Lactam Synthesis *via* the Intramolecular Hydroamidation of Alkynes Catalyzed by Palladium Complexes

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Preparation of Amido Alkynes: The amido alkynes **1** and **3a-i** were prepared from the commercially available alkynoic acids by a sequence of standard reactions. (See, Pinho, P.; Minnaard, A. J.; Feringa, B. L. *Org. Lett.*, **2003**, 5, 259-261 and references cited therein).

(1). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.69-1.60 (m, 2H), 1.88-1.79 (m, 2H), 2.27 (t, $J = 7.0$ Hz, 2H), 2.43 (t, $J = 7.0$ Hz, 2H), 4.43 (d, $J = 6.0$ Hz, 2H), 5.69 (br s, 1H), 7.37-7.23 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.1, 24.9, 28.1, 35.9, 43.4, 80.9, 89.6, 123.8, 127.3, 127.5, 127.6, 128.1, 128.5, 131.4, 138.3, 172.6; IR (KBr) 3292, 2947, 1720, 1643, 1541, 1209, 758, 690 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}$ ($\text{M}+\text{Na}$) 314.1515, found 314.1514; Anal. Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}$: C, 82.44; H, 7.26; N, 4.81, Found: C, 82.37; H, 7.31; N, 4.75.

(3a). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.58-1.50 (m, 2H), 1.77-1.69 (m, 2H), 2.30 (t, $J = 7.0$ Hz, 2H), 2.35 (t, $J = 7.0$ Hz, 2H), 2.41 (s, 3H), 7.36-7.22 (m, 7H), 7.92 (d, $J = 8.0$ Hz, 2H), 8.24 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.1, 21.8, 23.5, 27.8, 35.8, 81.2, 89.2, 123.6, 127.6, 128.1, 128.3, 129.6, 131.5, 135.4, 145.1, 170.1; IR (KBr) 3234, 2925, 1728, 1596, 1488, 1172, 866 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$ ($\text{M}+\text{Na}$) 378.1134, found 378.1133; Anal. Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$: C, 67.58; H, 5.95; N, 3.94. Found: C, 67.32; H, 5.99; N, 3.99.

(3b). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.57-1.47 (m, 2H), 1.77-1.66 (m, 2H), 2.36-2.28 (m, 7H), 2.40 (s, 3H), 7.06 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.96 (d, $J = 8.0$ Hz, 2H), 9.37 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 18.9, 21.2, 21.5, 23.4, 27.7, 35.5, 80.9, 88.5, 120.6, 128.1, 128.8, 129.5, 131.2, 135.5, 137.4, 144.9, 171.10; IR (KBr) 3236, 2927, 1724, 1596, 1444, 1174, 866, 659 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{S}$ ($\text{M}+\text{Na}$) 392.1291, found 392.1290; Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{S}$: C, 68.27; H, 6.27; N, 3.79. Found: C, 67.97; H, 6.26; N, 3.80.

(3c). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.51-1.43 (m, 2H), 1.71-1.61 (m, 2H), 2.30-2.21 (m, 4H), 2.35 (s, 3H), 3.72 (s, 3H), 6.72 (d, $J = 8.0$ Hz, 2H), 7.22 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.86 (d, $J = 8.0$ Hz, 2H), 8.44 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.0, 21.7, 23.5, 27.8, 35.7, 55.2, 80.9, 87.6, 113.8, 115.9, 128.3, 128.3, 129.6, 132.9, 135.6, 145.2, 159.1; IR (KBr) 3307,

2947, 1724, 1604, 1471, 1174, 846 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$ ($\text{M}+\text{Na}$) 408.1240, found 408.1240; Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$: C, 65.43; H, 6.01; N, 3.63. Found: C, 65.15; H, 6.04; N, 3.65.

(3d). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.53-1.44 (m, 2H), 1.73-1.63 (m, 2H), 2.32-2.26 (m, 4H), 2.39 (s, 3H), 5.90 (s, 2H), 6.67 (d, $J = 8.0$ Hz, 1H), 6.77 (br s, 1H), 6.84 (d, $J = 8.0$ Hz, 1H), 7.29 (d, $J = 8.0$ Hz, 2H), 7.91 (d, $J = 8.0$ Hz, 2H), 9.12 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 18.9, 21.4, 21.6, 23.4, 27.7, 35.6, 80.7, 87.5, 101.1, 108.2, 111.5, 116.9, 125.8, 128.2, 129.6, 135.5, 145.0, 147.2, 170.9; IR (KBr) 3311, 2947, 1720, 1600, 1440, 1172, 860, 663 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{S}$ ($\text{M}+\text{Na}$) 422.1033, found 422.1033; Anal. Calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{S}$: C, 63.14; H, 5.30; N, 3.51. Found: C, 63.04; H, 5.35; N, 3.66.

(3e). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.59-1.50 (m, 2H), 1.77-1.68 (m, 2H), 2.37-2.29 (m, 4H), 2.42 (s, 3H), 7.36-7.30 (m, 4H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.94 (d, $J = 8.0$ Hz, 2H), 8.98 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.0, 21.6, 23.4, 27.6, 35.6, 80.0, 90.4, 122.2, 128.3, 128.4, 129.6, 132.7, 133.5, 135.5, 145.2, 168.2; IR (KBr) 3315, 3147, 2933, 1720, 1596, 1488, 1174, 1089 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_3\text{SCl}$ ($\text{M}+\text{Na}$) 412.0745, found 412.0744; Anal. Calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_3\text{SCl}$: C, 61.61; H, 5.17; N, 3.59. Found: C, 61.42; H, 5.18; N, 3.52.

(3f). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.60-1.51 (m, 2H), 1.77-1.68 (m, 2H), 2.40-2.28 (m, 4H), 2.41 (s, 3H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.0$ Hz, 2H), 7.92 (d, $J = 8.0$ Hz, 2H), 8.50 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.0, 21.6, 23.4, 27.6, 27.6, 35.6, 79.9, 92.2, 122.6, 125.0, 125.1, 128.3, 129.7, 131.8, 135.5, 145.2, 170.9; IR (KBr) 3139, 2956, 1697, 1616, 1471, 1176, 1070 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{20}\text{NF}_3\text{O}_3\text{S}$ ($\text{M}+\text{Na}$) 446.1008, found 446.1006; Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{NF}_3\text{O}_3\text{S}$: C, 59.56; H, 4.76; N, 3.31. Found: C, 59.31; H, 4.82; N, 3.31.

(3g). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.62-1.51 (m, 2H), 1.79-1.69 (m, 2H), 2.32 (t, $J = 7.0$ Hz, 2H), 2.38 (t, $J = 7.0$ Hz, 2H), 2.42 (s, 3H), 3.91 (s, 3H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.40 (d, $J = 8.0$ Hz, 2H), 7.93 (d, $J = 8.0$ Hz, 2H), 7.93 (d, $J = 8.0$ Hz, 2H), 8.80 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.1, 21.6, 23.4, 27.6, 35.6, 52.2, 80.6, 92.8, 128.3, 128.6, 128.9, 129.4, 129.6, 129.7, 131.4, 135.6, 145.2, 166.7; IR (KBr) 3278, 2937, 1716, 1608, 1436, 1174, 856, 663 cm^{-1} ; ESI Calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$ ($\text{M}+\text{Na}$) 436.1189, found 436.1188, Anal. Calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$: C, 63.90; H, 5.61; N, 3.39. Found: C, 63.69; H, 5.56; N, 3.37.

(3h). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.63-1.51 (m, 2H), 1.79-1.70 (m, 2H), 2.30 (t, $J = 7.0$ Hz, 2H), 2.45-2.39 (m, 5H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.48 (d, $J = 8.0$ Hz, 2H), 7.92 (d, $J = 8.0$ Hz, 2H), 8.13 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.2, 21.6, 23.4, 27.4, 35.5, 79.7, 95.5,

123.4, 128.3, 129.6, 130.8, 132.2, 135.5, 145.2, 146.6, 170.6; IR (KBr) 3290, 2947, 1726, 1647, 1436, 1172, 852, 667 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_5\text{S}$ ($\text{M}+\text{Na}$) 423.0985, found 423.0985; Anal. Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_5\text{S}$: C, 59.99; H, 5.03; N, 7.00. Found: C, 59.85; H, 5.14; N, 6.83.

(3i). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.59-1.51 (m, 2H), 1.78-1.68 (m, 2H), 2.08 (t, $J = 8.0$ Hz, 2H), 2.41 (s, 3H), 7.37-7.23 (m, 7H), 7.91 (d, $J = 8.0$ Hz, 2H), 8.24 (bs, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 19.1, 23.6, 27.8, 35.7, 81.7, 89.5, 125.0, 127.6, 128.1, 128.3, 129.6, 131.5, 135.4, 145.1, 170.1; IR (KBr) 3043, 2931, 1760, 1629, 1431, 1147, 848 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$ ($\text{M}+\text{Na}$) 378.1134, found 378.1133; Anal. Calcd for $\text{C}_{19}\text{H}_{19}\text{NO}_3\text{S}$: C, 66.84; H, 5.61; N, 4.10. Found: C, 66.79; H, 5.33; N, 4.35.

General Procedure for Synthesis of Lactams. To a 5 mL screw capped vial equipped with a magnetic stirring bar were added the amido-alkyne **3a** (106 mg, 0.3 mmol), $\text{Pd}(\text{PPh}_3)_4$ (35 mg, 0.03 mmol), benzoic acid (7.5 mg, 0.06 mmol) and 1,4-dioxane (1.5 ml). After the mixture was stirred for 12h at 100 $^\circ\text{C}$, TLC was taken in order to confirm whole disappearance of the starting material. The solvent was removed under reduced pressure, and the residue was purified by short silica gel column with eluent (hexane:ethyl acetate, 9:1) to give **4a** (98.0 mg) in 92% yield.

(2). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 2.33 (t, $J = 7.0$ Hz, 2H), 2.51 (dt, $J = 15.0, 7.0$ Hz, 2H), 4.44 (d, $J = 6.0$ Hz, 2H), 5.70 (br s, 1H), 5.79 (dt, $J = 15.0, 7.0$ Hz, 1H), 6.24 (dd, $J = 15.0, 10.0$ Hz, 1H), 6.43 (d, $J = 15.0$ Hz, 1H), 6.70 (dd, $J = 15.0, 10.0$ Hz, 1H), 7.37-7.18 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 28.7, 35.9, 43.4, 126.1, 127.2, 127.3, 127.6, 128.5, 128.5, 128.8, 130.9, 131.6, 133.1, 137.3, 138.2, 172.1; IR (KBr) 3298, 2916, 1725, 1635, 1448, 993, 752, 694 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}$ ($\text{M}+\text{Na}$) 314.1515, found 314.1515; Anal. Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}$: C, 82.44; H, 7.26; N, 4.81. Found: C, 82.27; H, 7.27; N, 4.78.

(4a). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.96-1.72 (m, 2H), 2.08-2.02 (m, 2H), 2.38 (s, 3H), 2.51-2.43 (m, 2H), 5.40-5.35 (m, 1H), 6.14 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.49 (d, $J = 16.0$ Hz, 1H), 7.20 (d, $J = 8.0$ Hz, 2H), 7.34-7.23 (m, 5H), 7.89 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ

16.5, 21.6, 29.2, 33.4, 58.3, 126.5, 127.8, 128.0, 128.6, 128.9, 129.4, 132.7, 135.8, 136.2, 144.6, 170.4; IR (KBr) 3261, 2949, 1716, 1596, 1450, 1170, 813 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$ (M+Na) 378.1134, found 378.1134; Anal. Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$: C, 67.58; H, 5.95; N, 3.94. Found: C, 67.28; H, 5.81; N, 4.00.

(4b). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.90-1.65 (m, 2H), 2.02-1.94 (m, 2H), 2.28 (s, 3H), 2.32 (s, 3H), 2.44-2.33 (m, 2H), 5.33-5.28 (m, 1H), 5.99 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.39 (d, $J = 16.0$ Hz, 1H), 7.16-7.02 (m, 6H), 7.83 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 16.4, 21.1, 21.6, 27.4, 29.3, 33.4, 126.1, 126.4, 126.7, 128.3, 128.4, 128.9, 129.2, 129.5, 132.6, 137.9, 170.3; IR (KBr) 3261, 2920, 1716, 1596, 1450, 1292, 660 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{S}$ (M+Na) 392.1291, found 392.1290; Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{S}$: C, 68.27; H, 6.27; N, 3.79. Found: C, 67.98; H, 6.28; N, 3.84.

(4c). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 2.70-1.71 (m, 4H), 2.53-2.36 (m, 5H), 3.83 (s, 3H), 3.65 (s, 1H), 5.99 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.45 (d, $J = 16.0$ Hz, 1H), 6.86 (d, $J = 8.0$ Hz, 2H), 7.27-7.18 (m, 4H), 7.90 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 16.4, 21.5, 29.3, 33.4, 33.4, 55.3, 114.0, 125.5, 126.4, 127.8, 128.4, 128.9, 129.5, 129.6, 132.2, 144.5, 170.5; IR (KBr) 3294, 2949, 1720, 1604, 1512, 1251, 1170 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$ (M+Na) 408.1240, found 408.1241; Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$: C, 65.43; H, 6.01; N, 3.63. Found: C, 65.40; H, 6.02; N, 3.61.

(4d). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 2.03-1.65 (m, 4H), 2.33 (s, 3H), 2.44-2.34 (m, 2H), 5.31-5.25 (m, 1H), 5.94-5.85 (m, 3H), 6.34 (d, $J = 16.0$ Hz, 1H), 6.78-6.63 (m, 3H), 7.16 (d, $J = 8.0$ Hz, 2H), 7.83 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.9, 20.9, 21.5, 22.5, 31.5, 60.3, 101.1, 108.2, 121.4, 126.3, 128.2, 128.9, 129.3, 129.5, 129.6, 130.8, 131.8, 144.8, 171.2; IR (KBr) 3257, 2952, 1718, 1697, 1444, 1180, 670 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{S}$ (M+Na) 422.1033, found 422.1034; Anal. Calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_5\text{S}$: C, 63.14; H, 5.30; N, 3.51. Found: C, 63.36; H, 5.55; N, 3.71.

(4e). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.90-1.65 (m, 2H), 2.02-1.95 (m, 2H), 2.33 (s, 3H), 2.45-2.36 (m, 2H), 5.34-5.27 (m, 1H), 6.05 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.45 (d, $J = 16.0$ Hz, 1H), 7.25-7.13 (m, 6H), 7.82 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 16.4, 21.5, 27.3, 29.1, 33.4, 127.3, 127.7, 128.2, 128.6, 129.0, 129.3, 129.5, 131.6, 135.8, 144.8, 170.5; IR (KBr) 3246, 2954, 1716, 1595, 1490, 1332, 1085 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{20}\text{ClNO}_3\text{S}$ (M+Na) 412.0745, found 412.0745; Anal. Calcd for $\text{C}_{20}\text{H}_{20}\text{ClNO}_3\text{S}$: C, 61.61; H, 5.17; N, 3.59. Found: C, 61.30; H, 5.23; N, 3.59.

(4f). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.90-1.66 (m, 2H), 2.04-1.96 (m, 2H), 2.46-2.32 (m, 5H), 5.37-5.30 (m, 1H), 6.19 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.49 (d, $J = 16.0$ Hz, 1H), 7.20-7.14 (m, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.0$ Hz, 2H), 7.82 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 16.5, 21.6, 29.0, 33.4, 33.4, 58.1, 125.5, 125.6, 126.7, 129.0, 129.4, 130.8, 131.4, 136.2, 139.3, 144.8, 170.1; IR (KBr) 3271, 2958, 1720, 1618, 1452, 1348, 810 cm^{-1} ; ESI Calcd for $\text{C}_{21}\text{H}_{20}\text{F}_3\text{NO}_3\text{S}$ (M+Na) 446.1008, found 446.1007; Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{F}_3\text{NO}_3\text{S}$: C, 59.56; H, 4.76; N, 3.31. Found: C, 59.43; H, 4.85; N, 3.37.

(4g). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.95-1.73 (m, 4H), 2.39 (s, 3H), 2.52-2.39 (m, 2H), 3.92 (s, 3H), 5.42-5.37 (m, 1H), 6.26 (dd, $J = 16.0, 10.0$ Hz, 1H), 6.54 (d, $J = 16.0$ Hz, 1H), 7.21 (d, $J = 8.0$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 7.89 (d, $J = 8.0$ Hz, 2H), 7.98 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.6, 26.1, 30.6, 33.5, 38.3, 61.3, 126.5, 128.5, 128.6, 129.0, 129.3, 129.5, 129.9, 130.7, 131.9, 144.9, 166.7, 172.9; IR (KBr) 3247, 2952, 1720, 1641, 1452, 1311, 660 cm^{-1} ; ESI Calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$ (M+Na) 436.1189, found 436.1188; Anal. Calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$: C, 63.90; H, 5.61; N, 3.39. Found: C, 63.97; H, 5.67; N, 3.42.

(4h). Thick oil; ^1H NMR (400 MHz, CDCl_3) δ 1.90-1.80 (m, 1H), 2.55-2.30 (m, 6H), 3.50 (d, $J = 7.0$ Hz, 2H), 4.89 (t, $J = 7.0$ Hz, 1H), 5.30 (dd, $J = 15.0, 7.0$ Hz, 1H), 6.49 (dt, $J = 15.0, 7.0$ Hz, 1H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.85 (d, $J = 8.0$ Hz, 2H), 8.16 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.5, 21.5, 25.9, 30.5, 37.9, 123.7, 128.2, 129.3, 129.3, 130.6, 130.8, 135.8, 145.0, 146.6, 146.9, 173.1; IR (KBr) 3250, 2925, 1732, 1596, 1519, 1170, 680 cm^{-1} ; ESI Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_5\text{S}$ (M+Na) 423.0985, found 423.0986; Anal. Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_5\text{S}$: C, 59.99; H, 5.03; N, 7.00. Found: C, 59.93; H, 5.20; N, 6.84.

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 DATE Fri Apr 22 12:16:03 2005
 DATIM
 OBNUC ¹H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBF IN 10500.00 Hz
 POINT 32768
 FREQU 7993.60 Hz
 SCANS 8
 ACQTM 4.0983 sec
 PD 2.9010 sec
 PUL 5.50 usec
 IRNUC ¹H
 CTEMP 19.0 c
 SLVNT CDCL₃
 EXREF 5.30 ppm
 BF 0.10 Hz
 RGAIN 20

C:\Documents and Settings\All Users\Documents\DATA\MS21\huo\Gan\GCS16cc-H.als
huo573

8-5

DFILE C:\Documents and Settings\All Users\Documents
CONNT huos73
DATIM Wed Dec 15 15:04:14 2004
OBNUC 1H
EXAND NON
OBFREQ 399.65 MHz
OBFSET 124.00 KHz
OBF IN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 22.3 C
SLVNT CDCl3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 18

C:\Documents and Settings\All User\My Documents\DATA\A521\huo\huo801.a1s
huo801

6-5

DF:FILE C:\Documents and Settings\All User\My Documents\DATA\A521\huo\huo801.a1s
 COUNT hu0801
 DATE: Mon Dec 12 16:01:04 2005
 ORNUC 1H
 EXMOD NON
 OFRQ 399.65 MHz
 OBSET 124.00 KHz
 OFIN 10500.00 Hz
 POINT 32768
 FRECU 7993.60 Hz
 SCANS 8
 ACQTM 4.0993 sec
 PD 2.9010 sec
 IRRUC 5.50 usec
 PW1 24.9 c
 CTEP CDCL3
 SLWT 7.26 ppm
 EXREF 0.12 Hz
 RF 8
 RGAIN

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo622-3.als
huo622-3

DF:FILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo622-3
 COUNT huo622-3
 DATE: Mon Dec 12 10:42:08 2005
 DE:UC 1H
 EX:MOD NON
 OF:FRQ 399.65 MHz
 OF:SET 124.00 KHz
 OF:IN 10500.00 Hz
 PO:INT 32768
 FRE:QU 7993.60 Hz
 SC:ANS 8
 ACQ:TM 4.0993 sec
 PD 2.9010 sec
 PWT 5.50 usec
 IRR:UG 1H
 CT:MP 24.1 °C
 SL:VNT CDCL3
 EX:REF 0.00 ppm
 BF 0.12 Hz
 RG:AIN 16

C:\Documents and Settings\All Users\Documents\XDATA\621\huo\huo730.als
huo730

11-5

DFILE C:\Documents and Settings\All Users\XO
CONNT hu0730
DATIM Mon Dec 12 13:17:05 2005
OBNUG 1H
EXMOD NMR
OBFREQ 399.65 MHz
OBSSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 7
ACQTM
PD 4.0993 sec
PUL 2.9010 sec
PRG 5.50 usec
IRNUG 1H
CTEMP 24.9 °C
SLVNT CDCl3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 10

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DE FILE      C:\Documents and Settings\All Users\HD
COMMIT       hu0806
DATIM        Mon Dec 12 17:48:31 2005
OBINIC       1H
OBXEND       NON
OBFREQ       399.65 MHz
OBSET        124.00 KHz
OBFIN        10500.00 Hz
POINT        32768
FREQU        7993.60 Hz
SCANS        8
SCANS        4.0893 sec
ACQIM        2.9010 sec
PD           5.50 usec
PWT1
IRNOC        1H
CTEMP        25.0 c
SLVMT
EXREF
BF           7.26 ppm
RGAIN        0.12 Hz
           12

```

C:\Documents and Settings\All User\Documents\DATA\521\huo\huo617-ok.als
huo617

5-13

DFILE C:\Documents and Settings\All User\Documents\DATA\521\huo\huo617
CONTC hu0617
DATE Wed Mar 09 17:11:42 2005
GENUG 1H
EXMUG 1H
EXMUG NON
ORFREQ 399.65 MHz
ORFREQ 124.00 KHz
ORFREQ 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTIME 4.0993 sec
PD 2.9010 sec
PMT 5.50 usec
IRNUC 1H
CTEMP 21.3 C
SLVNT CDCl3
EXREF 7.24 ppm
BF 2.00 Hz
RGAIN 14

C:\Documents and Settings\All Users\Documents\MDATA\521\huo\huo807.als
huo807

71-5

D:\FILE C:\Documents and Settings\All Users\MD
 COMIT hu0807
 DATIM Mon Dec 12 18:49:49 2005
 DBNUC 1H
 EXMOD NCU
 DBFRQ 399.65 MHz
 DBSET 124.00 KHz
 DBFIN 10500.00 Hz
 POINT 32768
 FREQU 7993.60 Hz
 SCANS 8
 ACQTH
 PD 4.0993 sec
 PWT 2.9010 sec
 1RMUC 5.50 usec
 CTMP 1H
 SLWT 25.2 °
 EXREF CDCL3
 BF 7.26 ppm
 RGAIN 0.12 Hz
 15

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo712.als
huo712

SI-5

DFILE C:\Documents and Settings\All Users\Documents\huo712
CONHT hu0712
DATIM Wed Jun 29 20:14:16 2005
DENUM 1H
EXMOD NON
OFFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 29.5 C
SLVMT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 24

C:\Documents and Settings\K11\Users\Documents\DATA\K521\Whio\Kant\GS316cc-H.als
GS316cc

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DFILE C:\Documents and Settings\K11\Users\Documents\DATA\K521\Whio\Kant\GS316cc
CONHT 31
DATUM Wed Dec 15 15:04:14 2004
GENUG 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREOU 7993.80 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWT 5.50 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCl₃
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\NOA1A\5214\huc\huc0647.a1s
huc0647

DFILE C:\Documents and Settings\All Users\Documents\huc0647
 COUNT huc0647
 DATIM Thu Dec 15 20:39:27 2005
 ORNUC 1H
 EXAMOD NON
 ORFREQ 399.65 MHz
 ORSET 124.00 KHz
 ORFIN 10500.00 Hz
 POINT 32768
 FREQ 7993.60 Hz
 SCANS 8
 ACQTIM 4.0993 sec
 PD 2.9010 sec
 PWT 5.50 usec
 IRNUC 1H
 CTMP 26.1 °C
 SLVT CDCL3
 EXREF 7.24 ppm
 BF 0.12 Hz
 RGAIN 21

L1-5

C:\Documents and Settings\All Users\Documents\DATA621\huo\huo612-gpc-ok.ais
huo612-gpc

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DFILE C:\Documents and Settings\All Users\Documents\DATA621\huo\huo612-gpc
COMNT hu0612-gpc
DAT11 Thu Mar 10 21:12:58 2005
OBNUC 1H
EXMOD NON
OBR0 399.65 MHz
OBS0 124.00 KHz
OBS1 10500.00 Hz
POINT 32768
FREQ0 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PR1 5.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCl3
EXREF 7.24 ppm
BF 0.30 Hz
RGAIN 16

C:\Documents and Settings\All Users\Documents\DATA\K521\huo\huo808-ok.als
huo808-ok

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DFILE C:\Documents and Settings\All Users\Documents\huo808-ok
COMNT huo808-ok
DATIM Fri Dec 30 12:35:43 2005
ORNUC 1H
EXMOD NON
ORFREQ 399.65 MHz
ORFSET 124.00 KHz
ORFIN 10500.00 Hz
POINT 32788
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PPI 5.50 usec
IRNUC 1H
CTEMP 20.8 C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

C:\Documents and Settings\Xili User\Documents\DATA521\huo626-gpc3. a1s
huo626-gpc3

5-20

DF-FILE C:\Documents and Settings\Xili User\Documents\DATA521\huo626-gpc3
COMMIT hu0626-gpc3
DATE Tue Apr 05 11:30:30 2005
ORIGIN 1H
EXAMID MON
EXPTNO 399.65 MHz
ORFNO 124.00 KHz
ORF1N 10500.00 Hz
POINT 32768
FREQ0 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWT 5.50 usec
IRNUC 1H
CTEMP 21.5 C
SLVNT CDCL3
EXREF 7.26 ppm
RGAIN 2.00 Hz

C:\Documents and Settings\All Users\Documents\DATA\MS21\huo\huo731b-ok.a1s
huo731-ok

DFILE C:\Documents and Settings\All Users\Documents\huo731-ok
COMET hu731-ok
DATE Fri Dec 30 12:10:07 2005
NAME hu731
EXPNO 1
PROCNO 1
PROCNAME hu731
F2 - F1 399.65 MHz
F2 - F1 124.00 KHz
F2 - F1 10500.00 Hz
POINT 32/68
FREQ 7993.60 Hz
SCANS 8
ACQTIME 4.0993 sec
PD 2.9010 sec
PMT 5.50 usec
IRNUC 1H
CTEMP 20.5 c
SOLVENT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

C:\Documents and Settings\Kali\My Documents\DATA521\huo\huo816-2-ok.als
huo816-2-ok

DF:FILE C:\Documents and Settings\Kali\My Documents\DATA521\huo\huo816-2-ok.als
COMMIT huo816-2-ok
DATE Fri Dec 30 13:40:29 2005
NAME
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 kHz
OBSF IN 10500.00 Hz
POINT 32768
FREQD 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PMT 5.50 usec
IRNUC 1H
CTEMP 21.4 C
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

5-2.2

C:\Documents and Settings\All Users\Documents\XDA\AN521\huo\huo619-gpc.als
huo619-gpc

S-23

DFILE C:\Documents and Settings\All Users\Documents\XDA\AN521\huo\huo619-gpc.als
COMPT huo619-gpc
DATIM Sat Dec 24 14:44:49 2005
ORNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PMT 5.50 usec
IRNUC 1H
CTEMP 24.3 C
SLVNT CDCl3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 23

C:\Documents and Settings\All Users\Documents\DATA\621\huo\huo817-7ok.als
huo817-ok

DF:FILE C:\Documents and Settings\All Users\Documents\DATA\621\huo\huo817-7ok.als
 COMNT huo817-ok
 DATIM Thu Jan 05 21:48:30 2006
 ORNUC 1H
 EXMOD NON
 ORF-RQ 399.65 MHz
 ORSET 124.00 KHz
 ORF-IN 10500.00 Hz
 POINT 32768
 FREOU 7993.60 Hz
 SCANS 8
 ACQTM 4.0993 sec
 PD 2.9010 sec
 PNT 5.50 usec
 IRRNUC 1H
 CTENP 23.3 g
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 23

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C:\Documents and Settings\All User\Documents\DATA\K521\huo\huo715-ok. a1s
huo715

DE FILE C:\Documents and Settings\All User\sho
COMINT hu0715
DATE Thu Jan 05 21:33:19 2006
ORNUC 1H
EXMOD NON
ORFRO 399.65 MHz
ORSET 124.00 kHz
ORFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PMT 5.50 usec
IRNUC 1H
GTEMP 23.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

5-25