An Efficient One-Pot Synthesis of Novel Pyrimidoquinoline Derivatives under Microwave Irradiation without Catalyst

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A series of pyrimidoquinoline derivatives were synthesized through one-pot condensation of 2,6-diaminopyrimidin-4-one, aldehyde and cyclic a 1,3-dicarbonyl compound in glycol under microwave irradiation without catalyst. The protocol in the absence of catalyst has the advantage of good yield (87-95%), short reaction time (4-7 min) and an environmentally friendly technique.

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Pyrimidine and its derivatives have been studied for over a century due to a variety of chemical and biological significance. They have been reported as antibacterial, antiviral and antitumor agents [1]. A number of heterocylic compounds fused with pyrimidines are known for their varied biological activities [2-6].

Microwave activation as a non-conventional energy source has been adapted to the assembly of a library of compounds, and the protocol has the advantage of short reaction time and high yield.

In order to investigate new biological chemicals comprising the pyrimidine moiety, we employed the reaction to afford pyrimidoquinoline derivatives, which were synthesized by equimolecular amounts of 2,6-diaminopyrimidin-4-one with the cyclic 1,3-dicarbonyl compound and appropriate aldehyde without catalyst in a small amount of glycol under microwave irradiation (Scheme 1). After irradiation for 4-7 min, the pyrimidoquinoline derivatives with pyrimidine unit were obtained in excellent yields. Besides, compared with the traditional heating methodology in similar condition, we found that the reaction could complete within 7 min under microwave irradiation and at 100 °C within 2 h using standard thermal conditions. The results are listed in Table 1.

The solvent glycol as an energy transfer agent plays a critical role in the success of the reaction, owing to its high boiling point and good dehydrating properties. It can particularly accelerate dehydration in the reaction.

The procedure is easy to operate, and the workup procedure is just simple filtrations. Furthermore, the protocol can be applied not only for the aromatic aldehyde but also for aliphatic aldehyde.

Scheme 1

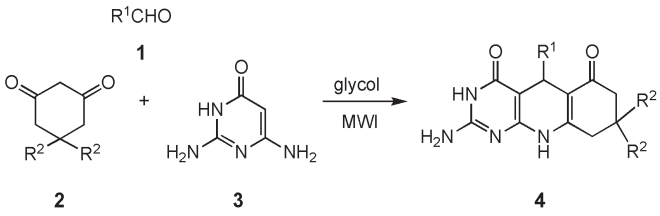


Table 1

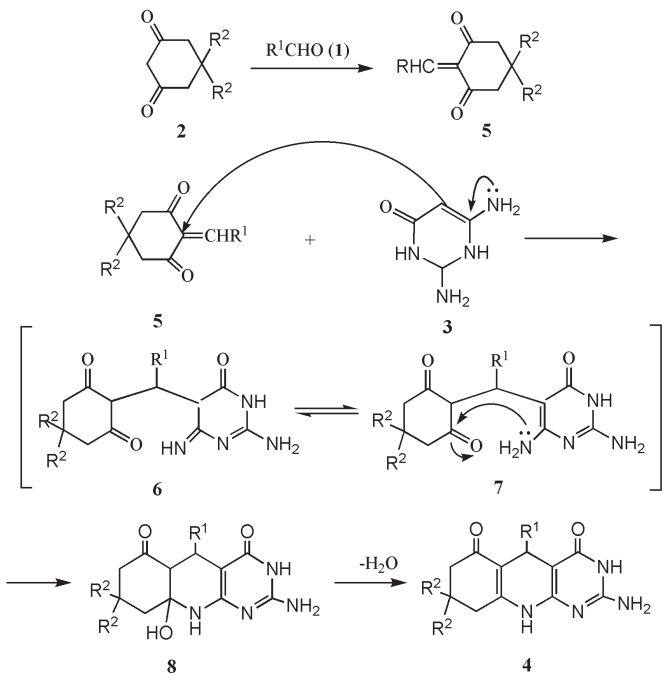
Synthesis of Pyrimidinoquinoline Derivatives Under Microwave Irradiation

Entry	R^1	R^2	Time (min)	Yield (%)	Mp (°C)
4a	4-Cl-C_6H_4	CH_3	6 [a] (120) [b]	95 [a] (91) [b]	>300
4b	2-Cl-C_6H_4	CH_3	6 [a] (100) [b]	91 [a] (90) [b]	>300
4c	4-NO_2-C_6H_4	CH_3	6	93	>300
4d	3-NO_2-C_6H_4	CH_3	7	92	>300
4e	3,4-$(OCH_3)_2$-C_6H_3	CH_3	5	95	>300
4f	3,4-OCH_2O-C_6H_3	CH_3	6	95	>300
4g	$CH_3CH_2CH_2$	CH_3	5	91	>300
4h	4-Cl-C_6H_4	H	5	92	>300
4i	3-NO_2-C_6H_4	H	5	92	>300
4j	4-NO_2-C_6H_4	H	5	94	>300
4k	4-Br-C_6H_4	H	4	91	>300
4l	4-OCH_3-C_6H_4	H	6	91	>300
4m	3,4-Cl_2-C_6H_3	H	5	90	>300
4n	$CH_3CH_2CH_2CH_2$	H	6	87	>300

[a] Method A: in glycol, under microwave irradiation; [b] Method B: in glycol at 100 °C.

This reaction may occur *via* a condensation, addition, cyclization, elimination mechanism (Scheme 2). The condensation between aldehyde and cyclic 1,3-dicarbonyl compound gave 2-arylidene-5,5-dimethyl-1,3-cyclohexanedione **5**. Michael addition between **5** and 2,6-diaminopyrimidin-4-one **3** then furnished the intermediate **6**, which isomerized to **7**. Intramolecular cyclodehydration of **8** gave **4**.

Scheme 2



In conclusion, we have disclosed a facile case, using the microwave heating mode in a small amount of glycol without catalyst. This present one-pot synthesis of pyrimidoquinoline derivatives is a simple, timesaving, high-yielding, and an environmentally friendly process.

Pyrimidoquinoline as a new class of compounds including the pyrimidine moiety are interesting new lead compounds for biological activity evaluation. This work is in progress in our laboratory.

EXPERIMENTAL

Microwave irradiation was carried out in a modified commercial microwave oven (2450 MHz, Nanjing Sanle) under atmospheric pressure. Melting points were determined in open capillaries and are uncorrected. The mass spectra were recorded on a LCQ Advantage instrument. The ir spectra were recorded on a Shimadzu spectrometer. ^{1}H and ^{13}C nmr spectra were measured on a DPX 400 spectrometer operating at 400 and 100 MHz respectively, using DMSO-d_6 as solvent and TMS as internal standard. Elemental analyses were determined by using a Perkin-Elmer 240c elemental analysis instrument.

General Procedure for Pyrimidinoquinoline Derivatives **4**.

The mixture of aldehyde (2 mmol), cyclic 1,3-dicarbonyl compound (2 mmol) and 2,6-diaminopyrimidin-4-one (2 mmol) in glycol (1 ml) was irradiated for 4-7 min with power 300 W. The achieved temperature in the mean reaction time was about 198 °C. The reaction mixture was cooled to room temperature and poured into water (50 mL), filtered and washed with ethanol (5 mL) to give the product. All products were characterized by ms, ir, ^{1}H and ^{13}C nmr spectral data as well as the elemental analyses.

5-(4-Chlorophenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4a**).

This compound was obtained according to the above general procedure; ms: m/z found 371, calcd 371 $(M+H)^+$; ir (KBr): ν 3468, 3246, 3189, 1660, 1621 cm^{-1}; ^{1}H nmr: δ 10.39 (s, 1H, NH), 9.35 (s, 1H, NH), 7.21 (d, 2H, J=8.4 Hz, ArH), 7.16 (d, 2H, J=8.4 Hz, ArH), 6.33 (s, 2H, NH_2), 4.78 (s, 1H, CH), 2.35-2.50 (m, 2H, CH_2), 1.95-2.18 (m, 2H, CH_2), 1.00 (s, 3H, CH_3), 0.88 (s, 3H, CH_3); ^{13}C nmr: δ 194.4 (C6), 162.0 (C4), 154.7 (C2), 154.6 (C1a), 152.1 (C9a), 147.2 (Ar-C), 130.5 (Ar-C), 129.9 (2Ar-C), 128.1 (2Ar-C), 110.0 (C6a), 92.3 (C4a), 50.8 (C5), 33.9 (C7), 32.7 ($2CH_3$), 29.6 (C9), 27.4 (C8).

Anal. Calcd. for $C_{19}H_{19}ClN_4O_2$: C, 61.54; H, 5.16; N, 15.11. Found: C, 61.32; H, 5.11; N, 15.22.

5-(2-Chlorophenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4b**).

This compound was obtained according to above general procedure; ms: m/z found 371, calcd 371 $(M+H)^+$; ir (KBr): ν 3406, 3247, 3188, 1671, 1624 cm^{-1}; ^{1}H nmr: δ 10.22 (s, 1H, NH), 9.34 (s, 1H, NH), 7.02-7.27 (m, 4H, ArH), 6.29 (s, 2H, NH_2), 5.09 (s, 1H, CH), 2.30-2.45 (m, 2H, CH_2), 1.87-2.14 (m, 2H, CH_2), 0.99 (s, 3H, CH_3), 0.87 (s, 3H, CH_3); ^{13}C nmr: δ 194.1 (C6), 162.9 (C4), 154.7 (C2), 154.6 (C1a), 152.2 (C9a), 145.3 (Ar-C), 133.0 (Ar-C), 132.5 (Ar-C), 129.5 (Ar-C), 127.4 (Ar-C), 126.7 (Ar-C), 110.0 (C6a), 92.3 (C4a), 50.8 (C5), 33.9 (C7), 32.7 ($2CH_3$), 29.6 (C9), 27.4 (C8).

Anal. Calcd. for $C_{19}H_{19}ClN_4O_2$: C, 61.54; H, 5.16; N, 15.11. Found: C, 61.20; H, 5.13; N, 15.12.

5-(4-Nitrophenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4c**).

This compound was obtained according to the above general procedure; ms: m/z found 382, calcd 382 $(M+H)^+$; ir (KBr): ν 3467, 3412, 3258, 1667, 1622 cm^{-1}; ^{1}H nmr: δ 10.44 (s, 1H, NH), 9.47 (s, 1H, NH), 8.07 (d, 2H, J=8.4 Hz, ArH), 7.42 (d, 2H, J=8.4 Hz, ArH), 6.39 (s, 2H, NH_2), 4.89 (s, 1H, CH), 2.38-2.45 (m, 2H, CH_2), 1.95-2.19 (m, 2H, CH_2), 1.00 (s, 3H, CH_3), 0.88 (s, 3H, CH_3); ^{13}C nmr: δ 196.6 (C6), 164.0 (C4), 158.0 (C2), 157.2 (C1a), 156.9 (C9a), 154.9 (Ar-C), 148.3 (Ar-C), 131.5 (2Ar-C), 125.8 (2Ar-C), 111.5 (C6a), 93.8 (C4a), 52.7 (C5), 37.3 (C7), 34.9 ($2CH_3$), 31.7 (C9), 29.6 (C8).

Anal. Calcd. for $C_{19}H_{19}N_5O_4$: C, 59.84; H, 5.02; N, 18.36. Found: C, 59.59; H, 5.11; N, 18.16.

5-(3-Nitrophenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4d**).

This compound was obtained according to the above general procedure; ms: m/z found 382, calcd 382 $(M+H)^+$; ir (KBr): ν

3469, 3346, 3256, 1665, 1619 cm^{-1}; ^1H nmr: δ 10.43 (s, 1H, NH), 9.48 (s, 1H, NH), 7.94-7.98 (m, 2H, ArH), 7.47-7.64 (m, 2H, ArH), 6.40 (s, 2H, NH_2), 4.90 (s, 1H, CH), 2.43-2.48 (m, 2H, CH_2), 1.96-2.21 (m, 2H, CH_2), 1.01 (s, 3H, CH_3), 0.89 (s, 3H, CH_3); ^{13}C nmr: δ 196.7 (C6), 165.0 (C4), 157.1 (C2), 156.9 (C1a), 154.9 (C9a), 152.5 (Ar-C), 150.2 (Ar-C), 137.2 (Ar-C), 131.9 (Ar-C), 127.4 (Ar-C), 123.1 (Ar-C), 110.0 (C6a), 92.3 (C4a), 50.8 (C5), 33.9 (C7), 32.7 ($2CH_3$), 29.6 (C9), 27.4 (C8).

Anal. Calcd. for $C_{19}H_{19}N_5O_4$: C, 59.84; H, 5.02; N, 18.36. Found: C, 59.62; H, 4.98; N, 18.25.

5-(3,4-Dimethoxyphenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4e**).

This compound was obtained according to the above general procedure; ms: m/z found 395, calcd 395 $(M-H)^-$; ir (KBr): ν 3427, 3267, 3176, 1670, 1620 cm^{-1}; ^1H nmr: δ 10.34 (s, 1H, NH), 9.27 (s, 1H, NH), 6.83 (s, 1H, ArH), 6.73 (d, 1H, J=8.4 Hz, ArH), 6.63 (d, 1H, J=8.4 Hz, ArH), 6.28 (s, 2H, NH_2), 4.76 (s, 1H, CH), 3.65 (s, 6H, OCH_3), 2.35-2.48 (m, 2H, CH_2), 1.97-2.19 (m, 2H, CH_2), 1.01 (s, 3H, CH_3), 0.93 (s, H, CH_3); ^{13}C nmr: δ 194.5 (C6), 161.8 (C4), 154.6 (C2), 154.5 (C1a), 151.7 (C9a), 148.5 (Ar-C), 147.4 (Ar-C), 140.7 (Ar-C), 119.7 (Ar-C), 112.4 (Ar-C), 111.9 (Ar-C), 110.3 (C6a), 92.9 (C4a), 56.1 ($2OCH_3$), 50.8 (C5), 33.3 (C7), 32.6 ($2CH_3$), 29.8 (C9), 27.2 (C8).

Anal. Calcd. for $C_{21}H_{24}N_4O_4$: C, 63.62; H, 6.10; N, 14.13. Found: C, 63.38; H, 6.18; N, 14.03.

5-(3,4-Methylenedioxyphenyl)-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (4f).

This compound was obtained according to above general procedure; ms: m/z found 381, calcd 381 $(M+H)^+$; ir (KBr): ν 3417, 3245, 3194, 1655, 1614 cm^{-1}; ^1H nmr: δ 10.36 (s, 1H, NH), 9.29 (s, 1H, NH), 6.59-6.70 (m, 3H, ArH), 6.30 (s, 2H, NH_2), 5.89 (s, 2H, CH_2), 4.72 (s, 1H, CH), 2.40-2.49 (m, 2H, CH_2), 1.98-2.17 (m, 2H, CH_2), 1.00 (s, 3H, CH_3), 0,91 (s, 3H, CH_3); ^{13}C nmr: δ 194.5 (C6), 162.1 (C4), 154.6 (C2), 154.4 (C1a), 151.8 (C9a), 147.1 (Ar-C), 145.5 (Ar-C), 142.5 (Ar-C), 120.6 (Ar-C), 110.3 (Ar-C), 108.7 (Ar-C), 108.1 (C6a), 101.0 (OCH_2O), 92.8 (C4a), 50.8 (C5), 33.7 (C7), 32.7 ($2CH_3$), 29.6 (C9), 27.4 (C8).

Anal. Calcd. for $C_{20}H_{20}N_4O_4$: C, 63.15; H, 5.30; N, 14.73. Found: C, 63.33; H, 5.38; N, 14.78.

5-Propyl-8,8-dimethyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4g**).

This compound was obtained according to the above general procedure; ms: m/z found 303, calcd 303 $(M+H)^+$; ir (KBr): ν 3332, 3223, 3112, 1660, 1622 cm^{-1}; ^1H nmr: δ 10.39 (s, 1H, NH), 9.01 (s, 1H, NH), 6.28 (s, 2H, NH_2), 3.82 (t, 1H, J=4.0 Hz, CH), 2.50-2.51 (m, 2H, CH_2), 2.18-2.33 (m, 2H, CH_2), 2.04-2.17 (m, 2H, CH_2), 1.29-1.44 (m, 2H, CH_2), 1.01 (s, 3H, CH_3), 0.95 (s, 3H, CH_3), 0.62 (t, 3H, J=7.2 Hz, CH_3); ^{13}C nmr: δ 194.8 (C6), 162.4 (C4), 155.5 (C2), 154.5 (C1a), 152.9 (C9a), 108.8 (C6a), 90.7 (C4a), 50.8 (C5), 32.5 ($2CH_3$), 29.8 (C7), 28.8 (C9), 28.0 (CH_2), 27.3 (C8), 26.8 (CH_2), 9.7 (CH_3).

Anal. Calcd. for $C_{16}H_{22}N_4O_2$: C, 63.56; H, 7.33; N, 18.53. Found: C, 63.58; H, 7.29; N, 18.47.

5-(4-Chlorophenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4h**).

This compound was obtained according to the above general procedure; ms: m/z found 341, calcd 341 $(M-H)^-$; ir (KBr): ν 3332, 3221, 3149, 1645, 1616 cm^{-1}; ^1H nmr: δ 10.38 (s, 1H, NH), 9.39 (s, 1H, NH), 7.22 (d, 2H, J=8.4 Hz, ArH), 7.19 (d, 2H, J=8.4 Hz, ArH), 6.32 (s, 2H, NH_2), 4.84 (s, 1H, CH), 2.50-2.56 (m, 2H, $COCH_2$), 2.17-2.23 (m, 2H, =C-CH_2), 1.89-1.90 (m, 2H, CH_2); ^{13}C nmr: δ 194.8 (C6), 162.1 (C4), 154.7 (C2), 154.5 (C1a), 154.1 (C9a), 147.2 (Ar-C), 130.5 (Ar-C), 129.8 (2Ar-C), 128.1 (2Ar-C), 111.0 (C6a), 92.2 (C4a), 37.3 (C5), 33.6 (C7), 27.1 (C9), 21.5 (C8).

Anal. Calcd. for $C_{17}H_{15}ClN_4O_2$: C, 59.57; H, 4.41; N, 16.34. Found: C, 59.39; H, 4.39; N, 16.26.

5-(3-Nitrophenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4i**).

This compound was obtained according to the above general procedure; ms: m/z found 352, calcd 352 $(M-H)^-$; ir (KBr) : ν 3334, 3225, 3156, 1648, 1619 cm^{-1}; ^1H nmr: δ 10.53 (s, 1H, NH), 9.51 (s, 1H, NH), 7.94-8.02 (m, 2H, ArH), 7.47-7.65 (m, 2H, ArH), 6.39 (s, 2H, NH_2), 4.96 (s, 1H, CH), 2.51-2.58 (m, 2H, $COCH_2$), 2.19-2.21 (m, 2H, =C-CH_2), 1.72-1.93 (m, 2H, CH_2); ^{13}C nmr: δ 194.9 (C6), 162.1 (C4), 154.9 (C2), 154.7 (C1a), 154.6 (C9a), 150.4 (Ar-C), 148.0 (Ar-C), 134.9 (Ar-C), 129.8 (Ar-C), 122.5 (Ar-C), 121.2 (Ar-C), 110.4 (C6a), 91.7 (C4a), 37.2 (C5), 34.5 (C7), 27.1 (C9), 21.4 (C8).

Anal. Calcd. for $C_{17}H_{15}N_5O_4$: C, 57.79; H, 4.28; N, 19.82. Found: C, 57.51; H, 4.25; N, 19.78.

5-(4-Nitrophenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4j**).

This compound was obtained according to the above general procedure; ms: m/z found 352, calcd 352 $(M-H)^-$; ir (KBr): ν 3328, 3221, 3149, 1648, 1619 cm^{-1}; ^1H nmr: δ 10.42 (s, 1H, NH), 9.50 (s, 1H, NH), 8.06 (d, 2H, J=8.4 Hz, ArH), 7.45 (d, 2H, J=8.4 Hz, ArH), 6.37 (s, 2H, NH_2), 4.95 (s, 1H, CH), 2.49-2.56 (m, 2H, $COCH_2$), 2.13-2.27 (m, 2H, =C-CH_2), 1.82-1.94 (m, 2H, CH_2); ^{13}C nmr: δ 194.8 (C6), 162.1 (C4), 155.8 (C2), 154.9 (C1a), 154.7 (C9a), 154.6 (Ar-C), 146.1 (Ar-C), 129.3 (2Ar-C), 123.6 (2Ar-C), 110.4 (C6a), 91.5 (C4a), 37.2 (C5), 34.8 (C7), 27.1 (C9), 21.4 (C8).

Anal. Calcd for $C_{17}H_{15}N_5O_4$: C, 57.79; H, 4.28; N, 19.82. Found: C, 57.48; H, 4.29; N, 19.71.

5-(4-Bromophenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4k**).

This compound was obtained according to the above general procedure; ms: m/z found 388, calcd 388 $(M+H)^+$; ir (KBr): ν 3338, 3226, 3149, 1645, 1615 cm^{-1}; ^1H nmr: δ 10.39 (s, 1H, NH), 9.39 (s, 1H, NH), 7.35 (d, 2H, J=8.4 Hz, ArH), 7.13 (d, 2H, J=8.4 Hz, ArH), 6.32 (s, 2H, NH_2), 4.82 (s, 1H, CH), 2.49-2.59 (m, 2H, $COCH_2$), 2.14-2.22 (m, 2H, =C-CH_2), 1.78-1.91 (m, 2H, CH_2); ^{13}C nmr: δ 194.8 (C6), 162.1 (C4), 154.7 (C2), 154.5 (C1a), 154.1 (C9a), 147.6 (Ar-C), 131.0 (2Ar-C), 130.3 (2Ar-C), 119.0 (Ar-C), 111.0 (C6a), 92.2 (C4a), 37.3 (C5), 33.7 (C7), 27.1 (C9), 21.5 (C8).

Anal. Calcd. for $C_{17}H_{15}BrN_4O_2$: C, 52.73; H, 3.90; N, 14.47. Found: C, 52.48; H, 3.88; N, 14.45.

5-(4-Methoxyphenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4l**).

This compound was obtained according to the above general procedure; ms: m/z found 339, calcd 339 $(M+H)^+$; ir (KBr): ν 3334, 3220, 3154, 1643, 1615 cm^{-1}; ^1H nmr: δ 10.32 (s, 1H, NH),

9.46 (s, 1H, NH), 7.13 (d, 2H, J=8.4 Hz, ArH), 6.74 (d, 2H, J=8.4 Hz, ArH), 6.37 (s, 2H, NH_2), 4.82 (s, 1H, CH), 3.69 (s, 3H, OCH_3), 2.49-2.58 (m, 2H, $COCH_2$), 2.18-2.23 (m, 2H, =C-CH_2), 1.75-1.92 (m, 2H, CH_2); ^{13}C nmr: δ 194.6 (C6), 162.7 (C4), 157.6 (Ar-C), 155.5 (C2), 154.9 (C1a), 154.3 (C9a), 130.0 (2Ar-C), 122.3 (Ar-C), 115.0 (2Ar-C), 110.2 (C6a), 91.8 (C4a), 37.1 (C5), 33.2 (C7), 27.2 (C9), 21.7 (C8).

Anal. Calcd. for $C_{18}H_{18}N_4O_3$: C, 63.89; H, 5.36; N, 16.56. Found: C, 63.58; H, 5.40; N, 16.49.

5-(3,4-Dichlorophenyl)-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4m**).

This compound was obtained according to the above general procedure; ms: m/z found 378, calcd 378 $(M+H)^+$; ir (KBr): ν 3335, 3222, 3155, 1649, 1613 cm^{-1}; ^{1}H nmr: δ 10.42 (s, 1H, NH), 9.46 (s, 1H, NH), 7.44-7.13 (m, 3H, ArH), 6.37 (s, 2H, NH_2), 4.82 (s, 1H, CH), 2.49-2.58 (m, 2H, $COCH_2$), 2.18-2.23 (m, 2H, =C-CH_2), 1.73-1.92 (m, 2H, CH_2); ^{13}C nmr: δ 194.8 (C6), 162.1 (C4), 154.9 (C2), 154.6 (C1a), 154.5 (C9a), 149.2 (Ar-C), 130.6 (Ar-C), 130.5 (Ar-C), 130.0 (Ar-C), 128.5 (Ar-C), 128.3 (Ar-C), 110.4 (C6a), 91.6 (C4a), 37.2 (C5), 33.9 (C7), 27.1 (C9), 21.4 (C8).

Anal. Calcd. for $C_{17}H_{14}Cl_2N_4O_2$: C, 54.13; H, 3.74; N, 14.85. Found: C, 54.45; H, 3.78; N, 14.73.

5-Butyl-5,6,7,8,9,10-hexahydro-2-aminopyrimido[4,5-*b*]quinoline-4,6-dione (**4n**).

This compound was obtained according to the above general procedure; ms: m/z found 289, calcd 289 $(M+H)^+$; ir (KBr): ν 3338, 3224, 3157, 1649, 1615 cm^{-1}; ^{1}H nmr: δ 10.34 (s, 1H, NH), 9.02 (s, 1H, NH), 6.23 (s, 2H, NH_2), 3.83 (t, 1H, J=4.8 Hz, CH), 2.38-2.50 (m, 2H, $COCH_2$), 2.18-2.26 (m, 2H, =C-CH_2), 1.78-1.90 (m, 2H, CH_2), 1.01-1.33 (m, 6H, CH_2), 0.77 (t, 3H, J=7.2 Hz, CH_3); ^{13}C nmr: δ 195.1 (C6), 162.4 (C4), 155.0 (C2), 154.4 (C1a), 154.3 (C9a), 110.8 (C6a), 91.5 (C4a), 38.0 (C5), 35.1 (C7), 27.7 (C9), 27.3 (CH_2), 27.1 (CH_2), 23.2 (C8), 22.0 (CH_2), 14.8 (CH_3).

Anal. Calcd. for $C_{15}H_{20}N_4O_2$: C, 62.48; H, 6.99; N, 19.43. Found: C, 62.95; H, 6.91; N, 19.46.

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