Synthesis of *trans*-6-(4-chlorobenzoyl)-7-(aryl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-diones using choline hydroxide as an efficient catalyst in an aqueous medium

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A green and efficient synthesis of the *trans*-6-(4-chlorobenzoyl)-7-(aryl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-diones has been achieved via a three-component, one-pot condensation of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide with 1,3-dimethylbarbituric acid and an aromatic aldehyde in the presence of catalytic amounts of choline hydroxide in water under reflux conditions. This gives *trans*-6-(4-chlorobenzoyl)-7-(aryl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-diones in excellent yield and in short reaction times.

Keywords: choline hydroxide, green chemistry, dihydrofuro[3,2-d]pyrimidine-2,4-dione, aryl aldehydes, 4-chlorophenacyl bromide, 1,3-dimethylbarbituric acid

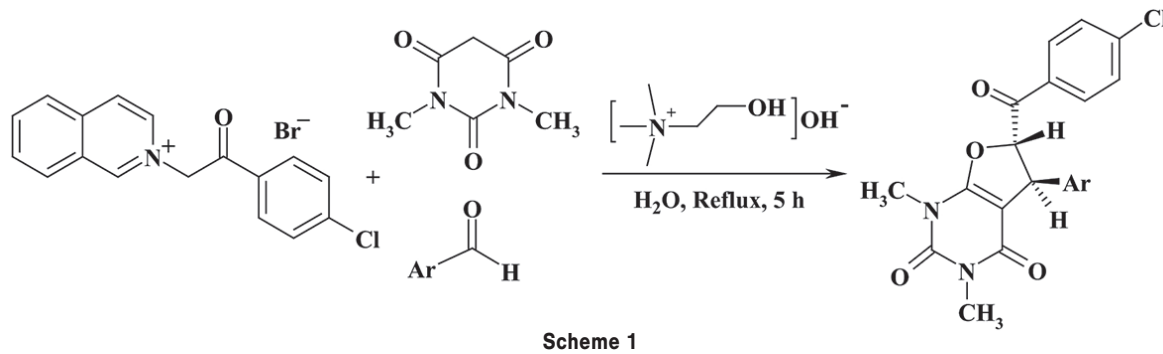
Among the heterocycles, the furo[2,3-*d*]pyrimidine derivatives are an important class of heterocyclic compounds in pharmaceutical research. Anti-fungal,[1] anti-folate,[2–4] anti-bacterial,[5] anti-tumor,[3,6] anti-viral[7,8] and anti-HCMV (human cytomegalovirus)[9] activities have been reported for these compounds. Recently, some furopyrimidines were shown to be potent vascular endothelial growth factor receptor-2 (VEGFR2) and epidermal growth factor receptor (EGFR) inhibitors.[10] A number of procedures for the synthesis of furo[2,3-*d*]pyrimidines have already been reported.[11,14] The methods reported previously for the synthesis of α-furo[2,3-*d*] pyrimidines suffer from severe disadvantages, such as hazardous solvents, longer reaction times, inadequate yields and use of an expensive, non-recoverable surfactant. Based on the above information and due to our interest in developing synthetic strategies for the construction of heterocyclic compounds, we have now used choline hydroxide in a new and rapid method affording excellent yields and as a green catalyst for the synthesis of furo[2,3-*d*]pyrimidines under reflux conditions.

Recently, organic reactions in aqueous media have attracted a great deal of attention[15] as a result of increasing interest in the concepts of sustainability and green chemistry.[16] In continuation of our previous work on the synthesis of heterocyclic compounds,[17,18] we decided to investigate the reaction of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** with 1,3-dimethylbarbituric acid **3** and an aromatic aldehyde **4** in the presence of catalytic amounts of choline hydroxide in water under reflux conditions (Scheme 1).

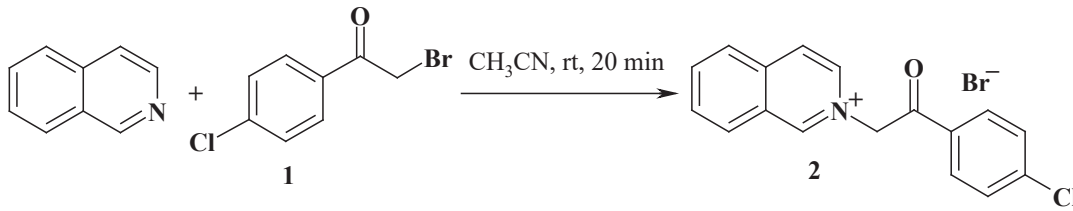
Results and discussion

Treatment of isoquinoline with 4-chlorophenacyl bromide **1** in acetonitrile after 20 min yielded the 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** in nearly quantitative yields (Scheme 2).

The one-pot, three-component reaction of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** with 1,3-dimethylbarbituric acid **3** and an aromatic aldehyde **4** in the presence of catalytic amounts of choline hydroxide in



Scheme 1



Scheme 2

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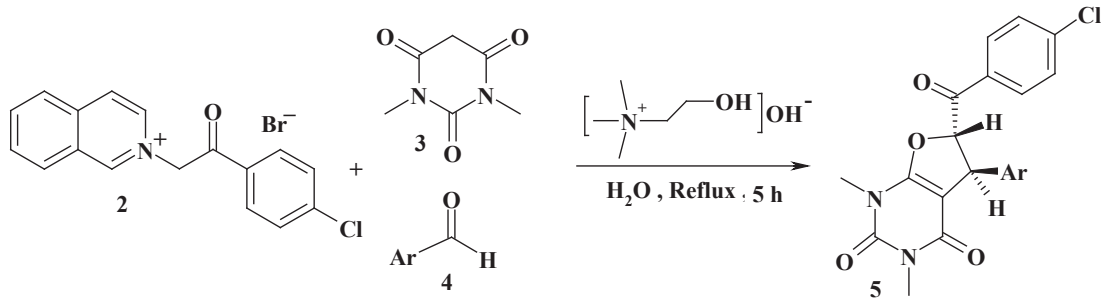
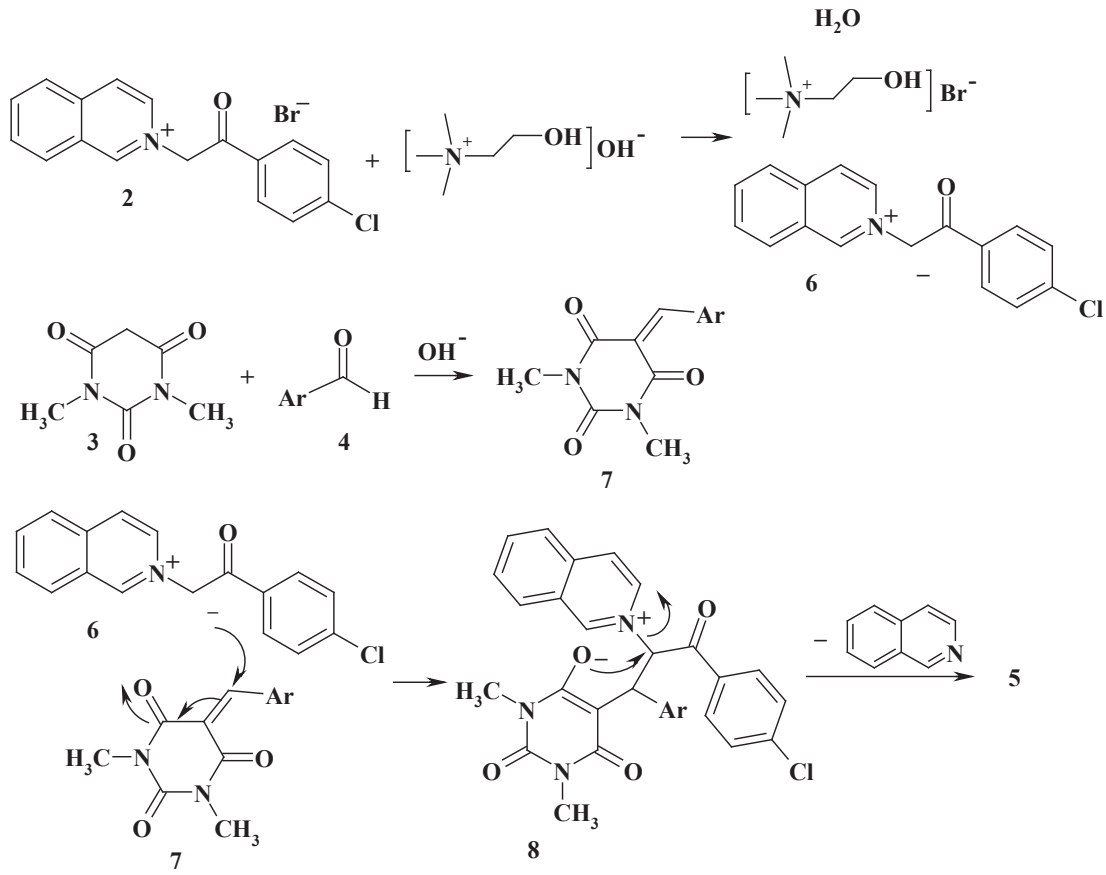


5	Ar	%Yield *	m.p.(°C)
a	C_6H_5	92	138-140
b	2-O_2N C_6H_4	95	141-143
c	4-O_2N C_6H_4	96	147-149
d	2-Cl C_6H_4	89	156-158
e	4-ClC_6H_4	92	158-160
f	2-FC_6H_4	93	137-139
g	4-CH_3O C_6H_4	89	143-145
h	4-F C_6H_4	96	160-162
i	4-Cl-3-O_2N C_6H_3	90	154-156
j	4-NC C_6H_4	93	145-147
k	4-CH_3 C_6H_4	91	161-163
l	4-Br C_6H_4	94	150-152

*Yields refer to the pure isolated prodoucts

Scheme 3



Scheme 4

refluxing water gave the corresponding products **5** in excellent yields (Scheme 3).

The structures of compounds **5a–l** were deduced from their elemental analyses and their IR, ^{1}H NMR and ^{13}C NMR spectra. The mass spectra of compounds **5a–l** are fairly similar and display molecular ion peaks. In the ^{1}H NMR spectra, the two protons at the 2,3-position of the dihydrofuran ring appeared as two doublets at 4.36 and 6.08 ppm with values of the vicinal

coupling constant J = 5.2 and 5.2 Hz, respectively. It has been reported that in *cis*-2,3-dihydrofuran the vicinal coupling constant of the two methine protons has a value in the range J = 7–10 Hz, while in *trans*-2,3-dihydrofuran J = 2.8–6 Hz. Hence, we concluded that the thermodynamically stable *trans* isomers of the 2,3-dihydrofuran derivatives were formed.[19]

A proposed mechanism for this reaction is shown in Scheme 4. The formation of the product can be explained as follows. The 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** undergoes deprotonation in the presence of aqueous choline hydroxide to give the reactive isoquinolinium ylid **6** at room temperature. The 1,3-dimethylbarbituric acid **3** reacts with aromatic aldehyde **4** in the presence of choline hydroxide to give the Knoevenagel product **7**. This reacts instantly with the isoquinolinium ylid **6** to form the zwitterionic intermediate **8**. The intermediate **8** undergoes cyclisation with the elimination of isoquinoline to give the desired product **5**.

In summary, we report a simple and efficient, one-pot condensation of 2-[2-(4-chlorophenyl)-2-oxoethyl)] isoquinolinium bromide with 1,3-dimethylbarbituric acid and an aromatic aldehyde in the presence of catalytic amounts of choline hydroxide in water at reflux conditions. This gives *trans*-6-(4-chlorobenzoyl)-7-(aryl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-diones. The advantages of this method are: readily available starting materials, short reaction times, easy and clean work-up and excellent yields.

Experimental

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed using a Heraeus CHN-O-Rapid Analyzer. Mass spectra were recorded on a Finnigan-MAT 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX-400 Avance spectrometer as solutions in $CDCl_3$ using tetramethylsilane (TMS) as internal standard. The 2-[2-(4-chlorophenyl)-2-oxoethyl)] isoquinolinium bromide **2** was prepared by the literature method.[20] Other chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General procedure

A magnetically stirred solution of 2-[2-(4-chlorophenyl)-2-oxoethyl)] isoquinolinium bromide (1 mmol), 1,3-dimethylbarbituric acid (1 mmol) and aryl aldehyde (1 mmol) in H_2O (10 mL) was treated with choline hydroxide (0.0121 g, 0.1 mmol) in H_2O (4 mL). The mixture was then refluxed for 5 h. The solid product was filtered and then recrystallised from ethanol to afford the pure product.

*trans-6-(4-Chlorobenzoyl)-7-phenyl-1,3-dimethyl-6,7-dihydrofuro [3,2-*d*]pyrimidine-2,4-dione* **(5a)**: Brown powder; m.p. 138–140 °C; IR (KBr) (ν_{max} cm^{-1}): 3454, 3300, 3189, 2904, 1662, 1560, 1468, 1419, 1369, 1216, 1025, 818, 789, 667, 604, 503; Anal. calcd for $C_{21}H_{17}ClN_2O_4$: C, 63.56; H, 4.32; N, 7.06; found: C, 63.72; H, 4.45; N, 7.07%; MS (*m/z*, %): 396 (7); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.12 (s, 3H, CH_3), 3.18 (s, 3H, CH_3) 4.36 (d, J = 5.2 Hz, 1H, CH), 6.08 (d, J 5.2 Hz, 1H CH), 7.29–8.05 (m, 9H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$: δ 29.6, 29.8, 41.9, 82.1, 121.6, 122.8, 125.9, 127.7, 128.6, 128.7, 130.2, 132.3, 138.7, 140.6, 151.4, 162.9, 188.6 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(2-nitrophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5b)**: Brown powder; m.p. 141–143 °C; IR (KBr) (ν_{max} cm^{-1}): 3425, 3380, 3035, 2900, 2220, 1660, 1570, 1513, 1421, 1370, 847, 823, 778, 742, 667, 576; Anal. calcd for $C_{21}H_{16}ClN_3O_6$: C, 57.09; H, 3.65; N, 9.51; found: C, 57.22; H, 3.76; N, 9.40%; MS (*m/z*, %): 441 (3); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.15 (s, 3H, CH_3), 3.19 (s, 3H, CH_3), 4.56 (d, J = 5.1 Hz, CH, 1H,), 6.12 (d, J = 5.1Hz CH, 1H), 7.61–8.07 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.6, 29.7, 37.3, 92.7, 81.1, 121.6, 123.1, 124.8, 126.8, 128.6, 128.9, 130.4, 132.3, 133.9, 137.7, 148.3, 151.4, 162.9, 188.5 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-nitrophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5c)**: Brown powder; m.p. 147–149 °C; IR (KBr) (ν_{max} cm^{-1}): 3395, 3195, 2905, 2630, 1662, 1572, 1504, 1418, 1331, 1267, 1124, 846, 817, 744, 664, 604; Anal. calcd for $C_{21}H_{16}ClN_3O_6$: C, 57.09; H, 3.65; N, 9.51; found: C, 57.20; H, 3.79; N, 9.43%; MS (*m/z*, %): 441 (5); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.11 (s, 3H, CH_3), 3.16 (s, 3H, CH_3) 4.36 (d, J = 4.8 Hz, CH, 1H), 6.08 (d, J = 4.8 Hz, CH, 1H), 7.58–8.42 (m, 8H, arom) ppm;^{13}C NMR (100.6 MHz, $CDCl_3$): δ 28.8, 29.1, 41.6, 84.3, 122.3, 123.3, 124.1, 128.1, 128.8, 130.4, 132.5, 138.5, 146.2, 146.7, 151.3, 163.3, 188.4 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(2-chlorophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5d)**: Brown powder; m.p. 156–158 °C; IR (KBr) (ν_{max} cm^{-1}): 3445, 3270, 3140, 2900, 1662, 1570, 1420, 1371, 1117, 1013, 817, 755, 667, 604, 574; Anal. calcd for $C_{21}H_{16}Cl_2N_2O_4$: C, 58.49; H, 3.74; N, 6.50; found: C, 58.63; H, 3.85; N, 6.38%; MS (*m/z*, %): 430 (8); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.10 (s, 3H, CH_3), 3.15 (s, 3H, CH_3), 4.48 (d, J = 5.2 Hz, CH, 1H), 5.98 (d, J = 5.2 Hz, CH, 1H), 6.05–8.06 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.6, 29.7, 36.8, 81.6, 108.8, 119.4, 120.9, 126.8, 127.3, 128.7, 129.1, 130.2, 132.3, 132.7, 133.0, 138.4, 151.4, 162.9, 188.1 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-chlorophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5e)**: Brown powder; m.p. 158–160 °C; IR (KBr) (ν_{max} cm^{-1}): 3425, 3280, 3140, 2900, 1659, 1570, 1420, 1371, 1115, 1016, 817, 667, 604, 516; Anal. calcd for $C_{21}H_{16}Cl_2N_2O_4$: C, 58.49; H, 3.74; N, 6.50; found: C, 58.65; H, 3.82; N, 6.35%; MS (*m/z*, %): 430 (10); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.22 (s, 3H, CH_3), 3.26 (s, 3H, CH_3), 3.96 (d, J = 5.2 Hz, CH, 1H), 6.46 (d, J = 5.2 Hz, CH, 1H), 7.30–8.02 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.7, 29.9, 48.3, 91.2, 108.7, 121.1, 124.0, 128.6, 129.1, 130.2, 131.5, 132.2, 138.3, 138.9, 151.7, 163.1, 188.4 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(2-fluorophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5f)**: Brown powder; m.p. 137–139 °C; IR (KBr) (ν_{max} cm^{-1}): 3447, 3280, 3040, 2905, 1666, 1570, 1513, 1421, 1250, 121, 823, 755, 604; Anal. calcd for $C_{21}H_{16}ClFN_2O_4$: C, 60.81; H, 3.89; N, 6.75; found: C, 60.93; H, 4.04; N, 6.63%; MS (*m/z*, %): 414 (4); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.11 (s, 3H, CH_3), 3.16 (s, 3H, CH_3), 4.08 (d, J = 5.2 Hz, CH, 1H), 6.13 (d, J = 5.2 Hz, CH, 1H), 7.07–8.10 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.5, 29.7, 35.2, 83.1, 115.4, 121.1, 122.9, 124.2, 127.3, 127.5, 128.7, 129.3, 130.2, 132.3, 138.7, 151.4, 160.2, 162.9, 188.2 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-methoxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5g)**: Brown powder; m.p. 143–145 °C; IR (KBr) (ν_{max} cm^{-1}): 3425, 3380, 3380, 3140, 1665, 1572, 1507, 1418, 1254, 1201, 1175; Anal. calcd for $C_{22}H_{19}ClN_2O_5$: C, 61.90; H, 4.49; N, 6.56; found: C, 62.02; H, 4.60; N, 6.70%; MS (*m/z*, %): 426 (8); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.12 (s, 3H, CH_3), 3.19 (s, 3H, CH_3), 3.81(s, 3H, OCH_3), 4.36 (d, J = 4.8 Hz, 1H), 6.09 (d, J = 4.8 Hz, 1H), 6.85–8.10 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.4, 29.6, 41.9, 55.8, 82.1, 114.2, 114.8, 121.3, 122.9, 128.7, 130.2, 132.3, 132.9, 138.7, 151.6, 157.8, 162.9, 188.4 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-fluorophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5h)**: Brown powder; m.p. 160–162 °C; IR (KBr) (ν_{max} cm^{-1}): 3435, 3297, 3135, 2904, 1664, 1565, 1425, 1272, 1017, 816, 667, 534; Anal. calcd for $C_{21}H_{16}ClFN_2O_4$: C, 60.81; H, 3.89; N, 6.75; found: C, 60.95; H, 4.01; N, 6.65%; MS (*m/z*, %): 414 (6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.10 (s, 3H, CH_3), 3.15 (s, 3H, CH_3), 4.24 (d, J = 5.0 Hz, CH, 1H), 6.10 (d, J = 5.0 Hz, CH, 1H,), 7.16–8.06 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.6, 29.7, 41.9, 82.1, 115.4, 121.2, 122.7, 128.7, 129.3, 130.2, 132.3, 136.4, 138.7, 151.4, 160.2, 162.7, 188.2 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-chloro-3-nitrophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5i)**: Brown powder; m.p. 154–156 °C; IR (KBr) (ν_{max} cm^{-1}): 3475, 3310, 3130, 2910, 1665, 1571, 1523, 1445, 1422, 819, 771, 745, 665, 604; Anal. calcd for $C_{21}H_{15}Cl_2N_3O_6$: C, 52.96; H, 3.17; N, 8.82; found: C, 53.11; H, 3.32; N, 8.70%; MS (*m/z*, %): 475 (5); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.16 (s, 3H, CH_3), 3.20 (s, 3H, CH_3), 4.40 (d, J = 4.8 Hz, CH, 1H,),

6.12 (d, *J* = 4.8 Hz CH, 1H), 7.59–8.32 (m, 7H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.1, 29.7, 40.7, 92.1, 122.3, 124.5, 125.5, 125.8, 128.6, 130.4, 132.1, 132.8, 135.5, 134.9, 138.4, 147.4, 151.3, 163.3, 188.8 ppm.

*trans-6-(4-Chlorobenzoyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4,6,7-hexa-hydrofuro[3,2-*d*]pyrimidin-7-yl)benzonitrile* **(5j)**: Brown powder; m.p. 145–147 °C; IR (KBr) (ν_{max} cm^{-1}): 3452, 3321, 3185, 2906, 1660, 1554, 1418, 1369, 1215, 817, 768, 565; Anal. calcd for $C_{22}H_{16}ClN_3O_4$: C, 62.64; H, 3.82; N, 9.96; found: C, 62.80; H, 3.70; N, 9.84%; MS (*m/z*, %): 421(7); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.11 (s, 3H, CH_3), 3.16 (s, 3H, CH_3), 4.35 (d, *J* = 4.8 Hz, CH, 1H), 6.08 (d, *J* = 4.8 Hz, CH, 1H), 7.60–8.07 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.4, 29.6, 41.9, 82.1, 109.8, 118.6, 122.1, 122.9, 127.5, 128.4, 130.2, 132.1, 132.3, 138.7, 144.9, 151.4, 162.9, 188.3 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-methylphenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5k)**: Brown powder; m.p. 161–163 °C; IR (KBr) (ν_{max} cm^{-1}): 3460, 3315, 3185, 2902, 1665, 1562, 1468, 1419, 1367, 1214, 757; Anal. calcd for $C_{22}H_{19}ClN_2O_4$: C, 64.32; H, 4.66; N, 6.82; found: C, 64.48; H, 4.50; N, 6.74%; MS (*m/z*, %): 410 (4); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.18 (s, 3H, CH_3), 3.12 (s, 3H, CH_3), 3.20 (s, 3H, CH_3), 4.44 (d, *J* = 5.4 Hz, CH, 1H), 6.05 (d, *J* = 5.4 Hz, CH, 1H), 7.02–8.05 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 21.4, 29.5, 29.7, 41.9, 82.1, 109.6, 121.7, 123.1, 127.6, 128.9, 130.2, 132.3, 135.6, 137.6, 138.7, 151.4, 162.9, 188.2 ppm.

*trans-6-(4-Chlorobenzoyl)-7-(4-bromophenyl)-1,3-dimethyl-6,7-dihydrofuro[3,2-*d*]pyrimidine-2,4-dione* **(5l)**: Brown powder; m.p. 150–152 °C; IR (KBr) (ν_{max} cm^{-1}): 3450, 3300, 3190, 2905, 1662, 1560, 1468, 1419, 1369, 1216, 818, 789, 667, 603, 505; Anal. calcd for $C_{21}H_{16}BrClN_2O_4$: C, 53.02; H, 3.39; N, 5.89; found: C, 53.15; H, 3.23; N, 5.77%; MS (*m/z*, %): 475 (10); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.13 (s, 3H, CH_3), 3.18 (s, 3H, CH_3), 4.22 (*J* = 5.2 Hz, CH, 1H), 6.32 (*J* = 5.2 Hz, CH, 1H), 7.32–8.02 (m, 8H, arom) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 29.6, 29.8, 42.1, 82.1, 121.3, 125.3, 126.6, 128.8, 129.8, 130.3, 131.4, 132.3, 139.0, 140.8, 151.4, 162.6, 188.7 ppm.

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