Electronic Supplementary Material (ESI) for Chemical Communications


Supplementary Information

# Direct use of dioxygen as oxygen source: catalytic oxidative synthesis of amides

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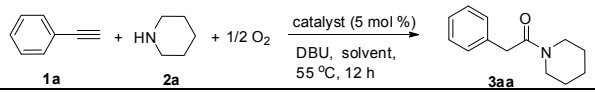
## 1. General Information

All commercially available reagent grade chemicals were purchased from Aldrich, Acros and Alfa Aesar Chemical Company and used as received without further purification unless otherwise stated. All solvents were dried according to standard procedures. $^{1}H$ NMR and $^{13}C$ NMR spectra were recorded in $CDCl_3$ on a Bruker Avance 600 spectrometer with TMS as internal standard (600 MHz $^{1}H$, 150 MHz $^{13}C$) at room temperature, and the chemical shifts ($\delta$) were expressed in ppm and $J$ values were given in Hz. The following abbreviations are used to indicate the multiplicity: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m) or broad (br). IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer. Mass analyses and HRMS were obtained on a Finnigan-LCQDECA mass spectrometer and a Bruker Daltonics Bio-TOF-Q mass spectrometer by the ESI method, respectively. Column chromatography was performed on silica gel (200 - 300 mesh).

## 2. Optimization of reaction conditions

***Table S1.*** The reaction of **1a** and **2a** in different conditions[a]



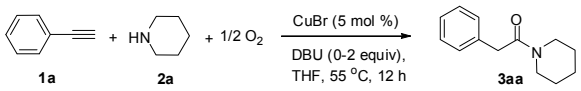

1a 2a 3aa

| entry | catalyst | solvent | yield (%)[b] |
|---|---|---|---|
| 1 | $RuCl_3$, or $RhCl_3$, or $Pd(OAc)_2$, or $AuBr_3$, or AgOTf, or $NiCl_2$, or $Ti(OiPr)_4$, or $(C_5H_5)_2ZrCl_2$, or $FeBr_3$ | THF | 0 |
| 2 | CuI | THF | 46 |
| 3 | $(CH_3CN)_4CuPF_6$ | THF | 60 |
| 4 | $Cu(OTf)_2$ | THF | 34 |
| 5 | CuCl | THF | 55 |
| 6 | $CuCl_2$ | THF | 53 |
| 7 | $CuBr_2$ | THF | 51 |
| 8 | CuBr | THF | 68 |
| 9 | CuBr | DME | 63 |
| 10 | CuBr | $CH_3CN$ | 53 |
| 11 | CuBr | Toluene | 55 |
| 12 | CuBr | 1,4-dioxane | 56 |
| 13 | CuBr | $CH_2Cl_2$ | 31[c] |
| 14 | CuBr | THF:$H_2O$ (10:1) | 43 |
| 15 | CuBr | $CH_3OH$ | 0 |
| 16 | CuBr | THF | 31[d] |
| 17 | CuBr | THF | 55[e] |
| 18 | CuBr | THF | 69[f] |
| 19 | CuBr | THF | 67[g] |

[a] Reaction conditions: alkyne **1a** (2.5 mmol), piperidine **2a** (0.5 mmol), catalyst (5 mol %), DBU (0.6 mmol), solvent (1.0 mL), 55 °C, 12 h, $O_2$ (balloon). [b] Isolated yields based on **2a**. [c] 40 °C. [d] alkyne **1a** (1 mmol). [e] alkyne **1a** (2 mmol). [f] alkyne **1a** (3 mmol). [g] alkyne **1a** (2.5 mmol), 65°C.

***Table S2.*** The effects of different amount of DBU on the reaction of **1a** with **2a**[a]

To investigate the yield or rate of this reaction affected by the amount of DBU, several control experiments were performed between phenylacetylene **1a** and piperidine **2a** under different conditions. As shown in Table S2, the reaction was conducted slowly and a low yield was obtained in the absence of DBU or when the loading of DBU was less than 1 equiv. When the amount of DBU was increased to 1.2 equiv, a good yield was obtained. Further increase of the amount of DBU, the yield of amide did not obviously improve.




| entry | DBU (equiv.) | **3aa** yield (%)[b] |
|---|---|---|
| 1 | - | 4% |
| 2 | 0.2 | 36% |
| 3 | 0.2 | 36.5% [c] |
| 4 | 0.5 | 45% |
| 5 | 1.2 | 68% |
| 6 | 2 | 69% |

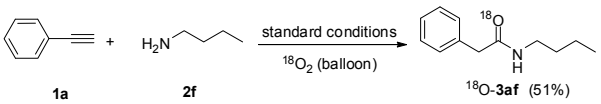
[a] Reaction conditions: alkyne **1a** (2.5 mmol), piperidine **2a** (0.5 mmol), CuBr (5 mol %), DBU (0-2 equiv), THF (1.0 mL), 55 °C, 12 h, $O_2$ (balloon). [b] Isolated yields based on **2a**. [c] Time: 24h.

## 3. General Procedure for Copper-Catalyzed Oxidative Amidation of Alkynes

Alkyne (2.5 mmol) was added to a mixture of amine (0.5 mmol), CuBr (5 mol %), and DBU (0.6 mmol) in THF (1 mL) (or without solvent) at room temperature under $O_2$ (balloon). The reaction mixture was stirred at 55 °C for 4-24 h. After cooling down to room temperature and concentrating in vacuum, the residue was purified by flash chromatography on a short silica gel to give the desired products.

## 4. Labeling Experiments.

### 4.1 Oxidative Amidation of Phenylacetylene with *n*-butylamine under $^{18}O_2$.



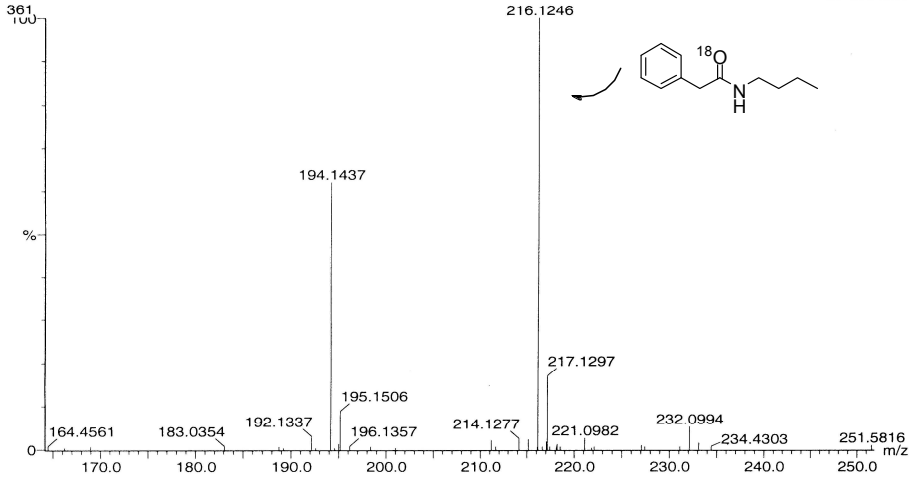

Phenylacetylene (2.5 mmol) was added to a mixture of *n*-butylamine (0.5 mmol), CuBr (5 mol %), and DBU (0.6 mmol) in THF (1 mL) at room temperature under $^{18}O_2$ (balloon). The reaction mixture was stirred at 55 °C for 4 h. After cooling down to room temperature and concentrating in vacuum, the residue was purified by flash chromatography on a short silica gel to give the $^{18}O$-labelled product **3af** in 51% yield.

The HRMS spectrum of $^{18}O$-**3af** for the reaction under $^{18}O_2$ (97%).

**Elemental Composition Report** **Page 1**

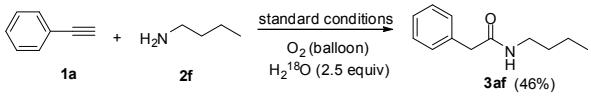
**Single Mass Analysis**
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
1193 formula(e) evaluated with 38 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-4 16O: 0-10 18O: 0-10 Na: 0-1
19:18:07 01-Feb-2010 TOF MS ES+




| | | | | | | |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 100.0 | 50.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 216.1246 | 216.1250 | -0.4 | -1.9 | 4.5 | 13.1 | C12 H17 N 18O Na |
| 194.1437 | 194.1431 | 0.6 | 3.1 | 4.5 | 5.4 | C12 H18 N 18O |

**4.2 Oxidative Amidation of Phenylacetylene with *n*-butylamine in the presence of $H_2{}^{18}O$ under $O_2$.**




Phenylacetylene (2.5 mmol) was added to a mixture of *n*-butylamine (0.5 mmol), $H_2{}^{18}O$ (1.25 mmol), CuBr (0.025 mmol), and DBU (0.6 mmol) in THF (1 mL) at room temperature under $O_2$ (balloon). The reaction mixture was stirred at 55 °C for 4 h. After cooling down to room temperature and concentrating in vacuum, the residue was purified by flash chromatography on a short silica gel to give the product **3af** in 46% yield.

The HRMS spectrum of **3af** for the reaction in the presence of $H_2{}^{18}O$.

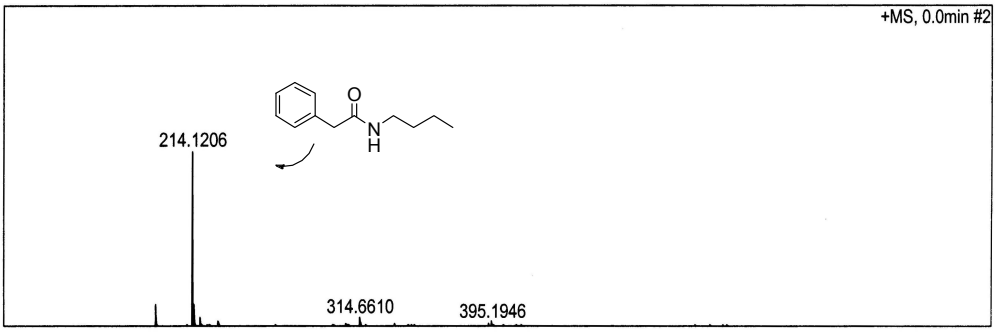
## Mass Spectrum SmartFormula Report

**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | E:\Data\micrOTOFQ\Chengdu_WZJ\20091216WU\WW091201-1.d | Acquisition Date | 12/17/2009 11:46:56 AM |
| Method | tune_low_20090830.m | Operator | Pu Hai |
| Sample Name | WW091201-1 | Instrument / Ser# | micrOTOF-Q II 10203 |
| Comment | | | |

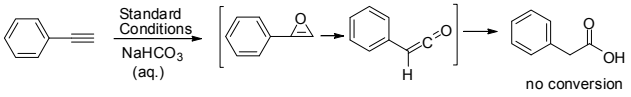
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.4 Bar |
| Focus | Not active | Set Capillary | 3500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 2000 m/z | Set Collision Cell RF | 100.0 Vpp | Set Divert Valve | Source |



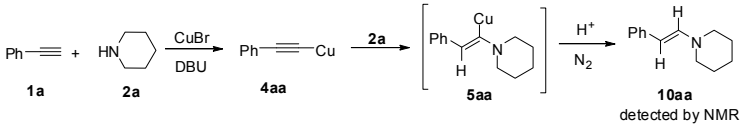

| Meas. m/z | # | Formula | Score | m/z | err [mDa] | err [ppm] | mSigma | rdb | e⁻ Conf | N-Rule |
|---|---|---|---|---|---|---|---|---|---|---|
| 214.1206 | 1 | C 12 H 17 N Na O | 100.00 | 214.1202 | -0.4 | -1.7 | 3.1 | 4.5 | even | ok |

## 5. Reaction of Phenylacetylene with Aqueous $NaHCO_3$



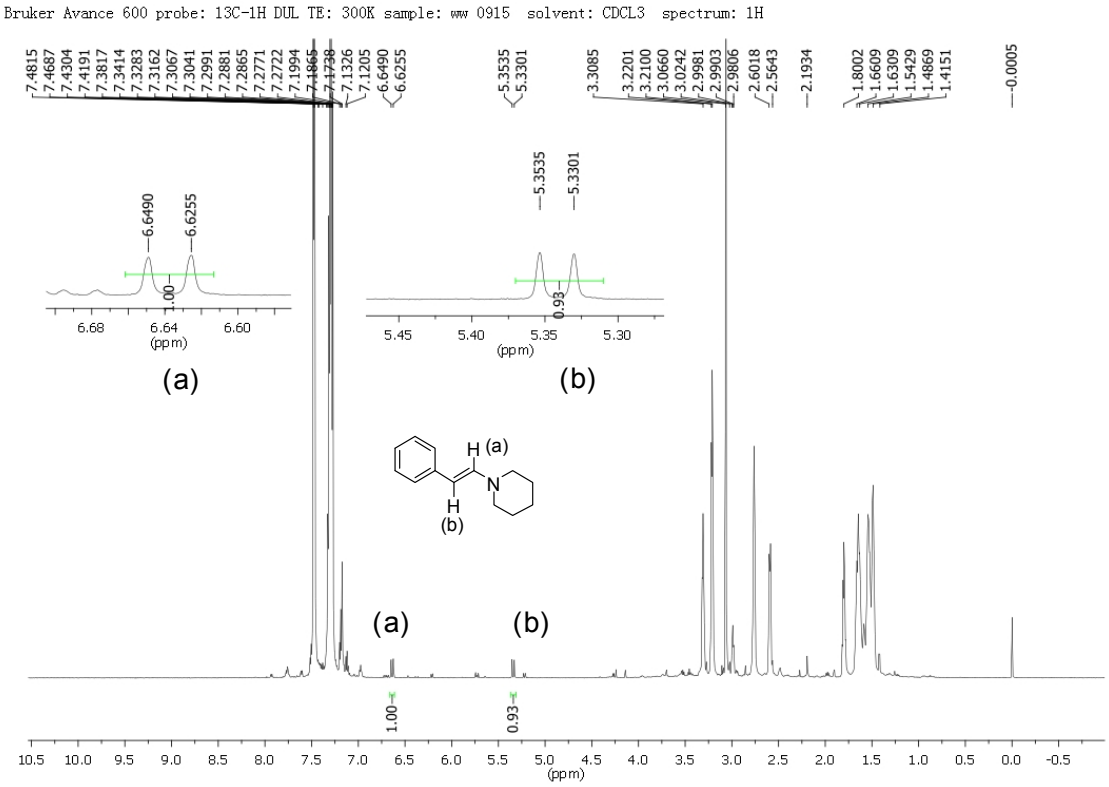

Phenylacetylene (2.5 mmol) was added to a mixture of CuBr (5 mol %), DBU (0.6 mmol), and $NaHCO_3$ (1 mmol) in a solution of THF (1 mL) and $H_2O$ (0.5 mL) at room temperature under $O_2$ (balloon). The reaction mixture was stirred at 55 °C for 12 h, and then the resulting mixture was quenched by saturated aqueous $Na_2S_2O_3$ solution (1 mL), acidified with dilute HCl solution, and extracted with diethyl ether (3 × 10 mL). The solution was concentrated in vacuum, no phenylacetic acid product was detected by TLC and MS.

## 6. Reaction of Phenylacetylene with Piperidine in the absence of Dioxygen




Phenylacetylene (4 mmol) was added to a mixture of piperidine (1 mmol), DBU (1.2 mmol), and CuBr (5 mol %) at room temperature under $N_2$. The reaction mixture was stirred at 65 °C for 2 h, and then the solution was quickly transferred to the NMR tube, and made $^1$H NMR ($CDCl_3$) monitoring experiment immediately, thus the enamine intermediate **10aa** was detected[1-3].

**Copy of $^1$H NMR Spectra for the monitoring experiment of enamine intermediate**




**Reference:**

(1) A. Tillack, H. Trauthwein, C. G. Hartung, M. Eichberger, A. Jansen and M. Bellar, *Monatsh. Chemie,* 2000, **131**, 1327-1334.

(2) C. R. V. Reddy, S. Urgaonkar and J. G. Verkade, *Org. lett.* 2005, **7**, 4427-4430.

(3) K. Sakai, T. Kochi and F. Kakiuchi. *Org. lett.* 2011, **13**, 3928-3931.

## 7. Control experiment of the isolated copper(I) acetylide with piperidine

Piperidine (1 mmol) was added to a mixture of phenylacetylene (1 mmol) and CuBr (1 mmol) in THF (0.6 mL) at room temperature under $N_2$. The reaction mixture was stirred at 55 °C for 20 mins. Then the resulting mixture was quickly filtered and washed by diethyl ether and n-hexane. The bright yellow solid was vacuum-dried, and 104 mg (64% yield) of copper(I) acetylide was obtained. Copper(I) acetylide **4aa** was determined by IR spectroscopy.[1-3] IR (KBr) ν/cm$^{-1}$: (C≡C) 1929 (w), 1584 (m), 1481 (s), 1440 (s), 745 (s), 682 (s), 549 (m).

When the reaction of isolated copper(I) acetylide **4aa** (0.25 mmol) with piperidine **2a** (0.5 mmol) was conducted at 55 °C in the presence of DBU (0.3 mmol) under $O_2$, the yellow solid copper(I) acetylide **4aa** was dissolved and the desired product **3aa** was obtained in 11% yield (The low yield of amide might be caused by the side reaction of oxidative dimerization of copper(I) acetylide). This result indicated that copper(I) acetylide might be a key intermediate in the present reaction system and supported the proposed mechansim.

Furthermore, the reaction of copper(I) acetylide **4aa** (0.25 mmol) with piperidine **2a** (0.25 mmol) was performed at 55 °C in the presence of DBU (0.3 mmol) and $N_2$. The yellow solid copper(I) acetylide **4aa** was completely dissolved. Unfortunately, no useful information for *α*-aminovinylcopper(I) complex **5aa** was obtained by NMR and HRMS analysis of reaction mixture, which might be caused by the high instability of intermediate **5aa**.

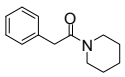
**Reference:**

(1) I. A. Garbusova, V. T. Alexanjan, L. A. Leites, I. R.Golding and A.M. Sladkov*, J. Organomet. Chem*., 1973, **54**, 341-344.

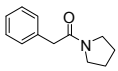
(2) W. M. Khairul, M. A. Fox, N. N. Zaitseva, M. Gaudio, D. S. Yufit, B. W. Skelton, A. H. White, J. A. K. Howard, M. I. Bruce and P. J. Low, *Dalton Trans*., 2009, 610–620.

(3) C. Shao, G. Cheng, D, Su,J. Xu, X. Wang and Y. Hua, *Adv. Synth. Catal*. 2010, **352**, 1587–1592.

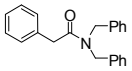
## 8. Characterization Data of Amides 3aa-3ma

**2-Phenyl-1-(piperidin-1-yl)ethanone (3aa)** 

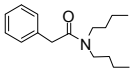
Compound **3aa** was obtained in 68% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$ = 0.38; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): *δ* 7.31 (t, *J* = 7.7 Hz, 2H), 7.26-7.22 (m, 3H), 3.73 (s, 2H), 3.57 (br.t, *J* = 5.3 Hz, 2H), 3.37 (br.t, *J* = 5.4 Hz, 2H), 1.58-1.56 (m, 2H), 1.52-1.51 (m, 2H), 1.35-1.34 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): *δ* 169.3, 135.4, 128.6, 128.5, 126.6, 47.3, 42.9, 41.2, 26.2, 25.5, 24.4; HRMS calc. for $C_{13}H_{17}NONa$ $(M+Na)^+$, 226.1202; found, 226.1210.

**2-Phenyl-1-(pyrrolidin-1-yl)ethanone (3ab)** 

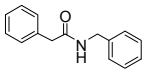
Compound **3ab** was obtained in 65% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 1:1 v/v): $R_f$= 0.22; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.32-7.27 (m, 4H), 7.25-7.22 (m, 1H), 3.66 (s, 2H), 3.49 (t, $J$ = 6.9 Hz, 2H), 3.42 (t, $J$ = 6.8 Hz, 2H), 1.94-1.89 (m, 2H), 1.86-1.81 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.5, 134.9, 128.9, 128.6, 126.7, 46.9, 45.9, 42.3, 26.1, 24.3; HRMS calc. for $C_{12}H_{15}NONa$ $(M+Na)^+$, 212.1046; found, 212.1051.

**N,N-Dibenzyl-2-phenylacetamide (3ac)** 

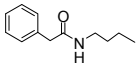
Compound **3ac** was obtained in 60% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 4:1 v/v): $R_f$= 0.53; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.35 (br.t, $J$ = 7.4 Hz, 2H), 7.32-7.23 (m, 9H), 7.19 (d, $J$ = 7.2 Hz, 2H), 7.10 (d, $J$ = 7.4 Hz, 2H), 4.62 (s, 2H), 4.43 (s, 2H), 3.79 (s, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 171.6, 137.3, 136.4, 135.0, 129.0, 128.9, 128.7, 128.6, 128.3, 127.7, 127.4, 126.9, 126.5, 50.3, 48.3, 41.0; HRMS calc. for $C_{22}H_{21}NONa$ $(M+Na)^+$, 338.1515; found, 338.1519.

**N,N-Dibutyl-2-phenylacetamide (3ad)** 

Compound **3ad** was obtained in 50% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 5:1 v/v): $R_f$= 0.28; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.30 (br.t, $J$ = 7.5 Hz, 2H), 7.25-7.21 (m, 3H), 3.69 (s, 2H), 3.32 (t, $J$ = 7.6 Hz, 2H), 3.20 (t, $J$ = 7.9 Hz, 2H), 1.54-1.44 (m, 4H), 1.32-1.24 (m, 4H); 0.92-0.89 (m, 6H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 170.4, 135.6, 128.7, 128.6, 126.6, 48.1, 45.7, 41.0, 31.1, 29.7, 20.2, 20.1, 13.8, 13.7; HRMS calc. for $C_{16}H_{25}NONa$ $(M+Na)^+$, 270.1828; found, 270.1834.

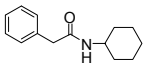
**N-Benzyl-2-phenylacetamide (3ae)** 

Compound **3ae** was obtained in 55% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 3:1 v/v): $R_f$= 0.21; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.34 (br.t, $J$ = 7.3 Hz, 2H), 7.30-7.23 (m, 6H), 7.17 (br.d, $J$ = 7.5 Hz, 2H), 5.72 (br.s, 1H), 4.41 (d, $J$ = 5.9 Hz, 2H), 3.62 (s, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 170.9, 138.2, 134.8, 129.4, 129.1, 128.7, 127.5, 127.4, 127.4, 43.8, 43.6; HRMS calc. for $C_{15}H_{15}NONa$ $(M+Na)^+$, 248.1046; found, 248.1043.

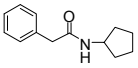
**N-Butyl-2-phenylacetamide (3af)** 

Compound **3af** was obtained in 53% yield according to the general procedure (4 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.25; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.36 (br.t, $J$ = 7.4 Hz, 2H), 7.30 (br.t, $J$ = 7.2 Hz, 1H), 7.25 (br.d, $J$ = 8.3 Hz, 2H), 5.32 (br.s, 1H), 3.57(s, 2H), 3.22-3.19 (m, 2H), 1.41-1.37 (m, 2H), 1.28-1.22 (m, 2H), 0.87 (t, $J$ = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 170.9,

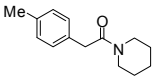
135.1, 129.4, 129.0, 127.3, 43.9, 39.4, 31.5, 20.0, 13.7; HRMS calc. for $C_{12}H_{17}NONa$ $(M+Na)^+$, 214.1202; found, 214.1208.

**N-Cyclohexyl-2-phenylacetamide (3ag)** 

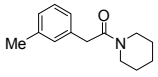
Compound **3ag** was obtained in 63% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 3:1 v/v): $R_f$= 0.32; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.35 (br.t, $J$ = 7.4 Hz, 2H), 7.28 (br.t, $J$ = 7.4 Hz, 1H), 7.25 (br.d, $J$ = 7.2 Hz, 2H), 5.22 (br.s, 1H), 3.79-3.73 (m, 1H), 3.54 (s, 2H), 1.84-1.82 (m, 2H), 1.62-1.54 (m, 3H), 1.36-1.29 (m, 2H), 1.14-1.07 (m, 1H), 1.04-0.98 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 170.0, 135.2, 129.4, 128.9, 127.2, 48.2, 44.0, 32.9, 25.5, 24.7; HRMS calc. for $C_{14}H_{19}NONa$ $(M+Na)^+$, 240.1359; found, 240.1358.

**N-Cyclopentyl-2-phenylacetamide (3ah)** 

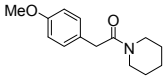
Compound **3ah** was obtained in 60% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 3:1 v/v): $R_f$= 0.30; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.35 (br.t, $J$ = 7.5 Hz, 2H), 7.28 (br.t, $J$ = 7.3 Hz, 1H), 7.24 (br.d, $J$ = 7.4 Hz, 2H), 5.29 (br.s, 1H), 4.21-4.15 (m, 1H), 3.54 (s, 2H), 1.95-1.90 (m, 2H), 1.58-1.52 (m, 4H), 1.27-1.23 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 170.5, 135.2, 129.3, 128.9, 127.2, 51.3, 43.9, 33.0, 23.6; HRMS calc. for $C_{13}H_{17}NONa$ $(M+Na)^+$, 226.1202; found, 226.1208.

**1-(Piperidin-1-yl)-2-*p*-tolylethanone (3ba)** 

Compound **3ba** was obtained in 63% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.32; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.14-7.10 (m, 4H), 3.68 (s, 2H), 3.56 (br.t, $J$ = 5.2 Hz, 2H), 3.36 (br.t, $J$ = 5.4 Hz, 2H), 2.32 (s, 3H), 1.58-1.55 (m, 2H), 1.52-1.51 (m, 2H), 1.36-1.35 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.5, 136.1, 132.3, 129.3, 128.4, 47.2, 42.9, 40.8, 26.2, 25.5, 24.4, 21.0; HRMS calc. for $C_{14}H_{19}NONa$ $(M+Na)^+$, 240.1359; found, 240.1363.

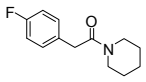
**1-(Piperidin-1-yl)-2-*m*-tolylethanone (3ca)** 

Compound **3ca** was obtained in 61% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.36; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.19 (br.t, $J$ = 7.6 Hz, 1H), 7.08 (s, 1H), 7.03 (br.d, $J$ = 7.4 Hz, 2H), 3.69 (s, 2H), 3.57 (br.t, $J$ = 5.5 Hz, 2H), 3.37 (br.t, $J$ = 5.5 Hz, 2H), 2.33 (s, 3H), 1.60-1.56 (m, 2H), 1.54-1.50 (m, 2H), 1.38-1.34 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.4, 138.3, 135.3, 129.3, 128.5, 127.4, 125.6, 47.3, 42.9, 41.1, 26.2, 25.5, 24.5, 21.4; HRMS calc. for $C_{14}H_{19}NONa$ $(M+Na)^+$, 240.1359; found, 240.1363.

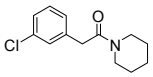
**2-(4-Methoxyphenyl)-1-(piperidin-1-yl)ethanone (3da)** 

Compound **3da** was obtained in 40% yield according to the general procedure (12 h). TLC (*n*-hexane :

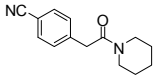
EtOAc, 2:1 v/v): $R_f$= 0.24; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.16 (d, $J$ = 8.6 Hz, 2H), 6.85 (d, $J$ = 8.6 Hz, 2H), 3.79 (s, 3H), 3.66 (s, 2H), 3.56 (br.t, $J$ = 5.3 Hz, 2H), 3.37 (br.t, $J$ = 5.5 Hz, 2H), 1.60-1.56 (m, 2H), 1.53-1.51 (m, 2H), 1.38-1.34 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.6, 158.3, 129.6, 127.5, 114.1, 55.3, 47.2, 42.9, 40.2, 26.2, 25.5, 24.4; HRMS calc. for $C_{14}H_{19}NO_2Na$ $(M+Na)^+$, 256.1308; found, 256.1304.

**2-(4-Fluorophenyl)-1-(piperidin-1-yl)ethanone (3ea)** 

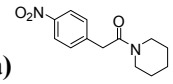
Compound **3ea** was obtained in 57% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.38; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.22-7.20 (m, 2H), 7.00 (br.t, $J$ = 8.6 Hz, 2H), 3.68 (s, 2H), 3.56 (br.t, $J$ = 5.4 Hz, 2H), 3.37 (br.t, $J$ = 5.4 Hz, 2H), 1.61-1.58 (m, 2H), 1.54-1.50 (m, 2H), 1.40-1.37 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.0, 162.5, 160.9, 131.1, 130.2, 130.1, 115.5, 115.4, 47.2, 42.9, 40.1, 26.3, 25.5, 24.4; HRMS calc. for $C_{13}H_{16}FNONa$ $(M+Na)^+$, 244.1108; found, 244.1131.

**2-(3-chlorophenyl)-1-(piperidin-1-yl)ethanone (3fa)** 

Compound **3fa** was obtained in 64% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.52; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.25-7.21 (m, 3H), 7.14 (d, $J$ = 7.3 Hz, 1H), 3.69 (s, 2H), 3.57 (t, $J$ = 5.5 Hz, 2H), 3.37 (t, $J$ = 5.5 Hz, 2H), 1.61-1.58 (m, 2H), 1.54-1.51 (m, 2H), 1.43-1.39 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 168.2, 137.8, 134.3, 129.8, 128.8, 126.9, 47.2, 43.0, 40.5, 26.3, 25.5, 24.4; HRMS calc. for $C_{13}H_{16}ClNONa$ $(M+Na)^+$, 260.0813; found, 260.0818.

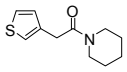
**2-(4-cyanophenyl)-1-(piperidin-1-yl)ethanone (3ga)** 

Compound **3ga** was obtained in 46% yield according to the general procedure (24 h). TLC (*n*-hexane : EtOAc, 1:1 v/v): $R_f$= 0.40; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.60 (d, $J$ = 8.2 Hz, 2H), 7.36 (d, $J$ = 8.2 Hz, 2H), 3.76 (s, 2H), 3.57 (t, $J$ = 5.5 Hz, 2H), 3.37 (t, $J$ = 5.5 Hz, 2H), 1.63-1.59 (m, 2H), 1.55-1.51 (m, 2H), 1.45-1.41 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 167.8, 141.0, 132.3, 129.8, 118.8, 110.7, 47.2, 43.0, 40.7, 26.3, 25.4, 24.3; HRMS calc. for $C_{14}H_{17}N_2O$ $(M+H)^+$, 229.1341; found, 229.1337.

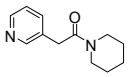
**2-(4-nitrophenyl)-1-(piperidin-1-yl)ethanone (3ha)** 

Compound **3ha** was obtained in 25% yield according to the general procedure (24 h). TLC (*n*-hexane : EtOAc, 1:1 v/v): $R_f$= 0.32; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 8.18 (d, $J$ = 8.6 Hz, 2H), 7.42 (d, $J$ = 8.5 Hz, 2H), 3.80 (s, 2H), 3.57 (t, $J$ = 5.5 Hz, 2H), 3.40 (t, $J$ = 5.5 Hz, 2H), 1.63-1.61 (m, 2H), 1.56-1.52 (m, 2H), 1.47-1.42 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 167.7, 146.9, 143.0,

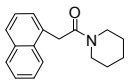
129.9, 123.8, 47.2, 43.1, 40.5, 26.4, 25.4, 24.3; HRMS calc. for $C_{13}H_{17}N_2O_3$ $(M+H)^+$, 249.1239; found, 249.1244.

**1-(piperidin-1-yl)-2-(thiophen-3-yl)ethanone( 3ia)** 

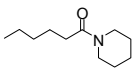
Compound **3ia** was obtained in 61% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.46; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.28-7.27 (m, 1H), 7.06 (t, $J$ =1.2 Hz, 1H), 7.01 (d, $J$ = 4.9 Hz, 1H), 3.72 (s, 2H), 3.57 (br.t, $J$ = 5.5 Hz, 2H), 3.38 (br.t, $J$ = 5.5 Hz, 2H), 1.61-1.58 (m, 2H), 1.54-1.51 (m, 2H), 1.41-1.37 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.2, 134.7, 127.5, 125.0, 121.3, 47.3, 42.7, 35.8, 25.9, 25.0, 24.4; HRMS calc. for $C_{11}H_{15}NSONa$ $(M+Na)^+$, 232.0767; found, 232.0775.

**1-(piperidin-1-yl)-2-(pyridin-3-yl)ethanone (3ja)** 

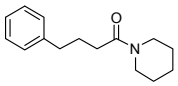
Compound **3ja** was obtained in 60% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 1:1 v/v): $R_f$= 0.20; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 8.49 (br.t, $J$ = 5.1 Hz, 2H), 7.64 (d, $J$ = 7.8 Hz, 1H), 7.27-7.25 (m, 1H), 3.71 (s, 2H), 3.57 (br.t, $J$ = 5.5 Hz, 2H), 3.42 (br.t, $J$ = 5.5 Hz, 2H), 1.64-1.60 (m, 2H), 1.56-1.52 (m, 2H), 1.47-1.43 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 168.2, 150.0, 148.2, 136.5, 130.7, 122.8, 47.2, 43.0, 37.8, 26.4, 25.4, 24.4; HRMS calc. for $C_{12}H_{16}N_2ONa$ $(M+Na)^+$, 227.1155; found, 227.1166.

**2-(naphthalen-1-yl)-1-(piperidin-1-yl)ethanone (3ka)** 

Compound **3ka** was obtained in 64% yield according to the general procedure (12 h). TLC (*n*-hexane : EtOAc, 2:1 v/v): $R_f$= 0.46; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.99 (d, $J$ = 8.4 Hz, 1H), 7.86 (d, $J$ = 8.0 Hz, 1H), 7.76 (d, $J$ = 8.2 Hz, 1H), 7.51 (t, $J$ = 7.6 Hz, 1H), 7.48 (t, $J$ = 7.4 Hz, 1H), 7.41 (t, $J$ = 7.7 Hz, 1H), 7.33 (d, $J$ = 7.0 Hz, 1H), 4.15 (s, 2H), 3.63 (br.t, $J$ = 5.2 Hz, 2H), 3.36 (br.t, $J$ = 5.4 Hz, 2H), 1.59-1.56 (m, 4H), 1.40-1.37 (m, 2H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 169.4, 128.8, 127.5, 126.3, 126.2, 125.7, 125.5, 123.5, 47.3, 43.0, 38.5, 26.3, 25.6, 24.5; HRMS calc. for $C_{17}H_{19}NONa$ $(M+Na)^+$, 276.1359; found, 276.1371.

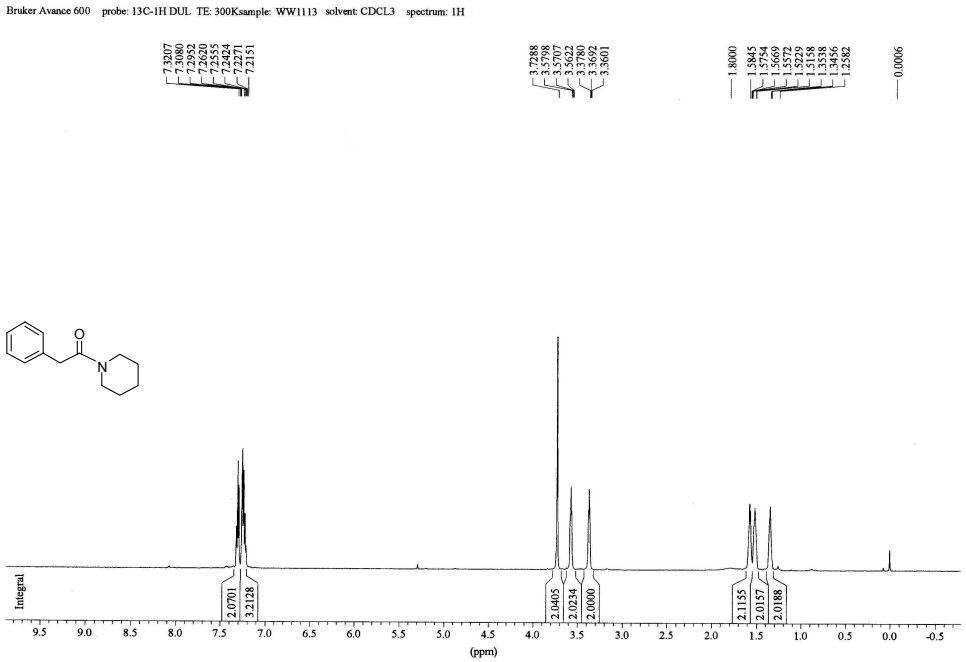
**1-(Piperidin-1-yl)hexan-1-one (3la)** 

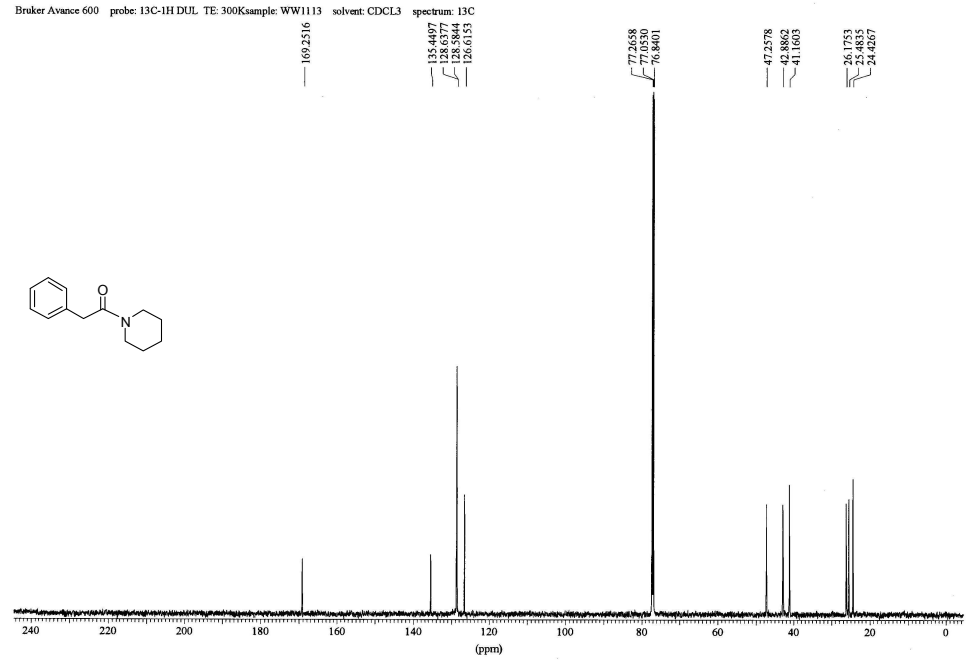
Compound **3la** was obtained in 33% yield according to the general procedure (neat conditions, 24 h). TLC (*n*-hexane : EtOAc, 3:1 v/v): $R_f$= 0.34; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 3.54 (br.t, $J$ = 5.4 Hz, 2H), 3.39 (br.t, $J$ = 5.3 Hz, 2H), 2.30 (br.t, $J$ = 7.7 Hz, 2H), 1.65-1.61 (m, 4H), 1.56-1.53 (m, 4H), 1.36-1.31 (m, 4H), 0.90 (br.t, $J$ = 6.8 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 171.5, 46.7, 42.6, 33.4, 31.7, 26.6, 25.6, 25.2, 24.6, 22.5, 13.9; HRMS calc. for $C_{11}H_{21}NONa$ $(M+Na)^+$, 206.1515; found,. 206.1534.

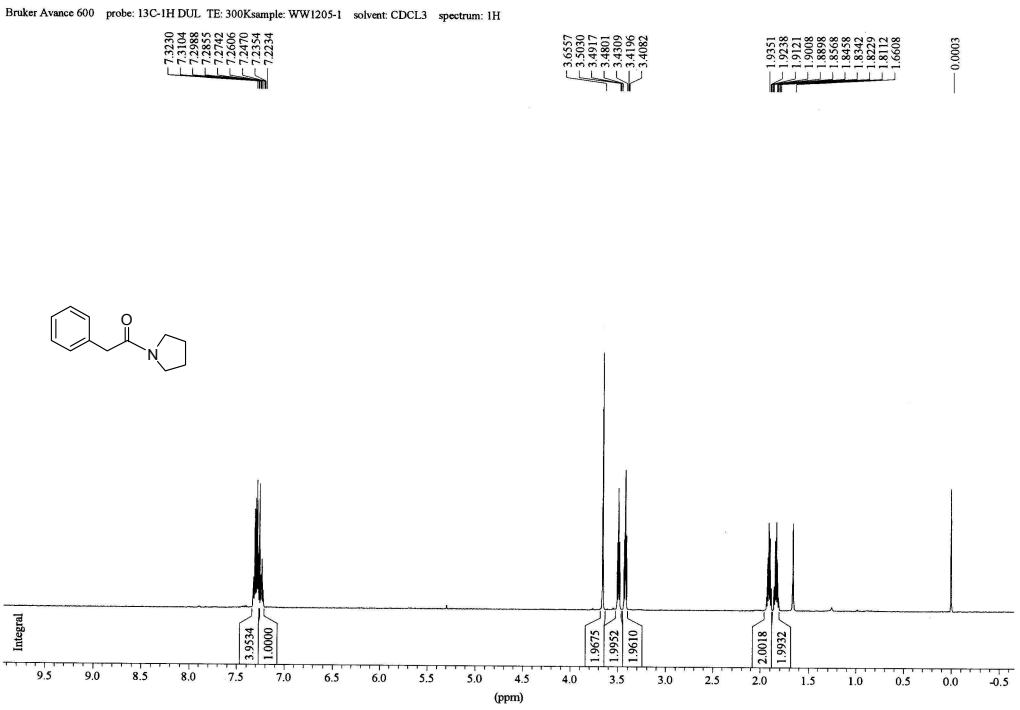
**4-Phenyl-1-(piperidin-1-yl)butan-1-one (3ma)** 

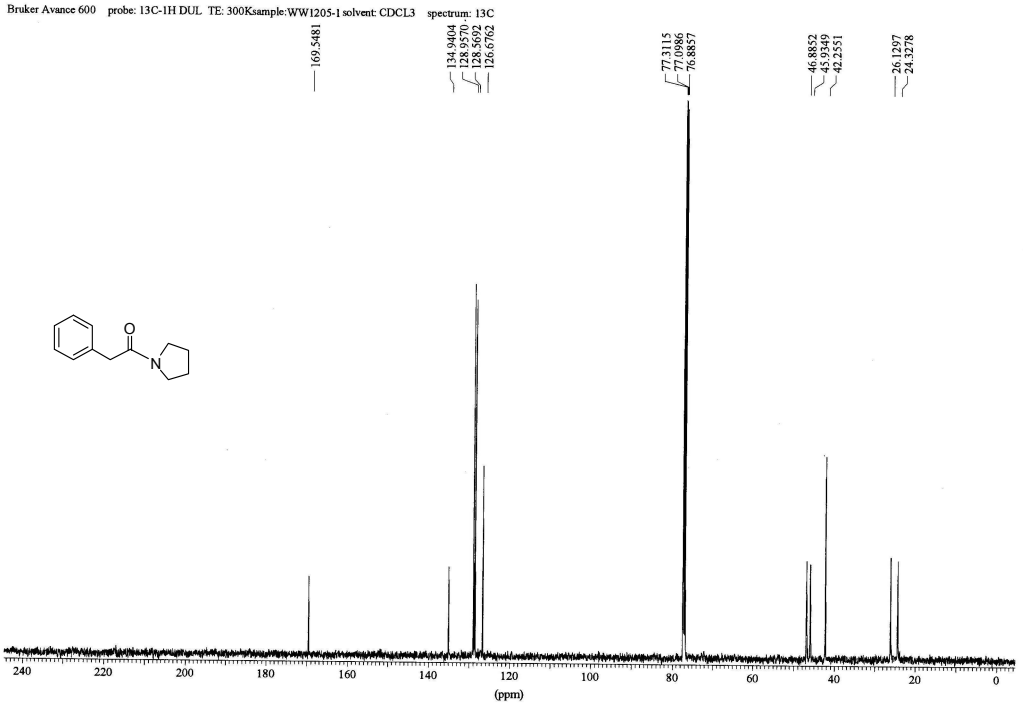
Compound **3ma** was obtained in 31% yield according to the general procedure (neat conditions, 24 h). TLC (*n*-hexane : EtOAc, 3:1 v/v): $R_f$= 0.40; $^1$H NMR ($CDCl_3$, 600 MHz, ppm): $\delta$ 7.29-7.26 (m, 2H), 7.20-7.17 (m, 3H), 3.54 (br.t, $J$ = 5.4 Hz, 2H), 3.32 (br.t, $J$ = 5.4 Hz, 2H), 2.68 (t, $J$ = 7.7 Hz, 2H), 2.32 (t, $J$ = 7.5 Hz, 2H), 1.99-1.94 (m, 2H), 1.64-1.60 (m, 2H), 1.54-1.51 (m, 4H); $^{13}$C NMR ($CDCl_3$, 150 MHz, ppm): $\delta$ 171.0, 141.8, 128.5, 128.3, 125.9, 46.6, 42.6, 35.4, 32.5, 26.8, 26.5, 25.6, 24.6; HRMS calc. for $C_{15}H_{21}NONa$ $(M+Na)^+$, 254.1515; found, 254.1519.

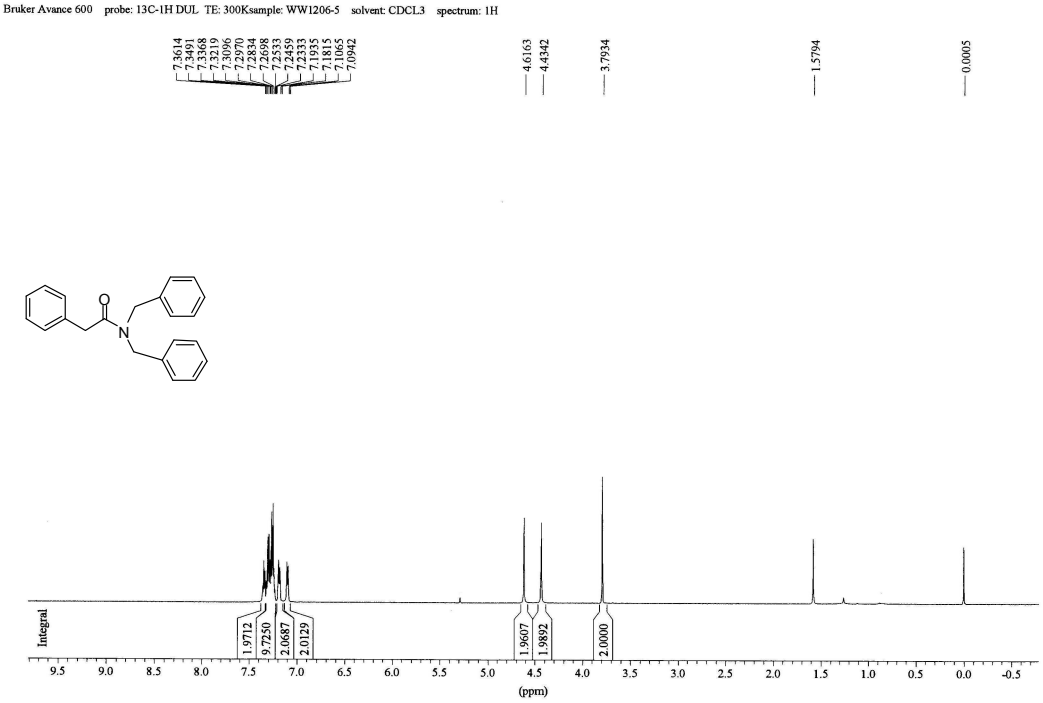
## 9. Copies of NMR Spectra for 3aa−3ma

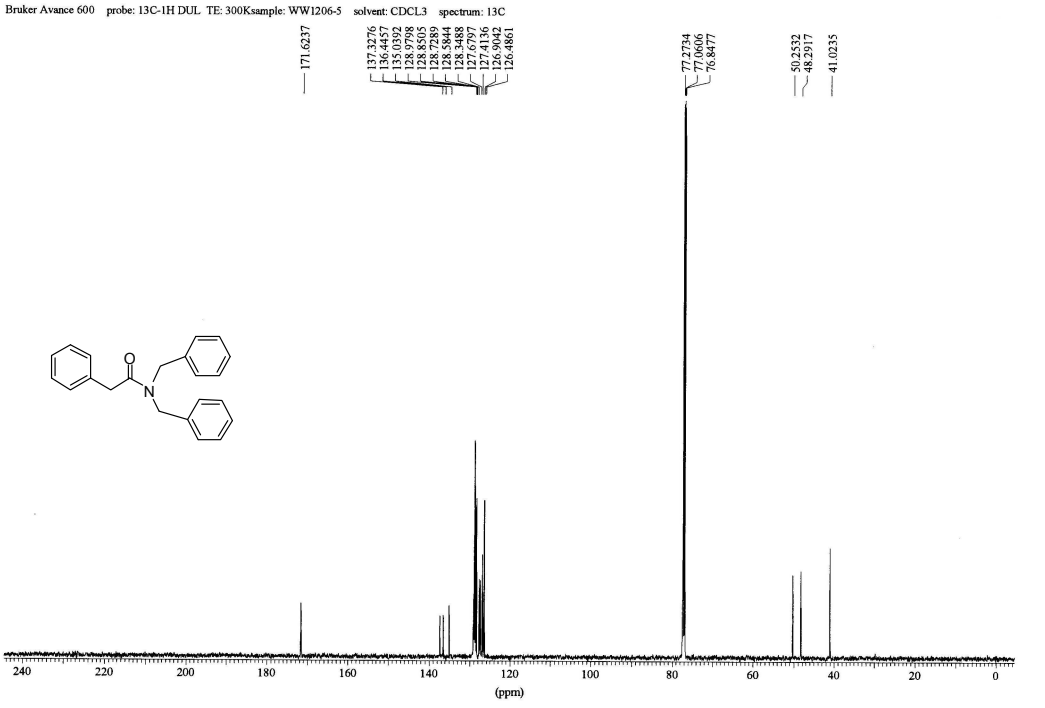


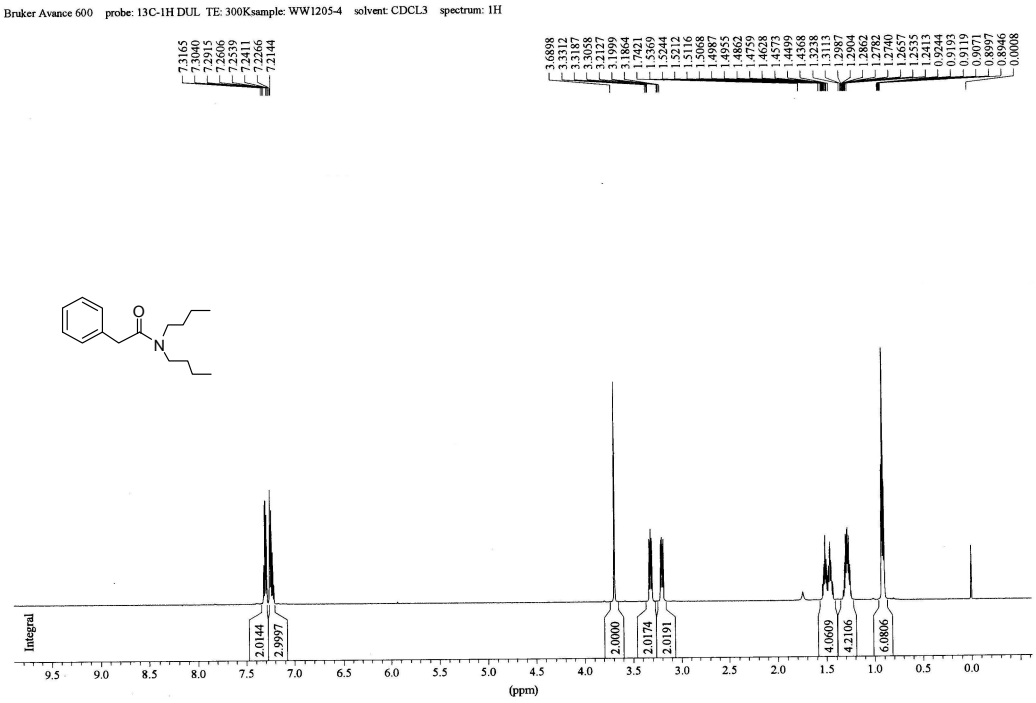




Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-1 solvent: CDCL3 spectrum: 1H
7.3230
7.3104
7.2988
7.2855
7.2742
7.2606
7.2470
7.2354
7.2234
3.6557
3.5030
3.4917
3.4801
3.4199
3.4106
3.4082
1.9351
1.9238
1.9121
1.9008
1.8898
1.8568
1.8458
1.8342
1.8229
1.8112
1.6608
0.0003
O
N
Integral
3.9534
1.0000
1.9675
1.9952
1.9610
2.0018
1.9932
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample:WW1205-1 solvent: CDCL3 spectrum: 13C
169.5481
134.9404
128.9570
128.5692
126.6762
77.3115
77.0986
76.8857
46.8852
45.9349
42.2551
26.1297
24.3278
O
N
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-5 solvent: CDCL3 spectrum: 1H
7.3614
7.3491
7.3368
7.3219
7.3096
7.2970
7.2834
7.2698
7.2533
7.2459
7.2333
7.1935
7.1815
7.1065
7.0942
4.6163
4.4342
3.7934
1.5794
0.0005
O
N
Integral
1.9712
9.7250
2.0687
2.0129
1.9607
1.9892
2.0000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-5 solvent: CDCL3 spectrum: 13C
171.6237
137.3276
136.4457
135.0392
128.9798
128.8565
128.7981
128.5844
128.3488
127.6797
127.4136
126.9042
126.4861
77.2734
77.0606
76.8477
50.2532
48.2917
41.0235
O
N
240 220 200 180 160 140 120 100 80 60 40 20 0
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-4 solvent: CDCL3 spectrum: 1H
7.3165
7.3040
7.2915
7.2606
7.2539
7.2411
7.2266
7.2144
3.6898
3.3312
3.3187
3.3058
3.2927
3.1999
3.1864
1.7421
1.5369
1.5244
1.5212
1.5116
1.5068
1.4987
1.4955
1.4862
1.4759
1.4628
1.4573
1.4499
1.4368
1.3238
1.3113
1.2987
1.2964
1.2862
1.2782
1.2740
1.2657
1.2535
1.2413
0.9244
0.9193
0.9119
0.9071
0.8997
0.8946
0.0008
Integral
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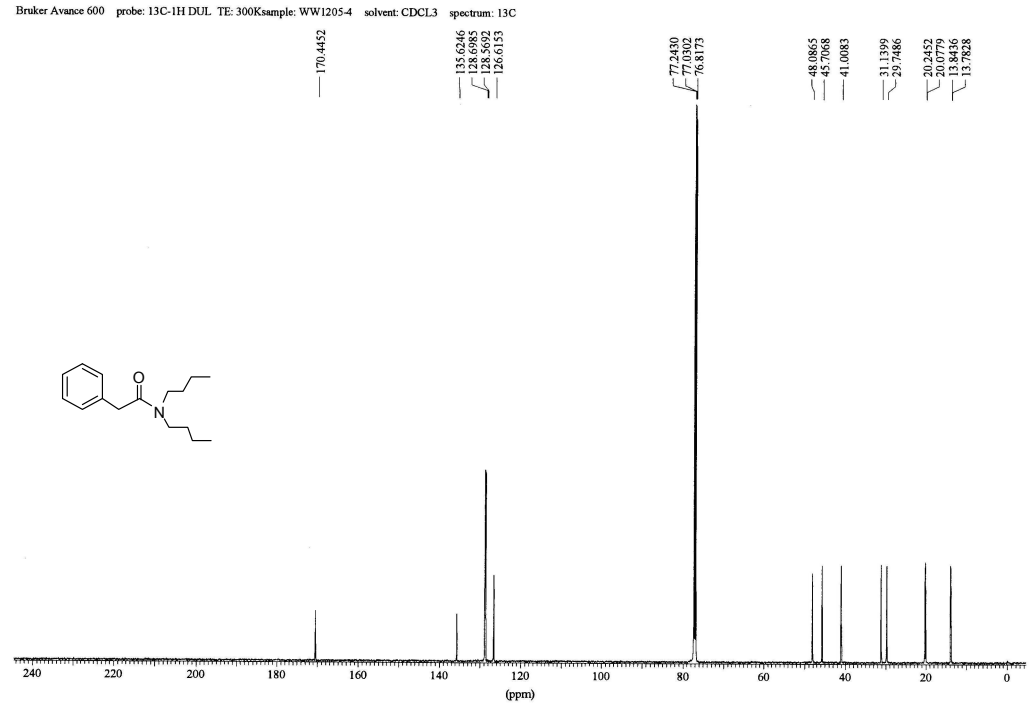
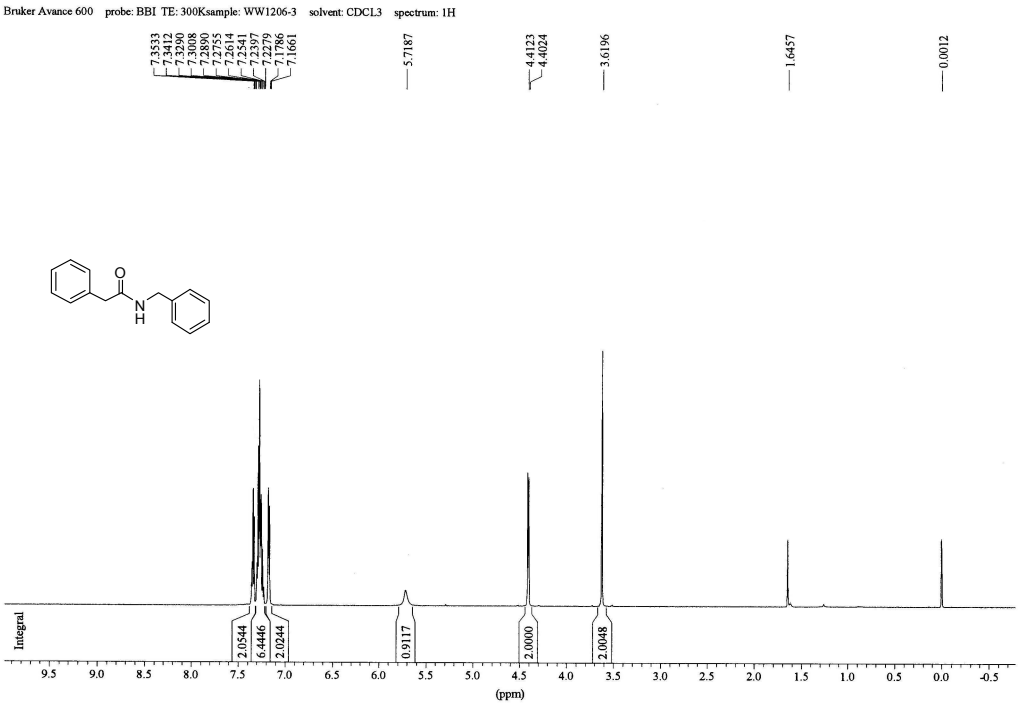
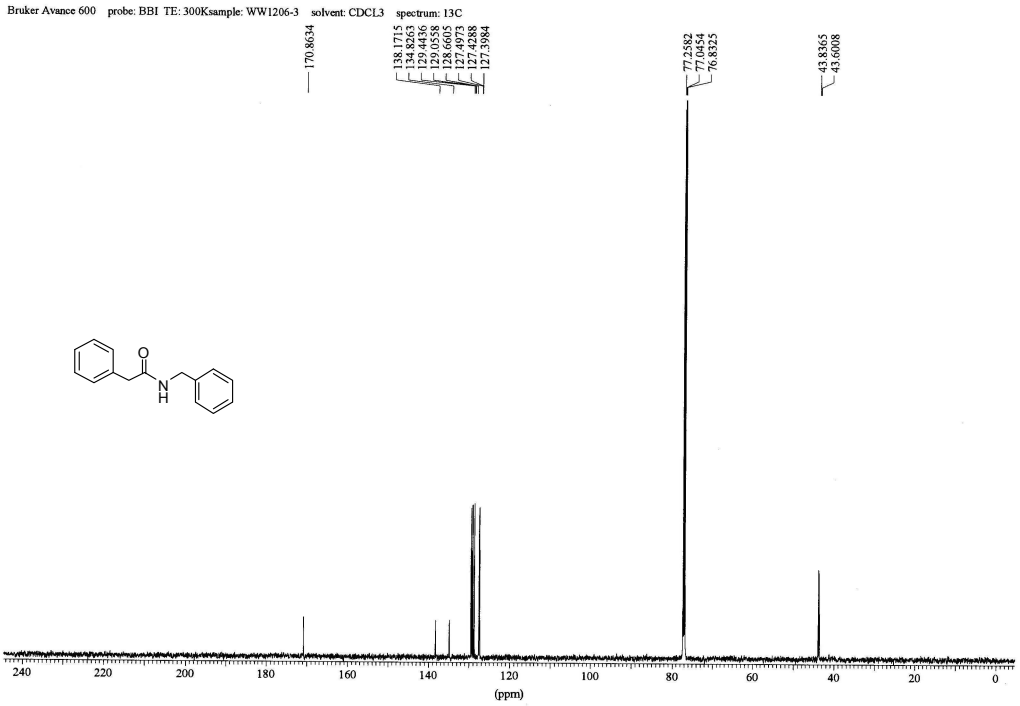
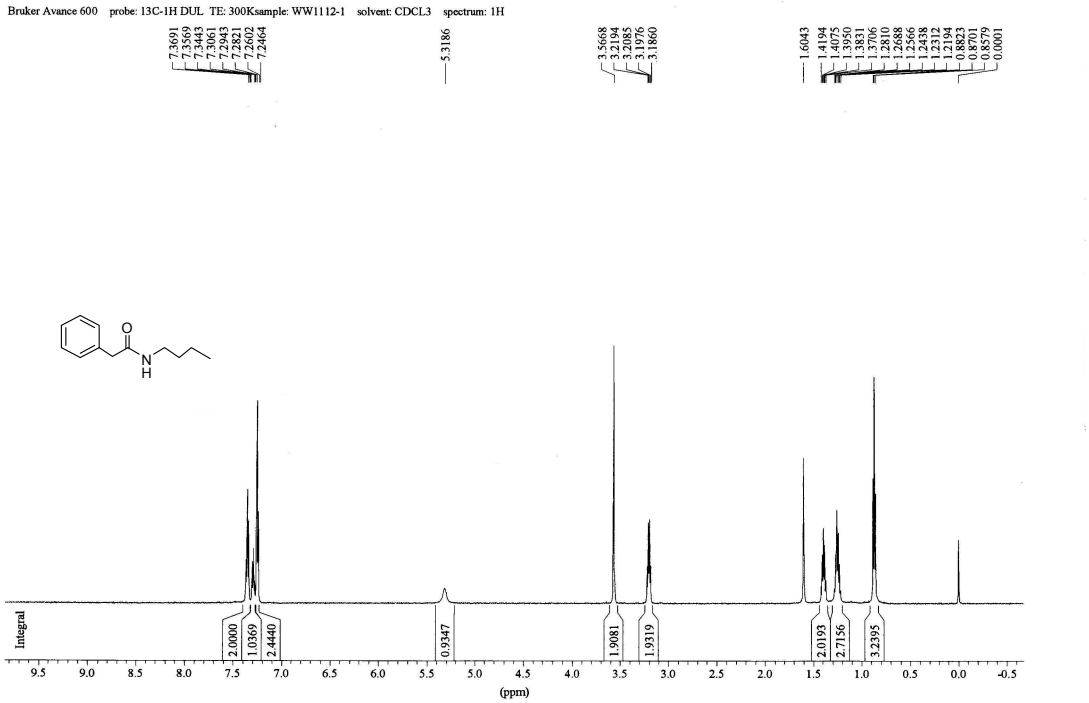
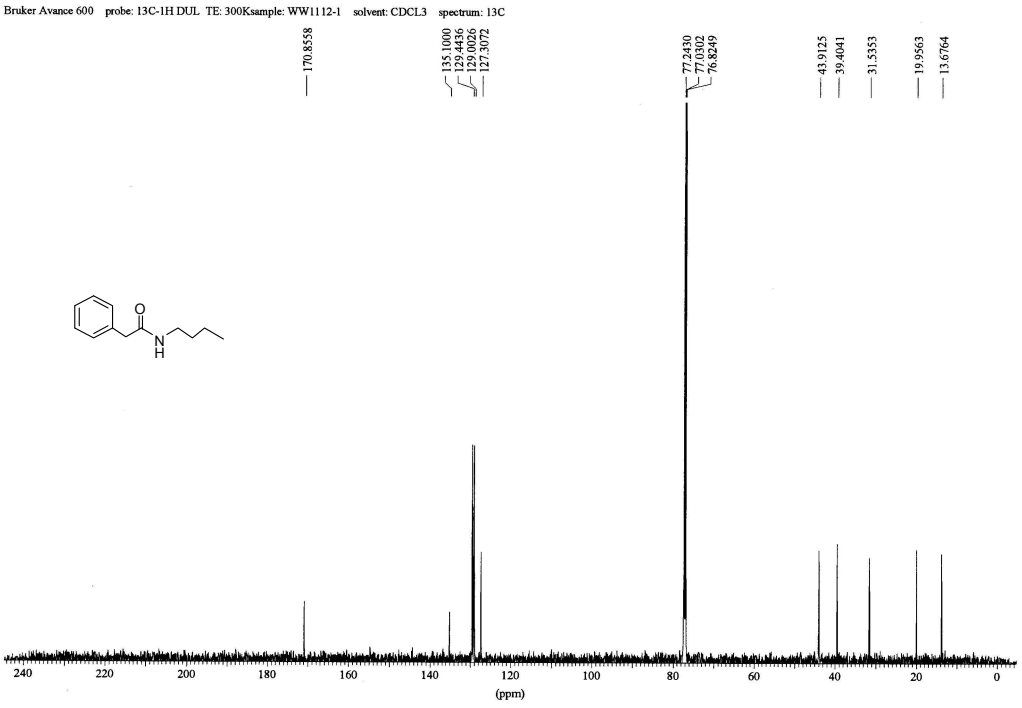
2.9997
2.0000
2.0174
2.0191
4.0609
4.2106
6.0806
(ppm)

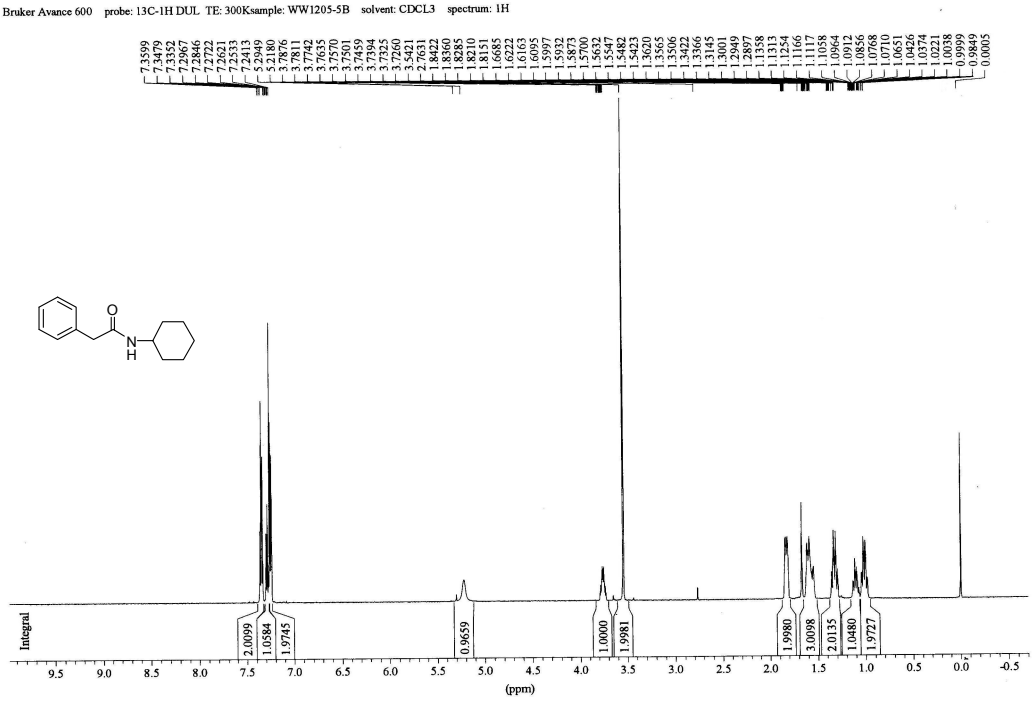

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-4 solvent: CDCL3 spectrum: 13C
170.4452
135.6246
128.6985
128.5692
126.6153
77.2430
77.0302
76.8173
48.0865
45.7068
41.0083
31.1399
29.7486
20.2452
20.0779
13.8436
13.7828
(ppm)

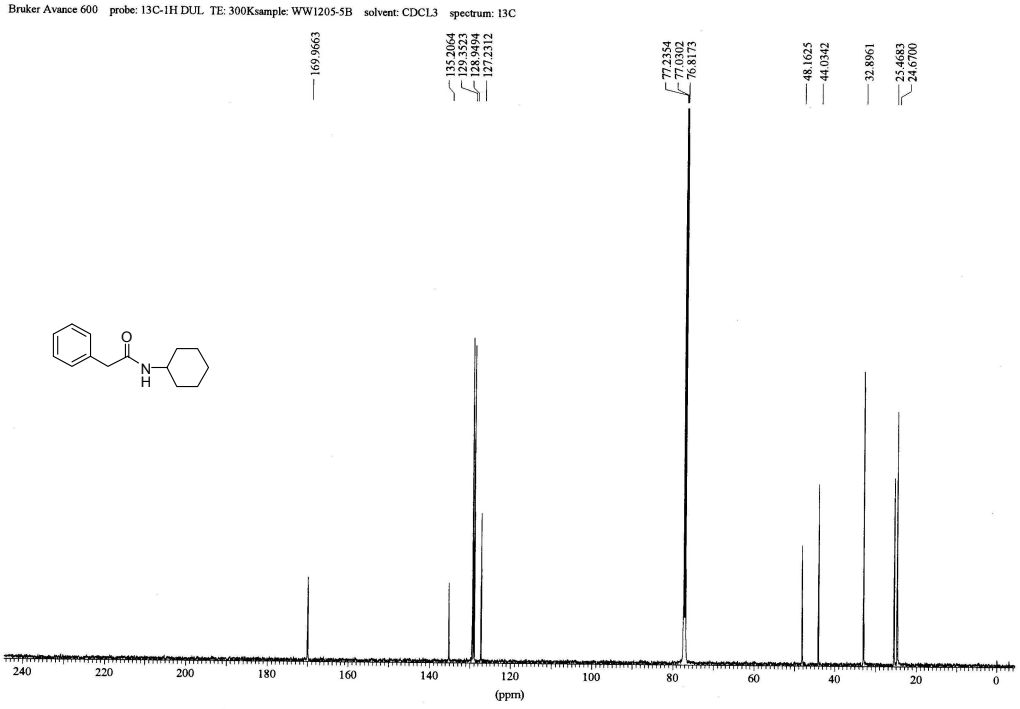

Bruker Avance 600 probe: BBI TE: 300Ksample: WW1206-3 solvent: CDCL3 spectrum: 1H
7.3533
7.3412
7.3290
7.3008
7.2890
7.2755
7.2614
7.2541
7.2397
7.2279
7.1796
7.1661
5.7187
4.4123
4.4024
3.6196
1.6457
0.0012
O
N
H
Integral
2.0544
6.4446
2.0244
0.9117
2.0000
2.0048
(ppm)

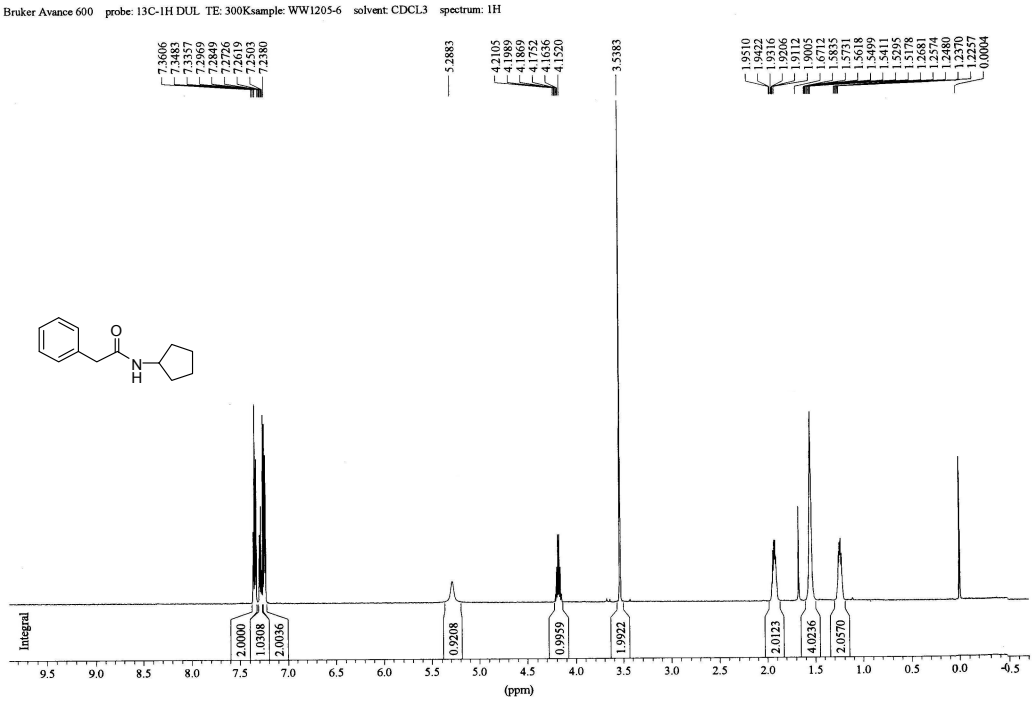

Bruker Avance 600 probe: BBI TE: 300Ksample: WW1206-3 solvent: CDCL3 spectrum: 13C
170.8634
138.1715
134.8263
129.4456
129.0158
128.6605
127.4973
127.4288
127.3984
77.2582
77.0454
76.8325
43.8365
43.6008
O
N
H
(ppm)

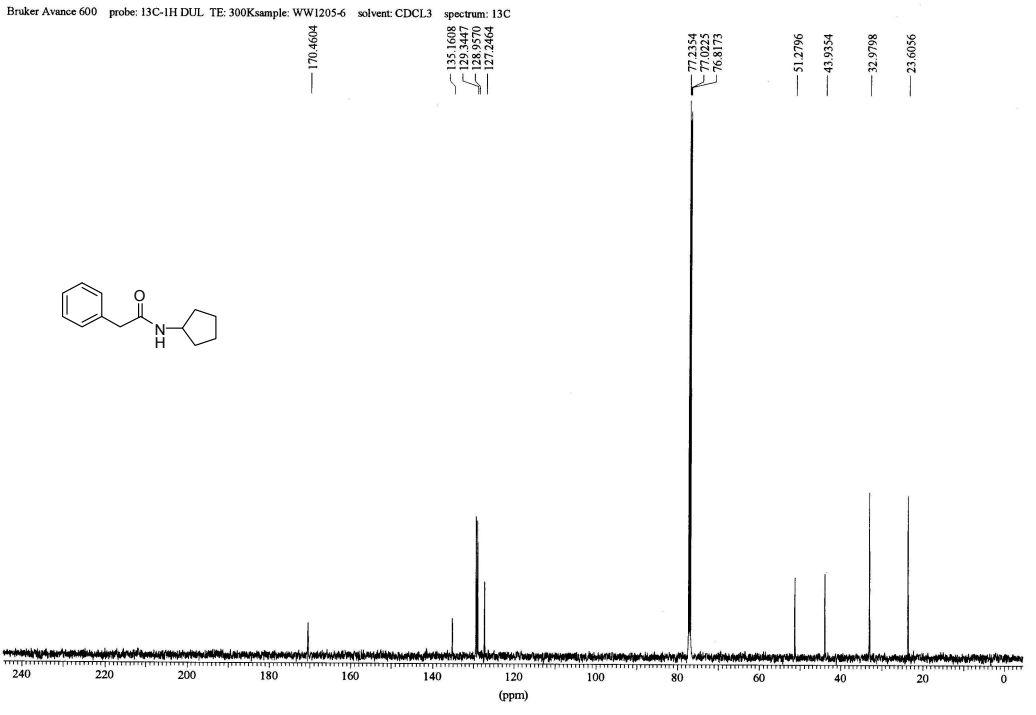

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1112-1 solvent: CDCL3 spectrum: 1H
7.3691
7.3569
7.3443
7.3061
7.2943
7.2821
7.2602
7.2464
5.3186
3.5668
3.2194
3.1985
3.1976
3.1860
1.6043
1.4194
1.4075
1.3950
1.3831
1.3706
1.2810
1.2688
1.2566
1.2438
1.2312
1.2194
0.8823
0.8701
0.8579
0.0001
Integral
2.0000
1.0369
2.4440
0.9347
1.9081
1.9319
2.0193
2.7156
3.2395
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1112-1 solvent: CDCL3 spectrum: 13C
170.8558
135.1000
129.4436
129.0026
127.3072
77.2430
77.0302
76.8249
43.9125
39.4041
31.5353
19.9563
13.6764
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-5B solvent: CDCL3 spectrum: 1H
Integral
2.0099
1.0584
1.9745
0.9659
1.0000
1.9981
1.9980
3.0098
2.0135
1.0480
1.9727
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-5B solvent: CDCL3 spectrum: 13C
169.9663
135.2064
129.3523
128.9494
127.2312
77.2354
77.0302
76.8173
48.1625
44.0342
32.8961
25.4683
24.6700
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-6 solvent: CDCL3 spectrum: 1H
7.3606
7.3483
7.3357
7.2969
7.2849
7.2726
7.2619
7.2503
7.2380
5.2883
4.2105
4.1989
4.1869
4.1752
4.1636
4.1520
3.5383
1.9510
1.9422
1.9316
1.9206
1.9113
1.9005
1.6712
1.5835
1.5731
1.5618
1.5499
1.5411
1.5295
1.5178
1.2684
1.2574
1.2480
1.2370
1.2257
0.0004
Integral
2.0000
1.0308
2.0036
0.9208
0.9959
1.9922
2.0123
4.0236
2.0570
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1205-6 solvent: CDCL3 spectrum: 13C
170.4604
135.1608
129.3447
128.9570
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77.2354
77.0225
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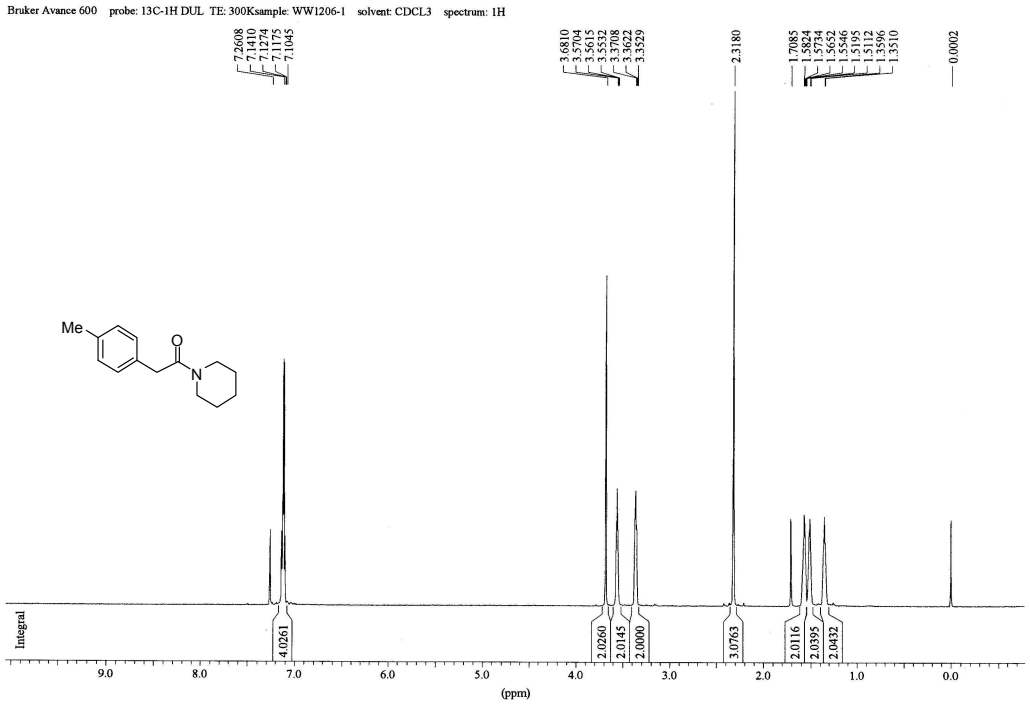
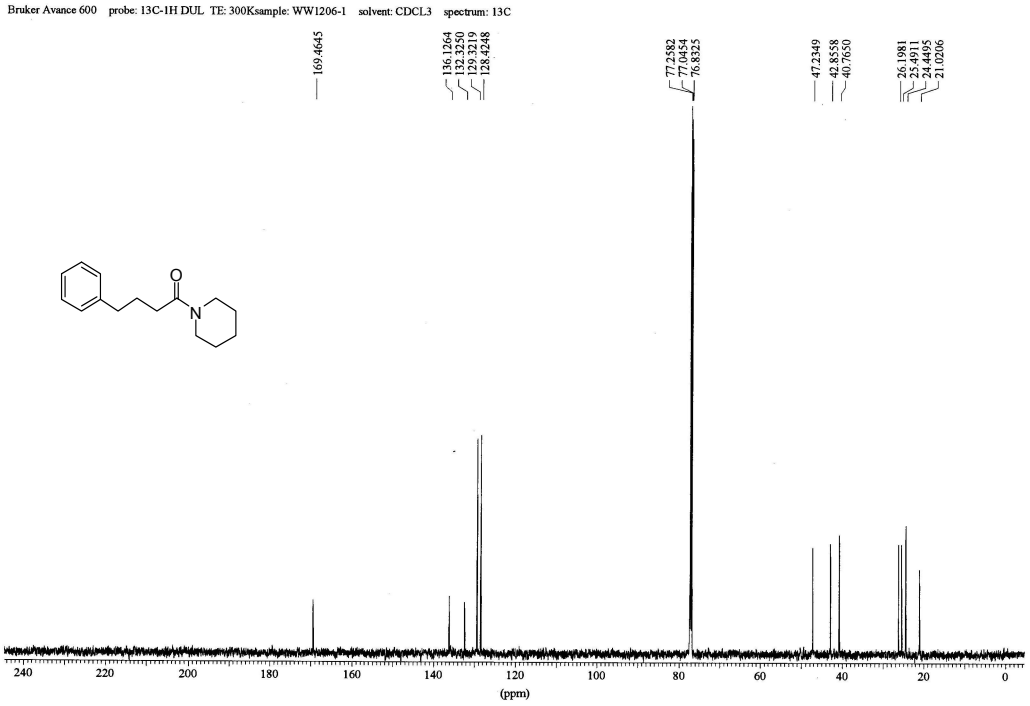
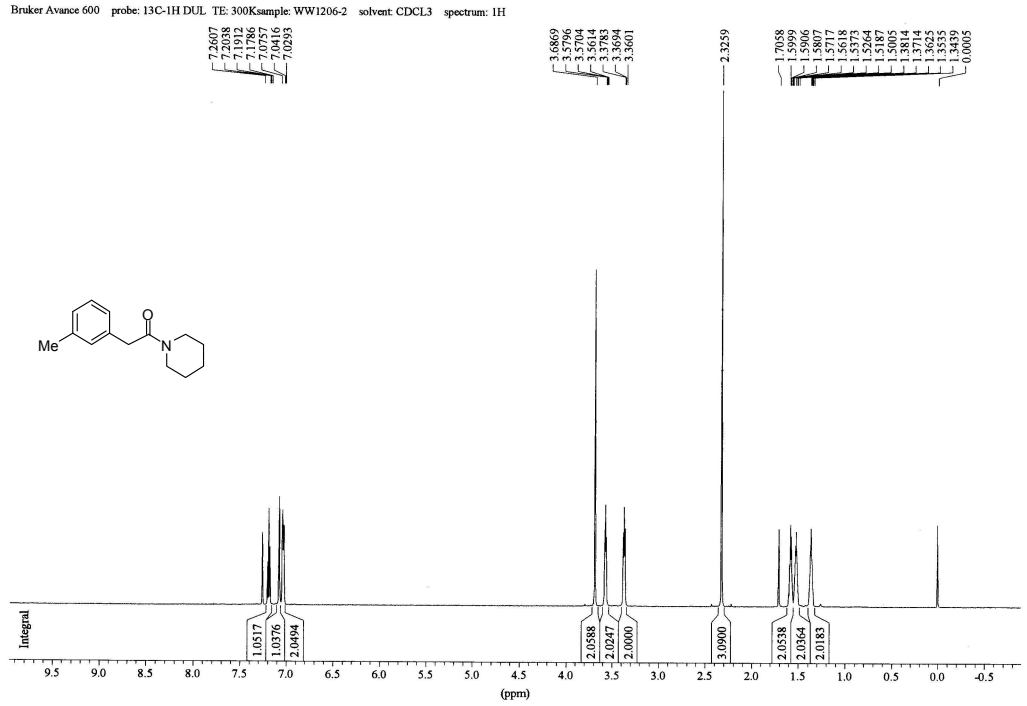
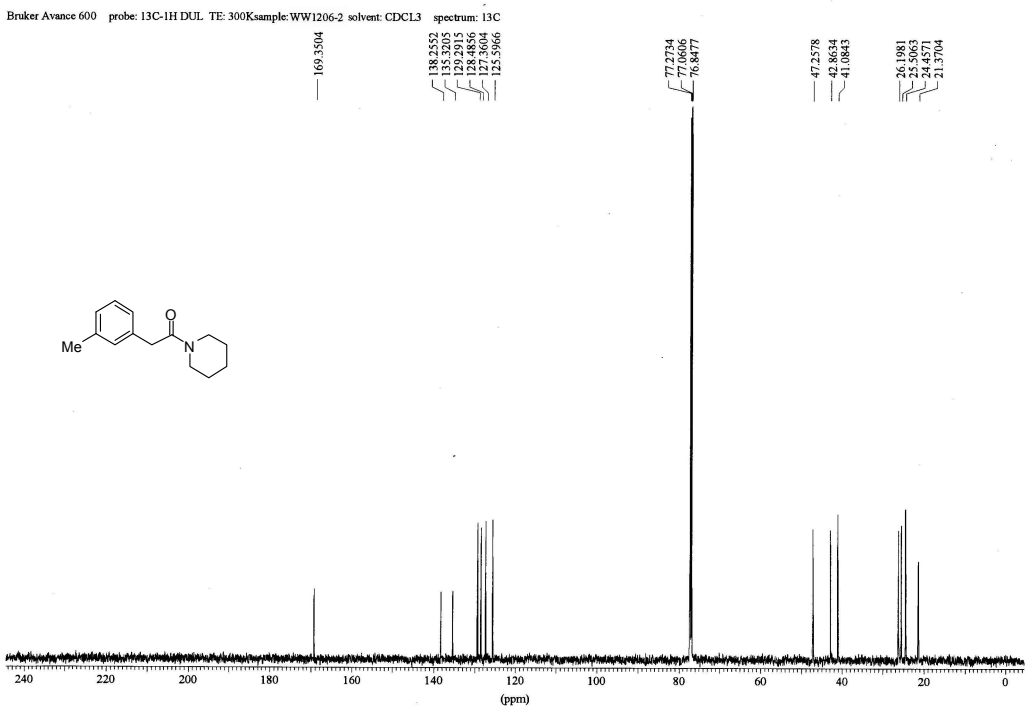
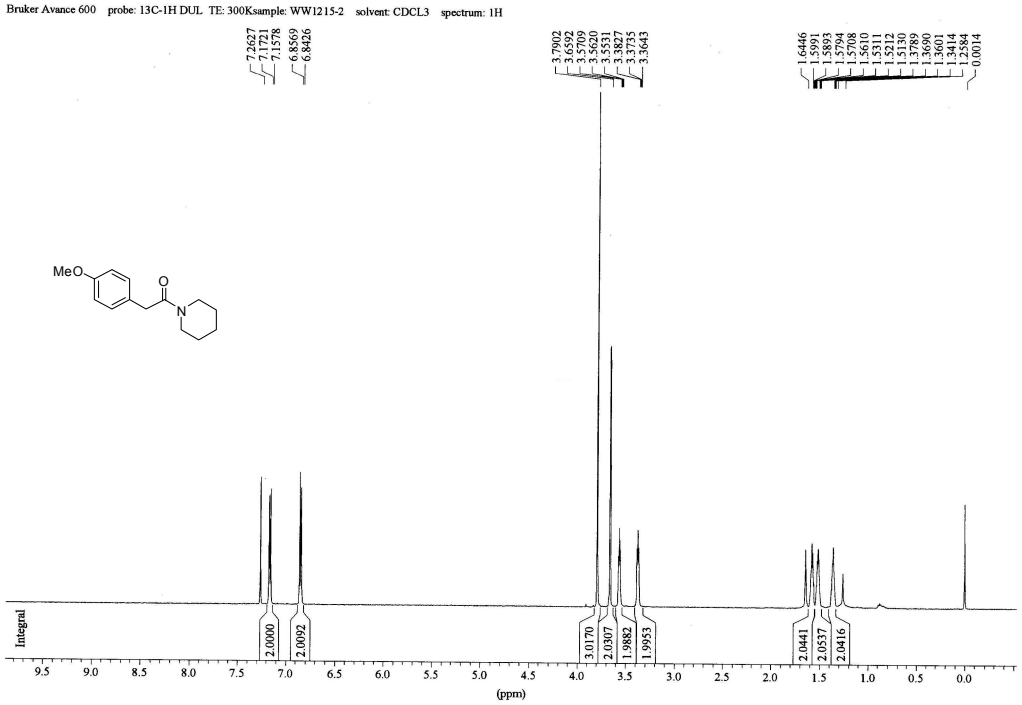
51.2796
43.9354
32.9798
23.6056
(ppm)

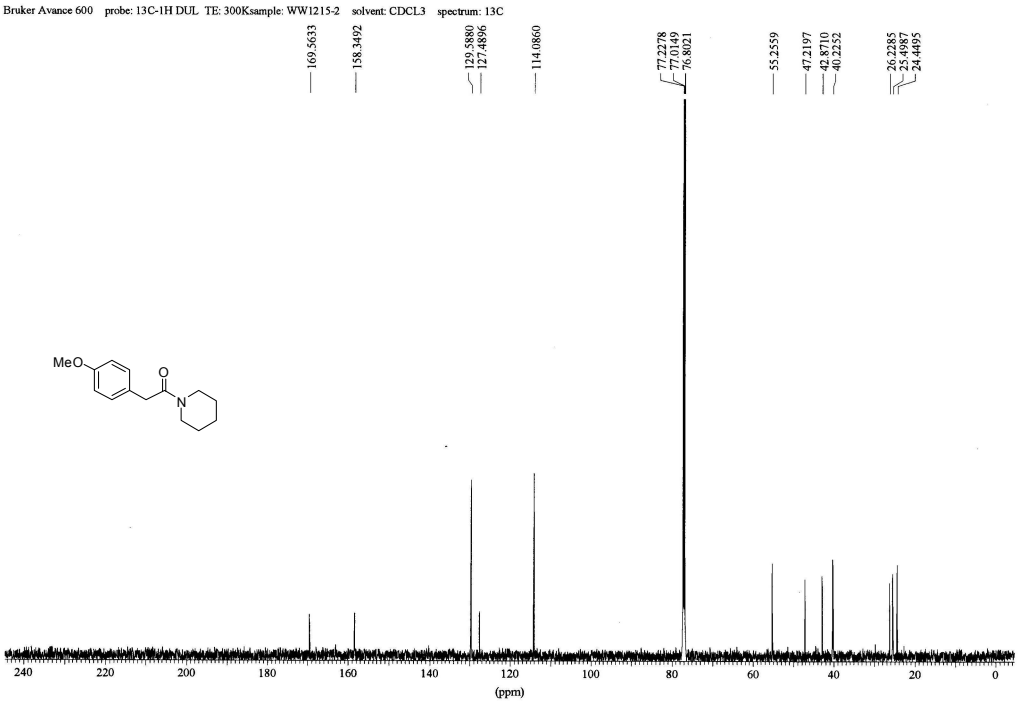

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-1 solvent: CDCL3 spectrum: 1H
7.2608
7.1410
7.1274
7.1175
7.1045
3.6810
3.5704
3.5615
3.5532
3.3708
3.3622
3.3529
2.3180
1.7085
1.5824
1.5734
1.5652
1.5546
1.5195
1.5112
1.3596
1.3510
0.0002
Me
O
N
Integral
4.0261
2.0260
2.0145
2.0000
3.0763
2.0116
2.0395
2.0432
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

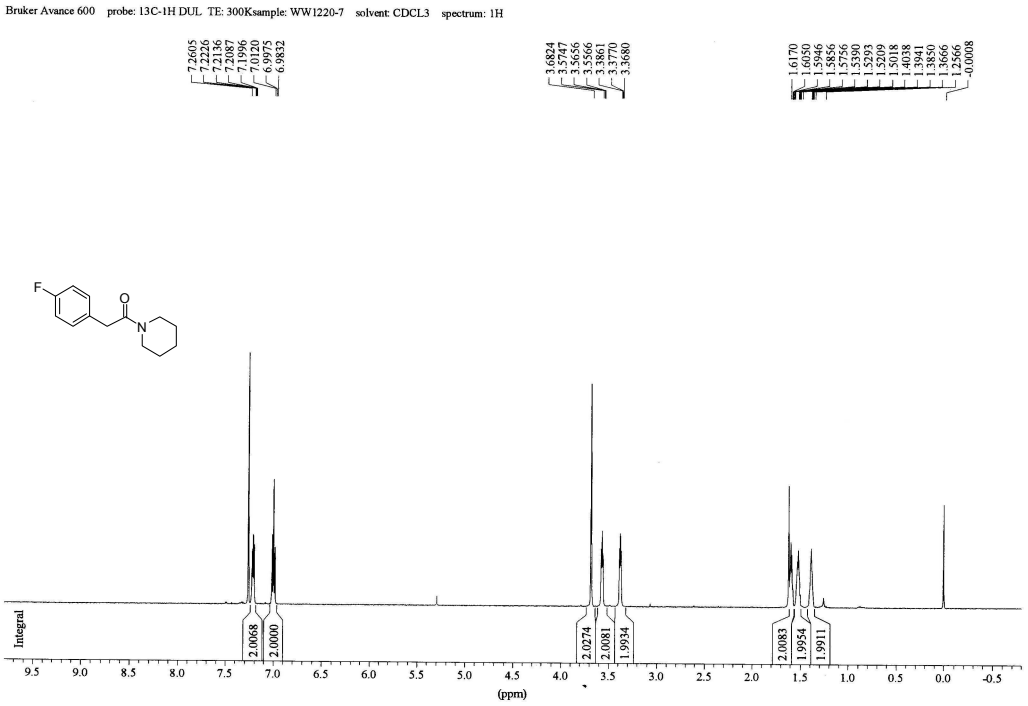

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-1 solvent: CDCL3 spectrum: 13C
169.4645
136.1264
133.3250
129.3719
128.4248
77.2582
77.0454
76.8325
47.2349
42.8558
40.7650
26.1981
25.4911
24.4495
21.0206
O
N
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

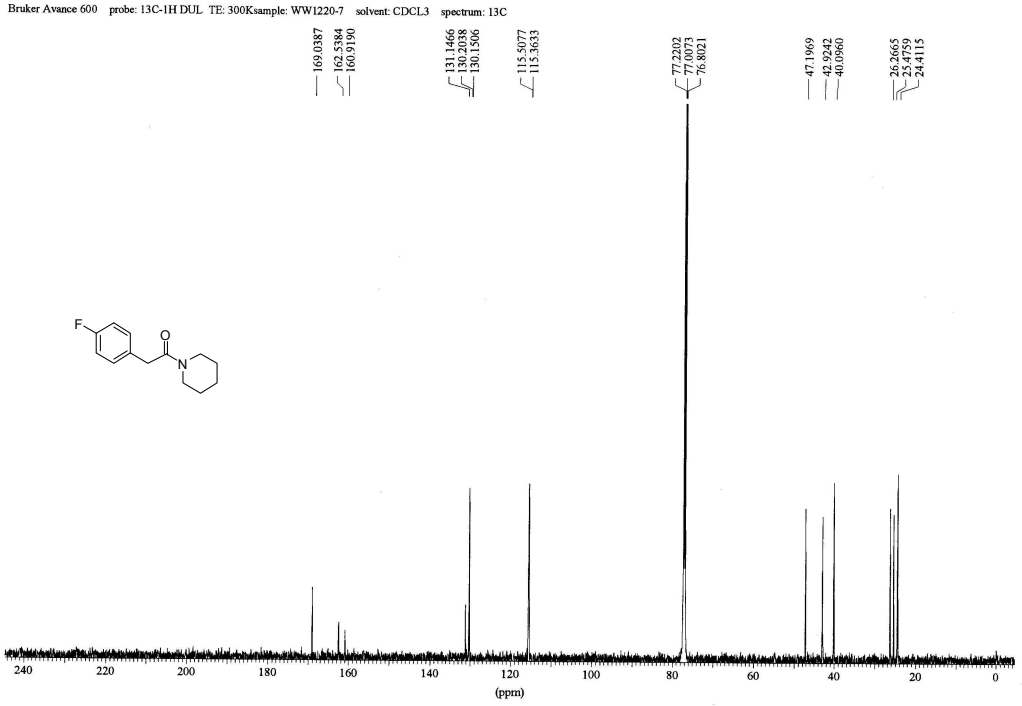

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-2 solvent: CDCL3 spectrum: 1H
7.2607
7.2038
7.1912
7.1786
7.0737
7.0416
7.0293
3.6869
3.5796
3.5704
3.5614
3.3783
3.3694
3.3601
2.3259
1.7058
1.5999
1.5906
1.5807
1.5717
1.5618
1.5373
1.5264
1.5187
1.5095
1.3814
1.3714
1.3625
1.3535
1.3439
0.0005
Me
O
N
Integral
1.0517
1.0376
2.0494
2.0588
2.0247
2.0000
3.0900
2.0538
2.0364
2.0183
(ppm)

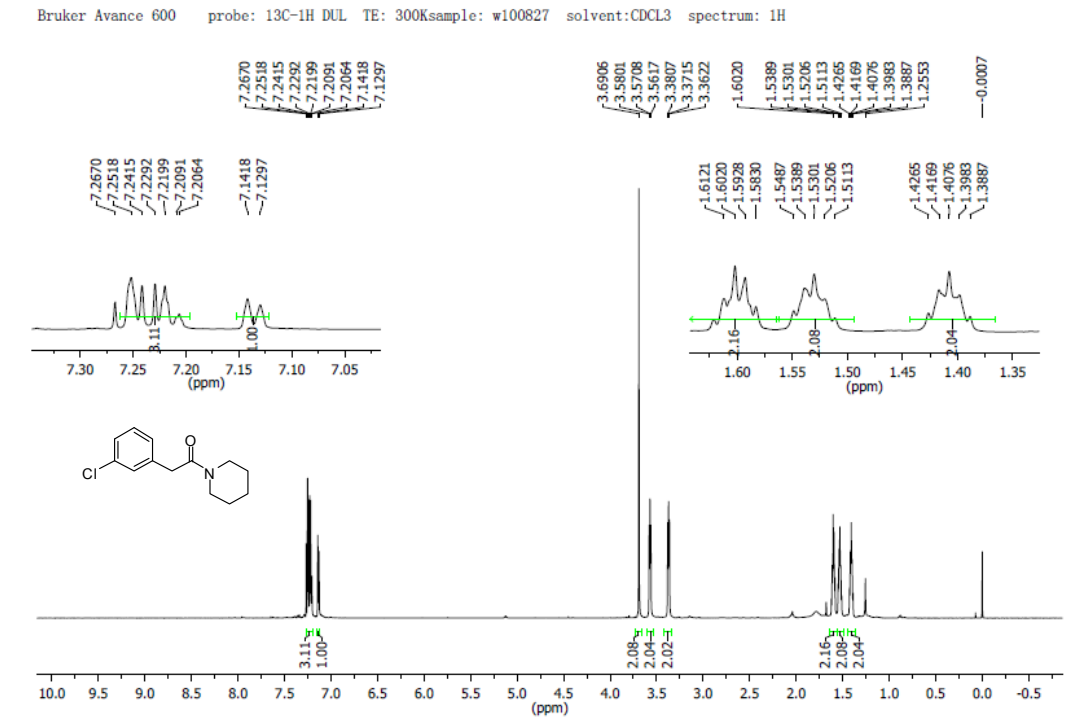

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-2 solvent: CDCL3 spectrum: 13C
169.3504
138.2552
135.3205
130.2015
128.4856
127.3604
125.5966
77.2734
77.0606
76.8477
47.2578
42.8634
41.0843
26.1981
25.5063
24.4571
21.3704
Me
O
N
(ppm)

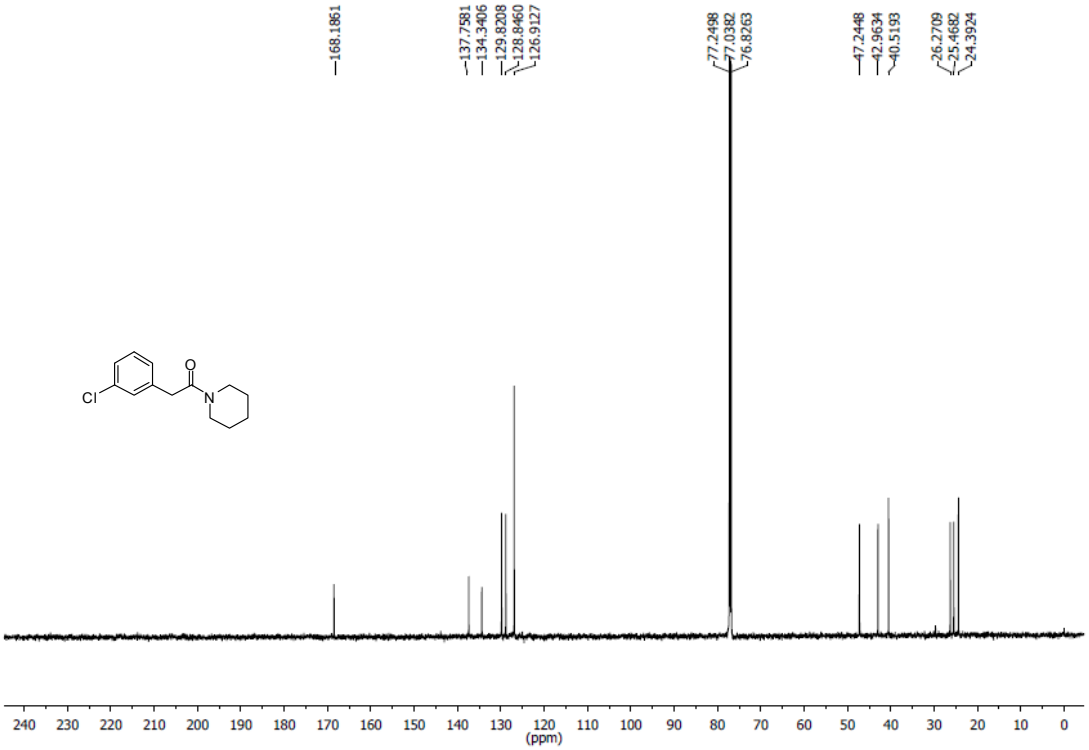

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1215-2 solvent: CDCL3 spectrum: 1H
7.2627
7.1721
7.1578
6.8569
6.8426
3.7902
3.6592
3.5709
3.5620
3.5531
3.3927
3.3735
3.3643
1.6446
1.5901
1.5893
1.5794
1.5708
1.5610
1.5311
1.5212
1.5130
1.3789
1.3690
1.3601
1.3414
1.2584
0.0014
MeO
O
N
Integral
2.0000
2.0092
3.0170
2.0307
1.9882
1.9953
2.0441
2.0537
2.0416
(ppm)

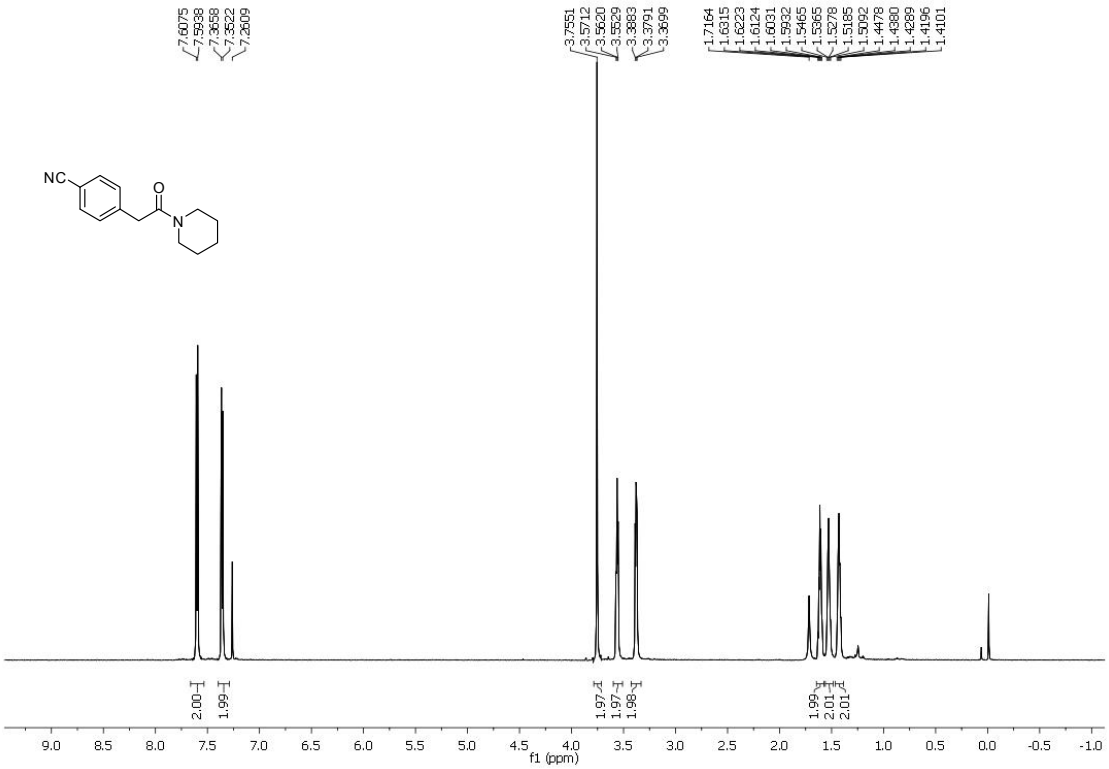

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1215-2 solvent: CDCL3 spectrum: 13C
169.5633
158.3492
129.5880
127.4896
114.0860
77.2278
77.0149
76.8021
55.2559
47.2197
42.8710
40.2252
26.2285
25.4987
24.4495
MeO
O
N
(ppm)

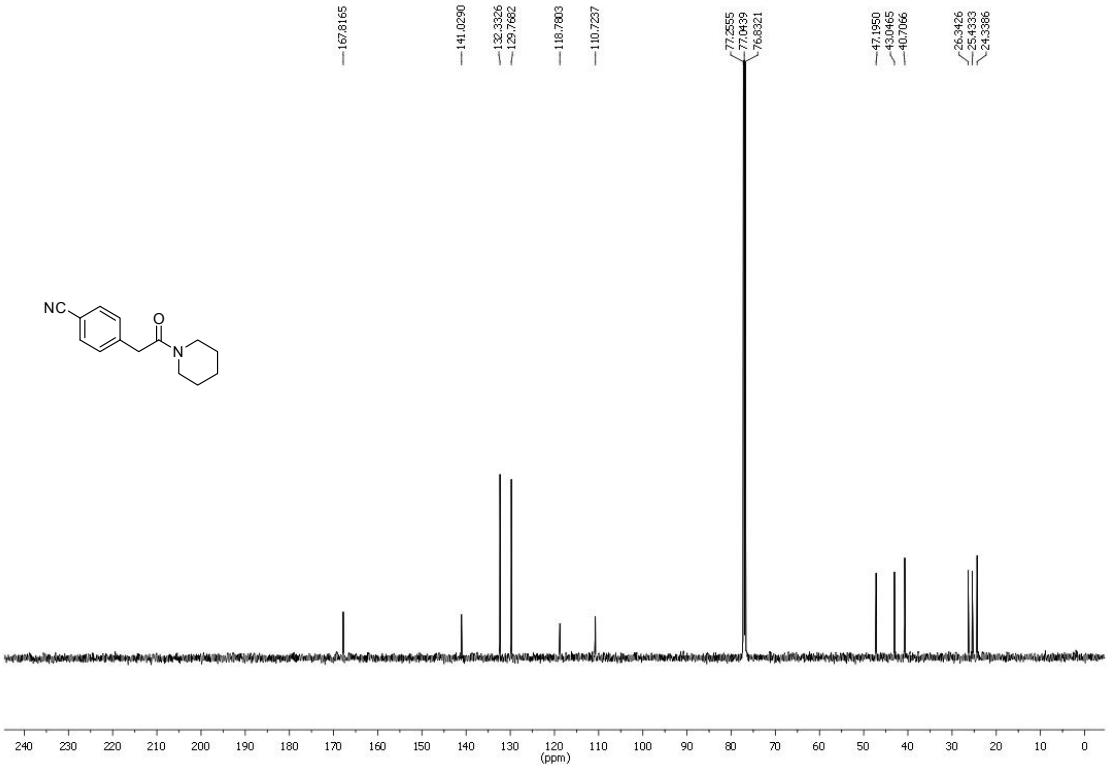

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1220-7 solvent: CDCL3 spectrum: 1H
7.2605
7.2226
7.2136
7.2087
7.1996
7.0120
6.9975
6.9832
3.6824
3.5747
3.5656
3.5566
3.3861
3.3770
3.3680
1.6170
1.6050
1.5946
1.5856
1.5756
1.5390
1.5293
1.5209
1.5018
1.4038
1.3941
1.3855
1.3666
1.2566
-0.0008
F
O
N
Integral
2.0068
2.0000
2.0274
2.0081
1.9934
2.0083
1.9954
1.9911
(ppm)

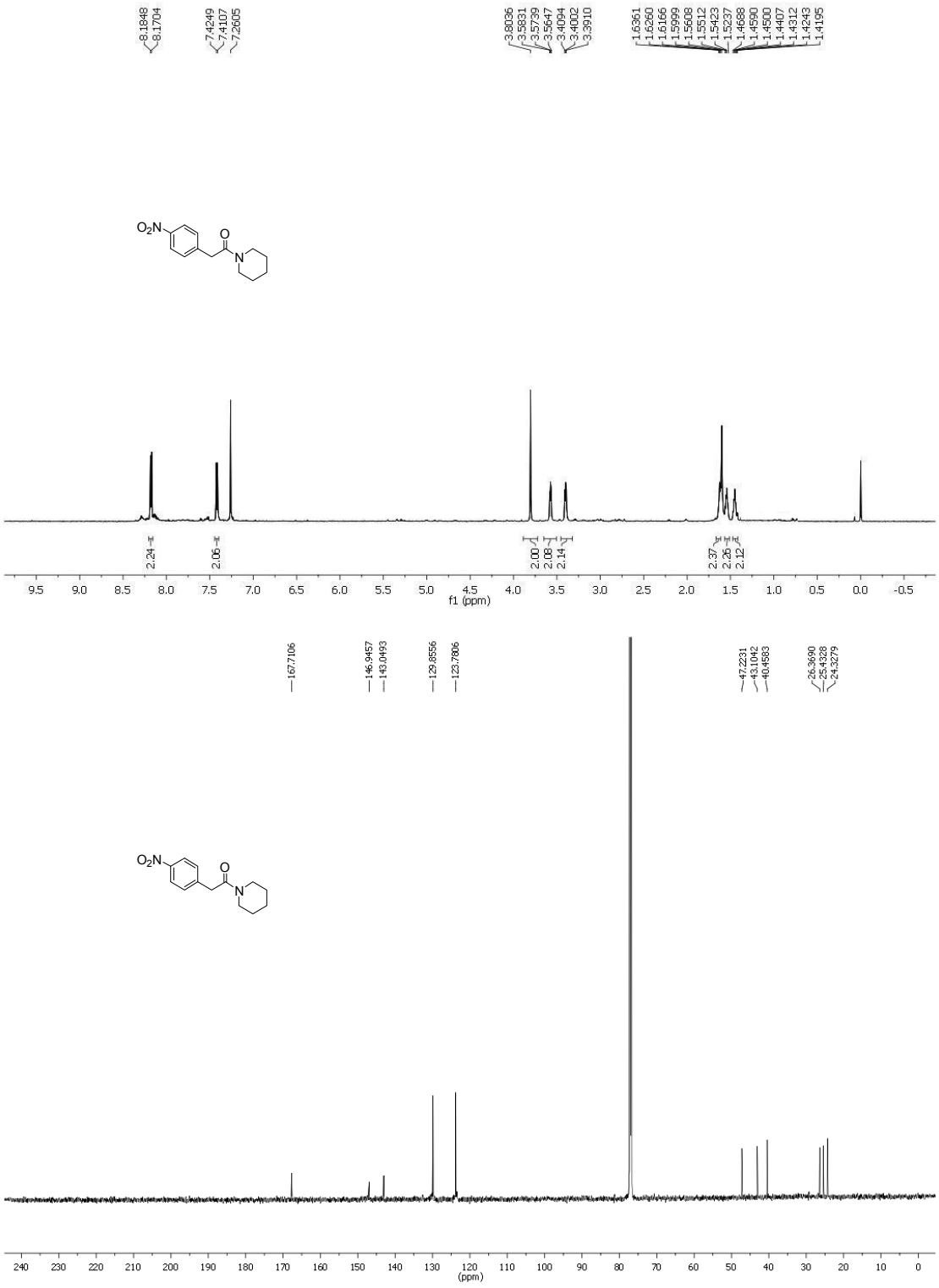

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1220-7 solvent: CDCL3 spectrum: 13C
169.0387
162.5384
160.9190
131.1466
130.2858
130.1506
115.5077
115.3633
77.2202
77.0073
76.8021
47.1969
42.9242
40.0960
26.2665
25.4759
24.4115
F
O
N
(ppm)

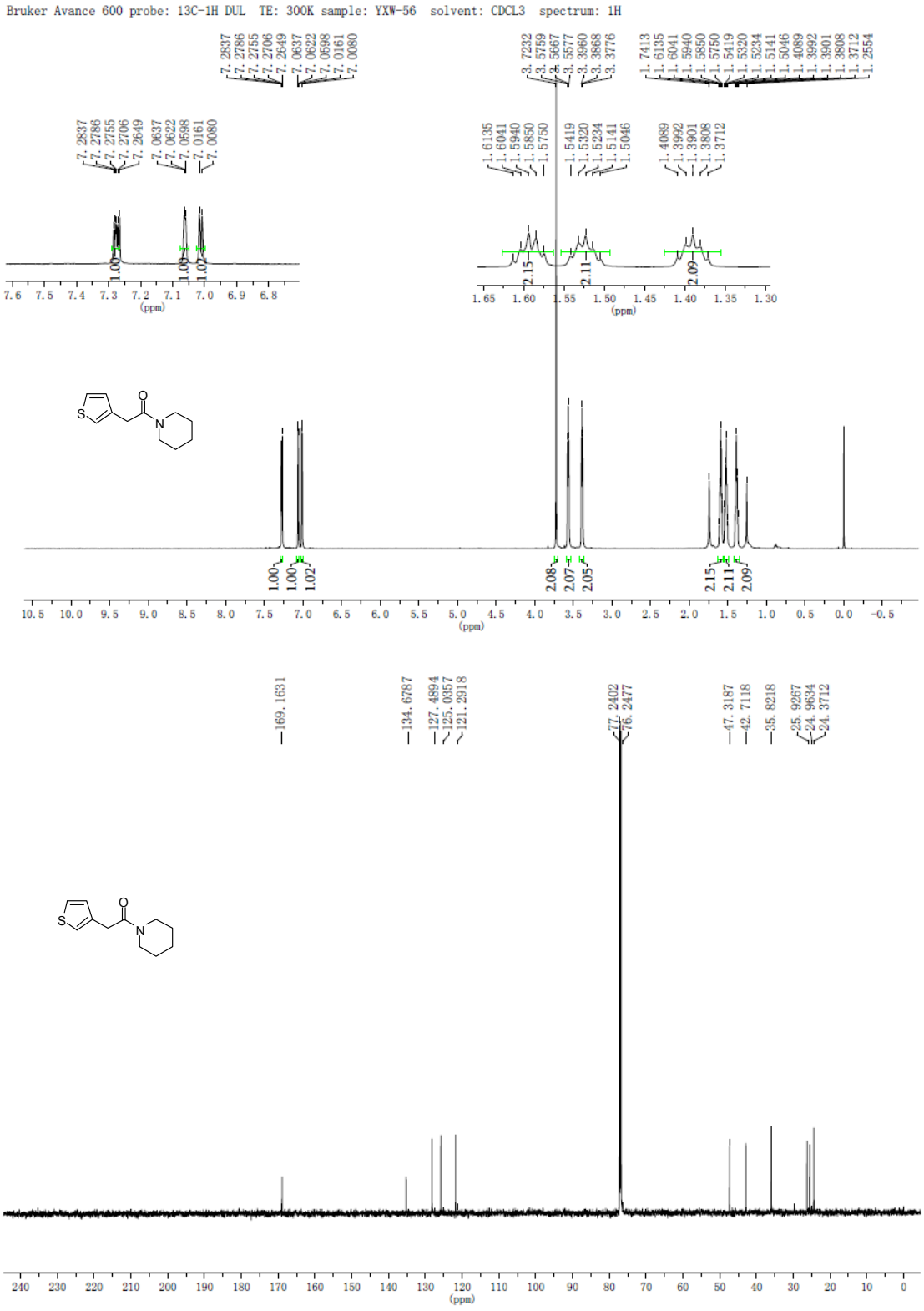

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: w100827 solvent:CDCL3 spectrum: 1H
(ppm)
Cl
O
N

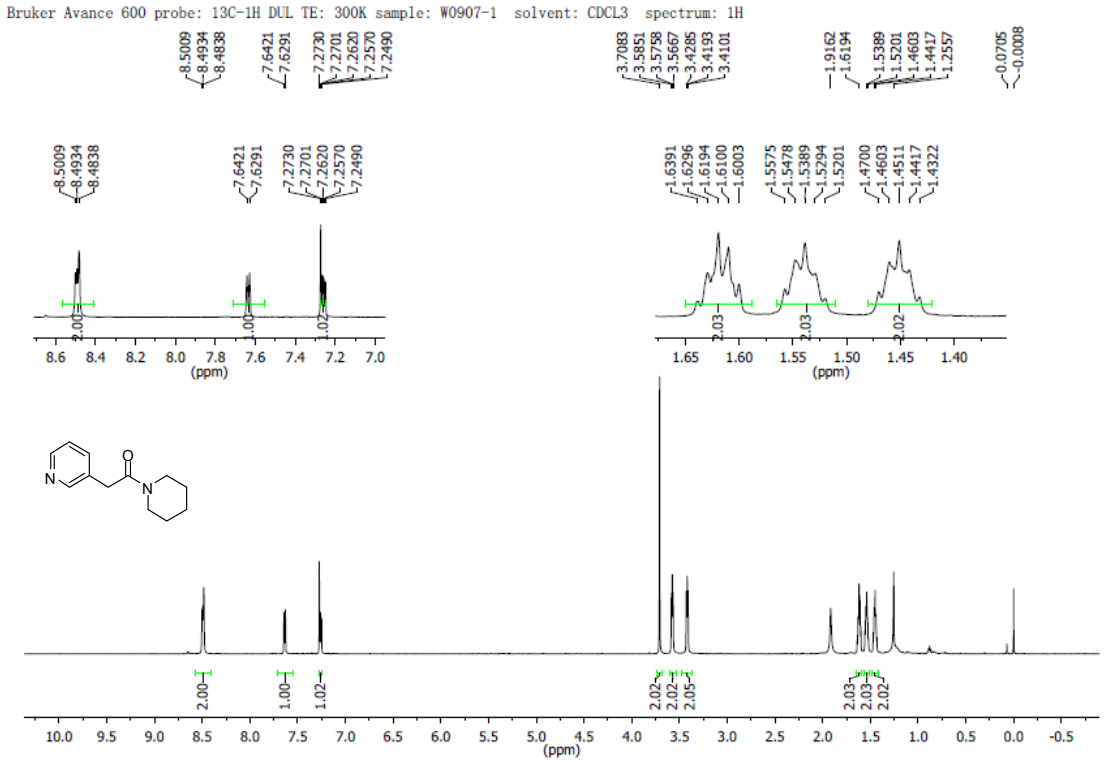

(ppm)
Cl
O
N

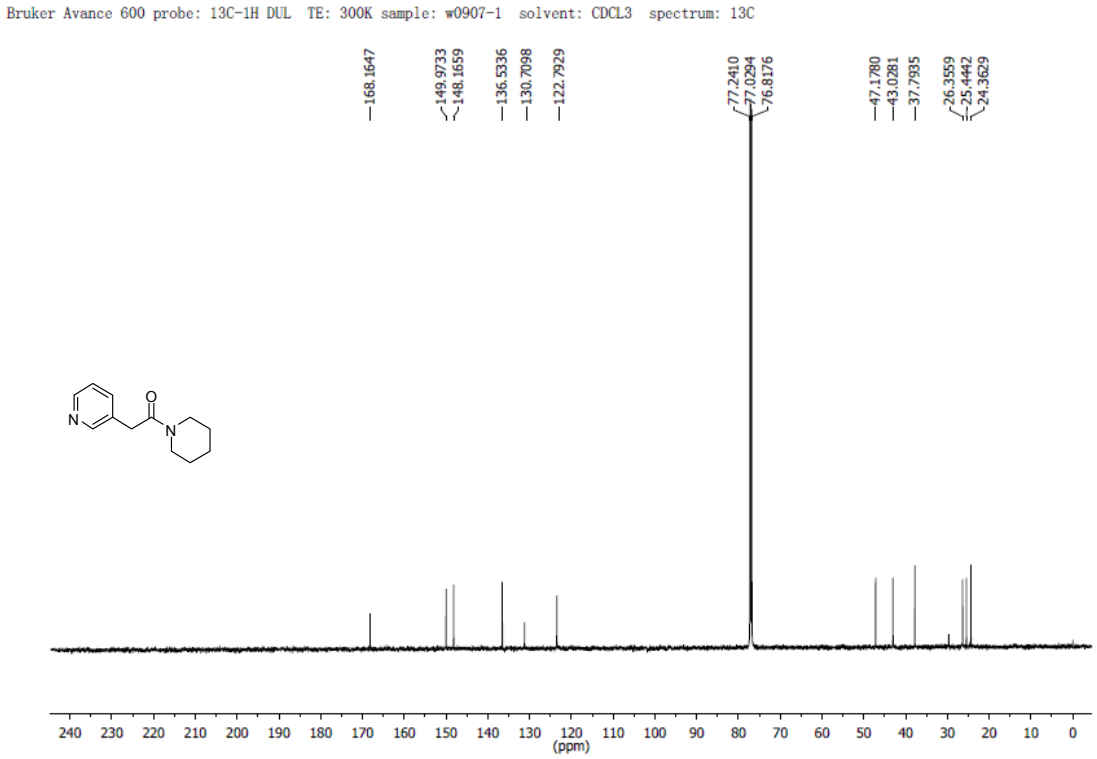

7.6075
7.5938
7.3658
7.3522
7.2609
3.7551
3.5712
3.5620
3.5529
3.3883
3.3791
3.3699
1.7164
1.6315
1.6223
1.6124
1.6031
1.5932
1.5465
1.5365
1.5278
1.5185
1.5092
1.4478
1.4380
1.4289
1.4196
1.4101
NC
O
N
2.00
1.99
1.97
1.97
1.98
1.99
2.01
2.01
f1 (ppm)

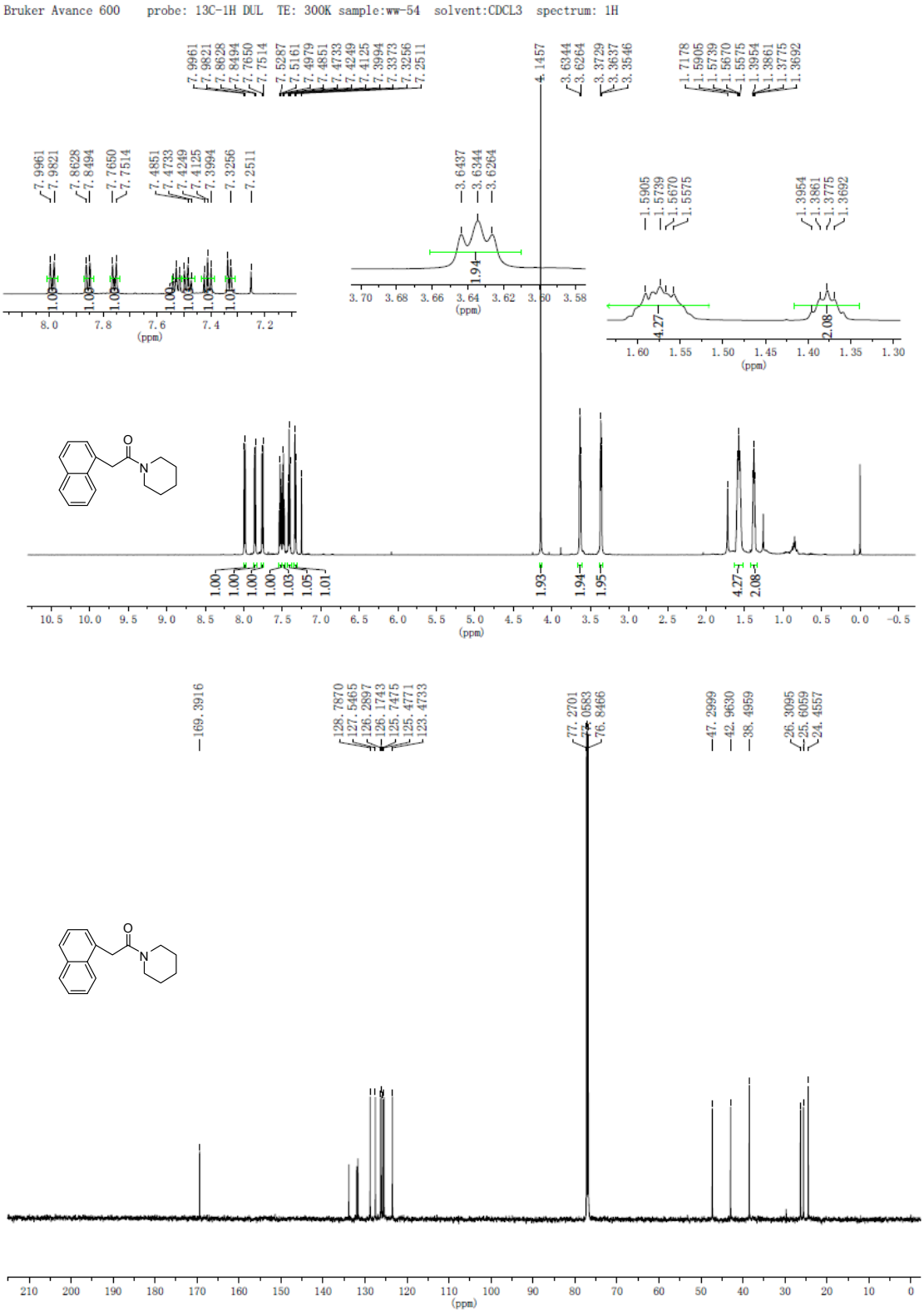

167.8165
141.0290
132.3326
129.7682
118.7803
110.7237
77.2555
77.0439
76.8321
47.1950
43.0465
40.7066
26.3426
25.4333
24.3386
NC
O
N
(ppm)

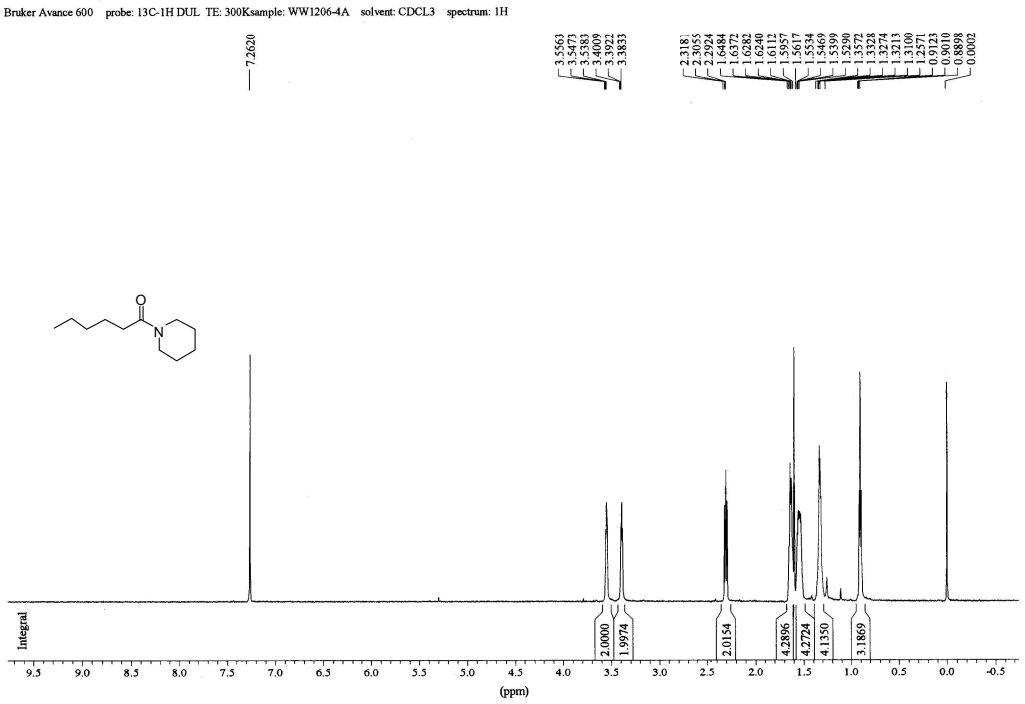

8.1848
8.1704
7.4249
7.4107
7.2605
3.8026
3.5831
3.5739
3.5647
3.4094
3.4002
3.3910
1.6361
1.6260
1.6166
1.5999
1.5608
1.5512
1.5423
1.5237
1.4688
1.4590
1.4500
1.4407
1.4312
1.4243
1.4195
O2N
O
N
2.24
2.06
2.00
2.08
2.14
2.37
2.26
2.12
f1 (ppm)
167.7106
146.9457
143.0493
129.8556
123.7806
47.2231
43.1042
40.4583
26.3690
25.4328
24.3279
(ppm)

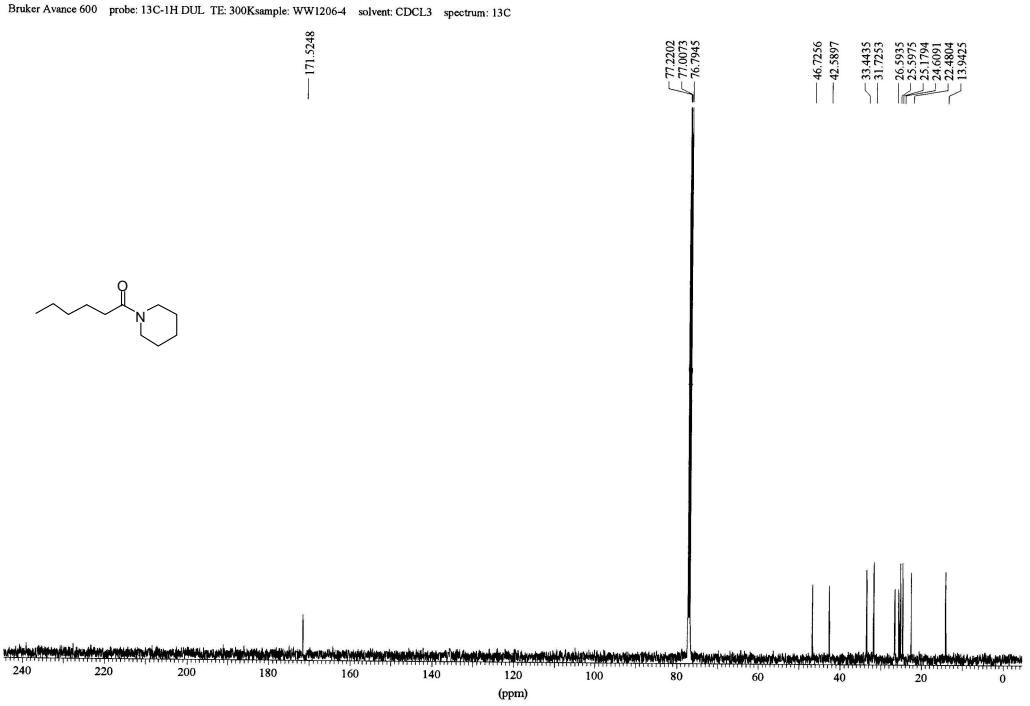

Bruker Avance 600 probe: 13C-1H DUL TE: 300K sample: YXW-56 solvent: CDCL3 spectrum: 1H
(ppm)
(ppm)
(ppm)
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300K sample: W0907-1 solvent: CDCL3 spectrum: 1H
8.5009
8.4934
8.4838
7.6421
7.6291
7.2730
7.2701
7.2620
7.2570
7.2490
3.7083
3.5851
3.5758
3.5667
3.4285
3.4193
3.4101
1.9162
1.6194
1.5389
1.5201
1.4603
1.4417
1.2557
0.0705
-0.0008
1.6391
1.6296
1.6194
1.6100
1.6003
1.5575
1.5478
1.5389
1.5294
1.5201
1.4700
1.4603
1.4511
1.4417
1.4322
2.00
1.00
1.02
2.03
2.03
2.02
(ppm)
2.02
2.02
2.05
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300K sample: w0907-1 solvent: CDCL3 spectrum: 13C
168.1647
149.9733
148.1659
136.5336
130.7098
122.7929
77.2410
77.0294
76.8176
47.1780
43.0281
37.7935
26.3559
25.4442
24.3629
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300K sample:ww-54 solvent:CDCL3 spectrum: 1H
7.9961
7.9821
7.8628
7.8494
7.7650
7.7514
7.5287
7.5161
7.4979
7.4851
7.4733
7.4249
7.4125
7.3994
7.3373
7.3256
7.2511
4.1457
3.6344
3.6264
3.3729
3.3637
3.3546
1.7178
1.5905
1.5739
1.5670
1.5575
1.3954
1.3861
1.3775
1.3692
(ppm)
169.3916
128.7870
127.5465
126.2897
126.1743
125.7475
125.4771
123.4733
77.2701
77.0583
76.8466
47.2999
42.9630
38.4959
26.3095
25.6059
24.4557
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-4A solvent: CDCL3 spectrum: 1H
7.2620
3.5563
3.5473
3.5383
3.4009
3.3922
3.3833
2.3181
2.3055
2.2924
1.6484
1.6372
1.6248
1.6140
1.6111
1.5957
1.5617
1.5534
1.5469
1.5399
1.3290
1.3272
1.3238
1.3274
1.3213
1.3100
1.2571
0.9123
0.9010
0.8898
0.0002
Integral
2.0000
1.9974
2.0154
4.2896
4.2724
4.1350
3.1869
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1206-4 solvent: CDCL3 spectrum: 13C
171.5248
77.2202
77.0073
76.7945
46.7256
42.5897
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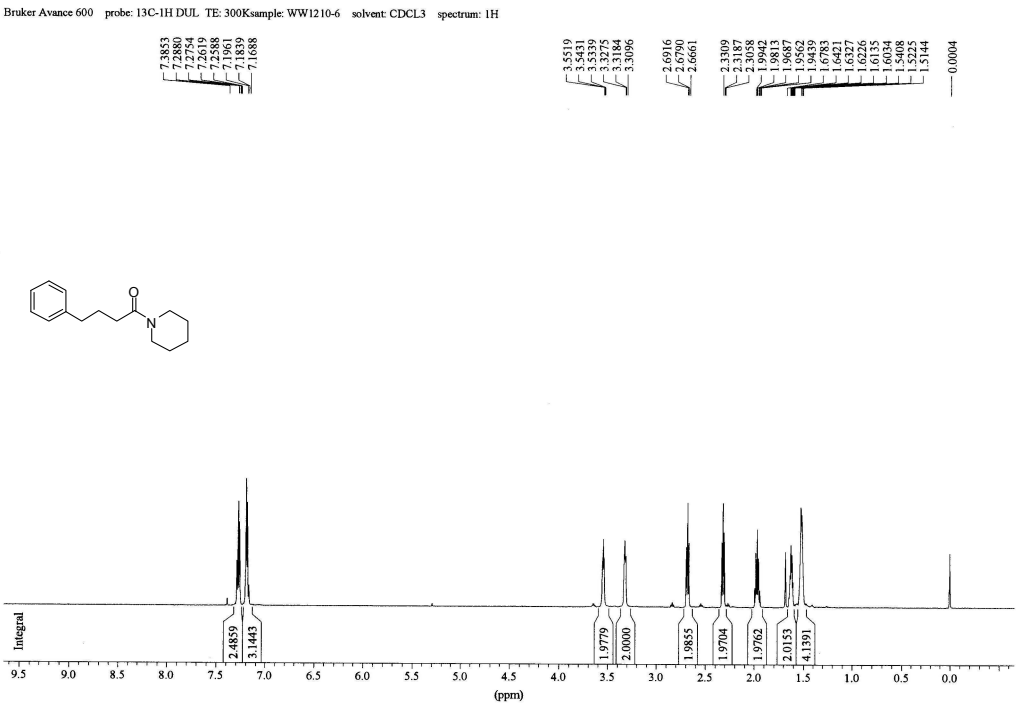
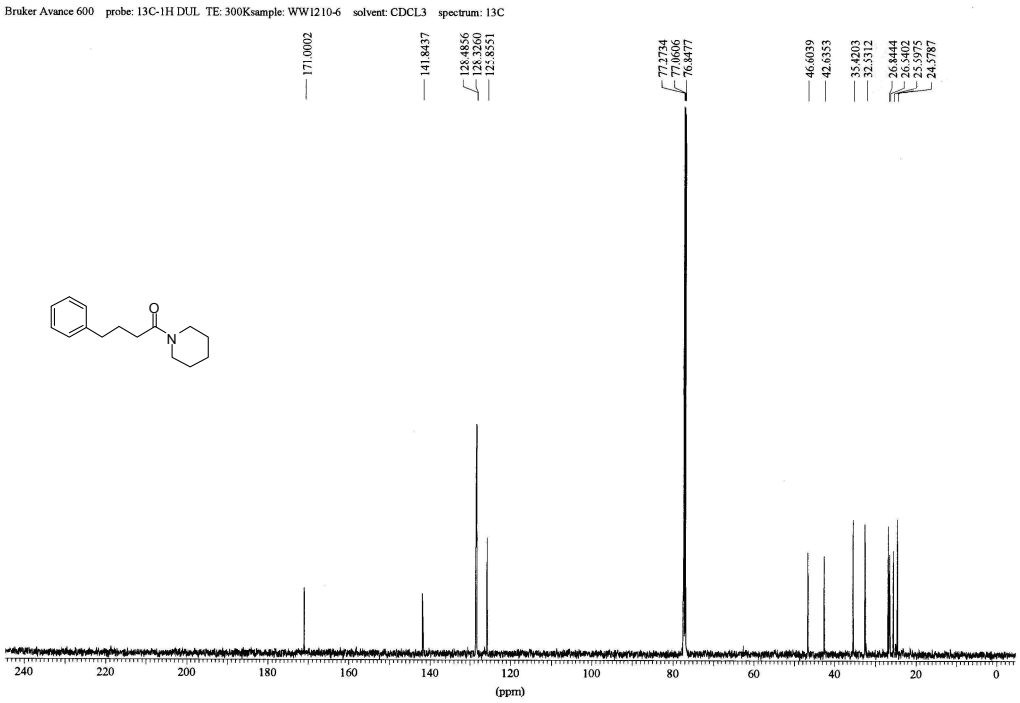
31.7253
26.5935
25.5975
25.1794
24.6091
22.4804
13.9425
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1210-6 solvent: CDCL3 spectrum: 1H
7.3853
7.2880
7.2754
7.2619
7.2588
7.2396
7.1839
7.1688
3.5519
3.5431
3.5299
3.3275
3.3184
3.3096
2.6916
2.6790
2.6661
2.3309
2.3187
2.3058
1.9942
1.9813
1.9687
1.9562
1.9439
1.6783
1.6421
1.6327
1.6226
1.6135
1.6034
1.5908
1.5225
1.5144
0.0004
Integral
2.4859
3.1443
1.9779
2.0000
1.9855
1.9704
1.9762
2.0153
4.1391
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: WW1210-6 solvent: CDCL3 spectrum: 13C
171.0002
141.8437
126.4856
125.3260
125.8551
77.2734
77.0606
76.8477
46.6039
42.6353
35.4203
32.5312
26.8444
26.5402
25.5975
24.5787
(ppm)