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Efficient Lactic Acid-catalyzed Route to Naphthopyranopyrimidines under Solvent-free Conditions

Fatemeh Noori Sadeh,¹ Nourallah Hazeri,¹ Malek Taher Maghsoodlou,¹ and Mojtaba Lashkari²

¹Department of Chemistry, Faculty of Sciences, University of Sistan and Baluchestan, P.O. Box 98135-674, Zahedan, Iran

²Faculty of Sciences, Velayat University, Iranshahr, Iran

Multi-component reactions (MCRs) are pivotal because of their versatility for the generation of interesting heterocyclic scaffolds and libraries of biologically active molecules. They have several notable advantageous features such as flexibility, operational simplicity, simple purification of products, minimum reaction steps, time and energy savings, and a high degree of atom economy.^{1–7} In order to avoid the use of organic solvents, solvent-free systems are also important for green chemical syntheses. Solvent-free reactions also have other advantages, encompassing simplicity of operation, reduction of reaction times, and increased yields due to compatibility of reagents.^{8,9}

Naphthopyranopyrimidines are key building blocks for the preparation of biologically active products and display antimicrobial,¹⁰ anticonvulsant,¹¹ antibacterial,¹² and antifungal¹³ activities. Lately Marugan has reported novel drugs for the treatment of sleep and anxiety disorders (*Figure 1*).¹⁴ Previous catalysts used for the preparation of naphthopyranopyrimidines from the condensation of aldehydes with β -naphthol (or 2,7-dihydroxynaphthalene) and 1,3-dimethylbarbituric acid include InCl_3 and P_2O_5 ,¹⁵ I_2/HOAc ,¹⁶ I_2 ,¹⁷ alum ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$),¹⁸ and ZnO NPs.¹⁹



Figure 1
Biologically active compounds containing naphthopyranopyrimidine

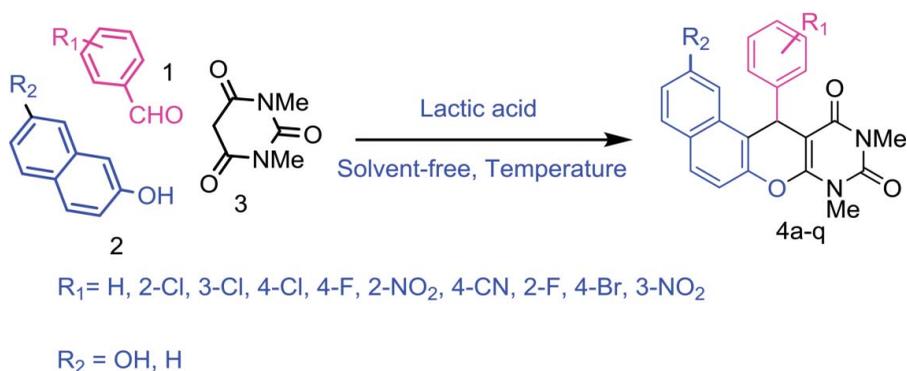
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Address correspondence to Nourallah Hazeri, Department of Chemistry, Faculty of Sciences, University of Sistan and Baluchestan, P.O. Box 98135-674, Zahedan, Iran. E-mail: nhazeri@chem.usb.ac.ir

As a part of our research program to develop greener methodologies,^{20–24} we investigated and now report the application of lactic acid as an efficient, non-toxic, natural, commercially available and inexpensive catalyst for the preparation of various naphthopyranopyrimidines in high yields.

In order to determine the optimum conditions for the synthesis of compounds **4a-i**, trial reactions on one mmole scale between benzaldehyde **1**, 2,7-dihydroxynaphthalene **2**, and 1,3-dimethylbarbituric acid **3** was studied (*Scheme 1*), initially, by carrying out the model reaction at different temperatures in the presence of lactic acid under solvent-free conditions (*Table 1*, *Entries 1–5*); the best result was obtained at 70°C (*Table 1*, *Entry 4*). Next, in order to optimize the amount of catalyst, the reaction was performed using various amounts of catalyst at 70°C (*Table 1*, *Entries 6, 7*), the best yield being obtained in the presence of 0.119 g of lactic acid.



Scheme 1

Lactic acid catalyzed one-pot synthesis of naphthopyranopyrimidines.

The used of lactic acid was extended in the condensation of benzaldehyde, β -naphthol, and 1,3-dimethylbarbituric acid to prepare compounds **4j-q** (*Scheme 1*). However, it was found that under the same conditions used for the preparation of compounds **4a-i**, this reaction was incomplete and a higher temperature and (100°C) was required to give satisfactory yields in the presence of 0.106 g lactic acid (*Table 2*).

The generality of this reaction was investigated using aldehydes (**1**, 1 mmol), 2,7-dihydroxynaphthalene or β -naphthol (**2**, 1 mmol), and 1,3-dimethylbarbituric acid (**3**, 1 mmol) under solvent-free conditions in the presence of lactic acid to generate

Table 1
Effect of Temperature and Amount of Catalyst

Entry	Tem (°C)	Catalyst (g)	Time (min)	Yield (%)
1	r.t	0.119	24(h)	-
2	50	0.119	110	60
3	60	0.119	90	65
4	70	0.119	40	87
5	80	0.119	35	87
6	70	0.08	40	75
7	70	0.093	40	79

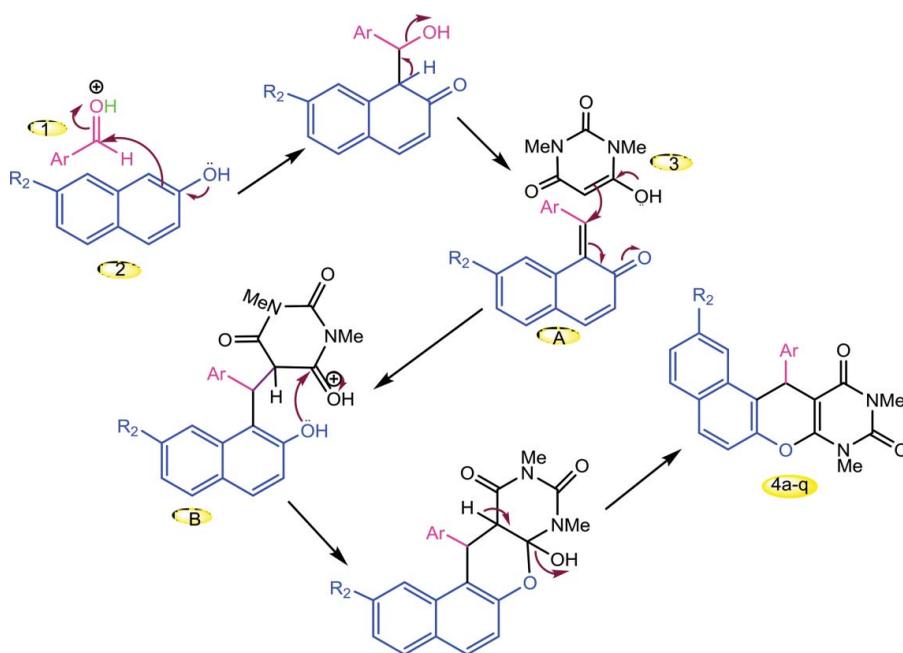
Table 2
Optimization of Reaction Condition

Entry	Tem (°C)	Catalyst (g)	Time (min)	Yield ^a (%)
1	70	0.093	180	45
2	80	0.093	90	63
3	90	0.093	45	76
4	100	0.093	20	80
5	110	0.093	20	81
6	100	0.083	20	75
7	100	0.106	20	84
8	100	0.119	20	83

compounds **4a-i** and compounds **4j-q**. The results are summarized in *Table 3*. A wide range of aldehydes containing electron-withdrawing and electron-donating groups were evaluated. It is noteworthy that the electronic nature of substituents of the aromatic aldehyde has no obvious effect on the multi-component reaction.

A possible mechanism shown in *Scheme 2* may proceed via the *o*-quinonemethides (*o*-*QMs*) intermediate **A**. The initial nucleophilic addition of 2,7-dihydroxynaphthalene or β -naphthol **2** to aldehyde **1**, assisted by lactic acid is followed by the Michael addition of the *o*-*QMs* to 1,3-dimethylbarbituric acid **3** to provide the intermediate **B**. Finally, the naphthopyranopyrimidines are obtained by intramolecular cyclization and loss of water.

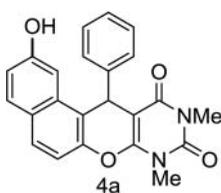
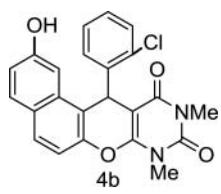
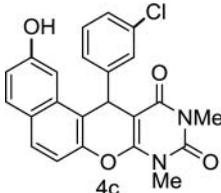
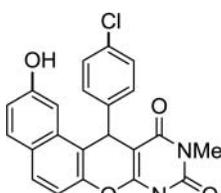
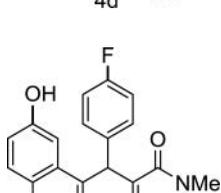
In summary, lactic acid was shown to be an efficient catalyst for the greener synthesis of naphthopyranopyrimidines. The most remarkable advantages for the use lactic acid as



Scheme 2

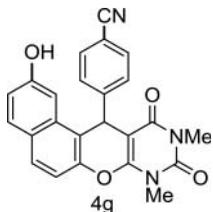
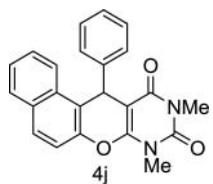
Proposed Mechanism for the Synthesis of Naphthopyranopyrimidines

Table 3
Lactic Acid Catalysed One-pot Three-component Synthesis of
Naphthopyranopyrimidines

Entry	R ₁	R ₂	Product	Time (min)	Yield ^a (%)	Mp (°C)	
						Found	Reported ^{Ref}
1	H	OH	 4a	40	87	>300	>300 ¹⁶
2	2-Cl	OH	 4b	50	89	>300	>300 ¹⁶
3	3-Cl	OH	 4c	45	87	>300	>300 ¹⁸
4	4-Cl	OH	 4d	40	93	>300	>300 ¹⁸
5	4-F	OH	 4e	50	95	>300	>300 ¹⁸

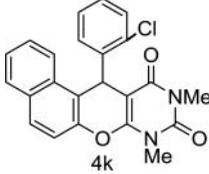
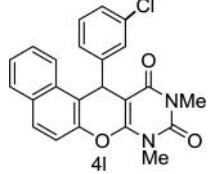
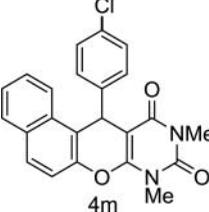
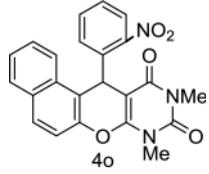
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Table 3
Lactic Acid Catalysed One-pot Three-component Synthesis of Naphthopyranopyrimidines (Continued)

Entry	R ₁	R ₂	Product	Time (min)	Yield ^a (%)	Mp (°C)	
						Found	Reported ^{Ref}
6	2-NO ₂	OH		45	89	>300	>300 ¹⁸
7	4-CN	OH		50	89	>300	>300 ¹⁸
8	2-F	OH		50	91	>300	This work
9	4-Br	OH		45	93	>300	This work
10	H	H		20	84	223–226	223–225 ¹⁷

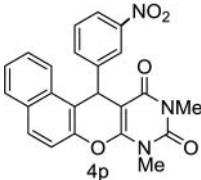
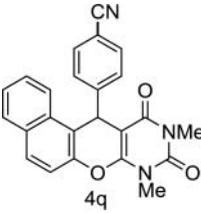
(Continued on next page)

Table 3
Lactic Acid Catalysed One-pot Three-component Synthesis of Naphthopyranopyrimidines (Continued)

Entry	R ₁	R ₂	Product	Time (min)	Yield ^a (%)	Mp (°C)	
						Found	Reported ^{Ref}
11	2-Cl	H		30	91	271–275	270–272 ¹⁵
12	3-Cl	H		30	89	233–237	221–223 ¹⁹
13	4-Cl	H		25	81	268–270	274–276 ¹⁷
14	4-F	H		25	84	298–301	303–305 ¹⁵
15	2-NO ₂	H		35	81	280–284	288–290 ¹⁵

(Continued on next page)

Table 3
Lactic Acid Catalysed One-pot Three-component Synthesis of Naphthopyranopyrimidines (Continued)

Entry	R ₁	R ₂	Product	Time (min)	Yield ^a (%)	Mp (°C)	
						Found	Reported ^{Ref}
16	3-NO ₂	H		25	95	313–315	310–312 ¹⁹
17	4-CN	H		30	90	290–293	288–290 ¹⁷

^aIsolated yield

catalyst were the generation of the desired product in high yields without by-products, with facile work-up, and purification.

Experimental Section

The melting points of all compounds were determined on an Electrothermal 9100 apparatus, and IR spectra were obtained on a FT-IR-JASCO-460 plus spectrometer. ¹H NMR and ¹³CNMR spectra were acquired on a Bruker DRX-300 and DRX-400 Avance instrument in DMSO at 400 and 300 MHz. Mass spectra were taken on an Agilent Technology (HP) spectrometer operating at an ionization potential of 70 eV. All reagents were purchased from Merck (Darmstadt, Germany) and Fluka (Buchs, Switzerland) and used without further purification. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer.

Preparation of 2-Hydroxy-12-aryl-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano-[2,3-d]pyrimidine-9,11-(10H)-dione and 12-Aryl-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)diones. General Procedure.

A mixture of aromatic aldehyde **1** (1.0 mmol), 2,7-dihydroxynaphthalene (weight) or β -naphthol **2** (weight, 1.0 mmol), 1,3-dimethylbarbituric acid **3** (weight, 1.0 mmol), and lactic acid (weight) at the optimum temperature (70°C or 100°C), was stirred without solvent in an oil bath. Completion of reaction was determined by TLC (solvent system). The crude reaction mixture was cooled to RT, the solid formed was suspended in ethanol (5 ml) and collected. The crude solid was recrystallized from ethanol. The structures of the compounds were confirmed by their IR, ¹H NMR and ¹³C NMR spectra and found to be identical with those reported in the literature. Correct combustion analyses were obtained for previously un-reported compounds **4h** and **4i**.

Analytical Data for Selected Compounds

2-Hydroxy-12-phenyl-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]-pyrimidine-9,11-(10H)dione (4a). White solid; IR (KBr, cm^{-1}): 3180, 3086, 2965, 1715, 1665, 1622, 1597, 1453, 1237, 1225, 1178. ^1H NMR (400 MHz), (DMSO-d₆): δ = 3.17 (s, 3H, CH₃), 3.51 (s, 3H, CH₃), 5.44 (s, 1H, CH), 7.00–7.87 (m, 10H, Ar-H), 9.97 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.16, 29.29, 36.04, 90.58, 105.98, 113.68, 115.47, 118.00, 126.19, 126.91, 128.63, 129.63, 130.75, 132.53, 144.61, 147.65, 150.48, 152.56, 157.06, 161.51.

2-Hydroxy-12-(2-Chlorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d] pyrimidine-9,11-(10H)dione (4b): White solid; IR (KBr, cm^{-1}): 3370, 3019, 2953, 1698, 1670, 1645, 1591, 1484, 1238, 1219, 1190. ^1H NMR (300 MHz), (DMSO-d₆): δ = 3.13 (s, 3H, CH₃), 3.47 (s, 3H, CH₃), 5.66 (s, 1H, CH), 7.01–7.80 (m, 9H, Ar-H), 9.92 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.13, 29.35, 34.40, 89.48, 106.30, 113.69, 114.68, 117.89, 126.11, 127.64, 128.67, 129.98, 130.07, 130.74, 132.59, 132.83, 132.90, 141.55, 147.69, 150.48, 152.78, 157.11, 161.24.

2-Hydroxy-12-(3-Chlorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d] pyrimidine-9,11-(10H)-dione (4c). White solid; IR (KBr, cm^{-1}): 3319, 3059, 2960, 1693, 1665, 1640, 1569, 1495, 1229, 1209, 1192. ^1H NMR (300 MHz), (DMSO-d₆): δ = 3.16 (s, 3H, CH₃), 3.47 (s, 3H, CH₃), 5.41 (s, 1H, CH), 6.99–7.86 (m, 9H, Ar-H), 10.01 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.21, 29.38, 35.90, 89.85, 105.81, 113.74, 114.59, 118.11, 126.19, 127.08, 127.41, 128.48, 130.03, 130.55, 130.89, 132.40, 133.19, 146.83, 147.73, 150.49, 152.76, 157.22, 161.56.

2-Hydroxy-12-(4-Chlorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d] pyrimidine-9,11-(10H)-dione (4d). white solid; IR (KBr, cm^{-1}): 3200, 3019, 2960, 1713, 1660, 1621, 1600, 1489, 1235, 1219, 1195. ^1H NMR (300 MHz), (DMSO-d₆): δ = 3.15 (s, 3H, CH₃), 3.45 (s, 3H, CH₃), 5.38 (s, 1H, CH), 6.98–7.83 (m, 9H, Ar-H), 9.98 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.19, 29.36, 35.57, 90.07, 105.89, 113.75, 114.85, 118.09, 126.21, 128.59, 129.93, 130.50, 130.87, 131.55, 132.44, 143.48, 147.68, 150.50, 152.67, 157.17, 161.55.

2-Hydroxy-12-(4-fluorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d] pyrimidine-9,11-(10H)-dione (4e). White solid; IR (KBr, cm^{-1}): 3186, 3080, 2960, 1712, 1661, 1621, 1599, 1452, 1236, 1223, 1179. ^1H NMR (300 MHz), (DMSO-d₆): δ = 3.17 (s, 3H, CH₃), 3.49 (s, 3H, CH₃), 5.45 (s, 1H, CH), 7.00–7.87 (m, 9H, Ar-H), 9.99 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.20, 29.38, 35.35, 90.39, 105.88 (d, $^2J_{\text{FC}}$ = 6.052 Hz, C₆H₄F), 113.83, 115.38 (d, $^2J_{\text{FC}}$ = 21.18 Hz, C₆H₄F), 118.04 (d, $^3J_{\text{FC}}$ = 7.56 Hz, C₆H₄F), 126.26, 129.87, 130.45 (d, $^3J_{\text{FC}}$ = 7.56 Hz, C₆H₄F), 130.90, 132.44, 140.72 (d, $^4J_{\text{FC}}$ = 3.02 Hz, C₆H₄F), 147.73, 150.54, 152.73, 156.98, 157.14, 161.56, 161.19 (d, $^1J_{\text{FC}}$ = 242.83 Hz, C₆H₄F), 161.60.

2-Hydroxy-8,12-dihydro-8,10-dimethyl-12-(2-nitrophenyl)-9H-naphtho[1',2':5,6]pyrano[2,3-d] pyrimidine-9,11-(10H)-dione(4f). White solid; IR (KBr, cm^{-1}): 3356, 3022, 2953, 1699, 1669, 1621, 1591, 1485, 1236, 1184. ^1H NMR (300 MHz), (DMSO-d₆): δ = 3.14 (s, 3H, CH₃), 3.47 (s, 3H, CH₃), 5.53 (s, 1H, CH), 6.97–7.99 (m, 9H, Ar-H), 10.00 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO-d₆): δ = 28.25, 29.44, 32.70, 91.27, 105.74, 113.86, 116.83, 118.12, 126.20, 126.71, 129.62, 130.88, 132.73, 135.74, 143.43, 147.78, 150.43, 152.76, 156.99, 161.77.

2-Hydroxy-12-(4-Cyanophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho

[1',2':5,6]pyra-no[2,3-d] pyrimidine-9,11-(10H)-dione (4g). White solid; IR (KBr, cm^{-1}): 3205, 3019, 2953, 2233, 1713, 1658, 1621, 1602, 1495, 1225, 1195. ^1H NMR (300 MHz), (DMSO- d_6): δ = 3.16 (s, 3H, CH_3), 3.49 (s, 3H, CH_3), 5.51 (s, 1H, CH), 7.00–7.89 (m, 9H, Ar-H), 10.02 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO- d_6): δ = 28.21, 29.43, 36.35, 89.47, 105.76, 109.88, 113.80, 114.22, 118.17, 119.13, 126.21, 129.78, 130.23, 130.96, 132.39, 132.66, 147.76, 147.76, 149.82, 150.50, 152.92, 157.28, 161.54.

2-Hydroxy-12-(2-fluorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho

[1',2':5,6]pyra-no[2,3-d] pyrimidine-9,11-(10H)-dione (4h). White solid; IR (KBr, cm^{-1}): 3377, 3079, 2952, 1699, 1666, 1643, 1590, 1489, 1231, 1216, 1185. ^1H NMR (300 MHz), (DMSO- d_6): δ = 3.14 (s, 3H, CH_3), 3.52 (s, 3H, CH_3), 5.61 (s, 1H, CH), 7.01–7.85 (m, 9H, Ar-H), 10.03 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO- d_6): δ = 28.15, 29.39, 30.74, 89.16, 105.36, 113.83, 114.39, 115.92 (d, $^2J_{\text{FC}} = 21.93$ Hz, $\text{C}_6\text{H}_4\text{F}$), 117.96, 124.80, 126.13, 129.15 (d, $^3J_{\text{FC}} = 8.32$ Hz, $\text{C}_6\text{H}_4\text{F}$), 129.87, 130.90, 131.18 (d, $^3J_{\text{FC}} = 12.86$ Hz, $\text{C}_6\text{H}_4\text{F}$), 131.68, 132.66, 147.74, 150.59, 153.10, 157.05, 150.54, 152.83, 160.13 (d, $^1J_{\text{FC}} = 246.61$ Hz, $\text{C}_6\text{H}_4\text{F}$), 161.41. MS m/z (%): 57.1 (34), 83.1 (22), 188.1 (14), 252.1 (21), 309.2 (100), 404.2 (M+, 20).

Anal. Calcd for $\text{C}_{23}\text{H}_{17}\text{FN}_2\text{O}_4$: C, 68.31; H, 4.24; N, 6.93. Found: C, 68.49; H, 4.28; N, 6.97.

2-Hydroxy-12-(4-bromophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho

[1',2':5,6]pyra-no[2,3-d] pyrimidine-9,11-(10H)-dione (4i). White solid; IR (KBr, cm^{-1}): 3202, 3019, 2960, 1713, 1661, 1621, 1596, 1491, 1236, 1219, 1195. ^1H NMR (300 MHz), (DMSO- d_6): δ = 3.16 (s, 3H, CH_3), 3.48 (s, 3H, CH_3), 5.41 (s, 1H, CH), 7.00–7.87 (m, 9H, Ar-H), 10.02 (s, 1H, OH). ^{13}C NMR (75.65 MHz, DMSO- d_6): δ = 28.20, 29.39, 35.65, 90.01, 105.81, 113.81, 114.82, 115.47, 118.01, 120.09, 126.24, 129.97, 130.64, 130.90, 131.52, 132.43, 133.02, 143.90, 147.70, 148.82, 150.52, 152.71, 157.01, 161.57. MS (m/z): 105.1 (13), 164.1 (28), 209.1 (21), 237.1 (15), 252.1 (56), 309.2 (100), 396.2 (14), 466.2 (M+, 30).

Anal. Calcd for $\text{C}_{23}\text{H}_{17}\text{BrN}_2\text{O}_4$: C, 59.37; H, 3.68; N, 6.02. Found: C, 59.55; H, 3.70; N, 6.09.

12-phenyl-8,10-dimethyl-12H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-dione (4j). White solid; IR (KBr, cm^{-1}): 3105, 2950, 1704, 1665, 1643, 1592, 1484, 1231, 1178. ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.16 (s, 3H, CH_3), 3.51 (s, 3H, CH_3), 5.67 (s, 1H, CH), 7.06–8.04 (m, 11H, Ar-H).

12-(3-Nitrophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione (4p). White solid; IR (KBr, cm^{-1}): 3103, 2951, 1705, 1663, 1639, 1596, 1479, 1232, 1177. ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.17 (s, 3H, CH_3), 3.54 (s, 3H, CH_3), 5.93 (s, 1H, CH), 6.98–8.00 (m, 10H, Ar-H).

12-(4-Cyanophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione (4q). White solid; IR (KBr, cm^{-1}): 3079, 2959, 2235, 1708, 1670, 1646, 1598, 1483, 1229, 1180. ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.17 (s, 3H, CH_3), 3.53 (s, 3H, CH_3), 5.65 (s, 1H, CH), 6.99–8.05 (m, 10H, Ar-H).

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