# Three-Component Reaction Discovery Enabled by Mass Spectrometry of Self-Assembled Monolayers

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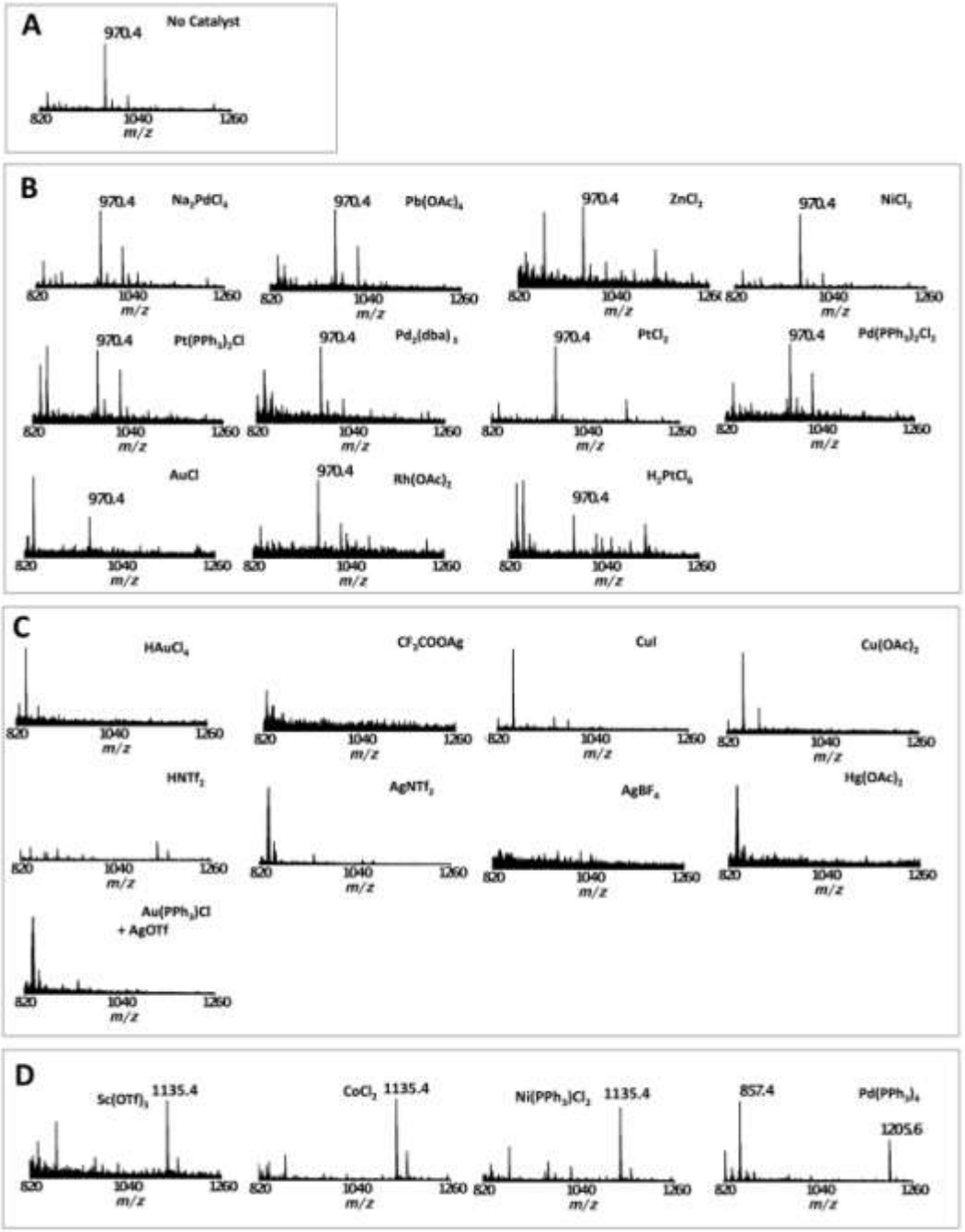
## Supplementary Information

# 1. SAMDI Reaction Screening

## Mass Spectrometry Data from SAMDI Screens




**Supplementary Figure S1**. Representative MALDI MS data of the three-component reaction screen. **A**: MALDI MS spectrum of the aldehyde-terminated monolayer, which was treated with 1-siloxy-1-hexyne (**2**) and 4-methoxy-

N-methyl aniline (**3**) in tetrahydrofuran, shows a major peak at m/z 970.4. **B**: MALDI MS spectra of the aldehyde-terminated monolayer after treatment with siloxy alkyne **2** and aniline **3** in the presence of several representative additives show major peaks at m/z 970.4, which is indicative of unreacted aldehyde. **C**: MALDI MS spectra of the aldehyde-terminated monolayer after treatment with siloxy alkyne **2** and aniline **3** in the presence of several additional additives shows disappearance of the peak at m/z 970.4 without formation of any new peaks. **D**. MALDI MS spectra of the aldehyde-terminated monolayer after treatment with siloxy alkyne **2** and aniline **3** in the presence of four catalysts, which resulted in formation of new major peaks. The peak at m/z 1135.4 (three spectra on the left) cannot be assigned at this point. The peaks at m/z 857.4 and 1205.6 (spectrum on the right) correspond to the sodium adduct of an alkanethiolate terminated in a three-component product following loss of the triisopropylsilyl (TIPS) group as well as the sodium adduct of a mixed disulfide produced from this three-component product and the background methylether-terminated alkanethiolate.

### Materials for SAMDI Screening

Reagents and anhydrous solvents for SAMDI screening were used as received without further purification. Glass slides for gold deposition were obtained from Fisher Scientific. (Tridecafluoro-1,1,2,2-tetrahydrooctyl)-1-trichlorosilane was purchased from Pfaltz & Bauer (Waterbury, CT). Glove bags were purchased from Glas-Col (Terre Haute, IN). Amino-EG6-undecanethiol was purchased from Dojindo (Rockville, MD). The methyl ether-terminated alkanethiol with a tri(ethylene glycol) spacer was synthesized according to a previously reported procedure.[1]

## 2. Three Component Reaction Scope Studies

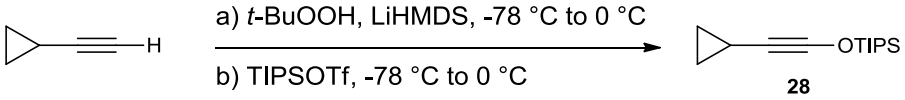
### General Procedures for Organic Synthesis

Hexanes (ACS grade), ethyl acetate (ACS grade), diethyl ether (ACS grade), and toluene (anhydrous) were purchased from Fisher Scientific and used without further purification. Tetrahydrofuran was distilled from sodium-benzophenone under a positive pressure of nitrogen. Unless otherwise noted, all reactions were performed under an inert atmosphere of nitrogen in flame-dried (10 x 75 mm) test tubes equipped with a stirbar and fitted with a rubber septum. Triisopropylsilyl trifluoromethanesulfonate (TIPSOTf) was distilled under reduced pressure over calcium hydride. 4-Methoxy-*N*-methylaniline was purified by flash chromatography prior to use (hexanes to 95:5 hexanes: EtOAc). Other commercially available reagents were obtained from Sigma-Aldrich, Strem, or Alfa Aesar Lancaster Synthesis and were used without further purification unless otherwise noted. Solution-phase reactions were monitored by thin layer chromatography (TLC) using Whatman precoated silica gel plates. Flash column chromatography was performed over Silacycle silica gel (230-400 mesh). $^1$H NMR spectra were recorded on a Bruker DMX-500 instrument. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance resulting from incomplete deuteration as the internal standard ($CH_3CN$: $\delta$ 1.94 or $CDCl_3$: $\delta$ 7.26). $^1$H NMR spectra for three-component products were recorded at elevated temperatures to ensure the appearance of all proton signals. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, ddd = doublet of doublet of doublets, br = broad, m = multiplet), coupling constants in

[1] Yeo, W. S.; Mrksich, M. *Langmuir* **2006**, *22*, 10816-10820.

Hertz (Hz), and integration. $^{13}C$ NMR spectra were recorded on a Bruker DMX-500 instrument using residual solvent peaks as internal standards ($CH_3CN$: δ 118.69 or $CDCl_3$ δ 77.23). High resolution mass spectra were recorded with a Waters Q-TOF Ultima tandem quandrupole/time-of-flight instrument.

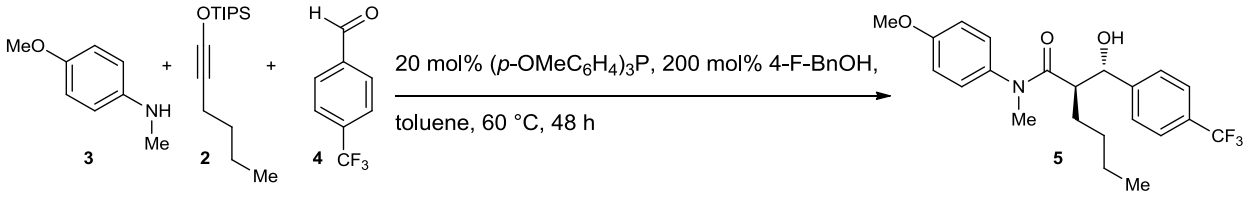
**Preparation of Siloxy Alkynes**



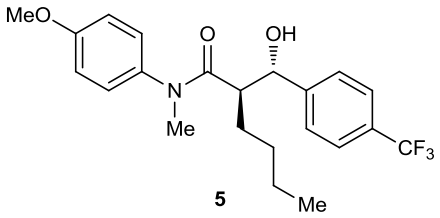

**((cyclopropylethynyl)oxy)triisopropylsilane** (**28**).
A 500-mL flame-dried, three-necked, round-bottomed flask equipped with a stir bar, fitted with rubber septa and a nitrogen inlet was charged with THF (125 mL) and cyclopropylacetylene (3.22 mL, 38.0 mmol). The resulting solution was cooled to -78 °C and anhydrous *t*-BuOOH (7.23 mL of a 5.78 M solution in nonane, 41.8 mmol) was added dropwise over a period of 10 minutes. CAUTION! SOLUTIONS OF OXIDANTS AND OXIDIZABLE SUBSTRATES ARE POTENTIALLY HAZARDOUS AND POSSIBLY SUBJECT TO VIOLENT DECOMPOSITION BY ADVENTITIOUS CATALYSIS. Freshly prepared LiHMDS (95.0 mL of a 1M solution in THF, 95.0 mmol) was added to the resulting mixture via syringe pump over a period of 30 minutes. The mixture was allowed to warm to 0 °C in an ice water bath, and was stirred for 2 h at this temperature. The reaction mixture was then cooled to -78 °C, treated dropwise over a period of 10 min with TIPSOTf (11.23 mL, 41.8 mmol), and was allowed to stir for 5 min at this temperature. The reaction vessel was transferred to a 0 °C ice water bath and was allowed to stir for an additional 40 minutes before being diluted with hexanes (200 mL). The resulting mixture was transferred to a separatory funnel and was washed with saturated aqueous $NaHCO_3$ (150 mL). The organic layer was collected, and the aqueous layer was extracted with hexanes (2 x 50 mL). The combined organic layers were washed with saturated aqueous $Na_2S_2O_3$ (125 mL) and brine (100 mL). The organic layer was collected, dried with $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by Kugelrohr distillation affording **28** as a clear oil (7.46 g, 31.3 mmol, 82 % yield). The synthesis and characterization of other siloxy alkynes used in this study has been previously reported.[2,3,4,5] $^1H$ NMR (500 MHz, $CDCl_3$) δ 1.27-1.20 (m, 3H), 1.10 (d, $J$ = 7.0 Hz, 18H), 1.05-1.03 (m, 1H), 0.62-0.58 (m, 2H), 0.46-0.42 (m, 2H); $^{13}C$ NMR (125 MHz, $CDCl_3$) 82.7, 34.3, 17.5, 12.0, 7.9, -1.4; HRMS ($EI^+$) calculated for $C_{14}H_{27}OSiF$ $[M]^+$ 238.1753, found 238.1766.

[2] Schramm, M. P.; Shubinets, V.; Kozmin, S. A. *Org. Synth*. **2010**, *87*, 253-263.
[3] Sun, J.; Keller, V. A.; Meyer, S. T.; Kozmin, S. A. *Adv. Synth. Catal*. **2010**, *352*, 839-842.
[4] Sun, J.; Kozmin, S. A. *Angew. Chem. Int. Ed*. **2006**, *45*, 4991-4993.
[5] Sweis, R.; Schramm, M. P.; Kozmin, S. A. *J. Am. Chem. Soc*. **2004**, *126*, 7442-7443.

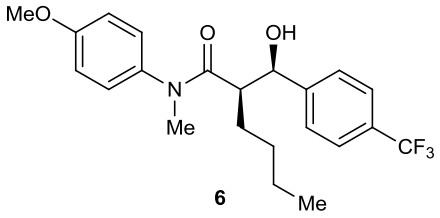
**Synthesis and Characterization of Three-Component Products**




All three-component products were synthesized according to the general procedure detailed in the methods section of the manuscript. For the reaction of 4-trifluoromethylbenzaldehyde with siloxy alkyne **2** and aniline **3**, both diastereomers of the product were isolated and characterized. For all other three-component reactions, only the major, *anti*-diastereomers were fully characterized due to the difficulty of obtaining sufficient amounts of the purified minor diastereomers.

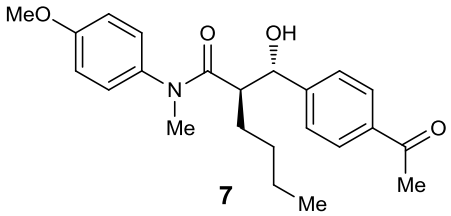



**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-5**). Following the standard procedure for three-component condensations, the reaction of 4-(trifluoromethyl)benzaldehyde (136.6 μl, 1.00 mmol) afforded ***anti*-5** as a white solid (178.2 mg, 0.435 mmol, 87% yield) after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). Crystallization of **5** was performed using hexanes:EtOAc (50:50). mp: 130-132 °C; $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.65 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.83 **(**d, *J* = 8.5, 2H), 6.71-5.54 (m, 2H), 4.99 (d, *J* = 7.5 Hz, 1H), 4.71 (dd, *J* = 7.0, 5.0 Hz, 1H), 3.80 (s, 3H), 3.08 (s, 3H), 2.64-2.59 (m, 1H), 1.75-1.66 (m, 1H), 1.54-1.45 (m, 1H), 1.24-1.12 (m, 4H), 0.84 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.0, 160.3, 150.5, 137.3, 130.1 (q, *J* = 31.8 Hz), 130.1, 128.1, 126.5 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.5 Hz), 115.7, 75.6, 56.6, 48.9, 37.9, 31.5, 30.4, 23.7, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_3F_3$ $[M+H]^+$ 410.1943, found 410.1950.



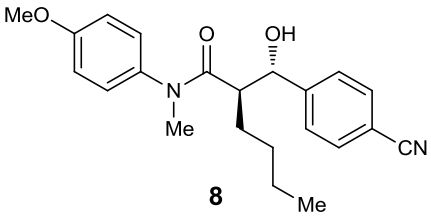

**(*RS*)-2-((*RS*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***syn*-6**). Following the standard procedure for three-component condensations, the reaction of 4-(trifluoromethyl)benzaldehyde (136.6 μl, 1.00 mmol) afforded ***syn*-6** as a white solid (5.5 mg, 0.013 mmol, 3% yield) after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc) and preparative TLC (70:30

hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.61 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.92 (d, *J* = 9.0 Hz, 2H), 6.86-6.78 (m, 2H), 4.93-4.89 (m, 1H), 4.10 (d, *J* = 2.5 Hz, 1H), 3.82 (s, 3H), 3.13 (s, 3H), 2.63-2.57 (m, 1H), 1.75-1.65 (m, 1H), 1.41-1.33 (m, 1H), 1.22-1.05 (m, 3H), 1.02-0.92 (m, 1H), 0.79 (t, *J* = 7.5 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) 175.8, 160.4, 149.1, 137.5, 130.2, 129.9 (q, *J* = 31.8 Hz), 128.0, 126.2 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.4 Hz), 115.9, 74.7, 56.6, 50.0, 38.0, 30.7, 28.5, 23.9, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_3F_3$ $[M+H]^+$ 410.1943, found 410.1946.



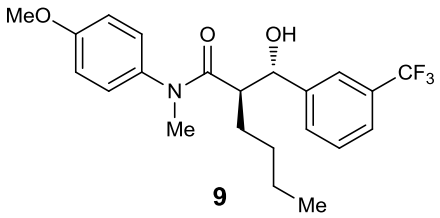

**7**

**(*RS*)-2-((*SR*)-(4-acetylphenyl)(hydroxy)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-7**). Following the standard procedure for three-component condensations, the reaction of 4-acetylbenzaldehyde (148.2 mg, 1.00 mmol) afforded ***anti*-7** as a white solid (167.2 mg, 0.436 mmol, 87% yield) after purification by flash chromatography (hexanes to 4:1 hexanes:EtOAc) $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.92 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.70-6.58 (m, 2H), 4.94 (d, *J* =7.5 Hz, 1H), 4.69 (dd, *J* = 7.5, 5.0 Hz, 1H), 3.80 (s, 3H), 3.08 (s, 3H), 2.71-2.61 (m, 1H), 2.58 (s, 3H), 1.73-1.66 (m, 1H), 1.52-1.48 (m, 1H), 1.22-1.10 (m, 4H), 0.83 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 199.0, 176.1, 160.3, 151.1, 137.7, 137.4, 130.2, 129.6, 127.7, 115.7, 75.8, 56.5, 48.8, 37.8, 31.5, 30.4, 27.5, 23.7, 14.5; HRMS (ESI) calculated for $C_{23}H_{30}NO_4$ $[M+H]^+$ 384.2175, found 384.2178.



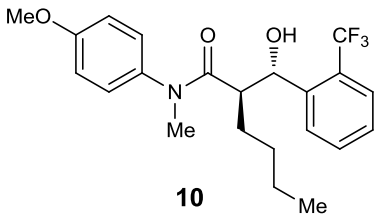

**8**

**(*RS*)-2-((*SR*)-(4-cyanophenyl)(hydroxy)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-8**). Following the standard procedure for three-component condensations, the reaction of 4-formylbenzonitrile (131.1 mg, 1.00 mmol) afforded ***anti*-8** (133.3 mg, 0.364 mmol, 73% yield) as a white solid after purification by flash chromatography (hexanes to 4:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 2H), 6.73-6.57 (m, 2H), 5.04 (d, *J* = 8.0 Hz, 1H), 4.69 (dd, *J* = 7.5, 4.5 Hz, 1H), 3.81 (s, 3H), 3.07 (s, 3H), 2.61 (ddd, *J* = 7.5, 6.5, 6.0 Hz, 1H), 1.72-1.65 (m, 1H), 1.53-1.47 (m, 1H), 1.23-1.13 (m, 4H), 0.83 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.9, 160.3 151.4, 137.2, 133.5, 130.1, 128.4, 120.2, 115.8, 112.0, 75.5, 56.6, 48.6, 37.8, 31.4, 30.4, 23.6, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}N_2O_3$ $[M+H]^+$ 367.2022, found 367.2024.



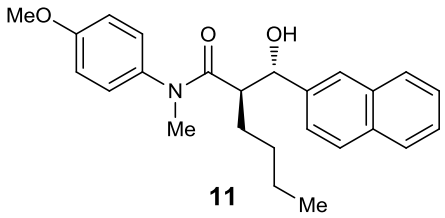

**9**

**(*RS*)-2-((*SR*)-hydroxy(3-(trifluoromethyl)phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-9**). Following the standard procedure for three-component condensations, the reaction of 3-(trifluoromethyl)benzaldehyde (131.1 μL, 1.00 mmol) afforded ***anti*-9** (129.3 mg, 0.316 mmol, 63% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.62 ( d, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.46-7.43 (m, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 6.67-6.50 (m, 2H), 5.05 (d, *J* = 8.0 Hz, 1H), 4.71 (dd, *J* = 7.5, 4.5 Hz, 1H), 3.79 (s, 3H), 3.06 (s, 3H), 2.60 (ddd, *J* = 6.0, 5.5, 5.0 Hz, 1H), 1.75-1.66 (m, 1H), 1.57-1.48 (m, 1H), 1.23-1.13 (m, 4H), 0.84 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.1, 160.3, 147.3, 137.3, 131.3, 131.2 (q, *J* =31.5 Hz), 130.5, 130.0, 125.9 (q, *J* = 269.9 Hz), 125.3 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 3.8 Hz), 115.7, 75.5, 56.5, 48.9, 37.8, 31.5, 30.4, 23.7, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_3F_3$ $[M+H]^+$ 410.1943, found 410.1943.



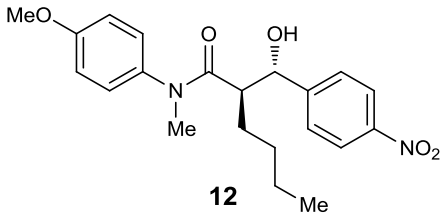

**10**

**(*RS*)-2-((*SR*)-hydroxy(2-(trifluoromethyl)phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-10**). Following the standard procedure for three-component condensations, the reaction of 2-(trifluoromethyl)benzaldehyde (131.9 μL, 1.00 mmol) afforded ***anti*-10** (150.6 mg, 0.368 mmol, 74% yield) as a white solid after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 345 K) δ 7.69-7.65 (m, 2H), 7.62 (d, *J* = 7.5 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H,), 6.77 (d, *J* = 8.5 Hz, 2H,), 6.50-6.28 (m, 2H), 5.28 (d, *J* = 7.5 Hz, 1H), 5.07-5.03 (m, 1H), 3.78 (s, 3H), 3.10 (s, 3H), 2.72-2.66 (m, 1H), 1.82-1.74 (m, 1H), 1.57-1.50 (m, 1H), 1.25-1.13 (m, 4H), 0.85 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.1, 160.3, 144.6, 137.2, 133.8, 129.8, 129.3, 129.2, 127.8 (q, *J* = 29.8 Hz), 127.11 (q, *J* = 6.3 Hz), 125.8 (q, *J* = 272.5 Hz), 115.7, 71.7, 56.6, 48.0, 37.8, 31.9, 30.4, 23.6, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_3F_3$ $[M+H]^+$ 410.1943, found 410.1942.



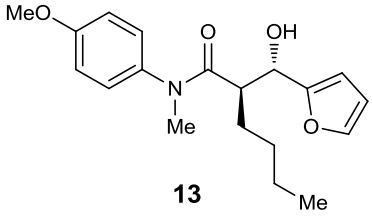

**11**

**(*RS*)-2-((*SR*)-hydroxy(naphthalen-2-yl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-11**). Following the standard procedure for three-component condensations, the reaction of 2-naphthaldehyde (156.2 mg, 1.00 mmol) afforded ***anti*-11** (134.0 mg, 0.342 mmol, 68% yield) as a white solid after purification by flash chromatography (hexanes to 90:10 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.92-7.88 (m, 1H), 7.87-7.83 (m, 2H), 7.66 (s, 1H), 7.54-7.49 (m, 2H), 7.29 (dd, *J* = 8.5, 1.5 Hz, 1H), 6.73 (d, *J* = 8.5 Hz, 2H), 6.65-6.48 (m, 2H), 4.85 (d, *J* = 7.5 Hz, 1H), 4.81-4.77 (m, 1H), 3.78 (s, 3H), 3.06 (s, 3H), 2.75-2.71 (m, 1H), 1.76-1.68 (m, 1H), 1.52-1.45 (m, 1H) 1.25-1.14 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.3, 160.1, 143.3, 137.5, 134.6, 134.2, 130.2, 129.2, 129.1, 129.0, 127.6, 127.2, 126.2, 125.8, 115.5, 76.5, 56.5, 49.0, 37.8, 31.7, 30.4, 23.7, 14.5; HRMS (ESI) calculated for $C_{25}H_{30}NO_3$ $[M+H]^+$ 392.2226, found 392.2228




**12**

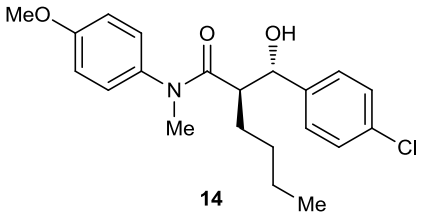
**(*RS*)-2-((*SR*)-hydroxy(4-nitrophenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-12**). Following the standard procedure for three-component condensations, the reaction of 4-nitrobenzaldehyde (151.1 mg, 1.00 mmol) afforded ***anti*-12** (127.0 mg, 0.329 mmol, 66% yield) as a white solid after purification by flash chromatgoraphy (hexanes to 9:1 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 330 K) δ 8.17 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 9.0 Hz, 2H), 6.86 (d, *J* = 9.0 Hz, 2H), 6.75-6.59 (m, 2H), 5.10 (d, *J* = 7.5 Hz, 1H), 4.75 (dd, *J* = 7.5, 5.0 Hz, 1H), 3.80 (s, 3H), 3.07 (s, 3H), 2.66-2.60 (m, 1H), 1.75-1.67 (m, 1H), 1.57-1.49 (m, 1H), 1.23-1.13 (m, 4H), 0.84 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.9, 160.3, 153.5, 148.7, 137.2, 130.1, 128.5, 124.7, 115.8, 75.3, 56.6, 48.6, 37.8, 31.4, 30.4, 23.6, 14.5; HRMS (ESI) calculated for $C_{21}H_{27}N_2O_5$ $[M+H]^+$ 387.1920, found 387.1915.




**13**

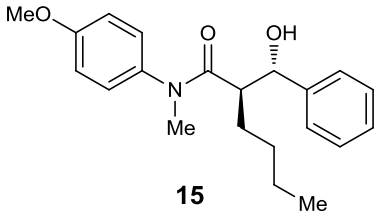
**(*RS*)-2-((*SR*)-furan-2-yl(hydroxy)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-13**). Following the standard procedure for three-component condensations, the reaction of 2-furaldehyde (82.9 μL, 1.00 mmol) afforded ***anti*-13** (97.8 mg, 0.295 mmol, 59% yield) as a white

solid after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 310 K) δ 7.42 (d, *J* = 2.5 Hz, 1H), 6.95-6.92 (m, 4H), 6.37 (dd, *J* = 3.5, 3.0 Hz, 1H), 6.20 (d, *J* = 3.0 Hz, 1H), 4.65-4.62 (m, 1H), 4.56 (d, *J* = 7.5 Hz, 1 H), 3.81 (s, 3H), 3.15 (s, 3H), 2.78-2.74 (ddd, *J* = 8.0, 6.5, 6.0 Hz, 1H), 1.60-1.53 (m, 1H), 1.36-1.29 (m, 1H), 1.15-1.07 (m, 4H), 0.81 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.2, 160.3, 158.0, 143.1, 137.8, 130.4, 115.8, 111.7, 107.8, 70.3, 56.6, 46.7, 38.0, 31.1, 30.3, 23.7, 14.5; HRMS (ESI) calculated for $C_{19}H_{26}NO_4$ $[M+H]^+$ 332.1862, found 332.1868.



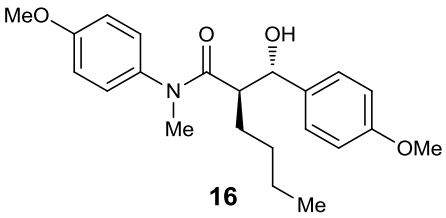
**14**

**(*RS*)-2-((*SR*)-(4-chlorophenyl)(hydroxy)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-14**). Following the standard procedure for three-component condensations, the reaction of 4-chlorobenzaldehyde (140.6 mg, 1.00 mmol) afforded ***anti*-14** (119.1 mg, 0.317 mmol, 63% yield) as a white solid after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.34 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.86 ( d, *J* = 9.0 Hz, 2H), 6.77-6.63 (m, 2H), 4.82 (d, *J* = 7.5 Hz, 1H), 4.62 (dd, *J* = 7.0, 5.5 Hz, 1H), 3.80 (s, 3H), 3.09 (s, 3H), 2.59 (ddd, *J* = 8.0, 7.0, 5.5 Hz, 1H), 1.70-1.62 (m, 1H), 1.47-1.39 (m, 1H), 1.21-1.08 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.1, 160.3, 144.7, 137.5, 133.7, 130.2, 129.5, 129.2, 115.7, 75.6, 56.6, 49.0, 37.9, 31.5, 30.4, 23.7, 14.5; HRMS (ESI) calculated for $C_{21}H_{27}NO_3Cl$ $[M+H]^+$ 376.1679, found 376.1679.



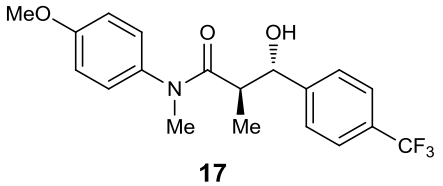
**15**

**(*RS*)-2-((*SR*)-hydroxy(phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (***anti*-15**). Following the standard procedure for three-component condensations, the reaction of benzaldehyde (101.1 μL, 1.00 mmol) afforded ***anti*-15** (99.3 mg, 0.291 mmol, 58% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.35-7.27 (m, 3H), 7.16 (m, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 6.72-6.55 (m, 2H), 4.76 (d, *J* = 7.5 Hz, 1H), 4.63-4.60 (m, 1H), 3.80 (s, 3H), 3.08 (s, 3H), 2.62-2.58 (ddd, *J* = 8.0, 7.0, 5.5 Hz, 1H), 1.72-1.64 (m, 1H), 1.47-1.39 (m, 1H), 1.21-1.11 (m, 4H), 0.81 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.3, 160.2, 145.9, 137.6, 130.3, 129.6, 128.6, 127.5, 115.6, 76.4, 56.5, 49.2, 37.8, 31.7, 30.5, 23.7, 14.5; HRMS (ESI) calculated for $C_{21}H_{28}NO_3$ $[M+H]^+$ 342.2069, found 342.2073.



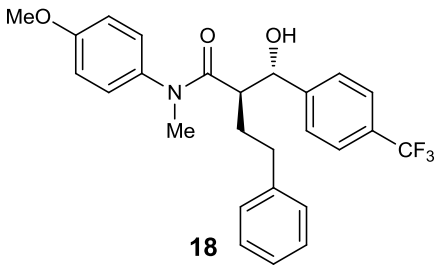

**16**

**(*RS*)-2-((*SR*)-hydroxy(4-methoxyphenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methylhexanamide** (**anti-16**). Following the standard procedure for three-component condensations, the reaction of *p*-anisaldehyde (121.7 μL, 1.00 mmol) afforded ***anti*-16** (82.0 mg, 0.221 mmol, 44% yield) as a white solid after purification by flash chromatography (hexanes to 4:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.07 (d, *J* = 8.5 Hz, 2H), 6.90-6.85 (m, 4H), 6.77-6.68 (m, 2H), 4.57-4.54 (m, 2H), 3.80 (m, 6H), 3.10 (s, 3H) 2.61-2.56 (m, 1H), 1.68-1.59 (m, 1H), 1.41-1.33 (m, 1H), 1.20-1.07 (m, 4H), 0.81 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.4, 160.4, 160.2, 137.9, 137.7, 130.4, 128.7, 115.6, 114.8, 76.1, 56.5, 56.3, 49.4, 37.9, 31.6, 30.5, 23.7, 14.5; HRMS (ESI) calculated for $C_{22}H_{30}NO_4$ $[M+H]^+$ 372.2175, found 372.2178.




**17**

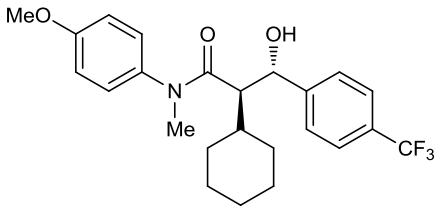
**(2*RS*,3*SR*)-3-hydroxy-*N*-(4-methoxyphenyl)-*N*,2-dimethyl-3-(4-(trifluoromethyl)phenyl) propanamide** (***anti*-17**). Following the standard procedure for three-component condensations, the reaction of 1-siloxy-1-propyne (159.3 mg, 0.750 mmol) afforded ***anti*-17** (138.5 mg, 0.377 mmol, 75% yield) as a white solid after purification by flash chromatography (hexanes to 4:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 315 K) δ 7.63 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.82-6.71 (m, 2H), 4.83 (d, *J* = 7.0 Hz, 1H), 4.66-4.61, (m, 1H), 3.80 (s, 3H), 3.06 (s, 3H), 2.71-2.64 (m, 1H), 1.06 (d, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.6, 160.4, 150.2, 137.5, 130.1 (q, *J* = 31.6 Hz), 129.7, 128.0, 126.4 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.8 Hz), 115.9, 77.1, 56.6, 43.7, 37.7, 16.4; HRMS (ESI) calculated for $C_{19}H_{21}NO_3F_3$ $[M+H]^+$ 368.1474, found 368.1479.




**18**

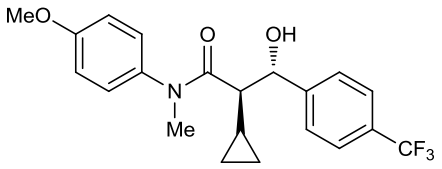
**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-(4-methoxyphenyl)-*N*-methyl-4-phenylbutanamide** (***anti*-18**). Following the standard procedure for three-component condensations, the reaction of siloxy alkyne **29** (226.9 mg, 0.750 mmol) afforded ***anti*-18** (165.0

mg, 0.361 mmol, 72% yield) after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.63 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.22 (t, *J* = 7.5 Hz, 2H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 2H), 6.77 (d, *J* = 9.0 Hz, 2H), 6.63-6.52 (m, 2H), 5.11 (d, *J* = 7.5 Hz, 1H), 4.80 (dd, *J* = 7.5, 4.5 Hz, 1H), 3.80 (s, 3H), 3.07 (s, 3H), 2.67 (ddd, *J* = 9.5, 5.0, 4.5 Hz, 1H), 2.60-2.53 (m, 2H), 2.10-1.87 (d, 2H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.8, 160.3, 150.3, 142.8, 137.1, 130.1 (q, *J* = 31.8 Hz), 129.9, 129.7, 129.6, 128.3, 127.3, 126.5 (q, *J* = 3.6 Hz), 125.9 (q, *J* = 269.6 Hz), 115.8, 74.8, 56.6, 48.2, 37.9, 34.0, 33.0; HRMS (ESI) calculated for $C_{26}H_{27}NO_3F_3$ $[M+H]^+$ 458.1943, found 458.1956.




**19**

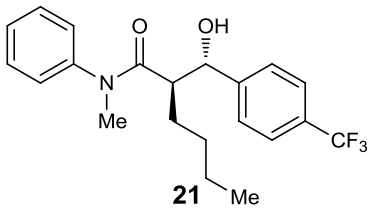
**(2*RS*,3*SR*)-2-cyclohexyl-3-hydroxy-*N*-(4-methoxyphenyl)-*N*-methyl-3-(4-(trifluoromethyl) phenyl)propanamide** (***anti*-19**). Following the standard procedure for three-component condensations, the reaction of 2-cyclohexyl-1-siloxy-ethyne (210.4 mg, 0.750 mmol) afforded ***anti*-19** (175.5 mg, 0.398 mmol, 80% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 350 K) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.72 (d, *J* = 8.5 Hz, 2H), 6.36-6.06 (m, 2H), 5.52 (d, *J* = 9.0 Hz, 1H), 4.93-4.88 (m, 1H), 3.78 (s, 3H), 3.03 (s, 3H), 2.37 (dd, *J* = 9.0, 3.0 Hz, 1H), 2.01-1.93 (m, 2H), 1.79-1.71 (m, 3H), 1.70-1.64 (m, 1H), 1.34-1.22 (m, 2H), 1.21-1.12 (m, 1H), 1.10-1.00 (m, 1H), 0.98-0.88 (m, 1H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.7, 160.3, 150.9, 136.7, 130.2 (q, *J* = 31.5), 129.9, 128.1, 126.6 (q, *J* = 3.8 Hz), 126.0 (q, *J* = 269.8 Hz), 115.5, 72.5, 56.5, 54.4, 39.7, 38.0, 33.2, 31.7, 27.7, 27.5, 27.5; HRMS (ESI) calculated for $C_{24}H_{29}NO_3F_3$ $[M+H]^+$ 436.2100, found 436.2108.



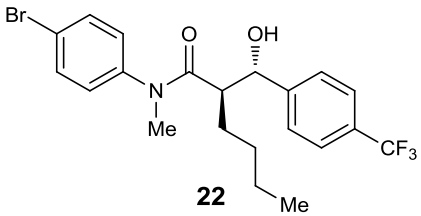

**20**

**(2*RS*,3*SR*)-2-cyclopropyl-3-hydroxy-*N*-(4-methoxyphenyl)-*N*-methyl-3-(4-(trifluoromethyl) phenyl)propanamide** (***anti*-20**). Following the standard procedure for three-component condensations, the reaction of siloxy alkyne **28** (178.8 mg, 0.750 mmol) afforded ***anti*-20** (175.5 mg, 0.433 mmol, 87% yield) as a white solid after purification by flash chromatography (hexanes to 4:1 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 9.0 Hz, 2H), 6.67-6.51 (m, 2H), 5.02 (d, *J* = 7.0 Hz, 1H), 4.88-4.84 (m, 1H), 3.79 (s, 3H), 3.10 (s, 3H), 1.94-1.89 (m, 1H), 1.18-1.11 (m, 1H), 0.47-0.41 (m, 1H), 0.38-0.31 (m, 1H), 0.08-0.02 (m, 1H), -0.18- -0.24 (m, 1H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.4, 160.3, 150.4, 137.2, 130.2 (q, *J* = 31.8 Hz), 130.1, 128.2, 126.4, (q, *J* = 31.8

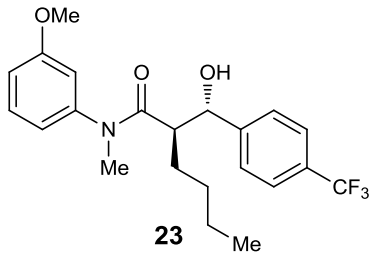
Hz), 126.0 (q, *J* = 269.6 Hz), 115.7, 76.8, 56.5, 53.4, 37.9, 13.4, 5.0, 4.6; HRMS (ESI) calculated for $C_{21}H_{23}NO_3F_3$ $[M+H]^+$ 394.1630, found 394.1633.




**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-methyl-*N*-phenylhexanamide** (***anti*-21**). Following the standard procedure for three-component condensations, the reaction of *N*-methylaniline (54.2 μL, 0.500 mmol) afforded ***anti*-21** (144.0 mg, 0.379 mmol, 76% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 315 K) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.35-7.30 (m, 5H), 6.83-6.70 (m, 2H), 4.97 (d, *J* = 7.0 Hz, 1H), 4.73-4.69 (m, 1H), 3.11 (s, 3H), 2.61 (ddd, *J* = 8.0, 5.5, 5.0 Hz, 1H,), 1.74-1.65 (m, 1H), 1.48-1.40 (m, 1H), 1.23-1.08 (m, 4H), 0.81 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.7, 150.4, 144.7, 130.8, 130.2 (q, 31.8 Hz), 129.2, 129.0, 128.2, 126.5 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.8 Hz), 75.7, 49.1, 37.8, 31.5, 30.4, 23.7, 14.5; HRMS (ESI) calculated for $C_{21}H_{25}NO_2F_3$ $[M+H]^+$ 380.1837, found 380.1836.

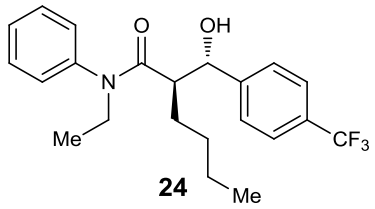



**(*RS*)-*N*-(4-bromophenyl)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-methylhexanamide** (***anti*-22**). Following the standard procedure for three-component condensations, the reaction of 4-bromo-*N*-methylaniline (62.8 μL, 0.500 mmol) afforded ***anti*-22** (170.0 mg, 0.371 mmol, 74% yield) as a white solid after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). $^1$H NMR (500 MHz, $CD_3CN$, 315 K) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.76 (d, *J* = 6.0 Hz, 2H), 4.75-4.68 (m, 2H), 3.11 (s, 3H), 2.64-2.58 (m, 1H), 1.71-1.61 (m, 1H), 1.38-1.28 (m, 1H), 1.21-1.05 (m, 4H), 0.80 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.4, 150.1, 144.0, 133.8, 131.1, 130.2 (q, *J* = 31.8 Hz), 128.3, 126.5 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.6 Hz), 122.3, 75.9, 49.4, 37.7, 31.4, 30.3, 23.7, 14.5; HRMS (ESI) calculated for $C_{21}H_{24}NO_2BrF_3$ $[M+H]^+$ 458.0943, found 458.0953.



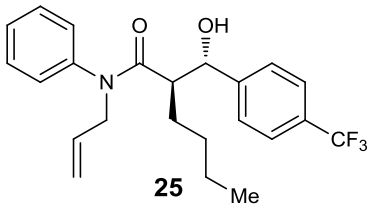

**23**

**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-(3-methoxyphenyl)-*N*-methylhexanamide** (***anti*-23**). Following the standard procedure for three-component condensations, the reaction of 3-methoxy-*N*-methylaniline (65.4 μL, 0.500 mmol) afforded ***anti*-23** (139.0 mg, 0.340 mmol, 68% yield) as a white solid after purification by flash chromatography (hexanes to 85:15 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.23 (t, *J* = 8.0 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 6.41-6.31 (m, 2H), 4.94 (d, *J* = 7.5 Hz, 1H), 4.74-4.70 (m, 1H), 3.75 (s, 3H), 3.11 (s, 3H), 2.70-2.63 (m, 1H), 1.74-1.65 (m, 1H), 1.52-1.44 (m, 1H), 1.23-1.09 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.6, 161.7, 150.3, 145.8, 131.5, 130.1 (q, *J* = 31.6 Hz), 128.1, 126.5 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.3 Hz), 121.0, 114.8, 114.7, 75.7, 56.5, 49.1, 37.7, 31.5, 30.5, 23.7, 14.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_3F_3$ $[M+H]^+$ 410.1943, found 410.1946.



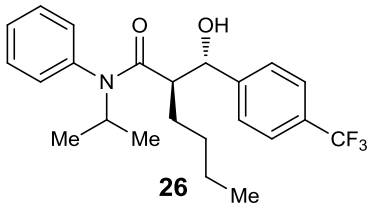

**24**

**(*RS*)-*N*-ethyl-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-phenylhexanamide** (***anti*-24**). Following the standard procedure for three-component condensations, the reaction of *N*-ethylaniline (62.9 μL, 0.500 mmol) afforded ***anti*-24** (153.7 mg, 0.391 mmol, 78% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.65 (d, *J* = 8.0 Hz, 2H), 7.35-7.28 (m, 5H), 6.75-6.50 (m, 2H), 5.09 (d, *J* = 8.0 Hz, 1H), 4.71 (dd, *J* = 7.5, 4.5 Hz, 1H), 3.69 (dq, *J* = 14.0, 7.0 Hz, 1H), 3.53 (dq, J = 14.0, 7.0 Hz, 1H), 2.51-2.46 (m, 1H), 1.76-1.67 (m, 1H), 1.58-1.49 (m, 1H), 1.25-1.10 (m, 4H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.83 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.2, 150.5, 142.8, 130.7, 130.2 (q, *J* = 31.6 Hz), 130.0, 129.4, 128.2, 126.4 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 269.4 Hz), 75.6, 49.1, 45.2, 31.6, 30.4, 23.7, 14.5, 13.5; HRMS (ESI) calculated for $C_{22}H_{27}NO_2F_3$ $[M+H]^+$ 394.1994, found 394.1996.



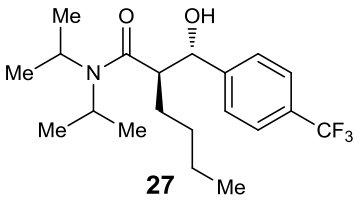

**25**

**(*RS*)-*N*-allyl-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-phenylhexanamide** (***anti*-25**). Following the standard procedure for three-component condensations, the reaction of *N*-allylaniline (67.8 μL, 0.500 mmol) afforded ***anti*-25** (115.1 mg, 0.284 mmol, 57% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.65 (d, *J* = 8.0 Hz, 2H), 7.37-7.29 (m, 5H), 6.73 (m, 2H), 5.74 (ddt, *J* = 12.0, 10.5, 6.0 Hz, 1H), 5.03 (d, *J* = 10.0 Hz, 1H), 4.98 (dd, *J* = 17.0, 1.5 Hz, 1H), 4.91 (d, *J* = 7.5 Hz, 1H), 4.76-4.71 (m, 1H), 4.29 (dd, *J* = 15.0, 6.0 Hz, 1H), 4.11 (dd, *J* = 15.0, 6.0 Hz 1H), 2.60-2.54 (m, 1H), 1.76-1.67 (m, 1H), 1.54-1.45 (m, 1H), 1.25-1.10 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.5, 150.3, 143.0, 134.6, 130.6, 130.2 (q, *J* = 31.6 Hz), 130.1, 129.3, 128.3, 126.5 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 268.8 Hz) 118.4, 75.6, 53.0, 49.3, 31.5, 30.4, 23.6, 14.5; HRMS (ESI) calculated for $C_{23}H_{27}NO_2F_3$ $[M+H]^+$ 406.1994, found 406.1998.




**26**

**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*-isopropyl-*N*-phenylhexanamide** (***anti*-26**). Following the standard procedure for three-component condensations, the reaction of *N*-isopropylaniline (72.4 μL, 0.500 mmol) afforded ***anti*-26** (106.1 mg, 0.260 mmol, 52% yield) as a white solid after purification by flash chromatography (hexanes to 9:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.66 (d, *J* = 8.0 Hz, 2H), 7.41-7.35 (m, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.24-7.15 (m, 1H), 7.13-7.05 (m, 1H), 5.99-5.91 (m, 1H), 5.25 (d, *J* = 8.0 Hz, 1H), 4.87-4.77 (m, 1H), 4.68 (dd, *J* = 7.0, 4.0 Hz, 1H), 2.31-2.28 (m, 1H), 1.76-1.67 (m, 1H), 1.63-1.55 (m, 1H), 1.26-1.12 (m, 4H), 0.96 (t, *J* = 6.5 Hz, 3H) 0.88-0.81 (m, 6H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.3, 150.6, 138.8, 131.8, 131.7, 130.2, 130.2, 130.1 (q, *J* = 31.9 Hz), 129.7, 128.2, 126.4 (q, *J* = 3.8 Hz), 125.9 (q, *J* = 270.0 Hz), 75.3, 49.7, 47.2, 31.6, 30.4, 23.7, 21.5, 21.3, 14.5; HRMS (ESI) calculated for $C_{23}H_{29}NO_2F_3$ $[M+H]^+$ 408.2150, found 408.2154.



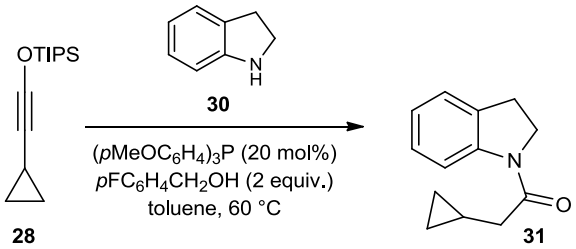

**27**

**(*RS*)-2-((*SR*)-hydroxy(4-(trifluoromethyl)phenyl)methyl)-*N*,*N*-diisopropylhexanamide** (***anti*-27**). Following the standard procedure for three-component condensations, the reaction of

diisopropylamine (35.1 μL, 0.250 mmol) afforded ***anti*-27** (28.3 mg, 0.076 mmol, 30% yield) as a white solid after purification by flash chromatography (hexanes to 12:1 hexanes:EtOAc). $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.63 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 5.76 (d, *J* = 8.5 Hz, 1H), 4.82 (dd, *J* = 8.0, 3.0 Hz, 1H), 3.87-3.78 (m, 1H), 3.34-3.20 (m, 1H), 3.05 (ddd, *J* = 9.0, 6.0, 3.5 Hz, 1H), 1.92-1.82 (m, 1H), 1.76-1.67 (m, 1H), 1.41-1.30 (m, 4H), 1.29 (d, *J* = 7.0 Hz, 3H), 1.14 (d, *J* = 7.0 Hz, 3H), 1.04 (d, *J* = 7.0 Hz, 3H), 0.82 (t, *J* = 7.5 Hz, 3H), 0.51 (d, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 176.0, 151.0, 129.8 (q, *J* = 31.3 Hz), 128.0, 126.2 (q, *J* = 3.8 Hz), 126.0 (q, *J* = 269.5 Hz), 75.8, 50.3, 48.1, 47.3, 32.5, 30.8, 24.0, 21.3, 21.2, 21.0, 20.6, 14.7; HRMS (ESI) calculated for $C_{20}H_{31}NO_2F_3$ $[M+H]^+$ 374.2307, found 374.2306.

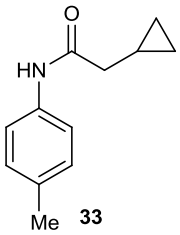
## 3. New Phosphine-Catalyzed Reactions

**Procedure for Acylation of Amines**.




To a solution of tris(4-methoxyphenyl)phosphine (17.6 mg, 0.050 mmol), siloxy alkyne **28** (89.4 mg, 0.375 mmol), and indoline **30** (29.8 μL, 0.250 mmol) in toluene (0.50 mL) was added 4-fluorobenzyl alcohol (54.6 μl, 0.50 mmol). The reaction was heated to 60 °C in an oil bath and was allowed to proceed for 48 hours. The reaction mixture was cooled to room temperature, and was concentrated by rotary evaporation. The crude residue was loaded directly onto a silica column and purified by flash chromatography (97.5:2.5 hexanes:EtOAc) affording amide **31** (47.7mg, 0.237 mmol, 95% yield) as a white solid.

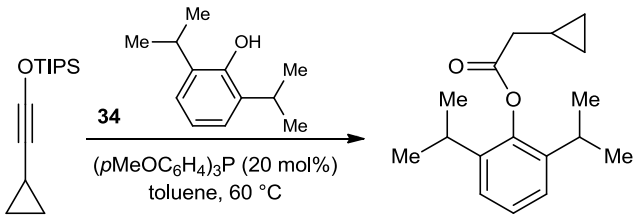
**2-cyclopropyl-1-(indolin-1-yl)ethanone** (**31**). $^{1}$H NMR (500 MHz, $CDCl_3$, 293 K) δ 8.27 (d, *J* = 8.0 Hz, 1H), 7.21-7.14 (m, 2H), 7.02-6.96 (m, 1H), 4.01 (t, *J* = 8.5 Hz, 2H), 3.18 (t, *J* = 8.5 Hz, 2H), 2.37 (d, *J* = 7.0 Hz, 2H), 1.22-1.12 (m, 1H), 0.64-0.59 (m, 2H), 0.25-0.17 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) 171.2, 143.3, 131.2, 127.8, 124.7, 123.7, 117.3, 48.1, 41.4, 28.3, 6.8, 4.7; HRMS (ESI) calculated for $C_{13}H_{16}NO$ $[M+H]^+$ 202.1232, found 202.1233.




**2-cyclopropyl-*N*-(*p*-tolyl)acetamide** (**33**). Following the general procedure for acylation of amines, the reaction of *p*-toluidine **32** (27.5 μL, 0.250 mmol) afforded amide **33** (45.5 mg, 0.240

mmol, 96% yield) as a white solid $^{1}$H NMR (500 MHz, $CDCl_3$, 293 K) δ 7.70 (br s, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 2.35-2.29 (m, 5H), 1.12-1.02 (m, 1H), 0.70-0.64 (m, 2H) 0.30-0.24 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) 170.8, 135.5, 134.0, 129.6, 120.2, 42.5, 21.0, 7.4, 4.9; HRMS (ESI) calculated for $C_{12}H_{16}NO$ $[M+H]^+$ 190.1232, found 190.1234.

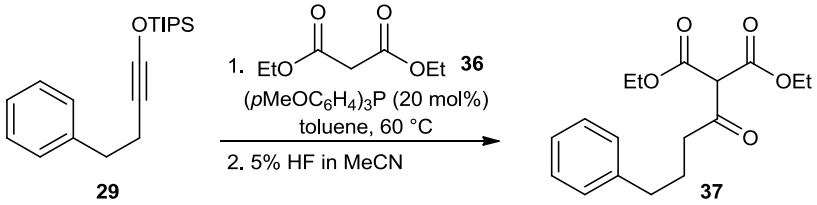
**Procedure for the Acylation of 2,6-Diisopropylphenol**.




**28** **35**

**2,6-diisopropylphenyl 2-cyclopropylacetate** (**35**). To a solution of tris(4-methoxyphenyl)-phosphine (17.6 mg, 0.050 mmol) and siloxy alkyne **28** (59.6 mg, 0.25 mmol) in toluene (0.50 mL) was added 2,6-diisopropylphenol (92.7 μL, 0.500 mmol). The reaction was heated to 60 °C in an oil bath and was allowed to proceed for 48 hours. The reaction mixture was cooled to room temperature, and was concentrated by rotary evaporation. The crude residue was loaded directly onto a silica column and purified by flash chromatography (200:1 hexanes:EtOAc) affording **35** as a clear oil (55.0 mg, 0.211 mmol, 85 % Yield). $^{1}$H NMR (500 MHz, $CDCl_3$, 293 K) δ 7.23-7.20 (m, 1H), 7.17-7.15 (m, 2H), 2.96 (septet, *J* = 7.0 Hz, 2H), 2.50 (d, *J* = 7.0 Hz, 2H), 1.29-1.24 (m, 1H), 1.21 (d, *J* = 7.0 Hz, 12H), 0.67-0.62 (m, 2H), 0.33-0.29 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) 172.0, 145.9, 140.5, 126.6, 124.1, 39.8, 27.7, 23.9, 23.0, 7.5, 4.8; HRMS (ESI) calculated for $C_{17}H_{24}O_2Na$ $[M+Na]^+$ 283.1674, found 283.1669.

**Procedure for the Acylation of Diethyl Malonate**.




**29** **37**

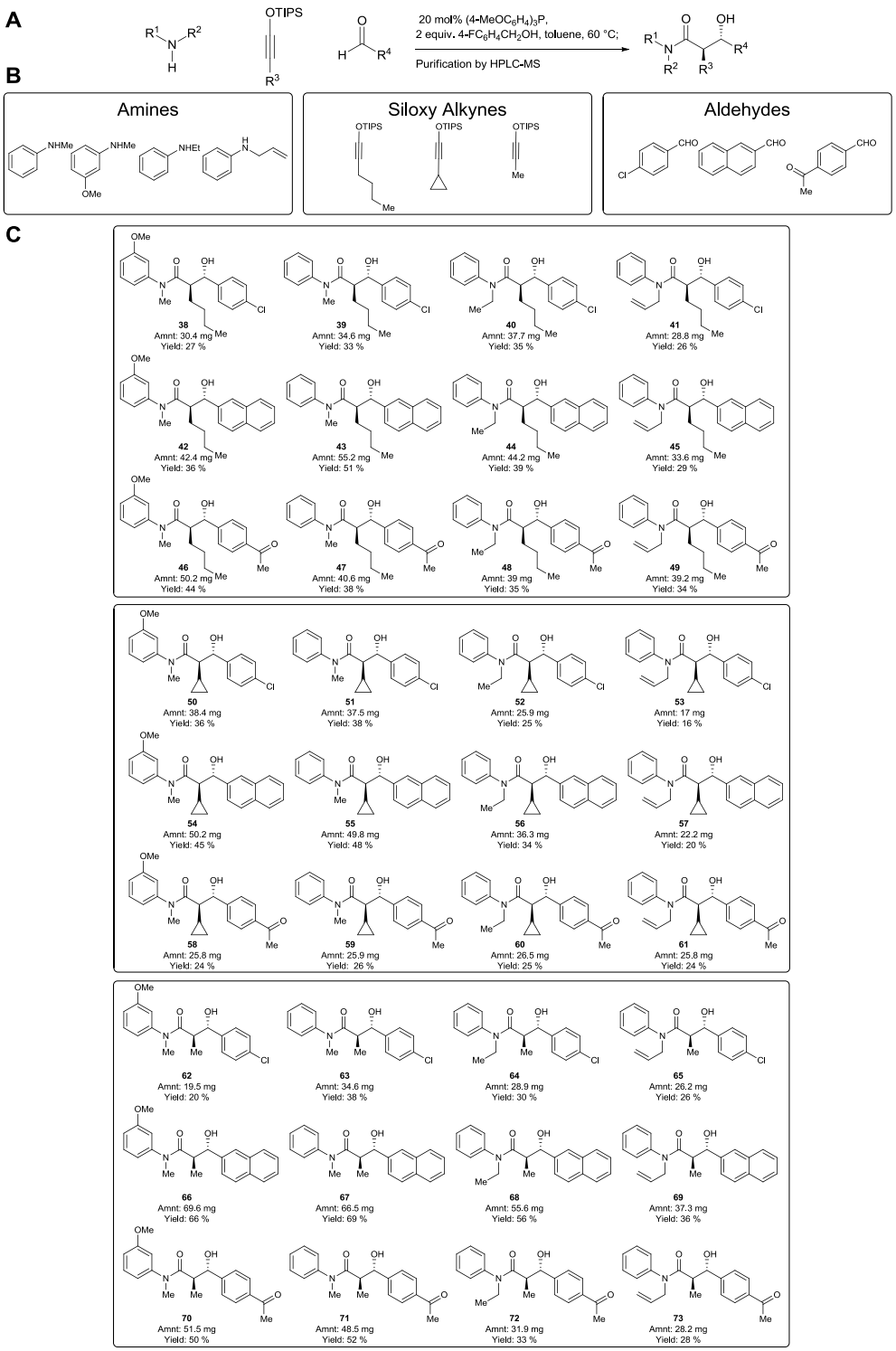
**Diethyl 2-(4-phenylbutanoyl)malonate** (**37**). Diethyl malonate (151.8 μL, 1.0 mmol) was added to a solution of tris(4-methoxyphenyl)phosphine (35.4 mg, 0.100 mmol) and siloxy alkyne **29** (151.3 mg, 0.3500 mmol) in toluene (0.50 mL). The reaction was heated to 60 °C in an oil bath and was allowed to proceed for 48 h. The reaction mixture was then cooled to room temperature and was treated with HF (500 μl, 5% aq., prepared by dilution of Fisher 49% aq. HF with $CH_3CN$). The reaction was allowed to proceed for an additional 2 h, the mixture was diluted with $CH_2Cl_2$ (5 mL) and washed with saturated aqueous $NaHCO_3$ (5 mL). The aqueous layer was extracted with $CH_2Cl_2$ (3 x 5 mL), the combined organic layers were dried with anhydrous $Na_2SO_4$, filtered, and concentrated via rotary evaporation. The crude mixture was purified by flash chromatography (hexanes to 97:3 hexanes:EtOAc) affording **37** as a clear oil (114.5 mg,

0.374 mmol, 75% yield) as a 1.5:1 mixture of enol and keto tautomers. $^{1}$H NMR (500 MHz, $CDCl_3$, 293 K) δ 13.44, 4.33 (s, s, total 1H), 7.30-7.25 (m, 2H), 7.21-7.15 (m, 3H), 4.29-4.16 (m, 4H), 2.69-2.61 (m, 3H), 2.50-2.45 (m, 1H), 2.01-1.92 (m, 2H), 1.33-1.24 (m, 6H); $^{13}$C NMR (125 MHz, $CDCl_3$) 198.9, 182.8, 171.4, 166.3, 164.8, 141.7, 141.6, 128.7, 128.6, 128.6, 126.2, 126.2, 100.1, 65.6, 62.5, 61.6, 61.2, 41.4, 35.6, 34.9, 33.6, 28.5, 25.1, 14.3, 14.3, 14.2; HRMS (ESI) calculated for $C_{17}H_{23}O_5$ $[M+H]^+$ 307.1545, found 307.1543.

## 4. Library Synthesis and Characterization

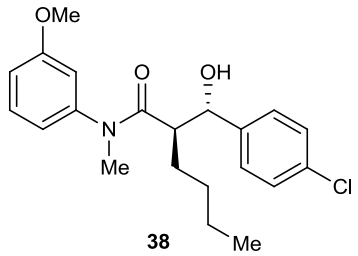
### General Procedure for the Synthesis of Library Compounds

A flame-dried test tube equipped with a stirbar was charged with 0.3 mmol of the requisite amine, and 0.6 ml of a 0.1M solution of tris(4-methoxyphenyl)phosphine in anhydrous toluene. To the resulting suspension was added the requisite aldehyde (0.6 mmol), siloxy alkyne (0.45 mmol) and 4-fluorobenzyl alcohol (65.6 μL, 0.6 mmol). The test tubes were sealed with septa, reactions were heated to 60 °C and were allowed to progress for 48 hours. The reactions were cooled to room temperature and concentrated by rotary evaporation. The resulting crude mixtures were diluted with DMSO until a final volume of 1.0 mL was reached. All library compounds were purified by preparative HPLC-MS on a Waters system composed of the following components: Waters 2545 binary gradient module, Waters 515 HPLC pump, Waters 3100 quadrupole mass spectrometer, Waters system fluidics organizer, Waters 2767 sample manager, Waters 2489 dual channel UV-Vis detector, Waters 2424 evaporative light scattering detector, and Masslynx software v4.1. Preparative HPLC conditions: Waters X-bridge Prep C18 5 μm OBD 19×150 mm column, flow rate 19.0 mL/min, injection volume 1.0 mL; mobile phase A: water with 0.1% formic acid; mobile phase B: methanol with 0.1% formic acid; typical gradient: 0-1.25 min 40-63% B, 1.25-7.25 min 63-77% B, 7.25-8.25 min 77-92% B, 8.25-11.85 min 92-100% B, 11.85-12 min 100-40% B. Fractions were collected by ES+ MS detection of product ion. The purity of all library compounds was then assessed by analytic HPLC-MS. Analytical HPLC conditions: ES Industries Sonoma C18 5 μm 100 Å 5 cm X 2.1 mm, flow rate 1.2 mL/min, injection volume 20 μL, ; mobile phase A: water with 0.1% formic acid; mobile phase B: methanol with 0.1% formic acid; typical gradient: 0-4.25 min 45-100% B, 4.25-5.00 min 100% B, 5.00-5.50 min 100-45% B, 5.50-6.50 min 45% B. Analytical traces for all library members are provided below. Compounds **41**, **43**, **50**, **68**, and **73** were randomly selected for further analysis by $^{1}$H and $^{13}$C NMR spectroscopy.




**Supplementary Figure S2**. **Synthesis of a 36-Member Library**. **A**: Reaction scheme and purification method. **B:** Structures of building blocks used for library construction. **C:** Structures of individual library members, as well as their isolated yields.

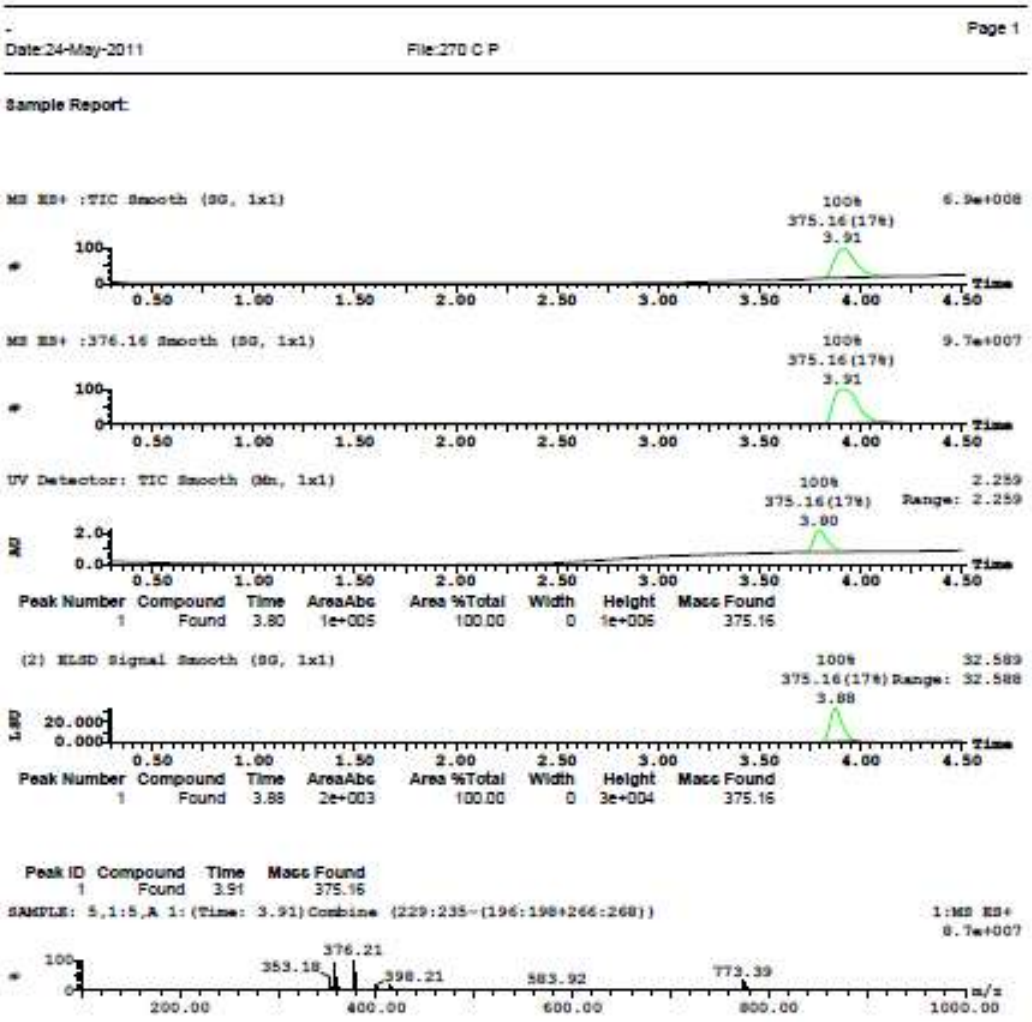
## Purity Reports for All Library Compounds

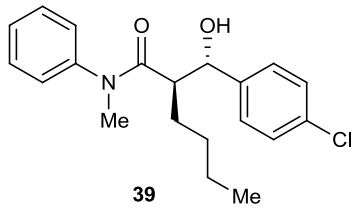



**38**

Chemical Formula: $C_{21}H_{26}ClNO_3$

Exact Mass: 375.16






**39**

Chemical Formula: $C_{20}H_{24}ClNO_2$

Exact Mass: 345.15



Date:24-May-2011 File:270 A P

**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 100% 5.7e+008

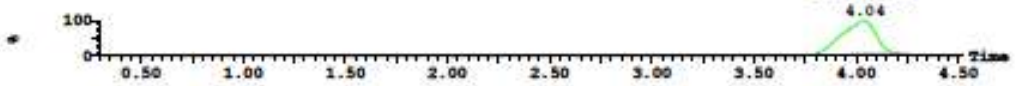
345.15(17%)

4.04


MS ES+ :346.15 Smooth (SG, 1x1) 100% 8.1e+007

345.15(17%)

4.04




UV Detector: TIC Smooth (Mn, 1x1) 1.207

Range: 1.207

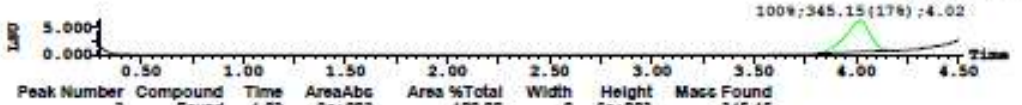
446;345.15(17%);3.94


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 3.66 | 1e+005 | 55.81 | 1 | 1e+005 | |
| 2 | Found | 3.94 | 1e+005 | 44.19 | 1 | 4e+005 | 345.15 |

(2) ELSD Signal Smooth (SG, 1x1) 6.370

Range: 6.369

100%;345.15(17%);4.02

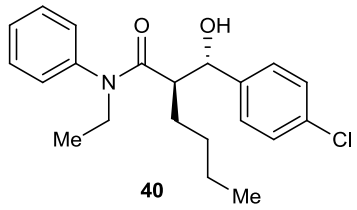



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.02 | 8e+002 | 100.00 | 0 | 6e+003 | 345.15 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 4.04 | 345.15 |

SAMPLE: S,1:3,A 2:(Time: 4.04)Combine (237:243-(204:206+1:-1)) 1:MS ES+

7.3e+007




**40**

Chemical Formula: $C_{21}H_{26}ClNO_2$

Exact Mass: 359.17

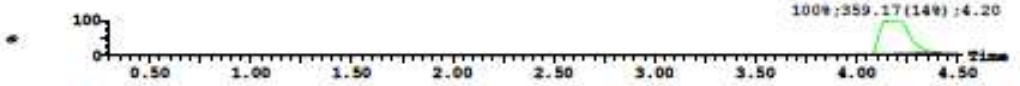


Date:24-May-2011 File:270 B P

**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 8.5e+008

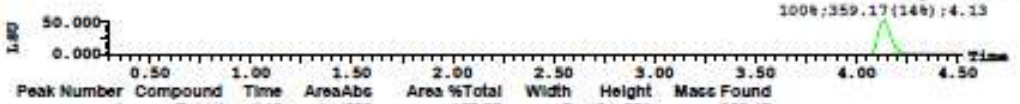

MS ES+ :360.17 Smooth (SG, 1x1) 9.3e+007




UV Detector: TIC Smooth (Mn, 1x1) 2.315
Range: 2.315


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 4.05 | 1e+005 | 100.00 | 1 | 1e+006 | 359.17 |

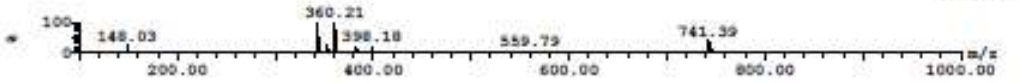
(2) ELSD Signal Smooth (SG, 1x1) 53.114
Range: 53.114

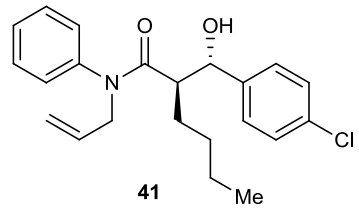



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 4.13 | 4e+003 | 100.00 | 0 | 5e+004 | 359.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 4.16 | 359.17 |

SAMPLE: 5,1:4,A 1:(Time: 4.20) Combine (246:252-(213:215+1:-1)) 1:MS ES+
8.8e+007






**41**

Chemical Formula: $C_{22}H_{26}ClNO_2$

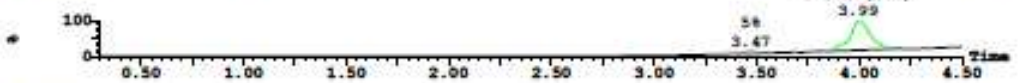
Exact Mass: 371.17



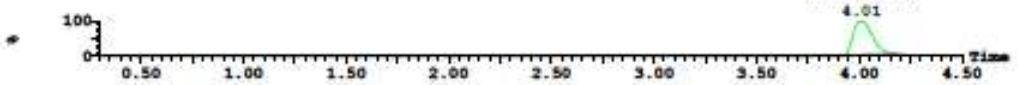
Date:24-May-2011 File:270 D P

**Sample Report:**

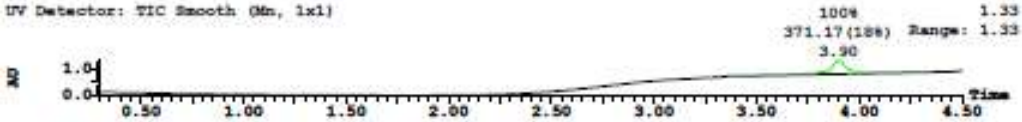
MS ES+ :TIC Smooth (SG, 1x1) 6.1e+008




MS ES+ :372.17 Smooth (SG, 1x1) 9.3e+007




UV Detector: TIC Smooth (Mn, 1x1) 1.33



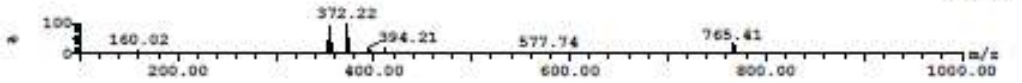

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.90 | 6e+004 | 100.00 | 1 | 5e+005 | 371.17 |

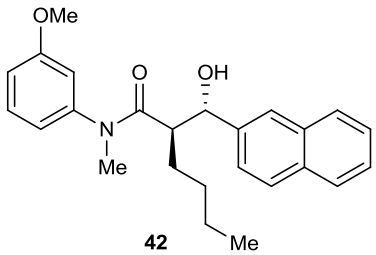
(2) ELSD Signal Smooth (SG, 1x1) 8.604


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.98 | 7e+002 | 100.00 | 0 | 8e+003 | 371.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.99 | 371.17 |

SAMPLE: 5,1:6,A 2:(Time: 4.01)Combine (235:241-(202:204+272:274)) 1:MS ES+ 7.4e+007






**42**

Chemical Formula: $C_{25}H_{29}NO_3$

Exact Mass: 391.21

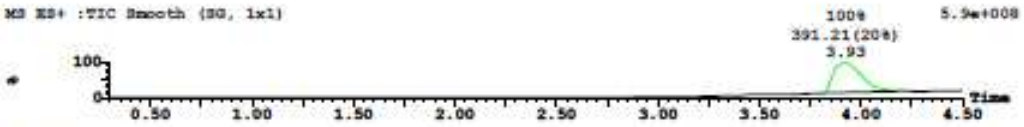
-

Page 1

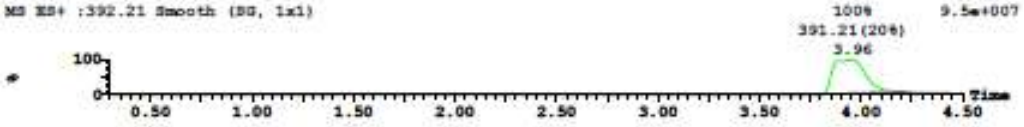
Date:24-May-2011 File:271 C P

**Sample Report:**

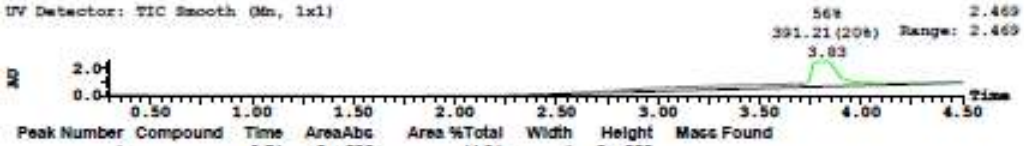
MS ES+ :TIC Smooth (SG, 1x1)




MS ES+ :392.21 Smooth (SG, 1x1)

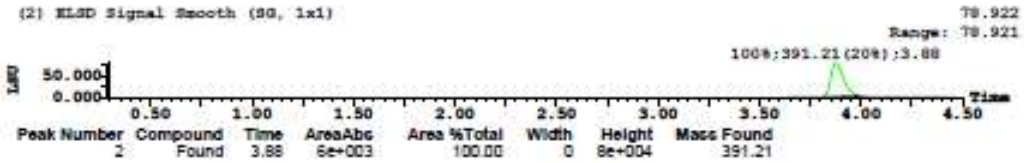



UV Detector: TIC Smooth (Mn, 1x1)




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 3.71 | 2e+005 | 44.31 | 1 | 2e+005 | |
| 2 | Found | 3.83 | 3e+005 | 55.69 | 1 | 2e+005 | 391.21 |

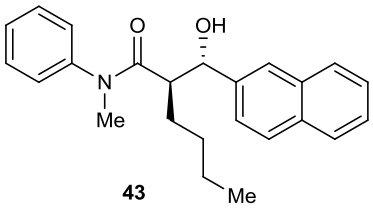
(2) ELSD Signal Smooth (SG, 1x1)




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.88 | 6e+003 | 100.00 | 0 | 8e+004 | 391.21 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.93 | 391.21 |

SAMPLE: 5,1:3,B 2:(Time: 3.96)Combine (232:238-(199:201+269:271))




**43**

Chemical Formula: $C_{24}H_{27}NO_2$
Exact Mass: 361.20

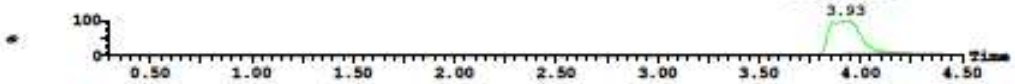


Date:24-May-2011 File:271 A P

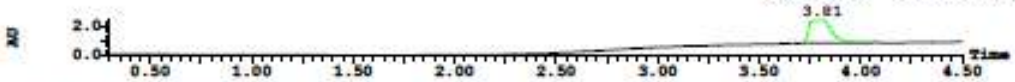
**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 100% 6.3e+008
361.20(18%)
3.91


MS ES+ :362.2 Smooth (SG, 1x1) 100% 9.4e+007
361.20(18%)
3.93

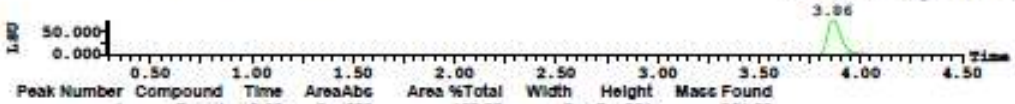



UV Detector: TIC Smooth (Mn, 1x1) 100% 2.459
361.20(18%) Range: 2.459
3.81




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.81 | 2e+005 | 100.00 | 0 | 2e+006 | 361.20 |

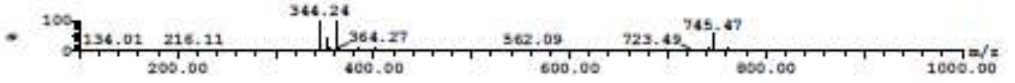
(2) ELSD Signal Smooth (SG, 1x1) 100% 74.890
361.20(18%) Range: 74.889
3.86

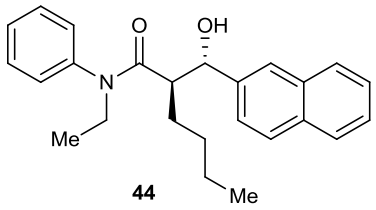



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.86 | 6e+003 | 100.00 | 0 | 7e+004 | 361.20 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.91 | 361.20 |

SAMPLE: 5,1:1,B 1:(Time: 3.93) Combine (230:236-(197:199+267:269)) 1:MS ES+
8.9e+007






**44**

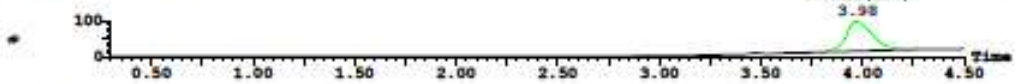
Chemical Formula: $C_{25}H_{29}NO_2$
Exact Mass: 375.22



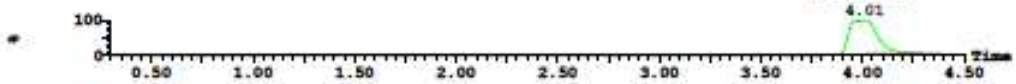
Date:24-May-2011 File:271 B P

**Sample Report:**

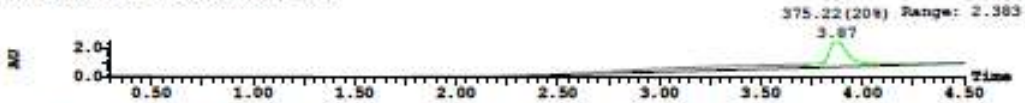
MS ES+ :TIC Smooth (SG, 1x1) 100% 375.22(20%) 3.98 6.1e+008




MS ES+ :376.22 Smooth (SG, 1x1) 100% 375.22(20%) 4.01 9.4e+007

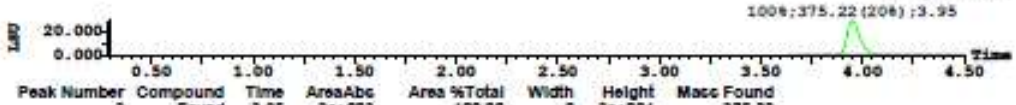



UV Detector: TIC Smooth (Mn, 1x1) 50% 375.22(20%) 3.87 2.383 Range: 2.383




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 3.73 | 2e+005 | 50.02 | 1 | 2e+005 | |
| 2 | Found | 3.87 | 2e+005 | 49.98 | 1 | 2e+006 | 375.22 |

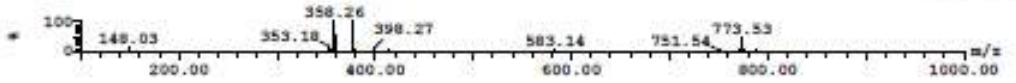
(2) ELSD Signal Smooth (SG, 1x1) 29.819 Range: 29.819 100%;375.22(20%);3.95

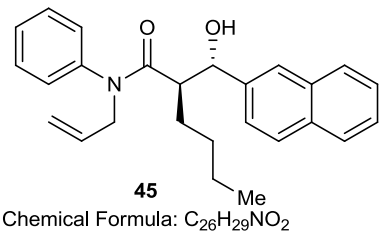



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.95 | 2e+003 | 100.00 | 0 | 3e+004 | 375.22 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.98 | 375.22 |

SAMPLE: 5,1:2,B 2:(Time: 4.01)Combine (235:241-(202:204+272:274)) 1:MS ES+ 8.9e+007





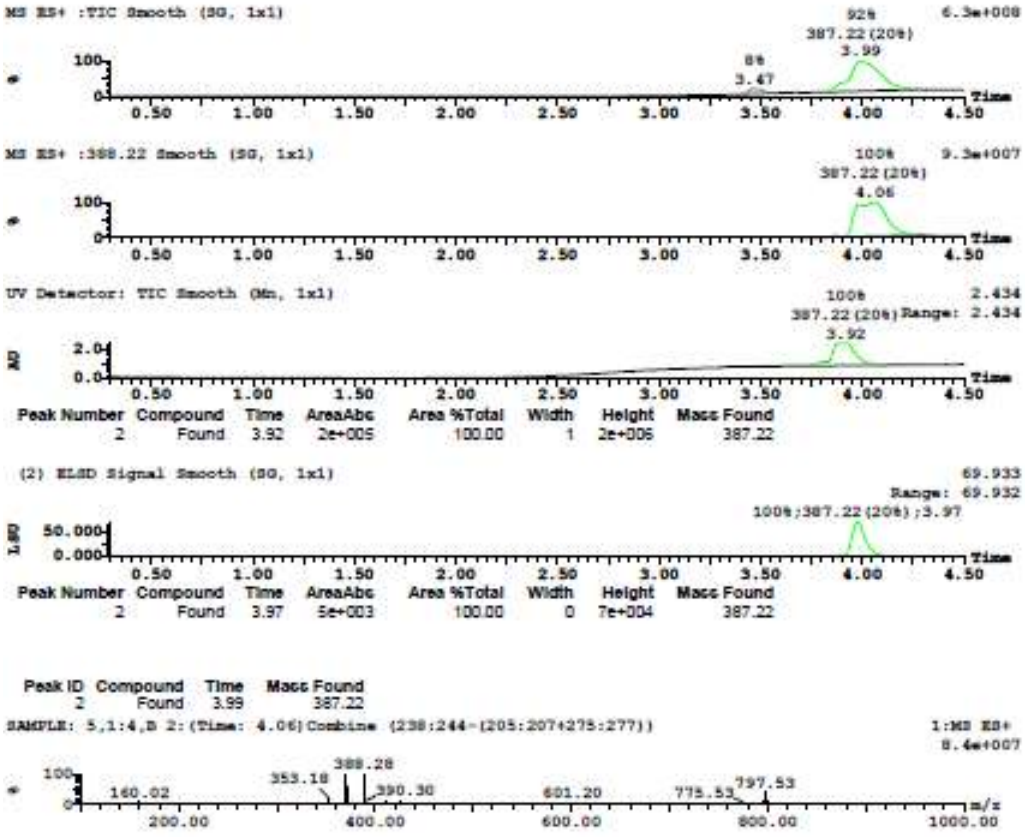

**45**

Chemical Formula: $C_{26}H_{29}NO_2$
Exact Mass: 387.22



Date:24-May-2011 File:271 D P

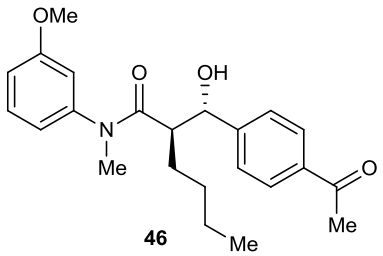
**Sample Report:**




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.92 | 2e+005 | 100.00 | 1 | 2e+006 | 387.22 |

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.97 | 5e+003 | 100.00 | 0 | 7e+004 | 387.22 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.99 | 387.22 |




**46**

Chemical Formula: $C_{23}H_{29}NO_4$

Exact Mass: 383.21

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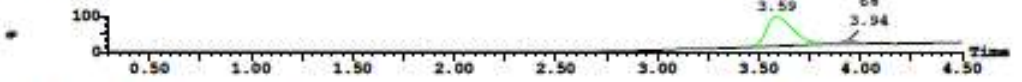
Page 1

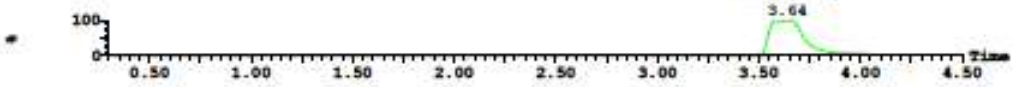
Date:24-May-2011 File:272 C P

**Sample Report:**

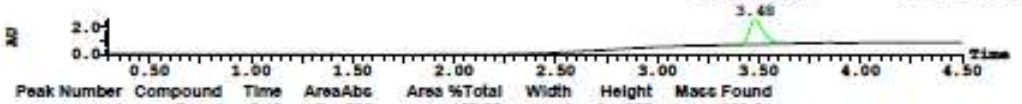
MS ES+ :TIC Smooth (SG, 1x1) 5.7e+008




MS ES+ :384.21 Smooth (SG, 1x1) 9.4e+007

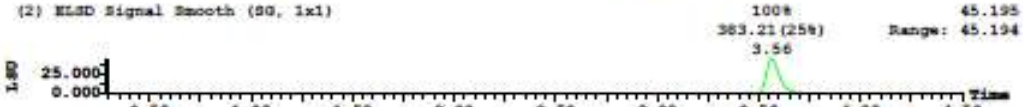



UV Detector: TIC Smooth (Mn, 1x1) 2.519




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.48 | 2e+005 | 100.00 | 1 | 2e+006 | 383.21 |

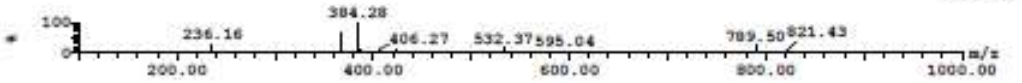
(2) ELSD Signal Smooth (SG, 1x1) 45.195

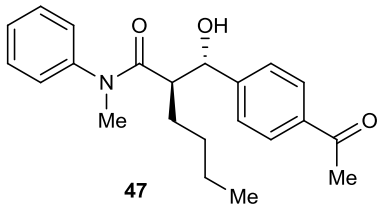



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.56 | 3e+003 | 100.00 | 0 | 5e+004 | 383.21 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.59 | 383.21 |

SAMPLE: 5,1:1,C 1:(Time: 3.64) Combine (213:219-(180:182+250:252)) 1:MS ES+ 9.0e+007






**47**

Chemical Formula: $C_{22}H_{27}NO_3$

Exact Mass: 353.20

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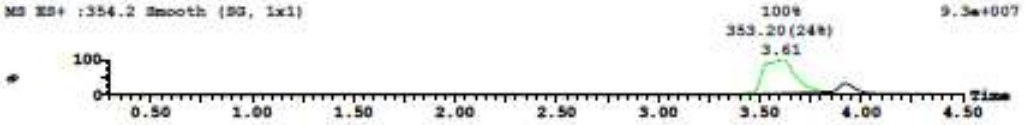
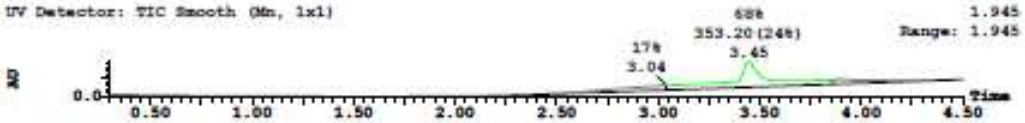
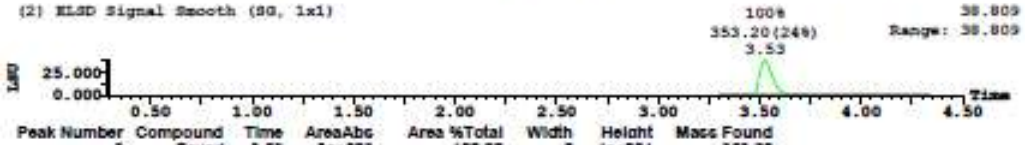
Date:24-May-2011 File:272 A.P

**Sample Report:**

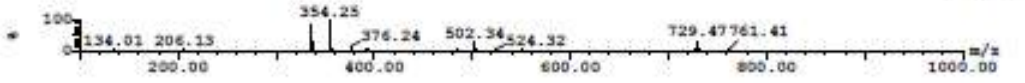

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 3.04 | 8e+004 | 17.24 | 1 | 3e+005 | |
| 2 | Found | 3.45 | 3e+005 | 67.68 | 1 | 1e+006 | 353.20 |
| 3 | | 3.90 | 7e+004 | 15.08 | 1 | 3e+005 | |

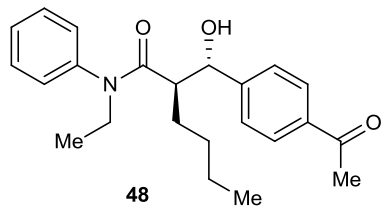



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.53 | 3e+003 | 100.00 | 0 | 4e+004 | 353.20 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.56 | 353.20 |

SAMPLE: 5,1:5,B 2:(Time: 3.61)Combine (211:217-(178:180+248:250))






**48**

Chemical Formula: $C_{23}H_{29}NO_3$

Exact Mass: 367.21

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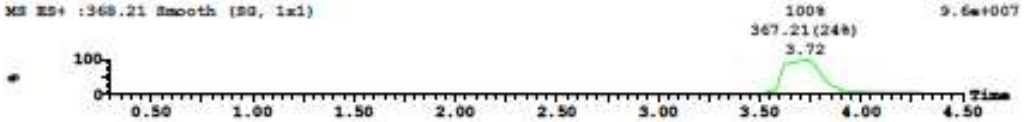
Page 1

Date:24-May-2011 File:272 B P

**Sample Report:**

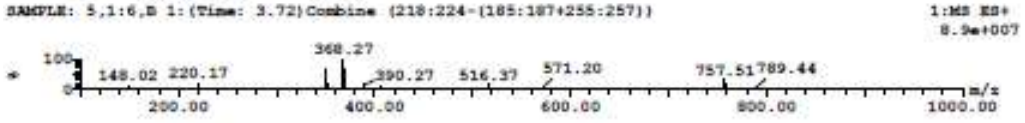



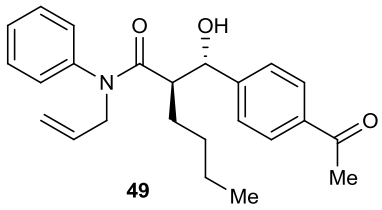


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.55 | 1e+005 | 100.00 | 0 | 2e+006 | 367.21 |


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.63 | 6e+003 | 100.00 | 0 | 7e+004 | 367.21 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.64 | 367.21 |






**49**

Chemical Formula: $C_{24}H_{29}NO_3$

Exact Mass: 379.21

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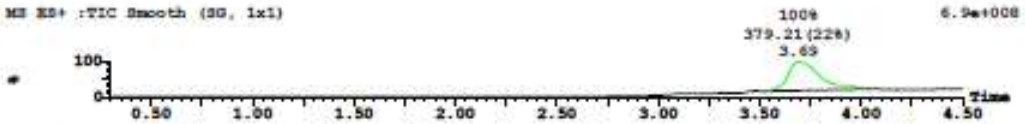
Page 1

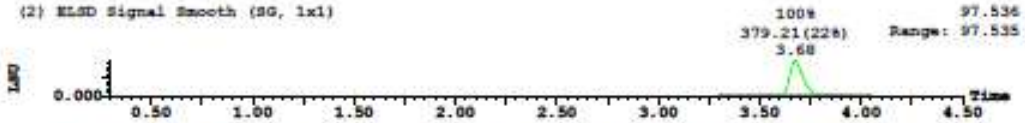
Date:24-May-2011 File:272 D P

**Sample Report:**



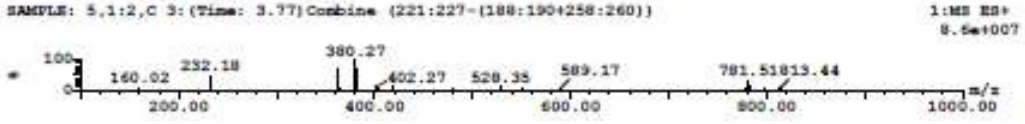


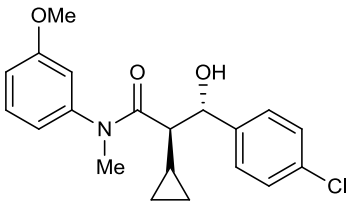

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 3.29 | 1e+005 | 26.65 | 1 | 3e+005 | |
| 2 | | 3.44 | 4e+004 | 8.24 | 0 | 3e+005 | |
| 3 | Found | 3.60 | 3e+005 | 65.11 | 1 | 2e+006 | 379.21 |




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 3 | Found | 3.68 | 8e+003 | 100.00 | 0 | 1e+005 | 379.21 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 3 | Found | 3.69 | 379.21 |

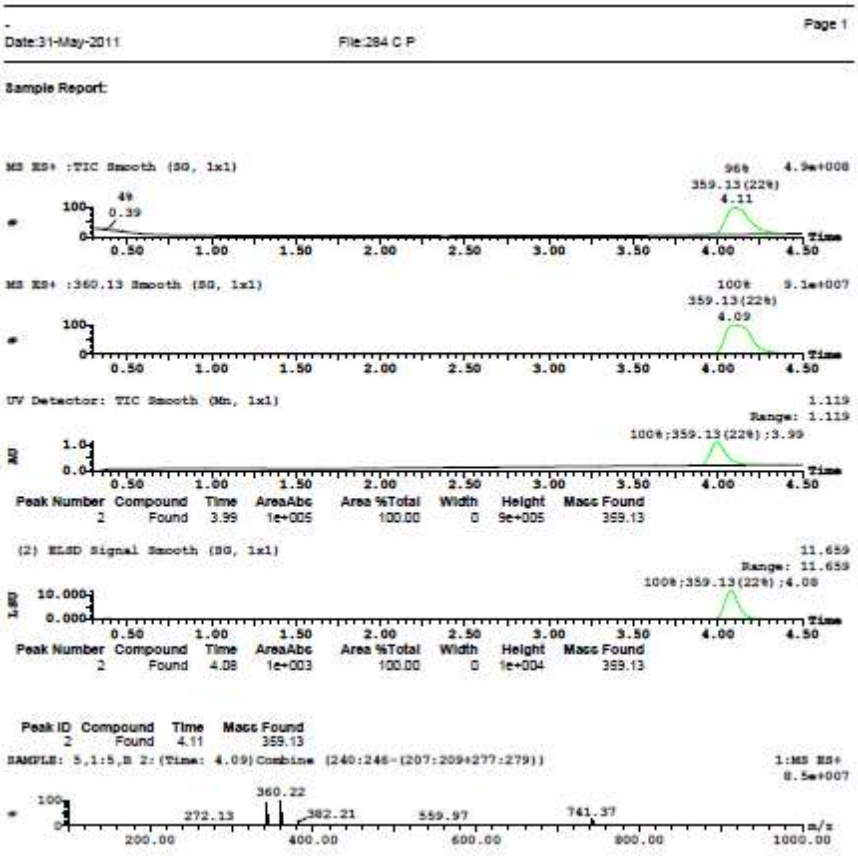


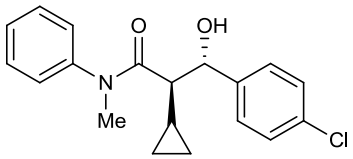



**50**

Chemical Formula: $C_{20}H_{22}ClNO_3$

Exact Mass: 359.13






**51**

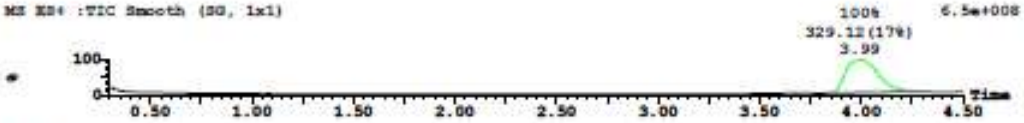
Chemical Formula: $C_{19}H_{20}ClNO_2$
Exact Mass: 329.12



Date:31-May-2011 File:284 A P

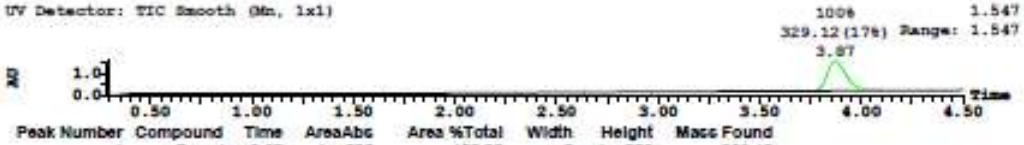
**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1)



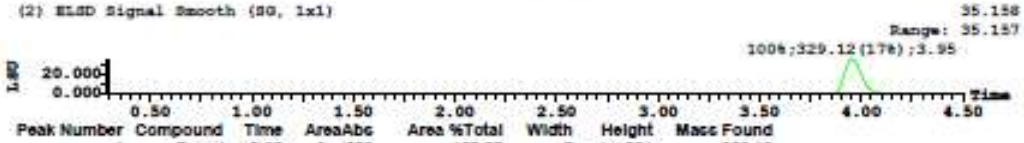

MS ES+ :330.12 Smooth (SG, 1x1)


UV Detector: TIC Smooth (Mn, 1x1)




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.87 | 1e+005 | 100.00 | 0 | 1e+006 | 329.12 |

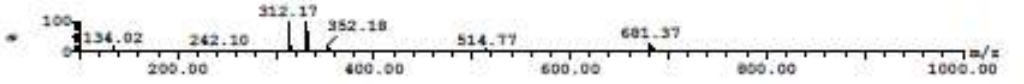
(2) ELSD Signal Smooth (SG, 1x1)

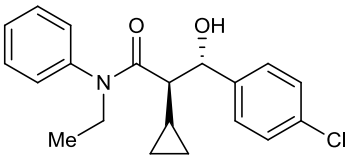



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.95 | 3e+003 | 100.00 | 0 | 4e+004 | 329.12 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.99 | 329.12 |

SAMPLE: 5,1:3,B 1:(Time: 3.96)Combine (232:238-(199:201+269:271))






**52**

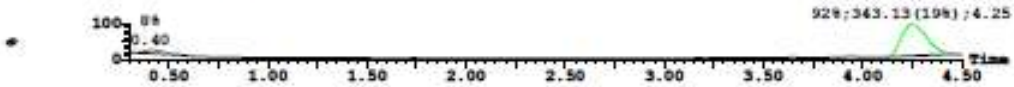
Chemical Formula: $C_{20}H_{22}ClNO_2$
Exact Mass: 343.13



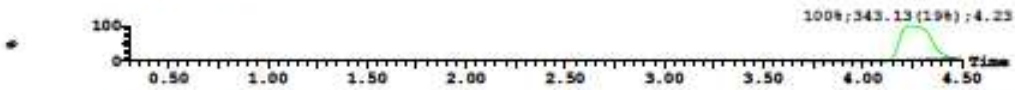
Date:31-May-2011 File:284 B P

**Sample Report:**

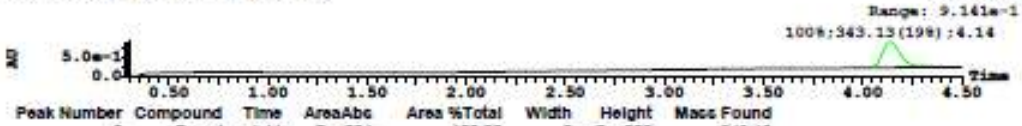
MS ES+ :TIC Smooth (SG, 1x1) 6.0e+008




MS ES+ :344.13 Smooth (SG, 1x1) 9.3e+007

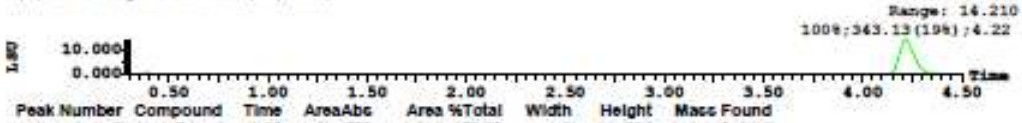



UV Detector: TIC Smooth (Mn, 1x1) 9.144e-1
Range: 9.141e-1




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.14 | 7e+004 | 100.00 | 0 | 7e+005 | 343.13 |

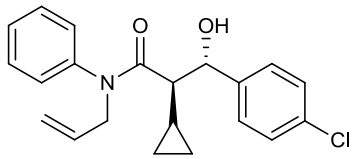
(2) ELSD Signal Smooth (SG, 1x1) 14.211
Range: 14.210




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.22 | 1e+003 | 100.00 | 0 | 1e+004 | 343.13 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 4.25 | 343.13 |

SAMPLE: 5,1:4,B 2:(Time: 4.23)Combine (240:254-(215:217+285:287)) 1:MS ES+ 8.2e+007



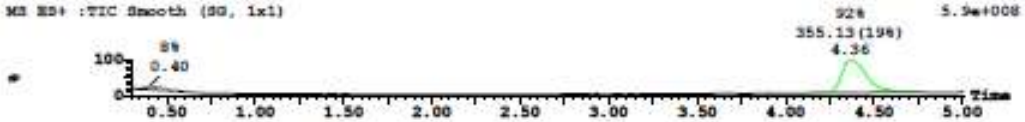

**53**

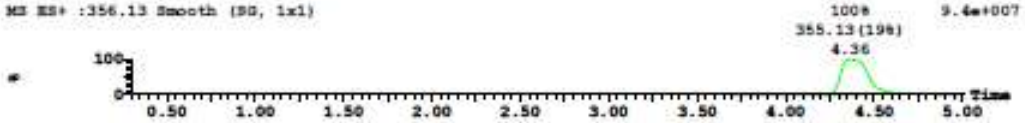
Chemical Formula: $C_{21}H_{22}ClNO_2$
Exact Mass: 355.13

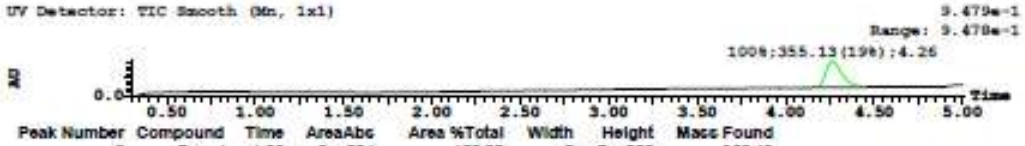


Date:31-May-2011 File:284 D P

**Sample Report:**



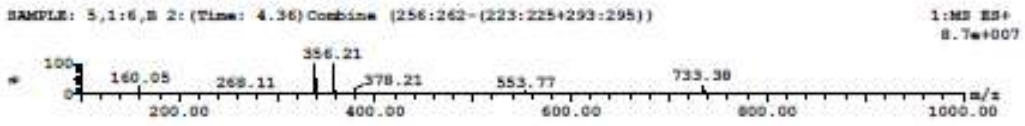



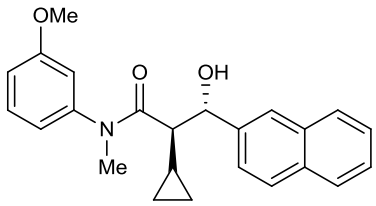




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.26 | 8e+004 | 100.00 | 0 | 7e+005 | 355.13 |


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.35 | 1e+003 | 100.00 | 0 | 2e+004 | 355.13 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 4.36 | 355.13 |





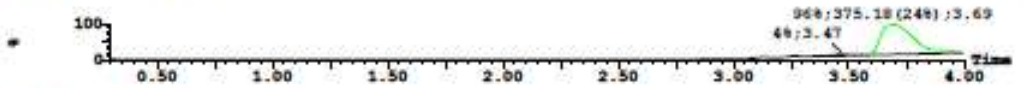

**54**

Chemical Formula: $C_{24}H_{25}NO_3$
Exact Mass: 375.18

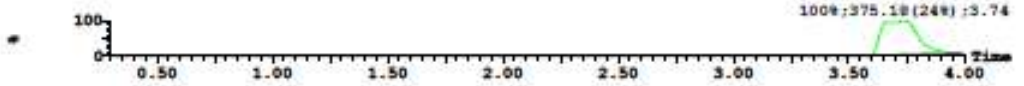

Date:25-May-2011 File:275 C P

**Sample Report:**

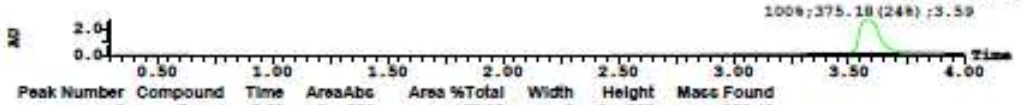
MS ES+ :TIC Smooth (SG, 1x1) 5.1e+008




MS ES+ :376.18 Smooth (SG, 1x1) 9.5e+007

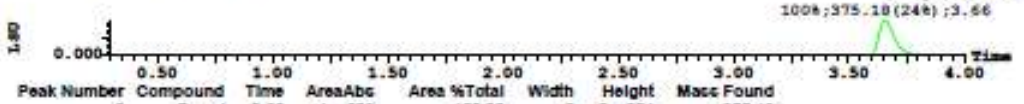



UV Detector: TIC Smooth (Mn, 1x1) 2.677
Range: 2.676




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.59 | 3e+005 | 100.00 | 0 | 2e+006 | 375.18 |

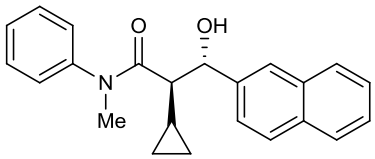
(2) ELSD Signal Smooth (SG, 1x1) 49.477
Range: 49.476




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.66 | 4e+003 | 100.00 | 0 | 5e+004 | 375.18 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.69 | 375.18 |

SAMPLE: 5,1:3,A 2:(Time: 3.74)Combine (219:225-(186:189+256:258)) 1:MS ES+
8.7e+007




**55**

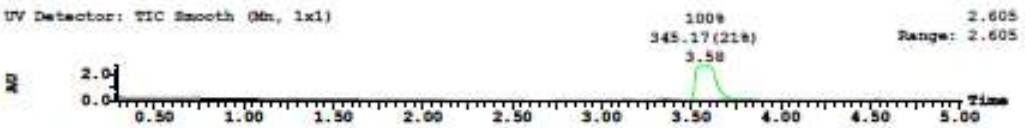
Chemical Formula: $C_{23}H_{23}NO_2$
Exact Mass: 345.17



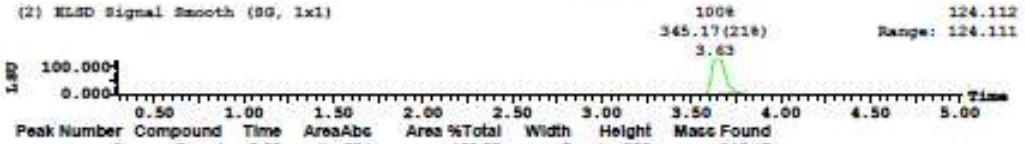
Date:25-May-2011 File:275 A.P

**Sample Report:**



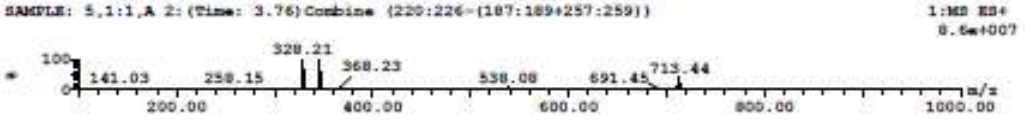


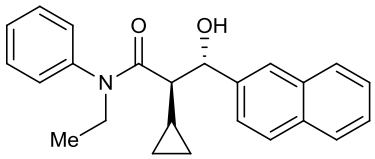

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.58 | 3e+005 | 100.00 | 0 | 3e+006 | 345.17 |




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.63 | 1e+004 | 100.00 | 0 | 1e+005 | 345.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.71 | 345.17 |






**56**

Chemical Formula: $C_{24}H_{25}NO_2$
Exact Mass: 359.19



Date:25-May-2011 File:275 B P

**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 5.8e+008

97%;359.19(21%);3.77

MS ES+ :360.19 Smooth (SG, 1x1) 9.2e+007

100%;359.19(21%);3.82

UV Detector: TIC Smooth (Mn, 1x1) 2.687
Range: 2.687

100%;359.19(21%);3.67

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.67 | 3e+006 | 100.00 | 1 | 2e+006 | 359.19 |

(2) ELSD Signal Smooth (SG, 1x1) 117.184
Range: 117.183

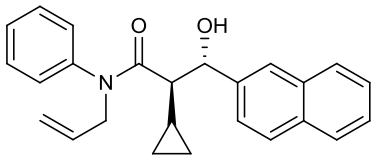
100%;359.19(21%);3.72

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.72 | 8e+003 | 100.00 | 0 | 1e+005 | 359.19 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.77 | 359.19 |

SAMPLE: 5,1:2,A 2:(Time: 3.82)Combine (224:230-(191:193+261:263)) 1:MS ES+ 8.6e+007

342.22
148.02
272.17
362.25
559.11
719.46
741.45
m/z




**57**

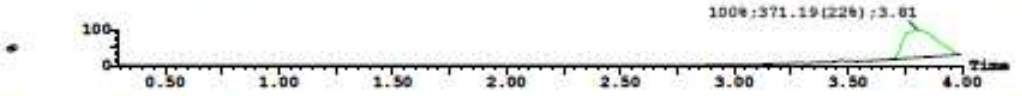
Chemical Formula: $C_{25}H_{25}NO_2$
Exact Mass: 371.19



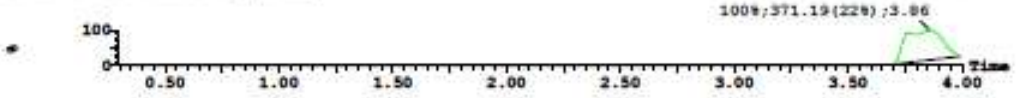
Date:25-May-2011 File:275 D P

**Sample Report:**

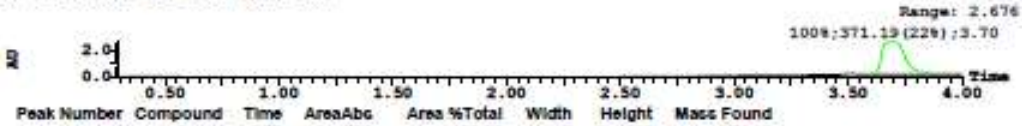
MS ES+ :TIC Smooth (SG, 1x1) 5.6e+008




MS ES+ :372.19 Smooth (SG, 1x1) 9.7e+007

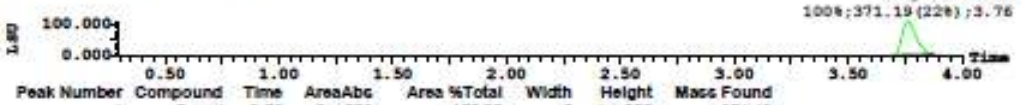



UV Detector: TIC Smooth (Mn, 1x1) 2.676
Range: 2.676




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.70 | 3e+005 | 100.00 | 0 | 2e+006 | 371.19 |

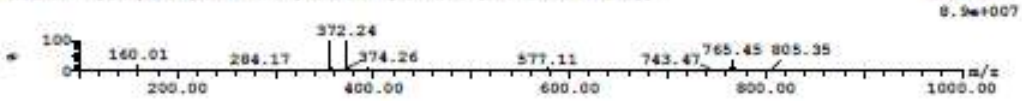
(2) ELSD Signal Smooth (SG, 1x1) 110.817
Range: 110.816

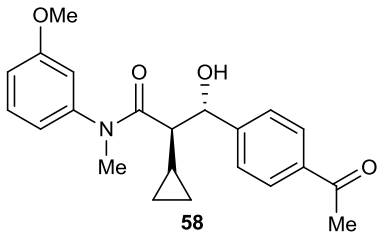



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.76 | 8e+003 | 100.00 | 0 | 1e+005 | 371.19 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.81 | 371.19 |

SAMPLE: 5,1:4,A 1:(Time: 3.86)Combine (226:232-(193:195+263:265)) 1:MS ES+
8.9e+007





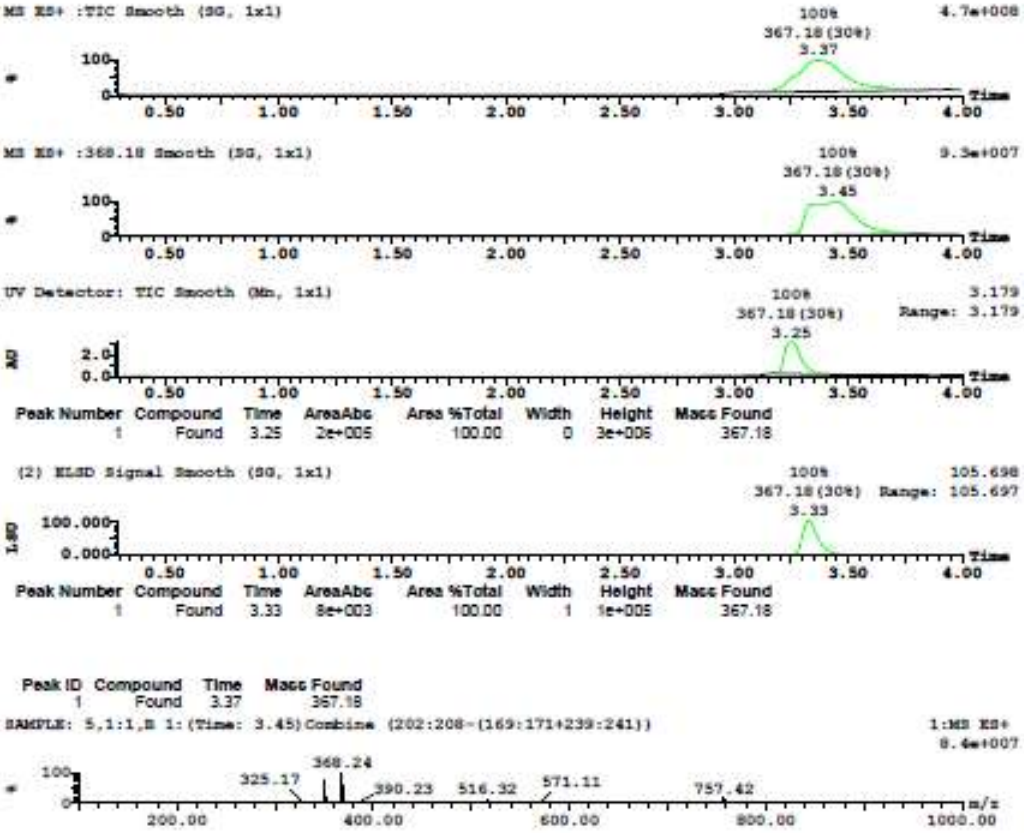

**58**

Chemical Formula: $C_{22}H_{25}NO_4$
Exact Mass: 367.18



Date:25-May-2011 File:276 C P

**Sample Report:**

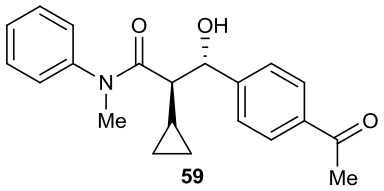



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.25 | 2e+006 | 100.00 | 0 | 3e+006 | 367.18 |

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.33 | 8e+003 | 100.00 | 1 | 1e+005 | 367.18 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.37 | 367.18 |

SAMPLE: 5,1:1,B 1:(Time: 3.45)Combine (202:208-(169:171+239:241))




**59**

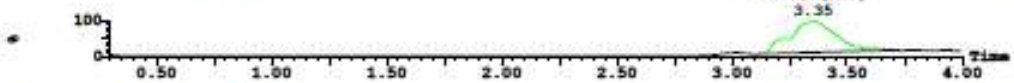
Chemical Formula: $C_{21}H_{23}NO_3$
Exact Mass: 337.17



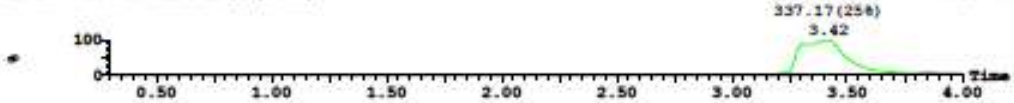
Date:25-May-2011 File:276 A.P

**Sample Report:**

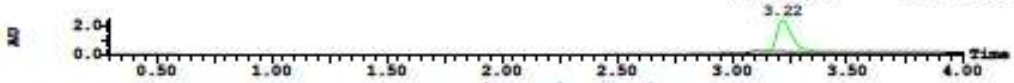
MS ES+ :TIC Smooth (SG, 1x1) 100% 5.4e+008
337.17(25%)
3.35




MS ES+ :338.17 Smooth (SG, 1x1) 100% 9.5e+007
337.17(25%)
3.42

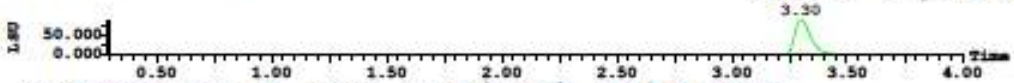



UV Detector: TIC Smooth (Mn, 1x1) 100% 2.444
337.17(25%) Range: 2.444
3.22




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.22 | 2e+005 | 100.00 | 0 | 2e+006 | 337.17 |

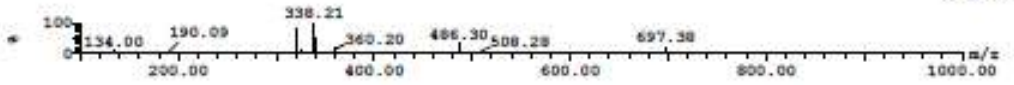
(2) ELSD Signal Smooth (SG, 1x1) 100% 87.314
337.17(25%) Range: 87.314
3.30

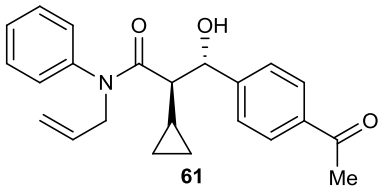



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.30 | 7e+003 | 100.00 | 0 | 9e+004 | 337.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.35 | 337.17 |

SAMPLE: 5,1:5,A 1:(Time: 3.42) Combine (200:206-(167:169+237:239)) 1:MS ES+
8.5e+007






**61**

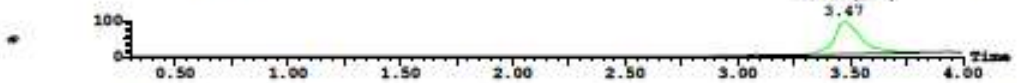
Chemical Formula: $C_{23}H_{25}NO_3$
Exact Mass: 363.18

-
Date:25-May-2011 File:276 D P

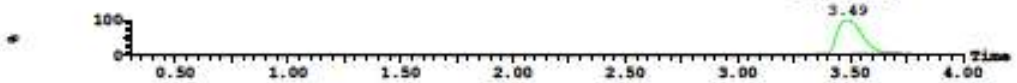


**Sample Report:**

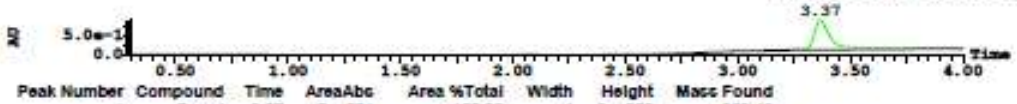
MS ES+ :TIC Smooth (SG, 1x1) 5.3e+008
100% 363.18(22%) 3.47




MS ES+ :364.18 Smooth (SG, 1x1) 9.4e+007
100% 363.18(22%) 3.49

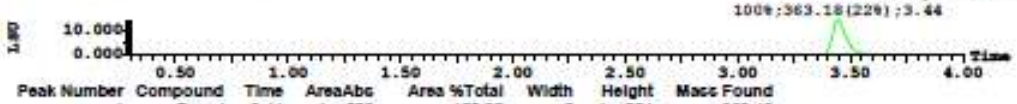



UV Detector: TIC Smooth (Mn, 1x1) 8.952e-1
100% 363.18(22%) 3.37 Range: 8.95e-1




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.37 | 7e+004 | 100.00 | 1 | 8e+005 | 363.18 |

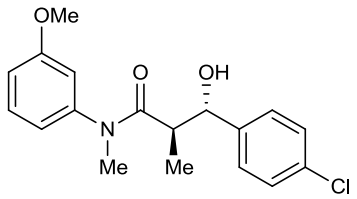
(2) ELSD Signal Smooth (SG, 1x1) 14.110
Range: 14.109
100%;363.18(22%);3.44




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.44 | 1e+003 | 100.00 | 0 | 1e+004 | 363.18 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.47 | 363.18 |

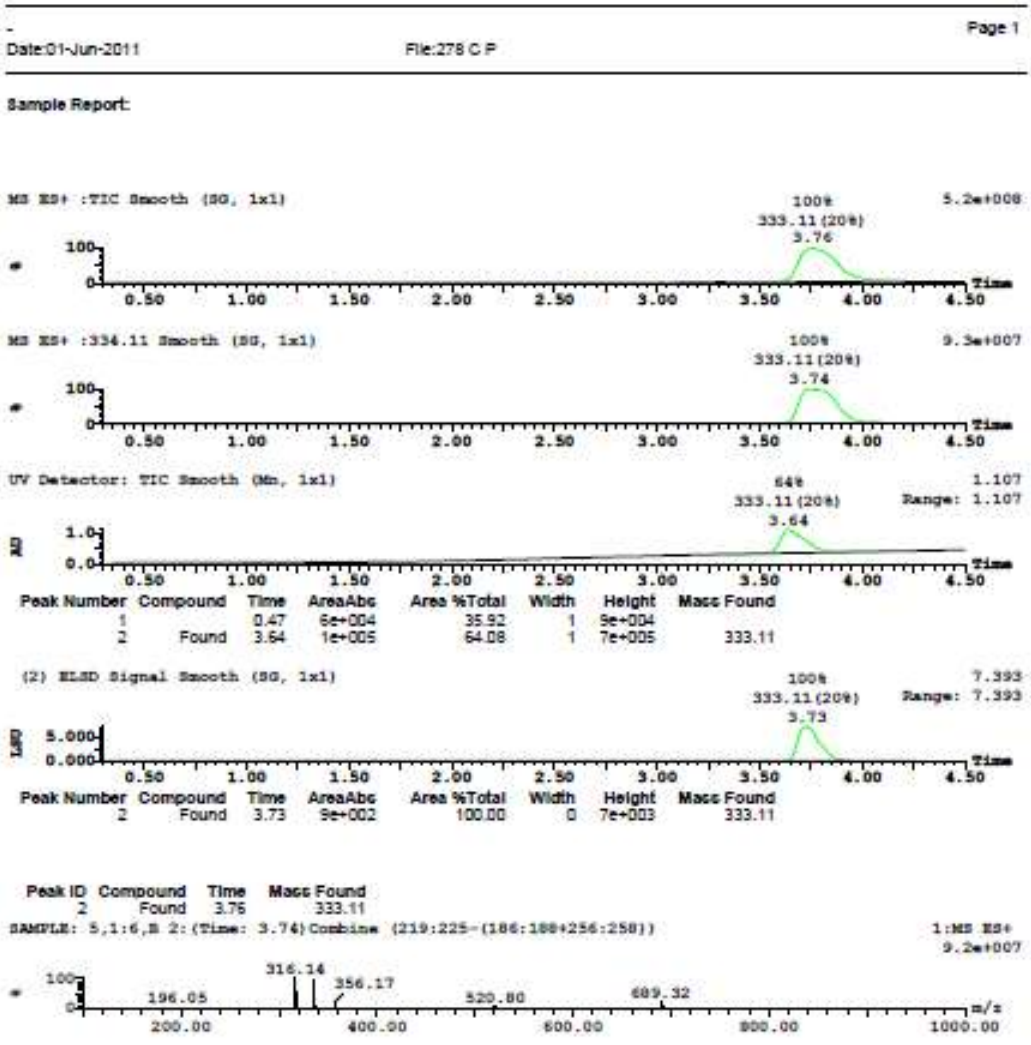
SAMPLE: 5,1:2,B 1:(Time: 3.49) Combine (204:210-(171:173+241:243)) 1:MS ES+
9.3e+007

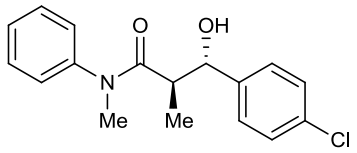



**62**

Chemical Formula: $C_{18}H_{20}ClNO_3$

Exact Mass: 333.11






**63**

Chemical Formula: $C_{17}H_{18}ClNO_2$

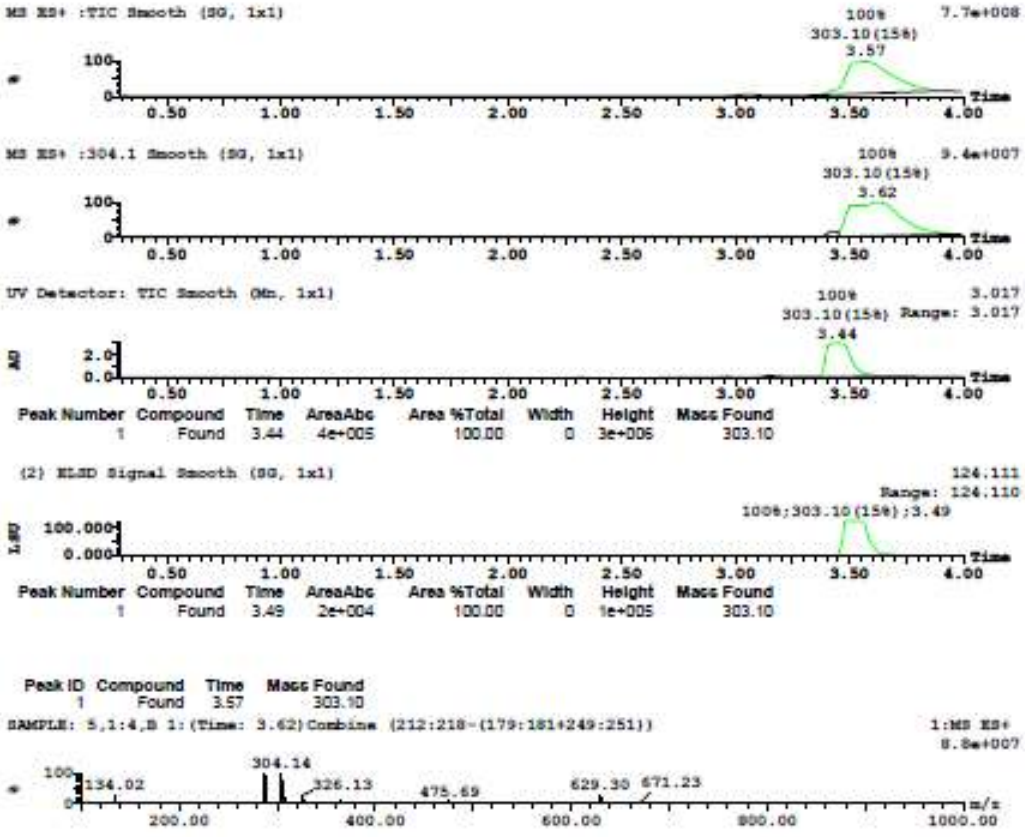
Exact Mass: 303.10

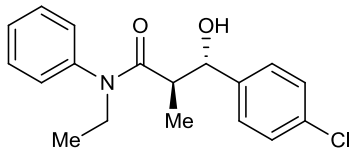
-

Page 1

Date:25-May-2011 File:278 A.P

**Sample Report:**






**64**

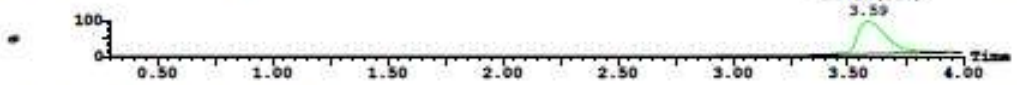
Chemical Formula: $C_{18}H_{20}ClNO_2$
Exact Mass: 317.12



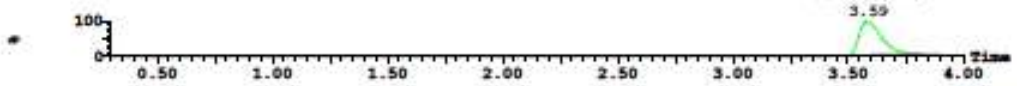
Date:25-May-2011 File:278 B P

**Sample Report:**

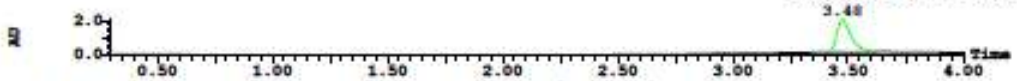
MS ES+ :TIC Smooth (SG, 1x1) 100% 317.12(13%) 3.59 6.5e+008




MS ES+ :318.12 Smooth (SG, 1x1) 100% 317.12(13%) 3.59 8.2e+007

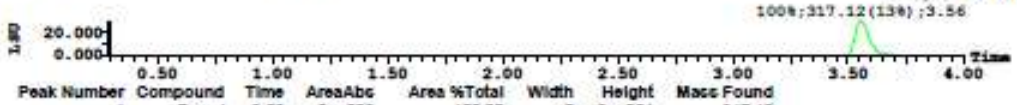



UV Detector: TIC Smooth (Mn, 1x1) 100% 317.12(13%) 3.48 2.105 Range: 2.105




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.48 | 1e+005 | 100.00 | 0 | 2e+006 | 317.12 |

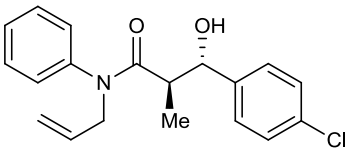
(2) ELSD Signal Smooth (SG, 1x1) 100%;317.12(13%);3.56 29.454 Range: 29.454




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | Found | 3.56 | 2e+003 | 100.00 | 0 | 3e+004 | 317.12 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 1 | Found | 3.59 | 317.12 |

SAMPLE: 5,1:5,B 1:(Time: 3.59) Combine (210:216-(177:179+247:249)) 1:MS ES+ 8.9e+007




**65**

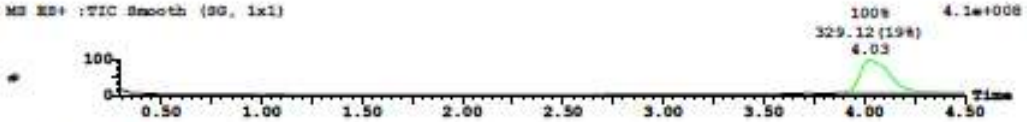
Chemical Formula: $C_{19}H_{20}ClNO_2$

Exact Mass: 329.12

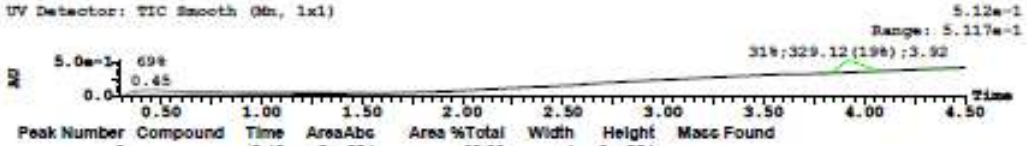


Date:01-Jun-2011 File:278 D F

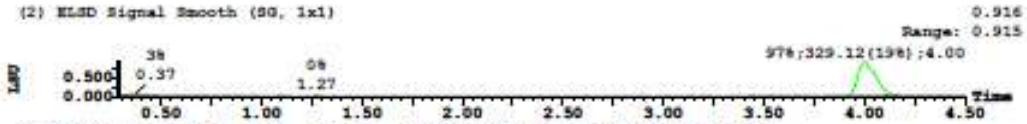
Sample Report:





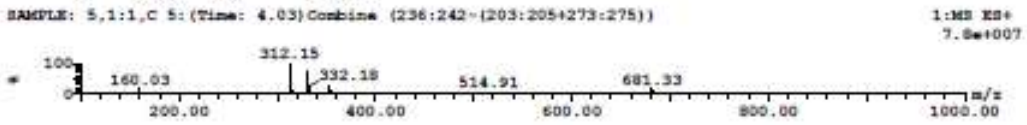


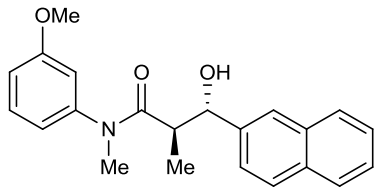

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | | 0.45 | 5e+004 | 68.66 | 1 | 8e+004 | |
| 5 | Found | 3.92 | 2e+004 | 31.34 | 1 | 2e+005 | 329.12 |




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.37 | 3e+000 | 2.68 | 0 | 4e+001 | |
| 3 | | 1.27 | 4e-001 | 0.44 | 0 | 5e+001 | |
| 4 | | 1.29 | 2e-001 | 0.15 | 0 | 2e+001 | |
| 5 | Found | 4.00 | 9e+001 | 96.73 | 0 | 9e+002 | 329.12 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 5 | Found | 4.03 | 329.12 |






**66**

Chemical Formula: $C_{22}H_{23}NO_3$

Exact Mass: 349.17

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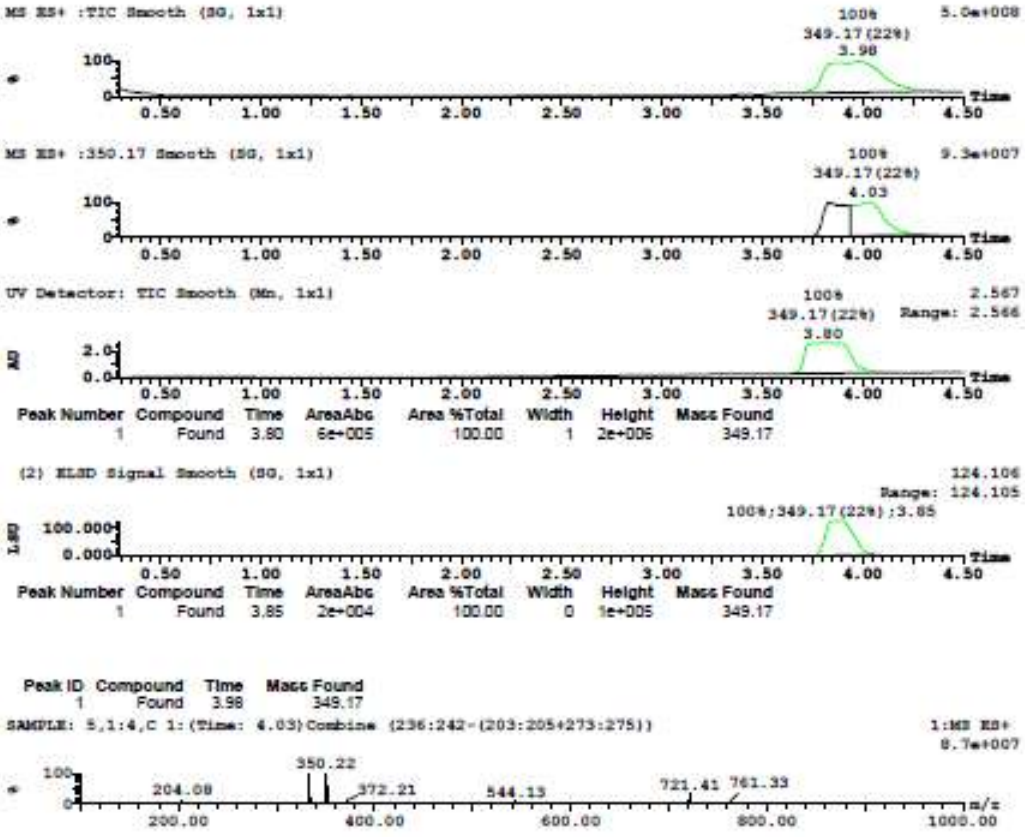
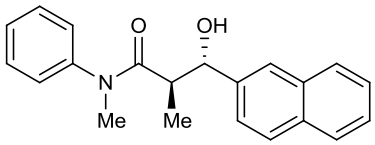
Page 1

Date:01-Jun-2011 File:279 C P

Sample Report:






**67**

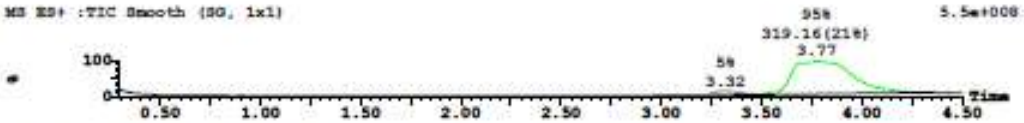
Chemical Formula: $C_{21}H_{21}NO_2$

Exact Mass: 319.16



Date:01-Jun-2011 File:279 A P

**Sample Report:**

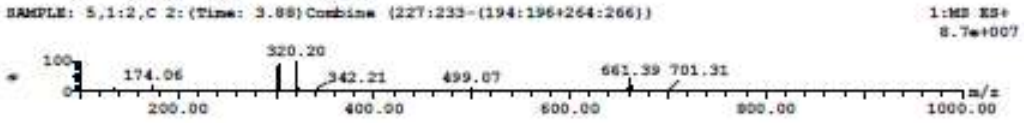


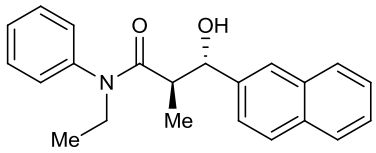



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.65 | 6e+005 | 100.00 | 1 | 2e+006 | 319.16 |


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.69 | 2e+004 | 100.00 | 0 | 1e+005 | 319.16 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.77 | 319.16 |






**68**

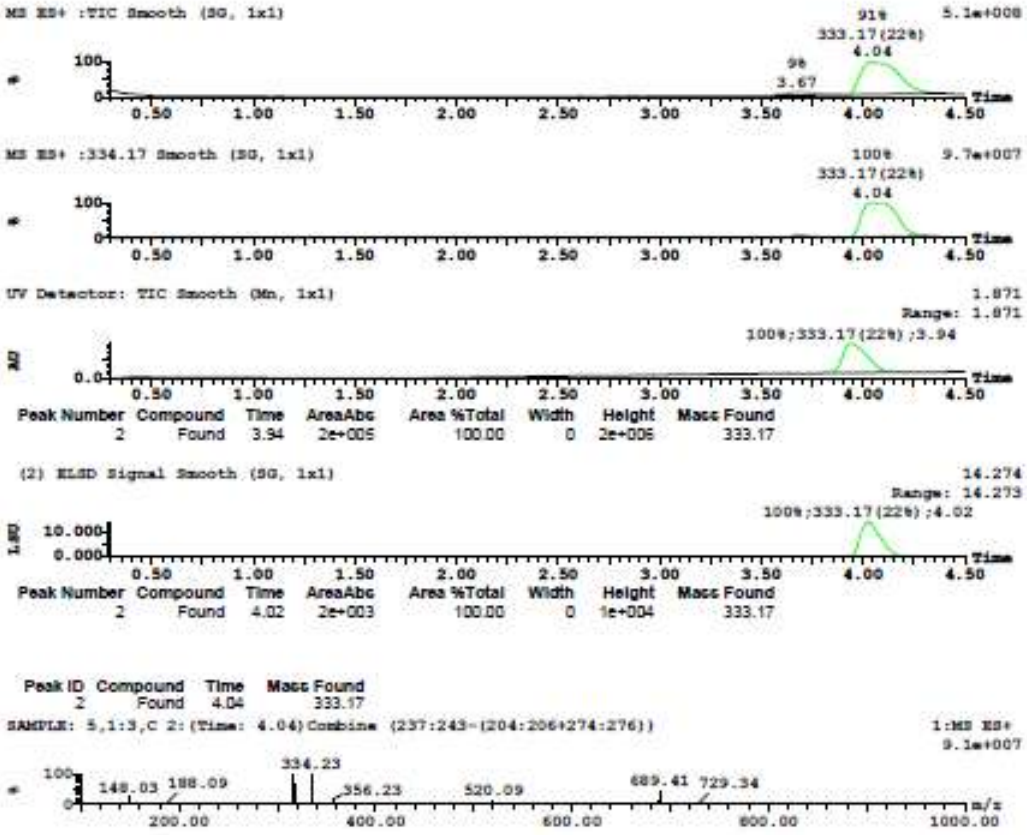
Chemical Formula: $C_{22}H_{23}NO_2$

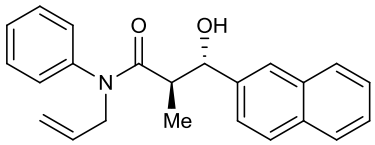
Exact Mass: 333.17



Date:01-Jun-2011 File:279 B P

**Sample Report:**






**69**

Chemical Formula: $C_{23}H_{23}NO_2$

Exact Mass: 345.17

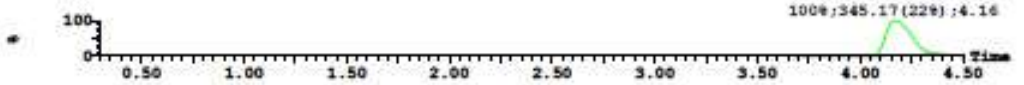


Date:01-Jun-2011 File:279 D P

**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 4.1e+008


MS ES+ :346.17 Smooth (SG, 1x1) 8.1e+007




UV Detector: TIC Smooth (Mn, 1x1) 1.151
Range: 1.151

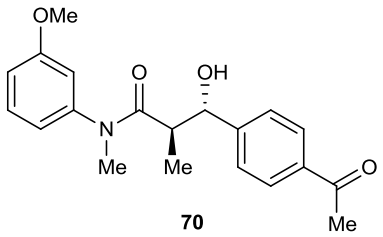

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.47 | 6e+004 | 41.91 | 1 | 9e+004 | |
| 2 | Found | 4.06 | 8e+004 | 58.09 | 1 | 6e+005 | 345.17 |

(2) ELSD Signal Smooth (SG, 1x1) 3.092
Range: 3.091


| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 4.14 | 3e+002 | 100.00 | 0 | 3e+003 | 345.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 4.16 | 345.17 |

SAMPLE: 5,1:5,C 2:(Time: 4.16)Combine (244:250-(211:213+281:283)) 1:MS ES+
8.9e+007




**70**

Chemical Formula: $C_{20}H_{23}NO_4$

Exact Mass: 341.16

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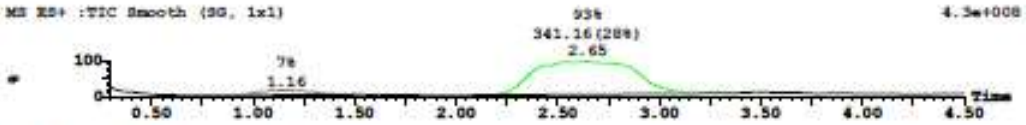
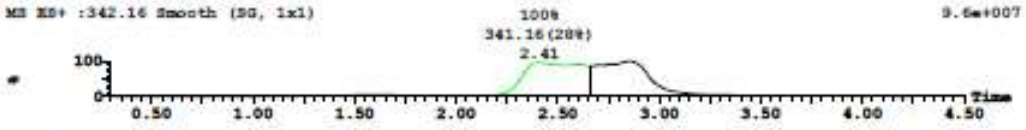
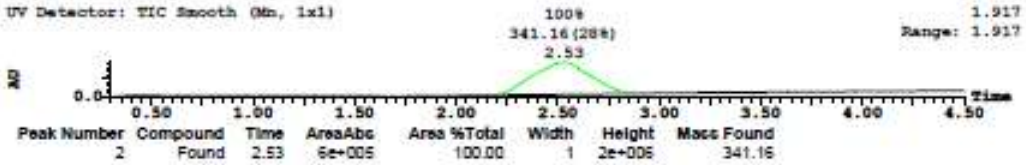
Page 1

Date:01-Jun-2011 File:280 C P

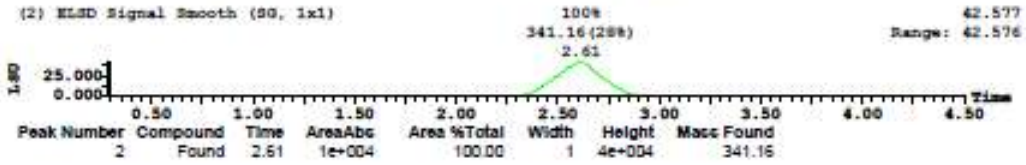
**Sample Report:**



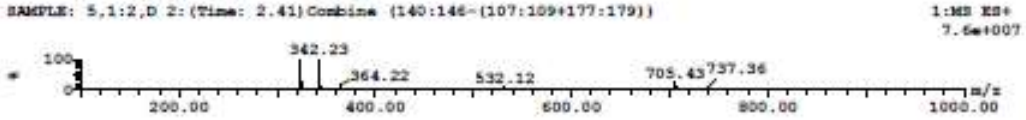



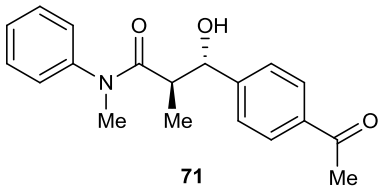

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 2.53 | 6e+005 | 100.00 | 1 | 2e+005 | 341.16 |




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 2.61 | 1e+004 | 100.00 | 1 | 4e+004 | 341.16 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 2.65 | 341.16 |






**71**

Chemical Formula: $C_{19}H_{21}NO_3$
Exact Mass: 311.15

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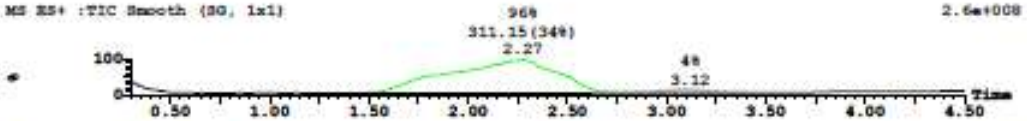
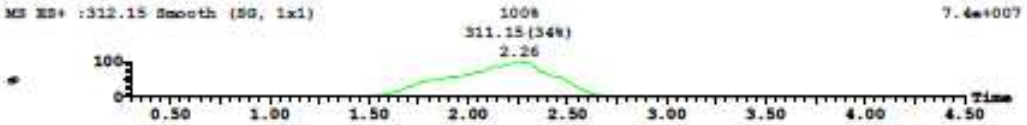
Page 1

Date:01-Jun-2011 File:280 A P

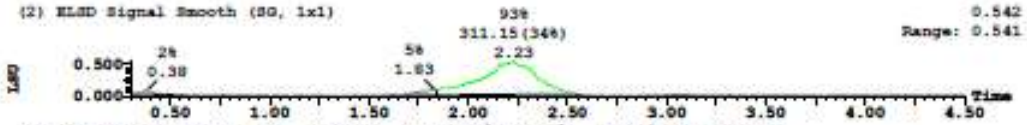
**Sample Report:**



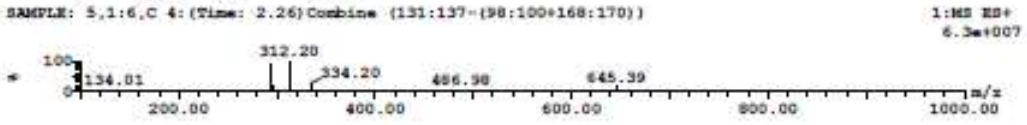


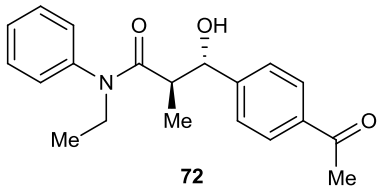
| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.40 | 5e+004 | 69.36 | 1 | 1e+005 | |
| 4 | Found | 2.19 | 2e+004 | 30.64 | 1 | 7e+004 | 311.15 |




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.38 | 3e+000 | 1.94 | 0 | 6e+001 | |
| 2 | | 1.63 | 1e-001 | 0.08 | 0 | 1e+001 | |
| 3 | | 1.83 | 9e+000 | 4.95 | 0 | 9e+001 | |
| 4 | Found | 2.23 | 2e+002 | 93.04 | 1 | 5e+002 | 311.15 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 4 | Found | 2.27 | 311.15 |






**72**

Chemical Formula: $C_{20}H_{23}NO_3$
Exact Mass: 325.17



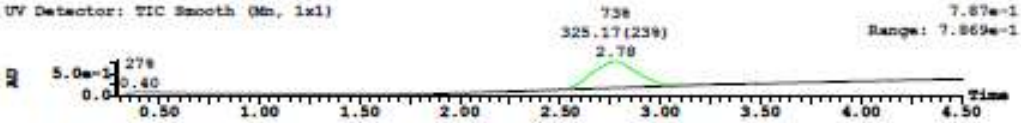
Date:01-Jun-2011 File:280 B P

**Sample Report:**

MS ES+ :TIC Smooth (SG, 1x1) 4.5e+008

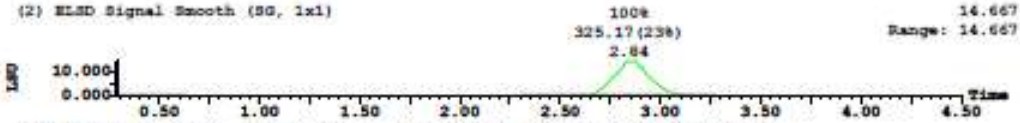

MS ES+ :326.17 Smooth (SG, 1x1) 9.5e+007


UV Detector: TIC Smooth (Mn, 1x1) 7.87e-1
Range: 7.869e-1




| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.40 | 6e+004 | 26.96 | 1 | 9e+004 | |
| 2 | Found | 2.78 | 2e+005 | 73.04 | 1 | 6e+005 | 325.17 |

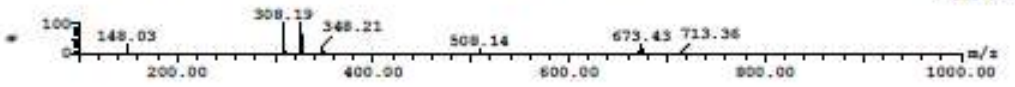
(2) ELSD Signal Smooth (SG, 1x1) 14.667
Range: 14.667

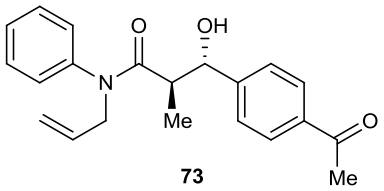



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 2.84 | 3e+003 | 100.00 | 1 | 1e+004 | 325.17 |

| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 2.93 | 325.17 |

SAMPLE: 5,1:1,D 2:(Time: 2.97)Combine (173:179-(140:142+210:212)) 1:MS ES+
9.2e+007





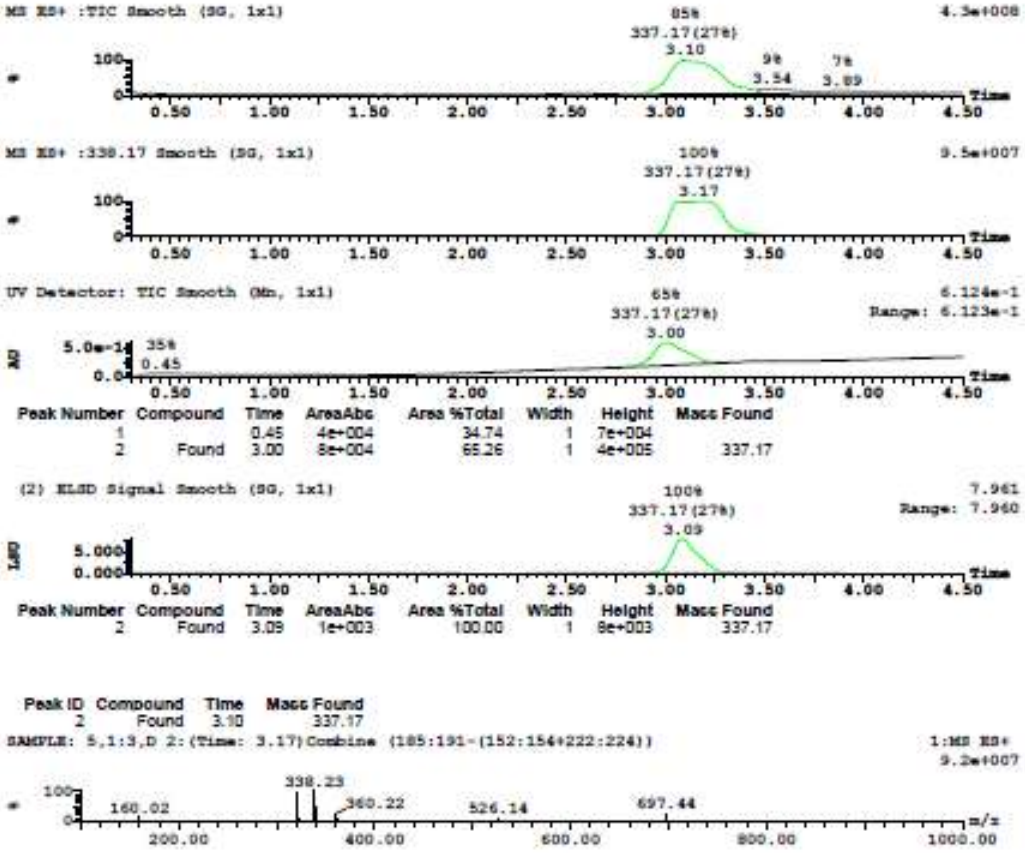

**73**

Chemical Formula: $C_{21}H_{23}NO_3$
Exact Mass: 337.17

-
Page 1

Date:01-Jun-2011 File:280 D P

**Sample Report:**

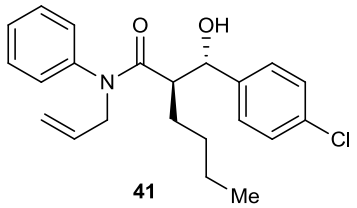



| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 1 | | 0.45 | 4e+004 | 34.74 | 1 | 7e+004 | |
| 2 | Found | 3.00 | 8e+004 | 65.26 | 1 | 4e+005 | 337.17 |

| Peak Number | Compound | Time | AreaAbs | Area %Total | Width | Height | Mass Found |
|---|---|---|---|---|---|---|---|
| 2 | Found | 3.09 | 1e+003 | 100.00 | 1 | 8e+003 | 337.17 |

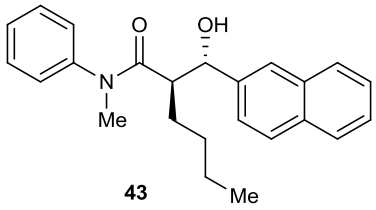
| Peak ID | Compound | Time | Mass Found |
|---|---|---|---|
| 2 | Found | 3.10 | 337.17 |

**Characterization of Randomly Selected Library Members**



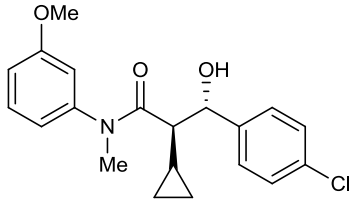

**41**

**(*RS*)-*N*-allyl-2-((*SR*)-(4-chlorophenyl)(hydroxy)methyl)-*N*-phenylhexanamide** (***anti*-41**). Following the standard procedure for the synthesis of library compounds, ***anti*-41** was obtained as a white solid. $^{1}$H NMR (500 MHz, $CD_3CN$, 320 K) δ 7.39-7.31 (m, 5H), 7.15 (d, *J* = 8.5 Hz, 2H); 6.88-6.72 (m, 2H), 5.80-5.69 (m, 1H), 5.04 (dd, *J* = 10.0, 1.5 Hz, 1H), 5.00 (dd, *J* = 17.0, 1.5 Hz, 1H), 4.77 (d, *J* = 7.0 Hz, 1H), 4.65-4.61 (m, 1H), 4.34-4.29 (m, 1H), 4.13-4.08 (m, 1H) 2.55-2.51 (m, 1H), 1.71-1.63 (m, 1H), 1.45-1.37 (m, 1H), 1.22-1.08 (m, 4H), 0.80 (t, *J* = 7.0 Hz, 3H) $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.3, 144.2, 142.8, 134.3, 133.5, 130.3, 129.8, 129.2, 129.0, 129.0, 118.1, 75.3, 52.7, 49.1, 31.2, 30.0, 23.3, 14.1




**43**

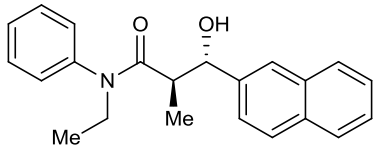
**(*RS*)-2-((*SR*)-hydroxy(naphthalen-2-yl)methyl)-*N*-methyl-*N*-phenylhexanamide** (***anti*-43**). Following the standard procedure for the synthesis of library compounds, ***anti*-43** was obtained as a white solid. $^{1}$H NMR (500 MHz, $CD_3CN$, 320 K) δ 7.92-7.88 (m, 1H), 7.86-7.82 (m, 2H), 7.66 (s, 1H), 7.55-7.48 (m, 2H), 7.31-7.21 (m, 4H), 6.75-6.68 (m, 2H), 4.82-4.78 (m, 2H), 3.10 (s, 3H), 2.74-2.69 (m, 1H), 1.74-1.67 (m, 1H), 1.45-1.38 (m, 1H), 1.22-1.05 (m, 4H), 0.79 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.6, 144.5, 142.9, 134.2,133.9, 130.3, 128.9, 128.8, 128.8, 128.7, 128.6, 127.3, 126.9, 126.0, 125.4, 76.2, 49.0, 37.4, 31.3, 30.1, 12.3, 14.2.




**50**

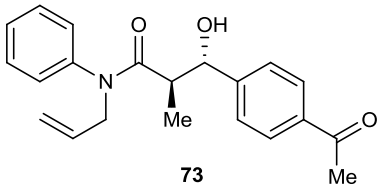
**(2*RS*,3*SR*)-3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-*N*-(3-methoxyphenyl)-*N*-methylpropanamide** (***anti*-50**). Following the standard procedure for the synthesis of library compounds, ***anti*-50** was obtained as a white solid. $^{1}$H NMR (500 MHz, $CD_3CN$, 330 K) δ 7.32 (d, *J* = 8.5 Hz, 2H), 7.23 (t, *J* = 8.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 2H), 6.87 (dd, *J* = 8.0, 2.5 Hz, 1H), 6.47-6.37 (m, 2H), 4.83-4.75 (m, 2H), 3.76 (s, 3H), 3.14 (s, 3H), 2.00-1.95 (m, 1H), 1.13-1.06 (m, 1H), 0.44-0.39 (m, 1H), 0.37-0.28 (m, 1H), 0.09-0.00 (m, 1H), -0.21- -0.31 (m, 1H); $^{13}$C

NMR (125 MHz, $CD_3CN$) δ 174.8, 161.3, 145.4, 144.3, 133.4, 131.1, 129.1, 129.0, 120.8, 114.5, 114.4, 76.3, 56.1, 53.4, 37.4, 13.0, 4.7, 4.1.




**68**

**(2*RS*,3*SR*)-*N*-ethyl-3-hydroxy-2-methyl-3-(naphthalen-2-yl)-*N*-phenylpropanamide** (***anti*-68**). Following the standard procedure for the synthesis of library compounds, ***anti*-68** was obtained as a white solid. $^1$H NMR (500 MHz, $CD_3CN$, 320 K) δ 7.91-7.87 (m, 1H), 7.85-7.81 (m, 2H), 7.67 (s, 1H), 7.53-7.47 (m, 2H), 7.35-7.24 (m, 4H), 6.79-6.63 (m, 2H), 4.83 (d, *J* = 7.0 Hz, 1H), 4.76-4.71 (m, 1H), 3.64 (dq, *J*= 14.0, 7.0 Hz, 1H), 3.44 (dq, *J*= 14.0, 7.0 Hz, 1H), 2.69-2.62 (m, 1H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.93 (t, *J* = 7.0 Hz, 3H) ; $^{13}$C NMR (125 MHz, $CD_3CN$) δ 175.8, 142.9, 142.7, 134.2, 133.9, 130.4, 129.4, 129.0, 128.8, 128.8, 128.6, 127.2, 126.8, 125.8, 125.4, 77.5, 44.5, 43.9, 16.2, 13.2.




**73**

**(2*RS*,3*SR*)-3-(4-acetylphenyl)-*N*-allyl-3-hydroxy-2-methyl-*N*-phenylpropanamide** (***anti*-73**). Following the standard procedure for the synthesis of library compounds, ***anti*-73** was obtained as a white solid. $^1$H NMR (500 MHz, $CD_3CN$, 320 K) δ 7.91 (d, *J* = 8.5 Hz, 2H), 7.39-7.35 (m, 3H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.92-6.84 (m, 2H), 5.78-5.69 (m, 1H), 5.06-4.96 (m, 2H), 4.69-4.63 (m, 2H), 4.24 (dd, *J* = 15.0, 6.0 Hz, 1H), 4.14 (dd, *J* = 15.0, 6.5 Hz, 1H), 2.69-2.61 (m, 1H), 2.57 (s, 3H), 1.08 (d, *J* = 7.0 Hz, 3H) ; $^{13}$C NMR (125 MHz, $CD_3CN$) δ 198.7, 175.8, 150.3, 142.9, 137.4, 134.3, 130.5, 129.4, 129.2, 129.1, 127.4, 117.9, 76.9, 52.5, 43.8, 27.1, 16.1.

## 5. Additional Product Characterization

### X-Ray Structure Determination of 5

**Data Collection**

An irregular broken fragment (0.32 x 0.28 x 0.24 mm) was selected under a stereo-microscope while immersed in Fluorolube oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered glass fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system at 100 K. Rotation and still images showed the diffractions to be sharp. Frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "full sphere" data set was obtained which samples approximately all of reciprocal space to a resolution of 0.75 Å using 0.3$^{o}$ steps in ω using 10 second integration times for each frame. Data was collected at 200 K. Cooling to 100 K resulted in crystal decomposition possibly due to a phase transition. Integration of intensities and refinement of cell parameters were done using SAINT. Absorption corrections were applied using SADABS based on redundant diffractions.[6]

**Structure solution and refinement**

The space group was determined as P1(bar) based on systematic absences and intensity statistics. Direct methods were used to locate all most C atoms from the E-map. Repeated difference Fourier maps allowed recognition of all expected C, N, O and F atoms. Following anisotropic refinement of all non-H atoms, ideal H-atom positions were calculated. The $CF_3$ group was disordered into two orientations with occupancies of 0.84 and 0.16, and they were refined as such. Final refinement was anisotropic for all non-H atoms, and isotropic-riding for H atoms. No other anomalous bond lengths or thermal parameters were noted. All ORTEP diagrams have been drawn with 50% probability ellipsoids. Further information is contained in the CIF file.

**Equations of interest:**

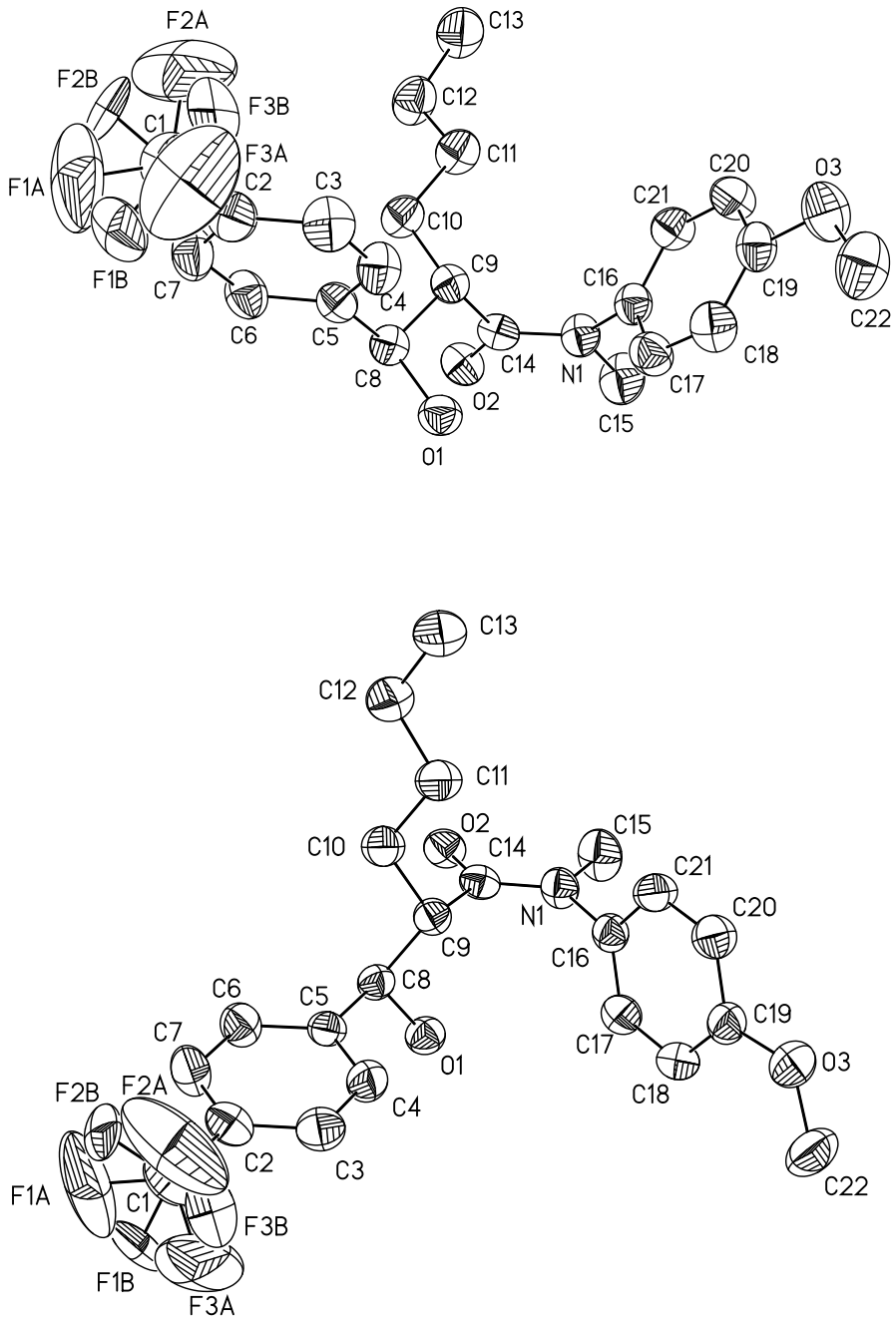
$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$ $\qquad$ $R1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

$wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$ $\qquad$ $GooF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$; $\qquad$ n = number of independent reflections;

q, a, b, P as defined in [5] $\qquad$ p = number of parameters refined.

[6] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).




**Supplementary Figure S3**. **Crystal structure of compound 5**. Both orientations of the $CF_3$ group are shown.

**Supplementary Table S1**. **Crystal data and structure refinement for compound 5** (**CCDC 810238**).

| | | |
|---|---|---|
| Identification Code | Mont01 | |
| CCDC Deposition Number | CCDC 810238 | |
| Empirical formula | $C_{22}H_{26}F_3NO_3$ | |
| Formula weight | 409.44 | |
| Crystallization Solvent | hexanes and ethyl acetate | |
| | **Data Collection** | |
| Temperature | 200 K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space Group | P1(bar) | |
| Unit cell dimensions | $a$ = 10.305(2) Å<br>$b$ = 11.201(3) Å<br>$c$ = 11.779(3) Å | $\alpha$ = 63.547(4) $^o$<br>$\beta$ = 65.449(4) $^o$<br>$\gamma$ = 66.927(4) $^o$ |
| Volume | 1071.0(4) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.270 $Mg/m^3$ | |
| Absorption coefficient | 0.101 $mm^{-1}$ | |
| F(000) | 432 | |
| Crystal size, color, habit | 0.32 x 0.28 x 0.24 mm, clear, fragment | |
| Theta range for data collection | 2.02 – 28.37 $^o$ | |
| Index ranges | $-13 \le h \le 13$, $-14 \le k \le 14$, $-15 \le l \le 15$ | |
| Reflections collected | 13,007 | |

| | |
|---|---|
| Independent reflections | 5,170 ($R_{int}$ = 0.0243) |

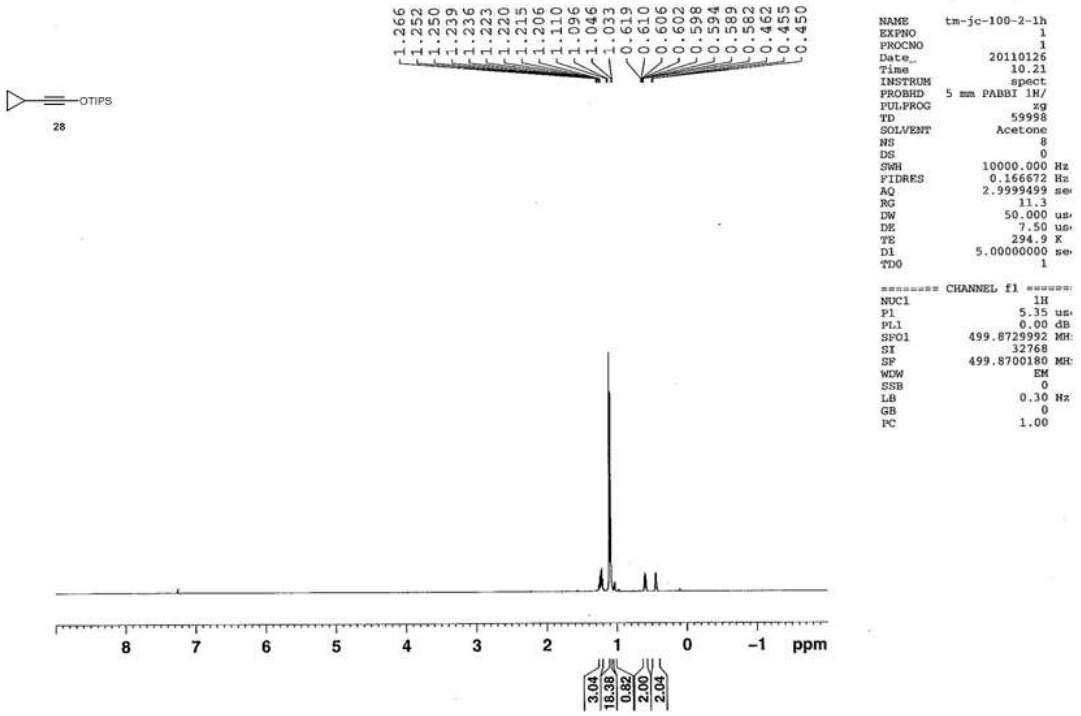
**Supplementary Table S1** (**cont**.)

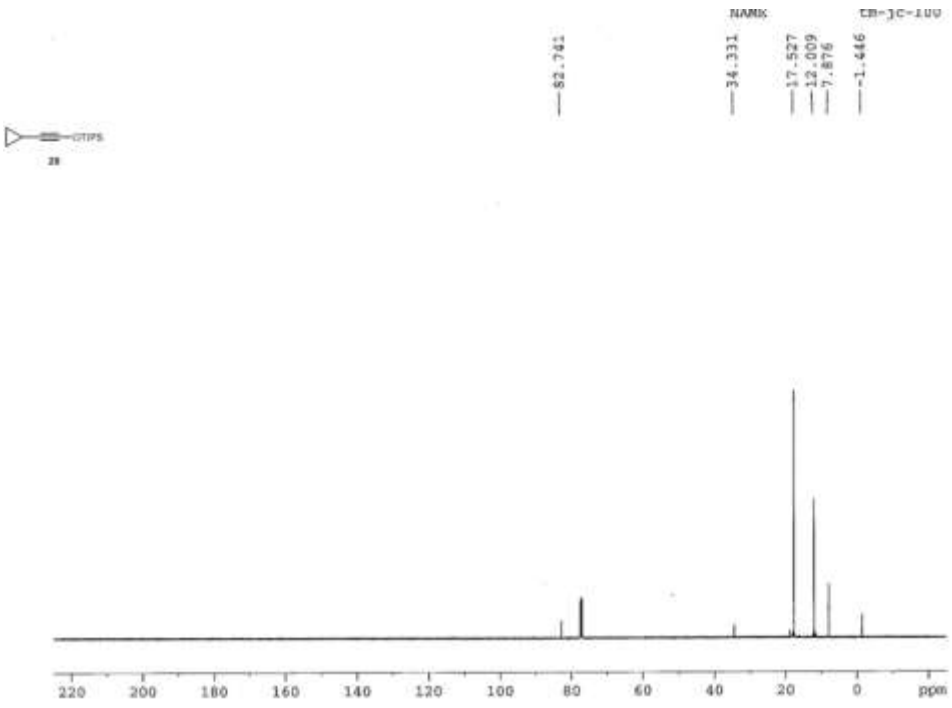
| | |
|---|---|
| Reflections with $I > 4\sigma(F_o)$ | 3,057 |
| Absorption correction | SADABS based on redundant diffractions |
| Max. and min. transmission | 1.0, 0.720 |

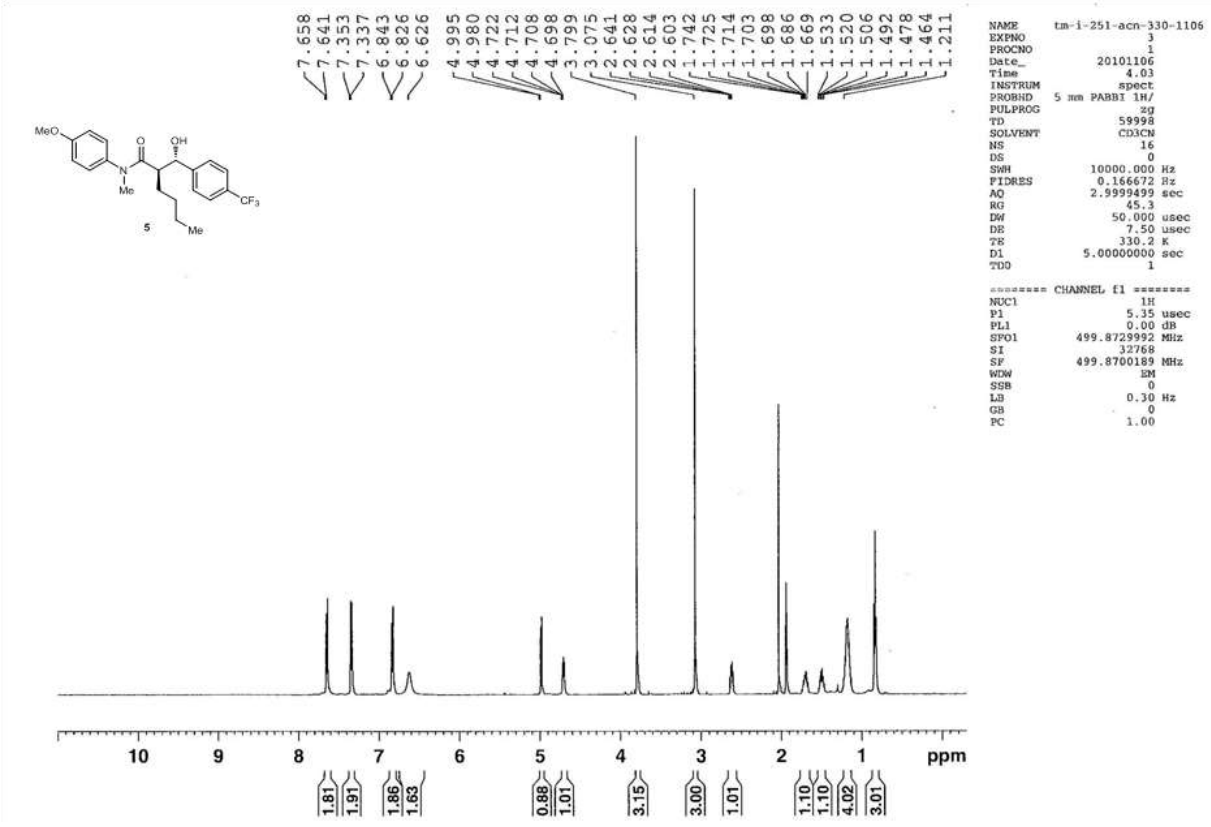
**Structure Solution and Refinement**

| | |
|---|---|
| Refinement method | Full-matrix least squares on $F^2$ |
| Weighting scheme | $w = q\,[\sigma^2 (F_o^2) + (aP)^2 + bP]^{-1}$ where:<br>$P = (F_o^2 + 2F_c^2)/3$, a = 0.0821, b = 0.0, q =1 |
| Data / restraints / parameters | 5170 / 0 / 293 |
| Goodness-of-fit on $F^2$ | 0.927 |
| Final R indices [I > 2 sigma(I)] | R1 = 0.0586, wR2 = 0.1402 |
| R indices (all data) | R1 = 0.0981, wR2 = 0.1594 |
| Largest diff. peak and hole | 0.311, -0.196 eÅ $^{-3}$ |

## NMR Spectra of New Compounds

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3.075
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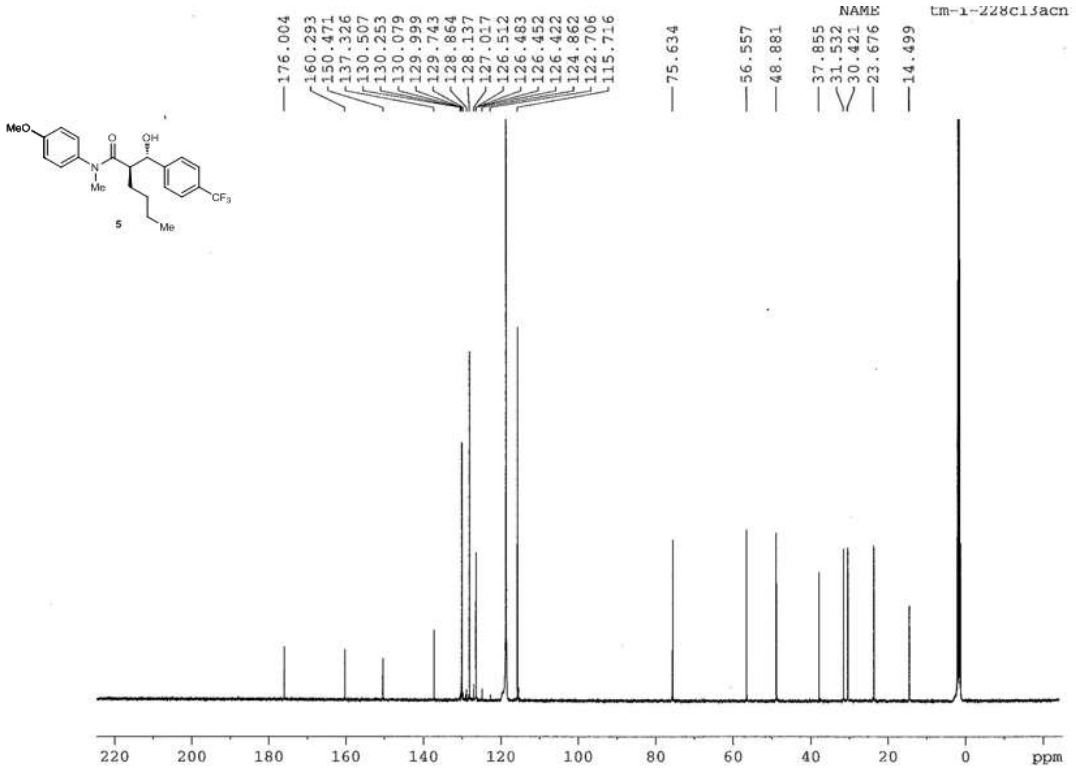
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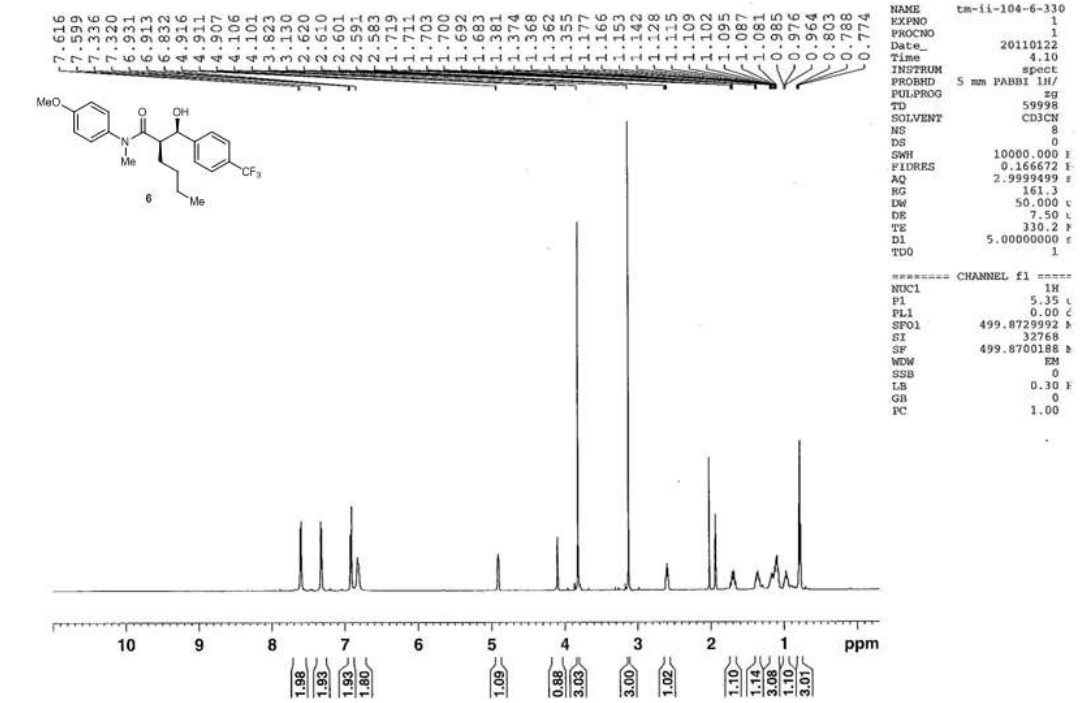
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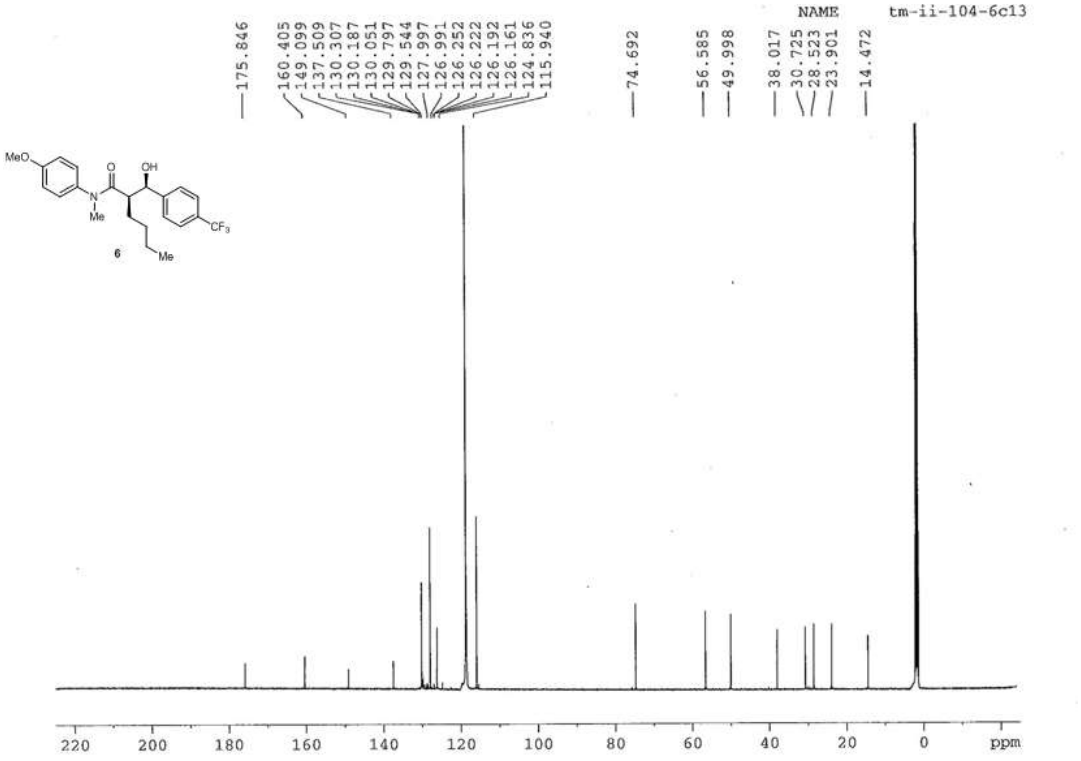
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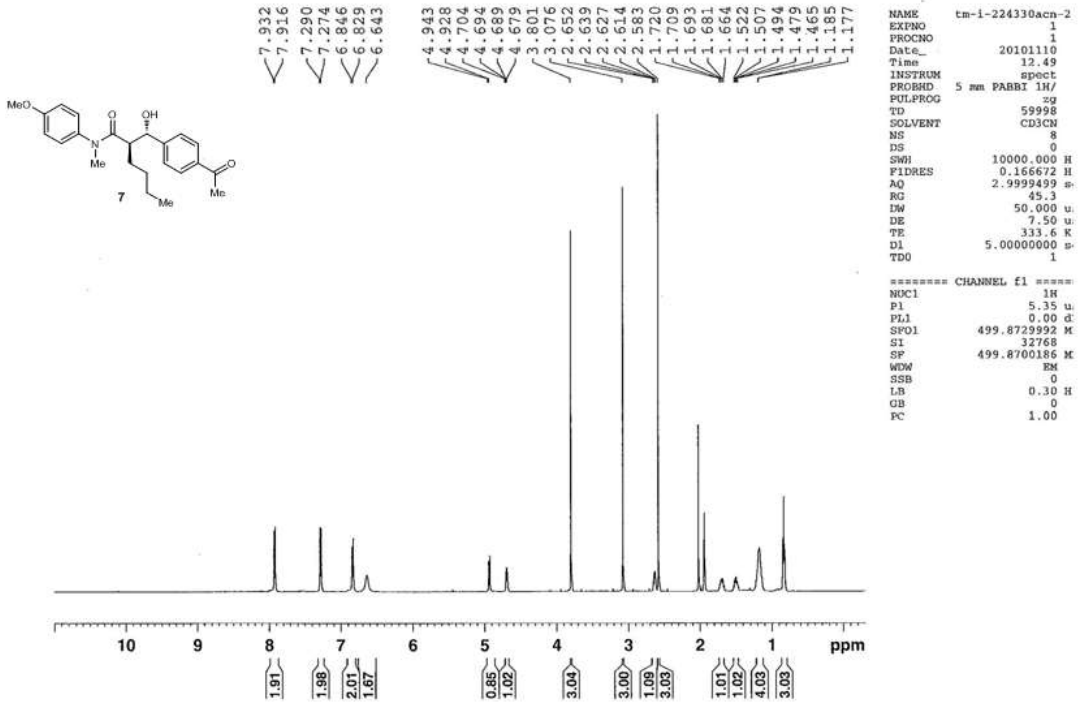
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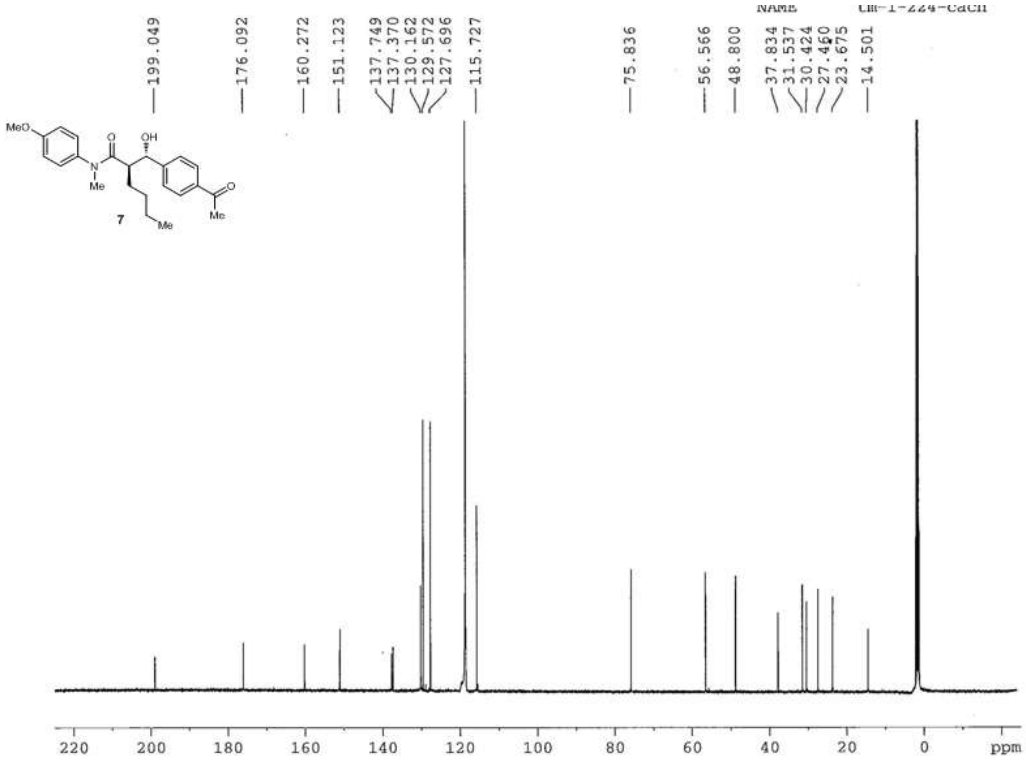
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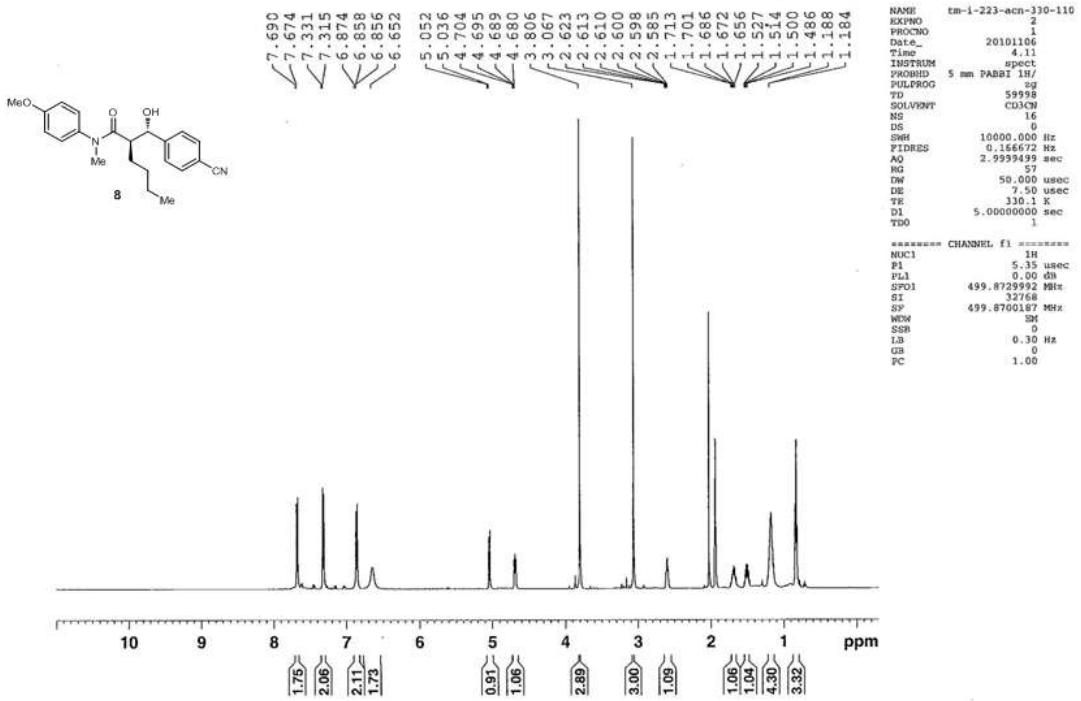
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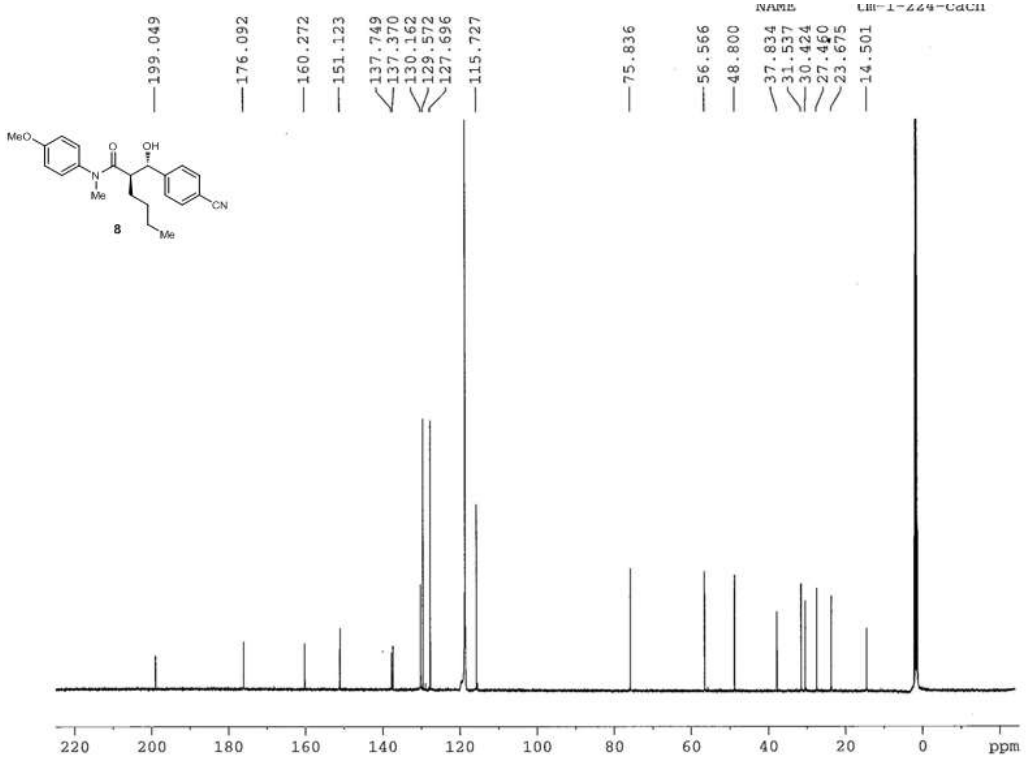
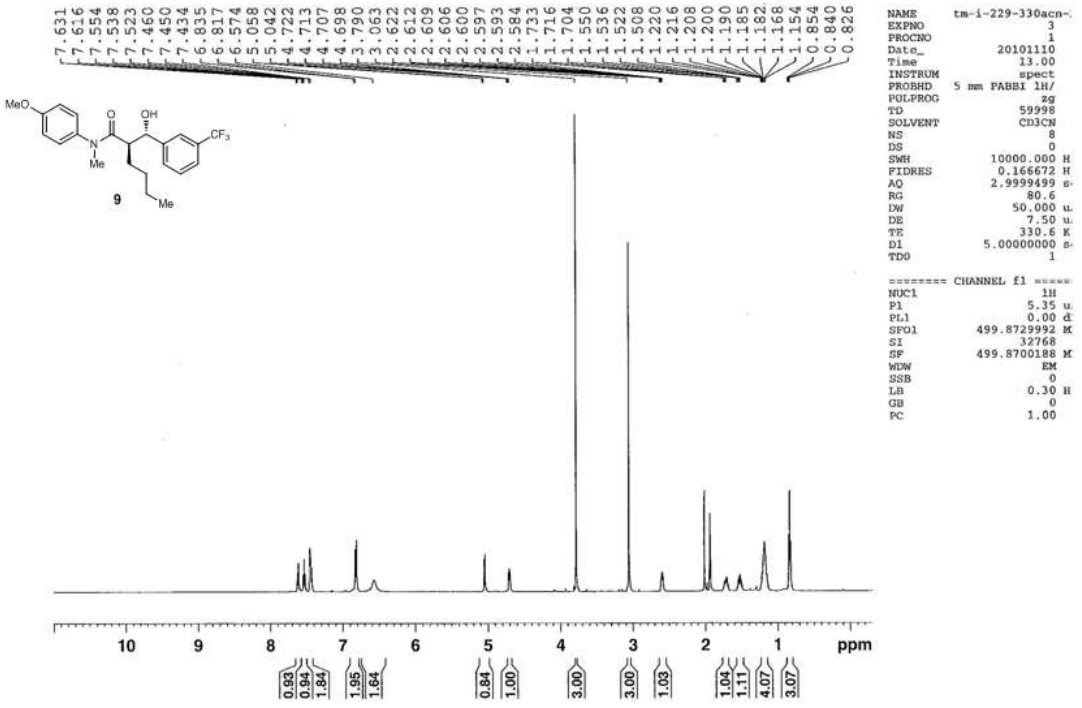
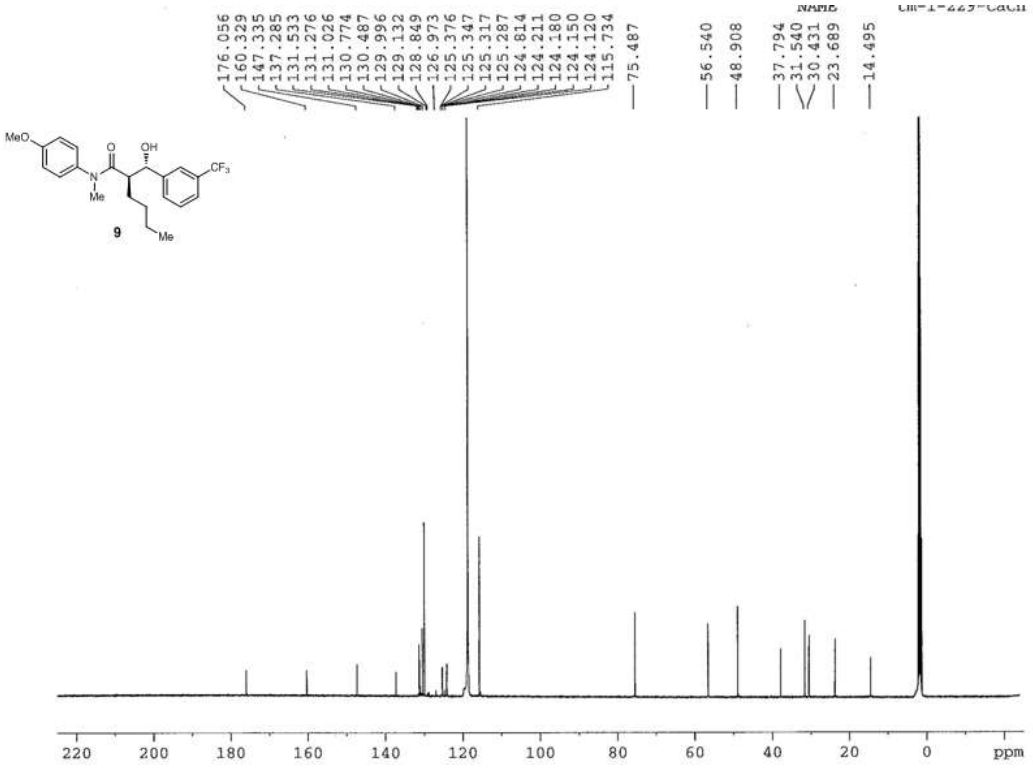
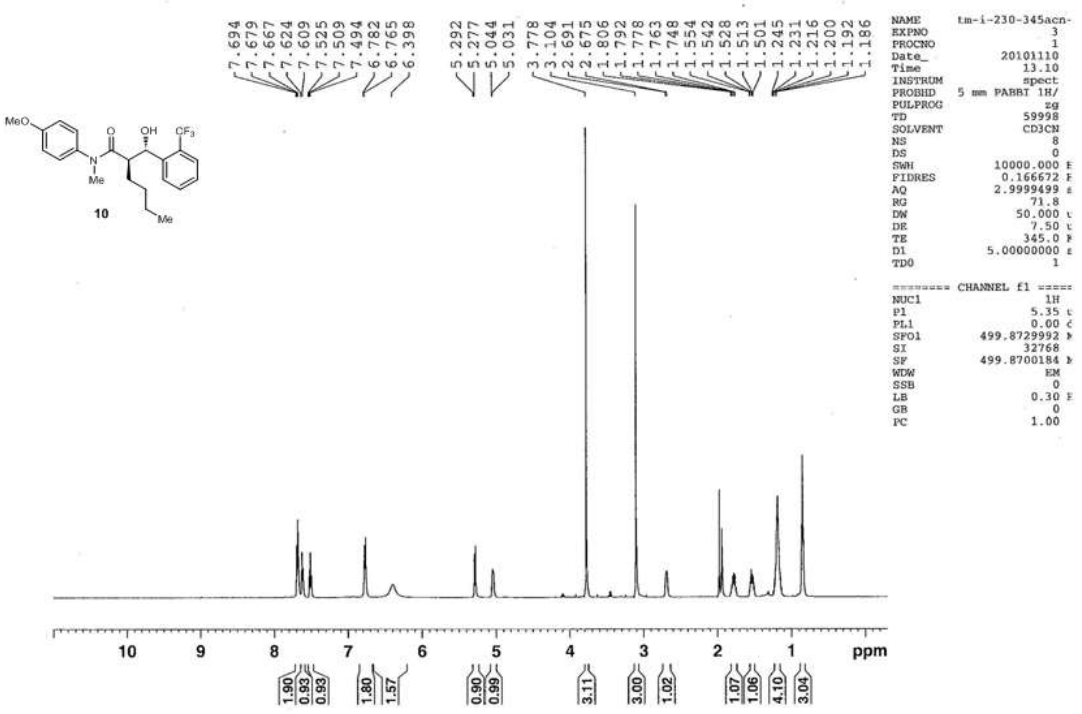
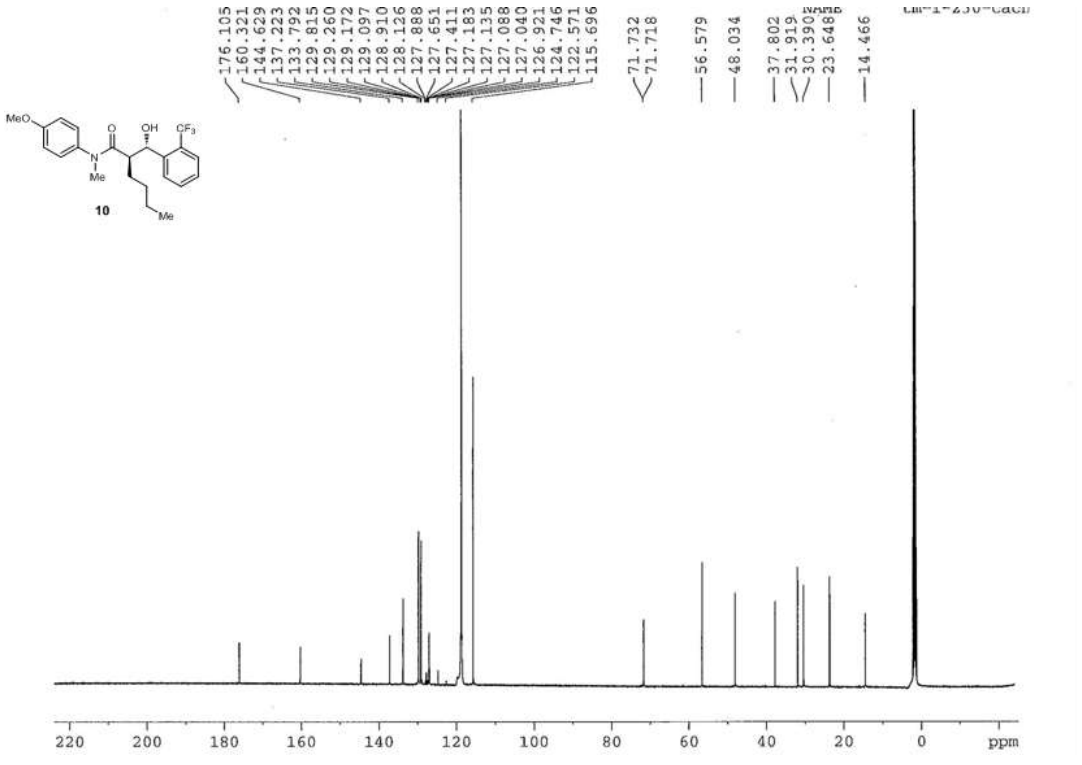
4.30
3.32

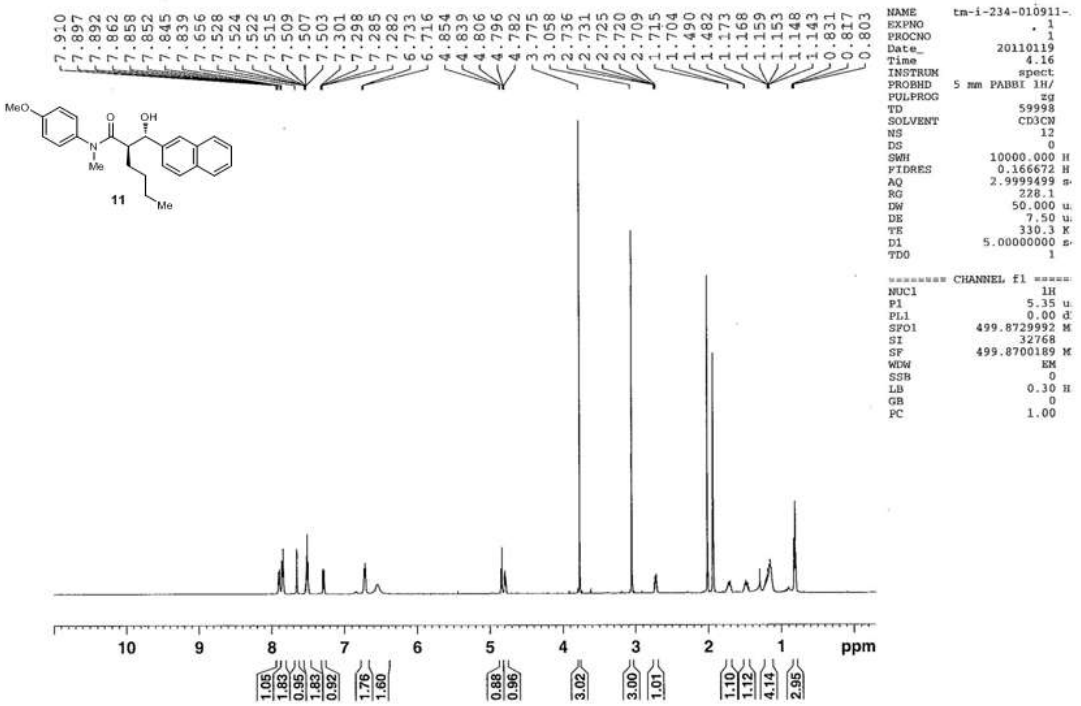

199.049
176.092
160.272
151.123
137.749
137.370
130.162
129.572
127.696
115.727
75.836
56.566
48.800
37.834
31.537
30.424
27.460
23.675
14.501
MeO
OH
N
Me
CN
8
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

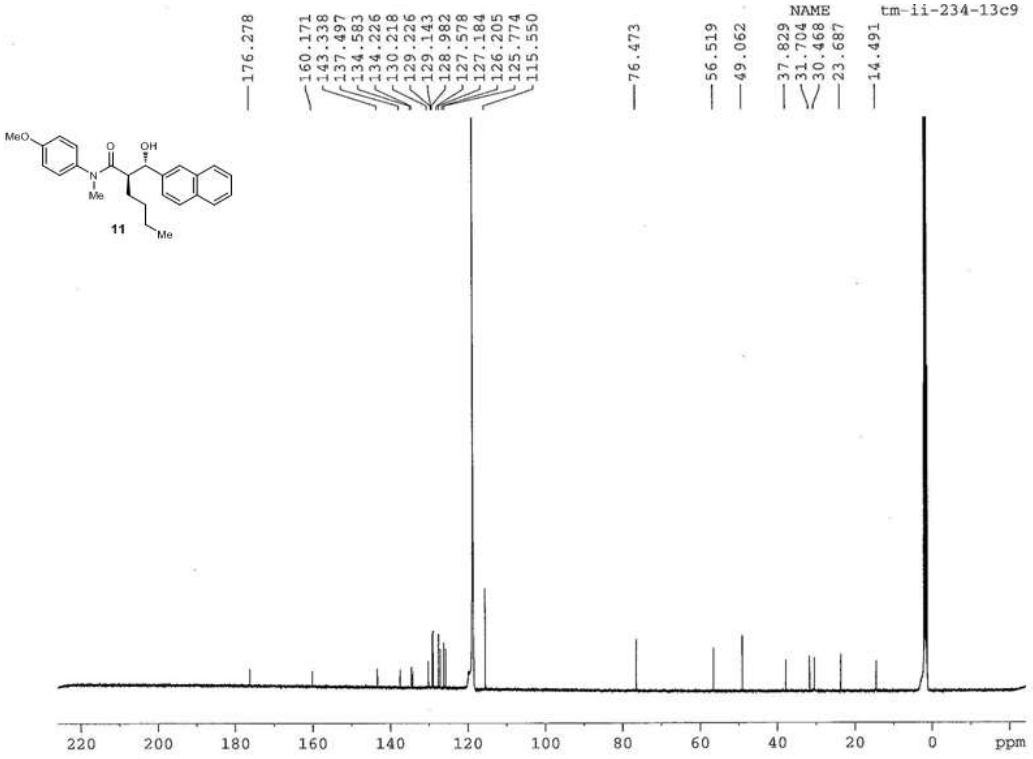

7.631 7.616 7.554 7.538 7.523 7.460 7.450 7.434 6.835 6.817 6.574 5.058 5.042 4.722 4.713 4.707 4.698 3.790 3.063 2.622 2.612 2.609 2.606 2.600 2.597 2.593 2.584 1.733 1.716 1.704 1.550 1.536 1.522 1.508 1.220 1.216 1.208 1.200 1.190 1.185 1.182 1.168 1.154 0.854 0.840 0.826
MeO
OH
CF3
N
Me
9
Me
NAME tm-i-229-330acn-
EXPNO 3
PROCNO 1
Date_ 20101110
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 H
FIDRES 0.166672 H
AQ 2.9999499 s
RG 80.6
DW 50.000 u
DE 7.50 u
TE 330.6 K
D1 5.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 u
PL1 0.00 d
SFO1 499.8729992 M
SI 32768
SF 499.8700188 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
0.93 0.94 1.84 1.95 1.64 0.84 1.00 3.00 3.00 1.03 1.04 1.11 4.07 3.07

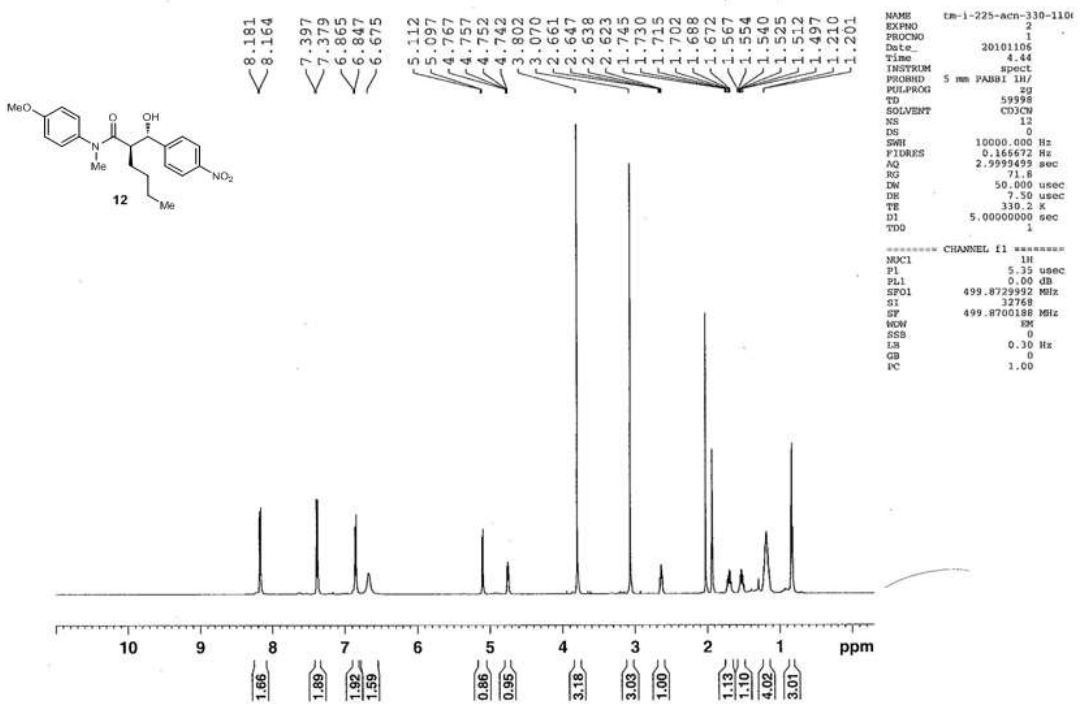

176.056 160.329 147.335 137.285 131.533 131.276 131.026 130.774 130.487 129.996 129.132 128.849 126.973 125.376 125.347 125.317 125.287 124.814 124.211 124.180 124.150 124.120 115.734 75.487 56.540 48.908 37.794 31.540 30.431 23.689 14.495
MeO
OH
CF3
N
Me
9
Me
220 200 180 160 140 120 100 80 60 40 20 0 ppm

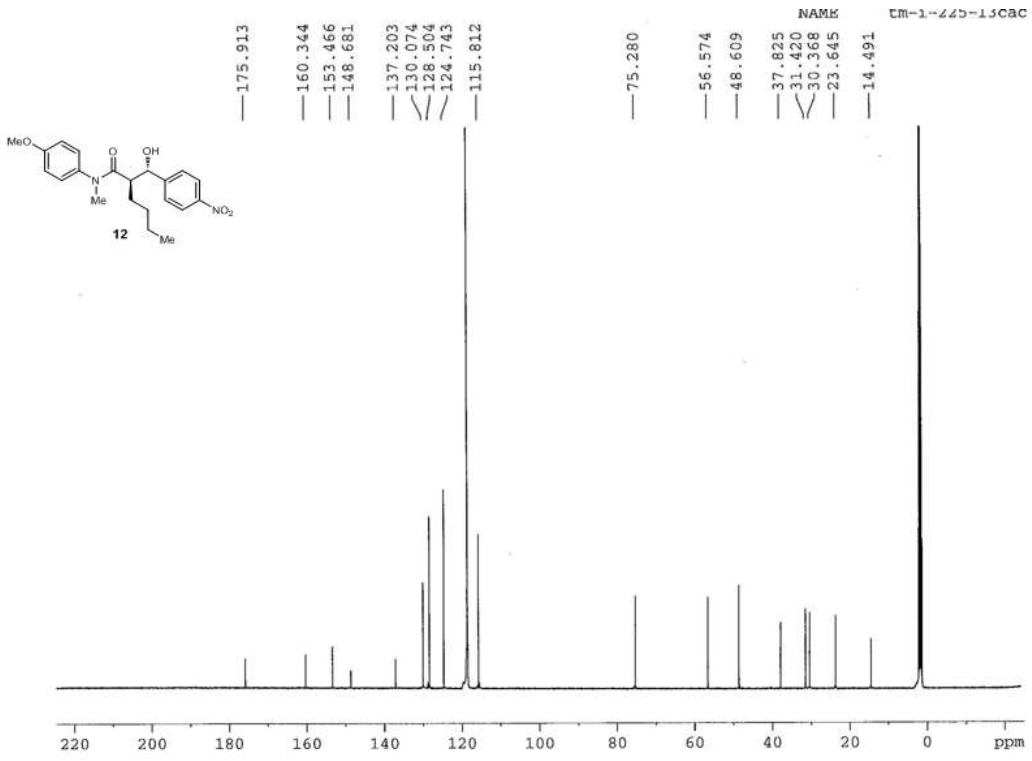

7.694
7.679
7.667
7.624
7.609
7.525
7.509
7.494
6.782
6.765
6.398
5.292
5.277
5.044
5.031
3.778
3.104
2.691
2.675
1.806
1.792
1.778
1.763
1.748
1.554
1.542
1.528
1.513
1.501
1.245
1.231
1.216
1.200
1.192
1.186
MeO
OH
CF3
N
Me
10
Me
NAME tm-i-230-345acn-
EXPNO 3
PROCNO 1
Date_ 20101110
Time 13.10
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000
FIDRES 0.166672
AQ 2.9999499
RG 71.8
DW 50.000
DE 7.50
TE 345.0
D1 5.00000000
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35
PL1 0.00
SFO1 499.8729992
SI 32768
SF 499.8700184
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.90
0.93
0.93
1.80
1.57
0.90
0.99
3.11
3.00
1.02
1.07
1.06
4.10
3.04

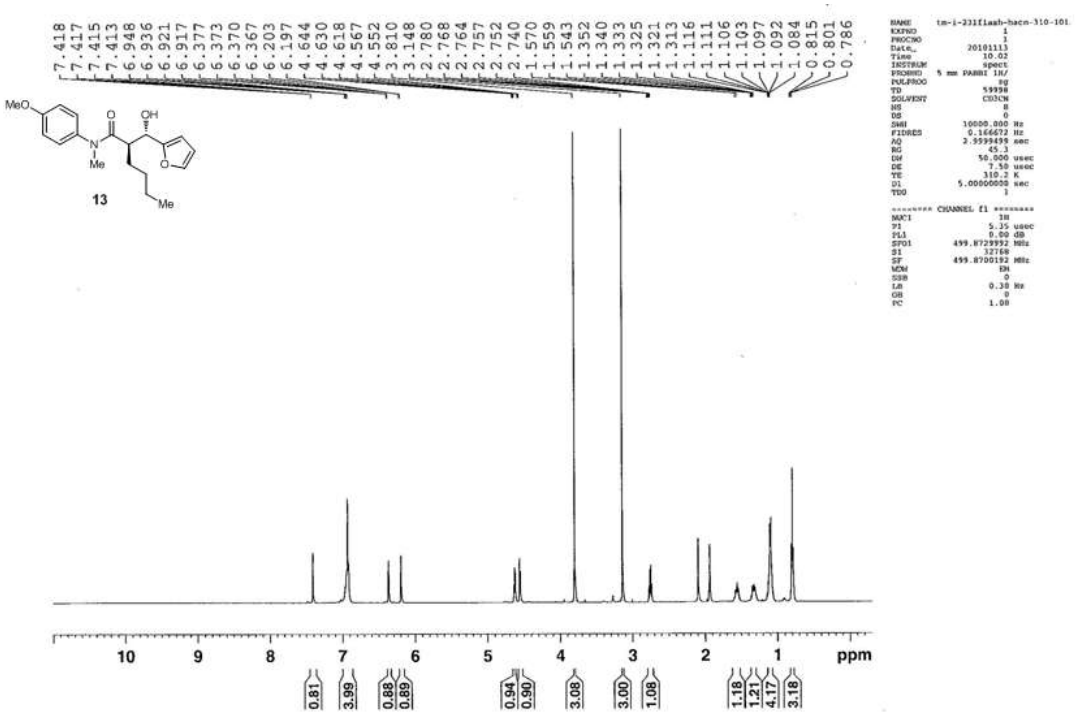

176.105
160.321
144.629
137.223
133.792
129.815
129.260
129.172
129.097
128.910
128.126
127.888
127.651
127.411
127.183
127.135
127.088
127.040
126.921
124.746
122.571
115.696
71.732
71.718
56.579
48.034
37.802
31.919
30.390
23.648
14.466
MeO
OH
CF3
N
Me
10
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

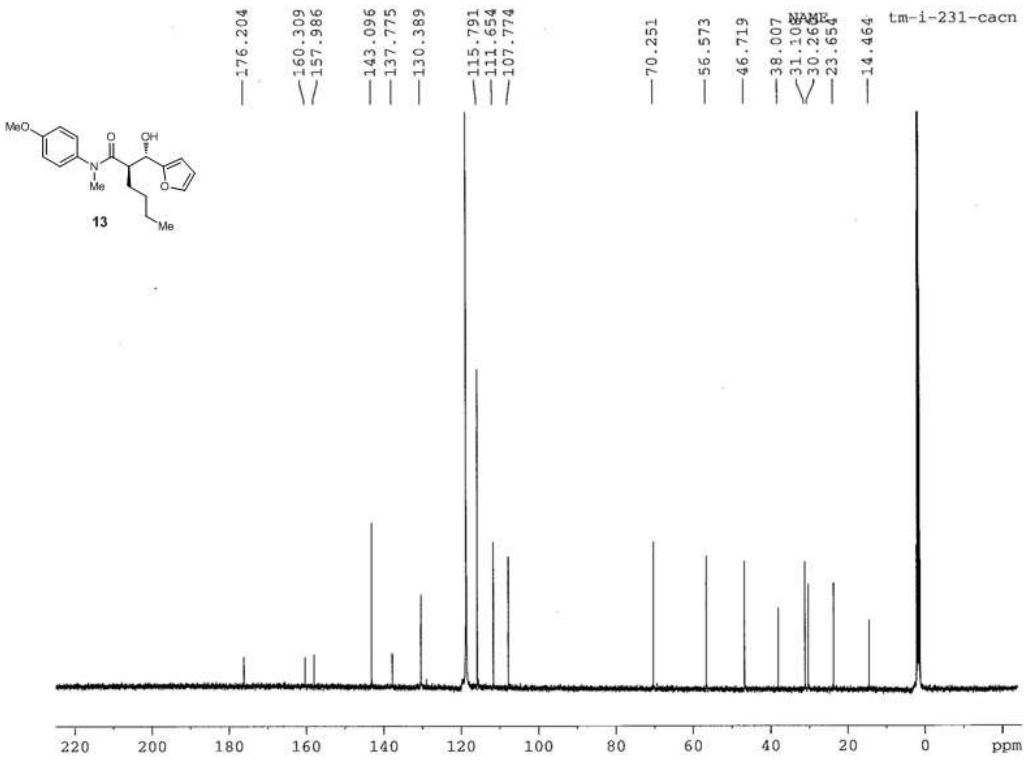

7.910
7.897
7.892
7.862
7.858
7.852
7.845
7.839
7.656
7.528
7.524
7.522
7.515
7.509
7.507
7.503
7.301
7.298
7.285
7.282
6.733
6.716
4.854
4.839
4.806
4.796
4.782
3.775
3.058
2.736
2.731
2.725
2.720
2.709
1.715
1.704
1.490
1.482
1.173
1.168
1.159
1.153
1.148
1.143
0.831
0.817
0.803
MeO
OH
N
Me
O
11
Me
NAME tm-i-234-010911-
EXPNO 1
PROCNO 1
Date_ 20110119
Time 4.16
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 12
DS 0
SWH 10000.000 H
FIDRES 0.166672 H
AQ 2.9999499 s
RG 228.1
DW 50.000 u
DE 7.50 u
TE 330.3 K
D1 5.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 u
PL1 0.00 d
SFO1 499.8729992 M
SI 32768
SF 499.8700189 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.05
1.83
0.95
1.83
0.92
1.76
1.60
0.88
0.96
3.02
3.00
1.01
1.10
1.12
4.14
2.95

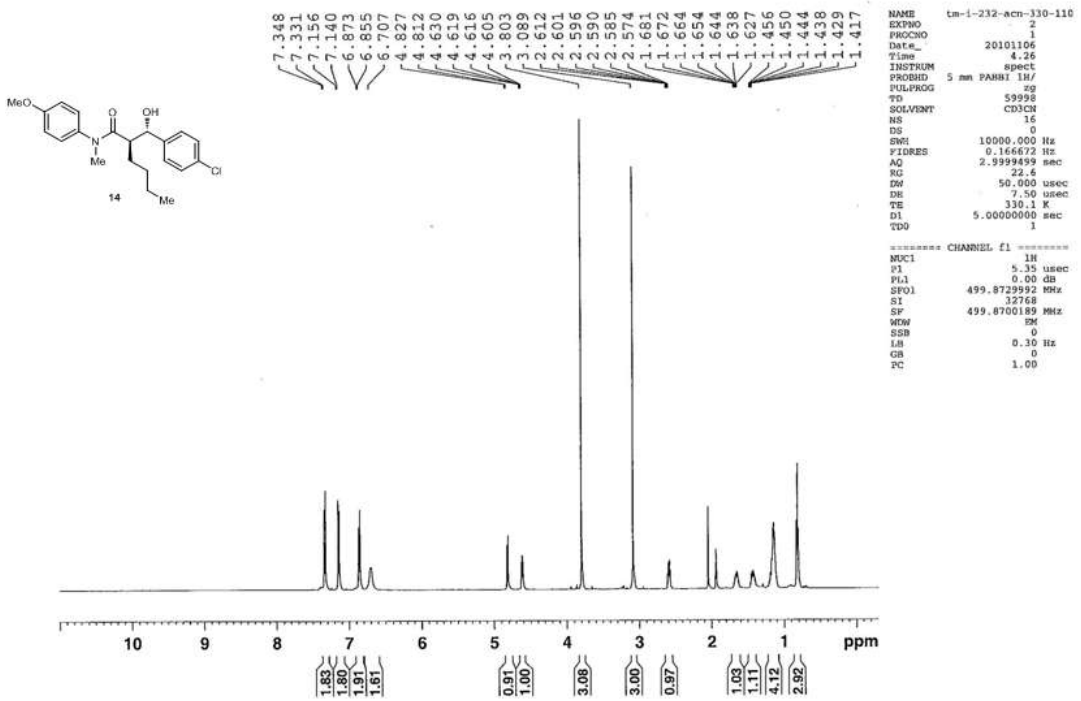

NAME tm-ii-234-13c9
176.278
160.171
143.338
137.497
134.583
134.226
130.218
129.226
129.143
128.982
127.578
127.184
126.205
125.774
115.550
76.473
56.519
49.062
37.829
31.704
30.468
23.687
14.491
MeO
OH
N
Me
O
11
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

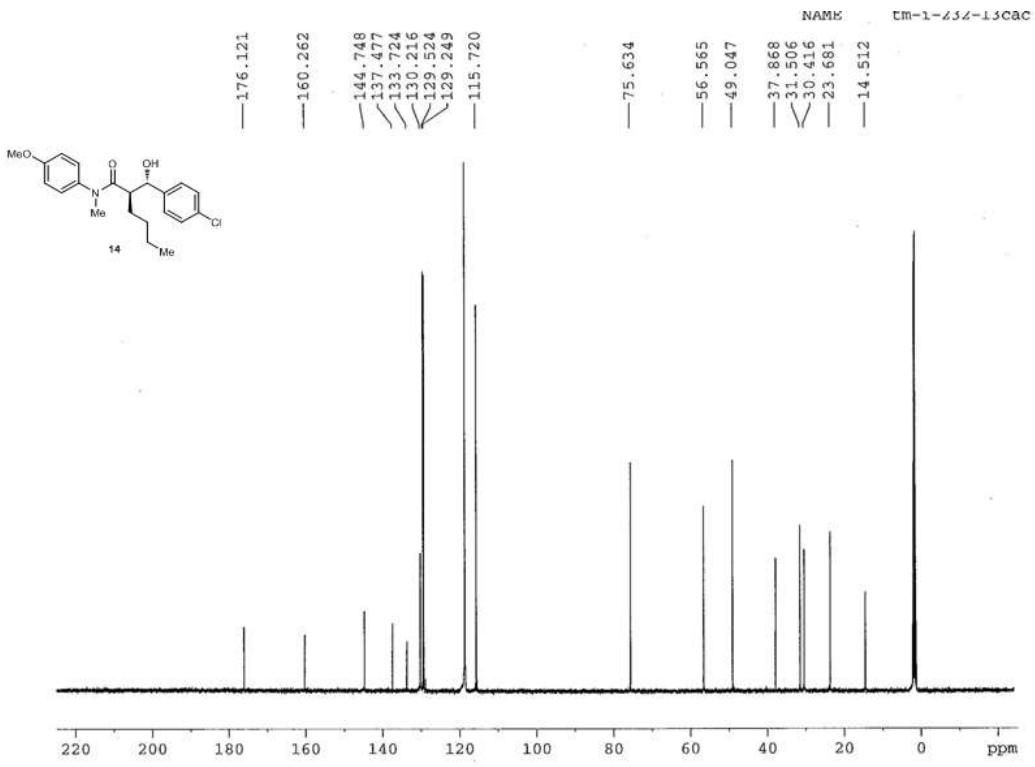

8.181
8.164
7.397
7.379
6.865
6.847
6.675
5.112
5.097
4.767
4.757
4.752
4.742
3.802
3.070
2.661
2.647
2.638
2.623
1.745
1.730
1.715
1.702
1.688
1.672
1.567
1.554
1.540
1.525
1.512
1.497
1.210
1.201
MeO
OH
N
Me
O
NO2
12
Me
NAME tm-i-225-acn-330-110
EXPNO 2
PROCNO 1
Date_ 20101106
Time 4.44
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 12
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 71.6
DW 50.000 usec
DE 7.50 usec
TE 330.2 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.66
1.89
1.92
1.59
0.86
0.95
3.18
3.03
1.00
1.13
1.10
4.02
3.01

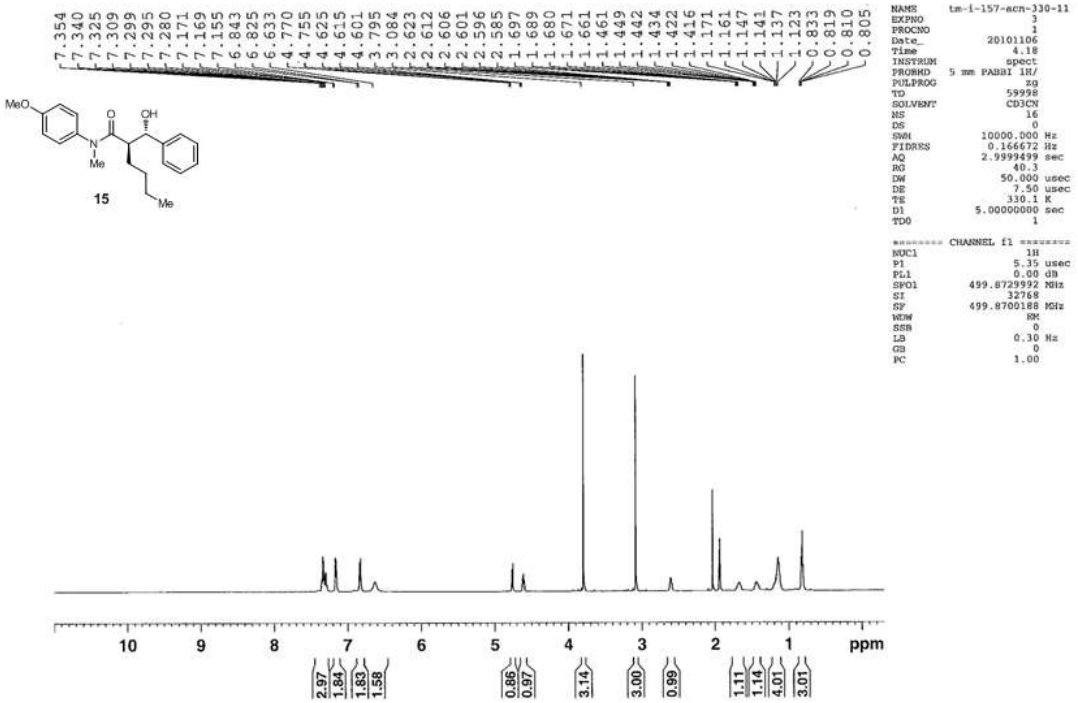

NAME tm-i-225-13cac
175.913
160.344
153.466
148.681
137.203
130.074
128.504
124.743
115.812
75.280
56.574
48.609
37.825
31.420
30.368
23.645
14.491
MeO
OH
N
Me
O
NO2
12
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

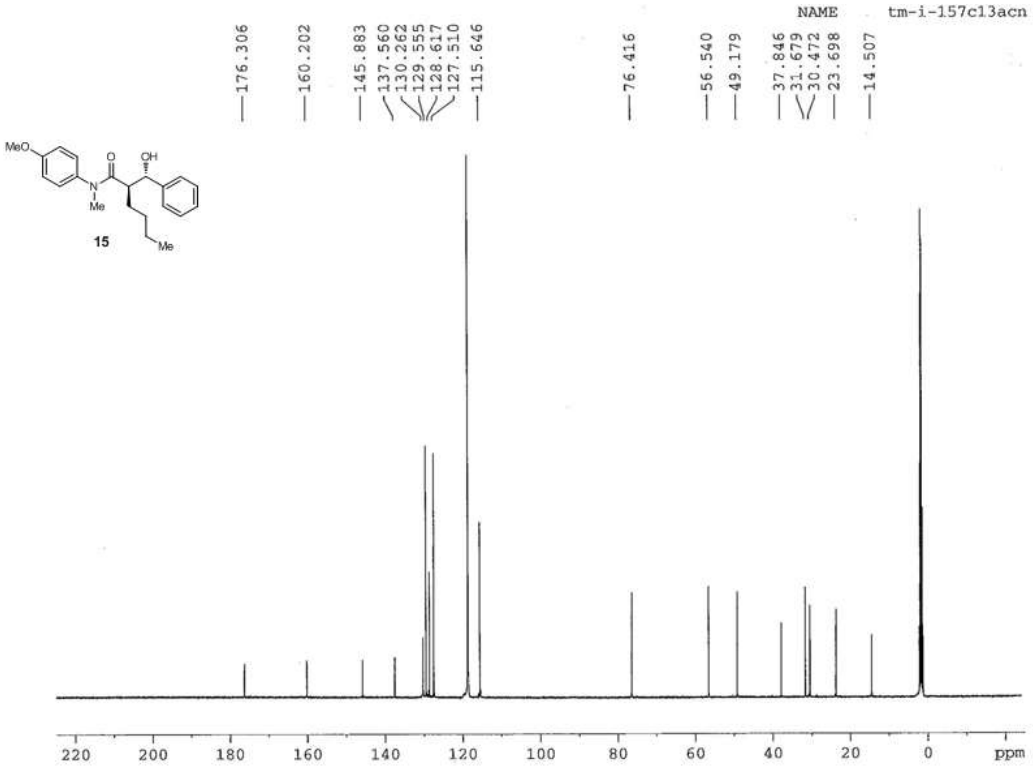

7.418
7.417
7.415
7.413
6.948
6.936
6.921
6.917
6.377
6.373
6.370
6.367
6.203
6.197
4.644
4.630
4.618
4.567
4.552
3.810
3.148
2.780
2.768
2.764
2.757
2.752
2.740
1.570
1.559
1.543
1.352
1.340
1.333
1.325
1.321
1.313
1.116
1.111
1.106
1.103
1.097
1.092
1.084
0.815
0.801
0.786
MeO
OH
N
Me
O
13
Me
NAME tm-i-231flash-hacn-310-101
EXPNO 1
PROCNO 1
Date_ 20101113
Time 10.02
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59958
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9599499 sec
RG 45.3
DW 50.000 usec
DE 7.50 usec
TE 310.2 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700192 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
0.81
3.99
0.88
0.89
0.94
0.90
3.08
3.00
1.08
1.18
1.21
4.17
3.18

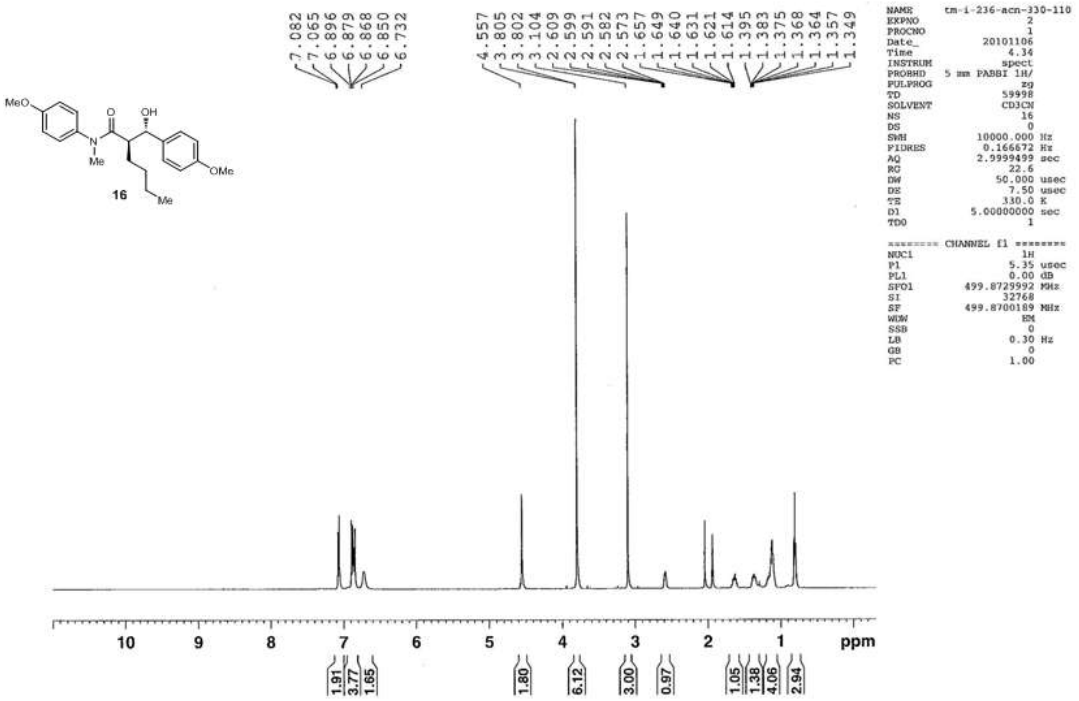

176.204
160.309
157.986
143.096
137.775
130.389
115.791
111.654
107.774
70.251
56.573
46.719
38.007
31.108
30.266
23.654
14.464
tm-i-231-cacn
MeO
OH
N
Me
O
13
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

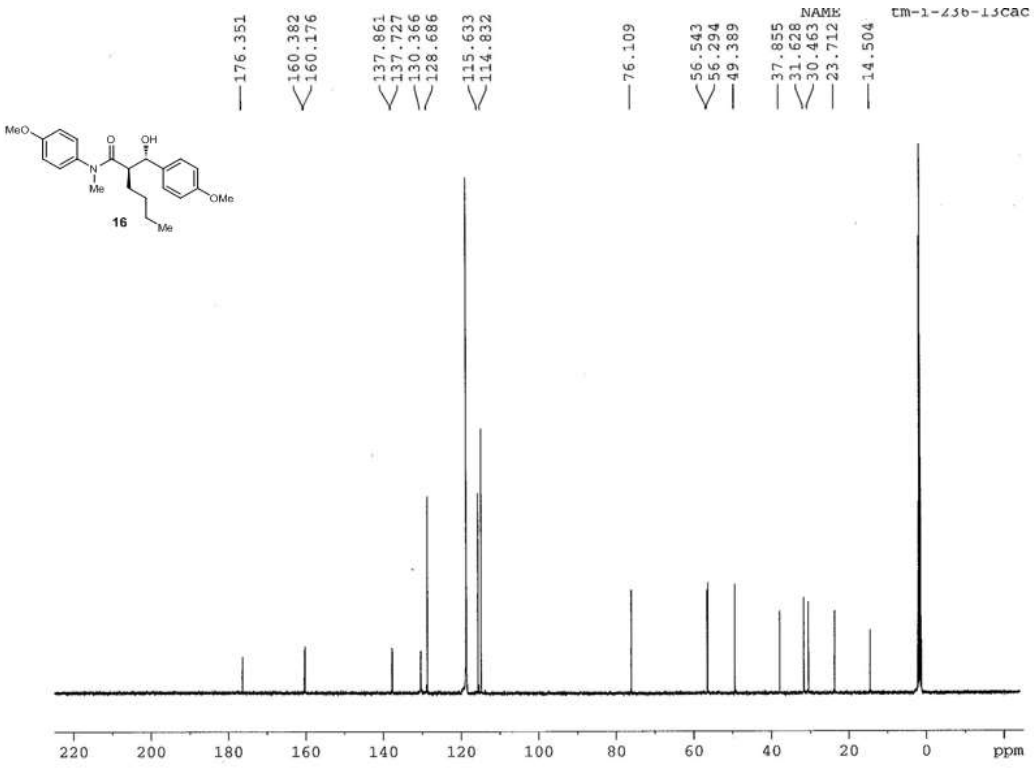

7.348
7.331
7.156
7.140
6.873
6.855
6.707
4.827
4.812
4.630
4.619
4.616
4.605
3.803
3.089
2.612
2.601
2.596
2.590
2.585
2.574
1.681
1.672
1.664
1.654
1.644
1.638
1.627
1.456
1.450
1.444
1.438
1.429
1.417
MeO
OH
N
Me
Cl
14
Me
NAME tm-1-232-acn-330-110
EXPNO 2
PROCNO 1
Date_ 20101106
Time 4.26
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 22.6
DW 50.000 usec
DE 7.50 usec
TE 330.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700189 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
1.83 1.80 1.91 1.61 0.91 1.00 3.08 3.00 0.97 1.03 1.11 4.12 2.92

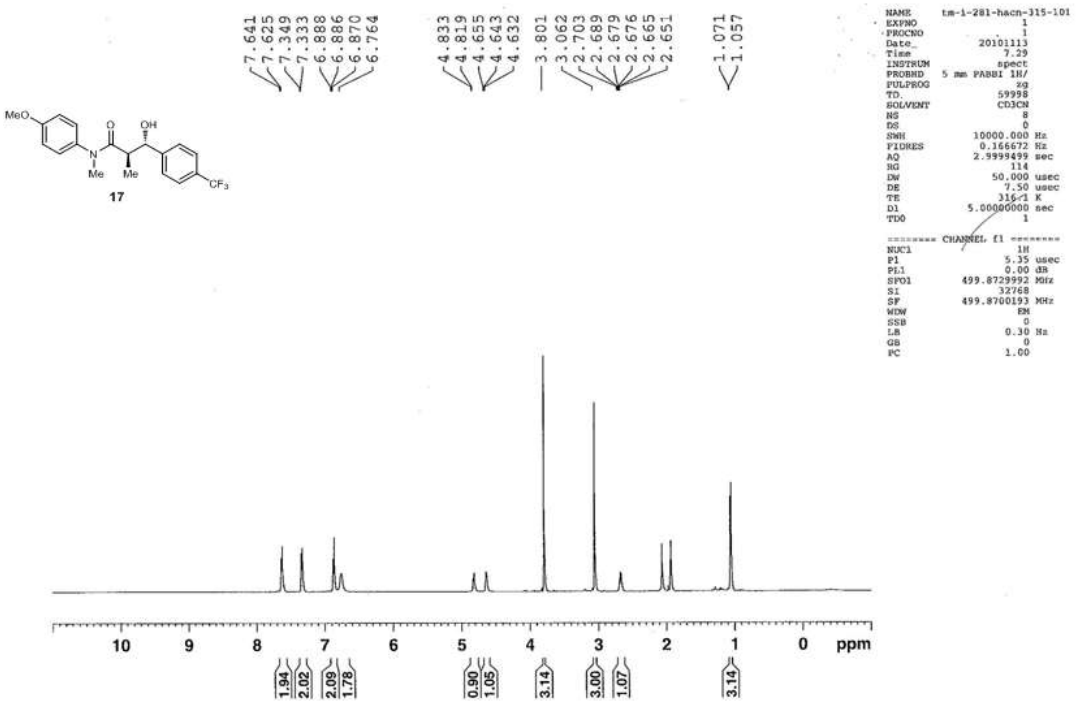

NAME tm-1-232-13Cac
176.121
160.262
144.748
137.477
133.724
130.216
129.524
129.249
115.720
75.634
56.565
49.047
37.868
31.506
30.416
23.681
14.512
MeO
OH
N
Me
Cl
14
Me
220 200 180 160 140 120 100 80 60 40 20 0 ppm

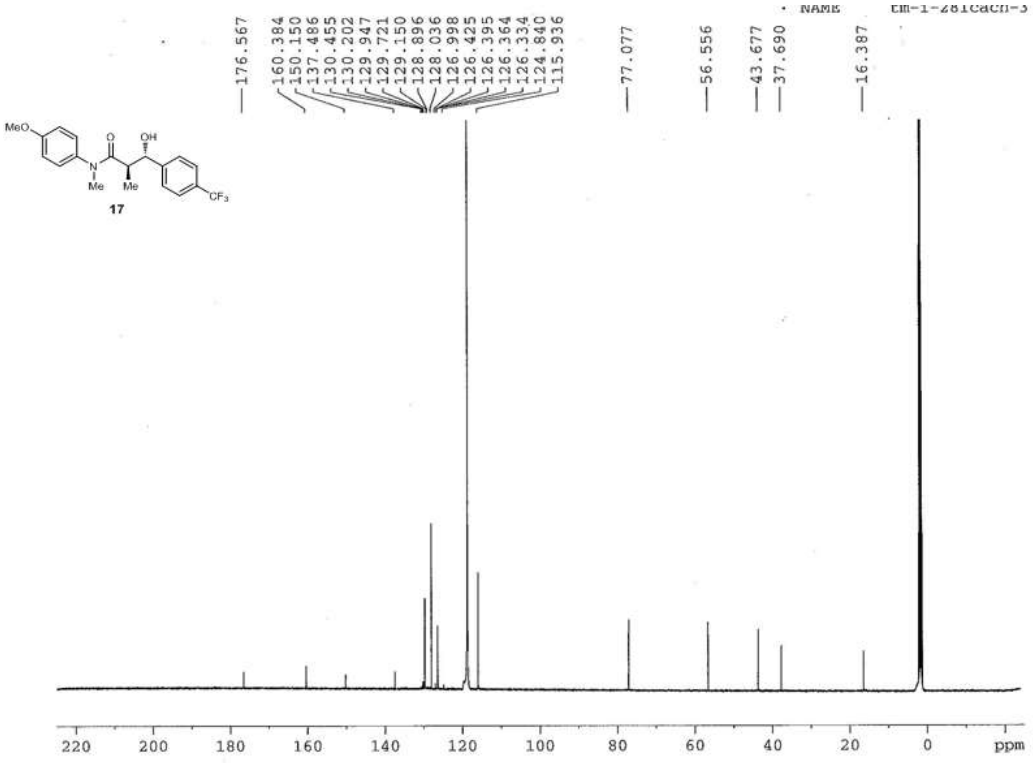

7.354 7.340 7.325 7.309 7.299 7.295 7.280 7.171 7.169 7.155 6.843 6.825 6.633 4.770 4.755 4.625 4.615 4.601 3.795 3.084 2.623 2.612 2.606 2.601 2.596 2.585 1.697 1.689 1.680 1.671 1.661 1.461 1.449 1.442 1.434 1.422 1.416 1.171 1.161 1.147 1.141 1.137 1.123 0.833 0.819 0.810 0.805
MeO
O
OH
N
Me
15
Me
NAME tm-i-157-acn-330-11
EXPNO 3
PROCNO 1
Date_ 20101106
Time 4.18
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 40.3
DW 50.000 usec
DE 7.50 usec
TE 330.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
2.97 1.84 1.83 1.58 0.86 0.97 3.14 3.00 0.99 1.11 1.14 4.01 3.01

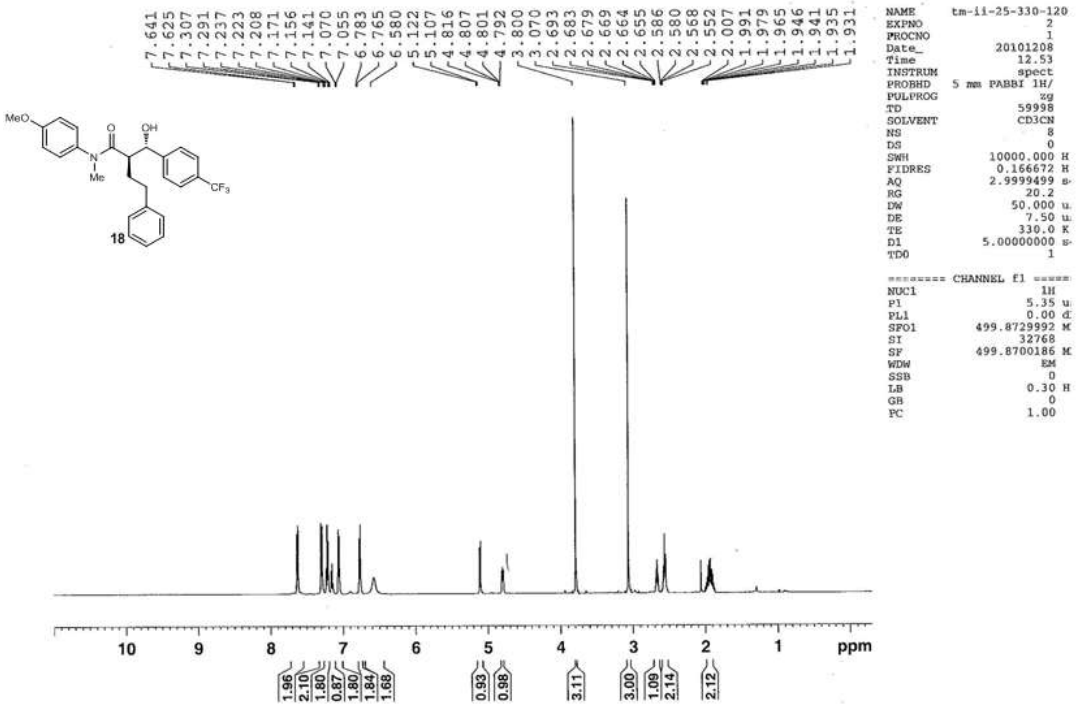

NAME tm-i-157c13acn
176.306 160.202 145.883 137.560 130.262 129.555 128.617 127.510 115.646 76.416 56.540 49.179 37.846 31.679 30.472 23.698 14.507
MeO
O
OH
N
Me
15
Me
220 200 180 160 140 120 100 80 60 40 20 0 ppm

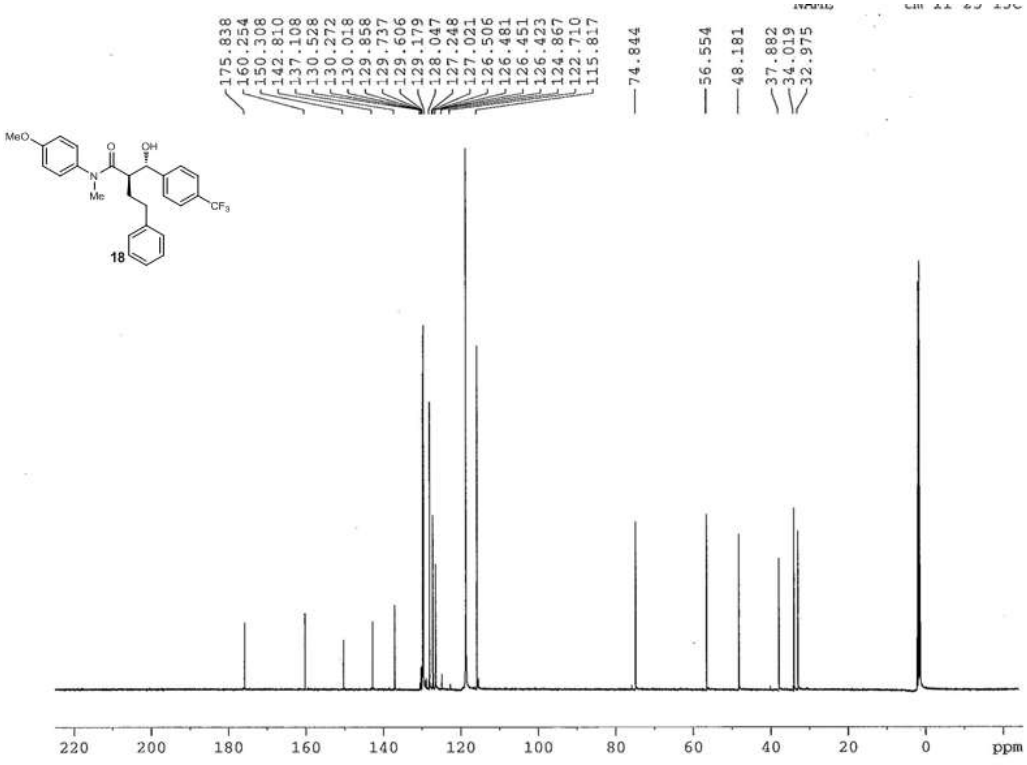

7.082
7.065
6.896
6.879
6.868
6.850
6.732
4.557
3.805
3.802
3.104
2.609
2.599
2.591
2.582
2.573
1.657
1.649
1.640
1.631
1.621
1.614
1.395
1.383
1.375
1.368
1.364
1.357
1.349
MeO
OH
N
Me
OMe
16
Me
NAME tm-1-236-acn-330-110
EXPNO 2
PROCNO 1
Date_ 20101106
Time_ 4.34
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 22.6
DW 50.000 usec
DE 7.50 usec
TE 330.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700189 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.91
3.77
1.65
1.80
6.12
3.00
0.97
1.05
1.38
4.06
2.94

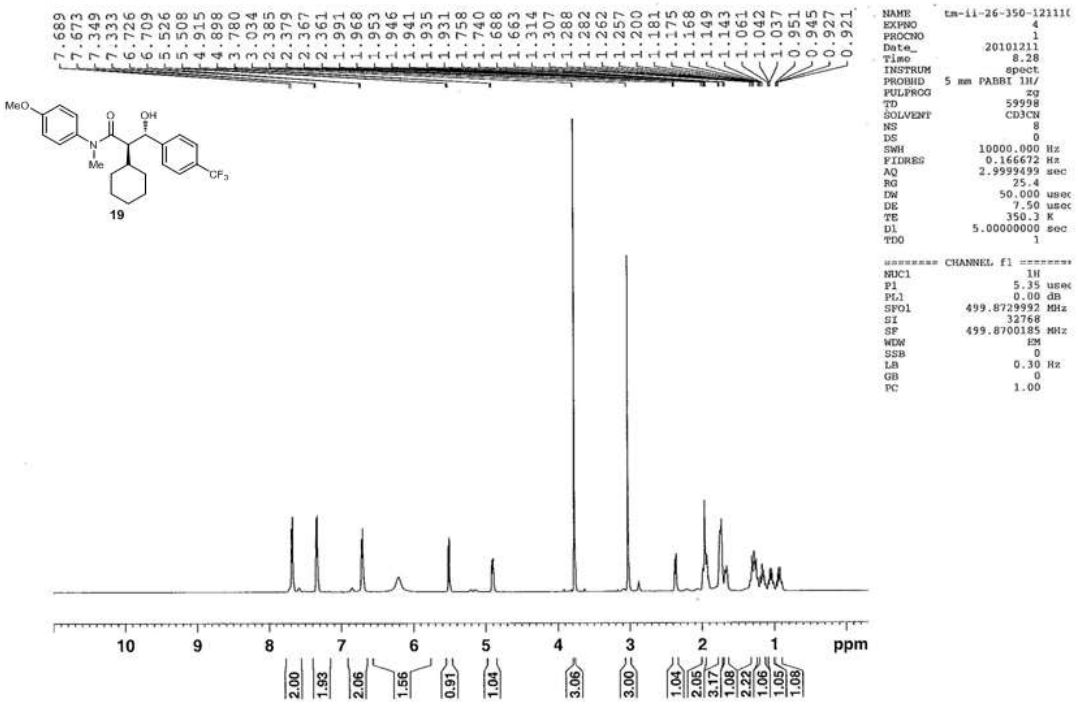

176.351
160.382
160.176
137.861
137.727
130.366
128.686
115.633
114.832
76.109
56.543
56.294
49.389
37.855
31.628
30.463
23.712
14.504
NAME tm-1-236-13cac
MeO
OH
N
Me
OMe
16
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

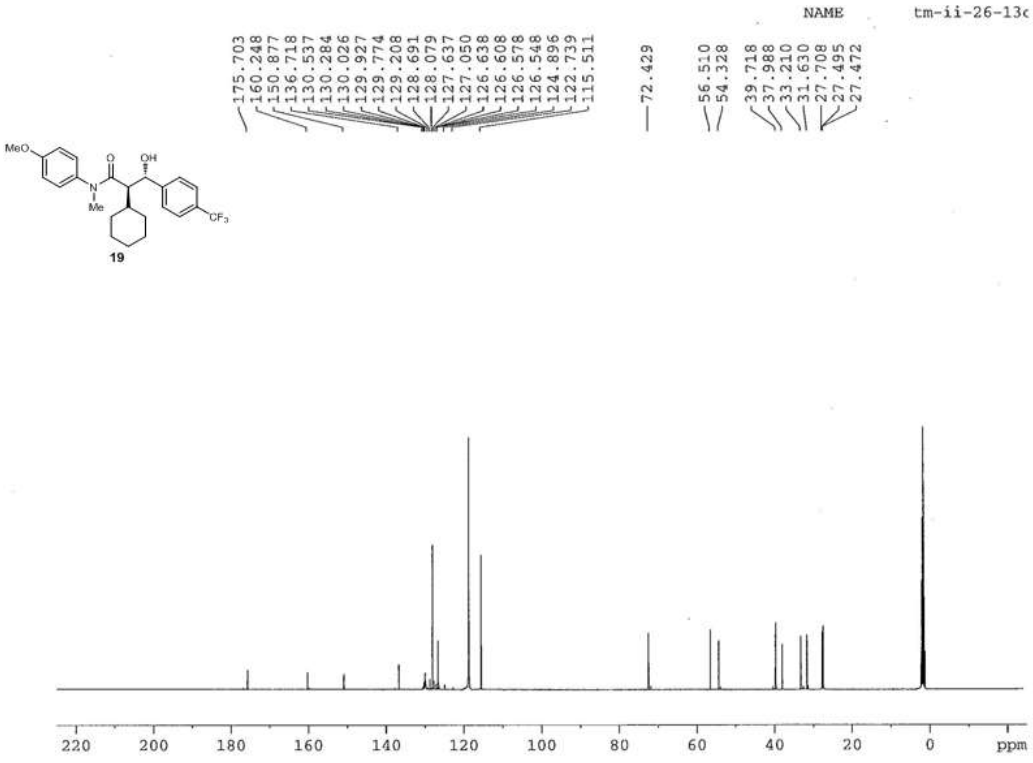

7.641
7.625
7.349
7.333
6.888
6.886
6.870
6.764
4.833
4.819
4.655
4.643
4.632
3.801
3.062
2.703
2.689
2.679
2.676
2.665
2.651
1.071
1.057
MeO
OH
N
Me
Me
CF3
17
NAME tm-1-281-hacn-315-101
EXPNO 1
PROCNO 1
Date_ 20101113
Time 7.29
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 114
DW 50.000 usec
DE 7.50 usec
TE 316.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.94
2.02
2.09
1.78
0.90
1.05
3.14
3.00
1.07
3.14

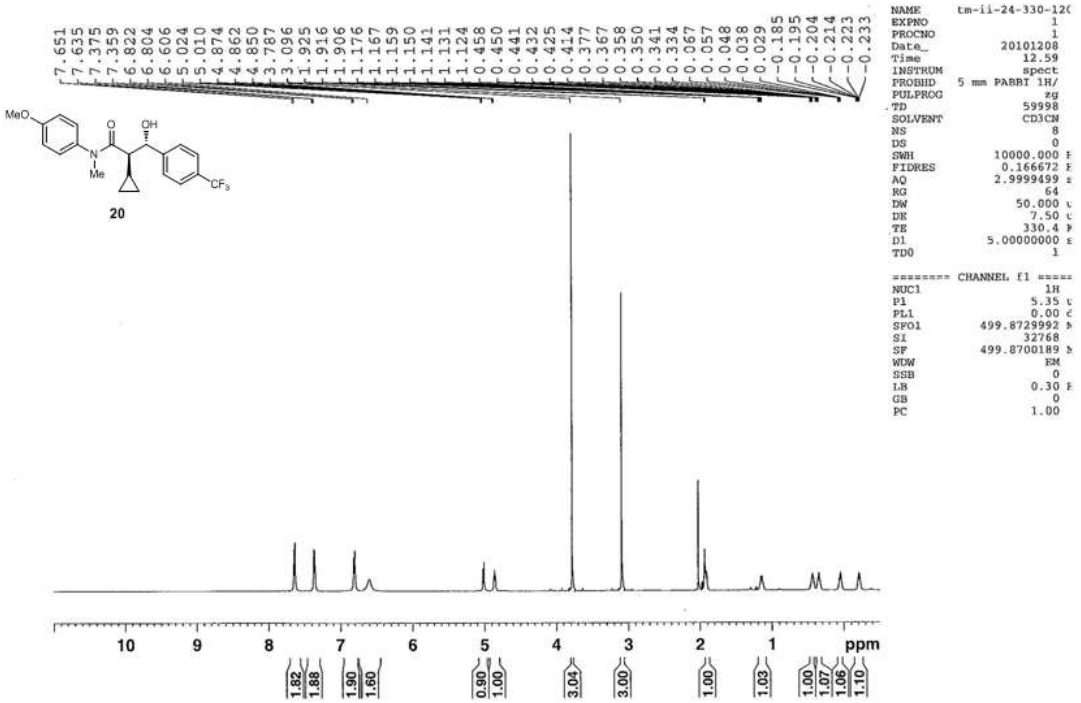

176.567
160.384
150.150
137.486
130.455
130.202
129.947
129.721
129.150
128.896
128.036
126.998
126.425
126.395
126.364
126.334
124.840
115.936
77.077
56.556
43.677
37.690
16.387
MeO
OH
N
Me
Me
CF3
17
220 200 180 160 140 120 100 80 60 40 20 0 ppm

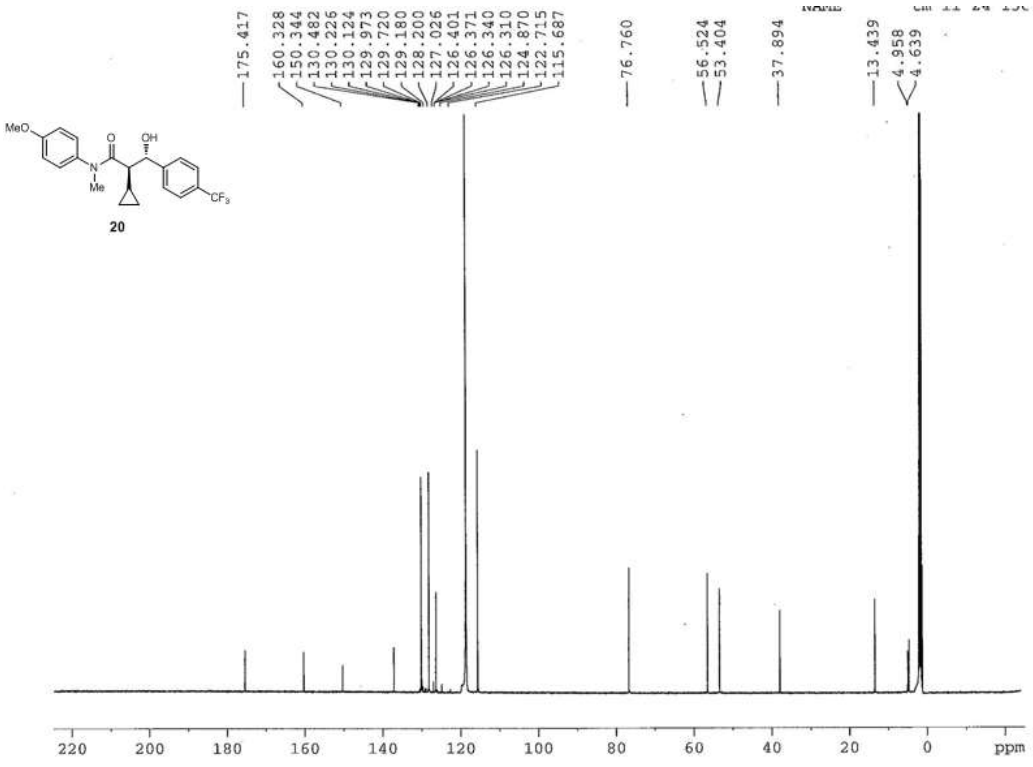

7.641
7.625
7.307
7.291
7.237
7.223
7.208
7.171
7.156
7.141
7.070
7.055
6.783
6.765
6.580
5.122
5.107
4.816
4.807
4.801
4.792
3.800
3.070
2.693
2.683
2.679
2.669
2.664
2.655
2.586
2.580
2.568
2.552
2.007
1.991
1.979
1.965
1.946
1.941
1.935
1.931
MeO
N
Me
O
OH
CF3
18
10
9
8
7
6
5
4
3
2
1
ppm
1.96
2.10
1.80
0.87
1.80
1.84
1.68
0.93
0.98
3.11
3.00
1.09
2.14
2.12
NAME tm-ii-25-330-120
EXPNO 2
PROCNO 1
Date_ 20101208
Time 12.53
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 H
FIDRES 0.166672 H
AQ 2.9999499 s
RG 20.2
DW 50.000 u
DE 7.50 u
TE 330.0 K
D1 5.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 u
PL1 0.00 d
SFO1 499.8729992 M
SI 32768
SF 499.8700186 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00

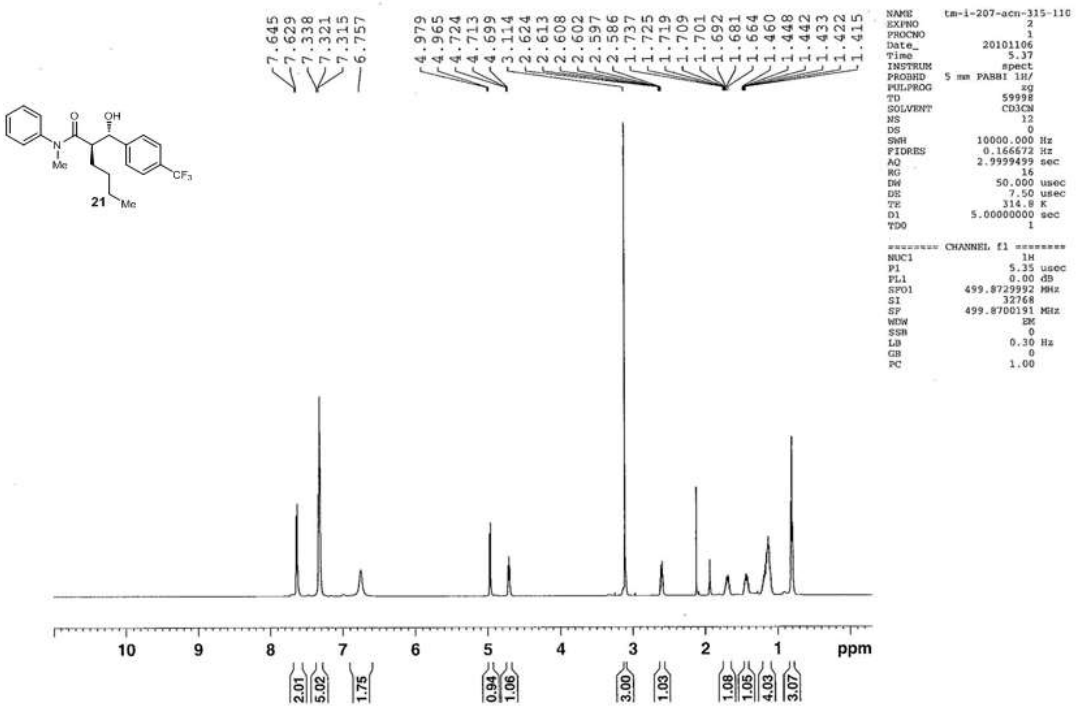

175.838
160.254
150.308
142.810
137.108
130.528
130.272
130.018
129.858
129.737
129.606
129.179
128.047
127.248
127.021
126.506
126.481
126.451
126.423
124.867
122.710
115.817
74.844
56.554
48.181
37.882
34.019
32.975
MeO
N
Me
O
OH
CF3
18
220
200
180
160
140
120
100
80
60
40
20
0
ppm

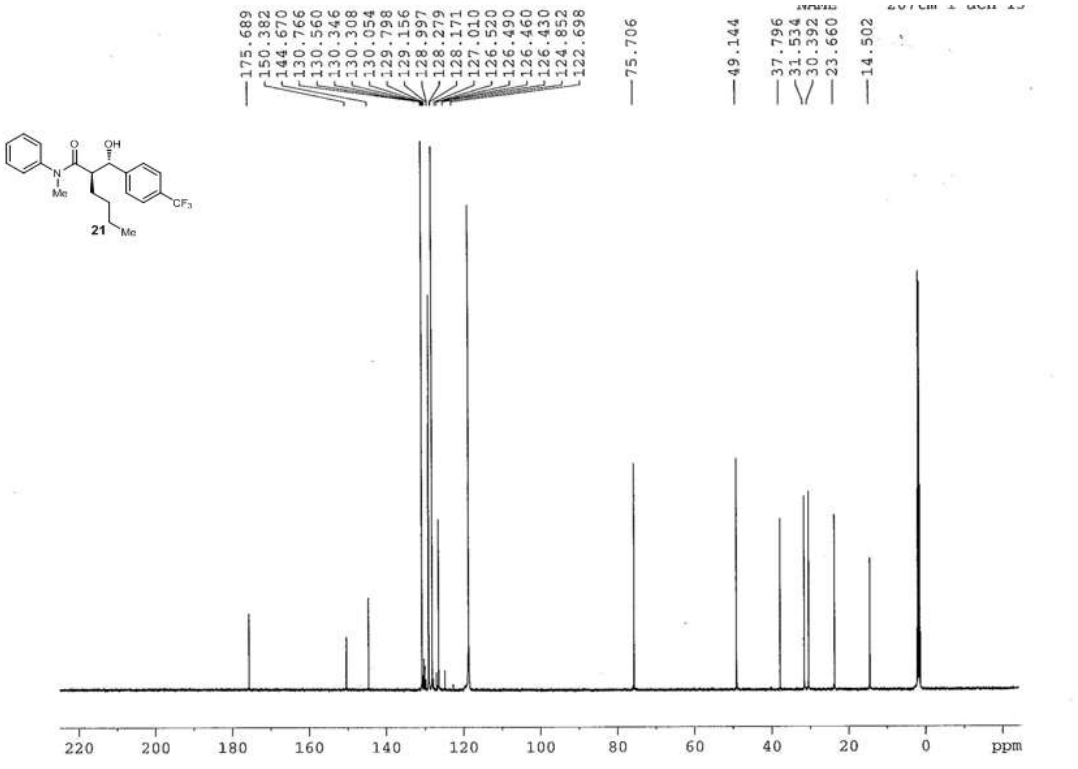

7.689
7.673
7.349
7.333
6.726
6.709
5.526
5.508
4.915
4.898
3.780
3.034
2.385
2.379
2.367
2.361
1.991
1.968
1.953
1.946
1.941
1.935
1.931
1.758
1.740
1.688
1.663
1.314
1.307
1.288
1.282
1.262
1.257
1.200
1.181
1.175
1.168
1.149
1.143
1.061
1.042
1.037
0.951
0.945
0.927
0.921
MeO
N
Me
O
OH
CF3
19
NAME tm-ii-26-350-12111(
EXPNO 4
PROCNO 1
Date_ 20101211
Time 8.28
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 25.4
DW 50.000 usec
DE 7.50 usec
TE 350.3 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700185 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
2.00 1.93 2.06 1.56 0.91 1.04 3.06 3.00 1.04 2.05 3.17 1.08 2.22 1.06 1.05 1.08

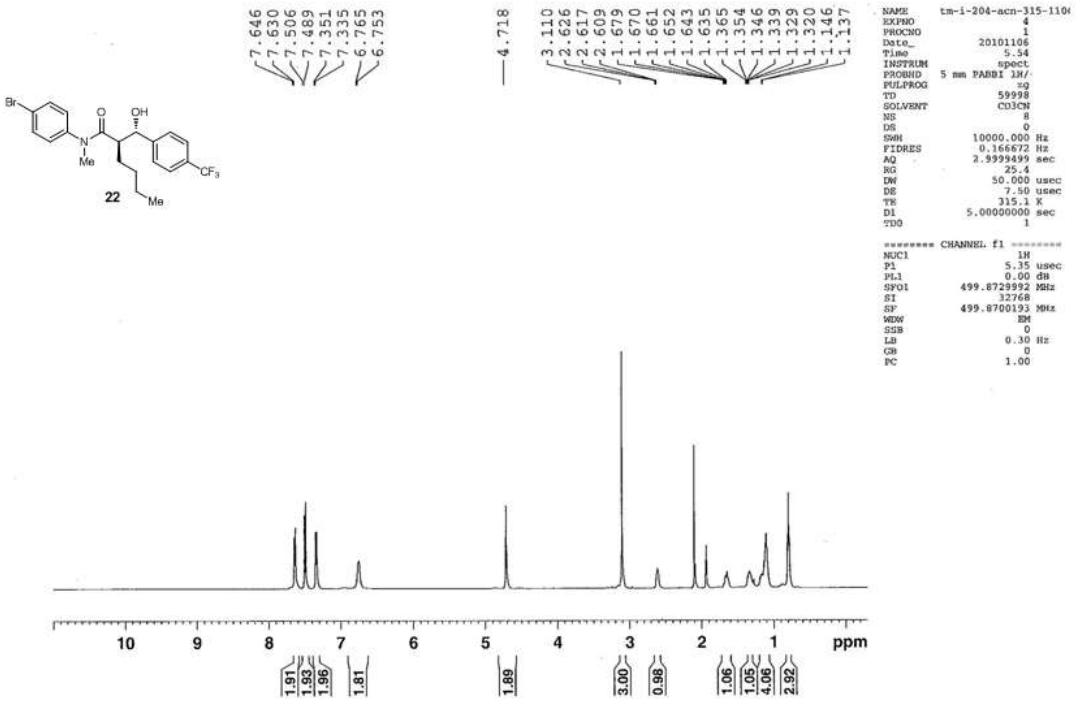

NAME tm-ii-26-13c
175.703
160.248
150.877
136.718
130.537
130.284
130.026
129.927
129.774
129.208
128.691
128.079
127.637
127.050
126.638
126.608
126.578
126.548
124.896
122.739
115.511
72.429
56.510
54.328
39.718
37.988
33.210
31.630
27.708
27.495
27.472
MeO
N
Me
O
OH
CF3
19
220 200 180 160 140 120 100 80 60 40 20 0 ppm

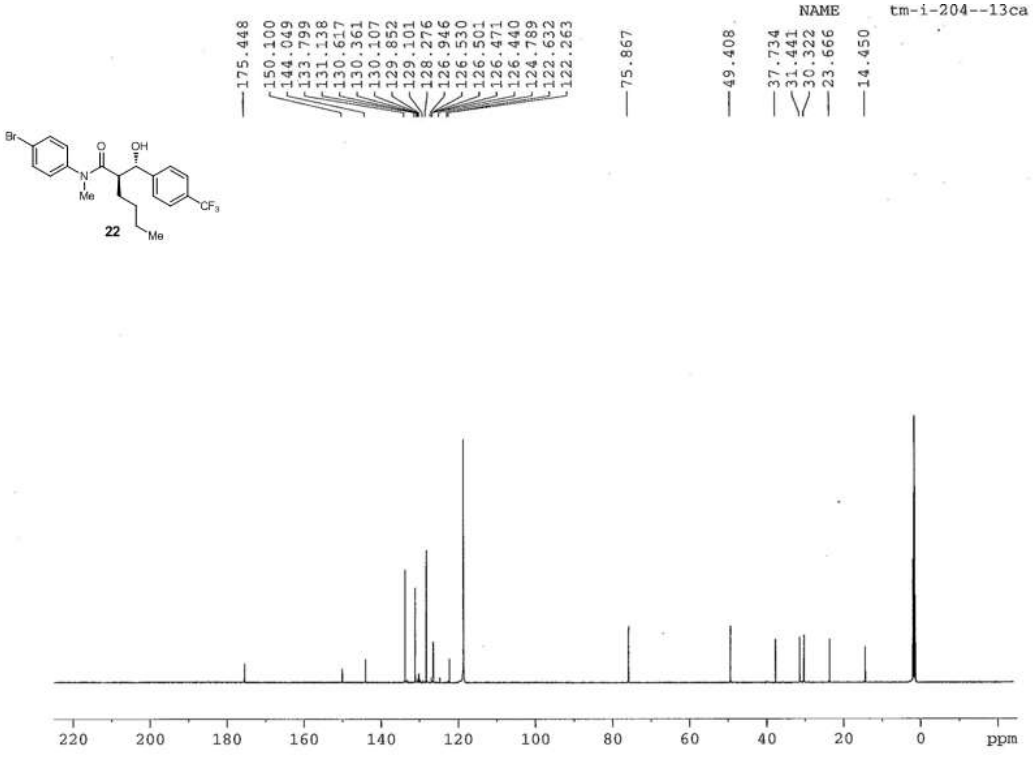

7.651
7.635
7.375
7.359
6.822
6.804
6.606
5.024
5.010
4.874
4.862
4.850
3.787
3.096
1.925
1.916
1.906
1.176
1.167
1.159
1.150
1.141
1.131
1.124
0.458
0.450
0.441
0.432
0.425
0.414
0.377
0.367
0.358
0.350
0.341
0.334
0.067
0.057
0.048
0.038
0.029
-0.185
-0.195
-0.204
-0.214
-0.223
-0.233
MeO
N
Me
O
OH
CF3
20
NAME tm-ii-24-330-12(
EXPNO 1
PROCNO 1
Date_ 20101208
Time 12.59
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000
FIDRES 0.166672
AQ 2.9999499
RG 64
DW 50.000
DE 7.50
TE 330.4
D1 5.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 5.35
PL1 0.00
SFO1 499.8729992
SI 32768
SF 499.8700189
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.82
1.88
1.90
1.60
0.90
1.00
3.04
3.00
1.00
1.03
1.00
1.07
1.06
1.10

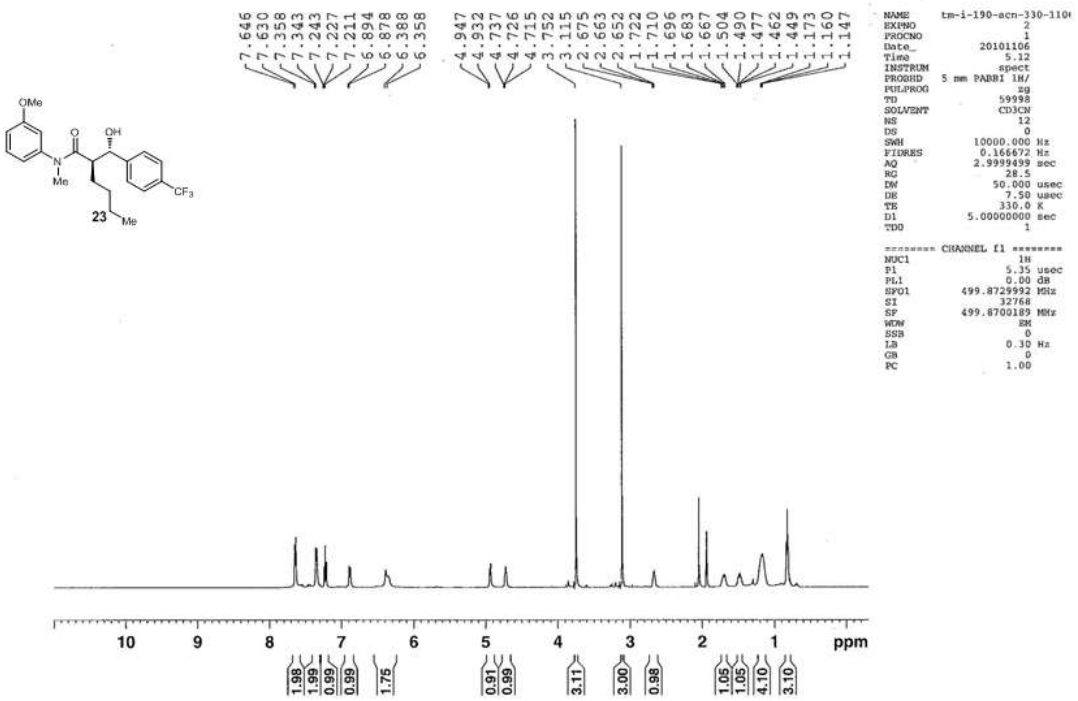

175.417
160.328
150.344
130.482
130.226
130.124
129.973
129.720
129.180
128.200
127.026
126.401
126.371
126.340
126.310
124.870
122.715
115.687
76.760
56.524
53.404
37.894
13.439
4.958
4.639
MeO
N
Me
O
OH
CF3
20
220
200
180
160
140
120
100
80
60
40
20
0
ppm

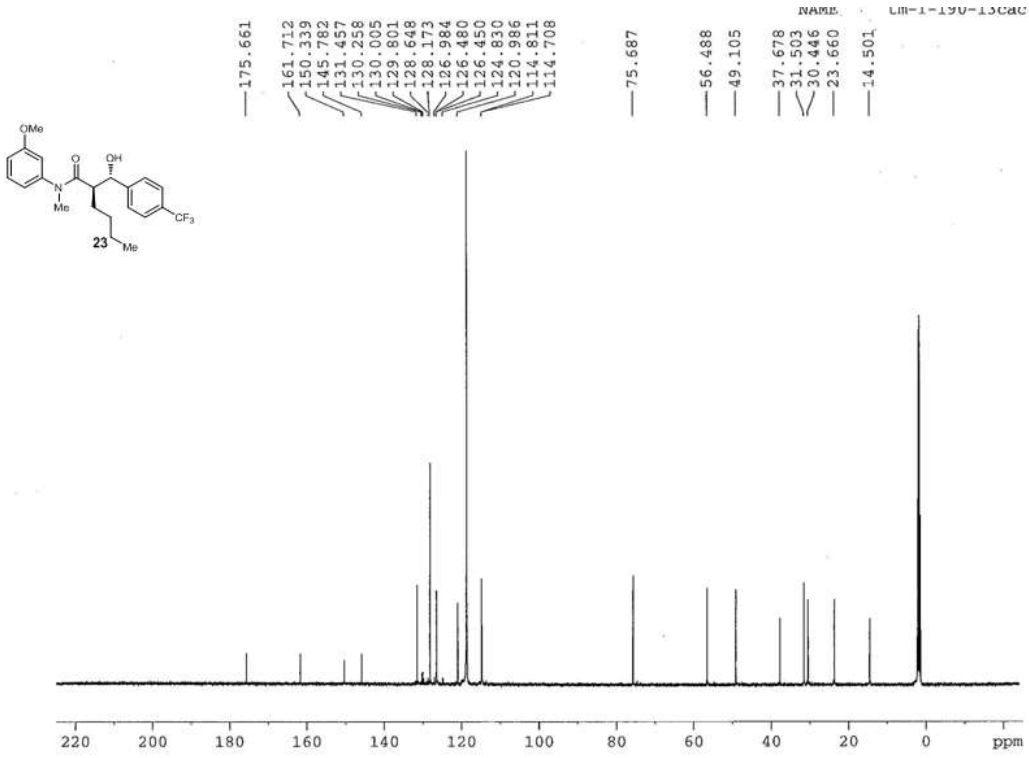

7.645
7.629
7.338
7.321
7.315
6.757
4.979
4.965
4.724
4.713
4.699
3.114
2.624
2.613
2.608
2.602
2.597
2.586
1.737
1.725
1.719
1.709
1.701
1.692
1.681
1.664
1.460
1.448
1.442
1.433
1.422
1.415
NAME tm-i-207-acn-315-110
EXPNO 2
PROCNO 1
Date_ 20101106
Time 5.37
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 12
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 16
DW 50.000 usec
DE 7.50 usec
TE 314.8 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700191 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
N
O
Me
21
Me
CF3
10
9
8
7
6
5
4
3
2
1
ppm
2.01
5.02
1.75
0.94
1.06
3.00
1.03
1.08
1.05
4.03
3.07

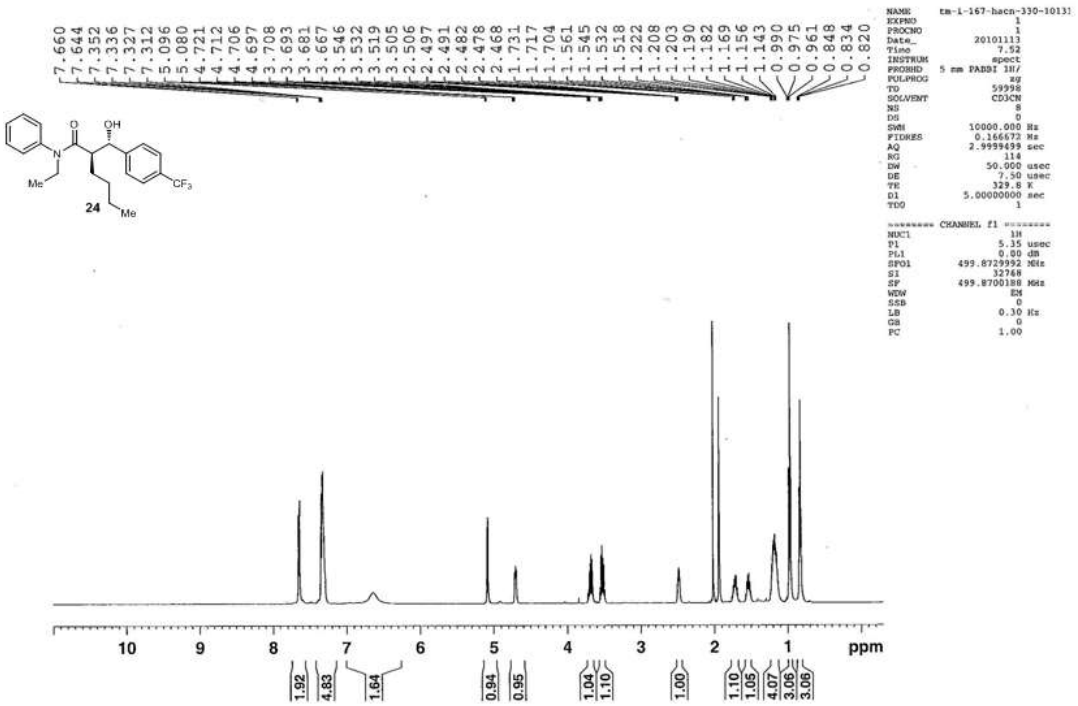

175.689
150.382
144.670
130.766
130.560
130.346
130.308
130.054
129.798
129.156
128.997
128.279
128.171
127.010
126.520
126.490
126.460
126.430
124.852
122.698
75.706
49.144
37.796
31.534
30.392
23.660
14.502
OH
N
O
Me
21
Me
CF3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

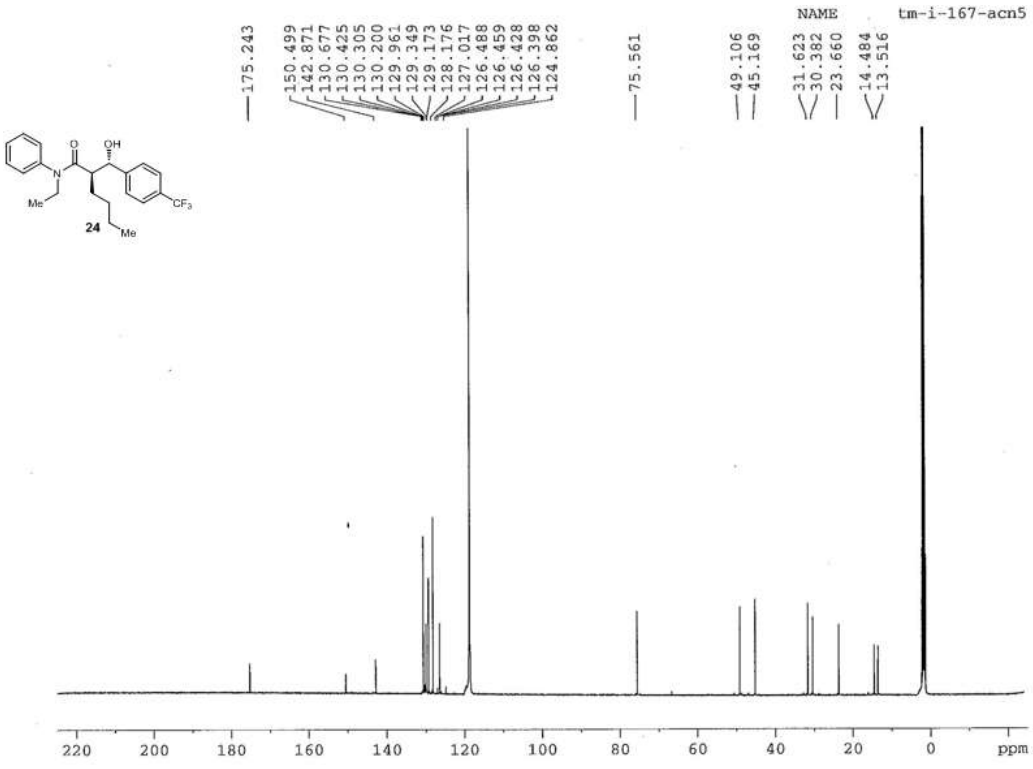

7.646
7.630
7.506
7.489
7.351
7.335
6.765
6.753
4.718
3.110
2.626
2.617
2.609
1.679
1.670
1.661
1.652
1.643
1.635
1.365
1.354
1.346
1.339
1.329
1.320
1.146
1.137
NAME tm-i-204-acn-315-110
EXPNO 4
PROCNO 1
Date_ 20101106
Time 5.54
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 25.4
DW 50.000 usec
DE 7.50 usec
TE 315.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
OH
N
Me
CF3
22
Me
10
9
8
7
6
5
4
3
2
1
ppm
1.91
1.93
1.96
1.81
1.89
3.00
0.98
1.06
1.05
4.06
2.92

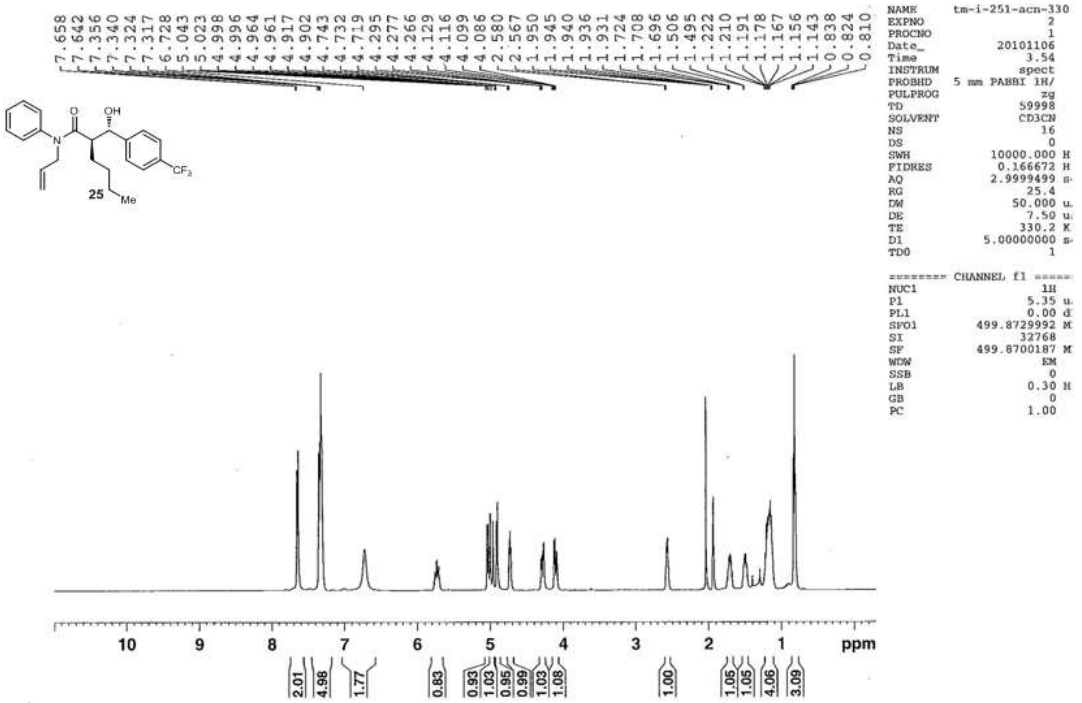

NAME tm-i-204--13ca
175.448
150.100
144.049
133.799
131.138
130.617
130.361
130.107
129.852
129.101
128.276
126.946
126.530
126.501
126.471
126.440
124.789
122.632
122.263
75.867
49.408
37.734
31.441
30.322
23.666
14.450
Br
O
OH
N
Me
CF3
22
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

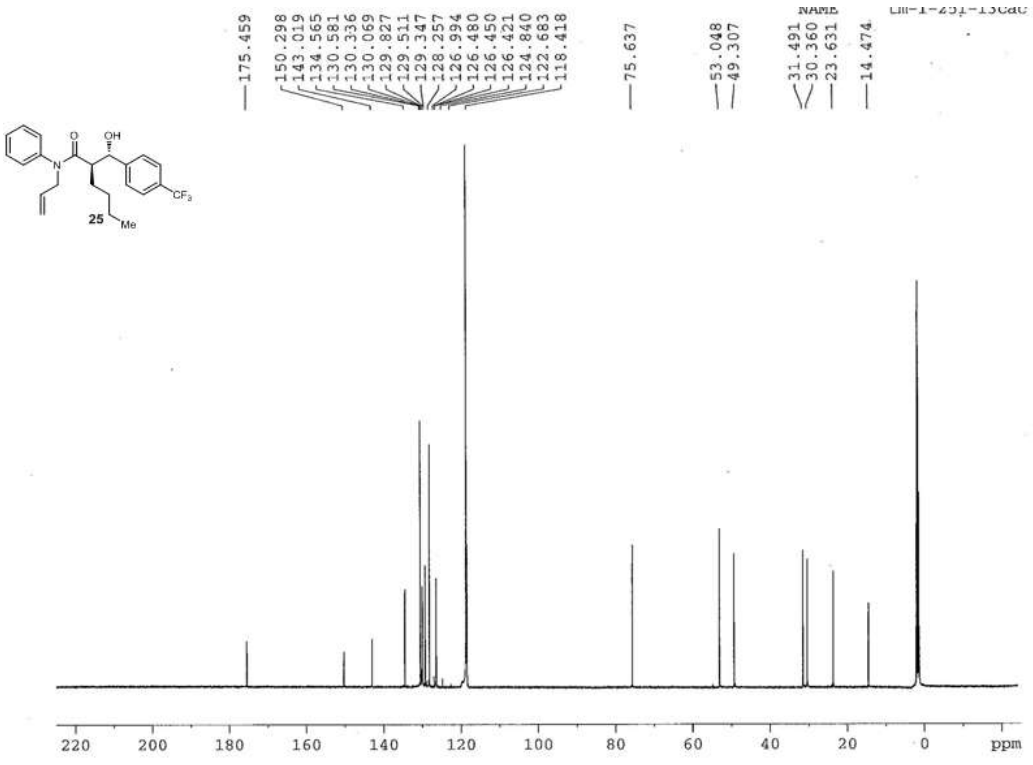

7.646
7.630
7.358
7.343
7.243
7.227
7.211
6.894
6.878
6.388
6.358
4.947
4.932
4.737
4.726
4.715
3.752
3.115
2.675
2.663
2.652
2.652
1.722
1.710
1.696
1.683
1.667
1.504
1.490
1.477
1.462
1.449
1.173
1.160
1.147
OMe
OH
N
Me
O
CF3
23
Me
NAME tm-i-190-acn-330-110
EXPNO 2
PROCNO 1
Date_ 20101106
Time 5.12
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 12
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 28.5
DW 50.000 usec
DE 7.50 usec
TE 330.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700189 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.98
1.99
0.99
0.99
1.75
0.91
0.99
3.11
3.00
0.98
1.05
1.05
4.10
3.10

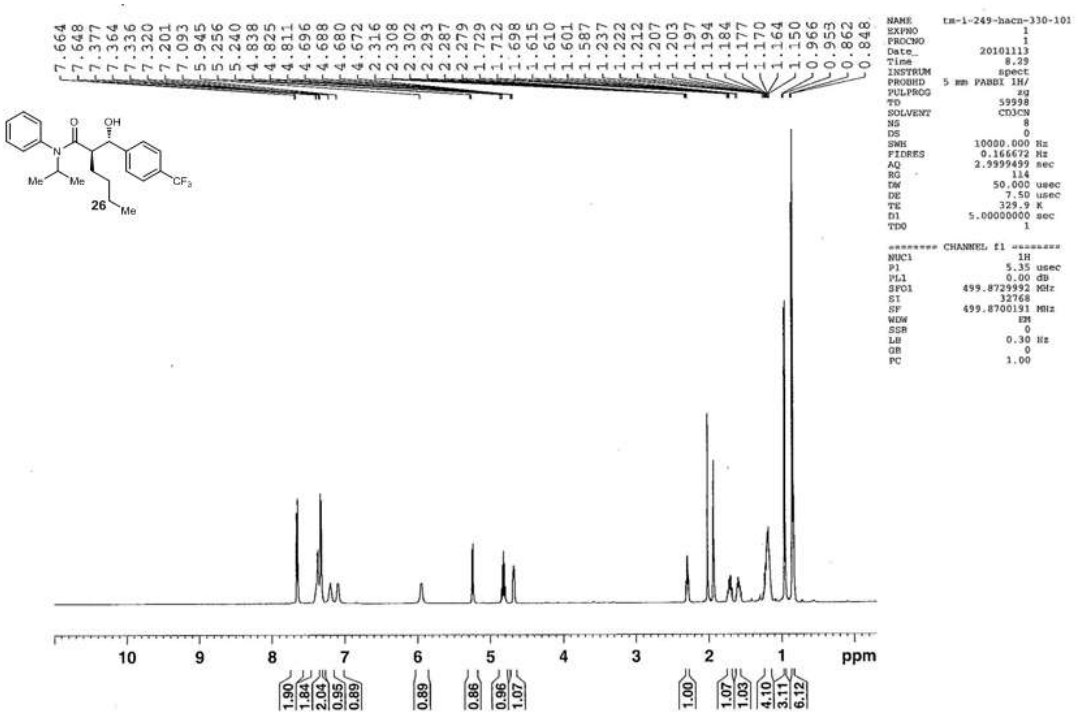

NAME tm-i-190-13Cac
175.661
161.712
150.339
145.782
131.457
130.258
130.005
129.801
128.648
128.173
126.984
126.480
126.450
124.830
120.986
114.811
114.708
75.687
56.488
49.105
37.678
31.503
30.446
23.660
14.501
OMe
OH
N
Me
O
CF3
23
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

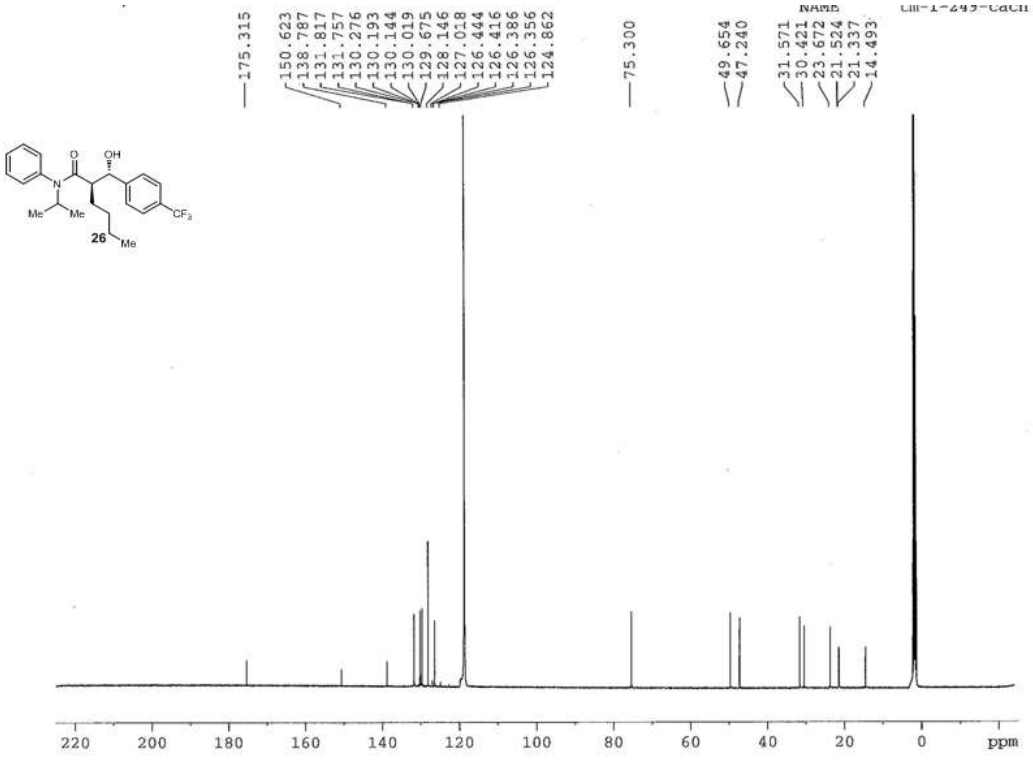

7.660
7.644
7.352
7.336
7.327
7.312
5.096
5.080
4.721
4.712
4.706
4.697
3.708
3.693
3.681
3.667
3.546
3.532
3.519
3.505
2.506
2.497
2.491
2.482
2.478
2.468
1.731
1.717
1.704
1.561
1.545
1.532
1.518
1.222
1.208
1.203
1.190
1.182
1.169
1.156
1.143
0.990
0.975
0.961
0.848
0.834
0.820
OH
N
O
Me
CF3
24
Me
NAME tm-i-167-hacn-330-10131
EXPNO 1
PROCNO 1
Date_ 20101113
Time 7.52
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 114
DW 50.000 usec
DE 7.50 usec
TE 329.8 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
1.92 4.83 1.64 0.94 0.95 1.04 1.10 1.00 1.10 1.05 4.07 3.06 3.06

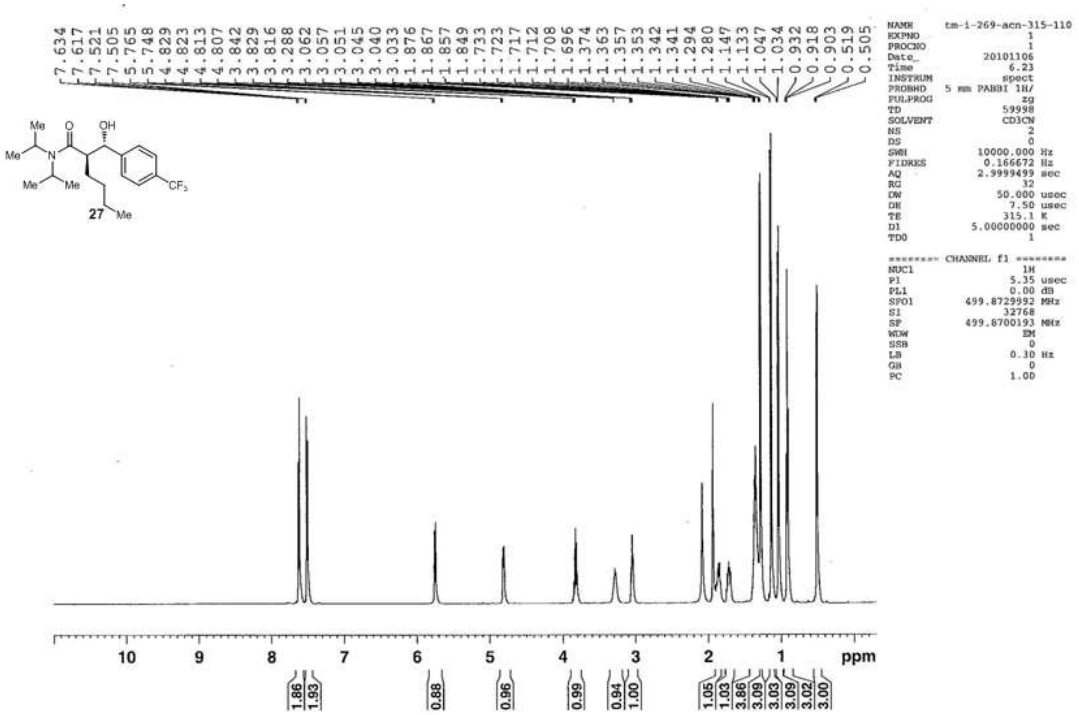

175.243
150.499
142.871
130.677
130.425
130.305
130.200
129.961
129.349
129.173
128.176
127.017
126.488
126.459
126.428
126.398
124.862
75.561
49.106
45.169
31.623
30.382
23.660
14.484
13.516
NAME tm-i-167-acn5
OH
N
O
Me
CF3
24
Me
220 200 180 160 140 120 100 80 60 40 20 0 ppm

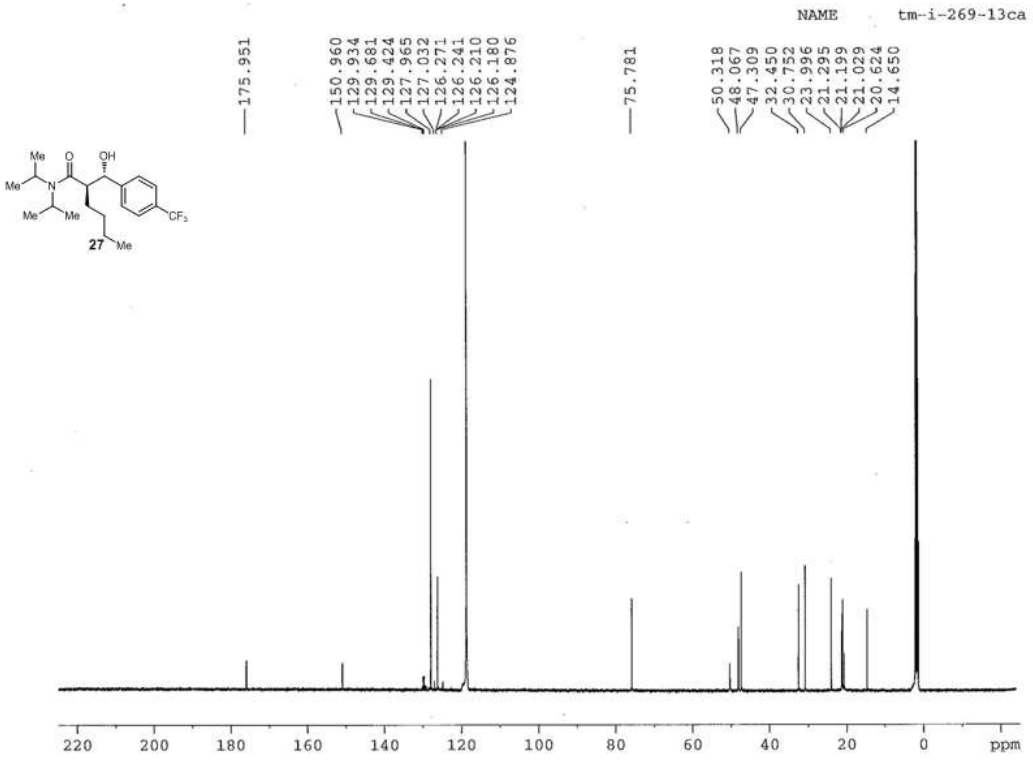

7.658
7.642
7.356
7.340
7.324
7.317
6.728
5.043
5.023
4.998
4.996
4.964
4.961
4.917
4.902
4.743
4.732
4.719
4.295
4.277
4.266
4.129
4.116
4.099
4.086
2.580
2.567
1.950
1.945
1.940
1.936
1.931
1.724
1.708
1.696
1.506
1.495
1.222
1.210
1.191
1.178
1.167
1.156
1.143
0.838
0.824
0.810
OH
O
N
CF3
25
Me
NAME tm-i-251-acn-330
EXPNO 2
PROCNO 1
Date_ 20101106
Time 3.54
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 16
DS 0
SWH 10000.000 H
FIDRES 0.166672 H
AQ 2.9999499 s
RG 25.4
DW 50.000 u
DE 7.50 u
TE 330.2 K
D1 5.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 u
PL1 0.00 d
SFO1 499.8729992 M
SI 32768
SF 499.8700187 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.01
4.98
1.77
0.83
0.93
1.03
0.95
0.99
1.03
1.08
1.00
1.05
1.05
4.06
3.09

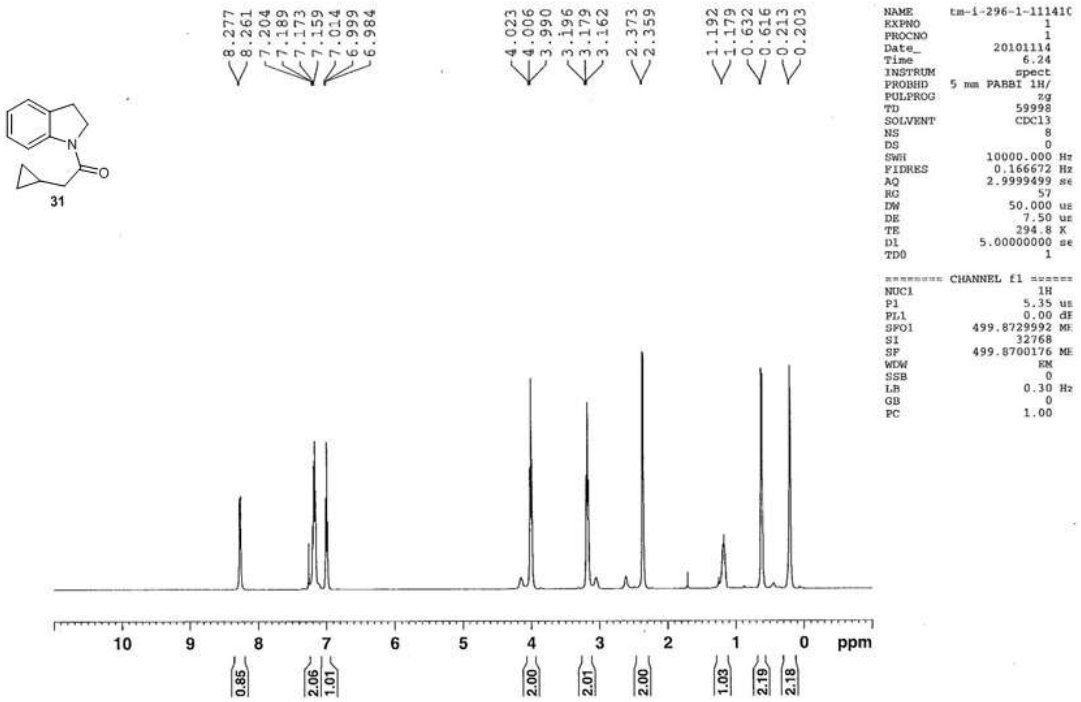

175.459
150.298
143.019
134.565
130.581
130.336
130.069
129.827
129.511
129.347
128.257
126.994
126.480
126.450
126.421
124.840
122.683
118.418
75.637
53.048
49.307
31.491
30.360
23.631
14.474
OH
O
N
CF3
25
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

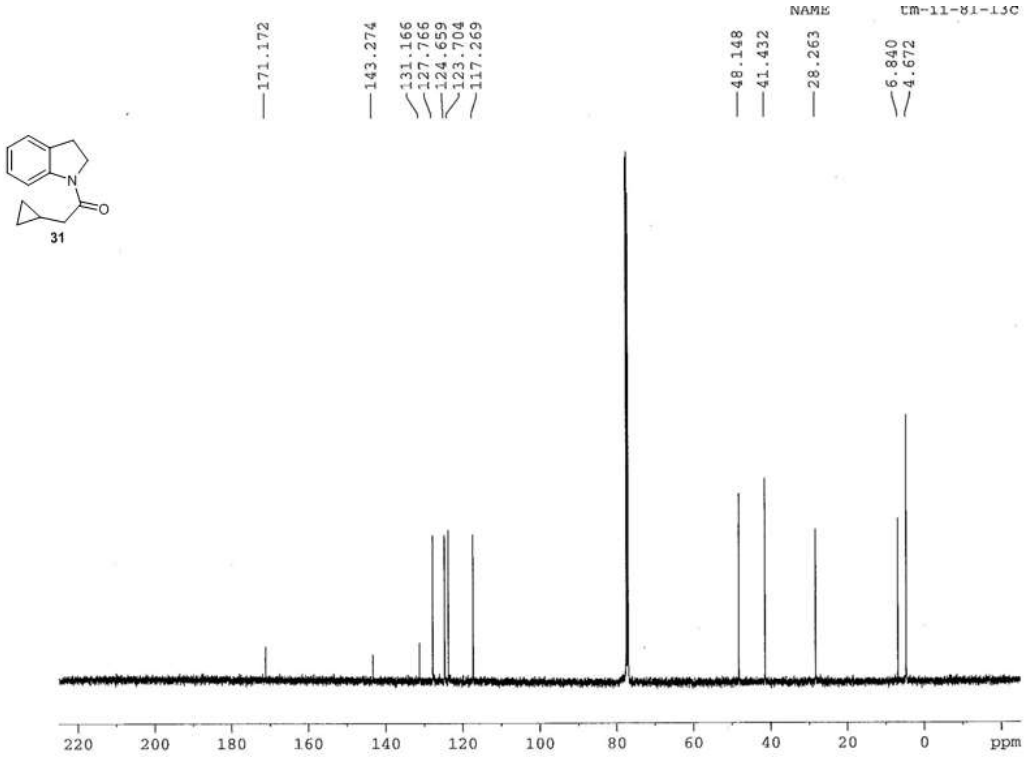

7.664
7.648
7.377
7.364
7.336
7.320
7.201
7.093
5.945
5.256
5.240
4.838
4.825
4.811
4.696
4.688
4.680
4.672
2.316
2.308
2.302
2.293
2.287
2.279
1.729
1.712
1.698
1.615
1.610
1.601
1.587
1.237
1.222
1.212
1.207
1.203
1.197
1.194
1.184
1.177
1.170
1.164
1.150
0.966
0.953
0.862
0.848
NAME tm-1-249-hacn-330-101
EXPNO 1
PROCNO 1
Date_ 20101113
Time 8.29
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 114
DW 50.000 usec
DE 7.50 usec
TE 329.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700191 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
N
O
Me
Me
CF3
26
Me
10
9
8
7
6
5
4
3
2
1
ppm
1.90
1.84
2.04
0.95
0.89
0.89
0.86
0.96
1.07
1.00
1.07
1.03
4.10
3.11
6.12

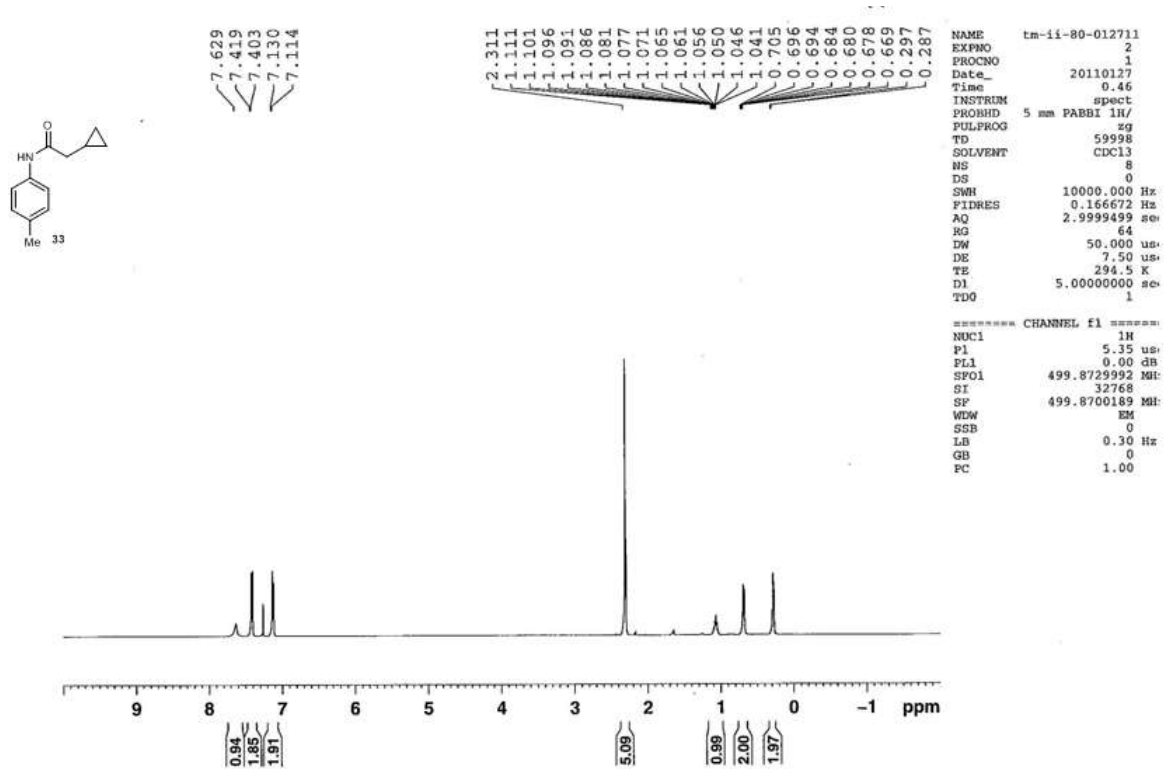

175.315
150.623
138.787
131.817
131.757
130.276
130.193
130.144
130.019
129.675
128.146
127.018
126.444
126.416
126.386
126.356
124.862
75.300
49.654
47.240
31.571
30.421
23.672
21.524
21.337
14.493
NAME tm-1-249-cdcl3
OH
N
O
Me
Me
CF3
26
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

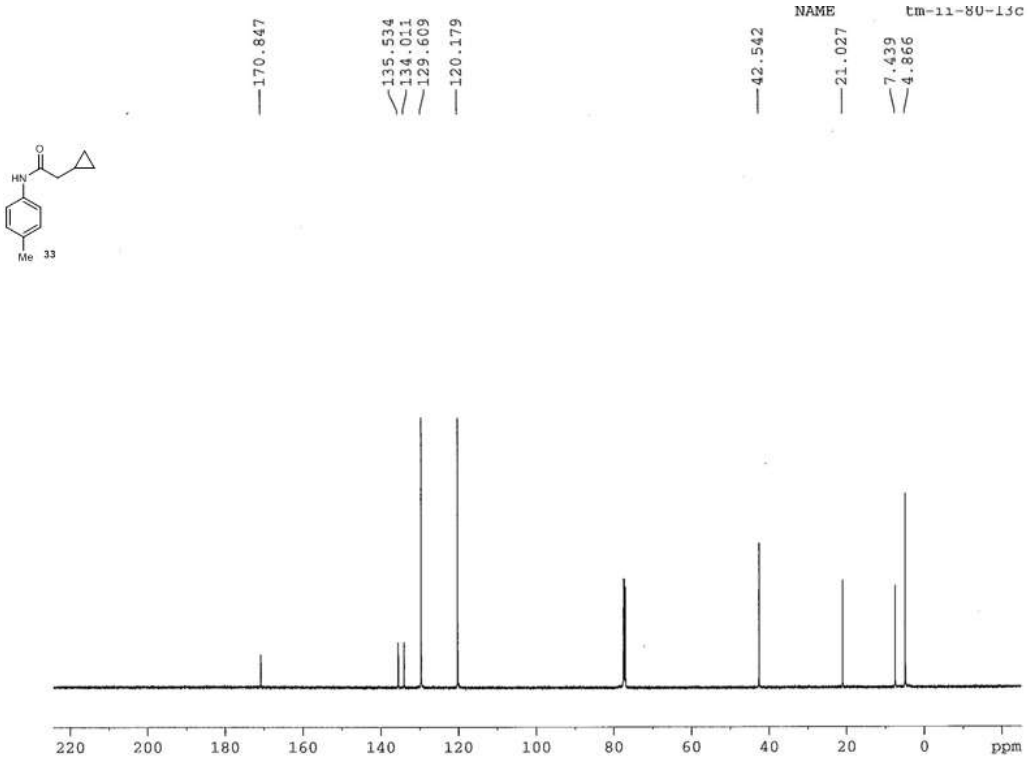

7.634
7.617
7.521
7.505
5.765
5.748
4.829
4.823
4.813
4.807
3.842
3.829
3.816
3.288
3.062
3.057
3.051
3.045
3.040
3.033
1.876
1.867
1.857
1.849
1.733
1.723
1.717
1.712
1.708
1.696
1.374
1.363
1.357
1.353
1.342
1.341
1.294
1.280
1.147
1.133
1.047
1.034
0.932
0.918
0.903
0.519
0.505
NAME tm-i-269-acn-315-110
EXPNO 1
PROCNO 1
Date_ 20101106
Time 6.23
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD3CN
NS 2
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 32
DW 50.000 usec
DE 7.50 usec
TE 315.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
OH
N
Me
Me
Me
Me
CF3
27
Me
10
9
8
7
6
5
4
3
2
1
ppm
1.86
1.93
0.88
0.96
0.99
0.94
1.00
1.05
1.03
3.86
3.09
3.03
3.09
3.02
3.00

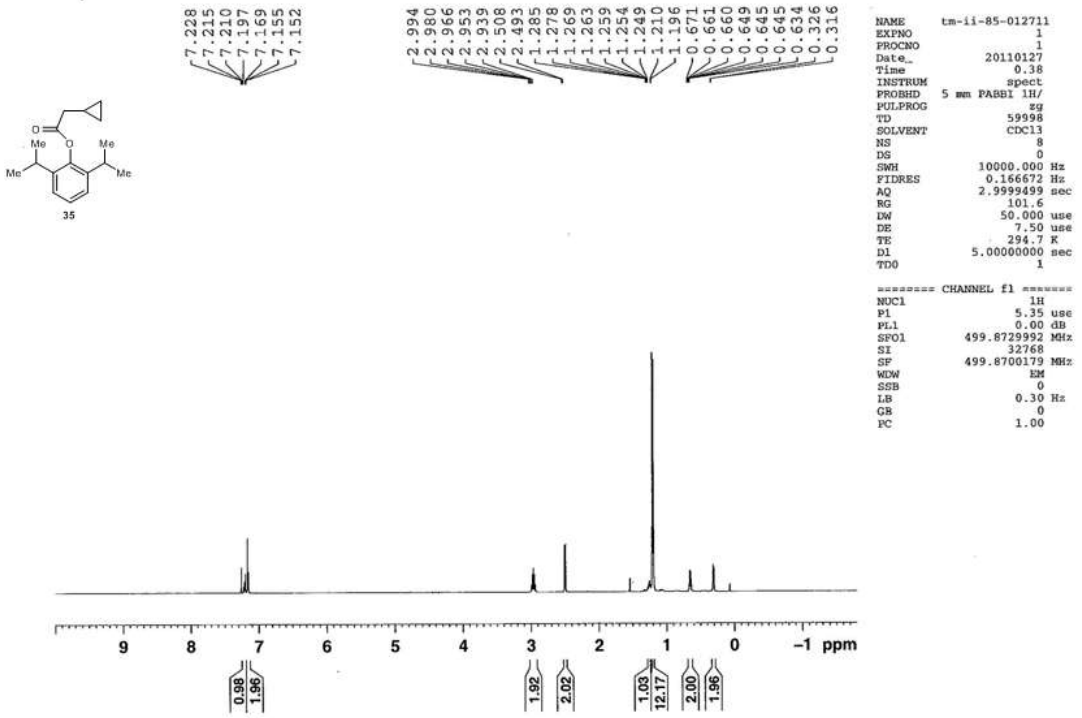

NAME tm-i-269-13ca
175.951
150.960
129.934
129.681
129.424
127.965
127.032
126.271
126.241
126.210
126.180
124.876
75.781
50.318
48.067
47.309
32.450
30.752
23.996
21.295
21.199
21.029
20.624
14.650
Me
O
OH
N
Me
Me
Me
Me
CF3
27
Me
220
200
180
160
140
120
100
80
60
40
20
0
ppm

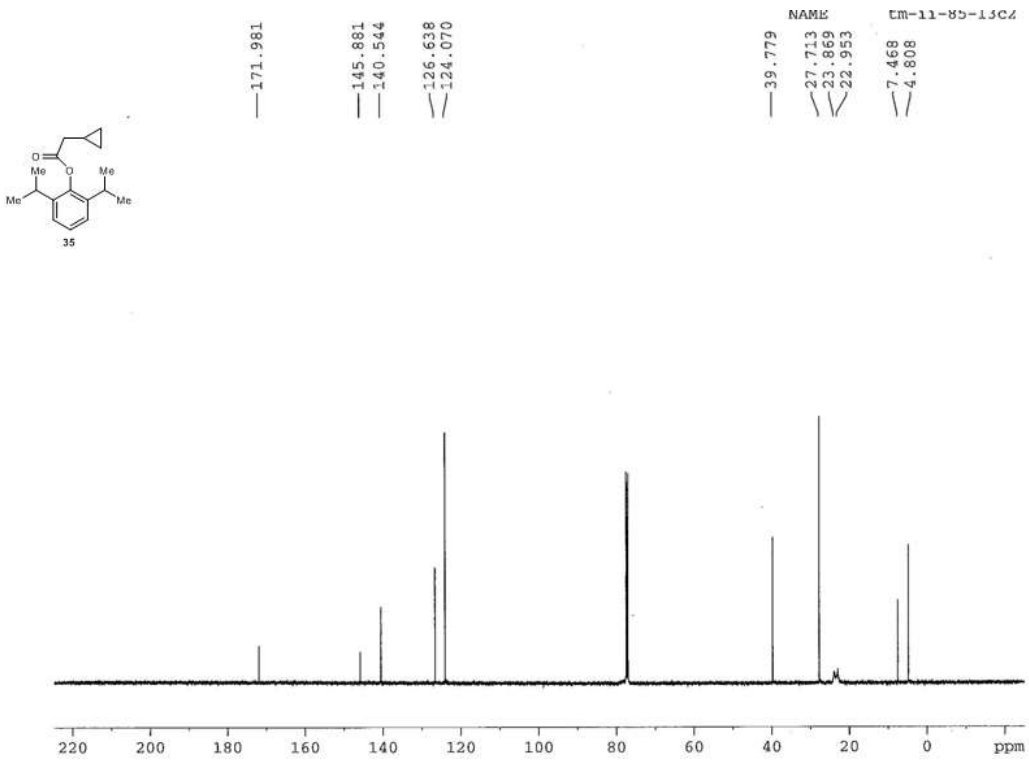

8.277
8.261
7.204
7.189
7.173
7.159
7.014
6.999
6.984
4.023
4.006
3.990
3.196
3.179
3.162
2.373
2.359
1.192
1.179
0.632
0.616
0.213
0.203
31
NAME tm-i-296-1-111410
EXPNO 1
PROCNO 1
Date_ 20101114
Time 6.24
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 se
RG 57
DW 50.000 us
DE 7.50 us
TE 294.8 K
D1 5.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 us
PL1 0.00 dB
SFO1 499.8729992 MH
SI 32768
SF 499.8700176 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.85 2.06 1.01 2.00 2.01 2.00 1.03 2.19 2.18

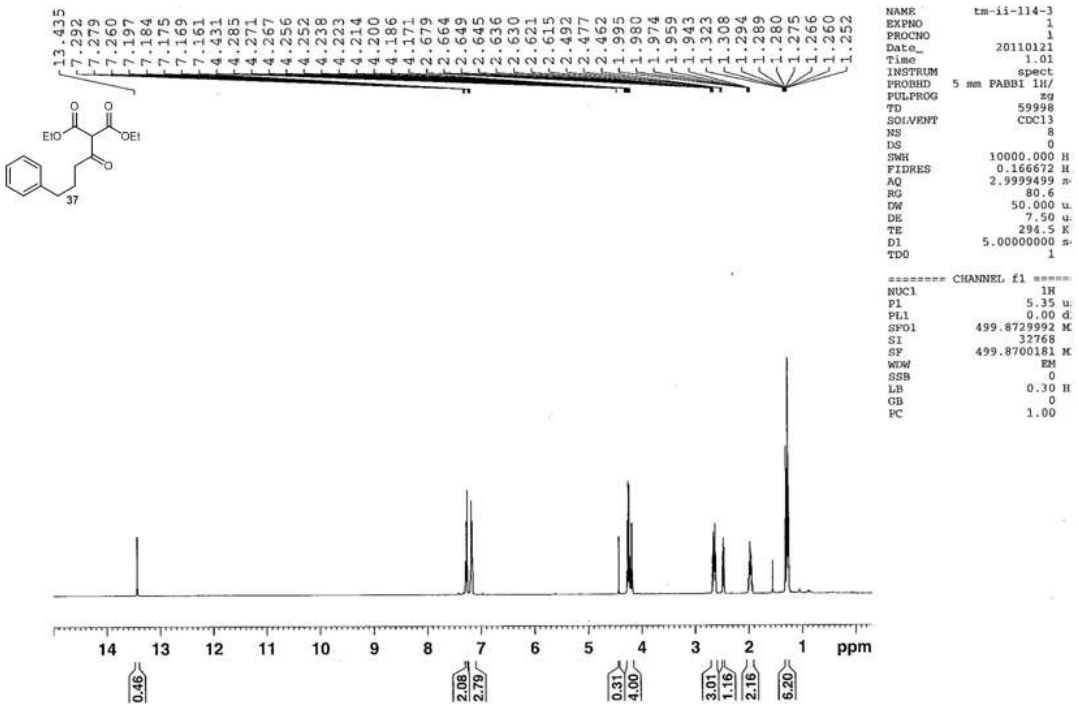

NAME tm-11-81-13C
171.172
143.274
131.166
127.766
124.659
123.704
117.269
48.148
41.432
28.263
6.840
4.672
31
220 200 180 160 140 120 100 80 60 40 20 0 ppm

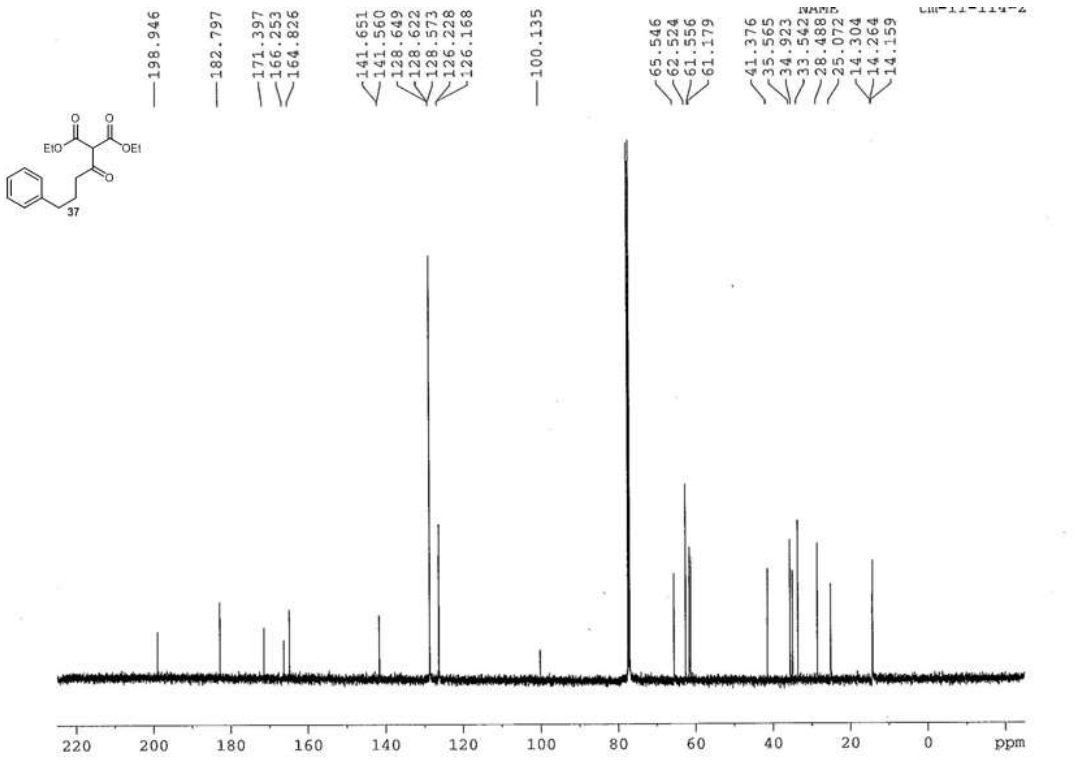

7.629
7.419
7.403
7.130
7.114
2.311
1.111
1.101
1.096
1.091
1.086
1.081
1.077
1.071
1.065
1.061
1.056
1.050
1.046
1.041
0.705
0.696
0.694
0.684
0.680
0.678
0.669
0.297
0.287
HN
Me
33
NAME tm-ii-80-012711
EXPNO 2
PROCNO 1
Date_ 20110127
Time 0.46
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 se
RG 64
DW 50.000 us
DE 7.50 us
TE 294.5 K
D1 5.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 5.35 us
PL1 0.00 dB
SFO1 499.8729992 MH
SI 32768
SF 499.8700189 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 −1 ppm
0.94
1.85
1.91
5.09
0.99
2.00
1.97

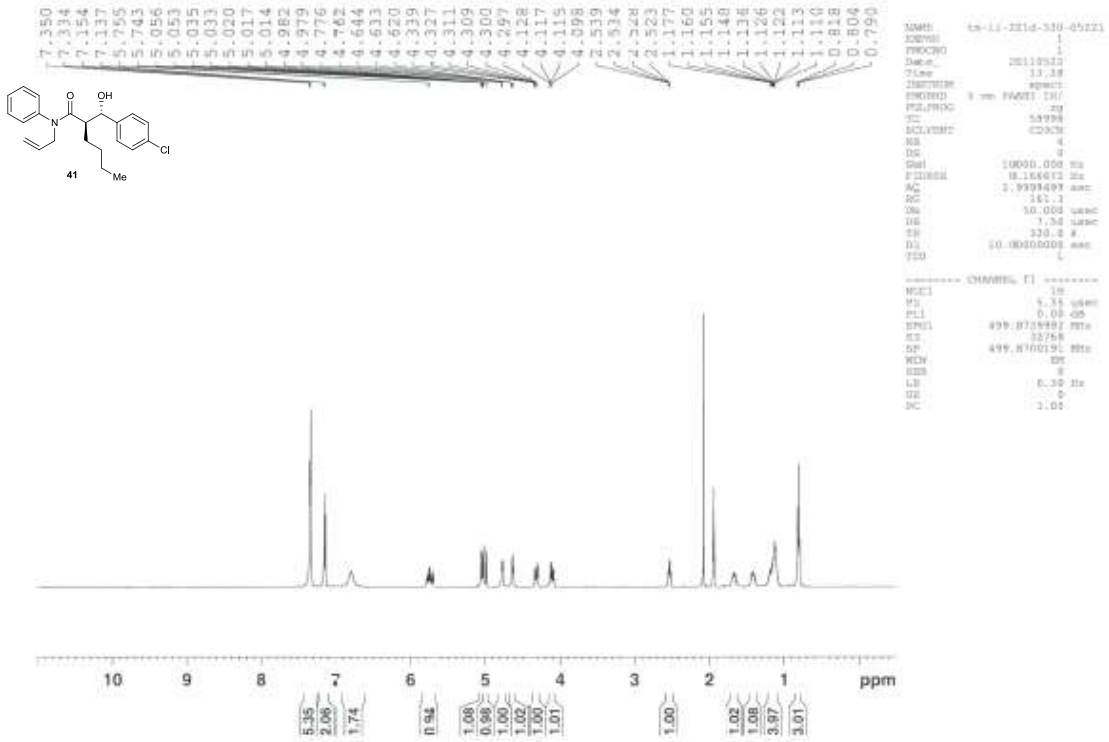

NAME tm-ii-80-13c
170.847
135.534
134.011
129.609
120.179
42.542
21.027
7.439
4.866
HN
Me
33
220 200 180 160 140 120 100 80 60 40 20 0 ppm

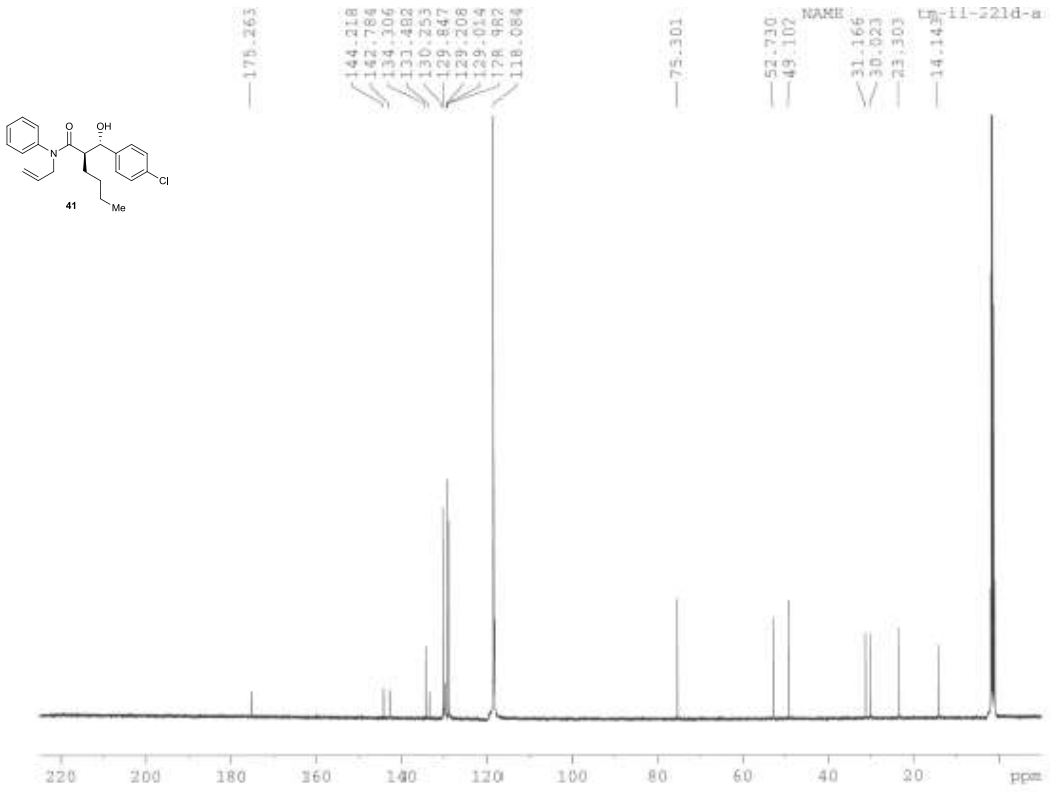

7.228
7.215
7.210
7.197
7.169
7.155
7.152
2.994
2.980
2.966
2.953
2.939
2.508
2.493
1.285
1.278
1.269
1.263
1.259
1.254
1.249
1.210
1.196
0.671
0.661
0.660
0.649
0.645
0.645
0.634
0.326
0.316
NAME tm-ii-85-012711
EXPNO 1
PROCNO 1
Date_ 20110127
Time 0.38
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 101.6
DW 50.000 use
DE 7.50 use
TE 294.7 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 use
PL1 0.00 dB
SFO1 499.8729992 MHz
SI 32768
SF 499.8700179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
Me
Me
O
O
35
9
8
7
6
5
4
3
2
1
0
−1 ppm
0.98
1.96
1.92
2.02
1.03
12.17
2.00
1.96

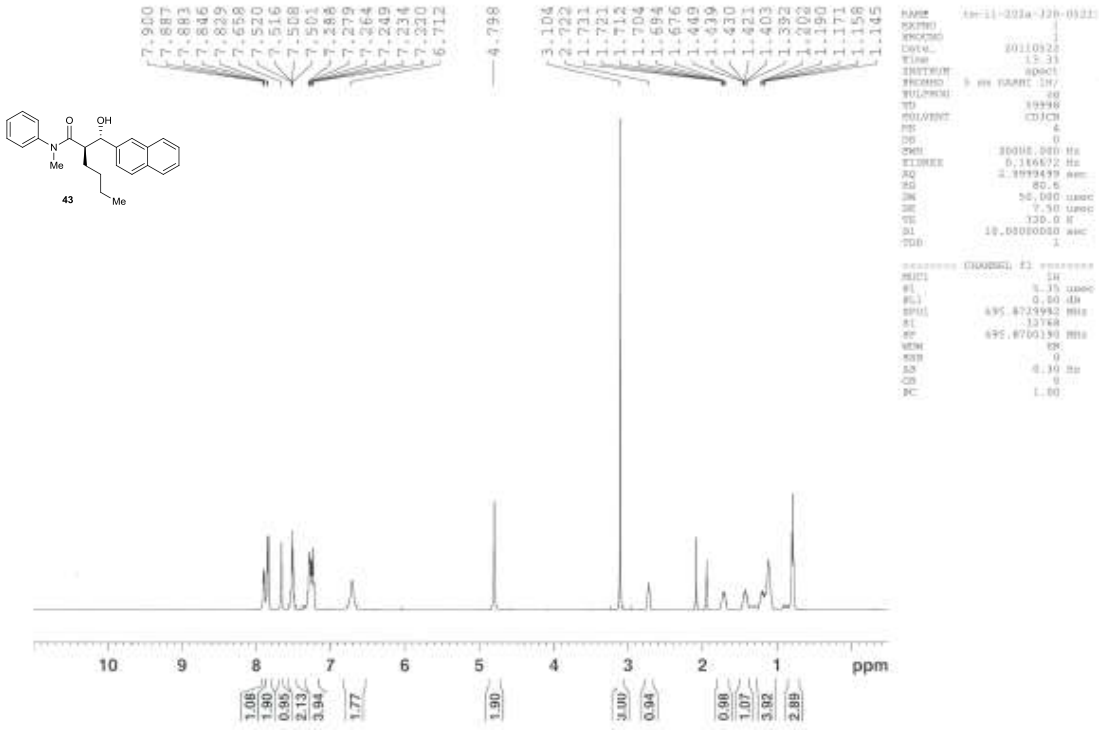

171.981
145.881
140.544
126.638
124.070
39.779
27.713
23.869
22.953
7.468
4.808
NAME tm-ii-85-13C2
Me
Me
Me
Me
O
O
35
220
200
180
160
140
120
100
80
60
40
20
0
ppm

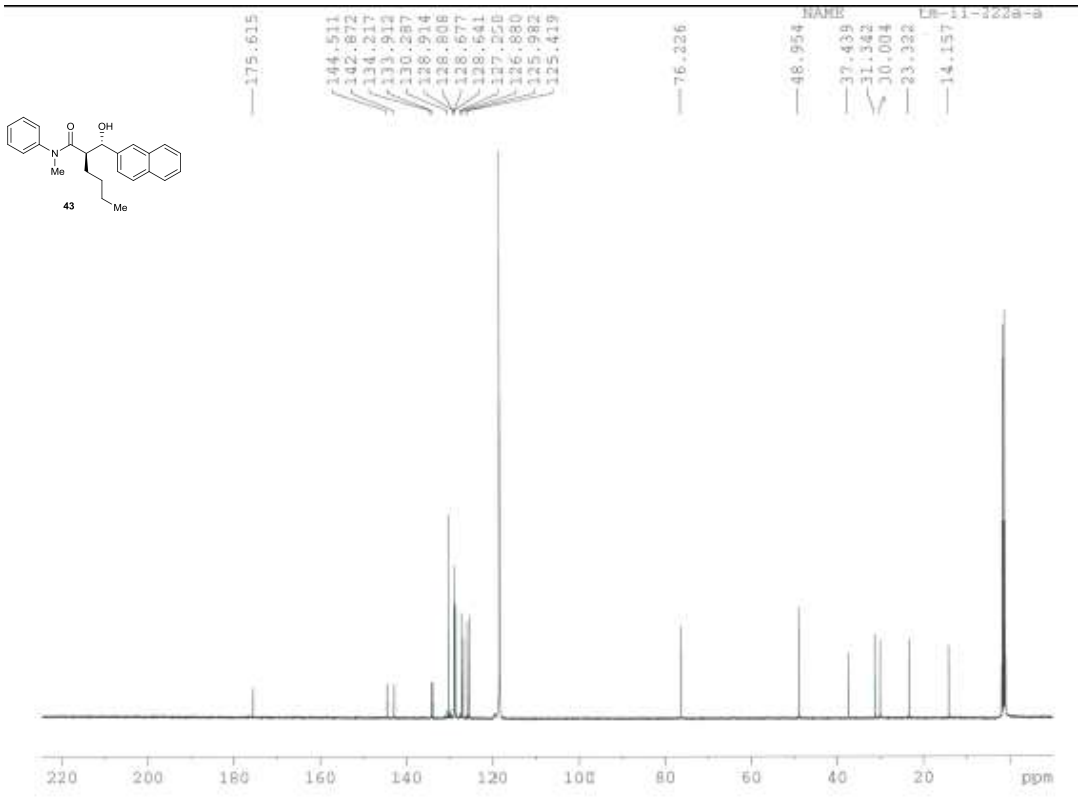

13.435
7.292
7.279
7.260
7.197
7.184
7.175
7.169
7.161
4.431
4.285
4.271
4.267
4.256
4.252
4.238
4.223
4.214
4.200
4.186
4.171
2.679
2.664
2.649
2.645
2.636
2.630
2.621
2.615
2.492
2.477
2.462
1.995
1.980
1.974
1.959
1.943
1.323
1.308
1.294
1.289
1.280
1.275
1.266
1.260
1.252
EtO
OEt
37
NAME tm-ii-114-3
EXPNO 1
PROCNO 1
Date_ 20110121
Time 1.01
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDC13
NS 8
DS 0
SWH 10000.000 H
FIDRES 0.166672 H
AQ 2.9999499 s
RG 80.6
DW 50.000 u
DE 7.50 u
TE 294.5 K
D1 5.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 5.35 u
PL1 0.00 d
SFO1 499.8729992 M
SI 32768
SF 499.8700181 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.46
2.08
2.79
0.31
4.00
3.01
1.16
2.16
6.20

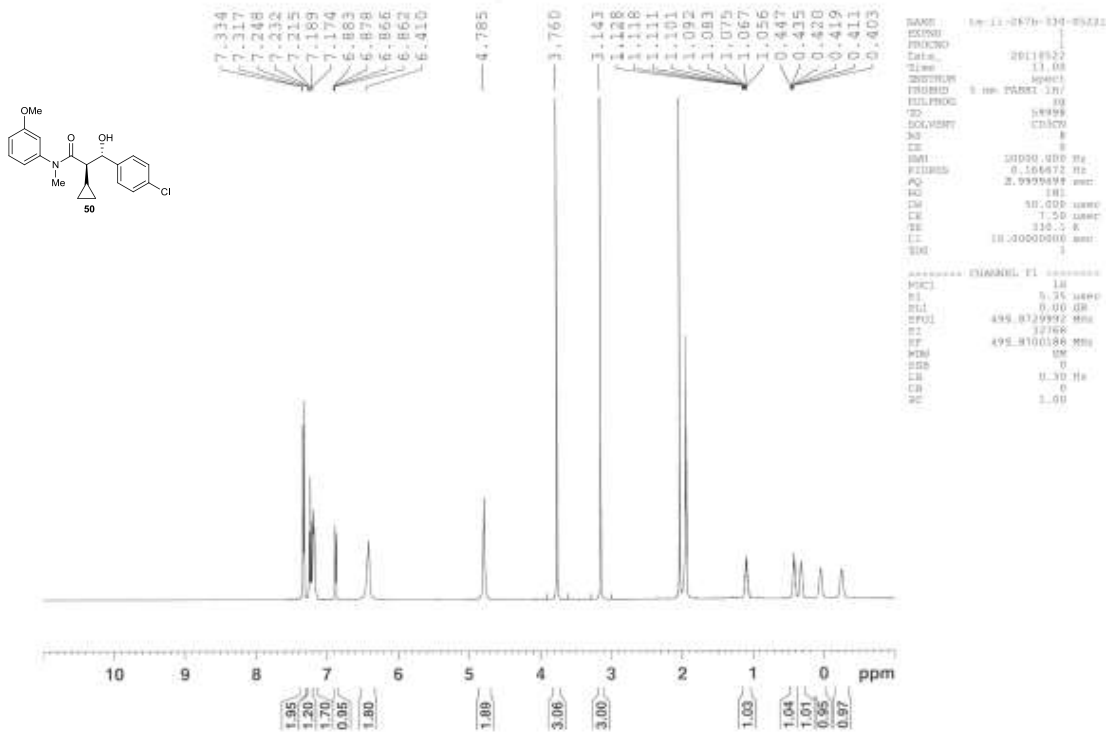

198.946
182.797
171.397
166.253
164.826
141.651
141.560
128.649
128.622
128.573
126.228
126.168
100.135
65.546
62.524
61.556
61.179
41.376
35.565
34.923
33.542
28.488
25.072
14.304
14.264
14.159
EtO
OEt
37
220 200 180 160 140 120 100 80 60 40 20 0 ppm

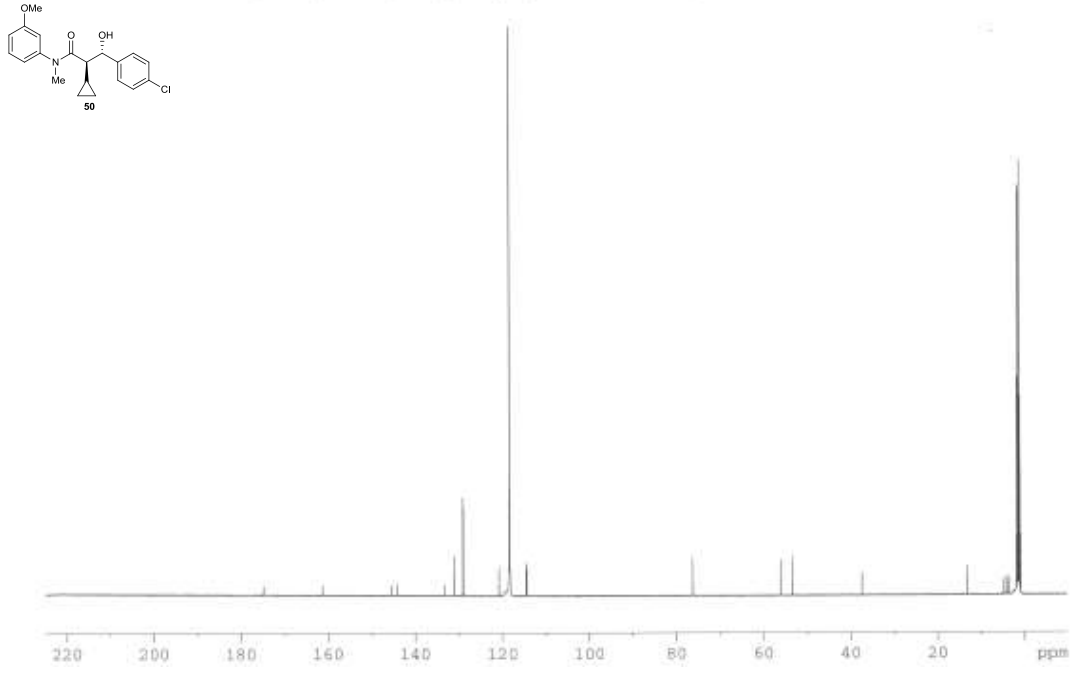

41
Me
Cl
OH
ppm

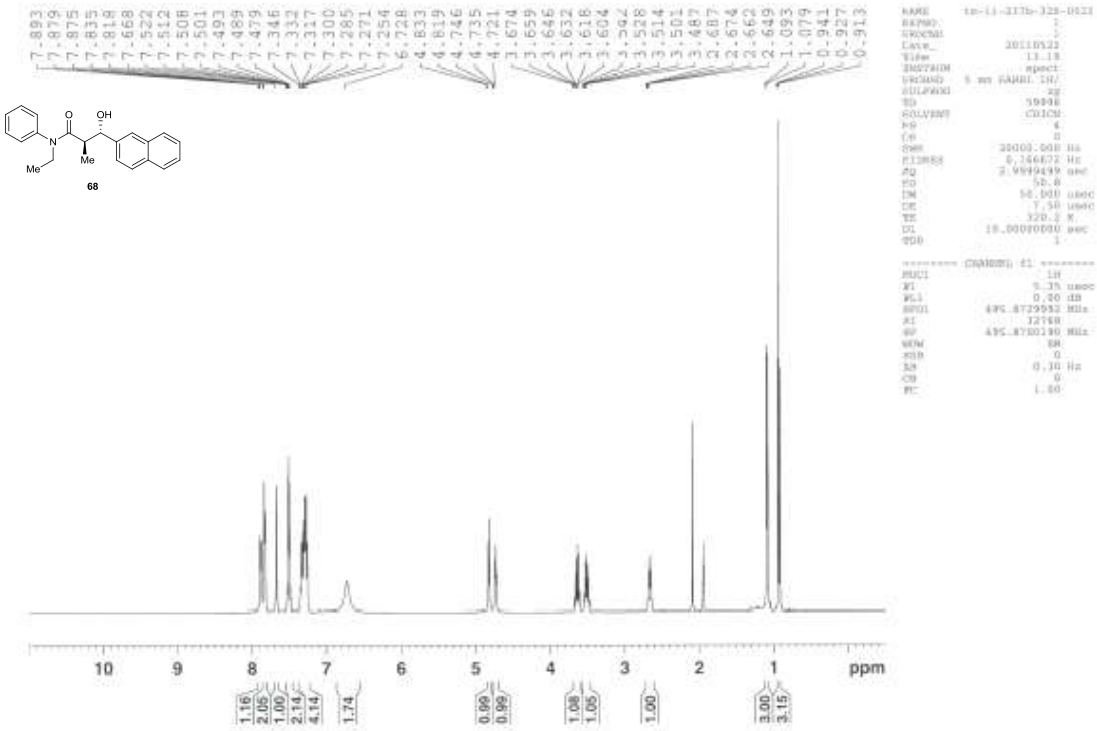

41
Me
Cl
OH
NAME
ppm

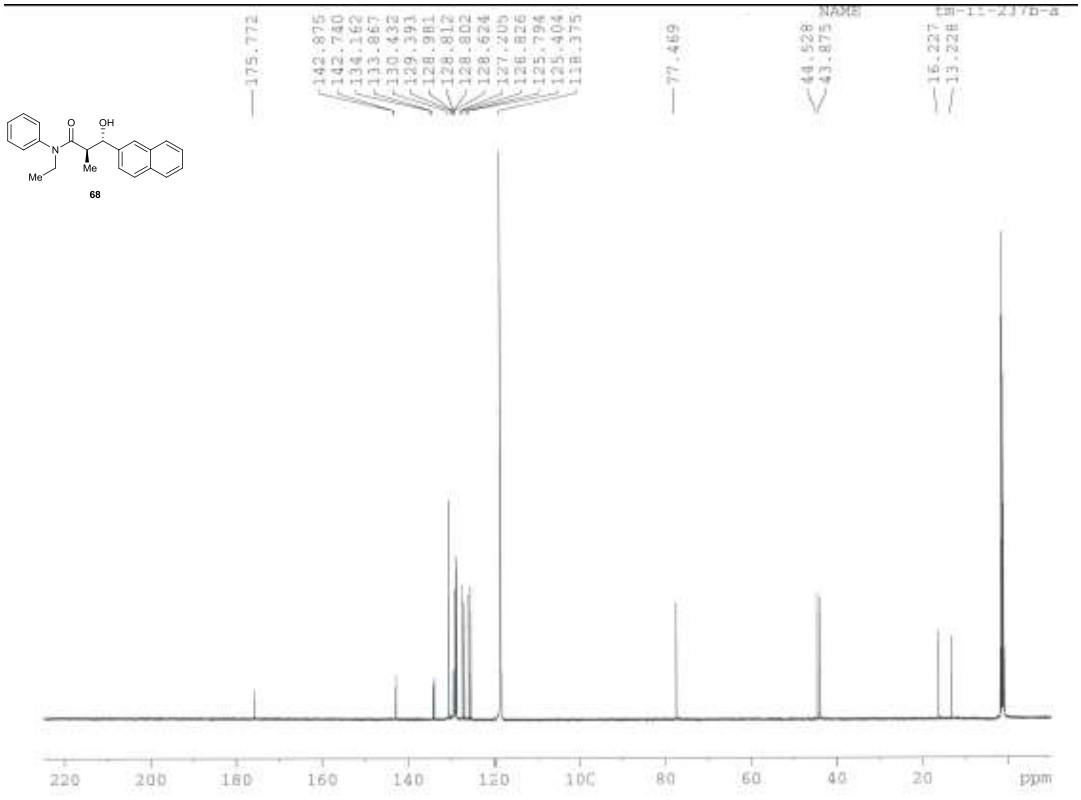

7.900
7.887
7.883
7.846
7.829
7.658
7.520
7.516
7.508
7.501
7.288
7.279
7.264
7.249
7.234
7.220
6.712
4.798
3.104
2.722
1.731
1.721
1.712
1.704
1.694
1.676
1.449
1.439
1.430
1.421
1.403
1.392
1.202
1.190
1.171
1.158
1.145
OH
N
Me
43
Me
10
9
8
7
6
5
4
3
2
1
ppm
1.08
1.90
0.95
2.13
3.94
1.77
1.90
3.00
0.94
0.98
1.07
3.02
2.89

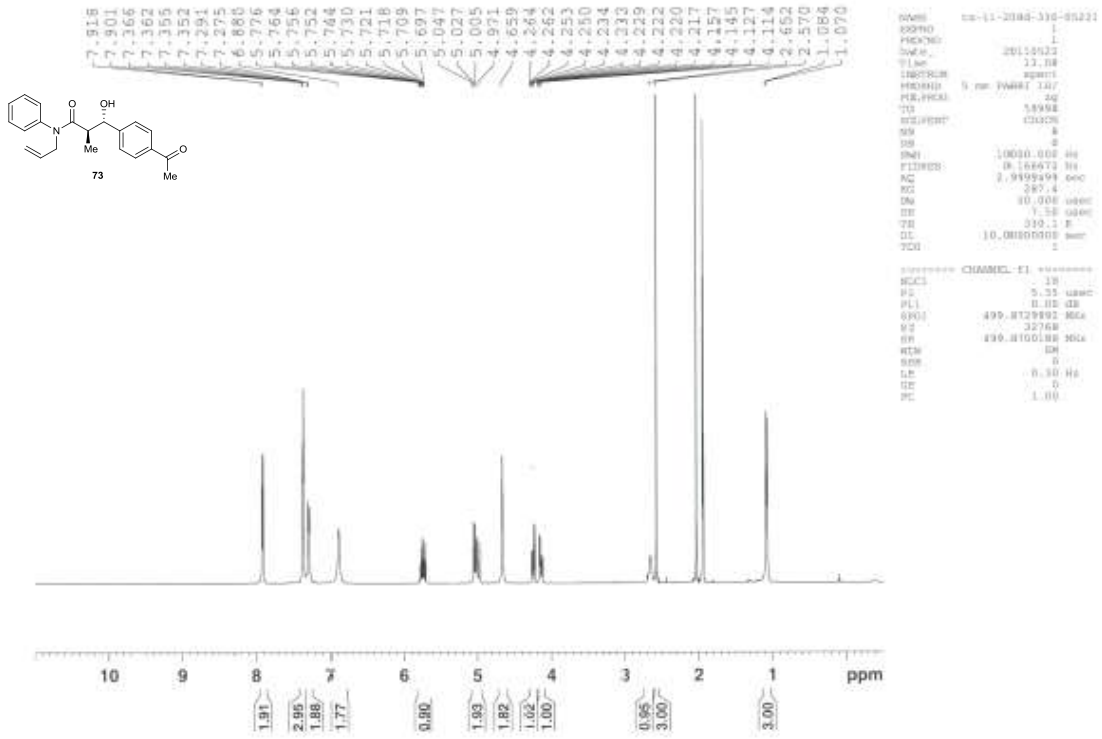

175.615
144.511
142.872
134.217
133.912
130.287
128.914
128.808
128.677
128.641
127.250
126.880
125.982
125.419
76.226
48.954
37.439
31.342
30.004
23.322
14.157
OH
N
Me
43
Me
220
200
180
160
140
120
100
80
60
40
20
ppm


OMe
O
OH
N
Me
Cl
50
7.334
7.317
7.248
7.224
7.215
7.188
7.174
6.883
6.878
6.866
6.862
6.410
4.785
3.760
3.143
3.128
3.118
3.111
3.102
3.083
3.075
3.067
3.056
0.447
0.435
0.420
0.419
0.411
0.403
10
9
8
7
6
5
4
3
2
1
0
ppm
1.95
1.20
1.70
0.95
1.80
1.00
3.06
3.00
1.03
1.04
1.01
0.95
0.97


OMe
O
OH
N
Me
Cl
50
174.836
161.314
145.408
144.272
133.354
131.088
129.082
128.966
120.803
114.526
114.380
76.335
56.160
53.404
37.389
13.045
4.724
4.124
220
200
180
160
140
120
100
80
60
40
20
ppm


68
ppm


68
ppm


7.918
7.901
7.366
7.362
7.355
7.352
7.291
7.275
6.888
5.776
5.764
5.756
5.752
5.744
5.730
5.721
5.718
5.709
5.697
5.047
5.027
5.005
4.971
4.659
4.264
4.262
4.253
4.250
4.234
4.233
4.229
4.222
4.220
4.217
4.157
4.145
4.127
4.114
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73
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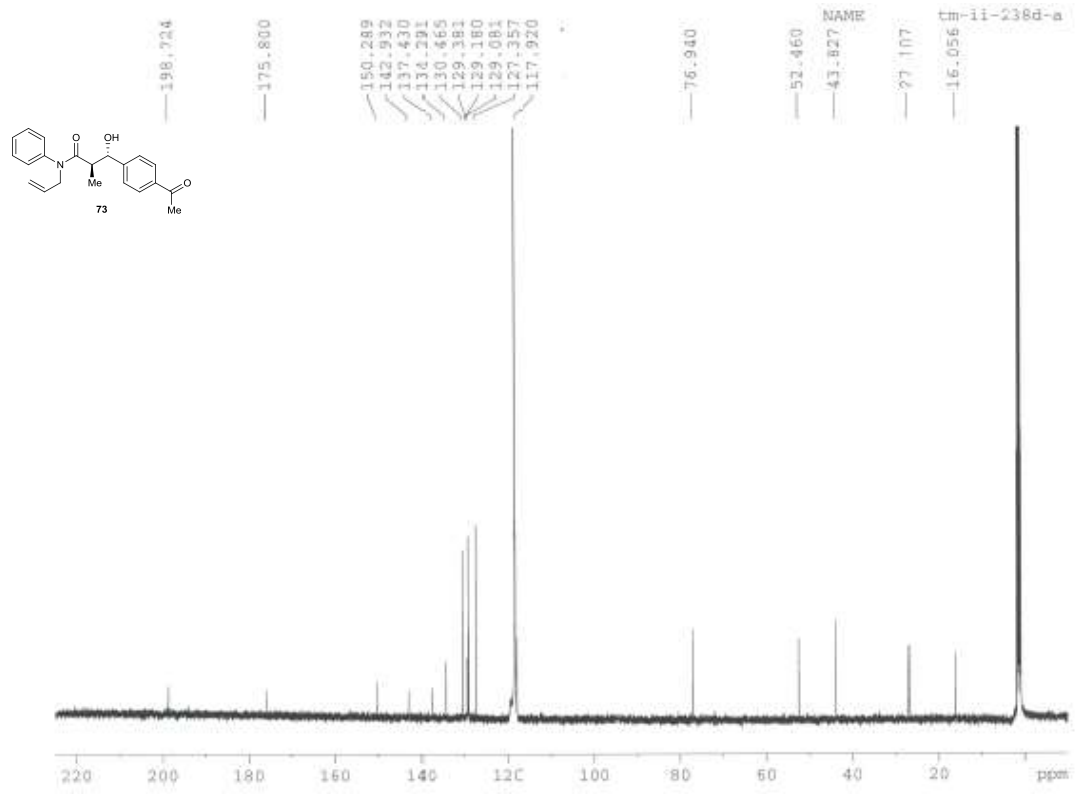
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