

Supporting Information

TriazineBis(pyridinium) Hydrogen Sulfate Ionic Liquid Immobilized on Functionalized Halloysite Nanotubes As An Efficient Catalyst for One-Pot Synthesis of Naphthopyranopyrimidines

Marzieh Samadani, Beheshteh Asadi, Iraj Mohammadpoor-Baltork,* Valiollah Mirkhani, Shahram Tangestaninejad and Majid Moghadam

Department of Chemistry, Catalysis Division, University of Isfahan, Isfahan 81746-73441, Iran, E-mail: imbaltork@sci.ui.ac.ir; Fax: +98 31 3668 9732; Tel.: +98 31 3793 4927

Table of Contents

1. General	S2
2. General procedure for Preparation of [(PATDBP)(HSO ₄) ₂ @HNT]	S2-S3
3. General procedure for synthesis of naphthopyranopyrimidine derivatives catalyzed by [(PATDBP)(HSO ₄) ₂ @HNT].....	S3
4. Spectroscopic data of the products.....	S3-S8
5. ¹ H NMR and ¹³ C NMR spectra of the products.....	S9-S25
6. References.....	S26

1. General

The chemicals used in this work were purchased from Fluka and Merck chemical companies. HNTs were purchased from Sigma-Aldrich (Germany) chemical company. The properties of HNTs are as follows: diameter = 30-70 nm, length = 1-3 μm , cation exchange capacity = 8.0 meq/g, density = 2.53 g/cm^3 , average hydrodynamic diameter = 399.7 nm, specific surface area ($a_{\text{s,BET}} = 39.156 \text{ m}^2\text{g}^{-1}$), total pore volume = 0.214 cm^3g^{-1}) and elemental compositions = $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot 2\text{H}_2\text{O}$ (1:1 ratio of Al/Si). Melting points were determined with a Stuart Scientific SMP2 apparatus. FT-IR spectra were recorded on a Nicolet-Impact 400D spectrophotometer. ^1H and ^{13}C NMR (400 and 100 MHz) spectra were recorded on a Bruker Avance 400 MHz spectrometer using CDCl_3 as solvent. Elemental analysis was performed on a LECO, CHNS-932 analyzer. Thermogravimetric analysis (TGA) was carried out on a Mettler TG50 instrument under air flow at a uniform heating rate of 20 $^\circ\text{C min}^{-1}$ in the range 30-740 $^\circ\text{C}$. The TGA instrument was re-calibrated at frequent intervals with standards; the accuracy was always better than $\pm 2.0\%$. The scanning electron microscope measurement was carried out on a Hitachi S-4700 field emission-scanning electron microscope (FE-SEM). The transmission electron microscopy (TEM) was carried out on a Philips CM10 instrument operating at 100 kV.

2. General procedure for Preparation of [(PATDBP)(HSO₄)₂@HNT]

Preparation of [PA@HNT]

First, HNT (2 g) was dispersed ultrasonically in 8 ml 3-aminopropyltriethoxysilane (APTS) for 5 min at room temperature. Then, the suspension was exposed to microwave irradiation in a closed vessel at 100 $^\circ\text{C}$ and a power of 25 W under constant stirring for 1 h. Finally, the precipitate (PA@HNT) was filtered, washed with ethanol ($2 \times 10 \text{ mL}$), and dried in a vacuum oven at 80 $^\circ\text{C}$.

Preparation of [PAT@HNT]

The obtained PA@HNT (2 g) was dispersed in 25 mL dry THF by sonication for 10 min and then, 1,3,5-trichlorotriazine (20 mmol, 3.68 g) and DIPEA (14.4 mmol, 2.5 mL) were added and the mixture was stirred for 24 hours under N_2 at 0 $^\circ\text{C}$. Finally, the precipitate PAT@HNT was filtered, washed with THF ($2 \times 10 \text{ mL}$) and acetone (10 mL), and dried in a vacuum oven at 80 $^\circ\text{C}$.

Preparation of [(PATDBP)Cl₂@HNT]

A mixture of PAT@HNT (2 g) and DIPEA (11.5 mmol, 2 mL) was intensely dispersed in 17 mL pyridine and then, the reaction mixture was refluxed for 24 hours. Finally, the mixture was filtered, washed with toluene three times and dried under vacuum to afford [(PATDBP)Cl₂@HNT].

Preparation of [(PATDBP)(HSO₄)₂@HNT]

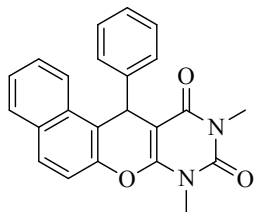
The nanocatalyst [(PATDBP)(HSO₄)₂@HNT] was generated by addition of NaHSO₄ (5 g) to a dispersed mixture of [(PATDBP)Cl₂@HNT] (2 g) in Mili-Q water (30 mL) at room temperature and then, was shaken for 24 hours. The resulting light brown solid was collected by filtration and washed with H₂O (3×10 mL) to remove the unreacted NaHSO₄, and finally dried in a vacuum oven at 80 °C for 5 hours.

3. General procedure for synthesis of naphthopyranopyrimidine derivatives catalyzed by [(PATDBP)(HSO₄)₂@HNT]

A mixture of β -naphthol (1 mmol), aromatic aldehyde (1 mmol), and *N,N*-dimethylbarbituric acid (1 mmol), and [(PATDBP)(HSO₄)₂@HNT] (2 mol%, 28 mg) was stirred at 100 °C under solvent-free conditions for the appropriate time according to Scheme 3. The progress of the reaction was tested by TLC (eluent: petroleum ether/EtOAc, 4:1). After completion of the reaction, the mixture was cooled to room temperature and CHCl₃ (5 mL) was added. The catalyst was quickly separated by centrifugation and washed with EtOH (5 mL). The products were obtained by recrystallization from EtOH and dried under reduced pressure. In some cases, the organic residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate) to provide the pure product in 90-98% isolated yields (Scheme 3, **4a-n**).

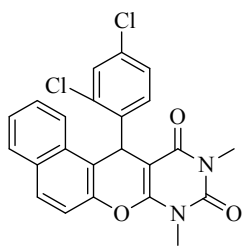
4. Spectroscopic data of the products:

8,10-Dimethyl-12-phenyl-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione (Scheme 3, **4a)**



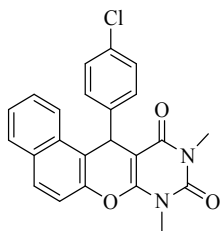
Mp 224-226 °C. (Lit.^[1] 223-225 °C) IR (KBr): ν_{\max} = 3105, 2922, 2856, 1709, 1645, 1594, 1486, 1452, 1233, 1179, 806, 745 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 3.36 (s, 3H), 3.63 (s, 3H), 5.84 (s, 1H), 7.11-7.15 (m, 1H), 7.21-7.25 (m, 2H), 7.38-7.51 (m, 5H), 7.83-7.88 (m, 2H), 7.98 (d, J = 8.4 Hz, 1H). Anal. Calcd for C₂₃H₁₈N₂O₃: C, 74.58; H, 4.90; N, 7.56. Found: C, 74.75; H, 4.86; N, 7.63.

12-(2,4-Dichlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4b)



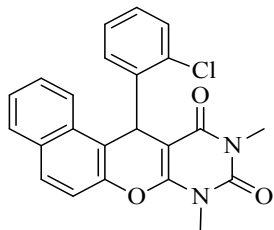
Mp 219-220 °C. (Lit.^[1] 219-221 °C) IR (KBr): ν_{\max} = 3058, 2949, 2884, 1707, 1646, 1593, 1482, 1452, 1399, 1182, 953, 840, 749 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 3.35 (s, 3H), 3.66 (s, 3H), 6.08 (s, 1H), 7.09 (dd, ¹ J = 8.4 Hz, ² J = 2.0 Hz, 1H), 7.24 (d, J = 8.4 Hz, 1H), 7.34 (d, J = 2.0 Hz, 1H), 7.38 (d, J = 9.2 Hz, 1H), 7.45-7.49 (m, 1H), 7.54 (td, ¹ J = 8.4 Hz, ² J = 1.2 Hz, 1H), 7.84 (t, J = 7.2 Hz, 2H), 8.11 (d, J = 8.0 Hz, 1H). Anal. Calcd for C₂₃H₁₆Cl₂N₂O₃: C, 62.88; H, 3.67; N, 6.38. Found: C, 62.74; H, 3.71; N, 6.27.

12-(4-Chlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4c)



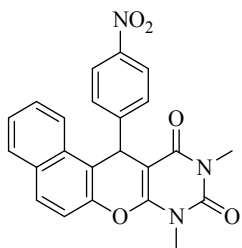
Mp 273-275 °C. (Lit.^[1] 274-276 °C) IR (KBr): ν_{\max} = 3046, 2955, 2924, 2852, 1710, 1671, 1598, 1484, 1452, 1226, 1071, 872, 808 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 3.27 (s, 3H), 3.54 (s, 3H), 5.72 (s, 1H), 7.08-7.11 (m, 2H), 7.22-7.24 (m, 2H), 7.32-7.43 (m, 3H), 7.75-7.82 (m, 3H). Anal. Calcd for C₂₃H₁₇ClN₂O₃: C, 68.23; H, 4.23; N, 6.92. Found: C, 68.37; H, 4.20; N, 6.99.

12-(2-Chlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4d)



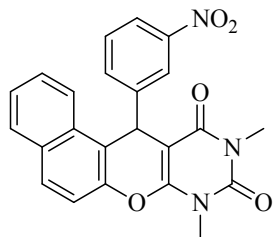
Mp 271-272 °C. (Lit.^[2] 270-272 °C) IR (KBr): ν_{\max} = 3058, 2951, 2922, 2854, 1707, 1647, 1595, 1482, 1451, 1398, 1236, 1184, 1038, 837, 754, 704 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 3.34 (s, 3H), 3.67 (s, 3H), 6.15 (s, 1H), 7.05-7.14 (m, 2H), 7.30-7.45 (m, 2H), 7.39 (d, J = 8.2 Hz, 1H), 7.44-7.47 (m, 1H), 7.52-7.56 (m, 1H), 7.83 (t, J = 7.2 Hz, 2H), 8.22 (d, J = 8.4 Hz, 1H). Anal. Calcd for C₂₃H₁₇ClN₂O₃: C, 68.23; H, 4.23; N, 6.92. Found: C, 68.36; H, 4.18; N, 7.01.

8,10-Dimethyl-12-(4-nitrophenyl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4e)



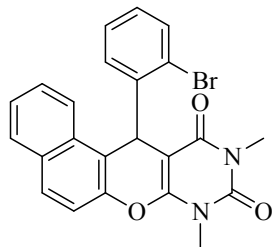
Mp 292-294 °C. (Lit. ^[1] 291-293 °C) IR (KBr): ν_{\max} = 3104, 2955, 2919, 1709, 1642, 1594, 1458, 1399, 1230, 1180, 823, 745. ¹H NMR (400 MHz, CDCl₃): δ = 3.36 (s, 3H), 3.65 (s, 3H), 5.93 (s, 1H), 7.47-7.51 (m, 3H), 7.57 (d, J = 8.8 Hz, 3H), 7.82-7.89 (m, 2H), 7.93 (d, J = 8.8 Hz, 1H), 8.09 (d, J = 8.8 Hz, 2H). Anal. Calcd for C₂₃H₁₇N₃O₅: C, 66.50; H, 4.12; N, 10.12. Found: C, 66.65; H, 4.18; N, 10.21.

3-(8,10-Dimethyl-9,11-dioxo-9,10,11,12-tetrahydro-8H-benzo[5,6]chromeno[2,3-d]pyrimidin-12-yl)phenyl nitrate (Scheme 3, 4f)



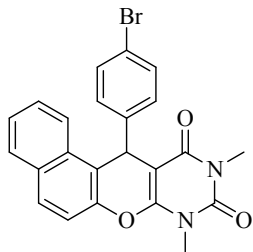
Mp 308-310 °C. (Lit. ^[2] 310-312 °C) IR (KBr): ν_{\max} = 3105, 2948, 2866, 1711, 1639, 1595, 1454, 1346, 1232, 1173, 952, 819, 754. ¹H NMR (400 MHz, CDCl₃): δ = 3.36 (s, 3H), 3.66 (s, 3H), 5.94 (s, 1H), 7.43-7.55 (m, 4H), 7.87 (t, J = 8.4 Hz, 2H), 7.93 (t, J = 8.0 Hz, 2H), 8.01 (d, J = 8.0 Hz, 1H), 8.06 (s, 1H). Anal. Calcd for C₂₃H₁₇N₃O₆: C, 64.04; H, 3.97; N, 9.74. Found: C, 63.93; H, 4.01; N, 9.62.

12-(2-Bromophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4g)



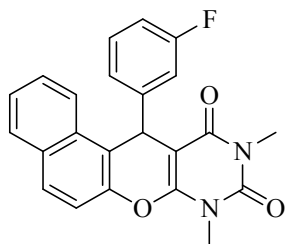
Mp 216-218 °C. (Lit. ^[1] 217-219 °C) IR (KBr): ν_{\max} = 3050, 2950, 2873, 1707, 1650, 1595, 1455, 1398, 1235, 1182, 828, 753. ¹H NMR (400 MHz, CDCl₃): δ = 3.34 (s, 3H), 3.65 (s, 3H), 6.09 (s, 1H), 6.94-6.98 (m, 1H), 7.10-7.14 (m, 1H), 7.23 (d, J = 7.2 Hz, 1H), 7.37 (d, J = 8.8 Hz, 1H), 7.42-7.46 (m, 1H), 7.51-7.55 (m, 2H), 7.82 (t, J = 8.4 Hz, 2H), 8.29 (d, J = 8.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.33, 151.97, 150.17, 146.14, 142.56, 133.01, 131.20, 131.14, 130.80, 129.37, 128.05, 127.80, 127.27, 127.07, 125.08, 124.09, 123.14, 116.88, 115.87, 90.41, 35.99, 28.68, 27.78. Anal. Calcd for C₂₃H₁₇BrN₂O₃: C, 61.48; H, 3.81; N, 6.23. Found: C, 61.33; H, 3.83; N, 6.15.

12-(4-Bromophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4h)



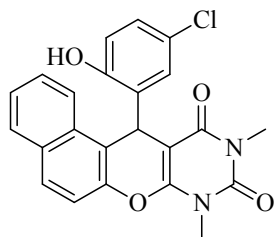
Mp 243-245 °C. (Lit. ^[1] 243-245 °C) IR (KBr): ν_{\max} = 3065, 2921, 2852, 1706, 1644, 1593, 1483, 1453, 1398, 1229, 1181, 961, 817, 746. ¹H NMR (400 MHz, CDCl₃): δ = 3.26 (s, 3H), 3.53 (s, 3H), 5.69 (s, 1H), 7.15-7.18 (m, 2H), 7.23-7.26 (m, 2H), 7.33-7.42 (m, 3H), 7.74-7.80 (m, 3H). Anal. Calcd for C₂₃H₁₇BrN₂O₃: C, 61.48; H, 3.81; N, 6.23. Found: C, 61.35; H, 3.86; N, 6.14.

12-(3-Fluorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4i)



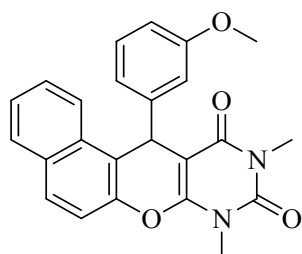
Mp 211-212 °C. (Lit. ^[3] 208-211 °C) IR (KBr): ν_{\max} = 3047, 2953, 2852, 1711, 1645, 1588, 1483, 1452, 1399, 1231, 1176, 946, 815, 748. ¹H NMR (400 MHz, CDCl₃): δ = 3.36 (s, 3H), 3.62 (s, 3H), 5.82 (s, 1H), 6.79-6.84 (m, 1H), 7.01 (dt, ¹ J = 10 Hz, ² J = 2 Hz, 1H), 7.17-7.26 (m, 2H), 7.41 (d, J = 8.8 Hz, 1H), 7.44-7.51 (m, 2H), 7.83-7.88 (m, 2H), 7.92 (d, J = 8.4 Hz, 1H). Anal. Calcd for C₂₃H₁₇FN₂O₃: C, 71.13; H, 4.41; N, 7.21. Found: C, 70.98; H, 4.43; N, 7.13.

12-(5-Chloro-2-hydroxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4j)



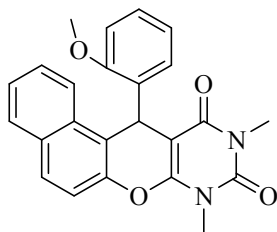
Mp 214-216 °C. IR (KBr): ν_{\max} = 3056, 2923, 2853, 1703, 1624, 1592, 1493, 1433, 1375, 1233, 1176, 959, 811, 746. ¹H NMR (400 MHz, CDCl₃): δ = 3.41 (s, 3H), 3.66 (s, 3H), 5.97 (s, 1H), 6.49 (s, 1H), 7.01-7.02 (m, 2H), 7.43 (d, J = 8.0 Hz, 1H), 7.45-7.49 (m, 2H), 7.63-7.67 (m, 2H), 7.84-7.88 (m, 1H), 7.91 (d, J = 8.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ = 164.40, 152.96, 151.78, 149.39, 146.95, 133.38, 131.41, 130.05, 129.71, 128.92, 128.08, 128.01, 127.70, 127.63, 125.79, 125.52, 123.08, 120.57, 115.80, 115.54, 89.88, 29.22, 28.90, 28.27. Anal. Calcd for C₂₃H₁₇ClN₂O₄: C, 65.64; H, 4.07; N, 6.66. Found: C, 65.53; H, 4.11; N, 6.54.

12-(3-Methoxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4k)



Mp 283-285 °C. (Lit. [2] 284-286 °C) IR (KBr): ν_{\max} = 3046, 2955, 2920, 1707, 1647, 1580, 1458, 1397, 1234, 1174, 815, 745. ¹H NMR (400 MHz, CDCl₃): δ = 3.27 (s, 3H), 3.53 (s, 3H), 3.65 (s, 3H), 5.73 (s, 1H), 6.59 (dd, ¹ J = 8.0 Hz, ² J = 2.0 Hz, 1H), 6.83 (s, 1H), 6.94 (d, J = 8.0 Hz, 1H), 7.08 (t, J = 8.0 Hz, 1H), 7.31-7.42 (m, 3H), 7.76 (t, J = 8.0 Hz, 2H), 7.91 (d, J = 8.0 Hz, 1H). Anal. Calcd for C₂₄H₂₀N₂O₄: C, 71.99; H, 5.03; N, 7.00. Found: C, 71.86; H, 5.09; N, 6.92.

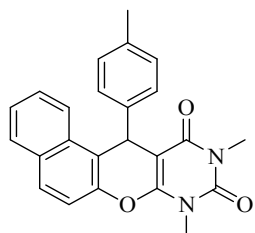
12-(2-Methoxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4l)



Mp 292-295 °C. (Lit. [2] 295-297 °C) IR (KBr): ν_{\max} = 3064, 2994, 2934, 2829, 1709, 1671, 1595, 1487, 1455, 1396, 1231, 1182, 1025, 809, 748. ¹H NMR (400 MHz, CDCl₃): δ = 3.33 (s, 3H), 3.67 (s, 3H), 3.88 (s, 3H), 6.06 (s, 1H), 6.83-6.89 (m, 2H), 7.11 (td, ¹ J = 8.0 Hz, ² J = 2.0 Hz, 1H), 7.35-7.44 (m, 3H), 7.47-

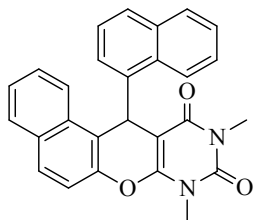
7.52 (m, 1H), 7.78-7.81 (m, 2H), 8.26 (d, $J = 8.4$ Hz, 1H). Anal. Calcd for $C_{24}H_{20}N_2O_4$: C, 71.99; H, 5.03; N, 7.00. Found: C, 72.08; H, 4.98; N, 7.07.

8,10-Dimethyl-12-(p-tolyl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4m)



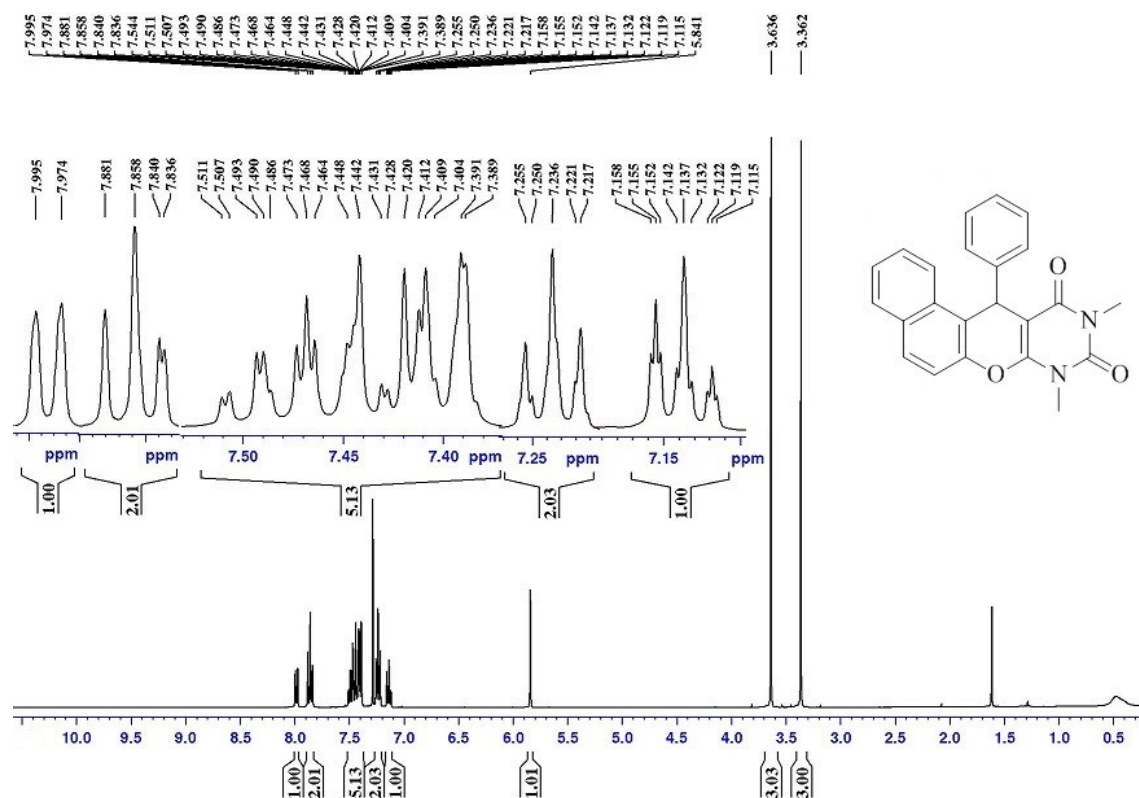
Mp 201-203 °C. (Lit. ^[1] 200-202 °C) IR (KBr): $\nu_{\max} = 3053, 2950, 2860, 1703, 1662, 1594, 1486, 1453, 1369, 1231, 1180, 1045, 811$. $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 2.14$ (s, 3H), 3.25 (s, 3H), 3.53 (s, 3H), 5.70 (s, 1H), 6.94 (d, $J = 8.0$ Hz, 2H), 7.17 (s, 1H), 7.18 (s, 1H), 7.31-7.41 (m, 3H), 7.73-7.77 (m, 2H), 7.89 (d, $J = 8.0$ Hz, 1H). Anal. Calcd for $C_{24}H_{20}N_2O_3$: C, 77.13; H, 4.79; N, 6.66. Found: C, 76.87; H, 4.83; N, 6.59.

8,10-Dimethyl-12-(naphthalen-1-yl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4n)

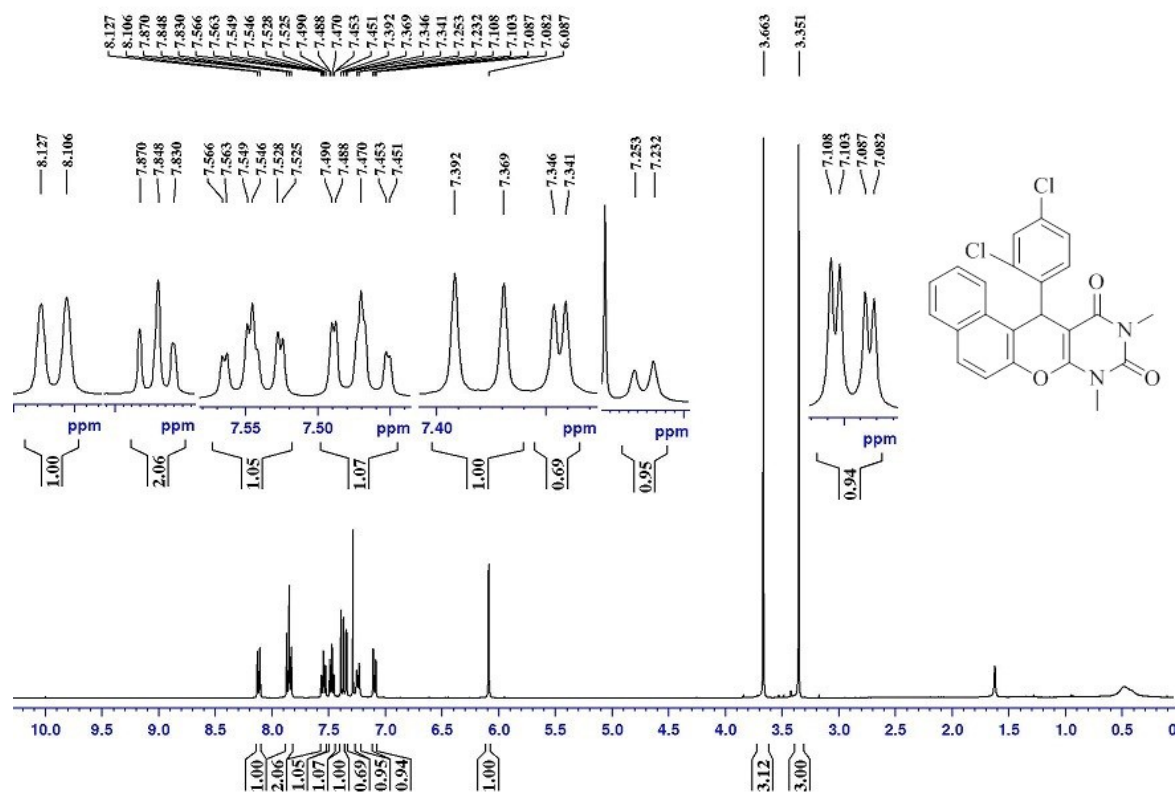


Mp 205-207 °C. IR (KBr): $\nu_{\max} = 3053, 2954, 2922, 2850, 1708, 1652, 1596, 1485, 1455, 1373, 1230, 1172, 1073, 951, 781$. $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 3.18$ (s, 3H), 3.61 (s, 3H), 6.51 (s, 1H), 7.16-7.17 (m, 3H), 7.27 (t, $J = 7.2$ Hz, 1H), 7.38 (d, $J = 8.8$ Hz, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 7.54-7.57 (m, 1H), 7.69-7.76 (m, 5H), 7.81 (d, $J = 8.4$ Hz, 1H), 9.10 (d, $J = 7.2$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 166.52, 162.20, 152.50, 147.02, 133.57, 131.71, 131.30, 131.00, 129.35, 128.75, 128.50, 127.67, 127.56, 126.59, 125.74, 125.47, 124.27, 123.87, 116.47, 77.23, 31.40, 29.20, 28.28$. Anal. Calcd for $C_{27}H_{20}N_2O_3$: C, 77.13; H, 4.79; N, 6.66. Found: C, 76.94; H, 4.86; N, 6.55.

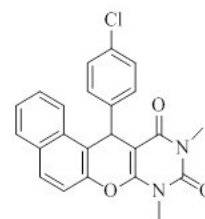
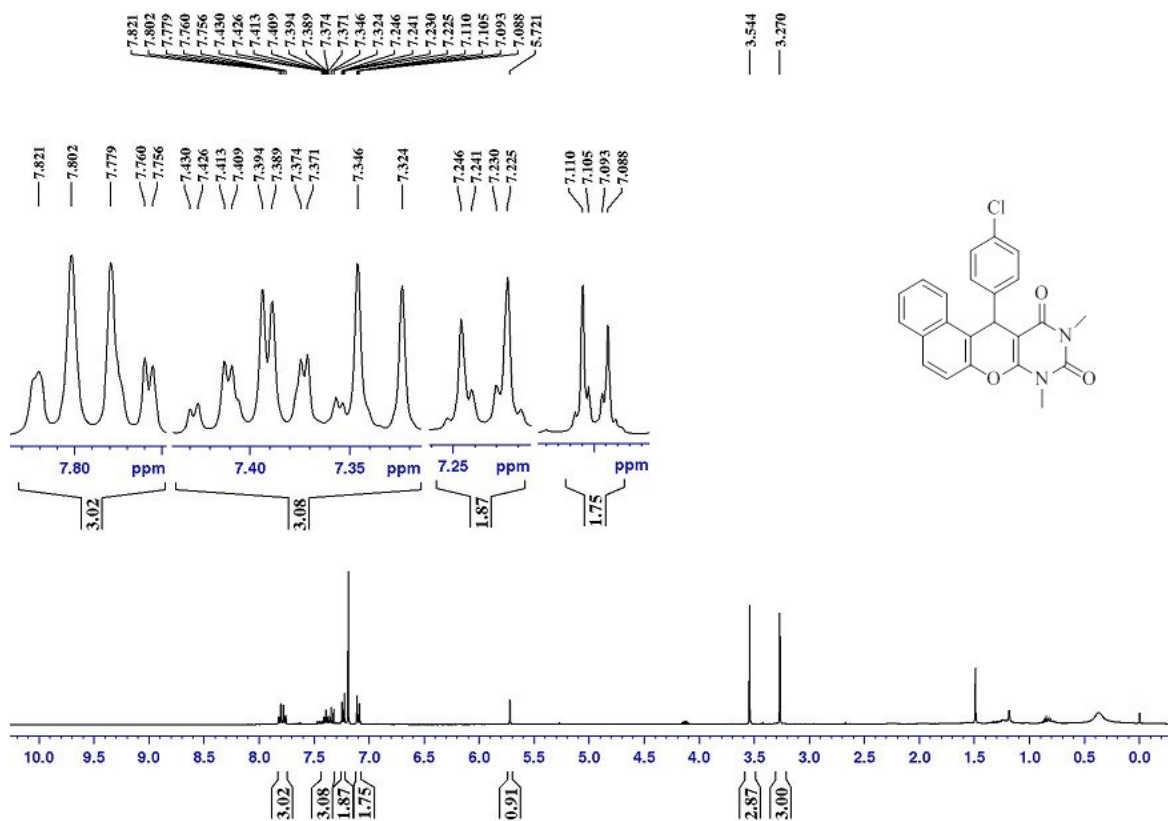
5. ^1H NMR and ^{13}C NMR Spectra of the Products:



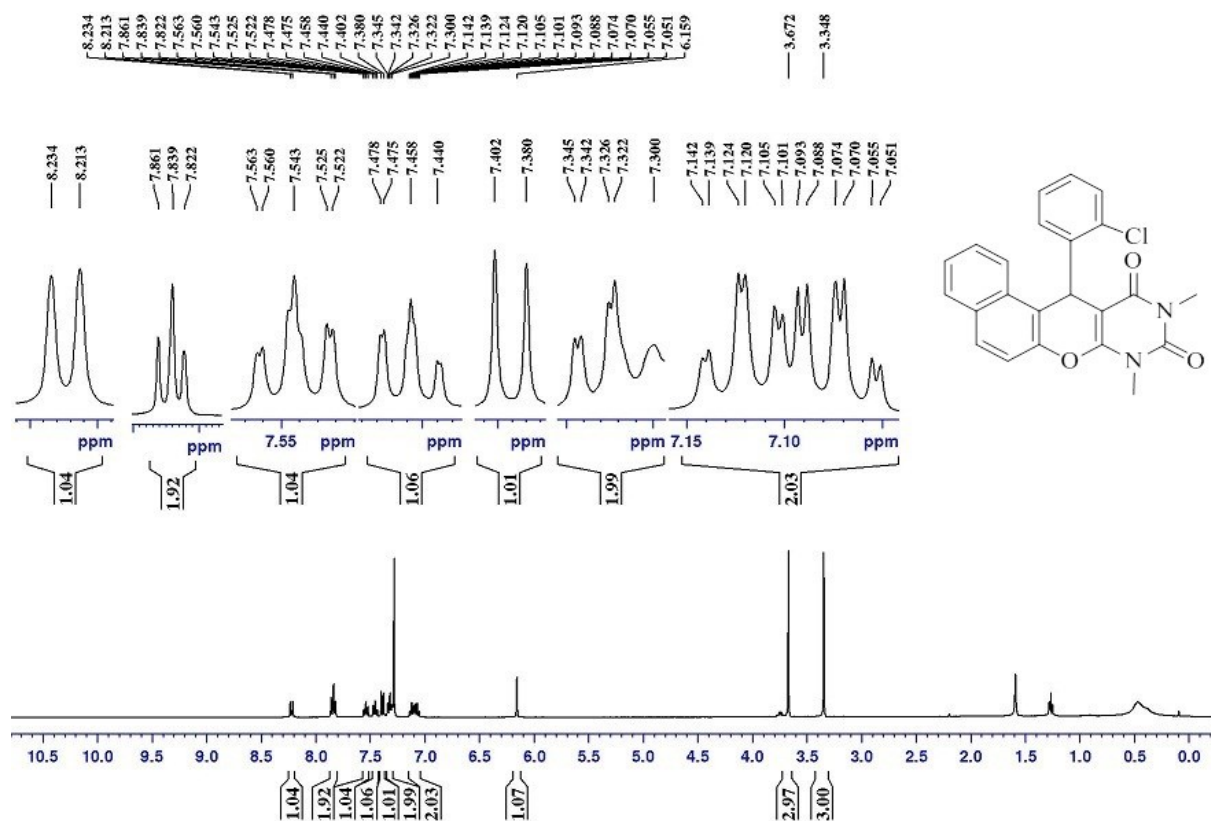
8,10-Dimethyl-12-phenyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4a); ^1H NMR (400 MHz, CDCl_3)



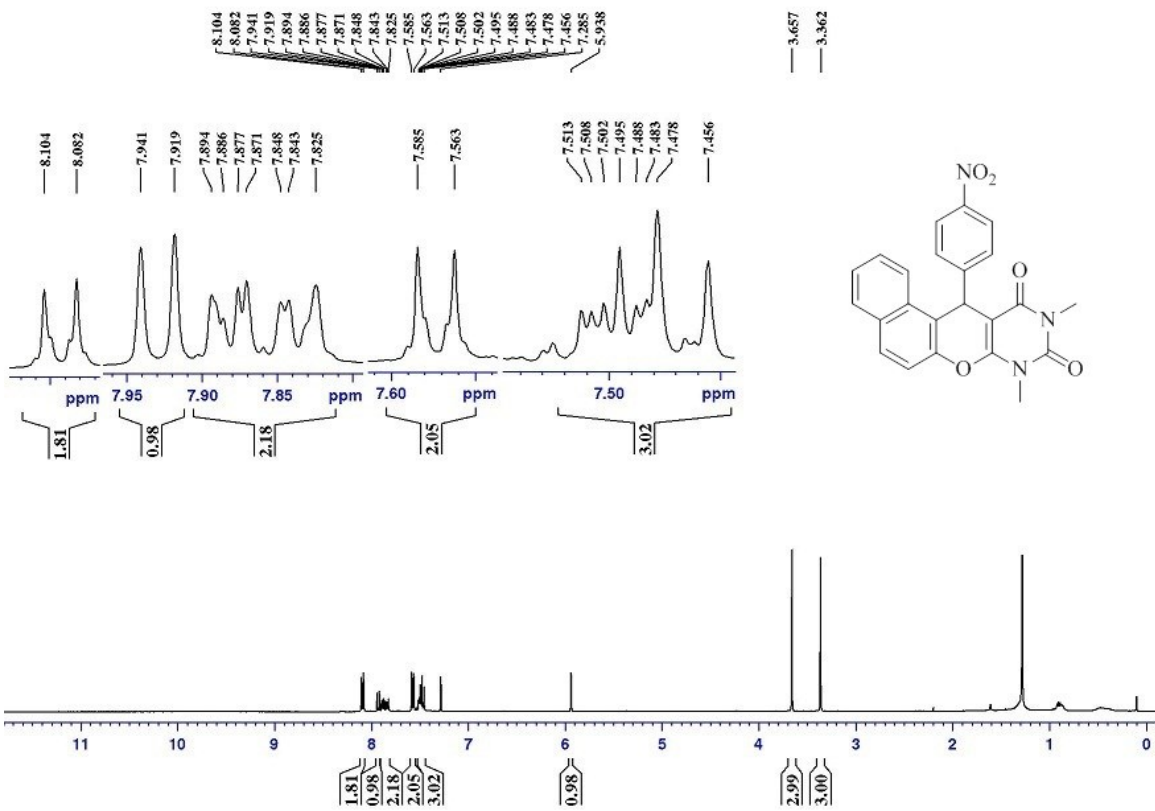
12-(2,4-Dichlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4b); ¹H NMR (400 MHz, CDCl₃)



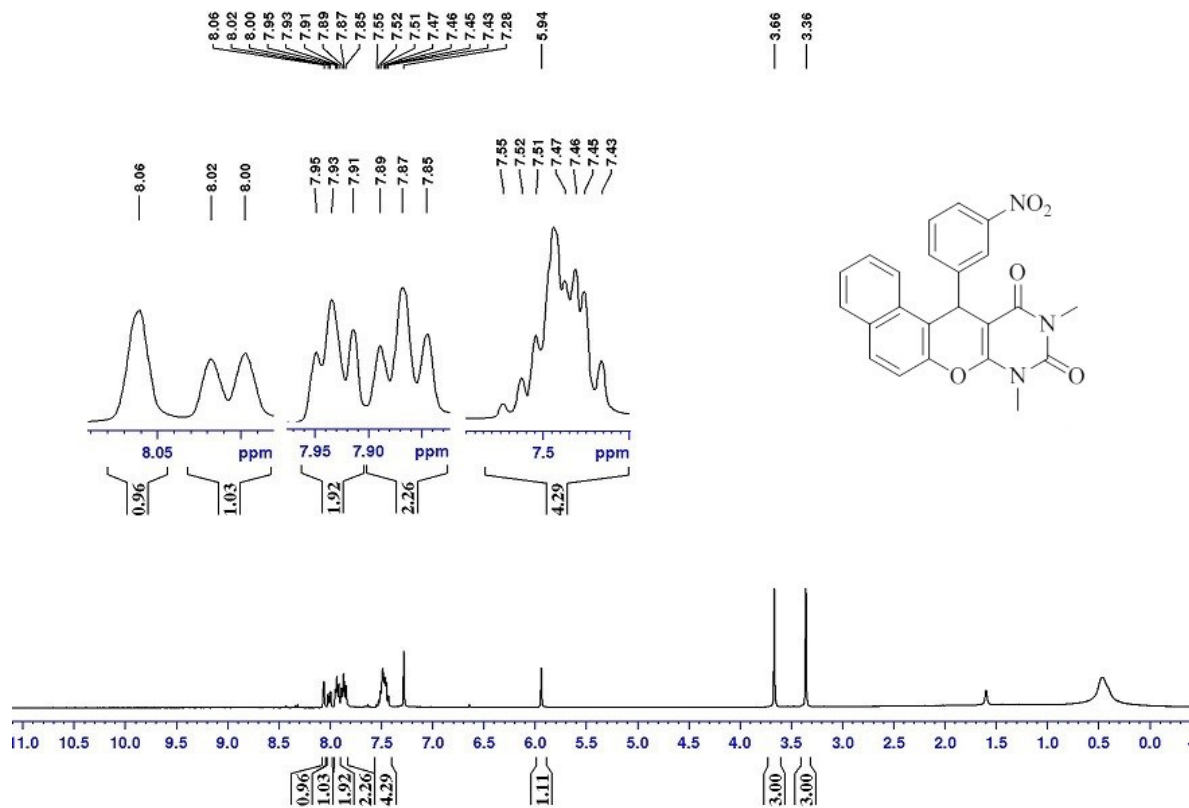
12-(4-Chlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4c); ^1H NMR (400 MHz, CDCl_3)



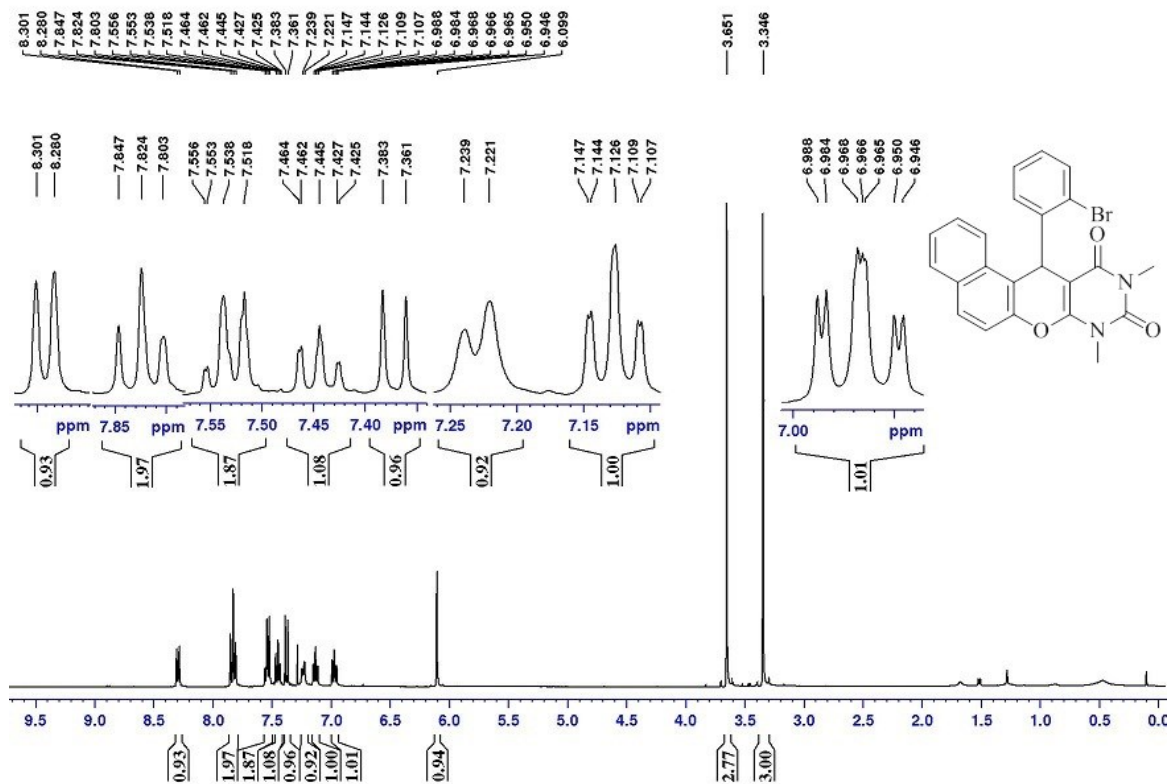
12-(2-Chlorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4d); ¹H NMR (400 MHz, CDCl₃)



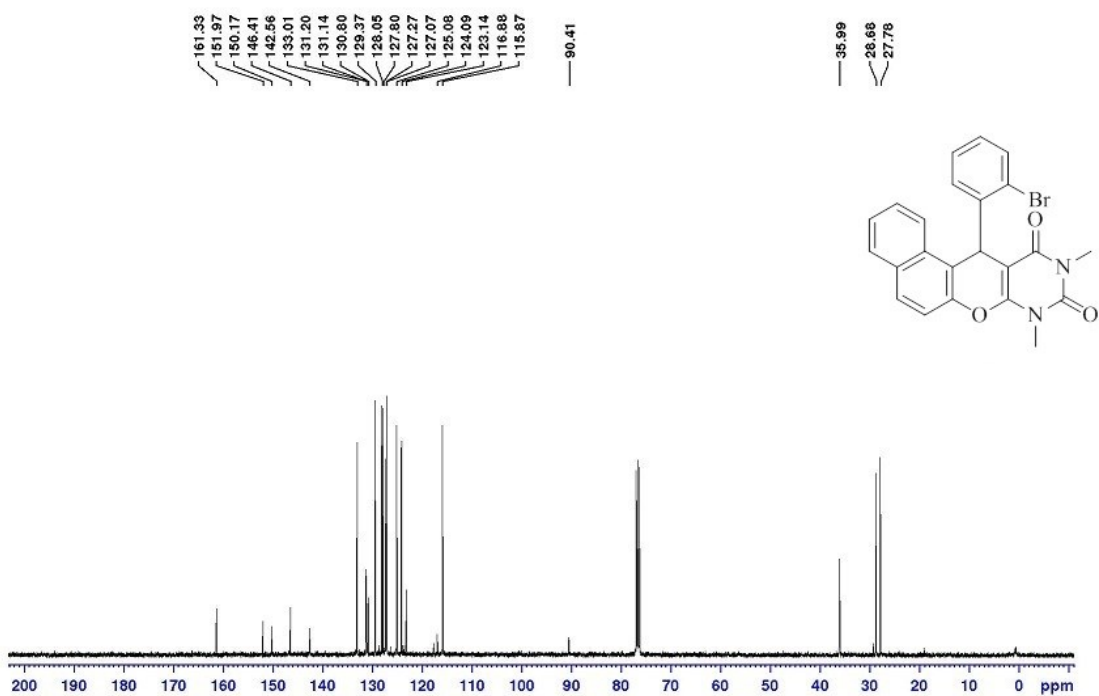
8,10-Dimethyl-12-(4-nitrophenyl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4e); ¹H NMR (400 MHz, CDCl₃)



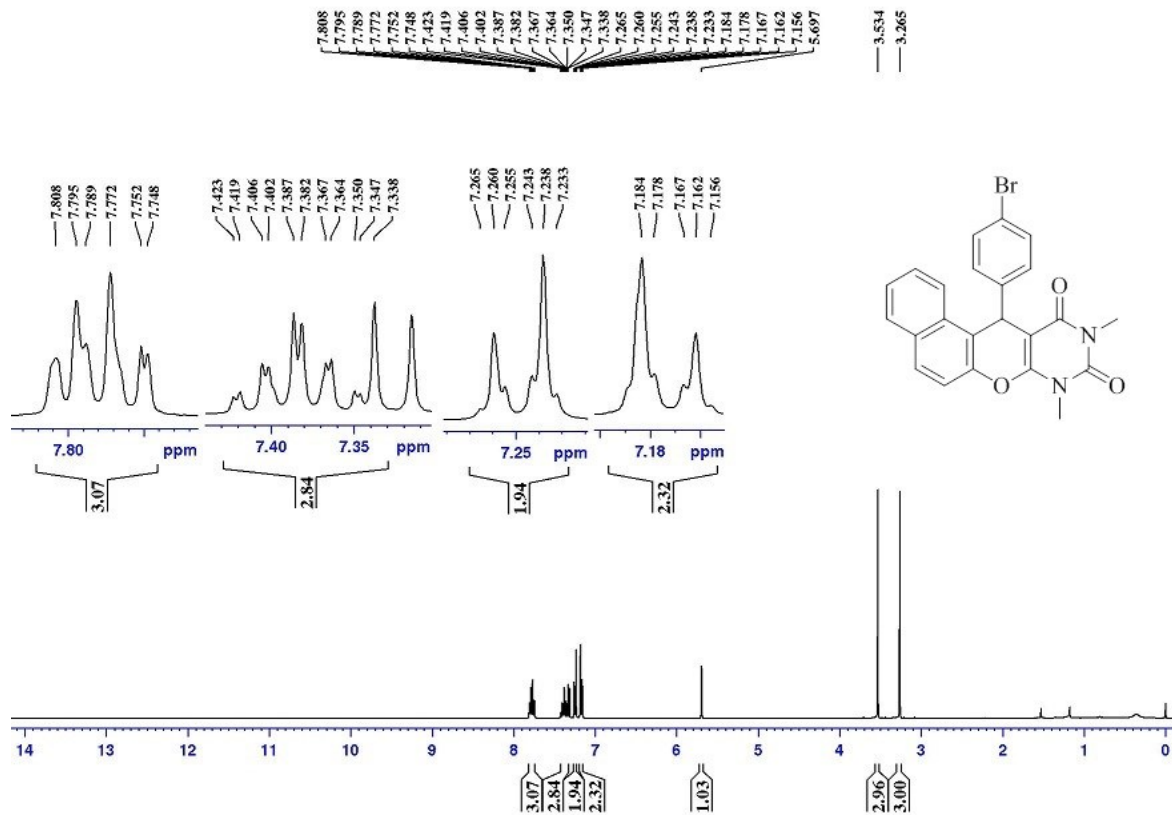
3-(8,10-Dimethyl-9,11-dioxo-9,10,11,12-tetrahydro-8H-benzo[5,6]chromeno[2,3-d]pyrimidin-12-yl)phenyl nitrate (Scheme 3, 4f); ¹H NMR (400 MHz, CDCl₃)



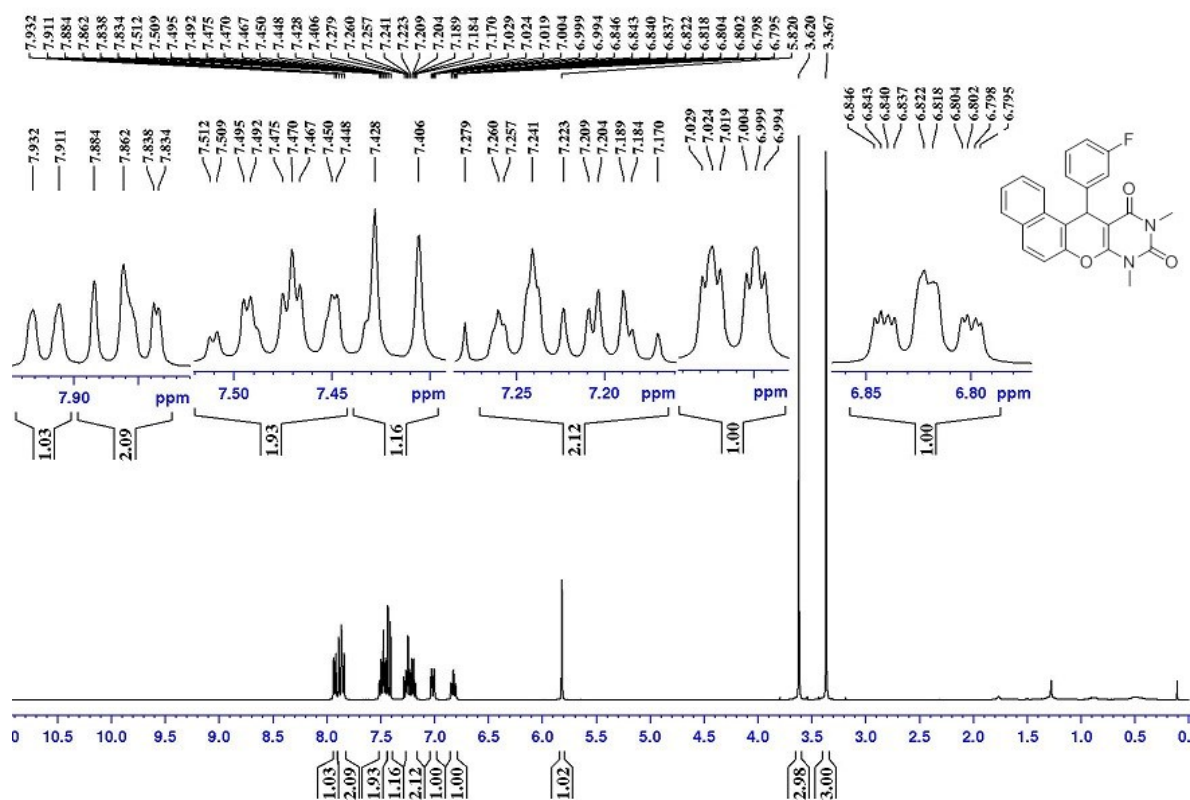
12-(2-Bromophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4g); ¹H NMR (400 MHz, CDCl₃)



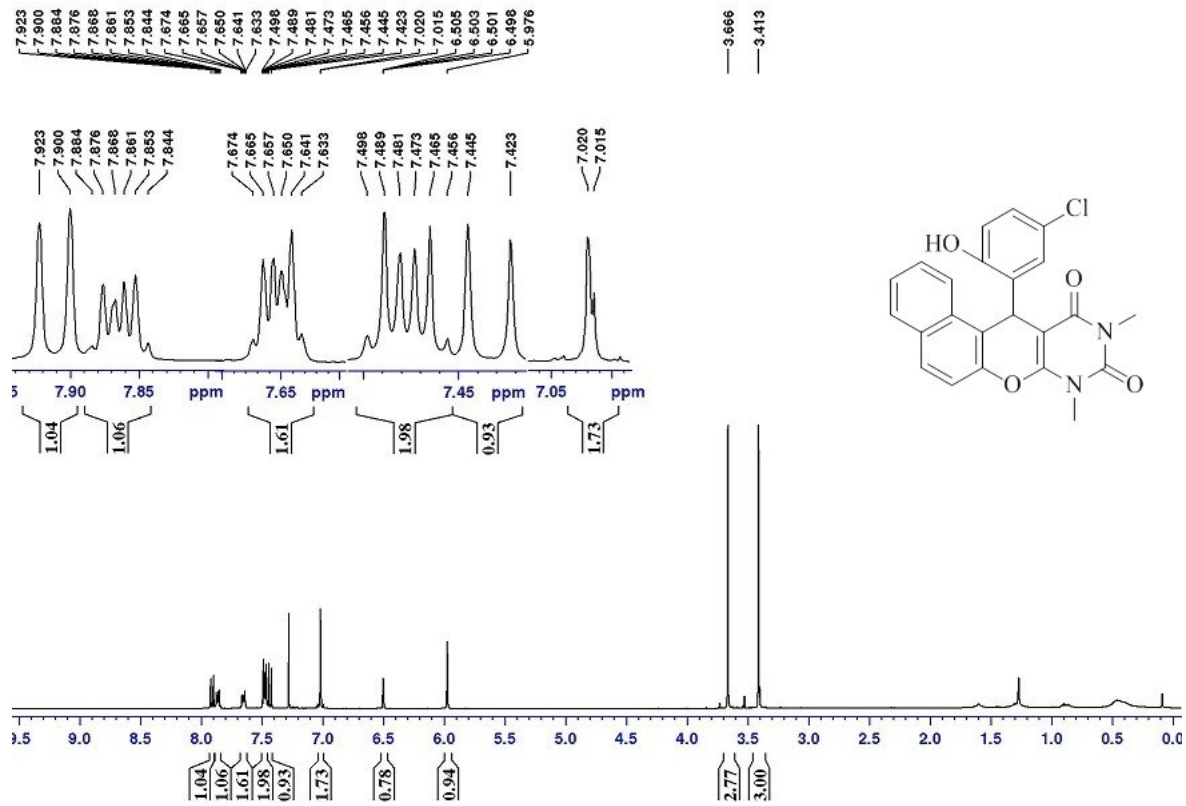
12-(2-Bromophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4g); ¹³C NMR (100 MHz, CDCl₃)



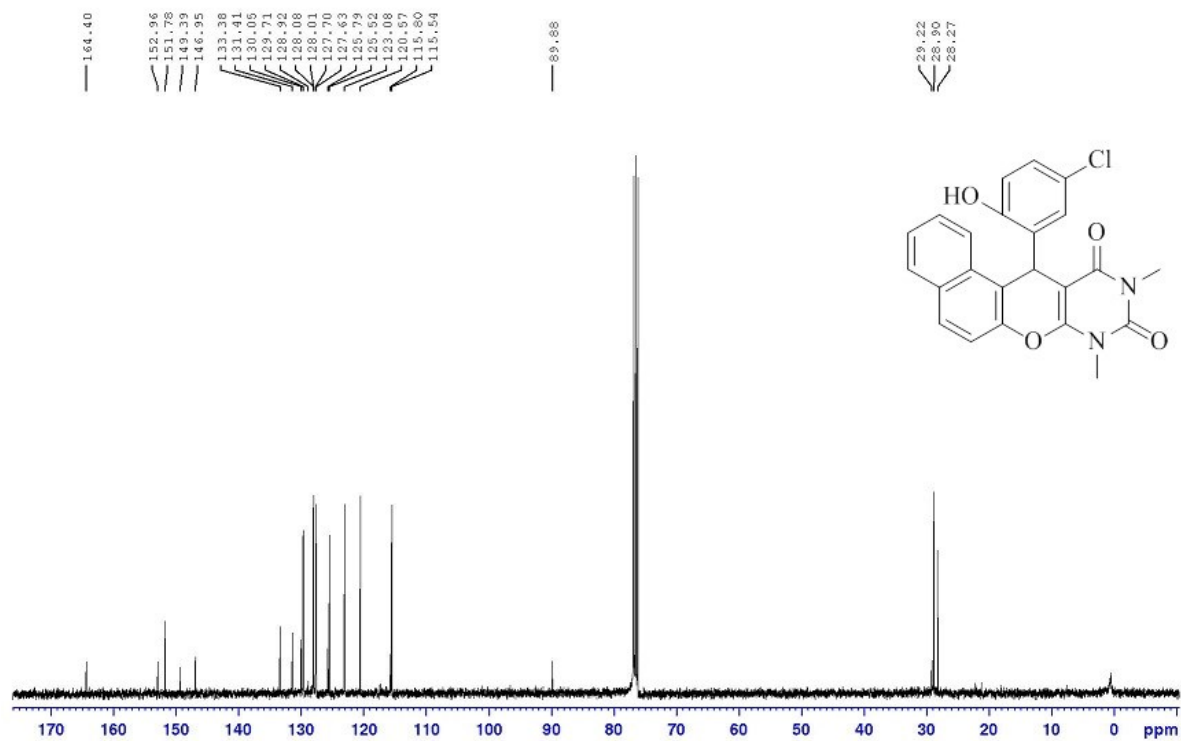
12-(4-Bromophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4h); ¹H NMR (400 MHz, CDCl₃)



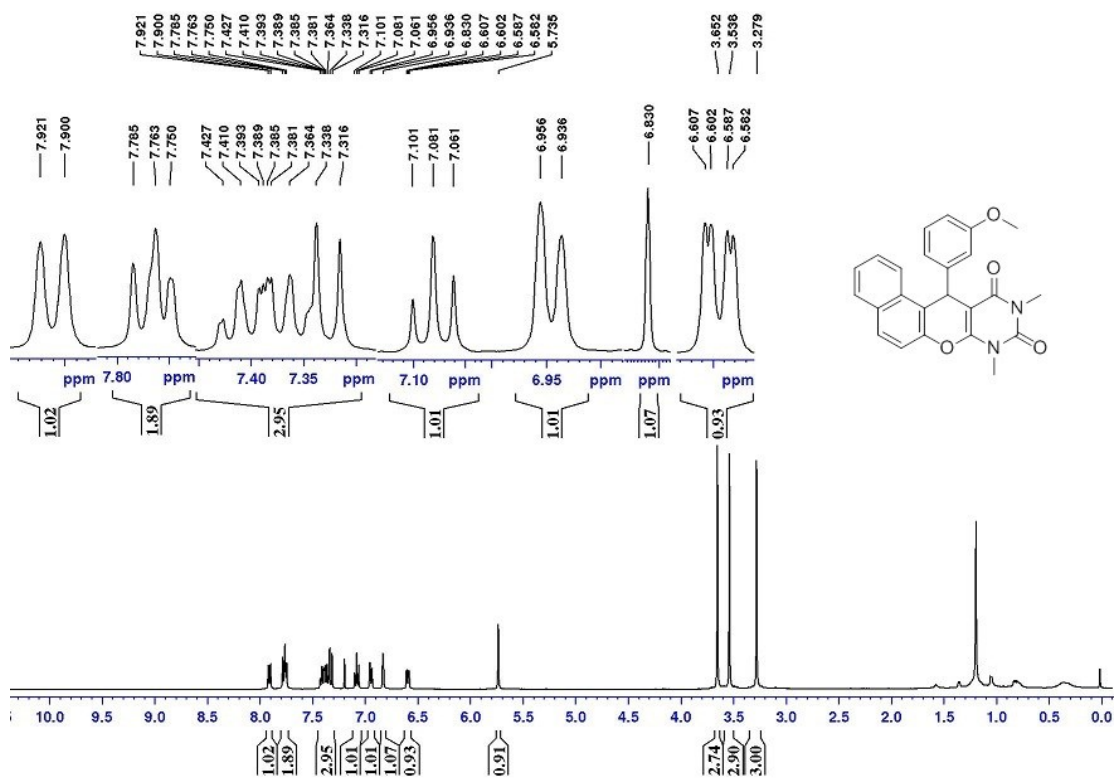
12-(3-Fluorophenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4i); ¹H NMR (400 MHz, CDCl₃)



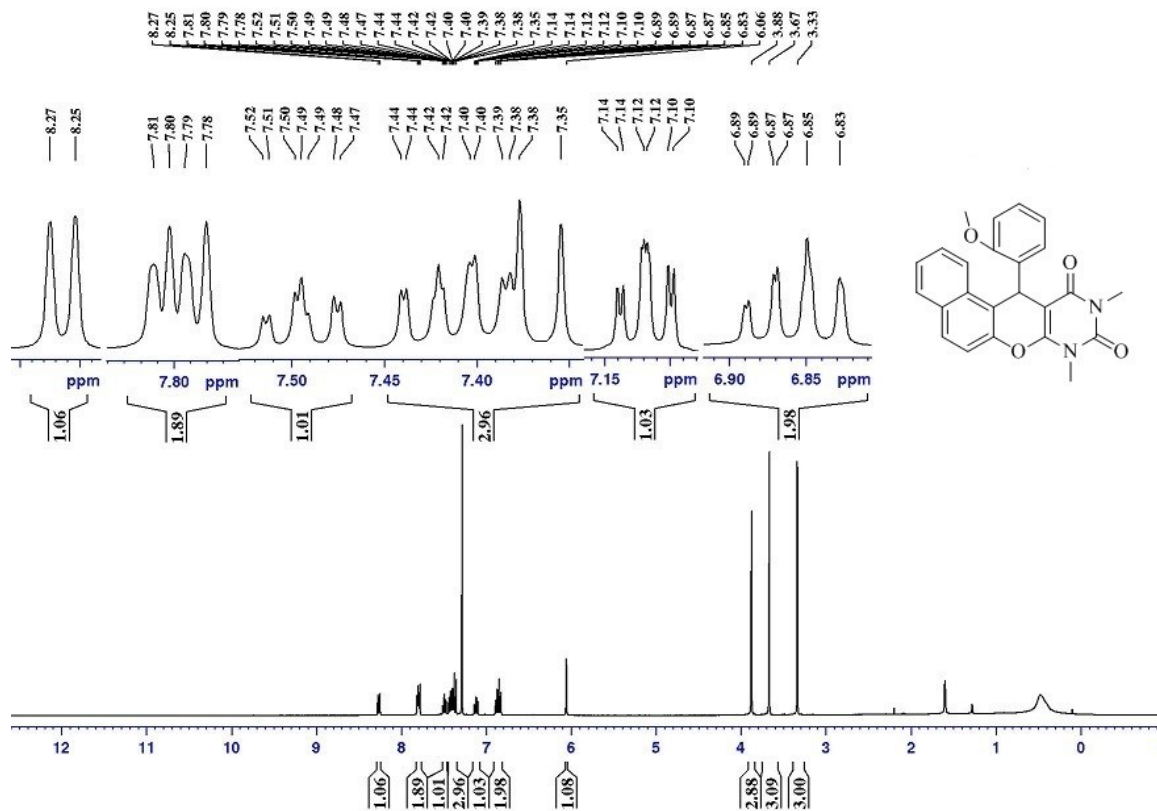
12-(5-Chloro-2-hydroxyphenyl)-8,10-dimethyl-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione (Scheme 3, 4j); ¹H NMR (400 MHz, CDCl₃)



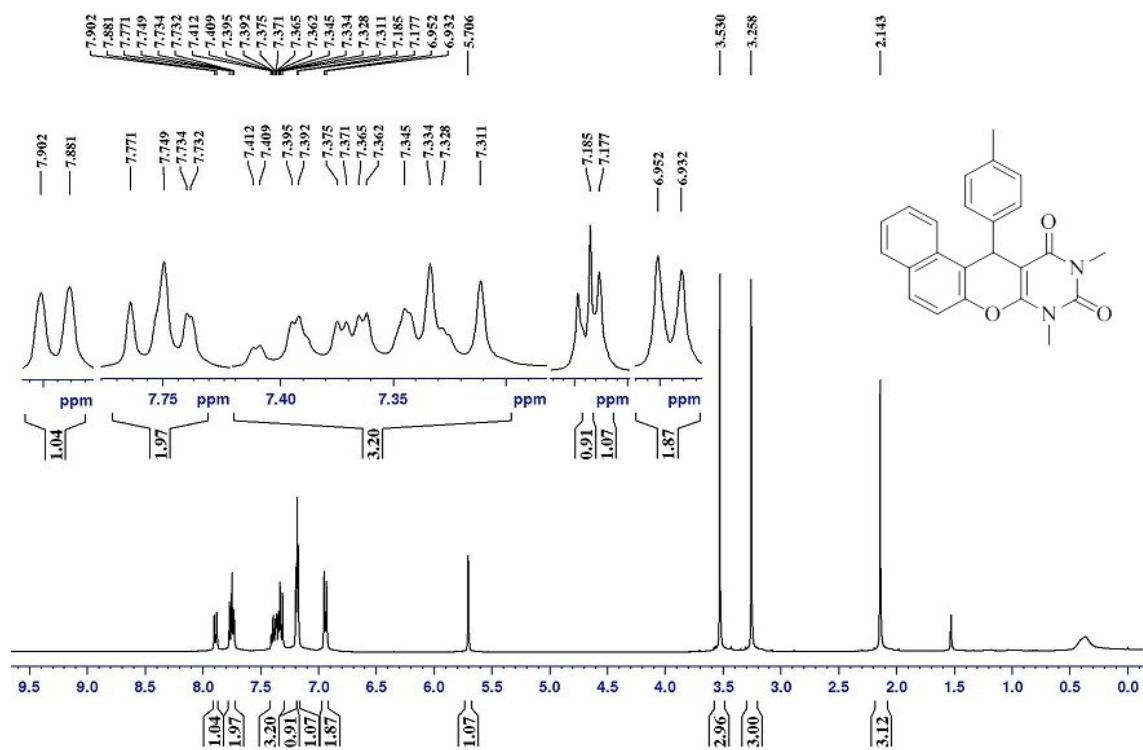
12-(5-Chloro-2-hydroxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4j); ^{13}C NMR (100 MHz, CDCl_3)



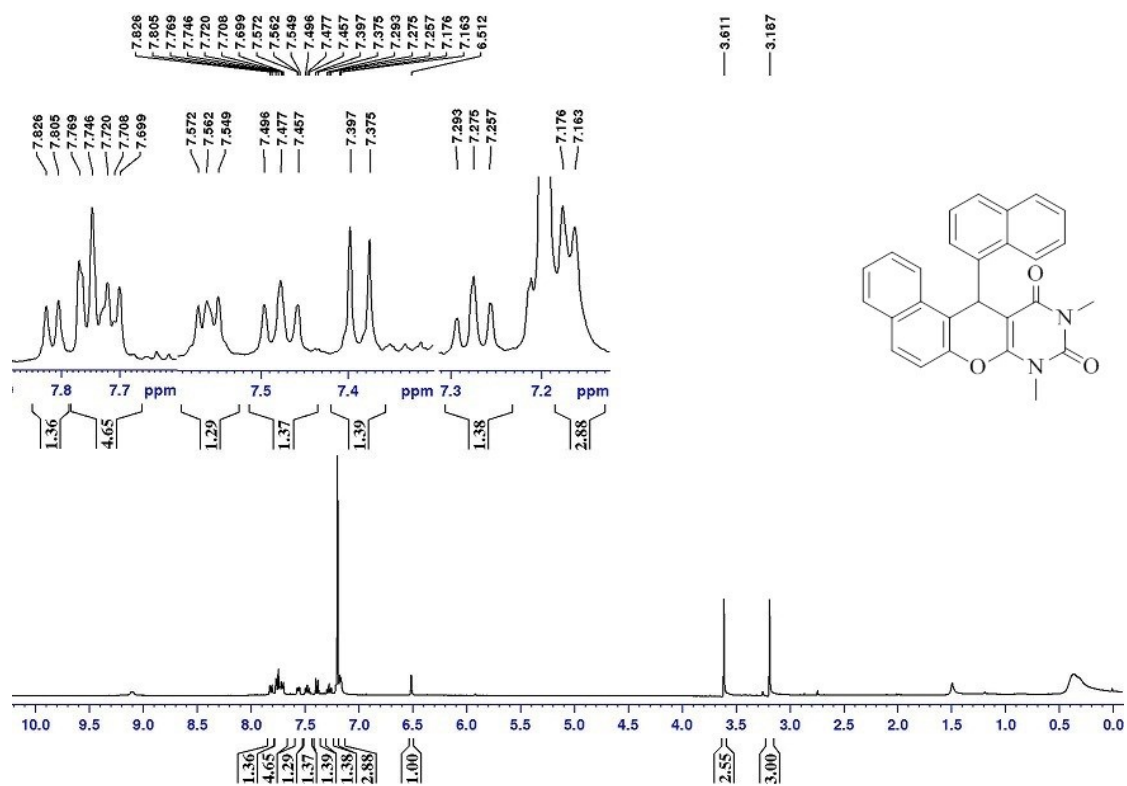
12-(3-Methoxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4k); ¹H NMR (400 MHz, CDCl₃)



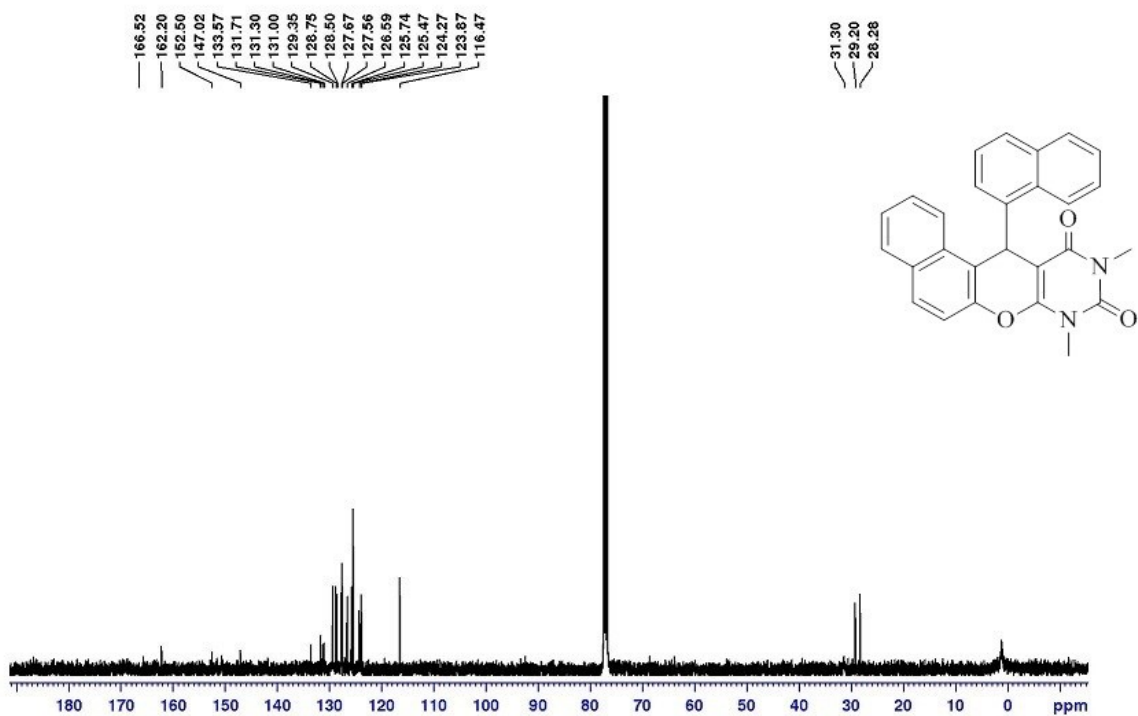
12-(2-Methoxyphenyl)-8,10-dimethyl-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4l); ¹H NMR (400 MHz, CDCl₃)



8,10-Dimethyl-12-(p-tolyl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4m); ¹H NMR (400 MHz, CDCl₃)



8,10-Dimethyl-12-(naphthalen-1-yl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione (Scheme 3, 4n); ¹H NMR (400 MHz, CDCl₃)



8,10-Dimethyl-12-(naphthalen-1-yl)-8H-benzo[5,6]chromeno[2,3-d]pyrimidine-9,11(10H,12H)-dione
 (Scheme 3, 4n); ¹³C NMR (100 MHz, CDCl₃)

6. References

- [1] K. P. Kumar, S. Satyanarayana, P. L. Reddy, G. Narasimhulu, N. Ravirala, and B. S. Reddy, *Tetrahedron Lett.*, 2012, **53**, 1738-1741.
- [2] M. Mohaqeq, J. Safaei-Ghomi, H. Shahbazi-Alavi, and R. Teymuri, *Polycycl. Aromat. Comp.*, 2017, **37**, 52-62.
- [3] M. Fatahpour, N. Hazeri, M. T. Maghsoodlou, and M. Lashkari, *Polycycl. Aromat. Comp.*, 2017, **39**, 311-317.