# EFFICIENT SYNTHESIS OF DIHYDROCHROMENO-[4,3-*b*]CHROMENONE DERIVATIVES IN AQUEOUS MEDIA

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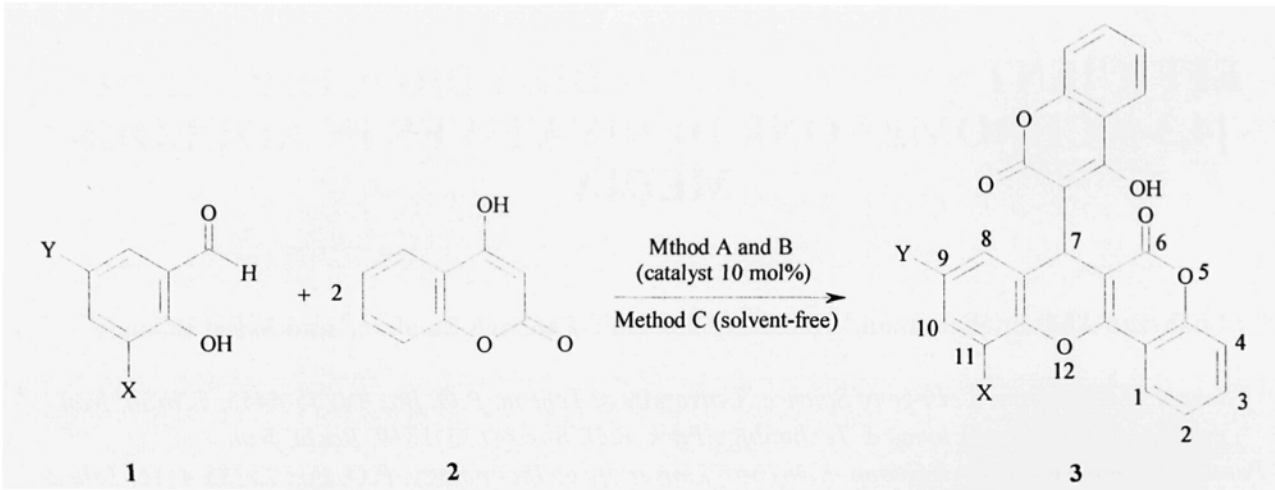
**Abstract**

Dihydrochromeno[4,3-b]chromenone derivatives were synthesized successfully via a pseudo three-component reaction of salicylaldehyde derivatives with 4-hydroxycoumarin in a ratio of 1:2 respectively in aqueous media catalyzed by diammonium hydrogen phosphate (DAHP) or S-proline at room temperature. This later modified by a solvent-free approach without catalyst at 80°C. These are the first reports on the synthesis of dihydrochromeno[4,3-b]chromenones by these procedures. The application of these methods to corresponding products are highlighted that can be adopted for simple operation, easy work-up, high yields, high-speed (specially under solvent-free approach) and environmentally benign procedure.

**Introduction**

The chromene framework is a structural feature of a vast number of synthetic biologically active compounds.[1,2] Chromene is an important class of compounds, which is found to possess some biological activities such as potassium channel opening and hypotensive,[3-6] Vasodilator and antihypertensive, β-adrenolytic[7] and antimicrobial[8] activities. Moreover substituted 4*H*-chromenes have been identified as a new class of anticancer compounds.[9] These facts called us to describe the efficient methods to this class of compounds. In this context, we have described the simple procedures for the preparation of 7-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-6*H*,7*H*-chromeno[4,3-*b*]chromen-6-ones **3a-h** in aqueous media or under solvent-free conditions.




1 2 3

| | X | Y |
|---|---|---|
| **a** | H | H |
| **b** | H | Br |
| **c** | Br | Br |
| **d** | Br | Cl |
| **e** | Cl | Cl |
| **f** | $OCH_3$ | H |
| **g** | H | $CH_3$ |
| **h** | H | $NO_2$ |

Method: A: 10 mol% diammonium hydrogen phosphate (DAHP), $H_2O$, r.t.
B: 10 mol% (*S*)-proline, $H_2O$, r.t.
C: Solvent-free, 80 °C.

**Scheme 1**

**Results and discussion:**

In continuation of our previous works on the development of new and efficient methods for the preparation of heterocyclic compounds, herein, we report the convenient and simple synthetic routes to 7-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-6*H*,7*H*-chromeno[4,3-*b*]chromen-6-ones **3a-h**.

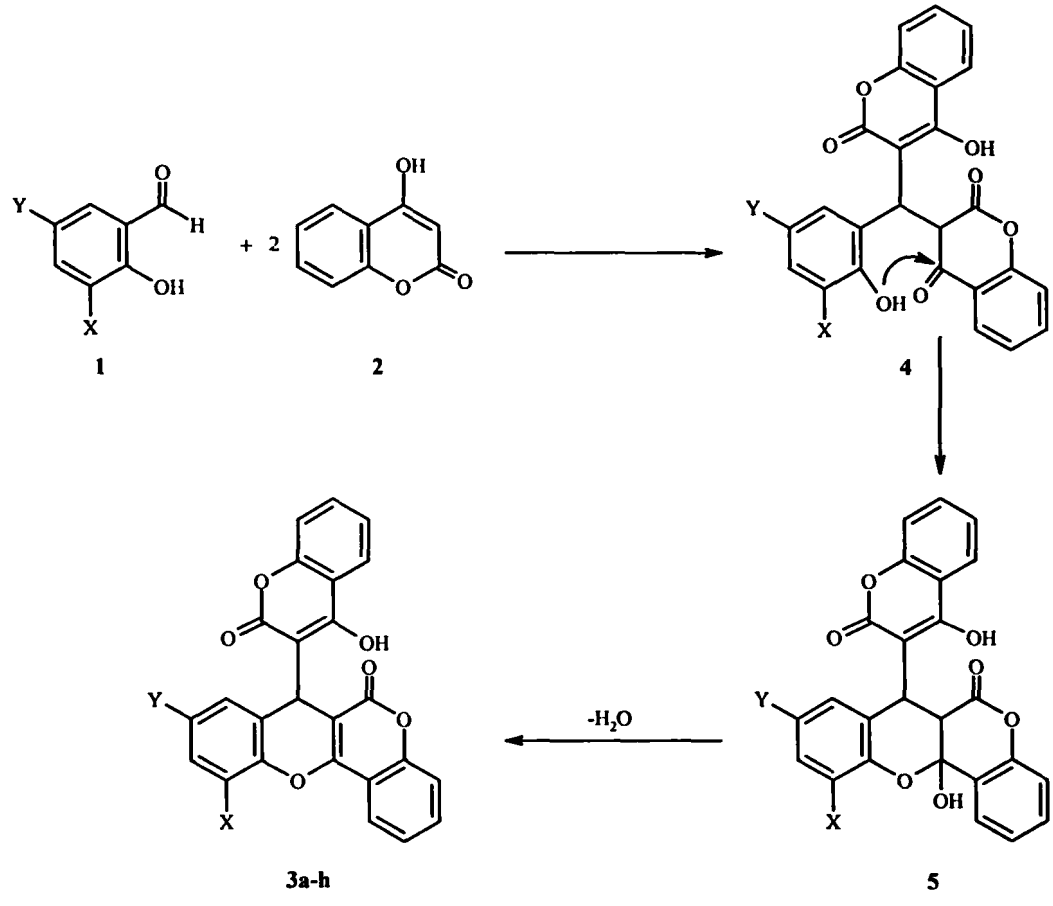
Toward this goal, a pseudo three-component reaction of salicylaldehyde derivatives with 4-hydroxycoumarin in a ratio of 1:2 respectively, in the presence of catalytic amounts (10 mol%) of DAHP as catalyst in aqueous media at room temperature, were investigated (Method **A**, **Scheme 1**). In spite of a few reports regarding the application of DAHP in organic synthesis,[10-15] we considered that DAHP to be ideal as a catalyst for this target. As shown in **Table 1**, the corresponding products **3a-h** were successfully synthesized in high yields within about 5 h.

Since recently *S*-proline was added as an efficient organocatalysts in some important organic reactions,[16] we have also used catalytic amounts (10 mol%) of *S*-proline as another efficient catalyst for this reaction in aqueous media at room temperature (Method **B**, **Scheme 1**). In this case the expected products **3a-h** were also obtained satisfactorially after about 6 h. The results are summarized in **Table 1**.

The employing solvent-free approach for this reaction was another goal of this study. When salicylaldehyde derivatives **1** and 4-hydroxycoumarin **2** in a ratio of 1:2 respectively were mixed without catalyst and under solvent-free conditions at 80°C, the corresponding products were obtained in high yields within about 2 h (Method **C**, **Scheme 1**).Various kind

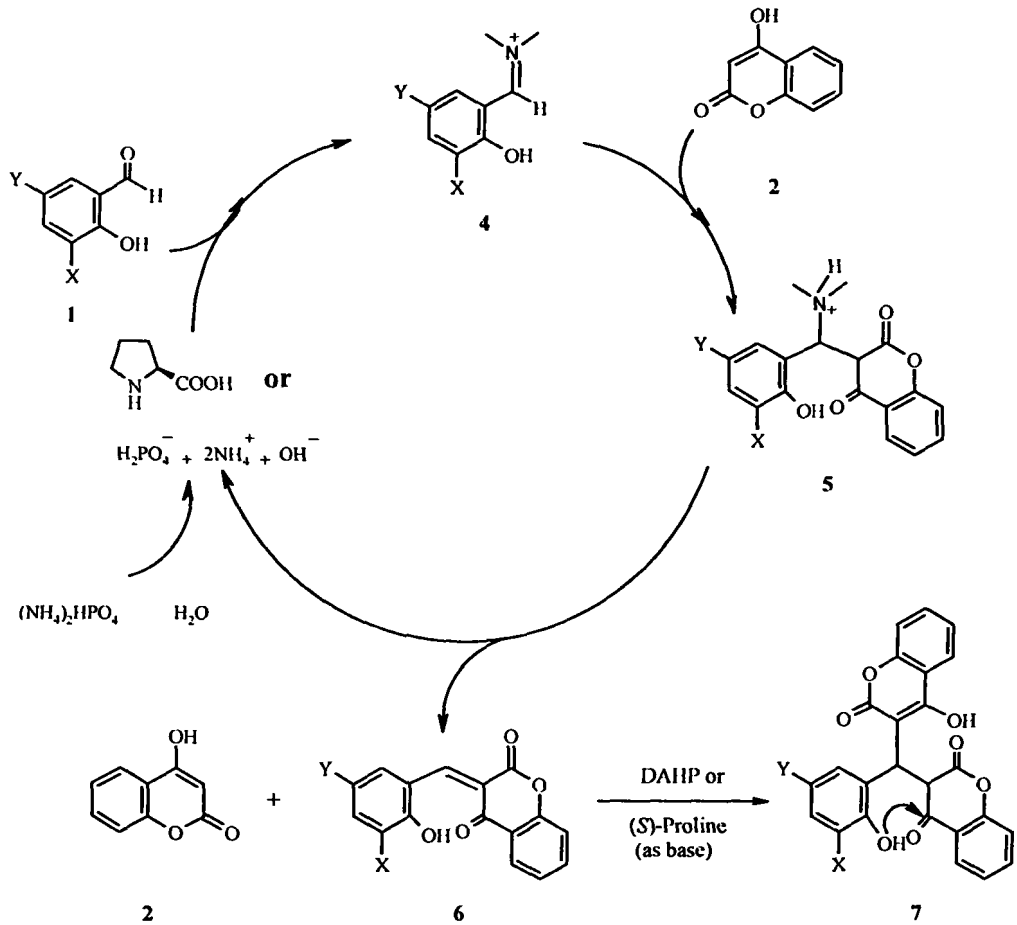
of salicylaldehyde derivatives were subjected to give the target compounds **3a-h**, and representative examples are shown in **Table 1**.

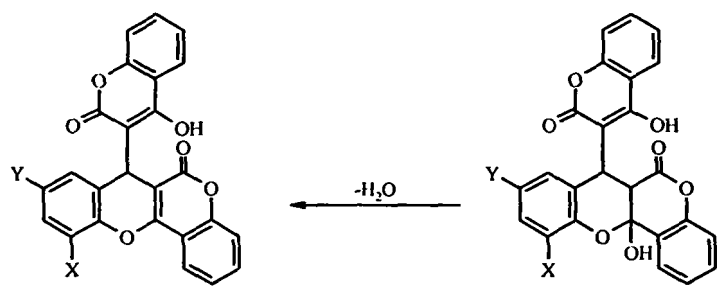
The neat pseudo three-component condensation reaction of 4-hydroxycoumarin with substituted salicylaldehydes yields unstable 3,3′-benzylidene bis(4-hydroxycoumarin) **4**, as intermediate which undergoes ring closure with the hydroxyl group of phenyl moiety, to produce chromeno[4,3-*b*]chromenone derivatives **3a-h** (**Scheme 2**).




**Scheme 1**

Although we have not established the mechanism of this reaction in the presence of catalytic amounts of DAHP or *S*-proline in aqueous media, a possible explanation is given in Scheme 3. We suggest that, DAHP or *S*-proline which possess the nitrogen heteroatom in their structure, can catalyze the formation of olefin **6** by conversion of carbonyl group of salicylaldehyde **1** to a more reactive iminium ion **4**, which reacts easily with one mole of 4-hydroxycoumarin **2** in a Knoevenagel condensation to produce olefin **6** *via* intermediate **5**. In second step DAHP or *S*-proline can also act as a mild base for the formation of intermediate **7**, readily prepared *in situ* from Michael addition of one remaining mole of 4-hydroxycoumarin **2** to olefin **6**, which generates product **3a-h**, after cyclization and hydrolysis of intermediate **8**.




| | X | Y |
|---|---|---|
| a | H | H |
| b | H | Br |
| c | Br | Br |
| d | Br | Cl |
| e | Cl | Cl |
| f | $OCH_3$ | H |
| g | H | $CH_3$ |
| h | H | $NO_2$ |

**Scheme 1**

**Table 1.** Synthesis of 7-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-6*H*,7*H*-chromeno[4, 3-*b*]chromen-6-ones **3a-h** in aqueous media using DAHP (Method **A**), *S*-proline (method **B**) as catalysts and under solvent-free conditions (Method **C**).

| product | X | Y | Mp (°C) | Yield* (%) | | |
|---|---|---|---|---|---|---|
| | | | | Method A | Method B | Method C |
| **3a** | H | H | 255-256 | 90 | 92 | 97 |
| **3b** | H | Br | 330-331 | 93 | 93 | 95 |
| **3c** | Br | Br | 208-210 | 89 | 90 | 93 |
| **3d** | Br | Cl | 204-206 | 90 | 91 | 95 |
| **3e** | Cl | Cl | 200-202 | 91 | 91 | 94 |
| **3f** | $OCH_3$ | H | 292-293 | 95 | 93 | 98 |
| **3g** | H | $CH_3$ | 328-329 | 93 | 94 | 95 |
| **3h** | H | $NO_2$ | 299-301 | 92 | 95 | 99 |

*Yields refer to pure isolated products characterized by IR, $^1$H and $^{13}$C NMR spectroscopic and Mass spectrometry data. A: the reaction was conducted in $H_2O$ using DAHP (10%) as catalyst at r.t.; B: the reaction was performed in $H_2O$ using *S*-proline as catalyst at r.t.; C: the reaction was carried out under solvent-free conditions at 80°C.

The structure of compounds **3a-h** were completely characterized by $^1$H NMR, $^{13}$C NMR and IR spectral data and their molecular weight confirmed by mass spectrometry. $^1$H NMR and $^{13}$C NMR spectroscopy especially were used to distinguish the structures of products. Thus, all of the products exhibited a singlet peak at about $\delta$ = 5.69-6.28 ppm for H-7 in the $^1$H NMR spectra, and also a distinctive signal at $\delta$ = 28.6-34.7 ppm for C-7 in the $^{13}$C NMR spectra. The mass spectra of these compounds detected the expected molecular ion signals.

In coclusion we have developed simple methods for the preparation of an important class of chromenes, which can be interested because of their usefulness as biologically active agents. In addition to their simplicity of operation, these methods have the advantages of high yields, easy work up and environmentally friendly procedure. The biologically activities of these compounds will be study.

## EXPERIMENTAL

Melting points were determined with an Electrothermal 9100 apparatus and were uncorrected. IR spectra were obtained on an ABB FT-IR (FTLA 2000) spectrometer. $^1$H NMR and $^{13}$C NMR spectra were recorded on a Bruker DRX-500 AVANCE at 500.1 and 125.8 MHz (respectively) using TMS as internal standard and DMSO-$d_6$ as solvent. ESI Mass spectra were recorded using ICR Apex-Qe instrument and EI Mass spectra data were obtained using a GC-MS Hewlett Packard instrument operating at ionization potential of 70 eV.

### General Procedure for the Preparation of compounds 3a-h

***Method A***: A mixture of salicylaldehyde derivatives **1** (1 mmol), 4-hydroxycoumarin (**2**, 2 mmol) and diammonium hydrogen phosphate (13.2 mg, 10 mol%) in $H_2O$ (10 mL) was stirred at room temperature for 5 h. After completion of the reaction as followed by TLC, the solid product was collected by filtration and purified by washing with cold 50% aq EtOH to afford the corresponding products.

***Method B***: A mixture of salicylaldehyde derivatives **1** (1 mmol), 4-hydroxycoumarin (**2**, 2 mmol) and *S*-proline (11.5 mg, 10 mol%) in $H_2O$ (10 mL) was stirred at room temperature for about 6 h. After completion of the reaction as monitored by TLC, the precipitate was collected by filtration and washed by cold 50% aq EtOH to afford the corresponding products.

***Method C*:** A mixture of neat salicylaldehyde derivatives **1** (1 mmol), 4-hydroxycoumarin (**2**, 2 mmol) were heated for 2 h at 80°C. Upon completion of the reaction as followed by TLC, 10 mL of 50% aq EtOH was added to the cooled mixture, which was then filtered. The pure products were obtained just by filtration.

**Selected data for 7-(4-hydroxy-2-oxo-2*H*-chromen-3-yl)-6*H*,7*H*-chromeno[4,3-*b*]chromen-6-ones (3a-h)**

*7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one*

(**3a**, $C_{25}H_{13}O_6$)

White solid, m.p. 255-256 °C; IR (KBr): $\nu_{max}$ = 3261, 1710, 1671, 1629 $cm^{-1}$; HR-MS (ESI-neg): $C_{25}H_{13}O_6$ $[M-1]^-$ Found. 409.07176, Calc. 409.07156; $^1$H-NMR ($\delta$, DMSO-$d_6$): 5.73 (s,1H, H-7), 7.13 (dt, 1H, $J$ = 7.1, 2.0 Hz, $H_{Ar}$), 7.20 (br d, 1H, $J$ = 7.5 Hz, $H_{Ar}$), 7.31 (m, 4H, $H_{Ar}$), 7.43 (d, 1H, $J$ = 8.2 Hz, $H_{Ar}$), 7.46 (t, 1H, $J$ = 7.5 Hz, $H_{Ar}$), 7.58 (dt, 1H, $J$ = 7.8, 1.3 Hz, $H_{Ar}$), 7.68 (dt, 1H, $J$ = 7.8, 1.3 Hz, $H_{Ar}$), 8.04 (br s, 1H, $H_{Ar}$), 8.07 (dd, 1H, $J$ = 7.9, 1.3 Hz, $H_{Ar}$), 12.25 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 28.7, 113.8, 116.1, 116.2, 116.4, 122.2, 122.6, 123.9, 124.5, 125.3, 128.3, 128.6, 132.1, 132.4, 149.2, 152.0, 152.2, 156.2, 160.3, 160.7 ppm.

*9-bromo-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one*

(**3b**, $C_{25}H_{12}BrO_6$)

White solid, m.p. 330-331 °C; IR (KBr): $\nu_{max}$ = 3270, 1707, 1670, 1629 $cm^{-1}$; HR-MS (ESI-neg): $C_{25}H_{12}{}^{81}BrO_6$ $[M-1]^-$ Found. 488.99683, Calc. 488.99712., $C_{25}H_{12}{}^{79}BrO_6$ $[M-1]^-$ Found. 486.98227, Calc. 486.98239; $^1$H-NMR ($\delta$, DMSO-$d_6$): 5.70 (s, 1H, H-7), 7.31 (m, 4H, $H_{Ar}$), 7.44 (m, 3H, $H_{Ar}$), 7.59 (t, 1H, $J$ = 7.5 Hz, $H_{Ar}$), 7.67 (t, 1H, $J$ = 7.5 Hz, $H_{Ar}$), 8.05 (m, 2H, $H_{Ar}$), 12.50 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 29.5, 114.4, 117.0, 117.2, 117.4, 117.5, 119.5, 123.5, 124.9, 125.4, 125.7, 131.7, 132.1, 133.2, 133.5, 149.5, 152.8, 153.1, 156.9, 161.1, 162.0 ppm.

*9,11-dibromo-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one* (**3c**, $C_{25}H_{11}Br_2O_6$)

Bright yellow solid, m.p. 208-210 °C; IR (KBr): $\nu_{max}$ = 3199, 1649, 1605, 1555 $cm^{-1}$; MS (EI, 70 eV): $C_{25}H_{11}Br_2O_6$ *m/z* (%) 570 ($M^+$+4, 14), 568 ($M^+$+2, 27), 566 ($M^+$, 14), 447(22), 420(47), 407(79), 317(16), 247(34), 219(18), 176(11), 162(83), 121(98), 120(100), 105(11), 92(69), 77(20), 63(36), 51(14); $^1$H-NMR ($\delta$, DMSO-$d_6$): 6.28 (s, 1H, H-7), 7.23 (m, 4H, $H_{Ar}$), 7.28 (d, 1H, $J$ = 2.3 Hz, $H_{Ar}$), 7.46 (d, 1H, $J$ = 2.3 Hz, $H_{Ar}$), 7.50 (dt, 2H, $J$ = 8.1, 1.3 Hz, $H_{Ar}$), 7.84 (dd, 2H, $J$ = 8.1, 1.3 Hz, $H_{Ar}$), 13.10 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 34.6, 103.8, 111.3, 112.6, 116.3, 120.8, 123.8, 125.0, 131.7, 131.9, 132.0, 136.2, 152.0, 153.2, 164.9, 168.4 ppm.

*9-bromo-11-chloro-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chro-men-6-one*

(**3d**, $C_{25}H_{11}BrClO_6$)

Pale yellow solid, m.p. 204-206 °C; IR (KBr): $\nu_{max}$ = 3195, 1651, 1606, 1556 $cm^{-1}$; MS (EI, 70 eV): $C_{25}H_{11}BrClO_6$ *m/z* (%) 528 ($M^+$+4, 8), 526 ($M^+$+2, 8), 524 ($M^+$, 27), 403(17), 376(41), 363(84), 317(7), 247(13), 219(12), 176(9), 162(77), 121(100), 105(13), 92(85), 77(20), 63(41), 51(16); $^1$H-NMR ($\delta$, DMSO-$d_6$): 6.28 (s, 1H, H-7), 7.16 (d, 1H, $J$ = 2.5 Hz, $H_{Ar}$),7.23 (m, 4H, $H_{Ar}$), 7.36 (d, 1H, $J$ = 2.5 Hz, $H_{Ar}$), 7.50 (dt, 2H, $J$ = 8.1, 1.5 Hz, $H_{Ar}$), 7.84 (dd, 2H, $J$ = 8.1, 1.5 Hz, $H_{Ar}$), 13.11 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): $\delta$ = 34.7, 103.8, 112.1, 116.3, 120.8, 123.7, 123.8, 125.0, 129.2, 129.4, 131.7, 135.7, 151.6, 153.2, 164.9, 168.4 ppm.

*9,11-dichloro-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one* (**3e**, $C_{25}H_{11}Cl_2O_6$)

Pale yellow solid, m.p. 200-202 °C; IR (KBr): $\nu_{max}$ = 3195, 1661, 1605, 1556 $cm^{-1}$; MS (EI, 70 eV): $C_{25}H_{11}Cl_2O_6$ *m/z* (%) 482 ($M^+$+4, 9), 480 ($M^+$+2, 33), 478 ($M^+$, 46), 357(41), 330(52), 317(100), 162(23), 121(48), 105(5), 92(20), 77(7), 51(5); $^1$H-NMR ($\delta$, DMSO-$d_6$): 6.26 (s, 1H, H-7), 7.11 (t, 2H, $J$ = 1.3 Hz, $H_{Ar}$), 7.24 (m, 4H, $H_{Ar}$), 7.50 (dt, 2H, $J$ = 8.1, 1.5 Hz, $H_{Ar}$), 7.84 (dd, 2H, $J$ = 8.1, 1.5 Hz, $H_{Ar}$), 13.21 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): $\delta$ = 34.5, 103.8, 116.3, 120.8, 121.7, 123.8, 123.8, 124.9, 126.5, 128.6, 131.7, 135.4, 150.6, 153.3, 164.9, 168.3 ppm.

*11-methoxy-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one*
(**3f**, $C_{26}H_{16}O_7$)
White solid, m.p. 292-293 °C; IR (KBr): $\nu_{max}$ = 3074, 1720, 1661, 1639 $cm^{-1}$; HR-MS (ESI-neg): $C_{26}H_{16}O_7$ $[M-1]^-$ Found. 439.08233, Calc. 439.08232; $^1$H-NMR ($\delta$, DMSO-$d_6$): 3.92 (s, 3H, $OCH_3$), 5.71 (s, 1H, H-7), 6.74 (d, 1H, $J$ = 7.4 Hz, $H_{Ar}$), 6.99 (d, 1H, $J$ = 7.9 Hz, $H_{Ar}$), 7.06 (t, 1H, $J$ = 7.9 Hz, $H_{Ar}$), 7.29 (m, 2H, $H_{Ar}$), 7.43 (d, 1H, $J$ = 8.3 Hz, $H_{Ar}$), 7.48 (t, 1H, $J$ = 7.6 Hz, $H_{Ar}$), 7.58 (t, 1H, $J$ = 7.6 Hz, $H_{Ar}$), 7.67 (t, 1H, $J$ = 7.4 Hz, $H_{Ar}$), 7.99 (m, 2H, $H_{Ar}$), 12.30 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 28.6, 56.0, 111.2, 113.9, 116.0, 116.2, 116.4, 119.6, 122.3, 123.7, 123.9, 124.6, 125.1, 132.1, 132.4, 147.3, 151.9, 152.1, 155.9, 160.3 ppm.

*9-methyl-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one*
(**3g**, $C_{26}H_{16}O_6$)
White solid, m.p. 328-329 °C; IR (KBr): $\nu_{max}$ = 3257, 1712, 1666, 1631 $cm^{-1}$; HR-MS (ESI-neg): $C_{26}H_{16}O_6$ $[M-1]^-$ Found. 423.08741, Calc. 423.08714; $^1$H-NMR ($\delta$, DMSO-$d_6$): 2.20 (s, 3H, $CH_3$), 5.69 (s, 1H, H-7), 6.98 (br s, 1H, $H_{Ar}$), 7.08 (d, 1H, $J$ = 7.1 Hz, $H_{Ar}$), 7.19 (t, 1H, $J$ = 8.3 Hz, $H_{Ar}$), 7. 31 (m, 2H, $H_{Ar}$), 7.44 (m, 2H, $H_{Ar}$), 7.58 (t, 1H, $J$ = 7.3 Hz, $H_{Ar}$), 7.66 (t, 1H, $J$ = 7.3 Hz, $H_{Ar}$), 8.05 (m, 2H, $H_{Ar}$), 12.61 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 20.2, 28.6, 100.4, 113.8, 115.9, 116.0, 116.1, 116.32, 121.8, 122.5, 123.8, 124.4, 128.5, 128.8, 132.0, 132.3, 134.3, 147.1, 151.9, 152.1, 156.2, 160.3, 160.5 ppm.

*9-nitro-7-(4-hydroxy-2-oxo-2H-chromen-3-yl)-6H,7H-chromeno[4,3-b]chromen-6-one*
(**3h**, $C_{25}H_{13}NO_8$)
White solid, m.p. 299-301 °C; IR (KBr): $\nu_{max}$ = 3239, 1699, 1637, 1569 $cm^{-1}$; HR-MS (ESI-pos): $C_{25}H_{13}NO_8$ $[M+1]^+$ Found. 456.07139, Calc. 456.07160; $^1$H-NMR ($\delta$, DMSO-$d_6$): 5.81 (s, 1H, H-7), 7.31 (d, 1H, $J$ = 8.2 Hz, $H_{Ar}$), 7.38 (t, 1H, $J$ = 7.3 Hz, $H_{Ar}$), 7.50 (m, 2H, $H_{Ar}$), 7.61 (m, 2H, $H_{Ar}$), 7.72 (t, 1H, $J$ = 7.3 Hz, $H_{Ar}$), 8.13 (m, 4H, $H_{Ar}$), 12.30 (br s, 1H, OH) ppm; $^{13}$C-NMR ($\delta$, DMSO-$d_6$): 28.7, 113.3, 116.0, 116.2, 116.5, 117.5, 122.6, 123.8, 124.0, 124.2, 124.3, 124.7, 132.4, 132.8, 144.2, 152.0, 152.3, 160.0 ppm.

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