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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Supporting Information

Experimental Section	S2
Copies of ¹ H and ¹³ C spectrum of new compounds	S13

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Tel 785.864.4496 Fax 785.864.5326 E-mail: jaube@ku.edu General. Compounds 1 and 2 have been previously reported. 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.

1-Azido-3-chloropropane (**A**). NaN₃ (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 Na₂SO₄ 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. ¹H NMR (400 MHz, CDCl₃) \Box 2.03 (m, 2H), 3.51 (t, J = 6.2 Hz, 2H), 3.65 (t, J = 6.2 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 32.3, 42.0, 48.7; IR (neat) 2100 cm⁻¹; MS (CI) m/z 120 (M⁺+1); HRMS calcd for C₃H₇ClN₃ (M⁺+1): 120.0344, found 120.0355.

$$N_3$$

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 Na₂SO₄ and concentrated to give an oil. Flash chromatography (10% EtOAc/hexane) afforded **B** (461 mg, 85%) as an oil. ¹H NMR (400 MHz, CDCl₃) \square 2.09 (m, 2H), 3.27 (t, J = 6.6 Hz, 2H), 3.46 (t, J = 6.4 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) \square 2.8, 32.7, 51.9; IR (neat) 2098 cm⁻¹; MS (CI) m/z 211 (M⁺+1); HRMS calcd for C₃H₇IN₃ (M⁺+1): 211.9685, found 211.9672.

$$N_3$$
 C

1-Azido-4-chlorobutane (C). ¹H NMR (400 MHz, CDCl₃) \Box 1.76 (m, 2H), 1.87 (m, 2H), 3.36 (t, J = 6.6 Hz, 2H), 3.53 (t, J = 6.5 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 26.6, 30.0, 44.8, 51.1; IR (neat) 2100 cm⁻¹; MS (CI) m/z 134 (M⁺+1); HRMS calcd for C₄H₉ClN₃ (M⁺+1):134.0445, found 134.0428.

1-Azido-4-iodobutane (**D**). ¹H NMR (400 MHz, CDCl₃) \Box 1.73 (m, 2H), 1.91 (m, 2H), 3.23 (t, J = 6.8 Hz, 2H), 3.36 (t, J = 6.6 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 6.1, 30.1, 30.8, 50.8; IR (neat) 2100 cm⁻¹; MS (CI) m/z 225 (M⁺+1); HRMS calcd for C₄H₈IN₃ (M⁺+1): 225.3953 found 225.3939.

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 ketone¹ (117 mg, 0.65 mmol) was added. After stirring for 1 h, 1-azido-4iodobutane **D** (294 mg, 1.31 mmol) was added dropwise. After 30 min at -78 °C, the reaction mixture was allowed to warm to 0 °C for 2 h. The reaction mixture was quenched with saturated NaHCO3, extracted with EtOAc, dried over Na2SO4 and concentrated to give an oil. Flash chromatography (40% EtOAc/hexane) afforded 107 mg (60%) of **3** as an oil: ¹H NMR (500 MHz, CDCl₃) \prod 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, CDCl₃) \prod 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.

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. ¹H NMR (500 MHz, CDCl₃) ☐ 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); ¹³C NMR (500 MHz, CDCl₃) ☐ 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 C₁₀H₁₀N₃O (M⁺+1): 194.1215, found 194.1222.

1-(4'-Chlorobutyl)-7a-hydroxyl-4,7-methano-1*H***-benzotriazole (5).** Prepared using the same procedure as above from norcamphor and alkyl azide \mathbb{C} . Yield 91%. ¹H NMR (500 MHz, CDCl₃) \square 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); ¹³C NMR (100.6 MHz, CDCl₃) \square 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 $\mathbb{C}_{11}H_{18}N_3$ OCl (M⁺+1): 244.1217, found 244.1205.

 \mathbf{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 Gassman² et al. Yield: 90%. Bp 65-66 °C (0.2 mm Hg), ¹H NMR (400 MHz, CDCl₃) [] 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); ¹³C NMR (100.6 MHz, CDCl₃) [] 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 C₁₀H₁₇O₂ (M⁺+1): 169.1229, found 169.1233.

 \mathbf{F}

2-[3-[(Trimethylsilyl)oxy]propoxy]bicyclo[2.2.1]hep-2-ene (F). F was made from E by procedure of Gassman² et al. Yield: 90%. ¹H NMR (400 MHz, CDCl₃) \square 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); ¹³C NMR (100.6 MHz, CDCl₃) \square -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 C₁₃H₂₅O₂Si (M⁺+1): 241.1526, found 241.1545.

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 K₂CO₃ (1.61 g, 11.7 mmol) at 0 °C, and the solution allowed to stir for 1 h. After filtration and evaporation, the residue was partitioned between water and CH₂Cl₂, and the organic layer was dried with Na₂SO₄. Removal of solvent gave **G** 1.90 g (98%) as a brown oil. ¹H NMR (400 MHz, CDCl₃) \square 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); ¹³C NMR (100.6 MHz, CDCl₃) \square 25.2, 28.6, 32.1, 41.2, 44.4, 47.4, 61.3, 67.2,196.3; IR (neat) 3418, 1614 cm⁻¹; MS (CI) m/z 169 (M⁺+1); HRMS calcd for C₁₀H₁₇O₂ (M⁺+1): 169.1229, found 169.1220.

2-[3-Azido-propoxy]bicyclo[2.2.1]hept-2-ene (6). To a solution of **G** (200 mg, 1.2 mmol), $Zn(N_3)_2 \cdot 2Py^3$ (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%). ¹H NMR (400 MHz, CDCl₃) \Box 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); ¹³C NMR (100.6 MHz, CDCl₃) \Box 25.3, 28.6, 28.9, 41.2,

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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. ¹H NMR (400 MHz, CDCl₃) \Box 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, \Box = 4.2 Hz, 2H), 5.57 (s, 1H), 7.28-7.43 (complex, 5H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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 C₁₄H₁₈N₃O (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, CDCl₃) \square 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, CDCl₃) \square 14.5, 23.0, 23.1, 27.1, 27.2, 30.4, 31.9, 34.5, 43.8, 44.7, 44.9,

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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.

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; ¹H NMR (400 MHz, CDCl₃) \Box 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); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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 C₁₃H₂₂N₃O (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 °C 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 °C, the reaction mixture was allowed to warm to 0

°C 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 NaHCO₃, extracted with EtOAc, dried over Na₂SO₄, filtered, and concentrated. Flash chromatography afforded 542 mg (95%) of **10** as an oil: 1 H NMR (400 MHz, CDCl₃) [] 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, CDCl₃) [] 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-1H-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, CDCl₃) \Box 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, CDCl₃) \Box 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. ¹H NMR (400 MHz, CDCl₃) \square 2.38 (pentet, J = 6.5 Hz, 2H), 2.54 (ddt, J = 2.0, 4.3,

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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); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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 C₉H₁₃N₃Cl (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. ¹H NMR (400 MHz, CDCl₃): \Box 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); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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.

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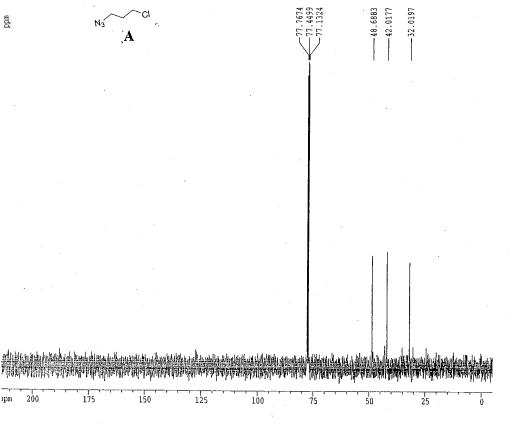
4-Methyl-5-ethyl-1-(4'-chlorobutyl)-1*H***-1,2,3-triazole** (**Table 1, entry 4).** Yield 32%; oil. ¹H NMR (400 MHz, CDCl₃) \Box 1.17 (t, J = 7.6 Hz, 3H), 1.79-1.86 (m, 2H), 2.02-2.09 (m, 2H), 2.29 (s, 3H), 2.64 (q, J = 7.6 Hz, 2H), 3.57 (t, J = 6.3 Hz, 2H), 4.25 (t, J = 7.1 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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.

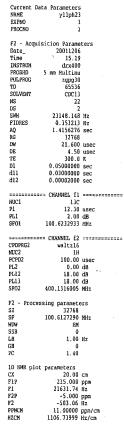
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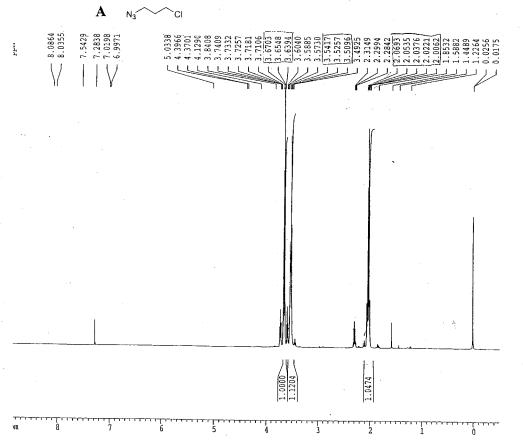
N- (4-Chlorobutyl)-2-methyl Butanamide (Table 1, entry 4) . Yield 30%; oil. ¹H NMR (400 MHz, CDCl₃) \Box 0.91 (t, J = 7.4 Hz, 3H), 1.13 (d, J = 6.9 Hz, 3H), 1.39-1.46 (m, 1H), 1.60-1.71 (m, 3H), 1.78-1.85 (m, 2H), 2.07-2.11 (m, 1H), 3.20-3.66 (m, 2H), 3.58 (t, J = 6.4 Hz, 2H); 5.66 (br s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) \Box 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 C₉H₁₈ClNO (M⁺+1): 192.1155, found 192.1158.

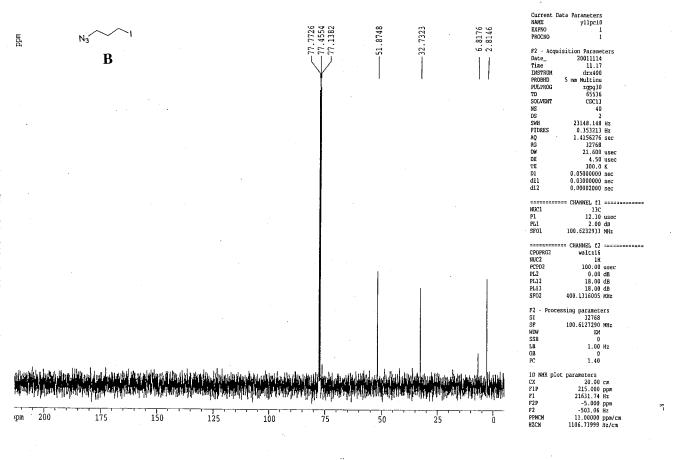
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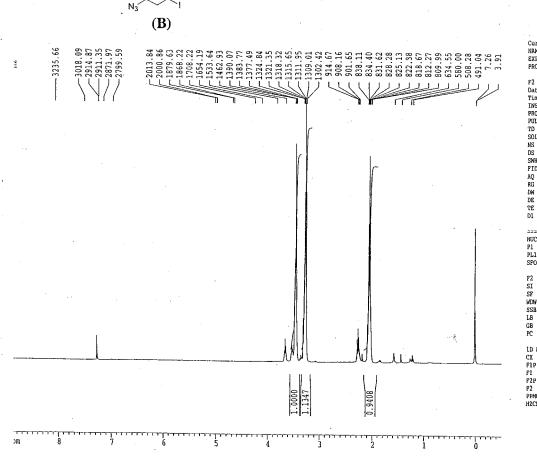
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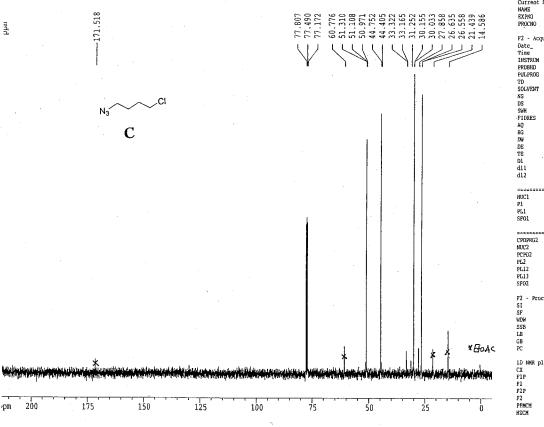


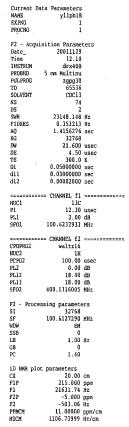








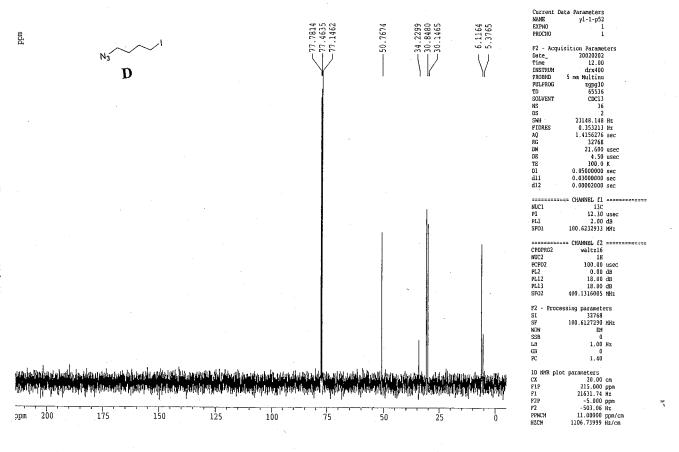




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F2P -0.500	
F2 -200.0	
	ppm/cm
HZCM 190.06175	5 Hz/cm



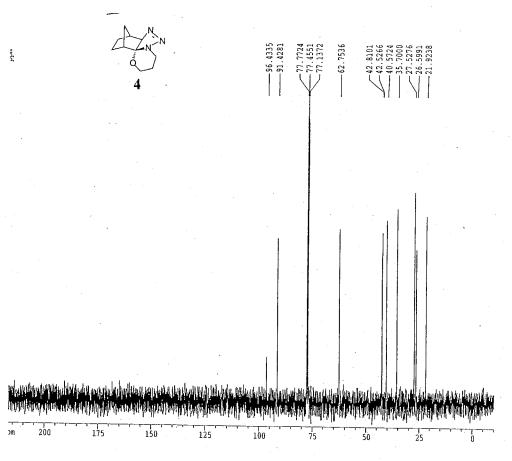
7 <u>1</u>

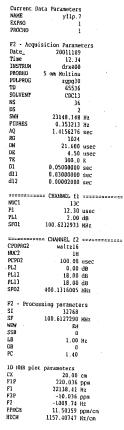
1

	(D) N ₃
ŗ i	8.0784 7.2838 7.2838 7.2838 7.2839 6.9054 6.9058 7.0200 6.9058 7.0200 7.
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	/ \
-	2.0000 2.4035 2.5454 1.9734
ттт)М	$\frac{1}{8}$ $\frac{1}{7}$ $\frac{1}{6}$ $\frac{1}{5}$ $\frac{1}{4}$ $\frac{1}{3}$ $\frac{1}{2}$ $\frac{1}{1}$

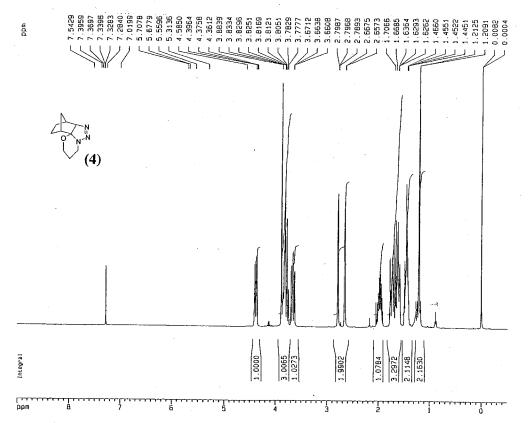
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Time	11.57	
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PROBHD	5 mm Multinu	
PULPROG	zq30	
TD	32768	
SOLVENT	CDC13	
NS	16	
DS	2	
SWH	4789,272	U e
FIDRES	0.146157	
AO	3.4210291	
RG		sec
DW DW	101.6	
	104.400	
DE		usec
TE	. 300.0	
DI	1.00000000	sec
	OIT - 1977 - 51	

NUC1	18	
Pl	7.70	
PL1	-6.00	
SF01	400.1320007	MH2
D2 D		
	sing paramete	ers
SI	16384	
SF	400.1300000	MHz
WDW	EM	
SSB	0	
LB	0.30	H2
GB	0	
PC	1.00	
1D NMR plot		
CX .	20.00	cm
F1P	9.000	ppm
F1	3601.17	Hz
F2P	-0.500	
F2	-200.07	
PPMCM	0.47500	
HZCM	190.06175	Hz/cm



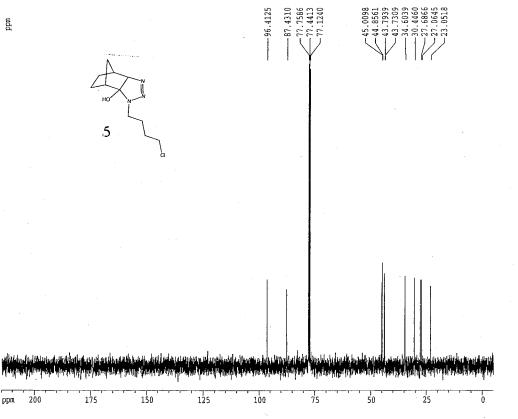


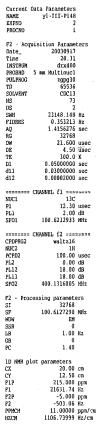
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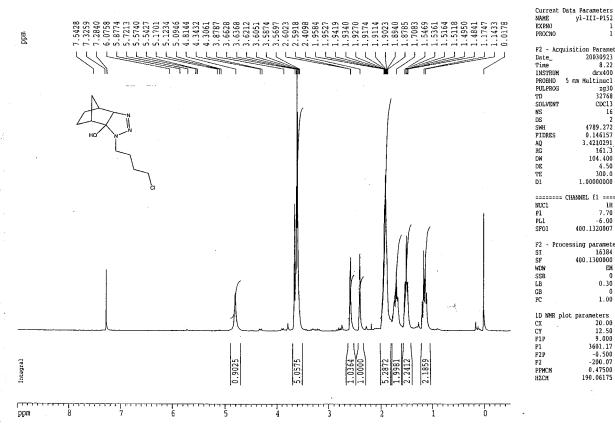


current bata	Parameters
NAME	ylip7
EXPNO	1
PROCNO	· i
	*
F2 - Acquisi	tion Parameters
Date	1011109
Time	
	12.28
INSTRUM	drx400
	mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	COC13
NS	15
0S	2 ·
SHH	4789.272 Hz
FIDRES	0.146157 Hz
AΩ	3.4210291 sec
RG	90.5
DW	104.400 usec
0E	4.50 usec
TE	300.0 K
Di .	1.00000000 sec
P1.	
-	7.70 usec
DE	4.50 usec
	100.1320007 HHz
1 DUN	111
PL1	-6.00 dB
F2 - Processi	ng parameters
SI	16384
SF 4	100.1300000 MHz
MDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC.	1.00
	1.00
10 NMA plat p	12mamakana
CX	
	·20.00 cm
FIR	9.000 ppm
F1	3601.17 Hz
F2P	~0.500 ppm
F2	-200.07 Hz
PPMCH	0.47500 ppm/cm
HZCM	190.06175 Hz/cm

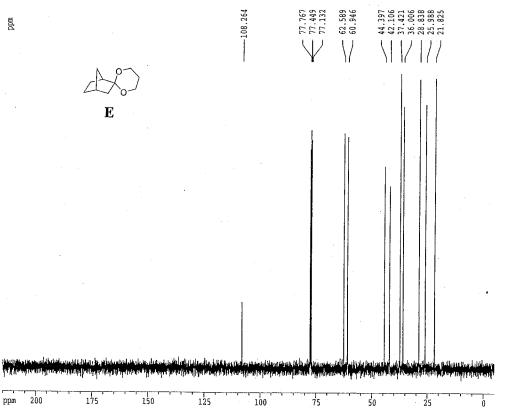
Current Data Parameters

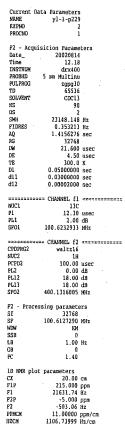


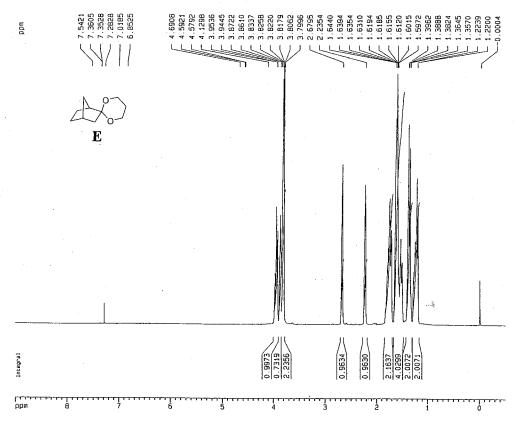




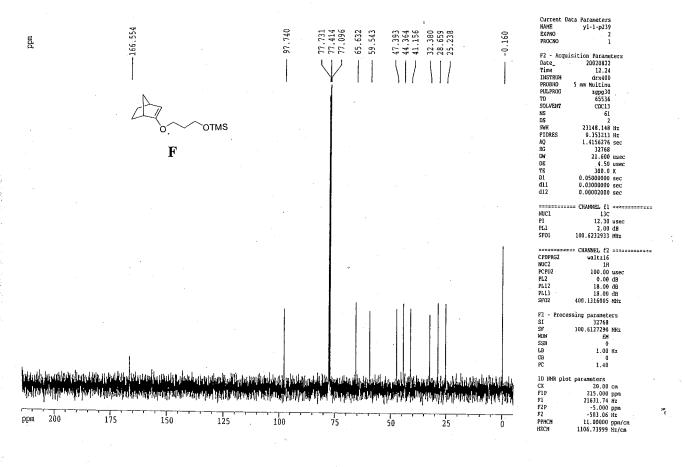
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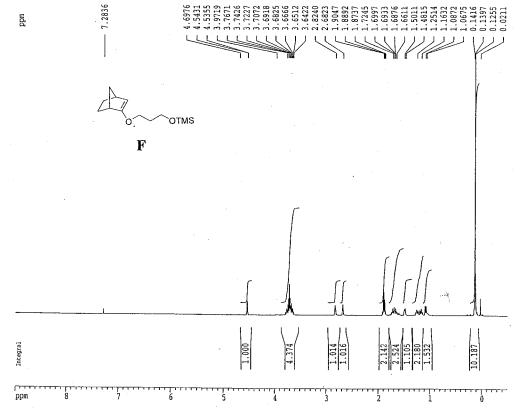






Current	Data Parameters
NAHE	v1-1-0229
EXPNO	1
PROCNO	i.
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F2 - Acc	uisition Parameters
Oate_	1020814
Time	12.14
INSTRUM	drx 400
PROBHO	5 mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	16
OS	5
SWH	47B9.272 Hz
FIORES	0.146157 Hz
AO	3.4210291 sec
RG	64
OH	104,400 usec
30	4.50 usec
TE	300.0 K
D1	1.00000000 sec
P1 DE	7.70 usec
SF01	4.50 usec
NUC1	400.1320007 HHz
PL1	1H
rt1	-6.00 dB
F2 - Pro	cessing parameters
SI	16384
SF	400 1300000 MHz
NDM	EH
SSB	0
LB	0.30 Hz
68	0
PC	1.00
	lot parameters
CX	20.00 cm
FIP	9.000 ppm
FT	3601.17 Hz
F2P	-0.500 ppm
F2	~200.07 Hz
PPMCM	0.47500 ppm/cm
HZCH	190.06175 Hz/cm

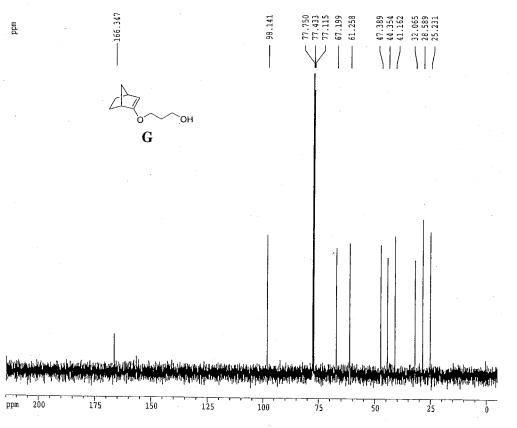


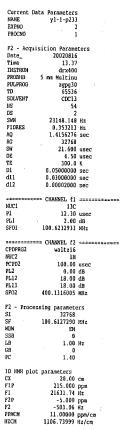


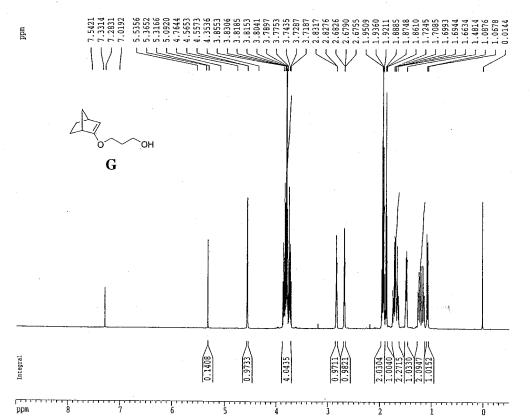
Current Data		
NAME	yl-1-p239	
EXPNO	. 1	
PROCNO	1	
F2 - Acquisi		ters
Date_ Time	20020822	
Time INSTRUM	12.21	
	drx400	
PROBHD 5 PULPROG	mm Multinu za30	
TĐ	2g30 32768	
SOLVENT	CDC13	
NS	16	
DS	2	
SWH	4789.272	H-
FIDRES	0.146157	
AO	3.4210291	
RG	143.7	566
ΓW	104.400	HSec
DE	4.50	
TE	300.0	
D1	1.00000000	
**********	CHANNEL fl	********
NUC1	18	
P1	7.70	usec
PL1	-6.00	
SF01	100.1320007	MHz
F2 - Process		ers
SI	16384	
	100.1300000	
WDW	EM 0	
SSB LB	0.30	
GB	0.30	nz
PC.	1.00	
rc .	1.00	
1D NMR plot p	narameters	
CX	20.00	CIN
FIP	9.000	
81	3601.17	Hz
F2P	-0.500	
F2	-200.07	
PPMCH	0.47500	
HZCM	190.06175	

S20

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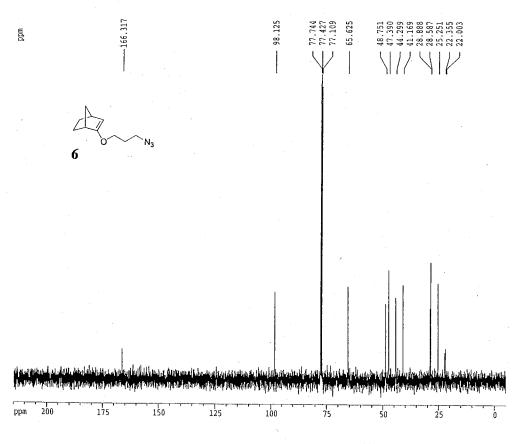


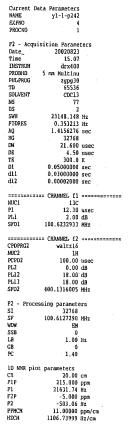


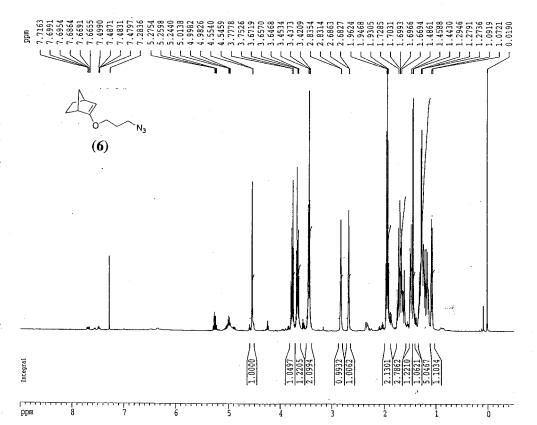
Current Data Param NAME yl-1 EXPNO PROCNO	
F2 - Acquisition Pa	arameters 20816
	13.35
	x400
PROBHD 5 mm Mul	ltinu
PULPROG	zq30
	12768
SOLVENT	DC13
NS	16
DS	2
SWH 4789).272 Hz
FIDRES 0.14	16157 Hz
	.0291 sec
	01.6
	.400 usec
DE	4.50 usec
TE :	00.0 K
	10000 sec
======= CHANNE	L fl ========
NUC1	1H
P1	7.70 usec
PL1 -	6.00 dB
SF01 400.132	0007 MHz
F2 - Processing par	amotore
r4 - riocessing par	unerers.
	.6384
SI 1	
SI 1	.6384
SI 1 SF 400.130	.6384 0000 MHz
SI 1 SF 400.130 WDW	.6384 10000 MHz EM
SI 1 SF 400.130 WDW SSB LB GB	.6384 10000 MHz EM 0
SI 1 SF 400.130 WDW SSB LB	.6384 HOOOO MHZ EM O O.30 H2
SI 1 SF 400.130 WDW SSB LB GB PC	.6384 10000 MHz EM 0 .30 Hz 0 1.00
SI 1 SF 400.130 WDW SSB LB GB PC	.6384 10000 MHz EM 0 0.30 Hz 1.00
SI 1 SP 400.130 WDW SSB LB GB PC 1D NNR plot paramet CX 2	.6384 00000 MHz EM 0 0.30 Hz 1.00
SI 1 SP 400.130 SP 400.130 SSB LB GB PC 1D NMR plot paramet CX 2 FIP 9	.6384 .6000 MHz EM 0 0.30 Hz 0 1.00 .ers .0.00 cm
SI 1 SF 400.130 MDW SSB LB GB PC 1D NNR plot paramet CX 2 PlP 9 F1 366	6.384 0000 MHz EM 0 0 0.30 Hz 0 1.00 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
SI 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	.5384 .0000 MHz EM .00.30 Hz .00 1.00 .00 cm .000 ppm 1.17 Hz .500 ppm
SI 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	6.5384 00000 MHz EM 0 0.30 Hz 0 1.00 ers 0.00 cm .000 ppm 1.17 Hz .500 ppm 0.07 Hz
SI 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	.5384 .0000 MHz EH .00 .30 Hz .00 .1.00 .1.00 .ers .0.00 cm .000 ppm .1.17 Hz .500 ppm .0.7 Hz
SI 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	6.5384 00000 MHz EM 0 0.30 Hz 0 1.00 ers 0.00 cm .000 ppm 1.17 Hz .500 ppm 0.07 Hz

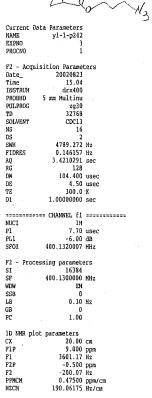
S21

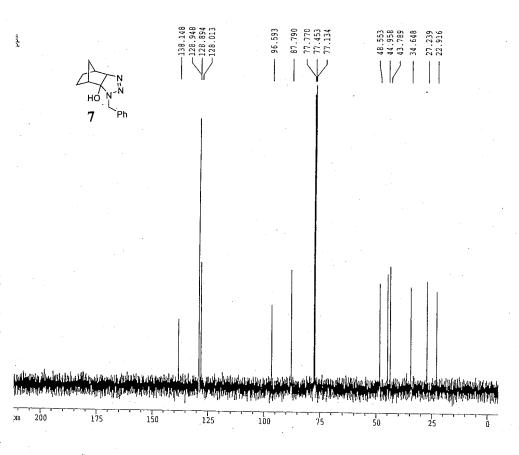
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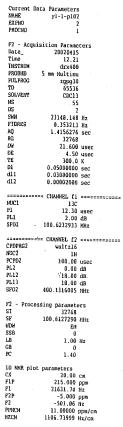


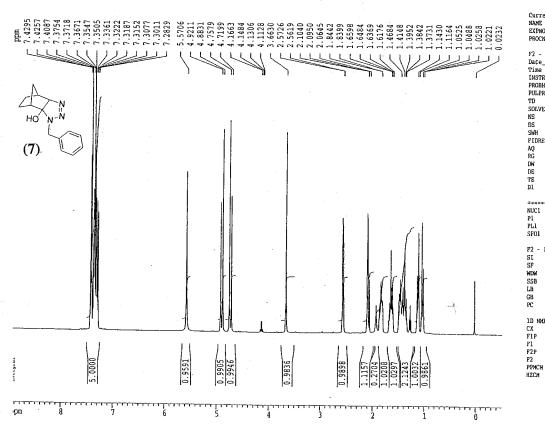




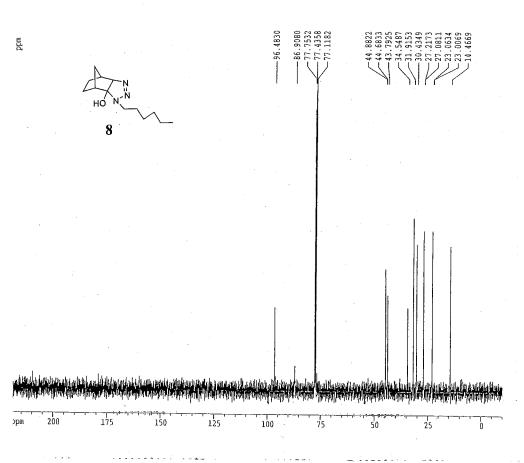


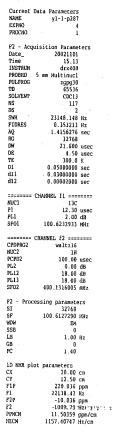


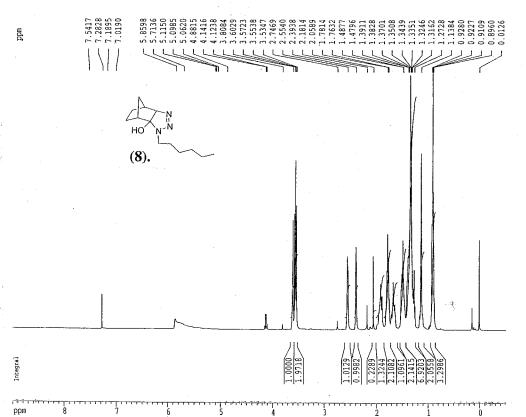


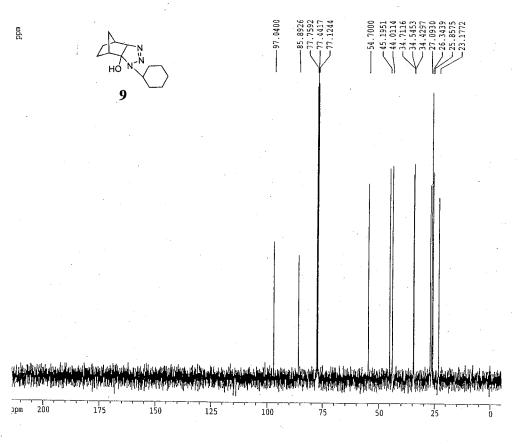


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PROCNO	ì
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F2 - Acc	puisition Parameters
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Time -	12.18
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PROBHD	5 mm Multinu
PULPROG	zq30
TD	12768
SOLVENT	CDC13
NS	16
DS	2
SWH	4789.272 H2
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 £1 =========
NUC1	18
P1	7.70 usec
PL1	-6.00 dB
SF01	400.1320007 MHz
F2 - Pro	cessing parameters
SI	16384
SF	400.1300000 MHz
WDW	EM ·
SSB	0
LB	0.30 Hz
GB	0
PC	1.00
	*
1D NMR o	lot parameters
CX	20.00 cm
FIP	9.000 ppm
F1	3601.17 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCH	0.47500 ppm/cm
HZCM	190.06175 Hz/cm

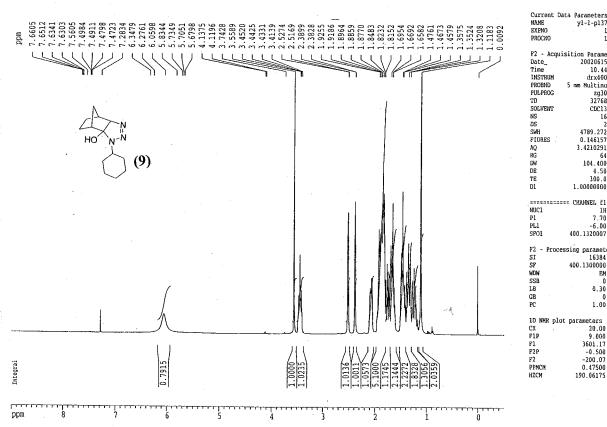




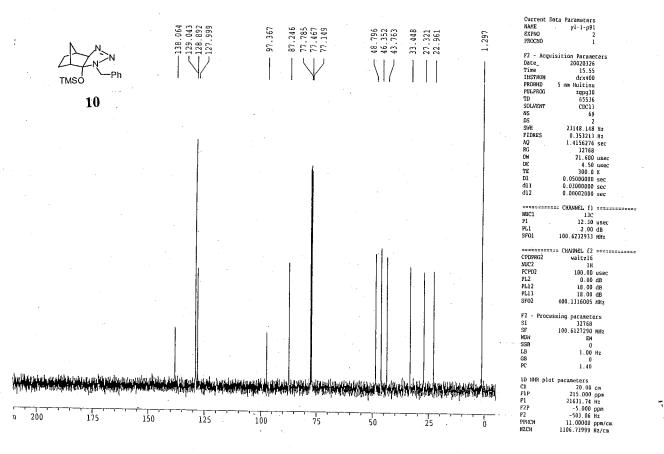


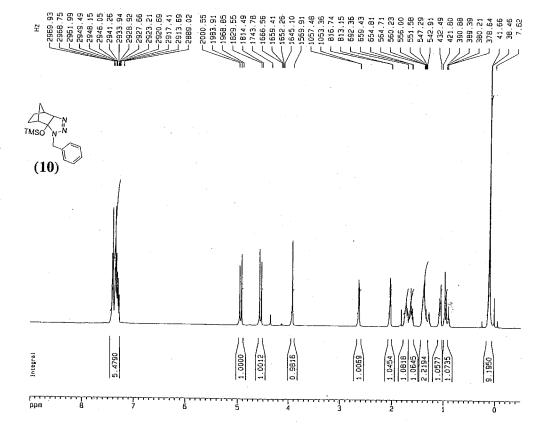




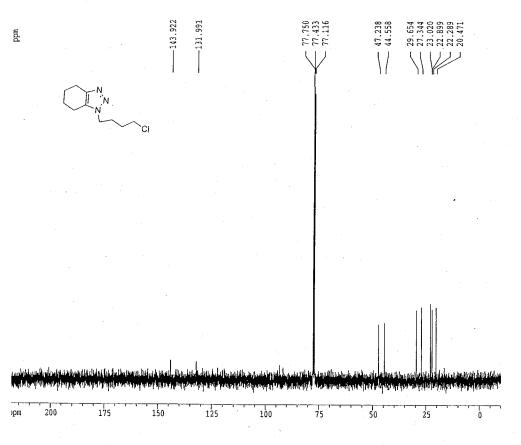


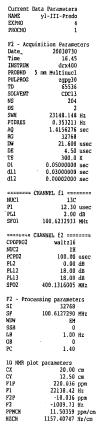
	Data Parameters
NAME	yl-1-p137
EXPNO	1
PROCNO	ī
	-
F2 - Acc	uisition Parameters
Date_	20020615
Time	
	10.44
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	16
DS	2
SWH	4789.272 Hz
FIDRES .	0.146157 Hz
AO	3.4210291 sec
RG	64
DW	104.400 usec
DE	4.50 usec
TE	
D1	300.0 K
ut	1.00000000 sec
	OUTS A PORT OF
	==== CHANNEL fl =========
NUC1	1H
P1	7.70 usec
PL1	-6.00 dB
SPO1	400.1320007 мHz
	cessing parameters
SI	16384
SF	400.1300000 MHz
WDW	EM
SSB	o .
LB	0.30 Hz
GB	0
PC	1.00
	1.00
ID NWD A	lot parameters
CX .	20.00 cm
F1P	
Fl	9.000 ppm 3601.17 Hz
F1 F2P	
	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.47500 ppm/cm
HZCM	190.06175 Hz/cm

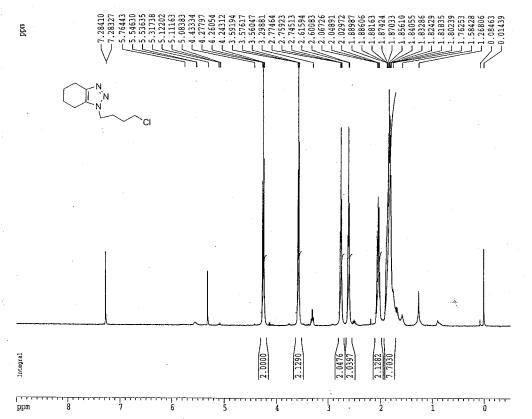




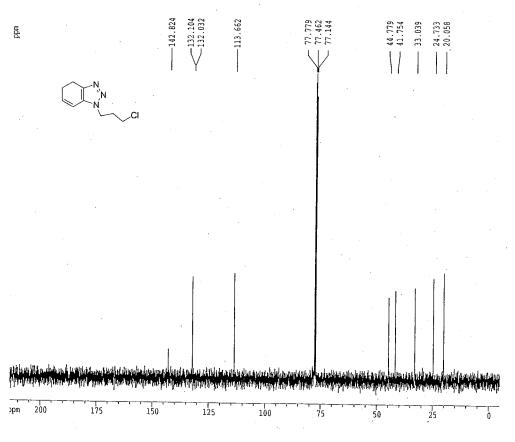
NAME	y1-1-091	
EXPNO	7. 1-pa;	
PROCNO		
FROCINO	1	
F2 - Acm	isition Parameters	
Date_	1020326	
Time	15.49	
INSTRUM	drx400	
PROBHO	5 mm Multinu	
PULPROG	2930	
TO	32768	
SOLVENT	CDC 13	
NS.		
05	16	
SWH	2	
FIDRES	4789.272 Hz	
AU L TOHE?	0.146157 Hz	
RG	3.4210291 sec	
DW .	64	
DN DE	104.400 usec	
ΙΕ	4.50 usec	
D1	300.0 K	
P1	1.00000000 sec	
DE	7.70 usec	
SF01	4.50 usec	
	400.1320007 MHz	
NUC1 -	1H	
PL1	-6.00 dB	
F2 - Broce	essing parameters	
SI	16384	
SE	400 . 1300000 HHz	
MOM	EH	
SSB	. 0	
LB	0.30 Hz	
68	u. 30 Hz	
PC	1.00	
, ,	1.00	
10 NMA 01	t parameters	
CX	20.00 cm	
FIP	9.000 ppm	
F1	3501.17 Hz	
F2P	~0.500 ppm	
F2	-200.07 Hz	
PPHCH	0.47500 ppm/cm	
HZCH	190.06175 Hz/cm	
112011	130.00175 HZ/CM	

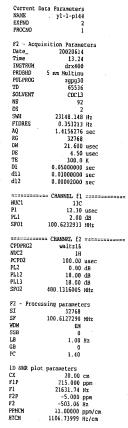


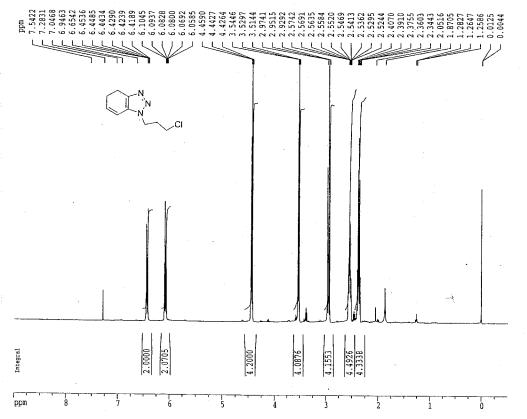




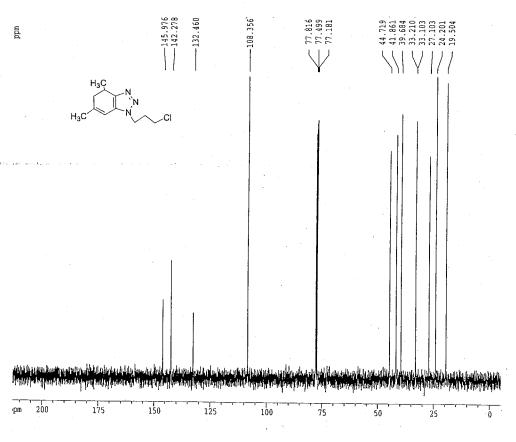
	a Parameters yl-III-Predo 3 1	
Date_ Time INSTRUM	ition Parame 2003D730 16.42 drx400 mm Multinucl 2g30 32768 CDC13 16 2 4789.272 0.146157 3.4210291 143.7 104.400	Hz Hz sec usec
DE		usec
TE	300.0	
D1	1.00000000	sec
======= CH	AARIDI EL	
NUC1 P1 PL1 SFO1	114	usec dB
NUC1 P1 PL1 SF01	1H 7.70 -6.00 400.1320007	usec dB MHz
NUC1 P1 PL1 SF01	1H 7.70 -6.00	usec dB MHz
NUC1 P1 PL1 SF01 F2 - Proces	1H 7.70 -6.00 400.1320007 sing paramete	usec dB MHz
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW	1H 7.70 -6.00 400.1320007 sing paramete 16384 400.1300000 EM	usec dB MHz
NUC1 P1 PL1 SF01 F2 - Proces: SI SF WDW SSB	1H 7.70 -6.00 400.1320007 sing paramete 16384 400.1300000 EM 0	usec dB MHz ers
NUC1 P1 P1 SF01 F2 - Proces SI SF WDW SSB LB	1H 7.70 -6.00 400.1320007 sing paramete 16384 400.1300000 EM 0	usec dB MHz ers
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW SSB LB GB	1H 7.70 -6.00 400.1320007 sing parametr 16384 400.1300000 EM 0 0.30	usec dB MHz ers
NUC1 P1 P1 SF01 F2 - Proces SI SF WDW SSB LB	1H 7.70 -6.00 400.1320007 sing paramete 16384 400.1300000 EM 0	usec dB MHz ers
NUC1 P1 PL1 SF01 F2 - Process SI SF WDW SSB LB GB PC 1D NMR plot	1H 7.70 -6.00 400.1320007 sing paramete 16384 400.1300000 EM 0 0.30 0 1.00	usec dB MHz ers MHz
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW SSB LB GB PC	1H 7.70 -6.00 400.1320007 sing parameti 16384 400.1300000 EM 0 0.30 0 1.00	usec dB MHz ers MHz Hz
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW SSB LB GB PC LB NMR plot CX CY	1H 7.70 -6.00 400.1320007 sing parameter 16384 400.1300000 EM 0 0.30 0 1.00 parameters 20.00 12.50	usec dB MHz ers MHz Hz
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW SSB LB GB PC	1H 7.70 400.00 1320007 sing parameter 16384 400.1320000 EM 0 0.30 0 1.00 parameters 20.00 12.50 9.000	usec dB MHz ers MHz Hz cm cm ppm
NUC1 P1 PL1 SF01 F2 - Proces SI SP WDW SSB LB GB PC 1D NMR plot CX CY F1P	1H 7.70 400.1320007 sing paramete 16384 400.1300000 EM 0 0.30 0 1.00 parameters 20.00 12.50 9.000 3601.17	usec dB MHz ers MHz Hz cm cm ppm
NUC1 P1 PL1 SF01 F2 - Proces SI SF WDW SSB LB GB PC 1D NMMR plot CX CY P1P F1	1H 7.70 400.00 1320007 sing parameter 16384 400.1320000 EM 0 0.30 0 1.00 parameters 20.00 12.50 9.000	usec dB MHz ers MHz H2 cm cm ppm Hz ppm
NUC1 P1 P1 P1 SF01 F2 - Proces SI SF WDW SSB LB GB PC 1D NMR plot CX CY P1P F1 F2P	1H 7,70 -6.00 400.1320007 sing paramet 1534 400.1300000 EM 0 0.100 parameters 20.00 12.55 9.000 3601.17 -0.500 -200.07 0.47500	usec dB MHz ers MHz Hz cm cm ppm Hz ppm Hz ppm/cm
NUC1 P1 PL1 SF01 F2 - Proces S1 SF WDM SSSB LB GB PC 1D NMR plot CX CY F1F F1 F1 F2P F2	1.H 7.70 400.1320007 sing paramet- 16384 400.1300000 0 0.30 0 1.00 parameters 20.00 12.50 9.000 3601.17 -0.500	usec dB MHz ers MHz Hz cm cm ppm Hz ppm Hz ppm/cm

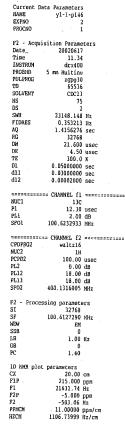


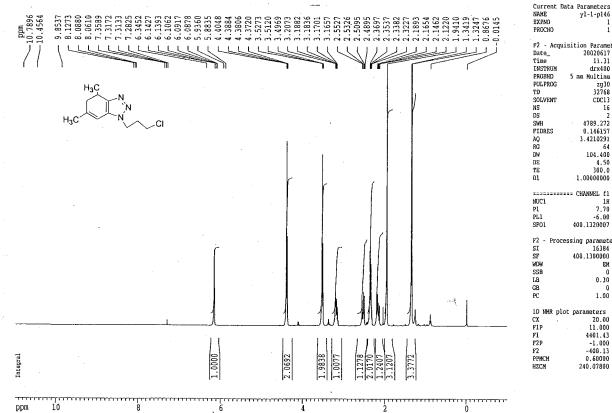




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RG	114	
DW .	104.400	
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TE	300.0	
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DI	1.00000000	sec
353355-4444	= CHANNET, F1	=========
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P1		usec
PL1	-6.00	
SFO1	400.1320007	
0.01	100.1320001	1412
F2 - Proces	sing paramet	ers
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SF	400.1300000	MH2
WDW	EM	
SSB	0	
LB	0.30	H2
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PC	1.00	
1D NMR plot	parameters	
CX .	20.00	CM
FIP	9.000	DDM
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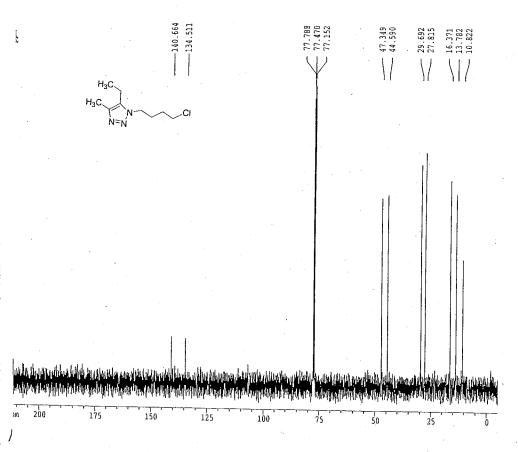


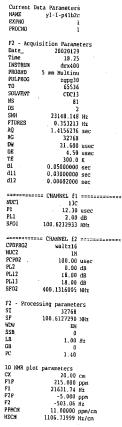


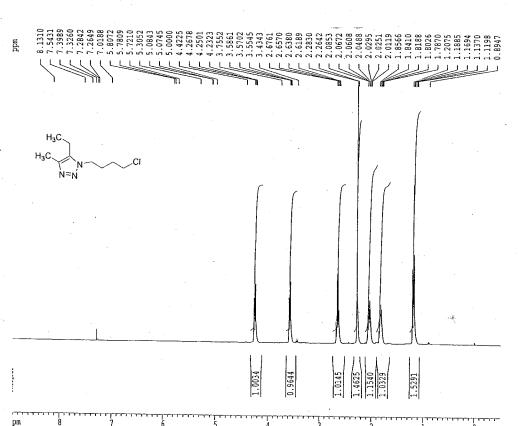


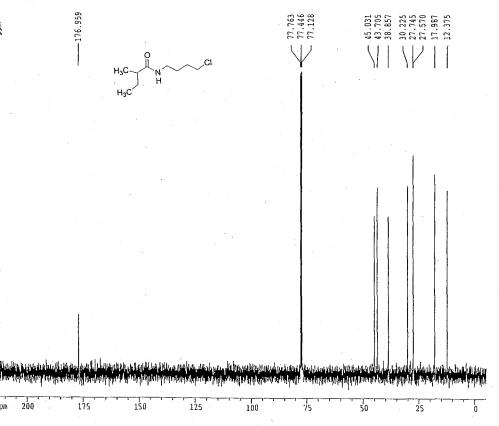
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DS	2	
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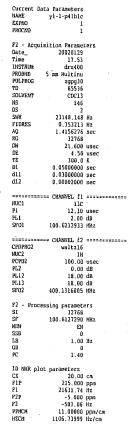
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SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	
	2.00	
ID NMR plot	narameters	
CX	20.00	Cm.
F1P	11.000	
ri	4401.43	
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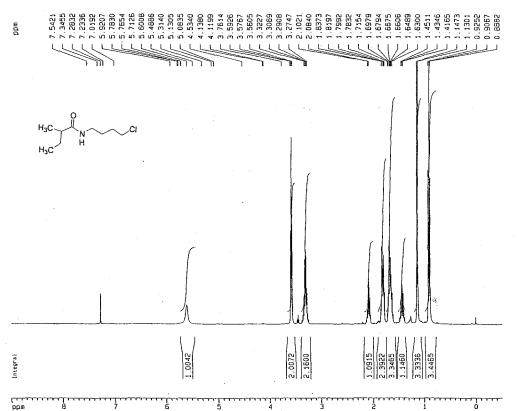












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PL 1	-6.00	dB		
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MDM	EM			
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LB	0.30	Hz		
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to NMR old	t parameters			
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F1	3601.17			
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Current Data Parameters