Base-Promoted Reactions of Bridged Ketones and 1,3- and 1,4-Haloalkyl Azides: Competitive Alkylation vs. Azidation Reactions of Ketone Enolates

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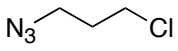
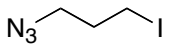
General. Compounds **1** and **2** have been previously reported.[1] **CAUTION**: Although we have not experienced any explosions, alkyl azides can be hazardous compounds. Accordingly, alkyl azides **A**–**D** were not distilled and in some cases used in crude form for safety reasons. We strongly recommend the use of these compounds with appropriate precautions, including the use of safety shields and the avoidance of very large-scale reactions.



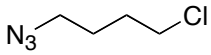
A

1-Azido-3-chloropropane (A). NaN_3 (1.0 g, 6.35 mmol) was added to a solution of 1-bromo-3-chloropropane (0.41 g, 6.35 mmol) in 20 mL of DMF at room temperature. The reaction mixture was allowed to stir for 20 h. The reaction mixture was partitioned between ether and water, and the organic layer was washed with water 3x, dried over Na_2SO_4 and concentrated to give **A** (0.71 g, 94%) as a colorless oil. The material was approximately 90% pure by NMR and used as obtained. ^{1}H NMR (400 MHz, $CDCl_3$) □ 2.03 (m, 2H), 3.51 (t, *J* = 6.2 Hz, 2H), 3.65 (t, *J* = 6.2 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) □ 32.3, 42.0, 48.7; IR (neat) 2100 cm^{-1}; MS (CI) *m/z* 120 (M^++1); HRMS calcd for $C_3H_7ClN_3$ (M^++1): 120.0344, found 120.0355.



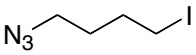
B

1-Azido-3-iodopropane (B). NaI (768 mg, 5.12 mmol) was added to a solution of **A** (306 mg, 2.56 mmol) in 15 mL of acetone and heated to reflux for 24 h. The reaction mixture was partitioned between EtOAc and water. The combined organic layers were dried over Na_2SO_4 and concentrated to give an oil. Flash chromatography (10% EtOAc/hexane) afforded **B** (461 mg, 85%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) 2.09 (m, 2H), 3.27 (t, J = 6.6 Hz, 2H), 3.46 (t, J = 6.4 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 2.8, 32.7, 51.9; IR (neat) 2098 cm^{-1}; MS (CI) *m/z* 211 (M^++1); HRMS calcd for $C_3H_7IN_3$ (M^++1): 211.9685, found 211.9672.



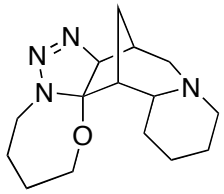
C

1-Azido-4-chlorobutane (C). ^{1}H NMR (400 MHz, $CDCl_3$) 1.76 (m, 2H), 1.87 (m, 2H), 3.36 (t, J = 6.6 Hz, 2H), 3.53 (t, J = 6.5 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 26.6, 30.0, 44.8, 51.1; IR (neat) 2100 cm^{-1}; MS (CI) *m/z* 134 (M^++1); HRMS calcd for $C_4H_9ClN_3$ (M^++1):134.0445, found 134.0428.



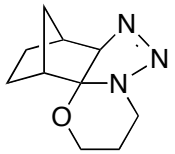
D

1-Azido-4-iodobutane (D). ^{1}H NMR (400 MHz, $CDCl_3$) 1.73 (m, 2H), 1.91 (m, 2H), 3.23 (t, J = 6.8 Hz, 2H), 3.36 (t, J = 6.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 6.1, 30.1, 30.8, 50.8; IR (neat) 2100 cm^{-1}; MS (CI) *m/z* 225 (M^++1); HRMS calcd for $C_4H_8IN_3$ (M^++1): 225.3953 found 225.3939.



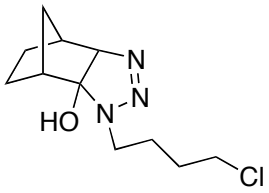
3

Triazoline 3. A 2.5 M solution of *n*-butyllithium in hexanes (0.31 mL, 0.78 mmol) was added dropwise to a solution of THF (8 mL) and diisopropylamine (0.086 g, 0.85 mmol) at 0 °C and allowed to stir under argon for 10 min. The mixture was cooled to -78 °C and ketone[1] (117 mg, 0.65 mmol) was added. After stirring for 1 h, 1-azido-4-iodobutane **D** (294 mg, 1.31 mmol) was added dropwise. After 30 min at -78 ºC, the reaction mixture was allowed to warm to 0 ºC for 2 h. The reaction mixture was quenched with saturated $NaHCO_3$, extracted with EtOAc, dried over Na_2SO_4, and concentrated to give an oil. Flash chromatography (40% EtOAc/hexane) afforded 107 mg (60%) of **3** as an oil: ^{1}H NMR (500 MHz, $CDCl_3$) □ 1.12-1.20 (m, 2H), 1.28-1.31 (m, 2H), 1.49-1.54 (m, 3H), 1.66-1.71 (m, 4H), 1.86 (m, 1H), 1.96-1.98 (m, 2H), 2.14-2.20 (m, 2H), 2.44 (s, 1H), 2.67 (m, 1H), 2.83 (m, 1H), 2.99 (m, 1H), 3.16 (m, 1H), 3.89 (m, 1H), 4.14 (m, 1H), 4.36 (s, 1H); ^{13}C NMR (500 MHz, $CDCl_3$) □ 25.0, 25.5, 28.2, 30.2, 30.4, 36.1, 40.9, 45.3, 47.8 (2C), 56.3, 62.1, 66.3, 85.3, 102.9; IR (neat) 2940, 2750 cm^{-1}; MS (CI) *m*/*z* 277 (M^++1): 97; HRMS calcd for $C_{15}H_{24}N_4O$ (M^++1): 277.2028, found 277.2016. A sample solidified on standing; the structure of compound **3** was confirmed through X-ray crystallography.



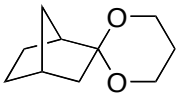
4

3,4,7,8,9,10-Hexahydro-2*H*,6a*H*-1-oxa-4a,5,6-triaza-7,10-methano-benzo[c]indene (4). Prepared using the same procedure as above from norcamphor (555 mg, 5.04 mmol) and 1-azido-3-iodopropane (1.06 g, 7.56 mmol). Flash chromatography (50% EtOAc/hexane) afforded 917 mg (94%) of triazoline **4** as an oil. ^{1}H NMR (500 MHz, $CDCl_3$) □ 1.21-1.22 (m, 2H), 1.45-1.47 (m, 2H), 1.61-1.71 (complex, 3H), 1.95-2.01 (m, 1H), 2.66-2.67 (m, 1H), 2.79-2.80 (m, 1H), 3.66-3.88 (m, 4H), 4.36-4.40 (m, 1H); ^{13}C NMR (500 MHz, $CDCl_3$) □ 21.9, 26.6, 27.5,35.7, 40.6, 42.5, 42.8, 62.8, 91.4, 96.4; IR (neat) 2950 cm^{-1}; MS (CI) *m/z* 194 (M^++1): 97; HRMS calcd for $C_{10}H_{16}N_3O$ (M^++1): 194.1215, found 194.1222.



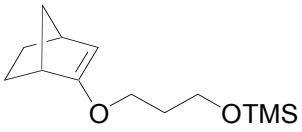
5

1-(4'-Chlorobutyl)-7a-hydroxyl-4,7-methano-1*H*-benzotriazole (5). Prepared using the same procedure as above from norcamphor and alkyl azide **C** . Yield 91%. ^{1}H NMR (500 MHz, $CDCl_3$) □ 1.17 (m, 2H), 1.50 (m, 2H), 1.70 (m, 1H), 1.89-1.97 (complex, 5H), 2.40 (s, 1H), 2.58 (m, 1H), 3.58-3.65 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) □ 23.1, 27.1, 27.7, 30.4, 34.6, 43.7, 43.8, 44.9, 45.0, 87.4, 96.4; IR (neat) 3168 cm^{-1}; MS (CI) *m/z* 244 (M^++1); HRMS calcd for $C_{11}H_{18}N_3OCl$ (M^++1): 244.1217, found 244.1205.



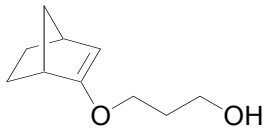
E

Spiro[bicyclo[2.2.1]heptane-2,2'-[1,3]dioxolane (E). E was made from norcamphor and 1,3-propanediol using a procedure of Gassman[2] et al. Yield: 90%. Bp 65-66 °C (0.2 mm Hg), ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.22 (m, 2H), 1.38 (m, 2H), 1.60-1.64 (complex, 4H), 1.75-1.83 (m, 2H), 2.33 (s, 1H), 2.68 (s, 1H), 3.80-3.95 (complex, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 21.8, 26.0, 28.8, 36.0, 37.4, 42.1, 44.4, 60.9, 62.6, 108.3; IR (neat) 2983 cm^{-1}; MS (CI) *m/z* 169 (M^++1); HRMS calcd for $C_{10}H_{17}O_2$ (M^++1): 169.1229, found 169.1233.



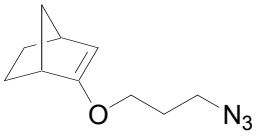
F

2-[3-[(Trimethylsilyl)oxy]propoxy]bicyclo[2.2.1]hep-2-ene (F). F was made from **E** by procedure of Gassman[2] et al. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.14 (s, 9H), 1.08 (d, *J* = 7.9 Hz, 1H), 1.13-1.25 (m, 2H), 1.47-1.50 (m, 1H), 1.66-1.70 (m, 2H), 1.89 (pentet, *J* = 5.9 Hz, 2H), 2.67-2.68 (m, 1H), 2.82-2.83 (m, 1H), 3.53-3.80 (complex, 4H), 4.54 (d, *J* = 3.1 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –0.2, 25.2, 28.7, 32.4, 41.2, 44.4, 47.4, 59.5, 65.6, 97.7, 116.6; IR (neat) 1614 cm^{-1}; MS (CI) *m/z* 241 (M^++1); HRMS calcd for $C_{13}H_{25}O_2Si$ (M^++1): 241.1526, found 241.1545.



G

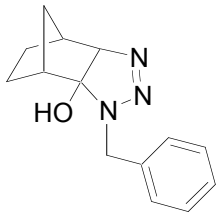
3- (Bicyclo[2.2.1]hept-2-en-2-yloxy)-propan-1-ol (G). To a solution of **F** (2.80 g, 11.7 mmol) in methanol was added K_2CO_3 (1.61 g, 11.7 mmol) at 0 ºC, and the solution allowed to stir for 1 h. After filtration and evaporation, the residue was partitioned between water and CH_2Cl_2, and the organic layer was dried with Na_2SO_4. Removal of solvent gave **G** 1.90 g (98%) as a brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07 (d, *J* = 7.9 Hz, 1H), 1.13-1.30 (m, 2H), 1.47-1.51 (m, 1H), 1.63-1.75 (m, 2H), 1.87 (t, *J* = 5.5 Hz, 1H), 1.94 (pentet, *J* = 5.9 Hz, 2H), 2.67-2.68 (m, 1H), 2.82-2.83 (m, 1H), 3.70-3.90 (complex, 4H), 4.55 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 25.2, 28.6, 32.1, 41.2, 44.4, 47.4, 61.3, 67.2, 96.3; IR (neat) 3418, 1614 cm^{-1}; MS (CI) *m/z* 169 (M^++1); HRMS calcd for $C_{10}H_{17}O_2$ (M^++1): 169.1229, found 169.1220.



6

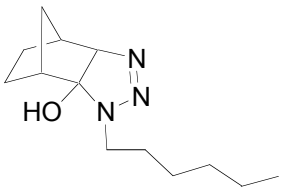
2-[3-Azido-propoxy]bicyclo[2.2.1]hept-2-ene (6). To a solution of **G** (200 mg, 1.2 mmol), $Zn(N_3)_2$•2Py[3] (275 mg, 0.9 mmol) and PPh_3 (620 mg, 2.4 mmol) in dry THF, was added diisopropylazodicarbonate (470 mg, 2.4 mmol) dropwise at room temperature, and the reaction mixture was allowed to stir overnight. The reaction was poured through Celite and concentrated to afford compound **6** (200 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07-1.96 (complex, 8H), 2.68-2.83 (m, 2H), 3.45 (t, *J* = 6.6 Hz, 2H), 3.65-3.78 (m, 2H), 4.54 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 25.3, 28.6, 28.9, 41.2,

44.3, 47.4, 48.8, 65.6, 98.1, 166.3; IR (neat) 2098, 1613 cm^{-1}; MS (CI) *m/z* 194 (M^++1); HRMS calcd for $C_{10}H_{16}N_3O$ (M^++1): 194.1215, found 194.1222.



7

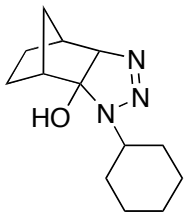
1-Benzyl-7a-hydroxy-4,7-methano-1*H*-benzotriazole (7). Prepared by the same procedure described in compound **10** from norcamphor and benzyl azide. Yield 67%; mp 111-118 °C. ^{1}H NMR (400 MHz, $CDCl_3$) □ 1.01-1.87 (complex, 6H); 2.08 (d, *J* = 3.0 Hz, 1H), 2.56 (d, *J* = 3.8 Hz, 1H), 3.66 (s, 1H), 4.80 (AB q, *J* = 15.2 Hz, □□ = 4.2 Hz, 2H), 5.57 (s, 1H), 7.28-7.43 (complex, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) □ 22.9, 27.2, 34.6, 43.8, 45.0, 48.5, 87.6, 96.6, 128.0, 128.9, 129.0, 138.2; IR (neat) 3165,1460 cm^{-1}; MS (CI) *m/z* 244 (M^++1); HRMS calcd for $C_{14}H_{18}N_3O$ (M^++1): 244.1450, found 244.1438.



8

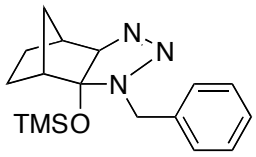
1-Hexyl-7a-hydroxy-4,7-methano-1*H*-benzotriazole (8). Prepared by the same procedure described in compound **10** from norcamphor and 1-azidohexane. Yield 93%; mp 65-67 °C. ^{1}H NMR (400 MHz, $CDCl_3$) □ 0.89-1.91 (complex, 18H), 2.22 (d, *J* = 6.9 Hz, 1H), 2.47 (d, *J* = 6.9 Hz, 1H), 3.55 (t, *J* = 7.6 Hz, 2H), 3.60 (br s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) □ 14.5, 23.0, 23.1, 27.1, 27.2, 30.4, 31.9, 34.5, 43.8, 44.7, 44.9,

87.6, 96.4; IR (neat): 3158, 1456, cm^{-1}; MS (CI) *m/z* 238 (M^++1); HRMS calcd for $C_{13}H_{24}N_3O$ (M^++1): 238.1919, found 238.1925.



9

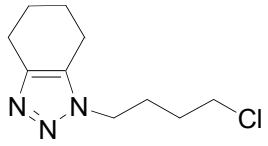
1-Cyclohexyl-7a-hydroxyl-4,7-methano-1*H*-benzotriazole (9). Prepared by the same procedure described in compound **10** from norcamphor and 1-azidocyclohexane. Yield 69%; mp 184-186 °C; ^{1}H NMR (400 MHz, $CDCl_3$) □ 1.12 (s, 2H), 1.21-2.09 (complex, 14H), 2.38 (d, *J* = 2.8 Hz, 1H), 2.51 (d, *J* = 4.2 Hz, 1H), 3.41-3.56 (m, 2H), 6.06 (br s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) □ 23.2, 25.9, 26.3, 27.1, 34.4, 34.7, 44.0, 45.2, 54.7, 85.9, 97.0; IR (neat) 3157, 1450 cm^{-1}; MS (CI) *m/z* 236 (M^++1); HRMS calcd for $C_{13}H_{22}N_3O$ (M^++1): 236.1763, found 236.1756.



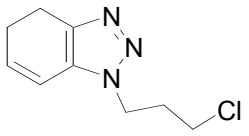
10

1-Benzyl-7a-trimethylsilyloxy-4,7-methano-1*H*-benzotriazole (10). A 2.5 M solution of *n*-butyllithium in hexanes (0.80 mL, 1.99 mmol) was added dropwise to a solution of THF (10 mL) and diisopropylamine (0.239 g, 2.36 mmol) at 0 ºC and allowed to stir under argon for 10 min. The mixture was cooled to -78 °C and norcamphor (200 mg, 1.81 mmol) was added. After stirring for 1 h, benzyl azide (480 mg, 3.61 mmol) was added dropwise. After 30 min at -78 ºC, the reaction mixture was allowed to warm to 0

ºC for 2 h. TMSCl (256 mg, 3.61 mmol) was added and the solution was stirred for another hour. The reaction mixture was quenched with saturated $NaHCO_3$, extracted with EtOAc, dried over Na_2SO_4, filtered, and concentrated. Flash chromatography afforded 542 mg (95%) of **10** as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) 0.09 (s, 9H), 0.86-1.06 (m, 3H), 1.26-1.40 (m, 3H), 1.58-1.71 (m, 2H), 2.02 (m, 1H), 2.62 (d, *J* = 4.4 Hz, 1H), 3.90, (s, 1H), 4.55 (d, *J* = 15.1 Hz, 1H), 4.83 (d, *J* = 15.1 Hz, 1H), 7.29-7.40 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 1.3, 23.0, 27.3, 33.4, 43.7, 46.4, 48.8, 87.2, 97.4, 128.0, 128.9, 129.0, 138.1; IR (neat) 2980 cm^{-1}; MS (CI) *m/z* 316 (M^++1): 258, 91; HRMS calcd for $C_{17}H_{26}N_3OSi$ (M^++1): 316.1845, found 316.1831.

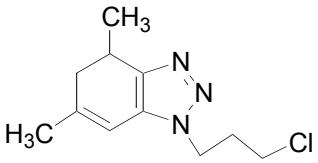


1-*N*- (4'-Chlorobutyl)-4,5,6,7-tetrahydro-1*H*-benzotriazole (Table 1, entry 1). The same procedure was followed as described above (no trapping agent was used). Yield: 95%; oil. ^{1}H NMR (400 MHz, $CDCl_3$) 1.71-1.88 (complex, 6H), 2.03 (m, 2H), 2.61 (t, *J* = 6.0 Hz, 2H), 2.76 (t, *J* = 5.6 Hz, 2H), 3.58 (t, *J* = 6.3 Hz, 2H), 4.26 (t, *J* = 6.9 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 20.5, 22.3, 22.9, 23.0, 27.3, 29.7, 44.6, 47.2, 132.0, 143.9; IR (neat) 1588, 1460 cm^{-1}; MS (CI) *m/z* 214 (M^++1); HRMS calcd for $C_{10}H_{17}N_3Cl$ (M^++1): 214.0955, found 214.0931.

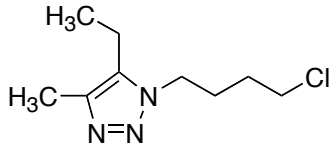


3'-Chloropropyl-4,5-dihydro-1*H*-benzotriazole (Table 1, entry 2). Yield 52%; oil. ^{1}H NMR (400 MHz, $CDCl_3$) 2.38 (pentet, *J* = 6.5 Hz, 2H), 2.54 (ddt, *J* = 2.0, 4.3,

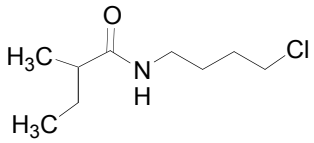
9.0 Hz, 2H), 2.95 (t, *J* = 9.0 Hz, 2H), 3.53 (t, *J* = 6.5 Hz, 2H), 4.44 (t, *J* = 6.5 Hz, 2H), 6.08 (dt, *J* = 4.3, 9.8 Hz, 1H), 6.44 (dt, *J* = 2.0, 9.8 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 20.1, 24.7, 33.0, 41.8, 44.8, 113.7, 132.0, 132.1, 142.8; IR (neat) 1572, 1449 cm^{-1}; MS (CI) *m/z* 198 (M^++1); HRMS calcd for $C_9H_{13}N_3Cl$ (M^++1): 198.0798, found 198.0788.



3'-Chloropropyl-4,6-dimethyl-4,5-dihydro-1*H*-benzotriazole (Table 1, entry 3) . Yield 69%; oil. ^{1}H NMR (400 MHz, $CDCl_3$): 1.33 (d, *J* = 6.9 Hz, 3H), 1.94 (s, 3H), 2.15 (dd, *J* = 8.0, 17.3 Hz, 1H), 2.35 (pentet, *J* = 9.7 Hz, 2H), 2.52 (dd, *J* = 8.0, 17.3 Hz, 1H), 3.17-3.22 (m, 1H), 3.51 (t, *J* = 6.1 Hz, 2H), 4.39 (t, *J* = 9.7 Hz, 2H), 6.13 (d, *J* = 1.4 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 19.5, 24.2, 27.1, 33.2, 39.7, 41.9, 44.7, 108.4, 132.5, 142.3, 146.0; IR (neat) 1567, 1444 cm^{-1}; MS (CI) *m/z* 226 (M^++1); HRMS calcd for $C_{11}H_{17}N_3Cl$ (M^++1): 226.1111, found 226.1132.



4-Methyl-5-ethyl-1-(4'-chlorobutyl)-1*H*-1,2,3-triazole (Table 1, entry 4). Yield 32%; oil. ^{1}H NMR (400 MHz, $CDCl_3$) 1.17 (t, *J* = 7.6 Hz, 3H), 1.79-1.86 (m, 2H), 2.02-2.09 (m, 2H), 2.29 (s, 3H), 2.64 (q, *J* = 7.6 Hz, 2H), 3.57 (t, *J* = 6.3 Hz, 2H), 4.25 (t, *J* = 7.1 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 10.8, 13.8, 16.4, 27.8, 29.7, 44.6, 47.4, 134.5, 140.7; IR (neat) 1567, 1444 cm^{-1}; MS (CI) *m/z* 202 (M^++1); HRMS calcd for $C_9H_{17}N_3Cl$ (M^++1): 202.1111, found 202.1097.



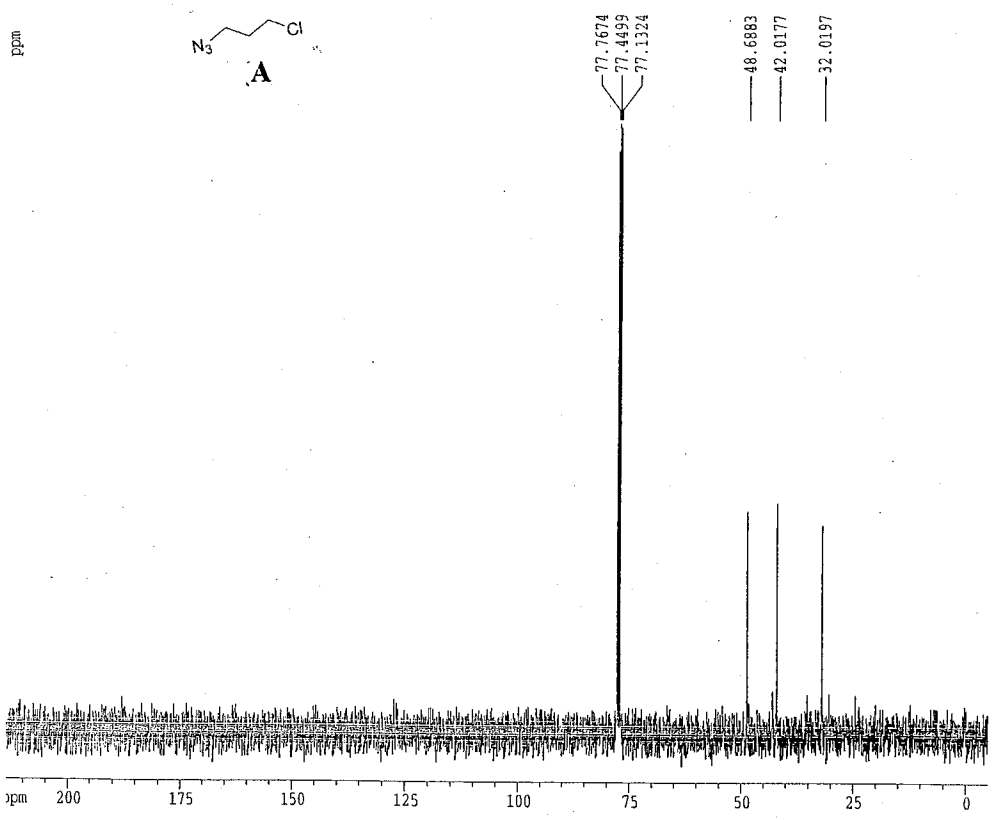
***N*- (4-Chlorobutyl)-2-methyl Butanamide (Table 1, entry 4) .** Yield 30%; oil. ^{1}H NMR (400 MHz, $CDCl_3$) 0.91 (t, J = 7.4 Hz, 3H), 1.13 (d, J = 6.9 Hz, 3H), 1.39-1.46 (m, 1H), 1.60-1.71 (m, 3H), 1.78-1.85 (m, 2H), 2.07-2.11 (m, 1H), 3.20-3.66 (m, 2H), 3.58 (t, J = 6.4 Hz, 2H); 5.66 (br s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) 12.4, 18.0, 27.6, 27.7, 30.2, 38.9, 43.7, 45.0, 177.0; IR (neat) 3288, 1649 cm^{-1}; MS (CI) *m/z* 192 (M^++1); HRMS calcd for $C_9H_{18}ClNO$ (M^++1): 192.1155, found 192.1158.

References

(1) Smith, B. T.; Wendt, J. A.; Aubé, J. *Org*. *Lett*. **2002**, *4*, 2577-2579.

(2) Gassman, P. G.; Burns, S. J.; Pfister, K. B. *J*. *Org*. *Chem*. **1993**, *58*, 1449-1457.

(3) Viaud, M. C.; Rollin, P. *Synthesis* **1990**, 130-132.

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EXPNO                       1
PROCNO                      1

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PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                         22
DS                          2
SWH                23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                      32768
DW                     21.600 usec
DE                      4.50 usec
TE                     300.0 K
D1                 0.05000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

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P1                      12.30 usec
PL1                      2.00 dB
SFO1               100.6232933 MHz

========== CHANNEL f2 ==========
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PCPD2                  100.00 usec
PL2                      0.00 dB
PL12                    18.00 dB
PL13                    18.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
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SF                 100.6127290 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
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F1                     21631.74 Hz
F2P                       -5.000 ppm
F2                      -503.06 Hz
PPMCM                  11.00000 ppm/cm
HZCM                1106.73999 Hz/cm
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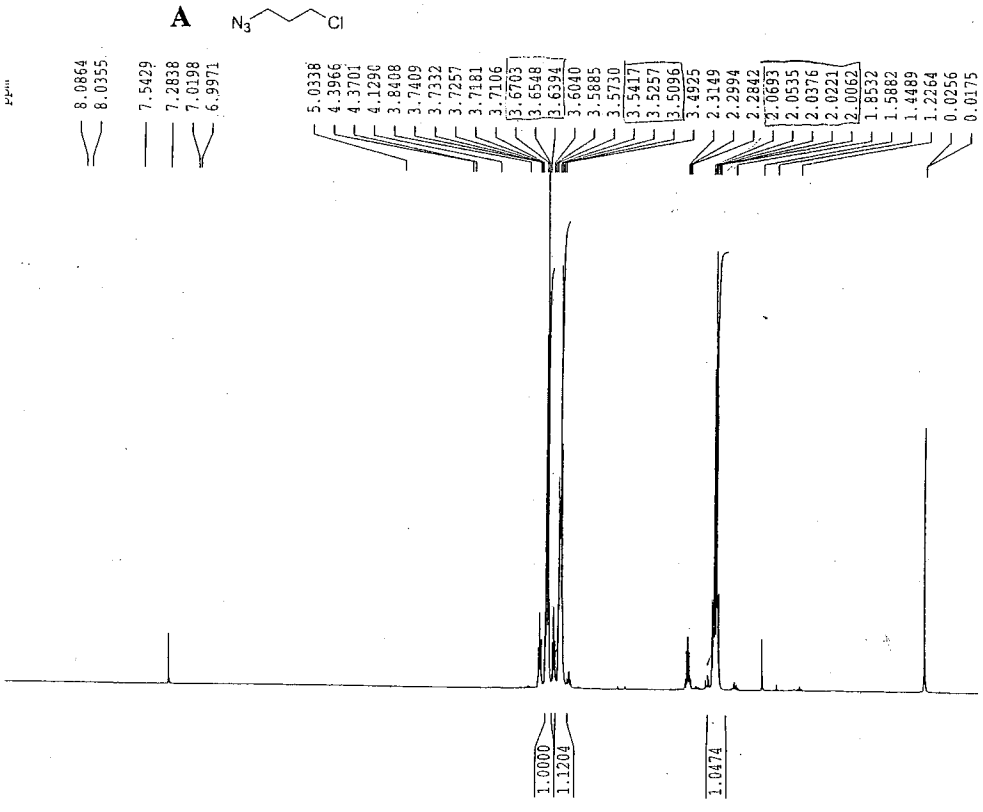


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Current Data Parameters
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EXPNO                       1
PROCNO                      1

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Time                   15.17
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PULPROG                zg30
TD                      32768
SOLVENT                CDCl3
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DS                          2
SWH                 4789.272 Hz
FIDRES             0.146157 Hz
AQ                 3.4210291 sec
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DW                    104.400 usec
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TE                     300.0 K
D1                 1.00000000 sec

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F2 - Processing parameters
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WDW                          EM
SSB                           0
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PC                         1.00

1D NMR plot parameters
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F2P                      -0.500 ppm
F2                     -200.07 Hz
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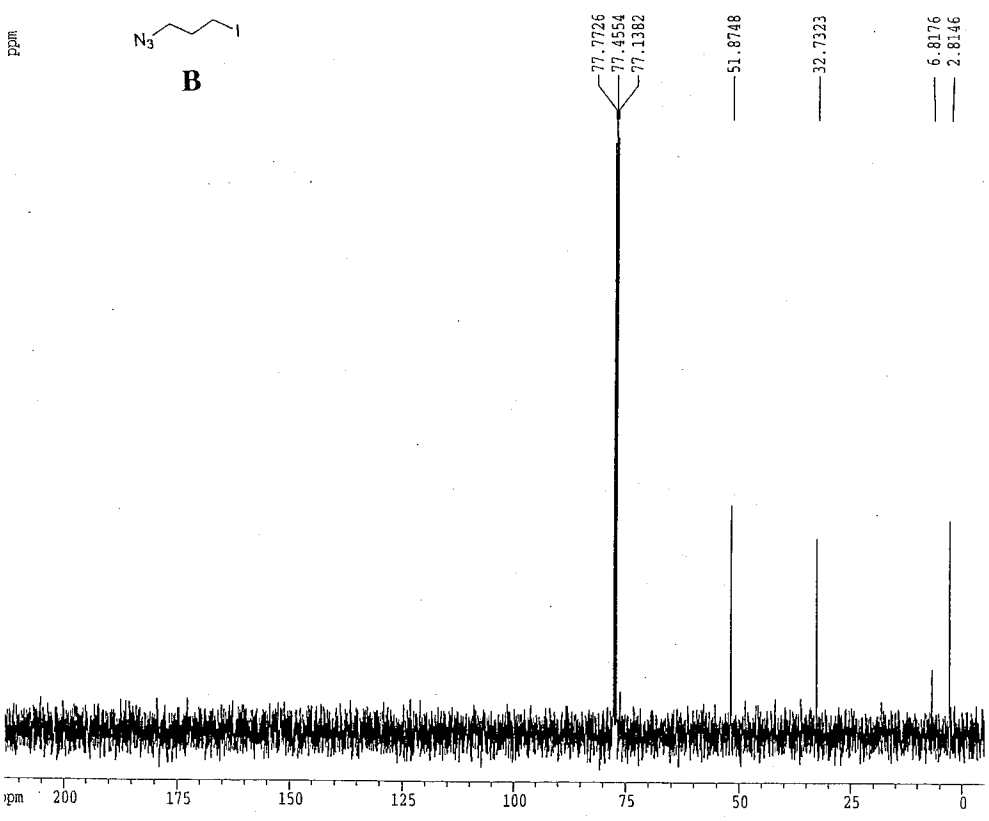


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PROCNO                1

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PULPROG         zgpg30
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SOLVENT          CDCl3
NS                  40
DS                   2
SWH          23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                1276B
DW               21.600 usec
DE                4.50 usec
TE              300.0 K
D1            0.05000000 sec
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============ CHANNEL f1 =============
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PL1               2.00 dB
SFO1        100.6232933 MHz

============ CHANNEL f2 =============
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NUC2                1H
PCPD2          100.00 usec
PL2               0.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM        1106.73999 Hz/cm
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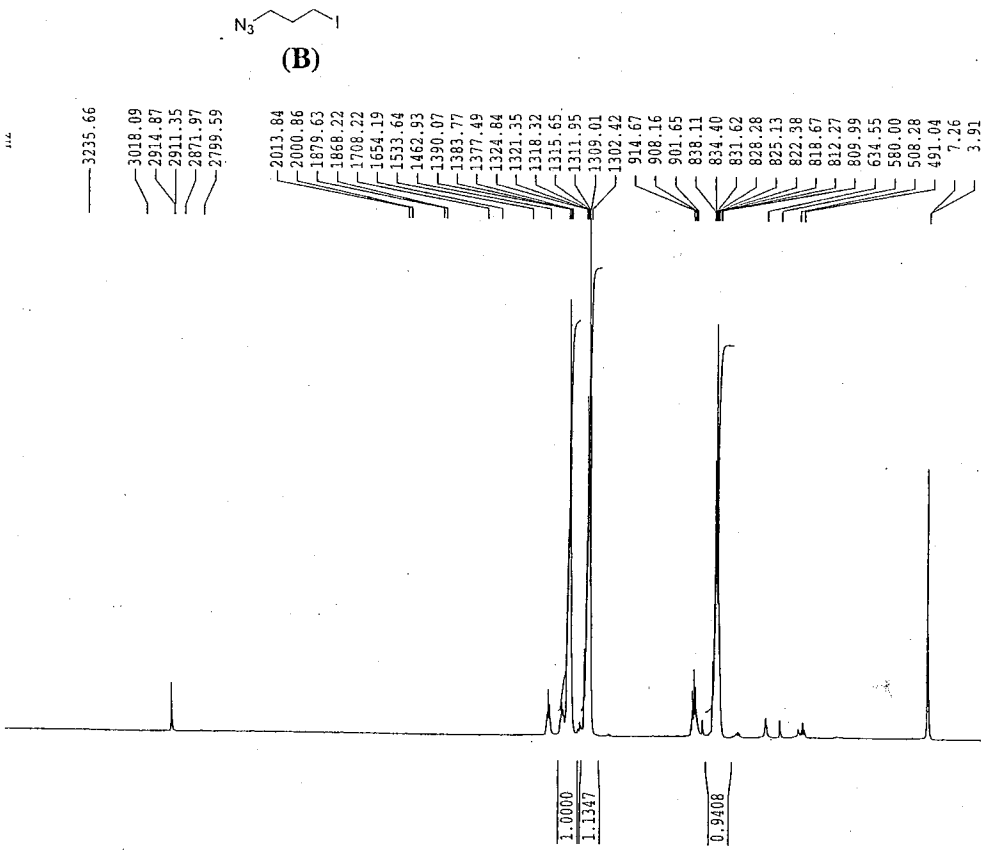


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PROCNO                1

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PROBHD     5 mm Multinu
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
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DW               104.400 usec
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D1            1.00000000 sec

============ CHANNEL f1 =============
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PL1               -6.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
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SSB                   0
LB                 0.30 Hz
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PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
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F2P              -0.500 ppm
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HZCM         190.06175 Hz/cm
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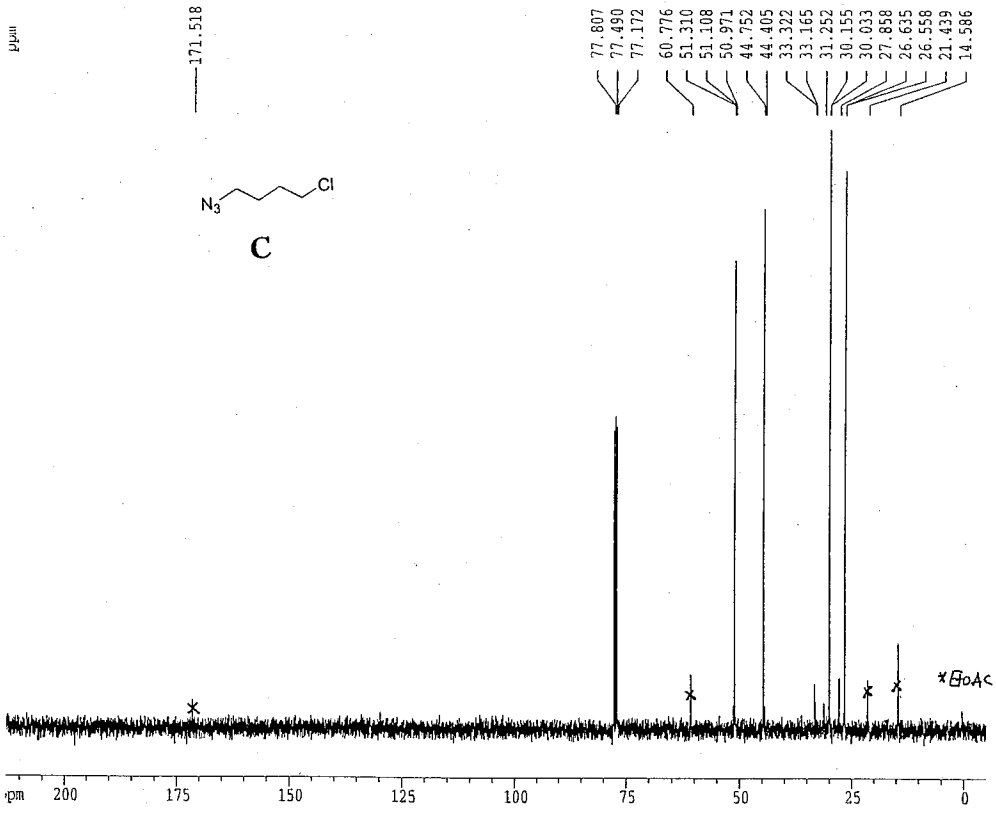


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EXPNO                       1
PROCNO                      1

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PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                         74
DS                          2
SWH                23148.146 Hz
FIDRES              0.353213 Hz
AQ                  1.4156276 sec
RG                      32768
DW                     21.600 usec
DE                      4.50 usec
TE                     300.0 K
D1                 0.05000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 ============
NUC1                      13C
P1                      12.30 usec
PL1                      2.00 dB
SFO1               100.6232933 MHz

============ CHANNEL f2 ============
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NUC2                       1H
PCPD2                  100.00 usec
PL2                      0.00 dB
PL12                    18.00 dB
PL13                    18.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
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SF                 100.6127290 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                   215.000 ppm
F1                  21631.74 Hz
F2P                    -5.000 ppm
F2                   -503.06 Hz
PPMCM                11.00000 ppm/cm
HZCM                 1106.73999 Hz/cm
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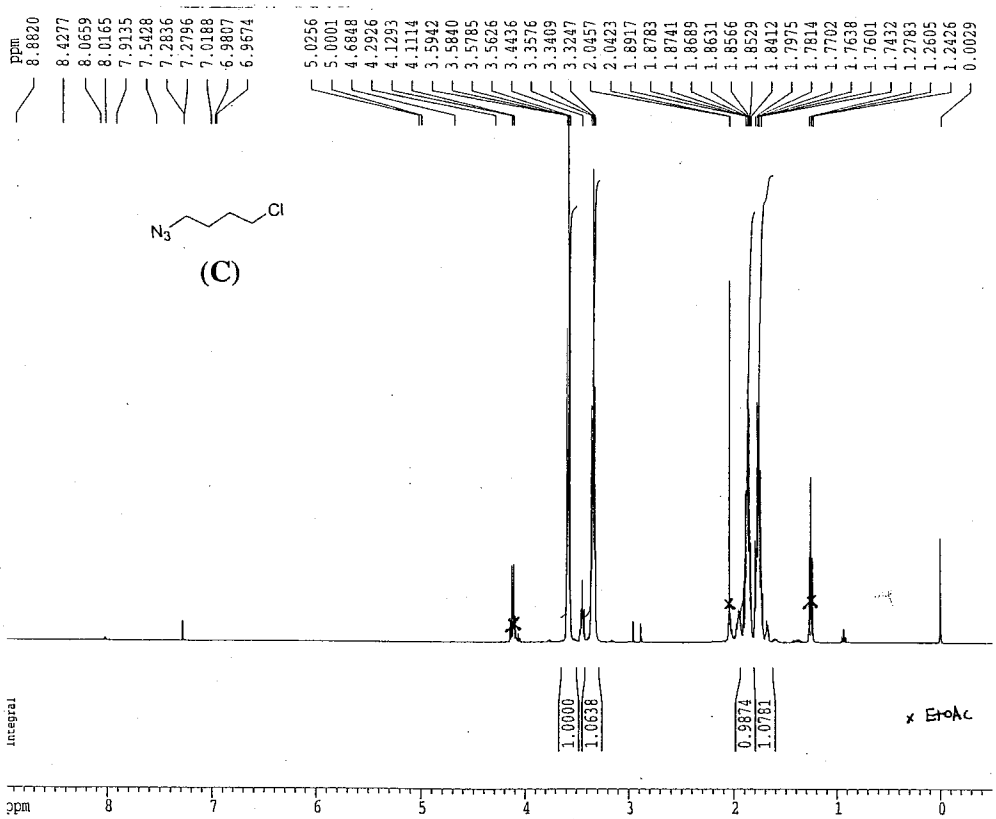


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PROCNO                      1

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Time                   12.05
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PULPROG                 zg30
TD                      32768
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                 4789.272 Hz
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TE                     300.0 K
D1                 1.00000000 sec

============ CHANNEL f1 ============
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PL1                    -6.00 dB
SFO1               400.1320007 MHz

F2 - Processing parameters
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SSB                         0
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PC                      1.00

1D NMR plot parameters
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F1P                     9.000 ppm
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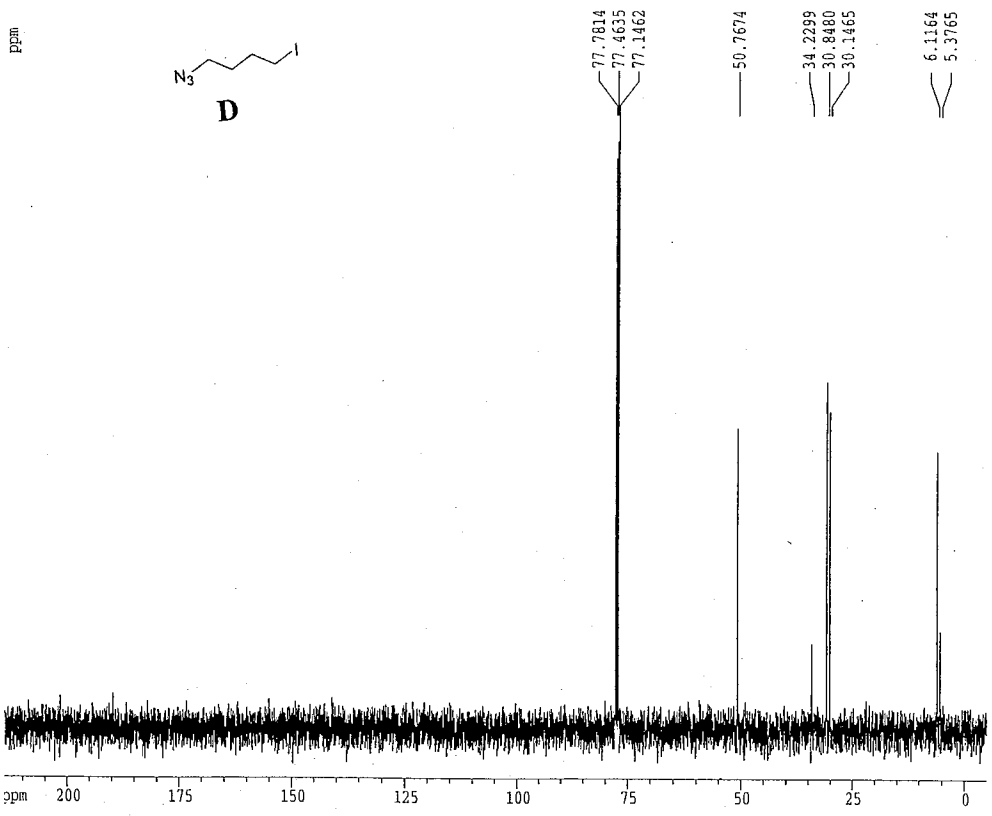

D

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PROCNO                 1

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PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   36
DS                    2
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ             1.4156276 sec
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DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1             0.05000000 sec
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d12            0.00002000 sec

============ CHANNEL f1 ============
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P1                12.30 usec
PL1                2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 ============
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NUC2                1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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SF           100.6127290 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
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F1             21631.74 Hz
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HZCM         1106.73999 Hz/cm
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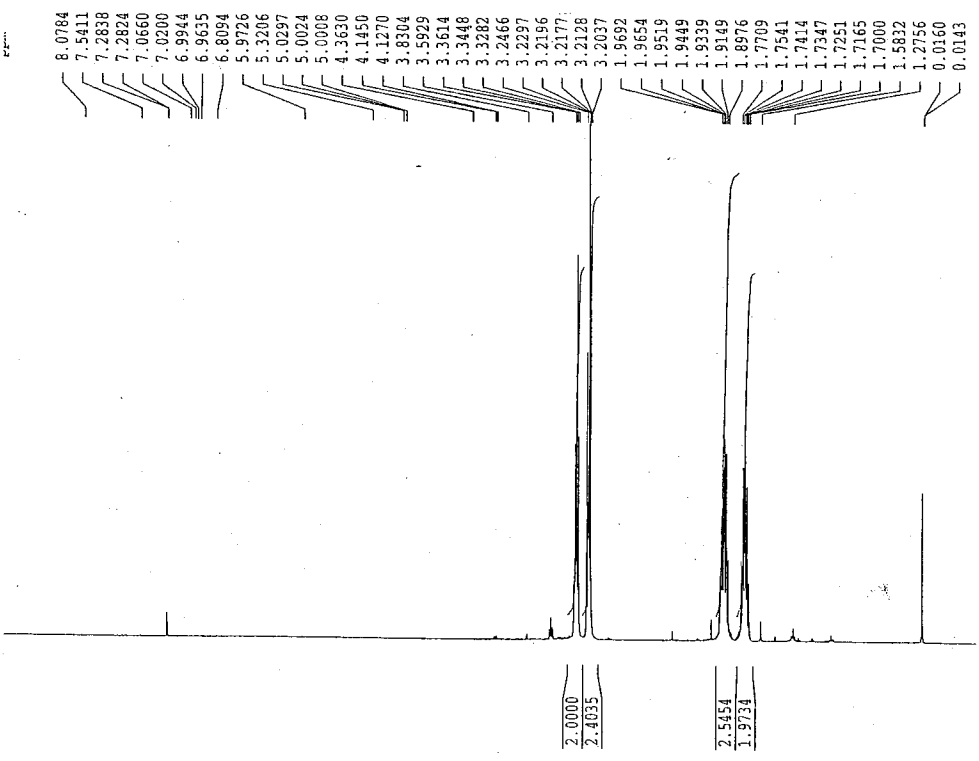
(D)

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PROCNO                 1

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NS                   16
DS                    2
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D1             1.00000000 sec

============ CHANNEL f1 ============
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SFO1         400.1320007 MHz

F2 - Processing parameters
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WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
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F1             3601.17 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
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HZCM         190.06175 Hz/cm
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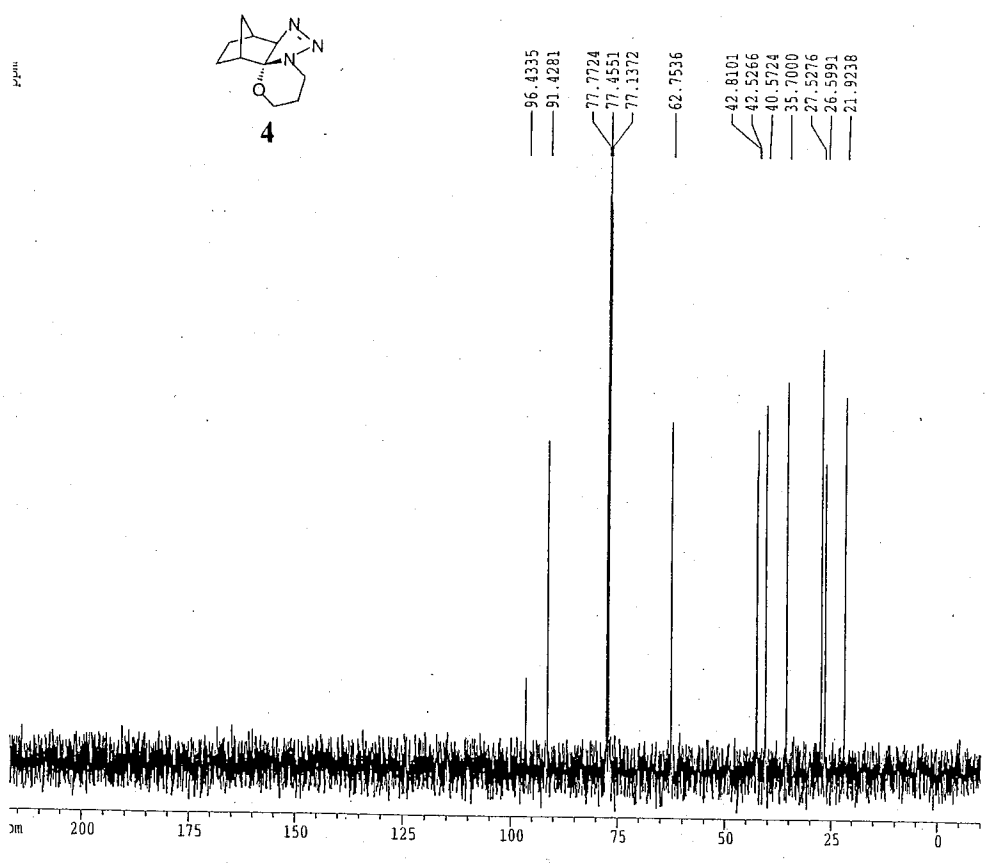


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PROCNO                     1

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NS                      16
DS                       2
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DE                   4.50 usec
TE                  300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                   12.30 usec
PL1                   2.00 dB
SFO1          100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 18.00 dB
PL13                 18.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
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SF            100.6127290 MHz
WDW                     EM
SSB                      0
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GB                       0
PC                    1.40

1D NMR plot parameters
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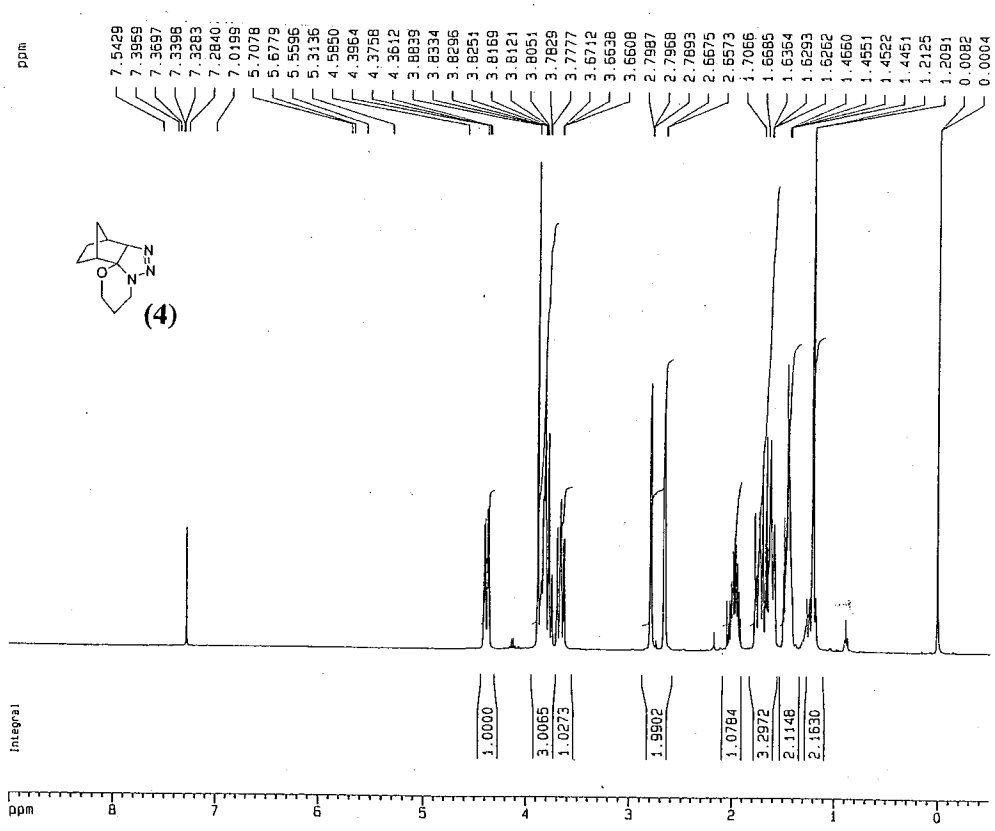


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PROCNO                    1

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TD                    32768
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NS                       16
DS                        2
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FIDRES             0.146157 Hz
AQ                3.4210291 sec
RG                      90.5
DW                 104.400 usec
DE                   4.50 usec
TE                  300.0 K
D1                1.00000000 sec
P1                    7.70 usec
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NUC1                    1H
PL1                  -6.00 dB

F2 - Processing parameters
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SF            400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                9.000 ppm
F1                 3601.17 Hz
F2P                -0.500 ppm
F2                 -200.07 Hz
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HZCM               190.06175 Hz/cm
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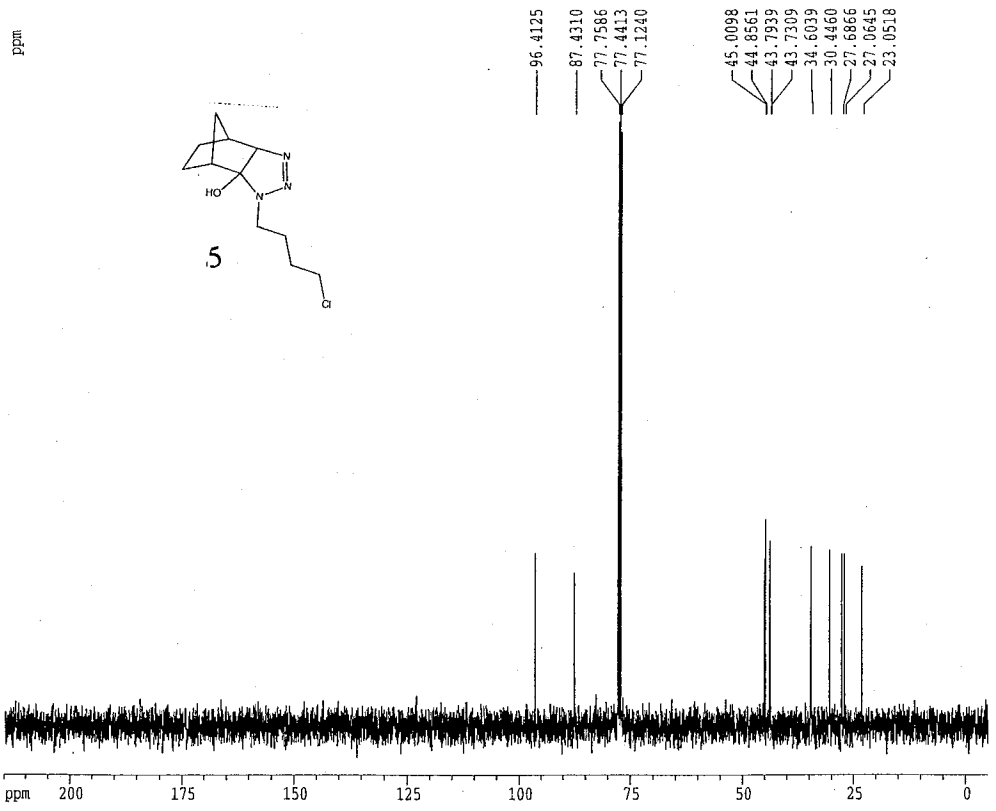


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PROCNO                 1

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TD                65536
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NS                   73
DS                    2
SWH           23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE               300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                12.30 usec
PL1                2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
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NUC2                  1H
PCPD2           100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
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F1             21631.74 Hz
F2P               -5.000 ppm
F2              -501.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
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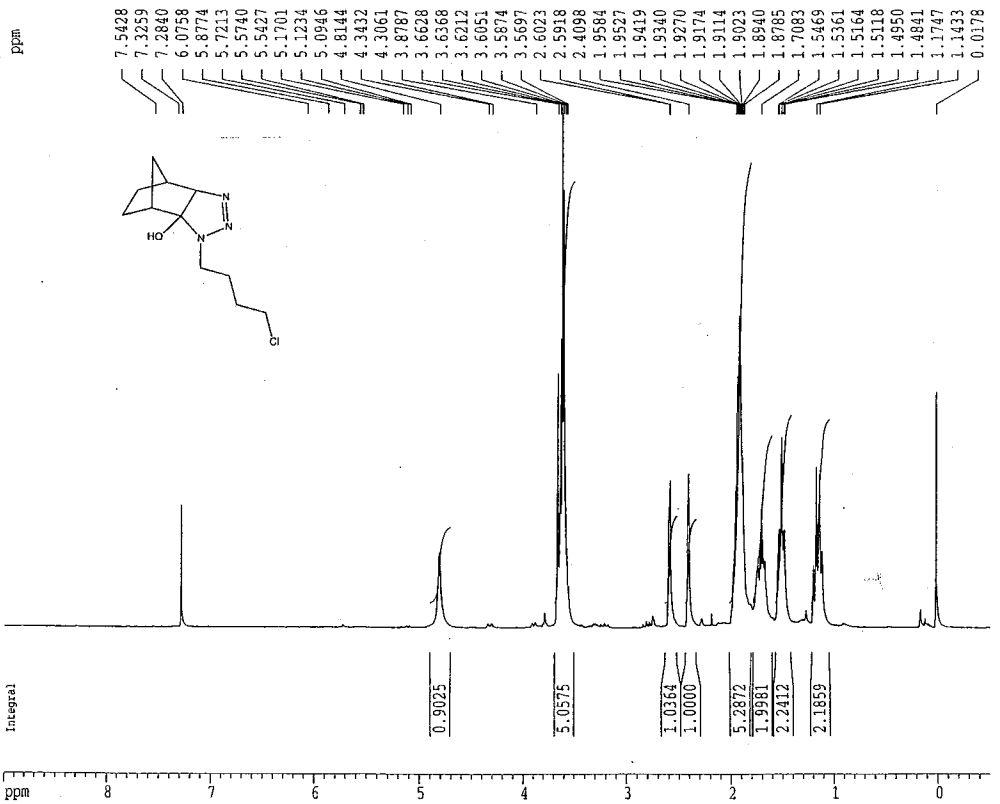


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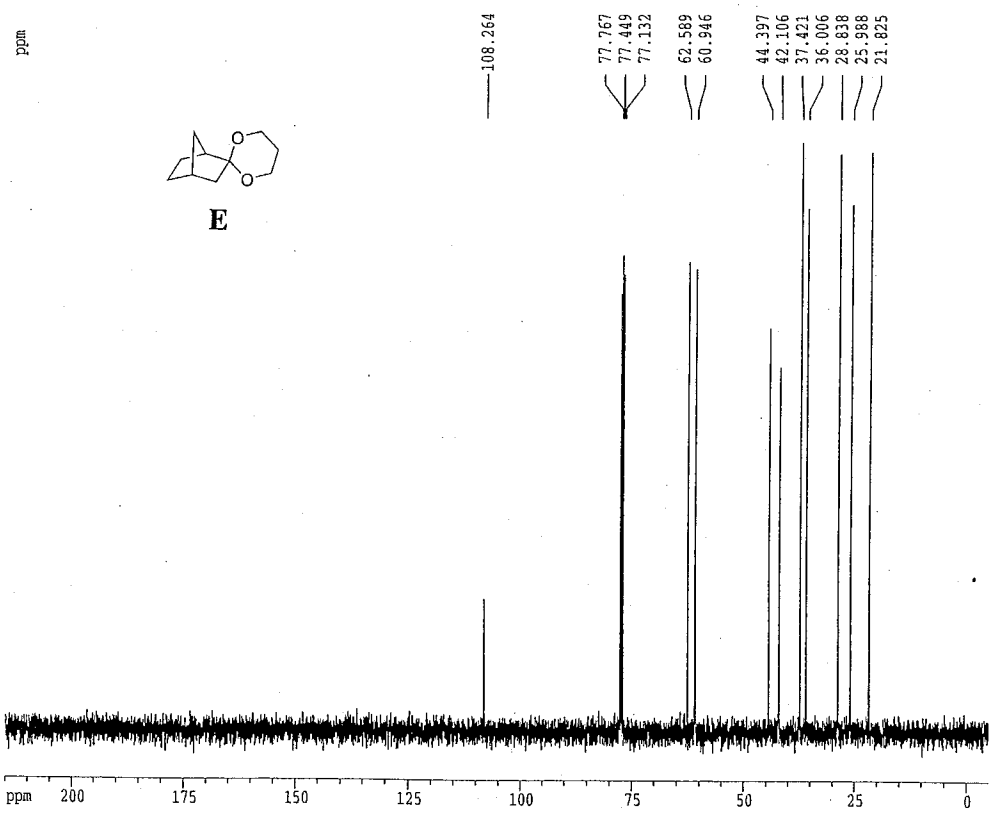
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                161.3
DW               104.400 usec
DE                4.50 usec
TE               300.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                7.70 usec
PL1               -6.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P              -0.500 ppm
F2              -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM         190.06175 Hz/cm
```




```
Current Data Parameters
NAME            yl-1-p229
EXPNO                   2
PROCNO                  1

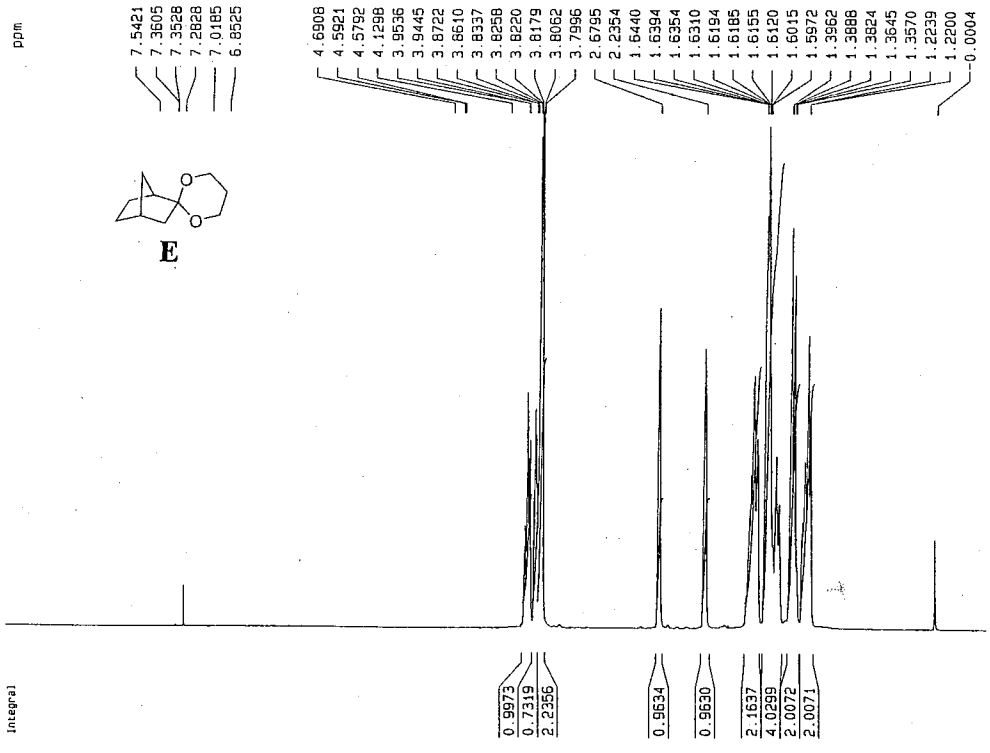
F2 - Acquisition Parameters
Date_          20020814
Time              12.18
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  90
DS                   2
SWH          23148.148 Hz
FIDRES           0.353213 Hz
AQ           1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE               300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1               13C
P1               12.30 usec
PL1               2.00 dB
SFO1        100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2               0.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127290 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX              20.00 cm
F1P            215.000 ppm
F1           21631.74 Hz
F2P             -5.000 ppm
F2            -503.06 Hz
PPMCM         11.00000 ppm/cm
HZCM        1106.73999 Hz/cm
```

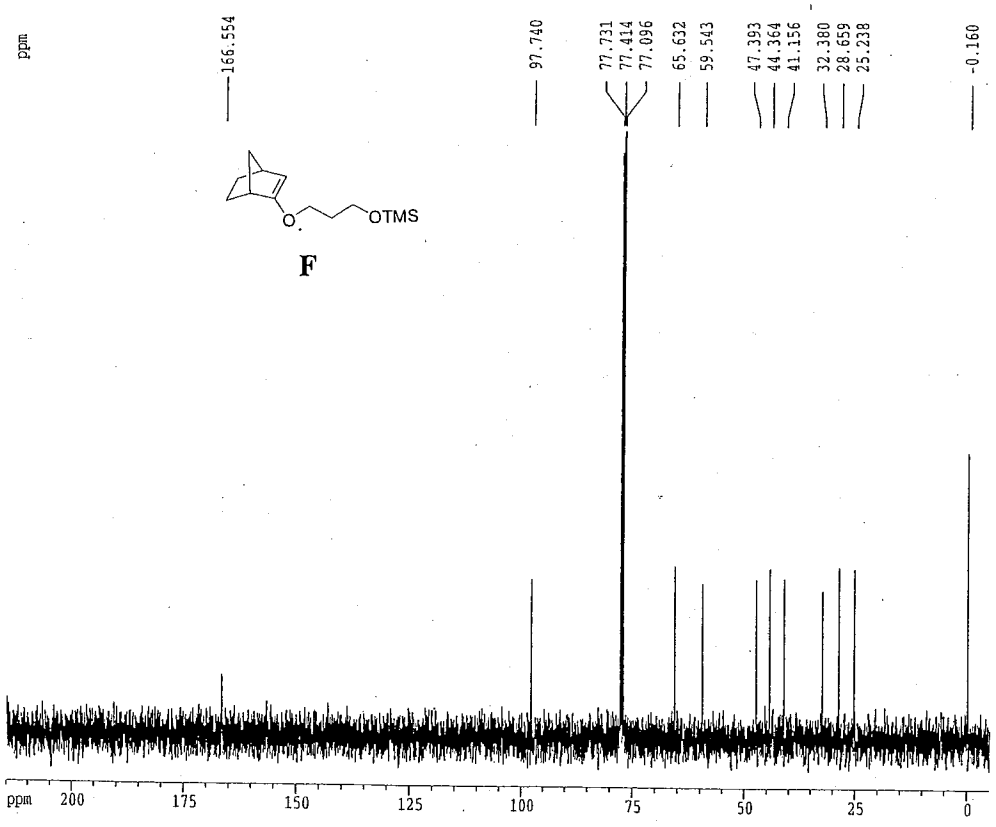



```
Current Data Parameters
NAME            yl-1-p229
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          1020814
Time              12.14
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           4789.272 Hz
FIDRES           0.146157 Hz
AQ           3.4210291 sec
RG                  64
DW              104.400 usec
DE                4.50 usec
TE               300.0 K
D1            1.00000000 sec
P1               7.70 usec
DE                4.50 usec
SFO1        400.1320007 MHz
NUC1                1H
PL1              -6.00 dB

F2 - Processing parameters
SI               16384
SF          400.1300000 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00

1D NMR plot parameters
CX              20.00 cm
F1P             9.000 ppm
F1           3601.17 Hz
F2P             -0.500 ppm
F2            -200.07 Hz
PPMCM         0.47500 ppm/cm
HZCM        190.06175 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p239
EXPNO                 2
PROCNO                1

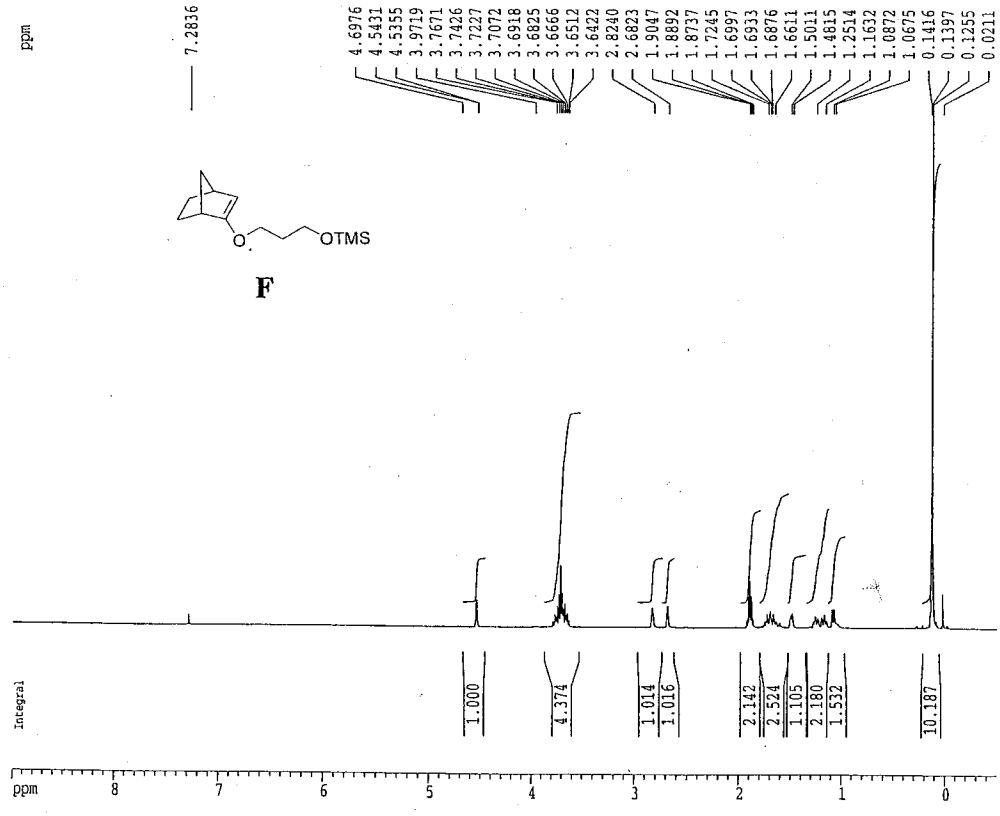
F2 - Acquisition Parameters
Date_         20020822
Time             12.24
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   61
DS                    2
SWH          23148.148 Hz
FIDRES        0.353211 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 ============
NUC1                 13C
P1                12.30 usec
PL1                2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
```



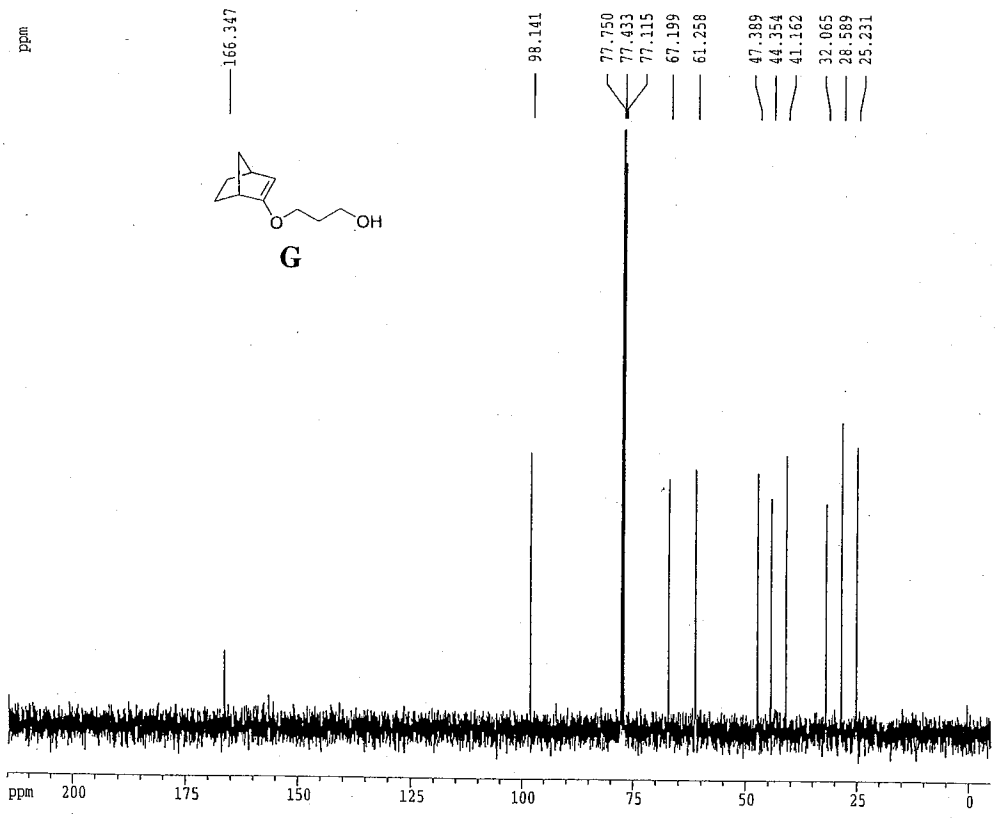

```
Current Data Parameters
NAME          yl-1-p239
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_         20020822
Time             12.21
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                143.7
DW              104.400 usec
DE                4.50 usec
TE                300.0 K
D1            1.00000000 sec

============ CHANNEL f1 ============
NUC1                 1H
P1                7.70 usec
PL1               -6.00 dB
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                16384
SF           400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P              -0.500 ppm
F2             -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM         190.06175 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p233
EXPNO                 2
PROCNO                1

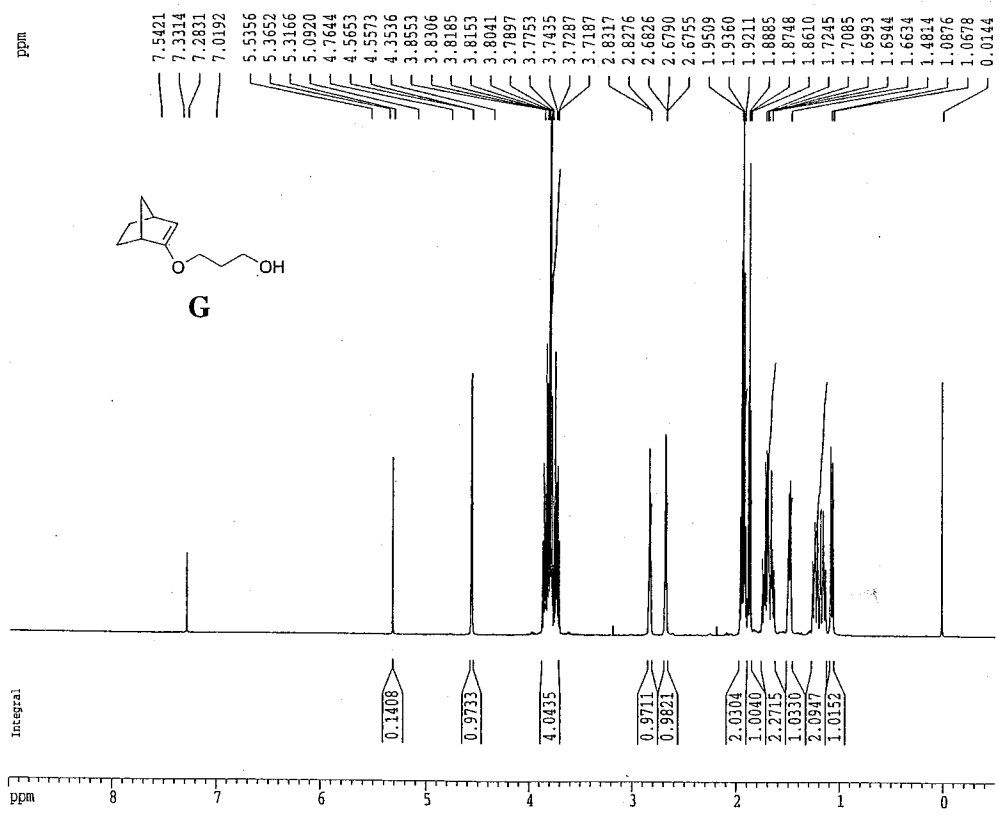
F2 - Acquisition Parameters
Date_          20020816
Time              13.37
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   54
DS                    2
SWH           23148.148 Hz
FIDRES            0.353213 Hz
AQ             1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1             0.05000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                 13C
P1                 12.30 usec
PL1                 2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 0.00 dB
PL12               18.00 dB
PL13               18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
```



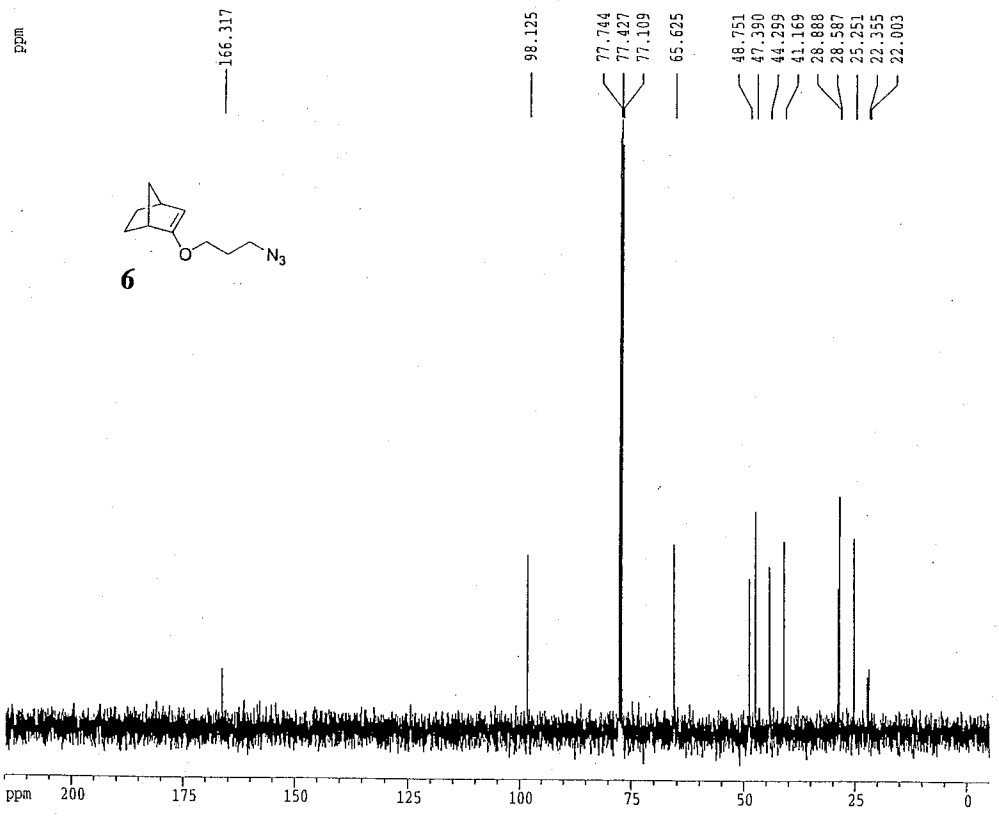

```
Current Data Parameters
NAME          yl-1-p233
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20020816
Time              13.35
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            4789.272 Hz
FIDRES            0.146157 Hz
AQ             3.4210291 sec
RG                101.6
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1             1.00000000 sec

============ CHANNEL f1 ============
NUC1                 1H
P1                 7.70 usec
PL1                -6.00 dB
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                16384
SF           400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P              -0.500 ppm
F2             -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM         190.06175 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p242
EXPNO                 4
PROCNO                1

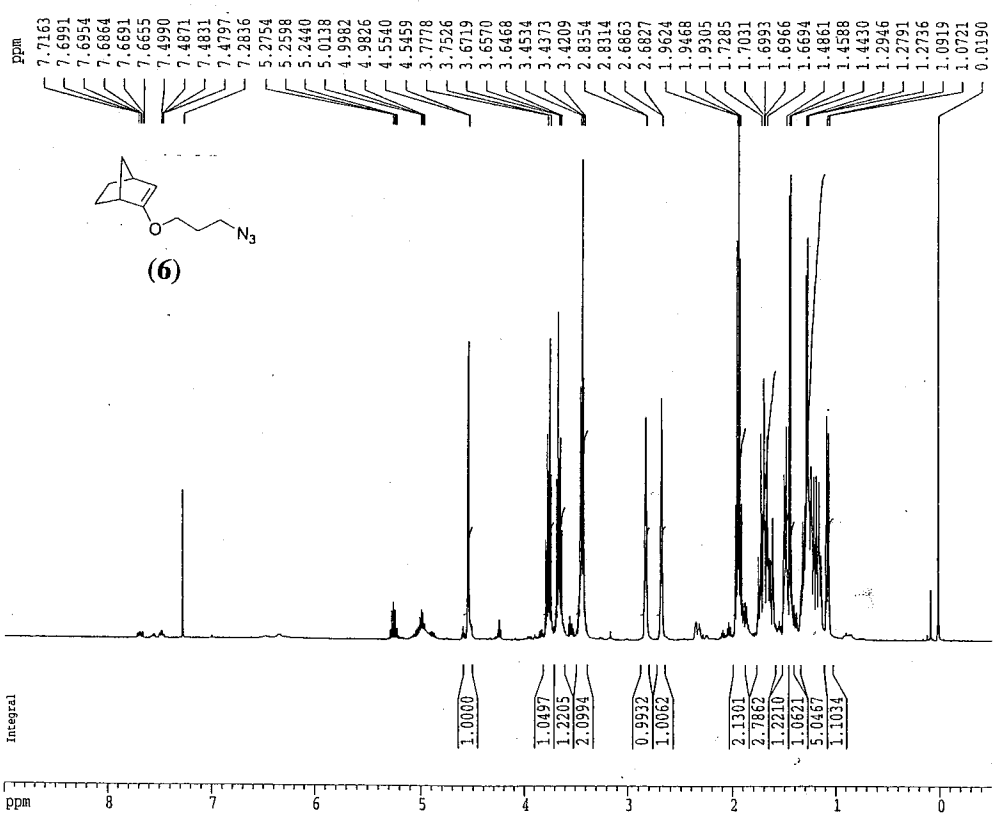
F2 - Acquisition Parameters
Date_          20020823
Time              15.07
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   77
DS                    2
SWH           23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

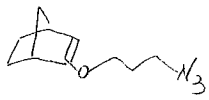
============ CHANNEL f1 =============
NUC1                 13C
P1                 12.30 usec
PL1                 2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 0.00 dB
PL12               18.00 dB
PL13               18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1              21631.74 Hz
F2P               -5.000 ppm
F2               -503.06 Hz
PPMCM            11.00000 ppm/cm
HZCM          1106.73999 Hz/cm
```





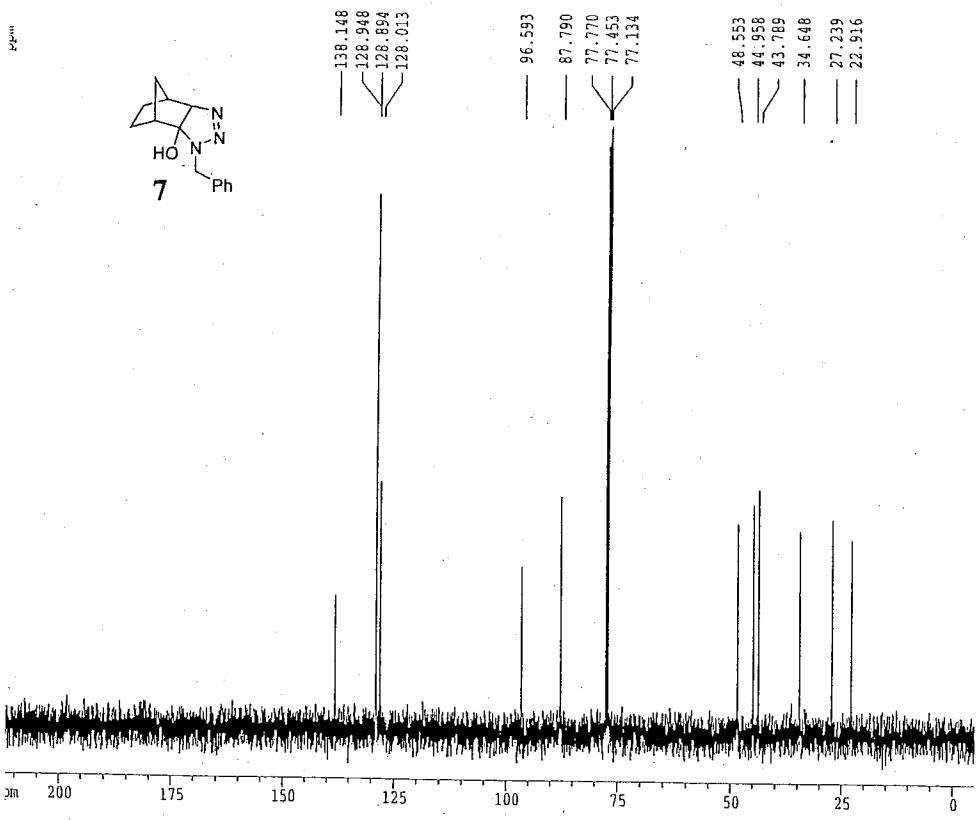

```
Current Data Parameters
NAME          yl-1-p242
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20020823
Time              15.04
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG           zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                  128
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1            1.00000000 sec

============ CHANNEL f1 =============
NUC1                 1H
P1                 7.70 usec
PL1                -6.00 dB
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2              -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM          190.06175 Hz/cm
```




```
Current Data Parameters
NAME            yl-1-p102
EXPNO                   2
PROCNO                  1

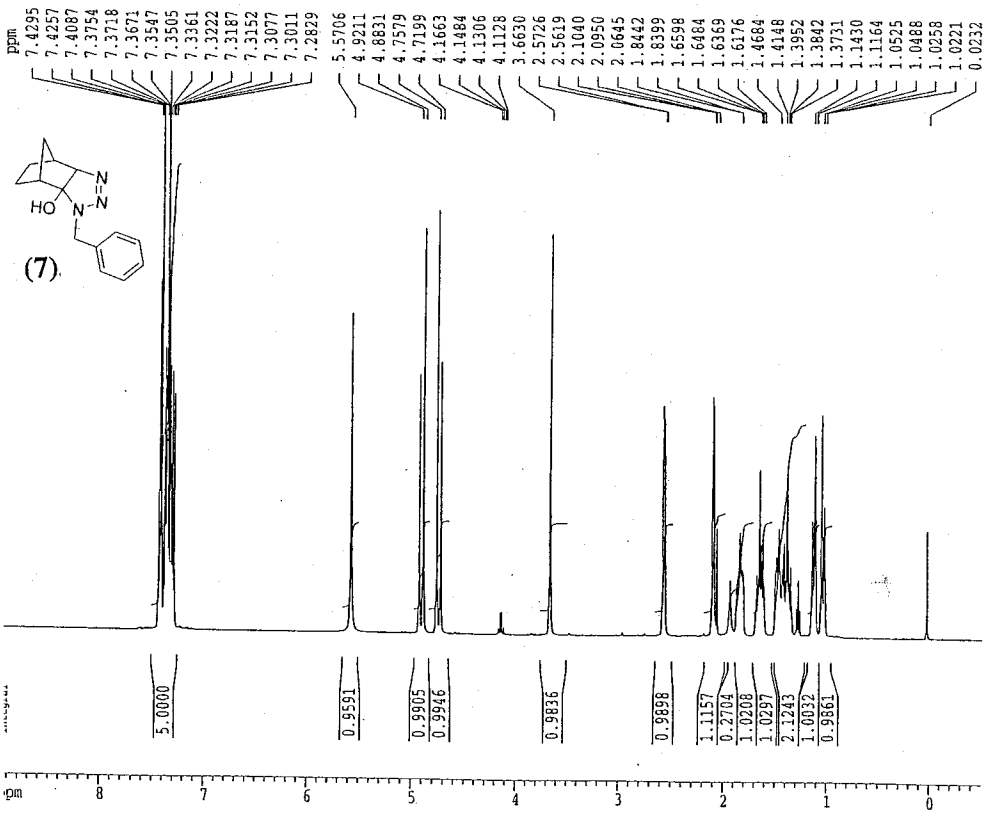
F2 - Acquisition Parameters
Date_           20020415
Time               12.21
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   55
DS                    2
SWH           23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               12.30 usec
PL1               2.00 dB
SFO1        100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               0.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM           1106.73999 Hz/cm
```



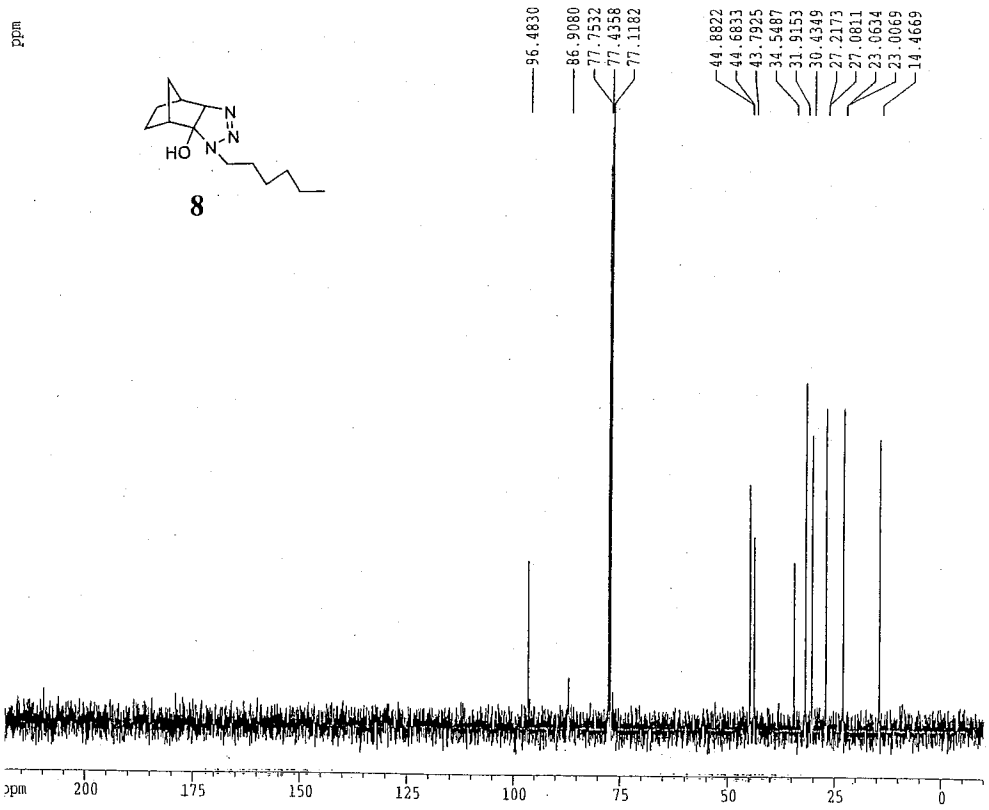

```
Current Data Parameters
NAME            yl-1-p102
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20020415
Time               12.18
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG             zg30
TD                12768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                101.6
DW              104.400 usec
DE                4.50 usec
TE                300.0 K
D1            1.00000000 sec

============ CHANNEL f1 ============
NUC1                1H
P1                7.70 usec
PL1               -6.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P              -0.500 ppm
F2             -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM           190.06175 Hz/cm
```




```
Current Data Parameters
NAME                  yl-1-p287
EXPNO                         4
PROCNO                        1

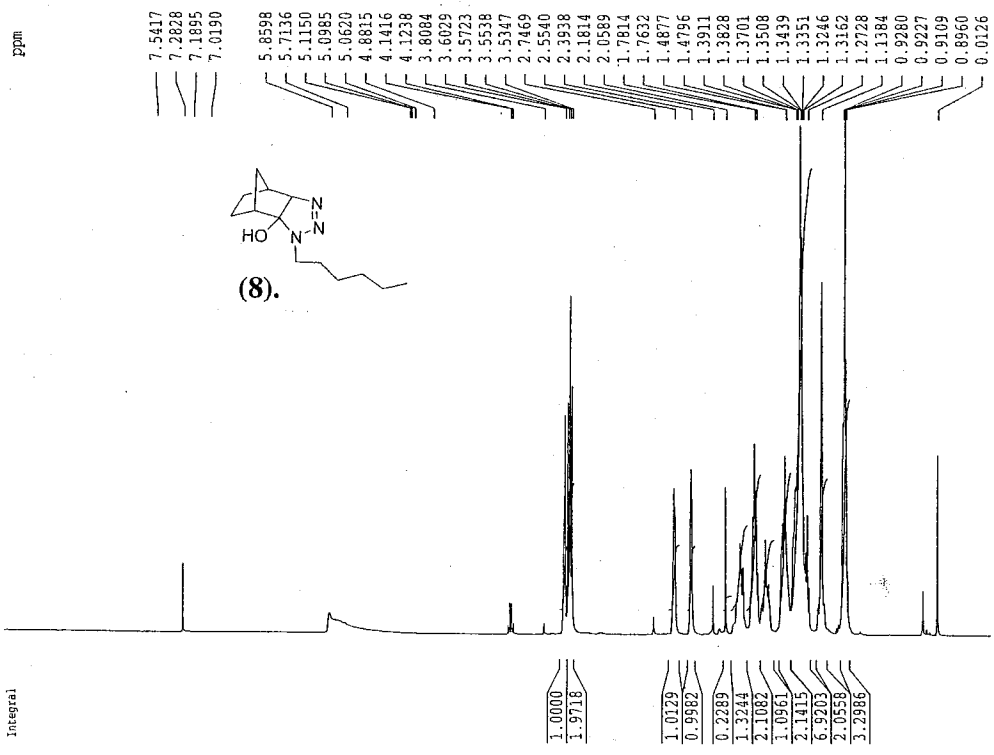
F2 - Acquisition Parameters
Date_                 20021101
Time                      15.13
INSTRUM                  drx400
PROBHD     5 mm Multinucl
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         117
DS                           2
SWH                 23148.148 Hz
FIDRES              0.353213 Hz
AQ                  1.4156276 sec
RG                       32768
DW                      21.600 usec
DE                       4.50 usec
TE                      300.0 K
D1                  0.05000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                       12.30 usec
PL1                       2.00 dB
SFO1               100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                       0.00 dB
PL12                     18.00 dB
PL13                     18.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127290 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
CY                       12.50 cm
F1P                    220.036 ppm
F1                   22138.42 Hz
F2P                    -10.036 ppm
F2                   -1009.73 Hz
PPMCM                11.50359 ppm/cm
HZCM                1157.40747 Hz/cm
```



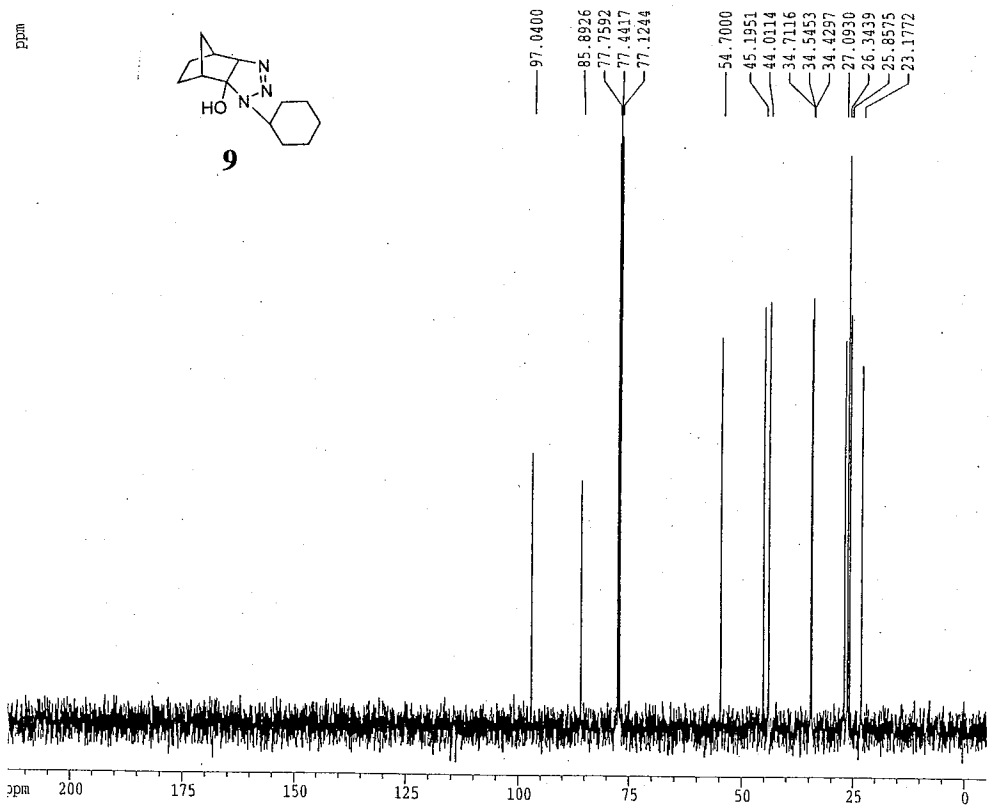

```
Current Data Parameters
NAME                  yl-1-p287
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20021101
Time                      15.10
INSTRUM                  drx400
PROBHD     5 mm Multinucl
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 4789.272 Hz
FIDRES              0.146157 Hz
AQ                  3.4210291 sec
RG                        90.5
DW                     104.400 usec
DE                       4.50 usec
TE                      300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                        7.70 usec
PL1                      -6.00 dB
SFO1               400.1320007 MHz

F2 - Processing parameters
SI                       16384
SF                 400.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       12.50 cm
F1P                      9.000 ppm
F1                    3601.17 Hz
F2P                     -0.500 ppm
F2                    -200.07 Hz
PPMCM                 0.47500 ppm/cm
HZCM                190.06175 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-pl37
EXPNO                 2
PROCNO                1

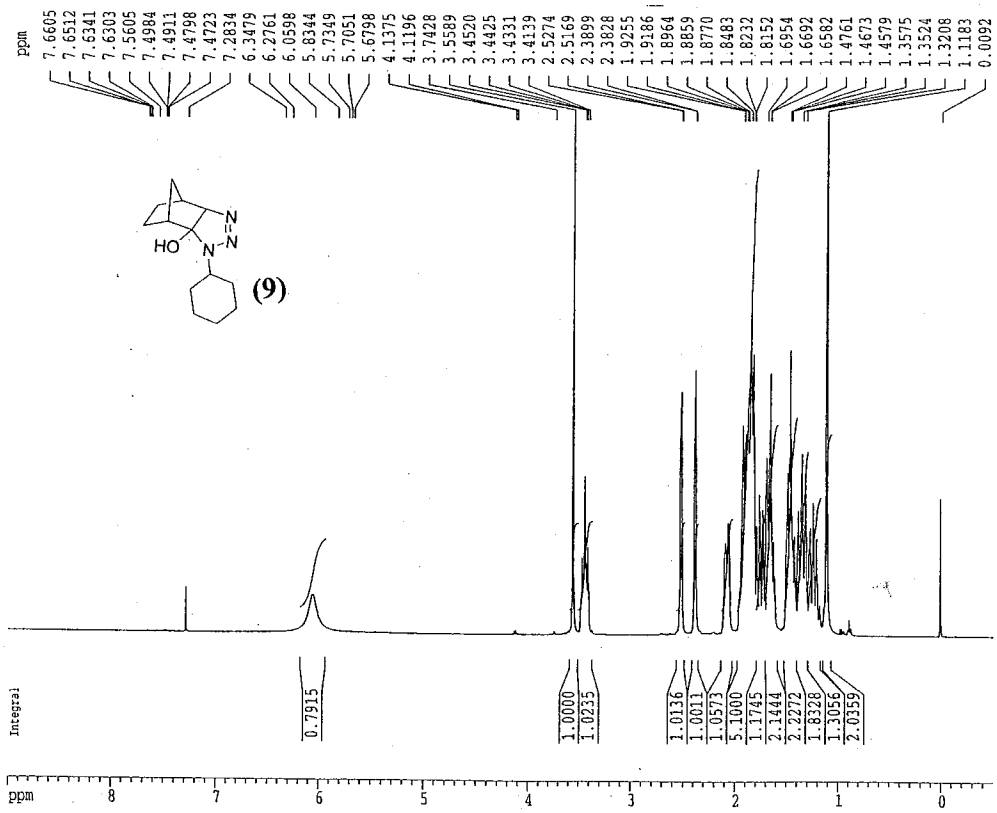
F2 - Acquisition Parameters
Date_          20020615
Time              10.49
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   70
DS                    2
SWH          23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1           0.05000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

============ CHANNEL f1 =============
NUC1                 13C
P1                 12.30 usec
PL1                 2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1            21631.74 Hz
F2P               -5.000 ppm
F2             -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
```



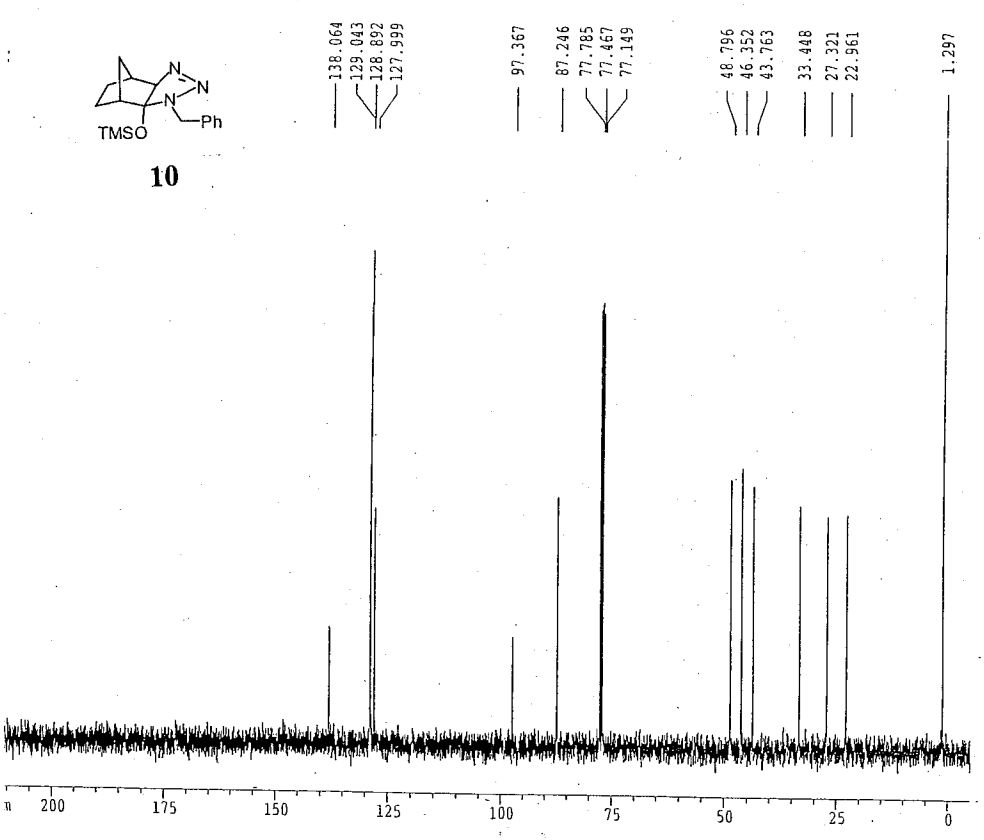

```
Current Data Parameters
NAME          yl-1-pl37
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20020615
Time              10.44
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG           zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                   64
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1           1.00000000 sec

============ CHANNEL f1 =============
NUC1                 1H
P1                 7.70 usec
PL1               -6.00 dB
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P                9.000 ppm
F1            3601.17 Hz
F2P               -0.500 ppm
F2             -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM         190.06175 Hz/cm
```




```
Current Data Parameters
NAME                  yl-1-p91
EXPNO                        2
PROCNO                       1

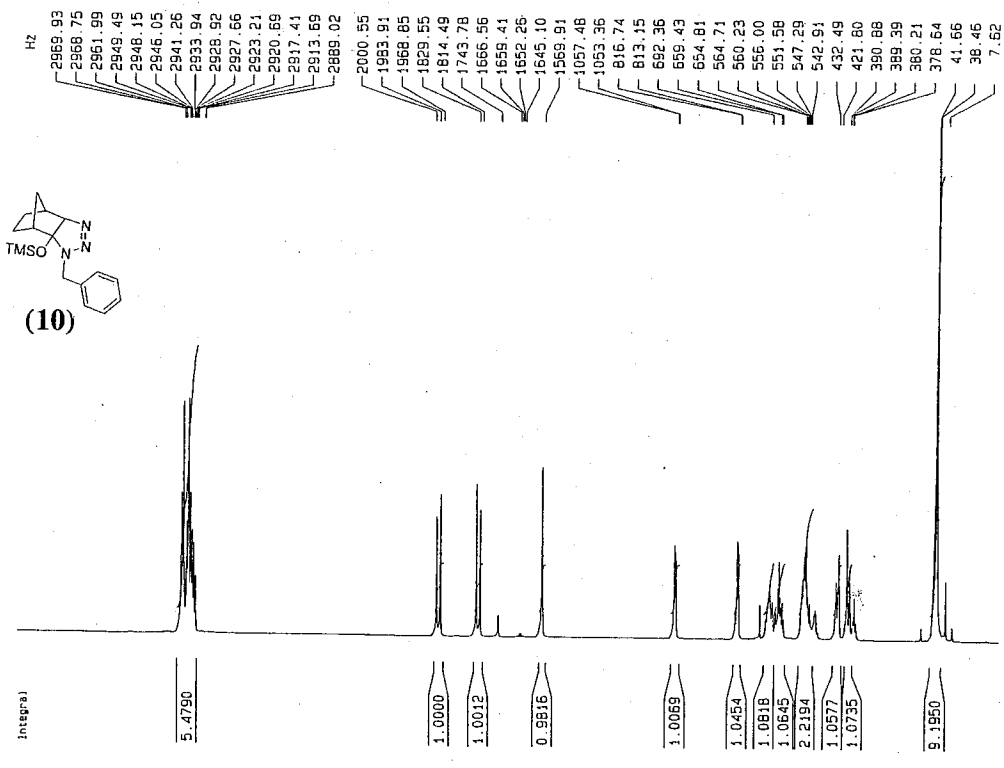
F2 - Acquisition Parameters
Date_               20020326
Time                    15.55
INSTRUM                 drx400
PROBHD       5 mm Multinu
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          69
DS                           2
SWH                 23148.148 Hz
FIDRES               0.353213 Hz
AQ                   1.4156276 sec
RG                       32768
DW                      21.600 usec
DE                       4.50 usec
TE                      300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                        13C
P1                       12.30 usec
PL1                       2.00 dB
SFO1              100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                       0.00 dB
PL12                     18.00 dB
PL13                     18.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127290 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                    21631.74 Hz
F2P                      -5.000 ppm
F2                     -503.06 Hz
PPMCM                  11.00000 ppm/cm
HZCM                1106.73999 Hz/cm
```

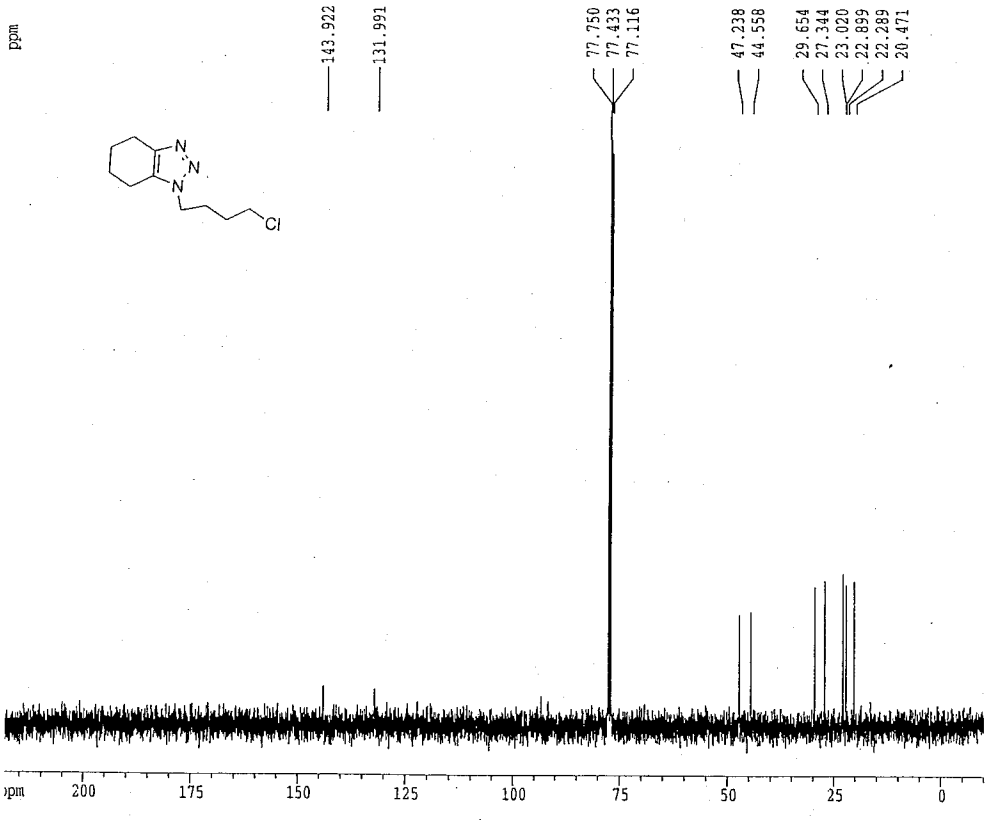



```
Current Data Parameters
NAME                  yl-1-p91
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               1020326
Time                    15.49
INSTRUM                 drx400
PROBHD       5 mm Multinu
PULPROG                 zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 4789.272 Hz
FIDRES               0.146157 Hz
AQ                   3.4210291 sec
RG                          64
DW                     104.400 usec
DE                       4.50 usec
TE                      300.0 K
D1                1.00000000 sec
P1                       7.70 usec
DE                       4.50 usec
SFO1              400.1320007 MHz
NUC1                        1H
PL1                      -6.00 dB

F2 - Processing parameters
SI                       16384
SF                400.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                      9.000 ppm
F1                    3601.17 Hz
F2P                      -0.500 ppm
F2                     -200.07 Hz
PPMCM                  0.47500 ppm/cm
HZCM                190.06175 Hz/cm
```




```
Current Data Parameters
NAME        yl-III-Predo
EXPNO                  4
PROCNO                 1

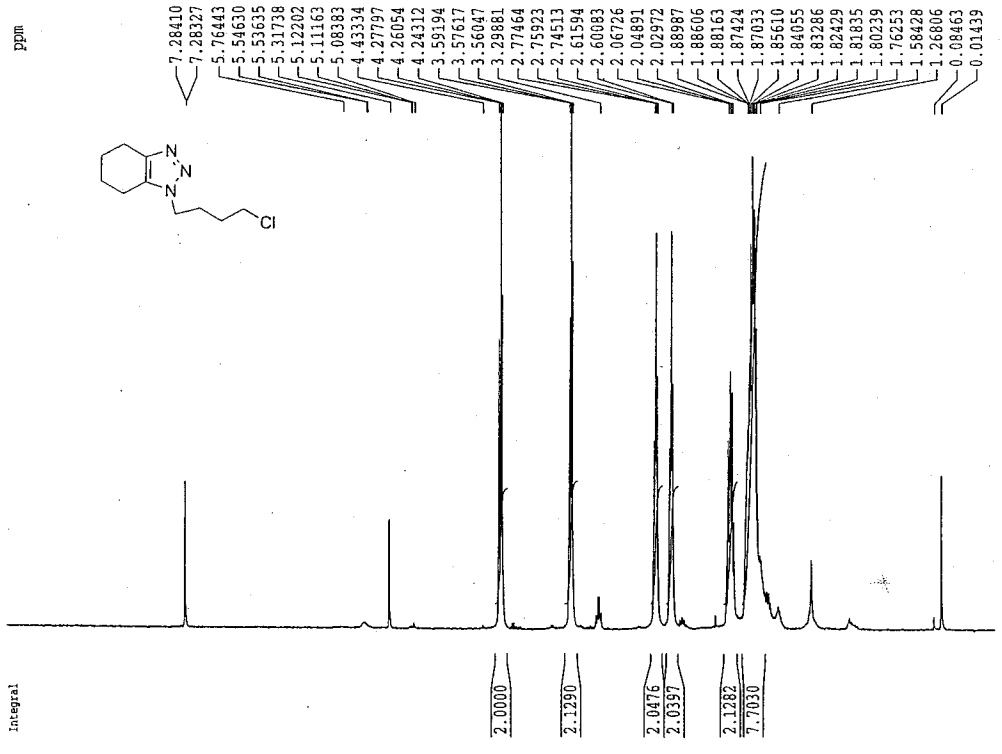
F2 - Acquisition Parameters
Date_          20030730
Time              16.45
INSTRUM          drx400
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  204
DS                    2
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1           0.05000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                12.30 usec
PL1                2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P              220.036 ppm
F1             22138.42 Hz
F2P              -10.036 ppm
F2             -1009.73 Hz
PPMCM          11.50359 ppm/cm
HZCM         1157.40747 Hz/cm
```



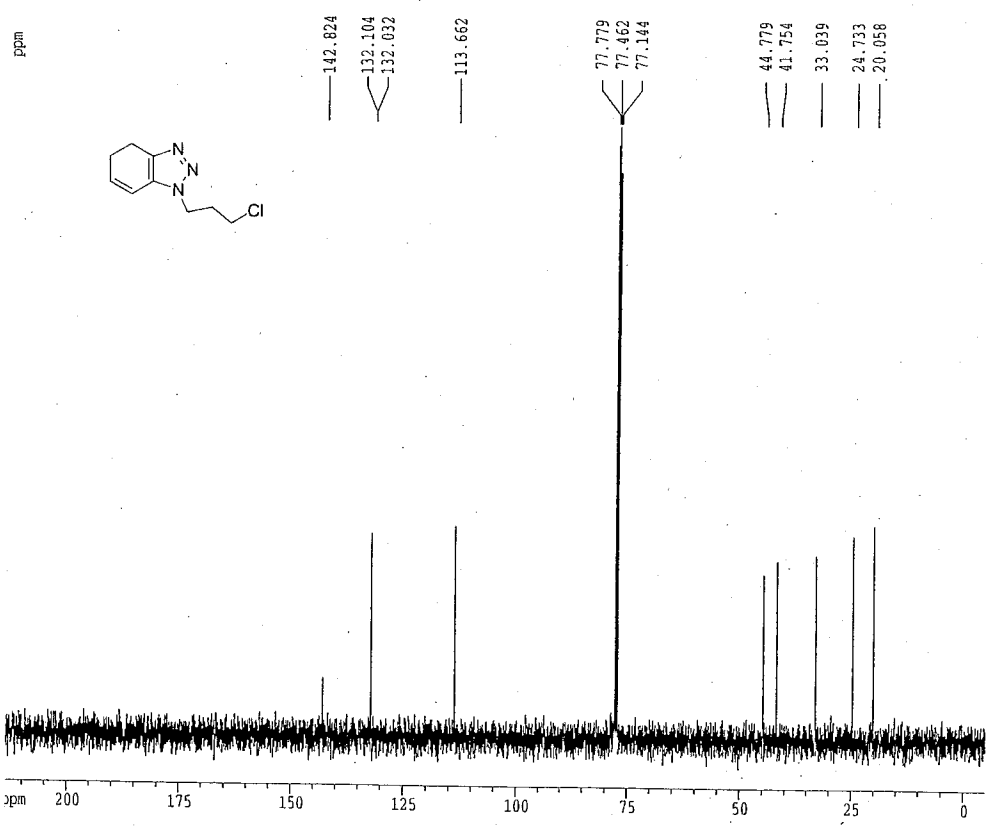

```
Current Data Parameters
NAME        yl-III-Predo
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_          20030730
Time              16.42
INSTRUM          drx400
PROBHD   5 mm Multinucl
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            4789.272 Hz
FIDRES         0.146157 Hz
AQ            3.4210291 sec
RG                143.7
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.70 usec
PL1               -6.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P               -0.500 ppm
F2             -200.07 Hz
PPMCM          0.47500 ppm/cm
HZCM         190.06175 Hz/cm
```




```
Current Data Parameters
NAME                  yl-1-p144
EXPNO                         2
PROCNO                        1

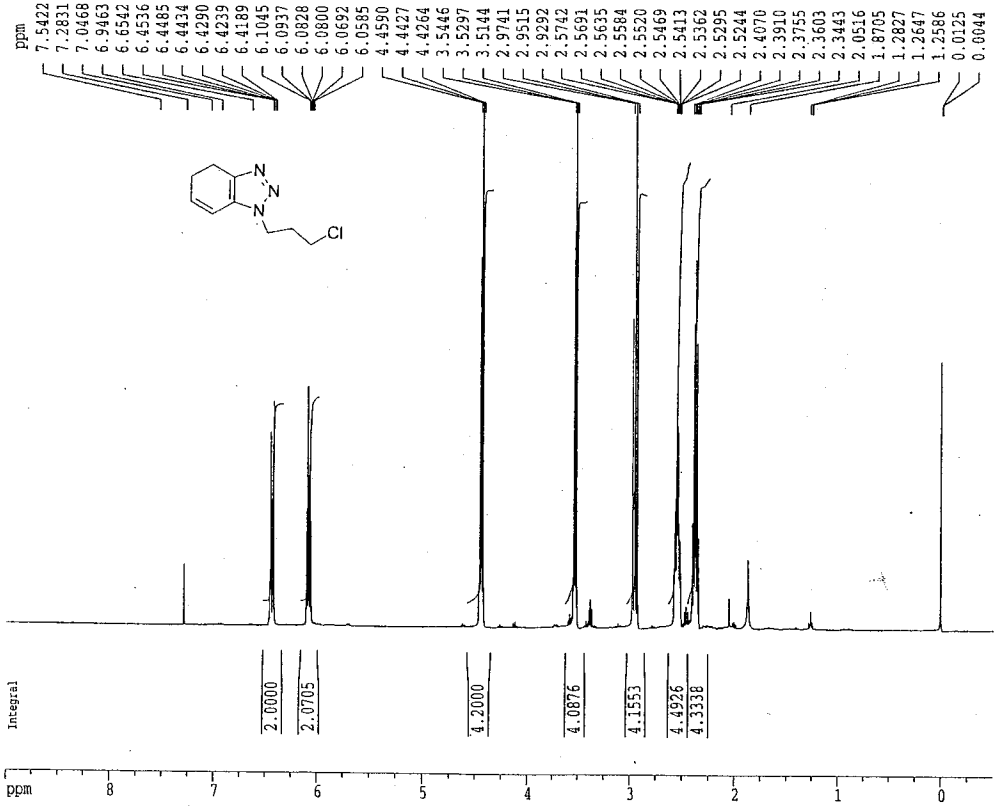
F2 - Acquisition Parameters
Date_                  20020614
Time                      13.24
INSTRUM                  drx400
PROBHD       5 mm Multinu
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          92
DS                           2
SWH                 23148.148 Hz
FIDRES                0.353213 Hz
AQ                    1.4156276 sec
RG                        32768
DW                       21.600 usec
DE                        4.50 usec
TE                       300.0 K
D1                    0.05000000 sec
d11                   0.03000000 sec
d12                   0.00002000 sec

============ CHANNEL f1 =============
NUC1                        13C
P1                        12.30 usec
PL1                        2.00 dB
SFO1                100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   100.00 usec
PL2                       0.00 dB
PL12                     18.00 dB
PL13                     18.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127290 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      215.000 ppm
F1                     21631.74 Hz
F2P                       -5.000 ppm
F2                      -503.06 Hz
PPMCM                  11.00000 ppm/cm
HZCM                1106.73999 Hz/cm
```



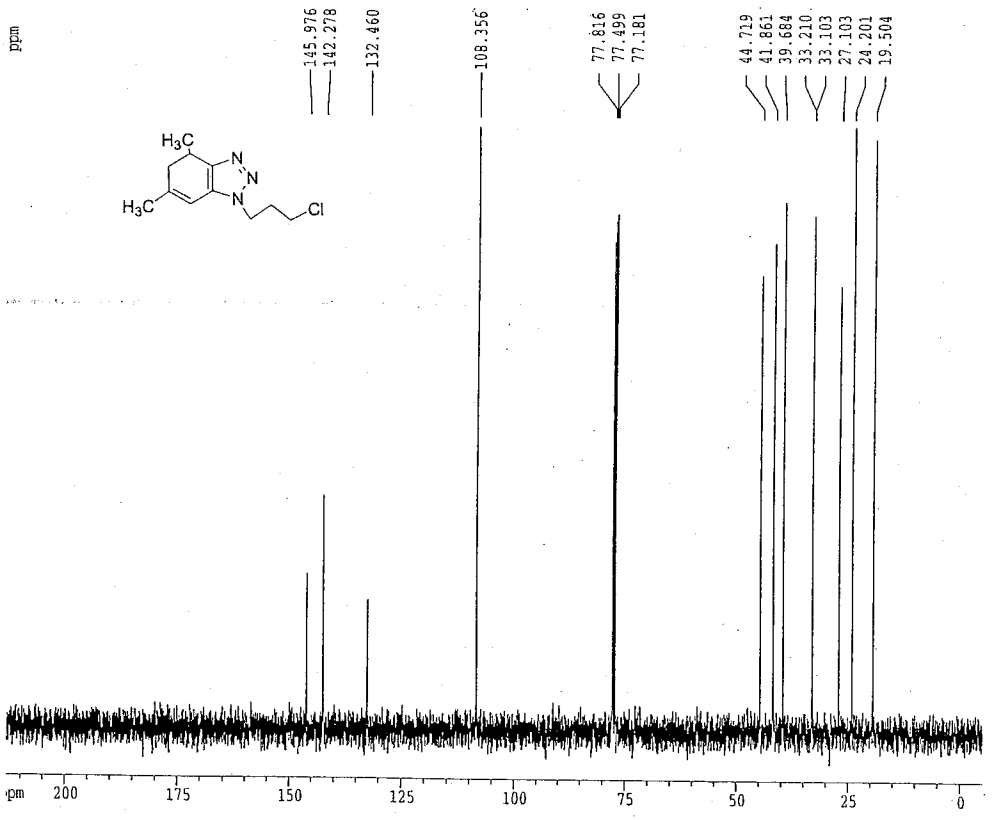

```
Current Data Parameters
NAME                  yl-1-p144
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20020614
Time                      13.22
INSTRUM                  drx400
PROBHD       5 mm Multinu
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                  4789.272 Hz
FIDRES                0.146157 Hz
AQ                    3.4210291 sec
RG                          114
DW                       104.400 usec
DE                        4.50 usec
TE                       300.0 K
D1                    1.00000000 sec

============ CHANNEL f1 =============
NUC1                         1H
P1                         7.70 usec
PL1                       -6.00 dB
SFO1                400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF                  400.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        20.00 cm
F1P                       9.000 ppm
F1                     3601.17 Hz
F2P                      -0.500 ppm
F2                      -200.07 Hz
PPMCM                  0.47500 ppm/cm
HZCM                 190.06175 Hz/cm
```




```
Current Data Parameters
NAME            yl-1-p146
EXPNO                   2
PROCNO                  1

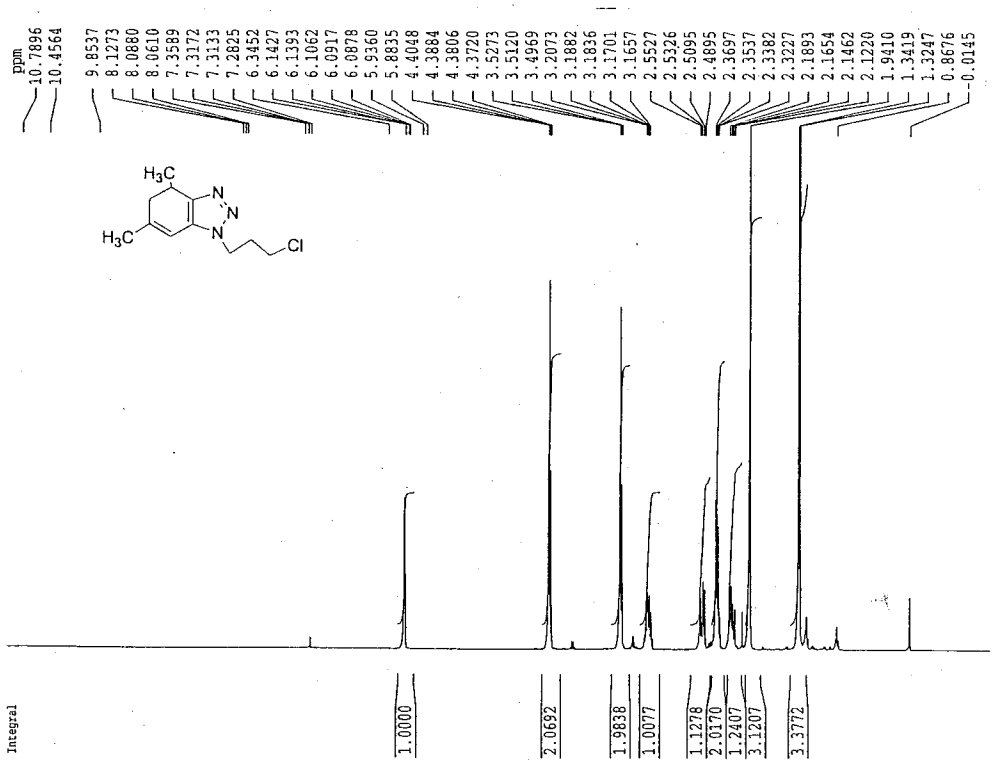
F2 - Acquisition Parameters
Date_         20020617
Time              11.34
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  75
DS                   2
SWH          23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                1280
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1            0.05000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                12.30 usec
PL1                2.00 dB
SFO1        100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM        1106.73999 Hz/cm
```



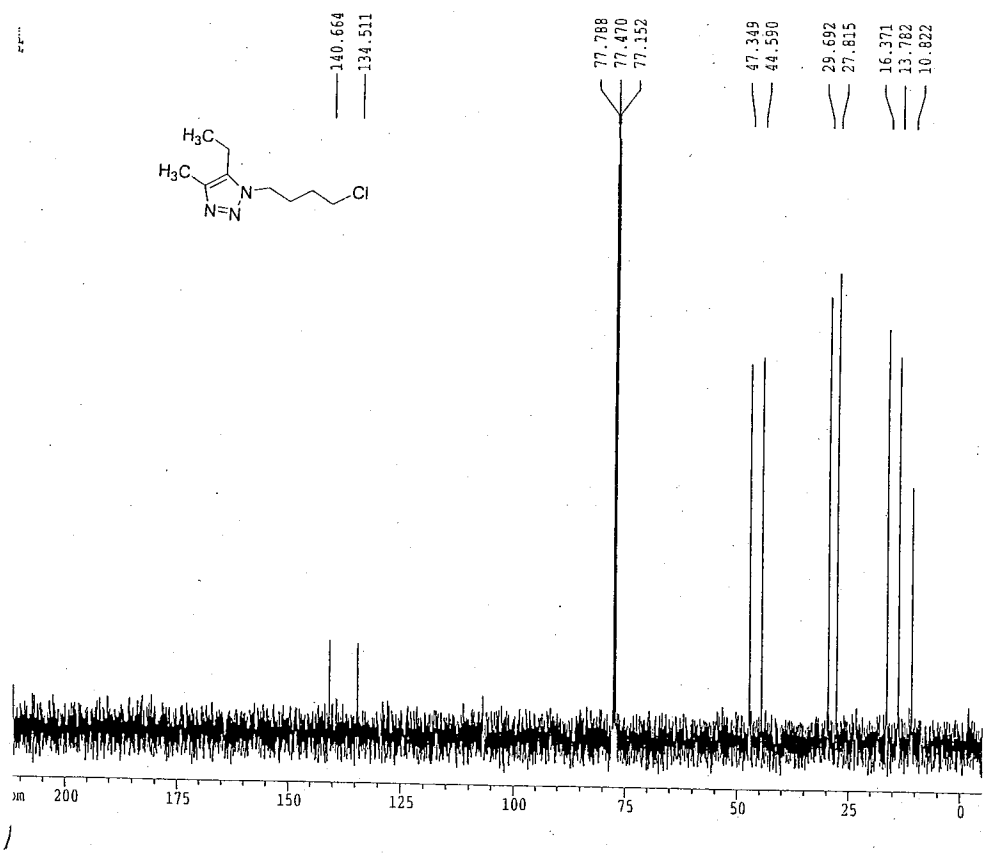

```
Current Data Parameters
NAME            yl-1-p146
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_         20020617
Time              11.31
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                  64
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1            1.00000000 sec

============ CHANNEL f1 ============
NUC1                1H
P1                7.70 usec
PL1               -6.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
F1P              11.000 ppm
F1             4401.43 Hz
F2P               -1.000 ppm
F2              -400.13 Hz
PPMCM          0.60000 ppm/cm
HZCM        240.07800 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p41b2c
EXPNO                   1
PROCNO                  1

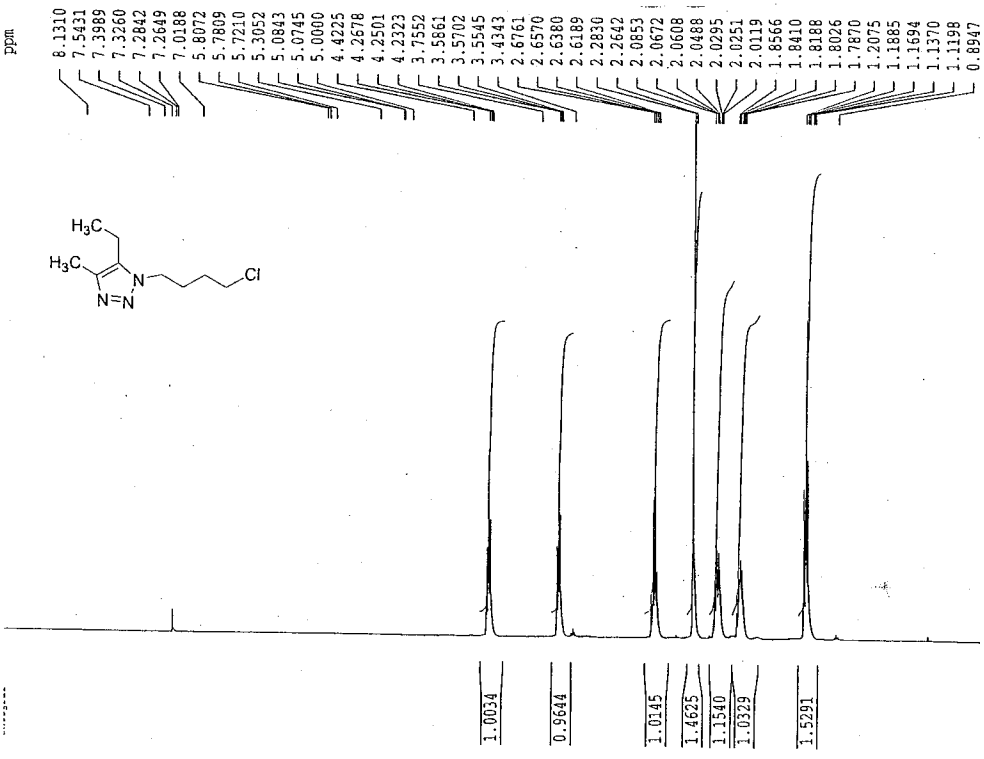
F2 - Acquisition Parameters
Date_            20020129
Time                18.25
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   81
DS                    2
SWH          23148.148 Hz
FIDRES        0.353213 Hz
AQ            1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1           0.05000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                12.30 usec
PL1                2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF          100.6127290 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
F1P                215.000 ppm
F1               21631.74 Hz
F2P                -5.000 ppm
F2                -503.06 Hz
PPMCM             11.00000 ppm/cm
HZCM          1106.73999 Hz/cm
```



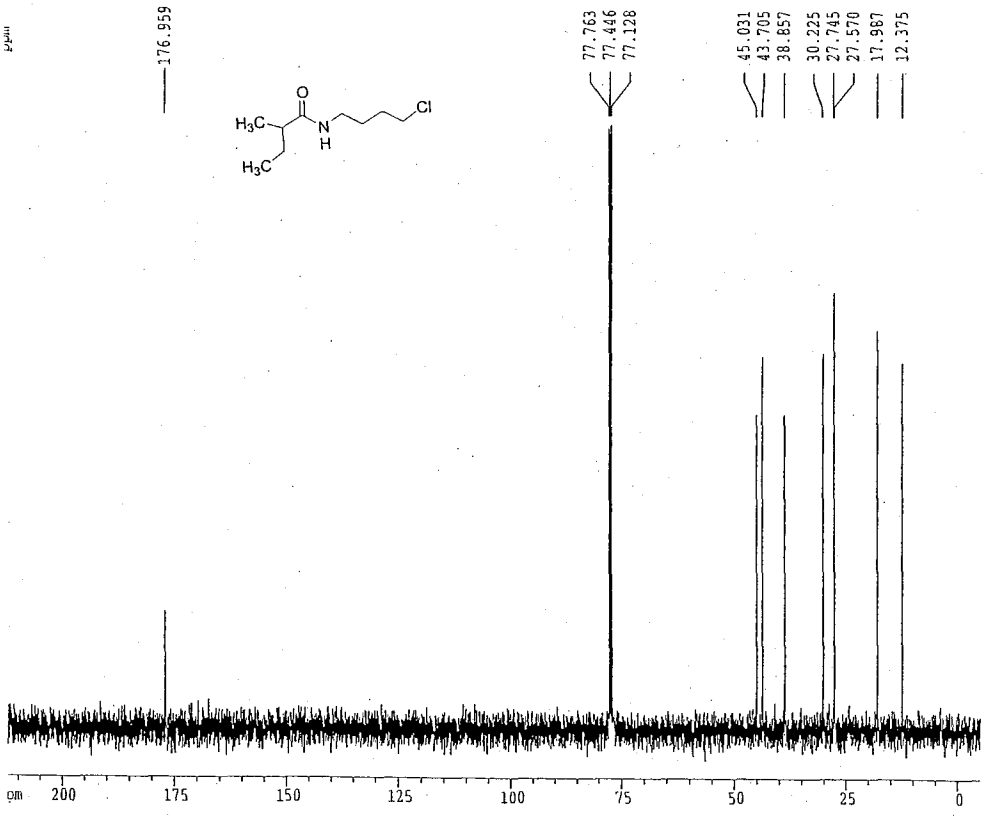

```
Current Data Parameters
NAME          yl-1-p41b2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20020129
Time                18.20
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                   64
DW              104.400 usec
DE                4.50 usec
TE                300.0 K
D1           1.00000000 sec

============ CHANNEL f1 ============
NUC1                1H
P1                7.70 usec
PL1               -6.00 dB
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                  16384
SF          400.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
F1P                 9.000 ppm
F1               3601.17 Hz
F2P                -0.500 ppm
F2               -200.07 Hz
PPMCM             0.47500 ppm/cm
HZCM          190.06175 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p41b1c
EXPNO                   1
PROCNO                  1

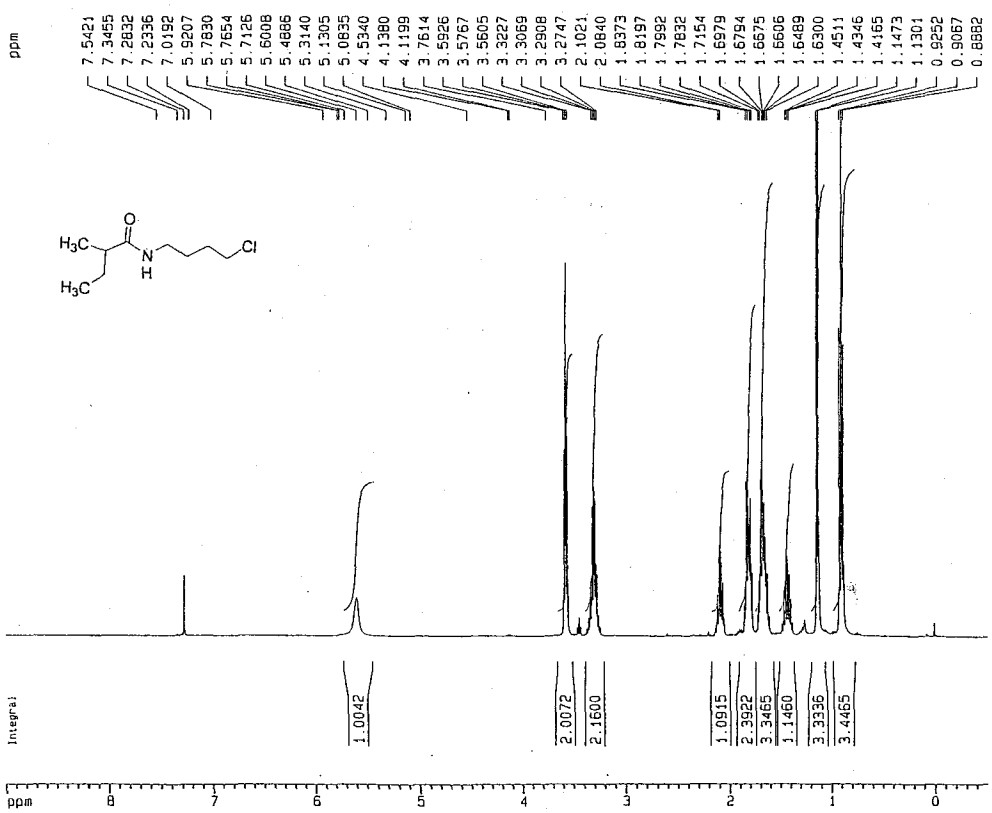
F2 - Acquisition Parameters
Date_           20020129
Time               17.53
INSTRUM           drx400
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  146
DS                    2
SWH           23148.148 Hz
FIDRES         0.353211 Hz
AQ             1.4156276 sec
RG                32768
DW               21.600 usec
DE                4.50 usec
TE                300.0 K
D1             0.05000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                12.30 usec
PL1                2.00 dB
SFO1         100.6232933 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127290 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM            11.00000 ppm/cm
HZCM          1106.73999 Hz/cm
```




```
Current Data Parameters
NAME          yl-1-p41D1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           1020129
Time              17.49
INSTRUM          drx400
PROBHD     5 mm Multinu
PULPROG           zg30
TD               32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                101.6
DW               104.400 usec
DE                4.50 usec
TE                300.0 K
D1            1.00000000 sec
P1                7.70 usec
DE                4.50 usec
SFO1         400.1320007 MHz
NUC1                1H
PL1               -6.00 dB

F2 - Processing parameters
SI                 16384
SF          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
PPMCM            0.47500 ppm/cm
HZCM          190.06175 Hz/cm
```