

Supporting Information

Palladium Catalyzed Annulation of 2-Iodobiphenyl with Non-terminal Alkene Enabled by Neighboring Group Assistance

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1. General

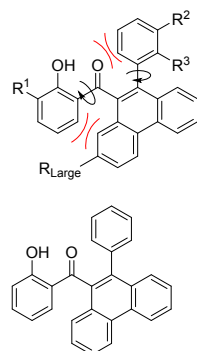
$\text{Pd}(\text{OAc})_2$ was purchased from Energy Chemical, 2'-OH Chalcone^[1] and 2-Iodobiphenyl^[2] derivatives were synthesized by the previous reported. Besides, all substrates and reagents were commercially available and used without further purification. TLC analysis was performed using pre-coated glass plates. Column chromatography was performed using silica gel (200–300 mesh). ^1H spectra were recorded in CDCl_3 and $\text{DMSO}-d_6$ on 600/400 MHz NMR spectrometers and resonances (δ) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C spectra were recorded in CDCl_3 and $\text{DMSO}-d_6$ on 150/100 MHz NMR spectrometers and resonances (δ) are given in ppm. HRMS were obtained on a Bruker 7-tesla FT-ICR MS equipped with an electrospray source. The X-ray crystal-structure determinations were obtained on a Bruker SMART APEX CCD system.

2. General procedure for synthesis of 3-32 (3 as an example)

A 25ml Schlenk-type tube (with a Teflon screw cap and a side arm) equipped with a magnetic stir bar was charged with the mixture of 2'-OH Chalcone **1** (0.1 mmol), 2-Iodobiphenyl **2** (0.15 mmol), $\text{Pd}(\text{OAc})_2$ (5 mol%), Na_2CO_3 (0.2 mmol) in DMF (1.0 mL). The reaction mixture was frozen with the liquid nitrogen and then the tube was evacuated and backfilled with argon (5 times). The mixture was first stirred at room temperature for 10 minutes and then stirred at 100 °C for 12 hours. After cooling to room temperature, the mixture was quenched with water (25 mL), extracted with EtOAc (3×50 mL), the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 100/1) to afford the product **3**.

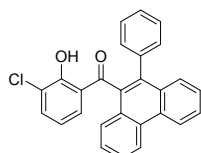
3. Characterization data for compounds

NOTE: For the compounds 3-32, asymmetrical substitutions on alkene substrate restrain the free rotation of the C-C bond due to the steric effects, resulting in unpredictable and unequal peak split of ^{13}C and ^1H NMR spectra for the corresponding compounds.



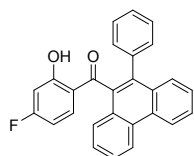
(2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (3):

Yield 76%; 28.4 mg; yellow solid; mp 198–201 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =11.99 (s, 1H), 8.81 (d, $J = 7.8$ Hz, 2H), 7.75 – 7.60 (m, 4H), 7.54 (s, 2H), 7.44 – 7.37 (m, 2H), 7.36 – 7.32 (m, 1H), 7.27 (d, $J = 6.3$ Hz, 1H), 7.22 – 7.17 (m, 2H), 7.13 (d, $J = 7.0$ Hz, 1H), 6.89 (d, $J = 8.2$ Hz, 1H), 6.59 (d, $J = 7.4$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =204.9, 162.4, 136.8, 136.7, 135.9, 133.8, 133.3, 131.3, 130.9, 130.7, 129.9, 129.8, 128.5, 128.3, 127.9, 127.8, 127.7, 127.6, 127.4, 127.2, 127.1, 126.3, 122.9, 122.7, 121.0, 118.8, 117.9. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{17}\text{O}_2^-$ (M-H) $^-$ 373.1307, found 373.1234.



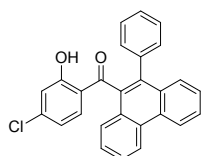
(3-chloro-2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (4):

Yield 46%; 18.7 mg; yellow solid; mp 118–121 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ =12.56 (s, 1H), 8.82 (d, $J = 8.4$ Hz, 2H), 7.73 (dt, $J = 15.1, 7.7$ Hz, 2H), 7.67 – 7.52 (m, 4H), 7.47 – 7.37 (m, 3H), 7.31 (s, 1H), 7.21 (s, 1H), 7.14 (t, $J = 6.5$ Hz, 2H), 6.57 (t, $J = 7.9$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =195.4, 158.3, 156.8, 141.4, 141.1, 140.6, 137.6, 135.7, 130.3, 129.9, 129.6, 128.9, 128.8, 128.4, 128.3, 128.2, 127.7, 127.2, 126.8, 122.5, 122.3, 121.4, 118.4. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{18}\text{ClO}_2$ (M+H) $^+$ 409.0917, found 409.1006.



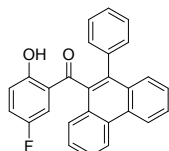
(4-fluoro-2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (5):

Yield 75%; 29.4 mg; yellow solid; mp 172–175 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =12.33 (s, 1H), 8.78 (d, $J = 8.3$ Hz, 2H), 7.74 – 7.66 (m, 2H), 7.65 (d, $J = 8.1$ Hz, 2H), 7.53 (dd, $J = 16.1, 8.1$ Hz, 2H), 7.43 – 7.36 (m, 2H), 7.29 (t, $J = 6.8$ Hz, 1H), 7.21 (dd, $J = 13.3, 6.4$ Hz, 2H), 7.12 (d, $J = 7.3$ Hz, 1H), 6.56 (d, $J = 10.2$ Hz, 1H), 6.29 (t, $J = 8.3$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =203.6, 167.5 (d, $J = 256.5$ Hz, $^1J_{\text{CF}}$), 165.0 (d, $J = 15.0$ Hz, $^3J_{\text{CF}}$), 136.7, 136.0, 135.7 (d, $J = 12.0$ Hz, $^3J_{\text{CF}}$), 133.4, 131.3, 130.7, 129.8, 128.4, 128.3, 127.9, 127.8, 127.7, 127.5, 127.2, 127.1, 126.2, 123.0, 122.7, 118.1, 107.2 (d, $J = 22.5$ Hz, $^2J_{\text{CF}}$), 104.6 (d, $J = 24.0$ Hz, $^2J_{\text{CF}}$). HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{18}\text{FO}_2^+$ (M+H) $^+$ 393.1213, found 393.1284.



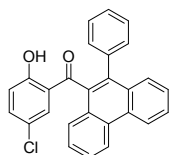
(4-chloro-2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (6):

Yield 63%; 25.7 mg; yellow solid; mp 139–142 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =12.42 (s, 1H), 8.75 (d, $J = 7.7$ Hz, 2H), 7.64 (dd, $J = 20.3, 17.4$ Hz, 3H), 7.50 (s, 2H), 7.36 (dd, $J = 24.3, 9.0$ Hz, 2H), 7.27 (s, 1H), 7.24 – 7.15 (m, 2H), 7.10 (dd, $J = 12.4, 8.7$ Hz, 2H), 6.98 (d, $J = 17.4$ Hz, 1H), 6.90 (s, 1H), 6.53 (d, $J = 7.8$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =204.1, 163.0, 142.4, 141.4, 136.6, 136.0, 134.2, 133.3, 131.3, 130.7, 129.8, 128.8, 128.3, 128.1, 127.9, 127.8, 127.7, 127.5, 127.2, 127.1, 126.1, 122.9, 122.7, 119.5, 118.0. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{18}\text{ClO}_2^+$ ($\text{M}+\text{H}$) $^+$ 409.0917, found 409.0988.



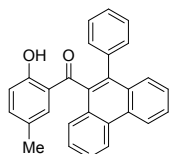
(5-fluoro-2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (7):

Yield 85%; 33.3 mg; yellow solid; mp 164–166 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =11.76 (s, 1H), 8.81 (d, $J = 8.3$ Hz, 2H), 7.77 – 7.69 (m, 2H), 7.69 – 7.60 (m, 2H), 7.61 – 7.52 (m, 2H), 7.40 (s, 2H), 7.29 (s, 1H), 7.23 (t, $J = 7.3$ Hz, 1H), 7.18 (d, $J = 7.2$ Hz, 1H), 7.08 (d, $J = 6.1$ Hz, 1H), 6.85 (dd, $J = 9.7, 6.2$ Hz, 2H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =204.2, 158.6, 154.6 (d, $J = 237.0$ Hz, $^1J_{\text{CF}}$), 136.6, 136.1, 133.1, 131.3, 130.8, 130.6, 129.9, 129.8, 128.7, 128.4, 128.2, 128.0, 127.8, 127.5, 127.3, 127.2, 126.0, 124.4 (d, $J = 24.0$ Hz, $^2J_{\text{CF}}$), 123.0, 122.7, 120.5, 119.3, 117.6 (d, $J = 22.5$ Hz, $^2J_{\text{CF}}$). HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{FO}_2^-$ ($\text{M}-\text{H}$) $^-$ 391.1213, found 391.1139.



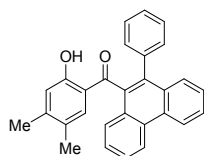
(5-chloro-2-hydroxyphenyl)(10-phenylphenanthren-9-yl)methanone (8):

Yield 77%; 31.4 mg; yellow solid; mp 192–195 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =11.92 (s, 1H), 8.83 (d, $J = 8.1$ Hz, 2H), 7.78 – 7.70 (m, 2H), 7.67 (dd, $J = 13.9, 8.0$ Hz, 2H), 7.57 (dd, $J = 16.9, 7.7$ Hz, 2H), 7.38 (s, 2H), 7.31 – 7.22 (m, 3H), 7.19 (d, $J = 6.0$ Hz, 1H), 7.14 (d, $J = 2.3$ Hz, 1H), 6.84 (d, $J = 8.9$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =204.2, 160.9, 136.7, 136.6, 136.2, 131.9, 131.2, 130.8, 130.6, 130.1, 129.9, 128.5, 128.3, 128.0, 127.8, 127.6, 127.4, 127.2, 126.0, 123.4, 123.1, 122.8, 121.4, 119.6. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{ClO}_2^-$ ($\text{M}-\text{H}$) $^-$ 407.0917, found 407.0844.



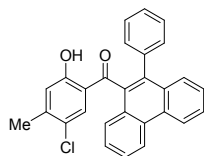
(2-hydroxy-5-methylphenyl)(10-phenylphenanthren-9-yl)methanone (9):

Yield 83%; 32.2 mg; yellow solid; mp 161–163 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.89$ (s, 1H), 8.80 (s, 2H), 7.68 (dd, $J = 21.8, 14.4$ Hz, 4H), 7.54 (s, 2H), 7.39 (d, $J = 10.4$ Hz, 2H), 7.26 (s, 1H), 7.20 (d, $J = 7.1$ Hz, 1H), 7.14 (d, $J = 6.9$ Hz, 2H), 6.98 (s, 1H), 6.79 (d, $J = 8.3$ Hz, 1H), 1.99 (s, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.8, 160.4, 137.9, 136.9, 135.7, 133.9, 132.7, 131.3, 130.9, 130.7, 129.9, 129.8, 128.6, 128.3, 127.9, 127.8, 127.6, 127.5, 127.4, 127.1, 127.0, 126.4, 122.9, 122.7, 120.6, 117.6, 20.2$. HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{19}\text{O}_2^-$ (M-H) $^-$ 387.1463, found 387.1391.



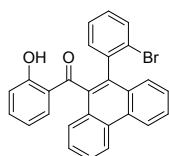
(2-hydroxy-4,5-dimethylphenyl)(10-phenylphenanthren-9-yl)methanone (10):

Yield 72%; 28.9 mg; white solid; mp 155–158 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.92$ (s, 1H), 8.82 (t, $J = 7.1$ Hz, 2H), 7.86 – 7.61 (m, 4H), 7.55 (d, $J = 7.1$ Hz, 2H), 7.40 (d, $J = 7.6$ Hz, 2H), 7.28 (s, 1H), 7.21 (s, 1H), 7.16 (s, 1H), 6.91 (s, 1H), 6.70 (s, 1H), 2.17 (s, 3H), 1.90 (s, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.0, 160.8, 147.6, 137.0, 135.6, 134.1, 133.1, 131.3, 130.9, 130.6, 129.8, 128.6, 128.3, 127.8, 127.7, 127.6, 127.4, 127.3, 127.2, 127.1, 127.0, 126.5, 122.8, 122.7, 119.0, 118.5, 20.6, 18.6$. HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{21}\text{O}_2^-$ (M-H) $^-$ 401.1620, found 401.1547.



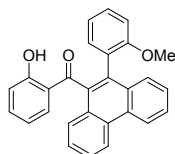
(5-chloro-2-hydroxy-4-methylphenyl)(10-phenylphenanthren-9-yl)methanone (11):

Yield 80%; 33.7 mg; white solid; mp 168–170 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.90$ (s, 1H), 8.82 (d, $J = 6.8$ Hz, 2H), 7.82 – 7.68 (m, 2H), 7.68 – 7.61 (m, 2H), 7.56 (d, $J = 5.6$ Hz, 2H), 7.39 (s, 2H), 7.31 – 7.23 (m, 2H), 7.20 (s, 1H), 7.12 (s, 1H), 6.78 (s, 1H), 2.27 (s, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=203.6, 160.8, 146.1, 136.8, 136.0, 133.2, 132.4, 131.3, 130.8, 130.7, 130.0, 129.9, 128.4, 128.3, 127.9, 127.8, 127.7, 127.5, 127.3, 127.1, 126.1, 124.1, 123.0, 122.7, 120.0, 119.9, 20.9$. HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{18}\text{ClO}_2^-$ (M-H) $^-$ 421.1074, found 421.1001.



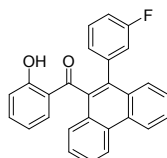
(10-(2-bromophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (12):

Yield 43%; 19.4 mg; yellow solid; mp 206–209 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=12.01$ (s, 1H), 8.80 (d, $J = 8.2$ Hz, 2H), 7.72 – 7.65 (m, 3H), 7.54 (d, $J = 5.1$ Hz, 2H), 7.43 – 7.38 (m, 2H), 7.36 – 7.32 (m, 1H), 7.29 – 7.26 (m, 1H), 7.19 (dd, $J = 11.8, 7.7$ Hz, 2H), 7.13 (d, $J = 7.1$ Hz, 1H), 6.89 (d, $J = 8.4$ Hz, 1H), 6.59 (t, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.9, 162.4, 136.8, 136.7, 135.9, 133.7, 133.3, 131.3, 130.8, 130.6, 129.9, 129.8, 128.5, 128.3, 127.8, 127.7, 127.5, 127.4, 127.2, 127.1, 126.3, 122.9, 122.7, 121.0, 118.8, 117.9$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{BrO}_2^-$ (M-H) $^-$ 451.0412, found 451.0339.



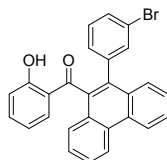
(2-hydroxyphenyl)(10-(2-methoxyphenyl)phenanthren-9-yl)methanone (13):

Yield 64%; 25.8 mg; yellow solid; mp 196–199 °C; $R_f = 0.3$ (silica gel, PE:EA = 50:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=12.01$ (s, 1H), 8.78 (s, 2H), 7.74 – 7.61 (m, 3H), 7.52 (d, $J = 5.5$ Hz, 3H), 7.36 (d, $J = 7.0$ Hz, 1H), 7.28 (dd, $J = 26.6, 6.5$ Hz, 3H), 6.96 (d, $J = 6.9$ Hz, 1H), 6.86 (d, $J = 7.9$ Hz, 1H), 6.68 (d, $J = 7.7$ Hz, 1H), 6.54 (s, 1H), 3.68 (s, 1H), 3.39 (s, 2H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=205.1, 162.2, 156.6, 136.5, 134.2, 133.7, 133.5, 132.8, 130.9, 130.6, 130.2, 130.0, 129.9, 128.8, 127.6, 127.4, 127.2, 127.0, 126.2, 125.2, 122.9, 122.6, 120.7, 120.4, 120.1, 118.9, 118.1, 117.9, 117.4, 110.0, 55.8, 54.7$. HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{19}\text{O}_3^-$ (M-H) $^-$ 403.1412, found 403.1340.



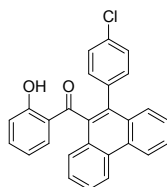
(10-(3-fluorophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (14):

Yield 86%; 33.7 mg; yellow solid; mp 217–220 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.99$ (d, $J = 10.5$ Hz, 1H), 8.80 (d, $J = 7.2$ Hz, 2H), 7.71 (dd, $J = 18.7, 8.5$ Hz, 2H), 7.63 (ddd, $J = 37.8, 28.0, 17.2$ Hz, 4H), 7.37 (s, 2H), 7.25 – 7.12 (m, 2H), 7.00 (s, 1H), 6.96 – 6.81 (m, 2H), 6.68 – 6.55 (m, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.6, 204.5, 162.5$ (d, $J = 7.5$ Hz, $^3J_{\text{CF}}$), 162.4 (d, $J = 247.5$ Hz, $^1J_{\text{CF}}$), 162.0 (d, $J = 246.0$ Hz, $^1J_{\text{CF}}$), 161.1, 139.0, 137.0, 136.9, 134.5, 134.4, 133.9, 133.3, 133.1, 130.7, 130.5, 130.3, 130.0, 129.9, 129.8, 129.7, 129.4 (d, $J = 7.5$ Hz, $^3J_{\text{CF}}$), 128.3, 127.7, 127.6, 127.4, 127.2, 126.4, 126.3, 125.7, 122.9 (d, $J = 21.0$ Hz, $^2J_{\text{CF}}$), 120.8 (d, $J = 22.5$ Hz, $^2J_{\text{CF}}$), 119.0, 118.9, 118.5 (d, $J = 21.0$ Hz, $^2J_{\text{CF}}$), 118.0, 117.0 (d, $J = 22.5$ Hz, $^2J_{\text{CF}}$), 115.0 (d, $J = 12.0$ Hz, $^3J_{\text{CF}}$), 114.9 (d, $J = 12.0$ Hz, $^3J_{\text{CF}}$). HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{FO}_2^-$ (M-H) $^-$ 391.1213, found 391.1140.



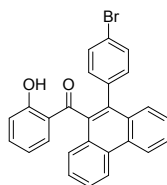
(10-(3-bromophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (15):

Yield 70%; 31.6 mg; yellow solid; mp 200–203 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.95$ (d, $J = 27.8$ Hz, 1H), 8.81 (d, $J = 7.4$ Hz, 2H), 7.81 – 7.64 (m, 3H), 7.63 – 7.47 (m, 4H), 7.42 – 7.27 (m, 3H), 7.16 (d, $J = 5.2$ Hz, 1H), 7.06 (s, 1H), 6.93 (t, $J = 9.3$ Hz, 1H), 6.63 (dt, $J = 25.5, 7.3$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.5, 162.4, 138.9, 137.1, 136.9, 134.2, 133.1, 131.0, 130.6, 130.5, 130.3, 130.0, 129.9, 129.3, 128.5, 128.3, 127.7, 127.6, 127.5, 127.3, 126.4, 122.9, 122.8, 122.4, 121.7, 120.8, 119.0, 118.9, 118.0$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{BrO}_2^-$ (M-H) $^-$ 451.0412, found 451.0339.



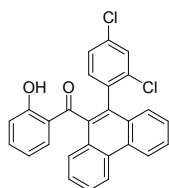
(10-(4-chlorophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (16):

Yield 72%; 29.4 mg; yellow solid; mp 183–186 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta=12.01$ (s, 1H), 8.78 (dd, $J = 8.3, 2.5$ Hz, 2H), 7.74 – 7.63 (m, 3H), 7.59 – 7.50 (m, 3H), 7.39 – 7.33 (m, 3H), 7.18 – 7.14 (m, 2H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.93 (d, $J = 8.4$ Hz, 1H), 6.58 (t, $J = 7.6$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta=204.6, 162.5, 137.0, 135.3, 134.5, 134.0, 133.9, 133.1, 132.7, 131.2, 130.6, 130.5, 129.8, 128.6, 128.3, 128.1, 127.7, 127.5, 127.4, 127.2, 126.4, 122.9, 122.8, 120.8, 118.9, 118.1$. HRMS (ESI) m/z calcd for: $\text{C}_{27}\text{H}_{16}\text{ClO}_2^-$ (M-H) $^-$ 407.0917, found 407.0844.



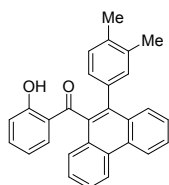
(10-(4-bromophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (17):

Yield 55%; 24.9 mg; yellow solid; mp 165–167 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=12.01$ (s, 1H), 8.79 (dd, $J = 7.6, 4.2$ Hz, 2H), 7.70 (dt, $J = 15.3, 7.5$ Hz, 2H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.55 (dd, $J = 21.9, 7.3$ Hz, 4H), 7.33 (ddd, $J = 19.7, 11.8, 4.4$ Hz, 3H), 7.15 (d, $J = 8.0$ Hz, 1H), 7.05 – 6.98 (m, 1H), 6.94 (d, $J = 8.4$ Hz, 1H), 6.59 (t, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.6, 162.5, 137.0, 135.7, 134.5, 133.9, 133.1, 133.0, 131.5, 131.4, 131.0, 130.6, 130.5, 129.8, 128.3, 127.7, 127.5, 127.4, 127.2, 126.4, 122.9, 122.8, 122.2, 120.8, 119.0, 118.1$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{BrO}_2^-$ (M-H) $^-$ 451.0412, found 451.0339.



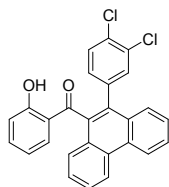
(10-(2,4-dichlorophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (18):

Yield 61%; 27.0 mg; yellow solid; mp 188–191 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.98$ (s, 1H), 8.77 (d, $J = 7.4$ Hz, 2H), 7.80 – 7.57 (m, 3H), 7.50 (dd, $J = 17.7, 8.4$ Hz, 2H), 7.37 (ddd, $J = 33.7, 15.6, 8.3$ Hz, 3H), 7.33 – 7.18 (m, 2H), 6.96 (ddd, $J = 32.6, 28.1, 8.1$ Hz, 2H), 6.68 – 6.55 (m, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.3, 203.5, 162.5, 162.3, 137.3, 137.0, 136.2, 134.9, 134.4, 134.3, 133.8, 133.1, 132.3, 132.0, 131.7, 130.5, 130.3, 130.2, 130.1, 129.9, 129.6, 129.4, 129.3, 128.4, 127.8, 127.7, 127.6, 127.4, 127.3, 127.1, 127.0, 126.8, 126.7, 126.5, 126.4, 122.9, 121.1, 120.4, 119.1, 118.9, 118.3, 117.8$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{15}\text{Cl}_2\text{O}_2^-$ (M-H) $^-$ 441.0527, found 441.0455.



(10-(3,4-dimethylphenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (19):

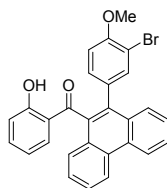
Yield 69%; 27.7 mg; white solid; mp 193–196 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=12.07$ (s, 1H), 8.78 (d, $J = 7.2$ Hz, 2H), 7.79 – 7.58 (m, 4H), 7.52 (s, 2H), 7.34 (s, 1H), 7.24 – 7.12 (m, 2H), 6.95 – 6.82 (m, 3H), 6.60 (s, 1H), 2.26 (s, 1H), 2.22 (s, 3H), 2.03 (s, 2H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=205.1, 162.4, 136.6, 136.5, 136.4, 136.1, 136.0, 135.8, 134.1, 133.5, 133.4, 132.4, 131.1, 130.6, 129.7, 129.5, 129.0, 128.7, 128.6, 127.9, 127.4, 127.3, 127.2, 127.0, 126.9, 126.2, 122.9, 122.6, 121.1, 118.8, 118.7, 117.8, 19.8, 19.5, 19.4$. HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{21}\text{O}_2^-$ (M-H) $^-$ 401.1620, found 401.1547.



(10-(3,4-dichlorophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (20):

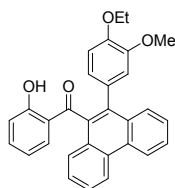
Yield 52%; 23.0 mg; white solid; mp 194–197 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.95$ (d, $J = 18.3$ Hz, 1H), 8.82 (s, 2H), 7.74 (d, $J = 17.1$ Hz, 2H), 7.66 (d, $J = 9.1$ Hz, 1H), 7.58 (s, 3H), 7.45 (d, $J = 46.7$ Hz, 2H), 7.26 (d, $J = 17.3$ Hz, 2H), 7.15 (s, 1H), 6.96 (s, 1H), 6.64 (d, $J = 18.6$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.3, 204.2, 162.6, 137.3, 137.1, 136.9, 134.3, 133.2, 133.1, 133.0, 132.3, 131.9, 130.8, 130.7, 130.4, 130.3, 130.2, 129.9, 129.2, 128.2$.

127.9, 127.7, 127.4, 127.2, 126.5, 123.0, 122.9, 120.9, 120.7, 119.1, 119.0, 118.3.
HRMS (ESI) m/z calcd for $C_{27}H_{15}Cl_2O_2^-$ (M-H) $^-$ 441.0527, found 441.0454.



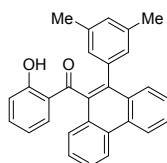
(10-(3-bromo-4-methoxyphenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (21):

Yield 64%; 30.8 mg; white solid; mp 221–223 °C; R_f = 0.3 (silica gel, PE:EA = 50:1, v/v); 1H NMR (600 MHz, $CDCl_3$) δ =12.02 (d, J = 15.9 Hz, 1H), 8.80 (d, J = 6.8 Hz, 2H), 7.82 – 7.59 (m, 4H), 7.56 (d, J = 8.0 Hz, 2H), 7.35 (dd, J = 25.4, 9.6 Hz, 2H), 7.16 (dd, J = 17.1, 7.6 Hz, 1H), 7.07 – 6.83 (m, 2H), 6.72 (d, J = 8.1 Hz, 1H), 6.63 (dd, J = 15.0, 7.1 Hz, 1H), 3.87 (d, J = 22.6 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ =204.8, 204.5, 162.5, 155.3, 137.0, 136.8, 136.0, 134.9, 134.3, 134.1, 133.2, 133.0, 131.6, 130.9, 130.7, 130.3, 130.0, 129.9, 128.4, 127.7, 127.6, 127.5, 127.3, 127.2, 126.4, 126.3, 122.9, 122.8, 120.7, 119.0, 118.9, 118.1, 118.0, 111.5, 111.4, 111.1, 110.9, 56.1. HRMS (ESI) m/z calcd for $C_{28}H_{18}BrO_3^-$ (M-H) $^-$ 481.0518, found 481.0445.



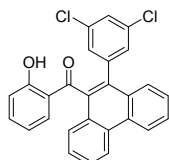
(10-(4-ethoxy-3-methoxyphenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (22):

Yield 68%; 30.4 mg; yellow solid; mp 105–108 °C; R_f = 0.3 (silica gel, PE:EA = 25:1, v/v); 1H NMR (600 MHz, $CDCl_3$) δ =12.11 (s, 1H), 8.79 (d, J = 5.4 Hz, 2H), 7.87 – 7.61 (m, 4H), 7.55 (dd, J = 14.9, 7.5 Hz, 2H), 7.38 – 7.31 (m, 1H), 7.25 – 7.18 (m, 1H), 6.99 – 6.84 (m, 3H), 6.69 (s, 1H), 6.59 (dd, J = 15.3, 7.5 Hz, 1H), 4.07 (ddd, J = 21.4, 13.9, 6.9 Hz, 2H), 3.84 (s, 1H), 3.46 (s, 2H), 1.54 – 1.39 (m, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ =205.2, 204.9, 162.5, 148.7, 148.2, 147.8, 136.7, 135.7, 133.9, 133.7, 133.3, 133.2, 131.0, 130.7, 129.7, 129.1, 128.9, 128.5, 127.7, 127.5, 127.4, 127.1, 126.2, 126.0, 123.9, 122.9, 122.7, 121.2, 118.7, 118.6, 118.1, 117.9, 114.8, 113.4, 111.9, 111.8, 64.1, 64.0, 56.0, 55.5, 14.7. HRMS (ESI) m/z calcd for $C_{30}H_{25}O_4^+$ (M+H) $^+$ 449.1675, found 449.1745.



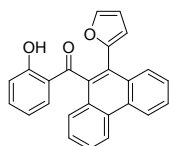
(10-(3,5-dimethylphenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (23):

Yield 79%; 31.7 mg; white solid; mp 186–188 °C; R_f = 0.3 (silica gel, PE:EA = 100:1, v/v); ^1H NMR (600 MHz, CDCl_3) δ =12.02 (s, 1H), 8.75 (d, J = 7.1 Hz, 2H), 7.68 (dd, J = 38.1, 13.8 Hz, 4H), 7.50 (s, 2H), 7.30 (s, 1H), 7.19 (d, J = 6.2 Hz, 1H), 7.02 (s, 1H), 6.94 – 6.80 (m, 2H), 6.71 (s, 1H), 6.56 (s, 1H), 2.29 (s, 3H), 2.06 (s, 3H). ^{13}C NMR (150 MHz, CDCl_3) δ =205.0, 162.3, 137.6, 136.9, 136.5, 136.2, 133.5, 133.4, 130.9, 130.6, 129.7, 129.3, 129.0, 128.5, 127.9, 127.4, 127.3, 126.9, 126.2, 122.8, 122.6, 121.1, 118.6, 117.7, 21.2, 21.0. HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{21}\text{O}_2^-$ (M-H) $^-$ 401.1620, found 401.1547.



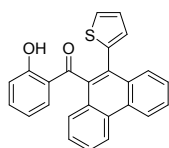
(10-(3,5-dichlorophenyl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (24):

Yield 70%; 30.9 mg; yellow solid; mp 222–224 °C; R_f = 0.3 (silica gel, PE:EA = 100:1, v/v); ^1H NMR (600 MHz, CDCl_3) δ =11.92 (s, 1H), 8.88 – 8.74 (m, 2H), 7.79 – 7.71 (m, 2H), 7.68 (d, J = 8.0 Hz, 1H), 7.62 – 7.53 (m, 3H), 7.41 (t, J = 7.7 Hz, 1H), 7.32 (d, J = 17.4 Hz, 2H), 7.14 (d, J = 8.0 Hz, 1H), 7.02 (s, 1H), 6.97 (d, J = 8.5 Hz, 1H), 6.66 (t, J = 7.4 Hz, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ =204.1, 162.5, 139.8, 137.2, 135.0, 134.3, 132.9, 130.6, 130.0, 129.8, 128.5, 128.2, 127.9, 127.7, 127.6, 127.4, 127.2, 126.5, 123.0, 122.9, 120.8, 119.1, 118.2. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{15}\text{Cl}_2\text{O}_2^-$ (M-H) $^-$ 441.0527, found 441.0455.



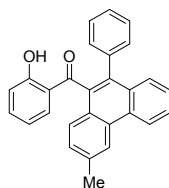
(10-(furan-2-yl)phenanthren-9-yl)(2-hydroxyphenyl)methanone (25):

Yield 74%; 26.9 mg; yellow solid; mp 203–205 °C; R_f = 0.3 (silica gel, PE:EA = 100:1, v/v); ^1H NMR (600 MHz, CDCl_3) δ =12.14 (s, 1H), 8.77 (dd, J = 8.2, 3.1 Hz, 2H), 7.97 (d, J = 8.2 Hz, 1H), 7.72 (dt, J = 15.3, 6.0 Hz, 3H), 7.63 (t, J = 7.6 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.37 (dd, J = 21.3, 13.1 Hz, 2H), 7.12 (d, J = 8.0 Hz, 1H), 6.98 (d, J = 8.4 Hz, 1H), 6.58 (t, J = 7.6 Hz, 1H), 6.49 (d, J = 2.9 Hz, 1H), 6.35 (s, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ =204.4, 162.5, 149.2, 143.2, 136.7, 135.2, 132.9, 130.8, 130.4, 129.9, 128.3, 127.9, 127.8, 127.5, 127.4, 127.3, 126.5, 125.7, 123.0, 122.8, 120.7, 118.8, 117.9, 113.3, 111.1. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{15}\text{O}_3^-$ (M-H) $^-$ 363.1099, found 363.1027.



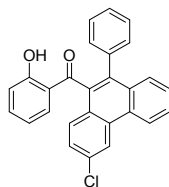
(2-hydroxyphenyl)(10-(thiophen-2-yl)phenanthren-9-yl)methanone (26):

Yield 41%; 15.6 mg; yellow solid; mp 208–211 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =12.05 (s, 1H), 8.77 (d, $J = 8.3$ Hz, 2H), 7.92 (d, $J = 6.4$ Hz, 1H), 7.70 (ddd, $J = 19.5, 13.0, 8.1$ Hz, 3H), 7.56 (dt, $J = 31.9, 7.4$ Hz, 2H), 7.36 (dd, $J = 21.6, 14.3$ Hz, 2H), 7.18 (d, $J = 7.2$ Hz, 1H), 7.07 – 6.88 (m, 3H), 6.61 (t, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =204.4, 162.4, 136.9, 136.7, 135.8, 133.3, 131.3, 130.5, 130.1, 128.6, 128.3, 127.6, 127.5, 127.4, 127.3, 127.2, 126.8, 126.5, 122.9, 122.6, 120.7, 118.9, 117.9. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{15}\text{O}_2\text{S}^-$ (M-H) $^-$ 379.0871, found 379.0798.



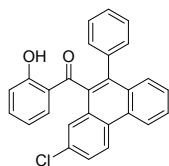
(2-hydroxyphenyl)(6-methyl-10-phenylphenanthren-9-yl)methanone (27):

Yield 57%; 22.0 mg; yellow solid; mp 166–168 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ =12.01 (s, 1H), 8.79 (d, $J = 8.0$ Hz, 1H), 8.58 (s, 1H), 7.69 (t, $J = 7.4$ Hz, 1H), 7.62 (d, $J = 7.9$ Hz, 1H), 7.58 – 7.49 (m, 2H), 7.37 (ddd, $J = 25.1, 15.2, 6.7$ Hz, 4H), 7.26 (t, $J = 6.8$ Hz, 1H), 7.17 (dd, $J = 12.6, 7.1$ Hz, 2H), 7.12 (d, $J = 7.2$ Hz, 1H), 6.88 (d, $J = 8.3$ Hz, 1H), 6.58 (t, $J = 7.4$ Hz, 1H), 2.61 (s, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ =205.0, 162.4, 137.0, 136.9, 136.6, 134.9, 133.7, 133.3, 131.4, 131.0, 130.4, 130.0, 129.9, 129.1, 128.3, 127.7, 127.6, 127.3, 126.9, 126.4, 126.1, 122.6, 121.0, 118.8, 117.9, 22.1. HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{19}\text{O}_2^-$ (M-H) $^-$ 387.1463, found 387.1390.



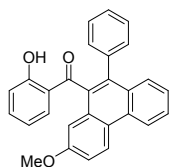
(6-chloro-10-phenylphenanthren-9-yl)(2-hydroxyphenyl)methanone (28):

Yield 43%; 17.5 mg; yellow solid; mp 183–185 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ =11.94 (s, 1H), 8.80 (s, 1H), 8.75 (d, $J = 8.3$ Hz, 1H), 7.78 (t, $J = 7.6$ Hz, 1H), 7.68 (d, $J = 8.1$ Hz, 1H), 7.62 (t, $J = 7.9$ Hz, 2H), 7.53 (d, $J = 8.7$ Hz, 1H), 7.44 (d, $J = 3.9$ Hz, 2H), 7.39 (t, $J = 7.9$ Hz, 1H), 7.34 – 7.30 (m, 1H), 7.23 – 7.13 (m, 3H), 6.93 (d, $J = 8.4$ Hz, 1H), 6.64 (t, $J = 7.6$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ =204.3, 162.5, 136.9, 136.5, 136.3, 133.4, 133.3, 133.2, 131.3, 131.1, 129.9, 129.7, 128.4, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 126.9, 122.8, 122.7, 120.9, 118.9, 118.0. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{ClO}_2^-$ (M-H) $^-$ 407.0917, found 407.0844.



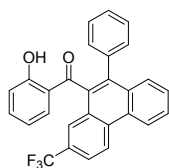
(7-chloro-10-phenylphenanthren-9-yl)(2-hydroxyphenyl)methanone (29):

Yield 72%; 29.3 mg; yellow solid; mp 184–186 °C; $R_f = 0.3$ (silica gel, PE:EA = 100:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=11.92$ (s, 1H), 8.69 (t, $J = 9.5$ Hz, 2H), 7.71 (t, $J = 7.4$ Hz, 1H), 7.68 – 7.55 (m, 3H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.38 (s, 2H), 7.33 (t, $J = 7.7$ Hz, 1H), 7.26 (s, 1H), 7.15 (dt, $J = 17.2, 7.4$ Hz, 3H), 6.88 (d, $J = 8.4$ Hz, 1H), 6.59 (t, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.0, 162.5, 137.2, 137.0, 136.4, 133.4, 133.1, 132.8, 131.1, 130.7, 130.2, 129.9, 129.5, 128.4, 128.2, 128.0, 127.9, 127.8, 127.7, 127.3, 125.2, 124.5, 122.6, 120.7, 118.9, 118.0$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{16}\text{ClO}_2^-$ (M-H) $^-$ 407.0917, found 407.0845.



(2-hydroxyphenyl)(7-methoxy-10-phenylphenanthren-9-yl)methanone (30):

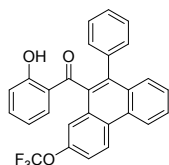
Yield 70%; 28.3 mg; yellow solid; mp 149–151 °C; $R_f = 0.3$ (silica gel, PE:EA = 50:1, v/v); $^1\text{H NMR}$ (600 MHz, CDCl_3) $\delta=12.00$ (s, 1H), 8.74 – 8.59 (m, 2H), 7.72 – 7.55 (m, 2H), 7.45 (t, $J = 7.5$ Hz, 1H), 7.43 – 7.35 (m, 2H), 7.36 – 7.28 (m, 2H), 7.26 (t, $J = 7.2$ Hz, 1H), 7.23 – 7.15 (m, 2H), 7.11 (d, $J = 7.5$ Hz, 1H), 7.00 (d, $J = 1.8$ Hz, 1H), 6.88 (dd, $J = 8.2, 4.7$ Hz, 1H), 6.59 (t, $J = 7.5$ Hz, 1H), 3.75 (s, 2H), 3.71 (s, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) $\delta=204.9, 162.4, 158.7, 158.5, 136.9, 136.8, 136.7, 136.6, 135.3, 134.3, 133.3, 133.2, 132.4, 131.3, 130.8, 129.9, 129.7, 128.4, 128.2, 127.8, 127.7, 127.6, 127.5, 127.4, 127.3, 127.2, 126.4, 126.3, 126.0, 125.0, 124.5, 124.3, 124.1, 122.4, 122.1, 121.0, 120.9, 118.8, 117.8, 117.4, 117.3, 108.6, 107.0, 55.3, 55.2$. HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{19}\text{O}_3^-$ (M-H) $^-$ 403.1412, found 403.1340.



(2-hydroxyphenyl)(10-phenyl-7-(trifluoromethyl)phenanthren-9-yl)methanone (31):

Yield 48%; 21.1 mg; yellow solid; mp 207–209 °C; $R_f = 0.3$ (silica gel, PE:EA = 50:1, v/v); $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta=11.91$ (s, 1H), 8.88 (dd, $J = 36.4, 8.6$ Hz, 2H), 7.93 (d, $J = 7.7$ Hz, 2H), 7.79 – 7.68 (m, 2H), 7.66 – 7.60 (m, 1H), 7.38 (dt, $J = 19.3, 8.7$ Hz, 4H), 7.21 (d, $J = 7.4$ Hz, 1H), 7.14 (t, $J = 8.7$ Hz, 2H), 6.91 (d, $J = 8.4$ Hz, 1H), 6.61 (t, $J = 7.6$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta=204.2, 162.5, 137.0, 135.8, 135.2, 133.1, 132.8, 131.3, 130.5, 129.8, 129.2$ (q, $J = 21.0$ Hz, $^2J_{\text{CF}}$), 128.6, 128.3, 128.0, 127.8, 126.6, 126.1 (q, $J = 267.0$ Hz, $^1J_{\text{CF}}$), 125.0 (q, $J = 6.0$ Hz, $^3J_{\text{CF}}$),

123.7, 123.4 (q, $J = 4.5$ Hz, $^3J_{CF}$), 120.8, 118.9, 118.1. HRMS (ESI) m/z calcd for $C_{28}H_{16}F_3O_2^-$ (M-H) $^-$ 441.1181, found 441.1108.



(2-hydroxyphenyl)(10-phenyl-7-(trifluoromethoxy)phenanthren-9-yl)methanone (32):

Yield 54%; 24.7 mg; yellow solid; mp 142–145 °C; $R_f = 0.3$ (silica gel, PE:EA = 50:1, v/v); 1H NMR (600 MHz, $CDCl_3$) $\delta = 11.89$ (s, 1H), 8.82 (d, $J = 9.1$ Hz, 1H), 8.74 (d, $J = 8.3$ Hz, 1H), 7.75 (t, $J = 7.5$ Hz, 1H), 7.67 (d, $J = 8.1$ Hz, 1H), 7.56 (t, $J = 7.6$ Hz, 2H), 7.48 (s, 1H), 7.40 (d, $J = 3.7$ Hz, 2H), 7.35 (t, $J = 7.7$ Hz, 1H), 7.28 (d, $J = 3.3$ Hz, 1H), 7.21 – 7.11 (m, 3H), 6.90 (d, $J = 8.4$ Hz, 1H), 6.61 (t, $J = 7.4$ Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) $\delta = 203.9, 162.6, 148.0, 137.7, 137.0, 136.4, 133.2, 133.0, 131.1, 130.8, 130.2, 129.8, 129.4, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.5, 125.0, 122.7, 120.7, 120.4$ (q, $J = 258.0$ Hz, $^1J_{CF}$), 120.2, 118.9, 118.1, 117.5. HRMS (ESI) m/z calcd for $C_{28}H_{16}F_3O_3^-$ (M-H) $^-$ 457.1130, found 457.1057.

4. Crystallographic data and molecular structure of compounds

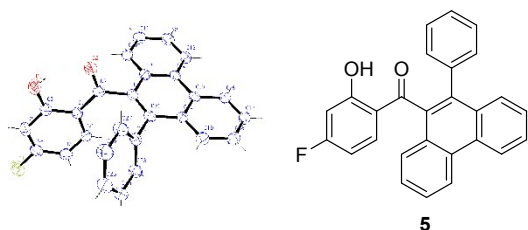


Figure 1. X-ray crystal structure of **5**

Crystal Data for Compound **5**: CCDC 2024940 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic.

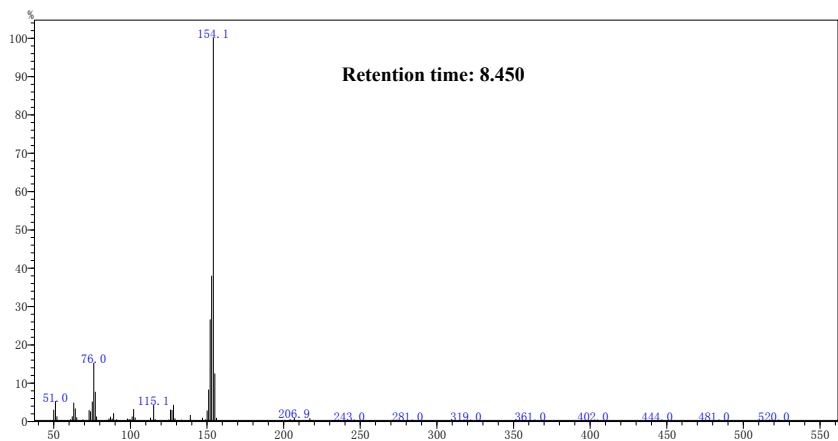


Figure 2. Detection of the biphenyl

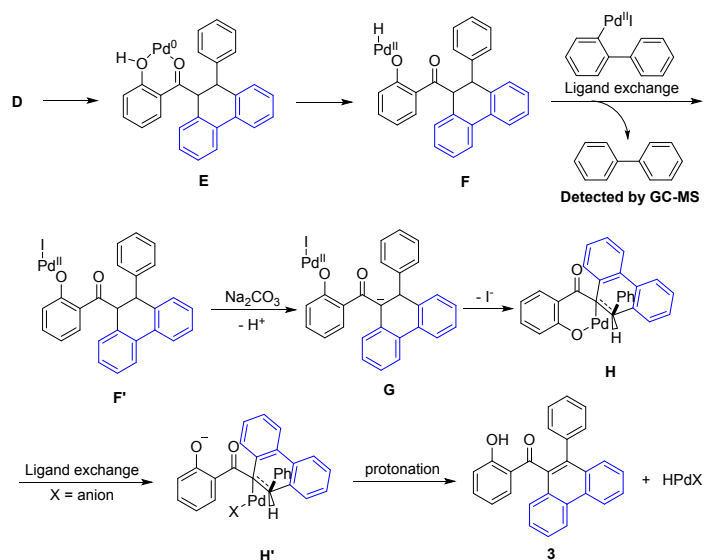


Figure 3. Possible pathway of dehydrogenation

7.¹H and ¹³C NMR spectra of compounds

