l**	5	70	228–229	242	394	40	0.11
13	4-NO_2	**4m**	5	70	>300	253	359	27	0.08
14	2-Cl	**4n**	5	75	223–224	256	358	38	0.09

[a]Relative fluorescence intensity; [b]fluorescence quantum yield.



Scheme 2 Possible transition states.

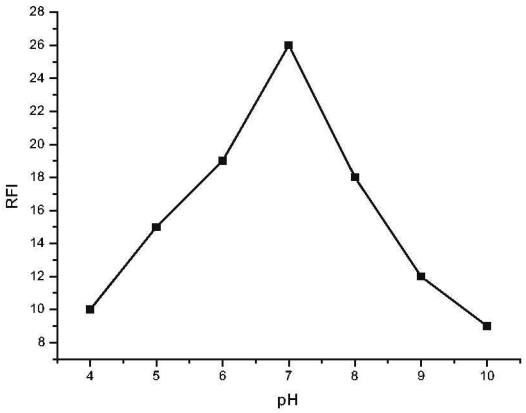


Fig 3 Influence of pH on Zn^{2+} determination.

After validating the good Zn^{2+} sensing ability of **4d**, the sensing selectivity of **4d** for Zn^{2+} was also investigated through a comparative study on the fluorescence responses of **4d** to different metal ions ($\lambda_{ex}/\lambda_{em}$ = 362 nm/405 nm). From Fig. 4, **4d** shows significant response to Zn^{2+} among the metal ions investigated, while its group 12 homologues, Cd^{2+}, and Hg^{2+}, only exhibit slight fluorescence responses under the same conditions. On the other hand, the fluorescence of **4d** shows no appreciable changes or slightly quenches with the addition of Na^+, Ca^{2+}, Hg^{2+}, Cu^{2+}, Pb^{2+}, Ba^{2+}, Fe^{2+}, Cd^{2+}, Ag^+ and Cr^{3+} (the possible competing cations when Zn^{2+} sensors are used in physiological studies). These results unambiguously demonstrate the applicability of **4d** as an efficient and special sensor to Zn^{2+}.

In summary, 4-aryl-6-phenyl-pyrimidin-2(1*H*)-ones have been synthesised from simple and available starting materials in one pot. The fluorescence properties of products were determined. The results indicated that products have electron-donating group in *para* position which presents the obvious fluorescence emission and can be readily monitored by both eyes and fluorescence spectroscopy in the presence of Zn^{2+}. The fluorescence sensing to Zn^{2+} of **4d** was studied in detail to develop a new efficient sensor to Zn^{2+}.

Experimental

All reagents were purchased from commercial sources and used without further purification. TLC analysis was performed with glass backed plates precoated with silica gel and examined under UV (254 nm). NMR spectra were measured in DMSO-d_6 with Me_4Si as the internal standards on a Bruker advance DPX-400 at room temperature. IR spectra were reported on Bruker FTIR spectrometer, absorbance is reported in cm^{-1}. Mass spectra was determined by using a Bruker MicrOTOF-QII high resolution mass spectrometer. The X-ray

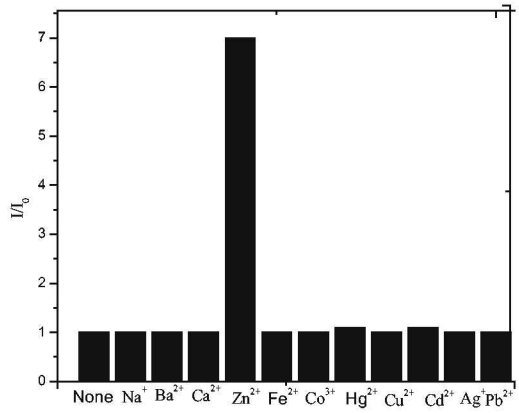


Fig. 4 Fluorescence responses of **4d** to different metal ions in Tris-HCl buffer solution (pH = 7.4). E_x = 362 nm, E_m = 429 nm, the concentration of **4d** and all of metal ions is 3.4×10^{-5} M.

structure determination for complex **4a** was given by Smartapex Bruke diffractometer using SHELXS-97 (Sheldrick, 1990) for structure solution and HELXL-97 (Sheldrick, 1997) for structure refinement. Elemental analyses were performed on a Perkin-Elmer-2400 elemental analyser. Fluorescence properties were reported on F-4500 Fluorescence Spectrophotometer (Hitachi, Japan).

General procedure for preparation of **4**

A mixture of acetophenone (2.0 mmol), urea (4.0 mmol), aromatic aldehyde (2.0 mmol) and 0.2 ml of concentrated HCl (36.5%, 0.24 mmol) was stirred in 0.5 ml of [BMIM][BF_4] at 120 °C for 3.5–5 h. The reaction mixture was then quenched water and the products were isolated by filtration followed by purification through simple recystallisation with EtOH–DMF.

4-phenyl-6-phenylpyrimidin-2(1H)-one **(4a)**: IR (KBr, ν, cm^{-1}): 3321, 2905, 1654, 1603, 770; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.17 (d, *J* = 7.2 Hz, 4H, ArH), 7.58–7.68 (m, 7H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 164.90, 157.00, 133.217, 132.29, 128.94, 128.05, 100.83, 56.03; Calcd for $C_{16}H_{12}N_2O$: C, 77.40; H, 4.87; N, 11.28. Found: C, 77.20; H, 5.11; N, 11.19%. MS (ESI) [M + H] found (expected): 249.1141 (249.1029).

4-(4-Chlorophenyl)-6-phenylpyrimidin-2(1H)-one **(4b)**: IR (KBr, ν, cm^{-1}): 3410, 2894, 1644, 1492, 818, 774; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.04 (br, s, 1H, NH), 8.11–8.20 (m, 4H, ArH), 7.49–7.60 (m, 6H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 160.20, 144.93, 135.65, 129.01, 127.75, 109.64; Calcd for $C_{16}H_{11}ClN_2O$: C, 67.97; H, 3.92; N, 9.91. Found: C, 68.20; H, 4.28; N, 9.60%. MS (ESI) [M + H] found (expected): 283.0625 (283.0639).

6-Phenyl-4-p-tolylpyrimidin-2(1H)-one **(4c)**: IR (KBr, ν, cm^{-1}): 3410, 2794, 1674, 1592, 848, 773; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.11–8.20 (m, 4H, ArH), 7.49–7.60 (m, 6H, ArH), 2.36 (s, 3H, CH), ^{13}C NMR (100 MHz, DMSO-d_6) δ: 142.10, 131.90, 129.91, 129.28, 128.02, 128.00, 21.49; Calcd for $C_{17}H_{14}N_2O$: C, 77.84; H, 5.38; N, 10.68. Found: C, 77.03; H, 5.13; N, 10.77%. MS (ESI) [M + H] found (expected): 263.1198 (263.1148).

4-(4-Methoxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4d)**: IR (KBr, ν, cm^{-1}): 3320, 3096, 1615, 1513, 809, 744; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.09 (tri, 2H, ArH), 7.99 (d, *J* = 7.6 Hz, 2H, ArH), 7.59 (d, *J* = 5.2 Hz, 3H, ArH), 7.39 (d, *J* = 8.0 Hz, 2H, ArH), 7.14 (m, 1H, ArH), 2.48 (s, 3H, CH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 162.28, 131.56, 129.56, 128.98, 127.70, 117.40, 55.66; Calcd for $C_{17}H_{14}N_2O_2$: C, 73.37; H, 5.07; N, 10.07. Found: C, 73.15; H, 4.80; N, 10.00%. MS (ESI) [M + H] found (expected): 279.1268 (279.1134).

4-(2-Methoxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4e)**: IR (KBr, ν, cm^{-1}): 3320, 3096, 1615, 1513, 809, 744; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.19 (tri, 2H, ArH), 7.97 (d, *J* = 7.6, 2H, ArH), 7.56 (d, *J* = 5.2 Hz, 3H, ArH), 7.39 (d, *J* = 8.0 Hz, 2H, ArH), 7.24(m, 1H, ArH), 2.58(s, 3H, CH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 162.58, 137.60, 129.66, 127.98, 127.80, 117.90, 55.56; Calcd for $C_{17}H_{14}N_2O_2$: C, 73.37; H, 5.07; N, 10.07. Found: C, 73.44; H, 4.77; N, 10.48. MS (ESI) [M + H] found (expected): 279.1185 (279.1134)%.

4-(4-Hydroxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4f)**: IR (KBr, ν,cm^{-1}): 3331, 3300, 2928, 1659, 1616, 779; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.88 (br, s, 1H, NH), 10.21 (s, 1H, OH), 8.08 (m, 4H, ArH), 7.56 (d, *J* = 7.2 Hz, 3H, ArH), 7.42 (s, 1H, ArH), 6.91 (d, *J* = 8.8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 161.02, 131.50, 129.70, 128.96, 127,67, 115.77; Calcd for $C_{16}H_{12}N_2O_2$: C,

72.72; H, 4.58; N, 10.60. Found: C, 72.42; H, 4.58; N, 10.50%. MS (ESI) [M + Na] found (expected): 287.0773 (287.0796).

4-(4-Bromophenyl)-6-phenylpyrimidin-2(1H)-one **(4g)**: IR (KBr, ν, cm^{-1}): 3401, 2876, 1698, 754; ^1H NMR (400 MHz, DMSO-d_6) δ: 8.16 (d, J = 8.4 Hz, 4H, ArH), 7.77 (d, J = 8.4 Hz, 2H, ArH); 7.56–7.66 (m, 4H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 160.68, 134.00, 131.98, 131.69, 129.78, 129.02, 127.74, 125.43; Calcd for $C_{16}H_{11}BrN_2O$: C, 58.74; H, 3.39; N, 8.56. Found: C, 58.77; H, 3.69; N, 8.44%. MS (ESI) [M + H] found (expected): 327.0096 (327.0134).

4-(1,2-Dihydro-2-oxo-6-phenylpyrimidine-4-yl)benzonitrile **(4h)**: IR (KBr, ν, cm^{-1}): 3420, 2894, 1620, 1680; ^1H NMR (400 MHz, DMSO-d_6) δ: 12.22 (br, s, 1H, NH), 8.41 (d, J = 8.0 Hz, 2H, ArH), 8.19 (d, J = 6.4 Hz, 2H, ArH), 8.05 (d, J = 8.4 Hz, 2H, ArH), 7.59 (m, 4H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ:159.70, 132.91, 129.06, 128.52, 127.77, 119.63, 118.63, 117.06, 113.71, 111.97; $C_{17}H_{11}N_3O_2$: C, 74.71; H, 4.06; N, 15.38. Found: C, 74.71; H, 4.29; N, 15.06%. MS (ESI) [M + H] found (expected): 274.0989 (274.0981).

4-(2,3-Dimethoxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4i)**: IR (KBr, ν, cm^{-1}): 3389, 2935, 1662, 1535, 1128, 868, 770; ^1H NMR (400 MHz, DMSO-d_6) δ: 8.00 (d, J = 6.8 Hz, 2H, ArH), 7.75 (d, J = 5.6 Hz, 1H, ArH), 7.56 (m, 4H, ArH), 7.09 (d, J = 8.0 Hz, 1H, ArH), 6.83 (t, 1H, ArH), 3.77 (s, 3H, OCH_3), 2.46 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 149.12, 132.07, 129.12, 127.99, 120.61, 118.03, 115.95, 56.00, 55.97; Calcd for $C_{18}H_{16}N_2O_3$: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.01; H, 5.08; N, 8.89%. MS (ESI) [M + H] found (expected): 309.1185 (309.1239).

4-(3,4-Dimethoxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4j)**: IR (KBr, ν, cm^{-1}):3200, 2835, 1575, 1169, 838, 762; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.95 (br, s, 1H, NH), 8.15 (d, J = 6.4 Hz, 2H, ArH), 7.82 (d, J = 8.4 Hz, 1H, ArH), 7.73 (s, 1H, ArH), 7.57 (m, 4H, ArH), 7.12 (d, J = 8.4 Hz, 1H, ArH), 3.88 (d, 6H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 145.02, 136.07, 128.12, 126.99, 121.61, 117.03, 116.95, 57.00, 55.96; Calcd for $C_{18}H_{16}N_2O_3$: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.01; H, 5.08; N, 8.89%. MS (ESI) [M + H] found (expected): 309.1338 (309.1240).

4-(2,4-Dichlorophenyl)-6-phenylpyrimidin-2(1H)-one **(4k)**: IR (KBr, ν, cm^{-1}): 3420, 2899, 1634, 1498, 828, 778; ^1H NMR (400 MHz, DMSO-d_6) δ: 12.04 (br, s, 1H, NH), 8.11–8.20 (m, 4H, ArH), 7.49–7.60 (m, 5H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 160.20, 144.93, 135.65,132.00, 129.00, 127.05, 117,26; Calcd for $C_{16}H_{10}Cl_2N_2O$: C, 60.59; H, 3.18; N, 8.83; Found: C, 60.45; H, 3.15; N, 8.59%. MS (ESI) [M + H] found (expected): 317.0515 (317.0249).

4-(3,4,5-Trimethoxyphenyl)-6-phenylpyrimidin-2(1H)-one **(4l)**: IR (KBr, ν, cm^{-1}): 3283, 2840, 1634, 1535, 1128, 868, 770; ^1H NMR (400 MHz, DMSO-d_6) δ: 12.00 (br, s, 1H, NH), 8.12 (d, J = 6.0 Hz, 2H, ArH), 7.53 (m, 6H, ArH), 3.88 (d, 9H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 153.27, 140.66, 131.70, 128.97, 127.99, 105.39, 60.36, 56.44; Calcd for $C_{19}H_{18}N_2O_4$: C, 67.44; H, 5.36; N, 8.28. Found: C, 67.41; H, 5.17; N, 8.27%; MS (ESI) [M + H] found (expected): 339.1448 (339.1345).

4-(4-Nitrophenyl)-6-phenylpyrimidin-2(1H)-one **(4m)**: IR (KBr, ν, cm^{-1}): 3418, 2918, 1681, 1560, 1351, 858, 758; ^1H NMR (400 MHz, DMSO-d_6) δ: 8.49 (d, J = 8.4 Hz, 1H, ArH), 8.39 (d, J = 8.8 Hz, 2H, ArH), 8.20 (d, J = 5.2 Hz, 1H, ArH), 7.99 (d, J = 8.4 Hz, 1H, ArH), 7.59 (d, J = 7.2 Hz, 2H, ArH), 7.29 (d, J = 8.4 Hz, 1H, ArH), 7.17 (s, 1H, ArH), 6.95 (d, J = 9.2 Hz, 1H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ:155.86, 154.02, 143.88, 137.83, 134.53, 131.15, 129.33, 128.80, 128.63, 128.51, 126.76, 126.72, 126.65, 71.11, 50.11; Calcd for $C_{16}H_{11}N_3O_3$: C, 65.53; H, 3.78; N, 14.33. Found: C, 65.85; H, 3.54; N, 14.40%. MS (ESI) [M + H] found (expected): 294.0828(294.0879).

4-(2-Chlorophenyl)-6-phenylpyrimidin-2(1H)-one **(4n)**: IR (KBr, ν, cm^{-1}): 3320, 2849, 1623, 1458, 1349, 772, 690; ^1H NMR (400 MHz, DMSO-d_6) 12.04 (br, s, 1H, NH), 8.12–8.29 (m, 4H, ArH), 7.69–7.76 (m, 6H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 156.97, 154.10, 149.20, 149.08, 146.51, 141.71, 138.68, 131.81, 129.07, 127.77, 123.21, 117.15, 69.19, 53.86, 49.93; Calcd for $C_{16}H_{11}ClN_2O$: C, 67.97; H, 3.92; N, 9.91; Found: C, 68.20; H, 4.28; N, 9.60%; MS (ESI) [M + H] found (expected): 283.0531 (283.0639).

Single crystal X-ray diffraction

The single crystal growth was carried out in EtOH at room temperature. Crystal data for **4a**: empirical formula: $C_{16}H_{12}N_2O$, colourless, crystal dimension: 0.32 × 0.19 × 0.16 mm, monoclinic, space group: P2(1)/c, a = 13.2000(2) Å, b = 7.3243(14) Å, c = 13.774(2) Å, α = 90°, β = 108.162°, γ = 90°, V = 1265.3(4) A^{-3}, Mr = 248.28, Z = 4, Dc = 1.303 Mg/m^{-3}, λ = 0.71073 Å, μ(Mo Kα) = 0.083 mm^{-1}, F(000) = 520, S = 1.012, R^1 = 0.0459, wR^2 = 0.0921, the number of reflections collected and the number of unique reflections is 6310 and 2234 respectively. Crystallographic data for the structure of **4a** reported have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC-649143.

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