

Platinum-Catalyzed Intermolecular Hydroamination of Vinyl Arenes with Carboxamides

Hua Qian and Ross A. Widenhoefer*

P. M. Gross Chemical Laboratory

Duke University

Durham, North Carolina 27708-0346

Supporting Information

Experimental procedures and spectroscopic and analytical data for new compounds and hydroamination products (20 pages).

General Methods. All reactions were performed in sealed glass tubes under a nitrogen atmosphere. NMR spectra were obtained on a Varian spectrometer operating at 400 MHz for ¹H and 100 MHz for ¹³C in CDCl₃ unless noted otherwise. Gas chromatography was performed on a Hewlett-Packard 5890 gas chromatography equipped with a 25 m polydimethylsiloxane capillary column. Flash column chromatography was performed employing 200-400 mesh silica gel (EM). Thin layer chromatography (TLC) was performed on silica gel 60 F₂₅₄ eluting with a 2:1 mixture of hexanes and ethyl acetate unless noted otherwise. IR spectra were obtained on a Bomen MB-100 FT-IR spectrometer. Elemental analyses were performed by Complete Analysis Laboratories (Parsippany, NJ). 1,4-Dioxane (Acros, anhydrous), [PtCl₂(H₂C=CH₂)]₂ (**1**) (Strem), phosphines (Strem), 2-oxazolidone, *p*-toluenesulfonamide, benzo[1,3]dioxole-5-carboxamide, and commercially available carboxamides and vinyl arenes (Acros or Aldrich) were used as received. Mesitylene was distilled over CaH₂ under nitrogen prior to use.

Substrates

Methyl terephthalamate (Table 2, entry 6).¹ A solution of methyl 4-chlorocarbonylbenzoate (1.0 g, 5.0 mmol) in THF (15 mL) was added slowly to concentrated aqueous ammonium hydroxide (16 N, 30 mL) at 0 °C and the resulting suspension was stirred for 1 h. The resulting precipitate was collected by filtration and recrystallized from methanol to give methyl terephthalamate as colorless needles (0.43 g, 48%). mp 210-212 °C.

¹H NMR (DMF-*d*₇): δ 8.32 (br s, 1 H), 8.14 (d, *J* = 8.8 Hz, 2 H), 8.08 (d, *J* = 8.8 Hz, 2 H), 7.64 (br s, 1 H), 3.94 (s, 3 H). ¹³C{¹H} NMR (DMF-*d*₇): δ 168.5, 167.0, 139.8, 133.3, 130.3, 128.9, 53.0.

4-(*tert*-Butyldimethylsiloxy)benzamide (Table 2, entry 13). A solution of 4-hydroxybenzamide (1.5 g, 10.9 mmol), TBDMSCl (2.0 g, 13.3 mmol), and imidazole (1.5 g, 22.0 mmol) in DMF (25 mL) was stirred at room temperature for 2 days. The reaction mixture was quenched with water and extracted with chloroform. The combined organic extracts were washed (water), dried (MgSO_4), and concentrated under vacuum. Chromatography of the residue (SiO_2 ; hexanes–ethyl acetate = 2:1) gave 4-(*tert*-butyldimethylsiloxy)benzamide (1.72 g, 63%) as a white solid. mp 143–144 °C. TLC: R_f = 0.11. ^1H NMR: δ 7.72 (d, J = 8.8 Hz, 2 H), 6.87 (d, J = 8.8 Hz, 2 H), 5.95 (br s, 2 H), 0.98 (s, 9 H), 0.22 (s, 6 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 169.4, 159.5, 129.5, 126.6, 120.4, 25.9, 18.6, –4.1. IR (neat, cm^{-1}): 3405 (s), 1653 (s). Anal. Calcd (found) for $\text{C}_{13}\text{H}_{21}\text{NO}_2\text{Si}$: C, 62.11 (61.97); H, 8.42 (8.58); N, 5.57 (5.71).

Hydroamination Products

N-(1-*p*-Tolylethyl)benzamide (2).² A solution of benzamide (121 mg, 1.0 mmol), 4-methylstyrene (0.53 mL, 4.0 mmol), **1** (15 mg, 0.025 mmol), and $\text{P}(4\text{-C}_6\text{H}_4\text{CF}_3)_3$ (23 mg, 0.050 mmol) in mesitylene (0.5 mL) was heated at 140 °C for 24 h, cooled to room temperature, and chromatographed (SiO_2 ; hexanes–ethyl acetate = 10:1 → 3:1) to give **2** (204 mg, 85%) as a white solid. In a separate, identical experiment, the crude reaction mixture was cooled to room temperature and chromatographed (SiO_2 ; hexanes) to give (*E*)-1,3-bis(*p*-tolyl)-1-butene (65 mg, 14% based on 4-methylstyrene) as a colorless oil, which was identified by comparison to an authentic sample.³

For 2: mp 119–120 °C. TLC: R_f = 0.55. ^1H NMR: δ 7.77–7.75 (m, 2 H), 7.51–7.47 (m, 1 H), 7.44–7.40 (m, 2 H), 7.29 (d, J = 8.0 Hz, 2 H), 7.17 (d, J = 8.0 Hz, 2 H), 6.29 (br d, J

= 6.4 Hz, 1 H), 5.31 (quintet, J = 7.2 Hz, 1 H), 2.34 (s, 3 H), 1.60 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.8, 140.5, 137.5, 131.8, 129.8, 128.9, 127.2, 126.6, 49.3, 22.0, 21.4.

All remaining hydroamination reactions were performed employing a procedure analogous to that used to synthesize **2** unless noted otherwise. Reaction temperatures and yields are provided in Table 2.

N-(1-Phenylethyl)benzamide (Table 2, entry 1).⁴ Reaction time = 45 h. ^1H NMR: δ 7.75 (d, J = 8.0 Hz, 2 H), 7.48-7.23 (m, 8 H), 6.35 (br s, 1 H), 5.32 (quintet, J = 6.8 Hz, 1 H), 1.58 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.9, 143.5, 134.9, 131.8, 129.1, 128.9, 127.8, 127.3, 126.6, 49.5, 22.1.

N-[1-(4-Chlorophenyl)ethyl]benzamide (Table 2, entry 2).⁵ Reaction time = 72 h. ^1H NMR: δ 7.74-7.72 (m, 2 H), 7.50-7.46 (m, 1 H), 7.42-7.38 (m, 2 H), 7.29 (s, 4 H), 6.25 (br s, 1 H), 5.28 (quintet, J = 6.8 Hz, 1 H), 1.56 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.9, 142.1, 134.7, 133.5, 131.9, 129.2, 128.9, 128.0, 127.2, 49.0, 22.1.

4-Methoxy-N-(1-p-tolylethyl)benzamide (Table 2, entry 3). Reaction time = 48 h. White solid. mp 148-150 °C. TLC: R_f = 0.36. ^1H NMR: δ 7.70 (d, J = 8.4 Hz, 2 H), 7.25 (d, J = 8.0 Hz, 2 H), 7.13 (d, J = 8.0 Hz, 2 H), 6.87 (d, J = 8.4 Hz, 2 H), 6.21 (br d, J = 7.2 Hz, 1 H), 5.26 (quintet, J = 7.2 Hz, 1 H), 3.81 (s, 3 H), 2.31 (s, 3 H), 1.55 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.3, 162.5, 140.7, 137.4, 129.7, 129.0, 127.3, 126.5, 114.0, 55.7, 49.2, 22.1, 21.4. IR (neat, cm^{-1}): 3326 (m), 1632 (s). Anal. Calcd (found) for $\text{C}_{17}\text{H}_{19}\text{NO}_2$: C, 75.81 (75.59); H, 7.11 (7.10); N, 5.20 (5.23).

4-Bromo-N-(1-p-tolylethyl)benzamide (Table 2, entry 4). Reaction time = 24 h. White solid. mp 150-151 °C. TLC: R_f = 0.57. ^1H NMR: δ 7.58 (d, J = 7.6 Hz, 2 H), 7.47 (d, J = 7.6 Hz, 2 H), 7.22 (d, J = 6.0 Hz, 2 H), 7.12 (d, J = 7.2 Hz, 2 H), 6.52 (br s, 1 H), 5.22

(quintet, $J = 6.8$ Hz, 1 H), 2.31 (s, 3 H), 1.54 (d, $J = 6.4$ Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 165.9, 140.3, 137.5, 133.7, 132.0, 129.7, 128.9, 126.5, 126.3, 49.4, 21.9, 21.4. IR (neat, cm^{-1}): 3300 (m), 1638 (s). Anal. Calcd (found) for $\text{C}_{16}\text{H}_{16}\text{BrNO}$: C, 60.39 (60.26); H, 5.07 (4.85); N, 4.40 (4.13).

N-(1-p-Tolylethyl)-4-trifluoromethylbenzamide (Table 2, entry 5). Reaction time = 24 h. White solid. mp 143-144 °C. TLC: $R_f = 0.69$. ^1H NMR: δ 7.83 (d, $J = 8.0$ Hz, 2 H), 7.64 (d, $J = 8.4$ Hz, 2 H), 7.25 (d, $J = 8.0$ Hz, 2 H), 7.15 (d, $J = 7.6$ Hz, 2 H), 6.34 (br d, $J = 6.8$ Hz, 1 H), 5.27 (quintet, $J = 6.8$ Hz, 1 H), 2.32 (s, 3 H), 1.58 (d, $J = 6.8$ Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 165.7, 140.2, 138.2, 137.6, 133.4 (q, $^2J_{\text{CF}} = 32$ Hz), 129.7, 127.8, 126.5, 125.8 (q, $^3J_{\text{CF}} = 3.5$ Hz), 124.0 (q, $^1J_{\text{CF}} = 271$ Hz), 49.6, 21.9, 21.3. ^{19}F NMR: δ -63.4. IR (neat, cm^{-1}): 3289 (m), 1643 (s). Anal. Calcd (found) for $\text{C}_{17}\text{H}_{16}\text{F}_3\text{NO}$: C, 66.44 (66.26); H, 5.25 (5.32); N, 4.56 (4.84).

Methyl N-(1-p-tolylethyl)terephthalamate (Table 2, entry 6). Reaction time = 30 h. White solid. mp 153-154 °C. TLC: $R_f = 0.30$. ^1H NMR (Figure S1): δ 8.07 (d, $J = 8.8$ Hz, 2 H), 7.81 (d, $J = 8.8$ Hz, 2 H), 7.29 (d, $J = 8.0$ Hz, 2 H), 7.18 (d, $J = 8.0$ Hz, 2 H), 6.37 (d, $J = 7.8$ Hz, 1 H), 5.30 (quintet, $J = 6.8$ Hz, 1 H), 3.93 (s, 3 H), 2.34 (s, 3 H), 1.61 (d, $J = 6.8$ Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR (Figure S2): δ 166.6, 166.0, 140.1, 138.9, 137.7, 133.0, 130.1, 129.8, 127.3, 126.6, 52.7, 49.6, 21.9, 21.4. IR (neat, cm^{-1}): 3294 (br s), 1723 (s), 1635 (s), 1538 (s), 1275 (s). HRMS Calcd (found) for $\text{C}_{18}\text{H}_{19}\text{NO}_3$: 297.1365 (297.1372).

4-Methyl-N-(1-p-tolylethyl)benzamide (Table 2, entry 7). Reaction time = 24 h. White solid. mp 116-118 °C. TLC: $R_f = 0.56$. ^1H NMR: δ 7.66 (d, $J = 8.0$ Hz, 2 H), 7.29 (d, $J = 8.0$ Hz, 2 H), 7.21 (d, $J = 8.0$ Hz, 2 H), 7.17 (d, $J = 8.0$ Hz, 2 H), 6.28 (br d, $J = 7.8$ Hz, 1 H), 5.30 (quintet, $J = 6.8$ Hz, 1 H), 2.39 (s, 3 H), 2.34 (s, 3 H), 1.59 (d, $J = 6.8$ Hz, 3

H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.8, 142.1, 140.6, 137.4, 132.1, 129.7, 129.5, 127.2, 126.5, 49.2, 22.0, 21.8, 21.4. IR (neat, cm^{-1}): 3305 (m), 1638 (s). Anal. Calcd (found) for $\text{C}_{17}\text{H}_{19}\text{NO}$: C, 80.60 (80.36); H, 7.56 (7.32); N, 5.53 (4.99).

2-Methyl-N-(1-p-tolylethyl)benzamide (Table 2, entry 8). Reaction time = 48 h. White solid. mp 126-127 °C. TLC: R_f = 0.61. ^1H NMR: δ 7.32-7.24 (m, 4 H), 7.18-7.13 (m, 4 H), 5.91 (br s, 1 H), 5.27 (quintet, J = 6.8 Hz, 1 H), 2.40 (s, 3 H), 2.32 (s, 3 H), 1.55 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 169.4, 140.4, 137.5, 136.4, 131.3, 130.1, 129.7, 126.9, 126.5, 126.0, 49.1, 22.1, 21.4, 20.1. IR (neat, cm^{-1}): 3258 (m), 1638 (s). Anal. Calcd (found) for $\text{C}_{17}\text{H}_{19}\text{NO}$: C, 80.60 (80.48); H, 7.56 (7.51); N, 5.53 (5.79).

N-(1-p-Tolylethyl)-2-naphthalcarboxamide (Table 2, entry 9). Reaction time = 24 h. White solid. mp 131-133 °C. TLC: R_f = 0.62. ^1H NMR: δ 8.28 (s, 1 H), 7.89-7.82 (m, 4 H), 7.57-7.50 (m, 2 H), 7.33 (d, J = 8.0 Hz, 2 H), 7.18 (d, J = 8.0 Hz, 2 H), 6.60 (br d, J = 7.2 Hz, 1 H), 5.37 (quintet, J = 6.8 Hz, 1 H), 2.35 (s, 3 H), 1.64 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.9, 140.5, 137.5, 135.0, 132.9, 132.2, 129.7, 129.2, 128.7, 128.0, 127.9, 127.6, 127.0, 126.6, 124.0, 49.4, 22.0, 21.4. IR (neat, cm^{-1}): 3300 (m), 1638 (s). Anal. Calcd (found) for $\text{C}_{20}\text{H}_{19}\text{NO}$: C, 83.01 (82.87); H, 6.62 (6.55); N, 4.84 (5.02).

N-[1-(p-tolyl)ethyl]-benzo[1,3]dioxole-5-carboxamide (Table 2, entry 10). Reaction time = 24 h. Pale yellow solid. mp 137-138 °C. TLC: R_f = 0.39. ^1H NMR (Figure S3): δ 7.26-7.22 (m, 4 H), 7.12 (d, J = 8.0 Hz, 2 H), 6.75 (d, J = 8.0 Hz, 1 H), 6.31 (d, J = 7.2 Hz, 1 H), 5.96 (s, 2 H), 5.22 (quintet, J = 6.8 Hz, 1 H), 2.30 (s, 3 H), 1.53 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR (Figure S4): δ 166.1, 150.5, 148.2, 140.6, 137.4, 129.7, 129.2, 126.5, 121.8, 108.2, 108.0, 101.9, 49.3, 22.0, 21.3. IR (neat, cm^{-1}): 3435 (br s), 1635 (s), 1479 (s), 1256 (s). HRMS Calcd (found) for $\text{C}_{17}\text{H}_{17}\text{NO}_3$: 283.1208 (283.1205).

N-[1-(*o*-tolyl)ethyl]benzamide (Table 2, entry 11). Reaction time = 72 h. Pale yellow solid. mp 130-131 °C. TLC: R_f = 0.33. ^1H NMR (Figure S5): δ 7.76 (d, J = 8.4 Hz, 2 H), 7.49 (7, J = 7.6 Hz, 1 H), 7.43-7.37 (m, 3 H), 7.26-7.19 (m, 3 H), 6.31 (br s, 1 H), 5.51 (quintet, J = 7.2 Hz, 1 H), 2.43 (s, 3 H), 1.59 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR (Figure S6): δ 166.7, 141.3, 136.5, 134.9, 131.8, 131.2, 128.9, 127.8, 127.2, 126.7, 125.1, 46.2, 21.4, 19.5. IR (neat, cm^{-1}): 3299 (m), 1630 (s). HRMS Calcd (found) for $\text{C}_{16}\text{H}_{17}\text{NO}$ (M^+): 239.1310 (239.1315).

N-[1-(2,4-Dimethylphenyl)ethyl]benzamide (Table 2, entry 12).⁶ Reaction time = 24 h. ^1H NMR: δ 7.73 (d, J = 8.0 Hz, 2 H), 7.45 (t, J = 8.0 Hz, 1 H), 7.37 (d, J = 8.0 Hz, 2 H), 7.25 (d, J = 8.0 Hz, 1 H), 7.01 (d, J = 9.2 Hz, 2 H), 6.38 (br s, 1 H), 5.45 (quintet, J = 6.8 Hz, 1 H), 2.36 (s, 3 H), 2.29 (s, 3 H), 1.55 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.6, 138.4, 137.3, 136.3, 134.8, 131.9, 131.6, 128.8, 127.2, 125.1, 45.9, 21.3, 21.2, 19.4.

4-(*tert*-Butyldimethylsiloxy)-N-[1-(2,4-dimethylphenyl)ethyl]benzamide (Table 2, entry 13). Reaction time = 44 h. Pale yellow solid. mp 103-105 °C. TLC: R_f = 0.64. ^1H NMR: δ 7.62 (d, J = 8.4 Hz, 2 H), 7.23 (d, J = 8.0 Hz, 1 H), 7.01 (d, J = 8.0 Hz, 1 H), 6.99 (s, 1 H), 6.80 (d, J = 8.8 Hz, 2 H), 6.08 (br d, J = 7.6 Hz, 1 H), 5.42 (quintet, J = 6.8 Hz, 1 H), 2.35 (s, 3 H), 2.28 (s, 3 H), 1.54 (d, J = 6.4 Hz, 3 H), 0.94 (s, 9 H), 0.17 (s, 6 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 166.3, 159.0, 138.5, 137.3, 136.4, 131.9, 128.9, 127.8, 127.2, 125.1, 120.3, 45.9, 25.9, 21.35, 21.26, 19.4, 18.6, -4.1. IR (neat, cm^{-1}): 3310 (m), 1638 (s). Anal. Calcd (found) for $\text{C}_{23}\text{H}_{33}\text{NO}_2\text{Si}$: C, 72.01 (71.90); H, 8.67 (8.94); N, 3.65 (4.00).

N-(1-Indanyl)benzamide (Table 2, entry 14).⁷ Reaction time = 48 h. ^1H NMR: δ 7.76-7.74 (m, 2 H), 7.48-7.44 (m, 1 H), 7.40-7.36 (m, 2 H), 7.31 (d, J = 7.2 Hz, 1 H), 7.24-7.17 (m, 3 H), 6.53 (br d, J = 7.6 Hz, 1 H), 5.63 (q, J = 7.6 Hz, 1 H), 2.98 (ddd, J = 4.0, 8.8,

16 Hz, 1 H), 2.87 (td, J = 8.0, 16 Hz, 1 H), 2.63 (dtd, J = 3.6, 6.8, 16.8 Hz, 1 H), 1.89 (qd, J = 8.4, 12.8 Hz, 1 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 167.5, 143.8, 143.5, 134.8, 131.8, 128.9, 128.4, 127.3, 127.1, 125.2, 124.4, 55.4, 34.4, 30.6.

3-(1-p-Tolylethyl)-2-oxazolidone (Table 15).⁸ Reaction time = 48 h. ^1H NMR: δ 7.17 (d, J = 8.0 Hz, 2 H), 7.11 (d, J = 8.0 Hz, 2 H), 5.12 (q, J = 6.8 Hz, 1 H), 4.23 (q, J = 8.4 Hz, 1 H), 4.15 (q, J = 8.8 Hz, 1 H), 3.44 (q, J = 8.4 Hz, 1 H), 3.10 (q, J = 8.4 Hz, 1 H), 2.29 (s, 3 H), 1.51 (d, J = 7.2 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 158.2, 137.7, 136.8, 129.5, 127.1, 62.1, 51.4, 40.2, 21.2, 16.6.

(N-1-p-Tolylethyl)valeramide (Table 2, entry 16). Reaction time = 48 h. Viscous pale yellow oil. TLC (hexanes–EtOAc = 1:1): R_f = 0.45. ^1H NMR (Figure S7): δ 7.21 (d, J = 8.0 Hz, 2 H), 7.14 (d, J = 8.0 Hz, 2 H), 5.71 (d, J = 6.4 Hz, 1 H), 5.10 (quintet, J = 7.2 Hz, 1 H), 2.33 (s, 3 H), 2.16 (t, J = 8.0 Hz, 2 H), 1.61 (quintet, J = 7.6 Hz, 2 H), 1.46 (d, J = 6.8 Hz, 3 H), 1.33 (sextet, J = 7.2 Hz, 2 H), 0.90 (t, J = 7.2 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR (Figure S8): δ 172.4, 140.7, 137.3, 129.6, 126.4, 48.6, 37.0, 28.1, 22.7, 22.0, 21.4, 14.1. IR (neat, cm^{-1}): 3289 (m), 1638 (s). HRMS Calcd (found) for $\text{C}_{14}\text{H}_{21}\text{NO}$ (M^+): 219.1623 (219.1628).

[N-1-(p-Methoxyphenyl)ethyl]valeramide (Table 2, entry 17). Reaction time = 48 h. Viscous colorless oil. TLC (hexanes–EtOAc = 1:1): R_f = 0.42. ^1H NMR (Figure S9): δ 7.23 (d, J = 8.8 Hz, 2 H), 6.85 (d, J = 8.4 Hz, 2 H), 5.80 (br d, J = 6.8 Hz, 1 H), 5.08 (quintet, J = 7.2 Hz, 1 H), 3.78 (s, 3 H), 2.14 (t, J = 7.6 Hz, 2 H), 1.59 (quintet, J = 7.6 Hz, 2 H), 1.45 (d, J = 7.2 Hz, 3 H), 1.31 (sextet, J = 7.2 Hz, 2 H), 0.89 (t, J = 7.2 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR (Figure S10): δ 172.4, 159.0, 135.8, 127.7, 114.2, 55.6, 48.2, 36.9, 28.1, 22.7, 21.9, 14.1. IR (neat, cm^{-1}): 3284 (m), 1644 (s). HRMS Calcd (found) for $\text{C}_{14}\text{H}_{22}\text{NO}_2$ (MH^+): 236.1651 (236.1645).

N-(1-p-Tolylethyl)-p-toluenesulfonamide (Table 2, entry 18).⁹ Reaction time = 24 h. ^1H NMR: δ 7.63 (d, J = 8.0 Hz, 2 H), 7.18 (d, J = 8.0 Hz, 2 H), 6.99 (s, 4 H), 4.98 (d, J = 6.8 Hz, 1 H), 4.41 (quintet, J = 6.8 Hz, 1 H), 2.39 (s, 3 H), 2.28 (s, 3 H), 1.40 (d, J = 6.8 Hz, 3 H). $^{13}\text{C}\{\text{H}\}$ NMR: δ 143.4, 139.4, 138.0, 137.5, 129.7, 129.5, 127.4, 126.4, 53.7, 23.8, 21.8, 21.3.

Figure S1. ^1H NMR spectrum of methyl *N*-(1-*p*-tolylethyl)terephthalamate.

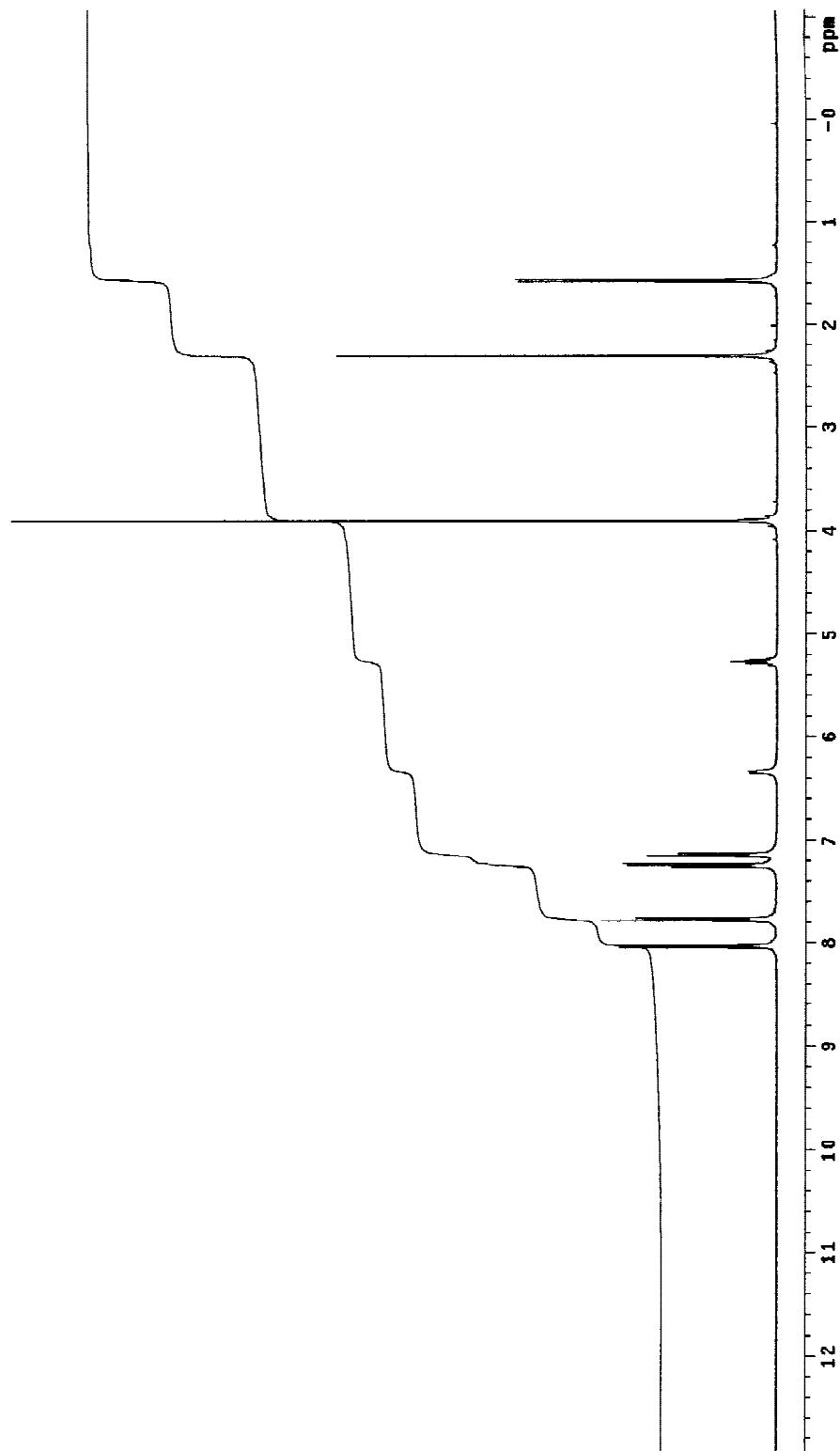


Figure S2. $^{13}\text{C}\{\text{H}\}$ NMR spectrum of methyl *N*-(1-*p*-tolylethyl)terephthalamate.

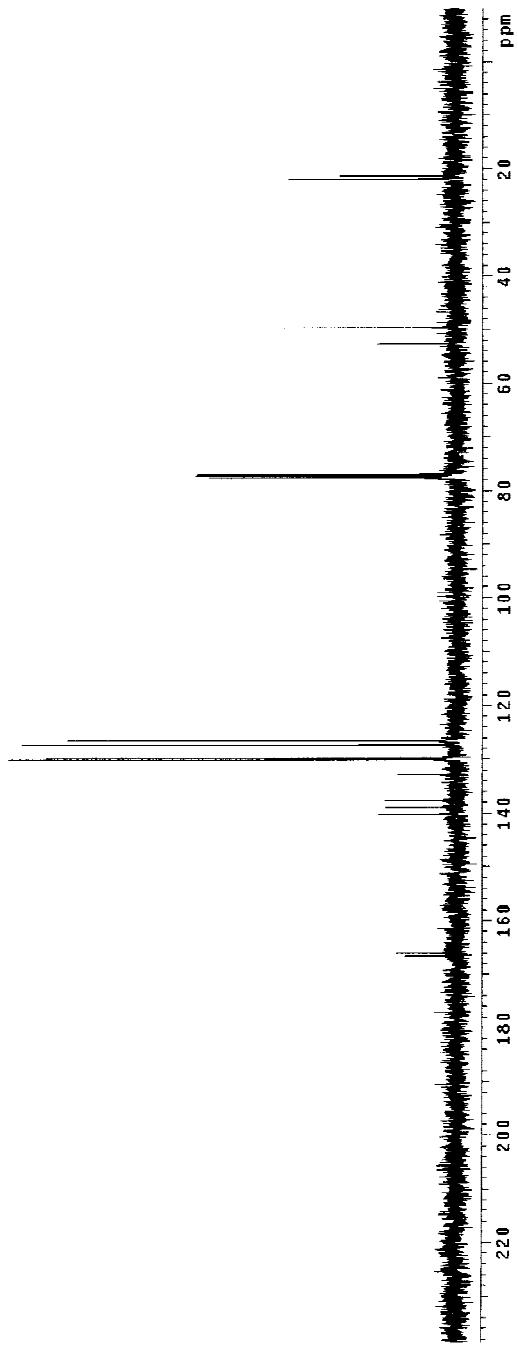


Figure S3. ^1H NMR spectrum of *N*-[1-(*p*-tolyl)ethyl]-benzo[1,3]dioxole-5-carboxamide.

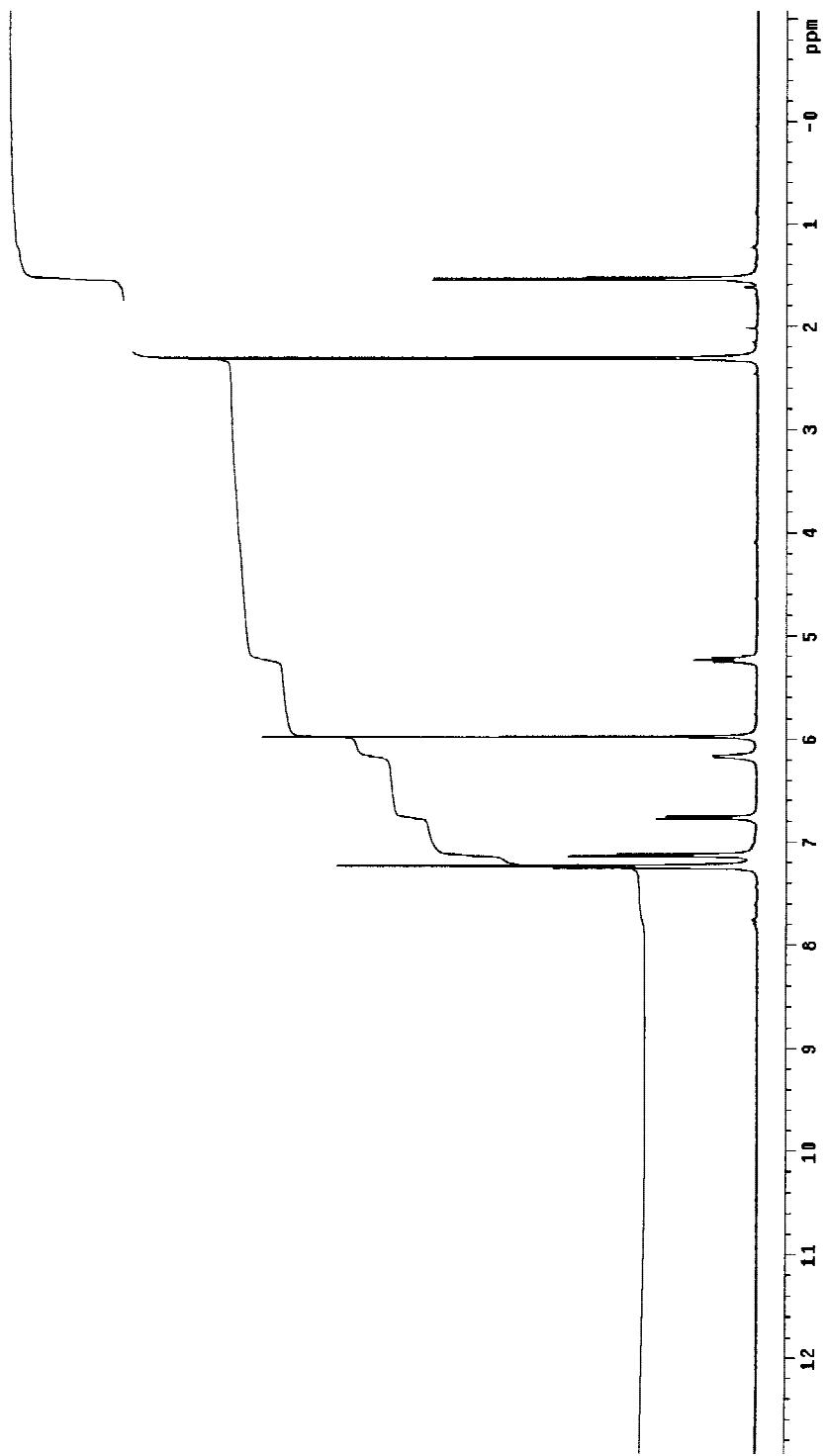


Figure S4. $^{13}\text{C}\{\text{H}\}$ NMR spectrum of *N*-[1-(*p*-tolyl)ethyl]-benzo[1,3]dioxole-5-carboxamide.

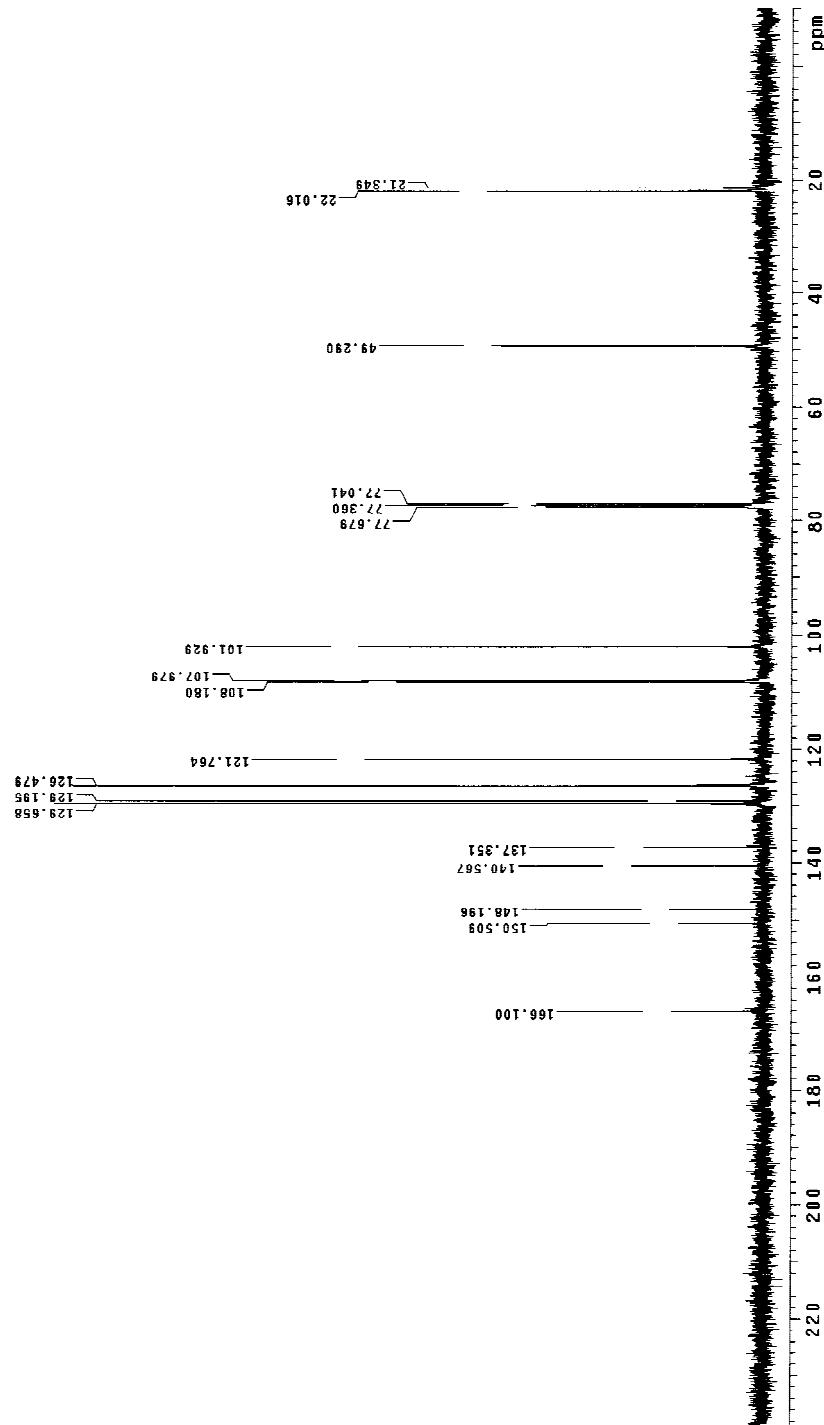


Figure S5. ^1H NMR spectrum of *N*-[1-(*o*-tolyl)ethyl]benzamide.

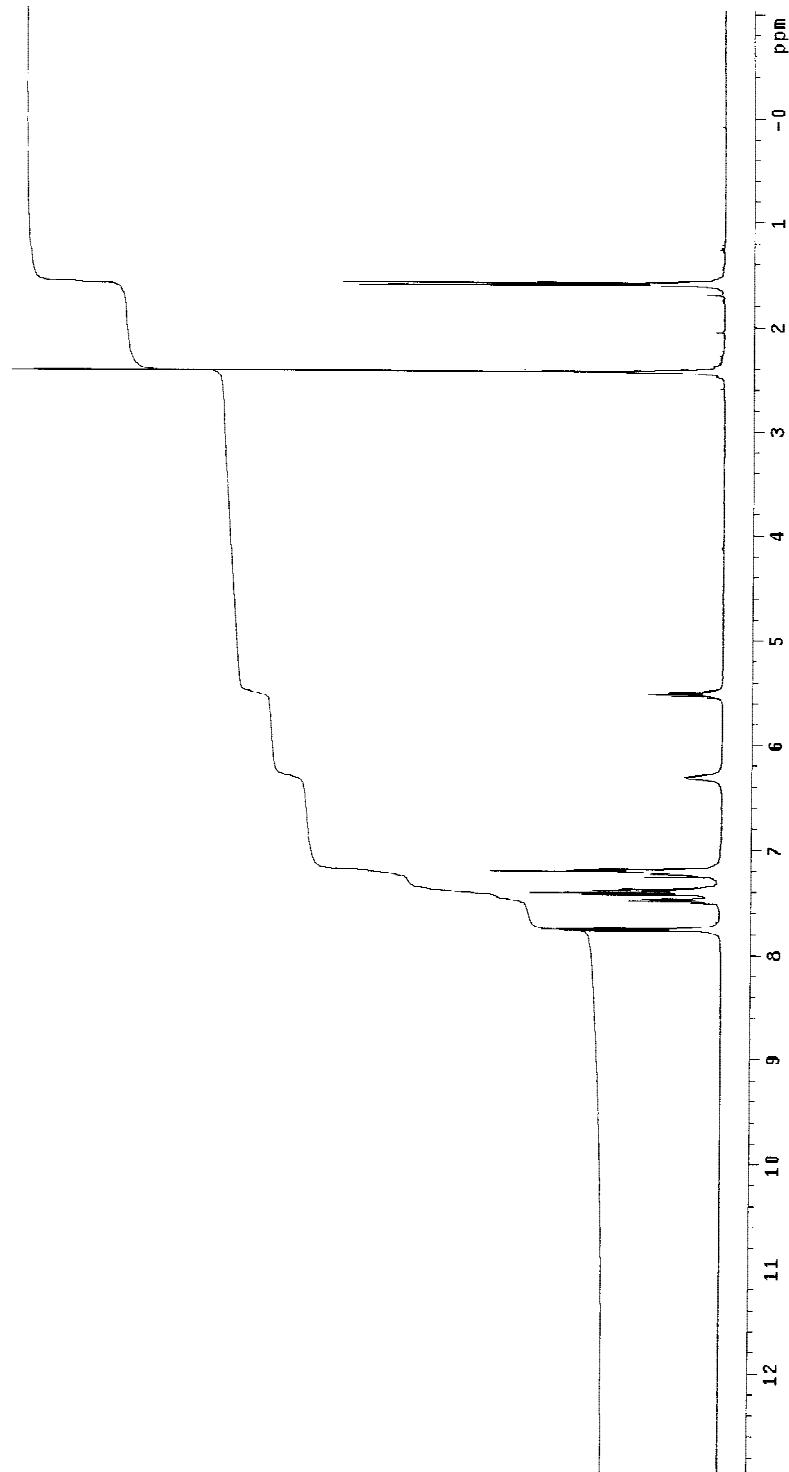


Figure S6. $^{13}\text{C}\{\text{H}\}$ NMR spectrum of *N*-[1-(*o*-tolyl)ethyl]benzamide.

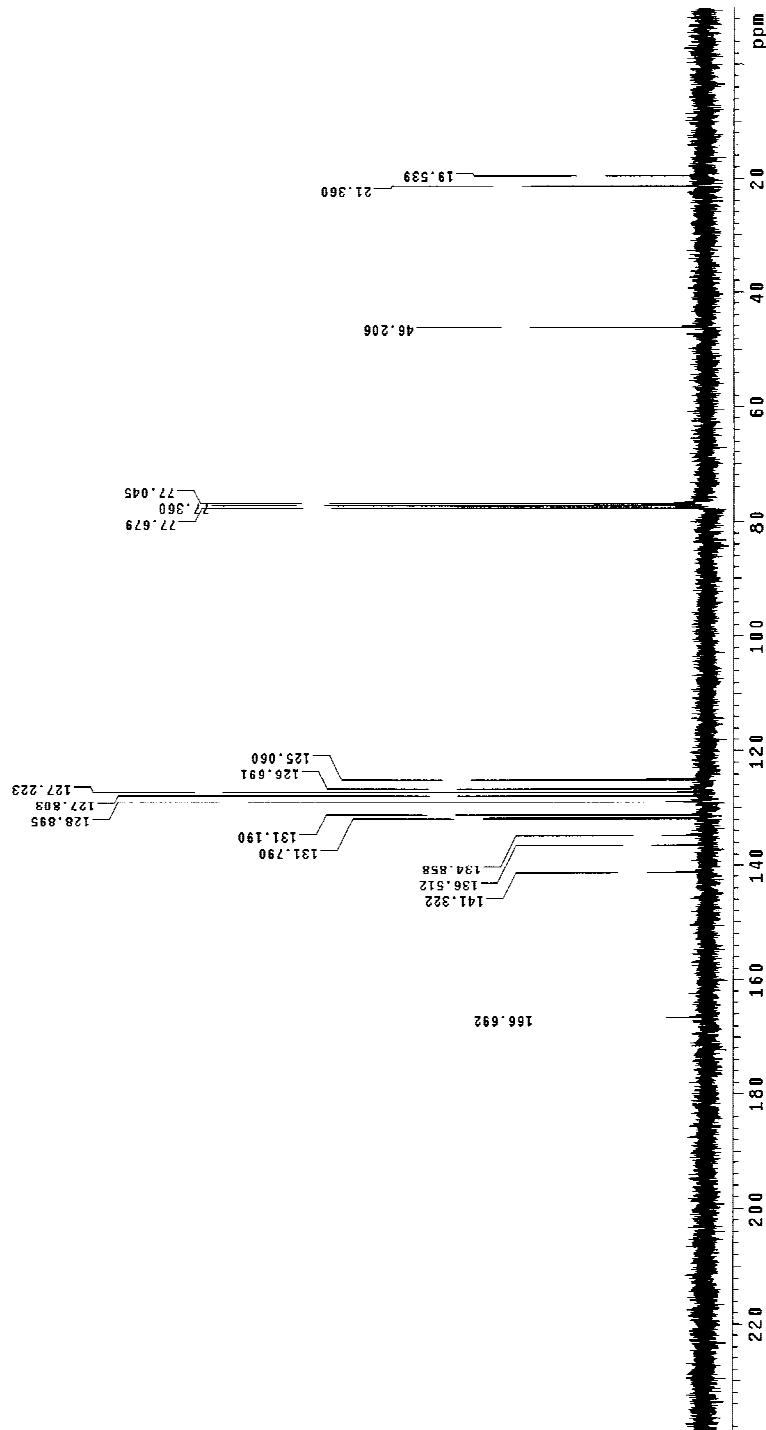


Figure S7. ^1H NMR spectrum of (*N*-1-*p*-tolylethyl)valeramide.

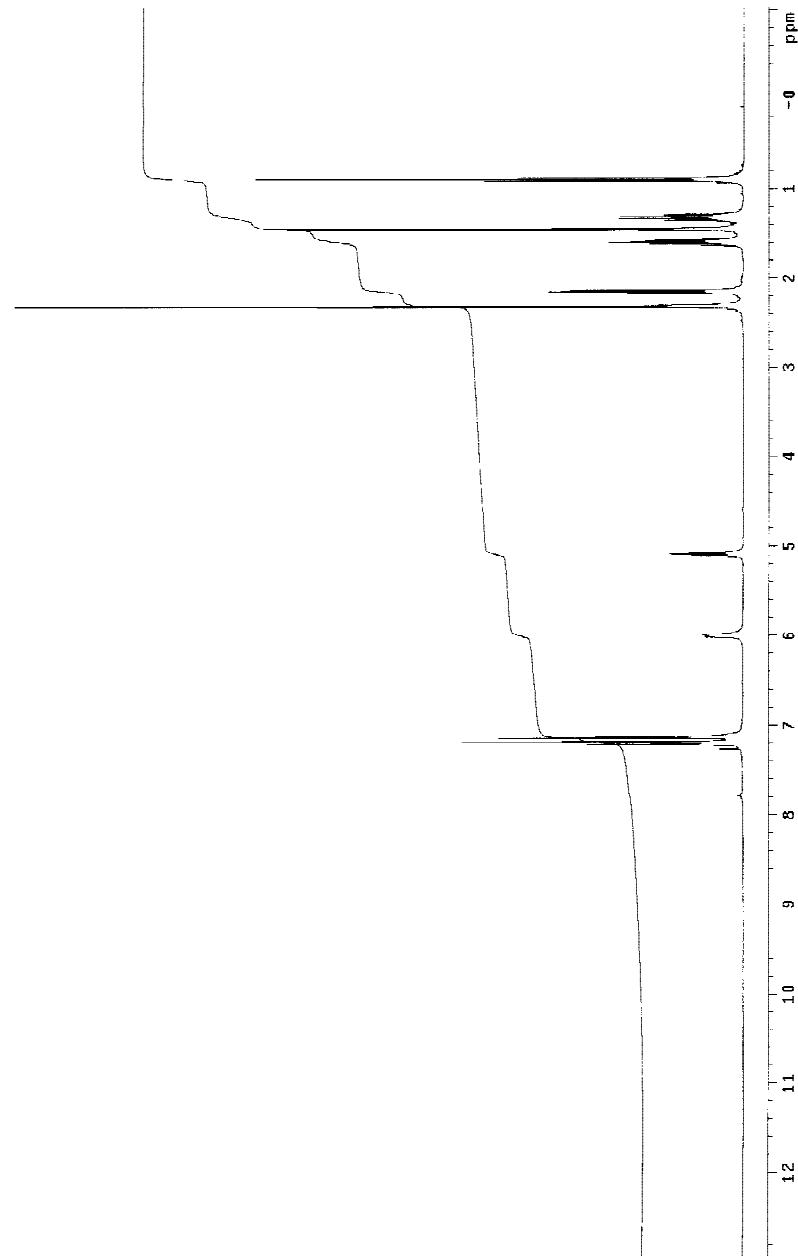


Figure S8. ^{13}C { ^1H } NMR spectrum of (*N*-1-*p*-tolylethyl)valeramide.

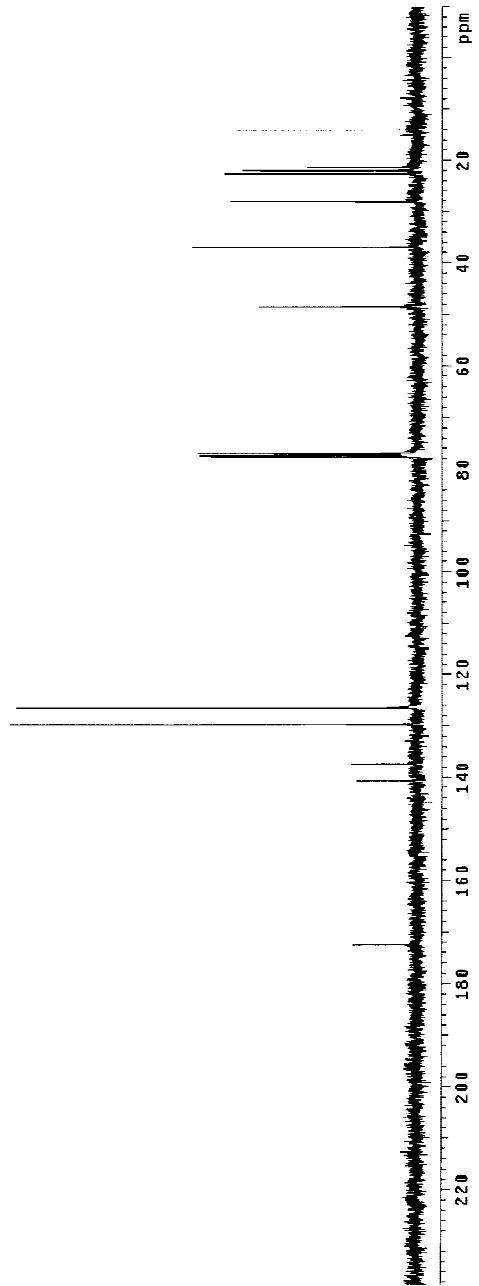


Figure S9. ^1H NMR spectrum of [N-1-(*p*-methoxyphenyl)ethyl]valeramide.

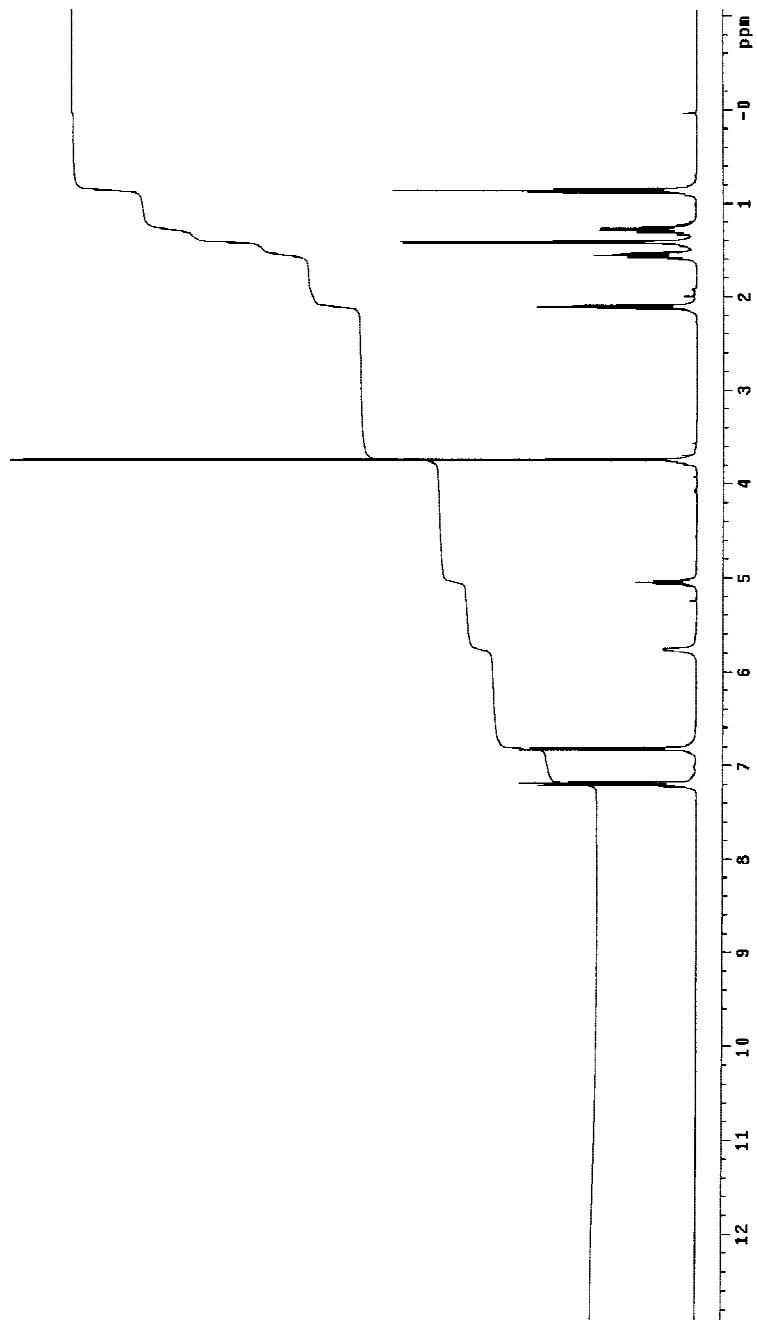
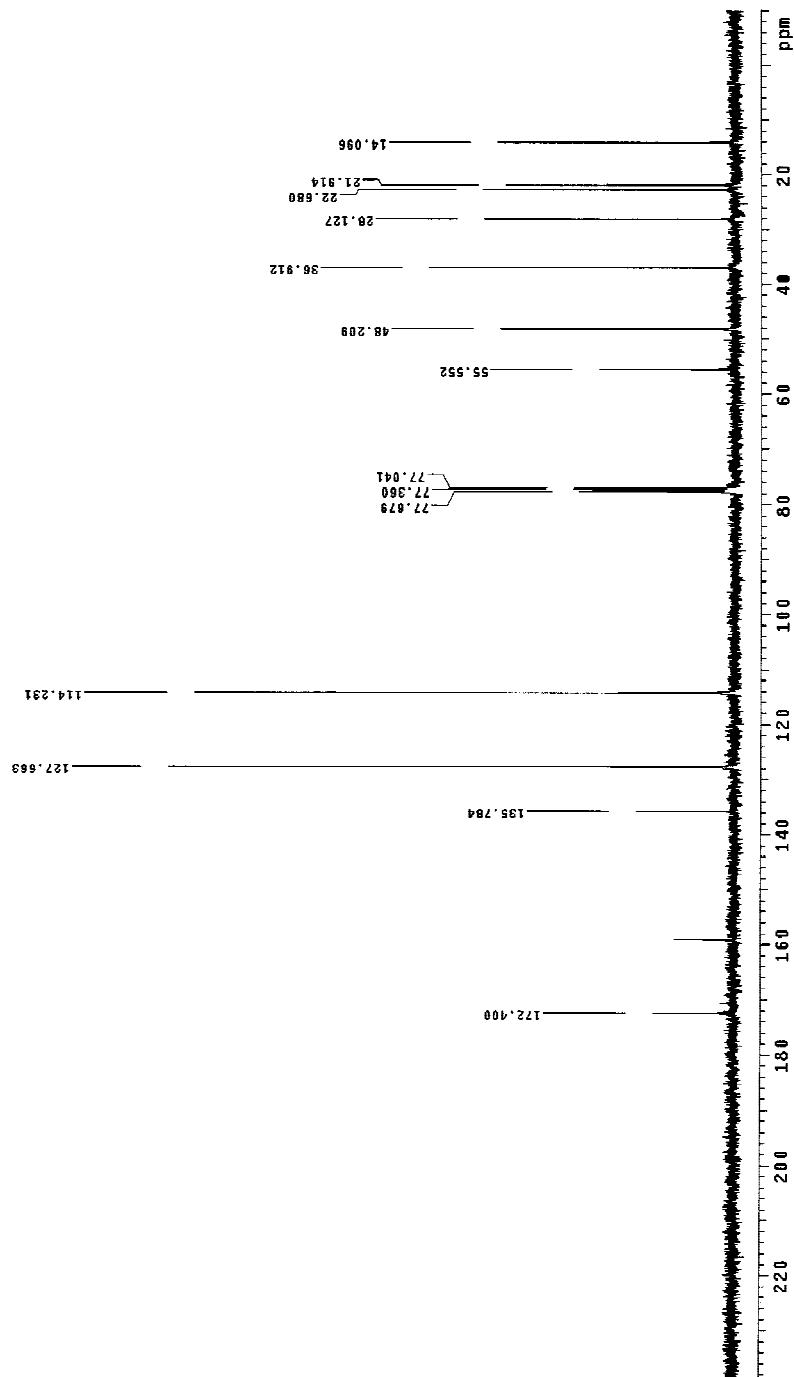


Figure S10. ^{13}C { ^1H } NMR spectrum of [N-1-(*p*-methoxyphenyl)ethyl]valeramide.



References

- 1) Plummer, B. F.; Menendez, M.; Songster, M. *J. Org. Chem.* **1989**, *54*, 718.
- 2) (a) Potapov, V. M.; Dem'yanovich, V. M.; Lazutina, L. I.; Terent'ev, A. P. *Zhurnal Obshchey Khimii* **1962**, *32*, 1187. (b) Vaughan, W. R.; Carlson, R. D. *J. Am. Chem. Soc.* **1962**, *84*, 769.
- 3) Tsuchimoto, T.; Kamiyama, S.; Negoro, R.; Shirakawa, E.; Kawakami, Y. *Chem. Commun.* **2003**, 852.
- 4) Noji, M.; Ohno, T.; Fuji, K.; Futaba, N.; Tajima, H.; Ishii, K. *J. Org. Chem.* **2003**, *68*, 9340.
- 5) (a) Smidt, H.; Fischer, A.; Fischer, P.; Schmid, R. D. *Biotechnology Techniques* **1996**, *10*, 335. (b) Potapov, V. M.; Dem'yanovich, V. M.; Terent'ev, A. P. *Zhurnal Obshchey Khimii* **1965**, *35*, 1340.
- 6) Terent'ev, A. P.; Potapov, V. M. *Zhurnal Obshchey Khimii* **1957**, *27*, 1092.
- 7) Moglioni, A. G.; Tombari, D. G.; Iglesias, G. Y. M. *J. Chem. Soc., Perkin Trans. 1* **1998**, *20*, 3459.
- 8) Timokhin, V. I.; Anastasi, N. R.; Stahl, S. S. *J. Am. Chem. Soc.* **2003**, *125*, 12996.
- 9) Au, S.-M.; Huang, J.-S.; Che, C.-M.; Yu, W.-Y. *J. Org. Chem.* **2000**, *65*, 7858.