Supporting Information

Copper-Mediated C—N Bond Formation *via* Direct Aminolysis of Dithioacetals

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I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel (300-400 mesh). ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C on a 500 MHz NMR spectrometer, and TMS as internal standard. IR spectra (KBr) were recorded in the range of 400 ~ 4000 cm^{-1} . Mass spectra were recorded on Agilent 1100 LCMsD mass spectrometer. C, H, N, elemental analyses were conducted with Bio-Rad Co's elemental analytical instrument.

II. Synthesis and the analytical data of the substrates 2a-b.

Precursors **1a** and **1b** were synthesized by a two-step reaction starting from ethyl acetoacetate in excellent yields according to the literature: Bi, X.; Liu, Q.; Sun, S.; Liu, J.; Pan, W.; Zhao, L.; Dong, D. *Synlett* **2005**, 49.

General Procedure for the preparation of **2a-b**: (with **2a** as an example): To a stirred solution of **1a** (2.93 g, 10.0 mmol) in EtOH (20 mL) at 0 °C, NaBH_4 (0.42 g, 11.0 mmol) was added slowly. After stirred for 20 min, the mixture was concentrated in vacuo and purified by silica gel column chromatography ($\text{EtOAc} : \text{CH}_3\text{COCH}_3 = 1 : 1$ as eluent) to give product **2a** (2.99 g, 97%).

2a: Yellow solid, m.p. decomposed at 200 °C. ^1H NMR (500 MHz, DMSO): δ = 1.62 (q, J = 13.5 Hz, 1H), 1.96 (d, J = 13.5 Hz, 1H), 2.57 (dd, J = 10.0, 14.5 Hz, 1H), 2.73 (d, J = 14.0 Hz, 2H), 2.87 (t, J = 13.0 Hz, 2H), 3.20 (dd, J = 2.5, 14.5 Hz, 1H), 4.40 (dd, J = 2.5, 10.0 Hz, 1H), 4.89 (s, 1H), 7.21 (dd, J = 5.5, 7.0 Hz, 1H), 7.29 (d, J = 8.0

Hz, 1H), 7.68 (t, $J = 7.5$ Hz, 1H), 8.48 (d, $J = 5.0$ Hz, 1H). ^{13}C NMR (100 MHz, DMSO): $\delta = 188.6, 175.1, 158.7, 148.8, 136.3, 123.6, 121.5, 87.2, 78.8, 41.8, 41.1, 31.7$ (2C), 25.1. IR (KBr, cm^{-1}): $\nu = 3427, 2896, 1701, 1580, 1438$. Anal. calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_3\text{S}_2$: C, 54.35; H, 4.89; N, 4.53; Found: C, 54.41; H, 4.93; N, 4.51.

2b: Yellow solid, m.p. decomposed at 206 °C. ^1H NMR (500 MHz, DMSO): $\delta = 1.62$ (q, $J = 13.0$ Hz, 1H), 1.93 (d, $J = 14.0$ Hz, 1H), 2.54 (dd, $J = 8.5, 14.5$ Hz, 1H), 2.69 (d, $J = 13.5$ Hz, 2H), 2.84 (t, $J = 13.0$ Hz, 2H), 3.04 (dd, $J = 2.5, 14.5$ Hz, 1H), 4.21 (dd, $J = 2.5, 8.5$ Hz, 1H), 4.83 (s, 1H), 7.23 (d, $J = 5.0$ Hz, 2H), 8.39 (d, $J = 5.0$ Hz, 2H). ^{13}C NMR (125 MHz, DMSO): $\delta = 188.6, 175.3, 149.1$ (2C), 147.6, 124.8 (2C), 86.7, 78.7, 42.0, 37.5, 31.8 (2C), 25.2. IR (KBr, cm^{-1}): $\nu = 3429, 2898, 1702, 1581, 1443$.

III. Synthesis of the substrates 5a-g

Procedure for the preparation of **4g**: To a stirred solution of **4f** (2.17 g, 10.0 mmol) in DMF (20 mL) at 0 °C was added POCl_3 (1.11 mL, 12.0 mmol) dropwise. The reaction mixture was stirred over a period of 3 h. Then the mixture was poured into water

under stirring. The precipitated solid was collected by filtration, washed with water, and dried in vacuo to afford the product **4g** (1.69 g, 85%).

4f: Light yellow solid. m.p.199-201°C. ^1H NMR (500 MHz, DMSO): δ = 2.09 (m, 5H), 2.86 (t, J = 6.5 Hz, 2H), 2.93 (t, J = 7.0 Hz, 2H), 7.49 (s, 1H), 7.83 (s, 1H). ^{13}C NMR (125 MHz, DMSO): δ = 191.2, 169.1, 160.4, 133.8, 29.2, 28.7, 28.6, 23.8. IR (KBr, cm^{-1}): ν = 3362, 3163, 2943, 2915, 1647, 1603, 1450, 1248, 679.

4g: Light yellow solid. m.p.110-112°C. ^1H NMR (500 MHz, CDCl_3): δ = 2.32 (t, J = 7.0 Hz, 2H), 2.42 (s, 3H), 2.98 (t, J = 7.0 Hz, 2H), 3.12 (t, J = 7.0 Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ = 190.8, 181.5, 117.6, 105.6, 30.0 (2C), 29.2, 23.5. IR (KBr, cm^{-1}): ν = 2924, 2195, 1646, 1406, 1258, 595.

Precursors **4a-e** and **5a-e** were synthesized in good yields according to the literature: John M. M.; Stephen R. S. *Tetrahedron* **1997**, 53, 17151-17162.

5f and **5g** were prepared by reduction of Mg in methanol and used directly to conduct the aminolysis reaction without further purification.

IV. Synthesis and the analytical data of products 3a-b and 6a-g.

General Procedure for the preparation of **3a**, **3b** and **6a-g** (with **3ad** as an example): Added Cu(OAc)₂·H₂O (0.10 g, 0.5 mmol) to the mixture of **2a** (0.309 g, 1.0 mmol) and n-butylamine (0.15 mL, 1.5 mmol) in THF(10 mL). The reaction mixture was stirred for 1 h at 50 °C and then filtrated, washed with CH₂Cl₂. The filtrate was concentrated in vacuo and purified by column chromatography over silica gel using EtOAc as eluent to give product **3ad** (0.263g, 96%).

3aa: White solid. ¹H NMR (400 MHz, DMSO): δ = 2.94 (m, 1H), 3.24 (dd, *J* = 3.6, 14.8 Hz, 1H), 4.96 (dd, *J* = 3.6, 9.2 Hz, 1H), 7.25 (dd, *J* = 4.8, 7.6 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.68 (dd, *J* = 9.2, 17.2 Hz, 0.6H), 7.72 (td, *J* = 2.0, 7.6 Hz, 1H), 7.83 (dd, *J* = 9.2, 17.2 Hz, 0.4H), 8.49 (d, *J* = 4.4 Hz, 1H), 8.97 (d br, 0.4H), 9.40 (d br, 0.6H), 9.55 (d br, 1H). ¹³C NMR (100 MHz, DMSO): δ = 196.5, 194.9, 171.7, 171.2, 156.8, 155.5, 154.6, 149.0, 136.4, 123.7, 121.8, 91.5, 91.0, 81.3, 80.1, 39.5. IR (KBr, cm⁻¹): ν = 3316, 1732, 1663, 1567, 1441, 1336, 1066. MS (EI): calcd *m/z* 218.07, found 219.2 [(M+1)]⁺; Anal. calcd for C₁₁H₁₀N₂O₃: C, 60.55; H, 4.62; N, 12.84; Found: C, 60.62; H, 4.67; N, 12.79.

3ab: White solid. ¹H NMR (500 MHz, CDCl₃): δ = 3.09 (m, 1H), 3.27 (m, 3H), 3.45 (m, 1H), 5.07 (m, 1H), 7.15 (m, 1H), 7.24 (m, 1H), 7.62 (m, 1H), 7.66 (d, *J* = 14.5 Hz, 0.6H), 7.79 (d, *J* = 15.0 Hz, 0.4H), 8.54 (m, 1H), 8.86 (br, 0.4H), 9.60 (br, 0.6H). ¹³C NMR (100 MHz, CDCl₃): δ = 198.6, 194.8, 173.9, 171.0, 157.3, 156.1, 149.2, 136.4, 124.0, 122.0, 92.8, 92.0, 82.7, 80.9, 39.9, 36.8, 36.6. IR (KBr, cm⁻¹): ν = 3446, 3246, 3210, 1739, 1660, 1641, 1594, 1504, 1352. MS (EI): calcd *m/z* 232.08, found 233.3 [(M+1)]⁺; Anal. calcd for C₁₂H₁₂N₂O₃: C, 62.06; H, 5.21; N, 12.06; Found: C, 62.21;

H, 5.33; N, 12.03.

3ac: White solid. ^1H NMR (300 MHz, CDCl_3): δ = 1.35 (m, 3H), 3.07 (m, 1H), 3.42 (m, 1H), 3.51 (m, 2H), 5.04 (m, 1H), 7.14 (m, 1H), 7.22 (m, 1H), 7.60 (m, 1H), 7.70 (d, J = 14.7 Hz, 0.6H), 7.82 (d, J = 15.0 Hz, 0.4H), 8.53 (m, 1H), 8.95 (br, 0.4H), 9.66 (br, 0.6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 198.4, 194.9, 173.8, 171.2, 156.4, 156.2, 155.5, 154.3, 149.3, 136.4, 124.1, 123.9, 121.8, 92.4, 91.6, 82.6, 80.9, 45.3, 45.1, 39.9, 39.8, 15.3. IR (KBr, cm^{-1}): ν = 3255, 2984, 1741, 1663, 1594, 1494. MS (EI): calcd m/z 246.1, found 247.3 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_3$: C, 63.40; H, 5.73; N, 11.38; Found: C, 63.35; H, 5.86; N, 11.47.

3ad: White solid. ^1H NMR (600 MHz, CDCl_3): δ = 0.97 (m, 3H), 1.40 (m, 2H), 1.66 (m, 2H), 3.10 (m, 1H), 3.45 (m, 3H), 5.07 (s, 1H), 7.17 (s, 1H), 7.27 (s, 1H), 7.64 (s, 1H), 7.67 (d, J = 14.4 Hz, 0.6 H), 7.80 (d, J = 15.0 Hz, 0.4H), 8.55 (s, 1H), 8.93 (br, 0.4H), 9.65 (br, 0.6H). ^{13}C NMR (100 MHz, CDCl_3): δ = 198.3, 194.8, 173.7, 171.1, 156.4, 156.2, 156.0, 154.8, 149.1, 136.3, 124.0, 121.8, 92.3, 91.5, 82.5, 80.8, 50.3, 50.1, 39.9, 31.8, 19.3, 13.4. IR (KBr, cm^{-1}): ν = 3446, 2961, 1741, 1664, 1561, 1495. MS (EI): calcd m/z 274.13, found 275.2 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_3$: C, 65.68; H, 6.61; N, 10.21; Found: C, 65.59; H, 6.67; N, 10.16.

3ae: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): δ = 3.06 (m, 1H), 3.41 (m, 1H), 4.59 (s, 2H), 5.05 (m, 1H), 7.13 (t, J = 6.0 Hz, 1H), 7.22 (m, 3H), 7.38 (m, 3H), 7.58 (t, J = 8.0 Hz, 1H), 7.76 (d, J = 13.0 Hz, 0.6H), 7.89 (d, J = 14.0 Hz, 0.4H), 8.51 (m, 1H), 9.24 (br, 0.4H), 9.92 (br, 0.6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 198.2, 194.8, 173.4, 170.9, 156.2, 156.0, 155.6, 154.5, 149.2, 149.1, 136.2, 133.9, 129.1 (2C), 128.7, 127.7 (2C), 123.9, 123.7, 121.6, 92.7, 92.0, 82.5, 80.8, 53.9, 53.7, 39.7. IR (KBr, cm^{-1}): ν = 3253, 3062, 2938, 1742, 1664, 1629, 1490, 1341. MS (EI): calcd m/z 308.12, found 309.1 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_4$: C, 70.12; H, 5.23; N, 9.09; Found: C, 70.18; H, 5.31; N, 9.04.

3af: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 1.26 (t, J = 6.5 Hz, 3H), 1.35 (t, J = 7.0 Hz, 3H), 3.06 (dd, J = 9.0, 14.0 Hz, 1H), 3.43 (d, J = 14.5 Hz, 1H), 3.54 (q, J = 7.0 Hz, 2H), 4.22 (m, 2H), 4.92 (br, 0.75H), 5.03 (br, 0.25H), 7.12 (s, 1H), 7.24 (d, J = 7.5 Hz, 1H), 7.38 (br, 0.75H), 7.51 (br, 0.25H), 7.59 (t, J = 7.5 Hz, 1H), 8.52 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ = 192.1, 174.4, 156.5, 154.2, 153.6, 149.1, 136.3, 124.0, 121.6, 90.0, 80.8, 52.9, 48.7, 47.7, 40.2, 14.4, 13.8. IR (KBr, cm^{-1}): ν = 2977, 2932, 1739, 1666, 1603, 1452, 1402, 1067. MS (EI): calcd m/z 274.13, found 275.2 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_3$: C, 65.68; H, 6.61; N, 10.21; Found: C, 65.66; H, 6.69; N, 10.31.

3ag: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 3.04 (dd, J = 8.5, 14.5 Hz, 1H), 3.40 (dd, J = 3.0, 14.5 Hz, 1H), 3.65 (s, 2H), 3.83 (s, 2H), 3.84 (s, 2H), 4.57 (s, 2H), 4.94 (dd, J = 3.5, 8.5 Hz, 0.75H), 5.07 (d, J = 6.5 Hz, 0.25H), 7.13 (t, J = 5.5 Hz, 1H), 7.22 (d, J = 8.0 Hz, 1H), 7.38 (s, 0.75H), 7.51 (s, 0.25H), 7.59 (t, J = 8.0 Hz, 1H), 8.51 (d, J = 3.5 Hz, 1H). ^{13}C NMR (125 MHz, DMSO): δ = 192.8, 173.9, 156.3, 153.6, 153.0, 148.9, 136.8, 124.3, 124.2, 122.0, 90.4, 81.8, 80.9, 67.0, 66.8, 56.8, 56.6, 53.5, 52.4, 40.1. IR (KBr, cm^{-1}): ν = 2960, 1748, 1669, 1609, 1578, 1436, 1334, 1059, 1003. MS (EI): calcd m/z 288.11, found 289.1 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_4$: C, 62.49; H, 5.59; N, 9.72; Found: C, 62.41; H, 5.66; N, 9.64.

3ah: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): δ = 3.18 (m, 1H), 3.49 (dd, J = 15.5, 18.0 Hz, 1H), 5.16 (m, 1H), 7.16 (s, 1H), 7.25 (m, 3H), 7.30 (d, J = 7.5 Hz, 1H), 7.45 (t, J = 7.5 Hz, 2H), 7.63 (t, J = 7.5 Hz, 1H), 8.19 (d, J = 14.0 Hz, 0.6H), 8.32 (d, J = 14.0 Hz, 0.4H), 8.54 (s, 1H), 10.57 (d, J = 13.5 Hz, 0.4H), 11.30 (d, J = 13.0 Hz, 0.6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 198.8, 195.2, 173.2, 170.4, 156.0, 155.8, 149.3, 148.2, 147.0, 137.2, 136.4, 130.2 (2C), 127.3, 127.1, 124.2, 123.9, 121.9, 117.8, 117.7, 94.8, 94.2, 83.0, 81.1, 39.7, 39.6. IR (KBr, cm^{-1}): ν = 3439, 3058, 1747, 1673, 1630, 1588, 1484, 1445, 1315, 1210. MS (EI): calcd m/z 294.1, found 295.1 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_3$: C, 69.38; H, 4.79; N, 9.52; Found: C, 69.47; H, 4.86; N, 9.39.

3ai: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): δ = 2.37 (s, 3H), 3.16 (m, 1H), 3.49 (m, 1H), 5.15 (m, 1H), 7.14 (m, 3H), 7.25 (m, 3H), 7.62 (t, J = 7.5 Hz, 1H), 8.15 (d, J = 11.5 Hz, 0.6H), 8.28 (d, J = 12.5 Hz, 0.4H), 8.55 (t, J = 5.5 Hz, 1H), 10.55 (d,

$J = 13.0$ Hz, 0.4 H), 11.28 (s, 0.6H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 198.7, 195.2, 173.3, 170.6, 156.1, 155.9, 149.4, 149.3, 148.1, 147.0, 137.5, 137.3, 136.4, 134.8, 130.7(2\text{C}), 124.2, 123.9, 121.9, 121.8, 117.7(2\text{C}), 94.4, 93.7, 82.9, 81.1, 39.8, 39.7, 20.9$. IR (KBr, cm^{-1}): $\nu = 3404, 1747, 1667, 1633, 1587, 1487, 1316$. MS (EI): calcd m/z 308.12, found 309.1 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_3$: C, 70.12; H, 5.23; N, 9.09; Found: C, 70.22; H, 5.36; N, 9.03.

3aj: White solid. ^1H NMR (400 MHz, DMSO): $\delta = 2.92$ (m, 1H), 3.25 (dd, $J = 3.2, 14.8$ Hz, 1H), 3.52 (dd, $J = 4.8, 9.2$ Hz, 4H), 4.95 (m, 2H), 7.25 (t, $J = 6.8$ Hz, 1H), 7.31 (d, $J = 7.6$ Hz, 1H), 7.73 (t, $J = 7.6$ Hz, 1H), 7.79 (d, $J = 15.2$ Hz, 0.6H), 7.92 (d, $J = 15.6$ Hz, 0.4H), 8.50 (d, $J = 4.0$ Hz, 1H), 9.48 (br, 0.4H), 9.45 (br, 0.6H). ^{13}C NMR (100 MHz, CDCl_3): $\delta = 196.3, 194.4, 172.2, 171.2, 156.9, 156.8, 156.0, 149.0, 136.5, 136.2, 123.8, 121.9, 90.6, 90.0, 81.5, 80.1, 59.7, 59.5, 52.2, 39.6$. IR (KBr, cm^{-1}): $\nu = 3220, 2927, 1724, 1672, 1629, 1489, 1444, 1350, 1064$. MS (EI): calcd m/z 262.1, found 263.2 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_4$: C, 59.54; H, 5.38; N, 10.68; Found: C, 59.47; H, 5.50; N, 10.72.

3ba: White solid. ^1H NMR (300 MHz, DMSO): $\delta = 2.85$ (dd, $J = 7.5, 14.7$ Hz, 1H), 3.13 (dd, $J = 3.6, 14.7$ Hz, 1H), 4.84 (dd, $J = 3.6, 7.8$ Hz, 1H), 7.24 (s, 2H), 7.62 (dd, $J = 9.0, 17.1$ Hz, 0.6H), 7.78 (dd, $J = 9.0, 17.1$ Hz, 0.4H), 8.45 (s, 2H), 8.93 (d br, 0.4H), 9.38 (d br, 0.6H), 9.55 (br, 1H). ^{13}C NMR (100 MHz, DMSO): $\delta = 196.0, 194.4, 171.5, 171.0, 155.6, 154.7, 149.3$ (2C), 145.5, 124.8 (2C), 91.5, 91.1, 81.1, 79.8, 36.0. IR (KBr, cm^{-1}): $\nu = 3319, 1738, 1666, 1609, 1576, 1433, 1334, 1058$. MS (EI): calcd m/z 218.07, found 219.3 $[(\text{M}+1)]^+$.

3bb: White solid. ^1H NMR (300 MHz, CDCl_3): δ = 2.99 (m, 1H), 3.23 (m, 4H), 4.77 (dd, J = 3.6, 6.6 Hz, 1H), 7.19 (d, J = 4.0 Hz, 2H), 7.59 (d, J = 14.7 Hz, 0.5H), 7.70 (d, J = 15.0 Hz, 0.5H), 8.49 (d, J = 4.0 Hz, 2H), 8.83 (br, 0.5H), 9.49 (br, 0.5H). ^{13}C NMR (100 MHz, CDCl_3): δ = 197.5, 193.8, 173.4, 170.6, 157.5, 156.0, 149.5 (2C), 144.7, 125.2 (2C), 92.7, 91.8, 82.2, 80.5, 36.9, 36.7, 36.5, 36.4. IR (KBr, cm^{-1}): ν = 3272, 1746, 1662, 1635, 1498, 1457. MS (EI): calcd m/z 232.08, found 233.2 $[(\text{M}+1)]^+$.

3bc: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 1.35 (m, 3H), 3.00 (m, 1H), 3.25 (m, 1H), 3.51 (m, 2H), 4.77 (dd, J = 4.0, 7.5 Hz, 1H), 7.21 (d, J = 6.0 Hz, 2H), 7.64 (d, J = 14.5 Hz, 0.5H), 7.75 (d, J = 14.5 Hz, 0.5H), 8.50 (d, J = 5.5 Hz, 2H), 8.93 (br, 0.5H), 9.60 (br, 0.5H). ^{13}C NMR (125 MHz, CDCl_3): δ = 197.5, 193.9, 173.5, 170.7, 155.6, 154.2, 149.7 (2C), 144.5, 124.9, 124.8, 92.5, 91.7, 82.3, 80.6, 45.3, 45.2, 36.5, 36.4, 15.2, 15.1. IR (KBr, cm^{-1}): ν = 3263, 1747, 1661, 1603, 1492, 1376, 1343, 1137, 1059.

3bd: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 0.95 (m, 3H), 1.36 (m, 2H), 1.63 (m, 2H), 2.97 (m, 1H), 3.23 (m, 1H), 3.42 (m, 2H), 4.75 (m, 1H), 7.21 (s, 2H), 7.59 (d, J = 14.5 Hz, 0.5H), 7.71 (d, J = 15.0 Hz, 0.5H), 8.53 (br, 2H), 8.90 (br, 0.5H), 9.57 (br, 0.5H). ^{13}C NMR (100 MHz, CDCl_3): δ = 197.5, 194.0, 173.4, 170.8, 156.2, 154.8, 149.6 (2C), 144.7, 124.9 (2C), 92.4, 91.6, 82.2, 80.5, 50.4, 50.3, 36.6, 36.5, 31.8, 19.4, 13.4. IR (KBr, cm^{-1}): ν = 3259, 2960, 2874, 1732, 1665, 1640, 1601, 1491, 1372. MS

(EI): calcd m/z 274.13, found 275.1 $[(M+1)]^+$.

3be: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): δ = 2.95 (m, 1H), 3.21 (m, 1H), 4.58 (s, 2H), 4.74 (dd, J = 4.0, 7.0 Hz, 1H), 7.17 (t, J = 5.0 Hz, 2H), 7.21 (m, 2H), 7.38 (m, 3H), 7.68 (s, 0.5H), 7.79 (s, 0.5H), 8.47 (t, J = 5.0 Hz, 2H), 9.26 (br, 0.5H), 9.77 (br, 0.5H). ^{13}C NMR (125 MHz, CDCl_3): δ = 197.3, 193.9, 173.0, 170.5, 155.8, 154.5, 149.5 (2C), 144.4, 144.3, 133.7, 129.1 (2C), 128.8, 127.6 (2C), 124.7, 124.6, 92.7, 91.9, 82.2, 80.4, 53.9, 53.7, 36.3. IR (KBr, cm^{-1}): ν = 3282, 3030, 1722, 1663, 1626, 1490, 1376. MS (EI): calcd m/z 308.12, found 309.2 $[(M+1)]^+$.

3bf: White solid. ^1H NMR (300 MHz, CDCl_3): δ = 1.19 (t, J = 6.9 Hz, 3H), 1.32 (t, J = 7.2 Hz, 3H), 2.96 (dd, J = 6.9, 14.4 Hz, 1H), 3.21 (dd, J = 3.6, 14.4 Hz, 1H), 3.50 (q, J = 7.2 Hz, 2H), 4.14 (d br, 2H), 4.66 (br, 1H), 7.18 (d, J = 5.7 Hz, 2H), 7.30 (br, 1H), 8.46 (d, J = 5.4 Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ = 191.1, 174.0, 154.1, 149.4 (2C), 144.6, 124.8 (2C), 89.9, 80.3, 52.9, 48.5, 36.6, 14.2, 13.6. IR (KBr, cm^{-1}): ν = 3412, 3036, 1754, 1663, 1596, 1448, 1389.

3bg: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 2.97 (dd, J = 7.5, 14.5 Hz, 1H), 3.24 (d, J = 14.0 Hz, 1H), 3.64 (s, 2H), 3.84 (d, J = 4.0 Hz, 2H), 4.52 (s, 2H), 4.69 (s, 0.75H), 4.81 (s, 0.25H), 7.21 (d, J = 5.0 Hz, 2H), 7.31 (s, 0.75H), 7.42 (s, 0.25H),

8.50 (d, $J = 5.0$ Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 191.8, 173.5, 153.7, 153.0, 148.9$ (2C), $145.7, 125.3$ (2C), $90.4, 81.4, 80.6, 67.0, 66.7, 56.9, 56.7, 53.6, 52.5, 37.0$. IR (KBr, cm^{-1}): $\nu = 2983, 2938, 2875, 1727, 1661, 1598, 1482, 1448, 1406, 1108, 1067$.

3bh: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): $\delta = 3.06$ (dd, $J = 7.0, 14.5$ Hz, 1H), **3.30 (m, 1H)**, **4.88 (m, 1H)**, 7.23 (m, 4H), 7.32 (dd, $J = 7.5, 15.5$ Hz, 1H), 7.46 (dd, $J = 7.5, 15.5$ Hz, 1H), 8.13 (d, $J = 14.5$ Hz, 0.25H), 8.24 (d, $J = 14.5$ Hz, 0.75H), 8.52 (d, $J = 5.5$ Hz, 2H), 10.54 (d, $J = 14.5$ Hz, 0.75H), 11.22 (d, $J = 11.5$ Hz, 0.25H). ^{13}C NMR (100 MHz, CDCl_3): $\delta = 197.7, 194.2, 172.7, 170.0, 149.8, 149.7, 148.7, 147.4, 144.3, 144.2, 137.1, 130.1$ (2C), $127.4, 127.3, 124.8, 124.7, 118.1, 118.0, 117.9, 94.5, 93.8, 82.6, 80.8, 36.5, 36.4$. IR (KBr, cm^{-1}): $\nu = 3227, 3058, 1753, 1676, 1633, 1589, 1489, 1446, 1333, 1208$. MS (EI): calcd m/z 294.1, found 295.2 $[(M+1)]^+$.

3bi: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): $\delta = 2.36$ (s, 3H), 3.04 (m, 1H), 3.28 (m, 1H), 4.85 (m, 1H), 7.11 (t, $J = 9.5$ Hz, 2H), 7.24 (m, 4H), 8.01 (d, $J = 14.0$ Hz, 0.5H), 8.19 (d, $J = 14.5$ Hz, 0.5H), 8.51 (d, $J = 5.0$ Hz, 2H), 10.51 (d, $J = 14.0$ Hz, 0.5H), 11.21 (d, $J = 13.0$ Hz, 0.5H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 197.5, 194.1, 172.8, 170.1, 149.6$ (2C), $148.3, 147.1, 144.1, 144.0, 137.7, 137.5, 134.4, 130.5$ (2C), 124.7 (2C), 117.7 (2C), $94.1, 93.3, 82.4, 80.6, 36.3, 20.8$. IR (KBr, cm^{-1}): $\nu = 3218, 3031, 1733, 1677, 1635, 1603, 1587, 1488, 1314$.

3bj: White solid. ^1H NMR (500 MHz, DMSO): δ = 2.85 (dd, J = 8.5, 14.5Hz, 1H), 3.15 (dd, J = 3.5, 14.5Hz, 1H), 3.47 (s, 2H), 3.51 (s, 2H), 4.87 (dd, J = 3.5, 8.0Hz, 1H), 4.97 (d, J = 4.0Hz, 1H), 7.27 (d, J = 4.5Hz, 2H), 7.74 (s, 0.6H), 7.89 (s, 0.4H), 8.46 (d, J = 5.0Hz, 2H), 9.50 (br, 0.4H), 9.94 (br, 0.6H). ^{13}C NMR (100 MHz, DMSO): δ = 195.7, 193.8, 172.0, 171.1, 156.8, 156.0, 149.3 (2C), 145.6, 124.8 (2C), 90.6, 90.1, 81.2, 79.8, 59.4, 52.3, 52.2, 36.2, 36.1. IR (KBr, cm^{-1}): ν = 3265, 1746, 1666, 1608, 1494, 1459, 1051. MS (EI): calcd m/z 262.1, found 263.1 $[(\text{M}+1)]^+$.

6a: White solid, m.p. 124-126 °C. ^1H NMR (500 MHz, CDCl_3): δ = 0.92 (t, J = 7.5 Hz, 3H), 1.36 (m, 2H), 1.61 (m, 2H), 1.94 (m, 2H), 2.43 (t, J = 6.5 Hz, 2H), 2.46 (t, J = 6.5 Hz, 2H), 3.38 (q, J = 7.0 Hz, 2H), 8.12 (d, J = 14.0 Hz, 1H), 11.16 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ = 199.6, 196.5, 158.8, 108.4, 50.1, 37.6, 37.2, 32.1, 19.9, 19.5, 13.4. IR (KBr, cm^{-1}): ν = 3243, 2941, 1682, 1612, 1543, 1428.

6b: White solid, m.p. 156-158 °C. ^1H NMR (500 MHz, CDCl_3): δ = 0.94 (t, J = 7.5 Hz, 3H), 1.04 (s, 6H), 1.38 (m, 2H), 1.63 (m, 2H), 2.32 (s, 2H), 2.36 (s, 2H), 3.40 (q, J = 6.5 Hz, 2H), 8.10 (d, J = 14.0 Hz, 1H), 11.11 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ = 198.9, 196.0, 158.3, 107.2, 51.3, 50.9, 50.0, 32.1, 31.1, 28.4 (2C), 19.5, 13.4. IR (KBr, cm^{-1}): ν = 3227, 2952, 2884, 1668, 1653, 1583, 1559, 1540, 1437. MS (EI):

calcd m/z 195.13, found 196.1 $[(M+1)]^+$; Anal. calcd for $C_{11}H_{17}NO_2$: C, 67.66; H, 8.78; N, 7.17; Found: C, 67.74; H, 8.86; N, 7.13.

6c: Light yellow solid, m.p. 64-66 °C. 1H NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, J = 7.5 Hz, 3H), 1.33 (m, 2H), 1.55 (m, 2H), 2.19 (s, 3H), 2.40 (s, 3H), 3.30 (m, 2H), 7.69 (d, J = 13.0 Hz, 1H), 11.01 (br, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 200.1, 194.2, 160.0, 111.2, 50.1, 32.5, 31.8, 27.3, 19.6, 13.5. IR (KBr, cm^{-1}): ν = 3184, 2962, 2936, 2875, 1653, 1616, 1587, 1398, 1314, 1255. MS (EI): calcd m/z 183.13, found 184.1 $[(M+1)]^+$; Anal. calcd for $C_{10}H_{17}NO_2$: C, 65.54; H, 9.35; N, 7.64; Found: C, 65.60; H, 9.47; N, 7.59.

6d: Light yellow liquid. 1H NMR (500 MHz, $CDCl_3$): δ = 0.87 (t, J = 7.0 Hz, 3H), 1.23 (t, J = 7.0 Hz, 3H), 1.30 (m, 2H), 1.53 (m, 2H), 2.39 (s, 3H), 3.27 (q, J = 6.5 Hz, 2H), 4.11 (q, J = 7.0 Hz, 2H), 7.91 (d, J = 14.0 Hz, 1H), 10.98 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 198.9, 167.1, 159.7, 99.7, 59.2, 49.6, 32.4, 30.6, 19.4, 14.3, 13.3. IR (KBr, cm^{-1}): ν = 3187, 2960, 2934, 2874, 1699, 1636, 1582, 1418.

6e: colorless liquid. 1H NMR (500 MHz, $CDCl_3$): δ = 0.91 (t, J = 7.0 Hz, 3H), 1.25 (t, J = 7.0 Hz, 3H), 1.30 (t, J = 7.0 Hz, 3H), 1.35 (m, 2H), 1.56 (m, 2H), 3.30 (q, J = 6.5 Hz, 2H), 4.14 (q, J = 7.0 Hz, 2H), 4.20 (q, J = 7.0 Hz, 2H), 7.96 (d, J = 14.0 Hz, 1H),

9.19 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ = 169.3, 166.1, 159.9, 89.1, 59.6, 59.4, 49.3, 32.6, 19.5, 14.3, 14.2, 13.4. IR (KBr, cm^{-1}): ν = 3276, 2960, 2875, 1684, 1652, 1464, 1428, 1378.

6f: Light yellow solid. ^1H NMR (500 MHz, CDCl_3): δ = 0.95 (t, J = 7.5 Hz, 3H), 1.39 (m, 2H), 1.61 (m, 2H), 2.24 (s, 3H), 3.34 (q, J = 6.5 Hz, 2H), 5.38 (s, 1H), 7.73 (d, J = 18.0 Hz, 1H), 9.31 (s, 1H), 10.70 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ = 194.2, 171.5, 160.4, 101.2, 49.9, 32.5, 25.7, 19.6, 13.5. IR (KBr, cm^{-1}): ν = 3385, 2958, 1647, 1616, 1551, 1410, 1327. MS (EI): calcd m/z 184.12, found 185.1 $[(\text{M}+1)]^+$; Anal. calcd for $\text{C}_9\text{H}_{16}\text{N}_2\text{O}_2$: C, 58.67; H, 8.75; N, 15.21; Found: C, 58.59; H, 8.84; N, 15.32.

6g: White solid. ^1H NMR (500 MHz, CDCl_3): δ = 0.94 (t, J = 3.0, 7.5 Hz, 3H), 1.36 (m, 2H), 1.59 (m, 2H), 2.30 (d, J = 3.5 Hz, 3H), 3.32 (q, J = 6.5 Hz, 2H), 7.27 (d, J = 14.0 Hz, 1H), 10.56 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ = 196.6, 158.8, 120.5, 81.6, 49.8, 32.2, 28.0, 19.4, 13.4. IR (KBr, cm^{-1}): ν = 3233, 2956, 2874, 216, 1684, 1653, 1395, 1300, 1225.

V. Copies of ^1H and ^{13}C NMR spectra for compounds 2a-b, 3a-b and 6a-g.

new experiment
 Archive directory: /export/home/pan/vmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: DMSO
 Ambient temperature
 INOVA-500 "HRENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.999 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.8949580 MHz
 DATA PROCESSING
 FT size 131072
 Total time 9 min, 24 sec

Current Data Parameters
 NAME SD-L100un
 EXPNO 297
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030717
 Time 21:12
 INSTRUM av400
 PROBNM 5 mm DUL 13C-1
 PULPROG zgpg
 TO 16384
 SOLVENT DMSO
 NS 512
 DS 2
 SMH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 4096
 DW 19.950 usec
 DE 6.50 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 2.30 usec
 PL1 -4.00 dB
 SFO1 100.5785294 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.20 dB
 SFO2 399.9515998 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5679540 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 21.00 cm
 CY 4.00 cm
 F1P 217.690 ppm
 F1 21892.32 Hz
 F2P -0.829 ppm
 F2 -83.32 Hz
 PRNCH 10.40563 ppm/cm
 HZDM 1046.46875 Hz/cm

new experiment
 Archive directory: /export/home/pan/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: DMSO
 Ambient temperature
 INOVA-500 "HENS500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.999 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.8049580 MHz
 DATA PROCESSING
 FT size 131072
 Total time 0 min, 24 sec

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "HENS500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.0 Hz
 128 repetitions
 OBSERVE C13, 125.6761083 MHz
 DECOUPLE H1, 499.8050805 MHz
 Power 48 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

mistakesvf92

Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NMR500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 7996.8 Hz
16 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec

3ab

3ab

Current Data Parameters
NAME 50-Liugu
EXPNO 277
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030703
Time 10.28
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 31852
DS 2
SWH 25062.656 Hz
FIDRES 1.529703 Hz
AQ 0.3268108 sec
RG 4096
DN 19.950 usec
DE 8.50 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 2.80 usec
PL1 -1.00 dB
SFO1 100.5785294 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 19.50 dB
SFO2 399.9515998 MHz

F2 - Processing parameters
SI 65536
SF 100.5675099 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
CY 15.00 cm
F1P 206.300 ppm
F1 20747.27 Hz
F2P 1.863 ppm
F2 187.39 Hz
PPMCH 9.73517 ppm/cm
HZCH 979.04169 Hz/cm

mistakesvf92

Archive directory: /export/home/pan/vnmrsvs/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NERUS50"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
F1 size 65536
Total time 0 min, 20 sec

3af

3af

Current Data Parameters
NAME SD-LiuQun
EXPNO 273
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030701
Time 14.58
INSTRUM in4000
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 16384
SOLVENT CDCl3
NS 2073
DS 2
SWH 25062.656 Hz
FIDRES 1.529703 Hz
AQ 0.3269108 sec
RG 4096
DW 19.950 usec
DE 6.50 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.80 usec
PL1 -3.00 dB
SFO1 100.5785294 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 10.50 dB
SFO2 399.9615998 MHz

F2 - Processing parameters
SI 65536
SF 100.575194 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
CY 8.00 cm
F1P 200.000 ppm
F1 20113.50 Hz
F2P 0.000 ppm
F2 0.00 Hz
PWCW 9.52381 ppm/cm
HZCW 957.78589 Hz/cm

S24

```
STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
INOVA-500 "NMRUS-500"
```

28ssg93-4ylan-1213

Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

INNOVA-500 "HREX500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.032 sec
Width 7996.0 Hz
100 repetitions
OBSERVE F1: 499.8025838 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec

3bc

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp: 7.5 C / 280.6 K

User: 1-14-87

File: b615-c13

INNOVA-500 "HREX500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
7800 repetitions
OBSERVE C13, 125.6754733 MHz
DECOUPLE H1, 499.8059805 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 hr, 29 min, 25 sec

3bc

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/pan/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 20.0 C / 293.1 K
 File: c187
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.989 sec
 Width 8000.0 Hz
 64 repetitions
 OBSERVE H1: 499.8025914 MHz
 DATA PROCESSING
 FT size 131072
 Total time 2 min, 59 sec

Current Data Parameters
 NAME 50-Liu0un
 EXPNO 274
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030701
 Time 15:57
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpgc
 TD 16384
 SOLVENT CDCl3
 NS 1718
 DS 2
 SWH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 4096
 OW 19.950 usec
 OE 6.50 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 2.80 usec
 PL1 -3.00 dB
 SFO1 100.5785294 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SFO2 399.9515998 MHz

F2 - Processing parameters
 S1 65536
 SF 100.5675152 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 21.00 cm
 CY 8.00 cm
 FIP 208.501 ppm
 F1 20988.47 Hz
 F2 0.084 ppm
 F2 68.76 Hz
 PPMCM 9.89608 ppm/cm
 HZCM 995.22406 Hz/cm

mistakesvf92

Archive directory: /export/home/pan/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient Temperature
File: c100
INOVA-500 "NMRUS00"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 7996.8 Hz
15 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
INOVA-500 "NMRUS00"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31821.0 Hz
640 repetitions
OBSERVE C13, 125.6754840 MHz
DECOUPLE H1, 499.8050905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-300BB ~JLUS88"
 Relax. delay 3.000 sec
 Pulse 45.0 degrees
 Acq. time 2.566 sec
 Width 6002.4 Hz
 8 repetitions
 OBSERVE H1, 300.0732340 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min, 47 sec

3bf

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 7800 repetitions
 OBSERVE C13, 125.6754940 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 48 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 4 hr, 29 min, 25 sec

3bf

mistakesvf92
 Archive directory: /export/home/pan/vnmr/sys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvents: CDCl3
 Ambient temperature
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 7996.8 Hz
 16 repetitions
 OBSERVE H1, 499.8025891 MHz
 DATA PROCESSING
 FT size 45536
 Total time 0 min, 40 sec

Current Data Parameters
 NAME 50-Liu0un
 EXPNO 275
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030701
 Time 16.31
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg
 TD 16384
 SOLVENT DMSO
 NS 7751
 DS 2
 SWH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 4096
 DW 19.950 usec
 DE 6.50 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 2.00 usec
 PL1 -3.00 dB
 SF01 100.5785294 MHz

***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SF02 399.9515998 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5673110 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 21.00 cm
 CY 7.00 cm
 F1P 201.222 ppm
 F1 20236.37 Hz
 F2P 2.978 ppm
 F2 299.52 Hz
 PPMCH 9.44016 ppm/cm
 HZCM 949.37372 Hz/cm

mistakesvf82

Archive directory: /export/home/pan/vnmr/sys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

INNOVA-500 "MENU500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.500 sec

Width 7396.8 Hz

8 repetitions

OBSERVE H1, 499.8025891 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 20 sec

3bh

3bh

Current Data Parameters

NAME SD-LiuJun

EXPNO 316

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030729

Time 9.16

INSTRUM av400

PROBHD 5 mm DUL 13C-1

PULPROG zgpg

TD 16384

SOLVENT CDCl3

NS 1622

DS 2

SWH 25062.656 Hz

FIDRES 1.529703 Hz

AQ 0.3269108 sec

RG 4096

DW 19.950 usec

DE 6.50 usec

TE 300.0 K

D1 0.69999999 sec

d11 0.03000000 sec

***** CHANNEL f1 *****

NUC1 13C

P1 2.30 usec

PL1 -4.00 dB

SFO1 100.5785294 MHz

***** CHANNEL f2 *****

CPOPRG2 waltz16

NUC2 1H

POPRG2 80.00 usec

PL2 -2.00 dB

PL12 18.20 dB

SFO2 399.9515998 MHz

F2 - Processing parameters

SF 65536

SF 100.5675213 MHz

WDW EM

SSB 0

LB 2.00 Hz

GB 0

PC 1.00

1D NMR plot parameters

CA 22.00 cm

CY 10.00 cm

F1P 213.826 ppm

F1 21503.00 Hz

F2P -1.587 ppm

F2 -157.59 Hz

PPHMC 9.79057 ppm/cm

HZCM 984.61340 Hz/cm

mistakesvf92
 Archive directory: /export/home/pan/vnmrsvs/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 7586.8 Hz
 8 repetitions
 OBSERVE H1, 499.8025891 MHz
 DATA PROCESSING
 FT size 65536
 Total time 9 min, 20 sec

3bi

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/fang/vnmrsvs/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 1280 repetitions
 OBSERVE C13, 125.6754940 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 48 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

3bi

STANDARD PROTON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: PROTON

Pulse Sequence: s2pul

Solvent: DMSO
 Temp: 25.0 C / 298.1 K
 INOVA-500 "MUS500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7596.8 Hz

8 repetitions

OBSERVE H1, 499.8049627 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS

Archive directory: /export/home/liu/vnmrsys/data
 Sample directory:
 File: CARBON

Pulse Sequence: s2pul

Solvent: DMSO

Temp: 7.5 C / 280.6 K

User: 1-14-87

INOVA-500 "MUS500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.390 sec

Width 31421.8 Hz

128 repetitions

OBSERVE C13, 125.6760285 MHz

DECOUPLE H1, 499.8074646 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 3 hr, 12 min, 26 sec

new experiment
 Archive directory: /export/home/pan/vmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.389 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.8025923 MHz
 DATA PROCESSING
 FT size 131072
 Total time 9 min, 24 sec

new experiment
 Archive directory: /export/home/fang/vmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-97
 File: f222-c13
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 784 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.8056905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

mistakesvf92

Archive directory: /export/home/pan/vnmrsvs/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
File: c364
INNOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 7996.6 Hz
8 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 20 sec

6a

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsvs/data
Sample directory:
File: C3600N
Pulse Sequence: s2pu1
Solvent: CDC13
Temp. 7.5 C / 280.6 K
User: 1-14-87
INNOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.6 Hz
256 repetitions
OBSERVE C13, 125.6754733 MHz
DECOUPLE H1, 499.8025891 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec

6a

mistakesvf92
 Archive directory: /export/home/pan/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NMRUS00"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 7996.8 Hz
 8 repetitions
 OBSERVE H1, 499.8025891 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 20 sec

6b

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "NMRUS00"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 640 repetitions
 OBSERVE C13, 125.6754733 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 48 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

6b

mistakesvf92
 Archive directory: /export/home/pan/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 7350.8 Hz
 8 repetitions
 OBSERVE H1, 499.8025891 MHz
 DATA PROCESSING
 FT size 65536
 Total time 8 min, 20 sec

6d

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 3584 repetitions
 OBSERVE C13, 125.6754733 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 48 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

6d

mistakesvf92

Archive directory: /export/home/pan/vnmr/sys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

INNOVA-500 "HREUS500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.500 sec

Width 7996.8 Hz

8 repetitions

OBSERVE H1, 499.8025891 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 20 sec

6e

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmr/sys/data

Sample directory:

File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 7.5 C / 280.6 K

User: 1-14-87

INNOVA-500 "HREUS500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31421.8 Hz

128 repetitions

OBSERVE C13, 125.6754733 MHz

DECOUPLE H1, 499.8058905 MHz

Power 48 dB

on during acquisition

off during delay

WALTZ-16 modulated

Line broadening 1.0 Hz

FT size 131072

Total time 3 hr, 12 min, 26 sec

6e

mistakesvf92

Archive directory: /export/home/pan/vnmrsvs/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 7996.8 Hz
64 repetitions
OBSERVE N1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 40 sec

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vnmrsvs/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 7.5 C / 280.6 K
User: 1-14-07
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3456 repetitions
OBSERVE C13, 125.8754733 MHz
DECOUPLE N1, 499.8050905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec

VI. Copies of ^1H NMR spectrum for the mixture of 2a and the corresponding aldehyde.

VII. Copies of the HMBC, HMQC and ^1H - ^1H COSY spectra of compound 3aa.