Synthesis of Dibenzo[*b,d*]pyran-6-ones based on [3+3] Cyclizations of 1,3-Bis(Silyl Enol Ethers) with 3-Silyloxy-2-en-1-ones

**Ibrar Hussain,^a Van Thi Hong Nguyen,^a Mirza Arfan Yawer,^a Tuan Thanh Dang,^a
Christine Fischer,^b Helmut Reinke,^a and Peter Langer*^{a, b}**

^a Institut für Chemie, Universität Rostock,

Albert-Einstein-Str. 3a, 18059 Rostock, Germany

Fax: +381 4986412, E-mail: peter.langer@uni-rostock.de

^b Leibniz-Institut für Katalyse e. V. an der Universität Rostock,

Albert-Einstein-Str. 29a, 18059 Rostock, Germany

Supporting Information

Contents:

S1: Experimental procedures

S 46: Copies of NMR spectra

S 248: ORTEP plot

Experimental Section

General Comments. All solvents were dried by standard methods and all reactions were carried out under an inert atmosphere. For ^1H and ^{13}C NMR spectra the deuterated solvents indicated were used. Mass spectrometric data (MS) were obtained by electron ionization (EI, 70 eV), chemical ionization (CI, H_2O) or electrospray ionization (ESI). For preparative scale chromatography, silica gel (60-200 mesh) was used. Melting points are uncorrected.

General procedure for the synthesis of salicylates **3 and **12**:** To a CH_2Cl_2 solution of silyl enol ether **2** (1.0 equiv.) and 1,3-bis(silyl enol ether) **1** (1.0 equiv.) was dropwise added TiCl_4 (1.0 equiv.) at $-78\text{ }^\circ\text{C}$ under argon atmosphere. The solution was stirred at $-78\text{ }^\circ\text{C}$ for 30 min and then allowed to warm to $20\text{ }^\circ\text{C}$ during 18 h. To the solution was added a saturated aqueous solution of NaHCO_3 . The organic layer was separated and the aqueous layer was repeatedly extracted with CH_2Cl_2 . The combined organic extracts were dried (Na_2SO_4) and filtered. The filtrate was concentrated *in vacuo* and the residue was purified by chromatography (silica gel, *n*-hexane/EtOAc) to give salicylates **3**. The synthesis of **3a** was previously reported.^{11a}

Methyl 4,5,6-trimethyl-2-hydroxybenzoate (3b). Starting with 1-methoxy-1,3-bis-(trimethylsilyloxy)buta-1,3-diene **1a** (1.04 g, 4.0 mmol), **2b** (0.744 g, 4.0 mmol) and TiCl_4 (0.760 g, 4.0 mmol) in CH_2Cl_2 (8 mL), **3b** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 20:1) as a colorless solid (0.395 g, 51%), mp. = $66\text{ }^\circ\text{C}$. ^1H NMR (CDCl_3 , 300 MHz): δ = 2.13 (s, 3 H, CH_3), 2.27 (s, 3 H, CH_3), 2.44 (s, 3 H, CH_3), 3.94 (s, 3 H, CH_3), 6.69 (s, 1 H, CH), 10.84 (s, 1

H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{C} = 15.2, 18.8, 21.5, 51.5 (CH_3), 111.3 (C), 116.2 (CH), 127.2, 138.1, 143.8, 159.0, 171.9 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3240 (w), 3002 (w), 2956 (s), 1657 (s), 1209 (s), 1236 (s), 1156 (s), 1065 (s), 805 (w). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 214 (4.38), 252 (3.88), 314 (3.58). MS (EI, 70 eV): m/z (%) = 193 (M^+ , 35), 162 (100), 134 (34), 28 (16). HRMS (EI, 70 eV). The exact molecular mass m/z = 194.0913 ± 2 ppm [M^+] for $\text{C}_{11}\text{H}_{14}\text{O}_3$ was confirmed by HRMS (EI, 70 eV). Anal. calcd. for $\text{C}_{11}\text{H}_{14}\text{O}_3$ (194.227): C 68.02, H 7.27; found: C 68.13, H 8.21.

Methyl 4,6-dimethyl-5-ethyl-2-hydroxybenzoate (3c). Starting with 1-methoxy-1,3-bis-(trimethylsilyloxy)buta-1,3-diene **1a** (0.52 g, 2.0 mmol), **2c** (0.400 g, 2.0 mmol) and TiCl_4 (0.38 g, 2.0 mmol) in CH_2Cl_2 (5 mL), **3c** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 20:1) as a colorless solid (0.185 g, 45%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.07 (t, J = 7.2 Hz, 3 H, CH_3), 2.47 (s, 3 H, CH_3), 2.61 (s, 3 H, CH_3), 2.63 (q, J = 7.1 Hz, 2 H, CH_2), 3.94 (s, 3 H, OCH_3), 6.68 (s, 1 H, CH), 10.54 (s, 1 H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{C} = 13.7, 18.1, 20.7 (CH_3), 22.3 (CH_2), 51.9 (CH_3), 111.6 (C), 116.8 (CH), 133.3, 137.8, 143.6, 159.3, 172.1 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3022 (m), 2969 (s), 1661 (s), 1445 (s), 1354 (s), 1228 (s), 1156 (s), 1074 (s). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 216 (4.35), 252 (3.78), 318 (3.31). MS (EI, 70 eV): m/z (%) = 208 (M^+ , 16), 176 (53), 161 (53), 28 (100). HRMS (EI, 70 eV): calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_3$ [M^+]: m/z = 208.1099; found: m/z = 208.1099 ± 2 ppm. Anal. calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_3$ (208.254): C 69.21, H 7.74; found: C 68.9, H 7.77.

Ethyl 4-methyl-6-phenyl-2-hydroxybenzoate (3d). The synthesis of **3d** has been previously reported.^{11a} Starting with 1-ethoxy-1,3-bis-(trimethylsilyloxy)buta-1,3-diene **1b** (1.300 g, 5.0

mmol), TiCl_4 (0.940 g, 5.0 mmol) CH_2Cl_2 (15 mL) and **2d** (1.170 g, 5.0 mmol), **3d** was isolated (0.399 g, 31%) as a colourless viscous oil by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 20:1). ^1H NMR (300 MHz, CDCl_3): δ = 0.74 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 2.34 (t, J = 7.1 Hz, 3 H, CH_3), 3.97 (q, J = 7.2 Hz, 2 H, OCH_2CH_3), 6.61 (m, J = 0.6 Hz, 1 H, Ar), 6.82 (m, J = 0.9 Hz, 1 H, Ar), 7.19-7.22 (m, 2 H, Ar), 7.29-7.32 (m, 2 H, Ar), 7.33-7.35 (m, 1 H, Ar), 10.92 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 12.9, 21.6 (CH_3), 60.8 (CH_2), 109.5, 116.8 (C), 116.2, 123.8, 126.6 (CH), 127.5 (2CH), 128.1 (2CH), 143.2, 144.7, 144.9, 161.8, 170.9 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3424 (w), 2986 (m), 1652 (s), 1617 (s), 1574 (m), 1445 (m), 1402 (s), 1375 (s), 1321 (s), 1276 (s), 1221 (s), 1105 (m), 1013 (m), 852 (m), 767 (s), 705 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 218.3 (4.48), 251.2 (3.86), 311.6 (3.72). MS (EI, 70 eV): m/z (%) = 257 ($[\text{M}^+ + 1]$, 6), 256 (M^+ , 34), 210 (100), 182 (28), 152 (11), 28 (7). Anal. calcd. for $\text{C}_{16}\text{H}_{16}\text{O}_3$: C 74.97, H 6.29; found C 74.78, H 6.28.

Methyl 2-hydroxy-3-methoxy-4,6-dimethylbenzoate (3e). Starting with **1c** (1.0 equiv., 0.870 g, 3.0 mmol), **2a** (1.0 equiv., 0.516 g, 3.0 mmol), CH_2Cl_2 (6 mL), and TiCl_4 (1.0 equiv., 0.560 g, 3.0 mmol), **3e** was isolated (0.312 g, 50%) by column chromatography (silica gel, *n*-hexane/EtOAc = 20:1) as a colourless viscous oil. ^1H NMR (300 MHz, CDCl_3): δ = 2.25 (s, 3 H, CH_3), 2.45 (s, 3 H, CH_3), 3.83 (s, 3 H, CH_3O), 6.52 (s, 1 H, CH), 11.46 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 16.1, 23.6, 52.1, 59.9 (CH_3), 111.0, 124.2, 135.4, 137.3, 144.7, 156.2, 172.3 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2984 (s), 2936 (s), 1656 (s), 1613 (s), 1573 (s), 1446 (s), 1402 (s), 1367 (s), 1304 (s), 1252 (s), 1206 (s), 1070 (s), 1003 (s), 954 (s), 859 (s), 809 (s). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 214.3 (4.39), 253.6 (3.99), 315.5 (3.49). MS (EI, 70 eV): m/z (%) = 210 (M^+ , 40), 178 (99), 149 (100), 135 (39), 106 (4), 77 (21), 28 (51). HRMS (ESI): calcd. for $\text{C}_{11}\text{H}_{15}\text{O}_3$ ($[\text{M} + 1]^+$): 211.09740;

found: 211.09674.

Ethyl 5,6,7,8-tetrahydro-2-hydroxynaphthalene-1-carboxylate (3f). Starting with **1a** (1.300 g, 5.0 mmol), TiCl_4 (0.94 g, 5.0 mmol), CH_2Cl_2 (15 mL), and **2e** (0.990 g, 5.0 mmol), **3f** was isolated (0.320 g, 30%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30 :1 $\rightarrow$ 20:1) as a colourless viscous oil. ^1H NMR (300 MHz, CDCl_3): δ = 1.37 (t, J = 7.1 Hz, 3 H, OCH_2CH_3), 2.03 (m, 4 H, CH_2), 2.82 (t, J = 6.6 Hz, 4 H, CH_2), 4.35 (q, J = 7.1 Hz, 2 H, OCH_2CH_3), 6.83 (t, J = 7.6 Hz, 1 H, Ar), 7.60 (m, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 14.3 (CH_3), 21.7, 25.2 (2 CH_2), 60.6 (CH_2), 119.1 (CH), 119.7, 123.4, 129.3 (C), 137.7 (CH), 154.9, 166.2 (C). MS (EI, 70 eV): m/z = 221 ($[\text{M}+1]^+$, 3), 220 (M^+ , 21), 174 (100), 146 (14), 118 (10), 28 (6). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2982 (s), 2934 (s), 2862 (s), 1726 (m), 1656 (s), 1597 (s), 1468 (s), 1402 (s), 1433 (s), 1374 (s), 1317 (s), 1223 (s), 1180 (s), 1133 (m), 1046 (m), 1010 (s), 820 (m), 739 (s). UV-VIS (CH_3CN , nm): λ_{max} (log ϵ) = 212.9 (4.25), 248.8 (3.74), 319.9 (3.47).

Methyl 5,6,7,8-tetrahydro-3-hydroxy-4-methylnaphthalene-12-carboxylate (3g). Starting with 1-methoxy-1,3-bis(trimethylsiloxy)buta-1,3-dien **1a** (1.300 g, 5.0 mmol), TiCl_4 (0.940 g, 5.0 mmol), CH_2Cl_2 (12 mL), and **2f** (1.060 g, 5.0 mmol), **3g** was isolated (0.352 g, 32%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 20:1) as a colourless solid. ^1H NMR (300 MHz, CDCl_3): δ = 1.66 - 1.80 (m, 4 H, 2 CH_2), 2.19 (s, 3 H, CH_3), 2.57 (m, J = 6.5 Hz, 2 H, CH_2), 2.98 (t, J = 6.3 Hz, 2 H, CH_2), 3.93 (s, 3 H, CH_3O), 6.69 (s, 1 H, CH, Ar), 10.84 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 20.4 (CH_3), 22.5, 23.1, 27.0 (CH_2), 51.9 (CH_3), 110.4 (C), 116.5 (CH), 127.7, 139.3, 144.7, 159.9, 172.2 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3429 (m), 2929 (s), 1657 (s), 1608 (s), 1443 (s), 1318 (s), 1234 (s), 1075 (m), 801 (m), 756 (m). UV-VIS (CH_3CN , nm): λ_{max}

(log ϵ) = 214.5 (4.32), 253.6 (3.92), 317.8 (3.49). MS (EI, 70 eV): m/z (%) = 221 ([M+1]⁺, 3), 220 (M⁺, 22), 189 (16), 188 (100), 160 (16), 132 (13), 28 (67). Anal. calcd. for C₁₃H₁₆O₃: C 70.89, H 7.32; found C 70.87, H 7.35.

Methyl 9,10-dihydro-3-hydroxy-1-methylphenanthrene-4-carboxylate (3h). Starting with 1-methoxy-1,3-bis(trimethylsiloxy)buta 1,3-diene **1a** (1.82 g, 7.0 mmol), **2h** (1.82 g, 7.0 mmol), TiCl₄ (1.32 g, 7.0 mmol) and CH₂Cl₂ (15 mL), **3h** was isolated (0.907 g, 48%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 → 20:1) as a colourless solid. ¹H NMR (300 MHz, CDCl₃): δ = 2.23 (s, 3 H, CH₃), 2.63 (m, J = 6.0 Hz, 2 H, CH₂), 2.81 (m, J = 7.3 Hz, 2 H, CH₂), 3.65 (s, 3 H, OCH₃), 7.18 - 7.21 (m, 2 H, Ar), 7.25-7.32 (m, 2 H, Ar). MS (EI, 70 eV): m/z (%) = 269 ([M+1]⁺, 5), 268 (M⁺, 29), 236 (100), 208 (28), 207 (35), 165 (30), 28 (7). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3415 (w, br), 2950 (w), 1723 (w), 1667 (m), 1609 (s), 1566 (s), 1435 (s), 1206 (m), 848 (m). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 203.5 (4.31), 229.9 (4.12), 300.9 (3.77), 338.7 (4.03).

Ethyl 3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12a). Starting with the bis(silyl-enol ether) (0.659 g, 2.4 mmol), TiCl₄ (0.455 g, 2.4 mmol), CH₂Cl₂ (8 mL) and the mono(silyl enol ether) (0.582 g, 2.2 mmol), **12a** was isolated (0.293 g, 47%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 → 20:1) as a colorless solid (mp = 91-93 °C). ¹H NMR (250 MHz, CDCl₃): δ = 0.78 (t, J = 7.2 Hz, 3 H, CH₃), 2.34 (s, 3 H, CH₃), 3.70 (s, 3 H, OCH₃), 3.97 (q, J = 7.3 Hz, 2 H, OCH₂CH₃), 6.87-6.58 (m, 1 H, Ar), 6.80-6.81 (m, 1 H, Ar), 6.85 (dd, J = 8.1 Hz, J = 1.2 Hz, 1 H, Ar), 6.99 (ddd, J = 7.3 Hz, J = 7.6 Hz, J = 1.2 Hz, 1 H, Ar), 7.15 (dd, J = 7.3 Hz, J = 1.5 Hz, 1 H, Ar), 7.32 (ddd, J = 8.6 Hz, J = 7.3 Hz, J = 1.8 Hz, 1 H, Ar), 10.93 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 13.5, 22.2 (CH₃), 55.6 (OCH₂CH₃), 60.8 (OCH₃),

109.9, 117.2, 120.5 (CH), 122.8 (C) 124.2, 128.4, 129.5 (CH), 140.8, 145.0, 151.6, 156.4, 161.4, 171.2 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3432(m), 2924 (s), 1655 (s), 1616 (m), 1462 (s), 1278 (m), 1217 (m), 1197 (m), 1107 (m), 1026 (m), 871 (w), 759 (m). MS (EI, 70 eV): m/z (%) = 286 (M^+ , 40), 255 (2), 240 (100), 211 (15), 197 (22), 169 (11), 152 (7), 141 (7), 115 (9), 77 (2). HRMS (EI): Calcd. for $\text{C}_{17}\text{H}_{18}\text{O}_4$: 286.11996; found: 286.120041.

Methyl 3-hydroxy-2'-methoxy-4,5-dimethyl[1,1'-biphenyl]-2-carboxylate (12b). Starting with the bis(silyl-enol ether) (0.604 g, 2.2 mmol), TiCl_4 (0.411 g, 2.2 mmol), CH_2Cl_2 (8 mL) and the mono(silyl enol ether) (0.529 g, 2.0 mmol), **12b** was isolated (0.117 g, 27%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 20:1) as a yellow viscous oil. ^1H NMR (250 MHz, CDCl_3): δ = 2.09 (s, 3 H, CH_3), 2.18 (s, 3 H, CH_3), 3.36 (s, 3 H, OCH_3), 3.38 (s, 3 H, OCH_3), 6.60 (s, 1 H, Ar), 6.86 (dd, J = 8.2 Hz, J = 0.9 Hz, 1 H, Ar), 6.99 (ddd, J = 8.1 Hz, J = 7.3 Hz, J = 0.9 Hz, 1 H, Ar), 7.19 (dd, J = 7.3 Hz, J = 1.8 Hz, 1 H, Ar), 7.31 (ddd, J = 8.2 Hz, J = 7.3 Hz, J = 1.8 Hz, 1 H, Ar), 11.07 (s, 1 H, OH). ^{13}C NMR (62 MHz, CDCl_3): δ = 20.5, 30.3 (CH_3), 51.4, 55.4 (OCH_3), 109.6, 120.4, 124.2 (CH), 125.5 (C) 128.2, 129.3 (CH), 132.2, 137.0, 143.2, 147.6, 156.1, 158.9, 171.9 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3000 (m), 2954 (m), 2951 (m), 2854 (m), 1657 (s), 1611 (m), 1439 (s), 1393 (s), 1269 (s), 1242 (s), 1195 (m), 1025 (s), 918 (w), 807 (m), 761 (s), 637 (w), 578 (w). MS (EI, 70 eV): m/z (%) = 286 (M^+ , 35), 254 (64), 239 (100), 223 (40), 211 (22), 181 (27), 165 (16), 152 (16), 131 (27), 115 (12), 77 (18). HRMS (EI): Calcd. for $\text{C}_{17}\text{H}_{18}\text{O}_4$: 286.11996; found: 286.119424.

Methyl 4-ethyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12c). Starting with the bis(silyl-enol ether) (0.692 g, 2.4 mmol), TiCl_4 (0.458 g, 2.4 mmol), CH_2Cl_2 (8 mL) and

the mono(silyl enol ether) (0.582 g, 2.2 mmol), **12c** was isolated (0.226 g, 34%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 → 20:1) as a colorless solid (mp = 123-125°C). ¹H NMR (250 MHz, CDCl₃): δ = 1.12 (t, *J* = 7.9 Hz, 3 H, CH₃), 2.28 (s, 3 H, CH₃), 2.68 (q, *J* = 5.8 Hz, 2 H, CH₂), 3.42 (s, 3 H, OCH₃), 3.65 (s, 3 H, OCH₃), 6.53 (s, 1 H, Ar), 6.80 (dd, *J* = 8.5 Hz, *J* = 1.2 Hz, 1 H, Ar), 6.94 (ddd, *J* = 8.5 Hz, *J* = 7.6 Hz, *J* = 1.2 Hz, 1 H, Ar), 7.14 (dd, *J* = 7.3 Hz, *J* = 1.5 Hz, 1 H, Ar), 7.24 (ddd, *J* = 8.4 Hz, *J* = 6.2 Hz, *J* = 2.1 Hz, 1 H, Ar), 10.92 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 13.2 (CH₃), 19.7 (CH₂), 19.8 (CH₃), 51.6, 55.6 (OCH₃), 109.7, 120.6 (CH), 123.5 (C), 124.7, 128.4, 129.5 (CH), 130.0, 137.3, 142.6, 144.3, 156.3, 159.0, 172.1 (C). IR (Nujol, cm⁻¹): $\tilde{\nu}$ = 3433 (w), 2954 (m), 2927 (m), 2870 (w), 1659 (s), 1605 (m), 1438 (s), 1278 (s), 1239 (s), 1197 (m), 1102 (m), 1026 (m), 812 (m), 756 (s), 639 (w), 575 (w). GC-MS (EI, 70 eV): *m/z* (%) = 300.2 (M⁺, 71), 268 (78), 253 (100), 250 (40), 239 (11), 237 (71), 225 (16), 223 (7), 181 (17), 152 (12), 115 (7), 77 (5). HRMS (EI): Calcd. for C₁₈H₂₀O₄: 300.13561; found: 300.135117.

Ethyl 4-hexyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12d). Starting with the bis(silyl-enol ether) (0.861 g, 2.4 mmol), TiCl₄ (0.448 g, 2.4 mmol), CH₂Cl₂ (8 mL) and the mono(silyl enol ether) (0.582 g, 2.2 mmol), **12d** was isolated (0.216 g, 26%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 → 20:1) as a yellow oil. ¹H NMR (250 MHz, CDCl₃): δ = 0.69 (t, *J* = 7.0 Hz, 3 H, CH₃), 0.83 (t, *J* = 6.4 Hz, 3 H, CH₃), 1.17-1.28 (m, 8 H, CH₂), 2.24 (s, 3 H, CH₃), 2.59 (t, *J* = 8.2 Hz, 2 H CH₂), 3.62 (s, 3 H, OCH₃), 3.89 (q, *J* = 7.3 Hz, 2 H, OCH₂CH₃) 6.57 (s, 1 H, Ar), 6.84 (dd, *J* = 7.3 Hz, *J* = 0.9 Hz, 1 H, Ar), 6.97 (ddd, *J* = 7.3 Hz, *J* = 6.4 Hz, *J* = 0.9 Hz, 1 H, Ar), 7.16 (dd, *J* = 7.3 Hz, *J* = 1.8 Hz, 1 H, Ar), 7.29 (ddd, *J* = 7.3 Hz, *J* = 6.4 Hz, *J* = 1.8 Hz, 1 H, Ar), 10.08 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 12.9, 14.0, 19.7

(CH₃), 22.6, 26.3, 29.6, 31.2, 31.8 (CH₂), 55.3 (OCH₃), 60.5 (OCH₂CH₃), 109.6 (CH), 110.4 (C), 120.3, 124.3 (CH), 127.9 (C), 128.7, 129.3 (CH), 132.5, 137.2, 142.4, 156.2, 159.1, 171.5 (C). IR (Nujol, cm⁻¹): $\tilde{\nu}$ = 2955 (m), 2926 (s), 2856 (m), 1656 (s), 1610 (m), 1462 (m), 1389 (m), 1270 (s), 1244 (m), 1180 (m), 1109 (m), 1029 (m), 750 (m). GC-MS (EI, 70 eV): m/z (%) = 370 (M⁺, 64), 324 (45), 309 (68), 307 (35), 254 (60), 253 (54), 239 (100), 223 (43), 181 (15), 115 (4), 77 (2). HRMS (EI): Calcd. for C₂₃H₃₀O₄ : 370.21386; found: 370.213501.

Methyl 4-heptyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12e). Starting with the bis(silyl-enol ether) (0.789 g, 2.2 mmol), TiCl₄ (0.407 g, 2.2 mmol), CH₂Cl₂ (8 mL) and the mono(silyl enol ether) (0.529 g, 2.0 mmol), **12e** was isolated (0.185 g, 25%) by column chromatography (silica gel, *n*-hexane/EtOAc = 40:1 → 20:1) as a yellow oil. ¹H NMR (250 MHz, CDCl₃): δ = 0.72 (t(br), J = 6.5 Hz, 3 H, CH₃), 1.09-1.12 (m, 10 H, CH₂), 2.14 (s, 3 H, CH₃), 2.49 (t, J = 7.9 Hz, 2 H, CH₂), 3.28 (s, 3 H, OCH₃), 3.50 (s, 3 H, OCH₃), 6.60 (s, 1 H, Ar), 6.70 (ddd, J = 8.4 Hz, J = 7.3 Hz, J = 0.9 Hz, 1 H, Ar), 6.86 (dd, J = 7.6 Hz, J = 0.6 Hz, 1 H, Ar), 7.20 (dd, J = 7.3 Hz, J = 1.8 Hz, 1 H, Ar), 7.30 (ddd, J = 8.2 Hz, J = 7.7 Hz, J = 1.8 Hz, 1 H, Ar), 10.82 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 15.3, 21.1 (CH₃), 23.9, 27.6, 30.2, 30.4, 32.9, 33.1 (CH₂), 52.6, 56.6 (OCH₃), 110.8 (CH), 111.6 (C), 123.9, 125.7 (CH), 129.4 (C), 129.9, 130.6 (CH), 133.5, 135.3, 138.3, 143.8, 157.3, 160.2 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2954 (s), 2927 (s), 2856 (s), 1663 (s), 1610 (m), 1458 (m), 1437 (m), 1392 (m), 1269 (s), 1244 (m), 1197 (m), 1145 (m), 1109 (m), 1030 (m), 815 (w). GC-MS (EI, 70 eV): m/z (%) = 370 (M⁺, 65), 339 (14), 338 (48), 323 (66), 281 (8), 254 (60), 253 (60), 239 (100), 223 (43), 211 (9), 181 (16), 165 (11), 152 (8), 115 (4), 77 (2), 43 (8). HRMS (EI): Calcd. for C₂₃H₃₀O₄ : 370.21386; found: 370.21397.

Ethyl 3-hydroxy-2'-methoxy-5-methyl-4-octyl[1,1'-biphenyl]-2-carboxylate (12f). Starting with the bis(silyl-enol ether) (0.928 g, 2.4 mmol), TiCl_4 (0.455 g, 2.4 mmol), CH_2Cl_2 (8 mL) and the mono(silyl enol ether) (0.582 g, 2.2 mmol), **12f** was isolated (0.457 g, 52%) by column chromatography (silica gel, *n*-hexane/EtOAc = 40:1 $\rightarrow$ 20:1) as a yellow oil. ^1H NMR (250 MHz, CDCl_3): δ = 0.74 (t, J = 7.3 Hz, 3 H, CH_3), 0.86 (t(br), J = 7.5 Hz, 3 H, CH_3), 1.23-1.37 (m, 12 H, CH_2), 2.30 (s, 3 H, CH_3), 2.64 (t, J = 7.6 Hz, 2 H, CH_3), 3.67 (s, 3 H, OCH_3), 3.94 (q, J = 6.7 Hz, 2 H, OCH_2CH_3), 6.58 (s, 1 H, Ar), 6.85 (dd, J = 7.9 Hz, J = 1.2 Hz, 1 H, Ar), 6.98 (ddd, J = 7.3 Hz, J = 6.9 Hz, J = 0.9 Hz, 1 H, Ar), 7.17 (dd, J = 7.3 Hz, J = 1.8 Hz, 1 H, Ar), 7.29 (ddd, J = 8.2 Hz, J = 7.7 Hz, J = 1.8 Hz, 1 H, Ar), 10.99 (s, 1 H, OH). ^{13}C NMR (62 MHz, CDCl_3): δ = 12.9, 14.1, 19.8 (CH_3), 22.6, 26.9, 28.8, 29.3, 29.5, 30.1, 31.9 (CH_2), 55.3 (OCH_3), 60.5 (OCH_2CH_3), 109.6 (CH), 110.4 (C), 120.2, 124.2 (CH), 127.9 (C), 128.7, 129.3 (CH), 132.5, 137.2, 142.4, 156.2, 159.1, 171.5 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2954 (s), 2926 (s), 2855 (w), 1657 (m), 1610 (m), 1490 (m), 1490 (w), 1389 (m), 1373 (m), 1274 (s), 1243 (m), 1180 (s), 1109 (m), 1036 (m), 750 (w). GC-MS (EI, 70 eV): m/z (%) = 398 (M^+ , 61), 352 (51), 338 (17), 337 (70), 281 (7), 254 (61), 253 (58), 239 (100), 223 (42), 211 (9), 181 (14), 115 (3), 77 (2), 43 (7). HRMS (EI): Calcd. for $\text{C}_{25}\text{H}_{34}\text{O}_4$: 398.24516; found: 398.24515.

Methyl 3-hydroxy-2'-methoxy-5-methyl-4-undecyl[1,1'-biphenyl]-2-carboxylate (12g).

Starting with the bis(silyl-enol ether) (0.683 g, 1.65 mmol), TiCl_4 (0.312 g, 1.65 mmol), CH_2Cl_2 (5 mL) and the mono(silyl enol ether) (0.396 g, 1.5 mmol), **12g** was isolated (0.304 g, 47%) by column chromatography (silica gel, *n*-hexane/EtOAc = 40:1 $\rightarrow$ 20:1) as a colorless oil. ^1H NMR (300 MHz, CDCl_3): δ = 0.70 (t(br), J = 7.5 Hz, 3 H, CH_3), 1.08-1.18 (m, 18 H, CH_2), 2.14 (s, 3 H, CH_3), 2.49 (t, J = 7.6 Hz, 2 H, CH_3), 3.28 (s, 3 H, OCH_3), 3.51 (s, 3 H, OCH_3), 6.48 (s, 1 H, Ar),

6.74 (dd, $J = 8.2$ Hz, $J = 0.7$ Hz, 1 H, Ar), 6.88 (ddd, $J = 7.4$ Hz, $J = 7.4$ Hz, $J = 0.9$ Hz, 1 H, Ar), 7.09 (dd, $J = 7.4$ Hz, $J = 1.7$ Hz, 1 H, Ar), 7.17 (ddd, $J = 8.1$ Hz, $J = 8.1$ Hz, $J = 1.7$ Hz, 1 H, Ar), 10.90 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 16.0, 21.7$ (CH_3), 24.6, 28.3, 30.7, 31.3, 31.3, 31.4, 31.5, 31.5, 32.0, 33.8, (CH_2), 53.2, 57.2 (OCH_3), 111.5 (CH), 112.3 (C), 122.3, 126.4 (CH), 129.6 (C), 130.1 (CH), 130.6, 131.3 (C), 134.2 (CH), 139.0, 144.8, 160.9, 173.9 (C). IR (Nujol, cm^{-1}): $\tilde{\nu} = 2951$ (s), 2924 (s), 2853 (s), 1744 (w), 1663 (s), 1609 (m), 1490 (w), 1457 (m), 1437 (s), 1392 (m), 1356 (w), 1273 (s), 1243 (s), 1170 (m), 1145 (m), 1109 (m), 1030 (m), 813 (w), 751 (m). GC-MS (EI, 70 eV): m/z (%) = 426 (M^+ , 51), 394 (49), 363 (6), 340 (11), 325 (24), 254 (53), 239 (67), 221 (100), 211 (7), 177 (17), 150 (15), 129 (21), 115 (83), 101 (35), 81 (51), 73 (73), 55 (46). HRMS (EI): Calcd. for $\text{C}_{27}\text{H}_{38}\text{O}_4$: 426.27646 found: 426.27560.

Methyl 3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12h). Starting with the bis(silyl-enol ether) (0.568 g, 2.2 mmol), TiCl_4 (0.414 g, 2.2 mmol), CH_2Cl_2 (5 mL) and the mono(silyl enol ether) (0.585 g, 2.0 mmol), **12h** was isolated (0.134 g, 22%) by column chromatography (silica gel, n -hexane/EtOAc = 30:1 $\rightarrow$ 20:1) as a yellow oil. ^1H NMR (300 MHz, CDCl_3): $\delta = 0.82$ (t, $J = 7.3$ Hz, 3 H, CH_3), 1.53 (m, 2 H, CH_2), 2.43 (t, $J = 7.4$ Hz, 2 H, CH_2), 3.34 (s, 3 H, OCH_3), 3.56 (s, 3 H, OCH_3), 6.46-6.47 (m, 1 H, Ar), 6.68 (m, 1 H, Ar), 6.72 (dd, $J = 8.1$ Hz, $J = 0.4$ Hz, 1 H, Ar), 6.85 (ddd, $J = 8.5$ Hz, $J = 7.4$ Hz, $J = 0.9$ Hz, 1 H, Ar), 7.05 (dd, $J = 7.4$ Hz, $J = 1.8$ Hz, 1 H, Ar), 7.15 (ddd, $J = 8.1$ Hz, $J = 8.1$ Hz, $J = 1.9$ Hz, 1 H, Ar), 10.60 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 12.8$ (CH_3), 22.7, 37.0 (CH_2), 50.4, 54.4 (OCH_3), 108.8 (CH), 109.8 (C), 115.3, 119.4, 122.5, 127.4, 128.4 (CH), 132.9, 139.3, 148.5, 155.1, 160.0, 170.4 (C). IR (Nujol, cm^{-1}): $\tilde{\nu} = 2961$ (m), 2934 (m), 2873 (w), 1665 (s), 1610 (s), 1570 (s), 1491 (m), 1437 (m), 1359 (w), 1269 (s), 1248 (s), 1025 (m), 807 (w), 753 (m). GC-MS (EI, 70 eV): m/z (%)

= 300 (M^+ , 42), 269 (23), 268 (100), 240 (43), 197 (12), 152 (6), 115 (5), 91 (2), 77 (2). HRMS (EI): Calcd. for $C_{23}H_{30}O_4$: 300.13561; found: 300.13695.

Methyl 3-hydroxy-2'-methoxy-4-methyl-5-propyl[1,1'-biphenyl]-2-carboxylate (12i). Starting with the bis(silyl-enol ether) (0.598 g, 2.2 mmol), $TiCl_4$ (0.414 g, 2.2 mmol), CH_2Cl_2 (5 mL) and the mono(silyl enol ether) (0.585 g, 2.0 mmol), **12i** was isolated (0.137 g, 21%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 20:1) as a yellow oil. 1H NMR (300 MHz, $CDCl_3$): δ = 0.90 (t, J = 7.4 Hz, 3 H, CH_3), 1.49-1.56 (m, 2 H, CH_2), 2.20 (s, 3 H, CH_3), 2.54 (t, J = 7.5 Hz, 2 H, CH_2), 3.40 (s, 3 H, OCH_3), 3.62 (s, 3 H, OCH_3), 6.52 (s, 1 H, Ar), 6.77 (dd, J = 8.0 Hz, J = 0.9 Hz, 1 H, Ar), 6.91 (ddd, J = 7.4 Hz, J = 7.4 Hz, J = 0.9 Hz, 1 H, Ar), 7.11 (dd, J = 7.4 Hz, J = 1.7 Hz, 1 H, Ar), 7.20 (ddd, J = 8.1 Hz, J = 7.4 Hz, J = 1.7 Hz, 1 H, Ar), 10.98 (s, 1 H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 10.2, 13.1 (CH_3), 22.2, 35.0 (CH_2), 50.4, 54.4 (OCH_3), 108.7 (CH), 109.2 (C), 119.4, 122.5 (CH), 122.6 (C), 127.2, 128.4 (CH), 131.4, 135.9, 146.3, 155.2, 158.2, 170.9 (C). IR (Nujol, cm^{-1}): $\tilde{\nu}$ = 2957 (s), 2929 (s), 2871 (m), 1663 (m), 1610 (m), 1584 (w), 1491 (m), 1437 (m), 1267 (s), 1243 (s), 1169 (m), 1029 (m), 810 (m), 752 (m). GC-MS (EI, 70 eV): m/z (%) = 314 (M^+ , 59), 282 (100), 267 (52), 251 (28), 239 (61), 211 (11), 181 (14), 115 (6), 91 (3), 77 (3).

Ethyl 4-ethyl-3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12j). Starting with the bis(silyl-enol ether) (0.660 g, 2.0 mmol), $TiCl_4$ (0.414 g, 2.2 mmol), CH_2Cl_2 (5 mL) and the mono(silyl enol ether) (0.585 g, 2.0 mmol), **12j** was isolated (0.203 g, 29%) by column chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 20:1) as a yellow oil. 1H NMR (300 MHz, $CDCl_3$): δ = 0.59 (t, J = 7.2 Hz, 3 H, CH_3), 0.81 (t, J = 7.4 Hz, 3 H, CH_3), 1.02 (t, J = 7.4 Hz, 3 H,

CH₃), 1.40-1.48 (m, 2 H, CH₂), 2.43 (t, $J = 7.2$ Hz, 2 H, CH₂), 2.56 (q, $J = 7.4$ Hz, 2 H, CH₂), 3.51 (s, 3 H, OCH₃), 3.79 (q, $J = 7.0$ Hz, 2 H, OCH₂CH₃), 6.40 (s, 1 H, Ar), 6.64-6.66 (m, 1 H, Ar), 6.78 (ddd, $J = 7.4$ Hz, $J = 7.2$ Hz, $J = 0.7$ Hz, 1 H, Ar), 6.98 (dd, $J = 7.4$ Hz, $J = 1.7$ Hz, 1 H, Ar), 7.09 (ddd, $J = 8.0$ Hz, $J = 7.6$ Hz, $J = 1.7$ Hz, 1 H, Ar), 10.96 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): $\delta = 14.1, 15.0, 15.2$ (CH₃), 20.3, 25.2, 36.4 (CH₂), 56.4 (OCH₃), 61.6 (OCH₂CH₃), 110.9 (CH), 111.5 (C), 121.4, 125.0, 129.1, 130.5 (CH), 130.6, 133.8, 138.4, 147.6, 157.4, 160.4, 172.6 (C). IR (Nujol, cm⁻¹): $\tilde{\nu} = 3057$ (w), 2961 (s), 2932 (s), 2872 (m), 2835 (w), 1657 (s), 1608 (m), 1560 (w), 1494 (w), 1463 (m), 1395 (m), 1373 (m), 1278 (s), 1245 (s), 1178 (s), 1111 (m), 1029 (m), 788 (m), 751 (m). GC-MS (EI, 70 eV): m/z (%) = 342 (M⁺, 58), 296 (100), 281 (71), 265 (46), 253 (80), 225 (8), 197 (5), 181 (10), 165 (11), 115 (5), 91 (3), 77 (3). HRMS (EI): Calcd. for C₂₁H₂₆O₄: 342.18256; found: 342.18256.

Ethyl 4-hexyl-3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12k). Starting with the bis(silyl-enol ether) (0.400 g, 1.1 mmol), TiCl₄ (0.209 g, 1.1 mmol) CH₂Cl₂ (3 mL) and the mono(silyl enol ether) (0.292 g, 1.0 mmol), **12k** was isolated (0.123 g, 32%) by column chromatography (silica gel, *n*-hexane/EtOAc = 40:1 → 20:1) as a colorless oil. ¹H NMR (300 MHz, CDCl₃): $\delta = 0.68$ (t, $J = 7.0$ Hz, 3 H, CH₃), 0.82 (t(br), $J = 7.2$ Hz, 3 H, CH₃), 0.89 (t, $J = 7.2$ Hz, 3 H, CH₃), 1.25-1.52 (m, 10 H, CH₂), 2.51 (t, $J = 8.3$ Hz, 2 H, CH₂), 2.58 (t, $J = 7.0$ Hz, 2 H, CH₂), 3.60 (s, 3 H, CH₃), 3.87 (q, $J = 7.0$ Hz, 2 H, OCH₂CH₃), 6.49 (s, 1 H, Ar), 6.74 (dd, $J = 8.1$ Hz, $J = 0.9$ Hz, 1 H, Ar), 6.88 (ddd, $J = 7.4$ Hz, $J = 7.4$ Hz, $J = 0.9$ Hz, 1 H, Ar), 7.08 (dd, $J = 7.4$ Hz, $J = 1.7$ Hz, 1 H, Ar), 7.20 (ddd, $J = 8.1$ Hz, $J = 8.0$ Hz, $J = 1.9$ Hz, 1 H, Ar), 11.04 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): $\delta = 13.4, 14.5, 14.6$ (CH₃), 23.1, 24.4, 26.5, 29.7, 30.0, 32.0, 35.7 (CH₂), 55.7 (OCH₂CH₃), 60.9 (OCH₃), 110.2 (CH), 110.7 (C), 120.7, 123.9, 128.4 (CH),

128.7 (C), 129.9 (CH), 133.1, 137.6, 147.6, 156.7, 159.7, 171.9 (C). IR (Nujol, cm^{-1}): $\tilde{\nu}$ = 2957 (s), 2929 (s), 2870 (m), 1658 (s), 1609 (m), 1560 (w), 1463 (m), 1400 (s), 1374 (s), 1270 (s), 1242 (s), 1179 (s), 1179 (s), 1110 (m), 1030 (m), 817 (w), 751 (s).

Methyl 3-hydroxy-2'-methoxy-5-propyl-4-undecyl[1,1'-biphenyl]-2-carboxylate (12l). Starting with the bis(silyl-enol ether) (0.911 g, 2.2 mmol), TiCl_4 (0.416 g, 2.2 mmol) CH_2Cl_2 (6 mL) and the mono(silyl enol ether) (0.584 g, 2.0 mmol), **12l** was isolated (0.396 g, 43%) by column chromatography (silica gel, *n*-hexane/EtOAc = 40:1 $\rightarrow$ 20:1) as a colorless oil. ^1H NMR (300 MHz, CDCl_3): δ = 0.78 (t (br), J = 6.3 Hz, 3 H, CH_3), 0.89 (t, J = 7.4 Hz, 3 H, CH_3), 1.16-1.18 (m, 20 H, CH_2), 2.50 (t, J = 6.4 Hz, 2 H, CH_2), 2.58 (t, J = 7.4 Hz, 2 H, CH_2), 3.36 (s, 3 H, OCH_3), 3.58 (s, 3 H, OCH_3), 6.49 (s, 1 H, Ar), 6.74 (dd, J = 8.1 Hz, J = 0.9 Hz, 1 H, Ar), 6.88 (ddd, J = 7.4 Hz, J = 7.4 Hz, J = 0.9 Hz, 1 H, Ar), 7.09 (dd, J = 7.4 Hz, J = 1.7 Hz, 1 H, Ar), 7.16 (ddd, J = 8.1 Hz, J = 8.1 Hz, J = 1.8 Hz, 1 H, Ar), 10.89 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 15.2, 15.4 (CH_3), 23.8, 23.9, 25.2, 27.3, 30.5, 30.7, 30.8, 30.8, 30.9, 31.4, 33.1, 36.5 (CH_2), 52.4, 56.5 (OCH_3), 110.8 (CH), 111. (C), 121.6, 124.6, 129.3 (CH), 129.5 (C), 130.6 (CH), 133.6, 138.2, 148.0, 157.4, 160.3, 173.1 (C). IR (Nujol, cm^{-1}): $\tilde{\nu}$ = 2954 (s), 2952 (s), 2853 (s), 1704 (w), 1637 (s), 1608 (m), 1560 (w), 1463 (m), 1437 (m), 1339 (m), 1273 (s), 1241 (s), 1198 (m), 1029 (m), 752 (m). GC-MS (EI, 70 eV): m/z (%) = 454 (M^+ , 67), 422 (40), 405 (19), 379 (100), 340 (8), 304 (8), 281 (44), 251 (23), 239 (46), 223 (10), 177 (19), 129 (18), 116 (28), 69 (36), 57 (57). HRMS (EI): Calcd. for $\text{C}_{29}\text{H}_{42}\text{O}_4$: 454.30776; found: 454.30717.

The synthesis of **3i** and **3j** has been previously reported.^{14b}

General procedure for the synthesis of triflates 4. To a CH₂Cl₂ solution of **4** (1.0 equiv.) was added pyridine (2.0 equiv.) at –78 °C. After 10 min, triflic anhydride (Tf₂O, 1.2 equiv.) was added at –78 °C. The solution was allowed to warm to 0 °C and was stirred for 4 h. The product was isolated by column chromatography (silica gel, CH₂Cl₂) as a colourless viscous oil.

Ethyl 2,4-dimethyl-6-(trifluoromethylsulfonyloxy)benzoate (4a). Starting with **3a** (0.415 g, 2.14 mmol) in 20 mL of CH₂Cl₂, pyridine (0.339 g, 4.28 mmol) and Tf₂O (0.727 g, 2.57 mmol), **4a** was isolated as a colourless oil (0.505 g, 73%). ¹H NMR (300 MHz, CDCl₃): δ = 1.39 (t, *J* = 7.1 Hz, 3 H, OCH₂CH₃), 2.37 (s, 3 H, CH₃), 2.41 (s, 3 H, CH₃), 4.39 (q, *J* = 7.1 Hz, 2 H, OCH₂CH₃), 6.95 (s, 1 H, Ar), 7.06 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.9, 20.2, 21.3 (CH₃), 26.0 (CH₂), 116.5 (C), 119.6 (CH), 124.3 (C), 131.3 (CH), 139.4, 142.2, 146.8, 165.1 (C). IR (KBr, cm^{–1}): $\tilde{\nu}$ = 2986 (m), 1733 (s), 1624 (s), 1569 (m), 1426 (s), 1274 (s), 1211 (s), 1142 (s), 1089 (s), 1021 (s), 957 (s), 856 (s), 821 (s), 764 (m), 607 (s). MS (EI, 70 eV): *m/z* (%) = 327 ([M+1]⁺, 5), 326 (M⁺, 76), 281 (96), 193 (4), 165 (100), 147 (45), 119 (27), 91 (86), 28 (37). Anal. calcd. for C₁₂H₁₃O₅SF₃: C 44.18, H 4.01; found: C 44.25, 3.97.

[2-(Methoxycarbonyl)-3,4,5-trimethylphenyl]-trifluoromethanesulfonate (4b). Starting with **3b** (0.432 g, 2.23 mmol) in 20 mL of CH₂Cl₂, pyridine (0.352 g, 4.46 mmol) and Tf₂O (0.758 g, 2.67 mmol), **4b** was isolated as a colorless oil (0.540 g, 74%). ¹H NMR (300 MHz, CDCl₃): δ = 2.19 (s, 3 H, CH₃), 2.29 (s, 3 H, CH₃), 2.33 (s, 3 H, CH₃), 3.94 (s, 3 H, CH₃O), 6.97 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 15.6, 17.7, 21.1, 52.6 (CH₃), 116.4 (C), 119.9 (CH), 125.2, 136.5, 136.8, 140.4, 143.8, 166.2 (C). MS (EI, 70 eV): *m/z* (%) = 327 ([M+1]⁺, 6), 326 (M⁺, 51), 295 (39), 193 (100), 162 (37), 161 (36), 133 (41), 91 (25), 28 (21).

[2-(Methoxycarbonyl)-4-ethyl-3,5-dimethylphenyl]-trifluoromethanesulfonate (4c). Starting with **3c** (0.610 g, 2.93 mmol) in 25 mL of CH₂Cl₂, pyridine (0.463 g, 5.86 mmol), and Tf₂O (0.996 g, 3.52 mmol), **4c** was isolated as a colorless oil (0.850 g, 85%). ¹H NMR (300 MHz, CDCl₃): δ = 1.11 (t, J = 7.1 Hz, 3 H, CH₃CH₂), 2.34 (t, J = 7.2 Hz, 3 H, CH₃), 2.37 (s, 3 H, CH₃), 2.66 (q, J = 6.9 Hz, 2 H, CH₃CH₂), 3.94 (s, 3 H, CH₃O), 6.96 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 16.6, 20.2 (CH₃), 22.6 (CH₂), 52.6 (CH₃), 116.4 (C), 120.4 (CH), 120.6, 125.6, 136.3, 140.0, 142.1, 143.8, 166.3 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2971 (s), 2884 (m), 1738 (s), 1607 (s), 1421 (s), 1278 (s), 1211 (s), 1147 (s), 1048 (s), 1012 (s), 954 (s), 857 (s), 819 (s), 607 (s). UV-VIS (CH₃CN, nm): λ_{max} (log ϵ) = 202.8 (4.47). MS (EI, 70 eV): m/z (%) = 342 ([M+2]⁺, 2), 341 ([M+1]⁺, 8), 340 (M⁺, 58), 309 (42), 207 (100), 176 (39), 175 (75), 146.5 (23), 119 (30), 91 (17), 28 (44). Anal. calcd. for C₁₃H₁₅O₅SF₃: C 45.89, H 4.44; found C 46.11, H 4.52.

[2-(Methoxycarbonyl)-5-methyl-3-phenylphenyl]-trifluoromethanesulfonate (4d). Starting with **3d** (0.307 g, 1.20 mmol) in 10 mL of CH₂Cl₂, pyridine (0.190 g, 2.40 mmol), and Tf₂O (0.409 g, 1.20 mmol), **4d** was isolated as a colourless solid (0.420 g, 90%). ¹H NMR (300 MHz, CDCl₃): δ = 1.02 (t, J = 7.1 Hz, 3 H, CH₃CH₂O), 2.46 (s, 3 H, CH₃), 4.13 (q, J = 7.0 Hz, 2 H, CH₃CH₂O), 7.14 (s, 1 H, Ar), 7.23 (m, 1 H, Ar), 7.33-7.36 (m, 2 H, Ar), 7.37-7.39 (m, 2 H, Ar), 7.42 (m, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.5 (CH₃), 21.4 (CH₂), 61.9 (CH₃), 120.7 (CH), 124.4 (C), 128.1 (CH), 128.2, 128.4 (2CH), 130.6 (CH), 139.1 (C), 142.1 (2C), 143.1, 146.4, 164.9 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3440 (w), 2987 (m), 1730 (s), 1617 (m), 1424 (s), 1272 (s), 1209 (s), 1139 (s), 1068 (m), 975 (s), 880 (m), 813 (s), 766 (m), 701 (m), 596 (s). MS (EI, 70 eV): m/z (%) = 389 ([M+1]⁺, 8), 388 (M⁺, 55), 210 (59), 182 (23), 174 (34), 152 (21), 114 (7), 32 (22), 28 (100). Anal. calcd. for C₁₇H₁₅O₅SF₃: C 52.59, H 3.89; found C 53.04, H 4.12.

[2-(Methoxycarbonyl)-6-methoxy-3,5-dimethylphenyl]-trifluoromethanesulfonate (4e).

Starting with **3e** (0.178 g, 0.85 mmol) in 8 mL of CH₂Cl₂, pyridine (0.135 g, 1.7 mmol) and Tf₂O (0.290 g, 1.02 mmol), **4e** was isolated as a colourless oil (0.187 g, 65%). ¹H NMR (300 MHz, CDCl₃): δ = 2.33 (s, 3 H, CH₃), 2.35 (s, 3 H, CH₃), 3.81 (s, 3 H, CH₃O), 3.92 (s, 3 H, CH₃O), 7.05 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 16.0, 19.8 (CH₃), 52.5, 61.0 (CH₃O), 116.4, 124.7 (C), 132.7 (CH), 133.8, 135.6, 139.8, 148.2, 165.2 (C). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 203.2 (4.51), 281.2 (3.42), 308.0 (3.14). MS (EI, 70 eV): *m/z* (%) = 344 ([M+2]⁺, 2), 343 ([M+1]⁺, 5), 342 (M⁺, 45), 311 (20), 209 (100), 177 (93), 148 (89), 77 (19), 28 (28). Anal. calcd. for C₁₂H₁₃O₅SF₃: C 42.12, H 3.83; found: C 42.48, H 3.91.

[5-(Ethoxycarbonyl)-1,2,3,4-tetrahydronaphthalen-6-yl]-trifluoromethanesulfonate (4f).

Starting with **12ad** (0.304 g, 1.38 mmol) in 12 mL CH₂Cl₂, pyridine (0.218 g, 2.76 mmol), Tf₂O (0.469 g, 1.66 mmol), **23f** was isolate as a colourless solid (0.407 g, 92%). ¹H NMR (300 MHz, CDCl₃): δ = 1.41 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.80 (m, 4 H, 2 CH₂), 2.81 (m, 4 H, 2 CH₂), 4.36 (q, *J* = 7.2 Hz, 2 H, OCH₂CH₃), 7.08 (d, 1 H, Ar), 7.18 (d, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.9 (CH₃), 22.1, 22.2, 27.0, 29.4, 62.1 (CH₂), 116.4, 118.3, 127.3 (C), 131.9 (CH), 137.5, 138.3, 144.0, 165.3 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2940 (m), 1733 (s), 1604 (m), 1468 (m), 1426 (s), 1258 (s), 1215 (s), 1140 (s), 1033 (m), 942 (s), 836 (s), 606 (m). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 275.5 (3.83). MS (EI, 70 eV): *m/z* (%) = 353 ([M+1]⁺, 2), 352 (M⁺, 11), 307 (16), 218 (31), 174 (26), 173 (100), 114 (9), 91 (8), 32 (17), 28 (77). Anal. calcd. for C₁₇H₁₅O₅SF₃: C 47.74, H 4.29; found C 48.33, H 4.45.

[5-(Methoxycarbonyl)-1,2,3,4-tetrahydro-8-methylnaphthalen-6-yl]-

trifluoromethanesulfonate (4g). Starting with **3g** (0.311 g, 1.41 mmol) in 15 mL of CH₂Cl₂, pyridine (0.223 g, 2.82 mmol) and Tf₂O (0.480 g, 1.70 mmol), **4g** was isolated as a colourless solid (0.370 g, 75%). ¹H NMR (300 MHz, CDCl₃): δ = 1.74-1.85 (m, 4 H, 2 CH₂), 2.26 (t, *J* = 6.5 Hz, 3 H, CH₃), 2.62 (t, *J* = 6.3 Hz, 2 H, CH₂) 3.92 (s, 3H, CH₃O), 6.94 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 19.9 (CH₃), 21.9, 22.2, 26.8, 27.8 (CH₂), 52.4 (CH₃), 116.3 (C), 119.6 (CH), 124.4, 136.7, 137.6, 140.9, 143.7, 166.1 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3443 (m), 2940 (m), 1735 (s), 1607 (m), 1423 (s), 1233 (s), 1214 (s), 1145 (s), 843 (m), 606 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 203.6 (4.52). MS (EI, 70 eV): *m/z* (%) = 353 ([M+1]⁺, 5), 352 (M⁺, 15), 219 (34), 187 (100), 131 (10), 91 (10), 28 (9). Anal. calcd. for C₁₄H₁₅O₅SF₃: C 47.73, H 4.29; found C 48.43, H 4.29.

[4-(Methoxycarbonyl)-9,10-dihydro-1-methylphenanthren-3-yl]-trifluoromethanesulfonate

(4h). Starting with **3h** (0.456 g, 1.70 mmol) in 15 mL CH₂Cl₂, pyridine (0.269 g, 3.4 mmol) and Tf₂O (0.578 g, 2.04 mmol), **4h** was isolated as a yellow solid (0.420 g, 61%). ¹H NMR (300 MHz, CDCl₃): δ = 2.41 (s, 3 H, CH₃), 2.76 (m, *J* = 4.0 Hz, 2.0 Hz, 2 H, CH₂), 2.82 (m, *J* = 4.0 Hz, 2.0 Hz, 2 H, CH₂), 3.84 (s, 3 H, CH₃O), 7.08 (s, 1 H, Ar), 7.25-7.29 (m, 3 H, Ar), 7.39 (dd, *J* = 1.7 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 20.3 (CH₃), 25.3, 28.5 (CH₂), 52.7 (CH₃), 116.4 (C), 121.2 (CH), 122.5 (C), 126.6, 126.7, 127.8, 128.6 (CH), 132.5, 135.7, 128.5, 138.6, 139.2, 145.1, 166.9 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3444 (m, br), 2959 (w), 1737 (s), 1605 (m), 1422 (s), 1278 (s), 1216 (s), 1141 (s), 950 (s), 878 (m), 609 (m). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 209.9 (4.53), 267.5 (4.12), 301.9 (3.79). MS (EI, 70 eV): *m/z* (%) = 401 ([M+1]⁺, 7), 400 (M⁺, 42), 267 (9), 236 (18), 235 (19), 208 (36), 207 (35), 179 (40), 178 (30), 165 (34), 32 (27), 28 (100). Anal. calcd. for C₁₈H₁₅O₅SF₃: C 54.00, H 3.78; found C 53.78, H 3.85.

[2-(Methoxycarbonyl)-4-(2-chloroethyl)-3,5-dimethylphenyl]-trifluoromethanesulfonate (4i).

Starting with **3i** (0.435 g, 1.79 mmol) in 18 mL of CH₂Cl₂, pyridine (0.283 g, 3.58 mmol) and Tf₂O (0.609 g, 2.15 mmol), **4i** was isolated as a colourless oil (0.619 g, 92%). ¹H NMR (300 MHz, CDCl₃): δ = 2.37 (s, 3 H, CH₃), 2.42 (s, 3 H, CH₃), 3.17 (m, *J* = 7.6 Hz, 2.0 Hz, 2 H, ClCH₂CH₂), 3.54 (m, *J* = 7.3 Hz, 2.4 Hz, 2 H, ClCH₂CH₂), 3.95 (s, 3 H, OCH₃), 7.01 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 17.0, 20.6 (CH₃), 32.9, 41.4 (CH₂), 52.7 (CH₃), 116.3 (C), 120.8 (CH), 126.1, 135.9, 137.4, 141.1, 144.7, 165.9 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2976 (m), 1738 (s), 1608 (m), 1426 (s), 1274 (s), 1217 (s), 1142 (s), 1014 (s), 953 (m), 866 (m), 606 (m). MS (EI, 70 eV): *m/z* (%) = 376 ([M+1]⁺, 12), 375 (M⁺, 3), 374 (39), 241 (55), 209 (23), 161 (24), 32 (24), 28 (100). Anal. calcd. for C₁₃H₁₄O₅ClSF₃: C 41.67, H 3.76; found C 42.18, H 3.71.

[2-(Methoxycarbonyl)-4-(2-bromoethyl)-3,5-dimethylphenyl]-trifluoromethanesulfonate (4j).

Starting with **3j** (0.425 g, 1.48 mmol) in 16 mL of CH₂Cl₂, pyridine (0.234 g, 2.96 mmol) and Tf₂O (0.503 g, 1.78 mmol), **4j** was isolated by column chromatography (silica gel; CH₂Cl₂) as a colourless oil (0.560 g, 90%). ¹H NMR (300 MHz, CDCl₃): δ = 2.67 (s, 3 H, CH₃), 2.41 (s, 3 H, CH₃), 3.23 (m, *J* = 7.6 Hz, 2.0 Hz, 2 H, BrCH₂CH₂), 3.54 (m, *J* = 7.5 Hz, 2.0 Hz, 2 H, BrCH₂CH₂), 3.95 (s, 3 H, OCH₃), 7.01 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 17.0, 20.5 (CH₃), 28.9, 33.3 (CH₂), 52.7 (CH₃), 116.3 (C), 120.9 (CH), 126.1, 136.9, 137.2, 140.9, 144.7, 165.8 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3446 (br, w), 2960 (w), 1737 (s), 1608 (m), 1425 (s), 1272 (s), 1215 (s), 1140 (s), 1014 (m), 863 (m), 793 (w), 607 (m). MS (EI, 70 eV): *m/z* (%) = 376 ([M+1]⁺, 12), 375 (M⁺, 3), 374 (39), 241 (55), 209 (23), 161 (24), 32 (24), 28 (100). Anal. calcd. for C₁₃H₁₄O₅BrSF₃: C 37.33, H 3.37; found C 38.67, H 3.41.

General procedure for the synthesis of biaryls 6 by Suzuki cross-coupling reactions. A dioxane solution of the boronic acid, potassium phosphate, Pd(PPh₃)₄ and triflate **4** was stirred at 110 °C for 4 h. After cooling to 20 °C, a saturated aqueous solution of NH₄Cl was added. The organic and the aqueous layer were separated and the latter was extracted with CH₂Cl₂. The combined organic layers were dried (Na₂SO₄), filtered and the filtrate was concentrated in *vacuo*. The residue was purified by chromatography (silica gel, *n*-hexane/EtOAc = 20:1).

Ethyl 2,4-dimethyl-6-(2-methoxyphenyl)benzoate (6a). Starting with 2-methoxyphenylboronic acid (**5a**, 1.3 equiv., 0.306 g, 2.01 mmol), potassium phosphate (1.6 equiv., 0.509 g, 2.40 mmol) and Pd(PPh₃)₄ (3 mol-%, 0.052 g, 0.045 mmol), dioxane (5 mL) and **4a** (1.0 equiv., 0.489 g, 1.5 mmol), **6a** was isolated as a colourless viscous oil (0.320 g, 75%). ¹H NMR (300 MHz, CDCl₃): δ = 0.89 (t, *J* = 7.1 Hz, 3 H, OCH₂CH₃), 2.34 (s, 3 H, CH₃), 2.41 (s, 3 H, CH₃), 3.74 (s, 3 H, CH₃O), 3.98 (q, *J* = 7.2 Hz, 2 H, OCH₂CH₃), 6.89-6.91 (d, *J* = 8.3 Hz, 1 H, Ar), 6.94-6.96 (dd, *J* = 1.0 Hz, 6.5 Hz, 1 H, Ar), 6.98 (s, 1 H, Ar), 7.02 (s, 1 H, Ar), 7.16-7.19 (dd, *J* = 1.7 Hz, 5.7 Hz, 1 H, Ar), 7.29-7.32 (td, *J* = 1.8 Hz, 6.8 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.6, 20.2, 21.2, 55.4 (CH₃), 60.3 (CH₂), 110.4 (C), 120.4, 128.6 (CH), 129.0 (2CH), 130.3 (C), 130.5 (CH), 130.6, 135.9, 137.5, 139.3, 156.3, 169.1 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2978 (s), 2931 (s), 1724 (s), 1605 (s), 1580 (m), 1497 (s), 1459 (s), 1434 (s), 1384 (m), 1265 (s), 1180 (s), 1107 (s), 1081 (s), 1027 (s), 861 (s), 754 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 210.3 (4.56), 281.7 (3.59). MS (EI, 70 eV): *m/z* (%) = 285 ([M+1]⁺, 7), 284 (M⁺, 100), 253 (64), 239 (58), 225 (61), 195 (32), 181 (28), 165 (27), 151 (15), 115 (5), 29 (12). HRMS (ESI) for C₁₈H₂₀O₃ [M+1]⁺: Calcd. 285.14907; found 285.14842. Anal. calcd. for C₁₈H₂₀O₃: C 76.02, H 7.09; found: C 75.82, H 6.61.

Methyl 2,3,4-trimethyl-6-(2-methoxyphenyl)benzoate (6b). Starting with **5a** (1.3 equiv., 0.296 g, 1.95 mmol), potassium phosphate (1.6 equiv., 0.509 g, 2.40 mmol) and Pd(PPh₃)₄ (3 mol-%, 0.052 g, 0.045 mmol), dioxane (5 mL), and **4b** (1.0 equiv., 0.510 g, 1.5 mmol), **6b** was isolated as a colourless solid (0.320 g, 73%). ¹H NMR (300 MHz, CDCl₃): δ = 2.24 (s, 3 H, CH₃), 2.31 (s, 3 H, CH₃), 2.32 (s, 3 H, CH₃), 3.51 (s, 3 H, CH₃O), 3.74 (s, 3 H, CH₃O), 6.89 (d, 1 H, CH), 6.92-6.98 (m, 1 H, CH), 7.00 (s, 1 H, CH), 7.14-7.18 (dd, 1 H, CH, Ar), 7.28-7.31 (dd, 1 H, CH, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 15.6, 17.6, 20.9, 51.4, 55.5 (CH₃), 110.5 (C), 120.3 (CH), 128.6, 129.8 (C), 130.0, 130.6 (CH), 131.9, 133.5, 133.9, 134.8, 137.7, 156.4, 170.4 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2997 (m), 2949 (m), 1719 (s), 1600 (m), 1497 (s), 1464 (s), 1436 (s), 1269 (s), 1239 (s), 1634 (s), 1108 (s), 1048 (s), 1269 (s), 1239 (s), 1164 (s), 1108 (s), 1048 (s), 772 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 210.3 (4.59), 281.0 (3.63). MS (EI, 70 eV): *m/z* (%) = 285.0 ([M+1]⁺, 2), 284.0 (M⁺, 17), 253.0 (21), 210.0 (43), 182.0 (15), 152.5 (4), 28.1 (100). HRMS (ESI) for C₁₈H₂₀O₃ ([M+1]⁺): Calcd. 285.14907; found 285.14871. Anal. calcd. for C₁₈H₂₀O₃: C 76.03, H 7.09; found: C 75.86, H 7.84.

Methyl 2,4-dimethyl-3-ethyl-6-(2-methoxyphenyl)benzoate (6c). Starting with **5a** (1.3 equiv., 0.170 g, 1.12 mmol), potassium phosphate (1.6 equiv., 0.292 g, 1.38 mmol) and Pd(PPh₃)₄ (3%mol 0.030 g, 0.026 mmol), 3 mL of dioxane, and **4c** (1.0 equiv., 0.305 g, 0.86 mmol), **25c** was isolated as colorless oil (0.245 g, 95%). ¹H NMR (300 MHz, CDCl₃): δ = 1.15 (t, *J* = 7.1 Hz, 3 H, CH₃CH₂), 2.34 (s, 3 H, CH₃), 2.35 (s, 3 H, CH₃), 2.72 (q, *J* = 7.0 Hz, 2 H, CH₃CH₂), 3.51 (s, 3 H, CH₃O), 3.74 (s, 3 H, CH₃O), 6.89-6.93 (m, 1 H, Ar), 6.95 - 6.98 (dd, *J* = 1.1 Hz, 6.3 Hz, 1 H, Ar), 6.99 (s, 1 H, Ar), 7.14 - 7.18 (dd, *J* = 1.8 Hz, 5.7 Hz, 1 H, Ar), 7.25-7.31 (m, *J* = 1.8 Hz, 5.7 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.1, 16.5, 19.6 (CH₃), 22.7 (CH₂), 51.3, 55.5 (OCH₃),

110.5, 120.3, 128.6 (CH), 130.0 (C), 130.2, 130.7 (CH), 132.3, 132.9, 134.1, 137.1, 140.4, 156.4, 170.5 (C). MS (EI, 70 eV): m/z (%) = 299 ($[M+1]^+$, 2), 298 (M^+ , 15), 267 (18), 234 (6), 209 (4), 165 (3), 32 (27), 28 (100).

Ethyl 4-methyl-6-(2-methoxyphenyl)-2-phenylbenzoate (6d). Starting with **5a** (1.3 equiv., 0.200 g, 1.32 mmol), potassium phosphate (1.6 equiv., 0.344 g, 1.62 mmol), $Pd(PPh_3)_4$ (3 mol-%, 0.035 g, 0.03 mmol), 3 mL of dioxane, and **4d** (1.0 equiv., 0.394 g, 1.01 mmol), **6d** was isolated as a colourless oil (0.338 g, 95%). 1H NMR (300 MHz, $CDCl_3$): δ = 0.73 (t, J = 7.1 Hz, 3 H, OCH_2CH_3), 2.43 (s, 3 H, CH_3), 3.74 (s, 3 H, CH_3O), 3.79 (q, J = 7.2 Hz, 2 H, OCH_2CH_3), 6.90 - 6.93 (d, J = 8.2 Hz, 1 H, Ar), 6.97 - 7.02 (td, J = 1.0 Hz, 6.4 Hz, 1 H, Ar), 7.15 - 7.19 (dq, J = 0.8 Hz, 7.4 Hz, 2 H, Ar), 7.23 - 7.30 (m, 1 H, Ar), 7.32 (m, 1 H, Ar), 7.29 - 7.41 (m, 5 H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.3, 21.3, 55.3 (CH_3), 60.3 (CH_2), 110.5, 120.4, 127.1, 128.0 (CH), 128.4 (2 CH), 128.9 (2 CH), 129.7 (C), 129.8, 130.5 (CH), 130.6 (C), 130.8 (CH), 137.7, 139.1, 140.7, 141.4, 156.4, 169.0 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2982 (m), 1728 (s), 1601 (m), 1496 (s), 1463 (s), 1259 (s), 1180 (m), 1100 (s), 1050 (m), 867 (m), 761 (s), 703 (m). MS (EI, 70 eV): m/z (%) = 347 ($[M+1]^+$, 17), 346 (M^+ , 74), 315 (100), 301 (14), 287 (98), 257 (22), 32 (18), 28 (97). UV-VIS (CH_3CN , nm): λ_{max} (log ϵ) = 211.2 (4.57), 280.7 (3.66). Anal. calcd. for $C_{23}H_{22}O_3$: C 79.74, H 6.40; found: C 79.66, H 7.04.

Methyl 2,4-dimethyl-5-methoxy-6-(2-methoxyphenyl)benzoate (6e). Starting with **5a** (1.3 equiv., 0.580 g, 0.38 mmol), potassium phosphate (1.6 equiv., 0.99 g, 0.47 mmol) and $Pd(PPh_3)_4$ (3 mol-%, 0.010 g, 0.009 mmol), 2 mL of dioxane, and **4e** (1.0 equiv., 0.100 g, 0.292 mmol), **6e** was isolated as a colourless viscous oil (0.072 g, 82%). 1H NMR (300 MHz, $CDCl_3$): δ = 2.30 (s, 3 H, CH_3), 2.31 (s, 3 H, CH_3), 3.36 (s, 3 H, CH_3O), 3.44 (s, 3 H, CH_3O), 3.91 (s, 3 H, CH_3O), 6.93 -

6.99 (m, 1 H, Ar), 7.04 (s, 1 H, Ar), 7.13 - 7.16 (dd, 1 H, Ar), 7.29 - 7.35 (m, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 16.3, 29.4, 51.4, 55.8, 60.1 (CH_3), 110.7, 120.1 (CH), 125.9 (C), 128.9 (CH), 130.4 (C), 131.0 (CH), 132.4, 132.5, 132.7, 132.9, 154.3, 157.1, 169.2 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2946 (s), 2839 (m), 1728 (s), 1601 (m), 1496 (s), 1460 (s), 1437 (s), 1279 (s), 1246 (s), 1195 (s), 1165 (s), 1056 (s), 1029 (s), 871 (m), 755 (s). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 202.8 (4.57), 280.1 (3.64). MS (EI, 70 eV): m/z (%) = 300 (M^+ , 6), 269 (3), 225 (2), 209 (3), 177 (2), 148 (3), 80 (4), 28 (100). HRMS (EI, 70 eV): $\text{C}_{18}\text{H}_{20}\text{O}_4$: Calcd. 300.13616; found: 300.13602

Ethyl 5,6,7,8-tetrahydro-2-(2-methoxyphenyl)naphthalene-1-carboxylate (6f). Starting with **5a** (1.3 equiv., 0.222 g, 1.46 mmol), potassium phosphate (1.6 equiv., 0.379 g, 1.79 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (3 mol-%, 0.039 g, 0.034 mmol), 5 mL of dioxane, and **4f** (1.0 equiv., 0.395 g, 1.12 mmol), **6f** was isolated as a colorless solid (0.273 g, 79%). ^1H NMR (300 MHz, CDCl_3): δ = 0.91 (t, J = 7.1 Hz, 3H, OCH_2CH_3), 1.81 (m, 4 H, 2 CH_2), 2.83 (m, 4 H, 2 CH_2), 3.75 (s, 3 H, CH_3O), 3.99 (q, J = 7.2 Hz, 2 H, OCH_2CH_3), 6.89 - 6.94 (m, 2 H, Ar), 7.08 - 7.18 (m, 3 H, Ar), 7.27 - 7.32 (m, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 13.6 (CH_3), 22.6, 22.9, 27.1, 29.7 (CH_2), 54.5 (CH_3), 60.4 (CH_2), 110.5, 120.3, 127.8, 128.7 (CH), 129.9 (C), 130.2, 130.8 (CH), 133.6 (C), 134.2 (2 C), 136.7, 156.6, 169.3 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2933 (m), 1724 (s), 1591 (m), 1468 (s), 1245 (s), 1139 (s), 1027 (m), 755 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 208.7 (4.56), 281.4 (3.69). MS (EI, 70 eV): m/z (%) = 310 (M^+ , 1), 264 (2), 214 (2), 32 (48), 28 (100). Anal. calcd. for $\text{C}_{20}\text{H}_{22}\text{O}_3$: C 77.39, H 7.14; found: C 77.38, H 6.30.

Methyl 5,6,7,8-tetrahydro-2-(2-methoxyphenyl)-4-methylnaphthalene-1-carboxylate (6g).

Starting with **5a** (1.3 equiv., 0.199 g, 1.31 mmol), potassium phosphate (1.6 equiv., 0.341 g, 1.61

mmol) and Pd(PPh₃)₄ (3 mol-% 0.035 g, 0.03 mmol), 3 mL of dioxane, and **4g** (1.0 equiv., 0.354 g, 1.0 mmol), **6g** was isolated as a colourless oil (0.190 g, 61%). ¹H NMR (300 MHz, CDCl₃): δ = 1.74 - 1.86 (m, 4 H, 2 CH₂), 2.24 (s, 3 H, CH₃), 2.66 (t, 2 H, CH₂), 2.83 (m, 2 H, CH₂), 3.50 (s, 3 H, CH₃O), 3.75 (s, 3 H, CH₃O), 6.85 - 6.93 (m, *J* = 1.0 Hz, 7.2 Hz, 1 H, Ar), 6.95 - 6.99 (dd, *J* = 1.1 Hz, 6.4 Hz, 1 H, Ar), 7.00 (s, 1 H, Ar), 7.15 - 7.18 (dd, *J* = 1.8 Hz, 5.7 Hz, 1 H, Ar), 7.25 - 7.31 (m, *J* = 1.8 Hz, 6.3 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 19.8 (CH₃), 22.6, 27.2, 27.9, 30.2 (CH₂), 51.3, 55.4 (CH₃), 110.4, 120.3, 128.6, 129.4 (CH), 130.0 (C), 130.6 (CH), 131.4, 133.9, 134.4, 135.1, 138.0, 156.3, 170.0 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3422 (w), 3002 (w), 2938 (s), 2836 (m), 1722 (s), 1600 (m), 1497 (m), 1462 (s), 1434 (s), 1247 (s), 1185 (m), 1162 (m), 1106 (m), 1029 (m), 764 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 210.0 (4.59), 281.3 (3.68). MS (EI, 70 eV): *m/z* (%) = 310 (M⁺, 1), 279 (2), 214 (7), 108 (2), 32 (24), 28 (100). HRMS (EI, 70 eV): calcd. for C₂₀H₂₂O₃: Calcd. 310.15689; found: 310.15590.

Methyl 9,10-dihydro-3-(2-methoxyphenyl)-1-methylphenanthrene-4-carboxylate (6h).

Starting with **5a** (1.3 equiv., 0.198 g, 1.30 mmol), potassium phosphate (1.6 equiv., 0.339 g, 1.60 mmol) and Pd(PPh₃)₄ (3 mol-%, 0.035 g, 0.030 mmol), 4 mL of dioxane, and **4h** (1.0 equiv., 0.400 g, 1.00 mmol), **6h** was isolated as a colourless solid (0.307 g, 86%), mp. = 126 °C. ¹H NMR (300 MHz, CDCl₃): δ = 2.38 (s, 3 H, CH₃), 2.79 (m, 2 H, CH₂CH₂), 2.86 (m, 2 H, CH₂CH₂), 3.47 (s, 3 H, CH₃O), 3.75 (s, 3 H, CH₃O), 6.92 - 6.95 (dd, *J* = 8.2 Hz, 1.0 Hz, 1 H, Ar), 6.97 - 7.02 (dt, *J* = 7.5 Hz, 1.0 Hz, 1 H, Ar), 7.09 (s, 1 H, Ar), 7.17 - 7.19 (m, 1 H, Ar), 7.20 (m, 1 H, Ar), 7.23 - 7.29 (m, 2 H, Ar), 7.32 - 7.36 (m, 2 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 20.1, 25.5 (CH₃), 28.9 (CH₂), 51.6, 55.5 (CH₃), 110.6, 120.4, 126.3, 126.8, 127.3, 127.4, 128.8 (CH), 128.9, 129.8 (C), 130.6, 131.5 (CH), 133.3, 134.1, 135.9, 136.3, 137.3, 138.6, 156.4, 170.9 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$

=1725 (m), 1495 (w), 1459 (w), 1279 (m), 1252 (m), 1131 (w), 754 (w). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 209.3 (4.59), 243.9 (4.34), 271.1 (4.21). MS (EI, 70 eV): m/z (%) = 359 ([M+1]⁺, 1), 358 (M⁺, 1), 214 (5), 184 (3), 32 (25), 28 (100). HRMS (ESI) for C₂₄H₂₂O₃ ([M+1]⁺): Calcd. 359.16472; found: 359.16431.

Methyl 2,4-dimethyl-6-(2,5-dimethoxyphenyl)-3-ethyl benzoate (6i). Starting with **5b** (1.3 equiv., 0.416 g, 2.28 mmol), potassium phosphate (1.6 equiv., 0.597 g, 2.82 mmol) and Pd(PPh₃)₄ (3 mol-%, 0.061 g, 0.053 mmol), 8 mL of dioxane, and **4c** (1.0 equiv., 0.598 g, 1.76 mmol), **6i** was isolated as a colourless oil (0.375 g, 65%). ¹H NMR (300 MHz, CDCl₃): δ = 1.15 (t, J = 7.2 Hz, 3 H, CH₃CH₂), 2.35 (s, 6 H, 2 CH₃), 2.72 (q, J = 7.5 Hz, 2 H, CH₃CH₂), 3.55 (s, 3 H, CH₃O), 3.69 (s, 3 H, CH₃O), 3.76 (s, 3 H, CH₃O), 6.77 (dd, J = 1.4 Hz, 1.0 Hz, 1 H, Ar), 6.82 (s, H, Ar) 6.83 (s, 1 H, Ar), 6.99 (s, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.1, 16.5, 19.9 (CH₃), 22.7 (CH₂), 51.5, 55.7, 56.2 (CH₃), 111.9, 113.6, 116.2, 130.1 (CH), 130.9, 132.2, 132.9, 133.9, 137.2, 140.5, 150.7, 153.3, 170.4 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2968 (m), 1728 (s), 1502 (s), 1465 (s), 1266 (s), 1220 (s), 1157 (m), 1108 (m), 1047 (s), 799 (m). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 208.0 (4.60), 301.5 (3.72). MS (EI, 70 eV): m/z (%) = 329 ([M⁺+1], 21), 328 (M⁺, 100), 298 (13), 297 (70), 265 (27), 239 (22), 196 (5), 165 (4), 28 (3). Anal. calcd. for C₂₀H₂₄O₄: C 73.14, H 7.37; found: C 73.33, H 7.73.

Ethyl 2,4-dimethyl-6-(2,4-dimethoxyphenyl)-3-ethyl-benzoate (6j). Starting with **5c** (1.3 equiv., 0.316 g, 1.74 mmol), potassium phosphate (1.6 equiv., 0.454 g, 2.14 mmol), Pd(PPh₃)₄ (3 mol-%, 0.046 g, 0.040 mmol), 6 mL of dioxane, and **4c** (1.0 equiv., 0.455 g, 1.34 mmol), **6j** was isolated as a colourless oil (0.215 g, 47%). ¹H NMR (300 MHz, CDCl₃): δ = 0.97 (t, J = 7.1 Hz, 3 H, CH₃CH₂), 1.13 (t, J = 7.5 Hz, 3 H, OCH₂CH₃), 2.34 (s, 3 H, CH₃), 2.35 (s, 3 H, CH₃), 2.68 (q, J =

7.6 Hz, 2 H, CH₃CH₂), 3.72 (s, 3 H, CH₃O), 3.82 (s, 3 H, CH₃O), 4.02 (q, $J = 7.1$ Hz, 2 H, OCH₂CH₃), 6.46 - 6.50 (m, 2 H, Ar), 6.97 (s, 1 H, Ar), 7.05 - 7.09 (m, 1 H, Ar). MS (EI, 70 eV): m/z (%) = 342 ([M+1]⁺, 1), 342 (M⁺, 8), 193 (10), 188 (13), 175 (11), 32 (22), 28 (100). IR (KBr, cm⁻¹): $\tilde{\nu} = 2969$ (s), 2935 (s), 1724 (s), 1611 (s), 1583 (s), 1510 (s), 1461 (s), 1416 (s), 1301 (s), 1260 (s), 1209 (s), 1162 (s), 1041 (s), 833 (m). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 215.7 (4.47), 284.2 (3.71).

Ethyl 2,4-dimethyl-6-(1,3-dimethoxyphenyl)benzoate (6k). Starting with **5d** (1.3 equiv., 0.378 g, 1.60 mmol), potassium phosphate (1.6 equiv., 0.543 g, 2.56 mmol), Pd(PPh₃)₄ (3 mol-%, 0.055 g, 0.048 mmol), 3 mL of dioxane, and **4a** (1.0 equiv., 0.500 g, 0.86 mmol), **6k** was isolated by chromatography (silica gel, *n*-hexane/EtOAc = 20:1) as a colorless solid (0.284 g, 59%). ¹H NMR (300 MHz, CDCl₃): $\delta = 2.34$ (s, 3 H, CH₃), 2.40 (s, 3 H, CH₃), 3.47 (s, 3 H, CH₃O), 3.71 (s, 6 H, 2 CH₃O), 6.59 (s, 1 H, Ar), 6.61 (s, 1 H, Ar), 6.93 (s, 1 H, Ar), 7.01 (s, 1 H, Ar), 7.21-7.27 (dd, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): $\delta = 20.4$, 21.3 (CH₃), 51.1 (OCH₃), 55.9 (2 OCH₃), 103.9 (2CH), 118.9 (C), 128.7, 129.7, 130.4 (CH), 130.9, 133.5, 135.9, 139.2 (C), 157.5 (2C), 169.5 (CO). MS (EI, 70 eV): m/z (%) = 301 ([M⁺+1], 14), 300 (M⁺, 75), 270 (85), 269 (85), 253 (54), 239 (13), 238 (13), 211 (16), 177 (56), 28 (100). IR (KBr, cm⁻¹): $\tilde{\nu} = 3427$ (w), 2943 (m), 1721 (s), 1596 (s), 1471 (s), 1437 (s), 1278 (s), 1257 (s), 1185 (m), 1107 (s), 785 (w). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 203.4 (4.72).

Methyl 3-(2-chloroethyl)-6-(*o*-methoxyphenyl)-2,4-dimethylbenzoate (6l). Starting with **5a** (1.3 equiv., 0.296 g, 1.95 mmol), potassium phosphate (1.6 equiv., 0.509 g, 2.40 mmol) and Pd(PPh₃)₄ (3 mol-%, 0.052 g, 0.045 mmol), 6 mL of dioxane, and **4i** (1.0 equiv., 0.561 g, 1.5

mmol), **6l** was isolated as a colorless oil (0.390 g, 78%). ^1H NMR (300 MHz, CDCl_3): δ = 2.37 (s, 3 H, CH_3), 2.39 (s, 3 H, CH_3), 3.19 (m, J = 6.0 Hz, 2 H, ClCH_2CH_2), 3.51 (s, 3 H, CH_3O), 3.58 (m, 2 H, ClCH_2CH_2), 3.70 (s, 3 H, CH_3O), 6.90-6.93 (dd, 1.8 Hz, 8.3 Hz, 1 H, Ar), 6.96-6.98 (dd, J = 1.0 Hz, 6.4 Hz, 1 H, Ar), 7.03 (s, 1 H, Ar), 7.12-7.15 (dd, J = 1.8 Hz, 5.7 Hz, 1 H, Ar), 7.27-7.33 (m, J = 1.8 Hz, 5.7 Hz, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 16.9, 20.3 (CH_3), 33.5, 41.8 (CH_2), 51.5, 55.5 (CH_3), 110.6, 120.4, 128.9 (CH), 129.5 (C), 130.5, 130.7 (CH), 132.8, 133.9, 134.2, 135.6, 138.0, 156.3, 170.1 (C). MS (EI, 70 eV): m/z (%) = 334 (M^+ , [^{37}Cl], 32), 333 (19), 332 (M^+ , [^{35}Cl], 100), 301 (83), 265 (74), 239 (17), 209 (25), 165 (12), 28 (33). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3440 (w), 2972 (m), 1728 (s), 1600 (m), 1497 (s), 1460 (m), 1439 (m), 1389 (m), 1269 (s), 1248 (s), 1158 (m), 1048 (m), 756 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 210.4 (4.61), 281.4 (3.64).

Methyl 3-(2-bromoethyl)-6-(*o*-methoxyphenyl)-2,4-dimethylbenzoate (6m). Starting with **5a** (1.3 equiv., 0.250 g, 1.64 mmol), potassium phosphate (1.6 equiv., 0.429 g, 2.02 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (3%mol, 0.044 g, 0.038 mmol), 4 mL of dioxane, and **4j** (1.0 equiv., 0.530 g, 1.26 mmol), **6m** was isolated as a colorless oil (0.425 g, 89%). ^1H NMR (300 MHz, CDCl_3): δ = 2.36 (s, 3 H, CH_3), 2.38 (s, 3 H, CH_3), 3.26 (m, J = 1.8 Hz, 2 H, BrCH_2CH_2), 3.41 (m, J = 1.8 Hz, 2 H, BrCH_2CH_2), 3.51 (s, 3 H, CH_3O), 3.74 (s, 3 H, CH_3O), 6.89-6.93 (m, J = 8.3 Hz, 1 H, Ar), 6.95-6.98 (dd, J = 1.1 Hz, 7.3 Hz, 1 H, Ar), 7.03 (s, 1 H, Ar), 7.12-7.15 (dd, J = 1.8 Hz, 5.7 Hz, 1 H, Ar), 7.26-7.32 (m, J = 1.8 Hz, 5.7 Hz, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 16.8, 20.2 (CH_3), 29.2, 33.9 (CH_2), 51.5, 54.4 (CH_3), 110.5, 120.3, 128.9 (CH), 129.5 (C), 130.5, 130.7 (CH), 132.8, 133.7, 135.2, 135.6, 137.8, 156.2, 169.9 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2949 (m), 1727 (s), 1599 (m), 1497 (m), 1459 (s), 1438 (s), 1267 (s), 1253 (s), 1159 (m), 1049 (m), 910 (m), 756 (m), 732 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 210.7 (4.55), 281.4 (3.65). MS (EI, 70 eV): m/z (%) = 378

(M^+ , [^{81}Br], 1), 376 (M^+ , [^{79}Br], 1), 267 (17), 235 (4), 32 (14), 28 (100). HRMS (ESI): Calcd. for $\text{C}_{19}\text{H}_{21}\text{O}_3\text{Br}$: 376.06740; found: 376.06727.

General procedure for synthesis of benzo[*c*]chromen-6-ones **7 and **13** by lactonization with BBr_3 .** To a CH_2Cl_2 solution of **6** was added BBr_3 at 0 °C. The solution was allowed to warm to 20 °C during 18 h. To the solution was added an aqueous solution of KO^tBu (0.1 M) and the solution was stirred for 15 min. The organic and the aqueous layer were separated and the latter was extracted with CH_2Cl_2 . The combined organic layers were dried (Na_2SO_4), filtered and the filtrate was concentrated *in vacuo*. The product was purified by chromatography (silica gel; *n*-hexane/ EtOAc = 20:1) as a colourless solid.

7,9-Dimethyl-6*H*-benzo[*c*]chromen-6-one (7a**).** Starting with **6a** (0.240 g, 0.85 mmol) in CH_2Cl_2 (2.5 mL), BBr_3 (0.423 g, 1.69 mmol) and KO^tBu (10 mL, 0.1 M aqueous solution), **7a** was isolated as a colourless solid (0.175 g, 92%), mp. = 155.5 °C. ^1H NMR (300 MHz, CDCl_3): δ = 2.51 (s, 3 H, CH_3), 2.84 (s, 3 H, CH_3), 7.22 (s, 1 H, Ar), 7.29 (d, 1 H, Ar), 7.35 (m, 1 H, Ar), 7.44 (m, 1 H, Ar), 7.81 (s, 1 H, Ar), 8.05 (d, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 21.9, 23.7 (CH_3), 117.3 (CH), 118.3 (C), 120.1, 122.9, 124.1, 130.1, 133.4 (CH), 136.1, 144.3, 144.8, 151.5, 153.2, 160.5 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3429 (m), 2921 (m), 1721 (s), 1610 (s), 1452 (s), 1422 (m), 1265 (s), 1224 (s), 1169 (s), 1052 (s), 859 (m), 752 (s). UV-VIS (CH_3CN , nm): λ_{max} (log ϵ) = 237.9 (4.58), 263.1 (4.05), 272.9 (4.04), 288.2 (3.71), 298.6 (3.87), 317.1 (3.87). MS (EI, 70 eV): m/z (%) = 225 ($[\text{M}+1]^+$, 16), 224 (M^+ , 100), 195 (12), 181 (15), 165 (13), 151 (10), 128 (2), 91 (13). HRMS (ESI): Calcd. for $\text{C}_{15}\text{H}_{12}\text{O}_2$ ($[\text{M}+1]^+$): 225.09155; found 225.09142. Anal. calcd. for $\text{C}_{15}\text{H}_{12}\text{O}_2$: C 80.34, H 5.39; found: C 80.02, H 5.65.

7,8,9-Trimethyl-6H-benzo[*c*]chromen-6-one (7b). Starting with **6b** (0.284 g, 0.95 mmol) in CH₂Cl₂ (3 mL), BBr₃ (0.477 g, 1.9 mmol), KO^{*t*}Bu (10 mL, 0.1 M aqueous solution), **7b** was isolated as a colourless solid (0.160 g, 71%), mp. = 183 °C. ¹H NMR (300 MHz, CDCl₃): δ = 2.35 (s, 3 H, CH₃), 2.49 (s, 3 H, CH₃), 2.84 (s, 3 H, CH₃), 7.25-7.32 (m, 2 H, 2 CH), 7.39-7.45 (m, 1 H, CH), 7.81 (s, 1 H, CH), 8.01-8.05 (dd, 1 H, CH). ¹³C NMR (75 MHz, CDCl₃): δ = 16.2, 18.4, 22.2 (CH₃), 116.9 (CH), 117.8, 118.4, 120.5 (C), 122.6, 123.9, 129.6 (CH), 133.2, 137.7, 142.2, 143.7, 151.2, 160.8 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3428 (m), 2927 (m), 1726 (s), 1606 (s), 1473 (s), 1272 (s), 1210 (s), 1119 (s), 1010 (s), 753 (s). UV-VIS (CH₃CN, nm): λ_{max} (log ϵ) = 239.5 (4.59), 265.4 (4.06), 275.1 (4.08), 289.7 (3.83), 299.5 (3.92), 322.2 (3.87). MS (EI, 70 eV): m/z (%) = 239.0 ([M+1]⁺, 16), 238.0 (M⁺, 100), 222.9 (28), 195 (17), 165.0 (11), 151.3 (5), 114.4 (3), 28.1 (21). HRMS (ESI): Calcd. for C₁₆H₁₄O₂ ([M+1]⁺): 239.10721; found 239.10736. Anal. calcd. for C₁₆H₁₄O₂: C 80.61, H 5.92; found: C 80.1, H 6.18.

8-Ethyl-7,9-dimethyl-6H-benzo[*c*]chromen-6-one (7c). Starting with **6c** (1.0 equiv., 0.240 g, 0.79 mmol), 3 mL of CH₂Cl₂, BBr₃ (2 equiv., 0.396 g, 1.58 mmol) and KO^{*t*}Bu (10 mL, 0.1 M aqueous solution), **7c** was isolated as a colourless solid (0.162 g, 81%), mp. = 144 °C. ¹H NMR (300 MHz, CDCl₃): δ = 0.87 (t, J = 7.5 Hz, 3 H, CH₃CH₂), 2.53 (s, 3 H, CH₃), 2.82 (q, J = 7.5 Hz, 2 H, CH₃CH₂), 2.86 (s, 3 H, CH₃), 7.25-7.32 (m, 2H, Ar), 7.39 - 7.44 (m, J = 1.5 Hz, 5.7 Hz, 1 H, Ar), 7.80 (s, 1 H, Ar), 8.01-8.04 (dd, J = 1.5 Hz, 6.7 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 13.2, 17.8, 21.3 (CH₃), 22.9 (CH₂), 116.9 (CH), 118.1, 118.3 (C), 121.0, 122.6, 123.9, 129.6 (CH), 133.4, 141.8, 143.26, 143.33, 151.2, 160.8 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2970 (m), 1722 (m), 1720 (s), 1600 (s), 1476 (s), 1455 (s), 1398 (s), 1221 (s), 1209 (s), 1129 (s), 1015 (s), 750 (s). UV-VIS

(CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 230.1 (4.23), 239.8 (4.6), 265.4 (4.08), 275.5 (4.10), 289.9 (3.83), 300.0 (3.94), 322.1 (3.88). MS (EI, 70 eV): m/z (%) = 253 ([M+1]⁺, 4), 252 (M⁺, 36), 237 (40), 210 (30), 182 (11), 128 (2), 32 (23), 28 (100). HRMS (ESI): Calcd. for C₁₇H₁₆O₂ ([M+1]⁺): 253.12285; found 253.12223. Anal. calcd. for C₁₇H₁₆O₂: C 80.93, H 6.39; found: C 80.71, H 7.07.

9-Methyl-7-phenyl-6H-benzo[c]chromen-6-one (7d). Starting with **6d** (0.355 g, 1.02 mmol), 5 mL of CH₂Cl₂, BBr₃ (0.513 g, 2.04 mmol) and KOtBu (10 mL, 0.1 M aqueous solution), **7d** was isolated as a colourless solid (0.212 g, 73%), mp. = 107 °C. ¹H NMR (300 MHz, CDCl₃): δ = 2.56 (s, 3 H, CH₃), 7.24 (d, 1 H, Ar), 7.30 - 7.35 (m, 4 H, Ar), 7.39 - 7.41 (m, 2 H, Ar), 7.42 - 7.45 (dd, 1 H, Ar), 7.47 - 7.49 (dd, 1 H, Ar), 7.97 (s, 1 H, Ar), 8.09 - 8.12 (dd, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 21.9 (CH₃), 116.2 (C), 117.4 (CH), 118.1 (C), 121.4, 122.9, 124.1, 127.2 (CH), 128.2 (2 CH), 130.3, 133.6 (CH), 136.1, 141.9, 144.4, 146.8, 151.6, 159.2 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3048 (w), 1737 (s), 1607 (s), 1448 (m), 1274 (m), 1252 (m), 1219 (m), 1072 (m), 1044 (m), 864 (w), 754 (s), 701 (m), 615 (w). UV-VIS (CH₃CN, nm): $\lambda_{\max}$ (log ϵ) = 211.0 (4.45), 239.1 (4.14), 290.3 (3.77), 272.9 (4.14), 301.3 (3.84), 322.1 (3.89). MS (EI, 70 eV): m/z (%) = 287 ([M+1]⁺, 0.4), 286 (M⁺, 3), 285 (4), 32 (29), 28 (100). HRMS (ESI) for C₂₀H₁₄O₂ ([M+1]⁺): Calcd. 287.10720; found 287.10668. Anal. calcd. for C₂₀H₁₄O₂: C 83.89, H 4.93; found: C 83.46, H 5.26.

10-Hydroxy-7,9-dimethyl-6H-benzo[c]chromen-6-one (7e). Starting with **6e** (0.15 mmol, 0.045 g), 2 mL of CH₂Cl₂, BBr₃ (0.075 g, 0.30 mmol) and KOtBu (5 mL, 0.1 M aqueous solution), **7e** was isolated as a yellow solid (0.029 g, 81%). ¹H NMR (300 MHz, CDCl₃): δ = 2.43 (s, 3 H, CH₃), 2.78 (s, 3 H, CH₃), 5.30 (s, 1 H, OH), 7.19 (d, J = 1.0 Hz, 1 H, Ar), 7.28 - 7.34 (m, 1.6 Hz, 6.1 Hz, 2 H, Ar), 7.40 - 7.43 (m, J = 1.5 Hz, 5.5 Hz, 1 H, Ar), 9.04 - 9.08 (dd, J = 1.7 Hz, 6.7 Hz, 1 H, Ar).

^{13}C NMR (75 MHz, CDCl_3): δ = 16.4, 23.6 (CH_3), 116.8 (CH), 119.2, 118.2 (C), 123.9 (CH), 128.4 (2 CH), 129.3 (CH), 129.4, 133.7 (C), 134.2 (2 C), 136.1, 149.5 (C). MS (EI, 70 eV): m/z (%) = 241 ($[\text{M}+1]^+$, 11), 240 (M^+ , 68), 169 (20), 152 (75), 139 (29), 44 (22), 28 (100). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3401 (br, w), 2923 (w), 2360 (w), 1701 (s), 1606 (w), 1259 (s), 1160 (w), 756 (w).

1,2,3,4-Tetrahydronaphtho[1,2-*c*]chromen-5-one (7f). Starting with **6f** (1.0 equiv., 0.238 g, 0.77 mmol), 4 mL of CH_2Cl_2 , BBr_3 (2 equiv., 0.384 g, 1.54 mmol), $\text{KO}t\text{Bu}$ (8 mL, 0.1 M aqueous solution), **7f** was isolated (0.164 g, 91%) by column chromatography (n -hexane/EtOAc = 20:1) as a colourless solid, mp. 143 °C. ^1H NMR (300 MHz, CDCl_3): δ = 1.80-1.89 (m, J = 1.5 Hz, 4 H, 2 CH_2), 2.90 (t, J = 6.0 Hz, 2 H, CH_2), 3.43 (t, J = 6.1 Hz, 2 H, CH_2), 7.28 - 7.33 (m, 2 H, Ar), 7.40-7.46 (m, 1 H, Ar), 7.49 - 7.52 (d, 1 H, Ar), 7.89 - 7.92 (d, 1 H, Ar), 8.01 - 8.04 (dd, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): δ = 21.8, 23.2, 29.3, 30.7 (CH_2), 116.9 (CH), 118.4 (C), 118.6 (CH), 119.3 (C), 122.6, 123.9, 129.6 (CH), 133.9, 135.7, 139.1, 142.9, 150.9, 160.2 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3421(w), 2924 (m), 1726 (s), 1596 (m), 1474 (m), 1291 (m), 1255 (m), 1207 (m), 1107 (s), 998 (m), 816 (m), 759 (s), 455 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 231.4 (4.53), 255.8 (4.02), 264.8 (4.03), 274.3 (4.09), 290.2 (3.75), 300.2 (3.83), 326.7 (3.94). MS (EI, 70 eV): m/z (%) = 251 ($[\text{M}+1]^+$, 1), 250 (M^+ , 8), 235 (5), 191 (2), 125 (8), 40(10), 32 (79). HRMS (ESI): Calcd. for $\text{C}_{20}\text{H}_{14}\text{O}_2$ ($[\text{M}+1]^+$): 251.10720; found 251.10670.

1,2,3,4-Tetrahydro-12-methylnaphtho[1,2-*c*]chromen-5-one (7g). Starting with **6g** (1.0 equiv., 0.140 g, 0.45 mmol), 2 mL of CH_2Cl_2 , BBr_3 (2.0 equiv., 0.225 g, 0.90 mmol) and $\text{KO}t\text{Bu}$ (5 mL, 0.1 M aqueous solution), **7g** was isolated as a colourless solid (0.080 g, 67%), mp. = 140 °C. ^1H NMR (300 MHz, CDCl_3): δ = 1.84 (m, 2 H, CH_2), 1.99-2.09 (m, 2 H, CH_2), 2.77 (t, 2 H, CH_2),

3.37 (m, 2 H, CH₂), 7.27 - 7.37 (m, 1 H, Ar), 7.47 - 7.53 (m, 1 H, Ar), 8.07 (s, 1 H, Ar), 8.23 - 8.28 (m, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 20.9 (CH₂), 22.1 (CH₃), 22.8, 27.9, 30.0 (CH₂), 116.9 (CH), 118.4, 119.9 (C), 120.2, 122.6, 123.9 (CH), 129.5 (C), 129.6 (CH), 138.1, 143.1, 144.3, 151.2, 160.4 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3434 (m), 2034 (s), 2966 (m), 1722 (s), 1603 (s), 1469 (m), 1261 (m), 1270 (m), 1110 (s), 756 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 205.2 (3.92), 240.3 (4.59), 265.8 (4.06), 275.5 (4.07), 289.9 (3.83), 300.1 (3.91), 323.2 (3.89). MS (EI, 70 eV): *m/z* (%) = 264 (M⁺, 4), 249 (4), 219 (3), 191 (5), 145 (4), 105 (4), 43 (5), 32 (24), 28 (100). HRMS (ESI): Calcd. for C₁₈H₁₆O₂ ([M+1]⁺): 265.12286; found: 265.12222. Anal. calcd. for C₁₈H₁₆O₂: C 81.79, H 6.10; found C 81.90, H 6.85.

1,2-Dihydro-14-methylphenanthro[4,3-*c*]chromen- 7-one (7h). Starting with **6h** (1.0 equiv., 0.280 g, 0.78 mmol), 4 mL of CH₂Cl₂, BBr₃ (2.0 equiv., 0.391 g, 1.56 mmol) and KO^tBu (10 mL, 0.1 M aqueous solution), **7h** was isolated as a colourless solid (0.215 g, 88%), mp. = 234 °C. ¹H NMR (300 MHz, CDCl₃): δ = 2.52 (s, 3 H, CH₃), 2.77 (m, *J* = 6.0 Hz, 2 H, CH₂), 2.88 (m, *J* = 6.4 Hz, 2 H, CH₂), 7.20 - 7.30 (m, *J* = 1.5 Hz, 3 H, Ar), 7.31 - 7.35 (m, *J* = 1.6 Hz, 1 H, Ar), 7.34 - 7.38 (m, *J* = 8.0 Hz, 1.6 Hz, 1 H, Ar), 7.44 - 7.49 (m, *J* = 1.5 Hz, 8.3 Hz, 1 H, Ar), 7.51 - 7.54 (dd, *J* = 7.2 Hz, 1.7 Hz, 1 H, Ar), 7.87 (s, 1 H, Ar), 8.06 - 8.09 (dd, *J* = 6.5 Hz, 1.5 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 20.9 (CH₃), 25.9, 28.8 (CH₂), 115.1 (C), 117.2 (CH), 118.2 (C), 121.6, 122.7, 124.0, 125.7, 126.8, 128.2, 129.9, 131.0 (CH), 133.2, 134.7, 137.8, 139.5, 141.1, 142.3, 151.4, 160.3 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3435 (br, m), 1726 (s), 1601 (m), 1452 (m), 1272 (m), 1210 (m), 1116 (s), 755 (s). UV-VIS (CH₃CN, nm): λ_{max} (logε) = 202.9 (4.63), 226.8 (4.31), 258.5 (4.49), 290.7 (4.13), 349.3 (3.99). MS (EI, 70 eV): *m/z* (%) = 312 (M⁺, 3), 311 (4), 252 (1),

202 (1), 105 (1), 32 (39), 28 (100). HRMS (ESI) for $C_{22}H_{16}O_2$ ($[M+1]^+$): Calcd. 313.12286; found 313.12245. Anal. calcd. for $C_{22}H_{16}O_2$: C 61.64, H 4.56; found: C 61.82, H 4.95.

8-Ethyl-2-hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7i). Starting with **6i** (1.0 equiv., 0.316 g, 0.96 mmol), 5 mL of CH_2Cl_2 , BBr_3 (4.0 equiv., 0.963 g, 3.85 mmol) and $KOtBu$ (20 mL, 0.1 M aqueous solution), **7i** was isolated as a red solid (0.203 g, 79%), mp. = 243 °C. 1H NMR (300 MHz, $(CD_3)_2CO$): δ = 1.12 (t, J = 7.5 Hz, 3 H, CH_3CH_2), 2.39 (s, 3 H, CH_3), 2.61 (s, 3 H, CH_3), 2.71 (q, J = 7.5 Hz, 2 H, CH_3CH_2), 6.59 (d, J = 8.8 Hz, 1 H, Ar), 6.97 (d, J = 8.9 Hz, 1 H, Ar), 7.54 (s, 1 H, Ar), 8.56 (s, 1 H, Ar), 8.73 (s, 1 H, OH). ^{13}C NMR (75 MHz, $(CD_3)_2CO$): δ = 13.5, 13.9, 21.2 (CH_3), 22.6 (CH_2), 119.5 (C), 119.9, 124.5, 125.7 (CH), 126.7 (C), 127.7 (CH), 130.0, 138.7, 143.0, 143.1, 144.2, 147.3, 152.3 (C). MS (EI, 70 eV): m/z (%) = 269 ($[M+1]^+$, 0.23), 268 (M^+ , 2), 253 (4), 151 (1), 121 (2), 40 (4), 32 (58), 28 (100). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3367 (s), 2966 (m), 1655 (s), 1609 (s), 1596 (s), 1461 (s), 1382 (m), 1276 (s), 1256 (m), 1199 (s), 1161 (m), 1089 (m), 943 (m), 822 (m), 612 (m). UV-VIS (CH_3CN , nm): λ_{max} (log ϵ) = 212.7 (4.24), 255.7 (4.74), 328.8 (3.39), 370.9 (3.63), 442.1 (3.57). Anal. calcd. for $C_{17}H_{16}O_3$: C 76.10, H 6.01; found C 76.26, H 6.48.

8-Ethyl-3-hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7j). Starting with **6j** (1.0 equiv., 0.055 g, 0.16 mmol), 1.5 mL of CH_2Cl_2 , BBr_3 (4.0 equiv., 0.160 g, 0.64 mmol) and $KOtBu$ (3 mL, 0.1 M aqueous solution), **7j** was isolated as a colourless solid (0.041 g, 96%). 1H NMR (300 MHz, $(CD_3)_2CO$): δ = 1.15 (t, J = 7.5 Hz, 3 H, CH_3CH_2), 2.06 (q, J = 7.5 Hz, 2 H, CH_3CH_2), 2.78 (s, 3 H, CH_3), 2.88 (s, 3 H, CH_3), 6.71 (d, 1 H, Ar), 6.86 (dd, 1 H, Ar), 7.90 (s, 1 H, Ar), 8.07 (d, 1 H, Ar), 9.11 (s, 1 H, OH). ^{13}C NMR (75 MHz, $(CD_3)_2CO$): δ = 14.3, 18.6, 21.8 (CH_3), 23.9 (CH_2),

103.9 (CH), 112.0 (C), 114.1 (CH), 118.2 (C), 122.2, 125.9 (CH), 135.7, 142.4, 143.4, 145.1, 154.2, 160.8, 161.7 (C). MS (EI, 70 eV): m/z (%) = 269 ($[M+1]^+$, 2), 268 (M^+ , 47), 253 (83), 152 (7), 32 (22), 28 (100). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3404 (s), 3371 (s), 2969 (m), 1700 (s), 1621 (s), 1457 (m), 1254 (m), 1216 (m), 1027 (w), 858 (w), 628 (w). UV-VIS (CH_3CN): λ_{max} ($\log \epsilon$) = 218.9 (4.39), 243.6 (4.51), 269.8 (4.05), 279.0 (4.13), 305.7 (4.01), 335.0 (3.89).

1-Hydroxy-7,9-dimethyl-6H-benzo[*c*]chromen-6-one (7k). Starting with **6k** (1.0 equiv., 0.063 g, 0.12 mmol), 1.5 mL of CH_2Cl_2 , BBr_3 (4.0 equiv., 0.120 g, 0.48 mmol) and KO^tBu (4 mL, 0.1 M aqueous solution), **7k** was isolated as a colourless solid (0.025 g, 87%). ^1H NMR (300 MHz, CDCl_3): δ = 2.47 (s, 3 H, CH_3), 2.78 (s, 3 H, CH_3), 6.81 - 6.84 (dd, 1 H, Ar), 6.89 - 6.92 (dd, 1 H, Ar), 7.28 (s, 1 H, Ar), 7.32 (d, 1 H, Ar), 9.01 (s, 1 H, Ar), 9.79 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 22.2, 24.0 (CH_3), 106.7 (C), 107.6, 112.2 (CH), 116.4 (C), 125.8, 130.2, 132.6 (CH), 136.5, 142.8, 144.7, 152.7, 156.8, 159.9 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3188 (m), 3117 (m), 1684 (s), 1606 (s), 1481 (s), 1376 (m), 1246 (s), 1175 (m), 1087 (m), 1034 (m), 740 (w). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 225.7 (4.50), 240.8 (4.53), 262.9 (4.10), 270.6 (4.09), 281.0 (4.00), 317.1 (3.84). MS (EI, 70 eV): m/z (%) = 242 ($[M+2]^+$, 1), 241 ($[M+1]^+$, 16), 240 (M^+ , 100), 225 (20), 197 (15), 181 (7), 114 (9), 28 (8). Anal. calcd. for $\text{C}_{15}\text{H}_{12}\text{O}_3$: C 74.97, H 5.03; found: C 75.32, H 5.48.

8-(2-Chloroethyl)-7,9-dimethyl-6H-benzo[*c*]chromen-6-one (7l). Starting with **6l** (1.0 equiv., 0.352 g, 1.05 mmol), 5 mL of CH_2Cl_2 , BBr_3 (2.0 equiv., 0.530 g, 2.11 mmol), and KO^tBu (15 mL, 0.1 M aqueous solution), **7l** was isolated as a colourless solid (0.240 g, 80%), mp. = 165 °C. ^1H NMR (300 MHz, CDCl_3): δ = 2.58 (s, 3 H, CH_3), 2.88 (s, 3 H, CH_3), 3.32 (m, J = 7.5 Hz, 2 H, ClCH_2CH_2), 3.61 (m, J = 7.6 Hz, 2 H, ClCH_2CH_2), 7.29-7.33 (m, 2 H, Ar), 7.42-7.45 (m, J = 1.5

Hz, 6.9 Hz, 1 H, Ar), 7.85 (s, 1 H, Ar), 8.02-8.05 (dd, $J = 1.6$ Hz, 6.4 Hz, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 18.4, 21.9$ (CH_3), 33.3, 41.9 (CH_2), 117.1 (CH), 117.9 (C), 121.4, 122.8, 124.1, 130.1 (CH), 130.7, 134.7, 137.0, 142.8, 144.1, 151.4, 160.5 (C). IR (KBr, cm^{-1}): $\tilde{\nu} = 2974$ (w), 1725 (s), 1601 (m), 1472 (m), 1266 (m), 1206 (m), 1119 (s), 1012 (m), 762 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 239.9 (4.61), 265.9 (4.10), 289.9 (3.84), 321.4 (3.98). MS (EI, 70 eV): m/z (%) = 288 ($[\text{M}+1]^+$, 12), 287 (M^+ , 4), 286 (27), 251 (44), 237 (51), 165 (8), 32 (25), 28 (100). Anal. calcd. for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{Cl}$: C 71.19, H 5.27; found C 71.11, H 5.78. HRMS (ESI): Calcd. for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{Cl}$ ($[\text{M}+1]^+$): 287.08389; found 287.08347

8-(2-Bromoethyl)-7,9-dimethyl-6H-benzo[*c*]chromen-6-one (7m). Starting with **6m** (1.0 equiv., 0.405 g, 1.07 mmol), 5 mL of CH_2Cl_2 , BBr_3 (2.0 equiv., 0.538 g, 2.15 mmol) and KO^tBu (5 mL, 0.1 M aqueous solution), **7m** was isolated as a colourless solid (0.270 g, 76%), mp. = 182 °C. ^1H NMR (300 MHz, CDCl_3): $\delta = 2.54$ (s, 3 H, CH_3), 2.83 (s, 3 H, CH_3), 3.36-3.47 (m, 4 H, BrCH_2CH_2), 7.29-7.32 (m, 2 H, Ar), 7.42-7.47 (m, $J = 1.5$ Hz, 5.8 Hz, 1 H, Ar), 7.83 (s, 1 H, Ar), 8.04 (dd, $J = 1.6$ Hz, 6.5 Hz, 1 H, Ar). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 18.3, 21.7$ (CH_3), 29.0, 33.6 (CH_2), 117.0 (CH), 117.9, 118.3 (C), 121.4, 122.7, 124.1, 130.1 (CH), 134.5, 137.9, 142.6, 143.8, 151.3, 160.3 (C). IR (KBr, cm^{-1}): $\tilde{\nu} = 2972$ (w), 1722 (s), 1603 (m), 1474 (m), 1400 (m), 1251 (m), 1205 (s), 1125 (m), 1016 (m), 733 (m). UV-VIS (CH_3CN , nm): λ_{max} ($\log \epsilon$) = 240.3 (4.62), 266.6 (4.13), 276.3 (4.18), 290.3 (3.89), 300.5 (3.96), 321.5 (3.91). MS (EI, 70 eV): m/z (%) = 332 ($[\text{M}^++1]$, 39), 331 (M^+ , 5), 330 (40), 251 (100), 237 (82), 165 (15), 32 (19), 28 (80). Anal. calcd. for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{Br}$: C 61.64, H 4.56; found C 61.82, H 4.95. HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{Br}$ ($[\text{M}+1]^+$): 331.03336; found 331.03336.

7-Hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13a). Starting with **12a** (0.278 g, 0.971 mmol) in CH₂Cl₂ (15 mL), BBr₃ (0.973 g, 3.89 mmol) and KO^{*t*}Bu (20 mL, 0.1 M aqueous solution), **13a** was isolated as a colourless solid (0.219 g, 92%), mp. = 150-151 °C. ¹H NMR (250 MHz, CDCl₃): δ = 2.42 (s, 3 H, CH₃), 6.77-6.78 (m, 1 H, Ar), 7.15 (s, 1 H, Ar), 7.21 (dd, *J* = 7.0 Hz, *J* = 1.5 Hz, 1 H, Ar), 7.25-7.27 (m, 1H,Ar), 7.37 (ddd, *J* = 8.3 Hz, *J* = 7.0 Hz, *J* = 1.5 Hz, 1 H, Ar), 7.90 (dd, *J* = 7.9 Hz, *J* = 1.2 Hz, 1 H, Ar), 11.20 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 23.0 (CH₃), 104.3 (C), 113.5, 117.4, 118.1 (CH), 118.7 (C) 123.7, 125.4, 130.9 (CH), 135.3, 149.4, 151.1, 162.8, 165.8 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3438 (m), 3068 (m), 2923 (w), 1680 (s), 1627 (s), 1568 (s), 1512 (w), 1456 (m), 1421 (m), 1276 (s), 1235 (s), 1208 (s), 1148 (m), 1078(s), 842 (m), 750 (s), 511 (m). GC-MS (EI, 70 eV): *m/z* (%) = 226 (M⁺, 100), 197 (20), 169 (8), 141 (8), 115 (11), 77 (3). HRMS (EI): Calcd. for C₁₄H₁₀O₃: 226.06245; found 226.061951.

7-Hydroxy-8,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (13b). Starting with **12b** (0.100 g, 0.349 mmol) in CH₂Cl₂ (10 mL), BBr₃ (0.349 g, 1.39 mmol) and KO^{*t*}Bu (10 mL, 0.1 M aqueous solution), **13b** was isolated as a colourless solid (0.058 g, 69%), mp. = 164-166 °C. ¹H NMR (250 MHz, CDCl₃): δ = 2.20 (s, 3 H, CH₃), 2.39 (s, 3 H, CH₃), 7.18 (s, 1 H, Ar), 7.28 (dd, *J* = 7.6 Hz, *J* = 1.2 Hz, 1 H, Ar), 7.34-7.35 (m, 1 H, Ar), 7.39 (ddd, *J* = 8.5 Hz, *J* = 6.8 Hz, *J* = 1.2 Hz, 1 H, Ar), 7.95 (dd, *J* = 7.9 Hz, *J* = 1.5 Hz, 1 H, Ar), 11.55 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 10.3, 20.3 (CH₃), 28.6 (CH₂), 102.4 (C), 112.2, 116.4 (CH), 117.4 (C), 121.8 (CH) 122.9 (C), 123.8, 128.7 (CH), 130.5, 146.6, 149.3, 158.9, 164.6 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2955 (m), 2923 (s), 2852 (m), 1670 (s), 1608 (m), 1560 (m), 1520 (m), 1457 (m), 1410 (m), 1361 (m), 1319 (m), 1280 (s), 1235 (s), 1246 (m), 1175 (m), 1136 (s), 1094 (m), 903 (w), 800 (w), 759 (s), 546 (w). GC-MS

(EI, 70 eV): m/z (%) = 240 (M^+ , 100), 225 (39), 211 (5), 197 (6), 181 (3), 165 (8), 152 (7), 139 (4), 115 (6), 76 (3). HRMS (EI): Calcd. for $C_{15}H_{12}O_3$: 240.07810; found 240.07779.

8-Ethyl-7-hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13c). Starting with **12c** (0.100 g, 0.333 mmol) in CH_2Cl_2 (10 mL), BBr_3 (0.334 g, 1.33 mmol) and $KOtBu$ (10 mL, 0.1 M aqueous solution), **13c** was isolated as a colourless solid (0.052 g, 62%), mp. = 109-111 °C. 1H NMR (250 MHz, $CDCl_3$): δ = 1.16 (t, J = 7.6 Hz, 3 H, CH_3), 2.41 (s, 3 H, CH_3), 2.70 (q, J = 7.3 Hz, 2 H, CH_2), 7.20 (s, 1 H, Ar), 7.22-7.24 (m, 1 H, Ar), 7.25-7.26 (m, 1 H, Ar), 7.36 (ddd, J = 7.6 Hz, J = 7.6 Hz, J = 1.5 Hz, 1 H, Ar), 7.89 (dd, J = 7.9 Hz, J = 1.2 Hz, 1 H, Ar), 11.50 (s, 1 H, OH). ^{13}C NMR (62 MHz, $CDCl_3$): δ = 12.8 (CH_3), 19.2 (CH_2), 20.4 (CH_3), 103.6 (C), 113.5, 117.4 (CH), 118.3 (C), 122.7, 124.8, 129.7 (CH), 130.5, 131.6, 146.5, 150.3, 159.8, 165.6 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3437 (m), 2967 (m), 1671 (s), 1622 (m), 1610 (m), 1560 (m), 1453 (m), 1404 (m), 1227 (s), 1177 (m), 1135 (m), 1110 (m), 903 (w), 807 (m), 769 (s). GC-MS (EI, 70 eV): m/z (%) = 254 (M^+ , 46), 240 (22), 239 (100), 152 (8), 115 (4), 76 (3). HRMS (EI): Calcd. for $C_{16}H_{14}O_3$: 254.09375; found 254.09304.

8-Hexyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13d). Starting with **12d** (0.100 g, 0.270 mmol) in CH_2Cl_2 (10 mL), BBr_3 (0.271 g, 1.079 mmol) and $KOtBu$ (10 mL, 0.1 M aqueous solution), **13d** was isolated as a colourless solid (0.061 g, 65%), mp. = 88-90 °C. 1H NMR (250 MHz, $CDCl_3$): δ = 1.33 (t(br), J = 7.0 Hz, 3 H, CH_3), 1.19-1.29 (m, 8 H, CH_2), 2.86 (s, 3 H, CH_3), 3.12 (t, J = 7.0 Hz, 2 H, CH_2), 7.19 (s, 1 H, Ar), 7.26 (ddd, J = 8.5 Hz, J = 8.2 Hz, J = 0.9 Hz, 1 H, Ar), 7.29-7.30 (m, 1 H, Ar), 7.38 (ddd, J = 7.3 Hz, J = 7.0 Hz, J = 1.5 Hz, 1 H, Ar), 7.94 (dd, J = 8.2 Hz, J = 1.2 Hz, 1 H, Ar), 11.52 (s, 1 H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.1, 19.8

(CH₃), 21.1, 25.1, 27.7, 28.7, 30.7 (CH₂), 102.7 (C), 112.6, 116.6 (CH), 117.6 (C), 121.9, 123.9 (CH), 128.6 (C), 128.7 (CH), 130.8, 145.8, 149.5, 159.2, 164.8 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3057 (w), 2955 (m), 2922 (s), 2853 (m), 1673 (s), 1624 (m), 1609 (m), 1556 (w), 1458 (m), 1404 (m), 1366 (w), 1227 (s), 1179 (m), 1137 (m), 1120 (m), 1037 (w), 906 (w), 804 (m), 769 (m), 751 (m), 621 (w). GC-MS (EI, 70 eV): m/z (%) = 310 (M⁺, 22), 292 (2), 263 (5), 240 (34), 239 (100), 165 (5), 115 (2). HRMS (EI): Calcd. for C₂₀H₂₂O₃: 310.15635; found 310.15649.

8-Heptyl-7-hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13e). Starting with **12e** (0.150 g, 0.405 mmol) in CH₂Cl₂ (12 mL), BBr₃ (0.406 g, 1.619 mmol) and KO^tBu (15 mL, 0.1 M aqueous solution), **13e** was isolated as a colourless solid (0.071 g, 54 %), mp. = 87-88 °C. ¹H NMR (250 MHz, CDCl₃): δ = 0.84 (t(br), J = 6.8 Hz, 3 H, CH₃), 1.21-1.26 (m, 10 H, CH₂), 2.42 (s, 3 H, CH₃), 2.68 (t, J = 7.3 Hz, 2 H, CH₂), 7.19 (s, 1 H, Ar), 7.21-7.24 (m, 1 H, Ar), 7.26-7.29 (m, 1 H, Ar), 7.36 (ddd, J = 7.9 Hz, J = 7.0 Hz, J = 1.5 Hz, 1 H, Ar), 7.93 (dd, J = 7.9 Hz, J = 2.1 Hz, 1 H, Ar), 11.52 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 13.9, 20.7 (CH₃), 22.5, 25.9, 28.7, 29.2, 29.8, 31.7 (CH₂), 103.5 (C), 113.5, 117.4 (CH), 118.4 (C), 122.7, 124.7 (CH), 129.4 (C), 129.7 (CH), 131.6, 146.7, 150.3, 159.9, 165.6 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3057 (m), 2956 (s), 2922 (s), 2853 (s), 1672 (s), 1624 (m), 1609 (m), 1559 (m), 1458 (m), 1404 (m), 1362 (w), 1269 (s), 1178 (m), 1137 (m), 1121 (m), 1045 (w), 906 (w), 811 (w), 768 (m), 625 (w). GC-MS (EI, 70 eV): m/z (%) = 324 (M⁺, 24), 253 (4), 240 (36), 239 (100), 165 (5), 115 (2), 77 (1). HRMS (EI): Calcd. for C₂₁H₂₄O₃: 324.17200; found 324.17233.

7-Hydroxy-9-methyl-8-octyl-6*H*-benzo[*c*]chromen-6-one (13f). Starting with **12f** (0.250 g, 0.627 mmol) in CH₂Cl₂ (20 mL), BBr₃ (0.651 g, 2.509 mmol) and KO^tBu (20 mL, 0.1 M aqueous

solution), **13d** was isolated as a colourless solid (0.128 g, 60%), mp. = 87-89 °C. ^1H NMR (300 MHz, CDCl_3): δ = 0.81 (t(br), J = 7.5 Hz, 3 H, CH_3), 1.18-1.21 (m, 12 H, CH_2), 2.37 (s, 3 H, CH_3), 2.63 (t, J = 7.0 Hz, 2 H, CH_2), 7.19 (s, 1 H, Ar), 7.22-7.24 (m, 1 H, Ar), 7.25-7.26 (m, 1 H, Ar), 7.35 (ddd, J = 8.1 Hz, J = 7.0 Hz, J = 1.8 Hz, 1 H, Ar), 7.89 (dd, J = 7.9 Hz, J = 1.2 Hz, 1 H, Ar), 11.46 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 13.9, 20.6 (CH_3), 22.5, 25.6, 28.5, 29.2, 29.3, 29.8, 31.3 (CH_2), 102.8 (C), 113.4, 117.5, 122.6, 124.7, 129.3 (CH), 129.6, 131.5, 146.6, 147.4, 150.2, 159.9, 165.6 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2950 (m), 2919 (s), 2850 (s), 1680 (s), 1625 (m), 1610 (m), 1556 (m), 1458 (m), 1405 (m), 1334 (m), 1273 (s), 1178 (m), 1135 (s), 1121 (m), 1045 (w), 906 (w), 757 (s), 625 (w), 550 (w), 471 (w). GC-MS (EI, 70 eV): m/z (%) = 338 (M^+ , 22), 253 (4), 240 (36), 239 (100), 207 (9), 152 (5), 115 (2), 77(1). HRMS (EI): Calcd. for $\text{C}_{22}\text{H}_{26}\text{O}_3$: 338.18765; found 338.18719.

7-Hydroxy-9-methyl-8-undecyl-6H-benzo[c]chromen-6-one (13g). Starting with **12g** (0.250 g, 0.586 mmol) in CH_2Cl_2 (20 mL), BBr_3 (0.587 g, 2.4 mmol) and $\text{KO}t\text{Bu}$ (20 mL, 0.1 M aqueous solution), **13g** was isolated as a colourless solid (0.158 g, 71%), mp. = 90–91 °C. ^1H NMR (300 MHz, CDCl_3): δ = 0.80 (t(br), J = 6.6 Hz, 3 H, CH_3), 1.17-1.18 (m, 18 H, CH_2), 2.38 (s, 3 H, CH_3), 2.63 (t, J = 7.4 Hz, 3 H, CH_3), 7.21 (dd, J = 6.8 Hz, J = 1.3 Hz, 1 H, Ar), 7.24 (m, 1 H, Ar), 7.26 (m, 1 H, Ar), 7.34 (ddd, J = 7.6 Hz, J = 7.0 Hz, J = 1.5 Hz, 1 H, Ar), 7.89 (dd, J = 7.6 Hz, J = 1.5 Hz, 1 H, Ar), 11.47 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 11.4, 21.1 (CH_3), 23.0, 26.5, 29.1, 29.7, 29.9, 30.0, 30.1, 30.3, 31.6, 32.3 (CH_2), 104.0 (C), 113.9, 117.9 (CH), 118.9 (C), 123.2, 125.2 (CH), 129.9 (C), 130.2 (CH), 132.1, 147.1, 150.8, 160.5, 166.1 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3435 (m), 3106 (m), 2955 (s), 2919 (s), 2848 (s), 1670 (s), 1672 (m), 1560 (w), 1458 (m), 1358 (w), 1274 (s), 1213 (m), 1135 (s), 1044 (w), 906 (w), 749 (s). GC-MS (EI, 70 eV): m/z (%) = 380

(M^+ , 16), 281 (3), 240 (35), 239 (100), 207 (13), 165 (4), 115 (2), 44(3). HRMS (EI): Calcd. for $C_{25}H_{32}O_3$: 380.23460; found 380.23443.

7-Hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13h). Starting with **12h** (0.100 g, 0.333 mmol) in CH_2Cl_2 (8 mL), BBr_3 (0.336 g, 1.341 mmol) and $KOtBu$ (20 mL, 0.1 M aqueous solution), **13h** was isolated as a colourless solid (0.065 g, 77%), mp. = 97-99 °C. 1H NMR (300 MHz, $CDCl_3$): δ = 0.92 (t, J = 7.2 Hz, 3 H, CH_3), 1.63-1.70 (m, 2 H, CH_2), 2.64 (t, J = 7.4 Hz, 2 H, CH_2), 6.84-6.85 (m, 1 H, Ar), 7.19 (s, 1 H, Ar), 7.27 (dd, J = 8.3 Hz, J = 0.7 Hz, 1 H, Ar), 7.32 (dd, J = 8.4 Hz, J = 0.9 Hz, 1 H, Ar), 7.41 (ddd, J = 8.4 Hz, J = 6.8 Hz, J = 1.7 Hz, 1 H, Ar), 7.97 (dd, J = 8.3 Hz, J = 1.7 Hz, 1 H, Ar), 11.22 (s, 1 H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, (CH₃), 24.3, 39.2 (CH_2), 104.5 (C), 112.9, 116.8, 118.1 (CH), 118.8 (C), 123.6, 125.3, 130.8 (CH), 135.3, 151.1, 154.0, 162.8, 165.7 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3436 (m), 3139 (w), 2962 (m), 2931 (m), 2872 (w), 1678 (s), 1625 (s), 1565 (s), 1511 (w), 1424 (s), 1319 (m), 1226 (s), 1104 (w), 1071 (s), 835 (w), 767 (s), 715 (m). GC-MS (EI, 70 eV): m/z (%) = 254 (M^+ , 65), 239 (14), 226 (100), 211 (2), 197 (13), 181 (11), 165 (5), 152 (10), 139 (7), 127 (3), 115 (10), 76 (3). HRMS (EI): Calcd. for $C_{16}H_{14}O_3$: 254.09375; found 254.09365.

7-Hydroxy-8-methyl-9-propyl-6*H*-benzo[*c*]chromen-6-one (13i). Starting with **12i** (0.076 g, 0.241 mmol) in CH_2Cl_2 (7 mL), BBr_3 (0.242 g, 0.964 mmol) and $KOtBu$ (20 mL, 0.1 M aqueous solution), **13i** was isolated as a colourless solid (0.047 g, 73%), mp. = 113-115 °C. 1H NMR (300 MHz, $CDCl_3$): δ = 0.96 (t, J = 7.2 Hz, 3 H, CH_3), 1.57-1.65 (m, 2 H, CH_2), 2.22 (s, 3 H, CH_3), 2.67 (t, J = 7.6 Hz, 2 H, CH_2), 7.19 (s, 1 H, Ar), 7.28 (dd, J = 8.3 Hz, J = 0.9 Hz, 1 H, Ar), 7.33 (m(br), 1 H, Ar), 7.38 (ddd, J = 8.7 Hz, J = 8.1 Hz, J = 1.5 Hz, 1 H, Ar), 7.96 (dd, J = 8.0 Hz, J =

1.7 Hz, 1 H, Ar), 11.57 (s, 1 H, OH). ^{13}C NMR (125 MHz, CDCl_3): δ = 11.1, 14.1 (CH_3), 23.3, 36.7 (CH_2), 103.5 (C), 112.6, 117.6 (CH), 118.6 (C), 122.9 (CH), 124.2 (C), 124.9, 129.8 (CH), 131.7, 150.4, 151.5, 160.4, 165.8 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3065 (m), 2965 (s), 2924 (s), 2853 (s), 1668 (s), 1624 (w), 1607 (m), 1516 (w), 1427 (m), 1326 (w), 1271 (s), 1157 (m), 1146 (m), 801 (m), 767 (s). GC-MS (EI, 70 eV): m/z (%) = 268 (M^+ , 100), 253 (48), 240 (74), 225 (37), 181 (5), 152 (12), 115 (7), 89 (3) 76 (3). HRMS (EI): Calcd. for $\text{C}_{17}\text{H}_{16}\text{O}_3$: 268.10940; found 268.10923.

8-Ethyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13j). Starting with **12j** (0.084 g, 0.246 mmol) in CH_2Cl_2 (7 mL), BBr_3 (0.246 g, 0.981 mmol) and $\text{KO}t\text{Bu}$ (20 mL, 0.1 M aqueous solution), **13j** was isolated as a colourless solid (0.049 g, 71%), mp. = 96-98 °C. ^1H NMR (300 MHz, CDCl_3): δ = 0.98 (t, J = 7.4 Hz, 3 H, CH_3), 1.12 (t, J = 7.4 Hz, 3 H, CH_3), 1.59-1.67 (m, 2 H, CH_2), 2.67 (q, J = 8.0 Hz, 2 H, CH_2), 2.72 (t, J = 7.6 Hz, 2 H, CH_2), 7.18 (s, 1 H, Ar), 7.26 (dd, J = 8.0 Hz, J = 0.7 Hz, 1 H, Ar), 7.32-7.34 (m, 1 H, Ar), 7.37 (ddd, J = 8.7 Hz, J = 7.0 Hz, J = 1.5 Hz, 1 H, Ar), 7.95 (dd, J = 7.9 Hz, J = 1.7 Hz, 1 H, Ar), 11.54 (s, 1 H, OH). ^{13}C NMR (75 MHz, CDCl_3): δ = 12.7, 13.1 (CH_3), 18.0, 23.3, 34.9 (CH_2), 102.6 (C), 111.7, 116.5 (CH), 117.6 (C), 121.8, 123.8, 128.8 (CH), 129.3, 130.8, 149.4, 149.9, 159.4, 164.8 (C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 2963 (s), 2930 (m), 2870 (m), 1669 (s), 1621 (m), 1609 (m), 1557 (m), 1468 (m), 1404 (m), 1332 (m), 1249 (s), 1139 (s), 1005 (w), 806 (w), 750 (s). GC-MS (EI, 70 eV): m/z (%) = 282 (M^+ , 72), 267 (100), 254 (6), 239 (36), 226 (21), 208 (5), 181 (7), 165 (11), 115 (4), 89 (2) 76 (3). HRMS (EI): Calcd. for $\text{C}_{18}\text{H}_{18}\text{O}_3$: 282.12505; found 282.12507.

8-Hexyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13k). Starting with **12k** (0.067 g, 0.168 mmol) in CH_2Cl_2 (6 mL), BBr_3 (0.168 g, 0.672 mmol) and $\text{KO}t\text{Bu}$ (20 mL, 0.1 M aqueous

solution), **13k** was isolated as a colourless solid (0.035 g, 62%), mp. = 78-80 °C. ¹H NMR (300 MHz, CDCl₃): δ = 0.83 (t(br), *J* = 7.6 Hz, 3 H, CH₃), 0.98 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.18-1.26 (m, 10 H, CH₂), 1.61 (t, *J* = 7.6 Hz, 2 H, CH₂), 2.66 (t, *J* = 7.6, 2 H, CH₂), 7.18 (s, 1 H, Ar), 7.27 (dd, *J* = 7.6 Hz, *J* = 0.9 Hz, 1 H, Ar), 7.32 (m, 1 H, Ar), 7.37 (ddd, *J* = 8.5 Hz, *J* = 7.0 Hz, *J* = 1.5 Hz, 1 H, Ar), 7.95 (dd, *J* = 8.2 Hz, *J* = 1.5 Hz, 1 H, Ar), 11.54 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 13.0, 13.2 (CH₃), 21.6, 23.2, 24.8, 28.4, 28.6, 30.6, 35.0 (CH₂), 102.5 (C), 111.7, 116.5 (CH), 117.6 (C), 121.8, 123.8 (CH), 128.2 (C), 128.8 (CH), 130.7, 149.4, 150.1, 159.4, 164.8 (C). IR (Nujol, cm⁻¹): $\tilde{\nu}$ = 2961 (s), 2924 (s), 2852 (m), 1683 (w), 1622 (w), 1611 (w), 1462 (w), 1409 (w), 1268 (s), 1122 (s), 1021 (m), 864 (w), 803 (m), 789 (m), 670 (w). GC-MS (EI, 70 eV): *m/z* (%) = 338 (M⁺, 34), 320 (5), 295 (12), 281 (10), 267 (100), 239 (28), 207 (9), 165 (18), 149 (13), 125 (18), 111 (30), 83 (50), 69 (60), 57 (80). HRMS (EI): Calcd. for C₂₂H₂₆O₃: 338.18765; found 338.18733.

7-Hydroxy-9-propyl-8-undecyl-6H-benzo[*c*]chromen-6-one (13l). Starting with **12l** (0.250 g, 0.551 mmol) in CH₂Cl₂ (10 mL), BBr₃ (0.552 g, 2.204 mmol) and KO^{*t*}Bu (10 mL, 0.1 M aqueous solution), **13l** was isolated as a colourless solid (0.168 g, 86%), mp. = 76 – 78 °C. ¹H NMR (300 MHz, CDCl₃): δ = 0.69 (t, *J* = 7.0 Hz, 3 H, CH₃), 0.86 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.05-1.08 (m, 20 H, CH₂), 1.48 (t, *J* = 7.4 Hz, 2 H, CH₂), 2.51 (t, *J* = 7.4, 2 H, CH₂), 7.05 (s, 1 H, Ar), 7.07 (m, 1 H, Ar), 7.12 (m, 1 H, Ar), 7.19 (ddd, *J* = 8.0 Hz, *J* = 7.4 Hz, *J* = 1.5 Hz, 1 H, Ar), 7.74 (dd, *J* = 8.0 Hz, *J* = 1.5 Hz, 1 H, Ar), 11.38 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 14.4, 14.6 (CH₃), 23.0, 24.6, 26.2, 29.7, 29.8, 29.9, 30.0, 30.0, 30.1, 30.4, 32.3, 36.4 (CH₂), 103.9 (C), 113.0, 117.8 (CH), 118.9 (C), 123.1, 125.1 (CH), 129.5 (C), 130.1 (CH), 132.0, 150.7, 151.4, 160.8, 166.1 (C). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3431 (m), 3062 (w), 2961 (s), 2919 (s), 2850 (s), 1660 (s), 1625 (m), 1611

(m), 1560 (w), 1467 (m), 1407 (s), 1332 (m), 1269 (s), 1179 (m), 1138 (s), 1124 (m), 811 (w), 753 (s). GC-MS (EI, 70 eV): m/z (%) = 408 (M^+ , 32), 365 (10), 291 (4), 268 (35), 267 (100), 239 (24), 226 (19), 208 (5), 165 (5), 115 (2), 41 (7). HRMS (EI): Calcd. for $C_{27}H_{36}O_3$: 408.26590; found: 408.26586.

Procedure for the synthesis of 4-hydroxy-4-(2-methoxyphenyl)-3-buten-2-one (10a). To a stirred solution of LDA (75.0 mmol) in THF (62 mL) was added acetone (2.904 g, 50.0 mmol) at -78 °C. After the solution was stirred for 1 h, 2-methoxy anisoyl chloride (10.235 g, 60.0 mmol) was added. The temperature of the solution was allowed to rise to 20 °C during 12 h. A saturated solution of NH_4Cl was added, the layers were separated, and the aqueous layer was extracted with ethylacetate (3 x 150 mL). The combined organic layers were dried (Na_2SO_4) and filtered, and the solvent was removed in vacuo. The residue was purified by chromatography (silica gel, *n*-hexane/EtOAc 30:1 → 20:1) to give **10a** as a yellowish oil (3.550 g, 37%, mixture of keto-enol tautomers). 1H NMR (250 MHz, $CDCl_3$): δ = 1.99 (s, 3 H, CH_3), 3.69 (s, 3 H, OCH_3), 6.28 (s, 1 H, CH), 6.76 (dd, J = 8.5 Hz, J = 0.9 Hz, 1 H, Ar), 6.84 (ddd, J = 7.3 Hz, J = 7.3 Hz, J = 0.9 Hz, 1 H, Ar), 7.22 (ddd, J = 8.5 Hz, J = 8.2 Hz, J = 1.8 Hz, 1 H, Ar), 7.70 (dd, J = 7.6 Hz, J = 1.8 Hz, 1 H, Ar), 15.3 (s(br), 1 H, OH). ^{13}C NMR (62 MHz, $CDCl_3$): δ = 26.1 (CH_3), 55.4 (OCH_3), 101.9, 111.6, 120.9, 130.8, 133.1 (CH), 134.8, 158.4, 181.3, 194.6 (C). IR (Nujol, cm^{-1}): $\tilde{\nu}$ = 3076 (w), 3005 (w) 2962 (w), 1721 (m), 1603 (s), 1490 (s), 1250 (s), 1164 (m), 1022 (m), 989 (m), 755 (m), 533 (w). MS (EI, 70 eV): m/z (%) = 192 ($[M]^+$, 12), 174 (10), 161 (54), 136 (10), 135 (100), 120 (5), 105 (4), 92 (11), 77 (25), 63.1 (5), 51 (5), 43 (11). HRMS (EI): Calcd. for $C_{11}H_{12}O_3$: 192.07810; found: 192.07797. Anal. calcd. for $C_{11}H_{12}O_3$: C 68.73, H 6.29; found: C 69.16, H 6.52.

Procedure for the synthesis of 1-hydroxy-1-(2-methoxyphenyl)-1-hexen-3-one (10b). To a stirred solution of LDA (6.0 mmol) in THF (5 mL) was added 2-Pentanone (0.344 g, 4.0 mmol) at -78 °C. After the solution was stirred for 1 h, 2-methoxy anisoyl chloride (0.818 g, 4.8 mmol) was added. The temperature of the solution was allowed to rise to 20 °C during 12 h. A saturated solution of NH₄Cl was added, the layers were separated, and the aqueous layer was extracted with ethylacetate (3 x 50 mL). The combined organic layers were dried (Na₂SO₄) and filtered, and the solvent was removed in vacuo. The residue was purified by chromatography (silica gel, *n*-hexane/EtOAc 30:1 → 20:1) to give **10b** as a yellowish oil (0.647 g, 73%, mixture of keto-enol tautomers). ¹H NMR (300 MHz, CDCl₃): δ = 0.86 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.53-1.61 (m, 2 H, CH₂), 2.26 (t, *J* = 7.3 Hz, 2 H, CH₂), 3.74 (s, 3 H, OCH₃), 6.32 (s, 1 H, CH), 6.80 (m, 1 H, Ar), 6.88 (dd, *J* = 7.6 Hz, *J* = 0.9 Hz, 1 H, Ar), 7.27 (ddd, *J* = 8.5 Hz, *J* = 8.4 Hz, *J* = 1.9 Hz, 1 H, Ar), 7.74 (dd, *J* = 7.8 Hz, *J* = 1.9 Hz, 1 H, Ar), 16.2 (s(br), 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 12.7 (CH₃), 18.2, 40.2 (CH₂), 54.5 (OCH₃), 100.3, 110.6, 119.6 (CH), 123.2 (C), 128.7, 131.9 (CH), 157.3, 180.5, 196.3 (C). IR (Nujol, cm⁻¹): $\tilde{\nu}$ = 2963 (m), 2935 (m), 2874 (w), 2839 (w), 1718 (w), 1668 (w), 1604 (s), 1490 (s), 1465 (m), 1293 (m), 1250 (s), 1165 (m), 1067 (w), 1067 (w), 1023 (m), 805 (w), 754 (m). MS (EI, 70 eV): *m/z* (%) = 220 ([M]⁺, 7), 202 (4), 189 (23), 177 (24), 135 (100), 120 (3), 105 (2), 92 (8), 77 (17), 63.1 (3), 43 (5). HRMS (EI): Calcd. for C₁₃H₁₆O₃: 220.10940; found: 220.10898. Anal. calcd. for C₁₃H₁₆O₃: C 70.88, H 7.32; found: C 70.55, H 7.58.

Procedure for the synthesis of 4-(2-methoxyphenyl)-4-[(trimethylsilyl)oxy]-3-buten-2-one (11a). To a stirred benzene solution (37 mL) of **10a** (2.854 g, 14.9 mmol) was added triethylamine (2.406 g, 23.8 mmol). After the solution was stirred for 2 h, trimethylchlorosilane (2.905 g, 26.7 mmol) was added. After the solution was stirred for 72 h, the solvent was removed in vacuo and

hexane (75 mL) was added to the residue to give a suspension. The later was filtered under argon atmosphere. The filtrate was concentrated in vacuo to give **11a** as yellowish oil (2.893 g, 73%, mixture of *E/Z*-isomers). ¹H NMR (250 MHz, CDCl₃): δ = 0.21 (s, 9 H, 3CH₃), 2.31 (s, 3 H, CH₃), 3.78 (s, 3 H, OCH₃), 6.16 (s, 1 H, CH), 6.84-6.86 (m, 1 H, Ar), 6.90 (dd, *J* = 8.7 Hz, *J* = 1.1 Hz, 1 H, Ar), 7.28-7.30 (m, 1 H, Ar), 7.51 (dd, *J* = 8.7 Hz, *J* = 2.1 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 0.16 (3 CH₃), 21.9 (CH₃), 55.1 (OCH₃), 101.3, 111.0, 119.8, 130.5, 131.5 (CH), 132.7, 157.2, 169.7, 191.3 (C).

Procedure for the synthesis of 1-(2-methoxyphenyl)-1-[(trimethylsilyl)oxy]-1-hexen-3-one (11b). To a stirred benzene solution (55 mL) of **10a** (5.0 g, 22.7 mmol) was added triethylamine (3.675 g, 36.3 mmol). After the solution was stirred for 2 h, trimethylchlorosilane (4.439 g, 40.8 mmol) was added. After the solution was stirred for 72 h, the solvent was removed in vacuo and hexane was added to the residue (250 mL) to give a suspension. The later was filtered under argon atmosphere. The filtrate was concentrated in vacuo to give **11b** as yellowish oil (5.024 g, 75%, mixture of *E/Z*-isomers). ¹H NMR (300 MHz, CDCl₃): δ = 0.23 (s, 9 H, 3CH₃), 0.90 (m, 3 H, CH₃), 1.51-1.59 (m, 2 H, CH₂), 2.72 (t, *J* = 7.6 Hz, CH₃), 3.75 (s, 3 H, OCH₃), 6.15 (s, 1 H, CH), 6.82-6.83 (m, 1 H, Ar), 6.89 (dd, *J* = 6.3 Hz, *J* = 0.5 Hz, 1 H, Ar), 7.26-7.28 (m, 1 H, Ar), 7.53 (dd, *J* = 8.3 Hz, *J* = 1.8 Hz, 1 H, Ar). ¹³C NMR (75 MHz, CDCl₃): δ = 0.5 (3 CH₃), 13.9 (CH₃), 20.3, 36.2 (CH₂), 55.6 (OCH₃), 101.3, 109.3 (CH), 111.0 (C), 120.2, 120.6, 131.6 (CH), 157.4, 173.3, 191.0 (C).

Copies of NMR spectra:

Methyl 4,5,6-trimethyl-2-hydroxybenzoate (3b).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 1H-NMR-Spektrum (Schnellmessung)
 Van: HV142
 Lösungsmittel: Deuteriochloroform, CDCl3
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV142
 EXPNO 1
 PROCNO 1
 USER speedy

F2 - Acquisition Parameters
 Date_ 20031205
 Time 11.20
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.110571 Hz
 AQ 4.5220342 sec
 RG 1430
 DW 69.000 usec
 DE 98.57 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 10.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300065 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ernst-Moritz-Arndt-Universitaet Greifswald
ARX300 - Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
Van: HV142
Loesungsmittel: Deuteriochloroform, CDCl3
Referenz: Loesungsmittel = 77.0 ppm
Mess temperatur: 298 K (25 oC)

Current Data Parameters
NAME VA HV142
EXPNO 13
PROCNO 1
USER lang

F2 - Acquisition Parameters
Date_ 20031223
Time 9.10
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 640
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.030000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 16384
SF 75.4677578 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

Methyl 4,6-dimethyl-5-ethyl-2-hydroxybenzoate (3c).

Ernst-Moritz-Arndt-Universität Greifswald

ARX300 – Kernresonanz-Spektrometer

¹H-NMR-Spektrum (Schnellmessung)

Van: HV176

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV176
EXPNO 1
PROCNO 1
USER speedy

F2 – Acquisition Parameters

Date_ 20040203
Time 10.06

INSTRUM spect

PROBHD 5 mm Multinu

PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 32

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 2048

DW 82.000 usec

DE 102.50 usec

TE 300.0 K

D1 1.00000000 sec

P1 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS ¹H

F2 – Processing parameters

SI 16384

SF 300.1300066 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitenbandenkoppelung)

Van: HV176

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Loesungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV176
EXPNO 13
PROCNO 1
USER lang

F2 - Acquisition Parameters
Date_ 20040304
Time 15.51
INSTRUM spect
PROBHD 5 mm Maltinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 256
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.0002000 sec
DL6 30.00 dB
D1 2.0000000 sec
CPDPRG walz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 3.00 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 16384
SF 75.4677512 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

Ethyl 4-methyl-6-phenyl-2-hydroxybenzoate (3d).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 13C-NMR-Spektrum, Protonenbreitbandentkoppelt
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Probenname:
 siehe nebenstehendes Parameterpanel
 (z.B.: NAME XY_kp89 bedeutet
 Auftraggeber: XY
 Probenname: kp89)

Current Data Parameters
 NAME VA_HV365
 EXPNO 13
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041122
 Time 13.53
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 131072
 SOLVENT CDCl3
 NS 384
 DS 0
 SWH 22727.273 Hz
 FIDRES 0.173395 Hz
 AQ 2.8836341 sec
 RG 32768
 DW 22.000 usec
 DE 31.43 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 1.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 20.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 32768
 SF 75.4677502 MHz
 WDW EM
 SSB 0
 LB 0.70 Hz
 GB 0
 PC 1.00

Methyl 2-hydroxy-3-methoxy-4,6-dimethylbenzoate (3e).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum

Var: HV292

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV292
EXPNO 1
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20040913
Time 8.52

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 16

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 1430

DW 82.000 usec

DE 102.50 usec

TE 300.0 K

D1 1.00000000 sec

P1 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS 1H

F2 – Processing parameters

SI 16384

SF 300.1300055 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV292
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV292
 EXPNO 13
 PROCNO 1
 USER lang
 F2 – Acquisition Parameters
 Date_ 20040913
 Time 9.16
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 512
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C
 F2 – Processing parameters
 SI 16384
 SF 75.4677480 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

Ethyl 5,6,7,8-tetrahydro-2-hydroxynaphthalene-1-carboxylate (3f).

Ernst-Moritz-Arnt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
IH-NMR-Spektrum

Var: HV378
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: TMS = 0.0 ppm
Mess temperatur: 298 K (25 °C)

Current Data Parameters
NAME VA HV378
EXPNO 1
PROCNO 1
USER lang

F2 - Acquisition Parameters
Date_ 20041217
Time 12.26
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 16
DS 0
SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 2860
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.00000000 sec
PI 3.00 usec
SFO1 300.1322456 MHz
NUCLEUS ¹H

F2 - Processing parameters
SI 16384
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV378
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV378
 EXPNO 13
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20041217
 Time 13.23
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 1784
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TB 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG walz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 2.50 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C

F2 - Processing parameters
 SI 32768
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 1.00

Methyl 5,6,7,8-tetrahydro-3-hydroxy-4-methylnaphthalene-12-carboxylate (3g).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Probenname:

Current Data Parameters
 NAME VA_HV247-1
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20040712
 Time 10.01
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 1024
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300045 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Methyl 9,10-dihydro-3-hydroxy-1-methylphenanthrene-4-carboxylate (3h).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Lösungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Probenname:
 siehe nebenstehendes Parameterpanel
 B.: NAME XY_kp89 bedeutet
 Auftraggeber: XY
 Probenname: kp89)

Current Data Parameters
 NAME VA_HV404R2
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041209
 Time 10:00
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 2860
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300061 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ethyl 3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12a).

HUSSAIN 40C, 1H, CDCL3

OK300S, 106
 AU PROG:
 X00, AU
 DATE 30-10-6'
 SF 250.133
 SY 250.0
 O1 4311.794
 SI 32768
 TD 32768
 SW 5000.000
 HZ/PT .305
 PW 0.0
 RD 0.0
 AQ 3.277
 RG 100
 NS 48
 TE 297
 FW 6300
 O2 2714.499
 DP 63L P0
 LB 0.0
 GB .100
 CX 23.00
 CY 12.00
 F1 12.000F
 F2 - .499F
 HZ/CM 135.936
 PPM/CM .543
 SR 2871.67

HUSSAIN 40C, 13C, CDCL3 ✓

Hussain AH40XX

Methyl 3-hydroxy-2'-methoxy-4,5-dimethyl[1,1'-biphenyl]-2-carboxylate (12b).

HUSSAIN 63C1, 1H, CDCL3

DZ010S.103
AU PROG:
X00.AU
DATE 1-12-6

SF 250.133
SY 250.0
O1 4311.794
SI 32768
TD 32768
SW 5000.000
HZ/PT .305

PW 0.0
RD 0.0
AQ 3.277
RG 80
NS 48
TE 297

FW 6300
O2 2714.499
DP 63L P0

LB 0.0
GB .100
CX 23.00
CY 12.00
F1 12.000f
F2 -.499f
HZ/CM 135.936
PPM/CM .543
SR 2886.01

Methyl 4-ethyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12 c).

HUSSAIN 52C1, 1H, CDCL3

NO100S.101
 AU PROG:
 X00 AU
 DATE 10-11-6
 SF 250.133
 SY 250.0
 O1 4311.794
 SI 32768
 TD 32768
 SW 5000.000
 HZ/PT 305
 PW 0.0
 RD 0.0
 AQ 3.277
 RG 40
 NS 48
 TE 297
 FW 6300
 O2 2714.499
 DP 63L P0
 LB 0.0
 GB 100
 CX 23.00
 CY 12.00
 F1 12.000
 F2 12.000
 HZ/CM 135.936
 PPM/CM 543
 SR 2872.28

Ethyl 4-hexyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12d).

HUSSAIN 65RC, H, CDCL3

DZ050S.106
 AU PROG:
 X00. AU
 DATE 5-12-6
 SF 250.13
 SY 250.0
 O1 4311.79
 SI 32768
 TD 32768
 SW 5000.00
 HZ/PT 30
 PW 0.0
 RD 0.0
 AQ 3.27
 RG 10
 NS 48
 TE 297
 FW 6300
 O2 2714.49
 DP 63L P0
 LB 0.0
 GB 10
 CX 23.00
 CY 12.00
 F1 12.00
 F2 -49
 HZ/CM 135.93
 PPM/CM 54
 SR 2874.11

Methyl 4-heptyl-3-hydroxy-2'-methoxy-5-methyl[1,1'-biphenyl]-2-carboxylate (12e).

Tuan DTT 161A

Current Data Parameters
NAME 061207
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061208
Time 0.43
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DCC13
NS 2500
DS 4
SWH 19230.770 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 32768
DM 26.000 usec
DE 37.14 usec
TE 300.0 K
D12 0.00002000 sec
DL6 21.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 100.00 usec
D11 0.03000000 sec
DL5 15.00 dB
P1 7.50 usec
SF01 75.4770618 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 75.4685040 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16225.73 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/1
HZCM 830.15356 Hz/cm

Ethyl 3-hydroxy-2'-methoxy-5-methyl-4-octyl[1,1'-biphenyl]-2-carboxylate (12f).

HUSSAIN 57C1 1H, CDCL3

N0170S.108
 AU PROG:
 X00.AU
 DATE 17-11-6
 SF 250.133
 SY 250.0
 O1 4311.794
 SI 32768
 TD 32768
 SW 5000.000
 HZ/PT .305
 PW 0.0
 RD 0.0
 AQ 3.277
 RG 4
 NS 48
 TE 297
 FW 6300
 O2 2714.499
 DP 63L P0
 LB 0.0
 GB .100
 CX 23.00
 CY 12.00
 F1 12.0000
 F2 -.499
 HZ/CM 135.936
 PPM/CM .543
 SR 2901.27

Methyl 3-hydroxy-2'-methoxy-5-methyl-4-undecyl[1,1'-biphenyl]-2-carboxylate (12g).

Tuan DTT172, 1H CDC13

Current Data Parameters
NAME 070205
EXPNO 130
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070205
Time 13:11

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30

TD 65536
SOLVENT CDC13

NS 40
DS 2

SWH 6250.000 Hz
FIDRES 0.096367 Hz

AQ 5.2429299 sec
RG 90

DW 80.000 usec
DE 114.29 usec

TE 300.0 K
D1 1.00000000 sec

P1 8.00 usec
SF01 300.1351790 MHz

NUCLEUS 1H

F2 - Processing parameters
SI 32768

SF 300.1334276 MHz
WDW EM

SSB 0
LB 0.30 Hz

GB 0
PC 1.00

4D NMR plot parameters
CX 20.00 cm

F1P 11.000 ppm
F1 3301.47 Hz

F2P -1.000 ppm
F2 -300.13 Hz

PPMCM 0.60000 ppm/cm
HZCM 180.08005 Hz/cm

Tuan DT1172

Methyl 3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12h).

Hussain 94R, 14 CDCl₃

Current Data Parameters
 NAME 070205
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070205
 Time 11.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 24
 DS 2
 SWH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 256
 DW 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1334165 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08005 Hz/cm

Hussain 94R

Methyl 3-hydroxy-2'-methoxy-4-methyl-5-propyl[1,1'-biphenyl]-2-carboxylate (12i).

Hussain 95C1 1H CDCl3

Current Data Parameters
NAME 070122
EXPNO 160
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070122
Time 11.52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.095367 Hz
AQ 5.2429299 sec
RG 256
DM 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 8.00 usec
SF01 300.1351790 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 32768
SF 300.1333961 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.000 ppm
F1 3301.47 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.08003 Hz/cm

Methyl 4-ethyl-3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12j).

Hussain 96C1 1H CDC13

Current Data Parameters
NAME 070122
EXPNO 180
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070122
Time 12.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.095367 Hz
AQ 5.2429299 sec
RG 180
DW 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 8.00 usec
SF01 300.1351790 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 300.1334309 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3301.47 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.08005 Hz/cm

Hussain 96c1

Ethyl 4-hexyl-3-hydroxy-2'-methoxy-5-propyl[1,1'-biphenyl]-2-carboxylate (12k).

Yaver Y69C1, 1H CDC13

Current Data Parameters
 NAME 070205
 EXPNO 70
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070205
 Time 11.36
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 24
 DS 2
 SMH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 180
 DW 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1334016 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08003 Hz/cm

Yawer Y69C1

Current Data Parameters
NAME 070205
EXPNO 310
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070206
Time 1.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 19230.770 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 32768
DM 26.000 usec
DE 37.14 usec
TE 300.0 K
D12 0.00002000 sec
DL6 21.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 100.00 usec
D11 0.03000000 sec
DL5 15.00 dB
P1 7.50 usec
SF01 75.4770618 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 75.4685631 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16225.74 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/c
HZCM 830.15424 Hz/cm

Methyl 3-hydroxy-2'-methoxy-5-propyl-4-undecyl[1,1'-biphenyl]-2-carboxylate (12l).

Tuan DTT173, 1H CDC13

Tuan DTT173

Ethyl 2,4-dimethyl-6-(trifluoromethylsulfonyloxy)benzoate (4a).

Ernst-Moritz-Amdt-Universität Greifswald

ARX300 - Kernresonanz-Spektrometer

¹H-NMR-Spektrum

Van: HV254

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Mess temperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV254
EXPNO 1
PROCNO 1
USER lang

F2 - Acquisition Parameters

Date_ 20040728
Time 12.24

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 16

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 2048

DW 82.000 usec

DE 102.50 usec

TB 300.0 K

D1 1.00000000 sec

P1 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS ¹H

F2 - Processing parameters

SI 16384

SF 300.1300059 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonen Breitbandkoppelt)
 Van: HV254
 Lösungsmittel: Deuteriochloroform, CDCl3
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV254
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20040728
 Time 13.01
 INSTRUM spect
 PROBHID 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 2.50 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 16384
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

[2-(Methoxycarbonyl)-3,4,5-trimethylphenyl]-trifluoromethanesulfonate (4b).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum

Var: HV311

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV311
EXPNO 1
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20050512
Time 15.26
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 32
DS 0
SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 1430
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 300.1322456 MHz
NUCLEUS 1H

F2 – Processing parameters
SI 16384
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV311
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)
 Messzeit: 13 Stunden 5 Minuten

Current Data Parameters
 NAME VA_HV311
 EXPNO 13
 PROCNO 1
 USER lang
 F2 – Acquisition Parameters
 Date_ 20050512
 Time 16.08
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 16896
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C
 F2 – Processing parameters
 SI 32768
 SF 75.4677505 MHz
 WDW EM
 SSB 0
 LB 0.70 Hz
 GB 0
 PC 0.80

[2-(Methoxycarbonyl)-4-ethyl-3,5-dimethylphenyl]-trifluoromethanesulfonate (4c).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer

¹H-NMR-Spektrum

Van: HV397

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV397
EXPNO 1
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20050104
Time 12.26

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 16

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 1430

DW 82.000 usec

DE 102.50 usec

TE 300.0 K

D1 1.00000000 sec

PI 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS ¹H

F2 – Processing parameters

SI 16384

SF 300.1300061 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Nguyen T. H. Van #V 397

[2-(Methoxycarbonyl)-5-methyl-3-phenylphenyl]-trifluoromethanesulfonate (4d).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelt)

an: HV308

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Messzeit: 1 Stunde 30 Minuten

Current Data Parameters
NAME VA_HV308
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20040928
Time 9:30
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 1792
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384
SF 75.4677499 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 0.90

[2-(Methoxycarbonyl)-6-methoxy-3,5-dimethylphenyl]-trifluoromethanesulfonate (4e).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV303
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)
 Messzeit: 12 Stunden 41 Minuten

Current Data Parameters
 NAME VA_HV303
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20050517
 Time 17.21
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 16384
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 32768
 SF 75.4677486 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

[5-(Ethoxycarbonyl)-1,2,3,4-tetrahydronaphthalen-6-yl]-trifluoromethanesulfonate (4f).

Ernst-Moritz-Arndt-Universität Gröfswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitenbandenkoppelt)

Van: HV370

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV370
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20041217
Time 10.09
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 2764
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384
SF 75.4677499 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00

[5-(Methoxycarbonyl)-1,2,3,4-tetrahydro-8-methylnaphthalen-6-yl]-trifluoromethanesulfonate (4g).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 -NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 nz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 °C)
 Auftraggeber und Probenname:
 siehe nebenstehendes Parameterpanel
 (z.B.: NAME XY_kp89 bedeutet
 Auftraggeber: XY
 Probenname: kp89)

Current Data Parameters
 NAME VA_HV385
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041109
 Time 8.32
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 24
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 2048
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300063 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Nguyen HV 385, 13C, CDC13

[4-(Methoxycarbonyl)-9,10-dihydro-1-methylphenanthren-3-yl]-trifluoromethanesulfonate (4h).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Problemname:

Current Data Parameters
 NAME VA_HV408R
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters

Date 20041210
 Time 9:45
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 48
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 4096
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1300065 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV408
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)
 Messzeit: 1 Stunde 25 Minuten

Current Data Parameters
 NAME VA_HV408
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20050418
 Time 13.52
 INSTRUM spect
 PROBHD 5 mm Multin
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 1792
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 PL 3.00 usec
 SFO1 75.4761177 M
 NUCLEUS 13C

F2 – Processing parameters
 SI 32768
 SF 75.4677495 MH
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 1.20

[2-(Methoxycarbonyl)-4-(2-chloroethyl)-3,5-dimethylphenyl]-trifluoromethanesulfonate (4i).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandentkoppelt)
 Van: HV337
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA HV337
 EXPNO 13
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20041014
 Time 13.18
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 1536
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C

F2 - Processing parameters
 SI 16384
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

[2-(Methoxycarbonyl)-4-(2-bromoethyl)-3,5-dimethylphenyl]-trifluoromethanesulfonate (4j).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer

¹H-NMR-Spektrum

Van: HV338

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV338
EXPNO 1
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20041012
Time 11.41
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 16
DS 0
SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 2860
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 300.1322456 MHz
NUCLEUS ¹H

F2 – Processing parameters
SI 16384
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandentkoppelt)
 Van: HV338
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV338
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20041012
 Time 12.56
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 1536
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.0000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.0300000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C

F2 – Processing parameters
 SI 16384
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

$C_{18}H_{20}O_3$

Exact Mass: 284.14

Mol. Wt.: 284.35

C, 76.03; H, 7.09; O, 16.88

Ethyl 2,4-dimethyl-6-(2-methoxyphenyl)benzoate (6a).

Ernst-Moritz-Armdt-Universität Greifswald

ARX300 – Kernresonanz-Spektrometer

¹H-NMR-Spektrum (Schnellmessung)

Van: HV275

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters	
NAME	VA_HV275
EXPNO	1
PROCNO	1
USER	speedy

F2 – Acquisition Parameters

Time	8.29	spec
INSTRUM	5 mm Multinu	
PROBHD	zg	
PULPROG	65536	
TD	CDC13	
SOLVENT	24	
NS	0	
DS	697.561 Hz	
SWH	0.093041 Hz	
FIDRES	5.3740020 sec	
AQ	2048	
RQ	82.000 usec	
DW	102.50 usec	
DE	300.0 K	
TE	1.0000000 sec	
DT	3.00 usec	
PI	300.1322456 MH	
SFO1	1H	
NUCLEUS		

F2 - Processing parameters	
SI	16384
SF	300:130077 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

Ernst-Moritz-Arndt-Universitaet Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV275
 Loesungsmittel: Deuteriochloroform, CDCl3
 Referenz: Loesungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 oC)

Current Data Parameters
 NAME VA_HV275
 EXPNO 13
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20040902
 Time 9.29
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 16384
 SF 75.4677512 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

C₁₈H₂₀O₃

Exact Mass: 284.14

Mol. Wt.: 284.35

C, 76.03; H, 7.09; O, 16.88

Methyl 2,3,4-trimethyl-6-(2-methoxyphenyl)benzoate (6b).

Ernst-Moritz-Arnt-Universität Greifswald

ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum (Schnellmessung)

Van: HV312

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV312
EXPNO 1
PROCNO 1
USER speedy

F2 – Acquisition Parameters

Date_ 20040910

Time 13.46

INSTRUM spect

PROBHD 5 mm Multinu

PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 32

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 2860

DW 82.000 usec

DE 102.50 usec

TE 300.0 K

D1 1.00000000 sec

P1 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS 1H

F2 – Processing parameters

SI 16384

SF 300.1300073 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppel)

Var: HV312

Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV312
EXPNO 13
PROCNO 1
USER lang

F2 - Acquisition Parameters

Date_ 20040928
Time 12.05
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 512
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.0002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG walz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters

SI 16384
SF 75.4677512 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 0.90

C₁₉H₂₂O₃

Exact Mass: 298.16

Mol. Wt.: 298.38

C, 76.48; H, 7.43; O, 16.09

Methyl 2,4-dimethyl-3-ethyl-6-(2-methoxyphenyl)benzoate (6c).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelt)

Van: HV319

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messzeit: 13 Stunden 30 Minuten

Current Data Parameters
NAME VA_HV319
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20040927
Time 16.38
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 16896
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384
SF 75.4677512 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 0.90

Ethyl 4-methyl-6-(2-methoxyphenyl)-2-phenylbenzoate (6d).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 1H-NMR-Spektrum
 Van: HV370
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV370
 EXPNO 1
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20041217
 Time 9.32
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 0
 SWH 6097.561 Hz
 FIDRES 0.093041 Hz
 AQ 5.3740020 sec
 RG 2860
 DW 82.000 usec
 DE 102.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 3.00 usec
 SFO1 300.1322456 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 300.1300062 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitenbandenkoppelt)

Van: HV370

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV370
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20041217
Time 10.09
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 2764
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384
SF 75.4677499 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00

Methyl 2,4-dimethyl-5-methoxy-6-(2-methoxyphenyl)benzoate (6e).

Universitaet Greifswald
ARX300-Kernresonanz-Spektrometer
1H-NMR-Spektrum (mit Automatik gemessen)

Loesungsmittel: CDCl3

Referenz TMS (oder LM, wenn TMS nicht erkennbar)

Messtemperatur: 298 K (25 oC)

Auftraggeber und Probenname:

siehe nebenstehendes Parameterpanel

(z.B.: NAME XY_kp89 bedeutet

Auftraggeber: XY

Probenname: kp89)

7.345
7.339
7.320
7.317
7.313
7.293
7.287
7.258
7.160
7.158
7.154
7.136
7.134
7.129
7.039
7.037
7.035
6.985
6.981
6.957
6.936
6.931

3.914
3.805
3.764
3.443
3.361

0.000

1.576

2.310
2.305

F2 - Acquisition Parameters
Date_ 20040924
Time 10.37
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8474.576 Hz
FIDRES 0.129312 Hz
AQ 3.8666739 sec
RG 1024
DW 59.000 usec
DE 84.29 usec
TE 300.0 K
D1 0.10000000 sec
P1 4.00 usec
SFO1 300.1316690 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 300.1300068 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV330
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)
 Messzeit: 13 Stunden 5 Minuten

Current Data Parameters
 NAME VA_HV330
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20041021
 Time 16.07
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 16896
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C

F2 – Processing parameters
 SI 16384
 SF 75.4677512 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 1.00

Ethyl 5,6,7,8-tetrahydro-2-(2-methoxyphenyl)naphthalene-1-carboxylate (6f).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Problemname:

Current Data Parameters
 NAME VA_HV394
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters

Date_ 20041119
 Time 9.43
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 24
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 2048
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1300072 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV394
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV394
 EXPNO 13
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20050103
 Time 14.23
 INSTRUM spect
 PROBD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 896
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 2.50 usec
 SFO1 75.4761177 MH
 NUCLEUS 13C

F2 - Processing parameters
 SI 16384
 SF 75.4677512 MHz
 WDW EM
 SSB 0
 LB 0.70 Hz
 GB 0
 PC 0.80

Methyl 9,10-dihydro-3-(2-methoxyphenyl)-1-methylphenanthrene-4-carboxylate (6h).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonen Breitbandkoppelt)
 Van: HV448
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Referenz: Lösungsmittel =
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV448
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20050209
 Time 14.38
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 2216
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 PL 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS ¹³C

F2 – Processing parameters
 SI 16384
 SF 75.4677493 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.95

Methyl 2,4-dimethyl-6-(2,5-dimethoxyphenyl)-3-ethyl benzoate (6i).

Universitaet Greifswald
 APX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)

Messtemperatur: 298 K (25 °C)

Auftraggeber und Probenname:

siehe nebenstehendes Parameterpanel

(z.B.: NAME XY_kp89 bedeutet

Auftraggeber: XY

Probenname: kp89)

7.257
6.998
6.826
6.818
6.769
6.766
6.761
6.758

3.782
3.758
3.745
3.729
3.687
3.545

1.562
1.258
1.171
1.146
1.120

0.000

Current Data Parameters
 NAME VA_HV399
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters

Date_ 20041124

Time 8.17

INSTRUM spect

PROBHD 5 mm Multinu

PULPROG zg

TD 65536

SOLVENT CDCl3

NS 24

DS 0

SWH 8474.576 Hz

FIDRES 0.129312 Hz

AQ 3.8666739 sec

RG 1024

DW 59.000 usec

DE 84.29 usec

TE 300.0 K

D1 0.10000000 sec

P1 4.00 usec

SFO1 300.1316690 MH

NUCLEUS 1H

F2 - Processing parameters

SI 32768

SF 300.1300073 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)

Van: HV399

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV399
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20050104
 Time 14.40
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 1544
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 16384
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 0.70 Hz
 GB 0
 PC 0.90

Ethyl 2,4-dimethyl-6-(2,4-dimethoxyphenyl)-3-ethylbenzoate (6j).

Current Data Parameters
 NAME VA_HV384
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041110
 Time 11.19
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 24
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQC 3.8666739 sec
 RG 512
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300084 MHz
 EM
 WDW 0
 SSB 0.10 Hz
 LB 0
 GB 0
 PC 1.00

Ethyl 2,4-dimethyl-6-(1,3-dimethoxyphenyl)benzoate (6k).

Methyl 3-(2-chloroethyl)-6-(*o*-methoxyphenyl)-2,4-dimethylbenzoate (6l).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 1H-NMR-Spektrum (mit Automatik gemessen)
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Probenname:

Current Data Parameters
 NAME VA_HV342
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041005
 Time 8.17
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 48
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 2860
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300065 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)

Van: HV342

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

```
Current Data Parameters
NAME      VA_HV342
EXPNO     13
PROCNO    1
USER      lang
```

F2 – Acquisition Parameters

Date_ 20041112
Time 15.16

Time 15.10 spect
INSTRUM 5 mm Multinu
PROBHD

PROBHD	5 mm Multihd
PULPROG	zgpg
TD	32768

TD	32768
SOLVENT	CDC13
NS	66560

NS	66560
DS	0
DS	21220 121 11-

SWH	21739.131 Hz
FIDRES	0.663426 Hz

AQ	0.7537140 sec
RG	32768

KU	32.76
DW	23.000 usec
DE	32.86 usec

DE	52.66 usec
TE	298.0 K
D12	0.00002000 sec

DL2	0.00000000 sec
DL6	30.00 dB
DL1	2.00000000 sec

D1	2.0000000 sec
CPDPRG	waltz16
D1	110.00 msec

P31	110.00 usec
D11	0.0300000 sec
D15	0.0000000 sec

Methyl 3-(2-bromoethyl)-6-(*o*-methoxyphenyl)-2,4-dimethylbenzoate (6m).

Current Data Parameters
 NAME VA_HV343
 EXPNO 1
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041005
 Time 8.28
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8474.576 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 715
 DW 59.000 usec
 DE 84.29 usec
 TE 300.0 K
 D1 0.10000000 sec
 P1 4.00 usec
 SFO1 300.1316690 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 32768
 SF 300.1300097 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV343
 Lösungsmittel: Deuteriochloroform, CDCl3
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV343
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters

Date_ 20041021
 Time 14:01
 INSTRUM spect
 PROBD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 384
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters

SI 16384
 SF 75.4677539 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 1.00

7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7a).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum (Schnellmessung)

Von: HV279

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV279
EXPNO 1
PROCNO 1
USER speedy

F2 - Acquisition Parameters
Date_ 20040818
Time 10.16
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 16
DS 0

SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 8192
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 3.00 usec
SFO1 300.1322456 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonen Breitbandentkoppelt)

Van: HV279

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298K (25 °C)

Messzeit: 1 Stunde 25 Minuten

Current Data Parameters
NAME VA_HV279
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20040830
Time 13.56

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768

SOLVENT CDCl₃
NS 1792

DS 0
SWH 21739.131 Hz

FIDRES 0.663426 Hz
AQ 0.7537140 sec

RG 32768
DW 23.000 usec

DE 32.86 usec
TE 298.0 K

D12 0.00002000 sec
DL6 30.00 dB

D1 2.00000000 sec
CPDPRG waltz16

P31 110.00 usec
D11 0.03000000 sec

DL5 22.00 dB
P1 3.00 usec

SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384

SF 75.4677499 MHz
WDW EM

SSB 0
LB 1.20 Hz

GB 0
PC 0.80

7,8,9-Trimethyl-6*H*-benzo[*c*]chromen-6-one (7b).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonen Breitbandkoppelt)
 Van: HV322
 Lösungsmittel: Deuteriochloroform, CDCl3
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV322
 EXPNO 13
 PROCNO 1
 USER lang

F2 - Acquisition Parameters
 Date_ 20041011
 Time 13.34
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 640
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.0002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG walz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 16384
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

8-Ethyl-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7c).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum (Schnellmessung)

Van: HV326

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV326
EXPNO 1
PROCNO 1
USER speedy

F2 - Acquisition Parameters

Date_ 20040920

Time 10.50

INSTRUM spect

PROBHD 5 mm Multinu

PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 16

DS 0

SWH 6097.561 Hz

FIDRES 0.093041 Hz

AQ 5.3740020 sec

RG 4096

DW 82.000 usec

DE 102.50 usec

TE 300.0 K

D1 1.00000000 sec

P1 3.00 usec

SFO1 300.1322456 MHz

NUCLEUS 1H

F2 - Processing parameters

SI 16384

SF 300.1300062 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelt)

Van: HV326

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: Loesungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV326
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20041108
Time 16:29
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 384
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 16384
SF 75.4677512 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

9-Methyl-7-phenyl-6*H*-benzo[c]chromen-6-one (7d).

Ernst-Moritz-Armdt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV375
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV375
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20041125
 Time 13.24
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 384
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 2.50 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 32768
 SF 75.4677539 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 1.00

10-Hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7e).

Current Data Parameters
NAME VA_HV341
EXPNO 1
PROCNO 1
USER automatic

F2 - Acquisition Parameters
Date_ 20040930
Time 9.21
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8474.576 Hz
FIDRES 0.129312 Hz
AQ 3.8666739 sec
RG 4096
DW 59.000 usec
DE 84.29 usec
TE 300.0 K
D1 0.10000000 sec
P1 4.00 usec
SFO1 300.1316690 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)

Van: HV341

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

```
Current Data Parameters
NAME      VA_HV341
EXPNO     13
PROCNO    1
USER      lang
```

F2 - Acquisition Parameters	
Date_	20041122
Time	17:13
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg
TD	32768
SOLVENT	CDCl3
NS	16384
DS	0
SWH	21739.131 Hz
FIDRES	0.663426 Hz
AQ	0.7537140 sec
RG	32768
DW	23.000 usec
DE	32.86 usec
TE	298.0 K
D12	0.00002000 sec
DL6	30.00 dB
D1	2.0000000 sec
CPDPRG	waltz16
P31	110.00 usec
D11	0.03000000 sec
DL5	22.00 dB
P1	2.50 usec
NUC1	75.4761177 MHz
NUCLEUS	¹³ C

F2 - Processing parameters	
SI	32768
SF	75.4677486 MHz
WDW	EM
SSB	0
LB	1.20 Hz
GB	0
PC	1.00

1,2,3,4-Tetrahydro-12-methylnaphtho[1,2-*c*]chromen-5-one (7g).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandentkoppelt)

Van: HV403

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: Loesungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV403
EXPNO 13
PROCNO 1
USER lang

F2 – Acquisition Parameters
Date_ 20050609
Time 14.35
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 1024
DS 0
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 3.00 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 32768
SF 75.4677505 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 0.95

1,2-Dihydro-14-methylphenanthro[4,3-c]chromen- 7-one (7h).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum

Van: HV415

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV415
EXPNO 1
PROCNO 1
USER lang

F2 - Acquisition Parameters
Date_ 20050201
Time 17.06
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 128
DS 0
SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 2860
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 300.1322456 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonen Breitbandentkoppelt)

Van: HV415

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: Lösungsmittel = 77.0 ppm

Messtemperatur: 298 K (25 °C)

Messzeit: 12 Stunden 40 Minuten

Current Data Parameters
NAME VA_HV415
EXPNO 13
PROCNO 1
USER lang

F2 - Acquisition Parameters
Date_ 20050201
Time 17.48

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 16384
DS 0

SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K

D12 0.0002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG walz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 3.00 usec

SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 16384

SF 75.4677480 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.90

8-Ethyl-2-hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7i).

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
1H-NMR-Spektrum (Schnellmessung)

Van: HV400

Lösungsmittel: deuteriertes Aceton, (CD₃)₂CO
Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV400
EXPNO 2
PROCNO 1
USER speedy

F2 – Acquisition Parameters
Date_ 20041125
Time 13.50
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 65536
SOLVENT CDCl₃
NS 16
DS 0

SWH 6097.561 Hz
FIDRES 0.093041 Hz
AQ 5.3740020 sec
RG 4096
DW 82.000 usec
DE 102.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 300.1332000 MHz
NUCLEUS 1H

F2 – Processing parameters
SI 16384
SF 300.1315640 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Nguyen HV 400

8-Ethyl-3-hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7j).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV409
 Lösungsmittel: deuteriertes Aceton, (CD₃)₂CO
 Referenz: Lösungsmittel = 30.5 ppm
 Messtemperatur: 298K (25 oC)

Current Data Parameters
 NAME VA_HV409
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20050606
 Time 15:08
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDC13
 NS 1408
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 32768
 SF 75.4680243 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.95

1-Hydroxy-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7k).

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 - Kernresonanz-Spektrometer
 1H-NMR-Spektrum (Schnellmessung)
 Van: HV401
 Lösungsmittel: deuteriertes Aceton, (CD₃)₂CO
 Referenz: TMS = 0.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV401
 EXPNO 2
 PROCNO 1
 USER speedy

F2 - Acquisition Parameters
 Date_ 20041125
 Time 13.45
 INSTRUM spect
 PROBD 5 mm Multinu
 PULPROG zg
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 0
 SWH 6097.561 Hz
 FIDRES 0.093041 Hz
 AQ 5.3740020 sec
 RG 4096
 DW 82.000 usec
 DE 102.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 3.00 usec
 SFO1 300.1352000 MH
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 300.1315640 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

Nguyen HV 401

Current Data Parameters
NAME 050119
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050120
Time 1.35
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3300
DS 4
SWH 19230.861 Hz
FIDRES 0.293440 Hz
AQ 1.7039860 sec
RG 32768
DM 26.000 usec
DE 37.14 usec
TE 300.0 K
D12 0.00002000 sec
DL6 21.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 100.00 usec
D11 0.03000000 sec
DL5 15.00 dB
P1 7.50 usec
SF01 75.4774203 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 75.4689594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F4P 215.000 ppm
F1 16225.83 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/
HZCM 830.15851 Hz/c

8-(2-Chloroethyl)-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (71).

Ernst-Moritz-Arndt-Universität Greifswald

ARX300 – Kernresonanz-Spektrometer

¹H-NMR – Spektrum (Schnellmessung)

Van: HV346a

Loesungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298 K (25 °C)

Current Data Parameters
NAME VA_HV346a
EXPNO 1
PROCNO 1
USER automatic

F2 – Acquisition Parameters
Date_ 20041007
Time 13.35

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg

TD 65536

SOLVENT CDCl₃

NS 32

DS 0

SWH 8474.576 Hz

FIDRES 0.129312 Hz

AQ 3.8666739 sec

RG 4096

DW 59.000 usec

DE 84.29 usec

TE 300.0 K

D1 0.10000000 sec

P1 4.00 usec

SFO1 300.1316690 MHz

NUCLEUS ¹H

F2 – Processing parameters

SI 32768

SF 300.1300059 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

Ernst-Moritz-Arndt-Universität Greifswald
 ARX300 – Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 Van: HV346
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME VA_HV346
 EXPNO 13
 PROCNO 1
 USER lang

F2 – Acquisition Parameters
 Date_ 20041026
 Time 16.14
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl₃
 NS 768
 DS 0
 SWH 21739.131 Hz
 FIDRES 0.663426 Hz
 AQ 0.7537140 sec
 RG 32768
 DW 23.000 usec
 DE 32.86 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 2.00000000 sec
 CPDPRG walz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 – Processing parameters
 SI 32768
 SF 75.4677499 MHz
 WDW EM
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.80

8-(2-Bromoethyl)-7,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (7m).

Universitaet Greifswald
 ARX300-Kernresonanz-Spektrometer
 13C-NMR-Spektrum, Protonenbreitbandenkoppelt
 Loesungsmittel: CDCl3
 Referenz: TMS (oder LM, wenn TMS nicht erkennbar)
 Messtemperatur: 298 K (25 oC)
 Auftraggeber und Probenname:
 siehe nebenstehendes Parameterpanel
 (z.B.: NAME XY_kp89 bedeutet
 Auftraggeber: XY
 Probenname: kp89)

Current Data Parameters
 NAME VA_HV347
 EXPNO 13
 PROCNO 1
 USER automatic

F2 - Acquisition Parameters
 Date_ 20041122
 Time 13:08
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 131072
 SOLVENT CDCl3
 NS 384
 DS 0
 SWH 22727.273 Hz
 FIDRES 0.173395 Hz
 AQ 2.8836341 sec
 RG 32768
 DW 22.000 usec
 DE 31.43 usec
 TE 298.0 K
 D12 0.00002000 sec
 DL6 30.00 dB
 D1 1.00000000 sec
 CPDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 20.00 dB
 P1 3.00 usec
 SFO1 75.4761177 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 32768
 SF 75.4677523 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

7-Hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13a).

HUSSAIN 48RC1, 1H, CDCL3

NO100S.103
 AU PROG:
 X00.AU
 DATE 10-11-6
 SF 250.13
 SY 250.0
 O1 4311.79
 SI 32768
 TD 32768
 SW 5000.00
 HZ/PT .30
 PW 0.0
 RD 0.0
 AQ 3.27
 RG 64
 NS 48
 TE 297
 FW 6300
 O2 2714.49
 DP 63L P0
 LB 0.0
 GB .10
 CX 23.00
 CY 12.00
 F1 12.00
 F2 .49
 HZ/CM 135.93
 PPM/CM .54
 SR 2871.97

Hussain 4BRC1

7-Hydroxy-8,9-dimethyl-6*H*-benzo[*c*]chromen-6-one (13b).

HUSSAIN 73C1, 1H, CDCL3

DZ040S.106
AU PROG:
X00.AU
DATE 4-12-6
SF 250.13
SY 250.0
O1 4311.79
SI 32768
TD 32768
SW 5000.00
HZ/PT .30
PW 0.0
RD 0.0
AQ 3.27
RG 200
NS 48
TE 297
FW 6300
O2 2714.49
DP 63L P0
LB 0.0
GB .10
CX 23.00
CY 12.00
F1 12.00
F2 -.49
HZ/CM 135.93
PPM/CM .54
SR 2871.36

8-Ethyl-7-hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13c).

HUSSAIN 54, 1H, CDCL3

N0130S.105
AU PROG:
X00.AU
DATE 13-11-6
SF 250.133
SY 250.0
01 4311.794
SI 32768
TD 32768
SW 5000.000
HZ/PT 300
PW 0.0
RD 0.0
AQ 3.27
RG 20
NS 48
TE 297
FW 6300
02 2714.495
DP 63L P0
LB 0.0
GB 100
CX 23.00
CY 12.00
F1 12.000
F2 -495
HZ/CM 135.936
PPM/CM .543
SR 2872.89

8-Hexyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13d).

HUSSAIN 77C1, H, CDCL3

DZ060S.102
 AU PROG:
 X00.AU
 DATE 6-12-6
 SF 250.133
 SY 250.0
 O1 4311.794
 SI 32768
 TD 32768
 SW 5000.000
 HZ/PT .305
 PW 0.0
 RD 0.0
 AQ 3.277
 RG 64
 NS 48
 TE 297
 FW 6300
 O2 2714.499
 DP 63L P0
 LB 0.0
 GB .100
 CX 23.00
 CY 12.00
 F1 12.0000
 F2 -.499
 HZ/CM 135.936
 PPM/CM .543
 SR 2871.67

Hussain 77C1

8-Heptyl-7-hydroxy-9-methyl-6*H*-benzo[*c*]chromen-6-one (13e).

YAER 82, 1H, CDCL3

DZ1105.106

AU PROG:

X00.AU

DATE 11-12-6

SF 250.133

SY 250.0

O1 4311.794

SI 32768

TD 32768

SW 5000.000

HZ/PT .305

PW 0.0

RD 0.0

AQ 3.277

RG 10

NS 48

TE 297

FW 6300

O2 2714.499

DP 63L P0

LB 0.0

GB .100

CX 23.00

CY 12.00

F1 12.0000f

F2 - .499f

HZ/CM 135.936

PPM/CM .543

SR 2872.59

7-hydroxy-9-methyl-8-octyl-6*H*-benzo[*c*]chromen-6-one (13f).

61

HUSSAIN ??, 1H, CDCL3

NO210S.101
AU PROG:
X00.AU
DATE 21-11-6

SF 250.133
SY 250.0
O1 4311.794
SI 32768
TD 32768
SW 5000.000
HZ/PT .305

PW 0.0
RD 0.0
AQ 3.277
RG 20
NS 48
TE 297

FW 6300
O2 2714.499
DP 63L P0

LB 0.0
GB .100
CX 23.00
CY 12.00
F1 12.000f
F2 - .499f
HZ/CM 135.936
PPM/CM .543
SR 2871.67

7-Hydroxy-9-methyl-8-undecyl-6*H*-benzo[*c*]chromen-6-one (13g).

7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13h).

Hussain AH 117 1H CDC13

Current Data Parameters
 NAME 070212
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070212
 Time 9.26
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 5700
 DW 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1333884 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08003 Hz/cm

Hussain AH 117

7-hydroxy-8-methyl-9-propyl-6*H*-benzo[*c*]chromen-6-one (13i).

Hussain 104C1 1H CDCL3

Current Data Parameters
 NAME 070126
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070126
 Time 9.08
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCL3
 NS 16
 DS 2
 SWH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 5700
 DW 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1333888 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08003 Hz/cm

Ibrar Hussain, 104Cl, 13C in CDCl3

Current Data Parameters
NAME 070131
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070131
Time 17.55
INSTRUM spect
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6144
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 57
DE 16.650 use
TE 6.00 use
D1 300.0 K
d11 2.00000000 sec
d12 0.03000000 sec
0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 use
PL1 4.80 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -3.00 dB
PL12 17.50 dB
PL13 20.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

8-ethyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13j).

Hussain 105C1 1H CDCl3

Current Data Parameters
 NAME 070126
 EXPNO 160
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070126
 Time 10.58
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 1430
 DW 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1333896 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08003 Hz/cm

Hussain 105c1

Current Data Parameters
NAME 070131
EXPNO 350
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070131
Time 21.19
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 4
SWH 19230.770 Hz
FIDRES 0.293436 Hz
AQ 1.7039860 sec
RG 32768
DM 26.000 usec
DE 37.14 usec
TE 300.0 K
D12 0.0002000 sec
DL6 21.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 100.00 usec
D11 0.03000000 sec
DL5 15.00 dB
P1 7.50 usec
SF01 75.4770618 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 75.4686695 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16225.76 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/
HZCM 830.15533 Hz/c

8-hexyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13k).

Yawer Y 74C1 1H, CDCl3

Current Data Parameters
NAME 070215
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070215
Time 10:59
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 6250.000 Hz
FIDRES 0.095367 Hz
AQ 5.2429299 sec
RG 1430
DM 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 8.00 usec
SF01 300.1351790 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 32768
SF 300.1333691 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.000 ppm
F1 3301.47 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.08003 Hz/cm

Yawer Y 74C1

8-heptyl-7-hydroxy-9-propyl-6*H*-benzo[*c*]chromen-6-one (13l).

Tuan DTT175, 4H C0C13

Current Data Parameters
 NAME 070207
 EXPNO 130
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070207
 Time 12.38
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT C0C13
 NS 16
 DS 2
 SWH 6250.000 Hz
 FIDRES 0.095367 Hz
 AQ 5.2429299 sec
 RG 90
 DM 80.000 usec
 DE 114.29 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.00 usec
 SF01 300.1351790 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 32768
 SF 300.1334232 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 3301.47 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 180.08005 Hz/cm

Tuan DTT175

4-hydroxy-4-(2-methoxyphenyl)-3-buten-2-one. (10a).

1-hydroxy-1-(2-methoxyphenyl)-1-hexen-3-one. (10b).

Yawer 38C1 1H CDC13

Current Data Parameters
NAME 070108
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070108
Time 11.05
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 32
DS 2
SWH 6250.000 Hz
FIDRES 0.095367 Hz
AQ 5.2429299 sec
RG 90
DM 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.0000000 sec
P1 8.00 usec
SF01 300.1351790 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 32768
SF 300.1333950 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.000 ppm
F1 3301.47 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCH 0.60000 ppm/cm
HZCM 180.08003 Hz/cm

4-(2-methoxyphenyl)-4-[(trimethylsilyl)oxy]-3-buten-2-one.(11a).

HUSSAIN 39R, 1H, CDCL3

N0030S.118
AU PROG:
X00.AU
DATE 3-11-6
SF 250.13
SY 250.0
01 4311.79
SI 32768
TD 32768
SW 5000.00
HZ/PT 30
PW 0.0
RD 0.0
AQ 3.27
RG 2
NS 48
TE 297
FW 6300
O2 2714.49
DP 63L P0
LB 0.0
GB 10
CX 23.00
CY 12.00
F1 12.00
F2 -49
HZ/CM 135.93
PPM/CM 54
SR 2871.36

Hussain 39H

1-(2-methoxyphenyl)-1-[(trimethylsilyl)oxy]-1-hexen-3-one. (11b).

Yawer Y 43M 1H CDC13

Current Data Parameters
NAME 070118
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070118
Time 9.53
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.095367 Hz
AQ 5.2429299 sec
RG 128
DW 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 8.00 usec
SF01 300.1351790 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 32768
SF 300.1333967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 11.000 ppm
F1 3301.47 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/c
HZCM 180.08003 Hz/cm

Yawer Y43M

Ortep plot

Figure 1. ORTEP-plot of 13c with 50% probability of the thermal ellipsoids