

# Regioselective synthesis of 4-acyl-1-hydroxy-2,3-benzodioxates by chelation-controlled [3+3] annulation of 3-acyl-4-ethoxy-2-oxo-3-enoates with 1,3-bis(trimethylsilyloxy)-1,3-butadienes

Abdolmajid Riahi,<sup>a, b</sup> Mohanad Shkoor,<sup>a</sup> Olumide Fatunsin,<sup>a</sup> Rasheed Ahmad Khera,<sup>a</sup> Christine Fischer,<sup>a</sup> and  
5 Peter Langer\*<sup>a, b</sup>

## Supporting Information

10

### Experimental Section

**General Comments.** All solvents were dried by standard methods and all reactions were carried out under an inert atmosphere. For <sup>1</sup>H and <sup>13</sup>C NMR spectra the deuterated  
15 solvents indicated were used. Mass spectrometric data (MS) were obtained by electron ionization (EI, 70 eV), chemical ionization (CI, isobutane) or electrospray ionization (ESI). For preparative scale chromatography silica gel 60 (0.063-0.200 mm, 70 – 230 mesh) was used.

20 **General procedure for the synthesis of 2a-f:** To a suspension of sodium ethoxide (1.0 equiv.) in benzene (0.5 mL / 1.0 mmol EtONa), was dropwise added diethyl oxalate (1.0 equiv.) at 0 °C followed by dropwise addition (during 30 min) of **1a-f** (1.0 equiv.). The temperature of the solution was allowed to warm to 20 °C during 14 h with stirring. To the solution was added hydrochloric acid (10%, 20 mL) and the organic and the aqueous  
25 layer were separated. The latter was extracted with ether (3×20 mL) and washed with brine. The combined organic layers were dried (Na<sub>2</sub>SO<sub>4</sub>), filtered and the filtrate was concentrated in vacuo to give products **2**. The synthesis of **2a-f** has been previously reported.

30 **General procedure for the synthesis of 3a-f:** To a solution of **2a-f** (1.0 equiv.) in acetic anhydride (2.0 equiv.) was added triethylorthoformiate (1.2 equiv.). The mixture was heated under reflux for 2 h at 120 °C and for further 4 h at 140 °C. The mixture was concentrated in vacuo to give **3** (92-99%).

35 **Ethyl 3-(ethoxymethylene)-2,4-dioxopentanoate (3a).** Starting with **2a** (4.30 g, 27.2 mmol), triethyl orthoformiate (5.16 g, 32.6 mmol), and acetic anhydride (8.60 g, 54.4 mmol), **3a** was isolated as a red oil (5.64 g, 97%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.28 (t, <sup>3</sup>J = 7.2 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.40 (t, <sup>3</sup>J = 7.1 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.36 (s, 3 H, CH<sub>3</sub>), 4.24 (q, <sup>3</sup>J = 7.2 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.35 (q, <sup>3</sup>J = 7.1 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.85 (s, 1 H, CH<sub>Oif</sub>).  
40 <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.7, 15.0, 30.4 (CH<sub>3</sub>), 61.8, 74.4 (OCH<sub>2</sub>), 117.4 (COCCO), 164.5 (CO), 169.4 (CH<sub>Oif</sub>), 186.8, 195.8 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2984 (w), 2940 (w),

2255 (w), 1780 (w), 1732 (m), 1661 (m), 1577 (m), 1473 (w), 1389 (w), 1367 (w), 1312 (m), 1255 (m), 1224 (m), 1172 (m), 1097 (m), 1022 (m), 907 (s), 862 (w), 725 (s), 684 (w), 648 (m), 601 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 214 ( $[M]^+$ , 1.4), 141 (100), 113 (55), 99 (23), 71 (82), 43 (48), 29 (20). HRMS (EI): Calcd. for  $C_{10}H_{14}O_5$  ( $[M]^+$ ): 214.08358; found: 214.083886.

**Ethyl 3-benzoyl-4-ethoxy-2-oxobut-3-enoate (3b).** Starting with **2b** (4.20 g, 19.1 mmol), triethyl orthoformate (5.04 g, 22.9 mmol), and acetic anhydride (8.40 g, 38.2 mmol), **3b** was isolated as a red oil (5.00 g, 96%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.15-1.33 (m, 6 H,  $2 \times OCH_2CH_3$ ), 4.10-4.29 (m, 4 H,  $2 \times OCH_2CH_3$ ), 7.35-7.53 (m, 3 H,  $3 \times CH_{Ph}$ ), 7.76-7.87 (m, 2 H,  $2 \times CH_{Ph}$ ), 7.93 (s, 1 H,  $CH_{Olf}$ ).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.8, 15.2 ( $CH_3$ ), 62.0, 73.4 ( $OCH_2$ ), 117.9 ( $COCCO$ ), 128.5 ( $2 \times CH_{Ar}$ ), 129.2 ( $2 \times CH_{Ar}$ ), 133.2 ( $CH_{Ar}$ ), 137.9 ( $C_{Ar}$ ), 162.8 ( $CO$ ), 166.4 ( $CH_{Olf}$ ), 183.4, 192.2 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu$  = 3062 (w), 2983 (w), 2928 (w), 1740 (m), 1681 (m), 1598 (m), 1489 (w), 1447 (m), 1387 (w), 1360 (m), 1264 (s), 1183 (s), 1157 (m), 1061 (m), 1010 (m), 954 (w), 893 (w), 760 (m), 690 (s), 631 (w), 587 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 276 ( $[M]^+$ , 2), 175 (11), 106 (10), 105 (100), 77 (39). HRMS (EI): Calcd. for  $C_{15}H_{16}O_5$  ( $[M]^+$ ): 276.09923; found: 276.099378.

**Ethyl 4-ethoxy-3-(4-methylbenzoyl)-2-oxobut-3-enoate (3c).** Starting with **2c** (4.50 g, 19.2 mmol), triethyl orthoformate (3.40 g, 23.0 mmol), and acetic anhydride (5.68 g, 38.4 mmol), **3c** was isolated as a red oil (5.46 g, 98%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.16-1.23 (m, 6 H,  $2 \times OCH_2CH_3$ ), 2.33 (s, 3 H,  $CH_3$ ), 4.10-4.17 (m, 4 H,  $2 \times OCH_2CH_3$ ), 7.17-7.19 (m, 2 H,  $2 \times CH_{Ar}$ ), 7.67-7.70 (m, 2 H,  $2 \times CH_{Ar}$ ), 7.89 (s, 1 H,  $CH_{Olf}$ ).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.8, 15.2, 21.7 ( $CH_3$ ), 61.9, 73.3 ( $OCH_2$ ), 118.1 ( $COCCO$ ), 129.2 ( $2 \times CH_{Ar}$ ), 129.4 ( $2 \times CH_{Ar}$ ), 135.3, 144.2 ( $C_{Ar}$ ), 162.8 ( $CO$ ), 166.6 ( $CH_{Olf}$ ), 183.3, 191.8 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu$  = 2981 (w), 2923 (w), 2871 (w), 1733 (m), 1677 (m), 1604 (s), 1579 (m), 1502 (m), 1447 (m), 1384 (m), 1265 (s), 1237 (s), 1180 (s), 1112 (m), 1059 (s), 1015 (s), 896 (m), 831 (m), 752 (m), 710 (m), 674 (m), 623 (m), 590 (m), 567 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 290 ( $[M]^+$ , 3), 175 (11), 218 (10), 217 (67), 120 (17), 119 (100), 91 (42), 65 (13). HRMS (EI): Calcd. for  $C_{16}H_{18}O_5$  ( $[M]^+$ ): 290.115247; found: 290.11488.

**Ethyl 4-ethoxy-3-(4-methoxybenzoyl)-2-oxobut-3-enoate (3d).** Starting with **2d** (3.80 g, 15.2 mmol), triethyl orthoformate (2.69 g, 18.2 mmol), and acetic anhydride (3.09 g, 30.4 mmol), **3d** was isolated as a red oil (4.41 g, 95%).  $^1H$  NMR (250 MHz,  $CDCl_3$ ):  $\delta$  = 1.19 (t,  $^3J$  = 7.1 Hz, 3 H,  $OCH_2CH_3$ ), 1.20 (t,  $^3J$  = 7.1 Hz, 3 H,  $OCH_2CH_3$ ), 3.79 (s, 3 H,  $OCH_3$ ), 4.12 (q,  $^3J$  = 7.2 Hz, 2 H,  $OCH_2CH_3$ ), 4.19 (q,  $^3J$  = 7.1 Hz, 2 H,  $OCH_2CH_3$ ), 6.83-6.87 (m, 2 H,  $CH_{PhOMe}$ ), 7.76-7.79 (m, 2 H,  $CH_{PhOMe}$ ), 7.89 (s, 1 H,  $CH_{Olf}$ ).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 12.9, 14.3 ( $CH_3$ ), 54.5 ( $OCH_3$ ), 61.3, 72.3 ( $OCH_2$ ), 112.7 ( $2 \times CH_{PhOMe}$ ), 117.2 ( $COCCO$ ), 129.6 ( $C_{Ar}$ ), 130.7 ( $2 \times CH_{PhOMe}$ ), 162.9 ( $CO$ ), 164.8 ( $CH_{Olf}$ ), 182.5 ( $C_{Ar}$ ), 189.7, 195.9 ( $CO$ ). IR (KBr,  $cm^{-1}$ ):  $\nu$  = 2983 (w), 2939 (w), 2905 (w), 2842 (w), 2254 (w), 1733 (m), 1674 (m), 1646 (m), 1599 (s), 1576 (m), 1510 (m), 1464 (w), 1444 (w), 1422 (w), 1385 (w), 1362 (w), 1309 (m), 1257 (s), 1170 (m), 1113 (w), 1029 (m), 911 (w), 846 (w), 806 (w), 777 (w), 731 (m), 648 (w), 609 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 306 ( $[M]^+$ , 2), 278 (3), 270 (1), 249 (2), 233 (26), 210 (3), 189 (2), 135 (100), 107 (8), 92 (8), 77 (14), 69 (3), 44 (2). HRMS (EI): Calcd. for  $C_{16}H_{18}O_6$  ( $[M]^+$ ): 306.10979; found: 306.110424

**Ethyl 3-(4-bromobenzoyl)-4-ethoxy-2-oxobut-3-enoate (3e).** Starting with **2e** (4.35 g, 14.60 mmol), triethyl orthoformate (2.60 g, 17.6 mmol), and acetic anhydride (3.73 g, 36.6 mmol), **3e** was isolated as a red oil (4.72 g, 92%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.29 (t, <sup>3</sup>J = 7.0 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.39 (t, <sup>3</sup>J = 6.9 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.21 (q, <sup>3</sup>J = 7.2 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.35 (q, <sup>3</sup>J = 7.0 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.72-7.74 (m, 2 H, CH<sub>PhBr</sub>), 7.82-7.85 (m, 2 H, CH<sub>PhBr</sub>), 8.01 (s, 1 H, CH<sub>Olf</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.8, 15.1 (CH<sub>3</sub>), 62.4, 73.6 (OCH<sub>2</sub>), 117.4 (COCCO), 128.3 (C<sub>Ar</sub>), 129.8 (2×CH<sub>PhBr</sub>), 131.8 (2×CH<sub>PhBr</sub>), 136.0 (C<sub>Ar</sub>), 162.7 (CO), 166.4 (CH<sub>Olf</sub>), 183.1, 191.2 (CO). IR (KBr, cm<sup>-1</sup>): ν = 3064 (w), 2982 (w), 2938 (w), 2873 (w), 1824 (w), 1732 (m), 1683 (m), 1651 (m), 1584 (s), 1481 (w), 1444 (w), 1395 (m), 1367 (m), 1302 (w), 1185 (m), 1068 (m), 1007 (s), 957 (m), 920 (m), 907 (m), 896 (m), 843 (m), 748 (m), 675 (m), 626 (m), 606 (m), 587 (m). GC-MS (EI, 70 eV): *m/z* (%) = 356 ([M]<sup>+</sup>, Br<sup>81</sup>, 10), 354 ([M]<sup>+</sup>, Br<sup>79</sup>, 16), 283 (60), 281 (62), 185 (98), 183 (100), 157 (18), 155 (19). HRMS (EI): Calcd. for C<sub>15</sub>H<sub>16</sub>O<sub>5</sub><sup>79</sup>Br ([MH]<sup>+</sup>): 355.01756; found: 355.01667.

**Diethyl 2-(ethoxymethylene)-3-oxosuccinate (3f).** Starting with **2f** (7.01 g, 37.2 mmol), triethyl orthoformate (7.53 mL, 45.0 mmol), and acetic anhydride (8.85 mL, 94.2 mmol), **3f** was isolated as a red oil (9.70 g, 99%). <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>): δ = 1.21 (t, <sup>3</sup>J = 7.8 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.30 (t, <sup>3</sup>J = 6.9 Hz, 6 H, 2×OCH<sub>2</sub>CH<sub>3</sub>), 4.16 (q, <sup>3</sup>J = 6.9 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.28 (q, <sup>3</sup>J = 7.1 Hz, 4 H, 2×OCH<sub>2</sub>CH<sub>3</sub>), 7.84 (s, 1 H, CH<sub>Olf</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 12.9, 13.1, 14.3 (CH<sub>3</sub>), 62.2, 62.2, 73.5 (OCH<sub>2</sub>), 108.5 (COCCO), 170.0 (CH<sub>Olf</sub>), 175.6, 182.1, 184.1 (CO). IR (KBr, cm<sup>-1</sup>): ν = 2985 (w), 2941 (w), 2908 (w), 1764 (m), 1737 (m), 1702 (m), 1582 (m), 1468 (w), 1447 (w), 1390 (w), 1370 (w), 1296 (m), 1240 (m), 1184 (s), 1156 (m), 1102 (s), 1009 (s), 866 (m), 852 (m), 784 (m), 755 (m), 657 (w), 605 (w). GC-MS (EI, 70 eV): *m/z* (%) = 244 ([M]<sup>+</sup>, 3), 144 (1), 113 (82), 85 (100), 83 (5), 55 (5), 39 (4), 29 (20).

**General procedure for the synthesis of 5a-al.** To a CH<sub>2</sub>Cl<sub>2</sub> solution (2 mL / 1 mmol of **3a-f**) of **3a-f** was added **4a-i** (1.1 mmol) and, subsequently, TiCl<sub>4</sub> (1.1 mmol) at -78 °C. The temperature of the solution was allowed to warm to 20 °C during 14 h with stirring. To the solution was added hydrochloric acid (10%, 20 mL) and the organic and the aqueous layer were separated. The latter was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 20 mL). The combined organic layers were dried (Na<sub>2</sub>SO<sub>4</sub>), filtered and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, heptanes / EtOAc) to give **5a-al**.

**2-Ethyl 1-methyl 3-acetyl-6-hydroxyphthalate (5a).** Starting with **3a** (0.321 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5a** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.191 g, 48%), mp. 95 - 97 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.17 (t, <sup>3</sup>J = 7.2 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.32 (s, 3 H, CH<sub>3</sub>), 3.72 (s, 3 H, OCH<sub>3</sub>), 4.20 (q, <sup>3</sup>J = 7.7 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 6.87 (d, <sup>3</sup>J = 9.0 Hz, 1 H, CH<sub>Ar</sub>), 7.73 (d, <sup>3</sup>J = 9.0 Hz, 1 H, CH<sub>Ar</sub>), 11.37 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 27.5 (CH<sub>3</sub>), 53.2 (OCH<sub>3</sub>), 61.8 (OCH<sub>2</sub>), 110.5 (CCOCH<sub>3</sub>), 118.5 (CH<sub>Ar</sub>), 126.9 (CCOCH<sub>3</sub>), 136.2 (CH<sub>Ar</sub>), 137.4 (CCOOC<sub>2</sub>H<sub>5</sub>), 164.9 (COH), 168.3, 169.4, 195.7 (CO). IR (neat, cm<sup>-1</sup>): ν = 3119 (w), 3076 (w), 2981 (w), 2919 (w), 2850 (w), 1729 (m), 1674 (s), 1580 (m), 1470 (w), 1443 (m), 1389 (w), 1362 (m), 1328 (m), 1304 (m), 1248 (s), 1207 (s), 1155 (m), 1137 (s), 1100 (m).

1026 (m), 965 (m), 937 (m), 872 (m), 847 (m), 811 (m), 757 (m), 733 (m), 706 (m), 688 (m), 647 (m), 598 (m), 580 (m), 540 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 266 ([M]<sup>+</sup>, 24), 251 (11), 221 (27), 220 (33), 192 (10), 191 (100), 190 (18), 189 (42), 188 (15), 162 (39), 120 (12), 119 (29), 43 (10). HRMS (EI): Calcd. for C<sub>13</sub>H<sub>14</sub>O<sub>6</sub> ([M]<sup>+</sup>): 266.07849; found: 266.079233.

5

**1-Ethyl 2-methyl 6-acetyl-3-hydroxy-4-methylphthalate (5b).** Starting with **3a** (0.321 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5b** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.210 g, 50%), mp. 76 – 78 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.22 (t, <sup>3</sup>J = 7.3 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.25 (s, 3 H, CH<sub>3</sub>), 2.47 (s, 3 H, CH<sub>3</sub>), 3.88 (s, 3 H, OCH<sub>3</sub>), 4.34 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.73 (s, 1 H, CH<sub>Ar</sub>), 11.78 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 15.9, 27.5 (CH<sub>3</sub>), 53.1 (OCH<sub>3</sub>), 61.5 (OCH<sub>2</sub>), 109.7 (CCOOCH<sub>3</sub>), 126.2, 128.1 (C<sub>Ar</sub>), 135.0 (CCOOC<sub>2</sub>H<sub>5</sub>), 136.5 (CH<sub>Ar</sub>), 163.4 (COH), 168.4, 170.0, 195.9 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 3067 (w), 2964 (w), 2929 (w), 2863 (w), 1726 (m), 1674 (s), 1564 (m), 1443 (m), 1407 (m), 1381 (w), 1363 (m), 1321 (m), 1251 (s), 1205 (s), 1172 (m), 1135 (s), 1090 (m), 1036 (s), 988 (m), 960 (m), 898 (m), 859 (m), 810 (m), 736 (m), 714 (m), 634 (m), 615 (m), 595 (m), 537 (s). GC-MS (EI, 70 eV):  $m/z$  (%) = 280 ([M]<sup>+</sup>, 27), 248 (11), 235 (29), 234 (52), 206 (10), 205 (100), 204 (16), 203 (47), 202 (66), 176 (33), 134 (10), 133 (25), 105 (11), 77 (11), 43 (12). HRMS (EI): Calcd. for C<sub>14</sub>H<sub>16</sub>O<sub>6</sub> ([M]<sup>+</sup>): 280.09414; found: 280.094215.

20

**Diethyl 6-acetyl-4-ethyl-3-hydroxyphthalate (5c).** Starting with **3a** (0.321 g, 1.5 mmol) and **4c** (0.499 g, 1.65 mmol), **5c** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a white solid (0.231 g, 50%), mp. 57 – 59 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.17 (t, <sup>3</sup>J = 7.6 Hz, 3 H, CH<sub>2</sub>CH<sub>3</sub>), 1.29-1.34 (m, 6 H, 2 OCH<sub>2</sub>CH<sub>3</sub>), 2.48 (s, 3 H, CH<sub>3</sub>), 2.67 (t, <sup>3</sup>J = 7.7 Hz, 2 H, CH<sub>2</sub>CH<sub>3</sub>), 4.30- 4.38 (m, 4 H, 2×OCH<sub>2</sub>CH<sub>3</sub>), 7.72 (s, 1 H, CH<sub>Ar</sub>), 11.91 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.5, 13.8, 13.9 (CH<sub>3</sub>), 23.2 (CH<sub>2</sub>), 27.7 (CH<sub>3</sub>), 61.6, 62.8 (OCH<sub>2</sub>), 109.9 (CCOOCH<sub>3</sub>), 126.5, 133.7, 134.8 (C<sub>Ar</sub>), 134.9 (CH<sub>Ar</sub>), 163.1 (COH), 168.5, 169.6, 196.1 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2981 (w), 2928 (w), 2894 (w), 1733 (s), 1659 (s), 1610 (w), 1555 (w), 1441 (m), 1423 (m), 1377 (w), 1363 (m), 1331 (s), 1257 (s), 1215 (s), 1144 (m), 1110 (w), 1034 (m), 1023 (s), 991 (m), 904 (m), 869 (m), 817 (m), 799 (m), 720 (m), 622 (m), 598 (w), 540 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 308 ([M]<sup>+</sup>, 23), 263 (17), 262 (10), 219 (44), 217 (39), 216 (99), 190 (11), 189 (15), 188 (100), 147 (16), 43 (13). HRMS (EI): Calcd. for C<sub>16</sub>H<sub>20</sub>O<sub>6</sub> ([M]<sup>+</sup>): 308.12544; found: 308.124935.

35

**1-Ethyl 2-methyl 6-acetyl-4-butyl-3-hydroxyphthalate (5d).** Starting with **3a** (0.321 g, 1.5 mmol) and **4d** (0.522 g, 1.65 mmol), **5d** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.280 g, 58%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.85 (t, <sup>3</sup>J = 7.2 Hz, 3 H, (CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>), 1.29 (t, <sup>3</sup>J = 7.9 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.32-1.26 (m, 2 H, CH<sub>2</sub>), 1.47-1.58 (m, 2 H, CH<sub>2</sub>), 2.47 (s, 3 H, CH<sub>3</sub>), 2.65 (t, <sup>3</sup>J = 7.9 Hz, 2 H, CH<sub>3</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>Ph), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.35 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.70 (s, 1 H, CH<sub>Ar</sub>), 11.76 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 12.8, 13.1 (CH<sub>3</sub>), 21.5 (CH<sub>2</sub>), 26.5 (CH<sub>3</sub>), 28.6, 30.2 (CH<sub>2</sub>), 52.1 (OCH<sub>3</sub>), 60.7 (OCH<sub>2</sub>), 108.8 (CCOOCH<sub>3</sub>), 125.3, 131.5, 133.8 (C<sub>Ar</sub>), 134.8 (CH<sub>Ar</sub>), 162.1 (COH), 167.6, 169.1, 195.2 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2955 (w), 2929 (w), 2895 (w), 1732 (m), 1671(s), 1604 (w), 1569 (w), 1438 (m), 1340 (m), 1300 (w), 1255 (s), 1218 (s), 1164 (m), 1140 (m), 1093 (w), 1034 (m), 964 (w), 814 (m), 776

(w), 719 (m), 614 (w), 593 (w), 550 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 322 ( $[M]^+$ , 17), 277 (20), 248 (19), 247 (35), 245 (35), 244 (96), 234 (31), 217 (16), 216 (100), 215 (16), 202 (51), 175 (15), 147 (13), 43 (14). HRMS (EI): Calcd. for  $C_{17}H_{22}O_6$  ( $[M]^+$ ): 322.14109; found: 322.141032.

5

**1-Ethyl 2-methyl 6-acetyl-4-hexyl-3-hydroxyphthalate (5e).** Starting with **3a** (0.321 g, 1.5 mmol) and **4f** (0.568 g, 1.65 mmol), **5e** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.310 g, 59%), mp. 76 – 78 °C.  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.77 (t,  $^3J$  = 6.9 Hz, 3 H,  $(CH_2)_5CH_3$ ), 1.13-1.24 (m, 6 H,  $3\times CH_2$ ), 1.27 (t,  $^3J$  = 6.9 Hz, 3 H,  $OCH_2CH_3$ ), 1.44-1.54 (m, 2 H,  $CH_2$ ), 2.43 (s, 3 H,  $CH_3$ ), 2.57 (t,  $^3J$  = 7.4 Hz, 2 H,  $PhCH_2(CH_2)_4CH_3$ ), 3.82 (s, 3 H,  $OCH_3$ ), 4.30 (q,  $^3J$  = 7.1 Hz, 2 H,  $OCH_2CH_3$ ), 7.65 (s, 1 H,  $CH_{Ar}$ ), 11.72 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 14.0, 14.1, 27.6 ( $CH_3$ ), 22.6, 29.0, 29.1, 30.0, 31.7 ( $CH_2$ ), 53.2 ( $OCH_3$ ), 61.8 ( $OCH_2$ ), 110.0 ( $CCOOCH_3$ ), 126.4, 132.7 ( $C_{Ar}$ ), 135.0 ( $CCOOEt$ ), 135.9 ( $CH_{Ar}$ ), 163.4 ( $COH$ ), 168.9, 170.1, 196.2 (CO). IR (neat,  $cm^{-1}$ ):  $\nu_{max}$  = 2955 (w), 2927 (m), 2856 (w), 1733 (m), 1671 (s), 1606 (w), 1569 (w), 1439 (m), 1421 (m), 1340 (m), 1255 (s), 1218 (s), 1162 (m), 1140 (m), 1098 (m), 1034 (m), 964 (m), 909 (w), 884 (w), 863 (w), 814 (m), 776 (m), 724 (m), 647 (w), 615 (w), 594 (w), 554 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 350 ( $[M]^+$ , 10), 335 (4), 305 (17), 272 (100), 248 (14), 234 (39), 215 (14), 202 (57), 187 (35), 176 (7), 146 (9), 105 (5), 77 (6), 43 (15). HRMS (EI): Calcd. for  $C_{19}H_{26}O_6$  ( $[M]^+$ ): 350.17239; found: 350.172364.

15

20

**1-Ethyl 2-methyl 6-acetyl-3-hydroxy-4-octylphthalate (5f).** Starting with **3a** (0.321 g, 1.5 mmol) and **4g** (0.614 g, 1.65 mmol), **5f** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a brown solid (0.334 g, 59%), mp. 105-107 °C.  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.82 (t,  $^3J$  = 7.2 Hz, 3 H,  $(CH_2)_7CH_3$ ), 1.22 (t,  $^3J$  = 7.0 Hz, 3 H,  $OCH_2CH_3$ ), 1.35-1.39 (m, 10 H,  $5\times CH_2$ ), 1.47-1.58 (m, 2 H,  $CH_2$ ), 2.47 (s, 3 H,  $CH_3$ ), 2.62 (t,  $^3J$  = 7.9 Hz, 2 H,  $CH_3(CH_2)_6CH_2Ph$ ), 3.86 (s, 3 H,  $OCH_3$ ), 4.33 (q,  $^3J$  = 7.4 Hz, 2 H,  $OCH_2CH_3$ ), 7.70 (s, 1 H,  $CH_{Ar}$ ), 11.77 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.8, 14.0 ( $CH_3$ ), 22.6 ( $CH_2$ ), 27.6 ( $CH_3$ ), 28.6, 29.1, 29.2, 29.4, 29.9, 31.9 ( $CH_2$ ), 53.1 ( $OCH_3$ ), 61.7 ( $OCH_2$ ), 109.8 ( $CCOOCH_3$ ), 126.3, 132.5, 134.8 ( $C_{Ar}$ ), 134.8 ( $CH_{Ar}$ ), 163.2 ( $COH$ ), 168.5, 170.2, 196.3 (CO). IR (neat,  $cm^{-1}$ ):  $\nu_{max}$  = 2954 (w), 2924 (m), 2854 (w), 1734 (m), 1672 (s), 1606 (w), 1569 (w), 1439 (m), 1421 (m), 1342 (m), 1257 (m), 1220 (s), 1173 (w), 1141 (m), 1103 (w), 1036 (m), 965 (w), 907 (w), 814 (w), 777 (w), 724 (w), 724 (w), 647 (w), 615 (w), 594 (w), 557 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 378 ( $[M]^+$ , 9), 333 (16), 303 (19), 301 (29), 300 (100), 248 (15), 234 (42), 215 (13), 203 (10), 202 (50), 188 (11), 187 (19), 176 (20), 43 (14). HRMS (EI): Calcd. for  $C_{21}H_{30}O_6$  ( $[M]^+$ ): 378.20369; found: 378.203766.

25

30

35

**2-Ethyl 1-methyl 3-benzoyl-6-hydroxyphthalate (5g).** Starting with **3b** (0.414 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5g** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.212 g, 65%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.22 (t,  $^3J$  = 8.8 Hz, 3 H,  $OCH_2CH_3$ ), 3.88 (s, 3 H,  $OCH_3$ ), 4.17 (q,  $^3J$  = 7.5 Hz, 2 H,  $OCH_2CH_3$ ), 6.99 (d,  $^3J$  = 9.0 Hz, 1 H,  $CH_{Ar}$ ), 7.36-7.42 (m, 3 H,  $CH_{Ph}$ ), 7.49-7.56 (m, 2 H,  $CH_{Ph}$ ), 7.66 (d,  $^3J$  = 9.0 Hz, 1 H,  $CH_{Ar}$ ), 11.14 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.9 ( $CH_3$ ), 53.1 ( $OCH_3$ ), 61.9 ( $OCH_2$ ), 110.7 ( $CCOOCH_3$ ), 118.1 ( $CH_{Ar}$ ), 128.3 ( $2\times CH_{Ph}$ ), 128.7 ( $C_{Ar}$ ), 129.9 ( $2\times CH_{Ph}$ ), 132.9 ( $CH_{Ph}$ ), 136.4 ( $CH_{Ar}$ ), 137.2 ( $CCOOEt$ ), 137.6 ( $C_{Ar}$ ), 163.6 ( $COH$ ), 167.6, 169.3, 194.7 (CO). IR (neat,  $cm^{-1}$ ):  $\nu_{max}$  = 3241 (w), 3078 (w), 2976 (w), 2952 (w),

40

45

2851 (w), 1732 (m), 1662 (s), 1579 (m), 1444 (m), 1389 (w), 1332 (m), 1313 (m), 1280 (m), 1237 (s), 1177 (s), 1146 (m), 1112 (s), 1025 (m), 950 (m), 939 (m), 923 (m), 847 (m), 810 (m), 784 (m), 740 (m), 718 (m), 691 (s), 633 (s), 566 (m), 544 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 328 ( $[M]^+$ , 44), 296 (14), 283 (25), 282 (35), 252 (33), 251 (38), 225 (16), 224 (100), 223 (12), 196 (19), 195 (19), 191 (33), 168 (19), 139 (26), 119 (21), 105 (58), 77 (47), 51 (10). HRMS (EI): Calcd. for  $C_{18}H_{16}O_6$  ( $[M]^+$ ): 328.09414; found: 328.094310.

**1-Ethyl 2-methyl 6-benzoyl-3-hydroxy-4-methylphthalate (5h).** Starting with **3b** (0.414 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5h** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.343 g, 67%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.23 (t,  $^3J$  = 7.9 Hz, 3 H,  $OCH_2CH_3$ ), 2.21 (s, 3 H,  $CH_3$ ), 3.89 (s, 3 H,  $OCH_3$ ), 4.17 (q,  $^3J$  = 7.6 Hz, 2 H,  $OCH_2CH_3$ ), 7.37-7.42 (m, 3 H,  $CH_{Ph}$ ), 7.50 (s, 1 H,  $CH_{Ar}$ ), 7.54-7.70 (m, 2 H,  $CH_{Ph}$ ), 11.32 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.8, 16.0 ( $CH_3$ ), 52.9 ( $OCH_3$ ), 61.8 ( $OCH_2$ ), 110.2 ( $CCOOCH_3$ ), 128.0 ( $C_{Ar}$ ), 128.4 ( $3 \times CH_{Ph}$ ), 129.9 ( $2 \times CH_{Ph}$ ), 132.4, 135.1 ( $C_{Ar}$ ), 136.3 ( $CH_{Ar}$ ), 144.0 ( $C_{Ar}$ ), 161.6 ( $COH$ ), 167.7, 170.1, 194.8 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu$  = 3062 (w), 2980 (w), 2926 (w), 2853 (w), 1729 (m), 1666 (w), 1597 (s), 1493 (w), 1448 (m), 1391 (w), 1366 (m), 1262 (s), 1236 (s), 1181 (m), 1124 (m), 1096 (m), 1076 (m), 1055 (m), 1013 (m), 961 (m), 931 (m), 894 (m), 870 (m), 833 (m), 763 (s), 700 (s), 628 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 342 ( $[M]^+$ , 9), 264 (14), 238 (13), 199 (9), 147 (100), 105 (65), 91 (13), 85 (16), 84 (10), 77 (24), 71 (18), 69 (36), 57 (26), 55 (10), 43 (14). HRMS (EI): Calcd. for  $C_{19}H_{18}O_6$  ( $[M]^+$ ): 342.10979; found: 342.109901.

**Diethyl 6-benzoyl-4-ethyl-3-hydroxyphthalate (5i).** Starting with **3b** (0.414 g, 1.5 mmol) and **4c** (0.499 g, 1.65 mmol), **9i** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.345 g, 62%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.98 (t,  $^3J$  = 7.0 Hz, 3 H,  $CH_2CH_3$ ), 1.18 (t,  $^3J$  = 7.2 Hz, 3 H,  $OCH_2CH_3$ ), 1.33 (t,  $^3J$  = 7.9 Hz, 3 H,  $OCH_2CH_3$ ), 2.59 (q,  $^3J$  = 7.5 Hz, 2 H,  $CH_2CH_3$ ), 4.09 (q,  $^3J$  = 7.2 Hz, 2 H,  $OCH_2CH_3$ ), 4.32 (q,  $^3J$  = 7.5 Hz, 2 H,  $OCH_2CH_3$ ), 7.0 (s, 1 H,  $CH_{Ar}$ ), 7.40-7.45 (m, 3 H,  $CH_{Ph}$ ), 7.91-7.94 (m, 2 H,  $CH_{Ph}$ ), 11.48 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.4, 14.0, 14.0 ( $CH_3$ ), 22.7 ( $CH_2$ ), 61.6, 62.5 ( $OCH_2$ ), 110.1 ( $CCOOEt$ ), 128.5 ( $C_{Ar}$ ), 128.9 ( $3 \times CH_{Ph}$ ), 129.9 ( $2 \times CH_{Ph}$ ), 132.4, 134.5 ( $C_{Ar}$ ), 136.3 ( $CH_{Ar}$ ), 144.1 ( $C_{Ar}$ ), 162.1 ( $COH$ ), 167.6, 169.7, 190.6 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu$  = 3062 (w), 2961 (w), 2925 (w), 2851 (w), 1729 (m), 1666 (w), 1598 (m), 1448 (m), 1391 (w), 1367 (m), 1262 (s), 1238 (s), 1181 (m), 1125 (m), 1095 (m), 1014 (m), 971 (w), 868 (w), 833 (w), 792 (w), 764 (m), 700 (m), 684 (m), 628 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 370 ( $[M]^+$ , 24), 325 (13), 324 (10), 279 (31), 278 (92), 251 (24), 250 (100), 194 (12), 165 (16), 152 (11), 147 (10), 105 (28), 77 (29), 29 (10). HRMS (EI): Calcd. for  $C_{21}H_{22}O_6$  ( $[M]^+$ ): 370.14109; found: 370.141006.

**Diethyl 6-benzoyl-4-ethyl-3-hydroxyphthalate (5j).** Starting with **3b** (0.414 g, 1.5 mmol) and **4d** (0.522 g, 1.65 mmol), **5j** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.375 g, 65%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.80 (t,  $^3J$  = 7.1 Hz, 3 H,  $(CH_2)_3CH_3$ ), 1.18 (t,  $^3J$  = 8.2 Hz, 3 H,  $OCH_2CH_3$ ), 1.22-1.27 (m, 2 H,  $(CH_2)_2CH_2CH_3$ ), 1.45-1.55 (m, 2 H,  $PhCH_2CH_2CH_2CH_3$ ), 2.58 (t,  $^3J$  = 7.0 Hz, 2 H,  $PhCH_2(CH_2)_2CH_3$ ), 3.85 (s, 3 H,  $OCH_3$ ), 4.14 (q,  $^3J$  = 7.5 Hz, 2 H,  $OCH_2CH_3$ ), 7.17-7.20 (m, 3 H,  $CH_{Ph}$ ), 7.23 (s, 1 H,  $CH_{Ar}$ ), 7.58 (d,  $^3J$  = 8.8 Hz, 2 H,  $CH_{Ph}$ ), 11.32 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.9, 21.6 ( $CH_3$ ), 22.6, 29.7, 31.6 ( $CH_2$ ), 52.9 ( $OCH_3$ ), 61.7

(OCH<sub>2</sub>), 110.1 (CCOOMe), 128.4 (C<sub>Ar</sub>), 129.0 (3×CH<sub>Ph</sub>), 130.2 (2×CH<sub>Ph</sub>), 132.2, 134.6 (C<sub>Ar</sub>), 135.9 (CH<sub>Ar</sub>), 144.1 (C<sub>Ar</sub>), 161.3 (COH), 168.1, 170.1, 194.9 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 2953 (w), 2925 (w), 2856 (w), 1734 (m), 1662 (m), 1604 (m), 1572 (w), 1439 (m), 1346 (m), 1234 (s), 1168 (m), 1096 (m), 1057 (m), 1016 (m), 956 (m), 915 (w), 865 (w), 812 (m), 761 (m), 721 (m), 585 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 384 ([M]<sup>+</sup>, 24), 339 (14), 310 (10), 307 (28), 306 (100), 296 (18), 279 (17), 278 (70), 277 (13), 265 (14), 264 (56), 263 (13), 249 (11), 237 (10), 152 (12), 105 (18), 77 (19). HRMS (EI): Calcd. for C<sub>22</sub>H<sub>24</sub>O<sub>6</sub> ([M]<sup>+</sup>): 384.15674; found: 384.156760.

**1-Ethyl 2-methyl 6-benzoyl-4-hexyl-3-hydroxyphthalate (5k).** Starting with **3b** (0.414 g, 1.5 mmol) and **4f** (0.568 g, 1.65 mmol), **5k** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.401 g, 65%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.80 (t, <sup>3</sup>*J* = 7.8 Hz, 3 H, (CH<sub>2</sub>)<sub>5</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>*J* = 8.5 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.23-1.28 (m, 6 H, 3×CH<sub>2</sub>), 1.45-1.55 (m, 2 H, CH<sub>2</sub>), 2.58 (t, <sup>3</sup>*J* = 7.1 Hz, 2 H, PhCH<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.16 (q, <sup>3</sup>*J* = 7.2 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.37-7.42 (m, 3 H, CH<sub>Ph</sub>), 7.51 (s, 1 H, CH<sub>Ar</sub>), 7.54-7.70 (m, 2 H, CH<sub>Ph</sub>), 11.35 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.9, 14.0 (CH<sub>3</sub>), 22.6, 28.8, 28.9, 29.7, 31.6 (CH<sub>2</sub>), 52.9 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.2 (CCOOEt), 128.0 (C<sub>Ar</sub>), 129.9 (3×CH<sub>Ph</sub>), 132.2 (2×CH<sub>Ph</sub>), 132.9, 134.9 (C<sub>Ar</sub>), 135.9 (CH<sub>Ar</sub>), 137.3 (C<sub>Ar</sub>), 161.7 (COH), 167.8, 169.9, 195.1 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 3059 (w), 2954 (w), 2926 (w), 2855 (w), 1733 (m), 1664 (s), 1597 (w), 1576 (w), 1439 (m), 1345 (m), 1232 (s), 1199 (m), 1169 (m), 1097 (w), 1056 (m), 1026 (w), 958 (m), 914 (w), 815 (m), 711 (m), 692 (m), 628 (m), 585 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 412 ([M]<sup>+</sup>, 14), 367 (13), 335 (29), 334 (100), 310 (11), 306 (17), 296 (27), 277 (11), 265 (17), 264 (60), 250 (13), 249 (43), 152 (14), 105 (24), 77 (21). HRMS (EI): Calcd. for C<sub>24</sub>H<sub>28</sub>O<sub>6</sub> ([M]<sup>+</sup>): 412.18804; found: 412.187659.

**1-Ethyl 2-methyl 6-benzoyl-3-hydroxy-4-octylphthalate (5l).** Starting with **3b** (0.414 g, 1.5 mmol) and **4g** (0.614 g, 1.65 mmol), **5l** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.435 g, 66%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.81 (t, <sup>3</sup>*J* = 7.7 Hz, 3 H, CH<sub>3</sub>), 1.20-1.26 (m, 13 H, 1×CH<sub>3</sub>, 5×CH<sub>2</sub>), 1.46-1.52 (m, 2 H, CH<sub>2</sub>), 2.58 (t, <sup>3</sup>*J* = 7.1 Hz, 2 H, PhCH<sub>2</sub>(CH<sub>2</sub>)<sub>6</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.15 (q, <sup>3</sup>*J* = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.37-7.41 (m, 3 H, CH<sub>Ph</sub>), 7.49 (s, 1 H, CH<sub>Ar</sub>), 7.66-7.69 (m, 2 H, CH<sub>Ph</sub>), 11.36 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.9, 14.0 (CH<sub>3</sub>), 22.6, 29.0, 29.1, 29.2, 29.3, 29.8, 31.8 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.2 (CCOOEt), 128.0 (C<sub>Ar</sub>), 128.3 (3×CH<sub>Ph</sub>), 130.0 (2×CH<sub>Ph</sub>), 132.9, 134.9 (C<sub>Ar</sub>), 135.9 (CH<sub>Ar</sub>), 137.2 (C<sub>Ar</sub>), 161.7 (COH), 167.8, 169.8, 195.1 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 3060 (w), 2953 (m), 2924 (w), 2853 (w), 1733 (m), 1665 (s), 1596 (w), 1576 (w), 1500 (w), 1440 (m), 1346 (m), 1252 (s), 1232 (s), 1199 (m), 1169 (m), 1097 (w), 1055 (m), 1026 (w), 1016 (w), 959 (m), 915 (w), 865 (w), 814 (m), 773 (w), 711 (m), 691 (m), 665 (w), 628 (w), 585 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 440 ([M]<sup>+</sup>, 12), 395 (14), 363 (30), 362 (100), 310 (12), 296 (32), 277 (11), 265 (16), 264 (54), 263 (12), 250 (14), 249 (25), 238 (28), 152 (12), 105 (25), 77 (17), 43 (8). HRMS (EI): Calcd. for C<sub>26</sub>H<sub>32</sub>O<sub>6</sub> ([M]<sup>+</sup>): 440.21934; found: 440.218868.

**1-Ethyl 2-methyl 6-benzoyl-3-hydroxy-4-nonylphthalate (5m).** Starting with **3b** (0.414 g, 1.5 mmol) and **4h** (0.638 g, 1.65 mmol), **5m** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.456 g, 67%). <sup>1</sup>H NMR (300 MHz,

CDCl<sub>3</sub>):  $\delta$  = 0.80 (t,  $^3J$  = 6.4 Hz, 3 H, (CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 1.18 (t,  $^3J$  = 7.4 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.25 (m, 12 H, 6×CH<sub>2</sub>), 1.45-1.52 (m, 2 H, CH<sub>2</sub>), 2.58 (t,  $^3J$  = 7.4 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.14 (q,  $^3J$  = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.36-7.41 (m, 3 H, CH<sub>Ph</sub>), 7.51 (s, 1 H, CH<sub>Ar</sub>), 7.68 (d,  $^3J$  = 8.0 Hz, 2 H, CH<sub>Ph</sub>), 11.35 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.9, 14.1 (CH<sub>3</sub>), 22.6, 29.0, 29.2, 29.3, 29.4, 29.5, 29.8, 31.9 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.8 (OCH<sub>2</sub>), 110.2 (CCOOMe), 128.1 (C<sub>Ar</sub>), 128.3 (2×CH<sub>Ph</sub>), 130.0 (2×CH<sub>Ph</sub>), 132.4 (C<sub>Ar</sub>), 132.9 (CH<sub>Ph</sub>), 135.0 (CCOOEt), 136.0 (CH<sub>Ar</sub>), 137.3 (C<sub>Ar</sub>), 161.8 (COH), 167.8, 169.9, 195.1 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 3059 (w), 2953 (w), 2923 (m), 2853 (m), 1734 (m), 1665 (m), 1597 (w), 1476 (w), 1440 (m), 1421 (m), 1346 (m), 1252 (m), 1233 (s), 1200 (m), 1170 (m), 1056 (m), 1026 (m), 960 (m), 865 (w), 768 (m), 711 (m), 692 (m), 665 (w), 628 (w), 586 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 454 ([M]<sup>+</sup>, 10), 409 (5), 376 (40), 296 (10), 264 (15), 242 (12), 169 (15), 129 (33), 116 (80), 105 (100), 97 (13), 84 (24), 77 (50), 57 (23), 43 (55). HRMS (EI): Calcd. for C<sub>27</sub>H<sub>34</sub>O<sub>6</sub> ([M]<sup>+</sup>): 454.23499; found: 454.236232.

15

**1-Ethyl 2-methyl 6-benzoyl-4-decyl-3-hydroxyphthalate (5n).** Starting with **3b** (0.414 g, 1.5 mmol) and **4i** (0.661 g, 1.65 mmol), **5n** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.463 g, 66%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.80 (t,  $^3J$  = 7.1 Hz, 3 H, (CH<sub>2</sub>)<sub>9</sub>CH<sub>3</sub>), 1.18 (t,  $^3J$  = 7.7 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.25 (m, 14 H, 7×CH<sub>2</sub>), 1.45-1.52 (m, 2 H, CH<sub>2</sub>), 2.58 (t,  $^3J$  = 7.2 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.14 (q,  $^3J$  = 7.7 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.36-7.41 (m, 3 H, CH<sub>Ph</sub>), 7.51 (s, 1 H, CH<sub>Ar</sub>), 7.67 (d,  $^3J$  = 8.9 Hz, 2 H, CH<sub>Ph</sub>), 11.35 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.9, 14.1 (CH<sub>3</sub>), 22.7, 29.0, 29.3, 29.4, 29.5, 29.6, 29.7, 29.9, 31.9 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.8 (OCH<sub>2</sub>), 110.2 (CCOOMe), 128.1 (C<sub>Ar</sub>), 128.3 (2×CH<sub>Ph</sub>), 130.0 (2×CH<sub>Ph</sub>), 132.3 (C<sub>Ar</sub>), 132.9 (CH<sub>Ph</sub>), 135.0 (CCOOEt), 136.0 (CH<sub>Ar</sub>), 137.3 (C<sub>Ar</sub>), 161.7 (COH), 167.8, 169.9, 195.1 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 2953 (w), 2922 (m), 2853 (m), 1734(m), 1665 (m), 1597 (w), 1576 (w), 1440 (m), 1346 (m), 1252 (s), 1233 (s), 1200 (m), 1056 (m), 1026 (m), 959 (m), 926 (w), 866 (w), 815 (m), 711 (m), 692 (m), 586 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 468 ([M]<sup>+</sup>, 32), 422 (23), 407 (8), 390 (100), 363 (13), 296 (55), 277 (13), 264 (75), 249 (26), 238 (48), 209 (6), 180 (7), 152 (7), 105 (15), 77 (5). HRMS (EI): Calcd. for C<sub>28</sub>H<sub>36</sub>O<sub>6</sub> ([M]<sup>+</sup>): 468.25064; found: 468.251834.

**1-Ethyl 2-methyl 3-hydroxy-6-(4-methylbenzoyl)phthalate (5o).** Starting with **3c** (0.435 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5o** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.353 g, 69 %), mp. 70-72 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.22 (t,  $^3J$  = 8.8 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.35 (s, 3 H, CH<sub>3</sub>), 3.87 (s, 3 H, OCH<sub>3</sub>), 4.17 (q,  $^3J$  = 7.5 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 6.99 (d,  $^3J$  = 9.0 Hz, 1 H, CH<sub>Ar</sub>), 7.17-7.20 (m, 2 H, CH<sub>Tol</sub>), 7.50-7.59 (m, 3 H, CH<sub>Ar,Tol</sub>), 11.10 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.9, 21.6 (CH<sub>3</sub>), 53.0 (OCH<sub>3</sub>), 61.8 (OCH<sub>2</sub>), 110.7 (CCOOCH<sub>3</sub>), 118.0 (CH<sub>Ar</sub>), 128.0 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 130.1 (2×CH<sub>Tol</sub>), 133.4 (C<sub>Ar</sub>), 136.2 (CH<sub>Ar</sub>), 137.4 (CCOOEt), 143.9 (C<sub>Ar</sub>), 163.4 (COH), 167.6, 169.3, 194.4 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 3030 (w), 2981 (w), 2955 (w), 2851 (w), 1731 (m), 1658 (s), 1604 (m), 1582 (m), 1441 (m), 1324 (m), 1245 (s), 1213 (s), 1181 (s), 1146 (m), 1114 (s), 1029 (s), 959 (m), 937 (m), 833 (m), 811 (m), 759 (m), 728 (m), 686 (m), 639 (m), 587 (s), 534 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 342 ([M]<sup>+</sup>, 42), 297 (22), 296 (28), 266 (30), 265 (32), 251 (12), 239 (16), 238 (100), 223 (16), 210 (14),

45



191 (23), 182 (11), 181 (15), 153 (13), 152 (11), 120 (10), 119 (75), 91 (43), 65 (13). HRMS (EI): Calcd. for C<sub>19</sub>H<sub>18</sub>O<sub>6</sub> ([M]<sup>+</sup>): 342.10979; found: 342.10099.

**1-Ethyl 2-methyl 3-hydroxy-4-methyl-6-(4-methylbenzoyl)phthalate (5p).** Starting with **3c** (0.435 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5p** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.373 g, 70%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.19 (t, <sup>3</sup>J = 8.4 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.20 (s, 3 H, CH<sub>3</sub>), 2.35 (s, 3 H, CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.15 (q, <sup>3</sup>J = 7.5 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.17-7.21 (m, 2 H, CH<sub>Tol</sub>), 7.37 (s, 1 H, CH<sub>Ar</sub>), 7.56-7.59 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 16.0, 21.7 (CH<sub>3</sub>), 52.9 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.1 (CCOOCH<sub>3</sub>), 127.8 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 129.7 (2×CH<sub>Tol</sub>), 132.2, 134.6, 134.7 (C<sub>Ar</sub>), 136.6 (CH<sub>Ar</sub>), 143.9 (C<sub>Ar</sub>), 161.9 (COH), 167.7, 170.0, 194.8 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 3030 (w), 2980 (w), 2954 (w), 1731 (m), 1660 (s), 1604 (m), 1573 (w), 1439 (m), 1408 (m), 1379 (w), 1408 (w), 1379 (w), 1338 (m), 1308 (m), 1236 (s), 1200 (m), 1167 (s), 1113 (w), 1054 (s), 1014 (m), 959 (m), 910 (w), 886 (w), 866 (w), 810 (m), 761 (m), 711 (m), 658 (w), 642 (w), 628 (w), 601 (m), 582 (m). GC-MS (EI, 70 eV): *m/z* (%) = 356 ([M]<sup>+</sup>, 57), 324 (26), 311 (27), 310 (55), 280 (24), 279 (49), 278 (100), 253 (13), 252 (83), 250 (11), 224 (11), 205 (13), 165 (10), 161 (17), 119 (55), 91 (30), 71 (11), 69 (14), 57 (16), 55 (10), 43 (10). HRMS (EI): Calcd. for C<sub>20</sub>H<sub>20</sub>O<sub>6</sub> ([M]<sup>+</sup>): 356.12544; found: 356.125057.

**1-Ethyl 2-methyl 4-butyl-3-hydroxy-6-(4-methylbenzoyl)phthalate (5q).** Starting with **3c** (0.435 g, 1.5 mmol) and **4d** (0.522 g, 1.65 mmol), **5q** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.481 g, 70%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.80 (t, <sup>3</sup>J = 7.7 Hz, 3 H, (CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>J = 8.8 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.22-1.27 (m, 2 H, (CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 1.45-1.55 (m, 2 H, CH<sub>2</sub>), 2.36 (s, 3 H, CH<sub>3</sub>), 2.61 (t, <sup>3</sup>J = 7.7 Hz, 2 H, PhCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>3</sub>), 3.85 (s, 3 H, OCH<sub>3</sub>), 4.16 (q, <sup>3</sup>J = 7.6 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.17-7.21 (m, 2 H, CH<sub>Tol</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.56-7.59 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.9, 14.0, 21.6 (CH<sub>3</sub>), 22.9, 29.8, 31.3 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 62.1 (OCH<sub>2</sub>), 110.1 (CCOOCH<sub>3</sub>), 128.2 (C<sub>Ar</sub>), 129.2 (2×CH<sub>Tol</sub>), 130.5 (2×CH<sub>Tol</sub>), 132.2, 134.6, 134.8 (C<sub>Ar</sub>), 136.6 (CH<sub>Ar</sub>), 143.7 (C<sub>Ar</sub>), 162.2 (COH), 168.1, 170.3, 195.1 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2955 (w), 2926 (w), 2858 (w), 1732 (m), 1662 (m), 1604 (m), 1572 (w), 1439 (m), 1421 (m), 1346 (m), 1310 (m), 1234 (s), 1206 (m), 1167 (m), 1093 (w), 1055 (m), 1016 (m), 960 (m), 911 (w), 865 (w), 836 (w), 812 (m), 761 (w), 729 (w), 664 (w), 585 (w). GC-MS (EI, 70 eV): *m/z* (%) = 398 ([M]<sup>+</sup>, 28), 353 (16), 324 (12), 321 (29), 320 (100), 310 (32), 293 (17), 292 (75), 291 (17), 279 (21), 278 (81), 277 (31), 263 (18), 251 (14), 250 (10), 165 (16), 119 (30), 91 (33). HRMS (EI): Calcd. for C<sub>23</sub>H<sub>26</sub>O<sub>6</sub> ([M]<sup>+</sup>): 398.17239; found: 398.172357.

**1-Ethyl 2-methyl 4-hexyl-3-hydroxy-6-(4-methylbenzoyl)phthalate (5r).** Starting with **3c** (0.435 g, 1.5 mmol) and **4f** (0.568 g, 1.65 mmol), **5r** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.415 g, 65%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.80 (t, <sup>3</sup>J = 7.1 Hz, 3 H, (CH<sub>2</sub>)<sub>5</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>J = 8.8 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.24 (m, 6 H, 3×CH<sub>2</sub>), 1.46-1.52 (m, 2 H, CH<sub>2</sub>), 2.36 (s, 3 H, CH<sub>3</sub>), 2.58 (t, <sup>3</sup>J = 7.0 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.15 (q, <sup>3</sup>J = 7.6 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.18-7.20 (m, 2 H, CH<sub>Tol</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.57-7.60 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.9, 14.0, 21.6 (CH<sub>3</sub>), 22.5, 29.0, 29.0, 29.7,

31.6 (CH<sub>2</sub>), 52.9 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.1 (CCOOMe), 128.4 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 130.2 (2×CH<sub>Tol</sub>), 132.2, 134.6, 134.7 (C<sub>Ar</sub>), 135.9 (CH<sub>Ar</sub>), 143.8 (C<sub>Ar</sub>), 161.5 (COH), 167.8, 170.0, 194.9 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 2953 (w), 2925 (w), 2856 (w), 1734 (m), 1663 (m), 1604 (m), 1572 (w), 1439 (m), 1419 (m), 1346 (m), 1234 (s), 1169 (m), 1096 (m), 1057 (m), 1016 (m), 956 (m), 914 (w), 866 (w), 812 (m), 762 (m), 722 (m), 585 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 426 ([M]<sup>+</sup>, 16), 381 (14), 349 (31), 348 (100), 324 (12), 320 (23), 310 (38), 291 (14), 279 (25), 278 (73), 277 (14), 264 (17), 263 (59), 252 (11), 203 (11), 165 (15), 119 (29), 91 (28), 29 (10). HRMS (EI): Calcd. for C<sub>25</sub>H<sub>30</sub>O<sub>6</sub> ([M]<sup>+</sup>): 426.20369; found: 426.203912.

10  
**1-Ethyl 2-methyl 3-hydroxy-6-(4-methylbenzoyl)-4-octylphthalate (5s)**

Starting with **3c** (0.435 g, 1.5 mmol) and **4g** (0.614 g, 1.65 mmol), **5s** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.435 g, 64%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.80 (t, <sup>3</sup>*J* = 7.1 Hz, 3 H, (CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>*J* = 8.9 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.27 (m, 10 H, 5×CH<sub>2</sub>), 1.46-1.53 (m, 2 H, CH<sub>2</sub>), 2.36 (s, 3 H, CH<sub>3</sub>), 2.58 (t, <sup>3</sup>*J* = 7.0 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>6</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.14 (q, <sup>3</sup>*J* = 7.5 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.17-7.20 (m, 2 H, CH<sub>Tol</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.57-7.60 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.8, 14.0, 21.6 (CH<sub>3</sub>), 22.6, 29.0, 29.2, 29.3, 29.3, 29.7, 31.8 (CH<sub>2</sub>), 52.9 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.1 (CCOOMe), 128.4 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 130.2 (2×CH<sub>Tol</sub>), 132.2, 134.6, 134.7 (C<sub>Ar</sub>), 135.8 (CH<sub>Ar</sub>), 143.8 (C<sub>Ar</sub>), 161.5 (COH), 167.8, 169.9, 194.8 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 2953 (w), 2923 (w), 2853 (w), 1735 (m), 1663 (m), 1605 (m), 1572 (w), 1439 (m), 1419 (m), 1346 (m), 1234 (s), 1169 (m), 1055 (m), 1016 (m), 960 (m), 915 (w), 866 (w), 812 (m), 761 (m), 719 (m), 585 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 454 ([M]<sup>+</sup>, 13), 409 (16), 408 (13), 377 (32), 376 (100), 348 (324), 311 (11), 310 (47), 291 (16), 279 (25), 278 (71), 277 (20), 265 (11), 264 (20), 263 (43), 253 (10), 252 (27), 250 (10), 207 (16), 165 (15), 119 (36), 91 (28), 44 (11), 43 (10), 41 (10), 29 (11). HRMS (EI): Calcd. for C<sub>27</sub>H<sub>34</sub>O<sub>6</sub> ([M]<sup>+</sup>): 454.23499; found: 454.234275.

1-Ethyl 2-methyl 3-hydroxy-6-(4-methylbenzoyl)-4-nonylphthalate (**5t**). Starting with **3c** (0.435 g, 1.5 mmol) and **4h** (0.638 g, 1.65 mmol), **5t** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.463 g, 66%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.80 (t, <sup>3</sup>*J* = 7.1 Hz, 3 H, (CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>*J* = 8.9 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.27 (m, 12 H, 6×CH<sub>2</sub>), 1.46-1.53 (m, 2 H, CH<sub>2</sub>), 2.36 (s, 3 H, CH<sub>3</sub>), 2.58 (t, <sup>3</sup>*J* = 7.0 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.14 (q, <sup>3</sup>*J* = 7.5 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.17-7.20 (m, 2 H, CH<sub>Tol</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.57-7.60 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  = 13.8, 14.0, 21.6 (CH<sub>3</sub>), 22.6, 29.0, 29.2, 29.3, 29.3, 29.4, 29.7, 31.8 (CH<sub>2</sub>), 52.9 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.1 (CCOOMe), 128.4 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 130.2 (2×CH<sub>Tol</sub>), 132.2, 134.6, 134.7 (C<sub>Ar</sub>), 135.8 (CH<sub>Ar</sub>), 143.8 (C<sub>Ar</sub>), 161.5 (COH), 167.8, 169.9, 194.8 (CO). IR (neat, cm<sup>-1</sup>):  $\nu$  = 2953 (w), 2923 (w), 2853 (w), 1735 (m), 1663 (m), 1605 (m), 1572 (w), 1439 (m), 1419 (m), 1346 (m), 1234 (s), 1169 (m), 1055 (m), 1016 (m), 960 (m), 915 (w), 866 (w), 812 (m), 761 (m), 719 (m), 585 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 468 ([M]<sup>+</sup>, 23), 423 (13), 390 (86), 363 (9), 324 (10), 310 (31), 291 (8), 278 (39), 263 (20), 169 (14), 135 (50), 119 (100), 91 (46), 84 (12), 57 (15). HRMS (EI): Calcd. for C<sub>28</sub>H<sub>37</sub>O<sub>6</sub> [(M+H)]<sup>+</sup>: 469.25847; found: 469.25827.

**1-Ethyl 2-methyl 4-decyl-3-hydroxy-6-(4-methylbenzoyl)phthalate (5u).** Starting with **3c** (0.435 g, 1.5 mmol) and **4i** (0.661 g, 1.65 mmol), **5u** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.470 g, 65%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.80 (t, <sup>3</sup>J = 6.4 Hz, 3 H, (CH<sub>2</sub>)<sub>9</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>J = 7.5 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.25 (m, 14 H, 7×CH<sub>2</sub>), 1.45-1.51 (m, 2 H, CH<sub>2</sub>), 2.35 (s, 3 H, CH<sub>3</sub>), 2.58 (t, <sup>3</sup>J = 7.5 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 3.85 (s, 3 H, OCH<sub>3</sub>), 4.14 (q, <sup>3</sup>J = 7.2 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.17-7.19 (m, 2 H, CH<sub>Tol</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.57-7.59 (m, 2 H, CH<sub>Tol</sub>), 11.32 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.9, 14.1, 21.7 (CH<sub>3</sub>), 22.7, 29.1, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 31.9 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.2 (CCOOMe), 128.5 (C<sub>Ar</sub>), 129.0 (2×CH<sub>Tol</sub>), 130.3 (2×CH<sub>Tol</sub>), 132.3, 134.7 (C<sub>Ar</sub>), 134.8 (CCOOEt), 136.0 (CH<sub>Ar</sub>), 143.8 (C<sub>Ar</sub>), 161.5 (COH), 167.9, 169.9, 194.8 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2953 (w), 2922 (m), 2853 (m), 1735 (m), 1664 (m), 1605 (m), 1573 (w), 1439 (m), 1420 (m), 1347 (m), 1235 (s), 1169 (m), 1057 (m), 1018 (m), 960 (m), 888 (w), 866 (w), 813 (m), 762 (m), 719 (m), 586 (m). GC-MS (EI, 70 eV): *m/z* (%) = 482 ([M]<sup>+</sup>, 14), 436 (14), 404 (74), 377 (18), 324 (11), 310 (32), 291 (13), 278 (62), 263 (29), 223 (9), 194 (11), 165 (16), 135 (100), 119 (84), 91 (49), 55 (22), 43 (91). HRMS (EI): Calcd. for C<sub>29</sub>H<sub>38</sub>O<sub>6</sub> ([M]<sup>+</sup>): 482.26629; found: 482.267429.

**1-Ethyl 2-methyl 3-hydroxy-6-(4-methoxybenzoyl)phthalate (5v).** Starting with **3d** (0.459 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5v** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.333 g, 62%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.21 (t, <sup>3</sup>J = 7.7 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 3.80 (s, 3 H, OCH<sub>3</sub>), 3.87 (s, 3 H, OCH<sub>3</sub>), 4.18 (q, <sup>3</sup>J = 7.8 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 6.84-6.87 (m, 2 H, CH<sub>PhOMe</sub>), 7.0 (d, <sup>3</sup>J = 8.8 Hz, 1 H, CH<sub>Ar</sub>), 7.51 (d, <sup>3</sup>J = 8.5 Hz, 1 H, CH<sub>Ar</sub>), 7.64-7.69 (m, 2 H, CH<sub>PhOMe</sub>), 11.56 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.7 (CH<sub>3</sub>), 52.7, 55.4 (OCH<sub>3</sub>), 61.7 (OCH<sub>2</sub>), 110.7 (CCOOCH<sub>3</sub>), 113.0 (2×CH<sub>PhOMe</sub>), 118.2 (CH<sub>Ar</sub>), 129.3, 129.7, 132.0 (C<sub>Ar</sub>), 132.5 (2×CH<sub>PhOMe</sub>), 135.9 (CH<sub>Ar</sub>), 137.0 (C<sub>Ar</sub>), 163.6 (COH), 167.4, 169.4, 193.4 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 3076 (w), 2979 (w), 2955 (w), 2903 (w), 2841 (w), 1729 (m), 1676 (m), 1655 (m), 1596 (s), 1509 (m), 1441 (m), 1310 (m), 1246 (s), 1218 (s), 1162 (s), 1146 (s), 1114 (m), 1025 (s), 958 (m), 912 (m), 842 (m), 811 (m), 771 (m), 728 (m), 689 (w), 635 (w), 593 (m), 535 (w). GC-MS (EI, 70 eV): *m/z* (%) = 358 ([M]<sup>+</sup>, 60), 313 (21), 312 (22), 282 (36), 281 (33), 255 (18), 254 (100), 253 (31), 226 (17), 225 (14), 191 (26), 135 (86), 119 (15), 107 (11), 92 (20), 77 (26). HRMS (EI): Calcd. for C<sub>19</sub>H<sub>18</sub>O<sub>7</sub> ([M]<sup>+</sup>): 358.10470; found: 358.104802.

**1-Ethyl 2-methyl 3-hydroxy-6-(4-methoxybenzoyl)-4-methylphthalate (5w).** Starting with **3d** (0.459 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5w** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.351 g, 63%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.18 (t, <sup>3</sup>J = 7.4 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.21 (s, 3 H, CH<sub>3</sub>), 3.81 (s, 3 H, OCH<sub>3</sub>), 3.85 (s, 3 H, OCH<sub>3</sub>), 4.11 (q, <sup>3</sup>J = 7.5 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 6.84-6.87 (m, 2 H, CH<sub>PhOMe</sub>), 7.36 (s, 1 H, CH<sub>Ar</sub>), 7.65-7.70 (m, 2 H, CH<sub>PhOMe</sub>), 11.26 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.8, 14.0 (CH<sub>3</sub>), 53.0, 55.8 (OCH<sub>3</sub>), 61.8, (OCH<sub>2</sub>), 110.1 (CCOOCH<sub>3</sub>), 113.5 (2×CH<sub>PhOMe</sub>), 127.7, 128.9, 129.9 (C<sub>Ar</sub>), 132.7 (2×CH<sub>PhOMe</sub>), 134.6 (C<sub>Ar</sub>), 136.1 (CH<sub>Ar</sub>), 161.6 (C<sub>Ar</sub>), 163.4 (COH), 168.1, 170.3, 193.9 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2979 (w), 2955 (w), 2936 (w), 2841 (w), 2254 (w), 1731 (m), 1671 (m), 1655 (m), 1596 (s), 1573 (m), 1509 (m), 1439 (m), 1413 (m), 1380 (w), 1340 (m), 1305 (m), 1238 (s), 1200 (m),

1161 (s), 1111 (m), 1054 (s), 1018 (m), 959 (m), 910 (m), 844 (m), 810 (m), 746 (m), 728 (m), 645 (m), 604 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 372 ( $[M]^+$ , 80), 340 (21), 327 (29), 326 (58), 296 (33), 295 (55), 294 (100), 269 (13), 268 (81), 267 (25), 240 (19), 239 (12), 238 (10), 205 (12), 135 (65), 133 (11), 92 (12), 77 (16). HRMS (EI): Calcd. for  $C_{20}H_{20}O_7$  ( $[M]^+$ ): 372.12035; found: 372.120601.

**Diethyl 4-ethyl-3-hydroxy-6-(4-methoxybenzoyl)phthalate (5x).** Starting with **3d** (0.459 g, 1.5 mmol) and **4c** (0.499 g, 1.65 mmol), **5x** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.335 g, 56%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.11 (t,  $^3J$  = 7.5 Hz, 3 H,  $CH_2CH_3$ ), 1.15 (t,  $^3J$  = 7.3 Hz, 3 H,  $OCH_2CH_3$ ), 1.31 (t,  $^3J$  = 7.9 Hz, 3 H,  $OCH_2CH_3$ ), 2.63 (q,  $^3J$  = 7.5 Hz, 2 H,  $CH_2CH_3$ ), 3.80 (s, 3 H,  $OCH_3$ ), 4.09 (q,  $^3J$  = 7.1 Hz, 2 H,  $OCH_2CH_3$ ), 4.32 (q,  $^3J$  = 7.2 Hz, 2 H,  $OCH_2CH_3$ ), 6.85-6.87 (m, 2 H,  $CH_{PhOMe}$ ), 7.35 (s, 1 H,  $CH_{Ar}$ ), 7.66-7.69 (m, 2 H,  $CH_{PhOMe}$ ), 11.38 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.4, 13.7, 13.7 ( $CH_3$ ), 22.9 ( $CH_2$ ), 55.5 ( $OCH_3$ ), 61.5, 62.5 ( $OCH_2$ ), 110.1 ( $CCOOEt$ ), 113.5 ( $2 \times CH_{PhOMe}$ ), 128.9, 129.9 ( $C_{Ar}$ ), 132.4 ( $2 \times CH_{PhOMe}$ ), 133.5, 134.3 ( $C_{Ar}$ ), 134.4 ( $CH_{Ar}$ ), 161.3 ( $C_{Ar}$ ), 163.5 (COH), 167.7, 169.5, 194.0 (CO). IR (neat,  $cm^{-1}$ ):  $\nu$  = 2975 (w), 2935 (w), 2840 (w), 1731 (m), 1657 (m), 1596 (s), 1573 (m), 1509 (m), 1442 (m), 1417 (m), 1374 (m), 1339 (m), 1305 (m), 1251 (s), 1235 (s), 1185 (m), 1163 (s), 1095 (m), 1053 (w), 1020 (s), 986 (m), 908 (m), 846 (m), 772 (m), 762 (m), 704 (m), 605 (m), 586 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 400 ( $[M]^+$ , 29), 355 (16), 354 (17), 310 (13), 309 (32), 308 (100), 282 (14), 281 (16), 280 (65), 279 (11), 219 (18), 147 (12), 135 (62), 107 (12), 92 (14), 77 (14). HRMS (EI): Calcd. for  $C_{22}H_{24}O_7$  ( $[M]^+$ ): 400.15165; found: 400.151332.

**1-Ethyl 2-methyl 3-hydroxy-6-(4-methoxybenzoyl)-4-pentylphthalate (5y).** Starting with **3d** (0.459 g, 1.5 mmol) and **4e** (0.456 g, 1.65 mmol), **5y** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.411 g, 64%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.75 (t,  $^3J$  = 7.2 Hz, 3 H,  $(CH_2)_4CH_3$ ), 1.14 (t,  $^3J$  = 7.3 Hz, 3 H,  $OCH_2CH_3$ ), 1.17-1.21 (m, 4 H,  $2 \times CH_2$ ), 1.43-1.51 (m, 2 H,  $CH_2$ ), 2.54 (t,  $^3J$  = 6.9 Hz, 2 H,  $ArCH_2(CH_2)_3CH_3$ ), 3.75 (s, 3 H,  $OCH_3$ ), 3.80 (s, 3 H,  $OCH_3$ ), 4.08 (q,  $^3J$  = 7.3 Hz, 2 H,  $OCH_2CH_3$ ), 6.79-6.84 (m, 2 H,  $CH_{PhOMe}$ ), 7.30 (s, 1 H,  $CH_{Ar}$ ), 7.60-7.65 (m, 2 H,  $CH_{PhOMe}$ ), 11.21 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.9, 14.0, ( $CH_3$ ), 22.4, 28.7, 29.7, 31.6 ( $CH_2$ ), 52.9, 55.5 ( $OCH_3$ ), 61.7 ( $OCH_2$ ), 110.1 ( $CCOOMe$ ), 113.6 ( $2 \times CH_{PhOMe}$ ), 128.8, 130.0, 132.3 ( $C_{Ar}$ ), 132.4 ( $2 \times CH_{PhOMe}$ ), 134.5 ( $CCOOEt$ ), 135.5 ( $CH_{Ar}$ ), 161.3 ( $C_{Ar}$ ), 163.6 (COH), 167.8, 169.9, 193.9 (CO). IR (neat,  $cm^{-1}$ ):  $\nu$  = 2955 (w), 2930 (w), 2858 (w), 1732 (m), 1659 (m), 1598 (m), 1573 (w), 1510 (w), 1440 (m), 1418 (m), 1351 (m), 1254 (s), 1242 (s), 1165 (s), 1057 (m), 1027 (m), 961 (w), 846 (m), 814 (m), 775 (w), 706 (w), 586 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 428 ( $[M]^+$ , 27), 383 (12), 350 (97), 340 (14), 326 (60), 294 (100), 239 (6), 203 (9), 147 (6), 135 (56), 92 (7), 77 (11), 41 (5). HRMS (EI): Calcd. for  $C_{24}H_{28}O_7$  ( $[M]^+$ ): 428.18295; found: 428.182763.

**1-Ethyl 2-methyl 4-hexyl-3-hydroxy-6-(4-methoxybenzoyl)phthalate (5z).** Starting with **3d** (0.459 g, 1.5 mmol) and **4f** (0.568 g, 1.65 mmol), **5z** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.404 g, 61%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 0.80 (t,  $^3J$  = 6.3 Hz, 3 H,  $(CH_2)_5CH_3$ ), 1.19 (t,  $^3J$  = 7.2 Hz, 3 H,  $OCH_2CH_3$ ), 1.21-1.28 (m, 6 H,  $3 \times CH_2$ ), 1.48-1.52 (m, 2 H,  $CH_2$ ), 2.59 (t,  $^3J$  = 7.5 Hz, 2 H,

$CH_2(CH_2)_4CH_3$ ), 3.80 (s, 3 H,  $OCH_3$ ), 3.85 (s, 3 H,  $OCH_3$ ), 4.12 (q,  $^3J = 7.2$  Hz, 2 H,  $OCH_2CH_3$ ), 6.85-6.87 (m, 2 H,  $CH_{PhOMe}$ ), 7.34 (s, 1 H,  $CH_{Ar}$ ), 7.66-7.69 (m, 2 H,  $CH_{PhOMe}$ ), 11.26 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta = 13.8, 14.0$  ( $CH_3$ ), 22.5, 28.9, 29.0, 29.7, 31.6 ( $CH_2$ ), 52.9, 55.4 ( $OCH_3$ ), 61.6 ( $OCH_2$ ), 110.1 ( $CCOOMe$ ), 113.5 ( $2 \times CH_{PhOMe}$ ), 128.7, 130.3, 132.1 ( $C_{Ar}$ ), 132.4 ( $2 \times CH_{PhOMe}$ ), 134.4 ( $C_{Ar}$ ), 135.4 ( $CH_{Ar}$ ), 161.2 ( $C_{Ar}$ ), 163.5 ( $COH$ ), 167.8, 169.8, 194.8 (CO). IR (neat,  $cm^{-1}$ ):  $\nu = 2954$  (w), 2926 (w), 2855 (w), 1732 (m), 1659 (m), 1609 (w), 1439 (m), 1351 (m), 1252 (s), 1240 (s), 1221 (m), 1164 (s), 1097 (w), 1058 (m), 1026 (m), 960 (w), 845 (w), 775 (w), 726 (w), 606 (w), 529 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 442 ( $[M]^+$ , 27), 397 (15), 365 (27), 364 (100), 342 (12), 340 (13), 336 (19), 327 (11), 326 (64), 307 (12), 295 (17), 294 (83), 280 (11), 279 (40), 135 (39), 94 (42), 55 (16). HRMS (ESI): Calcd. for  $C_{25}H_{31}O_7$  ( $[M+H]^+$ ): 443.20643; found: 443.20632.

**1-Ethyl 2-methyl 3-hydroxy-6-(4-methoxybenzoyl)-4-nonylphthalate (5aa).**

Starting with **3d** (0.459 g, 1.5 mmol) and **4h** (0.638 g, 1.65 mmol), **5aa** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.457 g, 63%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta = 0.79$  (t,  $^3J = 7.1$  Hz, 3 H,  $(CH_2)_8CH_3$ ), 1.19 (t,  $^3J = 7.5$  Hz, 3 H,  $OCH_2CH_3$ ), 1.21-1.26 (m, 12 H,  $6 \times CH_2$ ), 1.45-1.52 (m, 2 H,  $CH_2$ ), 2.58 (t,  $^3J = 7.5$  Hz, 2 H,  $ArCH_2(CH_2)_7CH_3$ ), 3.80 (s, 3 H,  $OCH_3$ ), 3.85 (s, 3 H,  $OCH_3$ ), 4.13 (q,  $^3J = 7.1$  Hz, 2 H,  $OCH_2CH_3$ ), 6.83-6.88 (m, 2 H,  $CH_{PhOMe}$ ), 7.34 (s, 1 H,  $CH_{Ar}$ ), 7.65-7.70 (m, 2 H,  $CH_{PhOMe}$ ), 11.26 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta = 13.9, 14.1$  ( $CH_3$ ), 22.6, 29.0, 29.2, 29.3, 29.4, 29.5, 29.8, 31.9 ( $CH_2$ ), 52.9, 55.5 ( $OCH_3$ ), 61.7 ( $OCH_2$ ), 110.1 ( $CCOOMe$ ), 113.6 ( $2 \times CH_{PhOMe}$ ), 128.8, 130.0, 132.3 ( $C_{Ar}$ ), 132.5 ( $2 \times CH_{PhOMe}$ ), 134.5 ( $CCOOEt$ ), 135.5 ( $CH_{Ar}$ ), 161.3 ( $C_{Ar}$ ), 163.6 ( $COH$ ), 167.8, 169.9, 193.8 (CO). IR (neat,  $cm^{-1}$ ):  $\nu = 2954$  (w), 2924 (m), 2853 (w), 2255 (w), 1732 (m), 1672 (m), 1659 (m), 1598 (m), 1574 (w), 1509 (w), 1440 (m), 1418 (m), 1348 (m), 1306 (m), 1253 (s), 1240 (s), 1201 (m), 1164 (s), 1111 (w), 1058 (m), 1028 (m), 961 (w), 907 (m), 867 (w), 845 (m), 727 (s), 647 (m), 606 (m), 586 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 484 ( $[M]^+$ , 23), 438 (26), 406 (100), 384 (19), 340 (15), 326 (80), 307 (14), 294 (83), 279 (35), 268 (17), 239 (17), 203 (7), 135 (34), 77 (4), 43 (7). HRMS (EI): Calcd. for  $C_{28}H_{36}O_7$  ( $[M]^+$ ): 484.24555; found: 484.245787.

**1-Ethyl 2-methyl 4-decyl-3-hydroxy-6-(4-methoxybenzoyl)phthalate (5ab).**

Starting with **3d** (0.459 g, 1.5 mmol) and **4i** (0.661 g, 1.65 mmol), **5ab** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.477 g, 64%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta = 0.81$  (t,  $^3J = 7.2$  Hz, 3 H,  $(CH_2)_9CH_3$ ), 1.15 (t,  $^3J = 8.9$  Hz, 3 H,  $OCH_2CH_3$ ), 1.17-1.22 (m, 14 H,  $7 \times CH_2$ ), 1.45-1.52 (m, 2 H,  $CH_2$ ), 2.58 (t,  $^3J = 7.5$  Hz, 2 H,  $CH_2(CH_2)_8CH_3$ ), 3.80 (s, 3 H,  $OCH_3$ ), 3.85 (s, 3 H,  $OCH_3$ ), 4.15 (q,  $^3J = 7.5$  Hz, 2 H,  $OCH_2CH_3$ ), 6.84-6.87 (m, 2 H,  $CH_{PhOMe}$ ), 7.34 (s, 1 H,  $CH_{Ar}$ ), 7.66-7.69 (m, 2 H,  $CH_{PhOMe}$ ), 11.26 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta = 13.9, 14.0$  ( $CH_3$ ), 22.6, 29.0, 29.3, 29.3, 29.4, 29.4, 29.5, 29.7, 31.8 ( $CH_2$ ), 52.9, 55.4 ( $OCH_3$ ), 61.6 ( $OCH_2$ ), 110.1 ( $CCOOMe$ ), 113.5 ( $2 \times CH_{PhOMe}$ ), 128.8, 129.9, 132.2 ( $C_{Ar}$ ), 132.4 ( $2 \times CH_{PhOMe}$ ), 134.4 ( $C_{Ar}$ ), 135.4 ( $CH_{Ar}$ ), 161.2 ( $C_{Ar}$ ), 163.5 ( $COH$ ), 167.8, 169.8, 193.8 (CO). IR (neat,  $cm^{-1}$ ):  $\nu = 2954$  (w), 2923 (w), 2853 (w), 1732 (m), 1673 (m), 1658 (w), 1598 (m), 1509 (m), 1440 (m), 1417 (m), 1306 (m), 1253 (s), 1240 (s), 1164 (s), 1057 (m), 1027 (m), 907 (m), 844 (m), 812 (m), 727 (s), 605 (w), 585 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 499 ( $[M+H]^+$ , 4.3), 498 (13), 453 (20), 452 (31), 421 (19), 420 (100), 392 (15), 340 (15), 327 (15), 326 (93), 307 (14), 295 (23), 294 (97),

280 (11), 279 (41), 268 (23), 135 (80), 77 (13), 55 (16), 44 (13), 43 (35), 41 (24). HRMS (ESI): Calcd. for  $C_{29}H_{39}O_7$  ( $[M+H]^+$ ): 499.26903; found: 499.26915.

**2-Ethyl 1-methyl 3-(4-bromobenzoyl)-6-hydroxyphthalate (5ac).** Starting with **3e** (0.532 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5ac** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.383 g, 63%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.23 (t,  $^3J$  = 7.5 Hz, 3 H,  $OCH_2CH_3$ ), 3.87 (s, 3 H,  $OCH_3$ ), 4.19 (q,  $^3J$  = 7.5 Hz, 2 H,  $OCH_2CH_3$ ), 7.0 (d,  $^3J$  = 9.0 Hz, 1 H,  $CH_{Ar}$ ), 7.50 (d,  $^3J$  = 8.5 Hz, 1 H,  $CH_{Ar}$ ), 7.54 ( $S_{br}$ , 4 H,  $CH_{PhBr}$ ), 11.56 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.9 ( $CH_3$ ), 53.1 ( $OCH_3$ ), 62.0 ( $OCH_2$ ), 110.9 ( $CCOOCH_3$ ), 118.2 ( $CH_{Ar}$ ), 128.2, 130.7 ( $C_{Ar}$ ), 131.4 ( $2\times CH_{PhBr}$ ), 131.7 ( $2\times CH_{PhBr}$ ), 135.8 ( $CCOOEt$ ), 136.1 ( $CH_{Ar}$ ), 137.6 ( $C_{Ar}$ ), 163.7 ( $COH$ ), 167.4, 169.2, 193.7 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu_{max}$  = 3085 (w), 2980 (w), 2955 (w), 2902 (w), 1729 (m), 1663 (s), 1583 (s), 1441 (m), 1394 (m), 1325 (m), 1248 (s), 1217 (s), 1174 (m), 1146 (m), 1118 (m), 1068 (m), 1029 (s), 959 (m), 937 (m), 838 (m), 811 (m), 762 (m), 731 (m), 679 (m), 630 (m), 583 (m), 533 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 408 ( $[M^+]$ ,  $^{81}Br$ , 57), 406 ( $[M^+]$ ,  $^{79}Br$ , 58), 376 (25), 374 (24), 363 (27), 362 (47), 361 (28), 360 (42), 335 (44), 333 (30), 331 (31), 330 (30), 329 (26), 308 (20), 305 (16), 304 (98), 303 (55), 302 (100), 301 (43), 280 (13), 276 (15), 275 (14), 274 (17), 273 (11), 251 (34), 250 (20), 249 (11), 238 (16), 223 (31), 222 (30), 219 (11), 217 (11), 194 (20), 191 (42), 185 (41), 183 (44), 167 (10), 157 (20), 155 (22), 139 (20), 138 (17), 119 (22). HRMS (EI): Calcd. for  $C_{18}H_{15}O_6Br$  ( $[M]^+$ ,  $^{79}Br$ ): 406.00465; found: 406.003513.

**1-Ethyl 2-methyl 6-(4-bromobenzoyl)-3-hydroxy-4-methylphthalate (5ad).** Starting with **3e** (0.532 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5ad** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.415 g, 66%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.20 (t,  $^3J$  = 7.6 Hz, 3 H,  $OCH_2CH_3$ ), 2.21 (s, 3 H,  $CH_3$ ), 3.86 (s, 3 H,  $OCH_3$ ), 4.14 (q,  $^3J$  = 7.5 Hz, 2 H,  $OCH_2CH_3$ ), 7.34 (s, 1 H,  $CH_{Ar}$ ), 7.54 ( $S_{br}$ , 4 H,  $CH_{PhBr}$ ), 11.36 (s, 1 H, OH).  $^{13}C$  NMR ( $CDCl_3$ , 75 MHz):  $\delta$  = 13.9, 15.9 ( $CH_3$ ), 53.0 ( $OCH_3$ ), 61.8 ( $OCH_2$ ), 110.0 ( $CCOOCH_3$ ), 127.0, 128.0 ( $C_{Ar}$ ), 131.4 ( $2\times CH_{PhBr}$ ), 131.6 ( $2\times CH_{PhBr}$ ), 134.2 ( $C_{Ar}$ ), 135.0 ( $CCOOEt$ ), 136.0 ( $C_{Ar}$ ), 136.1 ( $CH_{Ar}$ ), 162.0 ( $COH$ ), 167.5, 169.7, 194.1 ( $CO$ ). IR (neat,  $cm^{-1}$ ):  $\nu_{max}$  = 3056 (w), 2980 (w), 2955 (w), 1731 (m), 1663 (s), 1583 (m), 1439 (m), 1395 (m), 1337 (m), 1231 (s), 1199 (m), 1166 (s), 1110 (w), 1095 (w), 1069 (m), 1053 (s), 1008 (m), 958 (m), 910 (m), 865 (w), 873 (m), 810 (m), 765 (m), 731 (m), 679 (m), 635 (m), 579 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 422 ( $[M^+]$ ,  $^{81}Br$ , 51), 420 ( $[M^+]$ ,  $^{79}Br$ , 61), 391 (12), 390 (44), 389 (10), 388 (43), 377 (29), 376 (56), 375 (30), 374 (52), 346 (17), 345 (34), 343 (31), 342 (65), 319 (14), 318 (93), 317 (21), 316 (100), 290 (14), 288 (17), 265 (22), 264 (25), 252 (20), 237 (20), 235 (11), 205 (26), 185 (41), 183 (42), 181 (14), 180 (14), 157 (25), 155 (25), 152 (36), 151 (15), 133 (23), 76 (12). HRMS (EI): Calcd. for  $C_{19}H_{17}O_6Br$  ( $[M]^+$ ,  $^{79}Br$ ): 420.02030; found: 420.019964.

**Diethyl 6-(4-bromobenzoyl)-4-ethyl-3-hydroxyphthalate (5ae).** Starting with **3e** (0.532 g, 1.5 mmol) and **4c** (0.499 g, 1.65 mmol), **5ae** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.436 g, 65%).  $^1H$  NMR (300 MHz,  $CDCl_3$ ):  $\delta$  = 1.10 (t,  $^3J$  = 7.6 Hz, 3 H,  $CH_2CH_3$ ), 1.15 (t,  $^3J$  = 7.1 Hz, 3 H,  $OCH_2CH_3$ ), 1.29 (t,  $^3J$  = 7.2 Hz, 3 H,  $OCH_2CH_3$ ), 2.63 (q,  $^3J$  = 7.5 Hz, 2 H,  $CH_2CH_3$ ), 4.12 (q,  $^3J$  = 7.4 Hz, 2

H,  $\text{OCH}_2\text{CH}_3$ ), 4.32 (q,  $^3J = 7.5$  Hz, 2 H,  $\text{OCH}_2\text{CH}_3$ ), 7.33 (s, 1 H,  $\text{CH}_{\text{Ar}}$ ), 7.54 ( $\text{S}_{\text{br}}$ , 4 H,  $\text{CH}_{\text{PhBr}}$ ), 11.50 (s, 1 H, OH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta = 13.3, 13.7, 13.8$  ( $\text{CH}_3$ ), 22.9 ( $\text{CH}_2$ ), 61.7, 62.7 ( $\text{OCH}_2$ ), 110.2 ( $\text{CCOOEt}$ ), 127.0, 128.0, ( $\text{C}_{\text{Ar}}$ ), 131.4 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 131.6 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 133.7 ( $\text{C}_{\text{Ar}}$ ), 134.6 ( $\text{CH}_{\text{Ar}}$ ), 134.8, 136.0 ( $\text{C}_{\text{Ar}}$ ), 161.8 ( $\text{COH}$ ), 167.6, 169.4, 194.1 (CO). IR (neat,  $\text{cm}^{-1}$ ):  $\nu = 3056$  (w), 2977 (w), 2934 (w), 2873 (w), 1731 (m), 1662 (s), 1583 (m), 1442 (w), 1421 (m), 1394 (m), 1374 (m), 1337 (m), 1300 (m), 1227 (s), 1172 (s), 1095 (m), 1068 (m), 1052 (m), 1009 (m), 907 (m), 844 (m), 815 (m), 730 (m), 687 (m), 620 (m), 579 (m). GC-MS (EI, 70 eV):  $m/z$  (%) = 450 ( $[\text{M}^+]$ ,  $^{81}\text{Br}$ , 36), 448 ( $[\text{M}^+]$ ,  $^{79}\text{Br}$ , 38), 405 (18), 404 (18), 403 (19), 402 (18), 359 (30), 358 (100), 357 (33), 356 (95), 335 (10), 334 (10), 331 (19), 330 (89), 329 (23), 328 (84), 305 (11), 294 (10), 288 (13), 278 (25), 277 (70), 274 (11), 272 (11), 261 (10), 259 (11), 253 (10), 251 (11), 250 (10), 249 (21), 221 (12), 219 (17), 185 (71), 184 (14), 183 (71), 165 (28), 158 (33), 157 (27), 155 (27), 153 (10), 152 (14), 147 (19), 141 (13), 140 (12), 139 (16), 135 (12), 130 (23), 119 (10), 115 (22), 111 (10), 105 (10), 97 (14), 95 (11), 91 (12), 85 (11), 84 (14), 83 (15), 81 (12), 77 (15), 76 (13), 75 (13), 73 (20), 71 (52), 70 (13), 69 (25), 67 (11), 57 (23), 55 (24), 45 (12), 43 (87), 42 (10), 41 (29). HRMS (EI): Calcd. for  $\text{C}_{21}\text{H}_{21}\text{O}_6\text{Br}$  ( $[\text{M}]^+$ ,  $^{79}\text{Br}$ ): 448.05160; found: 448.051626.

**1-Ethyl 2-methyl 6-(4-bromobenzoyl)-3-hydroxy-4-pentylphthalate (5af).** Starting with **3e** (0.532 g, 1.5 mmol) and **4e** (0.546 g, 1.65 mmol), **5af** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.465 g, 65%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.80$  (t,  $^3J = 7.1$  Hz, 3 H,  $(\text{CH}_2)_4\text{CH}_3$ ), 1.15 (t,  $^3J = 6.9$  Hz, 3 H,  $\text{OCH}_2\text{CH}_3$ ), 1.18-1.23 (m, 4 H,  $2 \times \text{CH}_2$ ), 1.41-1.49 (m, 2 H,  $\text{CH}_2$ ), 2.53 (t,  $^3J = 7.2$  Hz, 2 H,  $\text{CH}_2(\text{CH}_2)_3\text{CH}_3$ ), 3.81 (s, 3 H,  $\text{OCH}_3$ ), 4.10 (q,  $^3J = 7.6$  Hz, 2 H,  $\text{OCH}_2\text{CH}_3$ ), 7.26 (s, 1 H,  $\text{CH}_{\text{Ar}}$ ), 7.48 ( $\text{S}_{\text{br}}$ , 4 H,  $\text{CH}_{\text{PhBr}}$ ), 11.30 (s, 1 H, OH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta = 13.9, 14.0$  ( $\text{CH}_3$ ), 22.4, 28.7, 29.7, 31.5 ( $\text{CH}_2$ ), 53.0 ( $\text{OCH}_3$ ), 61.8 ( $\text{OCH}_2$ ), 110.3 ( $\text{CCOOCH}_3$ ), 127.6, 128.1 ( $\text{C}_{\text{Ar}}$ ), 131.4 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 131.7 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 132.5 ( $\text{C}_{\text{Ar}}$ ), 135.0 ( $\text{CCOOEt}$ ), 135.6 ( $\text{CH}_{\text{Ar}}$ ), 136.0 ( $\text{C}_{\text{Ar}}$ ), 161.8 ( $\text{COH}$ ), 167.6, 169.7, 194.0 (CO). IR (neat,  $\text{cm}^{-1}$ ):  $\nu = 2955$  (w), 2929 (w), 2858 (w), 1731 (m), 1668 (m), 1585 (w), 1440 (m), 1348 (m), 1256 (s), 1174 (m), 1070 (w), 1055 (m), 1010 (m), 907 (m), 842 (w), 814 (w), 730 (m), 648 (w), 584 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 478 ( $[\text{M}^+]$ ,  $^{81}\text{Br}$ , 16), 476 ( $[\text{M}^+]$ ,  $^{79}\text{Br}$ , 16), 433 (14), 431 (13), 401 (27), 400 (100), 399 (29), 398 (94), 390 (12), 388 (13), 376 (25), 374 (26), 372 (37), 370 (35), 345 (12), 344 (45), 343 (21), 342 (45), 341 (13), 329 (20), 328 (11), 319 (21), 316 (12), 264 (14), 261 (14), 235 (11), 185 (22), 183 (22), 155 (15), 152 (16), 151 (10). HRMS (EI): Calcd. for  $\text{C}_{23}\text{H}_{25}\text{O}_6\text{Br}$  ( $[\text{M}]^+$ ,  $^{79}\text{Br}$ ): 476.08290; found: 476.082300.

**1-Ethyl 2-methyl 6-(4-bromobenzoyl)-4-hexyl-3-hydroxyphthalate (5ag).** Starting with **3e** (0.532 g, 1.5 mmol) and **4f** (0.569 g, 1.65 mmol), **5ag** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.516 g, 70%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.81$  (t,  $^3J = 7.1$  Hz, 3 H,  $(\text{CH}_2)_5\text{CH}_3$ ), 1.21 (t,  $^3J = 7.3$  Hz, 3 H,  $\text{OCH}_2\text{CH}_3$ ), 1.23-1.27 (m, 6 H,  $3 \times \text{CH}_2$ ), 1.45-1.52 (m, 2 H,  $\text{CH}_2$ ), 2.58 (t,  $^3J = 6.7$  Hz, 2 H,  $\text{ArCH}_2(\text{CH}_2)_4\text{CH}_3$ ), 3.86 (s, 3 H,  $\text{OCH}_3$ ), 4.15 (q,  $^3J = 7.5$  Hz, 2 H,  $\text{OCH}_2\text{CH}_3$ ), 7.32 (s, 1 H,  $\text{CH}_{\text{Ar}}$ ), 7.54 ( $\text{S}_{\text{br}}$ , 4 H,  $\text{CH}_{\text{PhBr}}$ ), 11.37 (s, 1 H, OH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta = 13.9, 14.0$  ( $\text{CH}_3$ ), 22.6, 29.0, 29.1, 29.8, 31.6 ( $\text{CH}_2$ ), 53.0 ( $\text{OCH}_3$ ), 61.8 ( $\text{OCH}_2$ ), 110.3 ( $\text{CCOOCH}_3$ ), 127.6, 128.1 ( $\text{C}_{\text{Ar}}$ ), 131.5 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 131.7 ( $2 \times \text{CH}_{\text{PhBr}}$ ), 132.6 ( $\text{C}_{\text{Ar}}$ ), 135.0 ( $\text{CCOOEt}$ ), 135.6 ( $\text{CH}_{\text{Ar}}$ ), 136.1 ( $\text{C}_{\text{Ar}}$ ), 161.9 ( $\text{COH}$ ), 167.7, 169.8, 194.1 (CO). IR (neat,  $\text{cm}^{-1}$ ):  $\nu = 2955$  (w), 2928 (w), 2856 (w), 1736 (w), 1672 (w), 1586 (w), 1441 (w), 1396 (w),

1350 (w), 1258 (w), 1235 (w), 1175 (w), 1058 (w), 962 (w), 909 (w), 732 (w), 649 (w), 586 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 492 ([M<sup>+</sup>], <sup>81</sup>Br, 21), 490 ([M<sup>+</sup>], <sup>79</sup>Br, 20), 445 (11), 414 (100), 376 (19), 355 (5), 344 (32), 263 (14), 235 (6), 200 (13), 183 (53), 155 (14), 129 (12), 116 (23), 57 (29), 43 (40). HRMS (EI): Calcd. for C<sub>24</sub>H<sub>27</sub>O<sub>6</sub>Br ([M]<sup>+</sup>, <sup>79</sup>Br): 490.09855; found: 490.098727.

**1-Ethyl 2-methyl 6-(4-bromobenzoyl)-3-hydroxy-4-nonylphthalate (5ah).** Starting with **3e** (0.532 g, 1.5 mmol) and **4h** (0.614 g, 1.65 mmol), **5ah** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.543 g, 68%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.80 (t, <sup>3</sup>J = 7.2 Hz, 3 H, (CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>J = 8.7 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.21-1.25 (m, 12 H, 6×CH<sub>2</sub>), 1.46-1.51 (m, 2 H, CH<sub>2</sub>), 2.58 (t, <sup>3</sup>J = 7.0 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.14 (q, <sup>3</sup>J = 7.6 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.31 (s, 1 H, CH<sub>Ar</sub>), 7.53 (s<sub>br</sub>, 4 H, CH<sub>PhBr</sub>), 11.36 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 13.9, 14.1 (CH<sub>3</sub>), 22.6, 29.0, 29.2, 29.3, 29.4, 29.5, 29.7, 31.8 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.8 (OCH<sub>2</sub>), 110.3 (CCOOCH<sub>3</sub>), 127.6, 128.1 (C<sub>Ar</sub>), 131.4 (2×CH<sub>PhBr</sub>), 131.7 (2×CH<sub>PhBr</sub>), 132.0 (C<sub>Ar</sub>), 135.0 (CCOOEt), 135.6 (CH<sub>Ar</sub>), 136.0 (C<sub>Ar</sub>), 161.9 (COH), 167.7, 169.8, 194.3 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 3085 (w), 2980 (w), 2955 (w), IR (KBr, cm<sup>-1</sup>): ν̄ = 2953 (w), 2924 (w), 2853 (w), 1734 (m), 1670 (m), 1585 (w), 1440 (m), 1349 (m), 1256 (s), 1174 (m), 1070 (w), 1010 (m), 961 (m), 842 (w), 814 (w), 733 (m), 648 (w), 584 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 534 ([M<sup>+</sup>], <sup>81</sup>Br, 26), 532 ([M<sup>+</sup>], <sup>79</sup>Br, 26), 489 (17), 488 (19), 487 (19), 486 (16), 457 (37), 456 (100), 454 (97), 390 (19), 388 (19), 376 (38), 375 (18), 374 (35), 345 (12), 344 (50), 343 (16), 342 (49), 330 (10), 329 (23), 327(19), 317 (11), 316 (34), 264 (12), 263 (19), 185 (24), 183 (24), 43 (16), 41 (13). HRMS (EI): Calcd. for C<sub>27</sub>H<sub>34</sub>O<sub>6</sub><sup>79</sup>Br ([M+H]<sup>+</sup>): 533.15333; found: 533.15235.

**1-Ethyl 2-methyl 6-(4-bromobenzoyl)-4-decyl-3-hydroxyphthalate (5ai).** Starting with **3e** (0.532 g, 1.5 mmol) and **4i** (0.661 g, 1.65 mmol), **5ai** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.567 g, 69%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.80 (t, <sup>3</sup>J = 7.3 Hz, 3 H, (CH<sub>2</sub>)<sub>9</sub>CH<sub>3</sub>), 1.18 (t, <sup>3</sup>J = 8.9 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.20-1.25 (m, 14 H, 7×CH<sub>2</sub>), 1.44-1.52 (m, 2 H, CH<sub>2</sub>), 2.58 (t, <sup>3</sup>J = 8.0 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 3.86 (s, 3 H, OCH<sub>3</sub>), 4.15 (q, <sup>3</sup>J = 7.3 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.32 (s, 1 H, CH<sub>Ar</sub>), 7.54 (s<sub>br</sub>, 4 H, CH<sub>PhBr</sub>), 11.36 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 12.9, 13.1 (CH<sub>3</sub>), 21.7, 28.0, 28.3, 28.4, 28.5, 28.6, 28.7, 28.8, 30.9 (CH<sub>2</sub>), 52.1 (OCH<sub>3</sub>), 60.8 (OCH<sub>2</sub>), 109.3 (CCOOCH<sub>3</sub>), 126.6, 127.1 (C<sub>Ar</sub>), 130.5 (2×CH<sub>PhBr</sub>), 130.7 (2×CH<sub>PhBr</sub>), 131.5, 134.0 (C<sub>Ar</sub>), 134.6 (CH<sub>Ar</sub>), 135.0 (CCOOEt), 160.9 (COH), 166.7, 168.8, 193.0 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2955 (w), 2926 (w), 2854 (w), 1734 (w), 1670 (w), 1586 (w), 1559 (w), 1507 (w), 1457 (w), 1438 (w), 1419 (w), 1351 (w), 1258 (w), 1238 (w), 1174 (w), 1070 (w), 1057 (w), 962 (w), 909 (w), 842 (w), 814 (w), 734 (w). GC-MS (EI, 70 eV):  $m/z$  (%) = 548 ([M<sup>+</sup>], <sup>81</sup>Br, 24), 546 ([M<sup>+</sup>], <sup>79</sup>Br, 23), 502 (25), 487 (8), 470 (100), 442 (7), 390 (24), 376 (40), 361 (8), 344 (51), 316 (38), 263 (22), 235 (9), 183 (21), 152 (8), 43 (20). HRMS (EI): Calcd. for C<sub>28</sub>H<sub>35</sub>O<sub>6</sub>Br ([M]<sup>+</sup>, <sup>79</sup>Br) : 546.16115; found: 546.162377.

**1,2-Diethyl 3-methyl 4-hydroxybenzene-1,2,3-tricarboxylate (5aj).** Starting with **3f** (0.366 g, 1.5 mmol) and **4a** (0.429 g, 1.65 mmol), **5aj** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.302 g, 68%), mp. 70-72 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.29 (t, <sup>3</sup>J = 7.2 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.33 (t, <sup>3</sup>J = 7.4 Hz, 3 H,



OCH<sub>2</sub>CH<sub>3</sub>), 3.88 (s, 3 H, OCH<sub>3</sub>), 4.26 (q, <sup>3</sup>J = 7.1 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.35 (q, <sup>3</sup>J = 7.2 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.00 (d, <sup>3</sup>J = 9.0 Hz, 1 H, CH<sub>Ar</sub>), 8.07 (d, <sup>3</sup>J = 9.0 Hz, 1 H, CH<sub>Ar</sub>), 11.54 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 14.2 (CH<sub>3</sub>), 53.1 (OCH<sub>3</sub>), 61.4, 61.8 (OCH<sub>2</sub>), 109.7 (CCOOCH<sub>3</sub>), 118.7 (CH<sub>Ar</sub>), 119.6 (C<sub>Ar</sub>), 137.0 (CH<sub>Ar</sub>), 138.7 (CCOOC<sub>2</sub>H<sub>5</sub>), 164.3 (COH), 165.1, 167.8, 169.4 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 3078 (w), 2983 (w), 2966 (w), 2940 (w), 2873 (w), 1729 (m), 1714 (s), 1682 (m), 1585 (m), 1443 (m), 1385 (w), 1346 (m), 1329 (m), 1304 (m), 1239 (s), 1202 (s), 1175 (m), 1148 (s), 1110 (m), 1024 (s), 983 (m), 938 (m), 867 (m), 855 (m), 811 (m), 752 (m), 708 (s), 648 (m), 632 (m), 562 (m). GC-MS (EI, 70 eV): m/z (%) = 296 ([M]<sup>+</sup>, 29), 264 (14), 250 (55), 222 (11), 208 (13), 191 (100), 164 (27), 148 (16), 119 (43), 92 (10). HRMS (ESI): Calcd. for C<sub>14</sub>H<sub>16</sub>O<sub>6</sub>Na ([M+Na]<sup>+</sup>): 319.07882; found: 319.0787.

### 1,2-Diethyl 3-methyl 4-hydroxy-5-methylbenzene-1,2,3-tricarboxylate (5ak).

Starting with **3f** (0.366 g, 1.5 mmol) and **4b** (0.457 g, 1.65 mmol), **5ak** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.321 g, 69%), mp. 73 – 75 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 1.29 (t, <sup>3</sup>J = 7.2 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 1.32 (t, <sup>3</sup>J = 7.2 Hz, 3 H, OCH<sub>2</sub>CH<sub>3</sub>), 2.22 (s, 3 H, CH<sub>3</sub>), 3.87 (s, 3 H, OCH<sub>3</sub>), 4.25 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.33 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.93 (s, 1 H, CH<sub>Ar</sub>), 11.81 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 14.2, 15.8 (CH<sub>3</sub>), 53.0 (OCH<sub>3</sub>), 61.3, 61.7 (OCH<sub>2</sub>), 108.9 (CCOOCH<sub>3</sub>), 118.7, 128.2 (C<sub>Ar</sub>), 136.3 (CCOOEt), 137.1 (CH<sub>Ar</sub>), 163.6 (COH), 164.7, 168.1, 169.9 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2984 (w), 2964 (w), 2936 (w), 2873 (w), 1753 (m), 1716 (m), 1662 (m), 1434 (m), 1416 (m), 1381 (m), 1367 (m), 1339 (m), 1271 (m), 1221 (s), 1194 (m), 1151 (m), 1097 (m), 1030 (m), 981 (m), 913 (m), 882 (w), 864 (w), 812 (m), 771 (m), 759 (m), 699 (m), 671 (m), 626 (m), 573 (m). GC-MS (EI, 70 eV): m/z (%) = 310 ([M]<sup>+</sup>, 19), 278 (14), 264 (39), 236 (8), 204 (100), 178 (11), 162 (7), 133 (21), 105 (8), 77 (5). HRMS (EI): Calcd. for C<sub>15</sub>H<sub>18</sub>O<sub>7</sub> ([M]<sup>+</sup>): 310.10470; found: 310.104607.

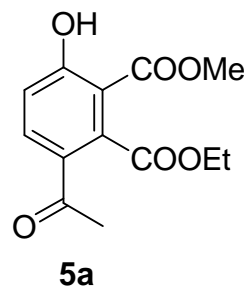
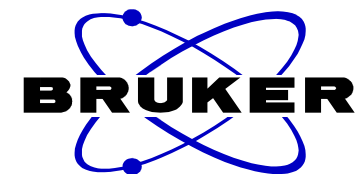
### 1,2-Diethyl 3-methyl 4-hydroxy-5-pentylbenzene-1,2,3-tricarboxylate (5al).

Starting with **3f** (0.366 g, 1.5 mmol) and **4e** (0.546 g, 1.65 mmol), **5al** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish solid (0.379 g, 69%), mp. 76 – 78 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.78 (t, <sup>3</sup>J = 7.3 Hz, 3 H, (CH<sub>2</sub>)<sub>4</sub>CH<sub>3</sub>), 1.18 – 1.29 (m, 10 H, 2×OCH<sub>2</sub>CH<sub>3</sub>, 2×CH<sub>2</sub>), 1.44 – 1.54 (m, 2 H, CH<sub>2</sub>), 2.55 (t, <sup>3</sup>J = 7.7 Hz, 2 H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>), 3.81 (s, 3 H, OCH<sub>3</sub>), 4.20 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 4.28 (q, <sup>3</sup>J = 7.4 Hz, 2 H, OCH<sub>2</sub>CH<sub>3</sub>), 7.85 (s, 1 H, CH<sub>Ar</sub>), 11.75 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.0, 14.1, 14.2 (CH<sub>3</sub>), 22.4, 28.7, 29.7, 31.5 (CH<sub>2</sub>), 53.0 (OCH<sub>3</sub>), 61.3, 61.7 (OCH<sub>2</sub>), 109.1 (CCOOCH<sub>3</sub>), 118.8, 132.8 (C<sub>Ar</sub>), 136.2 (CCOOEt), 136.4 (CH<sub>Ar</sub>), 163.4 (COH), 164.8, 168.2, 169.9 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2956 (w), 2930 (w), 2859 (w), 1739 (w), 1721 (m), 1671 (m), 1578 (w), 1440 (m), 1391 (w), 1368 (m), 1346 (m), 1251 (s), 1220 (s), 1197 (s), 1150 (2), 1111 (m), 1095 (m), 1044 (m), 922 (w), 894 (w), 865 (w), 815 (m), 768 (m), 731 (m), 700 (m), 630 (w), 590 (w), 534 (w). GC-MS (EI, 70 eV): m/z (%) = 366 ([M]<sup>+</sup>, 13), 321 (27), 291 (24), 278 (38), 264 (100), 232 (17), 217 (31), 204 (65), 189 (16), 131 (11), 105 (5). HRMS (ESI): Calcd. for C<sub>19</sub>H<sub>27</sub>O<sub>6</sub> ([M+H]<sup>+</sup>): 367.17513; found: 367.17459.

### Methyl 3-decyl-2-hydroxy-9,10-dioxo-9,10-dihydroanthracene-1-carboxylate (6).

A mixture of **5n** (0.252 g, 0.54 mmol) and of concentrated sulfuric acid (6.5 mL) was

stirred at 20 °C for 1 h. The solution was poured into ice water and the mixture was extracted with dichloromethane. The combined organic layers were dried (Na<sub>2</sub>SO<sub>4</sub>), filtered and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, heptanes/EtOAc = 20:1) to give **6** as a yellowish oil (0.193 mg, 85%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.78 (t, <sup>3</sup>J = 6.6 Hz, 3 H, (CH<sub>2</sub>)<sub>9</sub>CH<sub>3</sub>), 1.14-1.18 (m, 14 H, 7×CH<sub>2</sub>), 1.45-1.50 (m, 2 H, CH<sub>2</sub>), 2.56 (t, <sup>3</sup>J = 6.8 Hz, 2 H, ArCH<sub>2</sub>(CH<sub>2</sub>)<sub>8</sub>CH<sub>3</sub>), 3.93 (s, 3 H, OCH<sub>3</sub>), 7.33-7.38 (m, 3 H, CH<sub>Ar</sub>), 7.63-7.65 (m, 2 H, CH<sub>Ar</sub>), 11.42 (s, 1 H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 14.1 (CH<sub>3</sub>), 22.6, 28.9, 29.2, 29.3, 29.3, 29.5, 29.5, 29.7, 31.9 (CH<sub>2</sub>), 53.2 (OCH<sub>3</sub>), 110.4 (CCOOMe), 127.4 (C<sub>Ar</sub>), 128.4 (2×CH<sub>Ar</sub>), 128.7, 129.8, 130.0 (CH<sub>Ar</sub>), 131.0, 132.5, 136.3, 137.0 (C<sub>Ar</sub>), 161.9 (COH), 170.9, 192.4, 194.9 (CO). IR (neat, cm<sup>-1</sup>): ν̄ = 2953 (w), 2921 (m), 2851 (m), 1707(m), 1662 (m), 1597 (w), 1575 (w), 1440 (m), 14266 (m), 1344 (m), 1271 (m), 1234 (m), 1170 (m), 1054 (m), 1026 (m), 1000 (w), 959 (m), 927 (w), 819 (m), 766 (m), 723 (m), 693 (m), 652 (m), 607 (m), 585 (m). GC-MS (EI, 70 eV): *m/z* (%) = 422 ([M]<sup>+</sup>, 100), 405 (23), 404 (44), 391 (36), 345 (11), 309 (15), 296 (28), 278 (10), 265 (16), 264 (11), 238 (21), 231 (11), 165 (20), 152 (11), 105 (64), 77 (27), 43 (10), 41 (12). HRMS (ESI): Calcd. for C<sub>26</sub>H<sub>31</sub>O<sub>5</sub> ([M+H]<sup>+</sup>): 423.2166; found: 423.21684.

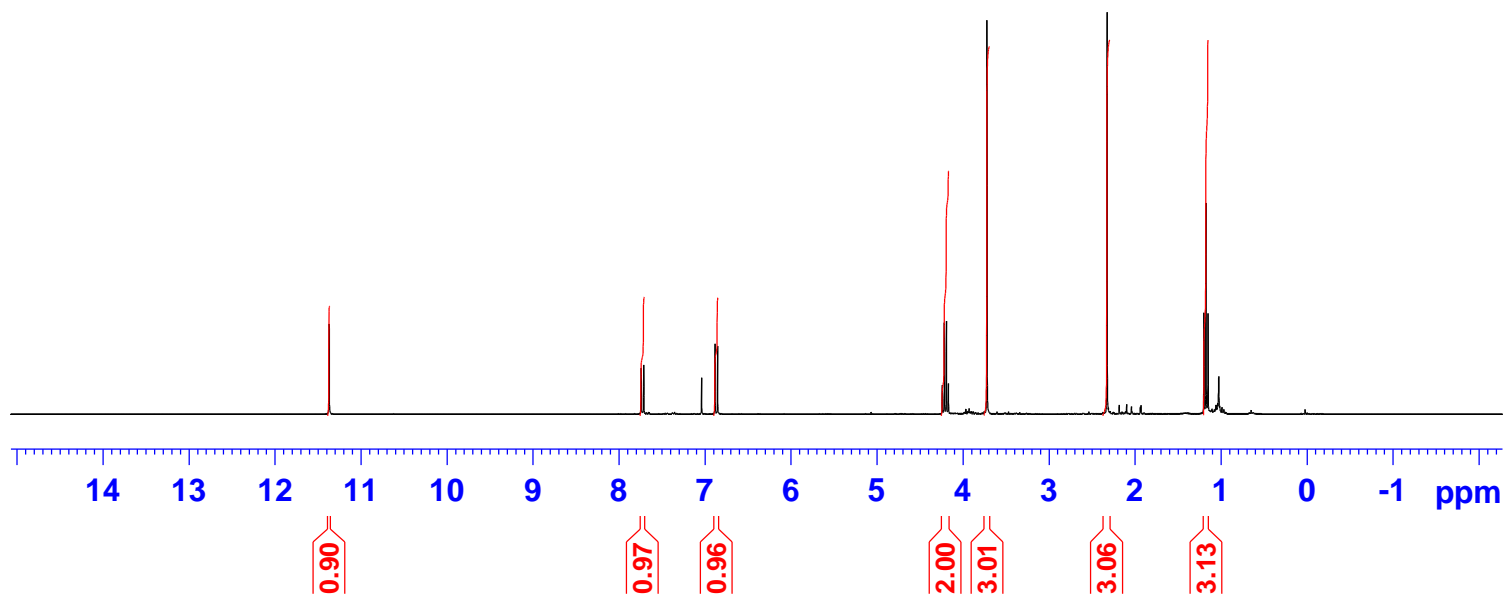


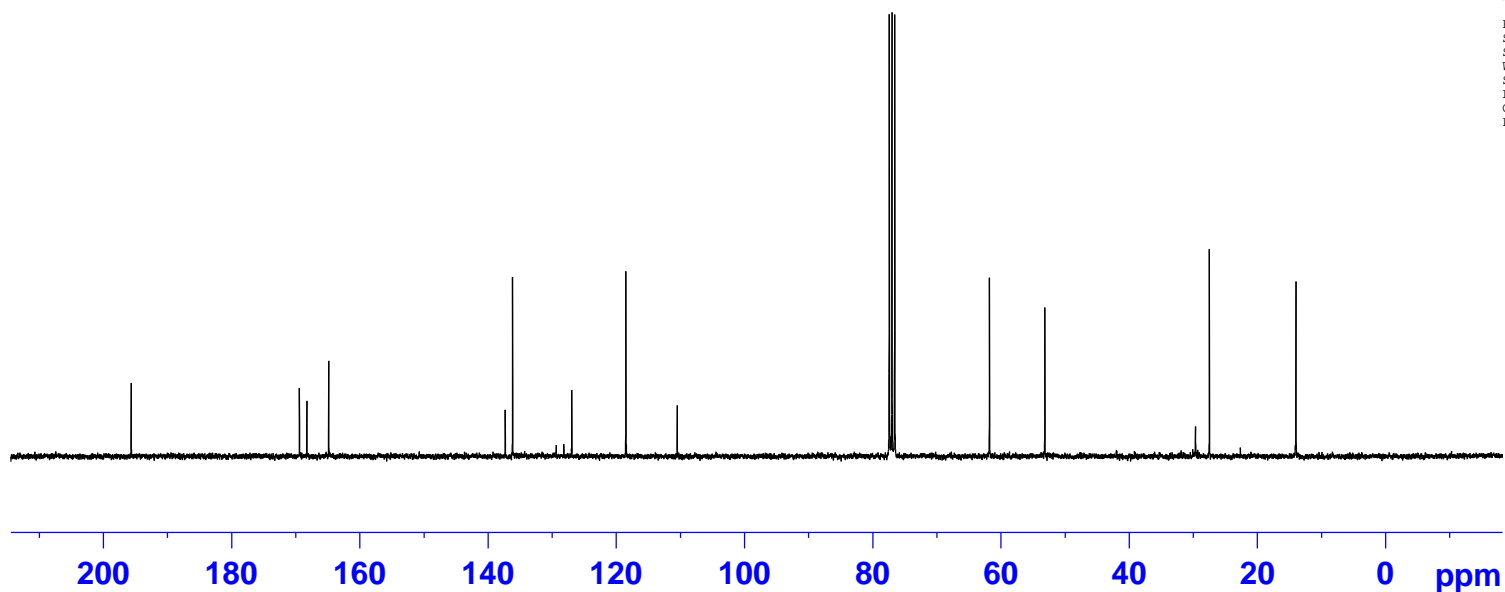
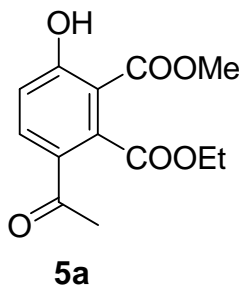
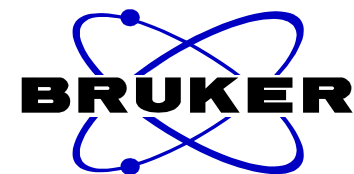
Current Data Parameters  
NAME 081128.u310  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081128  
Time 9.30  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300725 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





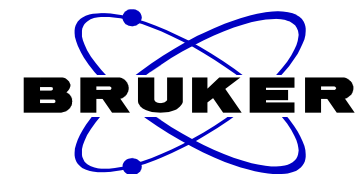
```
Current Data Parameters
NAME      081128.u333
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20081128
Time     21.27
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1024
DS       4
SWH      18028.846 Hz
FIDRES   0.275098 Hz
AQ       1.8175818 sec
RG       2050
DW       27.733 usec
DE       10.00 usec
TE       298.2 K
D1       2.0000000 sec
D11      0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -0.50 dB
PL1W     33.25691986 W
SFO1     75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    72.00 usec
PL2      0.00 dB
PL12     17.00 dB
PL13     17.00 dB
PL2W     11.25325108 W
PL12W    0.22453187 W
PL13W    0.22453187 W
SFO2     300.1312005 MHz

F2 - Processing parameters
SI       32768
SF       75.4677490 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

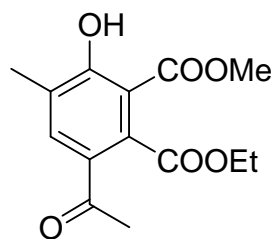


```
Current Data Parameters
NAME      081205.u313
EXPNO    10
PROCNO   1

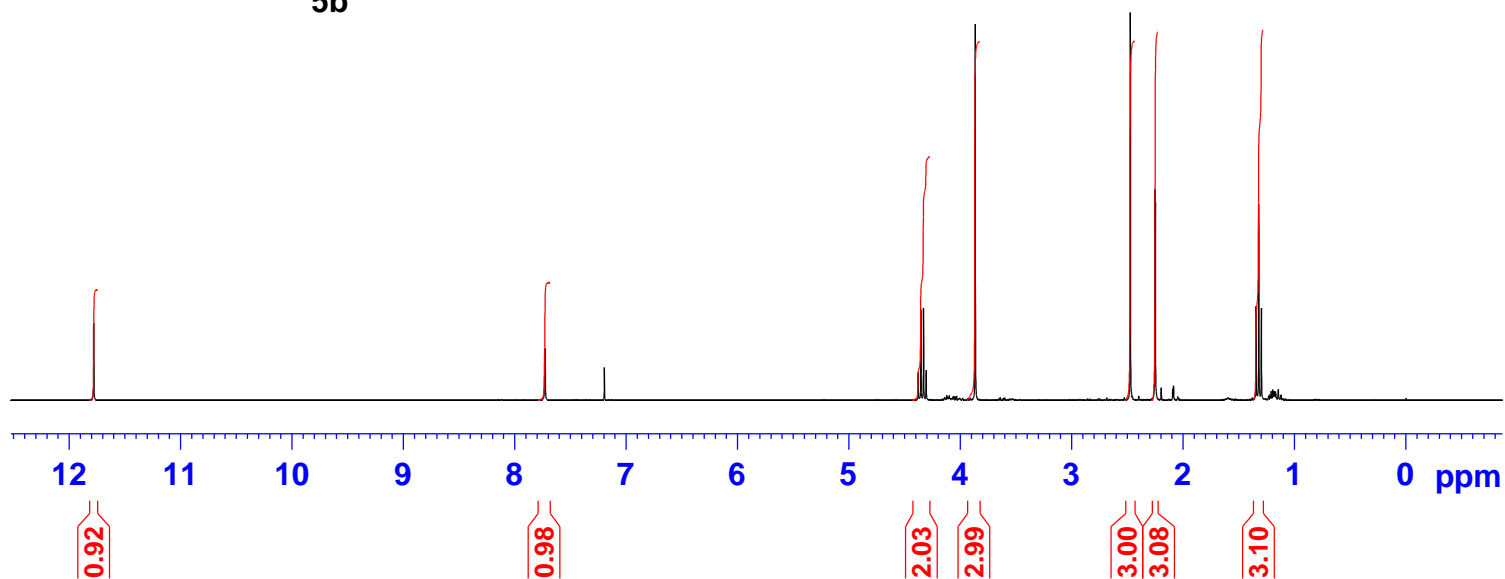
F2 - Acquisition Parameters
Date_    20081205
Time     9.05
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       101
DW       80.800 usec
DE       10.00 usec
TE       298.2 K
D1       1.00000000 sec
TD0      1

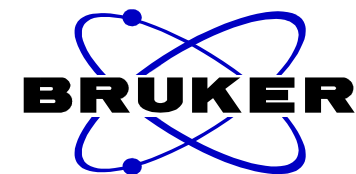
===== CHANNEL f1 =====
NUC1     1H
P1       10.00 usec
PL1      0.00 dB
PL1W     11.25325108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300259 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```



5b





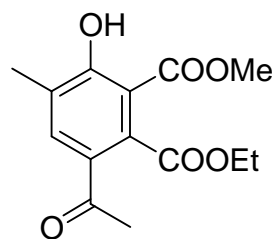
```
Current Data Parameters
NAME      081205.207
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20081205
Time     20.33
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       2048
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1845834 sec
RG       2050
DW       33.333 usec
DE       10.00 usec
TE       298.1 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999999 sec
TD0      1
```

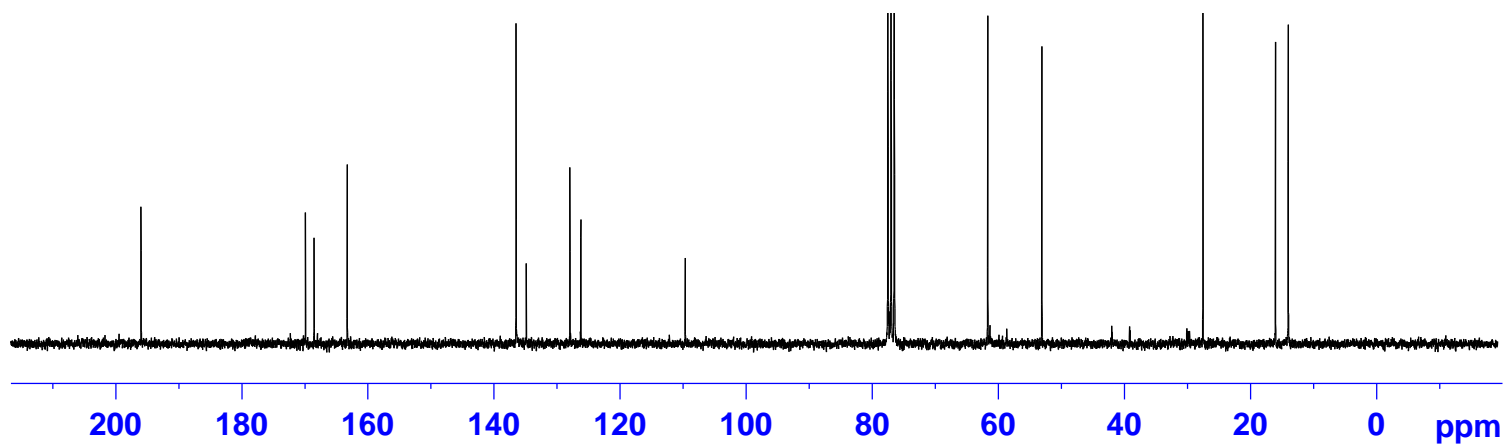
```
===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -1.00 dB
SFO1     62.9015280 MHz
```

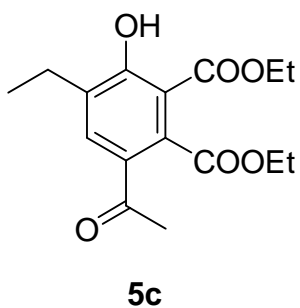
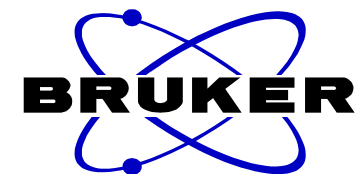
```
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     15.00 dB
PL13     15.00 dB
PL2      -2.50 dB
SFO2     250.1310005 MHz
```

```
F2 - Processing parameters
SI       32768
SF       62.8952390 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```



5b



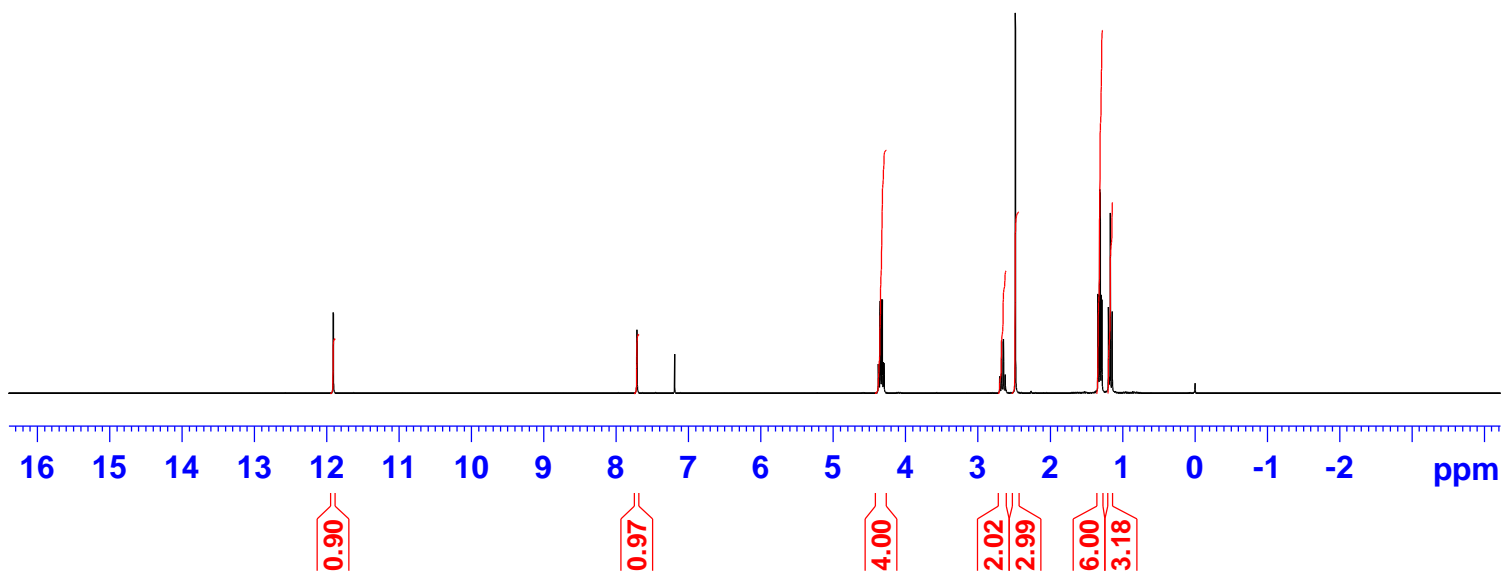


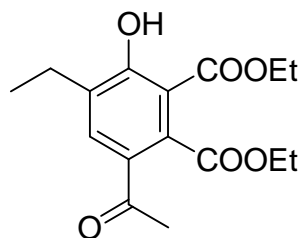
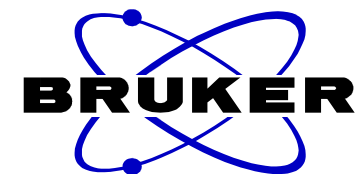
Current Data Parameters  
NAME 081211.u311  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081211  
Time 9.37  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

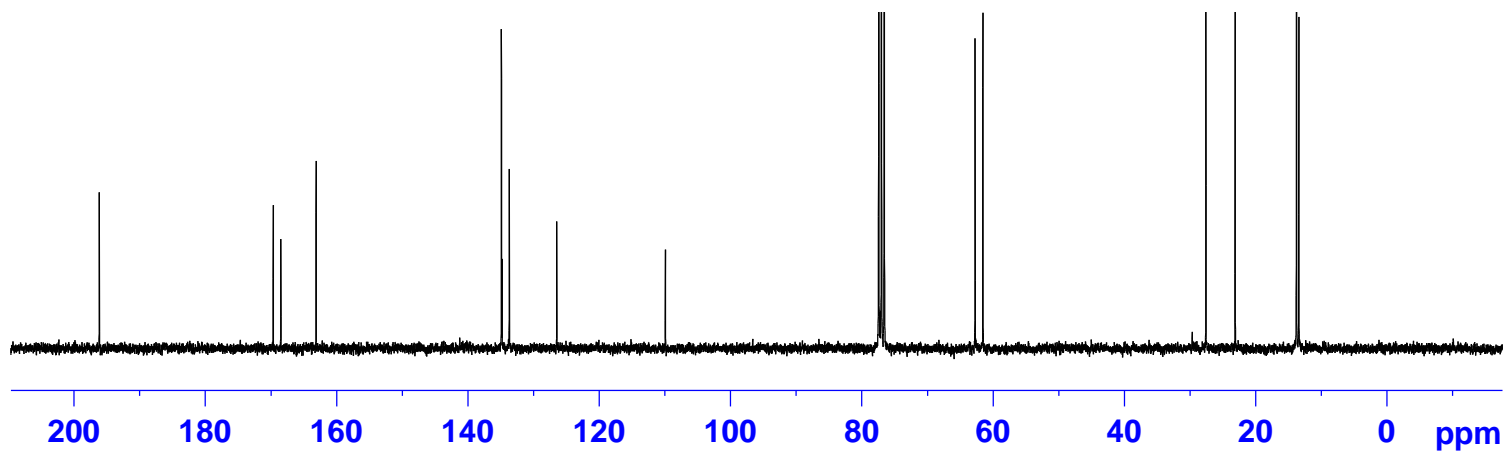
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300268 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





5c



```
Current Data Parameters
NAME      081211.u337
EXPNO    10
PROCNO   1

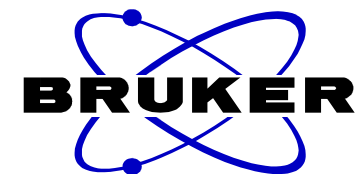
F2 - Acquisition Parameters
Date_    20081211
Time     19.19
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1024
DS       4
SWH      18028.846 Hz
FIDRES   0.275098 Hz
AQ       1.8175818 sec
RG       2050
DW       27.733 usec
DE       10.00 usec
TE       298.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -0.50 dB
PL1W     33.25691986 W
SFO1     75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    72.00 usec
PL2      0.00 dB
PL12     17.00 dB
PL13     17.00 dB
PL2W     11.25325108 W
PL12W    0.22453187 W
PL13W    0.22453187 W
SFO2     300.1312005 MHz

F2 - Processing parameters
SI       32768
SF       75.4677490 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```



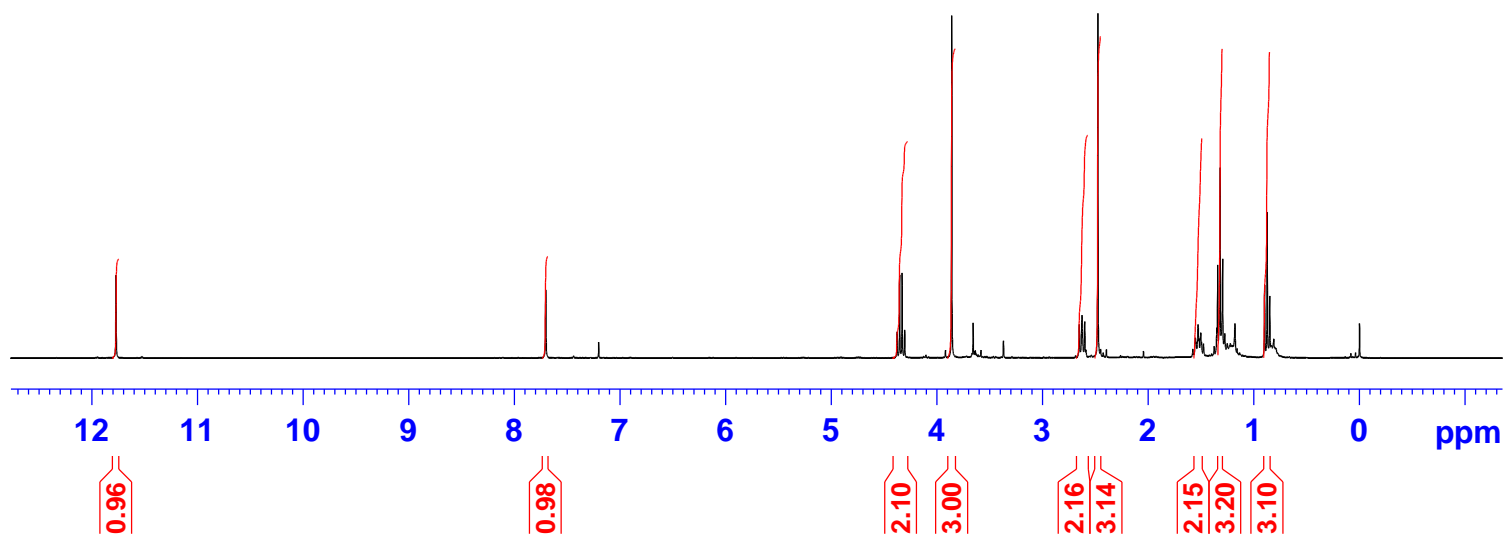
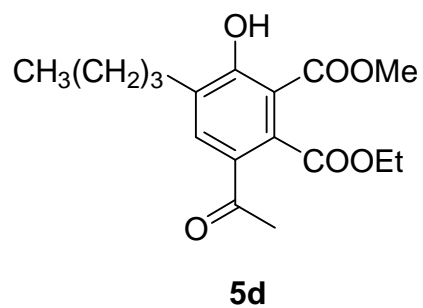


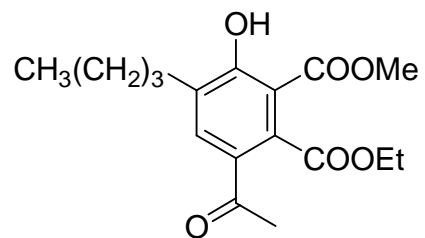
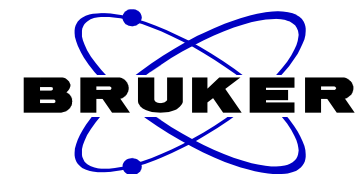
Current Data Parameters  
NAME 081211.u314  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081211  
Time 9.54  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 36  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

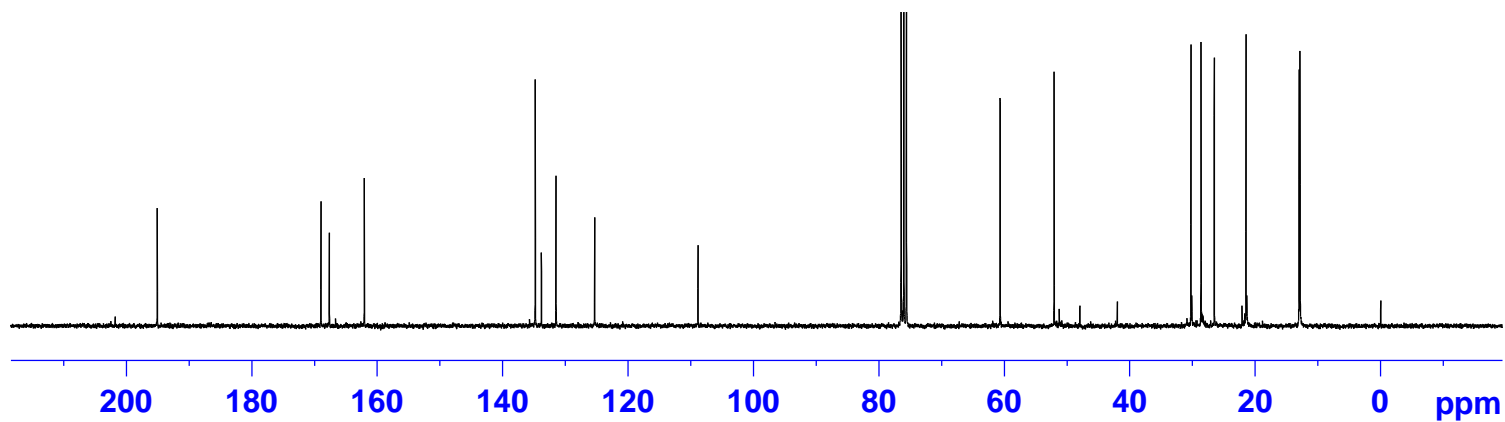
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300244 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





5d



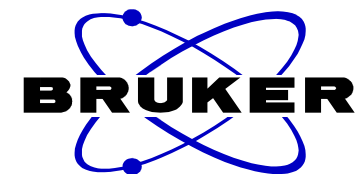
```
Current Data Parameters
NAME          081211.u330
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20081211
Time          13.02
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.25691986 W
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        72.00 usec
PL2           0.00 dB
PL12         17.00 dB
PL13         17.00 dB
PL2W         11.25325108 W
PL12W        0.22453187 W
PL13W        0.22453187 W
SFO2         300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4678247 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

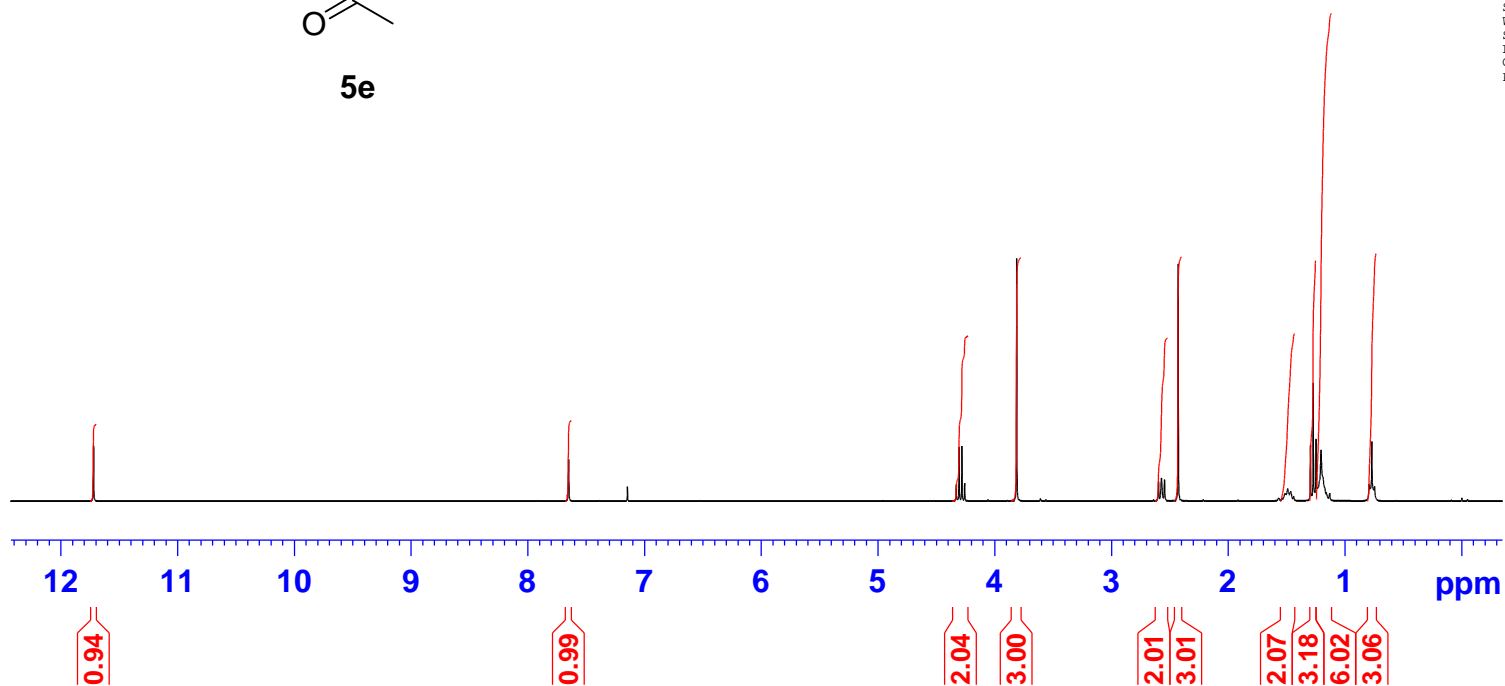
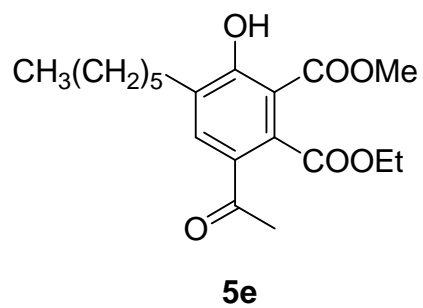


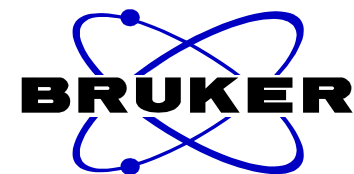
Current Data Parameters  
NAME 081217.u311  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081217  
Time 10.16  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 50.8  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300407 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





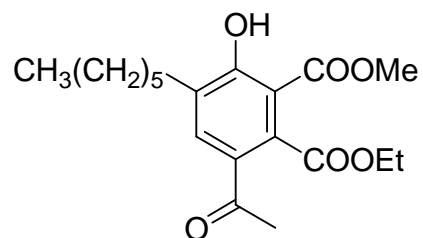
```
Current Data Parameters
NAME      081217.u327
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20081217
Time      18.19
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        1500
DS        4
SWH       18028.846 Hz
FIDRES    0.275098 Hz
AQ        1.8175818 sec
RG        2050
DW        27.733 usec
DE        10.00 usec
TE        298.2 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1

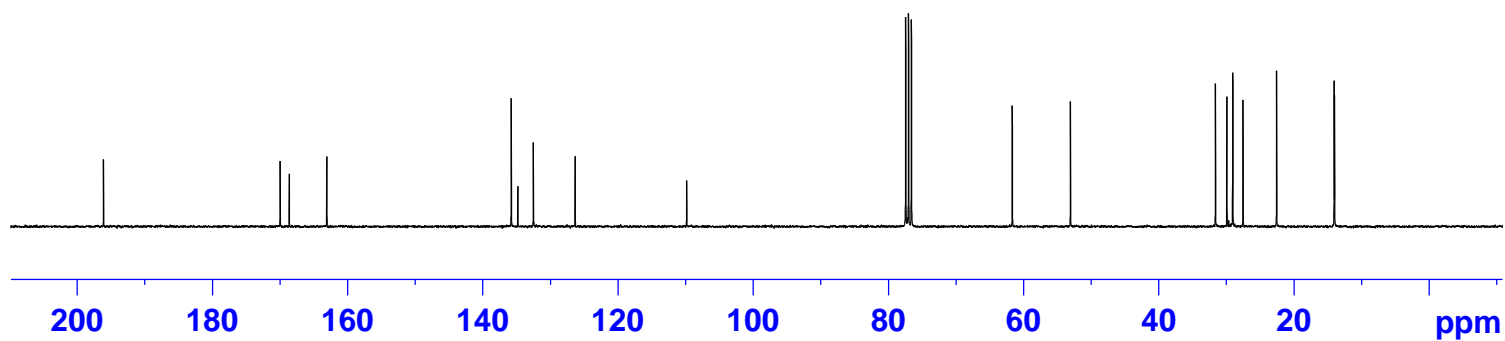
===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       -0.50 dB
PL1W      33.25691986 W
SFO1      75.4752953 MHz

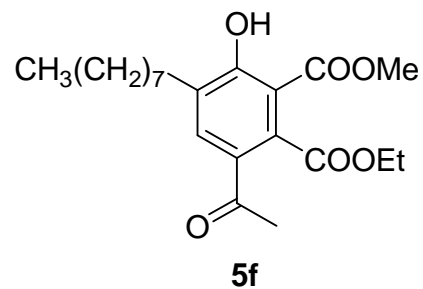
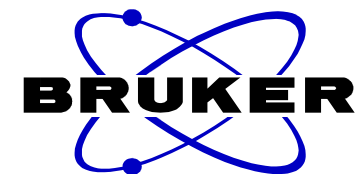
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     72.00 usec
PL2       0.00 dB
PL12      17.00 dB
PL13      17.00 dB
PL2W      11.25325108 W
PL12W     0.22453187 W
PL13W     0.22453187 W
SFO2      300.1312005 MHz

F2 - Processing parameters
SI        32768
SF        75.4677490 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```



5e



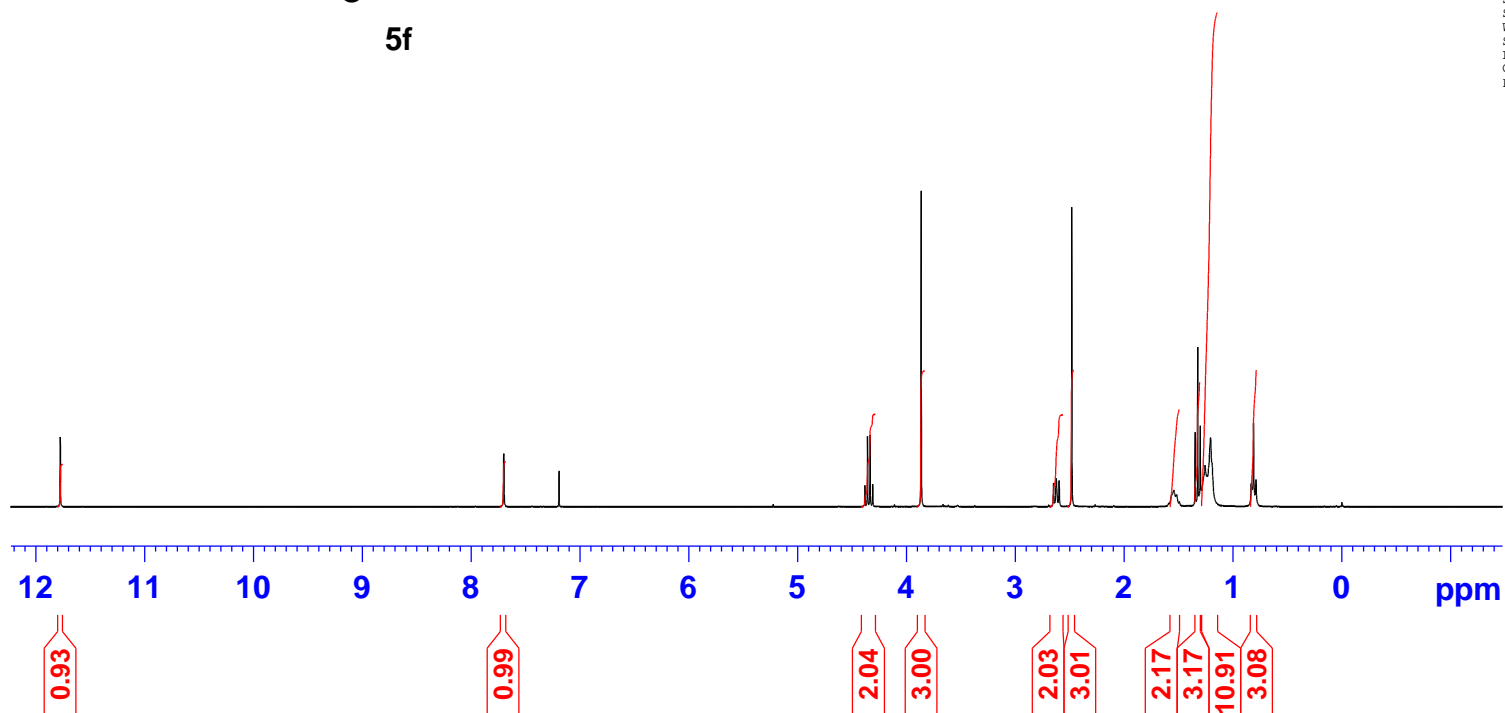


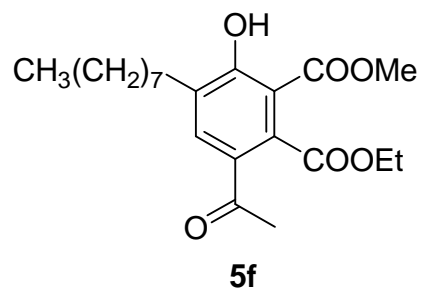
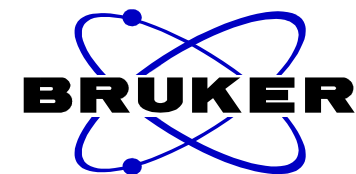
Current Data Parameters  
NAME 081205.u319  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081205  
Time 9.40  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 80.6  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
DL 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300270 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





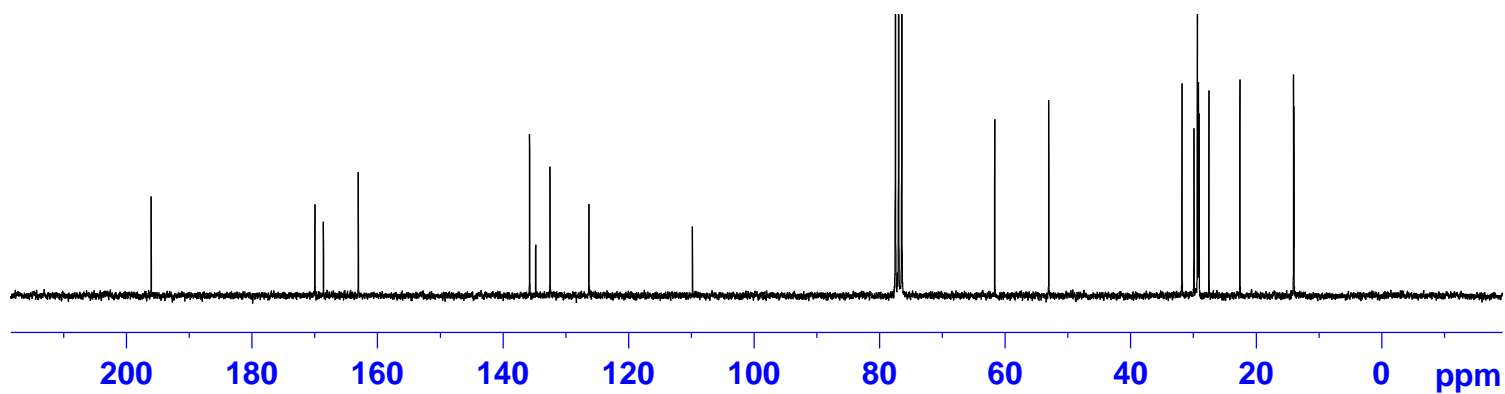
```
Current Data Parameters
NAME      081205.211
EXPNO    10
PROCNO   1

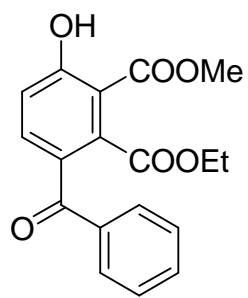
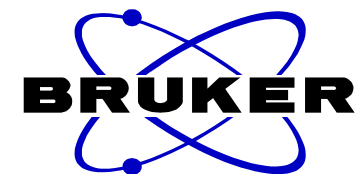
F2 - Acquisition Parameters
Date_    20081206
Time     5.30
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       2048
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1845834 sec
RG       2050
DW       33.333 usec
DE       10.00 usec
TE       298.1 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999999 sec
TD0      1
```

```
===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -1.00 dB
SFO1     62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     15.00 dB
PL13     15.00 dB
PL2      -2.50 dB
SFO2     250.1310005 MHz
```

```
F2 - Processing parameters
SI       32768
SF       62.8952390 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```





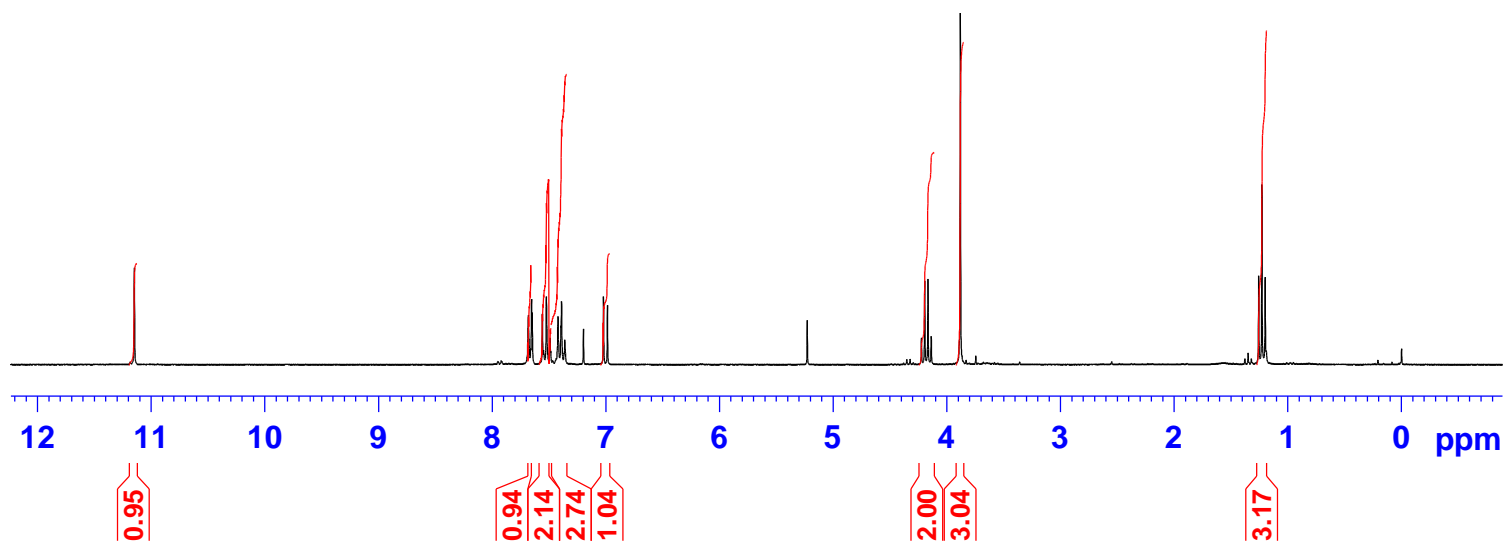
5g

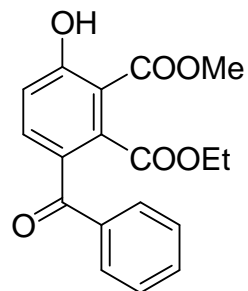
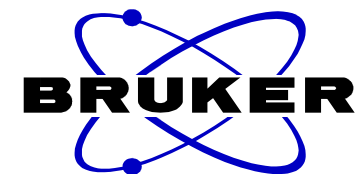
```
Current Data Parameters
NAME      081212.207
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20081212
Time      9.15
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       5165.289 Hz
FIDRES    0.078816 Hz
AQ        6.3439350 sec
RG        406
DW        96.800 usec
DE        10.00 usec
TE        298.3 K
D1        1.00000000 sec
TD0       1

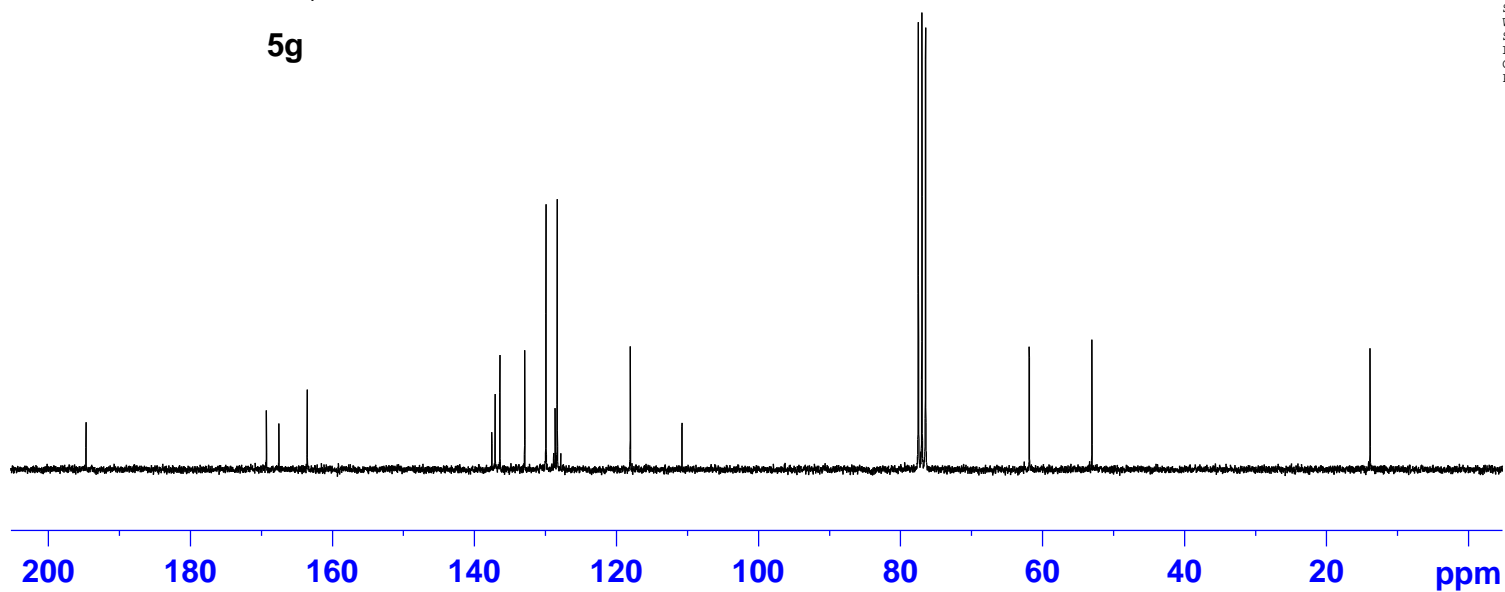
===== CHANNEL f1 =====
NUC1      1H
P1        10.00 usec
PL1       -2.50 dB
SFO1     250.1315447 MHz

F2 - Processing parameters
SI        32768
SF        250.1300162 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
```





5g



Current Data Parameters  
NAME 081212.214  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20081212  
Time 17.51  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 298.2 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





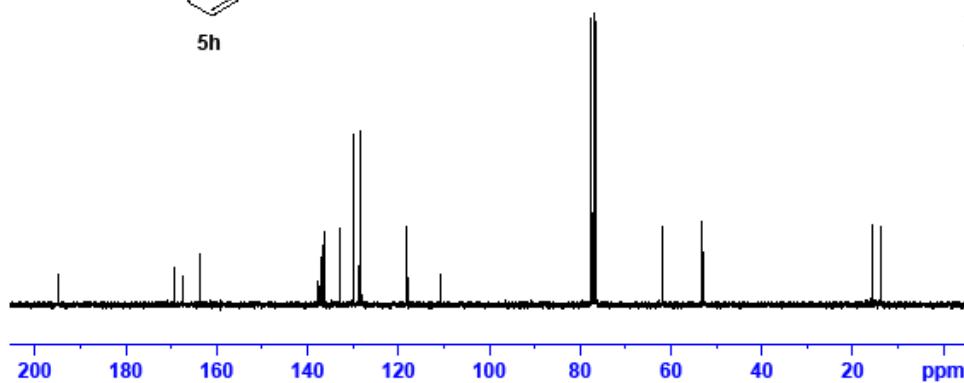
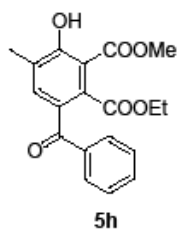
```
Current data parameters
NAME          081211_024
EXPNO        10
PROCNO       1

F1 - Acquisition parameters
Date_         20081212
Time         17.51
INSTRUM      spect
PROBHD      1 HX WARRD 5B-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           1024
DS           4
SWH          13000.000 MHz
FIDRES       0.118892 MHz
AQ           1.1164824 sec
RG           3280
SQ           23.133 UHRG
OR          10.00 UHRG
TE           299.2 K
SI           3.0000000 MHz
SFO          50.0000000 MHz
DELTA        1.8999999 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1            13.00 UHRG
PL1           -1.00 dB
SFO1          62.5012690 MHz

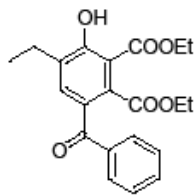
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          13C
P2            10.00 UHRG
PL2           18.00 dB
PL12          18.00 dB
PL13          18.00 dB
PL14          18.00 dB
SFO2          250.1310008 MHz

F1 - processing parameters
SI            32768
SF            62.5012690 MHz
MCH           63
AQ            0
CA            1.00 MHz
GB            0
PC            1.60
```

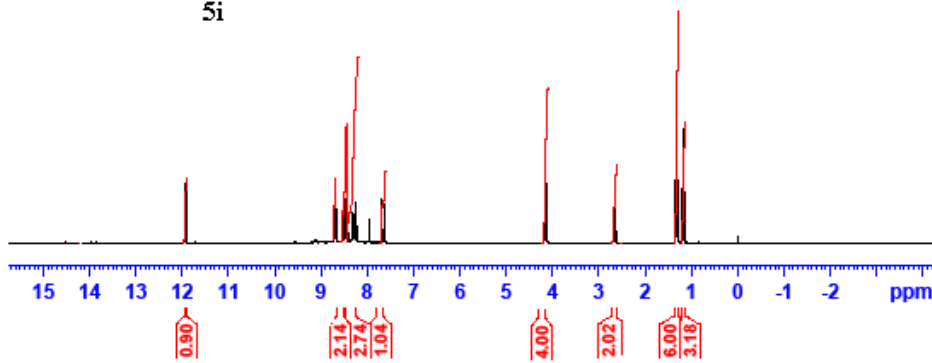


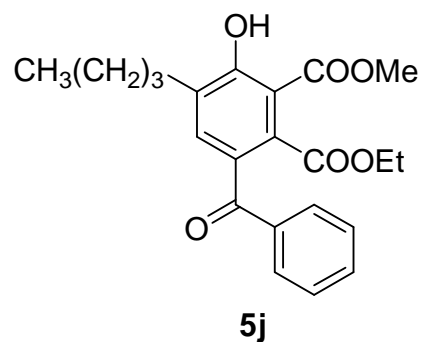
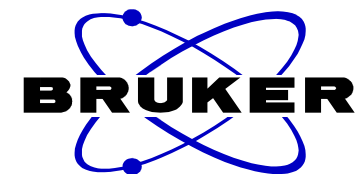


```
Current data parameters
NAME      082211.0811
EXPNO    10
PROCNO   1
-----
F2 - Acquisition parameters
Date_    20081211
Time     9.37
DIRNAME  NMR1
PROCNO   1
PULPROG  zgpg30
TD       65536
AQ       0.0211
RG       327.5
NA       1
DS       1
SWH      6199.119 Hz
FIDRES   0.000423 Hz
AQ       0.2593887 sec
RG       327.5
NA       1
DS       1
SWH      61.950 UHz
DE       10.00 UHz
TE       300.2 K
D1       1.00000000 sec
SFO      500
----- CHANNEL f1 -----
NUC1     13
P1       10.00 UHz
PC1      0.00 dB
PL1      11.0000000 Hz
SFO1     125.7613636 MHz
-----
F2 - processing parameters
SI       32768
SF       500.136098 MHz
RG       327.5
NA       0
DS       0.50 Hz
GB       0
PC       1.00
```



5i



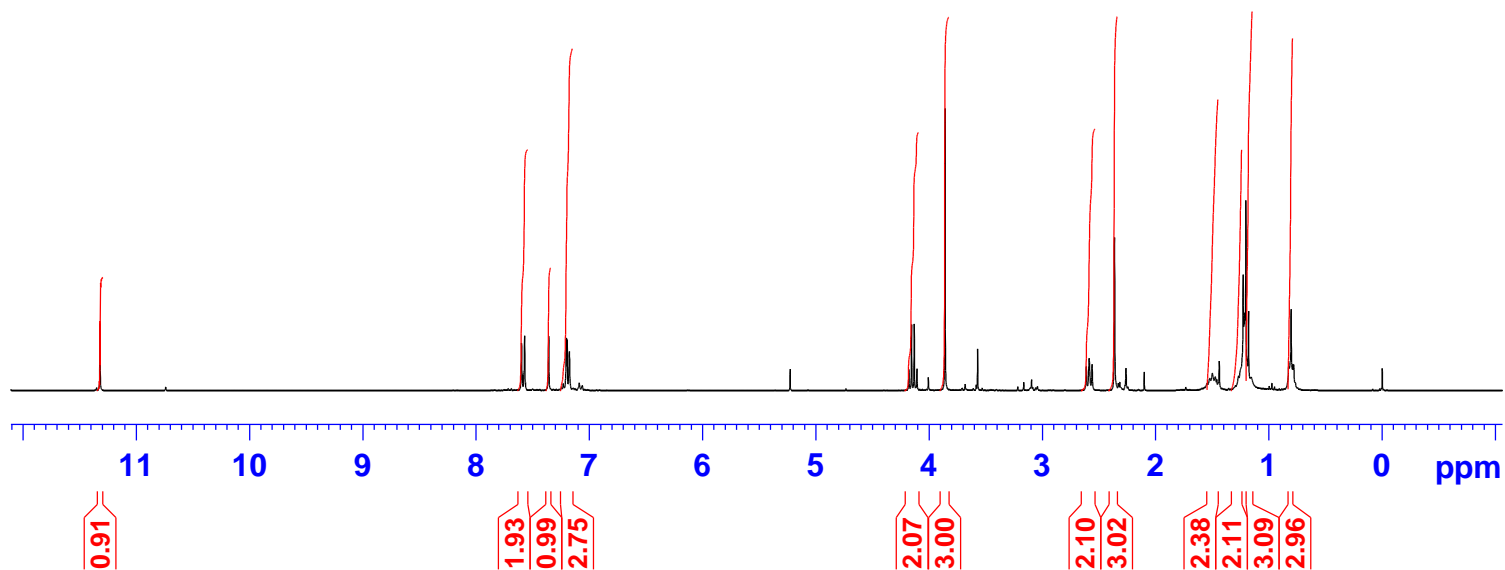


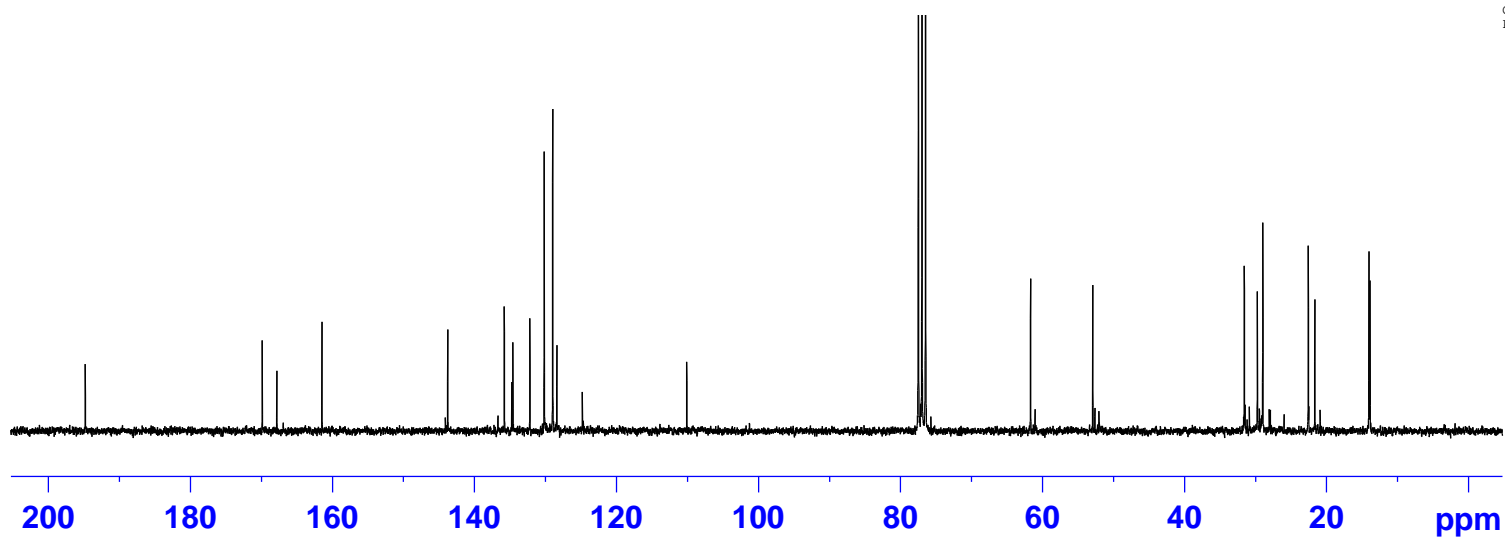
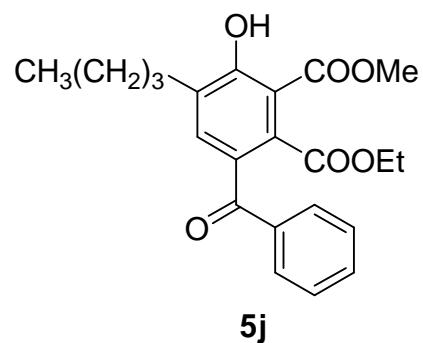
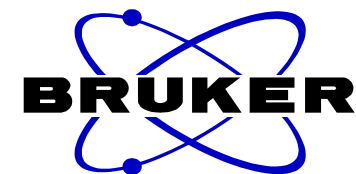
Current Data Parameters  
NAME 090116.u324  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090116  
Time 10.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 90.5  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300263 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





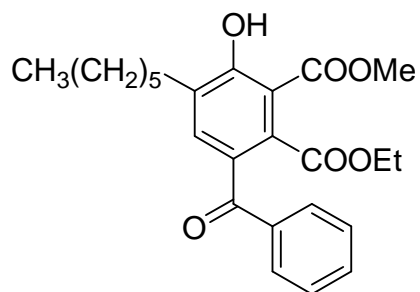
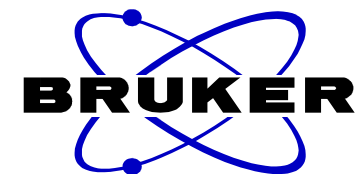
```
Current Data Parameters
NAME          090116.206
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090117
Time          16.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            1820
DW            33.333 usec
DE            10.00 usec
TE            297.9 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

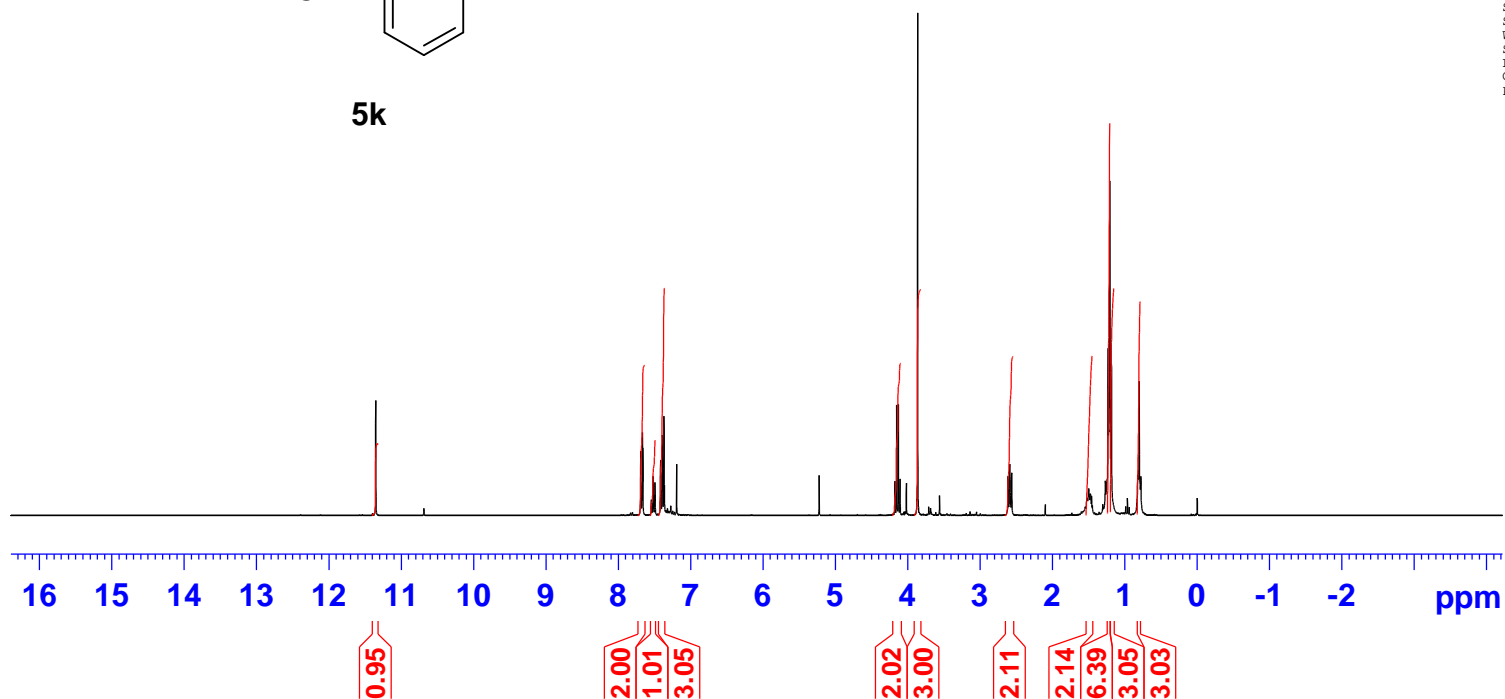
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1         62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         15.00 dB
PL13         15.00 dB
PL2          -2.50 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



5k

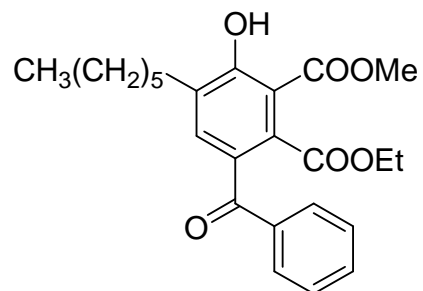
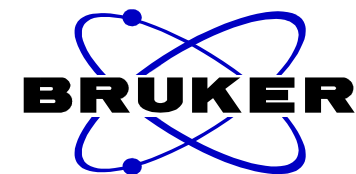


Current Data Parameters  
NAME 090116.u321  
EXPNO 10  
PROCNO 1

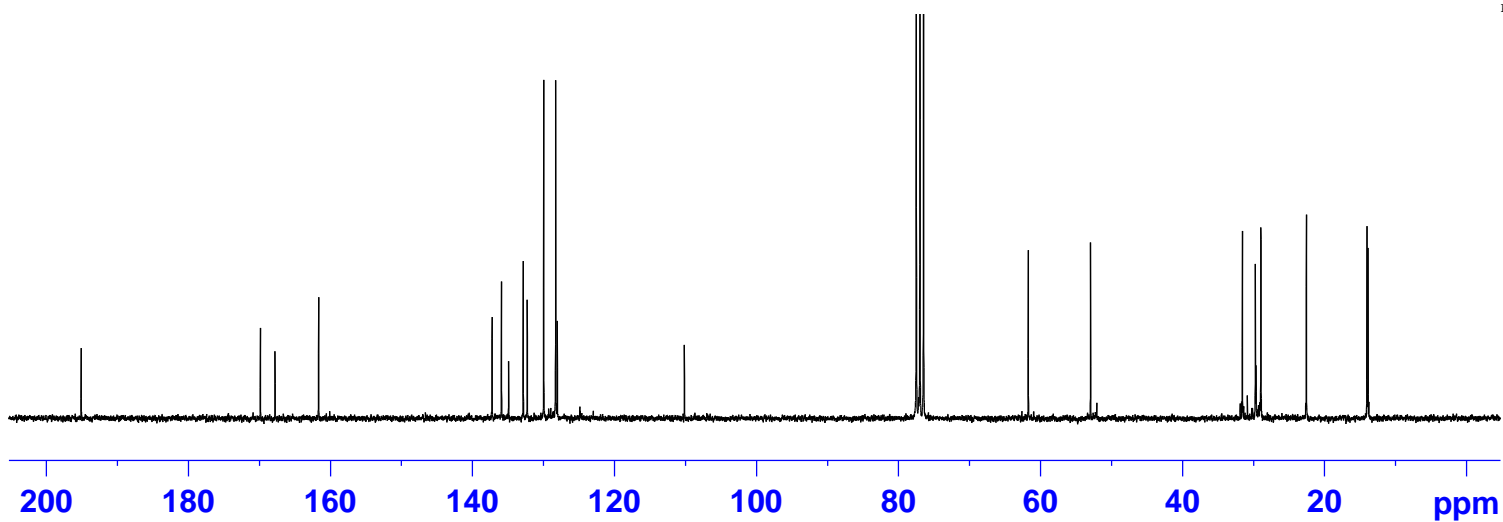
F2 - Acquisition Parameters  
Date\_ 20090116  
Time 9.59  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 80.6  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300263 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



5k



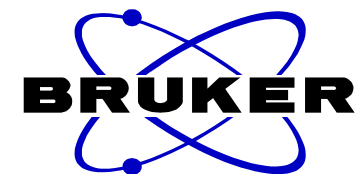
```
Current Data Parameters
NAME          090116.212
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090118
Time          10.53
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.0 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz
```

```
F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

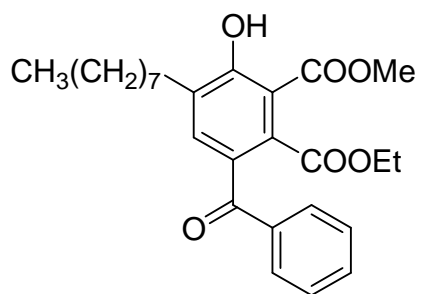


Current Data Parameters  
NAME 090116.u320  
EXPNO 10  
PROCNO 1

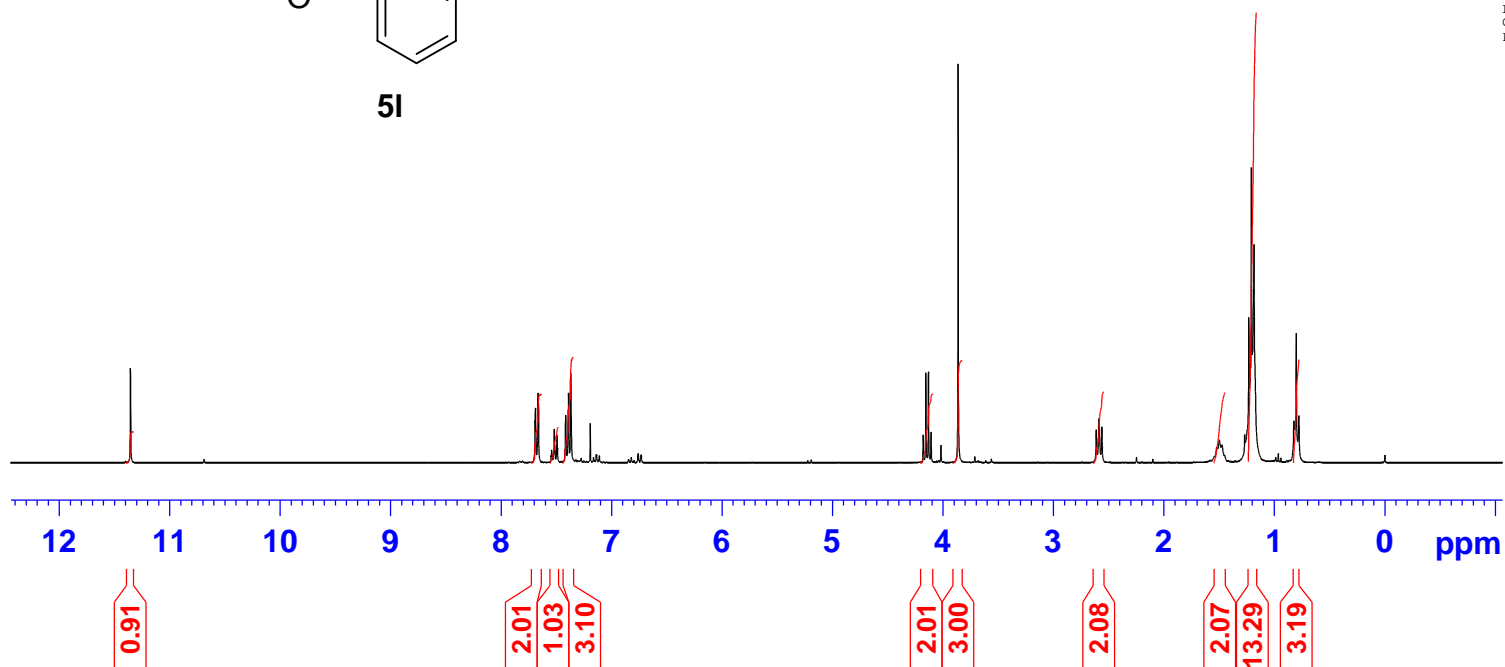
F2 - Acquisition Parameters  
Date\_ 20090116  
Time 9.53  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 71.8  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

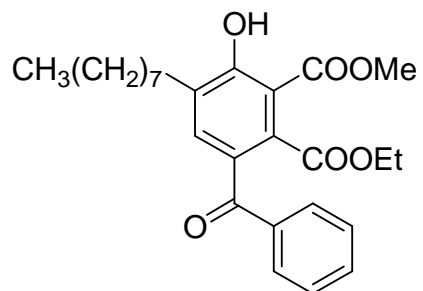
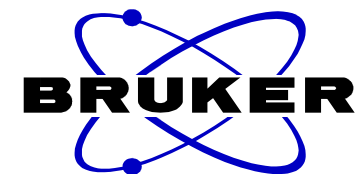
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300268 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

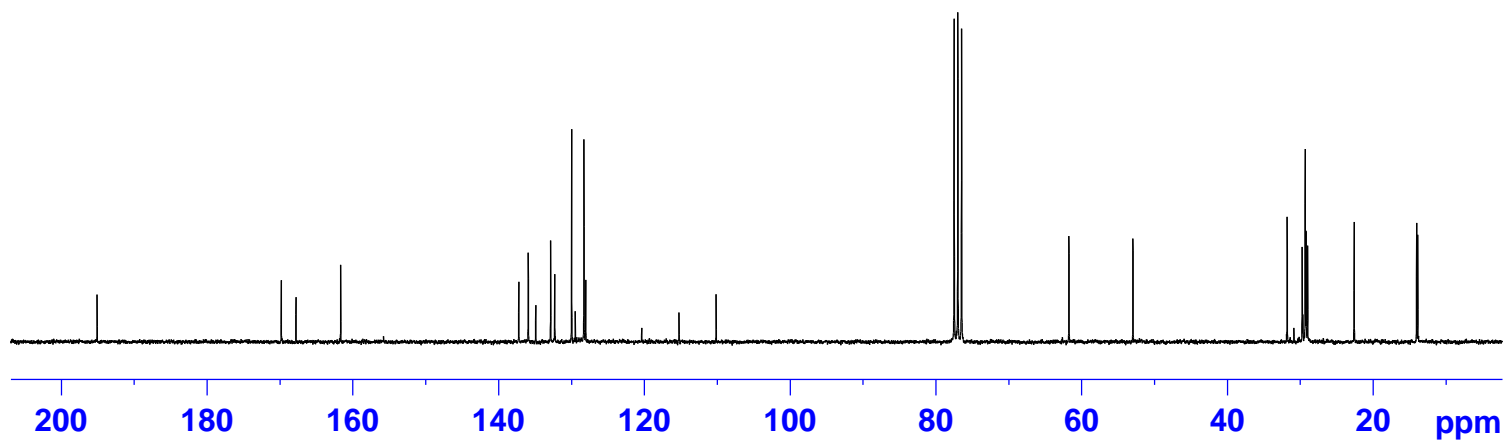


5I





51



```
Current Data Parameters
NAME      090116.211
EXPNO     11
PROCNO    1

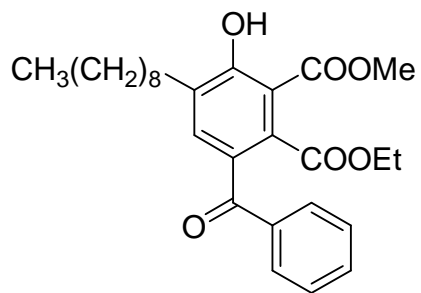
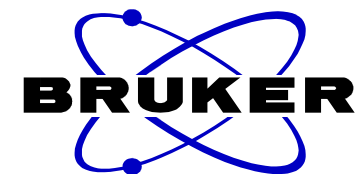
F2 - Acquisition Parameters
Date_     20090118
Time      7.46
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        2048
DS        4
SWH       15000.000 Hz
FIDRES    0.228882 Hz
AQ        2.1845834 sec
RG        2050
DW        33.333 usec
DE        10.00 usec
TE        298.0 K
D1        2.0000000 sec
d11       0.0300000 sec
DELTA     1.89999998 sec
TD0       1
```

```
===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       -1.00 dB
SFO1     62.9015280 MHz
```

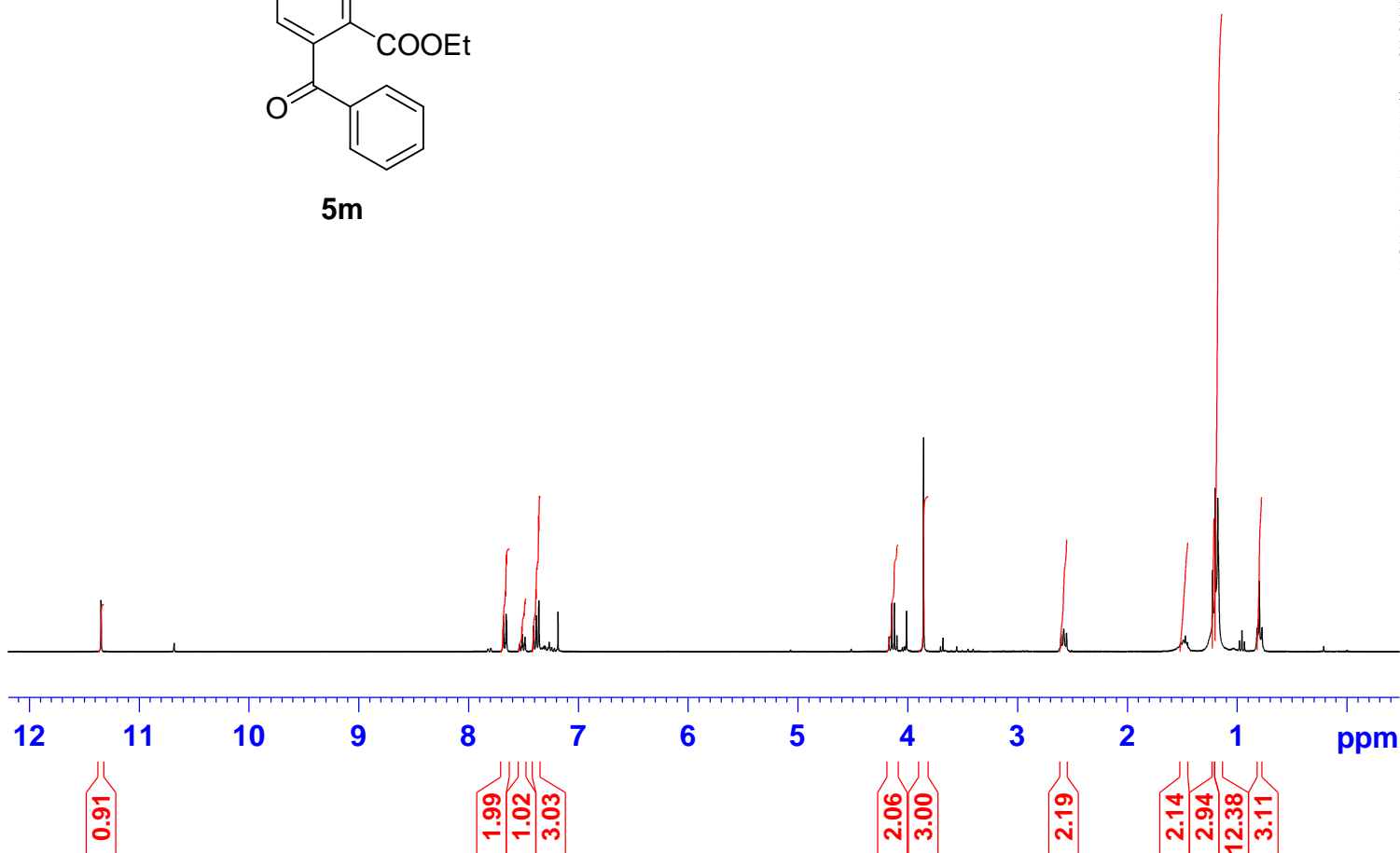
```
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     70.00 usec
PL12      15.00 dB
PL13      15.00 dB
PL2       -2.50 dB
SFO2     250.1310005 MHz
```

```
F2 - Processing parameters
SI        32768
SF        62.8952390 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```





5m

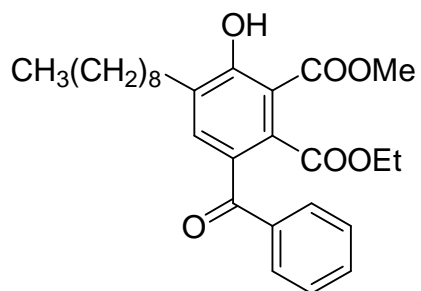
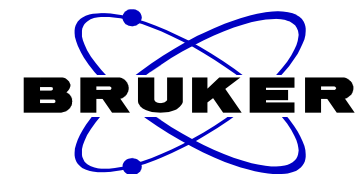


Current Data Parameters  
NAME 090206.u316  
EXPNO 10  
PROCNO 1

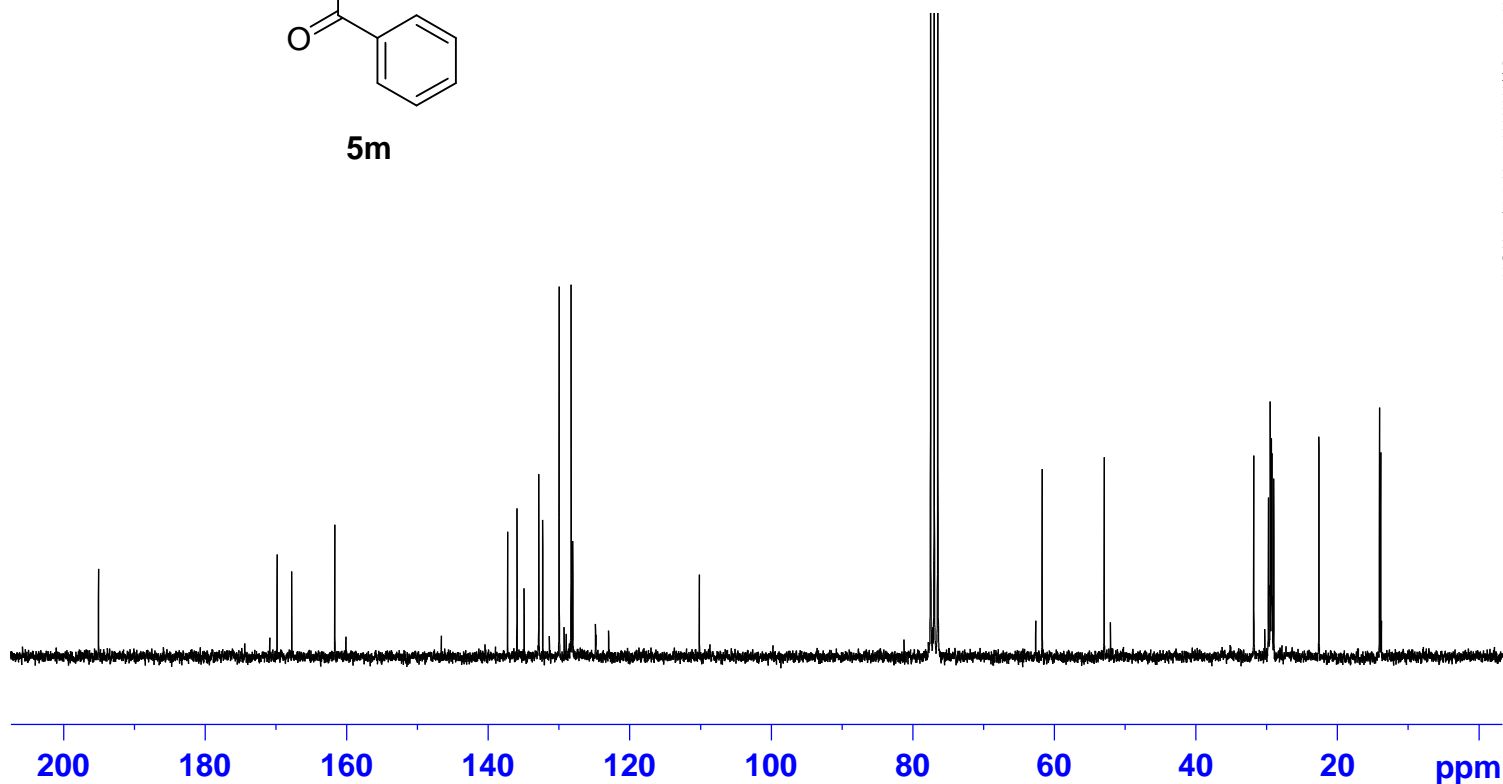
F2 - Acquisition Parameters  
Date\_ 20090206  
Time 9.24  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 90.5  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300289 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



5m



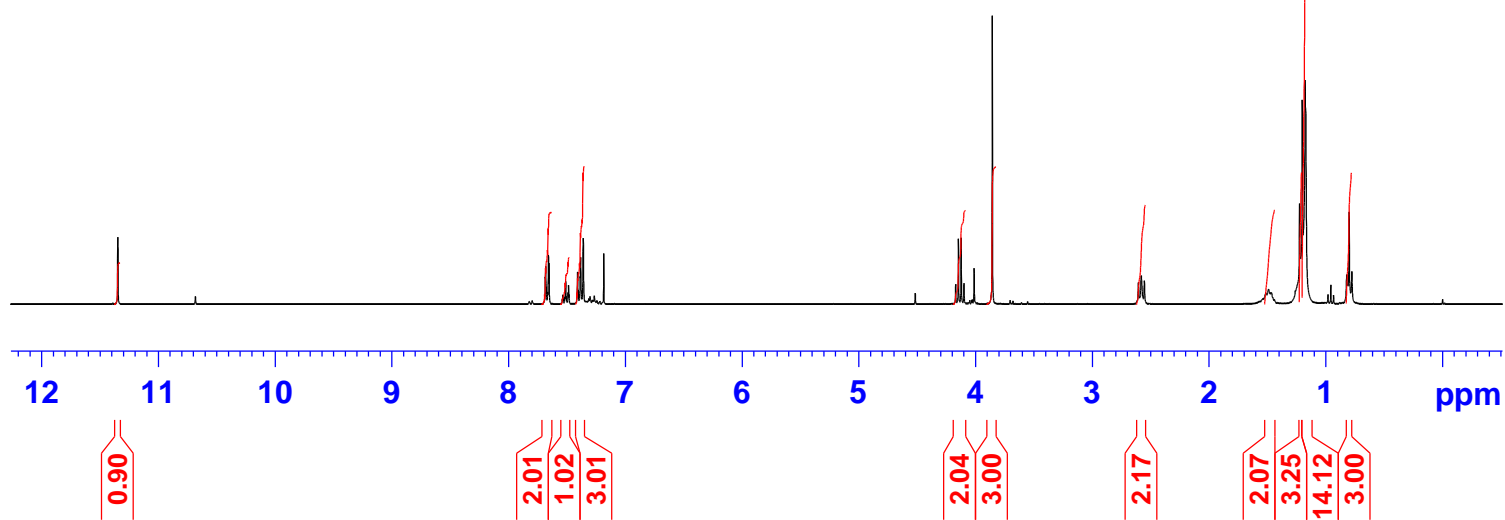
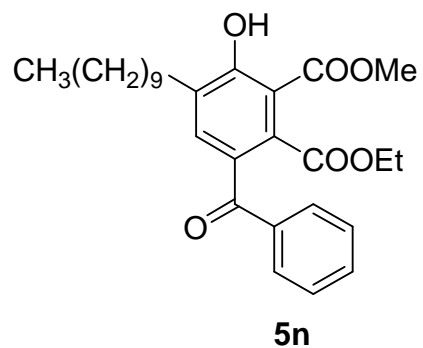
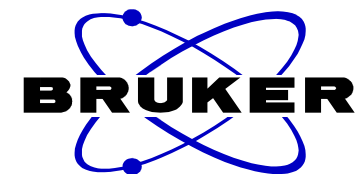
Current Data Parameters  
NAME 090206.218  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090208  
Time 3.39  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1500  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 297.9 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

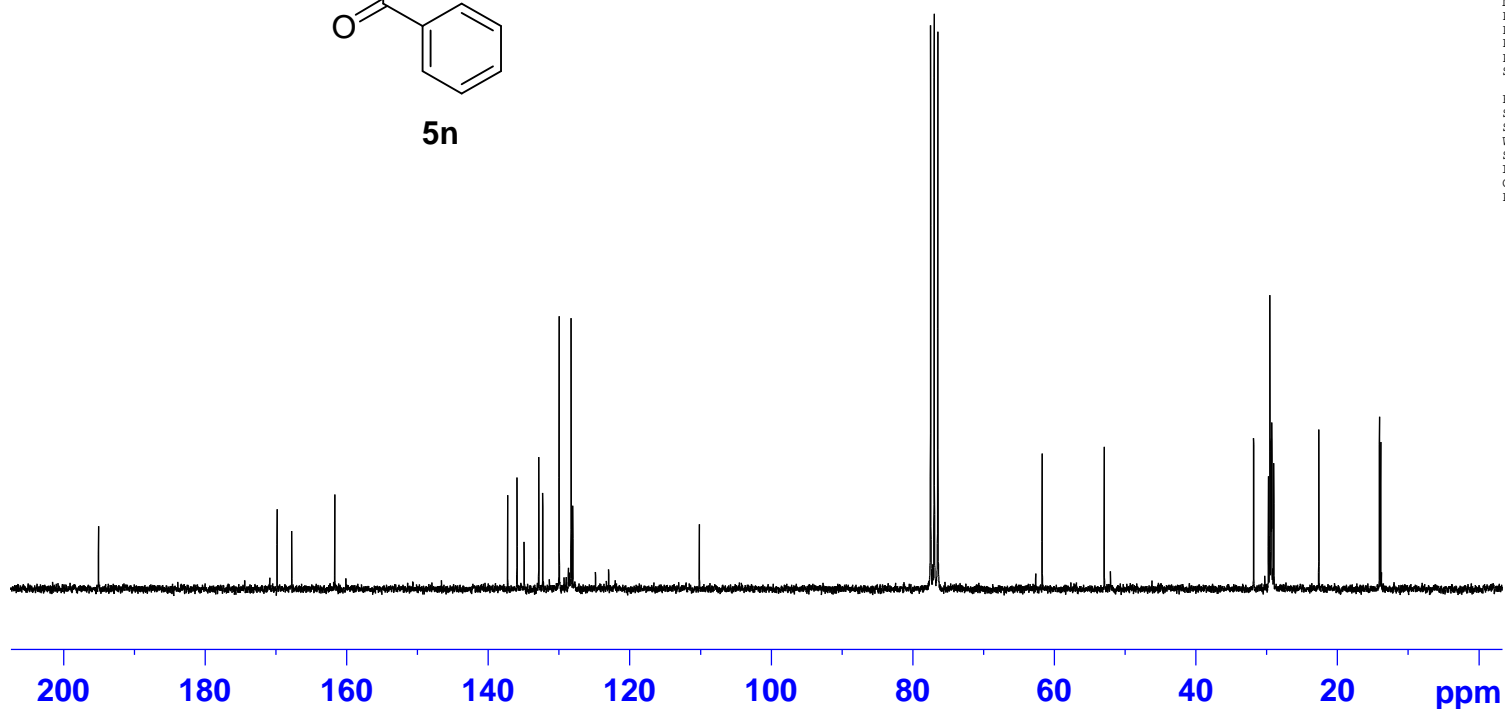
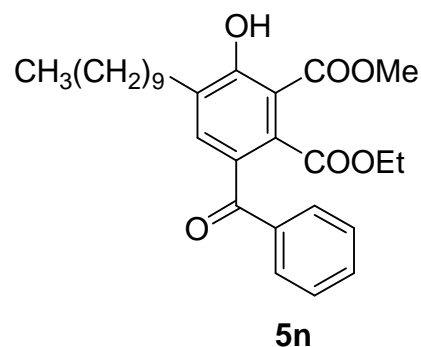
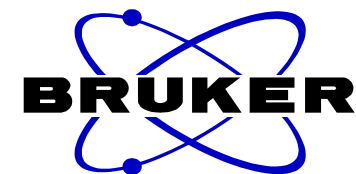


```
Current Data Parameters
NAME      090206.u317
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20090206
Time     9.32
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       90.5
DW       80.800 usec
DE       10.00 usec
TE       298.2 K
DL       1.0000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     1H
P1       10.00 usec
PL1      0.00 dB
PL1W     11.25325108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300289 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```



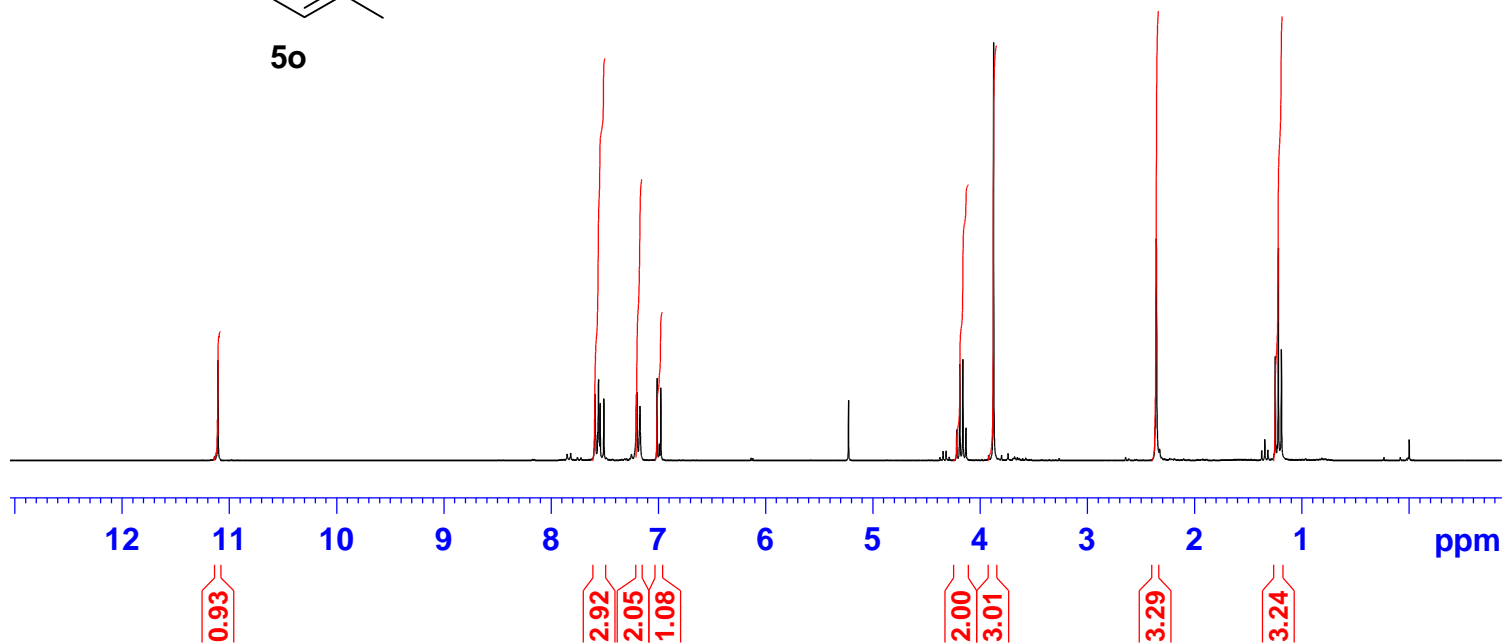
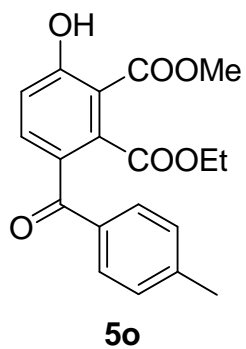
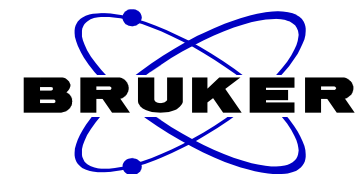
```
Current Data Parameters
NAME      090206.219
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20090208
Time     6.00
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1500
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1845834 sec
RG       2050
DW       33.333 usec
DE       10.00 usec
TE       298.1 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999999 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -1.00 dB
SFO1     62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     15.00 dB
PL13     15.00 dB
PL2      -2.50 dB
SFO2     250.1310005 MHz

F2 - Processing parameters
SI       32768
SF       62.8952390 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

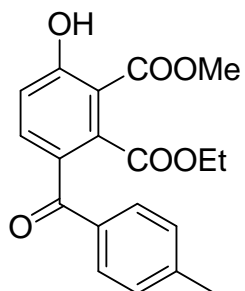
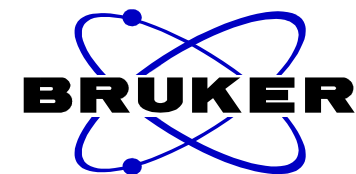


Current Data Parameters  
NAME 081212.209  
EXPNO 10  
PROCNO 1

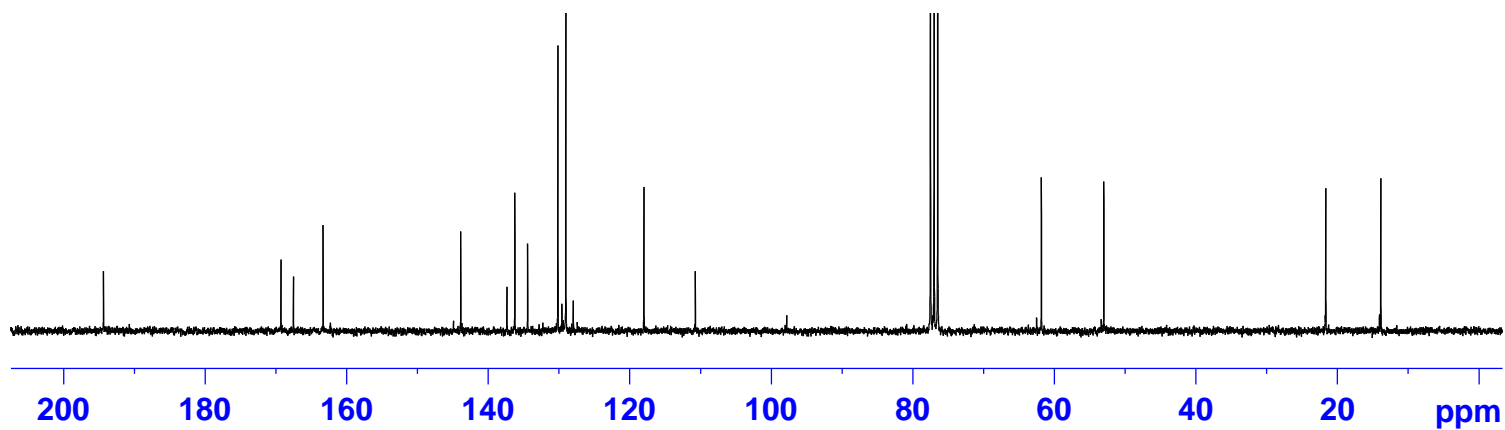
F2 - Acquisition Parameters  
Date\_ 20081212  
Time 9.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 5165.289 Hz  
FIDRES 0.078816 Hz  
AQ 6.3439350 sec  
RG 322  
DW 96.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 -2.50 dB  
SF01 250.1315447 MHz

F2 - Processing parameters  
SI 32768  
SF 250.1300159 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



5o



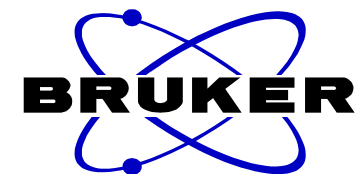
```
Current Data Parameters
NAME      081212.215
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20081212
Time      19.09
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH       15000.000 Hz
FIDRES    0.228882 Hz
AQ         2.1845834 sec
RG         2050
DW         33.333 usec
DE         10.00 usec
TE         298.4 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA     1.89999998 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -1.00 dB
SFO1       62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       70.00 usec
PL12        15.00 dB
PL13        15.00 dB
PL2         -2.50 dB
SFO2       250.1310005 MHz

F2 - Processing parameters
SI         32768
SF         62.8952390 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
```

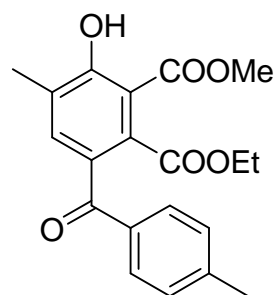


Current Data Parameters  
NAME 081218.u319  
EXPNO 10  
PROCNO 1

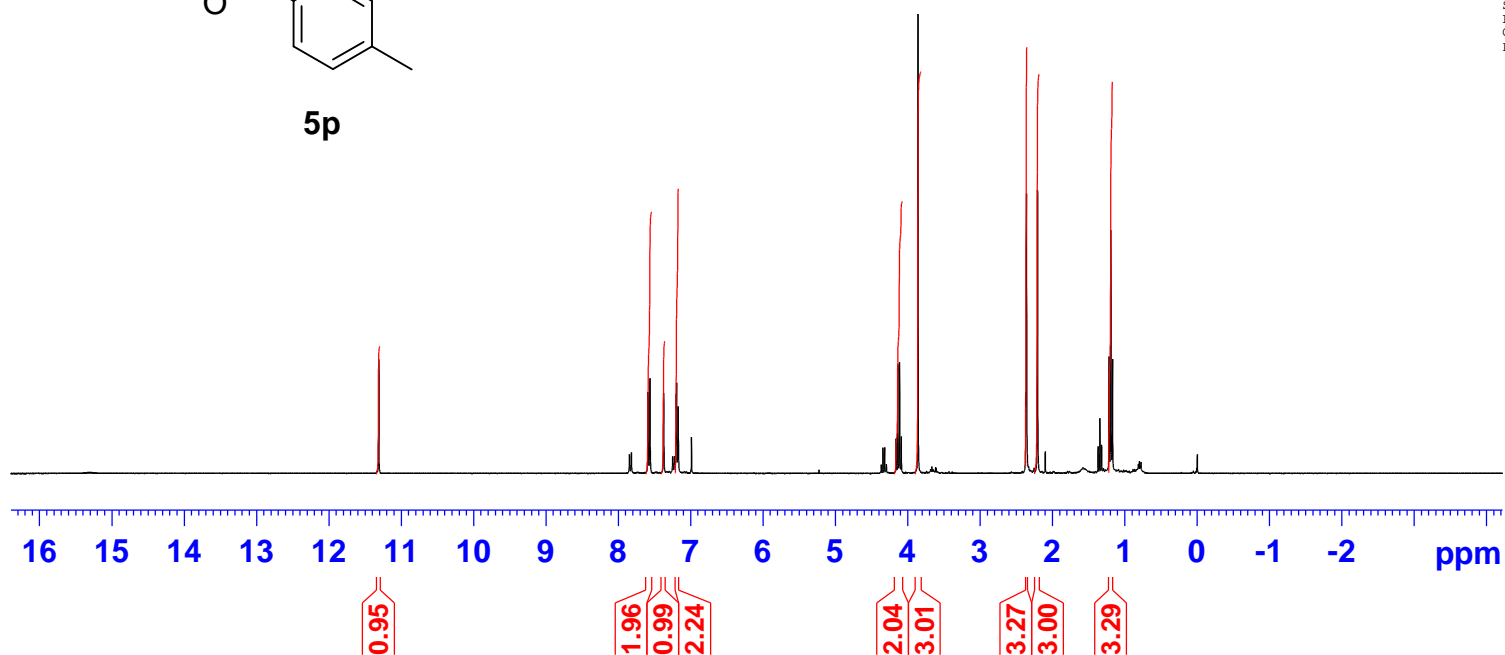
F2 - Acquisition Parameters  
Date\_ 20081218  
Time 10.00  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

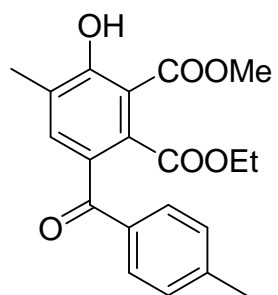
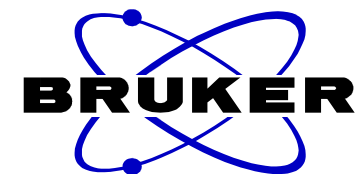
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300261 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

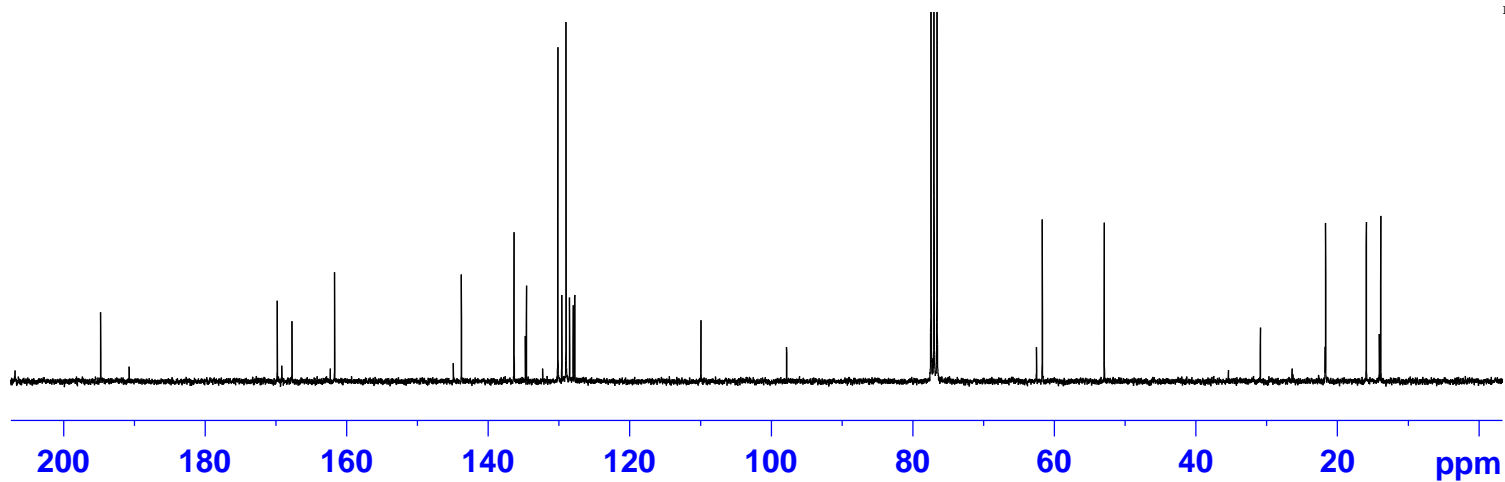


5p





5p



```
Current Data Parameters
NAME      081218.u335
EXPNO    11
PROCNO   1

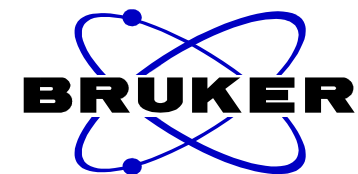
F2 - Acquisition Parameters
Date_    20081219
Time     1.05
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1500
DS       4
SWH      18028.846 Hz
FIDRES   0.275098 Hz
AQ       1.8175818 sec
RG       2050
DW       27.733 usec
DE       10.00 usec
TE       298.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -0.50 dB
PL1W     33.25691986 W
SFO1     75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    72.00 usec
PL2      0.00 dB
PL12     17.00 dB
PL13     17.00 dB
PL2W     11.25325108 W
PL12W    0.22453187 W
PL13W    0.22453187 W
SFO2     300.1312005 MHz

F2 - Processing parameters
SI       32768
SF       75.4677490 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```



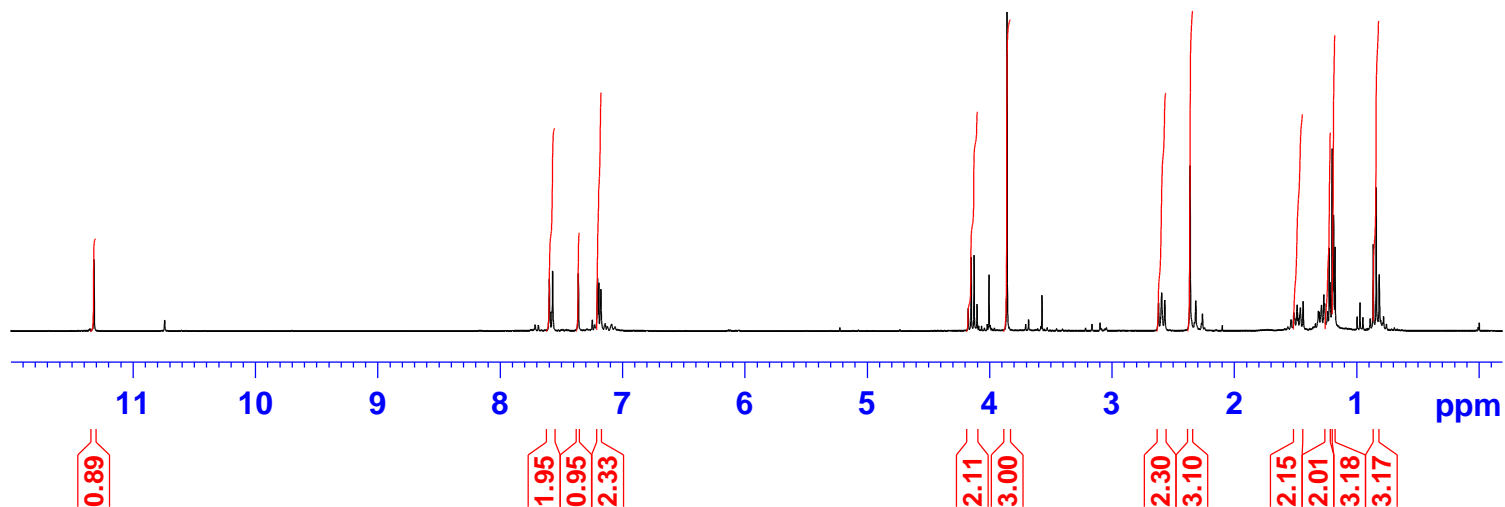
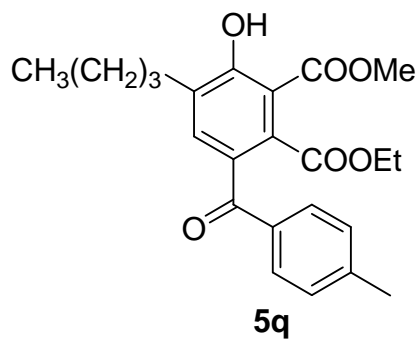


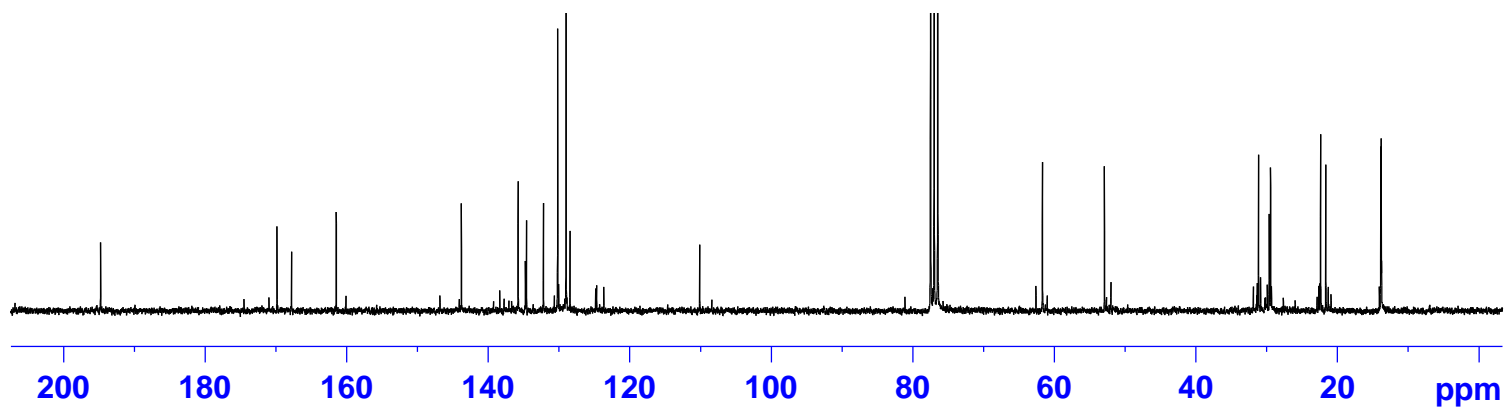
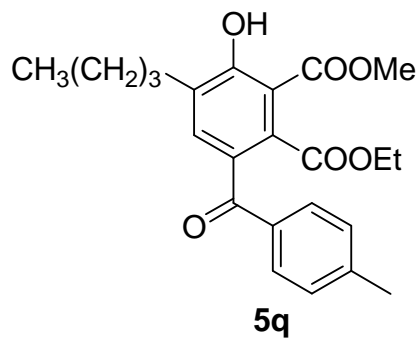
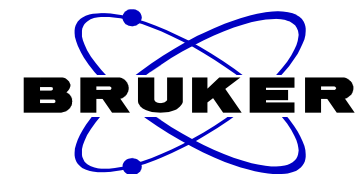
Current Data Parameters  
NAME 090116.u319  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090116  
Time 9.48  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 80.6  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300265 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





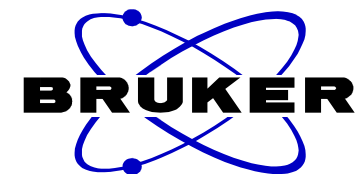
```
Current Data Parameters
NAME          090116.210
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090118
Time          4.39
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.1 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         15.00 dB
PL13         15.00 dB
PL2          -2.50 dB
SFO2         250.1310005 MHz
```

```
F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

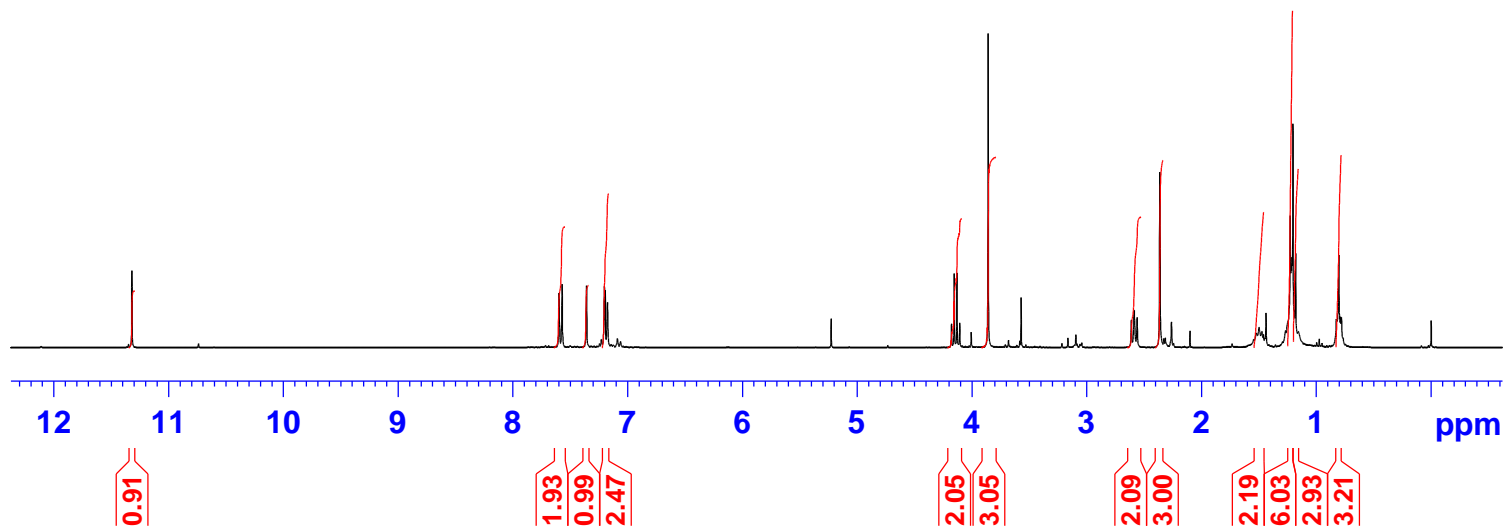
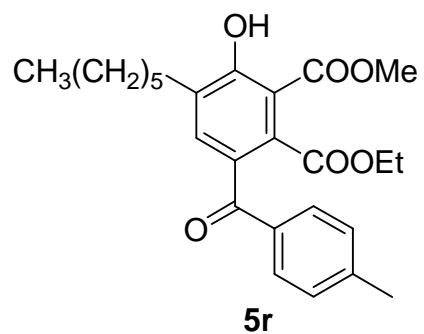


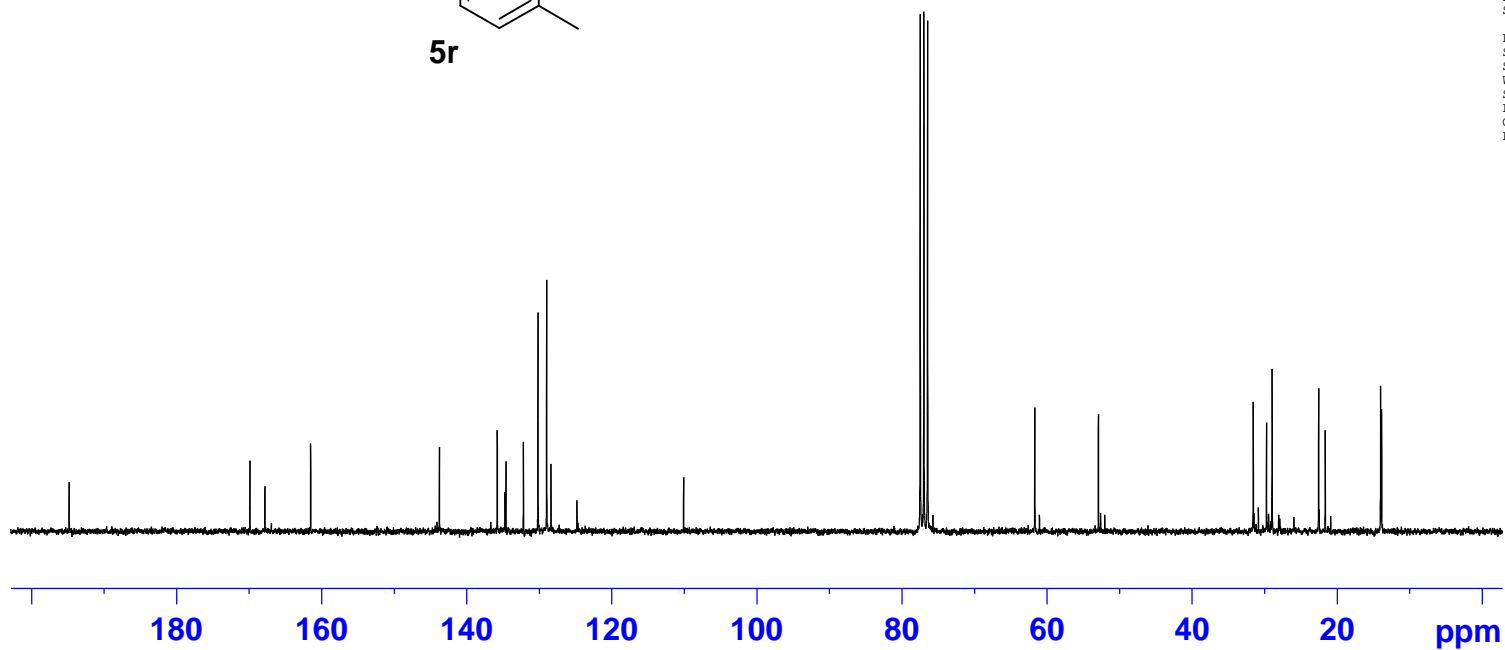
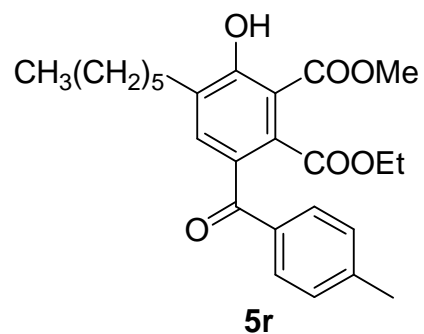
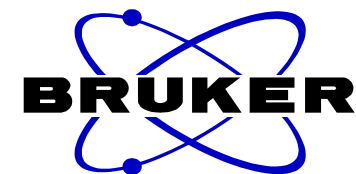
Current Data Parameters  
NAME 090116.u322  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090116  
Time 10.05  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 90.5  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300263 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





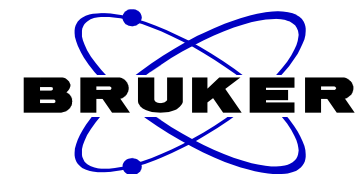
Current Data Parameters  
NAME 090116.208  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090117  
Time 22.25  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 2048  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 297.9 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

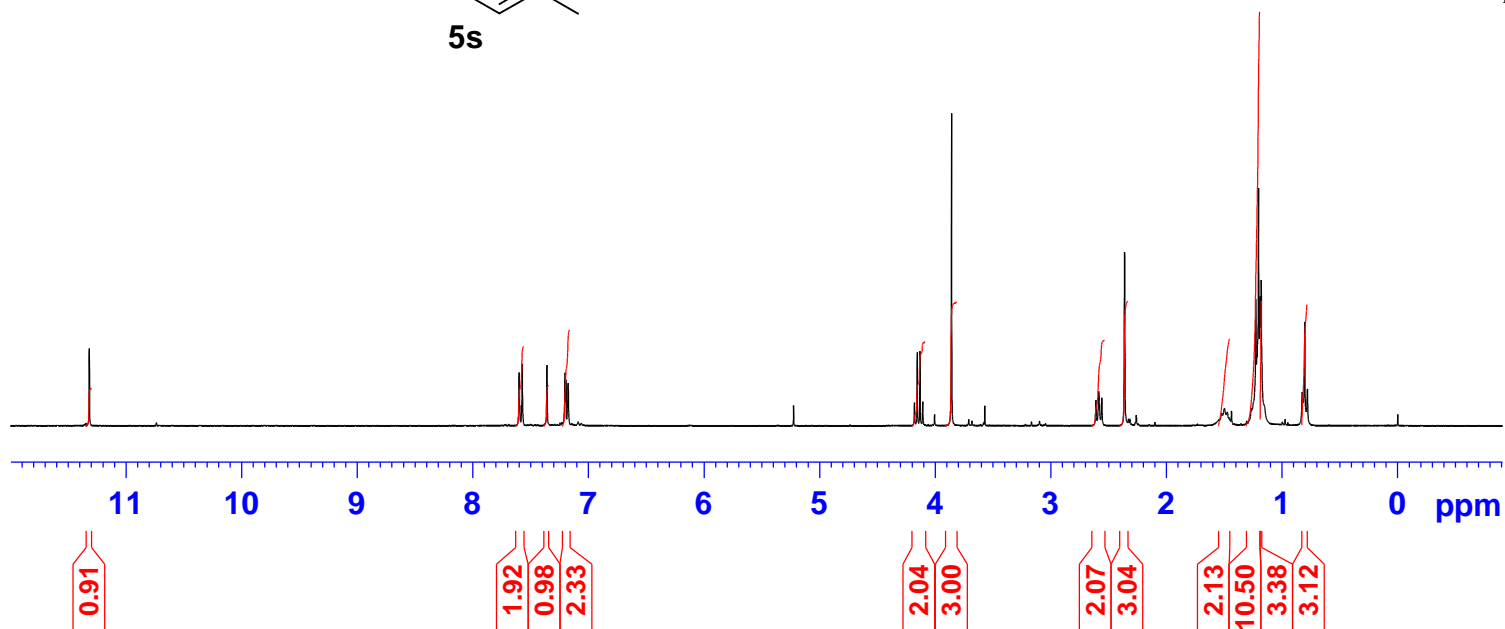
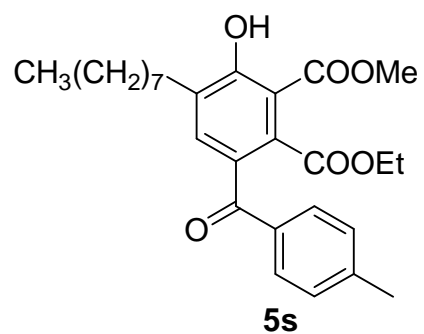


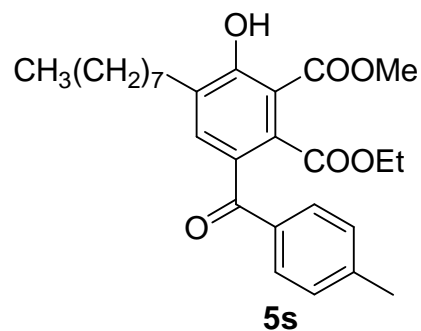
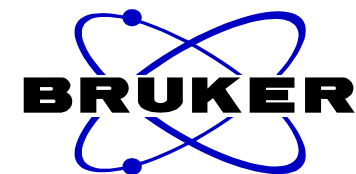
Current Data Parameters  
NAME 090116.u323  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090116  
Time 10.11  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 71.8  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
DL 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300264 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





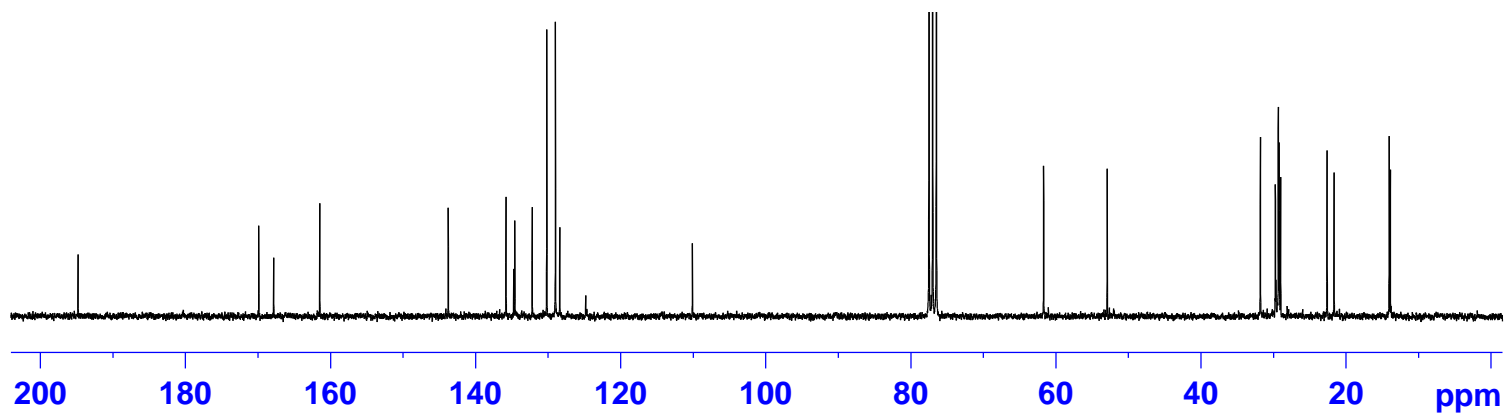
Current Data Parameters  
NAME 090116.207  
EXPNO 11  
PROCNO 1

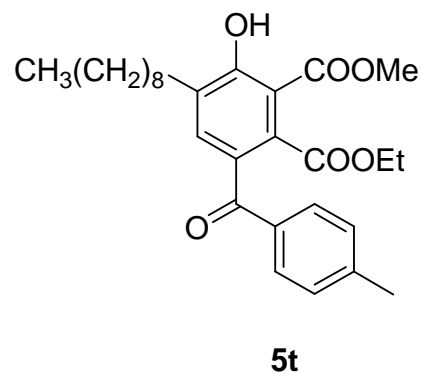
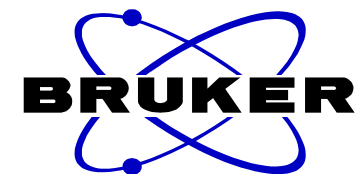
F2 - Acquisition Parameters  
Date\_ 20090117  
Time 19.19  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 2048  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 298.1 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



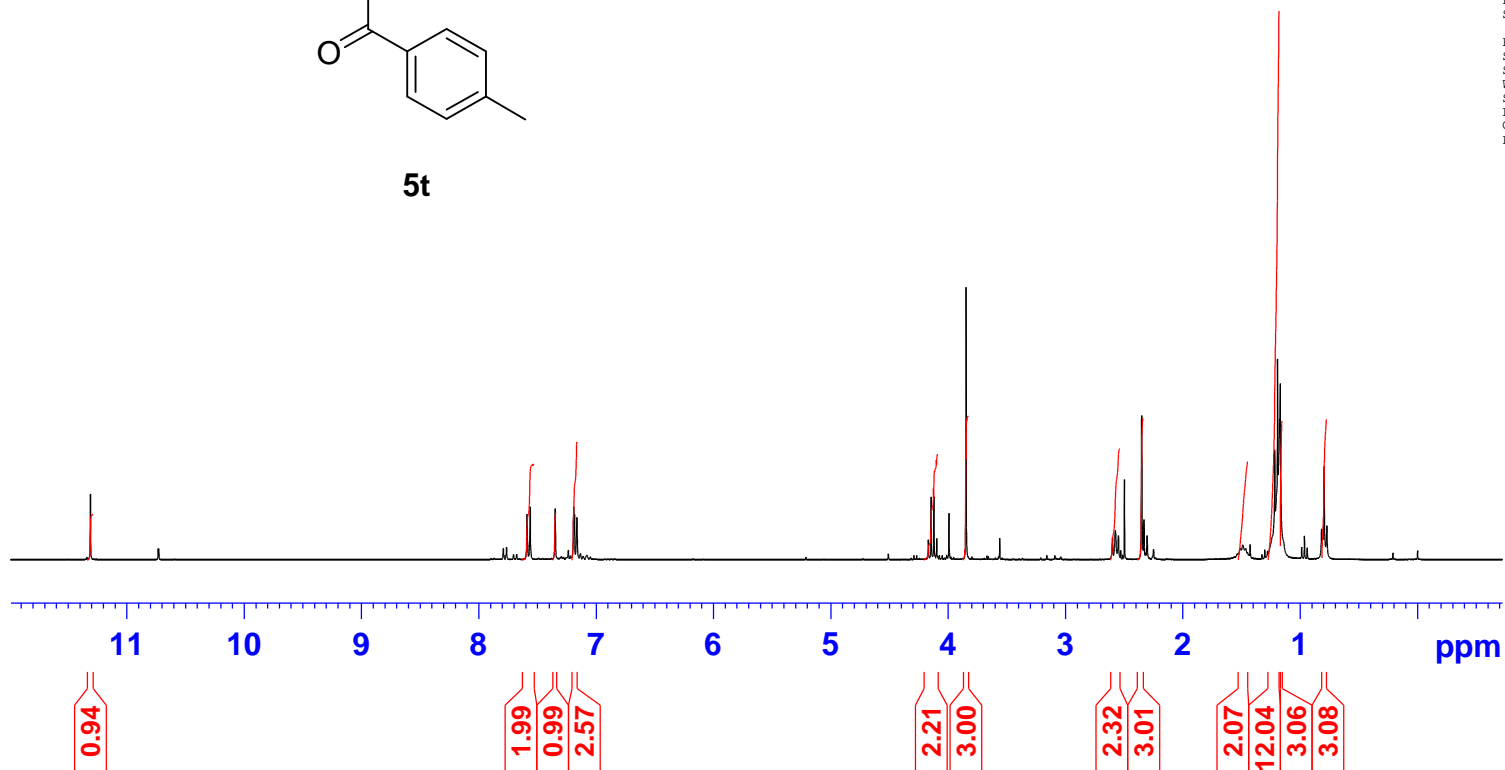


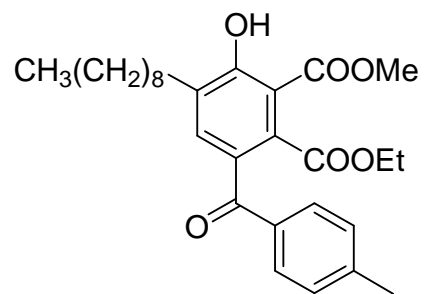
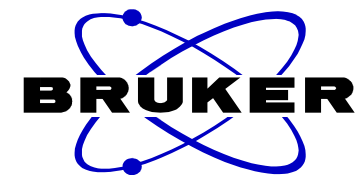
Current Data Parameters  
NAME 090211.u323  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090211  
Time 9.49  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 50.8  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

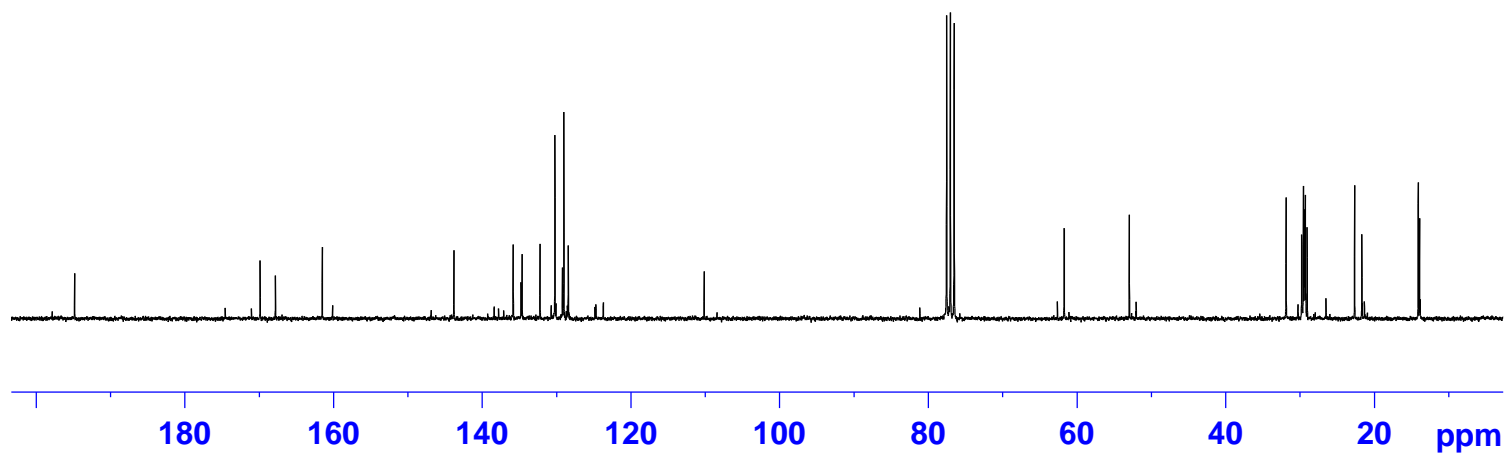
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300294 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





5t



```
Current Data Parameters
NAME          090212.209
EXPNO         11
PROCNO        1

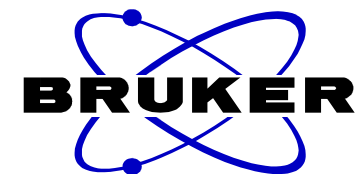
F2 - Acquisition Parameters
Date_         20090213
Time          2.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1500
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.0 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz

F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



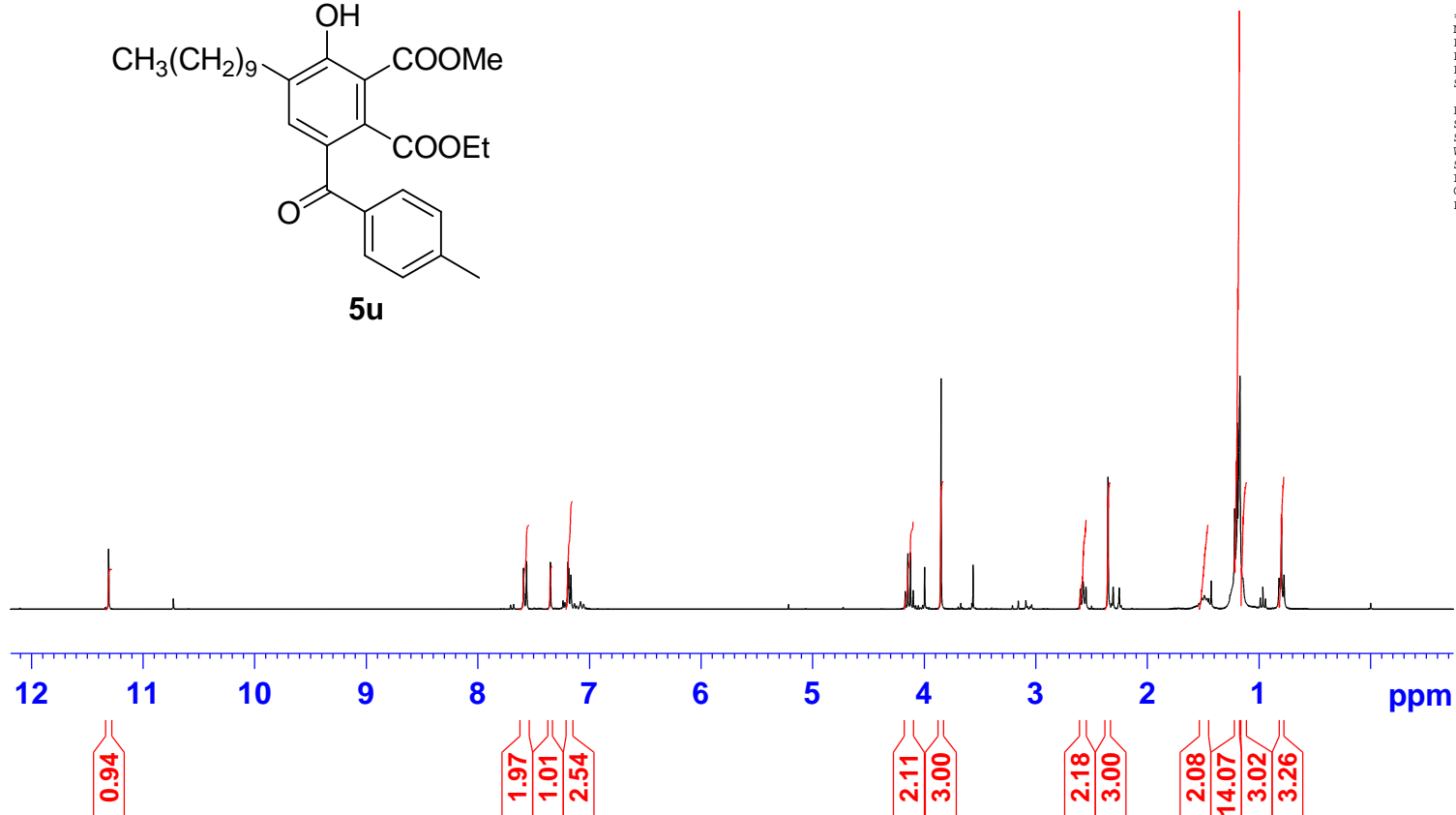
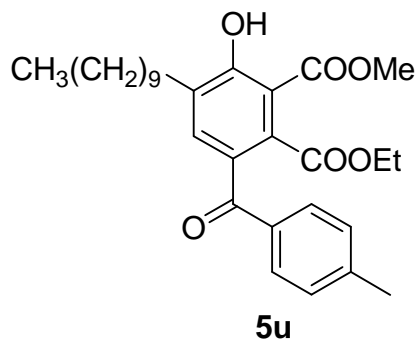


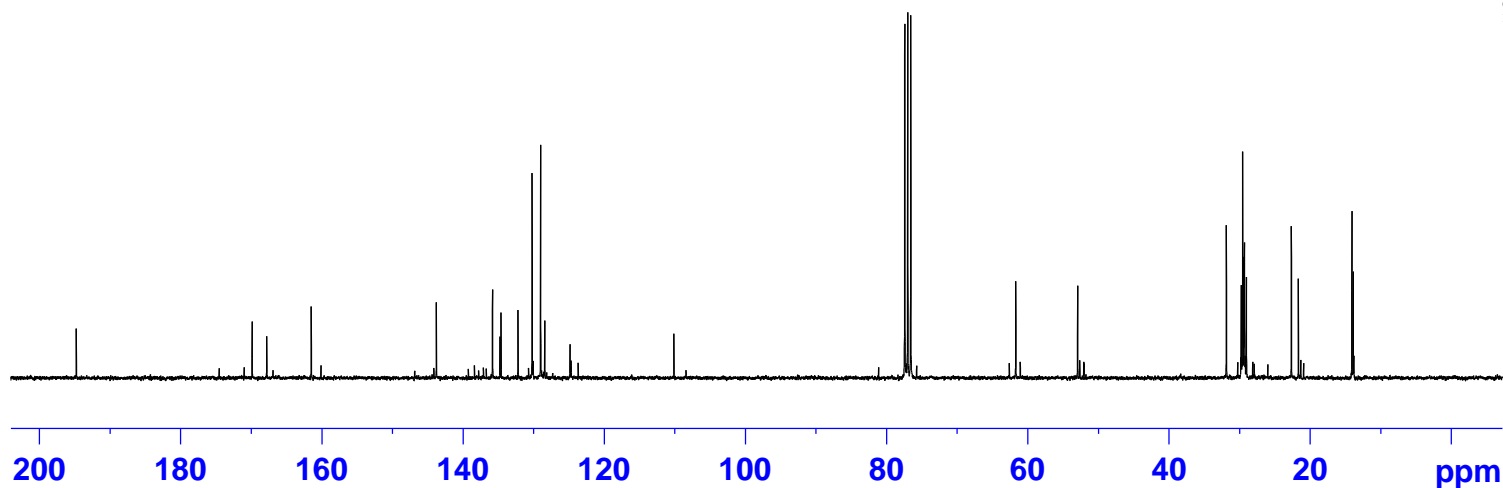
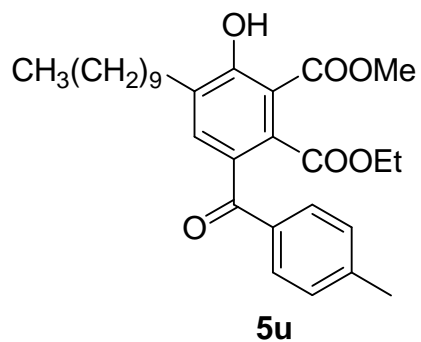
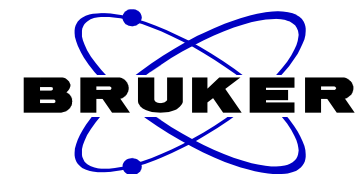
Current Data Parameters  
NAME 090211.u325  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090211  
Time 10.01  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 57  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300292 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





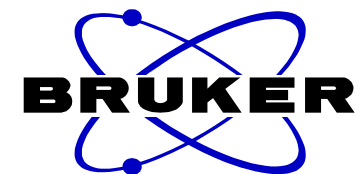
```
Current Data Parameters
NAME          090211.u357
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090212
Time          2.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1500
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            -0.50 dB
PL1W           33.25691986 W
SFO1           75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         72.00 usec
PL2            0.00 dB
PL12           17.00 dB
PL13           17.00 dB
PL2W           11.25325108 W
PL12W          0.22453187 W
PL13W          0.22453187 W
SFO2           300.1312005 MHz

F2 - Processing parameters
SI             32768
SF             75.4677490 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

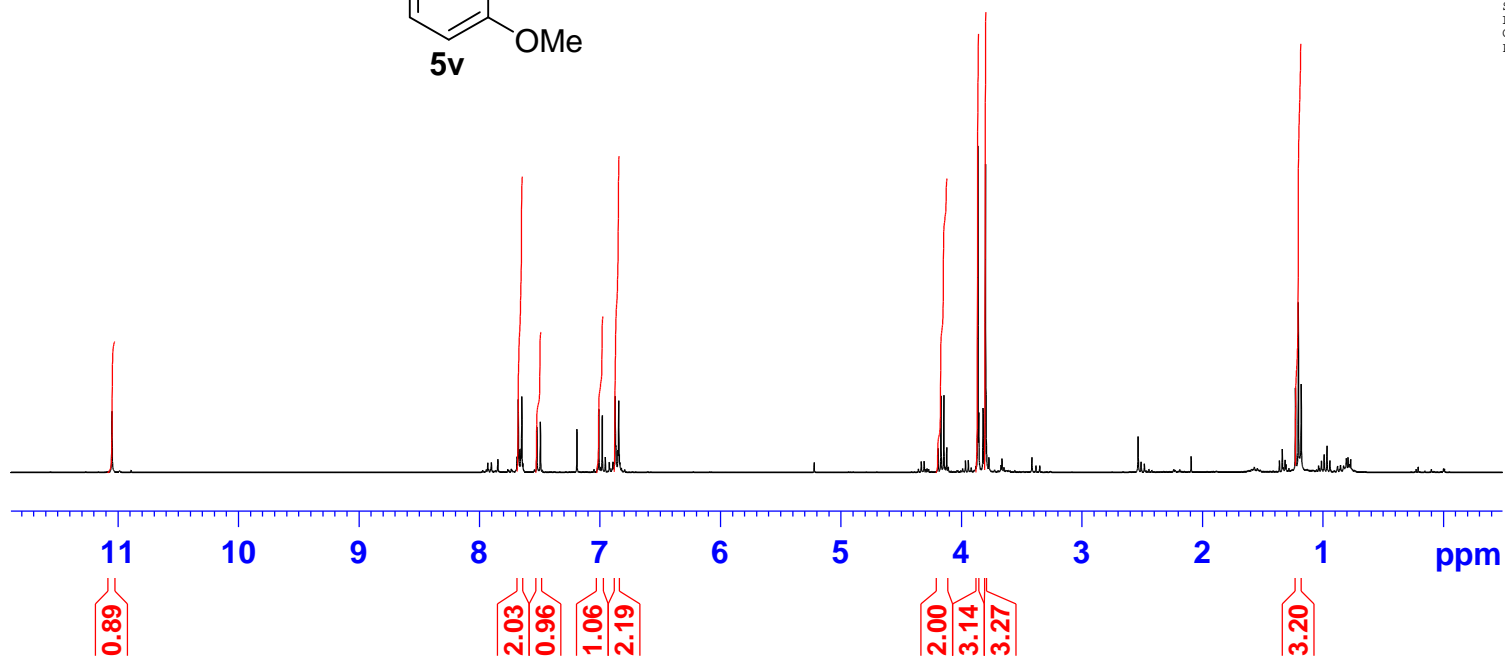
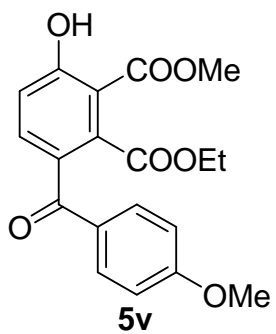


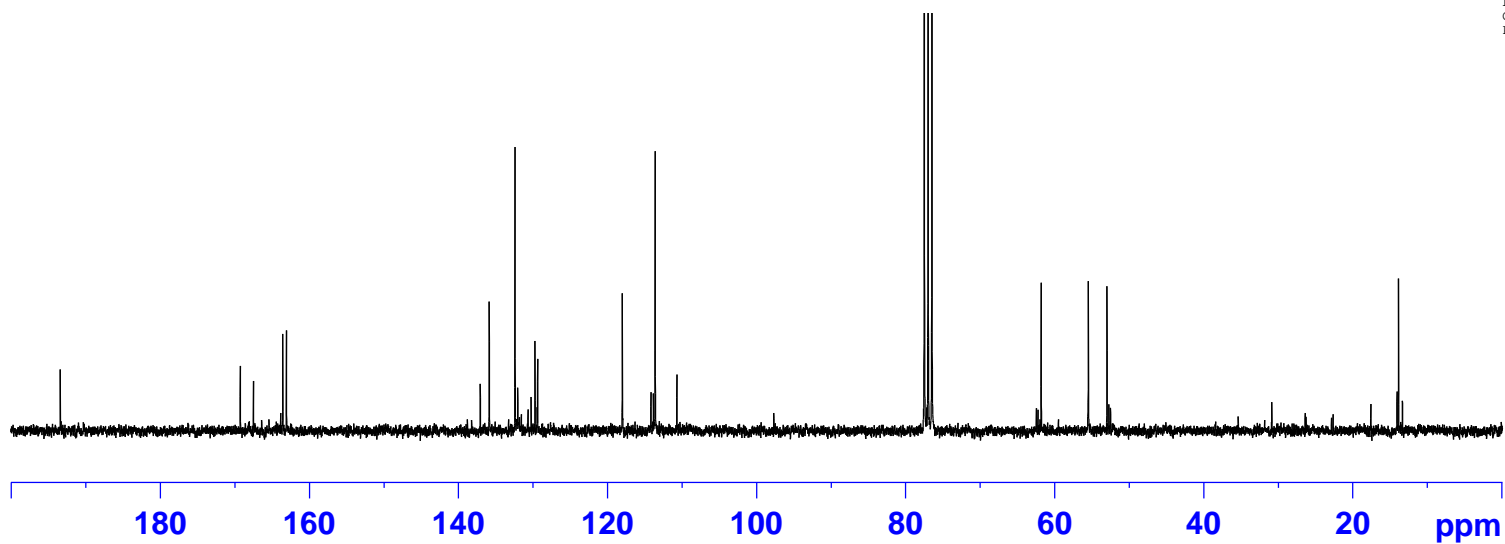
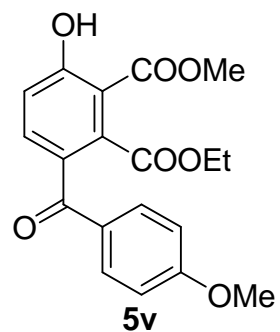
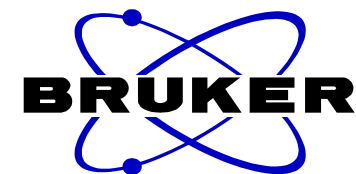
Current Data Parameters  
NAME 090120.u316  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090120  
Time 10.49  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 128  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300273 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





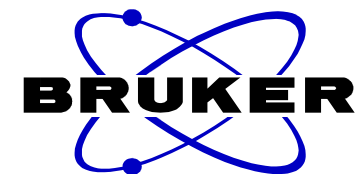
```
Current Data Parameters
NAME          090121.205
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090121
Time          18.34
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1         62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

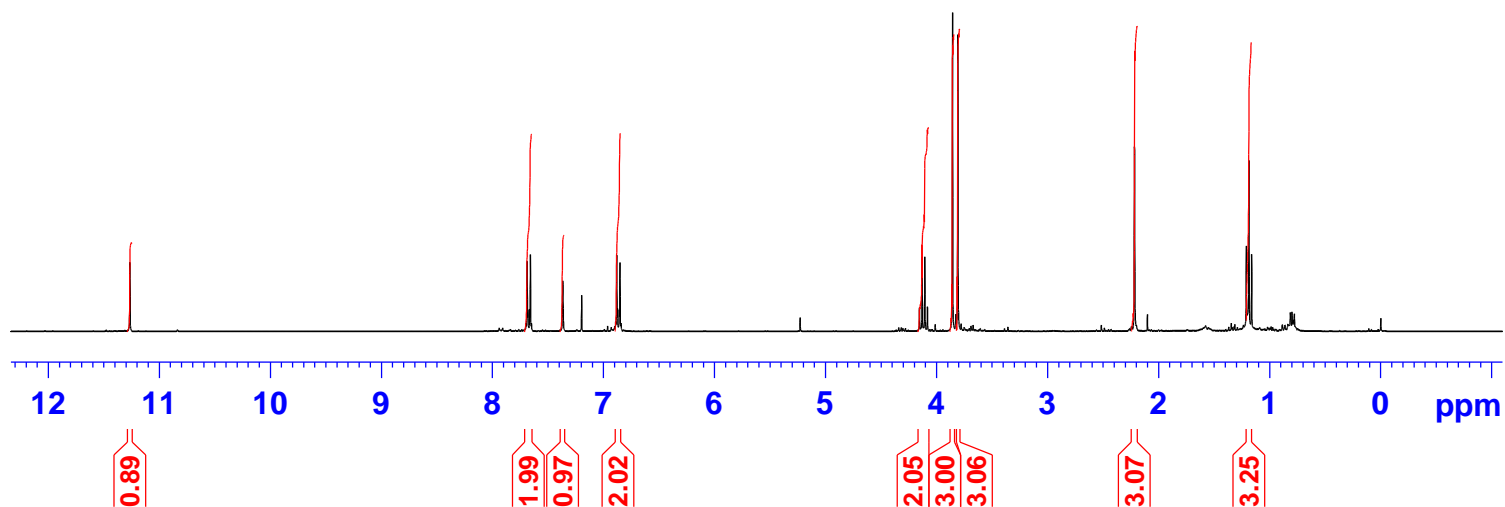
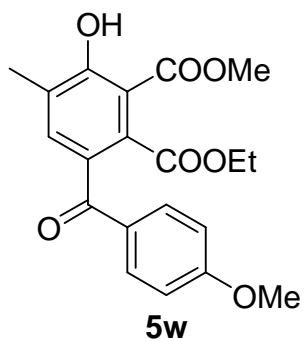


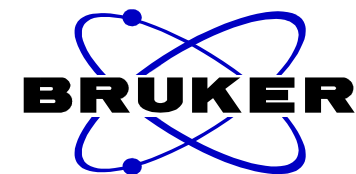
Current Data Parameters  
NAME 090120.u315  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090120  
Time 10.43  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300255 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





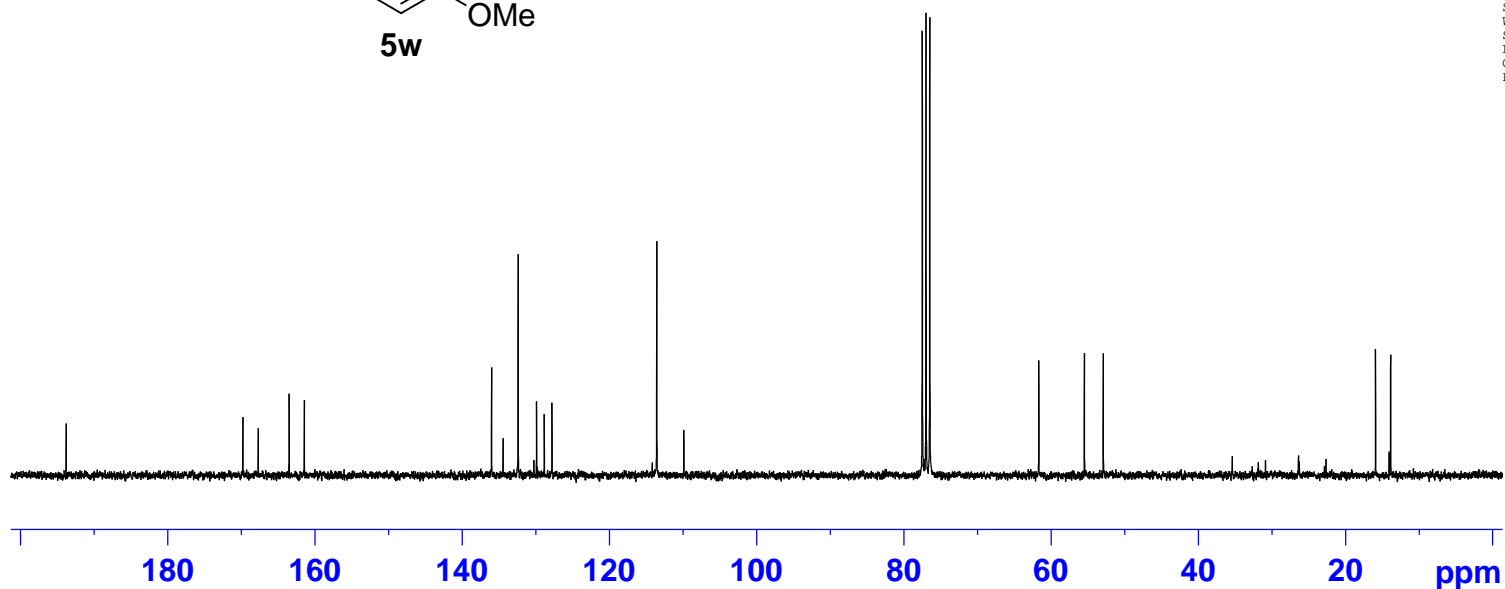
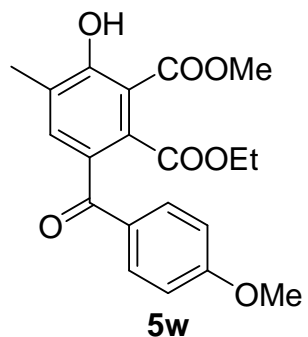
```
Current Data Parameters
NAME          090120.207
EXPNO         11
PROCNO        1

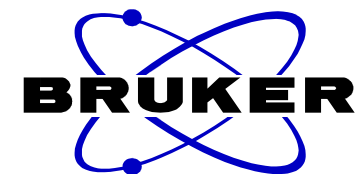
F2 - Acquisition Parameters
Date_         20090121
Time          0.29
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.0 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz
```

```
F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



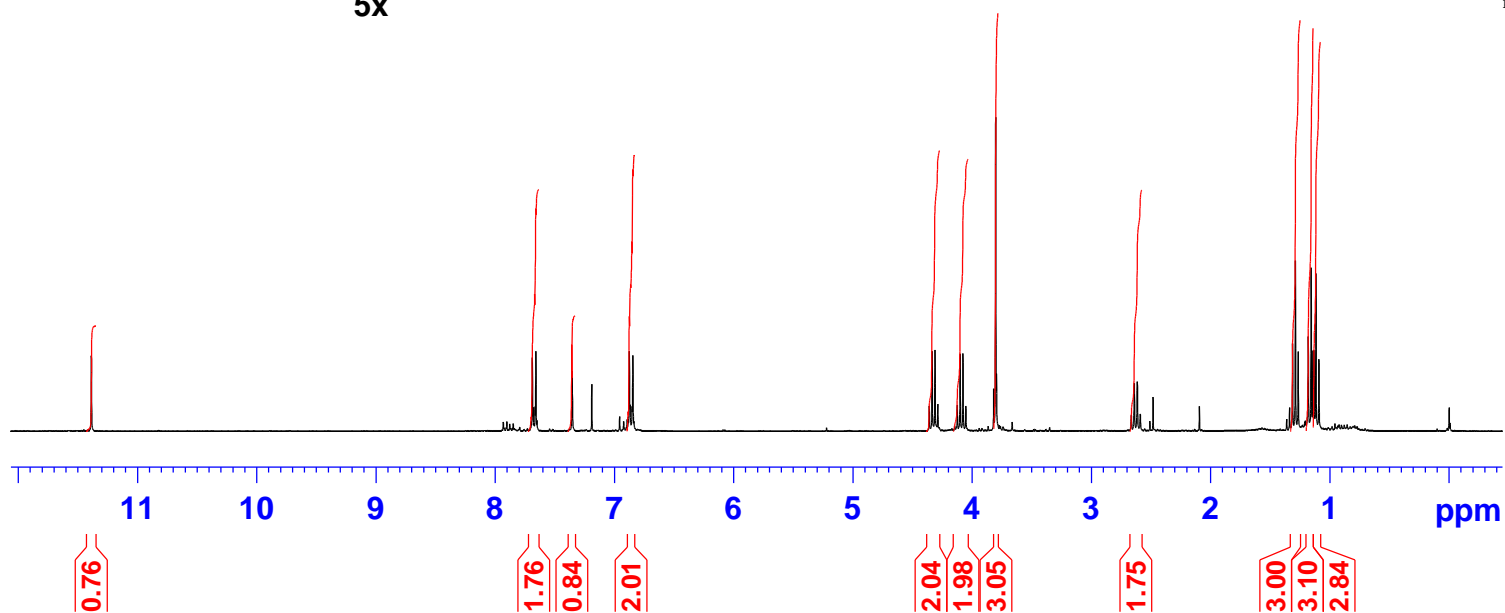
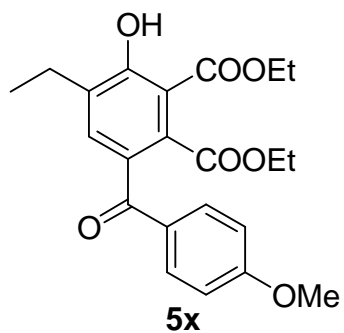


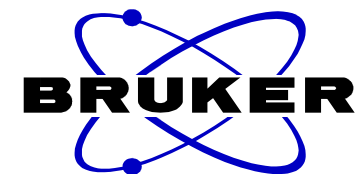
Current Data Parameters  
NAME 090120.u317  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090120  
Time 10.55  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300277 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





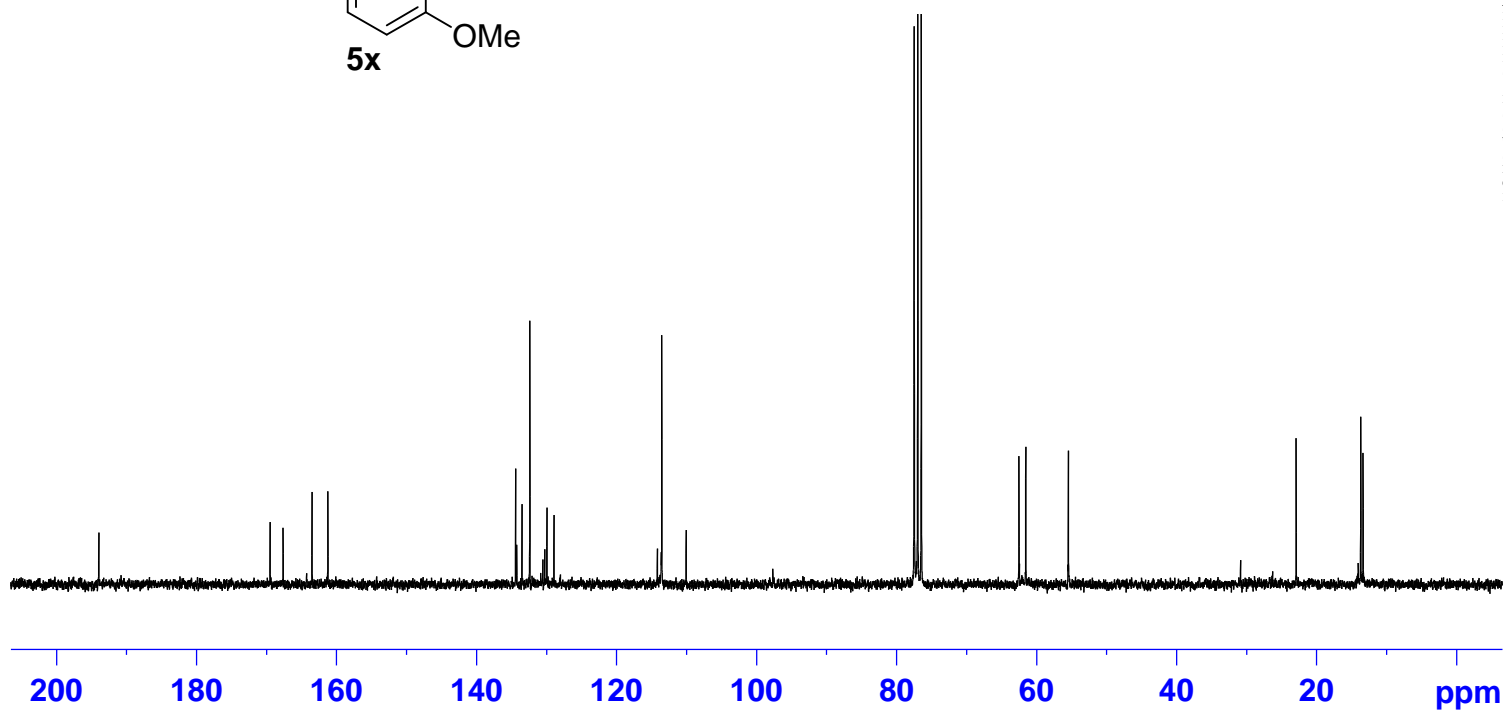
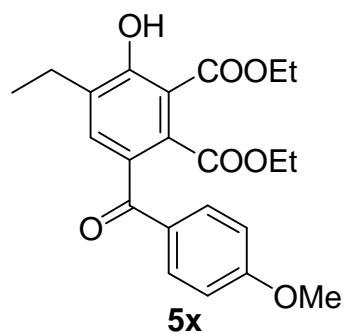
Current Data Parameters  
NAME 090121.206  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090121  
Time 20.10  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 298.2 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999998 sec  
TD0 1

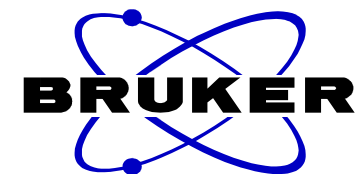
===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





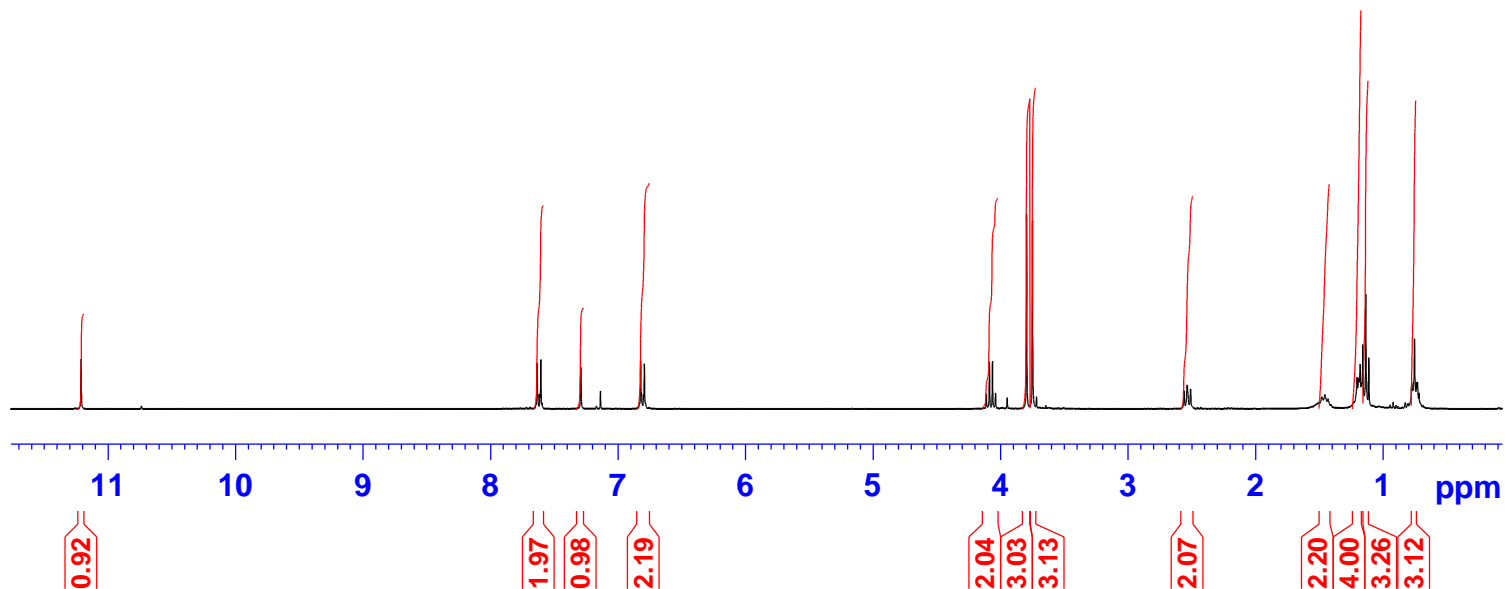
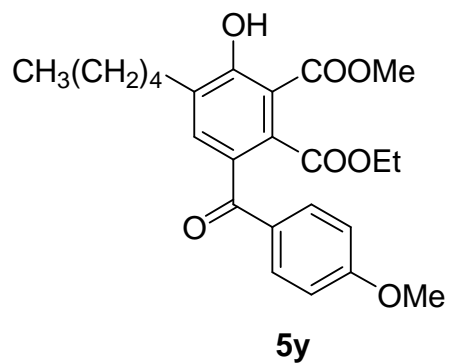


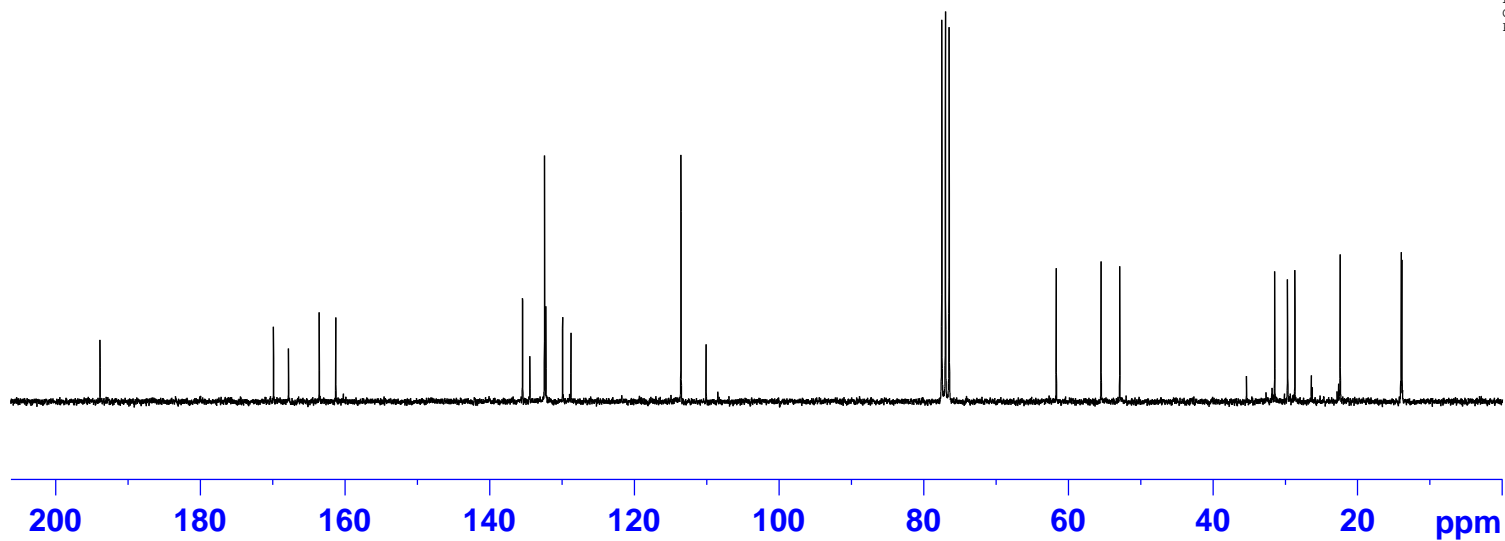
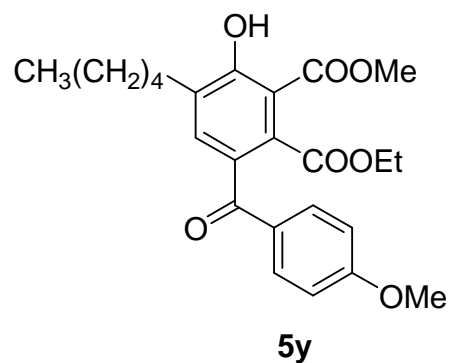
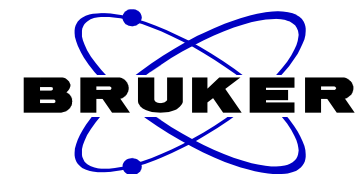
Current Data Parameters  
NAME 090128.u318  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090128  
Time 9.12  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 64  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300433 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





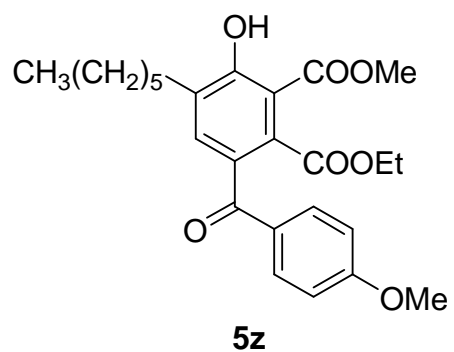
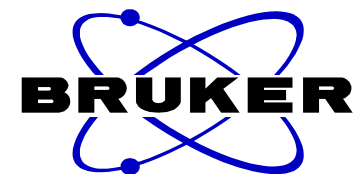
```
Current Data Parameters
NAME          090128.207
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090128
Time          19.50
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz

F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

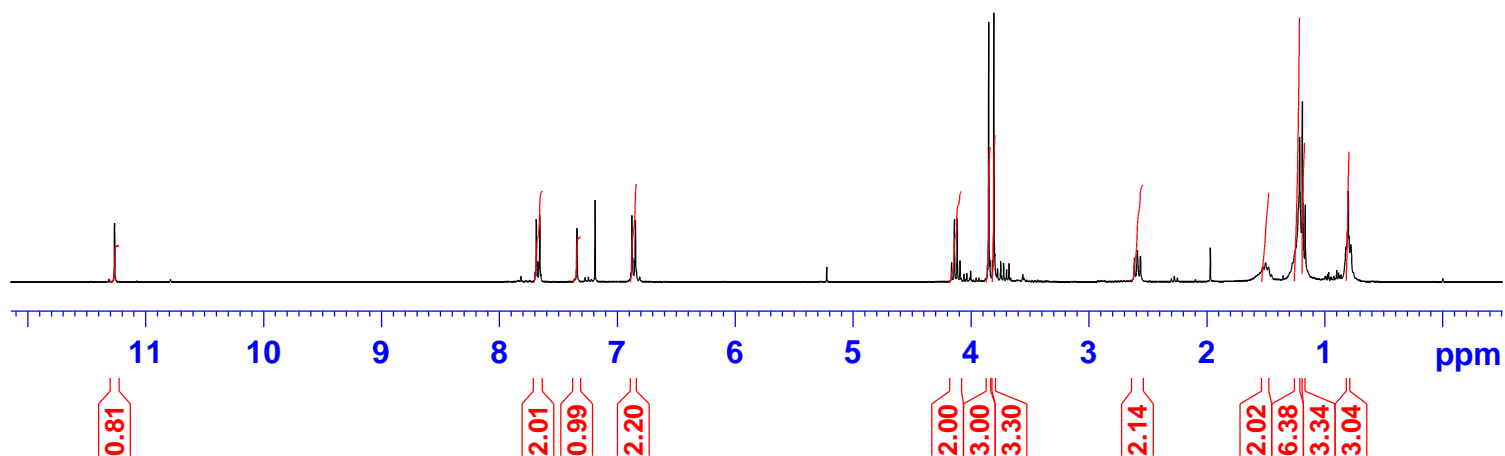


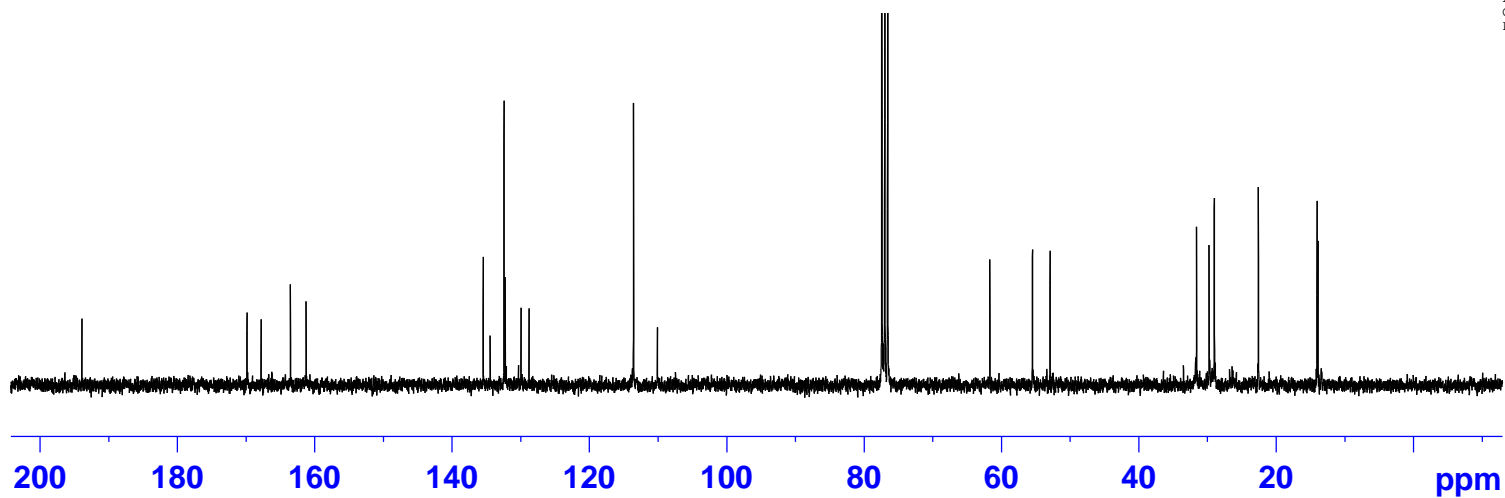
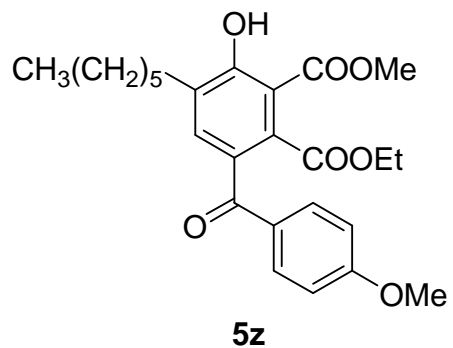
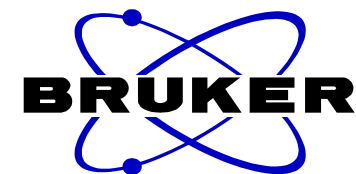
Current Data Parameters  
NAME 090206.u313  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090206  
Time 9.07  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 128  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300278 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





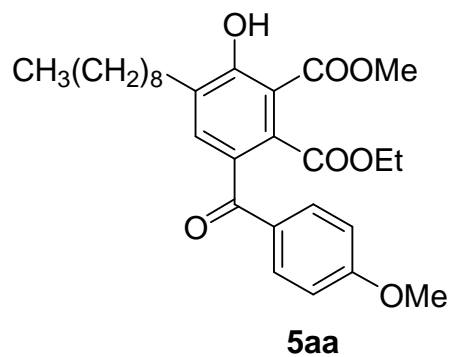
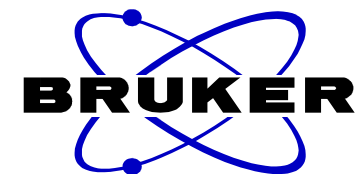
```
Current Data Parameters
NAME          090209.u333
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20090209
Time          12.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            947
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.25691986 W
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        72.00 usec
PL2           0.00 dB
PL12         17.00 dB
PL13         17.00 dB
PL2W         11.25325108 W
PL12W        0.22453187 W
PL13W        0.22453187 W
SFO2         300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

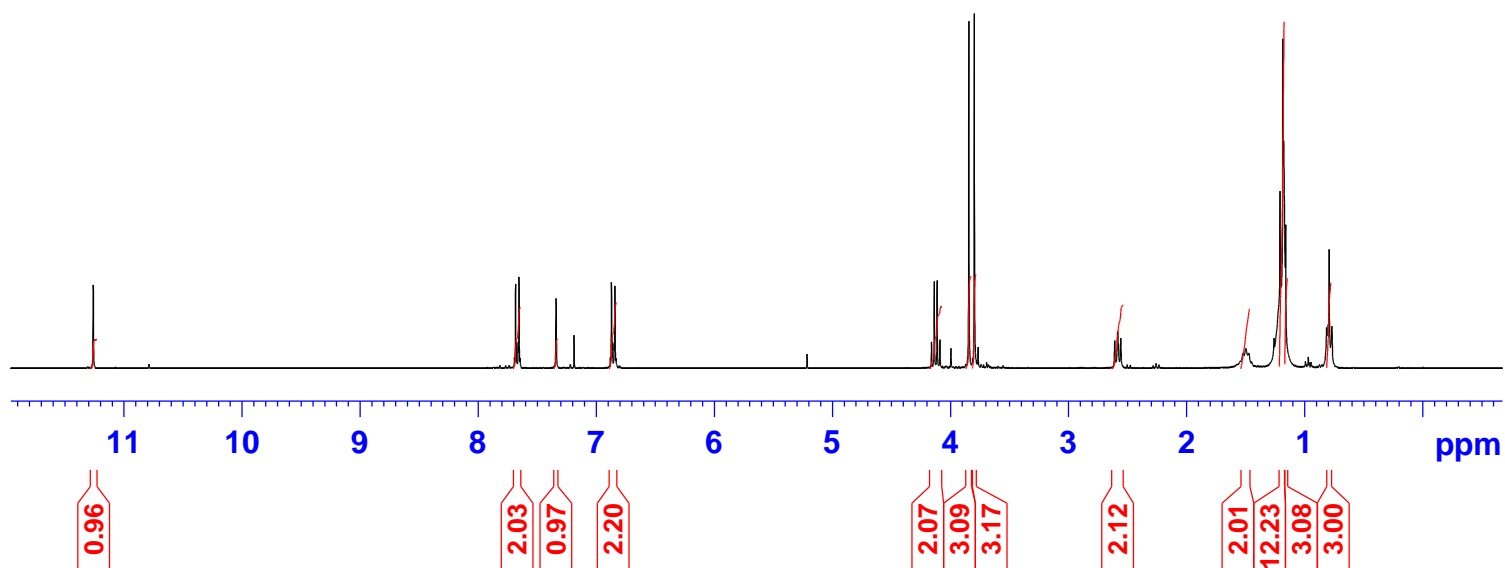


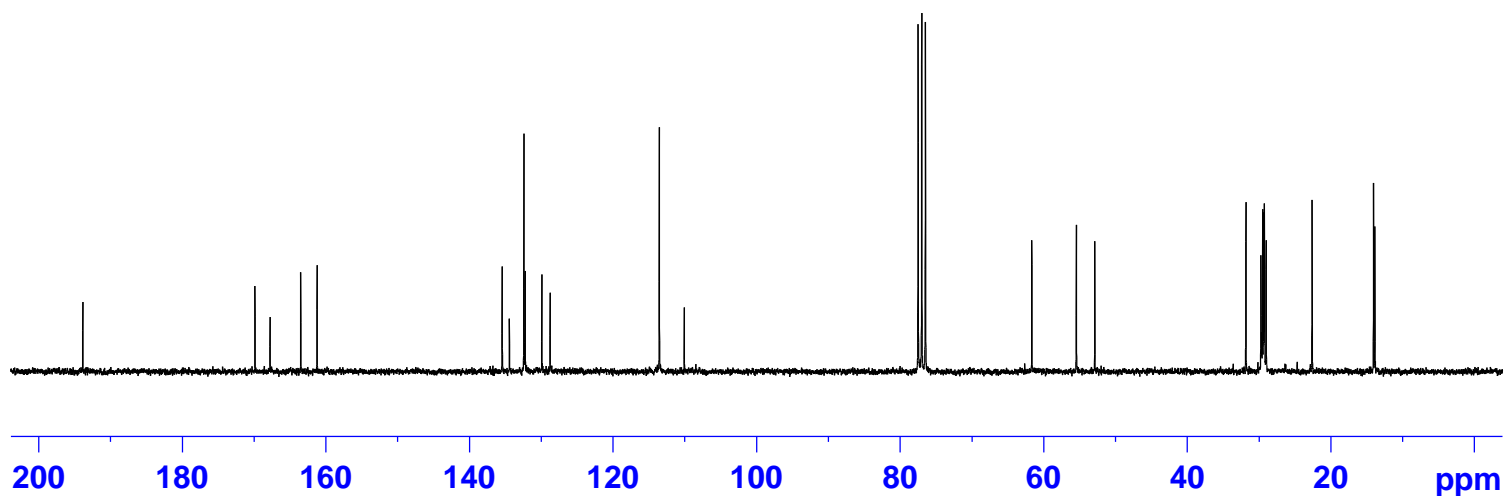
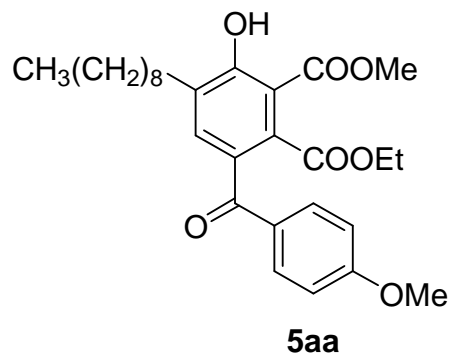
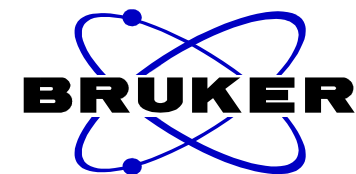
Current Data Parameters  
NAME 090128.u319  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090128  
Time 9.18  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 50.8  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300282 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





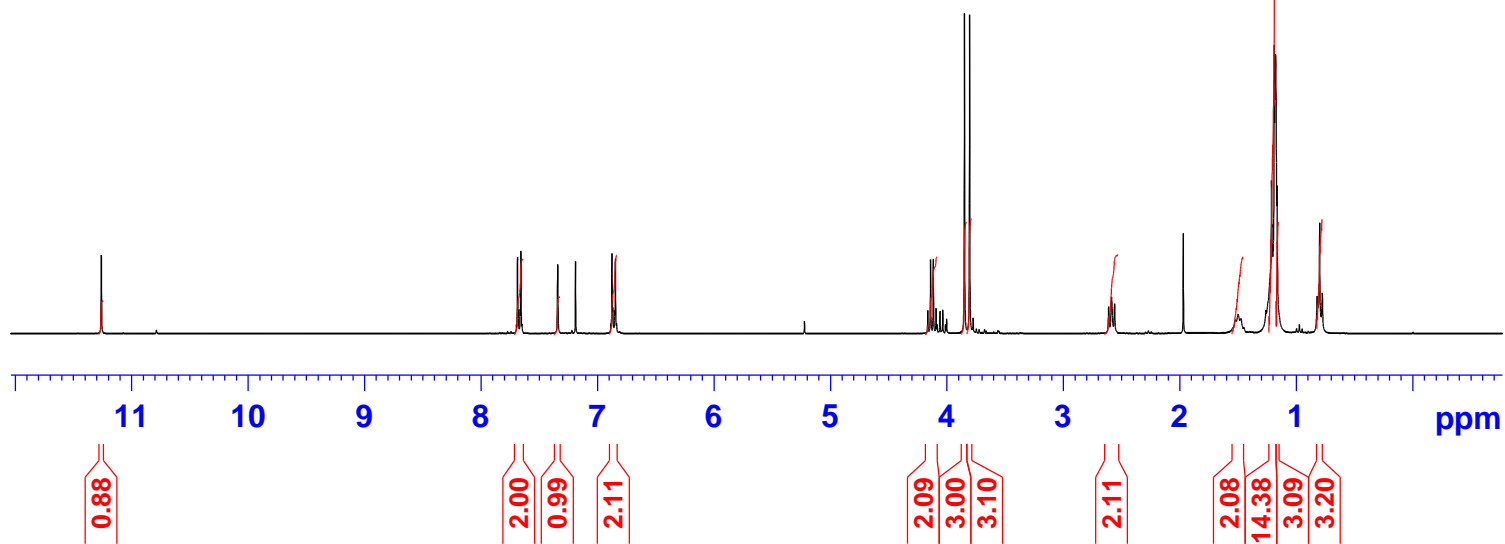
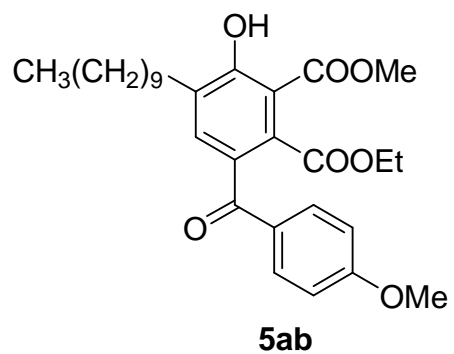
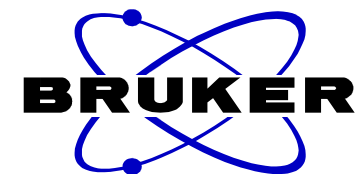
Current Data Parameters  
NAME 090128.206  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090128  
Time 18.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 2050  
DW 33.333 usec  
DE 10.00 usec  
TE 298.1 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 -1.00 dB  
SFO1 62.9015280 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 70.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 -2.50 dB  
SFO2 250.1310005 MHz

F2 - Processing parameters  
SI 32768  
SF 62.8952390 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

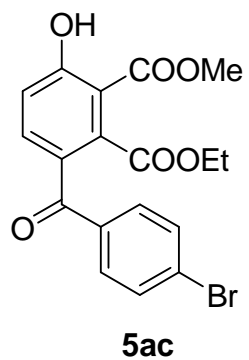
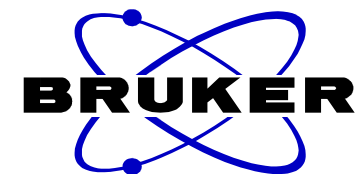


```
Current Data Parameters
NAME      090206.u312
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20090206
Time     9.02
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       114
DW       80.800 usec
DE       10.00 usec
TE       298.2 K
DL       1.00000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     1H
P1       10.00 usec
PL1      0.00 dB
PL1W     11.25325108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300280 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```

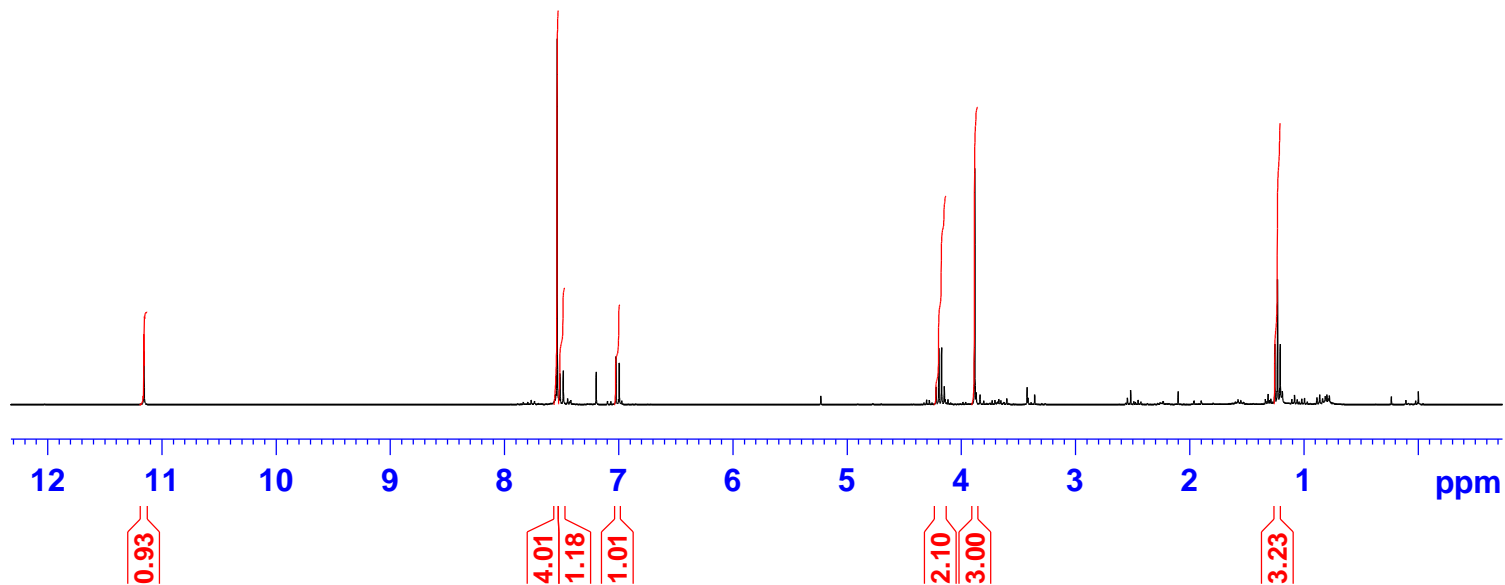


Current Data Parameters  
NAME 090120.u319  
EXPNO 10  
PROCNO 1

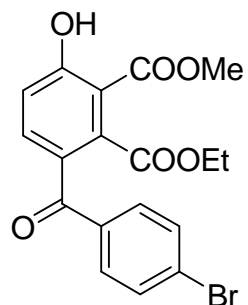
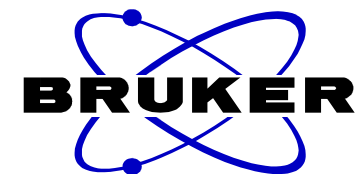
F2 - Acquisition Parameters  
Date\_ 20090120  
Time 11.06  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 128  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

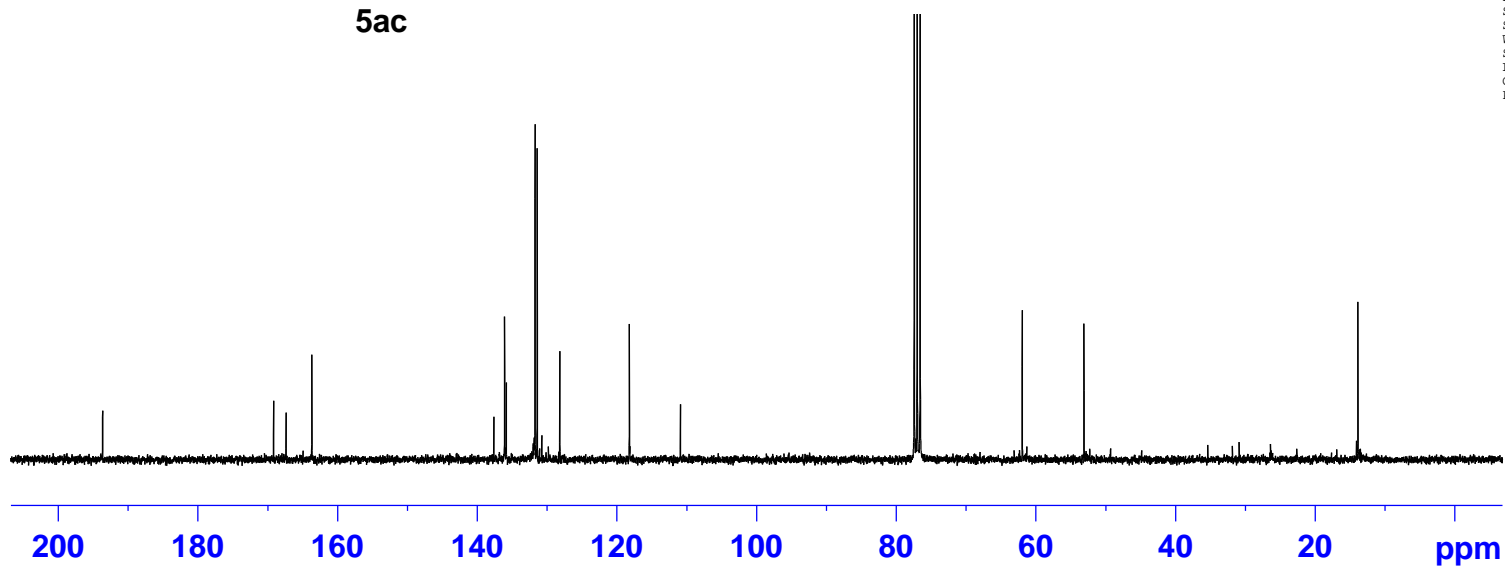
F2 - Processing parameters  
SI 32768  
SF 300.1300253 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







5ac



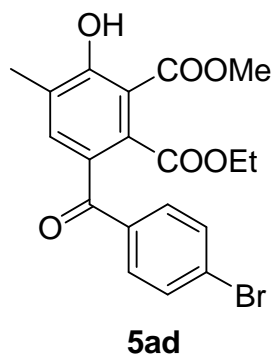
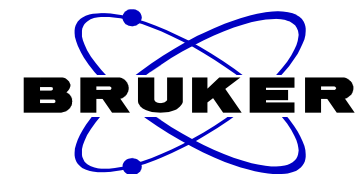
```
Current Data Parameters
NAME          090120.u334
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090121
Time          0.06
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            -0.50 dB
PL1W           33.25691986 W
SFO1           75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         72.00 usec
PL2            0.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W           11.25325108 W
PL12W          0.22453187 W
PL13W          0.22453187 W
SFO2           300.1312005 MHz

F2 - Processing parameters
SI             32768
SF             75.4677490 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

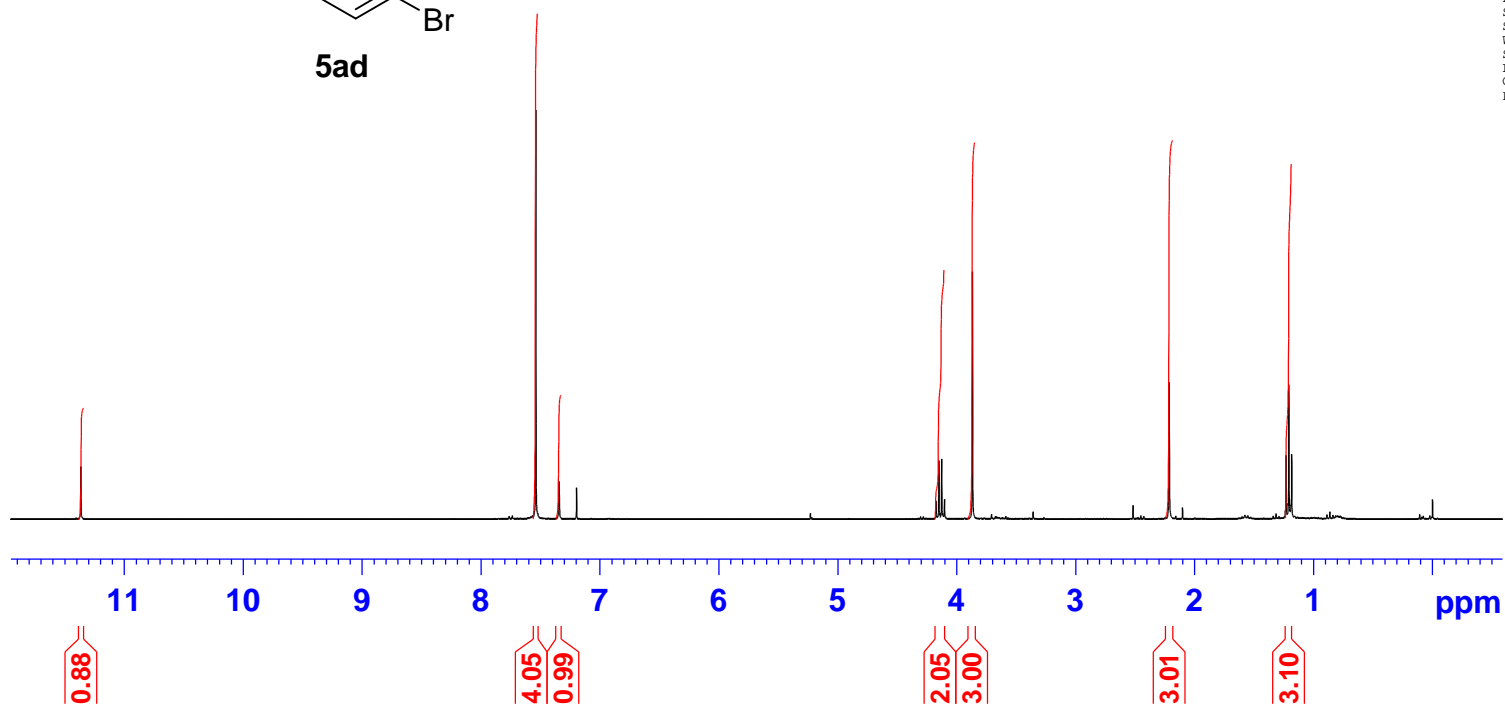


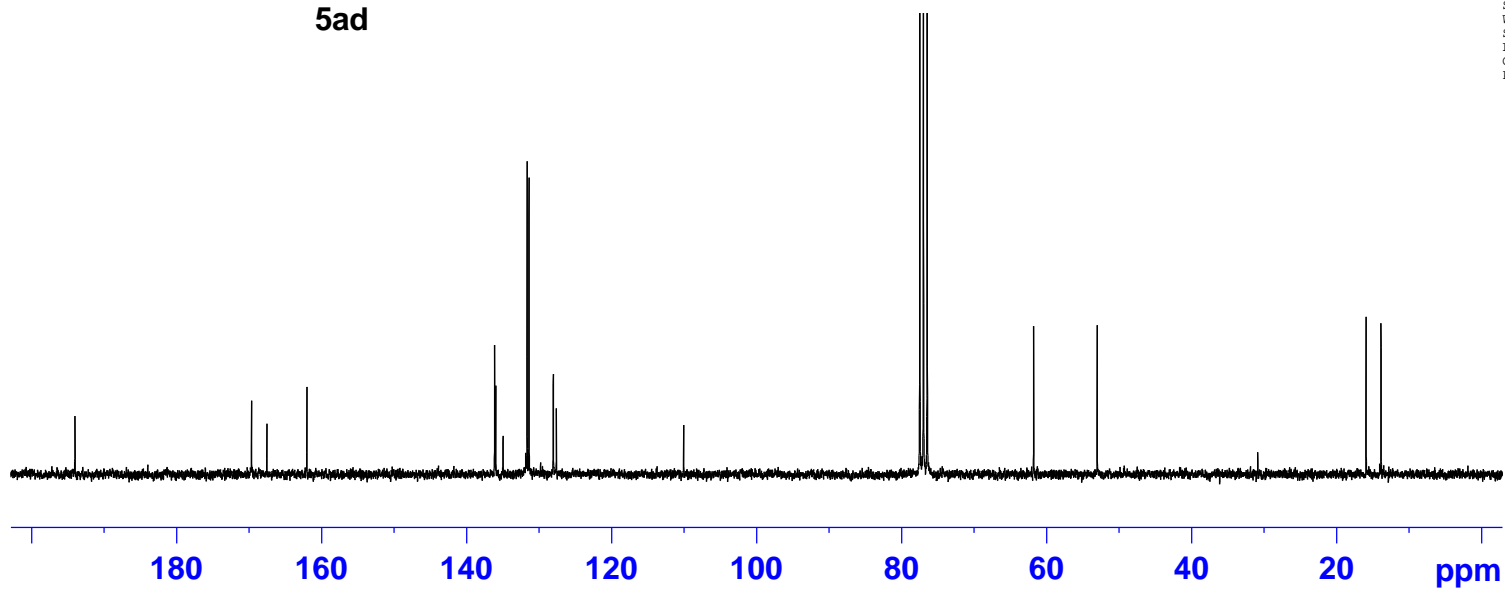
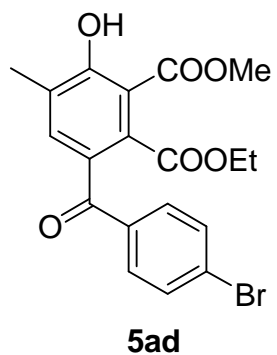
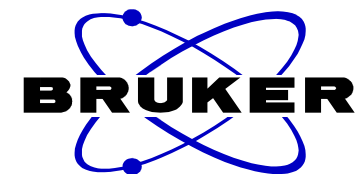
Current Data Parameters  
NAME 090120.u318  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090120  
Time 11.01  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 128  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300255 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





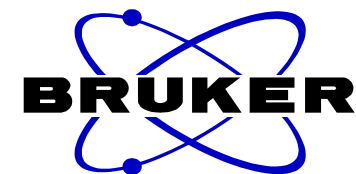
```
Current Data Parameters
NAME      090120.208
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20090121
Time     2.06
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zgpg30
TD       65536
SOLVENT  CDCl3
NS       1024
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1845834 sec
RG       2050
DW       33.333 usec
DE       10.00 usec
TE       298.0 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.89999998 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.00 usec
PL1      -1.00 dB
SFO1     62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     15.00 dB
PL13     15.00 dB
PL2      -2.50 dB
SFO2     250.1310005 MHz

F2 - Processing parameters
SI       32768
SF       62.8952390 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

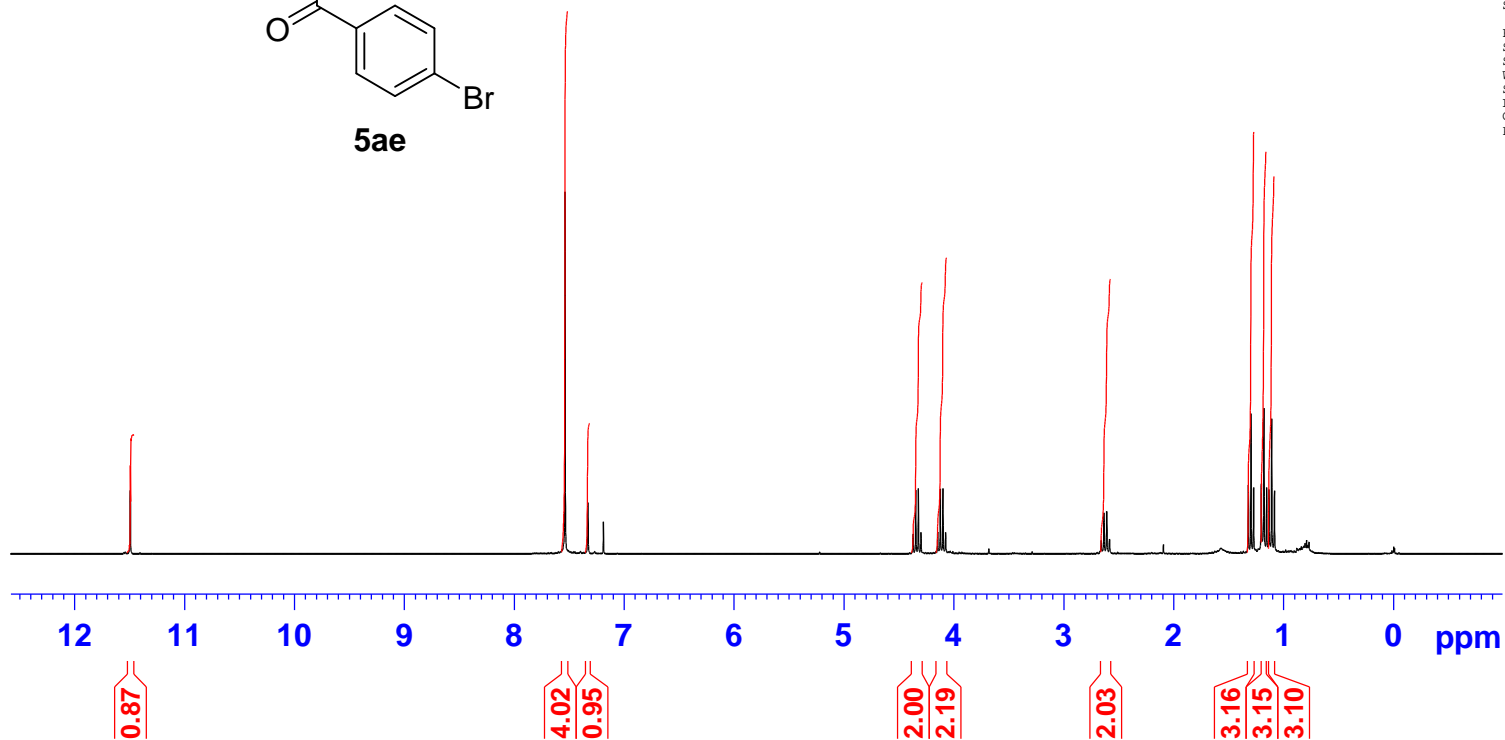
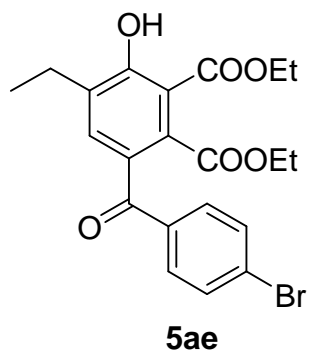


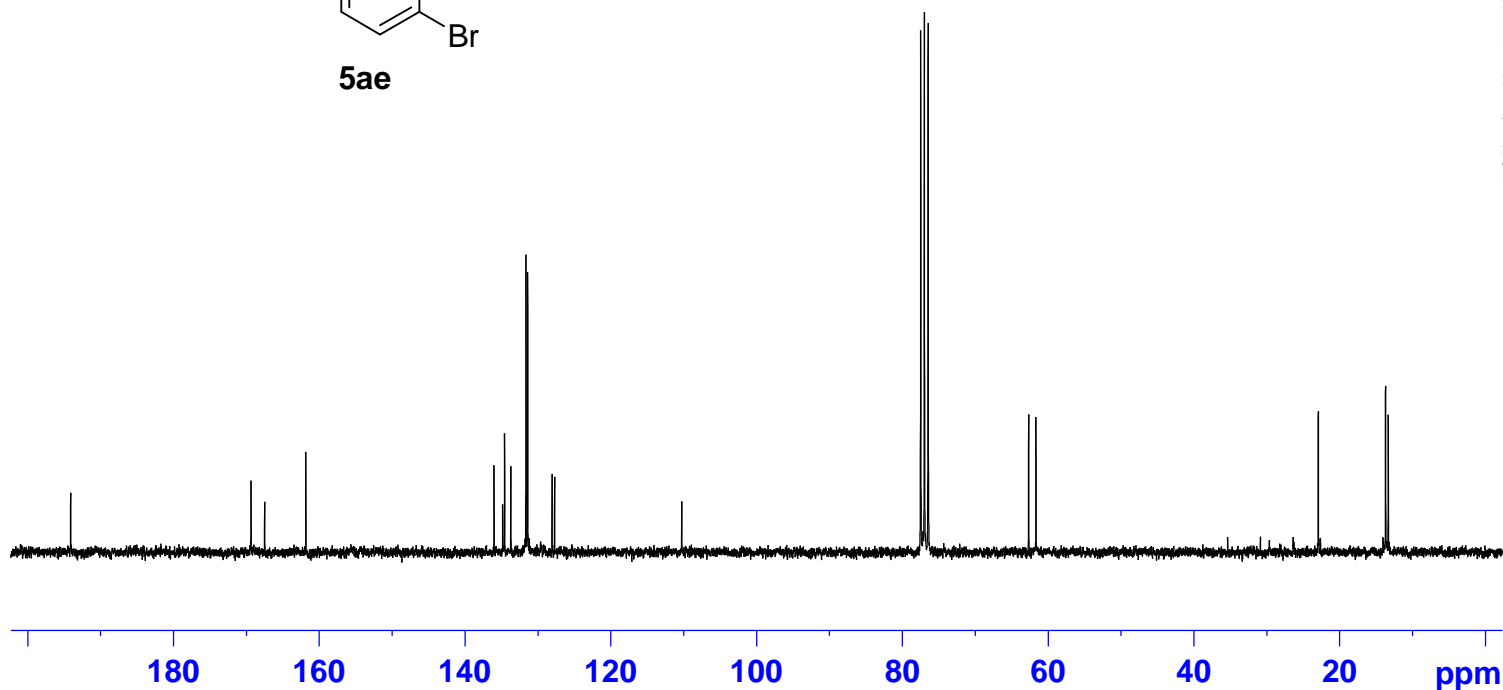
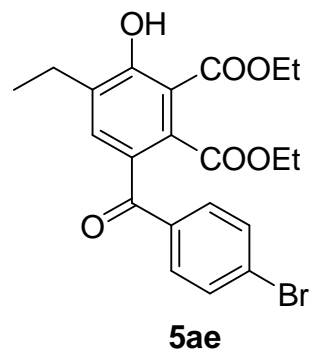
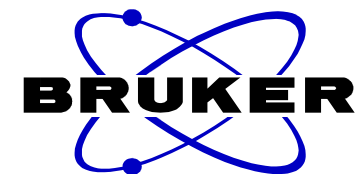
Current Data Parameters  
NAME 090120.u314  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090120  
Time 10.38  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 114  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300279 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





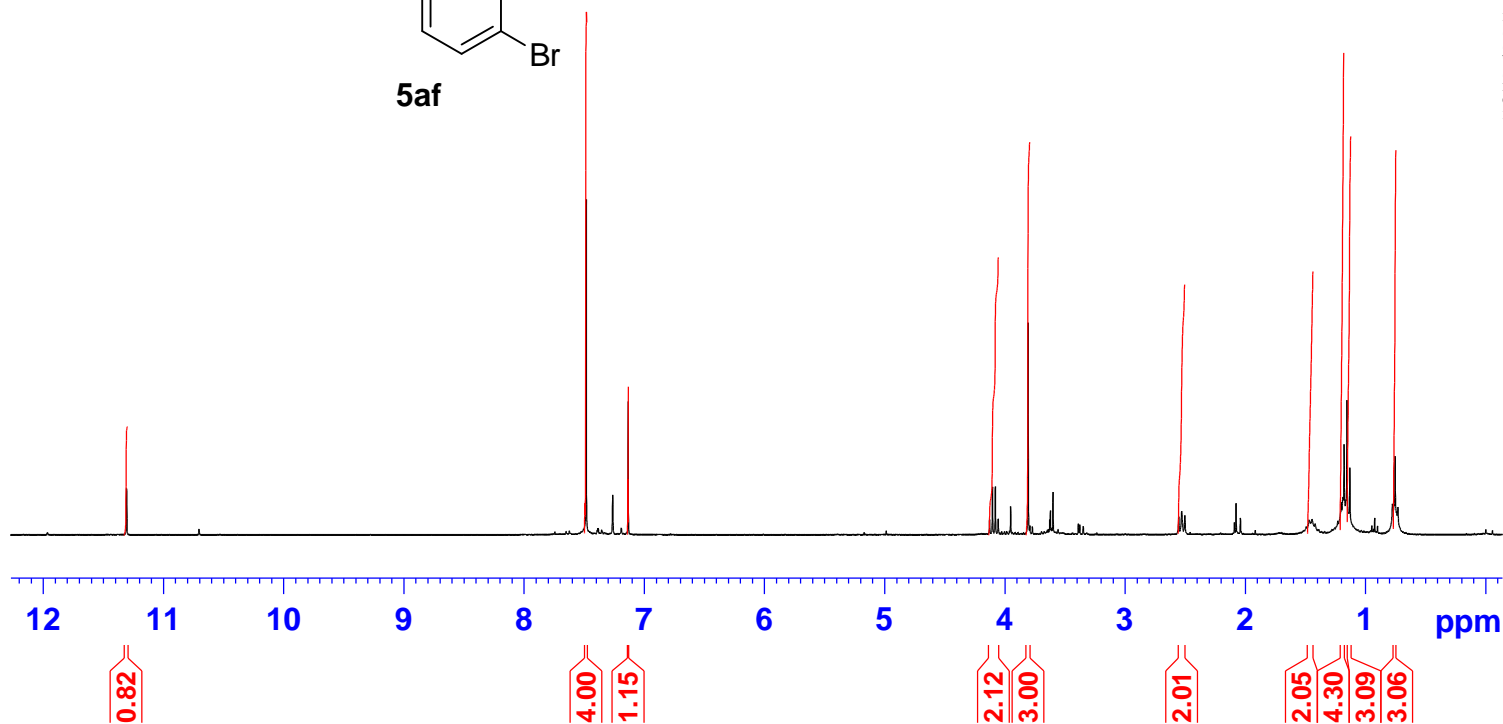
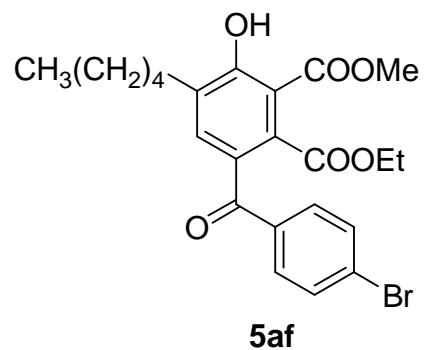
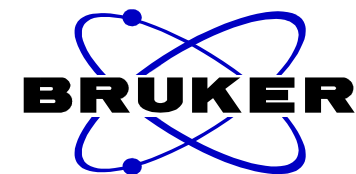
```
Current Data Parameters
NAME          090121.201
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090121
Time          8.41
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            901
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz
```

```
F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

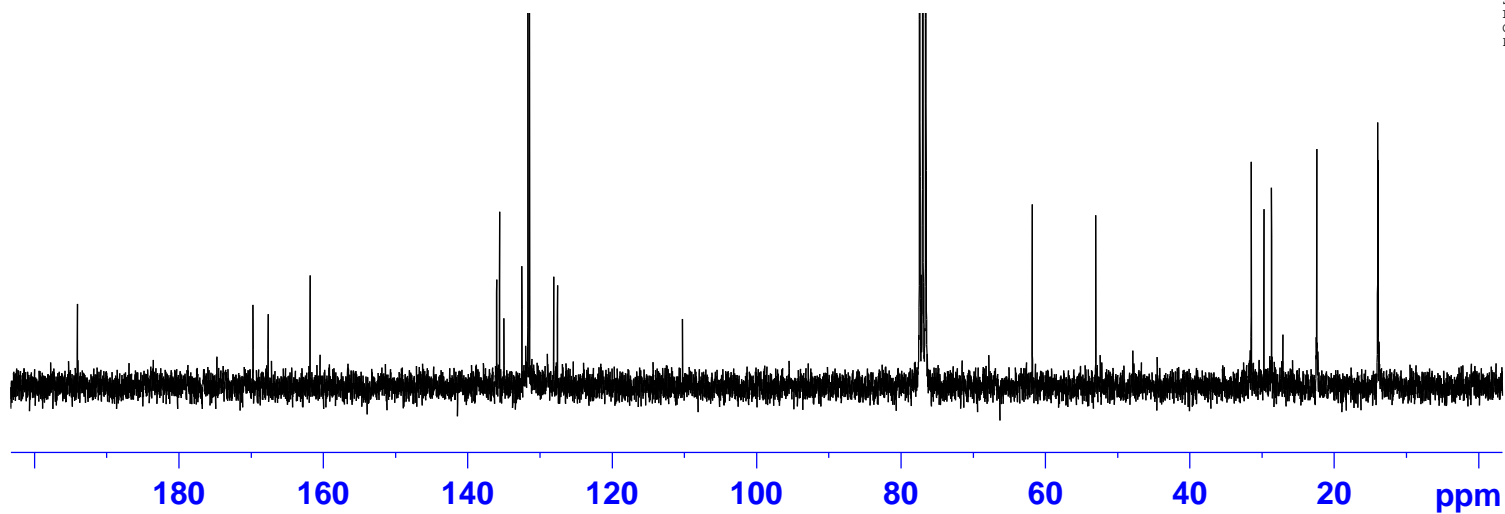
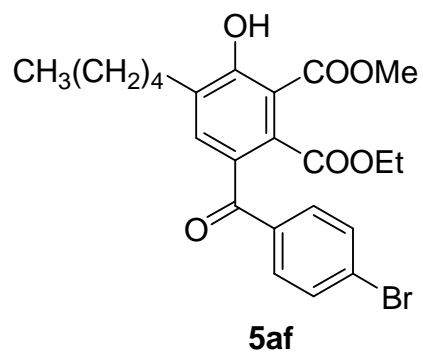
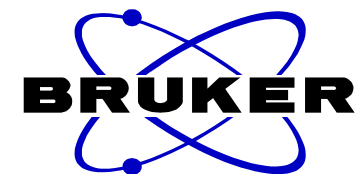


Current Data Parameters  
NAME 090129.u309  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090129  
Time 9.53  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 161  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
DL 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300445 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



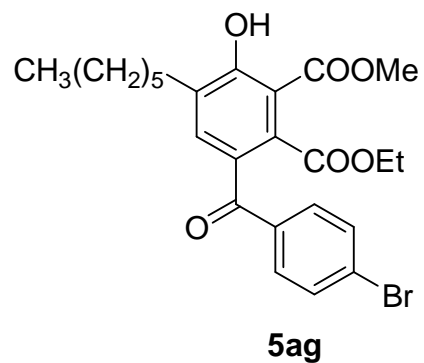
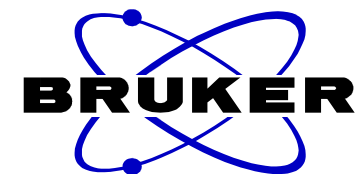
```
Current Data Parameters
NAME          090129.u340
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090129
Time          18.07
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1300
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.25691986 W
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         72.00 usec
PL2           0.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W          11.25325108 W
PL12W         0.22453187 W
PL13W         0.22453187 W
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

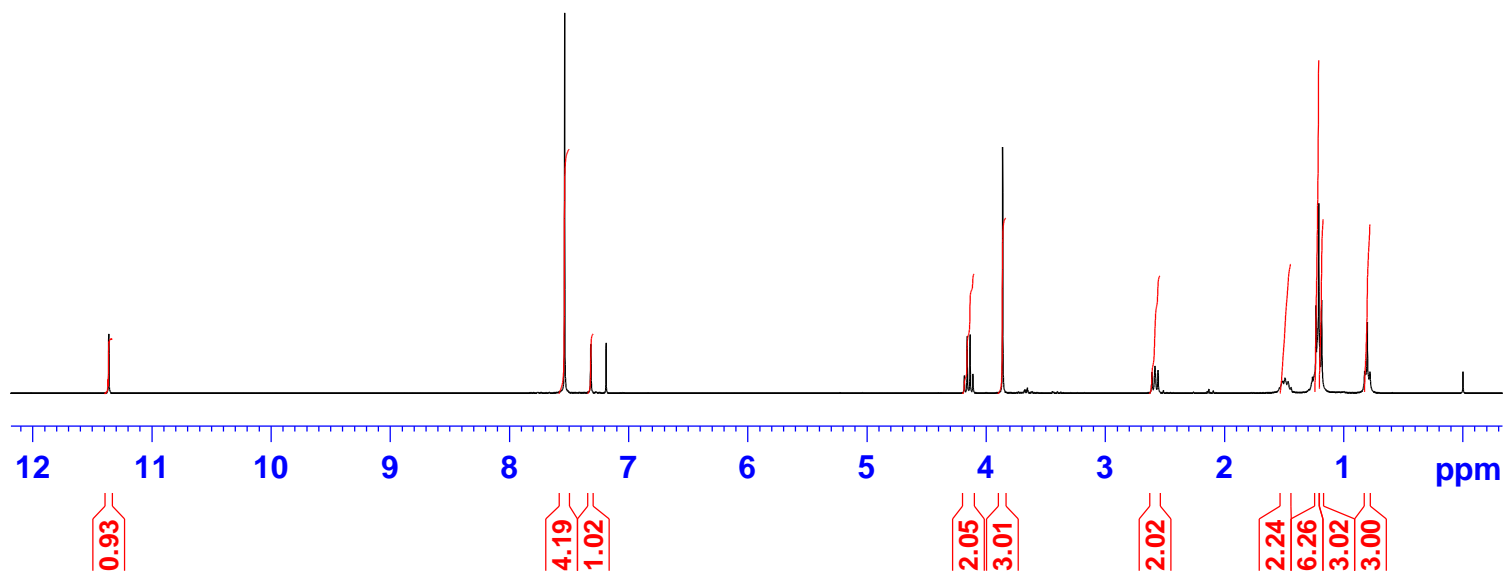


Current Data Parameters  
NAME 090204.u313  
EXPNO 10  
PROCNO 1

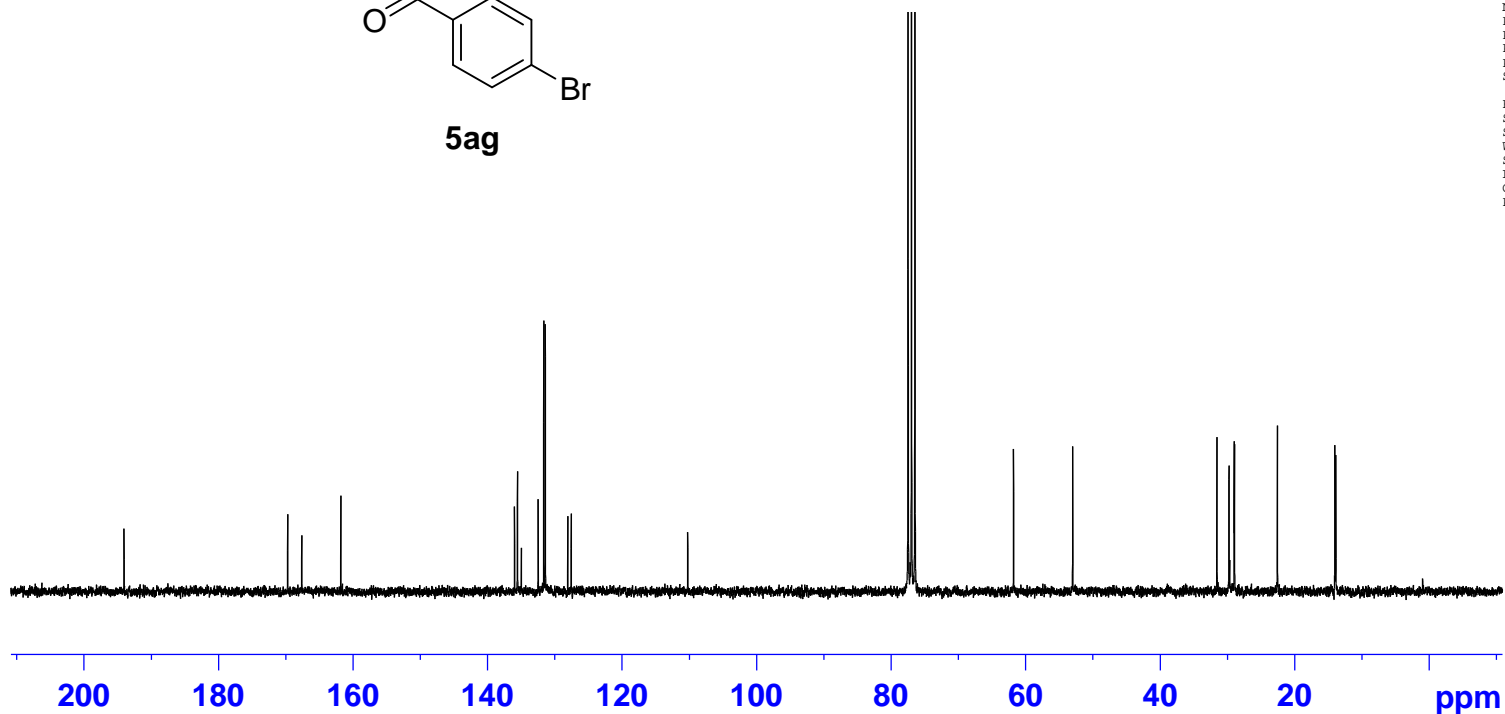
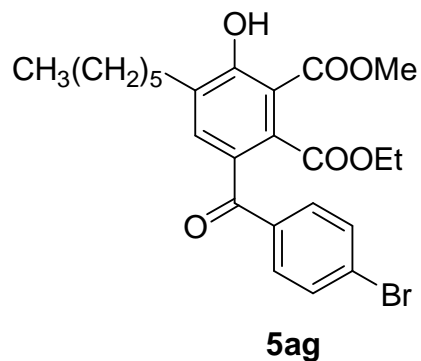
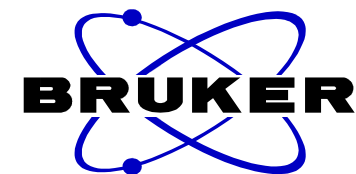
F2 - Acquisition Parameters  
Date\_ 20090204  
Time 9.25  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 128  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300280 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







```

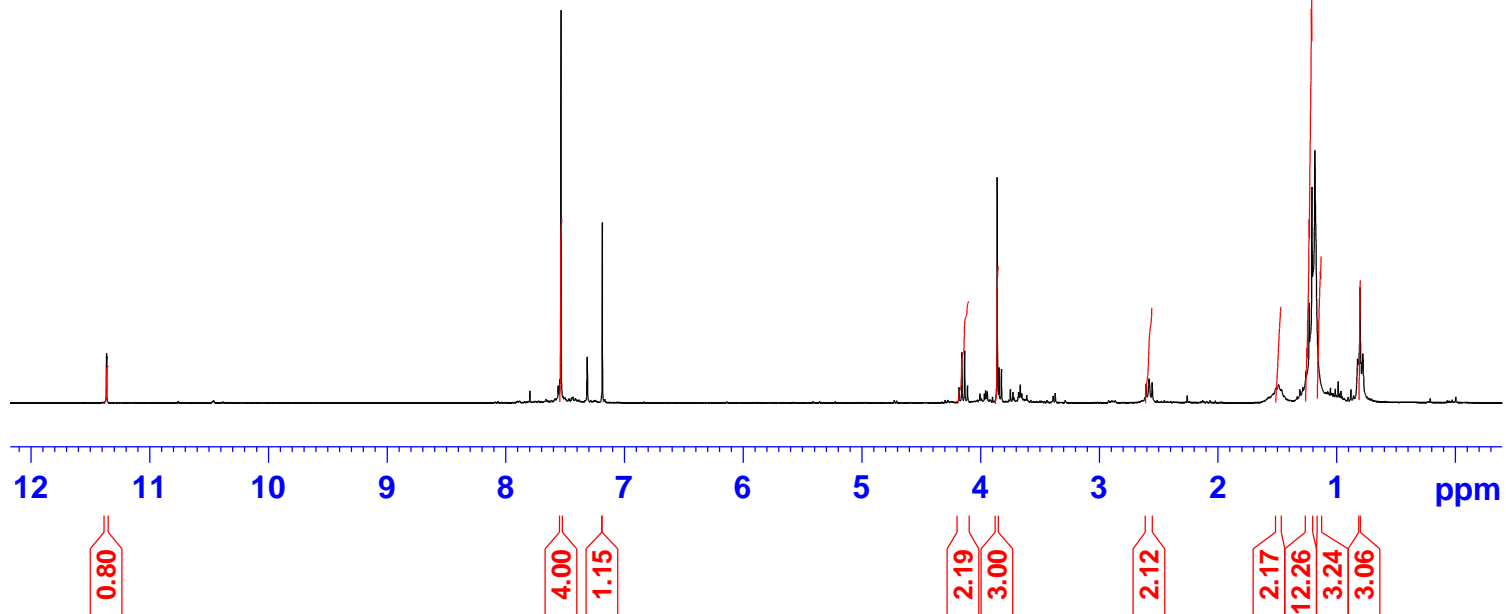
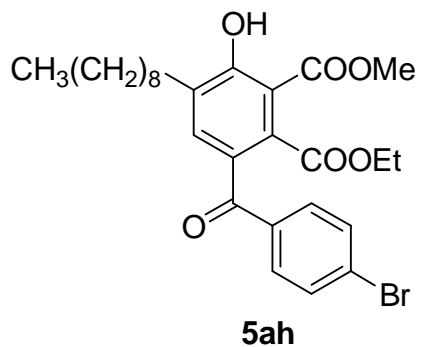
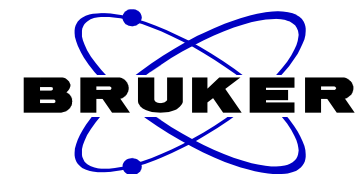
Current Data Parameters
NAME          090204.209
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090205
Time          7.05
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.0 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1          62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         70.00 usec
PL12          15.00 dB
PL13          15.00 dB
PL2           -2.50 dB
SFO2          250.1310005 MHz

F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

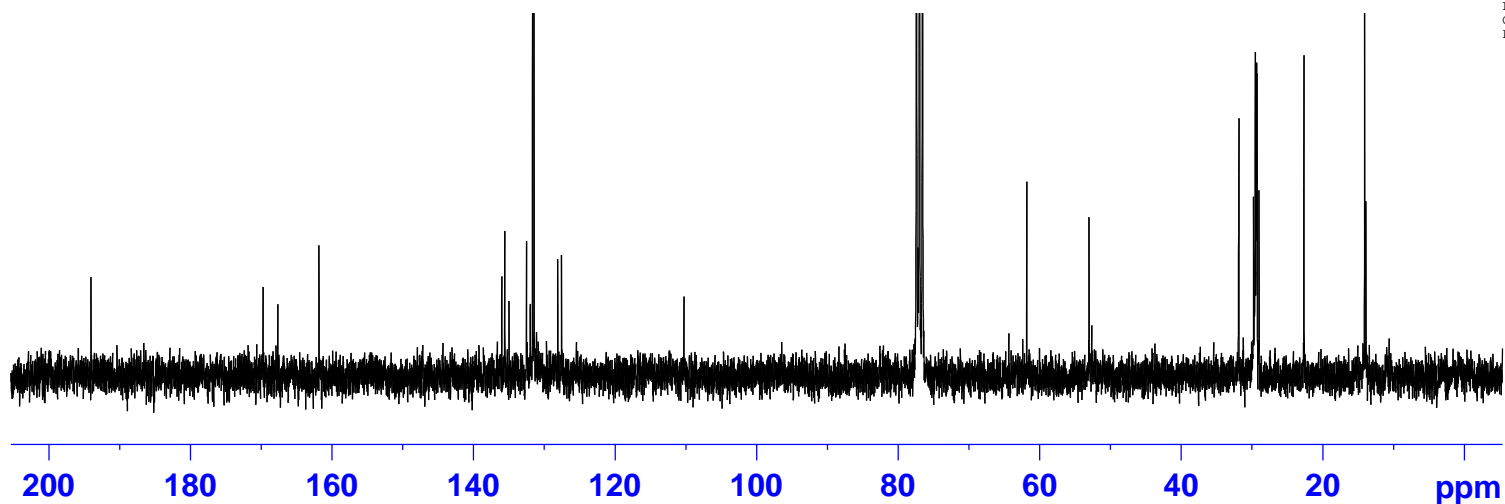
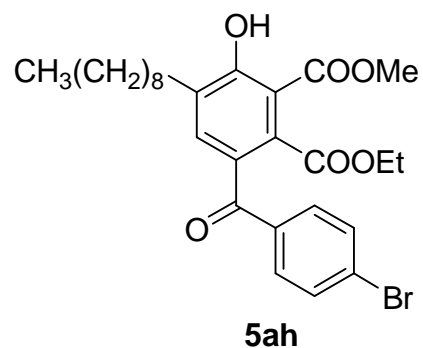
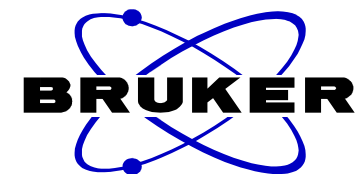


Current Data Parameters  
NAME 090129.u311  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090129  
Time 10.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 144  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300281 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



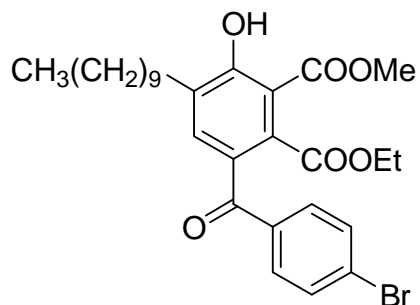
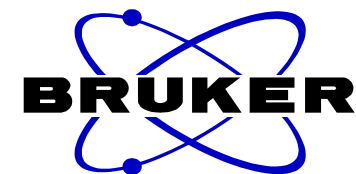
```
Current Data Parameters
NAME          090129.u345
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090130
Time          3.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1300
DS            4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            10.00 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

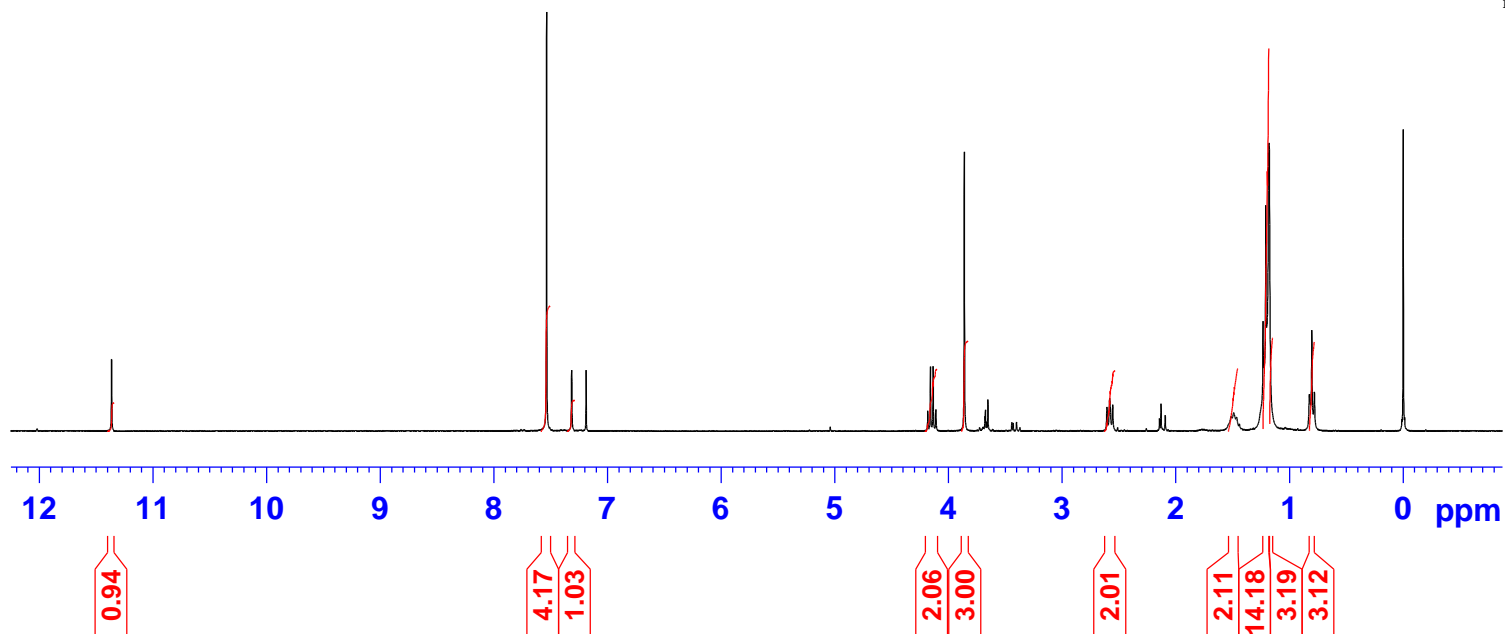
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.25691986 W
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         72.00 usec
PL2           0.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W          11.25325108 W
PL12W         0.22453187 W
PL13W         0.22453187 W
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



5ai

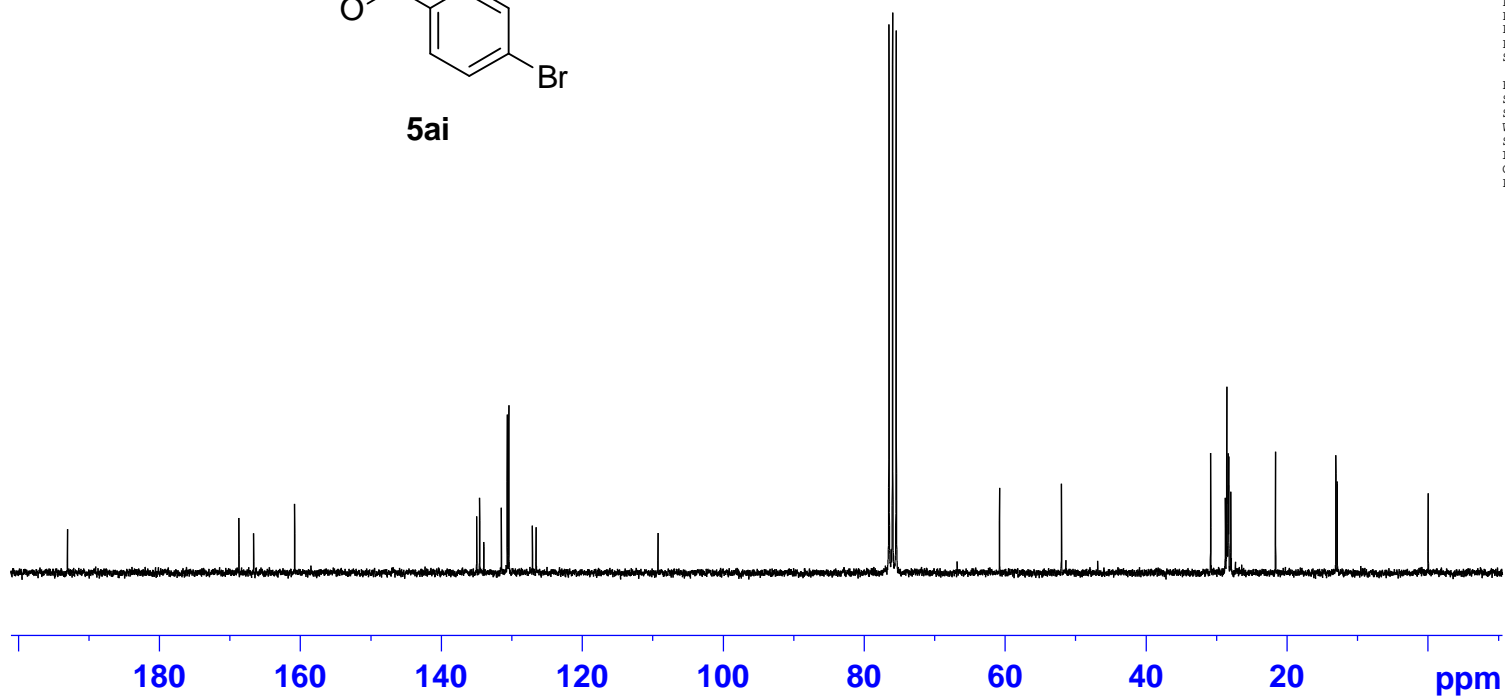
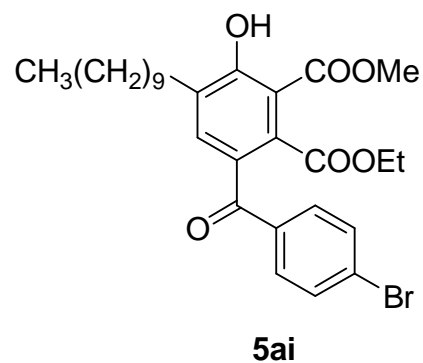
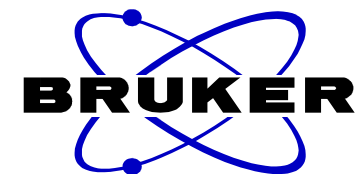


Current Data Parameters  
NAME 090204.u312  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090204  
Time 9.19  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 101  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300280 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



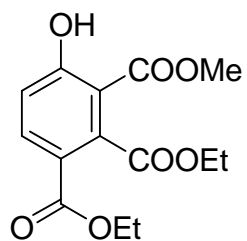
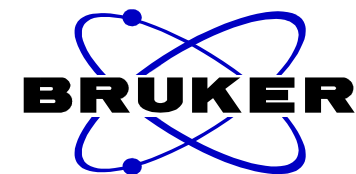
```
Current Data Parameters
NAME          090205.207
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090205
Time          23.36
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1500
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            297.9 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

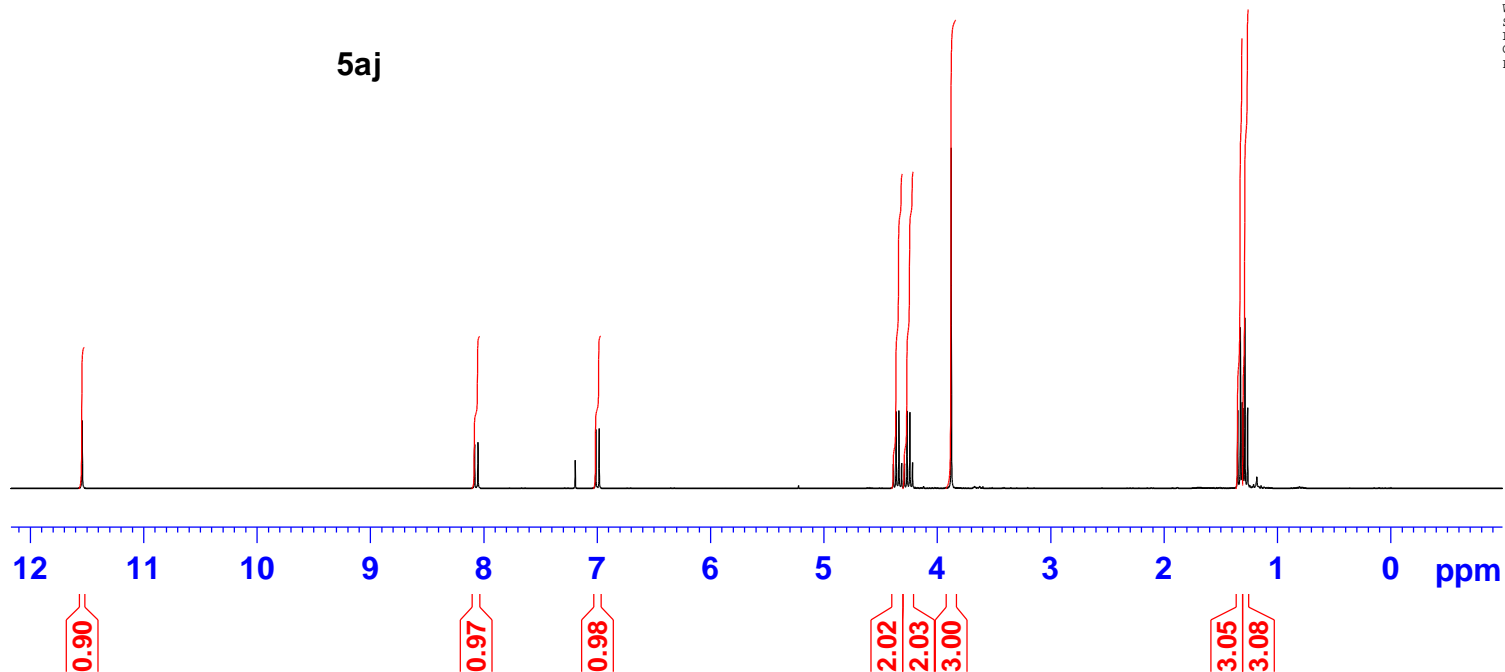
===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            -1.00 dB
SFO1           62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         70.00 usec
PL12           15.00 dB
PL13           15.00 dB
PL2            -2.50 dB
SFO2           250.1310005 MHz

F2 - Processing parameters
SI             32768
SF             62.8953018 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



5aj

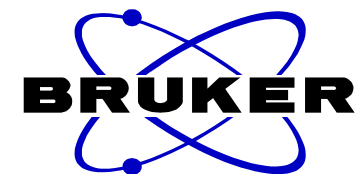


```
Current Data Parameters
NAME      090225.u314
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20090225
Time     9.32
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       114
DW       80.800 usec
DE       10.00 usec
TE       298.2 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       10.00 usec
PL1      0.00 dB
PL1W     11.25325108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300265 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```



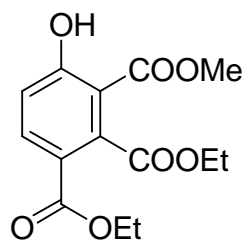
```
Current Data Parameters
NAME          090226.201
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20090226
Time          7.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            512
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.3 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

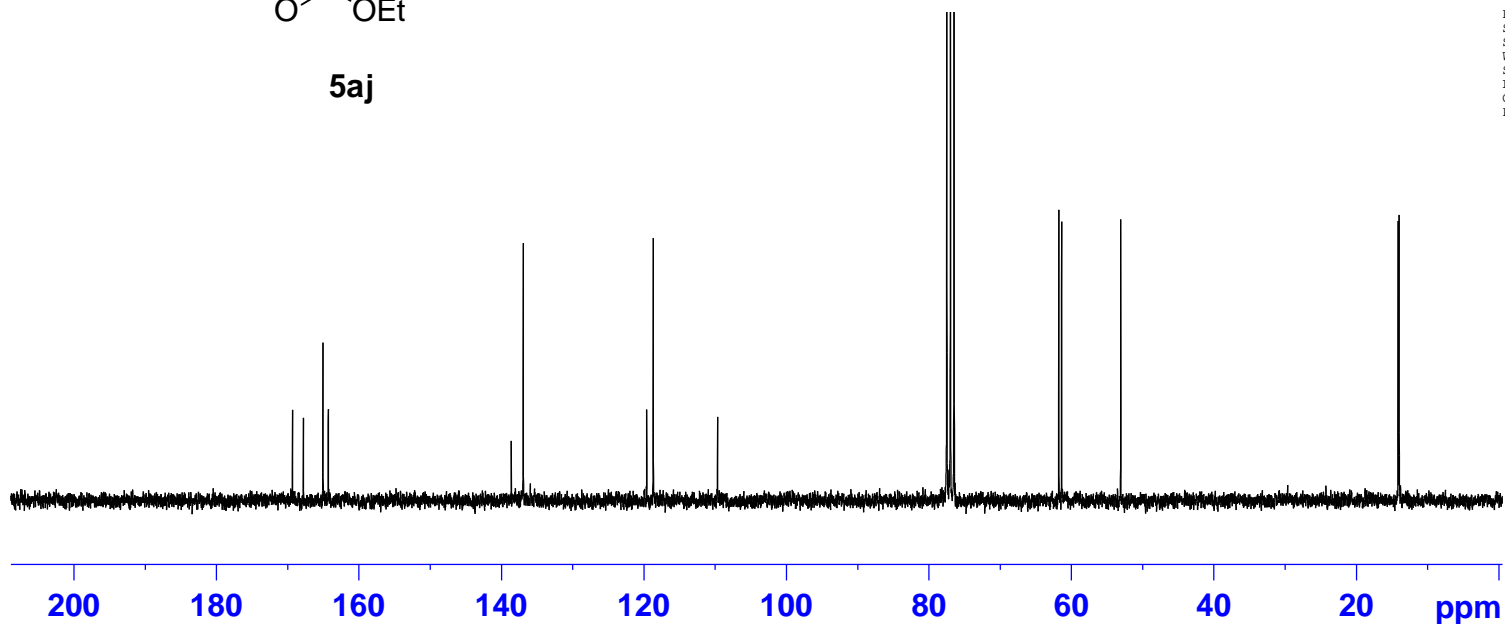
```
===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -1.00 dB
SFO1         62.9015280 MHz
```

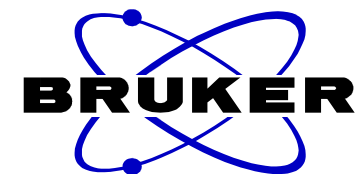
```
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         15.00 dB
PL13         15.00 dB
PL2          -2.50 dB
SFO2         250.1310005 MHz
```

```
F2 - Processing parameters
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```



5aj



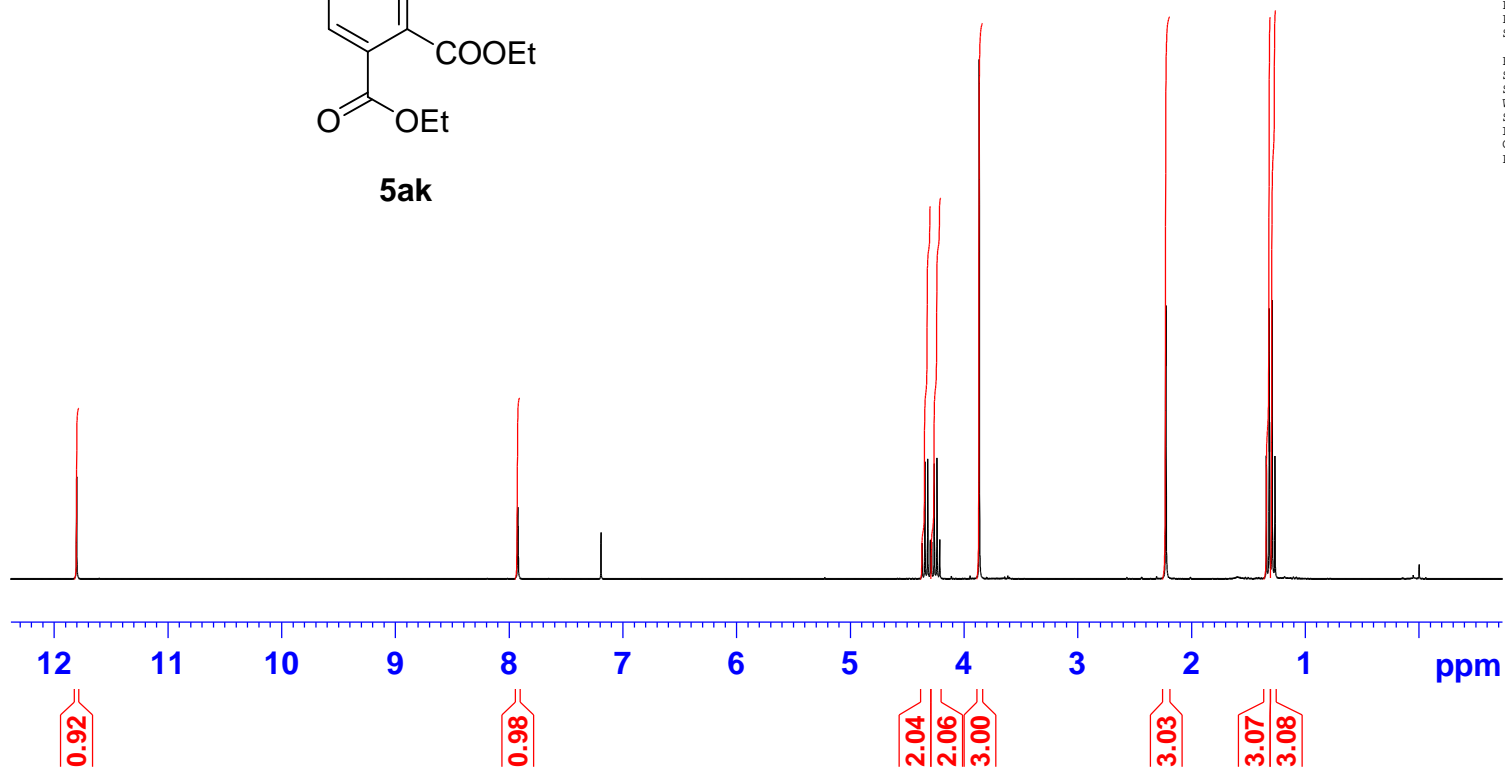
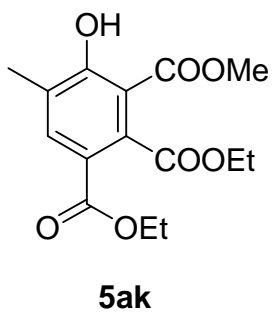


Current Data Parameters  
NAME 090220.u315  
EXPNO 10  
PROCNO 1

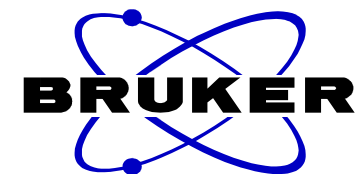
F2 - Acquisition Parameters  
Date\_ 20090220  
Time 9.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 114  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300268 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







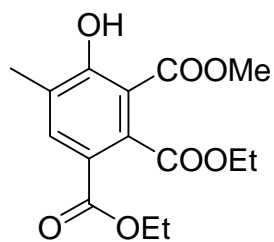
```
Current Data Parameters
NAME          090220.215
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20090222
Time          1.15
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1500
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            297.9 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1
```

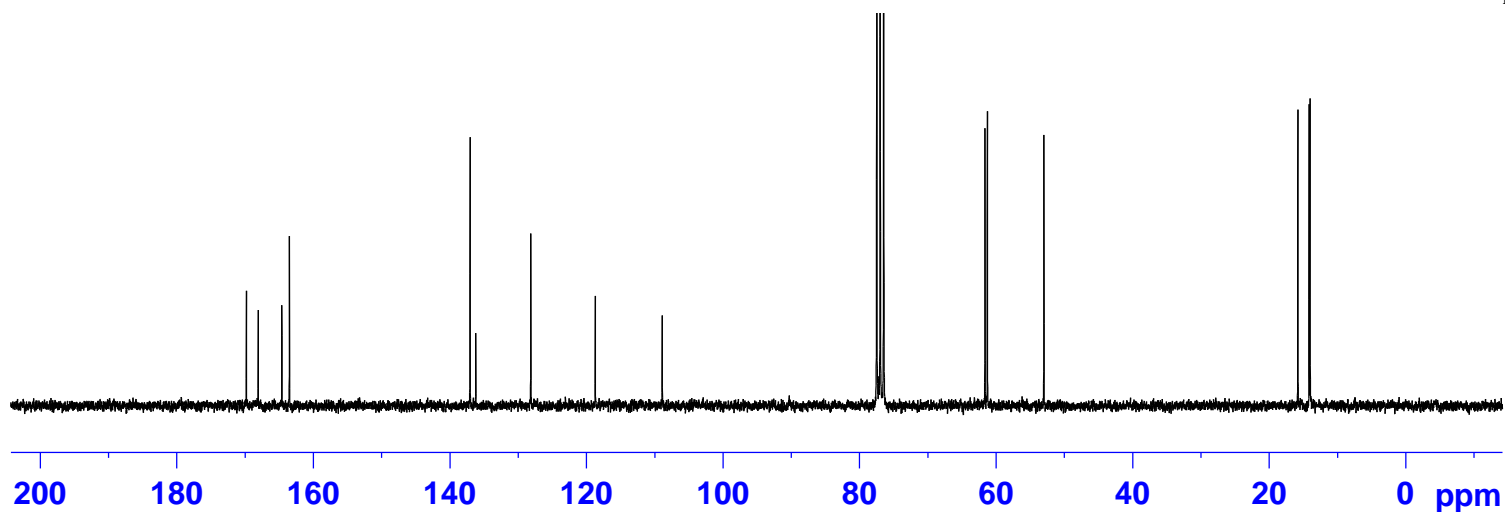
```
===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            -1.00 dB
SFO1           62.9015280 MHz
```

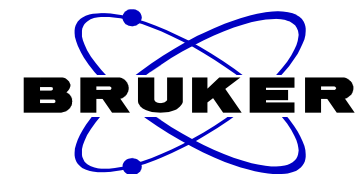
```
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         70.00 usec
PL12           15.00 dB
PL13           15.00 dB
PL2            -2.50 dB
SFO2           250.1310005 MHz
```

```
F2 - Processing parameters
SI             32768
SF             62.8952390 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



5ak



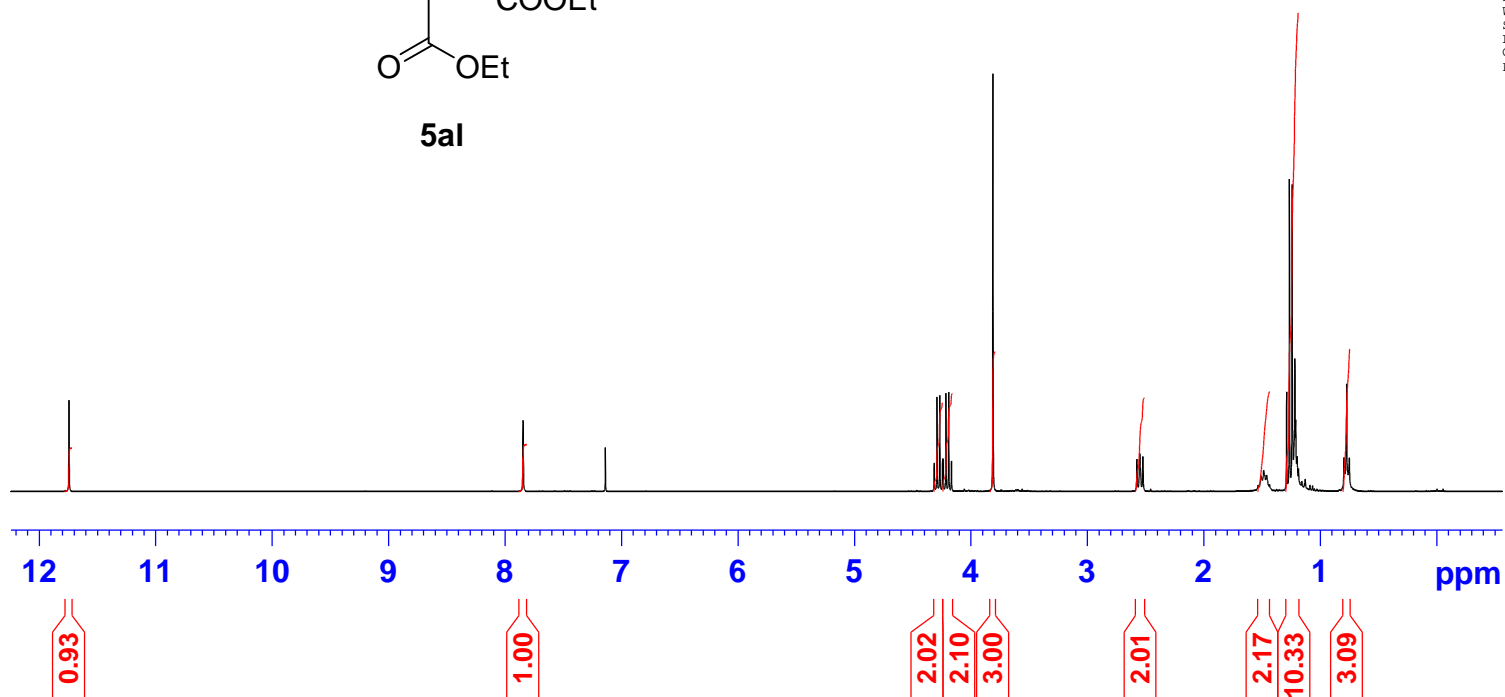
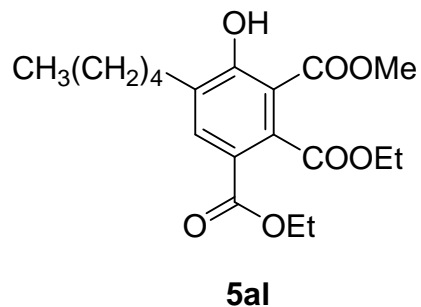


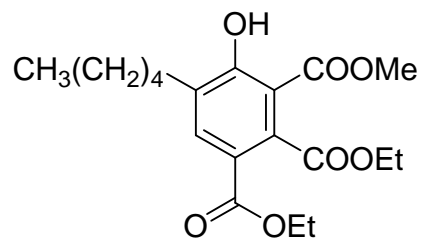
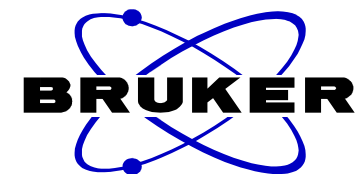
Current Data Parameters  
NAME 090225.u310  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090225  
Time 9.07  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.094423 Hz  
AQ 5.2953587 sec  
RG 90.5  
DW 80.800 usec  
DE 10.00 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

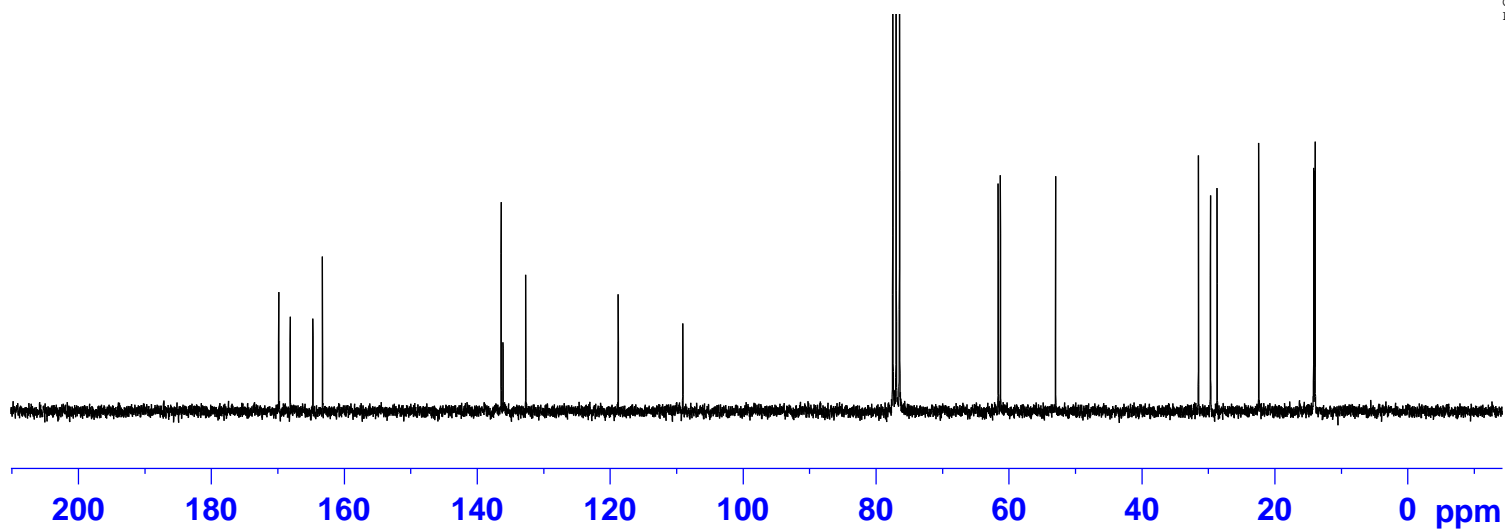
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 11.25325108 W  
SFO1 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300430 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





5al



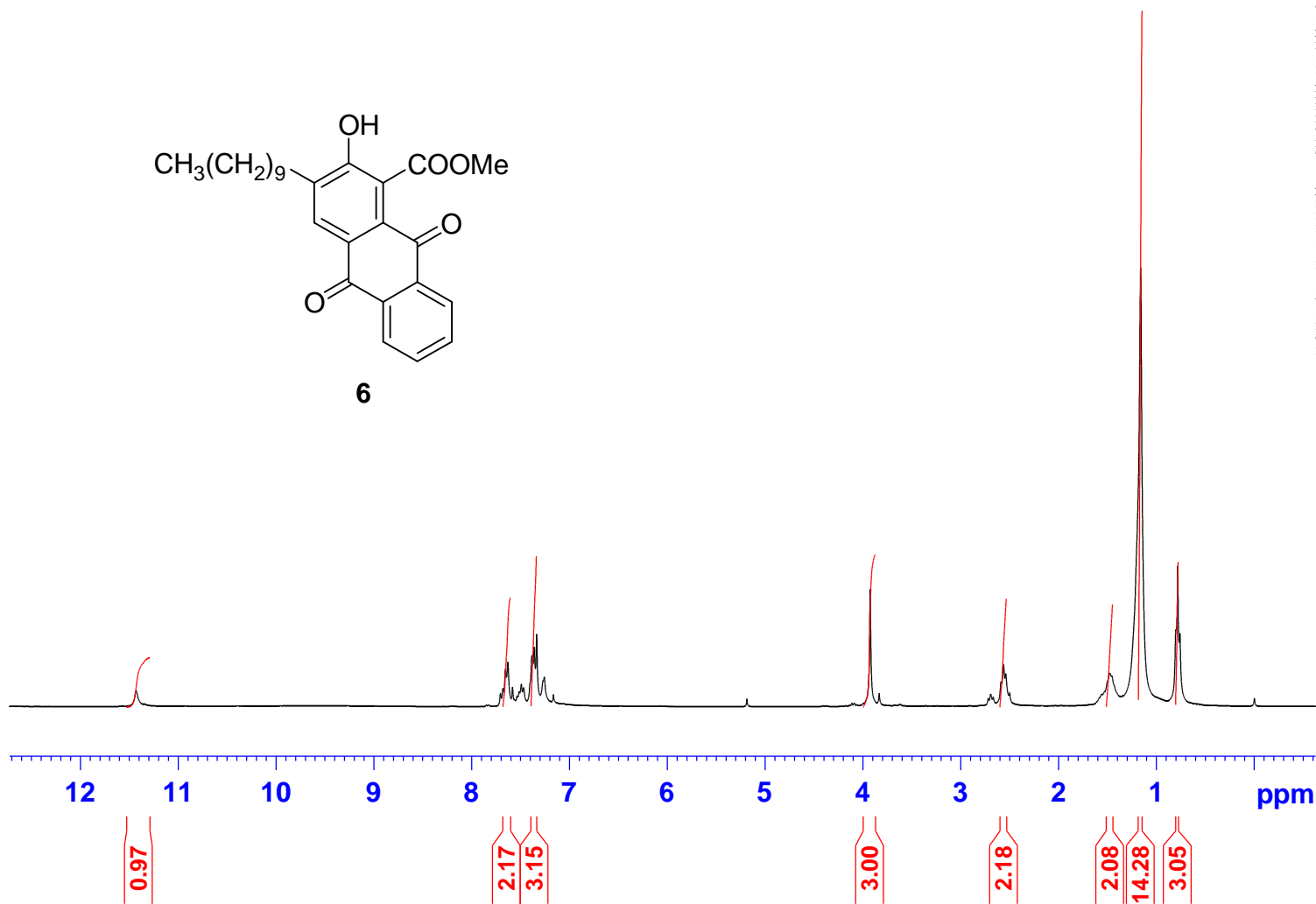
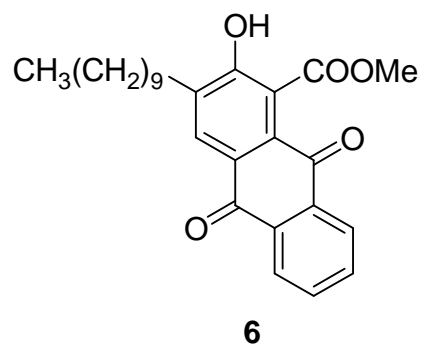
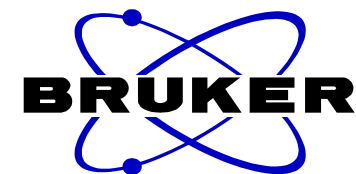
```
Current Data Parameters
NAME          090225.211
EXPNO         20
PROCNO        1

F2 - Acquisition Parameters
Date_         20090226
Time          4.26
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            512
DS            4
SWH           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1845834 sec
RG            2050
DW            33.333 usec
DE            10.00 usec
TE            298.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            -1.00 dB
SFO1           62.9015280 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         70.00 usec
PL12           15.00 dB
PL13           15.00 dB
PL2            -2.50 dB
SFO2           250.1310005 MHz
```

```
F2 - Processing parameters
SI             32768
SF            62.8952390 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



```
Current Data Parameters
NAME          090210.u316
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20090210
Time          9.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           6188.119 Hz
FIDRES        0.094423 Hz
AQ            5.2953587 sec
RG            22.6
DW            80.800 usec
DE            10.00 usec
TE            298.2 K
DL            1.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            10.00 usec
PL1           0.00 dB
PL1W          11.25325108 W
SFO1          300.1318534 MHz

F2 - Processing parameters
SI            32768
SF            300.1300379 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```