Alkyl–Alkyl Suzuki Cross-Couplings of Unactivated Secondary Alkyl Chlorides

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Supporting Information

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I. General

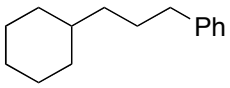
The following reagents were purchased and used as received: 9-BBN dimer (Aldrich), $NiBr_2$•diglyme (Aldrich; somewhat hygroscopic), KO*t*-Bu (Acros), *i*-BuOH (anhydrous, Aldrich), and *i*-Pr_2O (anhydrous, Aldrich). Molecular sieves (4 Å, powdered, <5 μ; Aldrich) were flame-dried. Ligand (±)-**1** was synthesized according to a procedure by Alper[1] and purified by flash chromatography (it is also available from Acros).

II. Suzuki Cross-Coupling Reactions

General procedure for the preparation of the boron reagent. 9-BBN dimer (220 mg, 0.90 mmol) was added to a flask (equipped with a side arm and a stir bar), which was then sealed with a septum and successively evacuated and back-filled with argon three times. *i*-Pr_2O (0.75 mL) and the alkene (1.8 mmol) were added, and the reaction mixture was stirred at 60 °C for 1 h. Next, the mixture was allowed to cool to r.t., and then KO*t*-Bu (150 mg, 1.2 mmol) and *i*-BuOH (180 μL, 2.0 mmol) were added in single portions under a positive pressure of argon. The resulting solution was stirred under argon for 30 min.

(1) Kuznetsov, V. F.; Jefferson, G. R.; Yap, G. P. A.; Alper, H. *Organometallics* **2002**, *21*, 4241–4248.

General procedure for the Suzuki reaction. $NiBr_2$•diglyme (21 mg, 0.060 mmol) and 4 Å molecular sieves (200 mg) were added to a 5-mL oven-dried round-bottom flask (equipped with a stir bar), which was then sealed with a septum and successively evacuated and back-filled with argon three times. *i*-Pr_2O (0.8 mL) was added, followed by ligand **1** (18 μL, 0.080 mmol). The mixture was stirred for 30 min, resulting in the formation of a pale-blue slurry. Next, the alkyl halide (1.0 mmol) and then the solution of the boron reagent (1.5 M; 1.8 mmol) were added by syringe. The reaction mixture was stirred vigorously at room temperature for 48 h. Then, it was passed through a short plug of silica gel, eluting with 1:1 Et_2O/hexanes. The solvent was removed, and the product was purified by flash chromatography.



(3-Cyclohexylpropyl)benzene (Table 2, entry 1) [170661-44-6]. Cyclohexyl chloride (119 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (cyclohexane). Colorless oil. First run: 160 mg (79%). Second run: 162 mg (80%).

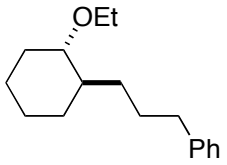
TLC: R_f = 0.7 (cyclohexane; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 7.21-7.14 (m, 3H), 2.56 (t, 2H, *J* = 7.8 Hz), 1.75-1.59 (m, 7H), 1.30-1.07 (m, 6H), 0.97-0.80 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.0, 128.4, 128.2, 125.6, 37.6, 37.2, 36.3, 33.4, 28.8, 26.7, 26.4.

FT-IR (neat) 2958, 2930, 2859, 1496, 1454, 745, 697 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{15}H_{22}$: 202.2, found: 202.2.



***trans*-(3-(2-Ethoxycyclohexyl)propyl)benzene (Table 2, entry 2).** *trans*-1-Chloro-2-ethoxycyclohexane (163 mg, 1.0 mmol; prepared according to a literature procedure[2]) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (2%→5% EtOAc/hexanes). Clear oil. First run: 166 mg (67%). Second run: 176 mg (71%).

TLC: R_f = 0.55 (10% EtOAc/hexanes; PMA).

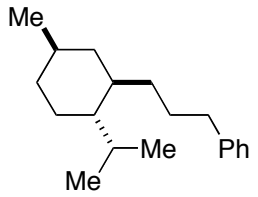
(2) Mendonca, G. F.; Sanseverino, A. M.; De Mattos, M. C. S. *Synthesis* **2003**, 45–48.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 7.20-7.14 (m, 3H), 3.68-3.60 (m, 1H), 3.37-3.29 (m, 1H), 2.85-2.77 (m, 1H), 2.68-2.51 (m, 2H), 2.07-2.01 (m, 1H), 1.87-1.50 (m, 7H), 1.37-1.07 (m, 5H), 1.17 (t, 3H, J = 7.0 Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 128.5, 128.3, 125.6, 82.2, 64.1, 43.2, 36.6, 32.2, 31.6, 30.6, 28.9, 25.6, 24.9, 15.8.

FT-IR (neat) 2928, 2858, 1496, 1449, 1104, 747, 698 cm^{-1}.

MS (EI) m/z (M^+) calcd for $C_{17}H_{26}O$: 246.2, found: 246.2.



(3-((1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl)propyl)benzene (Table 2, entry 3). (–)-Menthyl chloride (187 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (cyclohexane). Clear oil. First run: 135 mg (52%). Second run: 140 mg (54%).

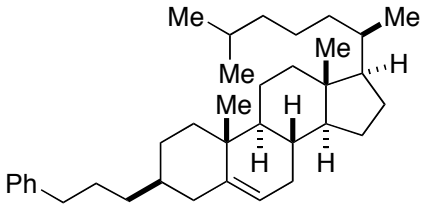
TLC: R_f = 0.7 (cyclohexane; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.25 (m, 2H), 7.21-7.15 (m, 3H), 2.66-2.49 (m, 2H), 2.00-1.90 (m, 1H), 1.75-1.40 (m, 6H), 1.35-1.05 (m, 3H), 1.11-0.72 (m, 9H), 0.70 (d, 3H, J = 6.9 Hz), 0.70-0.59 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.1, 128.5, 128.3, 125.7, 46.7, 41.4, 38.8, 36.7, 35.5, 33.0, 32.6, 28.0, 26.5, 24.5, 23.0, 21.8, 15.4.

FT-IR (neat) 2954, 2928, 2866, 1453, 1368, 745, 698 cm^{-1}.

MS (EI) m/z (M^+) calcd for $C_{19}H_{30}$: 258.2, found: 258.3.



3-(3-Phenylpropyl)cholest-5-ene (Table 2, entry 4). Cholesteryl chloride (405 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by

flash chromatography (hexanes). Waxy solid. mp 65–70 °C. First run: 326 mg (67%). Second run: 328 mg (67%). The product was isolated as a 2:1 (β:α) mixture of two diastereomers.[3]

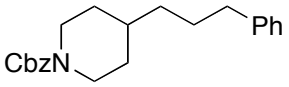
TLC: R_f = 0.5 (hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.25 (m, 2H), 7.20-7.14 (m, 3H), 5.29-5.25 (m, 1H, major diastereomer), 5.23-5.20 (m, 1H, minor diastereomer), 2.58 (t, 2H, *J* = 8.4 Hz), 2.10-1.85 (m, 45H), 0.67 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (both diastereomers) 143.7, 143.0, 128.50, 128.47, 128.4, 128.3, 125.7, 119.3, 57.0, 56.3, 50.6, 42.4, 40.0, 39.8, 39.6, 39.4, 37.5, 37.4, 37.0, 36.4, 36.3, 35.9, 34.2, 32.0, 29.3, 28.9, 28.4, 28.2, 24.4, 24.0, 23.0, 22.7, 21.0, 19.6, 18.8, 12.0.

FT-IR (neat) 2933, 2867, 1461, 1376, 746, 648 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{36}H_{56}$: 488.4, found: 488.5.



Benzyl 4-(3-phenylpropyl)piperidine-1-carboxylate (Table 2, entry 5). Benzyl 4-chloropiperidine-1-carboxylate (254 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5%→15% EtOAc/hexanes). White solid. mp 150 °C (dec). First run: 240 mg (71%). Second run: 229 mg (68%).

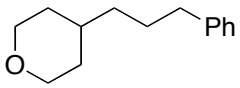
TLC: R_f = 0.15 (10% EtOAc/hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.20 (m, 7H), 7.20-7.10 (m, 3H), 5.11 (s, 2H), 4.15 (br s, 2H), 2.74 (br s, 2H), 2.59 (t, 2H, 7.6 Hz), 1.80-1.55 (m, 4H), 1.50-1.34 (m, 1H), 1.32-1.24 (m, 2H), 1.16-1.04 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4, 142.6, 137.1, 128.6, 128.45, 128.39, 128.0, 127.9, 125.8, 67.0, 45.0, 44.4, 36.2, 35.9, 28.6, 24.5.

FT-IR (neat) 2931, 2856, 1700, 1430, 1235, 749, 698 cm^{-1}.

MS (ESI) *m*/*z* (M^+) calcd for $C_{22}H_{27}NO_2$: 337.2, found: 338.2 (M + H^+), 360.2 (M + Na^+).



4-(3-Phenylproply)tetrahydro-2*H*-pyran (Table 2, entry 6). 4-Chlorotetrahydro-pyran (108 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5%→15% EtOAc/hexanes). Colorless oil. First run: 149 mg (73%). Second run: 141 mg (69%).

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(3) The stereochemistry was assigned by analogy to: Okamura, W. H.; Mitra, M. N.; Pirio, M. R.; Mourino, A.; Carey, S. C.; Norman, A. W. *J. Org. Chem.* **1978**, *43*, 574–580.

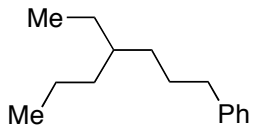
TLC: R_f = 0.25 (10% EtOAc/hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 7.20-7.14 (m, 3H), 3.93 (ddd, 2H, *J* = 11.5, 4.7, 1.2 Hz), 3.35 (td, 2H, 12.0, 1.8 Hz), 2.59 (t, 2H, 7.7 Hz), 1.70-1.42 (m, 5H), 1.35-1.20 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 128.5, 128.4, 125.8, 68.3, 36.7, 36.2, 35.1, 33.3, 28.4.

FT-IR (neat) 2928, 2842, 1096, 748, 699 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{14}H_{20}O$: 204.2, found: 204.2.



(4-Ethylheptyl)benzene (Table 2, entry 7). 3-Chlorohexane (139 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (cyclohexane). Colorless oil. First run: 145 mg (71%). Second run: 149 mg (73%).

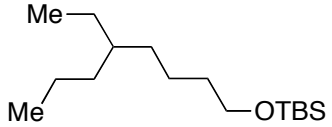
TLC: R_f = 0.65 (cyclohexane; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.25 (m, 2H), 7.21-7.15 (m, 3H), 2.58 (t, 2H, *J* = 7.7 Hz), 1.64-1.52 (m, 2H), 1.34-1.17 (m, 9H), 0.92-0.78 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.1, 128.5, 128.3, 125.7, 38.7, 36.6, 35.7, 33.0, 28.8, 26.0, 20.0, 14.6, 11.0.

FT-IR (neat) 2958, 2930, 1495, 1454, 746, 697 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{15}H_{24}$: 204.2, found: 204.3.



***tert*-Butyl(5-ethyloctyloxy)dimethylsilane (Table 2, entry 8).** 3-Chlorohexane (139 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of (but-3-enyloxy)(*tert*-butyl)dimethylsilane (prepared according to a literature procedure[4]) with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (hexanes). Colorless oil. First run: 180 mg (66%). Second run: 172 mg (63%).

TLC: R_f = 0.3 (hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.60 (t, 2H, *J* = 6.6 Hz), 1.55-1.45 (m, 2H), 1.38-1.15 (m, 11H), 0.88 (s, 9H), 0.87 (t, 3H, *J* = 7.2 Hz), 0.82 (t, 3H, *J* = 7.3 Hz), 0.05 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 63.4, 38.8, 35.7, 33.5, 33.1, 26.1, 26.0, 23.0, 20.0, 18.5, 14.5, 11.0, –5.1.

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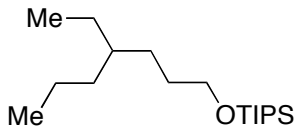
(4) Ferrie, L.; Reymond, S.; Capdevielle, P.; Cossy, J. *Org. Lett.* **2007**, *9*, 2461–2464.

FT-IR (neat) 2958, 2930, 2860, 1463, 1255, 1102, 836, 774 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd $C_{16}H_{36}OSi$: 272.2, found: 215.2 (M^+ – *t*-Bu).



(4-Ethylheptyloxy)triisopropylsilane (Table 2, entry 9). 3-Chlorohexane (139 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allyloxytriisopropylsilane (prepared according to a literature procedure[5]) with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (hexanes). Colorless oil. First run: 189 mg (63%). Second run: 198 mg (66%).

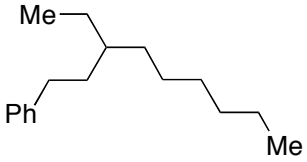
TLC: R_f = 0.3 (hexanes; PMA).

^1H NMR (400 MHz, $CDCl_3$) δ 3.65 (t, 2H, *J* = 6.9 Hz), 1.56-1.46 (m, 2H), 1.34-1.16 (m, 9H), 1.14-1.00 (m, 21H), 0.87 (t, 3H, *J* = 6.8 Hz), 0.83 (t, 3H, *J* = 7.3 Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ 64.1, 38.6, 35.7, 30.3, 29.1, 26.0, 20.0, 18.2, 14.6, 12.2, 11.0.

FT-IR (neat) 2959, 2942, 2867, 1464, 1105, 883 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{18}H_{40}OSi$: 300.3, found: 257.3 (M^+ – *i*-Pr).



(3-Ethylnonyl)benzene (Table 2, entry 10). (3-Chloropentyl)benzene (183 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of 1-hexene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (hexanes). Colorless oil. First run: 170 mg (73%). Second run: 172 mg (74%).

TLC: R_f = 0.7 (hexanes; UV).

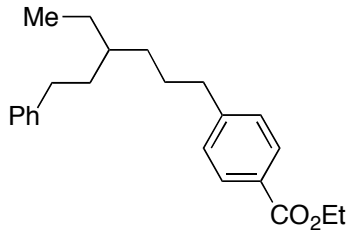
^1H NMR (400 MHz, $CDCl_3$) δ 7.31-7.25 (m, 2H), 7.21-7.15 (m, 3H), 2.58 (t, 2H, *J* = 7.8 Hz), 1.67-1.51 (m, 2H), 1.40-1.20 (m, 13H), 0.92-0.80 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 128.5, 128.4, 125.6, 38.8, 35.4, 33.3, 33.2, 32.1, 29.9, 26.7, 25.9, 22.8, 14.3, 10.9.

FT-IR (neat) 2959, 2925, 2857, 1454, 743, 697 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{17}H_{28}$: 232.2, found: 232.3.

(5) Frye, S. V.; Eliel, E. L. *J. Am Chem. Soc.* **1988**, *110*, 484–489.



Ethyl 4-(4-ethyl-6-phenylhexyl)benzoate (Table 2, entry 11). (3-Chloropentyl)benzene (183 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of ethyl 4-allylbenzoate (prepared according to a literature procedure[6]) with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5%→10% EtOAc/hexanes). Colorless oil. First run: 230 mg (71%). Second run: 234 mg (72%).

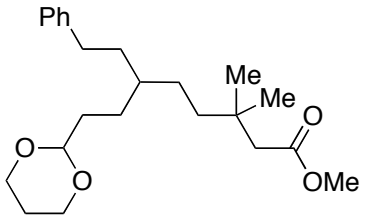
TLC: R_f = 0.3 (10% EtOAc/hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.89 (m, 2H), 7.30-7.20 (m, 4H), 7.20-7.10 (m, 3H), 4.33 (q, 2H, *J* = 7.1 Hz), 2.61 (t, 2H, *J* = 7.6 Hz), 2.52 (dd, 2H, *J* = 12.4, 5.5 Hz), 1.65-1.48 (m, 4H), 1.34 (t, 3H, *J* = 7.2 Hz), 1.40-1.23 (m, 5H), 0.82 (t, 3H, *J* = 7.0 Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 148.4, 143.2, 129.7, 128.6, 128.5, 128.45, 128.42, 125.7, 60.9, 38.5, 36.5, 35.2, 33.3, 32.7, 28.3, 25.8, 14.5, 10.9.

FT-IR (neat) 2933, 2859, 1718, 1275, 1106, 761, 699 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{23}H_{30}O_2$: 338.2, found: 338.3.



Methyl 6-(2-(1,3-dioxan-2-yl)ethyl)-3,3-dimethyl-8-phenyloctanoate (Table 2, entry 12). 2-(3-Chloro-5-phenylpentyl)-1,3-dioxane (139 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of 3,3-dimethyl-pent-4-enoic acid methyl ester with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (CH_2Cl_2→7.5% EtOAc/CH_2Cl_2). Colorless oil. First run: 309 mg (82%). Second run: 301 mg (80%).

TLC: R_f = 0.5 (20% EtOAc/hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.23 (m, 2H), 7.19-7.13 (m, 3H), 4.83 (t, 1H, *J* = 5.2 Hz), 4.13-4.07 (m, 2H), 3.76 (td, 2H, *J* = 11.8, 1.9 Hz), 3.63 (s, 3H), 2.61-2.54 (m, 2H), 2.14-2.01 (m, 1H), 1.62-1.50 (m, 5H), 1.42-1.24 (m, 9H), 0.98 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 143.1, 128.5, 128.4, 125.7, 102.8, 67.0, 51.2, 45.8, 38.9, 37.6, 35.5, 33.3, 33.1, 32.6, 27.6, 27.5, 27.4, 26.0.

FT-IR (neat) 2954, 2856, 1736, 1145, 746, 700 cm^{-1}.

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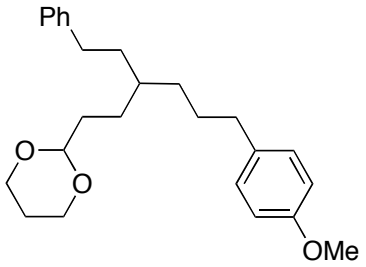
(6) Piazza, C.; Knochel, P. *Angew. Chem., Int. Ed.* **2002**, *41*, 3263–3265.

MS (EI) m/z (M^+) calcd for $C_{23}H_{36}O_4$: 376.3, found: 375.3 ($M^+ - H$).



2-(6-(4-Methoxyphenyl)-3-phenethylhexyl)-1,3-dioxane (Table 2, entry 13). 2-(3-Chloro-5-phenylpentyl)-1,3-dioxane (139 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of 4-allylanisole with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5%→10% EtOAc/hexanes). Colorless oil. First run: 314 mg (82%). Second run: 321 mg (84%).

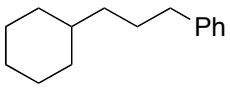
TLC: $R_f = 0.5$ (10% EtOAc/hexanes; PMA).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.22 (m, 2H), 7.18-7.12 (m, 3H), 7.09-7.05 (m, 2H), 6.84-6.79 (m, 2H), 4.46 (t, 1H, J = 5.2 Hz), 4.12-4.06 (m, 2H), 3.78 (s, 3H), 3.77-3.69 (m, 2H), 2.60-2.47 (m, 4H), 2.14-2.00 (m, 1H), 1.62-1.51 (m, 6H), 1.45-1.29 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 143.1, 135.0, 129.4, 128.5, 128.4, 125.7, 113.8, 102.8, 83.8, 67.0, 55.4, 36.9, 35.5, 33.1, 33.0, 32.4, 28.8, 27.5, 26.0.

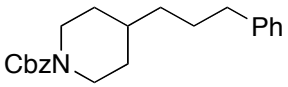
FT-IR (neat) 2928, 2856, 1512, 1245, 748, 700 cm^{-1}.

MS (EI) m/z (M^+) calcd for $C_{25}H_{34}O_3$: 382.2, found: 382.2.



(3-Cyclohexylpropyl)benzene (Table 3, entry 1) [170661-44-6]. Cyclohexyl bromide (163 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 152 mg (75%). Second run: 151 mg (75%).

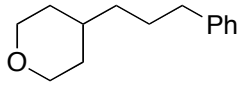
Table 3, entry 2. Cyclohexyl iodide (210 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 148 mg (73%). Second run: 158 mg (78%).



Benzyl 4-(3-phenylpropyl)piperidine-1-carboxylate (Table 3, entry 3). Benzyl 4-bromopiperidine-1-carboxylate (298 mg, 1.0 mmol) and a solution of the boron reagent prepared

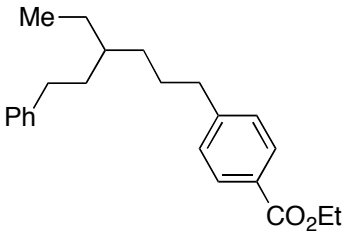
by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 217 mg (64%). Second run: 221 mg (66%).

Table 3, entry 4. Benzyl 4-iodopiperidine-1-carboxylate (345 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 216 mg (64%). Second run: 220 mg (65%).



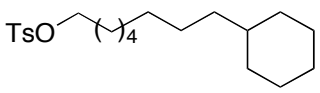
4-(3-Phenylproply)tetrahydro-2*H*-pyran (Table 3, entry 5). 4-Bromotetrahydropyran (165 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 144 mg (71%). 156 mg (76%).

Table 3, entry 6. 4-Iodotetrahydropyran (212 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 159 mg (78%). 155 mg (75%).



Ethyl 4-(4-ethyl-6-phenylhexyl)benzoate (Table 3, entry 7). (3-Bromopentyl)benzene (227 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of ethyl 4-allylbenzoate (prepared according to a literature procedure[6]) with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 260 mg (77%). Second run: 251 mg (74%).

Table 3, entry 8. (3-Iodopentyl)benzene (274 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of ethyl 4-allylbenzoate (prepared according to a literature procedure[6]) with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. First run: 254 mg (75%). Second run: 265 mg (78%).



8-Cyclohexyloctyl 4-methylbenzenesulfonate (Table 3, entry 9). 5-Chloropentyl 4-methylbenzenesulfonate (277 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylcyclohexane with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8

mmol) were used. The product was purified by flash chromatography (5% Et_2O/hexanes). Colorless oil. First run: 236 mg (64%). Second run: 230 mg (62%).

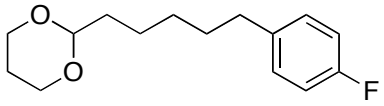
TLC: R_f = 0.5 (25% Et_2O/hexanes; $KMnO_4$).

^{1}H NMR ($CDCl_3$) δ 7.78 (d, 2H, *J* = 8.3 Hz), 7.34 (d, 2H, *J* = 8.3 Hz), 4.01 (t, 2H, *J* = 6.5 Hz), 2.44 (s, 3H), 1.71-1.55 (m, 7H), 1.32-1.07 (m, 16H), 0.90-0.78 (m, 2H).

^{13}C NMR ($CDCl_3$) δ 144.7, 133.6, 129.9, 128.0, 70.8, 37.8, 37.6, 33.6, 29.9, 29.5, 29.0, 28.9, 26.90, 26.87, 26.6, 25.4, 21.7.

FT-IR (neat) 3063, 3027, 2986, 2933, 2858, 1604, 1496, 1454, 1378, 1369, 1248, 1215, 1156, 1055, 859, 747, 699 cm^{-1}.

MS (ESI) *m*/*z* (M^+) calcd for $C_{21}H_{34}O_3S$: 366.2, found: 384.3 ($M^+ + NH_4$).



2-(5-(4-Fluorophenyl)pentyl)-1,3-dioxane (Table 3, entry 10). 2-(2-Bromoethyl)-1,3-dioxane (195 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of 1-allyl-4-fluorobenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5% Et_2O/hexanes). Pale-yellow oil. First run: 172 mg (68%). Second run: 181 mg (72%).

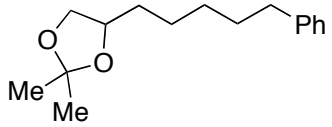
TLC: R_f = 0.3 (25% Et_2O/hexanes; UV).

^{1}H NMR ($CDCl_3$) δ 7.13-7.07 (m, 2H), 6.98-6.91 (m, 2H), 4.49 (t, 1H, *J* = 5.1 Hz), 4.12-4.06 (m, 2H), 3.79-3.70 (m, 2H), 2.56 (t, 2H, *J* = 7.6 Hz), 2.15-2.00 (m, 1H), 1.64-1.54 (m, 4H), 1.46-1.28 (m, 5H).

^{13}C NMR ($CDCl_3$) δ 129.8, 129.7, 115.2, 114.9, 102.4, 67.0, 35.3, 35.1, 31.6, 29.1, 26.0, 23.9.

FT-IR (neat) 2929, 2855, 1601, 1510, 1221, 1146, 998, 824 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{15}H_{21}FO_2$: 252.2, found: 251.1 (M^+ – H).



2,2-Dimethyl-4-(5-phenylpentyl)-1,3-dioxolane (Table 3, entry 11). 4-(2-Iodoethyl)-2,2-dimethyl-1,3-dioxolane (256 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 1.2 mL, 1.8 mmol) were used. The product was purified by flash chromatography (5% Et_2O/hexanes). Colorless oil. First run: 198 mg (79%). Second run: 194 mg (78%).

TLC: R_f = 0.5 (25% Et_2O/hexanes; $KMnO_4$).

^{1}H NMR ($CDCl_3$) δ 7.31-7.25 (m, 2H), 7.20-7.15 (m, 3H), 4.10-4.00 (m, 2H), 3.49 (t, 1H, *J* = 7.1 Hz), 2.61 (t, 2H, *J* = 7.6 Hz), 1.72-1.54 (m, 3H), 1.54-1.18 (m, 11H).

^{13}C NMR ($CDCl_3$) δ 142.8, 128.5, 128.4, 125.8, 108.7, 76.2, 69.7, 36.0, 33.7, 31.5, 29.4, 27.1, 25.9, 25.8.

FT-IR (neat) 3063, 3027, 2986, 2933, 2858, 1604, 1496, 1454, 1378, 1369, 1248, 1215, 1156, 1055, 859, 747, 699 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{16}H_{24}O_2$: 248.2, found: 248.2.

III. Kinetics Data

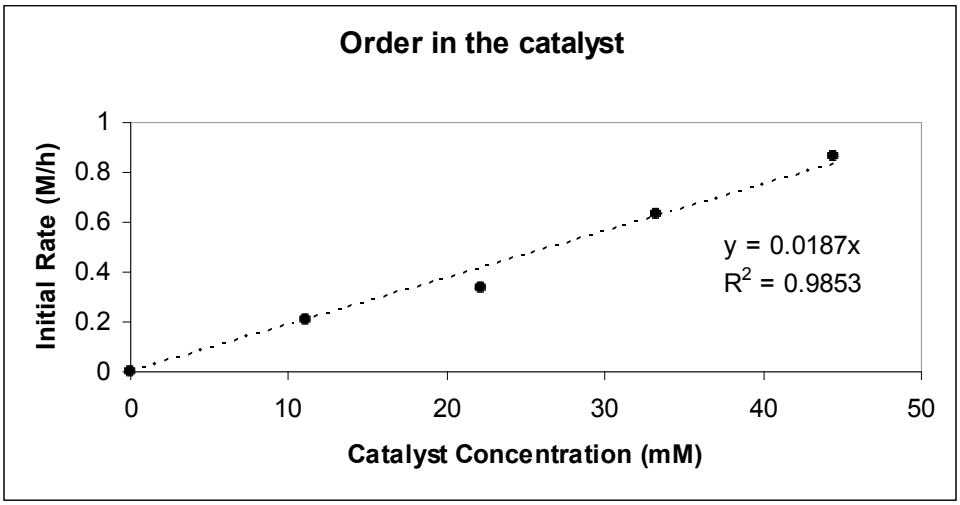
General procedure for the preparation of the boron reagent. In a glovebox, 9-BBN dimer (1.10 g, 4.5 mmol), *i*-Pr_2O (3.75 mL), and allylbenzene (1.19 mL, 9.0 mmol) were added to a 20-mL vial equipped with a stir bar. Then, the vial was capped, and the reaction mixture was stirred vigorously at 60 °C for 1 h. Next, the mixture was allowed to cool to r.t. and diluted with *i*-Pr_2O (6 mL). Then, KO*t*-Bu (0.73 g, 6.0 mmol) and *i*-BuOH (0.92 mL, 10 mmol) were added, and the resulting mixture was stirred at r.t. for 30 min.

General procedure for the Suzuki reaction. $NiBr_2$•diglyme (4.2 mg, 0.012 mmol), ligand **1** (3.6 μL, 0.016 mmol), and *i*-Pr_2O (0.10 mL) were added to a 4-mL vial equipped with a stir bar. The mixture was stirred for 30 min, resulting in the formation of a pale-blue slurry. Next, a solution of cyclohexyl bromide and tetradecane (calibrated internal standard) in *i*-Pr_2O were added, followed by the solution of the boron reagent. The reaction mixture was stirred vigorously at room temperature. Aliquots (100 μL) were removed after 5, 10, 15, and 20 minutes, and the amount of product was determined by GC analysis. The initial rates were determined by best fit of data from the first 20 minutes of reaction.

Order in the catalyst. Reactions were run with $[\text{electrophile}]_0$ = 0.37 M and $[\text{nucleophile}]_0$ = 0.67 M.

Table S1. Observed initial rates as a function of catalyst concentration.

$[\text{catalyst}]_0$ (mM)	rate_{obs} (M/h)
0	0
11	0.21
22	0.34
33	0.63
44	0.86

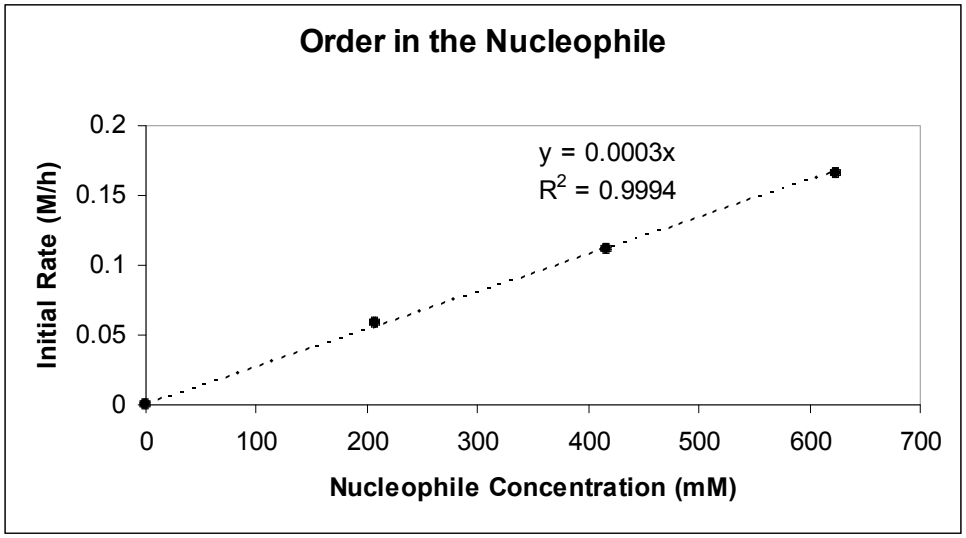


Order in the nucleophile. Reactions were run with $[\text{catalyst}]_0 = 0.022$ M and $[\text{electrophile}]_0 = 1.5$ M.

Table S2. Observed initial rates as a function of nucleophile concentration.

$[\text{nucleophile}]_0$ (mM)	rate_{obs} (M/h)
0	0
210	0.058
420	0.11
620	0.17

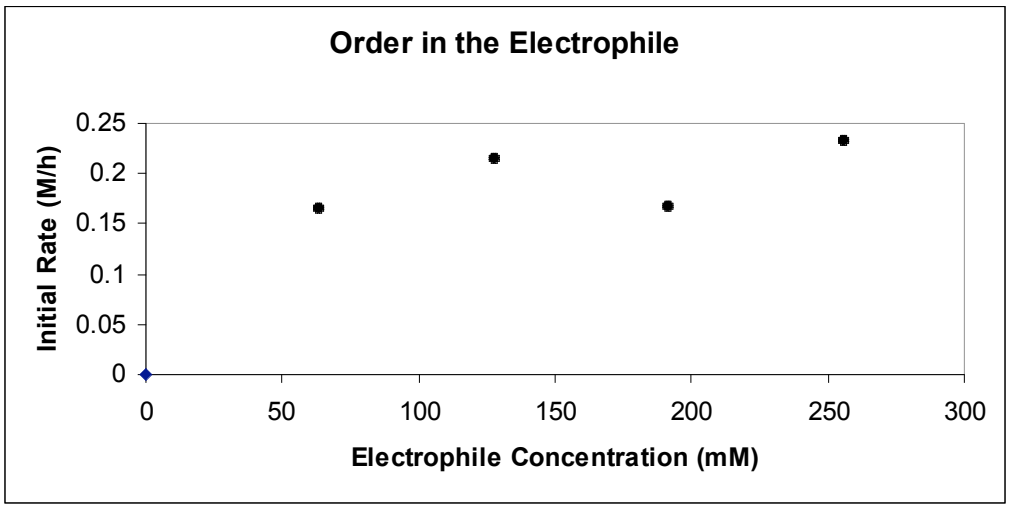
The rate of the reaction was not well-behaved at a higher concentration of nucleophile.



Order in the Electrophile. Reactions were run with $[catalyst]_0 = 0.015$ M and $[nucleophile]_0 = 0.92$ M.

Table S3. Observed initial rates as a function of electrophile concentration.

$[electrophile]_0$ (mM)	$rate_{obs}$ (M/h)
0	0
64	0.16
130	0.22
190	0.17
260	0.23



IV. ^{1}H NMR Spectra

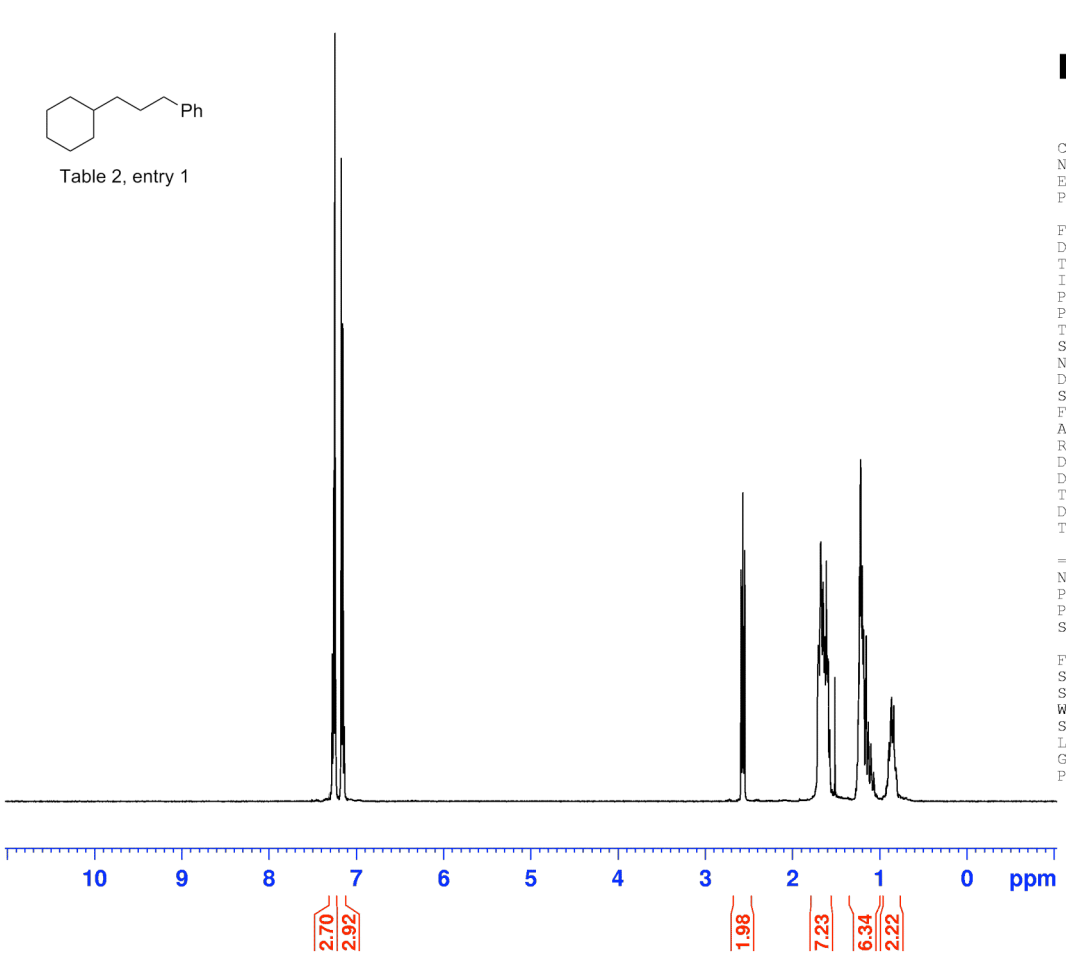


Table 2, entry 1

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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
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PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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SF          400.1300142 MHz
WDW                   EM
SSB                    0
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GB                     0
PC                  1.00
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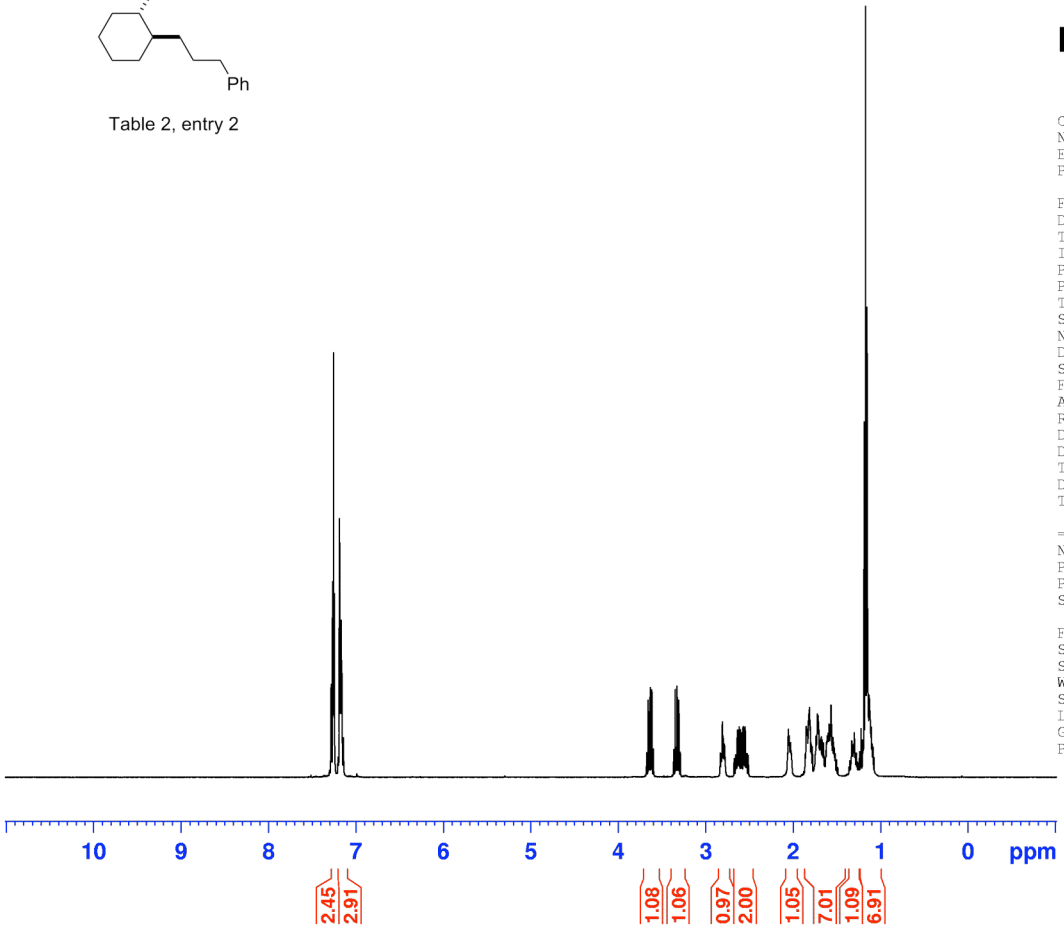
Table 2, entry 2

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EXPNO                  1
PROCNO                 1

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TD                 65536
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NS                    16
DS                     0
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FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   114
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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P1                 15.07 usec
PL1                 0.00 dB
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F2 - Processing parameters
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WDW                   EM
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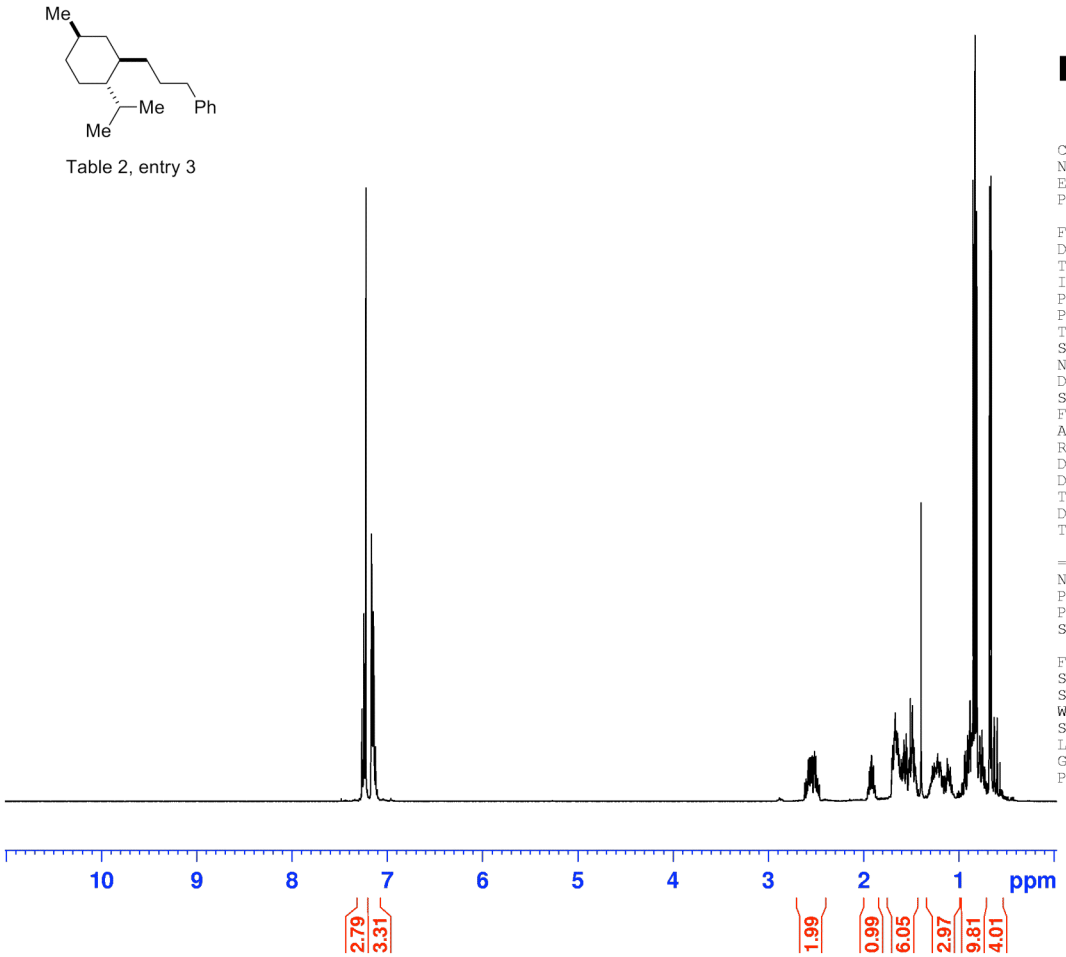

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Current Data Parameters
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EXPNO                  1
PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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WDW                   EM
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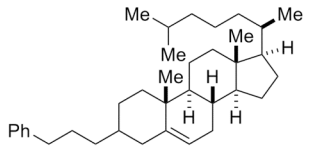

Table 2, entry 4


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EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
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Time               15.30
INSTRUM            spect
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TD                 65536
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NS                    16
DS                     0
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FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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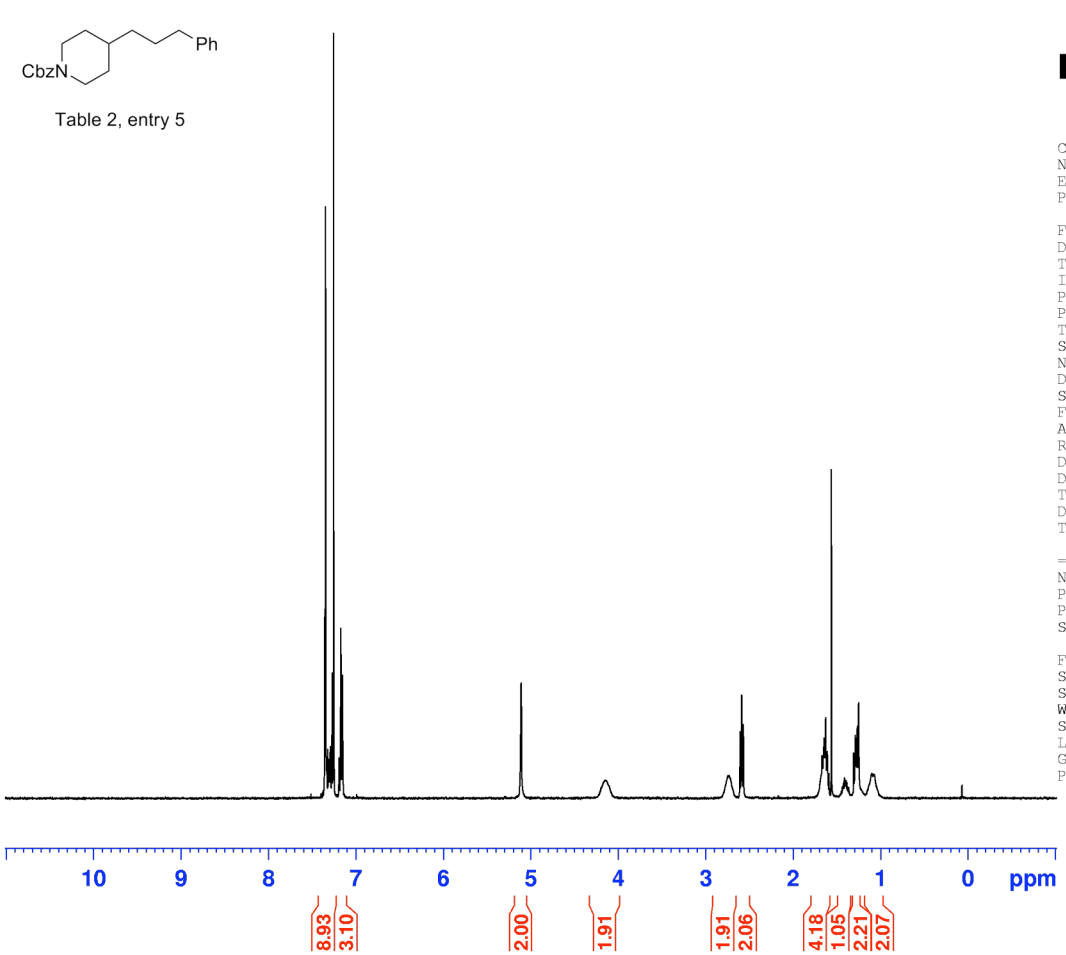

Table 2, entry 5

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PROCNO                 1

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DS                     0
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FIDRES          0.126314 Hz
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DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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PL1                 0.00 dB
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F2 - Processing parameters
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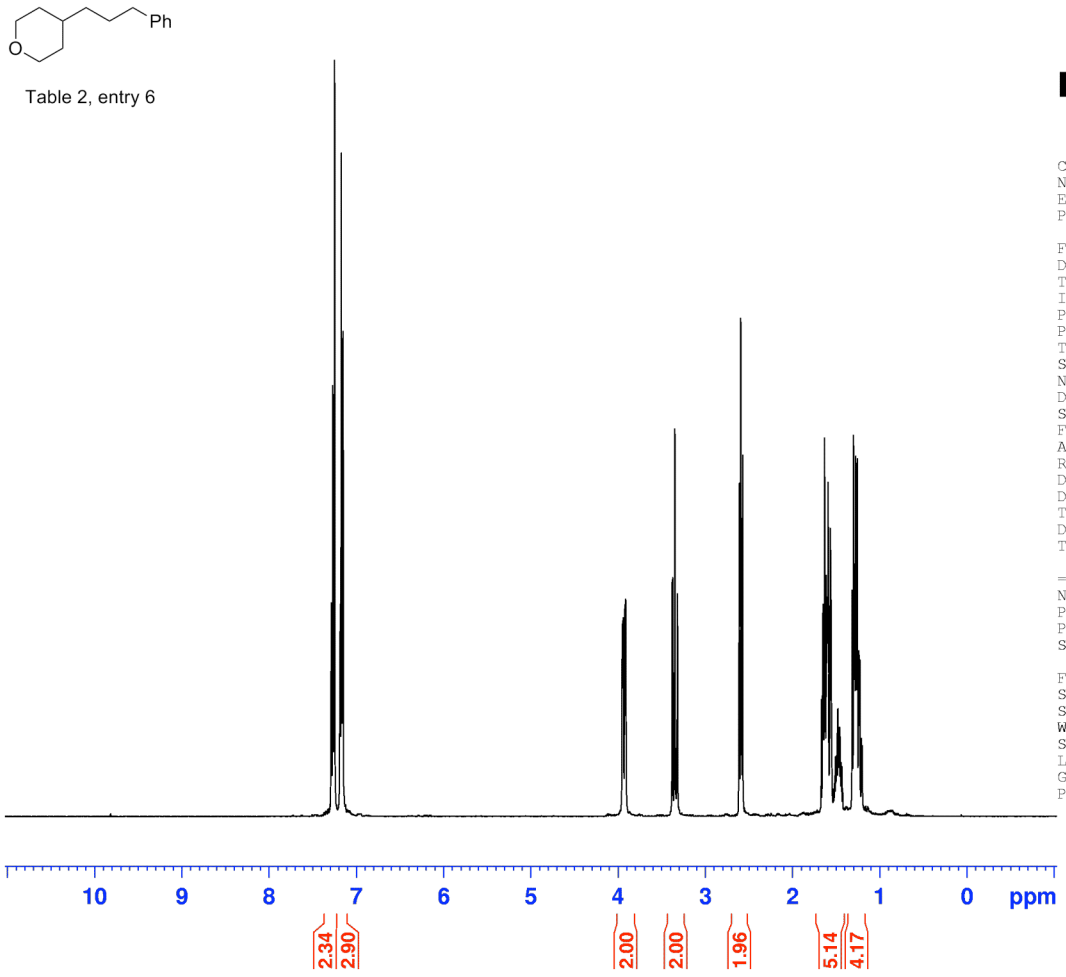

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EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
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INSTRUM            spect
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TD                 65536
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NS                    16
DS                     0
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FIDRES          0.126314 Hz
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RG                   128
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DE                 6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300139 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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Table 2, entry 7


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Current Data Parameters
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EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20081004
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INSTRUM                   spect
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SOLVENT
NS                           16
DS                            0
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FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                          128
DW                       60.400 usec
DE                        6.00 usec
TE                       298.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.07 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                 400.1300098 MHz
WDW                          EM
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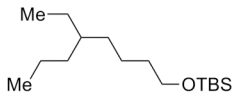

Table 2, entry 8


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EXPNO                         1
PROCNO                        1

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SOLVENT                   CDCl3
NS                           16
DS                            0
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                          128
DW                       60.400 usec
DE                        6.00 usec
TE                       298.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.07 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                 400.1300113 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```

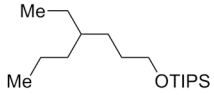


Table 2, entry 9


```
Current Data Parameters
NAME                  ZL-IV-028
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20081005
Time                      15.51
INSTRUM                   spect
PROBHD    5 mm BBO BB-1H
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            0
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                          128
DW                       60.400 usec
DE                        6.00 usec
TE                       298.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.07 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                 400.1300120 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```

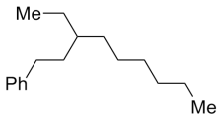


Table 2, entry 10


```
Current Data Parameters
NAME                  ZL-IV-015
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20081004
Time                      15.01
INSTRUM                   spect
PROBHD    5 mm BBO BB-1H
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            0
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                          128
DW                       60.400 usec
DE                        6.00 usec
TE                       298.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.07 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                  400.1300119 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```

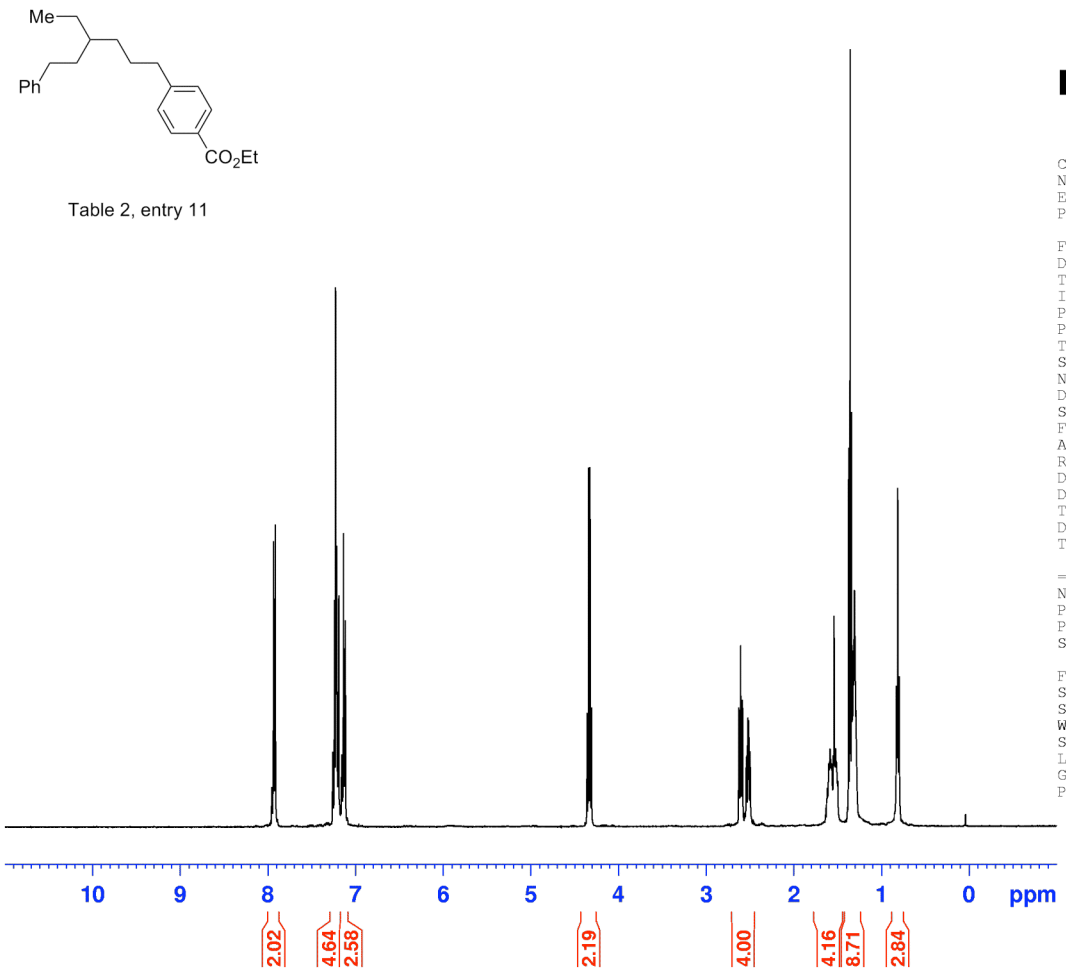


Table 2, entry 11

```
Current Data Parameters
NAME          ZL-IV-037A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081007
Time               10.42
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 456.1
DW                60.400 usec
DE                 6.00 usec
TE                292.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300220 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

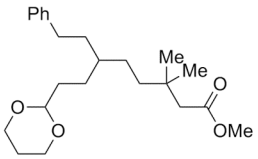


Table 2, entry 12


```
Current Data Parameters
NAME          ZL-IV-041A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081007
Time               10.57
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   362
DW                60.400 usec
DE                 6.00 usec
TE                292.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300097 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

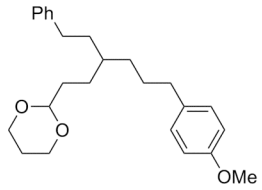


Table 2, entry 13

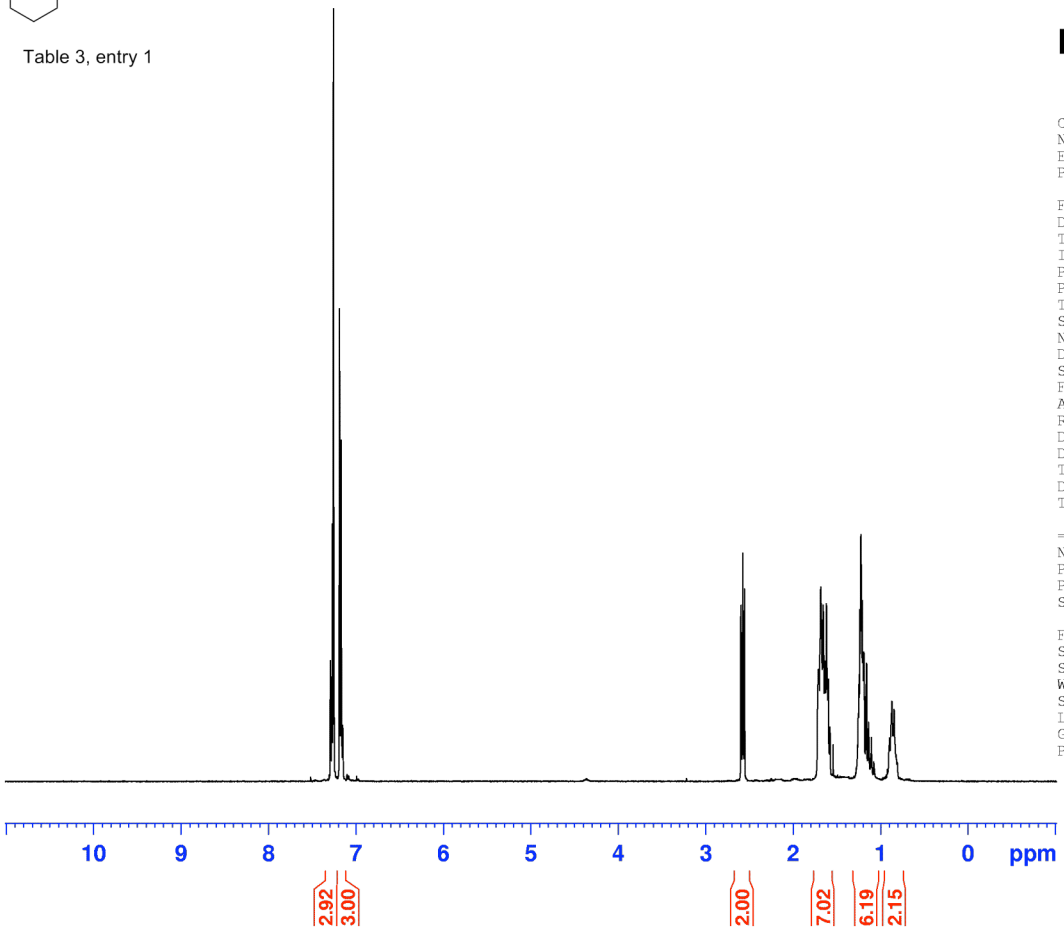

```
Current Data Parameters
NAME          ZL-IV-040B
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081008
Time               13.14
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                294.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300137 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Table 3, entry 1

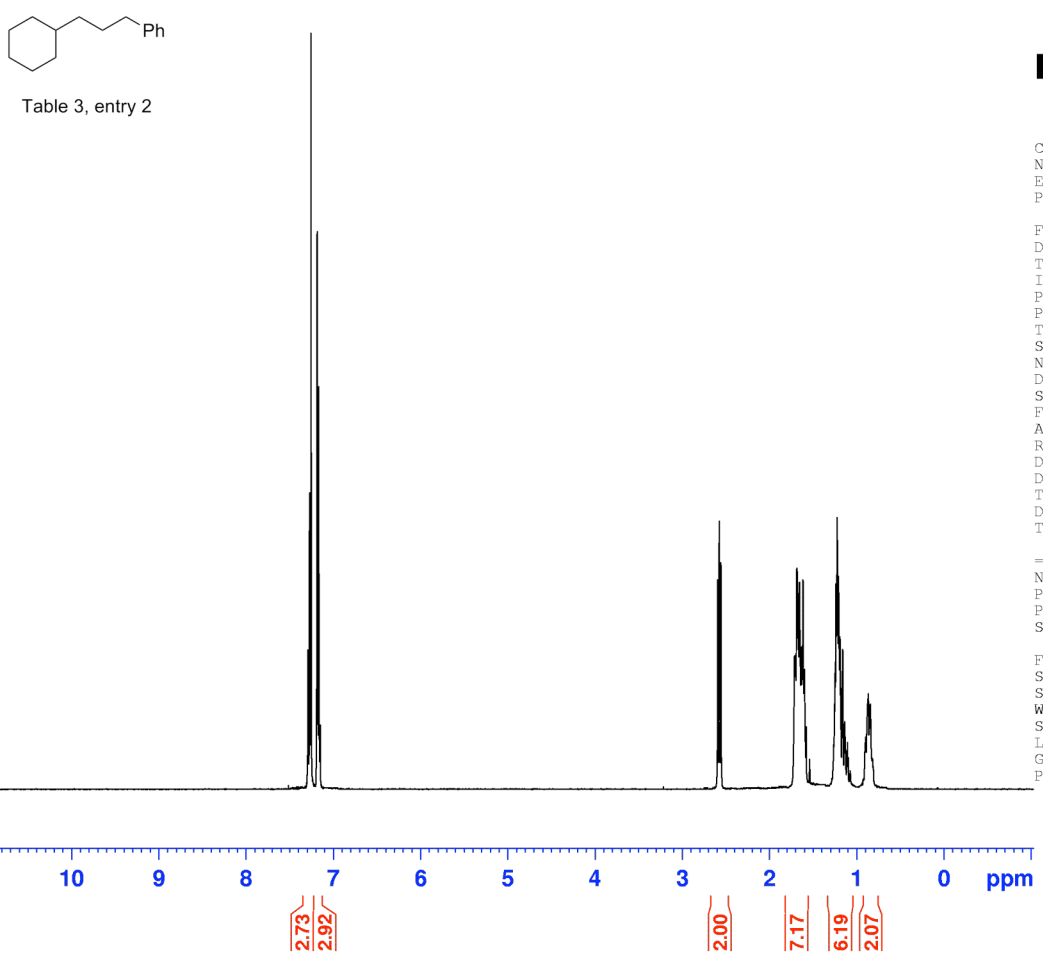



```
Current Data Parameters
NAME          ZL-IV-064A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081218
Time               12.05
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.07 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```




```
Current Data Parameters
NAME          ZL-IV-064C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081218
Time               12.50
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.07 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300099 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

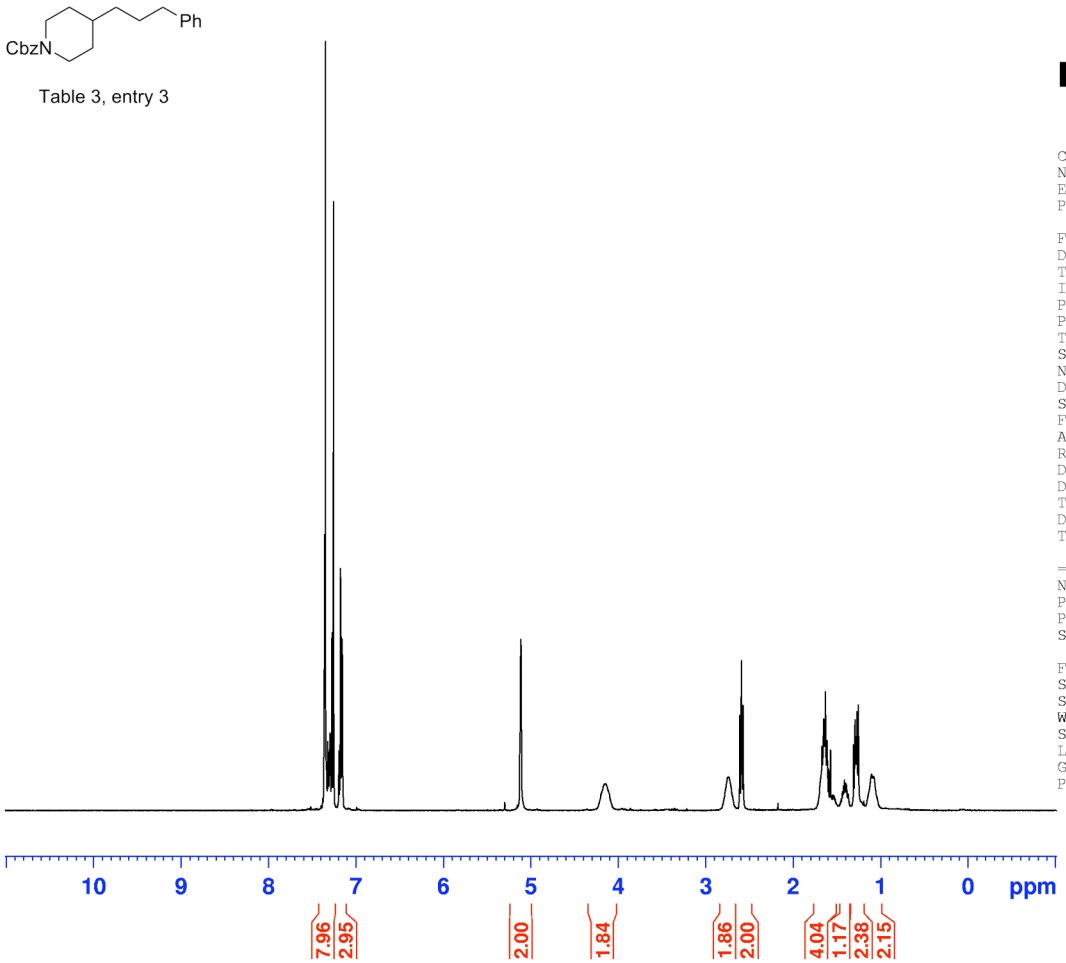


Table 3, entry 3

```
Current Data Parameters
NAME          ZL-IV-069C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081220
Time               16.35
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

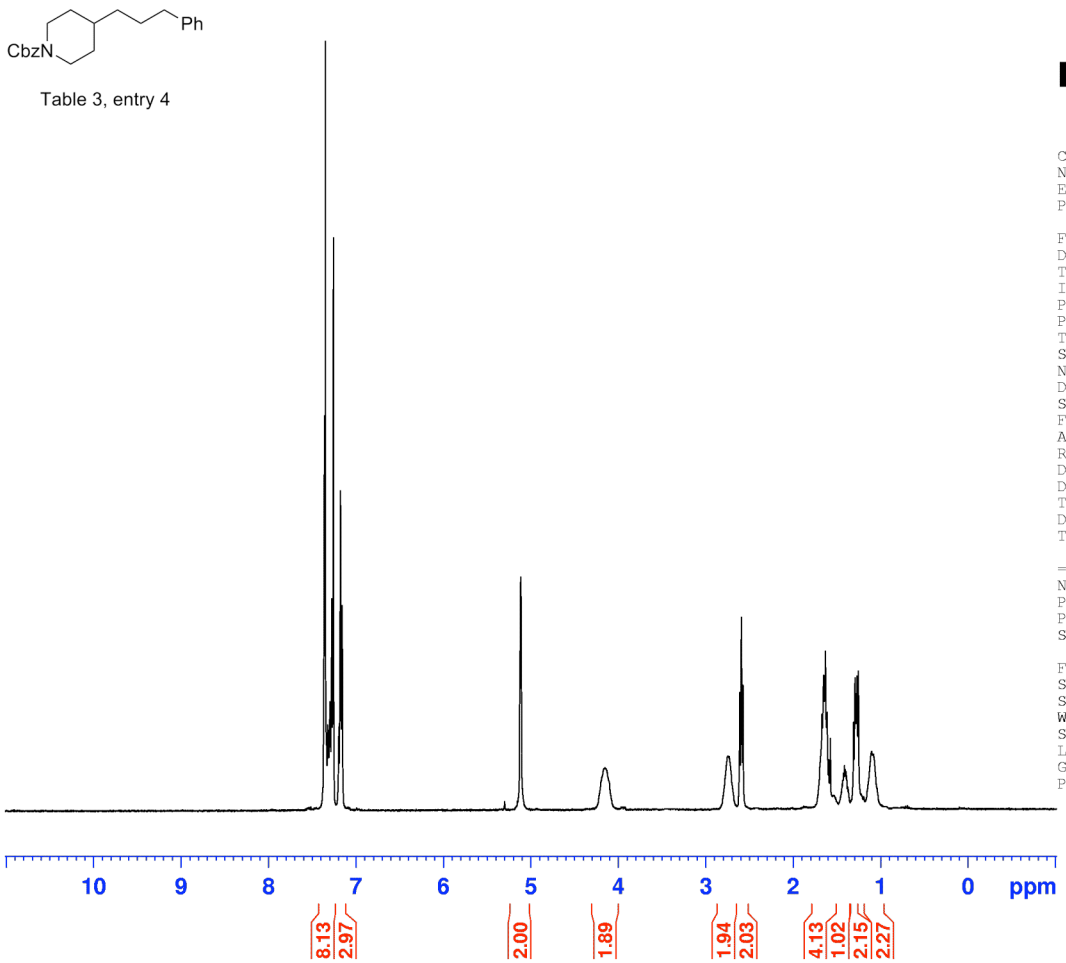


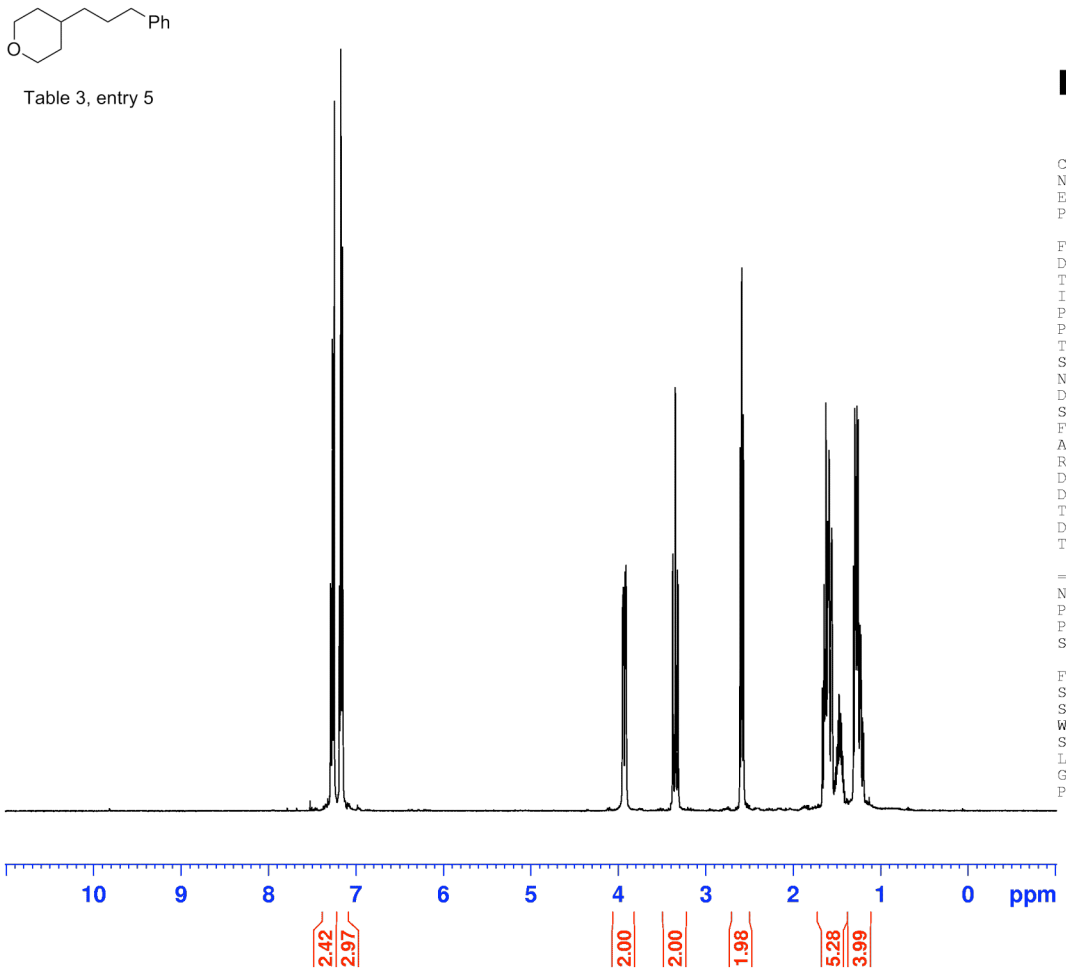
Table 3, entry 4

```
Current Data Parameters
NAME          ZL-IV-069A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081220
Time               15.13
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 456.1
DW                60.400 usec
DE                 6.00 usec
TE                291.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300094 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

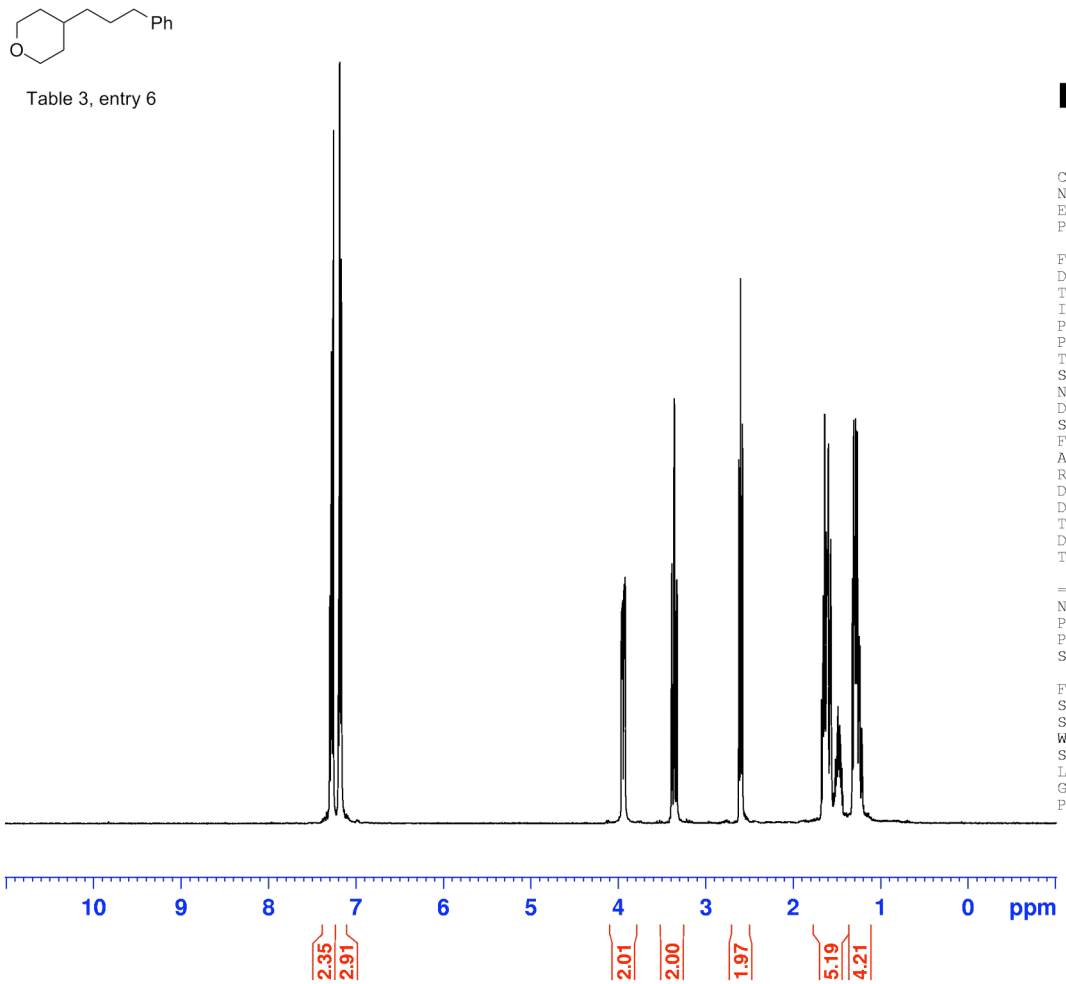



```
Current Data Parameters
NAME          ZL-IV-073B
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081218
Time               17.50
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300145 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



BRUKER

```
Current Data Parameters
NAME          ZL-IV-067B
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081218
Time               17.42
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

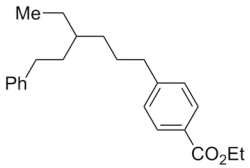
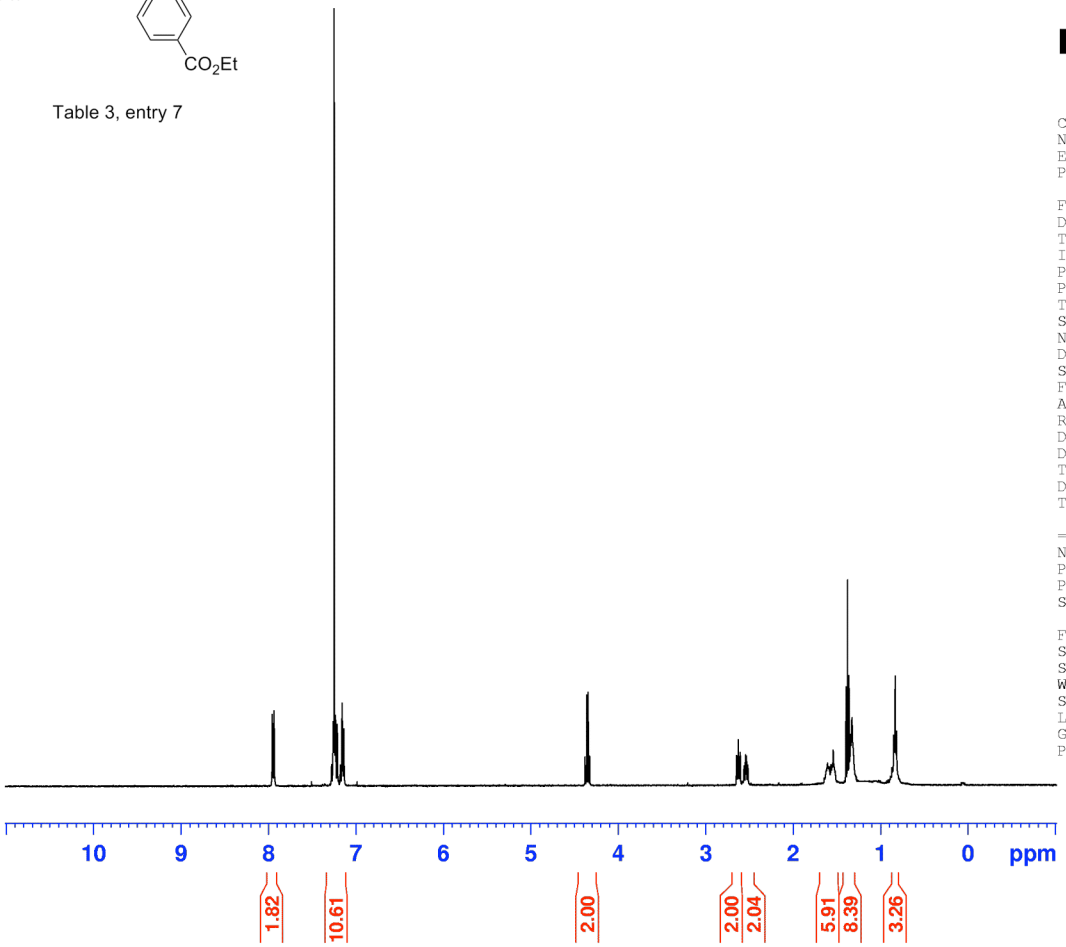


Table 3, entry 7




```
Current Data Parameters
NAME          ZL-IV-071A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081219
Time               16.28
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 456.1
DW                60.400 usec
DE                 6.00 usec
TE                291.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300128 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

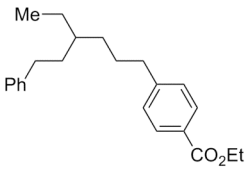
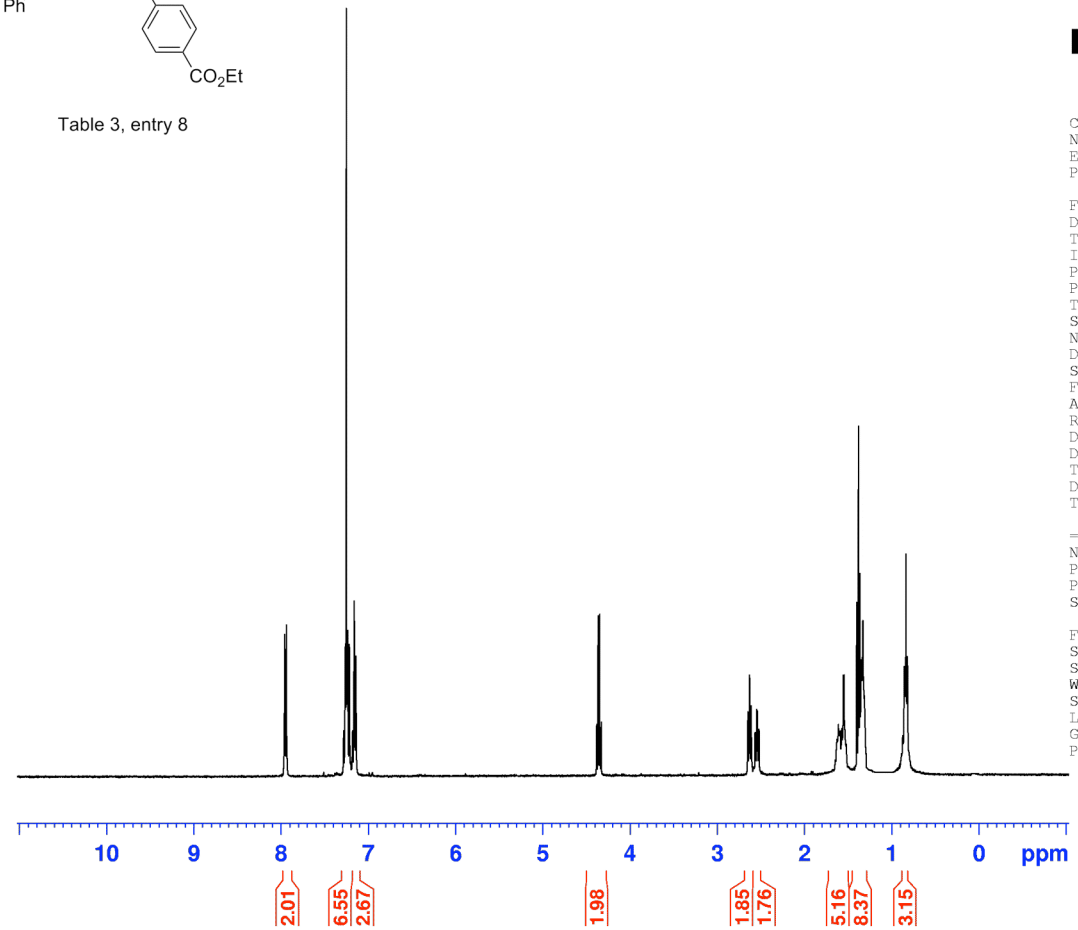


Table 3, entry 8




```
Current Data Parameters
NAME          ZL-IV-071C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081219
Time               17.11
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   512
DW                60.400 usec
DE                 6.00 usec
TE                291.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300112 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

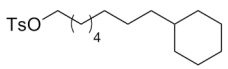
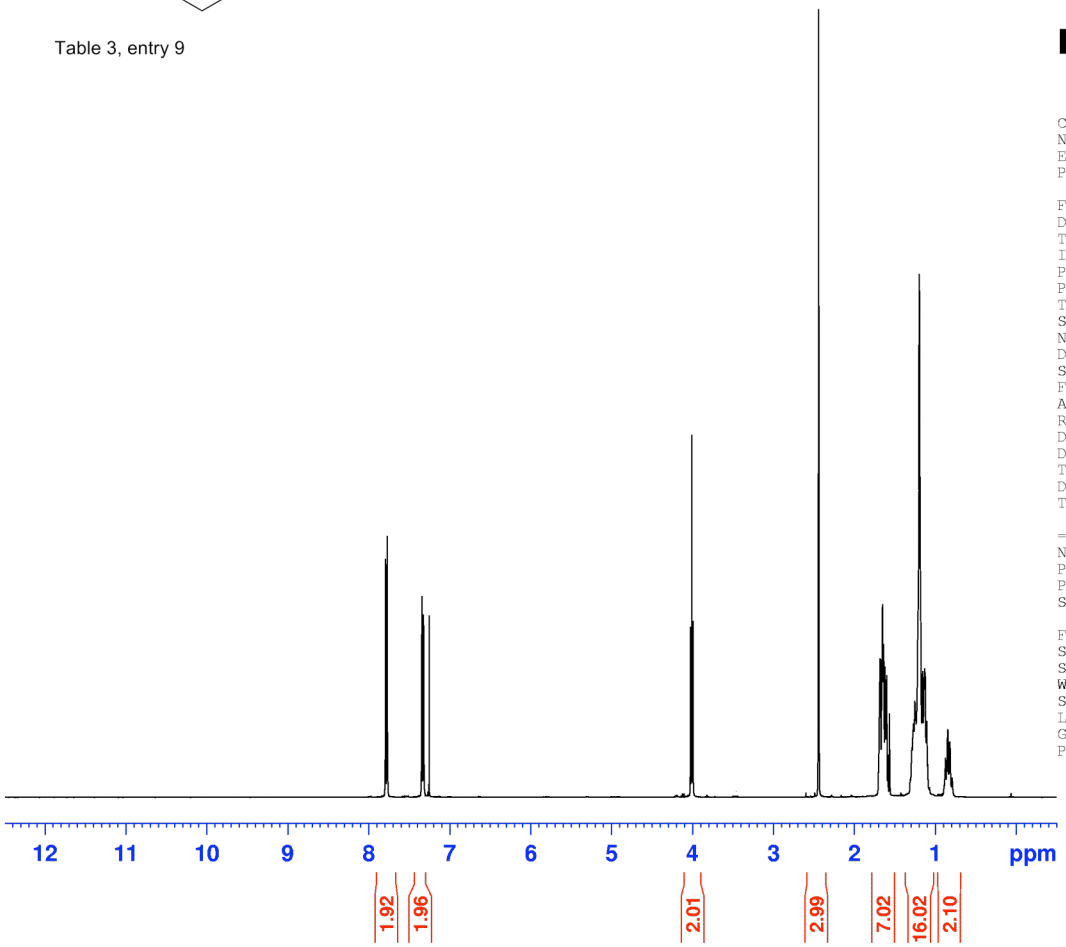


Table 3, entry 9




```
Current Data Parameters
NAME      ZL-V-200_1_col
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100417
Time               13.32
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300109 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

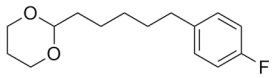
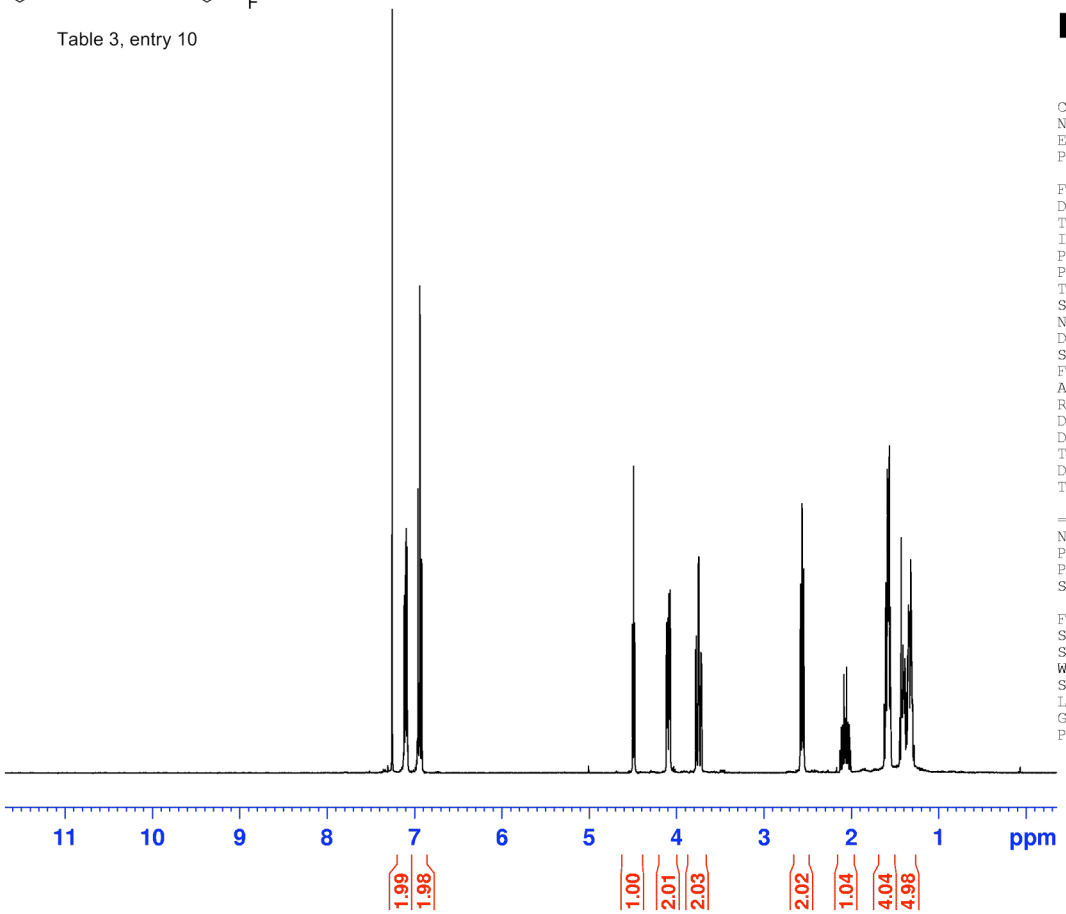


Table 3, entry 10




```
Current Data Parameters
NAME      ZL-V-190_Br_col1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20100416
Time               13.04
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   256
DW                60.400 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300103 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

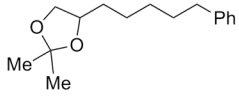


Table 3, entry 11


```
Current Data Parameters
NAME      ZL-V-201_1_col
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100417
Time               13.01
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 101.6
DW                60.400 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300091 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```