Supporting Information

Benzothiazines in Organic Synthesis. The Preparation of Enantiomerically-Pure 4-Substituted Quinolones

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Experimental Section

General procedure for synthesizing tertiary butyl esters 11e-11f: To 40 mL THF in 100 mL round bottomed flask was added *t*-BuOH (4 mmol). After stirring at 0 °C for 10 min, 2.5 M *n*-BuLi (4 mmol) was added dropwise. After 30 min, above LiO^{*t*}Bu solution was transferred to a mixture of **11b** or **11c** (1.0 mmol) in 40 mL THF at 0 °C. The mixture was further stirred at rt for 11 hr, quenched with water, washed with brine, extracted with ethyl acetate (3 × 40 mL), dried over MgSO₄, filtered, and concentrated. The crude product was purified by flash chromatography to afford compound **11e** or **11f**.

Compound 11e: colorless semi-solid; 90% yield; IR: 3060, 2974, 2974, 2925, 2246, 2214, 1728, 1605, 1495, 1450 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.07-8.04 (m, 2H), 7.65-7.54 (m, 3H), 7.02 (d, *J* = 7.8 Hz, 1H), 6.96 (d, *J* = 1.0 Hz, 1H), 6.74 (dd, *J* = 7.8, 1.3 Hz, 1H), 3.84-3.74 (m, 1H), 3.53 (dd, *J* = 12.6, 4.2 Hz, 1H), 3.24 (dd, *J* = 12.6, 11.3 Hz, 1H), 2.86 (dd, *J* = 16.1, 5.2 Hz, 1H), 2.64 (dd, *J* = 16.1, 8.3 Hz, 1H), 2.31 (s, 3H), 1.39 (s, 9H); ¹³C NMR (75 Hz, CDCl₃): δ 170.3, 144.7, 139.1, 138.3, 133.7, 129.3, 128.8, 125.1, 124.5, 121.6, 121.5, 81.2, 52.1, 37.8, 30.9, 27.9, 20.9; Anal. Calcd for C₂₁H₂₅NO₃S: C, 67.89; H, 6.78; N, 3.77; Found: C, 67.61; H, 6.63; N, 3.66; [α]_D²⁵ = 8.29 (c 1.23, CHCl₃).

Compound 11f: pale yellow oil; 92% yield; IR: 3064, 2974, 2933, 1723, 1572, 1462, 1368, 1258, 1151, 1106 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.11-8.07 (m, 2H), 7.64-7.51 (m, 3H), 6.69 (s, 1H), 6.60 (s, 1H), 3.89 (s, 3H), 3.77-3.56 (m, 1H), 3.52 (dd, *J* = 13.0, 4.4 Hz, 1H), 3.35 (dd, *J* = 13.0, 9.9 Hz, 1H), 2.80 (dd, *J* = 16.1, 5.6 Hz, 1H), 2.66 (dd, *J* = 16.1, 8.3 Hz, 1H), 2.31 (s, 3H), 1.36 (s, 9H); ¹³C NMR (75 Hz, CDCl₃): δ 170.4, 152.3, 139.5, 133.5, 131.7, 130.1, 129.2, 129.0, 125.6, 118.2, 111.6, 81.2, 56.0, 52.6, 37.8, 32.0, 27.9, 21.3; Anal. Calcd for C₂₂H₂₇NO₄S: C, 65.81; H, 6.78; N, 3.49; Found: C, 66.01; H, 6.75; N, 3.56; [α]_D²⁵ = -20.34 (c 1.16, CHCl₃).

General procedure for synthesizing 3,4-dihydro-1*H*-quinolin-2-ones: To a round bottomed flask under N₂ was charged with 6% sodium amalgam (10.0 equiv.), and dispersed in dry MeOH, followed by addition of solid Na₂HPO₄ (10.0 equiv.). The mixture was stirred at rt. for a few min. A 2,1-benzothiazine (1.0 equiv.) was added into the well-stirred dispersion of buffered sodium amalgam as

a solution in THF. The reaction mixture was stirred at rt overnight. The mixture was filtered through a pad of Celite and concentrated in *vacuo*, to give crude product. Purification of the product by flash chromatography afforded desired 3,4-dihydro-1*H*-quinolin-2-one.

(S)-6-Benzoyloxy-4-methyl-3,4-dihydro-1*H*-quinolin-2-one (12a): colorless solid, yield 89.2 %; mp: 133-134 °C; IR(KBr): 3199, 3064, 2958, 2884, 1670, 1507, 1376, 1245 cm⁻¹; ¹H NMR (250 MHz, CDCl₃): δ 8.76 (s, 1H), 7.45-7.32 (m, 5H), 6.85-6.72 (m, 3H), 5.03 (s, 2H), 3.12- 3.04 (m, 1H), 2.69 (dd, *J* = 16.0, 5.8 Hz, 1H), 2.38 (dd, *J* = 16.0, 7.4 Hz, 1H), 1.28 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (62.5 MHz, CDCl₃): δ 171.0, 155.0, 136.9, 130.2, 130.2, 128.6, 127.9, 127.5, 116.3, 113.8, 113.1, 70.5, 38.2, 31.0, 19.6; Anal. Calcd for C₁₇H₁₇NO₂: C, 76.38; H, 6.41. Found: C, 76.15; H, 6.59; [α]_D²⁵ = -14.0 (c 1.0, CHCl₃).

(S)-4,7-Dimethyl-3,4-dihydro-1*H*-quinolin-2-one (12b): colorless solid, yield 93 %; mp: 156-157 °C; IR(KBr): 3207, 3125, 3109, 3035, 2962, 2913, 1695, 1658, 1372 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.89 (s, 1H), 7.10 (d, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.68 (s, 1H), 3.14- 3.10 (m, 1H), 2.74 (dd, *J* = 16.0, 5.7 Hz, 1H), 2.44 (dd, *J* = 16.0, 7.3 Hz, 1H), 2.33 (s, 3H), 1.32 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 172.1, 137.4, 136.3, 126.2, 125.7, 124.0, 116.3, 38.5, 30.3, 20.9, 19.8; Anal. Calcd for C₁₁H₁₃NO: C, 75.48; H, 7.48; N, 7.99. Found: C, 75.28; H, 7.55; N, 7.92; [α]_D²⁵ = -40.56 (c 1.08, CHCl₃).

(R)-8-Methoxy-4,6-dimethyl-3,4-dihydro-1*H*-quinolin-2-one (12c): colorless solid, yield 91 %; mp: 70-71 °C; IR(KBr): 3215, 2958, 2929, 1683, 1597, 1495, 1360, 1290 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.72 (s, 1H), 6.62 (s, 1H), 6.59 (s, 1H), 3.84 (s, 3H), 3.11-3.04 (m, 1H), 2.68 (dd, *J* = 16.0, 5.8 Hz, 1H), 2.38 (dd, *J* = 16.0, 7.0 Hz, 1H), 2.32 (s, 3H), 1.27 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 169.6, 145.7, 132.7, 128.9, 118.9, 109.8, 55.7, 38.4, 30.9, 21.4, 19.8; Anal. Calcd for C₁₂H₁₅NO₂: C, 72.22; H, 7.37; N, 6.82. Found: C, 70.45; H, 7.44; N, 6.91; [α]_D²⁵ = 19.42 (c 1.03, CHCl₃).

(S)-4-Methyl-3,4-dihydro-1*H*-benzo[*h*]quinolin-2-one (12d): colorless solid, yield 71 %; mp: 165-167 °C; IR(KBr): 3247, 3121, 2958, 1669, 1573, 1463, 1391 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.20 (s, 1H), 7.86-7.83 (m, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.58-7.46 (m, 3H), 7.34 (d, *J* = 8.4 Hz, 1H), 3.31-3.25 (m, 1H), 2.88

(dd, $J = 15.9, 6.0$ Hz, 1H), 2.56 (dd, $J = 15.9, 5.6$ Hz, 1H), 1.36 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 171.1, 133.0, 130.8, 128.7, 126.5, 125.8, 124.9, 124.4, 123.3, 122.3, 119.2, 38.4, 31.5, 19.9; HRMS calcd for $\text{C}_{14}\text{H}_{13}\text{NONa}$ $[\text{M} + \text{Na}]^+$ 234.0889; Found 234.0871; $[\alpha]_D^{25} = -108.1$ (c 0.84, CHCl_3).

(E)-3-[2,5-Dibromo-4-(2-methoxycarbonyl-vinyl)-phenyl]-acrylic acid methyl ester (13): white solid; mp: 262-265 °C; IR: 3096, 3064, 2945, 1707, 1433, 1372, 1274 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 7.92 (d, $J = 16.0$ Hz, 2H), 7.82 (s, 2H), 6.42 (d, $J = 16.0$ Hz, 2H), 3.83 (s, 6H); ^{13}C NMR (125MHz, CDCl_3): δ 166.2, 141.0, 137.1, 132.0, 124.1, 122.5, 52.1; HRMS calcd for $\text{C}_{28}\text{H}_{24}\text{Br}_2\text{O}_8\text{Na}$ $[\text{M}_2 + \text{Na}]^+$ 830.8056; Found 830.8040.

Compound 14: pale yellow semi-solid, yield 81%; IR: 3060, 3011, 2945, 2921, 1707, 1625, 1482, 1405, 1319, 1164 cm^{-1} ; ^1H NMR (250 MHz, CDCl_3): δ 8.14 (d, $J = 16.1$ Hz, 2H), 7.91-7.85 (m, 4H), 7.58-7.47 (m, 6H), 7.22 (s, 2H), 6.32 (d, $J = 16.1$ Hz, 2H), 3.79 (s, 6H), 3.23 (s, 6H); ^{13}C NMR (62.5 MHz, CDCl_3): δ 167.8, 141.6, 139.3, 139.2, 133.3, 130.9, 129.5, 128.3, 121.2, 117.9, 51.5, 45.5; HRMS calcd for $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_6\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 575.1281; Found 575.1294; $[\alpha]_D^{25} = -112.3$ (c 2.19, CHCl_3).

Compound 15: yellow solid, yield 91%; mp>250 °C; IR: 3064, 2998, 2945, 1728, 1478, 1405, 1397, 1225 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 8.08-8.05 (m, 4H), 7.67-7.55 (m, 6H), 6.95 (s, 2H), 3.86-3.81 (m, 2H), 3.65 (s, 6H), 3.57 (dd, $J = 12.7, 4.3$ Hz, 2H), 3.27 (dd, $J = 12.6, 11.2$ Hz, 2H), 3.06 (dd, $J = 16.3, 4.9$ Hz, 2H), 2.75 (dd, $J = 16.3, 9.0$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3): δ 171.4, 138.9, 138.5, 133.8, 129.3, 128.9, 124.6, 120.9, 51.9, 51.8, 36.3, 31.1; HRMS calcd for $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_6\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 575.1281; Found 575.1314; $[\alpha]_D^{25} = -8.78$ (c 0.98, CHCl_3).

(S,S)-4,9-Dimethyl-1,3,4,6,8,9-hexahydro-pyrido[2,3-g]quinoline-2,7-dione (16): pale yellow solid, yield 65 %; mp>250 °C; IR(KBr): 3215, 3064, 2958, 2847, 1674, 1499, 1356, 1245 cm^{-1} ; ^1H NMR (300 MHz, DMSO): δ 9.94 (s, 2H), 6.69 (s, 2H), 3.33-3.32(m, 2H), 2.96-2.94 (m, 2H), 2.16 (dd, $J = 15.4, 6.8$ Hz, 2H), 1.13 (d, $J = 6.2$ Hz, 6H); HRMS calcd for $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 267.1104; Found 267.1103.

General procedure for synthesizing compounds 17a-17e: To 30 mL THF in 100 mL round bottomed flask was added **11f** (0.5 mmol). After stirring at $-50\text{ }^{\circ}\text{C}$ for 10 minutes, 1.3 M LiHMDS (0.55 mmol) was added dropwise. After 1 hour, then MeI (2.0 mmol) was added dropwise to the reaction mixture at $-50\text{ }^{\circ}\text{C}$. The mixture was stirred for 1 hour, quenched with water, washed with brine, extracted with ethyl acetate ($3 \times 30\text{ mL}$), dried over MgSO_4 , filtered, and concentrated. The crude product was purified by flash chromatography (30% Et_2O /Hexanes) to afford a pale yellow oil **17a** in 80% yield. IR: 3064, 2982, 2929, 2864, 1723, 1572, 1462, 1372, 1252, 1151 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 8.11-8.03 (m, 2H), 7.65-7.52 (m, 3H), 6.66 (s, 1H), 6.58 (s, 1H), 4.10 (s, 3H), 3.62-3.52 (m, 1H), 3.47-3.41 (m, 1H), 2.83 (dd, $J = 16.8, 6.3\text{ Hz}$, 1H), 2.64 (dd, $J = 16.8, 5.2\text{ Hz}$, 1H), 2.29 (s, 3H), 1.38 (d, $J = 6.8\text{ Hz}$, 3H) 1.34 (s, 9H); ^{13}C NMR (75 Hz, CDCl_3): δ 170.6, 152.0, 138.4, 133.4, 131.2, 129.7, 129.4, 129.1, 125.1, 119.1, 111.3, 81.0, 55.8, 55.1, 38.2, 36.0, 27.8, 21.2, 13.1; Anal. Calcd for $\text{C}_{23}\text{H}_{29}\text{NO}_4\text{S}$: C, 66.48; H, 7.03; N, 3.37; Found: C, 66.60; H, 7.15; N, 3.22; $[\alpha]^{25}_{\text{D}} = -66.42$ (c 1.09, CHCl_3).

Compound 17b: pale yellow oil, 44% yield; IR: 2978, 2933, 1723, 1580, 1478, 1258, 1151 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.09-8.06 (m, 2H), 7.64-7.54 (m, 3H), 6.65 (s, 1H), 6.53 (s, 1H), 3.87 (s, 3H), 3.54-3.51 (m, 1H), 3.49-3.46 (m, 1H), 2.55 (dd, $J = 16.5, 9.1\text{ Hz}$, 1H), 2.47 (dd, $J = 16.5, 6.0\text{ Hz}$, 1H), 2.28 (s, 3H), 2.26-2.21 (m, 1H), 1.59-1.53 (m, 1H), 1.26 (s, 9H), 1.06 (t, $J = 7.4\text{ Hz}$, 3H); ^{13}C NMR (125 Hz, CDCl_3): δ 170.5, 152.5, 141.2, 133.2, 130.7, 130.6, 129.2, 128.7, 124.7, 121.1, 111.7, 80.8, 62.6, 56.0, 39.1, 39.0, 27.8, 21.5, 21.1, 11.7; Anal. Calcd for $\text{C}_{24}\text{H}_{31}\text{NO}_4\text{SNa}[\text{M} + \text{Na}]^+$ 452.1866; Found 452.1880; $[\alpha]^{25}_{\text{D}} = -20.0$ (c 0.62, CHCl_3).

Compound 17c: colorless liquid, 83% yield; IR: 2978, 2931, 2869, 1724, 1476, 1257, 1099 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 8.10-8.07 (m, 2H), 7.63-7.52 (m, 3H), 6.66 (s, 1H), 6.50 (s, 1H), 5.81-5.73 (m, 1H), 5.16-5.17 (m, 2H), 3.88 (s, 3H), 3.73-3.70 (m, 1H), 3.51-3.48 (m, 1H), 2.95-2.90 (m, 1H), 2.61-2.44 (m, 2H), 2.32-2.20 (m, 1H), 2.28 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (75 Hz, CDCl_3): δ 170.4, 152.6, 140.8, 133.7, 133.2, 131.4, 129.2, 128.7, 124.5, 121.1, 118.8, 111.7, 80.7, 60.1, 55.9, 38.7, 38.5, 32.4, 27.8, 21.2; Anal. Calcd for $\text{C}_{25}\text{H}_{31}\text{NO}_4\text{SNa} [\text{M} + \text{Na}]^+$ 464.1871; Found 464.1875.

Compound 17d: colorless liquid, 79% yield; IR: 2978, 2929, 1719, 1478, 1266, 1151 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.06-8.04 (m, 2H), 7.61-7.58 (m, 1H), 7.53-7.50 (m, 2H), 7.29-7.26 (m, 2H), 7.22-7.19 (m, 1H), 7.15-7.13 (m, 2H), 6.69 (s, 1H), 6.51 (s, 1H), 3.99-3.96 (m, 1H), 3.90 (s, 3H), 3.57 (dd, J = 14.4, 4.6 Hz, 1H), 3.38-3.35 (m, 1H), 2.74 (dd, J = 14.4, 10.4 Hz, 1H), 2.42 (dd, J = 10.6, 7.0 Hz, 2H), 2.31 (s, 3H), 1.14 (s, 9H); ^{13}C NMR (125 Hz, CDCl_3): δ 170.0, 152.7, 140.9, 133.2, 131.0, 129.3, 129.2, 128.7, 128.6, 126.7, 121.3, 111.8, 80.6, 62.5, 56.0, 39.2, 39.0, 34.0, 27.7, 21.2; Anal. Calcd for $\text{C}_{29}\text{H}_{33}\text{NO}_4\text{SNa}[\text{M} + \text{Na}]^+$ 514.2022; Found 514.2019; $[\alpha]_D^{25} = 0.59$ (c 0.68, CHCl_3).

Compound 17e: colorless oil, 48% yield; IR: 2978, 2921, 2868, 1723, 1474, 1254, 1147 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.12-8.11 (m, 2H), 7.63-7.54 (m, 3H), 6.66 (s, 1H), 6.58 (s, 1H), 3.99 (dd, J = 5.6, 4.9 Hz, 1H), 3.91-3.88 (m, 1H), 3.86 (s, 3H), 3.69-3.56 (m, 3H), 3.49-3.39 (m, 3H), 3.31 (s, 3H), 2.70-2.67 (m, 2H), 2.28 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (125 Hz, CDCl_3): δ 170.5, 152.7, 140.0, 133.3, 130.9, 130.3, 129.1, 125.5, 120.7, 111.7, 80.8, 71.6, 70.4, 68.2, 61.2, 58.9, 55.9, 38.6, 37.0, 27.8, 21.2; Anal. Calcd for $\text{C}_{26}\text{H}_{35}\text{NO}_6\text{SNa}[\text{M} + \text{Na}]^+$ 512.2077; Found 512.2065; $[\alpha]_D^{25} = -22.4$ (c 0.58, CHCl_3).

(*R*)-4-Ethyl-8-methoxy-6-methyl-3,4-dihydro-1*H*-quinolin-2-one (18a): colorless liquid, yield 63 %; IR(KBr): 3235, 2958, 2933, 2868, 1683, 1605, 1499, 1446, 1368, 1290 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 7.67 (s, 1H), 6.58 (s, 2H), 3.84 (s, 3H), 2.80-2.77 (m, 1H), 2.71 (dd, J = 11.1, 6.2 Hz, 1H), 2.53 (dd, J = 11.1, 3.8 Hz, 1H), 2.31 (s, 3H), 1.64-1.54 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 169.7, 145.8, 132.3, 127.6, 123.1, 120.3, 109.8, 55.7, 38.0, 35.8, 27.0, 21.3, 11.4; Anal. Calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: C, 71.21; H, 7.81. Found: C, 71.35; H, 7.84; $[\alpha]_D^{25} = -18.89$ (c 0.9, CHCl_3).

(*R*)-8-Methoxy-6-methyl-4-propyl-3,4-dihydro-1*H*-quinolin-2-one (18b): colorless solid, yield 65 %; mp: 79-80 $^\circ\text{C}$; IR(KBr): 3213, 2960, 1682, 1600, 1495, 13590, 1293 cm^{-1} ; ^1H NMR (250 MHz, CDCl_3): δ 7.67 (s, 1H), 6.57 (s, 2H), 3.84 (s, 3H), 2.90-2.86 (m, 1H), 2.71 (dd, J = 16.1, 6.1 Hz, 1H), 2.48 (dd, J = 16.1, 3.8 Hz, 1H), 2.31 (s, 3H), 1.59-1.47 (m, 2H), 1.40-1.25 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR (62.5 MHz, CDCl_3): δ 169.8, 145.8, 132.4, 128.0, 123.1, 120.2,

113.3, 109.8, 55.7, 36.3, 36.2, 36.1, 30.3, 21.4, 20.0, 14.0; Anal. Calcd for $C_{14}H_{19}NO_2Na$ $[M + Na]^+$ 256.1308; Found 256.1307.

(R)-4-But-3-enyl-8-methoxy-6-methyl-3,4-dihydro-1H-quinolin-2-one (18c): colorless liquid, yield 51 %; IR(KBr): 3419, 3211, 3072, 2913, 2835, 1679, 1597, 1503, 1450, 1360 cm^{-1} ; 1H NMR (300 MHz, $CDCl_3$): δ 7.69 (s, 1H), 6.58 (s, 2H), 5.83-5.73 (m, 1H), 5.06-4.96 (m, 2H), 3.84 (s, 3H), 2.95-2.87 (m, 1H), 2.72 (dd, $J = 16.1, 6.1$ Hz, 1H), 2.50 (dd, $J = 16.1, 3.6$ Hz, 1H), 2.31 (s, 3H), 2.17-2.03 (m, 2H), 1.75-1.59 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 145.8, 137.8, 132.4, 127.5, 123.1, 120.2, 115.2, 109.9, 55.7, 36.0, 35.7, 33.1, 30.8, 21.3; Anal. Calcd for $C_{15}H_{19}NO_2Na$ $[M + Na]^+$ 268.1308; Found 268.1318; $[\alpha]^{25}_D = 4.07$ (c 1.62, $CHCl_3$).

(R)-8-Methoxy-6-methyl-4-phenethyl-3,4-dihydro-1H-quinolin-2-one (18d): colorless liquid, yield 60 %; IR(KBr): 3215, 2921, 2847, 1674, 1499, 1356, 1103 cm^{-1} ; 1H NMR (300 MHz, $CDCl_3$): δ 7.69 (s, 1H), 7.30-7.25 (m, 2H), 7.20-7.15 (m, 3H), 6.59 (s, 1H), 6.58 (s, 1H), 3.84 (s, 3H), 2.92-2.90 (m, 1H), 2.78-2.55 (m, 4H), 2.31 (s, 3H), 1.92-1.87 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 145.9, 141.5, 132.5, 128.4, 128.3, 127.5, 125.9, 123.1, 120.2, 110.0, 55.7, 36.0, 35.9, 35.5, 32.9, 21.4; Anal. Calcd for $C_{19}H_{21}NO_2Na$ $[M + Na]^+$ 318.1464; Found 318.1456; $[\alpha]^{25}_D = 5.78$ (c 0.9, $CHCl_3$).

(S)-8-Methoxy-6-methyl-4-vinyl-3,4-dihydro-1H-quinolin-2-one (18e): colorless solid, yield 72 %; mp: 54-55 $^{\circ}C$; IR(KBr): 3231, 2953, 2921, 1679, 1503 cm^{-1} ; 1H NMR (300 MHz, $CDCl_3$): δ 7.70 (s, 1H), 6.60 (s, 2H), 5.92-5.80 (m, 1H), 5.17-5.05 (m, 2H), 3.84 (s, 3H), 3.64-3.62 (m, 1H), 2.72 (dd, $J = 16.1, 6.1$ Hz, 1H), 2.69 (dd, $J = 16.1, 7.4$ Hz, 1H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 145.8, 137.9, 132.8, 125.5, 123.4, 119.9, 116.4, 110.2, 55.7, 40.4, 36.6, 21.3; Anal. Calcd for $C_{13}H_{15}NO_2Na$ $[M + Na]^+$ 240.0995; Found 240.0999; $[\alpha]^{25}_D = -16.77$ (c 0.6, $CHCl_3$).

(R)-7-Methyl-6,7-dihydro-4H-2-thia-4-aza-inden-5-one (20a): colorless solid, yield 87 %; mp: 166-168 $^{\circ}C$; IR(KBr): 3190, 3152, 2954, 1676, 1619, 1364 cm^{-1} ; 1H NMR (300 MHz, $CDCl_3$): δ 8.65 (s, 1H), 6.94-6.93 (m, 1H), 6.74 (d, $J = 3.0$ Hz, 1H), 3.11-3.05 (m, 1H), 2.65 (dd, $J = 15.9, 5.4$ Hz, 1H), 2.33 (dd, $J = 15.9, 10.9$ Hz, 1H), 1.35 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.7, 136.3,

133.4, 118.6, 103.3, 39.1, 28.4, 19.3; HRMS calcd for C₈H₉NOSNa [M + Na]⁺ 190.0297; Found 190.0312; [α]_D²⁵ = -17.3 (c 0.97, CHCl₃).

(S)-7-Methyl-6,7-dihydro-4H-thieno[3,2-b]pyridin-5-one (20b): colorless solid, yield 81 %; mp: 163-164 °C; IR(KBr): 3199, 3137, 2994, 1683, 1638, 1368 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.81 (s, 1H), 7.09 (d, *J* = 5.2 Hz, 1H), 6.66 (d, *J* = 5.2 Hz, 1H), 3.28-3.25 (m, 1H), 2.77 (dd, *J* = 16.0, 6.1 Hz, 1H), 2.44 (dd, *J* = 16.0, 10.2 Hz, 1H), 1.34 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 171.1, 134.7, 123.1, 122.6, 117.8, 39.7, 27.9, 21.0; HRMS calcd for C₈H₉NOSNa [M + Na]⁺ 190.0297; Found 190.0298; [α]_D²⁵ = -16.15 (c 1.3, CHCl₃).

(S)-7-Methyl-6,7-dihydro-4H-furo[3,2-b]pyridin-5-one (20c): colorless solid, yield 90 %; mp: 152-154 °C; IR(KBr): 3216, 3148, 3019, 2965, 1676, 1634, 1364 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.03 (s, 1H), 7.20 (dd, *J* = 1.9, 0.8 Hz, 1H), 6.17 (d, *J* = 1.9 Hz, 1H), 3.28-3.20 (m, 1H), 2.85 (dd, *J* = 16.4, 7.2 Hz, 1H), 2.47 (dd, *J* = 16.4, 8.6 Hz, 1H), 1.30 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 170.4, 141.3, 140.2, 121.8, 102.6, 40.4, 26.7, 18.5; HRMS calcd for C₈H₉NO₂Na [M + Na]⁺ 170.0525; Found 170.0530; [α]_D²⁵ = -33.8 (c 0.71, CHCl₃).

(S)-4,9-Dimethyl-1,3,4,9-tetrahydro-pyrido[2,3-b]indol-2-one (20d): pale yellow semi solid, yield 68 %; IR(KBr): 3207, 3129, 3.39, 2970, 1675, 1650, 1462, 1364 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 10.53 (s, 1H), 7.49-7.46 (m, 1H), 7.26-7.23 (m, 1H), 7.14-7.09 (m, 2H), 3.72 (s, 3H), 3.41-3.34 (m, 1H), 2.90 (dd, *J* = 16.1, 6.9 Hz, 1H), 2.55 (dd, *J* = 16.1, 5.9 Hz, 1H), 1.38 (d, *J* = 6.9 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 173.5, 134.7, 134.2, 125.2, 119.9, 119.5, 117.2, 108.8, 96.5, 40.6, 28.6, 25.2, 20.8; HRMS calcd for C₁₃H₁₄N₂ONa [M + Na]⁺ 237.0998; Found 237.1011; [α]_D²⁵ = 62.77 (c 1.66, CHCl₃).

11e

Current Data Parameters
NAME KH-III-12
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020628
Time 16.36
INSTRUM dx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 64
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

11e

ppm

Current Data Parameters
NAME XH-11T-12
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020628
Time 16.44
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 204
DS 4
SWH 18832.578 Hz
FIDRES 0.287362 Hz
AQ 1.7400136 sec
RG 32768
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.75 usec
PL1 6.00 dB
SF01 75.4760107 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 24.50 dB
SF02 300.1312005 MHz

F2 - Processing Parameters
SI 32768
SF 75.4677565 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 12.50 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41144 Hz/cm

Current Data Parameters

NAME XH-III-6-A1

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020620

Time 23.49

INSTRUM drx300

PROBHD 5 mm Multinuc1

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SWH 6172.839 Hz

FIDRES 0.188380 Hz

AQ 2.6542580 sec

RG 181

DW 81.000 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.50 usec

PL1 0.00 dB

SFO1 300.1318534 MHz

F2 - Processing parameters

SI 32768

SF 300.1300043 MHz

WDW EM

SSB 0

LB 0.20 Hz

GB 0

PC 1.30

1D NMR plot parameters

CX 20.00 cm

CY 12.50 cm

F1P 9.000 ppm

F1 2701.17 Hz

F2P -0.500 ppm

F2 -150.06 Hz

PPMCM 0.47500 ppm/cm

HZCM 142.56175 Hz/cm

Current Data Parameters

NAME XH-III-6-A1

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020620

Time 23.23

INSTRUM drx300

PROBHD 5 mm Multinuc1

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 440

DS 4

SWH 18832.578 Hz

FIDRES 0.287362 Hz

AQ 1.7400136 sec

RG 32768

DW 26.550 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C

P1 7.75 usec

PL1 6.00 dB

SFO1 75.4760107 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 100.00 usec

PL2 120.00 dB

PL12 24.50 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768

SF 75.4677531 MHz

MDN EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

CY 8.00 cm

F1P 200.000 ppm

F1 15093.55 Hz

F2P -10.000 ppm

F2 -754.68 Hz

PPMCM 10.50000 ppm/cm

HZCM 792.41138 Hz/cm

12a

Current Data Parameters
NAME XH-II-178
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020615
Time 17.26
INSTRUM arx250
PROBHD 5 mm GNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5208.333 Hz
FIDRES 0.158946 Hz
AQ 3.1457779 sec
RG 1430
DM 96.000 use
DE 137.14 use
TE 300.0 K
D1 1.0000000 sec
P1 8.70 use
SF01 250.1315321 MHz
1H

NUCLEUS 1H

S-14

F2 - Processing parameters
SI 16384
SF 250.1300087 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.50

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 2251.17 Hz
F2P -0.500 ppm
F2 -125.07 Hz
PPMCM 0.47500 ppm
HZCM 118.81175 Hz/

12a

Current Data Parameters
NAME XH-II-178
EXPNO 2
PROCNO 1

ppm

171.047
155.014
136.922
130.231
130.207
128.574
127.989
127.482
116.310
113.805
113.127
77.505
76.997
76.489
70.478
38.215
30.962
19.595

F2 - Acquisition Parameters
Date_ 20020615

Time 17.31
INSTRUM arx250
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 36864
SOLVENT CDCl3
NS 1326
DS 4
SWH 17241.379 Hz
FIDRES 0.467702 Hz
AQ 1.0691060 sec
RG 22800
DW 29.000 use
DE 41.43 use
TE 300.0 K
D12 0.00002000 sec
DL5 23.00 dB
CPDPRG waltz16
P31 103.00 use
D1 1.00000000 sec
P1 5.35 use

SF01 62.9023694 MHz
NUCLEUS 13C
D11 0.03000000 sec
F2 - Processing parameters
SI 32768
SF 62.8952419 MHz
WDW EM
SSB 0
LB 0.25 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F4P 200.000 ppm
F1 12579.05 Hz
F2P -10.000 ppm
F2 -628.95 Hz
PPMCM 10.50000 ppm
HZCM 660.40002 Hz/

12b

Current Data Parameters

NAME	XH-III-8
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20020627
Time	11.02
INSTRUM	DRX500
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	45056
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.229283 Hz
AQ	2.1807604 sec
RG	64
DW	48.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec

===== CHANNEL f1 =====

NUC1	¹ H
P1	13.25 usec
PL1	-3.00 dB
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.130000 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	10.000 ppm
F1	5001.30 Hz
F2P	-0.500 ppm
F2	-250.07 Hz
PPMCM	0.52500 ppm/cr
HZCM	262.56824 Hz/cm

12b

Current Data Parameters
NAME XH-III-B
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020625
Time 16.41

INSTRUM DRX500
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 202
DS 4
SWH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258188 sec
RG 16384
DM 12.600 usec
DE 5.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.24 usec
PL1 3.00 dB
SF01 125.7713108 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577995 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45691 Hz/cm

12C

ppm

Current Data Parameters
NAME XH-II-179
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 16.51

INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 479
DS 4
SWH 18832.578 Hz
FIDRES 0.287362 Hz
AQ 1.7400136 sec
RG 32768
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.75 usec
PL1 6.00 dB
SF01 75.4760107 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 24.50 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41138 Hz/cm

Current Data Parameters

NAME XH-IV-35-1

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030311

Time 16.03

INSTRUM drx300

PROBHD 5 mm Multinuc1

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SMH 6172.839 Hz

FIDRES 0.188380 Hz

AQ 2.6542580 sec

RG 64

DM 81.000 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.50 usec

PL1 0.00 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 1.30

1D NMR plot parameters

CX 20.00 cm

CY 9.00 cm

F1P 9.000 ppm

F1 2701.17 Hz

F2P -0.500 ppm

F2 -150.06 Hz

PPMCM 0.47500 ppm/cm

HZCM 142.56175 Hz/cm

12d

¹³C NMR

ppm

171.100

132.973

130.808

128.658

126.471

125.796

124.917

124.458

123.270

122.269

119.236

77.426

77.002

76.579

38.392

31.499

19.950

Current Data Parameters
NAME XH-IV-35
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030311

Time 14.41

INSTRUM drx300

PROBHD 5 mm Multinuc1

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 1696

DS 4

SWH 18850.141 Hz

FIDRES 0.287630 Hz

AQ 1.7383924 sec

H6 32768

DW 26.525 usec

DE 37.89 usec

TE 300.0 K

D1 1.00000000 sec

D11 0.03000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====

NUC1 ¹³C

P1 7.75 usec

PL1 6.00 dB

SFO1 75.4760107 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 ¹H

PCPD2 100.00 usec

PL2 120.00 dB

PL12 24.50 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768

SF 75.4677508 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

CY 25.00 cm

F1P 200.000 ppm

F1 15093.55 Hz

F2P -10.000 ppm

F2 -754.68 Hz

PPHCK 10.50000 ppm/cm

HZCM 792.41139 Hz/cm

Current Data Parameters
NAME XH-III-57
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020826
Time 16.30
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188360 Hz
AQ 2.6542580 sec
RG 2048
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

Current Data Parameters
NAME KH-III-57
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020830
Time 15.33
INSTRUM DRX500
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 807
DS 4
SMH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258188 sec
RG 16384
DM 12.600 usec
DE 5.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.24 usec
PL1 3.00 dB
SF01 125.7713108 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing Parameters
SI 32768
SF 125.7577910 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 50.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45679 Hz/cm

Current Data Parameters
 NAME XH-III-63-A1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020830
 Time 15.07
 INSTRUM arx250
 PROBN 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 715
 DW 96.000 use
 DE 137.14 use
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.70 use
 SF01 250.1315321 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 250.1300043 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 9.000 ppm
 F1 2251.17 Hz
 F2P 2.000 ppm
 F2 500.26 Hz
 PPMCM 0.35000 ppm
 HZCM 87.54550 Hz/

1.4

Current Data Parameters
NAME XH-III-63-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020830
Time 15.13

INSTRUM arx250
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 36864
SOLVENT CDCl3
NS 557
DS 4

SWH 17241.379 Hz
FIDRES 0.467702 Hz
AQ 1.0691060 sec
RG 22800

DW 29.000 use
DE 41.43 use
TE 300.0 K
D12 0.0002000 sec
DL5 23.00 dB

CPDPRG waltz16
P31 103.00 use
D1 1.0000000 sec
P1 5.35 use

SF01 62.9023694 MHz
NUCLEUS 13C
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 62.8952429 MHz

WDW EM
SSB 0
LB 1.00 Hz
GB 0

PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm

F1P 200.000 ppm
F1 12579.05 Hz
F2P -10.000 ppm
F2 -628.95 Hz

PPMCM 10.50000 ppm
HZCM 660.40009 Hz/

1.5

0.00004

Current Data Parameters
NAME XH-III-65-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020901
Time 14.08

INSTRUM gnx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 228.1
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

Current Data Parameters
NAME XH-III-65-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020901

Time 14.16
INSTRUM drx300
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2518
DS 4
SWH 18850.141 Hz
FIDRES 0.287630 Hz
AQ 1.7383924 sec
RG 32768
DM 26.525 usec
DE 37.89 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.75 usec
PL1 6.00 dB
SF01 75.4760107 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 24.50 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41138 Hz/cm

Current Data Parameters
 NAME XH-III-11-A1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020630
 Time 14.46
 INSTRUM drx300
 PROBD 5 mm Multinuc1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 64
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300001 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.30

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.47500 ppm/cm
 HZCM 142.56175 Hz/cm

Current Data Parameters
NAME XH-III-11-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020630
Time 14.51
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 135
DS 4
SWH 18832.578 Hz
FIDRES 0.287362 Hz
AQ 1.7400136 sec
RG 32768
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.75 usec
PL1 6.00 dB
SF01 75.4760107 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 24.50 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41144 Hz/cm

ppm

Current Data Parameters
 NAME XH-V-24-A1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030414
 Time 11.06

INSTRUM DRX500
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 57344
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 HZ
 FIDRES 0.180151 HZ
 AQ 2.7754996 sec
 RG 64
 DM 48.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.25 usec
 PL1 -3.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.130081 MHz
 WDW EM
 SSB 0
 LB 0.20 HZ
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 50.00 cm
 F1P 9.000 ppm
 F1 4501.17 HZ
 F2P -0.500 ppm
 F2 -250.06 HZ
 PPMCM 0.47500 ppm/cm
 HZCM 237.56174 HZ/cm

Current Data Parameters
 NAME KH-V-24-A1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030414
 Time 11.12

INSTRUM DRX500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 192

DS 4
 SMH 39681.812 Hz
 FIDRES 0.605496 Hz
 AQ 0.8258198 sec

RG 16384
 DM 12.600 usec
 DE 6.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.90 usec
 PL1 3.00 dB
 SFO1 125.7713109 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 88.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577971 MHz
 MDM EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 FLP 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

17c

Current Data Parameters
NAME XH-II-173-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 15.06
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 90.5
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300015 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPKM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

17c

Current Data Parameters
NAME XH-II-173-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020624
Time 15.13
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 180
DS 4
SWH 18832.578 Hz
FIDRES 0.287362 Hz
AQ 1.7400136 sec
RG 32768
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.75 usec
PL1 6.00 dB
SF01 75.4760107 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 24.50 dB
SF02 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677554 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 12.50 cm
FAP 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.50000 ppm/cm
HZCM 792.41144 Hz/cm

17d

Current Data Parameters
NAME XH-V-23-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030414
Time 10.47
INSTRUM DRX500
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 57344
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.180151 Hz
AQ 2.7754996 sec
RG 64
DM 48.400 usec
DE 6.00 usec
TE 296.7 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.25 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300109 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 25.00 cm
F1P 9.000 ppm
F1 4501.17 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 237.56174 Hz/cm

17d

Current Data Parameters
NAME XH-V-23-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030414
Time 10.55

INSTRUM DBX500
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 390
DS 4
SWH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258188 sec
RG 16384
DM 12.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

CHANNEL f1
NUC1 13C
P1 7.90 usec
PL1 3.00 dB
SF01 125.7713108 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 88.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578007 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCH 10.50000 ppm/cm
HZCM 1320.45679 Hz/cm

Current Data Parameters
 NAME XH-V-25-A1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030414
 Time 11.18
 INSTRUM DRX500
 PROBD 5 mm Multinuc1
 PULPROG zg30
 TD 57344
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 10330.578 HZ
 FIDRES 0.180151 HZ
 AQ 2.7754996 sec
 RG 64
 DM 48.400 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.25 usec
 PL1 -3.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.130068 MHz
 WDM EM
 SSB 0
 LB 0.20 HZ
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 9.000 ppm
 F1 4501.17 HZ
 F2P -0.500 ppm
 F2 -250.06 HZ
 PPMCM 0.47500 ppm/cm
 HZCM 237.56174 HZ/cm

17e

Current Data Parameters
NAME XH-V-25-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030414
Time 11.22
INSTRUM DRX500
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 222
DS 4
SMH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258188 sec
RG 16394
DM 12.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.90 usec
PL1 3.00 dB
SF01 125.7713108 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 88.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577983 MHz
MDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45679 Hz/cm

18a

Current Data Parameters
NAME XH-III-15-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020705
Time 14.22
INSTRUM DRX500
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 45056
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.229283 Hz
AQ 2.1807604 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 296.7 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.25 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 4501.17 Hz
F2 -0.500 ppm
PPMCM 0.47500 ppm/cr
HZCM 237.56175 Hz/cm

Current Data Parameters
NAME XH-III-15-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020705
Time 14.29
INSTRUM DRX500
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1394
DS 4
SWH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258188 sec
RG 16384
DM 12.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 8.24 usec
PL1 3.00 dB
SF01 125.7713108 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 107.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577935 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 50.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45691 Hz/cm

Current Data Parameters
 NAME XH-VI-9-A1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030814
 Time 18.02

F2 - Processing Parameters
 SI 16384
 SF 250.1300075 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 9.000 ppm
 F1 2251.17 Hz
 F2P -0.500 ppm
 F2 -125.07 Hz
 PPMCM 0.47500 ppm
 HZCM 118.81175 Hz/

Current Data Parameters

NAME	XH-VI-9-A1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030814
Time	18.10
INSTRUM	acx250
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	36864
SOLVENT	CDCl3
NS	9195
DS	4
SWH	17241.379 HZ
FIDRES	0.467702 HZ
AQ	1.0691060 sec
RG	22800
DM	29.000 use
DE	41.43 use
TE	300.0 K
D12	0.00002000 sec
DL5	23.00 dB
CPDPRG	waltz16
P31	103.00 use
D1	1.00000000 sec
P1	5.35 use
SFO1	62.9023694 MHz
NUCLEUS	¹³ C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	62.8952403 MHz
WDW	EM
SSB	0
LB	1.00 HZ
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	50.00 cm
F1P	200.000 ppm
F1	12579.05 HZ
F2P	-10.000 ppm
F2	-628.95 HZ
BPCKM	10.50000 ppm
HZCM	650.40002 HZ/

Current Data Parameters
NAME XH-III-14
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020703
Time 14.52
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 645.1
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

Current Data Parameters
 NAME XH-III-14
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020703
 Time 15.11
 INSTRUM drx300
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 725
 DS 4
 SMH 18832.578 Hz
 FIDRES 0.287362 Hz
 AQ 1.740136 sec
 RG 32768
 DM 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 7.75 usec
 PL1 6.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 24.50 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677496 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 792.41138 Hz/cm

¹H NMR

Current Data Parameters
NAME XH-VI-8-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030813
Time 14.35
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 64
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D31 0.00000000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 7.05 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47500 ppm/cm
HZCM 142.56175 Hz/cm

¹³C NMR

Current Data Parameters
 NAME XH-VI-8-A1
 EXRNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030813

Time 14.41
 INSTRUM drx300
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 917
 DS 4
 SMH 18832.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 sec
 RG 22528
 DM 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 D31 0.00000000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 8.50 usec
 PL1 6.00 dB
 SF01 75.4760107 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 25.60 dB
 SF02 300.1312005 MHz

F2 - Processing Parameters
 SI 32768
 SF 75.467502 MHz
 MDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 25.00 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 792.41138 Hz/cm

18e

Current Data Parameters
NAME XH-VI-10-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030815
Time 12.56
INSTRUM DRX500
PROBHD 5 mm Multinucl
PULPROG zg30
TD 57344
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 HZ
FIDRES 0.180151 HZ
AQ 2.7754996 sec
RG 64
RG 48.400 usec
DE 6.00 usec
TE 296.7 K
D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 13.25 usec
PL1 -3.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters

SF 32768
SF 500.130128 MHz
WDW EM
SSB 0
LB 0.20 HZ
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 12.50 cm
F1P 9.000 ppm
F1 4501.17 HZ
F2P 1.700 ppm
F2 850.22 HZ
PMCM 0.36500 ppm/cf
H2CM 182.54744 HZ/cm

18e

Current Data Parameters

NAME XH-VI-10-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030815
Time 12.59
INSTRUM DRX500
PROBHD 5 mm NUTINUC1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2720
DS 4
SWH 39681.812 Hz
FIDRES 0.605496 Hz
AQ 0.8258189 sec
RG 16384
DW 12.600 usec
DE 5.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.90 usec
PL1 3.00 dB
SF01 125.7713108 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 88.00 usec
PL2 0.00 dB
PL12 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577923 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 100.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45679 Hz/cm

Current Data Parameters

NAME XH-IV-146

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030317

Time 10.48

INSTRUM drx300

PROBHD 5 mm Multinuc1

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SWH 6172.839 Hz

FIDRES 0.188360 Hz

AQ 2.6542580 sec

RG 64

DM 81.000 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.50 usec

PL1 0.00 dB

SFO1 300.1318534 MHz

F2 - Processing parameters

SI 32768

SF 300.1300056 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.30

1D NMR plot parameters

CX 20.00 cm

CY 8.00 cm

F1P 9.000 ppm

F1 2701.17 Hz

F2P -0.500 ppm

F2 -150.06 Hz

PPMCM 0.47500 ppm/cm

HZCM 142.56175 Hz/cm

Current Data Parameters
 NAME XH-IV-146-A1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20030312
 Time 15.42
 INSTRUM arx250
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 36864
 SOLVENT CDCl3
 NS 407
 DS 4
 SWH 17241.379 Hz
 FIDRES 0.467702 Hz
 AQ 1.0691060 sec
 RG 22800
 DM 29.000 use
 DE 41.43 use
 TE 300.0 K
 D12 0.00002000 sec
 DL5 23.00 dB
 CPDPRG waltz16
 P31 103.00 use
 D1 1.00000000 sec
 P1 5.35 use
 SFO1 62.9023694 MHz
 NUCLEUS 13C
 D11 0.03000000 sec

F2 - Processing parameters

SI 32768
 SF 62.8952424 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot Parameters

CX 20.00 cm
 CY 10.00 cm
 F1P 200.000 ppm
 F1 12579.05 Hz
 F2P -10.000 ppm
 F2 -628.95 Hz
 PPMCM 10.50000 ppm
 HZCM 660.40002 Hz/

¹H NMR

20b

Current Data Parameters
NAME XH-IV-147
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030317
Time 10.50
INSTRUM dpx300
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 64
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D31 0.00000000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 7.50 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

¹³C NMR

20b

ppm

Current Data Parameters
NAME XH-IV-147
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030317

Time 10.57
INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2379
DS 4

SWH 18850.141 Hz
FIDRES 0.287630 Hz
AQ 1.7383924 sec

RG 32768

DW 26.525 usec
DE 37.89 usec

TE 300.0 K

DT 1.00000000 sec

d11 0.03000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====

NUC1 ¹³C

P1 7.75 usec

PL1 6.00 dB

SFO1 75.4760107 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 ¹H

PCPD2 100.00 usec

PL2 120.00 dB

PL12 24.50 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768

SF 75.4677514 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

CY 12.50 cm

F1P 200.000 ppm

F1 15093.55 Hz

F2P -10.000 ppm

F2 -754.68 Hz

PPMCM 10.50000 ppm/cm

HZCM 792.41198 Hz/cm

Current Data Parameters

NAME XH-IV-148-A1

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030312

Time 16.19

INSTRUM arx250

PROBHD 5 mm GNP 1H

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SWH 5208.333 Hz

FIDRES 0.158946 Hz

AQ 3.1457779 sec

RG 4096

DM 96.000 use

DE 137.14 use

TE 300.0 K

D1 1.0000000 sec

P1 8.70 use

SFO1 250.1315321 MHz

NUCLEUS 1H

F2 - Processing parameters

SI 16384

SF 250.1300065 MHz

WDW EM

SSB 0

LB 0.05 Hz

GB 0

PC 0.50

1D NMR plot parameters

CX 20.00 cm

CY 12.50 cm

F1P 9.000 ppm

F1 2251.17 Hz

F2P -0.500 ppm

F2 -125.07 Hz

PPMCM 0.47500 ppm

HZCM 118.81175 Hz/

Current Data Parameters
 NAME XH-IV-148-A1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030312
 Time 16.26

INSTRUM arx250
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 36864
 SOLVENT CDCl3
 NS 2193
 DS 4

SWH 17241.379 Hz
 FIDRES 0.467702 Hz
 AQ 1.0691060 sec

RG 22800

DW 29.000 use

DE 41.43 use

TE 300.0 K

D12 0.00002000 sec

DL5 23.00 dB

CPDPRG waltz16

P31 103.00 use

D1 1.00000000 sec

P1 5.35 use

SF01 62.9023694 MHz

NUCLEUS 13C

D11 0.03000000 sec

F2 - Processing parameters

SI 32768

SF 62.8952403 MHz

WDW EM

SSB 0

LB 1.20 Hz

GB 0

PC 1.20

1D NMR plot parameters

CX 20.00 cm

CY 25.00 cm

F1P 200.000 ppm

F1 12579.05 Hz

F2P -10.000 ppm

F2 -628.95 Hz

PPMCM 10.50000 ppm

20d

Current Data Parameters
NAME XH-IV-144-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030316
Time 17.29

INSTRUM drx300
PROBHD 5 mm Multinuc1
PULPROG zg30

TD 32768
SOLVENT CDCl3

NS 16
DS 2

SWH 6172.839 Hz
FIDRES 0.188380 Hz

AQ 2.6542580 sec
RG 64

DW 81.000 usec
DE 6.00 usec

TE 300.0 K
D1 1.00000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 7.50 usec

PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768

SF 300.1300118 MHz
WDW EM

SSB 0
LB 0.30 Hz

GB 0
PC 1.30

1D NMR plot parameters
CX 20.00 cm

CY 8.00 cm
F1P 11.000 ppm

F1 3301.43 Hz
F2P -0.500 ppm

F2 -150.07 Hz
PPMCM 0.57500 ppm/cm

HZCM 172.57475 Hz/cm

Integral
1.0000
1.0588
1.3224
1.9362

3.0480
1.0291
1.0410
1.0429
3.0650

20d

ppm

173.551

134.727

134.253

125.212

119.970

119.526

117.226

108.871

96.502

77.423

76.999

76.576

40.601

28.596

25.227

20.825

Current Data Parameters
NAME XH-IV-144-A1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030316
Time 17.34

INSTRUM dpx300
PROBHD 5 mm Multinuc1
PULPROG zgpg30

TD 65536
SOLVENT CDCl3
NS 2393

DS 4
SWH 18850.141 Hz
FIDRES 0.287630 Hz

AQ 1.7383924 sec
RG 32768

DW 26.525 usec
DE 37.89 usec

TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

D31 0.00000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.75 usec
PL1 6.00 dB

SFO1 75.4760107 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec

PL2 120.00 dB
PL12 24.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677519 MHz
WDW EM

SSB 0
LB 1.00 Hz
GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 11.00 cm
F1P 200.000 ppm

F1 15093.56 Hz
F2P -10.000 ppm
F2 -754.88 Hz

PPMCM 10.50000 ppm/cm
HZCM 792.41138 Hz/cm