Supporting Information

Eco-friendly diastereoselective synthesis of dihydrofuropyrido[2,3-d]pyrimidines via pyridinium ylides in water
Somayeh Ahadi, a Telma Kamranifard, a Mahsa Armaghan, b Hamid Reza Khavasi a and Ayoob Bazgir * a

a Department of Chemistry, Shahid Beheshti University G. C., Tehran 1983963113, Iran.
b Institute of Materials Research and Engineering, Agency for Science Technology and Research, 3 Research Link, S117602, Singapore
Email:a_bazgir@sbu.ac.ir

Table of Contents:

General methods S1
Synthesis of starting materials S2
General procedure for the synthesis of 4 and 9 S2-S12
Copies of 1H NMR and 13C NMR spectra S13-S48

General methods

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected IR spectra were recorded using a BOMEM MB-Series. 1H and 13C NMR spectra was recorded on a BRUKER DRX-300 AVANCE spectrometer. Chemical shifts are expressed in parts per million downfield from tetramethylsilane as an internal standard. Elemental analyses for C, H and N performed using a Heraeus CHN–O–Rapid analyzer. MS spectra were recorded on a Shimadzu QP 1100EX mass spectrometer operating at an ionization potential of 70 eV.

X-ray crystallography: The X-ray diffraction measurements were made on a STOE IPDS-II diffractometer with graphite monochromated Mo-Kα radiation. Cell constants and an orientation matrix for data collection were obtained by least-squares refinement of diffraction data from 4713 unique reflections for 4c. Data were collected at a temperature of 298(2) K to a maximum 20 value of 54.00° and in a series of ω scans in 1° oscillations and integrated using the Stoe X-AREA1 software package. The data were corrected for Lorentz and Polarizing effects. The structures were solved by direct methods and refined on F2 by full-matrix least-squares procedure. All hydrogen atoms were added at ideal positions and constrained to ride on their parent atoms, with Uiso(H) = 1.2Ueq. All refinements were performed using the X-STEP32 crystallographic software package.2 Complete crystallographic data for compound 4c has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 955501. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. Fax: 0044 1223 336 033. Email: deposit@ccdc.cam.ac.uk


Synthesis of starting material 1:
A mixture of 6-amino-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (1 mmol) and diethyl malonate (4 mmol) was heated at 220 °C for 3 h. Then, the precipitate was washed with ethanol to produce pure product 1.

Synthesis of starting material 3:
A mixture of phenacyl bromide (1 mmol), pyridin (1.2 mmol) in MeCN (2 mL) in the presence of K₂CO₃ (30 mol%) was refluxing for 2 h. Then the precipitated product was filtered and washed with MeCN to produce pure product.

General procedure for the synthesis of 4
A mixture of 5-hydroxy-1,3-dimethylpyrido[2,3-d]pyrimidine-2,4,7(1H,3H,8H)-trione (1.0 mmol), aromatic aldehydes (1.0 mmol) and 1-(2-oxo-2-phenylethyl)pyridin-1-ium (1 mmol) in H₂O (2 mL) in the presence of NEt₃
(30 mol%) was refluxing for 48 h. After completion of the reaction (TLC), the reaction mixture was cooled to room temperature. Then, the precipitated product was filtered and washed with ethanol (5 ml) to afford the pure product 4.

7-benzoyl-5-hydroxy-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4a)

Brown powder (yield 97%); mp 198-205 °C. IR (KBr) (νmax /cm⁻¹): 3379, 3051, 1678, 1600. ¹H NMR (300 MHz, DMSO-d₆): δH (ppm) 3.30 (3H, s, CH₃), 3.56 (3H, s, CH₃), 5.04 (1H, d, J= 4.5 Hz, CH), 5.99 (1H, d, J= 4.62 Hz, CH), 7.39-7.60 (5H, m, H-Ar), 7.85 (2H, d, J= 7.61 Hz, H-Ar), 8.14 (2H, d, J= 8.13 Hz, H-Ar), 12.40 (1H, s, OH). ¹³C NMR (75 MHz, DMSO-d₆): δC (ppm) 28.2, 30.5, 46.5, 88.6, 93.8, 100.0, 119.1, 122.2, 125.0, 129.7, 130.0, 135.1, 136.2, 155.1, 155.7, 164.8, 172.2, 194.7. Anal. Calcd for C₂₄H₁₈N₄O₇: C, 60.76; H, 3.82; N, 11.81. Found: C, 60.69; H, 3.77; N, 11.87.

7-benzoyl-5-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4,6,7-hexahydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidin-6-yl)benzoic acid (4b)

White powder (yield 60%); mp 227-233 °C. IR (KBr) (νmax /cm⁻¹): 3313, 3075, 3099, 1714, 1666, 1621. ¹H NMR (300 MHz, DMSO-d₆): δH (ppm) 3.22 (3H, s, CH₃), 3.49 (3H, s, CH₃), 4.92 (1H, bs, CH), 6.47 (1H, bs, CH), 7.33-7.79 (5H, m, H-Ar), 7.89-7.94 (4H, m, H-Ar), 12.64 (2H,
bs, 2OH). $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta$C (ppm) 28.2, 30.6, 46.5, 88.7, 93.8, 100.9, 128.6, 129.8, 129.9, 130.7, 131.0, 134.3, 145.7, 151.0, 154.0, 165.1, 166.0, 167.8, 171.8, 194.3. MS (EI, 70 eV) m/z: 473 (M$^+$. Anal. Calcd for C$_{25}$H$_{19}$N$_3$O$_7$: C, 63.42; H, 4.05; N, 8.88. Found: C, 63.37; H, 4.09; N, 8.80.

7-benzoyl-5-hydroxy-6-(4-methoxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4c)

Cream powder (yield 84%); mp 194-204 °C. IR (KBr) ($\nu_{\text{max}}$ /cm$^{-1}$): 3598, 1701, 1633. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta$H (ppm) 3.22 (3H, s, CH$_3$), 3.48 (3H, s, CH$_3$), 3.73 (3H, s, OCH$_3$), 4.70 (1H, bs, CH), 6.36 (1H, bs, CH), 6.91 (2H, bs, H-Ar), 7.10 (2H, bs, H-Ar), 7.54 (2H, bs, H-Ar), 7.70 (1H, bs, H-Ar), 7.87 (2H, bs, H-Ar), 12.40 (1H, s, OH). $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta$C (ppm) 28.2, 30.5, 46.3, 56.0, 89.4, 93.7, 101.5, 115.1, 129.3, 129.8, 132.9, 134.2, 135.1, 151.0, 153.8, 159.5, 165.0, 166.0, 171.8, 194.5. MS (EI, 70 eV) m/z: 459 (M$^+$). Anal. Calcd for C$_{25}$H$_{21}$N$_3$O$_6$: C, 65.35; H, 4.61; N, 9.15. Found: C, 65.73; H, 4.56; N, 9.21.

Crystal data for 4c C$_{25}$H$_{21}$N$_3$O$_6$ (CCDC 955501): M= 459.45 g/mol, monoclinic system, space group P21/a, a = 9.6442(12) Å, b = 14.0350(13) Å, c = 15.9534(18) Å, $\beta$ = 91.025(9)$^\circ$, V = 2159.0(4) Å$^3$, Z = 4, Dc = 1.413 g.cm$^{-3}$, $\mu$(Mo-K$\alpha$) = 0.103 mm$^{-1}$, crystal dimension of 0.25 x 0.20 x 0.15 mm. The structure was solved by using SHELXS. The structure refinement and data reduction was carried out with SHELXL of the X-Step32 suite of programs. The non-hydrogen atoms were refined anisotropically by full matrix least-squares on $F^2$ values to final $R_f$ = 0.0641, $wR_f$ = 0.1675 and S=0.958 with 313 parameters using 4713 independent reflection (0 range = 1.93 – 27.00$^\circ$). Hydrogen atoms were located from expected geometry and were not refined.
7-benzoyl-5-hydroxy-1,3-dimethyl-6-(3-nitrophenyl)-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-
d]pyrimidine-2,4(1H,3H)-dione (4d)

Cream powder (yield 81%); mp 187-194 °C. IR (KBr) (ν<sub>max</sub> /cm<sup>-1</sup>): 3452, 1708, 1624. <sup>1</sup>H NMR (300 MHz, DMSO-<sup>d6</sup>): δ<sub>H</sub> (ppm) 3.22 (3H, s, CH<sub>3</sub>), 3.48 (3H, s, CH<sub>3</sub>), 5.14 (1H, bs, CH), 6.53 (1H, bs, CH), 7.54-7.70 (5H, m, H-Ar), 7.91 (2H, d, J = 7.08 Hz, H-Ar), 8.08 (1H, bs, H-Ar), 8.18 (1H, d, J = 6.23 Hz, H-Ar), 12.47 (1H, s, OH). 1<sup>3</sup>C NMR (75 MHz, DMSO-<sup>d6</sup>): δ<sub>C</sub> (ppm) 28.2, 30.6, 45.9, 88.3, 93.7, 100.6, 123.2, 123.4, 129.7, 130.0, 131.2, 131.2, 134.5, 135.1, 142.9, 165.1, 171.7, 194.1. MS (EI, 70 eV) m/z: 474 (M<sup>+</sup>). Anal. Calcd for C<sub>24</sub>H<sub>18</sub>N<sub>4</sub>O<sub>7</sub>: C, 60.76; H, 3.82; N, 11.81. Found: C, 60.70; H, 3.78; N, 11.76.

-7-benzoyl-5-hydroxy-1,3-dimethyl-6-(thiophen-3-yl)-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-
d]pyrimidine-2,4(1H,3H)-dione (4e)

Light cream powder (yield 100%); mp 185 °C (dec). IR (KBr) (ν<sub>max</sub> /cm<sup>-1</sup>): 3635, 1705, 1671, 1621. <sup>1</sup>H NMR (300 MHz, DMSO-<sup>d6</sup>): δ<sub>H</sub> (ppm) 3.23 (3H, s, CH<sub>3</sub>), 3.48 (3H, s, CH<sub>3</sub>), 5.14 (1H, bs, CH), 5.14 (1H, bs, CH), 6.44 (1H, bs, CH), 7.00 (2H, bs, H-Ar), 7.47-7.73 (4H, m, H-Ar), 7.96 (2H, d, J = 4.67 Hz, H-Ar), 12.56 (1H, s, OH). 1<sup>3</sup>C NMR (75 MHz, DMSO-<sup>d6</sup>): δ<sub>C</sub> (ppm) 28.2, 30.6, 41.9, 89.1, 93.7, 101.2, 126.4, 128.1, 129.9, 134.1, 135.2, 143.9, 151.0, 154.3, 163.2, 165.3, 166.0, 171.5, 194.1. MS (EI, 70 eV) m/z: 435 (M<sup>+</sup>). Anal. Calcd for C<sub>22</sub>H<sub>17</sub>N<sub>3</sub>O<sub>5</sub>S: C, 60.68; H, 3.94; N, 9.65. Found: C, 60.78; H, 3.87; N, 9.73.
5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-phenyl-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4f)

Cream powder (yield 59%); mp 211 °C. IR (KBr) (ν_max/cm⁻¹): 3531, 1709, 1671, 1678, 1615. 

$^1$H NMR (300 MHz, DMSO-d₆): δ_H (ppm) 3.22 (3H, s, CH₃), 3.49 (3H, s, CH₃), 3.85 (3H, s, OCH₃), 4.75 (1H, bs, CH), 6.37 (1H, bs, CH), 7.06-7.34 (7H, m, H-Ar), 7.86 (2H, bs, H-Ar), 12.35 (1H, bs, OH). MS (EI, 70 eV) m/z: 459 (M⁺). Anal. Calcd for C₂₅H₂₁N₃O₆: C, 65.35; H, 4.61; N, 9.15. Found: C, 65.26; H, 4.67; N, 9.06.

5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4g)

Yellow powder (yield 60%); mp 195-200 °C. IR (KBr) (ν_max/cm⁻¹): 3602, 1704, 1672, 1625. 

$^1$H NMR (300 MHz, DMSO-d₆): δ_H (ppm) 3.22 (3H, s, CH₃), 3.48 (3H, s, CH₃), 3.85 (3H, s, OCH₃), 5.04 (1H, bs, CH), 6.46 (1H, bs, CH), 7.08 (2H, d, J= 8.21 Hz, H-Ar), 7.53 (2H, d, J= 7.76 Hz, H-Ar), 7.89 (2H, d, J= 7.92 Hz, H-Ar), 8.23 (2H, d, J= 7.60 Hz, H-Ar), 12.44 (1H, s, OH). 

$^{13}$C NMR (75 MHz, DMSO-d₆): δ_C (ppm) 28.2, 30.6, 46.3, 56.6, 88.1, 93.8, 100.7, 115.2, 124.8, 127.0, 129.9, 132.4, 147.8, 148.4, 151.0, 154.1, 165.0, 166.0, 192.2. MS (EI, 70 eV) m/z: 504 (M⁺). Anal. Calcd for C₂₅H₂₆N₄O₈: C, 59.52; H, 4.00; N, 11.11. Found: C, 59.61; H, 4.07; N, 11.07.
5-hydroxy-7-(4-methoxybenzoyl)-6-(4-methoxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4h)

Yellow powder (yield 94%); mp 163-170 °C. IR (KBr) (ν max/cm⁻¹): 3501, 1704, 1624. ¹H NMR (300 MHz, DMSO-d₆): δ_H (ppm) 3.22 (3H, s, CH₃), 3.33 (3H, s, CH₃), 3.49 (3H, s, OCH₃), 3.85 (3H, s, OCH₃), 4.68 (1H, bs, CH), 6.32 (1H, bs, CH), 6.91 (2H, d, J= 8.02 Hz, H-Ar), 7.06-7.15 (4H, m, H-Ar), 7.86 (2H, d, J= 8.47 Hz, H-Ar), 12.40 (1H, s, OH). MS (EI, 70 eV) m/z: 489 (M⁺). Anal. Calcd for C₂₆H₂₃N₃O₇: C, 63.80; H, 4.74; N, 8.58. Found: C, 63.74; H, 4.70; N, 8.52.

7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-phenyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4i)

Cream powder (yield 60%); mp 202-210 °C. IR (KBr) (ν max/cm⁻¹): 3075, 1706, 1670, 1628. ¹H NMR (300 MHz, DMSO-d₆): δ_H (ppm) 3.22 (3H, s, CH₃), 3.48 (3H, s, CH₃), 4.82 (1H, d, J= 2.76 Hz, CH), 6.41 (1H, d, J= 2.97 Hz, CH), 7.21 (2H, d, J= 6.75 Hz, H-Ar), 7.30-7.36 (3H, m, H-Ar), 7.76-7.83 (4H, d, m, H-Ar), 12.42 (1H, s, OH). ¹³C NMR (75 MHz, DMSO-d₆): δ_C (ppm) 28.2, 30.6, 46.5, 89.1, 93.8, 101.3, 128.2, 128.4, 129.3, 129.7, 131.8, 132.8, 133.4, 140.9, 151.0,
153.9, 165.1, 166.1, 171.8, 193.8. MS (EI, 70 eV) m/z: 509 (M$^+$, $^{81}$Br), 507 (M$^+$, $^{79}$Br). Anal. Calcd for C$_{24}$H$_{18}$BrN$_3$O$_5$: C, 56.71; H, 3.57; N, 8.27. Found: C, 56.63; H, 3.63; N, 8.34.

7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(4-nitrophenyl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4j)

Cream powder (yield 80%); mp 259 ºC (dec). IR (KBr) ($\nu_{\text{max}}$ / cm$^{-1}$): 3383, 1699, 1666, 1618. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ (ppm) 3.22 (3H, s, CH$_3$), 3.48 (3H, s, CH$_3$), 5.11 (1H, d, J = 3.12 Hz, CH), 6.49 (1H, d, J = 3.21 Hz, CH), 7.53 (2H, d, J = 8.13 Hz, H-Ar), 7.77-7.85 (4H, m, H-Ar). $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta_C$ (ppm) 28.2, 30.6, 45.9, 88.3, 93.8, 100.6, 124.8, 129.4, 129.9, 131.9, 132.9, 133.4, 147.8, 148.3, 151.0, 154.1, 165.2, 166.0, 171.6, 193.3. MS (EI, 70 eV) m/z: 554 (M$^+$, $^{81}$Br), 552 (M$^+$, $^{79}$Br). Anal. Calcd for C$_{24}$H$_{17}$BrN$_4$O$_7$: C, 52.10; H, 3.10; N, 10.13. Found: C, 52.05; H, 3.06; N, 10.05.

7-(4-bromobenzoyl)-5-hydroxy-6-(4-hydroxyphenyl)-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (4k)

Cream powder (yield 63%); mp 260 ºC (dec). IR (KBr) ($\nu_{\text{max}}$ / cm$^{-1}$): 3343, 1705, 1657, 1615. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ (ppm) 3.21 (3H, s, CH$_3$), 3.47 (3H, s, CH$_3$), 4.64 (1H, bs, CH), 6.31 (1H, bs, CH), 6.70 (2H, bs, H-Ar), 6.97 (2H, bs, H-Ar), 7.78 (4H, bs, H-Ar), 9.44 (1H, s, OH), 12.36 (1H, bs, OH). $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta_C$ (ppm) 28.2, 30.5, 46.2, 89.5, 93.8.
101.6, 116.5, 129.2, 131.2, 131.7, 132.8, 133.3, 151.1, 153.8, 157.7, 165.0, 166.1, 171.8, 194.0. MS (EI, 70 eV) m/z: 525 (M$^+$, $^{81}$Br), 523 (M$^+$, $^{79}$Br). Anal. Calcd for C$_{24}$H$_{18}$BrN$_3$O$_6$: C, 54.98; H, 3.46; N, 8.01. Found: C, 54.91; H, 3.41; N, 7.94.

*Due to very low solubility of the products 9, we can not report the $^{13}$C NMR date for these products.

7-benzoyl-5-hydroxy-1,3-dimethyl-6-(4-oxo-4H-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (9a)

Cream powder (yield 70%); mp 260 °C (dec). IR (KBr) ($\nu_{\text{max}}$/cm$^{-1}$): 3065, 1686, 1635. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$(ppm) 3.45 (6H, s, 2CH$_3$), 4.85 (1H, bs, CH), 6.37 (1H, bs, CH), 7.54-7.76 (6H, m, H-Ar), 7.97 (3H, bs, H-Ar), 8.27 (1H, bs, H-Ar), 12.42 (1H, bs, OH). MS (EI, 70 eV) m/z: 497 (M$^+$). Anal. Calcd for C$_{27}$H$_{19}$N$_3$O$_7$: C, 65.19; H, 3.85; N, 8.45. Found: C, 65.09; H, 3.79; N, 8.52.

7-benzoyl-6-(6-chloro-4-oxo-4H-chromen-3-yl)-5-hydroxy-1,3-dimethyl-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (9b)
Cream powder (yield 60%); mp 237-245 °C. IR (KBr) (ν max /cm\(^{-1}\)): 3428, 1708, 1676, 1632. \(^1\)H NMR (300 MHz, DMSO-\(d_6\)): δH (ppm) 3.23 (3H, s, CH\(_3\)), 3.46 (3H, s, CH\(_3\)), 4.87 (1H, bs, CH) 6.38 (1H, bs, CH), 7.22-7.99 (8H, m, H-Ar), 8.35 (1H, bs, H-Ar), 12.35 (1H, bs, OH). MS (EI, 70 eV) m/z: 532 (M\(^+\)). Anal. Calcd for C\(_{28}\)H\(_{21}\)N\(_3\)O\(_7\): C, 60.97; H, 3.41; N, 7.90. Found: C, 60.90; H, 3.48; N, 7.99.

![Chemical Structure 1](image1)

7-benzoyl-5-hydroxy-1,3-dimethyl-6-(6-methyl-4-oxo-4\(H\)-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1\(H,3\)\(H\))-dione (9c)

Cream powder (yield 100%); mp >260 °C. IR (KBr) (ν max /cm\(^{-1}\)): 3467, 1710, 1618. \(^1\)H NMR (300 MHz, DMSO-\(d_6\)): δH (ppm) 2.40 (3H, s, CH\(_3\)), 3.38 (3H, s, CH\(_3\)), 3.47 (3H, s, CH\(_3\)), 4.86 (1H, bs, CH), 6.37 (1H, bs, CH), 7.56-7.78 (6H, m, H-Ar), 8.01 (2H, d, J = 6.09 Hz, H-Ar), 8.26 (1H, bs, H-Ar), 12.39 (1H, bs, OH). MS (EI, 70 eV) m/z: 511 (M\(^+\)). Anal. Calcd for C\(_{28}\)H\(_{21}\)N\(_3\)O\(_7\): C, 65.75; H, 4.14; N, 8.22. Found: C, 65.82; H, 4.19; N, 8.16.

![Chemical Structure 2](image2)

5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6-(4-oxo-4\(H\)-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1\(H,3\)\(H\))-dione (9d)

Light cream powder (yield 85%); mp 243 °C (dec). IR (KBr) (ν max /cm\(^{-1}\)): 3087, 1700, 1670, 1636, 1608. \(^1\)H NMR (300 MHz, DMSO-\(d_6\)): δH (ppm) 3.23 (3H, s, CH\(_3\)), 3.47 (3H, s, CH\(_3\)), 3.85 (3H, s, CH\(_3\)), 4.83 (1H, bs, CH), 6.34 (1H, bs, CH), 7.08 (2H, d, J = 6.78 Hz, H-Ar), 7.49 (1H, bs,
H-Ar), 7.66 (1H, bs, H-Ar), 7.81 (1H, bs, H-Ar), 7.99 (3H, bs, H-Ar), 8.32 (1H, bs, H-Ar), 12.30 (1H, bs, OH). MS (EI, 70 eV) m/z: 527 (M^+). Anal. Calcd for C_{28}H_{21}N_{3}O_{8}: C, 63.76; H, 4.01; N, 7.97. Found: C, 63.69; H, 3.96; N, 7.91.

6-(6-chloro-4-oxo-4H-chromen-3-yl)-5-hydroxy-7-(4-methoxybenzoyl)-1,3-dimethyl-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (9e)

Cream powder (yield 90%); mp 244 °C (dec). IR (KBr) (ν_{max} /cm^{-1}): 3075, 1697, 1674, 1646, 1600. \(^1\)H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 3.27 (3H, s, CH_3), 3.51 (3H, s, CH_3), 3.88 (3H, s, CH_3), 4.93 (1H, d, J= 4.74 Hz, CH), 6.24 (1H, d, J= 4.62 Hz, CH), 7.09 (2H, d, J= 7.45 Hz, H-Ar), 7.68-7.84 (2H, m, H-Ar), 7.97-8.02 (4H, m, H-Ar), 8.25 (1H, s, H-Ar), 12.40 (1H, bs, OH). MS (EI, 70 eV) m/z: 562 (M^+). Anal. Calcd for C_{28}H_{20}ClN_{3}O_{8}: C, 59.85; H, 3.59; N, 7.48. Found: C, 59.94; H, 3.52; N, 7.53.

7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(4-oxo-4H-chromen-3-yl)-6,7-dihydrofuro[3′,2′:5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (9f)

Cream powder (yield 60%); mp >260 °C. IR (KBr) (ν_{max} /cm^{-1}): 3428, 3075, 1690, 1624. \(^1\)H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.78 (3H, s, CH_3), 3.03 (3H, s, CH_3), 4.38 (1H, d, J= 4.38 Hz, CH), 5.95 (1H, d, J= 3.54 Hz, CH), 6.94-7.07 (2H, m, H-Ar), 7.24-7.28 (3H, m, H-Ar), 7.48-
7.57 (3H, m, H-Ar), 7.89 (1H, s, H-Ar), 11.79 (1H, bs, OH). MS (EI, 70 eV) m/z: 577 (M⁺, ⁸¹Br), 575 (M⁺, ⁷⁹Br). Anal. Calcd for C₂₇H₁₈BrN₃O₇: C, 56.27; H, 3.15; N, 7.29. Found: C, 56.17; H, 3.08; N, 7.21.

7-(4-bromobenzoyl)-5-hydroxy-1,3-dimethyl-6-(6-methyl-4-oxo-4H-chromen-3-yl)-6,7-dihydrofuro[3',2':5,6]pyrido[2,3-d]pyrimidine-2,4(1H,3H)-dione (9g)

Brown powder (yield 65%); mp 245 °C (dec). IR (KBr) (νmax /cm⁻¹): 3464, 1698, 1675, 1637. ¹H NMR (300 MHz, DMSO-d₆): δH (ppm) 2.39 (3H, s, CH₃), 3.22 (3H, s, CH₃), 3.47 (3H, s, CH₃), 4.81 (1H, bs, CH), 6.37 (1H, bs, CH), 7.58-7.69 (2H, m, H-Ar), 7.79 (3H, bs, H-Ar), 7.91-7.93 (2H, m, H-Ar), 8.28 (1H, bs, H-Ar), 12.44 (1H, bs, OH). MS (EI, 70 eV) m/z: 591 (M⁺, ⁸¹Br), 589 (M⁺, ⁷⁹Br). Anal. Calcd for C₂₈H₂₀BrN₃O₇: C, 56.96; H, 3.41; N, 7.12. Found: C, 56.84; H, 3.48; N, 7.02.
Electronic Supplementary Material (ESI) for RSC Advances
This journal is © The Royal Society of Chemistry 2013

File: DI_90_X61  Date 2/19/81  Time 12:17:58
S=[112->123]  Bp=135  R1=384610,  RT=2.04  CT=294

SB=30  SE=520  DB=30  DE=520  N=0  Z=2  T=0.0  Fact[379->530] *16
S List >  S=[112->123]  B=0  Pos=3  Tot=3
S = [97->114] BP = 77 Bi = 90870. RT = 1.89 CT = 270
SB = 30 SE = 740 DB = 0 DE = 740 N = 0 Z = 2 T = 0.0 Fact[443->792] x 128
S List > S = [97->114] B = 0 Pos = 6 Tot = 6