Supplementary Information

Synthesis and Bacterial Biofilm Inhibition Studies of Ethyl *N*-(2-phenethyl) Carbamate Derivatives

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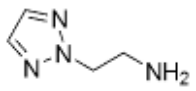
General Experimental

All reagents used for chemical synthesis were purchased from commercially available sources and used without further purification. Chromatography was performed using 60 Å mesh standard grade silica gel from Sorbtech. NMR solvents were obtained from Cambridge Isotope Labs and used as is. ^{1}H NMR (300 MHz or 400 MHz) and ^{13}C NMR (75 MHz or 100 MHz) spectra were recorded at 25ºC on Varian Mercury spectrometers. Chemical shifts (δ) are given in ppm relative to tetramethylsilane or the respective NMR solvent; coupling constants (*J*) are in hertz (Hz). Abbreviations used are s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublets, t = triplet, dt = doublet of triplets, bt = broad triplet, qt = quartet, m = multiplet, bm = broad multiplet and br = broad. Mass spectra were obtained at the NCSU Department of

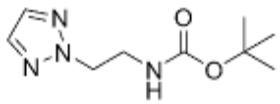
Chemistry Mass Spectrometry Facility. Funding was obtained from the North Carolina Biotechnology Center and the NCSU Department of Chemistry.

General Procedure for Compounds 2a-2h, 3a-3h, 4a-4h, 5a-5h, 6a-6h, 7a-7h, 11a-h and 12a-12h. Ten mL of dichloromethane and a stir bar was added to 100-200 mg of the amine. Two equivalents of triethylamine was then added and the reaction mixture was cooled to 0 ºC while stirring. Then, 0.9 equivalents of the chloroformate, isocyanate or thioisocyanate was added dropwise to the reaction mixture and was allowed to slowly warm to room temperature and continuted stirring overnight. The reaction mixture was then diluted with more dichloromethane, washed twice with 1N HCl, washed twice with brine, dried with sodium sulfate and then concentrated in vacuo.

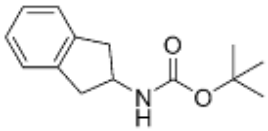
General Procedure for Compounds 8a-8h, 9a-9l and 10a-10l. Ten mL of dichloromethane and a stir bar was added to 100-200 mg of the amine. Two equivalents of triethylamine was then added and the reaction mixture was cooled to 0 ºC while stirring. Then, 0.9 equivalents of the chloroformate, isocyanate or thioisocyanate was added dropwise to the reaction mixture and was allowed to slowly warm to room temperature and continuted stirring overnight. The reaction mixture was then diluted with more dichloromethane, washed twice with brine, dried with sodium sulfate and then concentrated in vacuo. The crude mixture was then purified via flash chromatography on silica gel using a 2.5% - 10% methanol/dichlormethane eluent.



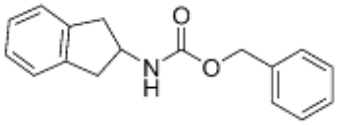
2-(2H-1,2,3-triazol-2-yl)ethanamine (**5**): White solid. mp=129-131 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (s, 2H), δ 4.55 (t, J = 1.2 Hz, 2H), δ 3.33 (t, J = 5.7 Hz, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.1, 51.3, 38.8 ppm; IR ν_{max} (cm^{-1}) 3054, 2987, 2306, 1421, 1258; HRMS (ESI) calcd for $C_4H_8N_4$ (M+) 113.0822, found 113.0819.



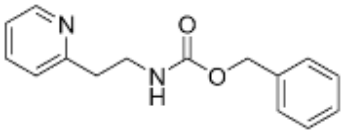
tert-butyl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamate (5e): White solid. mp=64-66 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55 (s, 2H), δ 5.15 (s, 1H), δ 4.49 (t, J = 5.7Hz, 2H), δ 3.63 (q, J = 5.7, 5.1 Hz, 2H), δ 1.36 (s, 9H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.9, 134.5, 79.8, 54.8, 40.2, 28.5, 27.8 ppm; IR ν_{max} (cm^{-1}) 3054, 2986, 1713, 1506; HRMS (ESI) calcd for $C_9H_{16}N_4O_2$ (M+) 235.1165, found 235.1169.



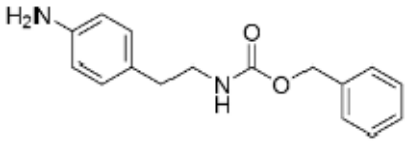
tert-butyl 2,3-dihydro-1H-inden-2-ylcarbamate (**3e**): White solid. mp=51-53 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 (m, 4H), δ 4.84 (s, 1H), δ 4.45 (s, 1H), δ 3.29 (d, J = 7.2 Hz, 1H), δ 3.24 (d, J = 6.9 Hz, 1H), δ 2.80 (d, J = 4.8 Hz, 1H), δ 2.75 (d, J = 4.8 Hz, 1H), δ 1.43 (s, 9H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ153.5, 140.9, 126.5, 124.7, 79.3, 51.9, 40.3, 28.4 ppm; IR ν_{max} (cm^{-1}) 3419, 2321, 1641; HRMS (ESI) calcd for $C_{17}H_{17}NO_2$ (M+) 256.1308, found 256.1308.



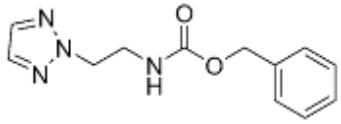
benzyl 2,3-dihydro-1H-inden-2-ylcarbamate (**3c**): Light yellow solid. mp=159-161 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (s, 4H), δ 7.13 (t, J = 1.2 Hz, 5H), δ 5.09 (s, 2H), δ 4.56 (s, 2H), δ 3.28 (m, 2H), δ 2.77 (m, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.3, 136.7, 128.8, 126.9, 125.0, 66.9, 51.8, 40.8 ppm; IR ν_{max} (cm^{-1}) 3419, 2977, 1691, 1643, 1265; HRMS (ESI) calcd for $C_{17}H_{17}NO_2$ (M+) 268.1332, found 268.1337.



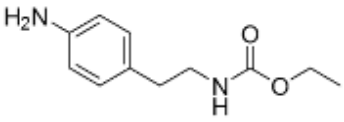
benzyl 2-(pyridin-2-yl)ethylcarbamate (**9c**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.43 (d, J = 4.8 Hz, 1H), δ 7.52 (t, J = 1.8 Hz, 1H), δ 7.23 (s, 5H), δ 7.06 (m, 2H), δ 5.91 (s, 1H), δ 5.04 (s, 2H), δ 3.57 (q, J = 6.3 Hz, 6.3 Hz, 2H), δ 2.93 (t, J = 6.6 Hz, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.7, 149.4, 137.0, 128.6, 127.1, 123.7, 121.7, 66.7, 40.6, 37.8 ppm; 3440, 2092, 1644, 1261; HRMS (ESI) calcd for $C_{15}H_{16}N_2O_2$ (M+) 257.1285, found 257.1288.



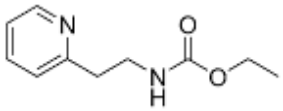
benzyl 4-aminophenethylcarbamate (**8c**): Light yellow solid. mp=70-73 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (s, 5H), δ 6.93 (d, J = 8.1 Hz, 2H), δ 6.58 (d, J = 8.4 Hz, 2H), δ 5.06 (s, 2H), δ 4.91 (s, 1H), δ 3.57 (s, 2H), δ 3.36 (q, J = 6.6, 6.6 Hz, 2H), δ 2.65 (t, J = 6.9 Hz, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.6, 145.2, 136.9, 129.8, 128.7, 115.6, 66.8, 58.8, 42.7, 35.4 ppm; IR ν_{max} (cm^{-1}) 3389, 1682, 1543; HRMS (ESI) calcd for $C_{16}H_{18}N_2O_2$ (M+) 271.1441, found 271.1446 .



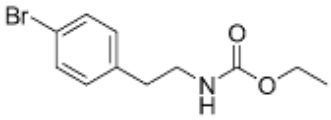
benzyl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamate (**5c**): Light yellow residue. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (s, 2H), δ 7.28 (m, 5H), δ 5.38 (s, 1H), δ 5.15 (s, 2H), δ 4.51 (t, J = 5.4 Hz, 2H), δ 3.75 (t, 6.3 Hz, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.5, 136.5, 134.5, 128.4, 67.1, 54.6, 40.6 ppm; IR ν_{max} (cm^{-1}) 3440, 3054, 2986, 2305, 1719; HRMS (ESI) calcd for $C_{12}H_{14}N_4O_2$ (M+) 269.1009, found 269.1011.



ethyl 4-aminophenethylcarbamate (**8d**): Light yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.95 (d, J = 8.1 Hz, 2H), δ 6.61 (d, J = 6.3 Hz, 2H), δ 4.89 (s, 1H), δ 4.08 (q, J = 6.9, 7.2 Hz, 2H), δ 3.74 (s, 2H), δ 3.33 (t, J = 6.3 Hz, 2H), δ 2.65 (t, J = 7.2 Hz, 2H), δ 1.19 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9, 145.0, 129.8, 115.7, 60.9, 42.6, 35.4, 14.9 ppm; IR ν_{max} (cm^{-1}) 3346, 2932, 1698, 1627; HRMS (ESI) calcd for $C_{11}H_{16}N_2O_2$ (M+) 209.1285, found 209.1278.

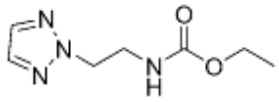


ethyl 2-(pyridin-2-yl)ethylcarbamate (**9d**): Light yellow solid. mp=56-58 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.41 (d, J = 4.8 Hz, 1H), δ 7.49 (t, J = 5.7 Hz, 1H), δ 7.02 (m, 2H), δ 5.77 (s, 1H), δ 4.00 (q, J = 6.9, 7.2 Hz, 2H), δ 3.50 (q, J = 6.3, 6.6 Hz, 2H), δ 2.89 (t, J = 6.6 Hz, 2H), δ 1.11 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5, 156.9, 149.3, 136.6, 123.5, 121.6, 60.6, 40.4, 37.9, 14.8 ppm; IR ν_{max} (cm^{-1}) 3435, 2091, 1641, 1259; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .

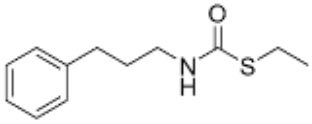


ethyl 4-bromophenethylcarbamate (**11d**): White solid. mp=63-65 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (d, J = 2.7 Hz, 2H), δ 7.07 (d, J = 8.7 Hz, 2H), δ 4.94 (s, 1H), δ 4.09 (q, J = 6.9, 7.2 Hz, 2H), δ 3.39 (q, J = 6.6, 6.9 Hz, 2H), δ 2.75 (t, 6.9 Hz, 2H), δ 1.21 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.8, 138.1, 131.7, 120.5, 61.0, 42.1, 35.8, 14.9 ppm; IR

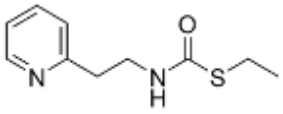
ν_{max} (cm^{-1}) 3348, 2975, 1691, 1537, 1260; HRMS (ESI) calcd for $C_{11}H_{14}BrNO_2$ (M+) 227.0281, found 227.0279.



ethyl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamate (**5d**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (s, 2H), δ 6.31 (s, 1H), δ 4.671 (t, J = 4.2 Hz, 2H), δ 4.17 (q, J = 4.5, 4.5 Hz, 2H), δ 3.31 (s, 2H), δ 1.21 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.1, 134.7, 54.2, 44.3, 41.8, 14.1 ppm; IR ν_{max} (cm^{-1}) 3434, 1642; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .

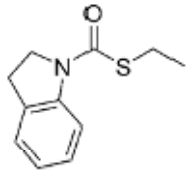


S-ethyl 3-phenylpropylcarbamothioate (**4b**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 – 7.10 (m, 5H), δ 6.24 (s, 1H), δ 3.26 (t, J = 5.7 Hz, 2H), δ 2.82 (q, J = 4.8, 6.9 Hz, 2H), δ 2.58 (t, J = 7.8 Hz, 2H), δ 1.76 (m, 2H), δ 1.26 (t, J = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.8, 141.7, 128.7, 126.5, 41.3, 33.4, 32.0, 24.5, 16.1 ppm; IR ν_{max} (cm^{-1}) 3318, 3027, 2929, 2867, 1650, 1624, 1215, 700; HRMS (ESI) calcd for $C_{12}H_{17}NOS$ (M+) 224.1104, found 224.1099.

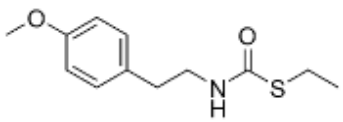


S-ethyl 2-(pyridin-2-yl)ethylcarbamothioate (**9b**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 (d, J = 3 Hz, 1H), δ 7.47 (t, J = 6.0 Hz, 1H), δ 7.01 (m, 2H), δ 6.88 (s, 1H), δ 3.58 (q, J = 4.8, 4.5, 2H), δ 2.89 (t, J = 5.1 Hz, 2H), δ 2.78 (q, J = 5.4, 5.4 Hz, 2H), δ 1.15 (t, J = 5.1 Hz, 3H)

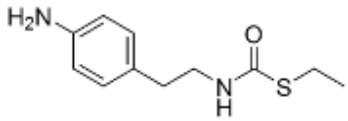
ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.3, 149.3, 136.8, 123.6, 121.8, 40.7, 37.4, 24.6, 15.9 ppm; IR ν_{max} (cm^{-1}) 3432, 2089, 1645, 1213; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .



S-ethyl indoline-1-carbothioate (**6b**): Light yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 (d, J = 5.7 Hz, 1H), δ 7.08 – 6.88 (m, 4H), δ 3.80 (t, J = 7.8 Hz, 2H), δ 2.94 (q, J = 6.0, 5.4 Hz, 2H), δ 2.89 (t, J = 3.3 Hz, 2H), δ 1.31 (t, J = 5.7 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.6, 143.0, 131.3, 127.6, 124.9, 123.6, 116.0, 47.3, 28.0, 24.7, 15.7 ppm; IR ν_{max} (cm^{-1}) 3053, 2932, 2253, 1712, 1598, 1650; HRMS (ESI) calcd for $C_{11}H_{13}NOS$ (M+) 208.0791, found 208.0788.

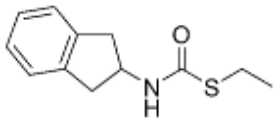


S-ethyl 4-methoxyphenethylcarbamothioate (**12b**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.09 (d, J = 8.4 Hz, 2H), δ 6.83 (d, J = 8.7, 2H), δ 5.96 (s, 1H), δ 3.50 (s, 3H), δ 3.45 (q, J = 6.3, 6.6 Hz, 2H), δ 2.91 (q, J = 3.3, 3.3 Hz, 2H), δ 2.75 (t, J = 7.5 Hz, 2H), δ 1.28 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 158.5, 130.9, 129.9, 114.2, 55.4, 43.0, 35.2, 24.4, 16.0 ppm; IR ν_{max} (cm^{-1}) 3323, 3034, 2962, 1640, 1514; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .

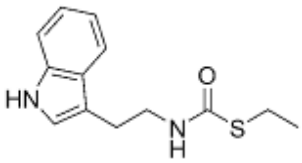


S-ethyl 4-aminophenethylcarbamothioate (**8b**): Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92 (d, J = 6.8, 2H), δ 6.59 (t, J = 6.4 Hz, 2H), δ 5.87 (s, 1H), δ 3.77 (s, 1H), δ 3.41 (t, J = 5.6 Hz, 2H), δ 2.86 (q, J = 7.2, 7.2 Hz, 2H), δ 2.67 (t, J = 6.4, 2H), δ 1.25 (t, J = 5.2 Hz, 3H) ppm;

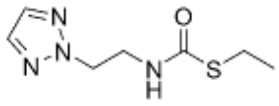
^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 145.1, 129.8, 115.7, 53.8, 43.0, 35.2, 24.5, 16.0 ppm; IR ν_{max} (cm^{-1}) 3434, 2929, 2086, 1647, 1517, 1263, 1219, 970; HRMS (ESI) calcd for $C_{11}H_{16}N_2OS$ (M+) 225.1056, found 225.1060.



S-ethyl 2,3-dihydro-1H-inden-2-ylcarbamothioate (**3b**): White solid. mp=107-110 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.15 (m, 4H), δ 6.08 (d, J = 5.4 Hz, 1H), δ 4.70 (s, 1H), δ 3.26 (dd, J = 7.2, 7.2 Hz, 2H), δ 2.86 (q, J = 5.4, 6.6 Hz, 2H), δ 1.29 (t, J = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 141.0, 127.1, 125.0, 52.7, 40.2, 24.5, 16.0 ppm; IR ν_{max} (cm^{-1}) 3258, 3019, 2945, 1628; HRMS (ESI) calcd for $C_{12}H_{15}NOS$ (M+) 222.0947, found 222.0944.

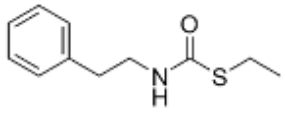


S-ethyl 2-(1H-indol-3-yl)ethylcarbamothioate (**10b**): Light yellow solid. mp=68-70 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.52 (s, 1H), δ 7.54 (d, J = 7.8, 1H), δ 7.24 (d, J = 8.1, 1H), δ 7.17 – 7.04 (m, 2H), δ 6.82 (d, J = 2.1 Hz, 1H), δ 5.74 (s, 1H), δ 3.51 (t, J = 6.3 Hz, 2H), δ 2.91 – 2.83 (m, 4H), δ 1.26 (t, J = 4.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 136.8, 127.6, 122.8, 119.7, 112.6, 42.1, 25.8, 24.7, 16.2 ppm; IR ν_{max} (cm^{-1}) 3418, 3055, 2932, 1657, 1496; HRMS (ESI) calcd for $C_{13}H_{16}N_2OS$ (M+) 249.1056, found 249.1052.

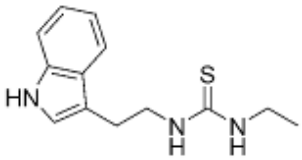


S-ethyl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamothioate (**5b**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (s, 1H), δ 6.14 (s, 1H), δ 4.56 (t, J = 5.1 Hz, 2H), δ 3.83 (q, J = 5.7, 5.7 Hz, 2H), δ 2.88 (q, J = 7.5, 7.5 Hz, 2H), δ 1.25 (t, J = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$)

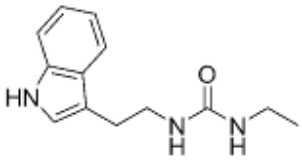
δ 202.1, 134.7, 54.3, 40.5, 24.6, 15.8 ppm; IR ν_{max} (cm^{-1}) 3434, 1647, 671; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .



S-ethyl phenethylcarbamothioate (**2b**): White solid. mp=95-98 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 (m, 5H), δ 6.02 (s, 1H), δ 3.53 (q, J = 6.6, 6.6 Hz, 2H), δ 2.89 (m, 4H), δ 1.30 (t, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 139.0, 129.1, 126.8, 42.9, 36.2, 24.5, 16.1 ppm; IR ν_{max} (cm^{-1}) 3399, 2968, 2929, 2870, 2088, 1650, 1498; HRMS (ESI) calcd for $C_{11}H_{15}NOS$ (M+) 210.0947, found 210.0944.

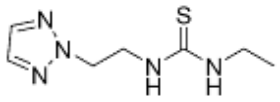


1-(2-(1H-indol-3-yl)ethyl)-3-ethylthiourea (**10h**): Light yellow residue. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67 (s, 1H), δ 7.58 (d, J = 7.2 Hz, 1H), δ 7.33 (d, J = 8.1 Hz, 1H), δ 7.18 (t, J = 6.9 Hz, 1H), δ 7.08 (t, J = 7.2 Hz, 1H), δ 6.86 (s, 1H), δ 6.24 (s, 2H), δ 3.69 – 2.94 (m, 6H), δ 1.00 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 180.9, 136.7, 127.4, 122.4, 118.9, 112.3, 45.0, 39.4, 25.3, 14.4 ppm; IR ν_{max} (cm^{-1}) 3418, 2975, 1634, 1569, 1265, 738, 702; HRMS (ESI) calcd for $C_{13}H_{17}N_3S$ (M+) 248.1216, found 248.1215.

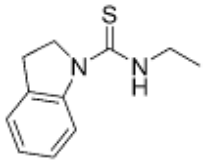


1-(2-(1H-indol-3-yl)ethyl)-3-ethylurea (**10g**): White solid. mp=109-112 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.55 (s, 1H), δ 7.57 (d, J = 7.8 Hz, 1H), δ 7.32 – 7.05 (m, 3H), δ 6.86 (s, 1H), δ 6.13 (s, 2H), δ 3.69 (m, 2H), δ 3.20 (m, 2H), δ 2.94 (t, J = 6.3 Hz, 2H), δ 0.97 (t, J = 7.2 Hz, 3H)

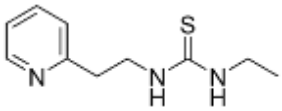
181.0, 136.6, 127.4, 122.9, 119.7, 112.3, 44.6, 39.0, 25.2, 14.3 ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 180.9, 136.6, 127.4, 122.9, 122.4, 119.7, 112.3, 111.9, 44.6, 38.9, 25.2, 14.4 ppm; IR ν_{max} (cm^{-1}) 3944, 3467, 3054, 2986, 2306, 1536; HRMS (ESI) calcd for $C_{13}H_{17}N_3O$ (M+) 232.1444, found 232.1445 .



1-(2-(2H-1,2,3-triazol-2-yl)ethyl)-3-ethylthiourea (**5h**): White solid. mp=91-93 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (d, J = 9.6 Hz 2H), δ 6.64 (s, 2H), δ 4.62 (q, J = 5.1, 3.9 Hz, 2H), δ 4.10 (q, J = 5.7, 5.4 Hz), δ 3.28 (t, J = 5.7 Hz, 2H), δ 1.15 (t, J = 9.3 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.1, 134.7, 100.4, 54.1, 44.2, 14.1 ppm; IR ν_{max} (cm^{-1}) 3419, 1640, 1551; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .

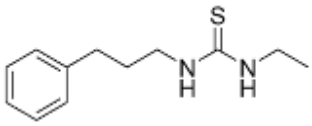


N-ethylindoline-1-carbothioamide (**6h**): Yellow solid. mp=88-91 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, J = 7.8 Hz, 2H), δ 7.08 – 7.01 (m, 2H), δ 6.85 (t, J = 7.2 Hz, 1H), δ 6.21 (t, J = 4.5 Hz, 1H), δ 4.09 (t, J = 8.4 Hz, 2H), δ 3.63 (m, 2H), δ 2.87 (t, J = 8.4 Hz, 2H), δ 1.18 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.9, 142.5, 134.0, 127.1, 125.9, 123.4, 114.9, 53.4, 40.3, 27.3, 14.6 ppm; IR ν_{max} (cm^{-1}) 3390, 3279, 3030, 2972, 1638, 1522; HRMS (ESI) calcd for $C_{11}H_{14}N_2S$ (M+) 207.0950, found 207.0946.

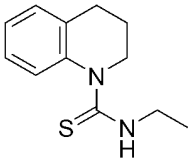


1-ethyl-3-(2-(pyridin-2-yl)ethyl)thiourea (**9h**): Light yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, J = 4.2 Hz, 1H), δ 7.43 (t, J = 7.5 Hz, 1H), δ 7.33 (s, 1H), δ 7.01 – 6.92 (m, 3H), δ 3.70

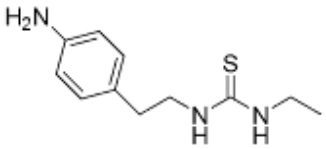
(t, J = 3.9 Hz, 2H), δ 3.21 (m, 2H), δ 2.85 (t, J = 6.3 Hz, 2H), δ 0.98 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.3, 181.0, 159.4, 150.0, 148.9, 137.1, 123.8, 121.9, 43.8, 39.0, 36.8, 24.7, 14.3 ppm; IR ν_{max} (cm^{-1}) 3396, 2974, 2100, 1641, 1556; HRMS (ESI) calcd for $C_{14}H_{17}N_5O_2S$ (M+) , found .



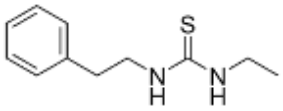
1-ethyl-3-(3-phenylpropyl)thiourea (**4h**): White solid. mp=51-53 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 – 7.11 (m, 5H), δ 6.42 (s, 2H), δ 3.42 (m, 2H), δ 3.34 (m, 2H), δ 2.61 (t, J = 7.5 Hz, 2H), δ 1.86 (m, 2H), 1.10 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.3, 141.4, 128.7, 126.3, 39.3, 33.9, 30.8, 14.6 ppm; IR ν_{max} (cm^{-1}) 3421, 3276, 3053, 2939, 2865, 1547, 1495; HRMS (ESI) calcd for $C_{12}H_{18}N_2S$ (M+) 223.1263, found 223.1261.



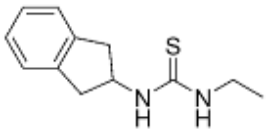
N-ethyl-3,4-dihydroquinoline-1(2H)-carbothioamide (7**h**): Light yellow solid. mp=55-57 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.09 – 6.97 (m, 4H), δ 6.22 (s, 1H), δ 4.09 (t, J = 6.3 Hz, 2H), δ 3.50 (m, 2H), δ 2.59 (t, J = 6.9 Hz, 2H), δ 1.84 (m, 2H), δ 1.02 (t, J = 3.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.9, 138.8, 134.0, 130.3, 126.8, 123.7, 49.1, 40.9, 26.7, 24.0, 14.3 ppm; IR ν_{max} (cm^{-1}) 3396, 3034, 2934, 2875, 2211, 1640, 1516; HRMS (ESI) calcd for $C_{12}H_{16}N_2S$ (M+) 221.1107, found 221.1105.



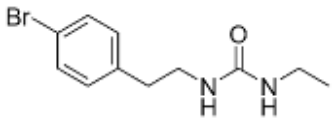
1-(4-aminophenethyl)-3-ethylthiourea (**8h**): Light yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.94 (d, J = 8.1, 2H), δ 6.57 (d, J = 4.5 Hz, 2H), δ 6.06 (d, J = 10.2 Hz, 2H), δ 3.60 (m, 4H), δ 3.26 (s, 2H), δ 2.72 (t, J = 6.9 Hz, 2H), δ 1.08 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.3, 145.3, 129.8, 128.3, 115.7, 46.1, 39.1, 34.6, 14.4 ppm; IR ν_{max} (cm^{-1}) 3408, 1626, 1553; HRMS (ESI) calcd for $C_{11}H_{17}N_3S$ (M+) 224.1216, found 224.1212.



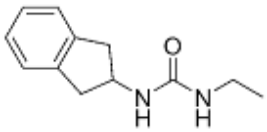
1-ethyl-3-phenethylthiourea (**2h**): White solid. mp=56-58 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 – 7.14 (m, 5H), δ 6.40 (d, J = 10.2 Hz, 2H), δ 3.68 (t, J = 5.1 Hz, 2H), δ 3.30 (m, 2H), δ 2.84 (t, J = 7.2 Hz, 2H), δ 1.08 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.4, 138.7, 129.0, 126.9, 45.9, 39.1, 35.6, 14.4 ppm; IR ν_{max} (cm^{-1}) 3420, 3269, 3054, 2984, 2935, 2875, 2685, 2306, 2253, 1711, 1546; HRMS (ESI) calcd for $C_{11}H_{16}N_2S$ (M+) 209.1107, found 209.1103.



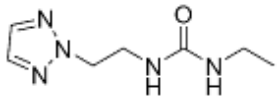
1-(2,3-dihydro-1H-inden-2-yl)-3-ethylthiourea (**3h**): Grey solid. mp=87-91 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.14 (m, 4H), δ 6.62 (s, 2H), δ 4.85 (m, 1H), δ 3.50 – 3.21 (m, 4H), δ 2.83 (dd, J = 5.4, 5.1 Hz, 2H), δ 1.10 (t, J = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.0, 140.8, 127.1, 125.0, 100.4, 55.5, 40.0, 14.6 ppm; IR ν_{max} (cm^{-1}) 3267, 3066, 2972, 1673, 1483, 1548; HRMS (ESI) calcd for $C_{12}H_{16}N_2S$ (M+) 221.1107, found 221.1106.



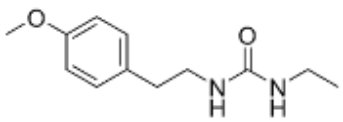
1-(4-bromophenethyl)-3-ethylurea (**11g**): White solid. mp=137-139 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (d, J = 8.1 Hz, 2H), δ 7.02 (d, J = 8.1 Hz, 2H), δ 5.46 (d, J = 16.5 Hz, 2H), δ 3.30 (m, 2H), δ 3.08 (q, J = 6.0, 6.9 Hz, 2H), δ 2.67 (t, J = 6.9 Hz, 2H), δ 1.05 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.1, 138.6, 131.7, 130.7, 120.3, 41.6, 36.3, 35.2, 15.8 ppm; IR ν_{max} (cm^{-1}) 3327, 2971, 1620, 1488; HRMS (ESI) calcd for $C_{11}H_{15}BrN_2O$ (M+) 271.0441, found 271.0439.



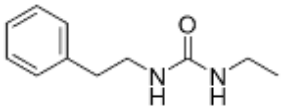
1-(2,3-dihydro-1H-inden-2-yl)-3-ethylurea (**3g**): White solid. mp=117 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.14 (m, 4H), δ 5.98 (s, 2H), δ 4.43 (m, 1H), δ 3.22 – 3.07 (m, 4H), δ 2.77 (dd, J = 6.3, 5.7 Hz, 2H), δ 1.07 (t, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.3, 141.4, 126.8, 124.9, 51.5, 40.7, 35.1, 15.9 ppm; IR ν_{max} (cm^{-1}) 3348, 2968, 1623, 1579, 1259, 736; HRMS (ESI) calcd for $C_{12}H_{16}N_2O$ (M+) 205.1335, found 205.1333.



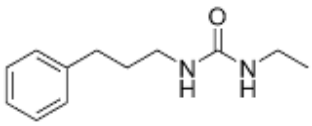
1-(2-(2H-1,2,3-triazol-2-yl)ethyl)-3-ethylurea (**5g**): White solid. mp=109 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (s, 2H), δ 6.63 (d, J = 25.5 Hz, 2H), δ 4.62 (m, 2H), δ 4.10 (t, J = 5.1 Hz, 2H), δ 3.28 (m, 2H), δ 1.16 (t, J = 8.7 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.6, 134.7, 54.3, 44.2, 38.7, 14.1 ppm; IR ν_{max} (cm^{-1}) 3434, 1640; HRMS (ESI) calcd for $C_7H_{13}N_5O$ (M+) 206.1012, found 206.1014.



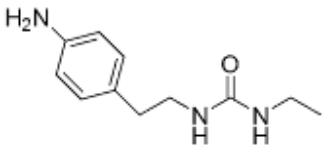
1-ethyl-3-(4-methoxyphenethyl)urea (**12g**): Light yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.03 (d, J = 8.7 Hz, 2H), δ 6.78 (t, J = 3.9 Hz, 2H), δ 5.85 (q, J = 5.1, 6.3 Hz, 2H), δ 3.73 (s, 3H), δ 3.31 (q, J = 6.6, 7.2 Hz, 2H), δ 3.12 (m, 2H), δ 2.67 (t, J = 7.5 Hz, 2H), δ 1.08 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 158.3, 131.7, 129.9, 114.1, 55.4, 42.1, 36.1, 35.1, 15.8 ppm; IR ν_{max} (cm^{-1}) 3374, 2977, 2837, 1630; HRMS (ESI) calcd for $C_{12}H_{18}N_2O_2$ (M+) 245.1260, found 245.1263.



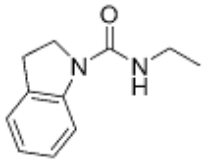
1-ethyl-3-phenethylurea (**2g)**: White solid. mp=75-77 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (m, 5H), δ 5.92 (d, J = 16.2 Hz, 2H), δ 3.86 (q, J = 6.6, 6.0 Hz, 2H), δ 3.14 (m, 2H), δ 2.78 (t, J = 7.8 Hz, 2H), δ 1.10 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 139.7, 129.0, 126.5, 41.9, 37.1, 35.1, 15.9 ppm; IR ν_{max} (cm^{-1}) 3359, 2972, 2873, 2239, 1633, 1259; HRMS (ESI) calcd for $C_{11}H_{16}N_2O$ (M+) 193.1335, found 193.1334.



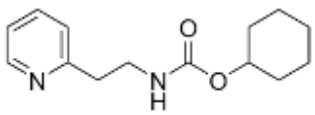
1-ethyl-3-(3-phenylpropyl)urea (**4g**): White solid. mp=48 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.2 (m, 5H), δ 6.15 (s, 1H), δ 6.08 (s, 1H), δ 3.26 (m, 4H), δ 2.69 (t, J = 9 Hz, 2H), δ 1.84 (m, 2H), δ 1.14 (t, J = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.0, 142.0, 128.6, 126.1, 40.0, 35.2, 33.5, 32.4, 15.9 ppm; IR ν_{max} (cm^{-1}) 3358, 2971, 2867, 1631; HRMS (ESI) calcd for $C_{12}H_{18}N_2O$ (M+) 207.1492, found 207.1489.



1-(4-aminophenethyl)-3-ethylurea (**8g**): Light yellow solid. mp= 101-103 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.94 (d, J = 8.4, 2H), δ 6.58 (d, J = 6.0 Hz, 2H), δ 5.05 (t, J = 3.3 Hz, 2H), δ 3.59 (s, 2H), δ 3.28 (q, J = 6.9, 6.9 Hz, 2H), δ 3.10 (m, 2H), δ 2.63 (t, J = 6.9 Hz, 2H), δ 1.04 (t, J = 7.2, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.0, 145.0, 129.8, 115.5, 42.1, 35.9, 15.7 ppm; IR ν_{max} (cm^{-1}) 3409, 1635, 1517, 1263; HRMS (ESI) calcd for $C_{11}H_{17}N_3O$ (M+) 208.1444, found 208.1445.

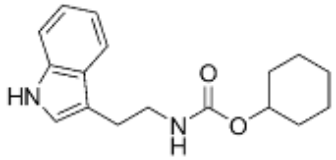


N-ethylindoline-1-carboxamide (**6g**): Light yellow solid. mp=111-113 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 (d, J = 8.1 Hz, 1H), δ 7.11 – 7.04 (m, 2H), δ 6.82 (t, J = 7.5 Hz, 1H), δ 5.09 (s, 1H), δ 3.78 (t, J = 8.7 Hz, 2H), δ 3.27 (m, 2H), δ 3.02 (t, J = 9.0 Hz, 2H), δ 1.15 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm; IR ν_{max} (cm^{-1}) 3403, 2959, 1646; HRMS (ESI) calcd for $C_{11}H_{14}N_2O$ (M+) 190.1179, found 190.1177.

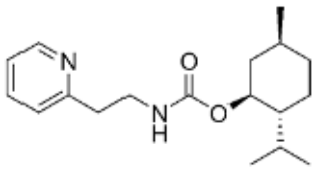


cyclohexyl 2-(pyridin-2-yl)ethylcarbamate (**9l**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 (d, J = 4.8 Hz, 1H), δ 7.43 (t, J = 9.9 Hz, 1H), δ 7.02 – 6.94 (m, 2H), δ 5.72 (s, 1H), δ 4.45 (d, J = 3.6 Hz, 1H), δ 3.40 (q, J = 6.6, 6.3 Hz, 2H), δ 2.83 (t, J = 6.6 Hz, 2H), δ 1.702 – 1.027 (m, 10H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.4, 156.5, 149.1, 136.8, 123.6, 121.6, 40.4, 37.9,

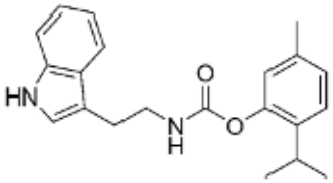
32.2, 25.5, 23.9 ppm; IR ν_{max} (cm^{-1}) 3944, 3692, 3054, 2987, 1709; HRMS (ESI) calcd for $C_{14}H_{20}N_2O_2$ (M+) 249.1598, found 249.1595.



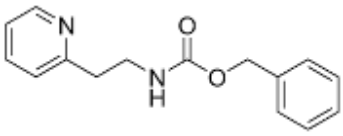
cyclohexyl 2-(1H-indol-3-yl)ethylcarbamate (**10l**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.74 (s, 1H), δ 7.65 (d, J = 6.9 Hz, 1H), δ 7.38 (d, J = 7.2 Hz, 1H), δ 7.20 (dd, J = 7.2, 7.2 Hz, 2H), δ 6.96 (s, 1H), δ 4.94 (s, 1H), δ 4.72 (s, 1H), δ 3.63 (d, J = 34.2 Hz, 2H), δ 2.98 (s, 2H), δ 1.91 – 1.31 (m, 10H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.8, 136.8, 127.6, 122.6, 119.5, 112.8, 73.3, 70.6, 41.6, 35.8, 32.4, 26.1, 24.5 ppm; IR ν_{max} (cm^{-1}) 3334, 2909, 2991, 1701;HRMS (ESI) calcd for $C_{17}H_{22}N_2O_2$ (M+) 287.1755, found 287.1752.



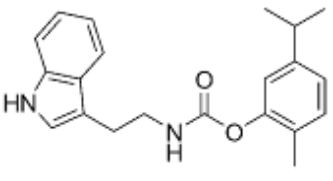
(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 2-(pyridin-2-yl)ethylcarbamate (**9k**): Yellow solid. mp=57-59 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 (d, J = 4.5 Hz, 1H), δ 7.45 (t, J = 5.7 Hz, 1H), δ 6.98 (m, 2H), δ 5.55 (s, 1H), δ 4.40 (t, J = 3.9 Hz, 1H), δ 3.43 (q, J = 6.3 Hz, 2H), δ 2.85 (t, J = 6.3 Hz, 2H), δ 2.01 – 0.62 (m, 19H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5, 156.7, 136.3, 123.6, 121.6, 74.2, 47.5, 41.6, 40.4, 37.9, 34.4, 31.5, 26.7, 23.6, 22.2, 20.9, 16.6 ppm; IR ν_{max} (cm^{-1}) 3406, 2954, 2868, 1695, 1694, 1514, 1260; HRMS (ESI) calcd for $C_{18}H_{28}N_2O_2$ (M+) 305.2224, found 305.2221.



2-isopropyl-5-methylphenyl 2-(1H-indol-3-yl)ethylcarbamate (**10i**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.71 (s, 1H), δ 7.67 (d, J = 7.5, 1H), δ 7.37 (d, J = 8.1 Hz, 1H), δ 7.25 (t, J = 7.2 Hz, 1H), δ 7.20 (t, J = 7.2 Hz, 1H), δ 6.96 (s, 1H), δ 4.92 (s, 1H), δ 3.52 (t, J = 6.3 Hz, 2H), δ 3.01 (t, J = 6.3 Hz, 2H), δ 1.26 – 1.03 (m, 6H), δ 0.99 (d, J = 6.6 Hz, 3H), δ 0.95 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.1, 136.8, 127.7, 122.6, 119.0, 112.9, 111.7, 74.9, 47.6, 41.8, 34.6, 31.7, 26.5, 23.8, 22.4, 21.2, 16.8 ppm; IR ν_{max} (cm^{-1}) 3408, 2962, 2926, 1717, 1620, 1502, 1457; HRMS (ESI) calcd for $C_{21}H_{24}N_2O_2$ (M+) 337.1911, found 337.1914.

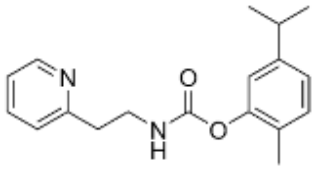


benzyl 2-(pyridin-2-yl)ethylcarbamate (**9c**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.34 (d, J = 6.6 Hz, 1H), δ 7.46 (t, J = 7.8 Hz, 1H), δ 6.98 (dd, J = 7.8, 7.5 Hz, 2H), δ 5.72 (s, 1H), δ 4.45 (d, J = 7.2 Hz, 1H), δ 3.42 (q, J = 6.6, 6.3 Hz, 2H), δ 2.83 (t, J = 6.6 Hz, 2H), δ 1.70 – 1.03 (m, 10H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.4, 156.5, 149.1, 136.8, 123.6, 121.6, 72.8, 40.4, 37.86, 32.2, 25.5, 23.9 ppm; IR ν_{max} (cm^{-1}) 3435, 2092, 1644, 1261; HRMS (ESI) calcd for $C_{15}H_{16}N_2O_2$ (M+) 257.1285, found 257.1282.

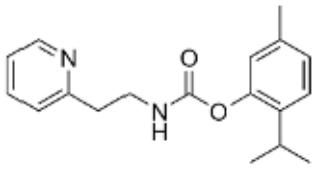


5-isopropyl-2-methylphenyl 2-(1H-indol-3-yl)ethylcarbamate (**10j**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.47 (s, 1H), δ 7.74 (d, J = 7.8 Hz, 1H), δ 7.37 – 7.05 (m, 6H), δ 6.95 (s, 1H), δ 5.35 (s,

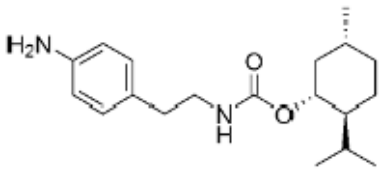
1H), δ 3.69 (t, J = 6.6 Hz, 2H), δ 3.10 (t, J = 6.6 Hz, 2H), δ 2.96 (m, 1H), δ 2.24 (s, 3H), δ 1.33 (d, J = 6.6 Hz, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.24, 149.7, 148.4, 136.8, 131.2, 128.2, 124.7, 122.7, 120.6, 119.6, 118.9, 112.5, 42.0, 33.9, 26.0, 24.3, 16.1 ppm; IR ν_{max} (cm^{-1}) 3336, 2969, 2926, 1719, 1503, 1457; HRMS (ESI) calcd for $C_{21}H_{24}N_2O_2$ (M+) 337.1911, found 337.1914.



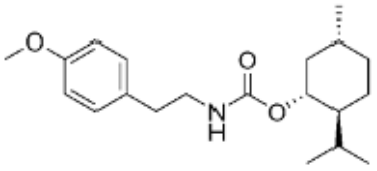
5-isopropyl-2-methylphenyl 2-(pyridin-2-yl)ethylcarbamate (**9j**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.55 (d, J = 3.9 Hz, 1H), δ 7.67 (t, J = 7.8 Hz, 1H), δ 7.27 – 6.88 (m, 5H), δ 6.46 (t, J = 5.4 Hz, 1H), δ 3.68 (q, J = 6.6, 6.3 Hz, 2H), δ 3.12 (t, J = 6.6 Hz, 2H), δ 2.83 (m, 1H), δ 2.13 (s, 3H), δ 1.23 (d, J = 5.1 Hz, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 155.0, 149.6, 148.1, 138.4, 131.0, 128.0, 124.5, 123.9, 122.4, 120.4, 40.8, 37.0, 33.8, 24.2, 15.9 ppm; IR ν_{max} (cm^{-1}) 3054, 2987, 2306, 1734, 1501; HRMS (ESI) calcd for $C_{18}H_{22}N_2O_2$ (M+) 299.1755, found 299.1753.



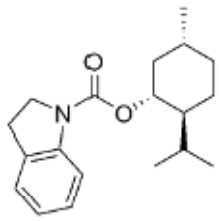
2-isopropyl-5-methylphenyl 2-(pyridin-2-yl)ethylcarbamate (**9i**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.54 (d, J = 4.8 Hz, 1H), δ 7.65 (t, J = 7.5 Hz, 1H), δ 7.25 – 6.94 (m, 4H), δ 6.83 (s, 1H), δ 6.47 (s, 1H), δ 3.68 (q, J = 6.0, 6.0 Hz, 2H), δ 3.07 (m, 2H), δ 2.27 (s, 3H), δ 1.16 (d, J = 6.0 Hz, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 155.4, 148.4, 138.2, 137.8, 136.5, 126.9, 126.5, 124.4, 123.4, 122.3, 40.8, 37.1, 27.1, 23.3, 21.1 ppm; IR ν_{max} (cm^{-1}) 3054, 2966, 1735, 1250; HRMS (ESI) calcd for $C_{18}H_{22}N_2O_2$ (M+) 299.1755, found 299.1759.



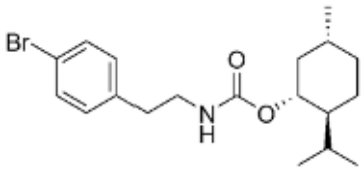
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-aminophenethylcarbamate (8a): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.93 (d, J = 8.1 Hz, 2H), δ 6.60 (d, J = 8.4 Hz, 2H), δ 4.79 (t, J = 5.7 Hz, 1H), δ 4.53 (t, J = 3.9 Hz, 1H), δ 3.59 (s, 2H), δ 3.33 (q, J = 4.5, 5.7 Hz, 2H), δ 2.65 (t, J = 6.6 Hz, 2H), δ 2.03 (d, J = 6.9 Hz, 1H), δ 1.87 (t, 6.6 Hz, 1H), δ 1.65 (d, 10.5 Hz, 2H), δ 1.43 (s, 3H), δ 1.25 (t, 6.4 Hz, 1H), δ 1.01 (q, J = 2.7, 2.7 Hz, 1H), δ 0.92 (m. 6 H), δ 0.79 (d, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.7, 145.1, 129.8, 128.9, 115.6, 47.6, 42.6, 41.7, 35.4, 34.5, 31.6, 26.5, 23.8, 22.3, 21.1, 16.7 ppm; IR ν_{max} (cm^{-1}) 3442, 2955, 2869, 1698, 1626, 1264; HRMS (ESI) calcd for $C_{19}H_{30}N_2O_2$ (M+) 319.2381, found 319.2383.



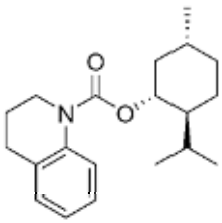
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-methoxyphenethylcarbamate (**12a**): White solid. mp=95-98 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.09 (d, J = 8.7 Hz, 2H), δ 6.84 (d, J = 8.7 Hz, 2H), δ 4.78 (t, J = 5.7 Hz, 1H), δ 4.56 (t, 3.9 Hz, 1H), δ 3.74 (s, 3H), δ 3.36 (q, J = 6.6, 5.7 Hz, 2H), δ 2.73 (t, 7.2 Hz, 2H), δ 1.99 (d, J = 4.8 Hz, 1H), δ 1.66 (t, J = 10.2 Hz, 1H), δ 1.48 (d, J = 3.0 Hz, 2H), δ 1.47 (t, J = 3.3 Hz, 1H), δ 1.07 (t, 3.5 Hz, 1H), δ 1.03 (q, 3.5, 3.0 Hz, 1H), δ 0.93 (m, 6H), δ 0.80 (d, 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.4, 156.7, 131.1, 129.9, 114.2, 55.4, 47.6, 41.7, 35.5, 34.6, 31.6, 26.5, 23.8, 22.3, 21.1, 16.7 ppm; IR ν_{max} (cm^{-1}) 3372, 2954, 2869, 1684, 1512, 1455, 1242, 1178, 1127, 1056, 623; HRMS (ESI) calcd for $C_{20}H_{31}NO_3$ (M+) 356.2196, found 356.2199.



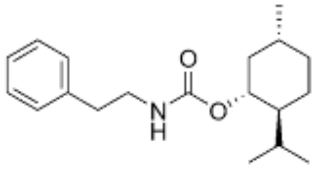
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl indoline-1-carboxylate (**6a**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 (s, 1H), δ 7.26 (m, 2H), δ 6.91 (m, 1H), δ 4.75 (s, 1H), δ 3.99 (s, 2H), δ 3.89 (t, 6.6 Hz, 2H), δ 2.19 (d, J = 8.7 Hz, 1H), δ 1.95 (s, 1H), δ 1.55 (d, J = 2.4 Hz, 2H), δ 1.41 (s, 2H), δ 1.12 (m, 2H), δ 0.84 (m, 10H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 127.7, 124.9, 122.5, 114.9, 47.8, 41.9, 34.7, 34.6, 31.7, 27.6, 26.7, 26.6, 23.8, 22.3, 21.1, 16.8, 16.7 ppm; IR ν_{max} (cm^{-1}) 3428, 2955, 2869, 1704, 1604, 1488; HRMS (ESI) calcd for $C_{19}H_{27}NO_2$ (M+) 324.1934, found 324.1929.



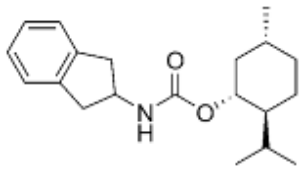
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-bromophenethylcarbamate (**11a**): White solid. mp=104-106 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, J = 5.7 Hz, 2H), δ 7.03 (d, 6.0 Hz, 2H), δ 4.74 (s, 1H), δ 4.51 (t, J = 2.7 Hz, 1H), δ3.35 (d, J = 4.5 Hz, 2H), δ 2.73 (s, 2H), δ 1.99 (d, J = 9.0 Hz, 1H), δ 1.64 (t, J = 1.5 Hz, 1H), δ 1.63 (t, J = 2.4 Hz, 2H), δ 1.41 (m, 1H), δ 1.04 (t, J = 3.8 Hz, 1H), δ 1.03 (q, J = 2.4 Hz, 2H), δ 0.97 (m, 6H), δ 0.76 (d, J = 5.1 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 1.56.6, 138.1, 131.8, 130.8, 120.5, 74.7, 47.6, 42.1, 41.7, 35.8, 34.5, 31.6, 26.5, 23.8, 22.3, 21.0, 16.7 ppm; IR ν_{max} (cm^{-1}) 3364, 2953, 2856, 1684, 1256; HRMS (ESI) calcd for $C_{19}H_{28}BrNO_2$ (M+) 404.1196, found 404.1191.



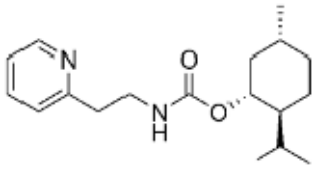
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 3,4-dihydroquinoline-1(2H)-carboxylate (**7a**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, J = 8.4 Hz, 1H), δ 7.18 (t, J = 1.5 Hz, 1H), δ 7.13 (d, J = 1.8 Hz, 1H), δ 6.97, J = 0.9 Hz, 1H), δ 4.77 (t, J = 4.2 Hz, 1H), δ 3.77 (t, J = 0.9 Hz, 2H), δ 2.78 (t, J = 6.6 Hz, 2H), δ 1.18 (d, J = 3.0 Hz, 1H), δ 1.99 (m, 3H), δ 1.68 (d, J = 2.7 Hz, 2H), δ 1.414 (m, 2H), δ 1.13 (m, 2H), δ 1.10 (m, 6H), δ 0.87 (d, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.9, 138.7, 129.9, 128.8, 126.1, 124.2, 123.6, 76.5, 47.5, 44.9, 41.7, 34.6, 31.7, 27.7, 26.7, 23.8, 23.8, 22.3, 21.1, 16.7 ppm; IR ν_{max} (cm^{-1}) 3434, 2954, 2870, 2105, 1694; HRMS (ESI) calcd for $C_{20}H_{29}NO_2$ (M+) 338.2091, found 338.2088.



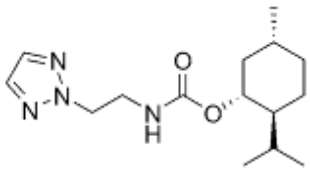
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl phenethylcarbamate (**2a**): White solid. mp=84-86 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (m, 5H), δ 4.91 (t, J = 4.2 Hz, 1H), δ 4.58 (s, 1H), δ 3.39 (s, 2H), δ 2.79 (t, J = 7.8 Hz, 2H), δ 2.05 (d, J = 8.7 Hz, 1H), δ 1.93 (t, J = 4.2 Hz, 1H), δ 1.66 (s, 2H), δ 1.47 (s, 1H), δ 1.28 (t, J = 3.8 Hz, 1H), δ 1.04 (q, 10.2, 9.3 Hz, 2H), δ 0.91 (m, 8H), δ 0.80 (d, J = 0.60, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.7, 139.2, 129.1, 128.9, 128.8, 128.7 , 126.6, 126.5, 74.6, 47.6, 45.3, 42.4, 41.9, 41.8, 36.5, 34.9, 31.6, 26.5, 23.8, 23.4, 22.6, 22.3, 21.1, 16.4 ppm; IR ν_{max} (cm^{-1}) 3360, 2957, 1682, 1526, 1259, 1024, 798; HRMS (ESI) calcd for $C_{19}H_{29}NO_2$ (M+) 343.2381, found 343.2383.



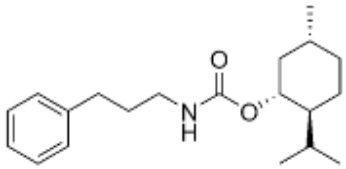
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 2,3-dihydro-1H-inden-2-ylcarbamate (**3a**): White solid. mp=167-170 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 (m, 4H), δ 4.99 (s, 1H), δ 4.57 (m, 2H), δ 3.25 (m, 3H), δ 2.75 (m, 2H), δ 2.02 (d, J = 11.7 Hz, 2H), δ 1.94 (t, J = 2.1 Hz, 1H), δ 1.67 (d, J = 12.9, 2H), δ 1.58 (s, 1H), δ 1.45 (t, J = 3.3 Hz, 1H), 1.05 (q, J = 3.0, 7.5 Hz, 2H), δ 0.93 (m, 6H), δ 0.78 (d, J = 7.2 Hz, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 126.9, 126.8, 124.9, 124.8, 51.6, 50.3, 47.6, 45.3, 41.7, 40.8, 40.6, 34.8, 34.5, 31.0, 31.6, 26.5, 23.7, 23.4, 22.5, 22.3, 21.3, 21.1, 16.6, 16.3 ppm; IR ν_{max} (cm^{-1}) 3409, 1688; HRMS (ESI) calcd for $C_{20}H_{29}NO_2$ (M+) 338.2091, found 338.2098.



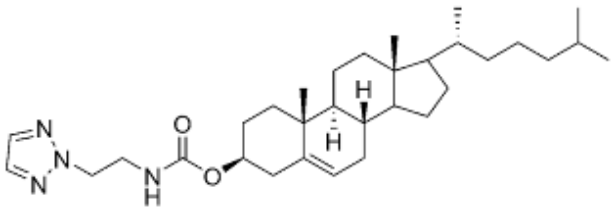
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 2-(pyridin-2-yl)ethylcarbamate (**9a**): White solid. mp=59-63 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.50 (q, J = 0.6, 2.4 Hz, 1H), δ 7.61 (t, 2.1 Hz, 1H), δ 7.13 (m, 2H), δ 5.75 (s, 1H), δ 4.54 (t, J = 7.2 Hz, 1H), δ 3.59 (q, J = 5.7, 6.3 Hz, 2H), δ 2.99 (t, J = 6.3 Hz, 2H), δ 2.03 (d, J = 11.7 Hz, 1H), δ 1.89 (m, 1H), δ 1.59 (tm, 2H), δ 1.27 (s, 1H), δ 1.27 (t, J = 10.8 Hz, 1H), δ 0.97 (m, 2H), δ 0.91 (m, 6H), δ 0.76 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5, 156.6, 140.3, 136.5, 123.6, 121.5, 74.2, 71.1, 50.1, 47.5, 41.6, 40.4, 37.9, 34.7, 31.8, 31.4, 26.3, 23.6, 20.9, 16.2 ppm; IR ν_{max} (cm^{-1}) 3406, 2954, 2868, 1695, 1694, 1514, 1260; HRMS (ESI) calcd for $C_{18}H_{28}N_2O_2$ (M+) 305.2224, found 305.2229.



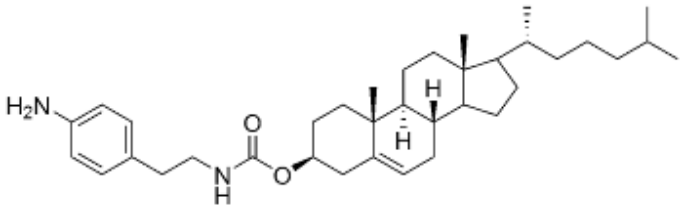
(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamate (**5a**): White residue. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (s, 2H), δ 5.11 (s, 1H), δ 4.54 (s, 2H), δ 3.72 (s, 2H), δ 3.24 (s, 1H), δ 1.90 (m, 2H), δ 1.61 (s, 1H), δ 1.52 (s, 2H), δ 1.45 (s, 1H), δ 1.24 (s, 1H), 1.09 (m, 2H), δ 1.05 (m, 8H), δ 0.76 (d, J = 4.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.5, 134.6, 74.8, 71.7, 54.7, 50.3, 47.7, 47.5, 41.8, 40.5, 34.5, 31.6, 26.5, 23.7, 22.2, 16.3 ppm; IR ν_{max} (cm^{-1}) 3434, 2955, 2088, 1642, 1256; HRMS (ESI) calcd for $C_{15}H_{26}N_4O_2$ (M+) 304.4125, found 304.4129.



(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 3-phenylpropylcarbamate (**4a**): White solid. mp=75-78 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 (t, J = 7.8 Hz, 2H), δ 7.16 (d, J = 7.2 Hz, 2H), δ 4.86 (s, 1H), δ 4.55 (t, J = 6.9 Hz, 1H), δ 3.201 (q, J = 6.6, 6.0 Hz, 2H), δ 2.64 (t, J = 7.5 Hz, 2H), δ 2.05 (d, J = 11.7 Hz, 1H), δ 1.95 (t, J = 2.7 Hz, 1H), δ 1.89 (m, 2H), δ 1.66 (d, J = 10.8 Hz, 2H), δ 1.48 (s, 1H), δ 1.06 (t, J = 8.4 Hz, 1H), δ 1.06 (m, 2H), δ 1.02 (m, 8H), δ 0.80 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9, 141.8, 128.7, 128.6, 126.2, 126.1, 74.6, 47.4, 41.8, 40.1, 34.6, 33.5, 33.3, 31.9, 31.6, 26.6, 23.8, 22.4, 16.8 ppm; IR ν_{max} (cm^{-1}) 3410, 3057, 2960, 1713, 1421, 1362, 1267, 1222, 191, 846, 736, 702; HRMS (ESI) calcd for $C_{20}H_{31}NO_2$ (M+) 340.2247, found 340.2241.

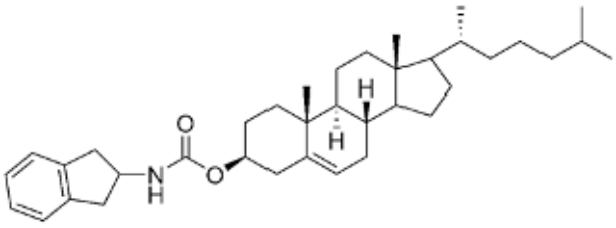


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 2-(2H-1,2,3-triazol-2-yl)ethylcarbamate (**5f**): White solid. mp= 135-139 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (s, 2H), δ 5.31 (t, 6.9 Hz, 1H), δ 4.53 (t, J = 5.1 Hz, 1H), δ 3.69 (q, J = 5.1 Hz, 2H) δ 3.47 (m, 2H), δ 2.49 (s, 1H), δ 2.45 (q, J = 9.3, 4.2 Hz, 2H), δ 1.96 (m, 6H), δ 1.54-1.30 (m, 11H), δ 1.25-1.09 (m, 14H), δ1.01-0.82 (m, 10H), δ0.65 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ141.1, 134,6, 121.8, 71.8, 57.0, 56.9, 50.3, 42.5, 40.0, 39.8, 37.5, 36.7, 36.4, 36.1, 32.1, 31.8, 28.5, 28.2, 24.5, 24.1, 23.1, 229, 21.3, 19.6, 19.5, 18.9, 12.1 ppm; IR ν_{max} (cm^{-1}) 3398, 2037, 1637; HRMS (ESI) calcd for $C_{32}H_{52}N_4O_2$ (M+) 547.3982, found 547.3981.

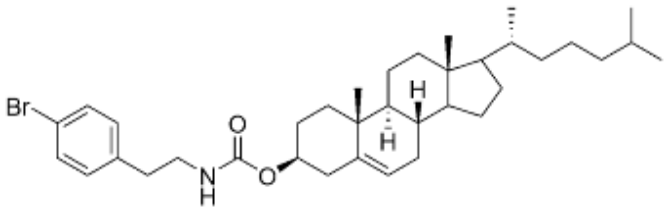


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-aminophenethylcarbamate (**8f**): Yellow hygroscopic solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.93 (d, J = 7.5 Hz, 2H), δ 6.588 (d, J = 7.5 Hz, 2H), δ 5.35 (m, 1H), δ 4.92 (s, 1H), δ 4.47 (m, 1H), δ3.32 (d, J = 6.0 Hz, 2H), δ 2.54 (t, J = 6.6 Hz, 2H), δ 2.31 (m, 2H), δ 2.02-1.98 (m, 5H), δ 1.54-1.27 (m, 11H), δ 1.10-0.85 (m, 24H), δ 0.67 (s, 3H)ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4, 145.2, 141.2, 140.1, 129.8, 128.8, 122.7, 115.6, 74.4, 57.-, 56.9, 56.4, 50.2, 42.6, 40.0, 39.8, 38.9, 37.3, 36.8, 36.5, 35.5, 32.1, 31.8, 28.4, 28.3, 24.5, 24.2, 23.2, 22.0, 21.3, 10.7, 19.5, 19.0,

12.1 ppm; IR ν_{max} (cm^{-1}) 3407, 2937, 2868, 2245, 1695, 1631, 1516; HRMS (ESI) calcd for $C_{36}H_{56}N_2O_2$ (M+) 549.4415, found 549.4420.

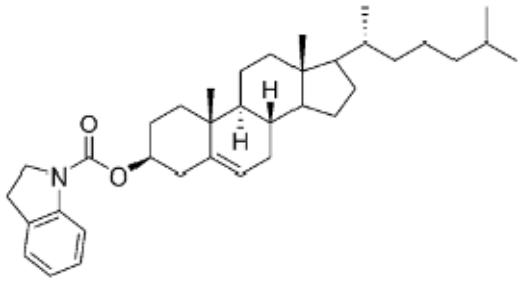


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 2,3-dihydro-1H-inden-2-ylcarbamate (**3f**): mp=114-116 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 (m, 4H), δ 5.37 (d, J = 6.6Hz, 1H), δ 5.15 (m, 1H), δ 4.52 (s, 1H), δ 3.40 (m, 1H), δ 3.24 (m, 2H), δ 2.78 (m, 2H), δ 2.27 (m, 3H), δ 2.04-1.84 (m, 5H), δ 1.55-1.26 (m, 11H), δ 1.12-0.87 (m, 23H), δ 0.69 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.4, 126.9, 125.0, 124.9, 122.8, 121.89, 71.9, 57.0, 56.9, 56.4, 52.4, 51.7, 50.4, 50.2, 42.5, 42.5, 40.8, 40.5, 40.1, 40.0, 39.8, 37.6, 36.8, 36.5, 36.1, 32.2, 32.1, 31.8, 28.5, 28.5, 24.6, 24.2, 23.1, 22.9, 21.4, 21.3, 19.7, 19.6, 19.0, 12.1 ppm; IR ν_{max} (cm^{-1}) 3358, 2937, 2867, 1693, 1551, 1466; HRMS (ESI) calcd for $C_{37}H_{55}NO_2$ (M+) 568.4125, found 568.4136.

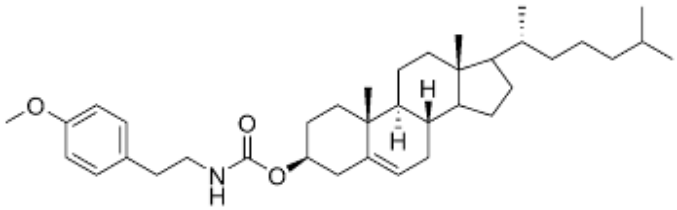


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-bromophenethylcarbamate (**11f**)**:** White solid. mp=142-145 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (d, J = 8.4 Hz, 2H), δ 6.99 (d, J = 8.4 Hz, 2H), δ 5.34 (s, 1H), δ 4.98 (t, J = 5.7, 1H), δ

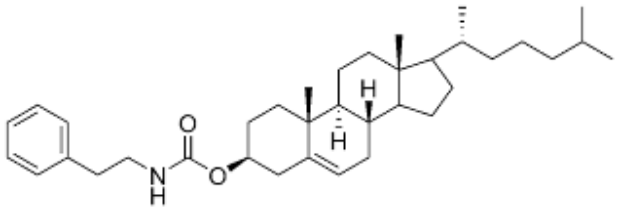
4.44 (m, 1H), δ 3.34 (q, J = 6.6 Hz, 1H), δ 2.72 (t, J = 6.6 Hz, 2H), δ 2.27 (m, 2H), δ 2.32-1.79 (m, 5H), δ 1.52-1.34 (m, 11H), δ 1.22-0.86 (m, 22H), δ 0.65 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.3, 156.3, 139.1, 138.1, 131.9, 131.7, 130.8, 122.8, 120.5, 74.5, 56.9, 57.4, 50.2, 42.5, 42.1, 39.9, 39.8, 38.8, 37.3, 36.8, 36.5, 36.1, 32.1, 29.6, 28.4, 28.2, 24.5, 24.2, 23.1, 22.9, 21.3, 19.6, 19.0, 12.1 ppm; IR ν_{max} (cm^{-1}) 3435, 2947, 2867, 2249, 1793, 1488; HRMS (ESI) calcd for $C_{36}H_{54}BrNO_2$ (M+) 634.3230, found 634.3210.



(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl indoline-1-carboxylate (**6f**): White solid. mp=157-159 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (s, 1H), δ 7.16 (t, 2H), δ 6.93 (t, J = 7.5 Hz, 1H), δ 5.44 (d, J = 4.5Hz, 1H), δ 4.68 (s, 1H), δ 3.98 (t, J = 7.5Hz, 2H), δ 3.07 (t, J = 8.7Hz, 2H), δ 2.07-1.84 (m, 5H), δ 1.59-1.28 (m, 11H), δ 1.21-1.04 (m, 10H), δ 1.03-0.90 (m, 12H), δ 0.72 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.3, 149.9, 139.9, 127.7, 122.9, 122.6, 115.1, 75.1, 56.9, 56.5, 50.3, 47.6, 42.6, 40.0, 39.8, 37.7, 37.3, 36.8, 36.8, 36.4, 32.2, 32.1, 31.8, 28.6, 28.3, 27.6, 24.6, 24.3, 23.2, 22.9, 21.4, 19.7, 19.0, 12.2 ppm; IR ν_{max} (cm^{-1}) 3444, 2944, 1699, 1604; HRMS (ESI) calcd for $C_{36}H_{53}NO_2$ (M+) 532.4150, found 532.4152.

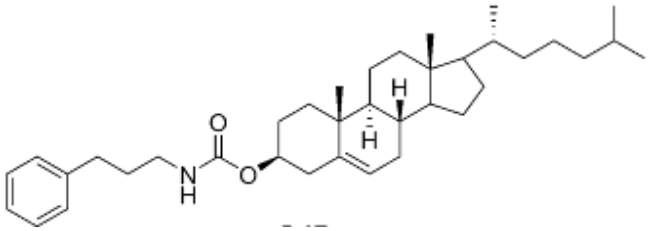


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-methoxyphenethylcarbamate (**12f**): White residue. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.10 (d, J = 6.6Hz, 2H), δ 6.83 (d, J = 6.6Hz, 2H), δ 5.37 (d, J = 4.8Hz, 1H), δ 4.88 (s, 1H), δ 4.48 (m, 1H), δ 3.75 (s, 3H), δ 3.36 (q, J = 6.0, 6.6Hz, 2H), δ 2.72 (t, J = 6.9Hz, 2H), δ 2.32 (m, 2H), δ 1.98-1.81 (m, 5H), δ 1.52-1.36 (m, 11H), δ 1.28-1.13 (m, 12H), δ 1.11-0.86 (m, 10H), δ 0.67 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.5, 156.4, 140.0, 131.1, 129.9, 122.7, 114.2, 114.1, 74.5, 56.9, 56.4, 55.4, 50.2, 42.5, 39.9, 39.8, 38.8, 37.3, 36.8, 36.5, 36.1, 35.5, 32.1, 28.5, 28.4, 28.3, 24.5, 24.1, 23.1, 22.9, 21.3, 19.6, 18.9, 12.1 ppm; IR ν_{max} (cm^{-1}) 3418, 2937, 2867, 1695, 1512, 1465, 1246, 733; HRMS (ESI) calcd for $C_{37}H_{57}NO_3$ (M+) 586.4231, found 586.4230.

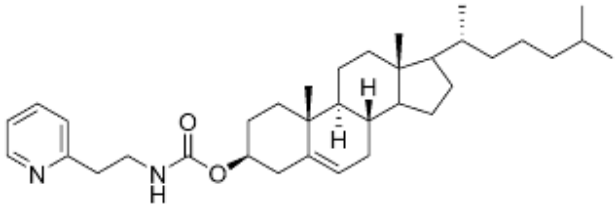


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl phenethylcarbamate (**2f**): White solid. mp=74-76 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (t, J = 1.2Hz, 2H), δ 7.18 (m, 3H), δ 5.36 (d, J = 2.7Hz, 1H), δ 4.83 (s, 1H), δ 4.48 (m, 1H), δ 3.39 (d, J = 4.5Hz, 2H), δ 2.33 (m, 2H), δ 2.02-1.82 (m, 5H), δ 1.55-1.33 (m, 18H), δ 1.12-1.03 (m, 7H), δ 0.96-0.86 (m, 9H), δ 0.67 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.36, 140.1, 129.1,

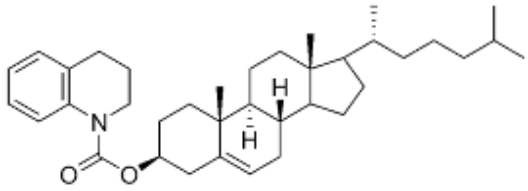
129.0, 128.8, 128.7, 126.7, 122.7, 56.0, 56.5, 50.3, 42.6, 42.4, 40.0, 39.8, 38.9, 37.3, 36.8, 36.5, 36.1, 32.2, 32.1, 28.5, 28.5, 28.3, 24.6, 23.2, 22.9, 21.3, 10.6, 10.0, 12.1 ppm; IR ν_{max} (cm^{-1}) 3419, 2944, 2867, 2089, 1694, 1255, 1137; HRMS (ESI) calcd for $C_{36}H_{55}NO_2$ (M+) 556.4125, found 556.4128.



(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 3-phenylpropylcarbamate (**4f**): White solid. mp=87-89 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 (t, J = 3.3 Hz, 2H), δ 7.25 (t, J = 5.4 Hz, 3H), δ 5.35 (d, J = 3.6 Hz, 1H), δ 4.89 (t, J = 4.5 Hz, 1H), δ 4.49 (m, 1H), δ 3.17 (d, J = 4.8 Hz, 2H), δ 2.35 (m, 2H), δ 2.01-1.78 (m, 8H), δ 1.55=1.41 (m, 11H), δ 1.29-1.12 (m, 12H), δ 1.05-0.91 (m, 11H), δ 0.67 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.5, 141.7, 140.1, 128.7, 128.6, 126.2, 122.7, 74.4, 56.9, 56.5, 50.3, 42.6, 50.7, 50.0, 39.8, 38.9, 37.3, 36.9, 36.5, 35.1, 33.4, 32.1, 31.9, 28.5, 28.4, 28.3, 24.5, 24.2, 23.2, 22.9, 21.3, 19.6, 19.0, 12.2 ppm; IR ν_{max} (cm^{-1}) 3419, 2944, 2867, 2089, 1694, 1255, 1137; HRMS (ESI) calcd for $C_{37}H_{57}NO_2$ (M+) 570.4282, found 570.4287.

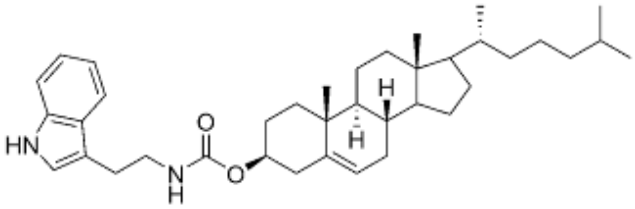


(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 2-(pyridin-2-yl)ethylcarbamate (**9f**) . White residue. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.50 (d, J = 3.6 Hz, 1H), δ 7.59 (t, 0.9Hz, 1H), δ 5.7 (d, J = 5.7 Hz, 1H), δ 7.09 (t, J = 5.7Hz, 1H), δ 5.73 (s, 1H), δ 5.35 (s, 1H), δ 4.49 (s, 1H), δ 3.58 (d, J = 4.2Hz, 2H), δ 2.99 (t, J = 4.8Hz, 2H), δ 2.34 (m, 2H), δ 2.02-1.81 (m, 5H), δ 1.54-1.34 (m, 10H), δ 1.19-1.08 (m, 11H), δ 1.06-0.86 (m, 14H), δ 0.68 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 156.3, 149.4, 140.0, 136.6, 123.6, 122.5, 121.6, 74.2, 56.9, 56.4, 50.2, 42.5, 40.4, 39.9, 39.7, 38.8, 37.0, 36.7, 36.4, 32.1, 32.0, 28.4, 28.4, 28.2, 24.5, 24.1, 23.0, 22.8, 21.2, 19.5, 18.9, 12.1 ppm; IR ν_{max} (cm^{-1}) 3434, 2938, 2869, 2094, 1708, 1641, 1264; HRMS (ESI) calcd for $C_{35}H_{54}N_2O_2$ (M+) 535.4259, found 535.4257.



(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 3,4-dihydroquinoline-1(2H)-carboxylate (**7f**): White solid. mp=107-110 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, J = 8.1Hz, 1H), δ 7.18 (t, J = 7.5 Hz, 1H), δ 7.09 (d, J = 7.2 Hz, 1H), δ 6.99 (t, J = 7.5 Hz, 1H), δ 5.41 (d, J = 4.21 Hz, 1H), δ 4.65 (m, 1H), δ 3.76 (t, J = 5.7 Hz, 2H), δ 2.77 (t, J = 6.6Hz, 2H), δ 2.45 (m, 2H), δ 2.04-1.89 (m, 7H), δ 1.86-1.55 (m, 8H), δ 1.44-1.15 (m, 10H),

δ 1.12-1.04 (m, 9H), δ 0.94-0.88 (m, 9H), δ 0.69 (s, 3H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.6, 140.0, 138.6, 130.1, 128.8, 126.1, 124.2, 123.6, 122.8, 75.9, 56.9, 56.4, 40.3, 44.9, 42.6, 39.9, 39.8, 38.8, 37.3, 36.9, 36.4, 36.1, 32.2, 32.1, 28.5, 28.4, 28.3, 27.7, 24.5, 24.1, 23.7, 23.1, 22.1, 21.3, 19.5, 18.9, 12.1 ppm; IR ν_{max} (cm^{-1}) 3408, 2962, 2927, 1717, 1620, 1602, 1457; HRMS (ESI) calcd for $C_{37}H_{55}NO_2$ (M+) 568.4125, found 568.4127.

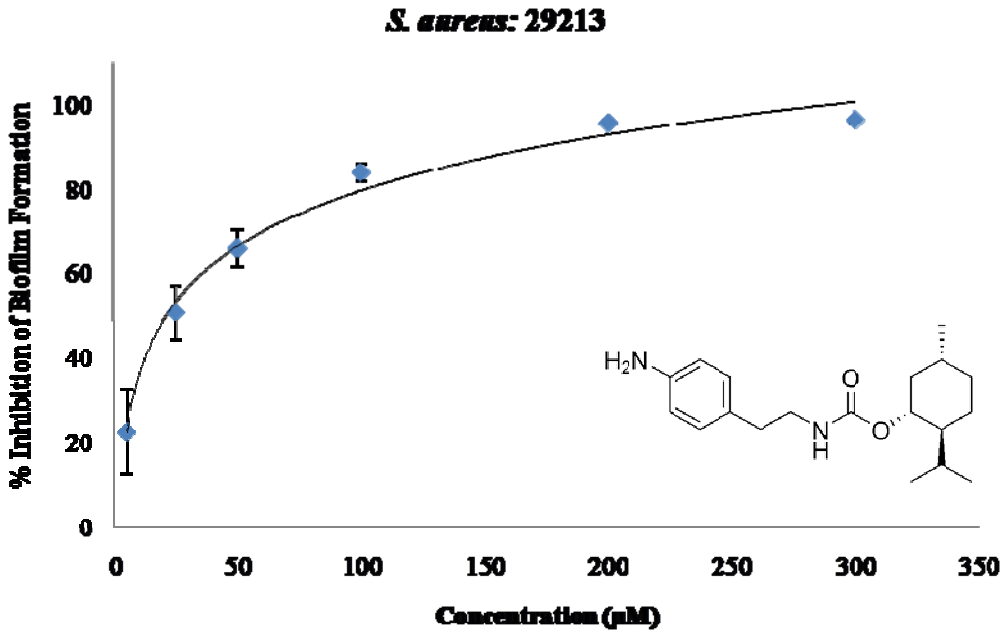


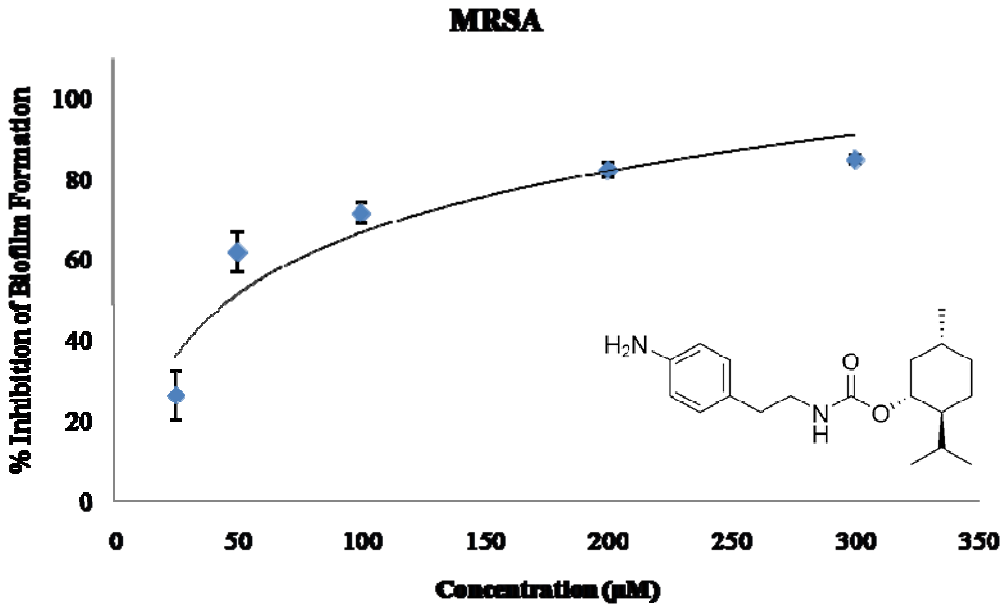
(3S,8S,9S,10R,13R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 2-(1H-indol-3-yl)ethylcarbamate (**10f**): Light yellow solid. mp=149-152 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.57 (s, 1H), δ 7.54 (d, J = 7.2 Hz, 1H), δ 7.26 (d, J = 7.2 Hz, 1H), δ 7.10 (m, 2H), δ 6.79 (s, 1H), δ 5.31 (s, 1H), δ 4.90 (s, 1H), δ 4.52 (s, 1H), δ 3.41 (s, 2H), δ 2.87 (s, 2H, δ 2.27 (s, 2H), δ 1.99-1.77 (m, 5H), δ 1.52-1.34 (m, 11H), δ 1.12-0.88 (m, 22H), δ 0.66 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.8, 140.0, 136.8, 127.6, 122.9, 122.6, 122.2, 119.5, 118.9, 112.8, 111.8, 74.7, 56.9, 56.5, 50.3, 42.6, 41.7, 40.1, 39.9, 38.9, 37.3, 36.8, 36.5, 36.2, 32.2, 28.6, 28.4, 24.7, 23.3, 22.9, 21.4, 19.7, 19.2, 12.2 ppm; IR ν_{max} (cm^{-1}) 3412, 3056, 2945, 2868, 2247, 1697, 1515; HRMS (ESI) calcd for $C_{36}H_{54}N_2O_2$ (M+) 547.4259, found 547.4257.

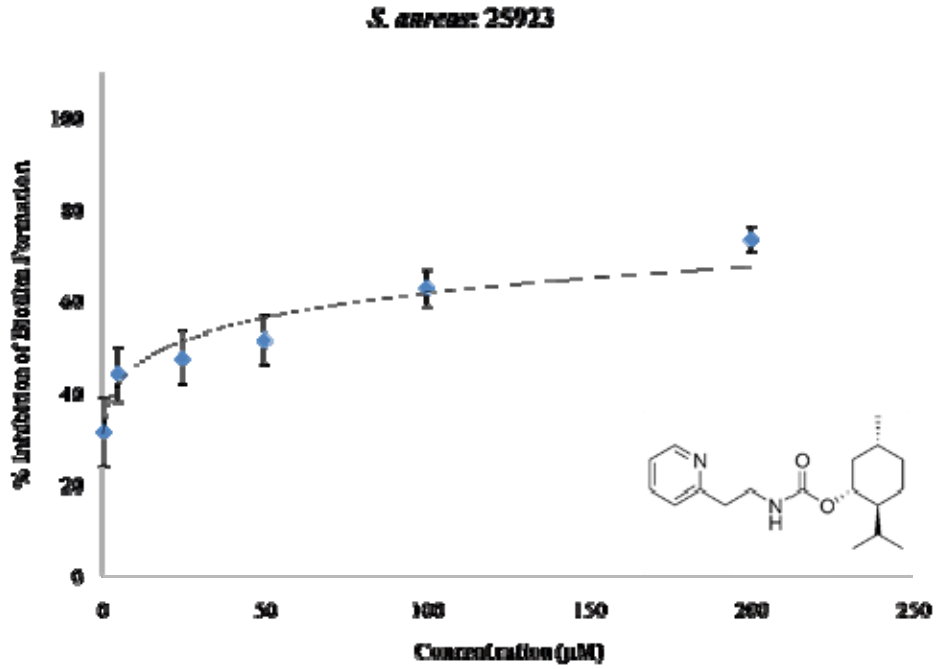
Biology Experimental:

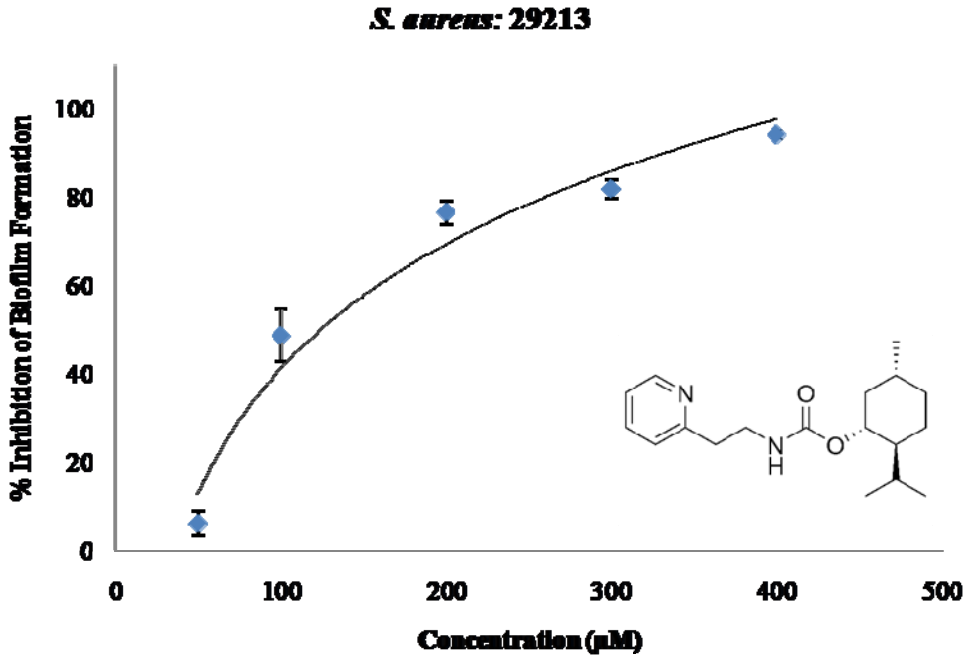
Procedure to Determine the Inhibitory Effect of Test Compounds on MRSA, *S. epidermidis, R. salexigens, E. Coli* and MDRAB Biofilm Formation: Inhibition assays

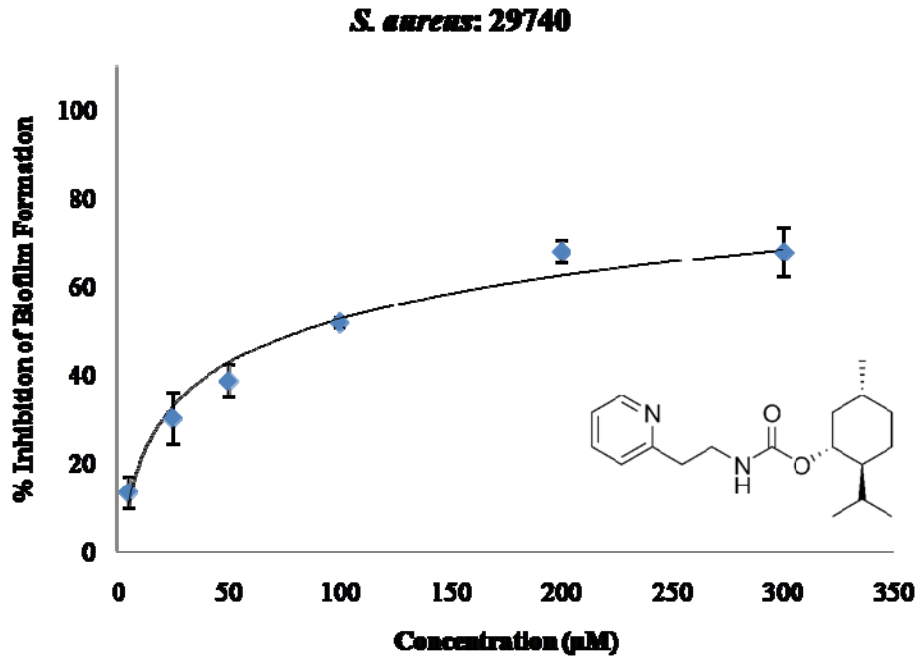
were performed by taking an overnight culture of bacterial strain and subculturing it at an OD_{600} of 0.01 into the necessary medium (brain heart infusion for *E. faecium*, tryptic soy broth with a 0.5% glucose supplement (TSBG) for MRSA, Luria-Bertani (LB) medium for MDRAB, tryptic soy broth with a 0.5% glucose supplement and a 3.0% NaCl supplement (TGN) for *R. salexigens*, Luria-Bertani (LB) medium for *E. coli* and tryptic soy broth with a 0.5% glucose supplement and a 3.0% NaCl supplement (TGN) for *S. epidermidis*. Stock solutions of predetermined concentrations of the test compound were then made in the necessary medium. These stock solutions were aliquoted (100 μL) into the wells of the 96-well PVC microtiter plate. Sample plates were then wrapped in GLAD Press n' Seal® followed by an incubation under stationary conditions for 24 h at 37 °C. After incubation, the medium was discarded from the wells and the plates were washed thoroughly with water. Plates were then stained with 100 μL of 0.1% solution of crystal violet (CV) and then incubated at ambient temperature for 30 min. Plates were washed with water again and the remaining stain was solubilized with 200 μL of 95% ethanol. A sample of 125 μL of solubilized CV stain from each well was transferred to the corresponding wells of a polystyrene microtiter dish. Biofilm inhibition was quantitated by measuring the OD_{540} of each well in which a negative control lane wherein no biofilm was formed served as a background and was subtracted out.

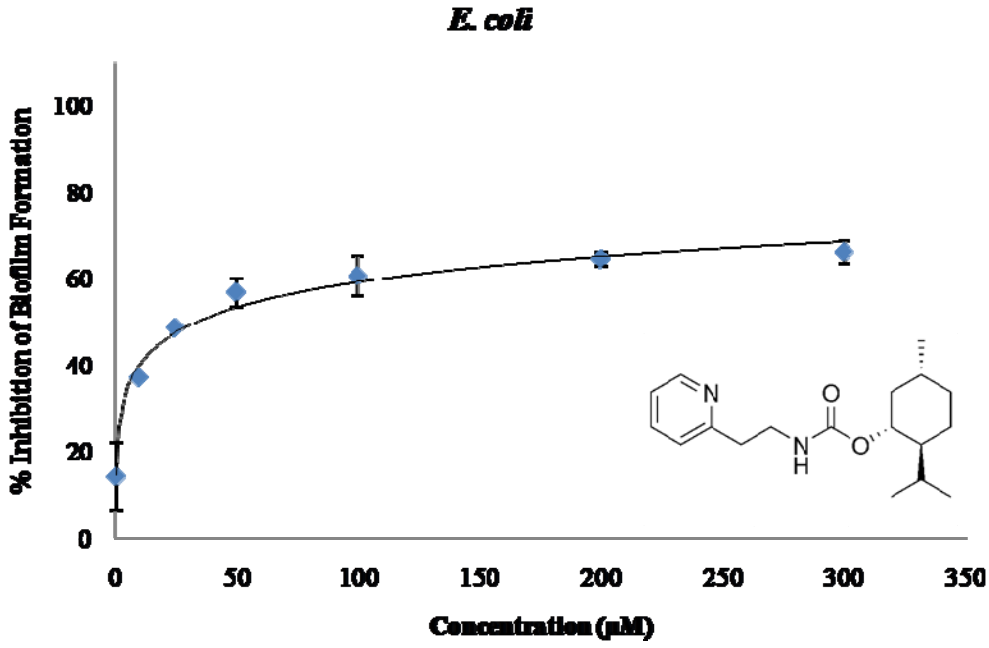

S. aureus: 29213
% Inhibition of Biofilm Formation
0
20
40
60
80
100
0
50
100
150
200
250
300
350
Concentration (μM)
H2N
O
N
H
O

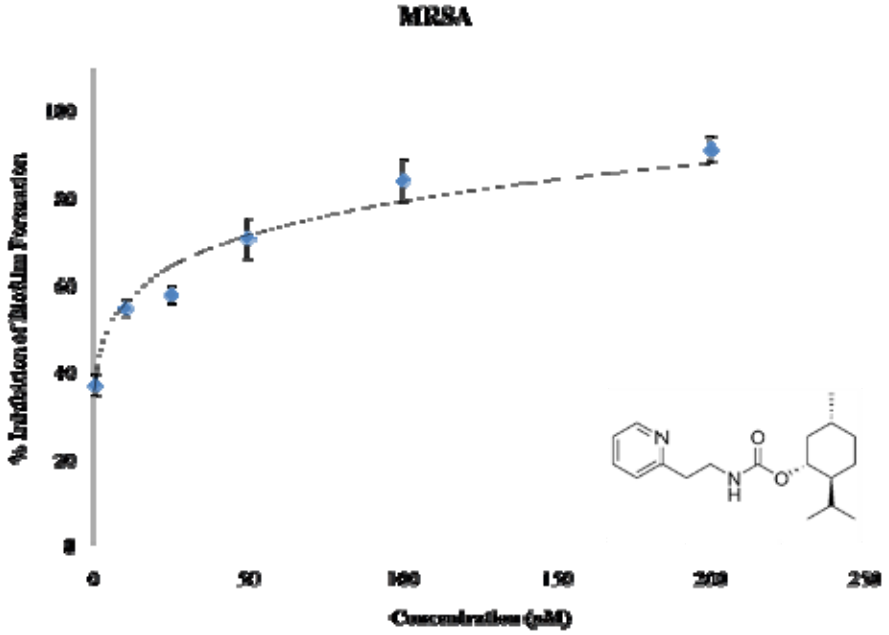

MRSA
% Inhibition of Biofilm Formation
0
20
40
60
80
100
0
50
100
150
200
250
300
350
Concentration (μM)
H2N
O
N
H
O

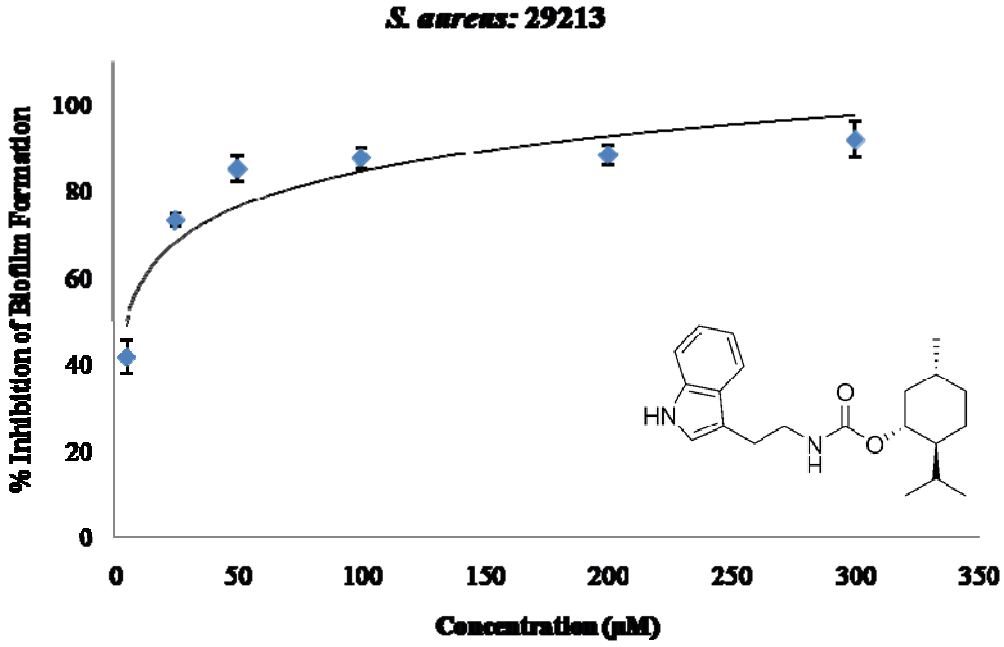

S. aureus: 25923
% Inhibition of Biofilm Formation
0
20
40
60
80
100
0
50
100
150
200
250
Concentration (μM)
N
O
N
H
O

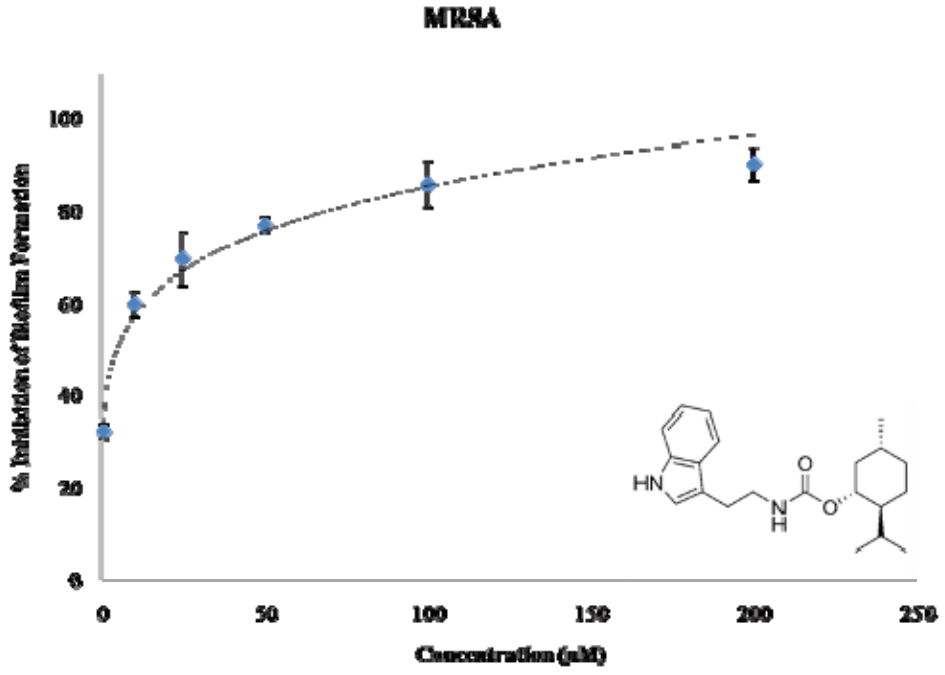

S. aureus: 29213
% Inhibition of Biofilm Formation
0
20
40
60
80
100
0
100
200
300
400
500
Concentration (μM)
N
O
N
H
O

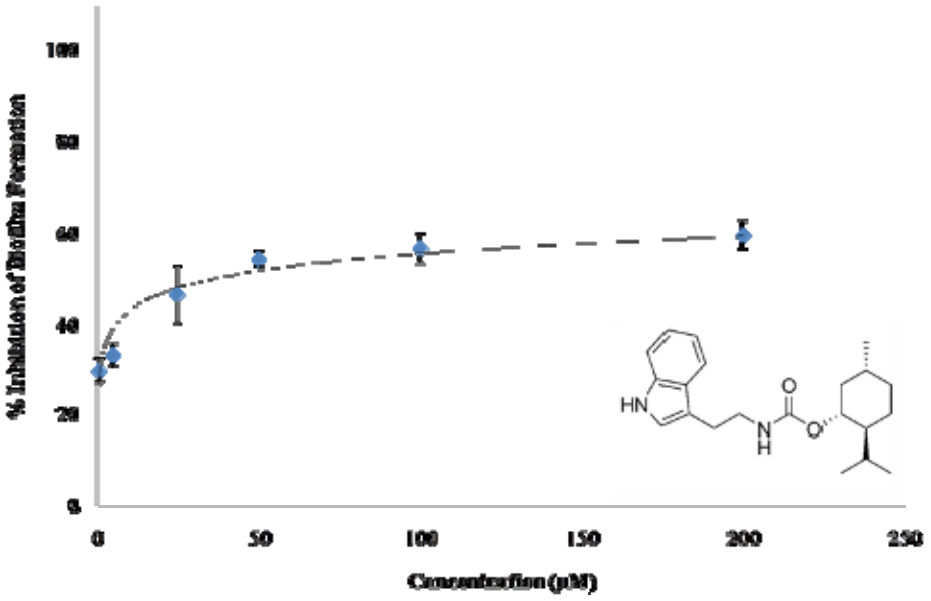

S. aureus: 29740
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
N
O
N
H
O

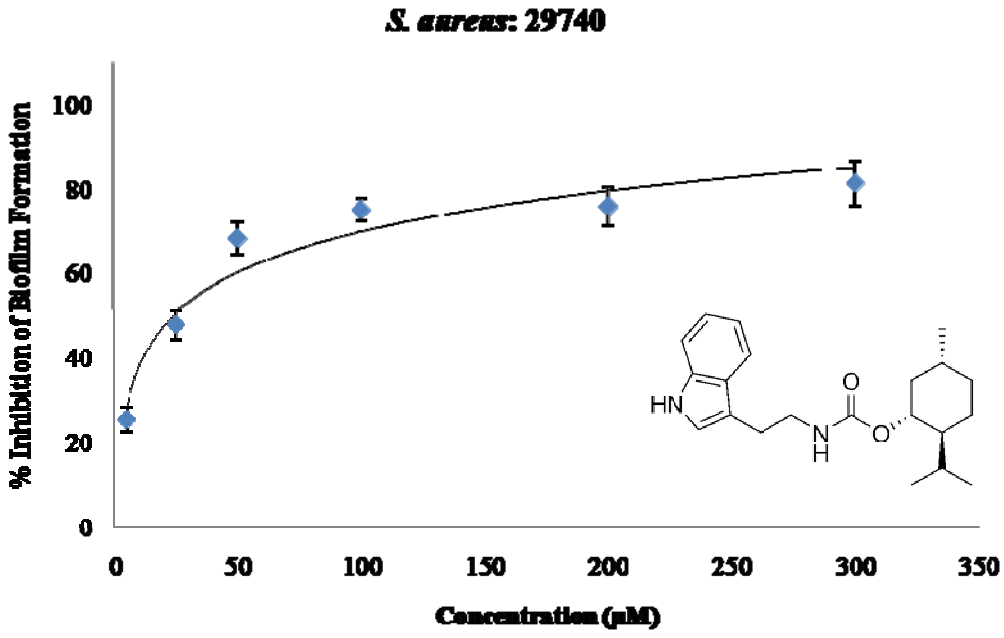

E. coli
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
N
O
N
H
O

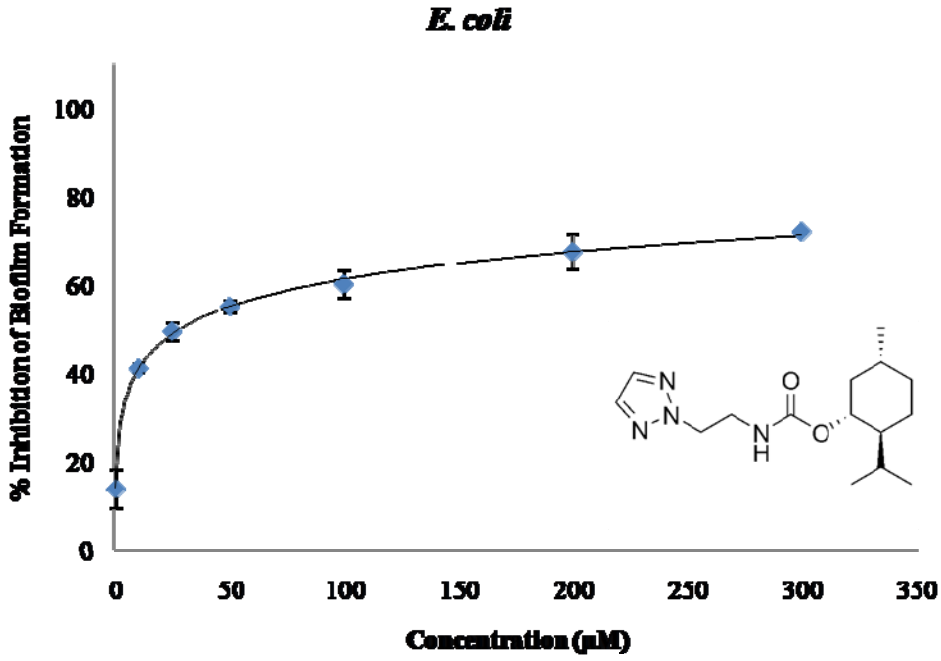

MRSA
% Inhibition of Biofilm Formation
Concentration (μM)
N
O
N
H
O

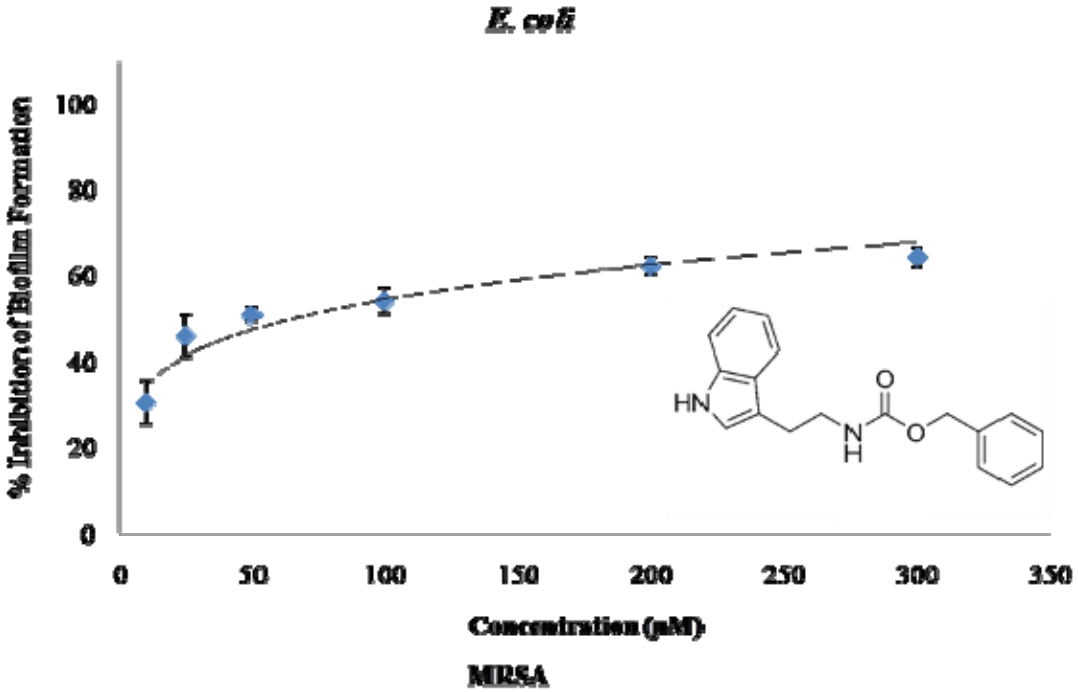

S. aureus: 29213
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
HN
N
H
O
O

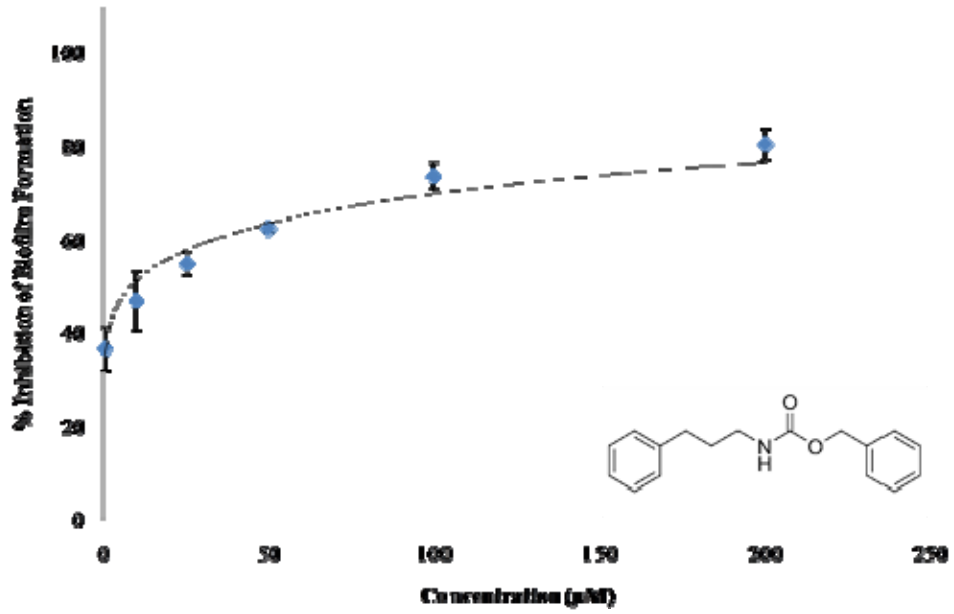

MRSA
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
Concentration (μM)
HN
O
N
H
O

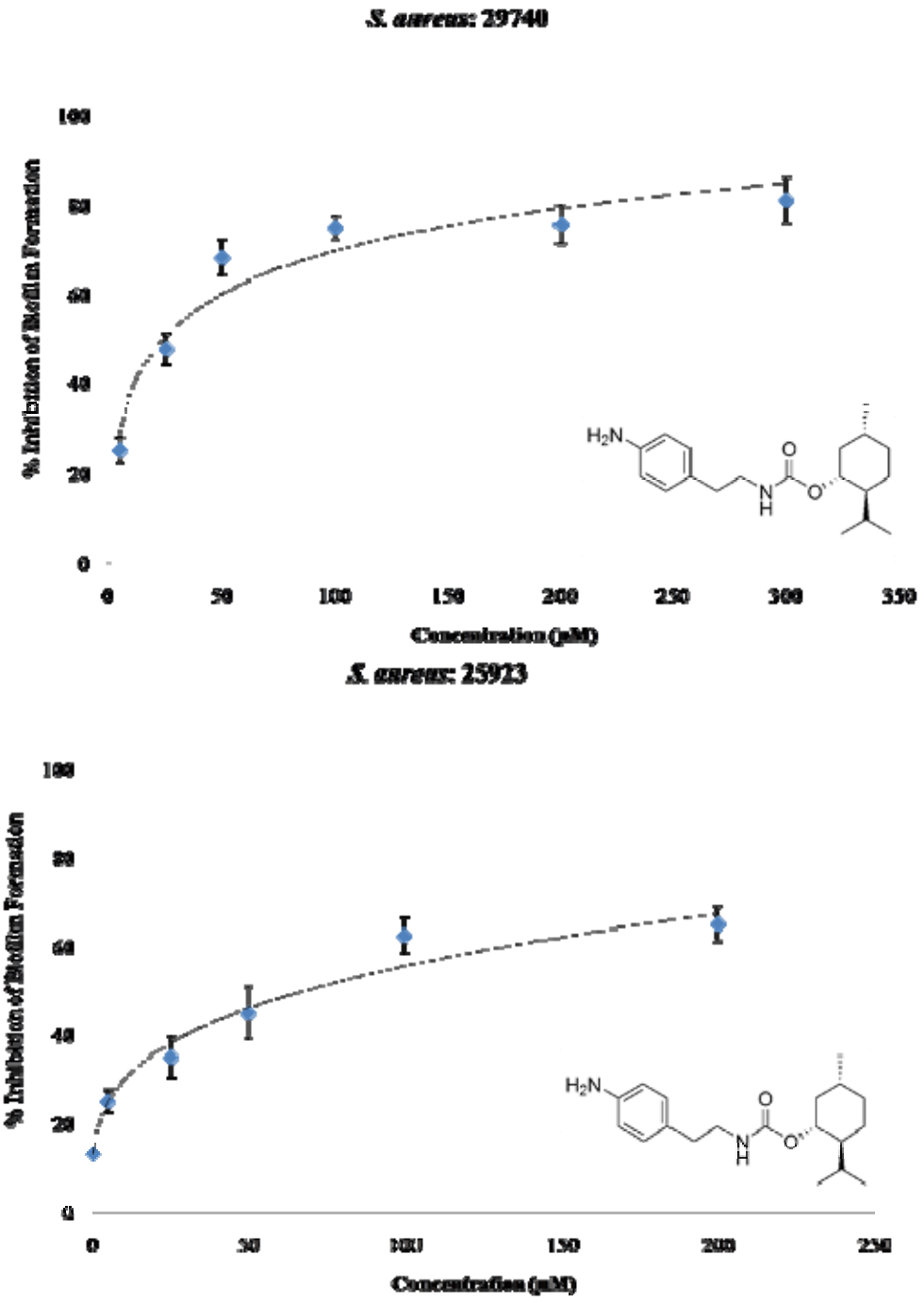

S. aureus: 25923
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
Concentration (μM)
HN
O
N
H
O


S. aureus: 29740
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
HN
O
N
H
O


E. coli
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
N
N
N
O
N
H
O


E. coli
% Inhibition of Biofilm Formation
100
80
60
40
20
0
0
50
100
150
200
250
300
350
Concentration (μM)
HN
N
H
O
O


MRSA
% Inhibition of Biofilm Formation
0
50
100
150
200
250
Concentration (μM)
N
H
O
O

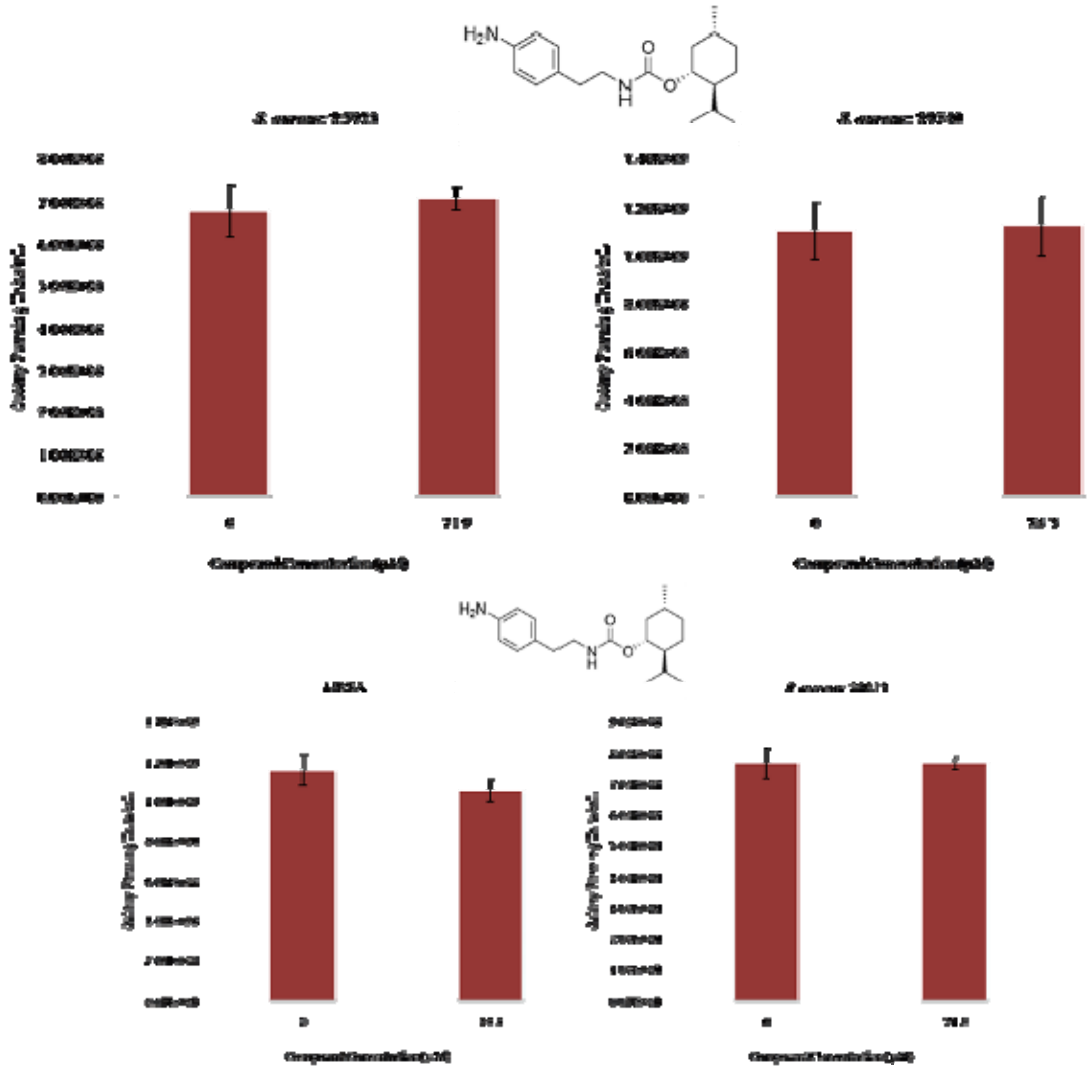


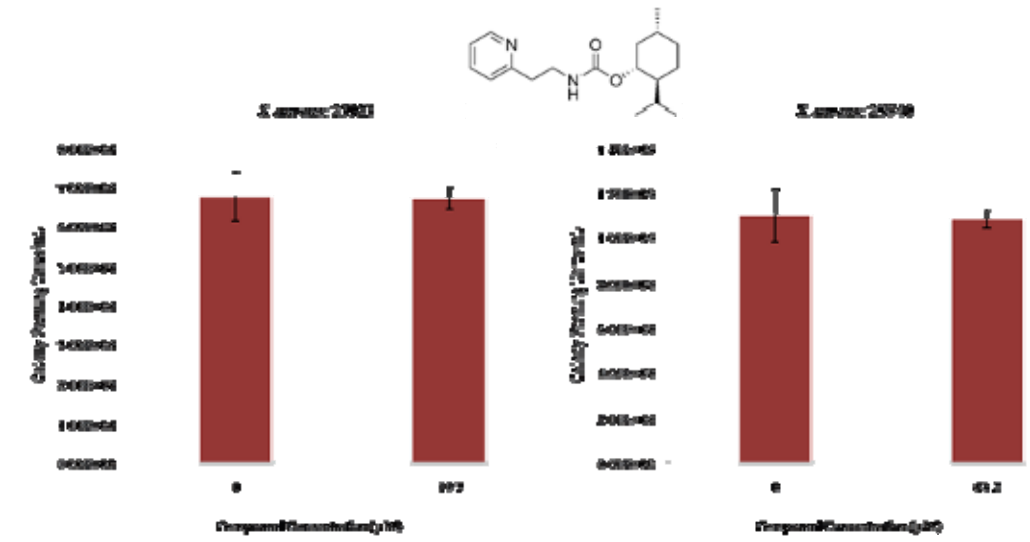
Procedure to determine the ability of test compounds to disperse MRSA and *E. coli* Preformed Biofilms: Dispersion assays were performed by taking an overnight culture of bacterial strain and subculturing it at an OD_{600} of 0.01 into the necessary medium (tryptic soy broth with a 0.5% glucose supplement (TSBG) for MRSA and Luria-Bertani (LB) medium

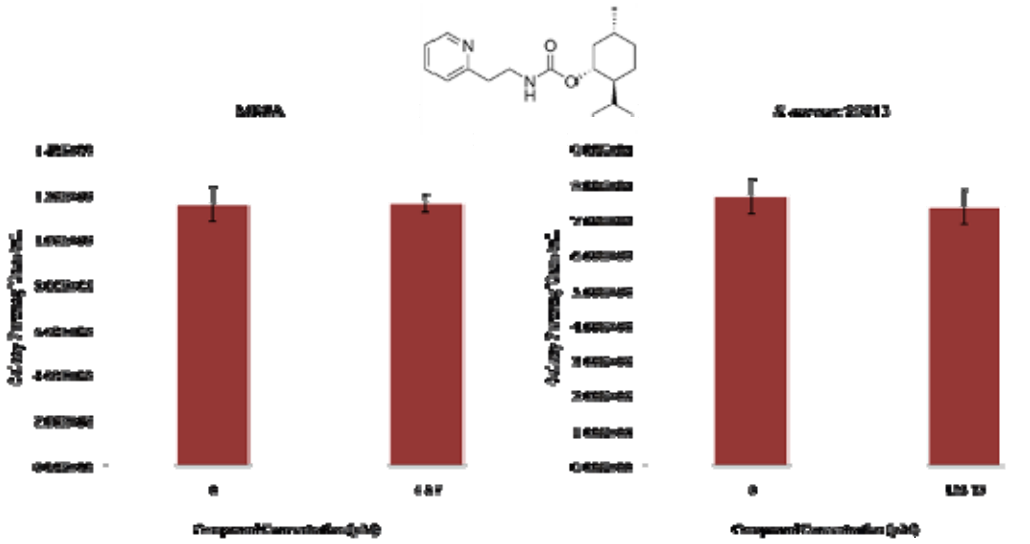
for *E. coli*. The resulting bacterial suspension was aliquoted (100 μL) into the wells of a 96-well PVC microtiter plate. Plates were then wrapped in GLAD Press n' Seal® followed by an incubation under stationary conditions at ambient temperature to establish the biofilms. After 24 h, the medium was discarded from the wells and the plates were washed thoroughly with water. Stock solutions of predetermined concentrations of the test compound were then made in the necessary medium. These stock solutions were aliquoted (100 μL) into the wells of the 96-well PVC microtiter plate with the established biofilms. Medium alone was added to a subset of the wells to serve as a control. Sample Plates were then incubated for 24 h at 37 °C. After incubation, the medium was discarded from the wells and the plates were washed thoroughly with water. Plates were then stained with 100 μL of 0.1% solution of crystal violet (CV) and then incubated at ambient temperature for 30 min. Plates were washed with water again and the remaining stain was solubilized with 200 μL of 95% ethanol. A sample of 125 μL of solubilized CV stain from each well was transferred to the corresponding wells of a polystyrene microtiter dish. Biofilm dispersion was quantitated by measuring the OD_{540} of each well in which a negative control lane wherein no biofilm was formed served as a background and was subtracted out.

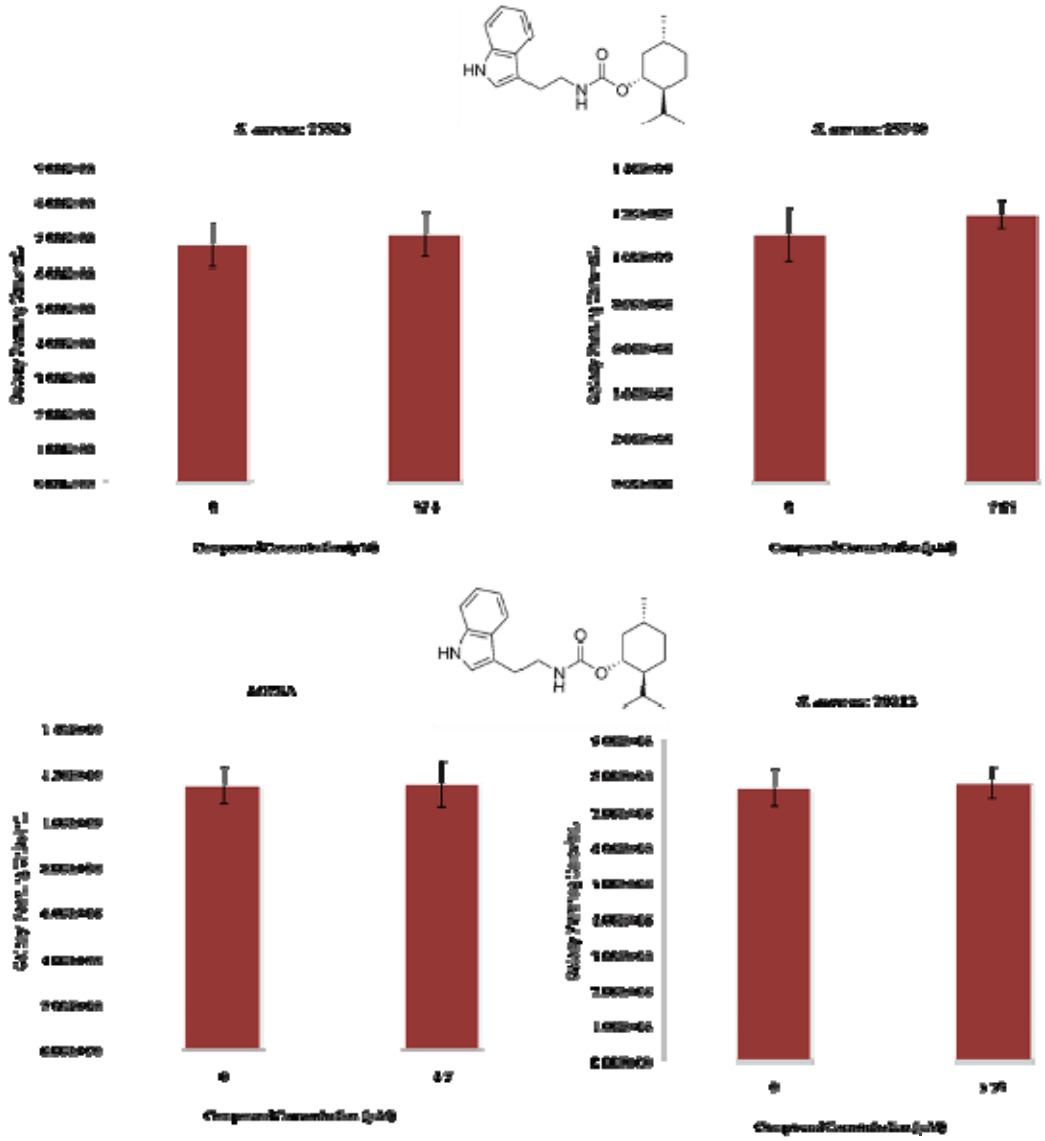
Colony Count Procedure to Determine the Effect of Leading Test Compounds on MRSA and *E. coli* planktonic viability: Colony counts were performed by taking an overnight culture of bacterial strain and subculturing it at an OD_{600} of 0.01 into the necessary medium (tryptic soy broth with a 0.5% glucose supplement (TSBG) for MRSA, Luria-Bertani (LB) medium for *E. coli*). The resulting bacterial suspension was then aliquoted (3.0 mL) into culture tubes. A test compound was then added to the medium of the test samples at a predetermined concentration to

the medium of the test samples. Controls were employed in which no test compound was added to the bacterial suspension. Samples were then placed in an incubator at 37 °C and shaken at 200 rpm until the OD_{600} of the control samples reached approximately 1.2. At this point, 100 µL was taken from each culture tube and then diluted serially into LB medium. Then, 10 µL was removed from each serial dilution and plated out on a square gridded Petri dish followed by 16 h of incubation at 37 °C to grow viable colonies, which were quantified through employment of the track-dilution method.



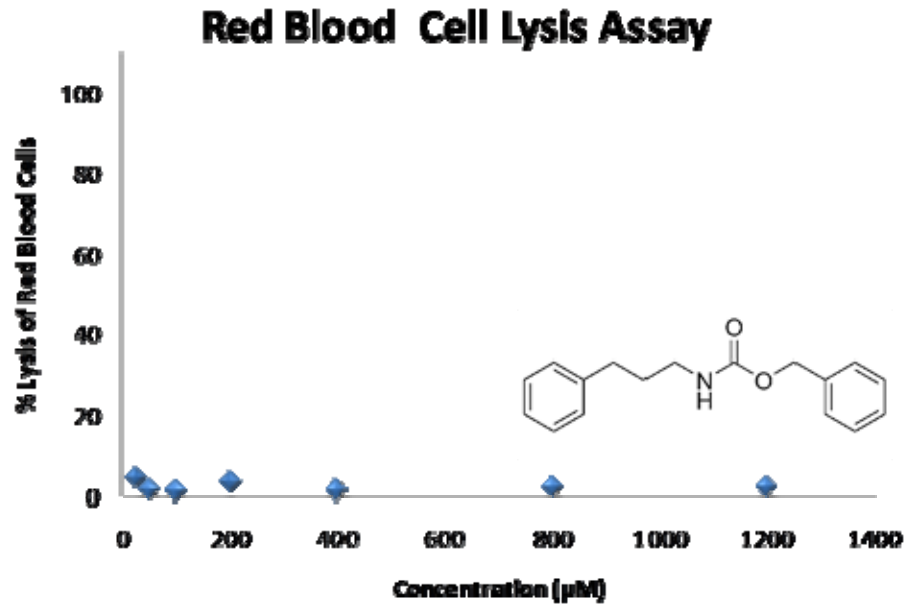


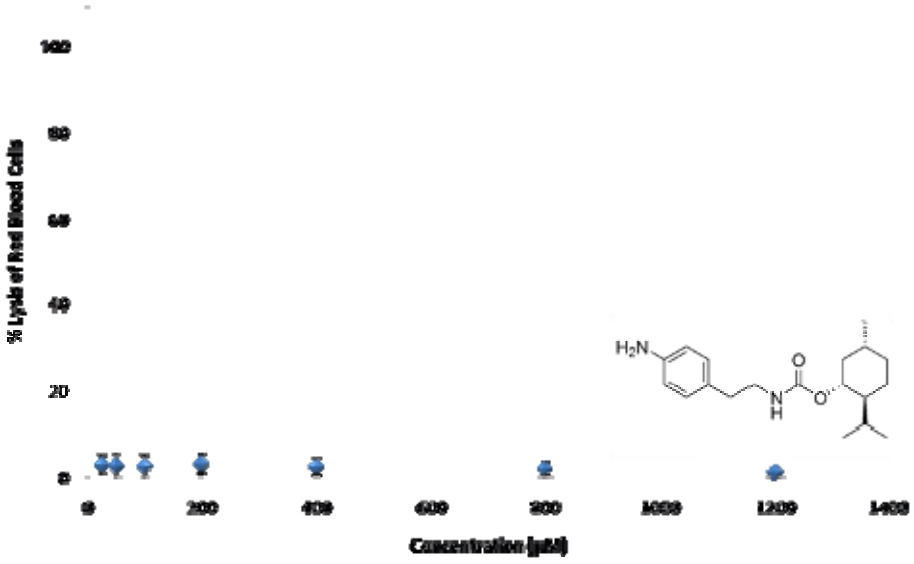


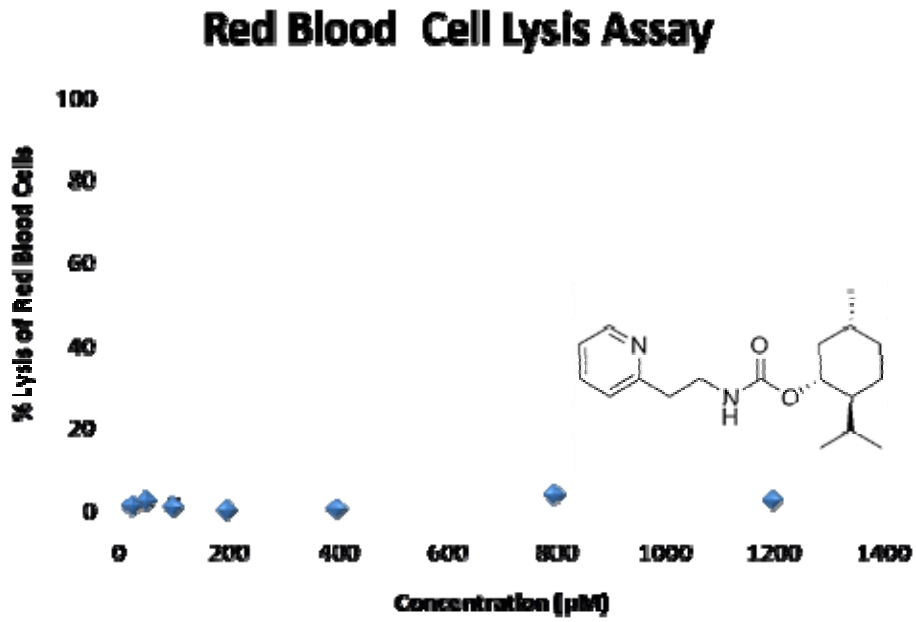


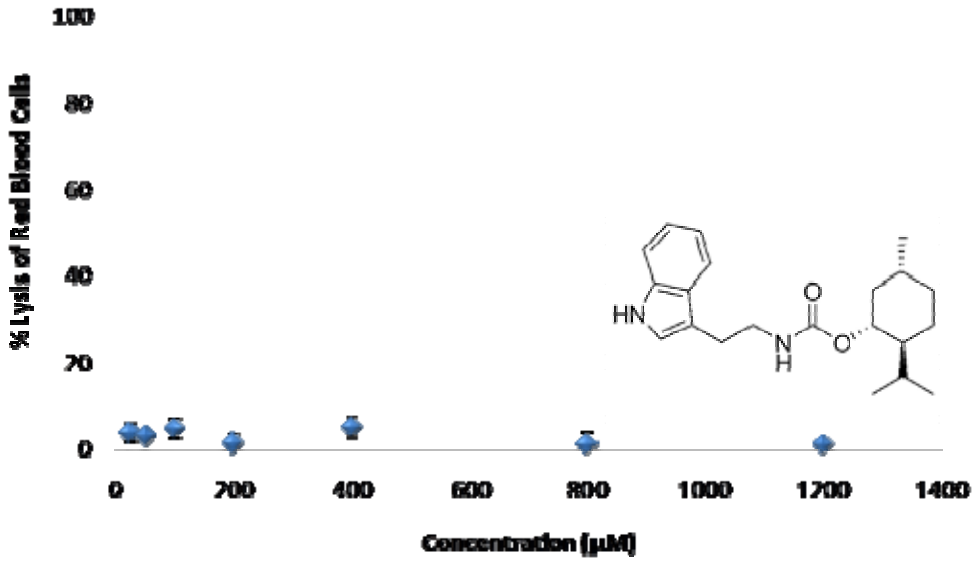
Red Blood Cell Hemolysis Assay. Hemolysis assays were performed on mechanically difibrinated sheep blood (Hemostat Labs: DSB100). 1.5 mL of blood was placed into a microcentrifuge tube and centrifuged at 10000 rpm for ten minutes. The supernatant was removed and then the cells were resuspended with 1 mL of phosphate-buffered saline (PBS). The suspension was centrifuged, the supernatant was removed and cells resuspended two more times. The final cell suspension was then diluted tenfold. Test compound solutions were made in PBS in small culture tubes and then added to aliquots of the tenfold suspension dilution. PBS

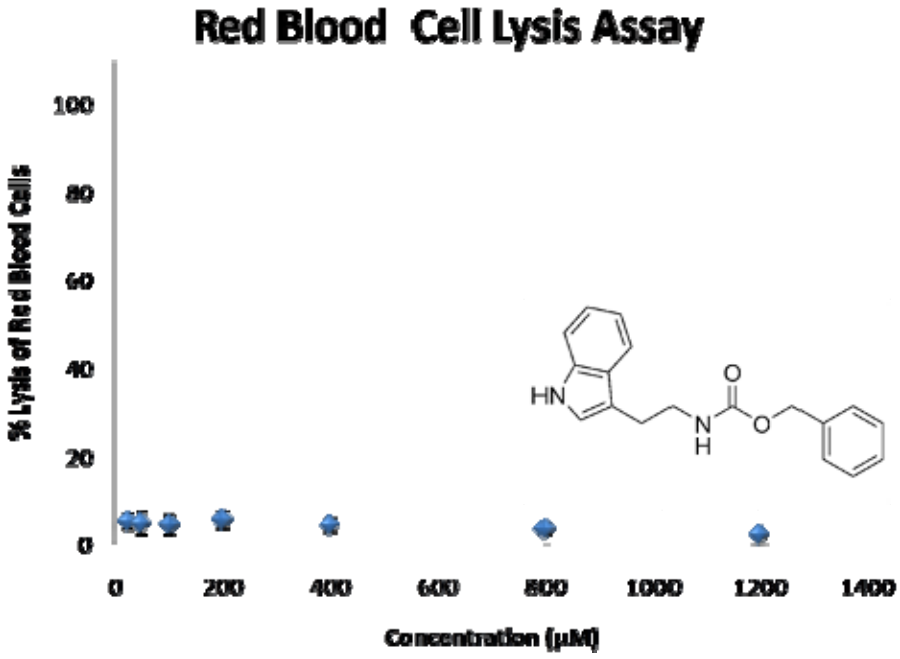
alone was used as a negative control and as a zero hemolysis marker whereas a 1% Triton X sample was used as a positive control and the 100 % lysis marker. Samples were then placed in an incubator at 37 °C while being shaken at 200 rpm for one hour. After one hour, the samples were transferred to microcentrifuge tubes and then centrifuged at 10000 rpm for ten minutes. The resulting supernatant was diluted by a factor of 40 in distilled water. The absorbance of the supernatant was measured with a UV spectrometer at a 540 nm wavelength.



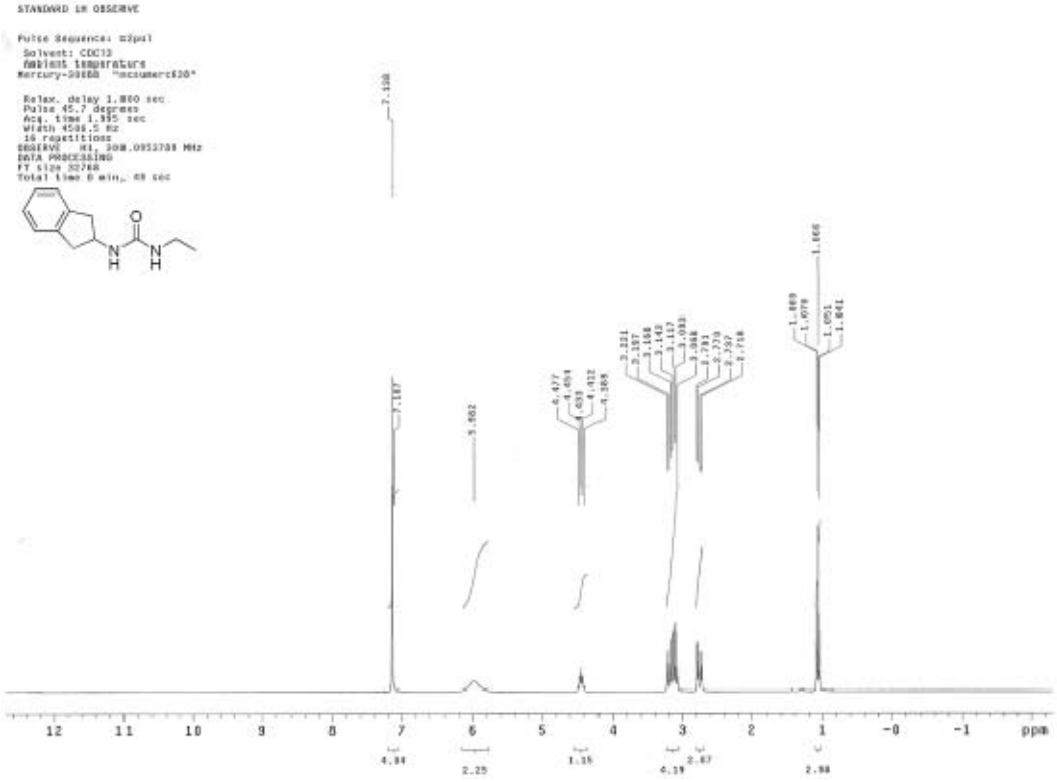


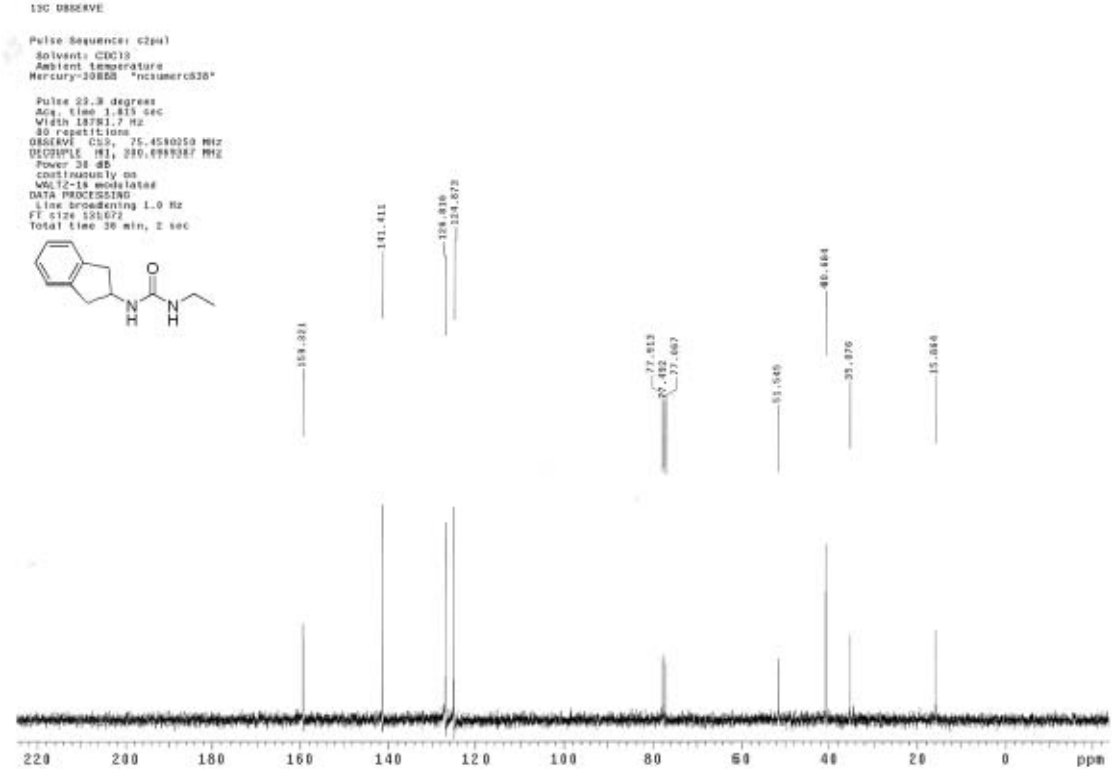

Red Blood Cell Lysis Assay
% Lysis of Red Blood Cells
100
80
60
40
20
0
0
200
400
600
800
1000
1200
1400
Concentration (μM)
N
O
N
H
O

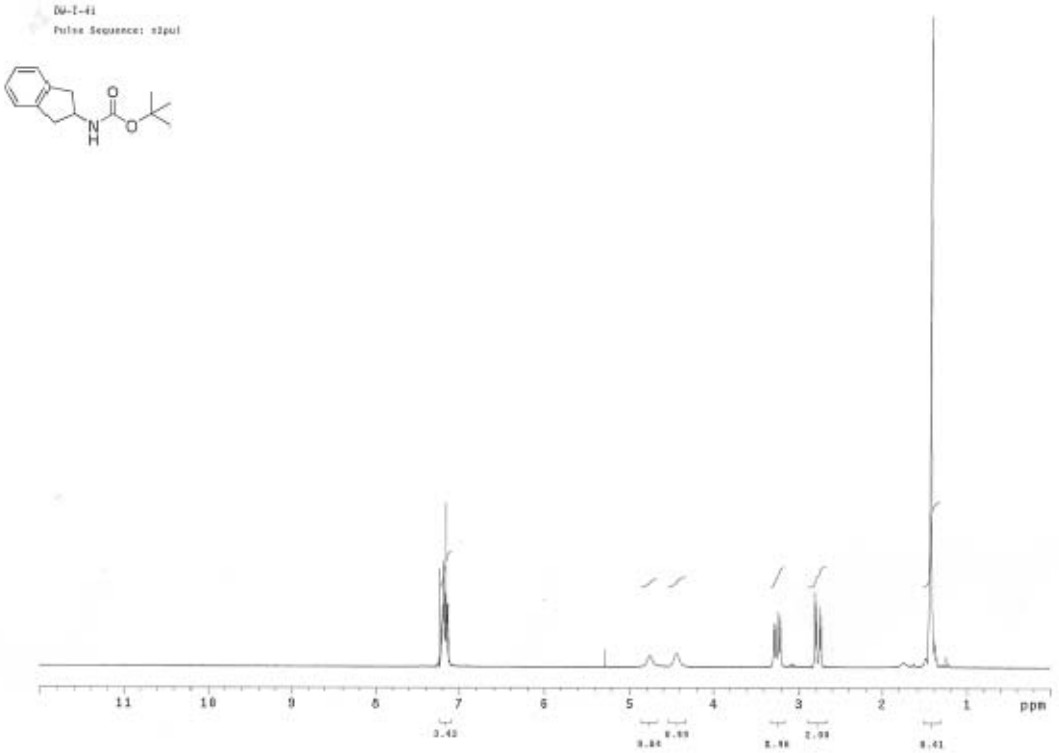

Red Blood Cell Lysis Assay
% Lysis of Red Blood Cells
100
80
60
40
20
0
0
200
400
600
800
1000
1200
1400
Concentration (μM)
HN
O
N
H
O

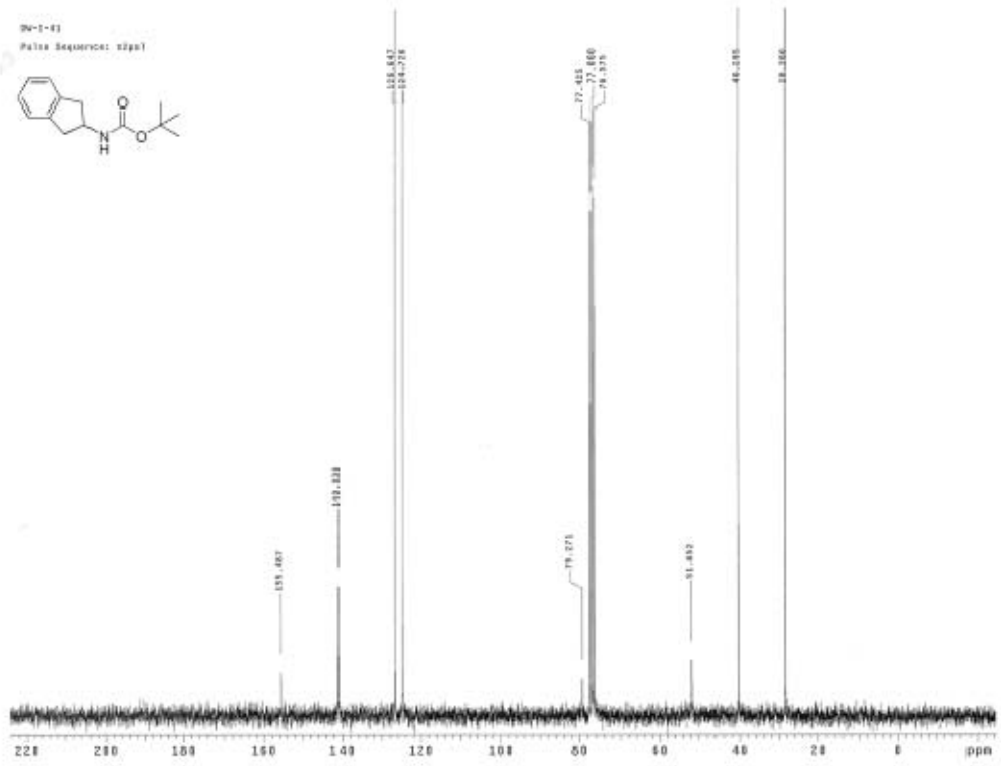

Red Blood Cell Lysis Assay
% Lysis of Red Blood Cells
100
80
60
40
20
0
0
200
400
600
800
1000
1200
1400
Concentration (µM)
HN
N
H
O
O

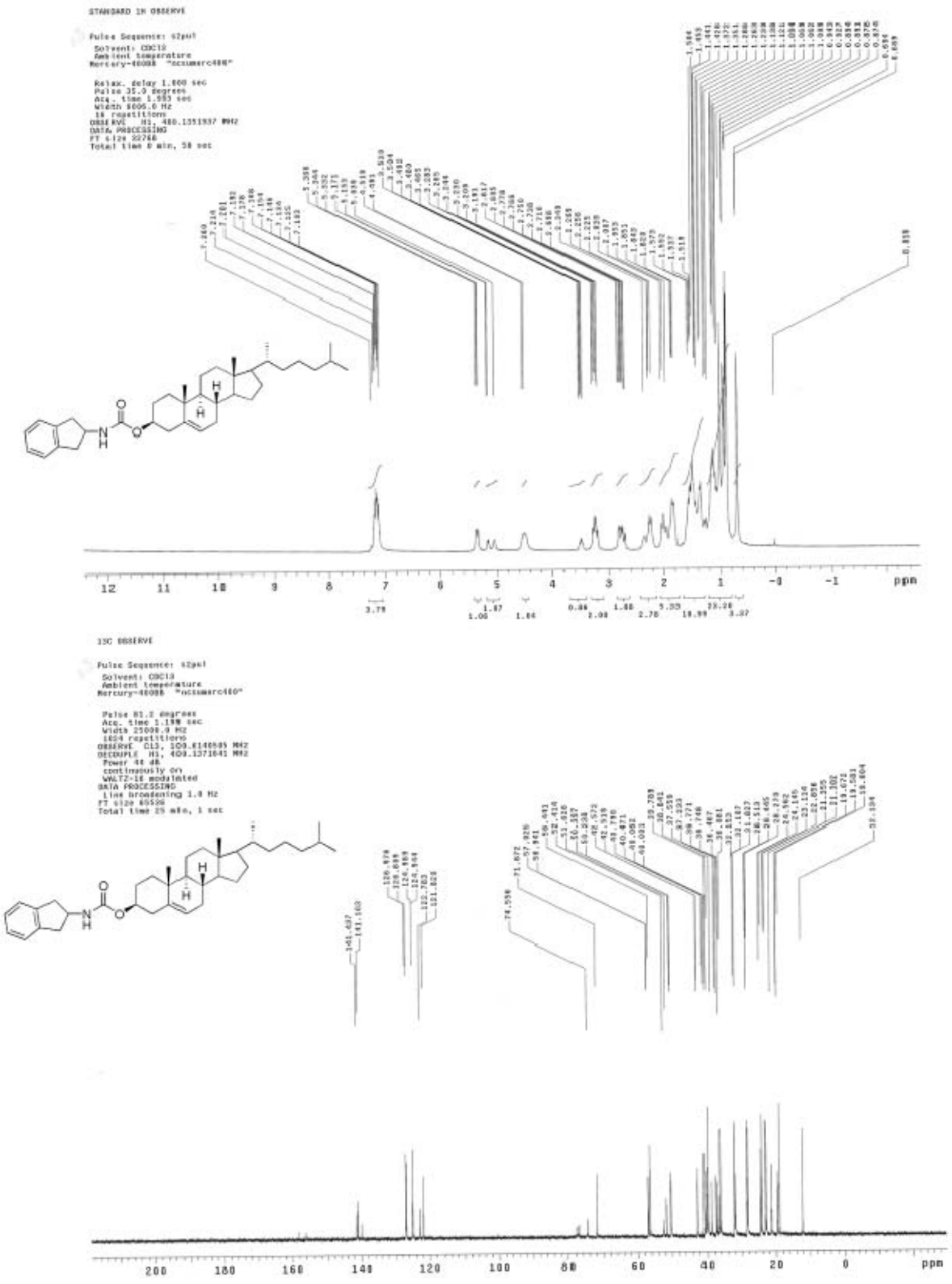
NMR Spectra

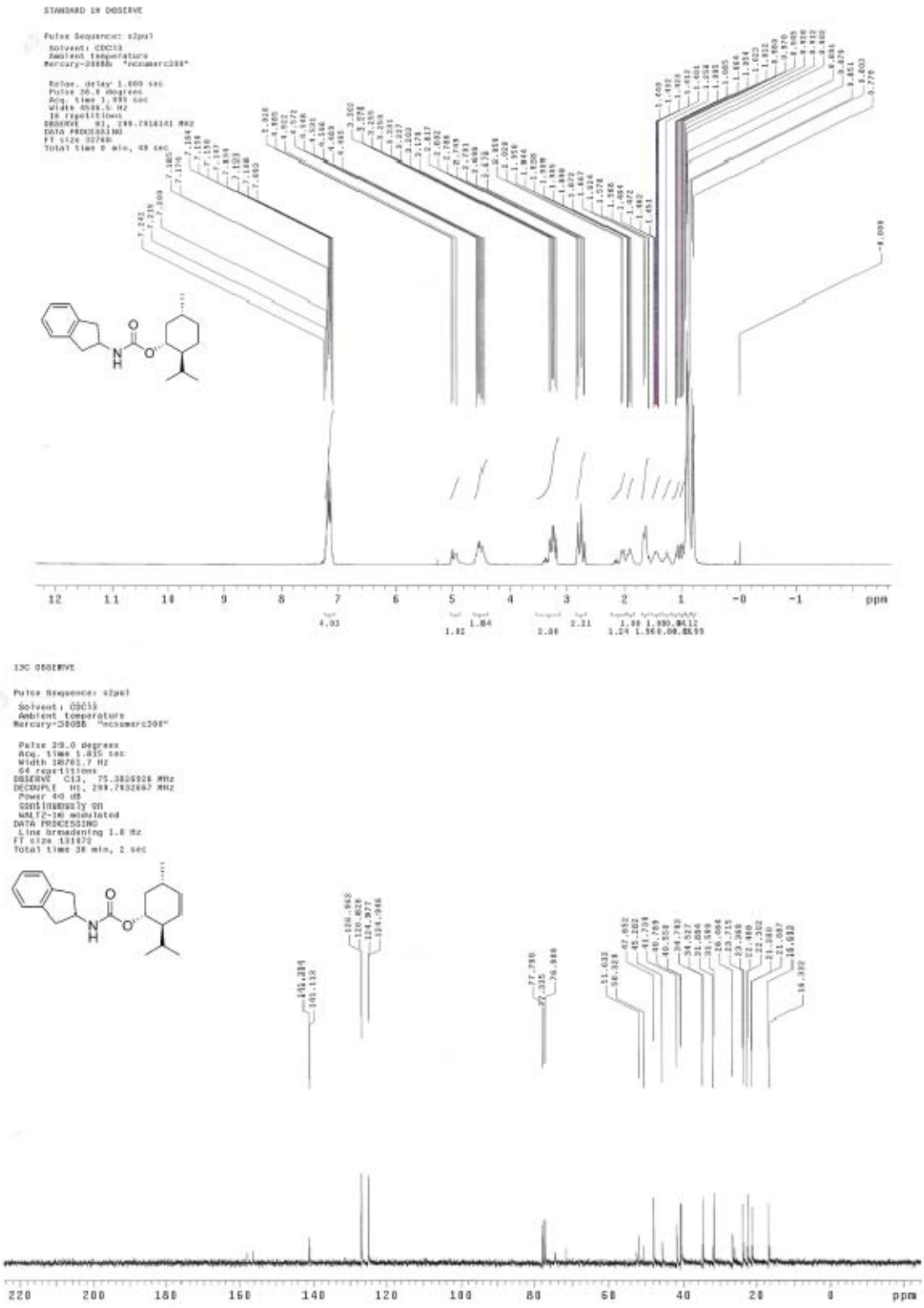

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4508.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
7.188
5.882
4.427
4.404
4.033
4.012
3.385
3.221
3.197
3.168
3.143
3.117
3.093
3.069
2.781
2.773
2.757
2.718
1.668
1.669
1.079
1.051
1.041
12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
4.84
2.25
1.15
4.19
2.67
2.98

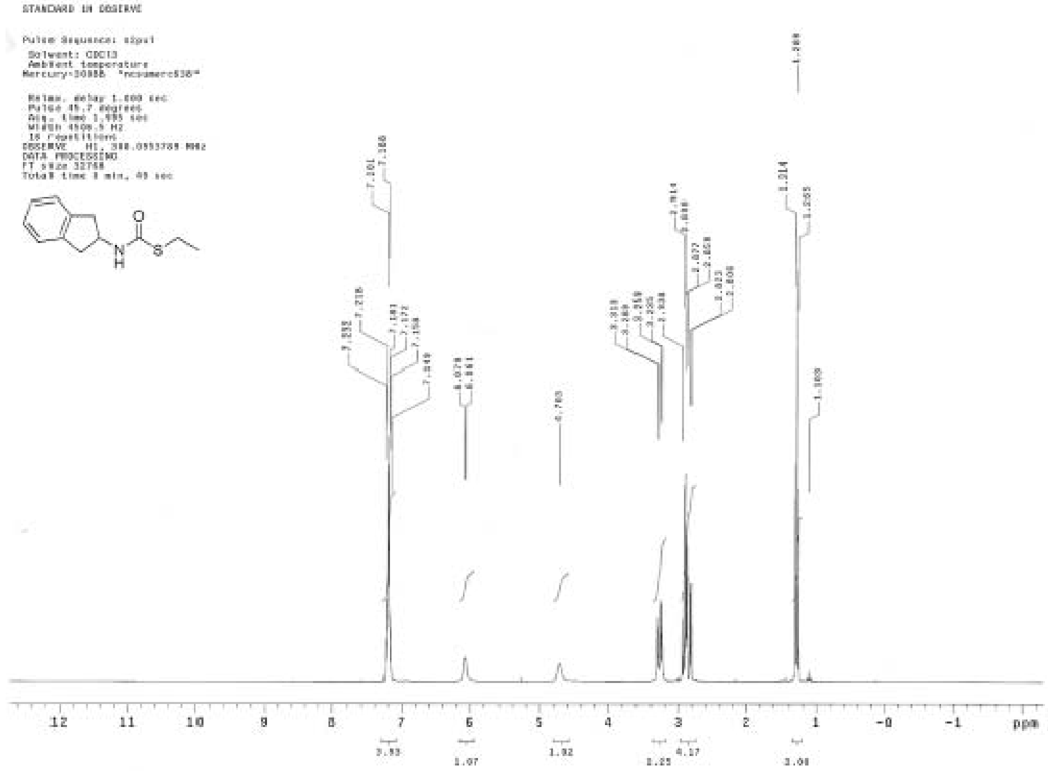

13C OBSERVE
Pulse Sequence: c2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18781.7 Hz
80 repetitions
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 30 min, 2 sec
158.331
141.411
126.816
124.672
77.613
77.492
77.067
51.545
40.464
35.876
15.864
220 200 180 160 140 120 100 80 60 40 20 0 ppm

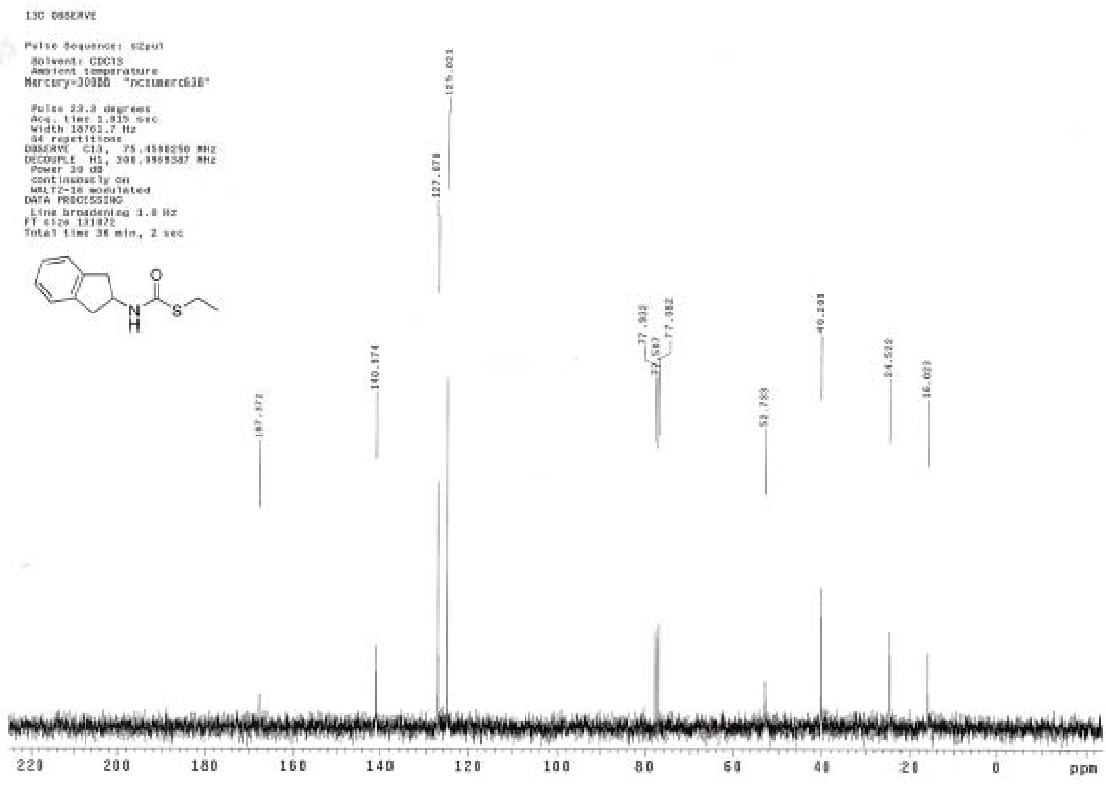

DW-I-41
Pulse Sequence: s2pul
11
10
9
8
7
6
5
4
3
2
1
ppm

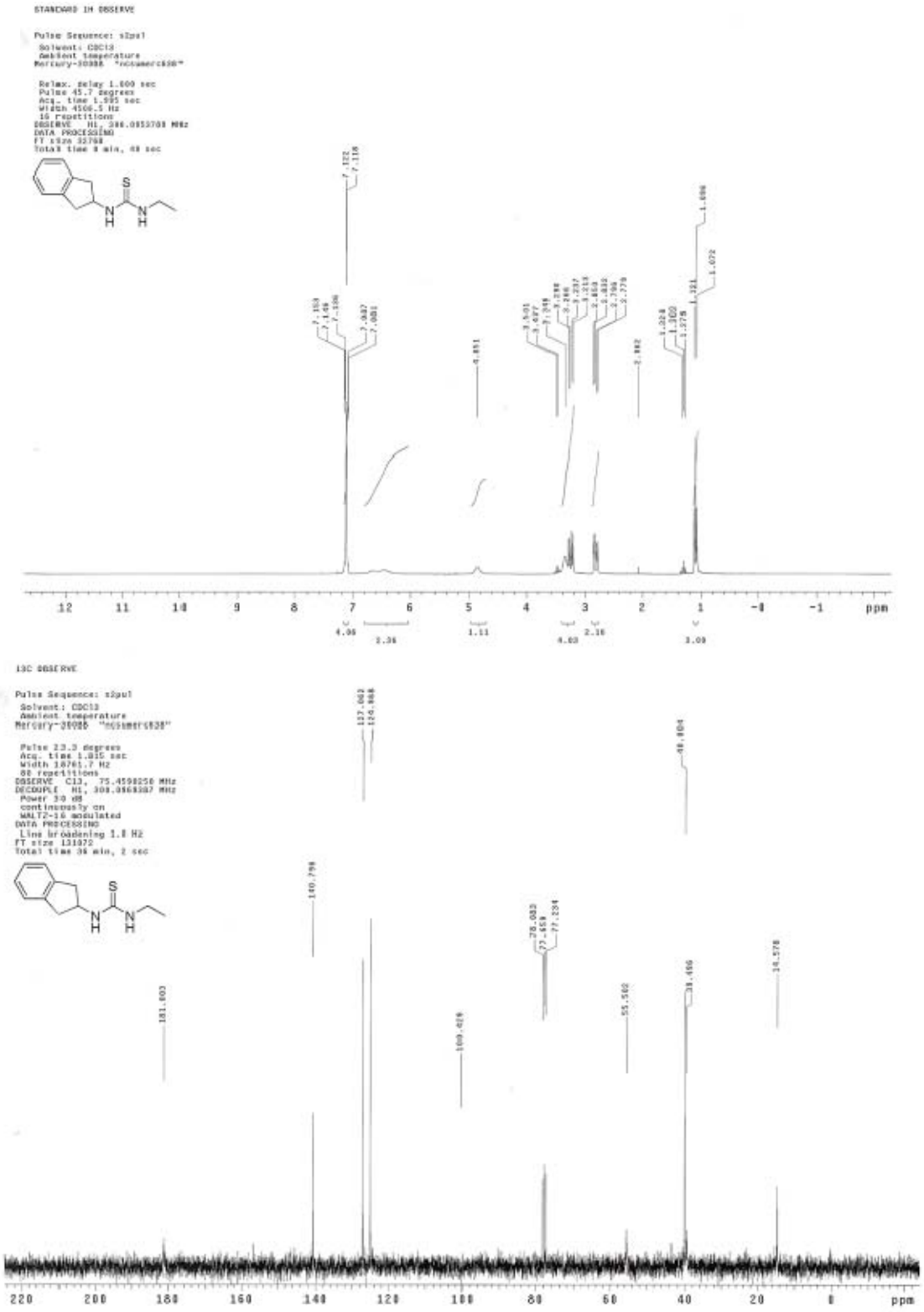

DW-I-41
Pulse Sequence: s2pul
155.467
140.838
79.271
51.462
220
200
180
160
140
120
100
80
60
40
20
0
ppm

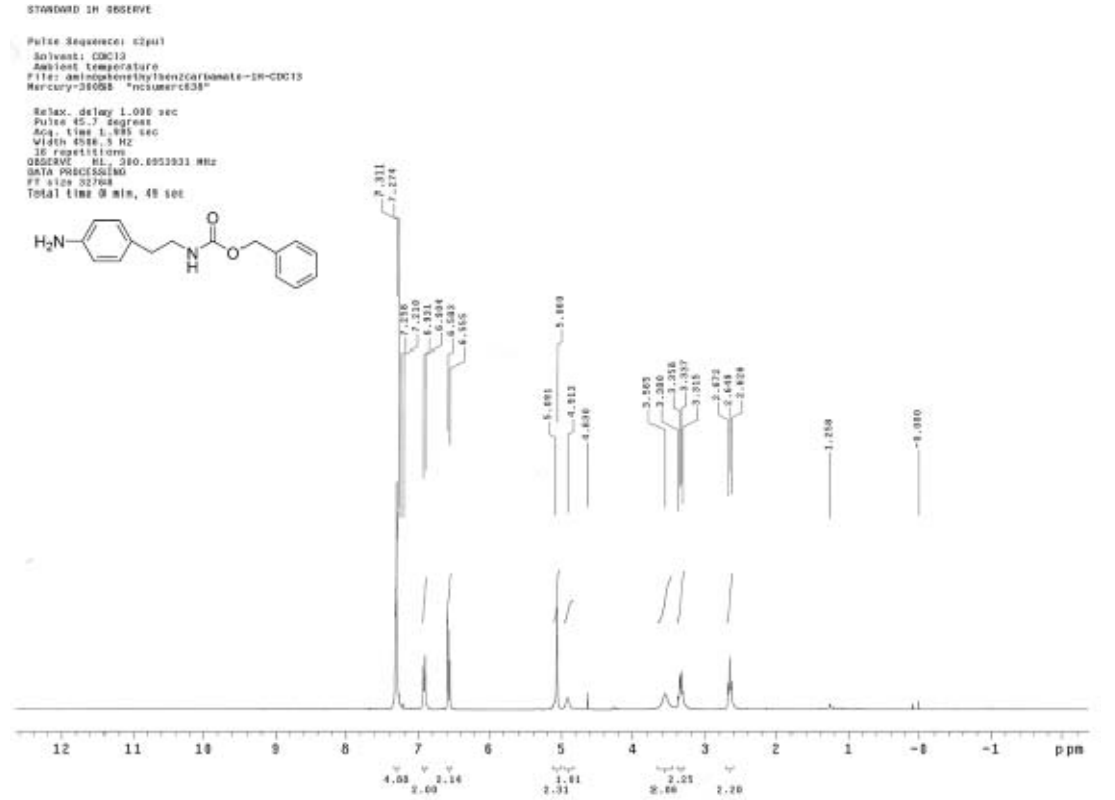


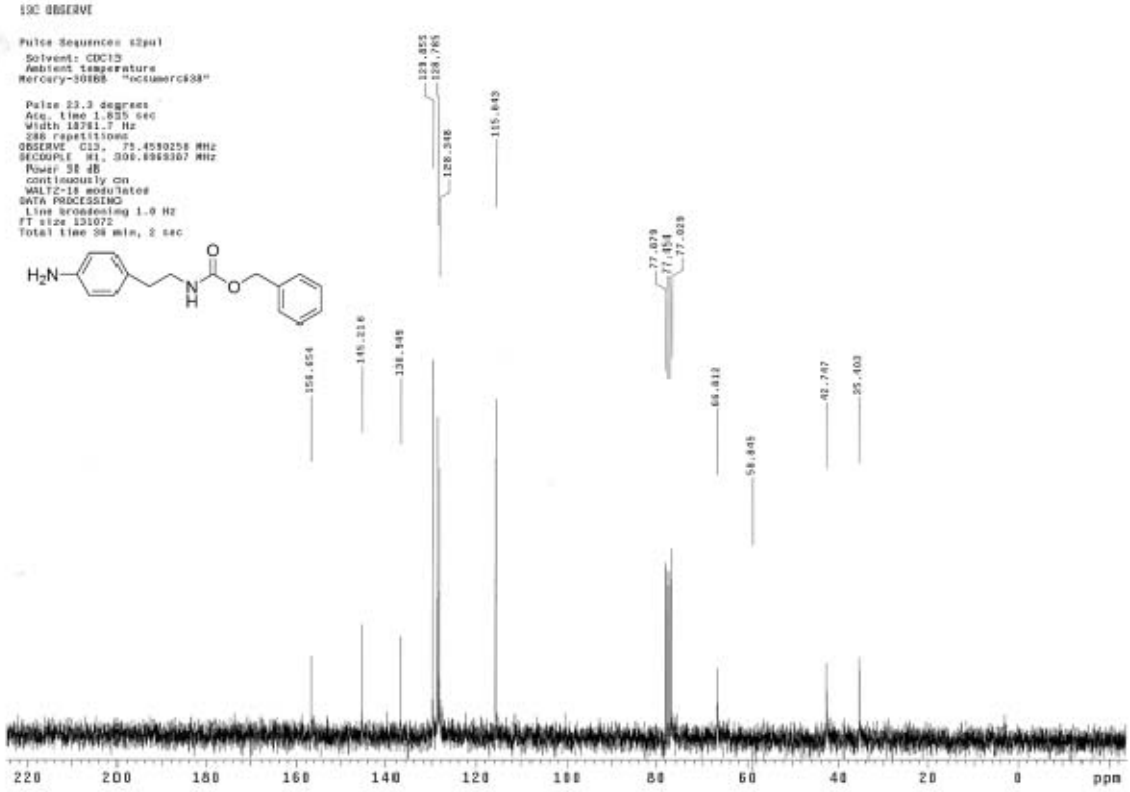


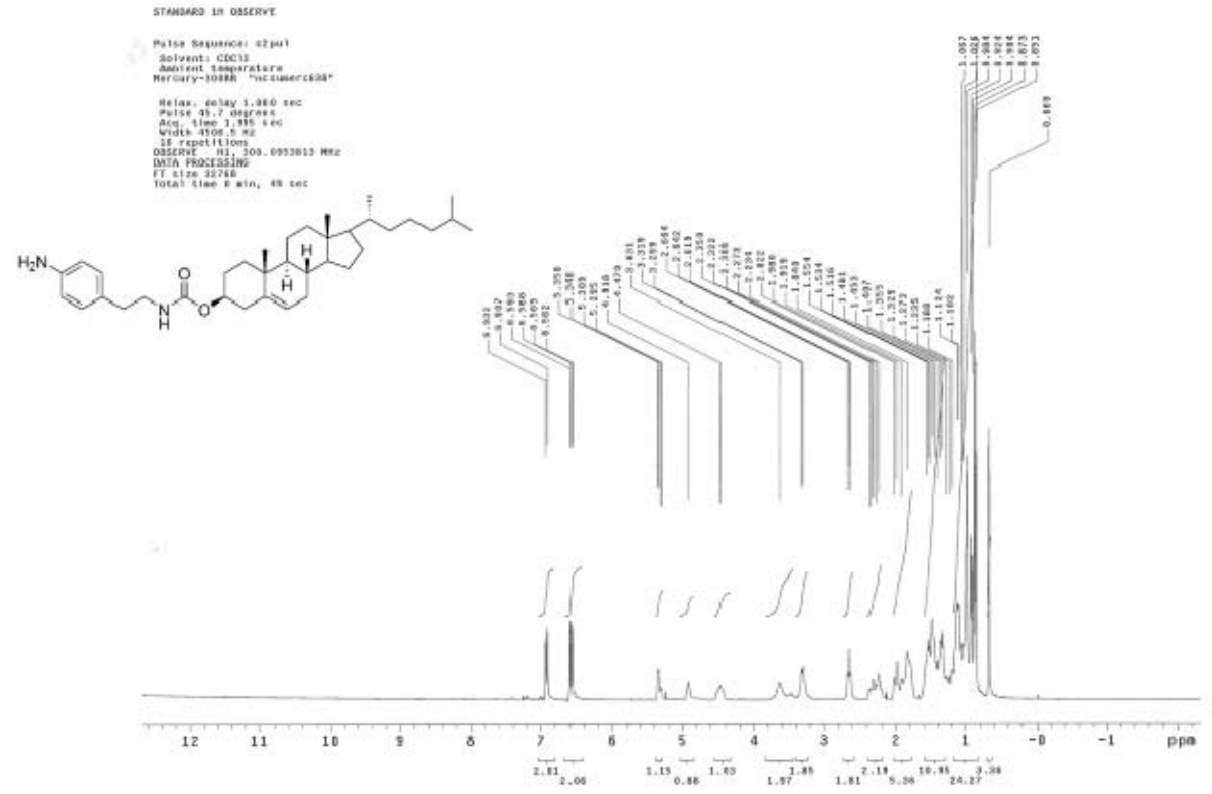


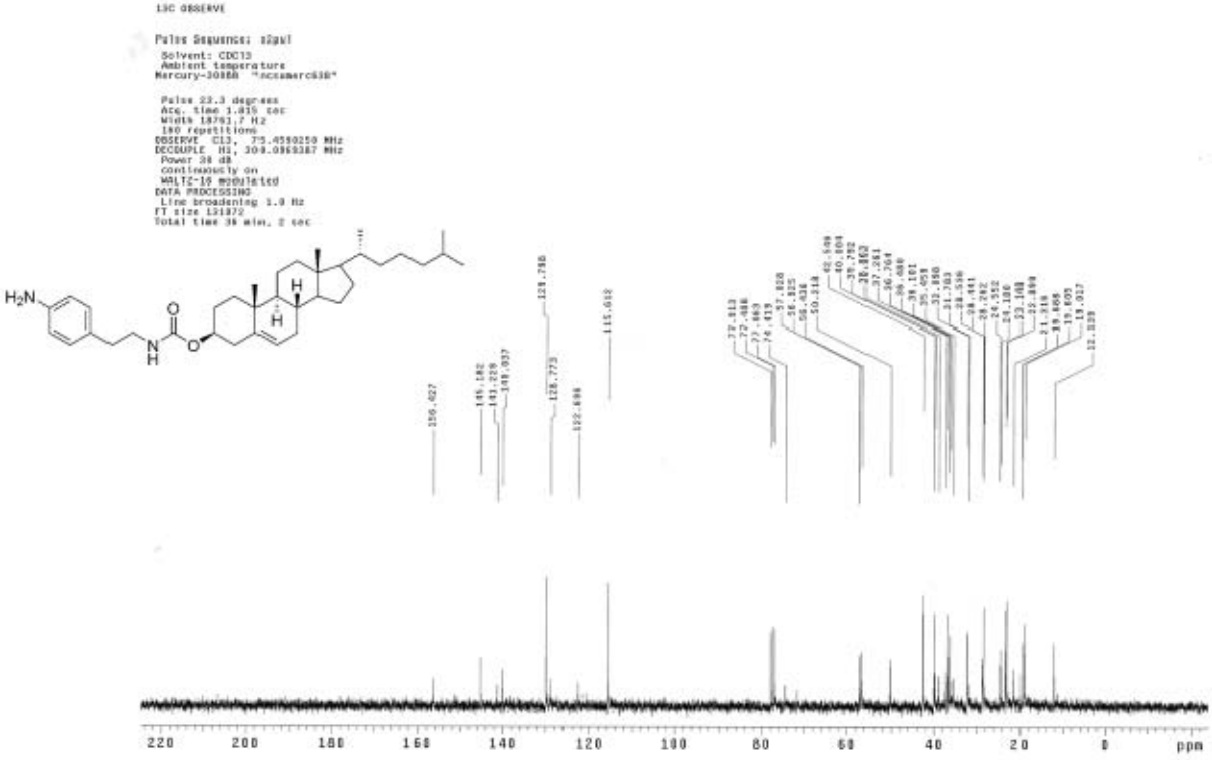


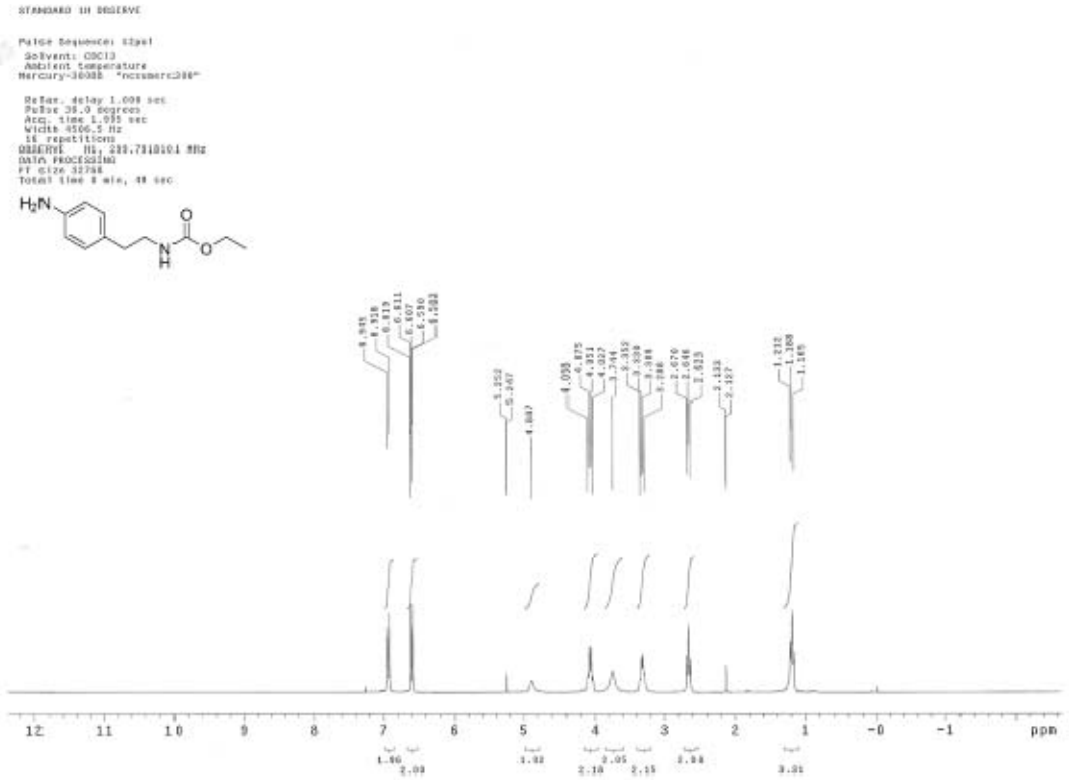

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.595 sec
Width 4506.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
88 repetitions
DECOUPLE H1, 300.0868387 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.8 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

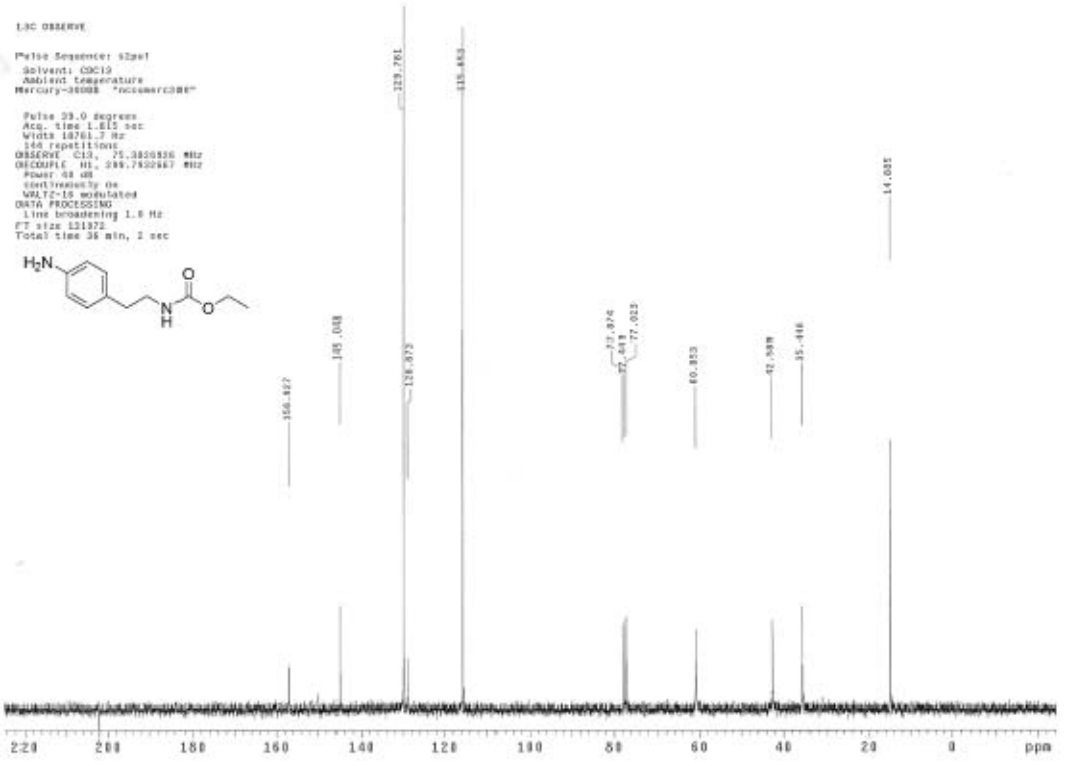

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.985 sec
Width 4508.5 Hz
16 repetitions
Total time 0 min, 48 sec
ppm

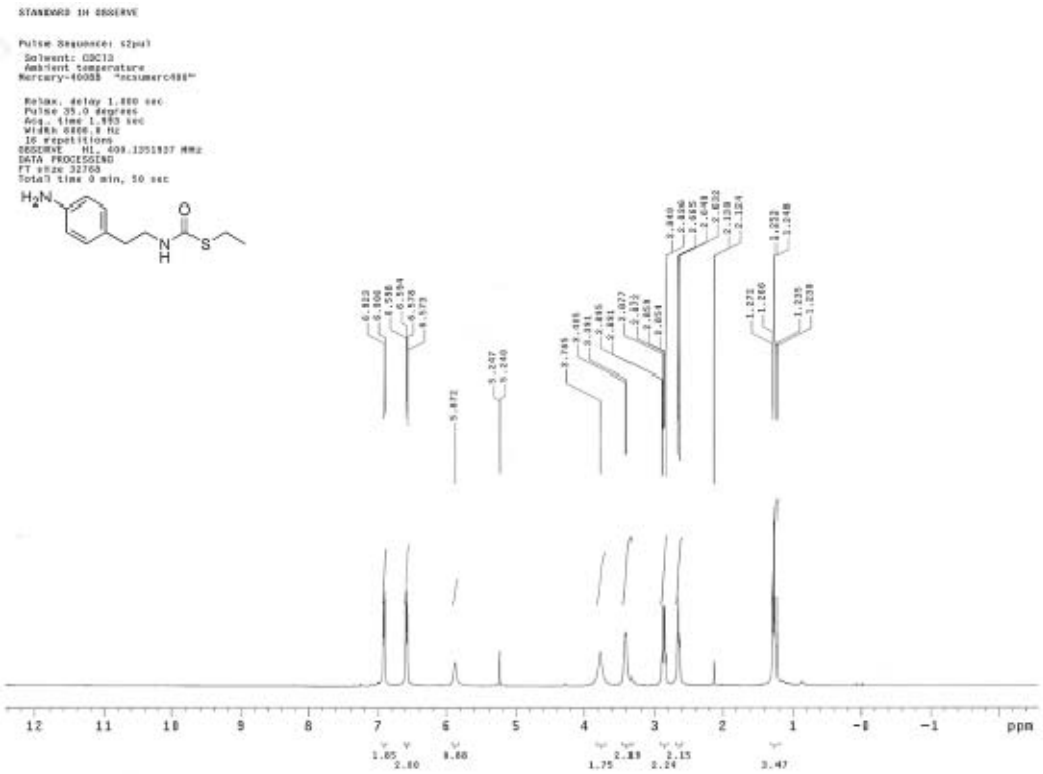

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.835 sec
Width 18781.7 Hz
288 repetitions
Power 38 dB
continuously on
WALTZ-16 modulated
Line broadening 1.0 Hz
Total time 38 min, 2 sec
ppm

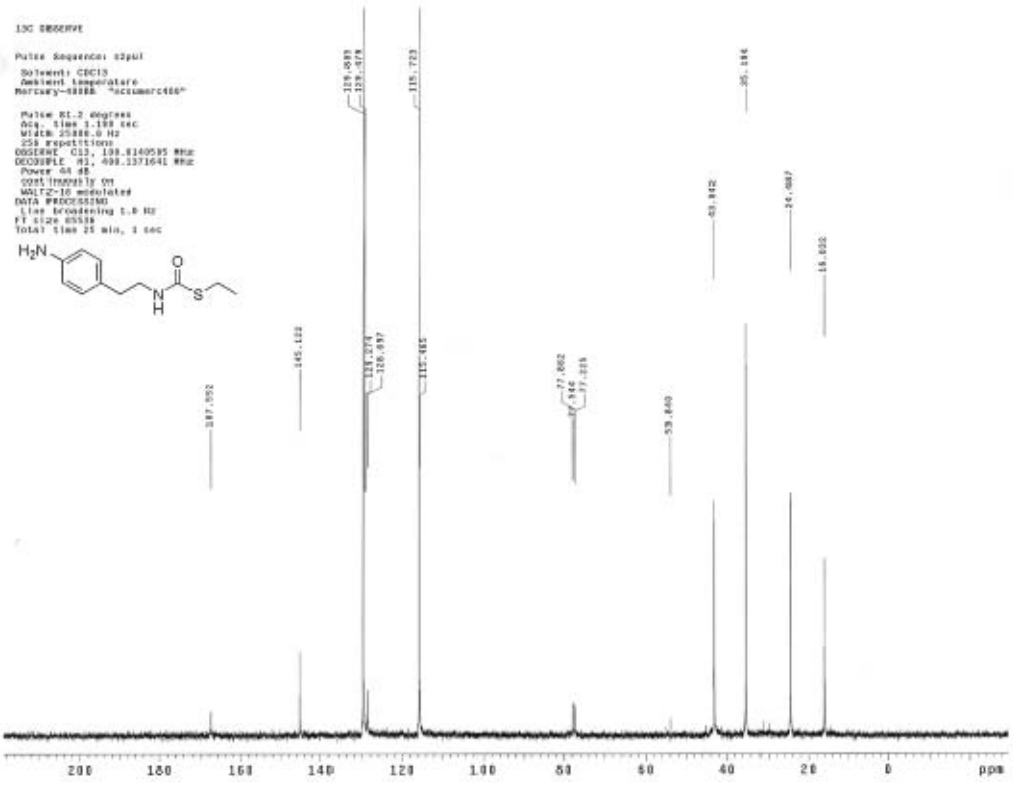

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "nccsamerc038"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4508.5 Hz
16 repetitions
OBSERVE H1, 300.0553813 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
H2N
O
N
H
O
H
ppm

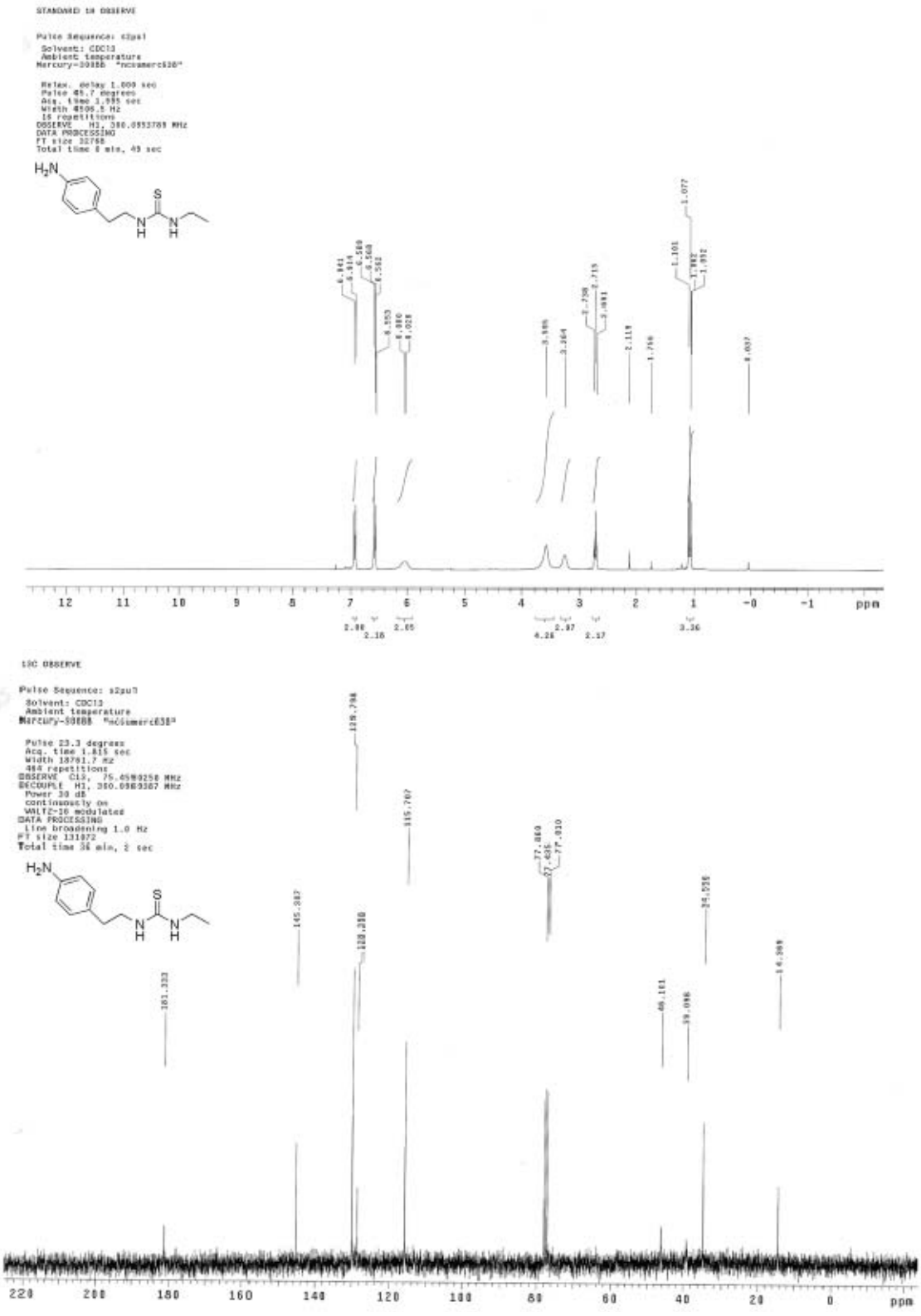

13C OBSERVE
Pulse Sequence: s2gxl
Solvent: CDCl3
Ambient temperature
Mercury-300BB "nccsamerc038"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
180 repetitions
OBSERVE C13, 75.4550259 MHz
DECOUPLE H1, 300.0869387 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 38 min, 2 sec
H2N
O
N
H
O
H
ppm

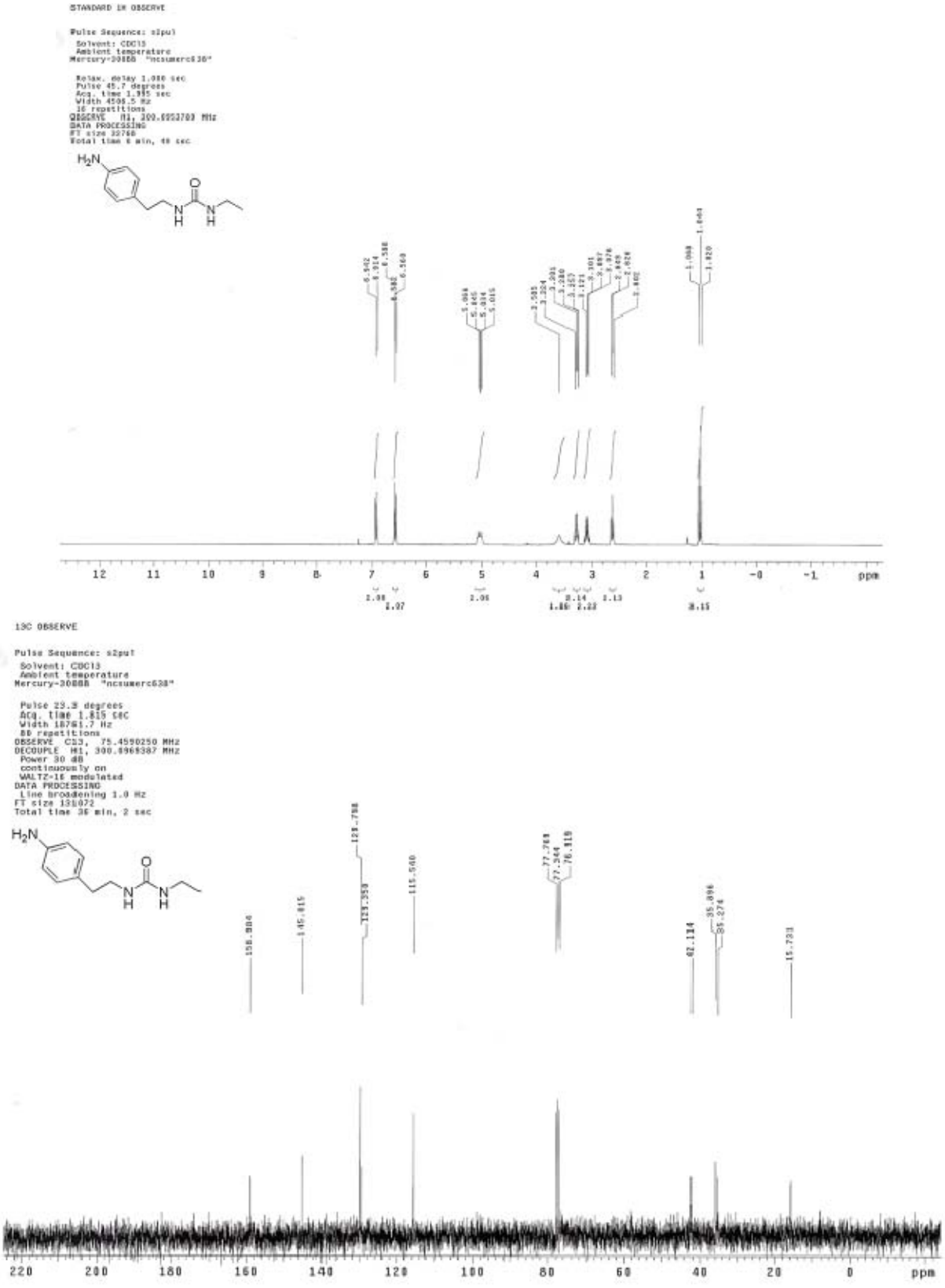


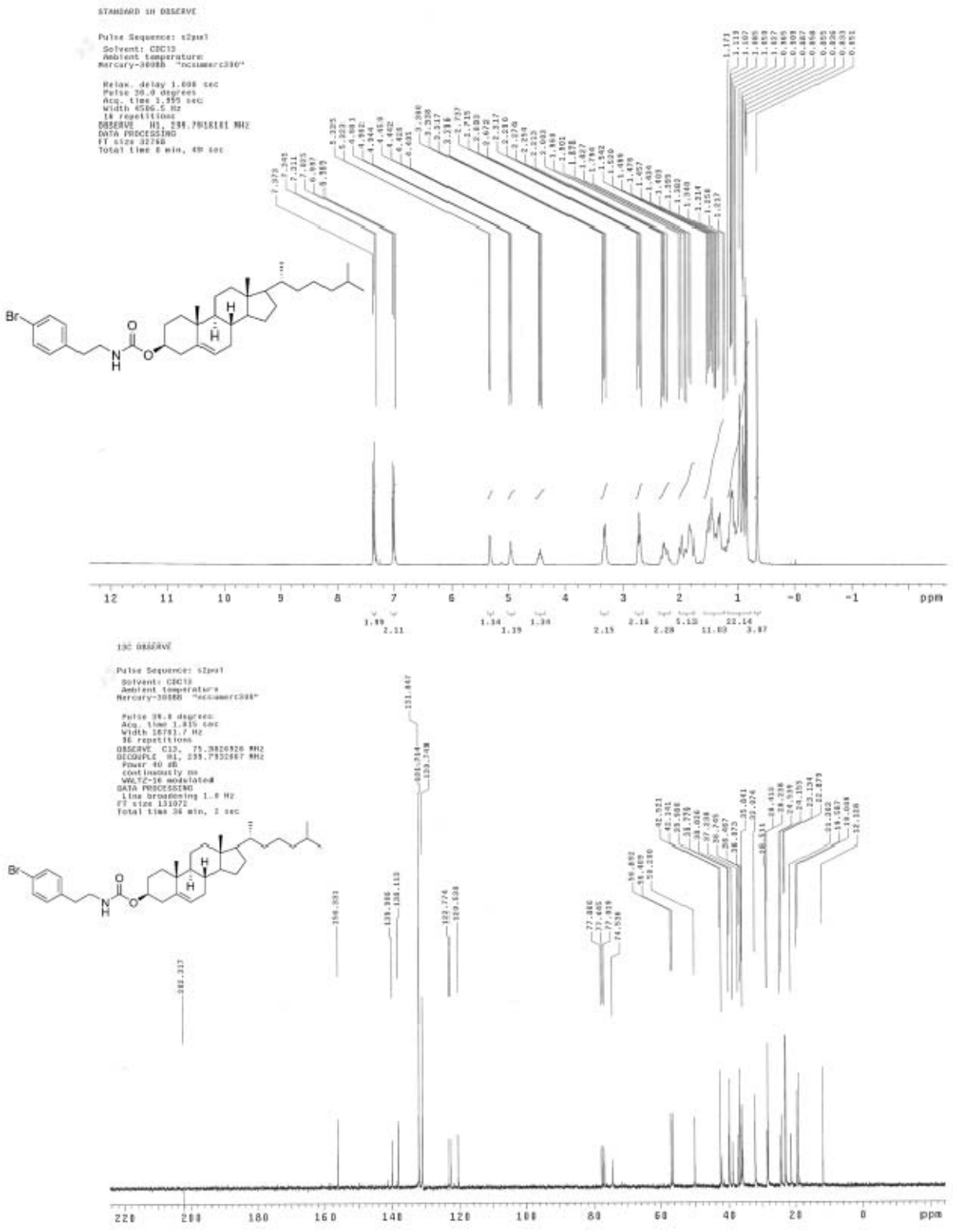


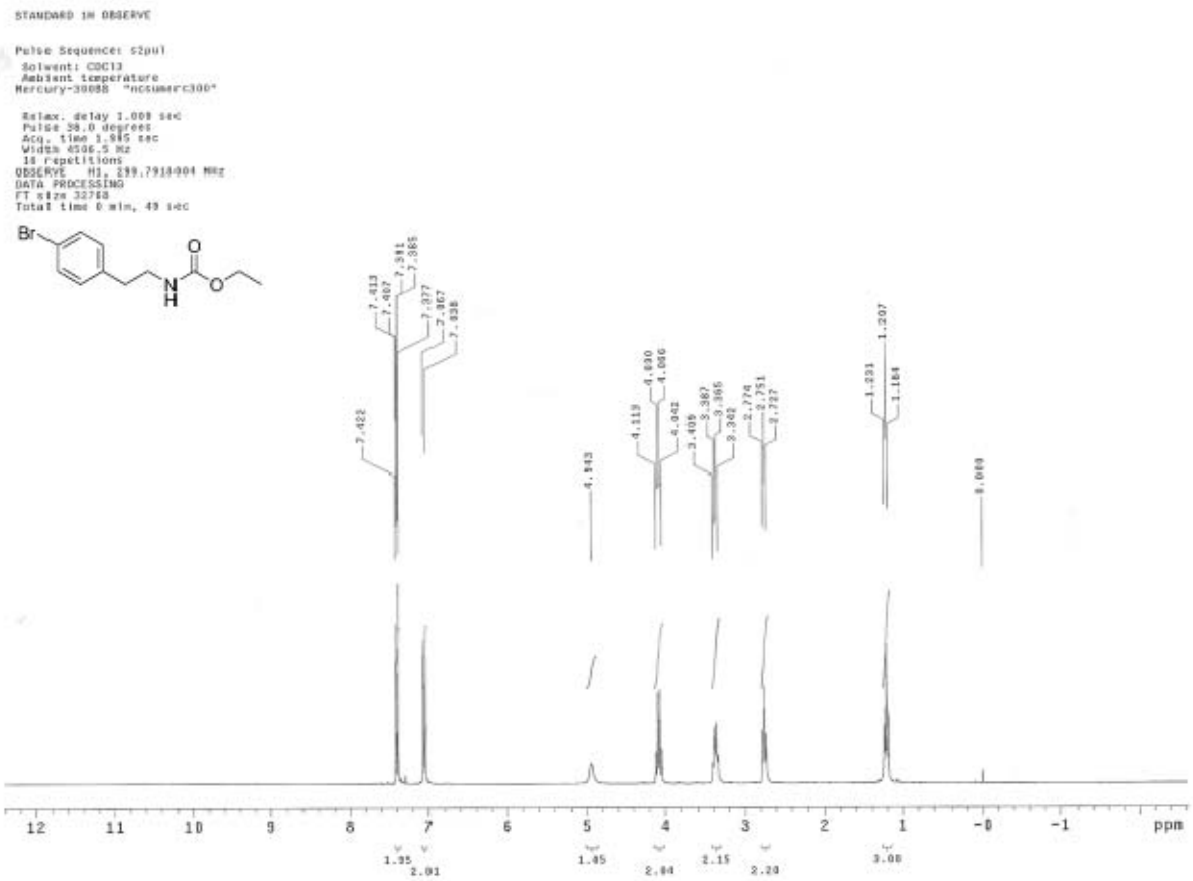


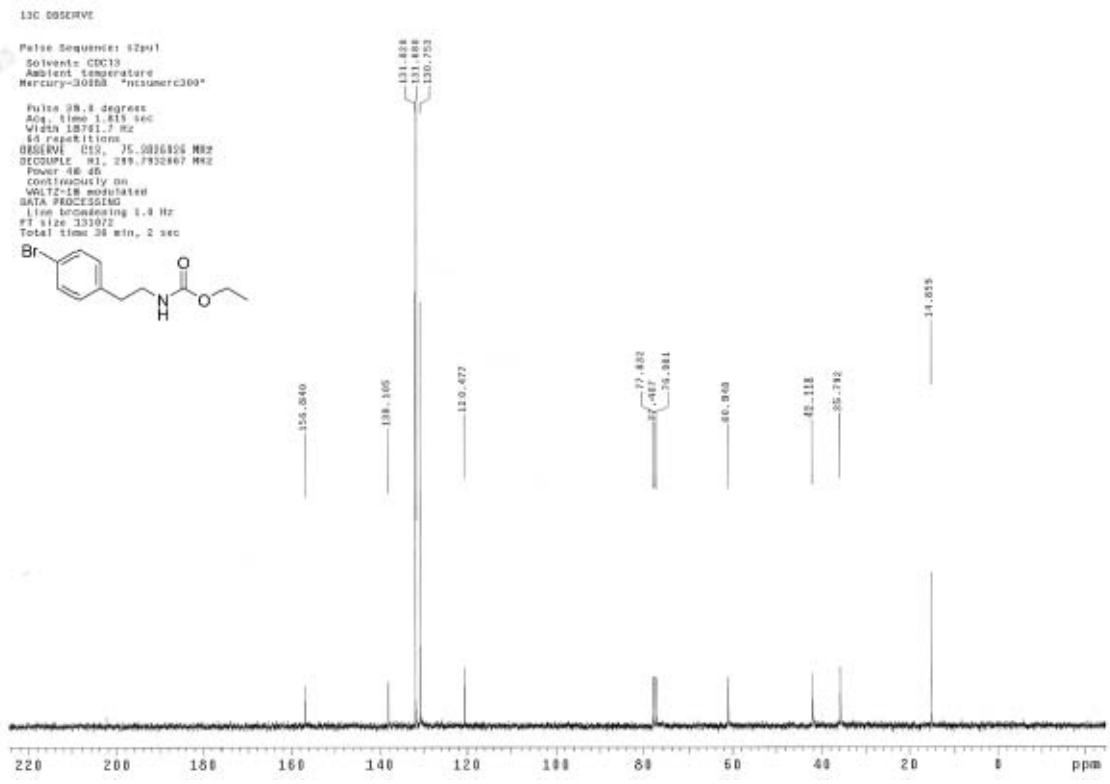


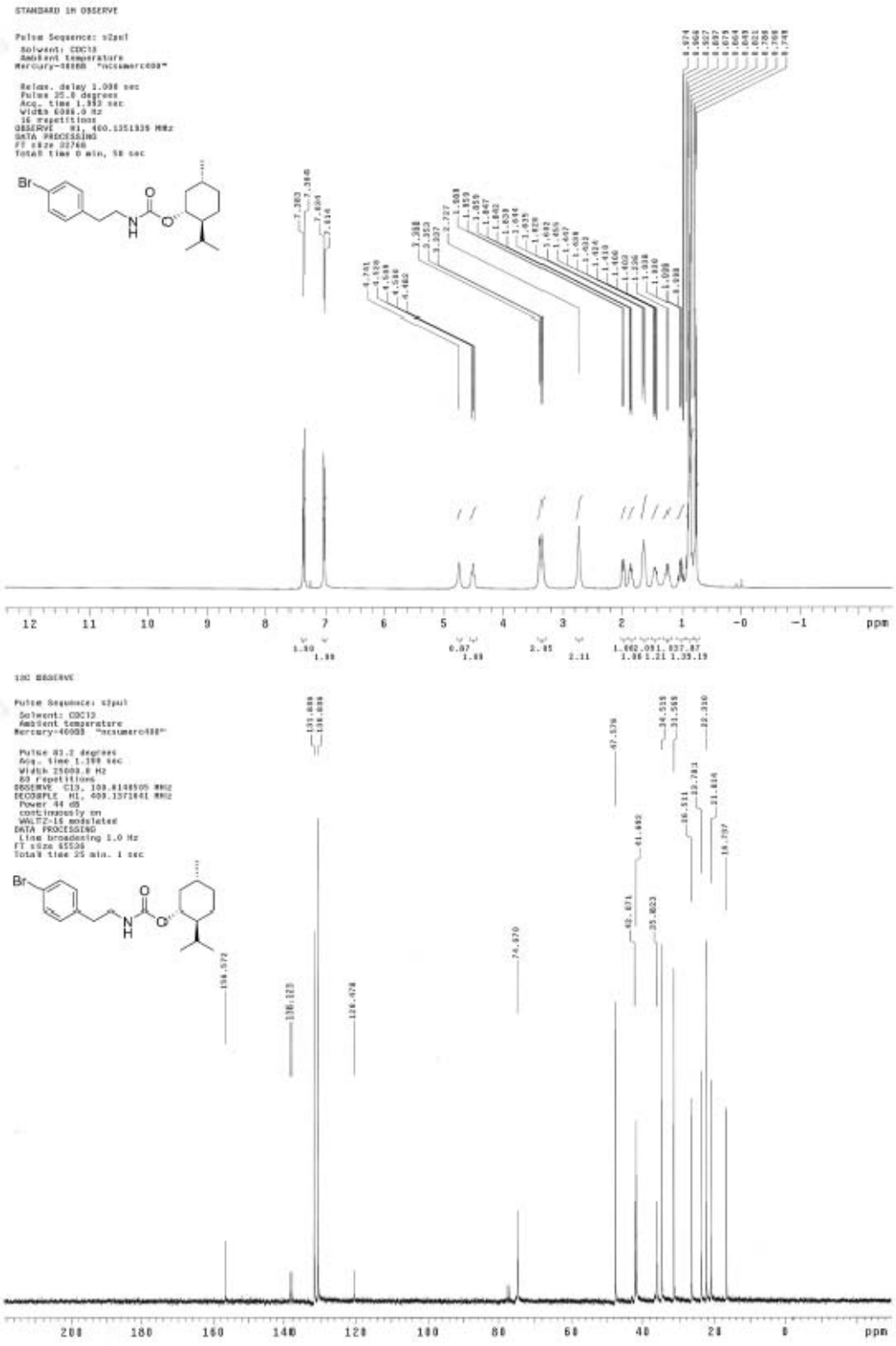

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsumerc638"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 3.595 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 300.0553705 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 45 sec
H2N
S
N
H
N
H
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsumerc638"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
468 repetitions
OBSERVE C13, 75.4698258 MHz
DECOUPLE H1, 300.0909307 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
H2N
S
N
H
N
H
220
200
180
160
140
120
100
80
60
40
20
0
ppm

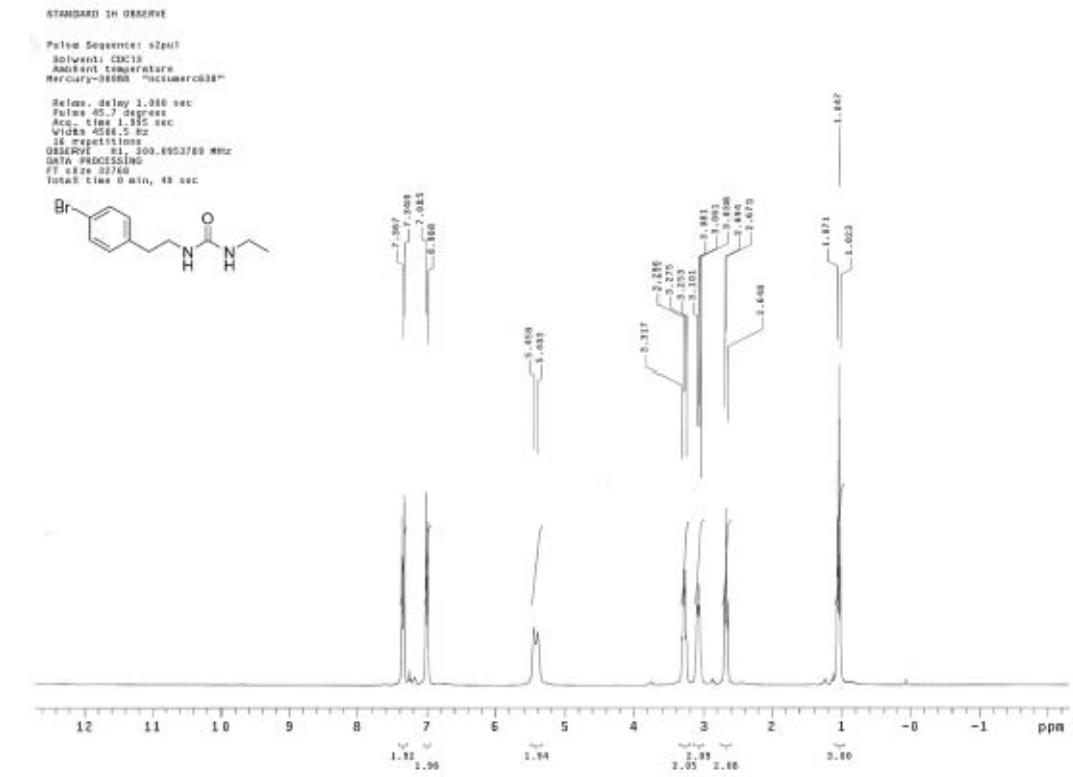

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 300.0053703 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
H2N
O
N
H
N
H
ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Pulse 23.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
80 repetitions
OBSERVE C13, 75.4590250 MHz
DECOUPLE H1, 300.0969387 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
H2N
O
N
H
N
H
158.884
145.815
129.788
129.359
115.560
77.769
77.344
76.919
62.134
35.096
34.274
15.783
ppm

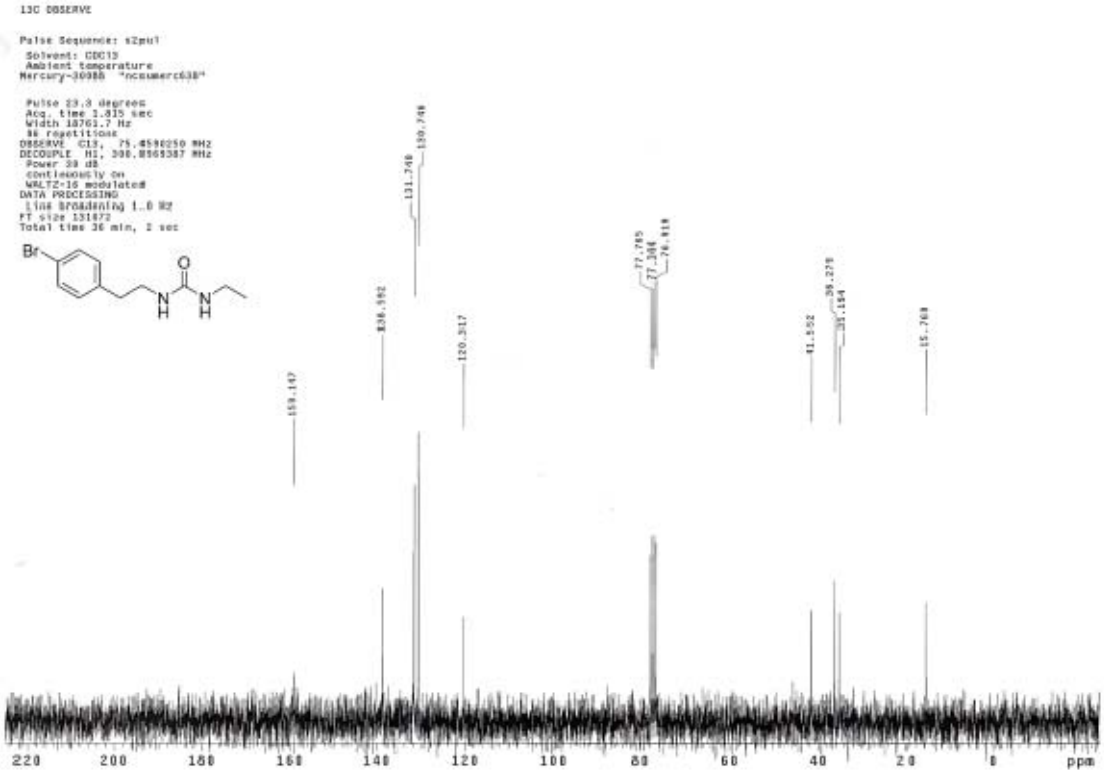

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc330"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.355 sec
Width 4586.5 Hz
16 repetitions
OBSERVE H1, 299.7916181 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
Br
O
N
H
H
H
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
2.11
1.14
1.19
1.38
2.15
2.18
2.28
5.13
11.03
22.14
3.87
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Pulse 30.0 degrees
Acq. time 1.815 sec
Width 18781.7 Hz
36 repetitions
OBSERVE C13, 75.3826926 MHz
DECOUPLE H1, 299.7932667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 1 sec
Br
O
N
H
H
H
220
200
180
160
140
120
100
80
60
40
20
0
ppm

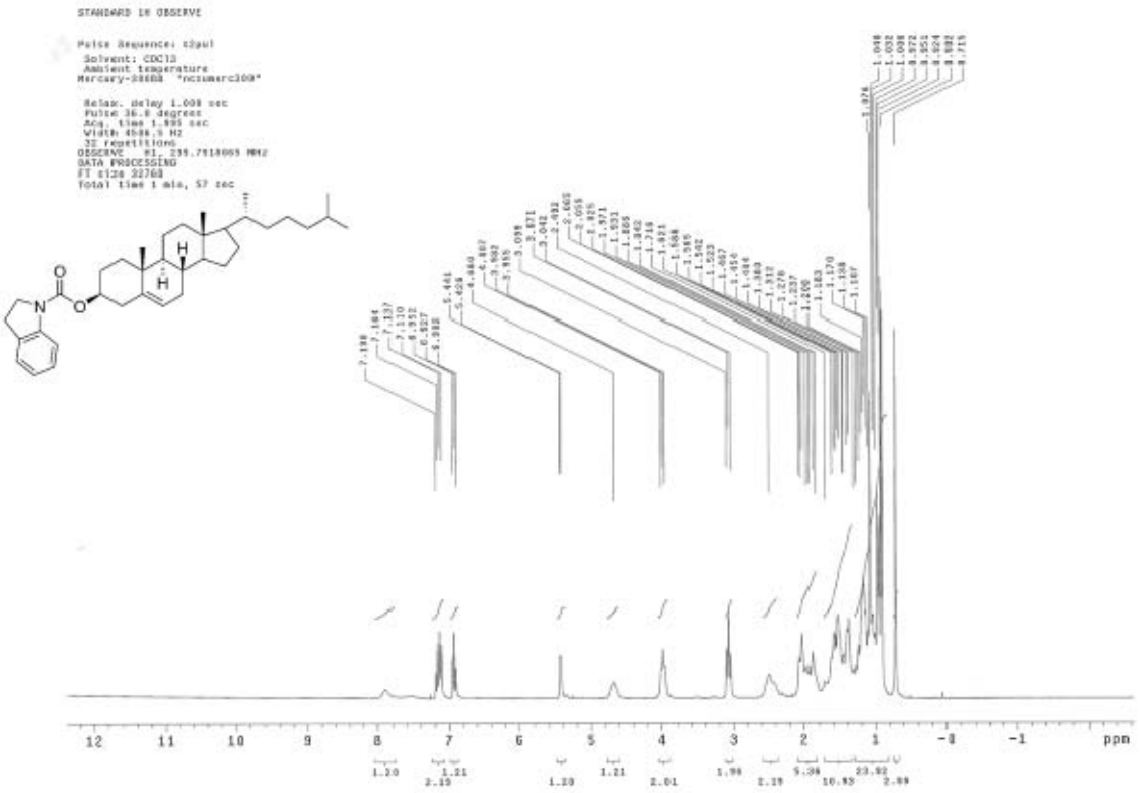

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.985 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 299.7918004 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
Br
O
N
H
O
7.422
7.413
7.407
7.391
7.385
7.377
7.367
7.838
4.943
4.119
4.030
4.016
4.002
3.405
3.387
3.365
3.342
2.774
2.751
2.727
1.231
1.207
1.184
0.000
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.95
2.01
1.45
2.84
2.15
2.20
3.00

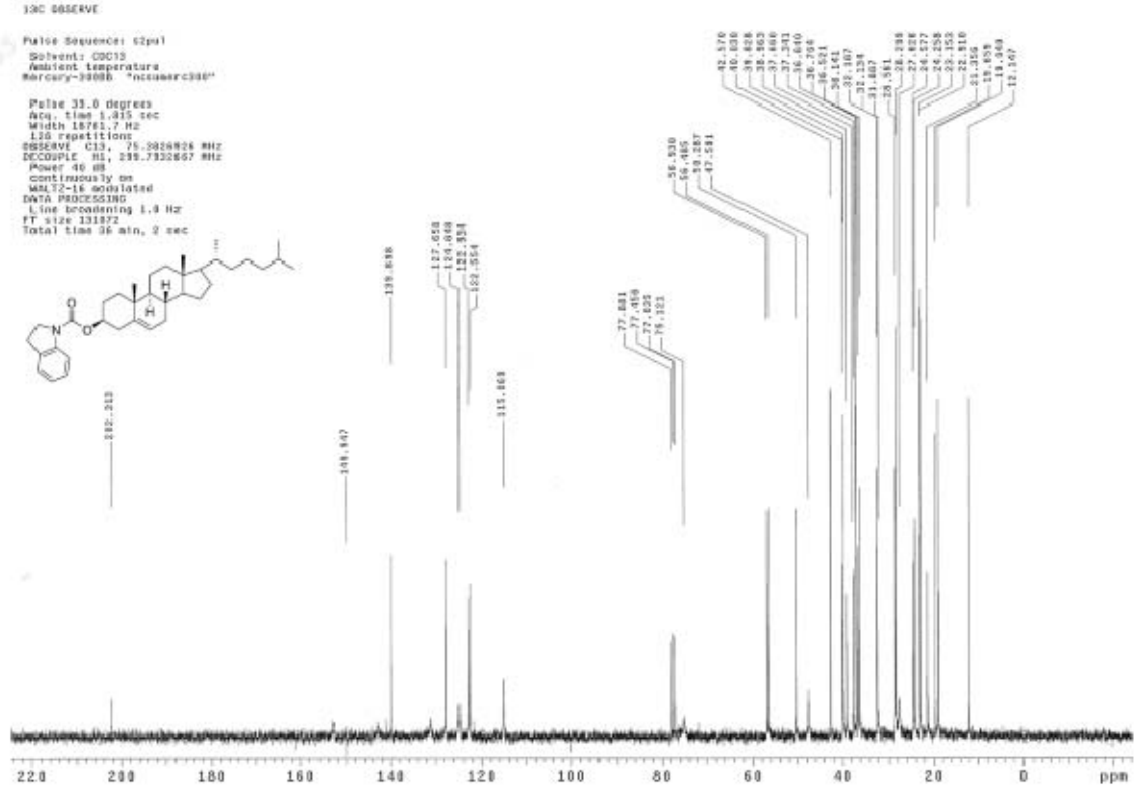

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 28.8 degrees
Acq. time 1.815 sec
Width 18781.7 Hz
64 repetitions
OBSERVE C13, 75.4836826 MHz
DECOUPLE H1, 299.7932867 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 30 min, 2 sec
Br
O
N
H
O
156.840
138.105
131.838
131.808
130.753
120.477
77.432
77.407
76.981
60.868
40.118
35.792
14.655
220
200
180
160
140
120
100
80
60
40
20
0
ppm

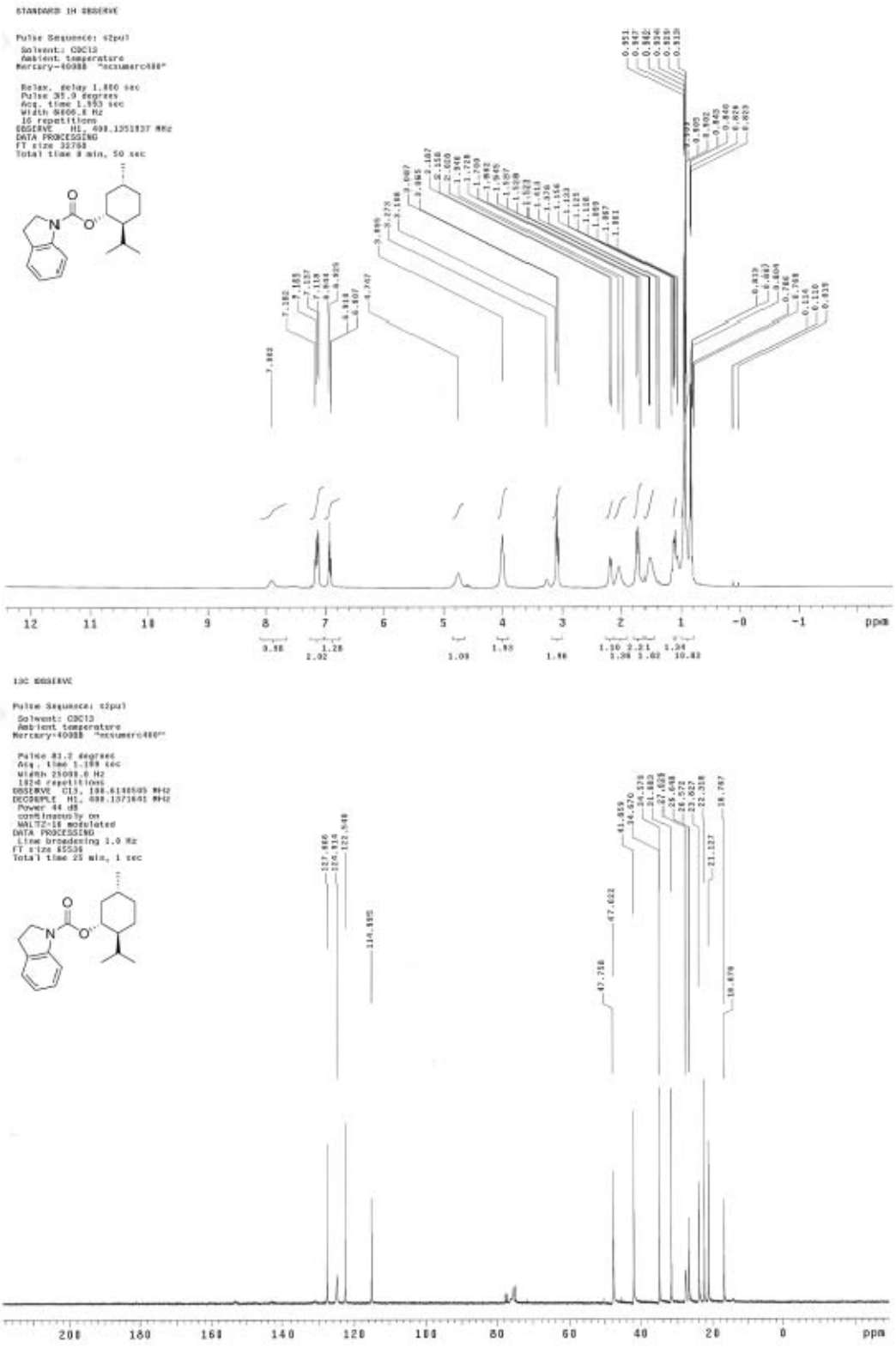


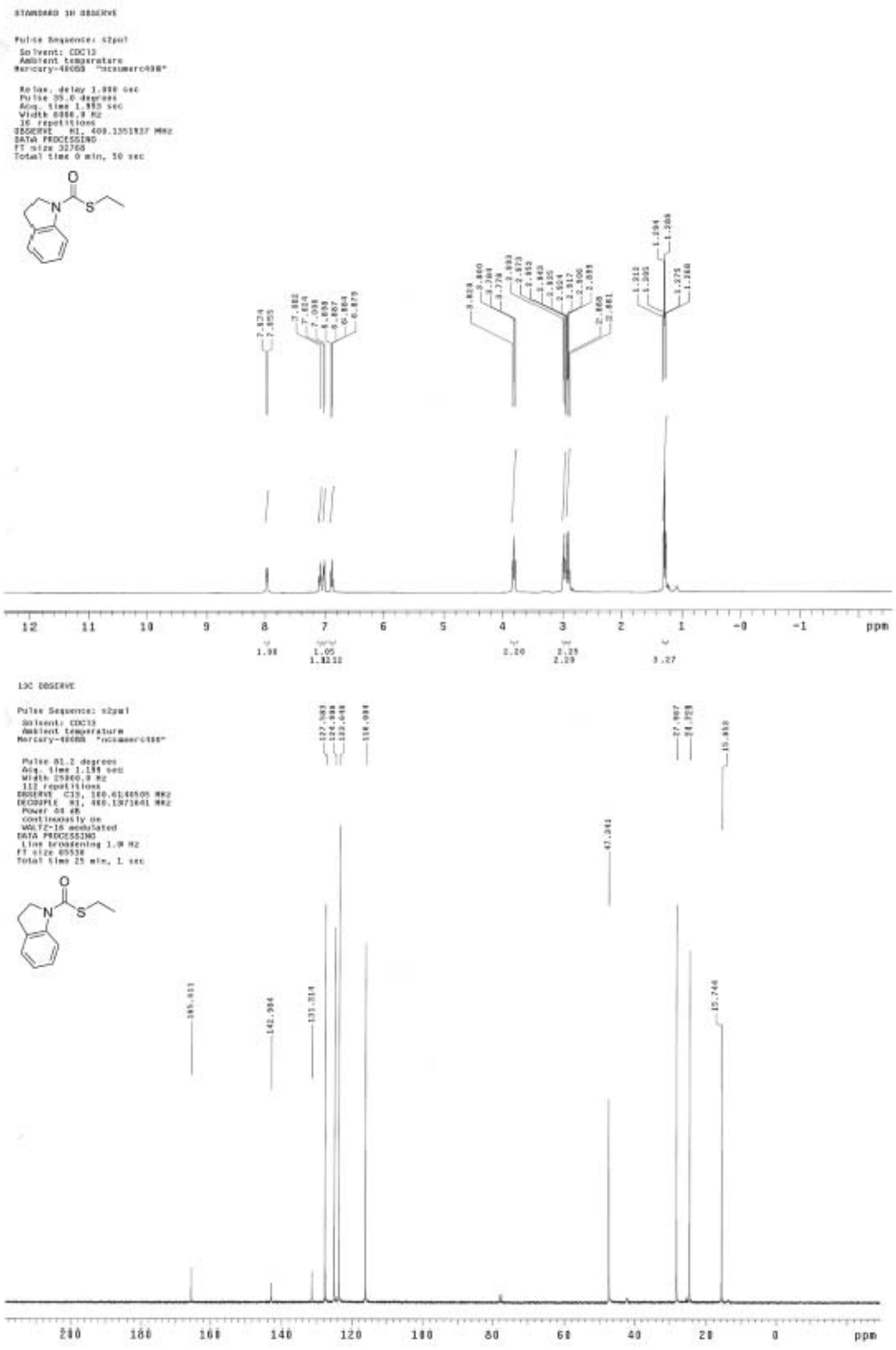

STANDARD 1H OBSERVE
Br
O
N
H
N
H
ppm

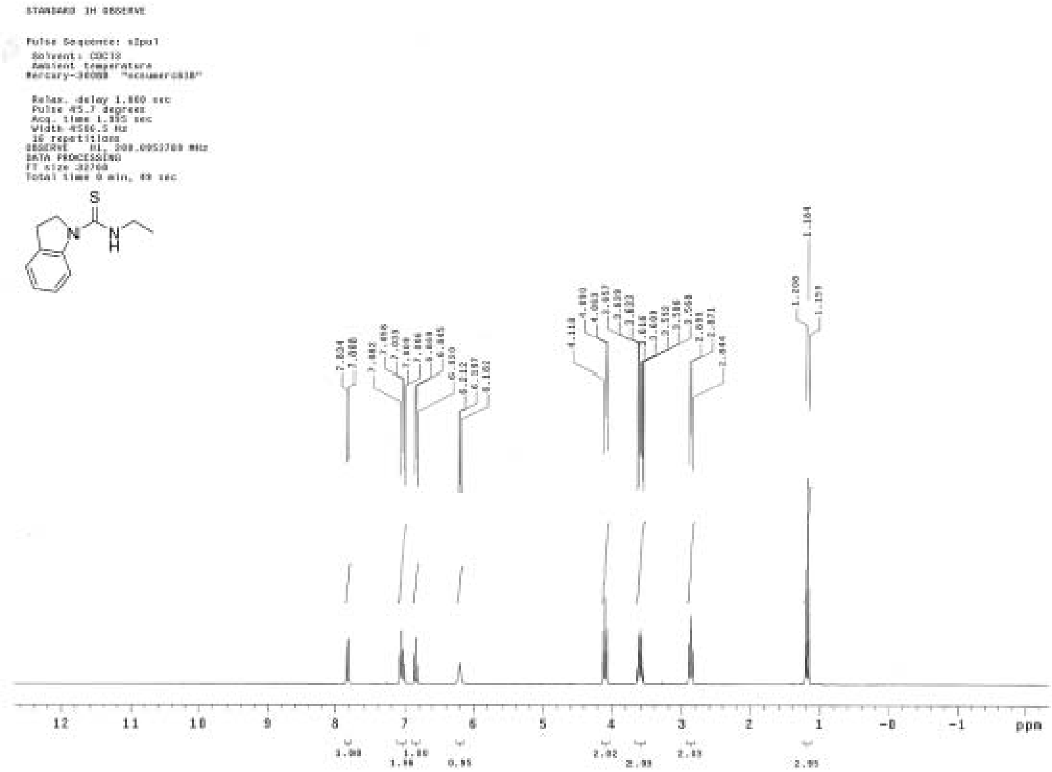

13C OBSERVE
Br
O
N
H
N
H
ppm

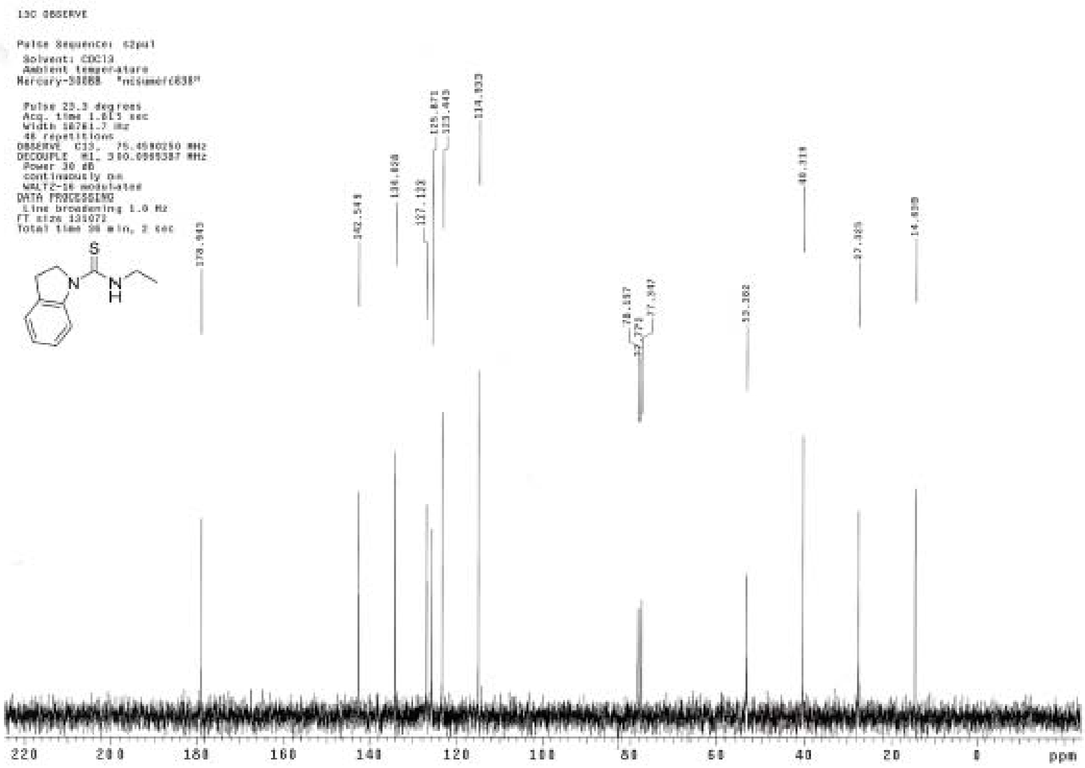

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 36.0 degrees
Acq. time 1.995 sec
Width 4504.5 Hz
32 repetitions
OBSERVE H1, 299.7918055 MHz
DATA PROCESSING
FT SIZE 32768
Total time 1 min, 57 sec
ppm

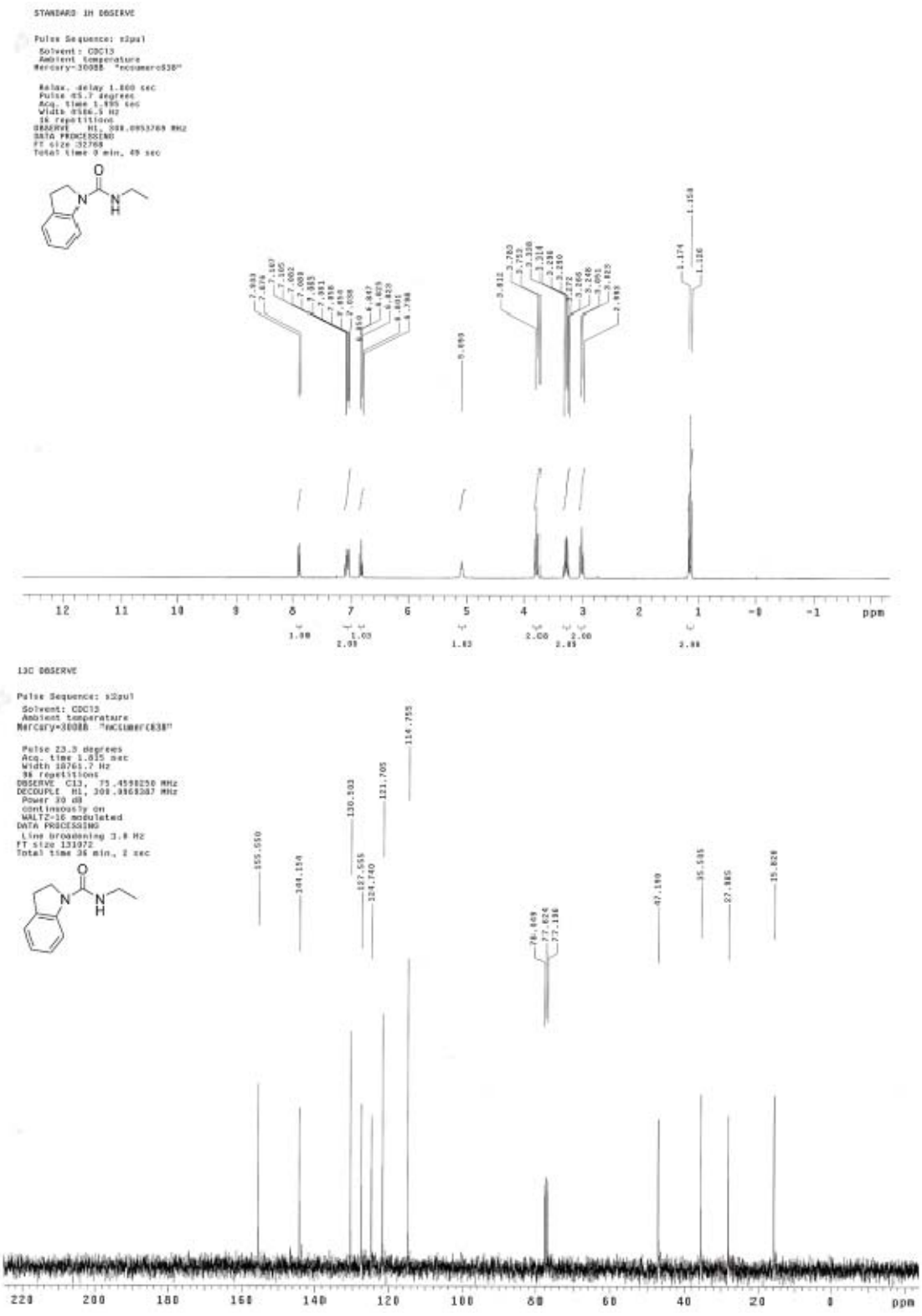

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 33.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
136 repetitions
OBSERVE C13, 75.3026826 MHz
DECOUPLE H1, 299.7932857 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

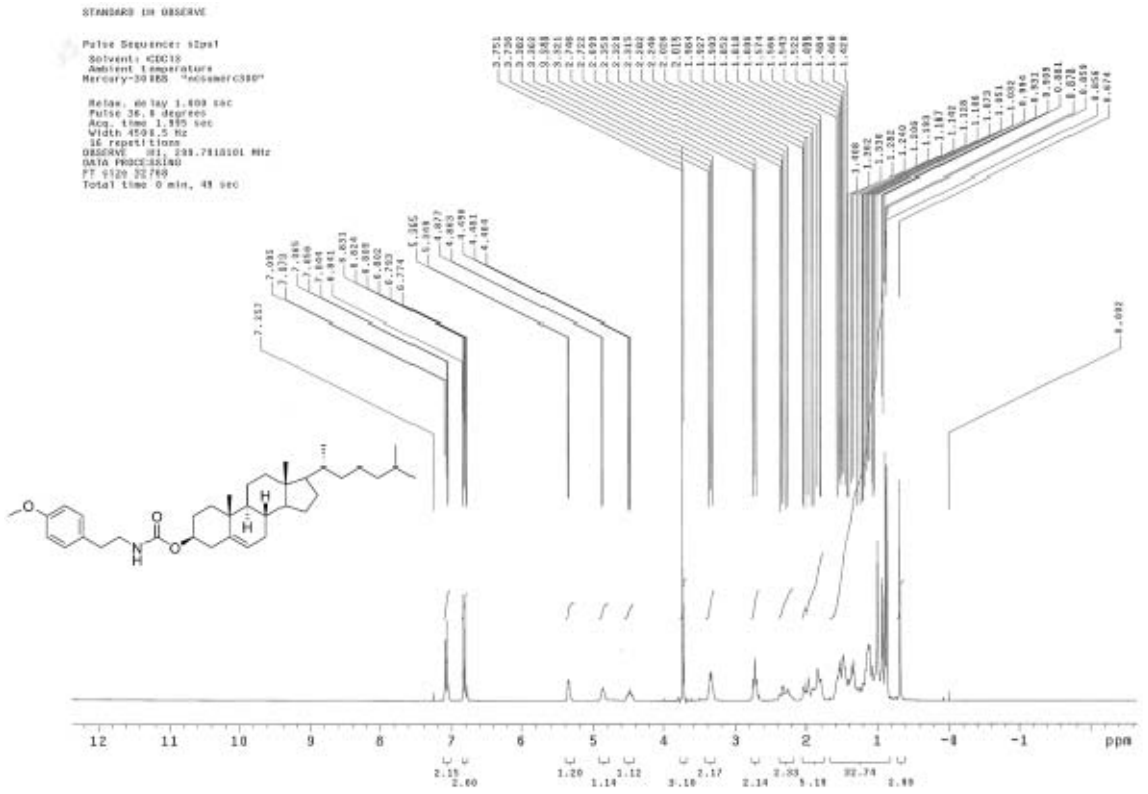


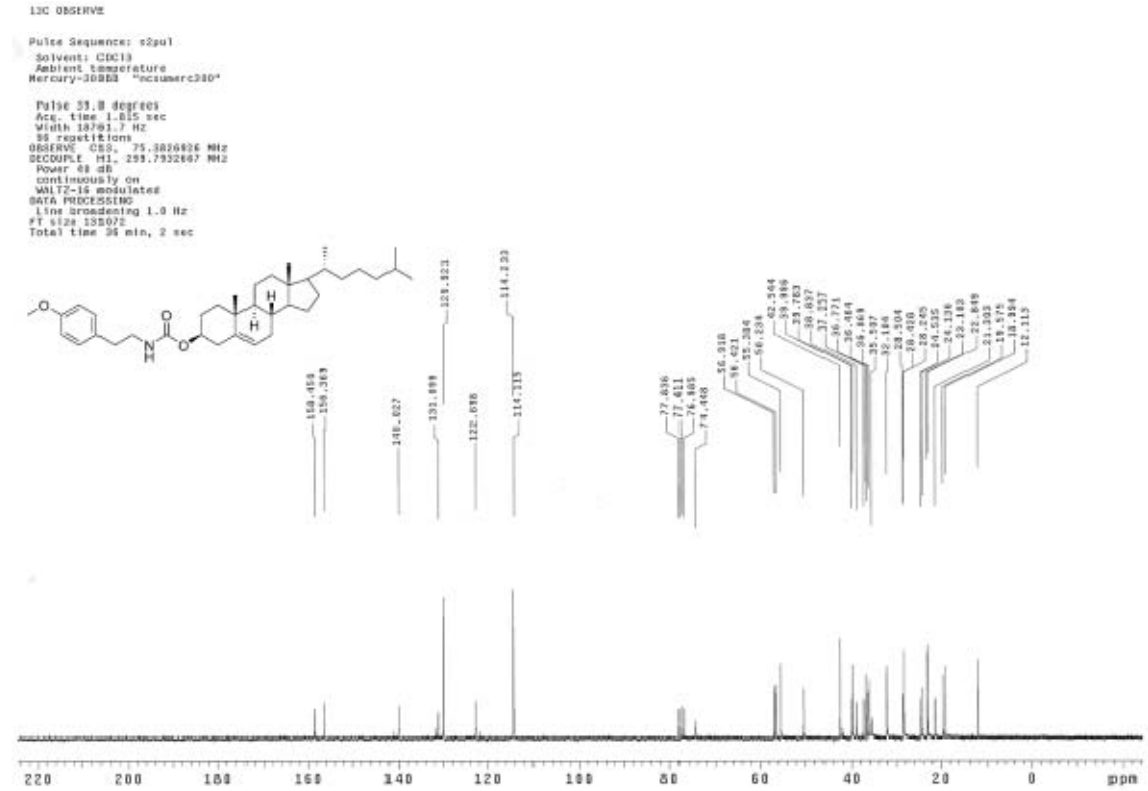


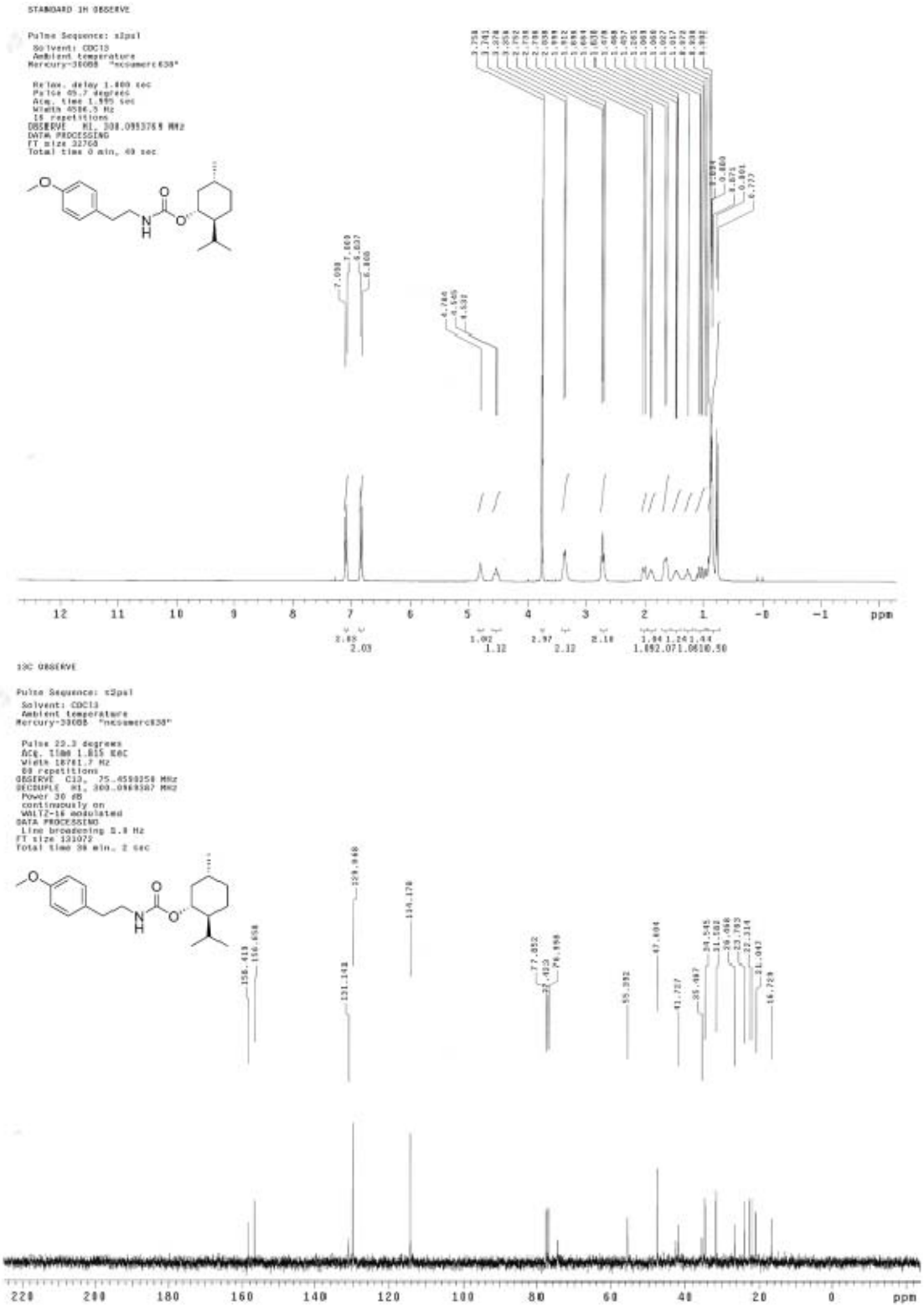


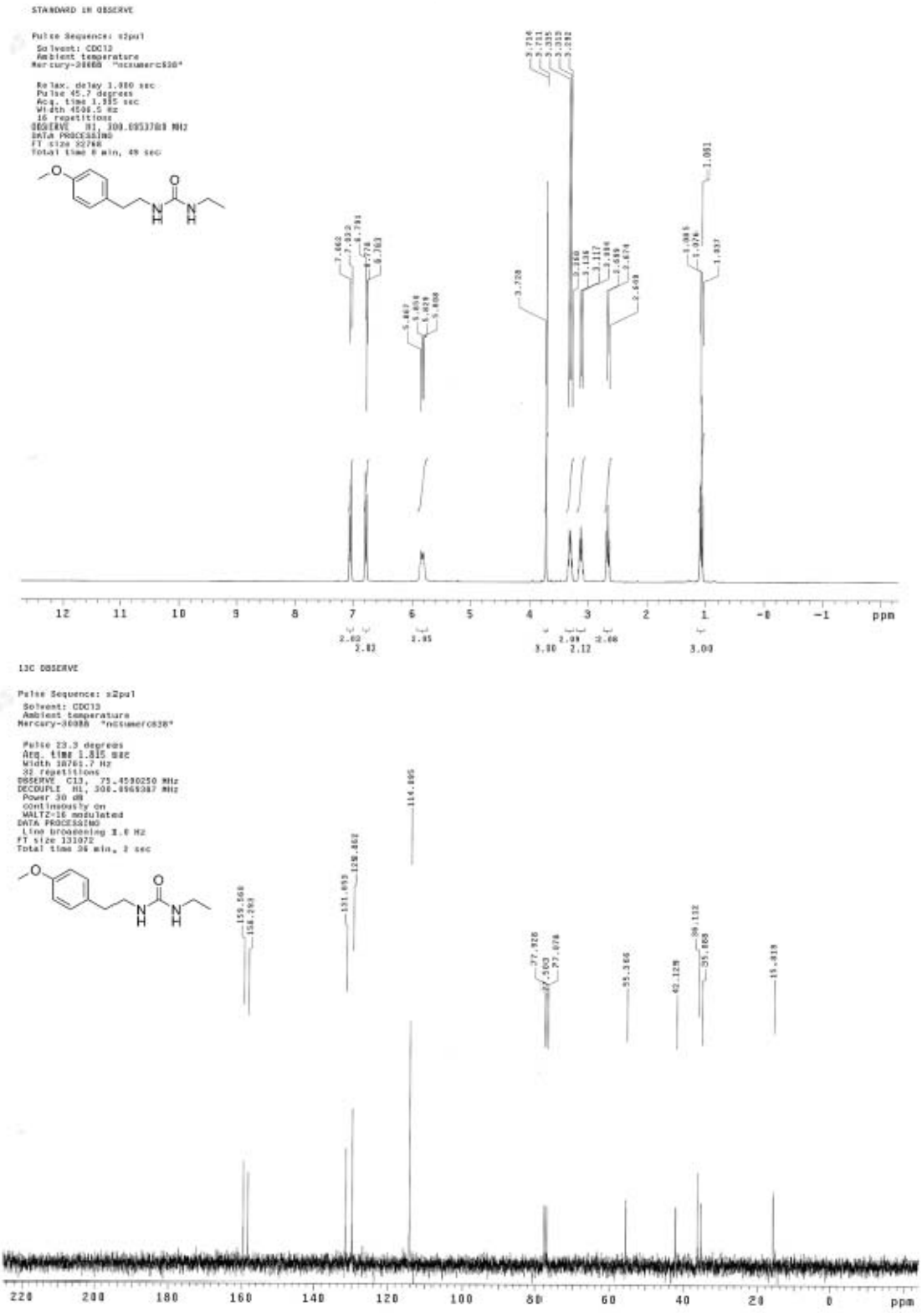


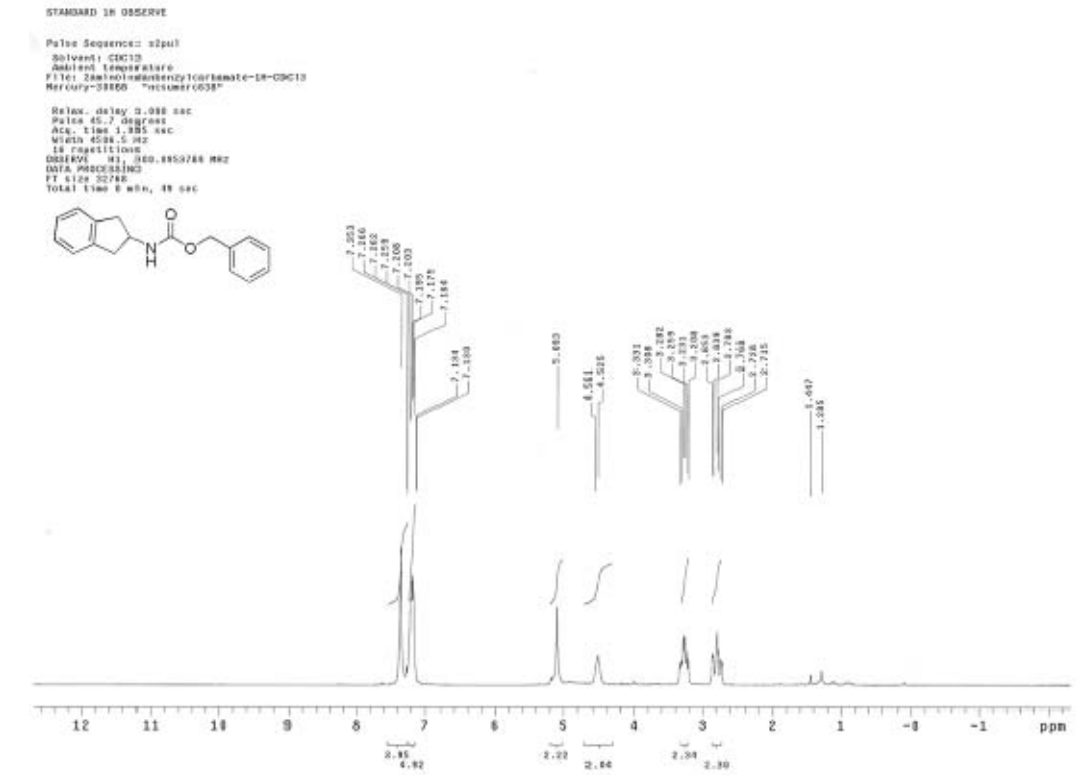

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsumerc838"
Relax. delay 1.800 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 300.0053769 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsumerc838"
Pulse 23.3 degrees
Acq. time 1.015 sec
Width 18761.7 Hz
96 repetitions
OBSERVE C13, 75.4598250 MHz
DECOUPLE H1, 300.1868387 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

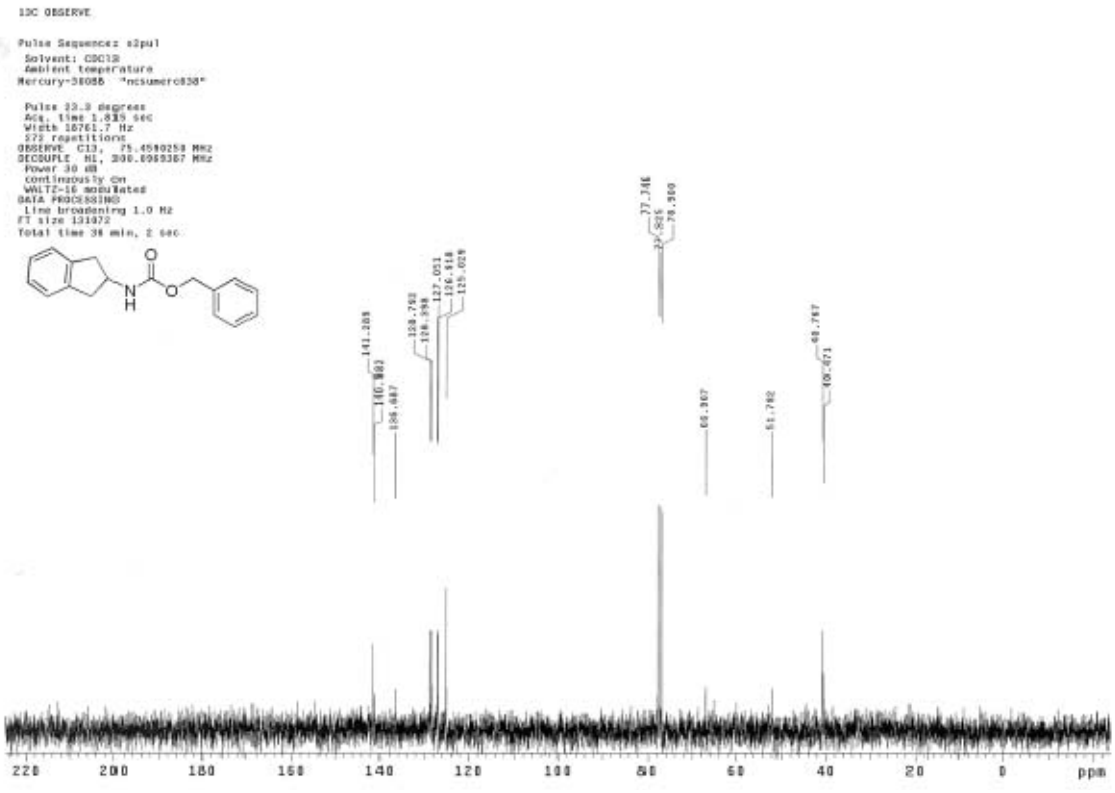

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Relax. delay 1.000 sec
Pulse 36.8 degrees
Acq. time 1.995 sec
Width 4598.5 Hz
16 repetitions
OBSERVE H1, 299.7818101 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
ppm

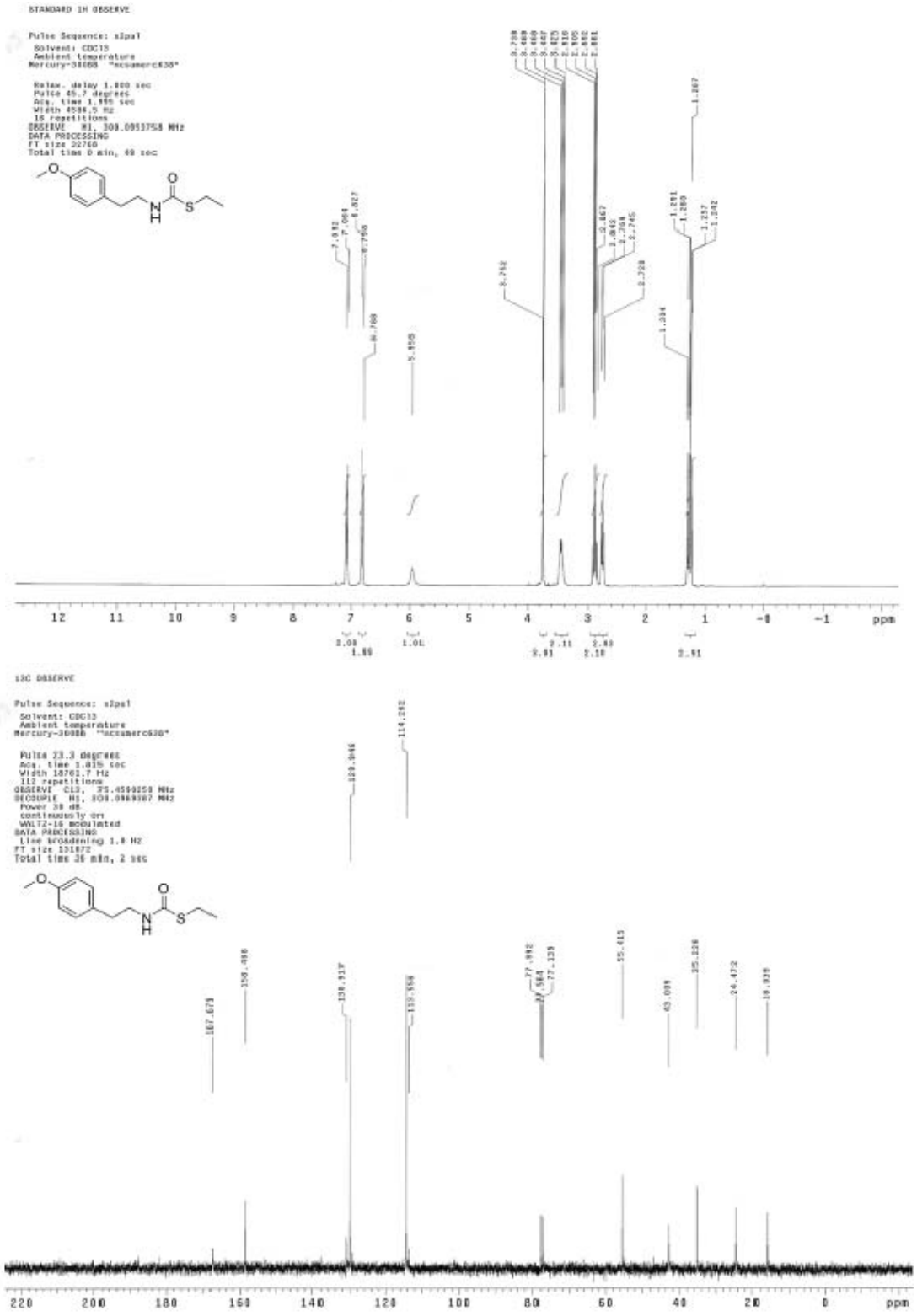

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Pulse 31.8 degrees
Acq. time 1.015 sec
Width 18761.7 HZ
96 repetitions
OBSERVE C13, 75.3826826 MHz
DECOUPLE H1, 299.7932667 MHz
Power 48 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

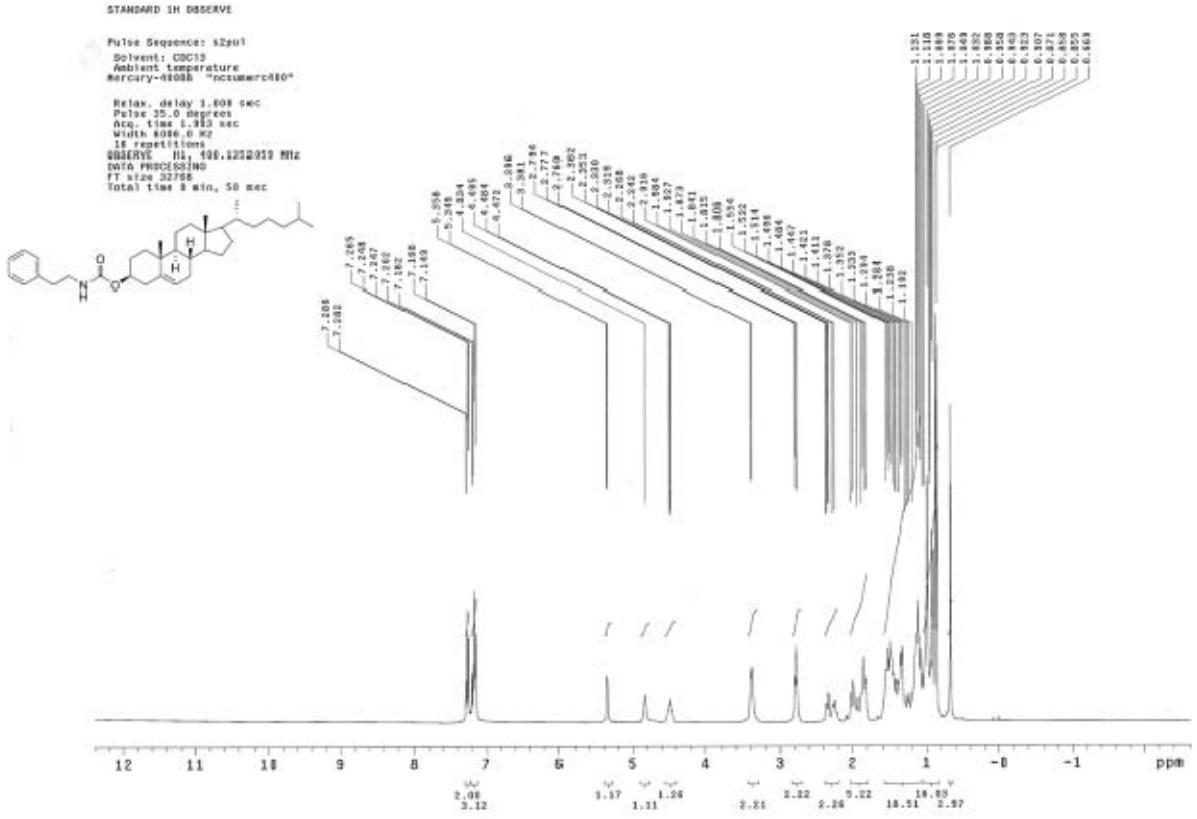

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsamerc630"
Relax. delay 1.000 sec
Pulse 85.7 degrees
Acq. time 1.595 sec
Width 4586.5 Hz
16 repetitions
OBSERVE H1, 300.0953769 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mcsamerc630"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
80 repetitions
OBSERVE C13, 75.4598358 MHz
DECOUPLE H1, 300.0969367 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FT size 131072
Total time 30 min, 2 sec
220 200 180 160 140 120 100 80 60 40 20 0 ppm

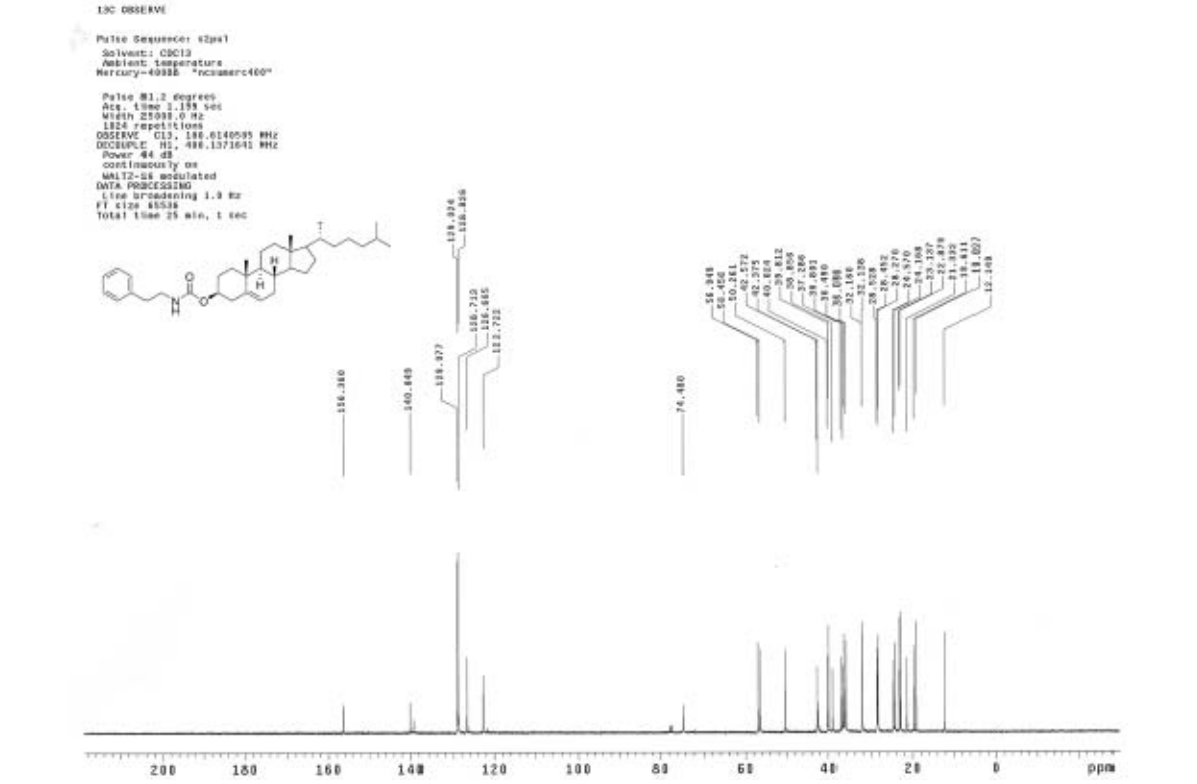

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.815 sec
Width 4506.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
32 repetitions
DECOUPLE H1, 300.0565387 MHz
Power 30 dB
continously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

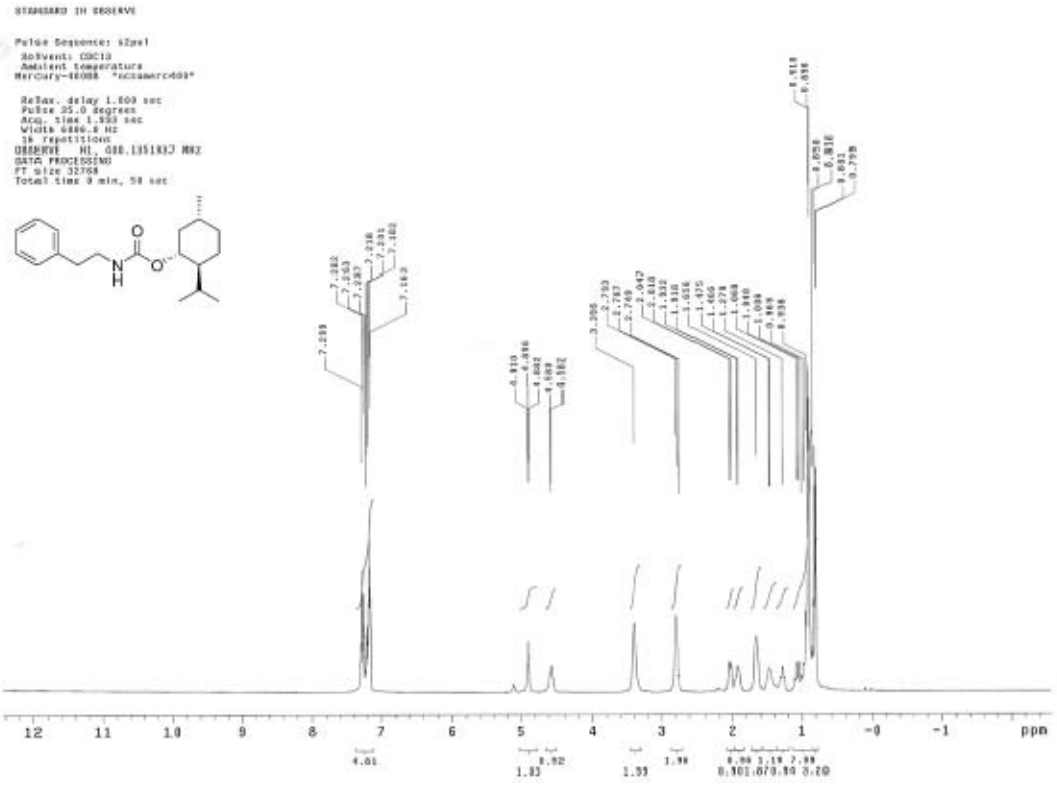

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Relax. delay 5.000 sec
Pulse 45.7 degrees
Acq. time 1.885 sec
Width 4598.5 Hz
16 repetitions
OBSERVE H1, 300.8953788 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
ppm

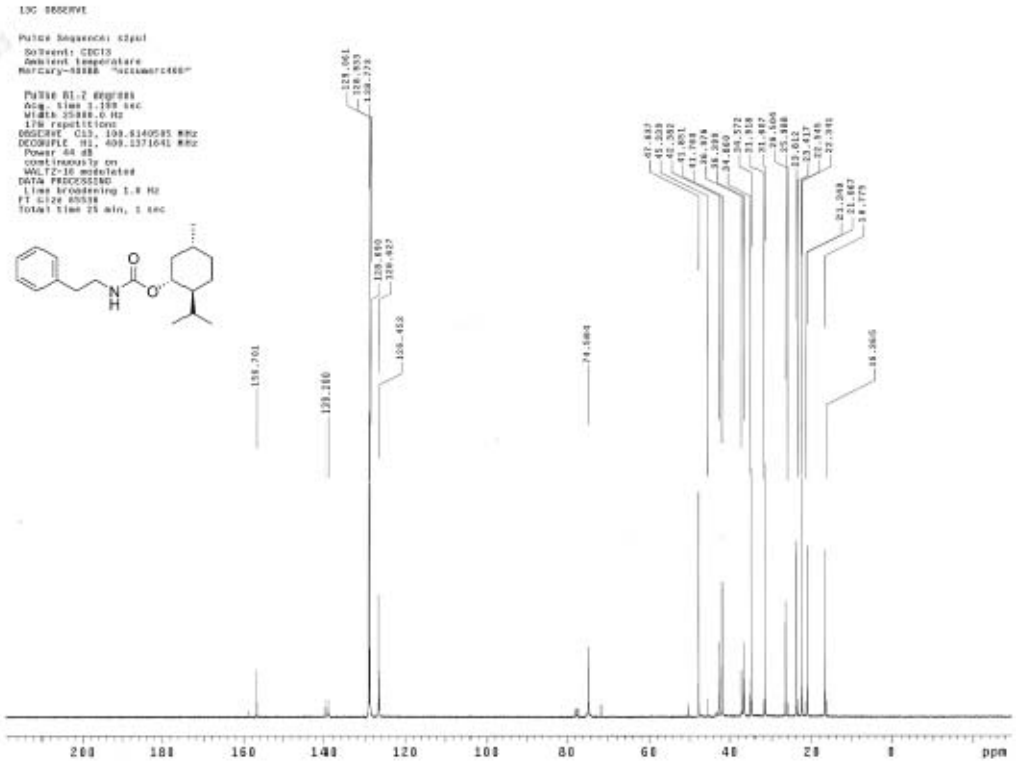

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Pulse 33.3 degrees
Acq. time 1.835 sec
Width 18761.7 Hz
272 repetitions
OBSERVE C13, 75.4580258 MHz
DECOUPLE H1, 300.0953367 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

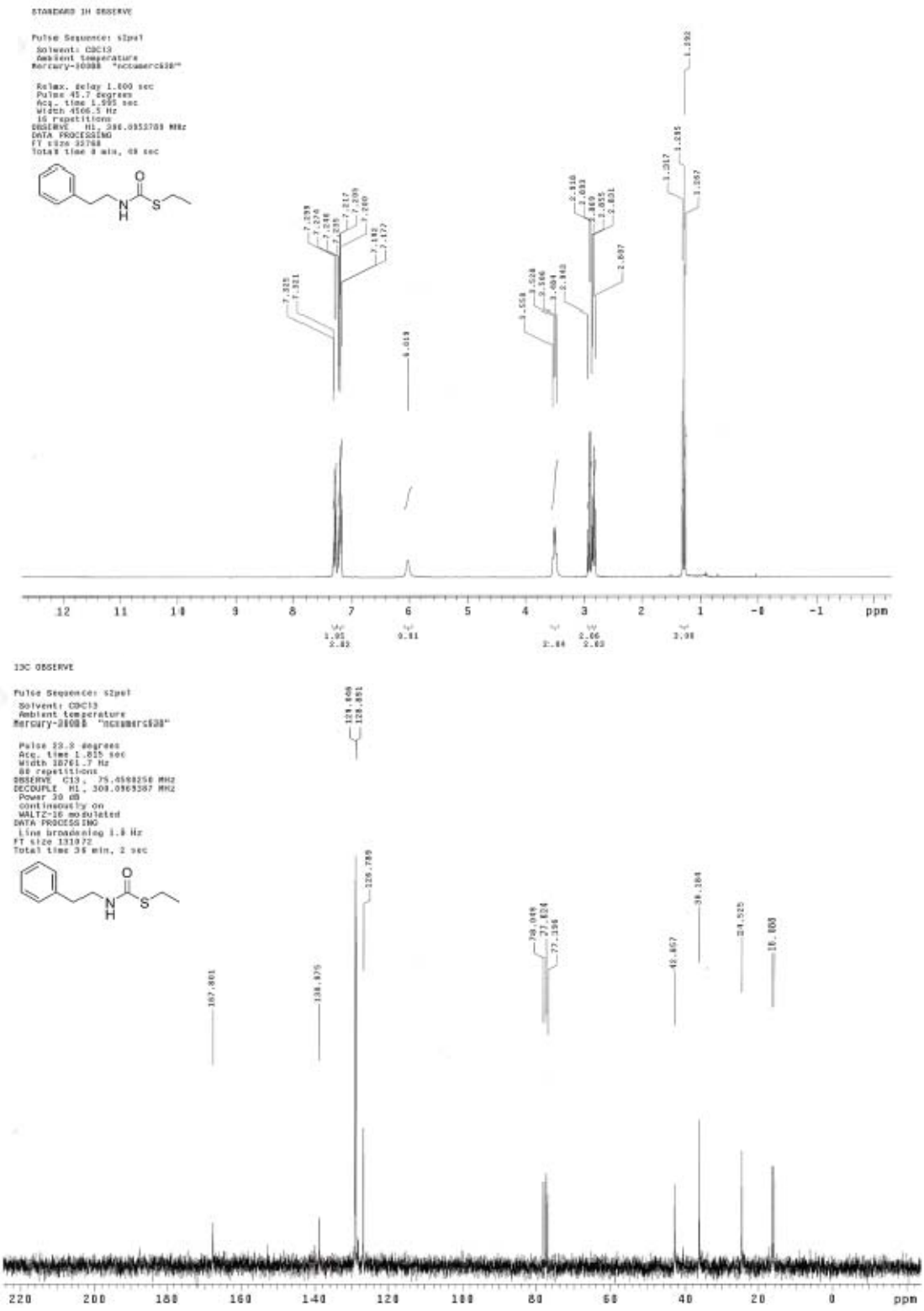


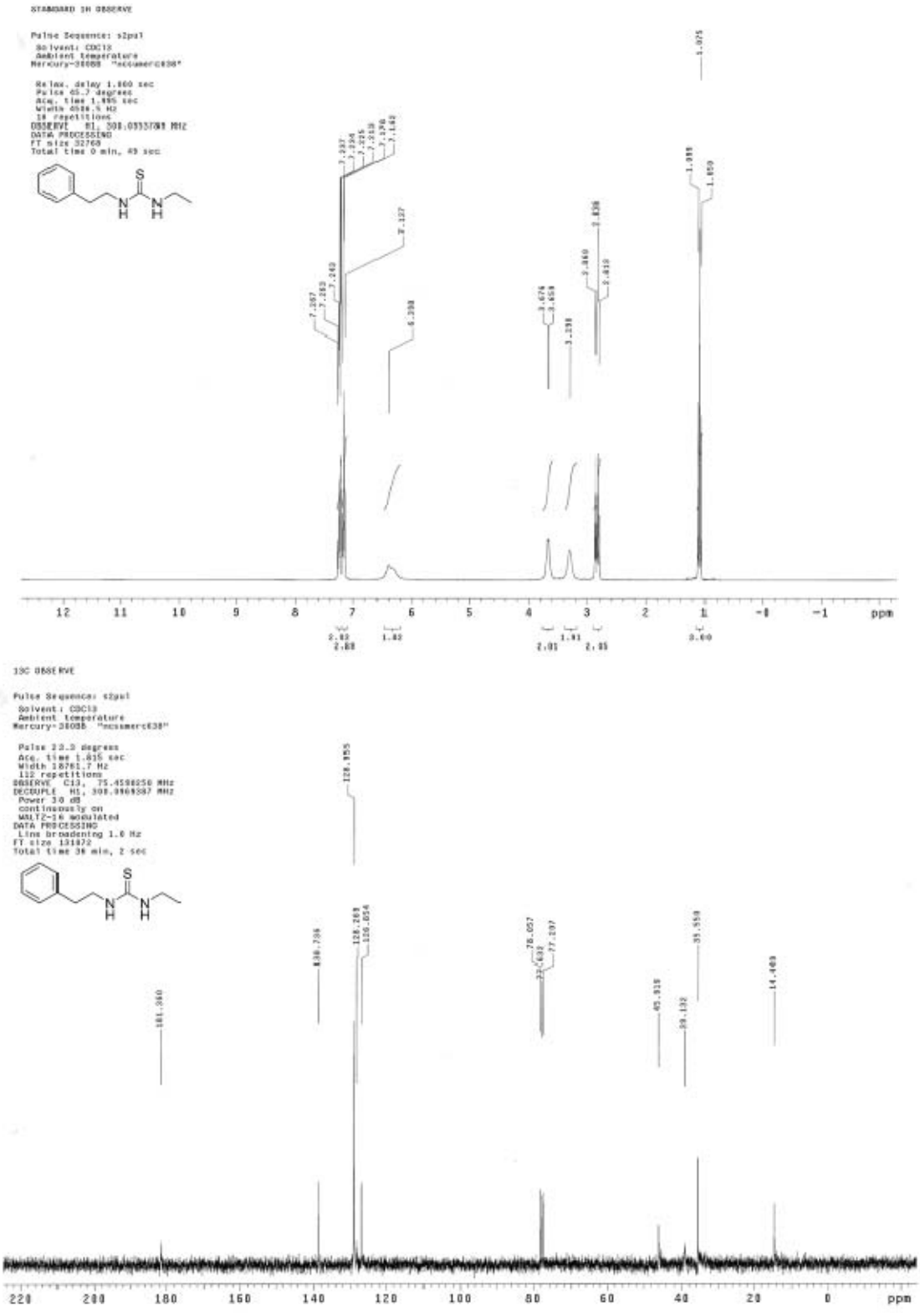

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsumerc400"
Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 1.993 sec
Width 4006.0 Hz
16 repetitions
OBSERVE H1, 400.1252059 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 50 sec
ppm

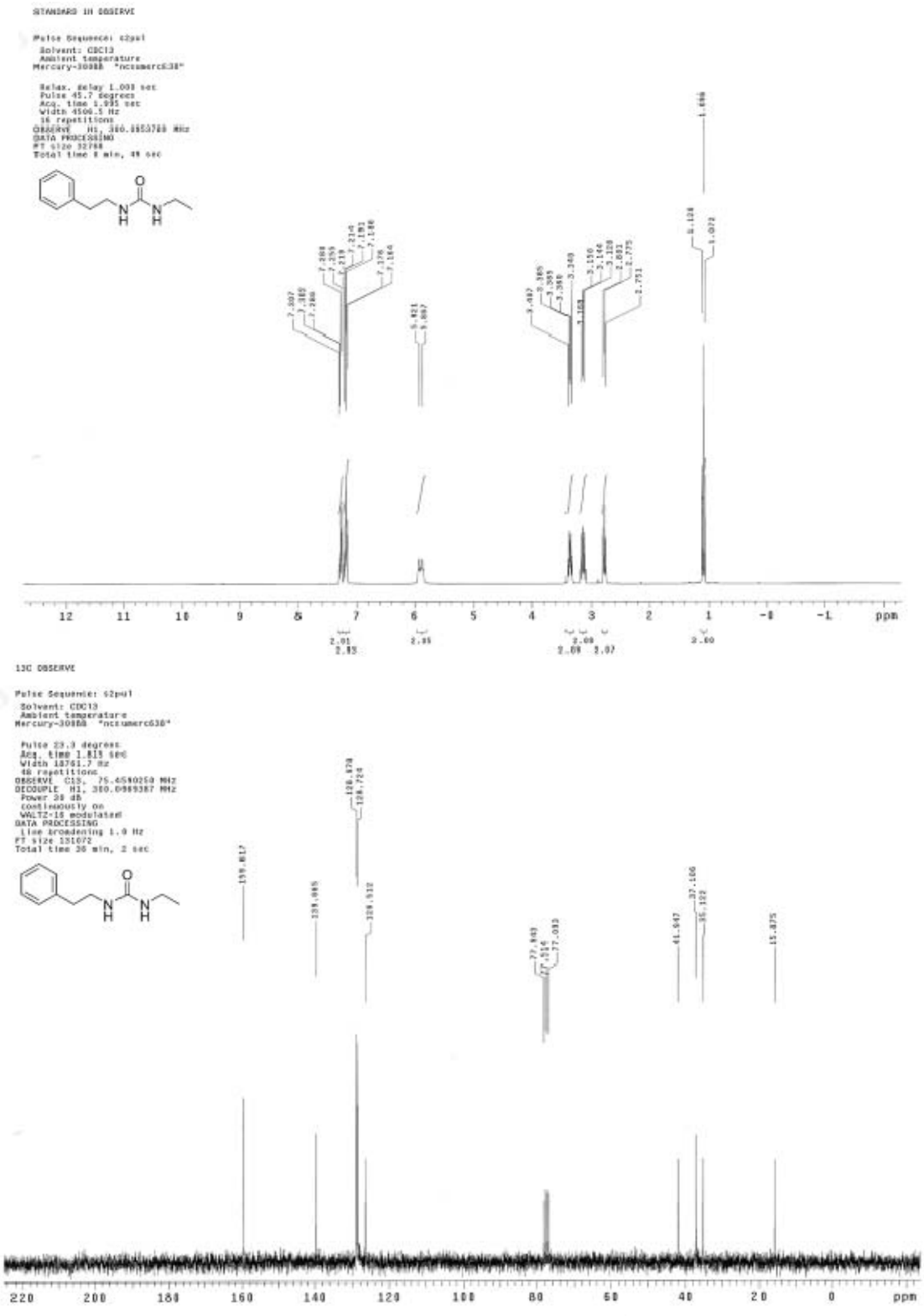

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsumerc400"
Pulse 81.2 degrees
Acq. time 1.199 sec
Width 25038.0 Hz
1024 repetitions
OBSERVE C13, 100.6140595 MHz
DECOUPLE H1, 400.1371641 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 25 min, 1 sec
ppm

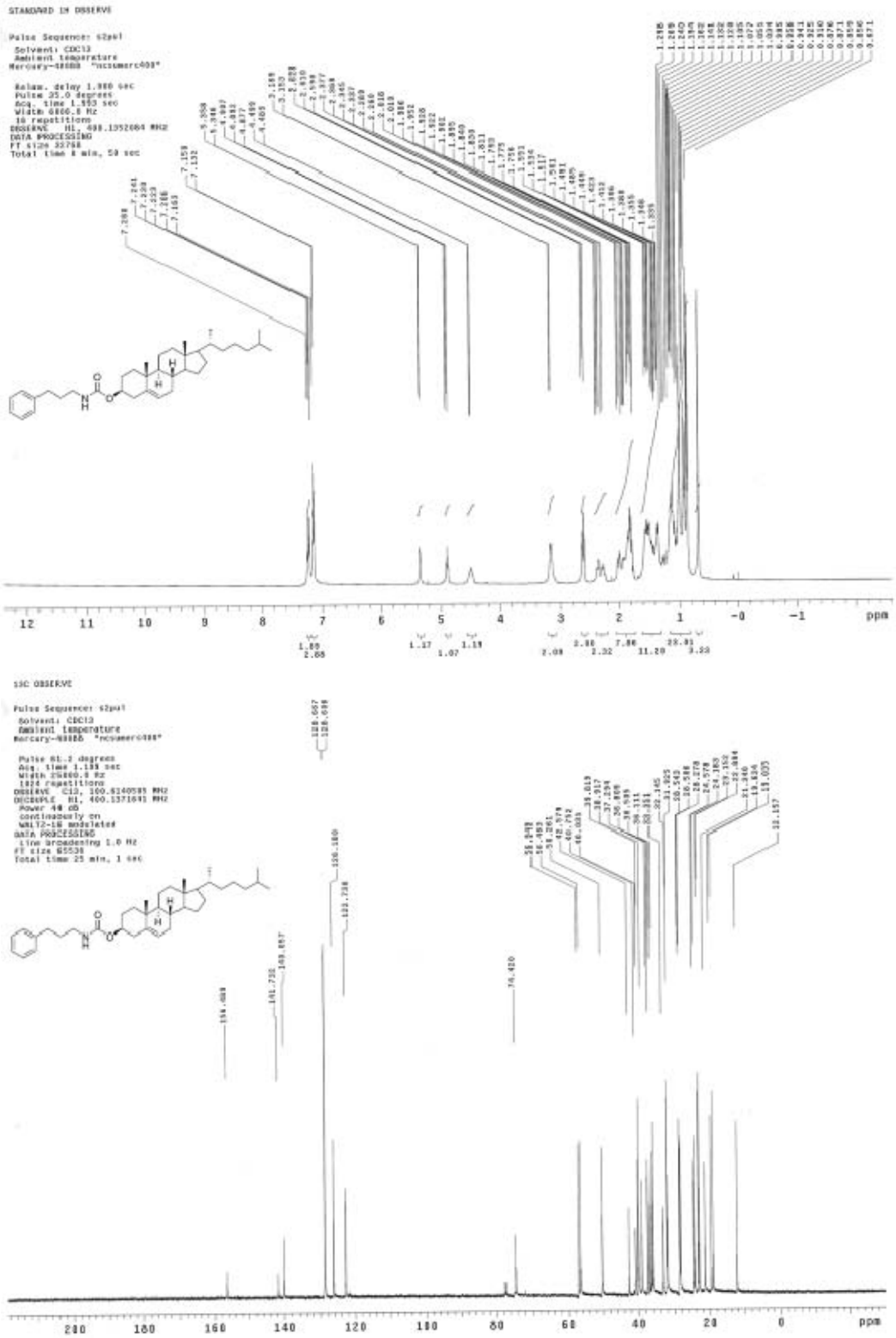


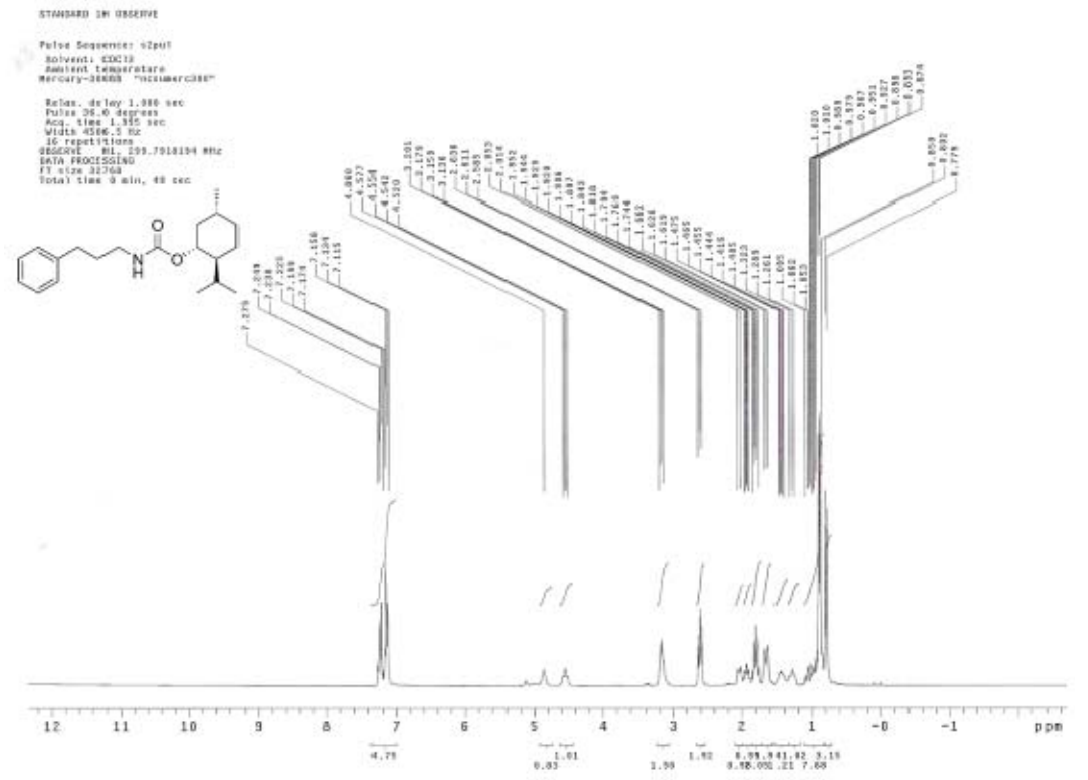


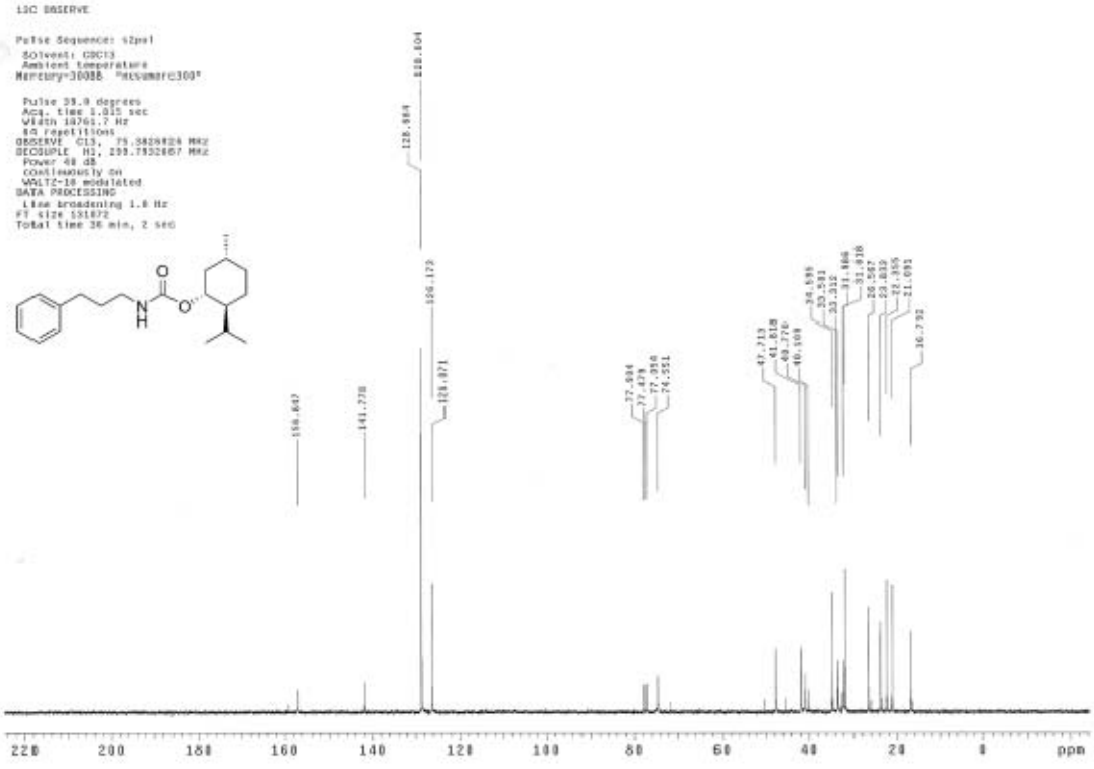


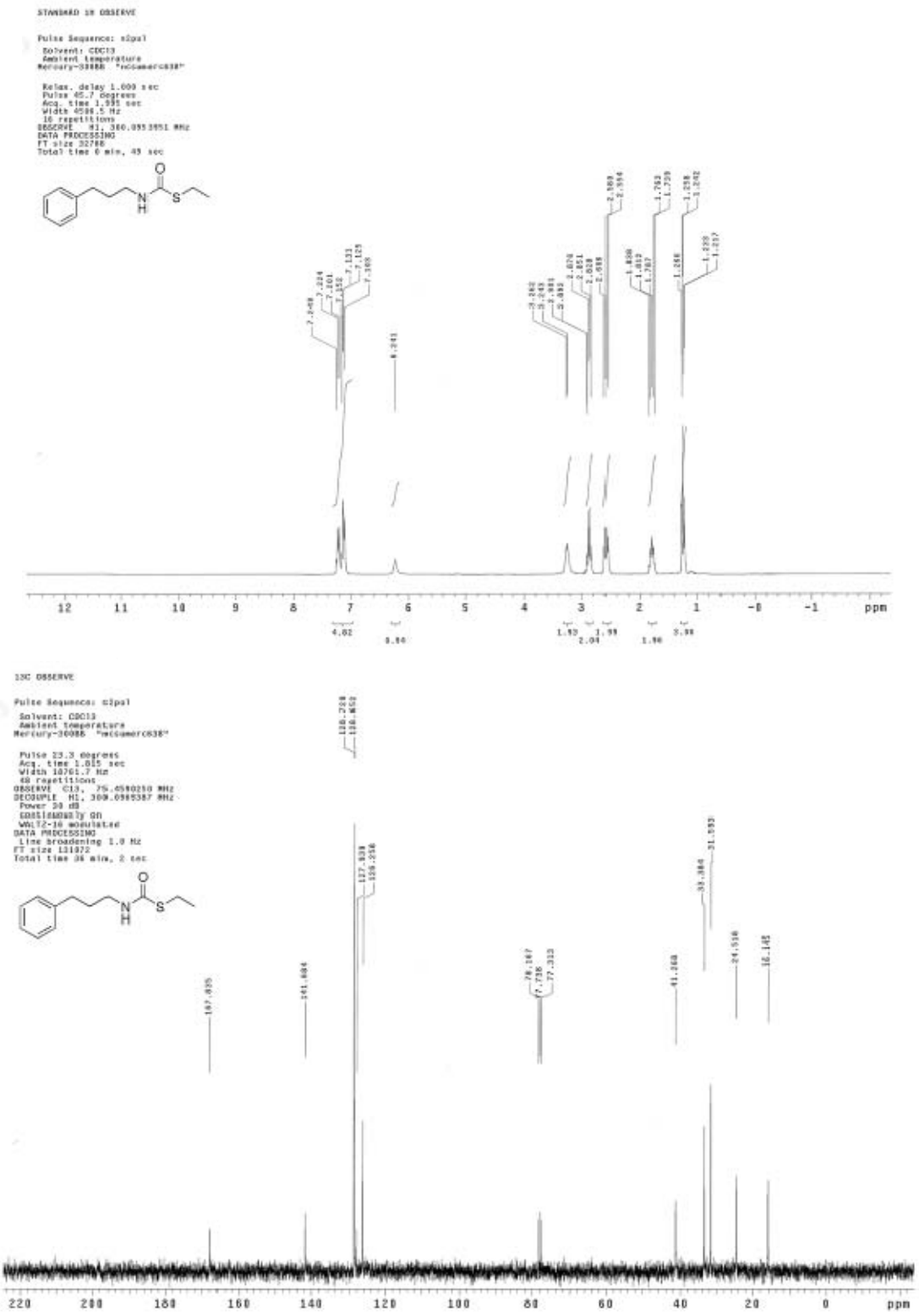


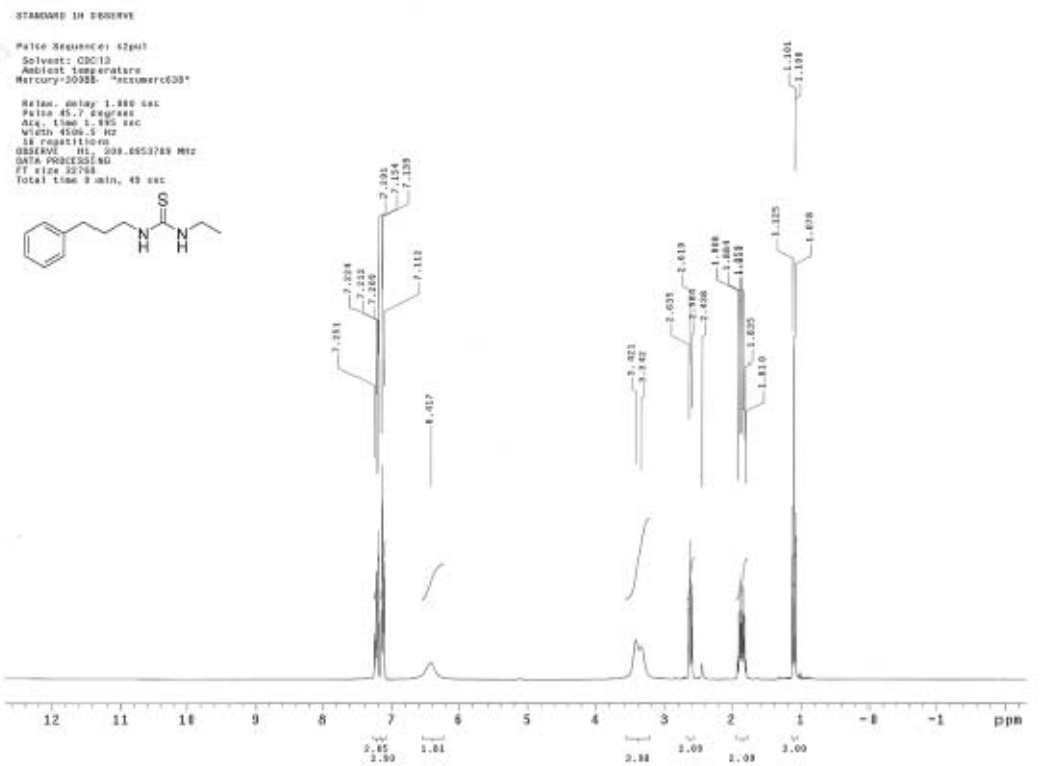

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.895 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 300.0853788 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
48 repetitions
OBSERVE C13, 75.4580250 MHz
DECOUPLE H1, 300.0989387 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
159.817
139.985
129.279
128.724
128.532
77.843
77.516
77.093
41.647
37.166
35.172
15.475
220 200 180 160 140 120 100 80 60 40 20 0 ppm

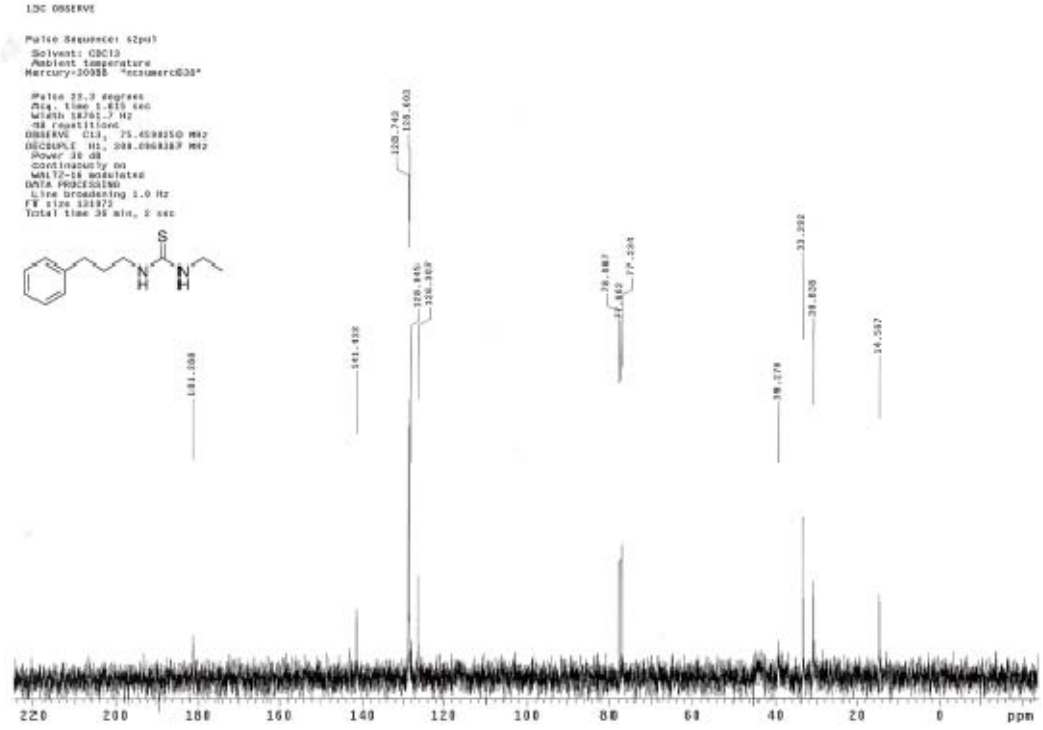


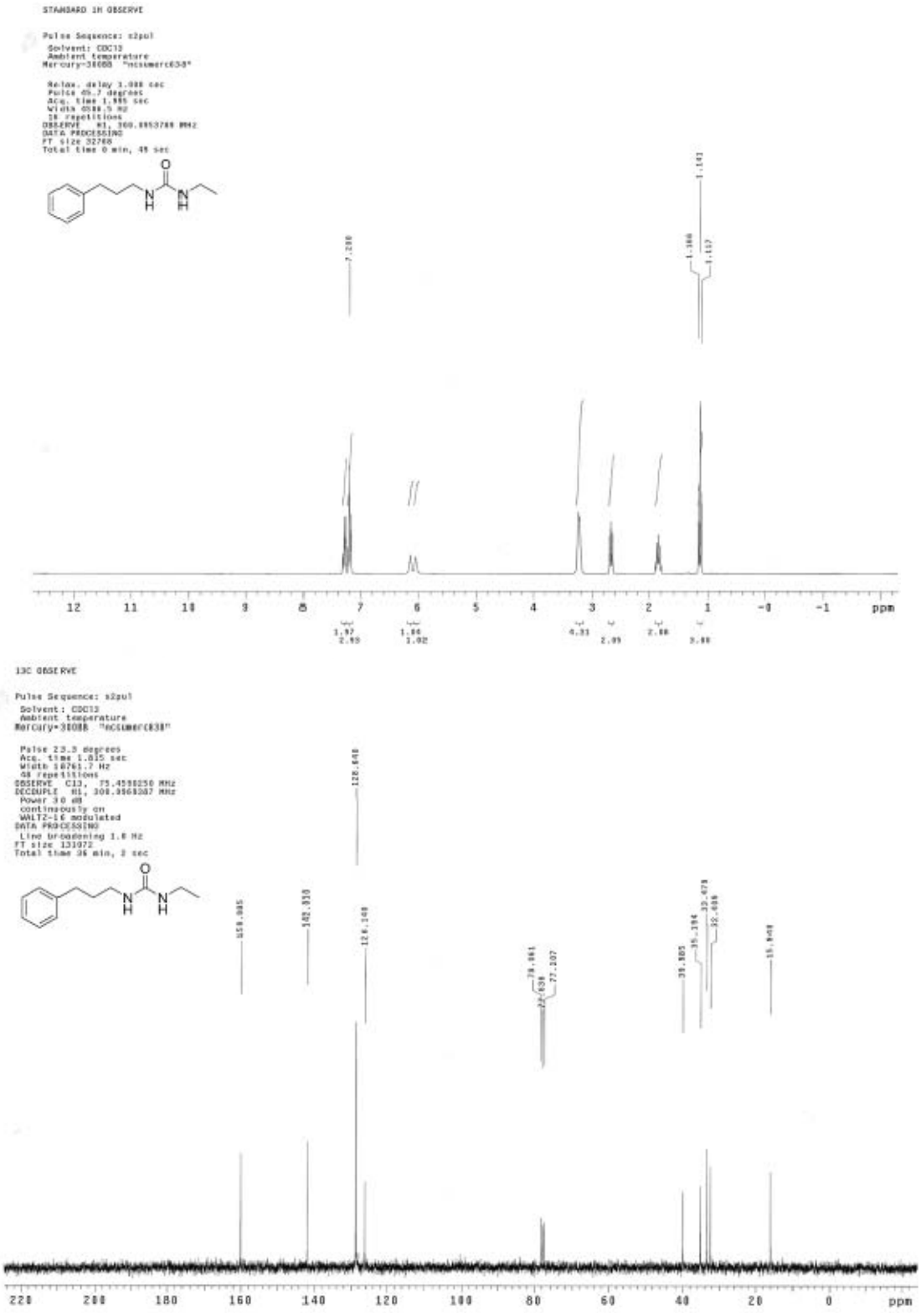


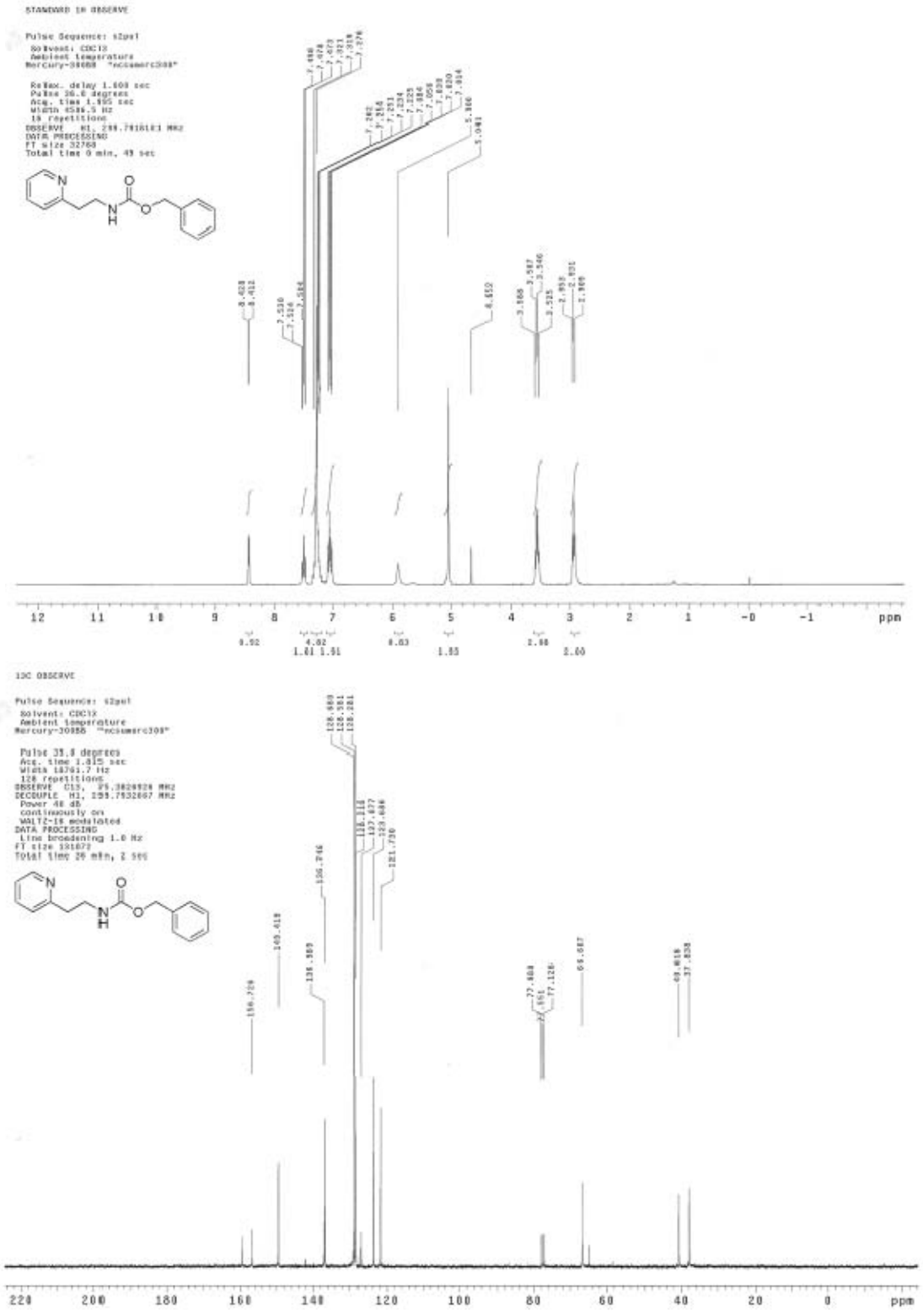


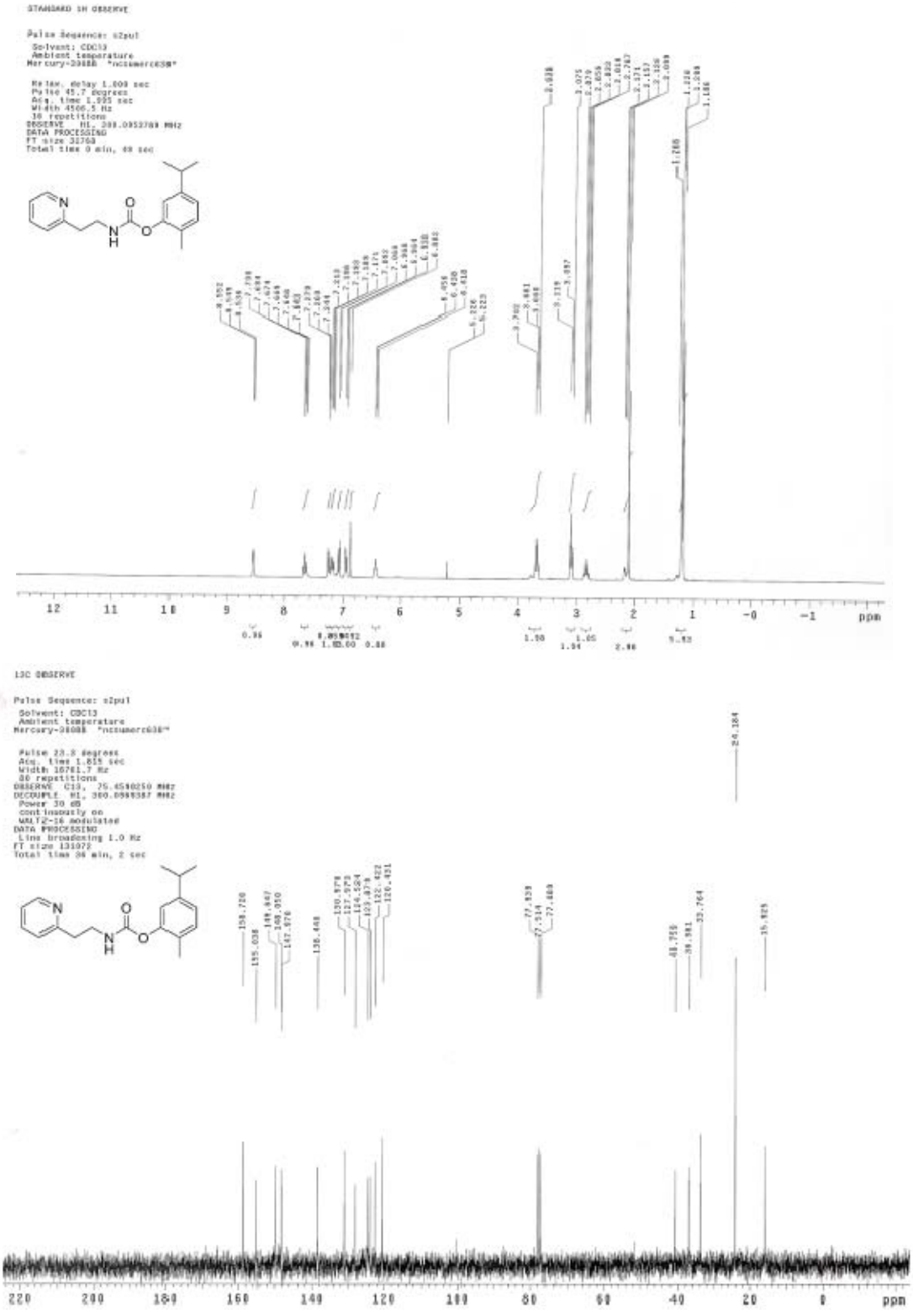


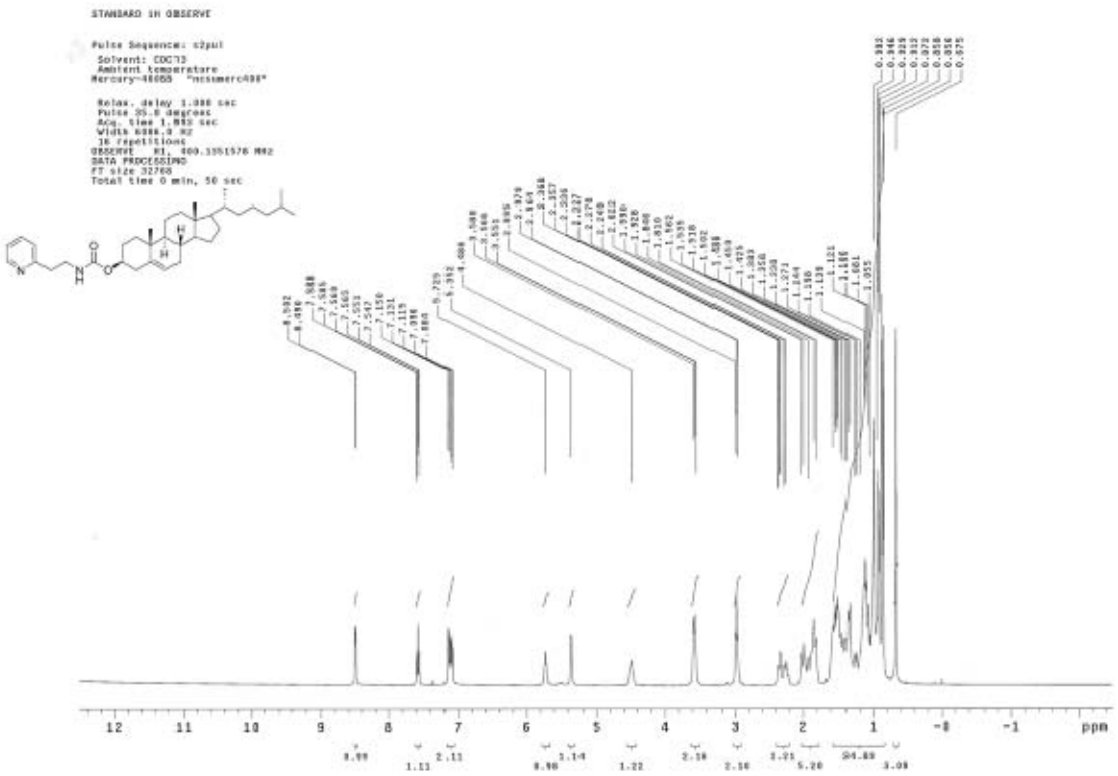


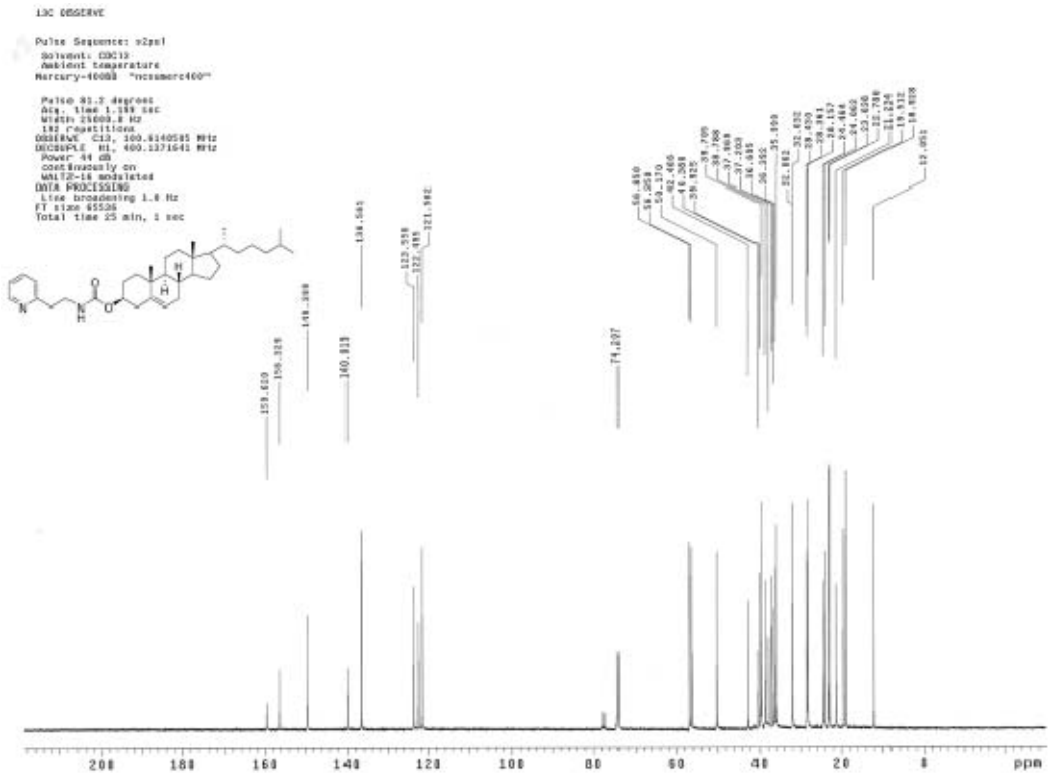


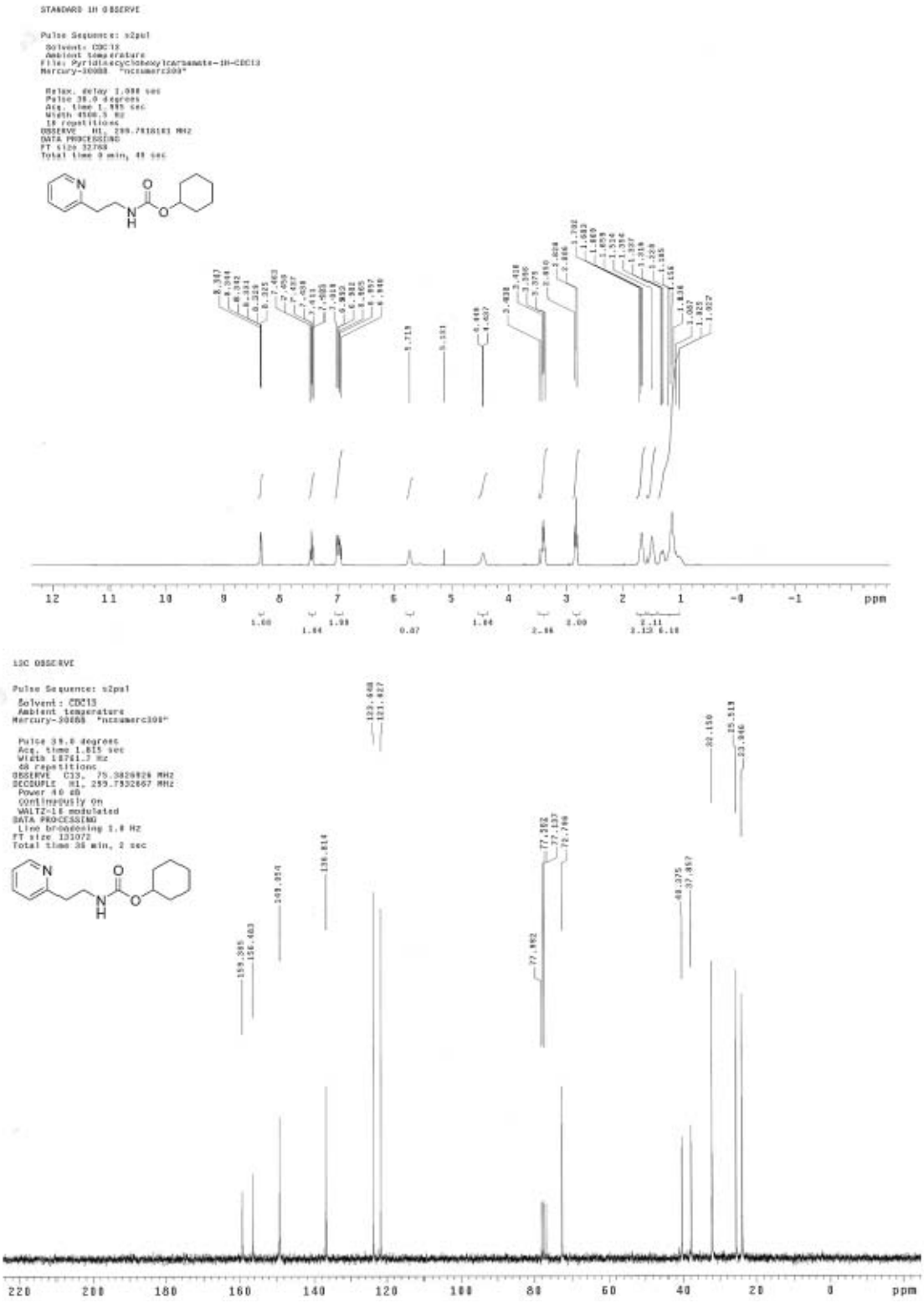

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc634"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 6588.5 Hz
16 repetitions
OBSERVE H1, 300.0053788 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 45 sec
7.288
1.141
1.366
1.117
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.92
2.00
1.84
1.92
4.33
2.09
2.08
3.00
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
48 repetitions
OBSERVE C13, 75.4550250 MHz
DECOUPLE H1, 300.0060307 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
159.885
142.330
126.648
126.148
79.861
77.207
39.805
35.284
33.479
32.496
15.948
220
200
180
160
140
120
100
80
60
40
20
0
ppm

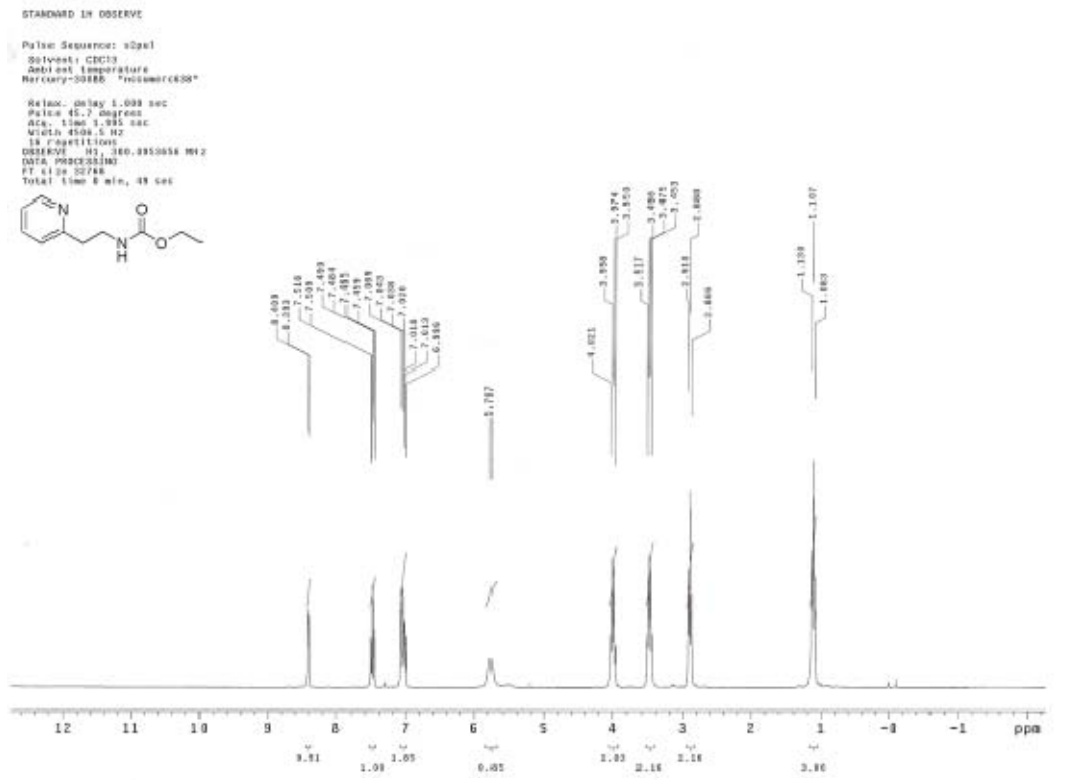


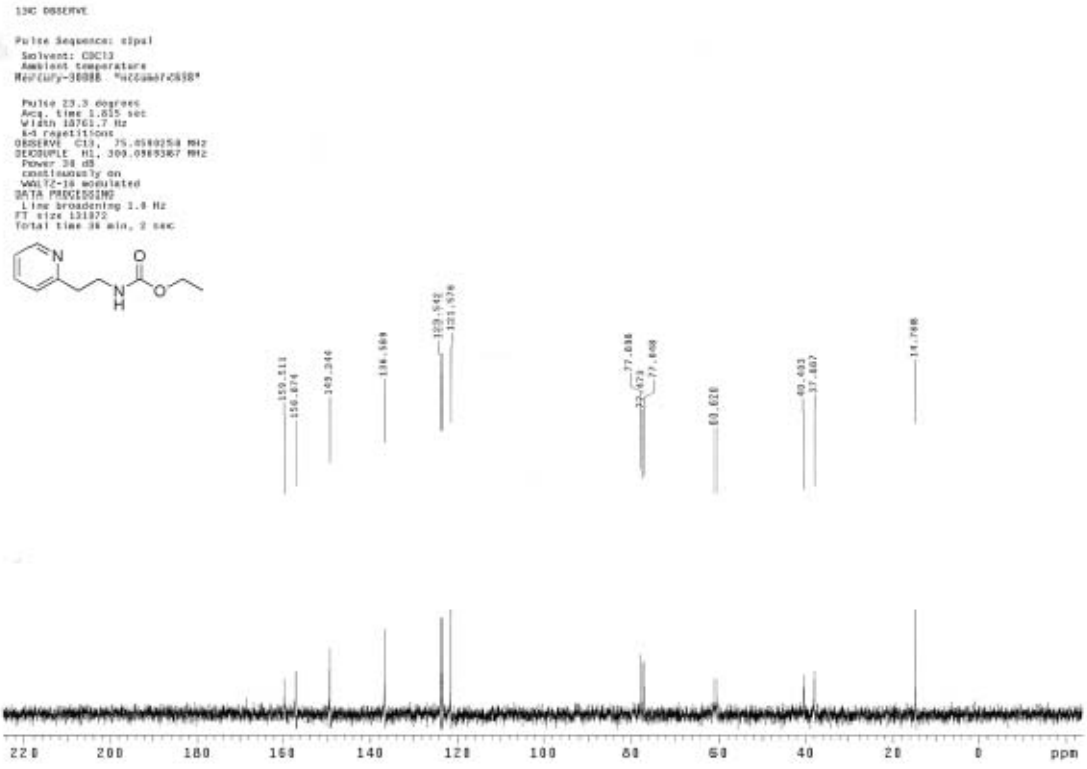


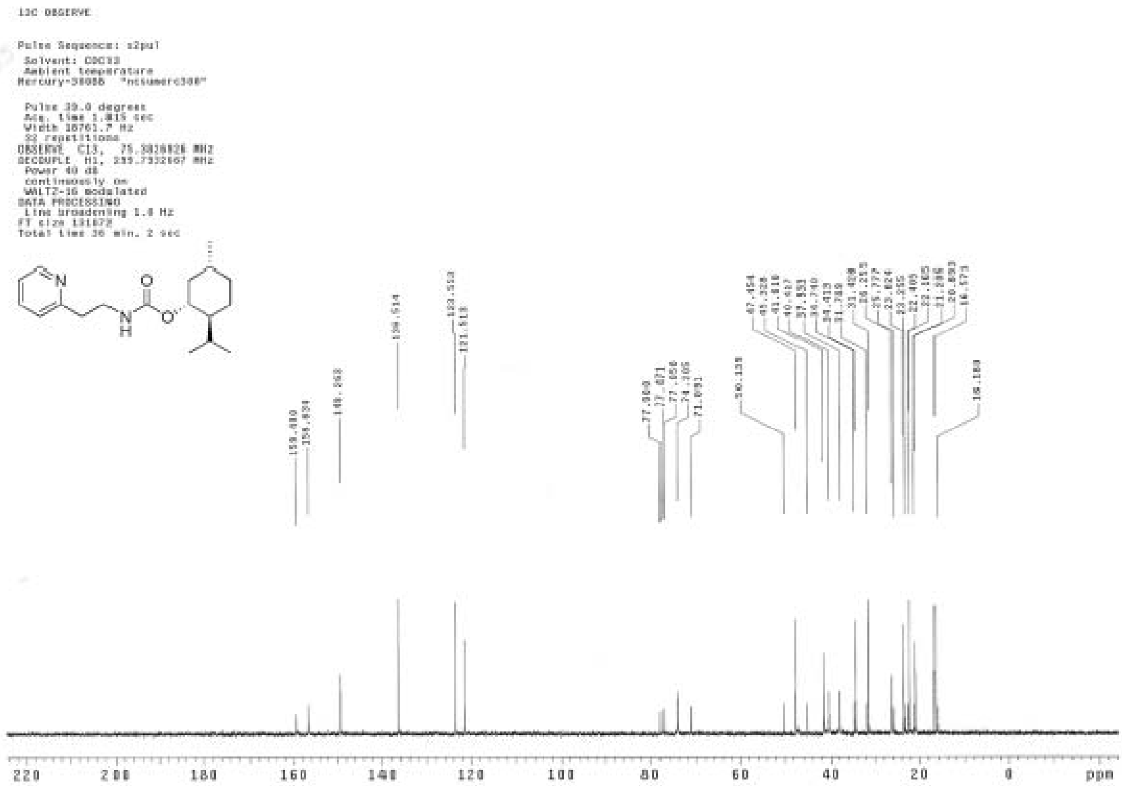

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsmerc400"
Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 1.855 sec
Width 6986.9 Hz
16 repetitions
OBSERVE H1, 400.1351576 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 50 sec
ppm

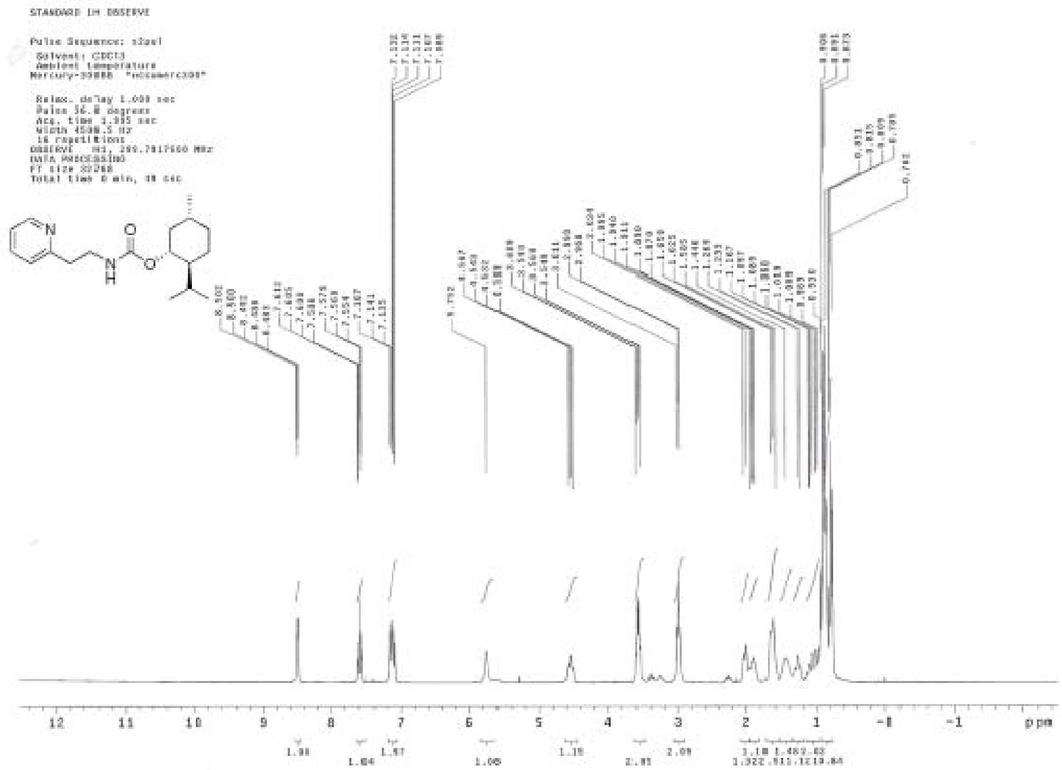

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsmerc400"
Pulse 81.2 degrees
Acq. time 1.188 sec
Width 25000.8 Hz
192 repetitions
OBSERVE C13, 100.6140585 MHz
DECOUPLE H1, 400.1371641 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.8 Hz
FT size 65536
Total time 25 min, 1 sec
ppm

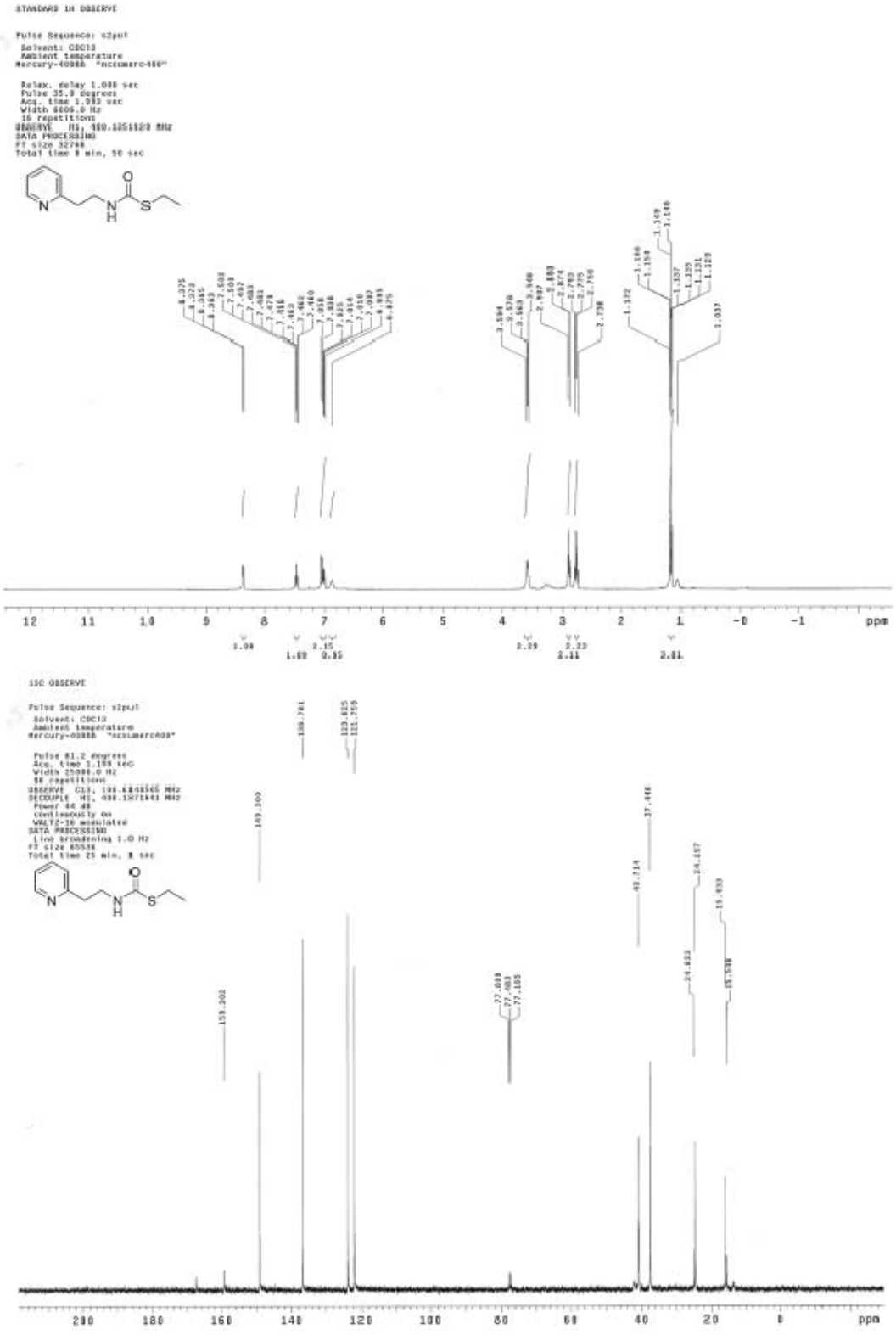

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.995 sec
Width 4508.5 Hz
16 repetitions
OBSERVE H1, 299.7818183 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
ppm
1.00
1.84
1.98
0.87
1.84
2.98
2.00
2.11
2.13
5.18
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Pulse 30.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
68 repetitions
OBSERVE C13, 75.3828926 MHz
DECOUPLE H1, 299.7932667 MHz
Power 40 dB
continously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
159.385
156.483
149.854
136.814
123.648
121.627
77.582
77.157
76.798
77.892
48.275
37.857
32.150
25.519
23.846
ppm

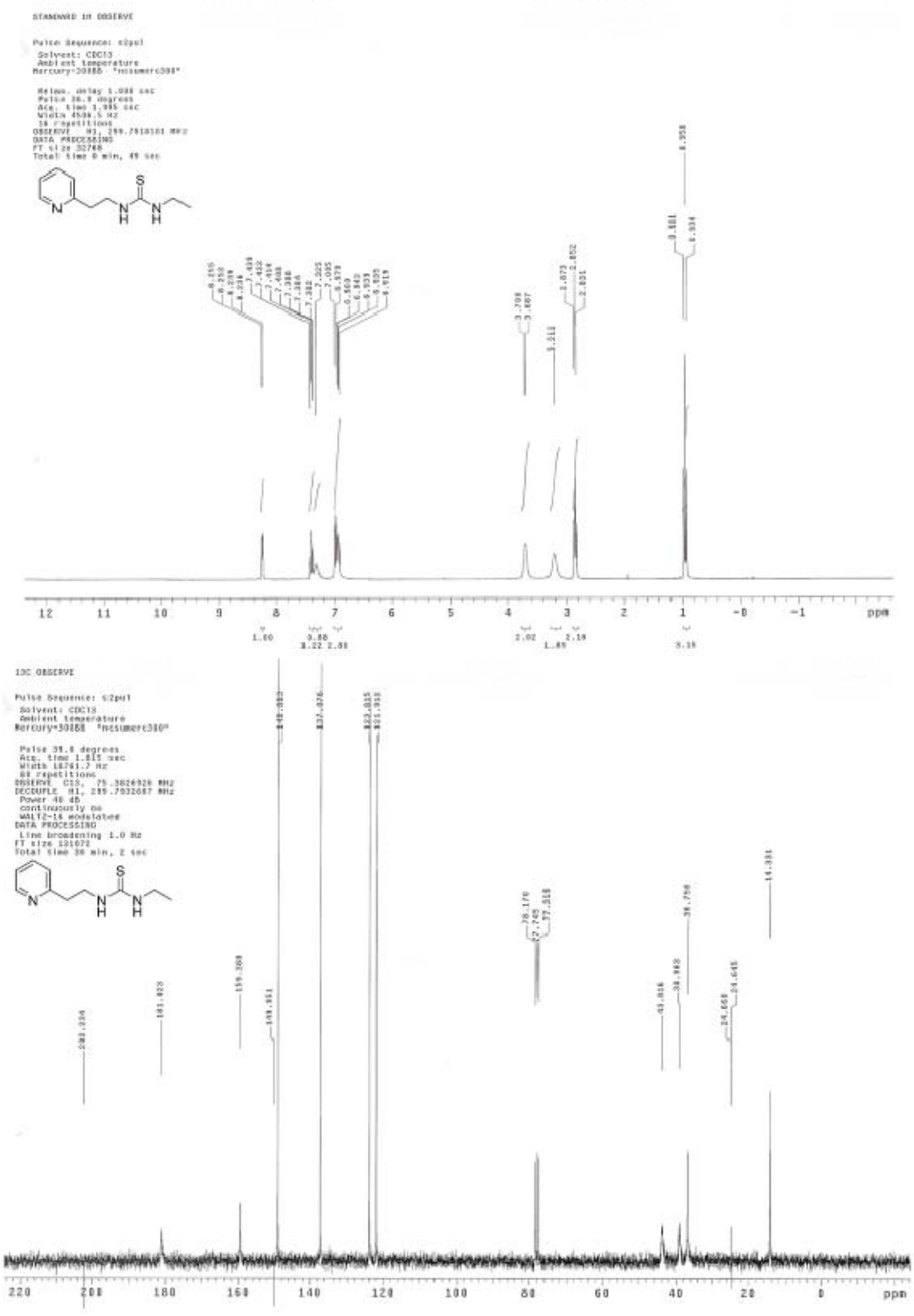

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4504.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
ppm

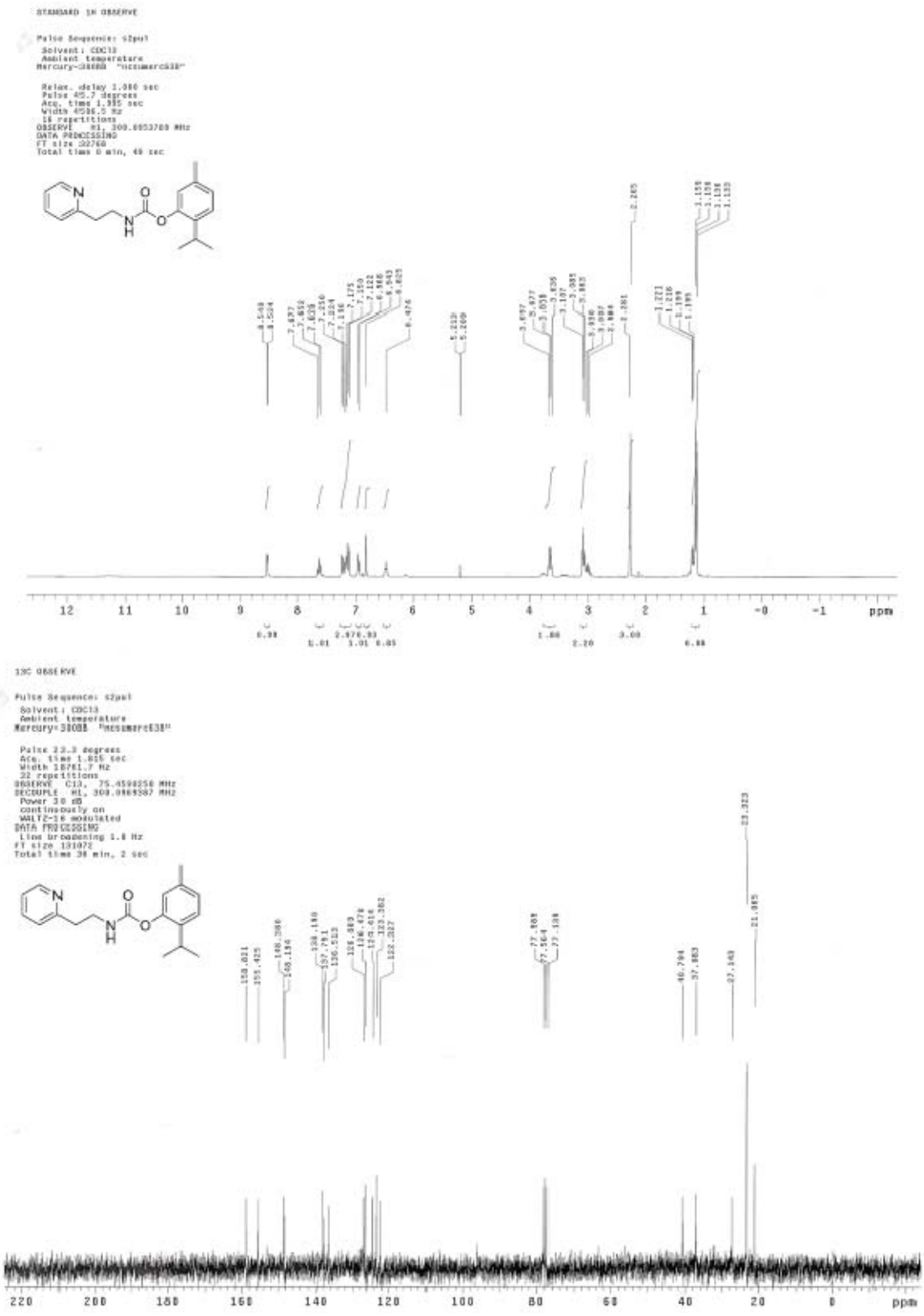

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
64 repetitions
DECOUPLE H1, 399.09893367 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 38 min, 2 sec
ppm

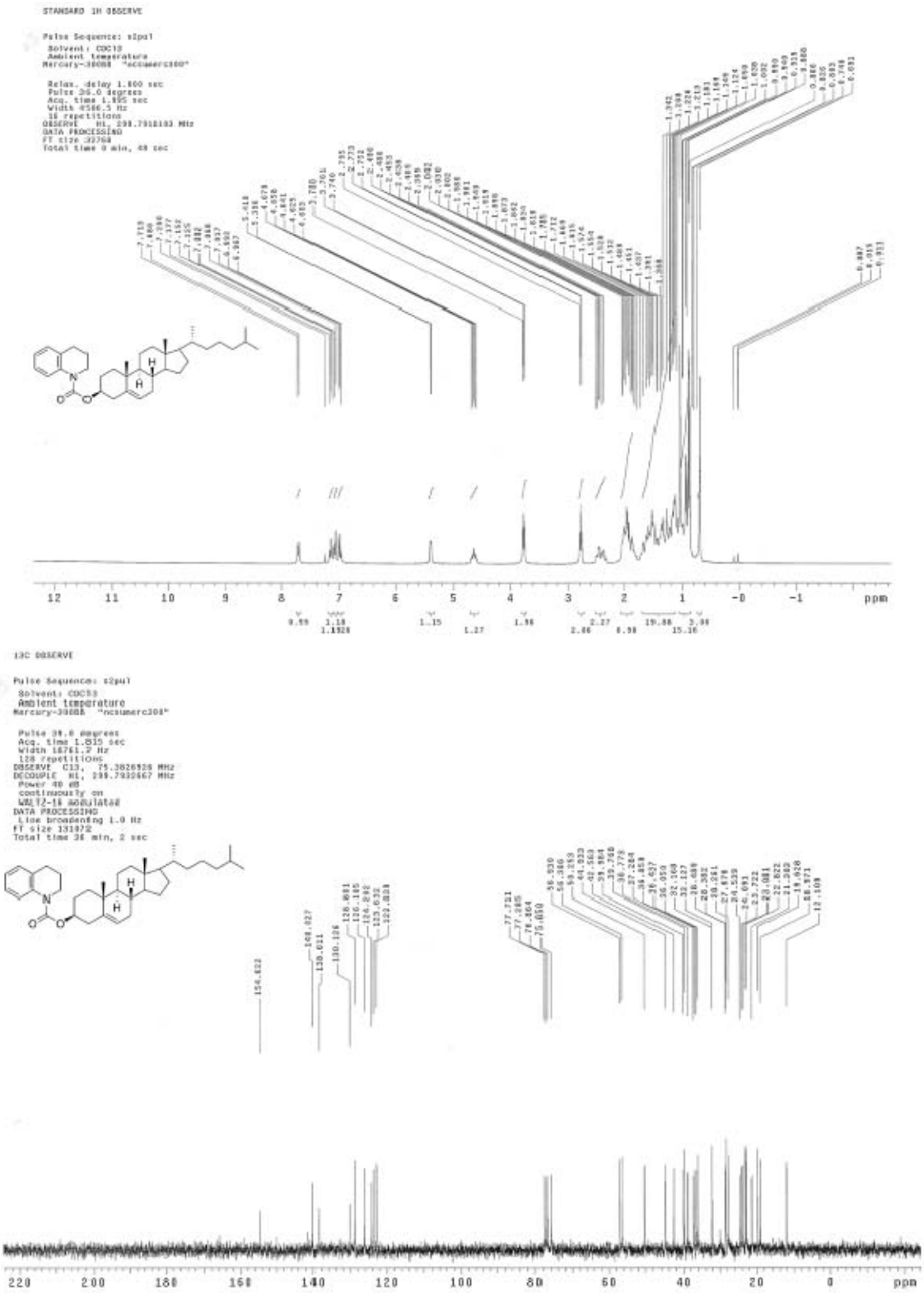


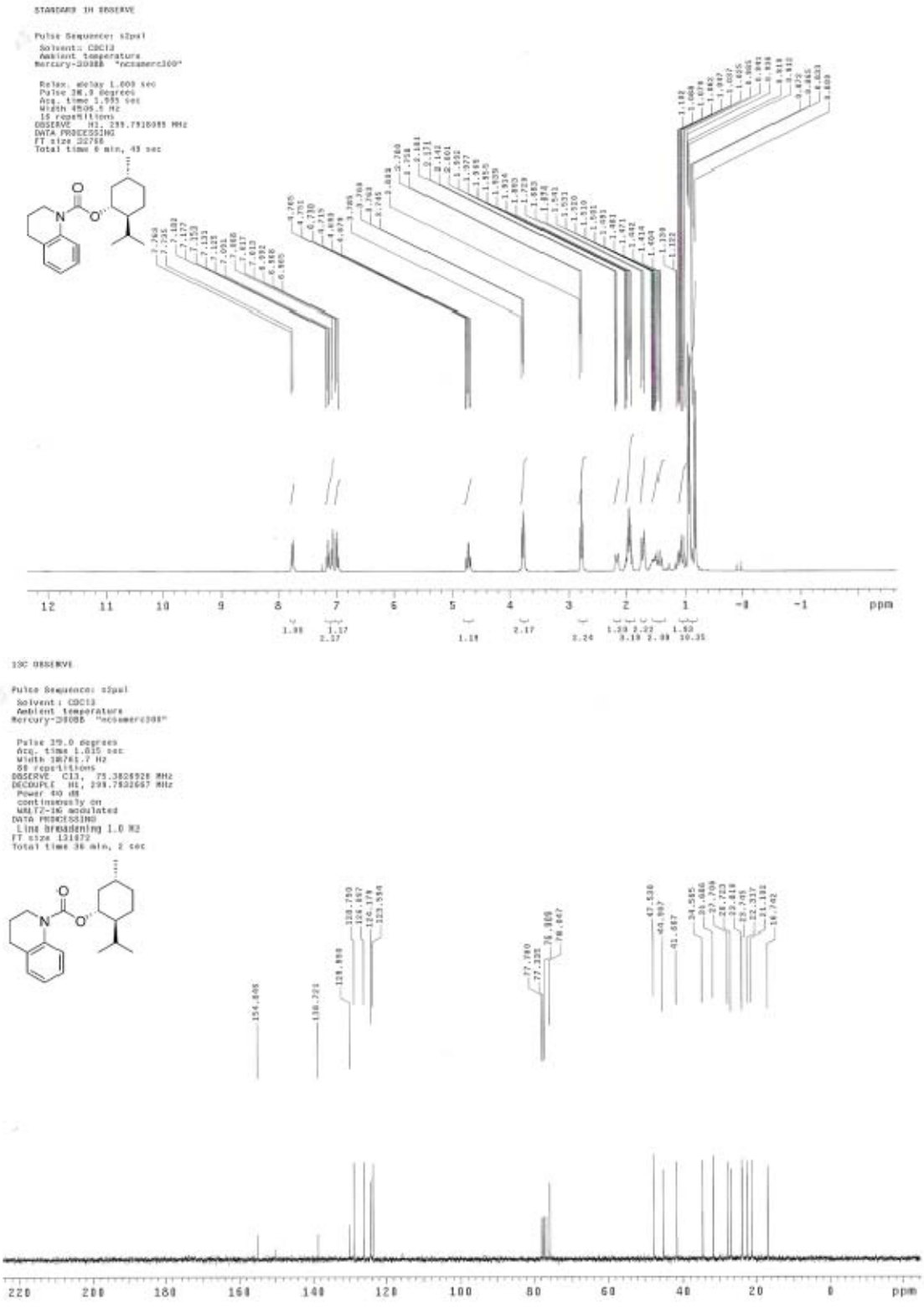


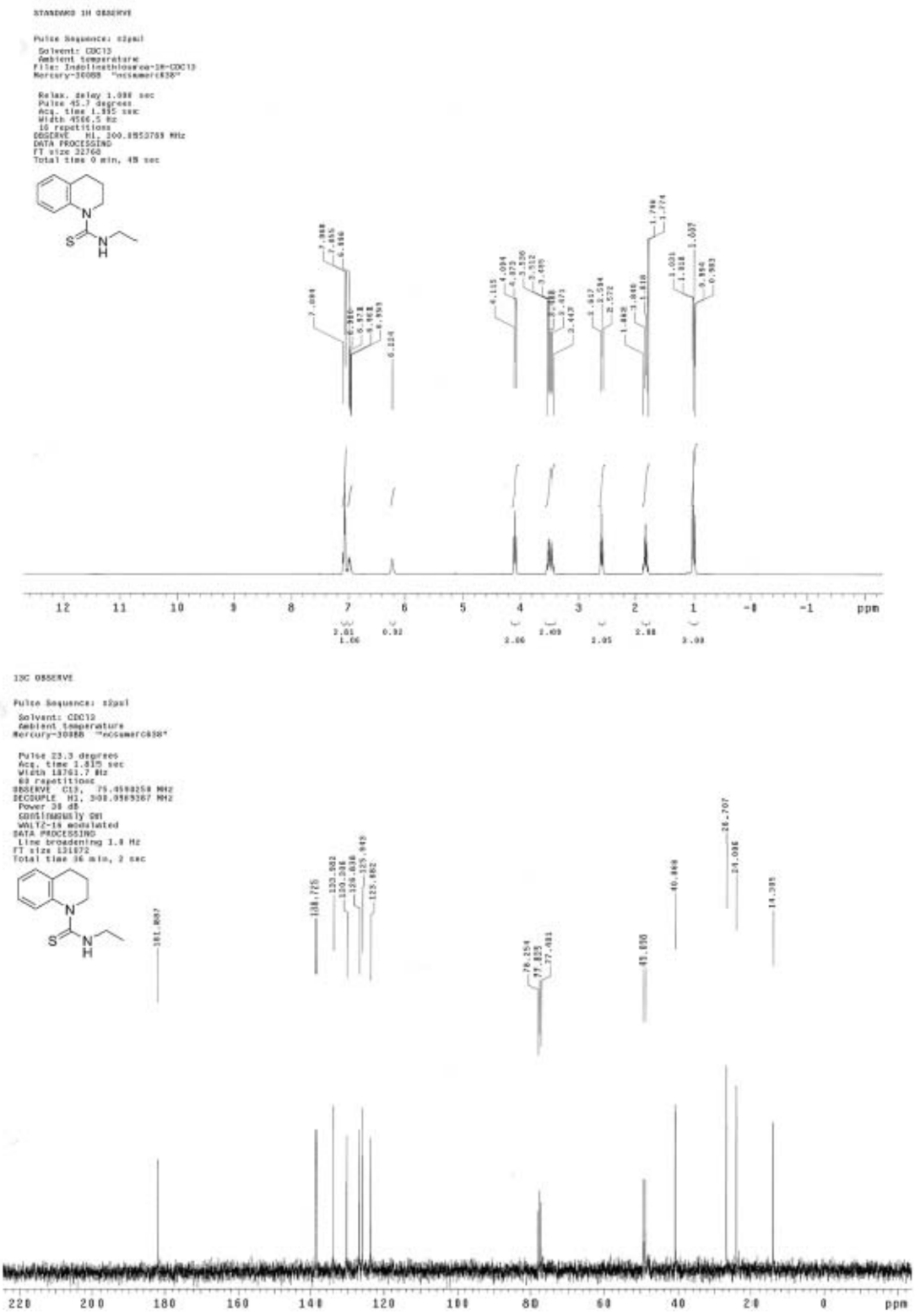


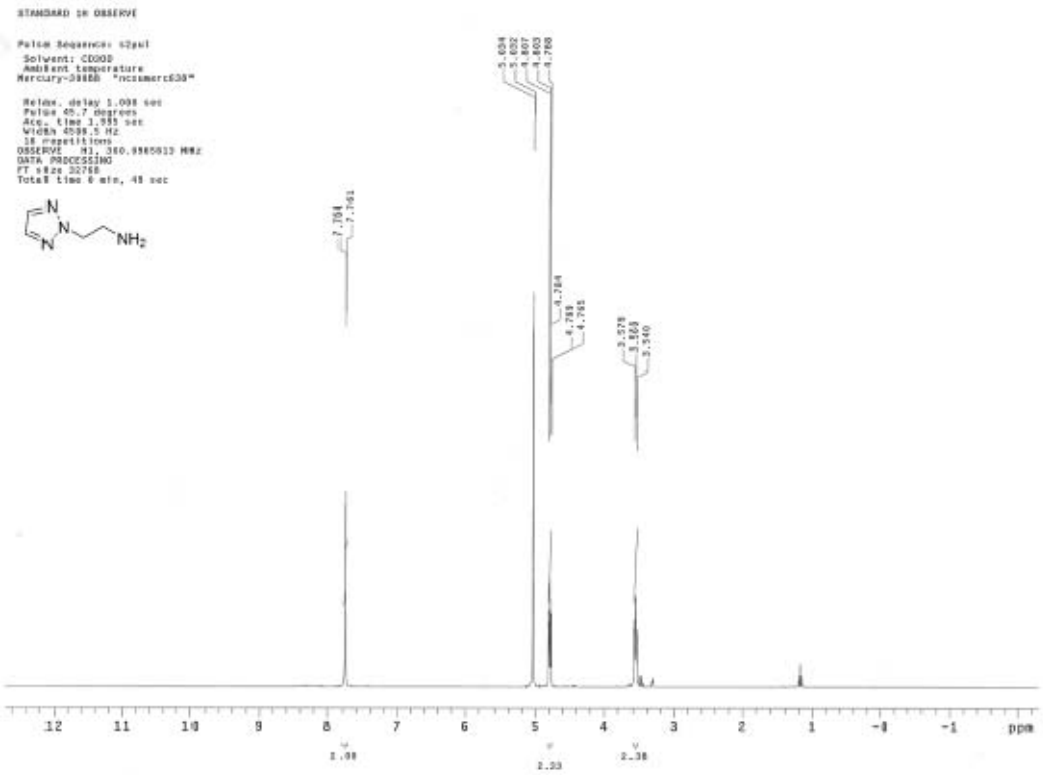


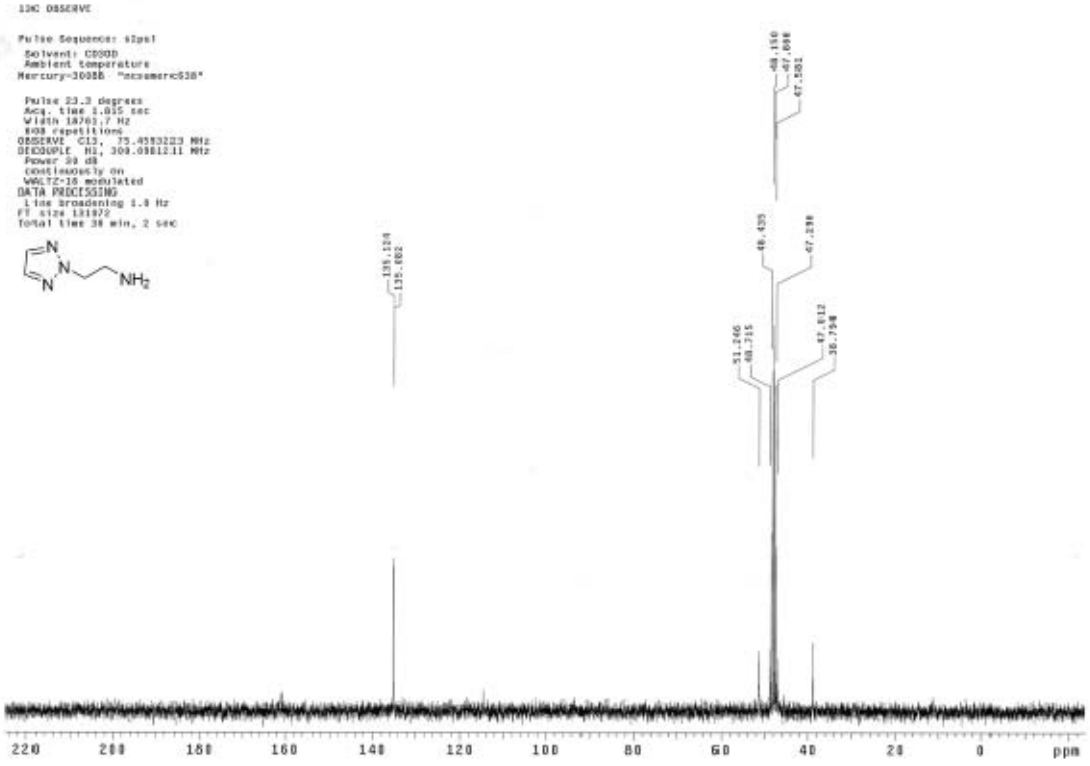


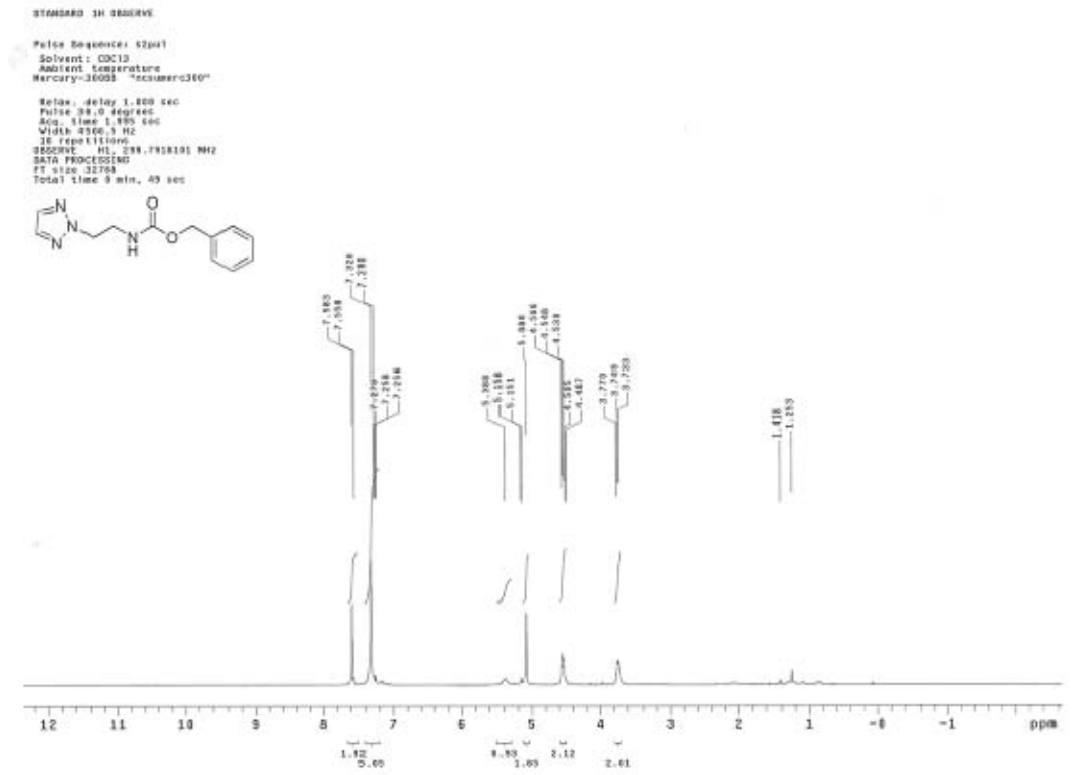

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Relax. delay 1.000 sec
Pulse 26.0 degrees
Acq. time 1.895 sec
Width 4586.5 Hz
16 repetitions
OBSERVE H1, 299.7918183 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Pulse 30.0 degrees
Acq. time 1.815 sec
Width 18761.2 Hz
128 repetitions
OBSERVE C13, 75.3826928 MHz
DECOUPLE H1, 299.7932667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 38 min, 2 sec
ppm

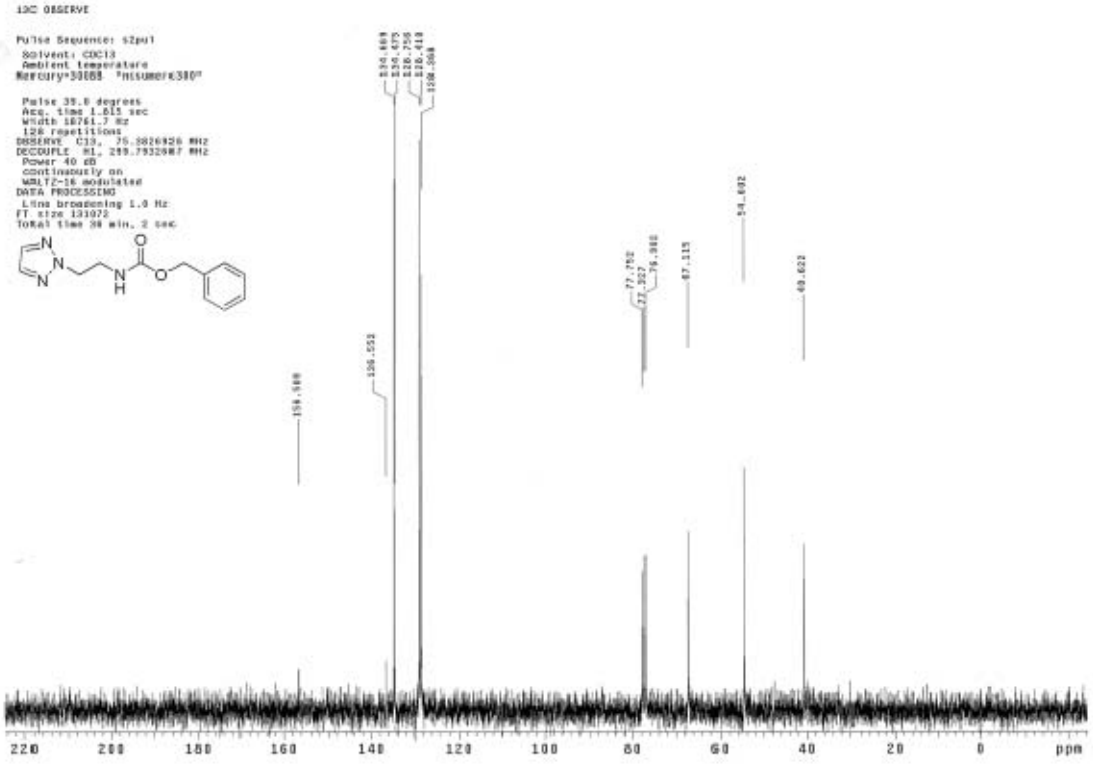


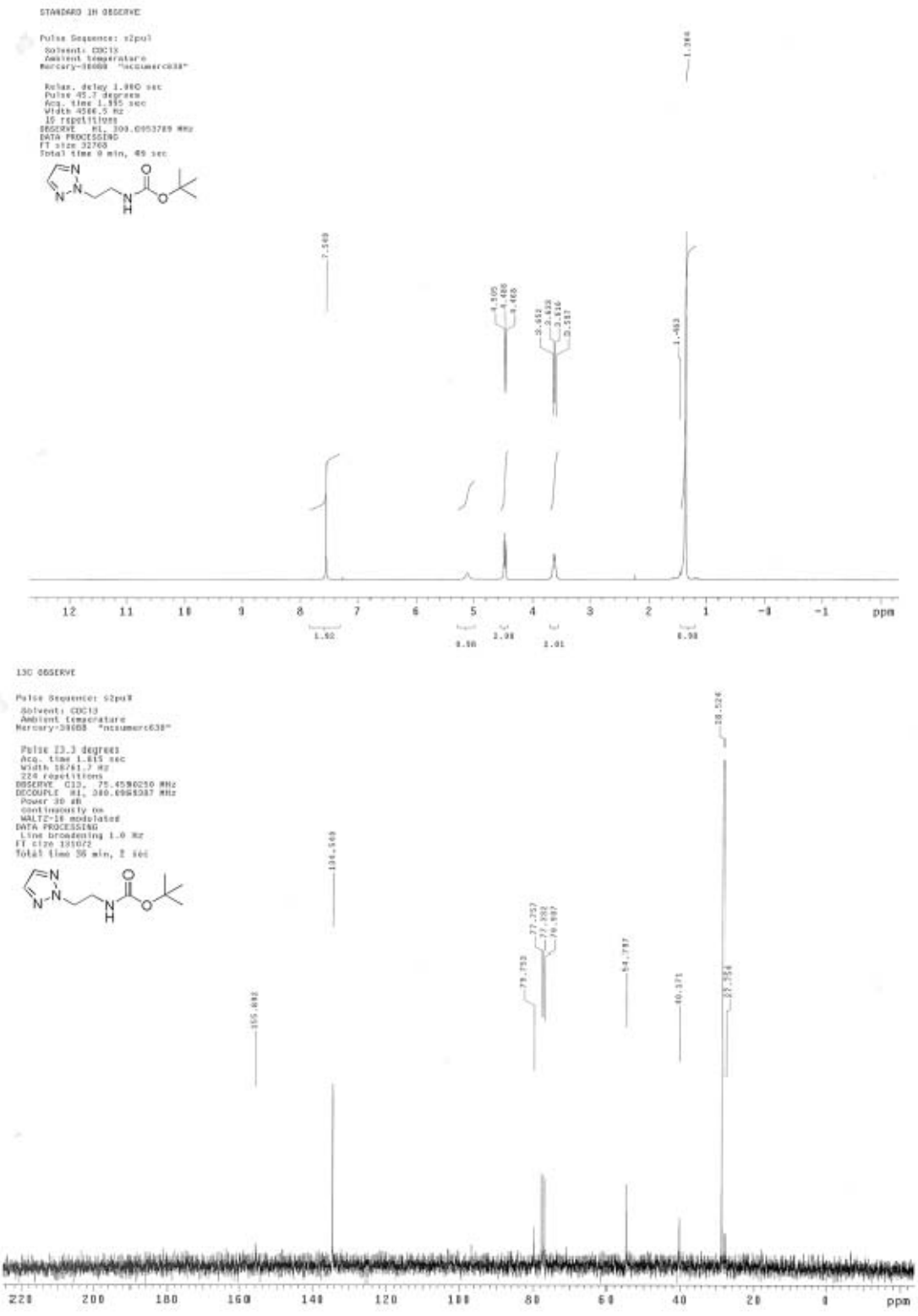


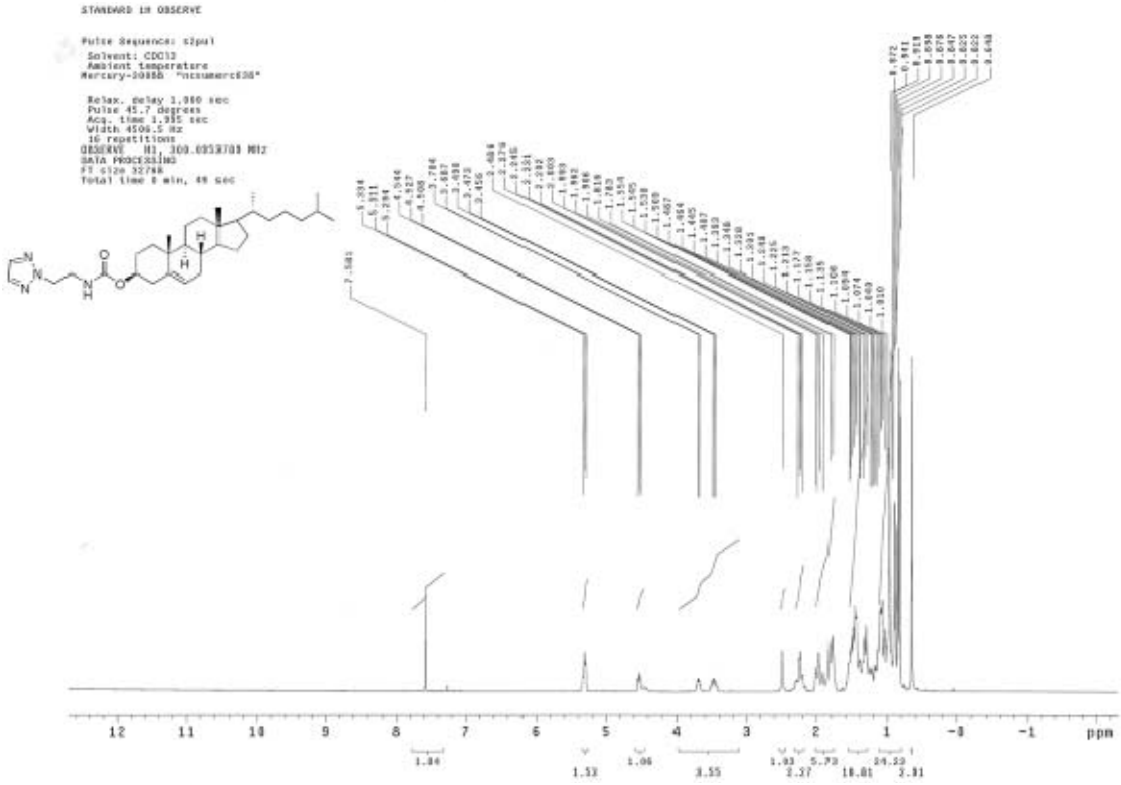

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
Mercury-300BB "ncsumerc638"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.955 sec
Width 4598.5 Hz
16 repetitions
OBSERVE H1, 300.0865013 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
NH2
ppm

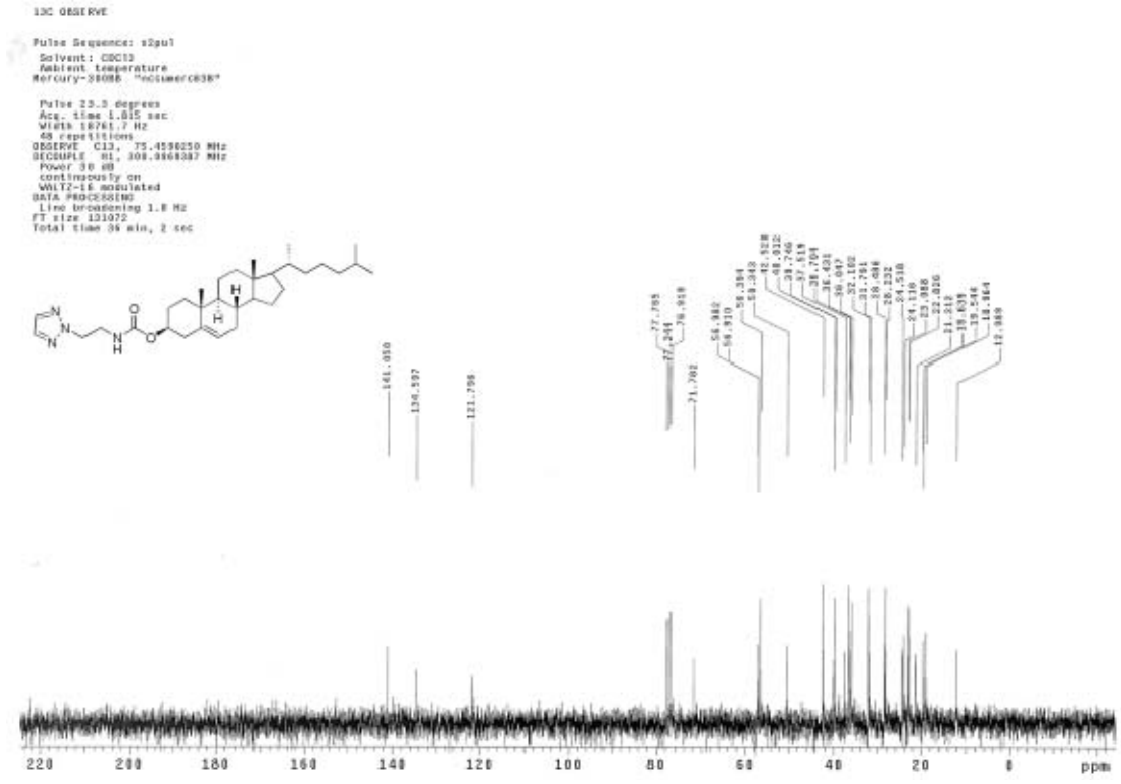

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
Mercury-300BB "ncsumerc638"
Pulse 23.3 degrees
Acq. time 1.015 sec
Width 18761.7 Hz
808 repetitions
OBSERVE C13, 75.4583223 MHz
DECOUPLE H1, 300.0812111 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.8 Hz
FT size 131072
Total time 38 min, 2 sec
NH2
ppm

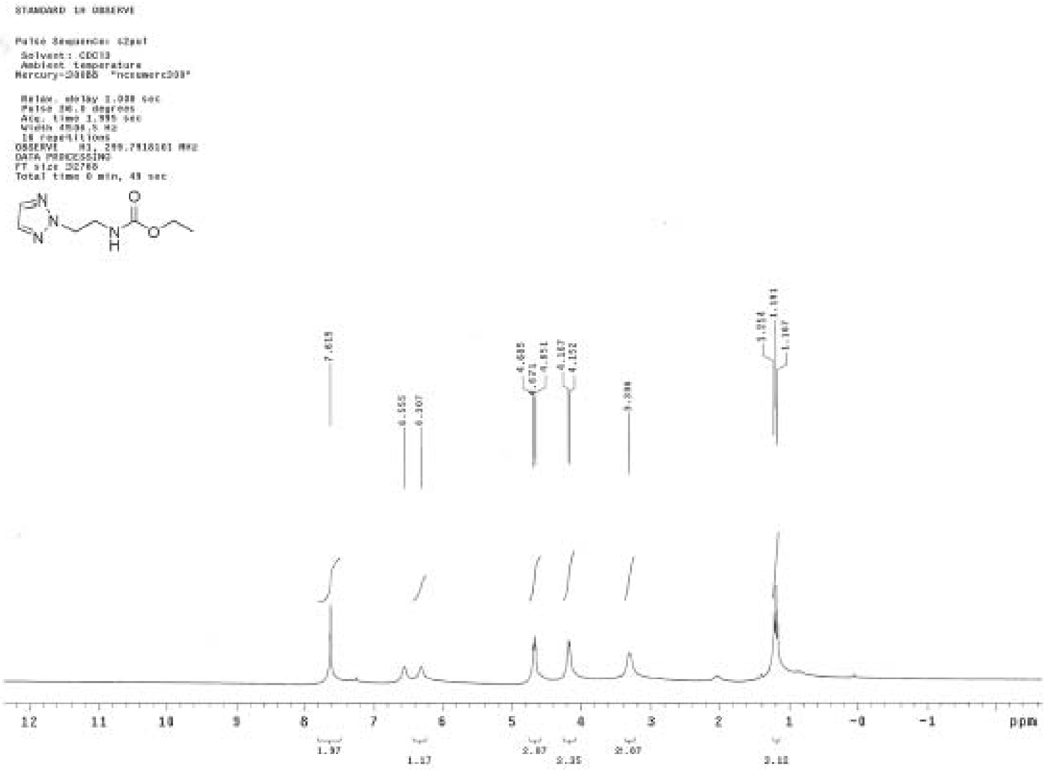


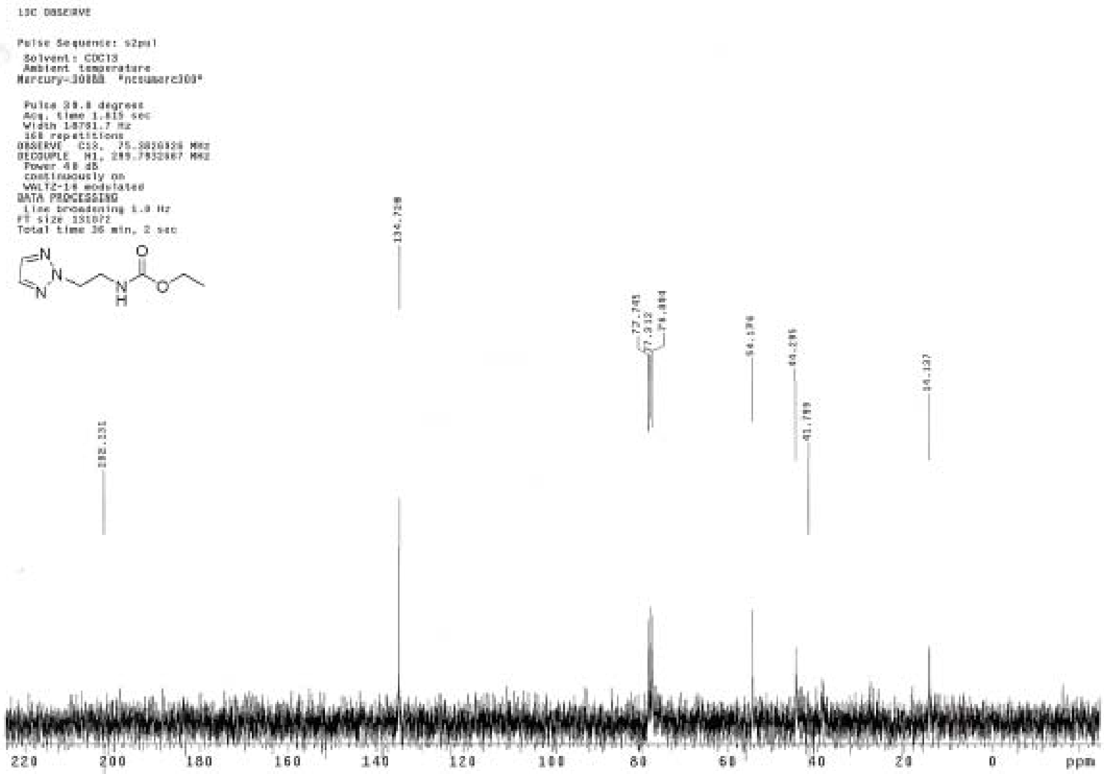


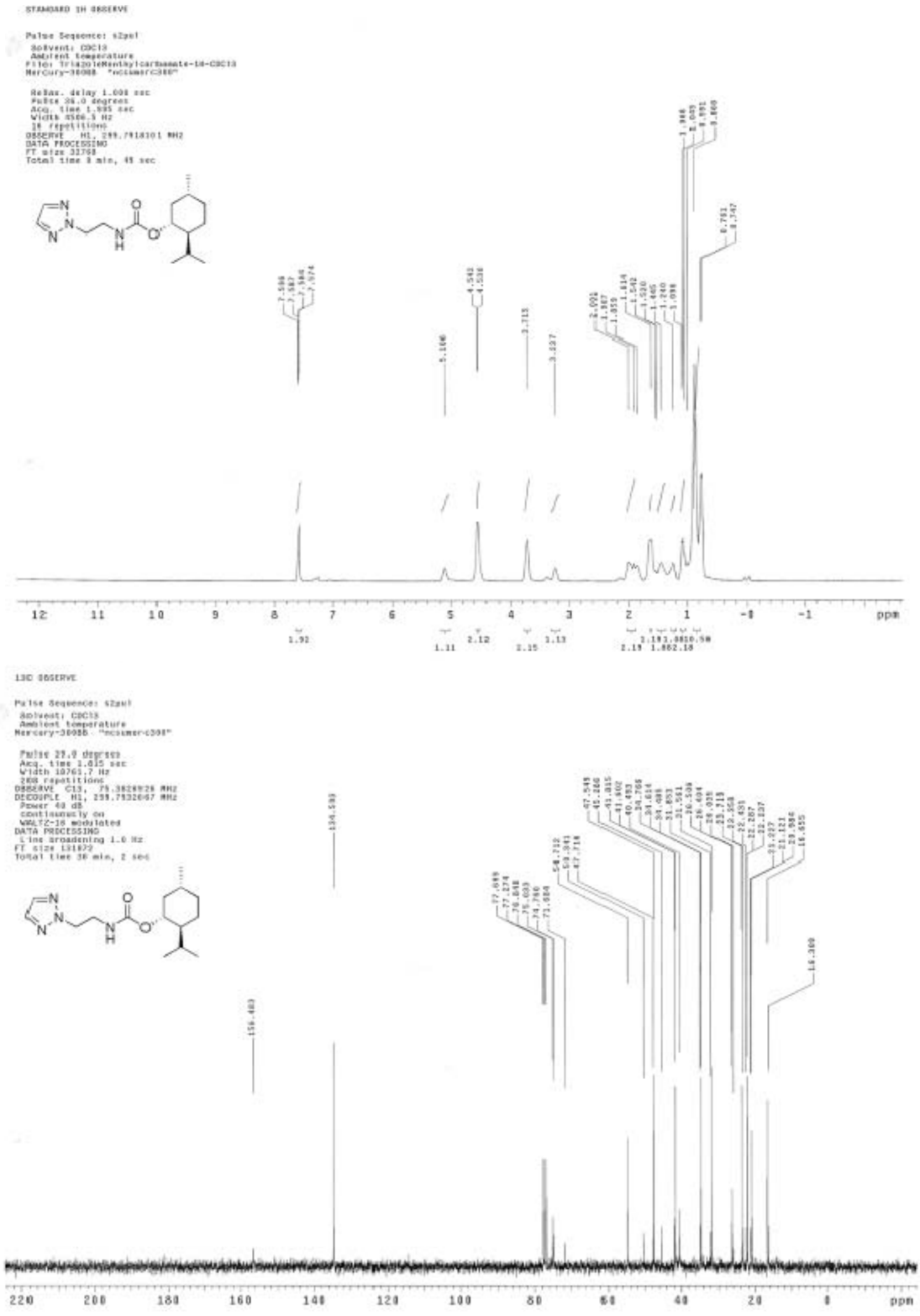

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.955 sec
Width 4566.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
7.569
4.505
4.486
4.468
3.552
3.533
3.516
3.497
1.483
1.384
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.92
0.98
1.98
1.01
8.98
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
224 repetitions
OBSERVE C13, 75.4590250 MHz
DECOUPLE H1, 300.0068387 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 56 min, 2 sec
134.569
155.693
79.753
77.257
77.000
76.747
54.797
40.171
28.524
27.354
220
200
180
160
140
120
100
80
60
40
20
0
ppm

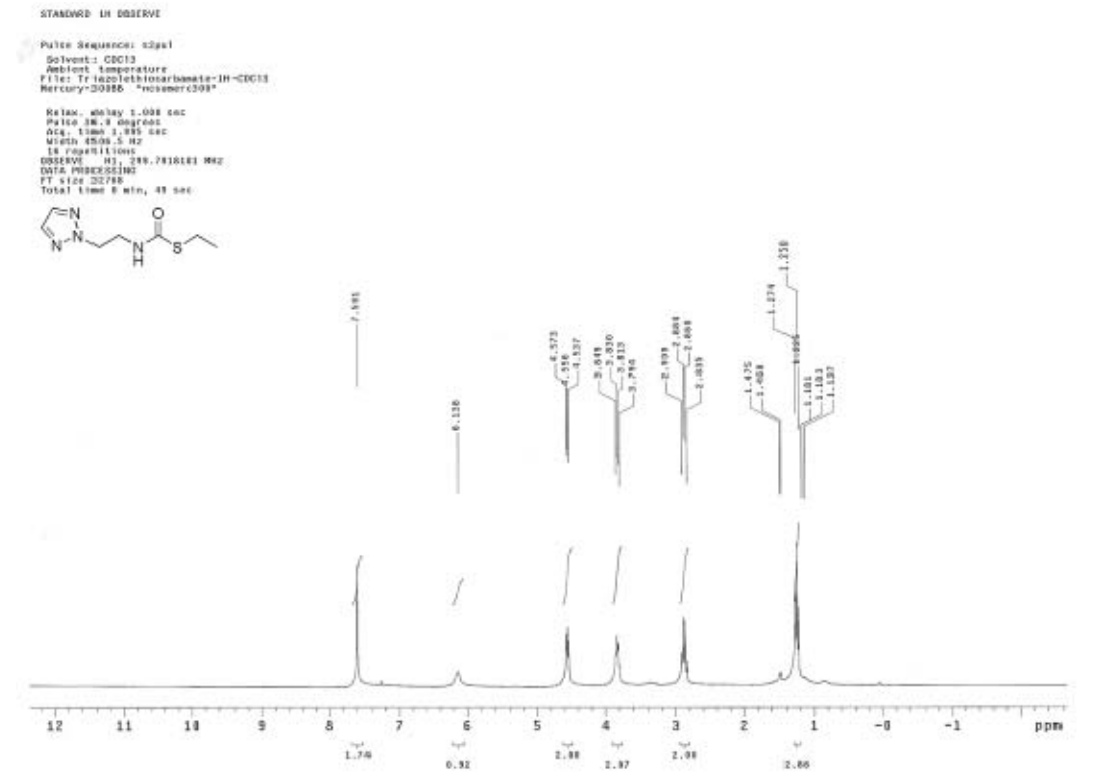

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc038"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 3.335 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 300.0353703 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
ppm

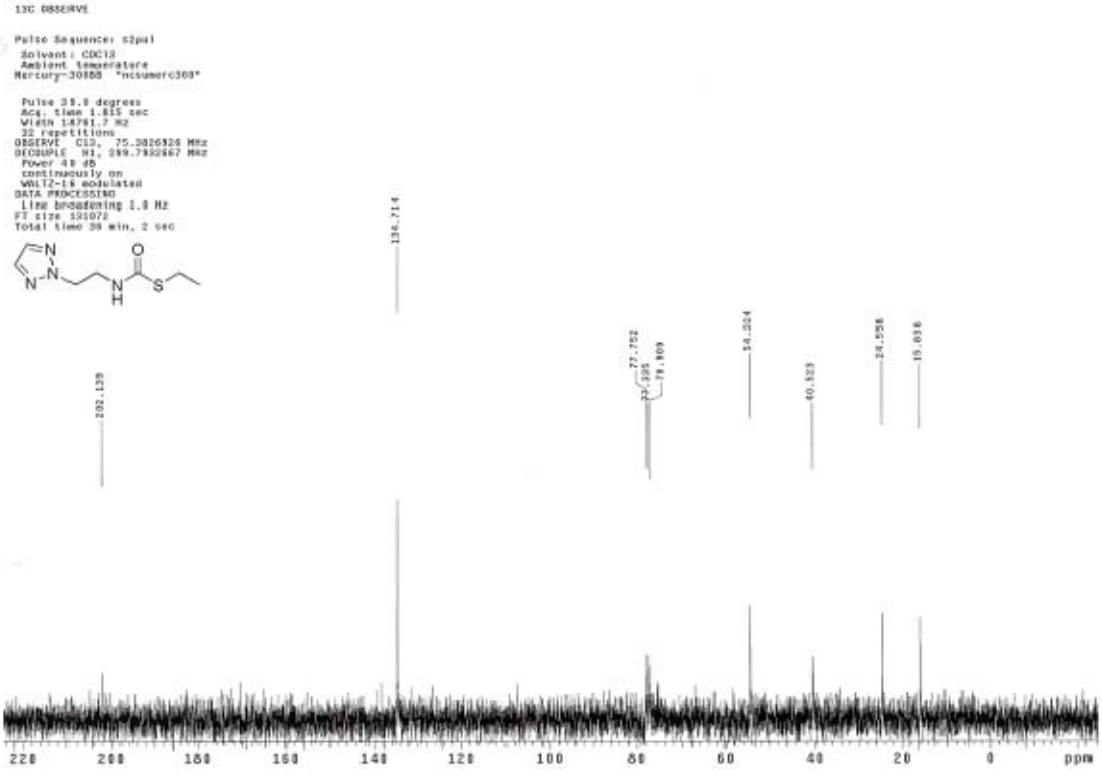

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc038"
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
68 repetitions
OBSERVE C13, 75.4598250 MHz
DECOUPLE H1, 300.0868387 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.8 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

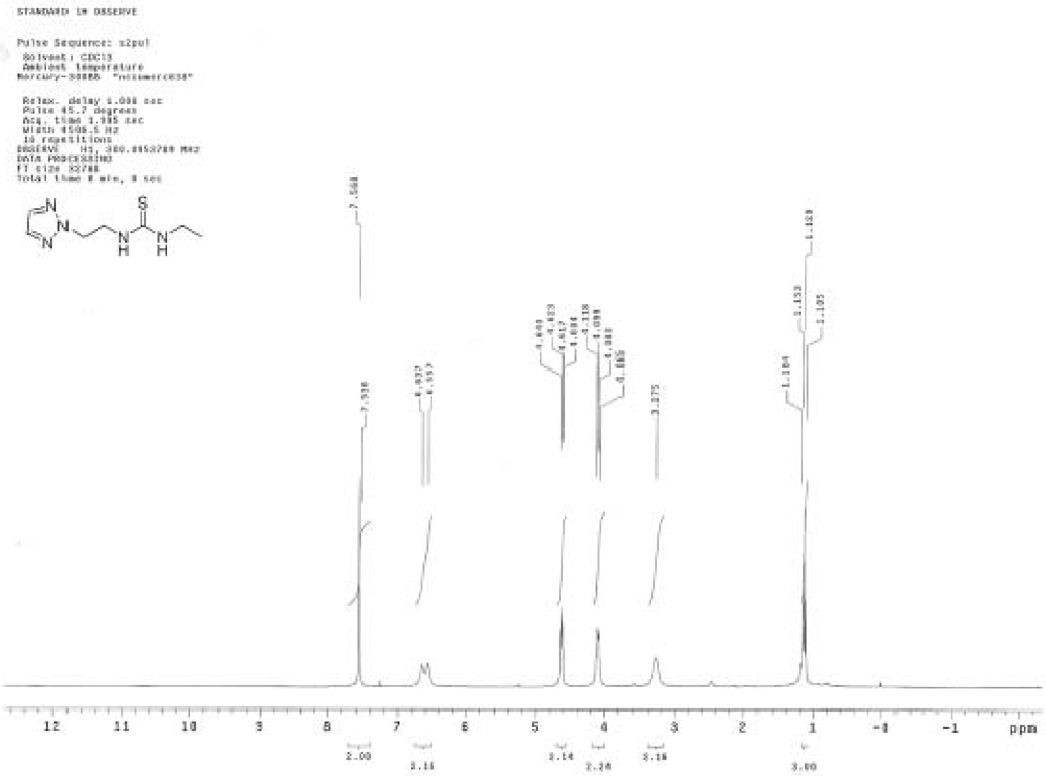

Solvent: CDCl3
Ambient temperature
DATA PROCESSING
ppm

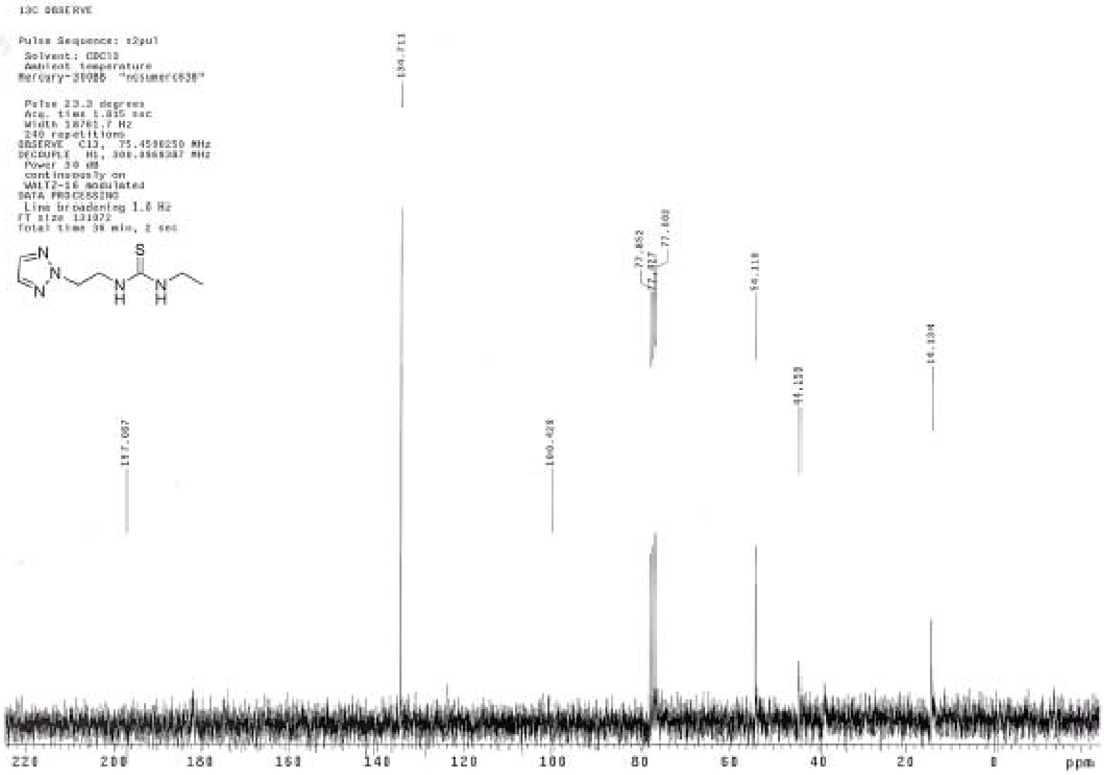

13C OBSERVE
Solvent: CDCl3
Ambient temperature
DATA PROCESSING
ppm

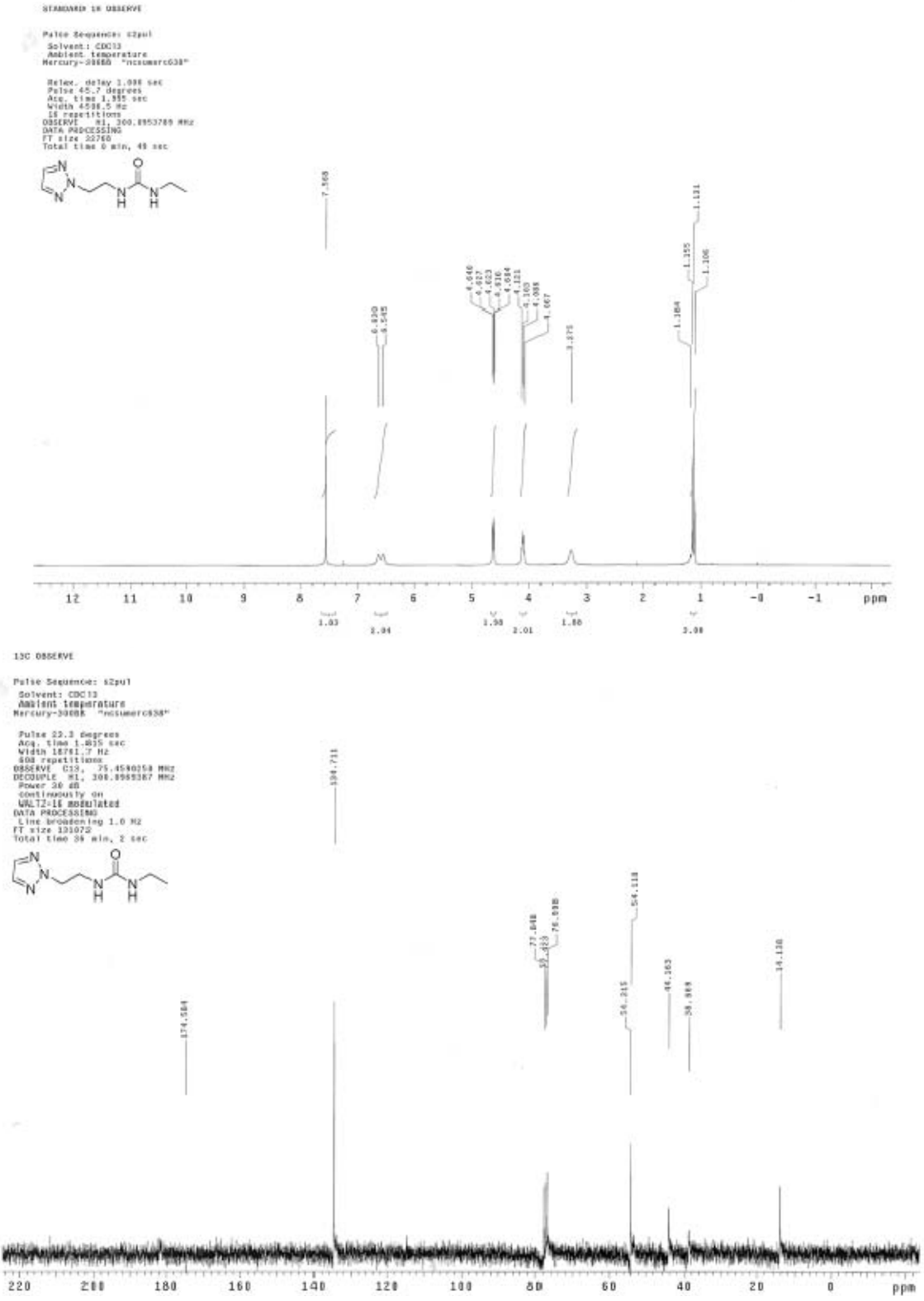


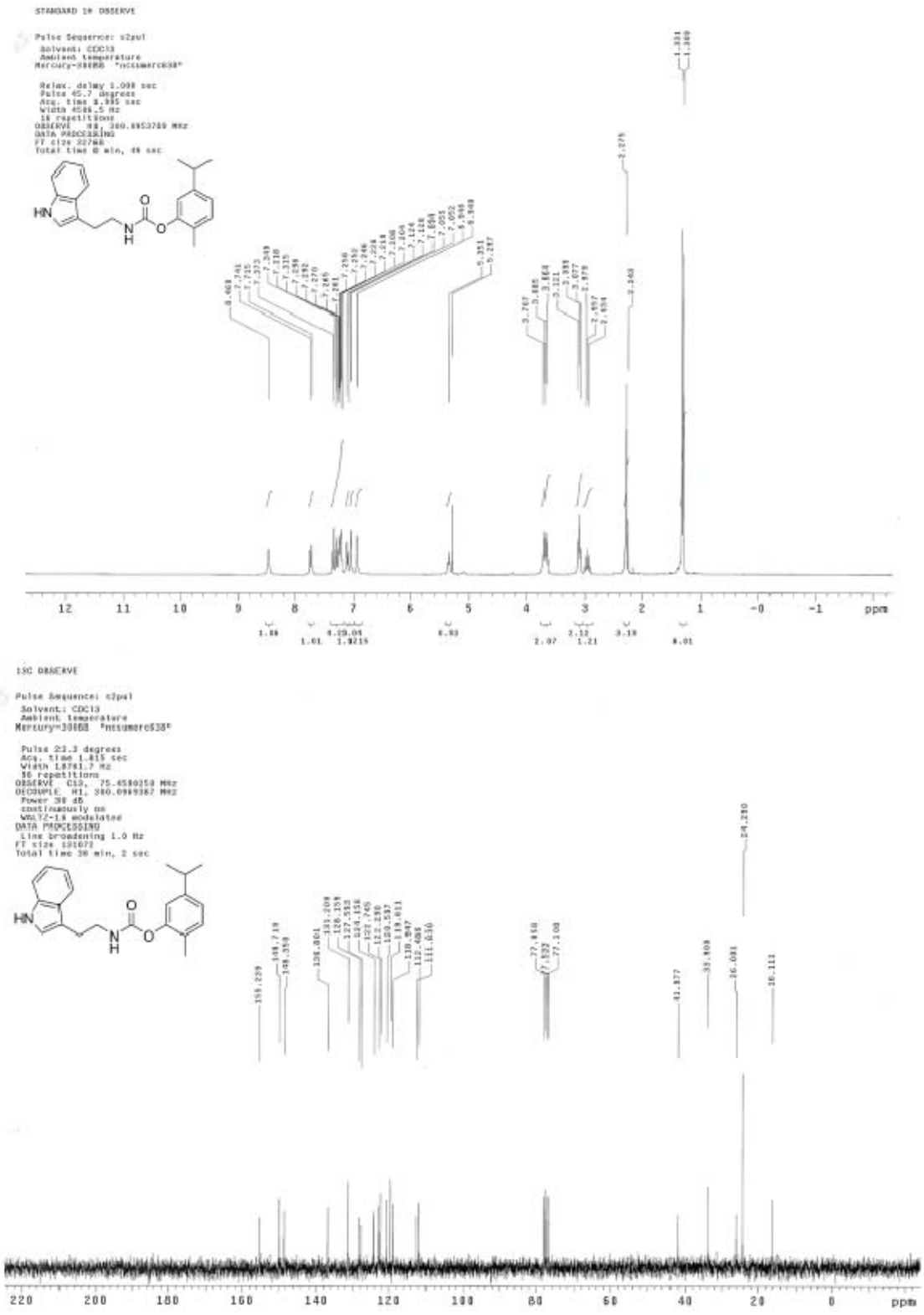

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Triazolethiourbamate-1H-CDCl3
Mercury-300BB "ncsumerc300"
Relax. delay 1.000 sec
Pulse 38.9 degrees
Acq. time 1.995 sec
Width 4508.5 Hz
16 repetitions
OBSERVE H1, 299.7818181 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
7.691
6.136
6.573
6.556
6.537
3.849
3.830
3.815
3.798
2.939
2.884
2.868
2.839
1.475
1.458
1.274
1.258
1.206
1.181
1.163
1.137
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.74
0.92
2.88
2.87
2.98
2.86

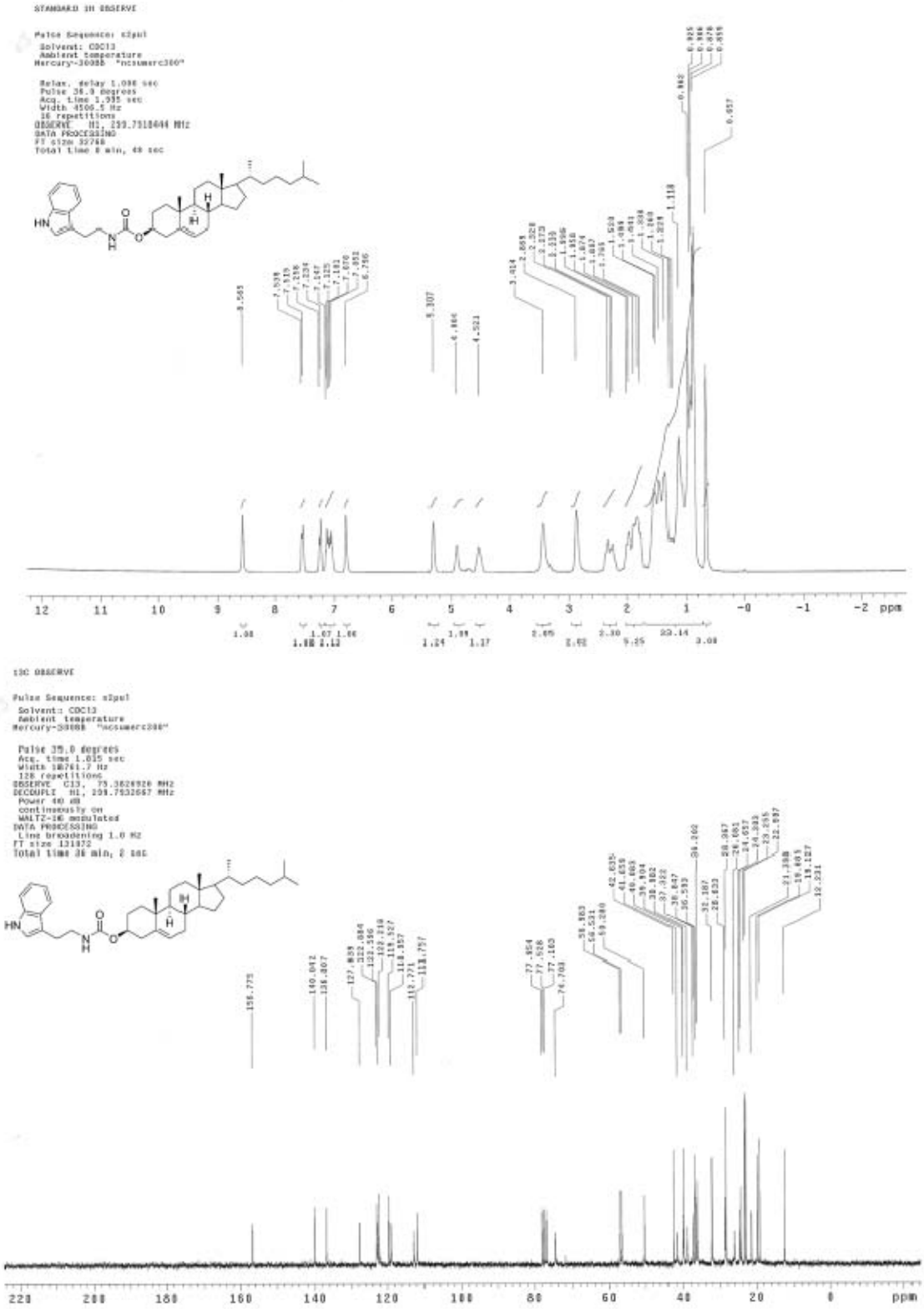

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"
Pulse 38.8 degrees
Acq. time 1.815 sec
Width 18781.7 Hz
32 repetitions
OBSERVE C13, 75.3826526 MHz
DECOUPLE H1, 299.7832667 MHz
Power 48 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 38 min, 2 sec
136.714
77.752
77.305
76.859
54.224
40.523
24.958
19.636
202.139
220
200
180
160
140
120
100
80
60
40
20
0
ppm

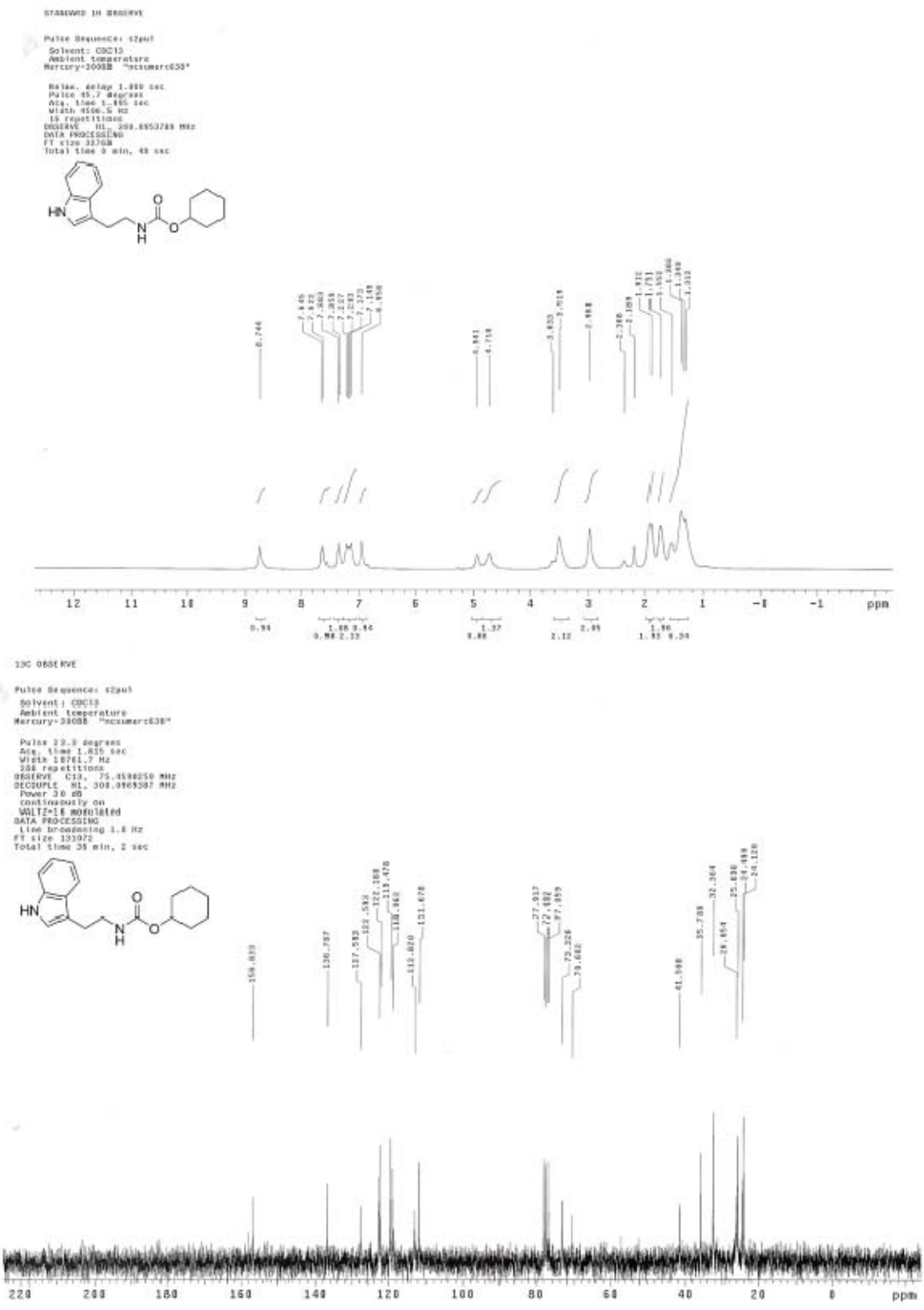


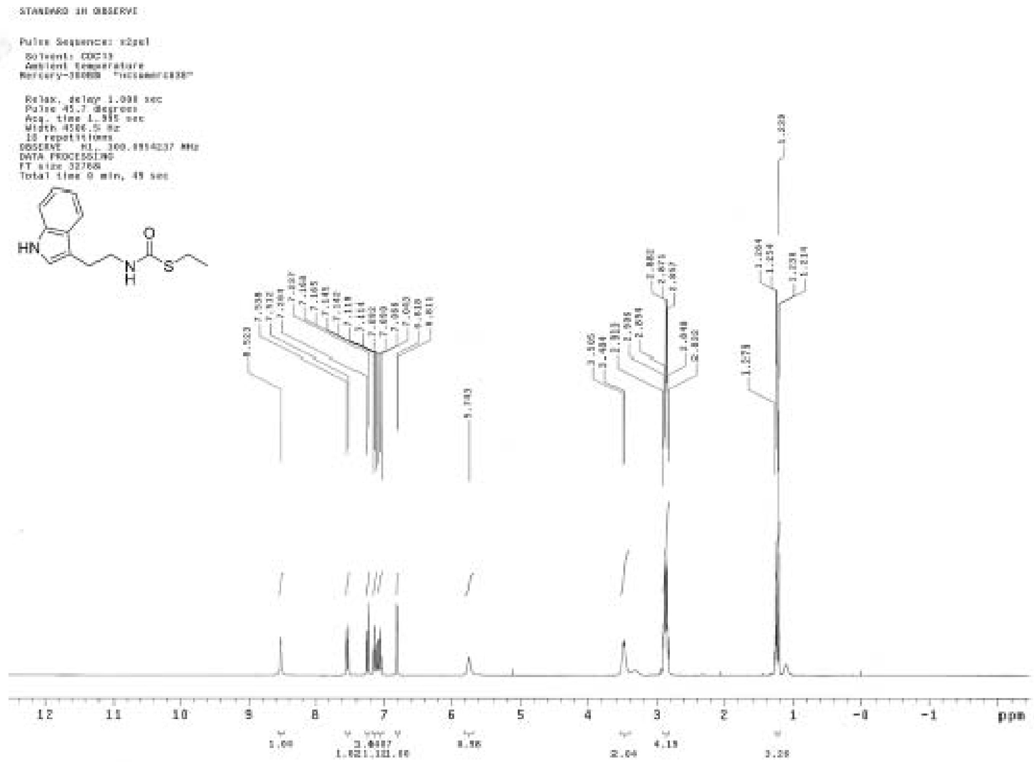


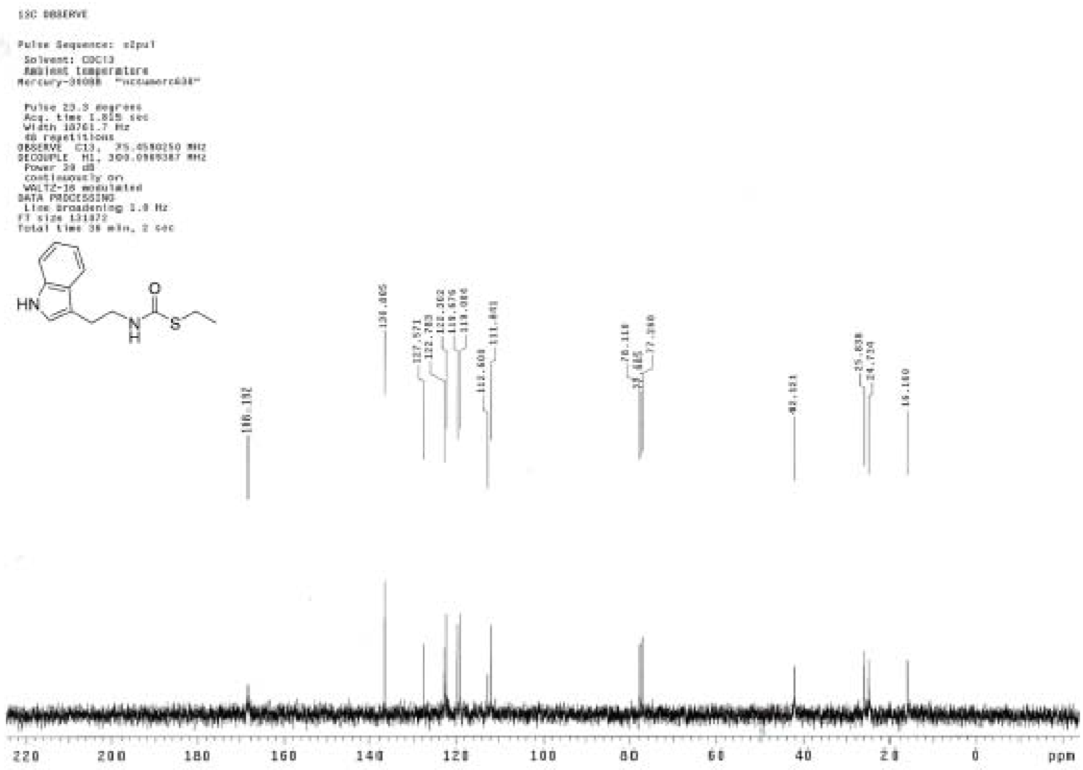

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.955 sec
Width 4598.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
7.568
6.820
6.545
4.046
4.027
4.023
4.018
4.004
4.121
4.103
4.098
4.067
3.375
1.384
1.355
1.131
1.106
12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
1.03
1.04
1.98
2.01
1.86
3.00
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 22.3 degrees
Acq. time 1.835 sec
Width 18761.7 Hz
408 repetitions
OBSERVE C13, 75.4580258 MHz
DECOUPLE H1, 300.0065387 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 38 min, 2 sec
154.711
77.048
76.838
54.118
54.215
46.163
38.869
14.130
174.564
220 200 180 160 140 120 100 80 60 40 20 0 ppm

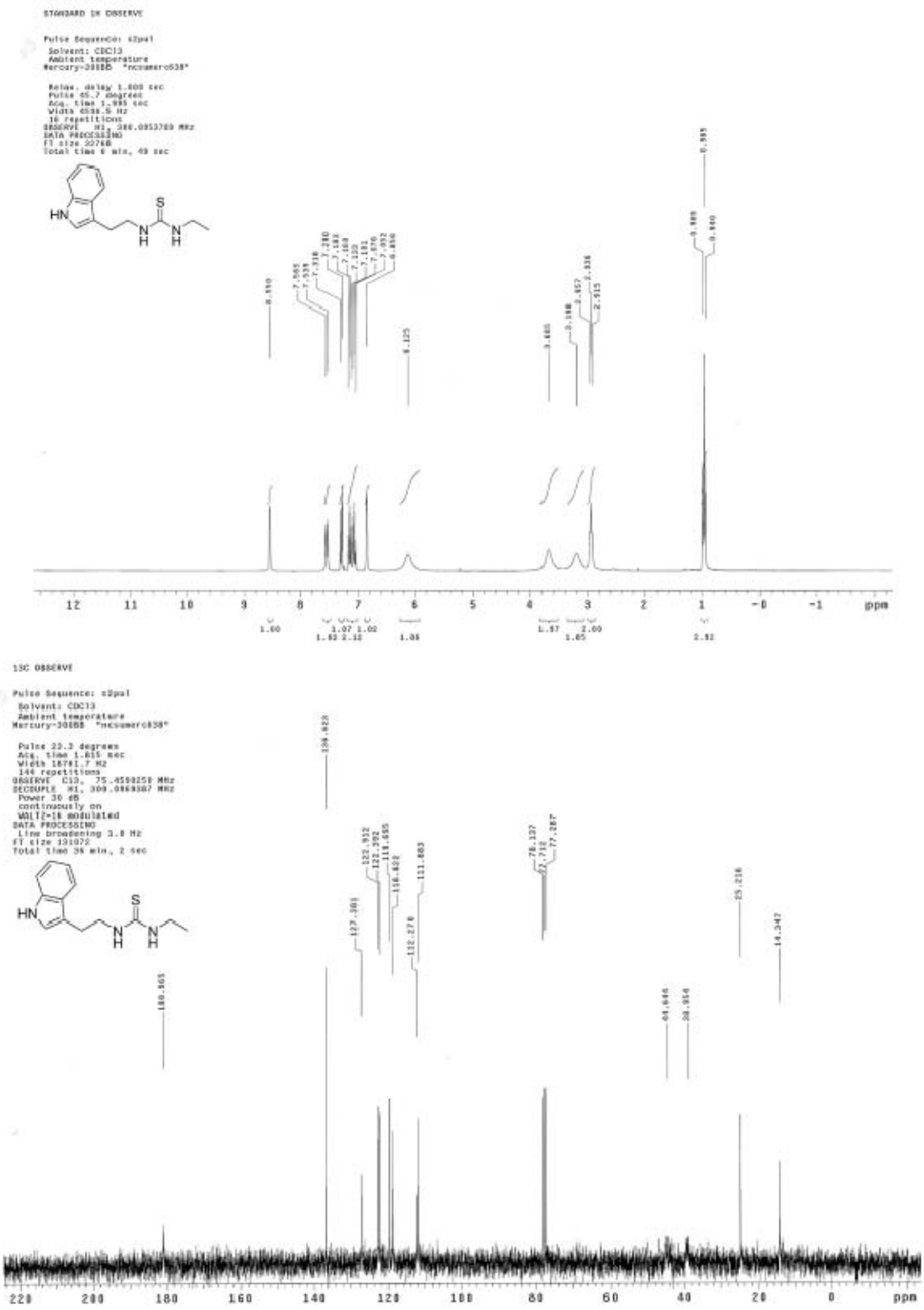


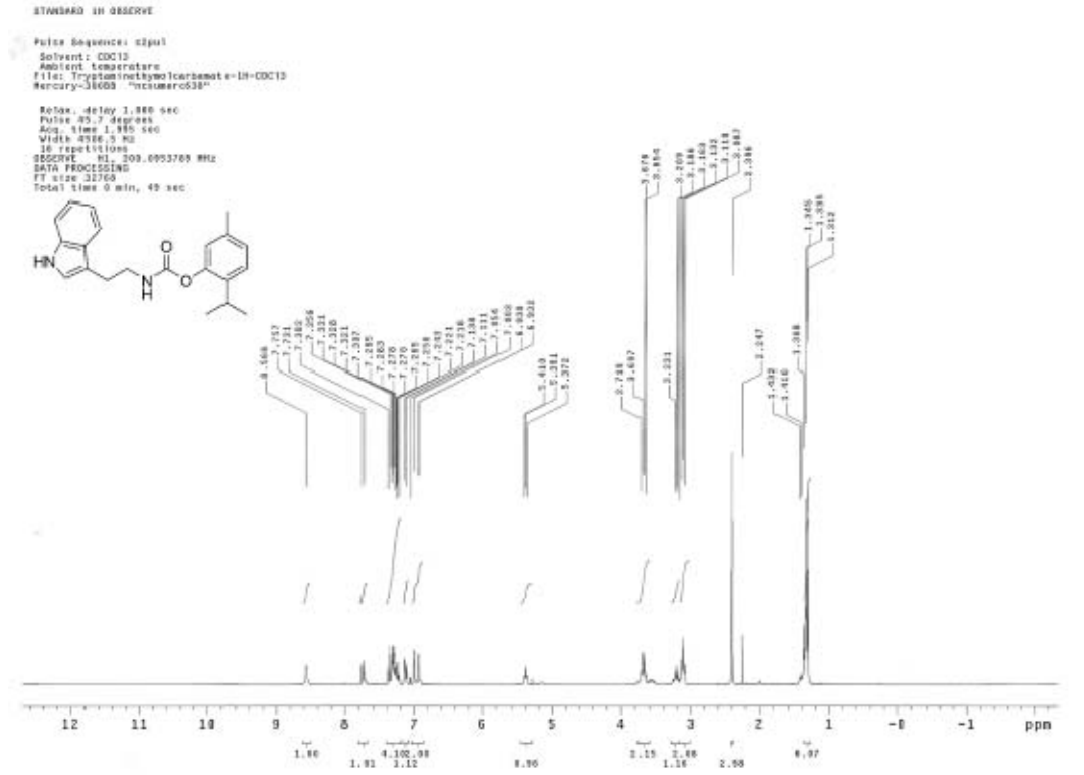

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 26.9 degrees
Acq. time 1.595 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 299.7518644 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 48 sec
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 35.0 degrees
Acq. time 1.015 sec
Width 18761.7 Hz
128 repetitions
OBSERVE C13, 75.3828928 MHz
DECOUPLE H1, 299.7532667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
ppm

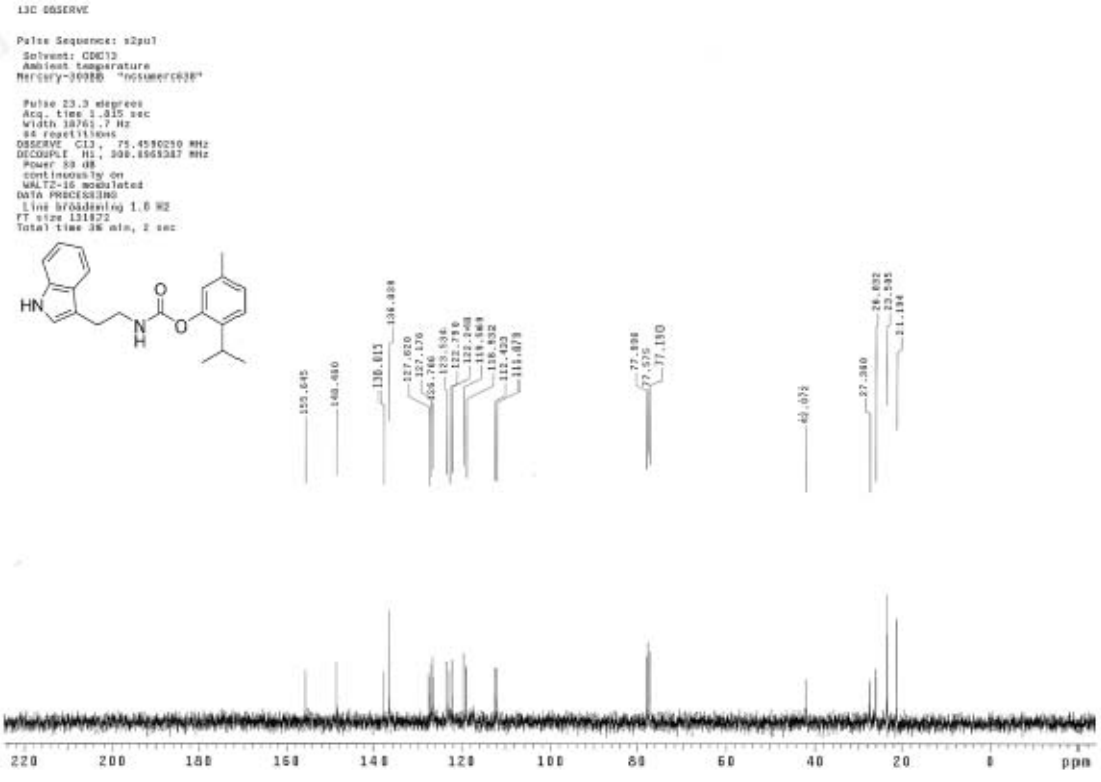







STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "nesumerc838"
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4598.5 Hz
16 repetitions
OBSERVE H1, 300.0353700 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
8.510
7.565
7.539
7.378
7.210
7.183
7.158
7.133
7.101
7.070
7.052
6.836
6.125
3.485
3.198
3.457
3.336
2.515
0.995
0.980
0.840
1.00
1.07 1.02
1.83 2.12
1.08
1.57
2.00
1.85
2.92
12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "nesumerc838"
Pulse 23.3 degrees
Acq. time 1.615 sec
Width 18781.7 Hz
144 repetitions
OBSERVE C13, 75.4598258 MHz
DECOUPLE H1, 300.0869387 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 36 min, 2 sec
180.965
139.828
127.301
122.932
122.392
118.695
118.402
111.883
132.276
78.327
77.710
77.267
61.646
38.954
25.236
14.347
220 200 180 160 140 120 100 80 60 40 20 0 ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Relax. delay 1.000 sec
Pulse 45.7 degrees
Acq. time 1.995 sec
Width 4586.5 Hz
16 repetitions
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec
HN
O
N
H
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Pulse 23.3 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
64 repetitions
DECOUPLE H1, 300.0565387 MHz
Power 53 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min, 2 sec
HN
O
N
H
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm