

**Palladium-Catalyzed Synthesis of Aryl Ketones by Coupling of  
Aryl Bromides with an Acyl Anion equivalent**

**Supporting Information**

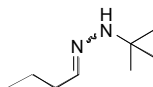
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**General Comments.** All reactions were performed under nitrogen atmosphere using standard Schlenk and drybox techniques. THF was dried by passing it through column of activated alumina. 1, 4-dioxane was purchased from commercial sources and was used under nitrogen without further purification.  $\text{Pd}_2(\text{dba})_3$ , DPEphos and Sodium *tert*-butoxide were purchased from commercial sources and were used without further purification. NMR spectra were recorded on a 400 MHz instrument. GC-MS data were obtained with an Alltech EC-1 capillary column.

### Synthesis of *N-tert-butyl hydrazones*

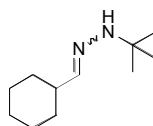
#### *N-tert-butyl-N'-butylidenehydrazine*<sup>1</sup> [50265-96-8]



A literature procedure<sup>1</sup> involving the reaction of *tert*-butylhydrazine hydrochloride neutralized with NaOH and the aldehyde in the presence of catalytic acetic acid was followed with butyraldehyde (2.40 ml, 26.8 mmol) and *tert*-butylhydrazine hydrochloride (4.00 g, 32.1 mmol). The crude product was distilled under reduced pressure to give the title compound as *ca.* 2.6 : 1 *E/Z* mixture. (2.80 g, 19.7 mmol, 74%)

<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  0.93 (t, 2.2H,  $J = 8.4$  Hz), 0.93 (t, 0.8 H,  $J = 8.4$  Hz), 1.16 (s, 6.5H), 1.19 (s, 2.5H), 1.45-1.62 (m, 2H), 2.00-2.05 (m, 0.5H), 2.13-2.18 (m, 1.5H), 4.66 (br, 1H), 6.48 (t, 0.3 H,  $J = 4.9$  Hz), 7.01 (t, 0.7 H,  $J = 5.3$  Hz); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.5, 13.8, 19.4, 20.1, 27.6, 28.1, 28.3, 34.0, 52.8, 52.9, 141.8, 142.5; MS, *m/e*: 142 [ $\text{M}^+$ ], 127 [ $\text{M}^+ - \text{Me}$ ].

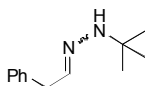
#### *N-tert-butyl-N'-cyclohexylmethylenehydrazine*



The literature procedure<sup>1</sup> involving the reaction of *tert*-butylhydrazine hydrochloride neutralized with NaOH and the aldehyde in the presence of catalytic acetic acid was followed with cyclohexanecarboxaldehyde (4.00 ml, 33.3 mmol) and *tert*-butylhydrazine hydrochloride (4.98 g, 40.0 mmol). The crude product was distilled under reduced pressure to give the title compound as *ca.* 12 : 1 *E/Z* mixture. (4.25 g, 23.3 mmol, 70%)

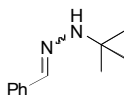
<sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.15-1.28 (m, 14H), 1.70-1.79 (m, 5H), 2.14-2.28 (m, 1H), 4.53 (br, 1H), 6.32 (d, 0.07 H,  $J = 7.2$  Hz), 6.92 (d, 0.93 H,  $J = 5.5$  Hz); <sup>13</sup>C NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.7, 25.9, 26.3, 26.5, 27.2, 28.7, 29.7, 31.1, 34.6, 40.8, 53.5, 146.5, 147.5; MS, *m/e*: 182 [ $\text{M}^+$ ], 167 [ $\text{M}^+ - \text{Me}$ ], 125 [ $\text{M}^+ - \text{tBu}$ ]; Anal. Calcd. for  $\text{C}_{11}\text{H}_{22}\text{N}_2$ : C, 72.47, H, 12.16, N, 15.37. Found: C, 72.48; H, 12.18, N, 15.62.

#### *N-tert-butyl-N'-phenethylidenehydrazine*



The literature procedure<sup>1</sup> involving the reaction of *tert*-butylhydrazine hydrochloride neutralized with NaOH and the aldehyde in the presence of catalytic acetic acid was followed with phenylacetaldehyde (4.70 ml, 40.1 mmol) and *tert*-butylhydrazine hydrochloride (6.00 g, 48.2 mmol). The crude product was distilled under reduced pressure to give the title compound as *ca.* 3 : 1 *E* / *Z* mixture. (6.47 g, 34.0 mmol, 85%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 1.19 (s, 9H), 3.42 (d, 0.5H, *J* = 4.9 Hz), 3.53 (d, 1.5H, *J* = 5.7 Hz), 4.69 (br, 1H), 6.68 (t, 0.25H, *J* = 4.9 Hz), 7.06 (t, 0.75H, *J* = 5.7 Hz), 7.20-7.35 (m, 5H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 28.4, 28.5, 33.1, 38.9, 53.3, 53.3, 126.4, 126.6, 128.5, 128.6, 128.8, 128.8, 137.2, 138.0, 139.4, 140.7; MS, *m/e*: 190 [M<sup>+</sup>], 167 [M<sup>+</sup>-Me], 91 [PhCH<sub>2</sub><sup>+</sup>].

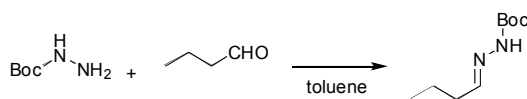
#### ***N*-Benzylidene-*N'*-*tert*-butylhydrazine<sup>2</sup> [32818-95-4]**



A literature procedure<sup>1</sup> involving the reaction of *tert*-butylhydrazine hydrochloride neutralized with NaOH and the aldehyde in the presence of catalytic acetic acid was followed with phenylaldehyde (2.00 ml, 18.0 mmol) and *tert*-butylhydrazine hydrochloride (2.69 g, 21.6 mmol). The crude product was distilled under reduced pressure to give the title compound as *ca.* >95 : <5 *E* / *Z* mixture. (2.36 g, 13.4 mmol, 74%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 1.21 (s, 9H), 5.47 (br, 1H), 7.15-7.19 (m, 1H), 7.24-7.27 (m, 2H), 7.48-7.52 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 28.6, 54.0, 125.9, 127.9, 128.4, 136.0, 138.4; MS, *m/e*: 176 [M<sup>+</sup>], 161 [M<sup>+</sup>-Me], 119 [M<sup>+</sup>-<sup>t</sup>Bu], 77 [Ph<sup>+</sup>].

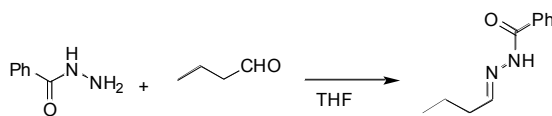
#### **Synthesis of other hydrazones**

##### ***N'*-Butylidene-hydrazinecarboxylic acid *tert*-butyl ester [149268-07-5]**



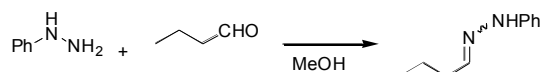
To a toluene (30 mL) solution of *tert*-butylcarbazate (2.00g, 15.1 mmol), butyraldehyde (1.63 ml, 18.2 mmol) was added at room temperature. The reaction mixture was stirred at 60 °C for 1 h. After the reaction, toluene was evaporated. The crude product was triturated with hexane (15 mL) and was filtered and dried under reduced pressure to give the title compound as white solid (1.98 g, 10.6 mmol, 57%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 0.94 (t, 3H, *J* = 7.4 Hz), 1.49-1.53 (m, 11H), 2.25 (t, 2H, *J* = 7.3 Hz), 7.21 (s, 1H), 8.14 (br, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.6, 19.9, 28.1, 34.0, 80.5, 147.3; MS, *m/e*: 186 [M<sup>+</sup>].

##### **Benzoic acid butylidenehydrazide [99840-61-6]**



To a THF (40 mL) solution of benzhydrazide (2.00 g, 14.7 mmol), butyraldehyde (1.71 mL, 19.1 mmol) was added and was stirred at room temperature for 16 h. After the reaction, THF was evaporated. The crude product was triturated with diethyl ether (25 mL) and was filtered then dried under reduced pressure to give the title compound as white solid (2.64 g, 13.9 mmol, 95%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  0.90 (t, 3H,  $J = 7.3$  Hz), 1.45-1.51 (m, 2H), 2.24 (t, 2H,  $J = 7.3$  Hz), 7.28-7.35 (m, 2H), 7.42-7.46 (m, 1H), 7.72 (t, 1H,  $J = 5.5$  Hz), 7.73-7.84 (m, 2H), 10.5 (br, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.8, 20.0, 34.4, 127.5, 128.4, 131.7, 133.2, 153.2, 164.6; MS, m/e: 190 [ $\text{M}^+$ ], 105 [ $\text{PhCO}^+$ ], 77 [ $\text{Ph}^+$ ].

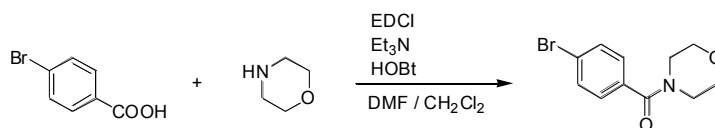
#### ***N*-Butylidene-*N'*-phenylhydrazine [940-54-5]**



To a methanol (40 mL) solution of phenylhydrazine (4.00 mL), butyraldehyde (4.80 mL, 52.7 mmol) was added and was stirred at room temperature for 16 h. After the reaction, methanol was evaporated. To the crude mixture, diethyl ether (40 mL) and magnesium sulfate was added. After filtration and concentration, the crude product was distilled under reduced pressure to give the title compound as *ca.* 4 : 1 *E* / *Z* mixture.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  0.89-0.94 (m, 3H), 1.43-1.54 (m, 2H), 1.97-2.02 (m, 0.4H), 2.14-2.19 (m, 1.6H), 6.43 (t, 0.2H,  $J = 5.1$  Hz), 6.73-6.83 (m, 1.6H), 6.90-6.92 (m, 1.6H), 7.00-7.20 (m, 3.6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.5, 13.6, 19.3, 20.1, 27.6, 33.8, 112.2, 112.7, 118.9, 119.8, 128.9, 129.0, 141.0, 141.4, 145.0, 145.3; MS, m/e: 162 [ $\text{M}^+$ ], 92 [ $\text{PhNH}^+$ ], 77 [ $\text{Ph}^+$ ].

### **Synthesis of aryl bromides**

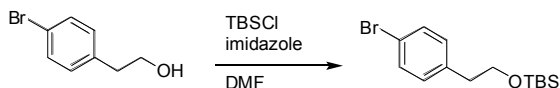
#### **Synthesis of (4-Bromophenyl)-morpholin-4-yl-methanone<sup>3</sup> [127580-92-1]**



To a mixture of 4-bromobenzoic acid (1.00 g, 4.97 mmol), morpholine (565  $\mu\text{L}$ , 6.46 mmol) and triethyl amine (1.00 mL, 7.46 mmol) in DMF (10 mL) and  $\text{CH}_2\text{Cl}_2$  (6 mL), EDCI (1.43 g, 7.46 mmol) and 1-hydroxybenzotriazole (336 mg, 2.49 mmol) were added at room temperature. The reaction mixture was stirred at room temperature for 20 h. After the reaction, water was added to the reaction mixture. The organic layer was extracted with ethyl acetate and was dried over magnesium sulfate. The crude product was purified by flash silica gel chromatography (65% EtOAc in hexane) to give the title compound as colorless solid. (1.34 g, 4.96 mmol, quant.)  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  3.44-3.73 (m, 8H), 7.29 (d, 2H,  $J = 8.4$  Hz), 7.56 (d, 2H,  $J = 8.4$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$

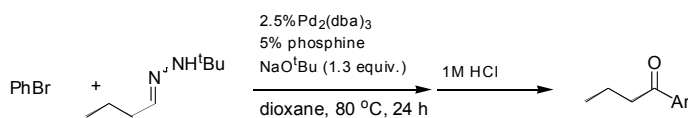
42.5(broad), 48.3(broad), 66.8, 124.2, 128.8, 131.8, 134.0, 169.3; MS, m/e 270 [ $M^+$ ], 184 [ $M^+ - C_4H_8NO$ ], 155 [ $BrC_6H_4^+$ ].

#### Synthesis of [2-(4-Bromophenyl)-ethoxy]-*tert*-butyl-dimethylsilane <sup>4</sup> [73899-15-7]



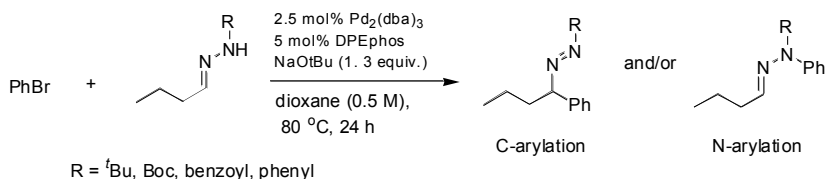
To a mixture of 4-bromophenethyl alcohol (1.50 g, 7.46 mmol) and imidazole (762 mg, 11.2 mmol) in DMF (15 mL), *tert*-butyldimethylsilyl chloride (1.35 g, 8.95 mmol) was added at room temperature. The reaction mixture was stirred at room temperature for 8 h. After the reaction, water was added at 0 °C. The organic layer was extracted with diethyl ether and was dried over magnesium sulfate. The crude product was purified by flash silica gel chromatography (2% EtOAc in hexane) to give the title compound as clear oil. (2.20 g, 6.98 mmol, 94%) <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 0.09 (s, 6H), 0.91 (s, 9H), 2.79 (d, 2H, *J* = 6.8 Hz), 3.81 (d, 2H, *J* = 6.8 Hz), 7.10 (d, 2H, *J* = 8.4 Hz), 7.41 (d, 2H, *J* = 8.4 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ -5.5, 18.2, 25.9, 38.9, 64.0, 119.9, 130.9, 131.2, 138.2; MS, m/e: 315 [ $M^+$ ], 257 [ $M^+ - C_6H_9$ ].

#### Ligand screening procedure for the cross-coupling of *N*-*tert*-butyl hydrazone with bromobenzene.



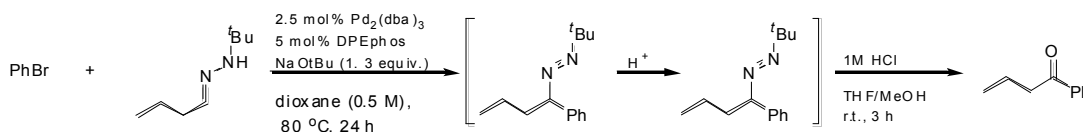
In a dry box, Pd<sub>2</sub>(dba)<sub>3</sub> (11.4 mg, 0.0125 mmol), phosphine (0.0250 mmol), and sodium *tert*-butoxide (62.5 mg, 0.0650 mmol) were dissolved in dioxane (0.5 mL). To this solution, *N*-*tert*-butyl-*N'*-butylidenehydrazine (85.3 mg, 0.600 mmol), bromobenzene (52.7 μl, 0.500 mmol), and dodecane (10.0 μl, 0.0439 mmol, as an internal standard) were added followed by dioxane (0.5 mL). The vial was sealed with a cap containing a PTFE septum and removed from the dry box. The reaction mixture was stirred at the indicated temperature for 24 h. After the reaction, the reaction mixture was filtered through celite pad and was dissolved in THF (1 mL) and MeOH (1 mL). To this solution, 1 M HCl (2 mL) was added, and the reaction mixture was stirred at room temperature for 3 h. After the hydrolysis, small aliquot was taken from the reaction mixture and was analyzed GC to determine conversion of bromobenzene and yield of butyrophenone.

#### Coupling of bromobenzene with various hydrazones.



In a dry box, Pd<sub>2</sub>(dba)<sub>3</sub> (11.4 mg, 0.0125 mmol), DPEphos (13.5 mg, 0.0250 mmol), and sodium tert-butoxide (62.5 mg, 0.0650 mmol) were dissolved in dioxane (0.5 mL). To this solution, hydrazone (0.600 mmol) and the bromobenzene (52.7 µl, 0.500 mmol) were added, followed by dioxane (0.5 mL). The vial was sealed with a cap containing a PTFE septum and removed from the dry box. The reaction mixture was stirred at the indicated temperature for 24 h. After the reaction, the reaction mixture was analyzed GC and TLC.

(R = *t*Bu): The crude product was hydrolyzed without isolating the C-arylation product due to instability of C-arylation product.

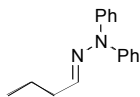


The reaction mixture was filtered through a silica gel plug (0.5 cm) and was washed with diethyl ether. The filtrate was concentrated and was dissolved in THF (3 mL) and MeOH (3 mL). To this solution, 1 M HCl (2 mL) was added, and the reaction mixture was stirred at room temperature for 3 h. After hydrolysis, the solvent was evaporated. The organic layer was extracted with diethyl ether twice and was dried over magnesium sulfate. After filtration and concentration of the organic layer, the reaction mixture was purified by preparative TLC (10% EtOAc in hexane) to give 72.4 mg (98%) of butyrophenone as a clear oil.

(R = Boc, benzoyl): No reaction was observed.

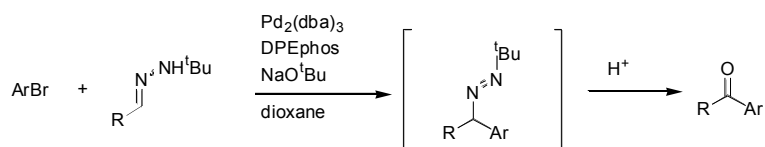
(R = phenyl): The reaction was filtered through a silica gel plug (0.5 cm) and was washed with diethyl ether. The crude mixture was concentrated and was purified by flash silica gel chromatography (5% EtOAc in hexane) to give 108.3 mg (91%) of the N-arylation product as pale brown oil.

#### ***N'*-Butylidene-*N,N*-diphenyl-hydrazine [861598-53-0]**



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 0.92 (t, 3H, *J* = 7.4 Hz), 1.47-1.53 (m, 2H), 2.23-2.29 (m, 2H), 6.53 (t, 1H, *J* = 5.4 Hz), 7.07-7.13 (m, 6H), 7.34-7.38 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.7, 20.3, 34.6, 122.3, 123.7, 129.6, 139.9, 144.3; MS, *m/e*: 238 [M<sup>+</sup>], 77 [Ph<sup>+</sup>].

**General procedure for the cross-coupling of *N*-*tert*-butyl hydrazone with aryl halide under optimized condition.**



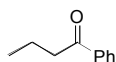
In a dry box,  $\text{Pd}_2(\text{dba})_3$  (11.4 mg, 0.0125 mmol), DPEphos (13.5 mg, 0.0250 mmol), and sodium *tert*-butoxide (62.5 mg, 0.0650 mmol) were dissolved in dioxane (0.5 mL). To this solution, *N-tert*-butyl hydrazone (0.600 mmol) and the aryl bromide (0.500 mmol) were added, followed by dioxane (0.5 mL). The vial was sealed with a cap containing a PTFE septum and removed from the dry box. The reaction mixture was stirred at the indicated temperature for 24 h. After the reaction, the reaction mixture was filtered through a silica gel plug (0.5 cm) and was washed with diethyl ether.

(R = *n*-Pr) The filtrate was concentrated and was dissolved in THF (3 mL) and MeOH (3 mL). To this solution, 1 M HCl (2 mL) was added, and the reaction mixture was stirred at room temperature for 3 h. After hydrolysis, the solvent was evaporated. The organic layer was extracted with diethyl ether twice and was dried over magnesium sulfate. After filtration and concentration of the organic layer, the crude product was purified by flash silica gel chromatography or preparative TLC.

(R = cyclohexyl or phenyl) The filtrate was concentrated and was dissolved in THF (3 mL). To this solution was added 10% aqueous sulfuric acid (2 mL), and the reaction mixture was stirred at room temperature for 24 h. After hydrolysis, the solvent was evaporated. The organic layer was extracted with diethyl ether (R = cyclohexyl) or ethyl acetate (R = phenyl) twice and was dried over magnesium sulfate. After filtration and concentration of the organic layer, the crude product was purified by flash silica gel chromatography or preparative TLC.

(R = benzyl) The filtrate was concentrated and was dissolved in THF/MeOH (3 mL / 3 mL). To this solution, 1 M HCl (2 mL) was added, and the reaction mixture was stirred at room temperature for 16 h. After hydrolysis, the solvent was evaporated. The organic layer was extracted with diethyl ether twice and was dried over magnesium sulfate. After filtration and concentration of the organic layer, the crude product was purified by flash silica gel chromatography or preparative TLC.

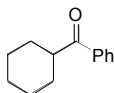
#### 1-Phenylbutan-1-one<sup>5</sup> [495-40-9]



The general procedure was followed with bromobenzene and *N-tert*-butyl-*N'*-butylidene-hydrazine. The reaction mixture was purified by preparative TLC (10% EtOAc in hexane) to give 72.4 mg (98%) of the product as a clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.01 (t, 3H,  $J = 7.4$  Hz), 1.73-1.82 (m, 2H), 2.95 (t, 2H,  $J = 7.3$  Hz), 7.44-7.48 (m, 2H), 7.53-7.69 (m, 1H), 7.96 (d, 2H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR

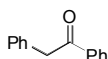
(100 MHz, CDCl<sub>3</sub>)  $\delta$  13.9, 17.7, 40.5, 128.0, 128.5, 132.8, 137.0, 200.4; MS, m/e: 148 [M<sup>+</sup>], 105 [PhCO<sup>+</sup>], 77 [Ph<sup>+</sup>].

**Cyclohexylphenylmethanone<sup>5</sup> [712-50-5]**



The general procedure was followed with bromobenzene and *N-tert*-butyl-*N'*-cyclohexylmethylenhydrazine. The reaction mixture was purified by preparative TLC (11% EtOAc in hexane) to give 76.6 mg (81%) of the product as a white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS)  $\delta$  1.19-1.55 (m, 5H), 1.72-1.76 (m, 1H), 1.83-1.91 (m, 4H), 3.23-3.30 (m, 1H), 7.44-7.48 (m, 2H), 7.52-7.56 (m, 1H), 7.94 (d, 2H, *J* = 8.2 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  25.9, 26.0, 29.4, 45.6, 128.2, 128.5, 132.7, 136.4, 203.8; MS, m/e: 188 [M<sup>+</sup>], 105 [PhCO<sup>+</sup>], 77 [Ph<sup>+</sup>].

**1,2-Diphenylethanone<sup>6</sup> [451-40-1]**



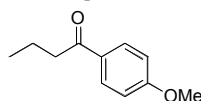
The general procedure was followed with bromobenzene and *N-tert*-butyl-*N'*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (6% EtOAc in hexane) to give 94.1 mg (96%) of the product as white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS)  $\delta$  4.29 (s, 2H), 7.23-7.28 (m, 3H), 7.31-7.35 (m, 2H), 7.44-7.48 (m, 2H), 7.54-7.58 (m, 1H), 8.00-8.03 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  45.5, 126.7, 128.6, 128.6, 128.6, 129.4, 133.1, 134.5, 136.5, 197.6; MS, m/e: 196 [M<sup>+</sup>], 105 [PhCO<sup>+</sup>], 91 [PhCH<sub>2</sub><sup>+</sup>], 77 [Ph<sup>+</sup>].

**Benzophenone<sup>7</sup> [119-61-9]**



The general procedure was followed with bromobenzene and *N*-Benzylidene-*N'*-*tert*-butylhydrazine. The reaction mixture was purified by flash silica gel chromatography (5% EtOAc in hexane) to give 77.0 mg (85%) of the product as a white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS)  $\delta$  7.45-7.49 (m, 4H), 7.56-7.59 (m, 2H), 7.79 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  128.2, 130.0, 132.4, 137.5, 196.7; MS, m/e: 182 [M<sup>+</sup>], 105 [PhCO<sup>+</sup>], 77 [Ph<sup>+</sup>].

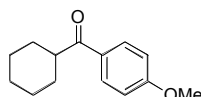
**1-(4-Methoxyphenyl)-butan-1-one<sup>8</sup> [4160-51-4]**



The general procedure was followed with 4-bromoanisole and *N-tert*-butyl-*N'*-butylidenehydrazine.

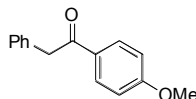
The reaction mixture was purified by flash silica gel chromatography (3% EtOAc in hexane) to give 83.7 mg (94%) of the product as pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  0.99 (t, 3H,  $J = 7.4$  Hz), 1.75 (m, 2H), 2.88 (t, 2H,  $J = 7.3$  Hz), 3.85 (s, 3H), 6.92 (d, 2H,  $J = 8.8$  Hz), 7.94 (d, 2H,  $J = 8.8$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.8, 17.9, 40.1, 55.3, 113.5, 130.1, 130.2, 163.2, 198.9; MS, m/e: 178 [ $\text{M}^+$ ], 135 [ $\text{MeOC}_6\text{H}_4\text{CO}^+$ ], 107 [ $\text{MeOC}_6\text{H}_4^+$ ].

**Cyclohexyl-(4-methoxyphenyl)-methanone<sup>9</sup> [7469-80-9]**



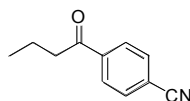
The general procedure was followed with 4-bromoanisole and *N-tert*-butyl-*N'*-cyclohexylmethylenhydrazine. The reaction mixture was purified by flash silica gel chromatography (3% EtOAc in hexane) to give 86.3 mg (79%) of the product as clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.21-1.55 (m, 5H), 1.71-1.74 (m, 1H), 1.81-1.88 (m, 4H), 3.18-3.26 (m, 1H), 3.85 (s, 3H), 6.93 (d, 2H,  $J = 8.8$  Hz), 7.94 (d, 2H,  $J = 8.8$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.9, 26.1, 29.6, 45.3, 55.4, 113.7, 129.2, 130.5, 163.2, 202.4; MS, m/e: 218 [ $\text{M}^+$ ], 135 [ $\text{MeOC}_6\text{H}_4\text{CO}^+$ ], 107 [ $\text{MeOC}_6\text{H}_4^+$ ].

**1-(4-Methoxyphenyl)-2-phenylethanone<sup>10</sup> [1023-17-2]**



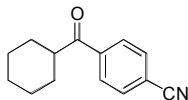
The general procedure was followed with 4-bromoanisole and *N-tert*-butyl-*N'*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (6% EtOAc in hexane) to give 103 mg (91%) of the product as pale yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  3.85 (s, 3H), 4.23 (s, 2H), 6.92 (d, 2H,  $J = 8.9$  Hz), 7.22-7.33 (m, 5H), 7.99 (d, 2H,  $J = 8.9$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  45.2, 55.4, 113.7, 126.7, 128.6, 129.3, 129.6, 130.9, 134.9, 163.7, 196.2; MS, m/e: 226 [ $\text{M}^+$ ], 135 [ $\text{MeOC}_6\text{H}_4\text{CO}^+$ ], 107 [ $\text{MeOC}_6\text{H}_4^+$ ].

**4-Butyrylbenzonitrile<sup>11</sup> [90819-57-1]**



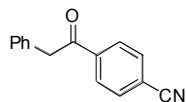
The general procedure was followed with 4-bromobenzonitrile and *N-tert*-butyl-*N'*-butylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (3% EtOAc in hexane) to give 61.0 mg (70%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.02 (t, 3H,  $J = 7.4$  Hz), 1.86-1.72 (m, 2H), 2.97 (t, 2H,  $J = 7.23$  Hz), 7.77 (d, 2H,  $J = 8.4$  Hz), 8.05 (d, 2H,  $J = 8.4$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.8, 17.5, 40.8, 116.2, 118.0, 128.5, 132.5, 140.1, 198.9; MS, m/e: 173 [ $\text{M}^+$ ], 130 [ $\text{NCC}_6\text{H}_4\text{CO}^+$ ], 102 [ $\text{NCC}_6\text{H}_4^+$ ].

#### 4-Cyclohexanecarbonylbenzonitrile



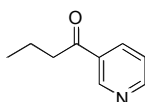
The general procedure was followed with 4-bromobenzonitrile and *N-tert*-butyl-*N'*-cyclohexylmethylenhydrazine. The reaction mixture was purified by preparative TLC (10% EtOAc in hexane) to give 49.2 mg (46%) of the product as pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.23-1.58 (m, 5H), 1.74-1.77 (m, 1H), 1.84-1.89 (m, 4H), 3.22 (tt, 1H,  $J = 11.3, 3.1$  Hz), 7.77 (d, 2H,  $J = 8.2$  Hz), 8.01 (d, 2H,  $J = 8.2$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.4, 25.6, 28.9, 45.7, 115.7, 117.8, 128.5, 132.3, 139.2, 202.2; MS,  $m/e$ : 213 [ $\text{M}^+$ ], 130 [ $\text{NCC}_6\text{H}_4\text{CO}^+$ ], 102 [ $\text{NCC}_6\text{H}_4^+$ ]; Anal. Calcd. for  $\text{C}_{14}\text{H}_{15}\text{NO}$ : C, 78.84, H, 7.09, N, 6.57. Found: C, 78.57; H, 6.89; N, 6.61.

#### 4-Phenylacetylbenzonitrile<sup>12</sup> [60694-99-7]



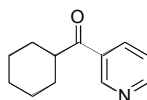
The general procedure was followed with 4-bromobenzonitrile and *N-tert*-butyl-*N'*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (12% EtOAc in hexane) to give 81.0 mg (73%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  4.30 (s, 2H), 7.24-7.30 (m, 3H), 7.33-7.36 (m, 2H), 7.75 (d, 2H,  $J = 8.2$  Hz), 8.08 (d, 2H,  $J = 8.2$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  45.8, 116.4, 117.8, 127.3, 128.9, 129.0, 129.3, 132.5, 133.4, 139.5, 196.2; MS,  $m/e$ : 221 [ $\text{M}^+$ ], 130 [ $\text{NCC}_6\text{H}_4\text{CO}^+$ ], 102 [ $\text{NCC}_6\text{H}_4^+$ ], 91 [ $\text{PhCH}_2^+$ ].

#### 1-Pyridin-3-yl-butan-1-one<sup>13</sup> [1701-70-8]



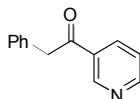
The general procedure was followed with 3-bromopyridine and *N-tert*-butyl-*N'*-butylidenehydrazine. After the hydrolysis, saturated aqueous sodium bicarbonate was added to the reaction mixture. The organic layer was extracted with ethyl acetate. The reaction mixture was purified by flash silica gel chromatography (40% EtOAc in hexane) to give 73.0 mg (98%) of the product as clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.02 (t, 3H,  $J = 7.4$  Hz), 1.87-1.73 (m, 2H), 2.98 (t, 2H,  $J = 7.2$  Hz), 7.43 (dd, 1H,  $J = 7.8, 4.8$  Hz), 8.24 (m, 1H), 8.78 (dd, 1H,  $J = 4.8, 1.6$  Hz), 9.18 (d, 1H,  $J = 1.9$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.6, 17.3, 40.6, 123.5, 132.1, 135.2, 149.4, 153.2, 198.9; MS,  $m/e$ : 149 [ $\text{M}^+$ ], 106 [ $\text{C}_5\text{H}_4\text{NCO}^+$ ], 78 [ $\text{C}_5\text{H}_4\text{N}^+$ ].

#### 1-Pyridin-3-yl-butan-1-one<sup>14</sup> [60148-00-7]



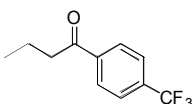
The general procedure was followed with 3-bromopyridine and *N-tert*-butyl-*N'*-cyclohexylmethylenehydrazine. After the hydrolysis, saturated aqueous sodium bicarbonate was added to the reaction mixture. The organic layer was extracted with ethyl acetate. The reaction mixture was flash silica gel chromatography (40% EtOAc in hexane) to give 80.3 mg (85%) of the product as clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.23-1.33 (m, 1H), 1.36-1.56 (m, 4H), 1.73-1.78 (m, 1H), 1.85-1.93 (m, 4H), 3.21-3.28 (m, 1H), 7.43 (dd, 1H,  $J = 7.9, 4.8$  Hz), 8.22 (m, 1H), 8.77 (d,  $J = 4.8$  Hz, 1H), 9.16 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.6, 25.7, 29.0, 45.9, 123.6, 131.3, 135.6, 149.6, 153.0, 202.4; MS,  $m/e$ : 189 [ $\text{M}^+$ ], 106 [ $\text{C}_5\text{H}_4\text{NCO}^+$ ], 78 [ $\text{C}_5\text{H}_4\text{N}^+$ ].

**2-Phenyl-1-pyridin-3-yl-ethanone<sup>15</sup> [14627-92-0]**



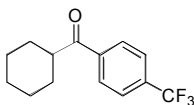
The general procedure was followed with 3-bromopyridine and *N-tert*-butyl-*N'*-phenethylidenehydrazine. After the hydrolysis, saturated aqueous sodium bicarbonate was added to the reaction mixture. The organic layer was extracted with ethyl acetate. The reaction mixture was purified by flash silica gel chromatography (45% EtOAc in hexane) to give 70.0 mg (71%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  4.29 (s, 2H), 7.26-7.41 (m, 6H), 8.24-8.27 (m, 1H), 8.8 (dd, 1H,  $J = 4.8, 1.3$  Hz), 9.23 (d, 1H,  $J = 1.9$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  45.7, 123.6, 127.1, 128.7, 129.3, 131.7, 133.4, 135.8, 149.9, 153.3, 196.3; MS,  $m/e$ : 197 [ $\text{M}^+$ ], 106 [ $\text{C}_5\text{H}_4\text{NCO}^+$ ], 78 [ $\text{C}_5\text{H}_4\text{N}^+$ ].

**1-(4-Trifluoromethylphenyl)-butan-1-one<sup>16</sup> [37851-10-8]**



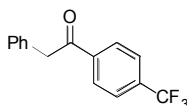
The general procedure was followed with 4-bromobenzotrifluoride and *N-tert*-butyl-*N'*-butylidenehydrazine. The reaction mixture was purified by preparative TLC (2.5% EtOAc in hexane) to give 97.2 mg (90%) of the product as clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.02 (t, 3H,  $J = 7.4$  Hz), 1.74-1.84 (m, 2H), 2.98 (t, 2H,  $J = 7.3$  Hz), 7.72 (d, 2H,  $J = 8.3$  Hz), 8.06 (d, 2H,  $J = 8.3$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.7, 17.5, 40.7, 123.5 (q,  $J = 272.6$  Hz), 125.70 (q,  $J = 3.7$  Hz), 128.4, 134.3 (q,  $J = 32.6$  Hz), 139.8, 199.4; MS,  $m/e$ : 216 [ $\text{M}^+$ ], 173 [ $\text{CF}_3\text{C}_6\text{H}_4\text{NCO}^+$ ], 145 [ $\text{CF}_3\text{C}_6\text{H}_4^+$ ].

**Cyclohexyl-(4-trifluoromethylphenyl)-methanone<sup>17</sup> [419543-02-5]**



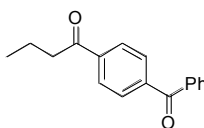
The general procedure was followed with 4-bromobenzotrifluoride and *N-tert*-butyl-*N'*-cyclohexylmethylenhydrazine. The reaction mixture was purified by preparative TLC (2.5% EtOAc in hexane) to give 85.0 mg (66%) of the product as clear oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.22-1.55 (m, 5H), 1.73-1.77 (m, 1H), 1.84-1.92 (m, 4H), 3.26 (tt, 1H,  $J = 11.4, 3.2$  Hz), 7.72 (d, 2H,  $J = 8.1$  Hz), 8.04 (d, 2H,  $J = 8.1$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.8, 25.9, 29.3, 46.0, 123.7 (q,  $J = 272.6$  Hz), 125.7 (q,  $J = 3.7$  Hz), 128.6, 134.1 (q,  $J = 32.6$  Hz), 139.2, 202.9; MS,  $m/e$ : 256 [ $\text{M}^+$ ], 173 [ $\text{CF}_3\text{C}_6\text{H}_4\text{NCO}^+$ ], 145 [ $\text{CF}_3\text{C}_6\text{H}_4^+$ ].

### 2-Phenyl-1-(4-(trifluoromethyl)phenyl)-ethanone<sup>18</sup> [61062-55-3]



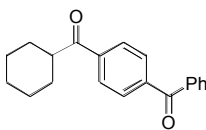
The general procedure was followed with 4-bromobenzotrifluoride and *N-tert*-butyl-*N'*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (2.5% EtOAc in hexane) to give 106 mg (80%) of the product as pale yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  4.31 (s, 2H), 7.25-7.36 (m, 5H), 7.71 (d, 2H,  $J = 8.5$  Hz), 8.10 (d, 2H,  $J = 8.5$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  45.8, 123.5 (q,  $J = 272.8$  Hz), 125.7 (q,  $J = 3.7$  Hz), 127.2, 128.8, 128.9, 129.3, 133.7, 134.4 (q,  $J = 32.7$  Hz), 139.1, 196.6; MS,  $m/e$ : 264 [ $\text{M}^+$ ], 173 [ $\text{CF}_3\text{C}_6\text{H}_4\text{NCO}^+$ ], 145 [ $\text{CF}_3\text{C}_6\text{H}_4^+$ ].

### 1-(4-Benzoylphenyl)-butan-1-one



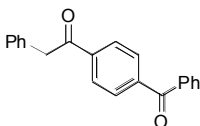
The general procedure was followed with 4-bromobenzophenone and *N-tert*-butyl-*N'*-butylidenehydrazine. The reaction mixture was purified by preparative TLC (4% EtOAc in hexane) to give 110 mg (87%) of the product as pale yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.03 (t, 3H,  $J = 7.4$  Hz), 1.76-1.85 (m, 2H), 3.00 (t, 2H,  $J = 7.3$  Hz), 7.45-7.51 (m, 2H), 7.60-7.64 (m, 1H), 7.80 (d, 2H,  $J = 7.2$  Hz), 7.86 (d, 2H,  $J = 8.3$  Hz), 8.06 (d, 2H,  $J = 8.3$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.7, 17.5, 40.8, 127.8, 128.4, 129.9, 130.0, 132.9, 136.8, 139.5, 141.0, 195.9, 199.7; MS,  $m/e$ : 252 [ $\text{M}^+$ ], 209 [ $\text{M}^+ - \text{C}_3\text{H}_7$ ], 105 [ $\text{PhCO}^+$ ], 77 [ $\text{Ph}^+$ ]; Anal. Calcd. for  $\text{C}_{17}\text{H}_{16}\text{O}_2$ : C, 80.93, H, 6.39. Found: C, 81.06; H, 6.30.

### (4-Benzoylphenyl)-cyclohexylmethanone



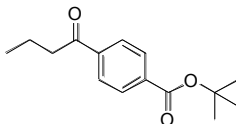
The general procedure was followed with 4-bromobenzophenone and *N-tert*-butyl-*N'*-cyclohexylmethylenhydrazine. The reaction mixture was purified by preparative TLC (3% EtOAc in hexane) to give 76.1 mg (52%) of the product as pale yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.24-1.56 (m, 5H), 1.74-1.77 (m, 1H), 1.85-1.94 (m, 4H), 3.26-3.30 (m, 1H), 7.49-7.51 (m, 2H), 7.61-7.63 (m, 1H), 7.79-7.81 (m, 2H), 7.86 (d, 2H,  $J = 8.4$  Hz), 8.03 (d, 2H,  $J = 8.3$  Hz),  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.8, 25.9, 29.3, 46.1, 128.1, 128.5, 130.1, 130.1, 133.0, 137.0, 139.1, 141.0, 196.0, 203.4; MS,  $m/e$ : 292 [ $\text{M}^+$ ], 209 [ $\text{M}^+ - \text{C}_6\text{H}_{11}$ ], 105 [ $\text{PhCO}^+$ ], 77 [ $\text{Ph}^+$ ]; Anal. Calcd. for  $\text{C}_{20}\text{H}_{20}\text{O}_2$ : C, 82.16, H, 6.89. Found: C, 81.94; H, 6.83.

#### 1-(4-Benzoylphenyl)-2-phenylethanone



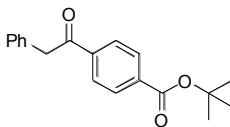
The general procedure was followed with 4-bromobenzophenone and *N-tert*-butyl-*N'*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (5% EtOAc in hexane) to give 143 mg (95%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  4.32 (s, 2H), 7.25-7.29 (m, 3H), 7.32-7.36 (m, 2H), 7.47-7.51 (m, 2H), 7.59-7.63 (m, 1H), 7.78-7.80 (m, 2H), 7.85 (d, 2H,  $J = 8.4$  Hz), 8.10 (d, 2H,  $J = 8.4$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ )  $\delta$  45.0, 1526.6, 128.3, 128.5, 128.7, 129.7, 129.7, 129.8, 133.2, 134.7, 136.4, 138.9, 140.7, 195.3, 197.4; MS,  $m/e$ : 300 [ $\text{M}^+$ ], 209 [ $\text{M}^+ - \text{C}_7\text{H}_7$ ], 105 [ $\text{PhCO}^+$ ], 77 [ $\text{Ph}^+$ ]; Anal. Calcd. for  $\text{C}_{17}\text{H}_{16}\text{O}_2$ : C, 83.98, H, 5.37. Found: C, 83.81; H, 5.50.

#### 4-Butyrylbenzoic acid *tert*-butyl ester



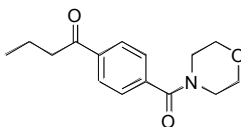
The general procedure was followed with *tert*-butyl 4-bromobenzoate and *N-tert*-butyl-*N'*-butylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (3% EtOAc in hexane) to give 93.6 mg (75%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.01 (t, 3H,  $J = 7.4$  Hz), 1.61, (s, 9H), 1.73-1.82 (m, 2H), 2.97 (t, 2H,  $J = 7.3$  Hz), 7.98 (d, 2H,  $J = 8.3$  Hz), 8.06 (d, 2H,  $J = 8.3$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.8, 17.6, 28.1, 40.8, 81.7, 127.8, 129.6, 135.6, 139.9, 164.9, 200.0; MS,  $m/e$ : 248 [ $\text{M}^+$ ], 205 [ $^t\text{BuO}_2\text{CC}_6\text{H}_4\text{CO}^+$ ], 175 [ $\text{M}^+ - \text{O}^t\text{Bu}$ ]; Anal. Calcd. for  $\text{C}_{15}\text{H}_{20}\text{O}_3$ : C, 72.55, H, 8.12. Found: C, 72.69; H, 8.40.

#### 4-Phenylacetylbenzoic acid *tert*-butyl ester



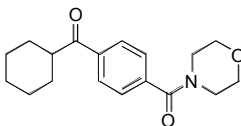
The general procedure was followed with *tert*-butyl 4-bromobenzoate and *N-tert*-butyl-*N*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (6% EtOAc in hexane) to give 134 mg (91%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.60 (s, 9H), 4.30 (s, 2H), 7.25-7.35 (m, 5H), 8.01-8.07 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  28.1, 45.9, 81.8, 127.0, 128.4, 128.7, 129.4, 129.7, 134.0, 135.8, 139.3, 164.8, 197.2; MS,  $m/e$ : 296 [ $\text{M}^+$ ], 205 [ $^t\text{BuO}_2\text{CC}_6\text{H}_4\text{CO}^+$ ]; Anal. Calcd. for  $\text{C}_{19}\text{H}_{20}\text{O}_3$ : C, 77.00; H, 6.80. Found: C, 77.01; H, 6.84.

#### 1-[4-(Morpholine-4-carbonyl)-phenyl]-butan-1-one



The general procedure was followed with (4-bromophenyl)-morpholin-4-yl-methanone and *N-tert*-butyl-*N*-butylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (70% EtOAc in hexane) to give 109 mg (83%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.01 (t, 3H,  $J = 7.4$  Hz), 1.72-1.82 (m, 2H), 2.96 (t, 2H,  $J = 7.3$  Hz), 3.42-3.89 (m, 8H), 7.50 (d, 2H,  $J = 7.8$  Hz), 8.01 (d, 2H,  $J = 7.8$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.6, 17.4, 40.4, 42.3 (broad), 47.9 (broad), 66.6, 127.1, 128.1, 137.7, 139.2, 169.1, 199.4; MS,  $m/e$ : 261 [ $\text{M}^+$ ], 218 [ $\text{M}^+ - \text{C}_3\text{H}_8$ ], 175 [ $\text{M}^+ - \text{C}_4\text{H}_8\text{NO}$ ]; Anal. Calcd. for  $\text{C}_{15}\text{H}_{19}\text{NO}_3$ : C, 68.94; H, 7.33; N, 5.36. Found: C, 68.68; H, 7.61; N, 5.34.

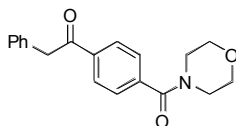
#### Cyclohexyl-[4-(morpholine-4-carbonyl)-phenyl]-methanone



The general procedure was followed with (4-bromophenyl)-morpholin-4-yl-methanone and *N-tert*-butyl-*N*-cyclohexylmethylenhydrazine. The reaction mixture was purified by flash silica gel chromatography (70% EtOAc in hexane) to give 90.1 mg (60%) of the product as white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.22-1.54 (m, 5H), 1.72-1.77 (m, 1H), 1.83-1.91 (m, 4H), 3.24 (tt, 1H,  $J = 11.4, 3.1$  Hz), 3.41-3.80 (m, 8H), 7.49 (d, 2H,  $J = 8.4$  Hz), 7.98 (d, 2H,  $J = 8.4$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  25.8, 25.9, 29.3, 42.5 (broad), 45.8, 48.1 (broad), 66.8, 127.3, 128.6, 137.3, 139.3, 169.4, 203.1;

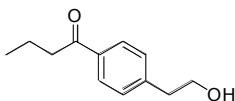
MS, m/e: 301 [ $M^+$ ], 218 [ $M^+ - C_6H_{11}$ ], 215 [ $M^+ - C_4H_8NO$ ]; Anal. Calcd. for  $C_{18}H_{23}NO_3$ : C, 71.73; H, 7.69; N, 4.65. Found: C, 71.47; H, 7.74; N, 4.48.

**1-[4-(Morpholine-4-carbonyl)-phenyl]-2-phenylethanone**



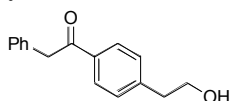
The general procedure was followed with (4-bromophenyl)-morpholin-4-yl-methanone and *N-tert*-butyl-*N*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (70% EtOAc in hexane) to give 136 mg (88%) of the product as white solid.  $^1H$  NMR (400 MHz,  $CDCl_3$ , TMS)  $\delta$  3.39-3.79 (m, 8H), 4.29 (s, 2H), 7.25-7.28 (m, 3H), 7.32-7.35 (m, 2H), 7.48 (d, 2H,  $J = 8.5$  Hz), 8.05 (d, 2H,  $J = 8.5$  Hz);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  42.5 (broad), 45.7, 48.1 (broad), 66.8, 127.1, 127.4, 128.8, 128.9, 129.4, 134.0, 137.4, 139.7, 169.2, 196.8; MS, m/e: 309 [ $M^+$ ], 218 [ $M^+ - C_7H_7$ ]; Anal. Calcd. for  $C_{19}H_{19}NO_3$ : C, 73.77; H, 6.19; N, 4.53. Found: C, 73.48; H, 6.12; N, 4.41.

**1-[4-(2-Hydroxyethyl)-phenyl]-butan-1-one**



The general procedure was followed with [2-(4-bromophenyl)-ethoxy]-*tert*-butyl-dimethylsilane and *N-tert*-butyl-*N*-butylidenehydrazine. After the hydrolysis step, the free alcohol was formed. The reaction mixture was purified by flash silica gel chromatography (45% EtOAc in hexane) to give 75.0 mg (78%) of the product as clear oil.  $^1H$  NMR (400 MHz,  $CDCl_3$ , TMS)  $\delta$  0.99 (t, 3H,  $J = 7.4$  Hz), 1.70-1.79 (m, 2H), 2.31 (br, 1H), 2.89-2.93 (m, 4H), 3.87 (t, 2H,  $J = 6.6$  Hz), 7.31 (d, 2H,  $J = 8.1$  Hz), 7.89 (d, 2H,  $J = 8.1$  Hz);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  13.8, 17.7, 39.0, 40.3, 63.0, 128.2, 129.1, 135.2, 144.3, 200.3; MS, m/e: 191 [ $M^+ - H$ ], 149 [ $M^+ - C_3H_7$ ]; Anal. Calcd. for  $C_{16}H_{16}O_2$ : C, 74.97; H, 8.39. Found: C, 74.74; H, 8.20.

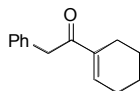
**1-[4-(2-Hydroxyethyl)-phenyl]-2-phenylethanone**



The general procedure was followed with [2-(4-bromophenyl)-ethoxy]-*tert*-butyl-dimethylsilane and *N-tert*-butyl-*N*-phenethylidenehydrazine. After the hydrolysis step, the free alcohol was formed. The reaction mixture was purified by flash silica gel chromatography (45% EtOAc in hexane) to give 117 mg (97%) of the product as white solid.  $^1H$  NMR (400 MHz,  $CDCl_3$ , TMS)  $\delta$  1.66 (br, 1H), 2.89 (t, 2H,  $J = 6.5$  Hz), 3.86 (t, 2H,  $J = 6.5$  Hz), 4.25 (s, 2H), 7.24-7.34 (m, 7H),

7.95 (d, 2H,  $J = 8.3$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  39.1, 45.5, 63.1, 126.9, 128.7, 129.0, 129.3, 129.4, 134.6, 134.9, 144.6, 197.3; MS,  $m/e$ : 239 [ $\text{M}^+ - \text{H}$ ]; Anal. Calcd. for  $\text{C}_{16}\text{H}_{16}\text{O}_2$ : C, 79.97; H, 6.71. Found: C, 79.75; H, 6.67.

**1-Cyclohex-1-enyl-2-phenylethanone<sup>19</sup> [31142-52-6]**



The general procedure was followed with 1-cyclohexenyl trifluoromethanesulfonate and *N-tert*-butyl-*N*-phenethylidenehydrazine. The reaction mixture was purified by flash silica gel chromatography (6% EtOAc in hexane) to give 52.3 mg (52%) of the product as clear oil.  $\delta$   $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  1.36-1.64 (m, 4H), 2.24-2.26 (m, 4H), 3.94 (s, 2H), 7.03 (t, 1H,  $J = 1.7$  Hz), 7.19-7.32 (m, 5H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  21.4, 21.9, 23.2, 26.2, 43.9, 126.5, 128.5, 129.2, 135.5, 139.0, 141.1, 198.6; MS,  $m/e$ : 200 [ $\text{M}^+$ ].

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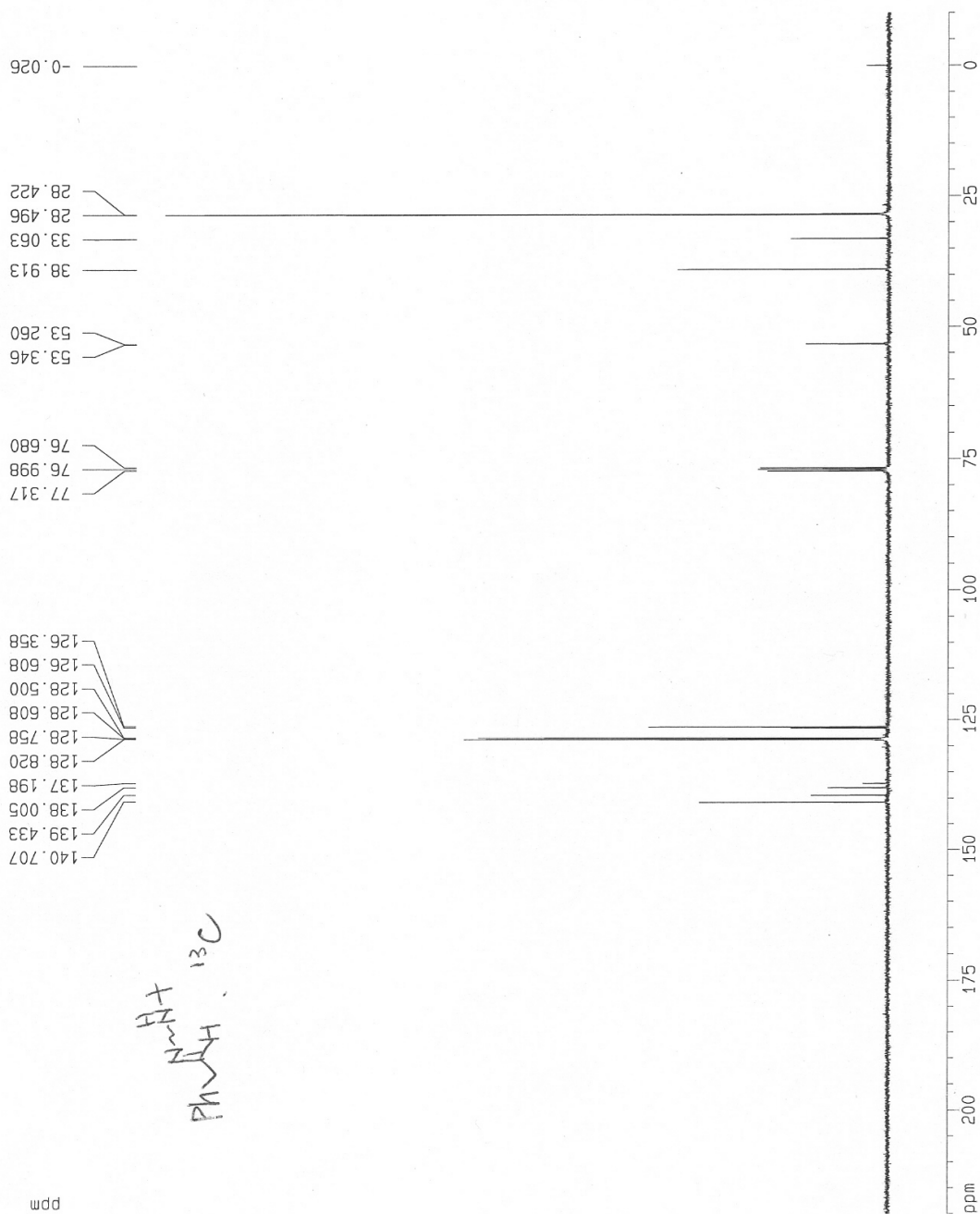
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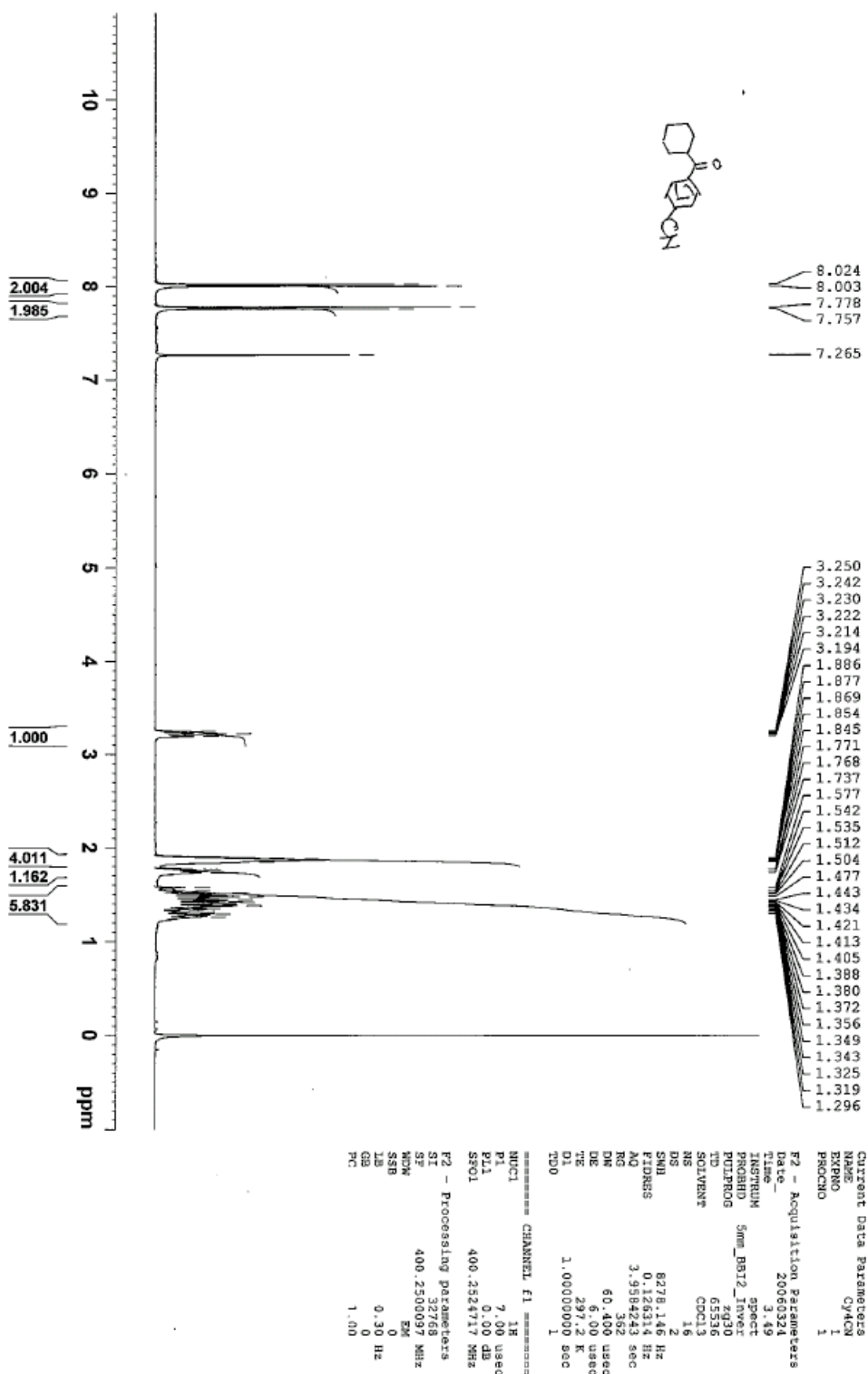
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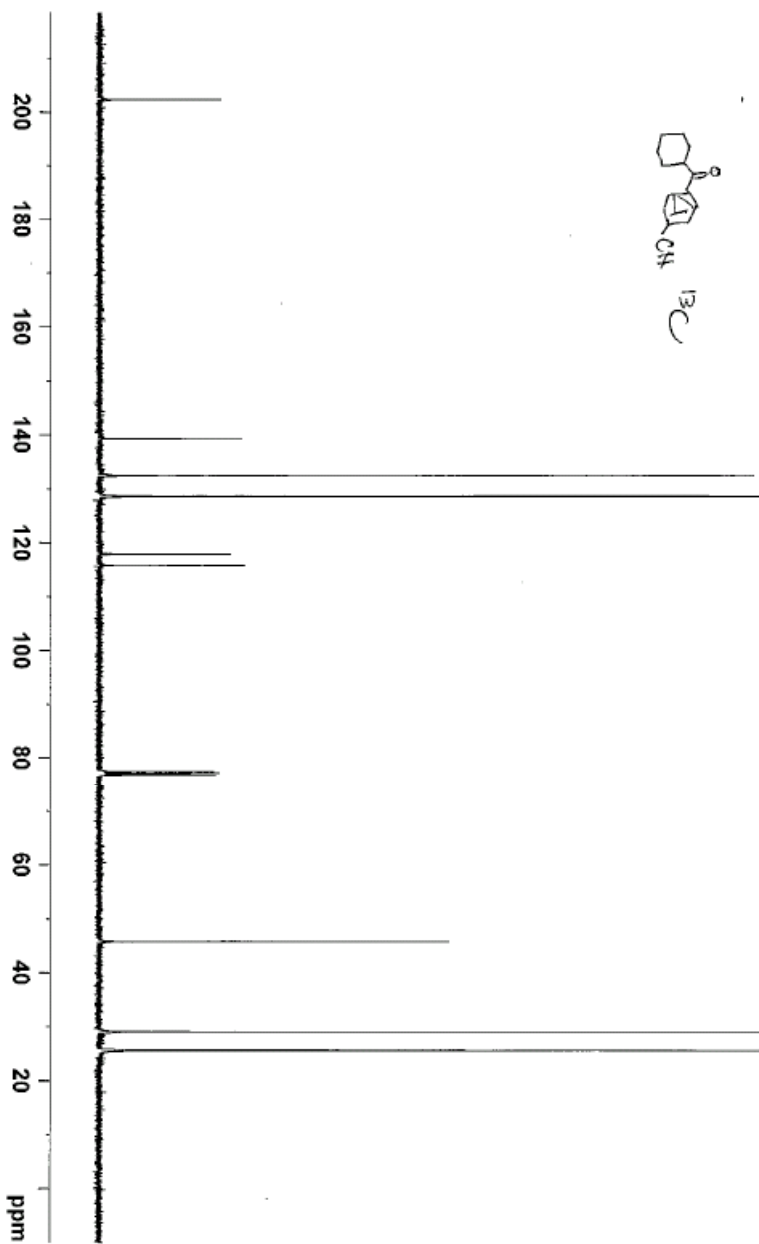
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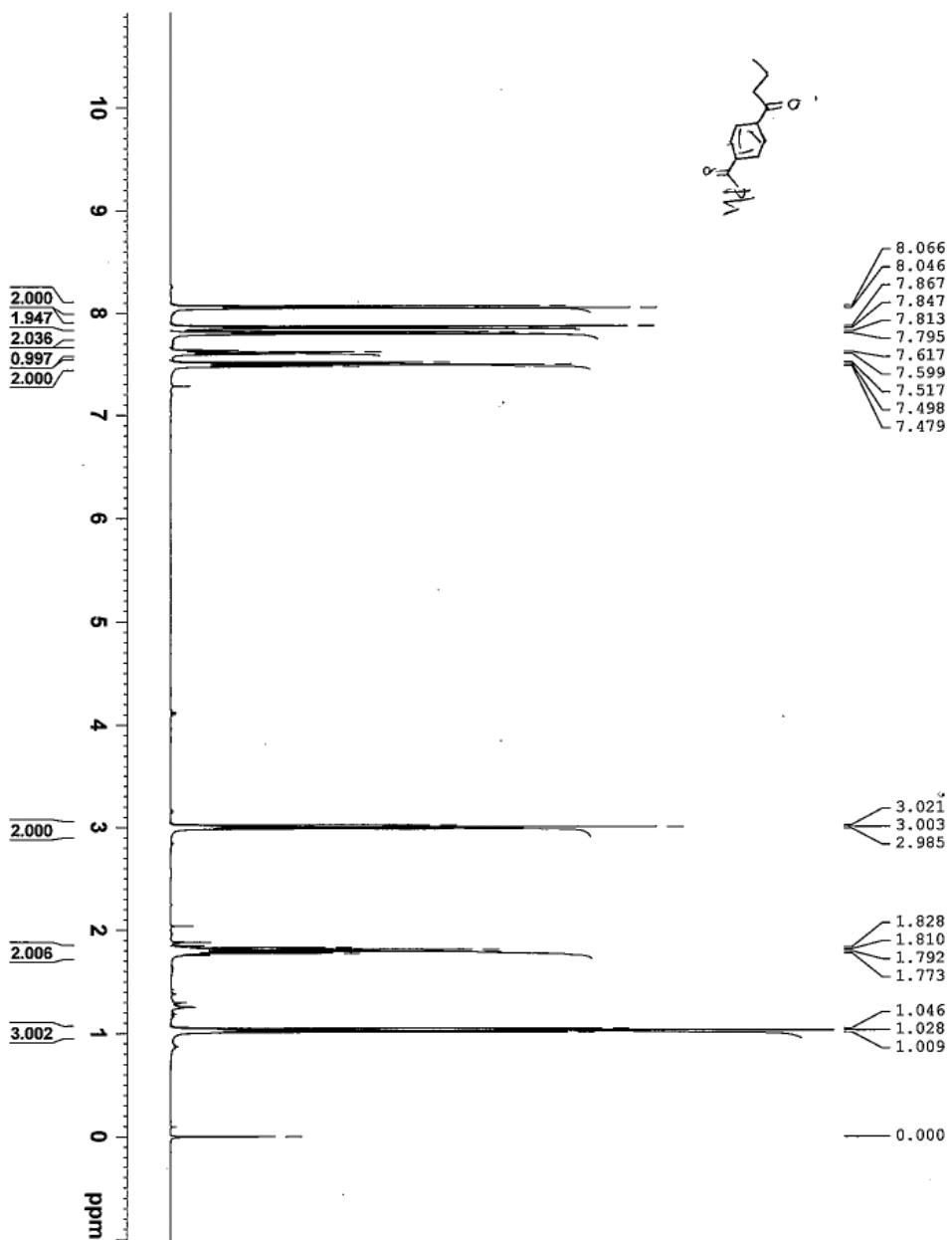
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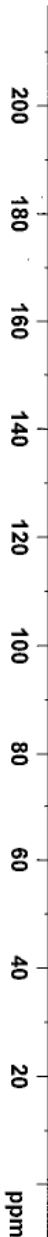
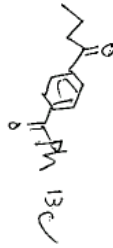


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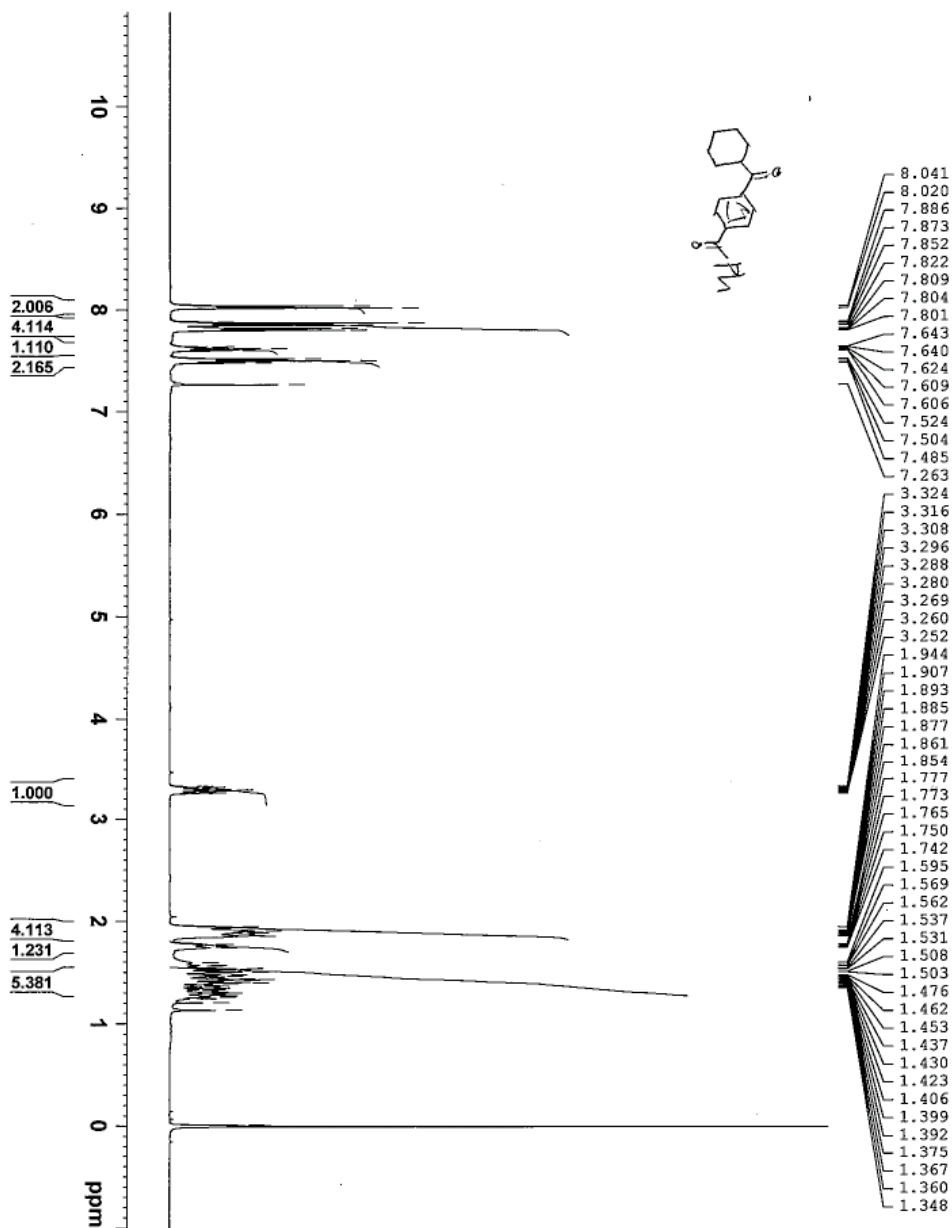
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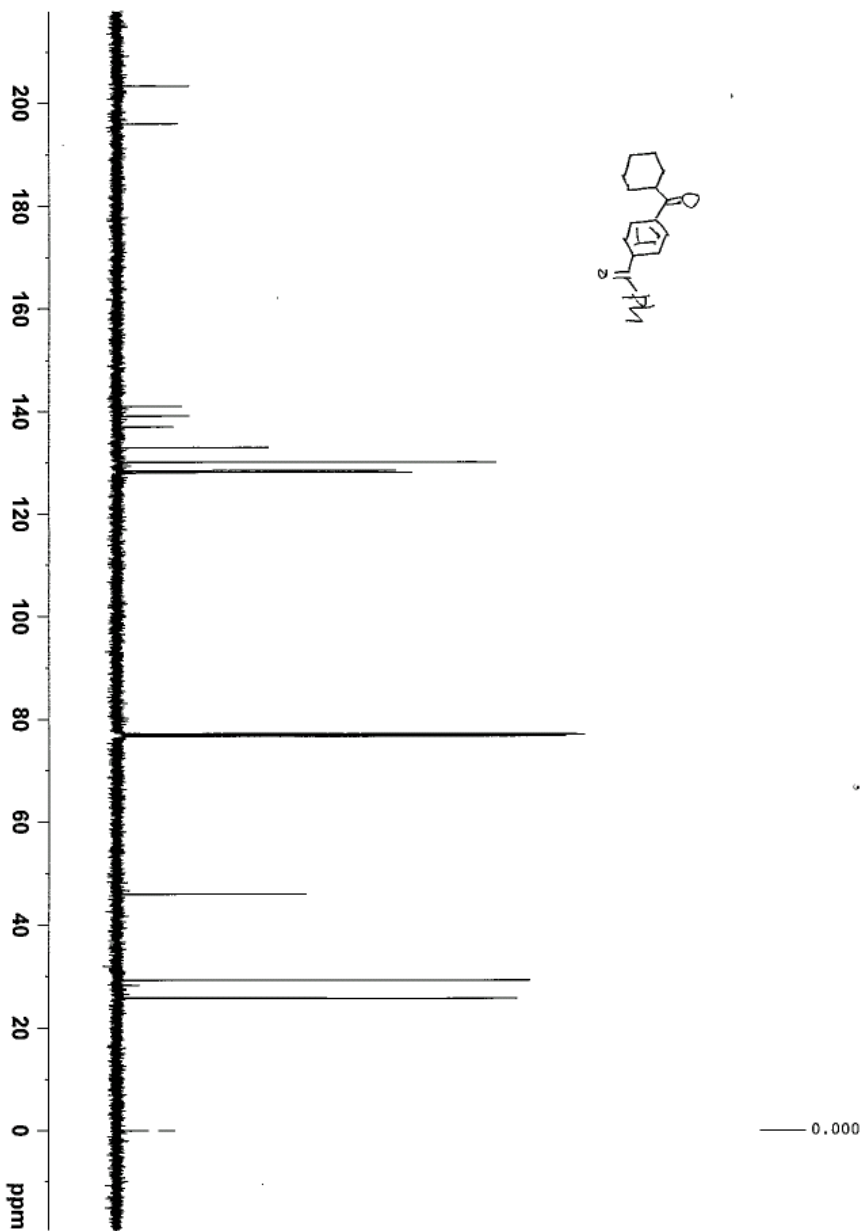


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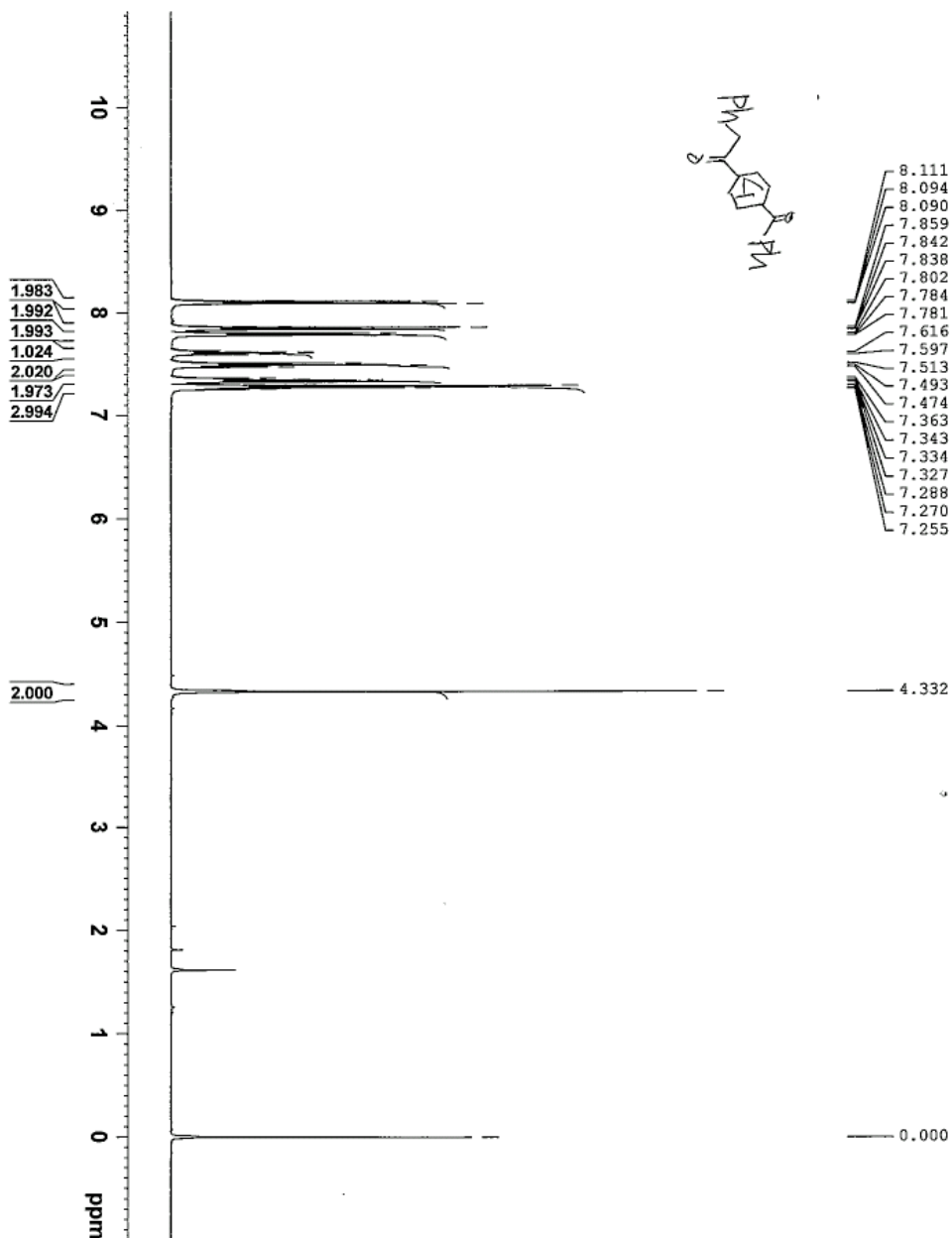
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S25

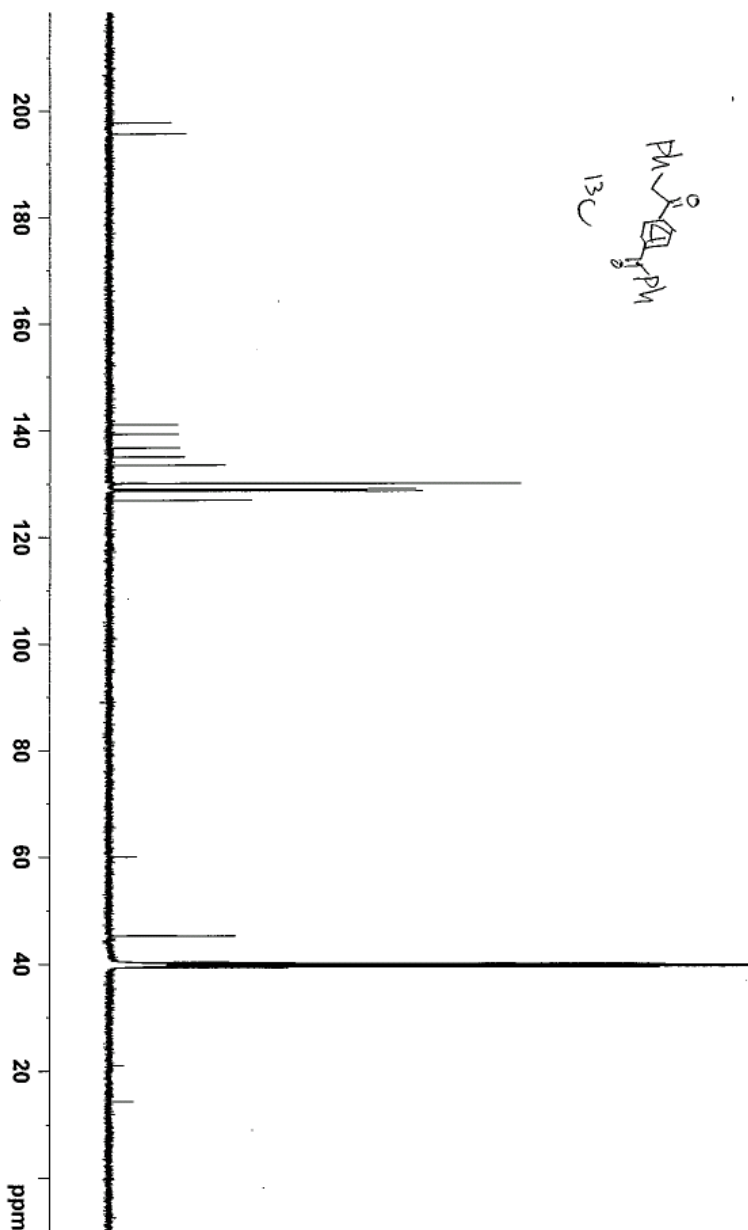
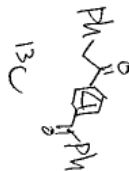


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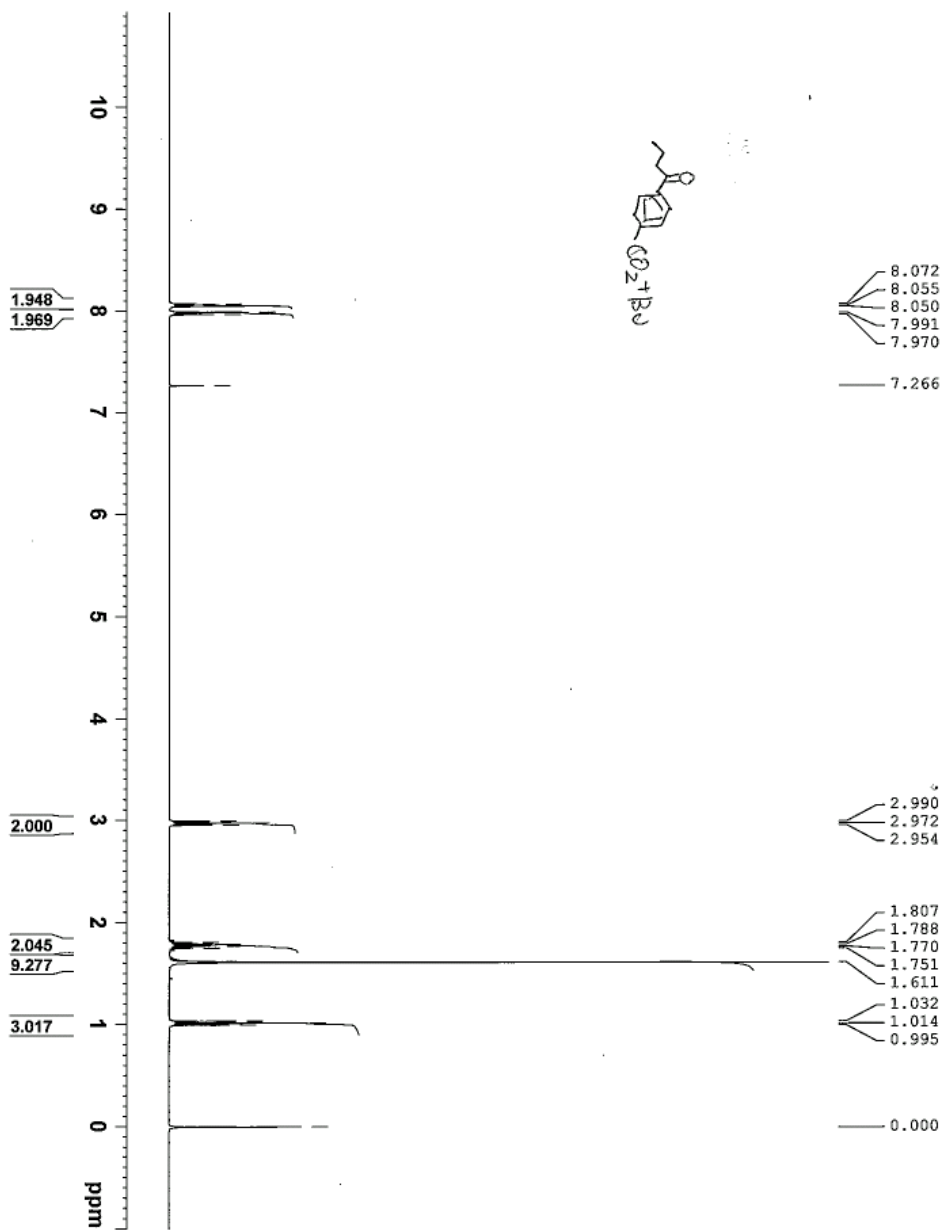


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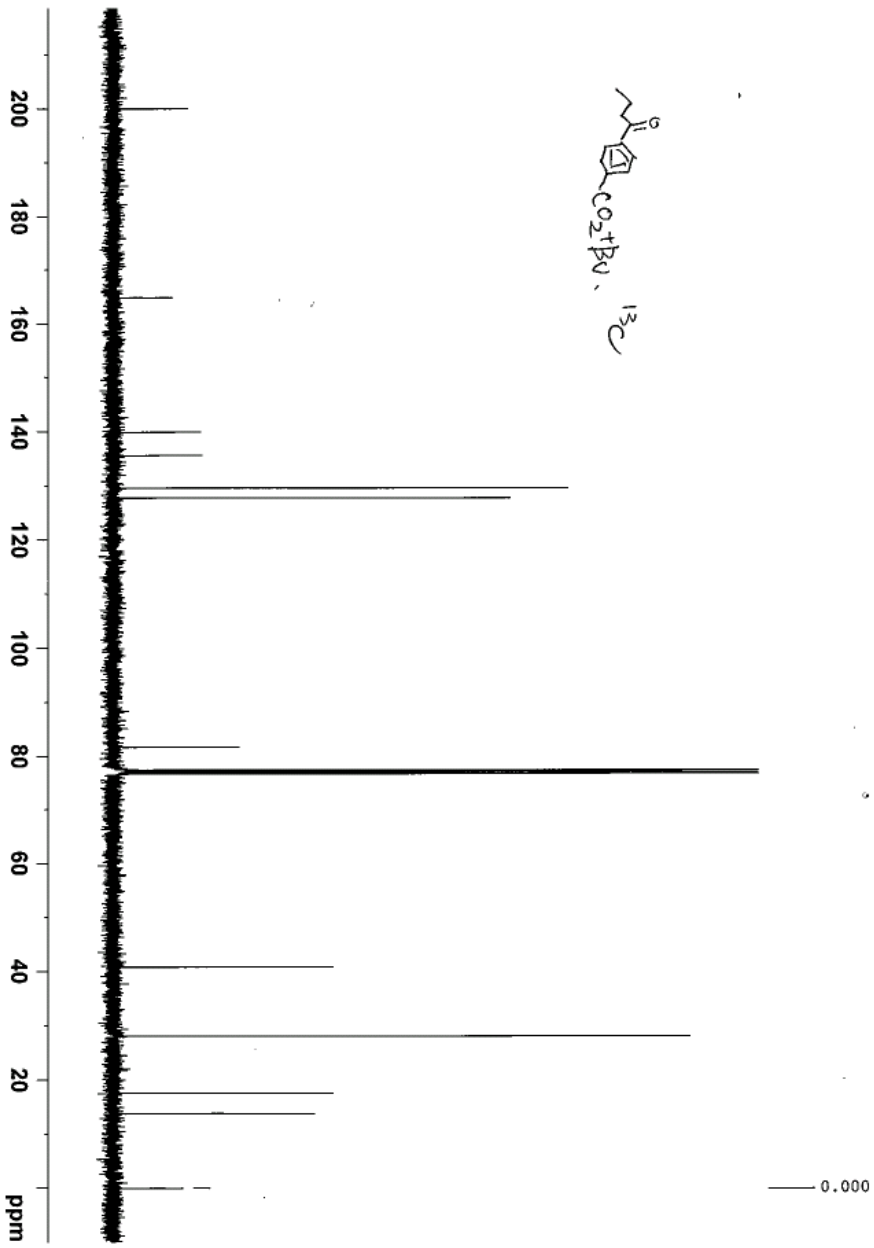
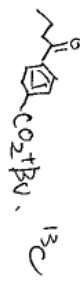
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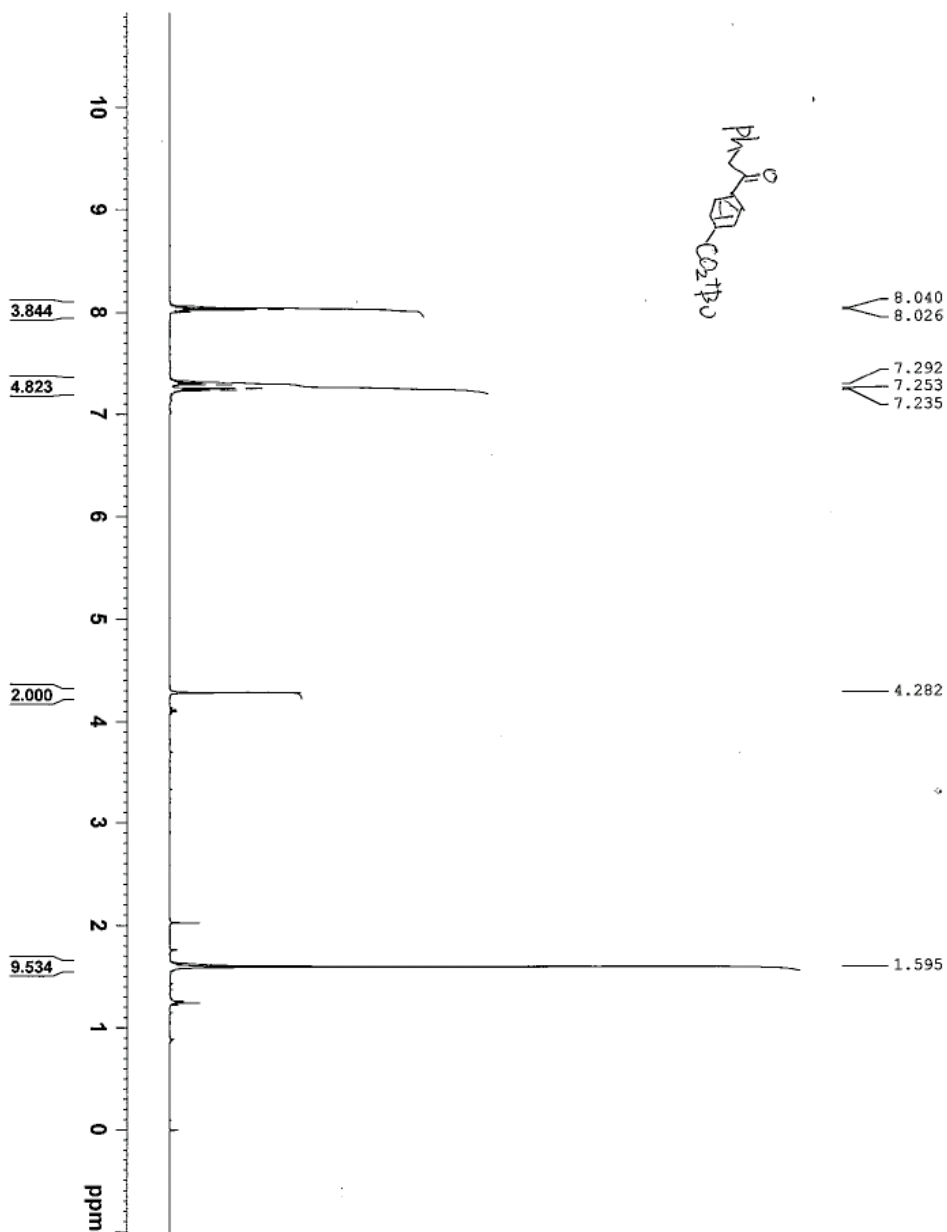


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NUC2 1H  
PCPD2 80.00 usec  
PL2 -6.00 dB  
PL12 18.00 dB

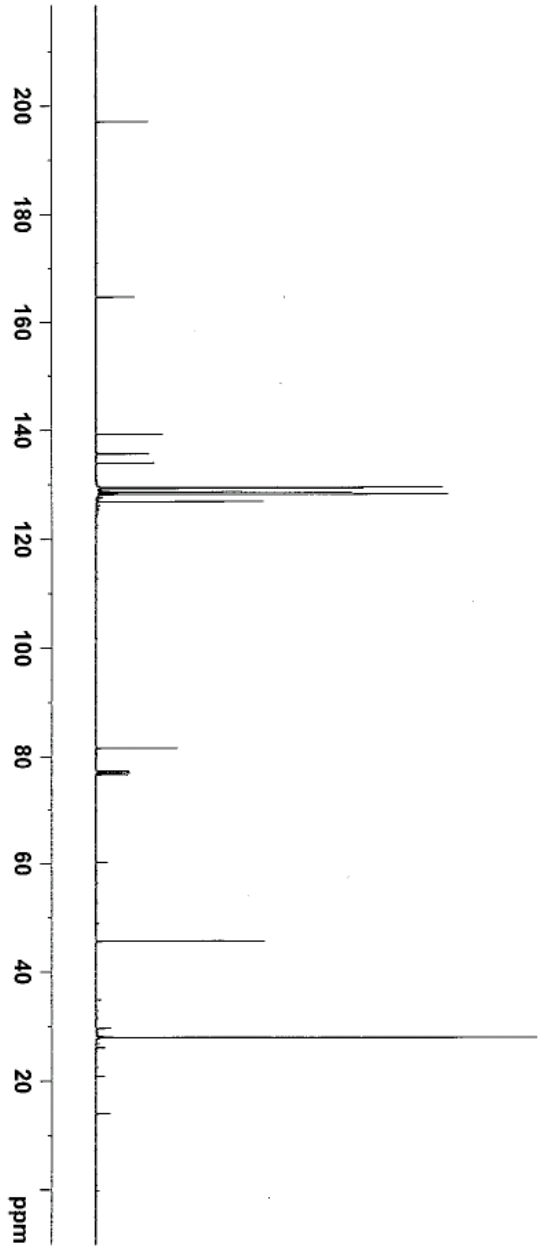
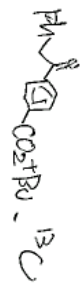


Current Data Parameters  
 NAME BnCOAC02Lbu  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20051210  
 Time\_ 8.11  
 INSTRUM spect  
 PROBDI 5mm\_QNP\_H-P-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.16314 Hz  
 AQ 3.9384243 sec  
 RG 226  
 AS 50.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUCL1 1H  
 P1 6.40 usec  
 PL1 -6.00 dB  
 SFO1 400.0384704 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.0360184 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



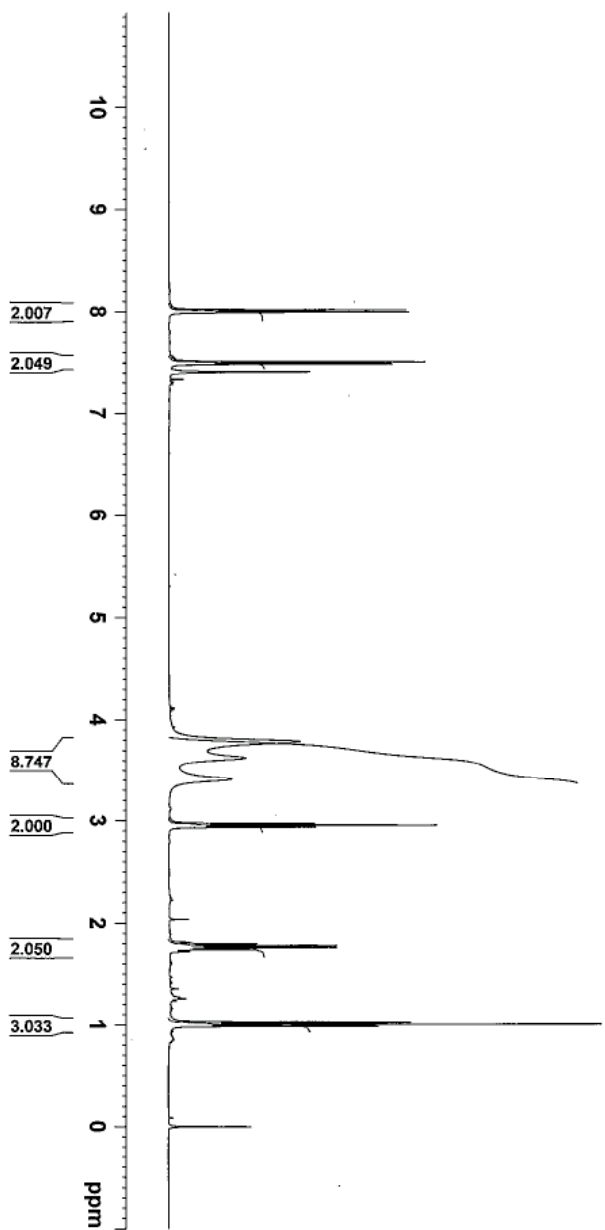
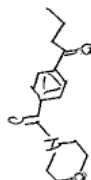
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Current Data Parameters
NAME      BnCO2CH3
EXPNO     2
PROCNO    1

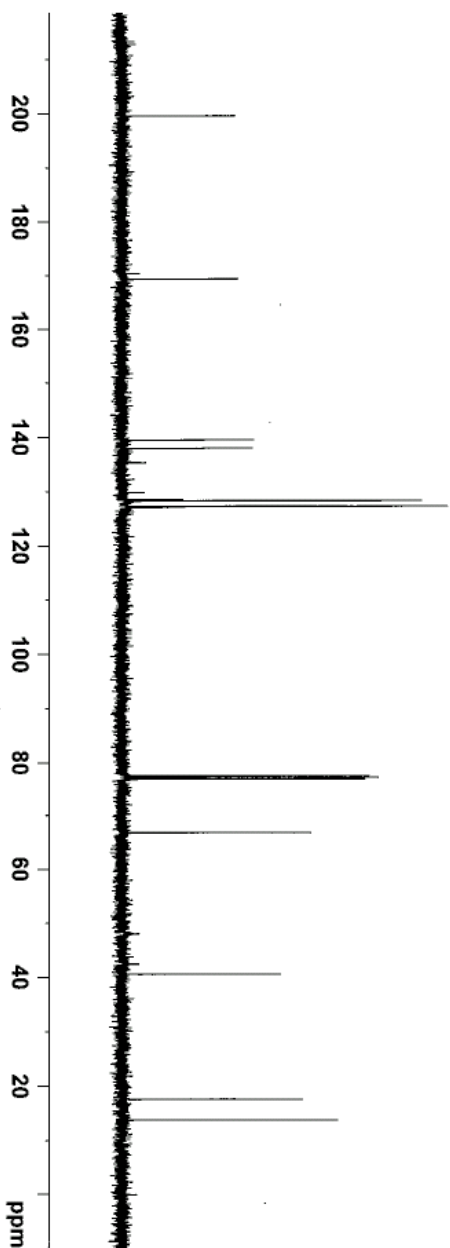
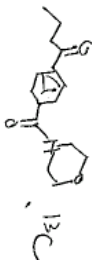
F2 - Acquisition Parameters
Date_     20051210
Time      8.42
INSTRUM   spect
PROBHD    5mm_QNP-1H-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         414
DS         4
SWH        25125.629 Hz
FIDRES     0.383387 Hz
AQ         1.3042164 sec
RG         4096
DN         19.900 usec
DE         6.00 usec
TE         300.0 K
D1         2.00000000 sec
d11        0.03000000 sec
d12        0.0002000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         6.55 usec
PL1        -3.00 dB
SFO1       100.6001570 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -6.00 dB
PL12       18.00 dB
PL13       18.00 dB
  
```



Current Data Parameters  
 NAME PROCARCOMO1  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20060403  
 Time 3:42  
 INSTRUM spect  
 PROBRD 5mm\_BB12\_inver  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 22.8  
 WC 60.7400 usec  
 DM 6400 usec  
 DE 296.2 K  
 TE 1.00000000 sec  
 TDO 1  
 CHANNEL F1  
 NUC1 1H  
 P1 7.00 usec  
 PL1 0.00 dB  
 SFO1 400.2524717 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.249821 MHz  
 MDW 0  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

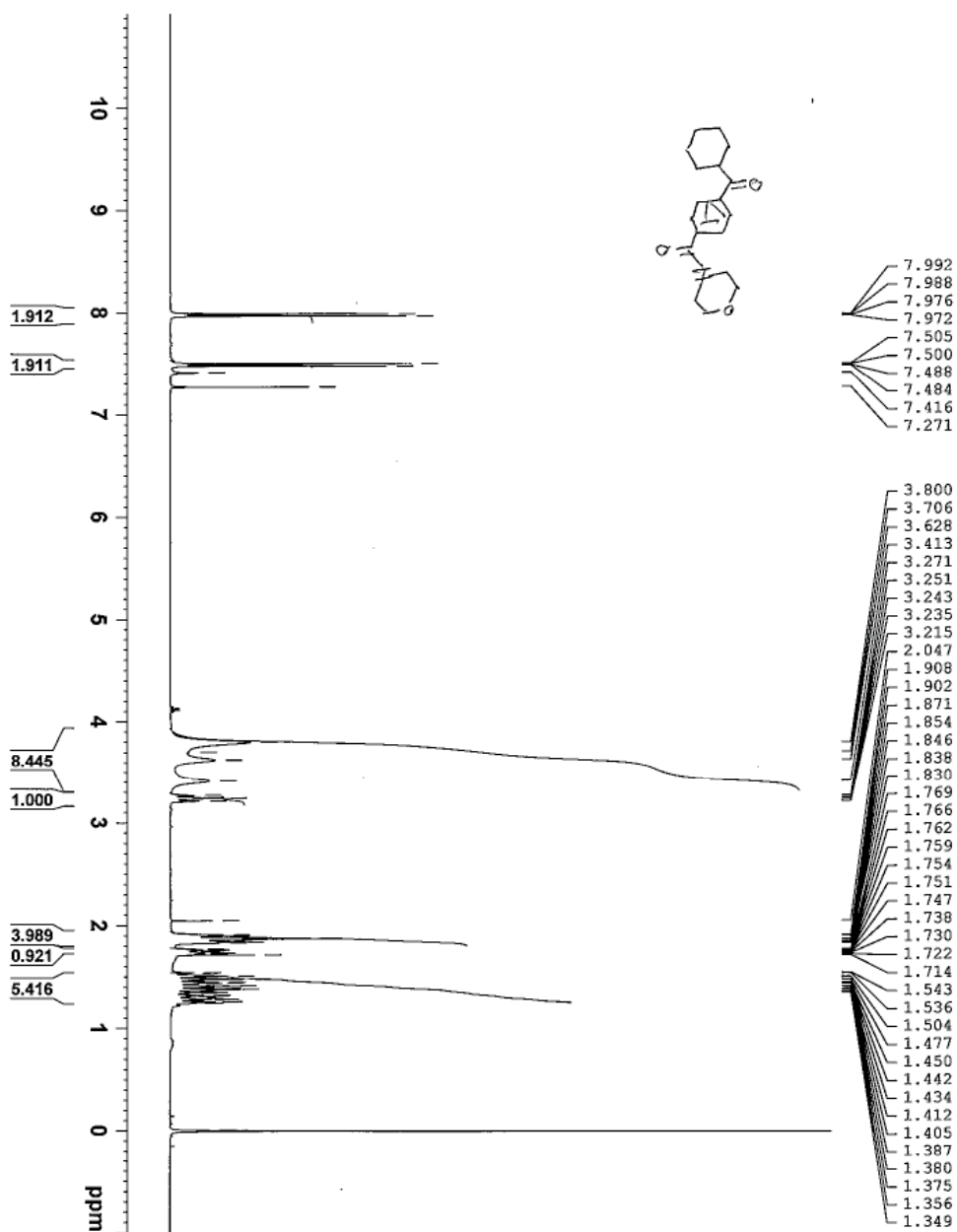


Current Data Parameters  
NAME PRCOA-COMOL  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20060409  
Time 5.45  
INSTRUM spect  
PROBHD 5mm\_BB12  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 65  
DS 4  
SWH 23980.814 Hz  
FIDRES 0.365918 Hz  
AQ 1.3664756 sec  
RG 2298.8  
DW 20.850 usec  
DE 6.00 usec  
TE 296.2 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

CHANNEL F1  
NUC1 13C  
P1 12.00 usec  
PL1 -3.00 dB  
SFO1 100.6530068 MHz

CHANNEL F2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 102.00 usec  
PL2 0.00 dB  
P112 23.00 dB

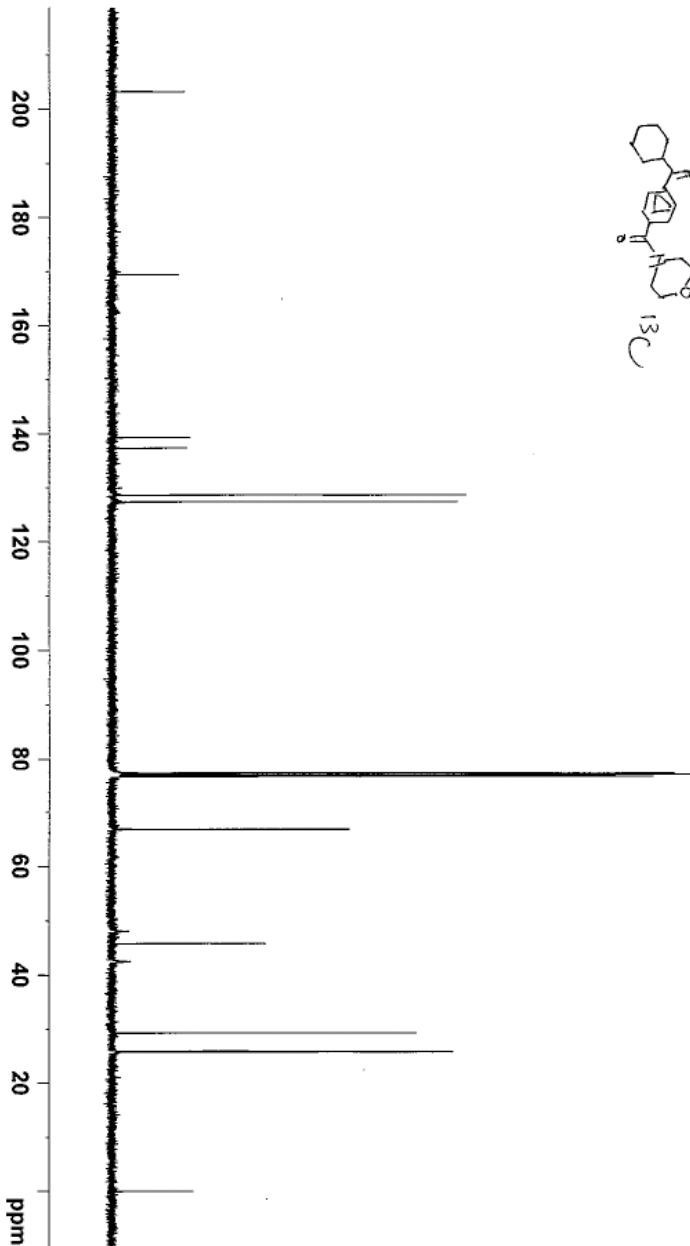
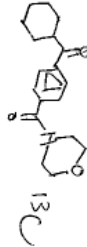


Current Data Parameters  
 NAME Cyclohexanone  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20060415  
 Time 8.05  
 INSTRUM spect  
 PULPROG zgpg30  
 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 7.50 usec  
 TE 295.2 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.40 usec  
 PL1 -6.00 dB  
 SFO1 400.0384704 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.0360083 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

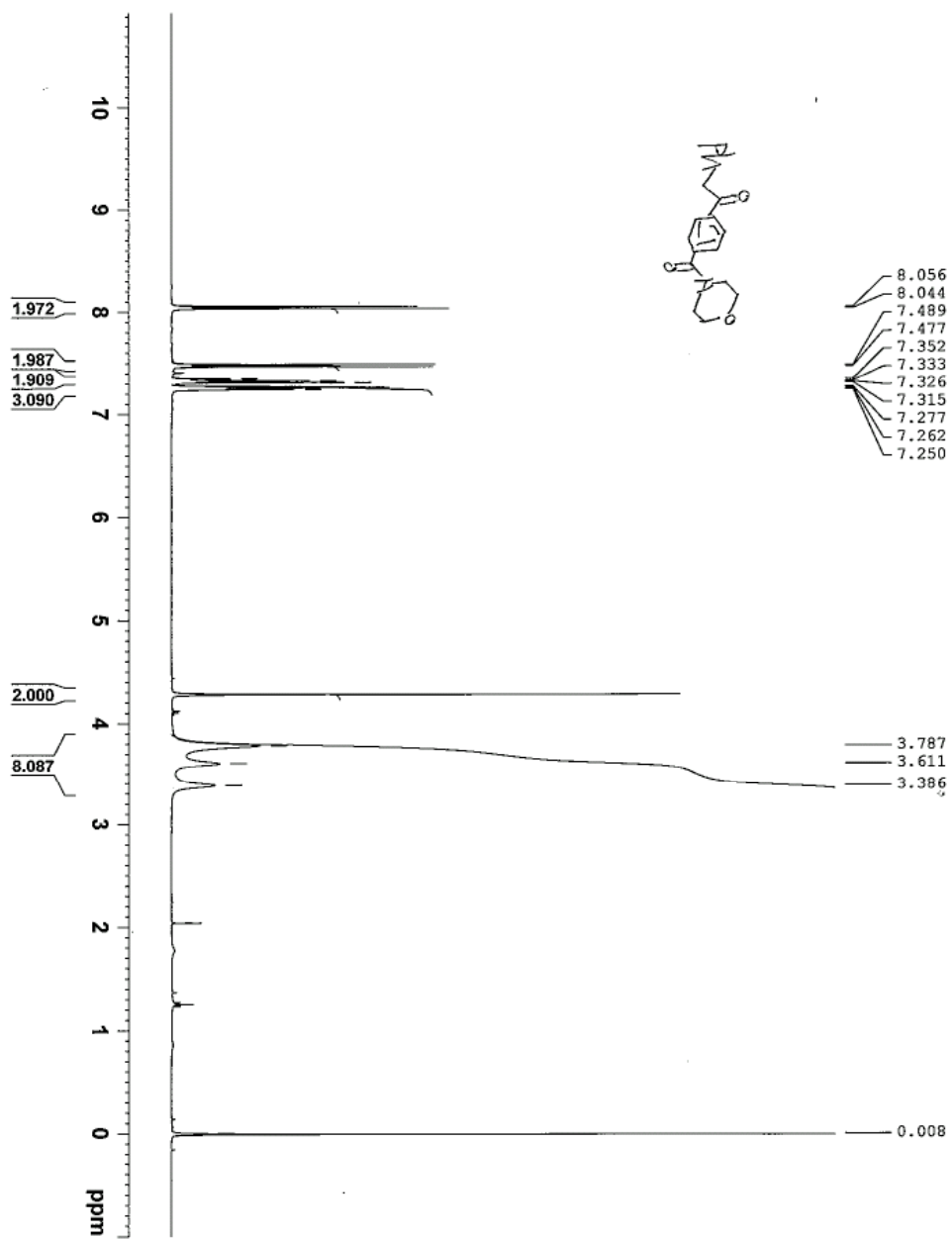


Current Data Parameters  
 NAME CYCOM61  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20060415  
 Time\_ 6.10  
 INSTRUM spect  
 PROBDI Summ\_CMP\_H-E-P-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CCl3  
 NS 838  
 DS 4  
 SWH 23980.814 Hz  
 FIDRES 0.365918 Hz  
 AQ 1.3664796 sec  
 RG 4597.6  
 DW 20.850 usec  
 DE 2.50 usec  
 TE 300.2 K  
 D1 2.0000000 sec  
 d11 0.0300000 sec  
 DELTA 1.8999998 sec  
 TD0 1

===== CHANNEL f1 13C =====  
 NUCL 13C  
 P1 6.60 usec  
 PL1 -3.00 dB  
 SFO1 100.5991911 MHz

===== CHANNEL f2 =====  
 CPROG2 waltz16  
 NUCL2 1H  
 P2 80.00 usec  
 PL2 -6.00 dB  
 P12 18.00 dB

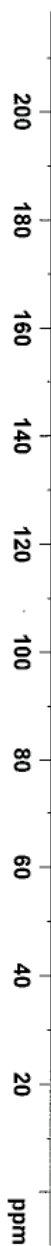
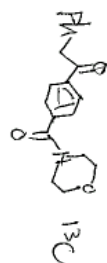


Current Data Parameters  
 NAME BnCOArCOMo1  
 EXNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080415  
 Time\_ 7.01  
 INSTRUM spect  
 PROBD 5mm\_CNP\_H-230  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 128  
 DW 60.409 usec  
 DE 7.59 usec  
 TE 295.2 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUCL1 1H  
 P1 6.40 usec  
 PL1 -6.00 dB  
 SFO1 400.0384704 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.0360113 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



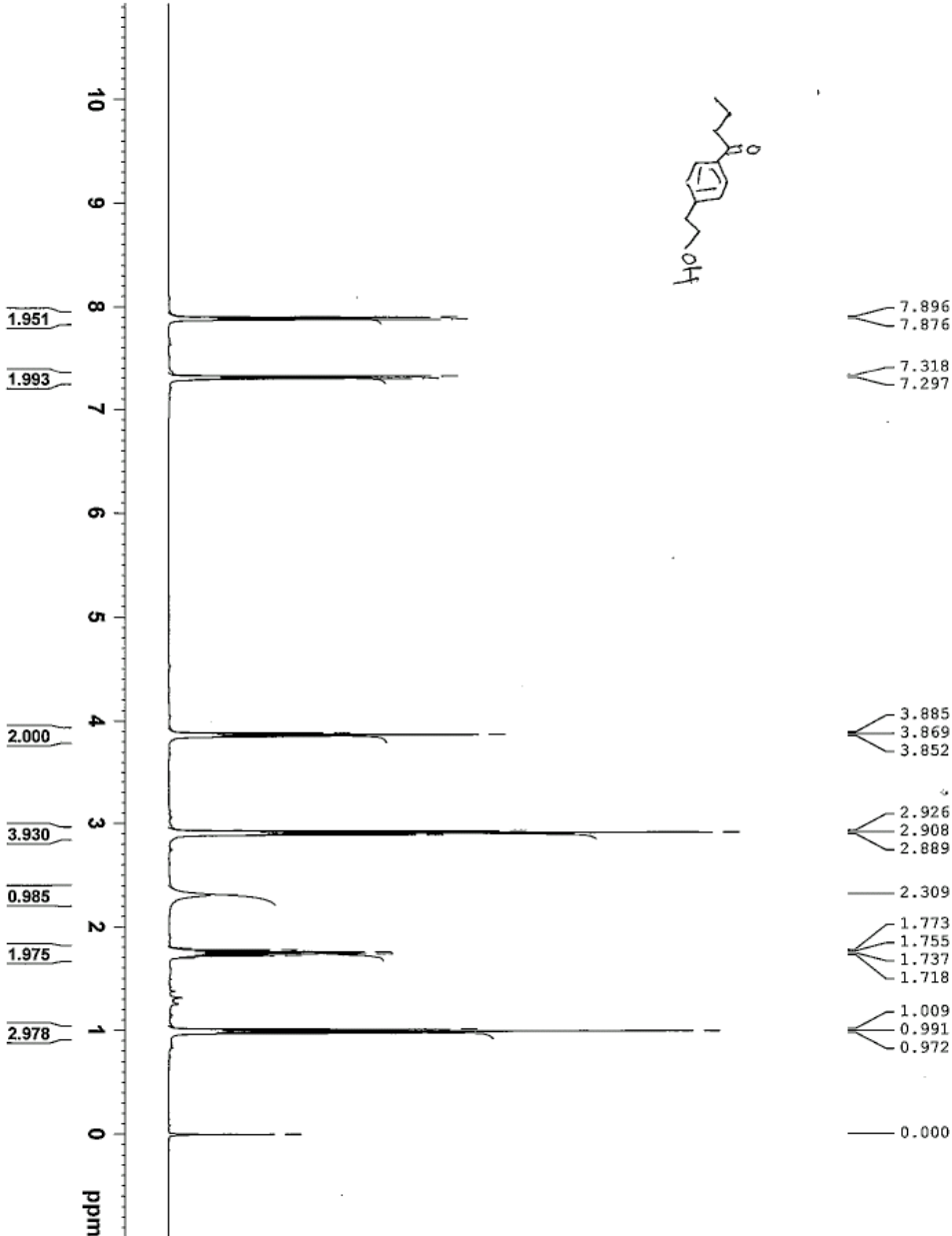
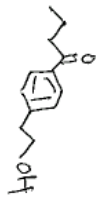
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Current Data Parameters
NAME      BNCORCOMO1
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20060413
Time      17.05
INSTRUM   spect
PROBHD    5mm_QNP-1H-13
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        795
DS        4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         1625.5
DW         20.850 usec
DE         7.50 usec
TE         295.2 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA      1.8939398 sec
TD0        1

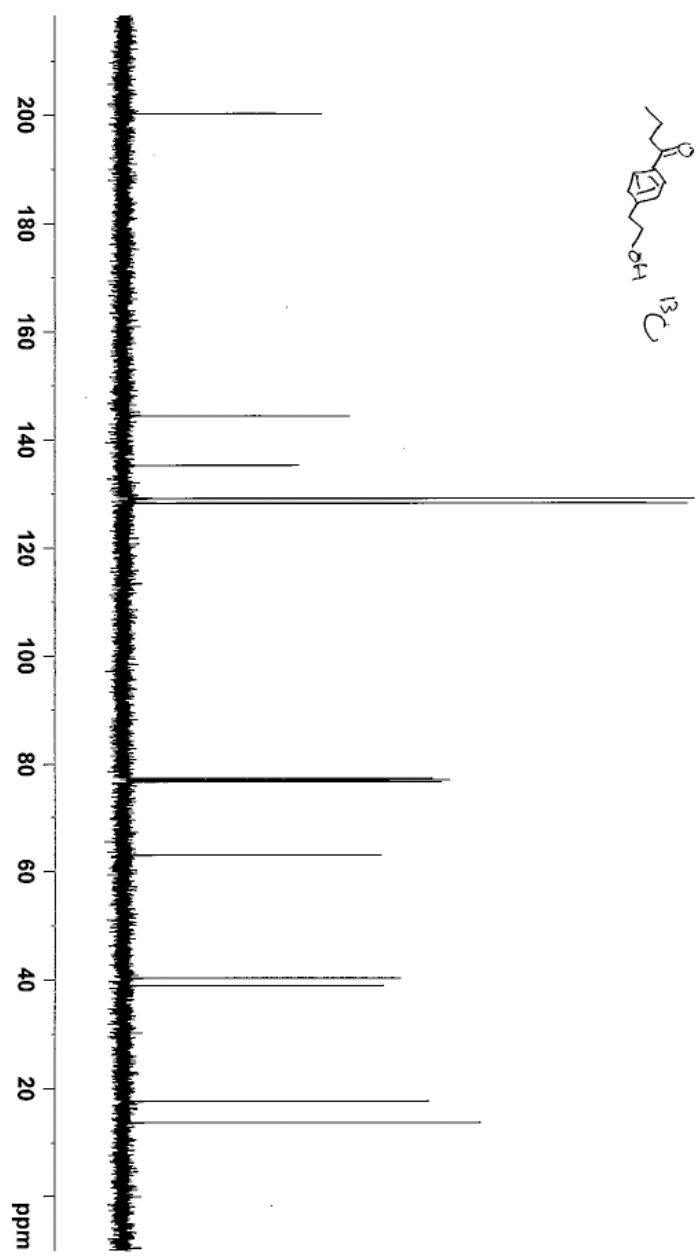
===== CHANNEL f1 =====
NUC1       13C
P1         6.60 usec
PL1        -3.00 dB
SFO1       100.5991911 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -6.00 dB
PL12       18.00 dB
  
```



7.896  
7.876  
7.318  
7.297  
3.885  
3.869  
3.852  
2.926  
2.908  
2.889  
2.309  
1.773  
1.755  
1.737  
1.718  
1.009  
0.991  
0.972  
0.000

Current Data Parameters  
NAME P:COA-CH2CH2OH  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20060416  
Time 2.39  
INSTRUM spect  
PROBHD 5mm\_BBI2\_293K  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 25.4  
DW 60.400 usec  
DE 296.2 K  
TE 1.00000000 sec  
D1 1  
TD0 1  
----- CHANNEL f1 -----  
NUC1 1H  
P1 7.00 usec  
PL1 0.00 dB  
SFO1 400.254717 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.2500020 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

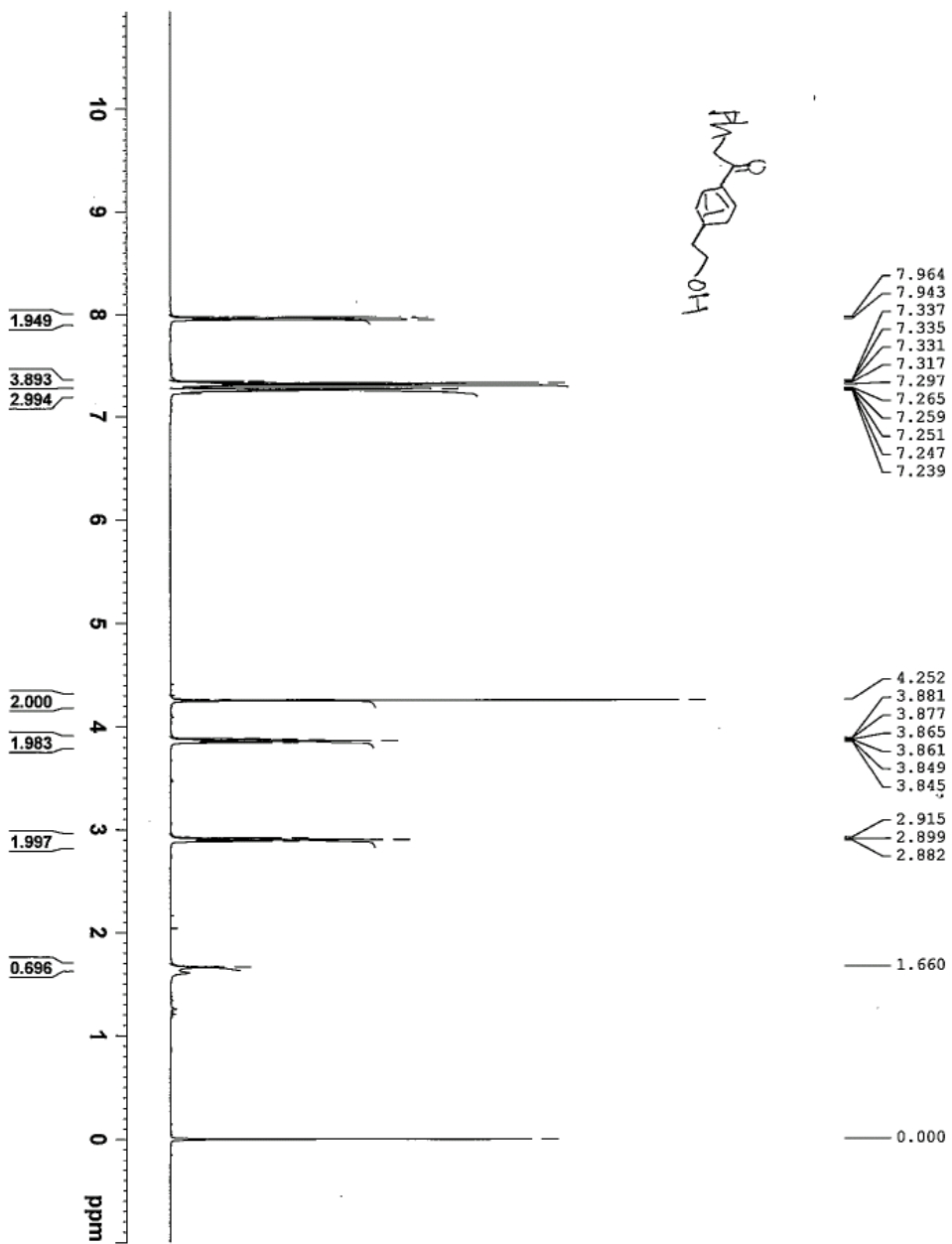


Current Data Parameters  
 NAME PICOARCHECH2OH  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090416  
 Time\_ 12:06  
 INSTRUM spect  
 PROBRD 5mm\_BBI2 Invec  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 107  
 DS 4  
 SWH 23980.814 Hz  
 FIDRES 0.365918 Hz  
 AQ 1.3664756 sec  
 RG 2896.3  
 DW 20.850 usec  
 DE 6.00 usec  
 TE 296.2 K  
 D1 2.0000000 sec  
 d11 0.1300000 sec  
 DELTA 1.8999999 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1  $^{13}\text{C}$   
 P1 12.00 usec  
 PL1 -3.00 dB  
 SFO1 100.6530068 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2  $^1\text{H}$   
 P2 102.00 usec  
 PL2 0.00 dB  
 P12 23.00 MHz

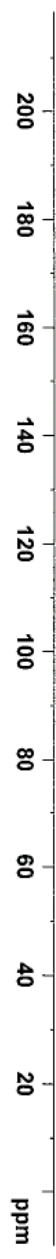
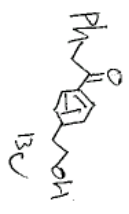


Current Data Parameters  
 NAME BmCOARCH2CH2OH  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20060421  
 Time\_ 7.02  
 INSTRUM spect  
 PROBU 5mm\_QNP\_H-  
 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  
 DB 7.50 usec  
 DE 235.2 K  
 TE 1.0000000 sec  
 D1 1  
 TDO

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.40 usec  
 PL1 -6.00 dB  
 SFO1 400.0384704 MHz

F2 - Processing Parameters  
 SI 32768  
 SF 400.0360158 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



```

Current Data Parameters
NAME      BnCOA1CH2CH2OH
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20060421
Time      7.08
INSTRUM   spect
PROBHD    5mm QNP 1H-13
PULPROG   zgpg30
TD         65536
SOLVENT    CDCl3
NS         326
DS         4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664796 sec
RG         1448.2
DW         20.850 usec
DE         7.50 usec
TE         295.2 K
D11        2.0000000 sec
d11        0.0300000 sec
DELTA      1.8993939 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         6.60 usec
PL1        -3.00 dB
SFO1       100.5991911 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -6.00 dB
PL12       18.00 dB
  
```