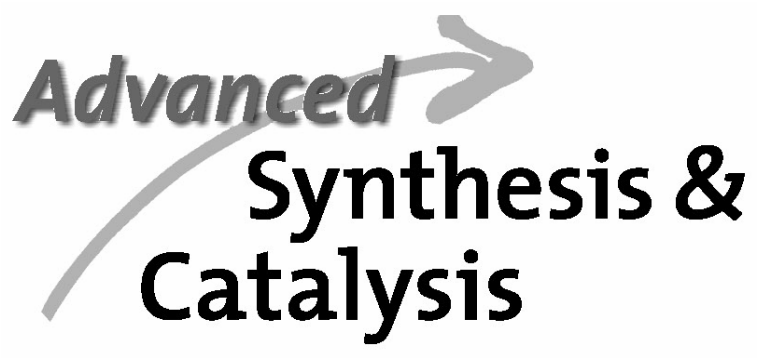

Supporting Information

Advanced Synthesis & Catalysis

Supporting Information

Enantioselective Organocatalytic Reactions of 4-Hydroxycoumarine and 4-Hydroxypyrone with α,β-Unsaturated Aldehydes – An Efficient Michael Addition-Acetalization Cascade to Chromenones, Quinolinones and Pyranones

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Experimental

General Remarks: Unless otherwise stated, all commercially available compounds were used as provided without further purification. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator, visualised by irradiation with UV light. Column chromatography was carried out using silica gel Merck 60 (particle size 0.040-0.063 mm).
^{1}H-NMR and ^{13}C-NMR were recorded on a Bruker AM 250, AV 300 or AV 400 spectrometer in $CDCl_3$. Chemical shifts (δ) are given in ppm, coupling constants (*J*) in Hz. Mass spectra (MS-ESI) were conducted on Micromass/ Waters Qtof Ultima 3 instrument and (MS-EI, 70 eV) were conducted on GC-MS Shimadzu QP2010 (column: Equity®-5, length × I.D. 30 m × 0.25 mm, df 0.25 µm, lot # 28089-U, Supelco). IR spectra were recorded on a Jasco FT/IR-420 spectrometer and are reported in terms of frequency of absorption (cm^{-1}).

General procedure: A screw capped test tube equipped with a stir bar was charged with aldehyde (1,30 equiv), catalyst (0,02 equiv) and solvent (0,20 M) at the temperature indicated. The cyclic 1,3-dicarbonyl (1.00 equiv) compound was then added. The mixture was allowed to stir at the temperature indicated for 48 – 96h. The crude reaction mixture was directly subjected to column chromatography (hexane: ethyl acetate 5:1 → hexane: ethyl acetate 1:1) on silica gel to afford the corresponding product.

(*4R*)-4-Ethyl-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(*2H*)-one (3a):
isolated as yellow solid; Mp: 120 - 121 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.75 – 7.71 (m, 1H), 7.47 – 7.42 (m, 1H), 7.23 – 7.15 (m, 2H), 5.66 (d, *J* = 8.8 Hz, 1H), 5.06 (bs, 1H), 2.87 – 2.80 (m, 1H), 2.29 (dt, *J* = 13.9 and 2.7 Hz, 1H), 2.11 – 1.88 (m, 2H), 1.39 – 1.37 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.93, 158.47, 152.50, 131.52, 123.76, 122.64, 116.30, 115.44, 104.69, 93.91, 31.44, 31.20, 26.48, 11.49; IR (NaCl): ν = 3372, 2965, 1683, 1622, 1573, 1277, 1174, 1104, 758 cm^{-1}; MS (EI): *m/z* (%) = 245.8 (5) $[M]^+$, 216.9 (20), 189 (26), 121 (100), 92 (51); $[\alpha]_D$ = -5.49 (c = 0.24, $CHCl_3$, 93% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL min-1, minor enantiomer: t_R = 13.26 min; major enantiomer: t_R = 15.78 min.

(*4R*)-2-Hydroxy-4-propyl-3,4-dihydropyrano-[3,2-*c*]-chromen-5(*2H*)-one (3b):
isolated as colorless solid; Mp: 64 - 66 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.78 – 7.75 (m, 1H), 7.50 – 7.44 (m, 1H), 7.24 – 7.19 (m, 2H), 5.66 (d, *J* = 8.0 Hz, 1H), 4.33 (bs, 1H), 2.98 – 2.91 (m, 1H), 2.28 (dt, *J* = 13.7 and 2.5 Hz, 1H), 1.95 – 1.87 (m, 1H), 1.59 – 1.25 (m, 3H), 0.95 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.65, 158.17, 152.62, 131.50, 123.75, 122.63, 116.41, 115.49, 105.00, 93.85, 35.94, 31.88, 29.76, 20.21, 13.95; IR (NaCl): ν = 3371, 2959, 1677, 1625, 1573, 1162, 1106, 1043, 758, 737 cm^{-1}; MS (EI) m/z (%) = 259.9 (8) $[M]^+$, 242.1 (5), 218.1 (11), 189.0 (82), 174.9 (17), 121.1 (100), 92.0 (47); $[\alpha]_D$ = +1.99 (c = 0.40, $CHCl_3$, 92% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 48.98 min; major enantiomer: t_R = 64.66 min.

(*4R*)-4-Butyl-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3c):
isolated as pale yellow solid; Mp: 101 – 103 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.78 – 7.74 (m, 1H), 7.50 – 7.44 (m, 1H), 7.27 – 7.18 (m, 2H), 5.66 (d, *J* = 8.7 Hz, 1H), 4.38 (bs, 1H), 2.96 – 2.90 (m, 1H), 2.28 (dt, *J* = 13.8 and 2.7 Hz, 1H), 1.99 -1.86 (m, 2H), 1.46 – 1.25 (m, 5H), 0.91 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.66, 158.16, 152.61, 131.49, 123.75, 122.63, 116.40, 115.49, 105.02, 93.85, 33.44, 31.86, 29.91, 29.20, 22.61, 13.98; IR (NaCl): ν = 3352, 2958, 2931, 1685, 1622, 1407, 1108, 758, 1011, 738 cm^{-1}; MS (EI) m/z (%) = 274.2 (7) $[M]^+$, 257.1 (7), 217.1 (18), 189.1 (42), 175.1 (22), 121.2 (100), 92 (27), 57.0 (67); $[\alpha]_D$ = -2.80 (c = 0.50, $CHCl_3$, 92% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 49.38 min; major enantiomer: t_R = 64.00 min.

(*4R*)-4-Heptyl-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3d):
isolated as colourless oil, yield: 99%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.77 – 7.74 (m, 1H), 7.49 – 7.43 (m, 1H), 7.25 – 7.17 (m, 2H), 5.66 (d, *J* = 8.7 Hz, 1H), 4.55 (s, 1H), 2.95 – 2.89 (m, 1H), 2.28 (dt, *J* = 13.9 and 2.6 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.35 – 1.26 (m, 11H), 0.89 – 0.84 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.71, 158.20, 152.58, 131.47, 123.74, 122.62, 116.37, 115.48, 104.99, 93.88, 33.76, 31.83, 31.80, 29.97, 29.50, 29.23, 27.09, 22.61, 14.04; IR (NaCl): ν = 3377, 2926, 2855, 1685, 1623, 1406, 1164, 1108, 758 cm^{-1}; MS (ESI) m/z (%) = 316.4 (9) $[M]^+$, 281.1 (8), 242.8 (12), 189.0 (56), 122.2 (8), 92.0 (36), 73.0 (100), 55.0 (66); $[\alpha]_D$ = +2.47 (c = 1.13, $CHCl_3$, 91% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 97/3, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 96.21 min; major enantiomer: t_R = 110.08 min.

(*4R*)-4-Decyl-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3e):

isolated as white solid; Mp: 82 – 84 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.76 – 7.73 (m, 1H), 7.48 – 7.42 (m, 1H), 7.25 – 7.16 (m, 2H), 5.66 (bs, 1H), 4.62 (s, 1H), 2.95 – 2.89 (m, 1H), 2.28 (dt, *J* = 13.8 and 2.6 Hz, 1H), 1.96 – 1.86 (m, 2H), 1.34 – 1.24 (m, 17H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.74, 158.23, 152.57, 131.47, 123.74, 122.62, 116.36, 115.48, 104.98, 93.89, 33.77, 31.87, 31.82, 29.97, 29.61, 29.59, 29.58, 29.29, 27.10, 22.64, 14.06; IR (NaCl): ν = 3372, 2925, 2853, 1714, 1684, 1622, 1162, 1109, 758 cm^{-1}; MS (EI) m/z (%) = 358.1 (16) $[M]^+$, 357.1 (55), 136.6 (4); $[\alpha]_D$ = +2.79 (c = 1.15, $CHCl_3$, 91% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 97/3, flow rate = 0.5 mL min^{-1}, minor enantiomer: t_R = 76.63 min; major enantiomer: t_R = 97.59 min.

(*4S*)-2-Hydroxy-4-phenyl-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3f):

performed with 2 equiv aldehyde. Isolated as white solid; Mp: 182 – 184 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81 – 7.85 (m, 1H), 7.57 – 7.52 (m, 1H), 7.33 – 7.26 (m, 4H), 7.24 – 7.19 (m, 1H), 7.17 – 7.15 (m, 2H), 5.50 (bs, 1H), 4.26 (dd, *J* = 5.7 and 3.5 Hz, 1H), 4.02 (bs, 1H), 2.31 (dt, *J* = 13.6 and 3.1 Hz, 1H), 2.24 (dd, *J* = 8.8 and 5.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.02, 159.72, 153.06, 142.75, 131.93, 128.73 (2C), 127.37 (2C), 126.89, 123.91, 122.84, 116.67, 115.29, 102.06, 93.74, 36.70, 35.65; IR (NaCl): ν = 3370, 1687, 1623, 1573, 1493, 1113, 1083, 759, 735, 701 cm^{-1}; MS (EI) m/z (%) = 294.9 (46) $[M]^+$; $[\alpha]_D$ = +32.92 (c = 0.24, $CHCl_3$, 89% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 14.32 min; major enantiomer: t_R = 24.17 min.

(*4R*)-4-(2-Bromophenyl)-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3g):

performed with 2 equiv aldehyde . Isolated as white solid; Mp: 173 – 175 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.90 - 7.88 (dd, *J* = 7.9 and 1.1 Hz, 1H), 7.64 – 7.62 (m, 1H), 7.60 – 7.55 (m, 1H), 7.36 – 7.31 (m, 2H), 7.21 – 7.16 (m, 1H), 7.13 – 7.09 (td, *J* = 7.7 and 1.6 Hz, 1H), 7.00 – 6.47 (dd, *J* = 7.5 and 1.5 Hz, 1H), 5.52 – 5.48 (m, 1H), 4.65 (dd, *J* = 6.2 and 3.3 Hz, 1H), 3.44 (d, *J* = 7.2 Hz, 1H), 2.45 (dt, *J* = 13.9 and 2.9 Hz, 1H), 2.24 (ddd, *J* = 13.9, 9.0 and 6.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 161.42, 160.03, 153.17, 141.26, 133.72, 132.09, 128.60, 128.44, 127.44, 124.20, 123.96, 122.84, 116.77, 115.16, 101.78, 93.82, 35.71, 34.34; IR (NaCl): ν = 3394, 1710, 1686, 1624, 1574, 1400, 1111, 1087, 759, 735 cm^{-1}; MS (EI) m/z (%) = 372.9 (100) $[M]^+$ (Br-pattern), 355.3 (10), 322.1 (8), 290.9 (9), 195.9 (6), 162.9 (9); $[\alpha]_D$ = +45.61(c = 0.35, $CHCl_3$, 93% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1}, minor enantiomer: t_R = 27.15 min; major enantiomer: t_R = 37.71 min.

(*4S*)-4-(4-Bromophenyl)-2-hydroxy-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3h): performed with 2 equiv aldehyde. Isolated as yellow solid; Mp: 168 – 170 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 – 7.85 (m, 1H), 7.59 – 7.54 (m, 1H), 7.44 – 7.42 (m, 2H), 7.34 – 7.30 (m, 2H), 7.08 – 7.06 (m, 2H), 5.52 (dd, *J* = 11.3 and 6.3 Hz, 1H), 4.23 (t, *J* = 4.9 Hz, 1H), 3.72 (d, J = 6.7 Hz, 1H), 2.28 – 2.25 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 161.95, 159.88, 153.01, 141.91, 132.12, 131.82 (2C), 129.11 (2C), 124.01, 122.87, 120.75, 116.69, 115.17, 101.62, 93.52, 39.50, 35.05; IR (NaCl): ν = 3402, 1685, 1623, 1572, 1490, 1073, 1007, 760, 736 cm^{-1}; MS (EI) m/z (%) = 372.9 (100) $[M]^+$ (Br-pattern), 360.1 (7), 144.8 (19); $[\alpha]_D$ = +44.80 (c = 0.25, $CHCl_3$, 94% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 75/25, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 10.68 min; major enantiomer: t_R = 21.00 min.

(*4S*)-2-Hydroxy-4-(4-methoxyphenyl)-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (3i): performed with 2.6 equiv aldehyde. Isolated as white solid; Mp: 79 – 81 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88 – 7.86 (m, 1H), 7.58 – 7.53 (m, 1H), 7.35 – 7.29 (m, 2H), 7.11 – 7.09 (m, 2H), 6.85 – 6.83 (m, 2H), 5.55 – 5.50 (m, 1H), 4.25 (dd, *J* = 5.8 and 3.3 Hz, 1H), 3.77 (s, 3H), 3.50 (d, *J* = 7.1 Hz, 1H), 2.32 (dt, *J* = 13.6 and 2.9 Hz, 1H), 2.22 (ddd, *J* = 13.5, 9.0 and 5.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 161.87, 159.41, 158.54, 153.11, 134.82, 131.89, 128.40, 123.89, 122.80, 116.71, 114.67, 114.22, 102.42, 93.78, 55.29, 36.94, 34.91; IR (NaCl): ν = 3393, 1684, 1621, 1510, 1248, 763 cm^{-1}; MS (EI) m/z (%) = 346.9 (34) $[M + Na]^+$, 325.0 (100), 308.3 (16), 162.9 (11); $[\alpha]_D$ = +50.23 (c = 0.21, $CHCl_3$, 94% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 85/15, flow rate = 1.0 mL min^{-1}, minor enantiomer: t_R = 17.24 min; major enantiomer: t_R = 31.23 min.

(*4R*)-4-Ethyl-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6a): isolated as colorless oil; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 5.73 (s, 1H), 5.48 – 5.42 (m, 1H), 2.74 – 2.64 (m, 1H), 2.22 – 2.14 (m, 1H), 2.18 (s, 3H), 2.08 – 1.90 (m, 2H), 1.82 – 1.68 (m, 1H), 1.34 – 1.22 (m, 1H), 0.98 (t, *J* = 6.3 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.75, 163.33, 160.58, 101.97, 100.36, 93.31, 31.14, 30.66, 26.15, 19.67, 11.39; IR (NaCl): ν = 3422, 1677, 1648, 1576 cm^{-1}; MS (EI) m/z (%) = 211.1 (19) $[M + H]^+$, 181.8 (43), 139.6 (100), 123.4 (29), 85.2 (80), 69.0 (94); $[\alpha]_D$ = -24.06 (c = 0.32, $CHCl_3$, 92% ee); HPLC conditions: AD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 58.50 min; major enantiomer: t_R = 63.34 min.

(*4R*)-2-Hydroxy-7-methyl-4-propyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6b):
isolated as colorless oil; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 5.73 (s, 1H), 5.46 (dd, *J* = 9.3 and 2.4 Hz, 2H), 2.82 – 2.73 (m, 1H), 2.20 – 2.12 (m, 1H), 2.18 (s, 3H), 1.93 – 1.66 (m, 2H), 1.59 – 1.19 (m, 3H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.89, 163.52, 160.45, 102.02, 100.47, 93.37, 35.62, 31.58, 29.01, 20.05, 19.60, 13.92; IR (NaCl): ν = 3394, 1683, 1651, 1575, 1184, 1139, 1001 cm^{-1}; MS (EI) m/z (%) = 225.2 (15) $[M + H]^+$, 181.9 (29), 153.7 (25), 139.6 (100), 123.5 (28), 85.1 (62), 69.0 (77), 53.0 (40); $[\alpha]_D$ = -43.08 (c = 0.07, $CHCl_3$, 92% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 45.33 min; major enantiomer: t_R = 51.09 min.

(*4R*)-4-Butyl-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6c):
isolated as colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.73 (s, 1H), 5.46 (dd, *J* = 9.2 and 2.0 Hz, 1H), 2.80 – 2.70 (m, 1H), 2.20 – 2.12 (m, 1H), 2.17 (s, 3H), 2.05 – 1.71 (m, 3H), 1.43 – 1.22 (m, 5H), 0.92 – 0.86 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 164.38, 162.87, 160.59, 102.20, 100.14, 93.23, 33.19, 31.85, 29.17, 29.12, 22.66, 19.73, 14.03; IR (NaCl): ν = 3371, 2957, 2930, 1684, 1651, 1577, 1448, 1184, 1139, 1002 cm^{-1}; MS (ESI) m/z (%) = 239.3 (12) $[M + H]^+$, 182.9 (14), 154.7 (14), 139.6 (100), 123.5 (23), 85 (54), 69 (58), 55 (48); $[\alpha]_D$ = -33.15 (c = 0.54, $CHCl_3$, 91% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 47.74 min; major enantiomer: t_R = 53.98 min.

(*4R*)-4-Heptyl-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6d):
isolated as colorless oil; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 5.73 - 5.72 (m, 1H), 5.47 – 5.44 (m, 1H), 3.87 – 3.85 (m, 1H), 2.80 – 2.73 (m, 1H), 2.21 – 2.13 (m, 1H), 2.17 (s, 3H), 2.05 – 1.65 (m, 3H), 1.35 – 1.19 (m, 10H), 0.89 – 0.84 (m, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.38, 162.87, 160.57, 102.21, 100.14, 93.23, 33.49, 31.82, 29.54, 29.27, 29.22, 27.00, 22.63, 19.73, 14.07; IR (NaCl): ν = 3421, 1647 cm^{-1}; MS (ESI) m/z (%) = 280.2 (17) $[M]^+$, 249.0 (6), 234.7 (4); $[\alpha]_D$ = -26.26 (c = 0.66, $CHCl_3$, 89% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 97/3, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 92.41 min; major enantiomer: t_R = 97.69 min.

(*4R*)-4-Decyl-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6e):
isolated as yellow solid; Mp: 46 – 48 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 5.73 – 5.72 (m, 1H), 5.48 – 5.43 (m, 1H), 3.72 (d, *J* = 7.0 Hz, 1H), 2.81 – 2.74 (m, 1H), 2.21 – 2.13 (m, 1H), 2.18 (s, 3H), 2.05 – 1.69 (m, 2H), 1.25 (bs, 17H), 0.90 – 0.85 (m, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.33, 162.81, 160.58, 102.22, 100.12, 93.22, 33.50, 31.89, 31.86, 29.62, 29.60, 29.32, 29.22, 27.02, 22.67, 19.73, 14.11; IR (NaCl): ν = 3405, 2924, 1683, 1651, 1576 cm^{-1}; MS (ESI) m/z (%) = 322.2 (21) $[M]^+$; $[\alpha]_D$ = -24.76 (c = 0.42, $CHCl_3$, 91% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 29.83 min; major enantiomer: t_R = 34.29 min.

(*4S*)-2-Hydroxy-7-methyl-4-phenyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6f):
isolated as yellow solid; Mp: 160 – 162 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.23 – 7.02 (m, 5H), 5.77 (s, 1H), 5.19 (dd, *J* = 8.7 and 2.6 Hz, 1H), 4.01 (dd, *J* = 5.7 and 3.7 Hz, 1H), 2.15 (s, 3H), 2.10 – 1.97 (m, 2H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.70, 164.03, 161.50, 142.80, 128.90, 128.58, 127.33, 126.76, 126.72, 100.16, 99.10, 93.20, 36.55, 34.88, 19.83; IR (NaCl): ν = 3360, 2961, 2854, 1708, 1654, 1577, 1452, 1193, 1118, 992, 763, 735, 702 cm^{-1}; MS (ESI) m/z (%) = 258.1 (100) $[M]^+$, 240.1 (16), 229.1 (73), 215.1 (23), 145.1 (45), 131.1 (57), 115.1 (42), 102.1 (60), 85.0 (43), 77.1 (38), 69.0 (31), 51.0 (23); $[\alpha]_D$ = -14.42 (c = 1.08, $CHCl_3$, 83% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 85/15, flow rate = 1.0 mL min^{-1}, minor enantiomer: t_R = 10.13 min; major enantiomer: t_R = 15.33 min.

(4S)-2-Hydroxy-4-(4-methoxyphenyl)-7-methyl-3,4-dihydropyrano-[4,3-b]-pyran-5(2H)-one (6g):
isolated as white solid; Mp: 166 – 168 °C; 1H NMR (300 MHz, $CDCl_3$): δ = 7.05 – 6.83 (system AB, *J* = 8.5 Hz, 4H), 5.83 – 5.85 (m, 1H), 5.26 – 5.23 (m, 1H), 4.07 (dd, *J* = 5.6 and 3.4 Hz, 1H), 3.77 (s, 3H), 2.25 – 2.17 (m, 2H), 2.23 (s, 3H), 2.08 (ddd, J = 13.5, 9.0 and 5.8 Hz, 1H); 13C NMR (75 MHz, $CDCl_3$): δ = 164.14, 163.64, 161.54, 158.45, 134.90, 128.36 (2C), 114.15 (2C), 99.89, 99.52, 93.22, 55.29, 36.95, 34.18, 19.87; IR (NaCl): = 3394, 1684, 1651, 1574, 1510, 1249, 1176, 1138, 1003 cm-1; MS (EI) m/z (%) = 288.0 (18) [M]+; = -15.59 (c = 0.30, CHCl3, 85% ee); HPLC conditions: AD-H column, n-hexane/2-propanol = 80/20, flow rate = 0.6 mL min-1, minor enantiomer: tR = 15.67 min; major enantiomer: tR = 26.08 min.

(*4R*)-4-(2-Chlorophenyl)-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6h): isolated as white solid; Mp: 245 – 247 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.40 – 7.36 (m, 1H), 7.16 – 7.10 (m, 2H), 6.94 – 6.90 (m, 1H), 5.84 (s, 1H), 5.27 – 5.23 (m, 1H), 4.48 (dd, *J* = 6.3 and 3.3 Hz, 1H), 3.29 (bs, 1H), 2.33 (dt, *J* = 14.3 and 3.1 Hz, 1H), 2.22 (s, 3H), 2.14 – 2.02 (m, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 164.79, 163.26, 161.88, 139.67, 133.65, 130.29, 128.40, 128.22, 126.73, 99.86, 98.57, 93.33, 34.11, 32.61, 19.94; IR (NaCl): ν = 3420, 1684, 1647, 1577, 1280, 1183, 1140 cm^{-1}; MS (EI) m/z (%) = 292.0 (16) $[M]^+$ (Cl-pattern); $[\alpha]_D$ = -18.69 (c = 1.07, MeOH, 95% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 39.71 min; major enantiomer: t_R = 42.18 min.

(*4S*)-4-(4-Bromophenyl)-2-hydroxy-7-methyl-3,4-dihydropyrano-[4,3-*b*]-pyran-5(2*H*)-one (6i): isolated as white solid; Mp: 86 – 88 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.41 – 7.01 (system AB, *J* = 8.5 Hz, 4H), 5.85 – 5.83 (m, 1H), 5.29 – 5.25 (m, 1H), 4.04 (t, *J* = 4.8 Hz, 1H), 2.22 (s, 4H), 2.13 – 2.09 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 164.61, 163.73, 161.82, 141.97, 131.72 (2C), 129.12 (2C), 120.60, 100.11, 98.85, 92.99, 36.51, 34.31, 19.85; IR (NaCl): ν = 3343, 1683, 1651, 1576, 1488, 1448, 1184, 1140, 1005, 737 cm^{-1}; MS (EI) m/z (%) = 336.9 (16) $[M]^+$ (Br-pattern); $[\alpha]_D$ = -26.57 (c = 0.29, $CHCl_3$, 82% ee); HPLC conditions: AD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL min^{-1}, minor enantiomer: t_R = 19.97 min; major enantiomer: t_R = 46.47 min.

(*4R*)-4-Propyl-3,4-dihydropyrano[3,2-*c*]-chromene-2,5-dione (7b):

To a dichloromethane solution (0.02 M) of **3b** (1.0 equiv) was added PCC (6.0 equiv). The resulting solution was stirred at room temperature for 24h and celite was added and filtered off. The solvent removed *in vacuo* and the residue was subjected to column chromatography. The product was isolated as white solid (94% yield); Mp: 115 – 117 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.84 (dd, *J* = 7.8 and 1.3 Hz, 1H), 7.63 – 7.56 (m, 1H), 7.38 – 7.31 (m, 2H), 3.34 – 3.25 (m, 1H), 2.96 (dd, *J* = 16.1 and 2.0 Hz, 1H), 2.84 (dd, *J* = 16.1 and 6.7 Hz, 1H), 1.74 – 1.62 (m, 1H), 1.56 – 1.25 (m, 3H), 0.93 (t, *J* = 7.1 Hz, 3H), 1.03 (t, *J* = 5.5 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 165.10, 161.08, 156.75, 152.96, 132.57, 124.53, 122.53, 116.78, 113.67, 107.83, 35.53, 33.30, 30.20, 19.52, 13.82; IR (NaCl): ν = 2953, 2930, 1787, 1714, 1649, 1386, 1131, 1097, 762, 739 cm^{-1}; MS (EI) m/z (%) = 257.8 (31) $[M]^+$, 215.0 (74), 187.0 (49), 131.0 (66), 121.1 (100), 103.1 (29), 92.0 (98), 77.0 (39), 63.0 (53), 53.0 (40); $[\alpha]_D$ = +30.84 (c = 0.35, $CHCl_3$, 89% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 90/10, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 16.28 min; major enantiomer: t_R = 22.24 min.

(4*R*)-4-Propyl-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (8b):

To a solution of **3b** (1.0 equiv) in ethanol was added sodium borohydride (10.0 equiv). The mixture was heated under reflux for 1 h. After cooling to room temperature, water was added and the resulting solution was acidified with sulphuric acid and extracted with dichloromethane. The combine extracts were concentrated under reduced pressure and to the residue was added conc. HCl. The resulting mixture was stirred at room temperature for 2 h. The clear solution was cooled, diluted with water and extracted with dichloromethane. The combined extracts washed with saturated aqueous sodium bicarbonate, water and dried over Na_2SO_4. The solvent was removed in vacuo and the residue was purified by column chromatography. The product was isolated as colourless oil in 83% yield. ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.70 (dd, *J* = 7.8 and 1.4 Hz, 1H), 7.48 – 7.41 (m, 1H), 7.26 – 7.19 (m, 2H), 4.50 – 4.42 (m, 1H), 4.28 – 4.16 (m, 1H), 2.84 – 2.76 (m, 1H), 1.98 – 1.93 (m, 2H), 1.91 – 1.80 (m, 1H), 1.53 – 1.16 (m, 3H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 162.66, 159.42, 152.30, 131.21, 123.60, 122.48, 116.42, 115.82, 105.57, 63.82, 35.56, 28.49, 24.42, 20.27, 14.03; IR (NaCl): ν = 2957, 2930, 1713, 1626, 1494, 1407, 1279, 1177, 1113, 1005, 757 cm^{-1}; MS (EI) m/z (%) = 244.0 (13) $[M]^+$, 201.1 (100), 187.1 (10), 145.1 (16), 128.1 (89), 115.1 (64), 92.1 (52), 77.1 (29), 63.0 (43), 53.0 (39); $[\alpha]_D$ = +6.12 (c = 0.49, $CHCl_3$, 91% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 16.23 min; major enantiomer: t_R = 19.09 min.

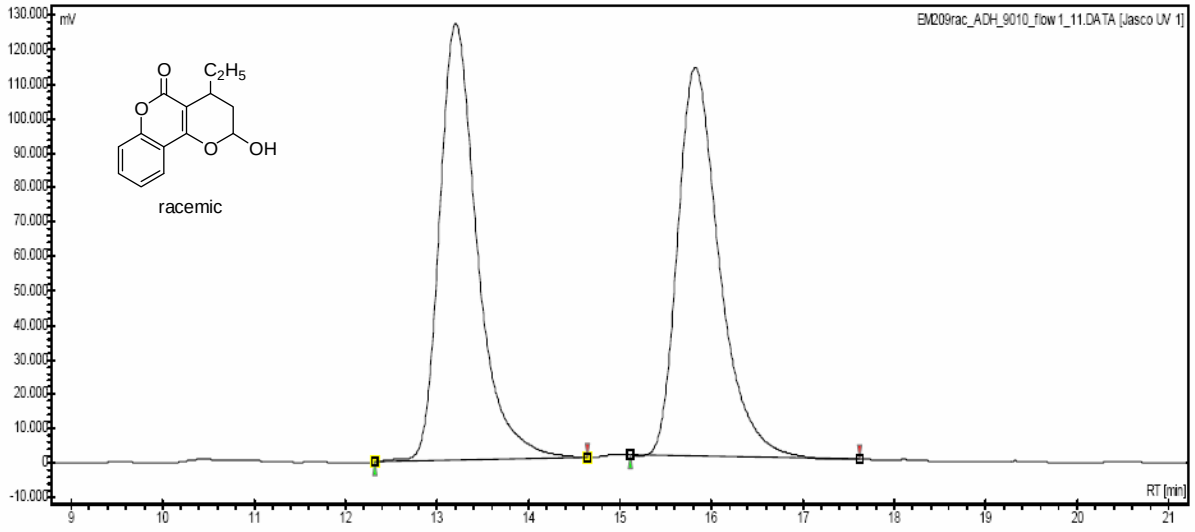
(*4R*)-4-Butyl-3,4-dihydro-*6H*-pyrano-[3,2-*c*]-quinoline-2,5-dione (10):

Synthesised according to the general procedure. The isolated intermediate was subsequently oxidized with PCC affording the product as white solid; Mp: 196 – 198 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.93 (d, *J* = 7.9 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.34 – 7.19 (m, 2H), 3.68 (s, 3H), 3.36 (dd, *J* = 13.0 and 6.7 Hz, 1H), 2.88 (d, *J* = 15.5 Hz, 1H), 2.71 (dd, *J* = 16.1 and 6.8 Hz, 1H), 1.67 – 1.56 (m, 1H), 1.44 – 1.16 (m, 5H), 0.82 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 166.65, 161.42, 153.23, 139.12, 131.31, 122.78, 122.37, 114.12, 113.93, 113.05, 33.45, 33.01, 30.41, 29.70, 28.48, 22.58, 13.86; IR (NaCl): ν = 2927, 2851, 1776, 1659, 1631, 1598, 1390, 1151, 757 cm^{-1}; MS (EI) m/z (%) = 285.1 (48) $[M]^+$, 229.1 (100), 201.1 (98), 149.0 (35), 77.1 (29); $[\alpha]_D$ = +32.42 (c = 0.33, $CHCl_3$, 89% ee); HPLC conditions: AD-H column, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 11.18 min; major enantiomer: t_R = 15.50 min.

(2*S*,4*S*)-2-(9*H*-Carbazol-9-yl)-4-phenyl-3,4-dihydropyrano-[3,2-*c*]-chromen-5(2*H*)-one (11): To a dichloromethane solution of **3f** (1.0 equiv) was added *p*-toluenesulfonic acid (0.3 equiv) and carbazole (1.1 equiv). The resulting solution was stirred for 48 h at room temperature. The crude reaction mixture was directly charged on silica gel and purified by column chromatography affording the product as white solid in 55% yield; Mp: 160 – 162 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 7.5 Hz, 2H), 7.68 (dd, *J* = 7.9 and 1.5 Hz, 1H), 7.54 – 7.47 (m, 1H), 7.39 – 7.13 (m, 13H), 6.51 (dd, *J* = 12.0 and 2.7 Hz, 1H), 4.50 (d, *J* = 4.2 Hz, 1H), 3.29 (ddd, *J* = 13.8, 12.0 and 5.5 Hz, 1H), 2.27 (dt, *J* = 13.7 and 2.2 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 161.85, 160.47, 153.10, 142.15, 138.81, 132.25, 129.06, 127.58, 127.42, 126.14, 124.16, 124.09, 122.89, 120.55, 120.48, 116.80, 114.95, 110.11, 102.35, 80.32, 36.48, 33.11; IR (NaCl): ν = 1714, 1629, 1490, 1454, 1389, 1307, 1265, 1072, 738, 703 cm^{-1}; MS (MALDI) m/z (%) = 444.7 (76) $[M + H]^+$, 283.4 (16), 252.1 (64), 237.1 (9), 193.3 (100); $[\alpha]_D$ = -21.54 (c = 0.98, $CHCl_3$, 85% ee); HPLC conditions: AS-H column, *n*-hexane/2-propanol = 50/50, flow rate = 0.6 mL min^{-1}, minor enantiomer: t_R = 19.41 min; major enantiomer: t_R = 29.97 min.

Chromatogram : EM209rac_ADH_9010_flow1_11

Data file: EM209rac_ADH_9010_flow1_11.DATA
Method: HPLC2_ADH_9010_flow1_acq30

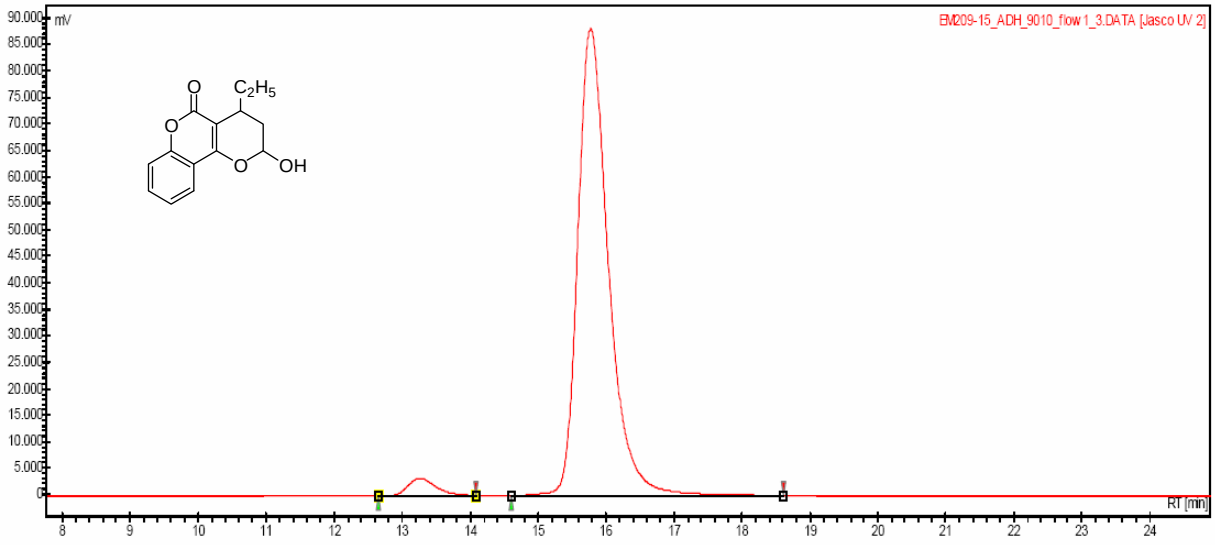


EM209rac_ADH_9010_flow1_11.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,320	13,208	14,641	50,534
2	15,110	15,825	17,624	49,466
Total				100,000

Chromatogram : EM209-15_ADH_9010_flow1_3

Data file: EM209-15_ADH_9010_flow1_3.DATA
Method: HPLC2_ADH_9010_flow1_acq30

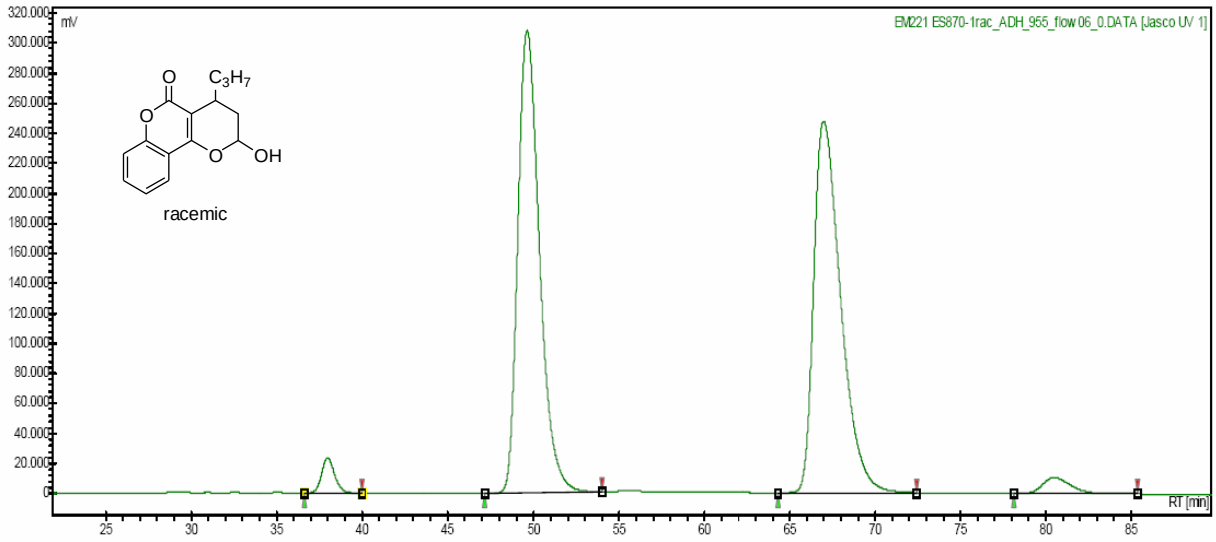


EM209-15_ADH_9010_flow1_3.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	12,652	13,258	14,088	3,401
2	14,613	15,775	18,619	96,599
Total				100,000

Chromatogram : ES870-1rac_ADH_955_flow06_0

Data file: ES870-1rac_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

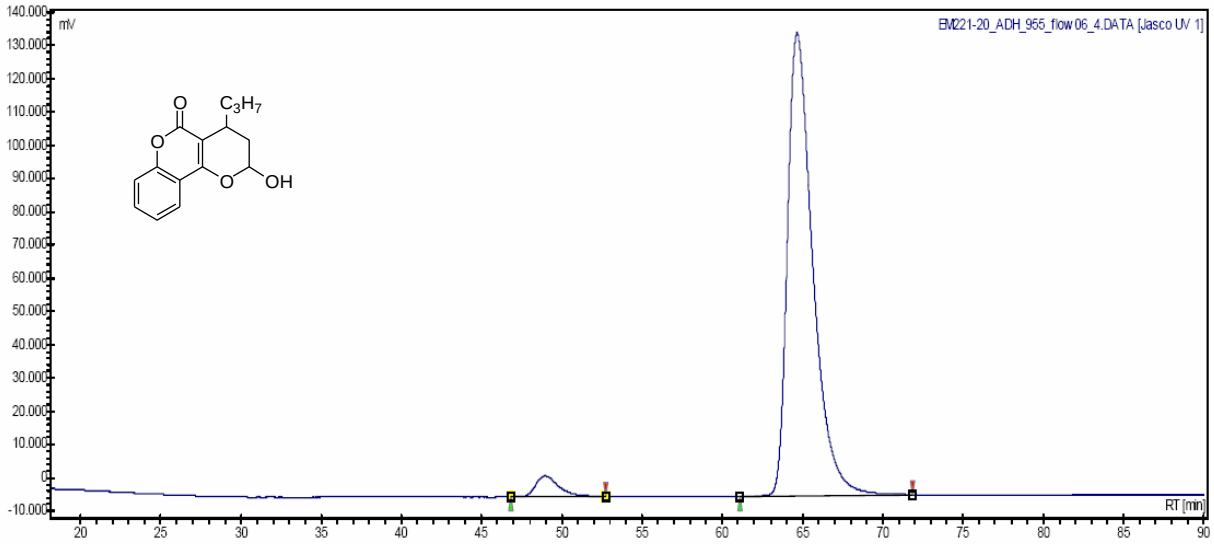


EM221 ES870-1rac_ADH_955_flow06_0.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	36,632	37,967	39,979	2,201
2	47,138	49,642	54,019	47,697
3	64,339	66,983	72,428	47,933
4	78,099	80,500	85,351	2,169
Total				100,000

Chromatogram : EM221-20_ADH_955_flow06_4

Data file: EM221-20_ADH_955_flow06_4.DATA
Method: HPLC2_ADH_955_flow06_acq90

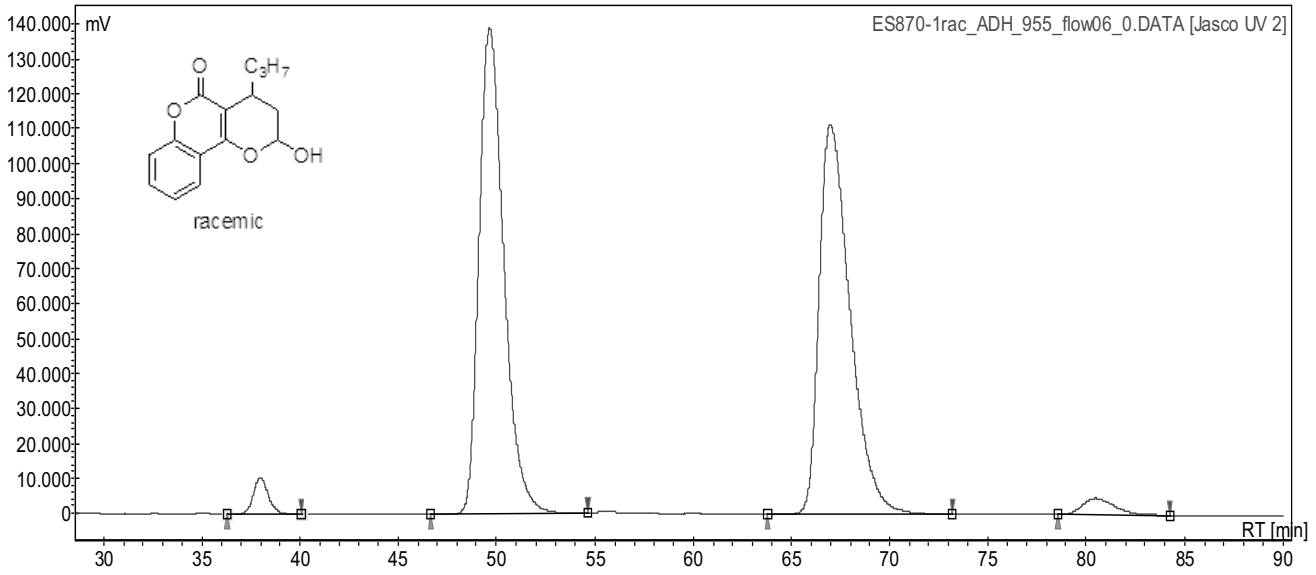


EM221-20_ADH_955_flow06_4.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	46,823	48,975	52,707	4,070
2	61,077	64,658	71,851	95,930
Total				100,000

Chromatogram : ES870-1rac_ADH_955_flow06_0

Data file: ES870-1rac_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

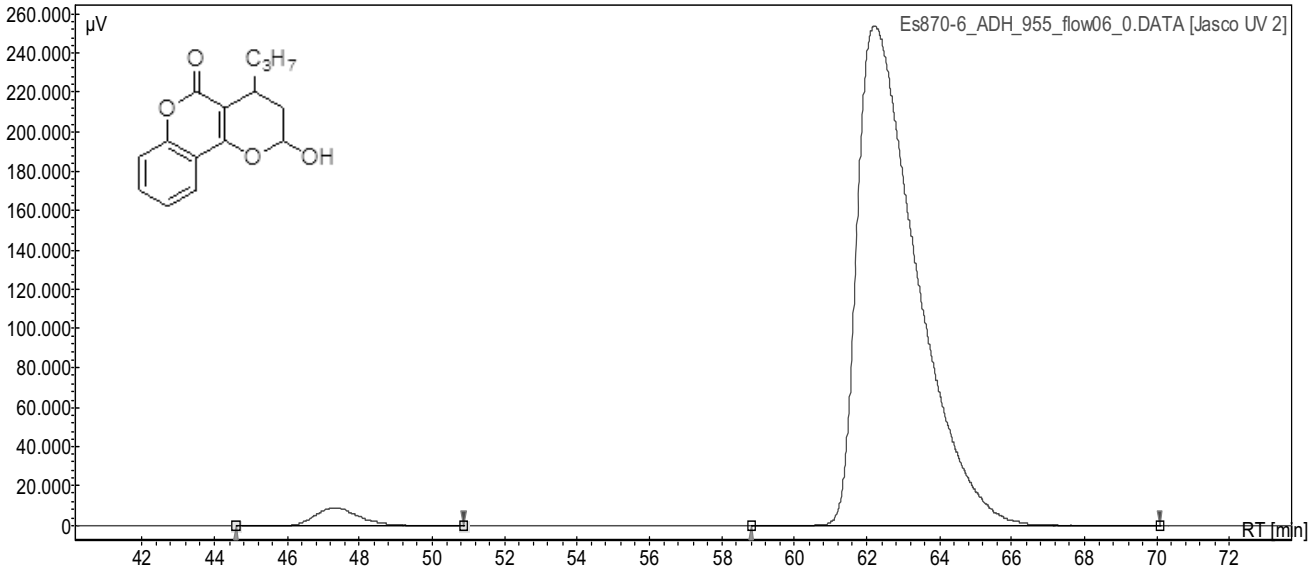


ES870-1rac_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	36,260	37,967	40,072	2,164
2	46,674	49,642	54,669	47,742
3	63,781	66,983	73,171	47,987
4	78,564	80,492	84,236	2,107
Total				100,000

Chromatogram : Es870-6_toluene_-20 grad_ADH_955_flow06_0

Data file: Es870-6_ADH_955_flow06_0.DATA
Method: HPLC1_ADH_955_flow06_acq_90

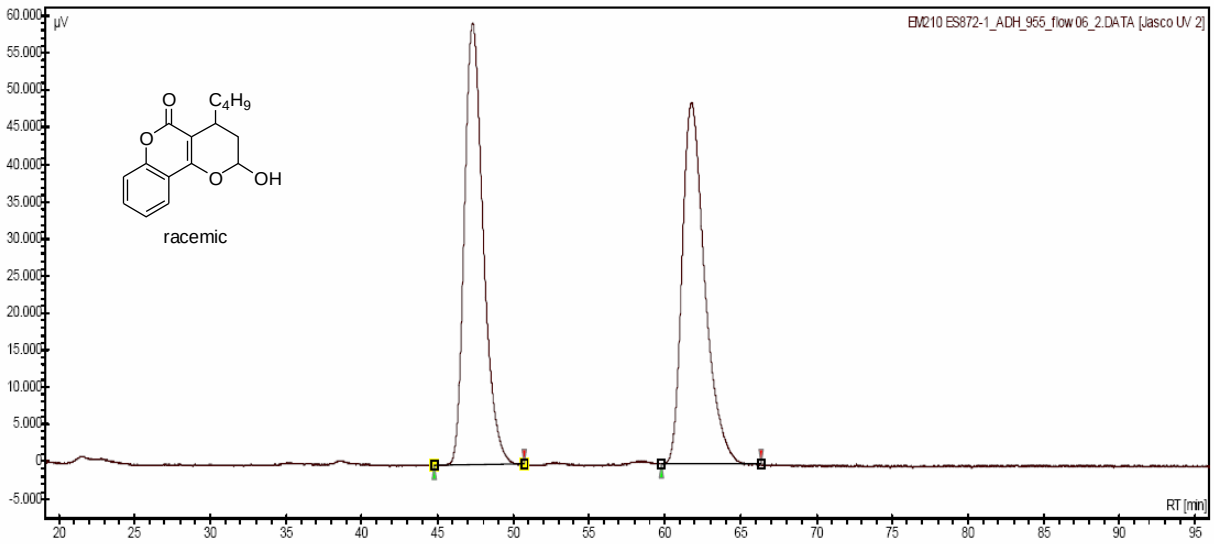


Es870-6_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	44,598	47,367	50,867	2,640
2	58,803	62,208	70,071	97,360
Total				100,000

Chromatogram : ES872-1_ADH_955_flow06_2

Data file: ES872-1_ADH_955_flow06_2.DATA
Method: HPLC1_ADH_955_flow06_acq_120

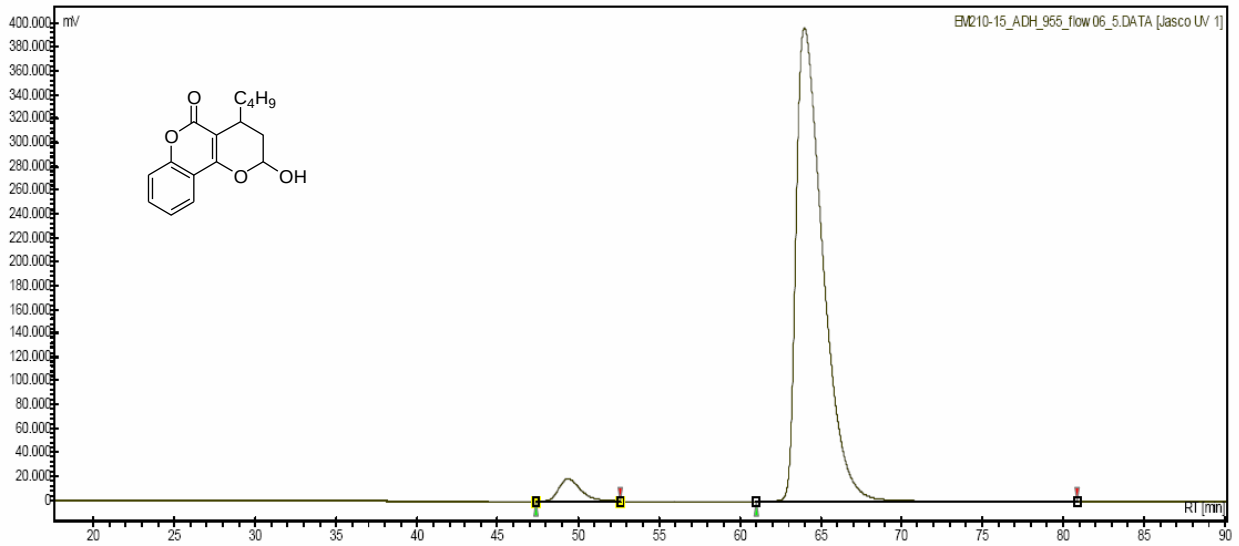


EM210 ES872-1_ADH_955_flow06_2.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	44,752	47,317	50,702	50,326
2	59,752	61,742	66,322	49,674
Total				100,000

Chromatogram : EM210-15_ADH_955_flow06_5

Data file: EM210-15_ADH_955_flow06_5.DATA
Method: HPLC2_ADH_955_flow06_acq90

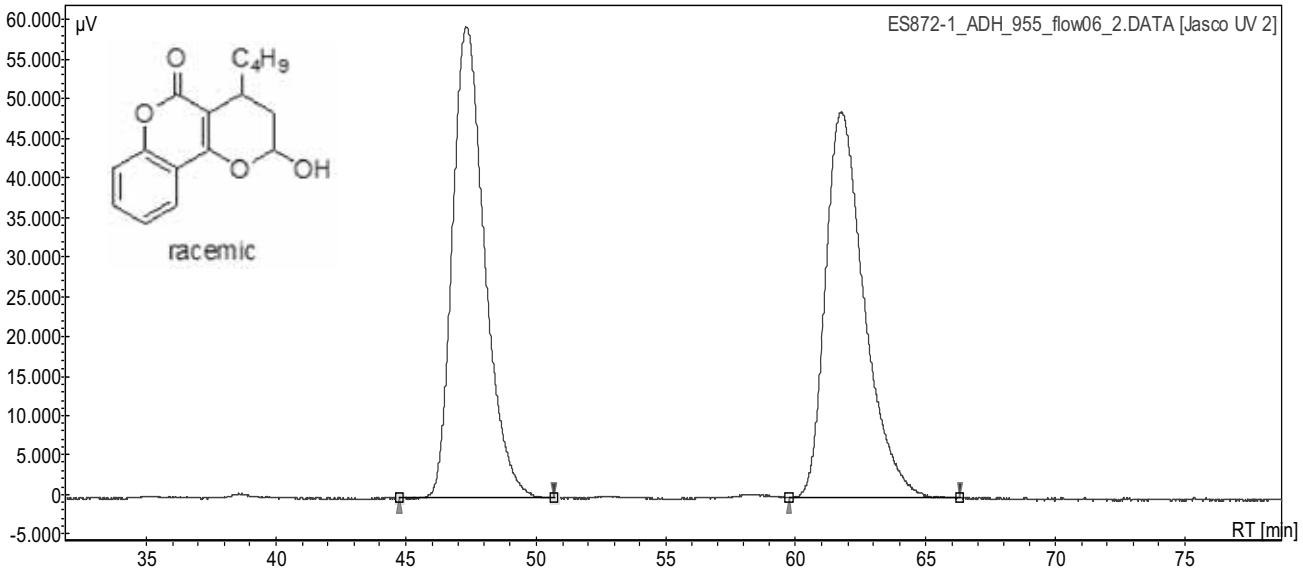


EM210-15_ADH_955_flow06_5.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	47,403	49,375	52,624	3,781
2	60,994	64,000	80,884	96,219
Total				100,000

Chromatogram : ES872-1_ADH_955_flow06_2

Data file: ES872-1_ADH_955_flow06_2.DATA
Method: HPLC1_ADH_955_flow06_acq_120

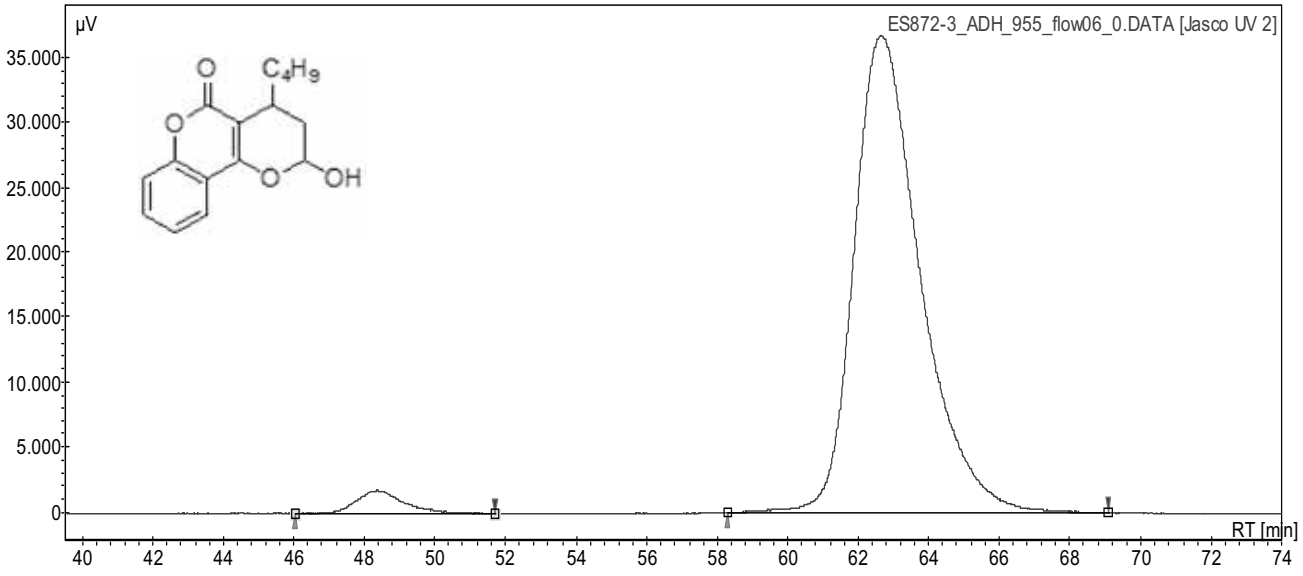


ES872-1_ADH_955_flow06_2.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	44,752	47,317	50,702	50,326
2	59,752	61,742	66,322	49,674
Total				100,000

Chromatogram : ES872-3_toluene_-20 grad_ADH_955_flow06_0

Data file: ES872-3_ADH_955_flow06_0.DATA
Method: HPLC1_ADH_955_flow06_acq_90

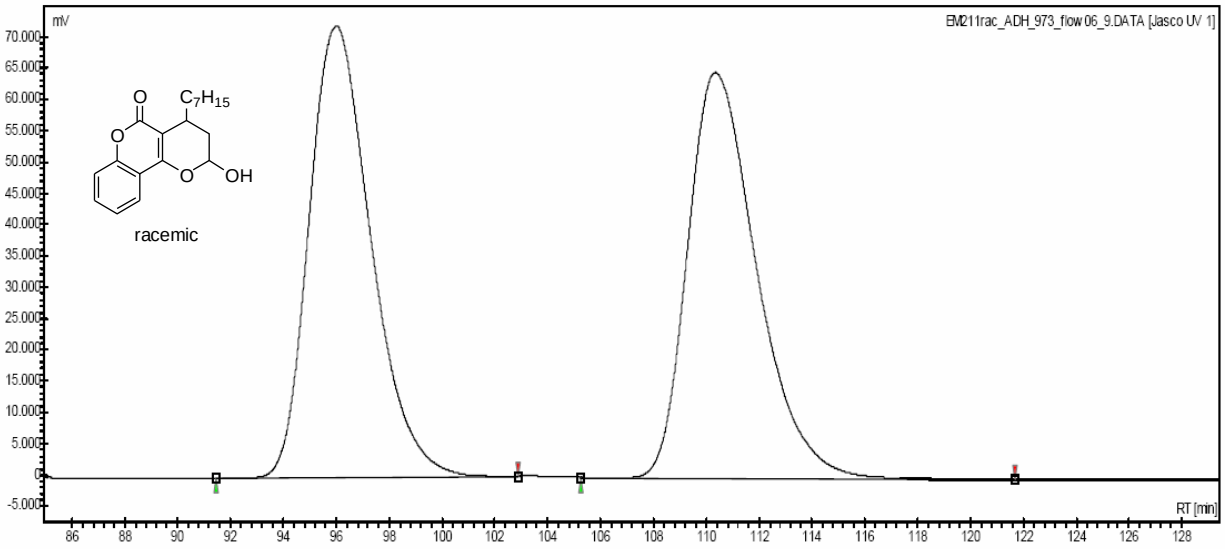


ES872-3_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	46,023	48,392	51,694	3,302
2	58,295	62,658	69,081	96,698
Total				100,000

Chromatogram : EM211rac_ADH_973_flow06_9

Data file: EM211rac_ADH_973_flow06_9.DATA
Method: HPLC2_ADH_973_flow06_acq150

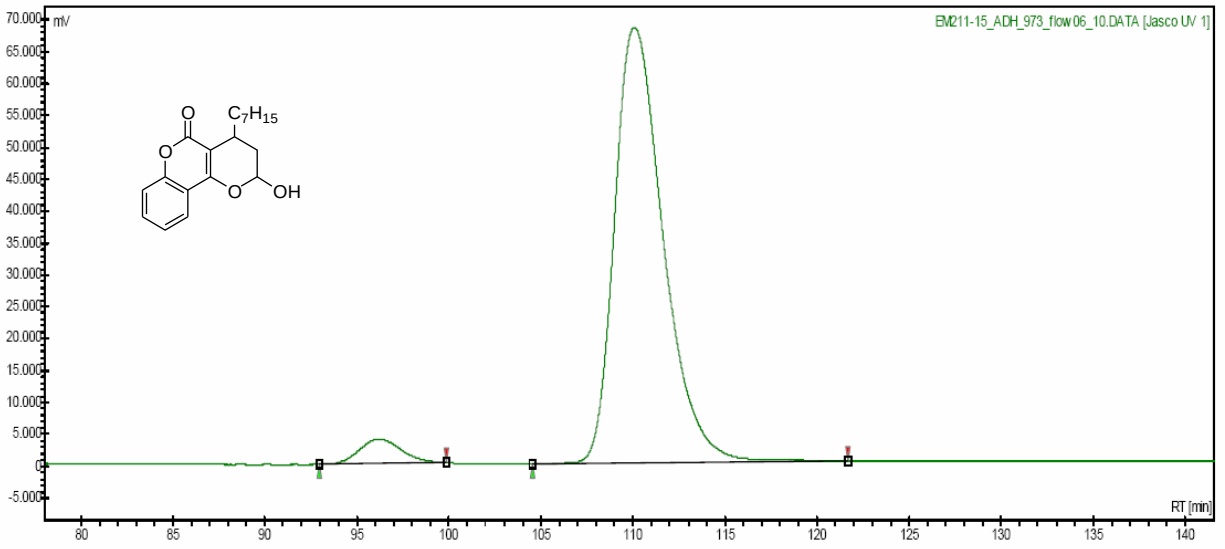


EM211rac_ADH_973_flow06_9.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	91,436	96,008	102,901	49,955
2	105,249	110,350	121,685	50,045
Total				100,000

Chromatogram : EM211-15_ADH_973_flow06_10

Data file: EM211-15_ADH_973_flow06_10.DATA
Method: HPLC2_ADH_973_flow06_acq150

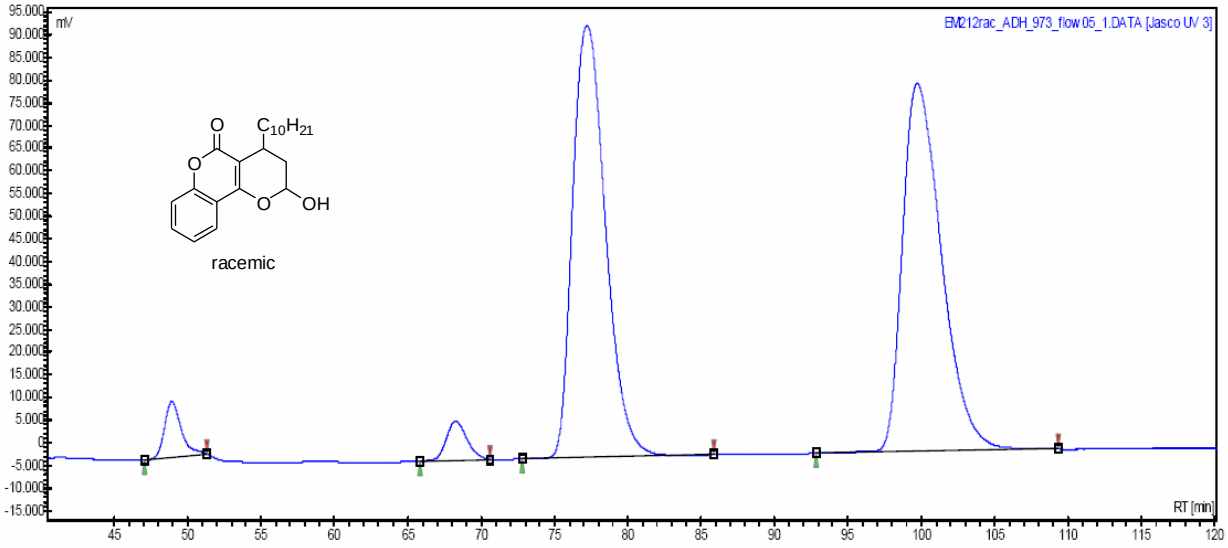


EM211-15_ADH_973_flow06_10.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	92,956	96,208	99,862	4,658
2	104,558	110,075	121,685	95,342
Total				100,000

Chromatogram : EM212rac_ADH_973_flow05_1

Data file: EM212rac_ADH_973_flow05_1.DATA
Method: HPLC2_ADH_973_flow05_acq140

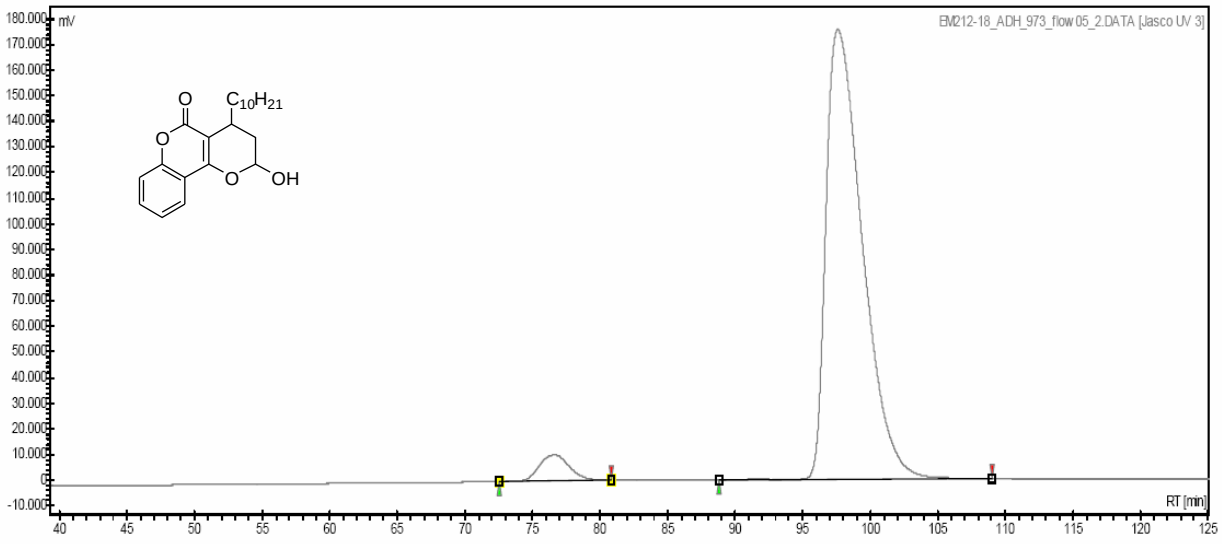


EM212rac_ADH_973_flow05_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
3	47,053	48,908	51,308	3,089
4	65,875	68,267	70,645	2,647
1	72,836	77,225	85,856	46,721
2	92,818	99,733	109,319	47,543
Total				100,000

Chromatogram : EM212-18_ADH_973_flow05_2

Data file: EM212-18_ADH_973_flow05_2.DATA
Method: HPLC2_ADH_973_flow05_acq140

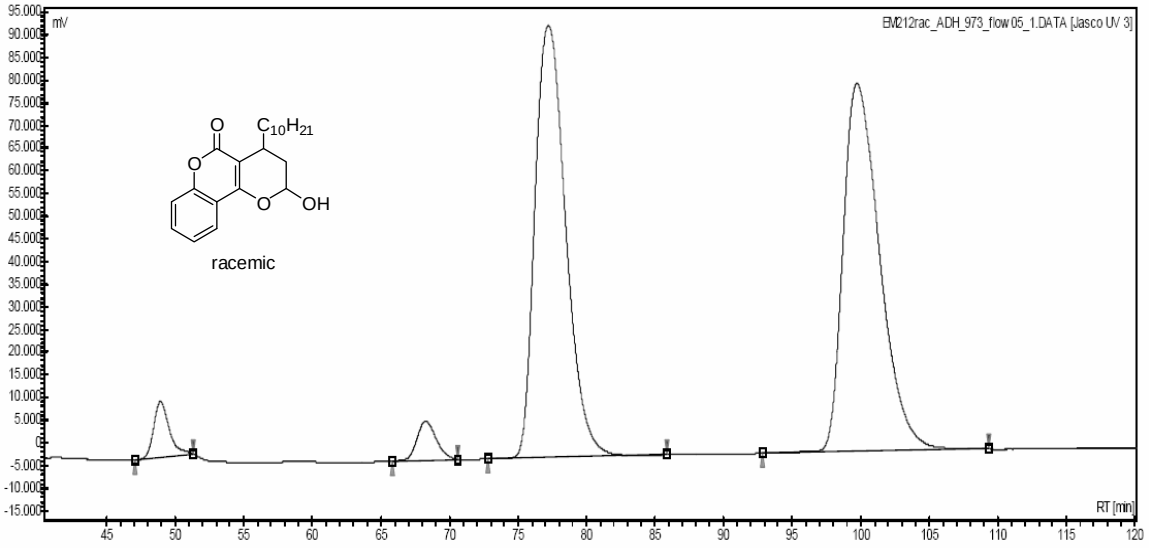


EM212-18_ADH_973_flow05_2.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	72,578	76,633	80,829	4,693
2	88,821	97,592	109,061	95,307
Total				100,000

Chromatogram : EM212rac_ADH_973_flow05_1

Data file: EM212rac_ADH_973_flow05_1.DATA
Method: HPLC2_ADH_973_flow05_acq140

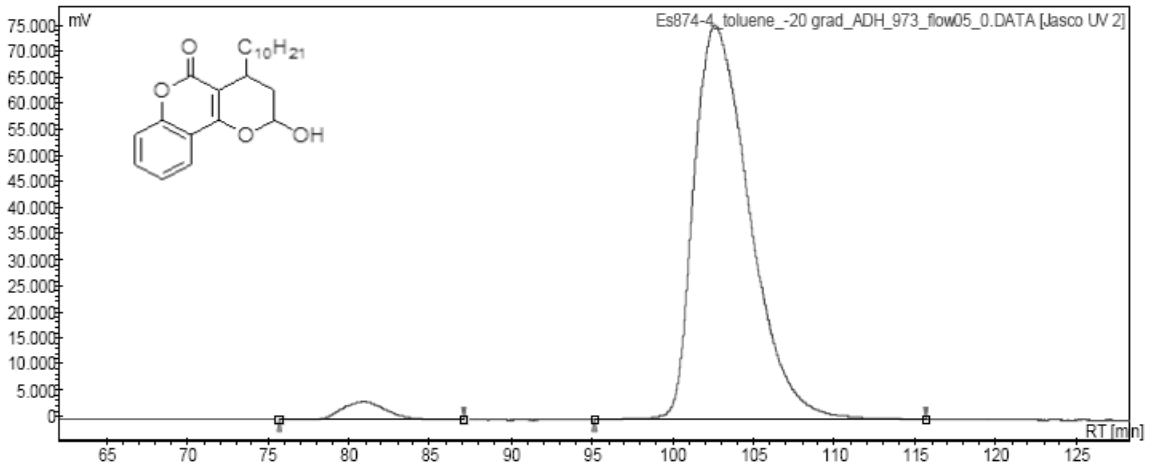


EM212rac_ADH_973_flow05_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
3	47,053	48,908	51,308	3,089
4	65,875	68,267	70,645	2,647
1	72,836	77,225	85,856	46,721
2	92,818	99,733	109,319	47,543
Total				100,000

Chromatogram : Es874-4_toluene_-20 grad_ADH_973_flow05_0

Data file: Es874-4_ADH_973_flow05_0.DATA
Method: HPLC2_ADH_973_flow05_acq140

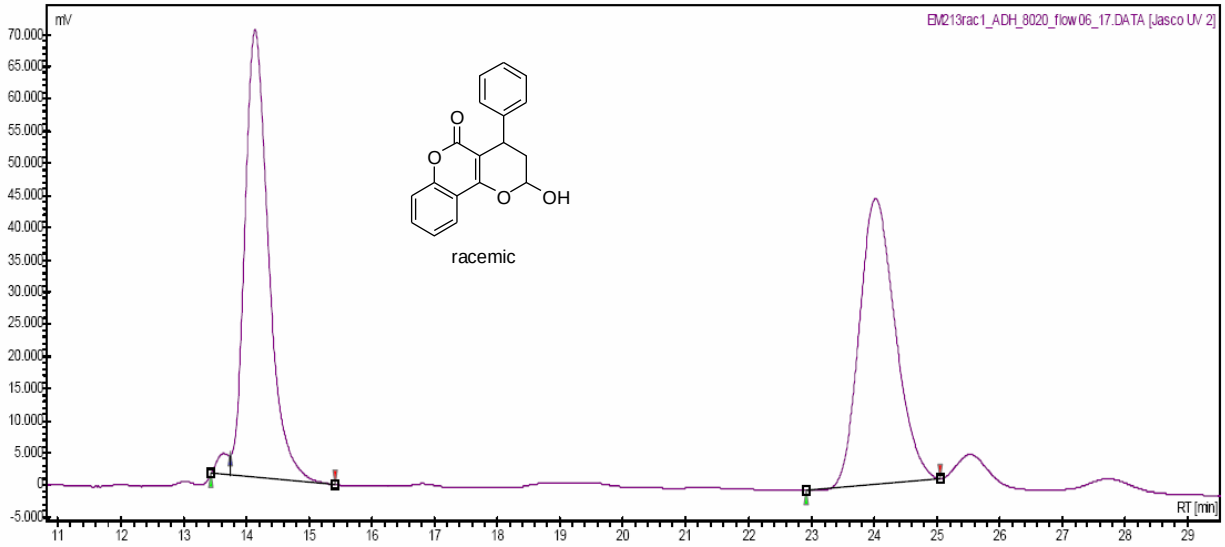


Es874-4_toluene_-20 grad_ADH_973_flow05_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	75,640	80,908	87,066	3,640
2	95,165	102,625	115,702	96,360
Total				100,000

Chromatogram : EM213rac1_ADH_8020_flow06_17

Data file: EM213rac1_ADH_8020_flow06_17.DATA
Method: HPLC2_ADH_8020_flow06_acq50

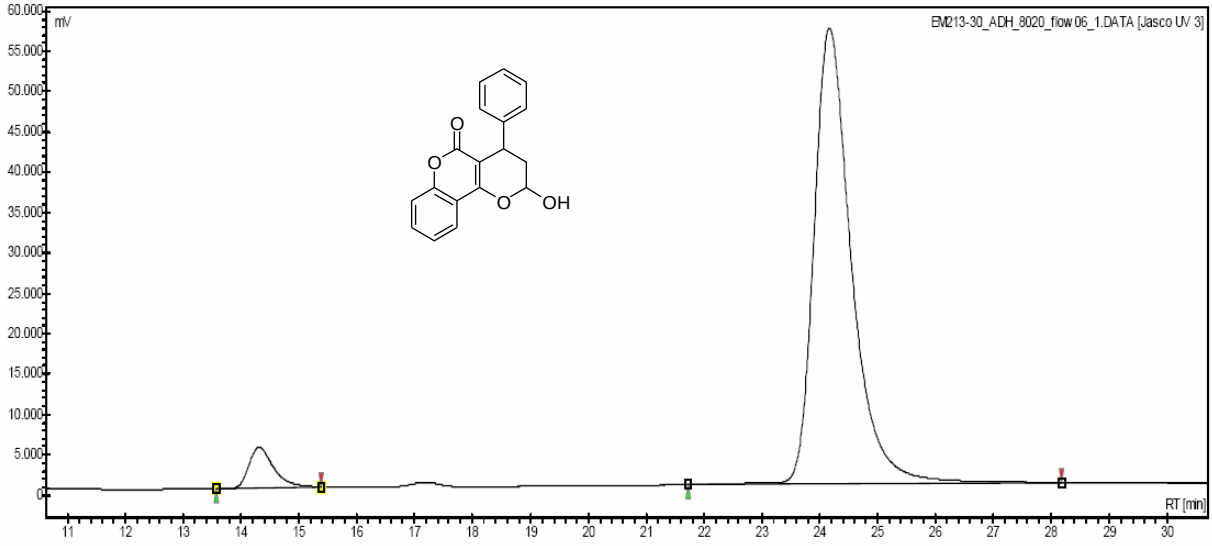


EM213rac1_ADH_8020_flow06_17.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,444	13,642	13,746	1,216
3	13,746	14,142	15,424	50,320
2	22,928	24,025	25,046	48,464
Total				100,000

Chromatogram : EM213-30_ADH_8020_flow06_1

Data file: EM213-30_ADH_8020_flow06_1.DATA
Method: HPLC2_ADH_8020_flow06_acq50

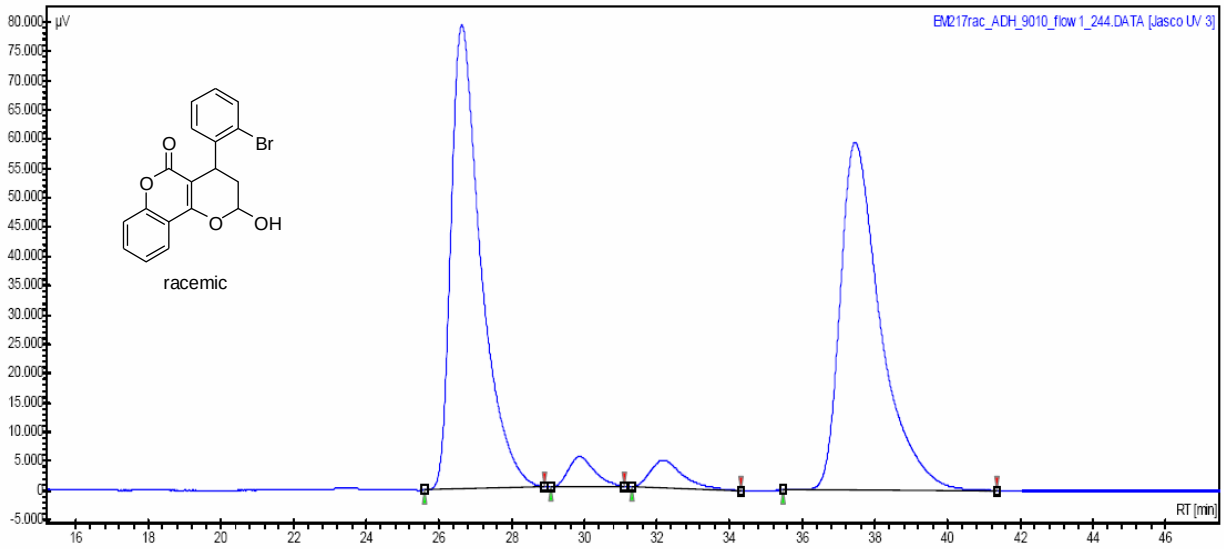


EM213-30_ADH_8020_flow06_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	13,582	14,317	15,378	5,617
2	21,731	24,167	28,177	94,383
Total				100,000

Chromatogram : EM217rac_ADH_9010_flow1_244

Data file: EM217rac_ADH_9010_flow1_244.DATA
Method: HPLC1_ADH_9010_flow1_acq_60

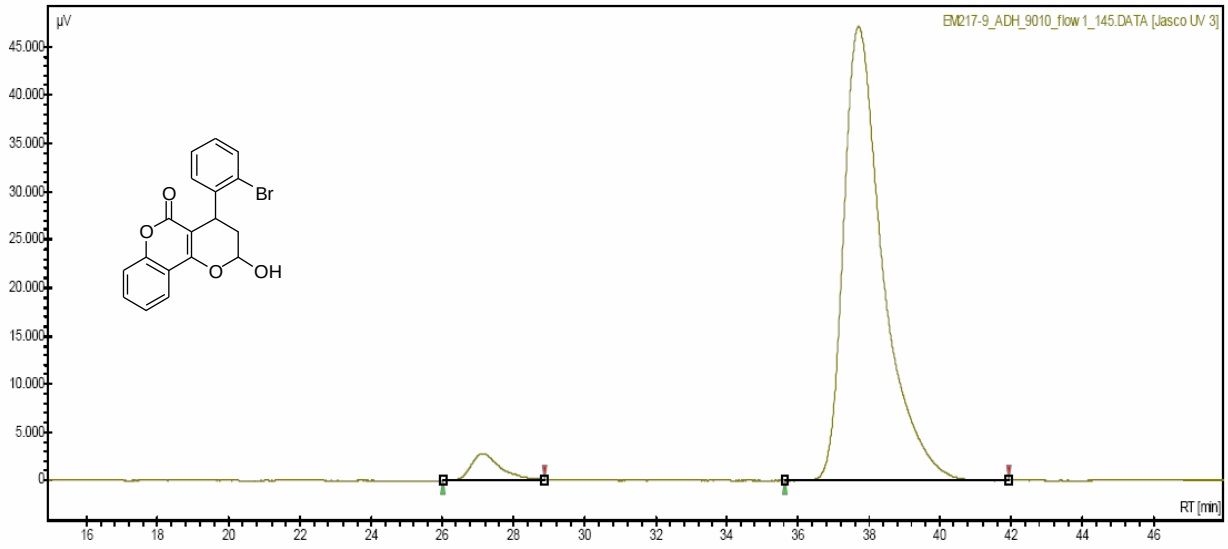


EM217rac_ADH_9010_flow1_244.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	25,606	26,633	28,913	46,953
2	29,073	29,875	31,100	2,659
3	31,314	32,200	34,301	2,981
4	35,475	37,458	41,343	47,407
Total				100,000

Chromatogram : EM217-9_ADH_9010_flow1_145

Data file: EM217-9_ADH_9010_flow1_145.DATA
Method: HPLC1_ADH_9010_flow1_acq_60

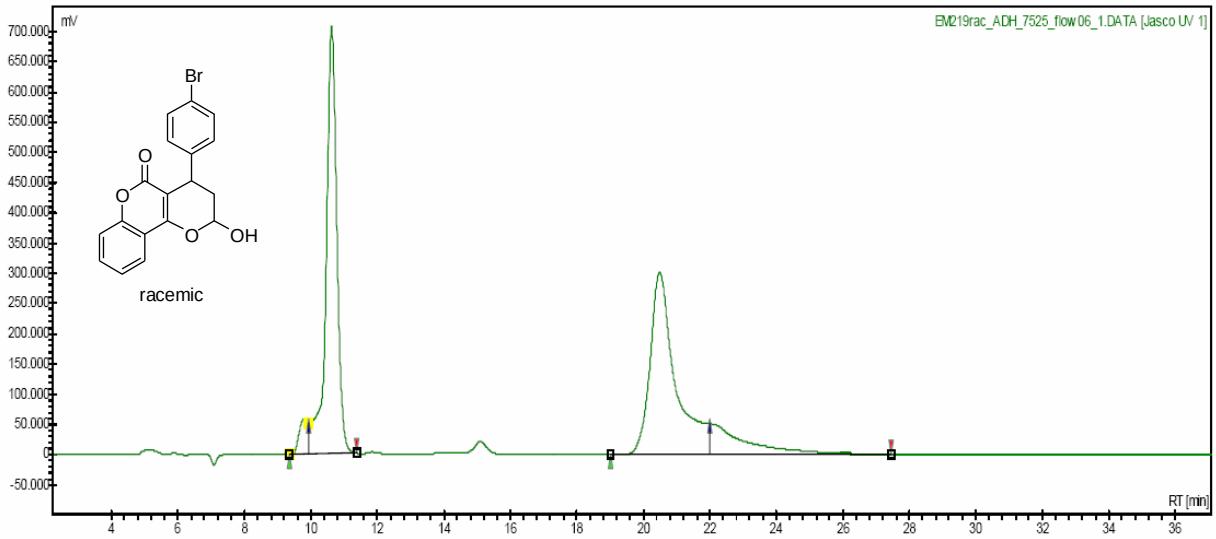


EM217-9_ADH_9010_flow1_145.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	26,022	27,150	28,895	4,152
2	35,635	37,708	41,934	95,848
Total				100,000

Chromatogram : EM219rac_ADH_7525_flow06_1

Data file: EM219rac_ADH_7525_flow06_1.DATA
Method: HPLC2_ADH_7525_flow06_acq45

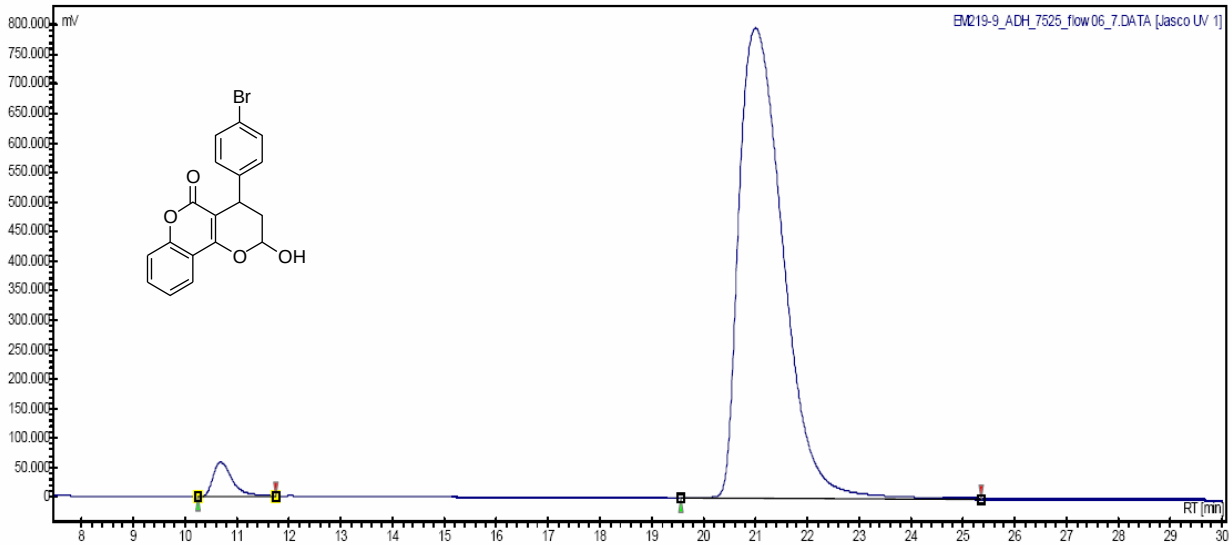


EM219rac_ADH_7525_flow06_1.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,365	9,792	9,945	2,966
3	9,945	10,625	11,395	43,554
2	19,019	20,500	22,003	43,430
4	22,003	22,003	27,472	10,050
Total				100,000

Chromatogram : EM219-9_ADH_7525_flow06_7

Data file: EM219-9_ADH_7525_flow06_7.DATA
Method: HPLC2_ADH_7525_flow06_acq30

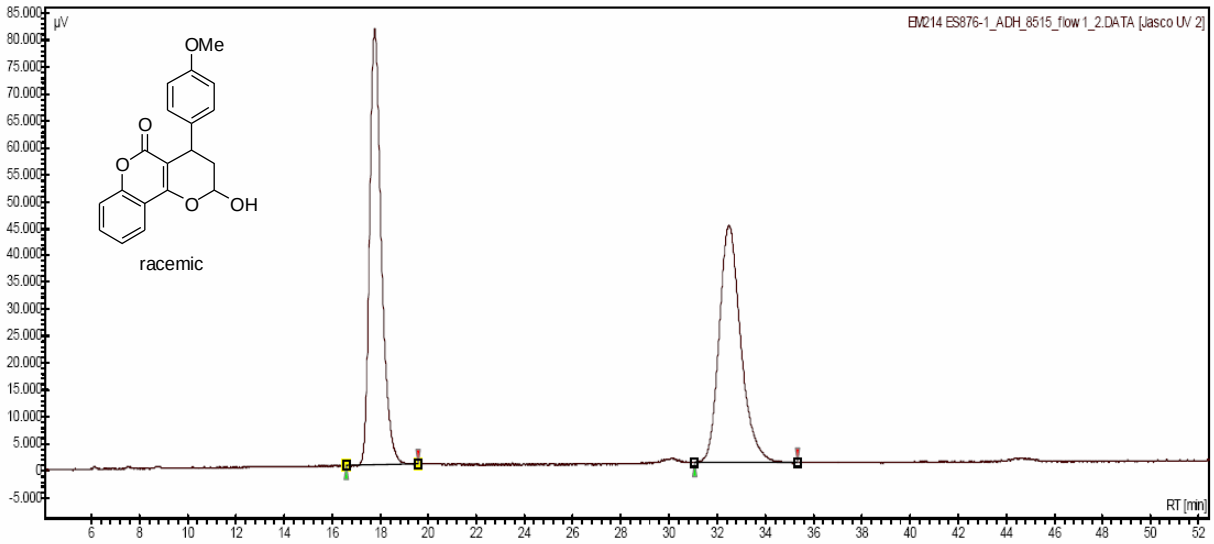


EM219-9_ADH_7525_flow06_7.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,249	10,683	11,740	3,113
2	19,558	21,000	25,359	96,887
Total				100,000

Chromatogram : ES876-1_ADH_8515_flow1_2

Data file: ES876-1_ADH_8515_flow1_2.DATA
Method: HPLC1_ADH_8515_flow1_acq_60

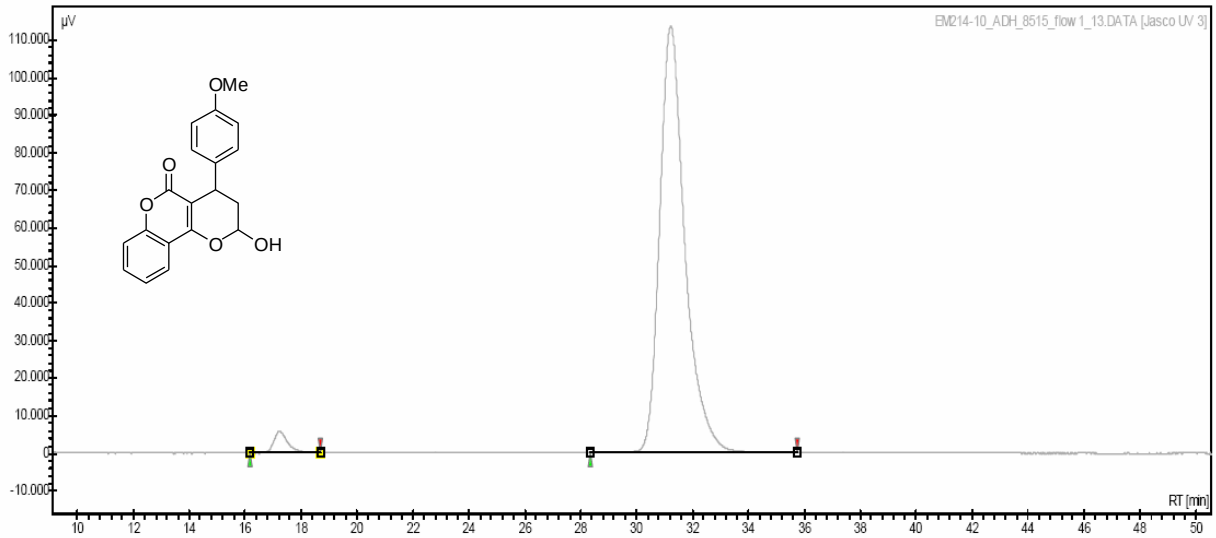


EM214 ES876-1_ADH_8515_flow1_2.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,612	17,775	19,587	50,051
2	31,054	32,483	35,331	49,949
Total				100,000

Chromatogram : EM214-10_ADH_8515_flow1_13

Data file: EM214-10_ADH_8515_flow1_13.DATA
Method: HPLC1_ADH_8515_flow1_acq_60

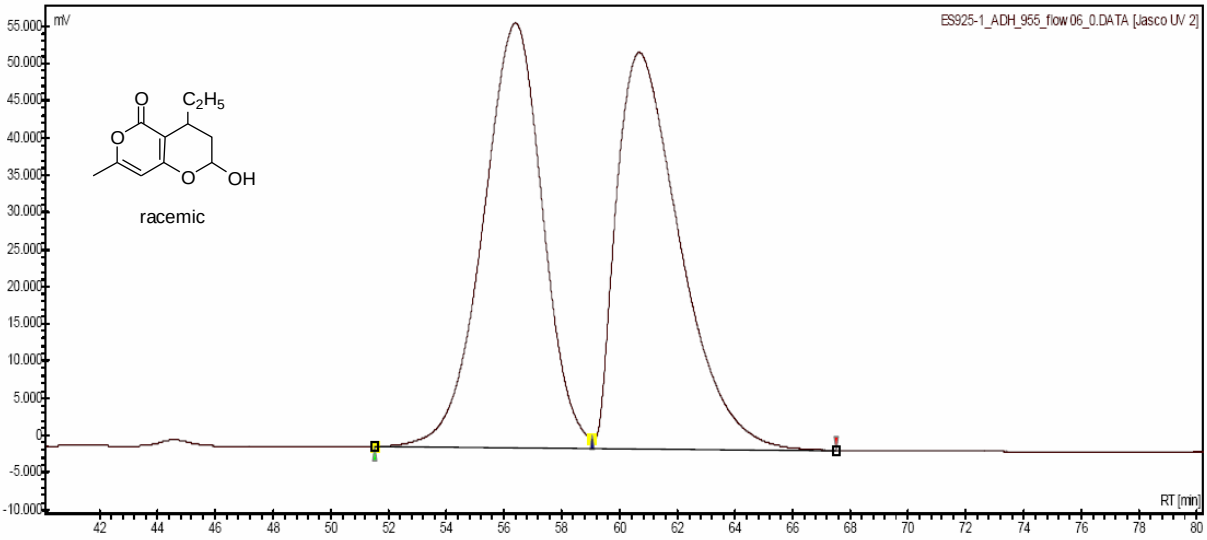


EM214-10_ADH_8515_flow1_13.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	16,188	17,242	18,674	2,775
2	28,343	31,225	35,746	97,225
Total				100,000

Chromatogram : ES925-1_ADH_955_flow06_0

Data file: ES925-1_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

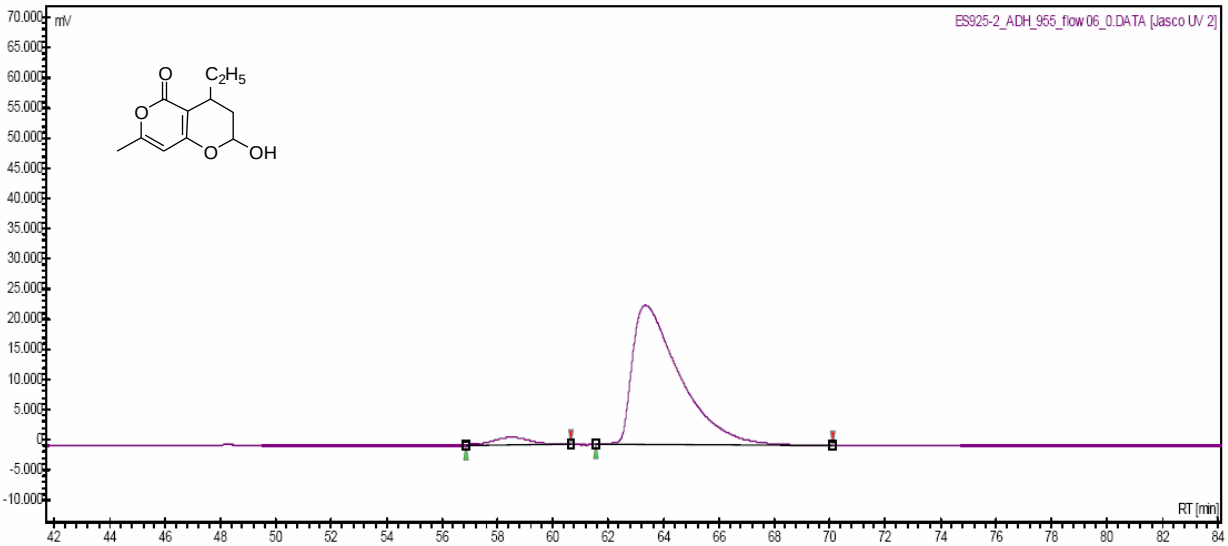


ES925-1_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	51,508	56,400	59,039	50,115
2	59,039	60,692	67,500	49,885
Total				100,000

Chromatogram : ES925-2_ADH_955_flow06_0

Data file: ES925-2_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

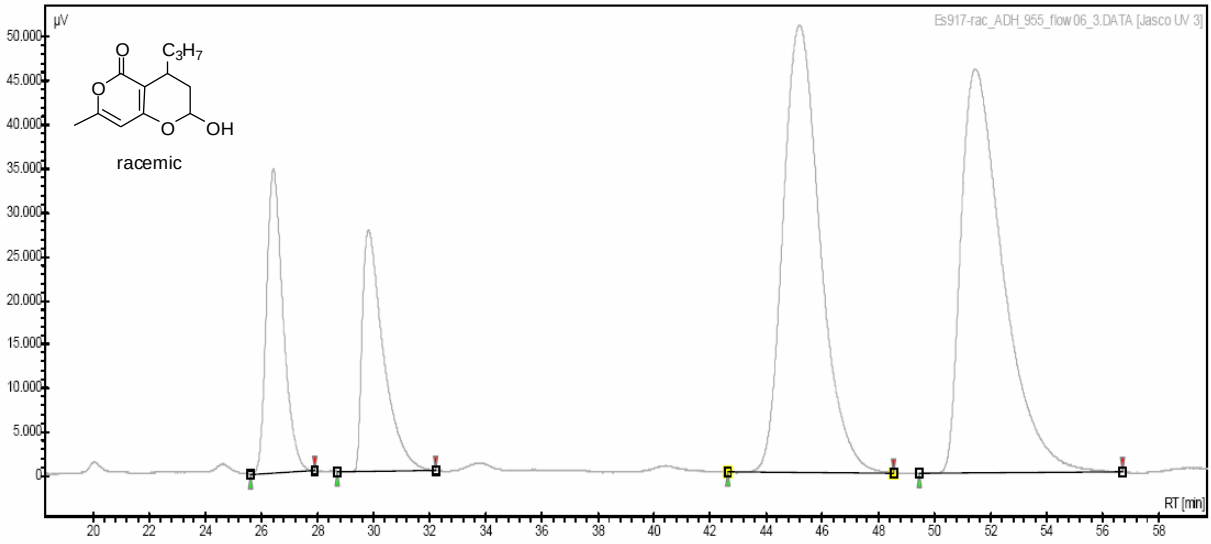


ES925-2_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
2	56,867	58,500	60,655	3,929
1	61,550	63,342	70,103	96,071
Total				100,000

Chromatogram : Es917-rac_ADH_955_flow06_3

Data file: Es917-rac_ADH_955_flow06_3.DATA
Method: HPLC1_ADH_955_flow06_acq_60

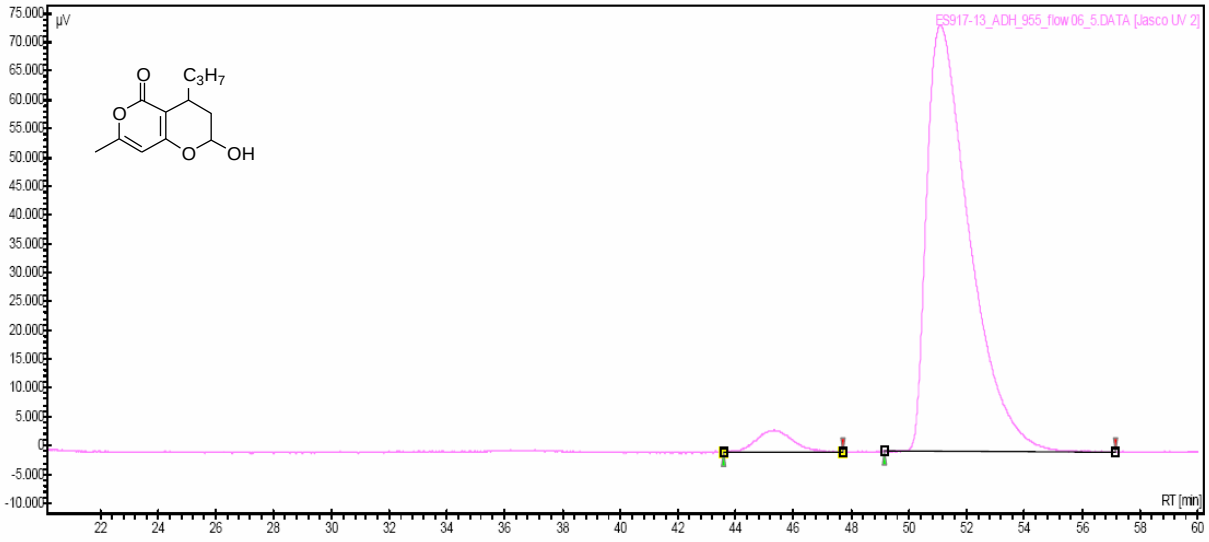


Es917-rac_ADH_955_flow06_3.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
3	25,599	26,425	27,893	11,607
4	28,698	29,817	32,231	11,647
1	42,645	45,192	48,533	37,906
2	49,463	51,450	56,715	38,840
Total				100,000

Chromatogram : ES917-13_ADH_955_flow06_5

Data file: ES917-13_ADH_955_flow06_5.DATA
Method: HPLC1_ADH_955_flow06_acq_60

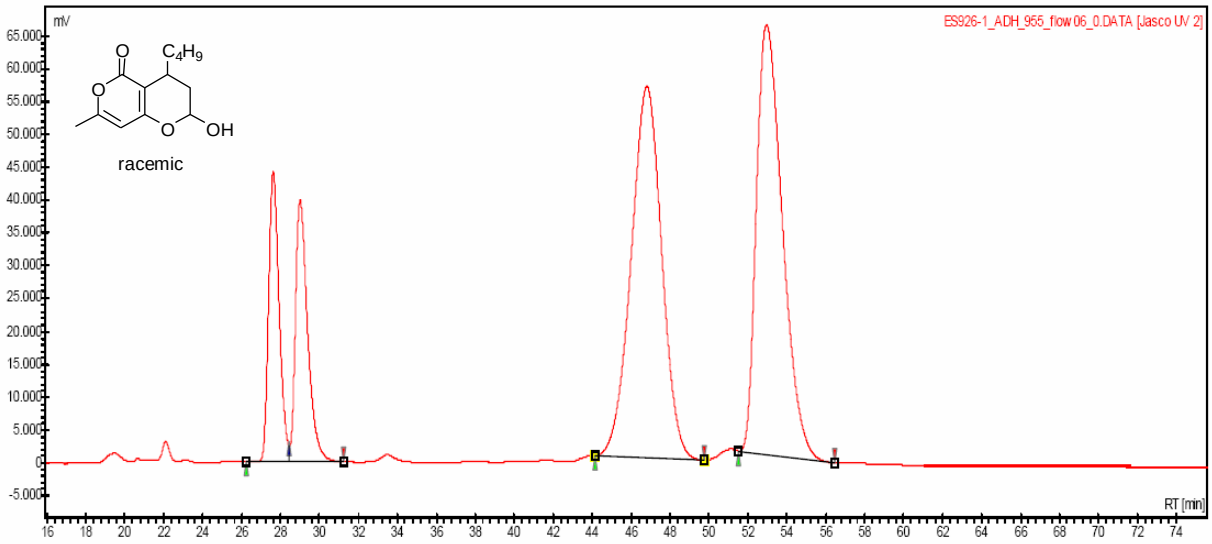


ES917-13_ADH_955_flow06_5.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	43,574	45,333	47,727	4,116
2	49,153	51,092	57,149	95,884
Total				100,000

Chromatogram : ES926-1_ADH_955_flow06_0

Data file: ES926-1_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

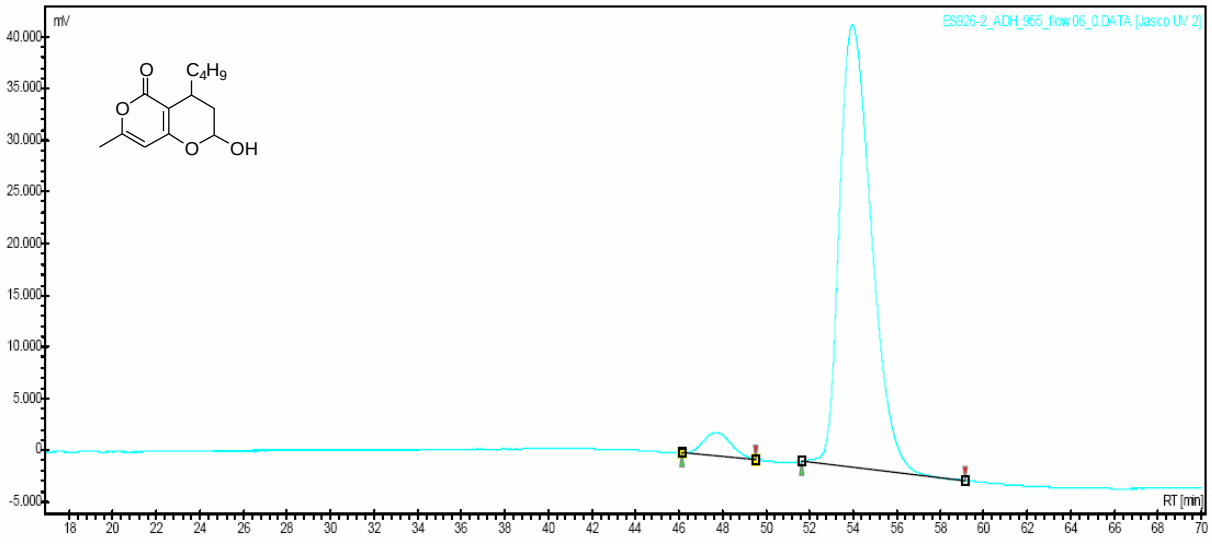


ES926-1_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
3	26,219	27,625	28,450	10,573
4	28,450	29,008	31,240	11,083
1	44,163	46,817	49,742	39,454
2	51,508	52,967	56,436	38,889
Total				100,000

Chromatogram : ES926-2_ADH_955_flow06_0

Data file: ES926-2_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq70

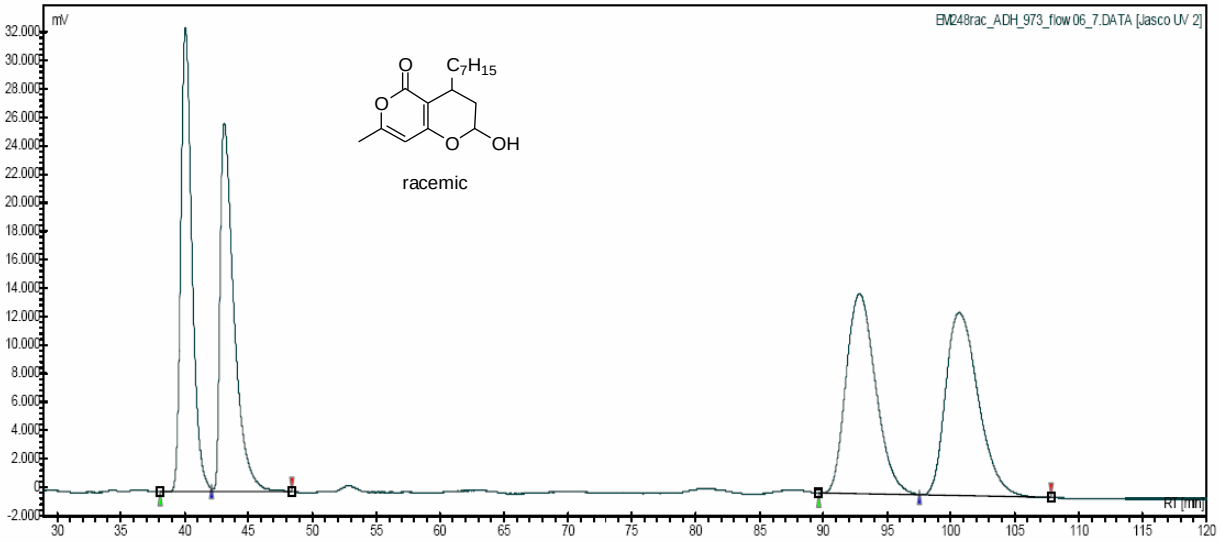


ES926-2_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	46,136	47,742	49,535	4,426
2	51,632	53,975	59,153	95,574
Total				100,000

Chromatogram : EM248rac_ADH_973_flow06_7

Data file: EM248rac_ADH_973_flow06_7.DATA
Method: HPLC2_ADH_973_flow06_acq120

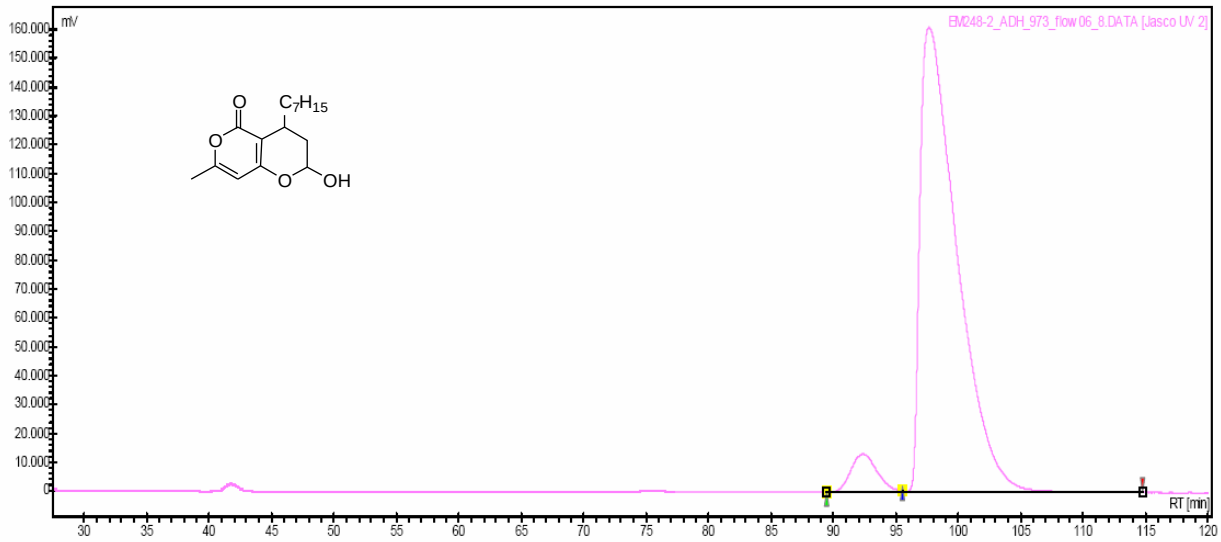


EM248rac_ADH_973_flow06_7.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	38,122	40,075	42,099	23,178
2	42,099	43,117	48,398	23,062
3	89,613	92,850	97,569	26,782
4	97,569	100,642	107,845	26,978
Total				100,000

Chromatogram : EM248-2_ADH_973_flow06_8

Data file: EM248-2_ADH_973_flow06_8.DATA
Method: HPLC2_ADH_973_flow06_acq120

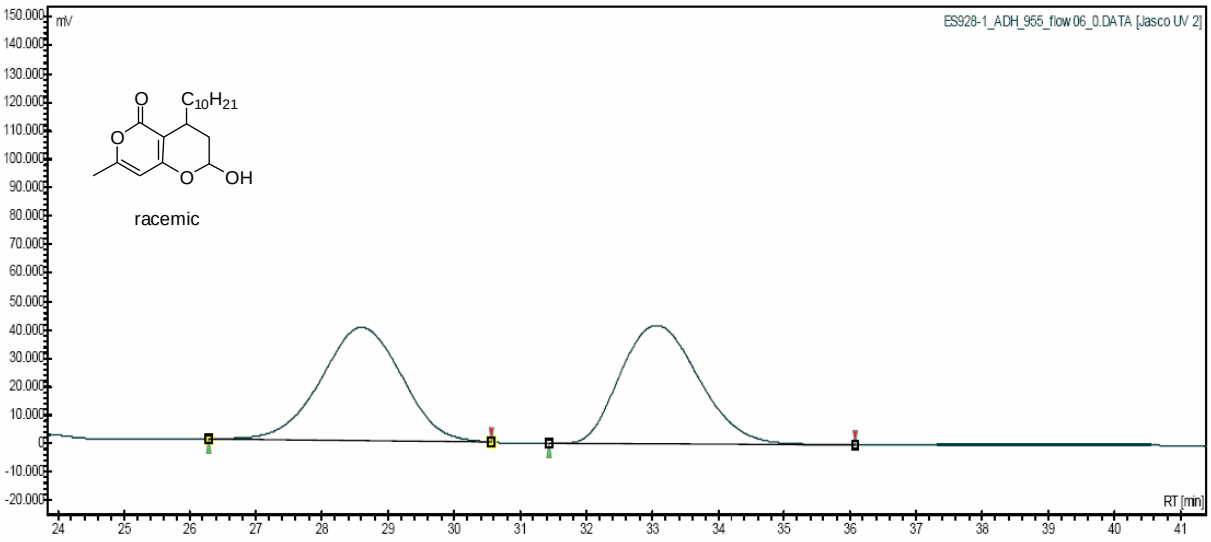


EM248-2_ADH_973_flow06_8.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	89,503	92,408	95,580	5,589
2	95,580	97,692	114,807	94,411
Total				100,000

Chromatogram : ES928-1_ADH_955_flow06_0

Data file: ES928-1_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq90

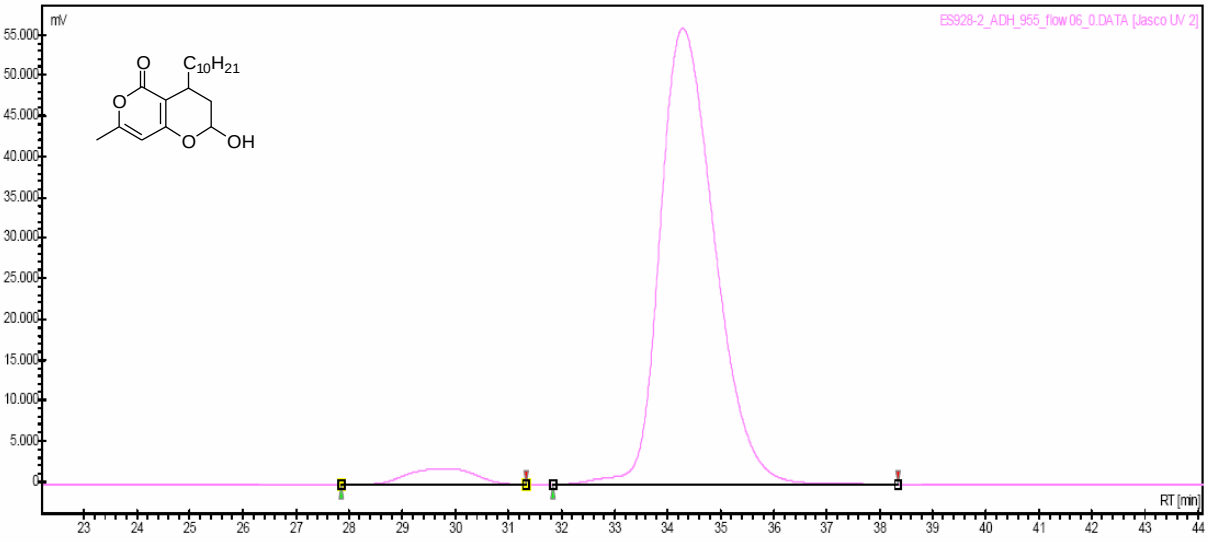


ES928-1_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	26,286	28,600	30,564	49,603
2	31,440	33,067	36,079	50,397
Total				100,000

Chromatogram : ES928-2_ADH_955_flow06_0

Data file: ES928-2_ADH_955_flow06_0.DATA
Method: HPLC2_ADH_955_flow06_acq45

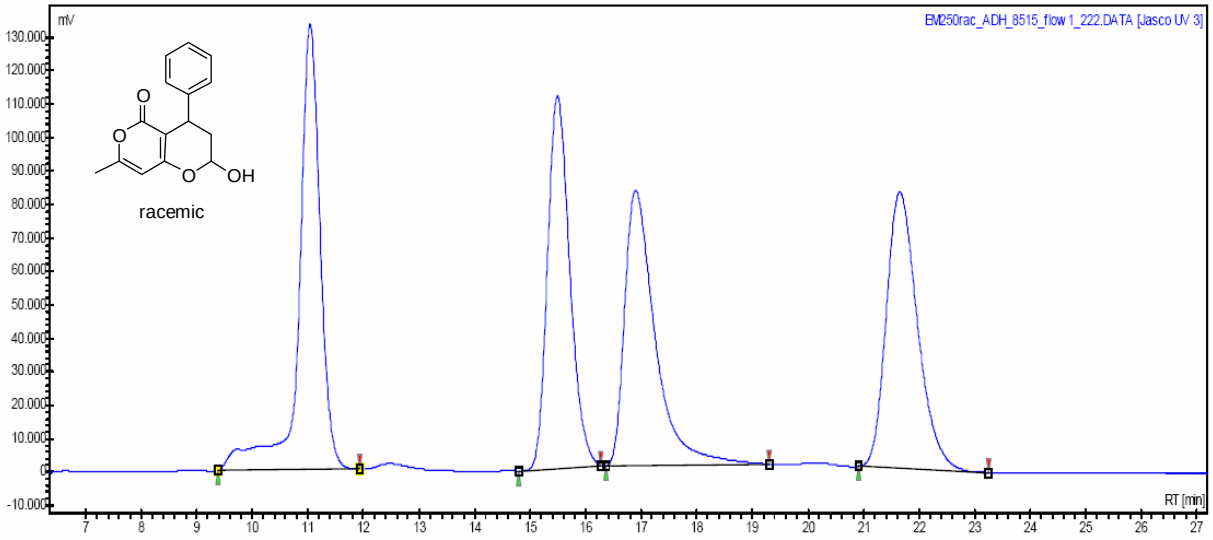


ES928-2_ADH_955_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	27,846	29,825	31,333	4,372
2	31,844	34,292	38,352	95,628
Total				100,000

Chromatogram : EM250rac_ADH_8515_flow1_222

Data file: EM250rac_ADH_8515_flow1_222.DATA
Method: HPLC2_ADH_8515_flow1_acq30

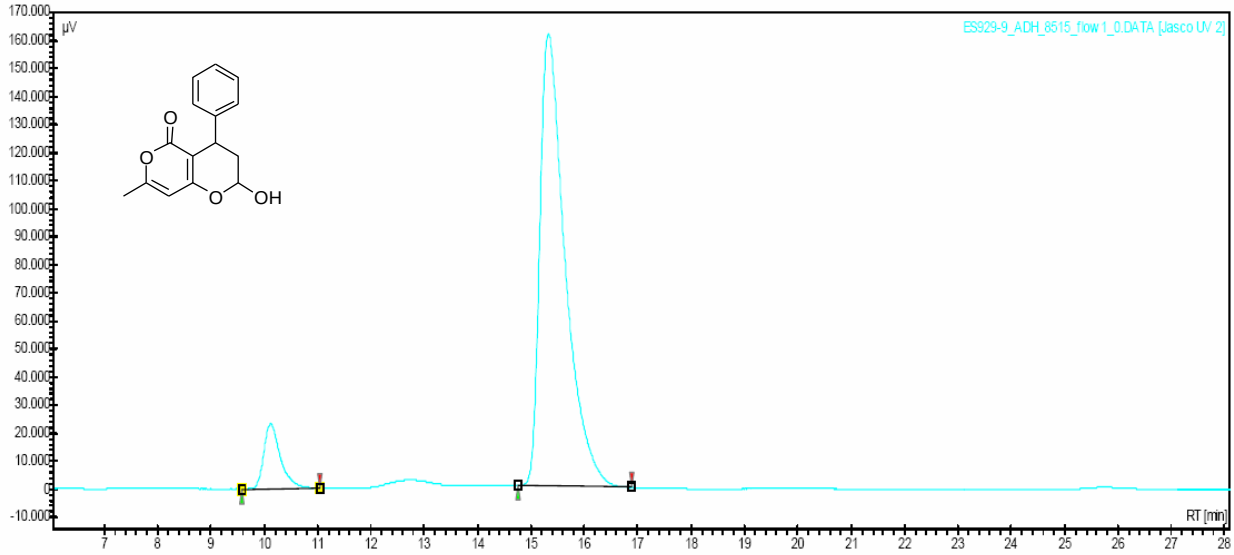


EM250rac_ADH_8515_flow1_222.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,390	11,042	11,932	27,501
2	14,783	15,492	16,271	23,705
3	16,364	16,900	19,308	24,069
4	20,919	21,650	23,244	24,725
Total				100,000

Chromatogram : ES929-9_ADH_8515_flow1_0

Data file: ES929-9_ADH_8515_flow1_0.DATA
Method: HPLC1_ADH_8515_flow1_acq_30

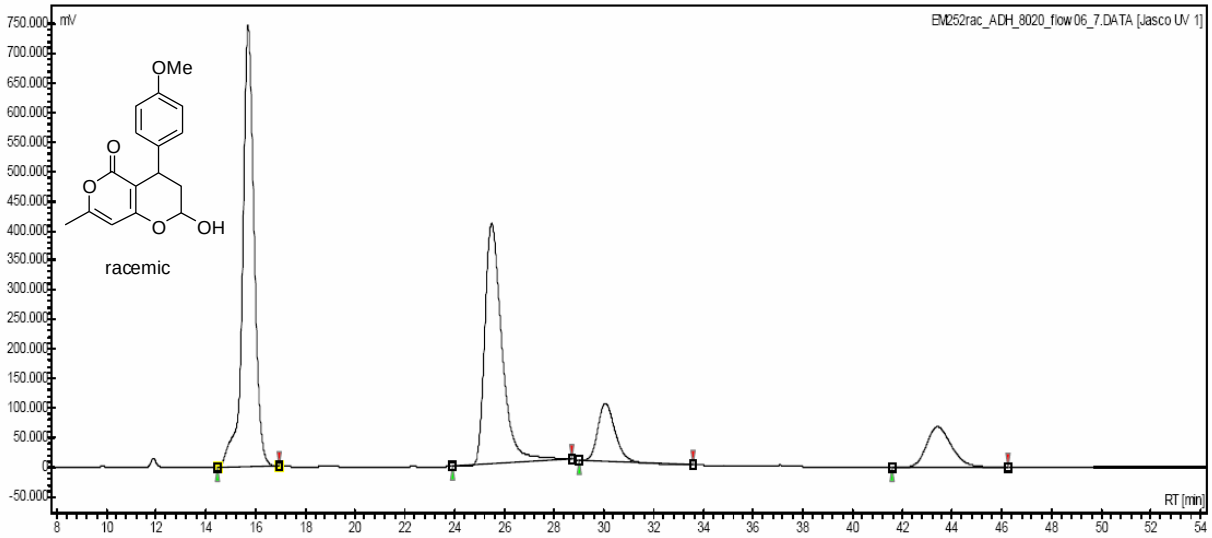


ES929-9_ADH_8515_flow1_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	9,576	10,125	11,033	8,581
2	14,752	15,325	16,890	91,419
Total				100,000

Chromatogram : EM252rac_ADH_8020_flow06_7

Data file: EM252rac_ADH_8020_flow06_7.DATA
Method: HPLC2_ADH_8020_flow06_acq60

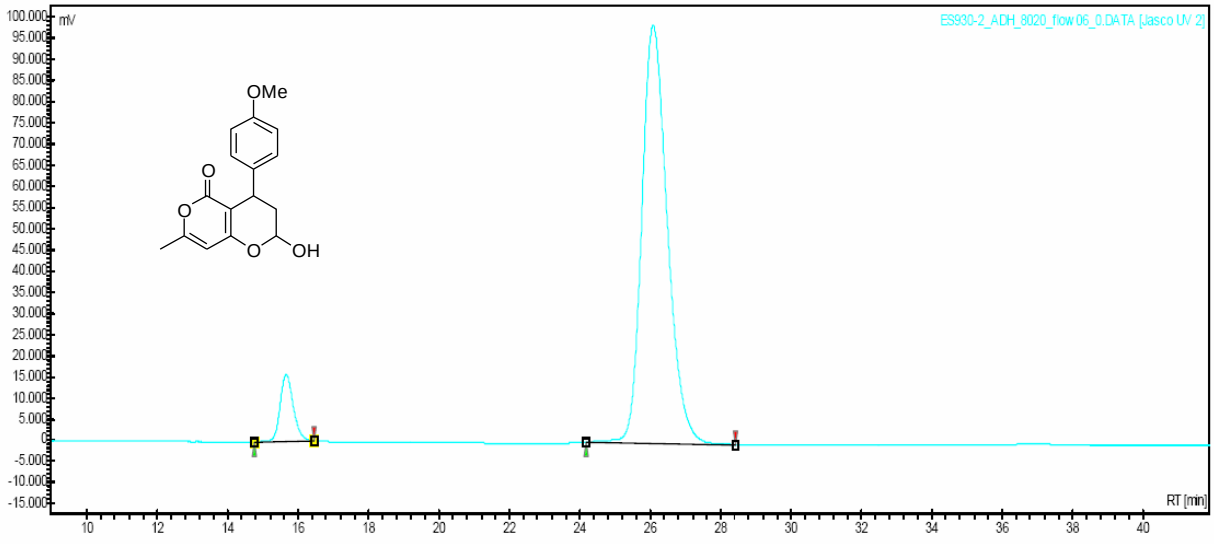


EM252rac_ADH_8020_flow06_7.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	14,475	15,700	16,961	45,317
2	23,923	25,492	28,729	36,931
3	29,006	30,067	33,591	8,662
4	41,602	43,425	46,243	9,091
Total				100,000

Chromatogram : ES930-2_ADH_8020_flow06_0

Data file: ES930-2_ADH_8020_flow06_0.DATA
Method: HPLC2_ADH_8020_flow06_acq90

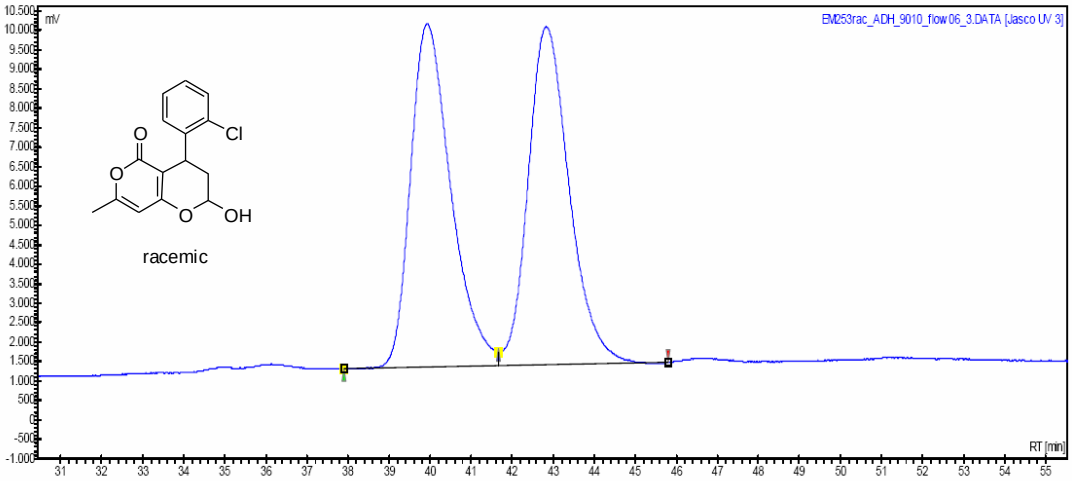


ES930-2_ADH_8020_flow06_0.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	14,750	15,667	16,468	7,648
2	24,169	26,083	28,434	92,352
Total				100,000

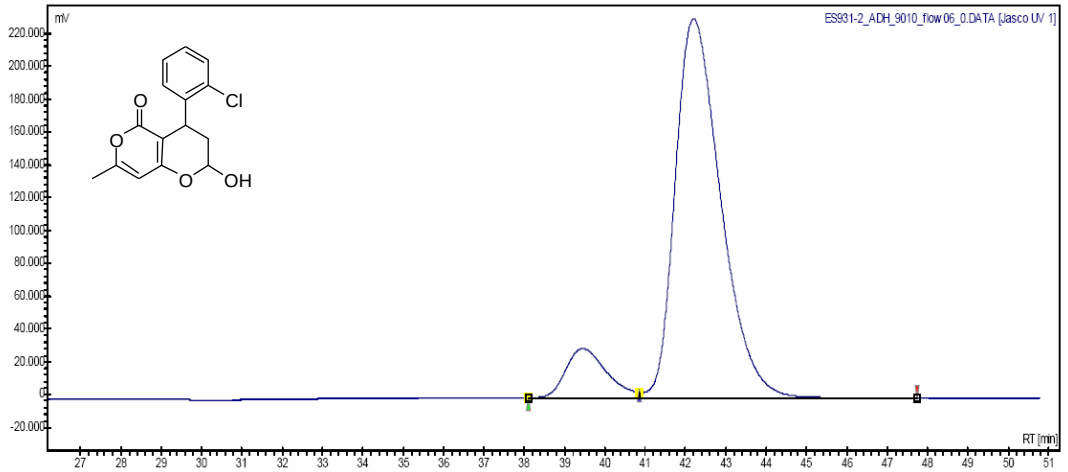
Chromatogram : EM253rac_ADH_9010_flow06_3

Data file: EM253rac_ADH_9010_flow06_3.DATA
Method: HPLC2_ADH_9010_flow06_acq60



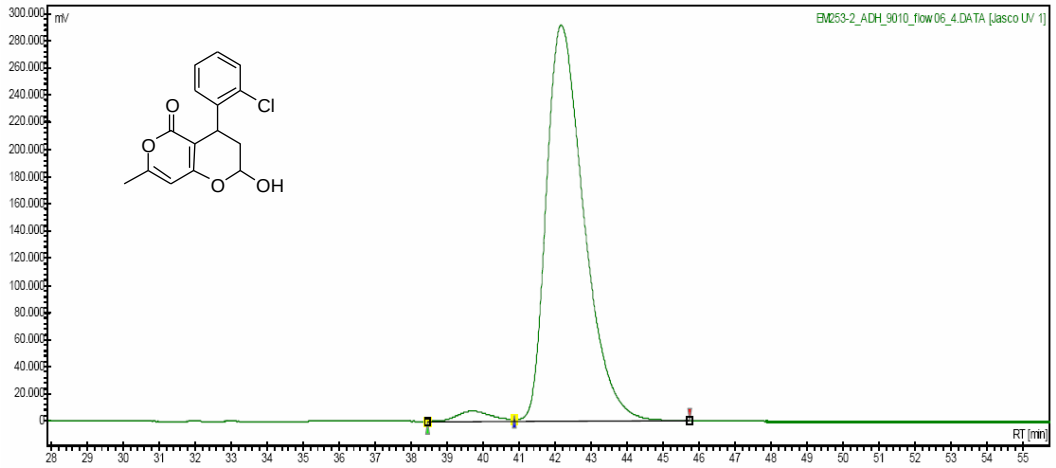
EM253rac_ADH_9010_flow06_3.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	37,901	39,933	41,657	49,816
2	41,657	42,825	45,801	50,184
Total				100,000



ES931-2_ADH_9010_flow06_0.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	38,105	39,458	40,863	10,379
2	40,863	42,200	47,736	89,621
Total				100,000

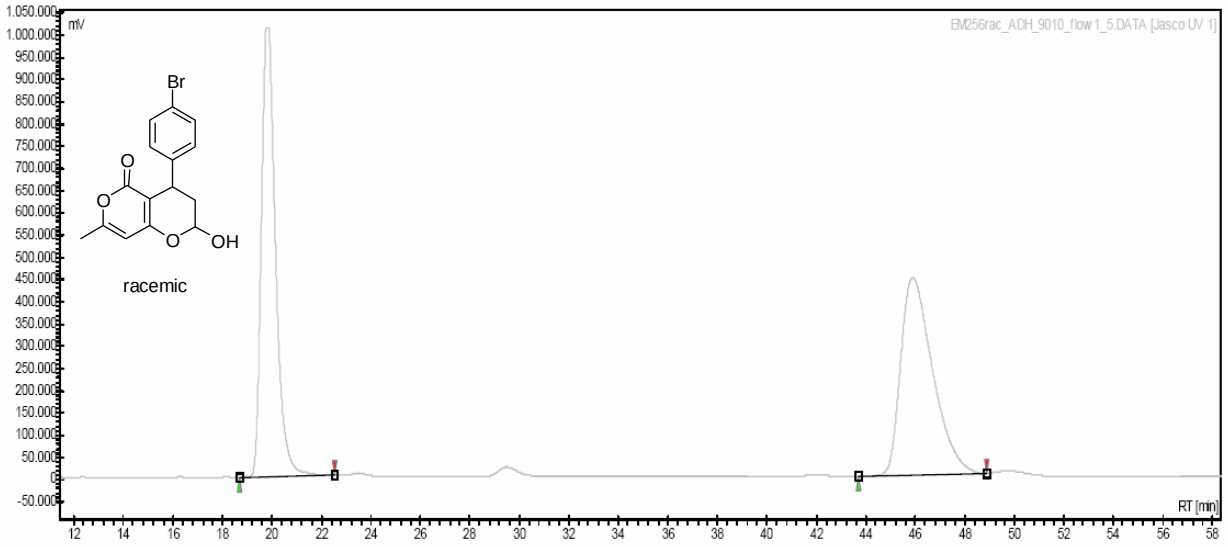


EM253-2_ADH_9010_flow06_4.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	38,453	39,708	40,884	2,276
2	40,884	42,175	45,746	97,724
Total				100,000

Chromatogram : EM256rac_ADH_9010_flow1_5

Data file: EM256rac_ADH_9010_flow1_5.DATA
Method: HPLC2_ADH_9010_flow1_acq60

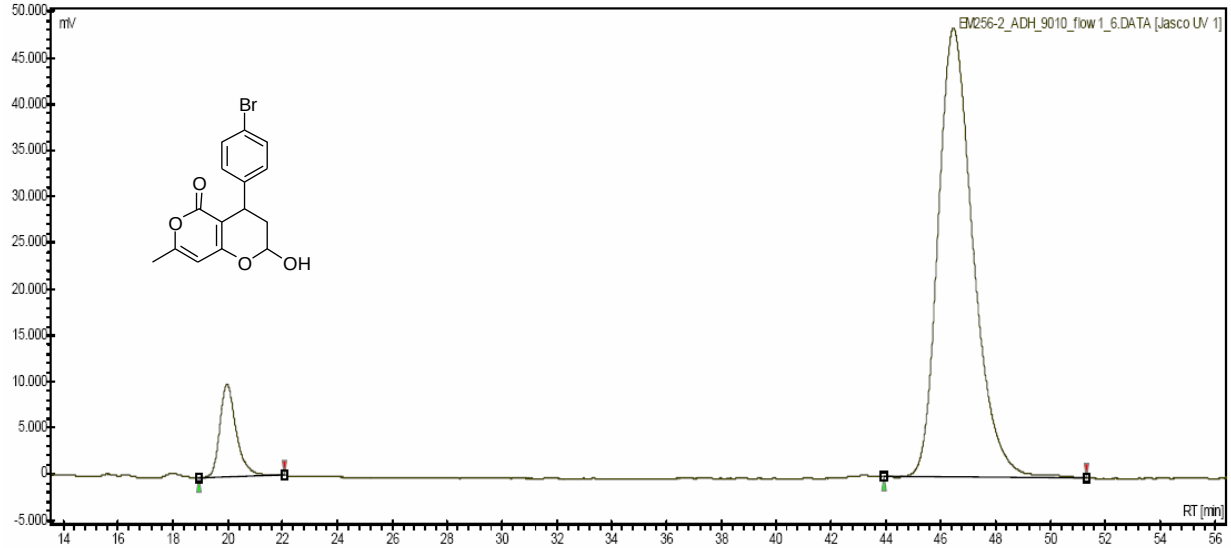


EM256rac_ADH_9010_flow1_5.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,674	19,767	22,541	50,270
2	43,702	45,892	48,895	49,730
Total				100,000

Chromatogram : EM256-2_ADH_9010_flow1_6

Data file: EM256-2_ADH_9010_flow1_6.DATA
Method: HPLC2_ADH_9010_flow1_acq60

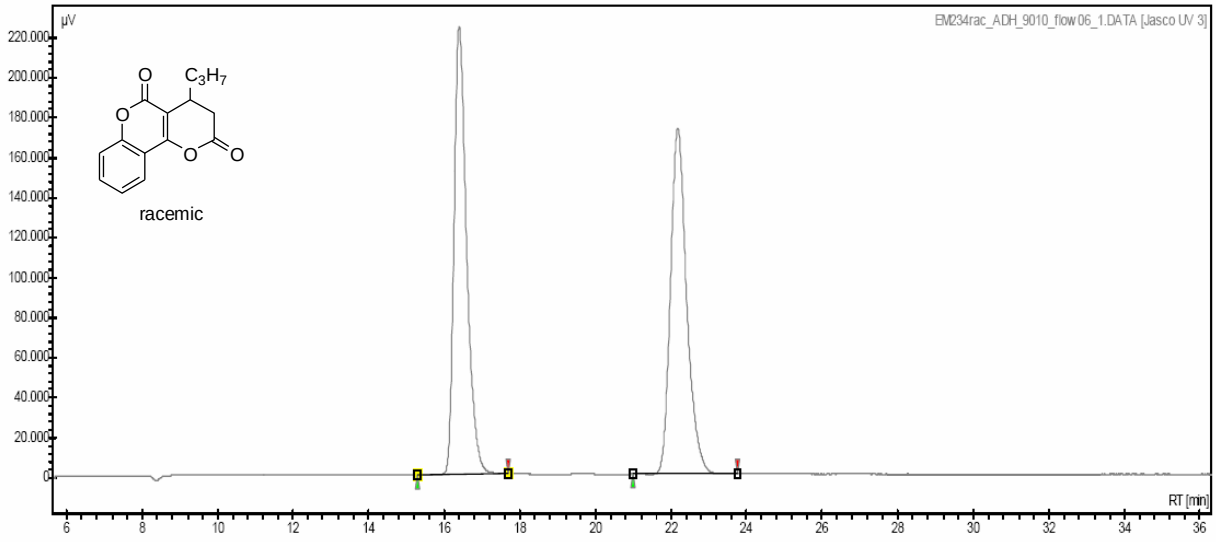


EM256-2_ADH_9010_flow1_6.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,950	19,967	22,044	8,833
2	43,923	46,467	51,326	91,167
Total				100,000

Chromatogram : EM234rac_ADH_9010_flow06_1

Data file: EM234rac_ADH_9010_flow06_1.DATA
Method: HPLC1_ADH_9010_flow06_acq_60

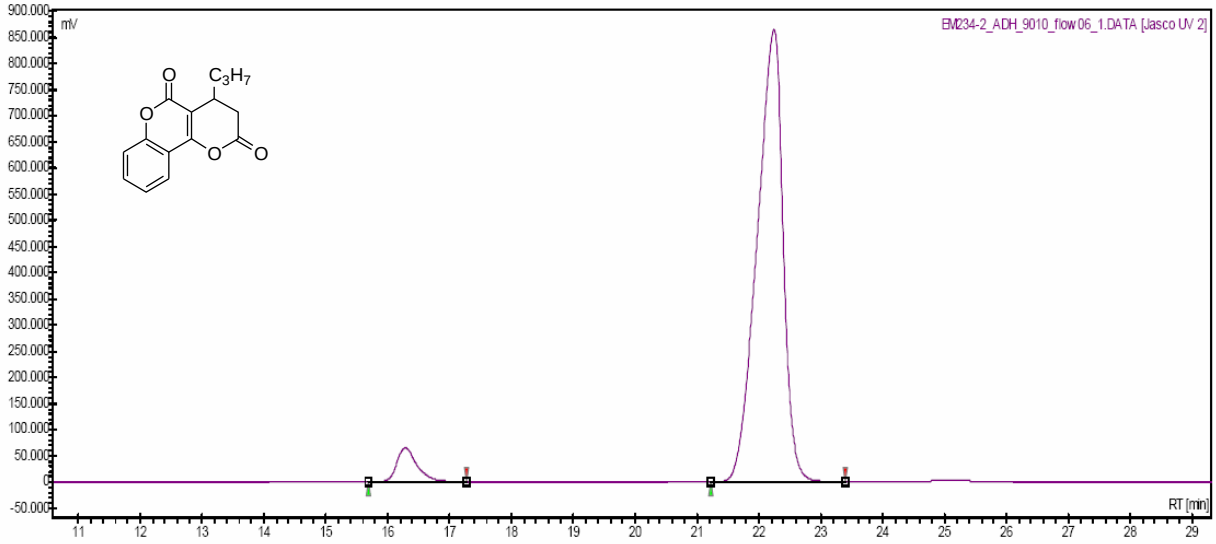


EM234rac_ADH_9010_flow06_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,304	16,400	17,680	50,152
2	20,994	22,183	23,757	49,848
Total				100,000

Chromatogram : EM234-2_ADH_9010_flow06_1

Data file: EM234-2_ADH_9010_flow06_1.DATA
Method: HPLC2_ADH_9010_flow06_acq30

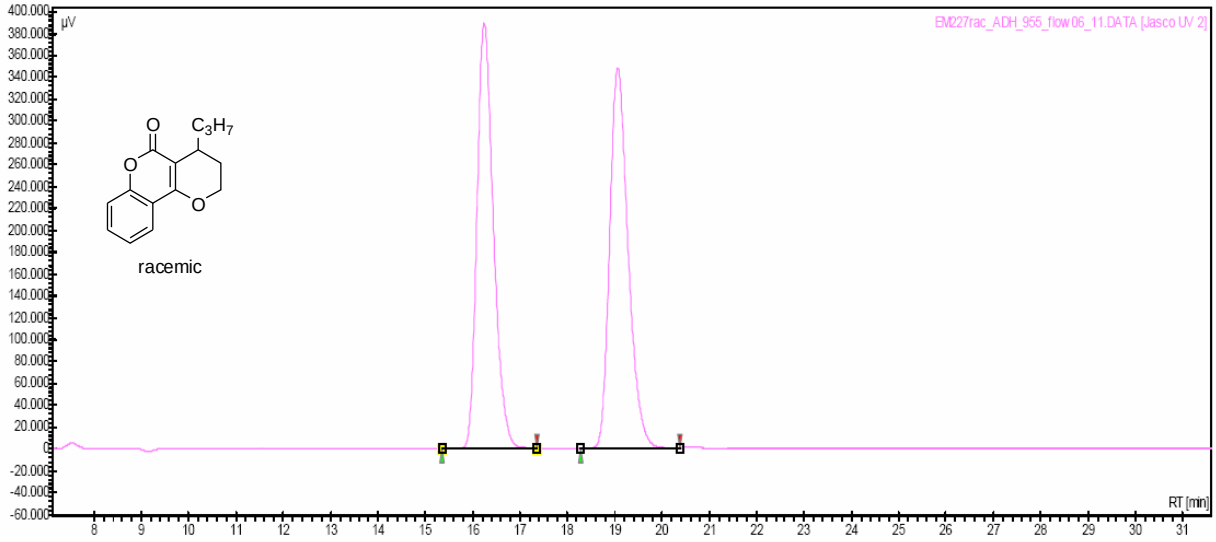


EM234-2_ADH_9010_flow06_1.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,691	16,283	17,265	5,402
2	21,215	22,242	23,398	94,598
Total				100,000

Chromatogram : EM227rac_ADH_955_flow06_11

Data file: EM227rac_ADH_955_flow06_11.DATA
Method: HPLC1_ADH_955_flow06_acq_60

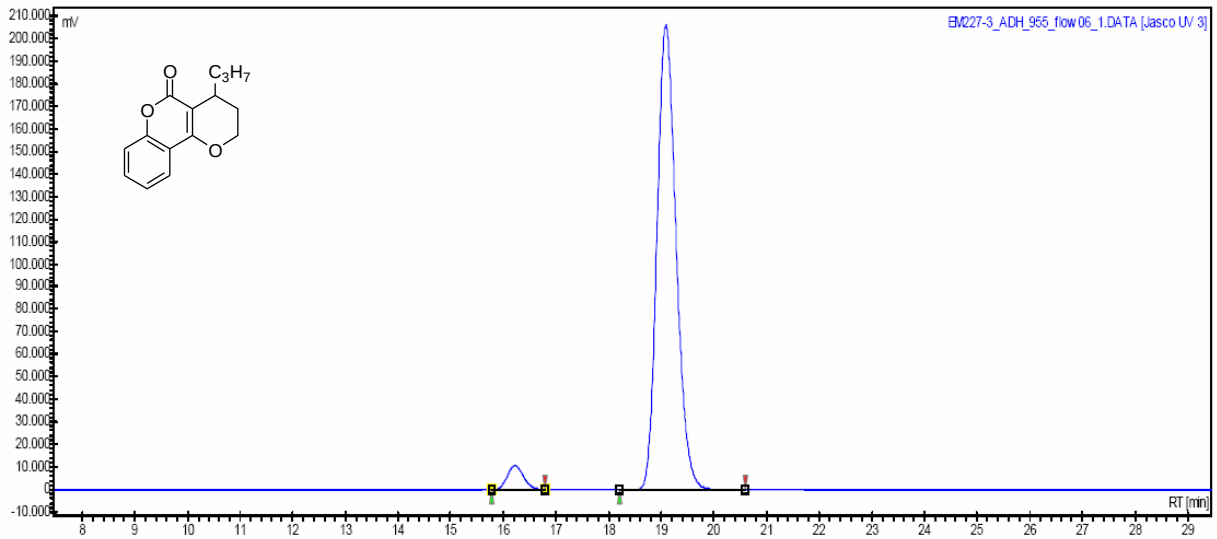


EM227rac_ADH_955_flow06_11.DATA [Jasco UV 2]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,359	16,242	17,348	49,947
2	18,287	19,067	20,387	50,053
Total				100,000

Chromatogram : EM227-3_ADH_955_flow06_1

Data file: EM227-3_ADH_955_flow06_1.DATA
Method: HPLC2_ADH_955_flow06_acq30

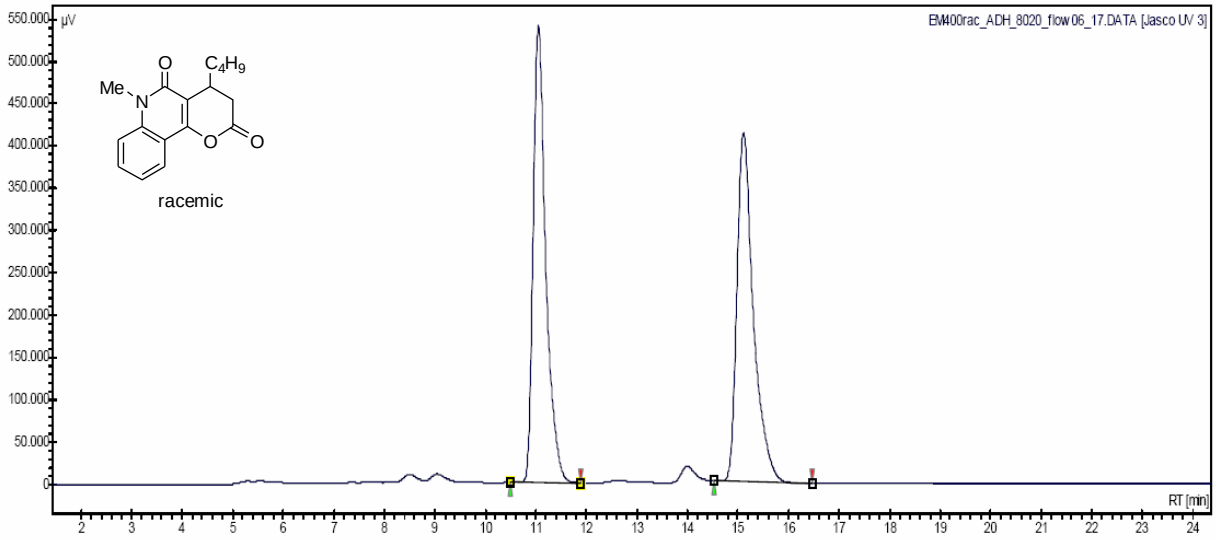


EM227-3_ADH_955_flow06_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	15,773	16,225	16,796	4,315
2	18,204	19,092	20,608	95,685
Total				100,000

Chromatogram : EM400rac_ADH_8020_flow06_17

Data file: EM400rac_ADH_8020_flow06_17.DATA
Method: HPLC1_ADH_8020_flow06_acq_60

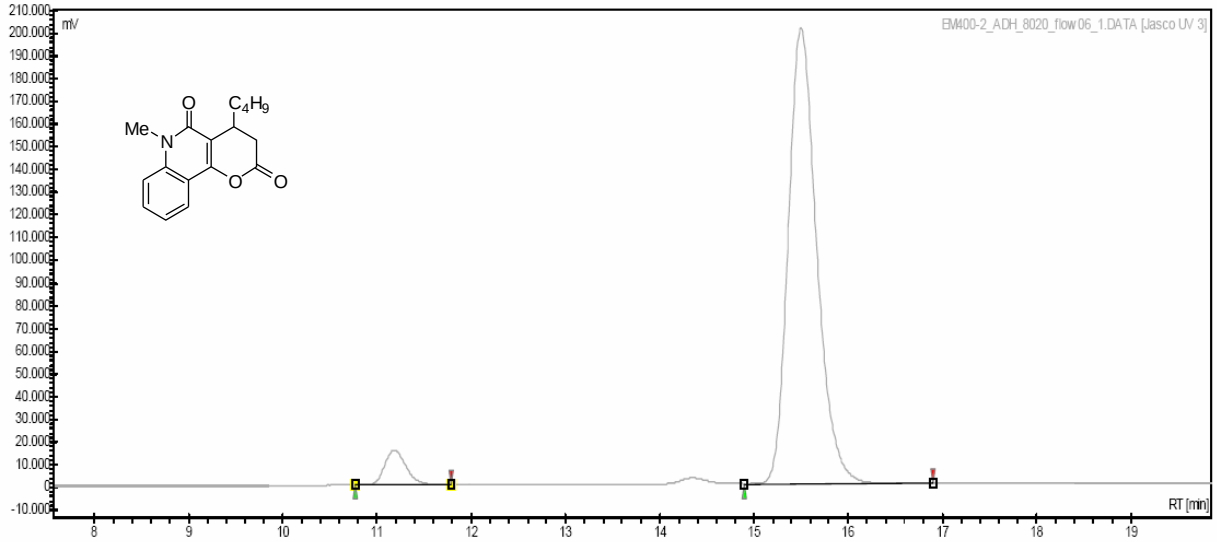


EM400rac_ADH_8020_flow06_17.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,497	11,050	11,878	49,996
2	14,530	15,108	16,464	50,004
Total				100,000

Chromatogram : EM400-2_ADH_8020_flow06_1

Data file: EM400-2_ADH_8020_flow06_1.DATA
Method: HPLC2_ADH_8020_flow06_acq20

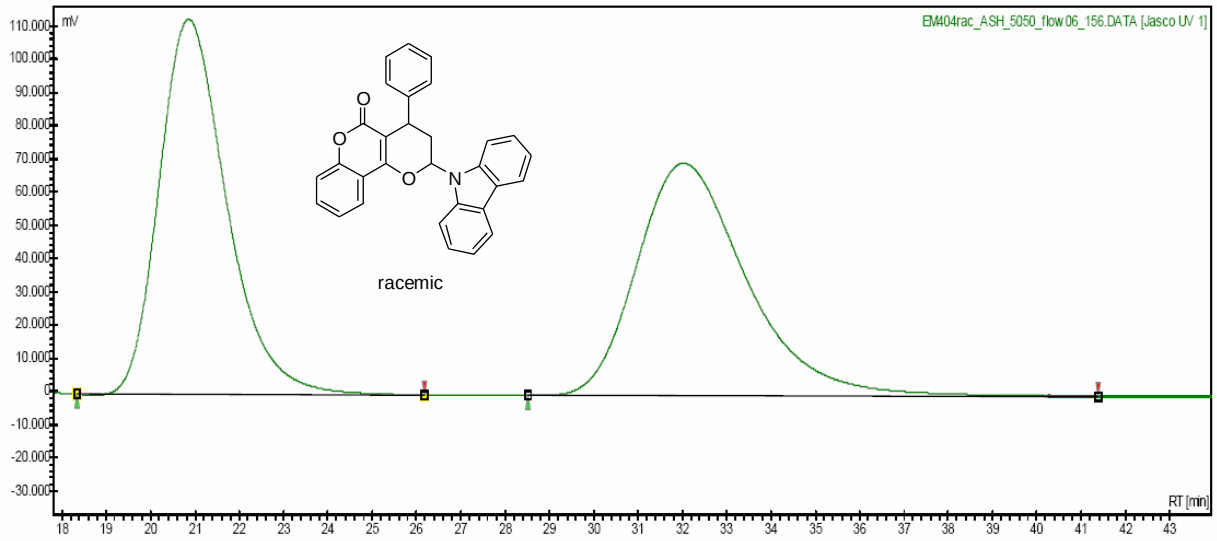


EM400-2_ADH_8020_flow06_1.DATA [Jasco UV 3]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	10,773	11,183	11,786	5,458
2	14,899	15,500	16,906	94,542
Total				100,000

Chromatogram : EM404rac_ASH_5050_flow06_156

Data file: EM404rac_ASH_5050_flow06_156.DATA
Method: HPLC2_ASH_5050_flow06_acq60

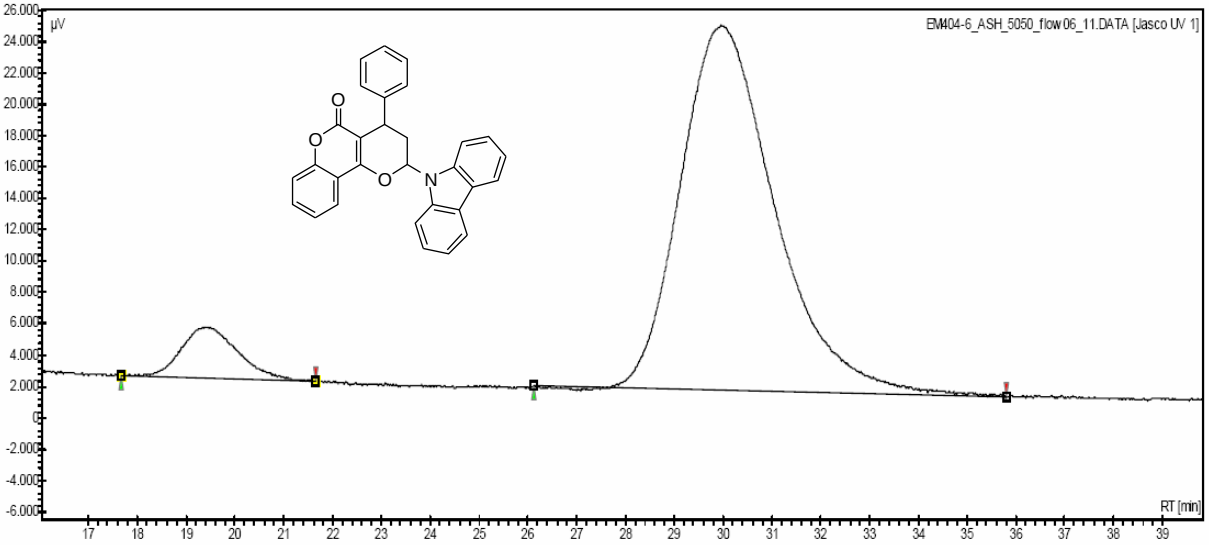


EM404rac_ASH_5050_flow06_156.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	18,343	20,858	26,188	49,749
2	28,508	32,025	41,381	50,251
Total				100,000

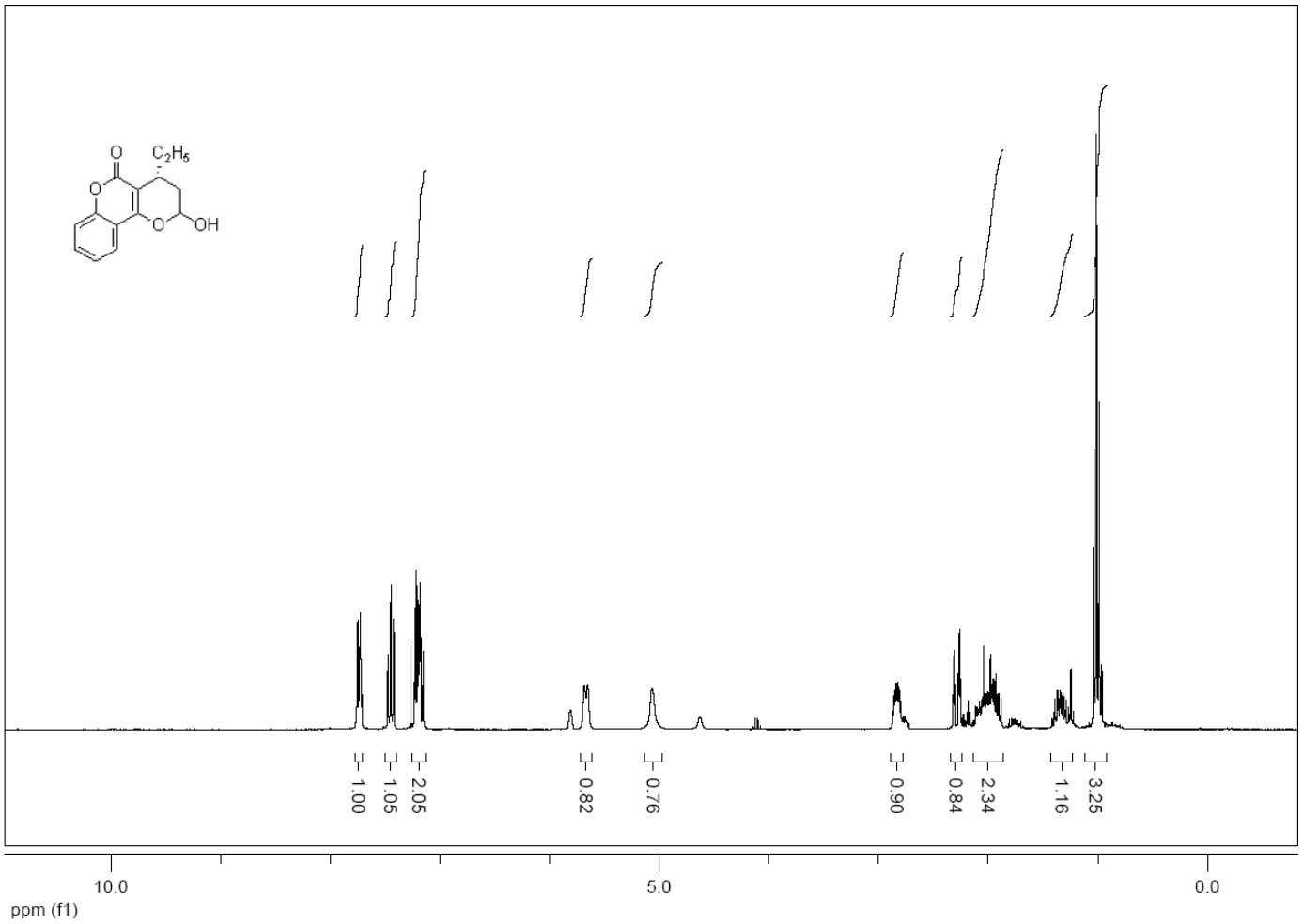
Chromatogram : EM404-6_ASH_5050_flow06_11

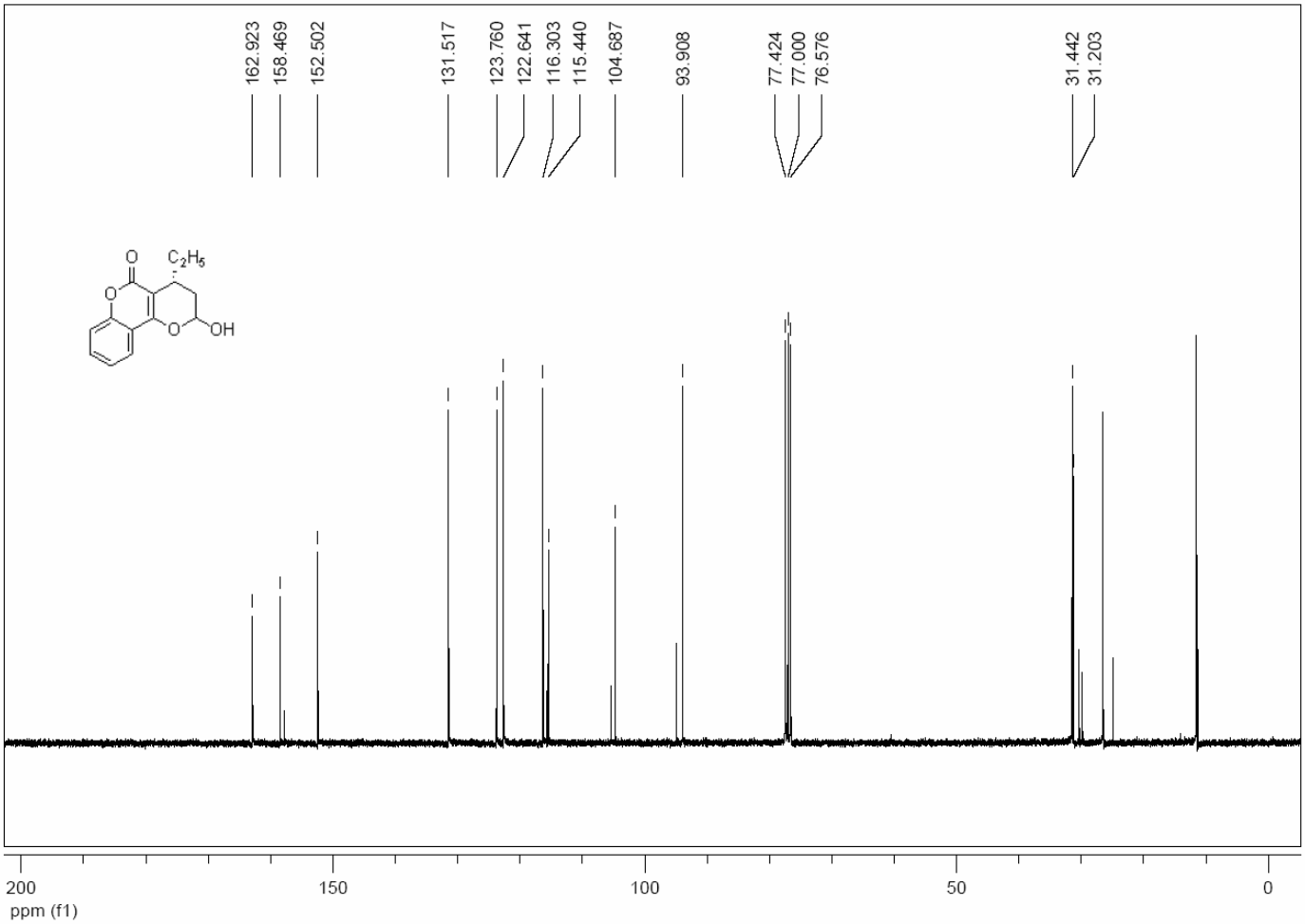
Data file: EM404-6_ASH_5050_flow06_11.DATA
Method: HPLC1_ASH_5050_flow06_acq_60

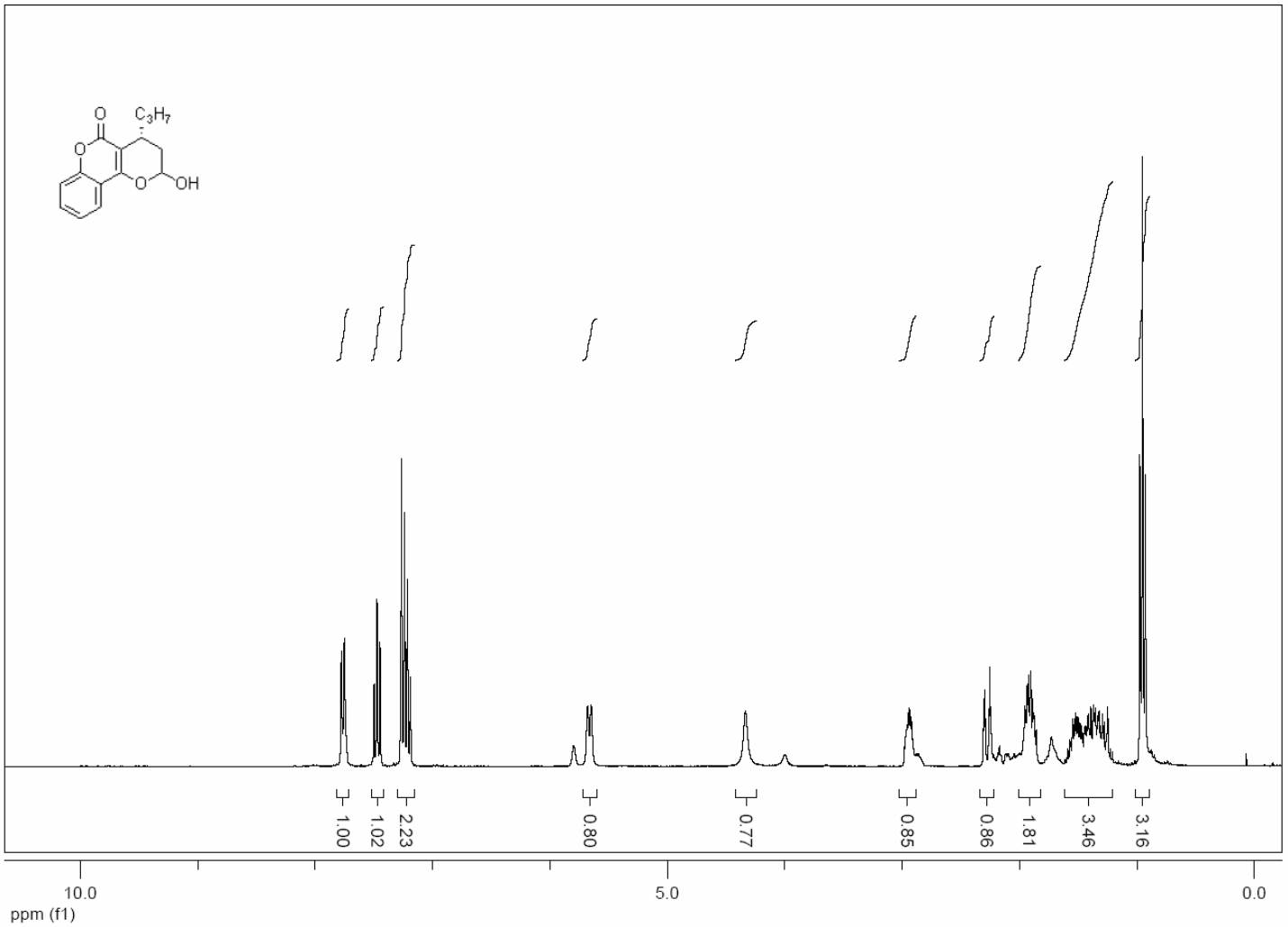


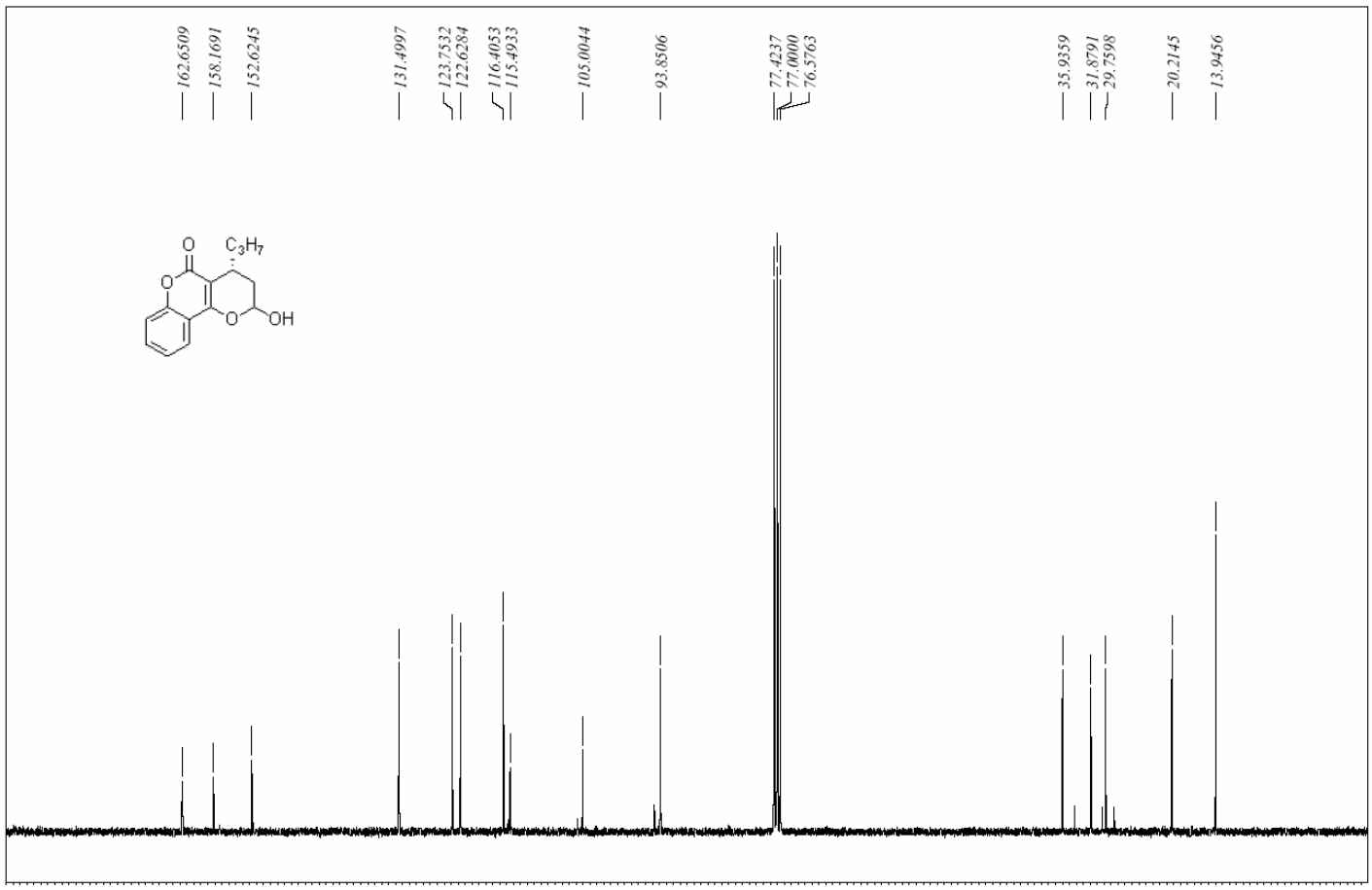
EM404-6_ASH_5050_flow06_11.DATA [Jasco UV 1]

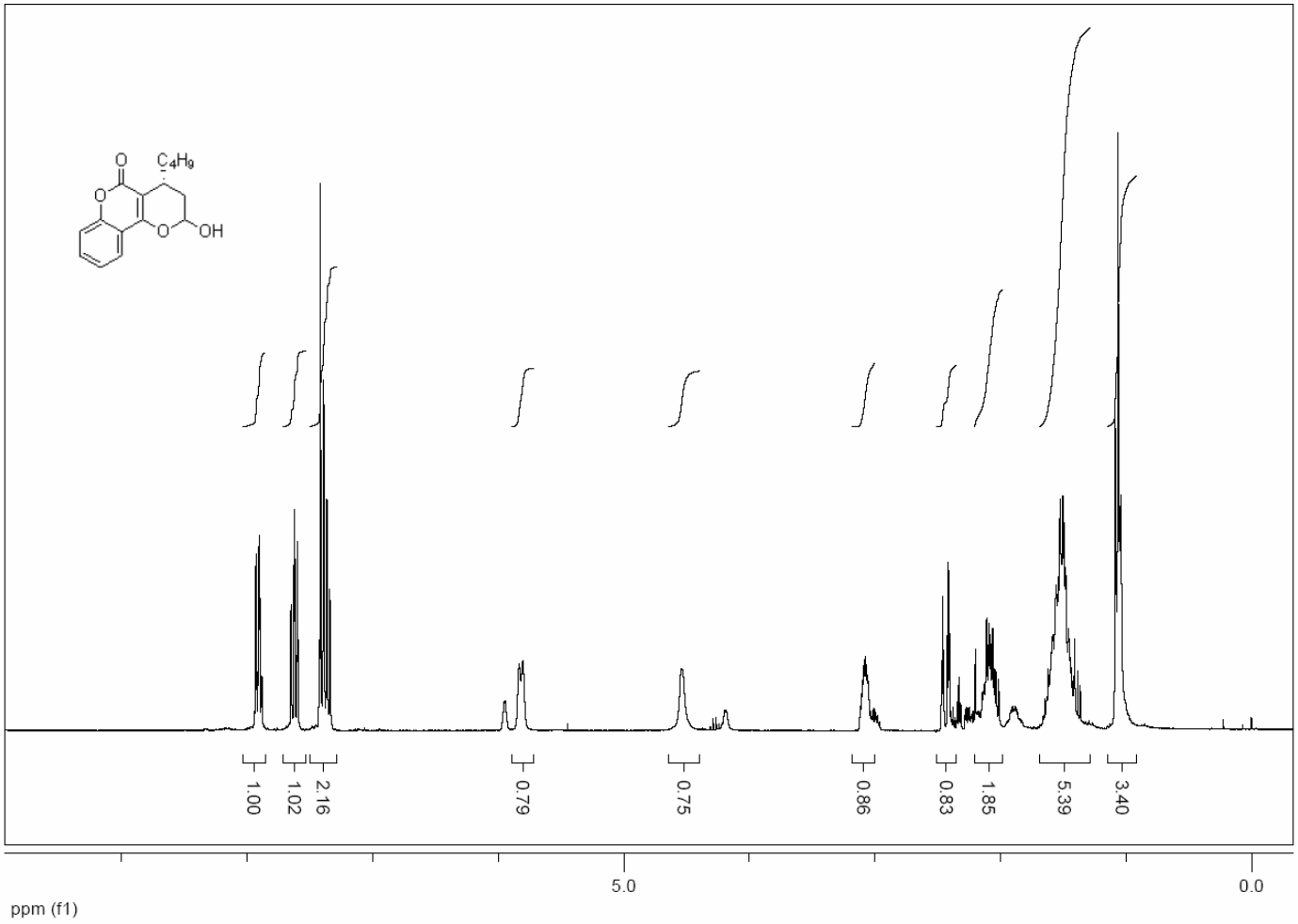
Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	17,680	19,408	21,657	7,680
2	26,133	29,967	35,801	92,320
Total				100,000

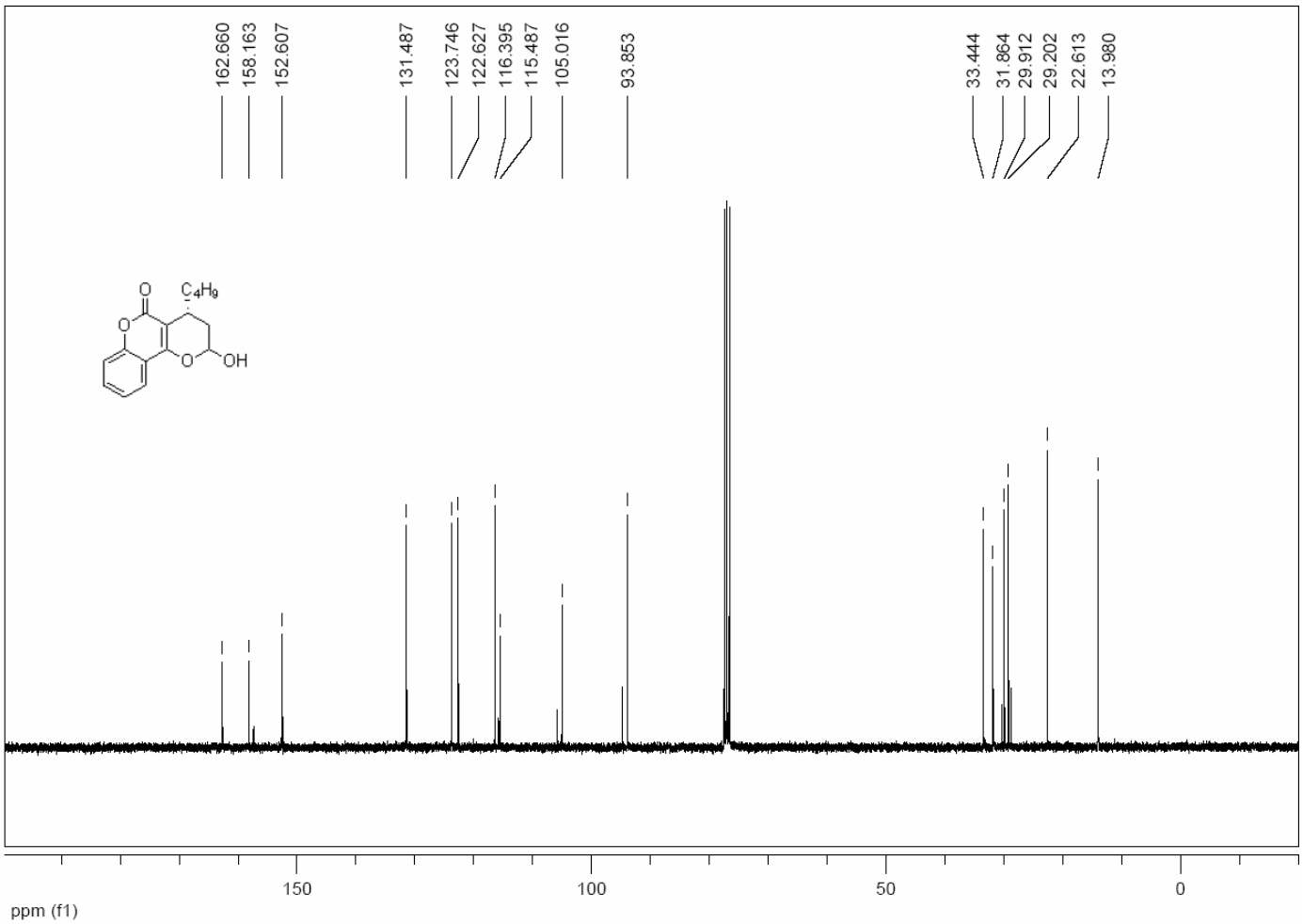

O
C_2H_5
O
O
OH
1.00
1.05
2.05
0.82
0.76
0.90
0.84
2.34
1.16
3.25
10.0
5.0
0.0
ppm (f1)

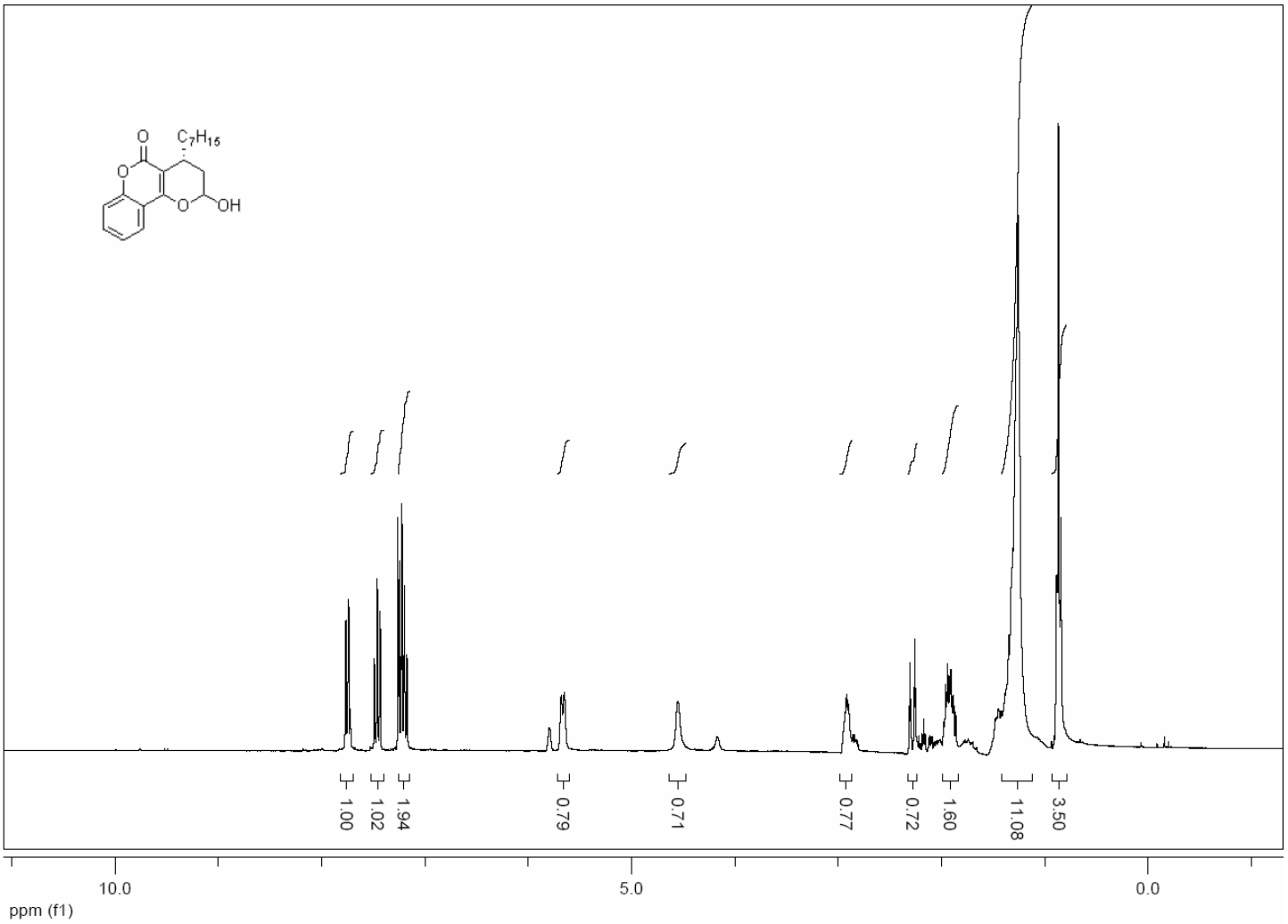

162.923
158.469
152.502
131.517
123.760
122.641
116.303
115.440
104.687
93.908
77.424
77.000
76.576
31.442
31.203
O
C_2H_5
O
O
OH
200
150
100
50
0
ppm (f1)

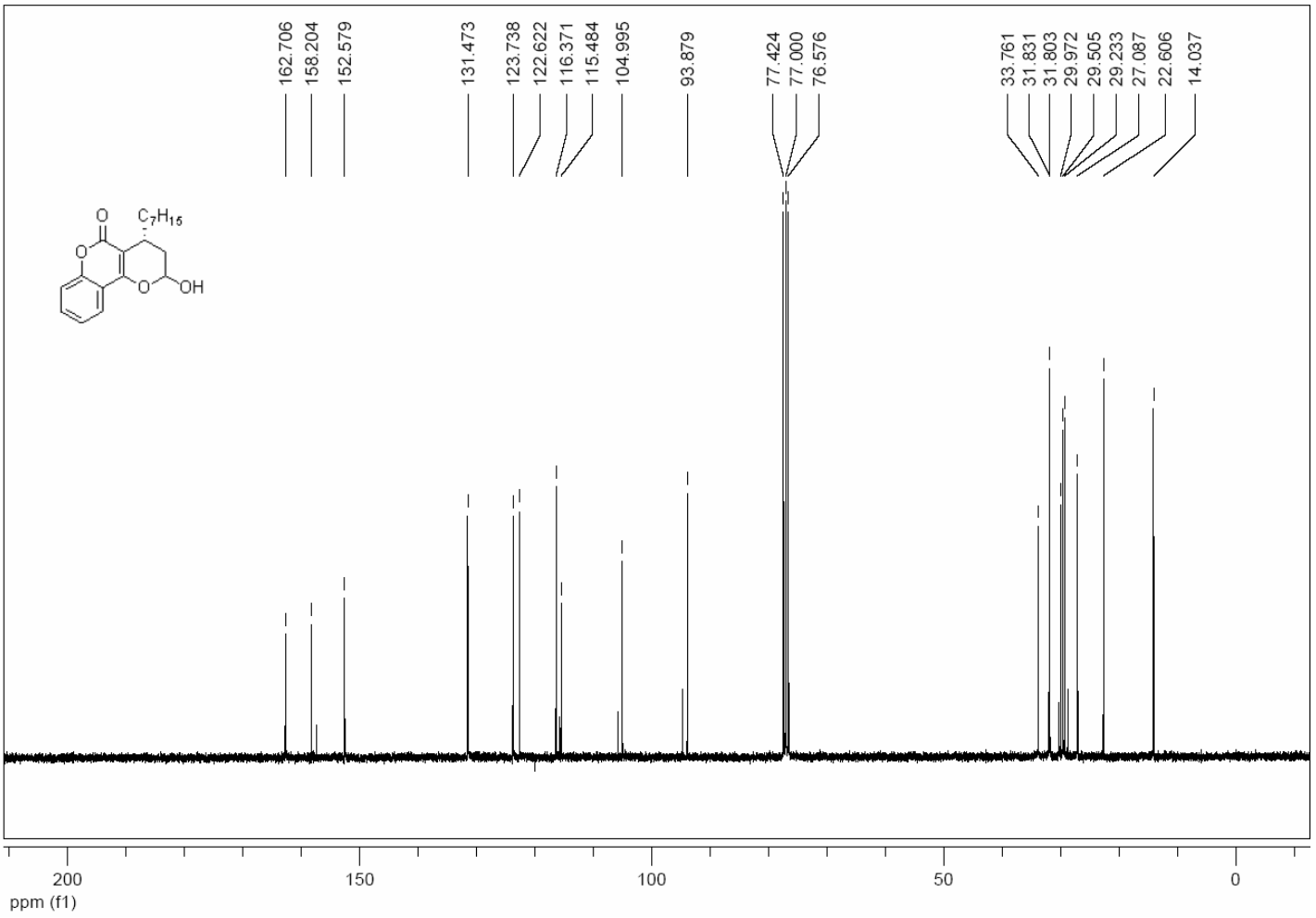

O
C3H7
O
O
OH
1.00
1.02
2.23
0.80
0.77
0.85
0.86
1.81
3.46
3.16
10.0
5.0
0.0
ppm (f1)

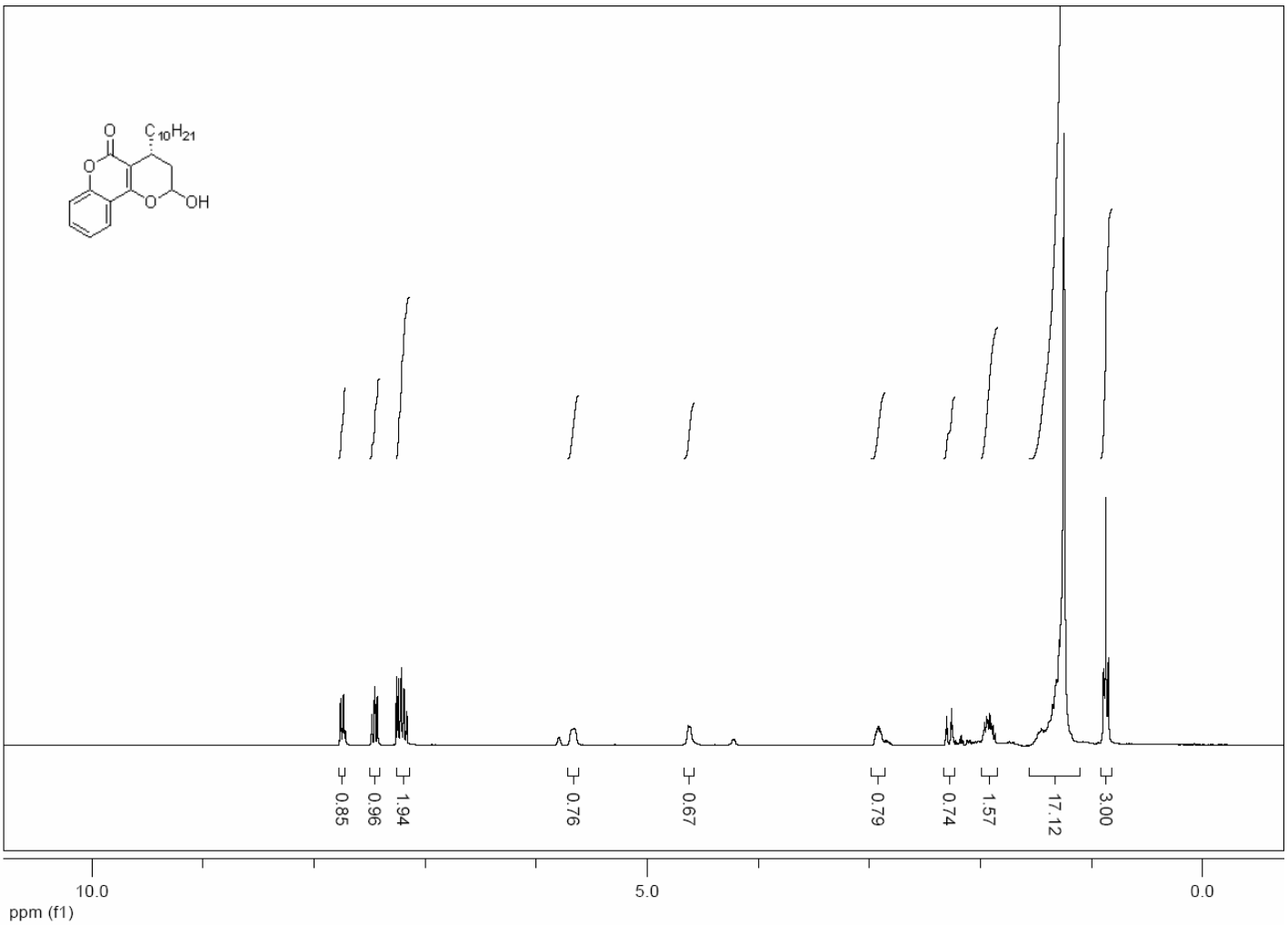

162.6509
158.1691
152.6245
131.4997
123.7532
122.6284
116.4053
115.4933
105.0044
93.8506
77.4237
77.0000
76.5763
35.9359
31.8791
29.7598
20.2145
13.9456
O
C3H7
O
O
OH

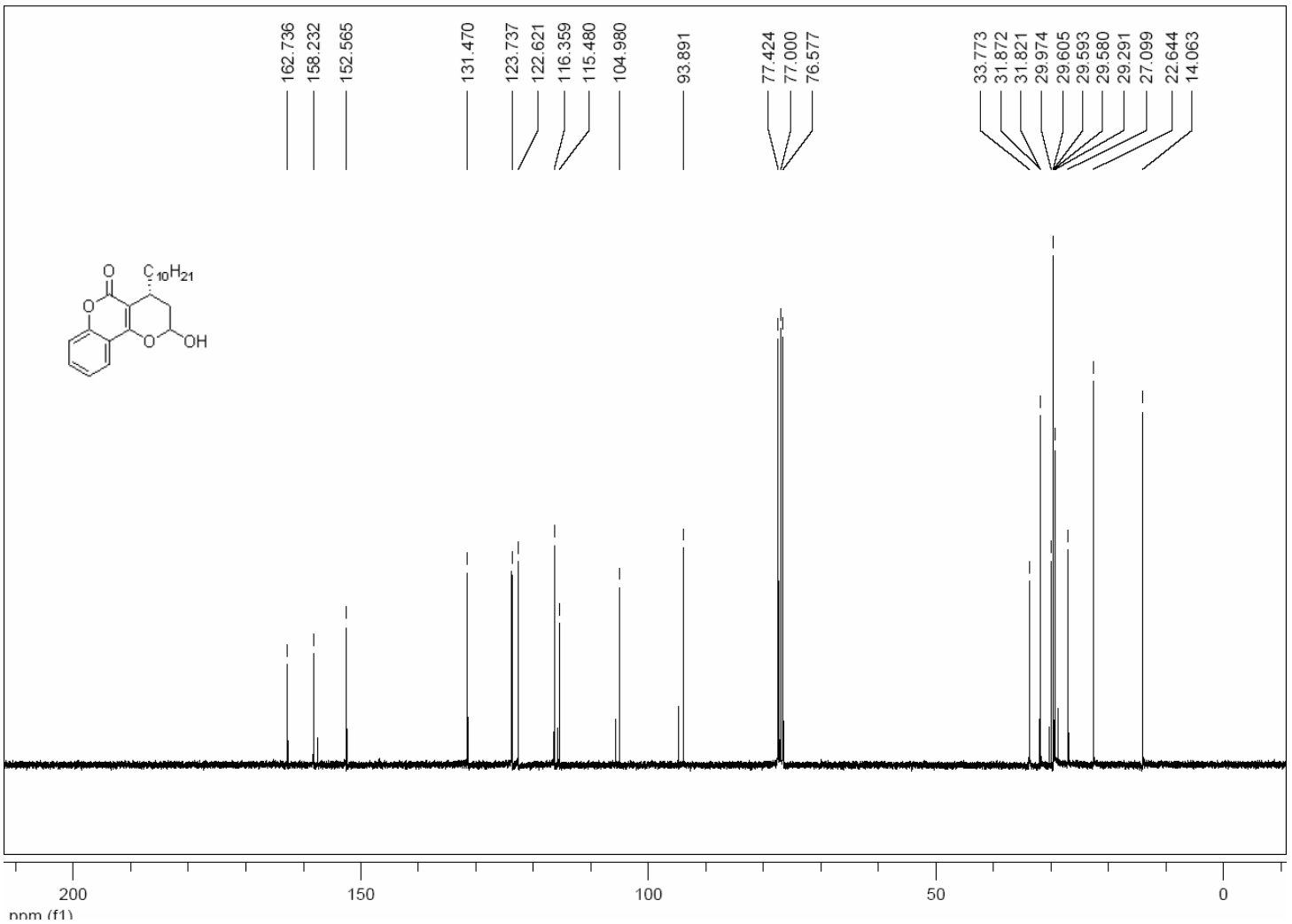

O
C4H9
O
O
OH
1.00
1.02
2.16
0.79
0.75
0.86
0.83
1.85
5.39
3.40
5.0
0.0
ppm (f1)

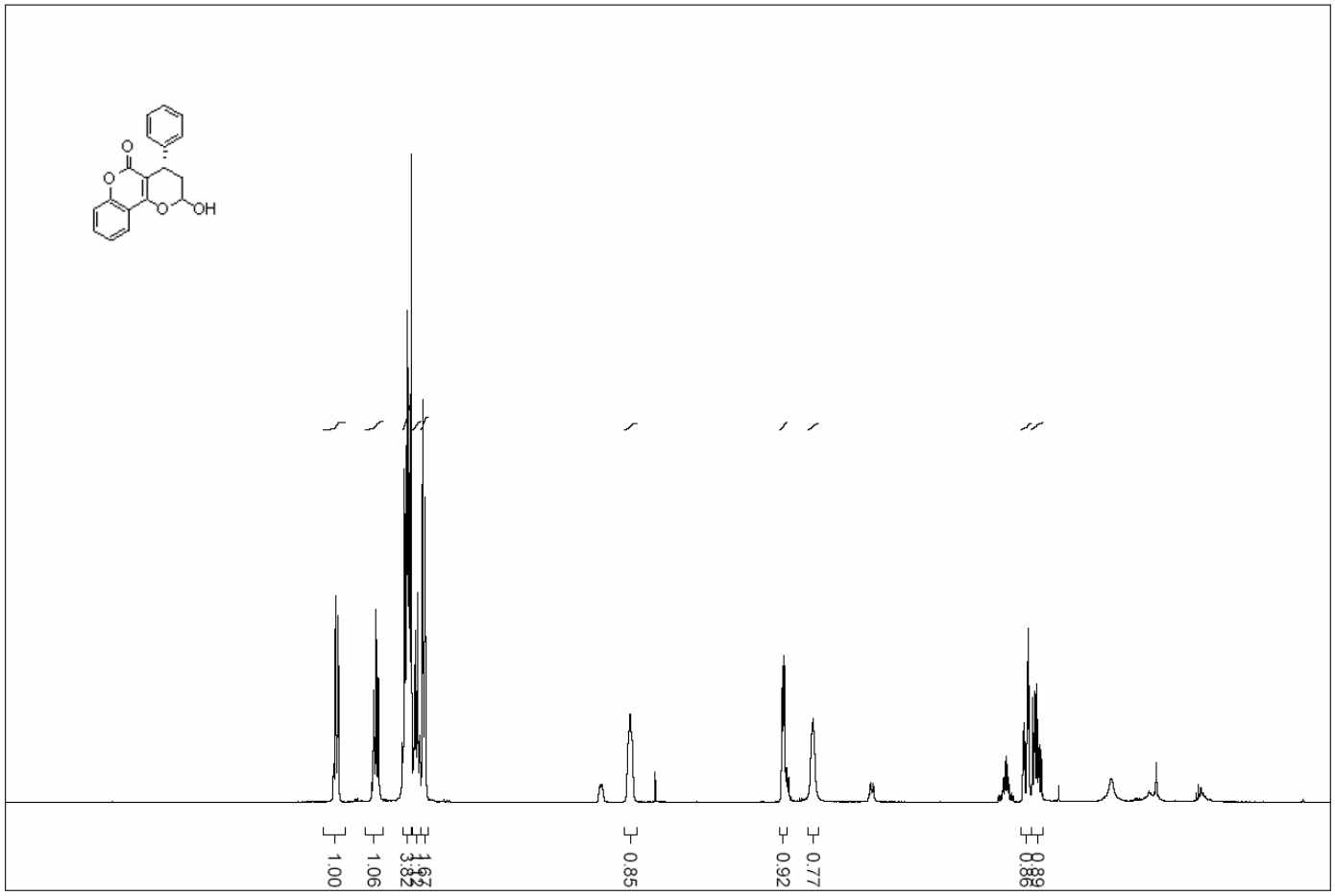

162.660
158.163
152.607
131.487
123.746
122.627
116.395
115.487
105.016
93.853
33.444
31.864
29.912
29.202
22.613
13.980
O
C4H9
O
O
OH
150
100
50
0
ppm (f1)

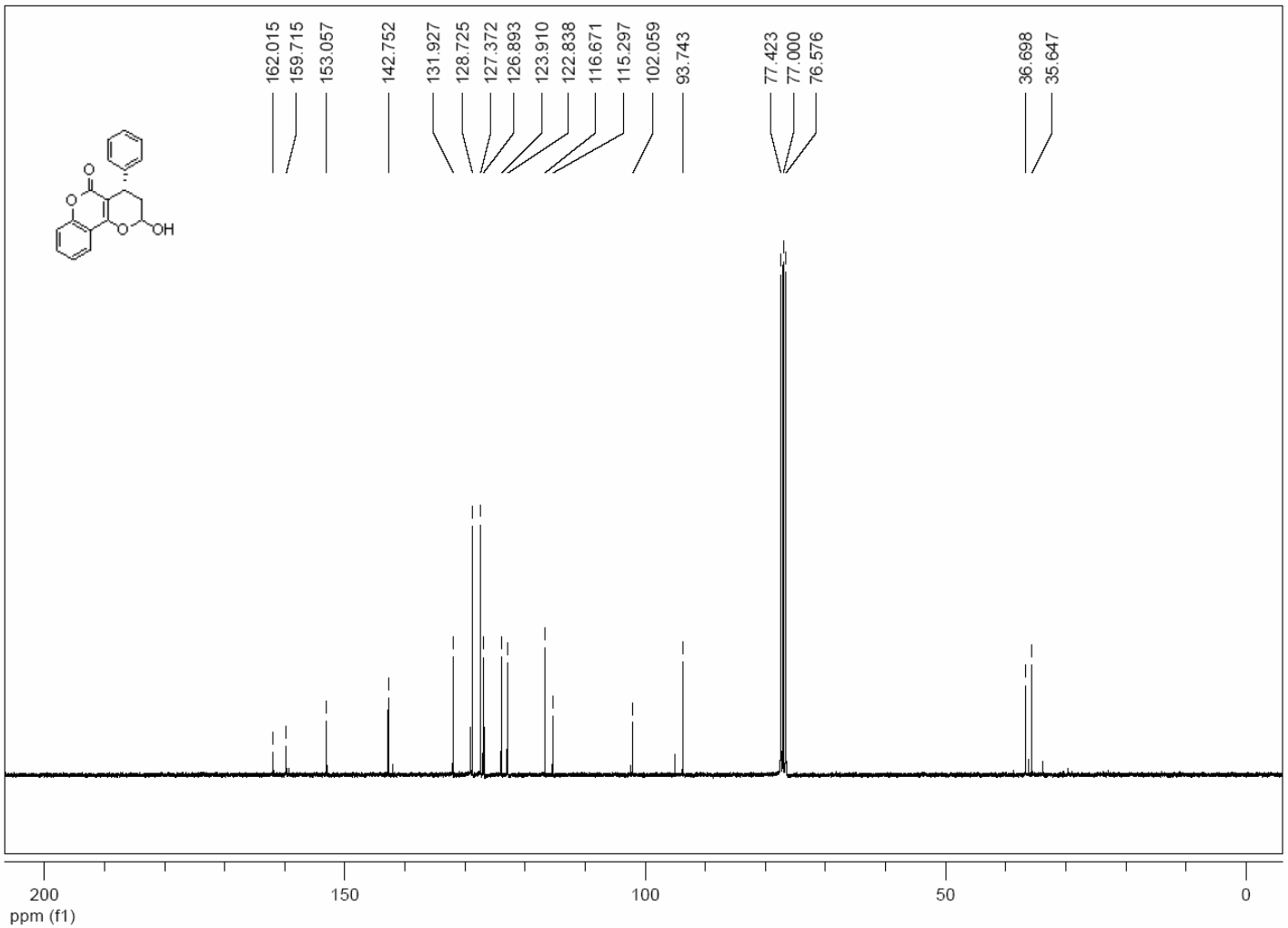

O
C7H15
O
O
OH
1.00
1.02
1.64
0.79
0.71
0.77
0.72
1.60
11.08
3.50
10.0
5.0
0.0
ppm (f1)

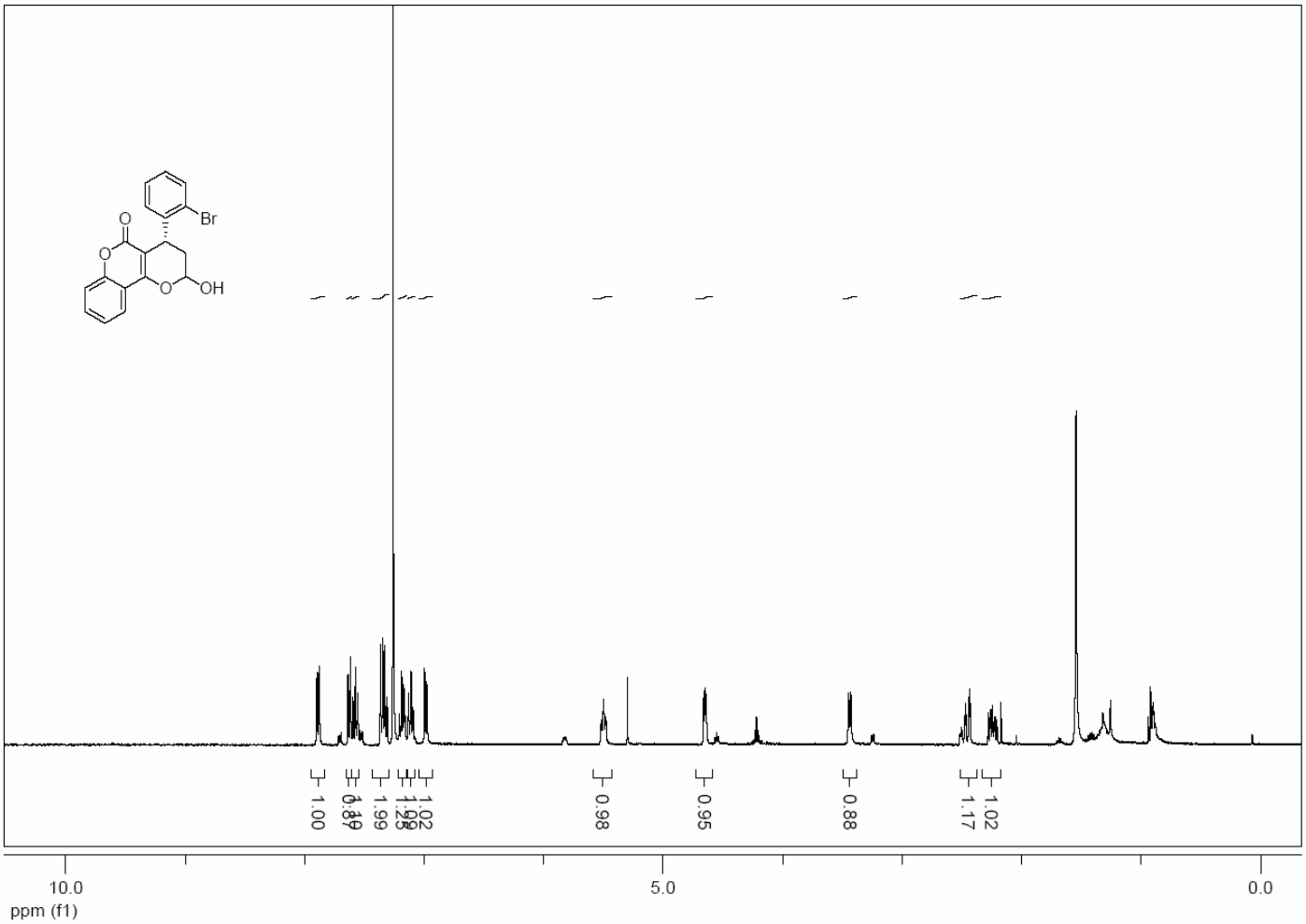

162.706
158.204
152.579
131.473
123.738
122.622
116.371
115.484
104.995
93.879
77.424
77.000
76.576
33.761
31.831
31.803
29.972
29.505
29.233
27.087
22.606
14.037
O
C7H15
O
O
OH
200
150
100
50
0
ppm (f1)

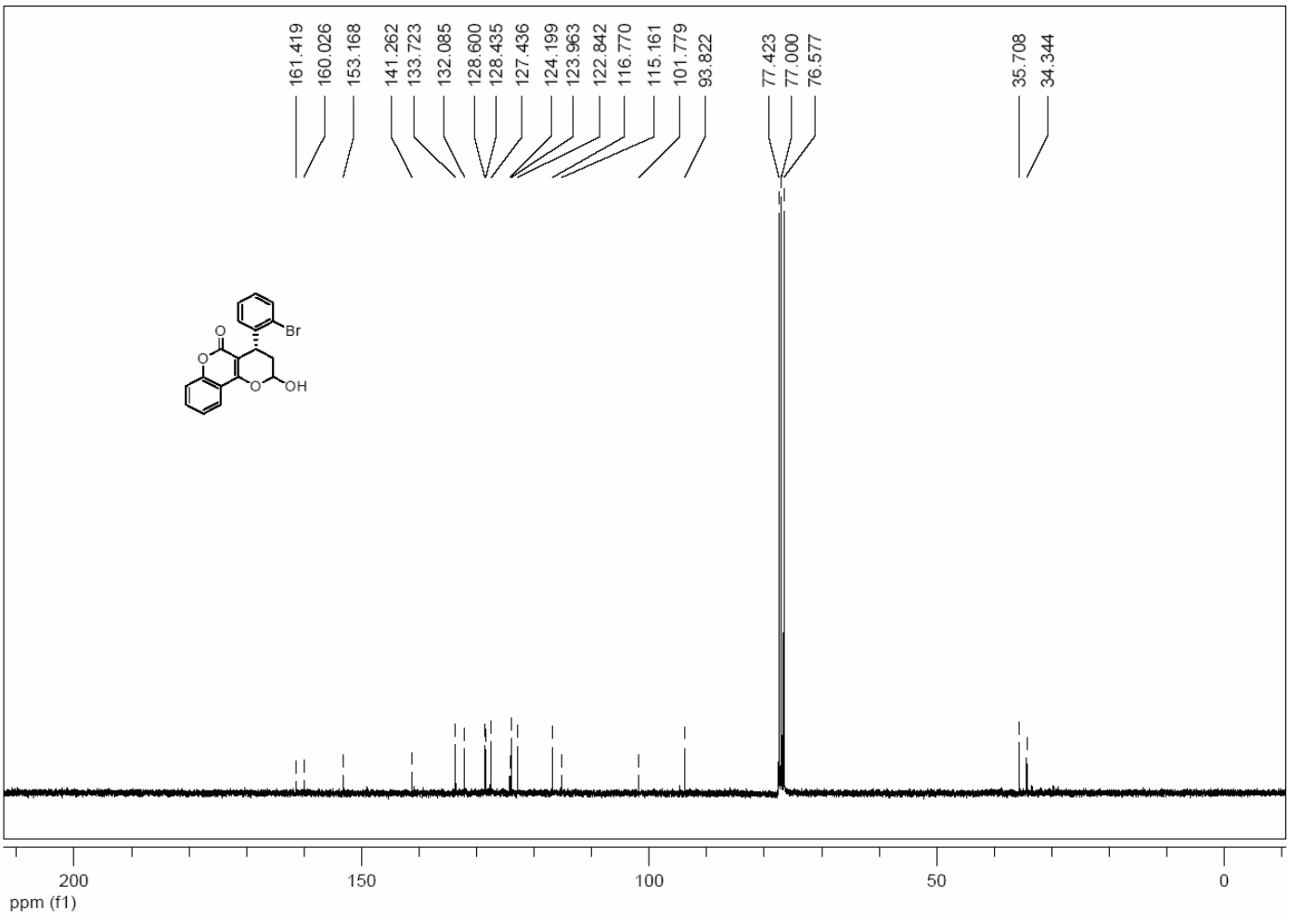

O
O
O
OH
$C_{10}H_{21}$
0.85
0.96
1.94
0.76
0.67
0.79
0.74
1.57
17.12
3.00
10.0
5.0
0.0
ppm (f1)

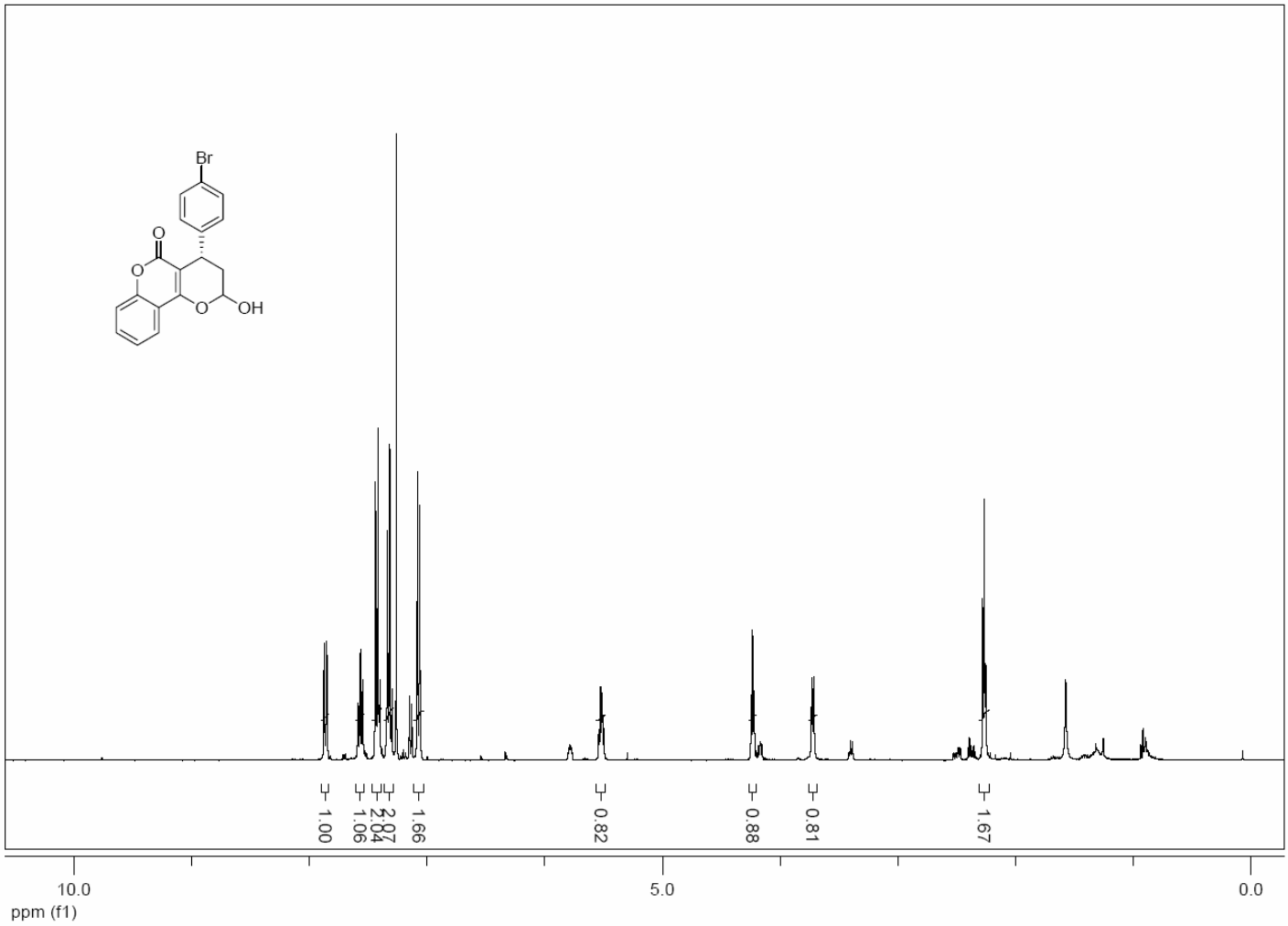

162.736
158.232
152.565
131.470
123.737
122.621
116.359
115.480
104.980
93.891
77.424
77.000
76.577
33.773
31.872
31.821
29.974
29.605
29.593
29.580
29.291
27.099
22.644
14.063
O
O
O
OH
$C_{10}H_{21}$
200
150
100
50
0
ppm (f1)

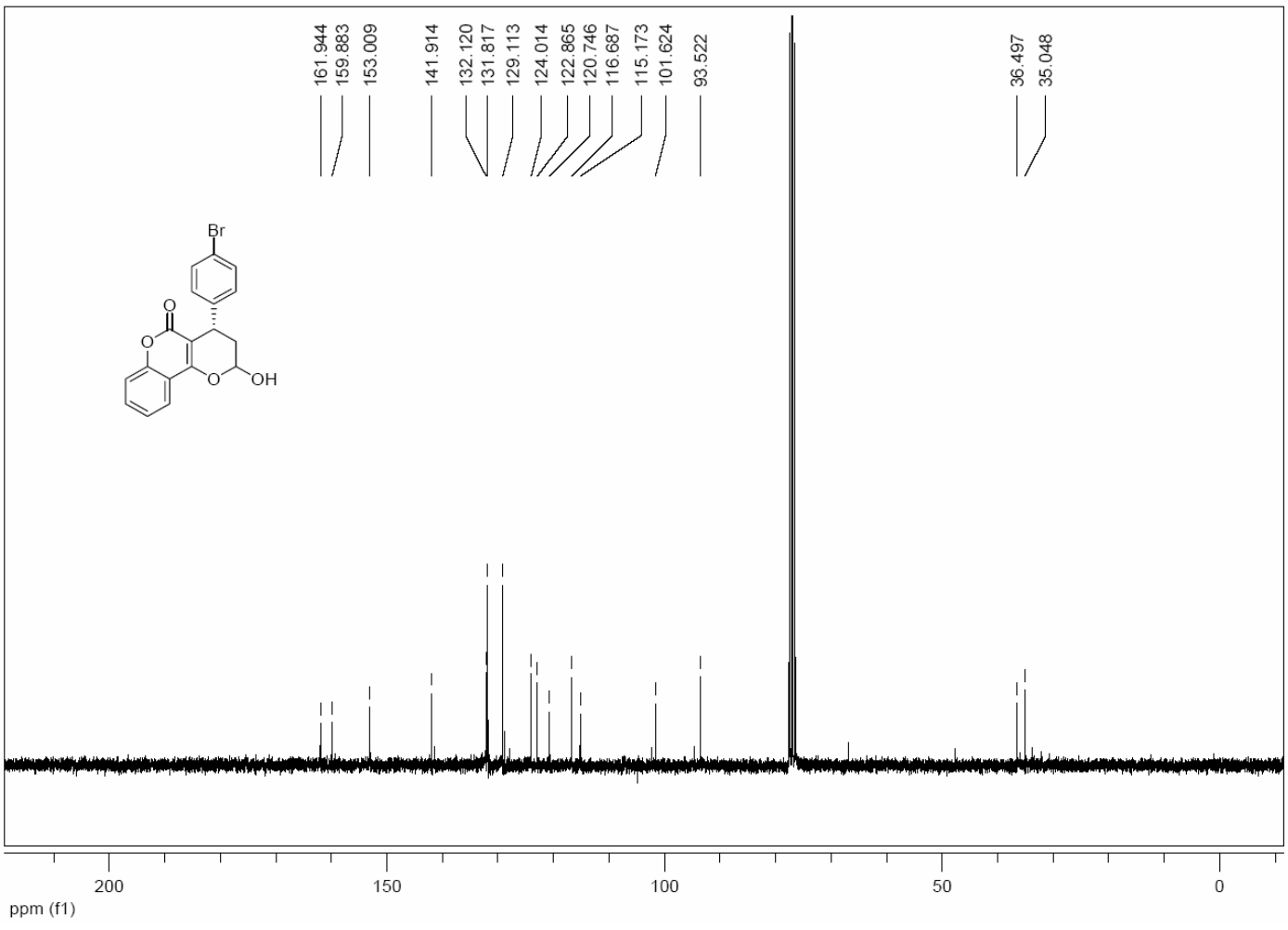

O
O
O
OH
1.00
1.09
3.82
1.97
0.85
0.92
0.77
0.88
0.89

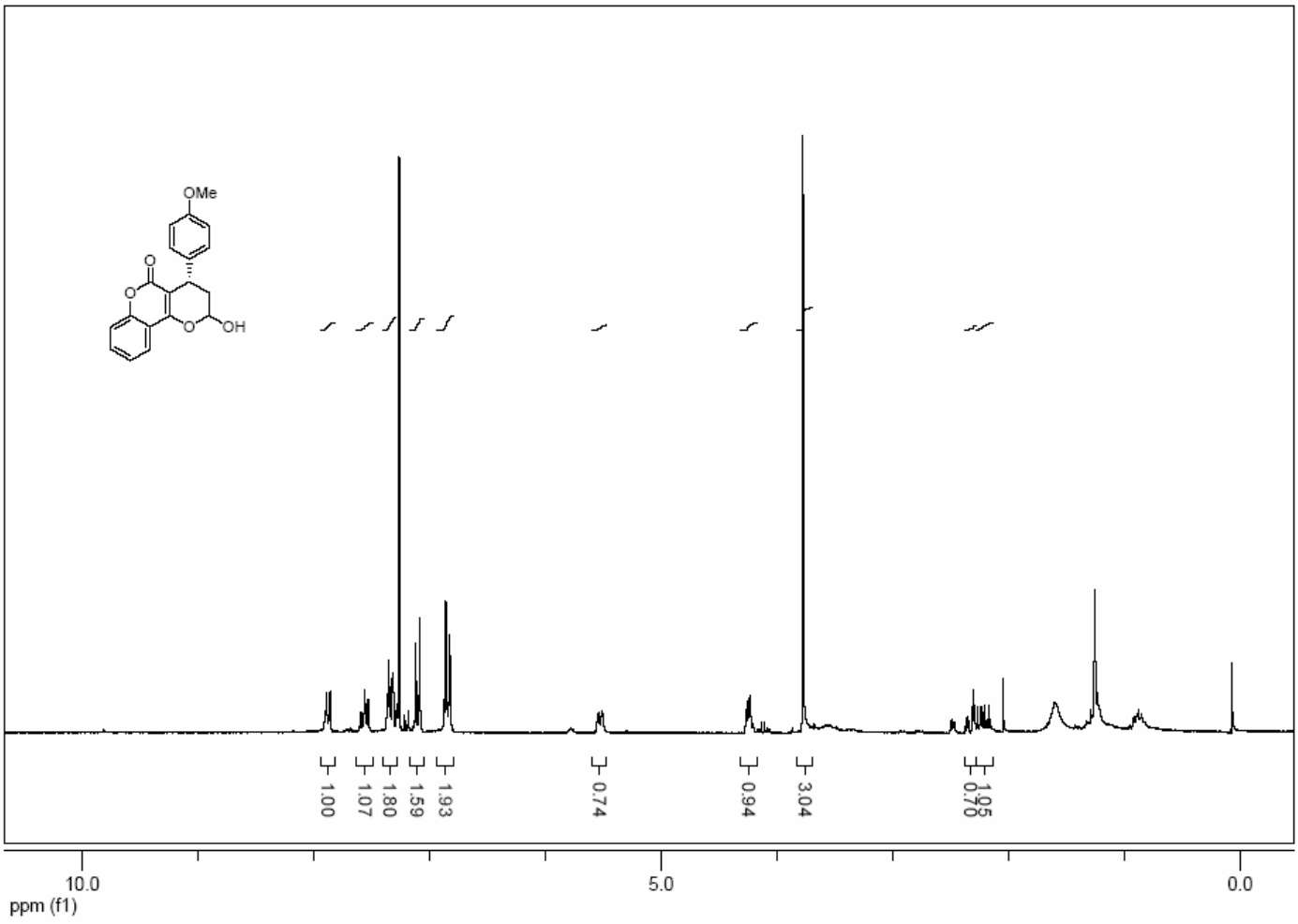

162.015
159.715
153.057
142.752
131.927
128.725
127.372
126.893
123.910
122.838
116.671
115.297
102.059
93.743
77.423
77.000
76.576
36.698
35.647
O
O
O
OH
200
150
100
50
0
ppm (f1)

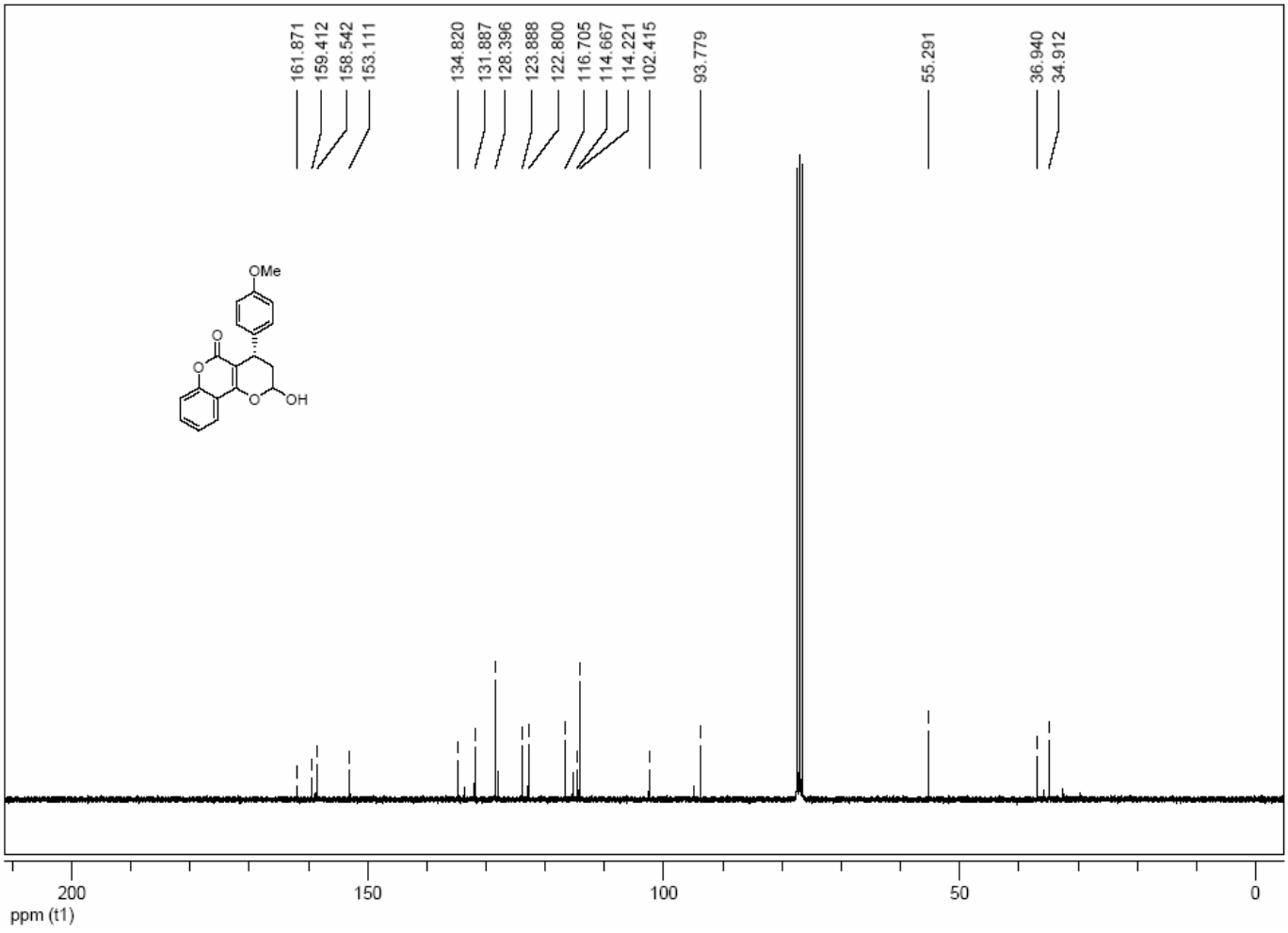

Br
O
O
O
OH
1.00
0.98
0.95
0.88
1.17
1.02
10.0
5.0
0.0
ppm (f1)

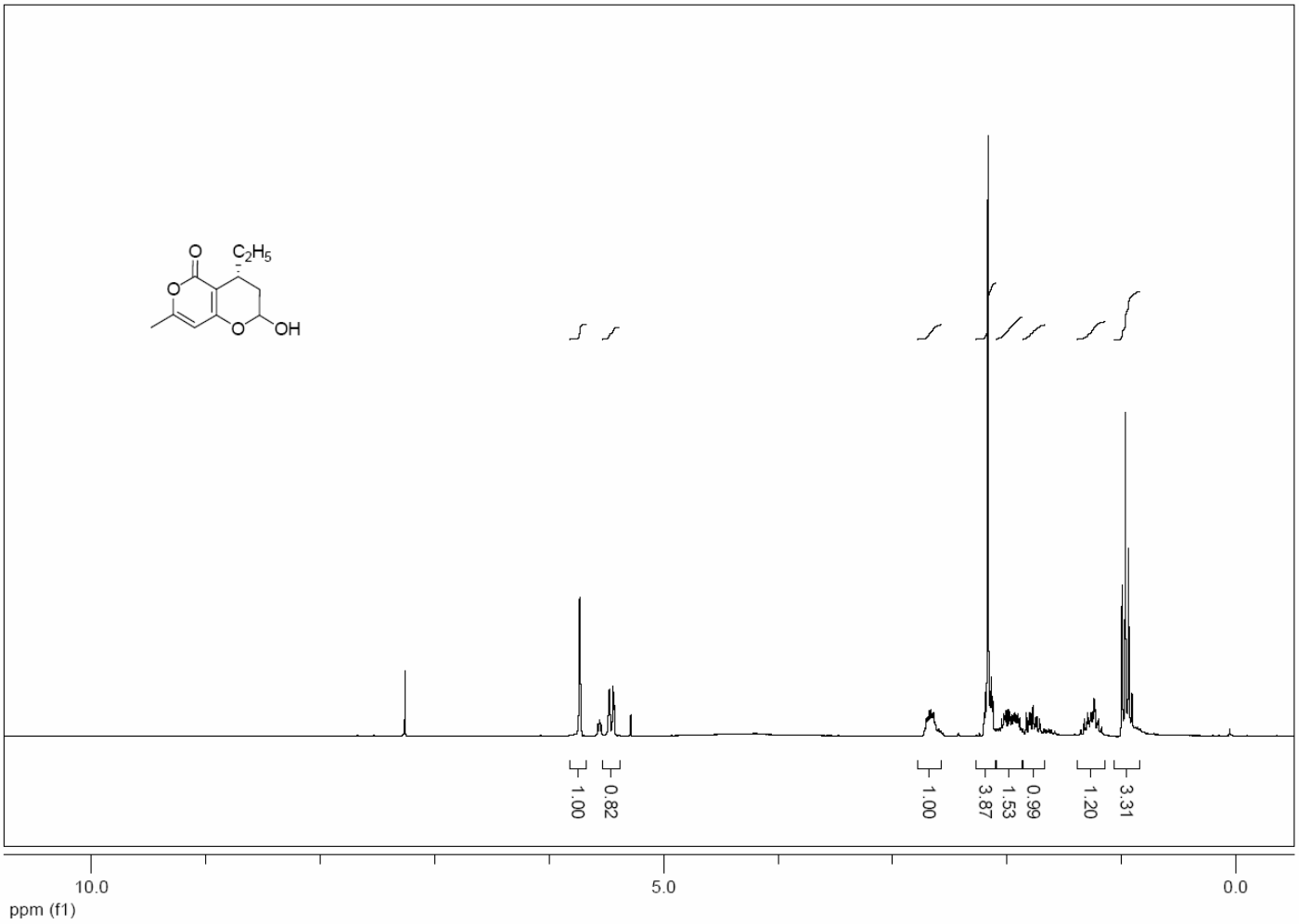

161.419
160.026
153.168
141.262
133.723
132.085
128.600
128.435
127.436
124.199
123.963
122.842
116.770
115.161
101.779
93.822
77.423
77.000
76.577
35.708
34.344
Br
O
O
O
OH
200
150
100
50
0
ppm (f1)

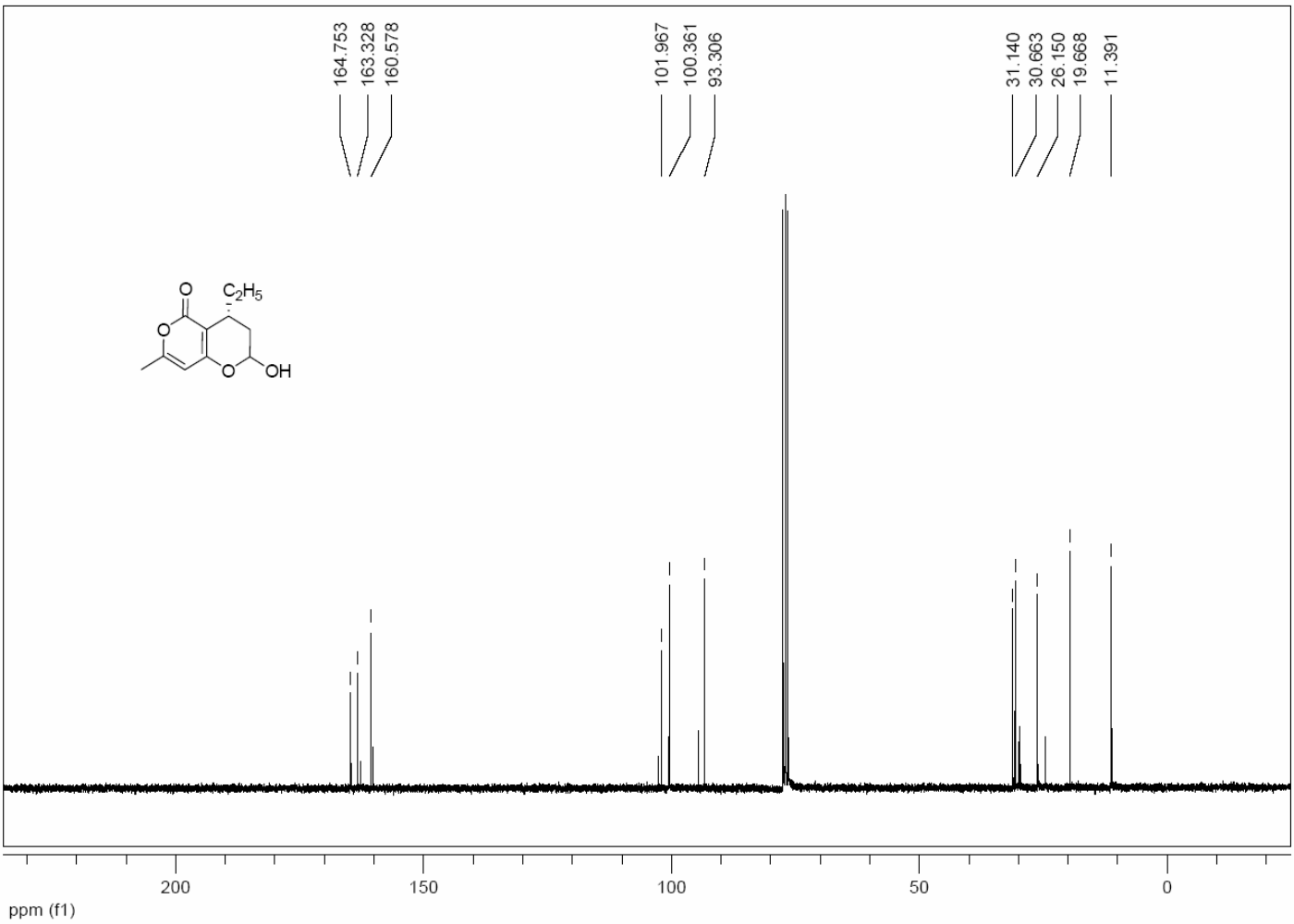

Br
O
O
O
OH
1.00
1.06
2.04
2.07
1.66
0.82
0.88
0.81
1.67
10.0
5.0
0.0
ppm (f1)

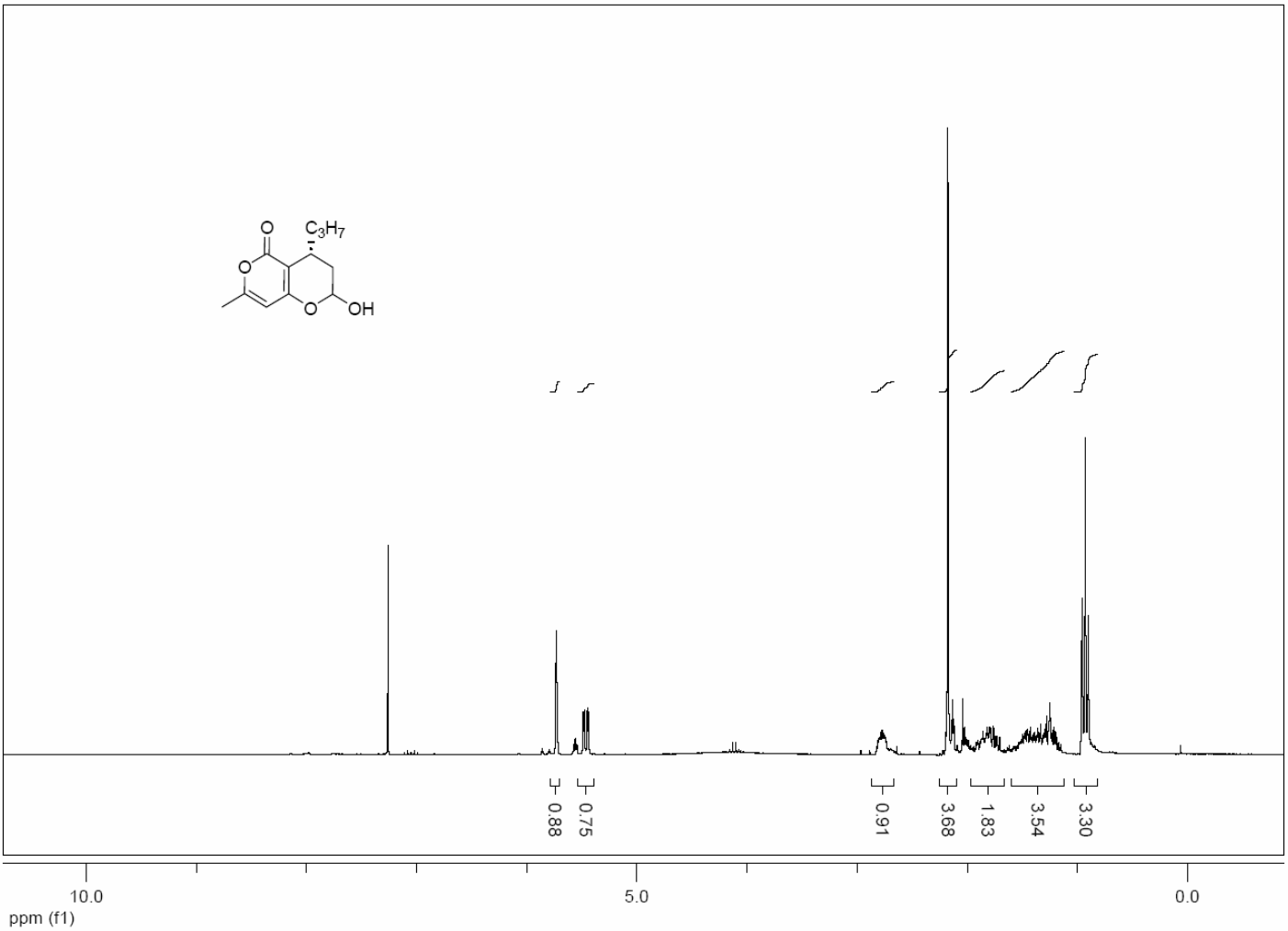

161.944
159.883
153.009
141.914
132.120
131.817
129.113
124.014
122.865
120.746
116.687
115.173
101.624
93.522
36.497
35.048
Br
O
O
O
OH
200
150
100
50
0
ppm (f1)

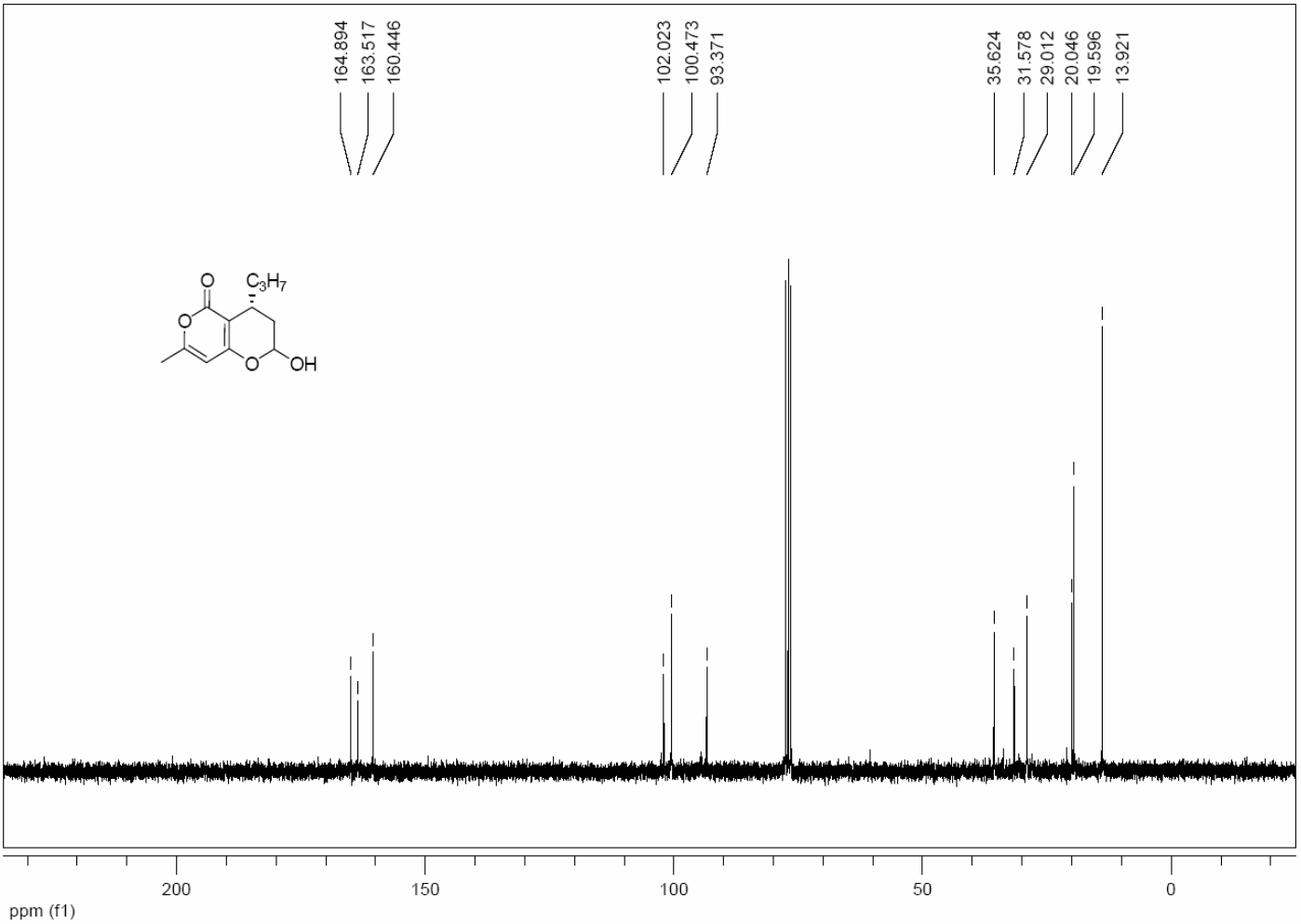

OMe
O
O
O
OH
1.00
1.07
1.80
1.59
1.93
0.74
0.94
3.04
0.70
1.05
10.0
5.0
0.0
ppm (f1)

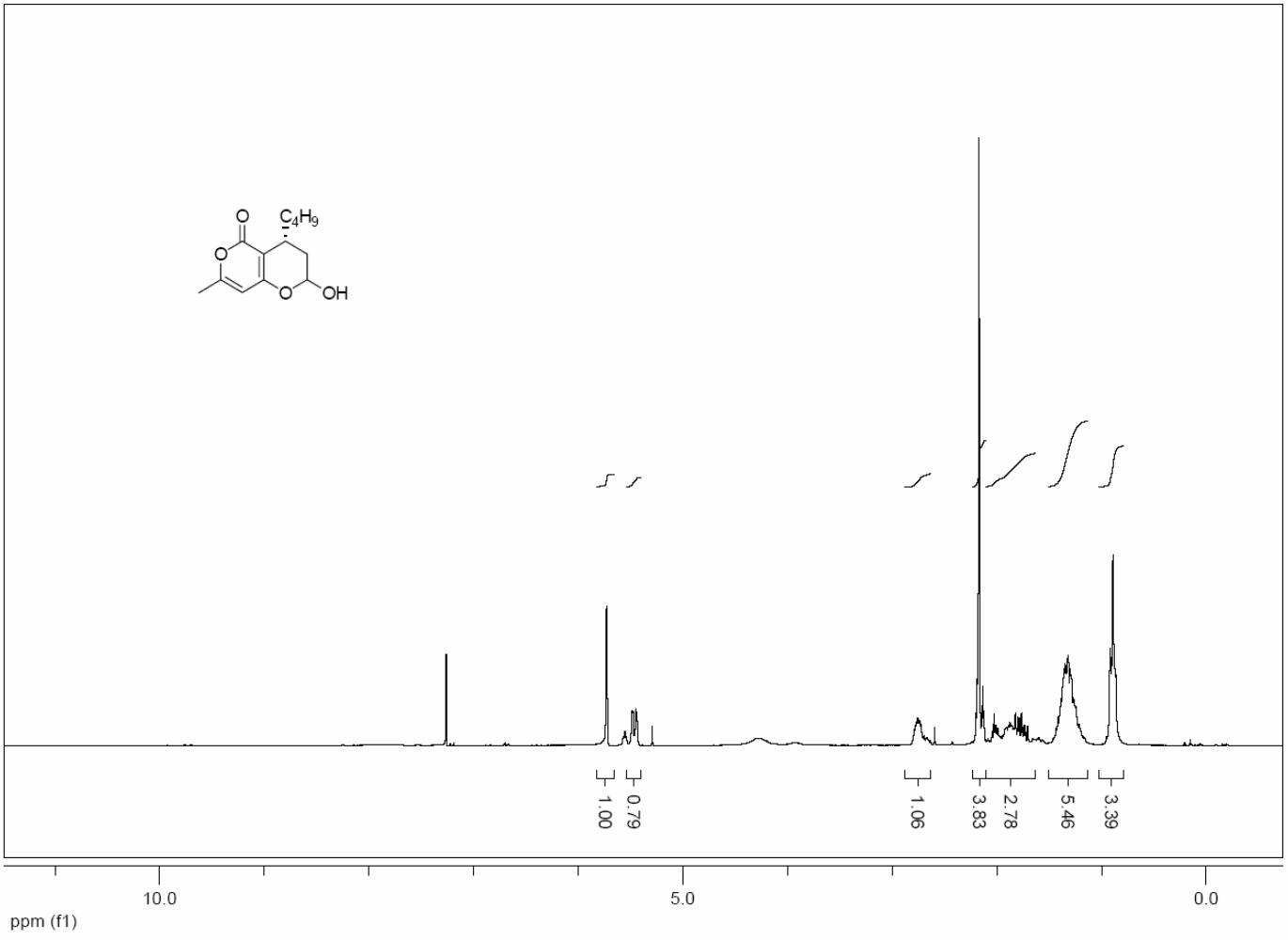

161.871
159.412
158.542
153.111
134.820
131.887
128.396
123.888
122.800
116.705
114.667
114.221
102.415
93.779
55.291
36.940
34.912
OMe
O
O
O
OH
200
150
100
50
0
ppm (t1)

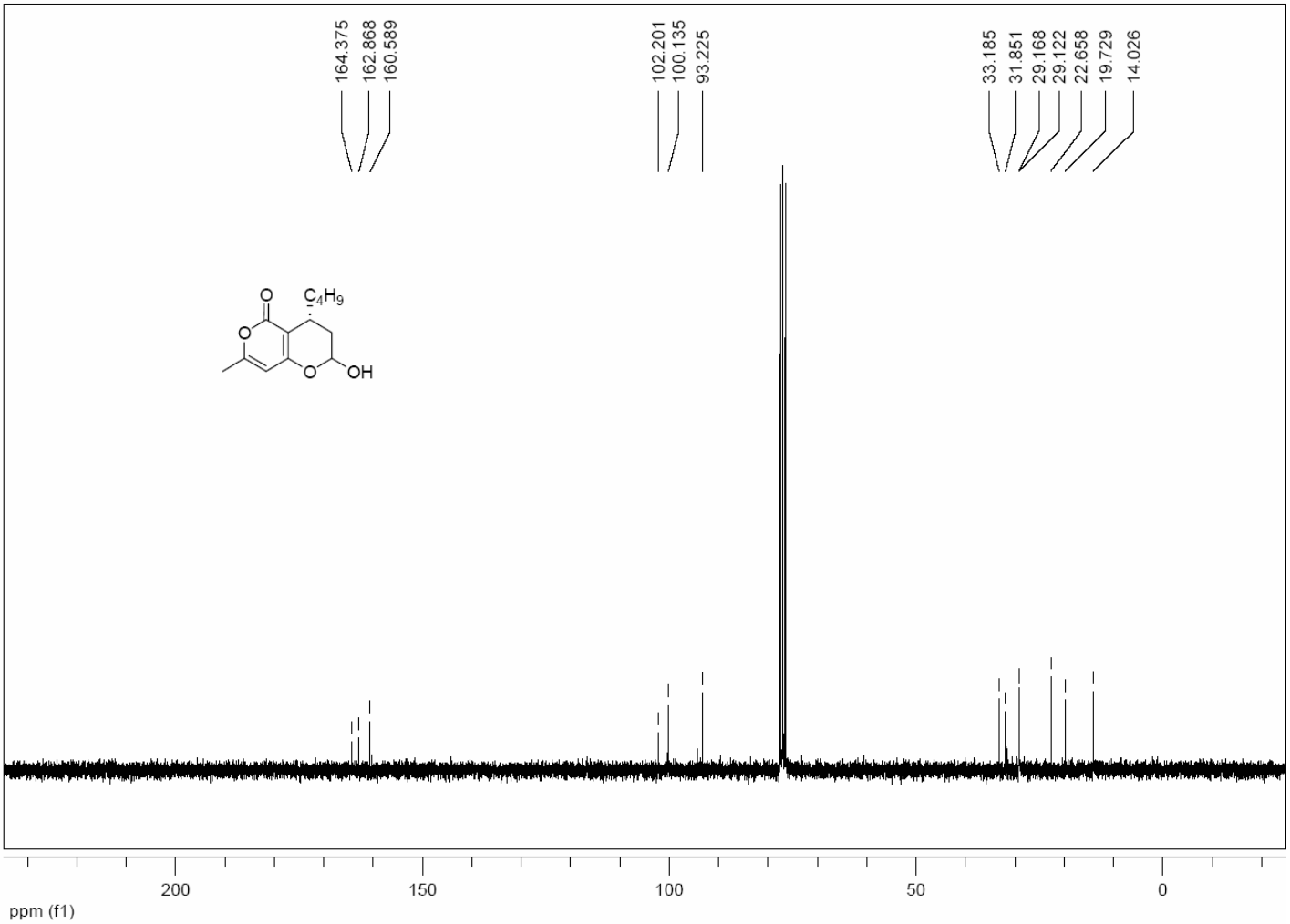

O
C_2H_5
O
O
OH
1.00
0.82
1.00
3.87
1.53
0.99
1.20
3.31
10.0
5.0
0.0
ppm (f1)

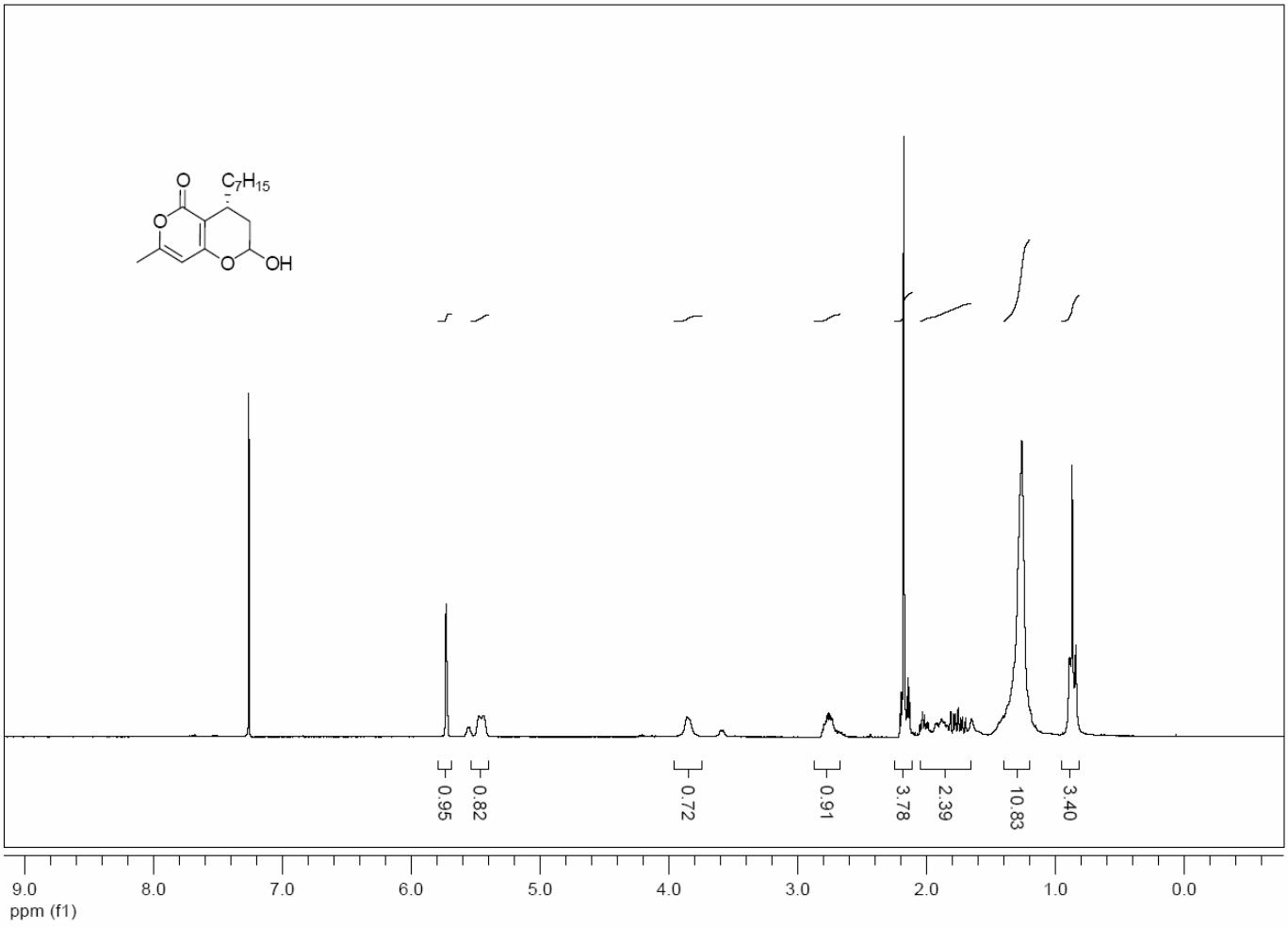

164.753
163.328
160.578
101.967
100.361
93.306
31.140
30.663
26.150
19.668
11.391
O
C_2H_5
O
O
OH
200
150
100
50
0
ppm (f1)

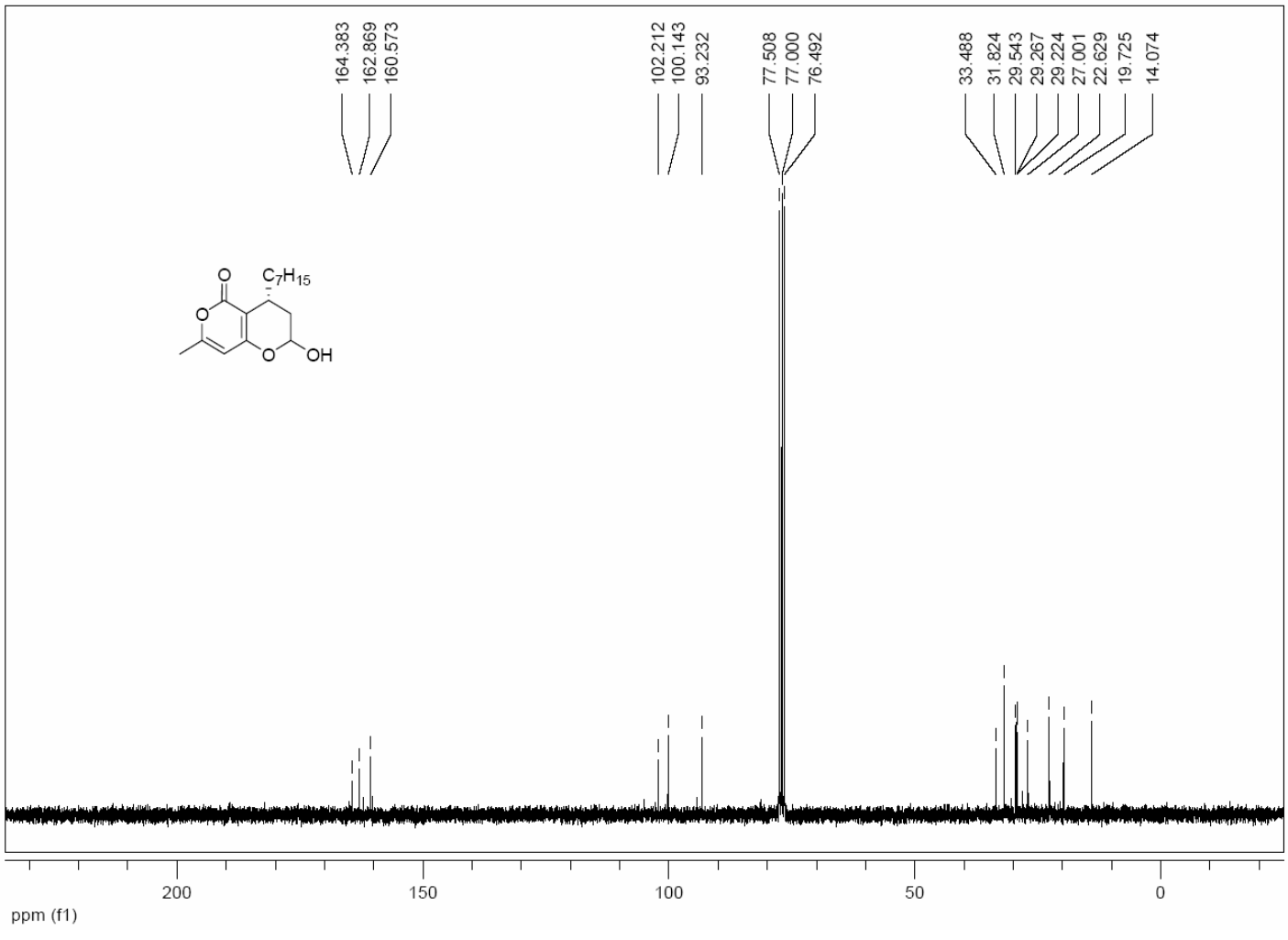

O
C3H7
O
O
OH
0.88
0.75
0.91
3.98
1.83
3.54
3.30
10.0
5.0
0.0
ppm (f1)

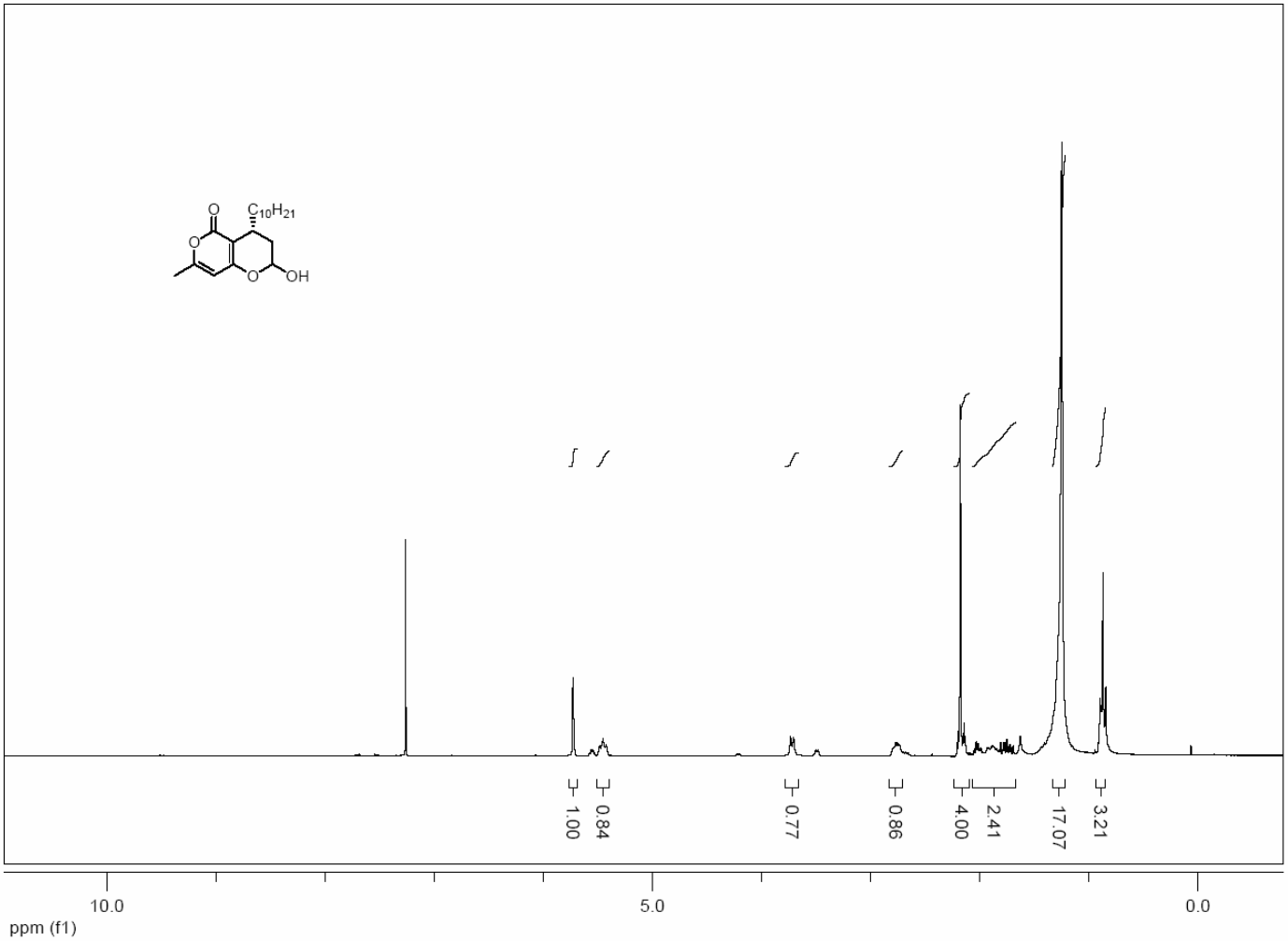

164.894
163.517
160.446
102.023
100.473
93.371
35.624
31.578
29.012
20.046
19.596
13.921
O
C3H7
O
O
OH
200
150
100
50
0
ppm (f1)

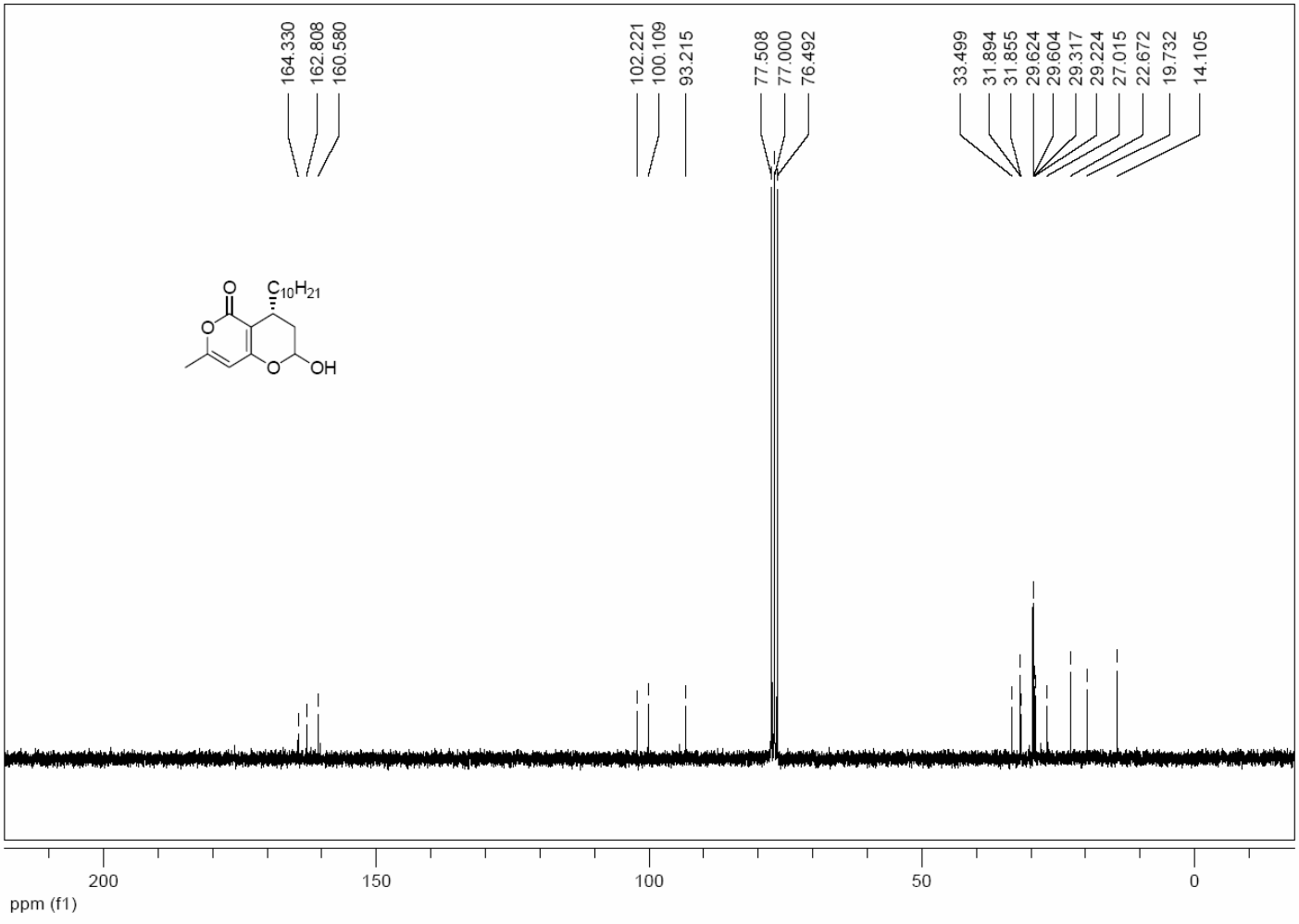

O
C4H9
O
O
OH
1.00
0.79
1.06
3.83
2.78
5.49
3.36
10.0
5.0
0.0
ppm (f1)

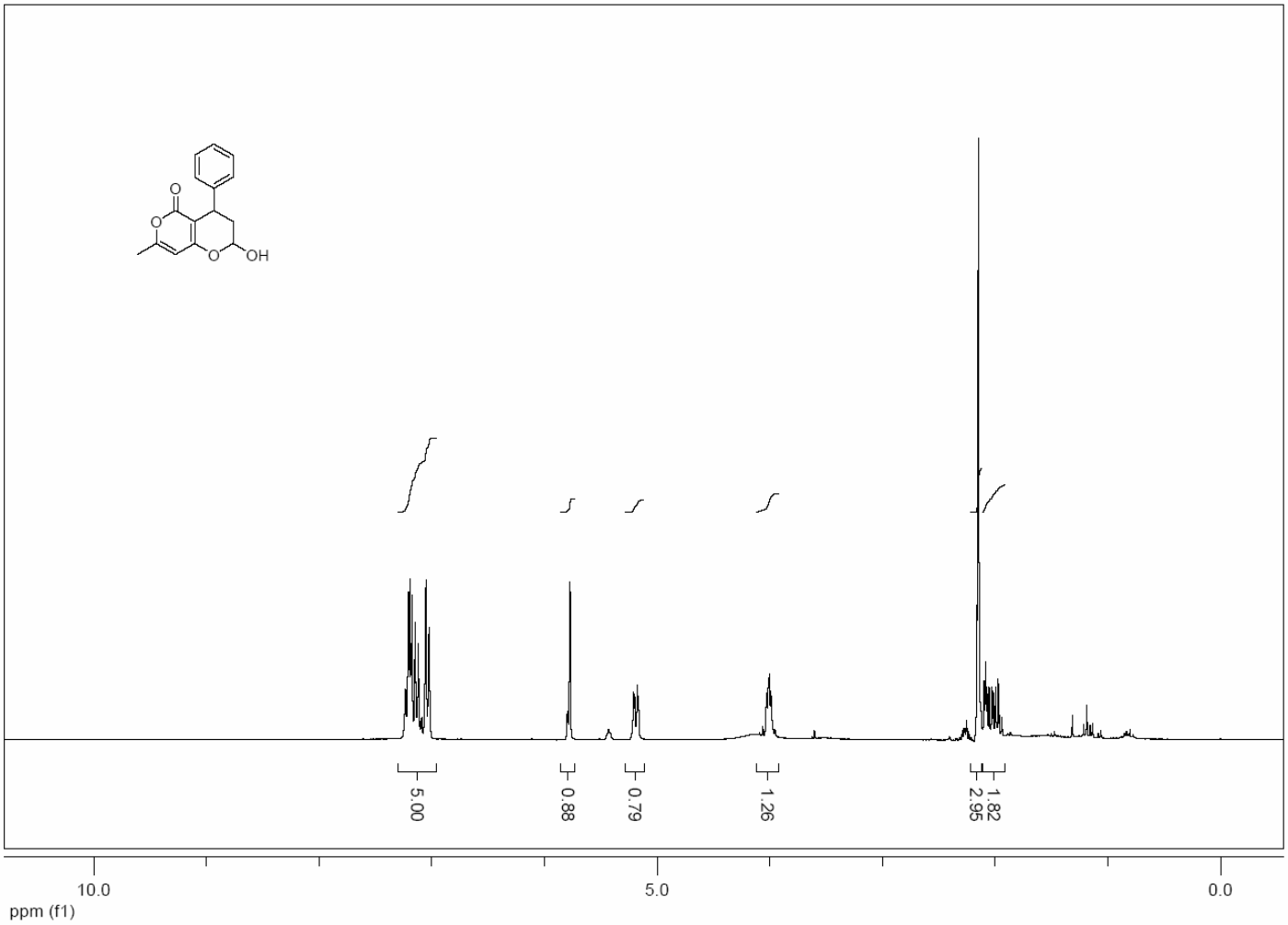

164.375
162.868
160.589
102.201
100.135
93.225
33.185
31.851
29.168
29.122
22.658
19.729
14.026
O
C4H9
O
O
OH
200
150
100
50
0
ppm (f1)

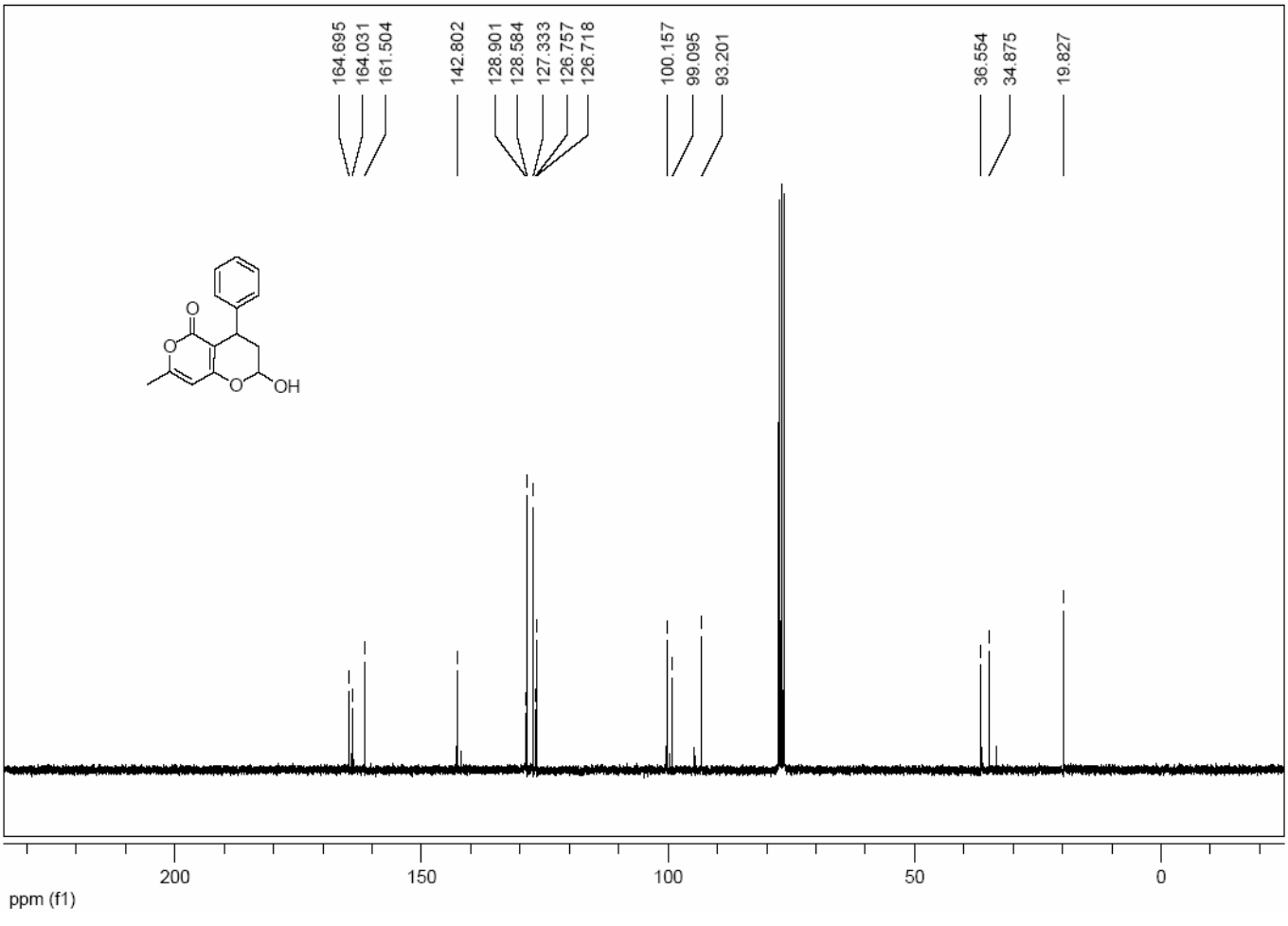

C7H15
O
O
O
OH
0.95
0.82
0.72
0.91
3.78
2.39
10.83
3.40
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

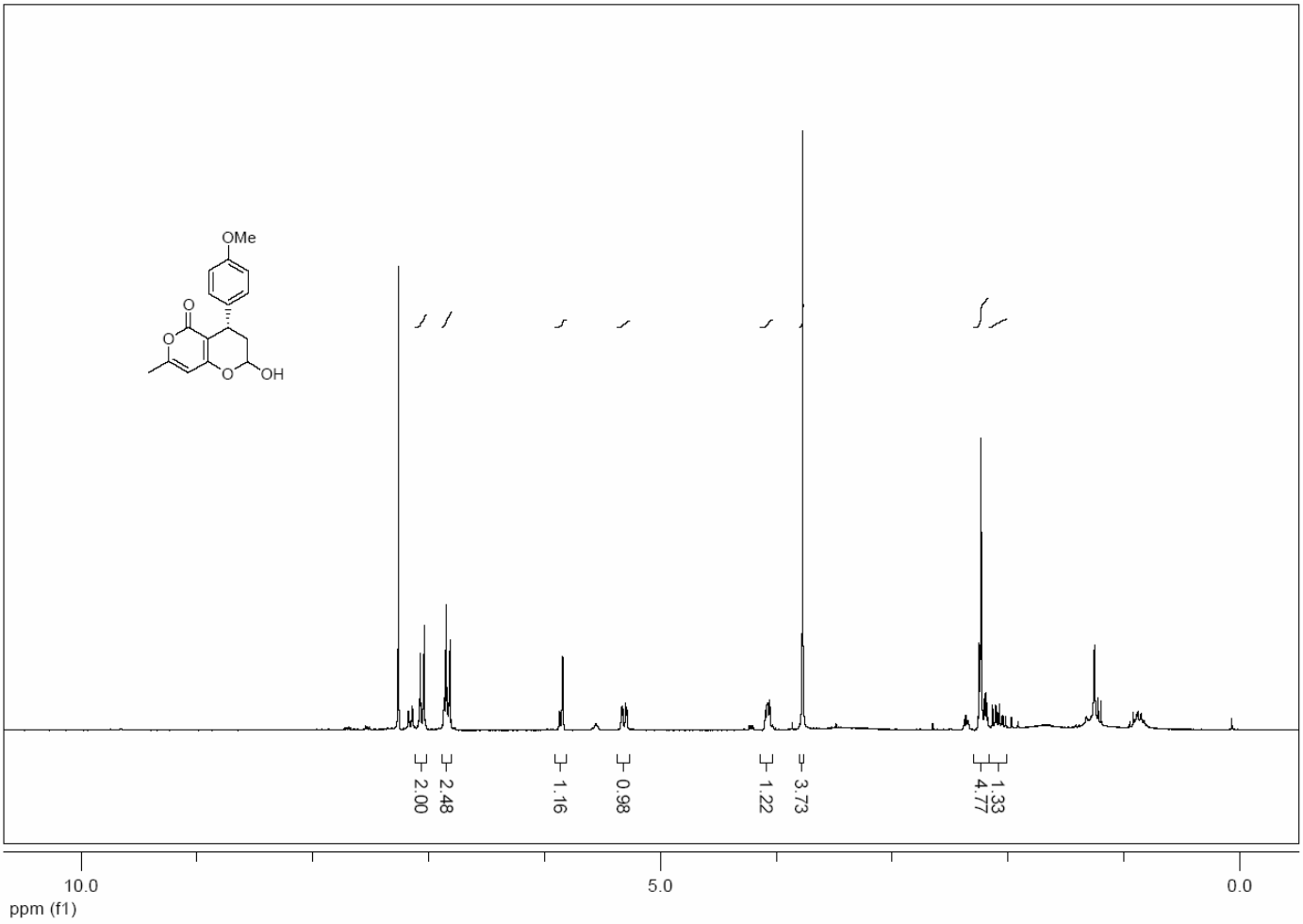

164.383
162.869
160.573
102.212
100.143
93.232
77.508
77.000
76.492
33.488
31.824
29.543
29.267
29.224
27.001
22.629
19.725
14.074
C7H15
O
O
O
OH
200
150
100
50
0
ppm (f1)

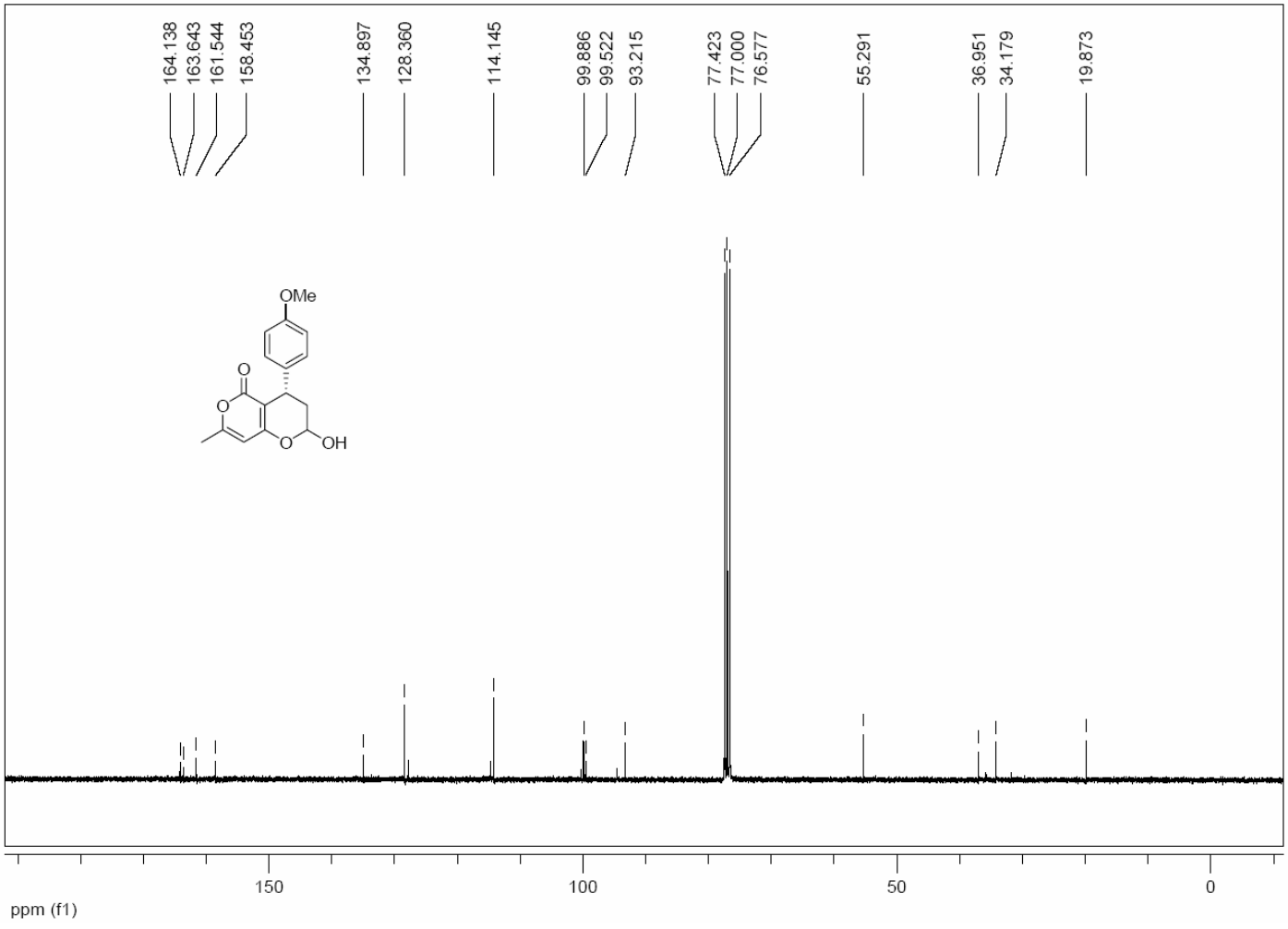

O
$C_{10}H_{21}$
O
O
OH
1.00
0.84
0.77
0.86
4.00
2.41
17.07
3.21
10.0
5.0
0.0
ppm (f1)

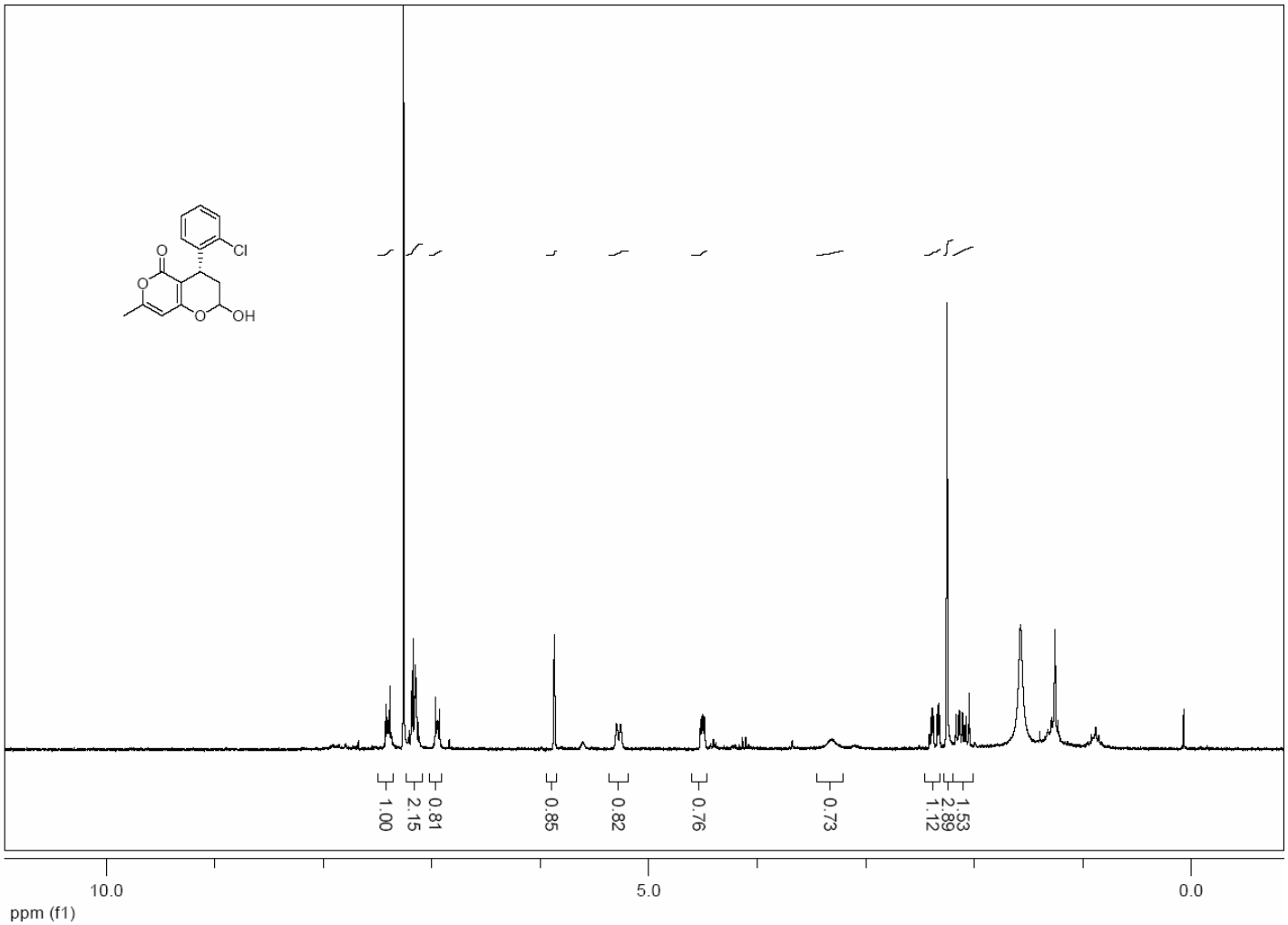

164.330
162.808
160.580
102.221
100.109
93.215
77.508
77.000
76.492
33.499
31.894
31.855
29.624
29.604
29.317
29.224
27.015
22.672
19.732
14.105
O
$C_{10}H_{21}$
O
O
OH
200
150
100
50
0
ppm (f1)

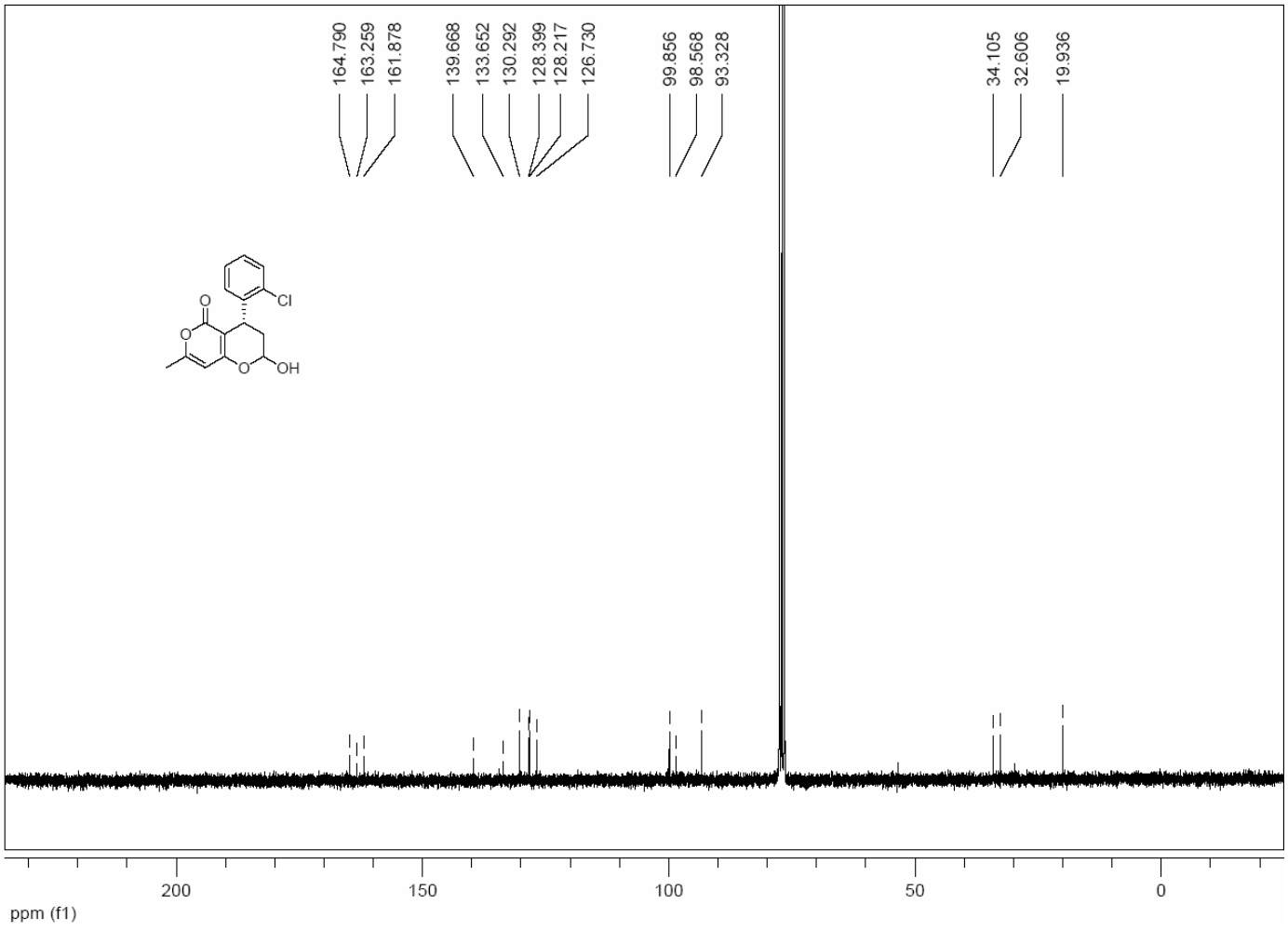

O
O
O
OH
5.00
0.88
0.79
1.26
2.95
1.82
10.0
5.0
0.0
ppm (f1)

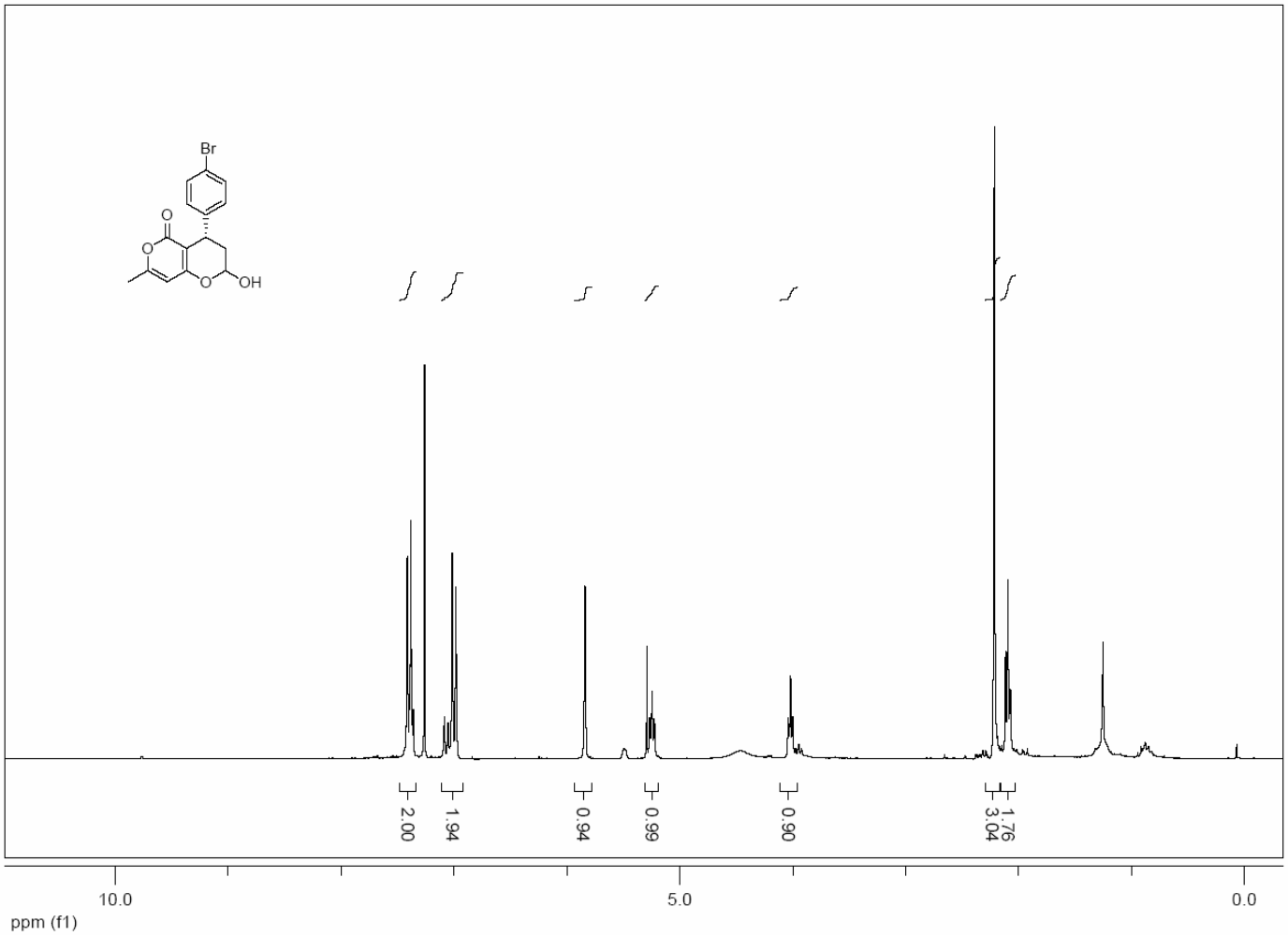

164.695
164.031
161.504
142.802
128.901
128.584
127.333
126.757
126.718
100.157
99.095
93.201
36.554
34.875
19.827
O
O
O
OH
200
150
100
50
0
ppm (f1)

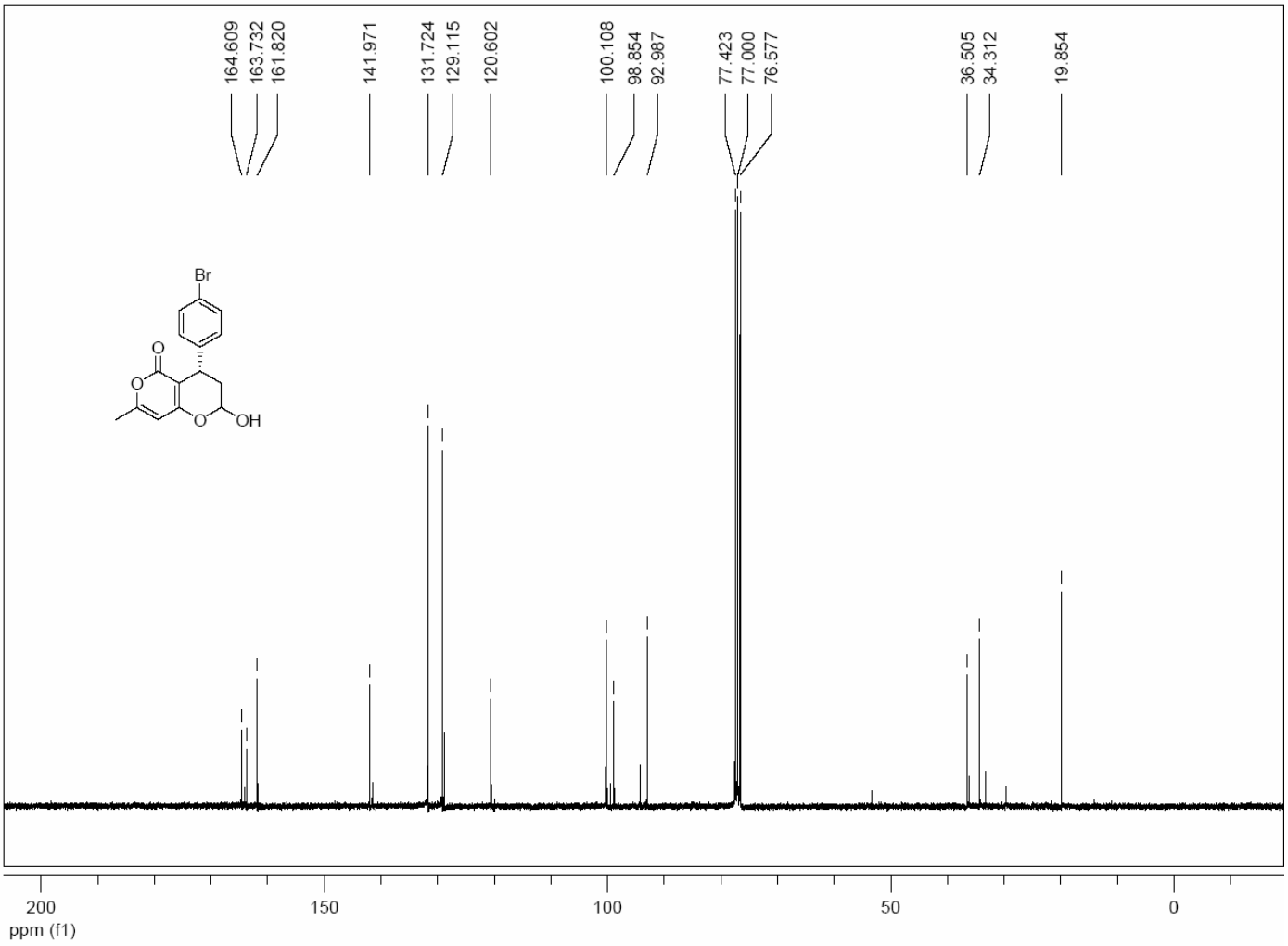

OMe
O
O
O
OH
2.00
2.48
1.16
0.98
1.22
3.73
4.77
1.33
10.0
5.0
0.0
ppm (f1)

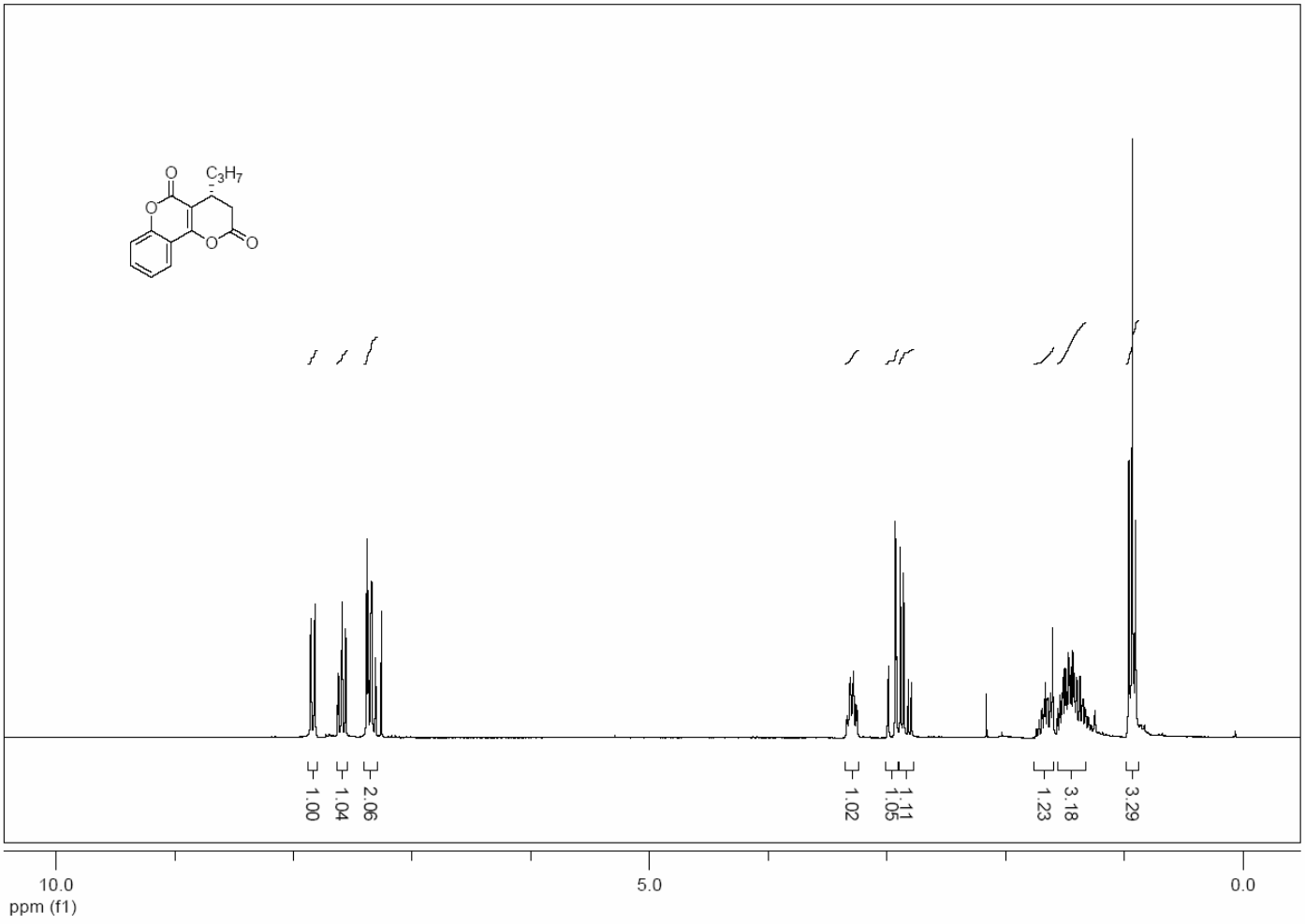

164.138
163.643
161.544
158.453
134.897
128.360
114.145
99.886
99.522
93.215
77.423
77.000
76.577
55.291
36.951
34.179
19.873
OMe
O
O
O
OH
150
100
50
0
ppm (f1)

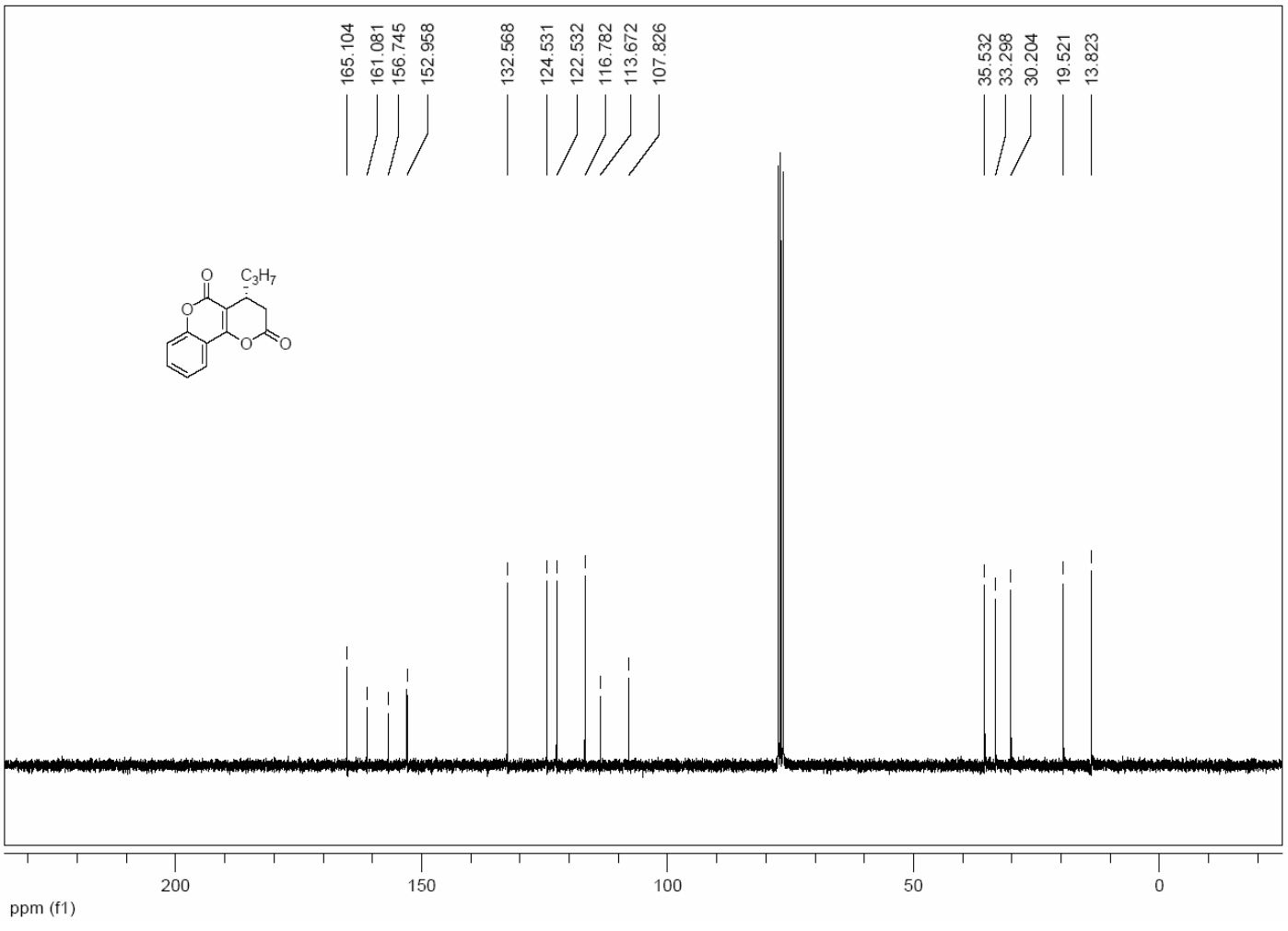

Cl
O
O
O
OH
1.00
2.15
0.81
0.95
0.82
0.76
0.73
1.12
2.89
1.53
10.0
5.0
0.0
ppm (f1)

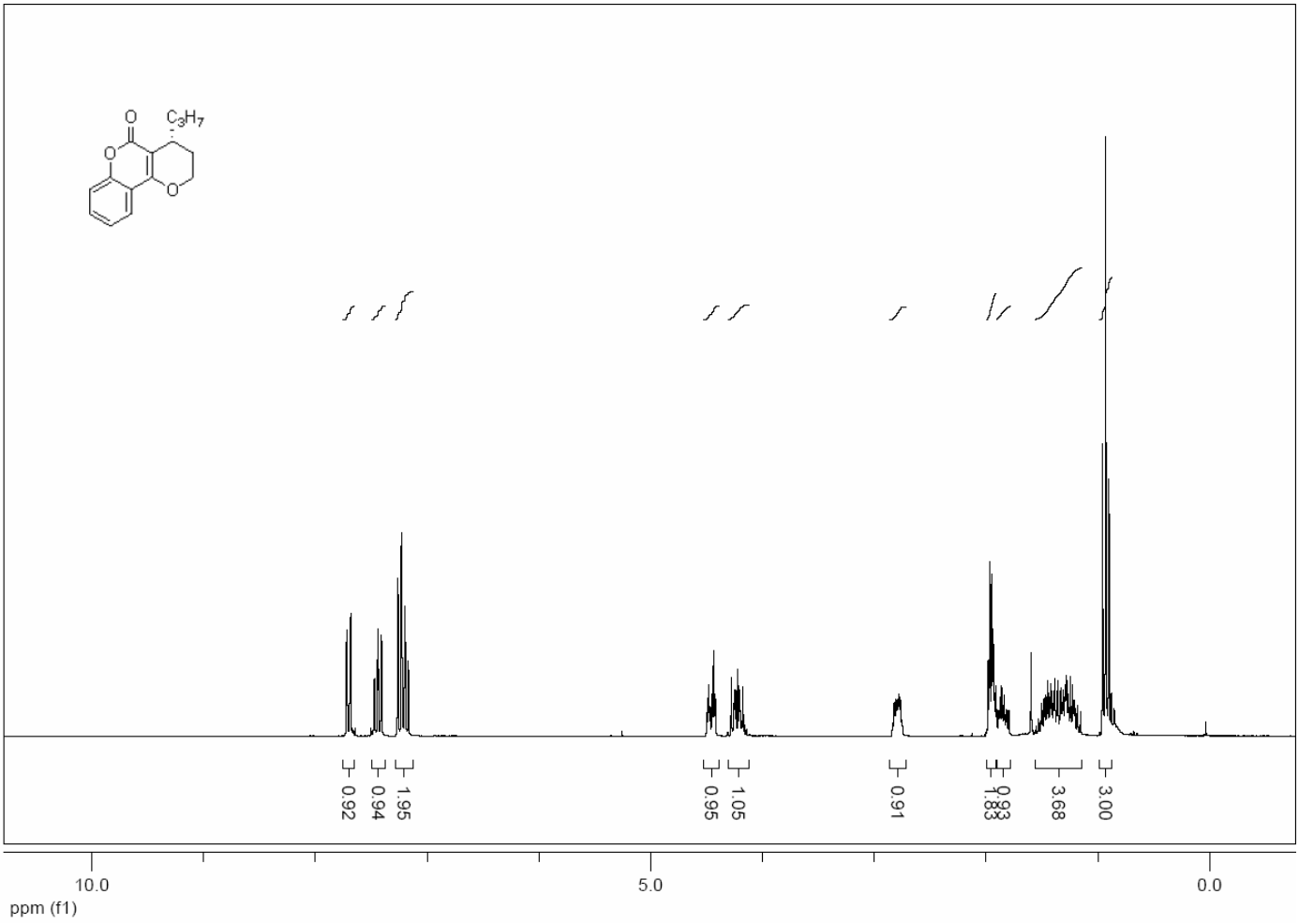

164.790
163.259
161.878
139.668
133.652
130.292
128.399
128.217
126.730
99.856
98.568
93.328
34.105
32.606
19.936
Cl
O
O
O
OH
200
150
100
50
0
ppm (f1)

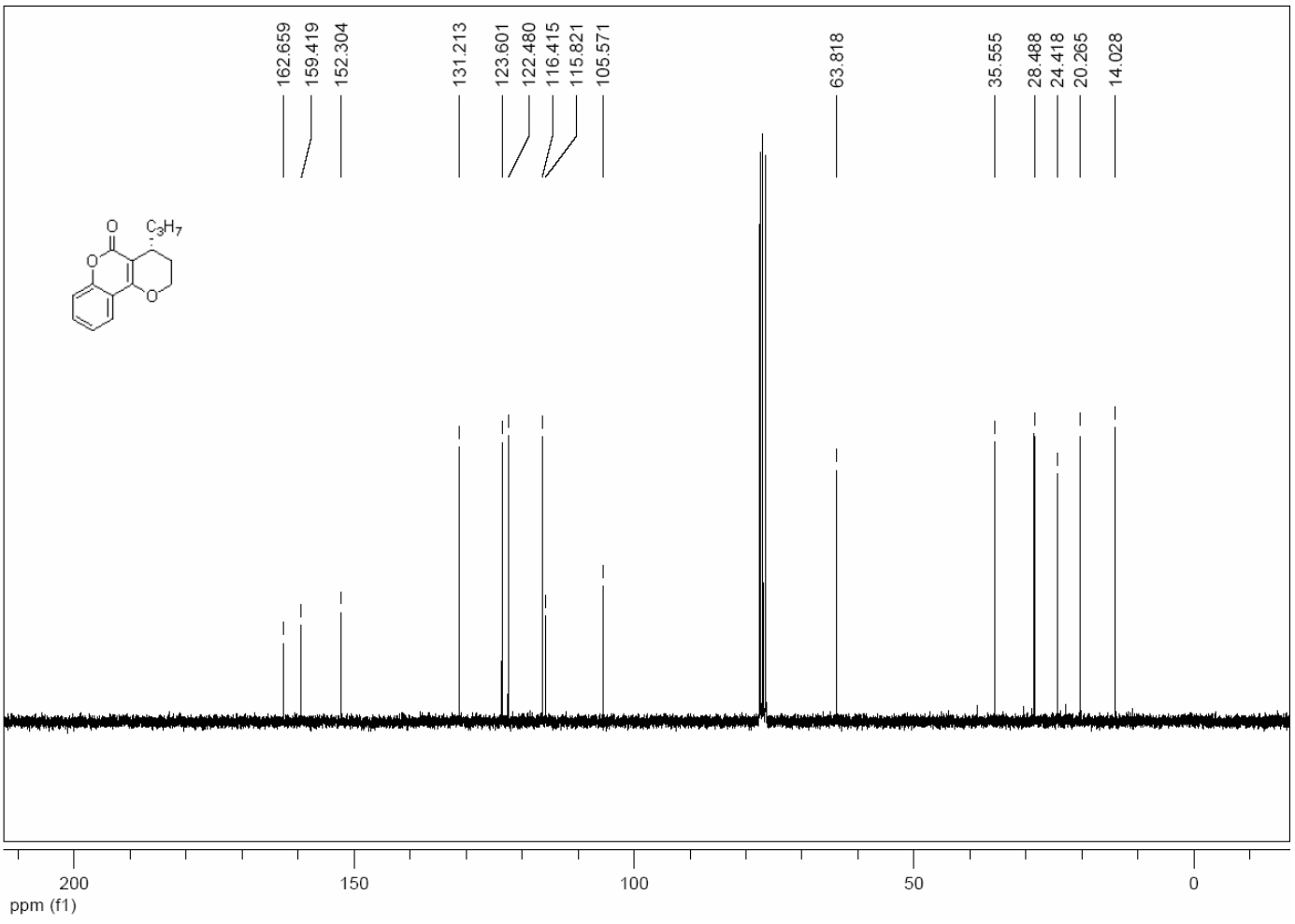

Br
O
O
O
OH
2.00
1.94
0.94
0.99
0.90
3.04
1.76
10.0
5.0
0.0
ppm (f1)

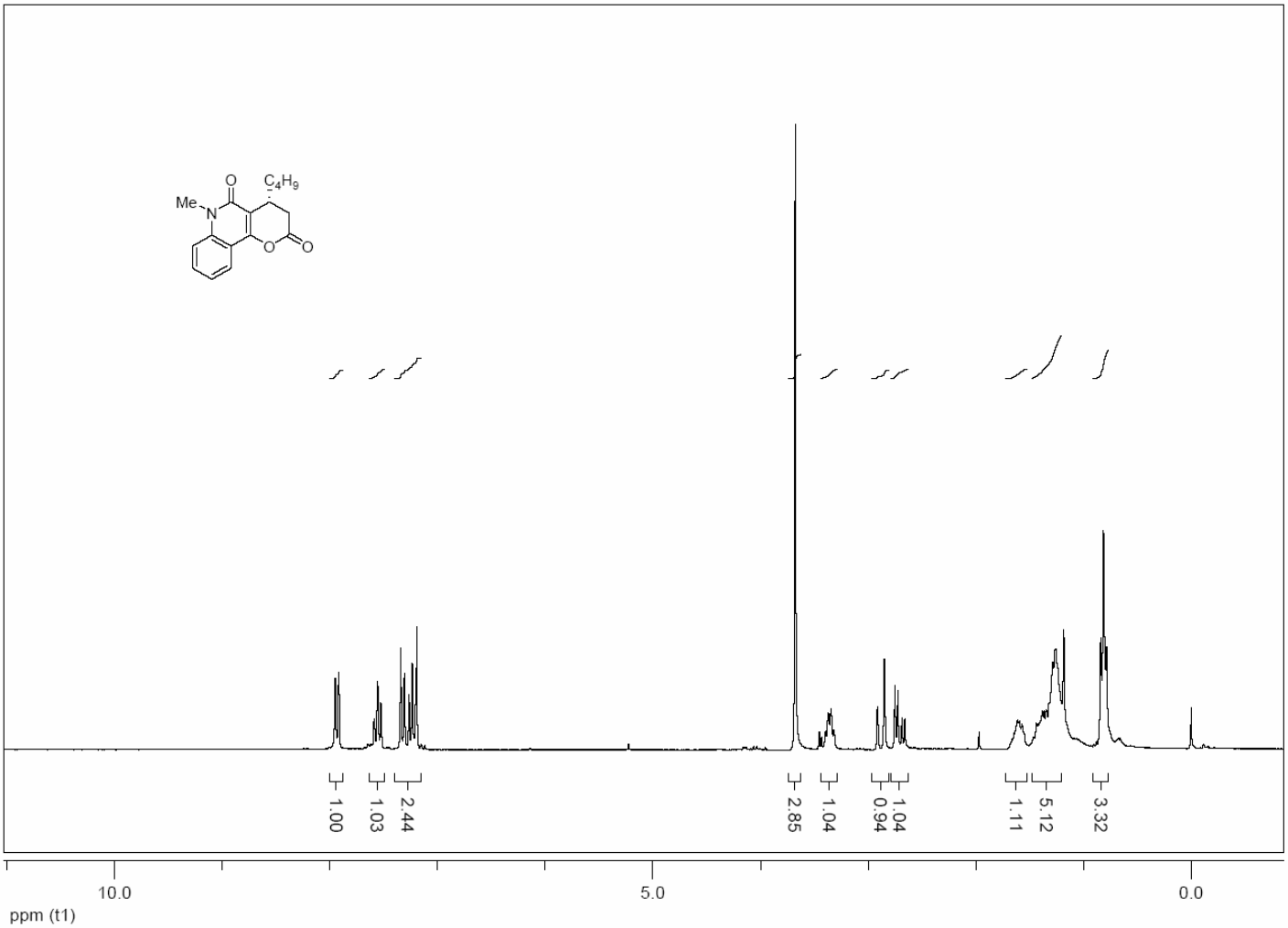

164.609
163.732
161.820
141.971
131.724
129.115
120.602
100.108
98.854
92.987
77.423
77.000
76.577
36.505
34.312
19.854
Br
O
O
O
OH
200
150
100
50
0
ppm (f1)

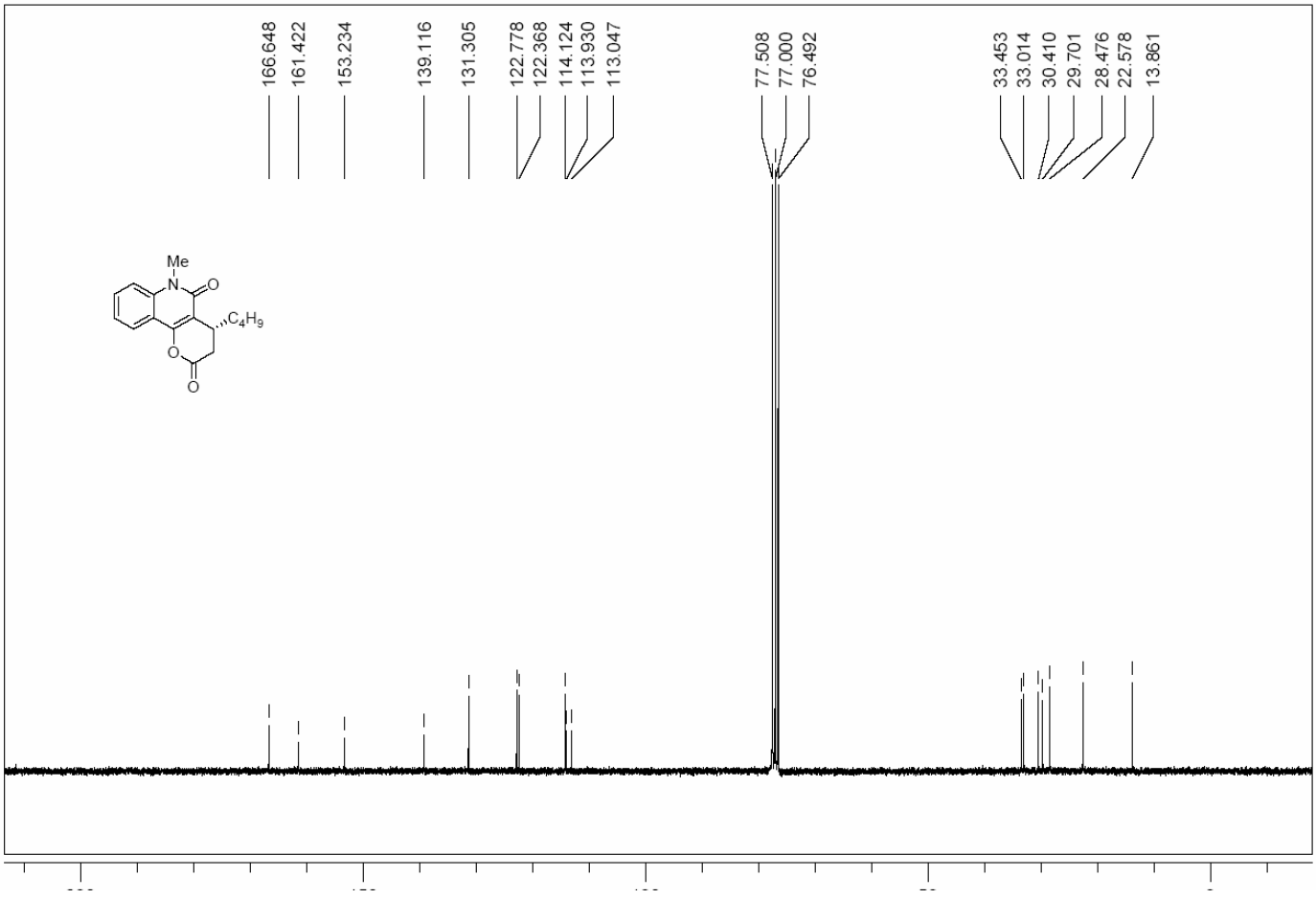

C3H7
1.00
1.04
2.90
1.02
1.11
1.05
1.23
3.18
3.29
10.0
5.0
0.0
ppm (f1)

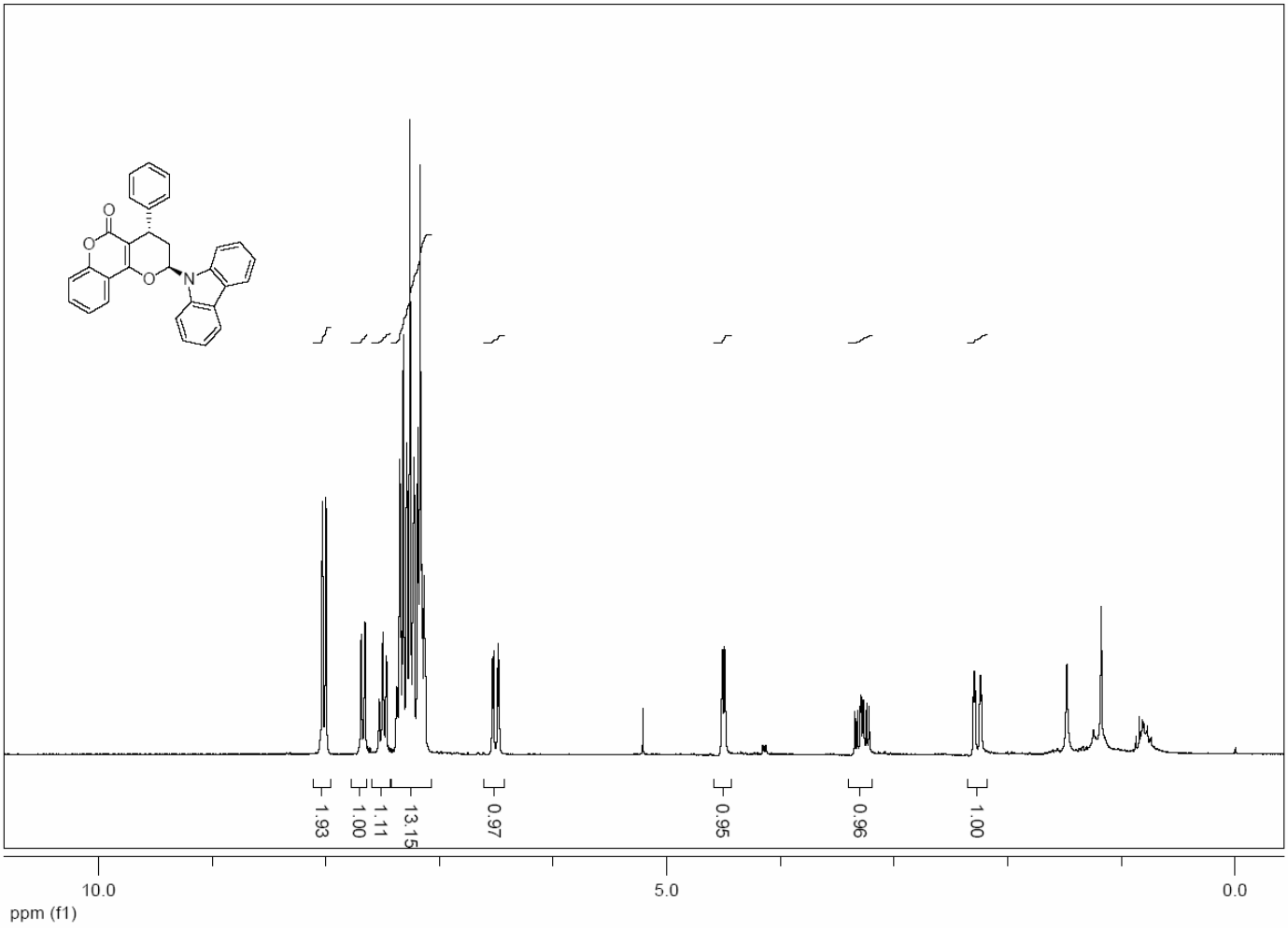

165.104
161.081
156.745
152.958
132.568
124.531
122.532
116.782
113.672
107.826
35.532
33.298
30.204
19.521
13.823
C3H7
200
150
100
50
0
ppm (f1)

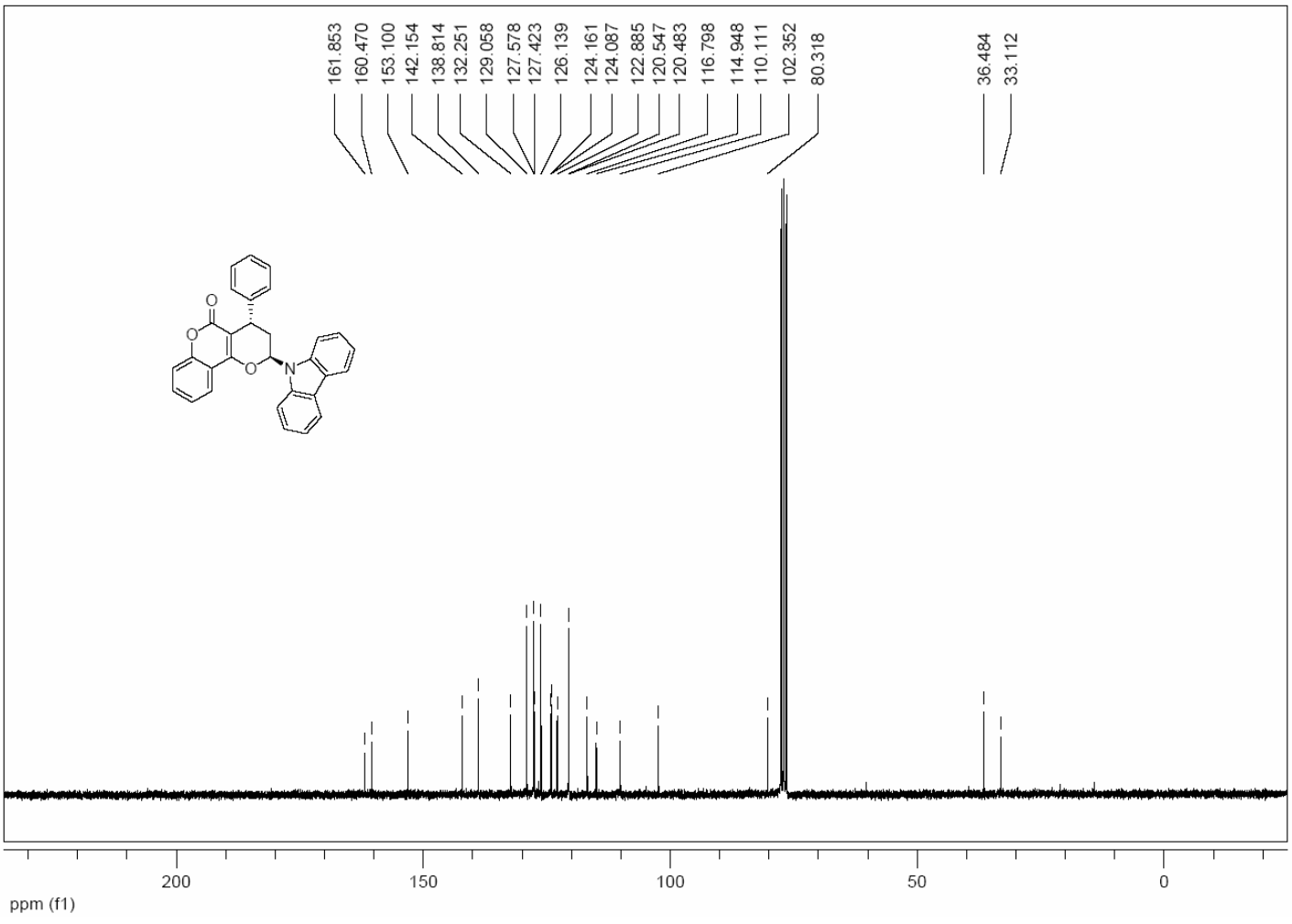

O
O
O
C_3H_7
0.92
0.94
1.95
0.95
1.05
0.91
0.93
1.83
3.68
3.00
10.0
5.0
0.0
ppm (f1)


162.659
159.419
152.304
131.213
123.601
122.480
116.415
115.821
105.571
63.818
35.555
28.488
24.418
20.265
14.028
O
O
O
C_3H_7
200
150
100
50
0
ppm (f1)


Me
N
O
O
O
C_4H_9
1.00
1.03
2.44
2.85
1.04
0.94
1.04
1.11
5.12
3.32
10.0
5.0
0.0
ppm (t1)


166.648
161.422
153.234
139.116
131.305
122.778
122.368
114.124
113.930
113.047
77.508
77.000
76.492
33.453
33.014
30.410
29.701
28.476
22.578
13.861
Me
N
O
O
O
C_4H_9


1.93
1.00
1.11
13.15
0.97
0.95
0.96
1.00
10.0
5.0
0.0
ppm (f1)


161.853
160.470
153.100
142.154
138.814
132.251
129.058
127.578
127.423
126.139
124.161
124.087
122.885
120.547
120.483
116.798
114.948
110.111
102.352
80.318
36.484
33.112
200
150
100
50
0
ppm (f1)