Supporting Information

Garcinia Xanthones as Orally Active Antitumor Agents

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Genaral Information

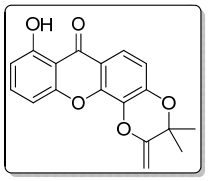
All reagents were purchased from commercial sources and, unless otherwise noted, were used without further purification. Organic solutions were concentrated in a rotary evaporator (Büchi Rotavapor) below 45 °C (70 °C for DMF) under reduced pressure. Reactions were monitored by thin-layer chromatography (TLC) on 0.25 mm silica gel plates (GF254) and visualized under UV light. Silica gel (60 Å, 300-400 mesh) was used for flash column chromatography. Melting points were determined with a Melt-Temp II apparatus and were reported without correction. IR spectra were recorded on a Nicolet iS10 Avatar FT-IR spectrometer using KBr film. The ^{1}H NMR and ^{13}C NMR spectra were measured on a Bruker AV-300 or Bruker AV-500 instrument using deuterated solvents with tetramethylsilane (TMS) as internal standard. Multiplicities were defined as s (singlet), d (doublet), t (triplet) or m (multiplet). EI-mass spectra were recorded with a Shimadzu GCMS-2010 mass spectrometer. High resolution mass spectra (HRMS) were recorded on a Water Q-Tof micro mass spectrometer. The purity (≥ 95%) of the compounds is verified by the HPLC study performed on Amethyst C18-P (4.6×150 mm, 5 μm, Merck) column using a mixture of solvent methanol/water or acetonitrile/water at the flow rate of 2ml/min and peak detection at 240 nm under UV.

Experimental and compound characterization

1,5-Dihydroxy-3,6-bis(2-methylbut-3-yn-2-yloxy)-9*H*-xanthen-9-one (7a). To a stirring solution of compound **7** (1.04 g, 4 mmol) in acetonitrile (20 mL) and tetrahydrofuran (10 mL) was added KI (1.99 g, 12 mmol), K_2CO_3 (1.82 g, 13.2 mmol), 3-chloro-3-methylbut-1-yne (2.23 mL, 20 mmol) and CuI (76 mg, 0.4 mmol) and stirred at 35 °C for 6 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford the titled compound as a light yellow solid (643 mg, 41%). m.p.: 149-150 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.75 (s, 6H, 2 × -CH_3), 1.79 (s, 6H, 2 × -CH_3), 2.71 (d, 2H, 2 × -C≡CH), 5.84 (s, 1H, Ar-OH), 6.66 (d, J = 2.4 Hz, 1H, Ar-H), 6.91 (d, J = 2.4 Hz, 1H, Ar-H), 7.54 (d, J = 9.0 Hz, 1H, Ar-H), 7.76 (d, J = 9.0 Hz, 1H, Ar-H), 12.84 (s, 1H, Ar-OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 29.6, 29.7, 72.8, 75.0, 75.3, 75.4, 84.6, 84.7, 97.6, 101.8, 104.1, 115.6, 116.0, 116.8, 136.4, 145.1, 147.3, 157.1, 162.9, 163.1, 180.6; IR (cm^{-1}, KBr film): 3291, 2989, 2935, 1649, 1604, 1571, 1442, 1326, 1287, 1202, 1131, 1095, 1061, 869, 817, 688; EI-MS *m/z*: 392[M]$^{+}$(6), 311(38), 260(36), 58(100); Anal. ($C_{23}H_{20}O_6$) C, H. Calc: 70.40, 5.14; found: 70.29, 5.12. This reaction also afforded cyclized product **7c** as a yellow oil (125 mg, 8%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.59 (s, 6H, 2 × -CH_3), 1.76 (s, 6 H, 2 × -CH_3), 2.74 (s, 1H, -C≡CH), 4.66 (d, J = 2.1 Hz, 1H, -C=C$\underline{H_2}$), 4.98 (d, J = 2.1 Hz, 1H, -C=C$\underline{H_2}$), 6.68 (d, J = 2.3 Hz, 1H, Ar-H), 6.87-6.92 (m, 2H, Ar-H), 7.80 (d, J = 8.8 Hz, 1H, Ar-H), 12.87 (s, 1H, Ar-OH); ^{13}C NMR (75 MHz, $CDCl_3$): 25.1, 29.7, 72.8, 74.0, 75.3, 84.8, 91.4, 97.7, 102.0, 104.2, 113.9, 115.4, 118.7, 129.5, 145.0, 147.0, 155.0, 156.9, 162.8, 162.9, 180.2; IR (cm^{-1}, KBr film): 3330, 3005, 2933, 1740, 1650, 1605, 1580, 1452, 1364, 1311, 1279, 1221, 1122, 1085, 870, 821; EI-MS *m/z:* 392[M]$^{+}$(6), 311(20), 260(10), 98(100).

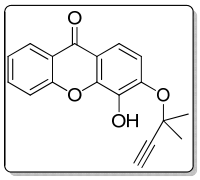
1,5-Dihydroxy-6-(2-methylbut-3-yn-2-yloxy)-9*H*-xanthen-9-one (8a). To a stirring solution of compound **8** (400 mg, 1.64 mmol) in acetonitrile (10 mL) and THF (5 mL) was added KI (544 mg, 3.28 mmol), K_2CO_3 (498 mg, 3.61 mmol), 3-chloro-3-methylbut-1-yne (0.46 mL, 4.10 mmol) and CuI (30 mg, 0.16 mmol) and stirred at 35 °C for 6 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford the titled compound as a light yellow solid (108 mg, 42%). m.p.: 142-144 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.60 (s, 6H, 2 × -CH_3), 2.72 (s, 1H, -C≡CH), 5.85 (s, 1H, Ar-OH), 6.80 (d, J = 8.1 Hz, 1H, Ar-H), 7.03 (d, J = 8.1 Hz, 1H, Ar-H), 7.56-7.61 (m, 2H, 2 × Ar-H), 7.80 (d, J = 9.0 Hz, 1H, Ar-H), 12.72 (s, 1H, Ar-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 29.2, 74.5, 75.0, 84.1, 106.7, 108.1, 110.1, 115.1, 115.6, 116.2, 135.9, 136.1, 144.7, 147.2, 155.8, 161.4, 181.4; IR (cm^{-1}, KBr film): 3392, 3280, 2956, 1741, 1646, 1605, 1576, 1464, 1381, 1275, 1260, 1229, 1067, 764, 750; EI-MS *m/z*: 310[M]$^{+}$(13),

244(100), 67(32). This reaction also afforded cyclized product **8c** as a yellow oil (10 mg, 2%). ^{1}H NMR

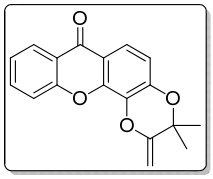


(300 MHz, $CDCl_3$): δ 1.60 (s, 6H, 2 × -CH_3), 4.67 (d, J = 2.3 Hz, 1H, -C=C$\underline{H}_2$), 4.99 (d, J = 2.3 Hz, 1H, -C=C$\underline{H}_2$), 6.81 (d, 1H, J = 4.1 Hz, Ar-H), 6.91 (d, 1H, J = 8.9 Hz, Ar-H), 7.01-7.06 (m, 1H, Ar-H), 7.56-7.61 (m, 1H, Ar-H), 7.83(d, J = 8.9 Hz, 1H, Ar-H), 12.77 (s, 1H, Ar-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 25.1, 74.2, 91.6, 107.1, 107.2, 108.6, 110.8, 112.6, 114.2, 115.3, 118.8, 129.9, 147.4, 154.9, 156.1, 161.9, 181.4; IR (cm^{-1}, KBr film): 3333, 2985, 2932, 1740, 1646, 1609, 1586, 1462, 1384, 1307, 1276, 1234, 1162, 1081, 795, 765,750; EI-MS *m/z*: 310[M]$^+$(54), 244(66), 43(100).

4-Hydroxy-3-(2-methylbut-3-yn-2-yloxy)-9*H*-xanthen-9-one (9a). 9a was prepared from **9** according

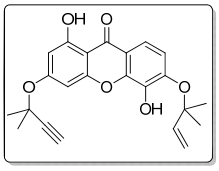


to the synthetic procedure described for **8a** as a yellow solid. Yield: 33%; m.p.: 171-173 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.79 (s, 6H, 2 × -CH_3), 2.70 (s, 1H, -C≡CH), 5.89 (s, 1H, Ar-OH), 7.39 (t, J = 7.2 Hz, 1H, Ar-H), 7.53-7.61 (m, 2H, 2 × Ar-H), 7.70-7.73 (m, 1H, Ar-H), 7.86 (d, J = 8.9 Hz, 1H, Ar-H), 8.34 (d, J = 8.8 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 29.7, 74.9, 75.3, 84.8, 115.7, 116.8, 118.1, 118.2, 121.6, 124.0, 126.7, 134.6, 136.4, 145.3, 147.0, 156.2, 176.7; IR (cm^{-1}, KBr film): 3300, 2973, 1726, 1641, 1599, 1576, 1464, 1381, 1275, 1255, 1063, 762, 748; EI-MS *m/z*: 294[M]$^+$(15), 253(29), 241(18), 58(100), 55(34). This reaction also formed cyclized product **9c** as a yellow oil. Yield:



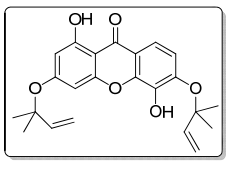
3%; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.53 (s, 6H, 2 × -CH_3), 4.59 (d, J = 2.3 Hz, 1H, -C=C$\underline{H}_2$), 4.91 (d, J = 2.3 Hz, 1H, -C=C$\underline{H}_2$), 6.83 (d, J = 8.8 Hz, 1H, Ar-H), 7.30-7.34 (m, 1H, Ar-H), 7.63-7.65 (m, 1H, Ar-H), 7.83 (d, J = 8.9 Hz, 1H, Ar-H), 8.27 (d, J = 7.2 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 24.1, 72.9, 90.3, 112.9, 115.7, 117.0, 118.5, 120.7, 123.1, 125.7, 129.0, 133.4, 144.2, 145.7, 154.1, 155.0, 175.2; IR (cm^{-1}, KBr film): 2975, 2922, 1726, 1640, 1599, 1569, 1465, 1382, 1280, 1260, 1152, 1071, 760,749; EI-MS *m/z*: 294[M]$^+$(79), 279(29), 228(100), 67(43).

1,5-Dihydroxy-6-(2-methylbut-3-en-2-yloxy)-3-(2-methylbut-3-yn-2-yloxy)-9*H*-xanthen-9-one (10).



To a stirring solution of compound **7a** (200 mg, 0.51 mmol) in ethanol (20 mL) was added Lindlar' catalyst (Pd, 10 wt% on barium sulfate, 20 mg) under hydrogen (1 atm) and stirred at 25 °C for 20 min. The reaction mixture was filtered and the filtration was concentrated.The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford the titled compound as a yellow oil (244 mg, 82%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.56 (s, 6H, 2 × -CH_3), 1.73 (s, 6H, 2 × -CH_3), 2.69 (s, 1H, -C≡CH), 5.24-5.32 (m, 2H, -CH=C$\underline{H}_2$), 6.00 (s, 1H, Ar-OH), 6.12-6.21 (m, 1H, -C$\underline{H}$=CH_2), 6.64 (d, J = 2.2 Hz, 1H, Ar-H), 6.89 (d, J = 2.2 Hz, 1H, Ar-H), 7.12 (d, J = 8.9 Hz, 1H, Ar-H), 7.65 (d, J = 8.9 Hz, 1H, Ar-H), 12.85 (s, 1H, Ar-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.9, 29.2, 72.3, 74.8, 82.4, 84.2, 97.1, 101.3, 103.5, 114.5, 114.8, 115.3, 115.7, 135.6, 142.2, 144.3, 147.5, 156.6, 162.3, 162.5, 180.1; IR (cm^{-1}, KBr film): 3398, 3298, 2925, 2861, 1740, 1650, 1604, 1573, 1443, 1325, 1277, 1204, 1129, 1090, 762, 750; EI-MS *m/z*: 364[M]$^+$(50), 379(100), 67(51).

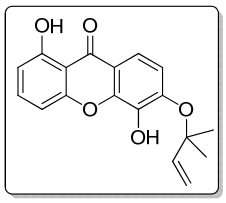
1,5-Dihydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (12). To a stirring solution of



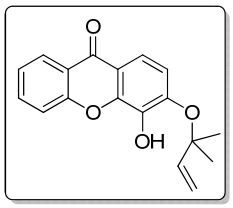
compound **7a** (200 mg, 0.51 mmol) in ethanol (20 mL) was added Lindlar' catalyst (Pd, 10 wt% on barium sulfate, 20 mg) under hydrogen (1 atm) and stirred at 25 °C for 40 min. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford the titled compound as a yellow solid (193 mg, 90%). m.p.: 125-127 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.56 (s, 6H, 2 × -CH_3), 1.58 (s, 6H, 2 × -CH_3), 5.27-5.29 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.15-6.17 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.42 (d, J = 2.2 Hz, 1H, Ar-H), 6.60 (d, J = 2.2 Hz, 1H, Ar-H), 7.11 (d, J = 8.9 Hz, 1H, Ar-H), 7.65 (d, J = 8.9 Hz, 1H, Ar-H), 12.82 (s, 1H, Ar-OH); IR (cm^{-1}, KBr film): 3441, 2960, 2924, 2854, 1649, 1598, 1439, 1283, 1261, 1124, 1104, 1008, 803, 764, 699; EI-MS *m/z*: 396[M]$^+$(92), 381(65), 341(100), 43(71); Anal. ($C_{23}H_{24}O_6$) C, H. Calc: 69.68, 6.10; found: 69.28, 6.08.

Compound **14** and **16** were prepared from compound **8a** and **9a**, respectively, according to the synthetic procedure described for **10**.

1,5-Dihydroxy-6-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (14). Yield: 89%; Yellow solid, m.p.: 150-152 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.61 (s, 6H, 2 × -CH_3), 5.27-5.35 (m, 2H, -CH=C$\underline{H}_2$), 5.88 (s, 1H, Ar-OH), 6.14-6.23 (m, 1H, -C$\underline{H}$=CH_2), 6.79 (d, J = 9.0 Hz, 1H, Ar-H), 7.03 (d, J = 9.0 Hz, 1H, Ar-H), 7.17 (d, J = 9.0 Hz, 1H, Ar-H), 7.58 (t, J = 9.0 Hz, 1H, Ar-H), 7.71(d, J = 9.0 Hz, 1H, Ar-H), 12.76 (s, 1H, Ar-OH); IR (cm^{-1}, KBr film): 3333, 2925, 2855, 1740, 1651, 1606, 1461, 1343, 1275, 1225, 1122, 1060, 763, 745; EI-MS *m/z*: 312[M]$^+$(22), 257(32), 43(100).

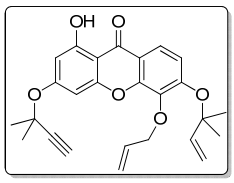


4-Hydroxy-3-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (16). Yield: 87%; white solid, m.p.: 100-102 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.60 (s, 6H, 2 × -CH_3), 5.26-5.35 (m, 2H, -CH=C$\underline{H}_2$), 5.90 (s, 1H, Ar-OH), 6.14-6.24 (m, 1H, -C$\underline{H}$=CH_2), 7.15 (d, J = 8.9 Hz, 1H, Ar-H), 7.37 (t, J = 7.5 Hz, 1H, Ar-H), 7.60 (d, J = 8.1 Hz, 1H, Ar-H), 7.69-7.72 (m, 2H, Ar-H), 8.32 (d, J = 8.1 Hz, 1H, Ar-H); IR (cm^{-1}, KBr film): 3375, 2924, 2854, 1740, 1645, 1603, 1462, 1384, 1233, 1073, 807, 733; EI-MS *m/z*: 294[M]$^+$(4), 122(36), 105(59), 57(100).

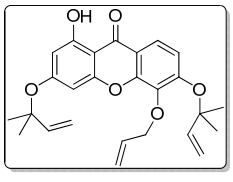


General procedure for allylation. To a stirring solution of required hydroxyxathone (**10**, **12**, **14** and **16**, 1 mmol) in acetone (20 mL) was added K_2CO_3 (276 mg, 2 mmol) and allyl bromide (242 mg, 2 mmol) and stirred at 35 °C for 4 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford allylated product **11**, **13**, **15** and **17**, respectively.

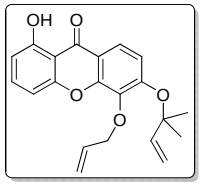
5-Allyloxy-1-hydroxy-6-(2-methylbut-3-en-2-yloxy)-3-(2-methylbut-3-yn-2-yloxy)-9*H*-xanthen-9-one (11). Yield: 80%; yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.58 (s, 6H, 2 × -CH_3), 1.75 (s, 6H, 2 × -CH_3), 2.71 (s, 1H, -C≡CH), 4.64-4.69 (m, 2H, -OCH_2-), 5.19-5.44 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.12-6.23 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.70 (d, J = 2.2 Hz, 1H, Ar-H), 6.83 (d, J = 2.0 Hz, 1H, Ar-H), 7.12 (d, J = 9.0 Hz, 1H, Ar-H), 7.84 (d, J = 9.0 Hz, 1H, Ar-H), 12.85 (s, 1H, Ar-OH); IR (cm^{-1}, KBr film): 3274, 3059, 2962, 2824, 1739, 1634, 1600, 1505, 1446, 1275, 1225, 1001, 764, 750; EI-MS *m/z*: 434[M]$^+$(45), 175(81), 69(100).



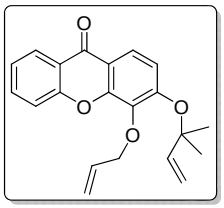
5-Allyloxy-1-hydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (13). Yield: 79%; yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.57 (s, 12H, 4 × -CH_3), 4.66 (d, J = 5.9 Hz, 2H, -OCH_2-), 5.18-5.31 (m, 6H, 3 × -CH=C$\underline{H}_2$), 6.11-6.17 (m, 3H, 3 × -C$\underline{H}$=CH_2), 6.43 (d, J = 2.2 Hz, 1H, Ar-H), 6.57 (d, J = 2.1 Hz, 1H, Ar-H), 7.10 (d, J = 9.0 Hz, 1H, Ar-H), 7.82 (d, J = 9.0 Hz, 1H, Ar-H), 12.80 (s, 1H, Ar-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 27.2, 27.4, 74.7, 81.3, 82.4, 97.9, 101.7, 103.6, 104.2, 104.3, 116.2, 117.2, 118.2, 120.1, 134.0, 138.6, 143.3, 143.5, 151.0, 155.3, 157.1, 162.8, 164.0, 180.5; IR (cm^{-1}, KBr film): 3339, 2925, 2850, 1741, 1640, 1599, 1450, 1332, 1269, 1187, 1131, 1063, 824, 736; EI-MS *m/z*: 436[M]$^+$(8), 69(100), 55(43).



5-Allyloxy-1-hydroxy-6-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (15). Yield: 87%; yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.59 (s, 6H, 2 × -CH_3), 4.69 (d, J = 5.8 Hz, 2H, -OCH_2-), 5.21-5.44 (m, 4H, 2 × -CH=CH_2), 6.13-6.24 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.79 (d, J = 7.8 Hz, 1H, Ar-H), 6.99 (d, J = 8.1 Hz, 1H, Ar-H), 7.14-7.19 (m, 1H, Ar-H), 7.57 (t, J = 8.1 Hz, 1H, Ar-H), 7.87 (d, J = 9.0 Hz, 1H, Ar-H), 12.75(s, 1H, OH); IR (cm^{-1}, KBr film): 3416, 2974, 2926, 1776, 1715, 1660, 1589, 1464, 1381, 1272, 1260, 1125, 748; EI-MS *m/z*: 352[M]$^+$(6), 284(60), 243(60), 69(61), 43(100).



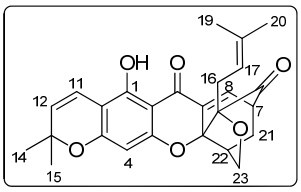
4-Allyloxy-3-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (17). Yield: 85%; light yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51 (s, 6H, 2 × -CH_3), 4.64 (d, J = 5.9 Hz, 2H, -OCH_2-), 5.11-5.38 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.08-6.17 (m, 2H, 2 × -C$\underline{H}$=CH_2), 7.06 (d, J = 9.0 Hz, 1H, Ar-$\underline{H}$), 7.29 (t, J = 7.5 Hz,



1H, Ar-H), 7.48 (d, J = 8.3 Hz, 1H, Ar-H), 7.60-7.63 (m, 1H, Ar-H), 7.87 (d, J = 9.0 Hz, 1H, Ar-H), 8.23 (d, J = 8.3 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 27.2, 74.7, 82.3, 114.2, 117.4, 117.6, 118.10, 118.15, 121.0, 121.6, 123.9, 126.6, 134.1, 134.4, 138.7, 143.5, 151.1, 155.1, 156.2, 176.6; IR (cm^{-1}, KBr film): 3091, 2985, 2932, 1741, 1645, 1601, 1568, 1462, 1440, 1381, 1275, 1233, 1125, 1079, 998, 927, 750; EI-MS *m/z*: 336[M]$^+$(4), 279(11), 206(100).

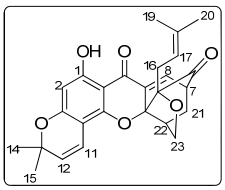
General procedure for Claisen/Diels-Alder cascade reaction. A solution of required substrate (**11**, **13**, **15** and **17**, 200 mg) in DMF (2 mL) was stirred under nitrogen at 120 °C for 2 h. The reaction mixture was then cooled to room temperature and concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford caged compound **18-19**, **20-21**, **22** and **23**, respectively.

8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (18). Yield: 31%; yellow solid, m.p.: 110-109 °C; ^{1}H



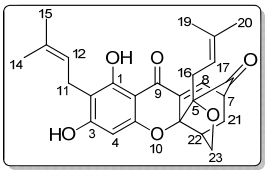
NMR (300 MHz, $CDCl_3$): δ 1.18 (s, 3H, C_{19}-H), 1.39 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 1.76-1.89 (m, 2H, C_{21}-H), 2.54-2.70 (m, 3H, C_{16}-H, C_{22}-H), 3.50-3.52 (m, 1H, C_7-H), 3.87 (d, J= 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.43-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.52 (d, J = 10.2 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.28 (d, J = 6.9 Hz, 1H, C_8-H), 12.74 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.7, 25.9, 27.2, 27.83, 27.88, 28.6, 41.2, 45.2, 73.7, 76.9, 82.2, 87.7, 96.7, 100.6, 102.6, 114.7, 117.2, 125.9, 130.7, 132.9, 135.2, 158.6, 159.9, 162.2, 178.5, 199.5; IR (cm^{-1}, KBr film): 3452, 2931, 2852, 1740, 1645, 1633, 1599, 1461, 1390, 1325, 1161, 1151, 1065, 828, 755; EI-MS *m/z*: 434[M]$^+$(23), 385(62), 133(100), 69(45); HRMS (ESI): calcd. for $C_{26}H_{26}O_6$ [M + H]$^+$ 435.1808, found 435.1803; HPLC (60% acetonitrile in water): t_R = 10.0 min, 99.0%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (19). Yield: 46%; yellow solid, m.p.: 102-104 °C; ^{1}H



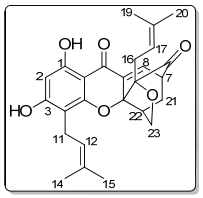
NMR (300 MHz, $CDCl_3$): δ 1.14 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.44-1.45 (m, 6H, C_{14}-H, C_{15}-H), 1.71-1.91 (m, 2H, C_{21}-H), 2.54-2.69 (m, 3H, C_{16}-H, C_{22}-H), 3.52-3.53 (m, 1H, C_7-H), 3.92 (d, J= 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.44 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.9 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H$_\alpha$), 5.55 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.01 (s, 1H, C_2-H), 6.56 (d, J = 10.2 Hz, 1H, C_{11}-H), 7.30 (d, J = 6.9 Hz, 1H, C_8-H), 12.56 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.1, 25.5, 27.7, 28.4, 28.5, 29.1, 41.9, 45.7, 74.3, 78.4, 82.7, 88.5, 97.8, 101.1, 101.7, 115.1, 117.4, 127.0, 31.4, 133.2, 135.8, 154.5, 162.8, 164.8, 179.0, 199.8; IR (cm^{-1}, KBr film): 3279, 2968, 2921, 1737, 1644, 1634, 1588, 1435, 1380, 1333, 1128, 826; EI-MS *m/z*: 434[M]$^+$(7), 169(90), 147(70), 119(60), 69(100); HRMS (ESI): calcd. for $C_{26}H_{26}O_6$ [M + H]$^+$ 435.1808, found 435.1819; HPLC (60% acetonitrile in water) : t_R = 19.7 min, 99.3%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (20). Yield: 37%; yellow solid, m.p.: 112-114 °C; ^{1}H NMR (300 MHz, $CDCl_3$):



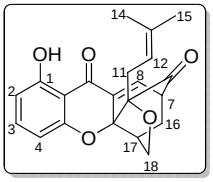
δ 1.14 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.55-2.71 (m, 5H, C_{21}-H, C_{22}-H, C_{16}-H), 3.35-3.37 (m, 2H, C_{11}-H), 3.50-3.52 (m, 1H, C_7-H), 3.88 (d, J= 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.51 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.22-5.26 (m, 1H, C_{12}-H), 6.02 (s, 1H, C_4-H), 7.28 (d, J = 7.2 Hz, 1H, C_8-H), 12.81 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.8, 18.1, 21.1, 25.77, 25.84, 29.8, 34.5, 36.6, 38.1, 45.7, 78.7, 83.3, 95.8, 101.3, 107.7, 117.1, 121.4, 133.1, 134.2, 134.9, 136.5, 159.6, 162.1, 164.7, 177.7, 198.9; IR (cm^{-1}, KBr film): 3347, 2968, 2926, 1740, 1644, 1634, 1598, 1430, 1380, 1272, 1130, 826, 742; EI-MS *m/z*: 436[M]$^+$(9), 169(86), 147(46), 69(100); HRMS (ESI): calcd. for $C_{26}H_{28}O_6$ [M + Na]$^+$ 459.1784, found 459.1796; HPLC (80% acetonitrile in water): t_R = 3.0 min, 99.2%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (21). Yield: 49%; yellow solid, m.p.: 143-144 °C; ^{1}H NMR (300 MHz, $CDCl_3$):



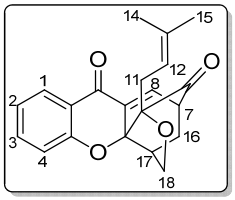
δ 1.10 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.73 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.52-2.71 (m, 5H, C_{21}-H, C_{22}-H, C_{16}-H), 3.27-3.35 (m, 2H, C_{11}-H), 3.47-3.60 (m, 1H, C_7-H), 3.91 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.38-4.41 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.3 Hz, J_2 = 7.2 Hz, 1H, C_{23}-H$_\alpha$), 5.15-5.25 (m, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 7.28 (d, J = 6.6 Hz, 1H, C_8-H), 12.41 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.9, 18.1, 21.9, 25.81, 25.86, 29.8, 34.7, 38.1, 45.7, 78.4, 83.5, 97.2, 101.5, 106.5, 111.4, 117.1, 121.4, 133.0, 134.2, 135.1, 136.5, 158.2, 163.1, 164.6, 178.2, 198.5; IR (cm^{-1}, KBr film): 3325, 2965, 2923, 1739, 1644, 1634, 1600, 1454, 1328, 1190, 1151, 1062, 832, 740; EI-MS *m/z*: 436[M]$^+$(14), 335(78), 169(100), 147(90), 69(95); HRMS (ESI): calcd. for $C_{26}H_{28}O_6$ $[M + H]^+$ 437.1964, found 437.1972; HPLC (80% acetonitrile in water): t_R = 3.8 min, 99.2%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (22). Yield: 71%; yellow solid; m.p.: 128-130 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.98 (s,



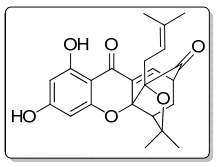
3H, C_{14}-H), 1.25 (s, 3H, C_{15}-H), 1.72-1.92 (m, 2H, C_{16}-H), 2.58-2.78 (m, 3H, C_{11}-H, C_{17}-H), 3.55 (dd, J_1 = 3.3 Hz, J_2 = 9.0 Hz, 1H, C_7-H), 3.91 (d, J = 6.0 Hz, 1H, C_{18}-H$_\beta$), 4.37-4.41 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 = 6.0 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.35-7.60 (m, 2H, C_3-H, C_8-H), 12.10 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.4, 24.9, 27.3, 28.3, 41.2, 45.4, 73.8, 81.8, 88.4, 105.7, 106.9, 109.0, 117.5, 132.1, 132.9, 135.4, 138.2, 159.0, 162.5, 180.6, 200.1; IR (cm^{-1}, KBr film): 3457, 2968, 2908, 1735, 1643, 1600, 1462, 1369, 1281, 1260, 1228, 1053, 1033, 801, 764, 750; EI-MS *m/z*: 352[M]$^+$(9), 324(78), 255(60), 227(100), 213(76), 69(64); HRMS (ESI): calcd. for $C_{21}H_{20}O_5$ $[2M + Na]^+$ 727.2519, found 727.2525; HPLC (80% acetonitrile in water): t_R = 5.8 min, 99.3%.

1-(3-Methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (23). Yield: 78%; white solid; m.p.: 114-116 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.94 (s, 3H, C_{14}-H),



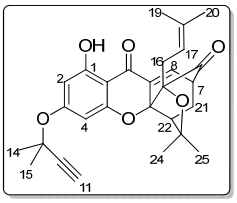
1.27 (s, 3H, C_{15}-H), 1.75-1.86 (m, 2H, C_{16}-H), 2.57-2.66 (m, 3H, C_{11}-H, C_{17}-H), 3.50-3.52 (m, 1H, C_7-H), 3.89 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.52-4.54 (m, 1H, C_{18}-H$_\alpha$), 7.02-7.07 (m, 2H, C_4-H, C_8-H), 7.28-7.31 (m, 1H, C_2-H), 7.50 (t, J = 7.6 Hz, 1H, C_3-H), 7.94 (d, J = 9.8 Hz, 1H, C_1-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.9, 25.2, 27.8, 29.0, 41.8, 45.7, 74.4, 82.9, 88.4, 118.2, 118.4, 119.3, 121.9, 127.0, 131.4, 134.4, 135.4, 136.2, 159.6, 176.2, 199.3; IR (cm^{-1}, KBr film): 2963, 2894, 1741, 1674, 1614, 1464, 1318, 1238, 1124, 751, 690; EI-MS *m/z*: 336[M]$^+$(3), 308(100), 239(35), 211(16); HRMS (ESI): calcd. for $C_{21}H_{20}O_4$ $[M + Na]^+$ 359.1259, found 359.1241; HPLC (80% acetonitrile in water): t_R = 6.0 min, 99.4%.

8,10-Hydroxy-3,3-dimethyl-10-(2-methylbut-3-yn-2-yloxy)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (28). Compound **28** was prepared according to our previously reported procedure [S1]. yellow solid; m.p: 168-170 °C; ^{1}H NMR (300 MHz, $CDCl_3$):



δ 1.11 (s, 3H), 1.23 (s, 3H), 1.33 (s, 3H), 1.61 (s, 3H), 2.26 (dd, J_1 =13.5 Hz, J_2 = 4.5 Hz, 1H), 2.37 (d, J = 9.6 Hz, 1H), 2.54 (d, J = 7.8 Hz, 2H), 3.42-3.46 (m, 2H), 4.34-4.40 (m, 1H), 5.96 (d, J = 2.1 Hz, 1H), 5.98 (d, J = 2.1 Hz, 1H), 7.35 (d, J = 6.9 Hz, 1H), 12.40 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.80, 26.20, 26.60, 30.06, 30.14, 32.22, 48.68, 49.94, 84.63, 85.46, 91.13, 107.14, 108.34, 109.40, 120.64, 135.84, 136.95, 137.30, 140.75, 159.52, 165.61, 182.36, 206.60. ESI-MS *m/z*: 336[M-H]$^-$; HRMS (ESI): calcd. for $C_{23}H_{24}O_6$ $[M + Na]^+$ 419.1471, found 419.1478; HPLC (80% acetonitrile in water): t_R = 4.1 min, 99.2%.

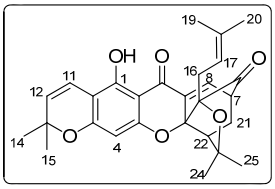
8-Hydroxy-1-(3-methylbut-2-en-yl)-3,3-dimethyl-10-(2-methylbut-3-yn-2-yloxy)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (24). To a stirring solution of compound **28** (200 mg, 0.43 mmol) in acetone (10 mL) was added KI (86 mg, 0.52 mmol), K_2CO_3 (72 mg, 0.52 mmol), 3-chloro-3-methylbut-1-yne (0.1 mL, 0.86 mmol) and CuI (8 mg, 43 μmol) and stirred at reflux for 30 min. The reaction mixture was filtered and the filtration was concentrated. The residue



was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford the titled compound as a yellow solid (170 mg, 85%). m.p.: 149-150 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{24}-H), 1.28 (s, 3H, C_{25}-H), 1.39 (s, 3H, C_{19}-H), 1.70 (s, 3H, C_{20}-H), 1.71 (s, 6H, C_{14}-H, C_{15}-H), 2.32 (dd, J_1 = 4.5 Hz, J_2 = 13.5 Hz, 1H, C_7-H), 2.42 (d, J = 9.6 Hz, 1H, C_{22}-H), 2.61 (d, J = 9.0 Hz, 2H, C_{16}-H), 2.68 (s, 1H, C_{11}-H), 3.48-3.50 (m, 2H, C_{21}-H), 4.41-4.45 (m, 1H, C_{17}-H), 6.33 (d, J = 2.1 Hz, 1H, C_4-H), 6.50 (d, J = 2.1 Hz, 1H, C_2-H), 7.41 (d, J = 6.9 Hz, 1H, C_8-H), 12.35 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3388, 2919, 2869, 1740, 1640, 1595, 1451, 1275, 1120, 1107, 1033,750; EI-MS *m/z*: 462[M]$^+$(5), 169(31), 69(100); Anal. ($C_{28}H_{30}O_6$) C, H. Calc: 72.71, 6.54; found: 72.46, 6.49.

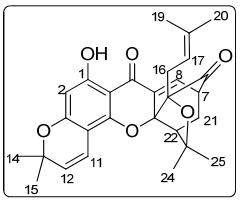
Preparation of caged compounds 29 and 30. A solution of compound **24** (100 mg, 0.22 mmol) in DMF (2 mL) was stirred under nitrogen at 120 °C for 2 h. The reaction mixture was then cooled to room temperature and concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford caged compounds **29** and **30** as isomers.

8-Hydroxy-1-(3-methylbut-2-en-yl)-3,3,11,11-tetramethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (29). Yield: 41%; m.p.: 121-123 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ1.15 (s, 3H, C_{19}-H), 1.27-1.28 (m, 4H, C_{20}-H, C_{21}-H_α), 1.40 (s, 3H, C_{24}-H), 1.44 (s, 6H, C_{14}-H, C_{15}-H), 1.67 (s, 3H, C_{25}-H), 2.32 (dd, J_1 = 13.5 Hz, J_2 = 4.4 Hz, 1H, C_{21}-H_β), 2.41 (d, J = 9.6 Hz, 1H, C_{22}-H), 2.59 (d, J = 8.1 Hz, 2H, C_{16}-H), 3.48 (t, J = 5.5 Hz, 1H, C_7-H), 4.44-4.49 (m, 1H, C_{17}-H), 5.52 (d, J = 10.0 Hz, 1H, C_{12}-H), 6.00 (s, 1H, C_4-H), 6.63 (d, J = 10.0 Hz, 1H, C_{11}-H), 7.40 (, J = 6.9 Hz, 1H, C_8-H), 12.71 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3398, 2941, 2863, 1740, 1649, 1630, 1599, 1460, 1360, 1285, 1195, 11245, 840, 750; EI-MS *m/z*: 462[M]$^+$(35), 169(100), 69(55); HRMS (ESI): calcd. for $C_{28}H_{30}O_6$ [M + H]$^+$ 463.2121, found 463.2125; HPLC (95% acetonitrile in water): t_R = 4.8 min, 98.8%.

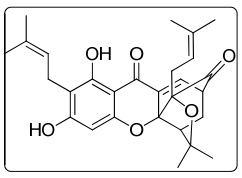
8-Hydroxy-1-(3-methylbut-2-en-yl)-3,3,11,11-tetramethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (30). Yield: 50%; m.p.: 113-114 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ1.08 (s, 3H, C_{19}-H), 1.29-1.30 (m, 4H, C_{20}-H, C_{21}-H_α), 1.38 (s, 3H, C_{24}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 1.67 (s, 3H, C_{25}-H), 2.34 (dd, J_1 = 13.5 Hz, J_2 = 4.5 Hz, 1H, C_{21}-H_β), 2.50 (d, J = 9.5 Hz, 1H, C_{22}-H), 2.56 (d, J = 7.5 Hz, 2H, C_{16}-H), 3.50 (t, J = 5.5 Hz, 1H, C_7-H), 4.45-4.50 (m, 1H, C_{17}-H), 5.55 (d, J = 10.1 Hz, 1H, C_{12}-H), 6.01 (s, 1H, C_4-H), 6.65 (d, J = 10.1 Hz, 1H, C_{11}-H), 7.46 (, J = 6.9 Hz, 1H, C_8-H), 12.66 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3300, 2970, 2913, 1741, 1640, 1634, 1590, 1455, 1375, 1260, 1131, 837, 747; EI-MS *m/z*: 462[M]$^+$(15), 169(90), 147(65), 69(100); HRMS (ESI): calcd. for $C_{28}H_{30}O_6$ [M + H]$^+$ 463.2121, found 463.2118; HPLC (95% acetonitrile in water): t_R = 4.1 min, 99.1%.

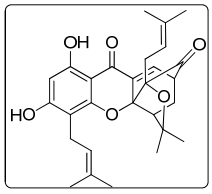
Compounds **30** and **31** was prepared according to our previously reported procedure [S2].

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (31). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H),1.30 (s,



3H),1.36 (dd, J_1 = 13.6 Hz, J_2 = 10.5 Hz, 1H), 1.40 (s, 3H), 1.67 (s, 3H), 1.77 (d, J = 1.1 Hz, 3H), 1.82 (s, 3H), 2.33 (dd, J_1 = 13.6 Hz, J_2 = 4.5 Hz, 1H), 2.39 (d, J = 9.6 Hz, 1H), 2.59-2.61 (m, 2H), 3.35-3.39 (m, 2H), 3.48 (dd, J_1 = 6.8, J_2 = 4.3 Hz, 1H), 4.44-4.48 (m, 1H), 5.23-5.26 (m, 1H), 6.08 (s, 1H), 6.60 (br s, 1H), 7.40 (d, J = 6.8 Hz, 1H), 12.80 (s, 1H); ESI-MS *m/z*: 463 [M-H]$^-$; HPLC (80% acetonitrile in water): t_R = 4.6 min, 98.9%.

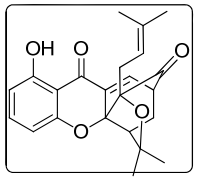
8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (32). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.04(s, 3H), 1.24-1.26 (m, 4H), 1.38 (s, 3H), 1.69 (s, 3H), 1.76 (s, 3H), 1.81 (s, 3H), 2.35 (dd, J_1 = 13.2 Hz, J_2 = 4.4 Hz, 1H), 2.46 (d, J = 9.2 Hz, 1H), 2.56-2.59 (m, 2H), 3.41-3.49 (m, 2H), 3.50-3.53 (m, 1H), 4.30-4.45 (m, 1H), 5.22-5.27 (m, 1H), 6.04 (s, 1H), 6.09 (br s, 1H), 7.46 (d, J = 6.8 Hz, 1H), 12.60 (s, 1H); ^{13}C NMR (75 MHz,



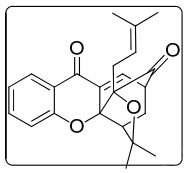
$CDCl_3$): δ 16.9, 18.2, 22.3, 25.6, 25.7, 25.9, 29.0, 29.2, 30.3, 47.0, 49.2, 83.2, 84.5, 90.5, 97.0, 101.0, 105.6, 117.7, 121.1, 133.3, 133.9, 134.9, 135.4, 157.9, 163.0, 163.9, 179.4, 203.1; ESI-MS *m/z*: 463 $[M-H]^-$; HRMS (ESI): calcd. for $C_{28}H_{32}O_6$ $[M + Na]^+$ 487.2097, found 487.2104; HPLC (80% acetonitrile in water): t_R = 3.8 min, 99.1%.

Compounds **33** and **34** was prepared according to our previously reported procedure [S3].

8-Hydroxy-1-(3-methylbut-2-en-yl)-3,3-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (33). yellow solid; mp: 130-132 °C. ^{1}H NMR (300 MHz, $CDCl_3$): 0.95 (s, 3H), 1.18-1.25 (m, 4H), 1.30 (s, 3H), 1.61 (s, 3H), 2.26 (dd, J_1 = 13.5 Hz, J_2 = 4.5 Hz, 1H), 2.37 (d, J = 9.6 Hz, 1H), 2.54 (d, J = 7.8 Hz, 2H), 3.44 (dd, J_1 = 6.9 Hz, J_2 = 4.5 Hz, 1H), 4.32-4.36 (m, 1H), 6.43 (dd, J_1 = 8.1 Hz, J_2 = 0.9 Hz, 1H), 6.45 (dd, J_1 = 8.1 Hz, J_2 = 0.9 Hz, 1H), 7.32 (t, J = 8.1 Hz, 1H), 7.41 (d, J = 9.6 Hz, 1H), 12.00 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 16.68, 24.95, 25.49, 29.05, 29.14, 30.27, 46.98, 48.82, 83.53, 84.46, 90.02, 106.12, 107.38, 109.37, 118.64, 133.88, 134.90, 135.30, 138.75, 159.59, 162.90, 181.33, 202.69; EI-MS *m/z*: 380$[M]^+$, 352; Anal. ($C_{23}H_{24}O_5$) C, H. Calc: 72.61, 6.36; found: 72.60, 6.67; HPLC (80% acetonitrile in water): t_R = 4.7 min, 99.5%.

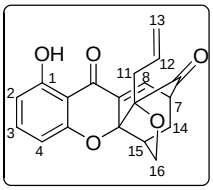


1-(3-Methylbut-2-en-yl)-3,3-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (34, Cluvenone). yellow solid; mp: 124-126 °C. ^{1}H NMR (300 MHz, $CDCl_3$): 0.84 (s, 3H), 1.15-1.30 (m, 7H), 1.66 (s, 3H), 2.27 (dd, J_1 = 13.5Hz, J_2 = 4.5Hz, 1H), 2.39 (d, J = 9.6Hz, 1H), 2.55 (d, J = 9.3Hz, 2H), 3.41-3.47 (m, 1H), 4.33-4.37 (m, 1H), 6.97-7.02 (m, 2H), 7.36 (d, J = 6.9 Hz, 1H), 7.45 (dd, J_1 = 8.4 Hz, J_2 = 7.2 Hz, 1H), 7.87 (d, J = 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 16.69, 25.14, 25.33, 29.12, 30.33, 30.93, 46.84, 48.83, 83.52, 84.62, 90.37, 118.10, 118.97, 119.14, 121.87, 126.99, 133.76, 134.78, 134.94, 136.21, 159.67, 176.52, 203.02. Anal. ($C_{23}H_{24}O_4$) C, H. Calc: 72.80, 6.64; found: 5.81, 6.60; HPLC (80% acetonitrile in water): t_R = 5.1 min, 99.4%.



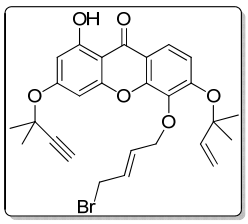
5,6-Bis(allyloxy)-1-hydroxy-9*H*-xanthen-9-one (35). To a stirring solution of compound **8** (1.22 g, 11 mmol) in acetone (20 mL) was added K_2CO_3 (1.66 g, 12 mmol) and allyl bromide (1.31 g, 11 mmol) and stirred at 35°C for 4 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford the titled compound as yellow solid (1.53 g, 95%). m.p.: 113-114°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.69-4.76 (m, 4H, 2 × OCH_2), 5.18-5.50 (m, 4H, 2 × CH=C$\underline{H_2}$), 5.98-6.25 (m, 2H, 2 × C$\underline{H}$=CH_2), 6.79 (d, J = 8.4 Hz, 1H, Ar-H), 6.98-7.01 (m, 2H, Ar-H), 7.60 (t, J = 8.4 Hz, 1H, Ar-H), 7.99 (d, J = 9.0 Hz, 1H, Ar-H), 12.72 (s, 1H, Ar-OH); IR (KBr, cm^{-1}): 3052, 1653, 1601, 1581, 1478, 1459, 1284, 1232, 1086, 792; EI-MS *m/z*: 324$[M]^+$(100), 283(80).

1-Allyl-8-hydroxy-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (36). A solution of compound **35** (100 mg, 0.30 mmol) in decalin (2 mL) was stirred under nitrogen at 180 °C for 3 h. The reaction mixture was then cooled to room temperature and purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford the titled compound **36** as a yellow solid (51 mg, 51%). m.p.: 127-128 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.83-1.93 (m, 2H, C_{14}-H), 2.46-2.54 (m, 1H, C_{15}-H), 2.62 (dd, J_1 = 3.3 Hz, J_2 = 7.8 Hz, 1H, C_{11}-H$_\alpha$), 2.78-2.85 (m, 1H, C_{11}-H$_\beta$), 3.53-3.56 (m, 1H, C_7-H), 3.89 (d, J = 8.1 Hz, 1H, C_{16}-H$_\alpha$), 4.50-4.59 (m, 2H, C_{13}-H), 4.74 (d, J = 10.2 Hz, 1H, C_{16}-H$_\beta$), 5.20-5.26 (m, 1H, C_{12}-H), 6.50-6.56 (m, 2H, C_2-H, C_4-H) 7.38 (d, J = 6.9 Hz, 1H, C_8-H), 7.44 (d, J = 8.4 Hz, 1H, C_3-H), 12.05 (s, 1H, C_1-OH); IR (KBr, cm^{-1}): 3413, 3076, 2982, 1734, 1643, 1604, 1463, 1377, 1229, 1034, 800, 780; EI-MS *m/z*: 324$[M]^+$(40), 296(100); HRMS (ESI) calcd. for $C_{19}H_{16}O_4$ $[M + Na]^+$ 347.0895, found 347.0872. HPLC (80% acetonitrile in water): t_R = 4.2 min, 98..5%

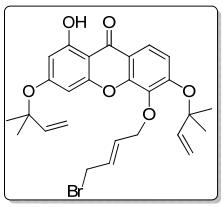


General procedure for preparation of 37, 39, 42 and 44. To a stirring solution of required hydroxyxathone (**10**, **12**, **14** and **16**, 1 mmol) in acetone (20 mL) was added K_2CO_3 (276 mg, 2 mmol) and (*E*)-1,4-dibromobut-2-ene (428 mg, 2 mmol) and stirred at 35°C for 4 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford product **37**, **39**, **42** and **44**, respectively.

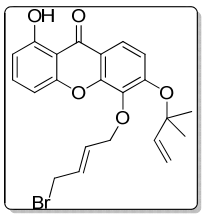
(*E*)-5-(4-Bromobut-2-enyloxy)-1-hydroxy-3-(2-methylbut-3-yn-2-yloxy)-6-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (37). Yield: 76%; yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.58 (s, 6H, 2 × -CH_3), 1.70 (s, 6H, 2 × -CH_3), 2.73 (s, 1H, -C≡CH), 3.97 (d, J = 5.2 Hz, 2H, -$BrCH_2$-), 4.66-4.69 (m, 2H, -OCH_2-), 5.20-5.29 (m, 2H, -CH=C$\underline{H}_2$), 6.09-6.23 (m, 3H, -C$\underline{H}$=CH_2, -C$\underline{H}$=C$\underline{H}$-), 6.70 (d, J = 2.0 Hz, 1H, Ar-$\underline{H}$), 6.83 (d, J = 2.0 Hz, 1H, Ar-H), 7.12 (d, J = 9.0 Hz, 1H, Ar-H), 7.84 (d, J = 9.0 Hz, 1H, Ar-H), 12.83 (s, 1H, Ar-OH); IR (cm^{-1}, KBr film): 3357, 3074, 2984, 2934, 1744, 1651, 1600, 1564, 1435, 1276, 1261, 1126, 1100, 1057, 995, 927, 818, 764, 749; EI-MS *m/z*: 528[M]$^+$(3), 526(3), 336(33), 206(48), 175(100), 161(57).



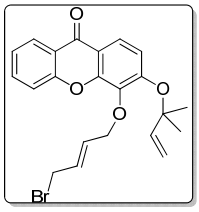
(*E*)-5-(4-Bromobut-2-enyloxy)-1-hydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (39). Yield: 76%; yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.50 (s, 12H, 4 × -CH_3), 3.89-3.91 (m, 2H, -$BrCH_2$-), 4.60 (d, J = 3.9 Hz, 2H, -OCH_2-), 5.13-5.25 (m, 4H, 2×-CH=C$\underline{H}_2$), 6.01-6.10 (m, 4H, 2×-C$\underline{H}$=CH_2, -C$\underline{H}$=C$\underline{H}$-), 6.37 (d, J = 2.1 Hz, 1H, Ar-H), 6.50 (d, J = 2.1 Hz, 1H, Ar-H), 7.04 (d, J = 9.0 Hz, 1H, Ar-H), 7.76 (d, J = 9.0 Hz, 1H, Ar-H), 12.73 (s, 1H, Ar-OH); IR (KBr, cm^{-1}): 3333, 2977, 2926, 1744, 1644, 1606, 1504, 1435, 1277, 1127, 1093, 972, 818, 751; EI-MS *m/z*: 530[M]$^+$(3), 528(3),313(56), 260(100), 83(90).



(*E*)-5-(4-Bromobut-2-enyloxy)-1-hydroxy-6-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (42). Yield: 74%; yellow solid; m.p.: 56-58 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.59 (s, 6H, 2 × -C$\underline{H}_3$), 3.86 (d, J = 3.3 Hz, 2H, -$BrCH_2$-), 4.70 (d, J = 3.9 Hz, 2H, -OCH_2-), 5.21-5.30 (m, 2H, -CH=C$\underline{H}_2$), 6.04-6.23 (m, 3H, -C$\underline{H}$=CH_2, -C$\underline{H}$=C$\underline{H}$-), 6.79 (d, J = 8.1 Hz, 1H, Ar-H), 6.99 (d, J = 8.4 Hz, 1H, Ar-H), 7.15 (d, J = 9.3 Hz, 1H, Ar-H), 7.57 (t, J = 8.4 Hz, 1H, Ar-H), 7.88 (d, J = 9.3 Hz, 1H, Ar-H), 12.71 (s, 1H, Ar-OH); IR (cm^{-1}, KBr film): 3286, 2979, 2932, 1740, 1645, 1601, 1560, 1462, 1439, 1381, 1269, 1232, 1079, 995, 930, 806, 735; EI-MS *m/z*: 446[M]$^+$(2), 444(2),297(32), 244(63), 69(100).

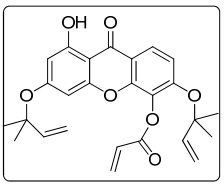


(*E*)-4-(4-Bromobut-2-enyloxy)-3-(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (44). Yield: 77%; yellow solid; m.p.: 65-67 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.58 (s, 6H, 2 × -C$\underline{H}_3$), 3.97-3.98 (m, 2H, -$BrCH_2$-), 4.72 (d, J = 3.8 Hz, 2H, -OCH_2-), 5.19-5.28 (m, 2H, -CH=C$\underline{H}_2$), 6.11-6.24 (m, 3H, -C$\underline{H}$=CH_2, -C$\underline{H}$=C$\underline{H}$-), 7.14 (d, J = 9.0 Hz, 1H, Ar-H), 7.38 (t, J = 7.2 Hz, 1H, Ar-H), 7.56 (d, J = 8.3 Hz, 1H, Ar-H), 7.69 (t, J = 7.1 Hz, 1H, Ar-H), 7.95 (d, J = 9.0 Hz, 1H, Ar-H), 8.31 (d, J = 8.0 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.3, 31.6, 33.0, 82.4, 114.3, 117.3, 117.6, 118.1, 121.2, 121.7, 124.0, 126.7, 129.8, 130.8, 134.5, 138.5, 143.4, 151.1, 155.1, 156.2, 176.6; IR (cm^{-1}, KBr film): 2985, 2932, 1740, 1645, 1620, 1601, 1567, 1462, 1439, 1378, 1275, 1233, 1128, 1079, 992, 810, 765, 750; EI-MS *m/z*: 430[M]$^+$(10), 428(10), 295(42), 69(100).

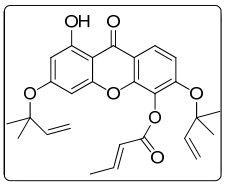


General procedure for preparation of 41a-d. To a stirring solution of corresponding substituted acrylic acid (0.77 mmol) in DCM (10 mL) was added DMAP (225 mg, 1.84 mmol) and EDC·HCl (166 mg, 0.92 mmol) and stirred at room temperature for 30 min, and then a solution of compound **12** (200 mg, 0.51 mmol) in DCM (5 mL) was added and the reaction mixture was stirred at room temperature for another 2 h. The reaction mixture was filtered and the filtration was concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford product **41a-d**, respectively.

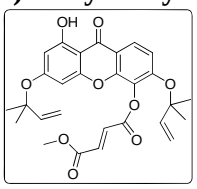
5-Acryloyloxy-1-hydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (41a). Yield: 80%; yollow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.50-1.61 (m, 12H, 4 × -CH_3), 5.22-5.27 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.09-6.14 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.43 (d, J = 2.1 Hz, 1H, Ar-H), 6.46 (d, J = 2.1 Hz, 1H, Ar-H), 6.62-6.64 (m, 3H, -OCOC$\underline{H}$=C$\underline{H}_2$), 7.17 (d, J = 9.0 Hz, 1H, Ar-H), 7.97 (d, J = 9.0 Hz, 1H, Ar-H), 12.76 (s, 1H, Ar-OH); EI-MS *m/z*: 450[M]$^+$(7), 433(10), 382(22), 314(18), 260(41), 69(100).



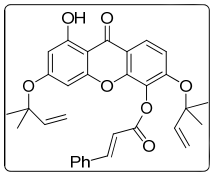
(*E*)-5-(But-2-enoyloxy)-1-hydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (41b). Yield: 71%; yollow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, 4 × -CH_3), 2.05-2.13 (m, 3H, -CH_3), 5.21-5.30 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.09-6.15 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.45 (d, J = 2.4 Hz, 2H, Ar-H), 6.51-6.75 (m, 1H, -OCOCH=C$\underline{H}$-), 7.10 (d, J = 9.0 Hz, 1H, Ar-H), 7.18-7.26 (m, 1H, -OCOC$\underline{H}$=CH-), 7.87 (d, J = 9.0 Hz, 1H, Ar-H), 12.69 (s, 1H, -OH); EI-MS *m/z*: 464[M]$^+$(8), 447(15), 396(21), 341(19), 69(100).



(*E*)-1-Hydroxy-5-(4-methoxy-4-oxobut-2-enoyloxy)-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (41c). Yield: 74%; yollow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, 4 × -CH_3), 3.81 (s, 3H, -OCH_3), 5.22-5.29 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.10-6.15 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.36 (d, J = 2.1 Hz, 2H, Ar-H), 7.08-7.19 (m, 2H, -CH=CH-), 7.41 (d, J = 9.0 Hz, 1H, Ar-H), 7.91 (d, J = 9.0 Hz, 1H, Ar-H), 12.63 (s, 1H, Ar-OH); EI-MS *m/z*: 508[M]$^+$ (9), 69(100).

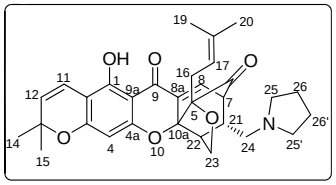


5-(Cinnamoyloxy)-1-hydroxy-3,6-bis(2-methylbut-3-en-2-yloxy)-9*H*-xanthen-9-one (41d). Yield: 75%; yollow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, 4 × -C$\underline{H}_3$), 5.21-5.30 (m, 4H, 2 × -CH=C$\underline{H}_2$), 6.09-6.15 (m, 2H, 2 × -C$\underline{H}$=CH_2), 6.43 (d, J = 2.4 Hz, 2H, Ar-H), 6.51-6.75 (m, 2H, -CH=CH-), 7.18 (d, J = 9.0 Hz, 1H, Ar-H), 7.38-7.57 (m, 5H, Ar-H), 7.91 (d, J = 9.0 Hz, 1H, Ar-H), 12.69 (s, 1H, Ar-OH); EI-MS *m/z*: 526[M]$^+$ (10), 498(17), 436(15), 380(26), 69(100).



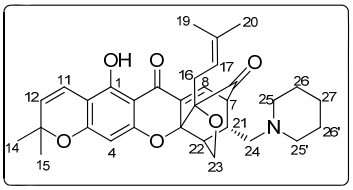
General procedure for preparation of caged compounds 46-52 and 53-59. To a stirring solution of compound **37** (0.20 mmol) in DMF (5 mL) was added potassium carbonate (0.40 mmol) and the corresponding amine (0.30 mmol) and the solution was stirred at 35 °C for 1 h. The reaction mixture was partitioned between ethyl acetate (15 mL) and water (20 mL). The water layer was extracted with ethyl acetate (15 mL × 2). The organic layer was combined, washed with saturated NaCl solution (10 mL × 2), dried over sodium sulfate, fiterted and concentrated to afford **38** as yellow oil. The yellow oil was then dissolved in DMF (3 mL) and the reaction solution was stirred at 120°C under nitrogen for 2 h. The reaction mixture was cooled to room temperature and concentrated. The residue was purified by column chromatography (16:1 petroleum ether/ethyl acetate) to afford **46-52** and **53-59** as isomers.

8-Hydroxy1-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-(pyrrolidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (46). Yield: 24%; yellow solid; m.p.: 184-185 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.26 (s, 3H, C_{19}-H), 1.30 (s, 3H, C_{20}-H), 1.43 (s, 3H, C_{15}-H), 1.45 (s, 3H, C_{14}-H), 1.79-1.81 (m, 4H, C_{26}-H, $C_{26'}$-H), 2.25-2.41 (m, 4H, C_{21}-H, C_{24}-H, C_{22}-H), 2.55-2.68 (m, 6H, C_{16}-H, C_{25}-H, $C_{25'}$-H), 3.64 (d, J = 4.5 Hz, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.43 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.9 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.53 (d, J = 10.2 Hz, 1H, C_{12}-H), 5.95 (s, 1H, C_4-H), 6.63 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.15 (d, J = 6.6 Hz, 1H, C_8-H), 12.73 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.2 (C-19), 22.5 (C-26), 24.5 (C-20), 26.7 (C-16), 28.7 (C-14), 28.7 (C-15), 39.8 (C-21), 46.2 (C-22), 49.0 (C-7), 53.1 (C-25), 57.4 (C-24), 73.5 (C-23), 77.5 (C-13), 81.2 (C-5), 87.2 (C-10a), 95.2 (C-4), 100.1 (C-9a), 102.1 (C-2), 114.2 (C-11), 116.6 (C-17), 125.4 (C-12), 128.6 (C-8), 131.9 (C-8a), 134.8 (C-18), 158.1 (C-4a), 159.4 (C-3), 161.7 (C-1), 177.9 (C-9), 199.0 (C-6); IR (cm^{-1}, KBr film): 3440, 2973, 2920, 1741, 1645, 1633, 1598, 1455, 1387, 1316, 1151, 1098, 883, 804; EI-MS

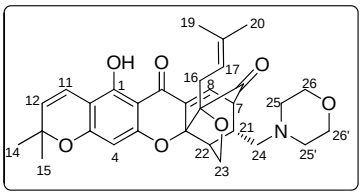


m/z: 517[M]$^+$(6), 420(60), 84(100), 58(72); HRMS (ESI): calcd. for $C_{31}H_{35}NO_6$ [M + H]$^+$ 518.2543, found 518.2559; HPLC (90% methanol in water): t_R = 7.8 min, 98.8%.

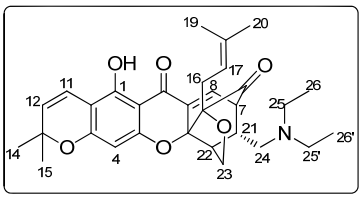
8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-(piperidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (47). Yield: 29%; yellow solid; m.p.: 192-194 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.16 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 1.47-1.69 (m, 6H, C_{26}-H, $C_{26'}$-H, C_{27}-H), 2.11-2.17 (m, 3H, C_{21}-H, C_{24}-H), 2.21-2.40 (m, 5H, C_{22}-H, C_{25}-H, $C_{25'}$-H), 2.54-2.69 (m, 2H, C_{16}-H), 3.63 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.84 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.53 (d, J = 9.9 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 10.2 Hz, 1H, C_{11}-H), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.75 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3451, 2935, 1740, 1651, 1634, 1599, 1456, 1320, 1276, 1261, 1168, 1057, 764, 750; EI-MS *m/z*: 531[M]$^+$(5), 434(59), 98(100), 58(50), 55(12); HRMS (ESI): calcd. for $C_{32}H_{37}NO_6$ [M + H]$^+$ 532.2699, found 532.2709; HPLC (95% methanol in water): t_R = 9.2 min, 99.3%.



8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-morpholinomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (48). Yield: 29%; yellow solid; m.p.: 177-178 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.17 (s, 3H, C_{19}-H), 1.39 (s, 3H, C_{20}-H), 1.68 (s, 6H, C_{14}-H, C_{15}-H), 2.11-2.16 (m, 2H, C_{24}-H), 2.20-2.31 (m, 2H, C_{21}-H, C_{22}-H), 2.38-2.39 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.59-2.66 (m, 2H, C_{16}-H), 3.62-3.69 (m, 5H, C_7-H, C_{26}-H, $C_{26'}$-H), 3.86 (d, J = 8.4 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.51 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.53 (dd, J = 9.9 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.14 (d, J = 6.9 Hz, 1H, C_8-H); 12.72 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3445, 2961, 2920, 1738, 1651, 1633, 1598, 1455, 1316, 1260, 1118, 1025, 801, 745; EI-MS *m/z* 533[M]$^+$(9), 436(62), 100(100), 70(8), 56(13); HRMS (ESI): calcd. for $C_{31}H_{35}NO_6$ [M + H]$^+$ 534.2492, found 534.2503; HPLC (90% methanol in water): t_R = 5.7 min, 99.3%.

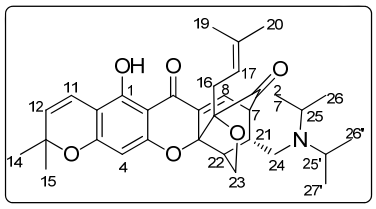


4-Diethylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (49). Yield: 30%; yellow solid; m.p.: 149-151°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.96-0.98 (m, 6H, C_{26}-H, $C_{26'}$-H), 1.16 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 2.13-2.29 (m, 6H, C_{24}-H, C_{25}-H, $C_{25'}$-H), 2.45-2.68 (m, 4H, C_{16}-H, C_{21}-H, C_{22}-H), 3.66 (d, J = 6.6 Hz, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.51 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.53 (d, J = 9.9 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3433, 2964, 2928, 1740, 1645, 1633, 1599, 1455, 1387, 1317, 1260, 1096, 1018, 801, 704; EI-MS *m/z*: 519[M]$^+$(4), 422(22), 86(100), 58(9); HRMS (ESI): calcd. for $C_{31}H_{37}NO_6$ [M + H]$^+$ 520.2699, found 520.2712; HPLC (90% methanol in water): t_R = 10.4 min, 99.3%.



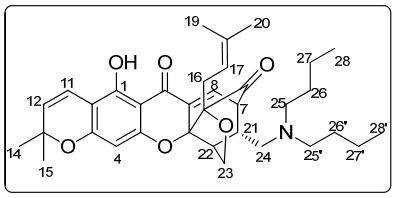
8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-dipropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (50). Yield: 30%; yellow solid; m.p.: 172-174 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.86 (t, J = 7.2 Hz, 6H, C_{27}-H, $C_{27'}$-H), 1.15 (s, 3H, C_{19}-H), 1.33-1.38 (m, 7H, C_{20}-H, C_{26}-H, $C_{26'}$-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 2.20-2.33 (m, 8H, C_{21}-H, C_{22}-H, C_{24}-H, C_{25}-H, $C_{25'}$-H), 2.55-2.63 (m, 2H, C_{16}-H), 3.67 (d, J = 6.9 Hz, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.53 (d, J = 9.9 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.78 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3427, 2961, 2932, 1740, 1645, 1634, 1599, 1452, 1390, 1313, 1263, 1172, 1095, 1065, 818, 803, 765; EI-MS *m/z*: 547[M]$^+$(8), 450(53), 114(100), 86(27), 72(20), 69(10), 58(9); HRMS (ESI): calcd. for $C_{33}H_{41}NO_6$ [M + H]$^+$ 548.3012, found 548.3021; HPLC (95% methanol in water): t_R = 10.0 min, 98.9%.

8-Hydroxy4-diisopropylaminomethyl-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (51). Yield: 25%; yellow



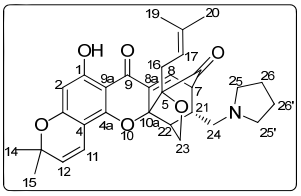
solid; m.p.: 185-186 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90-0.99 (m, 12H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.16 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.46 (s, 6H, C_{14}-H, C_{15}-H), 2.18-2.30 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.59-2.64 (m, 2H, C_{16}-H), 2.91-3.00 (m, 2H, C_{25}-H, $C_{25'}$-H), 3.64-3.67 (m, 1H, C_7-H), 3.81 (d, J= 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.53 (d, J = 10.2 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.64 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.17 (d, J = 6.9 Hz, 1H, C_8-H), 12.80 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3445, 2968, 2920, 1738, 1651, 1645, 1593, 1455, 1391, 1313, 1261, 1096, 1021, 801; EI-MS *m/z*: 547[M]$^+$(2), 114(100), 72(13), 69(5); HRMS (ESI): calcd. for $C_{33}H_{41}NO_6$ [M + H]$^+$ 548.3012, found 548.3023; HPLC (95% methanol in water): t_R = 14.5 min, 98.9%.

4-Dibutylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[3,2-*b*]xanthene-7,15-dione (52). Yield: 32%; yellow



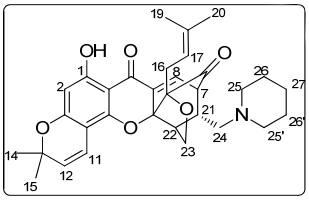
solid; m.p.: 200-202 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.89 (t, J= 7.2 Hz, 6H, C_{28}-H, $C_{28'}$-H), 1.17 (s, 3H, C_{19}-H), 1.26-1.34 (m, 8H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.39 (s, 3H, C_{20}-H), 1.44 (s, 6H, C_{14}-H, C_{15}-H), 2.12-2.23 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.26-2.41 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.59-2.63 (m, 2H, C_{16}-H), 3.65 (d, J = 6.6 Hz, 1H, C_7-H), 3.83 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.43-4.49 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.52 (d, J = 10.2 Hz, 1H, C_{12}-H), 5.96 (s, 1H, C_4-H), 6.63 (d, J = 10.2 Hz, 1H, C_{11}-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H); 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3421, 2962, 2932, 1741, 1691, 1644, 1599, 1457, 1095, 1022, 799, 731; EI-MS *m/z* 575[M]$^+$(5), 478(43), 142(100), 100(38), 58(14); HRMS (ESI): calcd. for $C_{35}H_{45}NO_6$ [M + H]$^+$ 576.3325, found 576.3336; HPLC (95% acetonitrile in water): t_R = 13.2 min, 99.6%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-(pyrrolidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (53). Yield: 35%; yellow



solid; m.p.: 179-180 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.44 (s, 3H, C_{15}-H), 1.45 (s, 3H, C_{14}-H), 1.70-1.80 (m, 4H, C_{26}-H, $C_{26'}$-H), 2.22-2.30 (m, 3H, C_{21}-H, C_{24}-H), 2.40-2.46 (m, 5H, C_{22}-H, C_{25}-H, $C_{25'}$-H), 2.58-2.65 (m, 2H, C_{16}-H), 3.63 (dd, J_1 = 2.1 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.89 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.42-4.44 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 8.4 Hz, 1H, C_{23}-H$_\alpha$), 5.54 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.01 (s, 1H, C_2-H), 6.54 (d, J = 6.9 Hz, 1H, C_{11}-H), 7.17 (d, J = 9.9 Hz, 1H, C_8-H), 12.58 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 17.1 (C-19), 23.6 (C-26), 25.5 (C-20), 27.8 (C-16), 28.4 (C-14), 28.5 (C-15), 41.1 (C-21), 47.3 (C-22), 50.2 (C-7), 54.1 (C-25), 58.5 (C-24), 74.7 (C-23), 78.4 (C-13), 82.2 (C-5), 88.6 (C-10a), 97.8 (C-2), 101.1 (C-9a), 101.7 (C-4), 115.1 (C-11), 117.5 (C-17), 127.0 (C-12), 129.9 (C-8), 132.7 (C-8a), 135.8 (C-18), 154.6 (C-4a), 162.8 (C-3), 164.8 (C-1), 178.9 (C-9), 199.9 (C-6); IR (cm^{-1}, KBr film): 3280, 2963, 2896, 1737, 1646, 1628, 1587, 1413, 1310, 1260, 1091, 1024, 865, 799, 705; EI-MS m/z: 517[M]$^+$(6), 420(59), 84(100); HRMS (ESI): calcd. for $C_{33}H_{41}NO_6$ [M + H]$^+$ 518.2543, found 518.2548; HPLC (90% methanol in water): t_R = 6.9 min, 98.6%.

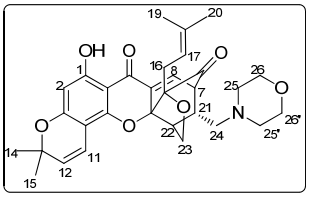
8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-(piperidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (54). Yield: 41%; yellow



solid; m.p.: 189-191°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.39 (s, 3H, C_{20}-H), 1.44 (s, 3H, C_{15}-H), 1.45 (s, 3H, C_{14}-H), 1.49-1.67 (m, 6H, C_{26}-H, $C_{26'}$-H, C_{27}-H), 2.00-2.16 (m, 2H, C_{24}-H), 2.20-2.40 (m, 5H, C_{21}-H, C_{25}-H, $C_{25'}$-H), 2.53-2.69 (m, H, C_{16}-H, C_{22}-H), 3.64 (dd, J_1 = 2.7 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.86-3.88 (m, 1H, C_{23}-H$_\beta$), 4.41-4.42 (m, 1H, C_{17}-H), 4.47-4.51 (m, 1H, C_{23}-H$_\alpha$), 5.54 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.55 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.16 (d, J = 6.6 Hz, 1H, C_8-H), 12.58 (s, 1H, C_1-OH); IR (cm^{-1}, KBr

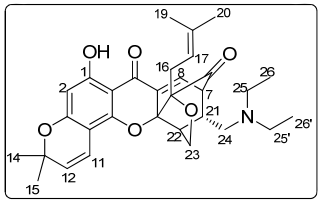
film): 3274, 2963, 2905, 1741, 1644, 1594, 1413, 1260, 1088, 1024, 864, 801, 701; EI-MS *m/z*: 531[M]$^+$(5), 434(49), 98(100), 58(39), 55(12); HRMS (ESI): calcd. for $C_{32}H_{37}NO_6$ [M + H]$^+$ 532.2699, found 532.2713; HPLC (95% methanol in water): t_R = 8.0 min, 99.2%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-morpholinomethyl-3,3a,4,5-tetrahydro -1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (55). Yield: 40%; yellow solid;



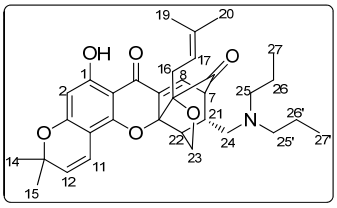
m.p.: 164-166 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 2.12-2.22 (m, 2H, C_{24}-H), 2.31-2.39 (m, 6H, C_{21}-H, C_{22}-H, C_{25}-H, $C_{25'}$-H), 2.58-2.65 (m, 2H, C_{16}-H), 3.60-3.76 (m, 5H, C_7-H, C_{26}-H, $C_{26'}$-H), 3.89 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.40-4.42 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.56 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.55 (d, J = 10.2 Hz, 1H, C_{11}-H), 7.17 (d, J = 6.6 Hz, 1H, C_8-H), 12.57 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3286, 2956, 2903, 1735, 1644, 1590, 1275, 1261, 1116, 1036, 801, 750; EI-MS *m/z*: 533[M]$^+$(4), 436(30), 100(100), 70(9), 56(13); HRMS (ESI): calcd. for $C_{31}H_{35}NO_7$ [M + H]$^+$ 534.2492, found 534.2498; HPLC (90% methanol in water): t_R = 5.0 min, 97.6%.

4-Diethylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (56). Yield: 40%; yellow solid;



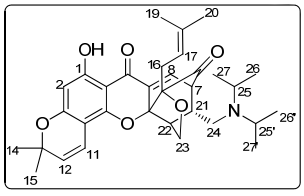
m.p.: 140-142 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.95 (m, 6H, C_{26}-H, $C_{26'}$-H), 1.16 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 2.16-2.20 (m, 6H, C_{24}-H, C_{25}-H, $C_{25'}$-H), 2.48-2.65 (m, 4H, C_{16}-H, C_{21}-H, C_{22}-H), 3.67 (d, J = 6.9 Hz, 1H, C_7-H), 3.89 (d, J = 7.5 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.43 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H$_\alpha$), 5.55 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.55 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.17 (d, J = 6.9 Hz, 1H, C_8-H), 12.60 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3422, 2962, 2920, 1738, 1644, 1634, 1587, 1434, 1319, 1257, 1125, 1101, 1063, 798, 751; EI-MS *m/z*: 519[M]$^+$(3), 422(21), 86(100), 58(10); HRMS (ESI): calcd. for $C_{31}H_{37}NO_6$ [M + H]$^+$ 520.2699, found 520.2708; HPLC (90% methanol in water): t_R = 8.9 min, 98.7%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-4-dipropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (57). Yield: 41%; yellow



solid; m.p.: 169-170 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.86 (t, J = 7.2 Hz, 6H, C_{27}-H, $C_{27'}$-H), 1.12 (s, 3H, C_{19}-H), 1.33-1.37 (m, 7H, C_{20}-H, C_{26}-H, $C_{26'}$-H), 1.44-1.46 (m, 6H, C_{14}-H, C_{15}-H), 2.15-2.37 (m, 8H, C_{21}-H, C_{22}-H, C_{24}-H, C_{25}-H, $C_{25'}$-H), 2.53-2.70 (m, 2H, C_{16}-H), 3.68 (d, J = 6.9 Hz, 1H, C_7-H), 3.89 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.40-4.42 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.3 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.50 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.55 (d, J = 10.2 Hz, 1H, C_{11}-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.61 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3416, 2962, 2938, 1738, 1644, 1635, 1587, 1431, 1260, 1128, 1103, 801; EI-MS *m/z*: 547[M]$^+$(8), 450(45), 114(100), 86(25), 72(19), 58(30); HRMS (ESI): calcd. for $C_{33}H_{41}NO_6$ [M + H]$^+$ 548.3012, found 548.3021; HPLC (95% acetonitrile in water): t_R = 12.0 min, 98.5%.

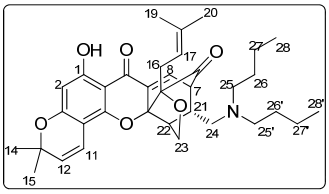
8-Hydroxy-4-diisopropylaminomethyl-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (58). Yield: 36%;



yellow solid; m.p.: 166-168 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.91-0.99 (m, 12H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.12 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.45 (s, 6H, C_{14}-H, C_{15}-H), 2.18-2.31 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.57-2.64 (m, 2H, C_{16}-H), 2.92-3.01 (m, 2H, C_{25}-H, $C_{25'}$-H), 3.67-3.69 (m, 1H, C_7-H), 3.86 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.43 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.55 (d, J = 10.2 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.56 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.19 (d, J = 6.9 Hz, 1H, C_8-H), 12.62 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3428, 2963, 2906, 1741, 1644, 1584, 1415, 1260, 1095, 1024, 799, 765; EI-MS *m/z*:

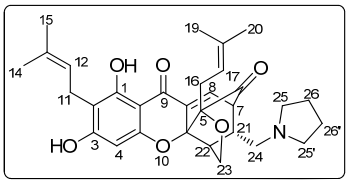
547[M]$^+$(2), 114(100), 72(14), 69(4); HRMS (ESI): calcd. for $C_{33}H_{41}NO_6$ [M + H]$^+$ 548.3012, found 548.3019; HPLC (95% methanol in water): t_R = 12.2 min, 99.0%.

4-Dibutylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-11,11-dimethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*,11*H*-furo[3,4-*g*]pyrano[2,3-*c*]xanthene-7,15-dione (59). Yield: 43%; yellow solid; m.p.: 190-191°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.87-0.92 (m, 6H, C_{28}-H, $C_{28'}$-H), 1.12 (s, 3H, C_{19}-H), 1.26-1.30 (m, 8H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.38 (s, 3H, C_{20}-H), 1.44-1.45 (m, 6H, C_{14}-H, C_{15}-H), 2.13-2.37 (m, 8H, C_{21}-H, C_{22}-H, C_{24}-H, C_{25}-H, $C_{25'}$-H), 2.53-2.69 (m, 2H, C_{16}-H), 3.67 (d, J = 6.6 Hz, 1H, C_7-H), 3.88 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.43 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.55 (d, J = 9.9 Hz, 1H, C_{12}-H), 6.02 (s, 1H, C_2-H), 6.55 (d, J = 9.9 Hz, 1H, C_{11}-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.61 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3421, 2963, 2927, 1738, 1649, 1644, 1584, 1416, 1261, 1090, 1013, 801, 765; EI-MS *m/z*: 575[M]$^+$(5), 478(36), 142(100), 100(34), 58(14); HRMS (ESI): calcd. for $C_{35}H_{45}NO_6$ [M + H]$^+$ 576.3325, found 576.3336; HPLC (95% acetonitrile in water): t_R = 13.6 min, 98.1%.

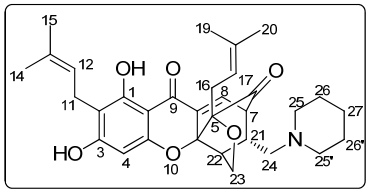


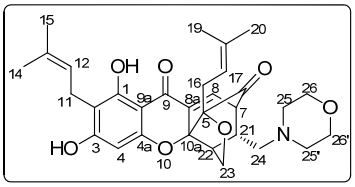
General procedure for preparation of caged compounds 61-81 and 82-103. To a stirring solution of compound **37** (0.20 mmol) in DMF (5 mL) was added potassium carbonate (0.40 mmol) and the corresponding amine or alcohol or acid (0.30 mmol) and the solution was stirred at 35 °C for 1 h. The reaction mixture was partitioned between ethyl acetate (15 mL) and water (20 mL). The water layer was extracted with ethyl acetate (15 mL × 2). The organic layer was combined, washed with saturated NaCl solution (10 mL × 2), dried over sodium sulfate, fiterted and concentrated to afford **40** as yellow oil. The yellow oil was then dissolved in DMF (3 mL) and the reaction solution was stirred at 120 °C under nitrogen for 2 h. The reaction mixture was cooled to room temperature and concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford **61-81** and **82-103** as isomers.

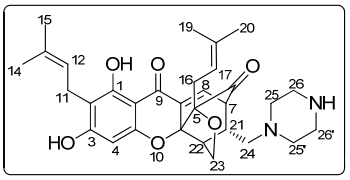
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-(pyrrolidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (60). Yield: 27%; yellow solid; m.p.: 162-163 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.72 (s, 3H, C_{15}-H), 1.78-1.80 (m, 7H, C_{14}-H, C_{26}-H, $C_{26'}$-H), 2.18-2.36 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.51-2.65 (m, 6H, C_{25}-H, $C_{25'}$-H, C_{16}-H), 3.07 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.58 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1 H, C_7-H), 3.81 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.34-4.43 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.20-5.24 (m, 1H, C_{12}-H), 5.86 (s, 1H, C_4-H), 7.12 (d, J = 6.9 Hz, 1H, C_8-H), 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3347, 2971, 2920, 1736, 1645, 1633, 1594, 1459, 1327, 1061, 819, 746; EI-MS *m/z*: 519[M]$^+$(6), 422(50), 84(100), 58(98), 55(18); HRMS (ESI): calcd. for $C_{31}H_{37}NO_6$ [M + H]$^+$ 520.2699, found 520.2709; HPLC (65% acetonitrile in water): t_R = 2.3 min, 99.0%.

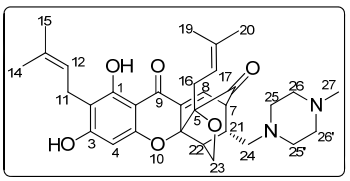


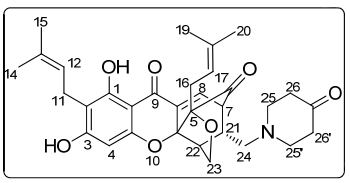
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-(piperidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (61). Yield: 25%; yellow solid; m.p. 200-202 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.10 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.40-1.42 (m, 2H, C_{27}-H), 1.52-1.54 (m, 4H, C_{26}-H, $C_{26'}$-H), 1.75 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.06-2.10 (m, 2H, C_{24}-H), 2.26-2.27 (m, 2H, C_{21}-H, C_{22}-H), 2.30-2.34 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.53-2.69 (m, 2H, C_{16}-H), 3.35 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.60 (dd, J_1 = 3.3 Hz, J_2 = 6.9 Hz, 1 H, C_7-H), 3.81 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.44 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23-5.25 (m, 1H, C_{12}-H), 5.95 (s, 1H, C_2-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.82 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3368, 2935, 1736, 1645, 1633, 1596, 1454, 1320, 1061, 817; EI-MS *m/z*: 533[M]$^+$(3), 436(30), 98(72), 58(100), 55(17); HRMS (ESI): calcd. for $C_{32}H_{39}NO_6$ [M + H]$^+$ 534.2856, found 536.2864; HPLC (65% acetonitrile in water): t_R = 7.2 min, 99.5%.



8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-morpholinomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-d]xanthene-7,13-dione (62). Yield: 29%; yellow solid; m.p.: 102-103 °C; 
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.05 (s, 3H, C_{19}-H), 1.39 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.07-2.14 (m, 2H, C_{24}-H), 2.21-2.30 (m, 2H, C_{21}-H, C_{22}-H), 2.34-2.44 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.54-2.70 (m, 2H, C_{16}-H), 3.33-3.36 (m, 2H, C_{11}-H), 3.62-3.63 (m, 1H, C_7-H), 3.64-3.69 (m, 4H, C_{26}-H, $C_{26'}$-H), 3.83 (d, J = 8.4 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.43 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.3 Hz, J_2 = 8.4 Hz, 1H, C_{23}-H$_\alpha$), 5.21-5.25 (m, 1H, C_{12}-H), 6.00 (s, 1H, C_4-H), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.78 (s, 1 H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.8 (C-19), 17.4 (C-14), 20.6 (C-11), 25.0 (C-20), 25.3 (C-15), 27.2 (C-16), 38.2 (C-21), 46.5 (C-22), 49.1 (C-7), 53.1 (C-25), 60.1(C-24), 66.3(C-26), 73.9 (C-23), 81.9 (C-5), 87.4 (C-10a), 107.3 (C-2), 100.6 (C-9a), 94.7 (C-4), 117.0 (C-17), 120.9 (C-12), 128.9 (C-8), 132.7 (C-8a), 133.4 (C-13), 135.5 (C-18), 158.5 (C-4a), 161.7 (C-1), 163.9 (C-3), 178.3 (C-9), 199.7 (C-6); IR (cm^{-1}, KBr film): 3280, 1969, 2924, 1736, 1644, 1633, 1598, 1457, 1381, 1117, 1087, 1068, 863, 816, 735; EI-MS *m/z*: 535[M]$^+$(3), 438(16), 382(6), 100(63), 58(100); HRMS (ESI): calcd. for $C_{31}H_{37}NO_7$ [M + H]$^+$ 536.2648, found 536.2657; HPLC (65% acetonitrile in water): t_R = 2.0 min, 99.1%.

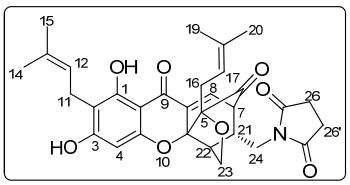
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-(piperazin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (63). Yield: 20%; yellow solid; m.p.: 159-160 °C; 
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.10 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.04-2.29 (m, 5H, C_{21}-H, C_{22}-H, C_{24}-H, N_{27}-H), 2.31-2.41 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.60-2.62 (m, 2H, C_{16}-H), 2.85-2.90 (m, 4H, C_{26}-H, $C_{26'}$-H), 3.33-3.35 (m, 2H, C_{11}-H), 3.60 (dd, J_1 = 3.3 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.80 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.41-4-43 (m, 1H, C_{17}-H), 4.49 (dd, J_1 = 3.9 Hz, J_2 = 8,1 Hz, 1H, C_{23}-H$_\alpha$), 5.23 (t, 1H, C_{12}-H), 5.94 (s, 1H, C_4-H), 7.12 (d, J = 6.6 Hz, 1H, C_8-H), 12.80 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3387, 2959, 2918, 1743, 1644, 1633, 1597, 1320, 1274, 1169, 1149, 1090, 818, 764, 749; EI-MS *m/z*: 534[M]$^+$(6), 437(31), 207(21), 99(100), 70(31), 56(46), 55(35); HRMS (ESI): calcd. for $C_{31}H_{38}N_2O_6$ [M + H]$^+$ 535.2808, found 535.2811; HPLC (50% acetonitrile in water): t_R = 1.7 min, 98.8%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-((4-methylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (64). Yield: 23%; yellow solid; m.p.: 227-229 °C; 
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.75 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.11-2.21 (m, 4H, C_{24}-H, C_{21}-H, C_{22}-H), 2.35 (s, 3H, C_{27}-H), 2.51-2.69 (m, 10H, C_{25}-H, $C_{25'}$-H, C_{26}-H, $C_{26'}$-H, C_{16}-H), 3.34-3.36 (m, 2H, C_{11}-H), 3.60-3.63 (m, 1H, C_7-H), 3.81 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.42 (m, 1H, C_{17}-H), 4.48 (dd, J_1 = 3.9 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H$_\alpha$), 5.24-5.26 (m, 1H, C_{12}-H), 5.99 (s, 1H, C_4-H), 7.12 (d, J = 6.9 Hz, 1H, C_8-H), 12.81 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3369, 2968, 2920, 1735, 1641, 1633, 1590, 1446, 1316, 1166, 1143, 1095, 1063, 815; EI-MS *m/z*: 548[M]$^+$(15), 451(65), 395(29), 113(100), 98(32), 70(82), 58(94); HRMS (ESI): calcd. for $C_{32}H_{40}N_2O_6$ [M + H]$^+$ 549.2965, found 549.2973; HPLC (60% acetonitrile in water): t_R = 1.7 min, 98.7%.

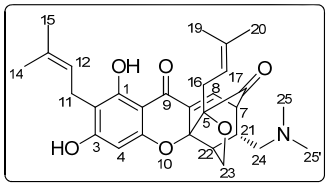
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-((4-oxopiperidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (65). Yield: 26%; yellow solid; m.p.: 168-170 °C; 
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.14 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.26-2.35 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.40-2.46 (m, 4H, C_{26}-H, $C_{26'}$-H), 2.52-2.80 (m, 6H, C_{16}-H, C_{25}-H, $C_{25'}$-H), 3.35-3.37 (m, 2H, C_{11}-H), 3.67 (d, J = 6.9 Hz, 1H, C_7-H), 3.84 (d, J = 8.4 Hz, 1H, C_{23}-H$_\beta$), 4.39-4.46 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.3 Hz, J_2 = 8.4 Hz, 1H, C_{23}-H$_\alpha$), 5.24-5.26 (m, 1H, C_{12}-H), 6.06 (s, 1H, C_4-H), 7.16 (d, J = 6.9 Hz, 1H, C_8-H), 12.80 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3265, 2964, 2914, 1739, 1712, 1644, 1633, 1599, 1453, 1315, 1190, 1152, 1057, 816, 736; EI-MS *m/z*: 547[M]$^+$(5),

450(17), 394(13), 112(100), 83(14), 58(26); HRMS (ESI): calcd. for $C_{32}H_{37}NO_7$ $[M + H]^+$ 548.2648, found 548.2655; HPLC (60% acetonitrile in water): t_R = 2.3 min, 99.0%.

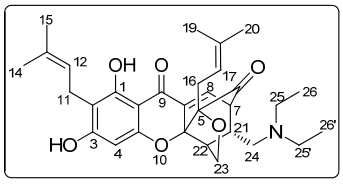
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-((2,4-dioxopyrrolidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (66). Yield: 24%; yellow solid; m.p.: 220-222 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.06 (s, 3H, C_{19}-H), 1.30 (s, 3H, C_{20}-H), 1.64 (s, 3H, C_{15}-H), 1.73 (s, 3H, C_{14}-H), 2.16-2.18 (m, 1H, C_{22}-H), 2.24-2.27 (m, 1H, C_{21}-H), 2.45-2.53 (m, 2H, C_{16}-H), 2.67 (s, 4H, C_{26}-H, $C_{26'}$-H), 3.23-3.31 (m, 5H, C_{11}-H, C_7-H, C_{24}-H), 3.80 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.31-4.33 (m, 1H, C_{17}-H), 4.39 (dd, J_1 = 3.6 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H$_\alpha$), 5.16 (t, J = 6.6 Hz, 1H, C_{12}-H), 5.90 (s, 1H, C_4-H), 6.43 (brs, 1H, C_3-OH), 7.12 (d, J = 6.6 Hz, 1H, C_8-H), 12.71 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3242, 2958, 2921, 1737, 1703, 1633, 1598, 1451, 1404, 1320, 1180, 1057, 818; EI-MS *m/z*: 547$[M]^+$(14), 407(93), 255(72), 69(100), 55(71); HRMS (ESI): calcd. for $C_{31}H_{33}NO_8$ $[M + H]^+$ 548.2284, found 548.2302; HPLC (50% acetonitrile in water): t_R = 20.4 min, 98.7%.



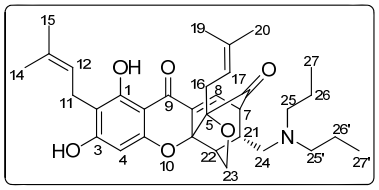
8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-dimethylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (67). Yield: 27%; yellow solid; m.p.: 165-166 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.72 (s, 3H, C_{15}-H), 1.80 (s, 3H, C_{14}-H), 2.08-2.18 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.22 (s, 6H, C_{25}-H, $C_{25'}$-H), 2.52-2.64 (m, 2H, C_{16}-H), 3.31 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.55 (dd, J_1 = 2.7 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.81 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.38-4.42 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23 (t, J = 6.6 Hz, 1H, C_{12}-H), 5.87 (s, 1H, C_4-H), 7.11 (d, J = 6.9 Hz, 1H, C_8-H), 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3246, 2964, 2912, 1740, 1634, 1594, 1452, 1319, 1272, 1188, 1053, 815, 768, 753; EI-MS *m/z*: 493$[M]^+$(8), 396(68), 84(14), 58(100); HRMS (ESI): calcd. for $C_{29}H_{35}NO_6$ $[M + H]^+$ 494.2543, found 494.2546; HPLC (60% acetonitrile in water): t_R = 2.3 min, 99.2%.



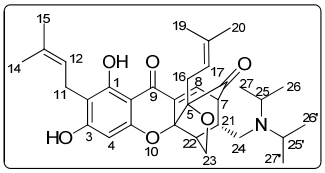
4-Diethylaminomethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (68). Yield: 32%; yellow solid; m.p.: 122-123 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.96 (t, J = 6.9 Hz, 6H, C_{26}-H, $C_{26'}$-H), 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.14-2.28 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.40-2.58 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.62-2.70 (m, 2H, C_{16}-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.64 (d, J = 6.6 Hz, 1H, C_7-H), 3.83 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.49 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.24 (t, J = 6.9 Hz, 1H, C_{12}-H), 5.98 (s, 1H, C_2-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.84 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3327, 2962, 2920, 1741, 1643, 1633, 1599, 1455, 1319, 1063, 815, 748; EI-MS *m/z*: 521$[M]^+$(6), 424(41), 86(100), 58(74); HRMS (ESI): calcd. for $C_{31}H_{39}NO_6$ $[M + H]^+$ 522.2856, found 522.2865; HPLC (65% acetonitrile in water): t_R = 5.7 min, 98.8%.



8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-dipropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (69). Yield: 30%; yellow solid; m.p.: 166-167 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.86 (t, J = 6.9 Hz, 6H, C_{27}-H, $C_{27'}$-H), 1.13 (s, 3H, C_{19}-H), 1.30-1.40 (m, 7H, C_{20}-H, C_{26}-H, $C_{26'}$-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.12-2.22 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.21-2.37 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.58-2.67 (m, 2H, C_{16}-H), 3.36 (d, J = 7.2 Hz, 2H, C_{11}-H), 3.66 (d, J = 6.9 Hz, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.19-5.27 (m, 1H, C_{12}-H), 6.00 (s, 1H, C_4-H), 7.14 (d, J = 6.6 Hz, 1H, C_8-H), 12.83 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3263, 2956, 2926, 1735, 1644, 1634, 1590, 1449, 1328, 1269, 1184, 1063, 818, 750; EI-MS *m/z*: 549$[M]^+$(11), 452(85), 114(100), 86(25), 72(24), 58(78); HRMS (ESI): calcd. for $C_{33}H_{43}NO_6$ $[M + H]^+$ 550.3169, found 550.3178; HPLC (65% acetonitrile in water): t_R = 9.3 min, 99.6%.

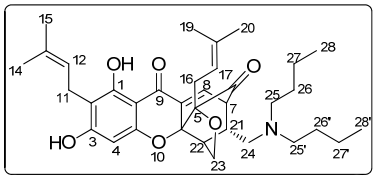


8,10-Dihydroxy-4-diisopropylaminomethyl-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (70). Yield: 27%; yellow solid; m.p.: 196-197 °C;



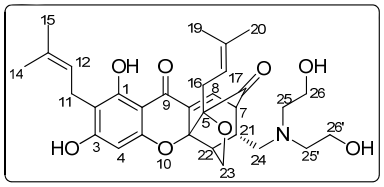
^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90-0.99 (m, 12H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.13 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.82 (s, 3H, C_{15}-H), 1.88 (s, 3H, C_{14}-H), 2.18-2.32 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.54-2.69 (m, 2H, C_{16}-H), 2.91-3.00 (m, 2H, C_{25}-H, $C_{25'}$-H), 3.36-3.38 (m, 2H, C_{11}-H), 3.64-3.67 (m, 1H, C_7-H), 3.82 (d, J = 7.5 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.51 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23-5.27 (m, 1H, C_{12}-H), 6.02 (s, 1H, C_4-H), 6.29 (brs, 1H, C_3-OH), 7.13 (d, J = 6.6 Hz, 1H, C_8-H), 12.87 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3323, 2963, 2919, 1735, 1644, 1634, 1596, 1455, 1322, 1182, 1060, 817, 737; EI-MS *m/z*: 549[M]$^+$(4), 114(100), 72(39), 58(57); HRMS (ESI): calcd. for $C_{33}H_{43}NO_6$ [M + H]$^+$ 550.3169, found 550.3173; HPLC (60% acetonitrile in water): t_R = 26.2 min, 99.5%.

4-Dibutylaminomethyl)-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (71). Yield: 31%; yellow solid; m.p.: 190-192 °C;



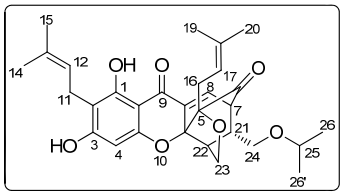
^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, J = 6.9 Hz, 6H, C_{28}-H, $C_{28'}$-H), 1.14 (s, 3H, C_{19}-H), 1.25-1.32 (m, 8H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.38 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.10-2.25 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.27-2.42 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.57-2.76 (m, 2H, C_{16}-H), 3.35 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.65 (d, J = 6.6 Hz, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.47-4.50 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23-5.27 (m, 1H, C_{12}-H), 5.98 (s, 1H, C_4-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.82 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3256, 2964, 2927, 1740, 1644, 1634, 1594, 1454, 1324, 1186, 1056, 820; EI-MS *m/z*: 577[M]$^+$(6), 480(44), 142(100), 100(25); HRMS (ESI): calcd. for $C_{35}H_{47}NO_6$ [M + H]$^+$ 578.3482, found 578.3474; HPLC (65% acetonitrile in water): t_R = 15.1 min, 99.3%.

4-Diethanolaminomethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (72). Yield: 28%; yellow solid; m.p.: 168-170 °C;



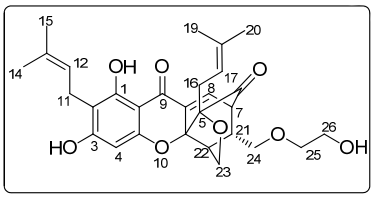
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.26-2.30 (m, 2H, C_{24}-H), 2.34-2.42 (m, 2H, C_{21}-H, C_{22}-H), 2.53-2.68 (m, 6H, C_{16}-H, C_{25}-H, $C_{25'}$-H), 3.06 (dd, J_1 = 7.5 Hz, J_2 = 14.7 Hz, 2H, C_{26}-OH, $C_{26'}$-OH), 3.32-3.35 (m, 2H, C_{11}-H), 3.59 (t, J = 4.8 Hz, 4H, C_{26}-H, $C_{26'}$-H), 3.66-3.69 (m, 1H, C_7-H), 3.88 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.39-4.41 (m, 1H, C_{17}-H), 4.48 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.22-5.26 (m, 1H, C_{12}-H), 6.03 (s, 1H, C_4-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3381, 2962, 2920, 1732, 1644, 1633, 1593, 1452, 1060, 765, 748; EI-MS *m/z*: 553[M]$^+$(6), 456(14), 174(17), 118(100), 58(17); HRMS (ESI): calcd. for $C_{31}H_{39}NO_8$ [M + H]$^+$ 554.2754, found 554.2764; HPLC (65% acetonitrile in water): t_R = 1.3 min, 98.8%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-isopropoxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (73). Yield: 27%; yellow solid; m.p.: 100-101 °C;



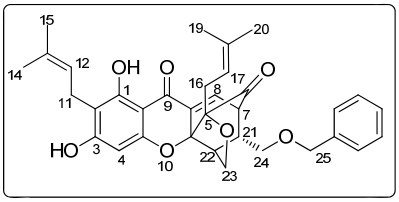
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09-1.13 (m, 9H, C_{19}-H, C_{26}-H, $C_{26'}$-H), 1.37 (s, 3H, C_{20}-H), 1.75 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.18-2.30 (m, 2H, C_{21}-H, C_{22}-H), 2.59-2.67 (m, 2H, C_{16}-H), 3.15-3.17 (m, 2H, C_{24}-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.46-3.50 (m, 1H, C_{25}-H), 3.69 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.89 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.48-4.52 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.22-5.25 (m, 1H, C_{12}-H), 6.01 (s, 1H, C_4-H), 6.80 (brs, 1H, C_3-OH), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.81 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3316, 2973, 2914, 1738, 1644, 1634, 1593, 1458, 1331, 1278, 1151, 1090, 815, 762, 745; EI-MS *m/z*: 508[M]$^+$(28), 480(30), 437(72), 407(100), 255(75), 69(82); HRMS (ESI): calcd. for $C_{30}H_{36}O_7$ [M-H]$^+$ 507.2383, found 507.2386; HPLC (60% acetonitrile in water): t_R = 2.7 min, 99.2%.

8,10-Dihydroxy-4-hydroxyethoxymethyl-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (74). Yield: 22%; yellow solid; m.p.: 68-69 °C; ^{1}H



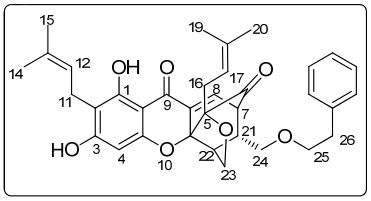
NMR (300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.75 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.22-2.24 (m, 1H, C_{22}-H), 2.32-2.41 (m, 1H, C_{21}-H), 2.59-2.65 (m, 2H, C_{16}-H), 3.25-3.36 (m, 4H, C_{11}-H, C_{24}-H), 3.49-3.52 (m, 2H, C_{26}-H), 3.66 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.71-3.73 (m, 2H, C_{25}-H), 3.89 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.40-4.42 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.22-5.26 (m, 1H, C_{12}-H), 6.01 (s, 1H, C_4-H), 6.78 (brs, 1H, C_3-OH), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.77 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3375, 3233, 2962, 2914, 1739, 1651, 1633, 1599, 1452, 1322, 1272, 1051, 813, 751; EI-MS *m/z*: 510[M]$^+$(13), 407(83), 255(48), 207(63), 78(67), 69(100), 55(58); HRMS (ESI): calcd. for $C_{29}H_{34}O_8$ [M-H]$^+$ 509.2175, found 509.2179; HPLC (65% acetonitrile in water): t_R = 1.5 min, 99.4%.

4-((Benzyloxy)methyl)-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (75). Yield: 20%; yellow solid; m.p.: 70-72 °C; ^{1}H



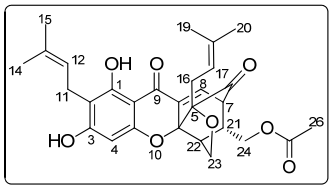
NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.75 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.17-2.19 (m, 1H, C_{22}-H), 2.34-2.39 (m, 1H, C_{21}-H), 2.54-2.71 (m, 2H, C_{16}-H), 3.22 (d, J = 7.8 Hz, 2H, C_{24}-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.69 (dd, J_1 = 3.0 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.39-4.50 (m, 4H, C_{17}-H, C_{23}-H$_\alpha$, C_{25}-H), 5.24 (t, J = 6.9 Hz, 1H, C_{12}-H), 5.99 (s, 1H, C_4-H), 7.06 (d, J = 6.9 Hz, 1H, C_8-H), 7.28-7.35 (m, 5H, Ph), 12.79 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3304, 2968, 2909, 1738, 1643, 1626, 1599, 1455, 1316, 1272, 1095, 1054, 813, 750, 695; EI-MS *m/z*: 556[M]$^+$(9), 528(12), 407(27), 255(19), 91(100), 69(29); HRMS (ESI): calcd. for $C_{34}H_{36}O_7$ [M-H]$^+$ 555.2383, found 555.2394; HPLC (60% acetonitrile in water): t_R = 5.2 min, 99.4%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-phenethoxylmethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (76). Yield: 21%; yellow solid; m.p.: 123-124 °C;



^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.05-2.09 (m, 1H, C_{22}-H), 2.29-2.31 (m, 1H, C_{21}-H), 2.53-2.65 (m, H, C_{16}-H), 2.80-2.85 (m, 2H, C_{26}-H), 3.16 (d, J = 7.8 Hz, 2H, C_{24}-H), 3.36 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.58-3.59 (m, 3H, C_7-H, C_{25}-H), 3.83 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.39-4.45 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23-5.25 (m, 1H, C_{12}-H), 6.00 (s, 1H, C_4-H), 6.78 (brs, 1H, C_3-OH), 7.03 (d, J = 6.6 Hz, 1H, C_8-H), 7.16-7.26 (m, 5H, Ph), 12.81 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3333, 2962, 2909, 1738, 1644, 1634, 1599, 1452, 1322, 1263, 1095, 806, 748, 695; EI-MS *m/z*: 570[M]$^+$(9), 542(17), 407(48), 255(22), 105(100), 69(28); HRMS (ESI): calcd. for $C_{35}H_{38}O_7$ [M-H]$^+$ 569.2539, found 569.2548; HPLC (60% acetonitrile in water): t_R = 6.3 min, 99.0%.

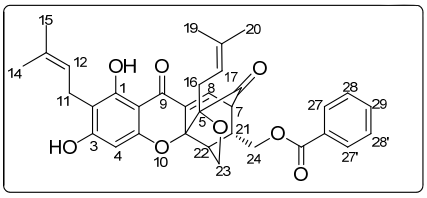
4-Acetoxymethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (77). Yield: 26%; yellow solid; m.p.: 182-184 °C; ^{1}H NMR



(300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.77 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.06 (s, 3H, C_{26}-H), 2.17-2.19 (m, 1H, C_{22}-H), 2.38-2.42 (m, 1H, C_{21}-H), 2.55-2.70 (m, 2H, C_{16}-H), 3.37 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.63 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.74-3.81 (m, 1H, C_{24}-H$_\alpha$), 3.89-3.97 (m, 2H, C_{23}-H$_\beta$, C_{24}-H$_\beta$), 4.40-4.41 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.3 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.22-5.25 (m, H, C_{12}-H), 6.00 (s, 1H, C_4-H), 6.39 (brs, 1H, C_3-OH), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.78 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3351, 2961, 2908, 1735, 1646, 1633, 1596, 1451, 1322, 1057, 813, 760; EI-MS *m/z*: 508[M]$^+$(36),

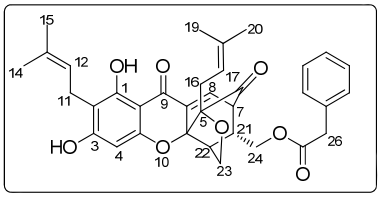
407(39), 255(32), 203(36), 69(100), 55(40); HRMS (ESI): calcd. for $C_{29}H_{32}O_8$ $[M-H]^+$ 507.2019, found 507.2016; HPLC (50% acetonitrile in water): t_R = 2.2 min, 99.5%.

4-Benzoyloxymethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (78). Yield: 25%; yellow solid; m.p.: 64-65 °C; ^{1}H



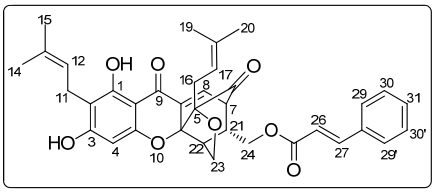
NMR (300 MHz, $CDCl_3$): δ 1.14 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.29-2.31 (m, 1H, C_{22}-H), 2.57-2.65 (m, 3H, C_{21}-H, C_{16}-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.71-3.73 (m, 1H, C_7-H), 3.95 (d, J = 7.8 Hz, 1H, C_{24}-H$_\alpha$), 4.06 (m, 1H, C_{24}-H$_\beta$), 4.17-4.19 (m, 1H, C_{23}-H$_\beta$), 4.35-4.40 (m, 1H, C_{17}-H), 4.53-4.54 (m, 1H, C_{23}-H$_\alpha$), 5.22-5.26 (m, 1H, C_{12}-H), 6.02 (s, 1H, C_4-H), 6.78 (brs, 1H, C_3-OH), 7.21 (d, J = 6.9 Hz, 1H, C_8-H), 7.43-7.48 (m, 2H, C_{28}-H, $C_{28'}$-H), 7.57-7.59 (m, 1H, C_{29}-H), 8.00 (d, J = 7.8 Hz, 2H, C_{27}-H, $C_{27'}$-H), 12.76 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3363, 2968, 2914, 1744, 1720, 1644, 1634, 1596, 1452, 1319, 1266, 1098, 1066, 1025, 815, 712; EI-MS *m/z*: 570$[M]^+$(11), 407(15), 122(37), 105(100), 77(70), 69(31), 51(30); HRMS (ESI): calcd. for $C_{34}H_{34}O_8$ $[M-H]^+$ 569.2175, found 569.2178; HPLC (50% acetonitrile in water): t_R = 4.0 min, 99.2%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-phenylacetyloxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (79). Yield: 23%; yellow solid; m.p.: 150-152 °C;



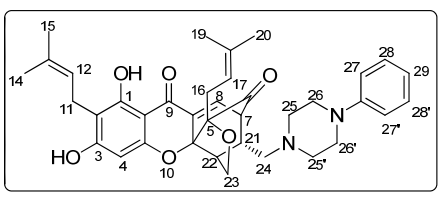
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.11-2.13 (m, 1H, C_{22}-H), 2.33-2.37 (m, 1H, C_{21}-H), 2.58-2.65 (m, 2H, C_{16}-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.50 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.61 (s, 2H, C_{26}-H), 3.75-3.92 (m, 3H, C_{24}-H, C_{23}-H$_\beta$), 4.37-4.44 (m, 2H, C_{17}-H, C_{23}-H$_\alpha$), 5.23-5.25 (m, 1H, C_{12}-H), 5.99 (s, 1H, C_4-H), 6.61 (brs, 1H, C_3-OH), 7.07 (d, J = 6.9 Hz, 1H, C_8-H), 7.24-7.36 (m, 5H, Ph), 12.75 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3357, 2968, 2914, 1744, 1644, 1634, 1452, 1322, 1275, 1260, 1151, 1048, 813, 751; EI-MS *m/z*: 584$[M]^+$(18), 556(17), 407(24), 255(19), 91(100), 69(42); HRMS (ESI): calcd. for $C_{35}H_{36}O_8$ $[M-H]^+$ 583.2332, found 583.2329; HPLC (60% acetonitrile in water): t_R = 3.8 min, 99.0%.

4-Cinnamyloxymethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (80). Yield: 22%; yellow solid; m.p.: 69-70 °C; ^{1}H



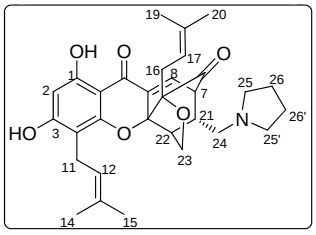
NMR (300 MHz, $CDCl_3$): δ 1.13 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.77 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.25-2.27 (m, 1H, C_{22}-H), 2.50-2.68 (m, 3H, C_{16}-H, C_{21}-H), 3.37 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.68-3.70 (m, 1H, C_7-H), 3.92-3.97 (m, 2H, C_{23}-H$_\beta$, C_{24}-H$_\alpha$), 4.04-4.10 (m, 1H, C_{24}-H$_\beta$), 4.41-4.42 (m, 1H, C_{17}-H), 4.53-4.54 (m, 1H, C_{23}-H$_\alpha$), 5.24-5.26 (m, 1H, C_{12}-H), 6.01 (s, 1H, C_4-H), 6.40 (d, J = 16.0 Hz, 1H, C_{26}-H), 6.55 (s, 1H, C_3-OH), 7.19 (d, J = 6.6 Hz, 1H, C_8-H), 7.38-7.45 (m, 3H, C_{230}-H, $C_{30'}$-H, C_{31}-H), 7.45-7.60 (m, 2H, C_{29}-H, $C_{29'}$-H), 7.68 (d, J = 16.0 Hz, 1H, C_{27}-H), 12.78 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3345, 2968, 2909, 1738, 1711, 1644, 1629, 1452, 1328, 1269, 1169, 1063, 818, 765, 750; EI-MS *m/z*: 596$[M]^+$(8), 568(19), 420(29), 407(34), 255(18), 131(100), 103(55), 69(34); HRMS (ESI): calcd. for $C_{36}H_{36}O_8$ $[M-H]^+$ 595.2332, found 595.2340; HPLC (60% acetonitrile in water): t_R = 7.0 min, 98.8%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-((4-phenylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (81). Yield: 30%; yellow solid; m.p.:

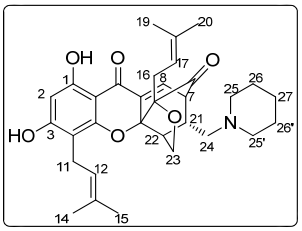


160-161 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.75 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.18-2.23 (m, 2H, C_{24}-H), 2.29-2.30 (m, 2H, C_{21}-H, C_{22}-H), 2.54-2.71 (m, 6H, C_{16}-H, C_{26}-H, $C_{26'}$-H), 3.14-3.17 (m, 4H, C_{25}-H, $C_{25'}$-H), 3.36 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.66 (dd, J_1 = 2.1 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.86 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.41-4.43 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 4.71 (s, 1H, C_3-OH), 5.24 (t, J = 6.6 Hz, 1H,

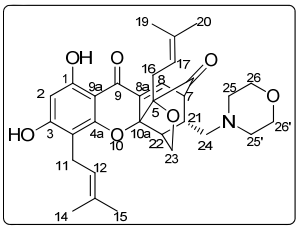
C_{12}-H), 6.00 (s, 1H, C_4-H), 6.84-6.93 (m, 3H, C_{28}-H, $C_{28'}$-H, C_{29}-H), 7.16 (d, J = 6.6 Hz, 1H, C_8-H), 7.24-7.38 (m, 2H, C_{27}-H, $C_{27'}$-H), 12.82 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3310, 2959, 2924, 1737, 1644, 1637, 1600, 1456, 1318, 1235, 1145, 1062, 816, 761, 741, 691; EI-MS *m/z*: 610$[M]^+$(28), 513(85), 457(35), 175(88), 160(41), 132(59), 104(35), 70(100); HRMS (ESI): calcd. for $C_{37}H_{42}N_2O_6$ $[M + H]^+$ 611.3121, found 611.3124; HPLC (60% acetonitrile in water): t_R = 8.2 min, 98.4%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-(pyrrolidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (82). Yield: 42%; yellow solid; m.p.: 137-138 °C; 

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.05 (s, 3H, C_{19}-H), 1.34 (s, 3H, C_{20}-H), 1.71 (s, 3H, C_{15}-H), 1.77 (s, 3H, C_{14}-H), 1.79-1.90 (m, 4H, C_{26}-H, $C_{26'}$-H), 2.15-2.42 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.48-2.68 (m, 6H, C_{25}-H, $C_{25'}$-H, C_{16}-H), 3.07 (d, J = 7.5 Hz, 2H, C_{11}-H), 3.54 (dd, J_1 = 2.1 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.86 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.34-4.36 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.13 (t, J = 6.6 Hz, 1H, C_{12}-H), 5.69 (s, 1H, C_2-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.46 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3440, 2973, 2785, 1741, 1644, 1633, 1598, 1455, 1316, 1169, 1151, 1098, 1063, 803; EI-MS *m/z*: 519$[M]^+$(6), 422(56), 84(100), 58(26), 55(9); HRMS (ESI): calcd. for $C_{31}H_{37}NO_6$ $[M + H]^+$ 520.2699, found 520.2706; HPLC (65% acetonitrile in water): t_R = 2.1 min, 99.3%.

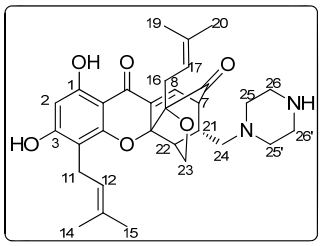
8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-(piperidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (83). Yield: 44%; yellow solid; m.p.: 176-178 °C; 

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.11 (s, 3H, C_{19}-H), 1.35 (s, 3H, C_{20}-H), 1.44-1.45 (m, 2H, C_{27}-H), 1.56-1.57 (m, 4H, C_{26}-H, $C_{26'}$-H), 1.72 (s, 3H, C_{15}-H), 1.79 (s, 3H, C_{14}-H), 2.12-2.30 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.37-2.40 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.47-2.67 (m, 2H, C_{16}-H), 3.25 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.57 (dd, J_1 = 2.7 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.85 (d, J = 8.1 Hz, 1H, C_{23}-H$_\beta$), 4.35-4.37 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.18 (t, J = 6.9 Hz, 1H, C_{12}-H), 5.81 (s, 1H, C_2-H), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.48 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3451, 2935, 1651, 1599, 1456, 1276, 1261, 1057, 764, 750; EI-MS *m/z*: 533$[M]^+$(7), 436(78), 380(8), 98(100), 58(57), 55(20); HRMS (ESI): calcd. for $C_{32}H_{39}NO_6$ $[M + H]^+$ 534.2856, found 534.2875; HPLC (65% acetonitrile in water): t_R = 3.0 min, 99.1%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-morpholinomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (84). Yield: 42%; yellow solid; m.p.: 127-128 °C; 

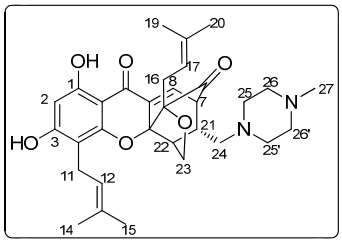
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.08 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.72 (s, 3H, C_{15}-H), 1.80 (s, 3H, C_{14}-H), 2.15-2.19 (m, 2H, C_{24}-H), 2.24-2.31 (m, 2H, C_{21}-H, C_{22}-H), 2.40-2.43 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.51-2.65 (m, 2H, C_{16}-H), 3.25-3.35 (m, 2H, C_{11}-H), 3.65 (dd, J_1 = 2.7 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.63-3.72 (m, 4H, C_{26}-H, $C_{26'}$-H), 3.86 (d, J = 8.4 Hz, 1H, C_{23}-H$_\beta$), 4.36-4.39 (m, 1H, C_{17}-H), 4.53 (dd, J_1 = 3.3 Hz, J_2 = 8.4 Hz, 1H, C_{23}-H$_\alpha$), 5.17-5.22 (m, 1H, C_{12}-H), 6.01 (s, 1H, C_2-H), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.49 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.5 (C-19), 17.3 (C-14), 21.2 (C-11), 25.0 (C-20), 25.2 (C-15), 27.1 (C-16), 38.1 (C-21), 46.7 (C-22), 49.2 (C-7), 53.1 (C-25), 60.1(C-24), 66.3 (C-26), 74.0 (C-23), 82.0 (C-5), 87.6 (C-10a), 96.2 (C-2), 100.7 (C-9a), 106.2 (C-4), 117.1 (C-17), 121.2 (C-12), 128.8 (C-8), 132.4 (C-8a), 133.1 (C-13), 135.4 (C-18), 157.1 (C-4a), 162.6 (C-1), 163.8 (C-3), 178.6 (C-9), 199.8 (C-6); IR (cm^{-1}, KBr film): 3266, 2959, 2920, 1739, 1645, 1634, 1598, 1433, 1381, 1270, 1174, 1112, 1065, 1036, 826, 737; EI-MS *m/z*: 535$[M]^+$(5), 438(26), 100(77), 58(100); HRMS (ESI): calcd. for $C_{31}H_{37}NO_7$ $[M + H]^+$ 536.2648, found 536.2658; HPLC (65% acetonitrile in water): t_R = 1.5 min, 99.2%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-(piperazin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (85). Yield: 30%; yellow solid; m.p.: 147-148 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.08 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.11-2.33 (m, 5H, C_{21}-H, C_{22}-H, C_{24}-H, N_{27}-H), 2.35-2.45 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.60-2.69 (m, 2H, C_{16}-H), 2.89-2.90 (m, 4H, C_{26}-H, $C_{26'}$-H), 3.28-3.40 (m, 2H, C_{11}-H), 3.63-3.65 (m, 1H, C_7-H), 3.85



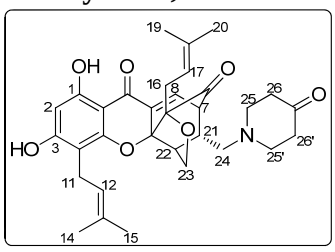
(d, J = 7.8 Hz, 1H, C_{23}-H_β), 4.39-4.40 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.20-5.22 (m, 1H, C_{12}-H), 5.97 (s, 1H, C_2-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.52 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3387, 2954, 2923, 1737, 1644, 1634, 1596, 1230, 1383, 1258, 1135, 1099, 1034, 799, 750; EI-MS *m/z*: 534$[M]^+$(6), 437(33), 99(100), 70(31), 56(51); HRMS (ESI): calcd. for $C_{31}H_{38}N_2O_6$ $[M + H]^+$ 535.2808, found 535.2816; HPLC (50% acetonitrile in water): t_R = 1.2 min, 98.3%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-((4-methylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (86). Yield: 38%; yellow solid; m.p.:



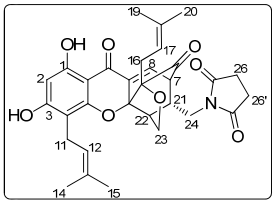
245-246 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.63 (s, 3H, C_{15}-H), 1.71 (s, 3H, C_{14}-H), 2.11-2.13 (m, 4H, C_{24}-H, C_{21}-H, C_{22}-H), 2.39 (s, 3H, C_{27}-H), 2.48-2.61 (m, 10H, C_{25}-H, $C_{25'}$-H, C_{26}-H, $C_{26'}$-H, C_{16}-H), 3.20-3.23 (m, 2H, C_{11}-H), 3.53 (d, J = 6.0 Hz, 1H, C_7-H), 3.78 (d, J = 7.8 Hz, 1H, C_{23}-H_β), 4.29-4.31 (m, 1H, C_{17}-H), 4.55 (dd, J_1 = 3.3 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.10-5.12 (m, 1H, C_{12}-H), 6.19 (s, 1H, C_2-H), 7.02 (d, J = 6.9 Hz, 1H, C_8-H), 12.40 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3374, 2962, 2920, 1735, 1644, 1633, 1593, 1425, 1381, 1311, 1263, 1128, 1084, 1057, 801, 733; EI-MS *m/z*: 548$[M]^+$(14), 451(54), 113(76), 98(31), 70(57), 58(100); HRMS (ESI): calcd. for $C_{32}H_{40}N_2O_6$ $[M + H]^+$ 549.2965, found 549.2972; HPLC (60% acetonitrile in water): t_R = 1.3 min, 99.1%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-((4-oxopiperidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (87). Yield: 40%; yellow solid; m.p.:



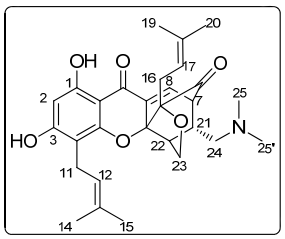
97-98 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09 (s, 3H, C_{19}-H), 1.38 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.82 (s, 3H, C_{14}-H), 2.20-2.36 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.42-2.46 (m, 4H, C_{26}-H, $C_{26'}$-H), 2.52-2.80 (m, 6H, C_{16}-H, C_{25}-H, $C_{25'}$-H), 3.33-3.35 (m, 2H, C_{11}-H), 3.69 (d, J = 6.9 Hz, 1H, C_7-H), 3.88 (d, J = 8.1 Hz, 1H, C_{23}-H_β), 4.38-4.40 (m, 1H, C_{17}-H), 4.54 (dd, J_1 = 3.3 Hz, J_2 = 8.4 Hz, 1H, C_{23}-H_α), 5.20-5.22 (m, 1H, C_{12}-H), 6.11 (s, 1H, C_2-H), 7.16 (d, J = 6.9 Hz, 1H, C_8-H), 12.46 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3250, 2969, 2919, 1737, 1712, 1644, 1634, 1596, 1428, 1273, 1175, 1137, 1085, 832, 734; EI-MS *m/z*: 547$[M]^+$(5), 450(17), 394(7), 112(100), 83(7), 58(12), 55(7); HRMS (ESI): calcd. for $C_{32}H_{37}NO_7$ $[M + H]^+$ 548.2648, found 548.2659; HPLC (60% acetonitrile in water): t_R = 1.7 min, 99.2%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-((2,4-dioxopyrrolidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (88). Yield: 39%; yellow solid; m.p.:



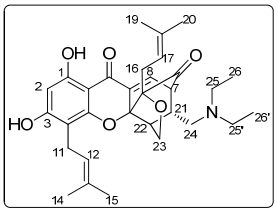
244-245 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.06 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.76 (s, 3H, C_{15}-H), 1.83 (s, 3H, C_{14}-H), 2.27-2.29 (m, 1H, C_{22}-H), 2.34-2.36 (m, 1H, C_{21}-H), 2.50-2.68 (m, 2H, C_{16}-H), 2.77 (s, 4H, C_{26}-H, $C_{26'}$-H), 3.31-3.34 (m, 2H, C_{11}-H), 3.36-3.41 (m, 3H, C_7-H, C_{24}-H), 3.94 (d, J= 8.1 Hz, 1H, C_{23}-H_β), 4.34-4.36 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.19 (t, J = 6.6 Hz, 1H, C_{12}-H), 6.06 (s, 1H, C_2-H), 6.80 (brs, 1H, C_3-OH), 7.24 (d, J = 6.6 Hz, 1H, C_8-H), 12.48 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3247, 2968, 2926, 1737, 1703, 1644, 1630, 1595, 1403, 1272, 1264, 1165, 1083, 820, 764, 750; EI-MS *m/z*: 547$[M]^+$(29), 519(20), 407(100), 311(73), 69(70), 55(48); HRMS (ESI): calcd. for $C_{31}H_{33}NO_8$ $[M + Na]^+$ 570.2104, found 570.2116; HPLC (60% acetonitrile in water): t_R = 7.1 min, 98.9%.

8,10-Dihydroxy-1,9-bis(3-methylbut-2-en-yl)-4-dimethylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (89). Yield: 40%; yellow solid; m.p.: 205-206 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.06 (s, 3H, C_{19}-H), 1.35 (s, 3H, C_{20}-H), 1.71 (s, 3H, C_{15}-H), 1.79 (s, 3H, C_{14}-H), 2.17-2.23 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.45 (s, 6H, C_{25}-H, $C_{25'}$-H), 2.52-2.67 (m, 2H, C_{16}-H), 3.24 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.55 (dd, J_1 = 2.1 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.86 (d, J = 8.1 Hz, 1H, C_{23}-H_β), 4.35-4.38 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.15 (t, J = 6.6 Hz,



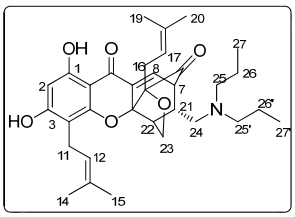
1H, C_{12}-H), 5.81 (s, 1H, C_2-H), 7.12 (d, J = 6.9 Hz, 1H, C_8-H), 12.49 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3363, 2962, 2914, 1738, 1643, 1629, 1596, 1431, 1275, 1257, 1131, 1048, 764, 750; EI-MS *m/z*: 493$[M]^+$(10), 396(91), 86(33), 71(18), 58(100), 55(19); HRMS (ESI): calcd. for $C_{29}H_{35}NO_6$ $[M + H]^+$ 494.2543, found 494.2551; HPLC (60% acetonitrile in water): t_R = 1.7 min, 99.2%.

4-Diethylaminomethyl-8,10-dihydroxy-1,9-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (90). Yield: 41%; yellow solid; m.p.: 120-121°C;



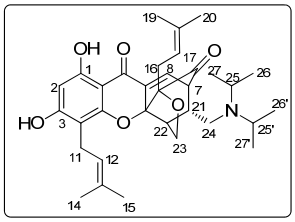
^{1}H NMR (300 MHz, $CDCl_3$): δ 0.96 (t, J = 6.9 Hz, 6H, C_{26}-H, $C_{26'}$-H), 1.08 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.15-2.30 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.42-2.57 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.60-2.69 (m, 2H, C_{16}-H), 3.32 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.65 (d, J = 6.6 Hz, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{23}-H_β), 4.38-4.40 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H_α), 5.20 (t, J = 7.2 Hz, 1H, C_{12}-H), 6.00 (s, 1H, C_2-H), 7.15 (d, J = 6.6 Hz, 1H, C_8-H), 12.49 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3286, 2958, 2926, 1735, 1644, 1634, 1602, 1434, 1275, 1134, 1087, 1057, 836, 742; EI-MS *m/z*: 521$[M]^+$(6), 424(51), 86(100), 58(23); HRMS (ESI): calcd. for $C_{31}H_{39}NO_6$ $[M + H]^+$ 522.2856, found 522.2865; HPLC (65% acetonitrile in water): t_R = 2.3 min, 98.8%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-dipropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (91). Yield: 41%; yellow solid; m.p.: 142-144 °C;



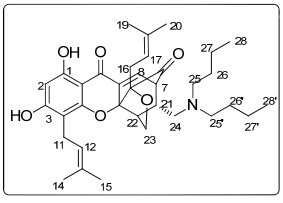
^{1}H NMR (300 MHz, $CDCl_3$): δ 0.86 (t, J = 7.2 Hz, 6H, C_{27}-H, $C_{27'}$-H), 1.09 (s, 3H, C_{19}-H), 1.31-1.41 (m, 7H, C_{20}-H, C_{26}-H, $C_{26'}$-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.15-2.24 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.24-2.38 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.50-2.65 (m, 2H, C_{16}-H), 3.32-3.34 (m, 2H, C_{11}-H), 3.67 (d, J = 6.9 Hz, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{23}-H_β), 4.38-4.41 (m, 1H, C_{17}-H), 4.50 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.18-5.20 (m, 1H, C_{12}-H), 6.00 (s, 1H, C_2-H), 7.13 (d, J = 6.9 Hz, 1H, C_8-H), 12.50 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3345, 2956, 2926, 1738, 1644, 1633, 1596, 1425, 1269, 1178, 1131, 1084, 827, 748; EI-MS *m/z*: 549$[M]^+$(6), 452(48), 114(100), 86(13), 72(11), 58(23); HRMS (ESI): calcd. for $C_{33}H_{43}NO_6$ $[M + H]^+$ 550.3169, found 550.3178; HPLC (65% acetonitrile in water): t_R = 5.5 min, 98.8%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-diisopropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (92). Yield: 38%; yellow solid; m.p.: 85-86 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ 0.90-0.99 (m, 12H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.10 (s, 3H, C_{19}-H), 1.33 (s, 3H, C_{20}-H), 1.71 (s, 3H, C_{15}-H), 1.80 (s, 3H, C_{14}-H), 2.18-2.32 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.55-2.64 (m, 2H, C_{16}-H), 2.94-3.01 (m, 2H, C_{25}-H, $C_{25'}$-H), 3.31-3.33 (m, 2H, C_{11}-H), 3.65-3.67 (m, 1H, C_7-H), 3.84 (d, J= 7.8 Hz, 1H, C_{23}-H_β), 4.40-4.42 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H_α), 5.19-5.21 (m, 1H, C_{12}-H), 5.72 (brs, 1H, C_3-OH), 5.97 (s, 1H, C_2-H), 7.13 (d, J = 6.6 Hz, 1H, C_8-H), 12.56 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3322, 2965, 2920, 1732, 1645, 1634, 1596, 1423, 1381, 1180, 826, 737; EI-MS *m/z*: 549$[M]^+$(10), 114(100), 72(88), 58(38); HRMS (ESI): calcd. for $C_{33}H_{43}NO_6$ $[M + H]^+$ 550.3169, found 550.3172; HPLC (60% acetonitrile in water): t_R = 10.8 min, 98.7%.

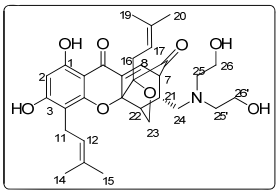
4-Dibutylaminomethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (93). Yield: 43%; yellow solid; m.p.: 130-131°C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ 0.89 (t, J = 6.9 Hz, 6H, C_{28}-H, $C_{28'}$-H), 1.09 (s, 3H, C_{19}-H), 1.28-1.31 (m, 8H, C_{26}-H, $C_{26'}$-H, C_{27}-H, $C_{27'}$-H), 1.37 (s, 3H, C_{20}-H), 1.71 (s, 3H, C_{15}-H), 1.79 (s, 3H, C_{14}-H), 2.15-2.27 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.32-2.43 (m, 4H, C_{25}-H, $C_{25'}$-H), 2.54-2.66 (m, 2H, C_{16}-H), 3.29-3.31 (m, 2H, C_{11}-H), 3.65 (d, J = 6.9 Hz, 1H, C_7-H), 3.86 (d, J = 7.8 Hz, 1H, C_{23}-H_β), 4.35-4.42 (m, 1H, C_{17}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H_α), 5.17-

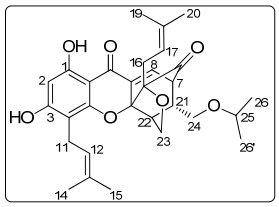
5.19 (m, 1H, C_{12}-H), 5.98 (s, 1H, C_2-H), 7.12 (d, J = 6.9 Hz, 1H, C_8-H); IR (cm^{-1}, KBr film): 3351, 2959, 2933, 1735, 1644, 1633, 1597, 1427, 1382, 1273, 1127, 1082, 828, 737; EI-MS *m/z*: 577$[M]^+$(10), 534(9), 480(85), 142(100), 100(43), 57(12); HRMS (ESI): calcd. for $C_{35}H_{47}NO_6$ $[M + H]^+$ 578.3482, found 578.3493; HPLC (65% acetonitrile in water): t_R = 15.7 min, 98.4%.

4-Diethanolaminomethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (94). Yield: 38%; yellow solid; m.p.: 128-130 °C;



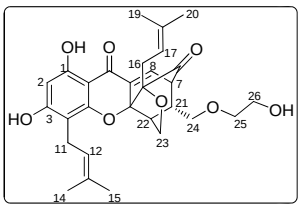
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.07 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.70 (s, 3H, C_{15}-H), 1.78 (s, 3H, C_{14}-H), 2.18-2.19 (m, 2H, C_{24}-H), 2.37-2.41 (m, 2H, C_{21}-H, C_{22}-H), 2.55-2.73 (m, 6H, C_{16}-H, C_{25}-H, $C_{25'}$-H), 3.11 (dd, J_1 = 7.2 Hz, J_2 = 14.7 Hz, 2H, C_{26}-OH, $C_{26'}$-OH), 3.28-3.30 (m, 2H, C_{11}-H), 3.58-3.62 (m, 4H, C_{26}-H, $C_{26'}$-H), 3.73-3.76 (m, 1H, C_7-H), 3.92 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.35-4.38 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 = 8.1 Hz, 1H, C_{23}-H$_\alpha$), 5.17-5.19 (m, 1H, C_{12}-H), 6.15 (s, 1H, C_2-H), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.48 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3357, 2962, 2926, 1732, 1642, 1593, 1431, 1272, 1071, 1036, 764, 750; EI-MS *m/z*: 553$[M]^+$(4), 522(8), 456(10), 118(100), 74(9), 56(8); HRMS (ESI): calcd. for $C_{31}H_{39}NO_8$ $[M + H]^+$ 554.2754, found 554.2768; HPLC (65% acetonitrile in water): t_R = 1.2 min, 98.7%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-isopropoxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (95). Yield: 40%; yellow solid; m.p.: 187-188 °C;



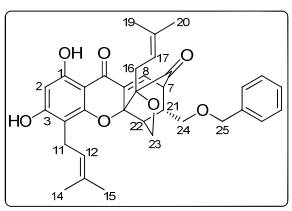
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09-1.12 (m, 9H, C_{19}-H, C_{26}-H, $C_{26'}$-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.19-2.34 (m, 2H, C_{21}-H, C_{22}-H), 2.54-2.65 (m, 2H, C_{16}-H), 3.16-3.20 (m, 2H, C_{24}-H), 3.34 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.47-3.51 (m, 1H, C_{25}-H), 3.70 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.92 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.38-4.40 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.19-5.21 (m, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 6.79 (brs, 1H, C_3-OH), 7.14 (d, J = 6.6 Hz, 1H, C_8-H), 12.49 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3439, 2979, 2914, 1738, 1644, 1634, 1593, 1431, 1275, 1125, 1081, 827, 768, 754; EI-MS *m/z*: 508$[M]^+$(42), 480(33), 437(58), 407(100), 311(69), 69(94); HRMS (ESI): calcd. for $C_{30}H_{36}O_7$ $[M+H]^+$ 509.2539, found 509.2508; HPLC (60% acetonitrile in water): t_R = 2.0 min, 98.8%.

8,10-Dihydroxy-4-hydroxyethoxymethyl-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (96). Yield: 36%; yellow solid; m.p.: 102-104 °C;



^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.20-2.22 (m, 1H, C_{22}-H), 2.38-2.40 (m, 1H, C_{21}-H), 2.51-2.70 (m, 2H, C_{16}-H), 3.27-3.34 (m, 4H, C_{11}-H, C_{24}-H), 3.50-3.53 (m, 2H, C_{26}-H), 3.66-3.74 (m, 3H, C_7-H, C_{25}-H), 3.92 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.37-4.39 (m, 1H, C_{17}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.17-5.19 (m, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 6.78 (brs, 1H, C_3-OH), 7.14 (d, J = 6.9 Hz, 1H, C_8-H), 12.46 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3398, 2962, 2926, 1735, 1645, 1634, 1434, 1272, 1116, 1063, 824, 742; EI-MS *m/z*: 510$[M]^+$(13), 407(83), 255(67), 207(79), 69(100), 55(60); HRMS (ESI): calcd. for $C_{29}H_{34}O_8$ $[M+H]^+$ 511.2332, found 511.2300; HPLC (50% acetonitrile in water): t_R = 1.1 min, 98.1%.

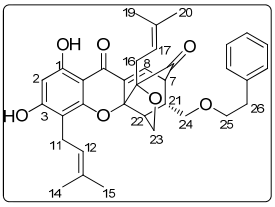
4-Benzyloxymethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (97). Yield: 34%; yellow solid; m.p. 59-60 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ 1.08 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.18-2.20 (m, 1H, C_{22}-H), 2.38-2.40 (m, 1H, C_{21}-H), 2.51-2.69 (m, 2H, C_{16}-H), 3.24 (d, J = 7.8 Hz, 2H, C_{24}-H), 3.32 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.70 (dd, J_1 = 3.0 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.90 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.36-4.40 (m, 1H, C_{17}-H), 4.44-4.52 (m, 3H, C_{23}-H$_\alpha$, C_{25}-H), 5.16-5.19 (m, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 6.78 (brs, 1H, C_3-OH), 7.06 (d, J = 6.6 Hz, 1H, C_8-H), 7.26-7.34 (m, 5H, Ph), 12.48 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3333, 2973, 2914, 1738, 1644, 1634, 1596, 1428, 1269, 1084, 830, 749, 695; EI-MS

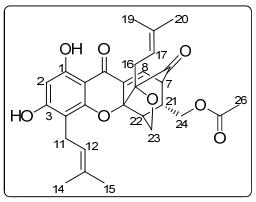
m/z: 556[M]$^+$(11), 407(24), 311(16), 91(100), 69(29); HRMS (ESI): calcd. for $C_{34}H_{36}O_7$ [M+H]$^+$ 557.2539, found 557.2547; HPLC (60% acetonitrile in water): t_R = 2.3 min, 98.5%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-phenethoxylmethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (98). Yield: 32%; yellow solid; m.p.: 44-45 °C; ^{1}H



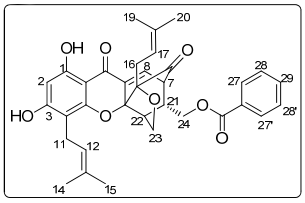
NMR (300 MHz, $CDCl_3$): δ 1.07 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.09-2.11 (m, 1H, C_{22}-H), 2.31-2.33 (m, 1H, C_{21}-H), 2.50-2.66 (m, 2H, C_{16}-H), 2.83 (t, J = 6.6 Hz, 2H, C_{26}-H), 3.18 (d, J = 7.8 Hz, 2H, C_{24}-H), 3.32 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.54-3.63 (m, 3H, C_7-H, C_{25}-H), 3.85 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.35-4.38 (m, 1H, C_{17}-H), 4.46 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.19 (t, J = 6.6 Hz, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 6.94 (brs, 1H, C_3-OH), 7.03 (d, J = 6.6 Hz, 1H, C_8-H), 7.16-7.29 (m, 5H, Ph), 12.49 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3298, 2973, 2909, 1741, 1643, 1633, 1600, 1428, 1275, 1107, 821, 748, 695; EI-MS *m/z*: 570[M]$^+$(17), 542(21), 407(46), 311(23), 105(100), 79(21), 69(29); HRMS (ESI): calcd. for $C_{35}H_{38}O_7$ [M+H]$^+$ 571.2696, found 571.2668; HPLC (60% acetonitrile in water): t_R = 2.8 min, 98.4%.

4-Acetoxymethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (99). Yield: 38%; yellow solid; m.p.: 138-140 °C; ^{1}H NMR



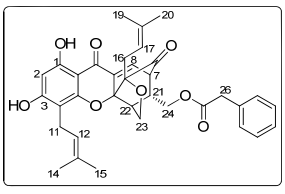
(300 MHz, $CDCl_3$): δ 1.09 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.07 (s, 3H, C_{26}-H), 2.19-2.21 (m, 1H, C_{22}-H), 2.41-2.43 (m, 1H, C_{21}-H), 2.52-2.71 (m, 2H, C_{16}-H), 3.32-3.34 (m, 2H, C_{11}-H), 3.64 (dd, J_1 = 3.0 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.76-3.82 (m, 1H, C_{24}-H$_\alpha$), 3.93-3.99 (m, 2H, C_{23}-H$_\beta$, C_{24}-H$_\beta$), 4.36-4.38 (m, 1H, C_{17}-H), 4.53 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.19 (t, J = 5.4 Hz, 1H, C_{12}-H), 6.06 (s, 1H, C_2-H), 6.75 (s, 1 H, C_3-OH), 7.15 (d, J = 6.9 Hz, 1H, C_8-H), 12.43 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3268, 2968, 2920, 1741, 1644, 1634, 1593, 1425, 1260, 1042, 801, 765, 749; EI-MS *m/z*: 508[M]$^+$(34), 407(37), 311(23), 69(100), 55(35); HRMS (ESI): calcd. for $C_{29}H_{32}O_8$ [M+H]$^+$ 509.2175, found 509.2170; HPLC (50% acetonitrile in water): t_R = 1.6 min, 98.4%.

4-Benzoyloxymethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (100). Yield: 36%; yellow solid; m.p.: 92-93 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ 1.09 (s, 3H, C_{19}-H), 1.37 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.30-2.32 (m, 1H, C_{22}-H), 2.58-2.68 (m, 3H, C_{21}-H, C_{16}-H), 3.27-3.41 (m, 2H, C_{11}-H), 3.72-3.76 (m, 1H, C_7-H), 3.98-4.01 (m, 1H, C_{24}-H$_\alpha$), 4.03-4.10 (m, 1H, C_{24}-H$_\beta$), 4.20-4.22 (m, 1H, C_{23}-H$_\beta$), 4.32-4.35 (m, 1H, C_{17}-H), 4.55-4.58 (m, 1H, C_{23}-H$_\alpha$), 5.19-5.21 (m, 1H, C_{12}-H), 6.07 (s, 1H, C_2-H), 6.90 (brs, 1H, C_3-OH), 7.21 (d, J = 6.6 Hz, 1H, C_8-H), 7.43-7.48 (m, 2H, C_{28}-H, $C_{28'}$-H), 7.57-7.60 (m, 1H, C_{29}-H), 8.00 (d, J = 7.8 Hz, 2H, C_{27}-H, $C_{27'}$-H), 12.44 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3369, 2956, 2914, 1744, 1723, 1644, 1634, 1600, 1428, 1268, 1110, 798, 709; EI-MS *m/z*: 570[M]$^+$(12), 407(21), 122(41), 105(100), 77(80), 58(37); HRMS (ESI): calcd. for $C_{34}H_{34}O_8$ [M + H]$^+$ 571.2332, found 459.1796; HPLC (50% acetonitrile in water): t_R = 2.7 min, 99.4%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-phenylacetyloxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (101). Yield: 36%; yellow solid; m.p.: 47-48 °C; ^{1}H



NMR (300 MHz, $CDCl_3$): δ 1.07 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.74 (s, 3H, C_{15}-H), 1.81 (s, 3H, C_{14}-H), 2.12-2.14 (m, 1H, C_{22}-H), 2.36-2.38 (m, 1H, C_{21}-H), 2.54-2.65 (m, 2H, C_{16}-H), 3.32 (d, J = 6.9 Hz, 2H, C_{11}-H), 3.51 (dd, J_1 = 3.0 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.62 (s, 2H, C_{26}-H), 3.78-3.86 (m, 2H, C_{24}-H), 3.91-3.93 (m, 1H, C_{23}-H$_\beta$), 4.42-4.43 (m, 1H, C_{17}-H), 4.44-4.45 (m, 1H, C_{23}-H$_\alpha$), 5.17-5.19 (m, 1H, C_{12}-H), 6.05 (s, 1H, C_2-H), 6.78 (brs, 1H, C_3-OH), 7.07 (d, J = 6.6 Hz, 1H, C_8-H), 7.24-7.34 (m, 5H, Ph), 12.42 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3351, 2962, 2902, 1735, 1644, 1635, 1593, 1425, 1272, 1137, 830, 803; EI-MS *m/z*: 584[M]$^+$(17), 556(24), 407(29),

311(20), 91(100), 69(36); HRMS (ESI): calcd. for $C_{35}H_{36}O_8$ $[M+H]^+$ 585.2488, found 585.2456; HPLC (60% acetonitrile in water): t_R = 2.6 min, 99.3%.

4-Cinnamyloxymethyl-8,10-dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (102). Yield: 37%; yellow solid; m.p.: 89-90 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{19}-H), 1.30 (s, 3H, C_{20}-H), 1.67 (s, 3H, C_{15}-H), 1.74 (s, 3H, C_{14}-H), 2.19-2.21 (m, 1H, C_{22}-H), 2.46-2.65 (m, 3H, C_{16}-H, C_{21}-H), 3.27 (d, J = 6.6 Hz, 2H, C_{11}-H), 3.64 (dd, J_1 = 3.0 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.84-3.91 (m, 2H, C_{23}-H$_\beta$, C_{24}-H$_\alpha$), 4.00-4.06 (m, 1H, C_{24}-H$_\beta$), 4.30-4.33 (m, 1H, C_{17}-H), 4.48 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 5.13 (t, J = 6.6 Hz, 1H, C_{12}-H), 5.99 (s, 1H, C_2-H), 6.37 (d, J = 16.0 Hz, 1H, C_{26}-H), 6.73 (brs, 1H, C_3-OH), 7.12 (d, J = 6.9 Hz, 1H, C_8-H), 7.34-7.35 (m, 3H, C_{30}-H, $C_{30'}$-H, C_{31}-H), 7.45-7.47 (m, 2H, C_{29}-H, $C_{29'}$-H), 7.62 (d, J = 16.0 Hz, 1H, C_{27}-H), 12.38 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3339, 2968, 2914, 1741, 1711, 1644, 1629, 1602, 1428, 1272, 1166, 765, 749; EI-MS *m/z*: 596$[M]^+$(10), 568(14), 407(24), 147(41), 131(100), 103(86), 69(41); HRMS (ESI): calcd. for $C_{36}H_{36}O_8$ $[M+H]^+$ 597.2488, found 597.2470; HPLC (60% acetonitrile in water): t_R = 3.0 min, 98.3%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-((4-phenylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (103). Yield: 43%; yellow solid; m.p.: 189-190 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.07 (s, 3H, C_{19}-H), 1.36 (s, 3H, C_{20}-H), 1.72 (s, 3H, C_{15}-H), 1.80 (s, 3H, C_{14}-H), 2.21-2.31 (m, 4H, C_{21}-H, C_{22}-H, C_{24}-H), 2.51-2.70 (m, 6H, C_{16}-H, C_{26}-H, $C_{26'}$-H), 3.16-3.18 (m, 4H, C_{25}-H, $C_{25'}$-H), 3.29-3.31 (m, 2H, C_{11}-H), 3.67 (dd, J_1 = 2.1 Hz, J_2 = 6.6 Hz, 1H, C_7-H), 3.88 (d, J = 7.8 Hz, 1H, C_{23}-H$_\beta$), 4.36-4.39 (m, 1H, C_{17}-H), 4.53 (dd, J_1 = 3.6 Hz, J_2 = 7.8 Hz, 1H, C_{23}-H$_\alpha$), 4.71 (s, 1H, C_3-OH), 5.16-5.19 (m, 1H, C_{12}-H), 5.98 (s, 1H, C_2-H), 6.85-6.93 (m, 3H, C_{29}-H, $C_{29'}$-H, C_{30}-H), 7.16 (d, J = 6.6 Hz, 1H, C_8-H), 7.24-7.38 (m, 2H, C_{28}-H, $C_{28'}$-H), 12.50 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3428, 2963, 2906, 1741, 1644, 1633, 1584, 1415, 1260, 1095, 1024, 799, 765, 750, 698; EI-MS *m/z*: 610$[M]^+$(35), 513(90), 175(81), 160(59), 132(59), 104(36), 70(100); HRMS (ESI): calcd. for $C_{37}H_{42}N_2O_6$ $[M + H]^+$ 611.3121, found 611.3120; HPLC (60% acetonitrile in water): t_R = 6.0 min, 99.2%.

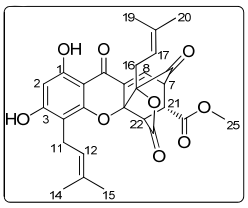
General procedure for preparation of caged compounds 104-107. A solution of required substrate (**41a-d**, 100 mg) in DMF (2 mL) was stirred under nitrogen at 120 °C for 2 h. The reaction mixture was then cooled to room temperature and concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford caged compound **104-107**.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-3,7,13-trione (104). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, C_{14}-H, C_{15}-H, C_{19}-H, C_{20}-H), 2.33-2.36 (m, 1H, C_{21}-H$_\alpha$), 2.55-2.90 (m, 4H, C_{16}-H, C_{21}-H$_\beta$, C_{22}-H), 3.23-3.26 (m, 2H, C_{11}-H), 3.74-3.76 (m, 1H, C_7-H), 4.24-4.28 (m, 1H, C_{17}-H), 5.06-5.10 (m, 1H，C_{12}-H), 6.02 (s, 1H, C_2-H), 6.37 (s, 1H, OH), 7.35 (d, J = 6.9 Hz, 1H, C_8-H), 12.25 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 2924, 2365, 1798, 1742, 1638, 1597, 1431, 1370, 1223, 1093, 797; EI-MS *m/z*: 450$[M]^+$(33), 396(89), 341(100), 273(80); HRMS (ESI): calcd. for $C_{26}H_{26}O_7$ $[M + H]^+$ 451.1757, found 451.1750; HPLC (80% acetonitrile in water): t_R = 2.5 min, 98.7%.

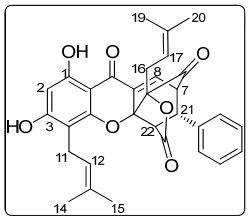
8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-methyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-3,7,13-trione (105). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, C_{14}-H, C_{15}-H, C_{19}-H, C_{20}-H), 1.73-1.77 (m, 3H, C_{24}-H), 2.54-2.57 (m, 1H, C_{21}-H), 2.76-2.80 (m, 1H, C_{22}-H), 3.13-3.17 (m, 2H, C_{16}-H), 3.63-3.67 (m, 1H, C_7-H), 3.75-3.81 (m, 2H, C_{11}-H), 4.25-5.03 (m, 1H, C_{17}-H), 5.06-5.10 (m, 1H, C_{12}-H), 6.02 (s, 1H, C_3-OH), 6.40 (s, 1H, C_2-H), 7.19 (d, J = 6.9 Hz, 1H, C_8-H), 12.26 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 2922, 2362, 1795, 1744, 1631, 1587, 1431, 1370, 1223, 1093, 799; EI-MS *m/z*: 464 $(M)^+$, 436, 396; HRMS (ESI): calcd. for $C_{27}H_{28}O_7$ $[M + H]^+$,

465.1913, found 465.1887; HPLC: (80% acetonitrile in water): t_R = 3.3 min, 97.9%.

8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-methoxycarbonyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-3,7,13-trione (106). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.61 (m, 12H, C_{14}-H, C_{15}-H, C_{19}-H, C_{20}-H), 2.33-2.37 (m, 1H, C_{21}-H), 2.55-2.90 (m, 4H, C_{16}-H, C_{21}-H, C_{22}-H), 3.23-3.27 (m, 2H, C_{11}-H), 3.53-3.57 (m, 1H, C_7-H), 3.76 (s, 3H, C_{25}-H), 4.24-4.28 (m, 1H, C_{17}-H), 5.07-5.10 (m, 1H, C_{12}-H), 6.07 (s, 1H, C_2-H), 7.35 (d, J = 6.9 Hz, 1H, C_8-H), 12.19 (s,1H, C_1-OH); IR (cm^{-1}, KBr film): 2960, 2852, 1804, 1745, 1639, 1599, 1432, 1311, 1181, 1140, 735; EI-MS *m/z*: 508 $(M)^+$, 453, 425; HRMS (ESI): calcd. for $C_{28}H_{28}O_9$ $[M + H]^+$ 509.1812, found 509.1817; HPLC (80% acetonitrile in water): t_R = 2.0 min, 97.2%.

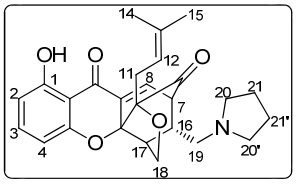


8,10-Dihydroxy-1,11-bis(3-methylbut-2-en-yl)-4-phenyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-3,7,13-trione (107). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51-1.60 (m, 12H, C_{14}-H, C_{15}-H, C_{19}-H, C_{20}-H), 2.61-3.01 (m, 3H, C_{16}-H$_\alpha$, C_{21}-H, C_{22}-H), 3.33-3.37 (m, 2H, C_{11}-H), 3.73-3.77 (m, 2H, C_{16}-H$_\beta$), 4.03-4.06 (m, 1H, C_7-H), 4.34-4.38 (m, 1H, C_{17}-H), 5.14-5.18 (m, 1H, C_{12}-H), 6.09 (s, 1H, C_3-OH), 6.7 (s, 1H, C_2-H), 7.06 (d, J = 6.9 Hz, 1H, C_8-H), 6.95-7.30 (m, 5H, Ph), 12.35 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3051, 2924, 2365, 1798, 1742, 1638, 1597, 1578, 1535, 1501, 1431, 1370, 1223, 1093, 797; EI-MS (m/z) 526 $(M)^+$, 452, 437, 396; HRMS (ESI): calcd. for $C_{32}H_{30}O_7$ $[M + H]^+$ 527.2070, found 527.2076; HPLC (80% acetonitrile in water): t_R = 3.0 min, 98.4%.

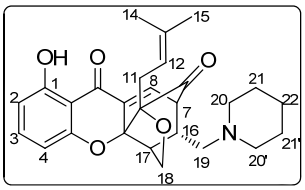


General procedure for preparation of caged compounds 108-129 and 130. To a stirring solution of compound **42 or 44** (0.20 mmol) in DMF (5 mL) was added potassium carbonate (0.40 mmol) and the corresponding amine or alcohol or acid (0.30 mmol) and the solution was stirred at 35°C for 1 h. The reaction mixture was partitioned between ethyl acetate (15 mL) and water (20 mL). The water layer was extracted with ethyl acetate (15 mL × 2). The organic layer was combined, washed with saturated NaCl solution (10 mL × 2), dried over sodium sulfate, fiterted and concentrated to afford **43 or 45** as yellow oil. The yellow oil was then dissolved in DMF (3 mL) and the reaction solution was stirred at 120 °C under nitrogen for 2 h. The reaction mixture was cooled to room temperature and concentrated. The residue was purified by column chromatography (8:1 petroleum ether/ethyl acetate) to afford **108-129** and **130**.

8-Hydroxy-1-(3-methyl-2–butenyl)-4-(pyrrolidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (108). Yield: 64%; yellow solid; m.p.: 148-150 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.04 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 1.74-1.76 (m, 4H, C_{21}-H, $C_{21'}$-H), 2.23-2.27 (m, 2H, C_{16}-H, C_{17}-H), 2.31-2.45 (m, 6H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.57-2.70 (m, 2H, C_{11}-H), 3.66-3.68 (m, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.54 (dd, J_1 = 3.3 Hz, J_2 =9.0 Hz, 1H, C_{18}-H$_\alpha$), 6.48 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.1 Hz, 1H, C_4-H), 7.23 (d, J = 6.9 Hz, 1H, C_8-H), 7.38 (t, J = 8.4 Hz, 1H, C_3-H), 12.12 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2985, 2926, 1738, 1644, 1600, 1462, 1382, 1275, 1046, 764, 750; EI-MS *m/z*: 435$[M]^+$(7), 338(51), 84(48), 58(100); HRMS (ESI): calcd. for $C_{26}H_{29}NO_5$ $[M + H]^+$ 436.2124, found 436.2122; HPLC (75% methanol in water): t_R = 6.6 min, 99.2%.



8-Hydroxy-1-(3-methyl-2–butenyl)-4-(piperidin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (109). Yield: 67%; yellow solid; m.p.: 138-140 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 1.40-1.53 (m, 6H, C_{21}-H, $C_{21'}$-H, C_{22}-H), 2.05-2.12 (m, 2H, C_{16}-H, C_{17}-H), 2.22-2.24 (m, 6H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.57-2.70 (m, 2H, C_{11}-H), 3.66-3.68 (dd, J_1 = 3.0 Hz, J_2 = 6.9 Hz, 1H, C_7-H), 3.85 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.23 (d, J = 6.9 Hz, 1H, C_8-H),



7.38 (t, J = 8.4 Hz, 1H, C_3-H), 12.13 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3452, 2985, 2914, 1738, 1683, 1644, 1600, 1462, 1380, 1276, 1261, 1228, 1141, 1045, 800, 761, 743; EI-MS *m/z*: 449$[M]^+$(5), 352(93), 98(100), 55(12); HRMS (ESI): calcd. for $C_{27}H_{31}NO_5$ $[M + H]^+$ 450.2280, found 450.2273; HPLC (75% methanol in water): t_R = 15.3 min, 99.3%.

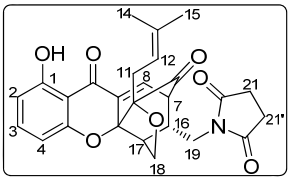
8-Hydroxy-1-(3-methyl-2–butenyl)-4-morpholinomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (110). Yield: 59%; yellow solid; m.p.: 202-204 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.06 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.08-2.21 (m, 2H, C_{16}-H, C_{17}-H), 2.30-2.44 (m, 6H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.57-2.70 (m, 2H, C_{11}-H), 3.68-3.69 (m, 5H, C_7-H, C_{21}-H, $C_{21'}$-H), 3.84 (d, J = 8.1 Hz, 1H, C_{18}-H$_\beta$), 4.36-4.41 (m, 1H, C_{12}-H), 4.53 (dd, J_1 = 3.9 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 9.3 Hz, 1H, C_2-H), 6.55 (d, J = 9.3Hz, 1H, C_4-H), 7.23 (d, J = 6.9 Hz, 1H, C_8-H), 7.40 (t, J = 8.4 Hz, 1H, C_3-H), 12.09 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2967, 2926, 1738, 1637, 1596, 1458, 1375, 1275, 1260, 1228, 1054, 1033, 807, 764, 750; EI-MS *m/z*: 451$[M]^+$(12), 354(94), 169(20), 100(100), 56(38); HRMS (ESI): calcd. for $C_{26}H_{29}NO_6$ $[M + H]^+$ 452.2073, found 452.2066; HPLC (60% acetonitrile in water): t_R = 10.0 min, 99.4%.

8-Hydroxy-1-(3-methyl-2–butenyl)-4-(piperazin-1-ylmethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (111). Yield: 51%; yellow solid; m.p.: 239-241 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.34 (s, 3H, C_{15}-H), 2.13-2.37 (m, 12H, C_{16}-H, C_{17}-H ,C_{19}-H, C_{20}-H, $C_{20'}$-H, C_{21}-H, $C_{21'}$-H), 2.57-2.65 (m, 2H, C_{11}-H), 3.66-3.68 (m, 1H, C_7-H), 3.84 (d, J = 6.0 Hz, 1H, C_{18}-H$_\beta$), 4.35-4.37 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 9.3 Hz, 1H, C_2-H), 6.55 (d, J = 9.3 Hz, 1H, C_4-H), 7.22 (d, J = 6.9 Hz, 1H, C_8-H), 7.39 (t, J = 8.1 Hz, 1H, C_3-H), 12.09 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3463, 2963, 2808, 1740, 1644, 1600, 1462, 1374, 1262, 1229, 1161, 1060, 1030, 802, 742; EI-MS *m/z*: 450$[M]^+$(11), 353(91), 99(100), 84(36), 70(34), 56(48); HRMS (ESI): calcd. for $C_{26}H_{30}N_2O_5$ $[M + H]^+$ 451.2233, found 451.2216; HPLC (95% methanol in water): t_R = 3.1 min, 98.6%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-((4-methylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (112). Yield: 55%; yellow solid; m.p.: 145-147 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.10-2.27 (m, 6H, C_{16}-H, C_{17}-H, C_{21}-H, $C_{21'}$-H), 2.35 (s, 3H, C_{22}-H), 2.51-2.57 (m, 6H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.62-2.70 (m, 2H, C_{11}-H), 3.65-3.68 (m, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.39 (m, 1H, C_{12}-H), 4.53 (dd, J_1 = 3.3 Hz, J_2 =8.1 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.55 (d, J = 8.4 Hz, 1H, C_4-H), 7.22 (d, J = 6.9 Hz, 1H, C_8-H), 7.41 (t, J = 8.4 Hz, 1H, C_3-H), 12.10 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2932, 2796, 1740, 1644, 1600, 1462, 1375, 1230, 1160, 1101, 1060, 1028, 801, 739; EI-MS *m/z*: 464$[M]^+$(16), 367(100), 113(79), 98(53), 70(92); HRMS (ESI): calcd. for $C_{27}H_{32}N_2O_5$ $[M + H]^+$ 465.2389, found 465.2380; HPLC (60% acetonitrile in water): t_R = 8.2 min, 99.5%.

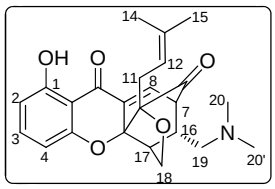
8-Hydroxy-1-(3-methylbut-2-en-yl)-4-((4-oxopiperidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (113). Yield: 60%; yellow solid; m.p.: 182-184 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.03 (s, 3H, C_{14}-H), 1.36 (s, 3H, C_{15}-H), 2.17-2.44 (m, 6H, C_{16}-H, C_{17}-H, C_{21}-H, $C_{21'}$-H), 2.58-2.71 (m, 8H, C_{11}-H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 3.72-3.74 (m, 1H, C_7-H), 3.86 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.37-4.40 (m, 1H, C_{12}-H), 4.56 (dd, J_1 = 3.9 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.51 (d, J = 8.4 Hz, 1H, C_2-H), 6.56 (d, J = 8.4 Hz, 1H, C_4-H), 7.25 (d, J = 6.9 Hz, 1H, C_8-H), 7.41 (t, J = 8.4 Hz, 1H, C_3-H), 12.08 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3404, 2973, 2920, 1741, 1641, 1593, 1458, 1375, 1225, 1060, 1033, 807, 754; EI-MS *m/z*: 463$[M]^+$(8), 366(45), 112(100), 73(44); HRMS (ESI): calcd. for $C_{27}H_{29}NO_6$ $[M + H]^+$ 464.2073, found 464.2069; HPLC (60% acetonitrile in water): t_R = 8.2 min, 99.0%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-((2,4-oxopyrrolidin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (114). Yield: 52%; yellow solid; m.p.: 220-222 °C;



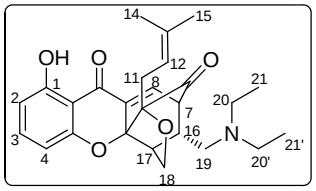
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.28-2.30 (m, 1H, C_{16}-H), 2.35-2.39 (m, 1H, C_{17}-H), 2.57-2.65 (m, 2H, C_{11}-H), 2.71-2.76 (m, 4H, C_{20}-H, $C_{20'}$-H), 3.37-3.42 (m, 3H, C_{19}-H, C_7-H), 3.92 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.35-4.37 (m, 1H, C_{12}-H), 4.51 (dd, J_1 = 3.6 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.56 (d, J = 8.4 Hz, 1H, C_4-H), 7.33 (d, J = 6.6 Hz, 1H, C_8-H), 7.40 (t, J = 8.4 Hz, H, C_3-H), 12.09 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3451, 2973, 2914, 1738, 1705, 1646, 1602, 1458, 1375, 1349, 1222, 1172, 803; EI-MS *m/z*: 463[M]$^+$(5), 323(100), 239(46), 227(77), 69(57), 55(47); HRMS (ESI): calcd. for $C_{26}H_{25}NO_7$ [M + Na]$^+$ 486.1529, found 486.1516; HPLC (75% methanol in water): t_R = 1.3 min, 99.3%.

4-Dimethylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (115). Yield: 57%; yellow solid; m.p.: 170-172 °C; ^{1}H NMR



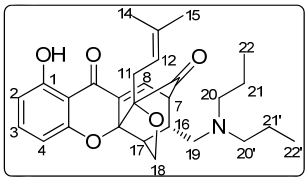
(300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.19-2.33 (m, 8H, C_{16}-H, C_{17}-H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.57-2.65 (m, 2H, C_{11}-H), 3.65-3.66 (m, 1H, C_7-H), 3.87 (d, J = 7.2 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.54 (dd, J_1 = 3.6 Hz, J_2 =7.5 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 7.8 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.23 (d, J = 6.9 Hz, 1H, C_8-H), 7.39 (t, J = 8.4 Hz, 1H, C_3-H), 12.10 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3451, 2973, 2896, 1738, 1640, 1590, 1461, 1375, 1260, 1228, 1045, 1028, 801, 764, 750; EI-MS *m/z*: 409[M]$^+$(6), 312(40), 84(56), 58(100); HRMS (ESI): calcd. for $C_{24}H_{27}NO_5$ [M + H]$^+$ 410.1967, found 410.1963; HPLC (60% acetonitrile in water): t_R = 6.9 min, 98.8%.

4-Diethylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (116). Yield: 68%; yellow solid; m.p.: 104-105 °C; ^{1}H NMR



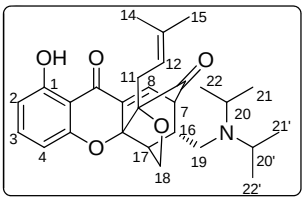
(300 MHz, $CDCl_3$): δ 0.95 (t, 6H, C_{21}-H, $C_{21'}$-H), 1.03 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.10-2.26 (m, 4H, C_{16}-H, C_{17}-H, C_{19}-H), 2.35-2.56 (m, 4H, C_{20}-H, $C_{20'}$-H), 2.61-2.70 (m, 2H, C_{11}-H), 3.69-3.71 (m, 1H, C_7-H), 3.85 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.39-4.41 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 =7.5 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.22 (d, J = 6.9 Hz, 1H, C_8-H), 7.40 (t, J = 8.1 Hz, 1H, C_3-H), 12.12 (s, 1H, C_1-OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 11.2, 16.4, 24.9, 27.3, 39.7, 46.3, 47.1, 49.6, 55.1, 74.0, 81.9, 87.6, 105.7, 106.9, 108.9, 117.5, 131.0, 132.2, 135.4, 138.1, 159.1, 162.5, 180.5, 199.8; IR (cm^{-1}, KBr film): 3457, 2968, 2932, 1741, 1644, 1600, 1463, 1374, 1276, 1261, 1057, 804, 764, 750; HPLC: EI-MS *m/z*: 437[M]$^+$(7), 340(46), 86(100), 58(13); HRMS (ESI): calcd. for $C_{26}H_{31}NO_5$ [M + H]$^+$ 438.2280, found 438.2279; HPLC (60% acetonitrile in water): t_R = 26.3 min, 98.6%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-dipropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (117). Yield: 70%; yellow solid; m.p.: 116-118 °C; ^{1}H NMR



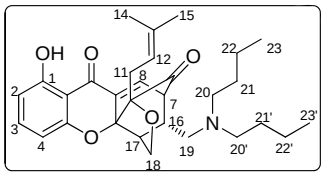
(300 MHz, $CDCl_3$): δ 0.95 (t, J = 6.6 Hz, 6H, C_{22}-H, $C_{22'}$-H), 1.03 (s, 3H, C_{14}-H), 1.35-1.43 (m, 7H, C_{15}-H, C_{21}-H, $C_{21'}$-H), 2.14-2.40 (m, 8H, C_{16}-H, C_{17}-H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 2.56-2.70 (m, 2H, C_{11}-H), 3.70-3.72 (m, 1H, C_7-H), 3.86 (d, J = 8.4 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.9 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.1 Hz, 1H, C_2-H), 6.54 (d, J = 8.1Hz, 1H, C_4-H), 7.22 (d, J = 6.9 Hz, 1 H, C_8-H), 7.39 (t, J = 8.4 Hz, 1H, C_3-H), 12.13 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3445, 2962, 2932, 1738, 1641, 1596, 1464, 1378, 1275, 1260, 1063, 1031, 804, 763, 747; EI-MS *m/z*: 465[M]$^+$(5), 368(38), 114(95), 84(100); HRMS (ESI): calcd. for $C_{28}H_{35}NO_5$ [M + H]$^+$ 466.2593, found 466.2591; HPLC (85% methanol in water): t_R = 26.3 min, 99.1%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-diisopropylaminomethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (118). Yield: 56%; yellow solid; m.p.: 156-158 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90-1.02 (m, 15H, C_{14}-H, C_{21}-H, $C_{21'}$-H, C_{22}-H, $C_{22'}$-H), 1.35 (s, 3H, C_{15}-H),



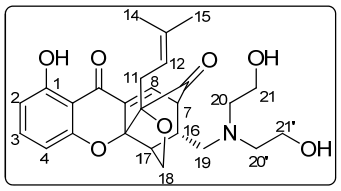
2.20-2.32 (m, 4H, C_{16}-H, C_{17}-H, C_{20}-H, $C_{20'}$-H), 2.61-2.64 (m, 2H, C_{11}-H), 2.92-3.00 (m, 2H, C_{19}-H), 3.69-3.71 (m, 1H, C_7-H), 3.84 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.38-4.40 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.23 (d, J = 6.9 Hz, 1H, C_8-H), 7.39 (t, J = 8.4 Hz, 1H, C_3-H), 12.15 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2966, 2920, 1740, 1644, 1600, 1462, 1376, 1261, 1229, 1061, 1030, 803, 764, 749; EI-MS *m/z*: 465[M]$^+$(3), 114(100), 72(21); HRMS (ESI): calcd. for $C_{28}H_{35}NO_5$ [M + H]$^+$ 466.2593, found 466.2587; HPLC (85% methanol in water): t_R = 9.5 min, 98.4%.

4-Dibutylaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (119). Yield: 60%; yellow solid; m.p.: 96-98 °C; ^{1}H NMR



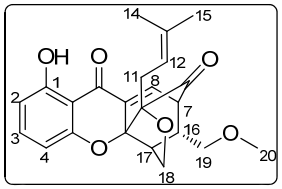
(300 MHz, $CDCl_3$): δ 0.89 (t, J = 6.9 Hz, 6H, C_{23}-H, $C_{23'}$-H), 1.03 (s, 3H, C_{14}-H), 1.24-1.35 (m, 11H, C_{15}-H, C_{21}-H, $C_{21'}$-H, C_{22}-H, $C_{22'}$-H), 2.12-2.41 (m, 8H, C_{16}-H, C_{17}-H, C_{19}-H , C_{20}-H, $C_{20'}$-H), 2.57-2.65 (m, 2H, C_{11}-H), 3.68-3.71 (m, 1H, C_7-H), 3.85 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.37-4.42 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 =7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.21 (d, J = 6.9 Hz, 1H, C_8-H), 7.38 (t, J = 8.4 Hz, 1H, C_3-H), 12.14 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2956, 2926, 2869, 1738, 1644, 1372, 1275, 1260, 1057, 1030, 804, 749; EI-MS *m/z*: 493[M]$^+$(7), 396(58), 142(76), 86(66), 84(100), 57(59); HRMS (ESI): calcd. for $C_{20}H_{39}NO_5$ [M + H]$^+$ 494.2906, found 494.2900; HPLC (95% acetonitrile in water): t_R = 7.9 min, 99.5%.

4-Diethanolaminomethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (120). Yield: 62%; yellow solid; m.p.: 154-156 °C; ^{1}H NMR



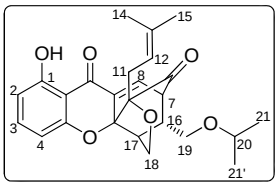
(300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.24-2.41 (m, 4H, C_{16}-H, C_{17}-H, C_{19}-H), 2.54-2.74 (m, 6H, C_{11}-H, C_{20}-H, $C_{20'}$-H), 3.59-3.62 (m, 4H, C_{21}-H, $C_{21'}$-H), 3.78-3.81 (m, 1H, C_7-H), 3.91 (d, J = 7.8 Hz, 1H, C_{18}-H$_\beta$), 4.37-4.38 (m, 1H, C_{12}-H), 4.52 (dd, J_1 = 3.6 Hz, J_2 =8.1 Hz, 1 H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.4 Hz, 1H, C_2-H), 6.54 (d, J = 8.4 Hz, 1H, C_4-H), 7.27 (d, J = 6.9 Hz, 1H, C_8-H), 7.39 (t, J = 8.4 Hz, 1H, C_3-H), 12.10 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3375, 2956, 2914, 2816, 1735, 1641, 1596, 1461, 1372, 1278, 1257, 1057, 1031, 804, 764, 750; EI-MS *m/z*: 469[M]$^+$(6), 372(20), 118(100); HRMS (ESI): calcd. for $C_{26}H_{31}NO_7$ [M + H]$^+$ 470.2179, found 470.2179; HPLC (50% acetonitrile in water): t_R = 6.0 min, 98.7%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-methoxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (121). Yield: 59%; yellow solid; m.p.: 145-147 °C; ^{1}H NMR (300



MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.17-2.24 (m, 1H, C_{16}-H), 2.37 (m, 1H, C_{17}-H), 2.57-2.66 (m, 2H, C_{11}-H), 3.09-3.20 (m, 2H, C_7-H, C_{18}-H$_\beta$), 3.30 (s, 3H, C_{20}-H), 3.68-3.70 (m, 1H, C_{19}-H$_{\alpha,}$), 3.89-3.91 (m, 1H, C_{19}-H$_\beta$), 4.37-4.39 (m, 1H, C_{12}-H), 4.51-4.55 (m, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.3 Hz, 1H, C_2-H), 6.55 (d, J = 8.3 Hz, 1H, C_4-H), 7.22 (d, J = 6.8 Hz, 1H, C_8-H), 7.39 (t, J = 8.3Hz, C_3-H), 12.10 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3451, 2991, 2891, 1742, 1643, 1601, 1462, 1376, 1230, 1109, 1031, 803, 747; EI-MS *m/z*: 396[M]$^+$(7), 323(100), 227(87), 69(91); HRMS (ESI): calcd. for $C_{23}H_{24}O_6$ [M + Na]$^+$ 419.1471, found 419.1468; HPLC (75% methanol in water): t_R = 7.7 min, 98.8%.

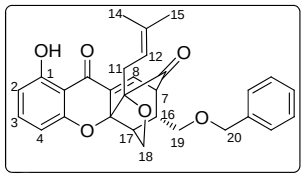
8-Hydroxy-1-(3-methylbut-2-en-yl)-4-(isopropoxymethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (122). Yield: 64%; yellow solid; m.p.: 158-160 °C; ^{1}H NMR



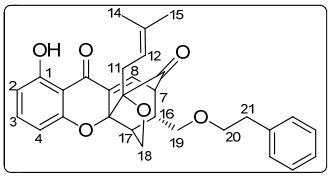
(300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.10-1.12 (m, 6H, C_{21}-H, $C_{21'}$-H), 1.35 (s, 3H, C_{15}-H), 2.17-2.20 (m, 1H, C_{16}-H), 2.30-2.35 (m, 1H, C_{17}-H), 2.57-2.71 (m, 2H, C_{11}-H), 3.12-3.22 (m, 2H, C_7-H, C_{18}-H$_\beta$), 3.45-3.53 (m, 1H, C_{20}-H), 3.72-3.75 (m, 1H, C_{19}-H$_{\alpha,}$), 3.89-3.92 (m, 1H, C_{19}-H$_\beta$), 4.37-4.39 (m, 1H, C_{12}-H), 4.51-4.55 (m, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.3 Hz, 1H, C_2-H), 6.55 (d, J = 8.1

Hz, 1H, C_4-H), 7.22 (d, J = 6.9 Hz, 1H, C_8-H), 7.39 (t, J = 8.3 Hz, 1H, C_3-H), 12.11 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3445, 2976, 2903, 1737, 1642, 1600, 1461, 1380, 1275, 1250, 1229, 1086, 1031, 806, 764, 749; EI-MS *m/z*: 424$[M]^+$(2), 227(18), 137(23), 69(100); HRMS (ESI): calcd. for $C_{25}H_{28}O_6$ $[M + Na]^+$ 447.1414, found 447.1400; HPLC (75% methanol in water): t_R = 13.8 min, 97.9%.

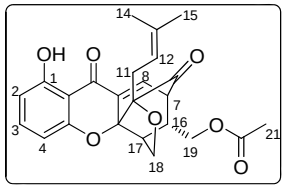
4-Benzyloxymethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (123). Yield: 57%; yellow solid; m.p.: 87-89 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.34 (s, 3H, C_{15}-H), 2.18-2.21 (m, 1H, C_{16}-H), 2.37-2.41 (m, 1H, C_{17}-H), 2.57-2.65 (m, 2H, C_{11}-H), 3.22-3.24 (m, 2H, C_7-H, C_{18}-H$_\beta$), 3.71-3.75 (m, 1H, C_{12}-H), 3.87-3.90 (m, 1H, C_{18}-H$_\alpha$), 4.36-4.53 (m, 4H, C_{19}-H, C_{20}-H), 6.48 (d, J = 8.3 Hz, 1H, C_2-H), 6.55 (d, J = 8.3 Hz, 1H, C_4-H), 7.13 (d, J = 6.7 Hz, 1H, C_8-H), 7.28-7.41 (m, 6H, C_3-H, Ph), 12.09 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3469, 2963, 2914, 1742, 1644, 1600, 1462, 1376, 1272, 1260, 1229, 1106, 1031, 803, 749, 692; EI-MS *m/z*: 472$[M]^+$(2), 323(19), 227(23), 91(100), 69(50); HRMS (ESI): calcd. for $C_{29}H_{28}O_6$ $[M + Na]^+$ 495.1784, found 495.1775; HPLC (85% methanol in water): t_R = 9.9 min, 99.2%.



8-Hydroxy-1-(3-methylbut-2-en-yl)-4-(phenethoxymethyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (124). Yield: 59%; yellow solid; m.p.: 105-107 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.00 (s, 3H, C_{14}-H), 1.34 (s, 3H, C_{15}-H), 2.12-2.17 (m, 1H, C_{16}-H), 2.32 (m, 1H, C_{17}-H), 2.56-2.64 (m, 2H, C_{11}-H), 2.83 (t, J= 6.5 Hz, 2H, C_{21}-H), 3.15-3.17 (m, 2H, C_7-H, C_{18}-H$_\beta$), 3.55-3.63 (m, 3H, C_{19}-H$_\alpha$, C_{20}-H), 3.82-3.85 (m, 1H, C_{19}-H$_\beta$), 4.34-4.36 (m, 1H, C_{12}-H), 4.45-4.49 (m, 1H, C_{18}-H$_\alpha$), 6.48 (d, J = 8.3 Hz, 1H, C_2-H), 6.54 (d, J = 8.3 Hz, 1H, C_4-H), 7.09 (d, J = 6.8 Hz, 1H, C_8-H), 7.16-7.29 (m, 5H , Ph), 7.39 (t, J = 8.3 Hz, 1H, C_3-H), 12.12 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3475, 2908, 1742, 1644, 1600, 1462, 1376, 1275, 1229, 1108, 1031, 803, 750, 704; EI-MS *m/z*: 486$[M]^+$(3), 323(53), 227(32), 105(100), 69(32); HRMS (ESI): calcd. for $C_{30}H_{30}O_6$ $[M + Na]^+$ 509.1940, found 509.1933; HPLC (85% methanol in water): t_R = 11.9 min, 99.0%.



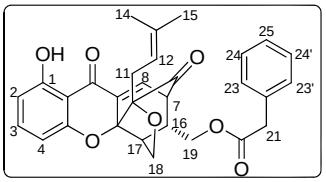
4-(Acetoxymethyl)-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (125). Yield: 59%; yellow solid; m.p.: 176-178 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.07 (s, 3H, C_{20}-H), 2.24-2.26 (m, 1H, C_{16}-H), 2.41-2.43 (m, 1H, C_{17}-H), 2.63-2.66 (m, 2H, C_{11}-H), 3.65-3.68 (m, 1H, C_7-H), 3.76-3.83 (m, 1H, C_{18}-H$_\beta$), 3.91-3.97 (m, 2H, C_{19}-H), 4.36-4.38 (m, 1H, C_{12}-H), 4.52-4.57 (m, 1H, C_{18}-H$_\alpha$), 6.49 (d, J = 8.3 Hz, 1H, C_2-H), 6.56 (d, J = 8.3 Hz, 1H, C_4-H), 7.23 (d, J = 6.8 Hz, 1H, C_8-H), 7.40 (t, J = 8.3 Hz, C_3-H), 12.04 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3463, 2962, 2903, 1741, 1644, 1602, 1462, 1374, 1228, 1042, 803, 768, 749; EI-MS *m/z*: 424$[M]^+$(10), 323(51), 227(74), 69(100); HRMS (ESI): calcd. for $C_{24}H_{24}O_7$ $[M +Na]^+$ 447.1420, found 447.1405; HPLC (75% methanol in water): t_R = 6.4 min, 99.1%.



4-Benzoyloxymethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (126). Yield: 67%; yellow solid; m.p.: 168-170 °C; ^{1}H-NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.17-2.19 (m, 1H, C_{16}-H), 2.34-2.36 (m, 1H, C_{17}-H), 2.61-2.68 (m, 4H, C_{11}-H, C_{16}-H, C_{17}-H), 3.76-3.77 (m, 1H, C_7-H), 3.97 (d, J = 8.0 Hz, 1H, C_{18}-H$_\beta$), 4.04-4.11 (m, 1H, C_{19}-H$_\alpha$), 4..17-4.23 (1H, C_{19}-H$_\beta$), 4.37-4.39 (m, 1H, C_{12}-H), 4.58 (dd, J_1 = 3.9 Hz, J_2=7.8 Hz, 1H, C_{18}-H$_\alpha$), 6.50 (d, J = 8.3 Hz, 1H, C_2-H), 6.56 (d, J = 8.3Hz, 1H, C_4-H), 7.29 (d, J = 6.9 Hz, 1H, C_8-H), 7.37-7.48 (m, 3H, C_3-H, C_{23}-H, $C_{23'}$-H), 7.57-7.62 (m, 1H, C_{24}-H), 7.99 (d, J = 7.5 Hz, 2H, C_{22}-H, $C_{22'}$-H), 12.05 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3428, 2968, 2897, 1742, 1722, 1644, 1601, 1462, 1369, 1275, 1266, 1225, 1104, 1025, 804, 750, 712; EI-MS *m/z*: 486$[M]^+$(2), 336(36), 240(32), 105(100), 77(54), 69(44); HRMS

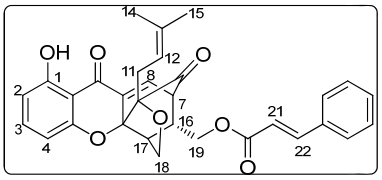
(ESI): calcd. for $C_{29}H_{26}O_7$ $[M + H]^+$ 487.1757, found 487.1737; HPLC (85% methanol in water): t_R = 8.6 min, 98.4%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-phenylacetyloxymethyl-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (127). Yield: 66%; yellow solid; m.p.: 125-127 °C; ^{1}H NMR



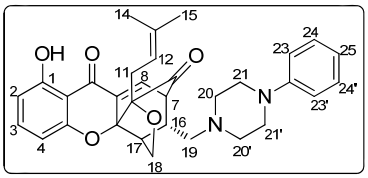
(300 MHz, $CDCl_3$): δ 1.01 (s, 3H, C_{14}-H), 1.34 (s, 3H, C_{15}-H), 2.15-2.17 (m, 1H, C_{16}-H), 2.36-2.40 (m, 1H, C_{17}-H), 2,61-2.64 (m, 2H, C_{11}-H), 3.51-3.55 (m, 1H, C_7-H), 3,62 (s, 2H, C_{20}-H), 3.77-3.91 (m, 3H, C_{18}-H_β, C_{19}-H), 4.43-4.45 (m, 1H, C_{12}-H), 4.46-4.47 (m, 1H, C_{18}-H_α), 6.48 (d, J = 8.3 Hz, 1H, C_2-H), 6.56 (d, J = 8.3 Hz, 1H, C_4-H), 7.15 (d, J = 6.8 Hz, 1H, C_8-H), 7.24-7.42 (m, 6H, C_3-H, Ph), 12.03 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 2973, 2890, 1740, 1644, 1600, 1462, 1372, 1259, 1230, 1154, 1022, 803, 711, 742; EI-MS *m/z*: 500$[M]^+$(2), 336(30), 239(26), 227(29), 91(100), 69(39); HRMS (ESI): calcd. for $C_{30}H_{28}O_7$ $[M + Na]^+$ 523.1733, found 523.1713; HPLC (85% methanol in water): t_R = 7.4 min, 98.7%.

4-Cinnamyloxymethyl-8-hydroxy-1-(3-methylbut-2-en-yl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (128). Yield: 63%; yellow solid; m.p.: 161-163 °C; ^{1}H NMR



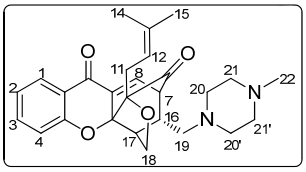
(300 MHz, $CDCl_3$): δ 1.03 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.17-2.18 (m, 1H, C_{16}-H), 2.30-2.32 (m, 1H, C_{17}-H), 2,60-2.68 (m, 2H, C_{11}-H), 3.71-3.75 (m, 1H, C_7-H), 3.93-3.99 (m, 2H, C_{19}-H), 4.05-4.11 (m, 1H, C_{18}-H_β), 4.37-4.39 (m, 1H, C_{12}-H), 4.55-4.59 (m, 1H, C_{18}-H_α), 6.41 (d, J = 16Hz, 1H, C_{22}-H), 6.50 (d, J = 8.2 Hz, 1H, C_2-H), 6.56 (d, J = 8.1 Hz, 1H, C_4-H), 7.28 (d, J = 7.8 Hz, 1H, C_8-H), 7.37-7.54 (m, 6H , C_3-H, Ph), 7.67 (d, J = 16.0 Hz, 1H, C_{21}-H), 12.06 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3463, 2962, 2909, 1743, 1714, 1644, 1601, 1462, 1376, 1275, 1260, 1162, 1042, 803, 765, 749; HPLC: 97.9%; t_R 10.83 min; EI-MS *m/z*: 512$[M]^+$(2), 484(12), 336(47), 240(36), 131(100), 103(58), 69(41); HRMS (ESI): calcd. for $C_{31}H_{28}O_7$ $[M + H]^+$ 513.1913, found 513.1897; HPLC (85% methanol in water): t_R = 10.8 min, 98.9%.

8-Hydroxy-1-(3-methylbut-2-en-yl)-4-((4-phenylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (129). Yield: 69%; yellow solid; m.p.: 104-106 °C;



^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 (s, 3H, C_{14}-H), 1.35 (s, 3H, C_{15}-H), 2.12-2.33 (m, 4H, C_{16}-H, C_{17}-H, C_{19}-H), 2.50-2.66 (m, 6H, C_{11}-H, C_{21}-H, $C_{21'}$-H), 3.15-3.18 (m, 4H, C_{20}-H, $C_{20'}$-H), 3.69-3.73 (m, 1H, C_7-H), 3.87 (d, J = 7.8 Hz, 1H, C_{18}-H_β), 4.37-4.40 (m, 1H, C_{12}-H), 4.54 (dd, J_1 = 3.6 Hz, J_2 = 8.1 Hz, 1H, C_{18}-H_α), 6.50 (d, J = 8.4 Hz, 1H, C_2-H), 6.55 (d, J = 8.4 Hz, 1H, C_4-H), 6.84-6.93(m, 3H, C_{24}-H, $C_{24'}$-H, C_{25}-H), 7.24-7.29 (m, 3H, C_8-H, C_{23}-H, $C_{23'}$-H), 7.39 (t, J = 8.4 Hz, 1H, C_3-H), 12.11 (s, 1H, C_1-OH); IR (cm^{-1}, KBr film): 3457, 3069, 2962, 2890, 2814, 1740, 1644, 1600, 1493, 1463, 1372, 1260, 1231, 1060, 1025, 803, 758, 695; EI-MS *m/z*: 526$[M]^+$(21), 429(100), 175(45), 160(52), 70(53); HRMS (ESI): calcd. for $C_{32}H_{34}N_2O_5$ $[M + H]^+$ 527.2546, found 527.2547; HPLC (75% methanol in water): t_R = 28.7 min, 98.9%.

1-(3-Methylbut-2-en-yl)-4-((4-methylpiperazin-1-yl)methyl)-3,3a,4,5-tetrahydro-1,5-methano-1*H*,7*H*-furo[3,4-*d*]xanthene-7,13-dione (130). Yield: 56%; yellow solid; m.p.: 154-156 °C; ^{1}H NMR



(300 MHz, $CDCl_3$): δ 0.93 (s, 3H, C_{14}-H), 1.28 (s, 3H, C_{15}-H), 2.10-2.20 (m, 6H, C_{16}-H, $C_{17'}$-H, C_{21}-H, $C_{21'}$-H), 2.27 (s, 3H, C_{22}-H), 2.30-2.48 (m, 6H, C_{19}-H, C_{20}-H, $C_{20'}$-H), 3.62-3.64 (m, 1H, C_7-H), 3.85 (d, J = 7.8 Hz, 1 H, C_{18}-H_β), 4.39-4.40 (m, 1H, C_{12}-H), 4.54 (dd, J_1 = 3.2 Hz, J_2 = 7.6 Hz, 1 H, C_{18}-H_α), 7.01-7.09 (m, 2H, C_2-H, C_4-H), 7.16 (d, J = 6.8 Hz, 1H, C_8), 7.51 (t, J = 7.2 Hz, 1H, C_3-H), 7.96 (d, J = 7.8 Hz, 1H, C_1-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.9, 25.2, 27.8, 38.9, 46.0, 47.3, 49.8, 53.1, 55.0, 60.1, 74.5, 82.5, 88.4, 118.1, 118.3, 119.3, 121.9, 127.1, 130.0, 133.9, 135.4, 136.2, 159.7, 176.1, 200.3; IR (cm^{-1}, KBr film): 2932, 2798, 1739, 1672, 1646, 1614, 1462, 1310, 1262, 1126, 1068,752; EI-MS *m/z*: 448$[M]^+$(14), 351(100), 113(67),

70(93); HRMS (ESI): calcd. for $C_{27}H_{33}N_2O_4$ $[M + H]^+$ 449.2440, found 449.2435; ; HPLC (60% acetonitrile in water): t_R = 8.9 min, 99.1%.

References

[S1]. Li, N.-G.; Wang, J.-X.; Liu, X.-R.; Lin, C.-J.; You, Q.-D.; Guo, Q.-L. A novel and efficient route to the construction of the 4-oxa-tricyclo[4.3.1.0]decan-2-one scaffold. *Tetrahedron Lett.* **2007**, *48*, 6586-6589.

[S2]. Sun, H.; Chen, F.; Wang, X.; Liu, Z.; Yang, Q.; Zhang, X.; Zhu, J.; Qiang, L.; Guo, Q.; You, Q. Studies on chemical modification and biology of a natural product, Gambogic acid (IV): Identification of IκB Kinase-beta (IKKβ) as the molecular target and determination of the pharmacophoric scaffold. *Eur. J. Med. Chem.* **2012**, *51*, 110-123.

[S3]. Wang, X.; Lu, N.; Yang, Q.; Gong, D.; Lin, C.; Zhang, S.; Xi, M.; Gao, Y.; Wei, L.; Guo, Q.; You, Q. Studies on chemical modification and biology of a natural product, gambogic acid (III): Determination of the essential pharmacophore for biological activity. *Eur. J. Med. Chem.* **2011**, *46*, 1280-1290.

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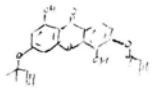

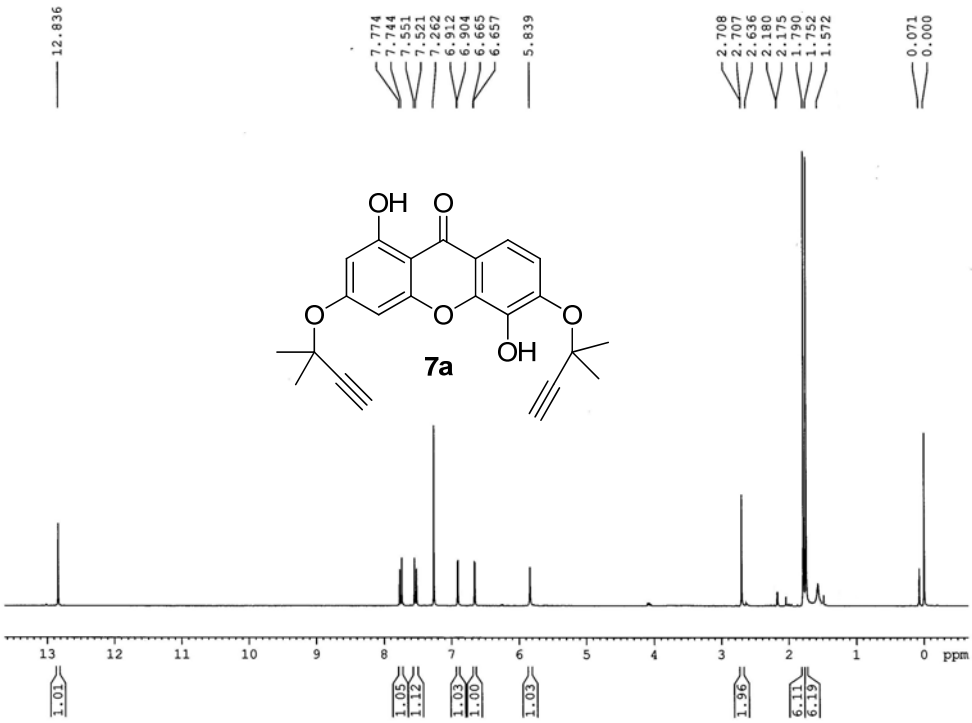


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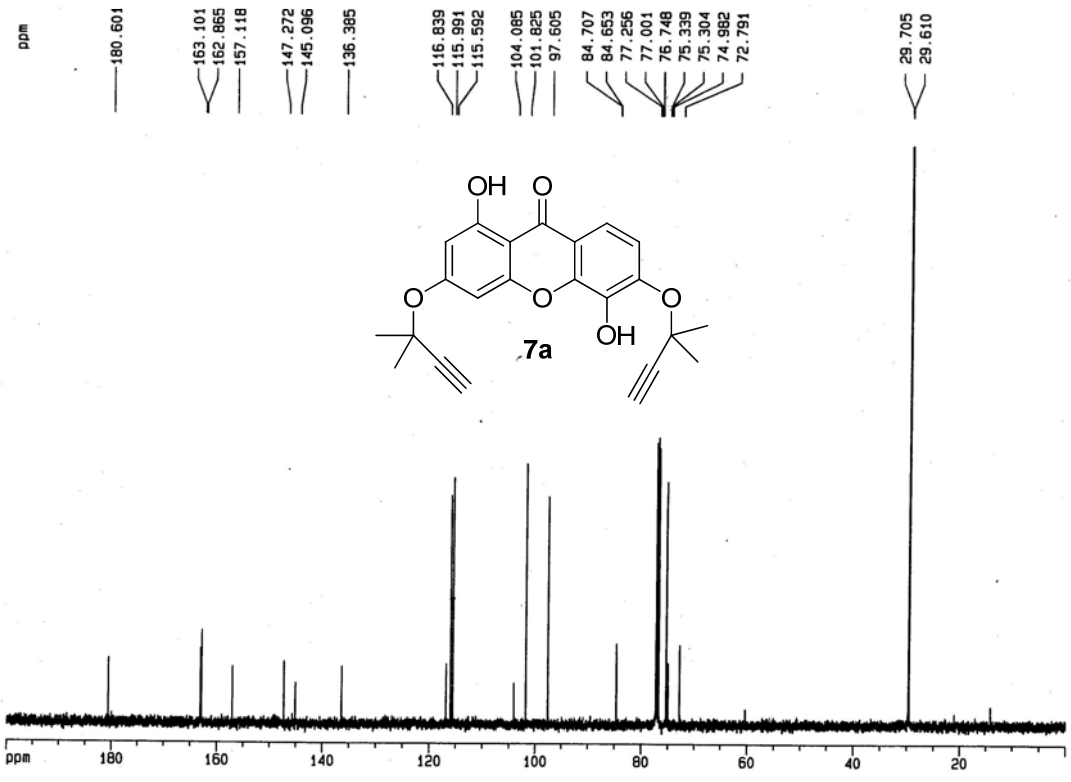


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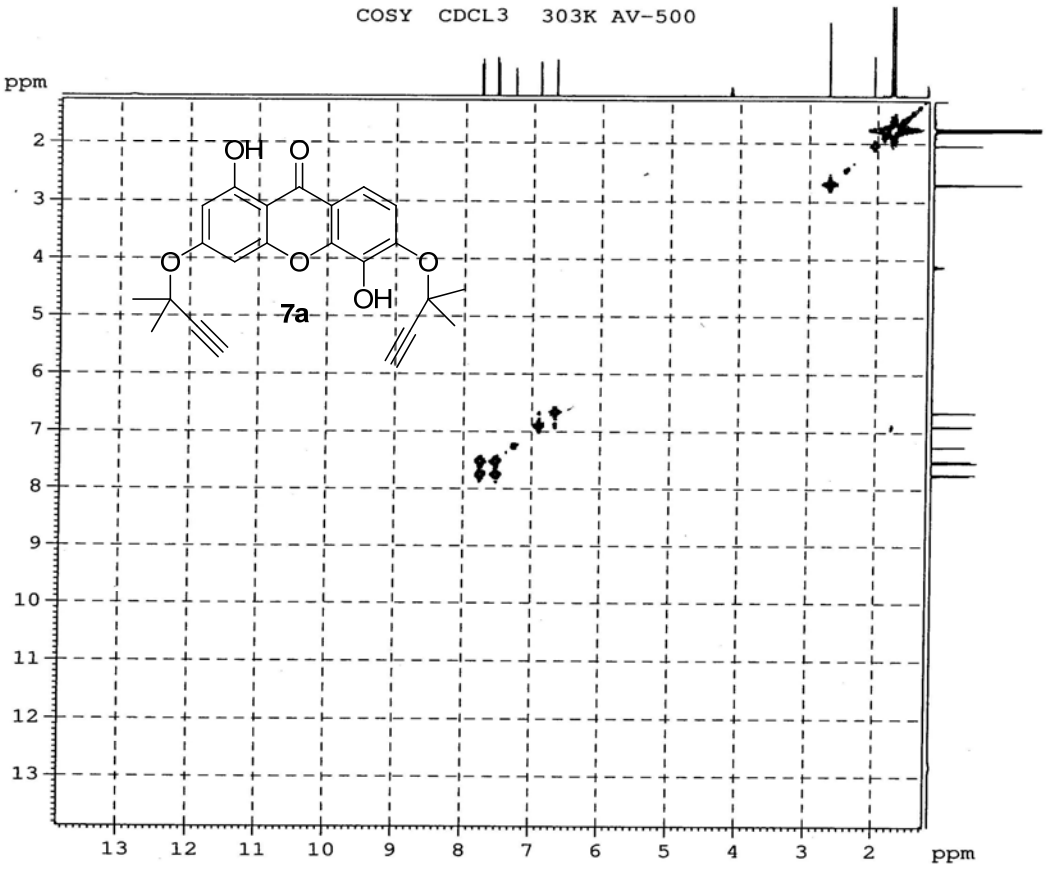
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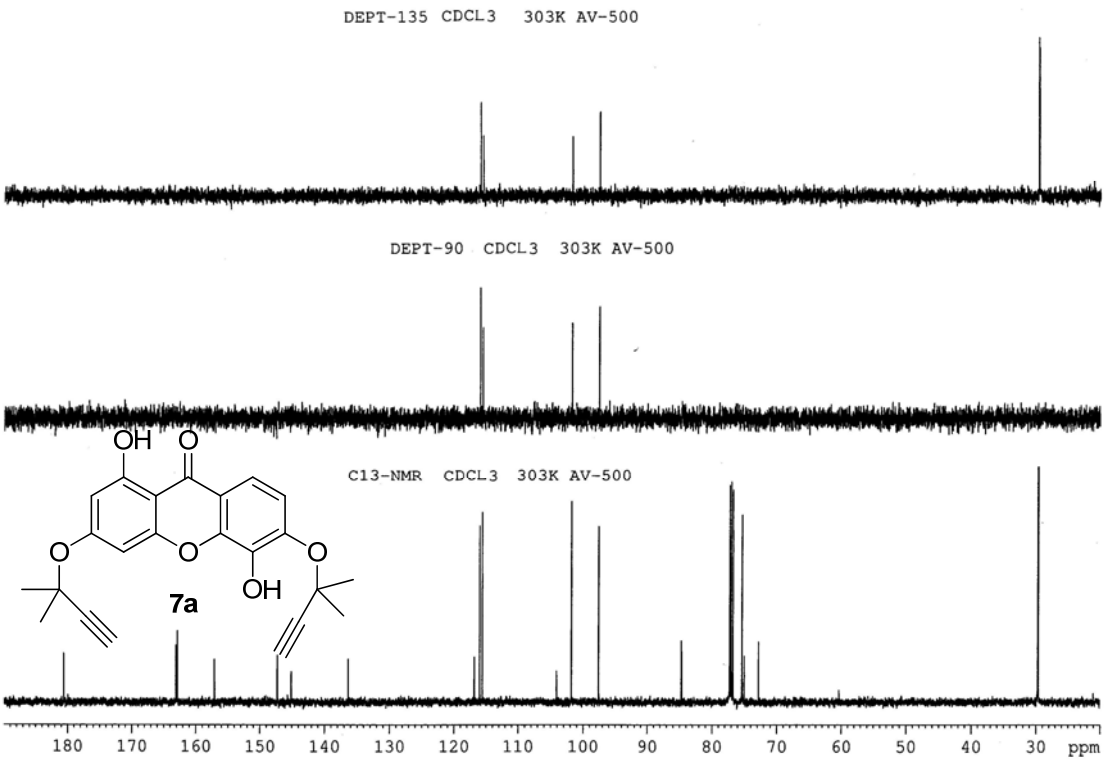
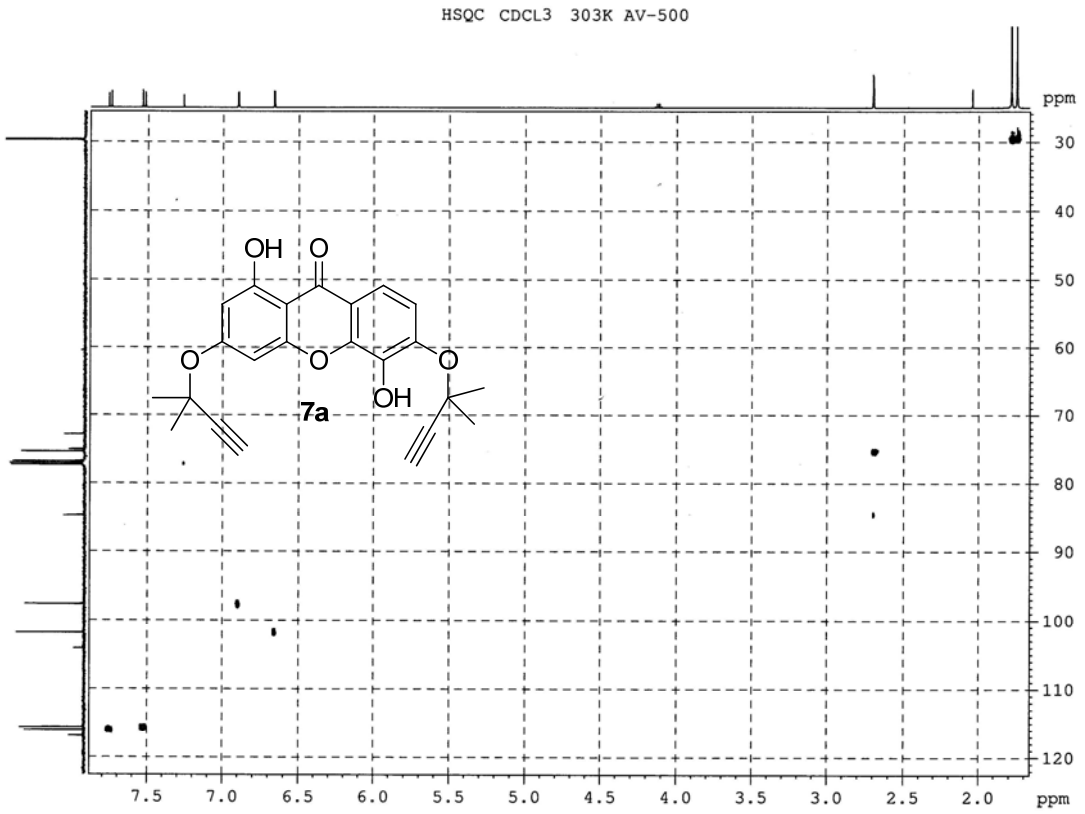
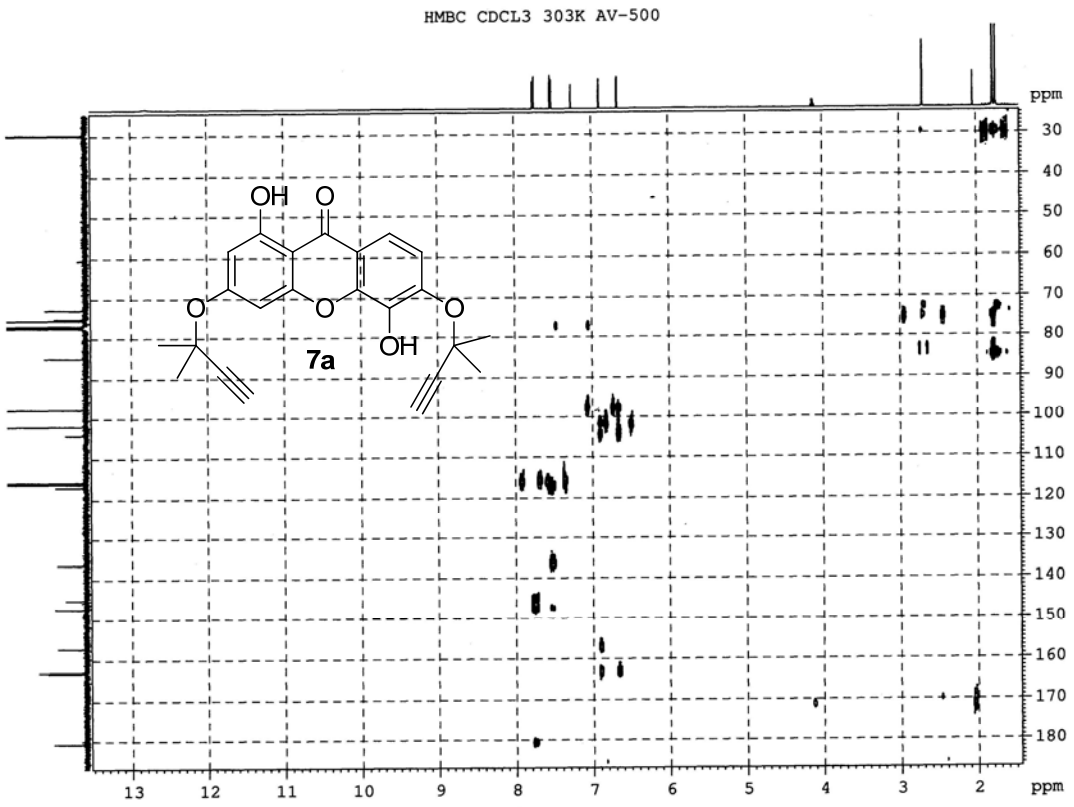
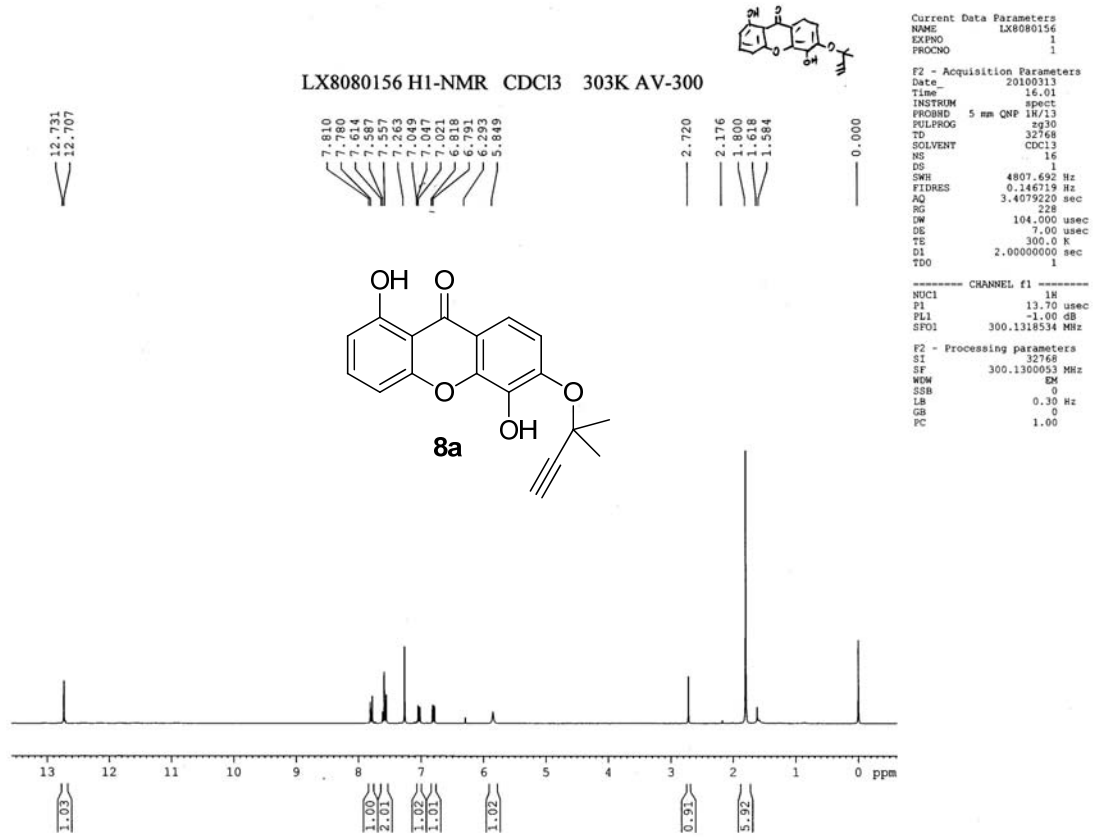
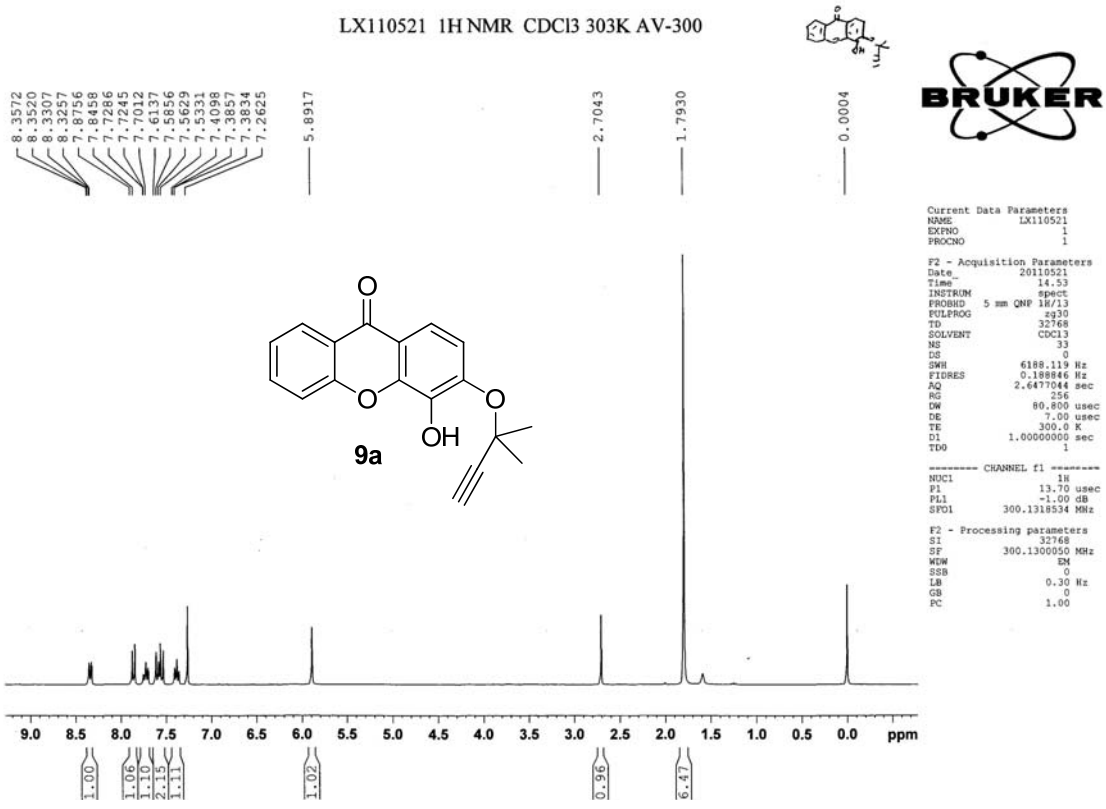
7a
13 12 11 10 9 8 7 6 5 4 3 2 ppm

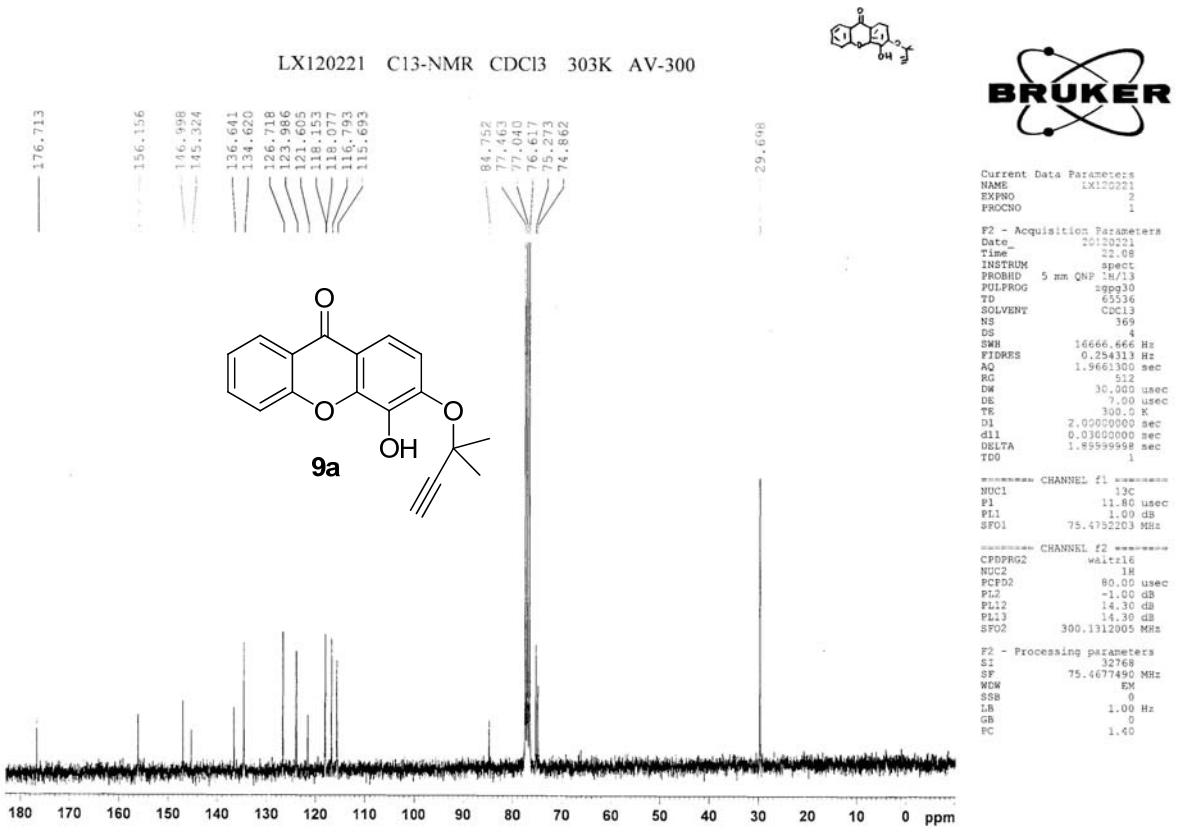

DEPT-135 CDCL3 303K AV-500
DEPT-90 CDCL3 303K AV-500
C13-NMR CDCL3 303K AV-500
OH
O
O
O
O
OH
7a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

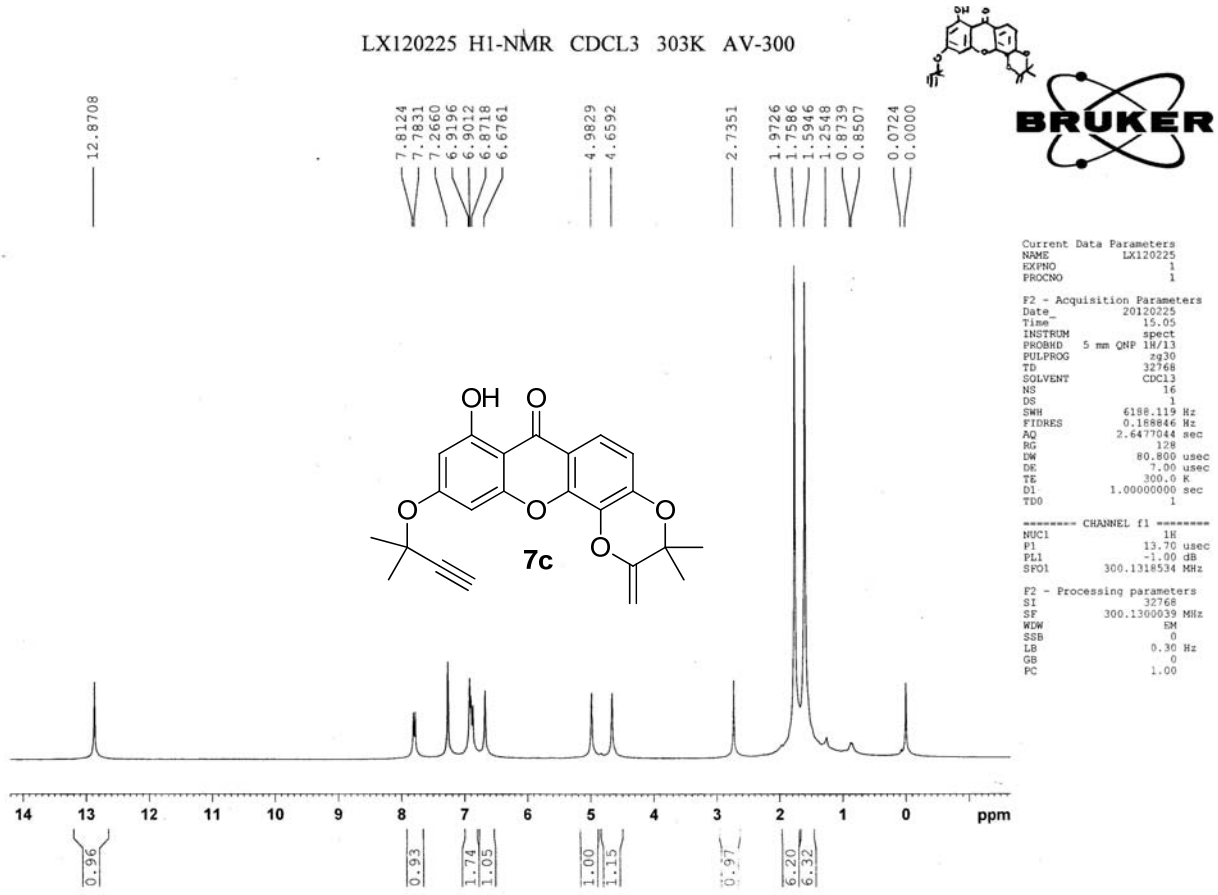

HSQC CDCL3 303K AV-500
OH
O
O
O
O
OH
7a
ppm
30
40
50
60
70
80
90
100
110
120
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm


HMBC CDCL3 303K AV-500
OH
O
O
O
O
OH
7a
ppm
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
13
12
11
10
9
8
7
6
5
4
3
2
ppm

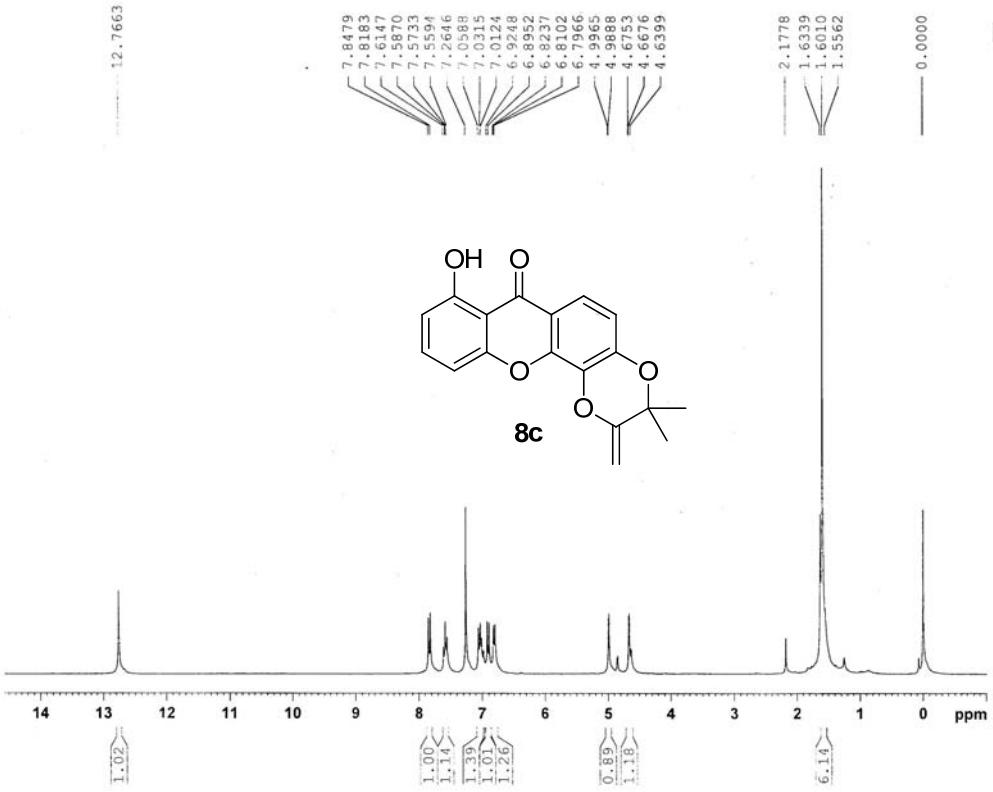

LX8080156 H1-NMR CDCl3 303K AV-300
12.731
12.707
7.810
7.780
7.614
7.587
7.557
7.263
7.049
7.047
7.021
6.818
6.791
6.293
5.849
2.720
2.176
1.800
1.618
1.584
0.000
OH
O
O
OH
8a
Current Data Parameters
NAME LX8080156
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100313
Time 16.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 228
DW 104.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300053 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.03
1.00
2.01
1.02
1.01
1.02
0.91
5.92


LX110521 1H NMR CDCl3 303K AV-300
8.3572
8.3520
8.3307
8.3257
7.8756
7.8458
7.7286
7.7245
7.7012
7.6137
7.5856
7.5629
7.5331
7.4098
7.3857
7.3834
7.2625
5.8917
2.7043
1.7930
0.0004
O
O
O
OH
9a
BRUKER
Current Data Parameters
NAME LX110521
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110521
Time 14.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 33
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 256
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.06
1.10
2.15
1.11
1.02
0.96
6.47


LX120221 C13-NMR CDCl3 303K AV-300
176.713
156.156
146.998
145.324
136.641
134.620
126.718
123.986
121.605
118.153
118.077
116.793
115.693
84.752
77.463
77.040
76.617
75.273
74.862
29.698
9a
Current Data Parameters
NAME LX120221
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120221
Time 22.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 369
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 512
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.85999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


LX120225 H1-NMR CDCL3 303K AV-300
12.8708
7.8124
7.7831
7.2660
6.9196
6.9012
6.8718
6.6761
4.9829
4.6592
2.7351
1.9726
1.7586
1.5946
1.2548
0.8739
0.8507
0.0724
0.0000
7c
Current Data Parameters
NAME LX120225
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120225
Time 15.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 128
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
0.93
1.74
1.05
1.00
1.15
0.97
6.20
6.32

ZXJ120218-2 H1-NMR CDCL3 303K AV-300



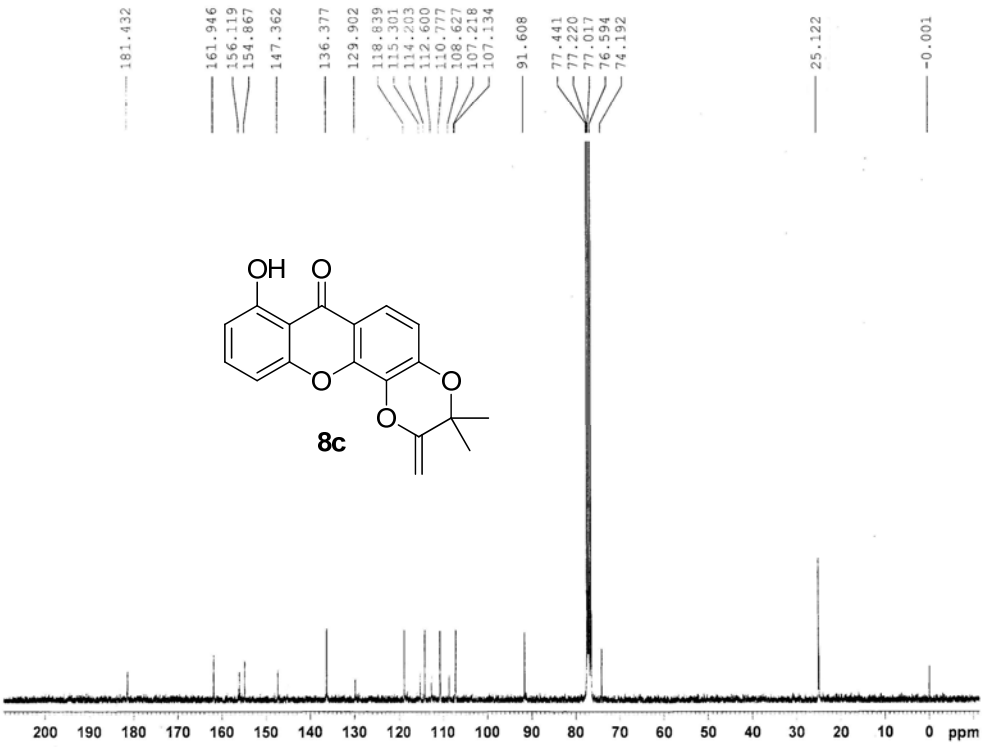
Current Data Parameters
NAME ZXJ120218-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120218
Time 15.33
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 161
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ZXJ120218-2 C13-NMR CDCl3 303K AV-300



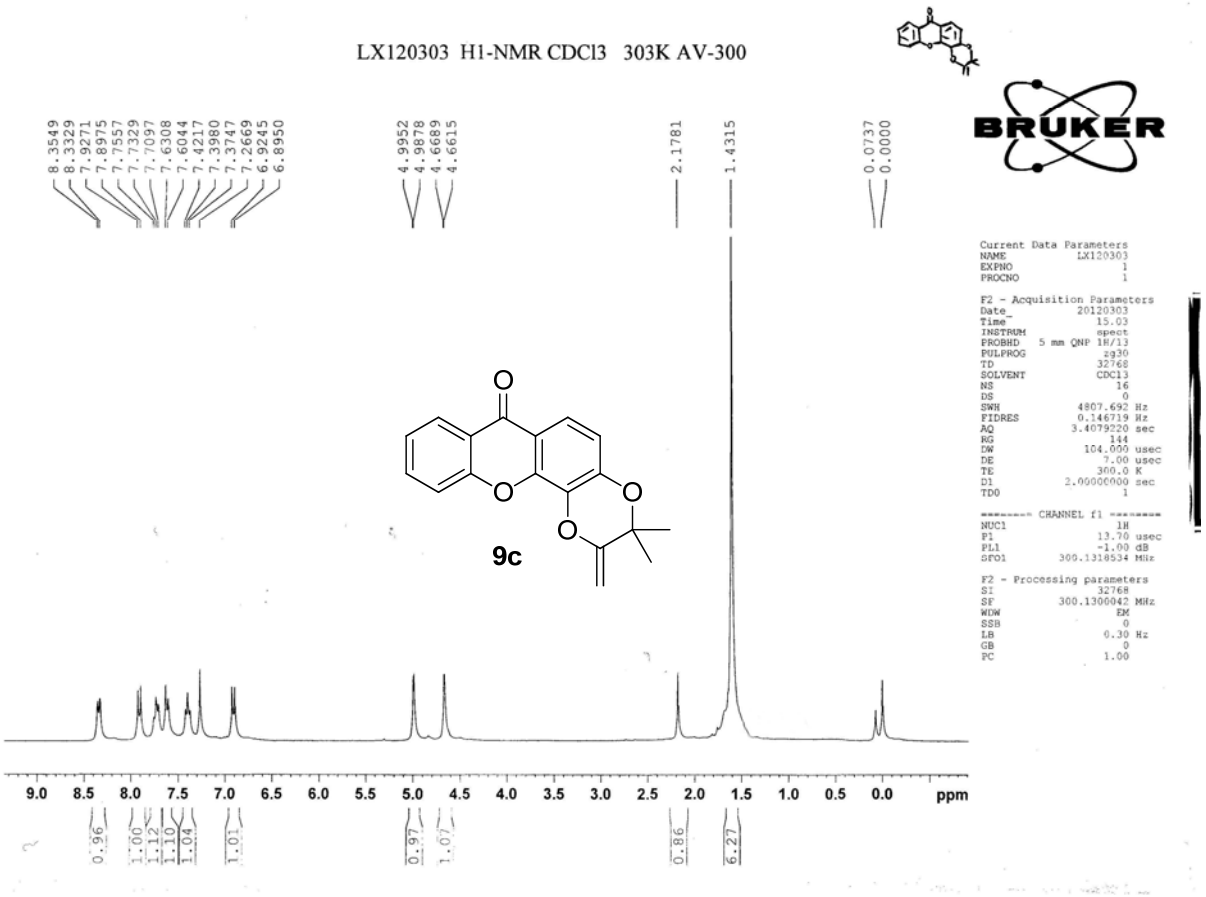
Current Data Parameters
NAME ZXJ120218-2
EXPNO 2
PROCNO 1

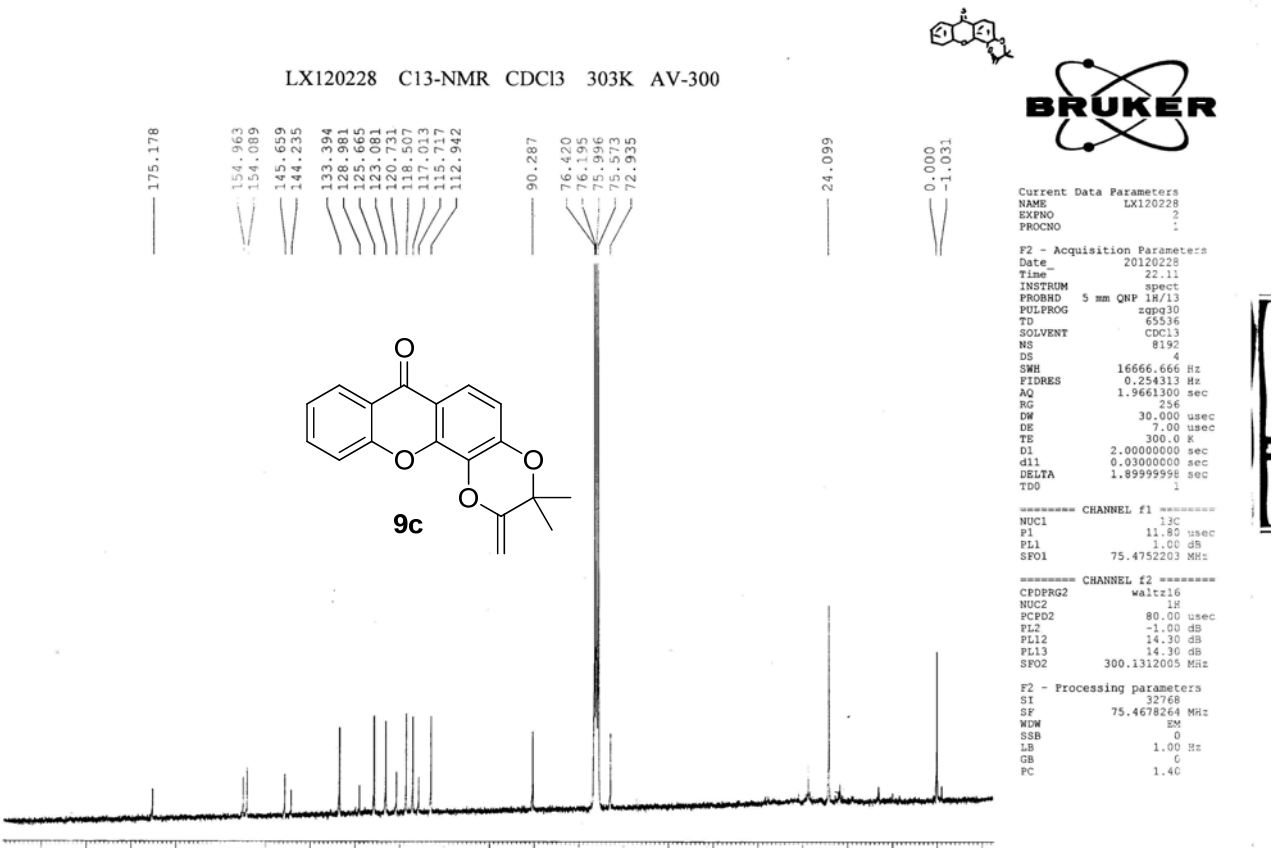
F2 - Acquisition Parameters
Date_ 20120218
Time 16.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3547
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 256
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

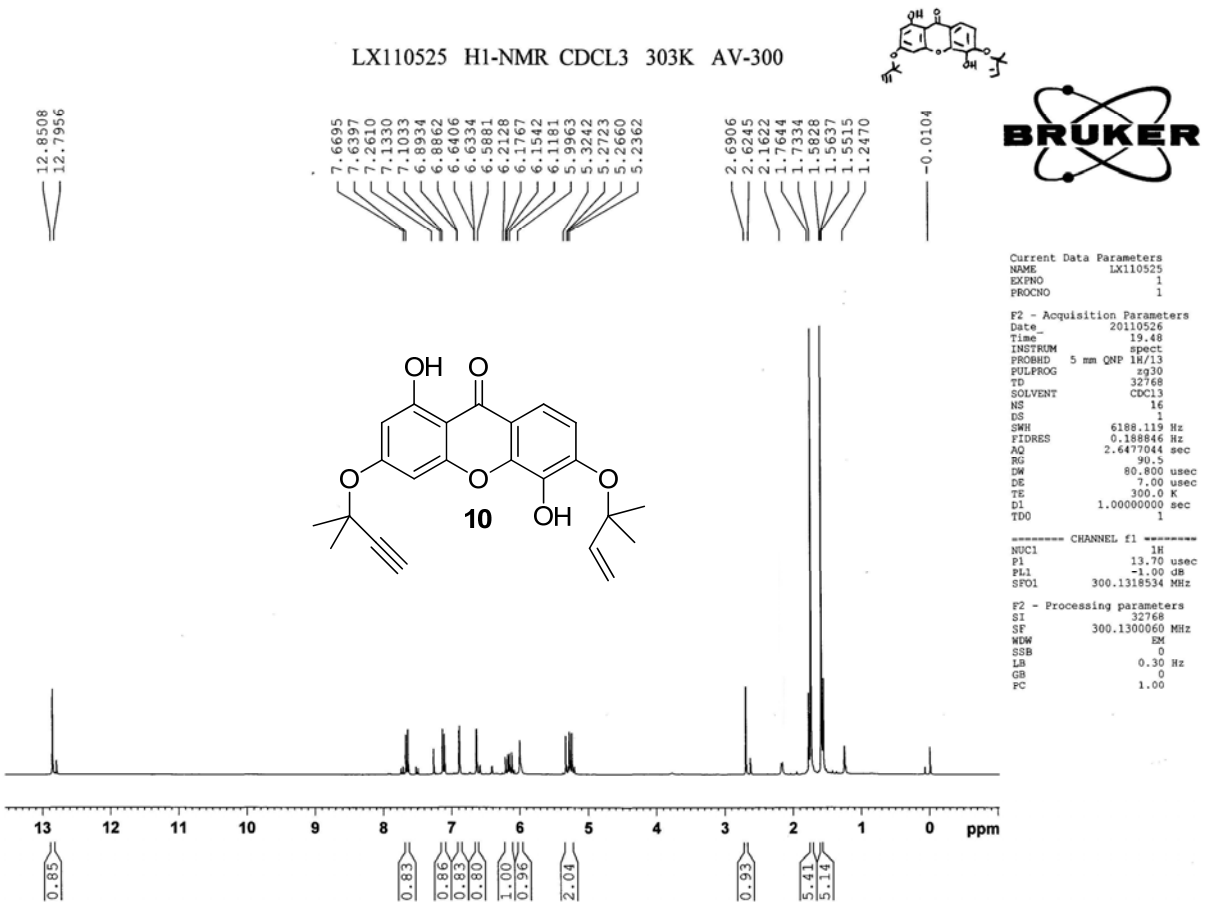
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz

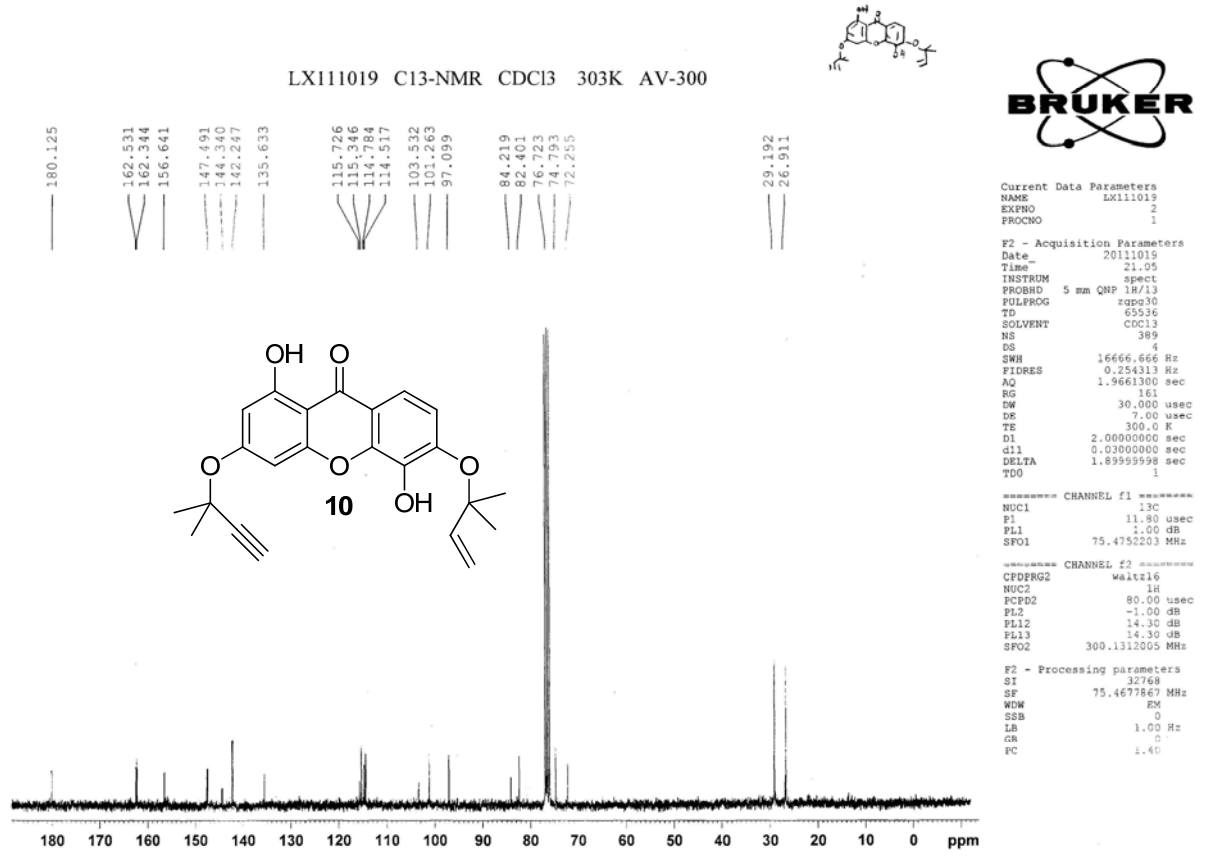
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677493 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

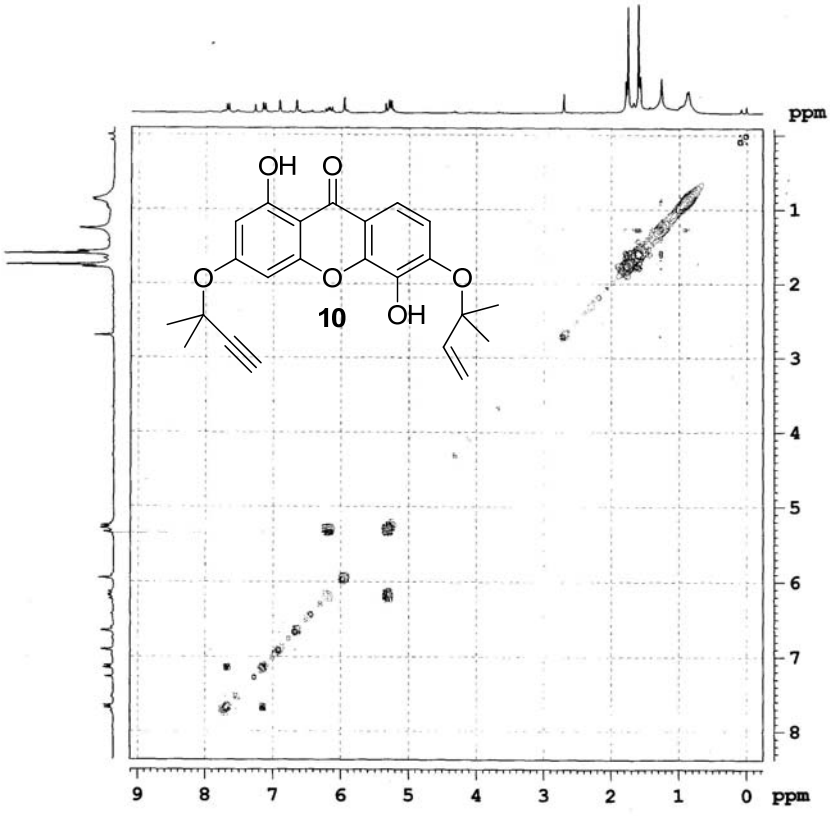

LX120303 H1-NMR CDCl3 303K AV-300
BRUKER
8.3549
8.3329
7.9271
7.8975
7.7557
7.7329
7.7097
7.6308
7.6044
7.4217
7.3980
7.3747
7.2669
6.9245
6.8950
4.9952
4.9878
4.6689
4.6615
2.1781
1.4315
0.0737
0.0000
9c
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.96
1.00
1.12
1.10
1.04
1.01
0.97
1.07
0.86
6.27
Current Data Parameters
NAME LX120303
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120303
Time 15.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 144
DW 104.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


LX120228 C13-NMR CDCl3 303K AV-300
BRUKER
175.178
154.963
154.089
145.659
144.235
133.394
128.981
125.665
123.081
120.731
118.507
117.013
115.717
112.942
90.287
76.420
76.195
75.996
75.573
72.935
24.099
0.000
-1.031
9c
Current Data Parameters
NAME LX120228
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120228
Time 22.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8192
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 256
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4678264 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

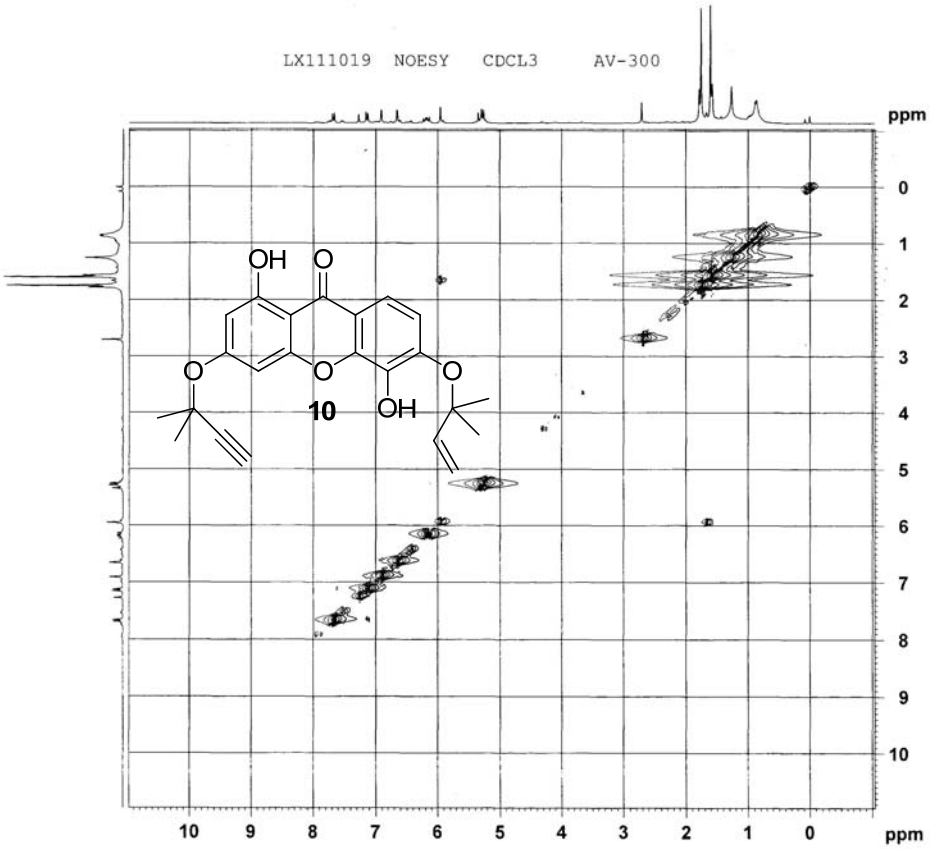

LX110525 H1-NMR CDCL3 303K AV-300
12.8508
12.7956
7.6695
7.6397
7.2610
7.1330
7.1033
6.8934
6.8862
6.6406
6.6334
6.5881
6.2128
6.1767
6.1542
6.1181
5.9963
5.3242
5.2723
5.2660
5.2362
2.6906
2.6245
2.1622
1.7644
1.7334
1.5828
1.5637
1.5515
1.2470
-0.0104
OH
O
O
O
O
OH
10
Current Data Parameters
NAME LX110525
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110526
Time 19.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 90.5
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.85
0.83
0.86
0.83
0.80
1.00
0.96
2.04
0.93
5.41
5.14


LX111019 C13-NMR CDCl3 303K AV-300
180.125
162.531
162.344
156.641
147.491
144.340
142.247
135.633
115.726
115.346
114.784
114.517
103.532
101.263
97.099
84.219
82.401
76.723
74.793
72.255
29.192
26.911
OH
O
O
O
O
OH
10
Current Data Parameters
NAME LX111019
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111019
Time 21.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 389
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 161
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

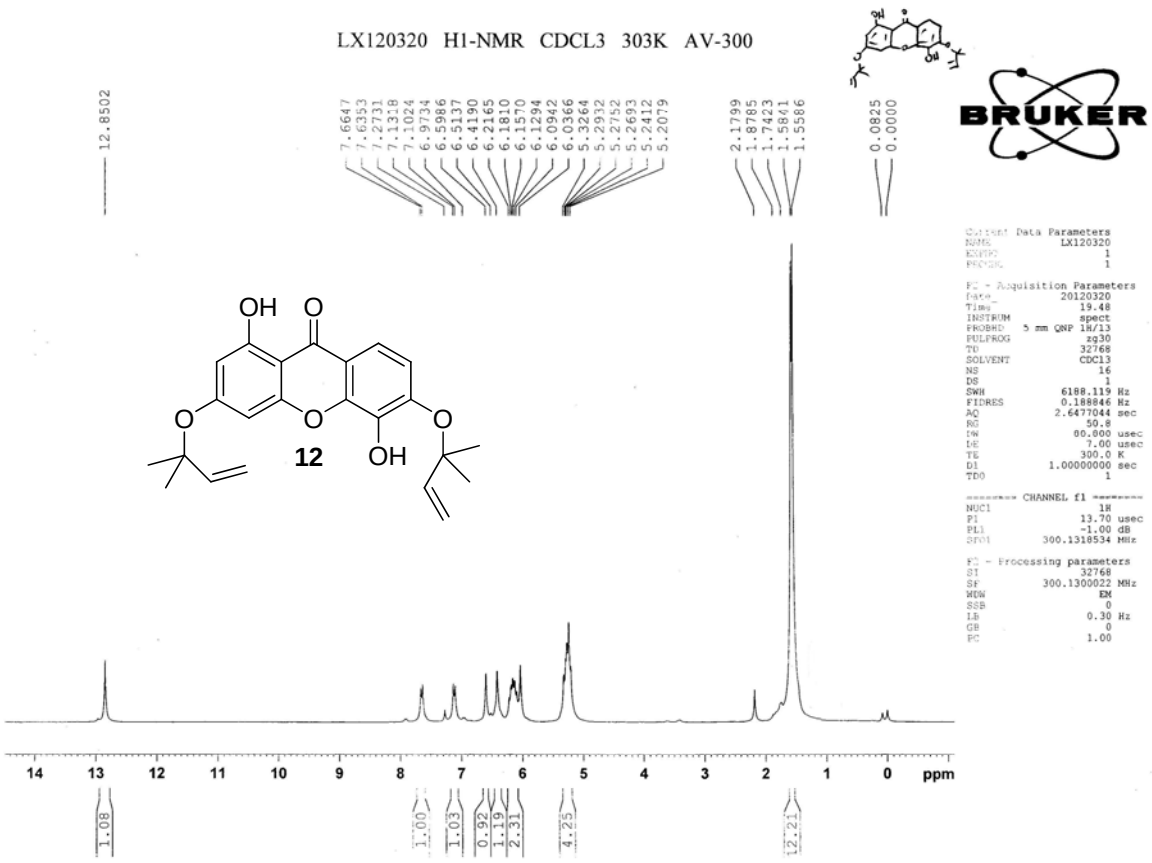
LX111019 H-H COSY CDCl3 303K AV-300

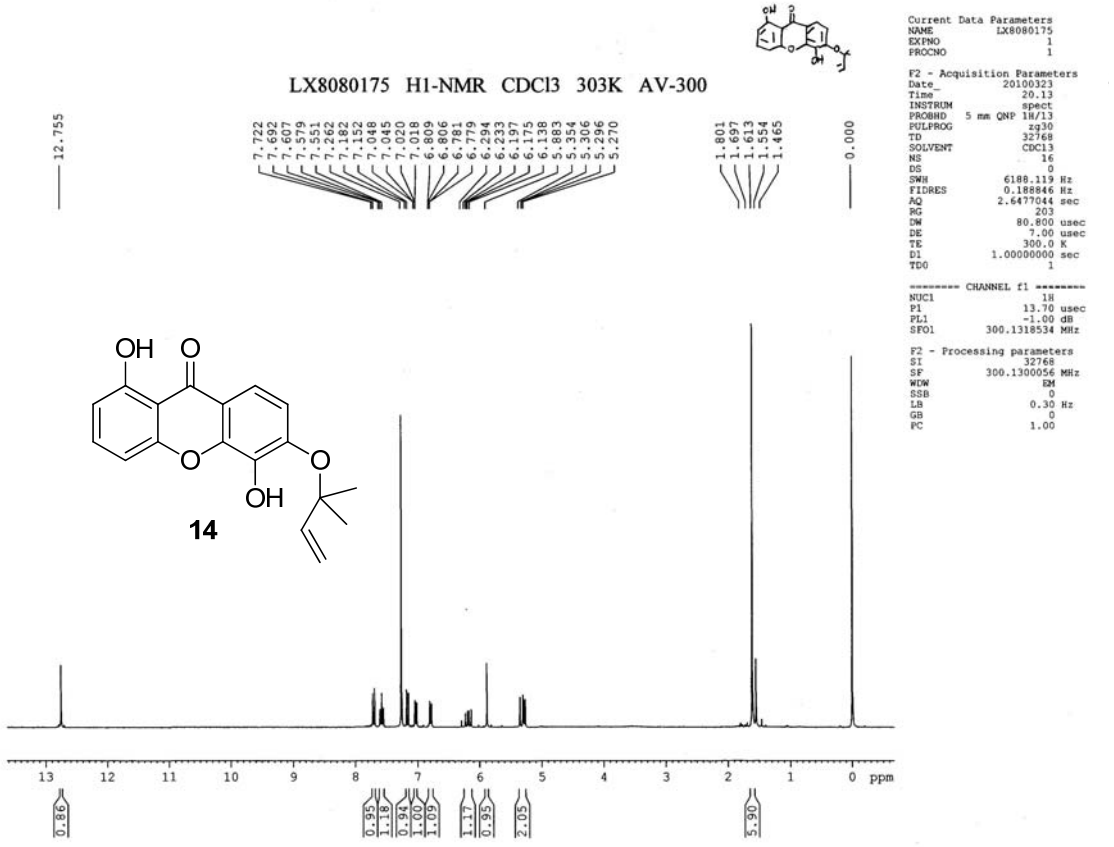


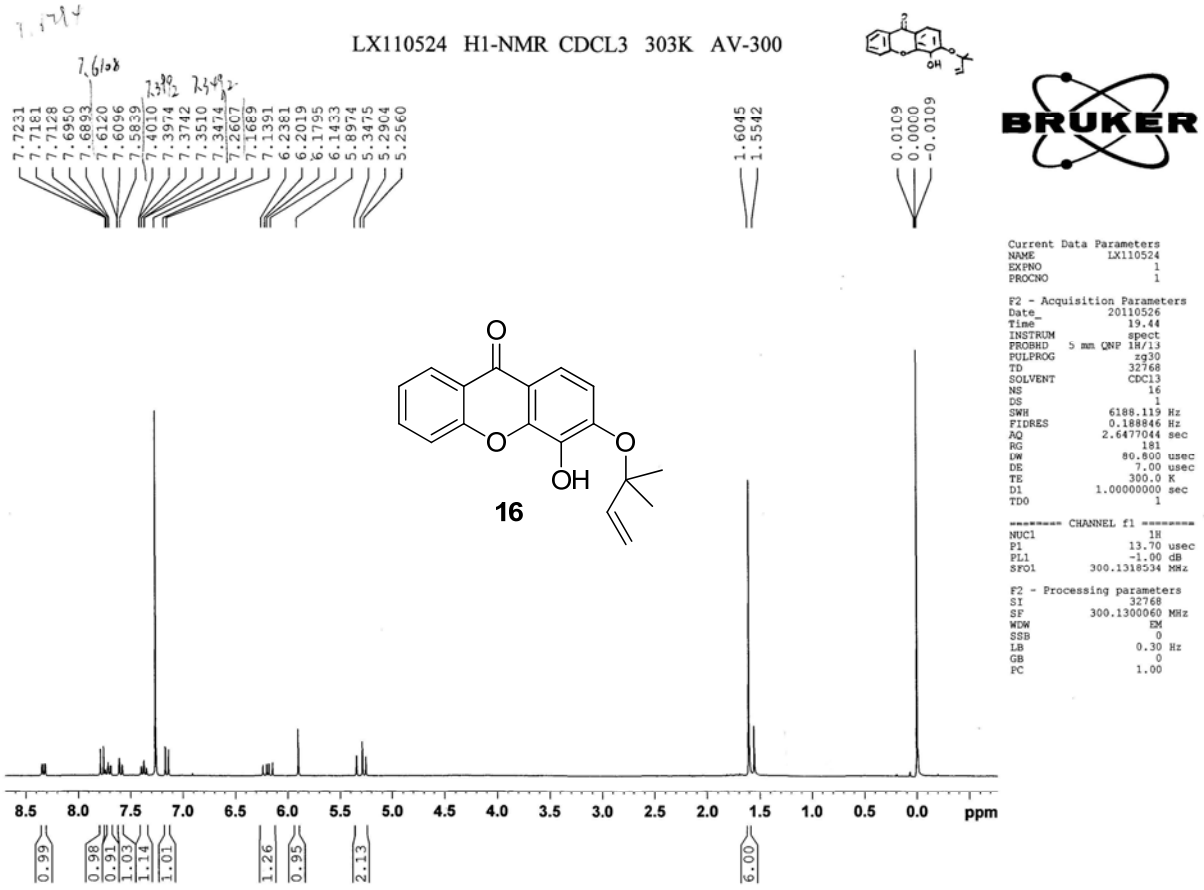

```
Current Data Parameters
NAME                 LX111019
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20111019
Time                   22.45
INSTRUM                spect
PROBHD    5 mm QNP 1H/13
PULPROG            cosygpqf
TD                      2048
SOLVENT                CDCl3
NS                          2
DS                          8
SWH                4045.307 Hz
FIDRES             1.975248 Hz
AQ                 0.2531828 sec
RG                        114
DW                   123.600 usec
DE                     7.00 usec
TE                    300.0 K
d0                 0.00000300 sec
D1                 1.48689198 sec
d13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00024720 sec

======== CHANNEL f1 ========
NUC1                       1H
P0                     13.70 usec
P1                     13.70 usec
PL1                    -1.00 dB
SFO1           300.1319508 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPZ1                  10.00 %
GPZ2                  10.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
ND0                         1
TD                        106
SFO1               300.132 MHz
FIDRES          38.163277 Hz
SW                    13.478 ppm
FnMODE                     QF

F2 - Processing parameters
SI                       1024
SF             300.1300000 MHz
WDW                      SINE
SSB                         0
LB                     0.00 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2                        QF
SF             300.1300000 MHz
WDW                      SINE
SSB                         0
LB                     0.00 Hz
GB                          0
```

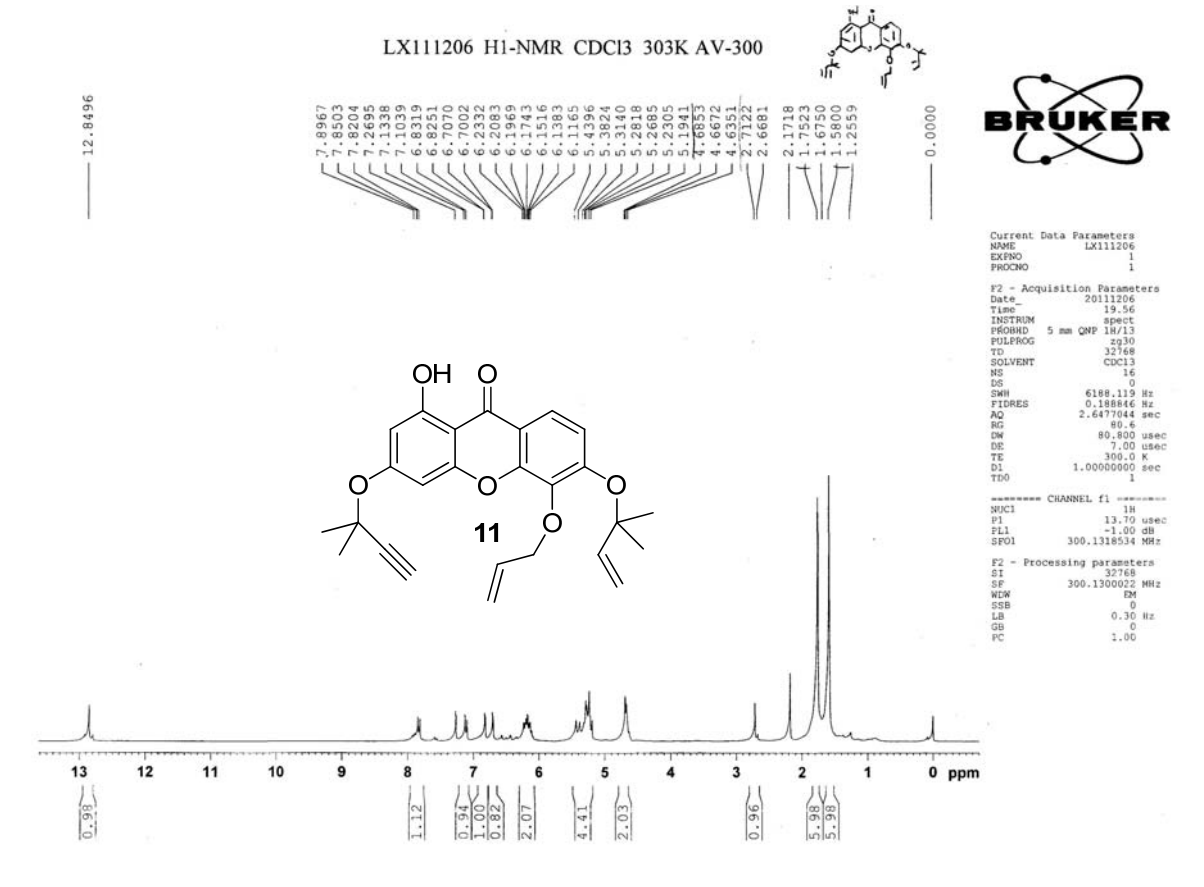


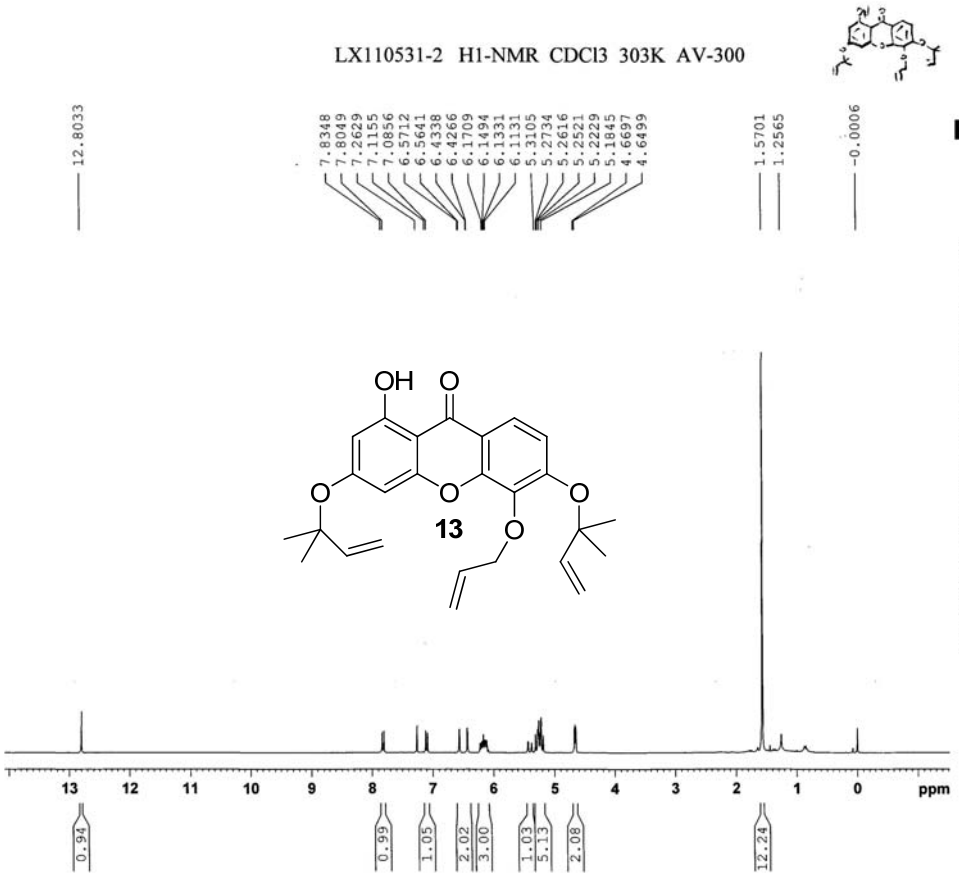

```
Current Data Parameters
NAME                 LX111019
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_               20111020
Time                   23.37
INSTRUM                spect
PROBHD    5 mm QNP 1H/13
PULPROG            noesyph
TD                      40960
SOLVENT                CDCl3
NS                         16
DS                          4
SWH                3605.769 Hz
FIDRES             0.088031 Hz
AQ                 5.6798368 sec
RG                         64
DW                   138.667 usec
DE                     7.00 usec
TE                    300.0 K
d0                 0.00012138 sec
D1                 2.00000000 sec
D8                 0.80000001 sec
IN0                0.00027765 sec
ST1CNT                    100

======== CHANNEL f1 ========
NUC1                       1H
P1                     13.70 usec
PL1                    -1.00 dB
SFO1           300.1315007 MHz

F1 - Acquisition parameters
ND0                         1
TD                        200
SFO1              300.1315 MHz
FIDRES          18.008284 Hz
SW                    12.000 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                       1024
SF             300.1300096 MHz
WDW                      SINE
SSB                         2
LB                     0.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
SI                       1024
MC2             States-TPPI
SF             300.1300096 MHz
WDW                      SINE
SSB                         2
LB                     0.00 Hz
GB                          0
```


LX120320 H1-NMR CDCL3 303K AV-300
BRUKER
12
OH
O
OH
ppm


LX8080175 H1-NMR CDCl3 303K AV-300
14
OH
O
OH
ppm


LX110524 H1-NMR CDCL3 303K AV-300
BRUKER
16
Current Data Parameters
NAME LX110524
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110526
Time 19.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 181
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


LX111206 H1-NMR CDCl3 303K AV-300
BRUKER
11
Current Data Parameters
NAME LX111206
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111206
Time 19.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 80.6
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

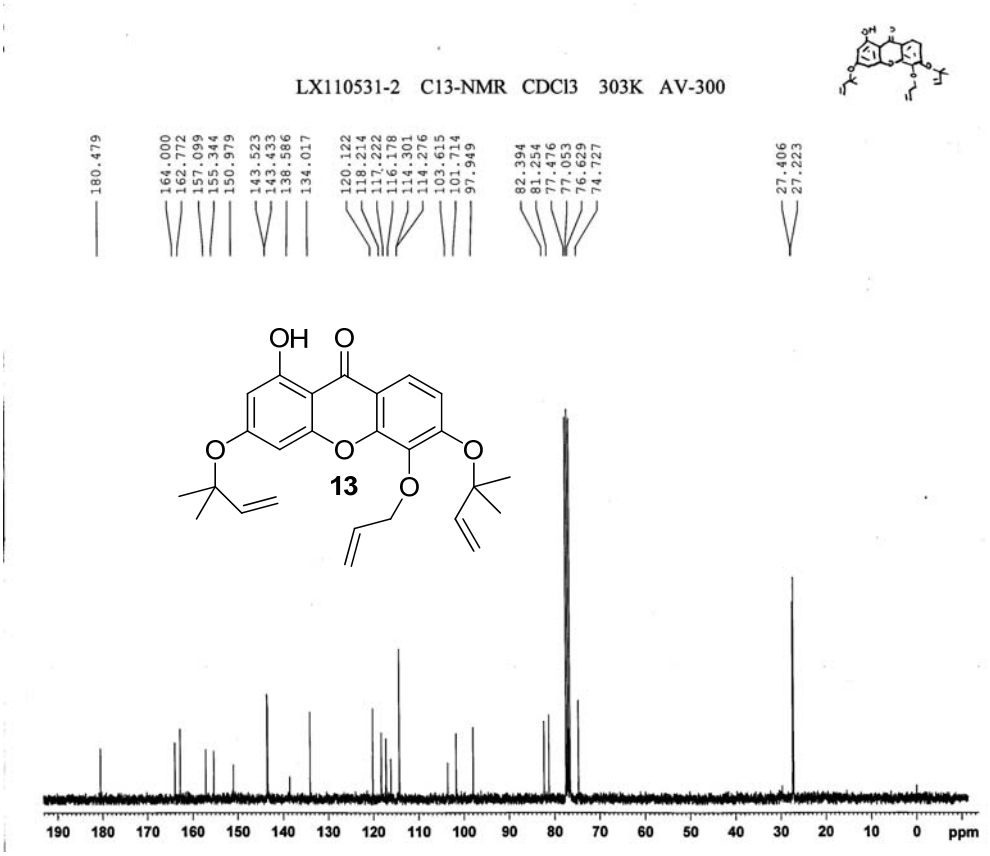


Current Data Parameters
NAME LX110531-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110531
Time 20.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 80.6
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



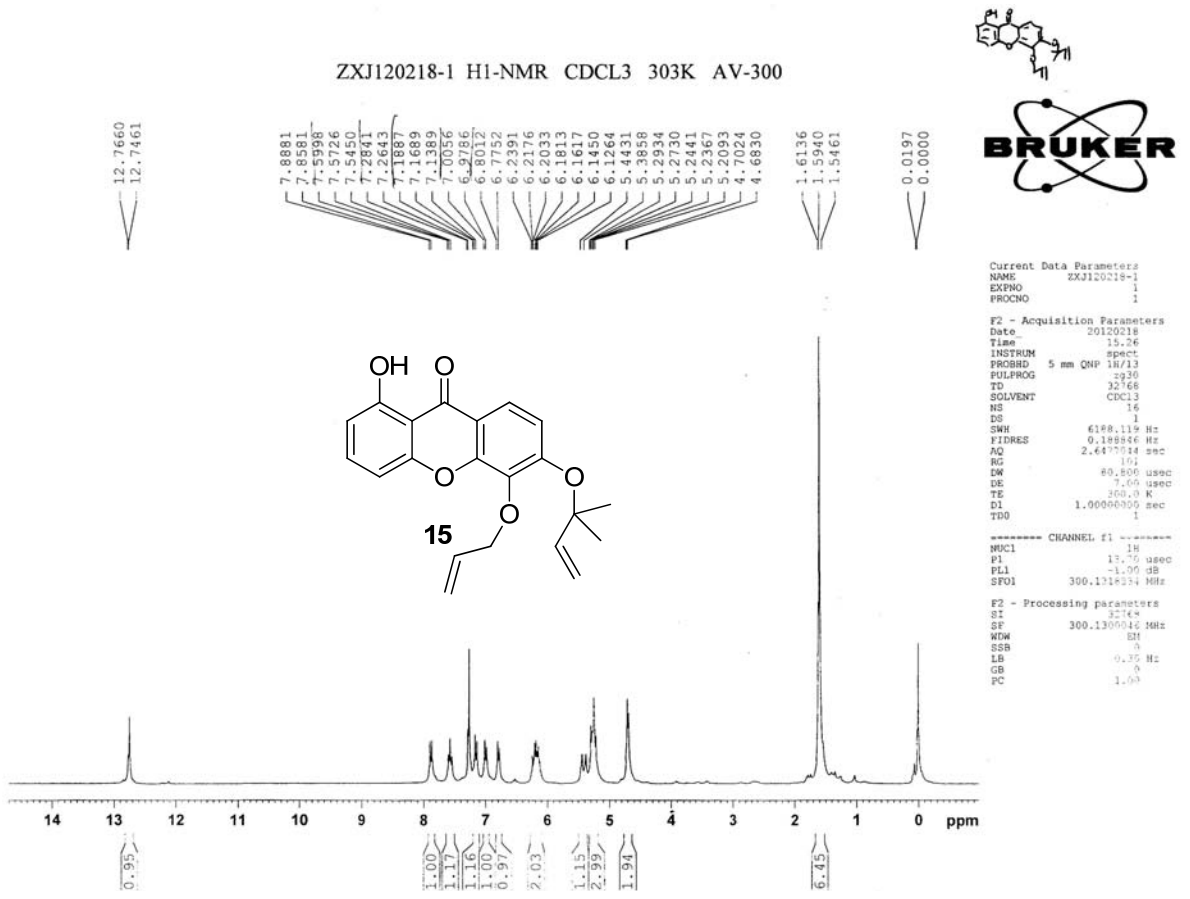
Current Data Parameters
NAME LX110531-2
EXPNO 2
PROCNO 1

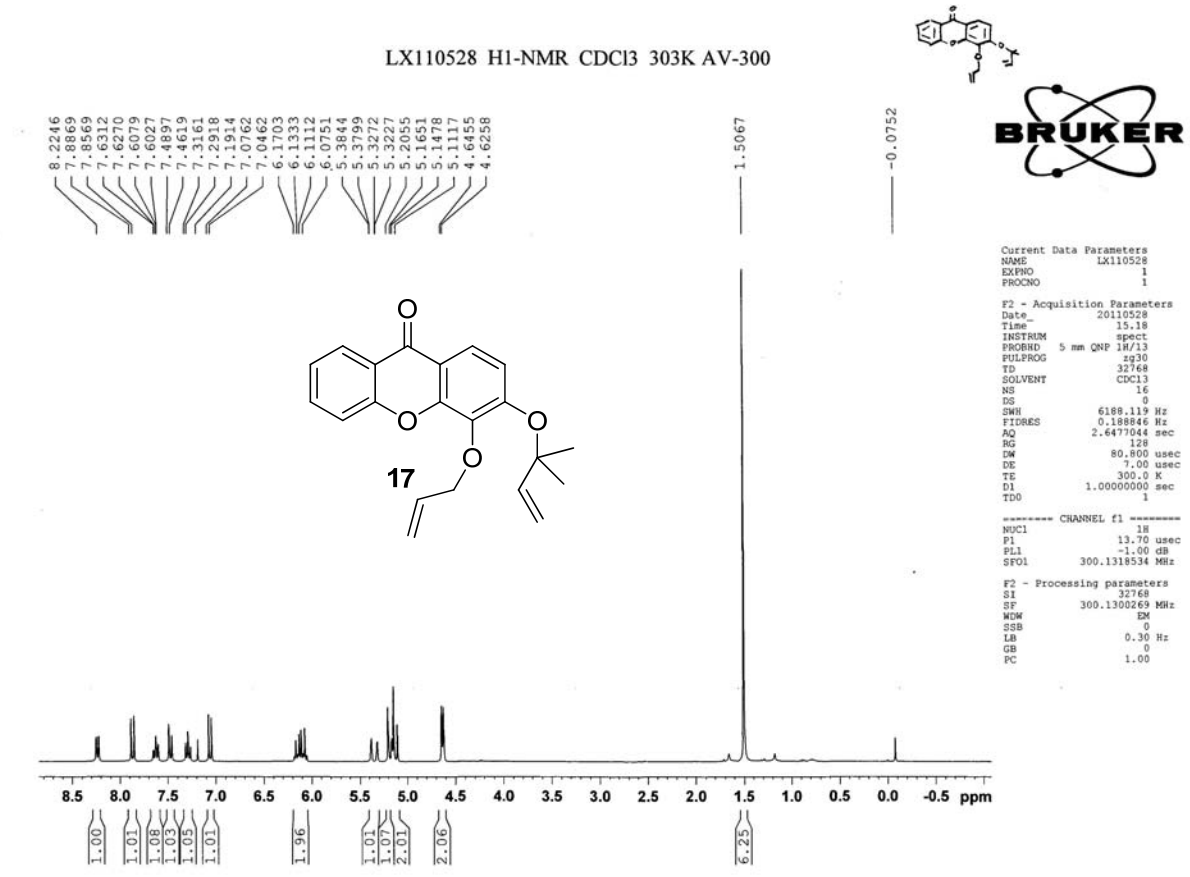
F2 - Acquisition Parameters
Date_ 20110531
Time 20.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 168
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 114
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

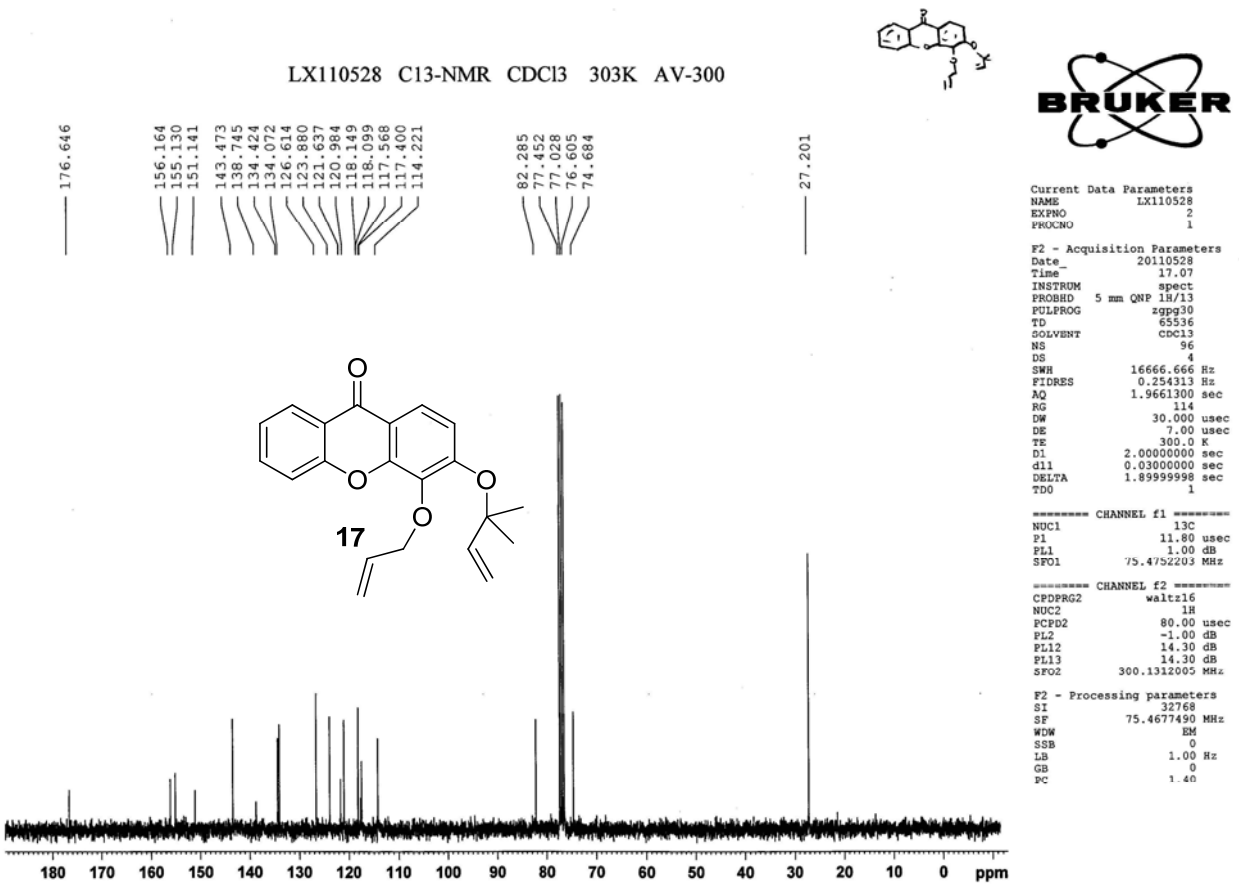
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz

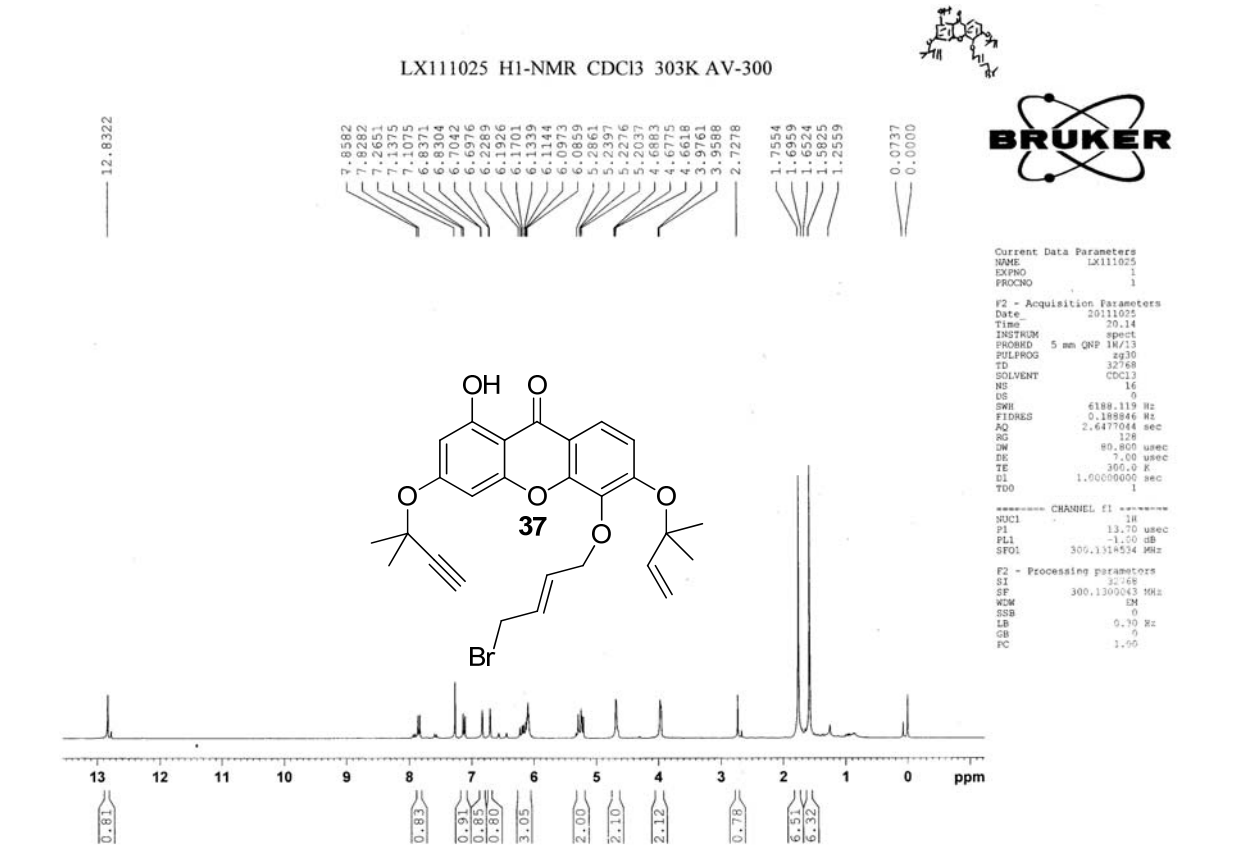
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz

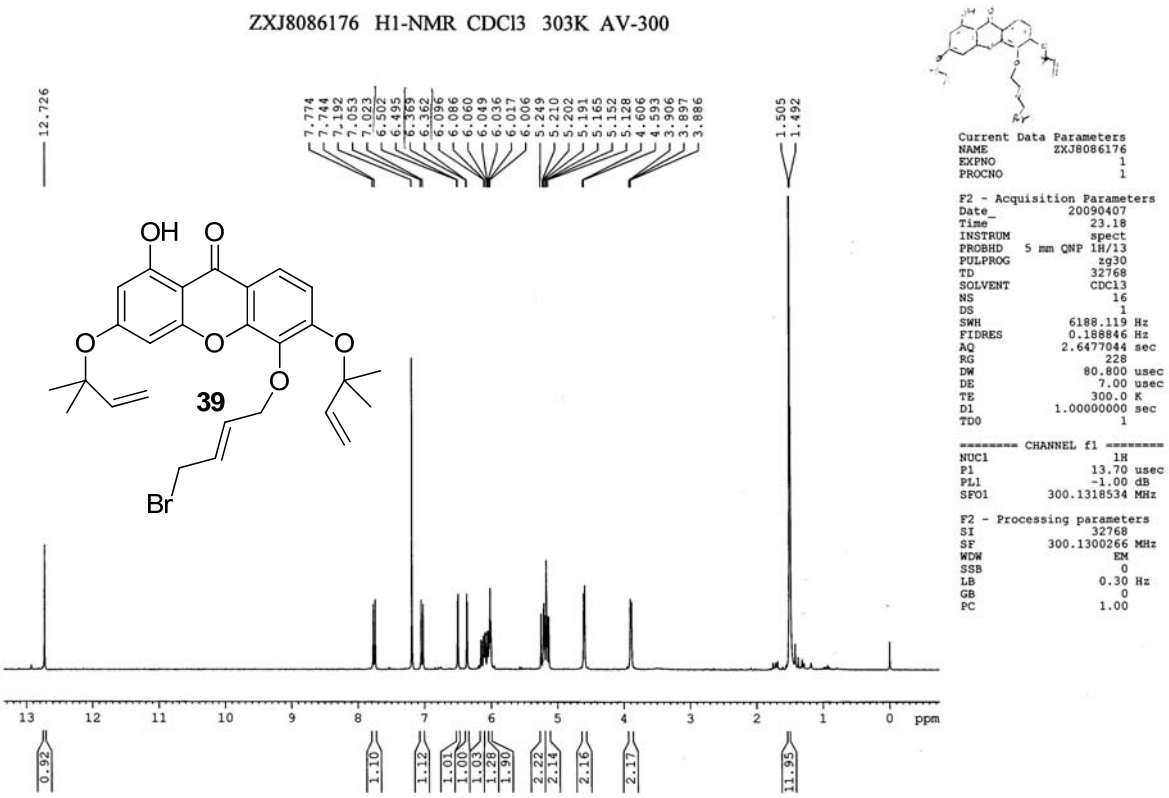
F2 - Processing parameters
SI 32768
SF 75.4677466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

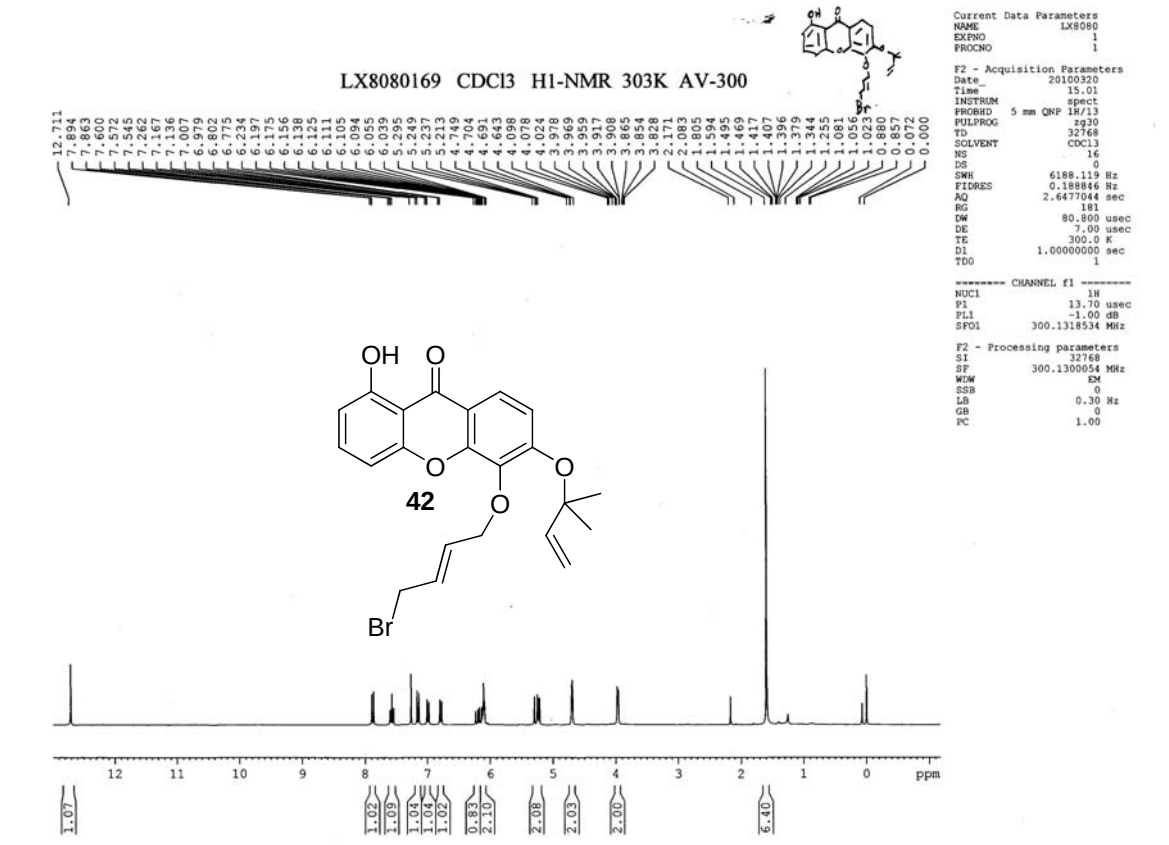

ZXJ120218-1 H1-NMR CDCL3 303K AV-300
BRUKER
12.7660
12.7461
7.8881
7.8581
7.5998
7.5726
7.5450
7.2841
7.2643
7.1887
7.1689
7.1389
7.0056
6.9786
6.8012
6.7752
6.2391
6.2176
6.2033
6.1813
6.1617
6.1450
6.1264
5.4431
5.3858
5.2934
5.2730
5.2441
5.2367
5.2093
4.7024
4.6830
1.6136
1.5940
1.5461
0.0197
0.0000
OH
O
O
O
O
15
Current Data Parameters
NAME ZXJ120218-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120218
Time_ 15.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 161
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318234 MHz
F2 - Processing parameters
SI 32768
SF 300.1300042 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
1.00
1.17
1.16
1.00
0.97
2.03
1.15
2.99
1.94
6.45

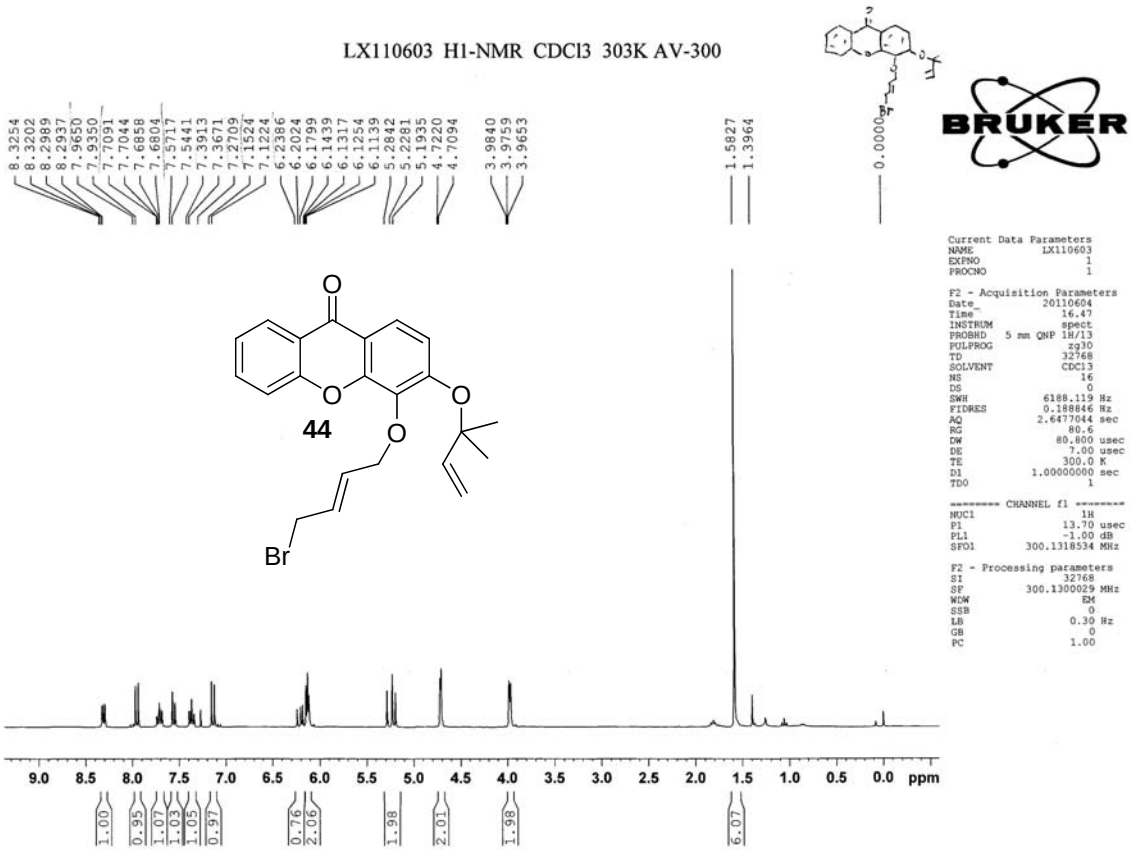

LX110528 H1-NMR CDCl3 303K AV-300
BRUKER
8.2246
7.8869
7.8569
7.6312
7.6270
7.6079
7.6027
7.4897
7.4619
7.3161
7.2918
7.1914
7.0762
7.0462
6.1703
6.1333
6.1112
6.0751
5.3844
5.3799
5.3272
5.3227
5.2055
5.1651
5.1478
5.1117
4.6455
4.6258
1.5067
-0.0752
O
O
O
O
17
Current Data Parameters
NAME LX110528
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110528
Time_ 15.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 128
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300269 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
1.00
1.01
1.08
1.03
1.05
1.01
1.96
1.01
1.07
2.01
2.06
6.25

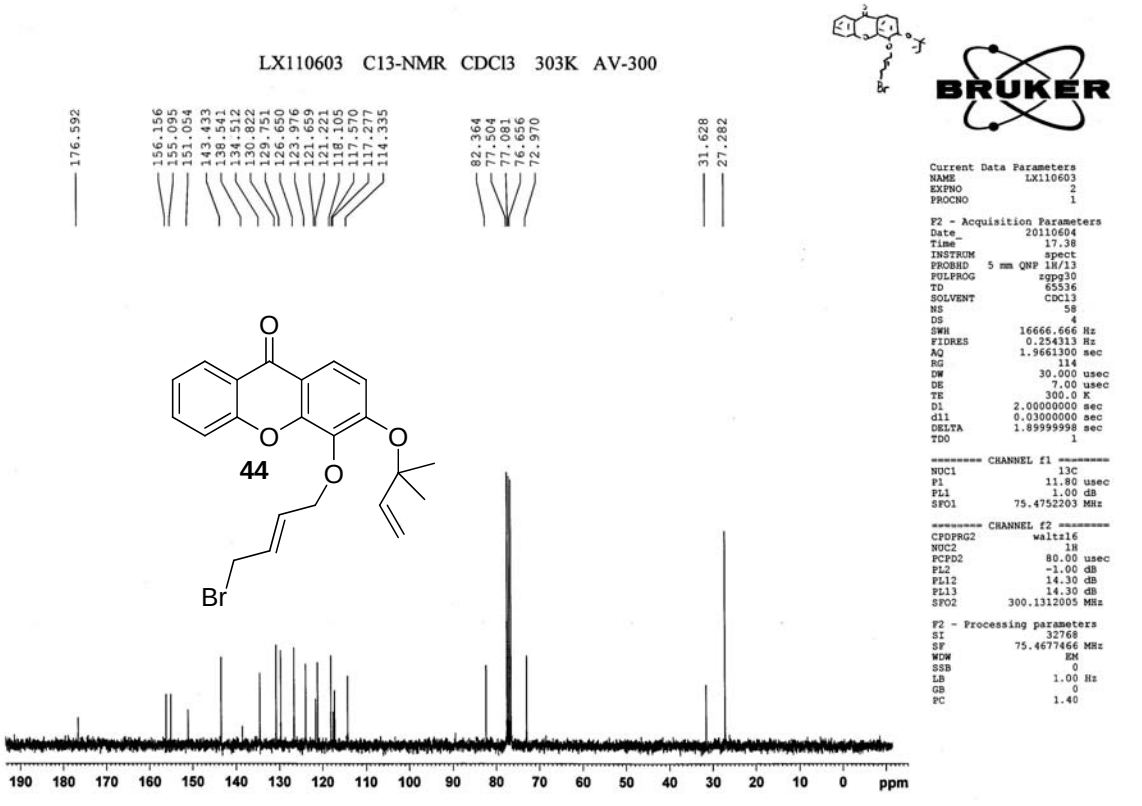

LX110528 C13-NMR CDCl3 303K AV-300
176.646
156.164
155.130
151.141
143.473
138.745
134.424
134.072
126.614
123.880
121.637
120.984
118.149
118.099
117.568
117.400
114.221
82.285
77.452
77.028
76.605
74.684
27.201
17
Current Data Parameters
NAME LX110528
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110528
Time 17.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 114
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

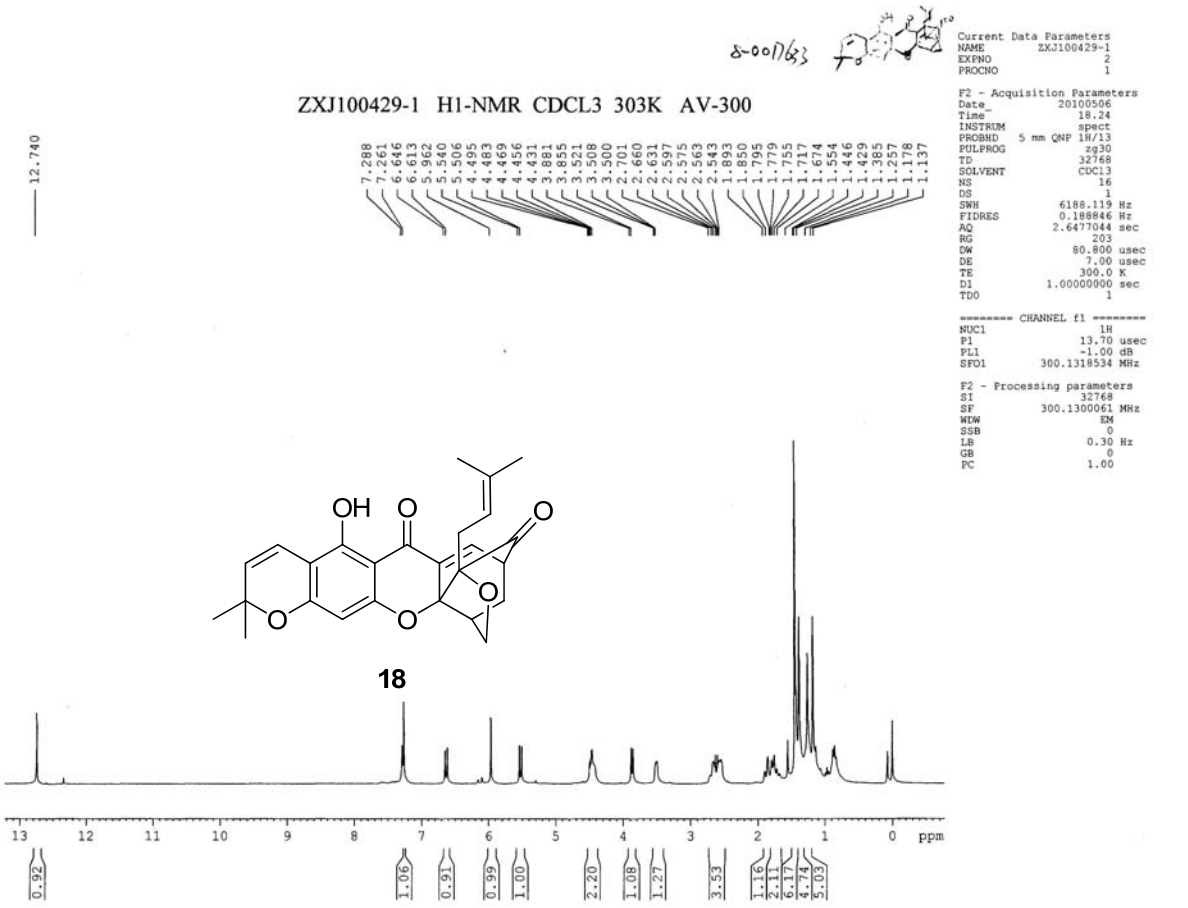

LX111025 H1-NMR CDCl3 303K AV-300
12.8322
7.8582
7.8282
7.2651
7.1375
7.1075
6.8371
6.8304
6.7042
6.6976
6.2289
6.1926
6.1701
6.1339
6.1144
6.0973
6.0859
5.2861
5.2397
5.2276
5.2037
4.6883
4.6775
4.6618
3.9761
3.9588
2.7278
1.7554
1.6959
1.6524
1.5825
1.2559
0.0737
0.0000
OH
37
Br
Current Data Parameters
NAME LX111025
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111025
Time 20.14
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 128
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1314534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.81
0.83
0.91
0.85
0.80
3.05
2.00
2.10
2.12
0.78
6.51
6.32

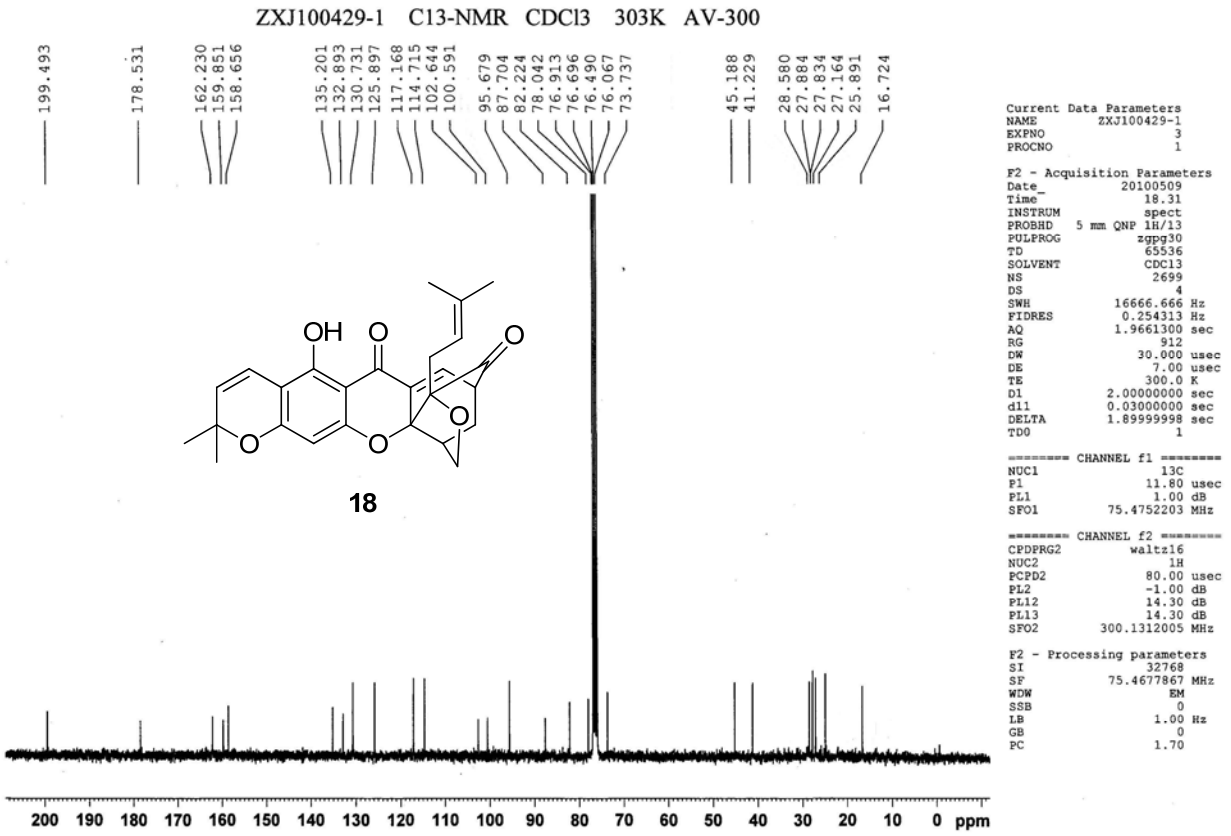

ZXJ8086176 H1-NMR CDCl3 303K AV-300
OH
O
O
O
O
O
39
Br
Current Data Parameters
NAME ZXJ8086176
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090407
Time 23.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300266 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

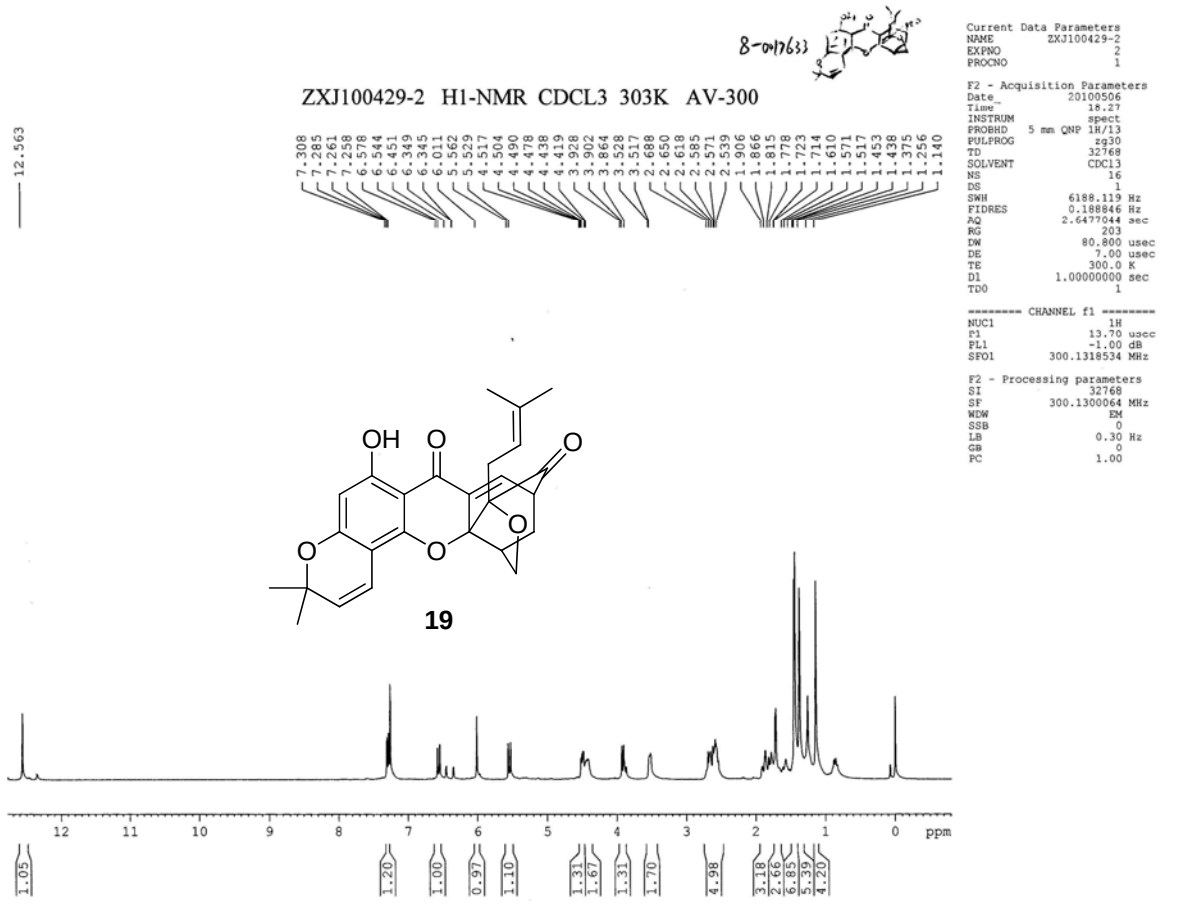

LX8080169 CDCl3 H1-NMR 303K AV-300
OH
O
O
O
O
42
Br
Current Data Parameters
NAME LX8080
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100320
Time 15.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 181
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


LX110603 H1-NMR CDCl3 303K AV-300
BRUKER
44
Br
Current Data Parameters
NAME LX110603
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110604
Time 16.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 80.6
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

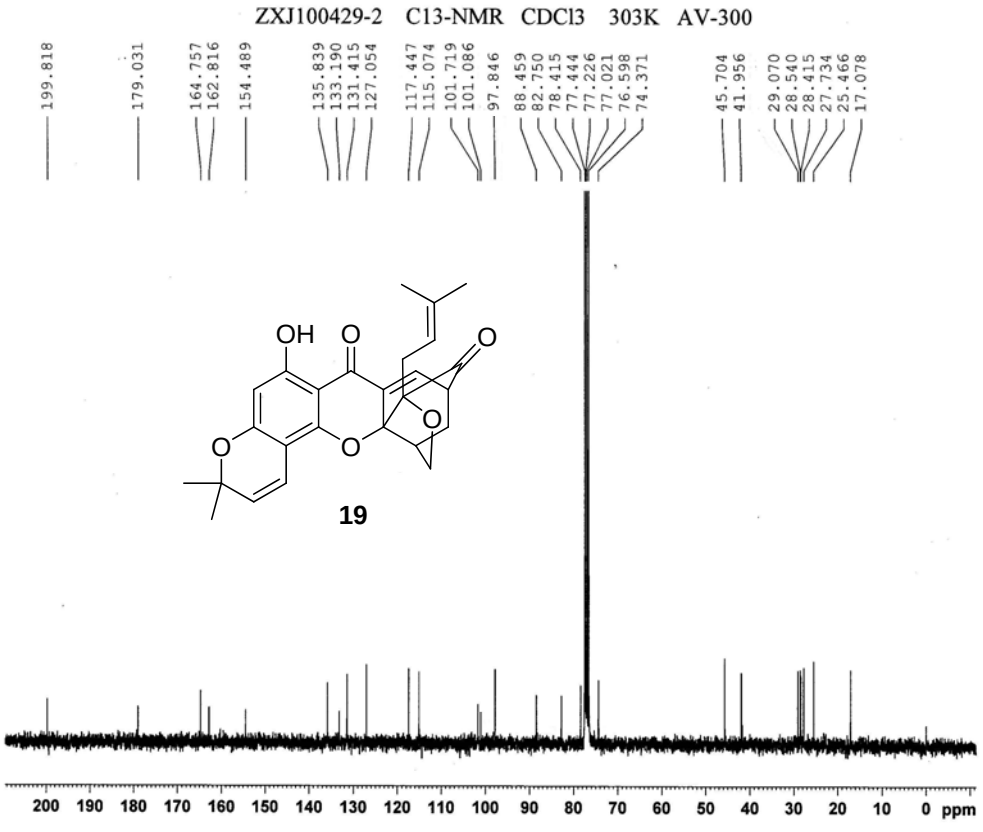

LX110603 C13-NMR CDCl3 303K AV-300
BRUKER
44
Br
Current Data Parameters
NAME LX110603
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110604
Time 17.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 58
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.96613300 sec
RG 114
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ZXJ100429-1 H1-NMR CDCL3 303K AV-300
18
Current Data Parameters
NAME ZXJ100429-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100506
Time 18.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 203
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ZXJ100429-1 C13-NMR CDCl3 303K AV-300
18
Current Data Parameters
NAME ZXJ100429-1
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100509
Time 18.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2699
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 912
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.70

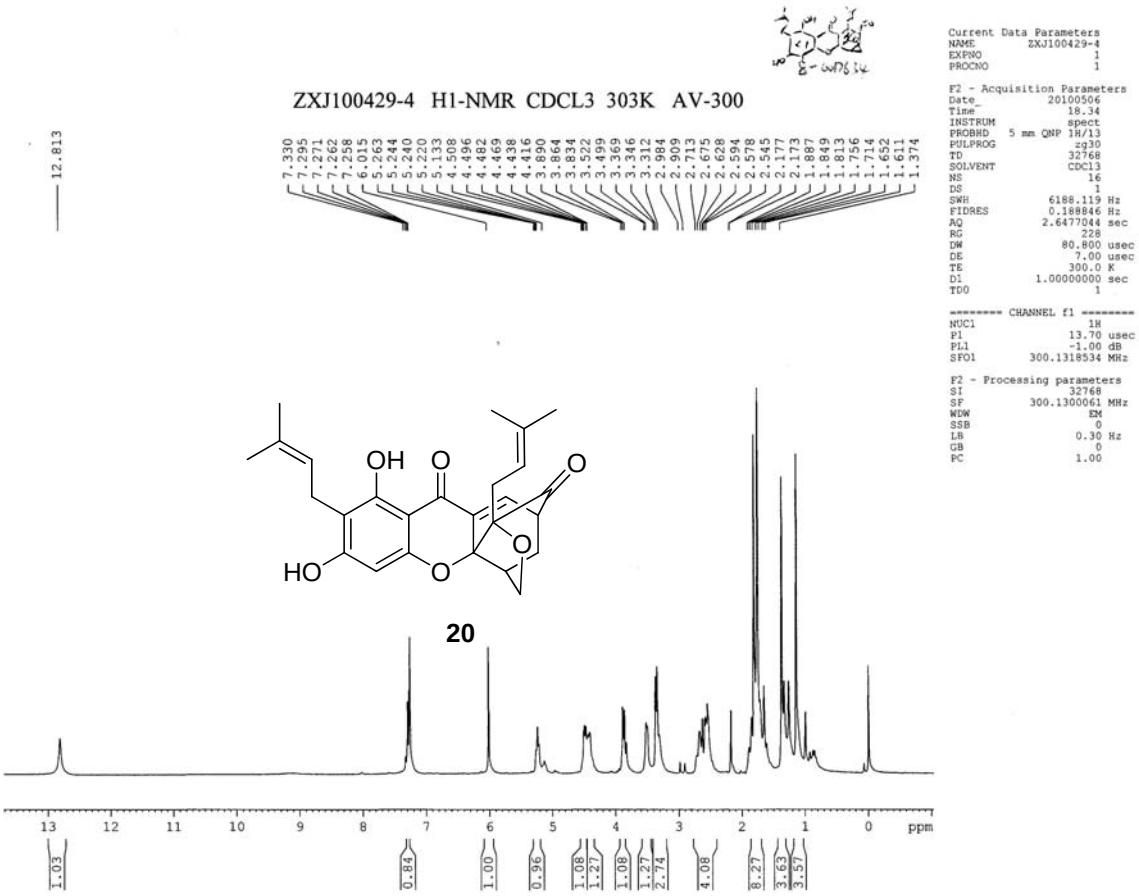



```
Current Data Parameters
NAME          ZXJ100429-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100506
Time_              18.27
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    1
SWH            6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 sec
RG                  203
DW                80.800 usec
DE                 7.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                13.70 usec
PL1               -1.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300064 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```




```
Current Data Parameters
NAME          ZXJ100429-2
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100509
Time_              23.31
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1708
DS                    4
SWH           16666.666 Hz
FIDRES        0.254313 Hz
AQ            1.9661300 sec
RG                  912
DW                30.000 usec
DE                 7.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                11.80 usec
PL1                1.00 dB
SFO1        75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              14.30 dB
PL13              14.30 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677469 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                2.00
```

ZXJ100429-4 H1-NMR CDCL3 303K AV-300



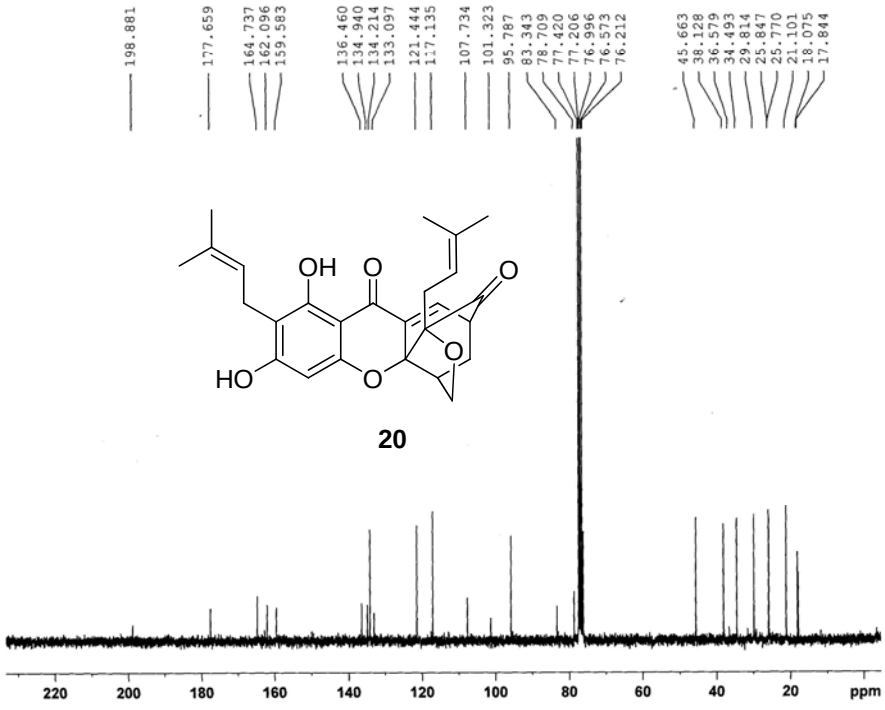
Current Data Parameters
NAME ZXJ100429-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100506
Time 18.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

C13-NMR CDCL3 303K AV-300



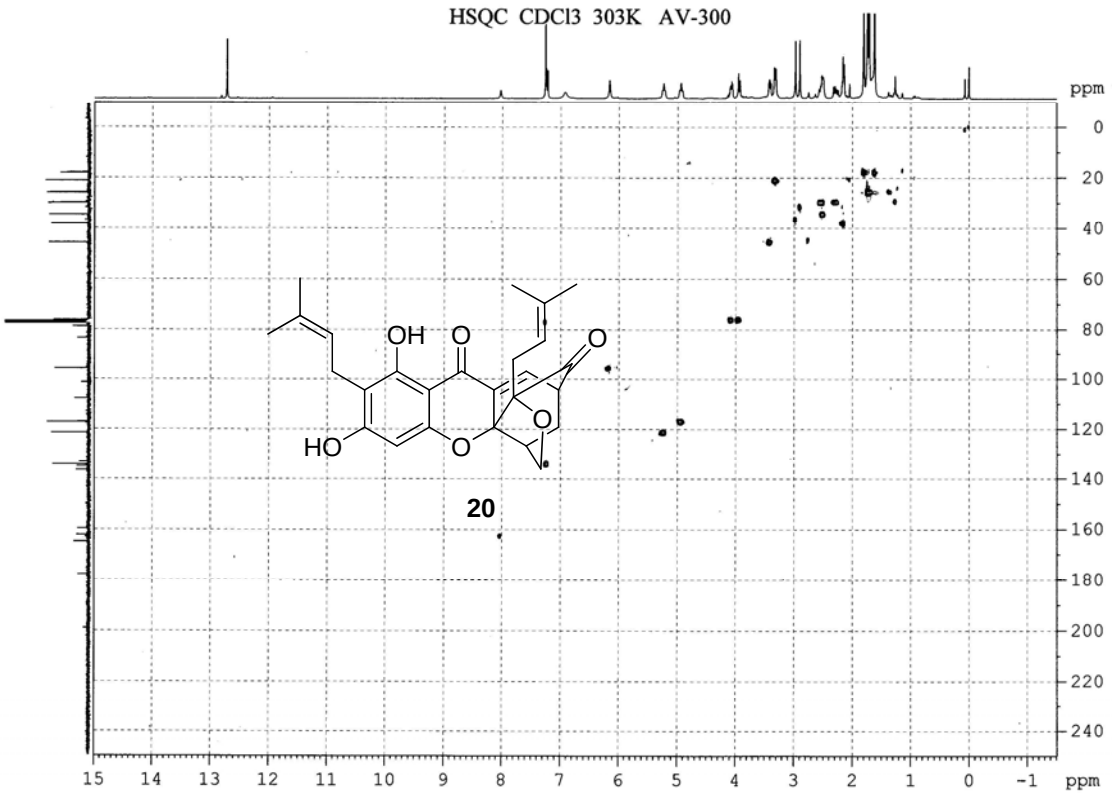
Current Data Parameters
NAME LNG0511305
EXPNO 3
PROCNO 1

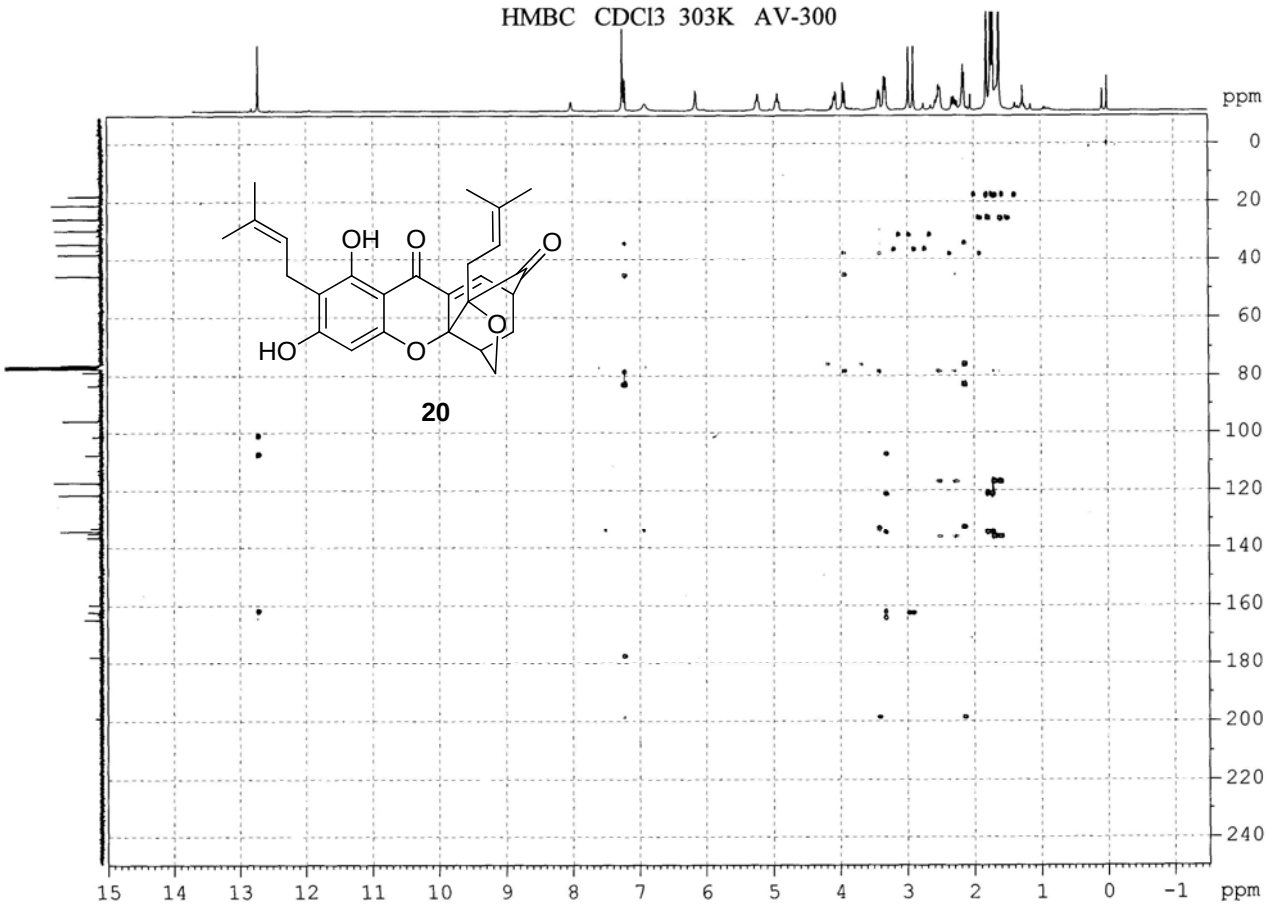
F2 - Acquisition Parameters
Date_ 20070402
Time 9.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 2187
DS 2
SWH 19531.250 Hz
FIDRES 0.596046 Hz
AQ 0.8389108 sec
RG 287
DW 25.600 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4768051 MHz

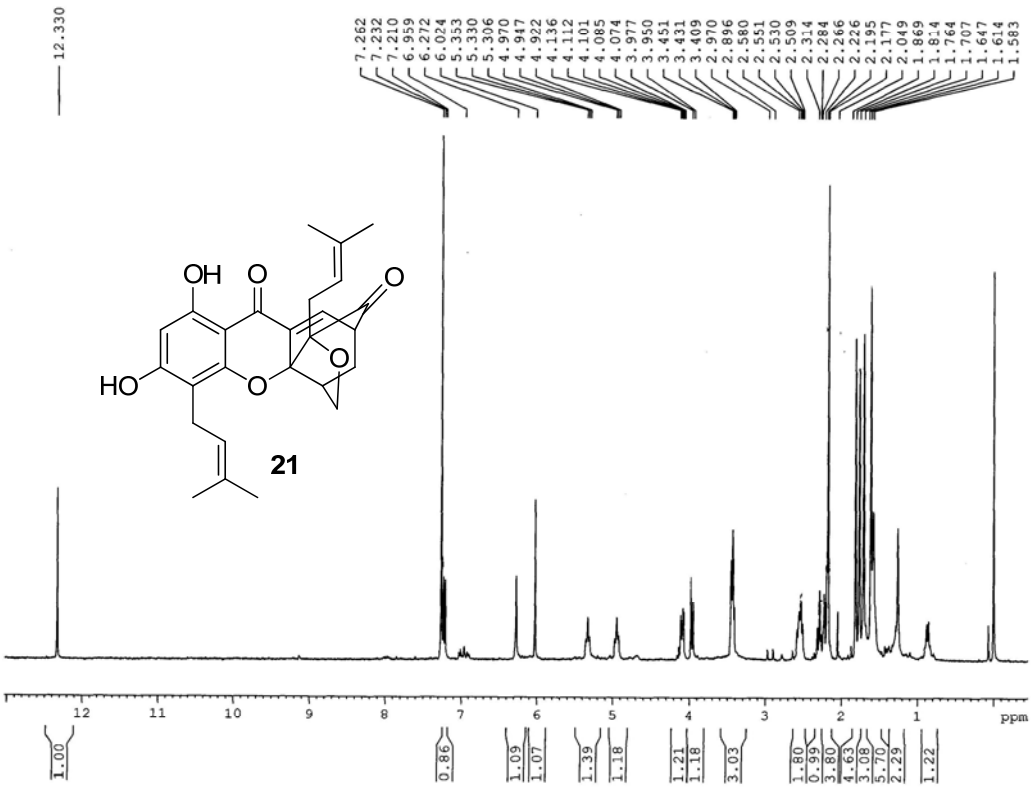
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
SFO2 300.1312005 MHz

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


HSQC CDCl3 303K AV-300
OH
O
O
HO
O
O
20
ppm
0
20
40
60
80
100
120
140
160
180
200
220
240
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm

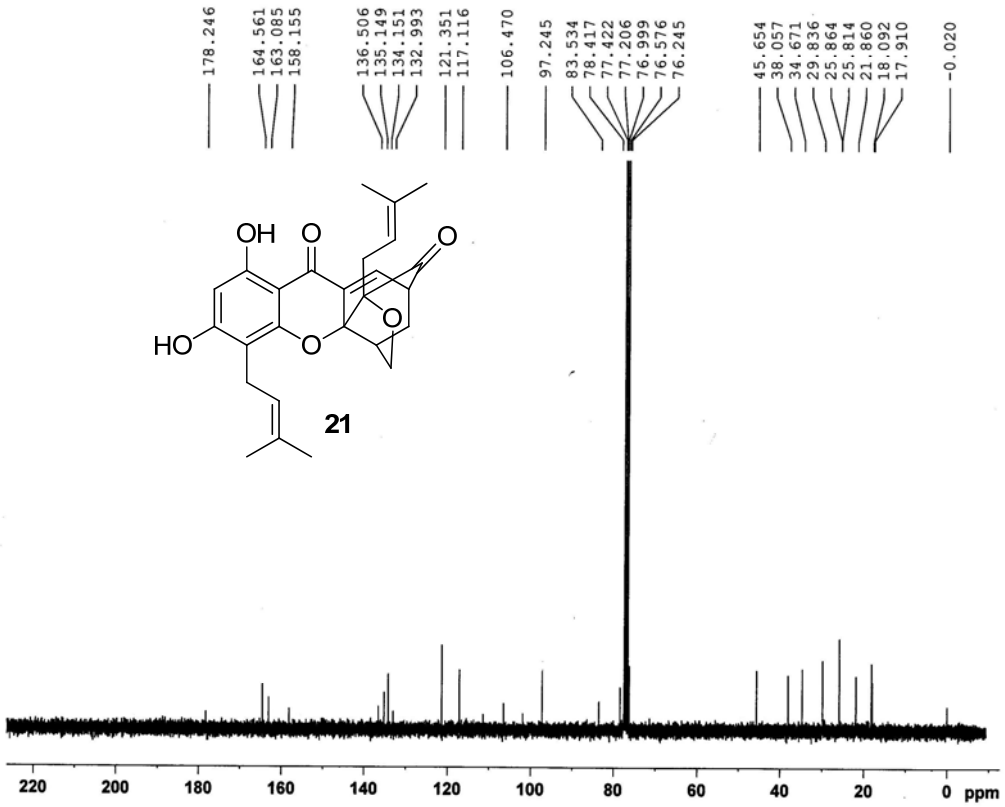

HMBC CDCl3 303K AV-300
OH
O
O
HO
O
O
20
ppm
0
20
40
60
80
100
120
140
160
180
200
220
240
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm

H1-NMR CDCL3 303K AV-300

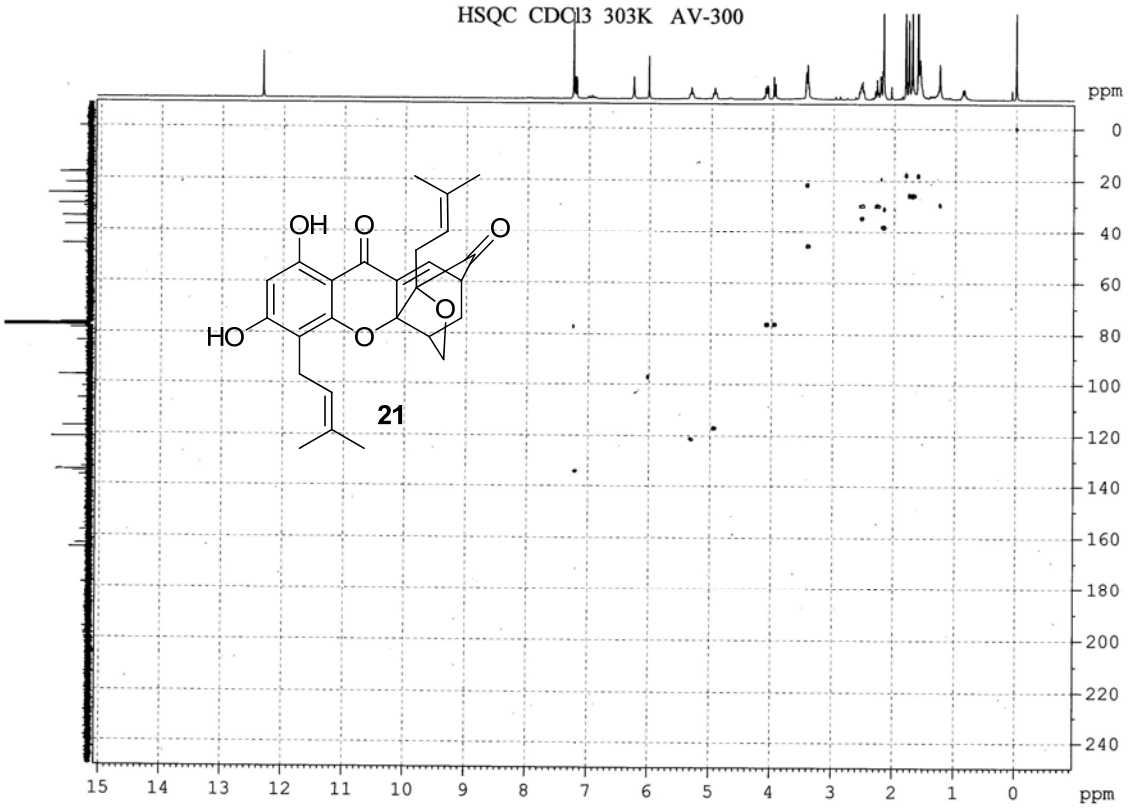


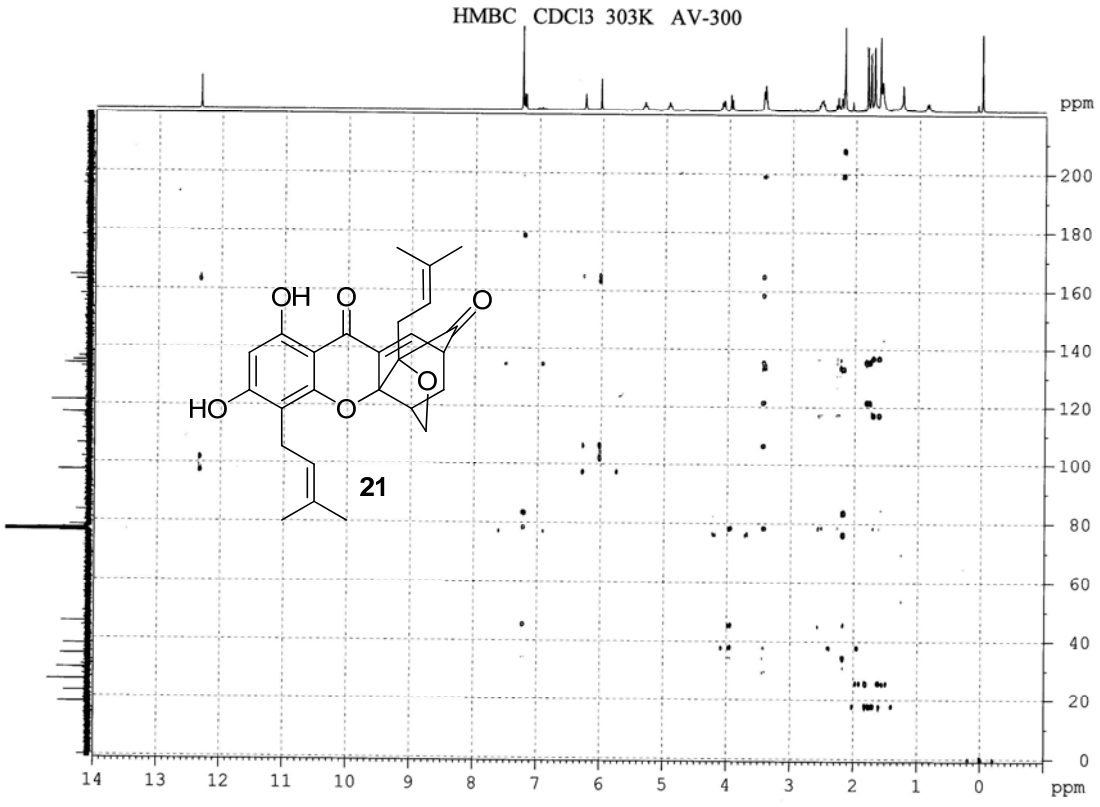

```
Current Data Parameters
NAME          LNG0511303
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070317
Time               13.29
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH          7500.000 Hz
FIDRES          0.114441 Hz
AQ            4.3691168 sec
RG                   181
DW              66.667 usec
DE                7.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.70 usec
PL1               -1.00 dB
SFO1        300.1330013 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300049 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```

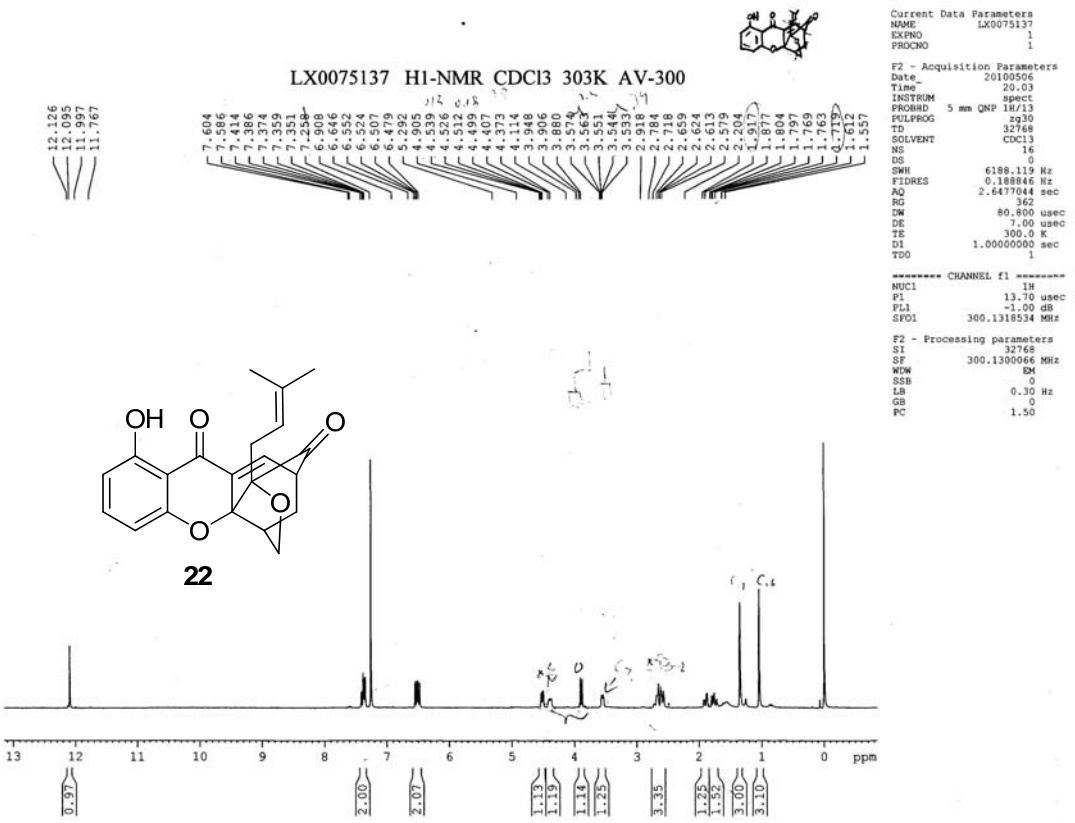
C13-NMR CDCL3 303K AV-300

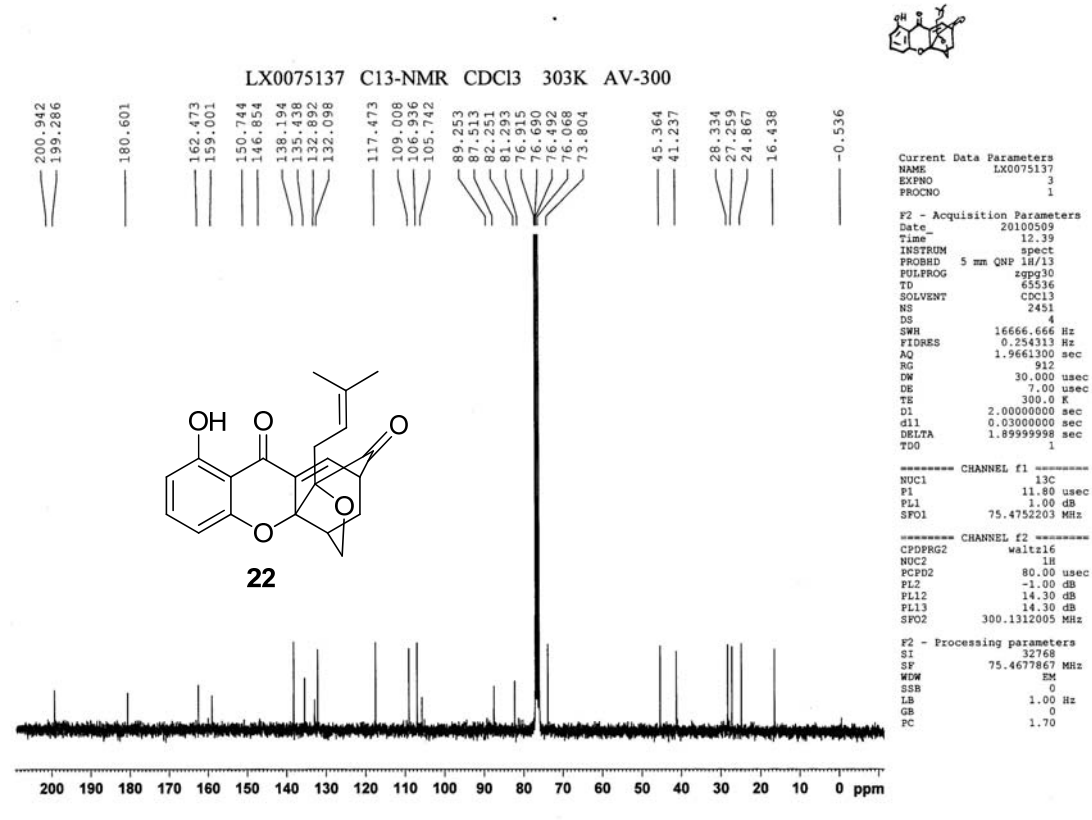



```
Current Data Parameters
NAME          LNG0511303
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070319
Time                9.30
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  5243
DS                     2
SWH          19531.250 Hz
FIDRES          0.298023 Hz
AQ            1.6777716 sec
RG                   287
DW              25.600 usec
DE                7.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.80 usec
PL1                1.00 dB
SFO1         75.4768051 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              14.30 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677490 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                  1.40
```

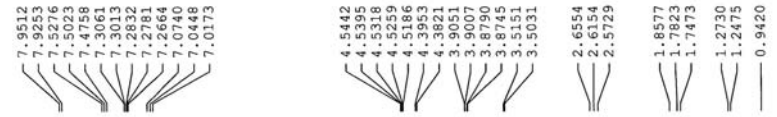

HSQC CDCl3 303K AV-300
OH
O
O
HO
O
O
21
ppm
0
20
40
60
80
100
120
140
160
180
200
220
240
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
ppm

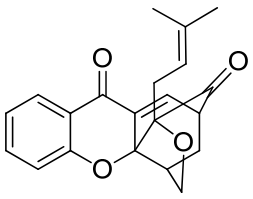

HMBC CDCl3 303K AV-300
OH
O
O
HO
O
O
21
ppm
200
180
160
140
120
100
80
60
40
20
0
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
ppm


LX0075137 H1-NMR CDCl3 303K AV-300
22
ppm

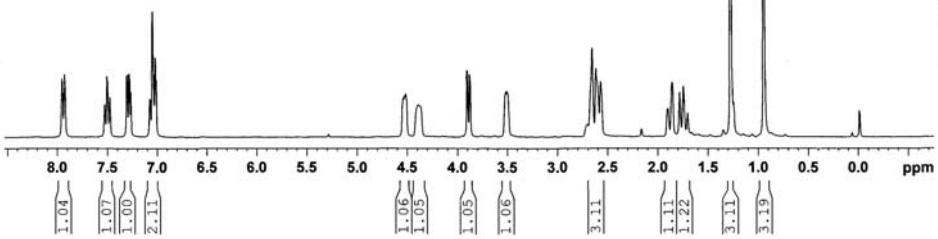

LX0075137 C13-NMR CDCl3 303K AV-300
22
ppm

LX110608 H1-NMR CDCl3 303K AV-300



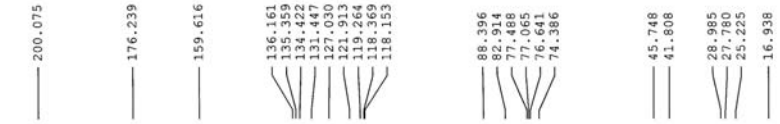


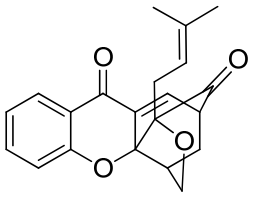
23



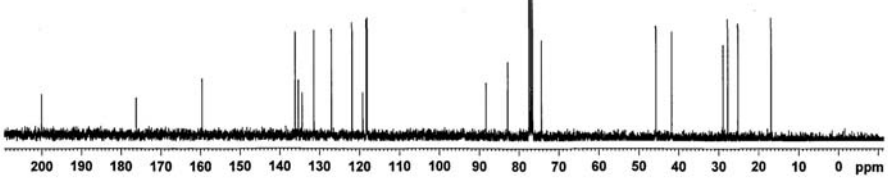

```
Current Data Parameters
NAME                  LX110608
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20110609
Time                   20.03
INSTRUM                spect
PROBHD     5 mm QNP 1H/13
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                4807.692 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                      101
DW                 104.000 usec
DE                   7.00 usec
TE                   300.0 K
D1                2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.70 usec
PL1                  -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300046 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                    1.00
```

LX110608 C13-NMR CDCl3 303K AV-300



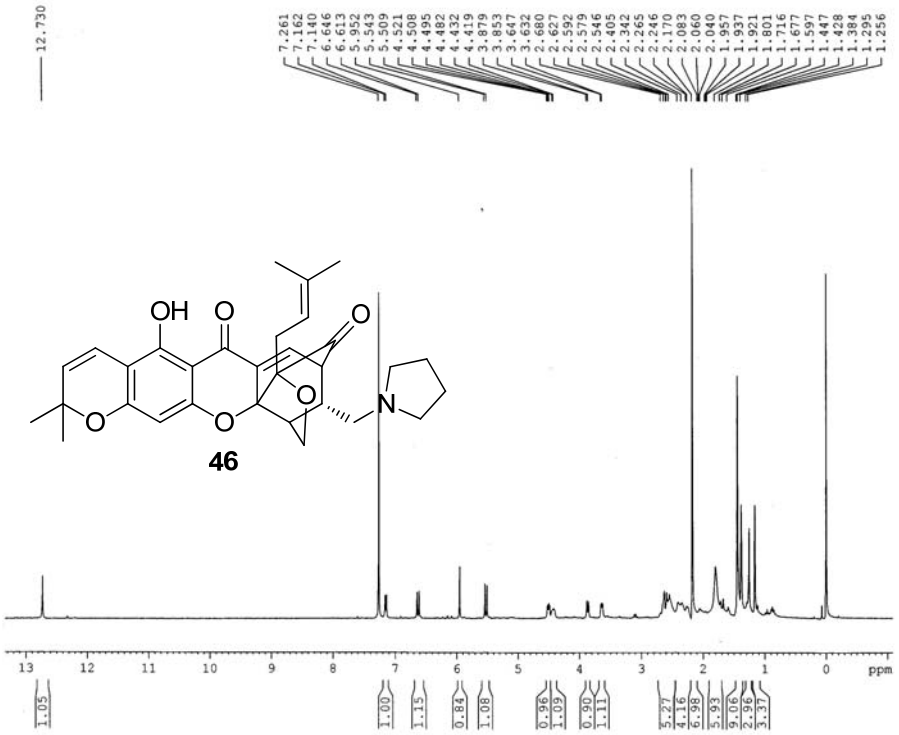


23




```
Current Data Parameters
NAME                  LX110608
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20110609
Time                   20.10
INSTRUM                spect
PROBHD     5 mm QNP 1H/13
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       81
DS                        4
SWH               16666.666 Hz
FIDRES            0.254313 Hz
AQ                1.9661300 sec
RG                      114
DW                  30.000 usec
DE                   7.00 usec
TE                   300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   11.80 usec
PL1                   1.00 dB
SFO1           75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 14.30 dB
PL13                 14.30 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF             75.4677466 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                    1.40
```

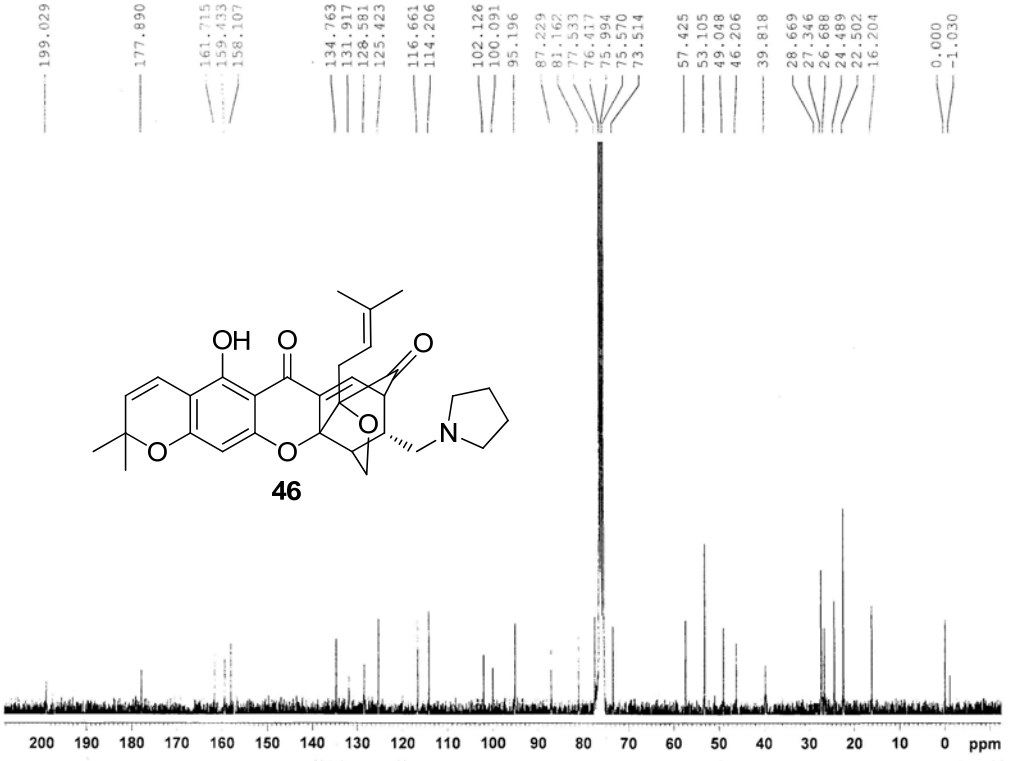
ZXJ090902-1 H1-NMR CDCl3 303K AV-300



46

```
Current Data Parameters
NAME          ZXJ090902-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameter
Date_              20090908
Time_                 21.09
INSTRUM               spect
PROBHD  5 mm QNP 1H/13
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               6188.119 Hz
FIDRES            0.188846 Hz
AQ                2.6477044 se
RG                     228
DW                  80.800 us
DE                   7.00 us
TE                   300.0 K
D1                1.00000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                    1H
P1                  13.70 us
PL1                 -1.00 dB
SFO1          300.1318534 MH

F2 - Processing parameters
SI                   32768
SF                300.1300056 MH
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

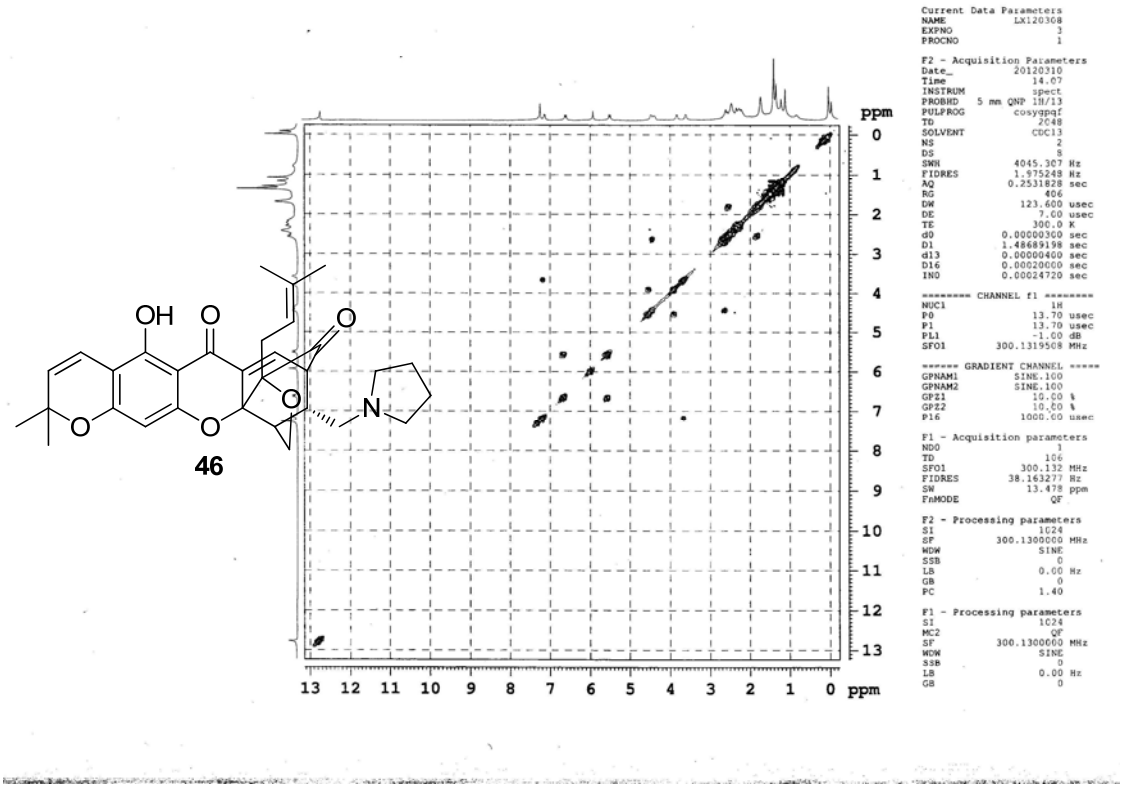
LX120308 C13-NMR CDCl3 303K AV-300



46

```
Current Data Parameters
NAME          LX120308
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_              20120308
Time_                 22.25
INSTRUM               spect
PROBHD  5 mm QNP 1H/13
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    8192
DS                       4
SWH              16666.666 Hz
FIDRES            0.254313 Hz
AQ                1.9661300 sec
RG                     256
DW                 30.000 usec
DE                   7.00 usec
TE                   300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                 11.80 usec
PL1                 1.00 dB
SFO1          75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              80.00 usec
PL2                 -1.00 dB
PL12                14.30 dB
PL13                14.30 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF                75.4678257 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

LX120308 H–H COSY CDCl3 303K AV–300



Current Data Parameters
NAME LX120308
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120310
Time 14.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 2
DS 8
SWH 4045.307 Hz
FIDRES 1.975248 Hz
AQ 0.2531828 sec
RG 406
DW 123.600 usec
DE 7.00 usec
TE 300.0 K
d0 0.00000300 sec
D1 1.48689198 sec
d13 0.00000400 sec
D16 0.00020000 sec
INO 0.00024720 sec

======== CHANNEL f1 ========
NUC1 1H
P0 13.70 usec
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1319508 MHz

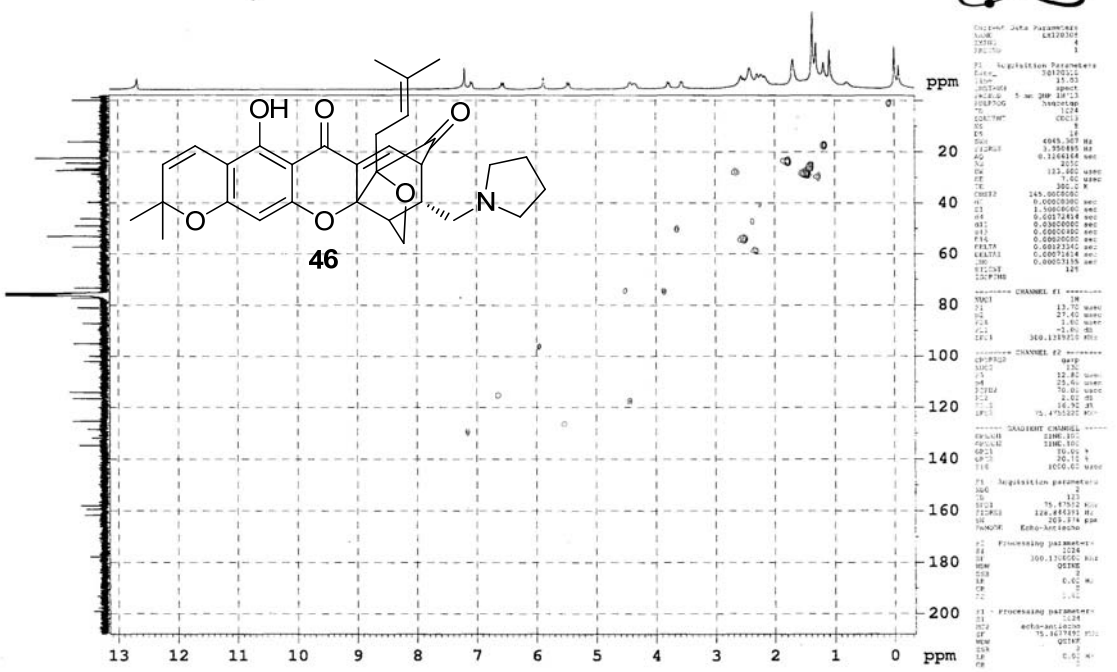
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

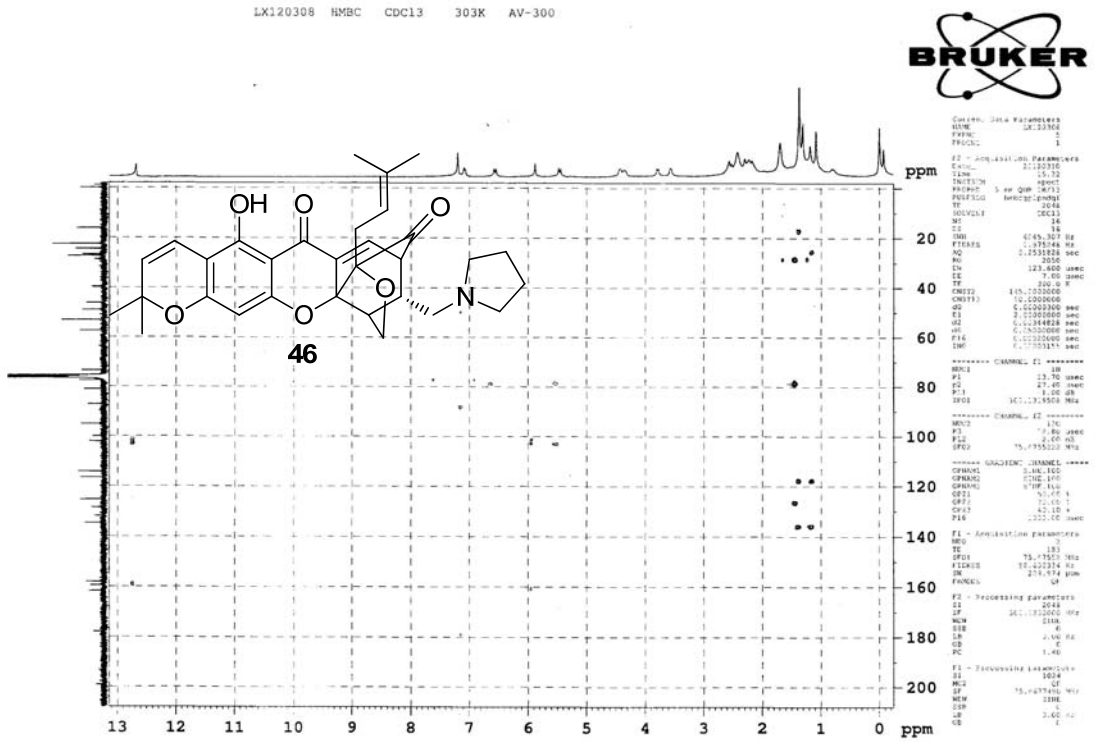
F1 - Acquisition parameters
ND0 1
TD 106
SFO1 300.132 MHz
FIDRES 38.163277 Hz
SW 13.478 ppm
FnMODE QF

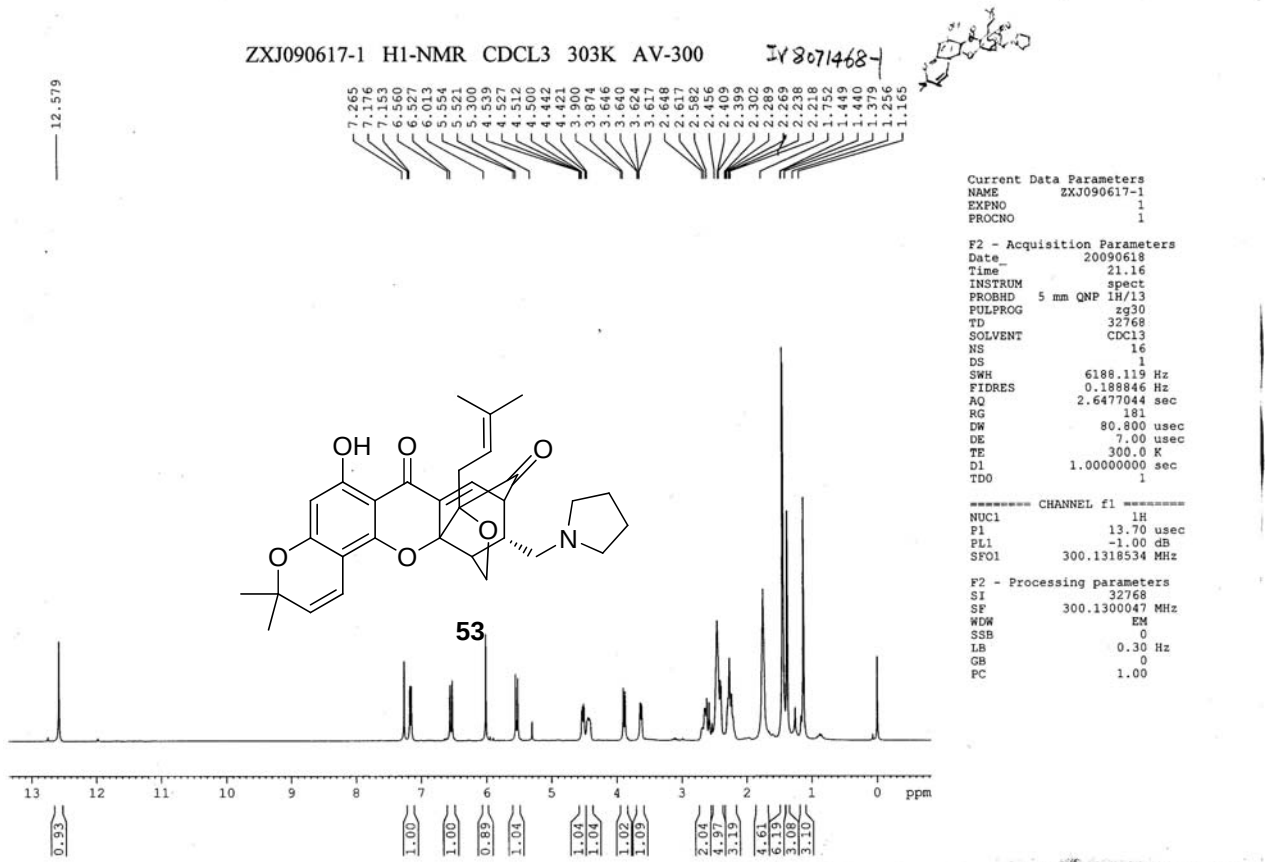
F2 - Processing parameters
SI 1024
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1300060 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

LX120308 HSQC CDCl3 303K AV-300







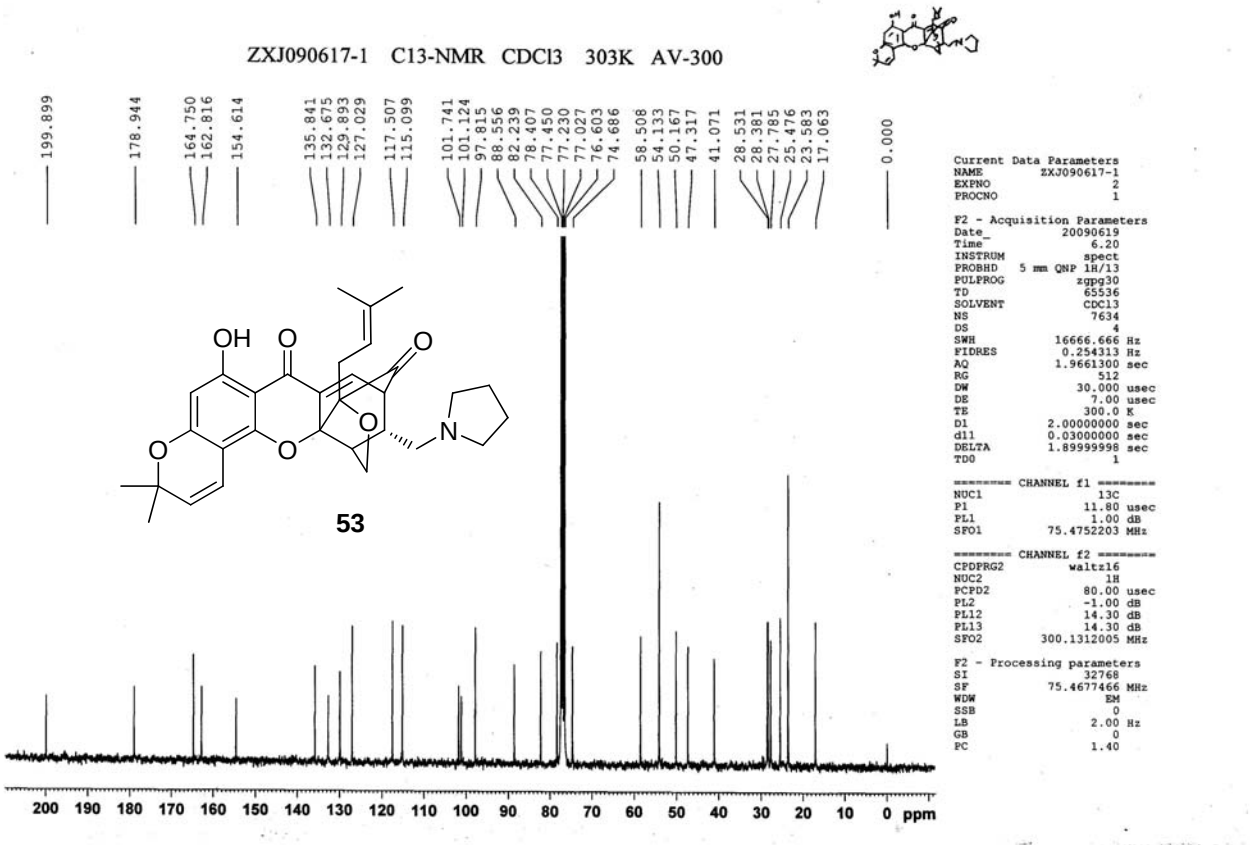
Current Data Parameters
NAME ZXJ090617-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090618
Time 21.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 181
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

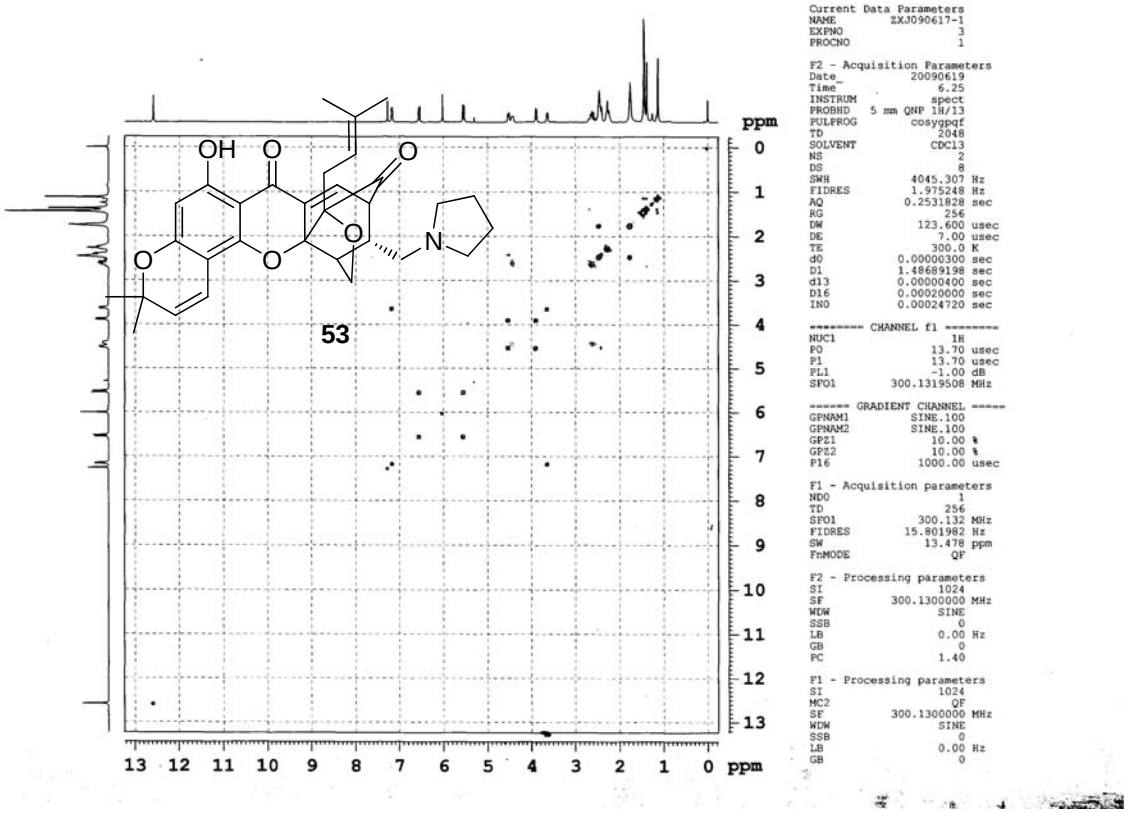
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

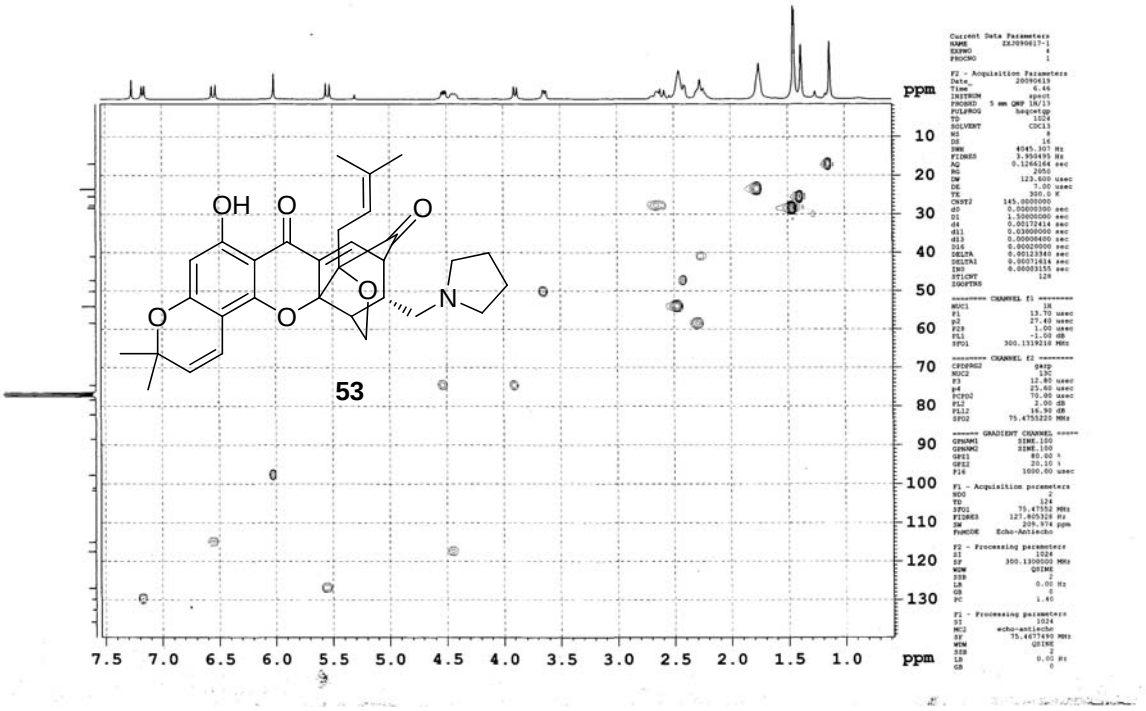
ZXJ090617-1 C13-NMR CDCl3 303K AV-300



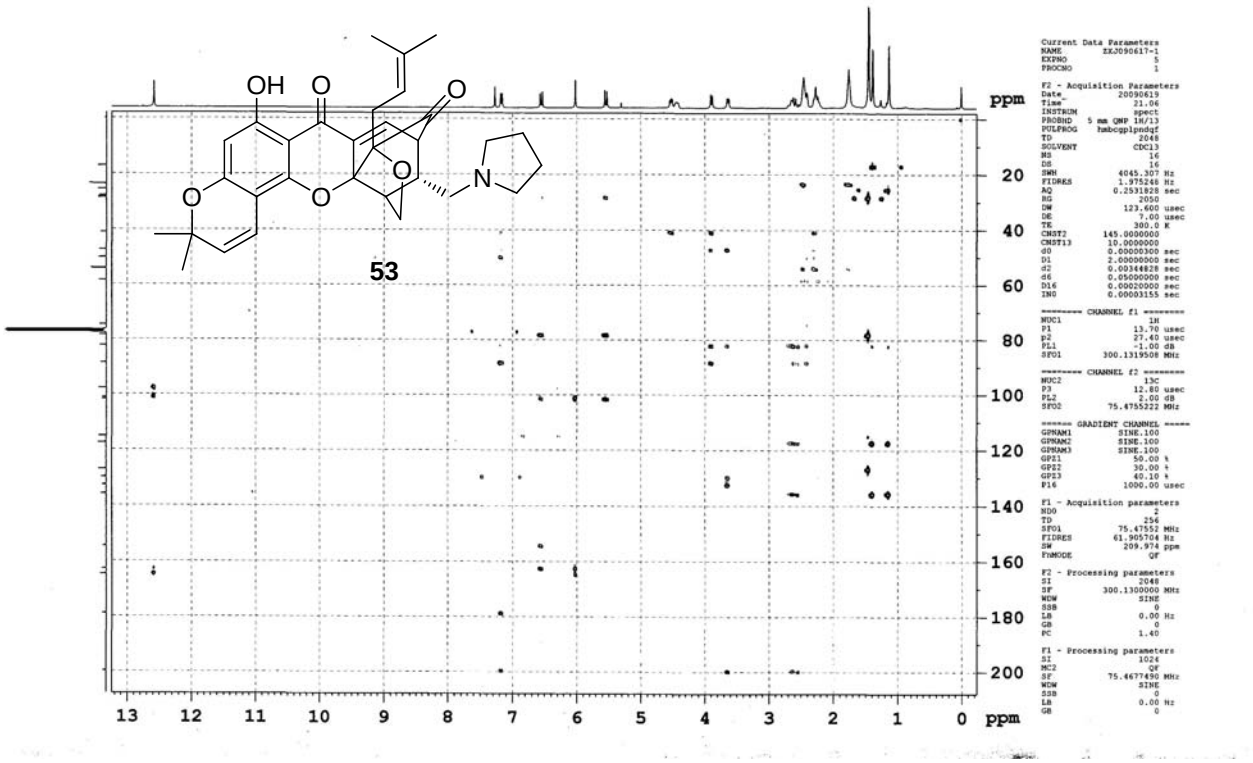
ZXJ090617-1 H-H COSY CDCl3 303K AV-300

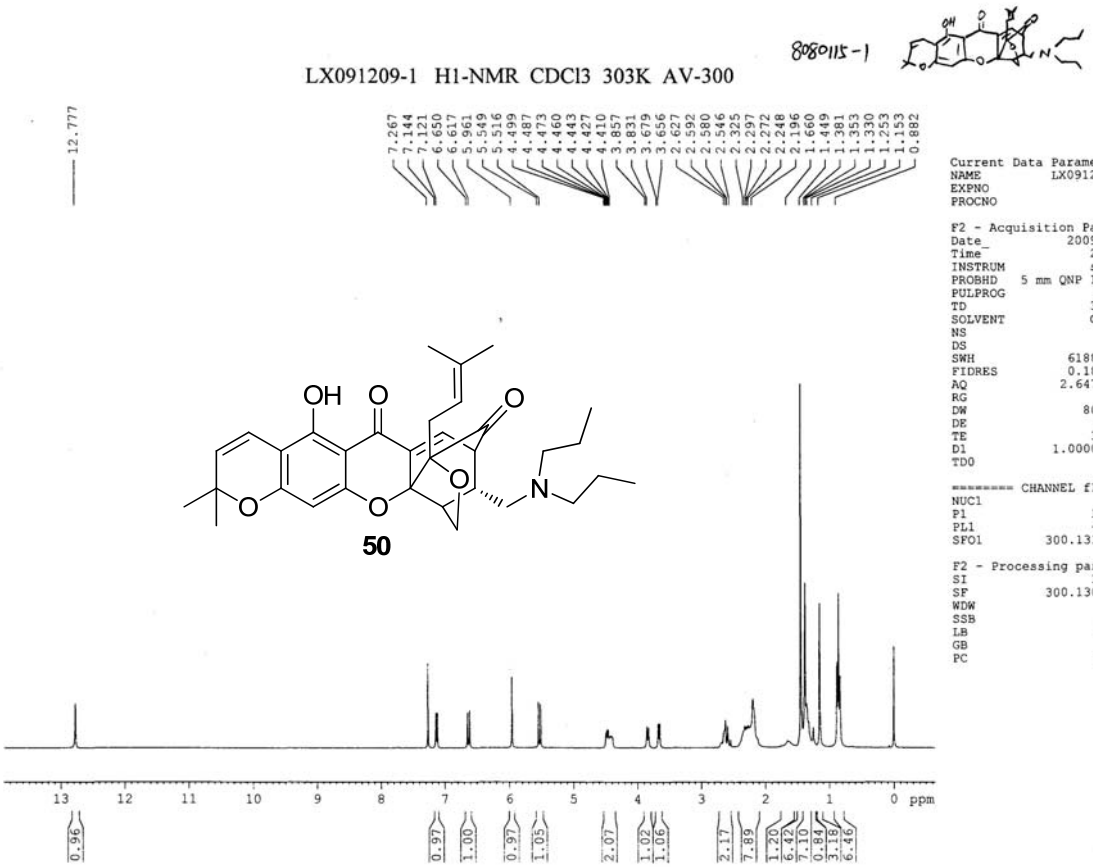


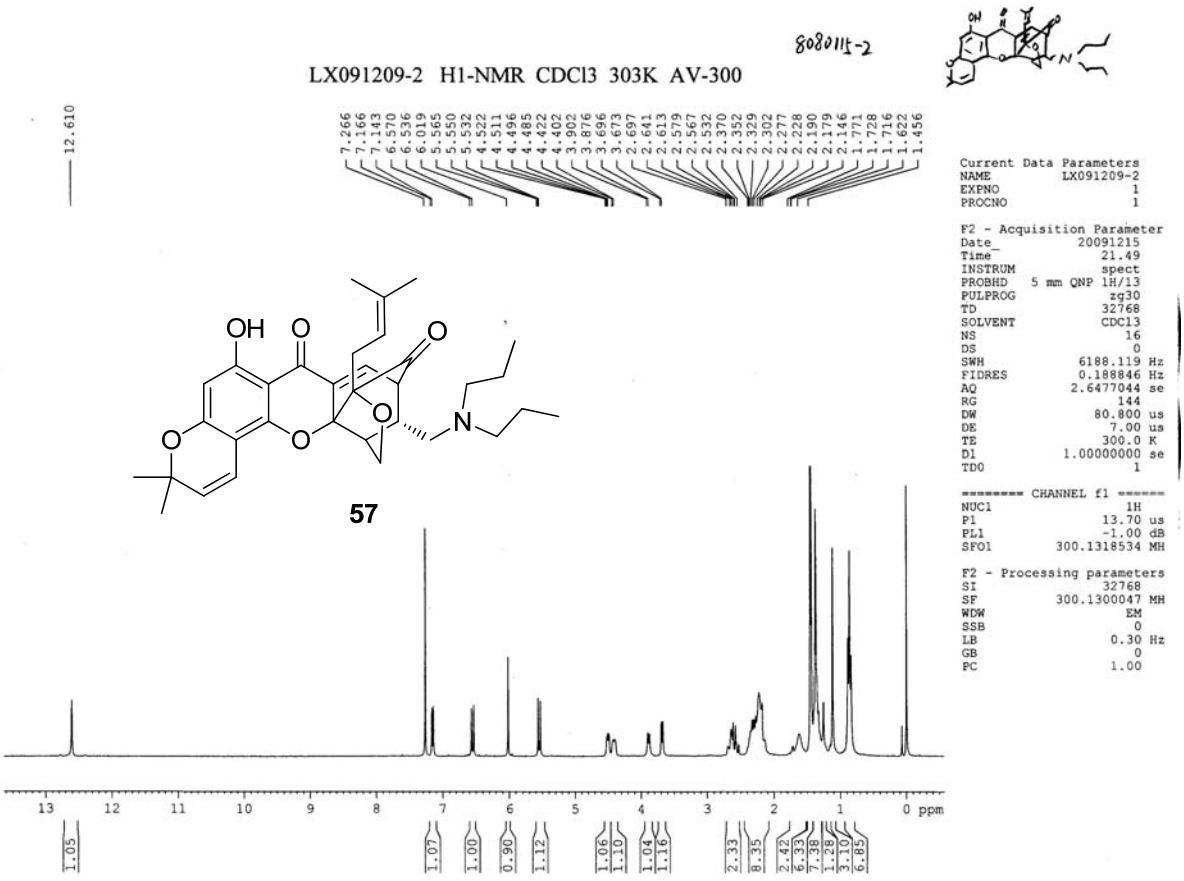
ZXJ090617-1 HSQC CDCl3 303K AV-300

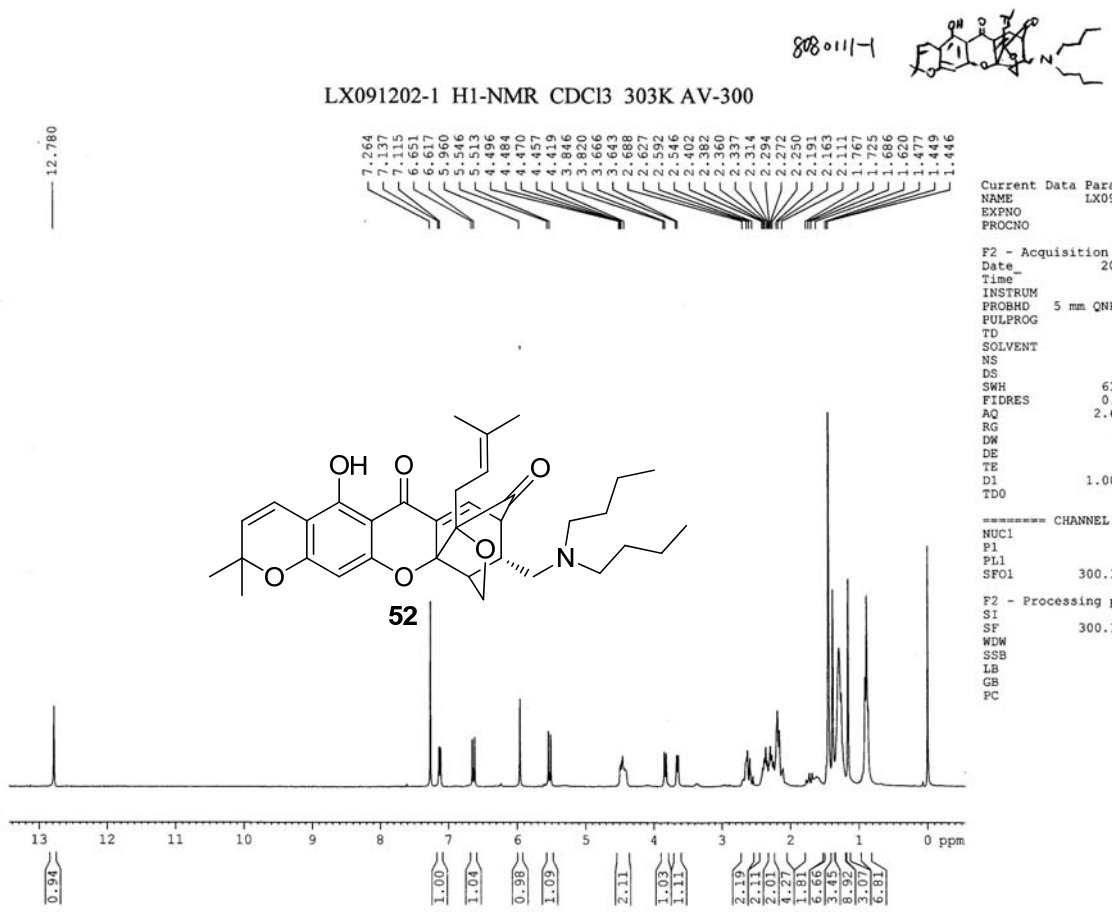


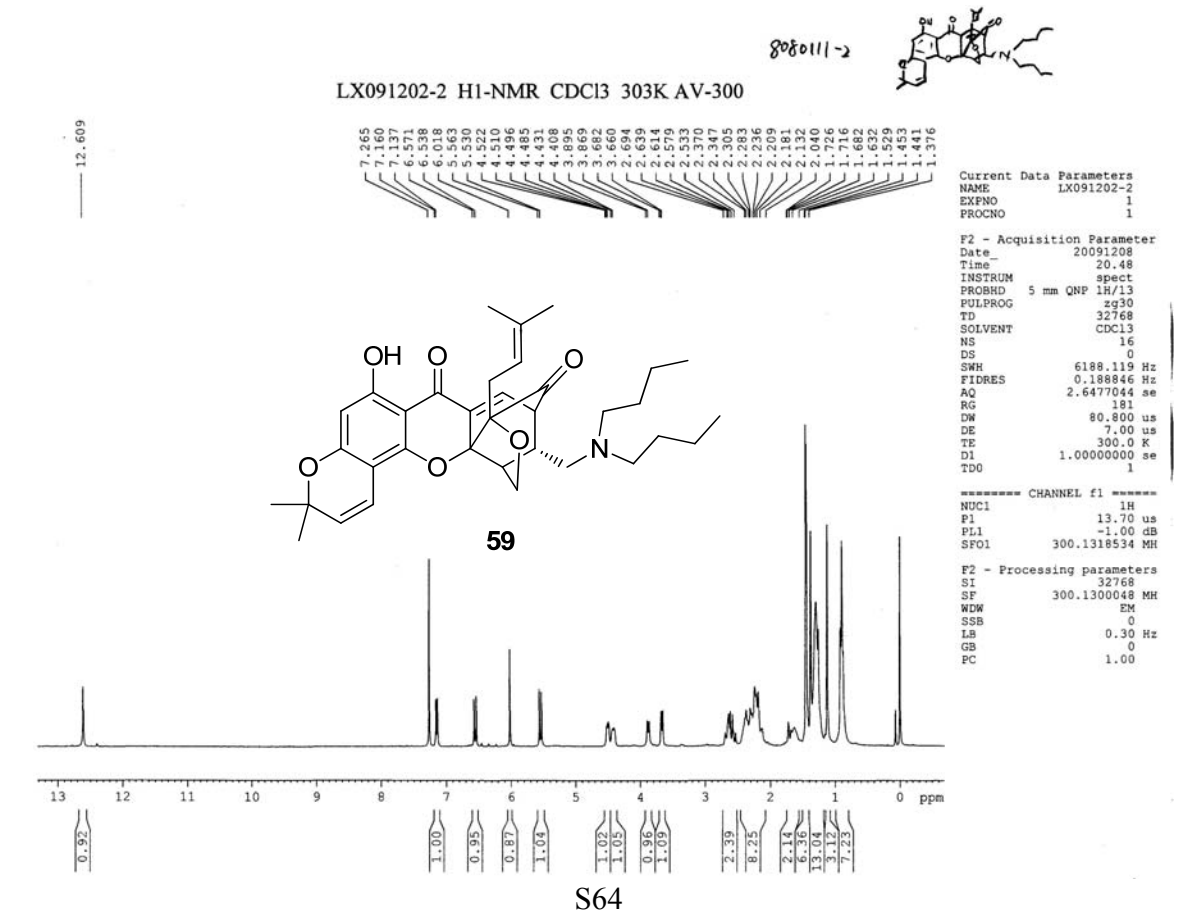
ZXJ090617-1 HMBC CDCl3 303K AV-300



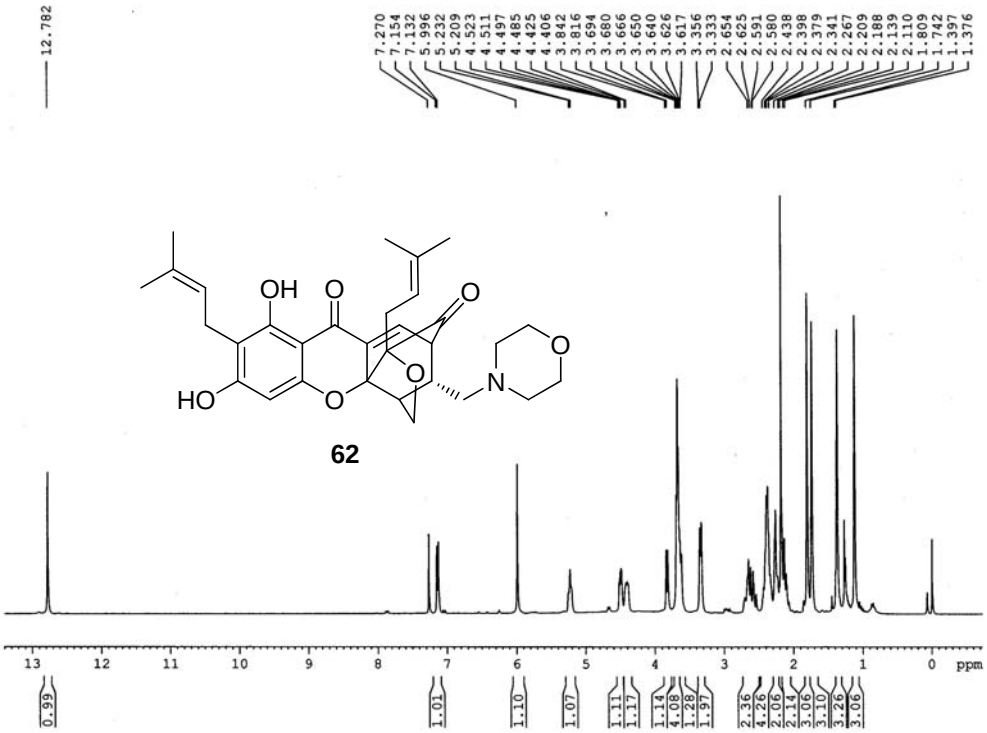

8080115-1
LX091209-1 H1-NMR CDCl3 303K AV-300
12.777
7.267 7.144 7.121 6.650 6.617 5.961 5.549 5.516 4.499 4.487 4.473 4.460 4.443 4.427 4.410 3.857 3.831 3.679 3.656 2.627 2.592 2.580 2.546 2.325 2.297 2.272 2.248 2.196 2.160 1.449 1.381 1.353 1.330 1.253 1.153 0.882
OH O O O N
50
Current Data Parameters
NAME LX091209-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091215
Time 21.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 se
RG 144
DW 80.800 us
DE 7.00 us
TE 300.0 K
D1 1.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.70 us
PL1 -1.00 dB
SFO1 300.1318534 MH
F2 - Processing parameters
SI 32768
SF 300.1300042 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96 0.97 1.00 0.97 1.05 2.07 1.02 1.06 2.17 7.89 1.20 6.42 7.10 0.84 3.18 6.46


8080115-2
LX091209-2 H1-NMR CDCl3 303K AV-300
12.610
7.266 7.166 7.143 6.570 6.536 6.019 5.565 5.550 5.532 4.522 4.511 4.496 4.485 4.422 4.402 3.902 3.876 3.696 3.673 2.697 2.641 2.613 2.579 2.567 2.532 2.370 2.352 2.329 2.302 2.277 2.228 2.190 2.179 2.146 1.771 1.728 1.716 1.622 1.456
OH O O O N
57
Current Data Parameters
NAME LX091209-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091215
Time 21.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 se
RG 144
DW 80.800 us
DE 7.00 us
TE 300.0 K
D1 1.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.70 us
PL1 -1.00 dB
SFO1 300.1318534 MH
F2 - Processing parameters
SI 32768
SF 300.1300047 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.05 1.07 1.00 0.90 1.12 1.06 1.10 1.04 1.16 2.33 8.35 2.42 6.33 7.38 1.28 3.10 6.85


LX091202-1 H1-NMR CDCl3 303K AV-300
52
Current Data Parameters
NAME LX091202-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091208
Time 20.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 se
RG 181
DW 80.800 us
DE 7.00 us
TE 300.0 K
D1 1.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.70 us
PL1 -1.00 dB
SFO1 300.1318534 MH
F2 - Processing parameters
SI 32768
SF 300.1300049 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


LX091202-2 H1-NMR CDCl3 303K AV-300
59
Current Data Parameters
NAME LX091202-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091208
Time 20.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 se
RG 181
DW 80.800 us
DE 7.00 us
TE 300.0 K
D1 1.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.70 us
PL1 -1.00 dB
SFO1 300.1318534 MH
F2 - Processing parameters
SI 32768
SF 300.1300048 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

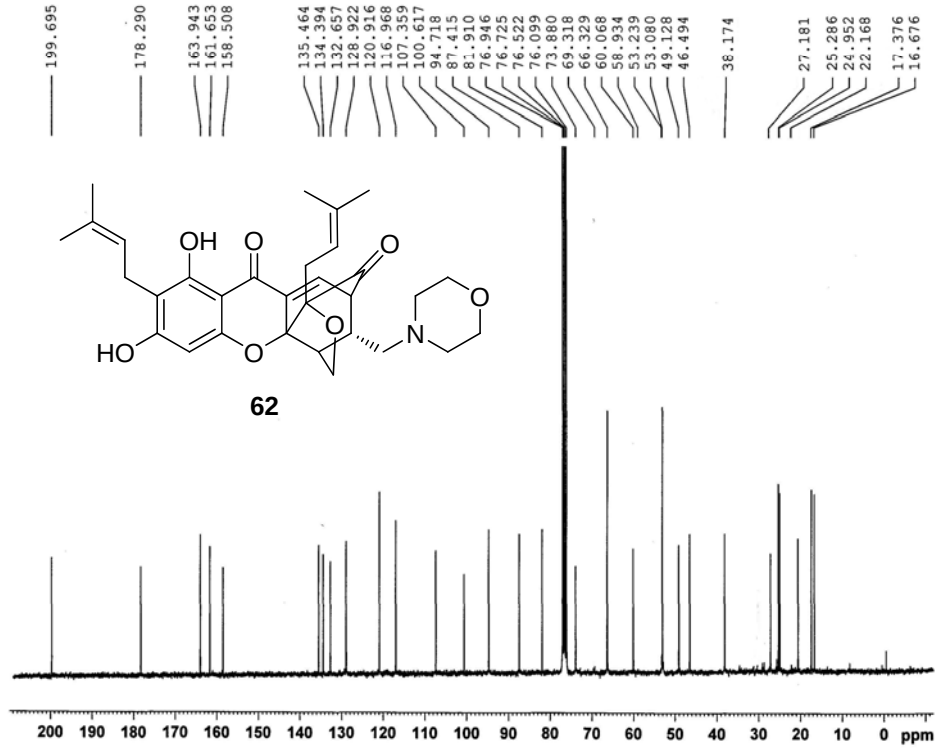
ZXJ8086204-2 H1-NMR CDCL3 303K AV-300



62

```
Current Data Parameters
NAME        ZXJ8086204-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090424
Time               21.28
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                  71.8
DW                 80.800 usec
DE                  7.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.70 usec
PL1                -1.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF            300.1300037 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

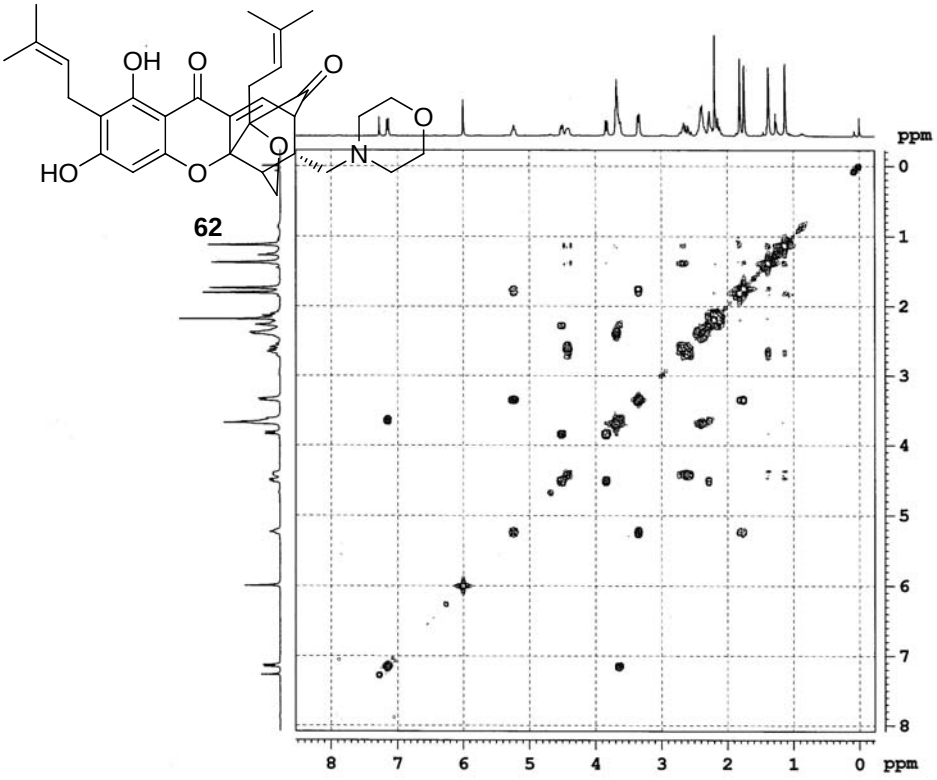
ZXJ8086204-2 C13-NMR CDCl3 303K AV-300



62

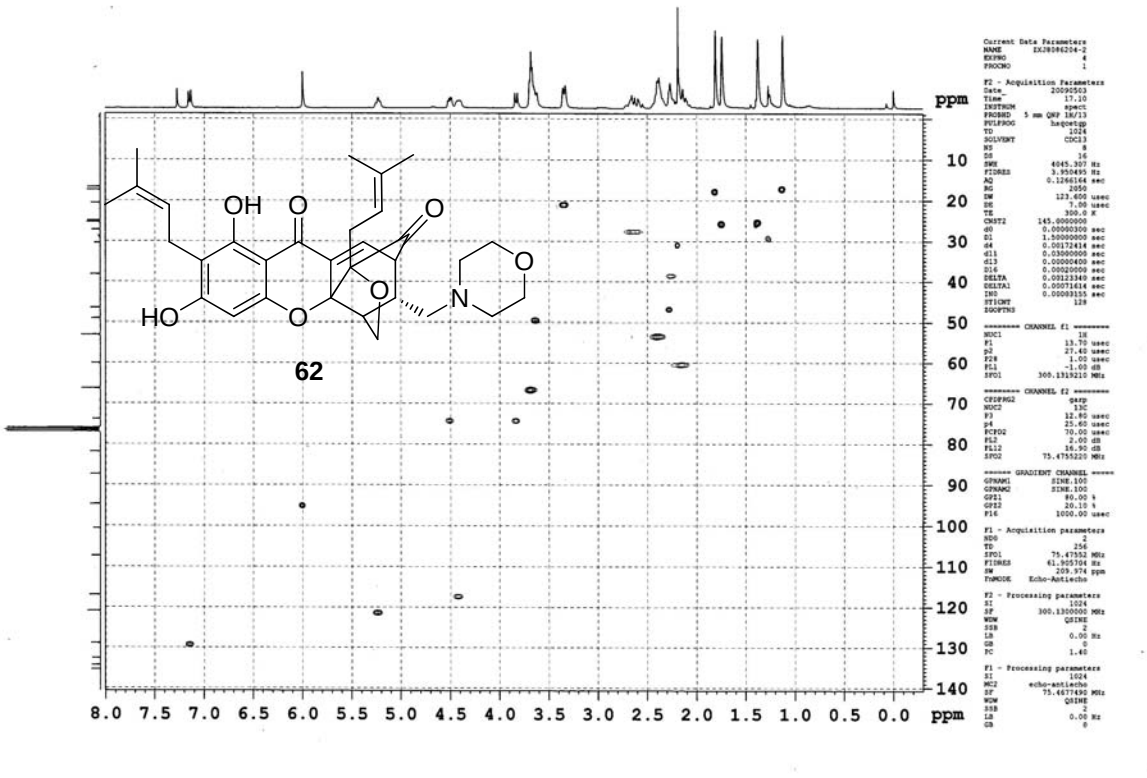
```
Current Data Parameters
NAME        ZXJ8086204-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090502
Time               18.36
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7148
DS                     4
SWH            16666.666 Hz
FIDRES          0.254313 Hz
AQ              1.9661300 sec
RG                   512
DW                 30.000 usec
DE                  7.00 usec
TE                 300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.80 usec
PL1                 1.00 dB
SFO1          75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.30 dB
PL13               14.30 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677867 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

ZXJ8086204-2 H-H COSY CDCl3 303K AV-300



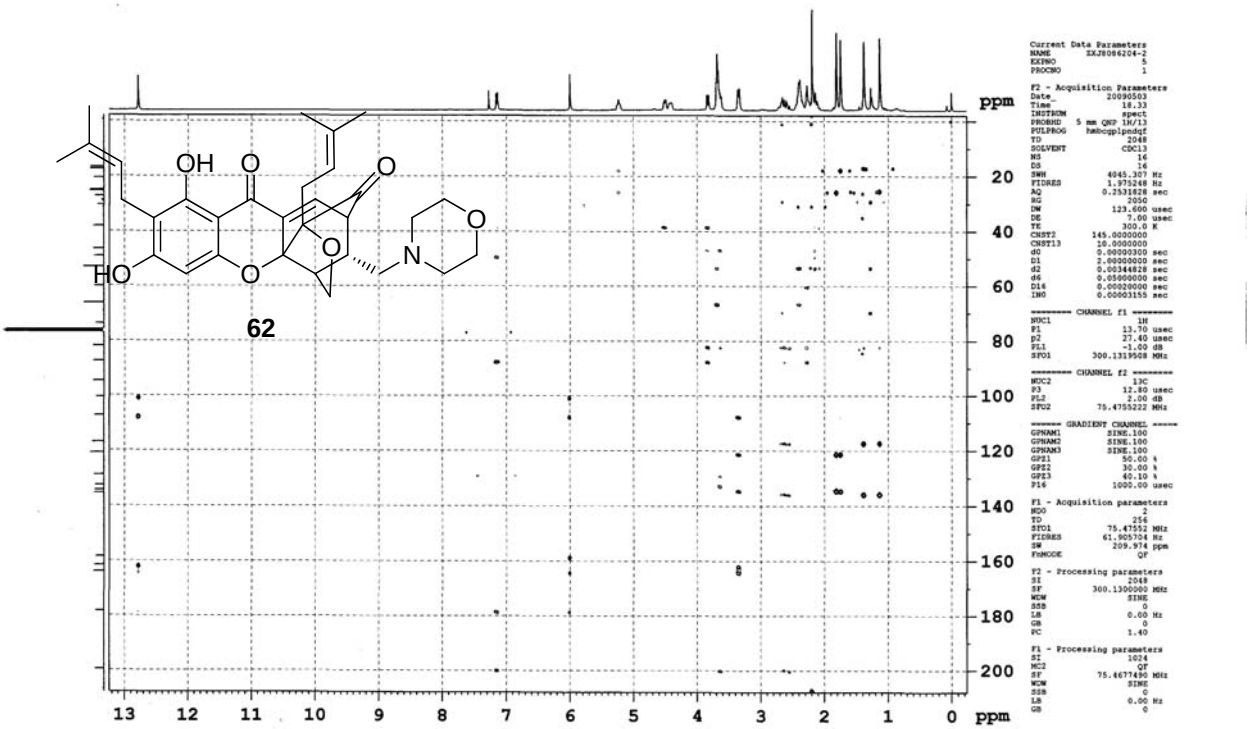

```
Current Data Parameters
NAME                ZXJ8086204-2
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                    20090503
Time                        13.53
INSTRUM                     spect
PROBHD    5 mm QNP 1H/13
PULPROG                 cosygpqf
TD                          2048
SOLVENT                     CDCl3
NS                             2
DS                             8
SWH                  4045.307 Hz
FIDRES               1.975248 Hz
AQ                   0.2531828 sec
RG                           114
DW                     123.600 usec
DE                       7.00 usec
TE                      300.0 K
d0                   0.00000300 sec
D1                   1.48689198 sec
d13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00024720 sec

========== CHANNEL f1 ==========
NUC1                         1H
P0                         13.70 usec
P1                         13.70 usec
PL1                        -1.00 dB
SFO1                300.1319508 MHz

======= GRADIENT CHANNEL ======
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                      10.00 %
GPZ2                      10.00 %
P16                     1000.00 usec

F1 - Acquisition parameters
ND0                            1
TD                           256
SFO1                 300.132 MHz
FIDRES              15.801982 Hz
SW                       13.478 ppm
FnMODE                       QF

F2 - Processing parameters
SI                          1024
SF                   300.1300000 MHz
WDW                        SINE
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                          1024
MC2                           QF
SF                   300.1300000 MHz
WDW                        SINE
SSB                           0
LB                         0.00 Hz
GB                            0
```

ZXJ8086204-2 HSQC CDCl3 303K AV-300




```
Current Data Parameters
NAME                ZXJ8086204-2
EXPNO                          4
PROCNO                         1

F2 - Acquisition Parameters
Date_                    20090503
Time                        17.10
INSTRUM                     spect
PROBHD    5 mm QNP 1H/13
PULPROG                 hsqcetgp
TD                          1024
SOLVENT                     CDCl3
NS                             8
DS                            16
SWH                  4045.307 Hz
FIDRES               3.950495 Hz
AQ                   0.1266164 sec
RG                          2050
DW                     123.600 usec
DE                       7.00 usec
TE                      300.0 K
CNST2               145.0000000
d0                   0.00000300 sec
D1                   1.50000000 sec
d4                   0.00172414 sec
d11                  0.03000000 sec
d13                  0.00000400 sec
D16                  0.00020000 sec
DELTA                0.00123340 sec
DELTA1               0.00071614 sec
IN0                  0.00008315 sec
TDI                         128
TDIGHT
ZGOPTNS

========== CHANNEL f1 ==========
NUC1                         1H
P1                         13.70 usec
p2                         27.40 usec
P28                         1.00 usec
PL1                        -1.00 dB
SFO1                300.1319210 MHz

========== CHANNEL f2 ==========
CPDPRG2                    garp
NUC2                        13C
P3                         12.80 usec
p4                         25.60 usec
PCPD2                      70.00 usec
PL2                         2.00 dB
PL12                       16.90 dB
SFO2                75.4755220 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                      80.00 %
GPZ2                      20.10 %
P16                     1000.00 usec

F1 - Acquisition parameters
ND0                            2
TD                           256
SFO1                75.4775552 MHz
FIDRES              61.905704 Hz
SW                       209.974 ppm
FnMODE       Echo-Antiecho

F2 - Processing parameters
SI                          1024
SF                   300.1300000 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                          1024
MC2                 echo-antiecho
SF                   75.4677490 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
```

ZXJ8086204-2 HMBC CDCl3 303K AV-300



Current Data Parameters
NAME ZXJ8086204-2
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090503
Time 18.33
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmbcgplpndqf
TD 2048
SOLVENT CDCl3
NS 16
DS 16
SWH 4045.307 Hz
FIDRES 1.975248 Hz
AQ 0.2531828 sec
RG 2050
DW 123.600 usec
DE 7.00 usec
TE 300.0 K
CNST2 145.0000000
CNST13 10.0000000
d0 0.00003009 sec
D1 2.00000000 sec
d2 0.00344828 sec
d6 0.05000000 sec
D16 0.00020000 sec
IN0 0.00003155 sec

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
p2 27.40 usec
PL1 -1.00 dB
SFO1 300.1319508 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 12.80 usec
PL2 2.00 dB
SFO2 75.4755222 MHz

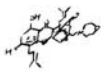
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

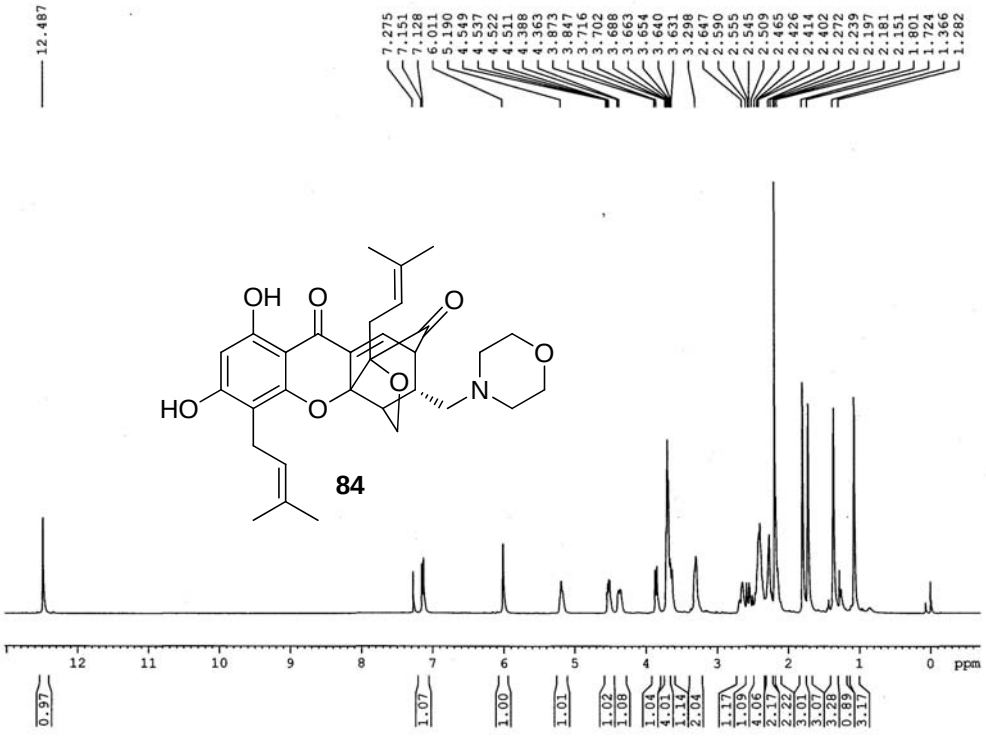
F1 - Acquisition parameters
ND0 2
TD 256
SFO1 75.47552 MHz
FIDRES 61.905704 Hz
SW 209.974 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 75.4677490 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

ZXJ8086204-1 H1-NMR CDCL3 303K AV-300





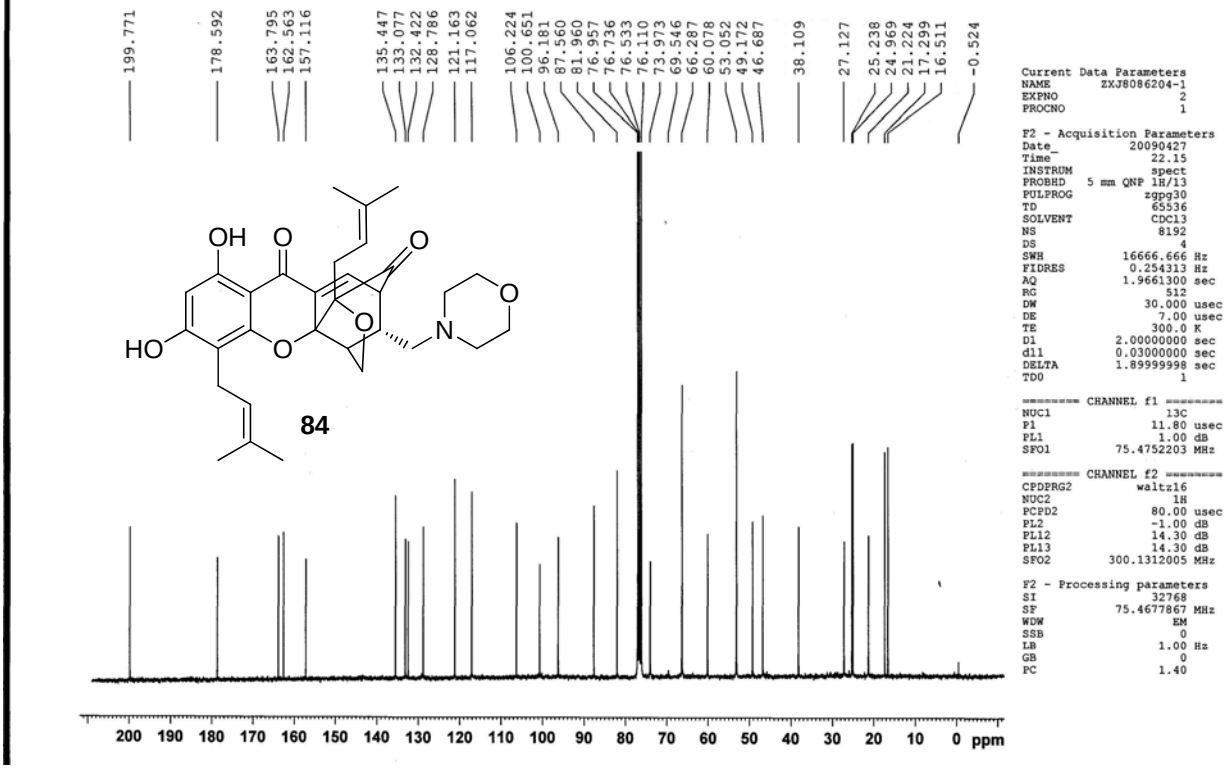
Current Data Parameters
NAME ZXJ8086204-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090424
Time 21.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 71.8
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

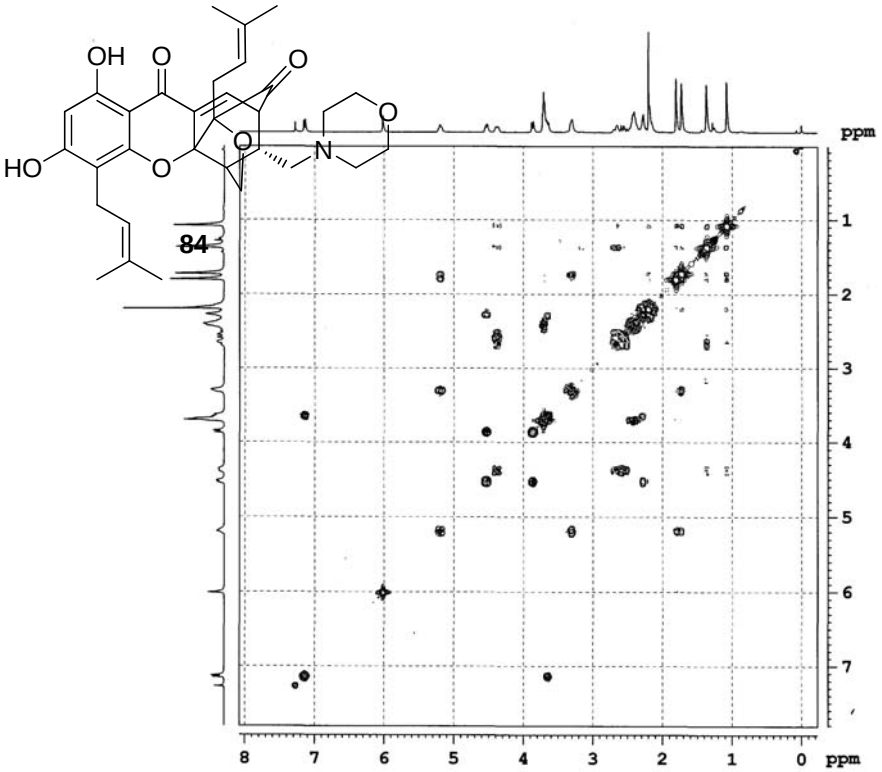
F2 - Processing parameters
SI 32768
SF 300.1300024 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ZXJ8086204-1 C13-NMR CDCl3 303K AV-300



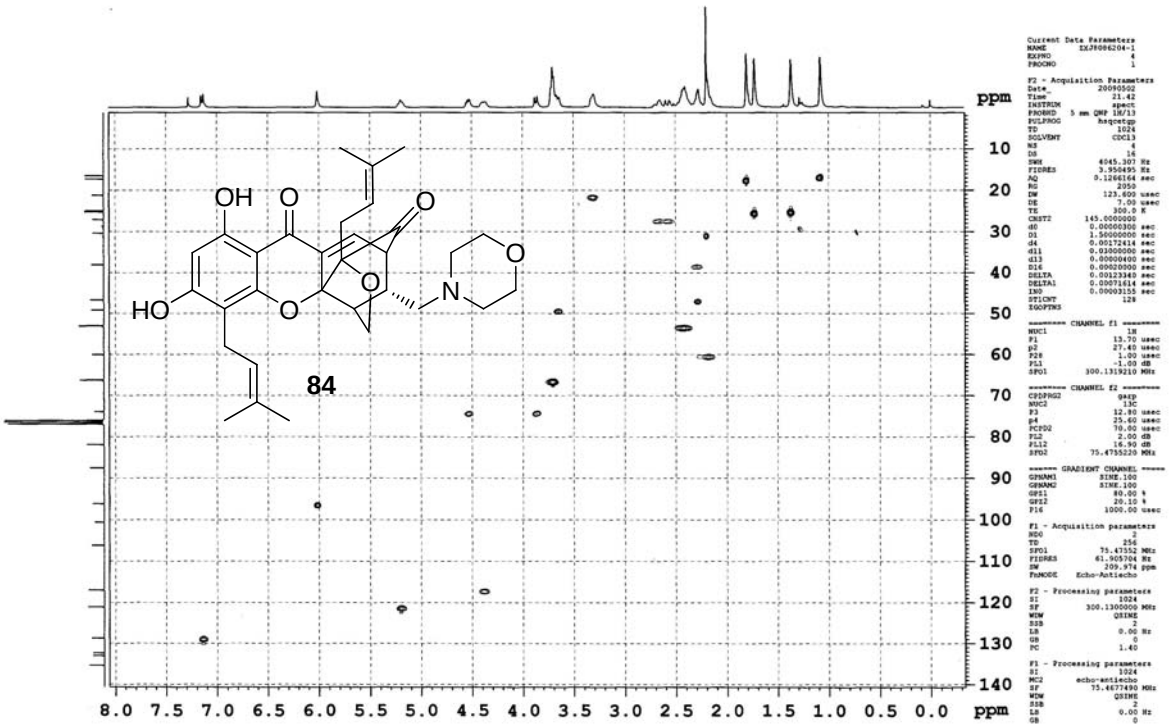

```
Current Data Parameters
NAME          ZXJ8086204-1
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090427
Time                 22.15
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   8192
DS                      4
SWH            16666.666 Hz
FIDRES          0.254313 Hz
AQ              1.9661300 sec
RG                    512
DW                30.000 usec
DE                 7.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.80 usec
PL1                  1.00 dB
SFO1           75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                14.30 dB
PL13                14.30 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677867 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

ZXJ8086204-1 H-H COSY CDCl3 303K AV-300

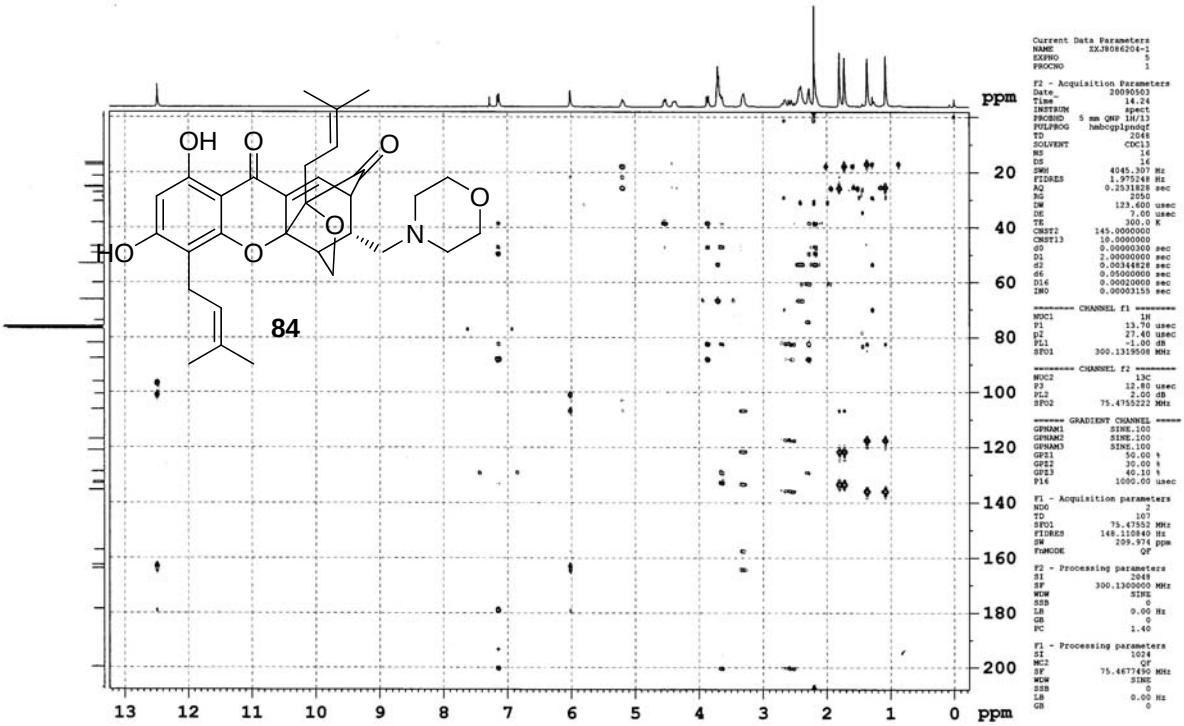



```
Current Data Parameters
NAME          ZXJ8086204-1
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090502
Time                 21.02
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG          cosygpqf
TD                    2048
SOLVENT             CDCl3
NS                       2
DS                       8
SWH             4045.307 Hz
FIDRES          1.975248 Hz
AQ              0.2531828 sec
RG                     114
DW                123.600 usec
DE                 7.00 usec
TE                300.0 K
d0             0.00000300 sec
D1             1.48689198 sec
d13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00024720 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                  13.70 usec
P1                  13.70 usec
PL1                 -1.00 dB
SFO1          300.1319508 MHz

====== GRADIENT CHANNEL =====
GPNAM1            SINE.100
GPNAM2            SINE.100
GPZ1                10.00 %
GPZ2                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
NDO                      1
TD                     256
SFO1           300.132 MHz
FIDRES         15.801982 Hz
SW                 13.478 ppm
FnMODE                  QF

F2 - Processing parameters
SI                    1024
SF           300.1300000 MHz
WDW                  SINE
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF           300.1300000 MHz
WDW                  SINE
SSB                     0
LB                   0.00 Hz
GB                      0
```

ZXJ8086204-1 HSQC CDCl3 303K AV-300



ZXJ8086204-1 HMBC CDCl3 303K AV-300



LX090609-2 H1-NMR CDCl3 303K AV-300

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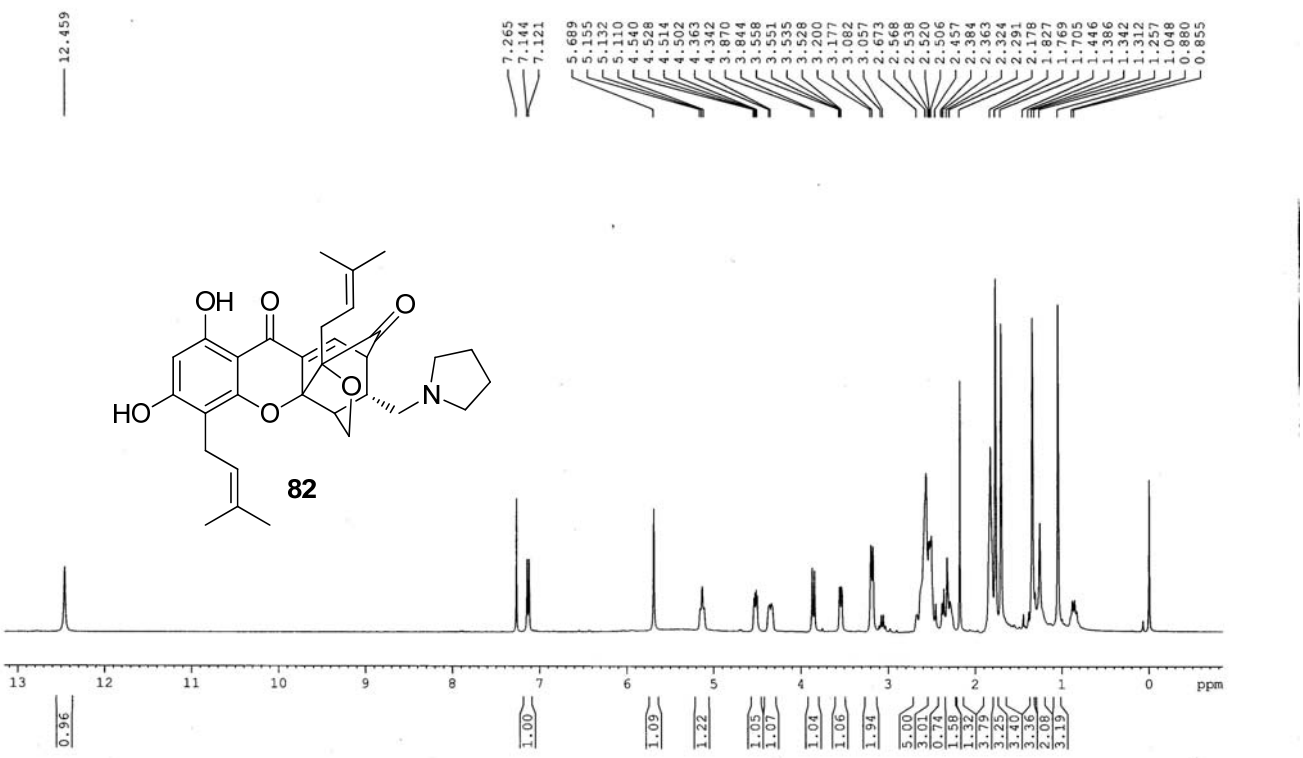
LX090609-1 H1-NMR CDCl3 303K AV-300



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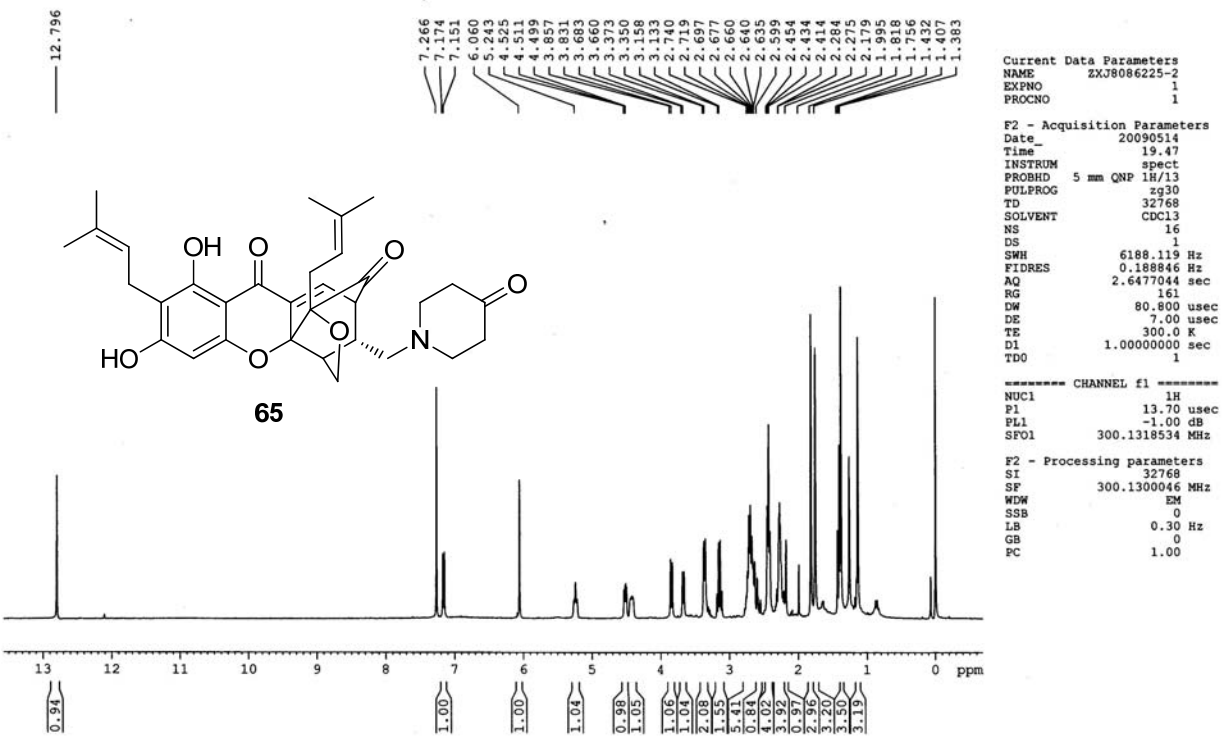
ZXJ8086225-2 H1-NMR CDCl3 303K AV-300



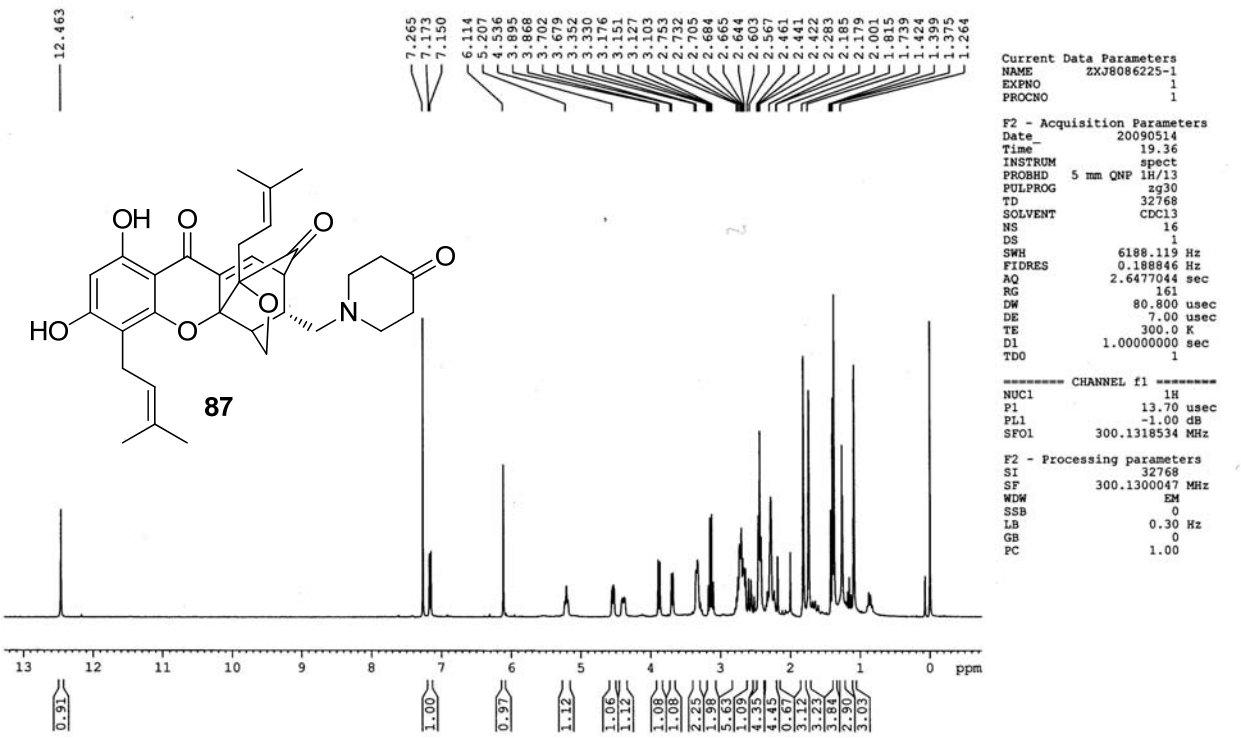
Current Data Parameters
NAME ZXJ8086225-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090514
Time 19.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 161
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300046 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ZXJ8086225-1 H1-NMR CDCl3 303K AV-300



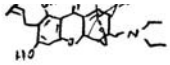
Current Data Parameters
NAME ZXJ8086225-1
EXPNO 1
PROCNO 1

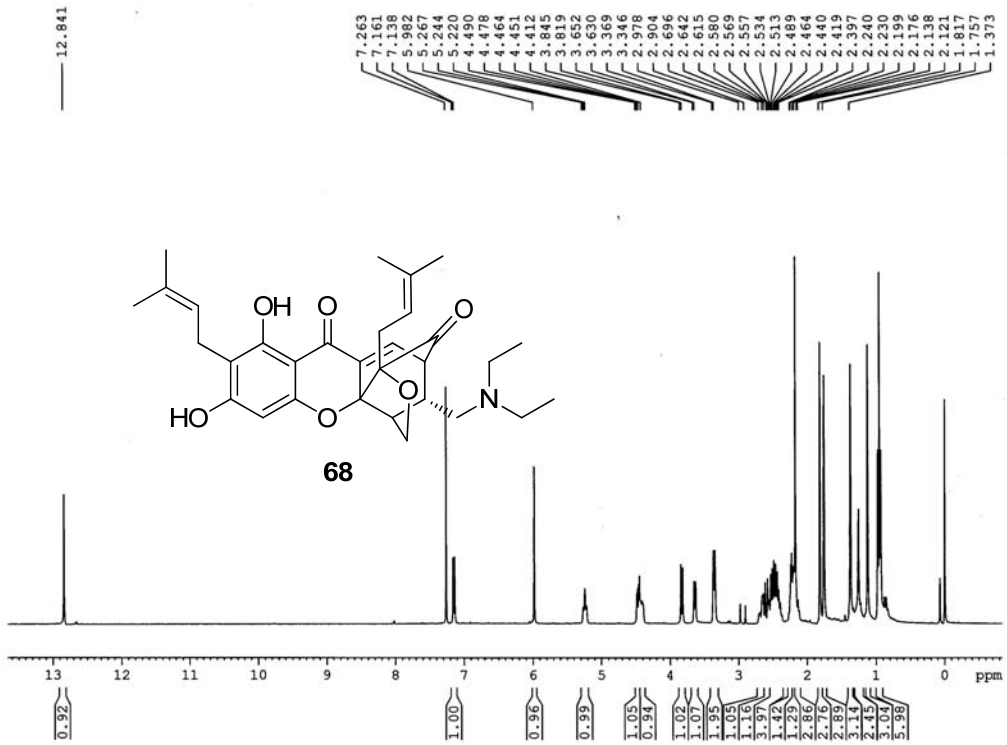
F2 - Acquisition Parameters
Date_ 20090514
Time 19.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 161
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LX8015903-2 H1-NMR CDCl3 303K ARV-300





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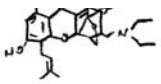
Current Data Parameters
NAME LX8015903-2
EXPNO 1
PROCNO 1

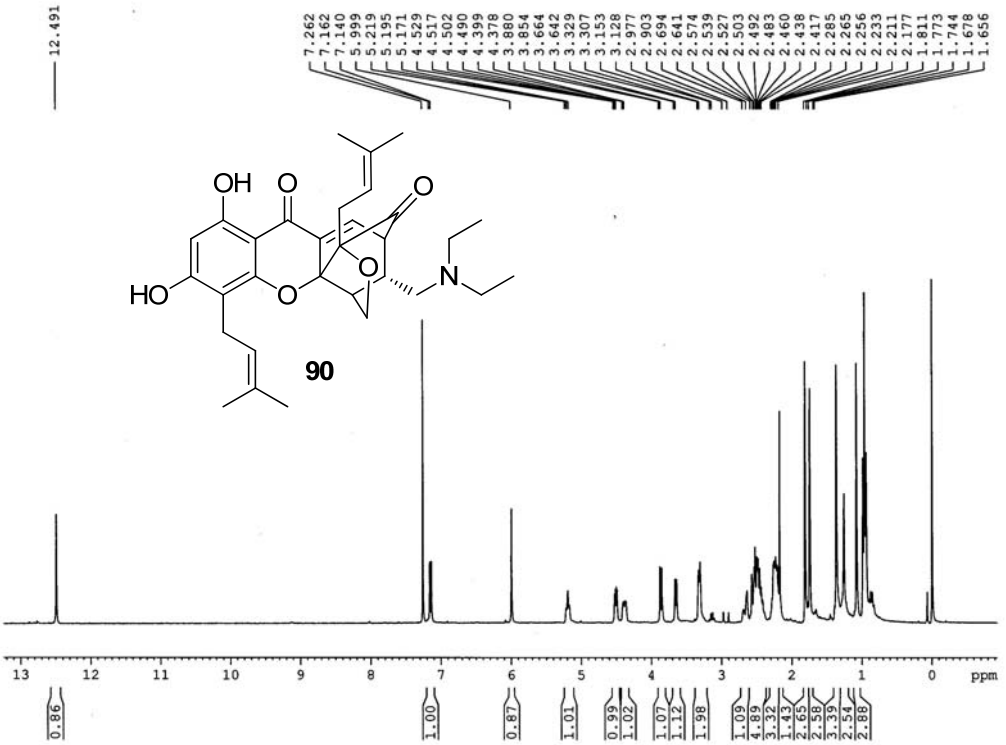
F2 - Acquisition Parameters
Date_ 20090507
Time 22.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 144
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300053 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LX8015903-1 H1-NMR CDCl3 303K ARV-300





90

Current Data Parameters
NAME LX8015903-1
EXPNO 1
PROCNO 1

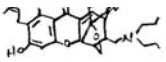
F2 - Acquisition Parameters
Date_ 20090507
Time 21.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

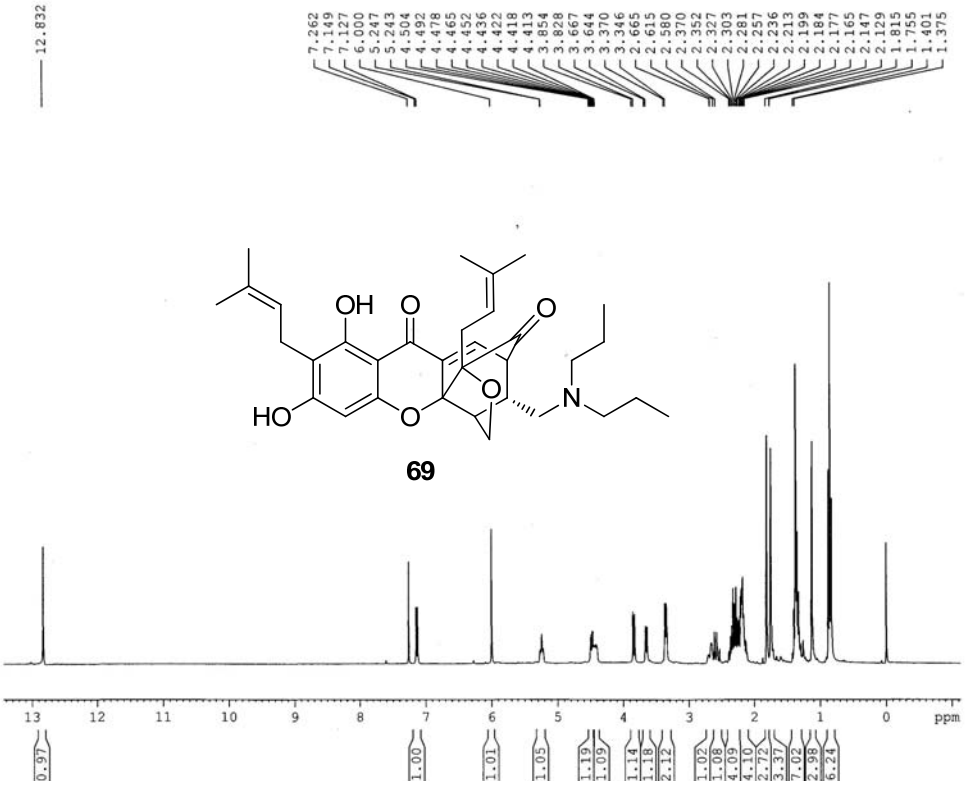
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LX090710-2 H1-NMR CDCL3 303K AV-300

801988-2



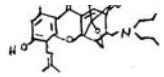


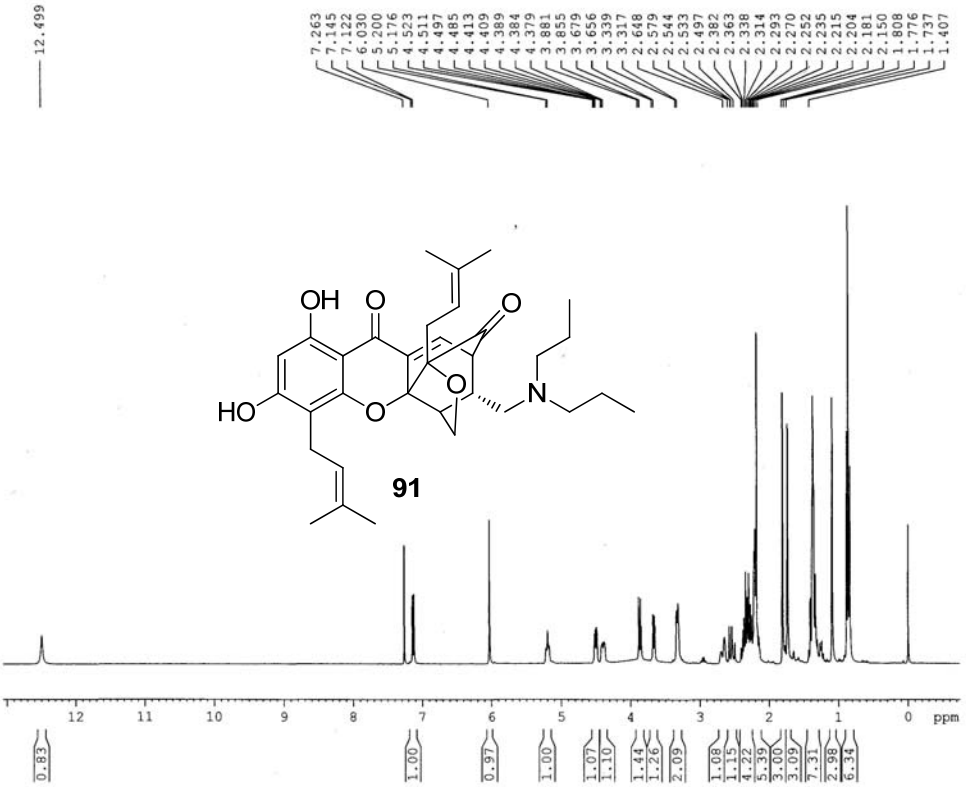
69


```
Current Data Parameters
NAME            LX090710-2
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameter
Date_             20090714
Time                 19.58
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 se
RG                    144
DW                 80.800 us
DE                  7.00 us
TE                 300.0 K
D1            1.00000000 se
TD0                     1

======== CHANNEL f1 ======
NUC1                   1H
P1                 13.70 us
PL1                -1.00 dB
SFO1         300.1318534 MH

F2 - Processing parameters
SI                  32768
SF           300.1300056 MH
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

LX090710-1 H1-NMR CDCL3 303K AV-300

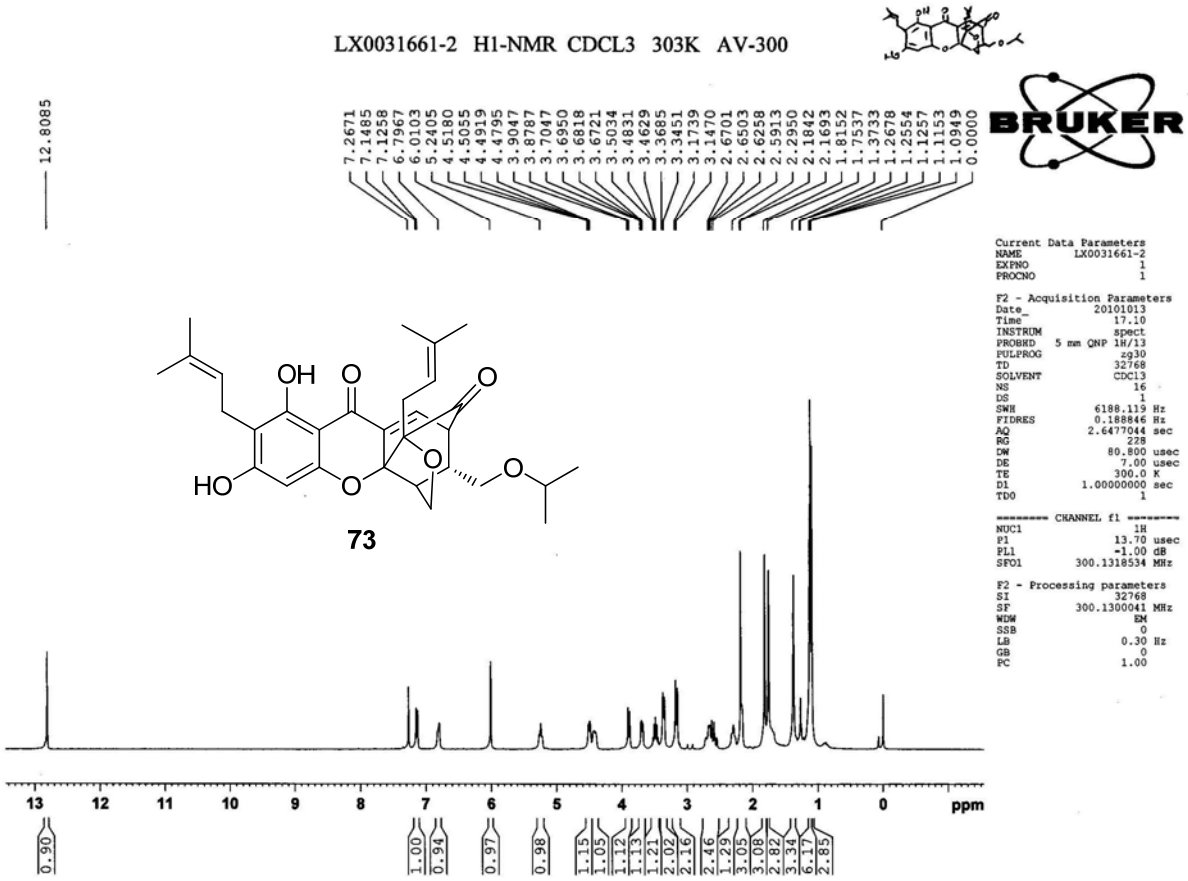
801988-1

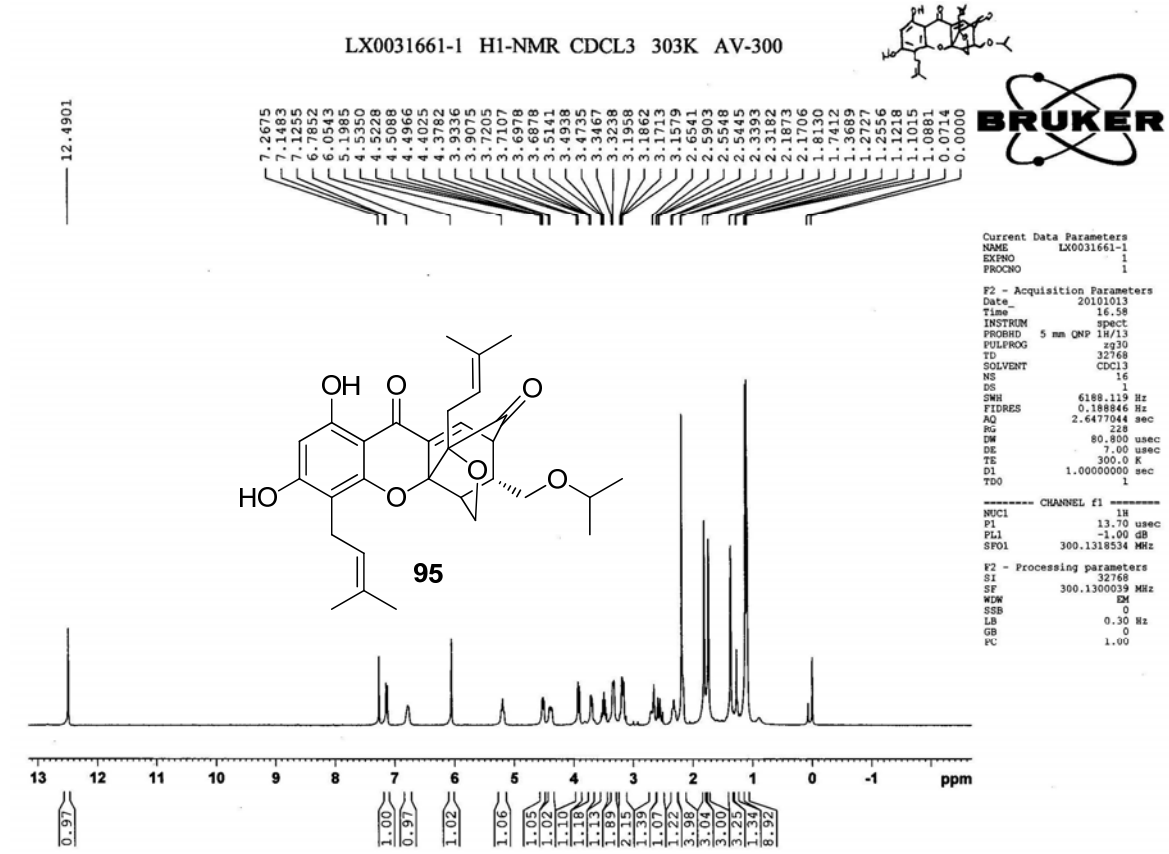


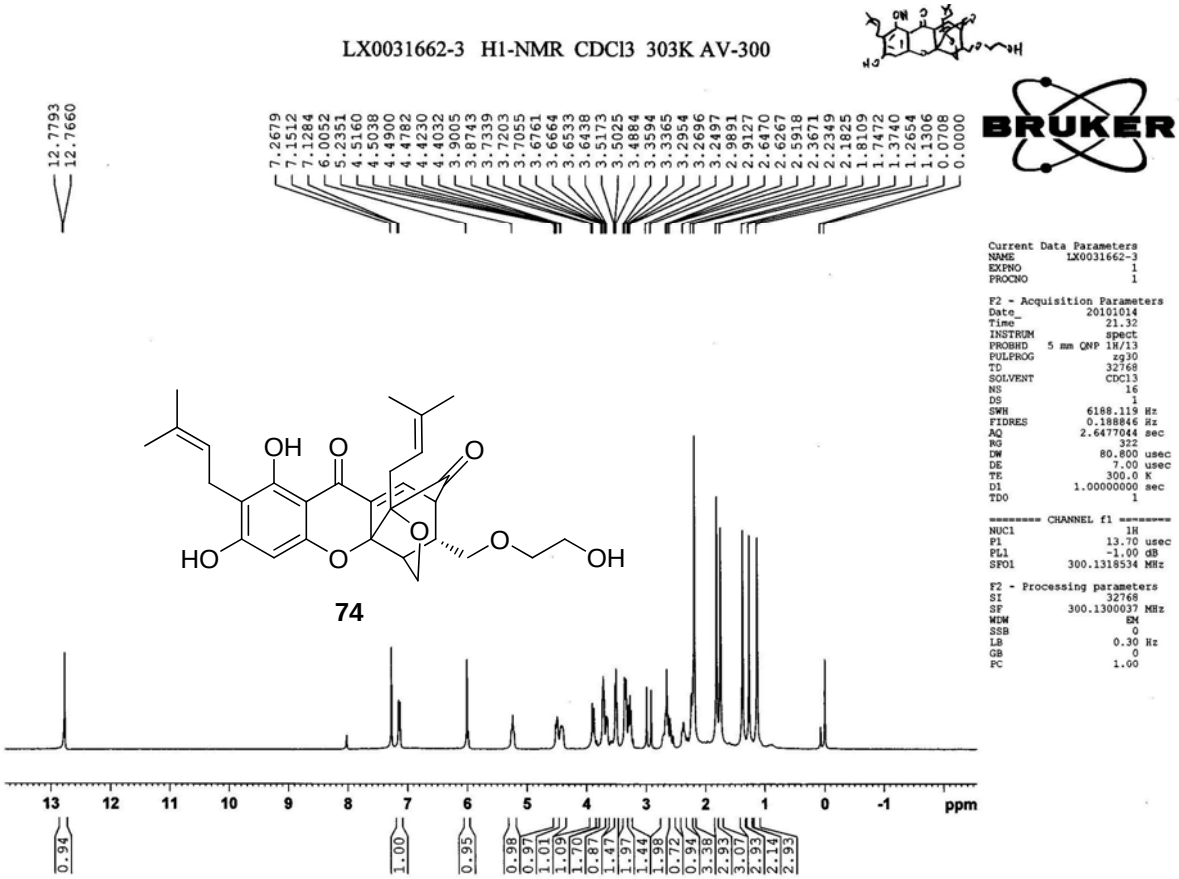


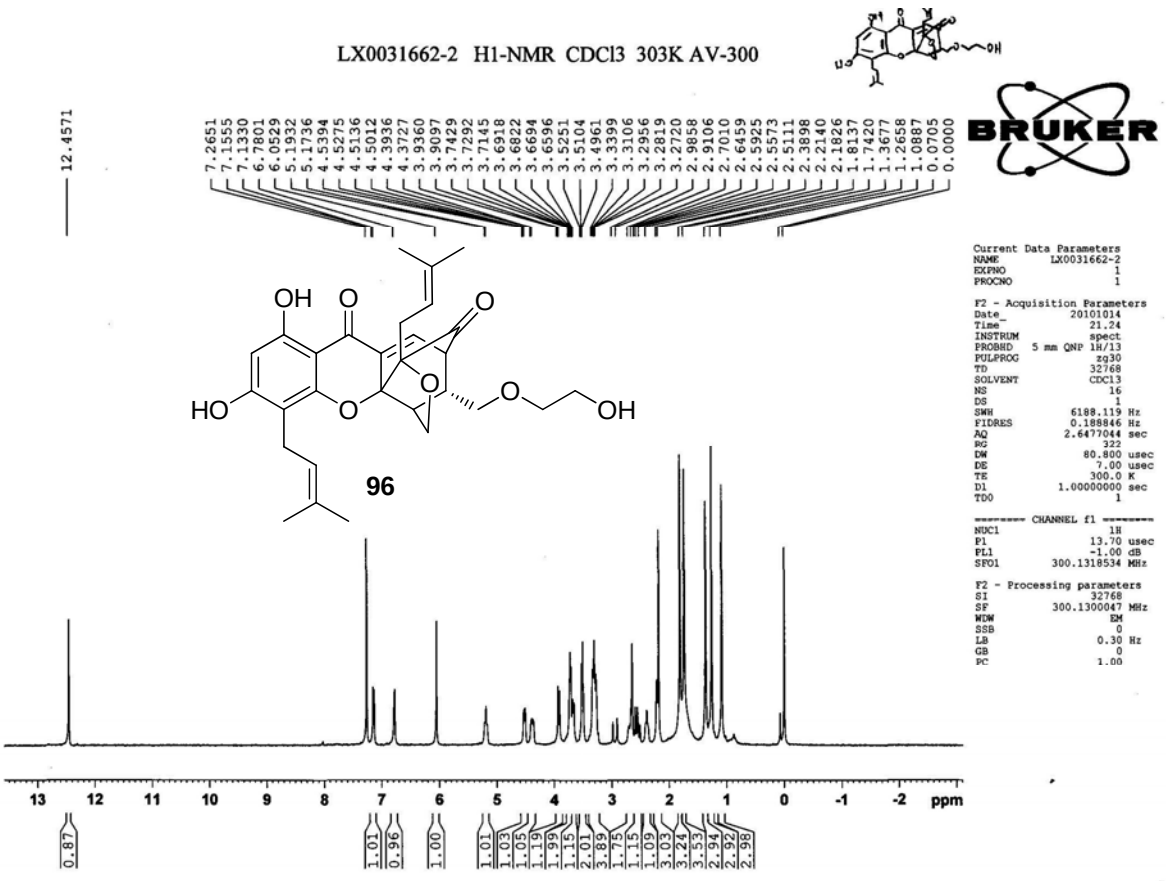
91

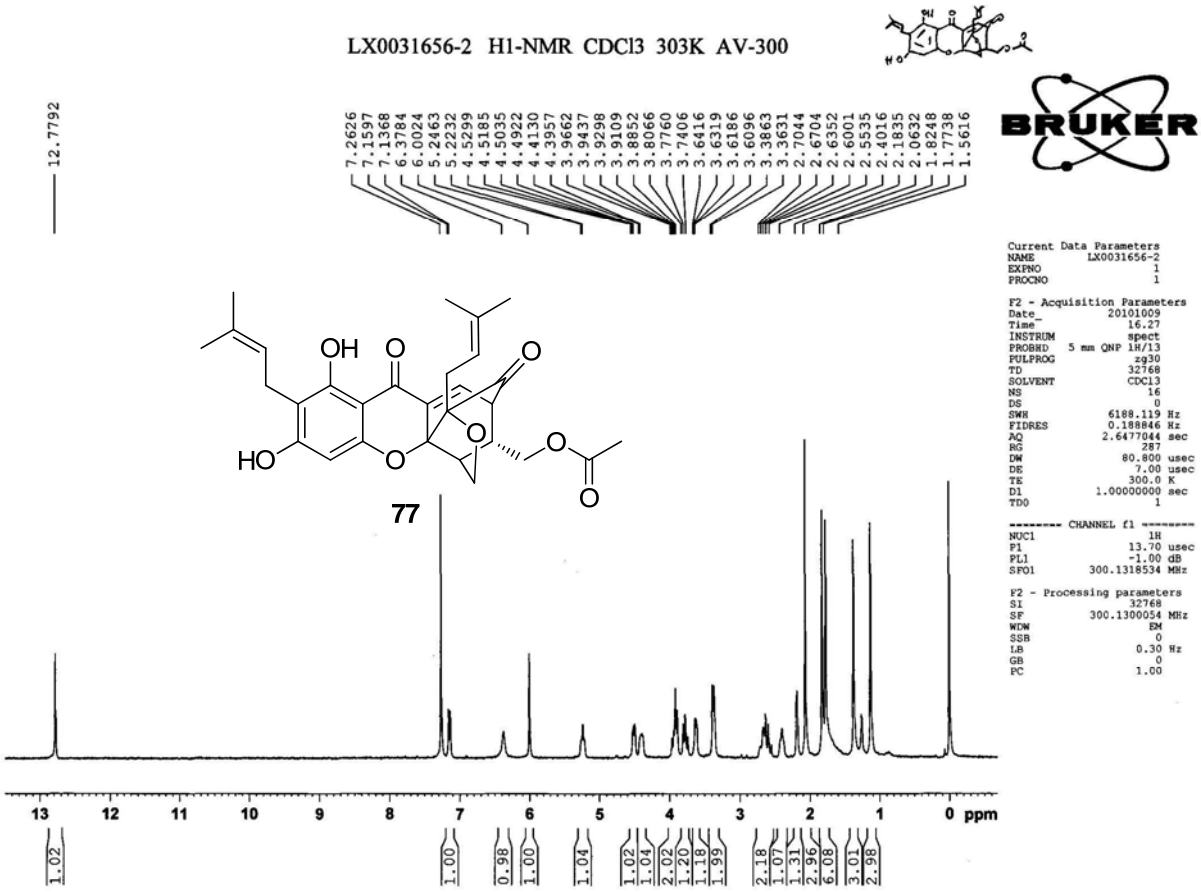

```
Current Data Parameters
NAME            LX090710-1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameter
Date_             20090714
Time                 19.51
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 se
RG                    144
DW                 80.800 us
DE                  7.00 us
TE                 300.0 K
D1            1.00000000 se
TD0                     1

======== CHANNEL f1 ======
NUC1                   1H
P1                 13.70 us
PL1                -1.00 dB
SFO1         300.1318534 MH

F2 - Processing parameters
SI                  32768
SF           300.1300052 MH
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

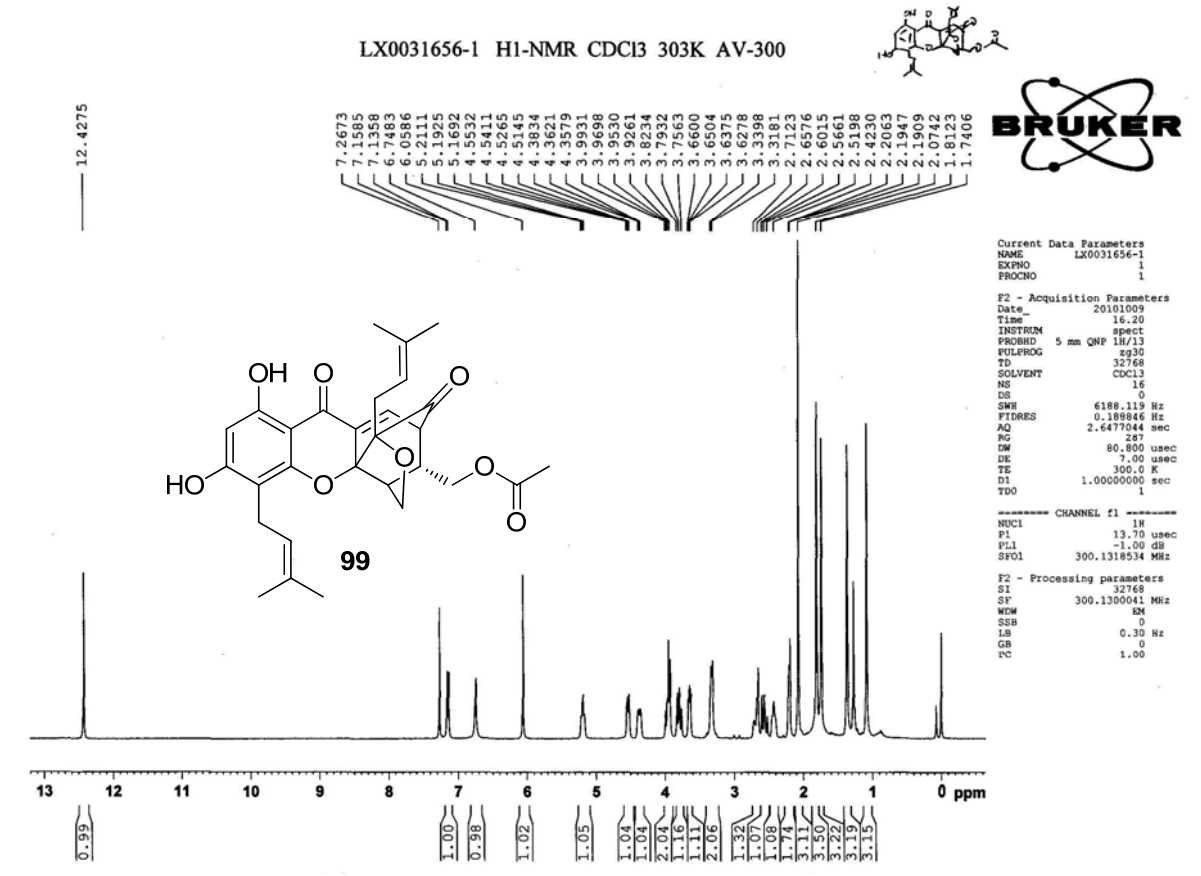

LX0031661-2 H1-NMR CDCL3 303K AV-300
BRUKER
12.8085
7.2671
7.1485
7.1258
6.7967
6.0103
5.2405
4.5180
4.5055
4.4919
4.4795
3.9047
3.8787
3.7047
3.6950
3.6818
3.6721
3.5034
3.4831
3.4629
3.3685
3.3451
3.1739
3.1470
2.6701
2.6503
2.6258
2.5913
2.2950
2.1842
2.1693
1.8152
1.7537
1.3733
1.2678
1.2554
1.1257
1.1153
1.0949
0.0000
Current Data Parameters
NAME LX0031661-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101013
Time 17.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
O
O
HO
O
O
O
73
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.90 1.00 0.94 0.97 0.98 1.15 1.05 1.12 1.13 1.21 2.02 2.16 2.46 1.29 3.05 3.08 2.82 3.34 6.17 2.85

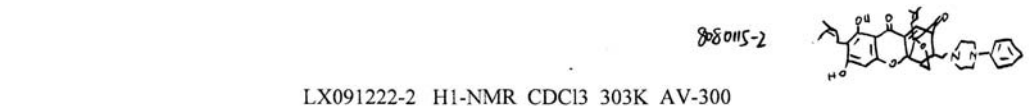

LX0031661-1 H1-NMR CDCL3 303K AV-300
BRUKER
12.4901
7.2675
7.1483
7.1255
6.7852
6.0543
5.1985
4.5350
4.5228
4.5088
4.4966
4.4025
4.3782
3.9336
3.9075
3.7205
3.7107
3.6978
3.6878
3.5141
3.4938
3.4735
3.3467
3.3238
3.1958
3.1862
3.1713
3.1579
2.6541
2.5903
2.5548
2.5445
2.3393
2.3182
2.1873
2.1706
1.8130
1.7412
1.3689
1.2727
1.2556
1.1218
1.1015
1.0881
0.0714
0.0000
Current Data Parameters
NAME LX0031661-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101013
Time 16.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
O
O
HO
O
O
O
95
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.97 1.00 0.97 1.02 1.06 1.05 1.02 1.10 1.18 1.13 1.89 2.15 1.39 1.07 1.22 3.98 3.04 3.00 3.25 1.34 8.92


LX0031662-3 H1-NMR CDCl3 303K AV-300
BRUKER
74
Current Data Parameters
NAME LX0031662-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101014
Time 21.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 322
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300037 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

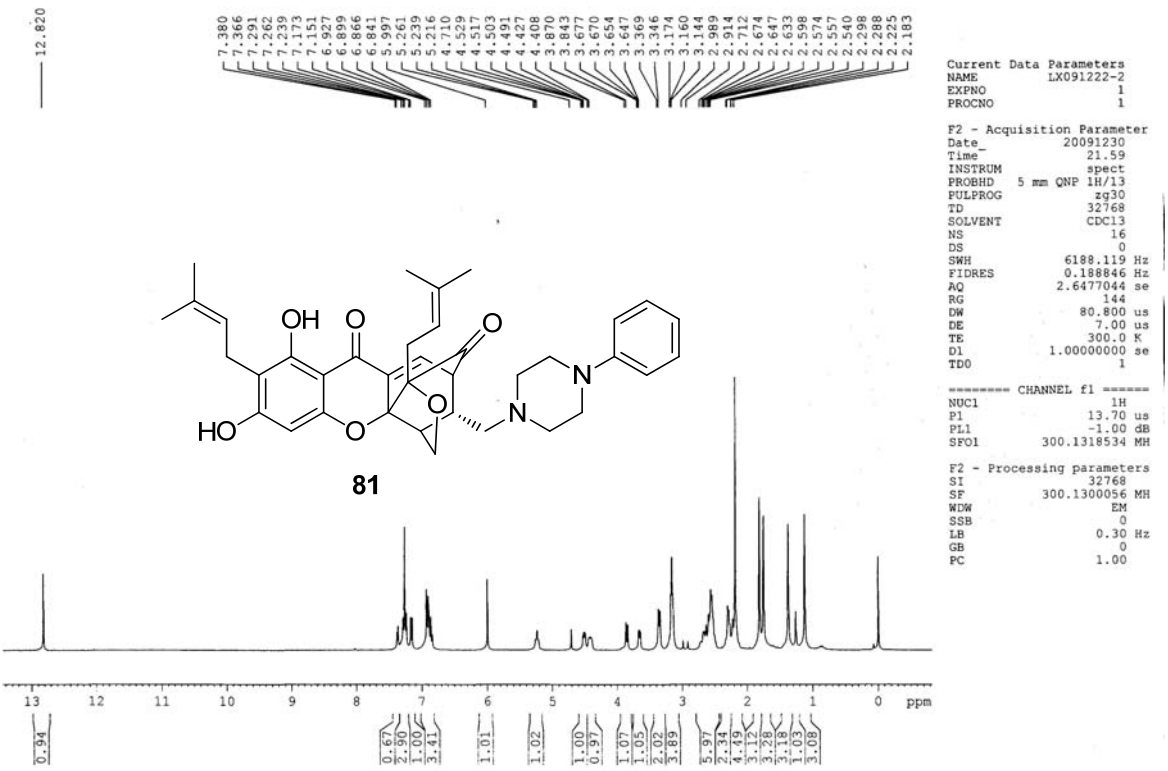

LX0031662-2 H1-NMR CDCl3 303K AV-300
BRUKER
96
Current Data Parameters
NAME LX0031662-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101014
Time 21.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 322
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


LX0031656-2 H1-NMR CDCl3 303K AV-300
BRUKER
77
Current Data Parameters
NAME LX0031656-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101009
Time 16.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 287
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

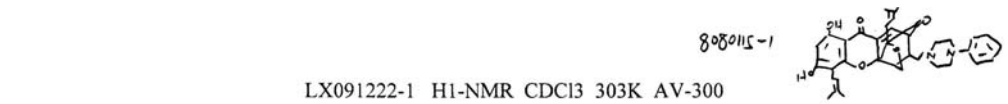

LX0031656-1 H1-NMR CDCl3 303K AV-300
BRUKER
99
Current Data Parameters
NAME LX0031656-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101009
Time 16.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 287
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm



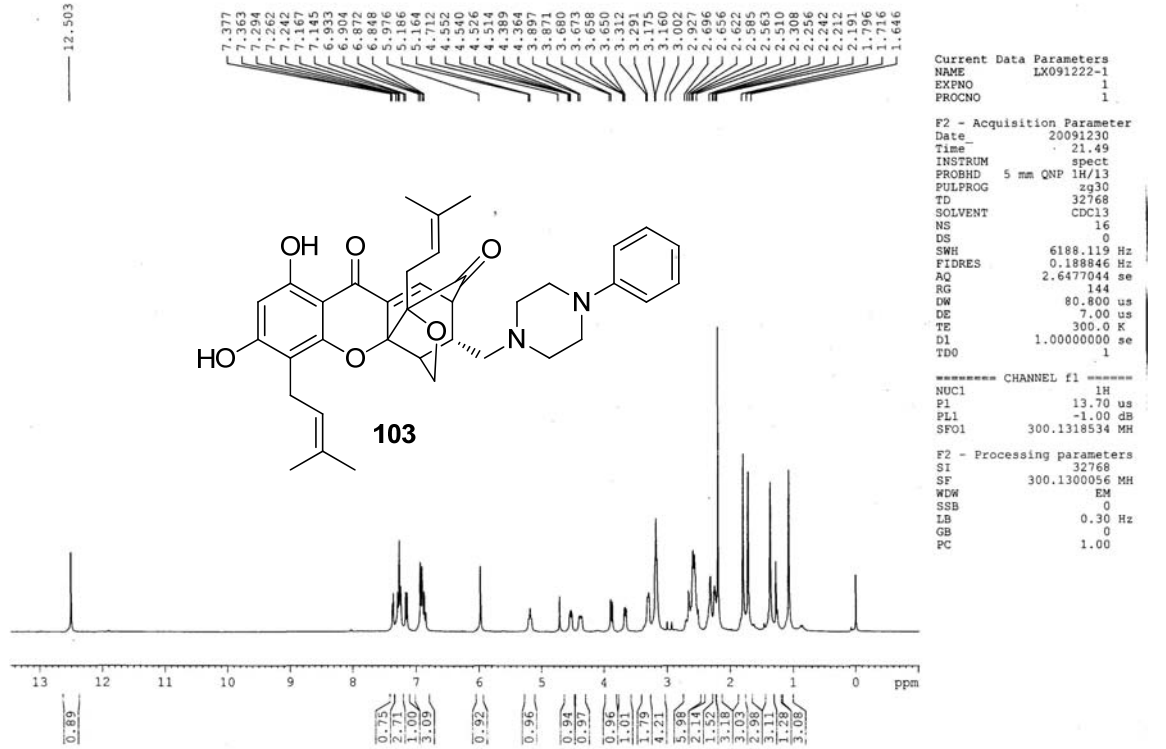
LX091222-2 H1-NMR CDCl3 303K AV-300



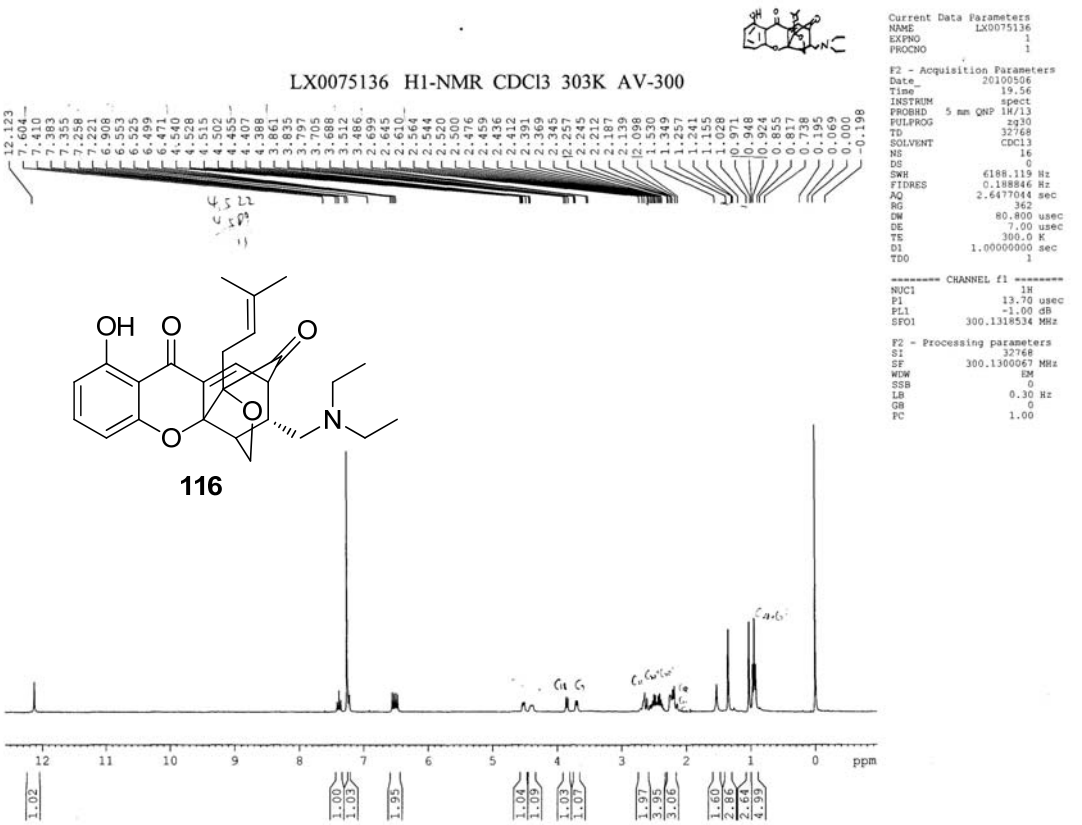

```
Current Data Parameters
NAME            LX091222-2
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameter
Date_             20091230
Time                 21.59
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 se
RG                    144
DW                80.800 us
DE                 7.00 us
TE                300.0 K
D1           1.00000000 se
TD0                     1

======== CHANNEL f1 ======
NUC1                   1H
P1                 13.70 us
PL1                -1.00 dB
SFO1         300.1318534 MH

F2 - Processing parameters
SI                  32768
SF           300.1300056 MH
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

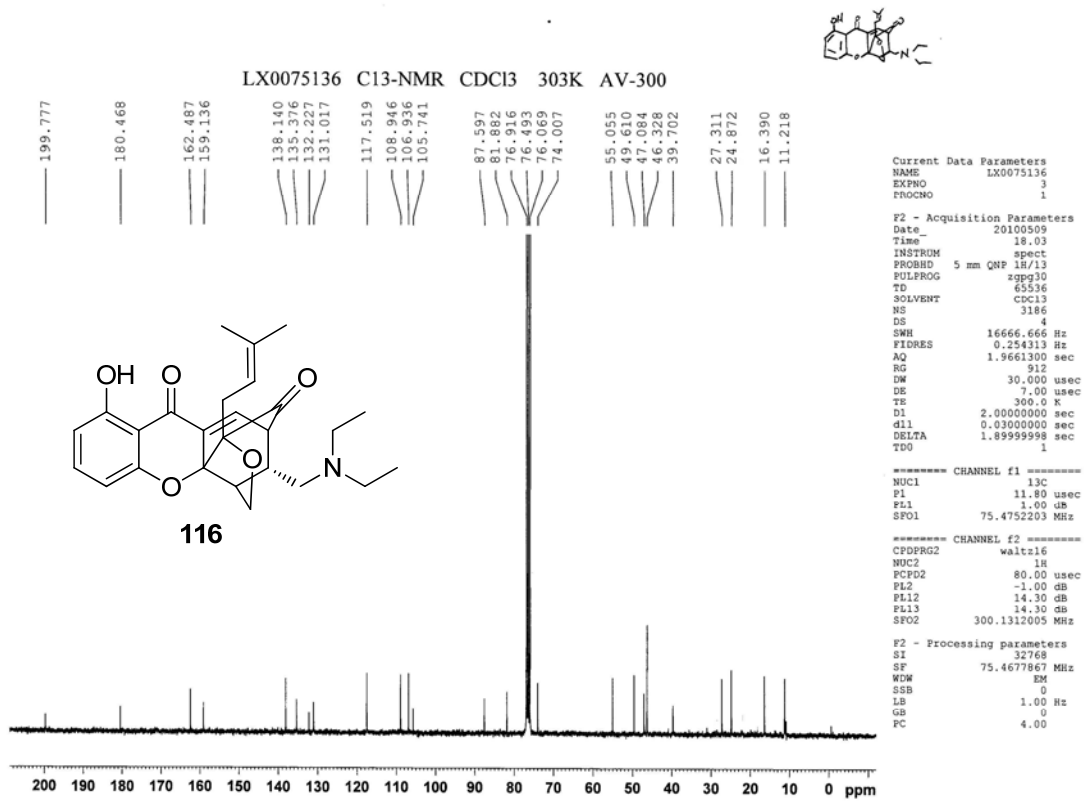


LX091222-1 H1-NMR CDCl3 303K AV-300

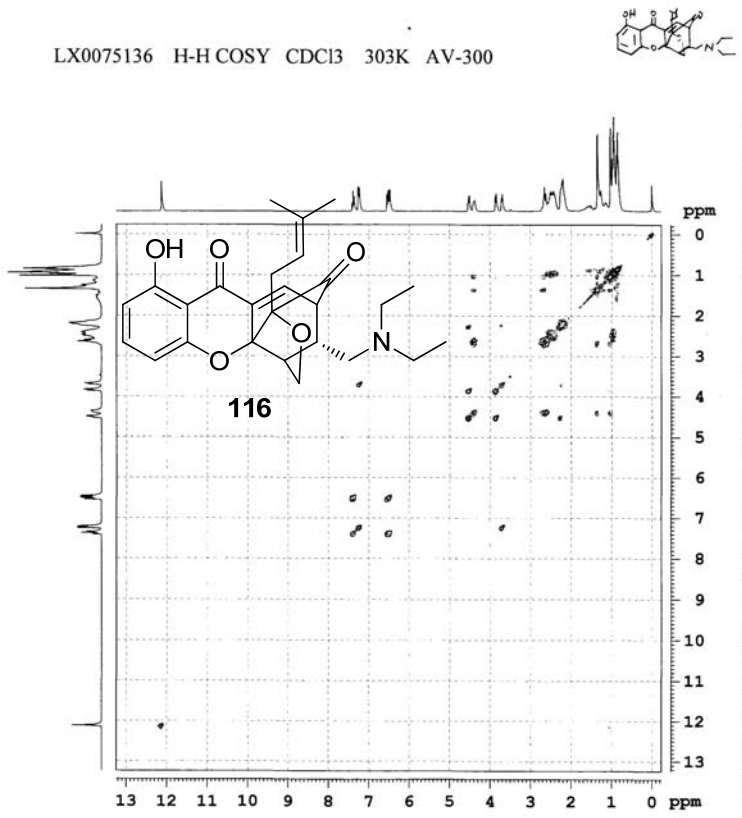



```
Current Data Parameters
NAME            LX091222-1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameter
Date_             20091230
Time                 21.49
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 se
RG                    144
DW                80.800 us
DE                 7.00 us
TE                300.0 K
D1           1.00000000 se
TD0                     1

======== CHANNEL f1 ======
NUC1                   1H
P1                 13.70 us
PL1                -1.00 dB
SFO1         300.1318534 MH

F2 - Processing parameters
SI                  32768
SF           300.1300056 MH
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```


LX0075136 H1-NMR CDCl3 303K AV-300
116
Current Data Parameters
NAME LX0075136
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100506
Time 19.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 362
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

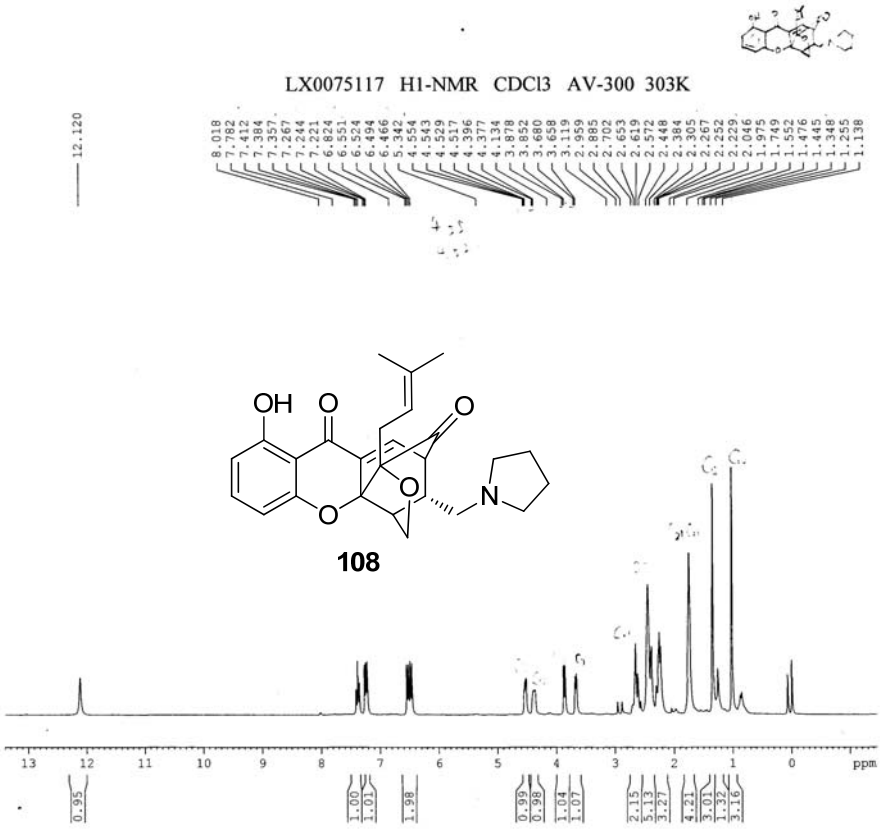

LX0075136 C13-NMR CDCl3 303K AV-300
116
Current Data Parameters
NAME LX0075136
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100509
Time 18.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3186
DS 4
SWH 16666.666 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 912
DW 30.000 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 1.00 dB
SFO1 75.4752203 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.30 dB
PL13 14.30 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00

LX0075136 H-H COSY CDCl3 303K AV-300

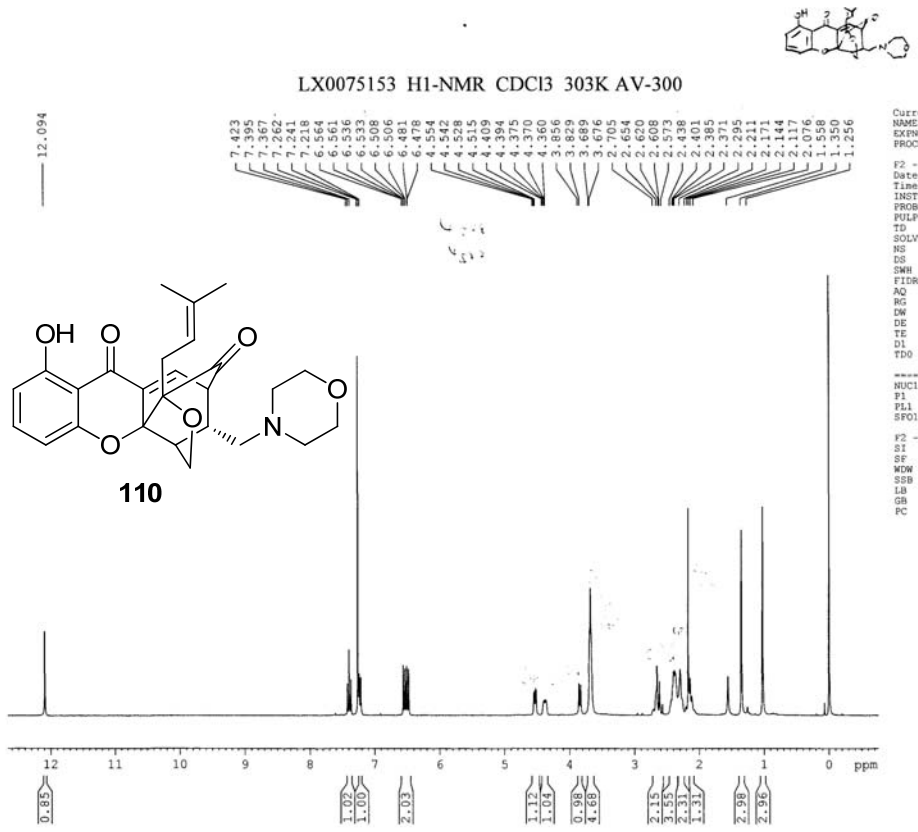


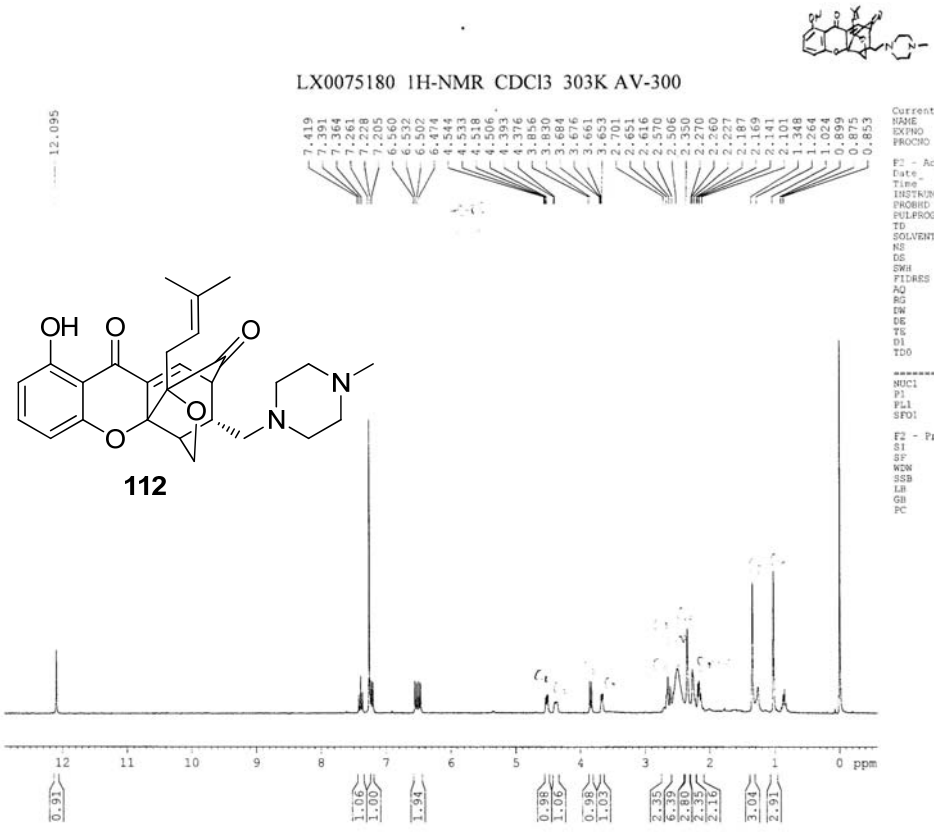

```
Current Data Parameters
NAME                  LX0075136
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_               20100509
Time                   18.09
INSTRUM                spect
PROBHD     5 mm QNP 1H/13
PULPROG             cosygpqf
TD                      2048
SOLVENT                CDCl3
NS                            2
DS                            8
SWH                4045.307 Hz
FIDRES             1.975248 Hz
AQ                 0.2531828 sec
RG                          322
DW                   123.600 usec
DE                     7.00 usec
TE                     300.0 K
d0                 0.00000300 sec
D1                 1.48689198 sec
d13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00024720 sec

======== CHANNEL f1 ========
NUC1                       1H
P0                      13.70 usec
P1                      13.70 usec
PL1                     -1.00 dB
SFO1               300.1319508 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                     10.00 %
GPZ2                     10.00 %
P16                    1000.00 usec

F1 - Acquisition parameters
ND0                           1
TD                          256
SFO1                300.132 MHz
FIDRES             15.801982 Hz
SW                     13.478 ppm
FnMODE                      QF

F2 - Processing parameters
SI                         1024
SF                 300.1300000 MHz
WDW                        SINE
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                 300.1300000 MHz
WDW                        SINE
SSB                           0
LB                         0.00 Hz
GB                            0
```

LX0075117 H1-NMR CDCl3 AV-300 303K

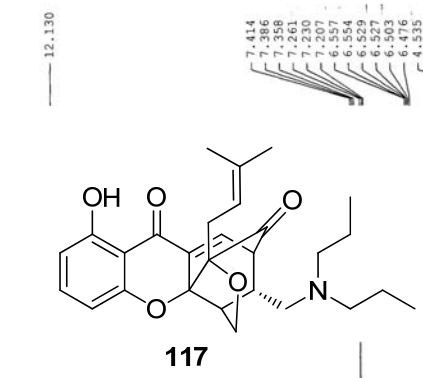



```
Current Data Parameters
NAME                  LX0075117
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20100422
Time                   22.04
INSTRUM                spect
PROBHD     5 mm QNP 1H/13
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                           16
DS                            1
SWH                6188.119 Hz
FIDRES             0.188846 Hz
AQ                 2.6477044 sec
RG                          128
DW                    80.800 usec
DE                     7.00 usec
TE                     300.0 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.70 usec
PL1                     -1.00 dB
SFO1               300.1318534 MHz

F2 - Processing parameters
SI                         32768
SF                 300.1300047 MHz
WDW                           EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```


LX0075153 H1-NMR CDCl3 303K AV-300
110
Current Data Parameters
NAME LX0075153
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100515
Time 15.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 228
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


LX0075180 1H-NMR CDCl3 303K AV-300
112
Current Data Parameters
NAME LX0075180
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100605
Time 15.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 322
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

LX0075163 H1-NMR CDCl3 303K AV-300



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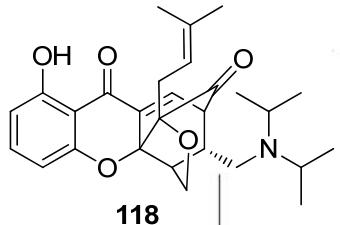
LX0075169 H1-NMR CDCl3 303K AV-300



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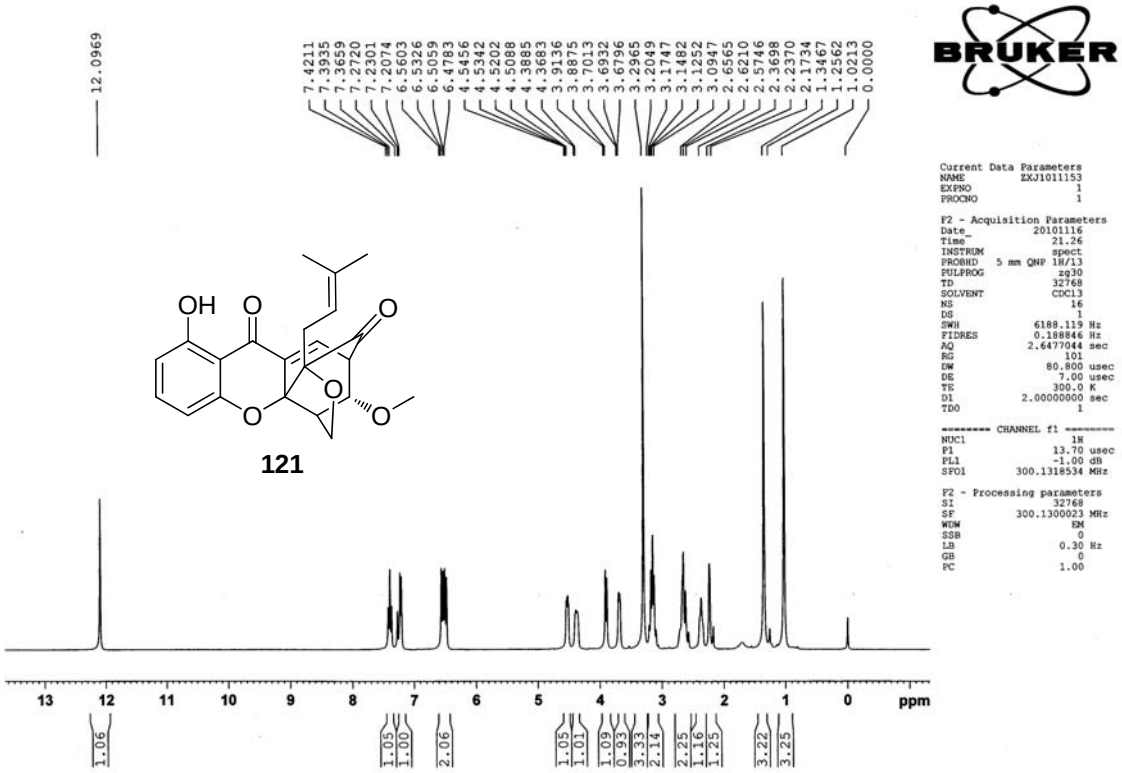
ZXJ1011153 HNMR CDCl3 303K AV-300



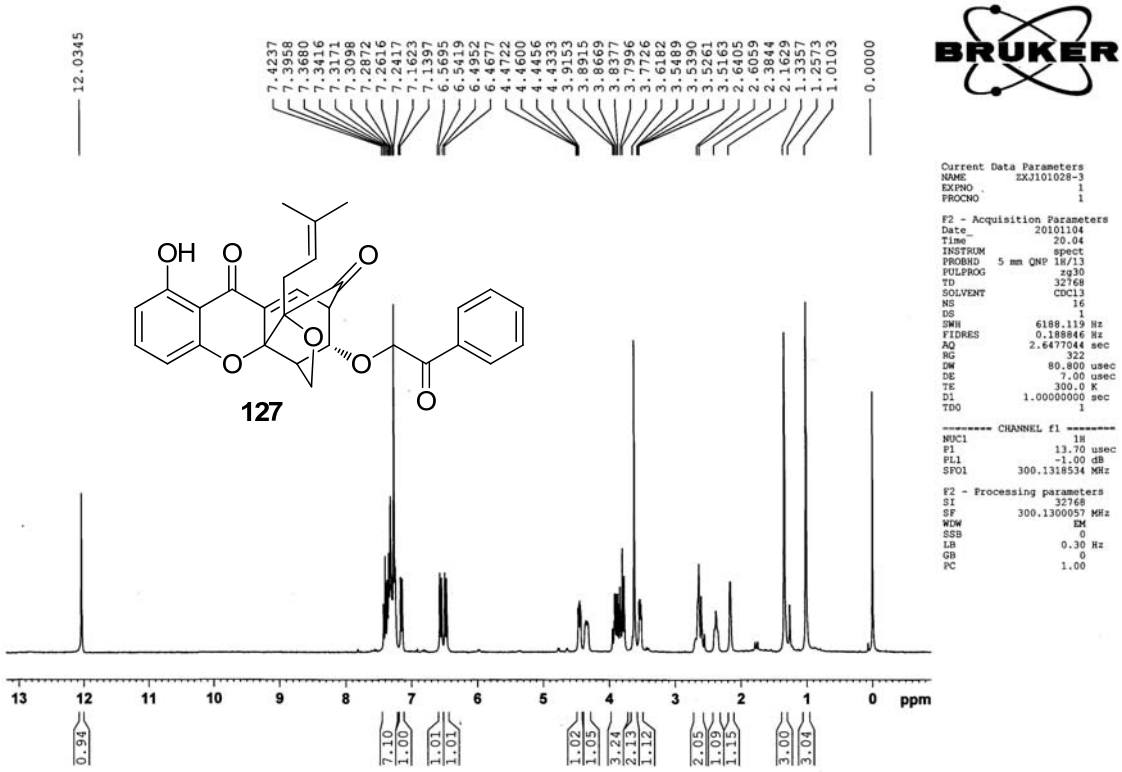
Current Data Parameters
NAME ZXJ1011153
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101116
Time 21.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 101
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300023 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ZXJ101028-3 H1-NMR CDCl3 303K AV-300



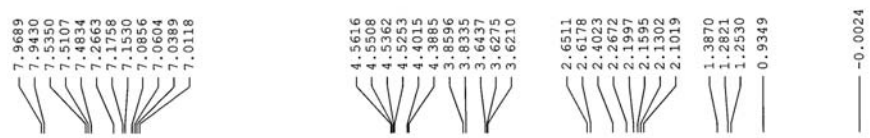
Current Data Parameters
NAME ZXJ101028-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101104
Time 20.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 322
DW 80.800 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LX110609 H1-NMR CDCl3 303K AV-300

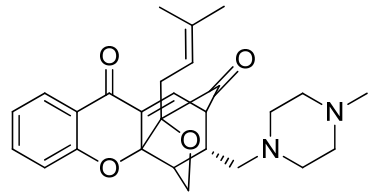


```
Current Data Parameters
NAME                  LX110609
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110609
Time                     20.16
INSTRUM                  spect
PROBHD     5 mm QNP 1H/13
PULPROG                  zg30
TD                      32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 4807.692 Hz
FIDRES              0.146719 Hz
AQ                  3.4079220 sec
RG                         161
DW                  104.000 usec
DE                    7.00 usec
TE                    300.0 K
D1                  2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.70 usec
PL1                      -1.00 dB
SFO1                300.1318534 MHz

F2 - Processing parameters
SI                          32768
SF                  300.1300046 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



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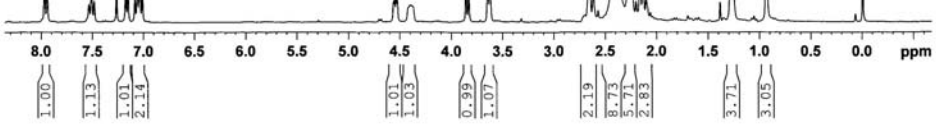
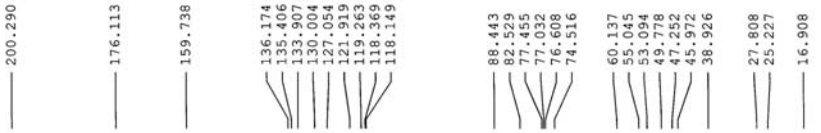


LX110609 C13-NMR CDCl3 303K AV-300



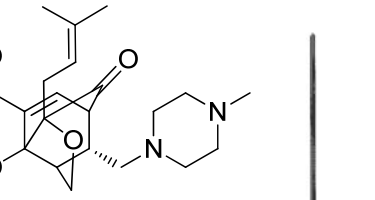
```
Current Data Parameters
NAME                  LX110609
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110609
Time                     20.20
INSTRUM                  spect
PROBHD     5 mm QNP 1H/13
PULPROG                zgpg30
TD                      65536
SOLVENT                  CDCl3
NS                         169
DS                           4
SWH                16666.666 Hz
FIDRES              0.254313 Hz
AQ                  1.9661300 sec
RG                         114
DW                   30.000 usec
DE                    7.00 usec
TE                    300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       11.80 usec
PL1                       1.00 dB
SFO1                75.4752203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      -1.00 dB
PL12                     14.30 dB
PL13                     14.30 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                          32768
SF                  75.4677466 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



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