

## Supporting Information for

*N*-Amidation by the Copper-Mediated Cross-coupling of Organostannanes and Boronic Acids with *O*-Acetyl

Hydroxamic Acids

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Experimental procedures and compound characterizations S-2-S-20

Photocopies of spectra S-21-S-74

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## General Methods

All reactions were performed under an atmosphere of dry argon in oven-dried glassware. THF, DMA, DMF, and toluene were dried over 4Å molecular sieves and titrated for water level prior to use with a Fisher Coulomatic K-F titrater. hexanes, ethyl acetate (EtOAc), and diethyl ether (Et<sub>2</sub>O) were obtained from Aldrich and used as purchased. 'Brine' refers to a saturated aqueous solution of NaCl. Purification by flash chromatography was performed using Whatman 60Å 230-400 mesh SiO<sub>2</sub> with compressed air as a source of positive pressure. Purification by preparative plate chromatography was performed on EM Science Kieselgel 0.5 mm or 1 mm 60F<sub>254</sub> plates. Analytical thin-layer chromatography (TLC) was carried out using Merck Kieselgel 60F<sub>254</sub> plates with visualization by UV or phosphomolybdic acid.

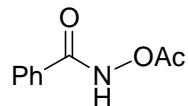
<sup>1</sup>H NMR spectra were recorded on a INOVA 400 MHz NMR spectrometer at room temperature in CDCl<sub>3</sub> and were internally referenced to CDCl<sub>3</sub> (7.27 ppm); <sup>13</sup>C NMR spectra were recorded on a 100 MHz NMR spectrometer at room temperature in CDCl<sub>3</sub> and were internally referenced to CDCl<sub>3</sub> (77.23 ppm). Data are reported in the following order: chemical shifts are given (δ); multiplicities are indicated (br (broadened), s (singlet), d (doublet), t (triplet), q (quartet), hex (hextet), hept (heptet), m (multiplet), exch (exchangeable), app (apparent); coupling constants, *J*, are reported (Hz); integration is provided. Infrared spectra were recorded ASI ReactIR 1000FT-IR spectrometer with a silicone probe. Peaks are reported (cm<sup>-1</sup>) with the following relative intensities: s (strong, 67-100%), m (medium, 40-67%), w (weak, 20-40%) and br (broad). GC-MS spectra were recorded on a Shimadzu Gas Chromatograph GC-17A, Mass Spectrometer QP-5000. GC/MS analysis was carried out on a bonded 5% diphenylsiloxane capillary column (30 m, 0.25 mm id, 0.25 μm df). Uncalibrated melting points were taken on a Thomas-Hoover melting point apparatus in open capillary tubes

## Starting Materials

*N*-Benzoyl-*N*-phenylhydroxylamine, benzoyl chloride, benzohydroxamic acid, acetic anhydride, acetyl chloride, 2-thiophene carbonyl chloride, *t*-butylcarbonyl chloride, *p*-toluoyl chloride, *O*-methylhydroxylamine hydrochloride, *O*-phenylhydroxylamine hydrochloride, phenylacetyl chloride, *m*-anisoyl chloride, *p*-nitrobenzoyl chloride, hydroxylamine hydrochloride, CuOAc, CuCl, CuI, CuCN, *p*-tolylboronic acid, phenylboronic acid, 2-phenyl-1,3,2-dioxaborinane, TiCl<sub>4</sub>, Sc(OTf)<sub>3</sub>, CuPF<sub>6</sub>(CH<sub>3</sub>CN)<sub>4</sub> and boron trifluoride etherate were purchased from Aldrich. All boronic acids except *p*-tolylboronic acid and phenylboronic acid were obtained from Frontier Scientific, Inc. All organostannanes were obtained from Synthonix. 1-Naphthohydroxamic acid and 2-picolinoyl

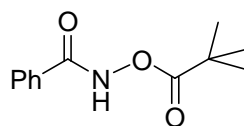
chloride hydrochloride were obtained from TCI America Inc. Copper(I) thiophene-2-carboxylate (CuTC)<sup>1</sup> and copper(I) diphenylphosphinate (CuDPP)<sup>2</sup> were prepared following the literature procedures.

### ***O*-Acetyl benzohydroxamic acid<sup>3</sup>**



Benzohydroxamic acid (1.385 g, 10 mmol), CH<sub>2</sub>Cl<sub>2</sub> (30 mL) and aqueous NaOH (2 M, 5.5 mL) were charged in a round bottomed flask. Ac<sub>2</sub>O (1.12 g, 11 mmol) was added via syringe. After stirring at room temperature for 2 h, the CH<sub>2</sub>Cl<sub>2</sub> layer was separated. The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (10 mL) and the combined CH<sub>2</sub>Cl<sub>2</sub> layers were washed with brine and dried over MgSO<sub>4</sub>. Evaporation of the solvent gave the product as a white solid (1.59 g, 89%). Mp 124-125 °C (ethyl ether, lit. {126.5 °C (hexanes/ether)}<sup>3</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.47 (s, 1 H), 7.84 (d, *J* = 7.2 Hz, 2 H), 7.59 (t, *J* = 7.6 Hz, 1 H), 7.48 (t, *J* = 7.6 Hz, 2 H), 2.31 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3147 (m), 2961 (m), 1793 (s), 1649 (s).

### ***N*-(2,2-Dimethyl-1-oxopropoxy)benzamide<sup>4</sup>**



Benzohydroxamic acid (0.693 g, 5 mmol) was dissolved in THF (30 mL) in a round bottomed flask. *t*-Butylcarbonyl chloride (0.603 g, 5 mmol) was added via syringe, followed by Et<sub>3</sub>N (0.607 g, 6 mmol). A white precipitate was observed. The suspension was stirred at room temperature for 2 h. Ethyl ether (20 mL) was added and the reaction was washed with water twice and then brine. After drying the organic layer over MgSO<sub>4</sub>, evaporation of the solvent gave the product as an off-white solid. Recrystallization from hexanes/ether gave the product as a white solid in 72% yield (0.796 g). Mp 169-170 °C (lit. {170-171.5 °C}<sup>4</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.51 (s, 1 H), 7.80 (d, *J* = 7.6 Hz, 2 H), 7.54 (t, *J* = 7.6 Hz, 1 H), 7.43 (t, *J* = 7.6 Hz, 2 H), 1.35 (s, 9 H). IR (neat, cm<sup>-1</sup>): 3204 (br), 2980 (s), 1783 (s), 1652 (s), 1517 (m), 1482 (m).

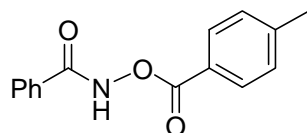
<sup>1</sup> Allred, G.; Liebeskind, L. S. *J. Am. Chem. Soc.* **1996**, *118*, 2748-2749.

<sup>2</sup> Wittenberg, R.; Srogl, J.; Egi, M.; Liebeskind, L. S. *Org. Lett.* **2003**, *5*, 3033-3035.

<sup>3</sup> Just, G.; Dahl, K. *Tetrahedron* **1968**, *24*, 5251-5269.

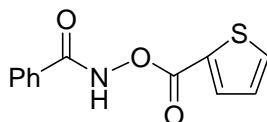
<sup>4</sup> Miller, M. J.; DeBons, F. E.; Loudon, G. M. *J. Org. Chem.* **1977**, *42*, 1750-1761.

#### ***N*-(4-Methylbenzoyloxy)benzamide<sup>5</sup>**



Benzohydroxamic acid (0.693 g, 5 mmol) was dissolved in 1,4-dioxane (40 mL) in a round bottomed flask. Pyridine (0.400 g, 5 mmol) was added via syringe. After addition of *p*-toluoyl chloride (0.850 g, 5.5 mmol), a white precipitate was observed. The suspension was stirred at room temperature overnight. EtOAc (20 mL) was added and the reaction was washed with 1 M HCl, water and then brine. After drying the organic layer over MgSO<sub>4</sub>, evaporation of the solvent gave a white solid. Recrystallization from hexanes/EtOAc gave the product as colorless needle-shape crystals in 75% yield (0.956 g). Mp 154-156 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.86 (br, s, 1 H), 8.03 (d, *J* = 10.8 Hz, 2 H), 7.87 (d, *J* = 9.2 Hz, 2 H), 7.57 (t, *J* = 10 Hz, 1 H), 7.46 (t, *J* = 10 Hz, 2 H), 7.28 (d, *J* = 10.8 Hz, 2 H), 2.44 (s, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.7, 165.5, 145.5, 132.9, 131.1, 130.3, 129.7, 129.0, 127.7, 123.9, 22.0. IR (neat, cm<sup>-1</sup>): 3150 (m), 2953 (m), 1764 (s), 1706 (m), 1648 (s), 1583 (m). HRMS (FAB) Calcd for C<sub>15</sub>H<sub>13</sub>O<sub>3</sub>NLi (M+Li<sup>+</sup>): 262.1055. Found: 262.1058.

#### ***N*-(Thiophene-2-carbonyloxy)benzamide<sup>6</sup>**

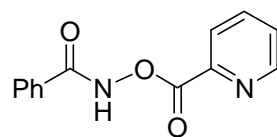


Benzohydroxamic acid (0.693 g, 5 mmol) was dissolved in THF (20 mL) and CH<sub>2</sub>Cl<sub>2</sub> (20 mL) in a round bottomed flask. 2-Thiophene carbonyl chloride (0.733 g, 5 mmol) was added to the solution via syringe, followed by Et<sub>3</sub>N (0.607 g, 6 mmol). A white precipitate was observed. The suspension was stirred at 50 °C for 2 h after which CH<sub>2</sub>Cl<sub>2</sub> (20 mL) was added and the reaction was washed with water twice then brine. After drying the organic layer over MgSO<sub>4</sub>, evaporation of the solvent gave the product as a yellow solid. Recrystallization from hexanes/ether gave the product as light yellow crystals (1.0 g, 81%). Mp 131-132.5 °C (lit. {133-133.5 °C (EtOAc)}<sup>6</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.49 (br, s, 1 H), 8.02 (dd, *J* = 2.4, 1.2 Hz, 1 H), 7.86-7.88 (m, 2 H), 7.72 (dd, *J* = 3.6, 1.2 Hz, 1 H), 7.60 (tt, *J* = 7.2, 2.0 Hz, 1 H), 7.47-7.51 (m, 2 H), 7.19 (dd, *J* = 5.2, 4.0 Hz, 1 H). IR (neat, cm<sup>-1</sup>): 3188 (m), 2953 (m), 1752 (s), 1660 (s).

<sup>5</sup> Alexandrou, N. E.; Nicolaides, D.N. *Tetrahedron Lett.* **1966**, 7, 2497-2499.

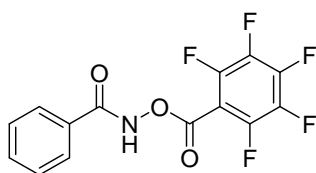
<sup>6</sup> Jones, L. W.; Hurd, C. D. *J. Am. Chem. Soc.* **1921**, 43, 2422-2448.

### ***N*-(2-Pyridinylcarbonyloxy)benzamide<sup>7</sup>**



2-Picolinoyl chloride hydrochloride (1.0 g, 5.2 mmol) was dissolved in THF (20 mL) in a round bottomed flask. Et<sub>3</sub>N (0.526 g, 5.2 mmol) was added to the suspension via syringe, and a white precipitate was observed. Then benzohydroxamic acid (0.693 g, 5 mmol) was dissolved in THF (20 mL) and slowly added, followed by additional Et<sub>3</sub>N (0.526 g, 5.2 mmol). More white precipitate was observed. After stirring at room temperature under argon overnight, the solid was filtered and washed with EtOAc (10 mL). The filtrate was washed with water and then brine. After drying over MgSO<sub>4</sub>, evaporation of the solvent gave a light yellow solid. Recrystallization from Et<sub>2</sub>O/CH<sub>2</sub>Cl<sub>2</sub> gave the product as an off-white solid in 52% yield (0.629 g). Mp 80-81°C. (lit. {155-166 °C }<sup>7</sup>) <sup>1</sup>H NMR (400 MHz, *d*<sub>6</sub>-DMSO) δ 8.82 (d, *J* = 4.8 Hz, 1 H), 8.22 (d, *J* = 8.0 Hz, 1 H), 8.09 (td, *J* = 8.0, 1.2 Hz, 1 H), 7.90 (d, *J* = 8.0 Hz, 2 H), 7.74-7.77 (m, 1 H), 7.64 (t, *J* = 6.8 Hz, 1 H), 7.56 (t, *J* = 7.6 Hz, 2 H), -0.27 (br, s, 1 H). <sup>13</sup>C NMR (100 MHz, *d*<sup>6</sup>-DMSO) δ 164.9, 163.3, 150.3, 145.4, 138.0, 132.5, 130.9, 128.8, 128.4, 127.5, 125.7. IR (neat, cm<sup>-1</sup>): 3157 (m), 2930 (m), 1776 (s), 1664 (s), 1282 (s), 1239 (s), 1061 (s), 695 (s). HRMS (FAB) Calcd for C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N<sub>2</sub> (M+H<sup>+</sup>): 243.0770. Found: 243.0771.

### ***N*-(penta-Fluorobenzoyloxy)benzamide**

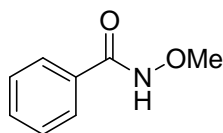


Benzohydroxamic acid (1.38 g, 10 mmol) and Et<sub>3</sub>N (1.21 g, 12 mmol) were dissolved in CH<sub>2</sub>Cl<sub>2</sub> (30 mL) in a round-bottom flask and cooled to -78 °C under protection of argon. Pentafluorobenzoyl chloride (2.53 g, 11 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was added to the mixture via syringe. The suspension was stirred for 2 h and slowly warmed to room temperature. The organic phase was washed with brine and dried over MgSO<sub>4</sub>. Evaporation of the solvent gave the crude product. Recrystallization from CH<sub>2</sub>Cl<sub>2</sub>/hexanes gave a white solid (2.81 g, 85%) Mp 142-143 °C (ethyl ether). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.56 (s, 1H), 7.89 (d, *J* = 7.2 Hz, 2 H), 7.64 (t, *J* = 7.2

<sup>7</sup> Misra, B. N.; Sharma, R. P.; Diksha. *Indian J. Chem. Sec. B*. **1986**, 25, 1182-1183.

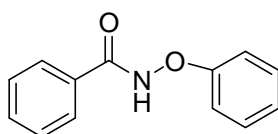
Hz, 1 H), 7.53 (t,  $J = 8.0$  Hz, 2 H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 166.7, 158.2, 133.4, 130.1, 129.1, 127.8. IR (neat,  $\text{cm}^{-1}$ ): 3244 (m), 1783 (s), 1654 (s), 1497 (s). HRMS (FAB) Calcd for  $\text{C}_{14}\text{H}_7\text{O}_3\text{NF}_5(\text{M}+\text{H}^+)$ : 332.0340. Found. 332.0336.

#### ***O*-Methylbenzohydroxamic acid<sup>8</sup>**



*O*-Methylhydroxylamine hydrochloride (0.852 g, 10 mmol) and  $\text{K}_2\text{CO}_3$  (2.792 g, 20 mmol) were dissolved in water (20 mL) and EtOAc (20 mL) in a round bottomed flask. The solution was cooled to 0 °C in an ice bath. Benzoyl chloride (1.406 g, 10 mmol) was then added via syringe. After stirring at room temperature for 2 h, the aqueous layer was removed. The organic layer was washed with water then brine. After drying over  $\text{MgSO}_4$ , evaporation of the solvent gave a sticky residue. Recrystallization from hexanes/ $\text{Et}_2\text{O}$  gave the product as a white solid in 70% yield (1.057 g). Mp 59.5-61.0 °C (lit. {61-62 °C (hexanes/ $\text{Et}_2\text{O}$ )}<sup>8</sup>).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  10.2 (br, s, 1 H), 7.76 (d,  $J = 7.2$  Hz, 2 H), 7.54 (t,  $J = 7.6$  Hz, 1 H), 7.45 (t,  $J = 7.6$  Hz, 2 H), 3.88 (s, 3 H). IR (neat,  $\text{cm}^{-1}$ ): 3196 (s), 2980 (m), 1648 (s), 1579 (m), 1517 (m).

#### ***O*-Phenylbenzohydroxamic acid<sup>9</sup>**



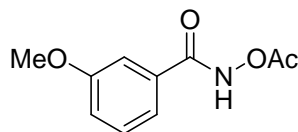
*O*-Phenylhydroxylamine hydrochloride (0.750 g, 5 mmol) and  $\text{Na}_2\text{CO}_3$  (1.06 g, 10 mmol) were dissolved in water (20 mL) and EtOAc (20 mL) in a round bottomed flask. Benzoyl chloride (0.703 g, 5 mmol) was added via syringe. After stirring at room temperature for 2 h, the aqueous layer was removed. The organic layer was washed with water then brine. After drying over  $\text{MgSO}_4$ , evaporation of the solvent gave a yellow solid. Recrystallization from hexanes/ $\text{Et}_2\text{O}$  gave the product as a white solid in 73% yield (0.777 g). Mp 135-136 °C (lit. {137-139 °C ( $\text{EtOAc}$ )}<sup>9</sup>).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.89 (br, s, 1 H), 7.78 (d,  $J = 7.6$  Hz, 2 H), 7.50 (t,  $J = 7.6$  Hz, 1 H), 7.36 (t,  $J = 7.6$  Hz, 2 H), 7.26 (t,  $J = 7.6$  Hz, 2 H), 7.00-7.07 (m, 3 H). IR (neat,  $\text{cm}^{-1}$ ): 3173 (s), 2953 (m), 1656 (s),

<sup>8</sup> Johnson, J. E.; Nalley, E. A.; Kunz, Y. K.; Springfield, J. R. *J. Org. Chem.* **1976**, *41*, 252-259.

<sup>9</sup> Baughman, R. G.; Fountain, K. R.; Fountain, D. P.; Tappmeyer, A. M. *J. Org. Chem.* **1989**, *54*, 5819-5821.

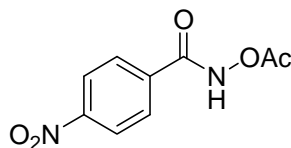
1590 (s), 1513 (m), 1486 (s).

***O*-Acetyl-3-methoxybenzohydroxamic acid**



Hydroxylamine hydrochloride (2.78 g, 40 mmol) and NaOH pellets (2 g, 50 mmol) were dissolved in water (20 mL) and THF (20 mL). *m*-Methoxybenzoyl chloride (1.723 g, 10 mmol) was then added via syringe. After stirring the mixture at room temperature overnight, 2 M HCl was added to acidify the solution to pH=1. EtOAc (10 mL) was added to the organic layer that was washed with brine, then dried over MgSO<sub>4</sub>. Evaporation of the solvent gave 0.939 g of a light orange solid, which was then dissolved in THF (30 mL). After purging with argon, AcCl (0.453 g, 5.8 mmol) and Et<sub>3</sub>N (0.587 g, 5.8 mmol) were added subsequently via syringe. The suspension was stirred at room temperature overnight. After filtration of the solid, EtOAc (20 mL) was added to the filtrate that was washed with water, then brine. Drying over MgSO<sub>4</sub> and evaporation of the solvent gave a white solid. Recrystallization from hexanes/EtOAc gave the product as white crystals in 40% yield (0.836 g). Mp 119-120 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.88 (br, s, 1 H), 7.29-7.35 (m, 3 H), 7.04-7.07 (m, 1 H), 3.80 (s, 3 H), 2.23 (s, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.3, 166.4, 160.0, 132.1, 130.0, 119.6, 119.3, 112.5, 55.6, 18.5. IR (neat, cm<sup>-1</sup>): 3181 (m), 2937 (m), 1795 (s), 1660 (s), 1583 (s), 1177 (s). HRMS (FAB) Calcd for C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>NLi (M+Li<sup>+</sup>): 216.0848. Found: 216.0848.

***O*-Acetyl-4-nitrobenzohydroxamic acid<sup>10</sup>**

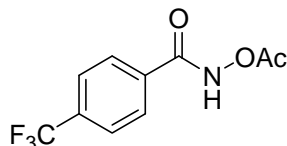


Hydroxylamine hydrochloride (2.78 g, 40 mmol) and NaOH pellets (2 g, 50 mmol) were dissolved in water (20 mL) and THF (20 mL). *p*-Nitrobenzoyl chloride (1.874 g, 10 mmol) was dissolved in THF (30 mL) and added via syringe. After stirring the mixture at room temperature overnight, 2 M HCl was added to acidify the solution to pH=1. EtOAc (10 mL) was added to the organic layer that was washed with brine, then dried over MgSO<sub>4</sub>.

<sup>10</sup> (a) Dessolin, M.; Laloi-Diard, M.; Vilkas, M. *Bull. Soc. Chim. Fr.* **1970**, 7, 2573-2580. (b) Exner, O.; Simon, W. *Collect. Czech. Chem. Commun.* **1965**, 30, 4078-4094.

Evaporation of the solvent gave 1.290 g of an off-white solid, which was then dissolved in THF (30 mL). AcCl (0.569 g, 7.3 mmol) and Et<sub>3</sub>N (0.739 g, 7.3 mmol) were added subsequently via syringe. The suspension was stirred at room temperature overnight. After filtration of the solid, EtOAc (10 mL) was added to the filtrate which was washed with water, then brine. Drying over MgSO<sub>4</sub> and evaporation of the solvent gave a white solid. Recrystallization from Et<sub>2</sub>O/CH<sub>2</sub>Cl<sub>2</sub> gave the product as needle-shape yellow crystals in 51% yield (1.14 g). Mp 181-183 °C. <sup>1</sup>H NMR (400 MHz, *d*6-acetone) δ 11.6 (br, s, 1 H), 8.38 (d, *J* = 8.0 Hz, 2 H), 8.13 (d, *J* = 8.0 Hz, 2 H), 2.24 (s, 3 H). <sup>13</sup>C NMR (100 MHz, *d*6-DMSO) δ 168.5, 162.7, 149.6, 136.6, 129.0, 123.9, 18.1. IR (neat, cm<sup>-1</sup>): 3150 (s), 2964 (m), 1799 (s), 1671 (s), 1602 (m), 1571 (m). HRMS (FAB) Calcd for C<sub>9</sub>H<sub>8</sub>O<sub>5</sub>N<sub>2</sub>Li (M+Li<sup>+</sup>): 231.0593. Found: 231.0594.

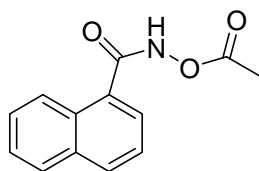
#### ***O*-Acetyl-(4-trifluoromethyl)benzohydroxamic acid**



Hydroxylamine hydrochloride (2.78 g, 40 mmol) and NaOH pellets (2 g, 50 mmol) were dissolved in water (20 mL) and THF (20 mL). *p*-(Trifluoromethyl)benzoyl chloride (2.150 g, 10 mmol) was added via syringe. After stirring the mixture at room temperature overnight, 2 M HCl was added to acidify the solution to pH=1. EtOAc (10 mL) was added to the organic layer that was washed with brine, then dried over MgSO<sub>4</sub>. Evaporation of the solvent gave 2.0 g of an off-white solid, which was then dissolved in THF (30 mL). AcCl (0.765 g, 9.8 mmol) and Et<sub>3</sub>N (0.992 g, 9.8 mmol) were added subsequently via syringe. The suspension was stirred at room temperature overnight. After filtration of the solid, EtOAc (10 mL) was added to the filtrate that was washed with water, then brine. Drying over MgSO<sub>4</sub> and evaporation of the solvent gave a white solid. Recrystallization from EtOAc/CH<sub>2</sub>Cl<sub>2</sub> gave the product as needle-shaped white crystals in 55% yield (1.359 g). Mp 140-141 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.50 (br, s, 1 H), 7.93 (d, *J* = 11.2 Hz, 2 H), 7.73 (d, *J* = 11.2 Hz, 2 H), 2.31 (s, 3 H). <sup>13</sup>C NMR (100 MHz, *d*6-DMSO) δ 168.5, 163.3, 134.9, 132.0 (q, *J* = 31.9 Hz), 128.4, 125.7 (q, *J* = 3.8 Hz), 123.8 (q, *J* = 270.8 Hz), 18.1. IR (neat, cm<sup>-1</sup>): 3142 (m), 2961 (m), 1799 (s), 1710 (s), 1656 (m). HRMS (FAB) Calcd for C<sub>10</sub>H<sub>8</sub>O<sub>3</sub>NF<sub>3</sub>Li (M+Li<sup>+</sup>): 254.0616. Found: 254.0616.

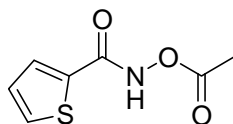
#### ***O*-Acetyl 1-naphthohydroxamic acid**





1-Naphthohydroxamic acid (0.936 g, 5 mmol) was dissolved in THF (60 mL) in a round bottomed flask. AcCl (0.408 g, 5.2 mmol) was added to the solution via syringe, followed by Et<sub>3</sub>N (0.526 g, 5.2 mmol). The suspension was stirred at room temperature overnight. The precipitate was filtered and washed with EtOAc (20 mL). The filtrate was washed with water and then brine. After drying over MgSO<sub>4</sub>, evaporation of the solvent gave a light yellow solid. Recrystallization from Et<sub>2</sub>O/CH<sub>2</sub>Cl<sub>2</sub> gave the product as an off-white solid in 68% yield (0.779 g). Mp 105-106 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.17 (br, s, 1 H), 8.35 (d, *J* = 8.4 Hz, 1 H), 8.00 (d, *J* = 8.8 Hz, 1 H), 7.89 (d, *J* = 8.0 Hz, 1 H), 7.77 (dd, *J* = 6.8, 1.2 Hz, 1 H), 7.54-7.82 (m, 2 H), 7.49 (t, *J* = 7.6 Hz, 1 H), 2.34 (s, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.0, 167.5, 133.6, 131.9, 130.4, 129.3, 128.4, 127.6, 126.8, 126.4, 125.2, 124.6, 18.3. IR (neat, cm<sup>-1</sup>): 3150 (s), 2953 (m), 1791 (s), 1660 (s), 1513 (m). HRMS (FAB) Calcd for C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>NLi (M+Li<sup>+</sup>): 236.0899. Found: 236.0910.

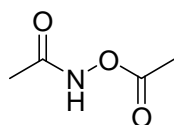
#### ***N*-Acetoxy-2-thiophenecarboxamide**



Hydroxylamine hydrochloride (2.78 g, 40 mmol) and NaOH pellets (2 g, 50 mmol) were dissolved in water (20 mL) and THF (20 mL). 2-Thiophenecarbonyl chloride (1.46 g, 10 mmol) was then added via syringe. After stirring the mixture at room temperature overnight, 2 M HCl was added to acidify the solution to pH=1. EtOAc (10 mL) was added to the organic layer that was washed with brine, then dried over MgSO<sub>4</sub>. Evaporation of the solvent gave a yellow solid which was then dissolved in THF (30 mL). After purging with argon, AcCl (0.765 g, 9.8 mmol) and Et<sub>3</sub>N (0.992 g, 9.8 mmol) were added subsequently via syringe. The suspension was stirred at room temperature overnight. After filtration of the solid, EtOAc (10 mL) was added to the filtrate that was washed with water, then brine. Drying over MgSO<sub>4</sub> and evaporation of the solvent gave a yellow solid. Recrystallization from hexanes/EtOAc gave the product as white crystals in 75% yield (1.39 g). Mp 93-94 °C (ethyl ether). <sup>1</sup>H NMR (400

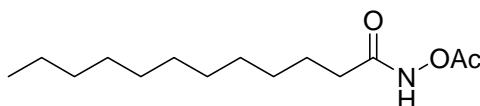
MHz, CDCl<sub>3</sub>)  $\delta$  9.37 (s, 1 H), 7.69 (m, 1 H), 7.61 (d,  $J$  = 5.6 Hz, 1 H), 7.15 (m, 1 H), 2.30 (s, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) 169.4, 161.7, 133.7, 132.1, 130.7, 128.1, 18.5. IR (neat, cm<sup>-1</sup>): 3102 (w), 1782 (s), 1643 (s). HRMS (FAB) Calcd for C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>NS (M+H<sup>+</sup>): 186.0219. Found: 186.0215.

### ***O*-Acetyl acetylhydroxamic acid<sup>11</sup>**



Acetyl hydroxamic acid (1.5 g, 20 mmol), CH<sub>2</sub>Cl<sub>2</sub> (30 mL), and NaOH (2 M, 11 mL) were charged to a round bottomed flask. Ac<sub>2</sub>O (2.2 g, 22 mmol) was added via syringe. After stirring at room temperature for 2 h, the CH<sub>2</sub>Cl<sub>2</sub> layer was separated. The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (10 mL) and the CH<sub>2</sub>Cl<sub>2</sub> layers were combined and washed with brine and dried over MgSO<sub>4</sub>. Evaporation of the solvent gave the product as a white solid (1.7 g, 76%). Mp 85-86 °C (hexanes/EtOAc, lit. {88-90 °C (hexanes/ether)}<sup>11</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.91 (s, 1 H), 2.24 (s, 3 H), 2.07 (s, 3 H). IR (neat, cm<sup>-1</sup>): 2957 (m), 1787 (s), 1668 (s)

### ***O*-Acetyl laurohydroxamic acid**

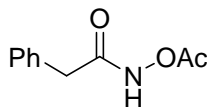


Laurohydroxamic acid<sup>12</sup> (2.2 g, 10 mmol), CH<sub>2</sub>Cl<sub>2</sub> (30 mL) and NaOH (2 M, 11 mL) were charged to a round bottomed flask. Ac<sub>2</sub>O (1.1 g, 11 mmol) was added via syringe. After stirring at room temperature for 2 h, the CH<sub>2</sub>Cl<sub>2</sub> layer was separated. The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (10 mL). The CH<sub>2</sub>Cl<sub>2</sub> layers were combined and washed with brine and dried over MgSO<sub>4</sub>. Evaporation of the solvent gave the product as a white solid (2.2 g, 85%). Mp 95-96 °C (hexanes/EtOAc). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.59 (s, 1 H), 2.24 (m, 5 H), 1.68 (m, 2 H), 1.28 (br, 16 H), 0.87 (t,  $J$  = 7.2 Hz, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.7, 169.0, 33.0, 32.1, 29.8, 29.6, 29.53, 29.50, 29.3, 25.3, 22.8, 18.4, 14.3. IR (neat, cm<sup>-1</sup>): 2916 (s), 2847 (s), 1790 (s), 1661 (s). HRMS(FAB) Calcd for C<sub>14</sub>H<sub>28</sub>O<sub>3</sub>N (M+H<sup>+</sup>): 258.2063. Found: 258.2057.

<sup>11</sup> Narita, M.; Akiyama, M.; Okawara, M. *Bull. Chem. Soc. Jpn.* **1971**, *44*, 437-441.

<sup>12</sup> Laurohydroxamic acid was prepared by the procedure: Reddy, A. S.; Kumar, M. S.; Reddy, G. R. *Tetrahedron Lett.* **2000**, *41*, 6285-6288.

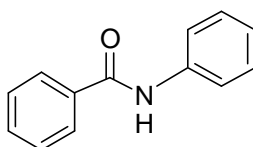
### ***N*-Acetoxy-2-phenylacetamide<sup>13</sup>**



Hydroxylamine hydrochloride (2.78 g, 40 mmol) and NaOH pellets (2 g, 50 mmol) were dissolved in water (20 mL) and EtOAc (20 mL). Phenylacetyl chloride (1.578 g, 10 mmol) was then added via syringe. After stirring the mixture at room temperature overnight, 2 M HCl was added to acidify the solution to pH=1. The organic layer was separated, washed with brine then dried over MgSO<sub>4</sub>. Evaporation of the solvent gave 0.830 g of an off-white solid, which was then dissolved in THF (30 mL). After purging with argon, AcCl (0.453 g, 5.8 mmol) and Et<sub>3</sub>N (0.587 g, 5.8 mmol) were added subsequently via syringe. The suspension was stirred at room temperature overnight. After filtration of the solid, EtOAc (10 mL) was added to the filtrate which was washed with water, then brine. Drying over MgSO<sub>4</sub> and evaporation of the solvent gave a white solid. Recrystallization from hexanes/Et<sub>2</sub>O gave the product as needle-shape colorless crystals in 52% yield (1.0 g). Mp 139-140 °C (lit. {141-142 °C}<sup>13</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.86 (br, s, 1 H), 7.30-7.38 (m, 5 H), 3.65 (s, 2 H), 2.18 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3142 (s), 2937 (m), 1791 (s), 1702 (s), 1652 (s).

**General procedure for Cu(I) mediated reaction of *O*-substituted aryl/alkylhydroxamic acid and arylboronic acid.** *O*-Substituted hydroxamic acid (0.2 mmol), boronic acid (0.24 mmol) and CuTC (0.2 mmol) were added to a Schlenk tube. After flushing with argon, THF (5 mL) was added via syringe. The reaction mixture was stirred under the protection of argon at 60 °C for 16 h. Ethyl ether (10 mL) was added to the mixture. The organic layer was washed with brine, dried over MgSO<sub>4</sub> and evaporated. The residue was then subjected to preparative plate silica gel chromatography using hexanes/EtOAc as eluent.

### ***N*-Phenylbenzamide<sup>14</sup>**



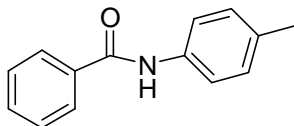
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a light yellow solid (30 mg, 74%). Mp

<sup>13</sup> Cherest, M.; Lusinch, X. *Tetrahedron* **1986**, 42, 3825-3840.

<sup>14</sup> Huang, H. H.; Tan, B. G. *J. Chem. Soc. Perkin Trans.2* **1983**, 233-235.

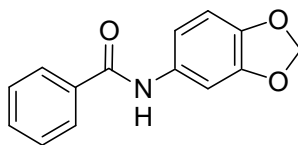
161-161.5 °C (hexanes/EtOAc, lit. {162-163 °C (ethanol)}<sup>14</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.97 (br, s, 1 H), 7.86 (d, *J* = 7.6 Hz, 2 H), 7.64 (d, *J* = 7.6 Hz, 2 H), 7.34-7.56 (m, 5 H), 7.15 (t, *J* = 7.6 Hz, 1 H). IR (neat, cm<sup>-1</sup>): 3343 (s), 3053 (m), 2918 (m), 1656 (s), 1598 (s), 1525 (s), 1440 (s).

#### ***N*-*p*-Tolylbenzamide<sup>15</sup>**



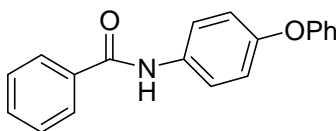
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a light yellow solid (32 mg, 77%). Mp 157-158 °C (hexanes/EtOAc, {lit. 158 °C}<sup>15</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.99 (br, s, 1 H), 7.84 (d, *J* = 8.0 Hz, 2 H), 7.42-7.53 (m, 5 H), 7.14 (d, *J* = 8.4 Hz, 2 H), 2.34 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3312 (s), 3053 (m), 2918 (m), 1648 (s), 1579 (m), 1513 (m).

#### ***N*-(1,3-Benzodioxol-5-yl)benzamide<sup>16</sup>**



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a yellow solid (30 mg, 63%). Mp 134-135 °C (hexanes/EtOAc). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.13 (br, s, 1 H), 7.81 (d, *J* = 8.4 Hz, 2 H), 7.38-7.52 (m, 3 H), 7.30 (d, *J* = 0.8 Hz, 1 H), 6.90 (dd, *J* = 8.4, 2.0 Hz, 1 H), 6.72 (d, *J* = 8.4 Hz, 1 H), 5.93 (s, 2 H). IR (neat, cm<sup>-1</sup>): 3308 (s), 2891 (m), 1644 (s), 1540 (m), 1490 (m).

#### ***N*-(4-Phenoxyphenyl)benzamide<sup>17</sup>**



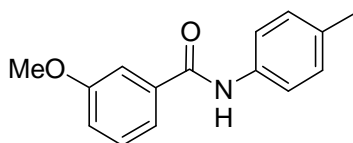
<sup>15</sup> Haridasan, V. K.; Ajayaghosh, A.; Pillai, V. N. Rajasekharan *J. Org. Chem.* **1987**, 52, 2662-2665.

<sup>16</sup> (a) Commercially available from Exploratory Library. (b) Monteil, A.; Simond, J.; Combourieu, M. Eur. Pat. Appl. (1985), EP 84-401919 19840926.

<sup>17</sup> (a) Konieczny, J. M.; Wunder, S. L. *Macromolecules* **1996**, 29, 7613-7615. (b) Ruane, P. H.; Ahmed, A. R.; McClelland, R. A. *J. Chem. Soc. Perkin Trans. 2* **2002**, 312-317.

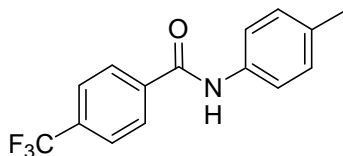
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (40 mg, 68%). Mp 159-160 °C (diethyl ether). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.93 (br, s, 1 H), 7.86 (d, *J* = 7.6 Hz, 2 H), 7.45-7.61 (m, 5 H), 7.31-7.36 (m, 2 H), 6.99-7.12 (m, 5 H). IR (neat, cm<sup>-1</sup>): 3347 (m), 3057 (m), 1652 (s), 1606 (m), 1525 (m), 1505 (s), 1486 (s).

### 3-Methoxy-*N-p*-tolylbenzamide<sup>18</sup>



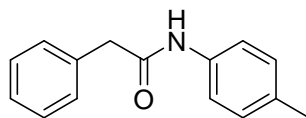
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (28 mg, 58%). Mp 129-130 °C (hexanes/EtOAc, lit. {132 °C (ethanol)}<sup>18</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98 (br, s, 1 H), 7.51 (d, *J* = 8.4 Hz, 2 H), 7.31-7.42 (m, 3 H), 7.14 (d, *J* = 8.4 Hz, 2 H), 7.05 (dq, *J* = 7.6 Hz, 1.4, 1 H), 3.82 (s, 3 H), 2.33 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3296 (m), 2922 (m), 1648 (s), 1594 (s), 1517 (s).

### *N*-(4-Methylphenyl)-4-(trifluoromethyl)benzamide<sup>19</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (46 mg, 81%). Mp 236-238 °C (hexanes/EtOAc). <sup>1</sup>H NMR (400 MHz, *d*<sub>6</sub>-acetone) δ 9.66 (br, s, 1 H), 8.18 (d, *J* = 8.4 Hz, 2 H), 7.86 (d, *J* = 8.4 Hz, 2 H), 7.72 (dd, *J* = 8.0, 1.6 Hz, 2 H), 7.18 (d, *J* = 8.4 Hz, 2 H), 2.31 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3424 (m), 3061 (m), 3007 (m), 1710 (s), 1420 (m), 1363 (s).

### 2-Phenyl-*N-p*-tolyl-acetamide<sup>20</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a light yellow solid (34 mg, 76%). Mp

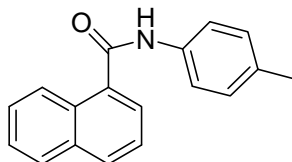
<sup>18</sup> Grammaticakis, P. *Bull. Soc. Chim. Fr.* **1964**, 5, 924-935.

<sup>19</sup> Commercially available from Exploratory Library.

<sup>20</sup> House, H. O.; Berkowitz, W. F. *J. Org. Chem.* **1963**, 28, 307-311.

131-132.5 °C (hexanes/EtOAc, lit. {132-133.8 °C (hexanes/EtOAc)}<sup>20</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.30-7.40 (m, 8 H), 7.07 (d, *J* = 8.4 Hz, 2 H), 3.70 (s, 2 H), 2.29 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3300 (m), 3034 (m), 1656 (s), 1606 (s), 1536 (s), 1517 (s).

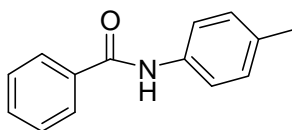
#### Naphthalene-1-carboxylic acid *p*-tolyl-amide<sup>21</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (34 mg, 65%). Mp 190-191 °C (hexanes/EtOAc, lit. {191.9-192.9 °C}<sup>21</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.34-8.36 (m, 1 H), 7.94 (d, *J* = 8.0 Hz, 1 H), 7.88-7.90 (m, 1 H), 7.17 (d, *J* = 6.4 Hz, 2 H), 7.53-7.58 (m, 4 H), 7.45 (t, *J* = 7.6 Hz, 1 H), 7.19 (d, *J* = 8.0 Hz, 2 H), 2.36 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3235 (m), 2922 (m), 1648 (s), 1598 (m), 1517 (s).

**General procedure for Cu(I) mediated reaction of *O*-acetyl aryl/heteroaryl/alkylhydroxamic acid with organostannane.** *O*-Substituted hydroxamic acid (0.2 mmol), organostannane (0.24 mmol) and CuDPP (0.4 mmol) were added to a Schlenk tube. After flushing with argon, DMF (5 mL) was added via syringe. The reaction mixture was stirred under the protection of argon at 60 °C for 12 h. Ethyl ether (10 mL) was added to the mixture. The organic layer was washed with brine, dried over MgSO<sub>4</sub> and evaporated. The residue was then subjected to preparative plate silica chromatography using hexanes/EtOAc as eluent.

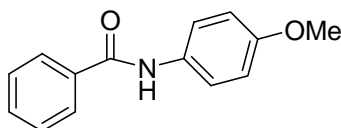
#### *N*-*p*-Tolylbenzamide<sup>15</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a light yellow solid (33 mg, 80%). Mp 157-158 °C (hexanes/EtOAc, {lit. 158 °C}<sup>15</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.99 (br, s, 1 H), 7.84 (d, *J* = 8.0 Hz, 2 H), 7.42-7.53 (m, 5 H), 7.14 (d, *J* = 8.4 Hz, 2 H), 2.34 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3312 (s), 3053 (m), 2918 (m), 1648 (s), 1579 (m), 1513 (m).

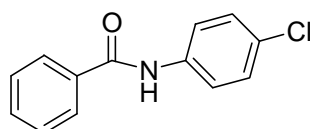
<sup>21</sup> Jart, A. *Acta Polytech. Scand., Chem. Met. Ser.* **1965**, *44*, 1.

### ***N*-*p*-Methoxyphenyl-benzamide<sup>22</sup>**



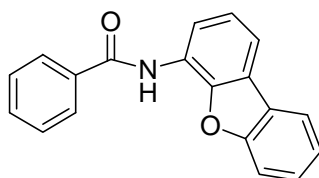
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (34 mg, 76%). Mp 156-157 °C (hexanes/EtOAc, lit. {156-157°C}<sup>22</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 7.2 Hz, 2 H), 7.79 (s, 1 H), 7.55-7.47 (m, 5 H), 6.92 (d, *J* = 7.2 Hz, 2 H), 3.82 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3331 (m), 3053 (m), 1644 (s), 1602 (m), 1517 (s).

### ***N*-*p*-Chlorophenyl-benzamide<sup>23</sup>**



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (26 mg, 56%). Mp 188-190 °C (hexanes/EtOAc, lit. {190-191°C}<sup>23</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.88 (d, *J* = 6.8 Hz, 2 H), 7.80 (s, 1 H), 7.62-7.49 (m, 5 H), 7.36 (d, *J* = 8.8 Hz, 2 H). IR (neat, cm<sup>-1</sup>): 3335 (m), 1648 (s), 1594 (m), 1521 (s).

### ***N*-4-Dibenzofuranylbenzamide**

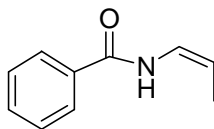


Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (42 mg, 73%). Mp 195-196 °C (hexanes/EtOAc). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.55 (d, *J* = 7.6 Hz, 1 H), 8.48 (s, 1 H), 8.05-7.97 (m, 3 H), 7.74 (dd, *J* = 8.0, 1.2 Hz, 1 H), 7.65-7.56 (m, 4 H), 7.52-7.48 (m, 1 H), 7.42-7.28 (m, 2 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.8, 155.9, 146.0, 134.9, 132.3, 129.1, 127.5, 127.4, 124.7, 124.5, 124.0, 123.8, 123.4, 121.2, 118.2, 116.2, 111.8. IR (neat, cm<sup>-1</sup>): 3239 (m), 1648 (s), 1525 (s). HRMS (FAB) Calcd for C<sub>19</sub>H<sub>14</sub>O<sub>2</sub>N (M+H<sup>+</sup>): 288.1019. Found: 288.1015.

<sup>22</sup> Katritzky, A. R. Cai, C.; Singh, S. K. *J. Org. Chem.* **2006**, *71*, 3375-3380.

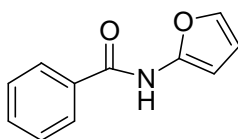
<sup>23</sup> Singh, H; Aggarwal, S. K.; Malhotra, N. *Synthesis* **1983**, *10*, 791-793.

***N*-(*(Z)*-Prop-1-enyl)benzamide**<sup>24</sup>



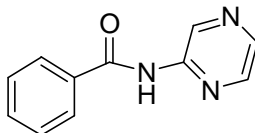
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded light yellow oil (22 mg, 66%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.83-7.81 (m, 2 H), 7.58-7.46 (m, 4 H), 6.99-6.93 (m, 1 H), 4.98-4.94 (m, 1H), 1.73 (dd, *J* = 7.2, 2.0 Hz, 3 H). IR (neat, cm<sup>-1</sup>): 2926 (s), 1656 (s), 1606 (m), 1529 (s).

***N*-(Furan-2-yl)benzamide**<sup>25</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (27 mg, 73%). Mp 116-118 °C (hexanes/EtOAc, lit. {121-122°C}<sup>25</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.26 (s, 1 H), 7.88 (d, *J* = 7.6 Hz, 2 H), 7.59-7.48 (m, 3 H), 7.12 (dd, *J* = 2.0, 1.2 Hz, 1 H), 6.50 (d, *J* = 3.2 Hz, 1 H), 6.49 (dd, *J* = 3.2, 2.0 Hz, 1 H). IR (neat, cm<sup>-1</sup>): 3065 (m), 1660 (s), 1606 (m), 1540 (s).

***N*-(Pyrazin-2-yl)benzamide**<sup>26</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (17 mg, 42%). Mp 169-170 °C (hexanes/EtOAc, lit. {170°C}<sup>26</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.74 (s, 1 H), 8.51 (s, 1H), 8.41 (d, *J* = 2.4 Hz, 1 H), 8.29 (s, 1H), 7.96-7.94 (m, 2 H), 7.62-7.52 (m, 3 H). IR (neat, cm<sup>-1</sup>): 3242 (m), 3065 (m), 1675 (s), 1583 (m), 1532 (s).

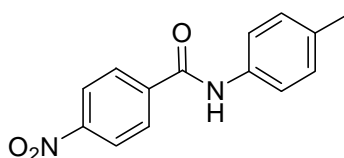
***N*-(4-Methylphenyl)-4-nitrobenzamide**<sup>27</sup>

<sup>24</sup> Liskamp, R. M. J.; Blom, H. J.; Nivard, R. J. F.; Ottenheijm, H. C. J. *J. Org. Chem.* **1983**, *48*, 2733-2736.

<sup>25</sup> Padwa, A.; Crawford, K. R.; Rashatasakhon, P.; Rose, M. *J. Org. Chem.* **2003**, *68*, 2609-2617.

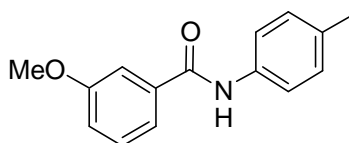
<sup>26</sup> Barlin, G. B.; Brown, D. J.; Kadunc, Z.; Petric, A.; Stanovnik, B.; Tisler, M. *Aust. J. Chem.* **1983**, *36*, 1215-1220.





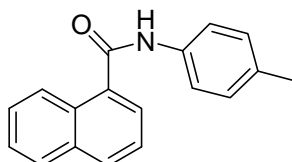
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (37 mg, 74%). Mp 198-199 °C (hexanes/EtOAc, lit. {201-203°C}<sup>27</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.34 (d, *J* = 9.2 Hz, 2 H), 8.05 (d, *J* = 8.4 Hz, 2 H), 7.84 (s, 1 H), 7.53 (d, *J* = 8.0 Hz, 2 H), 7.21 (d, *J* = 8.0 Hz, 2 H), 2.36 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3312 (m), 1644 (s), 1598 (m), 1525 (s).

### 3-Methoxy-*N-p*-tolylbenzamide<sup>28</sup>



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (34 mg 71%). Mp 125-126 °C (hexanes/EtOAc, lit. {132 °C (ethanol)}<sup>28</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.82 (s, 1 H), 7.53 (d, *J* = 8.4 Hz, 2 H), 7.43-7.37 (m, 3 H), 7.19 (d, *J* = 8.4 Hz, 2 H), 7.09 (m, 1 H), 3.87 (s, 3 H), 2.35 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3296 (m), 3034 (m), 1648 (s), 1594 (s), 1517 (s).

### Naphthalene-1-carboxylic acid *p*-tolyl-amide<sup>29</sup>



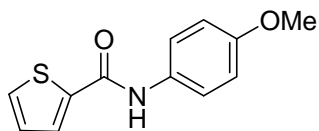
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (43 mg 83%). Mp. 192-193 °C (hexanes/EtOAc, lit. {191.9-192.9 °C}<sup>29</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.39 (d, *J* = 7.6 Hz, 1 H), 7.98 (d, *J* = 8.0 Hz, 1 H), 7.92-7.90 (m, 1 H), 7.76 (d, *J* = 6.8 Hz, 1 H), 7.64-7.50 (m, 6 H), 7.23 (d, *J* = 8.4 Hz, 2 H), 2.37 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3235 (m), 3046 (m), 1652 (s), 1598 (s), 1517 (s).

### *N*-(4-Methoxyphenyl)thiophene-2-carboxamide<sup>30</sup>

<sup>27</sup> Han, K-J; Tae, B. S.; Kim, M. *Org. Prep. Proced. Int.* **2005**, 37, 198-203.

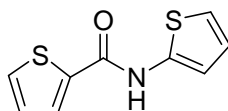
<sup>28</sup> Grammaticakis, P. *Bull. Soc. Chim. Fr.* **1964**, 5, 924-935.

<sup>29</sup> Jart, A. *Acta Polytech. Scand., Chem. Met. Ser.* **1965**, 44, 1.



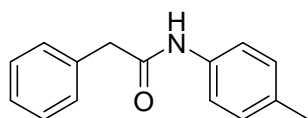
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (42 mg, 90%). Mp 139-140 °C (hexanes/EtOAc, lit. {137-140 °C (hexanes/EtOAc)}<sup>30</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69 (s, 1 H), 7.62 (d, *J* = 3.6 Hz, 1 H), 7.54-7.50 (m, 3 H), 7.12 -7.10 (m, 1 H), 6.91-6.88 (m, 2 H), 3.81 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3281 (m), 3003 (m), 1633 (s), 1602 (m), 1513 (s).

***N*-(Thiophen-2-yl)thiophene-2-carboxamide<sup>31</sup>**



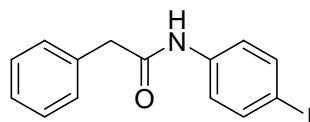
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (25 mg, 60%). Mp 205-206 °C <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.46 (s, 1 H), 7.68 (dd, *J* = 3.6, 1.2 Hz, 1 H), 7.58 (dd, *J* = 5.2, 1.2 Hz, 1 H), 7.15 (dd, *J* = 5.2, 3.6 Hz, 1 H), 6.95 (dd, *J* = 5.6, 1.2 Hz, 1 H), 6.91 (dd, *J* = 5.6, 3.6 Hz, 1 H), 6.80 (dd, *J* = 3.6, 1.2 Hz, 1 H). IR (neat, cm<sup>-1</sup>): 3227 (m), 1617 (s), 1563 (s), 1513 (s).

**2-Phenyl-*N*-*p*-tolylacetamide<sup>17</sup>**



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (33 mg, 74%). Mp 130-131 °C (hexanes/EtOAc, lit. {132-133.8 °C (hexanes/EtOAc)}<sup>17</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41-7.28 (m, 7 H), 7.09 (d, *J* = 8.4 Hz, 2 H), 7.04 (s, 1 H), 3.73 (s, 2 H), 2.29 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3300 (m), 1656 (s), 1606 (s), 1536 (s).

***N*-(4-Iodophenyl)-2-phenylacetamide<sup>32</sup>**



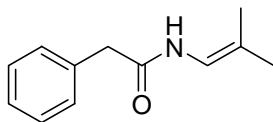
<sup>30</sup> Lee, C. K.; Yu, J. S.; Ji, Y. R. *J. Heterocycl. Chem.*, **2002**, 39, 1219-1227.

<sup>31</sup> Brovelli, F.; del Valle, M. A.; Diaz, F. R.; Bernede, J. C. *Bol. Soc. Chil. Quim.*, **2001**, 46, 319-337.

<sup>32</sup> Chattaway, F. D.; Constable, A. *J. Chem. Soc., Trans.*, **1914**, 105, 124-131.

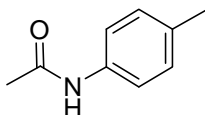
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (40 mg, 60%). Mp 195-196 °C (hexanes/EtOAc, lit. {200 °C }<sup>32</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.59-7.57 (m, 2 H), 7.44-7.32 (m, 5 H), 7.21-7.19 (m, 2 H), 7.02 (s, 1 H), 3.74 (s, 2 H). IR (neat, cm<sup>-1</sup>): 3273 (m), 3026 (m), 1656 (s), 1583 (m), 1525 (s).

***N*-(2-Methylprop-1-enyl)-2-phenylacetamide<sup>33</sup>**



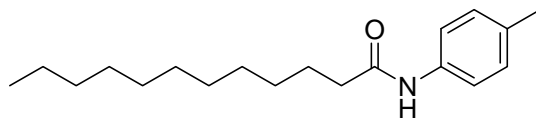
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (24 mg, 64%). Mp 98-99 °C (hexanes/EtOAc, lit. {102 °C (hexanes/EtOAc)}<sup>33</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42-7.28 (m, 5 H), 6.64 (s, 1 H), 6.51-6.48 (m, 1 H), 3.65 (s, 2 H), 1.65 (s, 3 H), 1.35 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3300 (m), 2930 (m), 1660 (s), 1637 (s), 1529 (s).

***N*-*p*-Tolylacetamide<sup>34</sup>**



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (25 mg, 85%).. Mp 146-147 °C (hexanes/EtOAc, lit. {146-148 °C (hexanes/EtOAc)}<sup>34</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.38 (d, *J* = 8.4 Hz, 2 H), 7.13 (d, *J* = 8.4 Hz, 2 H), 2.31 (s, 3 H), 2.16 (s, 3 H). IR (neat, cm<sup>-1</sup>): 3289 (m), 2922 (m), 1660 (s), 1602 (s), 1532 (s).

***N*-*p*-Tolyldecylamide<sup>35</sup>**



Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (38 mg, 66%).. Mp 83-84 °C (hexanes/EtOAc, lit. {82-83 °C (hexanes/EtOAc)}<sup>35</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.40 (d, *J* = 8.0 Hz, 2 H), 7.18 (s, 1 H), 7.13 (d, *J* = 8.0 Hz, 2 H), 2.35-2.31 (m, 5 H), 1.73-1.70 (m, 2 H), 1.32-1.26 (br, 16 H), 0.90 (t, *J* = 6.8 Hz,

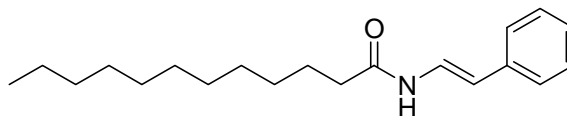
<sup>33</sup> Zehavi, U.; Ben-Ishai, D. *J. Org. Chem.* **1961**, 26, 1097-1101.

<sup>34</sup> Lobo, A. M.; Marques, M. M.; Prabhakar, S.; Rzepa, H. S. *J. Org. Chem.* **1987**, 52, 2925-2927.

<sup>35</sup> Robertson, P. W. *J. Chem. Soc., Trans.*, **1919**, 115, 1210-1223.

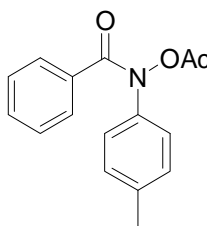
3 H). IR (neat,  $\text{cm}^{-1}$ ): 3312 (m), 2922 (s), 1660 (s), 1529 (s).

***N*-(*E*)- $\beta$ -Styryldodecanamide**



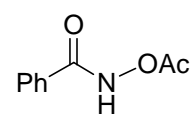
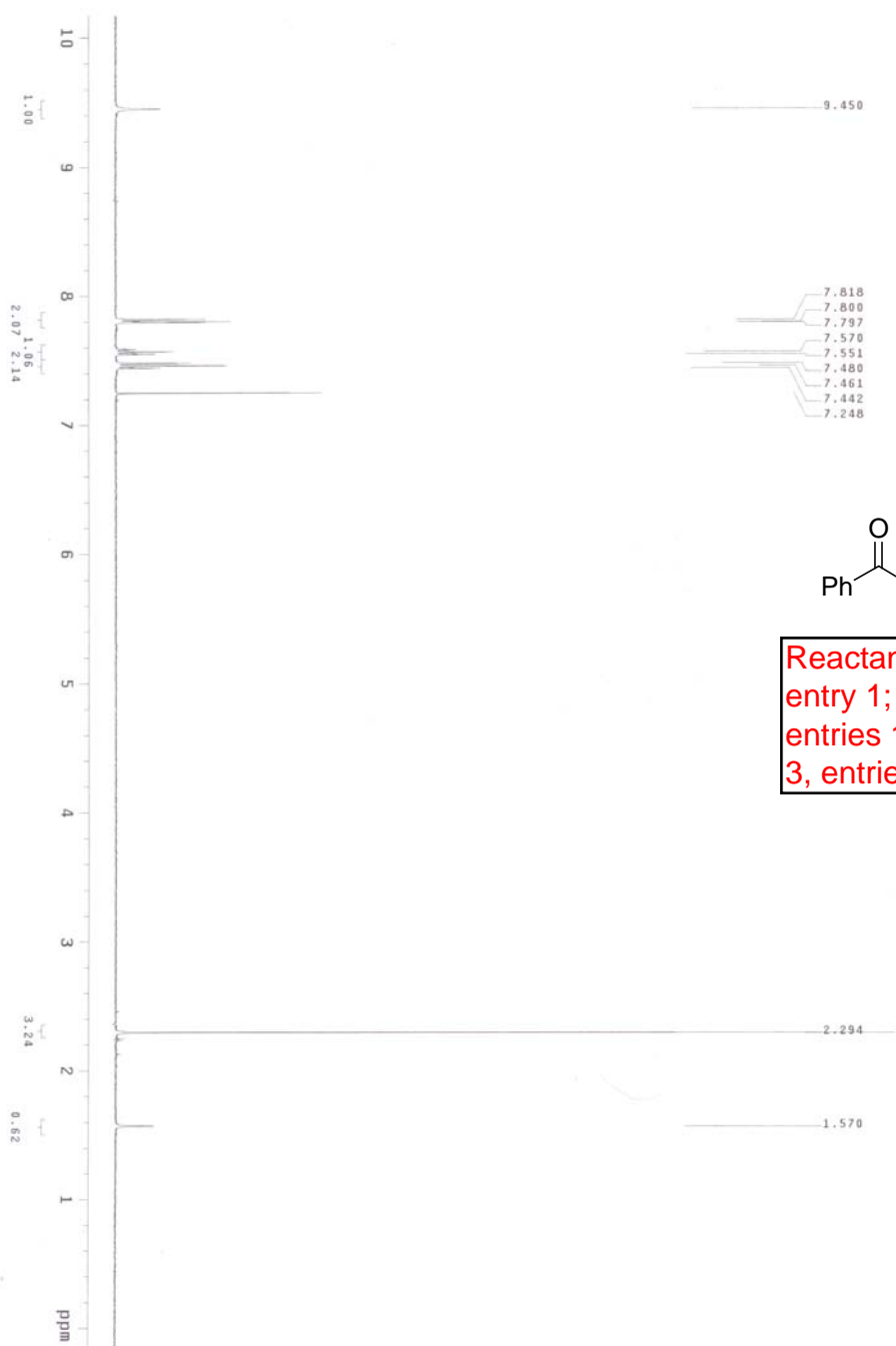
Purification by preparative TLC (hexanes/EtOAc 2:1) afforded a white solid (31 mg, 52%). Mp 103-104 °C (hexanes/EtOAc)  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 (dd,  $J$  = 14.4, 10.8 Hz, 1 H), 7.38-7.26 (m, 5 H), 7.19-7.15 (m, 1 H), 2.35-2.31 (m, 5 H), 6.11 (d,  $J$  = 14.4 Hz, 1H), 2.31 (t,  $J$  = 7.6 Hz, 2H), 1.71-1.66 (m, 2 H), 1.32-1.26 (br, 16 H), 0.90 (t,  $J$  = 6.8 Hz, 3 H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 136.2, 128.8, 126.8, 125.7, 122.8, 112.5, 37.0, 32.1, 29.8, 29.6, 29.5, 29.4, 25.7, 22.8, 14.3. IR (neat,  $\text{cm}^{-1}$ ): 3305 (m), 2916 (s), 2849 (m), 1664 (m), 1639 (s), 1522 (m). HRMS (FAB) Calcd for  $\text{C}_{20}\text{H}_{32}\text{ON}$  ( $\text{M}+\text{H}^+$ ): 302.2478. Found: 302.2474.

***N*-(acetyloxy)-*N*-(4-methylphenyl)-benzamide**

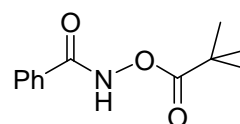


*O*-Acetyl benzohydroxamic acid (36 mg, 0.2 mmol), *p*-tolyl boronic acid (33 mg, 0.24 mmol) and  $\text{Cu}(\text{OAc})_2$  (36 mg, 0.2 mmol) were added to a flask. DMF (5 mL) was added via syringe. The reaction mixture was stirred under air at 60 °C for 12 h. Ethyl ether (10 mL) was added to the mixture. The organic layer was washed with brine, dried over  $\text{MgSO}_4$  and evaporated. The residue was then subjected to preparative plate silica gel chromatography using hexanes/EtOAc as eluent. The product was obtained as a yellow oil (23 mg, 43%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54 (d,  $J$  = 7.2 Hz, 2 H), 7.38 (t,  $J$  = 7.2 Hz, 1 H), 7.29 (m, 2 H), 7.21 (d,  $J$  = 8.8 Hz, 2 H), 7.11 (d,  $J$  = 8.0 Hz, 2 H), 2.32 (s, 3 H), 2.19 (s, 3 H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 167.0, 139.1, 138.2, 133.4, 131.1, 130.0, 129.0, 128.2, 127.5, 21.3, 18.6. IR (neat,  $\text{cm}^{-1}$ ): 3069 (m), 2964 (m), 1795 (s), 1675 (s), 1509 (m). HRMS (FAB) Calcd for  $\text{C}_{16}\text{H}_{16}\text{O}_3\text{N}$  ( $\text{M}+\text{H}^+$ ): 270.1124. Found: 270.1120.

Under the same reaction conditions using CuTC in place of  $\text{Cu}(\text{OAc})_2$  the product of the reaction was *N*-*p*-tolylbenzamide (56%). Characterization data is provided above.



Reactant, Table 1,  
entry 1; Table 2,  
entries 1-6; Table  
3, entries 1-7.



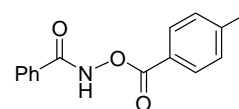
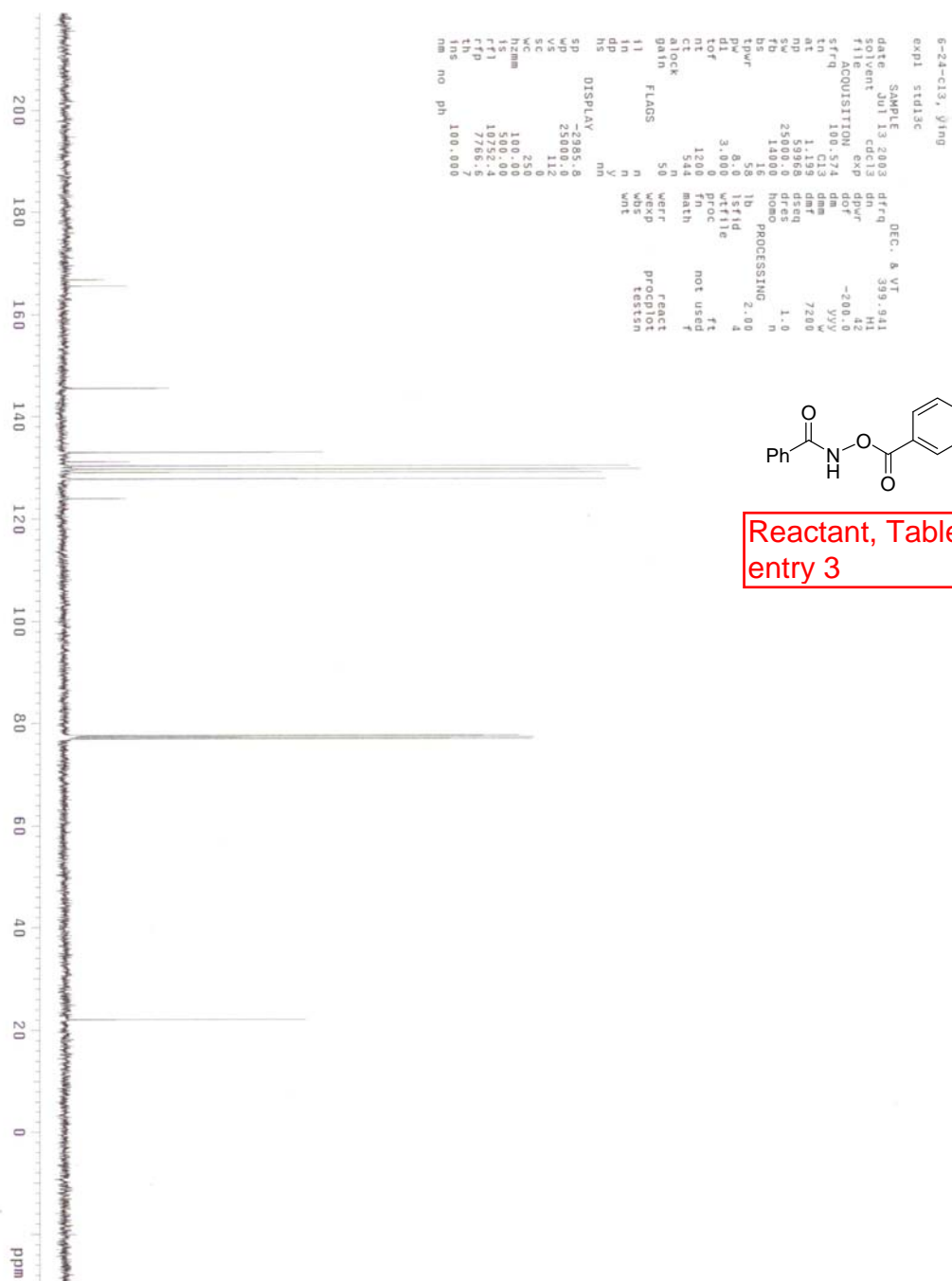
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sfrq 399.941 dm nm  
tn 11 dm c  
nt 9.212 dres 208  
sw 5203.3 dres 1.0  
fb 3000 homo n  
bs 16 PROCESSING 0.28  
cpwr 58 wfile  
dl 1.000 proc ft  
tof 207.0 fn not used  
nt 8 math f  
ct 8 wstr react  
block not used wexp procp101  
gain not used wts  
11 11 FLAGS n wnt  
1n n  
dp y  
bs DISPLAY nm  
sp -390.3  
wp 5203.3  
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vc 126  
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hzem 20.81  
15 119.78  
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ins nm cdc ph

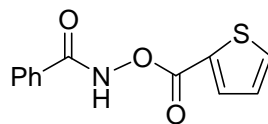




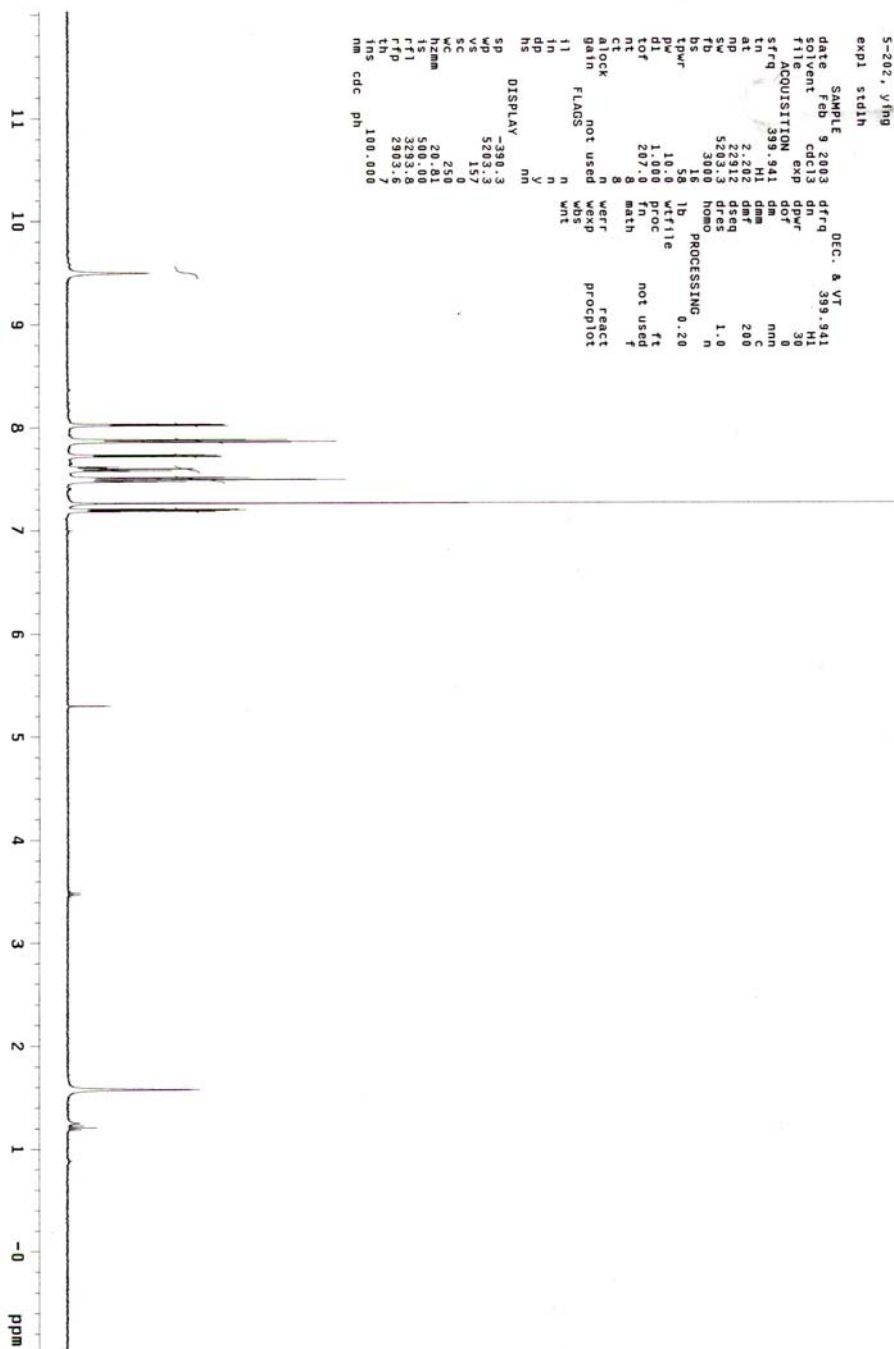


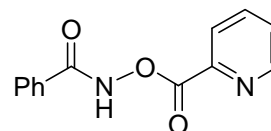
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entry 3



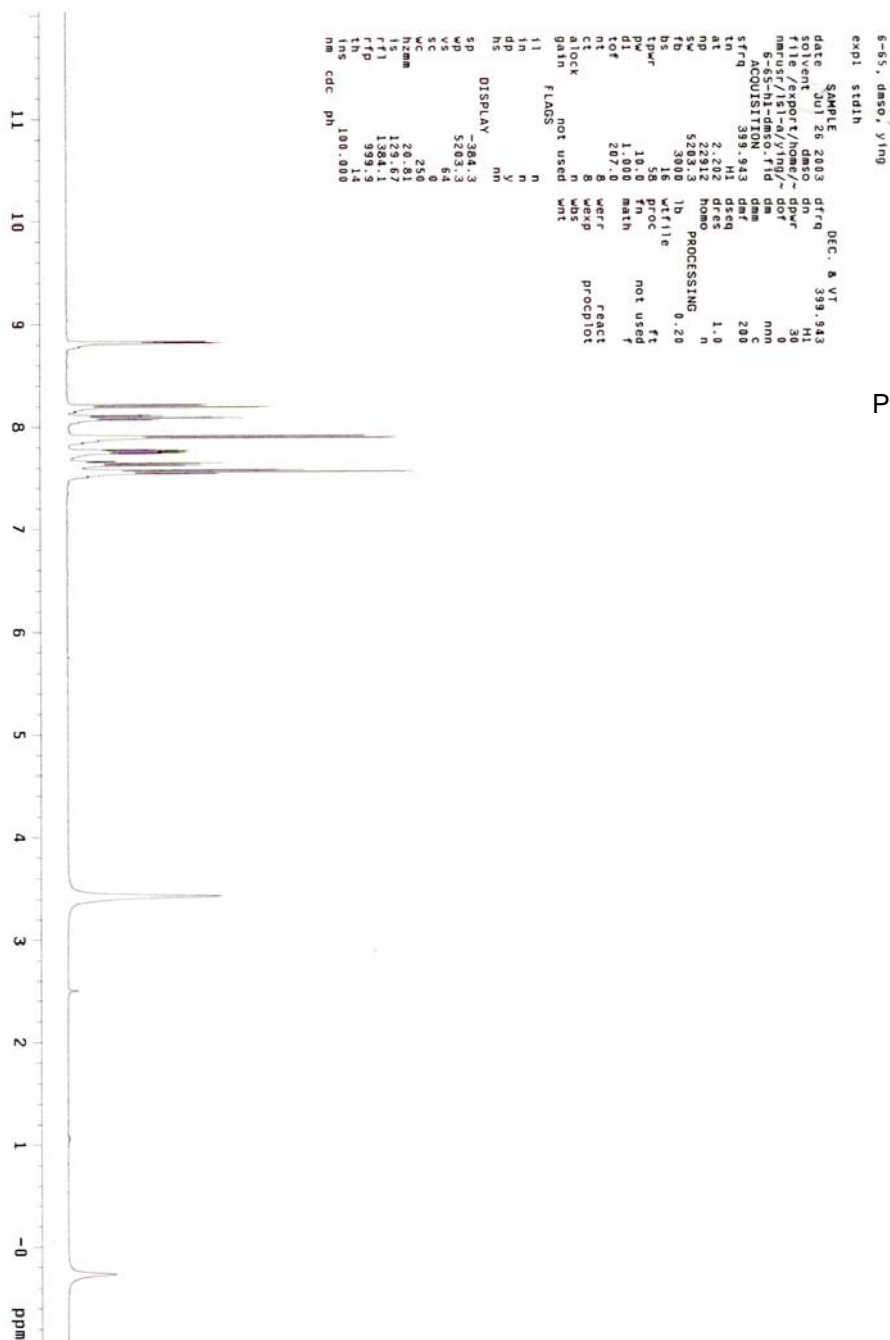


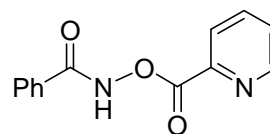
Reactant, Table 1,  
entry 4





Reactant, Table 1,  
entry 5

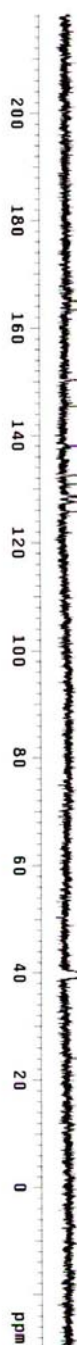


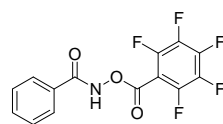
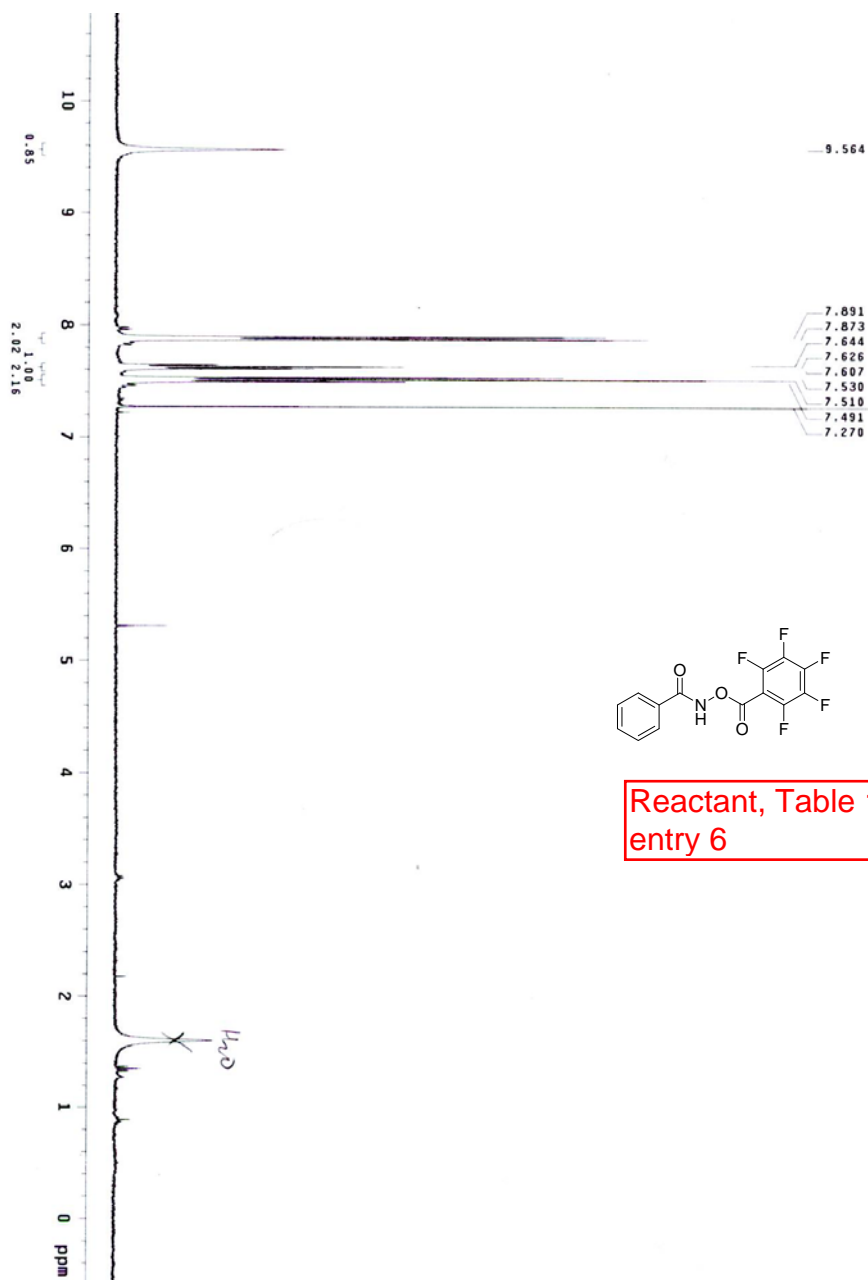
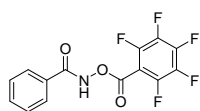


Reactant, Table 1,  
entry 5

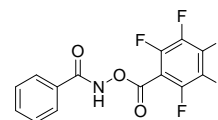
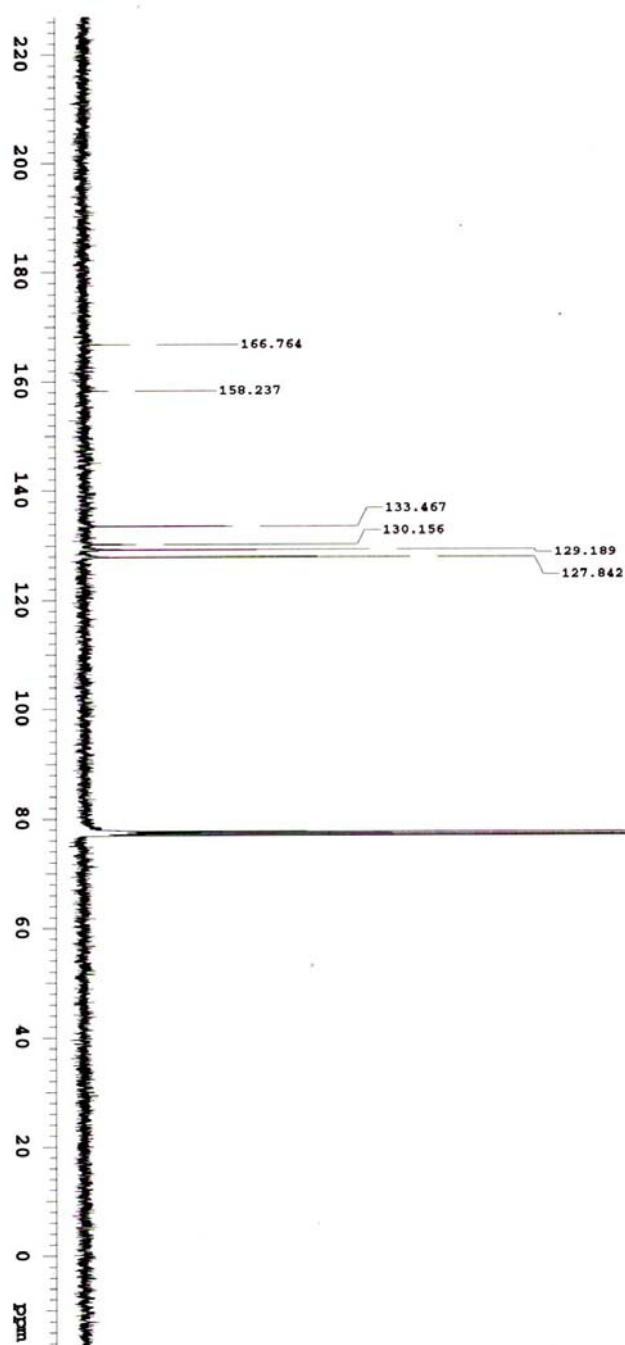
6-65, c13, dms0, y1ng  
expt std13c

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file	exp dpr	dot	-280.0
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tn	C13 dm	w	
nt	1.133 dm	7200	
sw	5968 dres	1.0	
fb	25000.0 dres		
bs	14000 homo		
PROCESSING			
dpr	16	2.00	
sw	8.00	15fid	4
nt	3.000	wfride	
ct	0	proc	ft
nt	1200	fn	not used
ct	64	math	f
block	50	weff	react
gain	n	weff	procp1ot
ll	n	testin	
in	n		
sp	nm		
DISPLAY			
sp	-3046.7		
wp	25000.0		
vc	96		
mc	250		
hzm	100.00		
ts	500.00		
fti	3020.0		
th	3973.5		
ins	100.000		
nm	no ph		

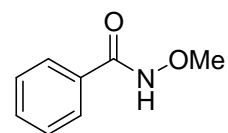
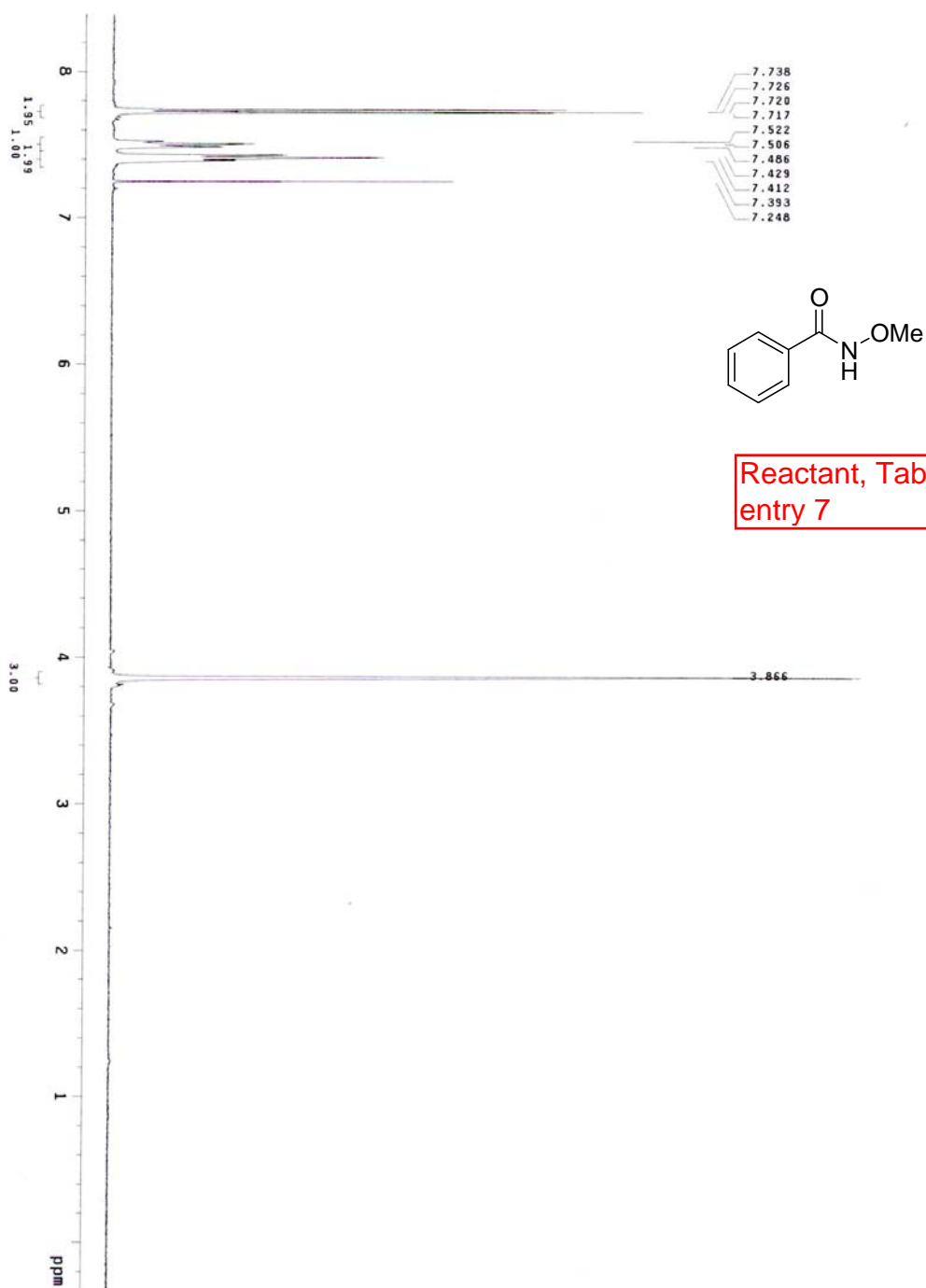




Reactant, Table 1,  
entry 6

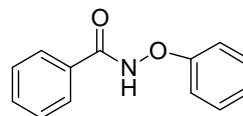


Reactant, Table 1,  
entry 6

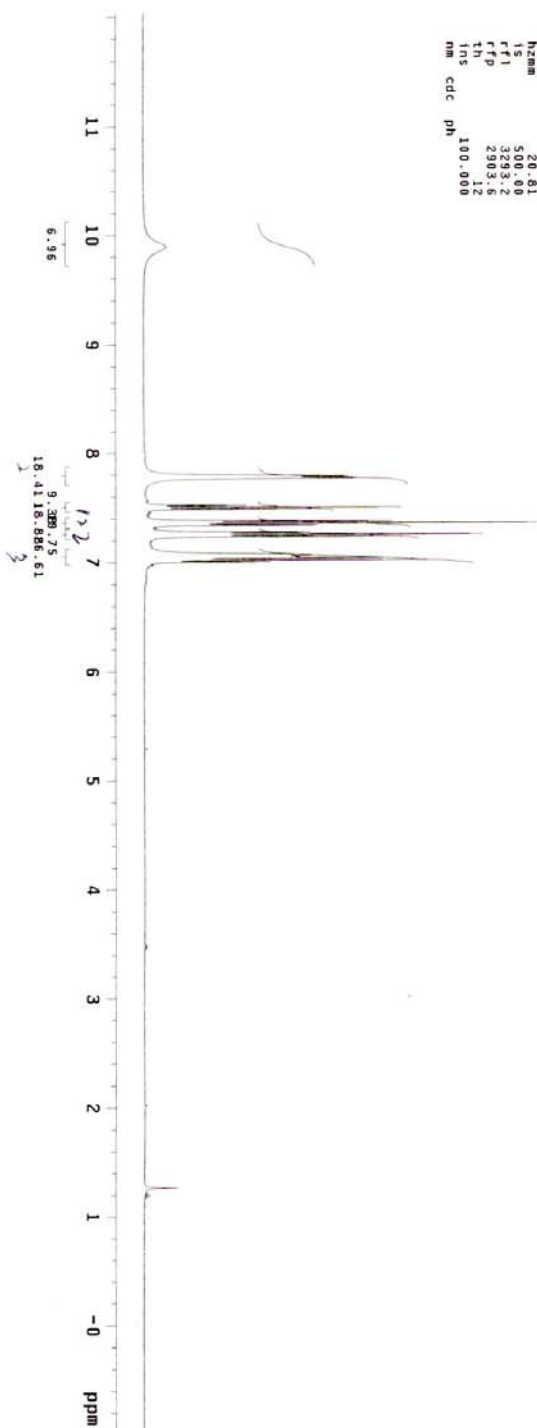


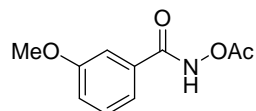
Reactant, Table 1,  
entry 7

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 solvent dcl3 do 30  
 file cdc13 dpr 0  
 ACQUISITION  
 sfrq 399.941 da 0  
 tn 30  
 at 2.202 dm 200  
 nu 2.202 dm 200  
 su 5203.3 dres 1.0  
 fb 3000 homo  
 bs 16  
 tpur 56 1b  
 pw 10.0 wflle 0.20  
 dl 1.000 proc ft  
 nt 207.8 math not used  
 ct 8  
 alock n weff react  
 gain not used wexp procpot  
 FLAGS  
 l1 n  
 l2 n  
 dn y  
 hs  
 DISPLAY  
 sp -389.6  
 wp 5203.3  
 vs 69  
 sc 250  
 hzmm 20.81  
 ls 500.00  
 rfi 3293.2  
 rfp 2903.6  
 th 12  
 nm cdc ph 100.000

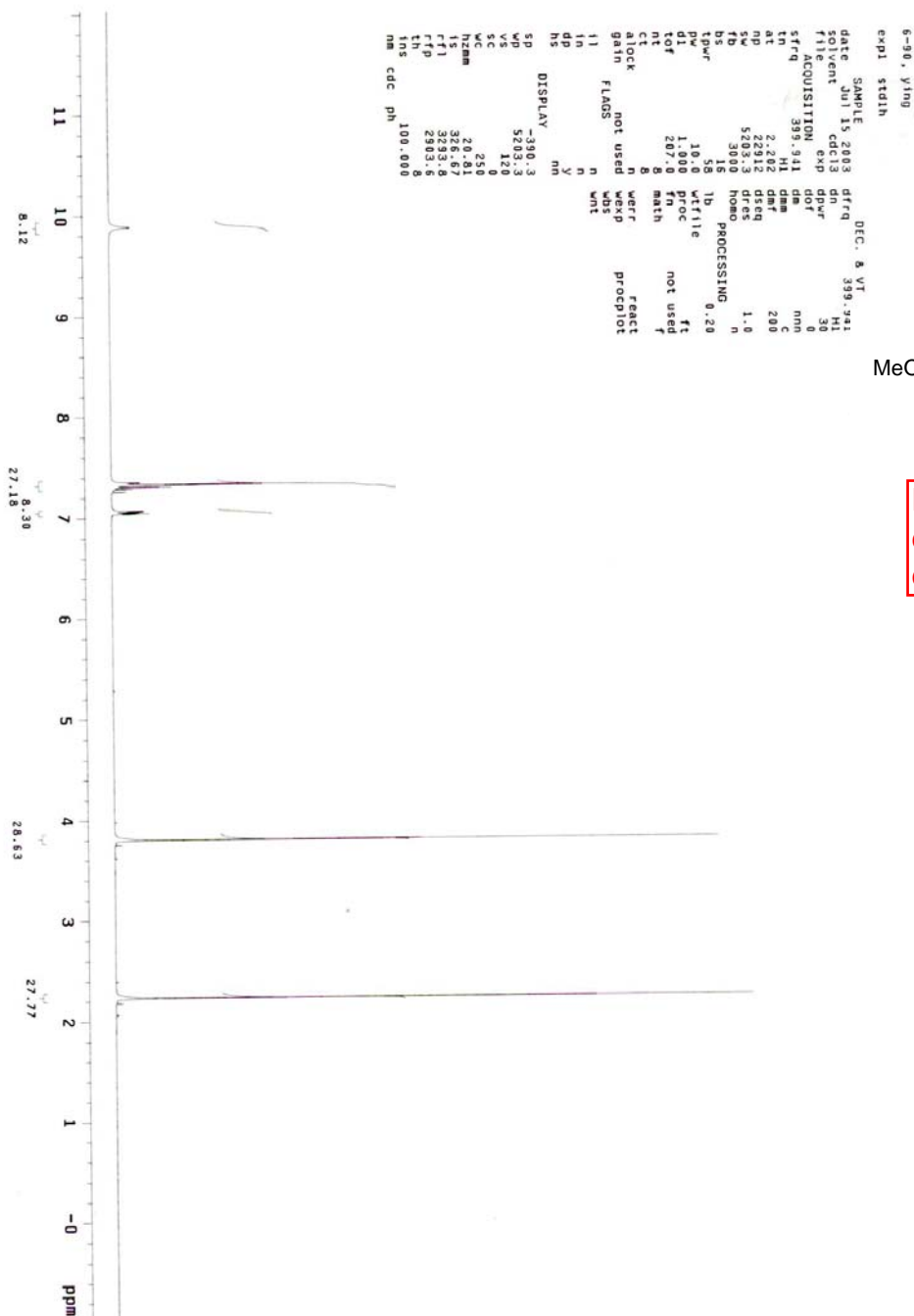


Reactant, Table 1,  
 entry 8

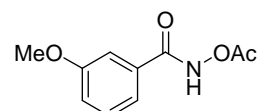




Reactant, Table 2,  
entry 7; Table 3,  
entry 9.



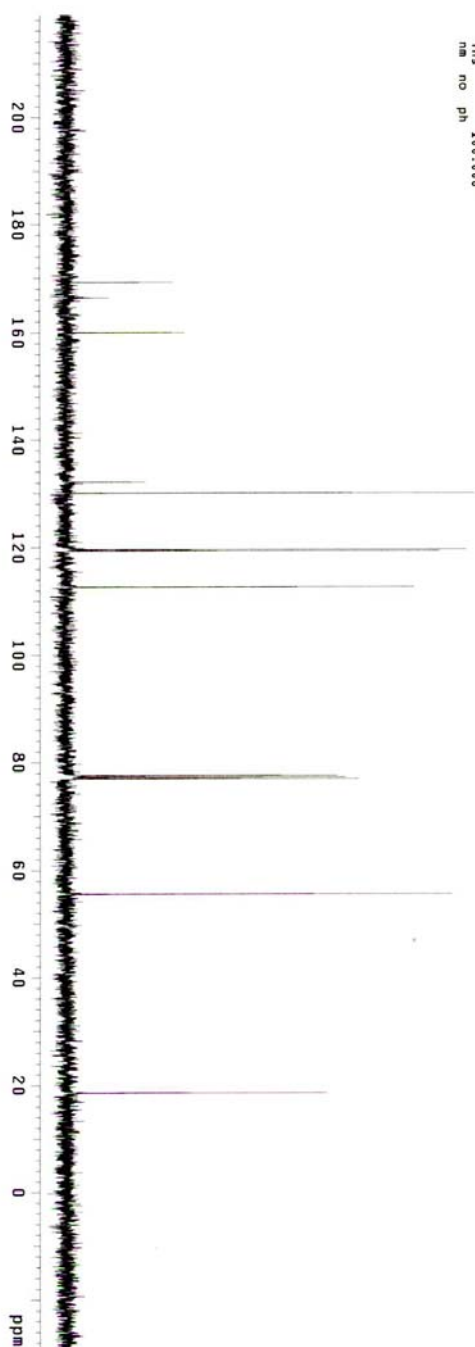


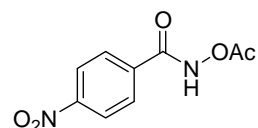


Reactant, Table 2,  
entry 7; Table 3,  
entry 9.

6-90, C13, y/mg  
exptl std13C

SAMPLE DEC. 8 VT  
date 2001.15.2003 dfrq 359.941  
solvent d11 cdcl3 dn 41  
file exp dpufr -200.0  
ACQUISITION  
sfrq 100.574 dm yyy  
an 1.133 dmf  
ap 5998.8 dseq 7200  
sw 25000.0 dres 1.0  
fb 14000 homo n  
bs 1.6 PROCESSING 2.00  
pufr 8.0 1b  
di 3.000 wfile 4  
nt 0 proc ft  
ct 800 fn not used  
sc 400 match f  
alock n  
gain 50 wefr react  
11 flags n wep proclot  
1n n wts testis  
dn n  
hs n  
DISPLAY  
sp -2988.9  
wp 25000.0  
vc 7.0  
sc 0  
wc 250  
hzmm 100.90  
1s 500.90  
1f 10.00  
rfd 7785.5  
th 5  
ins 100.000  
nm no ph



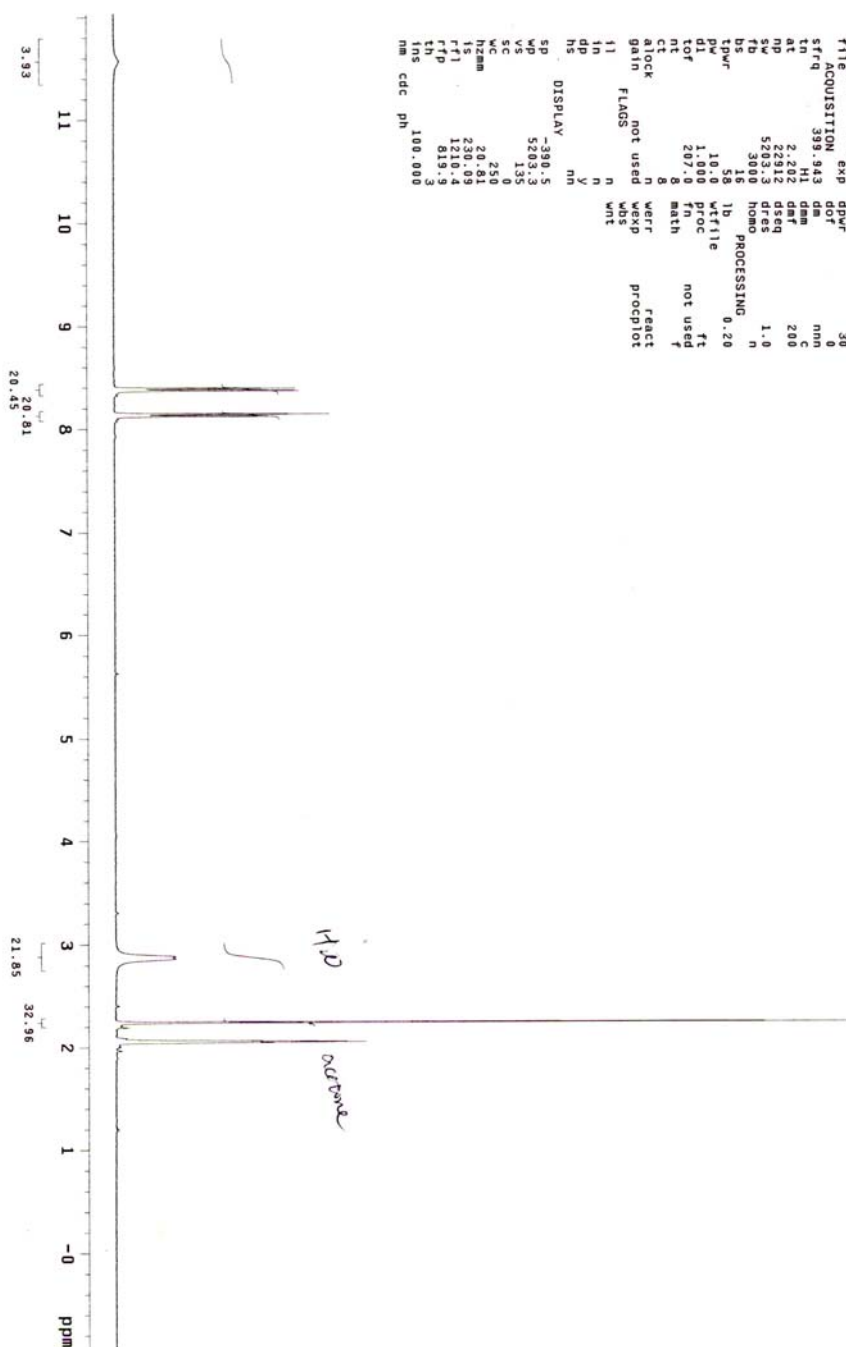


Reactant, Table 3,  
entry 8

6-80 in acetone, ying

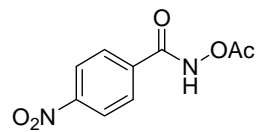
exp1 std1h

SAMPLE date 4 2003 dfreq DEC. 399.943  
 solvent acetone dn H1  
 file 110 dpr 30  
 acquisition exp dm  
 sfreq 399.943  
 tn H1 dm 200  
 at 2.202 dmf  
 np 52912 dseq 1.0  
 pw 3000 homo  
 bs 16 PROCESSING 6.20  
 tpwr 58 1b wtfile  
 dl 10.0 proc ft  
 nt 207.8 math not used f  
 ct alock n weff react  
 gain not used weff procpilot  
 flags n n  
 in n y  
 dp n  
 hs n  
 DISPLAY nm  
 SD -390.5  
 WP 5293.3  
 VS 135  
 SC 0  
 WC 250  
 HSC 250  
 15mm 230.09  
 rfi 1210.4  
 rfp 819.9  
 th 100.000  
 nm cdc ph

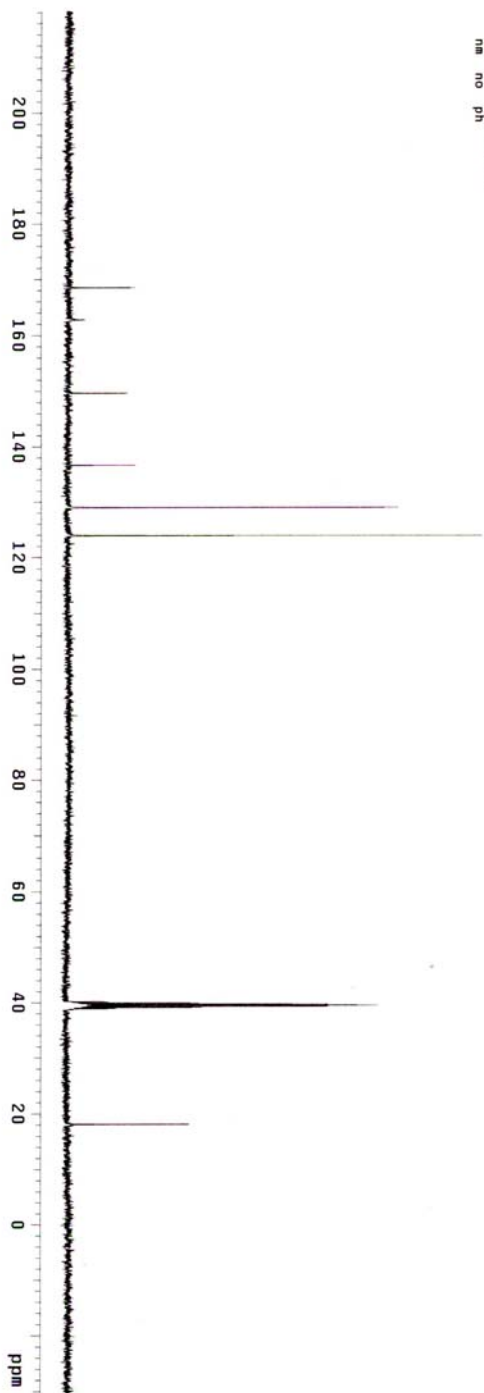


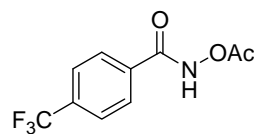
```
6-80, c13, dms0, ying
expl std13c
```

SAMPLE										DEC. 4 VI	
date	Jul 15 2003	dfrq	399.9433	HL	42						
solvent	dmsd	dn									
file											
ACQUISITION											
sfreq	100.613	exp	dpr	dot							
at	1	dm									
nu	1.139	dm									
np	55968	dreq									
sw	2500.0	dot									
fb	14000	homo									
bs											
tpwr	36	lb									
dw	8.0	stfid									
nv	3.000	white									
tof	0	proc									
nt	860	n									
ct	96	math									
gain	50										
FLAGS											
l1		n	err	react							
l1n		n	wbp	procpri							
dp		y	ent	testin							
hs		mh									
DISPLAY											
sp	-3048.2										
wp	2500.0										
vc	7.4										
sc	25.0										
h2o	3.0										
bzdm	100.00										
ls	50.0										
rfl	7021.5										
rfd	3973.3										
th	10										
ins	100.000										
am	no	ph									



Reactant, Table 3,  
entry 8

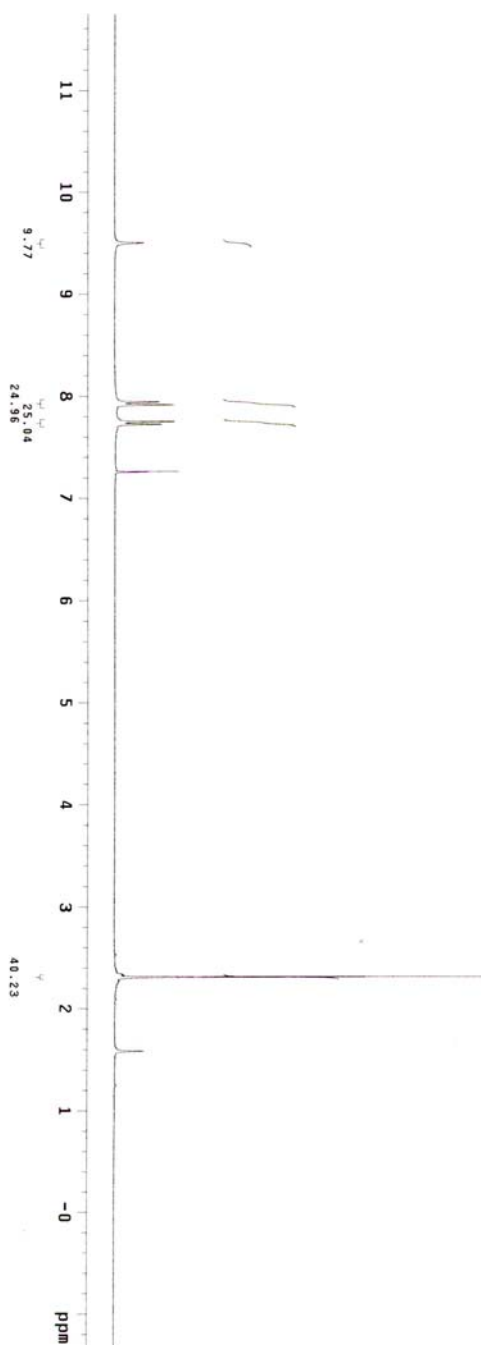


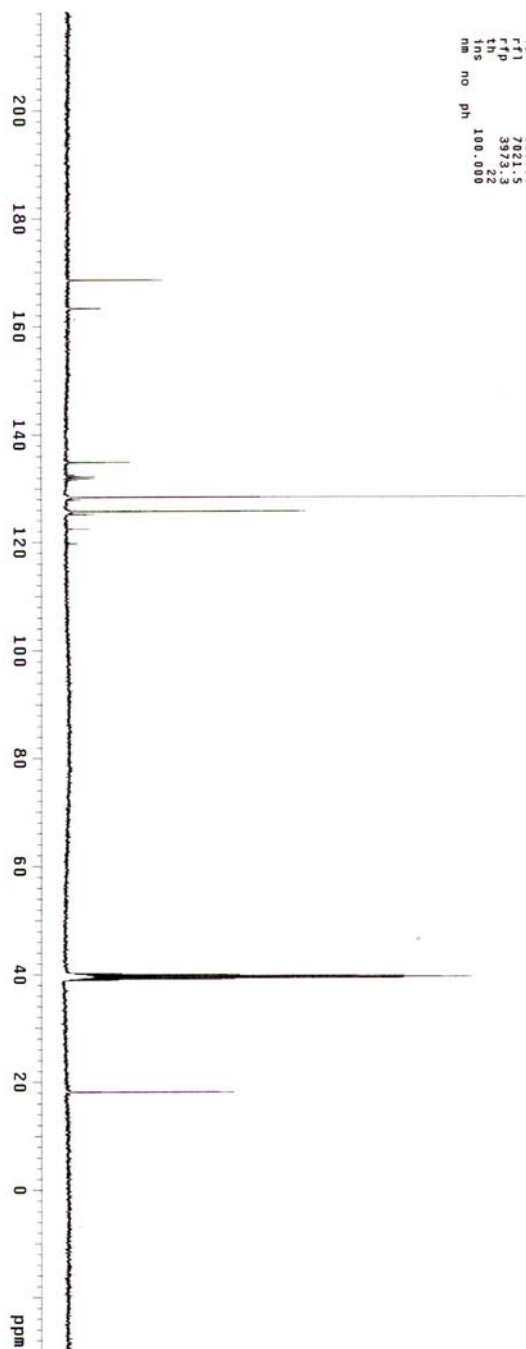


Reactant, Table 2,  
entry 8

6-96, yling  
expt szpul

SAMPLE date Jun 17 2003 temp not used  
solvent cdcl3 gain not used  
f11 250.131111 not used  
F1 ACQUISITION exp hst not used  
sw 3930.8 pw30 19.500  
at 2.500 aifa 10.000  
pd 19504 11 FLAOS  
pr 12200 11  
bs 4 in ny  
d1 2.000 dp y  
nt 8 hs  
ct TRANSMITTER H1 1b PROCESSING 0.20  
tn 300.065 f1 not used  
sfrq 65.2 sp DISPLAY  
tof 3930.8  
lpwr 3930.8  
pw 10.000 rfp 2176.5  
dn DECOUPLER C13 rfp 176.0  
dot 0 1p PLOT  
dm 250  
dmm 333  
dpr 333  
dbr 5600 vs  
dbr 14  
at cdc ph

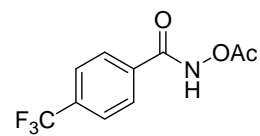




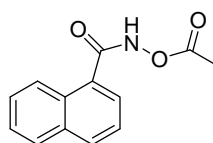
```

6-96, c13, dmsd
expt 1d13c
SAMPLE
date Jul 15 2003 DEC. 8 VT
solvent dmsd dn 399.943
file exp dpr 42
ACQUISITION
sfreq 100.575 dm yyy
nu1 11.14 dm
nu2 1.193 dmf
sw 59958 dseq 7200
fb 25000.0 dres 1.0
bs 14000 homo n
pc 5.3 1b PROCESSING 2.00
pw 8.0 1sfid 4
pr 3.000 wfile 0
di 0 proc 0
tof 800 fn 0
nt 500 match 0
a1ock n not used
gain 50 wep f
flags n react
nbs wbs procplot
hs n testis
DISPLAY
sp -3048.2
wp 25000.0
vc 0
sc 0
wc 250
hzam 100.00
fs 500.00
ftf 3973.3
th 22
ins 100.000
nm no ph

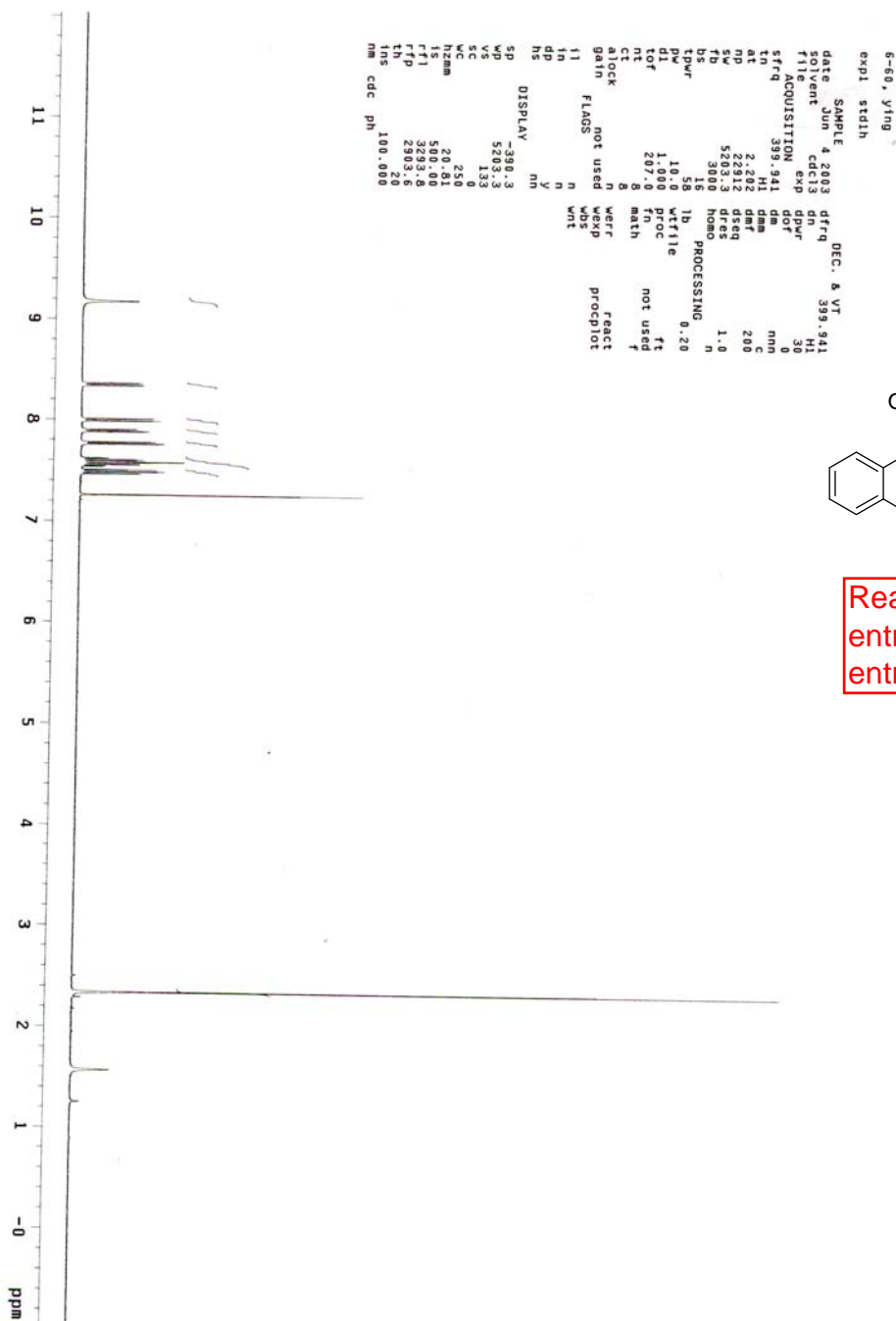
```

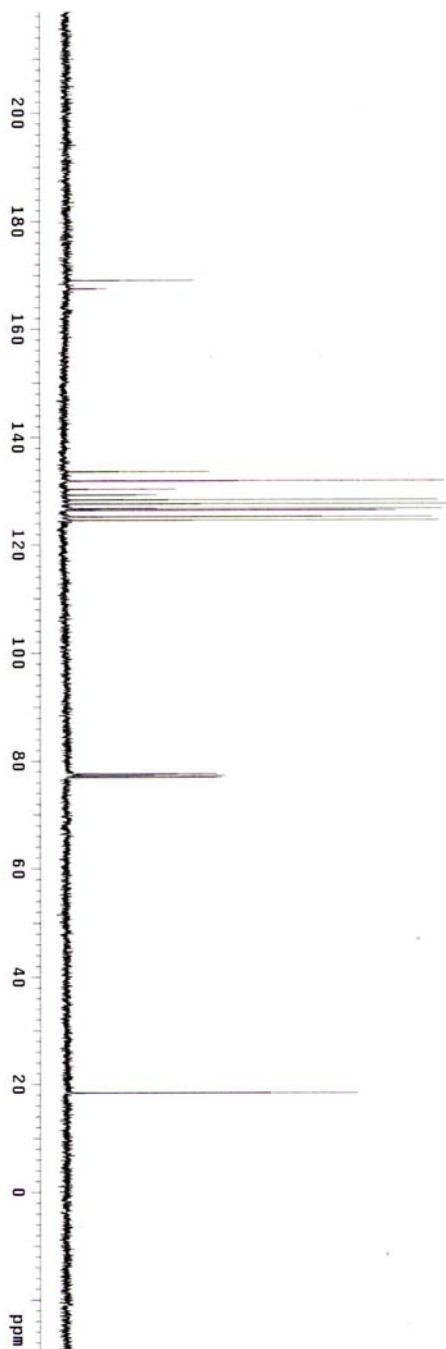


Reactant, Table 2,  
entry 8

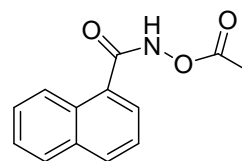


Reactant, Table 2,  
entry 10, Table 3,  
entry 10.





6-60, C13, y1ng  
 exp1 std13c  
 SAMPLE DEC. 8 VT  
 date Jul 16 2003 dfrq 399.941  
 solvent CDCl3 d1 42  
 file exp dof -200.0  
 ACQUISITION exp dof -200.0  
 sfrq 100.574 dm yyy  
 tn C13 dm v  
 1 100.574 7200  
 np 59968 dseq 1.0  
 sw 25000.0 dres n  
 tb 14000 homo  
 bs 16 PROCESSING 2.00  
 dpr 50 1b 4  
 pwr 8.00 1sfid 4  
 d1 3.000 wfile  
 nt 0 proc ft  
 ct 800 fn not used  
 clock 64 math f  
 gain 50 weff react  
 11 FLAGS n wep procplot  
 1n n wds testin  
 1p y  
 1s nm  
 DISPLAY  
 sp -2995.0  
 wp 25000.0  
 sc 71  
 wc 250  
 hzem 100.00  
 15 500.00  
 ffl 10761.6  
 th 7706.8  
 ins 100.000  
 nm no ph

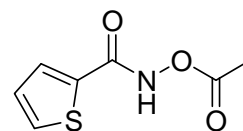
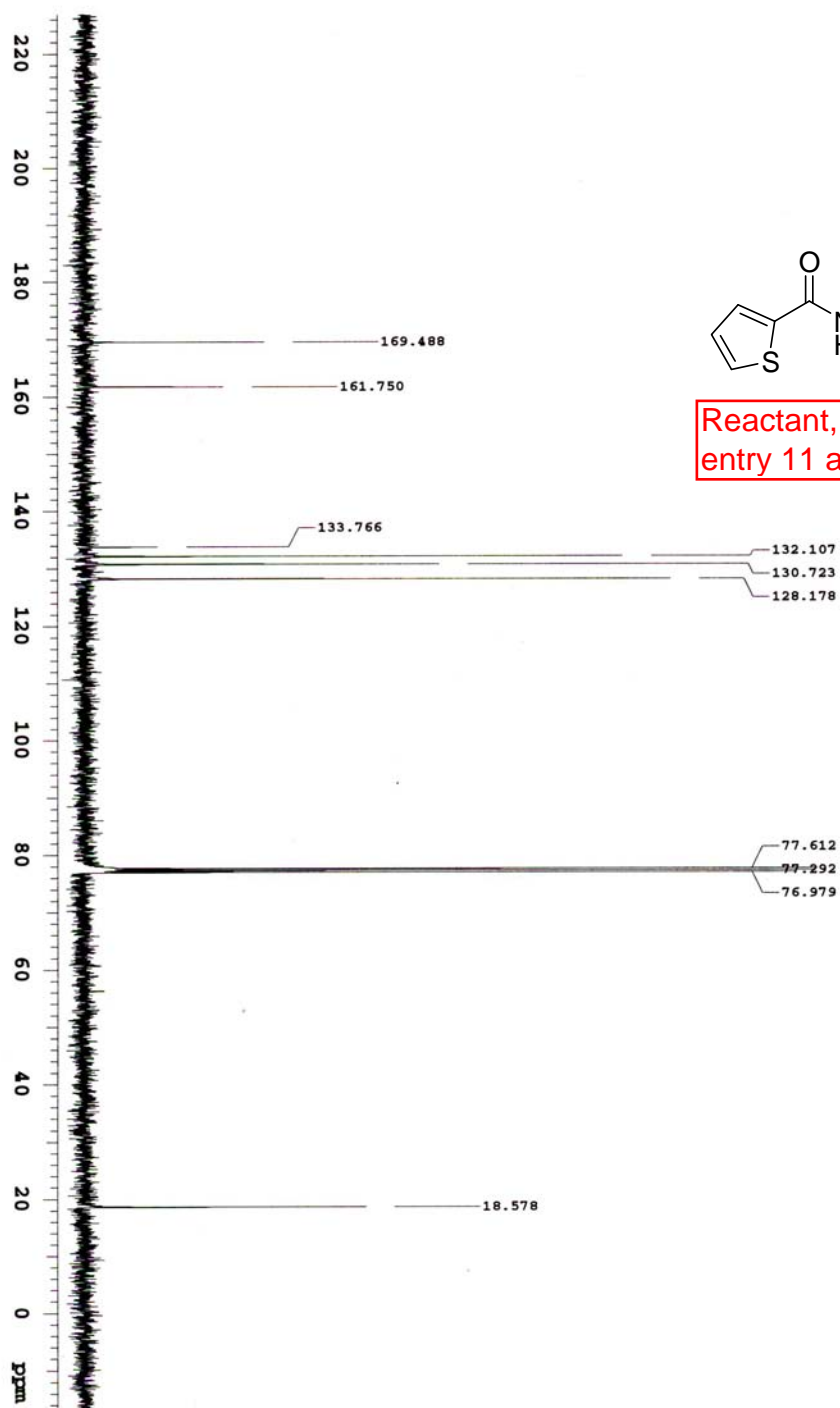


Reactant, Table 2,  
 entry 10, Table 3,  
 entry 10.

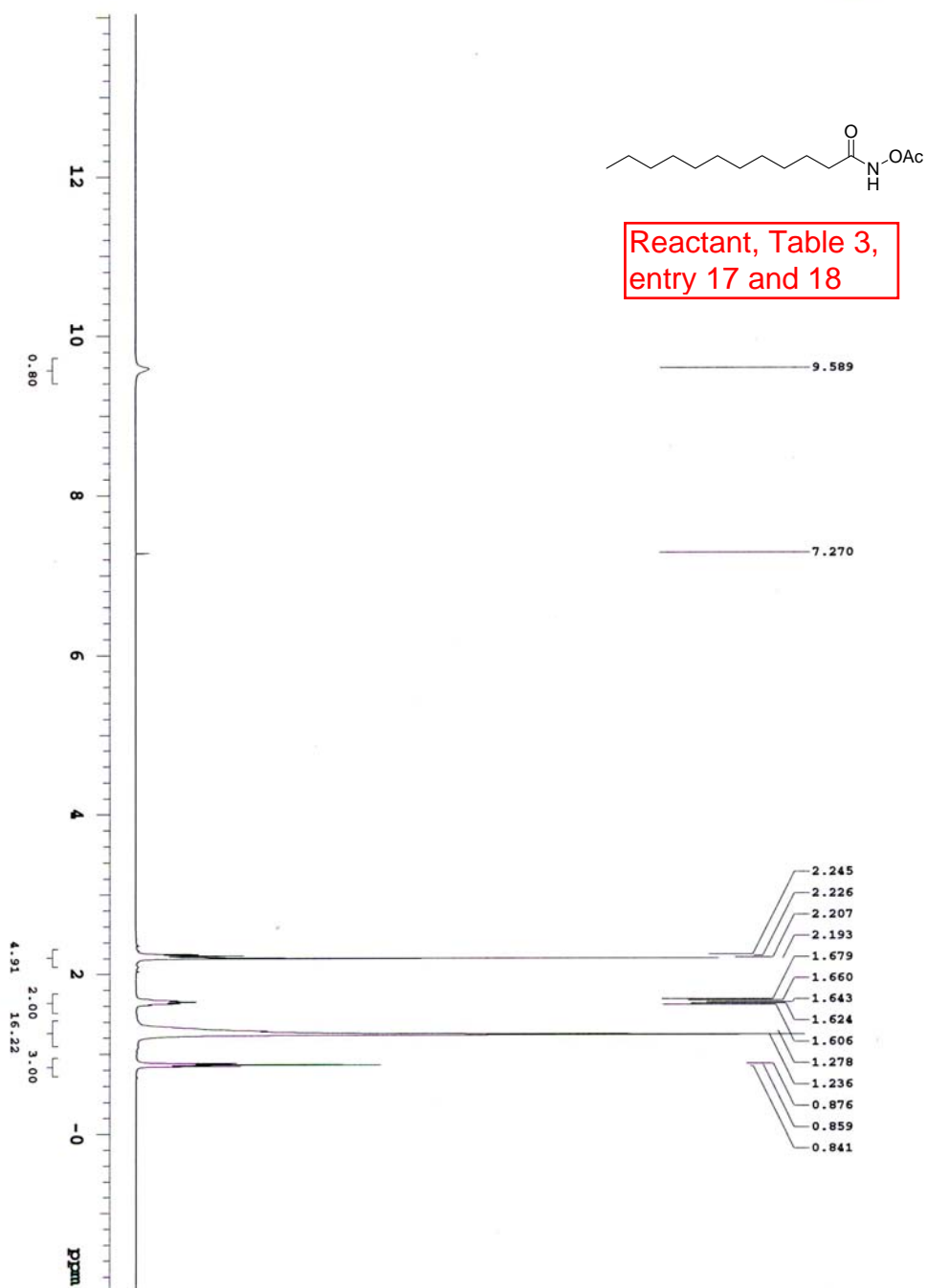


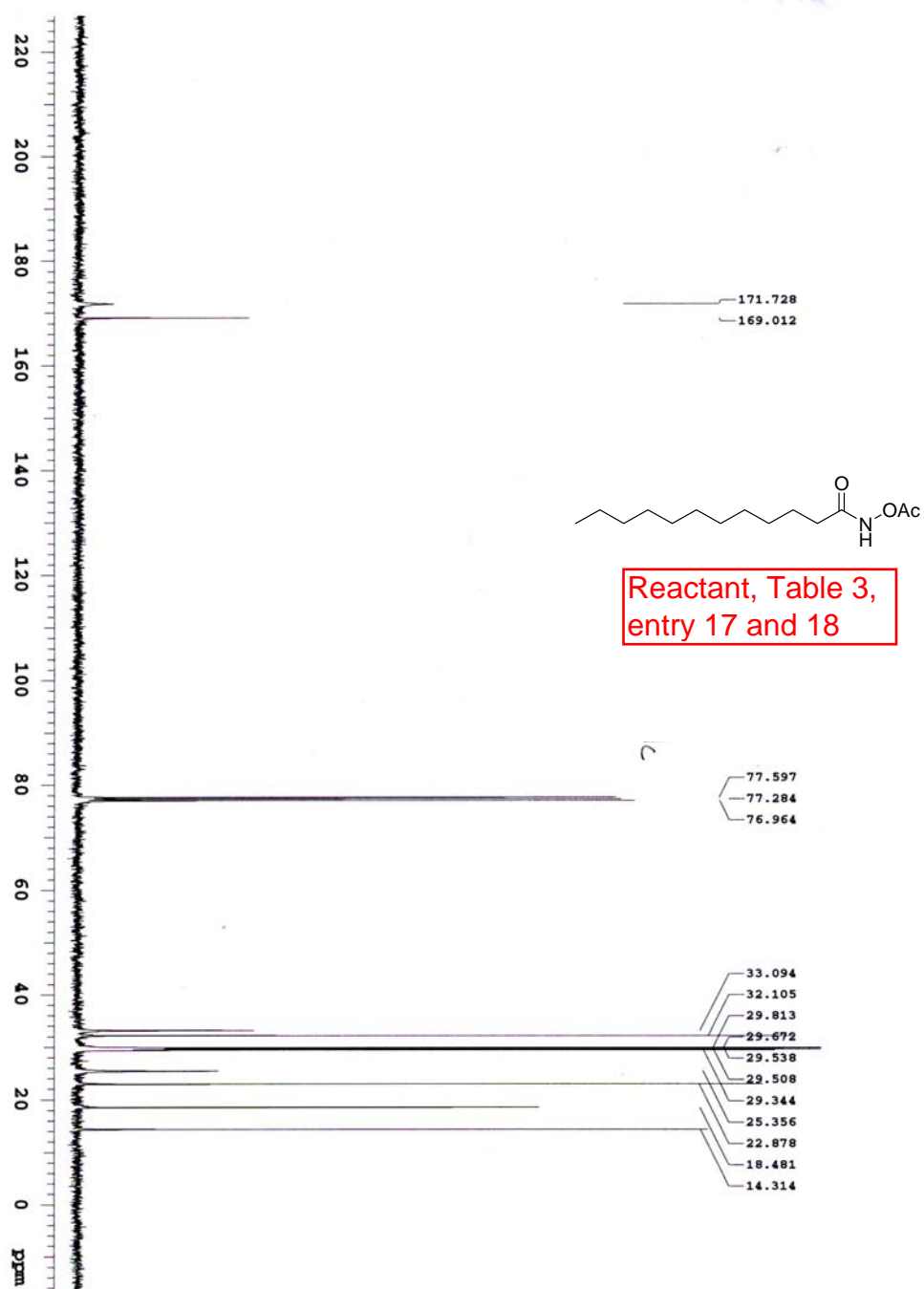
Reactant, Table 3,  
entry 11 and 12

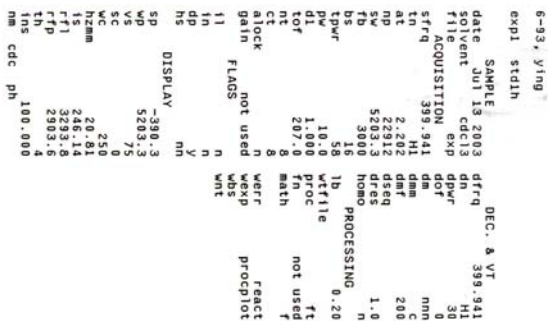
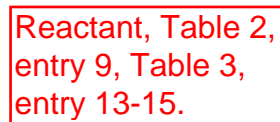


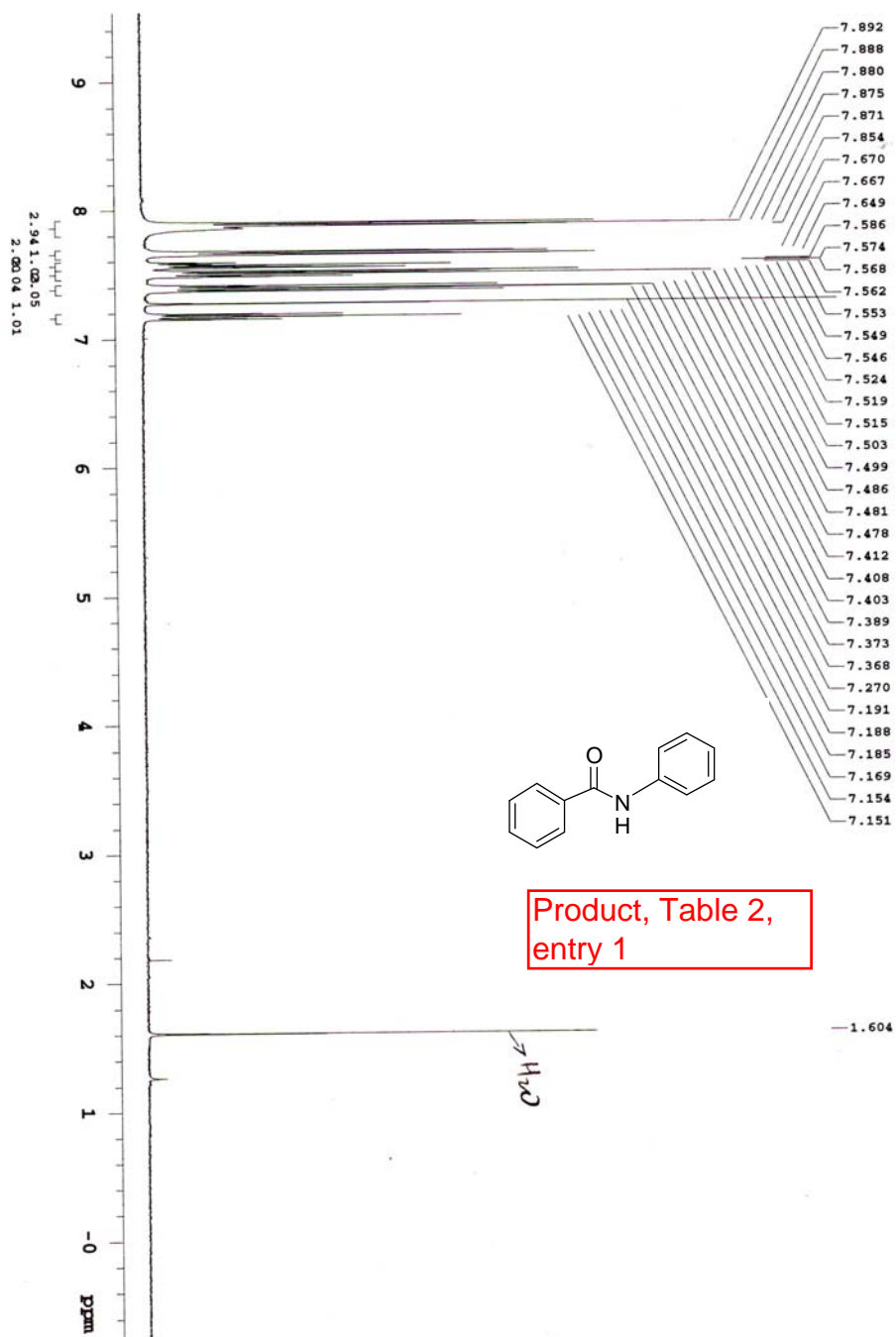


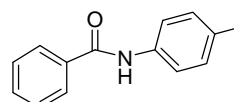
Reactant, Table 3,  
entry 11 and 12







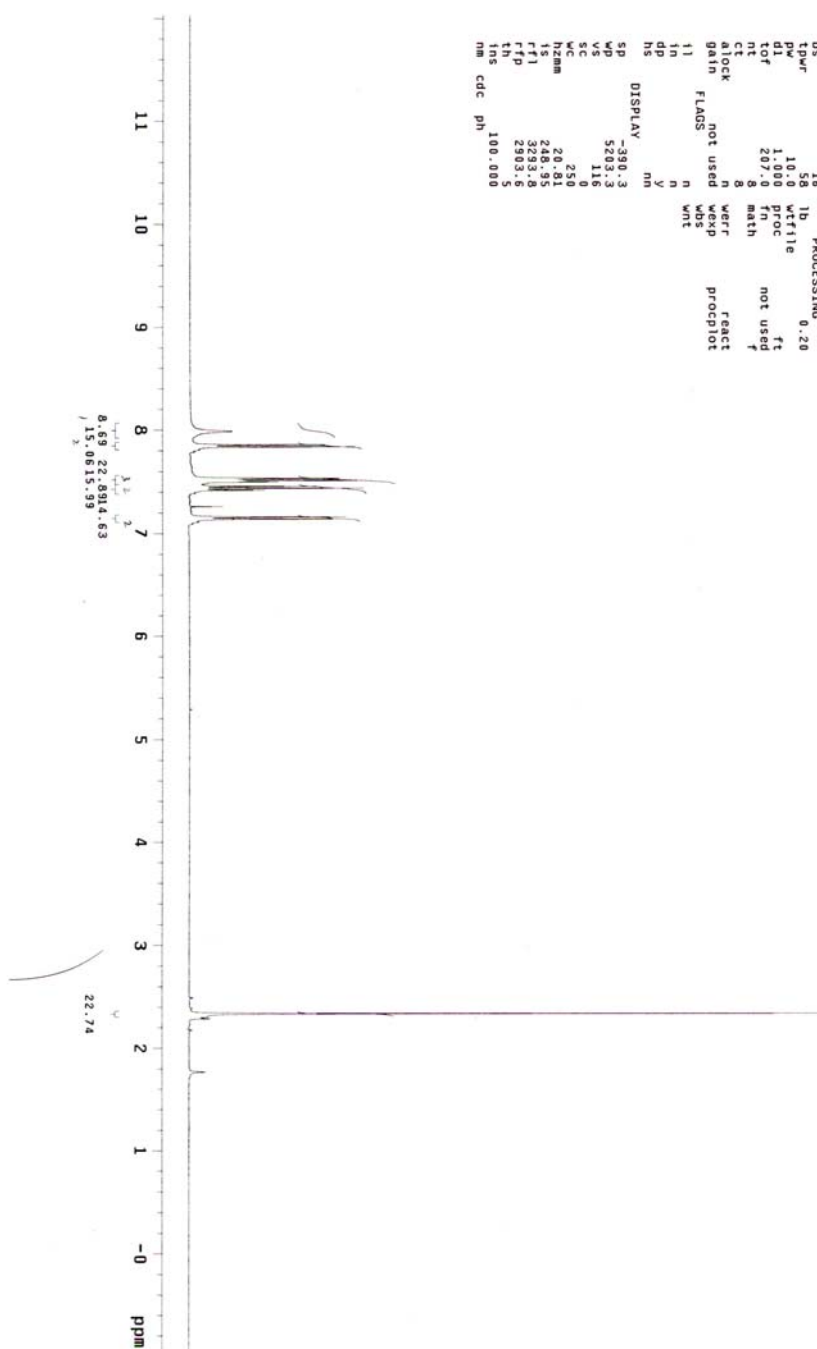


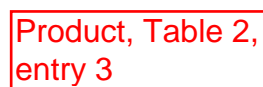


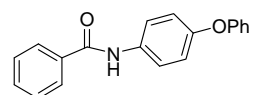
Product, Table 1,  
all entries; Table 2,  
entry, 2, Table 3,  
entry 1

6-5, ying  
expl stdth  
SAMPLE  
date Jul 27 2003  
solvent d1  
file cdc13  
ACQUISITION  
exp dpr  
sfreq 399.941  
at 2.202  
np 22912  
sw 5203.3  
db 3000  
tprf 58  
pw 10.0  
d1 1.000  
tof 207.0  
ct 8  
clock not used  
gain n  
wnt n  
11 n  
11 n  
dp n  
hs n  
DISPLAY  
sp -380.3  
vp 5203.3  
wc 1.0  
sc 0  
hzm 20.81  
f1 320.95  
rfd 2903.6  
th 5  
ins 100.000  
nm cdc ph

DEC. & VT  
399.941  
HI  
38  
0  
nm  
206  
1.0  
n  
PROCESSING  
0.20  
lb  
wf file  
proc ft  
fn not used  
math f  
react  
wexp  
procplot





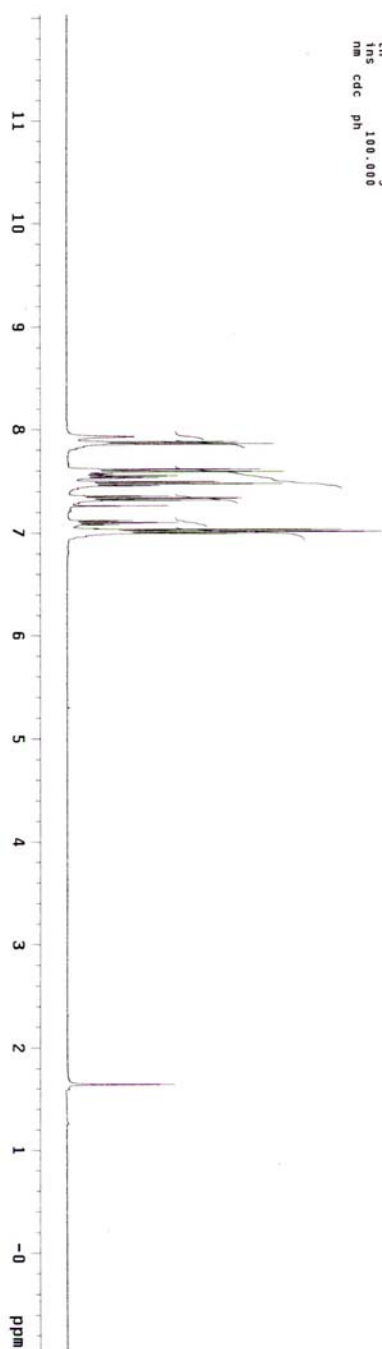


Product, Table 2,  
entry 4

6-26, ying  
expt stdth

SAMPLE DEC. & VT  
date Jul 27 2003 dfrq 399.941  
solvent d<sub>2</sub>cld<sub>3</sub> d<sub>2</sub>o 30  
file exp d<sub>2</sub>o 30  
ACQUISITION  
sfrq 399.941 dm nm  
tn 2.20 d<sub>2</sub>o 200  
np 22912 dseq 1.0  
sw 5203.3 dres n  
fb 3000 homo  
bs 1.6  
d<sub>2</sub>o 1.6 PROCESSING 0.20  
pwr 10.0 wfile ft  
dl 1.000 proc f  
tof 207.0 fn not used  
nt 8 math f  
ct 8  
clock 8  
gain not used  
flags not used  
wbs wexp react  
wnt wnt procplot

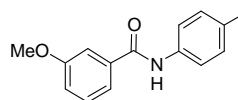
DISPLAY  
sp -390.3  
wp 5203.3  
vc 30  
wc 250  
h<sub>2</sub>mm 20.01  
ts 287.83  
ft 3203.6  
th 2903.5  
ins 100.000  
nm cdc ph



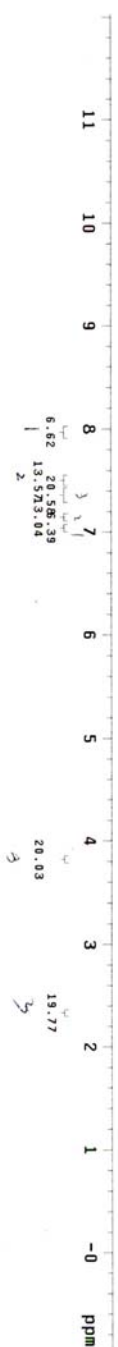


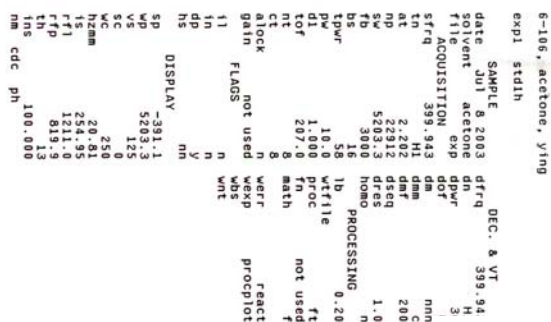
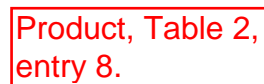
exp	stain
1	1
2	2
3	3
4	4
5	5
6	6
7	7
8	8
9	9
10	10
11	11
12	12
13	13
14	14
15	15
16	16
17	17
18	18
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96	96
97	97
98	98
99	99
100	100

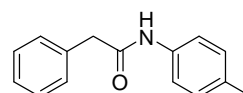
nm cdc ph



Product, Table 2, entry 7; Table 3, entry 9.

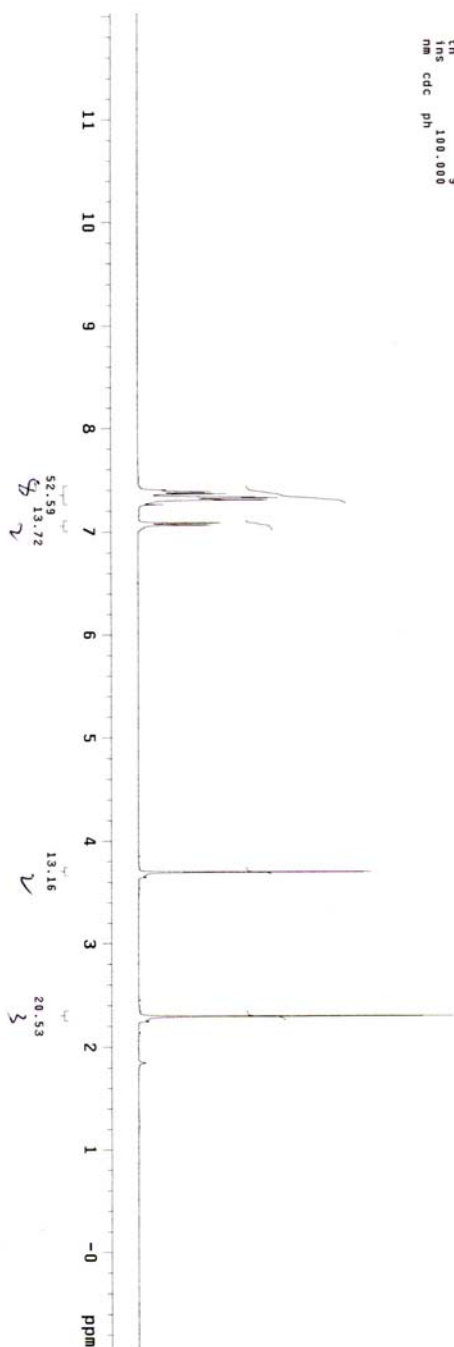


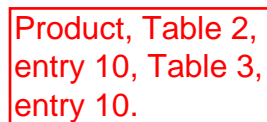


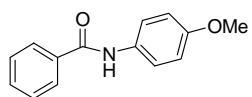


Product, Table 2,  
entry 9; Table 3,  
entry 13.

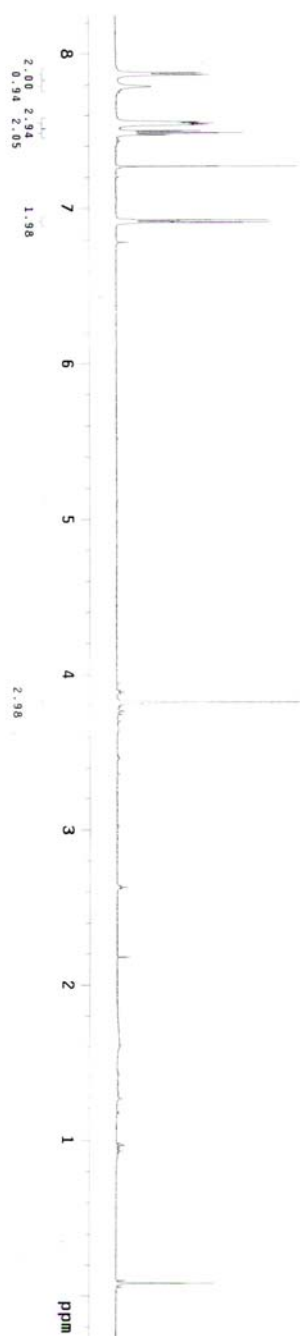
6-81, ying  
expl stdh  
SAMPLE  
date 2003 DEC. 8 VT  
solvent cdcl3 dn 399.941  
file  
ACQUISITION  
exp 30  
sfrq 399.941 dm 0  
tn 2.20 dmf 200  
np 22912 dseq 1.0  
sv 5203.3 dres  
fb 3000 homo  
bs 1b PROCESSING 0.20  
pw 10.0 wfile  
di 1.000 proc ft  
tof 207.0 fn not used  
nt 8 math f  
ct 0  
alock n  
gain not used  
wbs wep react  
procplot  
11 n  
1n n  
dn n  
hs n  
DISPLAY  
sp -390.3  
wp 5203.3  
sc 0  
wc 250  
hzmm 20.91  
1s 79.79  
1f 2003.6  
th 2003.6  
ins 100.000  
nm cdc ph

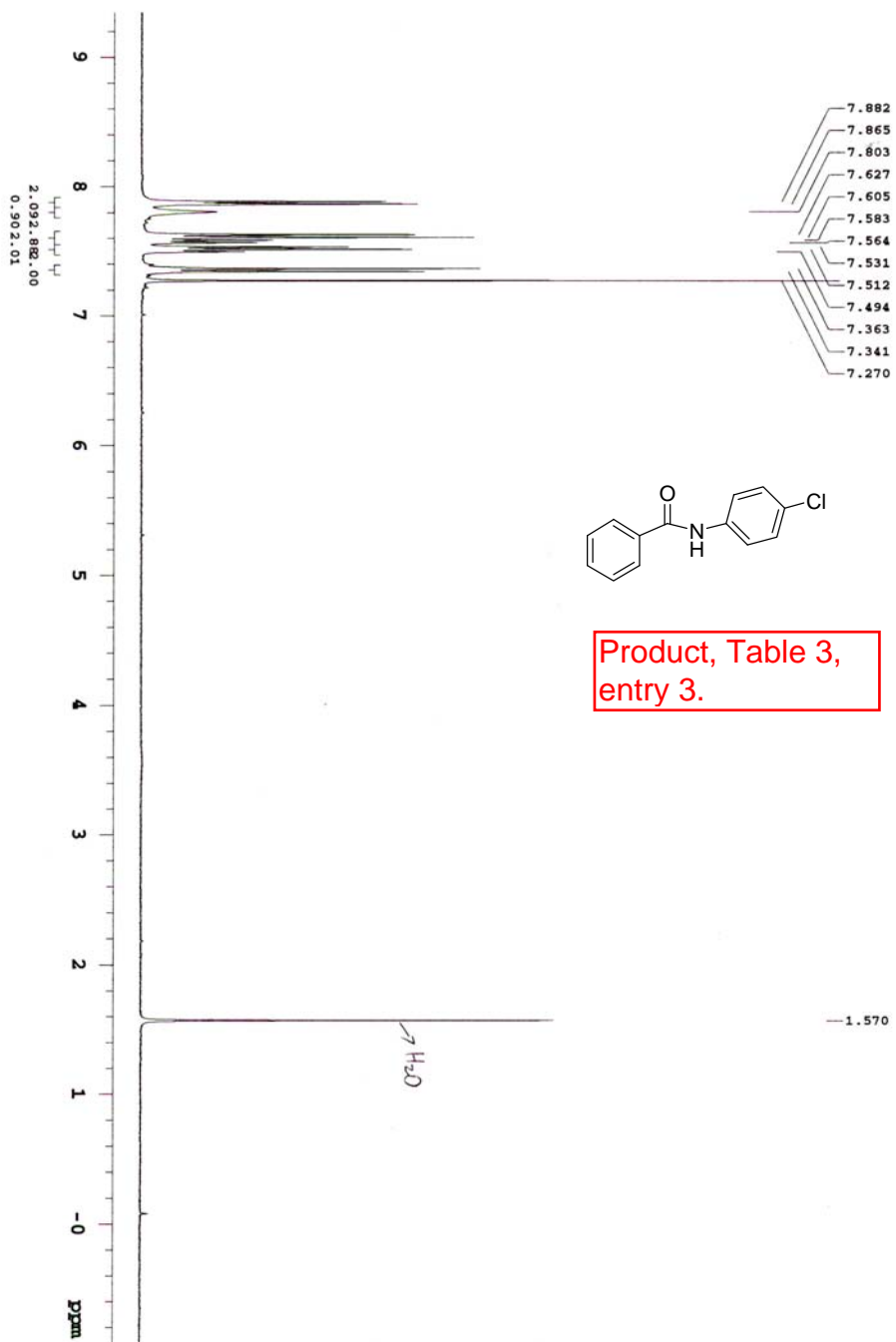


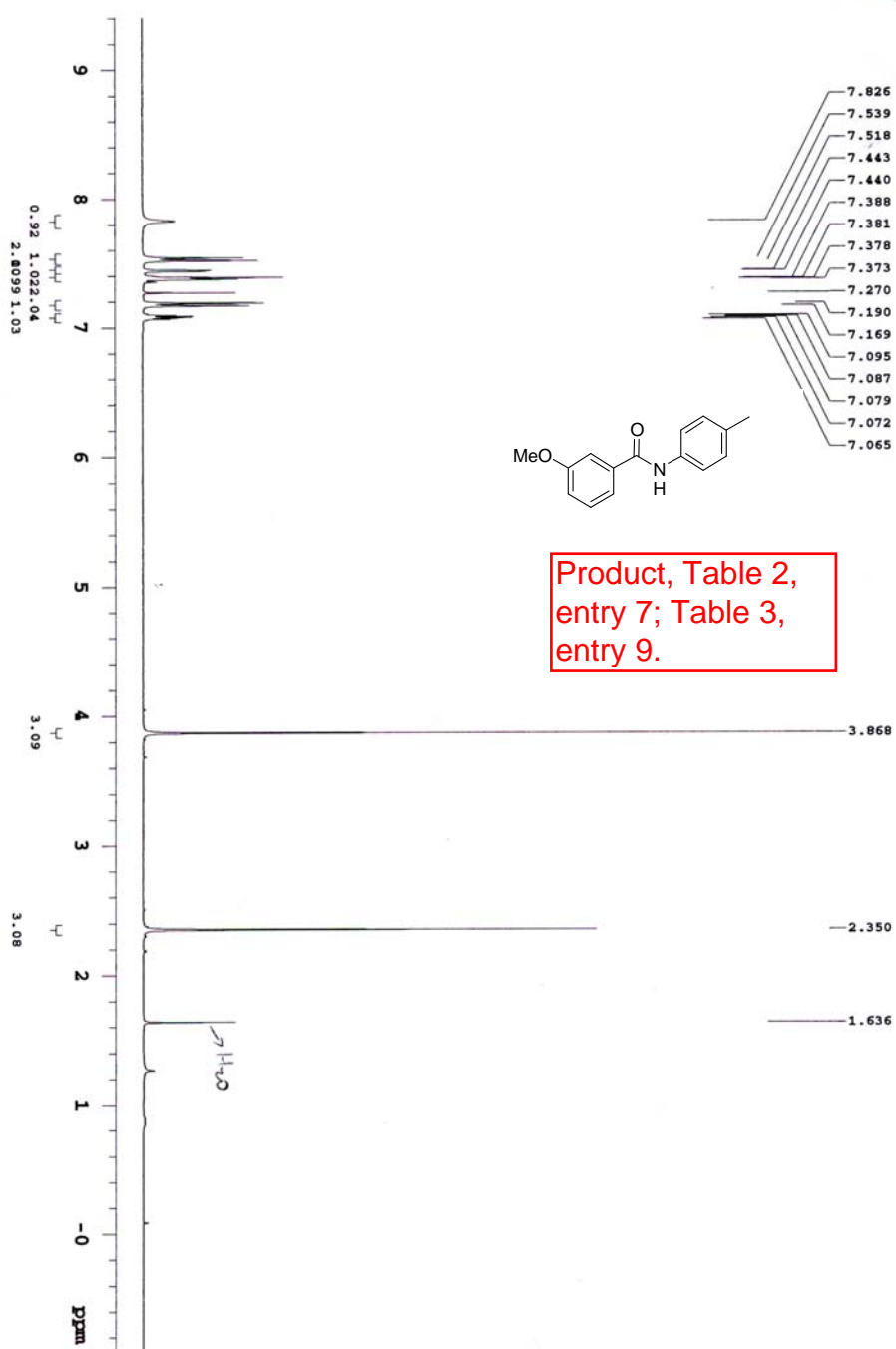




Product, Table 3,  
entry 2.



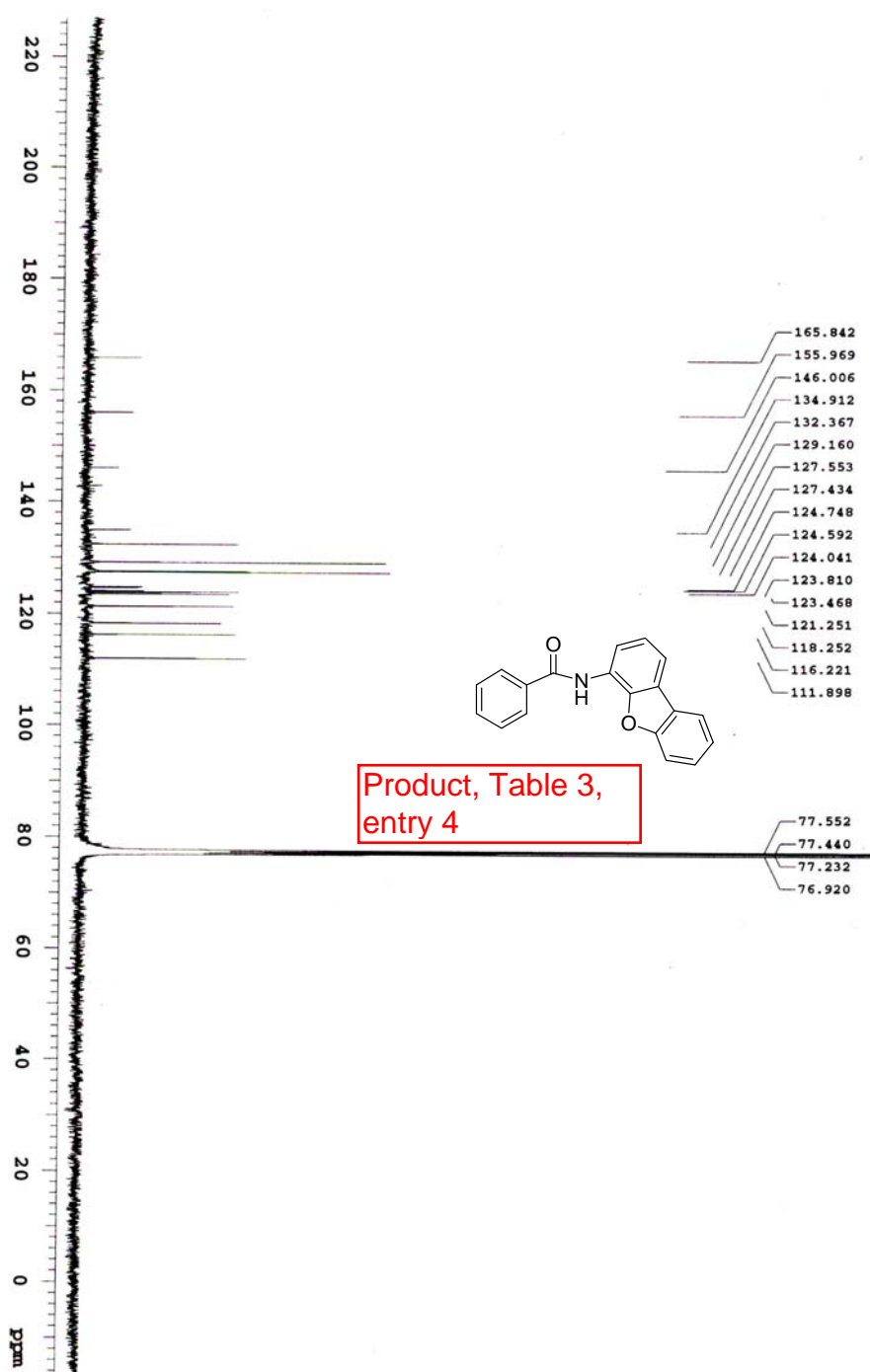


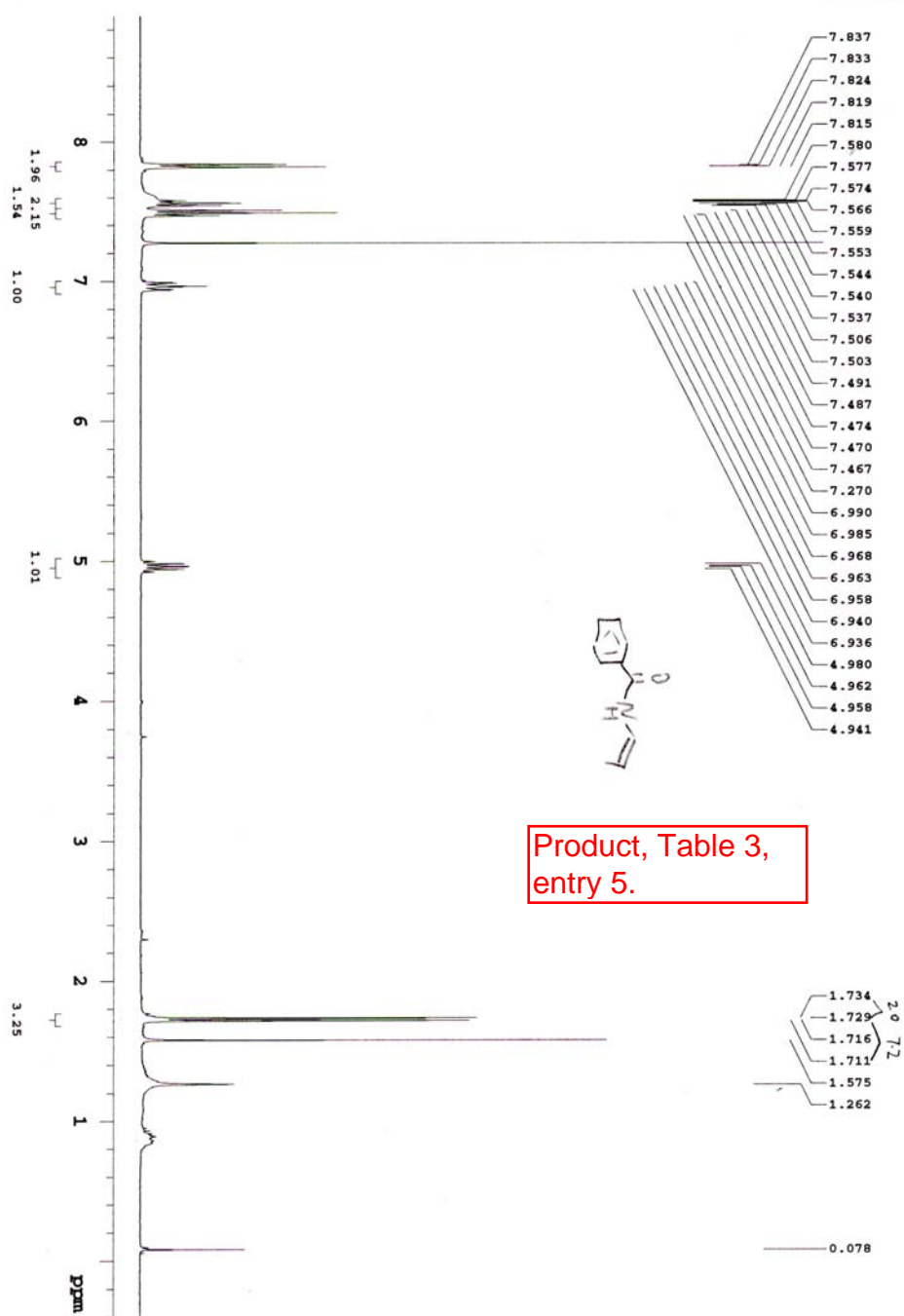


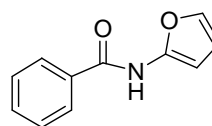
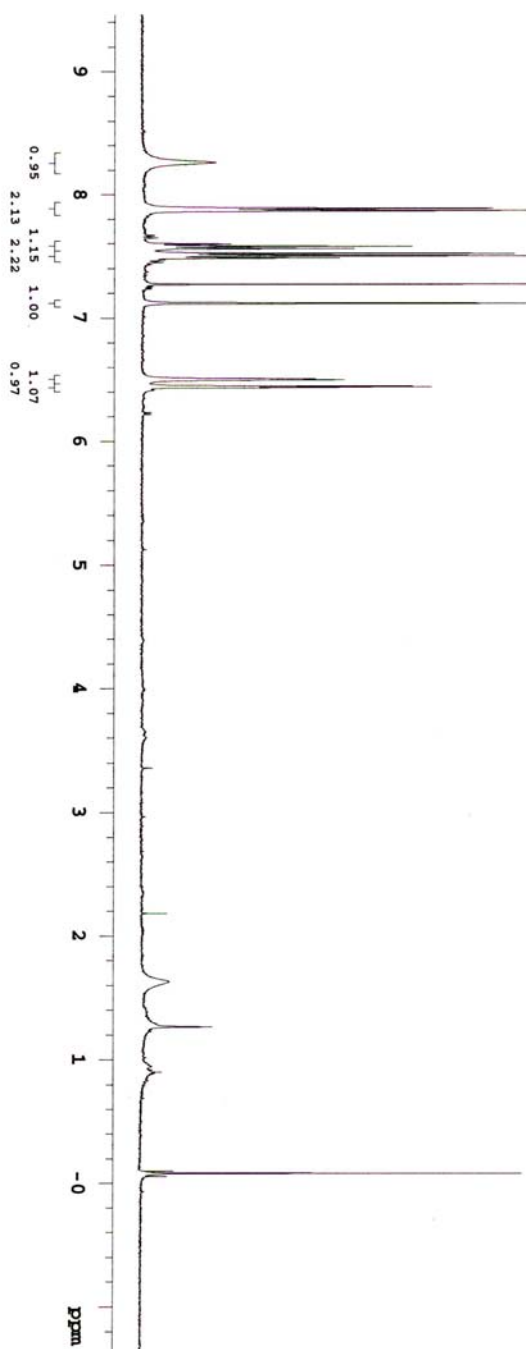
Product, Table 2,  
entry 7; Table 3,  
entry 9.



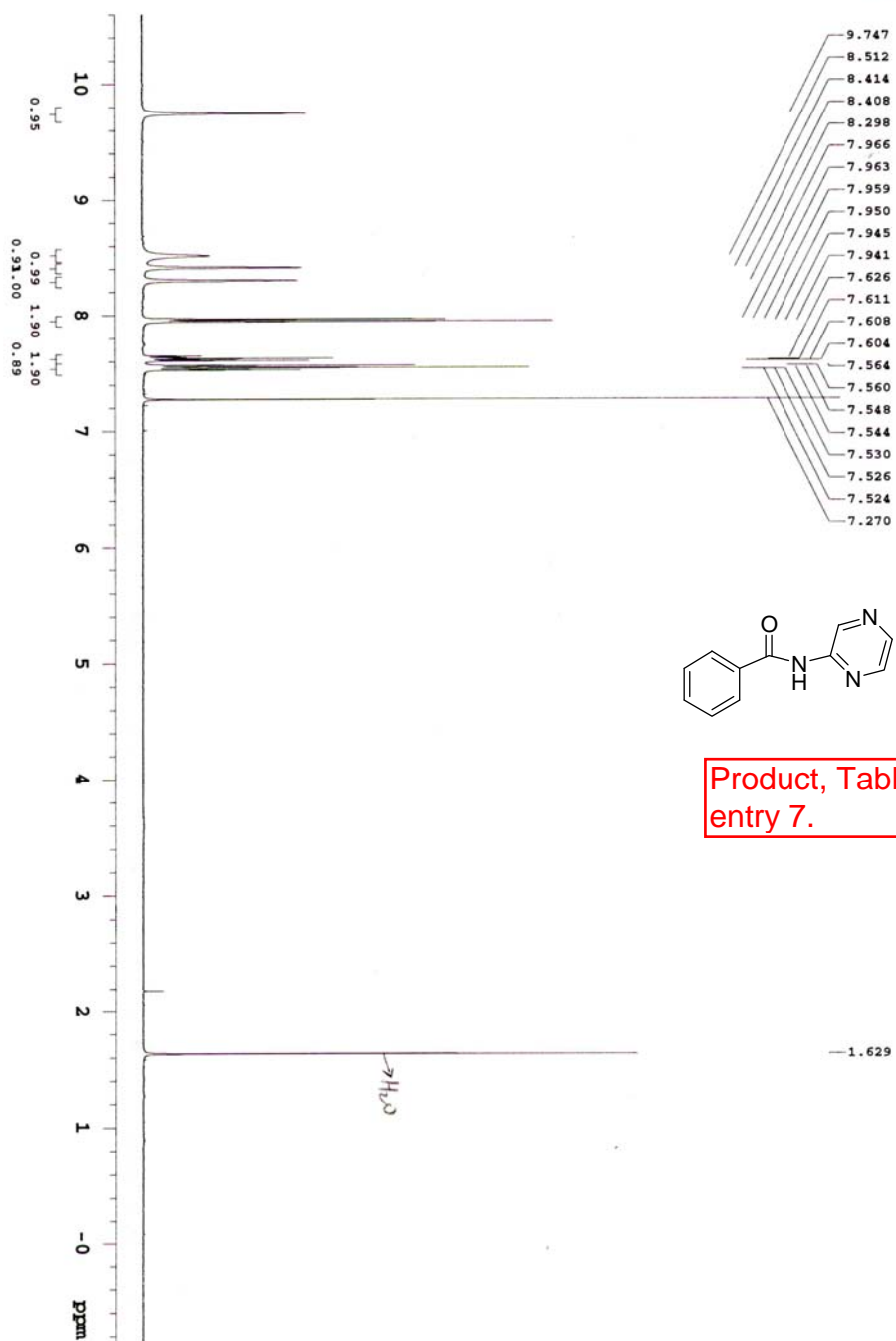




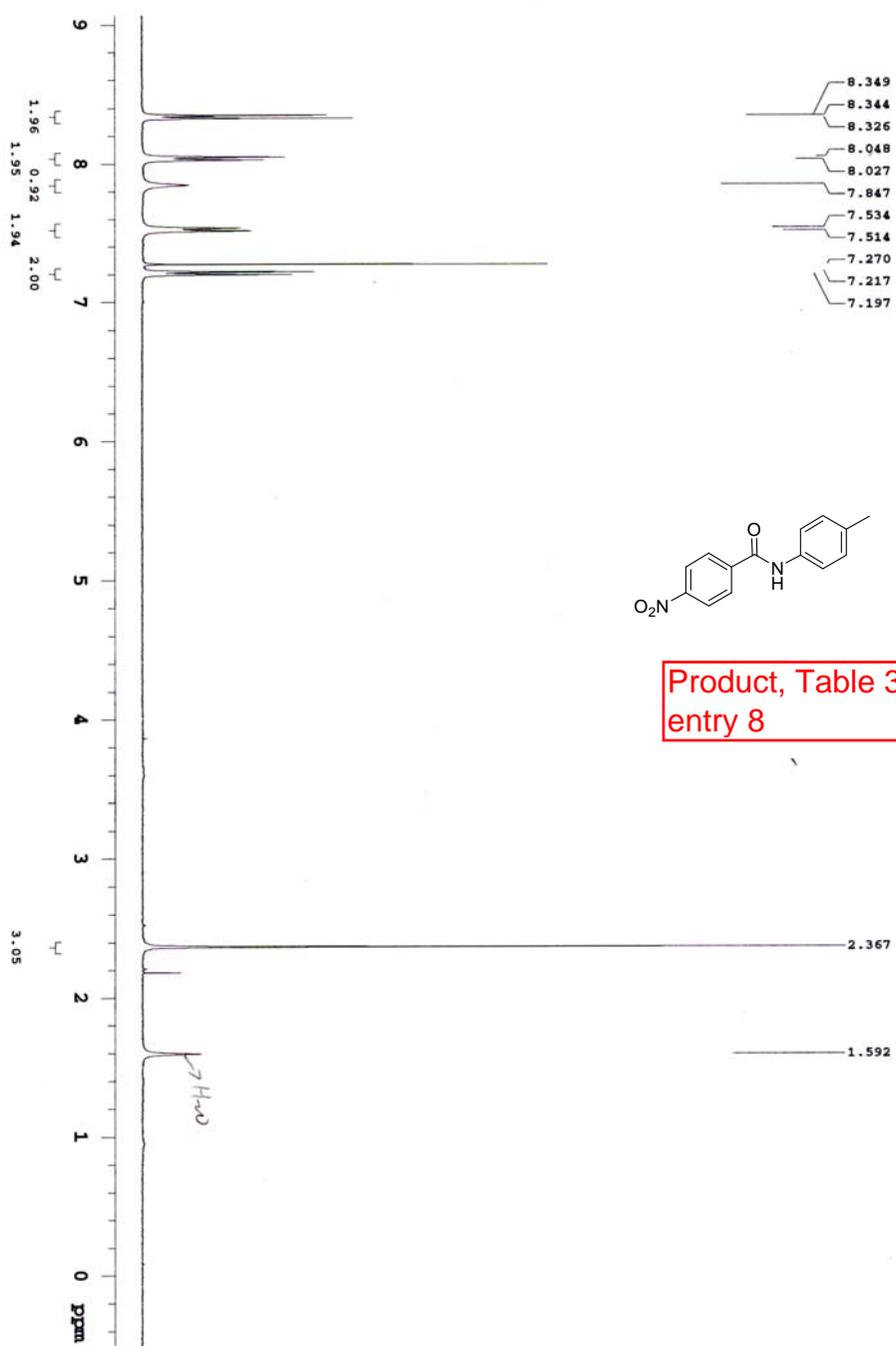




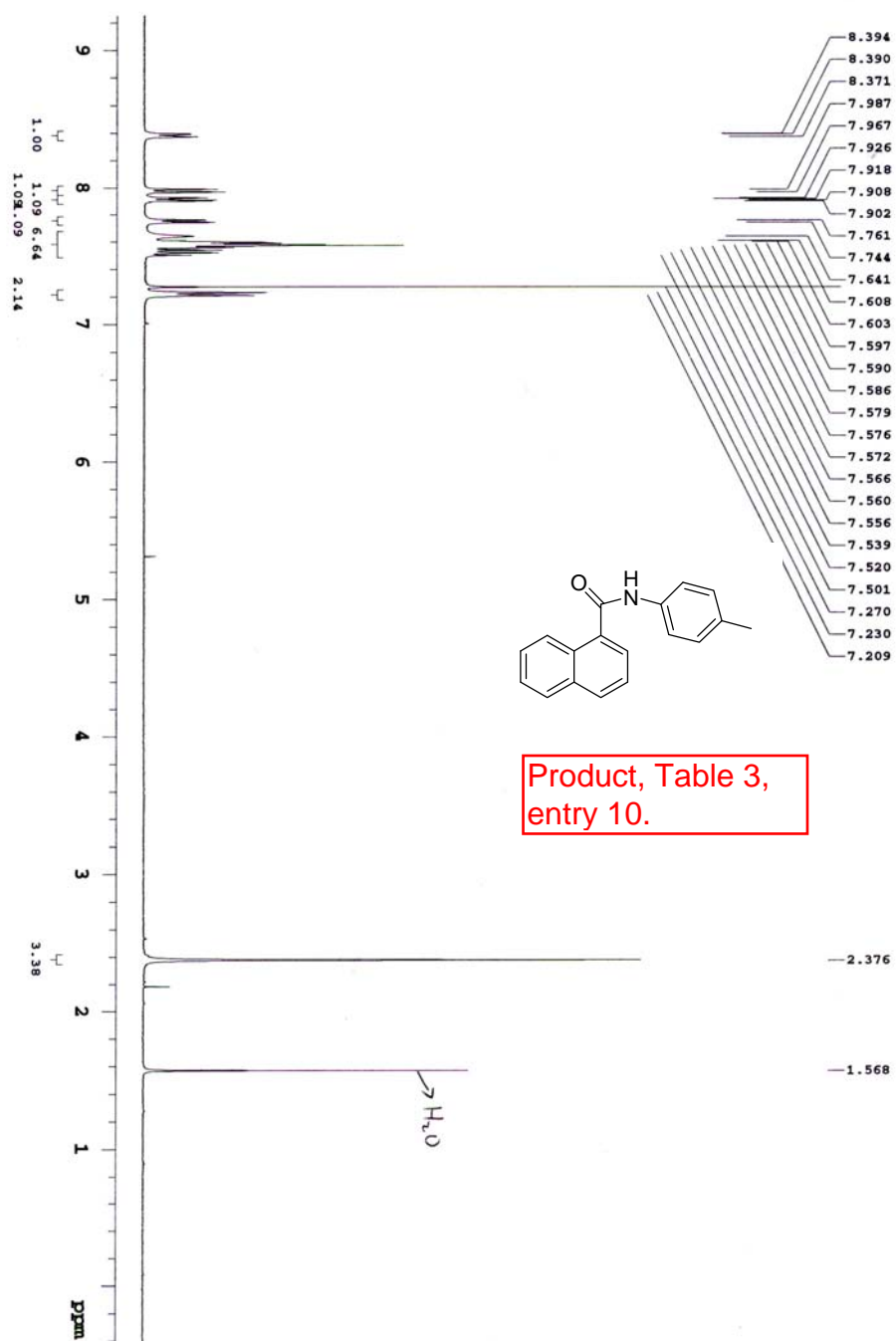
Product, Table 3,  
entry 6.



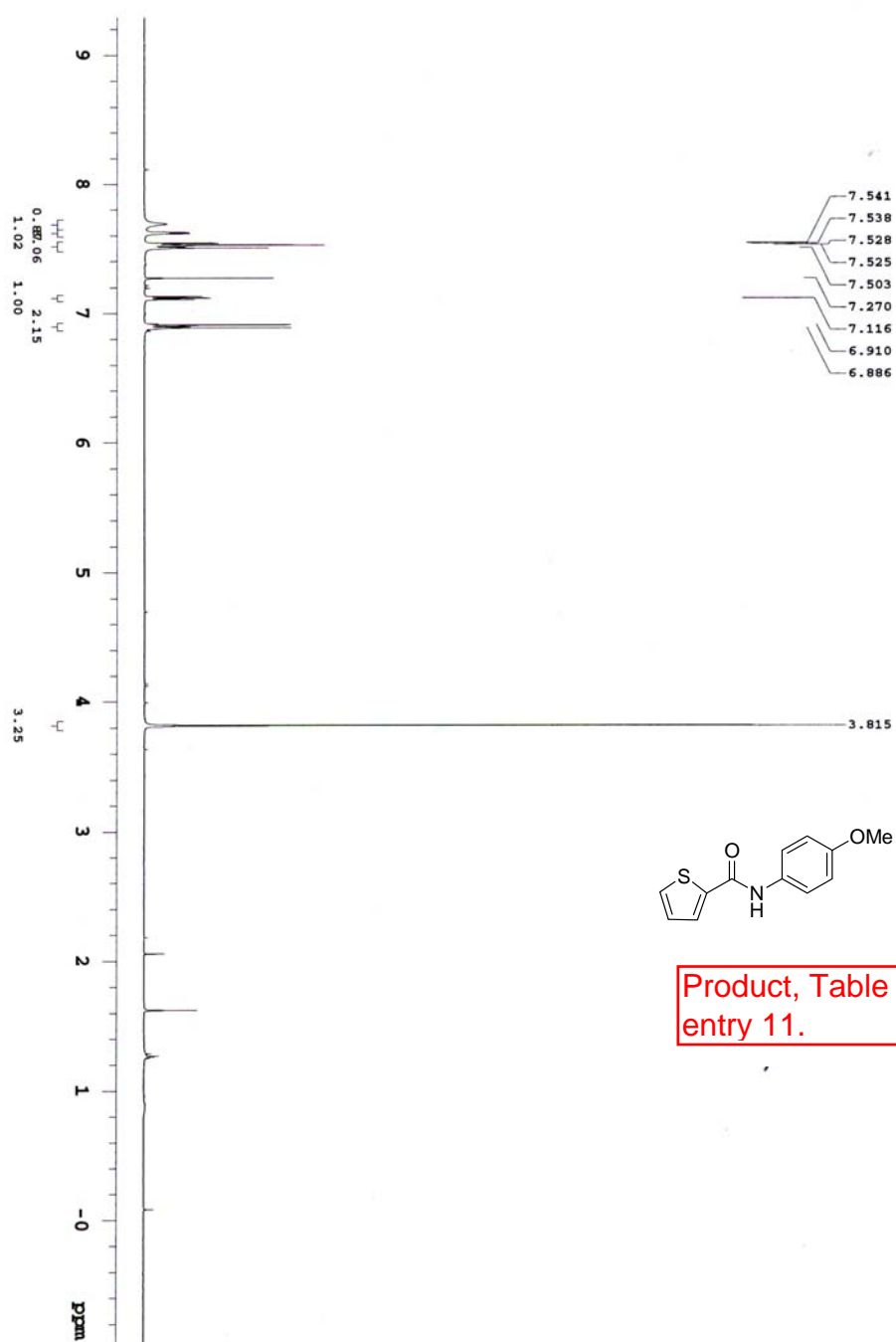
Product, Table 3,  
entry 7.



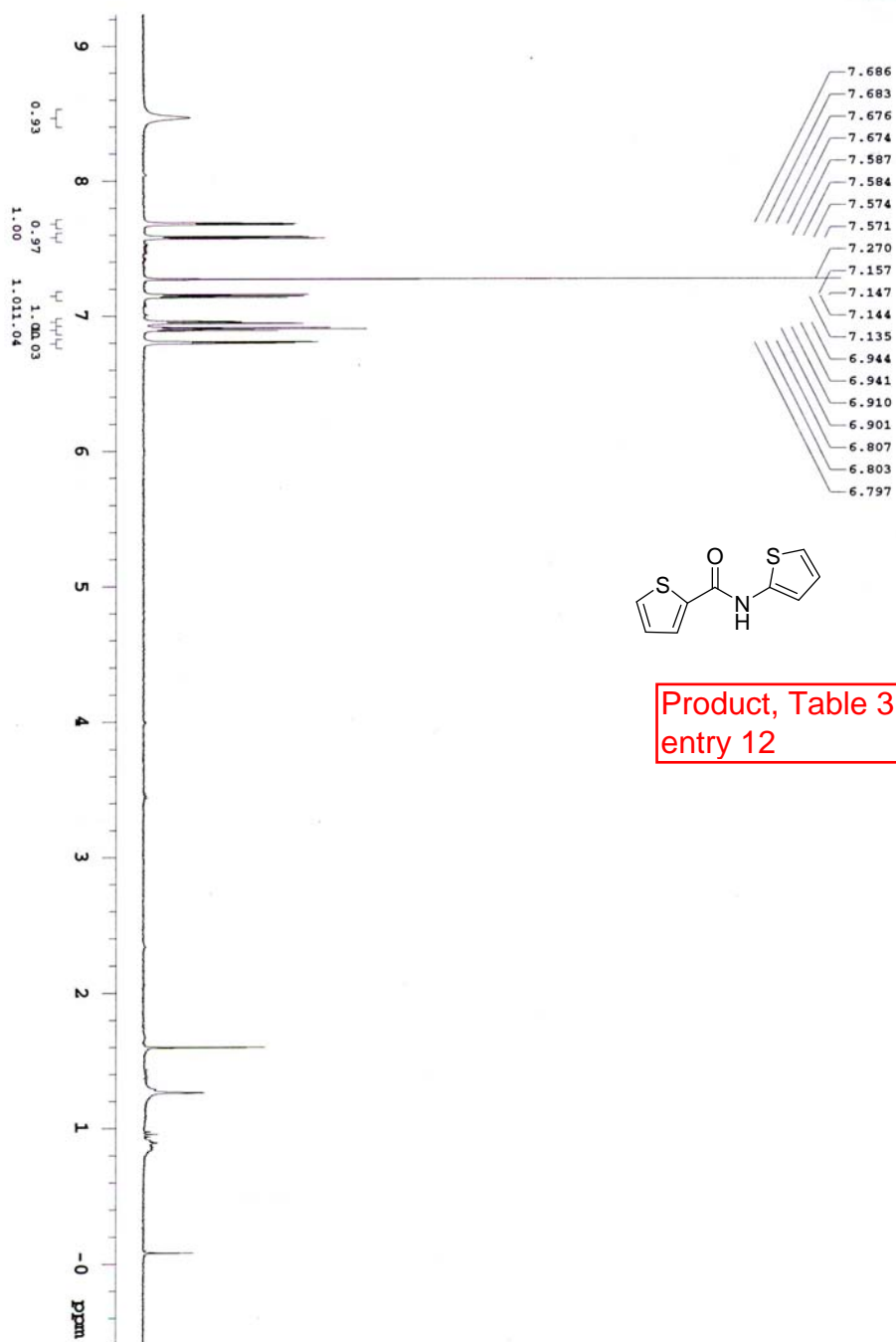
Product, Table 3,  
entry 8



Product, Table 3,  
entry 10.

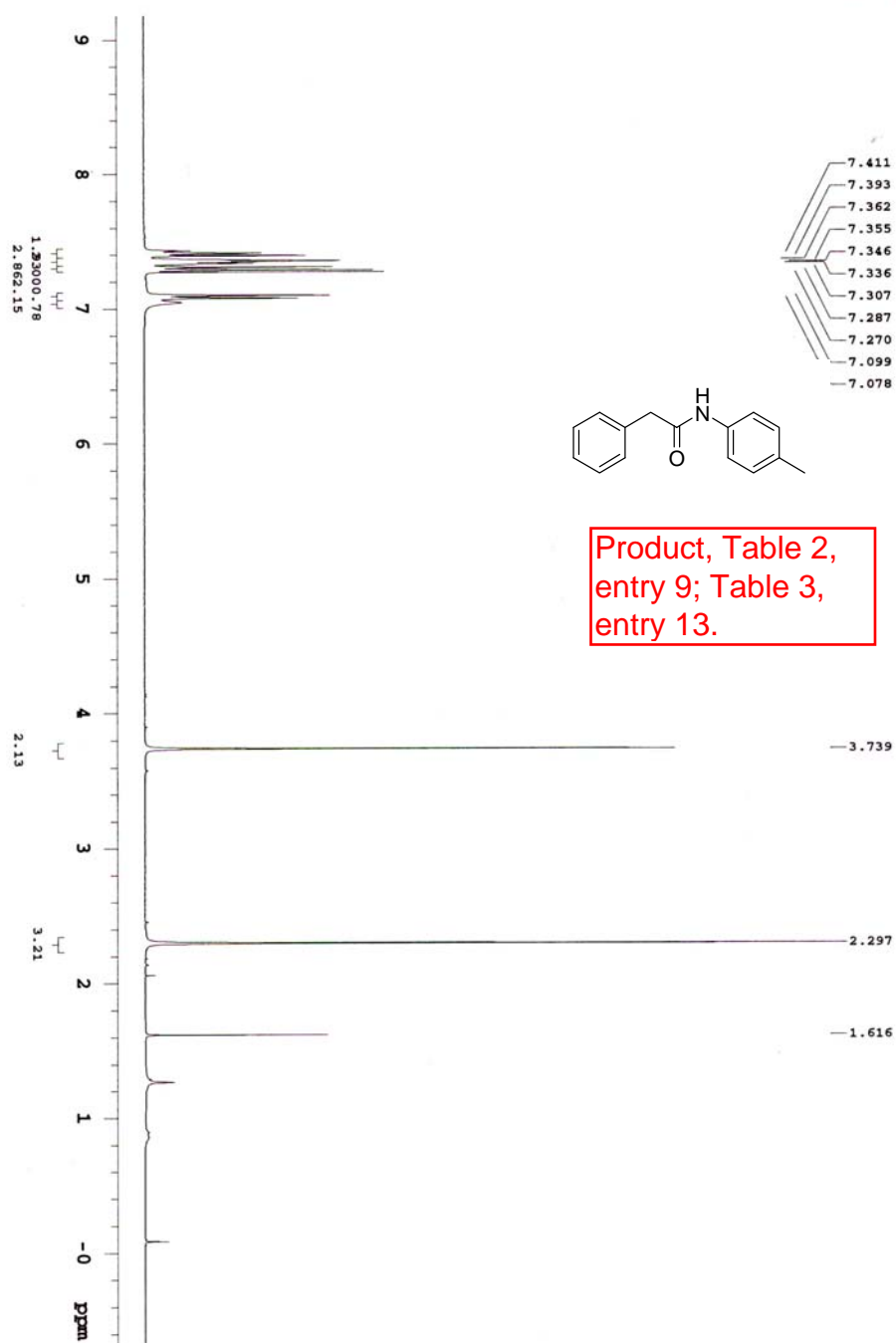


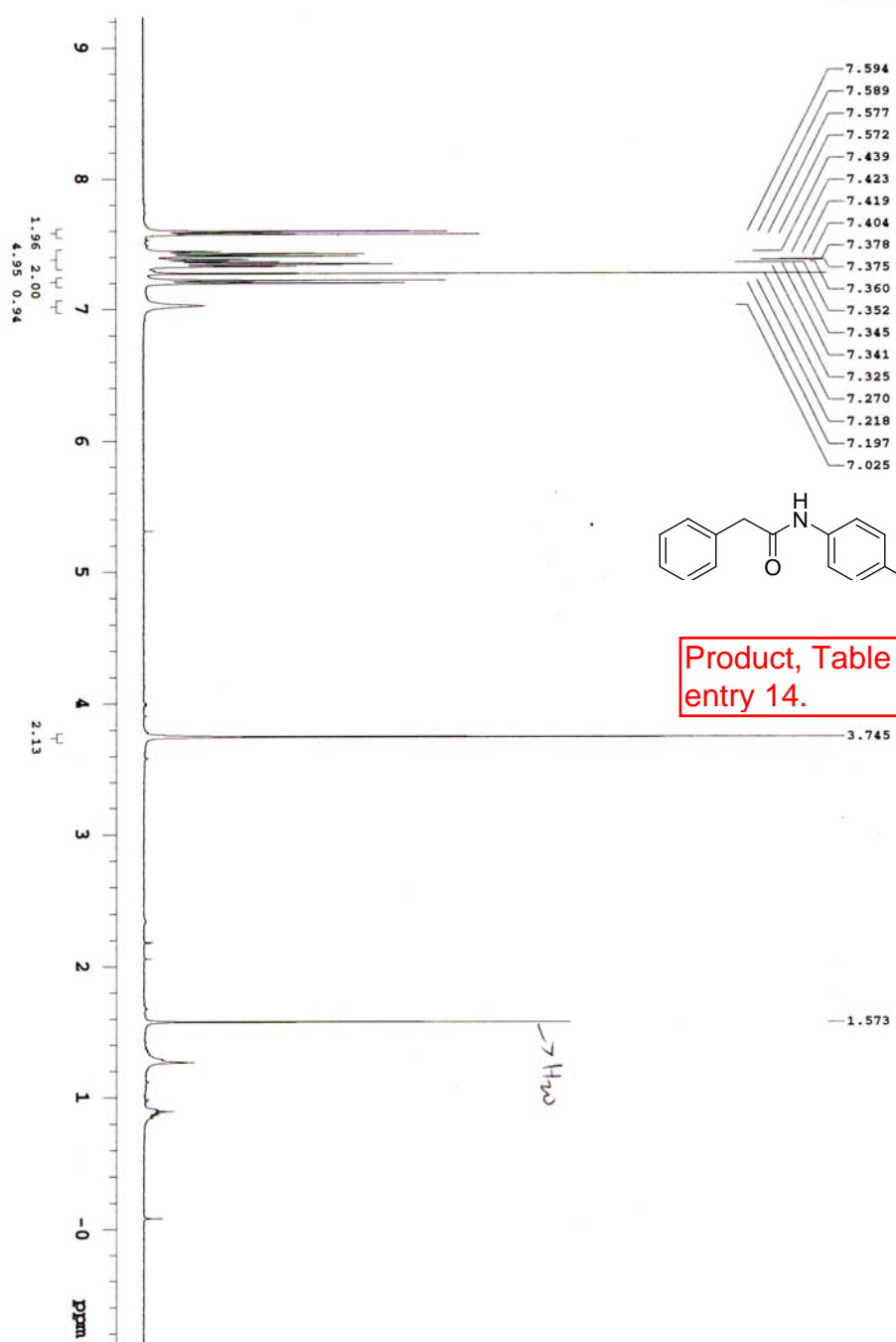
Product, Table 3,  
entry 11.

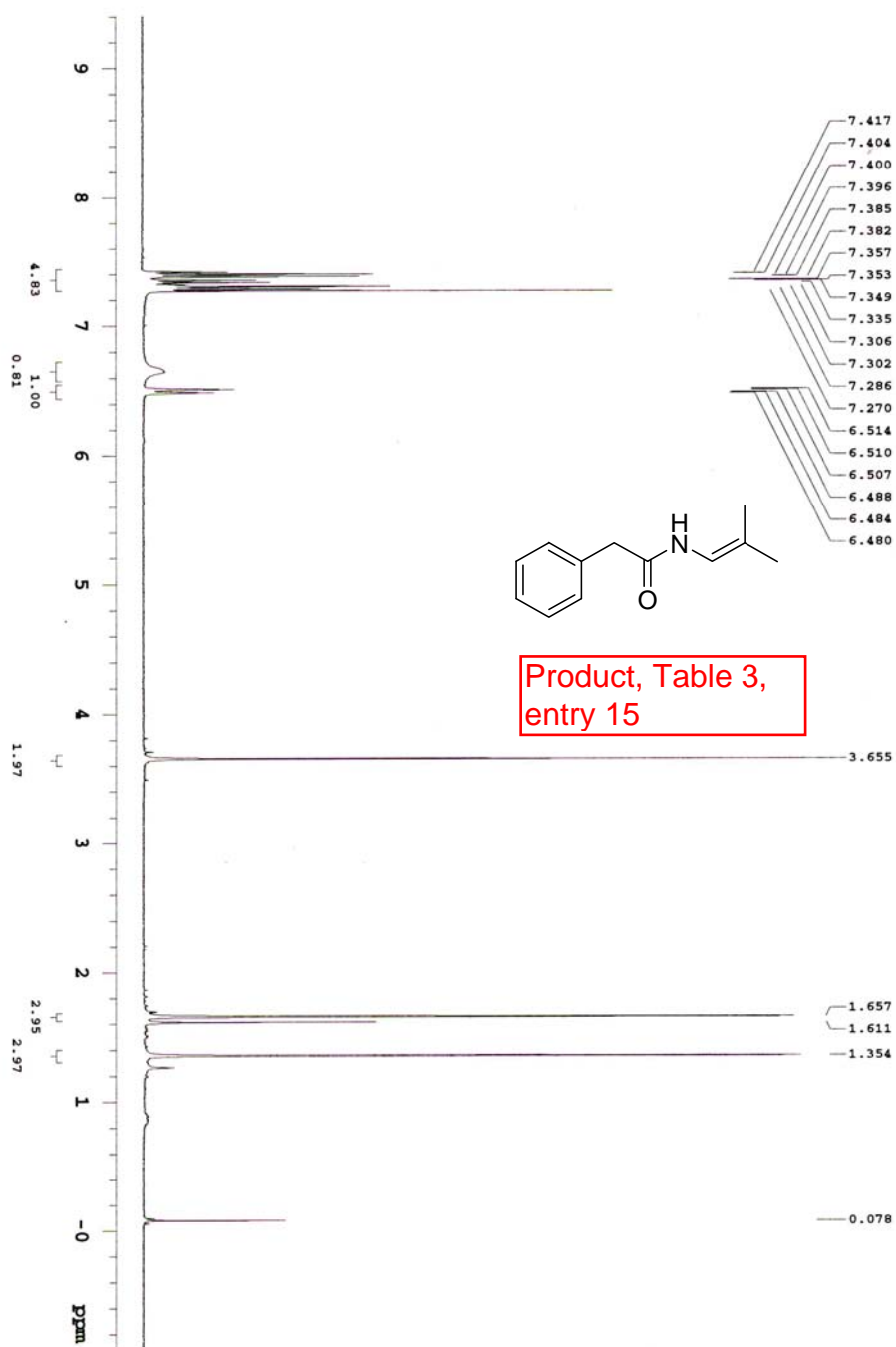


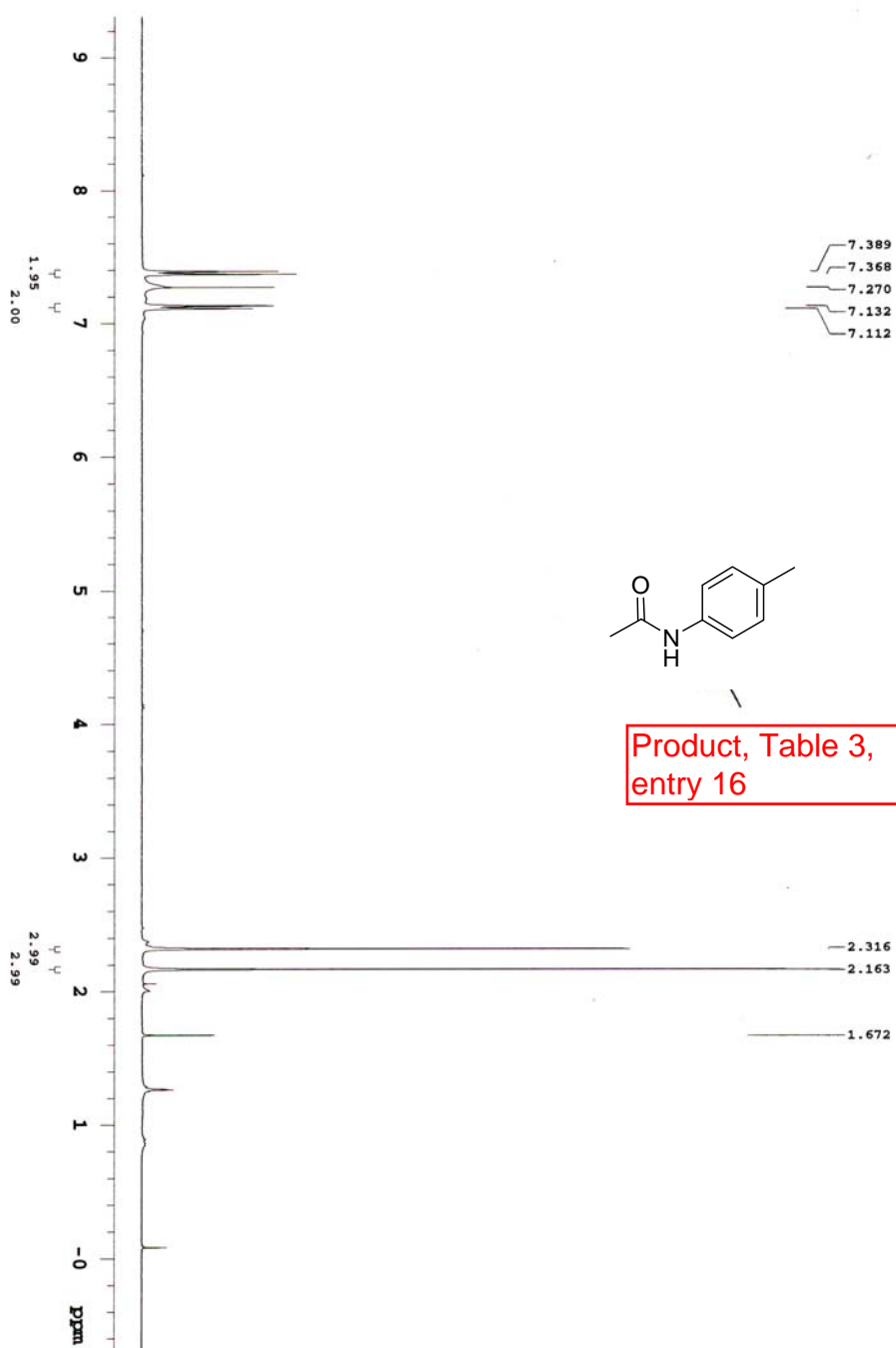
Product, Table 3,  
entry 12

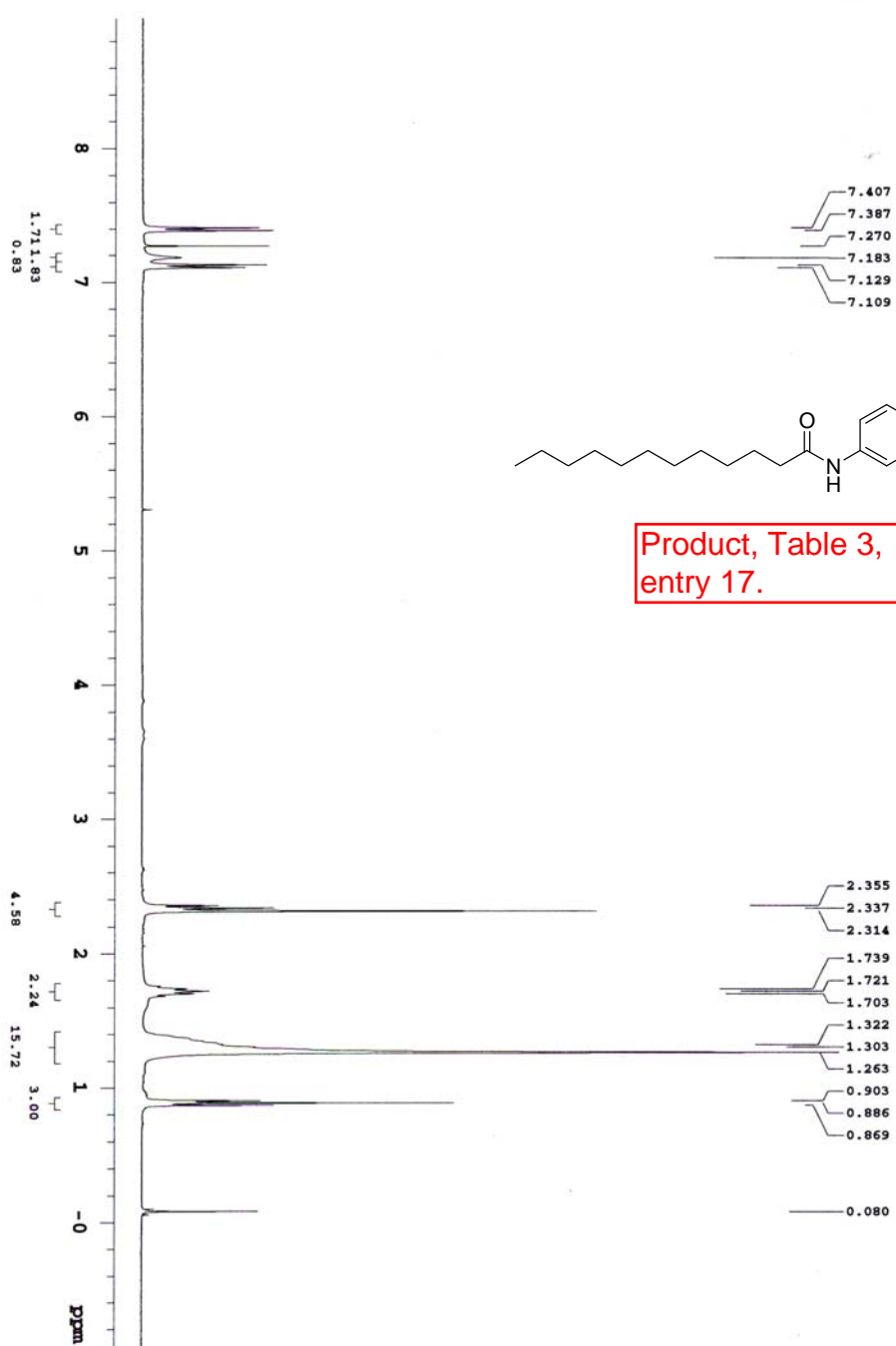




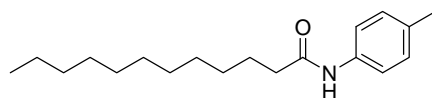
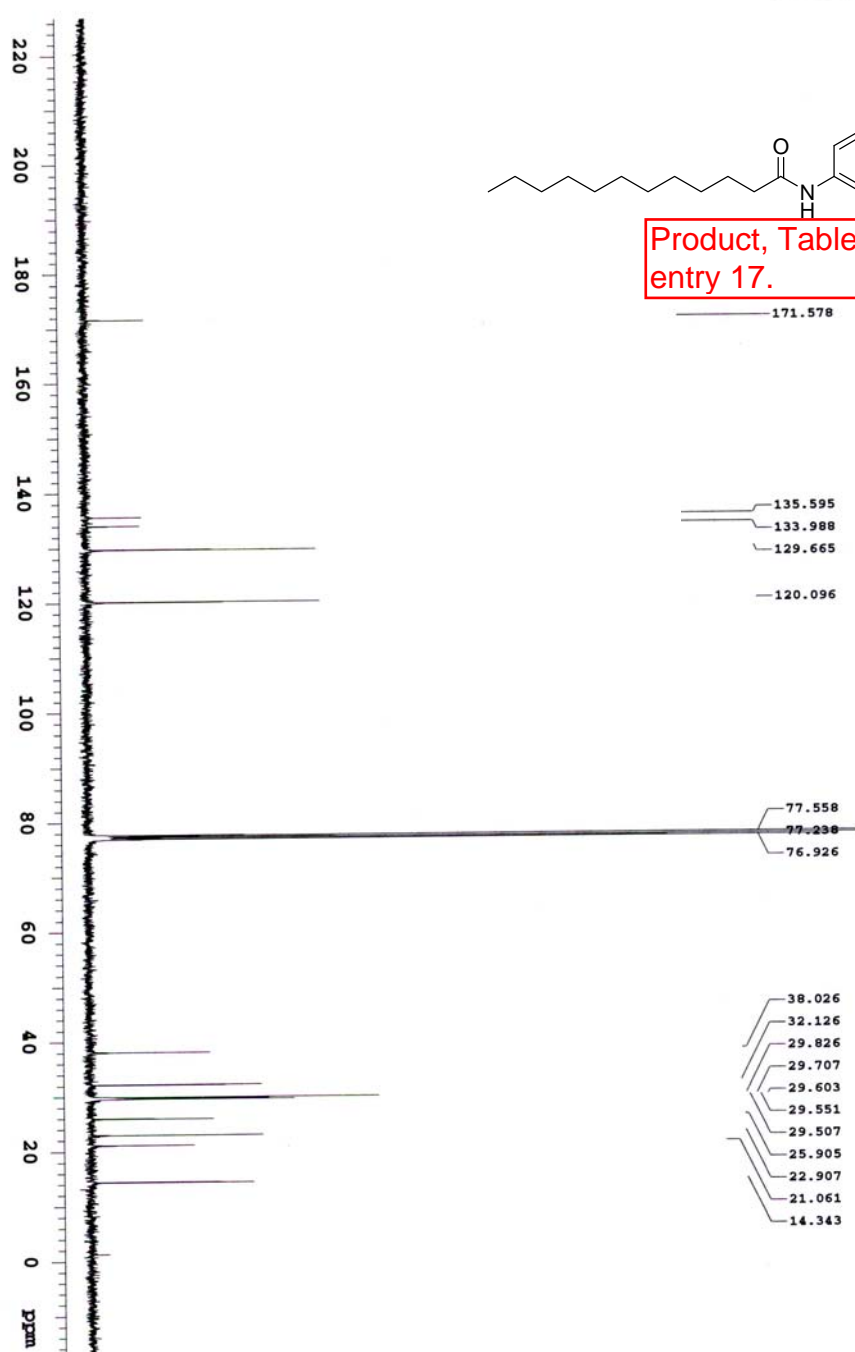




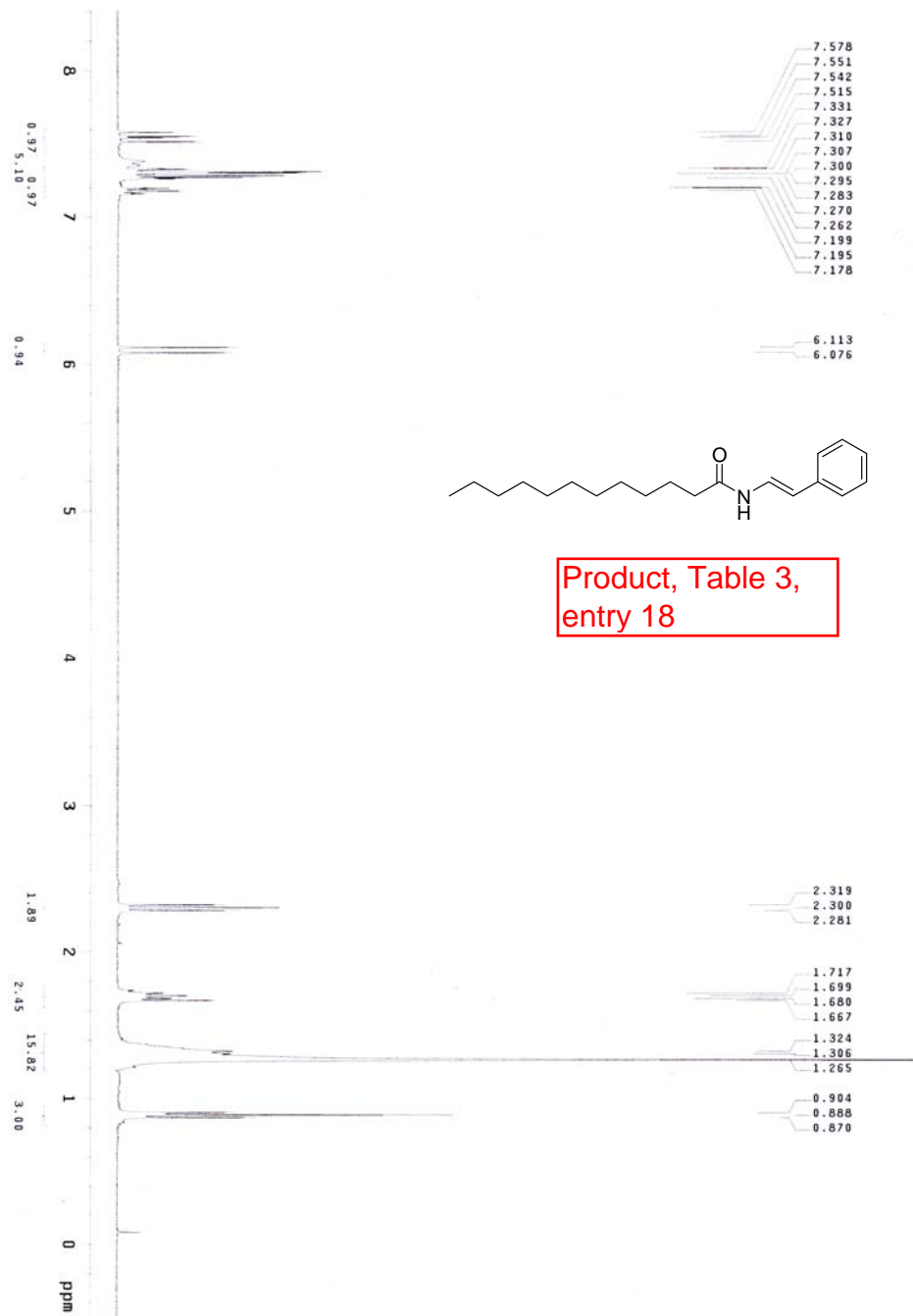


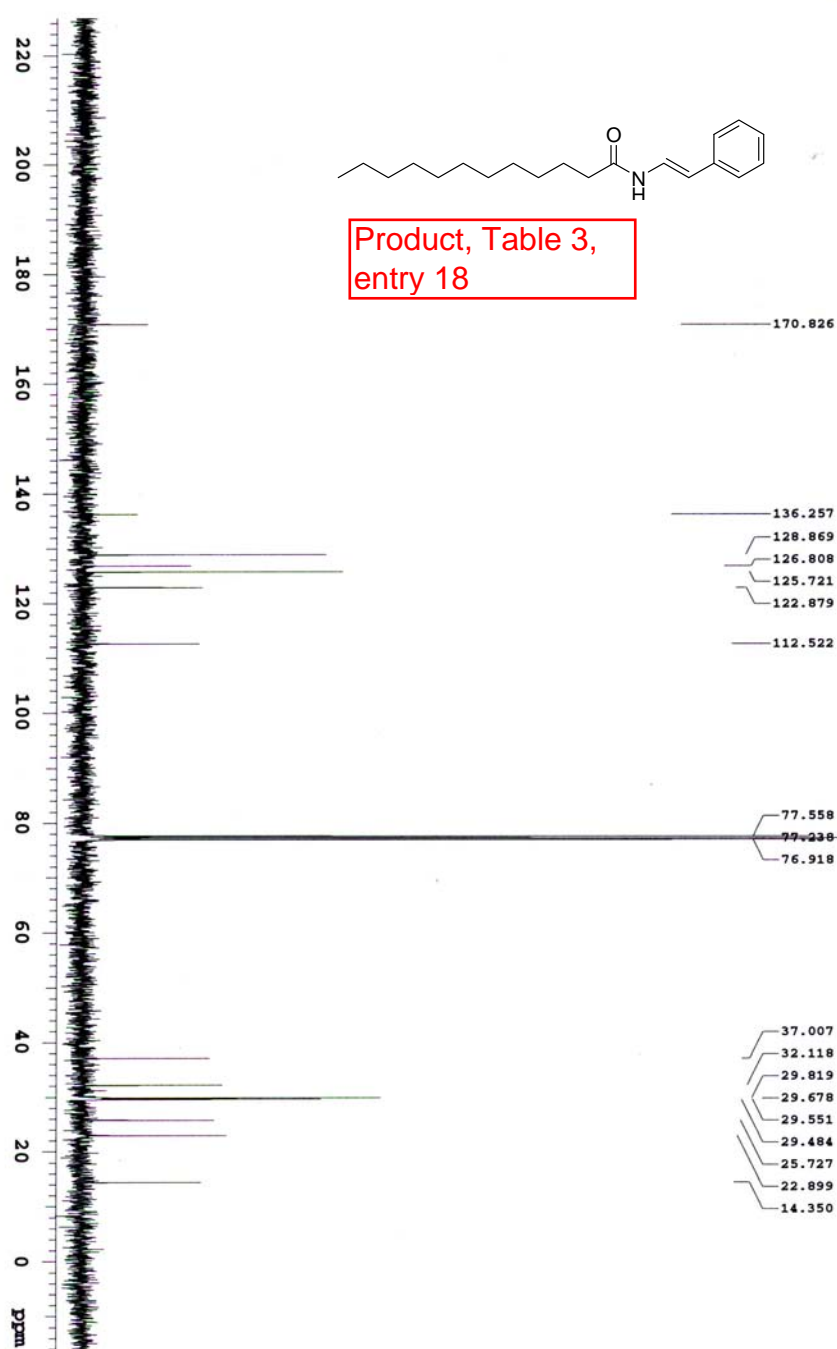


Product, Table 3,  
entry 17.

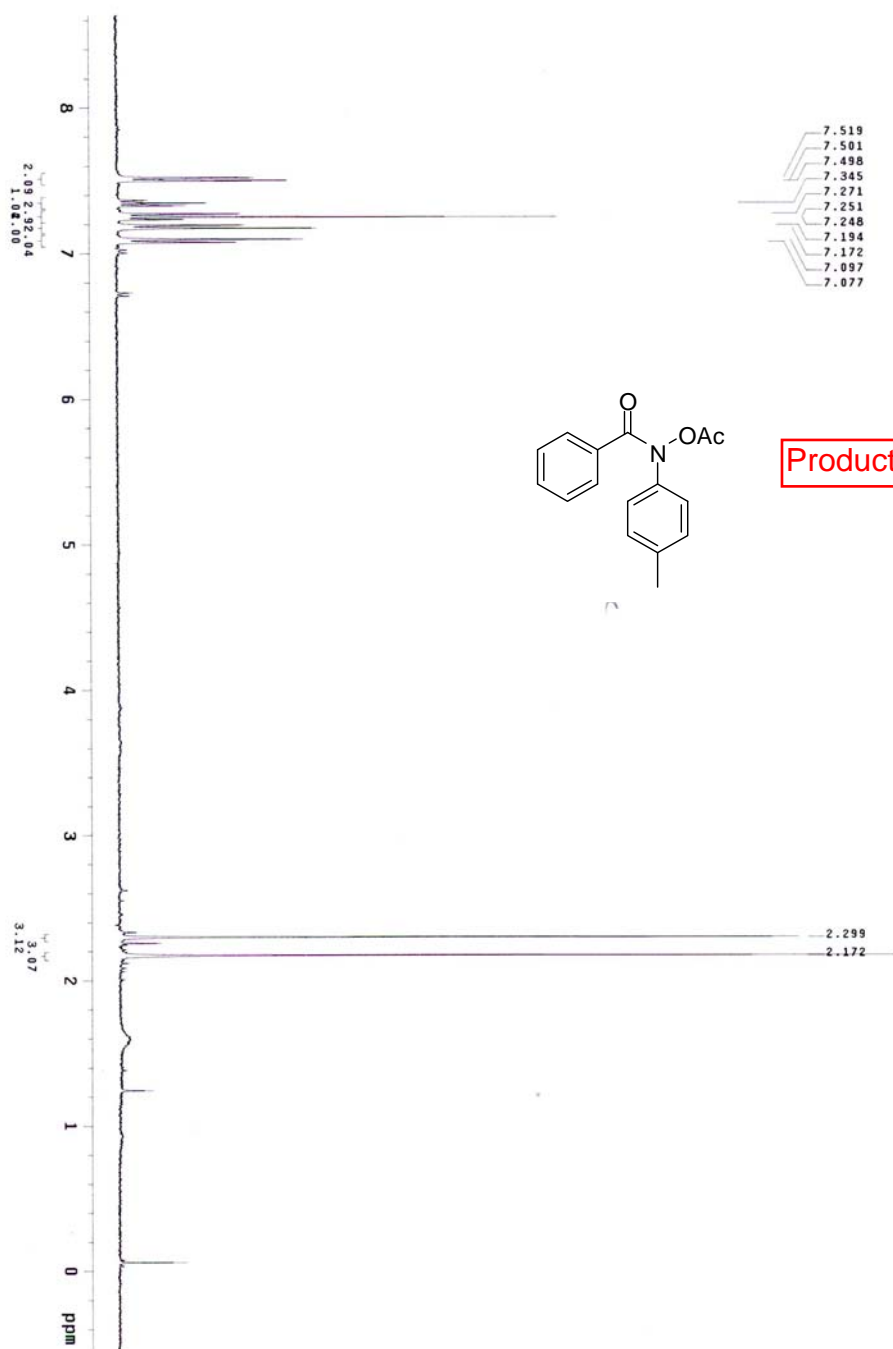


Product, Table 3,  
entry 17.









Product, Scheme 4

Product, Scheme 4

