General procedure for the synthesis of coumarines

Phenol derivative (2.5 mmol), β-diketone (2.5 mmol) and catalytic amount of meglumine sulfate (0.04 g) were mixed and heated at 100°C in thermal conditions. In microwave conditions, the reaction mixture subjected to microwave irradiation at power of 600W and 60-70°C for 6 minutes. The progress of the reaction was monitored by TLC. After the completion of reaction, the reaction mixture was cooled to room temperature and 10 ml of EtOH was added. Then, obtained mixture poured into crushed ice and stirred for 10 min. The crude product was collected by filtration, washed with ice cold distilled water and recrystallized from hot ethanol to afford pure corresponding pure product.

The characterization data for synthesized coumarins are given below:

7-hydroxy-4-methyl-chromen-2-one (3a). Yellow powder: m.p 185-186°C; IR (KBr) \( \nu_{\text{max}} \): 3498, 3113, 1605, 1669, 1450, 1390, 1213 \text{ cm}^{-1}; ^1H\text{ NMR (Acetone-d}_6, 400 \text{ MHz})\): \( \delta \) 9.62 (s, 1H, OH), 7.52 (d, J= 8.4 Hz, 1H, H-5), 6.83 (d, J= 8.4 Hz, 1H, H-6), 6.38 (s, 1H, H-8), 6.25 (s, 1H, H-3), 2.56 (s, 3H, CH$_3$-C-4) ppm; $^{13}$C NMR (100 MHz, Acetone-d$_6$): \( \delta \) 161.18 (C-2), 160.39 (C-7), 155.39 (C-4), 153.15 (C-9), 126.36 (C-5), 112.70 (C-10), 112.61 (C-6), 110.75 (C-3), 102.39...
(C-8), 17.67 (CH₃) ppm; Anal. Found: C, 67.97; H, 4.49; O, 27.25%. Calcd for C₁₀H₈O₃: C, 68.18; H, 4.55; O, 27.27%.

7,8-Dihydroxy-4-methyl-chromen-2-one (3b). Orange powder: m.p 243-245°C; IR (KBr) νₘₐₓ: 3413, 3233, 1690, 1584, 11371, 1057 cm⁻¹; ¹H NMR (DMSO-d₆, 400 MHz): δ 9.96 (s, 1H, OH), 9.10 (s, 1H, OH), 7.06 (d, J= 8.4 Hz, 1H, H-5), 6.79 (d, J= 8.4 Hz, 1H, H-6), 6.12 (s, 1H, H-3), 3.11 (s, 3H, CH₃-C-4) ppm; Anal. Found: C, 62.41; H, 4.15; O, 33.31%. Calcd for C₁₀H₈O₄: C, 62.46; H, 4.16; O, 33.33%.

4-methyl-benzo[h]chromen-2-one (3c). Yellow powder: m.p 153-155°C; IR (KBr) νₘₐₓ: 3057, 2917, 1910, 1714, 1373, 1471, 1080 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz): δ 7.59-8.57 (m, 6H, ArH), 6.38 (s, 1H, H-3), 6.63 (s, 3H, CH₃-C-4) ppm; Anal. Found: C, 79.92; H, 4.75; O, 15.21%. Calcd for C₁₄H₁₀O₂: C, 80.00; H, 4.76; O, 15.23%.

5,7-Dihydroxy-4-methyl-chromen-2-one (3d). Orange powder: m.p 282-285°C; IR (KBr) νₘₐₓ: 3435, 3137, 2687, 1668, 1551, 1471, 1390, 1299, 1158 cm⁻¹; ¹H NMR (DMSO-d₆, 400 MHz): δ 10.53 (s, 1H, OH), 10.31 (s, 1H, OH), 6.23 (s, 1H, H-3), 6.14 (s, 1H, H-8), 5.83 (s, 1H, H-6), 3.29 (s, 3H, CH₃-C-4) ppm; Anal. Found: C, 62.43; H, 4.13; O, 33.32%. Calcd for C₁₀H₈O₄: C, 62.5; H, 4.16; O, 33.33%.

5-hydroxy-4,7-dimethyl-chromen-2-one (3e). Yellow powder: m.p 250-253°C; IR (KBr) νₘₐₓ: 3272, 2979, 2922, 1822, 1681, 1603, 1385, 1281, 1093 cm⁻¹; ¹H NMR (DMSO-d₆, 400 MHz): δ 10.51 (s, 1H, OH), 6.58 (s, 1H, H-8), 6.53 (s, 1H, H-6), 6.01 (s, 1H, H-3), 2.49 (s, 3H, CH₃), 2.23
(s, 3H) ppm; Anal. Found: C, 64.46; H, 5.24; O, 25.25%. Calcd for C_{11}H_{10}O_3: C, 64.47; H, 5.26; O, 25.26%.

**7-hydroxy-4-phenyl-chromen-2-one (3f):** Bright pink powder: m.p 257-259°C; IR (KBr) \( \nu_{\text{max}} \): 3104, 2802, 2599, 1696, 1610, 1444, 1375, 1238 cm\(^{-1}\); \(^1\)H NMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 10.42 (s, 1H, OH), 7.46-7.49 (m, 5H, Ph), 7.19 (d, J= 8.4 Hz, 1H, H-5), 6.76 (s, 2H, H-6, H-8), 6.09 (s, 1H, H-3) ppm; Anal. Found: C, 70.85; H, 3.92; O, 25.18%. Calcd for C\(_{15}\)H\(_{10}\)O\(_4\): C, 70.87; H, 3.94; O, 25.19%.

**7,8-Dihydroxy-4-phenyl-chromen-2-one (3g).** Bright yellow powder: m.p 194-197°C; IR (KBr) \( \nu_{\text{max}} \): 3402, 1890, 1696, 1606, 1450, 1356, 1210, 1071 cm\(^{-1}\); \(^1\)H NMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 10.24 (s, 1H, OH), 9.48 (s, 1H, OH), 7.5 (m, 5H, ArH), 6.7 (d, J=2.8 Hz, H-5, H-6), 6.12 (s, 1H, ArH) ppm; \(^13\)C NMR (DMSO-d\(_6\), 100 MHz): \( \delta \) 160.57 (C=O), 156.42 (C-4), 150.09 (C-9), 144.35 (C-7), 135.91 (C-8), 133.09-128.88 (6C of ph), 117.77 (C-10), 112.79 (C-6), 111.96 (C-5) ppm; Anal. Found: C, 70.85; H, 3.93; O, 25.17%. Calcd for C\(_{15}\)H\(_{10}\)O\(_4\): C, 70.87; H, 3.94; O, 25.19%.

**5,7-Dihydroxy-4-phenyl-chromen-2-one (3h).** Bright orange powder: m.p 245-247°C; IR (KBr) \( \nu_{\text{max}} \): 3217, 1665, 1609, 1460, 1371, 1295, 1146, 1073; \(^1\)H NMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 10.42 (s, 1H, OH), 10.13 (s, 1H, OH), 7.33 (s, 5H, ArH), 6.24 (s, 1H, H-3), 6.13 (s, 1H, H-8), 5.7 (s, 1H, H-6) ppm; \(^13\)C NMR (DMSO-d\(_6\), 100 MHz): \( \delta \) 162.18 (C=O), 160.40 (C-7), 157.57 (C-4), 157.21 (C-5), 156.3 (C-4), 140.02-127.73 (6C of ph), 110.62 (C-10), 101.03 (C-3), 99.58 (C-8),
95.10 (C-6) ppm; Anal. Found: C, 70.86; H, 3.92; O, 25.20%. Calcd for C_{15}H_{10}O_{4}: C, 70.87; H, 3.94; O, 25.19%.

7-amino-4-methyl-chromen-2-one (3i). Bright yellow powder: m.p 222-223 °C; IR (KBr) \( \nu_{\text{max}} \); 3356, 2989, 1696, 1451, 1486, 1213, 1092 cm\(^{-1}\); \(^1\)H NMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 7.34 (d, \( J=8.7 \) Hz, 1H, H-5), 6.74 (d, \( J=8.7 \) Hz, 1H, H-6), 6.39 (s, 1H, H-8), 5.84 (s, 1H, H-3), 3.92 (s, 2H, CH\(_2\)-C-4) ppm; Anal. Found: C, 68.55; H, 5.13; O, 18.30%. Calcd for C\(_{10}\)H\(_9\)O\(_2\): C, 68.57; H, 5.14; O, 18.29%.

7-methoxy-4-methyl-chromen-2-one (3j). White powder m.p 160-161 °C; IR (KBr) \( \nu_{\text{max}} \); 3355, 2983, 1698, 1482, 1207, 1113 cm\(^{-1}\); \(^1\)HNMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 7.52 (d, \( J=8.1 \) Hz, 1H, H-5), 6.86 (d, \( J=8.1 \) Hz, 1H, H-6), 6.38 (s, 1H, H-8), 6.25 (s, 1H, H-3), 3.68 (s, 3H, OCH\(_3\)), 2.56 (s, 3H, CH\(_3\)-C-4) ppm; Anal. Found: C, 69.47; H, 5.24; O, 25.25%. Calcd for C\(_{11}\)H\(_{10}\)O\(_3\): C, 69.47; H, 5.26; O, 25.26%.

4-methyl-chromen-2-one (3l). Bright pink powder :m.p 77-79 °C; IR (KBr) \( \nu_{\text{max}} \); 3342, 2983, 1690, 1490, 1210, 1027 cm\(^{-1}\); \(^1\)HNMR (CDCl\(_3\), 400MHz): \( \delta \) 7.63 (d, \( J=6.0 \) Hz, 1H, H-5), 7.18-7.48 (m, 3H, H-6, H-8), 6.36 (s, 1H, H-3), 2.38 (s, 3H, CH\(_3\)-C-4) ppm; Anal. Found: C, 74.95; H, 5.01; O, 20.05%. Calcd for C\(_{10}\)H\(_8\)O\(_2\): C, 75.00; H, 5.00; O, 20.00%.

6-hydroxy-4-methyl-chromen-2-one (3m): Yellow powder m.p 180-182 °C; IR (KBr) \( \nu_{\text{max}} \); 3377, 2981, 1682, 1617, 1493, 1201, 1099 cm\(^{-1}\); \(^1\)HNMR (DMSO-d\(_6\), 400 MHz): \( \delta \) 8.92 (s, 1H, OH), 7.25 (s, 1H, H-5), 6.86 (d, \( J = 8.4 \) Hz, 1H, H-8), 6.58 (d, \( J = 8.4 \) Hz, 1H, H-7), 6.32 (s, 1H,
H-3), 2.38 (s, 3H, CH$_3$-C-4) ppm; Anal. Found: C, 68.21; H, 4.58; O, 27.26%. Calcd for C$_{10}$H$_8$O$_3$: C, 68.18; H, 4.55; O, 27.27%.
$^1$HNMR of 3a
$^{13}$CNMR of 3a
$^1$HNMR of 3b
$^{1}$HNMR of 3C
$^1$HNMR of 3d
$^1$HNMR of 3e
$^1$HNMR of 3f
$^1$HNMR of 3g
$^{13}$C NMR of 3g
Expand $^{13}$CNMR of 3g
$^1$HNMR of 3h
$^{13}$CNMR of 3h
$^1$HNMR of 3i
HNMR of 3j
$^1$HNMR of 31
"$^1$HNMR of 3m"