

Diversity-Oriented Synthesis of 1-Hydroxy-2,4-benzodioxanes by Regioselective [3+3] Cyclocondensations of 1,3-Bis(silyloxy)-1,3-butadienes with 3-Alkoxy- and 3-Silyloxy-2-alkoxycarbonyl-2-en-1-ones

Mohanad Shkooor,^a Abdolmajid Riahi,^{a, b} Olumide Fatunsin,^a Ibrar Hussain,^a Mirza A. Yawer,^a Mathias Lubbe,^a Stefanie Reim,^a Helmut Reinke,^b Christine Fischer,^b and Peter Langer*^{a, b}

^a Institut für Chemie, Universität Rostock,, Albert-Einstein-Str. 3a, 18059 Rostock, Germany. Fax: 00 49 381 49864112; Tel: 0049 381 4986410; E-mail: peter.langer@uni-rostock.de

^b Leibniz-Institut für Katalyse e. V. an der Universität Rostock,, Albert-Einstein-Str. 29a, 18059 Rostock, Germany.

Supporting Information

15

Experimental Section

General Comments. All solvents were dried by standard methods and all reactions were carried out under an inert atmosphere. For ¹H and ¹³C NMR spectra the deuterated 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.

General procedure for the synthesis of 1-hydroxy-2,4-benzodioxanes 4a-o. To a CH₂Cl₂ solution (2 mL / 1.0 mmol of **3**) of **3** (1.0 equiv.) was added **1** (1.0 equiv.) and,

subsequently, TiCl_4 (1.0 equiv.) at $-78\text{ }^\circ\text{C}$. The temperature of the solution was allowed to warm to $20\text{ }^\circ\text{C}$ 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 later was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were dried (Na_2SO_4), filtered and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, *n*-heptane / EtOAc) to give product **4**.

1-Ethyl 3-methyl 4-hydroxy-2,6-dimethylisophthalate (4a): Starting with **3** (0.489 g, 2.0 mmol) and **1a** (0.521 g, 2.0 mmol), **4a** was isolated after chromatography (silica gel, heptanes/EtOAc) as an orange oil (0.149 g, 30%). ^1H NMR (CDCl_3 , 250 MHz): $\delta = 1.36$ (t, $^3J = 7.1$ Hz, 3H, OCH_2CH_3), 2.25 (s, 3H, CH_3), 2.44 (s, 3H, CH_3), 3.94 (s, 3H, OCH_3), 4.36 (q, $^3J = 7.1$ Hz, 2H, OCH_2CH_3), 6.69 (s, 1H, CH_{Ar}), 11.23 (s, 1H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 14.2$ (OCH_2CH_3), 20.0, 20.2 (CH_3), 52.2 (OCH_3), 61.1 (OCH_2CH_3), 110.4 (C_{Ar}), 116.9 (CH_{Ar}), 128.6, 137.8, 141.8 (C_{Ar}), 162.6 (COH), 169.7 (CO), 171.7 (CO). IR (neat, cm^{-1}): $\tilde{\nu} = 3421$ (br, w), 2983 (m), 2956 (m), 1725 (s), 1666 (s), 1606 (m), 1579 (m), 1444 (s), 1360 (s), 1324 (s), 1259 (s), 1232 (s), 1185 (s), 1115 (s), 1053 (m), 1036 (m). MS (GC/MS, 70 eV): m/z (%) = 252 (M^+ , 27), 220 (100), 207 (28), 175 (61). Anal.: calcd. for $\text{C}_{13}\text{H}_{16}\text{O}_5$ (252.26): C, 61.90; H, 6.39. Found: C, 61.69; H, 6.51.

Diethyl 4-hydroxy-2,5,6-trimethylisophthalate (4b): Starting with **3** (0.305 g, 1.25 mmol) and **1b** (0.325 g, 1.25 mmol), **4b** was isolated after chromatography (silica gel, heptanes/EtOAc) as a slightly yellow solid (167 mg, 48%), mp. = $66\text{-}67\text{ }^\circ\text{C}$. ^1H NMR (CDCl_3 , 250 MHz): $\delta = 1.44 - 1.35$ (m, 6H, $2\text{OCH}_2\text{CH}_3$), 2.17 (s, 3H, PhCH_3), 2.21 (s, 3H, PhCH_3), 2.44 (s, 3H, PhCH_3), 4.38 (q, $^3J = 7.0$ Hz, 2H, OCH_2CH_3), 4.43 (q, $^3J = 7.0$ Hz, 2H, OCH_2CH_3), 11.68 (s, 1H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 11.5$ (CH_3), 14.1, 14.2 (OCH_2CH_3), 17.5, 20.1, (CH_3), 61.1 (OCH_2CH_3), 61.7 (OCH_2CH_3), 110.0, 123.2, 128.6, 133.8, 139.2 (C_{Ar}), 160.7 (COH), 170.1, 171.9, (CO). IR (Nujol, cm^{-1}): $\tilde{\nu} = 1722$ (s), 1656 (s), 1598 (m), 1576 (m). MS (EI, 70 eV): m/z (%) = 280 (M^+ , 37), 235 (60), 234 (100), 206 (87), 189 (34), 178 (48), 177 (26). Anal.: calcd. for $\text{C}_{15}\text{H}_{20}\text{O}_5$ (280.32): C, 64.27; H, 7.19. Found: C, 64.04; H, 7.40.

Diethyl 5-ethyl-4-hydroxy-2,6-dimethylisophthalate (4c): Starting with **3** (0.305 g, 1.25 mmol) and **1c** (0.378 g, 1.25 mmol), **4c** was isolated after chromatography (silica gel, heptanes/EtOAc) as colourless oil (0.181 g, 49%). ¹H NMR (CDCl₃, 250 MHz): δ = 1.10 (t, ³J = 7.6 Hz, 3H, CH₂CH₃), 1.44 – 1.36 (m, 6H, 2 OCH₂CH₃), 2.24 (s, 3H, PhCH₃), 2.43 (s, 3H, PhCH₃), 2.70 (q, ³J = 7.6 Hz, 2H, CH₂CH₃), 4.38 (q, ³J = 7.0 Hz, 2H, OCH₂CH₃), 4.42 (q, ³J = 7.0 Hz, 2H, OCH₂CH₃), 11.63 (s, 1H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.0, 14.1, 14.2, 16.6, 19.3, 20.2 (CH₃), 61.1, 61.7 (OCH₂CH₃), 110.2, 128.8, 129.2, 134.0, 138.5 (C_{Ar}), 160.6 (COH), 170.6, 171.8 (CO). IR (neat, cm⁻¹): $\tilde{\nu}$ = 3422 (br, m), 2938 (s), 2937 (s), 2970 (m), 2875 (m), 1727(s), 1656 (s), 1600 (s), 1574 (s). MS (EI, 70 eV): *m/z* (%) = 294 (M⁺, 20), 249 (25), 248 (37), 220 (100), 203 (15), 192 (31). Anal.: calcd. for C₁₆H₂₂O₅ (294.34): C, 65.29; H, 7.53. Found: C, 65.27; H, 7.68.

1-Ethyl 3-methyl 5-hexyl-4-hydroxy-2,6-dimethylisophthalate (4d). Starting with **3** (0.488 g, 2.0 mmol) and **1d** (0.757 g, 2.2 mmol), **4d** was isolated after chromatography (silica gel, heptanes/EtOAc) as a colourless viscous oil (0.236 g, 35%). ¹H NMR (250 MHz, CDCl₃): δ = 0.81 (t(br), ³J = 7.4 Hz, 3 H, CH₃), 1.19–1.25 (m, 8 H, 4 CH₂), 1.31 (t, ³J = 7.6 Hz, 3 H, OCH₂CH₃), 2.16 (s, 3 H, PhCH₃), 2.34 (s, 3 H, PhCH₃), 2.59 (t, ³J = 7.4 Hz, 2 H, PhCH₂), 3.88 (s, 3 H, OCH₃), 4.32 (q, ³J = 7.6 Hz, 2 H, OCH₂CH₃), 11.40 (s, 1 H, OH). ¹³C NMR (75 MHz, CDCl₃): δ = 15.2, 15.3, 18.0, 21.0 (CH₃), 23.7, 27.3, 29.7, 32.4, 34.4 (CH₂), 53.6 (OCH₃), 62.3 (OCH₂CH₃), 111.1, 129.3, 130.0, 135.1, 140.0 (C_{Ar}), 161.9 (COH), 171.8, 173.5 (CO). IR (Neat, cm⁻¹): $\tilde{\nu}$ = 2962 (m), 1723 (m), 1663 (m), 1439 (m), 1394 (m), 1229 (s), 1194 (s), 1151 (s), 1101 (m), 1033 (m), 844 (w), 723 (w). GC-MS (EI, 70 eV): *m/z* (%) = 336 ([M⁺], 49), 289 (100), 276 (87), 259 (19), 234 (54), 206 (31), 187 (43), 178 (13), 159 (6), 91 (9), 77 (8), 43 (7). HRMS (EI): Calcd. for C₁₉H₂₈O₅: 336.19313; found: 336.19263.

1-Ethyl 3-methyl 4-hydroxy-2,6-dimethyl-5-nonylisophthalate (4e). Starting with **3** (0.488 g, 2.0 mmol) and **1e** (0.850 g, 2.2 mmol), **4e** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.302 g, 40%). ¹H NMR (250 MHz, CDCl₃): δ = 0.80 (t, ³J = 7.3 Hz, 3 H, (CH₂)₈CH₃), 1.19 - 1.24 (m, 14 H, 7 CH₂), 1.31 (t, ³J = 7.3 Hz, 3 H, OCH₂CH₃), 2.15 (s, 3 H, PhCH₃), 2.34 (s, 3 H, PhCH₃), 2.57 (t, ³J = 6.7 Hz, 2 H, PhCH₂), 3.87 (s, 3 H, OCH₃), 4.30 (q, ³J = 7.1 Hz, 2 H, OCH₂CH₃), 11.48 (s, 1 H, OH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 14.0, 14.1, 16.8, 20.0$ (CH_3), 22.6, 26.1, 28.8, 29.3, 29.5, 29.6, 29.9, 31.8 (CH_2), 52.2 (OCH_3), 61.3 (OCH_2), 109.9 (CCOOCH_3), 128.1 (COOC_2H_5), 128.8, 133.8, 138.8 (C_{Ar}), 160.7 (COH), 170.6, 172.3 (CO). IR (neat, cm^{-1}): $\nu = 2953$ (w), 2922 (m), 2852 (w), 1725 (m), 1657 (m), 1598 (w), 1572 (w), 1439 (m), 1411 (w), 1362 (m), 1328 (m), 1267 (m), 1217 (s), 1192 (m), 1155 (m), 1123 (m), 1094 (w), 1073 (w), 1033 (m), 972 (w), 858 (w), 809 (m), 756 (w), 684 (w), 662 (w), 580 (w), 541 (w). GC-MS (EI, 70 eV): m/z (%) = 378 ($[\text{M}]^+$, 52), 333 (20), 332 (118), 331 (100), 329 (17), 318 (29), 301 (10), 275 (14), 235 (10), 234 (60), 233 (38), 206 (41), 187 (24). HRMS (EI): Calcd. for $\text{C}_{22}\text{H}_{34}\text{O}_5$ ($[\text{M}]^+$): 378.240088; found: 378.239837.

10

1-Ethyl 3-methyl 5-decyl-4-hydroxy-2,6-dimethylisophthalate (4f). Starting with **3** (0.488 g, 2.0 mmol) and **1** (0.881 g, 2.2 mmol), **4f** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.321 g, 41%). ^1H NMR (250 MHz, CDCl_3): $\delta = 0.80$ (t, $^3J = 7.3$ Hz, 3 H, $(\text{CH}_2)_9\text{CH}_3$), 1.13 - 1.23 (m, 16 H, 8 CH_2), 1.30 (t, $^3J = 7.3$ Hz, 3 H, OCH_2CH_3), 2.15 (s, 3 H, PhCH_3), 2.33 (s, 3 H, PhCH_3), 2.57 (t, $^3J = 6.7$ Hz, 2 H, PhCH_2), 3.87 (s, 3 H, OCH_3), 4.30 (q, $^3J = 7.2$ Hz, 2 H, OCH_2CH_3), 11.47 (s, 1 H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 14.0, 14.1, 16.8, 20.0$ (CH_3), 22.6, 26.1, 28.8, 29.3, 29.5, 29.6, 29.6, 29.9, 31.8 (CH_2), 52.2 (OCH_3), 61.3 (OCH_2), 109.9 (CCOOCH_3), 128.1 (COOC_2H_5), 128.8, 133.8, 138.9 (C_{Ar}), 160.7 (COH), 170.6 172.3 (CO). IR (neat, cm^{-1}): $\nu = 2953$ (w), 2922 (m), 2852 (w), 1725 (m), 1657 (m), 1597 (w), 1572 (w), 1439 (m), 1409 (w), 1362 (m), 1328 (m), 1264 (m), 1216 (s), 1191 (m), 1155 (m), 1123 (m), 1093 (w), 1070 (w), 1033 (m), 959 (w), 858 (w), 808 (m), 761 (w), 721(w), 685 (w), 662 (w), 580 (w), 539 (w). GC-MS (EI, 70 eV): m/z (%) = 392 ($[\text{M}]^+$, 42), 347 (23), 346 (24), 345 (100), 343 (22), 332 (17), 315 (10), 275 (13), 261 (10), 235 (10), 234 (51), 233 (38), 206 (40), 177 (11), 43 (13). HRMS (EI): Calcd. for $\text{C}_{23}\text{H}_{36}\text{O}_5$ ($[\text{M}]^+$): 392.25573; found: 392.255696.

25

3-Ethyl 5-methyl 6-hydroxy-2,4,4'-trimethylbiphenyl-3,5-dicarboxylate (4g). Starting with **3** (0.488 g, 2.0 mmol) and **1g** (0.771 g, 2.2 mmol), **4g** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.260 g, 38%). ^1H NMR (250 MHz, CDCl_3): $\delta = 1.24$ (t, $^3J = 7.0$ Hz, 3 H, OCH_2CH_3), 1.90 (s, 3H, PhCH_3), 2.27 (s, 3H, PhCH_3), 2.36 (s, 3H, PhCH_3), 3.83 (s, 3 H, OCH_3), 4.26 (q, $^3J = 7.0$ Hz, 2 H,

30

OCH_2CH_3), 6.94 - 6.97 (m, 2 H, CH_{Ar}), 7.02 - 7.08 (m, 2 H, CH_{Ar}), 11.18 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.1, 18.2, 20.1, 21.2 (CH_3), 52.3 (OCH_3), 61.2 (OCH_2), 110.6 ($CCOOCH_3$), 128.8 (C_{Ar}), 129.0 ($CCOOC_2H_5$), 129.2 (2 CH_{Ar}), 129.7 (2 CH_{Ar}), 133.0 (C_{Ar}), 135.8, 137.0, 139.5 (C_{Ar}), 160.1 (COH), 170.1, 171.9 (CO). IR (neat, cm^{-1}): ν = 2954 (w), 2929 (w), 2871 (w), 1721 (m), 1658 (m), 1598 (w), 1568 (w), 1513 (w), 1438 (m), 1363 (w), 1330 (m), 1213 (s), 1203 (s), 1098 (m), 1075 (w), 1035 (m), 958 (w), 923 (w), 842 (w), 821 (w), 809 (m), 760 (w), 729 (w), 710 (w), 686 (w), 666 (w), 611 (w), 580 (w), 539 (w). GC-MS (EI, 70 eV): m/z (%) = 342 ($[M]^+$, 50), 310 (100), 297 (17), 282 (22), 265 (13), 253 (12), 236 (11), 209 (16), 165 (14), 119 (5), 43 (5). HRMS (EI): Calcd. for $C_{20}H_{22}O_5$ ($[M]^+$): 342.14618; found: 342.146101.

3-Ethyl 5-methyl 4'-chloro-6-hydroxy-2,4-dimethyl-biphenyl-3,5-dicarboxylate (4h). Starting with **3** (0.488 g, 2.0 mmol) and **1h** (0.816 g, 2.2 mmol), **4h** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.270 g, 37%). 1H NMR (250 MHz, $CDCl_3$): δ = 1.16 (t, 3J = 6.9 Hz, 3 H, OCH_2CH_3), 1.78 (s, 3 H, $PhCH_3$), 2.27 (s, 3 H, $PhCH_3$), 3.74 (s, 3 H, OCH_3), 4.16 (q, 3J = 6.9 Hz, 2 H, OCH_2CH_3), 6.89 - 6.93 (m, 2 H, 2 CH_{Ar}), 7.02 - 7.08 (m, 2 H, 2 CH_{Ar}), 11.26 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 15.1, 19.1, 21.2 (CH_3), 53.3 (OCH_3), 62.2 (OCH_2), 111.5 ($CCOOCH_3$), 128.7 (C_{Ar}), 129.6 (2 CH_{Ar}), 129.9 ($CCOOC_2H_5$), 132.2 (2 CH_{Ar}), 134.3, 135.5, 137.4, 140.3 (C_{Ar}), 160.0 (COH), 170.8, 172.8 (CO). IR (neat, cm^{-1}): ν = 2954 (w), 2930 (w), 2871 (w), 1722 (m), 1658 (m), 1604 (w), 1591 (w), 1571 (w), 1491 (w), 1438 (m), 1408 (w), 1363 (w), 1329 (m), 1204 (s), 1100 (m), 1086 (m), 1034 (m), 1014 (m), 985 (w), 957 (w), 831 (m), 809 (m), 758 (w), 689 (w), 665 (w), 612 (w), 579 (w). GC-MS (EI, 70 eV): m/z (%) = 364 ($[M]^+$, ^{37}Cl , 12), 362 ($[M]^+$, ^{35}Cl , 39), 332 (^{37}Cl , 29), 331 (23), 330 (^{35}Cl , 100), 317 (15), 302 (18), 285 (12), 274 (8), 165 (14), 128 (6), 86 (9), 43 (5). HRMS (EI): Calcd. for $C_{19}H_{19}ClO_5$ ($[M]^+$, ^{35}Cl): 362.09155; found: 362.090638.

3-Ethyl 5-methyl 6-hydroxy-4'-methoxy-2,4-dimethyl-biphenyl-3,5-dicarboxylate (4i). Starting with **3** (0.488 g, 2.0 mmol) and **1i** (0.806 g, 2.2 mmol), **4i** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellowish oil (0.245 g, 34%). 1H NMR

(250 MHz, CDCl₃): δ = 1.32 (t, 3J = 7.1 Hz, 3 H, OCH₂CH₃), 1.96 (s, 3 H, PhCH₃), 2.42 (s, 3 H, PhCH₃), 3.77 (s, 3 H, OCH₃), 3.89 (s, 3 H, OCH₃), 4.32 (q, 3J = 6.9 Hz, 2 H, OCH₂CH₃), 6.88 - 6.93 (m, 2 H, 2 CH_{Ar}), 7.02 - 7.08 (m, 2 H, 2 CH_{Ar}), 11.26 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.2, 18.2, 20.1 (CH₃), 52.3, 55.3 (OCH₃), 61.2 (OCH₂), 110.6 (CCOOCH₃), 113.9 (2 CH_{Ar}), 128.1 (C_{Ar}), 128.7 (CCOOC₂H₅), 128.9 (C_{Ar}), 130.9 (2 CH_{Ar}), 135.8, 139.7 (C_{Ar}), 158.8 (C_{Ar}), 160.2 (COH), 170.2, 171.9 (CO). IR (neat, cm⁻¹): ν = 3033 (w), 2995 (w), 2953 (w), 2906 (w), 2835 (w), 1719 (m), 1656 (w), 1608 (w), 1509 (s), 1439 (m), 1364 (w), 1331 (m), 1300 (w), 1242 (s), 1207 (s), 1174 (s), 1100 (m), 1073 (w), 1030 (s), 985 (w), 956 (w), 830 (m), 810 (m), 764 (w), 686 (w), 637 (w), 581 (w), 555 (w), 526 (m). GC-MS (EI, 70 eV): m/z (%) = 358 ([M]⁺, 5), 326 (10), 270 (21), 121 (100), 78 (6). HRMS (EI): Calcd. for C₂₀H₂₂O₆ ([M]⁺): 358.14109; found: 358.140227.

1-Ethyl 3-methyl 4-hydroxy-5-methoxy-2,6-dimethylisophthalate (4j): Starting with **3** (0.305 g, 1.25 mmol) and **1j** (0.363 g, 1.25 mmol), **4j** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellow oil (0.230 g, 65%). ¹H NMR (CDCl₃, 250 MHz): δ = 1.39 (t, 3J = 7.3 Hz, 3H, OCH₂CH₃), 2.23 (s, 3H, PhCH₃), 2.41 (s, 3H, PhCH₃), 3.97 (s, 3H, OCH₃), 3.98 (s, 3H, OCH₃), 4.38 (q, 3J = 7.3 Hz, 2H, OCH₂CH₃), 11.30 (s, 1H, OH), ¹³C NMR (CDCl₃, 75 MHz): δ = 13.3, 14.2, 19.8 (CH₃), 52.4, 60.1 (OCH₃), 61.3 (OCH₂CH₃), 111.8, 128.4, 131.7, 134.0, 144.7 (C_{Ar}), 156.1 (COH), 169.5, 171.7 (CO). IR (neat, cm⁻¹): $\tilde{\nu}$ = 3422 (br, w), 2982 (m), 2957 (m), 2939 (m), 2838 (s), 1726 (s), 1683 (s), 1600 (m), 1578 (m). MS (EI, 70 eV): m/z (%) = 282 (M⁺, 35), 250 (93), 237 (23), 222 (100), 205 (26), 194 (43). Anal.: calcd. for C₁₄H₁₈O₆ (282.29): C, 59.57; H, 6.43. Found: C, 59.65; H, 6.44.

Diethyl 4-hydroxy-2,6-dimethyl-5-phenoxyisophthalate (4k). Starting with **3** (0.366 g, 1.5 mmol) and **1k** (0.577 g, 1.6 mmol), **4k** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as reddish viscous oil (0.260 g, 48%). ¹H NMR (250 MHz, CDCl₃): δ = 1.31 (t, 3J = 7.4 Hz, 3 H, OCH₂CH₃), 1.34 (t, 3J = 7.6 Hz, 3 H, OCH₂CH₃), 2.07 (s, 3 H, PhCH₃), 2.42 (s, 3 H, PhCH₃), 4.33 (q, 3J = 7.3 Hz, 2 H, OCH₂CH₃), 4.37 (q, 3J = 7.4 Hz, 2 H, OCH₂CH₃), 6.76 (d, 3J = 7.4 Hz, 2 H, CH_{Ar}), 6.89 (m, 1 H, CH_{Ar}), 6.94 (m, 2 H, CH_{Ar}), 11.27 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 12.7, 13.1, 19.0, 19.2 (CH₃),

60.1, 61.1 (OCH₂CH₃), 110.6 (CCOOC₂H₅), 113.6 (2 CH_{Ar}), 115.2 (CH_{Ar}), 128.7 (CCOOC₂H₅), 130.7 (2 CH_{Ar}), 130.9, 132.6, 137.9, 154.9 (C_{Ar}), 156.5 (COH), 168.2, 169.9 (CO). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2979 (w), 1722 (s), 1658 (m), 1489 (m), 1367 (m), 1261 (m), 1215 (s), 1046 (s), 749 (s), 688 (m). GC-MS (EI, 70 eV): *m/z* (%) = 358 ([M⁺], 63), 312 (64), 283 (100), 267 (21), 240 (12), 211 (6), 181 (5), 161 (11), 105 (61), 77 (22). HRMS (EI): Calcd. for C₂₀H₂₂O₆: 358.14109; found: 358.14174.

Diethyl 4-hydroxy-2,6-dimethyl-5-(2-tolyloxy)isophthalate (4l). Starting with **3** (0.488 g, 2.0 mmol) and **1l** (0.837 g, 2.2 mmol), **4l** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a reddish viscous oil (0.312 g, 42%). ¹H NMR (250 MHz, CDCl₃): δ = 1.32 (t, ³*J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.35 (t, ³*J* = 7.4 Hz, 3 H, OCH₂CH₃), 2.05 (s, 3 H, PhCH₃), 2.24 (s, 3 H, PhCH₃), 2.43 (s, 3 H, PhCH₃), 4.35 (q, ³*J* = 7.4 Hz, 2 H, OCH₂CH₃), 4.38 (q, ³*J* = 7.6 Hz, 2 H, OCH₂CH₃), 6.54 (s, 1 H, CH_{Ar}), 6.56 (m, 2 H, 2 CH_{Ar}), 6.75 (d, ³*J* = 7.5 Hz, 1 H, CH_{Ar}), 11.20 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 15.6, 16.0, 16.1, 21.9, 23.3 (CH₃), 63.2, 64.0 (CH₂), 113.1 (CH_{Ar}), 114.2 (C_{Ar}), 117.1, 124.7, 130.5 (CH_{Ar}), 131.3, 135.4, 136.8, 140.9, 141.6 (C_{Ar}), 157.8 (COH), 159.4 (C_{Ar}), 171.1, 172.8 (CO). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2979 (m), 1723 (s), 1658 (s), 1586 (m), 1444 (s), 1367 (m), 1321 (s), 1218 (s), 1139 (s), 1032 (s), 939 (m), 771 (m), 686 (m). MS (EI, 70 eV): *m/z* (%) = 372 ([M⁺], 33), 326 (49), 297 (84), 281 (12), 254 (13), 225 (4), 119 (100). HRMS (EI): Calcd. for C₂₁H₂₄O₆: 372.15673; found: 372.15665.

Diethyl 4-hydroxy-2,6-dimethyl-5-(3-tolyloxy)isophthalate (4m). Starting with **3** (0.488 g, 2.0 mmol) and **1m** (0.836 g, 2.2 mmol), **4m** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a reddish viscous oil (0.382 g, 51%). ¹H NMR (250 MHz, CDCl₃): δ = 1.31 (t, ³*J* = 7.4 Hz, 3 H, OCH₂CH₃), 1.35 (t, ³*J* = 7.6 Hz, 3 H, OCH₂CH₃), 2.07 (s, 3 H, PhCH₃), 2.22 (s, 3 H, PhCH₃), 2.42 (s, 3 H, PhCH₃), 4.36 (q, ³*J* = 7.2 Hz, 2 H, OCH₂CH₃), 4.38 (q, ³*J* = 7.6 Hz, 2 H, OCH₂CH₃), 6.56 (m, 2 H, 2 CH_{Ar}), 6.72 (d, ³*J* = 7.6 Hz, 1 H, CH_{Ar}), 7.06 (m, 1 H, CH_{Ar}), 11.25 (s, 1 H, OH). ¹³C NMR (62 MHz, CDCl₃): δ = 13.7, 14.1, 14.2, 20.0, 21.4 (CH₃), 61.4, 61.8 (CH₂), 111.5 (CH_{Ar}), 112.3 (C_{Ar}), 115.3, 122.8 (CH_{Ar}), 128.6 (C_{Ar}), 129.2 (CH_{Ar}), 133.5, 135.0, 139.0, 139.7 (C_{Ar}), 155.9 (COH), 157.5 (C_{Ar}), 169.3, 170.9 (CO). IR (neat, cm⁻¹): $\tilde{\nu}$ = 1660 (s), 1619 (m), 1452 (s), 1343 (s), 1219

(m), 1136 (s), 966 (m), 882 (m), 694 (m). MS (EI, 70 eV): m/z (%) = 372 ($[M^+]$, 28), 326 (52), 297 (66), 254 (28), 119 (100). HRMS (EI): Calcd. for $C_{21}H_{24}O_6$: 372.15673; found: 372.15665.

3-Acetyl-4-hydroxy-2,6-dimethyl-benzoic acid ethylester (4n): Starting with **3** (0.305 g, 1.25 mmol) and **1n** (0.305 g, 1.25 mmol), **4n** was isolated after chromatography (silica gel, *n*-heptane/EtOAc) as a yellow oil (0.113 g, 38%). 1H NMR ($CDCl_3$, 250 MHz): δ = 1.39 (t, 3J = 7.0 Hz, 3H, OCH_2CH_3), 2.27 (s, 3H, $PhCH_3$), 2.49 (s, 3H, $PhCH_3$), 2.63 (s, 3H, $PhCH_3$), 4.39 (q, 3J = 7.0 Hz, 2H, OCH_2CH_3), 6.69 (s, 1H, CH_{Ar}), 11.83 (s, 1H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.2, 20.1, 20.6, 33.1 (CH_3), 61.3 (OCH_2CH_3), 117.5 (CH_{Ar}), 120.6, 128.4, 136.1, 142.3 (C_{Ar}), 161.8 (COH), 169.5, 205.7 (CO). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3337 (s, br), 2982 (s), 2935 (m), 2909 (m), 2874 (w), 1722 (s), 1695 (s), 1631 (s), 1599 (s). MS (EI, 70 eV): m/z (%) = 236 (M^+ , 60), 221 (100), 207 (37), 193 (83), 191 (79), 175 (42), 91 (35). Anal.: calcd. for $C_{13}H_{16}O_4$ (236.26): C, 66.09; H, 6.83. Found: C, 66.05; H, 6.90.

3-Benzoyl-4-hydroxy-2,6-dimethyl-benzoic acid ethylester (4o). Starting with **3** (0.305 g, 1.25 mmol) and **1o** (0.386 g, 1.25 mmol), **4o** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellow oil (0.135 g, 36%); 1H NMR ($CDCl_3$, 250 MHz): δ = 1.36 (t, 3J = 7.0 Hz, 3H, OCH_2CH_3), 1.96 (s, 3H, $PhCH_3$), 2.32 (s, 3H, $PhCH_3$), 4.35 (q, 3J = 7.0 Hz, 2H, OCH_2CH_3), 7.73 – 7.42 (m, 5H, 5 CH_{Ar}), 6.73 (s, 1H, CH_{Ar}), 8.66 (s, 1H, OH). ^{13}C NMR ($CDCl_3$, 63 MHz): δ = 14.2, 19.8, 20.1 (CH_3), 61.2 (OCH_2CH_3), 116.3 (CH_{Ar}), 121.6, 128.1 (C_{Ar}), 128.8 (2 CH_{Ar}), 129.1 (2 CH_{Ar}), 133.2 (CH_{Ar}), 135.7, 139.4, 140.9 (C_{Ar}), 158.1 (COH), 169.4, 200.1 (CO). IR (KBr, cm^{-1}): ν = 3305 (w, br), 2979 (w), 1712 (s), 1667 (s), 1595 (s), 1448 (m), 1363 (m), 1309 (m), 1283 (m), 1228 (s), 1174 (s), 1132 (s), 1052 (m), 937 (w), 922 (s), 851 (m), 806 (w), 758 (w), 711 (s), 686 (s), 622 (s), 587 (m). MS (EI, 70 eV): m/z (%) = 299 ($[M+1]^+$, 9), 298 ($[M^+]$, 51), 251 (100), 224 (18), 193 (10), 175 (17), 105 (32), 91 (9), 77 (26). HRMS (EI, 70 eV): calcd. for $C_{18}H_{18}O_4$ (M^+): 298.1200, found: 298.1193.

Typical experimental procedure for the synthesis of 7a-ae. To a CH₂Cl₂ solution (2 mL / 1 mmol of **6a-e**) of **6a-e** was added **1** (1.1 mmol) and, subsequently, TiCl₄ (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 CH₂Cl₂ (3 × 20 mL). The combined organic layers were dried (Na₂SO₄), filtered and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, heptanes / EtOAc) to give **7a-ae**.

10

Dimethyl 4-hydroxy-2-methylisophthalate (7a). Starting with **6a** (0.237 g, 1.5 mmol) and **1a** (0.429 g, 1.7 mmol), **7a** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish solid (0.144 g, 43%), mp. = 88 - 90 °C. ¹H NMR (250 MHz, CDCl₃): δ = 2.63 (s, 3 H, PhCH₃), 3.79 (s, 3 H, OCH₃), 3.91 (s, 3 H, OCH₃), 6.78 (d, ³J = 8.7 Hz, 1 H, CH_{Ar}), 7.76 (d, ³J = 8.9 Hz, 1 H, CH_{Ar}), 10.98 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 20.0 (CH₃), 52.0, 52.5 (OCH₃), 114.5 (CCOOCH₃), 115.2 (CH_{Ar}), 123.9 (CCOOCH₃), 135.8 (CH_{Ar}), 143.6 (C_{Ar}), 163.9 (COH), 168.0, 171.7 (CO). IR (KBr, cm⁻¹): ν̄ = 3339 (w), 2989 (w), 2959 (w), 2924 (w), 2853 (w), 1715 (m), 1688 (m), 1651 (m), 1583 (m), 1537 (m), 1430 (m), 1386 (m), 1321 (m), 1243 (m), 1195 (s), 1151 (s), 1050 (m), 1018 (m), 960 (m), 944 (m), 858 (m), 797 (s), 754 (m), 707 (s), 652 (m), 560 (m). GC-MS (EI, 70 eV): m/z (%) = 224 ([M]⁺, 31), 193 (30), 192 (100), 161 (56), 160 (26), 149 (13), 133 (12), 132 (12), 105 (10), 77 (15), 51 (11). HRMS (EI): Calcd. for C₁₁H₁₂O₅ ([M]⁺): 224.06792; found: 224.067341.

25 **Dimethyl 4-hydroxy-2,5-dimethylisophthalate (7b).** Starting with **6a** (0.237 g, 1.5 mmol) and **1p** (0.457 g, 1.7 mmol), **7b** was isolated after chromatography (silica gel, heptanes/EtOAc) as a white solid (0.205 g, 57%), mp. = 110 - 112 °C. ¹H NMR (250 MHz, CDCl₃): δ = 2.15 (s, 3 H, PhCH₃), 2.60 (s, 3 H, PhCH₃), 3.78 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.75 (s, 1 H, CH_{Ar}), 11.22 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 15.7, 19.8 (CH₃), 51.9, 52.4 (OCH₃), 113.7 (CCOOCH₃), 123.0 (C_{Ar}), 124.0 (CCOOCH₃), 136.3 (CH_{Ar}), 140.6 (C_{Ar}), 162.4 (COH), 168.2, 172.1 (CO). IR (KBr, cm⁻¹): ν̄ = 3349 (w), 2992 (w),

30

2953 (w), 2931 (w), 2853 (w), 1716 (m), 1693 (m), 1667 (m), 1579 (m), 1537 (m), 1434 (m), 1384 (w), 1330 (m), 1229 (m), 1190 (s), 1144 (s), 1049 (m), 1013 (m), 959 (m), 855 (m), 796 (s), 745 (m), 702 (m), 651 (m), 606 (m). GC-MS (EI, 70 eV): m/z (%) = 238 ($[M]^+$, 33), 207 (33), 206 (100), 178 (65), 175 (31), 163 (26), 91 (16), 65 (12). HRMS (EI): Calcd. for $C_{12}H_{14}O_5$ ($[M]^+$): 238.08358; found: 238.083755.

3-Ethyl 1-methyl 5-ethyl-4-hydroxy-2-methylisophthalate (7c). Starting with **6a** (0.237 g, 1.5 mmol) and **1c** (0.499 g, 1.7 mmol), **7c** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.200 g, 50%). 1H NMR (250 MHz, $CDCl_3$): δ = 1.05 (t, 3J = 7.5 Hz, 3 H, CH_2CH_3), 1.27 (t, 3J = 6.7 Hz, 3 H, OCH_2CH_3), 2.48 (q, 3J = 7.3 Hz, 2 H, CH_2CH_3), 2.52 (s, 3 H, $PhCH_3$), 3.71 (s, 3 H, OCH_3), 4.30 (q, 3J = 7.0 Hz, 2 H, OCH_2CH_3), 7.55 (s, 1 H, CH_{Ar}), 11.21 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 13.5, 14.1, 19.9 (CH_3), 23.1 (CH_2), 52.0 (OCH_3), 62.2 (OCH_2), 114.3 ($CCOOC_2H_5$), 123.4 ($CCOOC_2H_5$), 129.8 (C_{Ar}), 134.9 (CH_{Ar}), 140.8 (C_{Ar}), 162.5 (COH), 168.5, 171.9 (CO). IR (neat, cm^{-1}): ν = 2967 (w), 2874 (w), 1718 (m), 1655 (m), 1580 (w), 1429 (m), 1396 (w), 1372 (m), 1326 (m), 1261 (m), 1227 (s), 1199 (s), 1154 (s), 1095 (w), 1043 (m), 1019 (m), 959 (w), 844 (w), 809 (m), 780 (m), 743 (w), 650 (m), 535 (w). GC-MS (EI, 70 eV): m/z (%) = 266 ($[M]^+$, 25), 221 (17), 220 (63), 193 (12), 192 (100), 189 (15), 177 (10), 77 (11), 29 (7). HRMS (EI): Calcd. for $C_{14}H_{18}O_5$ ($[M]^+$): 266.11488; found: 266.115159.

20

Dimethyl 5-butyl-4-hydroxy-2-methylisophthalate (7d). Starting with **6a** (0.237 g, 1.5 mmol) and **1q** (0.522 g, 1.7 mmol), **7d** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.218 g, 52%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.85 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_3CH_3$), 1.25 – 1.35 (m, 2 H, CH_2), 1.48 – 1.53 (m, 2 H, CH_2), 2.54 (t, 3J = 7.5 Hz, 2 H, $PhCH_2$), 2.58 (s, 3 H, $PhCH_3$), 3.79 (s, 3 H, OCH_3), 3.90 (s, 3 H, OCH_3), 7.62 (s, 1 H, CH_{Ar}), 11.21 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 12.9, 18.9 (CH_3), 21.5, 28.9, 30.4 (CH_2), 50.9, 51.5 (OCH_3), 113.0 ($CCOOC_2H_5$), 122.1 (C_{Ar}), 127.6 ($CCOOC_2H_5$), 134.8 (CH_{Ar}), 139.6 (C_{Ar}), 161.1 (COH), 167.4, 171.2 (CO). IR (neat, cm^{-1}): ν = 3024 (w), 2989 (w), 2957 (w), 2932 (w), 2865 (w), 1713 (s), 1659 (m), 1608 (w), 1579 (w), 1431 (s), 1377 (w), 1337 (m), 1259 (m), 1228 (s), 1199 (s), 1152 (s), 1049 (m), 994 (m), 962 (m), 894 (m), 872 (w), 808 (m), 767 (m), 653 (m), 544 (w). GC-MS (EI, 70 eV): m/z (%)

30

= 280 ($[M]^+$, 34), 249 (20), 248 (19), 231 (15), 220 (39), 219 (18), 217 (14), 207 (12), 206 (100), 205 (23), 189 (19), 178 (40), 173 (34), 91 (10), 77 (11). HRMS (EI): Calcd. for $C_{15}H_{20}O_5$ ($[M]^+$): 280.13053; found: 280.130756.

5 **Dimethyl 5-hexyl-4-hydroxy-2-methylisophthalate (7e)**. Starting with **6a** (0.237 g, 1.5 mmol) and **1d** (0.568 g, 1.7 mmol), **7e** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.254 g, 55%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.72 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_5CH_3$), 1.11 – 1.20 (m, 6 H, 3 CH_2), 1.40 – 1.47 (m, 2 H, CH_2), 2.44 (t, 3J = 7.5 Hz, 2 H, $PhCH_2$), 2.51 (s, 3 H, $PhCH_3$), 3.71 (s, 3 H, OCH_3), 3.82 (s,
10 3 H, OCH_3), 7.54 (s, 1 H, CH_{Ar}), 11.13 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.0, 19.8 (CH_3), 22.6, 29.1, 29.2, 29.7, 31.7 (CH_2), 51.9, 52.4 (OCH_3), 113.9 ($CCOOCH_3$), 123.1 (C_{Ar}), 128.7 ($CCOOCH_3$), 135.7 (CH_{Ar}), 140.5 (C_{Ar}), 162.0 (COH), 168.3, 172.2 (CO). IR (neat, cm^{-1}): ν = 2952 (w), 2926 (w), 2856 (w), 1719 (m), 1659 (m), 1608 (w), 1580 (w), 1432 (m), 1335 (m), 1227 (s), 1194 (s), 1150 (s), 1045 (m), 990 (m), 961 (w), 886 (w), 808
15 (m), 779 (m), 725 (w), 651 (m), 555 (w). GC-MS (EI, 70 eV): m/z (%) = 308 ($[M]^+$, 37), 277 (23), 276 (20), 259 (16), 248 (40), 247 (31), 245 (11), 233 (13), 220 (10), 219 (17), 217 (14), 207 (16), 206 (100), 205 (33), 178 (35), 173 (42), 91 (10), 77 (10), 43 (8). HRMS (EI): Calcd. for $C_{17}H_{24}O_5$ ($[M]^+$): 308.16183; found: 308.161283.

20 **Dimethyl 5-hexyl-4-hydroxy-2-methylisophthalate (7f)**. Starting with **6a** (0.237 g, 1.5 mmol) and **1r** (0.591 g, 1.7 mmol), **7f** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.246 g, 51%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.78 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_6CH_3$), 1.18 – 1.22 (m, 8 H, 4 CH_2), 1.48 – 1.54 (m, 2 H, CH_2), 2.53 (t, 3J = 7.5 Hz, 2 H, $PhCH_2$), 2.58 (s, 3 H, $PhCH_3$), 3.79 (s, 3 H, OCH_3), 3.89 (s,
25 3 H, OCH_3), 7.62 (s, 1 H, CH_{Ar}), 11.22 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 13.0, 18.8 (CH_3), 21.6, 27.7, 27.8, 28.2, 28.7, 30.8 (CH_2), 50.9, 51.4 (OCH_3), 112.9 ($CCOOCH_3$), 122.1 (C_{Ar}), 127.6 ($CCOOCH_3$), 134.7 (CH_{Ar}), 139.6 (C_{Ar}), 161.1 (COH), 167.4, 171.4 (CO). IR (neat, cm^{-1}): ν = 3400 (w), 2952 (w), 2923 (w), 2853 (w), 1720 (m), 1659 (m), 1610 (w), 1579 (w), 1433 (m), 1379 (w), 1336 (m), 1228 (s), 1195 (s), 1152 (s), 1046 (m), 997 (w), 889
30 (w), 809 (m), 779 (m), 723 (w), 651 (m), 553 (w). GC-MS (EI, 70 eV): m/z (%) = 322 ($[M]^+$, 33), 291 (23), 290 (19), 273 (14), 262 (32), 247 (29), 233 (11), 231 (14), 219 (17), 207 (17),

206 (100), 205 (33), 178 (33), 173 (40), 91 (10), 77 (9), 43 (10), 29 (8). HRMS (EI): Calcd. for C₁₈H₂₆O₅ ([M]⁺): 322.17748; found: 322.177264.

Dimethyl 4-hydroxy-2-methyl-5-octylisophthalate (7g). Starting with **6a** (0.237 g, 1.5 mmol) and **1s** (0.614 g, 1.7 mmol), **7g** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.241 g, 48%). ¹H NMR (250 MHz, CDCl₃): δ = 0.80 (t, ³J = 7.5 Hz, 3 H, (CH₂)₇CH₃), 1.15 – 1.23 (m, 10 H, 5 CH₂), 1.51 – 1.53 (m, 2 H, CH₂), 2.54 (t, ³J = 7.7 Hz, 2 H, PhCH₂), 2.59 (s, 3 H, PhCH₃), 3.80 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.21 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.0, 19.8 (CH₃), 22.6, 29.2, 29.4, 29.5, 29.6, 29.7, 31.8 (CH₂), 51.9, 52.4 (OCH₃), 113.9 (CCOOCH₃), 123.1 (C_{Ar}), 128.8 (CCOOCH₃), 135.7 (CH_{Ar}), 140.6 (C_{Ar}), 162.0 (COH), 168.4, 172.1 (CO). IR (neat, cm⁻¹): ν_{max} = 2952 (w), 2924 (w), 2853 (w), 1720 (m), 1699 (m), 1605 (w), 1552 (w), 1434 (m), 1377 (w), 1336 (m), 1229 (s), 1202 (s), 1153 (s), 1045 (m), 996 (w), 911 (w), 809 (m), 759 (m), 700 (m), 652 (w), 566 (w). GC-MS (EI, 70 eV): *m/z* (%) = 336 ([M]⁺, 35), 305 (20), 304 (14), 287 (13), 276 (32), 247 (34), 245 (17), 233 (12), 219 (18), 207 (15), 206 (100), 205 (30), 178 (33), 173 (39), 91 (11), 43 (10), 41 (10), 29 (7). HRMS (EI): Calcd. for C₁₉H₂₈O₅ ([M]⁺): 336.19313; found: 336.193115.

Dimethyl 4-hydroxy-2-methyl-5-nonylbenzene-1,3-dioate (7h). Starting with **6a** (0.237 g, 1.5 mmol) and **1e** (0.522 g, 1.7 mmol), **7h** was isolated after chromatography (silica gel, heptanes/EtOAc) as a white solid (0.273 g, 52%), mp. 56 - 57 °C. ¹H NMR (250 MHz, CDCl₃): δ = 0.80 (t, ³J = 7.3 Hz, 3 H, (CH₂)₈CH₃), 1.19 – 1.26 (m, 12 H, 6 CH₂), 1.47 – 1.55 (m, 2 H, CH₂), 2.54 (t, ³J = 7.2 Hz, 2 H, PhCH₂), 2.59 (s, 3 H, PhCH₃), 3.80 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.20 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.1, 18.8 (CH₃), 21.6, 28.2, 28.3, 28.4, 28.5, 28.7, 29.9, 30.9 (CH₂), 50.9, 51.4 (OCH₃), 112.9 (CCOOCH₃), 122.1 (CCOOCH₃), 127.6 (C_{Ar}), 134.7 (CH_{Ar}), 139.6 (C_{Ar}), 161.0 (COH), 167.3, 171.2 (CO). IR (KBr, cm⁻¹): ν_{max} = 3000 (w), 2954 (m), 2914 (m), 2851 (m), 1709 (m), 1665 (m), 1607 (w), 1579 (m), 1471 (m), 1437 (w), 1426 (w), 1382 (w), 1335 (m), 1252 (w), 1229 (s), 1210 (s), 1194 (s), 1149 (s), 1045 (m), 1003 (m), 986 (m), 972 (m), 961 (m), 920 (m), 887 (s), 802 (m), 791 (m), 781 (m), 743 (m), 714 (m), 659 (m), 610 (m), 561 (w). GC-MS (EI, 70 eV): *m/z* (%) = 350 ([M]⁺, 34), 319 (20), 290 (24), 259 (15), 247

(27), 233 (10), 219 (15), 206 (100), 192 (9), 173 (39), 163 (7), 147 (6), 119 (3), 91 (7), 77 (7), 55 (4), 41 (10). HRMS (EI): Calcd. for $C_{20}H_{30}O_5$ ($[M]^+$): 350.20878; found: 350.208651

3-(2-Methoxyethyl) 1-methyl 4-hydroxy-2-methylbenzene-1,3-dioate (7i). Starting with **6a** (0.237 g, 1.5 mmol) and **1t** (0.479 g, 1.7 mmol), **7i** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.193 g, 48%). 1H NMR (250 MHz, $CDCl_3$): δ = 2.66 (s, 3 H, $PhCH_3$), 3.36 (s, 3 H, OCH_3), 3.66 (t, 3J = 4.9 Hz, 2 H, $OCH_2CH_2OCH_3$), 3.80 (s, 3 H, OCH_3), 4.47 (t, 3J = 4.7 Hz, 2 H, $OCH_2CH_2OCH_3$), 6.78 (d, 3J = 8.7 Hz, 1 H, CH_{Ar}), 7.78 (d, 3J = 9.0 Hz, 1 H, CH_{Ar}), 10.51 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 20.6 (CH_3), 52.7, 59.6 (OCH_3), 65.2, 70.5 (OCH_2), 115.9 (CH_{Ar}), 116.0 ($CCOOCH_2CH_2OCH_3$), 124.7 ($CCOOCH_3$), 136.5 (CH_{Ar}), 144.3 (C_{Ar}), 164.0 (COH), 168.6, 171.1 (CO). IR (KBr, cm^{-1}): ν_{max} = 3281 (w), 2951 (w), 2924 (w), 2851 (w), 1716 (m), 1661 (w), 1588 (m), 1470 (w), 1434 (w), 1378 (w), 1315 (w), 1224 (m), 1200 (m), 1048 (s), 1028 (s), 955 (m), 868 (m), 834 (m), 804 (m), 783 (m), 709 (m), 653 (m), 608 (m), 543 (m). GC-MS (EI, 70 eV): m/z (%) = 268 ($[M]^+$, 17), 237 (8), 192 (100), 161 (32), 133 (5), 105 (6), 77 (7), 59 (11), 45 (5). HRMS (EI): Calcd. for $C_{13}H_{16}O_6$ ($[M]^+$): 268.09414; found: 268.094150.

Dimethyl 2-hydroxy-4,4'-dimethylbiphenyl-3,5-dicarboxylate (7j). Starting with **6a** (0.237 g, 1.5 mmol) and **1g** (0.403 g, 1.65 mmol), **7j** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish solid (0.250 g, 53 %), mp. = 83 - 85 °C. 1H NMR (250 MHz, $CDCl_3$): δ = 2.32 (s, 3 H, $PhCH_3$), 2.65 (s, 3 H, $PhCH_3$), 3.80 (s, 3 H, OCH_3), 3.93 (s, 3 H, OCH_3), 7.15 – 7.19 (m, 2 H, 2 CH_{Ar}), 7.34 – 7.37 (m, 2 H, 2 CH_{Ar}), 7.83 (s, 1 H, CH_{Ar}), 11.04 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 18.9, 20.2 (CH_3), 51.0, 51.6 (OCH_3), 114.3 ($CCOOCH_3$), 122.6 ($CCOOCH_3$), 126.9 (C_{Ar}), 128.0 (2 CH_{Ar}), 128.2 (2 CH_{Ar}), 132.6 (C_{Ar}), 135.4 (CH_{Ar}), 136.4, 141.0 (C_{Ar}), 159.5 (COH), 167.0, 171.0 (CO). IR (KBr, cm^{-1}): ν_{max} = 3027 (w), 3012 (w), 2953 (w), 2924 (w), 2853 (w), 1771 (w), 1718 (m), 1684 (w), 1663 (m), 1653 (m), 1636 (w), 1616 (w), 1608 (w), 1576 (w), 1558 (w), 1540 (w), 1533 (w), 1516 (w), 1507 (w), 1497 (w), 1489 (w), 1472 (w), 1456 (w), 1436 (m), 1399 (w), 1338 (m), 1240 (m), 1209 (m), 1103 (w), 1052 (w), 1028 (w), 958 (w), 910 (w), 822 (w), 783 (w), 733 (m), 668 (w), 650 (w), 617 (w), 608 (w), 567 (w), 541 (w). GC-MS (EI,

70 eV): m/z (%) = 314 ($[M]^+$, 42), 282 (100), 267 (5), 251 (14), 239 (16), 222 (13), 195 (14), 165 (13), 152 (12), 132 (12), 119 (29), 105 (18), 91 (19), 69 (22), 57 (29), 43 (20). HRMS (EI): Calcd. for $C_{18}H_{18}O_5$ ($[M]^+$): 314.11488; found: 314.114827

Dimethyl 4'-chloro-2-hydroxy-4-methylbiphenyl-3,5-dicarboxylate (7k). Starting with **6a** (0.237 g, 1.5 mmol) and **1h** (0.612 g, 1.7 mmol), **7k** was isolated after chromatography (silica gel, heptanes/EtOAc) as brownish crystals (0.241 g, 48%), mp. = 160 - 161 °C. 1H NMR (250 MHz, $CDCl_3$): δ = 2.65 (s, 3 H, $PhCH_3$), 3.80 (s, 3 H, OCH_3), 3.93 (s, 3 H, OCH_3), 7.28 – 7.33 (m, 2 H, 2 CH_{Ar}), 7.38 – 7.43 (m, 2 H, 2 CH_{Ar}), 7.81 (s, 1 H, CH_{Ar}), 11.36 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 19.1 (CH_3), 51.1, 51.7 (OCH_3), 114.0 ($CCOOCH_3$), 122.9 ($CCOOCH_3$), 125.6 (C_{Ar}), 127.4 (2 CH_{Ar}), 129.6 (2 CH_{Ar}), 132.6, 134.0 (C_{Ar}), 135.4 (CH_{Ar}), 141.8 (C_{Ar}), 159.7 (COH), 166.8, 171.0 (CO). IR (KBr, cm^{-1}): ν = 3000 (w), 2951 (w), 1716 (m), 1662 (m), 1603 (m), 1579 (m), 1562 (w), 1492 (m), 1439 (m), 1425 (m), 1389 (m), 1375 (m), 1323 (m), 1301 (m), 1239 (m), 1194 (s), 1171 (s), 1105 (m), 1085 (m), 1051 (m), 1025 (m), 1010 (m), 949 (m), 926 (m), 876 (m), 825 (m), 806 (m), 782 (m), 771 (m), 746 (m), 719 (m), 675 (m), 650 (m), 629 (m), 613 (m), 540 (m). GC-MS (EI, 70 eV): m/z (%) = 336 ($[M]^+$, ^{37}Cl , 6), 334 ($[M]^+$, ^{35}Cl , 17), 302 (100), 296 (20), 270 (12), 242 (7), 215 (4), 152 (11), 125 (8), 104 (6), 86 (10), 43 (9). HRMS (EI): Calcd. for $C_{17}H_{15}O_5Cl$ ($[M]^+$): 334.06025; found: 334.059873.

20

Dimethyl 4-hydroxy-5-methoxy-2-methylbenzene-1,3-dioate (7l). Starting with **6a** (0.237 g, 1.5 mmol) and **1j** (0.360 g, 1.7 mmol), **7l** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.198 g, 52%). 1H NMR (250 MHz, $CDCl_3$): δ = 2.45 (s, 3 H, $PhCH_3$), 3.76 (s, 3 H, OCH_3), 3.79 (s, 3 H, OCH_3), 3.86 (s, 3 H, OCH_3), 7.32 (s, 1 H, CH_{Ar}), 9.41 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 18.7 (CH_3), 52.0, 52.6, 56.2 (OCH_3), 115.5 (CH_{Ar}), 117.6 ($CCOOCH_3$), 122.3 ($CCOOCH_3$), 133.4, 145.3 (C_{Ar}), 151.6 (COH), 167.7, 170.4 (CO). IR (KBr, cm^{-1}): ν = 3400 (w), 3001 (w), 2951 (w), 2842 (w), 1713 (m), 1662 (m), 1610 (w), 1587 (m), 1492 (m), 1433 (m), 1385 (w), 1357 (m), 1305 (m), 1282 (m), 1200 (s), 1167 (s), 1082 (s), 1048 (m), 1029 (s), 959 (m), 914 (w), 886 (m), 858 (w), 806 (w), 780 (m), 730 (m), 688 (w), 671 (w), 647 (w), 621 (w), 592 (w), 553 (w). GC-MS (EI, 70 eV): m/z (%) = 254 ($[M]^+$, 38), 222 (100), 207 (4), 194 (53), 179 (20), 163

(11), 147 (18), 136 (15), 119 (5), 107 (3), 92 (7), 77 (10), 64 (5), 53 (5), 39 (5). HRMS (EI): Calcd. for $C_{12}H_{14}O_6$ ($[M]^+$): 254.07849; found: 254.078254.

Dimethyl 2-ethyl-4-hydroxy-5-methylisophthalate (7m). Starting with **6b** (0.258 g, 1.5 mmol) and **1p** (0.457 g, 1.7 mmol), **7m** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.196 g, 52%). 1H NMR (250 MHz, $CDCl_3$): δ = 1.14 (t, 3J = 7.7 Hz, 3 H, CH_2CH_3), 2.21 (s, 3 H, $PhCH_3$), 3.09 (q, 3J = 7.1 Hz, 2 H, CH_2CH_3), 3.79 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 7.63 (s, 1 H, CH_{Ar}), 11.14 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.8, 15.0 (CH_3), 24.2 (CH_2), 50.9, 51.5 (OCH_3), 112.2 ($CCOOCH_3$), 121.5 (C_{Ar}), 123.4 ($CCOOCH_3$), 136.0 (CH_{Ar}), 145.6 (C_{Ar}), 161.4 (COH), 167.3, 171.2 (CO). IR (neat, cm^{-1}): ν = 2952 (w), 2930 (w), 2854 (w), 1719 (m), 1658 (m), 1610 (w), 1578 (w), 1432 (m), 1380 (w), 1333 (m), 1300 (m), 1277 (m), 1225 (s), 1192 (s), 1149 (s), 1071 (m), 1017 (m), 983 (m), 886 (w), 813 (m), 776 (m), 732 (m), 675 (m), 644 (m). GC-MS (EI, 70 eV): m/z (%) = 252 ($[M]^+$, 32), 221 (27), 220 (100), 189 (24), 177 (11), 170 (12), 161 (11), 160 (19), 133 (9), 132 (10), 103 (9), 77 (14). HRMS (EI): Calcd. for $C_{13}H_{16}O_5$ ($[M]^+$): 252.09923; found: 252.099203.

3-Ethyl 1-methyl 2,5-diethyl-4-hydroxyisophthalate (7n). Starting with **6b** (0.258 g, 1.5 mmol) and **1c** (0.499 g, 1.7 mmol), **7n** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.210 g, 50%). 1H NMR (250 MHz, $CDCl_3$): δ = 1.11-1.19 (m, 6 H, 2 CH_3), 1.37 (t, 3J = 7.4 Hz, 3 H, OCH_2CH_3), 2.58 (q, 3J = 7.4 Hz, 2 H, $PhCH_2$), 3.11 (q, 3J = 7.5 Hz, 2 H, $PhCH_2$), 3.79 (s, 3 H, OCH_3), 4.39 (q, 3J = 7.4 Hz, 2 H, OCH_2CH_3), 7.63 (s, 1 H, CH_{Ar}), 11.22 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 13.4, 13.9, 15.2 (CH_3), 21.9, 23.9, 50.9 (CH_2), 61.1 (OCH_2), 112.4 ($CCOOC_2H_5$), 121.6 ($CCOOCH_3$), 129.0 (C_{Ar}), 134.0 (CH_{Ar}), 145.3 (C_{Ar}), 161.0 (COH), 167.3, 170.6 (CO). IR (neat, cm^{-1}): ν = 2968 (w), 2935 (w), 2875 (w), 1722 (m), 1657 (m), 1608 (w), 1578 (w), 1429 (w), 1373 (w), 1323 (w), 1274 (m), 1229 (m), 1200 (m), 1156 (w), 1093 (w), 1061 (w), 1021 (w), 979 (w), 909 (w), 847 (w), 818 (w), 733 (m), 648 (w). GC-MS (EI, 70 eV): m/z (%) = 280 ($[M]^+$, 36), 249 (11), 235 (23), 234 (100), 206 (17), 203 (20), 191 (16), 175 (11), 174 (37), 147 (15), 146 (16), 91 (13), 77 (10). HRMS (EI): Calcd. for $C_{15}H_{20}O_5$ ($[M]^+$): 280.13053; found: 280.130632.

Dimethyl 5-butyl-2-ethyl-4-hydroxyisophthalate (7o). Starting with **6b** (0.258 g, 1.5 mmol) and **1q** (0.522 g, 1.7 mmol), **7o** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.229 g, 52%). ¹H NMR (250 MHz, CDCl₃): δ = 0.86 (t, ³J = 7.4 Hz, 3 H, (CH₂)₃CH₃), 1.14 (t, ³J = 7.4 Hz, 3 H, CH₂CH₃), 1.25 – 1.34 (m, 2 H, CH₂), 1.45 – 1.54 (m, 2 H, CH₂), 2.55 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 3.08 (q, ³J = 7.4 Hz, 2 H, PhCH₂), 3.78 (s, 3 H, OCH₃), 3.91 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.9, 16.0 (CH₃), 22.6, 25.1, 29.6, 31.4 (CH₂), 51.9, 52.5 (OCH₃), 113.4 (CCOOCH₃), 122.6 (C_{Ar}), 128.8 (CCOOCH₃), 135.9 (CH_{Ar}), 146.4 (C_{Ar}), 162.0 (COH), 168.2, 172.1 (CO). IR (neat, cm⁻¹): ν̄ = 2954 (w), 2930 (w), 2873 (w), 2256 (w), 1721 (m), 1662 (m), 1606 (w), 1579 (w), 1434 (m), 1337 (w), 1276 (m), 1228 (m), 1202 (m), 1153 (w), 1068 (w), 986 (w), 908 (m), 816 (w), 732 (s), 648 (w). GC-MS (EI, 70 eV): *m/z* (%) = 294 ([M]⁺, 47), 263 (27), 262 (41), 247 (12), 245 (23), 233 (18), 231 (20), 221 (14), 220 (100), 219 (26), 203 (55), 192 (13), 187 (28), 160 (15), 159 (11), 131 (11), 103 (12), 91 (13), 77 (14). HRMS (EI): Calcd. for C₁₆H₂₂O₅ ([M]⁺): 294.14618; found: 294.146337.

Dimethyl 2-ethyl-5-hexyl-4-hydroxyisophthalate (7p). Starting with **6b** (0.258 g, 1.5 mmol) and **1d** (0.568 g, 1.7 mmol), **7p** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.251 g, 52%). ¹H NMR (250 MHz, CDCl₃): δ = 0.79 (t, ³J = 7.6 Hz, 3 H, (CH₂)₅CH₃), 1.13 (t, ³J = 7.4 Hz, 3 H, CH₂CH₃), 1.21 – 1.29 (m, 6 H, 3 CH₂), 1.46 – 1.53 (m, 2 H, CH₂), 2.53 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 2.99 (q, ³J = 7.3 Hz, 2 H, PhCH₂), 3.78 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.61 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.0, 16.0, (CH₃), 22.6, 25.0, 29.1, 29.2, 29.8, 31.7 (CH₂), 51.9, 52.5 (OCH₃), 113.3 (CCOOCH₃), 122.6 (C_{Ar}), 128.8 (CCOOCH₃), 135.9 (CH_{Ar}), 146.4 (C_{Ar}), 161.9 (COH), 168.2, 172.1 (CO). IR (neat, cm⁻¹): ν̄ = 2953 (w), 2927 (w), 2856 (w), 2255 (w), 1719 (m), 1660 (m), 1606 (w), 1578 (w), 1433 (m), 1334 (m), 1272 (m), 1227 (s), 1200 (s), 1151 (m), 1087 (w), 1068 (w), 1047 (w), 992 (w), 906 (s), 816 (w), 729 (s), 648 (m). GC-MS (EI, 70 eV): *m/z* (%) = 323 (10), 322 ([M]⁺, 50), 291 (28), 290 (38), 273 (25), 262 (11), 261 (43), 259 (16), 247 (11), 234 (11), 233 (21), 231 (49), 221 (16), 220 (100), 219 (31), 205 (10), 192 (14), 187 (31), 160 (15), 131 (11), 103 (11), 91 (12), 77 (11). HRMS (EI): Calcd. for C₁₈H₂₆O₅ ([M]⁺): 322.17748; found: 322.177229.

Dimethyl 2-ethyl-5-heptyl-4-hydroxyisophthalate (7q). Starting with **6b** (0.258 g, 1.5 mmol) and **1r** (0.591 g, 1.7 mmol), **7q** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.257 g, 51%). ¹H NMR (250 MHz, CDCl₃): δ = 0.81 (t, ³J = 7.6 Hz, 3 H, (CH₂)₆CH₃), 1.14 (t, ³J = 7.6 Hz, 3 H, CH₂CH₃), 1.20 – 1.27 (m, 8 H, 4 CH₂), 1.49 – 1.54 (m, 2 H, CH₂), 2.53 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 3.08 (q, ³J = 7.4 Hz, 2 H, PhCH₂), 3.79 (s, 3 H, OCH₃), 3.91 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.1, 16.0, (CH₃), 22.6, 25.1, 29.1, 29.2, 29.4, 29.8, 31.8 (CH₂), 51.9, 52.5 (OCH₃), 113.2 (CCOOCH₃), 122.7 (C_{Ar}), 129.0 (CCOOCH₃), 136.2 (CH_{Ar}), 146.6 (C_{Ar}), 162.3 (COH), 168.4, 172.2 (CO). IR (neat, cm⁻¹): ν̄ = 2953 (w), 2927 (m), 2855 (w), 2258 (w), 1722 (m), 1662 (m), 1606 (w), 1579 (w), 1434 (m), 1335 (w), 1275 (m), 1228 (m), 1202 (m), 1152 (w), 1069 (w), 990 (w), 908 (m), 816 (w), 733 (m), 648 (w). GC-MS (EI, 70 eV): *m/z* (%) = 337 (10), 336 ([M]⁺, 45), 305 (26), 304 (35), 287 (24), 275 (10), 273 (15), 262 (10), 261 (41), 247 (11), 246 (49), 234 (10), 233 (20), 221 (18), 220 (100), 205 (10), 192 (15), 161 (10), 160 (13), 159 (11), 131 (11), 103 (10), 91 (11), 43 (10). HRMS (ED): Calcd. for C₁₉H₂₈O₅ ([M]⁺): 336.19313; found: 336.193054.

Dimethyl 2-ethyl-4-hydroxy-5-octylisophthalate (7r). Starting with **6b** (0.258 g, 1.5 mmol) and **1s** (0.614 g, 1.7 mmol), **7r** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellow oil (0.263 g, 50%). ¹H NMR (250 MHz, CDCl₃): δ = 0.79 (t, ³J = 7.5 Hz, 3 H, (CH₂)₇CH₃), 1.13 (t, ³J = 7.6 Hz, 3 H, CH₂CH₃), 1.22 – 1.36 (m, 10 H, 5 CH₂), 1.47 – 1.56 (m, 2 H, CH₂), 2.52 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 3.06 (q, ³J = 7.5 Hz, 2 H, PhCH₂), 3.79 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.7, 15.9, (CH₃), 22.1, 25.1, 28.9, 29.0, 29.2, 29.3, 29.8, 31.6 (CH₂), 51.6, 52.4 (OCH₃), 112.8 (CCOOCH₃), 121.7 (C_{Ar}), 128.7 (CCOOCH₃), 135.9 (CH_{Ar}), 146.1 (C_{Ar}), 161.9 (COH), 167.7, 171.9 (CO). IR (neat, cm⁻¹): ν̄ = 2954 (w), 2925 (m), 2854 (w), 2255 (w), 1745 (w), 1711 (w), 1658 (m), 1604 (w), 1569 (w), 1462 (w), 1329 (w), 1231 (w), 1153 (w), 908 (m), 845 (w), 734 (s), 649 (w). GC-MS (EI, 70 eV): *m/z* (%) = 350 (10), 318 (12), 228 (28), 220 (20), 155 (10), 130 (10), 129 (44), 116 (100), 111 (11), 101 (10), 98 (11), 97 (15), 95 (12), 85 (21), 83 (16), 81 (17), 71 (30), 69 (40), 57 (43), 55

(26), 43 (32), 41 (19). HRMS (ESI, [M-H]⁻): calcd. for C₂₀H₂₉O₅: 349.20205; found: 349.20192.

Dimethyl 2-ethyl-4-hydroxy-5-nonylbenzene-1,3-dioate (7s). Starting with **6b** (0.258 g, 1.5 mmol) and **1e** (0.638 g, 1.65 mmol), **7s** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellow oil (0.278 g, 51%). ¹H NMR (250 MHz, CDCl₃): δ = 0.81 (t, ³J = 6.5 Hz, 3 H, (CH₂)₈CH₃), 1.14 (t, ³J = 7.0 Hz, 3 H, CH₃), 1.18 – 1.25 (m, 12 H, 6 CH₂), 1.49 – 1.54 (m, 2 H, CH₂), 2.54 (t, ³J = 7.0 Hz, 2 H, PhCH₂), 3.08 (q, ³J = 7.5 Hz, 2 H, PhCH₂), 3.80 (s, 3 H, OCH₃), 3.91 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.2, 15.1 (CH₃), 21.7, 24.1, 26.4, 28.2, 28.3, 28.5, 28.8, 30.3, 30.8 (CH₂), 50.9, 51.5 (OCH₃), 112.3 (CCOOCH₃), 121.6 (CCOOCH₃), 127.8 (C_{Ar}), 135.0 (CH_{Ar}), 145.5 (C_{Ar}), 161.0 (COH), 167.4, 171.1 (CO). IR (KBr, cm⁻¹): ν̄ = 2925 (s), 2854 (m), 1749 (w), 1717 (m), 1662 (m), 1617 (w), 1577 (w), 1559 (w), 1540 (w), 1507 (w), 1456 (w), 1436 (m), 1331 (w), 1273 (w), 1229 (m), 1203 (m), 1153 (w), 1070 (w), 994 (w), 909 (m), 817 (w), 734 (m), 668 (w), 649 (w). GC-(EI, 70 eV): *m/z* (%) = 364 ([M⁺], 47), 332 (49), 315 (19), 273 (41), 242 (21), 220 (100), 187 (21), 160 (9), 129 (26), 116 (54), 97 (12), 85 (7), 69 (11), 57 (14), 43 (16). HRMS (EI): Calcd. for C₂₁H₃₂O₅ ([M]⁺): 364.22443; found: 364.223933.

Dimethyl 5-decyl-2-ethyl-4-hydroxybenzene-1,3-dioate (7t). Starting with **6b** (0.258 g, 1.5 mmol) and **1f** (0.661 g, 1.65 mmol), **7t** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellow oil (0.278 g, 49%). ¹H NMR (250 MHz, CDCl₃): δ = 0.80 (t, ³J = 6.6 Hz, 3 H, (CH₂)₉CH₃), 1.13 (t, ³J = 8.6 Hz, 3 H, CH₃), 1.16 – 1.24 (m, 14 H, 7 CH₂), 1.46 – 1.54 (m, 2 H, CH₂), 2.53 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 3.08 (q, ³J = 7.4 Hz, 2 H, PhCH₂), 3.79 (s, 3 H, OCH₃), 3.91 (s, 3 H, OCH₃), 7.62 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 13.1, 15.3 (CH₃), 21.8, 24.2, 28.2, 28.3, 28.4, 28.5, 28.6, 28.7, 28.8, 30.9, (CH₂), 50.8, 51.5 (OCH₃), 112.5 (CCOOCH₃), 121.8 (CCOOCH₃), 127.9 (C_{Ar}), 134.9 (CH_{Ar}), 145.5 (C_{Ar}), 161.0 (COH), 167.4, 171.2 (CO). IR (KBr, cm⁻¹): ν̄ = 2953 (m), 2925 (s), 2854 (m), 1723 (m), 1663 (m), 1608 (w), 1578 (w), 1435 (m), 1336 (m), 1275 (m), 1228 (m), 1203 (m), 1153 (m), 1069 (w), 995 (w), 909 (m), 817 (w), 734 (m), 649 (w). GC-MS (EI, 70 eV): *m/z* (%) = 378 ([M⁺], 43), 363 (3), 346 (48), 329 (19), 311

(11), 287 (40), 275 (9), 261 (36), 233 (19), 220 (100), 207 (63), 187 (21), 160 (9), 115 (32), 95 (11), 73 (21), 55 (13), 43 (16). HRMS (EI): Calcd. for $C_{22}H_{34}O_5$ ($[M]^+$): 378.24008; found: 378.240020.

5 **Dimethyl 4-ethyl-2-hydroxy-4'-methylbiphenyl-3,5-dicarboxylate (7u)**. Starting with **6b** (0.258 g, 1.5 mmol) and **1g** (0.525 g, 1.7 mmol), **7u** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light red oil (0.250 g, 51%). 1H NMR (250 MHz, $CDCl_3$): δ = 1.19 (t, 3J = 7.5 Hz, 3 H, CH_2CH_3), 2.31 (s, 3 H, $PhCH_3$), 3.11 (q, 3J = 7.5 Hz, 2 H, CH_2CH_3), 3.79 (s, 3 H, OCH_3), 3.93 (s, 3 H, OCH_3), 7.15 - 7.19 (m, 2 H, 2
10 CH_{Ar}), 7.33 - 7.36 (m, 2 H, 2 CH_{Ar}), 7.82 (s, 1 H, CH_{Ar}), 10.60 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 15.9, 21.2 (CH_3), 25.2 (CH_2), 52.0, 52.7 (OCH_3), 115.2 ($CCOOCH_3$), 122.9 (C_{Ar}), 127.9 ($CCOOCH_3$), 129.1 (2 CH_{Ar}), 129.5 (2 CH_{Ar}), 133.5 (C_{Ar}), 136.5 (CH_{Ar}), 137.5, 147.5 (C_{Ar}), 160.0 (COH), 167.7, 171.6 (CO). IR (neat, cm^{-1}): ν_{max} = 2950 (w), 2874 (w), 1719 (m), 1660 (m), 1605 (m), 1561 (m), 1514 (w), 1431 (m), 1397 (m), 1332 (m), 1293
15 (m), 1231 (s), 1198 (s), 1174 (s), 1081 (m), 1029 (m), 961 (m), 932 (m), 822 (m), 778 (m), 745 (m), 680 (w), 656 (m), 539 (m). GC-MS (EI, 70 eV): m/z (%) = 328 ($[M]^+$, 40), 297 (47), 296 (100), 253 (12), 165 (7). HRMS (EI): Calcd. for $C_{19}H_{20}O_5$ ($[M]^+$): 328.13053; found: 328.130425.

20 **Dimethyl 4-hydroxy-5-methyl-2-propylisophthalate (7v)**. Starting with **6c** (0.279 g, 1.5 mmol) and **1p** (0.457 g, 1.7 mmol), **7v** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light red oil (0.203 g, 51%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.85 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_3CH_3$), 1.41 - 1.50 (m, 2 H, $CH_2CH_2CH_3$), 2.10 (s, 3 H, $PhCH_3$), 2.98 (t, 3J = 8.2 Hz, 2 H, $CH_2CH_2CH_3$), 3.73 (s, 3 H, OCH_3), 3.86 (s, 3 H, OCH_3), 7.58 (s, 1
25 H, CH_{Ar}), 11.09 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.5, 15.8 (CH_3), 22.3, 33.6 (CH_2), 51.9, 52.6 (OCH_3), 113.2 ($CCOOCH_3$), 122.9 (C_{Ar}), 124.4 ($CCOOCH_3$), 136.8 (CH_{Ar}), 145.2 (C_{Ar}), 162.4 (COH), 168.3, 172.3 (CO). IR (neat, cm^{-1}): ν_{max} = 2953 (w), 2927 (w), 2855 (w), 1720 (m), 1658 (m), 1609 (w), 1580 (w), 1432 (m), 1379 (w), 1331 (m), 1265 (m), 1222 (s), 1192 (s), 1150 (s), 1063 (m), 1017 (m), 985 (m), 912 (w), 843 (w), 813 (m), 760 (m), 731
30 (m), 678 (w), 647 (w). GC-MS (EI, 70 eV): m/z (%) = 266 ($[M]^+$, 28), 235 (29), 234 (100),

219 (12), 203 (24), 187 (14), 178 (14), 177 (9), 175 (11), 163 (9), 157 (8), 147 (9), 91 (10), 77 (8). HRMS (EI): Calcd. for $C_{14}H_{18}O_5$ ($[M]^+$): 266.11488; found: 266.114914.

3-Ethyl 1-methyl 5-ethyl-4-hydroxy-2-propylisophthalate (7w). Starting with **6c** (0.279 g, 1.5 mmol) and **1c** (0.499 g, 1.65 mmol), **7w** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.233 g, 53%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.76 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_2CH_3$), 0.99 (t, 3J = 7.5 Hz, 3 H, CH_2CH_3), 1.23 (t, 3J = 7.4 Hz, 3 H, OCH_2CH_3), 1.32 – 1.47 (m, 2 H, $CH_2CH_2CH_3$), 2.44 (q, 3J = 7.6 Hz, 2 H, $PhCH_2CH_3$), 2.91 (q, 3J = 7.4 Hz, 2 H, $PhCH_2CH_2$), 3.65 (s, 3 H, OCH_3), 4.25 (q, 3J = 7.6 Hz, 2 H, OCH_2CH_3), 7.48 (s, 1 H, CH_{Ar}), 11.08 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.8, 15.3, 15.8 (CH_3), 24.2, 26.5, 34.9 (CH_2), 53.2 (OCH_3), 63.4 (OCH_2CH_3), 114.7 ($CCOCH_2CH_3$), 124.1 (C_{Ar}), 131.3 ($CCOCH_3$), 136.3 (CH_{Ar}), 146.1 (C_{Ar}), 163.4 (COH), 169.7, 173.0 (CO). IR (neat, cm^{-1}): ν = 2963 (w), 2933 (w), 2872 (w), 2255 (w), 1716 (m), 1655 (m), 1607 (w), 1578 (w), 1428 (w), 1396 (w), 1373 (w), 1321 (w), 1298 (w), 1263 (w), 1223 (s), 1154 (m), 1097 (w), 1054 (w), 1020 (w), 971 (w), 906 (m), 868 (w), 845 (w), 818 (w), 729 (s), 648 (w), 581 (w). GC-MS (EI, 70 eV): m/z (%) = 294 ($[M]^+$, 38), 263 (14), 249 (22), 248 (100), 233 (17), 230 (14), 217 (21), 215 (11), 198 (17), 192 (48), 191 (11), 177 (12), 173 (12), 171 (13), 115 (10), 91 (13), 77 (10). HRMS (EI): Calcd. for $C_{16}H_{22}O_5$ ($[M]^+$): 294.14618; found: 294.146042.

20

Dimethyl 5-butyl-4-hydroxy-2-propylisophthalate (7x). Starting with **6c** (0.279 g, 1.5 mmol) and **1q** (0.522 g, 1.7 mmol), **7x** was isolated after chromatography (silica gel, heptanes/EtOAc) as a light yellowish oil (0.236 g, 51%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.82 - 0.93 (m, 6 H, 2 CH_3), 1.25 – 1.35 (m, 2 H, $(CH_2)_2CH_2CH_3$), 1.45 – 1.55 (m, 4 H, 2 CH_2), 2.54 (t, 3J = 7.4 Hz, 2 H, $PhCH_2$), 3.03 (t, 3J = 7.4 Hz, 2 H, $PhCH_2$), 3.79 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 13.9, 14.6 (CH_3), 22.6, 25.3, 29.5, 31.4, 33.6 (CH_2), 51.9, 52.5 (OCH_3), 113.4 ($CCOCH_3$), 122.8 (C_{Ar}), 128.7 ($CCOCH_3$), 135.9 (CH_{Ar}), 145.0 (C_{Ar}), 161.9 (COH), 168.3, 172.1 (CO). IR (neat, cm^{-1}): ν = 2955 (w), 2930 (w), 2871 (w), 2255 (w), 1719 (m), 1661 (m), 1606 (w), 1579 (w), 1434 (m), 1335 (w), 1297 (w), 1265 (w), 1226 (m), 1199 (m), 1153 (m), 1088 (w), 1063 (w), 999 (w), 907 (m), 816 (w), 731 (s), 649 (w). GC-MS (EI, 70

eV): m/z (%) = 309 (10), 308 ($[M]^+$, 54), 277 (36), 276 (41), 259 (30), 247 (20), 245 (38), 235 (13), 234 (100), 233 (29), 219 (11), 217 (53), 216 (19), 202 (11), 201 (17), 191 (13), 187 (10), 184 (10), 178 (22), 173 (14), 115 (16), 91 (14), 77 (12). HRMS (ED): Calcd. for $C_{17}H_{24}O_5$ ($[M]^+$): 308.16183; found: 308.162084.

5

Dimethyl 5-hexyl-4-hydroxy-2-propylisophthalate (7y). Starting with **6c** (0.279 g, 1.5 mmol) and **1d** (0.568 g, 1.7 mmol), **7y** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.227 g, 45%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.80 (t, 3J = 7.4 Hz, 3 H, $(CH_2)_5CH_3$), 0.90 (t, 3J = 7.4 Hz, 3 H, $(CH_2)_2CH_3$), 1.17 – 1.32 (m, 6 H, 3 CH_2), 1.43 – 1.56 (m, 4 H, 2 CH_2), 2.53 (t, 3J = 7.4 Hz, 2 H, $PhCH_2$), 3.03 (t, 3J = 7.5 Hz, 2 H, $PhCH_2$), 3.78 (s, 3 H, OCH_3), 3.90 (s, 3 H, OCH_3), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.0, 14.6 (CH_3), 22.6, 25.3, 29.1, 29.2, 29.8, 31.7, 33.6 (CH_2), 51.9, 52.6 (OCH_3), 113.7 ($CCOOCH_3$), 122.9 (C_{Ar}), 128.8 ($CCOOCH_3$), 136.0 (CH_{Ar}), 145.0 (C_{Ar}), 162.3 (COH), 168.4, 172.3 (CO). IR (neat, cm^{-1}): ν = 2954 (w), 2927 (m), 2857 (w), 2255 (w), 1720 (m), 1660 (m), 1606 (w), 1579 (w), 1433 (m), 1331 (w), 1298 (w), 1261 (w), 1226 (s), 1199 (s), 1152 (m), 1092 (w), 1062 (w), 995 (w), 972 (w), 907 (m), 816 (w), 731 (s), 649 (w). GC-MS (EI, 70 eV): m/z (%) = 337 (34), 336 ($[M]^+$, 98), 306 (19), 305 (91), 304 (96), 303 (14), 289 (21), 288 (13), 287 (75), 276 (23), 275 (89), 274 (14), 273 (76), 262 (18), 261 (37), 249 (11), 248 (54), 247 (72), 246 (17), 245 (87), 244 (13), 235 (55), 234 (100), 233 (88), 229 (12), 219 (22), 217 (14), 216 (32), 215 (32), 206 (11), 205 (13), 203 (18), 202 (23), 201 (44), 192 (13), 191 (24), 189 (11), 187 (14), 184 (14), 178 (37), 175 (18), 174 (12), 173 (22), 159 (11), 157 (14), 147 (12), 146 (10), 145 (13), 115 (12), 91 (12). HRMS (ED): Calcd. for $C_{19}H_{28}O_5$ ($[M]^+$): 336.19313; found: 336.192594.

25 **Dimethyl 5-heptyl-4-hydroxy-2-propylisophthalate (7z).** Starting with **6c** (0.279 g, 1.5 mmol) and **1r** (0.591 g, 1.7 mmol), **7z** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.242 g, 46%). 1H NMR (250 MHz, $CDCl_3$): δ = 0.79 (t, 3J = 7.6 Hz, 3 H, $(CH_2)_6CH_3$), 0.91 (t, 3J = 7.5 Hz, 3 H, $(CH_2)_2CH_3$), 1.13 – 1.30 (m, 8 H, 4 CH_2), 1.45 – 1.58 (m, 4 H, 2 CH_2), 2.54 (t, 3J = 7.6 Hz, 2 H, $PhCH_2$), 3.03 (t, 3J = 7.5 Hz, 2 H, $PhCH_2$), 3.79 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 14.0, 14.6 (CH_3), 22.7, 25.4, 29.1, 29.5, 29.6, 29.8,

30

31.8, 33.6 (CH₂), 51.9, 52.5 (OCH₃), 113.6 (CCOOCH₃), 122.8 (C_{Ar}), 128.8 (CCOOCH₃), 135.8 (CH_{Ar}), 145.0 (C_{Ar}), 162.1 (COH), 168.4, 172.2 (CO). IR (neat, cm⁻¹): ν = 2954 (w), 2926 (m), 2855 (w), 1720 (m), 1661 (m), 1607 (w), 1578 (w), 1434 (m), 1329 (w), 1298 (w), 1262 (w), 1226 (s), 1199 (m), 1152 (w), 1093 (w), 1063 (w), 994 (w), 908 (w), 816 (w), 732 (s), 649 (w). GC-MS (EI, 70 eV): m/z (%) = 351 (13), 350 ([M]⁺, 56), 319 (41), 318 (43), 301 (34), 289 (10), 287 (36), 276 (12), 275 (50), 262 (10), 261 (12), 259 (43), 248 (11), 247 (25), 235 (17), 234 (100), 233 (44), 219 (11), 216 (12), 203 (11), 202 (12), 201 (21), 191 (14), 178 (22), 173 (14), 145 (10). HRMS (EI): Calcd. for C₂₀H₃₀O₅ ([M]⁺): 350.20878; found: 350.208521.

10

Dimethyl 4-hydroxy-5-octyl-2-propylisophthalate (7aa). Starting with **6c** (0.279 g, 1.5 mmol) and **1s** (0.614 g, 1.7 mmol), **7aa** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.246 g, 45%). ¹H NMR (250 MHz, CDCl₃): δ = 0.80 (t, ³J = 7.4 Hz, 3 H, (CH₂)₇CH₃), 0.90 (t, ³J = 7.6 Hz, 3 H, (CH₂)₂CH₃), 1.17 – 1.30 (m, 10 H, 5 CH₂), 1.46 – 1.57 (m, 4 H, 2 CH₂), 2.53 (t, ³J = 7.6 Hz, 2 H, PhCH₂), 3.03 (t, ³J = 7.5 Hz, 2 H, PhCH₂), 3.79 (s, 3 H, OCH₃), 3.90 (s, 3 H, OCH₃), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.0, 14.6 (CH₃), 22.6, 25.3, 29.1, 29.2, 29.4, 29.5, 29.8, 31.8, 33.6 (CH₂), 51.8, 52.5 (OCH₃), 113.7 (CCOOCH₃), 123.1 (C_{Ar}), 129.0 (CCOOCH₃), 136.1 (CH_{Ar}), 145.4 (C_{Ar}), 162.1 (COH), 168.2, 172.2 (CO). IR (neat, cm⁻¹): ν = 2954 (w), 2926 (m), 2855 (w), 2256 (w), 1721 (w), 1662 (m), 1606 (w), 1579 (w), 1434 (m), 1331 (w), 1298 (w), 1262 (w), 1227 (m), 1200 (m), 1152 (w), 1094 (w), 1062 (w), 995 (w), 908 (m), 817 (w), 733 (s), 649 (w). GC-MS (EI, 70 eV): m/z (%) = 364 ([M]⁺, 47), 333 (42), 332 (73), 315 (28), 301 (33), 289 (11), 276 (21), 275 (74), 273 (53), 261 (13), 248 (17), 247 (32), 235 (26), 234 (100), 233 (69), 228 (16), 219 (14), 217 (11), 216 (22), 215 (10), 203 (13), 202 (16), 201 (32), 191 (14), 185 (11), 184 (11), 179 (11), 178 (25), 175 (14), 173 (17), 158 (10), 157 (12), 155 (13), 147 (10), 145 (10), 130 (11), 129 (45), 117 (13), 116 (85), 115 (11), 101 (10), 98 (11), 91 (11), 85 (19), 83 (12), 81 (10), 71 (30), 69 (25), 57 (42), 55 (23). HRMS (EI): Calcd. for C₂₁H₃₂O₅ ([M]⁺): 364.22443; found: 364.224478.

Dimethyl 4-hydroxy-5-nonyl-2-propylbenzene-1,3-dioate (7ab). Starting with **6c** (0.279 g, 1.5 mmol) and **1e** (0.638 g, 1.7 mmol), **7ab** was isolated after chromatography

(silica gel, heptanes/EtOAc) as a yellowish oil (0.284 g, 50%). ^1H NMR (250 MHz, CDCl_3): δ = 0.80 (t, 3J = 6.4 Hz, 3 H, $(\text{CH}_2)_8\text{CH}_3$), 0.91 (t, 3J = 7.5 Hz, 3 H, $(\text{CH}_2)_2\text{CH}_3$), 1.15 – 1.26 (m, 14 H, 7 CH_2), 1.52 – 1.54 (m, 2 H, CH_2), 2.54 (t, 3J = 7.5 Hz, 2 H, PhCH_2), 3.00 – 3.06 (m, 2 H, PhCH_2), 3.79 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): δ = 13.0, 13.9 (CH_3), 21.9, 24.4, 26.1, 28.2, 28.3, 28.5, 28.8, 30.3, 30.9 32.6 (CH_2), 50.9, 51.5 (OCH_3), 112.6 (CCOOCH_3), 121.9 (CCOOCH_3), 127.9 (C_{Ar}), 135.1 (CH_{Ar}), 144.2 (C_{Ar}), 161.2 (COH), 167.6, 171.2 (CO). IR (KBr, cm^{-1}): ν = 2954 (m), 2925 (s), 2854 (m), 1722 (m), 1662 (m), 1608 (w), 1578 (w), 1435 (m), 1330 (w), 1299 (w), 1262 (m), 1227 (s), 1200 (m), 1153 (m), 1094 (w), 1063 (w), 995 (w), 908 (m), 817 (w), 733 (s), 650 (w). GC-MS (EI, 70 eV): m/z (%) = 378 ($[\text{M}^+]$, 50), 368 (5), 346 (57), 329 (21), 275 (40), 242 (22), 234 (100), 201 (17), 178 (18), 158 (12), 129 (37), 116 (78), 97 (16), 85 (12), 69 (17), 57 (23), 43 (21). HRMS (EI): Calcd. for $\text{C}_{22}\text{H}_{34}\text{O}_5$ ($[\text{M}^+]$): 378.24008; found: 378.239947.

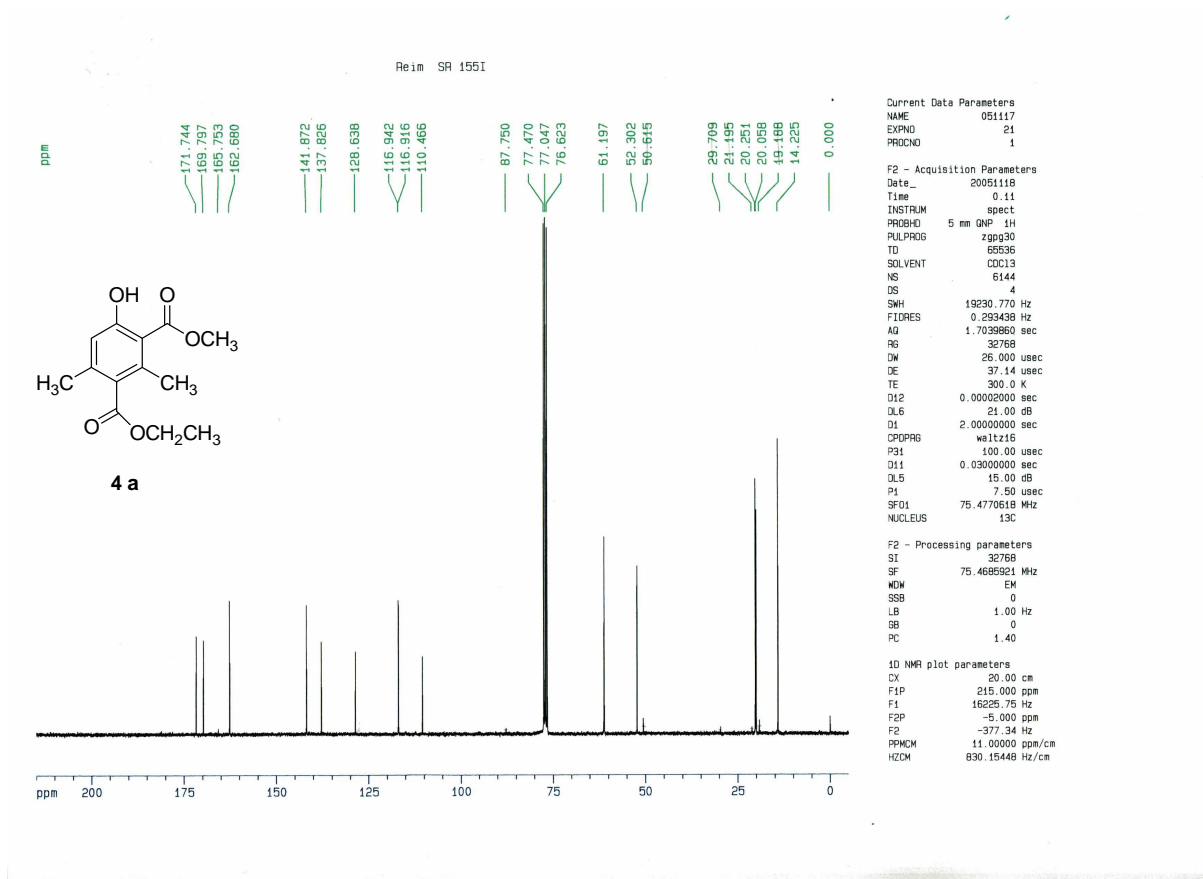
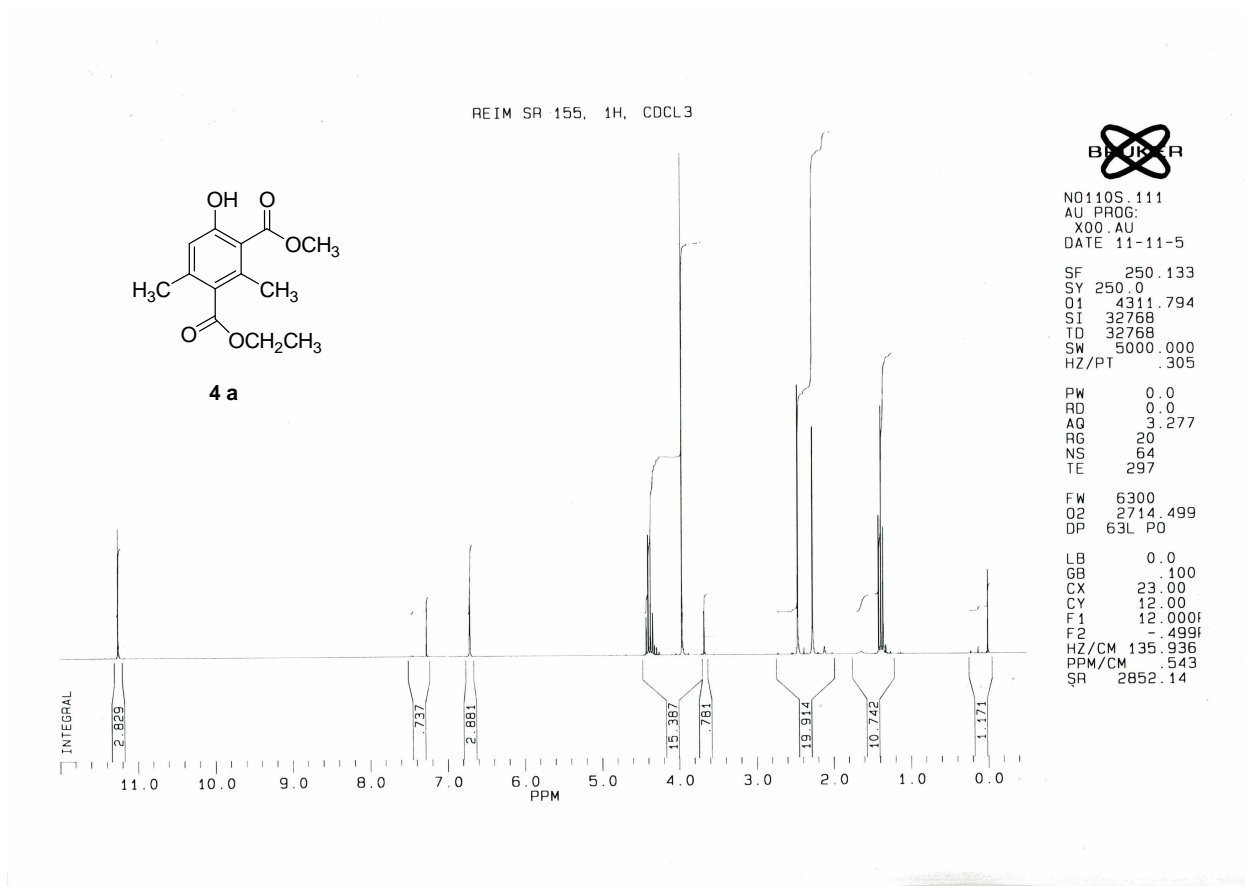
Dimethyl 5-decyl-4-hydroxy-2-propylbenzene-1,3-dioate (7ac). Starting with **6c** (0.279 g, 1.5 mmol) and **1f** (0.661 g, 1.7 mmol), **7ac** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.323 g, 55%). ^1H NMR (250 MHz, CDCl_3): δ = 0.80 (t, 3J = 6.5 Hz, 3 H, $(\text{CH}_2)_9\text{CH}_3$), 0.91 (t, 3J = 7.2 Hz, 3 H, $(\text{CH}_2)_2\text{CH}_3$), 1.16 – 1.26 (m, 16 H, 8 CH_2), 1.49 – 1.54 (m, 2 H, CH_2), 2.53 (t, 3J = 7.7 Hz, 2 H, PhCH_2), 3.01 – 3.06 (m, 2 H, PhCH_2), 3.79 (s, 3 H, OCH_3), 3.91 (s, 3 H, OCH_3), 7.61 (s, 1 H, CH_{Ar}), 11.07 (s, 1 H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): δ = 13.9, 14.7 (CH_3), 22.7, 25.2, 29.2, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 31.9, 33.8 (CH_2), 51.9, 52.7 (OCH_3), 113.3 (CCOOCH_3), 122.9 (CCOOCH_3), 128.8 (C_{Ar}), 136.0 (CH_{Ar}), 145.1 (C_{Ar}), 162.0 (COH), 168.4, 172.2 (CO). IR (KBr, cm^{-1}): ν = 2954 (m), 2955 (m), 2854 (m), 1723 (m), 1662 (m), 1606 (w), 1579 (w), 1434 (m), 1331 (w), 1298 (w), 1261 (w), 1227 (m), 1200 (m), 1152 (w), 1095 (w), 1061 (w), 996 (w), 908 (w), 817 (w), 734 (m), 650 (w). GC-MS (EI, 70 eV): m/z (%) = 392 ($[\text{M}^+]$, 50), 360 (43), 343 (20), 301 (30), 275 (31), 247 (20), 234 (100), 219 (8), 201 (15), 178 (18), 145 (7), 116 (12), 91 (5), 69 (4), 55 (7), 43 (11). HRMS (EI): Calcd. for $\text{C}_{23}\text{H}_{36}\text{O}_5$ ($[\text{M}^+]$): 392.25573; found: 392.255638.

6-Ethyl 2-methyl 3-hydroxy-4-methylbiphenyl-2,6-dicarboxylate (7ad). Starting with **6d** (0.372 g, 1.5 mmol) and **1p** (0.457 g, 1.7 mmol), **7ad** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.272 g, 58%). ¹H NMR (300 MHz, CDCl₃): δ = 1.17 (t, ³J = 7.0 Hz, 3 H, OCH₂CH₃), 2.24 (s, 3 H, PhCH₃), 3.89 (s, 3 H, OCH₃), 4.14 (q, ³J = 7.5 Hz, 2 H, OCH₂CH₃), 7.02 - 7.06 (m, 3 H, 3 CH_{Ar}), 7.33 - 7.36 (m, 2 H, 2 CH_{Ar}), 7.88 (s, 1 H, CH_{Ar}), 11.04 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 14.0, 15.8 (CH₃), 51.9 (OCH₃), 61.4 (OCH₂), 112.7 (CCOOCH₃), 123.9 (CCOOC₂H₅), 126.0 (C_{Ar}), 128.2 (CH_{Ar}), 128.4 (2 CH_{Ar}), 128.7 (2 CH_{Ar}), 133.7 (CH_{Ar}), 135.6, 135.9 (C_{Ar}), 167.5 (COH), 171.4, 173.1 (CO). IR (KBr, cm⁻¹): ν̄ = 3059 (w), 2981 (w), 2953 (w), 2905 (w), 2872 (w), 1737 (m), 1686 (m), 1662 (m), 1597 (m), 1578 (m), 1494 (w), 1381 (w), 1366 (m), 1264 (m), 1193 (s), 1148 (s), 1076 (m), 1022 (m), 998 (m), 942 (w), 870 (w), 814 (m), 755 (m), 687 (s), 647 (m), 572 (m). GC-MS (EI, 70 eV): *m/z* (%) = 314 ([M]⁺, 51), 283 (20), 282 (100), 254 (22), 253 (96), 237 (27), 210 (12), 209 (47), 208 (23), 181 (10), 153 (15), 152 (10). HRMS (EI): Calcd. for C₁₈H₁₈O₅ ([M]⁺): 314.11488; found: 314.114952.

15

Diethyl 4-ethyl-3-hydroxybiphenyl-2,6-dicarboxylate (7ae). Starting with **6d** (0.372 g, 1.5 mmol) and **1c** (0.499 g, 1.7 mmol), **7ae** was isolated after chromatography (silica gel, heptanes/EtOAc) as a yellowish oil (0.308 g, 60%). ¹H NMR (300 MHz, CDCl₃): δ = 0.57 (t, ³J = 7.3 Hz, 3 H, OCH₂CH₃), 0.77 (t, ³J = 7.4 Hz, 3 H, OCH₂CH₃), 1.16 (t, ³J = 7.3 Hz, 3 H, PhCH₂CH₃), 2.61 (q, ³J = 7.5 Hz, 2 H, PhCH₂CH₃), 3.74 - 3.85 (m, 4 H, 2 OCH₂CH₃), 6.99 - 7.04 (m, 2 H, 2 CH_{Ar}), 7.17 - 7.19 (m, 3 H, 3 CH_{Ar}), 7.62 (s, 1 H, CH_{Ar}), 11.17 (s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ = 12.8, 13.4, 13.6 (CH₃), 22.9, 60.7, 61.3 (CH₂), 113.0 (CCOOCH₃), 124.5 (CCOOC₂H₅), 126.6 (CH_{Ar}), 127.1 (2 CH_{Ar}), 128.3 (2 CH_{Ar}), 131.9 (CH_{Ar}), 134.4, 141.2, 142.3 (C_{Ar}), 161.5 (COH), 168.4, 171.3 (CO). IR (KBr, cm⁻¹): ν̄ = 3058 (w), 2978 (w), 2935 (w), 2874 (w), 2254 (w), 1708 (m), 1657 (m), 1602 (w), 1571 (w), 1443 (m), 1398 (w), 1367 (m), 1329 (m), 1306 (m), 1281 (m), 1265 (m), 1214 (s), 1178 (s), 1125 (m), 1095 (w), 1066 (w), 1020 (m), 907 (m), 884 (w), 866 (w), 819 (w), 763 (w), 728 (s), 698 (s), 669 (w), 648 (w). GC-MS (EI, 70 eV): *m/z* (%) = 342 ([M]⁺, 35), 297 (16), 296 (51), 268 (19), 267 (100), 251 (12), 223 (36), 222 (11), 165 (13), 152 (13). HRMS (EI): Calcd. for C₂₀H₂₂O₅ ([M]⁺): 342.14618; found: 342.146145.

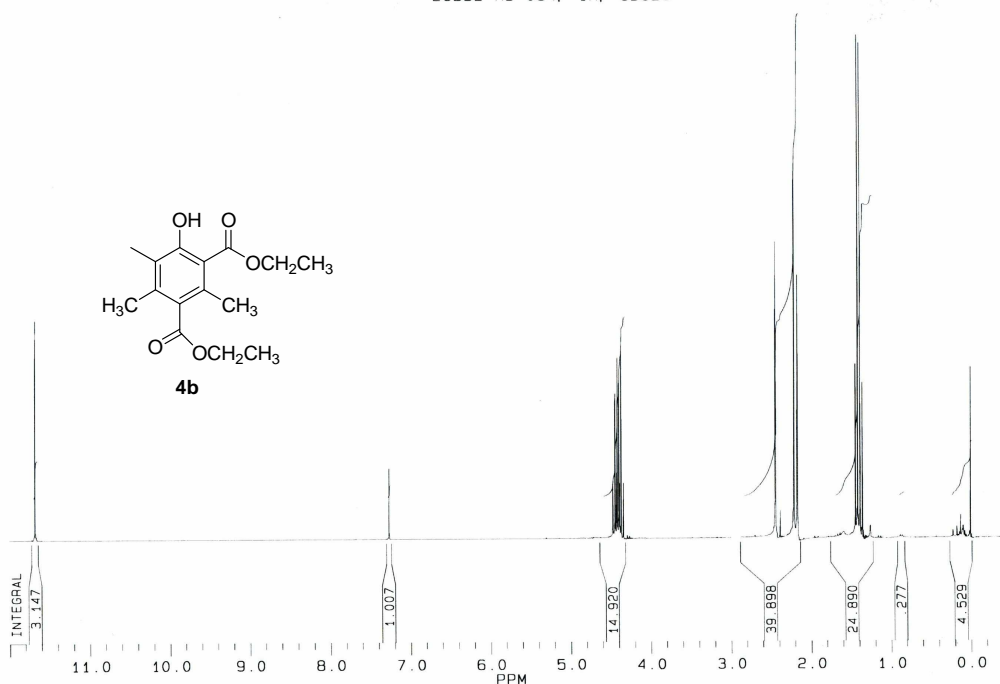
30



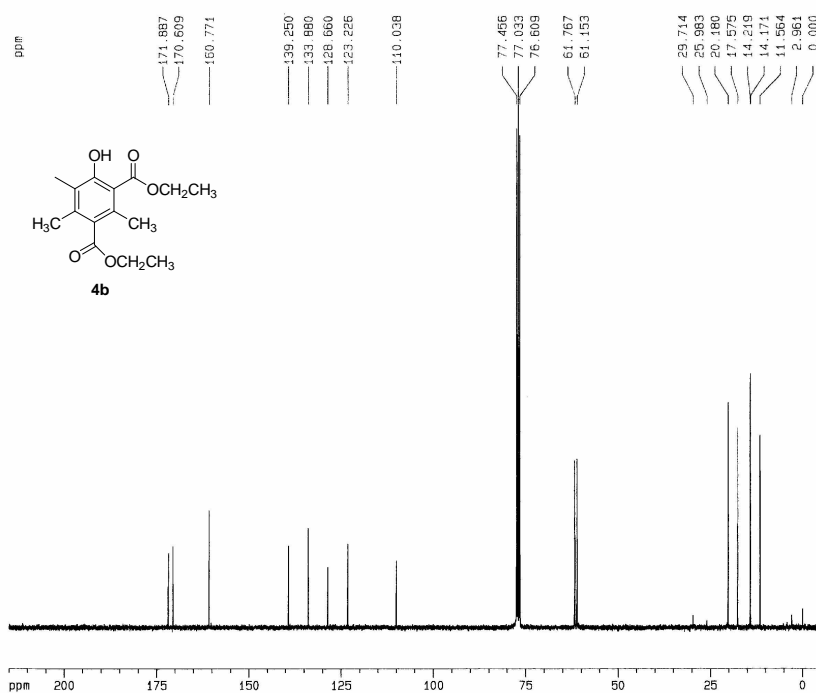
LUBBE MD 034, 1H, CDCL3



DZ0905_104
AU PROG:
X00.AU
DATE 9-12-5
SF 250.133
SY 250.0
Q1 4311.794
SI 32768
TD 32768
SW 5000.000
HZ/PT .305
PW 0.0
RD 0.0
AQ 3.277
RG 20
NS 56
TE 297
FW 6300
Q2 2714.499
DP 63L P0
LB 0.0
GB .100
CX 23.00
CY 12.00
F1 12.000f
F2 .499f
HZ/CM 135.936
PPM/CM .543
SR 2853.36

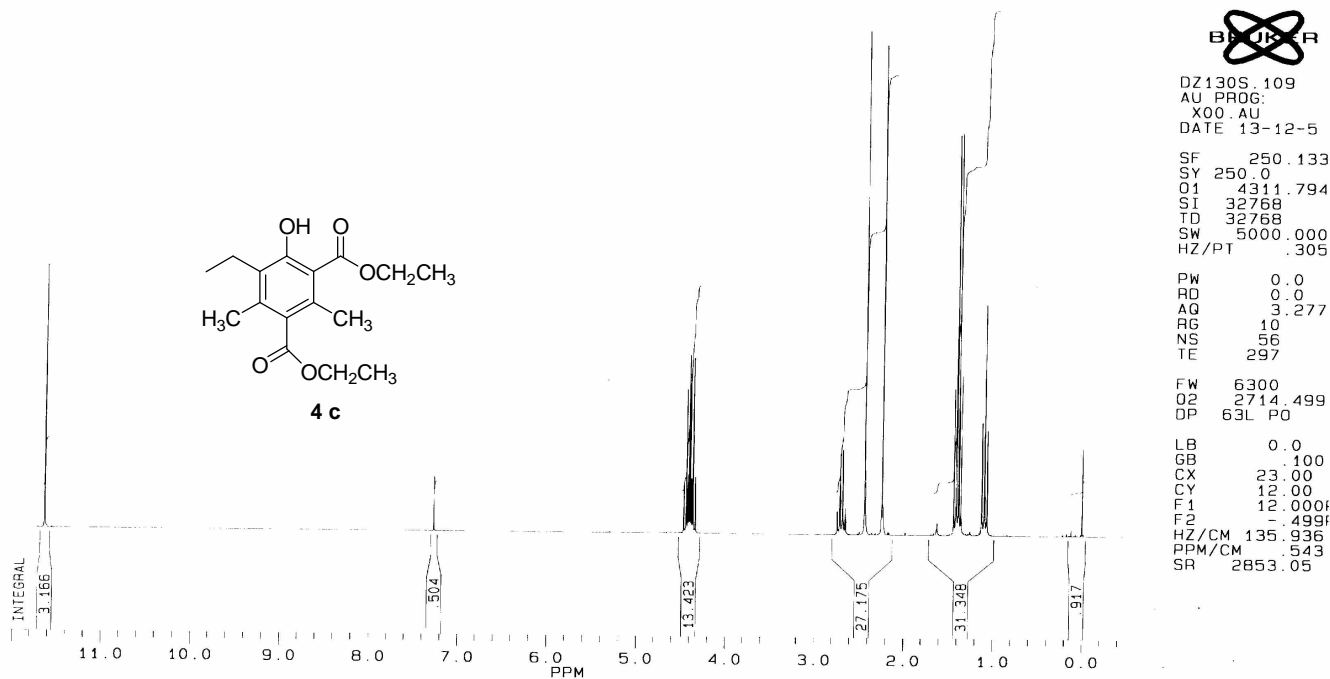


Lubbe MD034

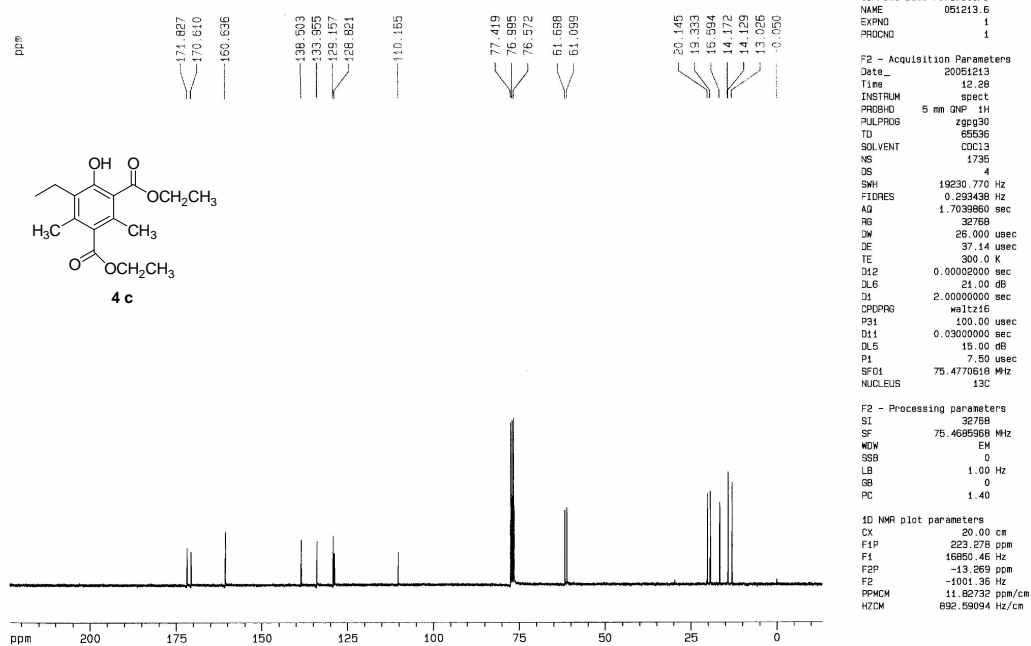


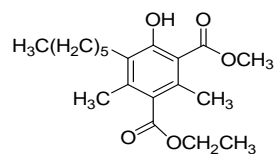
Current Data Parameters
NAME 051209
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051209
Time 15.36
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
DS 4
SMH 19230.770 Hz
FIDRES 0.293438 Hz
AQ 1.703960 sec
RG 32768
DM 26.000 usec
DE 37.14 usec
TE 300.0 K
D12 0.0002000 sec
DL6 21.00 dB
D1 2.0000000 sec
CPDPRG waltz16
P21 100.00 usec
D11 0.0300000 sec
DL5 15.00 dB
P1 7.50 usec
SFO1 75.4770518 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 32768
SF 75.4685921 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16225.75 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.15448 Hz/cm

LUBBE MD 036, 1H, CDCL3

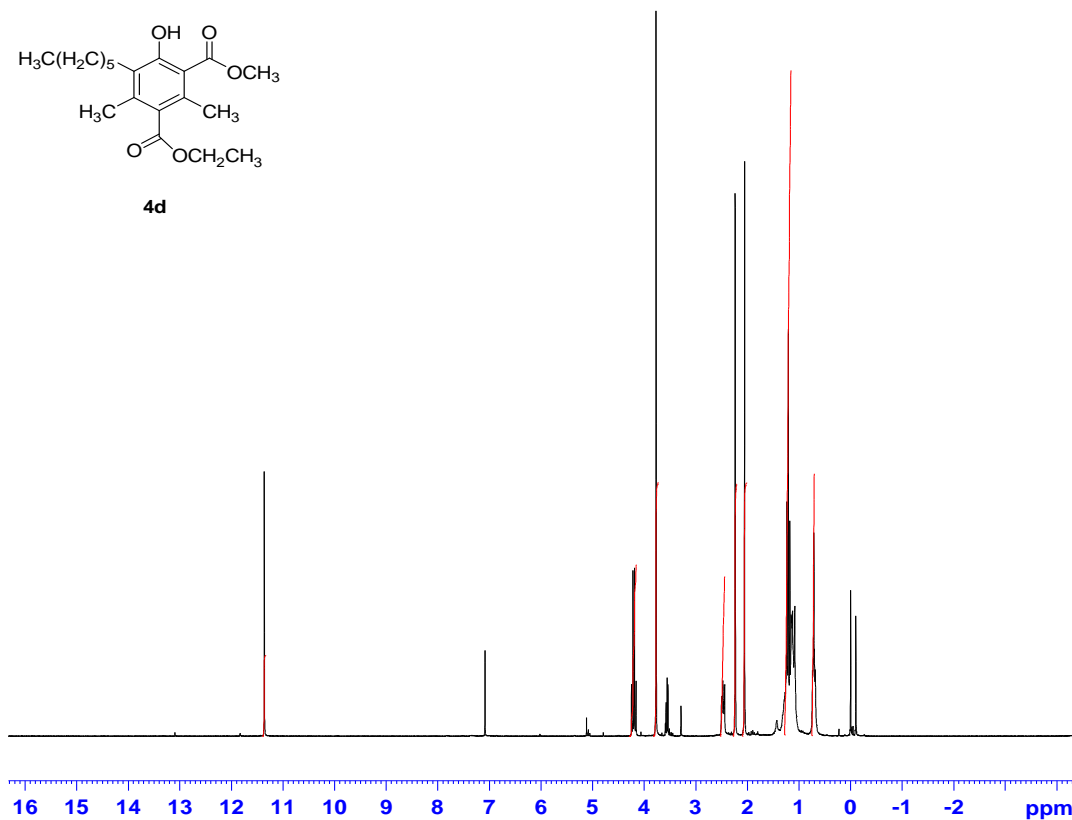


Lubbe MD 036, 13C





4d



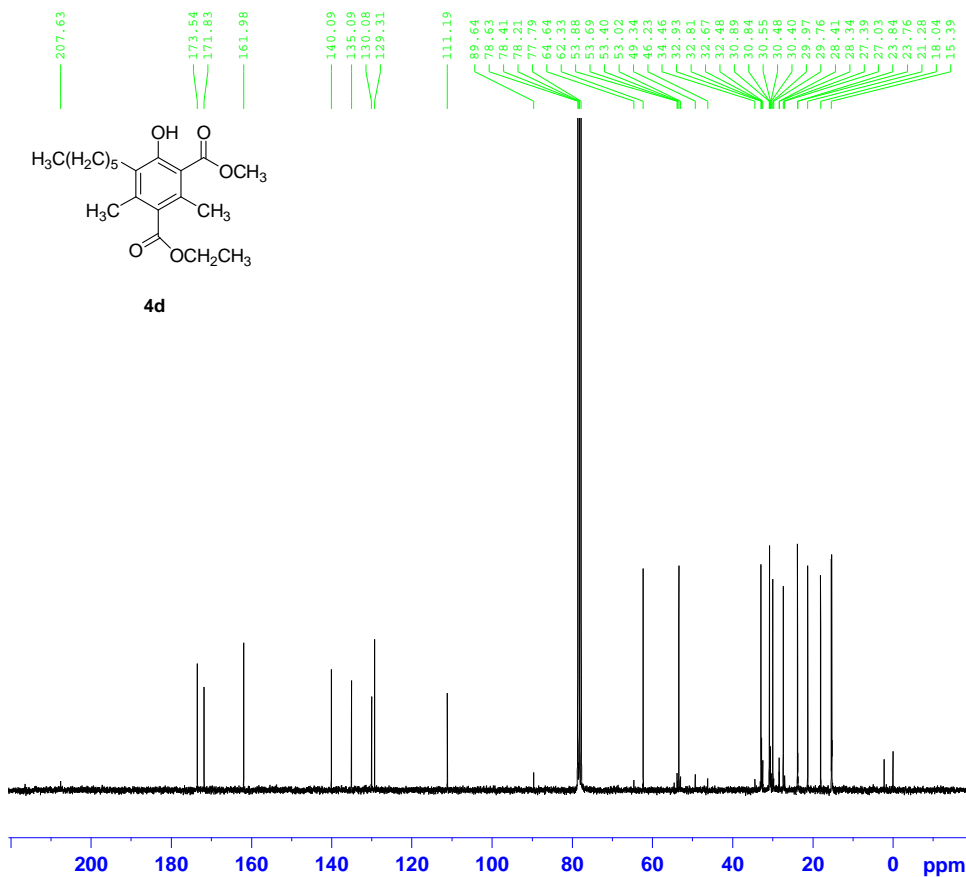
```

Current Data Parameters
NAME          071112.225
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_         20071112
Time          11.11
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          5165.289 Hz
FIDRES      0.078816 Hz
AQ          6.3439350 sec
RG           406
DM           96.800 usec
DE           10.00 usec
TE           297.9 K
D1           1.00000000 sec
TDO          1

***** CHANNEL f1 *****
NUC1         1H
P1           11.40 usec
PL1          -3.00 dB
SFO1        250.1315447 MHz

F2 - Processing parameters
SI           32768
SF          250.1300441 MHz
WDW          EM
SSB          0
LB           0.50 Hz
GB           0
PC           1.00
    
```



```

Current Data Parameters
NAME          071113.u303
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_         20071113
Time          13.27
INSTRUM      spect
PROBHD       5 mm PABBO BH-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          18028.646 Hz
FIDRES      0.275098 Hz
AQ          1.8175818 sec
RG           2050
DM           27.733 usec
DE           10.00 usec
TE           298.3 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TDO          1

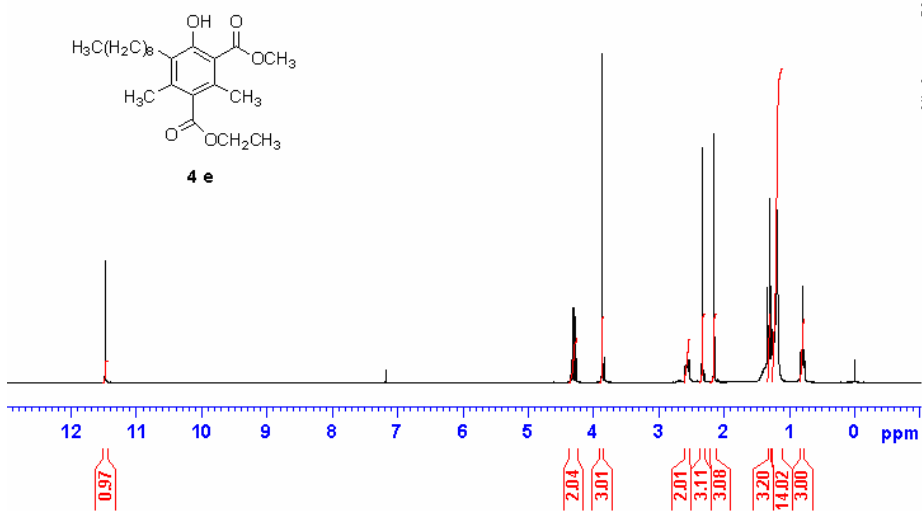
***** CHANNEL f1 *****
NUC1         13C
P1           10.00 usec
PL1          -0.50 dB
SFO1        75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2      waltz16
NUC2         1H
PCPD2       72.00 usec
PL12         17.00 dB
PL13         17.00 dB
PL2          0.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI           32768
SF          75.4676578 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```



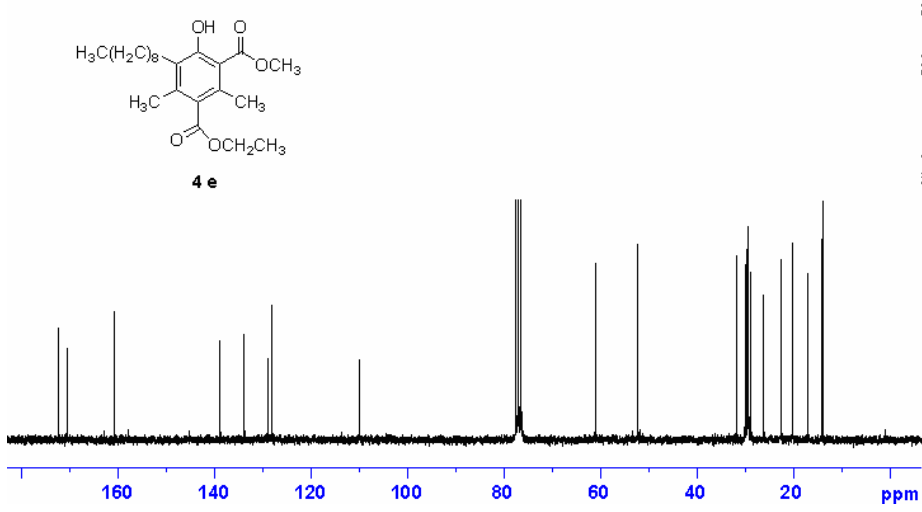
Current Data Parameters
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20080915
 Time 11:29
 INSTRUM spect
 PROCNO 5
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5262.249 Hz
 F2 FREQS 400.146360 MHz
 AQ 6.2450000 sec
 RG 322
 DR 96.488 mmcs
 DE 18.81 mmcs
 TE 300.2 K
 D1 1.80000000 sec
 TDR 1



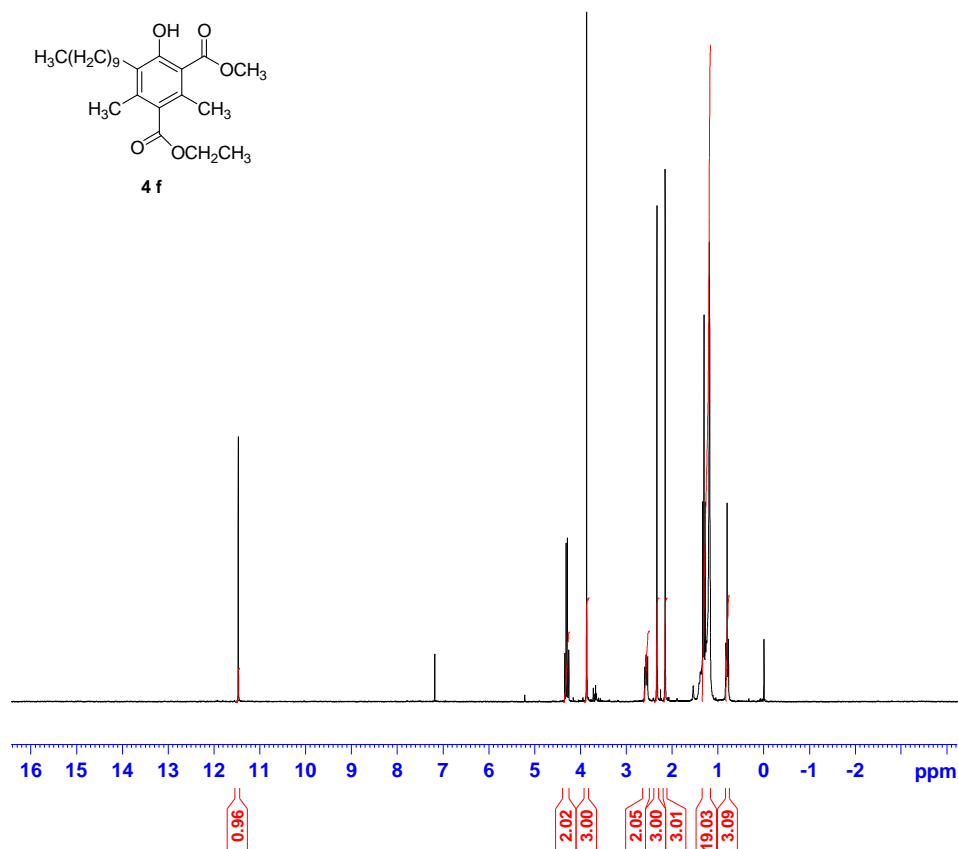
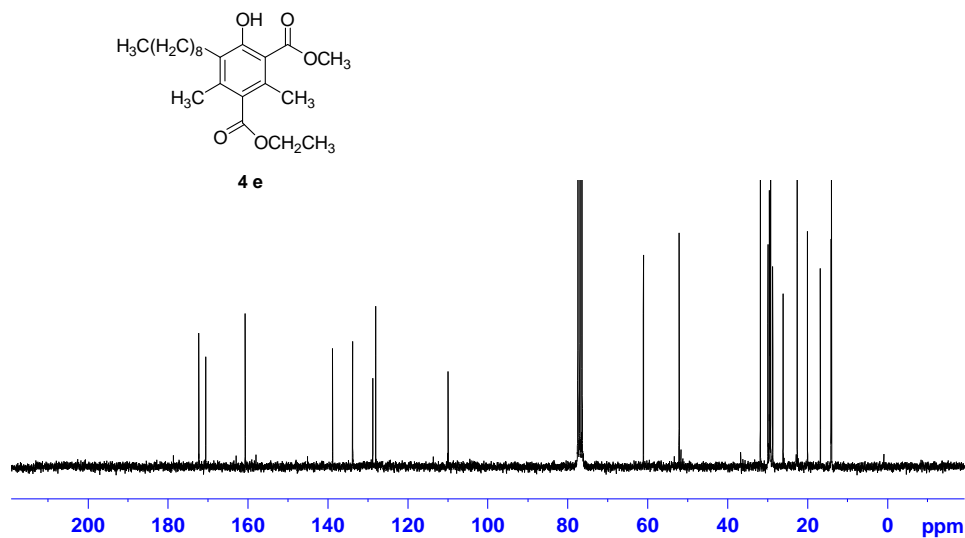
----- CHANNEL F1 -----
 FQ1 30.00 MHz
 PL1 -5.00 dB
 SFO1 258.1328441 MHz
 F2 - Processing parameters:
 SI 32768
 SF 258.1328441 MHz
 WHW 60
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

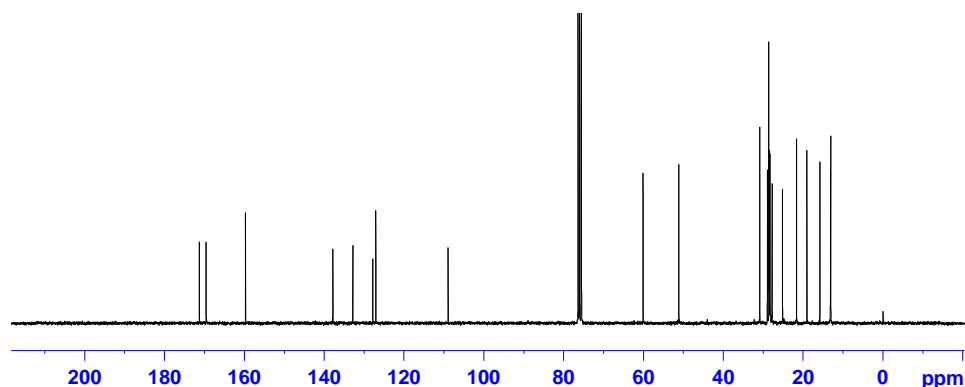
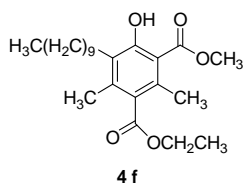


Current Data Parameters
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20080915
 Time 11:29
 INSTRUM spect
 PROCNO 5
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5262.249 Hz
 F2 FREQS 400.146360 MHz
 AQ 6.2450000 sec
 RG 322
 DR 96.488 mmcs
 DE 18.81 mmcs
 TE 300.2 K
 D1 1.80000000 sec
 TDR 1



----- CHANNEL F1 -----
 FQ1 30.00 MHz
 PL1 -5.00 dB
 SFO1 258.1328441 MHz
 ----- CHANNEL F2 -----
 CFREQ2 400.146360 MHz
 FQ2 30.00 MHz
 PL2 14.00 dB
 PL3 14.00 dB
 PL4 -5.00 dB
 SFO2 258.1328441 MHz
 F2 - Processing parameters:
 SI 32768
 SF 400.1463600 MHz
 WHW 60
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00





```

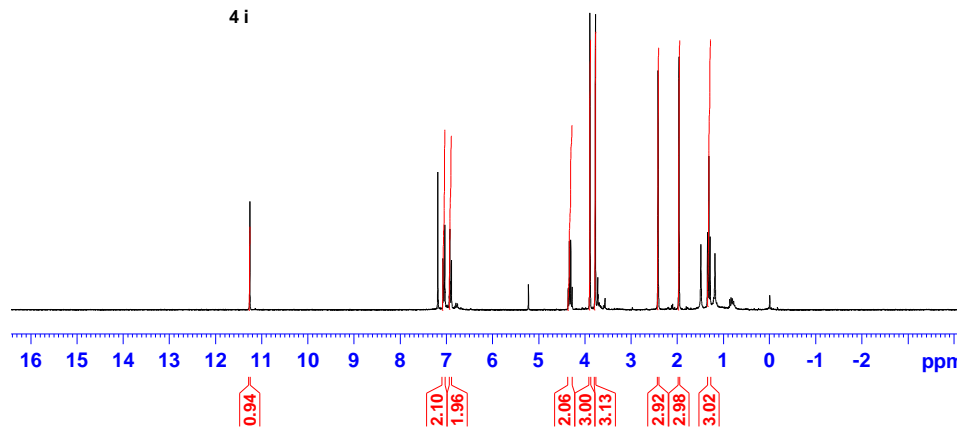
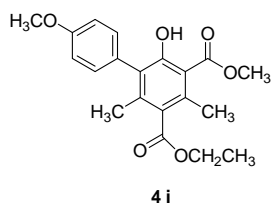
Current Data Parameters
NAME          080930.u311
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_        20080930
Time         21.08
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          18028.846 Hz
FIDRES       0.275998 Hz
AQ           1.8178818 sec
RG           1030
RW           27.733 usec
DE           6.50 usec
TE           298.2 K
D1           2.0000000 sec
D11          0.0300000 sec
TDO          1

***** CHANNEL f1 *****
NUC1          13C
P1           10.00 usec
PL1          -0.50 dB
PL1W         33.25691896 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
PL1W         0.22453187 W
PL13W        0.22453187 W
SFO2         300.1312005 MHz

F2 - Processing parameters
SI           32768
SF           75.4678249 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
CB           0
PC           1.40
    
```



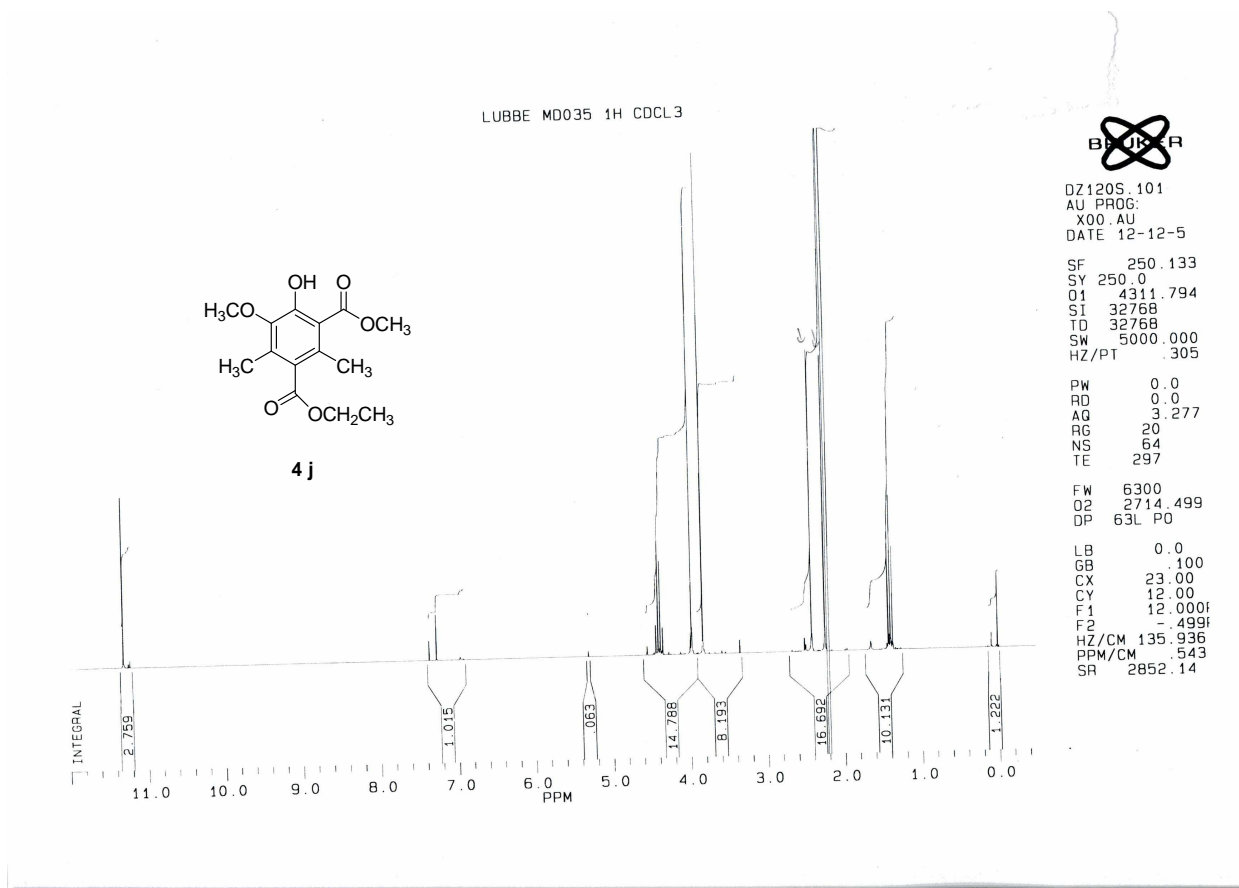
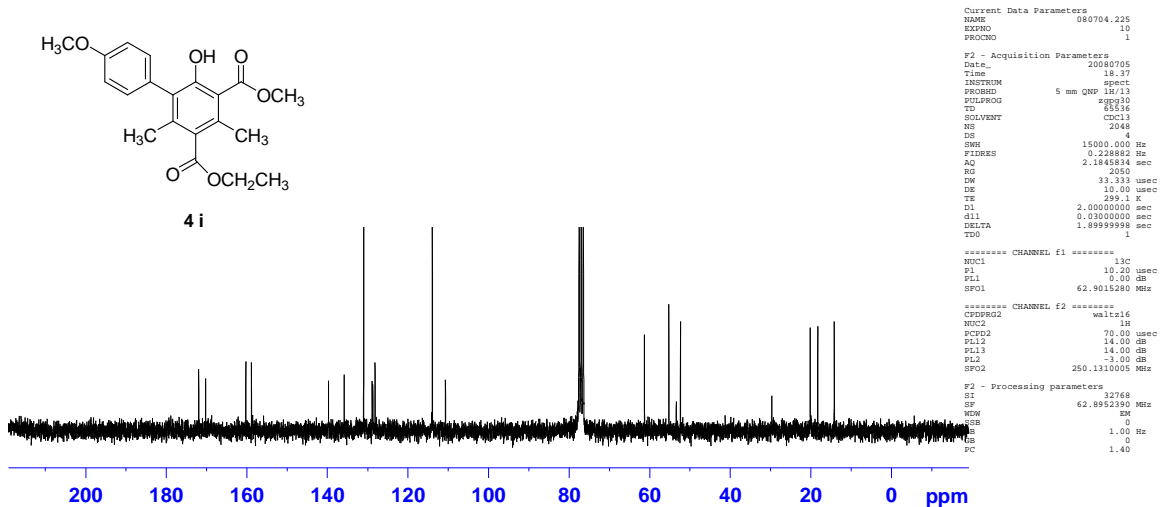
```

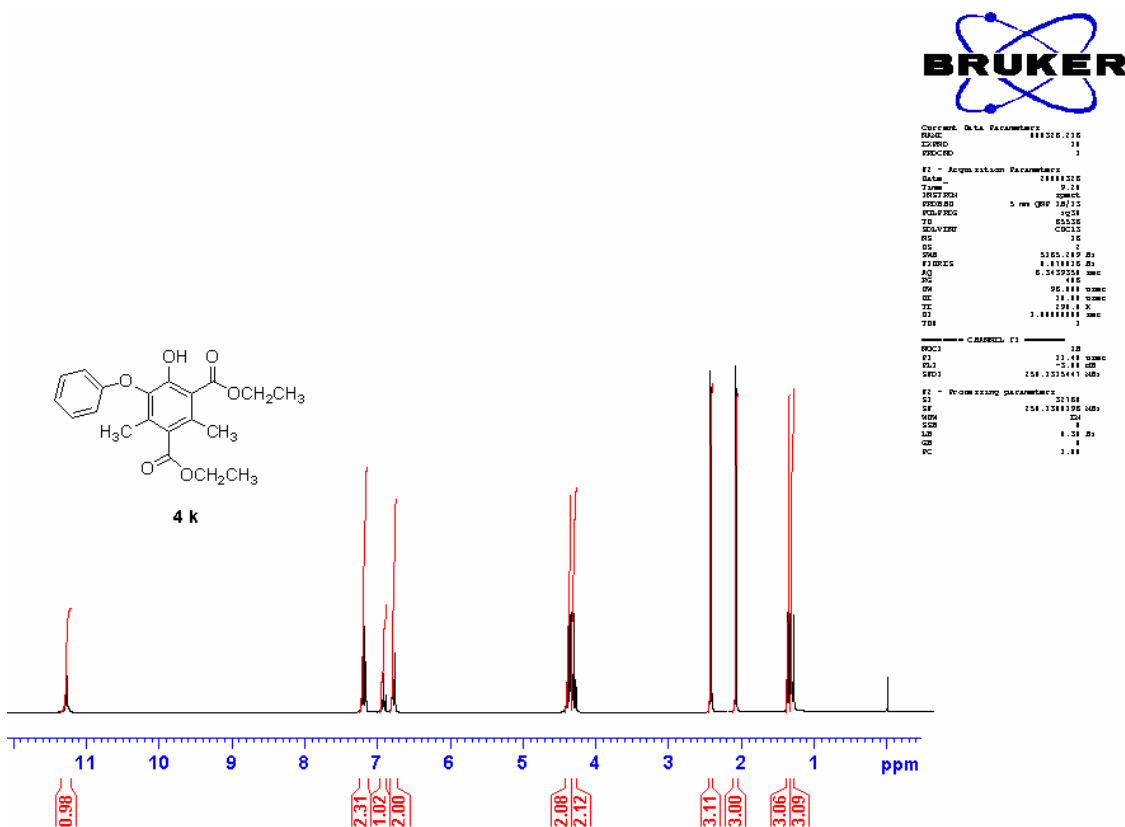
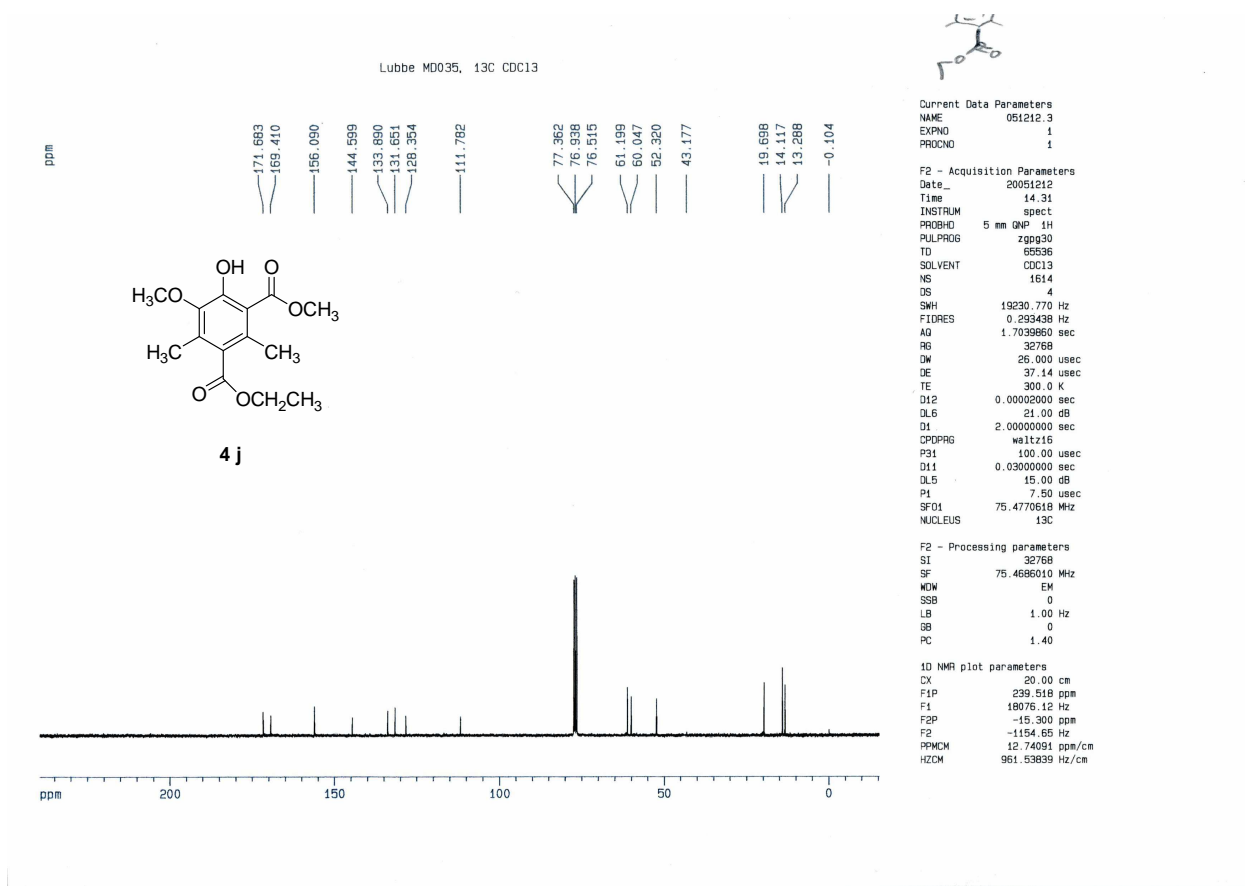
Current Data Parameters
NAME          080704.205
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080704
Time         8.41
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          5165.289 Hz
FIDRES       0.078816 Hz
AQ           6.9439350 sec
RG           1290
RW           96.800 usec
DE           10.00 usec
TE           297.7 K
D1           1.0000000 sec
TDO          1

***** CHANNEL f1 *****
NUC1          1H
P1           11.40 usec
PL1          -3.00 dB
SFO1         250.1315447 MHz

F2 - Processing parameters
SI           32768
SF           250.1300186 MHz
WDW          EM
SSB           0
LB           0.30 Hz
GB           0
CB           1.00
PC           1.00
    
```







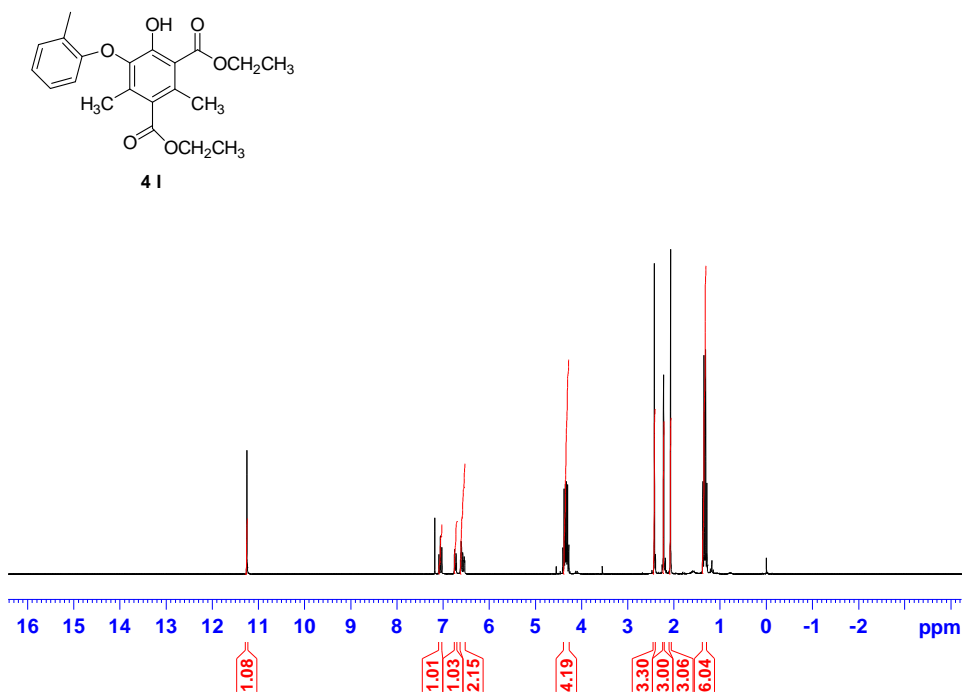
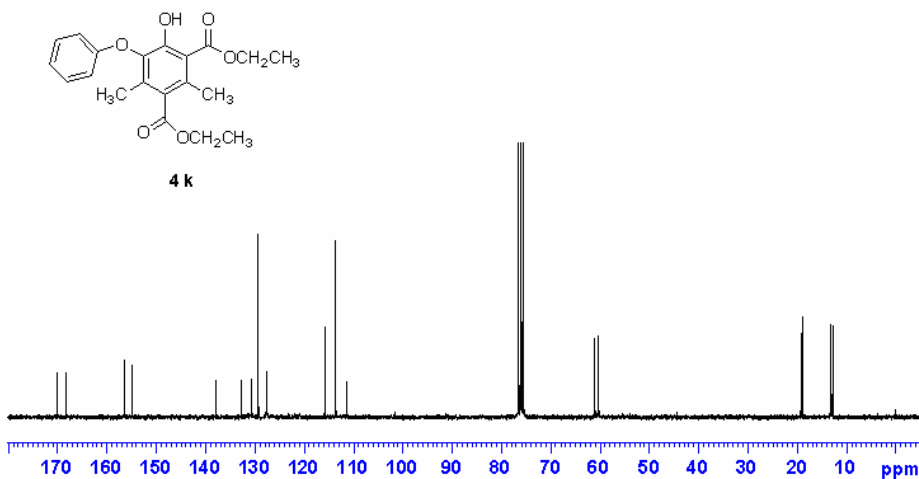
```

Current Data Parameters
NAME      071112.221
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20071112
Time     10.44
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       5165.289 Hz
FIDRES   0.078816 Hz
AQ        6.3439350 sec
RG         512
AW        96.800 usec
DE        10.00 usec
TE        299.0 K
D1        1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      13C
P1         11.40 usec
PL1        -1.00 dB
SFO1      250.1315447 MHz

F2 - Processing parameters
SI         32768
SF         250.130200 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



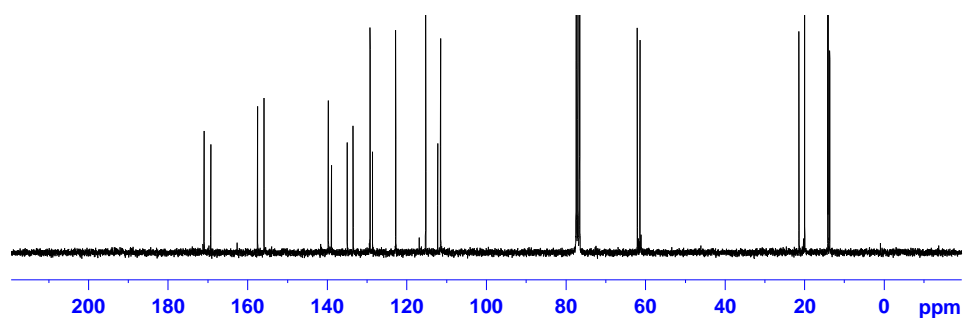
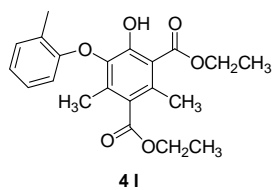
```

Current Data Parameters
NAME      071112.221
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20071112
Time     10.44
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       5165.289 Hz
FIDRES   0.078816 Hz
AQ        6.3439350 sec
RG         512
AW        96.800 usec
DE        10.00 usec
TE        299.0 K
D1        1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      13C
P1         11.40 usec
PL1        -1.00 dB
SFO1      250.1315447 MHz

F2 - Processing parameters
SI         32768
SF         250.130200 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

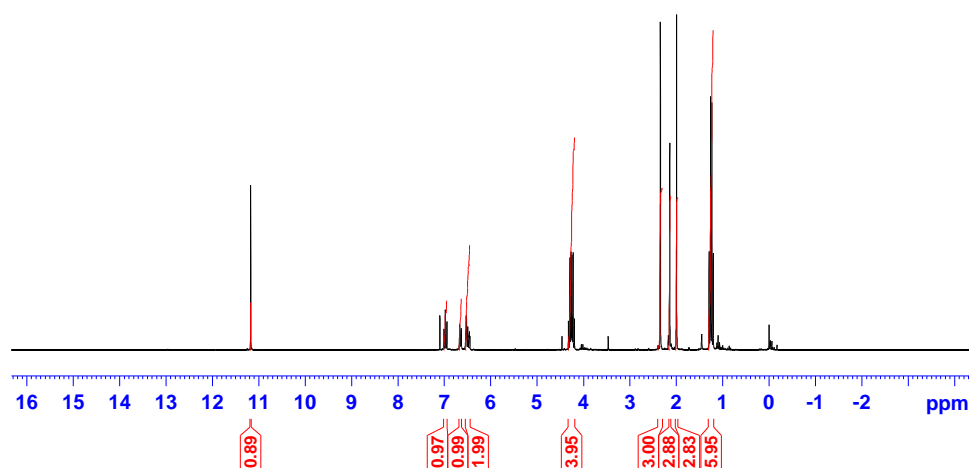
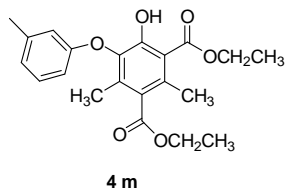
Current Data Parameters
NAME          071113.u307
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20071114
Time         0.45
INSTRUM      spect
PROBHD       5 mm TAMB0 BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          18028.846 Hz
FIDRES      0.275598 Hz
AQ          1.8175818 sec
RG          2050
DW          27.133 usec
DE          10.00 usec
TE          298.4 K
D1          2.0000000 sec
d11         0.0300000 sec
DELTA       1.8999998 sec
TD0         1

***** CHANNEL f1 *****
NUC1         13C
P1          10.00 usec
PL1         -0.50 dB
SFO1        75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2     waltz16
NUC2         1H
PCPD2       72.00 usec
PL12        17.00 dB
PL13        17.00 dB
PL2         0.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI          32768
SF          75.4677490 MHz
RG          662
WDW          EM
SSB          0
LB          1.00 Hz
GB          0
PC          1.40
    
```



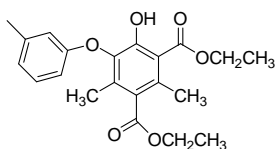
```

Current Data Parameters
NAME          071102.224
EXPNO        10
PROCNO       1

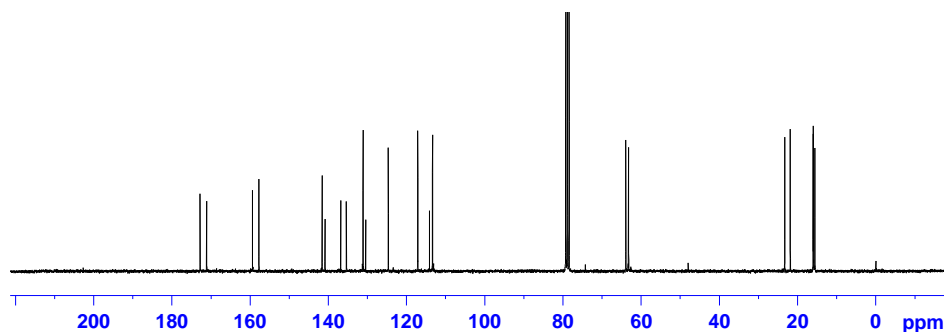
F2 - Acquisition Parameters
Date_        20071102
Time         12.06
INSTRUM      spect
PROBHD       5 mm QNP
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          5165.289 Hz
FIDRES      0.078816 Hz
AQ          6.3439350 sec
RG          662
DW          96.800 usec
DE          10.00 usec
TE          298.0 K
D1          1.0000000 sec
TD0         1

***** CHANNEL f1 *****
NUC1         1H
P1          11.40 usec
PL1         -3.00 dB
SFO1        250.1315447 MHz

F2 - Processing parameters
SI          32768
SF          250.1300415 MHz
RG          662
WDW          EM
SSB          0
LB          0.30 Hz
GB          0
PC          1.00
    
```

4 m



```

Current Data Parameters
NAME      071105.u302
EXPNO    1
PROCNO   1

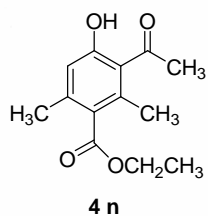
F2 - Acquisition Parameters
Date_    20071105
Time     13.03
INSTRUM  spect
PROBHD   5 mm PABBO 5H
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1536
DS       4
SWH      18028.846 Hz
FIDRES   0.276598 Hz
AQ       1.8775818 sec
RG       2050
DW       27.733 usec
DE       10.00 usec
TE       298.2 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999999 sec
T30      1

***** CHANNEL f1 *****
NUC1      13C
P1        10.00 usec
PL1      -0.50 dB
SFO1      75.4752653 MHz

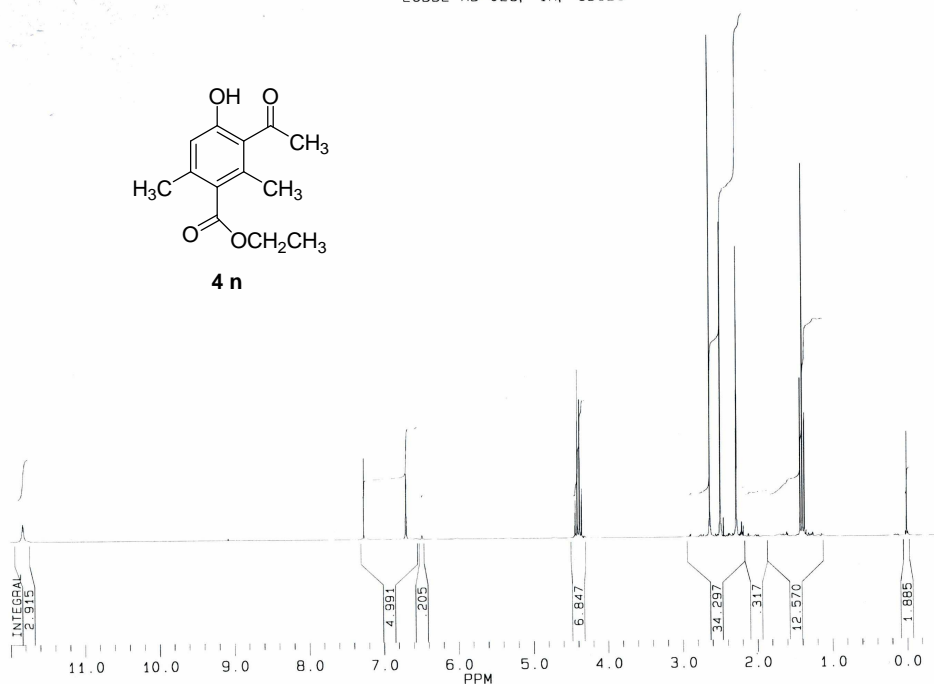
***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPD2     72.00 usec
PL12     17.00 dB
PL13     17.00 dB
PL2      0.00 dB
SFO2     300.1312005 MHz

F2 - Processing parameters
SI        32768
SF        75.4676069 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

LUBBE MD 029, 1H, CDCl3



4 n



```

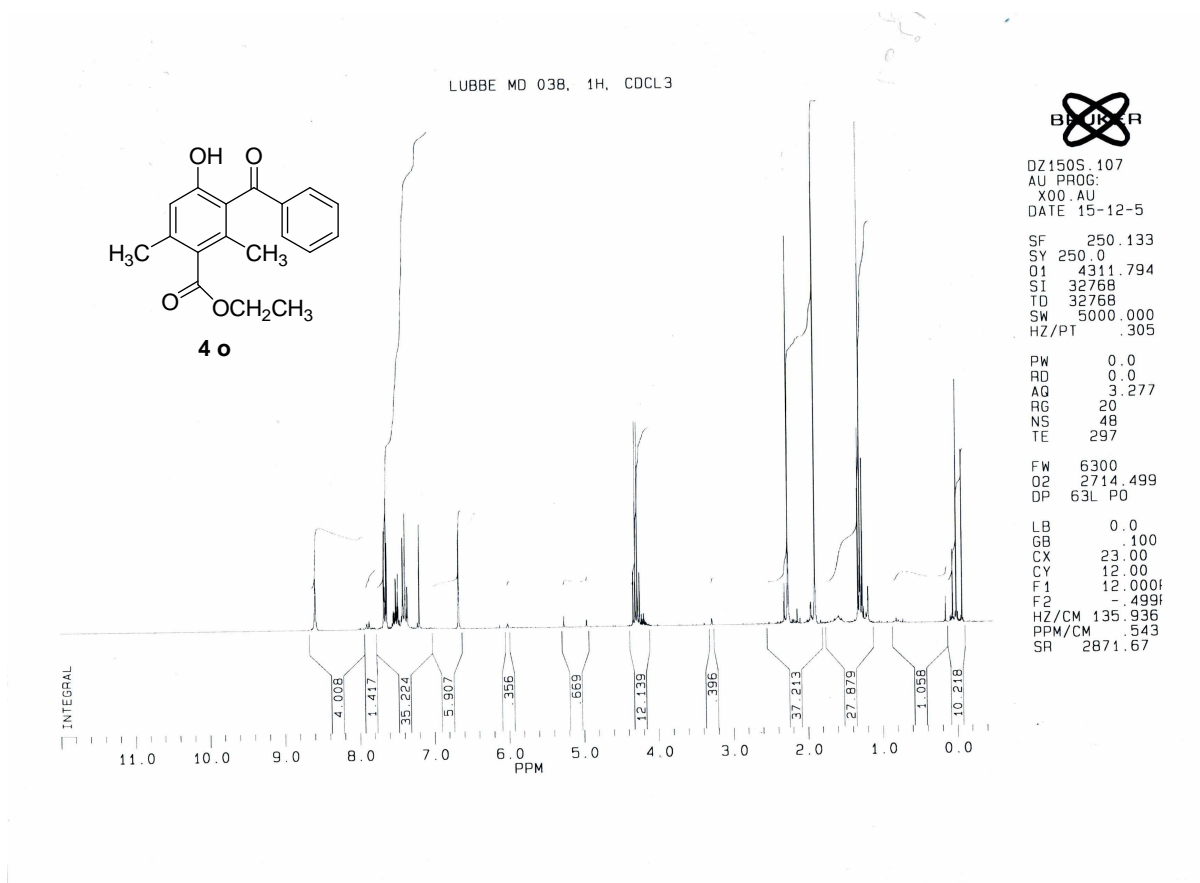
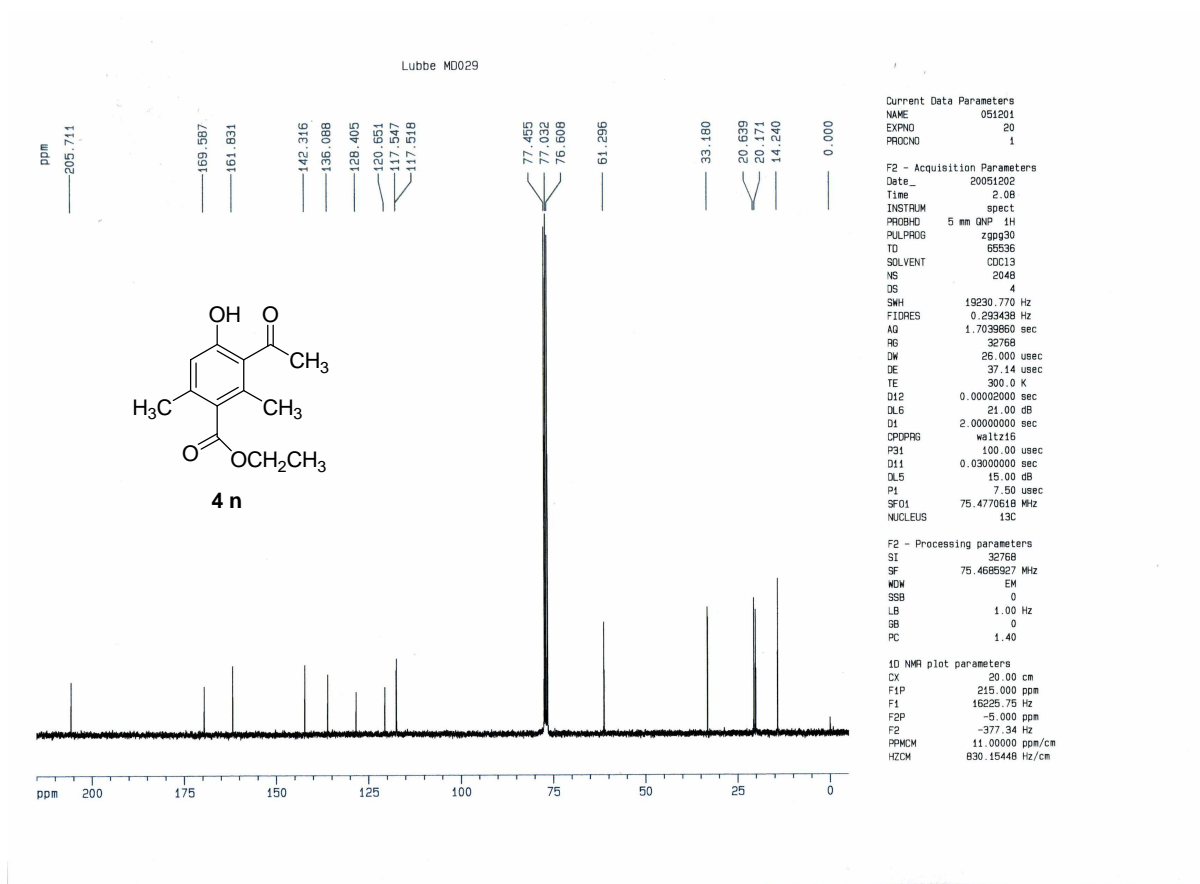
DZ010S.101
AU PROG:
X00.AU
DATE 1-12-5

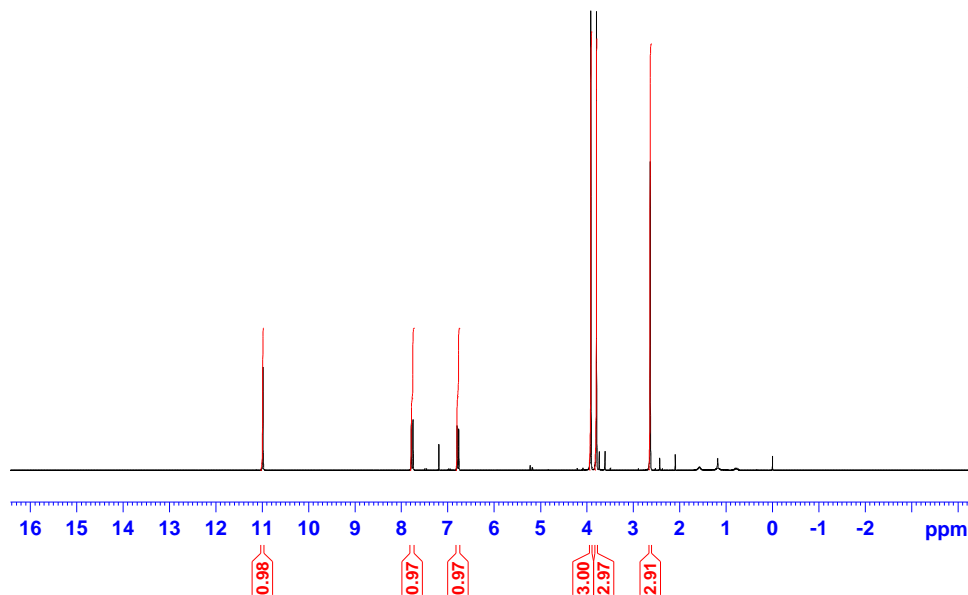
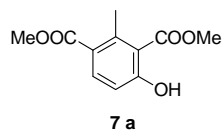
SF      250.133
SY      250.0
O1      4311.794
SI      32768
TD      32768
SW      5000.000
HZ/PT   .305

PW      0.0
RD      0.0
AQ      3.277
RG      40
NS      64
TE      297

FW      6300
O2      2714.499
DP      63L PO

LB      0.0
GB      .100
CX      23.00
CY      12.00
F1      12.000f
F2      - .499f
HZ/CM   135.936
PPM/CM  .543
SR      2853.05
    
```





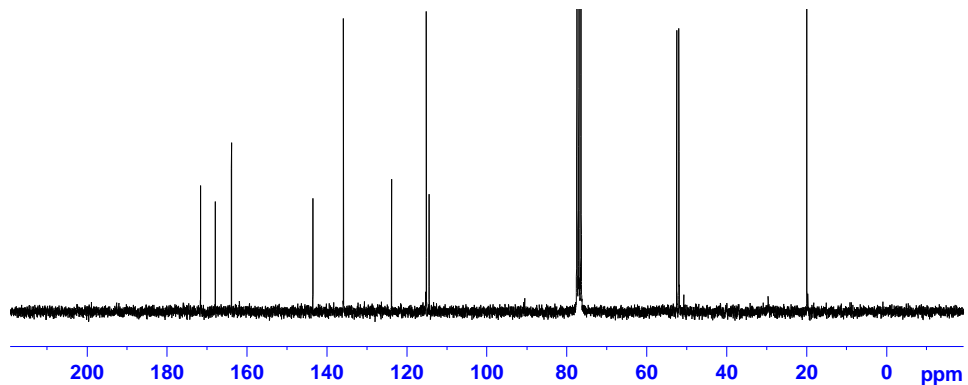
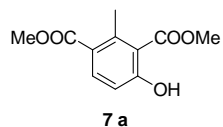
```

Current Data Parameters
NAME      080804.211
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080804
Time     8.49
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       5165.288 Hz
FIDRES   0.078816 Hz
AQ        6.343950 sec
RG        645
RW        96.800 usec
DE        10.00 usec
TE        298.3 K
D1        1.0000000 sec
TDO       1

***** CHANNEL f1 *****
NUC1      13C
P1        11.40 usec
PL1       -3.00 dB
SFO1      250.1315447 MHz

F2 - Processing parameters
SI        32768
SF        250.1300174 MHz
MORF     EM
SGB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

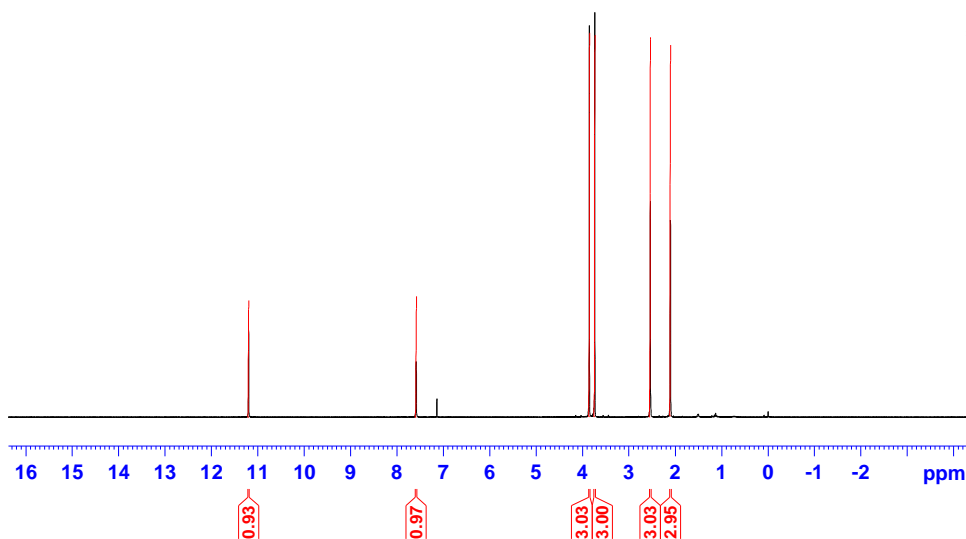
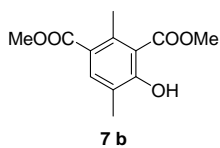
Current Data Parameters
NAME      080804.236
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080805
Time     3.08
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        4
SWH       15000.000 Hz
FIDRES   0.228882 Hz
AQ        2.184834 sec
RG        2050
RW        33.333 usec
DE        10.00 usec
TE        298.3 K
D1        2.0000000 sec
d11      0.8800000 sec
DELTA    1.8999999 sec
TDO       1

***** CHANNEL f1 *****
NUC1      13C
P1        10.20 usec
PL1       0.00 dB
SFO1      62.9015280 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -3.00 dB
SFO2     250.1310005 MHz

F2 - Processing parameters
SI        32768
SF        62.8932390 MHz
MORF     EM
SGB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

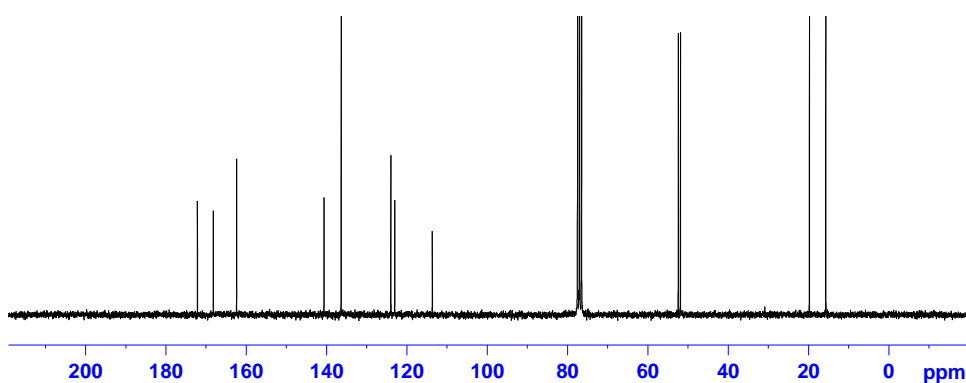
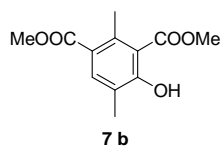


```
Current Data Parameters
NAME      080807.227
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080807
Time     13.39
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      5165.288 Hz
FIDRES   0.078816 Hz
AQ       6.343950 sec
RG       512
DW       96.800 usec
DE       10.00 usec
TE       298.8 K
D1       1.0000000 sec
TDO      1

***** CHANNEL f1 *****
NUC1     1H
P1       11.40 usec
PL1      -3.00 dB
SFO1     250.131447 MHz

F2 - Processing parameters
SI       32768
SF       250.130093 MHz
MDW      EM
SGB      0
LB       0.30 Hz
GB       0
PC       1.00
```



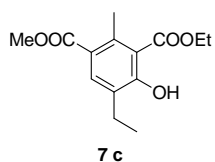
```
Current Data Parameters
NAME      080808.226
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080809
Time     19.11
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1500
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1848834 sec
RG       2050
DW       33.333 usec
DE       10.00 usec
TE       298.3 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999999 sec
TDO      1

***** CHANNEL f1 *****
NUC1     13C
P1       10.00 usec
PL1      0.00 dB
SFO1     62.9015280 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -3.00 dB
SFO2     250.1310005 MHz

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



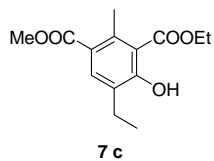
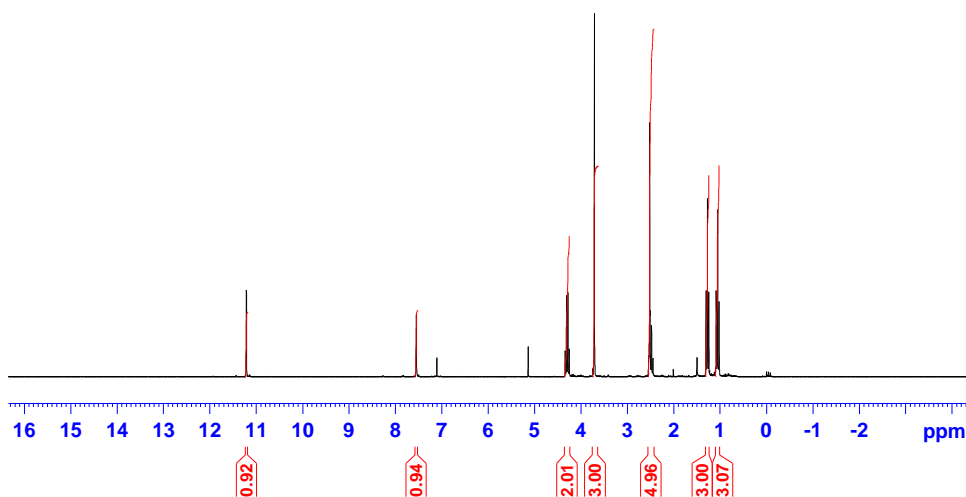
```

Current Data Parameters
NAME      080812.216
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20080812
Time     11.43
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      5165.288 Hz
FIDRES   0.078816 Hz
AQ       6.3439350 sec
RG       512
RW       96.800 usec
DE       10.00 usec
TE       296.8 K
D1       1.0000000 sec
TDO      1

***** CHANNEL f1 *****
NUC1     1H
P1       11.40 usec
PL1     -3.00 dB
SFO1    250.131447 MHz

F2 - Processing parameters
SI       32768
SF       250.130038 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

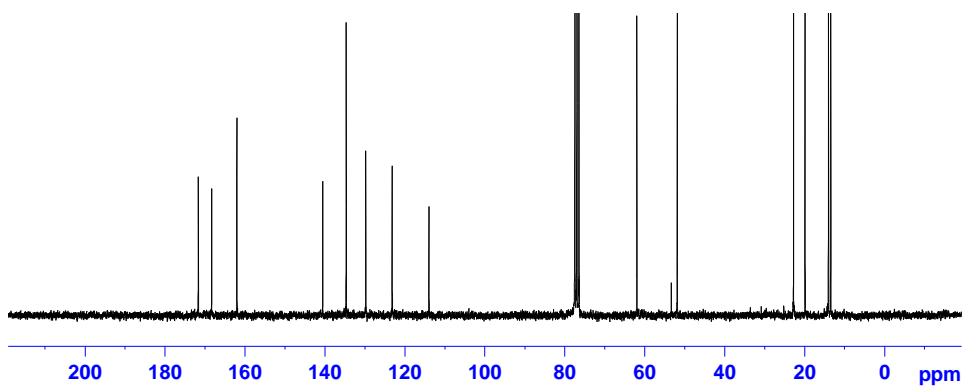
Current Data Parameters
NAME      080815.223
EXPNO    10
PROCNO   1

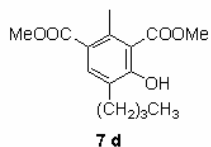
F2 - Acquisition Parameters
Date_    20080816
Time     10.21
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       2048
DS       4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ       2.1848834 sec
RG       2050
RW       33.333 usec
DE       10.00 usec
TE       297.1 K
D1       2.0000000 sec
d11      0.8300000 sec
DELTA    1.8999998 sec
TDO      1

***** CHANNEL f1 *****
NUC1     13C
P1       10.20 usec
PL1      0.00 dB
SFO1    62.9015280 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -3.00 dB
SFO2    250.1310005 MHz

F2 - Processing parameters
SI       32768
SF       62.8932380 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```





```

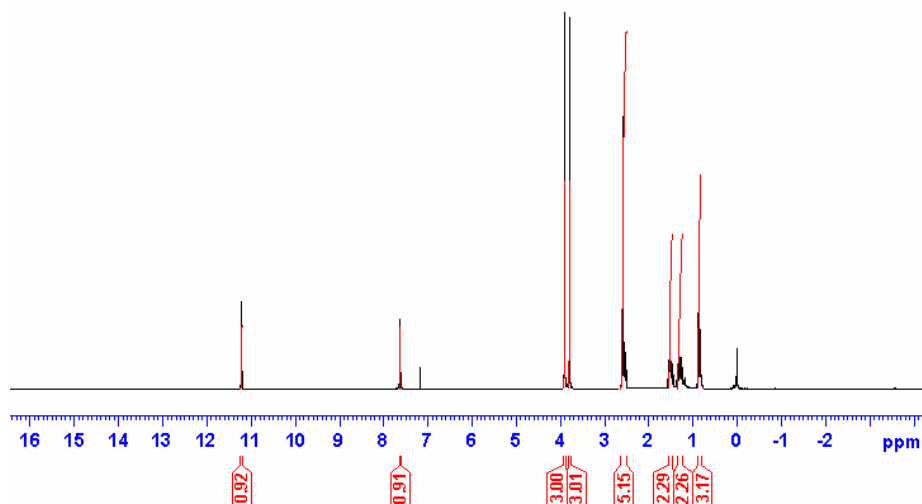
Current Data Parameters
NAME          080815.224
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080816
Time         14.40
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           3672
DS           4
SWH          15000.000 Hz
FIDRES       0.238882 Hz
AQ           2.1845834 sec
RG           2050
EW           33.333 usec
DE           10.00 usec
TE           297.7 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1            10.20 usec
PL1           0.00 dB
SFO1         62.9015280 MHz

===== CHANNEL F2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         14.00 dB
PL13         14.00 dB
PL2          -3.00 dB
SFO2         250.1310005 MHz

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



```

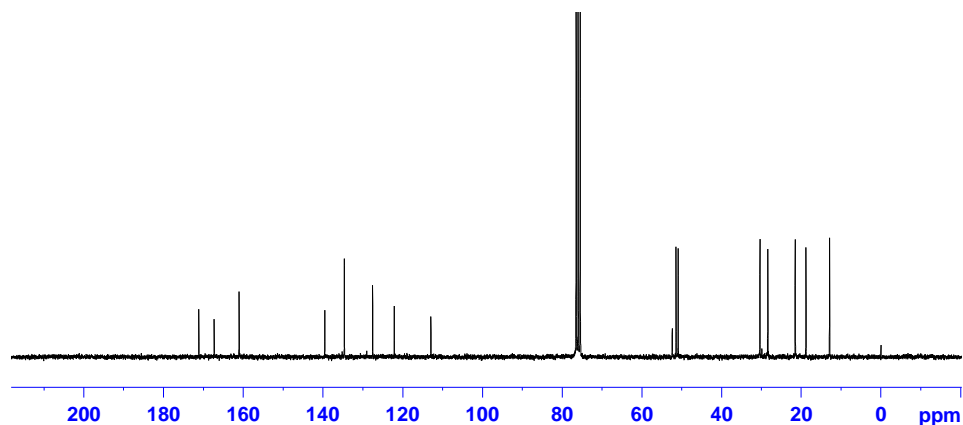
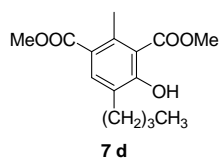
Current Data Parameters
NAME          080815.224
EXPNO        10
PROCNO       1

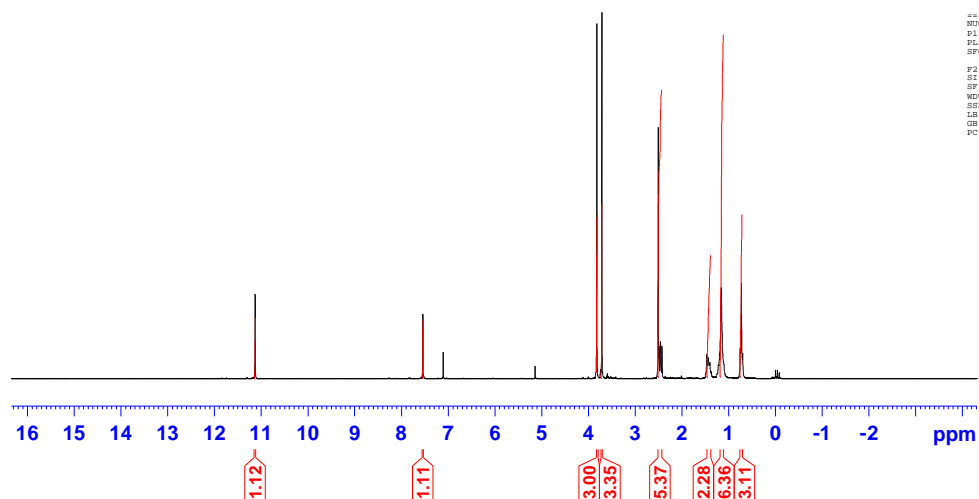
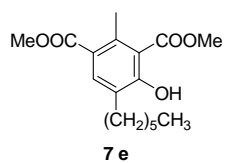
F2 - Acquisition Parameters
Date_        20080816
Time         14.40
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           3672
DS           4
SWH          15000.000 Hz
FIDRES       0.238882 Hz
AQ           2.1845834 sec
RG           2050
EW           33.333 usec
DE           10.00 usec
TE           297.7 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1            10.20 usec
PL1           0.00 dB
SFO1         62.9015280 MHz

===== CHANNEL F2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         14.00 dB
PL13         14.00 dB
PL2          -3.00 dB
SFO2         250.1310005 MHz

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





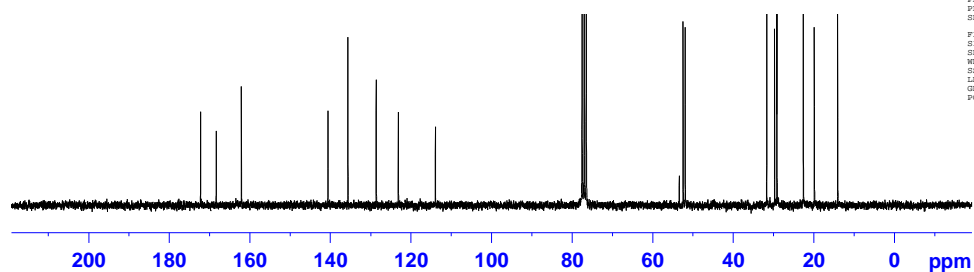
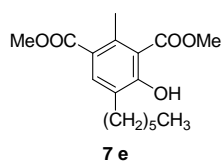
```

Current Data Parameters
NAME      080812.215
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20080812
Time     11.36
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       5165.289 Hz
FIDRES   0.078816 Hz
AQ        6.1483350 sec
RG        575
DW        96.800 usec
DE        10.00 usec
TE        296.9 K
D1        1.00000000 sec
DELTA    1
TDO       1

***** CHANNEL f1 *****
NUC1      1H
P1        11.40 usec
PL1       -3.50 dB
SFO1      250.1315447 MHz

F2 - Processing parameters
SI        32768
SF        250.130386 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

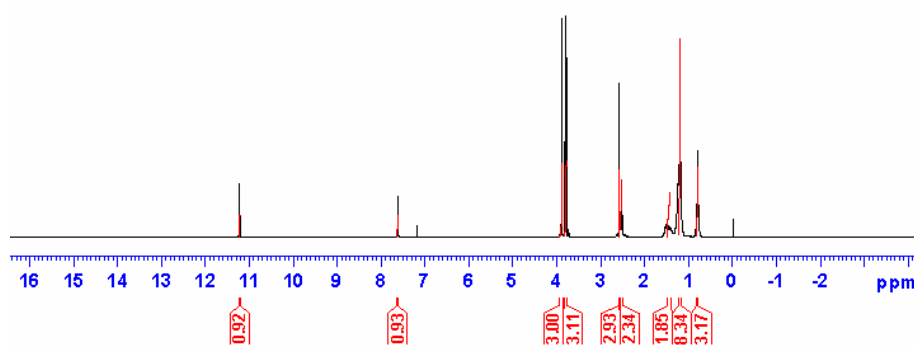
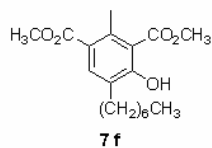
Current Data Parameters
NAME      080814.224
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080815
Time     5.17
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
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        297.3 K
D1        2.00000000 sec
d11      0.03000000 sec
DELTA    1.89999998 sec
TDO       1

***** CHANNEL f1 *****
NUC1      13C
P1        10.20 usec
PL1       0.00 dB
SFO1      62.9015280 MHz

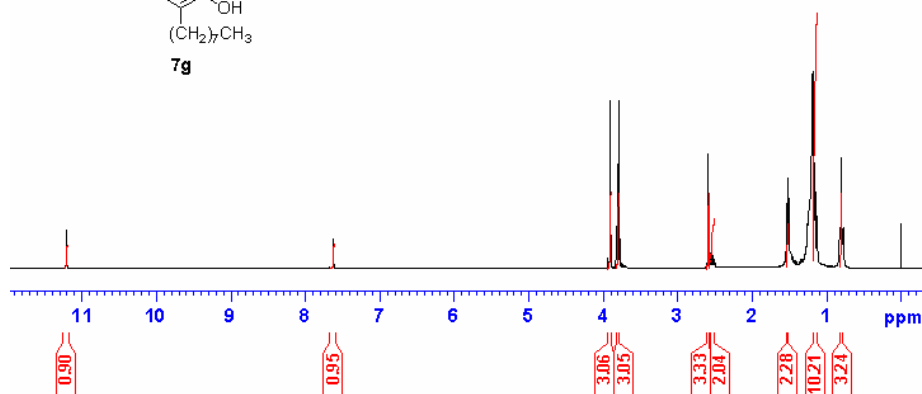
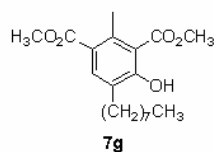
***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -3.00 dB
SFO2     250.1310005 MHz

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      001812_214
EXPNO    11
PROCNO   1
----- ACQUISITION PARAMETERS
Date_    20080812
Time     11.30
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        4
SWH       5161.248 Hz
F2 (MHz)  101.62513 MHz
AQ        6.1618218 sec
RG         324
SI         8
SF         250.1361910 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
----- CHANNEL f1 -----
NUC1      13C
P1         12.00 usec
PL1        0.00 dB
SFO1      101.62513 MHz
F2 - Processing parameters
SI         65536
SF         250.1361910 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
    
```



```

CURRENT DATA PARAMETERS
NAME      001812_231
EXPNO    11
PROCNO   1
----- ACQUISITION PARAMETERS
Date_    20080812
Time     11.49
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        4
SWH       5161.248 Hz
F2 (MHz)  101.62513 MHz
AQ        6.1618218 sec
RG         324
SI         8
SF         250.1361910 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
----- CHANNEL f1 -----
NUC1      13C
P1         12.00 usec
PL1        0.00 dB
SFO1      101.62513 MHz
F2 - Processing parameters
SI         65536
SF         250.1361910 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
    
```



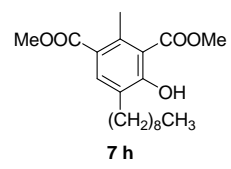
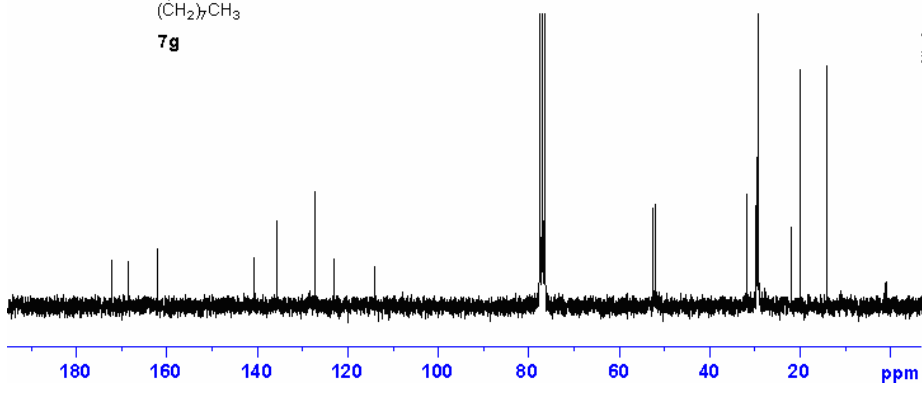
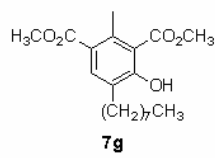

```

Current Data Parameters
NAME      080905.u311
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20080905
Time     10.06
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.118 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       45.2
DM       80.800 usec
DE       6.50 usec
TE       298.2 K
D1       1.00000000 sec
TDO      1

***** CHANNEL f1 *****
NUC1     1H
P1       10.00 usec
PL1     0.00 dB
PL12    11.25325108 W
SF01    300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300250 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



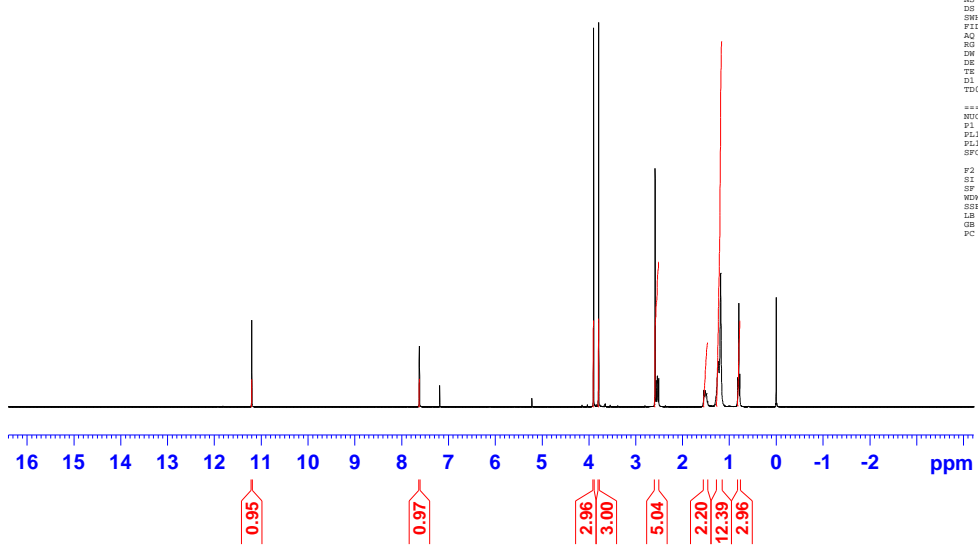
```

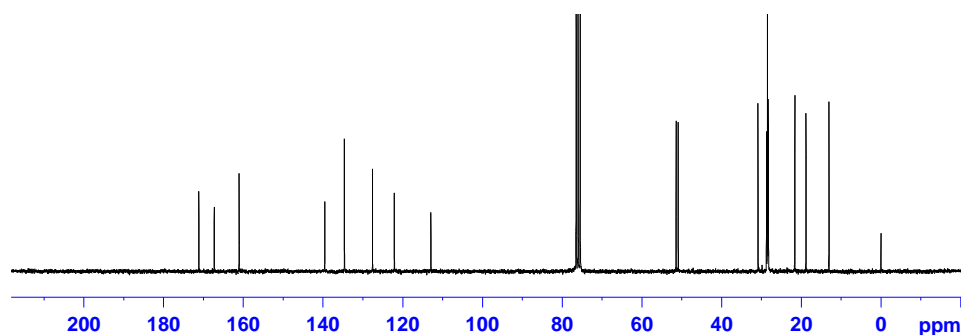
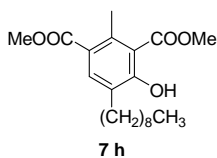
Current Data Parameters
NAME      080905.u311
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20080905
Time     10.06
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      6188.118 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       45.2
DM       80.800 usec
DE       6.50 usec
TE       298.2 K
D1       1.00000000 sec
TDO      1

***** CHANNEL f1 *****
NUC1     1H
P1       10.00 usec
PL1     0.00 dB
PL12    11.25325108 W
SF01    300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300250 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```





```

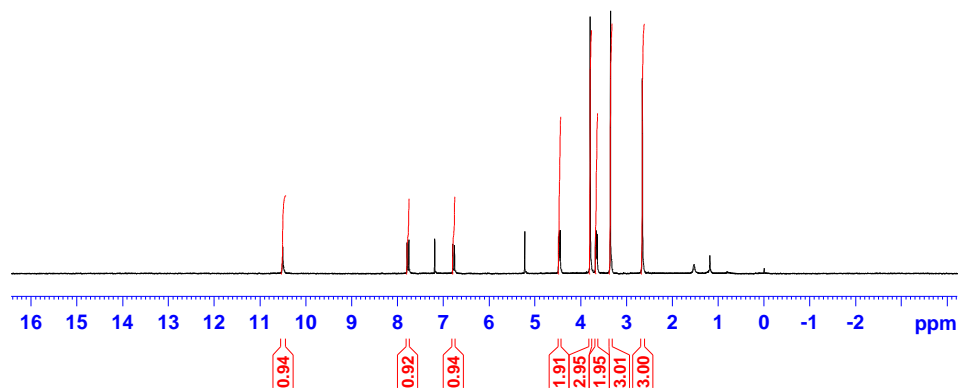
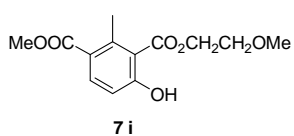
Current Data Parameters
NAME      080905.210
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080906
Time     14.47
INSTRUM spect
PROBHD   5 mm QNP 1H/13
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       298.2 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999998 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.20 usec
PL1      0.00 dB
SFO1     62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2     1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -1.00 dB
SFO2     250.1310005 MHz

F2 - Processing parameters
SI       32768
SF       62.8953014 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```



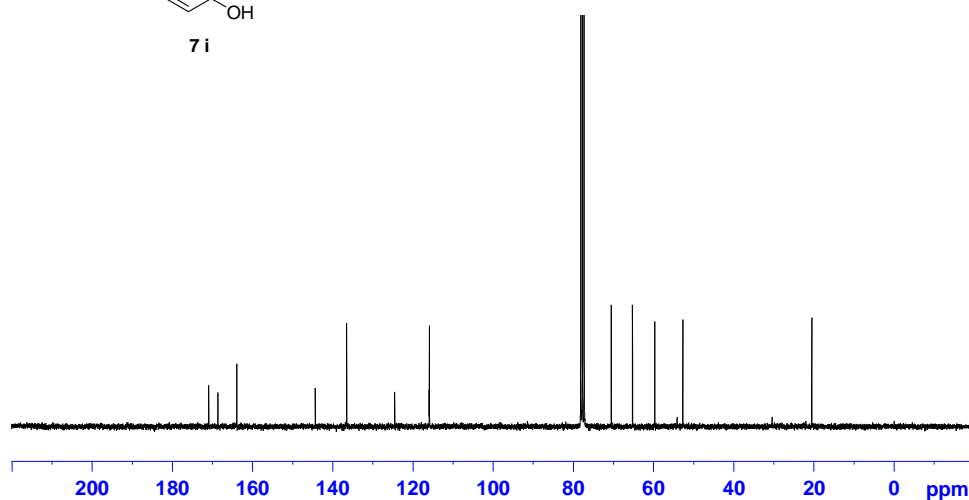
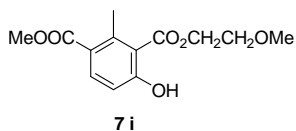
```

Current Data Parameters
NAME      080909.228
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080909
Time     14.32
INSTRUM spect
PROBHD   5 mm QNP 1H/13
PULPROG zgpg30
TD       65536
SOLVENT  cdcl3
NS       16
DS       2
SWH      5165.288 Hz
FIDRES   0.078816 Hz
AQ       6.3439350 sec
RG       724
DW       98.800 usec
DE       10.00 usec
TE       298.2 K
D1       1.0000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       11.40 usec
PL1      -3.00 dB
SFO1     250.1315447 MHz

F2 - Processing parameters
SI       32768
SF       250.1300177 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

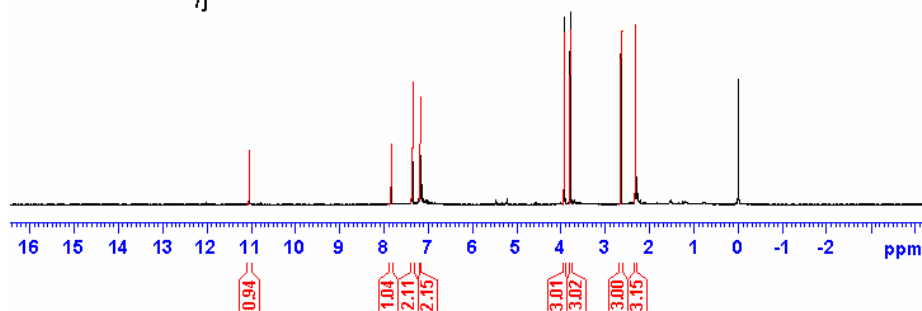
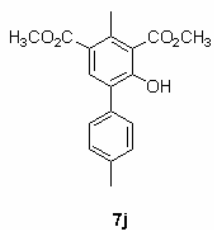
Current Data Parameters
NAME          080910.u301
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_         20080910
Time          9.35
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zgpg30
TD            65536
SOLVENT      CDCl3
NS            1389
DS            4
SWH           18028.846 Hz
FIDRES       0.275098 Hz
AQ            1.8176818 sec
RG            1150
RW            27.733 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

***** CHANNEL f1 *****
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W         33.2569396 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
PL1W         0.22453187 W
PL13W        0.22453187 W
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4676931 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



```

Current Data Parameters
NAME          080924.u316
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_         20080924
Time          13.31
INSTRUM      spect
PROBHD       5 mm QNP 1H/1
PULPROG      zgpg30
TD            65536
SOLVENT      CDCl3
NS            1389
DS            4
SWH           5385.189 Hz
FIDRES       0.118816 Hz
AQ            6.5429258 sec
RG            915
RW            27.733 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

***** CHANNEL f1 *****
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W         33.2569396 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
PL1W         0.22453187 W
PL13W        0.22453187 W
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4676931 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



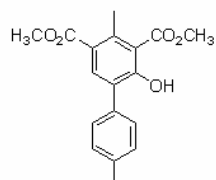
```

Current Data Parameters
NAME      080924.219
EXPNO     1
PROCNO    1

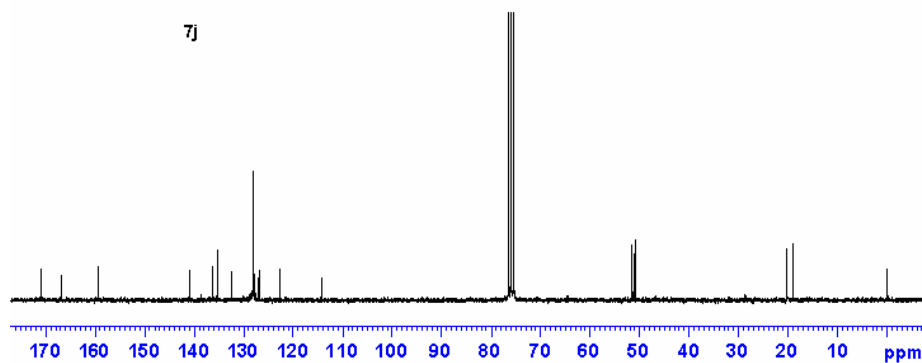
F2 - Acquisition Parameters
Date_     20080924
Time      18.11
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        5165.289 Hz
FIDRES    0.078816 Hz
AQ         6.3439950 sec
RG         406
DK         96.800 usec
DE         10.00 usec
TE         295.6 K
D1         1.0000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         11.40 usec
PL1        -3.00 dB
SFO1       250.131447 MHz

F2 - Processing parameters
SI         32768
SF         250.1300213 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



7j



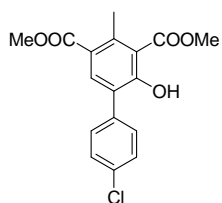
```

Current Data Parameters
NAME      080924.212
EXPNO     1
PROCNO    1

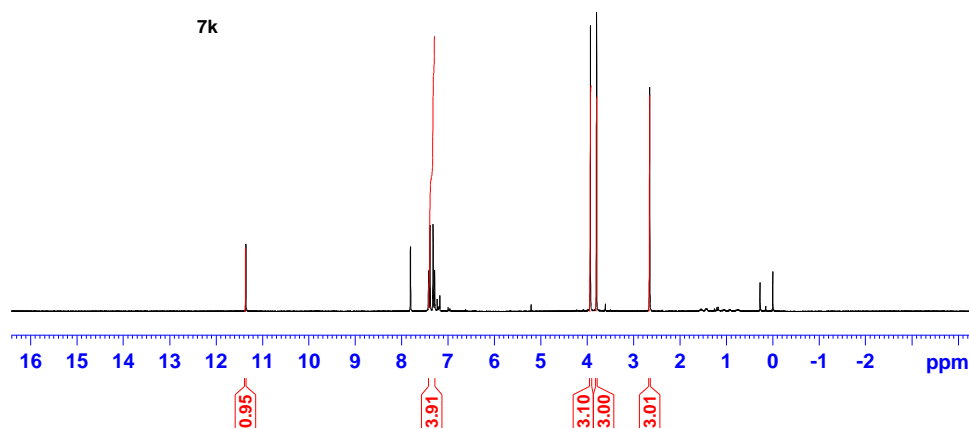
F2 - Acquisition Parameters
Date_     20080924
Time      10.50
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        5165.289 Hz
FIDRES    0.078816 Hz
AQ         6.3439950 sec
RG         406
DK         96.800 usec
DE         10.00 usec
TE         295.6 K
D1         1.0000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       1H
P1         11.40 usec
PL1        -3.00 dB
SFO1       250.131447 MHz

F2 - Processing parameters
SI         32768
SF         250.1300213 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



7k





```

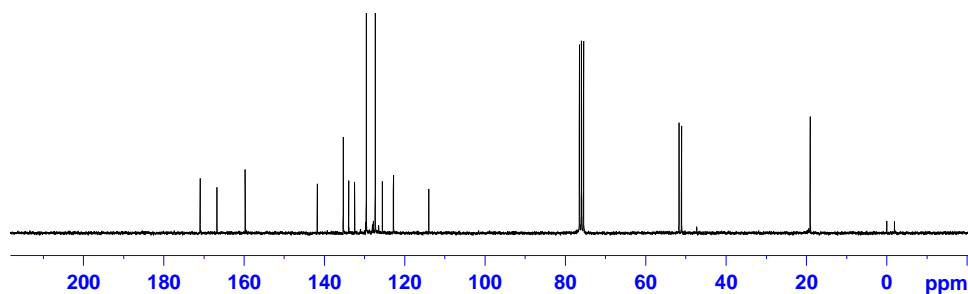
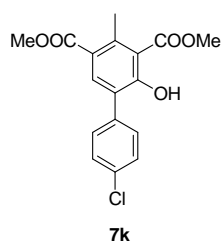
Current Data Parameters
NAME      080924.218
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20080924
Time     16.39
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1500
DS        4
SWH      15000.000 Hz
FIDRES   0.228882 Hz
AQ        2.1848534 sec
RG        2050
RW        33.333 usec
DE        10.00 usec
TE        296.7 K
D1        2.0000000 sec
d11       0.0300000 sec
DELTA    1.8999999 sec
TD0       1

***** CHANNEL f1 *****
NUC1      13C
P1        10.20 usec
PL1       0.00 dB
SFO1      62.9015280 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPD2    70.00 usec
PL12     14.00 dB
PL13     14.00 dB
PL2      -3.00 dB
SFO2     250.1310005 MHz

F2 - Processing parameters
SI        32768
SF        62.8953032 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



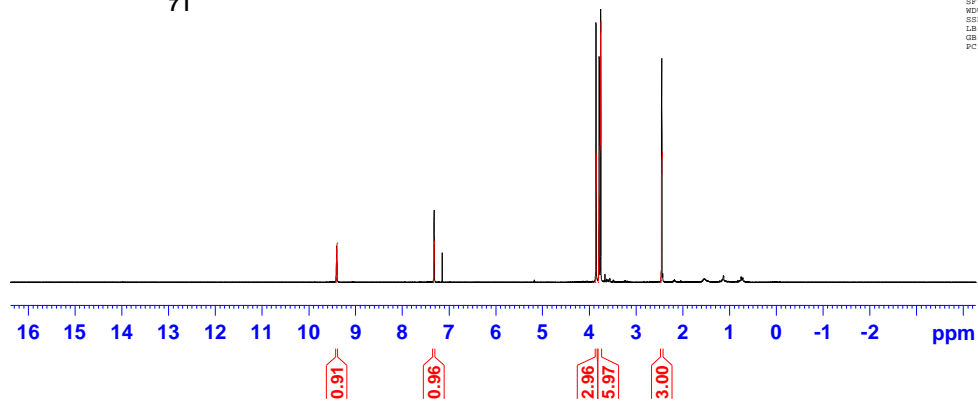
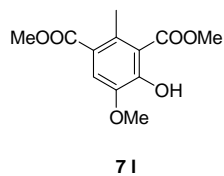
```

Current Data Parameters
NAME      080926.208
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080926
Time     9.52
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH      5165.288 Hz
FIDRES   0.078816 Hz
AQ        6.3439350 sec
RG        512
RW        96.800 usec
DE        10.00 usec
TE        296.5 K
D1        1.0000000 sec
TD0       1

***** CHANNEL f1 *****
NUC1      1H
P1        11.40 usec
PL1       -3.00 dB
SFO1     250.1314447 MHz

F2 - Processing parameters
SI        32768
SF        250.1302287 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

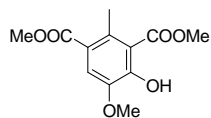




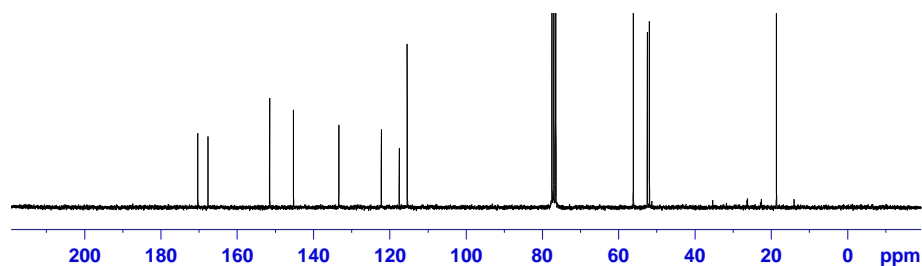
```

Current Data Parameters
NAME          080926.216
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080927
Time          11.49
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SFR           15000.000 Hz
FIDRES        0.228882 Hz
AQ            2.1848354 sec
RG            2050
DM            33.333 usec
DE            10.00 usec
TE            297.3 K
D1            2.0000000 sec
d11           0.1000000 sec
DELTA         1.8999999 sec
TDO           1
    
```



7 l



```

***** CHANNEL f1 *****
NUC1          13C
P1            10.20 usec
PL1           0.00 dB
SFO1          62.9015280 MHz

***** CHANNEL f2 *****
CROSS2       wait16
NUC2          1H
P2           70.00 usec
PL2           14.00 dB
PL12          14.00 dB
PL3           -3.00 dB
SFO2          250.1310005 MHz

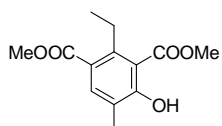
F2 - Processing parameters:
SI            32768
SF            62.8952390 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



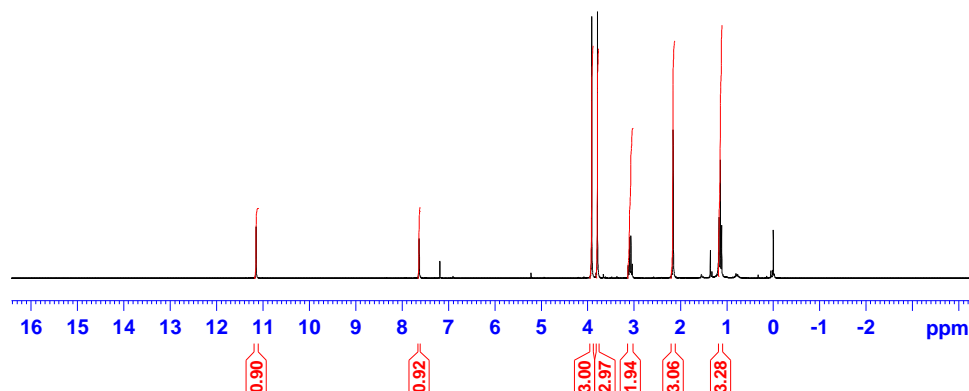
```

Current Data Parameters
NAME          080821.213
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080821
Time          9.38
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SFR           5165.289 Hz
FIDRES        0.078816 Hz
AQ            6.1433350 sec
RG            456
DM            96.000 usec
DE            10.00 usec
TE            296.7 K
D1            1.0000000 sec
TDO           1
    
```



7 m



```

***** CHANNEL f1 *****
NUC1          1H
P1            11.40 usec
PL1           -3.00 dB
SFO1          250.1315447 MHz

F2 - Processing parameters:
SI            32768
SF            250.1300184 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

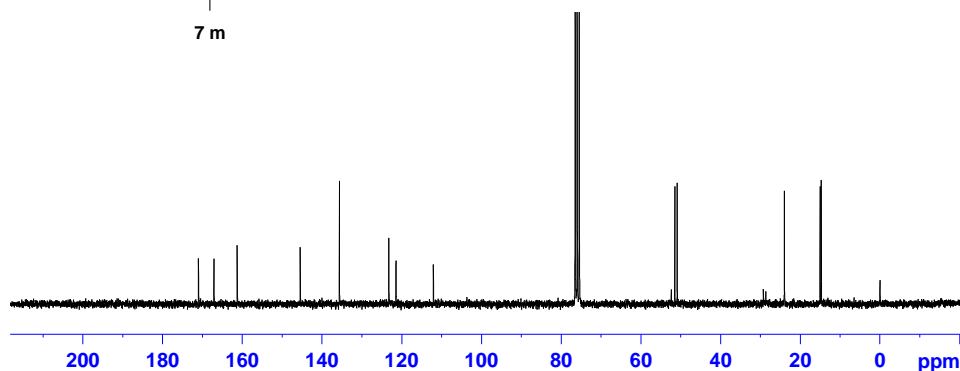
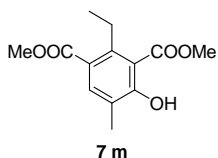
Current Data Parameters
NAME          080821.235
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080822
Time         0.37
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           1024
DS           4
SWH          15000.000 Hz
FIDRES       0.228882 Hz
AQ           2.1845834 sec
RG           2050
DM           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          0.00 dB
SFO1         62.9015260 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        70.00 usec
PL12         14.00 dB
PL13         14.00 dB
PL2          -3.00 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI           32768
SF           62.8953016 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```



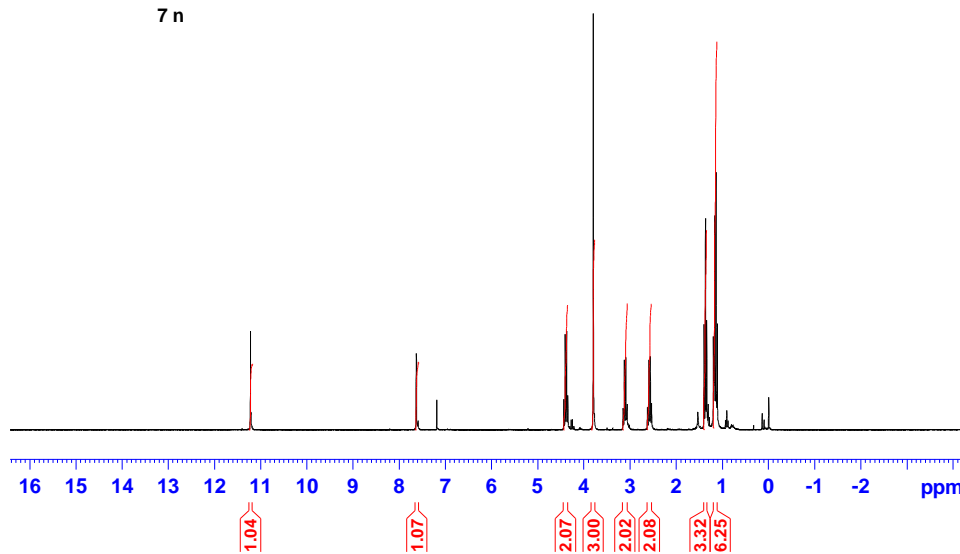
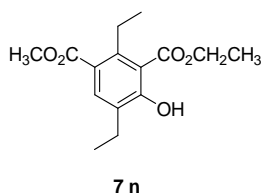
```

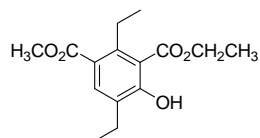
Current Data Parameters
NAME          080903.207
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080903
Time         10.09
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          5165.289 Hz
FIDRES       0.078816 Hz
AQ           6.3439350 sec
RG           812
DM           96.800 usec
DE           10.00 usec
TE           298.2 K
D1           1.0000000 sec
TD0          1

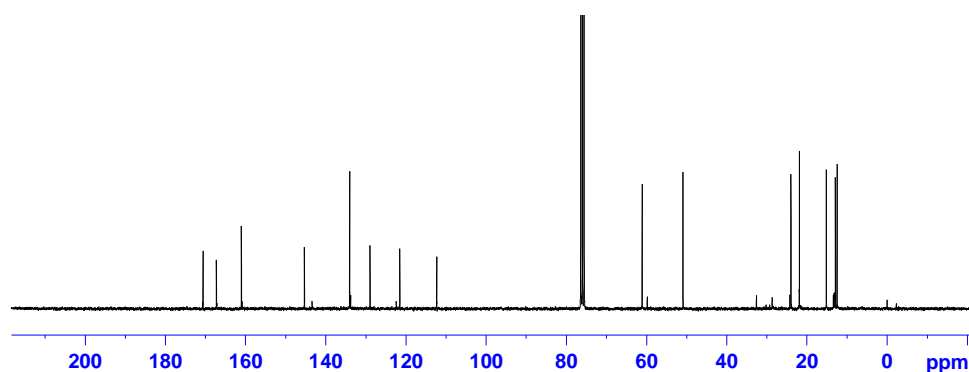
===== CHANNEL f1 =====
NUC1         1H
P1           11.40 usec
PL1          -3.00 dB
SFO1         250.1315447 MHz

F2 - Processing parameters
SI           32768
SF           250.1300186 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```





7 n



```

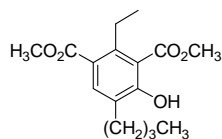
Current Data Parameters
NAME          080903.u311
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_         20080904
Time          3.32
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zgpg30
TD            65536
SOLVENT      CDCl3
NS            2048
DS            4
SWH           18028.846 Hz
FIDRES       0.275098 Hz
AQ            1.8176818 sec
RG            912
RW            27.733 usec
DE            6.50 usec
TE            298.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TDO           1

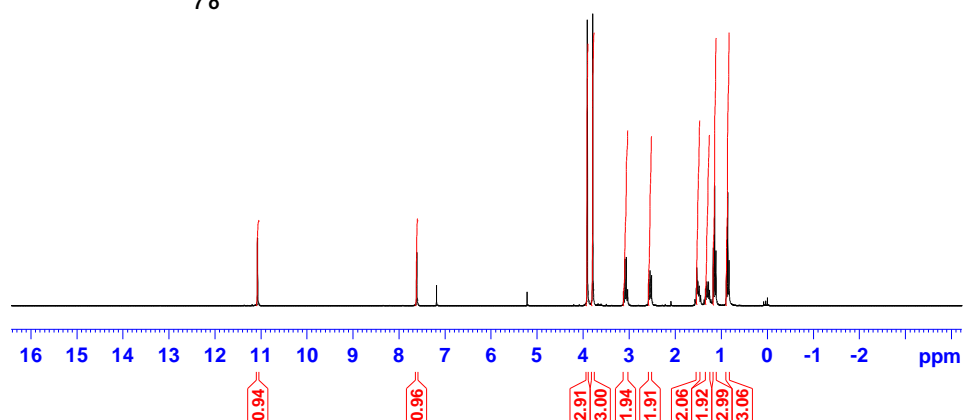
***** CHANNEL f1 *****
NUC1          13C
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.2569386 W
SFO1          75.4752953 MHz

***** 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
PL1W          0.22453187 W
PL13W         0.22453187 W
SFO2          300.1312005 MHz

F2 - Processing parameters
SI            32768
SF            75.4678249 MHz
WDW           EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
    
```



7 o



```

Current Data Parameters
NAME          080911.221
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_         20080911
Time          14.01
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD            65536
SOLVENT      CDCl3
NS            16
DS            4
SWH           5165.262 Hz
FIDRES       0.078816 Hz
AQ            6.3493950 sec
RG            406
RW            96.800 usec
DE            10.00 usec
TE            298.2 K
D1            1.0000000 sec
TDO           1

***** CHANNEL f1 *****
NUC1          1H
P1            11.40 usec
PL1            3.00 dB
SFO1          250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.1300184 MHz
WDW           EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```




```

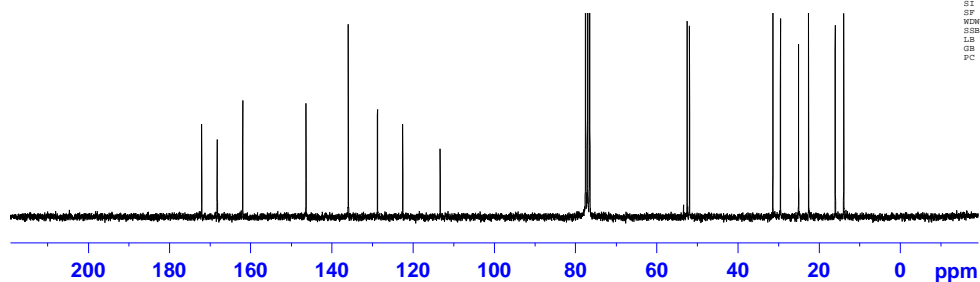
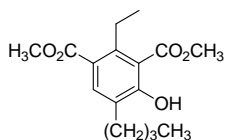
Current Data Parameters
NAME          080911.232
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080911
Time          23.37
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SMB           15000.000 Hz
FIDRES       0.228882 Hz
AQ           2.1845014 sec
RG            2050
DM            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            10.20 usec
PL1           0.00 dB
SFO1         62.901260 MHz

***** CHANNEL f2 *****
CPDPRG2       waltz16
NUC2          13C
PCPD2         70.00 usec
PL12          14.00 dB
PL13          14.00 dB
PL2           -3.00 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
    
```



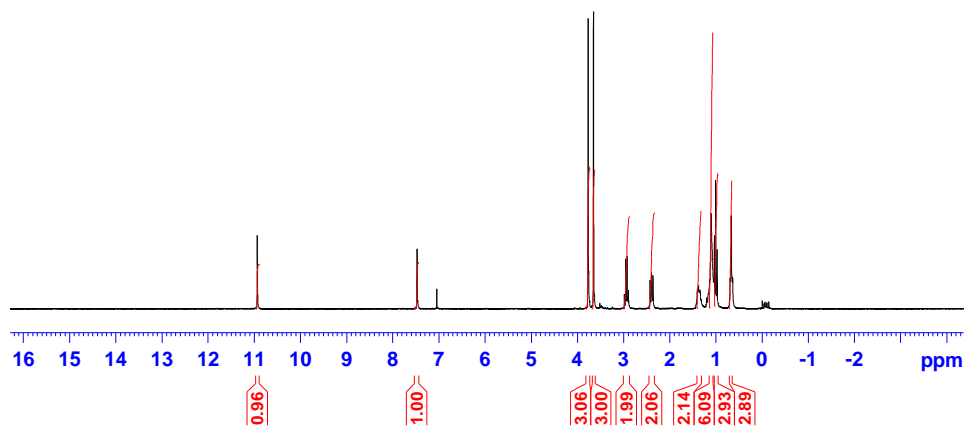
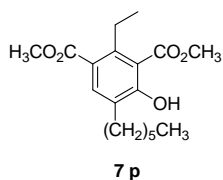
```

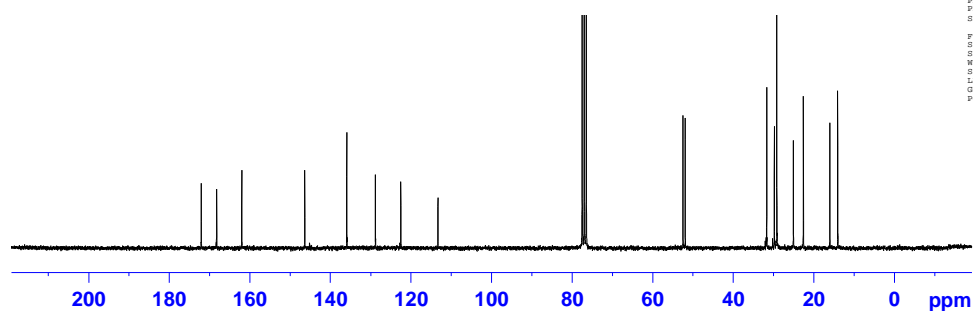
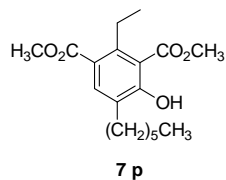
Current Data Parameters
NAME          080903.208MS 115
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080903
Time          10.19
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SMB           5165.289 Hz
FIDRES       0.078816 Hz
AQ           6.3433350 sec
RG            406
DM            96.800 usec
DE            10.00 usec
TE            298.1 K
D1            1.0000000 sec
TD0           1

***** CHANNEL f1 *****
NUC1          1H
P1            11.40 usec
PL1           -3.00 dB
SFO1         250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.1300532 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```





```

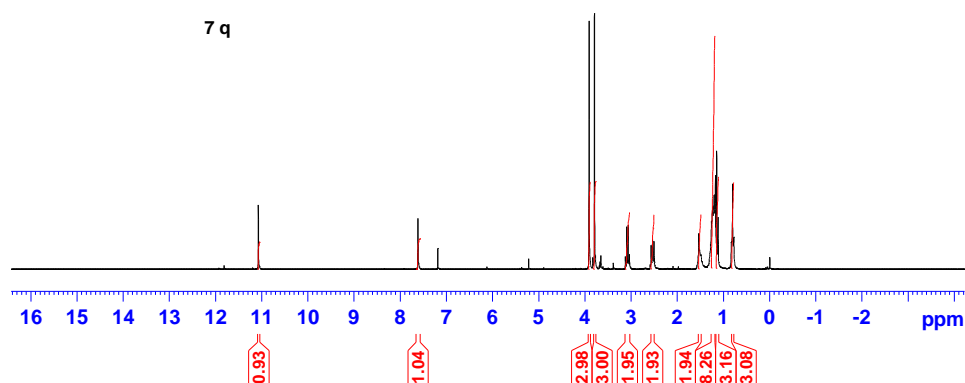
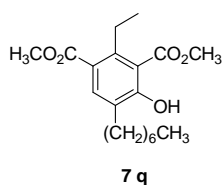
Current Data Parameters
NAME          080903.226
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080903
Time         20:57
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SFO1         15000.000 Hz
FIDRES       0.228882 Hz
AQ           2.1846834 sec
RG           2050
DE           33.933 usec
TE           298.2 K
DE           10.00 usec
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TDO          1

***** CHANNEL f1 *****
NUC1          13C
P1            10.20 usec
PL1           0.00 dB
SFO1         62.9015280 MHz

***** CHANNEL f2 *****
CPDPRG2      waltz16
NUC2          1H
P2            70.00 usec
PL2           14.00 dB
PL12          14.00 dB
PL13          14.00 dB
PL3           -3.00 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
    
```



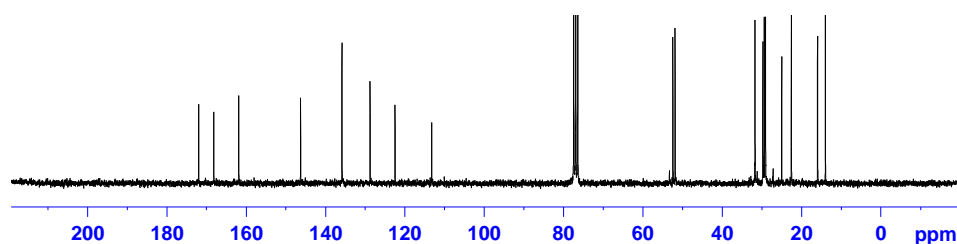
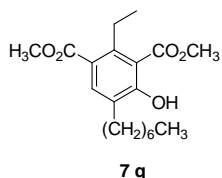
```

Current Data Parameters
NAME          080911.218
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080911
Time         13:42
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SFO1         5165.289 Hz
FIDRES       0.076816 Hz
AQ           6.3439350 sec
RG           652
DE           96.800 usec
TE           298.2 K
DE           10.00 usec
D1           1.0000000 sec
TDO          1

***** CHANNEL f1 *****
NUC1          1H
P1            11.40 usec
PL1           -3.00 dB
SFO1         250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.1300184 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.00
    
```



```

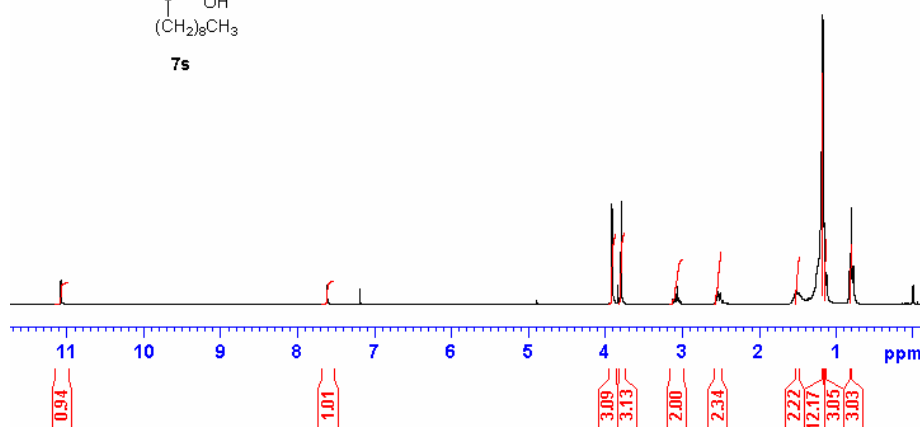
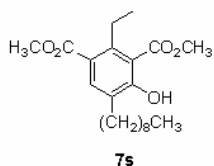
Current Data Parameters
NAME      080911.234
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080912
Time     5.49
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       2048
DS       4
SWH      15000.000 Hz
FIDRES   0.228892 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       10.20 usec
PL1      0.00 dB
SFO1     62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2  wait16
NUC2      1H
P2       70.00 usec
PL2      14.00 dB
PL12     14.00 dB
PL13     14.00 dB
PL2     -3.00 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      080911.234
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20080912
Time     5.49
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       2048
DS       4
SWH      15000.000 Hz
FIDRES   0.228892 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       10.20 usec
PL1      0.00 dB
SFO1     62.9015280 MHz

===== CHANNEL f2 =====
CPDPRG2  wait16
NUC2      1H
P2       70.00 usec
PL2      14.00 dB
PL12     14.00 dB
PL13     14.00 dB
PL2     -3.00 dB
SFO2     250.1310005 MHz

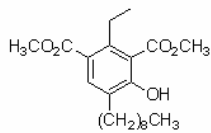
F2 - Processing parameters
SI       32768
SF       62.8952390 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```



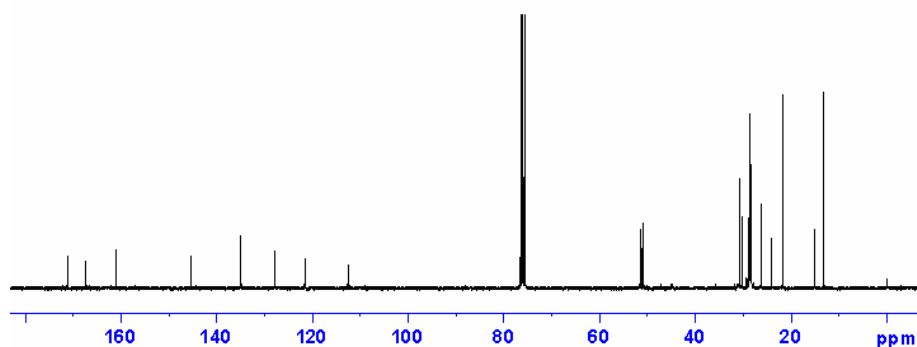
```

Current Data Parameters
NAME      818216-0518
EXPNO    3
PROCNO   3

F2 - Acquisition Parameters
Date_    20110916
Time     12:35
INSTRUM  spect
PROBHD   5 mm BBOBO MM-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1444
DS        4
SWH       38828.846 Hz
F2IDRES  0.215499 Hz
AQ        0.2015618 sec
RG         743
RW         0.11355 sec
AQ        0.2015618 sec
TE        298.2 K
DE        2.8888888 sec
D1        0.2015618 sec
D11       0.2015618 sec
TDR       3
    
```



7s



```

----- CHANNEL f1 -----
NUC1     13C
P1        30.00 usec
PL1       0.00 dB
PL12      1.00 dB
PL13      1.00 dB
PL14      1.00 dB
PL15      1.00 dB
PL16      1.00 dB
PL17      1.00 dB
PL18      1.00 dB
PL19      1.00 dB
PL20      1.00 dB
SFO1      101.625318 MHz

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2     13C
P2        12.00 usec
PL2       0.00 dB
PL22      1.00 dB
PL23      1.00 dB
PL24      1.00 dB
PL25      1.00 dB
PL26      1.00 dB
PL27      1.00 dB
PL28      1.00 dB
PL29      1.00 dB
PL30      1.00 dB
SFO2      50.787659 MHz

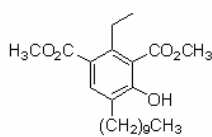
F2 - Processing parameters
SI        32768
SF        101.625318 MHz
WDW       EM
SSB       0
GB        0
CB        0
PC        1.00
    
```



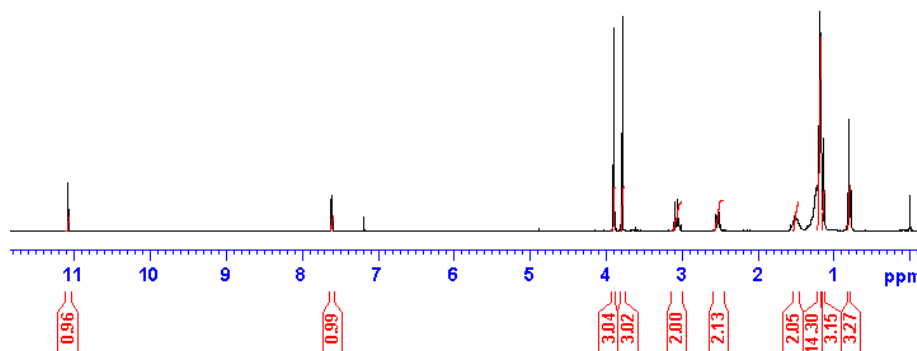
```

Current Data Parameters
NAME      818216-0518
EXPNO    3
PROCNO   3

F2 - Acquisition Parameters
Date_    20110916
Time     12:35
INSTRUM  spect
PROBHD   5 mm BBOBO MM-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1444
DS        4
SWH       38828.846 Hz
F2IDRES  0.215499 Hz
AQ        0.2015618 sec
RG         743
RW         0.11355 sec
AQ        0.2015618 sec
TE        298.2 K
DE        2.8888888 sec
D1        0.2015618 sec
D11       0.2015618 sec
TDR       3
    
```



7t



```

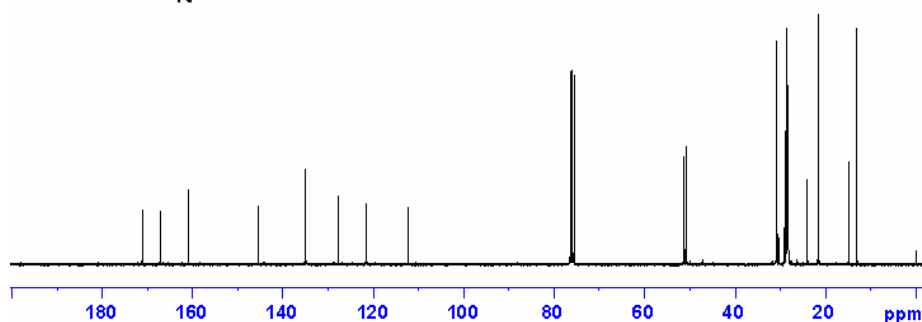
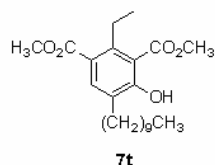
----- CHANNEL f1 -----
NUC1     1H
P1        30.00 usec
PL1       0.00 dB
PL12      1.00 dB
PL13      1.00 dB
PL14      1.00 dB
PL15      1.00 dB
PL16      1.00 dB
PL17      1.00 dB
PL18      1.00 dB
PL19      1.00 dB
PL20      1.00 dB
SFO1      500.136251 MHz

F2 - Processing parameters
SI        32768
SF        500.136251 MHz
WDW       EM
SSB       0
GB        0
CB        0
PC        1.00
    
```



```
Current Data Parameters
NAME: 000018_0011
EXPNO: 18
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20080920
Time: 0.55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 4
SWH: 28821.840 Hz
F2FREQ: 400.146399 MHz
AQ: 1.1215019 sec
RG: 3128
SQ: 17.1322 sec
DE: 0.38 umol
TE: 298.2 K
D1: 2.80000000 sec
d11: 0.20000000 sec
TDR: 1
```



```
===== CHANNEL f1 =====
NUC1: 13C
P1: 12.00 umol
PL1: -5.00 dB
PL24: 22.5000000 MHz
SFO1: 101.6261250 MHz

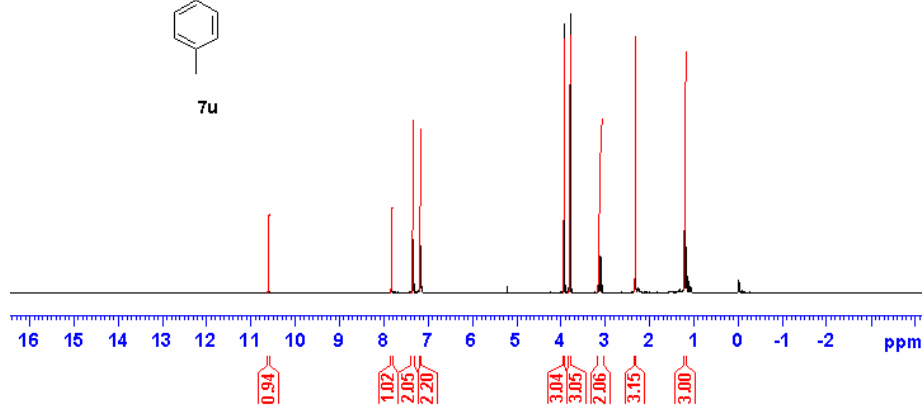
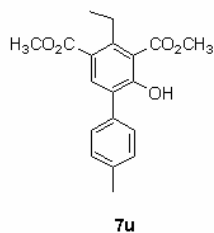
===== CHANNEL f2 =====
NAME: 1H
NUC2: 1H
P2: 12.00 umol
PL2: 0.00 dB
PL31: 11.00 dB
PL32: 11.00 dB
PL33: 11.00 dB
PL34: 11.0000000 MHz
PL35: 0.22452001 MHz
PL36: 0.22452001 MHz
SFO2: 500.1364912 MHz

F2 - Processing parameters
SI: 32768
SF: 101.6261250 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.00
```



```
Current Data Parameters
NAME: 000018_0012
EXPNO: 18
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20080920
Time: 21.55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 4
SWH: 5265.289 Hz
F2FREQ: 400.146399 MHz
AQ: 6.3429254 sec
RG: 455
SQ: 96.888 sec
DE: 0.38 umol
TE: 298.2 K
D1: 2.80000000 sec
d11: 0.20000000 sec
TDR: 1
```



```
===== CHANNEL f1 =====
NUC1: 1H
P1: 12.00 umol
PL1: -5.00 dB
PL24: 22.5000000 MHz
SFO1: 500.1364912 MHz

F2 - Processing parameters
SI: 32768
SF: 500.1364912 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.00
```



```

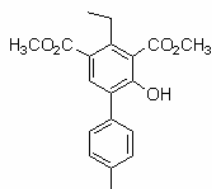
Current Data Parameters:
NAME: 000000000
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters:
Date_: 20080904
Time: 1.42
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2048
DS: 4
SWH: 12881.880 Hz
F2DRES: 0.248880 Hz
AQ: 1.2140254 sec
RG: 3158
SI: 327.332 mmHg
DE: 18.88 mmHg
TE: 296.2 K
D1: 2.80000000 sec
d11: 0.00000000 sec
DELTA: 1.49999999 sec
TDR: 1

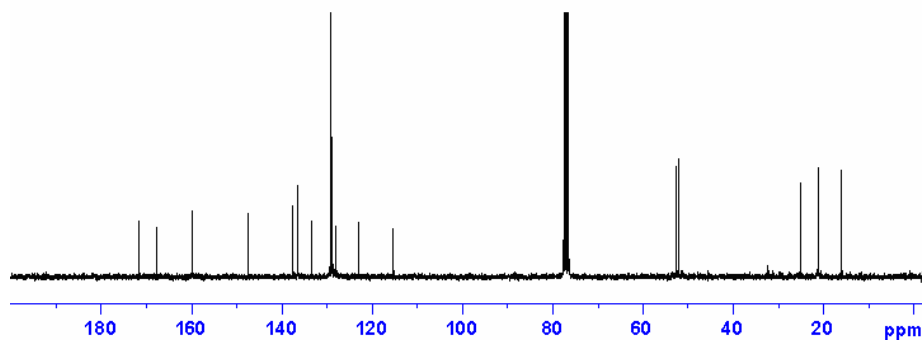
===== CHANNEL f1 =====
NUC1: 13C
P1: 18.00 mmHg
PL1: 0.00 dB
SFO1: 101.625375 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 18.88 mmHg
PL12: 14.00 dB
PL13: 14.00 dB
PL2: -5.00 dB
SFO2: 500.136099 MHz

F2 - Processing parameters:
SI: 32736
SF: 101.625375 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.48
    
```



7u



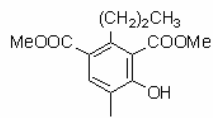
```

Current Data Parameters:
NAME: 000000000
EXPNO: 1
PROCNO: 1

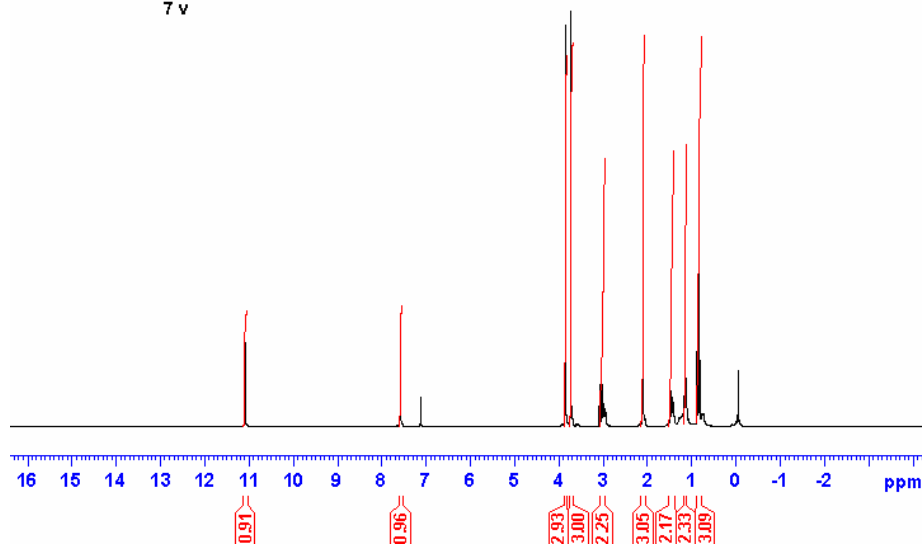
F2 - Acquisition Parameters:
Date_: 20080903
Time: 9.44
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2048
DS: 4
SWH: 5265.249 Hz
F2DRES: 0.418880 Hz
AQ: 0.2440254 sec
RG: 3158
SI: 327.332 mmHg
DE: 18.88 mmHg
TE: 296.2 K
D1: 1.80000000 sec
TDR: 1

===== CHANNEL f1 =====
NUC1: 1H
P1: 18.00 mmHg
PL1: -5.00 dB
SFO1: 500.136099 MHz

F2 - Processing parameters:
SI: 32736
SF: 500.136099 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.48
    
```



7v





```

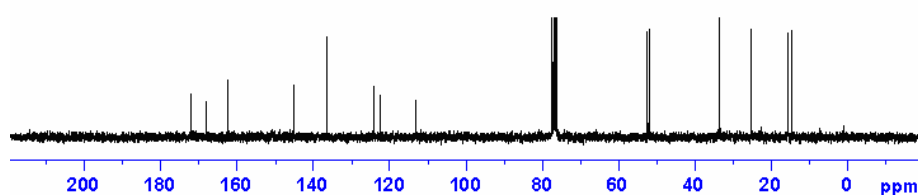
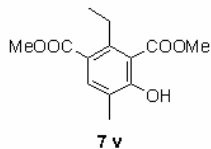
Current Data Parameters
NAME          080903.206
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080903
Time          2.13
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           5165.289 Hz
FIDRES        0.078816 Hz
AQ            6.3439350 sec
RG            322
DM            96.800 usec
DE            10.00 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            18.00 usec
PL1           0.00 dB
SFO1          101.625381 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         78.00 usec
PL12          14.00 dB
PL13          14.00 dB
PL14          -3.00 dB
SFO2          250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.130528 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



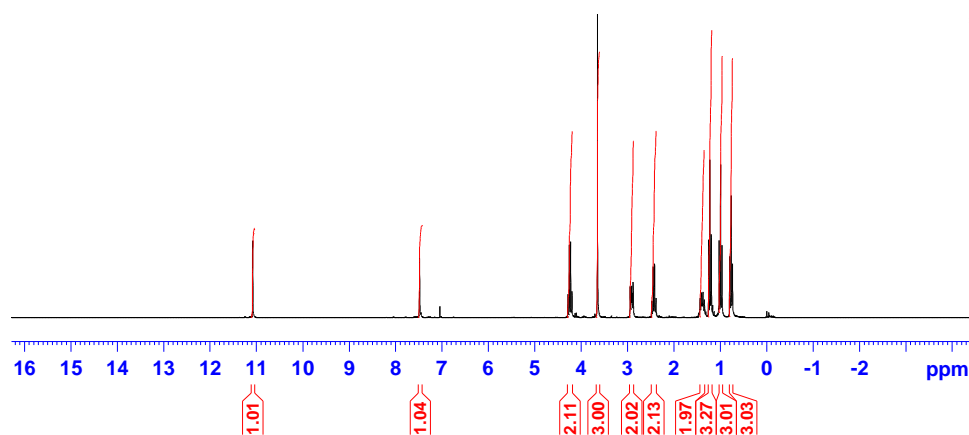
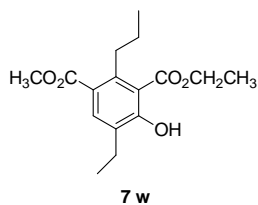
```

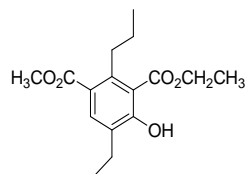
Current Data Parameters
NAME          080903.206
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20080903
Time          10.01
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           5165.289 Hz
FIDRES        0.078816 Hz
AQ            6.3439350 sec
RG            322
DM            96.800 usec
DE            10.00 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1

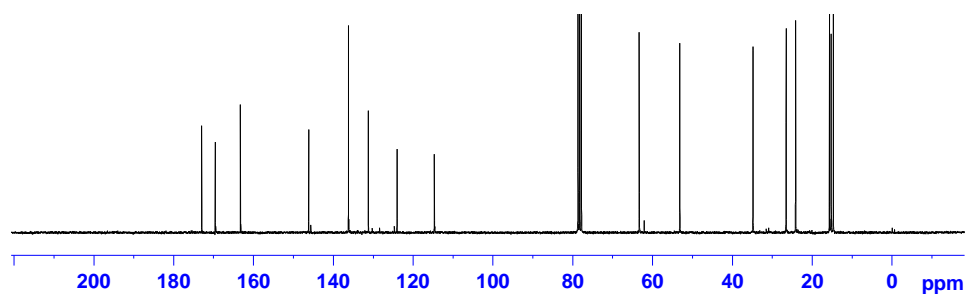
===== CHANNEL f1 =====
NUC1          1H
P1            11.40 usec
PL1           -3.00 dB
SFO1          250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.130528 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```





7 w



```

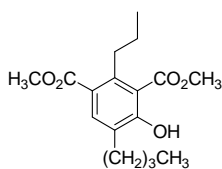
Current Data Parameters
NAME          080903.u312
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080904
Time         6.24
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          18028.846 Hz
FIDRES       0.275998 Hz
AQ           1.8178818 sec
RG           1030
RW           27.733 usec
DE           6.50 usec
TE           298.2 K
D1           2.0000000 sec
D11          0.0300000 sec
TDO          1

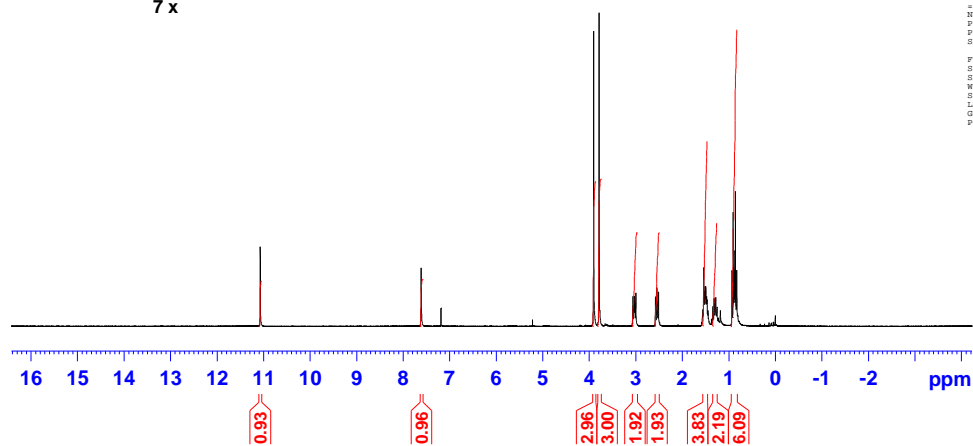
***** CHANNEL f1 *****
NUC1          13c
P1            10.00 usec
PL1           -0.50 dB
PL1W          33.2569186 W
SFO1          75.4752953 MHz

***** 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            75.4676524 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
CB            0
PC            1.40
    
```



7 x



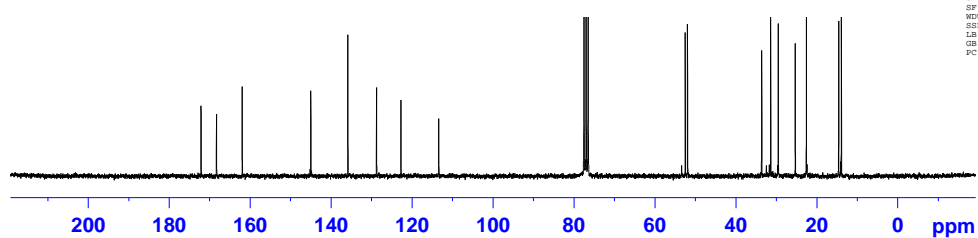
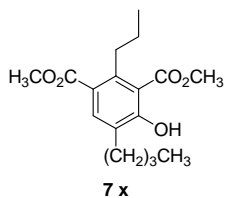
```

Current Data Parameters
NAME          080911.217
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080911
Time         13.35
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          5165.289 Hz
FIDRES       0.078816 Hz
AQ           6.3439250 sec
RG           362
RW           96.800 usec
DE           10.00 usec
TE           298.2 K
D1           1.0000000 sec
TDO          1

***** CHANNEL f1 *****
NUC1          1H
P1            11.40 usec
PL1           -3.00 dB
SFO1          250.1315447 MHz

F2 - Processing parameters
SI            32768
SF            250.1300187 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
CB            1.00
PC            1.00
    
```

```

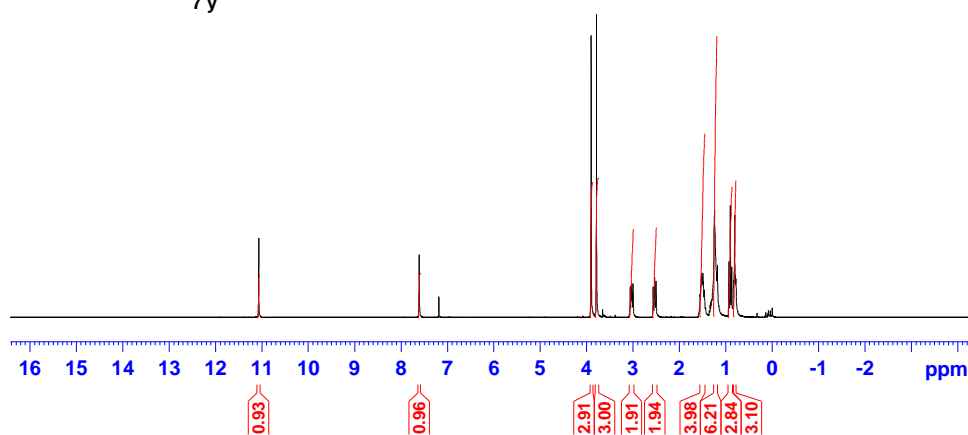
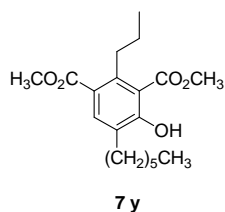
Current Data Parameters
NAME      080911.233
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080912
Time     2.43
INSTRUM spect
PROBHD   5 mm QNP 1H/13
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        2048
DS        4
SWH       15000.000 Hz
FIDRES   0.228882 Hz
AQ        2.1845834 sec
RG        2050
DM        33.333 usec
DE        10.00 usec
TE        298.2 K
D1        2.0000000 sec
d11       0.0300000 sec
DELTA    1.8999998 sec
TDO       1

***** CHANNEL f1 *****
NUC1      13C
P1        10.20 usec
PL1       0.00 dB
SFO1      62.901260 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPD2     70.00 usec
PL12      14.00 dB
PL13      14.00 dB
PL2       -3.00 dB
SFO2      250.1310005 MHz

F2 - Processing parameters
SI        32768
SF        62.8952350 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



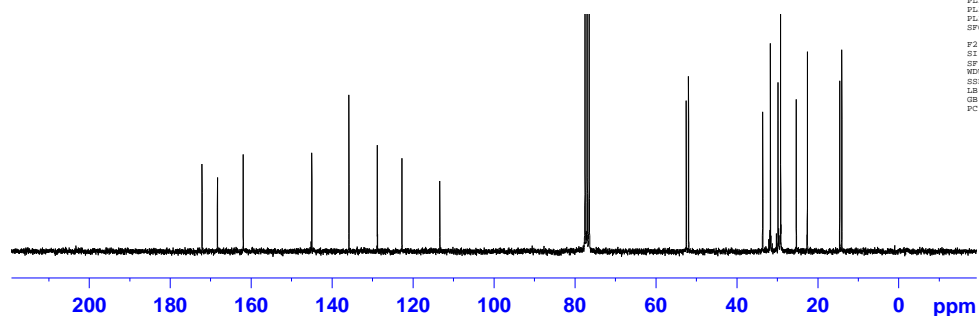
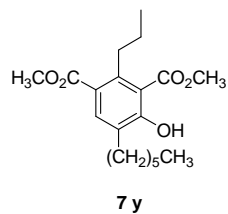
```

Current Data Parameters
NAME      080903.212
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080903
Time     11.04
INSTRUM spect
PROBHD   5 mm QNP 1H/13
PULPROG zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       5165.289 Hz
FIDRES   0.078816 Hz
AQ        6.3439350 sec
RG        362
DM        96.800 usec
DE        10.00 usec
TE        298.2 K
D1        1.0000000 sec
TDO       1

***** CHANNEL f1 *****
NUC1      1H
P1        11.40 usec
PL1       -3.00 dB
SFO1      250.131447 MHz

F2 - Processing parameters
SI        32768
SF        250.1300189 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

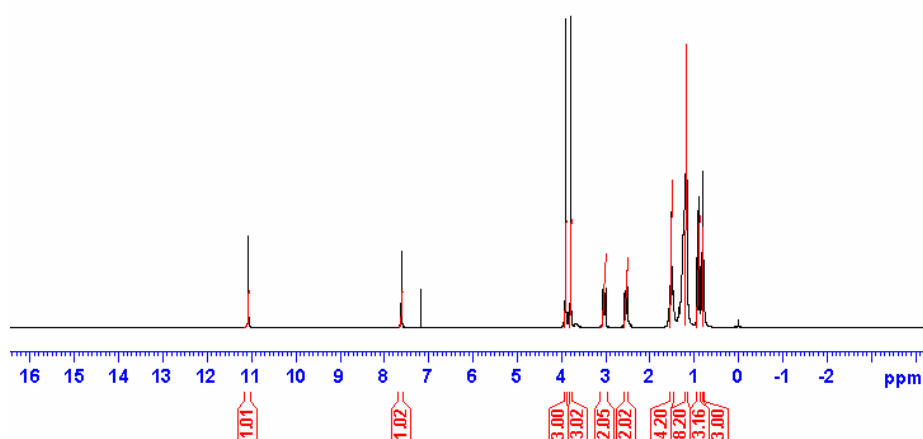
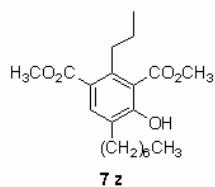
Current Data Parameters
NAME          060903.227
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20080904
Time         0.03
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          15000.000 Hz
FIDRES       0.228882 Hz
AQ           2.1845834 sec
RG           2050
RW           33.333 usec
DE           10.00 usec
TE           298.2 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TDO          1

===== CHANNEL f1 =====
NUC1          13C
P1           10.20 usec
PL1          0.00 dB
SFO1         62.901280 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         14.00 dB
PL13         14.00 dB
PL2          -3.00 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI           32768
SF           62.8952300 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```



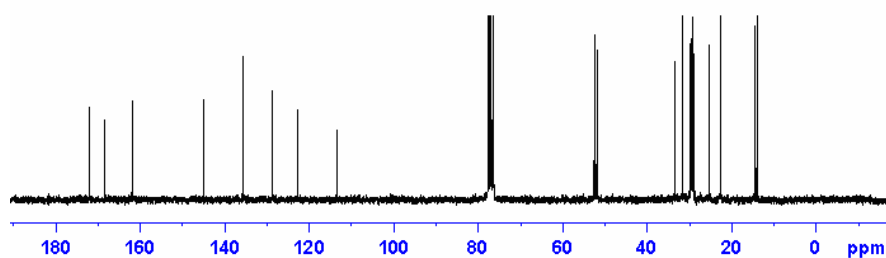
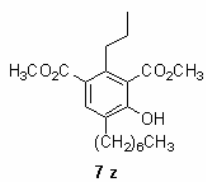
```

Current Data Parameters
NAME          060903.228
EXPNO        11
PROCNO       2

F2 - Acquisition Parameters
Date_        20080904
Time         0.03
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          5085.289 MHz
FIDRES       0.218826 MHz
AQ           6.3429258 sec
RG           486
RW           96.188 usec
DE           10.00 usec
TE           298.2 K
D1           3.0000000 sec
TDO          1

===== CHANNEL f1 =====
NUC1          1H
P1           13.18 usec
PL1          -5.18 dB
SFO1         250.1310441 MHz

F2 - Processing parameters
SI           32768
SF           250.1310441 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.18
    
```



```

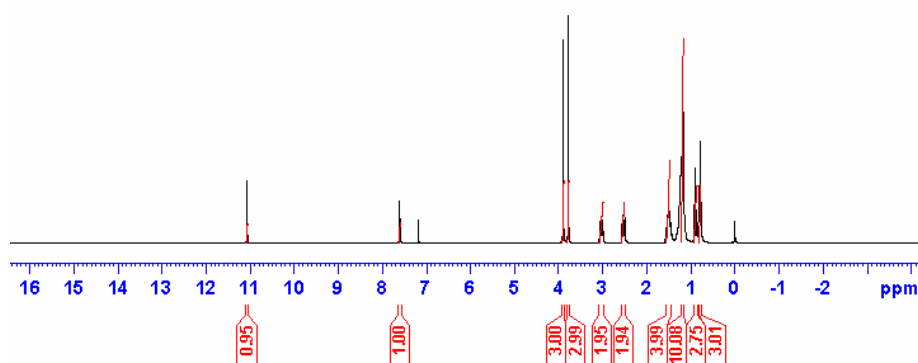
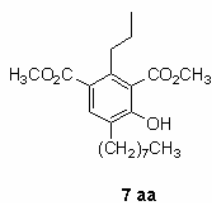
=====
CURRENT DATA PARAMETERS
NAME      081112_222
EXPNO    13
PROCNO   1

F2 - ACQUISITION PARAMETERS
Date_    20110114
Time     18.28
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        2
SWH       15180.888 Hz
FIDRES    0.20282 Hz
AQ         2.1805514 sec
RG         250
DN         95.253 uHz
DC         10.00 uHz
VE         288.2 Hz
SI         2.0000000 sec
SFO       101.6253000 MHz
AQ1       1.0000000 sec
DELTA     1.0000000 sec
TDS       1

===== CHANNEL f1 =====
NUC1      13C
P1         18.28 uHz
PC1       0.00 dB
SFO1      101.6253000 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      78.00 uHz
PC12       1.00 dB
PC13       19.00 dB
PC2        7.00 dB
SFO2      400.1462000 MHz

F2 - PROCESSING PARAMETERS
SI         32768
SF         101.6253000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00
=====
    
```



```

=====
CURRENT DATA PARAMETERS
NAME      081112_218
EXPNO    13
PROCNO   1

F2 - ACQUISITION PARAMETERS
Date_    20110114
Time     11.31
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        2
SWH       11657.288 Hz
FIDRES    0.20282 Hz
AQ         4.9489250 sec
RG         250
DN         95.253 uHz
DC         10.00 uHz
VE         288.2 Hz
SI         1.0000000 sec
SFO       101.6253000 MHz
AQ1       1.0000000 sec
DELTA     1.0000000 sec
TDS       1

===== CHANNEL f1 =====
NUC1      1H
P1         11.30 uHz
PC1       0.00 dB
SFO1      400.1462000 MHz

F2 - PROCESSING PARAMETERS
SI         32768
SF         101.6253000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00
=====
    
```



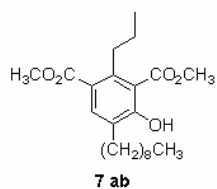
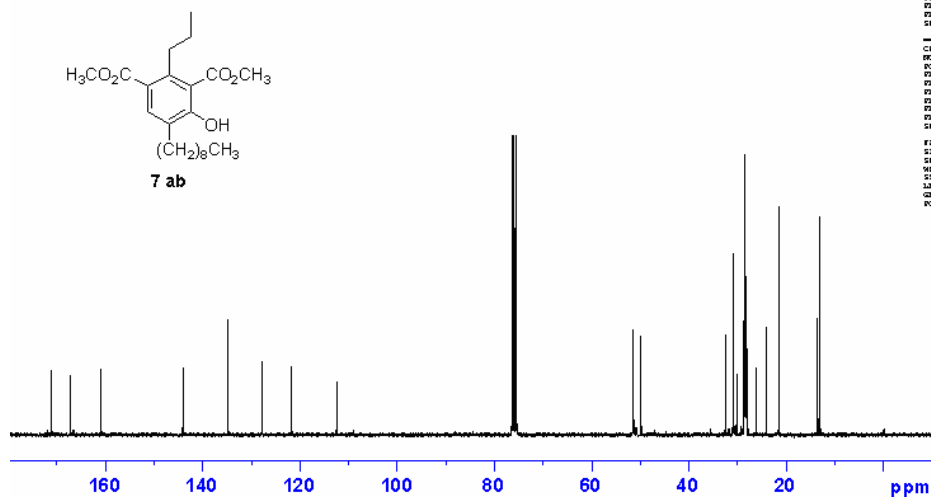

```

Current Data Parameters
NAME      080918.u318
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080918
Time     11.51
INSTRUM  spect
PROBHD   5 mm PABBO 90
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SFR      6188.119 Hz
FIDRES   0.094423 Hz
AQ        5.2953587 sec
RG         50.8
DW        80.800 usec
DE        6.50 usec
TE        298.2 K
D1        1.0000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       0.00 dB
PL1W     11.2525108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI        32768
SF        300.1300251 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



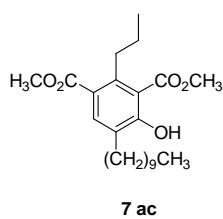
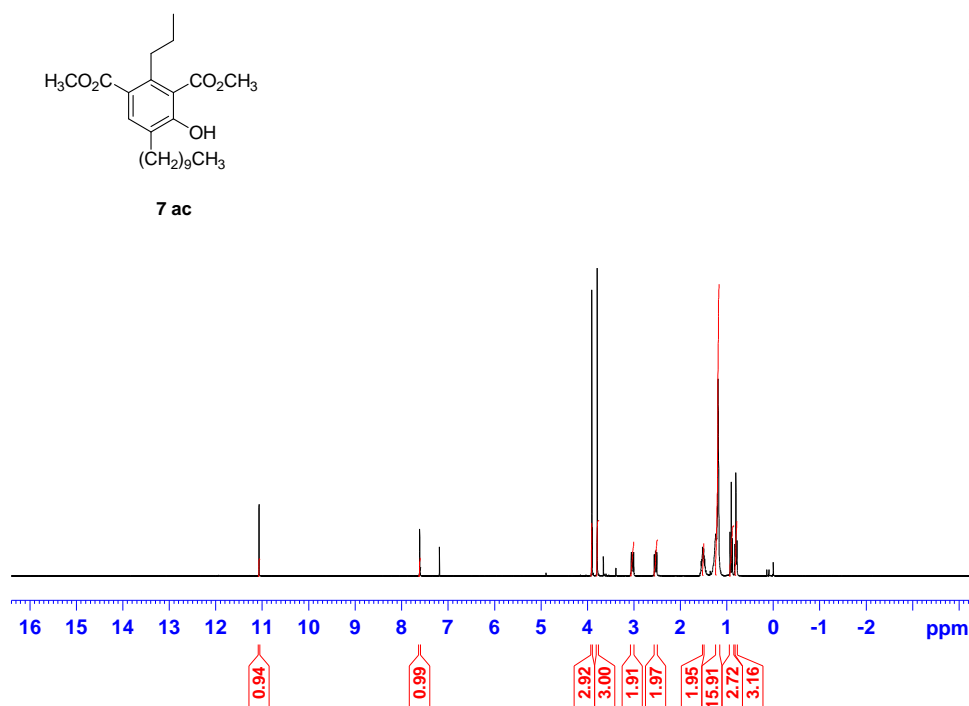
```

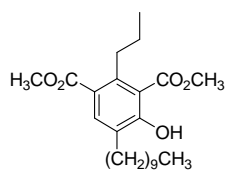
Current Data Parameters
NAME      080918.u318
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20080918
Time     11.51
INSTRUM  spect
PROBHD   5 mm PABBO 90
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SFR      6188.119 Hz
FIDRES   0.094423 Hz
AQ        5.2953587 sec
RG         50.8
DW        80.800 usec
DE        6.50 usec
TE        298.2 K
D1        1.0000000 sec
TD0       1

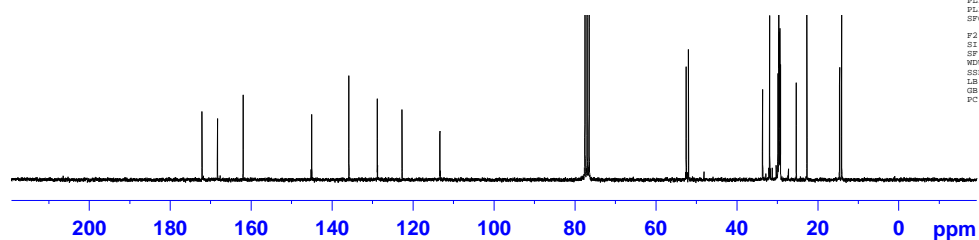
===== CHANNEL f1 =====
NUC1      1H
P1        10.00 usec
PL1       0.00 dB
PL1W     11.2525108 W
SFO1     300.1318534 MHz

F2 - Processing parameters
SI        32768
SF        300.1300251 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```





7 ac



```

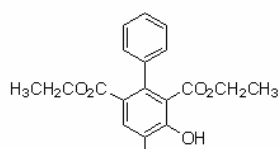
Current Data Parameters
NAME          080918_215
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_        20080919
Time         7.08
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          15000.000 Hz
FIDRES       0.228882 Hz
AQ           2.1845834 sec
RG           2050
SW           33.333 usec
DE           10.00 usec
TE           296.3 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TDO         1

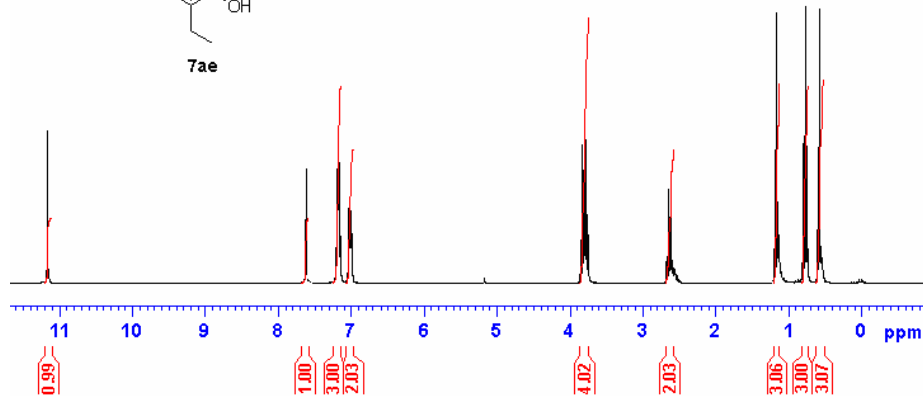
===== CHANNEL f1 =====
NUC1          13C
P1           10.20 usec
PL1          0.00 dB
SFO1         62.9012880 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL12         14.00 dB
PL13         14.00 dB
PL2          -3.00 dB
SFO2         250.1310005 MHz

F2 - Processing parameters
SI           32768
SF           62.8952950 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```



7ae



```

Current Data Parameters
NAME          080918_222
EXPNO        11
PROCNO       1

F2 - Acquisition Parameters
Date_        20080919
Time         14.41
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2048
DS           4
SWH          5085.249 Hz
FIDRES       0.818826 Hz
AQ           0.2439551 sec
RG           486
SW           26.118 usec
DE           10.00 usec
TE           296.3 K
D1           1.8000000 sec
DELTA        1.8000000 sec
TDO         1

===== CHANNEL f1 =====
NUC1          1H
P1           13.00 usec
PL1          -5.00 dB
SFO1         250.1310005 MHz

F2 - Processing parameters
SI           32768
SF           250.1310005 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```



```

Current Data Parameters
NAME: 010911_001
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20090904
Time : 11.58
INSTRUM : spect
PROBHD : 5 mm QNP 1H/13
PULPROG : zgpg30
TD : 65536
SOLVENT : CDCl3
NS : 1024
DS : 4
SWH : 12581.880 Hz
F2RES : 0.220000 Hz
AQ : 1.2140254 sec
RG : 3154
SI : 327.322 mmHg
DE : 10.00 mmHg
TE : 298.2 K
D1 : 2.0000000 sec
d11 : 0.1000000 sec
DELTA : 1.4999999 sec
TD0 : 1

===== CHANNEL f1 =====
NUC1 : 13C
P1 : 10.00 mmHg
PL1 : 0.00 dB
SFO1 : 101.625380 MHz

===== CHANNEL f2 =====
CPDPRG2 : waltz16
NUC2 : 1H
PCPD2 : 10.00 mmHg
PL12 : 14.00 dB
PL13 : 14.00 dB
PL2 : -5.00 dB
SFO2 : 500.136185 MHz

F2 - Processing parameters
SI : 32768
SF : 101.625380 MHz
WDW : EM
SSB : 0
LB : 0.00 Hz
GB : 0
PC : 1.48
    
```

