Electronic Supplementary Information

Highly active and selective synthesis of imines from alcohols and amines or nitroarenes catalyzed by Pd/DNA in water with dehydrogenation

Lin Tang, Huayin Sun, Yunfeng Li, Zhenggen Zha, Zhiyong Wang*

Hefei National Laboratory for Physical Sciences at Microscale, CAS Key Laboratory of Soft Matter Chemistry and Department of Chemistry,
University of Science and Technology of China, Hefei, 230026, P. R. China

Contents:

General Remarks

All substrates were purchased commercially without further purification. Fish sperm DNA (CAS: 100403-24-5) were purchased from BBI. Starch soluble (CAS: 9005-25-8), Gum Arabic powder (CAS: 9000-01-5), Polyvinyl pyrrolidone (PVP K-30, CAS: 9003-39-8), were purchased from Sinopharm Chemical Reagent Co., Ltd. UV-Vis tests were taken on a Persee TU1901 UV-Vis spectromter. All the solutions were diluted to similar concentration and gained the desirable normalized curves. The morphology and size of the Pd NPs were characterized on transmission electron microscopy (TEM) (JEOL–2010 and Hitachi H7650). The diluted solutions of the as-synthesized Pd-DNA nanohybrid were used as samples directly and dried on the carbon-coated Cu grids. X-ray powder diffraction (XRD) experiments were carried out with a Philips X'Pert Pro Super diffractometer with Cu KR radiation (λ = 1.54178 Å). The accurate concentrations of Pd/DNA nanohybrid and other supported Pd catalysts were directly determined by ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometer) using Perkin Elmer Optima 7300 DV. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded on a Bruker AC-300 FT (^{1}H NMR 300 MHz, ^{13}C NMR 75 MHz) or Bruker AC-400 FT(^{1}H NMR 400 MHz, ^{13}C NMR 100 MHz) using TMS as internal reference. Infrared samples of DNA and Pd/DNA were recorded on a Thermo Scientific Nicolet 8700 spectrometer. HRMS were recorded on a MicroMass GCT TOF-MS. GC-MS samples were recorded on a Shimadzu QP-5050 GC-MS system. And the yields were determined using 1,3,5-trimethylbenzene as an internal standard. X-ray photoelectron spectroscopy (XPS) measurements were performed on an ESCALAB 250 electron spectrometer using monochromatized Al Kα excitation source ($h\nu$ = 1486.6 eV). H_2 was detected by a online mass spectroscopy (Hiden-QIC20).

General procedures for the synthesis of heterogeneous Pd catalysts

0.2 mmol of the corresponding metal salt ($PdCl_2$, $KAuCl_4$, $AgNO_3$, K_2PtCl_6) and 20 mg of fish sperm DNA were dissolved in 24 ml Tris buffer (10mM, pH = 7.4). The combined solution was stirred for 24 h to ensure the corresponding metal ion (Pd^{2+}, Ag^+, Au^{3+}, Pt^{4+}) thoroughly bind to DNA. After this aging process, 1.0 mmol of freshly dissolved $NaBH_4$ in 16 ml Tris buffer was added dropwise under N_2 atmosphere at 0 °C. After reduction, the solution was stirred for another 24 h in N_2 from 0 °C to room temperature to obtain the resulting M/DNA nanohybrids (c.a. 5 mM in Tris). This synthesis method can be scaled up to hundreds of millilitres easily at a time.

Other water-soluble palladium nanoparticles, such as Pd/starch, Pd/PVP and Au/gum arabic, were was synthesized by similar procedure according to the synthesis of M/DNA. For example, 0.05 mmol of $PdCl_2$ and 50 mg of water-soluble starch were dissolved in 5 ml H_2O. The combined solution was stirred for 24 h. After this aging process, 0.25 mmol of freshly dissolved $NaBH_4$ in 5 ml H_2O was added dropwise under N_2 atmosphere at 0 °C. After reduction, the solution was stirred for another 24 h in N_2 from 0 °C to room temperature to obtain the resulting Pd/starch (c.a. 5 mM in H_2O).

The Pd/diatomite nanoparticles were synthesized according to previous report.[s1] The

Pd/MMT nanoparticles were synthesized according the similar method using Na-MMT instead of diatomite. Pd/C (10%) was supplied by Sinopharm Chemical Reagent Co., Ltd.

Reference

S1 Z. H. Zhang, Z. Y. Wang, *J. Org. Chem.*, 2006, **71**, 7485.

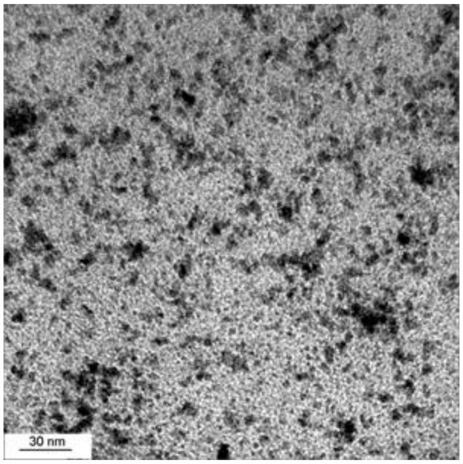
General procedure for the tandem imination

Synthesis of imine from alcohol and amine: For M/DNA nanohybrids: To 2 ml of as-synthesized Pd/DNA nanohybrids were precipitated by adding excess EtOH (3 times of the volume). After sitting for 1 h and then centrifuged at 6000 r min^{-1} for 4 min, the decantate was poured off. The obtained solid residue was dried by N_2 flow and redispersed in a water solution (1 mL) containing $LiOH•H_2O$ (15.7 mg, 0.375 mmol). Benzyl alcohol (29.7 mg, 0.275 mmol) and aniline (23.3 mg, 0.25 mmol) were then added to the solution. The air in the reaction mixture was removed under vacuum and the reaction vessel refilled with N_2. This procedure was repeated for three times. The reaction mixture was then stirred under an N_2 balloon at 50 °C for 12 h. After the reaction was finished, 3 times the volume of EtOH and 5 times the volume of EtOAc was added to the reaction mixture. This mixture was left undisturbed for 2 h to allow precipitation and then centrifuged at 6000 r min^{-1} for 4 min. The decantate was poured off. The solid residue was dried and used as the catalyst for the next round. The decantate was evaporated with a rotary evaporator. The obtained mixture was dissolved in EtOAc and yield with conversion were determined by GC-MS using 1,3,5-trimethylbenzene as an internal standard. And the reaction was carried out at a higher scale with alcohol (5.1 mmol) and amine (5.0 mmol) to afford the pure imine by recrystallization or vacuum distillation. The resulting products were characterized by ^{1}H NMR, ^{13}C NMR and HRMS.

Synthesis of imines from alcohols and nitroarenes compound: The tandem reaction was carried out with alcohol (1.0 mmol) and nitroarene (0.25 mmol) for 24 h under the standard reaction conditions.

Detailed characterization of heterogeneous Pd catalysts

TEM characterization of Pd/DNA



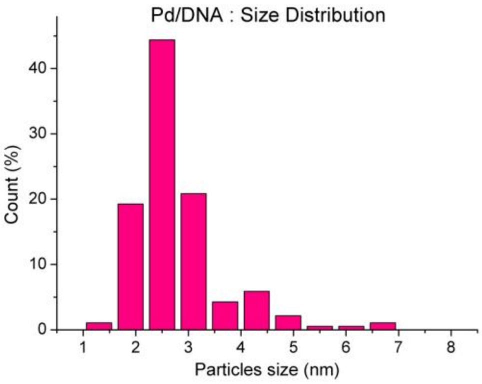


Fig. ESI-1. Pd/DNA average diameter = 2.87 nm.

XRD patterns for Pd/DNA nanohybrids

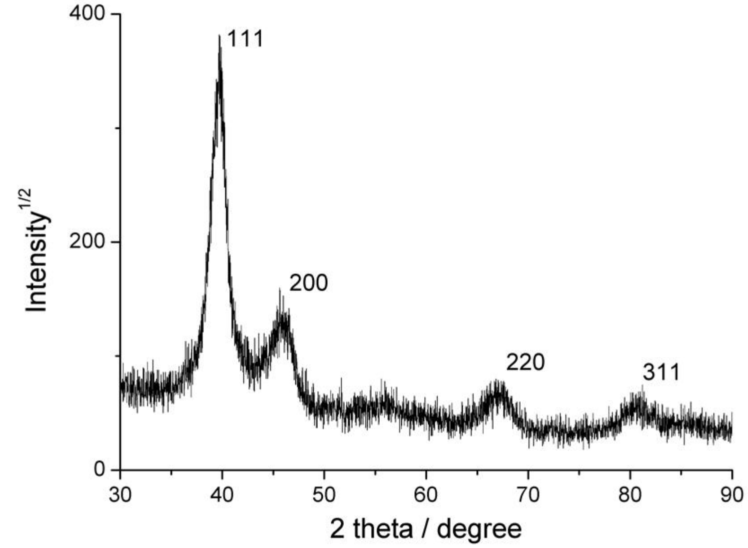


Fig. ESI-2 XRD patterns of Pd/DNA

Uv-Vis Spectra of Pd/DNA compared with only DNA and NaBH$_4$

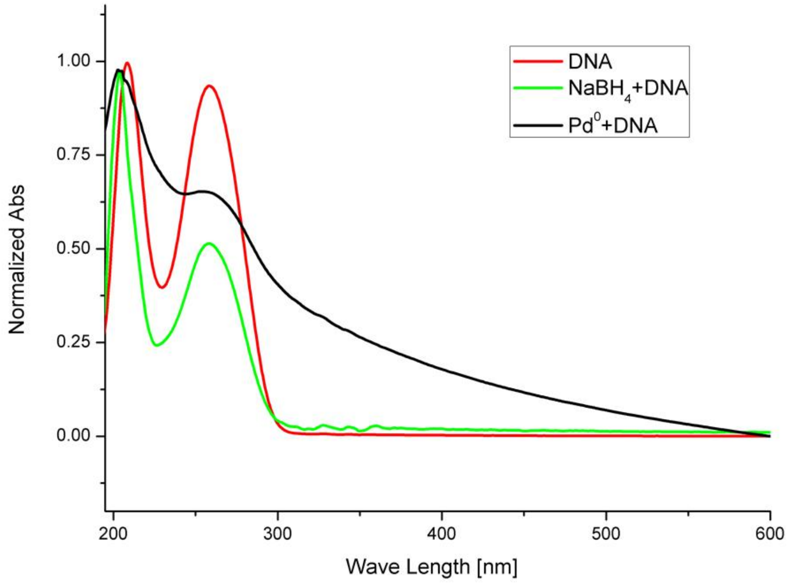


Fig. ESI-3 Uv-Vis spectra of DNA (red line), Pd/DNA (black line) and DNA after only adding $NaBH_4$ (green line).

IR spectra of DNA and Pd/DNA.

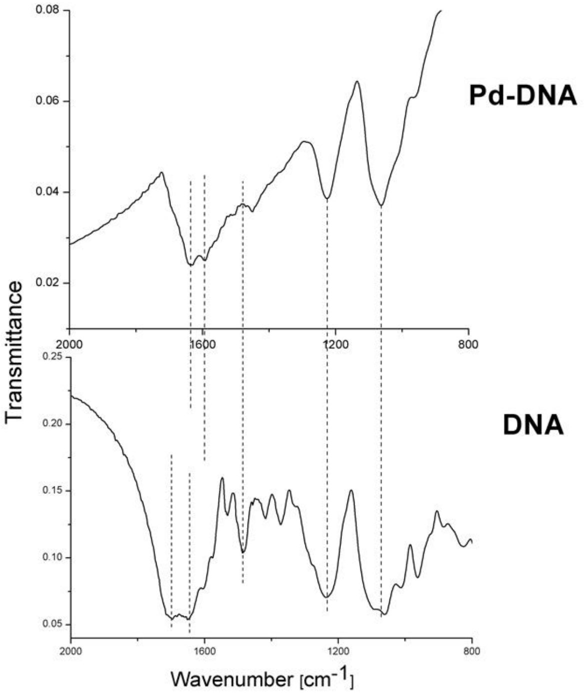
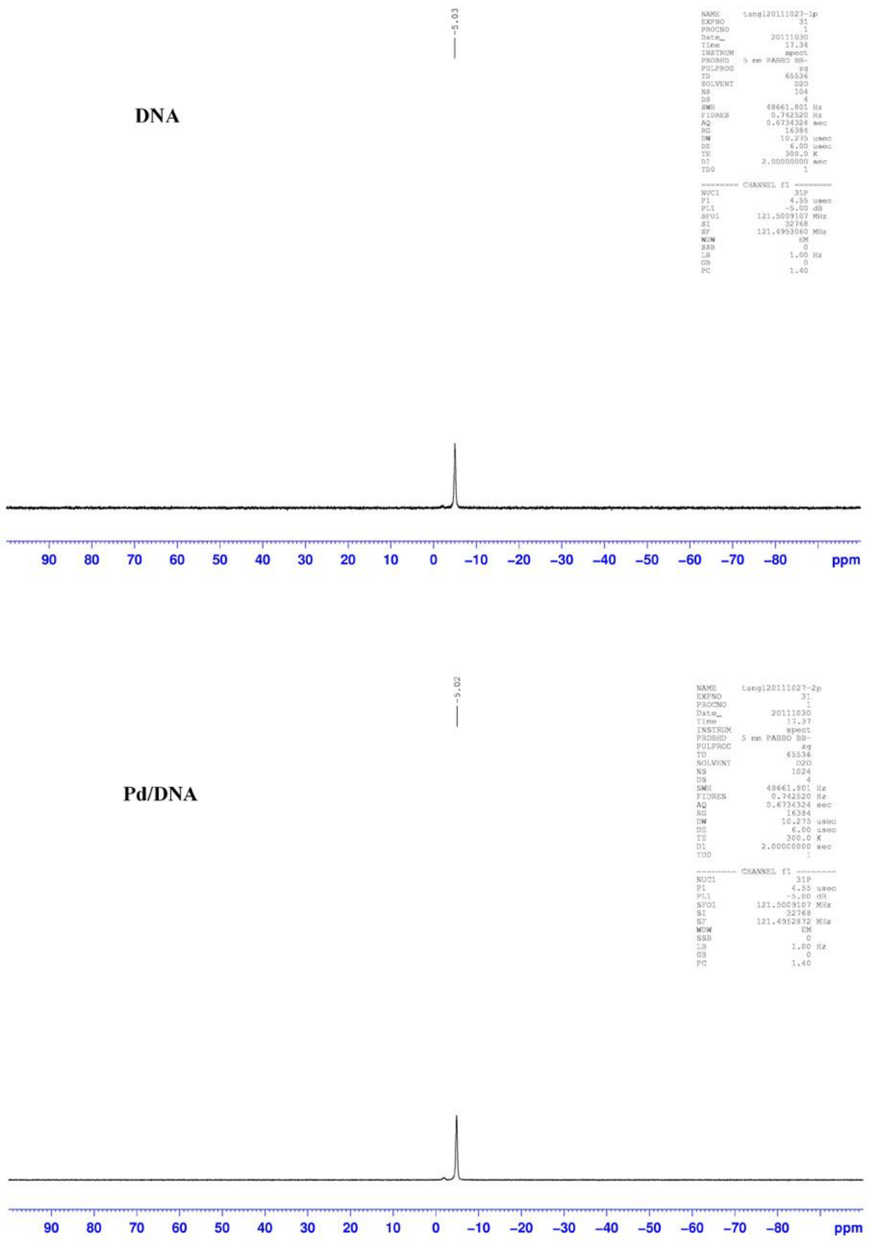


Fig. ESI-4 IR spectra of Pd/DNA and DNA

No obvious variation was found before and after reduction at 1060 and 1230 cm^{-1}, which were the symmetric and antisymmetric stretching modes of PO^{2-}, showing that Pd NPs didn't bind to this area. The variation at 1650 - 1685 cm^{-1}, which was contributed from C=C, C-N, C=O stretching modes, indicated that the base pairs of the DNA may interacted with Pd NPs. Especially, the weakening of 1480 cm^{-1} (C=N) implied that Pd NPs may had interaction with the nitrogen atom of the base pairs. All of these indicated that Pd/DNA nanohybrid was formed.

^{31}P NMR spectra of DNA and Pd/DNA.



```
NAME     tang120111027-1p
EXPNO                  31
PROCNO                  1
Date_            20111030
Time                17.34
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  65536
SOLVENT               D2O
NS                    104
DS                      4
SWH             48661.801 Hz
FIDRES           0.742520 Hz
AQ              0.6734324 sec
RG                  16384
DW                 10.275 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  31P
P1                   4.55 usec
PL1                 -5.00 dB
SFO1          121.5009107 MHz
SI                  32768
SF            121.4953060 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

```
NAME     tang120111027-2p
EXPNO                  31
PROCNO                  1
Date_            20111030
Time                17.37
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  65536
SOLVENT               D2O
NS                   1024
DS                      4
SWH             48661.801 Hz
FIDRES           0.742520 Hz
AQ              0.6734324 sec
RG                  16384
DW                 10.275 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  31P
P1                   4.55 usec
PL1                 -5.00 dB
SFO1          121.5009107 MHz
SI                  32768
SF            121.4952872 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Fig. ESI-5 ^{31}P NMR spectra of DNA and Pd/DNA

The ^{31}P NMR spectra of pure fish sperm DNA in Tris and the Pd/DNA solution which was gained through precipitating and re-dissolving procedure by adding excess EtOH indicated that after pouring out the fair solution, DNA remained in the black precipitation. Secondly, the chemical shift of ^{31}P in Pd/DNA system was almost the same as the pure DNA showed that Pd did not bind with the P atoms in the DNA.

The precedure for the detection of the hydrogen

The reaction mixture was stirred under an N_2 balloon at 50 °C in the reaction vessel (see Experimental Section for details). After the reaction was finished, the balloon was closed. Then the balloon was connect to an online mass spectroscopy (Hiden-QIC20). we set N_2 as the carrier gas to detect the H_2 liberated from this reaction. Opening the piston of the balloon, the gas entered into the system of this online mass spectroscopy. When H_2 had been detected, the piston was closed. This procedure was repeated for three times.

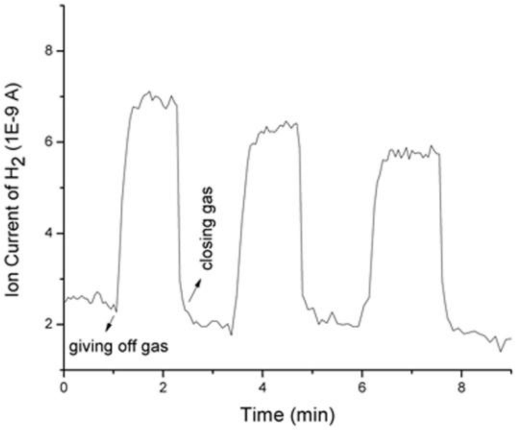


Fig. ESI-6 H_2 was detected by a online mass spectroscopy (Hiden-QIC20).

Characterization of Pd/DNA after recycles

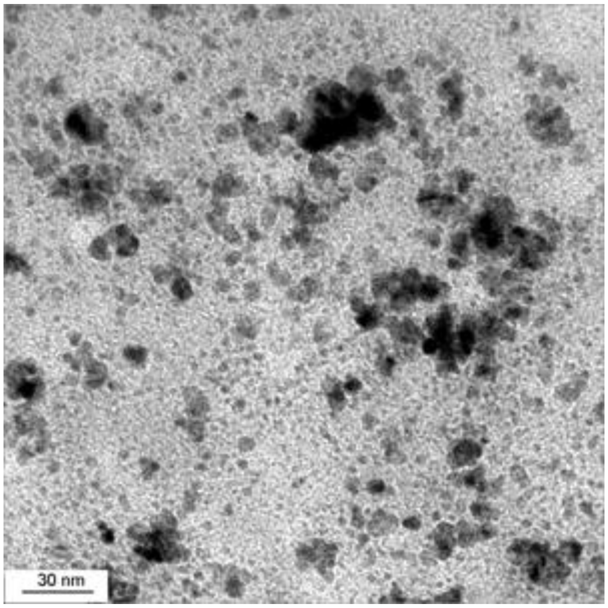


Fig. ESI-7 TEM image of the Pd/DNA after fifth round

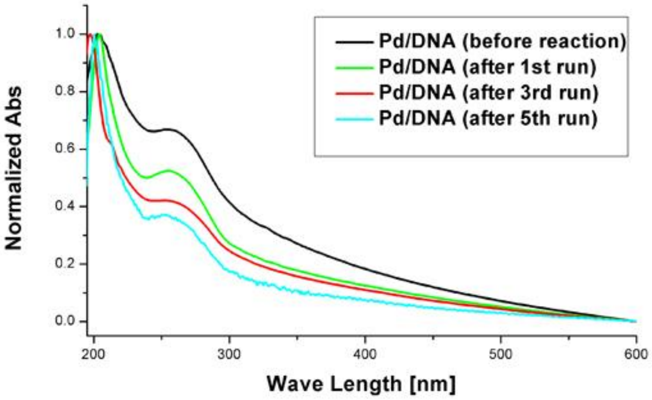


Fig. ESI-8 UV-Vis of the Pd/DNA before reaction, after first, third and fifth round

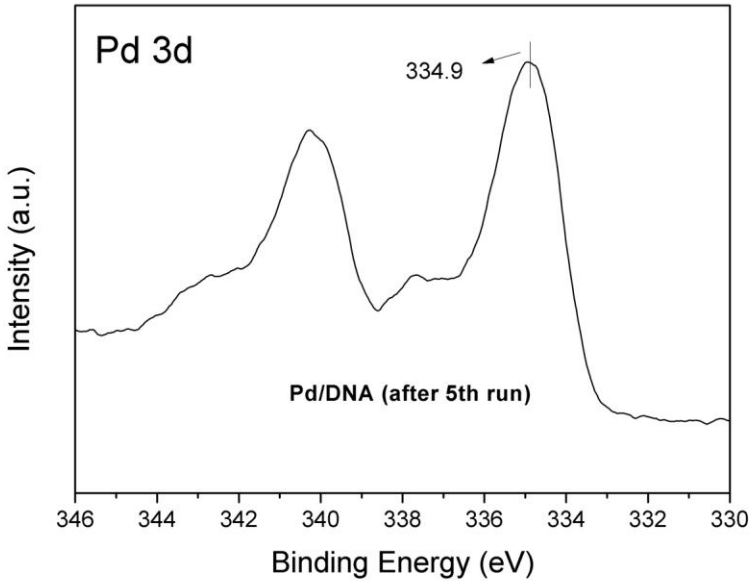


Fig. ESI-9 Pd 3d XPS analysis of Pd/DNA after fifth round

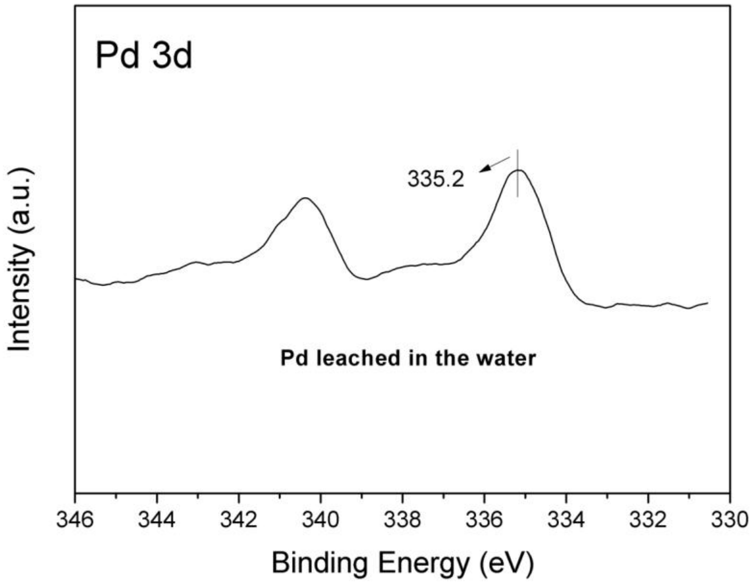


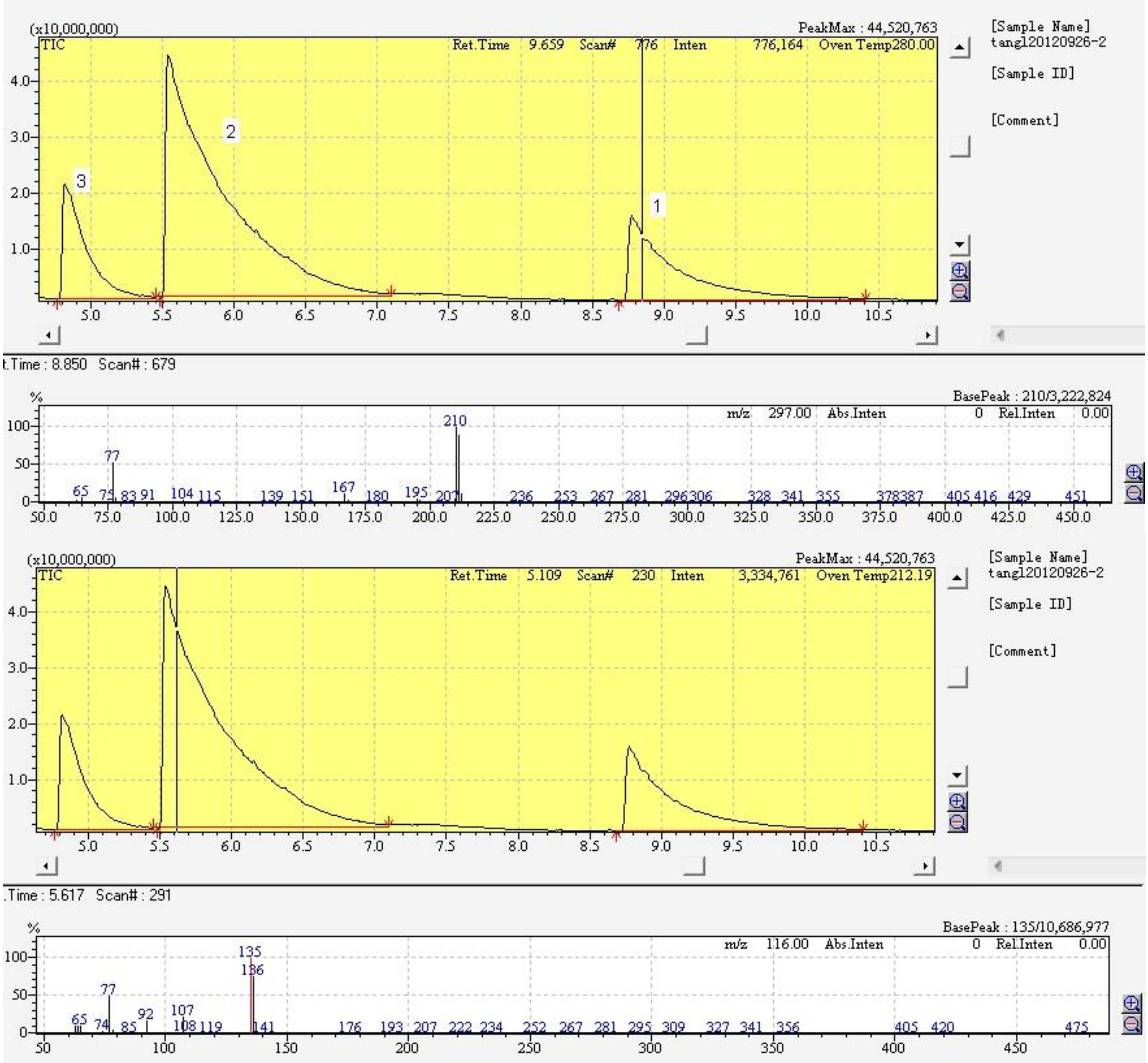
Fig. ESI-10 Pd 3d XPS analysis of Pd leached in the water after reaction

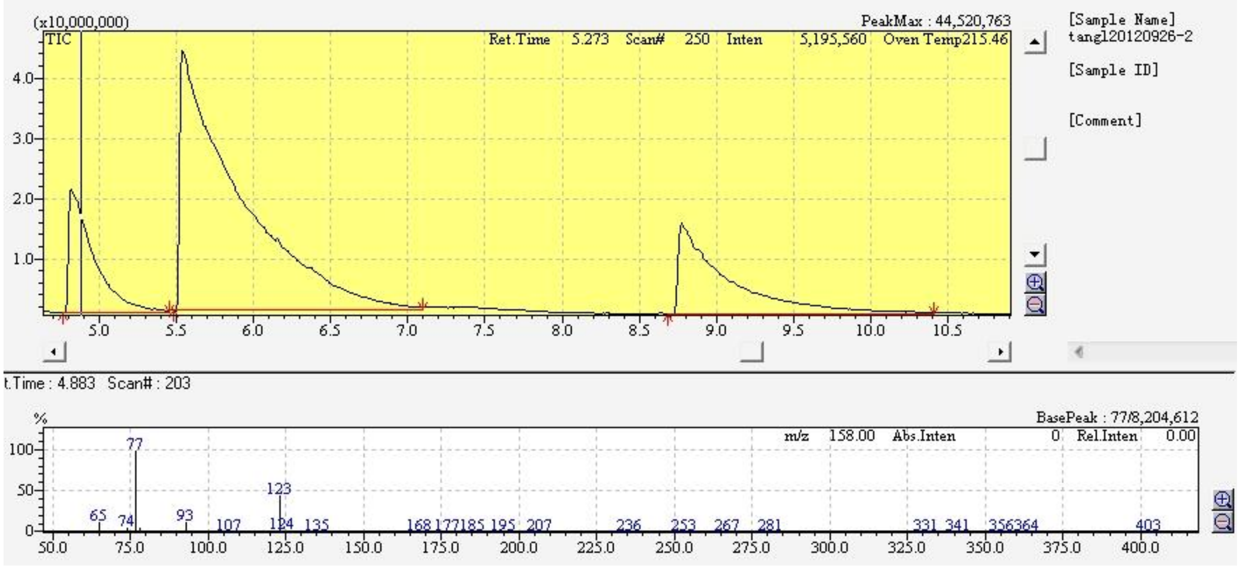
ICP-OES measurement of Pd/DNA

For the as-synthesized Pd/DNA nanohybrids (2 mL), with the aid of centrifugation at 5000 r/min for 5 minutes, then the decantate were poured out. The residues were dried by N_2 flow. And it was dissolved in aqua regia with constant volume of 50 ml. The accurate palladium content was 0.774 mg with ICP-OES measurement, after five cycles the Pd/DNA content was decreased to 0.577mg.

GC-MS analysis for the reaction of 4-methoxybenzyl alcohol and 4-methylbenzyl alcohol with nitrobenzene

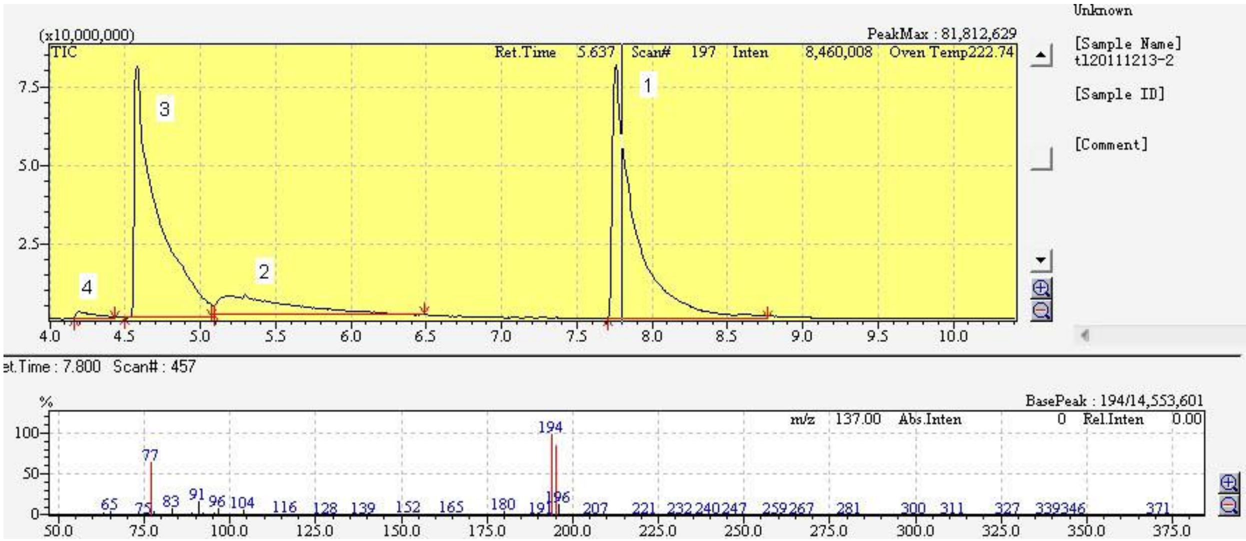
1. The GC-MS analysis for the reaction of 4-methoxybenzyl alcohol with nitrobenzene.

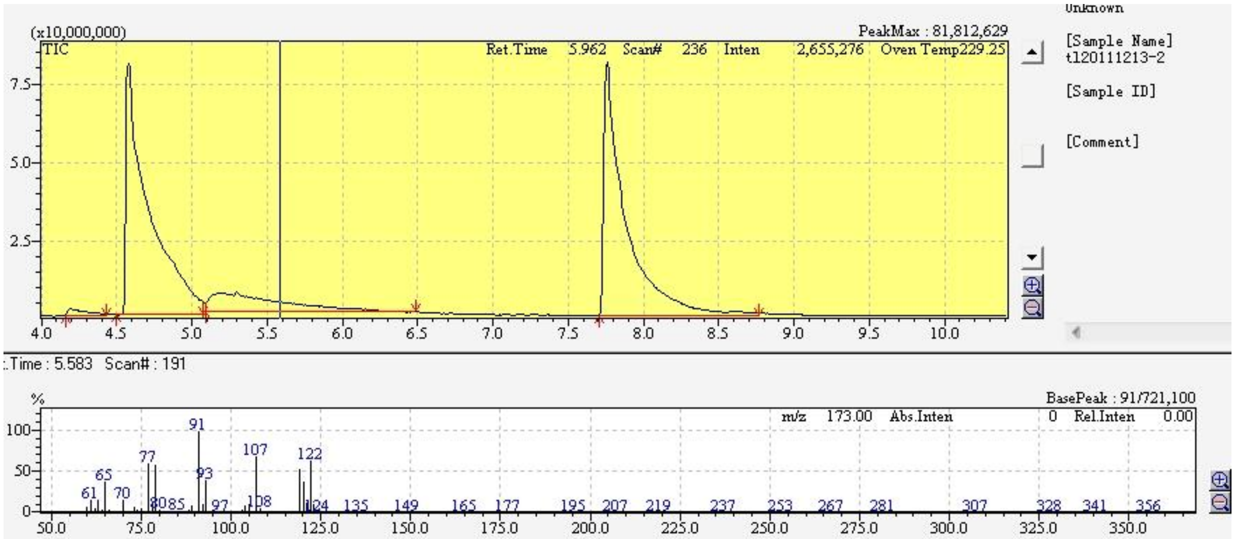


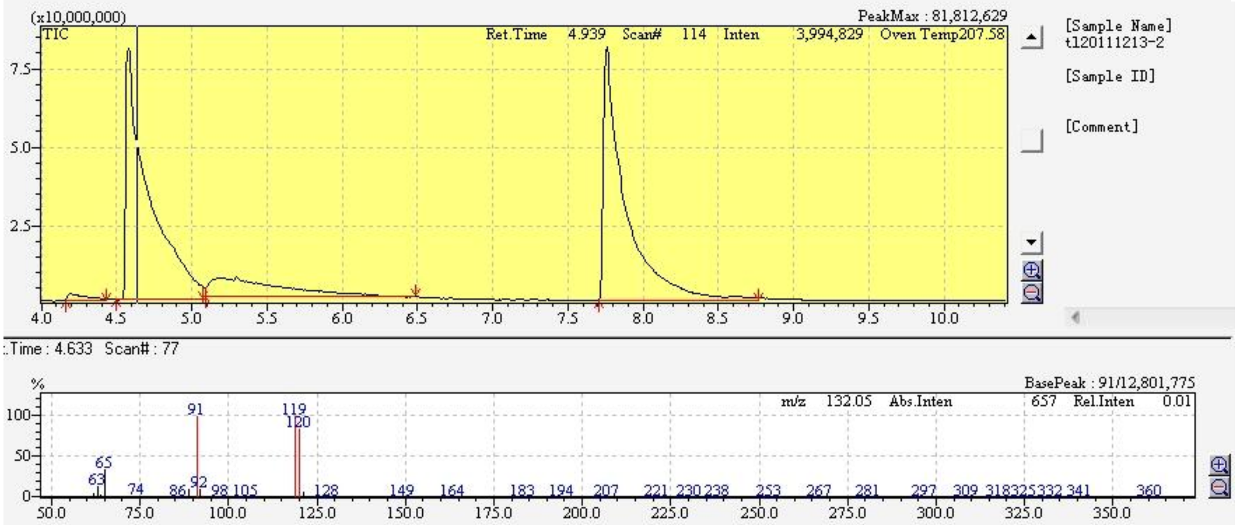


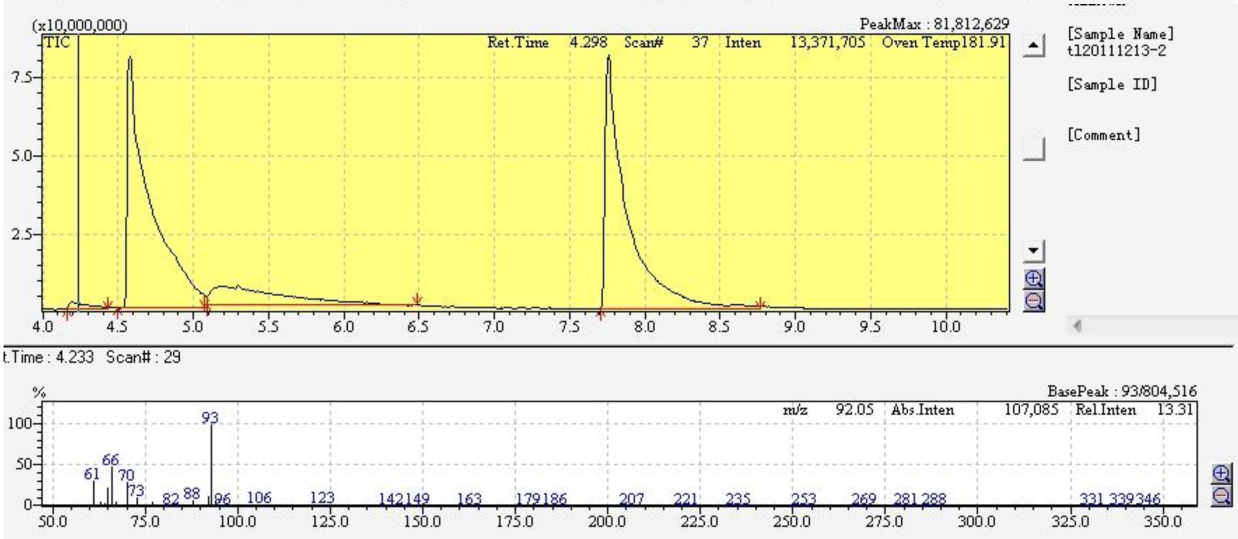
The MS analysis indicated that the peak of 1, 2 and 3 belonged to *N*-(4-methoxybenzylidene)aniline, 4-methoxy benzaldehyde and nitrobenzene respectively. This meant that 4-methoxybenzyl alcohol was almost completely converted to 4-methoxy benzaldehyde. A poor reduction of nitrobenzene into aniline was obseverd. A low yield of *N*-(4-methoxybenzylidene)aniline were observed either.

2. The GC-MS analysis for the reaction of 4-methylbenzyl alcohol with nitrobenzene.




Unknown
(x10,000,000)
TIC
PeakMax : 81,812,629
Ret.Time 5.962 Scan# 236 Inten 2,655,276 Oven Temp229.25
7.5
5.0
2.5
4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 8.0 8.5 9.0 9.5 10.0
[Sample Name]
t120111213-2
[Sample ID]
[Comment]
.Time : 5.583 Scan# : 191
%
BasePeak : 91/721,100
m/z 173.00 Abs.Inten 0 Rel.Inten 0.00
100
50
0
91
107
122
77
65
93
61
70
80 85
97
108
124 135 149 165 177 195 207 219 237 253 267 281 307 328 341 356
50.0 75.0 100.0 125.0 150.0 175.0 200.0 225.0 250.0 275.0 300.0 325.0 350.0

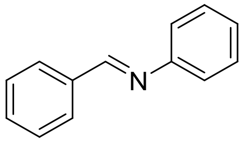

(x10,000,000)
TIC
PeakMax : 81,812,629
Ret.Time 4.939 Scan# 114 Inten 3,994,829 Oven Temp207.58
7.5
5.0
2.5
4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 8.0 8.5 9.0 9.5 10.0
[Sample Name]
t120111213-2
[Sample ID]
[Comment]
.Time : 4.633 Scan# : 77
%
BasePeak : 91/12,801,775
m/z 132.05 Abs.Inten 657 Rel.Inten 0.01
100
50
0
91
119
120
65
63
74
86
92
98 105
128 149 164 183 194 207 221 230 238 253 267 281 297 309 318325332 341 360
50.0 75.0 100.0 125.0 150.0 175.0 200.0 225.0 250.0 275.0 300.0 325.0 350.0


(x10,000,000)
TIC
PeakMax : 81,812,629
Ret.Time 4.298 Scan# 37 Inten 13,371,705 Oven Temp181.91
7.5
5.0
2.5
4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 8.0 8.5 9.0 9.5 10.0
[Sample Name]
t120111213-2
[Sample ID]
[Comment]
t.Time : 4.233 Scan# : 29
%
BasePeak : 93/804,516
m/z 92.05 Abs.Inten 107,085 Rel.Inten 13.31
100
50
0
93
66
61
70
73
82 88
96 106 123 142149 163 179186 207 221 235 253 269 281 288 331 339346
50.0 75.0 100.0 125.0 150.0 175.0 200.0 225.0 250.0 275.0 300.0 325.0 350.0

The MS analysis indicated that the peak of 1, 2 3 and 4 belong to *N*-(4-methylbenzylidene)aniline, 4-methylbenzyl alcohol, 4-methylbenzaldehyde and aniline respectively. The 4-methoxybenzyl alcohol was converted to 4-methoxy benzaldehyde with a good yield, and an excellent reduction of nitrobenzene and an excellent yield of *N*-(4-methylbenzylidene)aniline were observed.

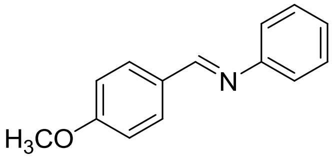
Characterization data of products

N-Benzylideneaniline (3aa)



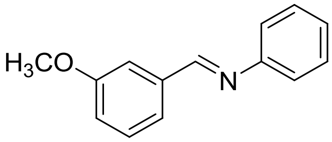
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.45 (s, 1 H), 7.92-7.89 (m, 2H), 7.47 (m, 3H), 7.39 (t, J = 9. 0 Hz, 2H), 7.23 (t, J = 9.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.4, 152.1, 136.3, 131.4, 129.1, 128.81, 128.77, 125.9, 120.9; HRMS (M+) calcd for $C_{13}H_{11}N$: 181.0891, found 181.0894.

N-(4-methoxybenzylidene)aniline (3ba)



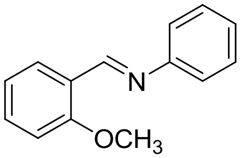
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.38 (s, 1H), 7.86-7.83 (m, 2H), 7.40-7.35 (m, 2H), 7.23-7.17 (m, 3H), 6.99-6.96 (m, 2H), 3.87 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 162.3, 159.7, 152.4, 130.5, 129.3, 129.1, 125.6, 120.9, 114.2, 55.4; HRMS (M+) calcd for $C_{14}H_{13}NO$: 211.0997 found 211.0995.

N-(3-methoxybenzylidene)aniline (3ca)



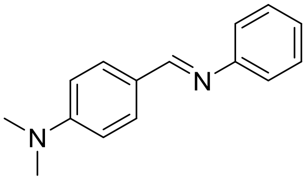
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.42 (s, 1H), 7.53 (s, 1H), 7.53-7.34 (m, 4H), 7.25-7.20 (m, 3H), 7.06-7.02 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.3, 160.1, 152.0, 137.7, 129.7, 129.2, 126.0, 122.4, 120.9, 118.4, 111.9, 55.5; HRMS (M+) calcd for $C_{14}H_{13}NO$: 211.0997 found 211.0994.

N-(2-methoxybenzylidene)aniline (3da)



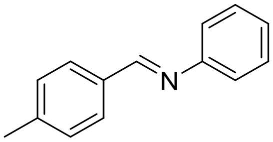
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.92 (s, 1H), 8.17-8.14 (m, 1H), 7.46-7.35 (m, 3H), 7.25-7.18 (m, 3H), 7.07-7.02 (m, 1H), 6.95 (d, J_{CF} = 6.0 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 159.5, 156.4, 152.8, 132.7, 129.0, 127.5, 125.6, 124.8, 121.1, 120.9, 111.1, 55.5; HRMS (M+) calcd for $C_{14}H_{13}NO$: 211.0997 found 211.0996.

N,N-dimethyl-4-((phenylimino)methyl)aniline (3ea)



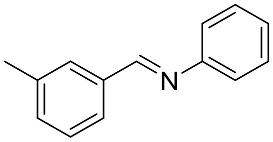
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.33 (s, 1H), 7.87 (d, J = 8.7 Hz, 2H), 7.37 (t, J = 7.5 Hz, 2H), 7.20 (d, J = 7.5 Hz, 3H), 6.74 (d, J = 8.7 Hz, 2H), 3.06 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.3, 153.1, 152.7, 130.6, 129.2, 125.1, 124.7, 121.1, 111.7, 40.3; HRMS (M+) calcd for $C_{15}H_{16}N_2$: 224.1313 found 224.1319.

N-(4-methylbenzylidene)aniline (3fa)



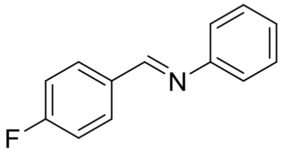
This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (2% Et_3N) v/v) to give the product as a white solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.41 (s, 1H), 7.79 (d, J = 8.0 Hz, 2H), 7.40-7.35 (m, 2H), 7.27 (d, J = 7.9 Hz, 2H), 7.20 (d, J = 8.0 Hz, 3H), 2.41 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.3, 152.3, 141.9, 133.7, 129.5, 129.1, 128.8, 125.7, 120.9, 21.6; HRMS (M+) calcd for $C_{14}H_{13}N$: 195.1048 found 195.1047.

N-(3-methylbenzylidene)aniline (3ga)



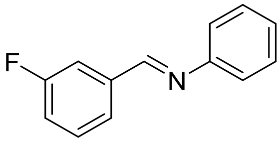
This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (2% Et_3N) v/v) to give the product as a yellow oil. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.39 (s, 1H), 7.74 (s, 1H), 7.64 (d, J = 8.5 Hz, 1H), 7.39-7.33 (m, 3H), 7.31-7.25 (m, 1H), 7.22-7.18 (m, 3H), 2.39 (s, 3H); 13C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.6, 152.2, 138.6, 136.2, 132.3, 129.1, 129.0, 128.7, 126.4, 125.9, 120.9, 21.3; HRMS (M+) calcd for $C_{14}H_{13}N$: 195.1048 found 195.1045.

N-(4-fluorobenzylidene)aniline (3ia)



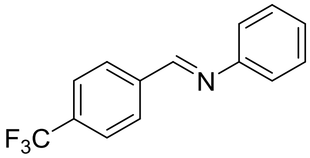
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.42 (s, 1H), 7.94-7.87 (m, 2H), 7.43-7.36 (m, 2H), 7.24-7.12 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 164.7 (d, J_{CF} = 250.5 Hz), 158.8, 151.9, 132.6 (d, J_{CF} = 2.8 Hz), 130.8 (d, J_{CF} = 8.7 Hz), 129.2, 126.0, 120.8, 115.9 (d, J_{CF} = 21.8 Hz); HRMS (M+) calcd for $C_{13}H_{10}FN$: 199.0797 found 199.0795.

N-(3-fluorobenzylidene)aniline (3ja)



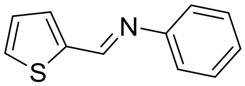
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a yellow oil. ^1H NMR (300 MHz, d^6-acetone): δ [ppm] = 8.61 (s, 1H), 7.79-7.73 (m, 2H), 7.60-7.53 (m, 1H), 7.45-7.40 (m, 2H), 7.33-7.24 (m, 4H); ^{13}C NMR (75 MHz, d^6-acetone): δ [ppm] = 164.7 (d, J_{CF} = 243 Hz), 160.6 (d, J_{CF} = 3.0 Hz), 153.3, 140.9 (d, J_{CF} = 7.8 Hz), 132.4 (d, J_{CF} = 7.8Hz), 130.9, 127.9, 126.9 (d, J_{CF} = 3.0 Hz), 122.7, 119.7 (d, J_{CF} = 21.7 Hz), 115.9 (d, J_{CF} = 22.5 Hz); HRMS (M+) calcd for $C_{13}H_{10}FN$: 199.0797 found 199.0801.

N-(4-(trifluoromethyl)benzylidene)aniline (3ka)



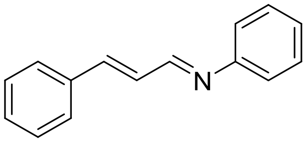
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] =8.51 (s, 1H), 8.02 (d, *J* = 6.0 Hz, 2H), 7.73 (d, *J* = 6.0 Hz, 2H), 7.44-7.39 (m, 2H), 7.29-7.22 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 158.5, 151.4, 139.3, 132.1 (q, J_{CF} = 32.3 Hz), 129.9, 129.3, 129.0, 126.6, 125.7 (q, J_{CF} = 3.8 Hz), 122.1 (q, J_{CF} = 270 Hz), 120.9; HRMS (M+) calcd for $C_{14}H_{10}F_3N$: 249.0765 found 249.0771.

N-(Thiophen-2-ylmethylene)aniline (3la)



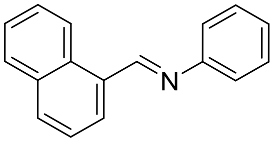
This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (1% Et_3N) v/v) to give the product as a yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.55 (s, 1H), 7.51-7.47 (m, 2H), 7.40-7.35 (m, 2H), 7.23-7.19 (m, 3H), 7.13-7.11 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 153.0, 151.4, 142.8, 132.2, 130.3, 129.1, 127.7, 126.1, 121.0; HRMS (M+) calcd for $C_{11}H_9NS$: 187.0456 found 187.0459.

N-(3-Phenylallylidene)aniline (3ma)



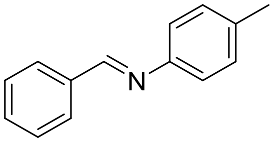
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.27-8.25 (m, 1H), 7.55-7.52 (m, 2H), 7.42-7.32 (m, 5H), 7.19 (t, *J* = 6.0 Hz, 3H), 7.14-7.12 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 161.6, 151.8, 144.0, 135.6, 129.6, 129.2, 128.9, 128.7, 127.5, 126.1, 120.9; HRMS (M+) calcd for $C_{15}H_{13}N$: 207.1048 found 207.1044.

N-(naphthalen-1-ylmethylene)aniline (3na)



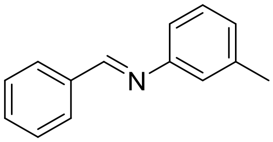
This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (2% Et_3N) v/v) to give the product as a light yellow solid. ^1H NMR (300 MHz, d^6-acetone): δ [ppm] = 9.32 (d, *J* = 9 Hz, 1H), 9.18 (s, 1H), 8.17 (d, *J* = 9 Hz, 1H), 8.07 (d, *J* = 8.3 Hz, 1H), 8.01 (d, *J* = 7.6 Hz, 1H), 7.68-7.57 (m, 3H), 7.47-7.42 (m, 2H), 7.37-7.34 (m, 2H), 7.29-7.24 (m, 1H); ^{13}C NMR (75 MHz, d^6-acetone):δ [ppm] = 162.0, 154.4, 135.8, 133.6, 133.3, 133.2, 132.3, 130.9, 130.4, 129.1, 128.0, 127.6, 127.0, 126.5, 122.7; HRMS (M+) calcd for $C_{17}H_{13}N$: 231.1048 found 231.1050.

N-benzylidene-4-methylaniline (3ab)



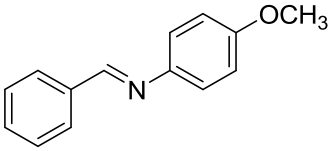
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.46 (s, 1H), 7.91-7.88 (m, 2H), 7.46 (m, 3H), 7.21-7.12 (m, 4H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 159.6, 149.5, 136.4, 135.8, 131.2, 129.8, 128.74, 128.72, 120.8, 21.0; HRMS (M+) calcd for $C_{14}H_{13}N$: 195.1048, found 195.1047.

N-benzylidene-3-methylaniline (3ac)



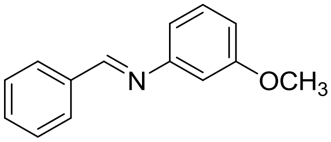
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a faint yellow oil. ^1H NMR (300 MHz, d^6-acetone): δ [ppm] = 8.57 (s,1H), 7.98-7.95 (m, 2H), 7.54-7.49 (m, 3H), 7.31-7.25 (m, 1H), 7.07-7.04 (m, 3H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, d^6-acetone):δ [ppm] = 161.6, 153.9, 140.5, 138.4, 132.9, 130.61, 130.38, 130.33, 128.2, 123.2, 119.6, 22.2; HRMS (M+) calcd for $C_{14}H_{13}N$: 195.1048, found 195.1046.

N-benzylidene-4-methoxyaniline (3ae)



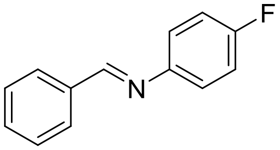
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.48 (s, 1H), 7.90-7.87 (m, 2H), 7.47-7.45 (m,3H), 7.52-7.21 (m, 2H), 6.95-6.92 (m, 2H), 3.83 (s,3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 158.4, 158.3, 144.9, 136.4, 131.0, 128.7, 128.6, 122.2, 114.4, 55.5; HRMS (M+) calcd for $C_{14}H_{13}NO$: 211.0997, found 211.0996.

N-benzylidene-3-methoxyaniline (3af)



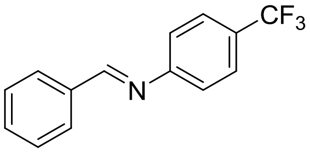
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ [ppm] = 8.47 (s, 1H), 7.92-7.90 (m, 2H), 7.50-7.48 (m, 3H), 7.30 (t, J = 8.0 Hz, 1H), 6.82-6.79 (m, 3H), 3.85 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.6, 160.5, 153.6, 136.3, 131.5, 130.0, 129.0, 128.9,113.0, 112.0,106.8, 55.4; HRMS (M+) calcd for $C_{14}H_{13}NO$: 211.0997, found 211.0990.

N-benzylidene-4-fluoroaniline (3ah)



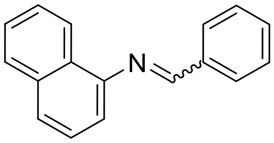
This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (2% Et_3N) v/v) to give the product as a faint yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.44 (s, 1H), 7.89 (s, 2H), 7.48 (s, 3H), 7.25-7.20 (m, 2H), 7.09-7.08 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 161.3 (d, J_{CF} = 243.0 Hz), 160.2, 148.05 (d, J_{CF} = 3 Hz), 136.1, 131.5, 128.80, 128.78, 122.3 (d, J_{CF} = 8.3 Hz), 115.9 (d, J_{CF} = 22.5 Hz); HRMS (M+) calcd for $C_{13}H_{10}FN$: 199.0797, found 199.0798.

N-benzylidene-4-trifluoromethylaniline (3ai)



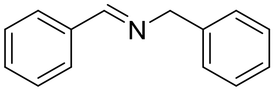
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.42 (s, 1H), 7.92-7.89 (m, 2H), 7.64 (d, J = 8.3 Hz, 2H), 7.53-7.45 (m, 3H), 7.24 (d, J = 8.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 161.9, 155.3, 135.7, 132.0, 129.1, 128.9, 127.7 (q, J_{CF} = 32.3 Hz), 126.4 (q, J_{CF} = 3.8 Hz), 124.3 (q, J_{CF} = 270 Hz), 121.0; HRMS (M+) calcd for $C_{14}H_{10}F_3N$: 249.0765 found 249.0766.

N-benzylidenenaphthalen-1-amine (3aj)



This compound was prepare according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc = 10:1 (2% Et_3N) v/v) to give the product as a light yellow solid. ^{1}H NMR (300 MHz, d^6-acetone): δ [ppm] = 8.68 (s) and 8.67 (s, E and Z, 1H), 8.39-8.36 (m, 1H), 8.11-8.08 (m, 2H), 7.93-7.90 (m, 1H), 7.78 (d, J = 9 Hz, 1H), 7.57-7.48 (m, 6H), 7.18 (d, 7.3 Hz, 1H); ^{13}C NMR (75 MHz, d^6-acetone): δ [ppm] = 162.2, 150.9, 138.5, 135.9, 133.2, 130.8, 130.64, 130.55, 129.4, 128.1, 127.9, 127.44, 127.36, 125.5, 114.4; HRMS (M+) calcd for $C_{17}H_{13}N$: 231.1048 found 231.1047.

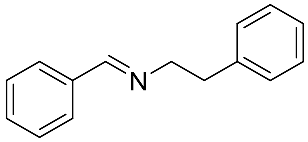
N-benzylidenebenzylamine (3ak)



This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.38 (s, 1H), 7.79-7.76 (m, 2H), 7.42-7.38 (m, 3H), 7.34-7.33 (m, 4H),7.30-7.23 (m, 1H), 4.81 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 162.0, 139.3, 136.2, 130.7, 128.59, 128.49, 128.28, 127.98, 127.0, 65.0; HRMS

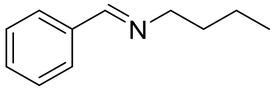
(M+) calcd for $C_{14}H_{13}N$: 195.1048, found 195.1045.

N-benzylidene-2-phenylethanamine (3al)



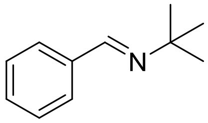
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a colourless oil. ^{1}H NMR (400 MHz, d^{6}-acetone): δ [ppm] = 8.27 (s, 1H), 7.79 (t, *J* = 1.6 Hz, 2H), 7.46 (d, *J* = 5.2 Hz, 3H), 7.30 (d, *J* = 4.0 Hz, 4H), 7.22-7.18 (m, 1H), 3.88 (t, *J* = 7.2 Hz, 2H), 3.01 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, d^{6}-acetone): δ [ppm] = 161.1, 140.3, 136.8, 130.5, 129.1, 128.6, 128.3, 128.1, 126.0, 62.7, 37.3; HRMS (M+) calcd for $C_{15}H_{15}N$: 209.1204, found 209.1211.

N-benzylidenebutan-1-amine (3am)



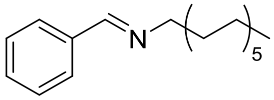
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a yellow oil. ^{1}H NMR (300 MHz, d^{6}-acetone): δ [ppm] = 8.36 (s, 1H), 7.82-7.79 (m, 2H), 7.47 (t, *J* = 4.0 Hz, 3H), 3.62 (t, *J* = 9.2 Hz, 2H), 1.73-1.63 (m, 2H), 1.49-1.36 (m, 2H), 0.97 (t, *J* = 10.0 Hz, 3H); ^{13}C NMR (75 MHz, d^{6}-acetone): δ [ppm] = 160.4, 136.8, 130.4, 128.6, 128.0, 60.9, 33.0, 20.2, 13.3; HRMS (M+) calcd for $C_{11}H_{15}N$: 161.1204, found 161.1209.

N-benzylidenetertbutylamine (3an)



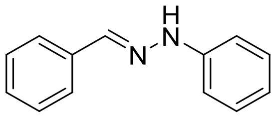
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.27 (s, 1H), 7.76-7.73 (m, 2H), 7.40-7.37 (m, 3H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 155.1, 137.2, 130.1, 128.5, 127.9, 57.2, 29.7; HRMS (M+) calcd for $C_{11}H_{15}N$: 161.1204 found 161.1202.

N-benzylidenedodecylamine (3ap)



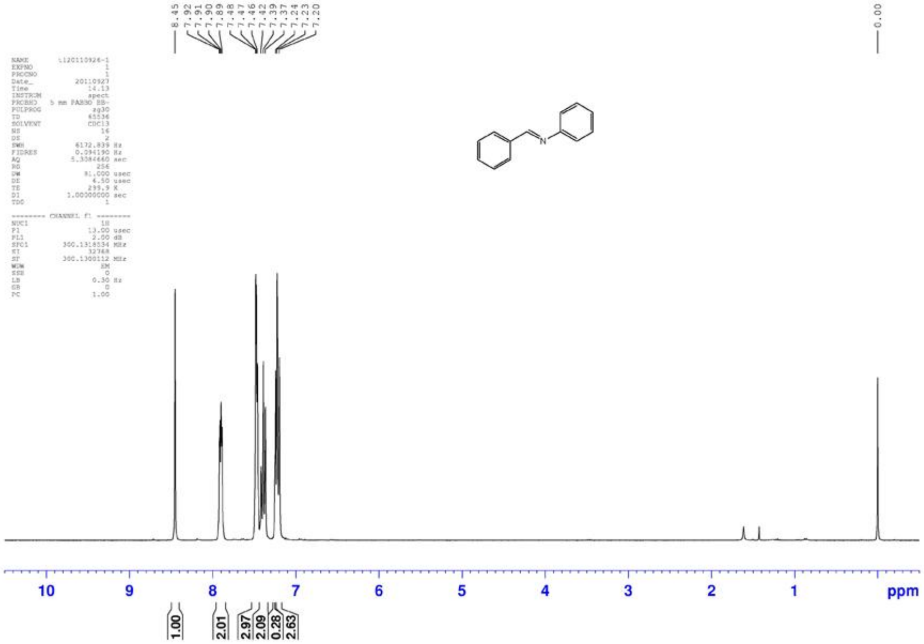
This compound was prepared according to the general procedure and purified by vacuum distillation to give the product as a colourless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 8.27 (s, 1H), 7.72 (d, *J* =3.0 Hz, 2H), 7.40 (t, *J* = 3.0 Hz, 3H), 3.60 (t, *J* = 6.0Hz, 2H), 1.69 (t, *J* = 6.0 Hz, 2H), 1.29 (d, *J* = 20.6 Hz, 18H), 0.88 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 160.6, 136.4, 130.4, 128.5, 128.0, 61.8, 31.9, 31.0, 29.68, 29.64, 29.61, 29.47, 29.35, 27.4, 22.7, 14.1; HRMS (M+) calcd for $C_{19}H_{31}N$: 273.2457 found 273.2461.

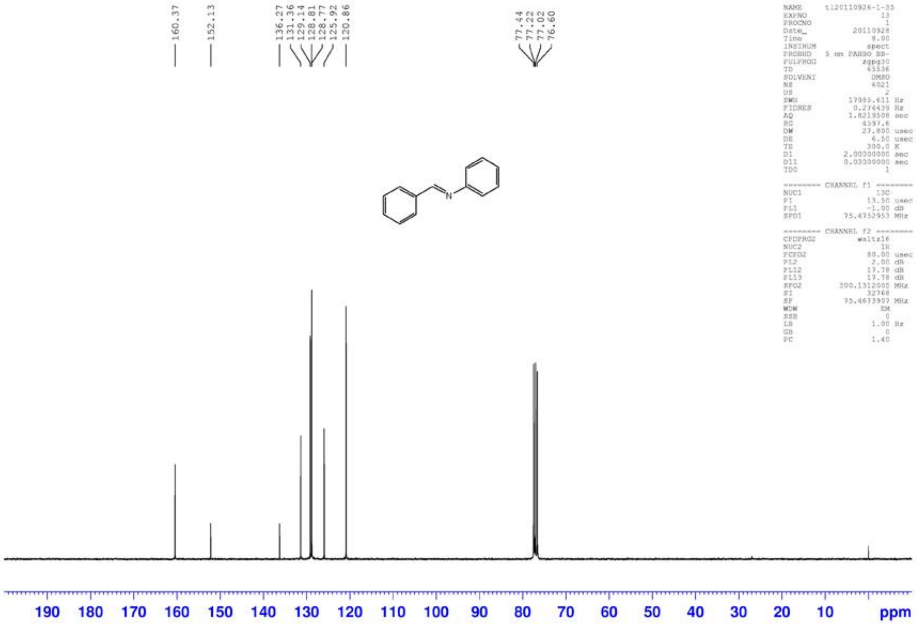
1-benzylidene-2-phenylhydrazine (3aq)

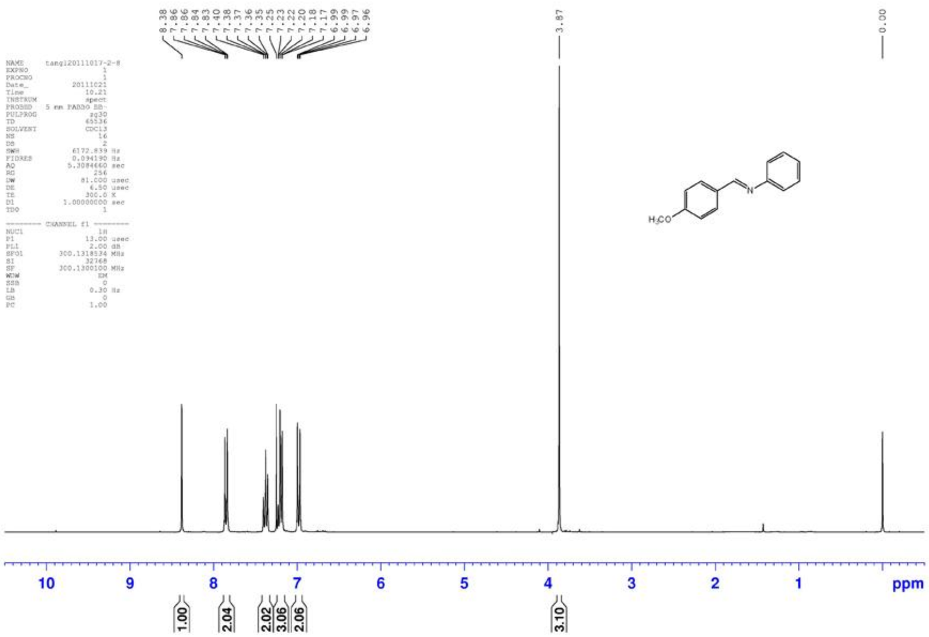


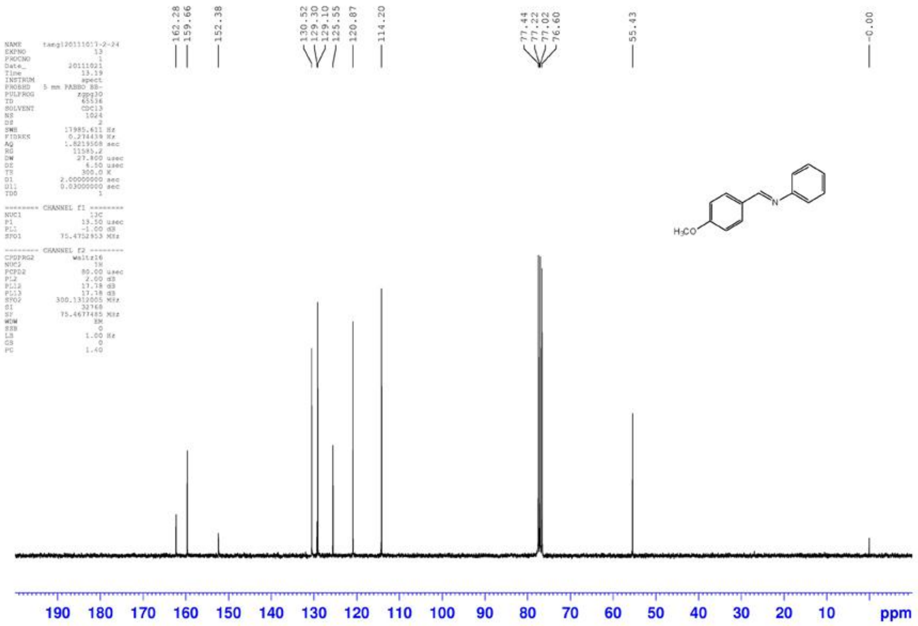
This compound was prepared according to the general procedure and purified by recrystallization with Et_2O/hexane to give the product as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ [ppm] = 7.67-7.64 (m, 3H), 7.39-7.31 (m, 2H), 7.30-7.24 (m, 3H), 7.12-7.10 (m, 2H), 6.89-6.84 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 144.7, 137.3, 135.3, 129.3, 128.6, 128.4, 126.2, 120.1, 112.8; HRMS (M+) calcd for $C_{13}H_{12}N_2$: 196.1000 found 196.1011.

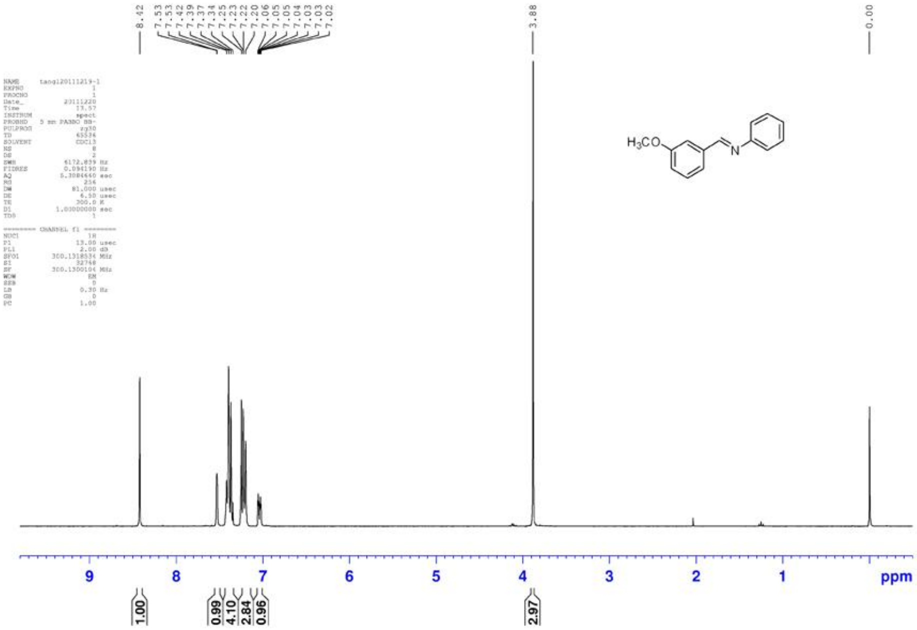
NMR Spectra of products

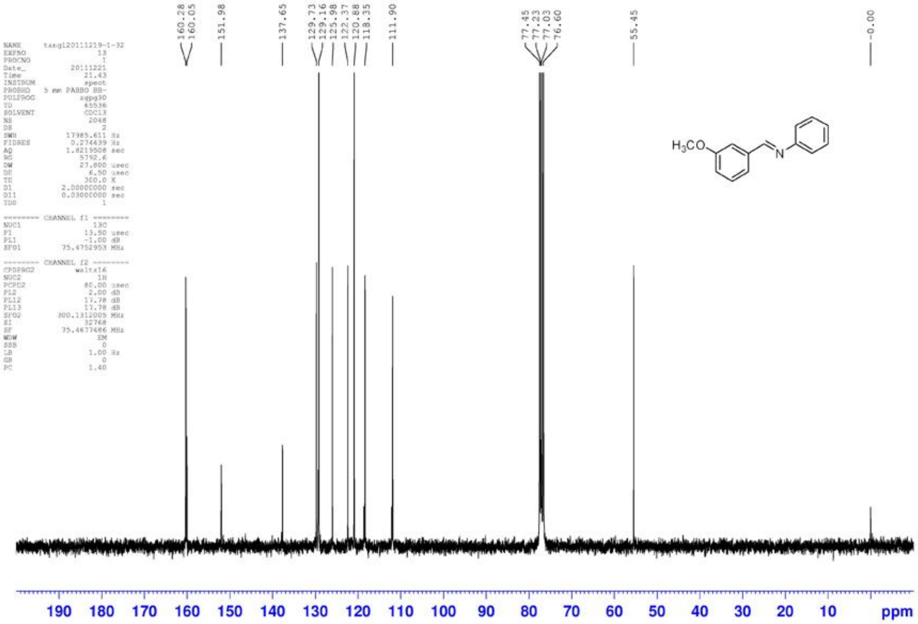


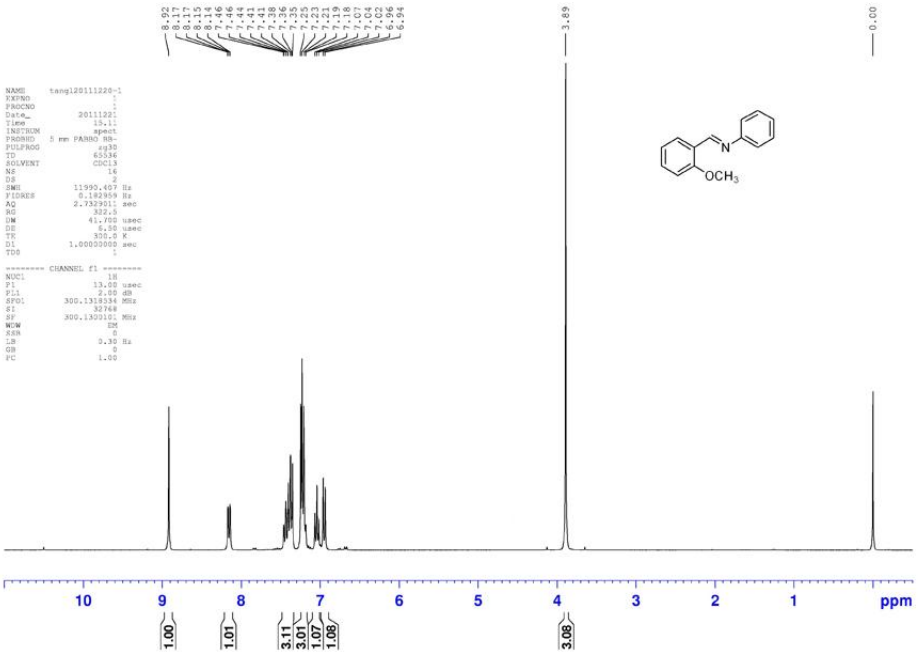


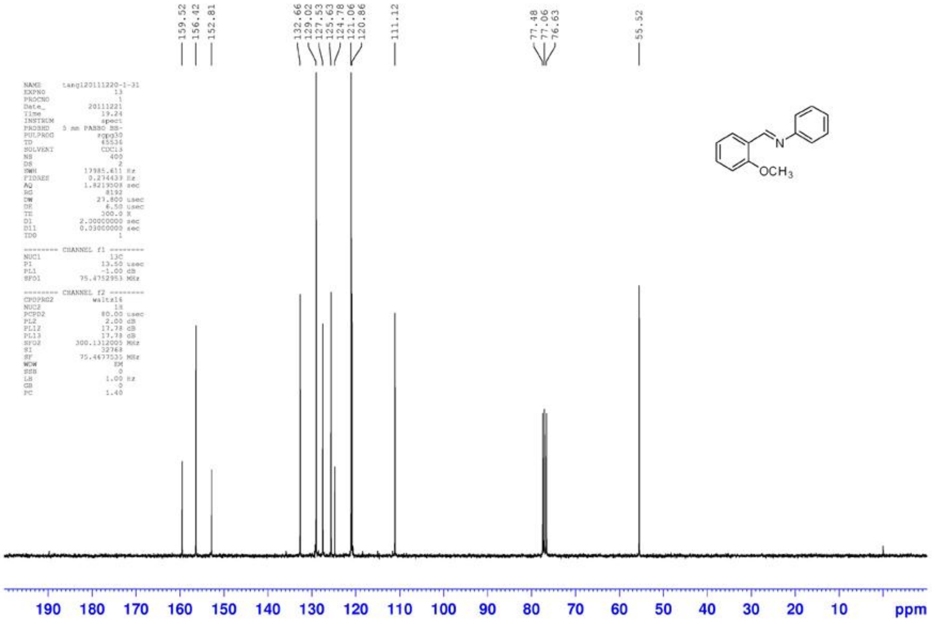

8.38
7.86
7.84
7.83
7.40
7.38
7.37
7.36
7.35
7.25
7.23
7.22
7.20
7.18
7.17
6.99
6.97
6.96
3.87
0.00
NAME tang120111017-2-8
EXPNO 1
PROCNO 1
Date_ 20111021
Time 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3CO
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.04
2.02
3.06
2.06
3.10

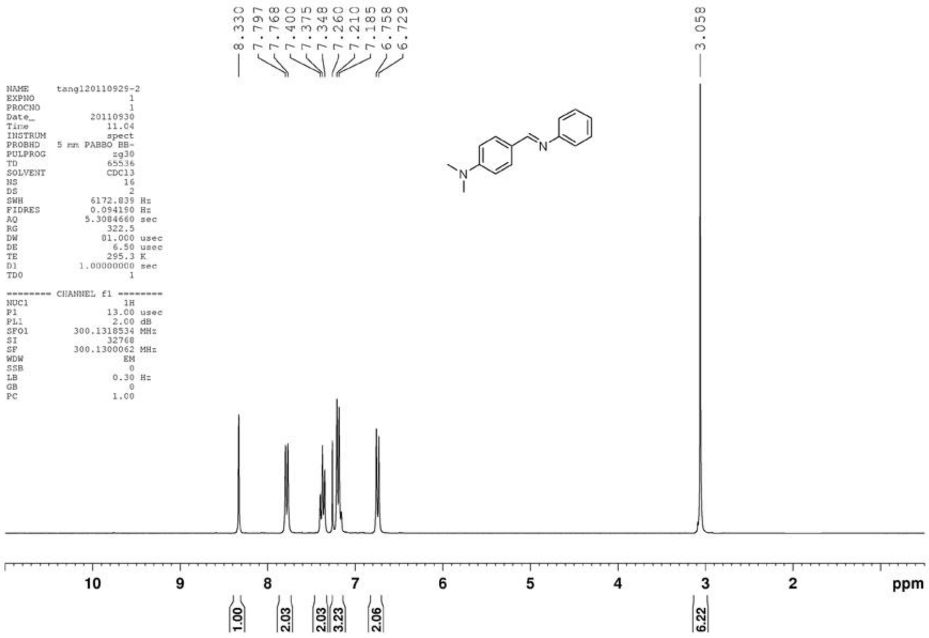

162.28
159.66
152.38
130.52
129.30
129.10
125.55
120.87
114.20
77.44
77.22
77.02
76.60
55.43
-0.00
NAME tang120111017-2-24
EXPNO 13
PROCNO 1
Date_ 20111021
Time 13.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 11585.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3CO
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.42
7.53
7.53
7.42
7.39
7.37
7.34
7.25
7.23
7.22
7.20
7.06
7.05
7.05
7.04
7.03
7.03
7.02
3.88
0.00
H_3CO
N
9
8
7
6
5
4
3
2
1
ppm
1.00
0.99
4.10
2.84
0.96
2.97

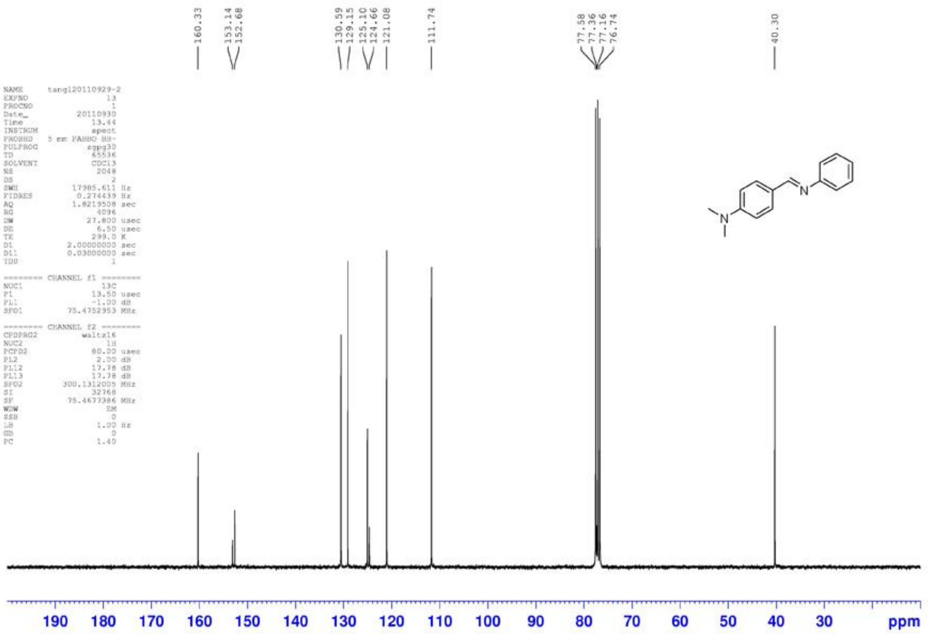

160.28
160.05
151.98
137.65
129.73
129.16
125.98
122.37
120.88
118.35
111.90
77.45
77.23
77.03
76.60
55.45
-0.00
H_3CO
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.92
8.17
8.17
8.15
8.14
7.46
7.44
7.41
7.41
7.38
7.36
7.35
7.25
7.23
7.21
7.19
7.18
7.07
7.04
7.02
6.96
6.94
3.89
0.00
NAME tangl20111220-1
EXPNO 1
PROCNO 1
Date_ 20111221
Time 15.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 322.5
DW 41.700 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
OCH3
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
3.11
3.01
1.07
1.08
3.08

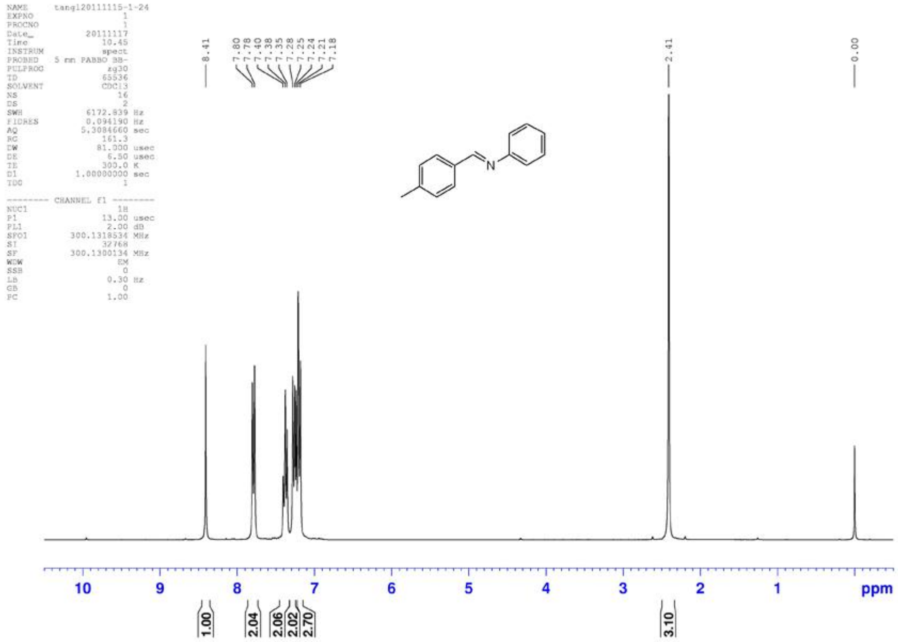

159.52
156.42
152.81
132.66
129.02
127.53
125.63
124.78
121.06
120.86
111.12
77.48
77.06
76.63
55.52
NAME tangl20111220-1-31
EXPNO 13
PROCNO 1
Date_ 20111221
Time 19.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
OCH3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

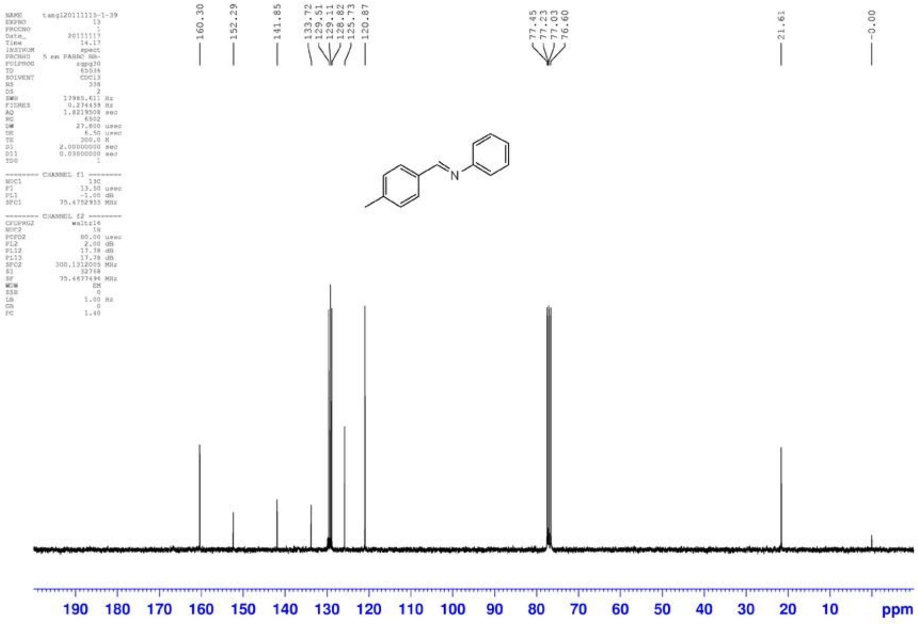


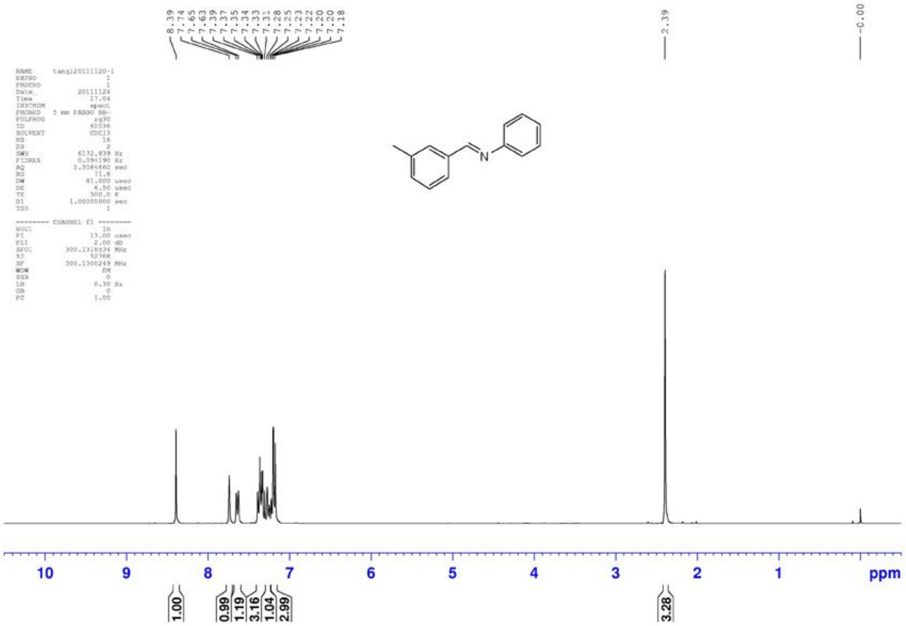
```
NAME     tangl20110929-2
EXPNO                  1
PROCNO                 1
Date_           20110930
Time               11.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ             5.3084660 sec
RG                 322.5
DW                81.000 usec
DE                  6.50 usec
TE                 295.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                 2.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300062 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

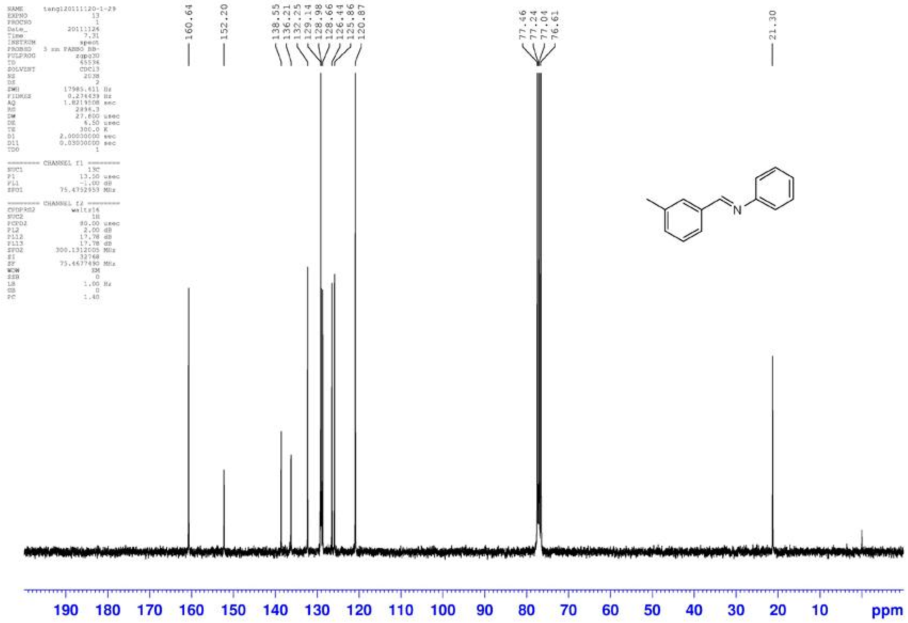


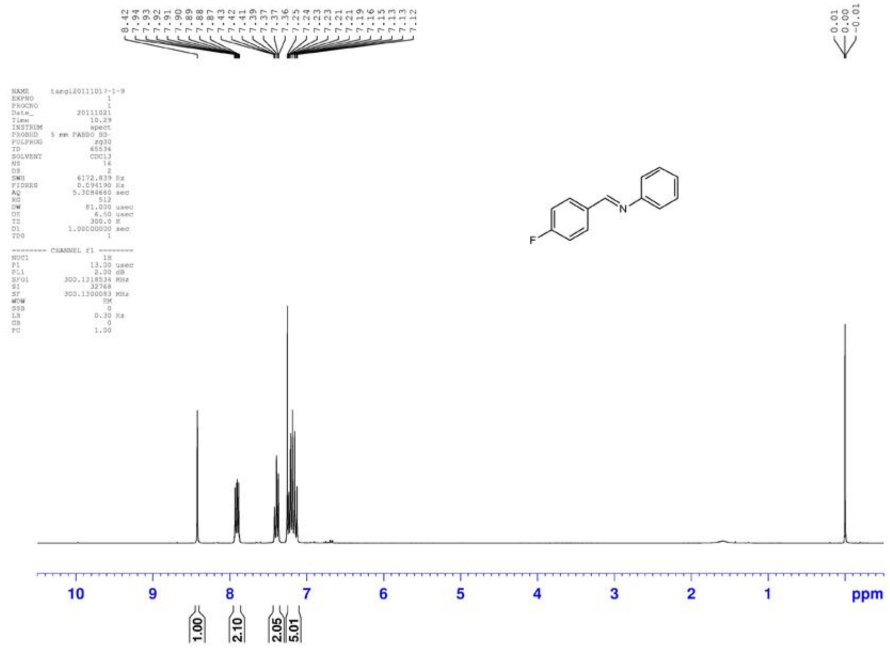
```
NAME     tangl20110929-2
EXPNO                 13
PROCNO                 1
Date_           20110930
Time               13.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     2
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                  4096
DW                27.800 usec
DE                  6.50 usec
TE                 299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 13.50 usec
PL1                -1.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 2.00 dB
PL12               17.78 dB
PL13               17.78 dB
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677386 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

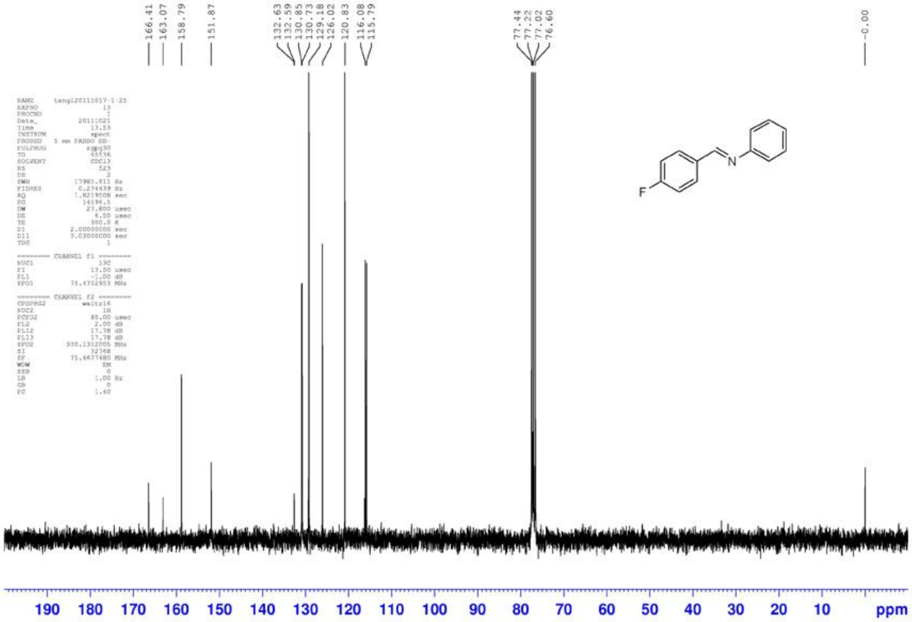

8.41
7.80
7.78
7.40
7.38
7.35
7.28
7.25
7.24
7.21
7.18
2.41
0.00
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.04
2.06
2.02
2.70
3.10

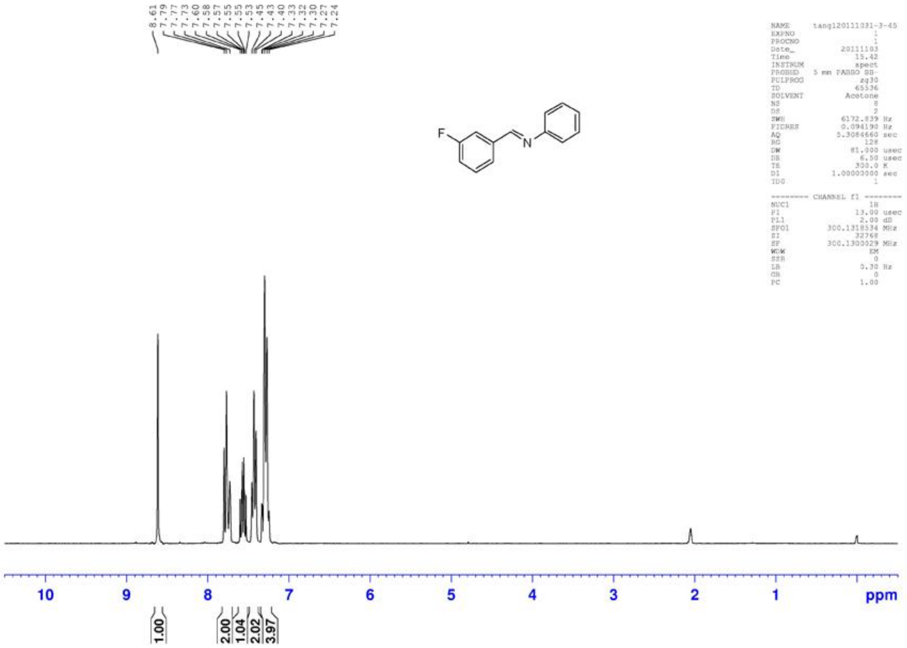

160.30
152.29
141.85
133.72
129.51
129.11
128.82
125.73
120.87
77.45
77.23
77.03
76.60
21.61
0.00
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

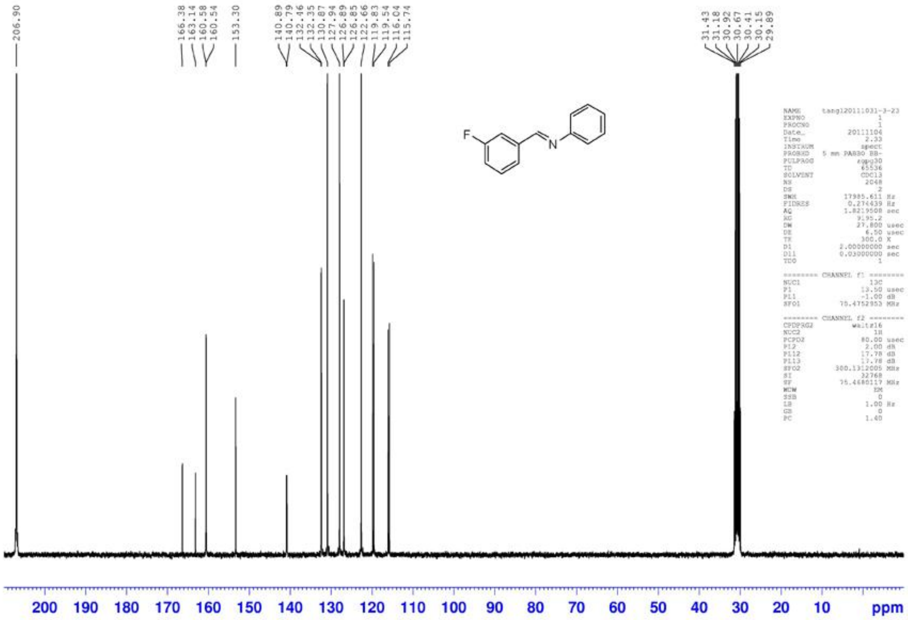

8.39
7.74
7.65
7.63
7.39
7.37
7.35
7.34
7.33
7.31
7.28
7.25
7.23
7.22
7.20
7.20
7.18
2.39
-0.00
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.99
1.19
3.16
1.04
2.99
3.28

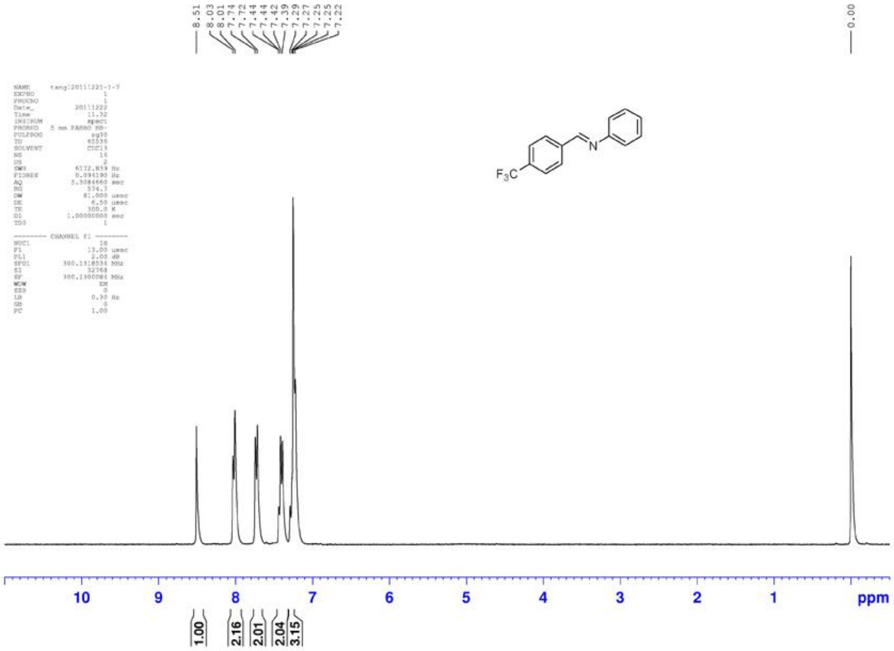

160.64
152.20
138.55
136.21
132.25
129.14
128.98
128.66
126.44
125.86
120.87
77.46
77.24
77.04
76.61
21.30
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

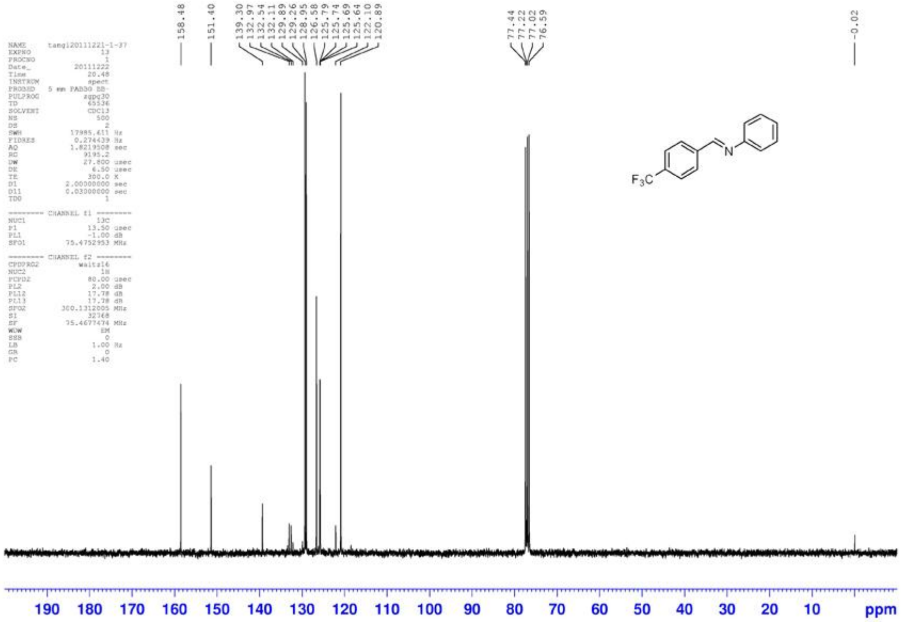

8.42
7.94
7.93
7.92
7.91
7.90
7.89
7.88
7.87
7.43
7.42
7.41
7.39
7.37
7.37
7.36
7.25
7.24
7.23
7.23
7.21
7.21
7.19
7.16
7.15
7.13
7.13
7.12
0.01
0.00
-0.01
F
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.10
2.05
5.01

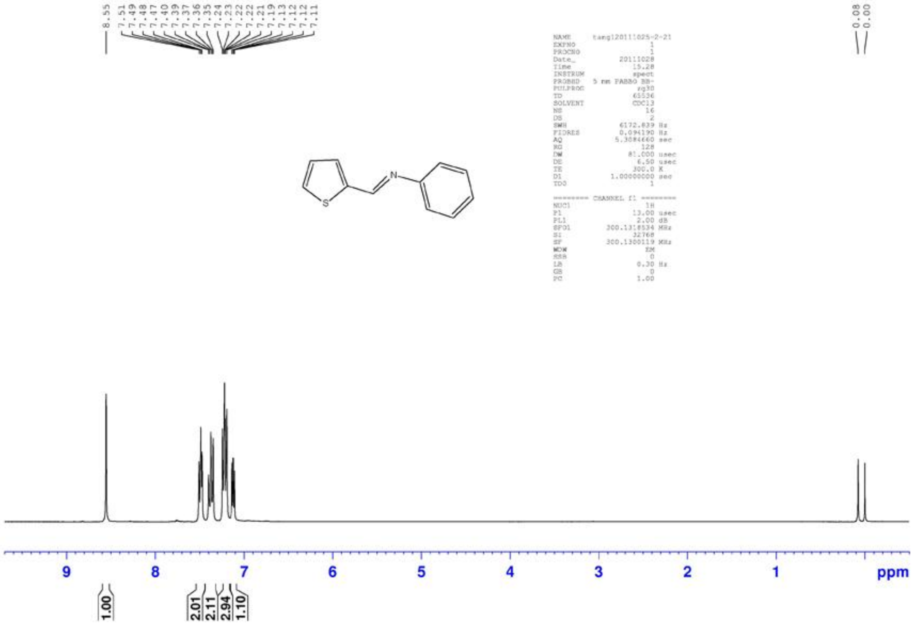

166.41
163.07
158.79
151.87
132.63
132.59
130.85
130.73
129.18
126.02
120.83
116.08
115.79
77.44
77.22
77.02
76.60
-0.00
F
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

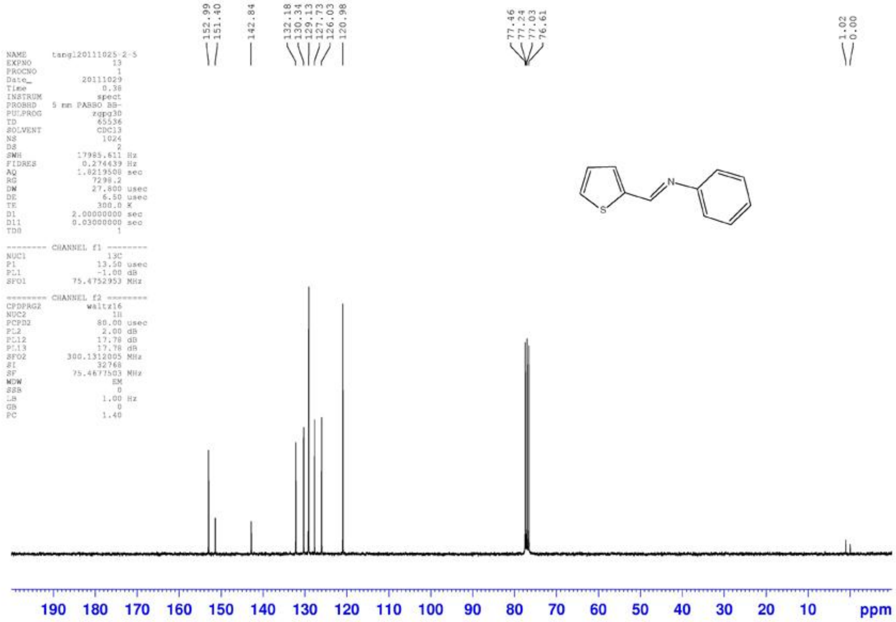

8.61
7.79
7.77
7.73
7.60
7.58
7.57
7.55
7.55
7.53
7.45
7.43
7.40
7.33
7.32
7.30
7.27
7.24
F
N
NAME tang120111031-3-45
EXPNO 1
PROCNO 1
Date_ 20111103
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300029 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
1.00
2.00
1.04
2.02
3.97

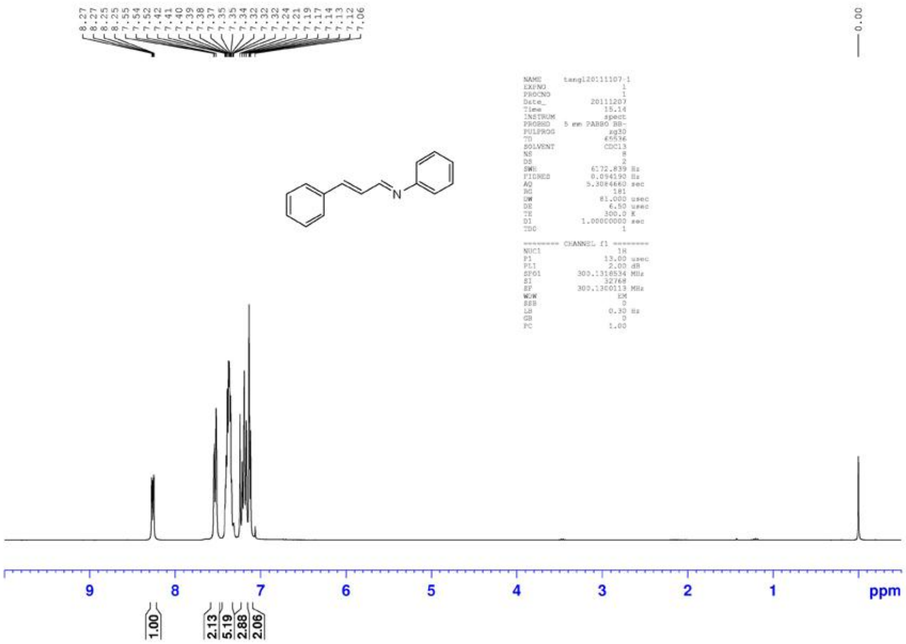

206.90
166.38
163.14
160.58
160.54
153.30
140.89
140.79
132.46
132.35
130.87
127.94
126.89
126.85
122.66
119.83
119.54
116.04
115.74
31.43
31.18
30.92
30.67
30.41
30.15
29.89
F
N
NAME tang120111031-3-23
EXPNO 1
PROCNO 1
Date_ 20111104
Time 2.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 9195.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4680117 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

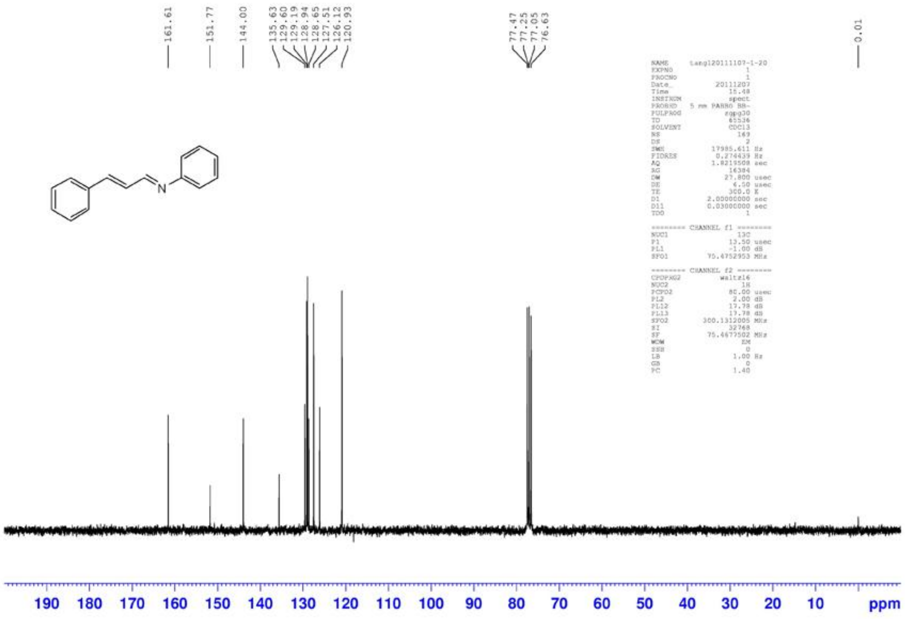

8.51
8.03
8.01
7.74
7.72
7.44
7.44
7.42
7.39
7.29
7.27
7.25
7.22
0.00
F_3C
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.16
2.01
2.04
3.15

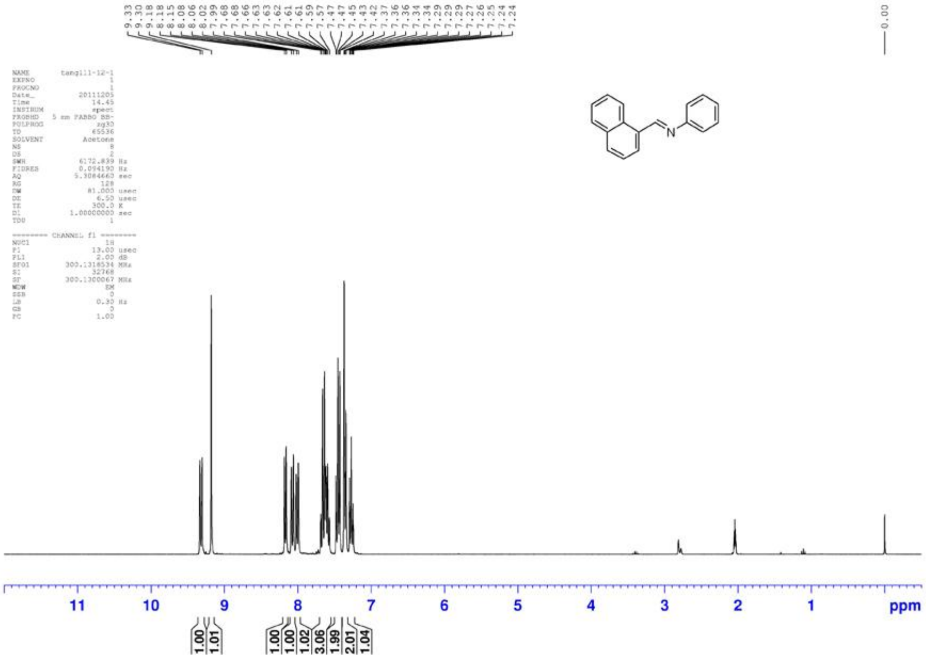

158.48
151.40
139.30
132.97
132.54
132.11
129.89
129.26
128.95
126.58
125.79
125.74
125.69
125.64
122.10
120.89
77.44
77.22
77.02
76.59
-0.02
F_3C
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

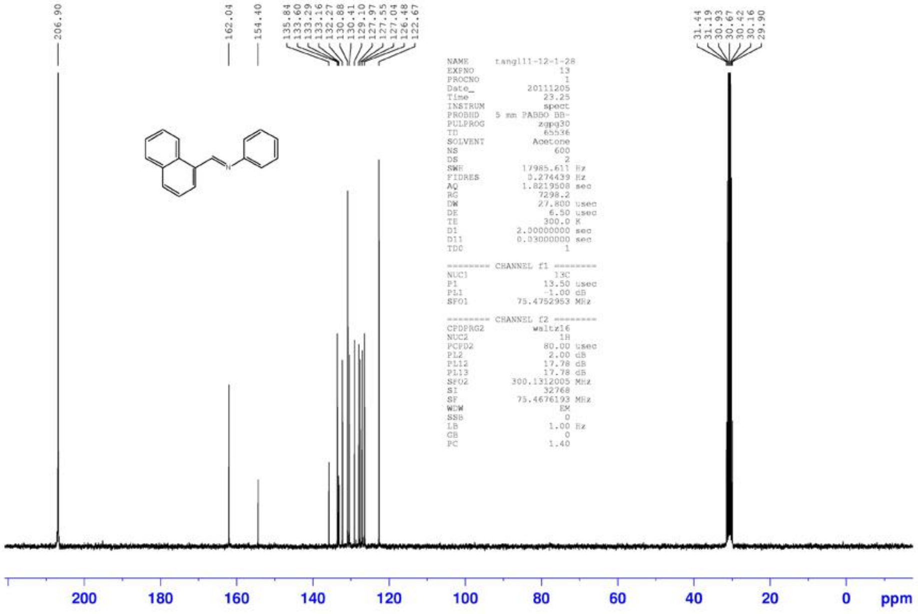

8.55
7.51
7.49
7.48
7.47
7.40
7.39
7.37
7.36
7.35
7.24
7.23
7.22
7.22
7.21
7.19
7.13
7.12
7.12
7.11
0.08
0.00
NAME tang120111025-2-21
EXPNO 1
PROCNO 1
Date_ 20111028
Time 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
S
N
9
8
7
6
5
4
3
2
1
ppm
1.00
2.01
2.11
2.94
1.10

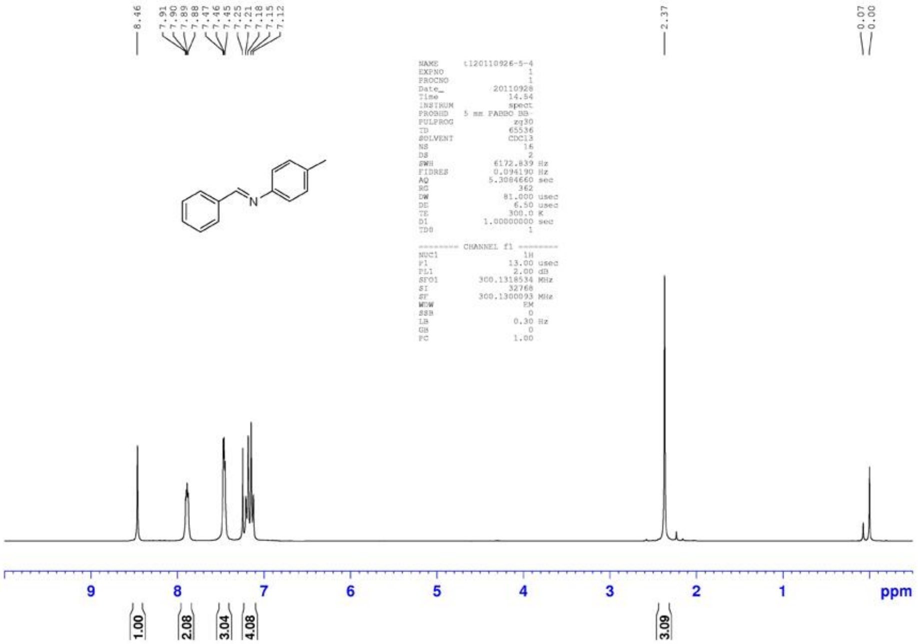

152.99
151.40
142.84
132.18
130.34
129.13
127.73
126.03
120.98
77.46
77.24
77.03
76.61
1.02
0.00
NAME tang120111025-2-5
EXPNO 13
PROCNO 1
Date_ 20111029
Time 0.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 7298.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677503 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
S
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

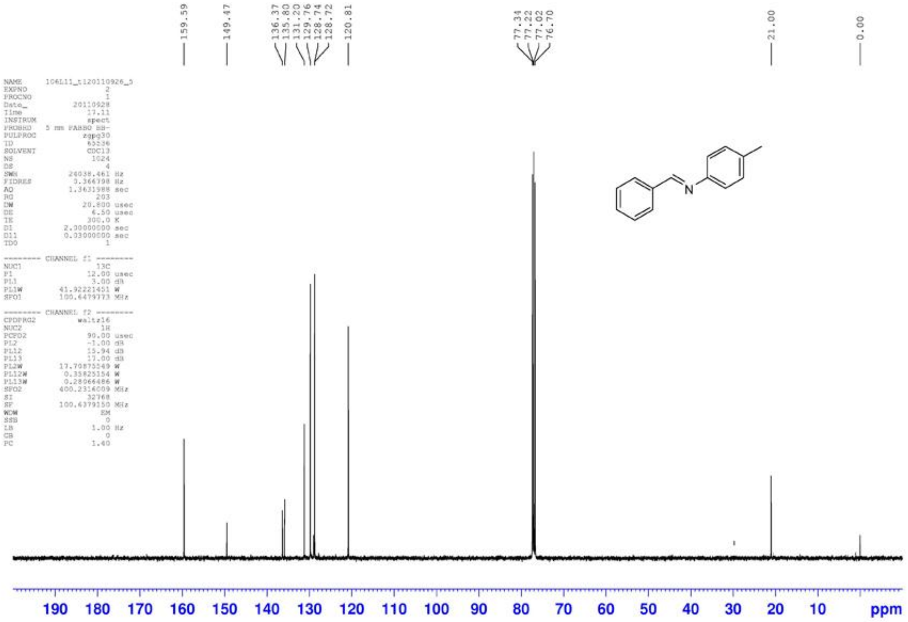

8.27
8.27
8.25
8.25
7.55
7.54
7.52
7.42
7.41
7.40
7.39
7.38
7.37
7.35
7.35
7.34
7.32
7.32
7.32
7.24
7.21
7.19
7.17
7.14
7.13
7.12
7.06
0.00
N
9
8
7
6
5
4
3
2
1
ppm
1.00
2.13
5.19
2.88
2.06

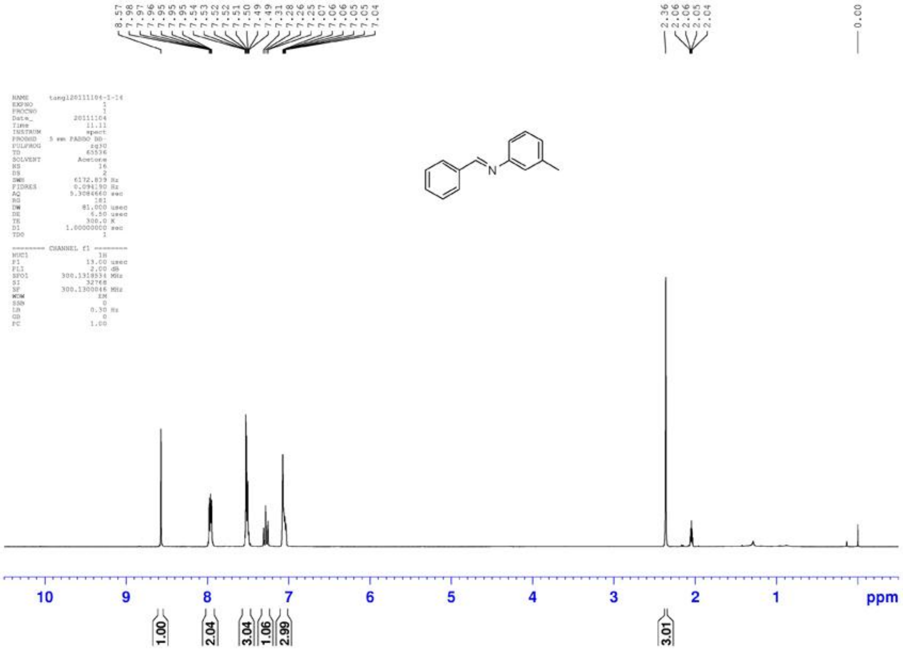

161.61
151.77
144.00
135.63
129.60
129.19
128.94
128.65
127.51
126.12
120.93
77.47
77.25
77.05
76.63
0.01
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


9.33
9.30
9.18
8.18
8.15
8.08
8.06
8.02
7.99
7.68
7.68
7.66
7.63
7.63
7.62
7.61
7.61
7.59
7.57
7.47
7.47
7.45
7.43
7.42
7.37
7.36
7.36
7.34
7.34
7.29
7.29
7.29
7.27
7.26
7.25
7.24
7.24
0.00
NAME tangl11-12-1
EXPNO 1
PROCNO 1
Date_ 20111205
Time 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.00
1.00
1.02
3.06
1.99
2.01
1.04

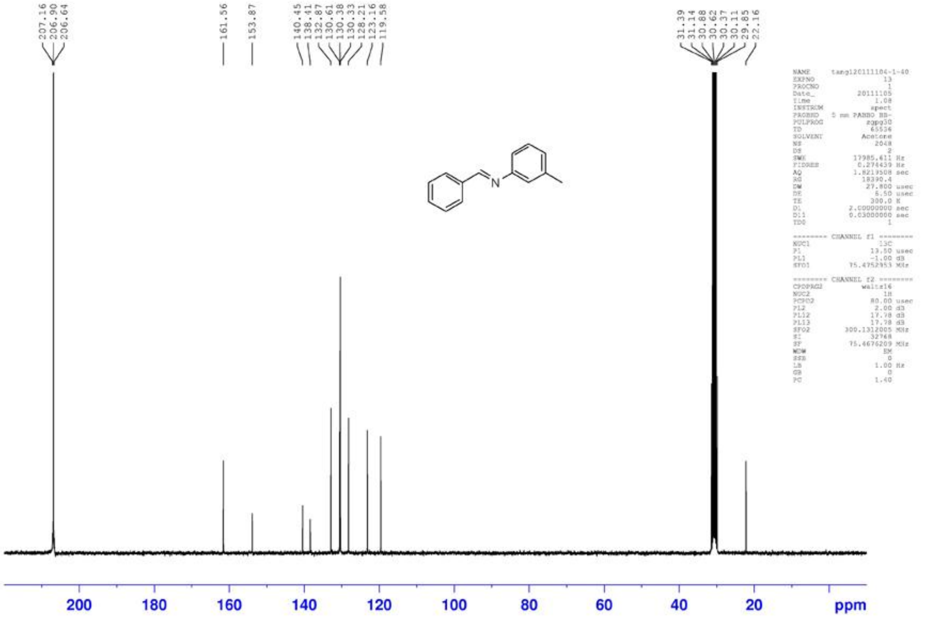

206.90
162.04
154.40
135.84
133.60
133.29
133.16
132.27
130.88
130.41
129.10
127.97
127.55
127.04
126.48
122.67
31.44
31.19
30.93
30.67
30.42
30.16
29.90
NAME tangl11-12-1-28
EXPNO 13
PROCNO 1
Date_ 20111205
Time 23.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 600
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 7298.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4676193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
200
180
160
140
120
100
80
60
40
20
0
ppm


8.46
7.91
7.90
7.89
7.88
7.47
7.46
7.45
7.25
7.21
7.18
7.15
7.12
2.37
0.07
0.00
N
NAME t120110926-5-4
EXPNO 1
PROCNO 1
Date_ 20110928
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
2.08
3.04
4.08
3.09

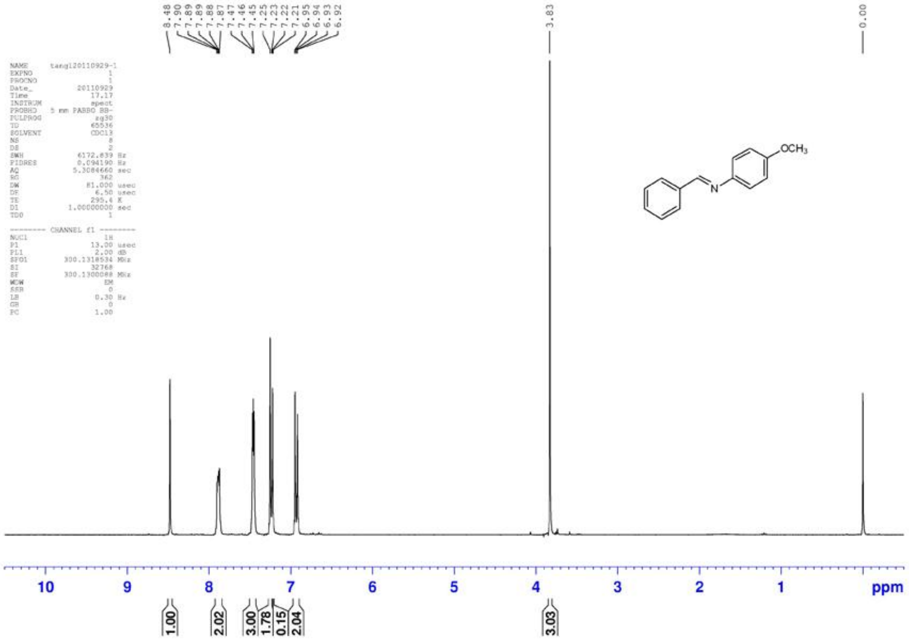

159.59
149.47
136.37
135.80
131.20
129.76
128.74
128.72
120.81
77.34
77.22
77.02
76.70
21.00
0.00
NAME 106L11_t120110926_5
EXPNO 2
PROCNO 1
Date_ 20110928
Time 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28066486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379150 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

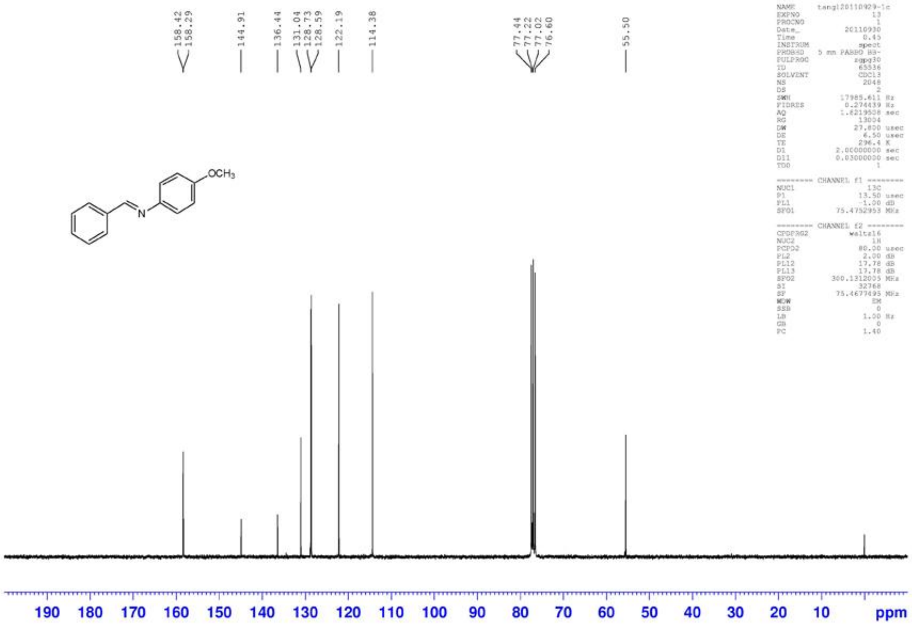


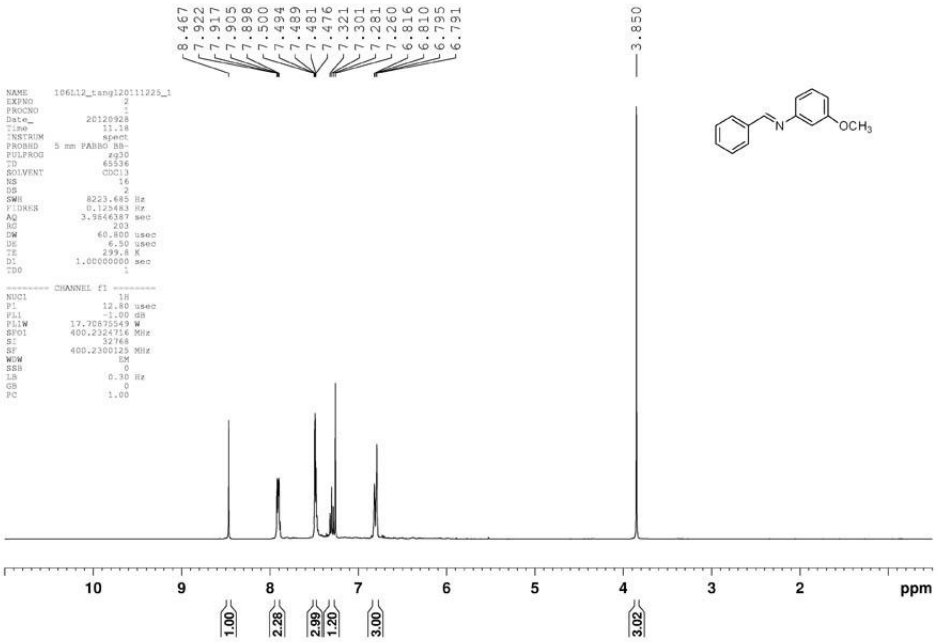
```
NAME     tangl20111104-1-14
EXPNO                     1
PROCNO                    1
Date_              20111104
Time                  11.11
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        2
SWH            6172.839 Hz
FIDRES         0.094190 Hz
AQ            5.3084660 sec
RG                      181
DW               81.000 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                13.00 usec
PL1                2.00 dB
SFO1        300.1318534 MHz
SI                    32768
SF          300.1300046 MHz
WDW                      EM
SSB                       0
LB                 0.30 Hz
GB                        0
PC                     1.00
```

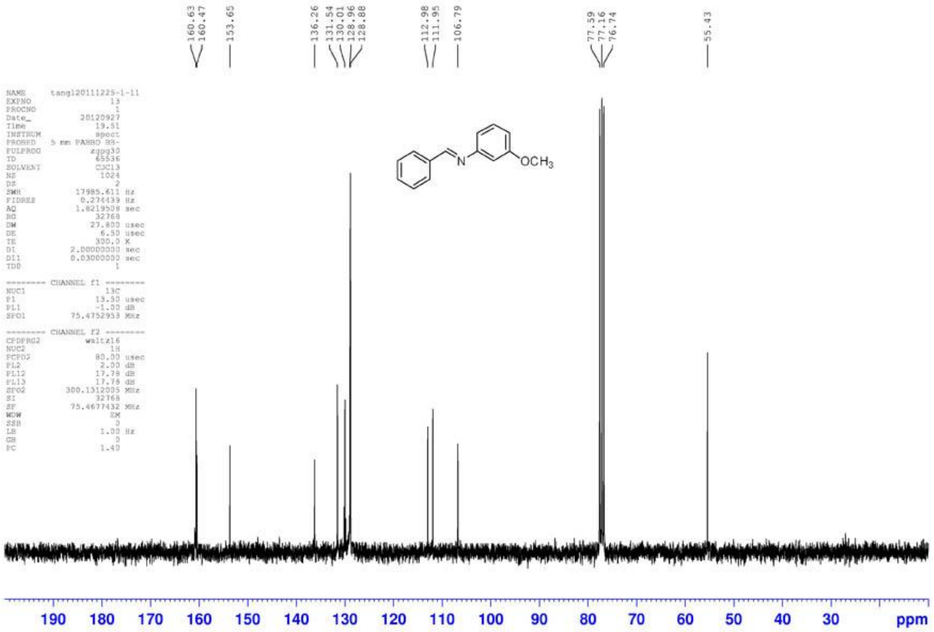


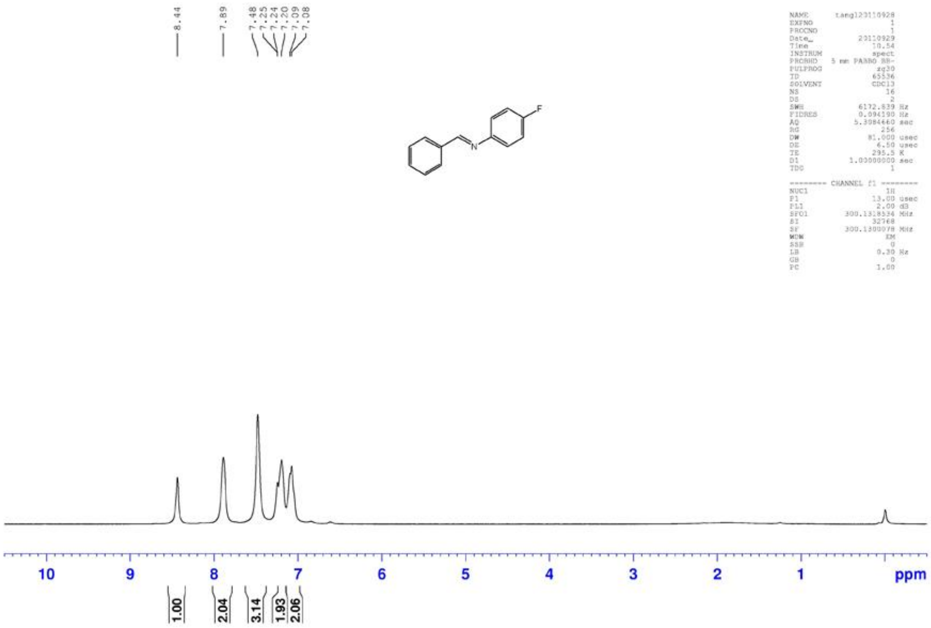
```
NAME     tangl20111104-1-40
EXPNO                    13
PROCNO                    1
Date_              20111105
Time                   1.08
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                     2048
DS                        2
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                  18390.4
DW               27.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                13.50 usec
PL1               -1.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2             80.00 usec
PL2                2.00 dB
PL12              17.78 dB
PL13              17.78 dB
SFO2        300.1312005 MHz
SI                    32768
SF           75.4676209 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                     1.40
```


8.48
7.90
7.89
7.89
7.88
7.87
7.47
7.46
7.45
7.25
7.23
7.22
7.21
6.95
6.94
6.93
6.92
3.83
0.00
NAME tangl20110929-1
EXPNO 1
PROCNO 1
Date_ 20110929
Time 17.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OCH3
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.02
3.00
1.78
0.15
2.04
3.03

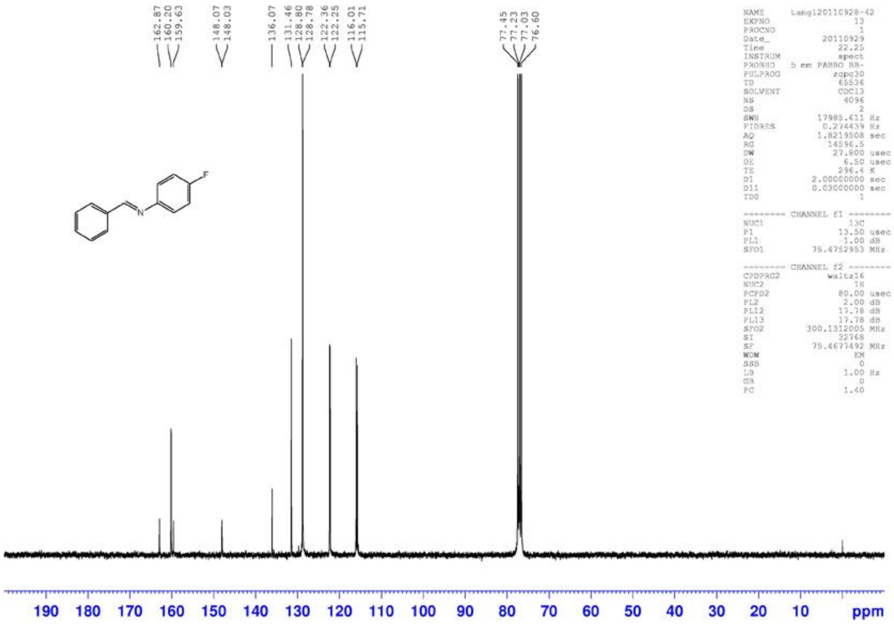

158.42
158.29
144.91
136.44
131.04
128.73
128.59
122.19
114.38
77.44
77.22
77.02
76.60
55.50
NAME tangl20110929-1c
EXPNO 13
PROCNO 1
Date_ 20110930
Time 0.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677495 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OCH3
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.467
7.922
7.917
7.905
7.898
7.500
7.494
7.489
7.481
7.476
7.321
7.301
7.281
7.260
6.816
6.810
6.795
6.791
3.850
NAME 106L12_tang120111225_1
EXPNO 2
PROCNO 1
Date_ 20120928
Time 11.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
OCH3
10
9
8
7
6
5
4
3
2
ppm
1.00
2.28
2.99
1.20
3.00
3.02

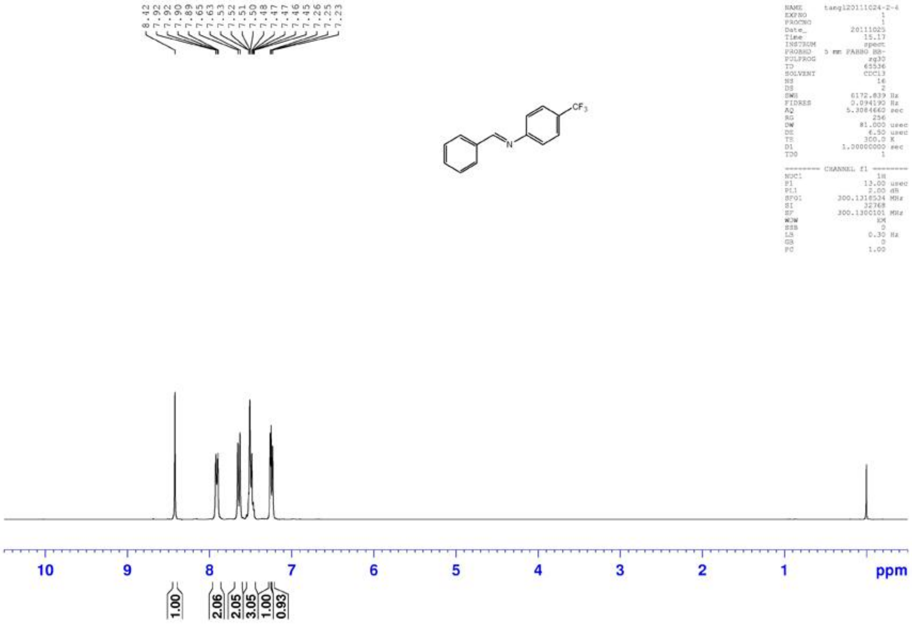

160.63
160.47
153.65
136.26
131.54
130.01
128.96
128.88
112.98
111.95
106.79
77.59
77.16
76.74
55.43
NAME tang120111225-1-11
EXPNO 13
PROCNO 1
Date_ 20120927
Time 19.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677432 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
OCH3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm



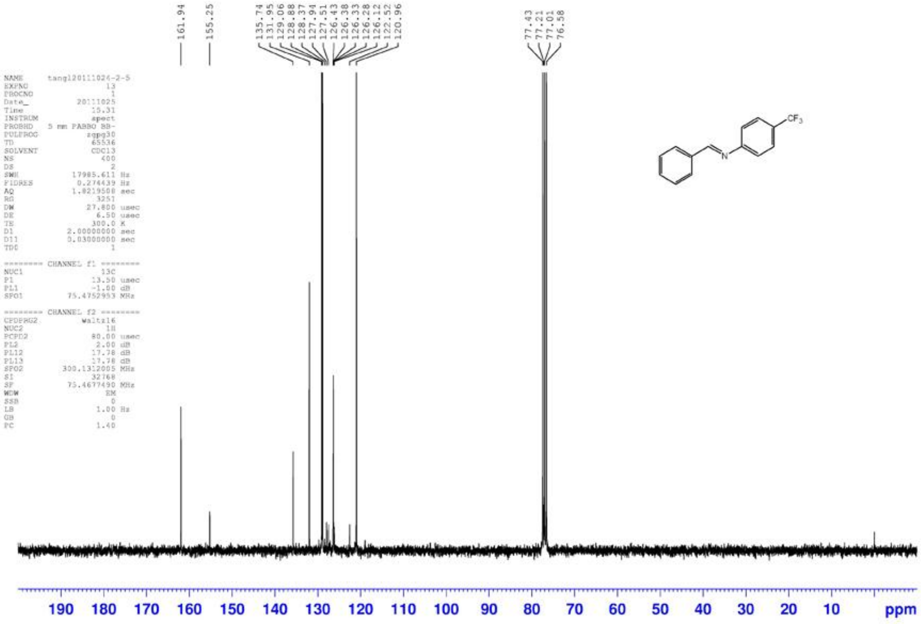
```
NAME       tang120110928
EXPNO                  1
PROCNO                 1
Date_           20110929
Time               10.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ             5.3084660 sec
RG                   256
DW                81.000 usec
DE                  6.50 usec
TE                 295.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                 2.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300078 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



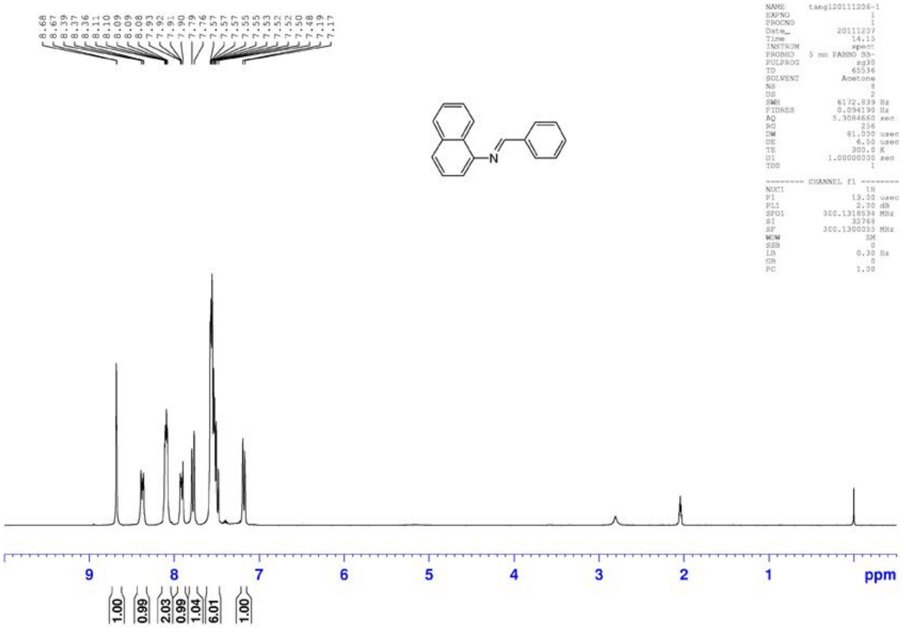
```
NAME       tang120110928-42
EXPNO                    13
PROCNO                    1
Date_              20110929
Time                  22.25
INSTRUM               spect
PROBHD      5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     4096
DS                        2
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                  14596.5
DW                   27.800 usec
DE                     6.50 usec
TE                    296.4 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    13.50 usec
PL1                   -1.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    2.00 dB
PL12                  17.78 dB
PL13                  17.78 dB
SFO2            300.1312005 MHz
SI                    32768
SF               75.4677492 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

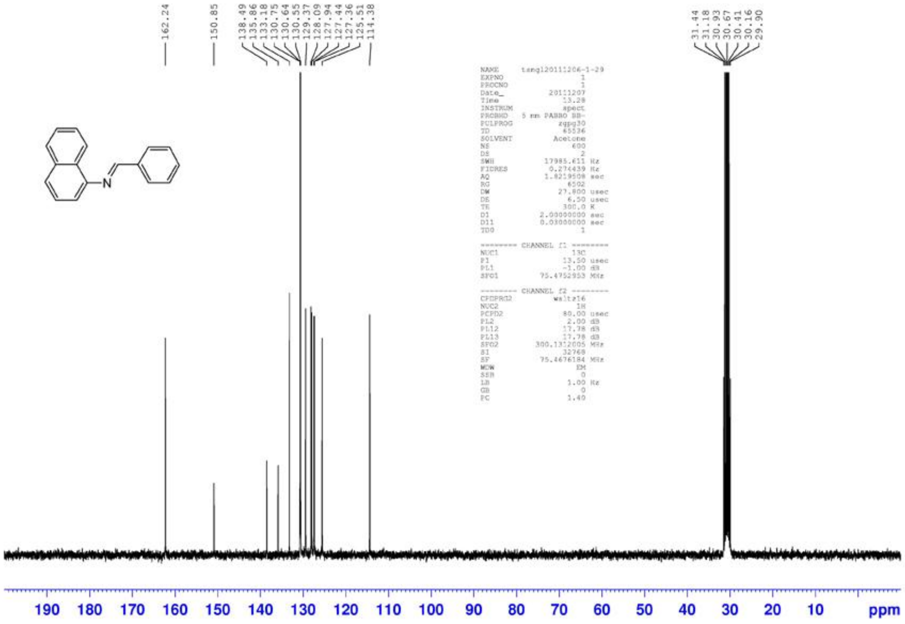


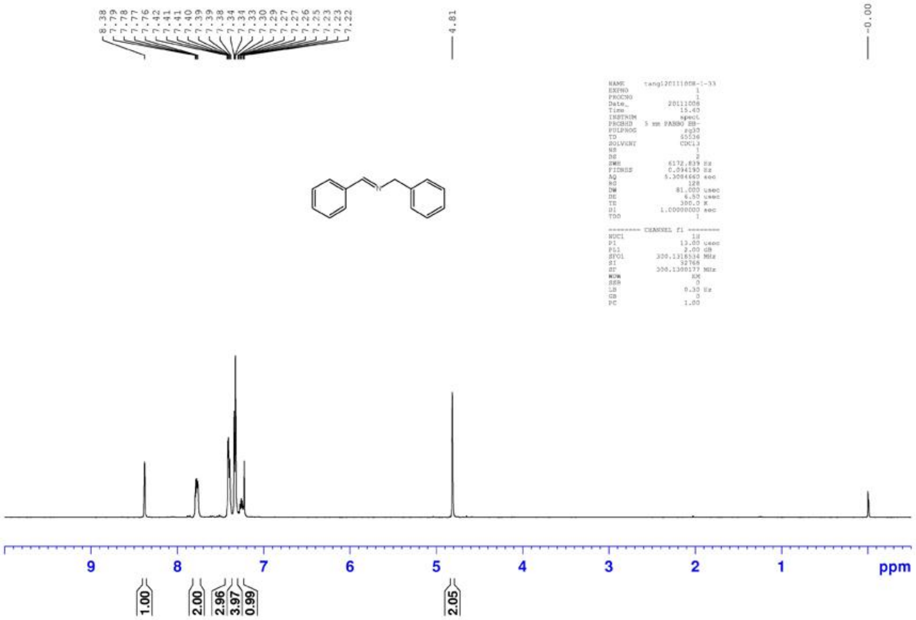
```
NAME       tang120111024-2-4
EXPNO                      1
PROCNO                     1
Date_               20111025
Time                   15.17
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                 5.3084660 sec
RG                       256
DW                    81.000 usec
DE                      6.50 usec
TE                     300.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.00 usec
PL1                     2.00 dB
SFO1             300.1318524 MHz
SI                     32768
SF               300.1300101 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



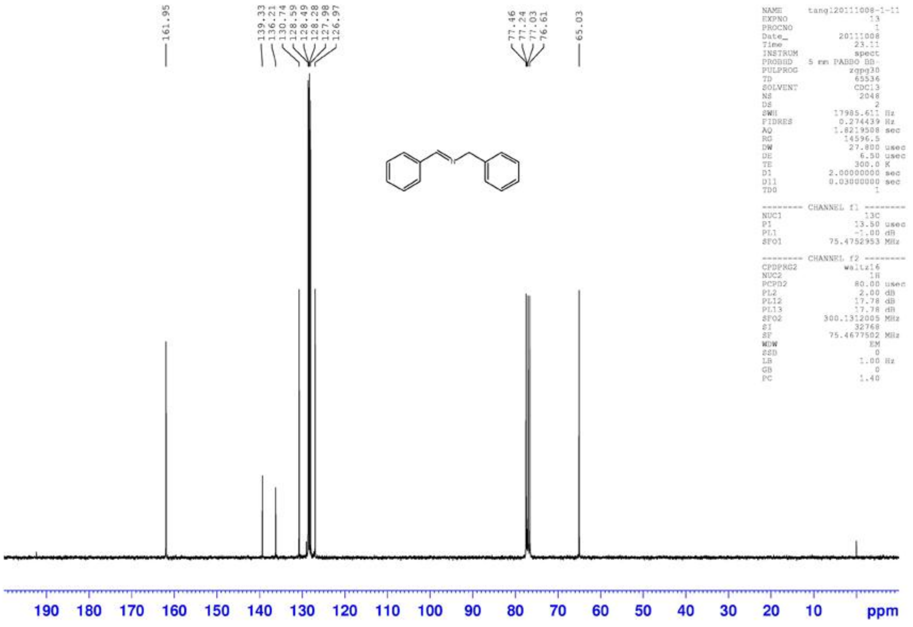
```
NAME       tang120111024-2-5
EXPNO                     13
PROCNO                     1
Date_               20111025
Time                   15.31
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       400
DS                         2
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                 1.8219500 sec
RG                      3251
DW                    27.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                    -1.00 dB
SFO1              75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     2.00 dB
PL12                   17.78 dB
PL13                   17.78 dB
SFO2             300.1312005 MHz
SI                     32768
SF                75.4677490 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```


8.68
8.67
8.39
8.37
8.36
8.11
8.10
8.09
8.09
8.08
7.93
7.92
7.91
7.90
7.79
7.76
7.57
7.57
7.57
7.55
7.55
7.53
7.52
7.52
7.50
7.48
7.19
7.17
N
NAME tang120111206-1
EXPNO 1
PROCNO 1
Date_ 20111207
Time 14.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
0.99
2.03
0.99
1.04
6.01
1.00

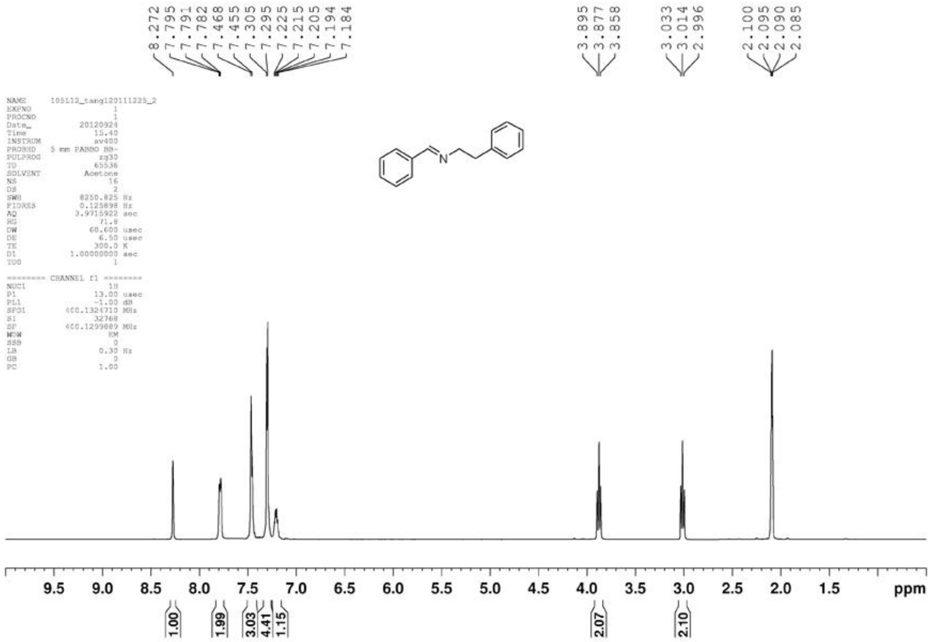

162.24
150.85
138.49
135.86
133.18
130.75
130.64
130.55
129.37
128.09
127.94
127.44
127.36
125.51
114.38
31.44
31.18
30.93
30.67
30.41
30.16
29.90
N
NAME tang120111206-1-29
EXPNO 1
PROCNO 1
Date_ 20111207
Time 13.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 600
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 6502
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4676184 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



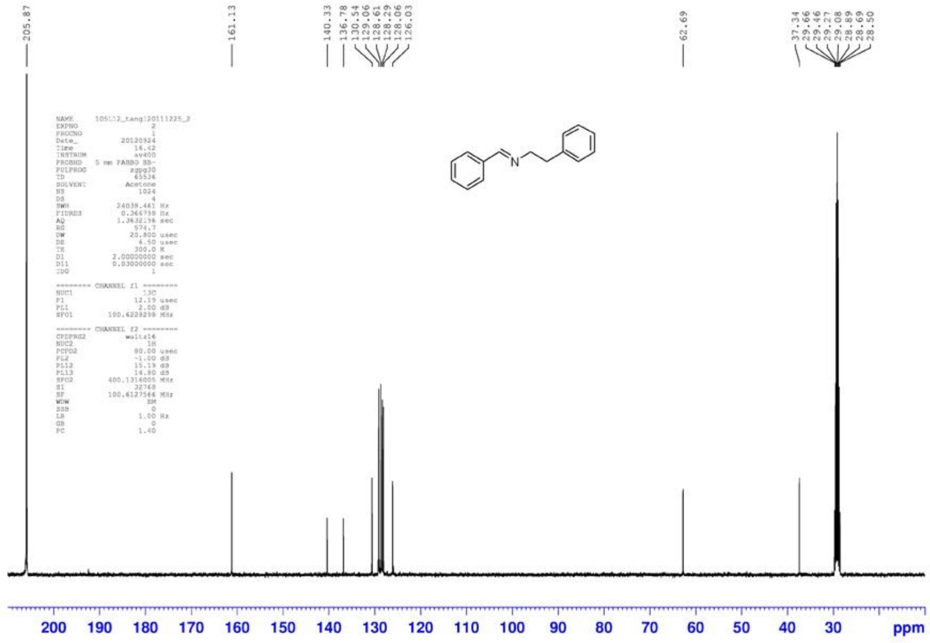
```
NAME        tang120111008-1-33
EXPNO                        1
PROCNO                       1
Date_                 20111008
Time                     15.40
INSTRUM                  spect
PROBHD      5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           1
DS                           2
SWH                   6172.839 Hz
FIDRES                0.094190 Hz
AQ                   5.3084660 sec
RG                         128
DW                      81.000 usec
DE                        6.50 usec
TE                       300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.00 usec
PL1                       2.00 dB
SFO1               300.1318534 MHz
SI                       32768
SF                 300.1300177 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



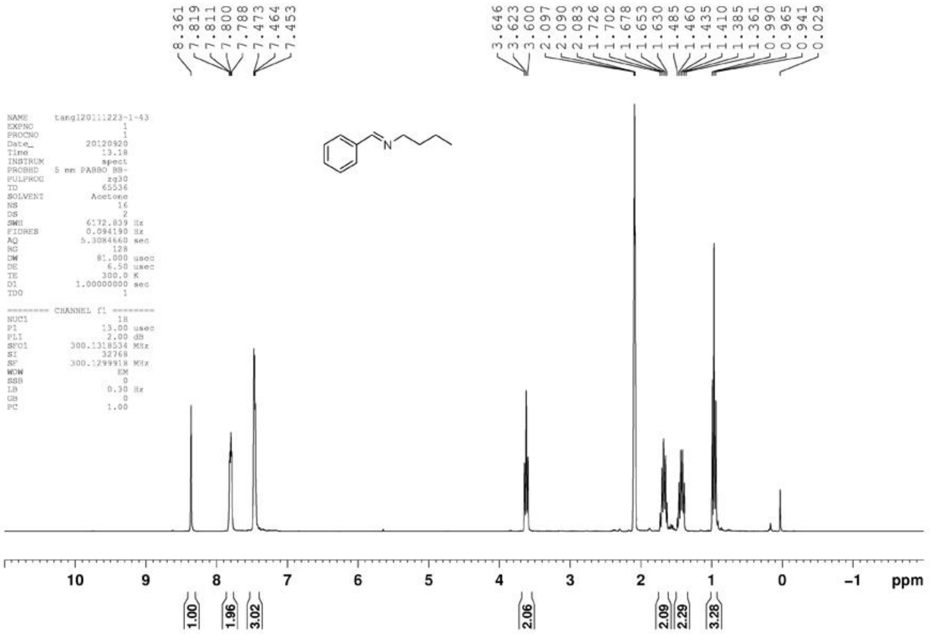
```
NAME        tang120111008-1-11
EXPNO                       13
PROCNO                       1
Date_                 20111008
Time                     23.11
INSTRUM                  spect
PROBHD      5 mm PABBO BB-
PULPROG                zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           2
SWH                  17985.611 Hz
FIDRES                0.274439 Hz
AQ                   1.8219508 sec
RG                     14596.5
DW                      27.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                      -1.00 dB
SFO1                75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       2.00 dB
PL12                     17.78 dB
PL13                     17.78 dB
SFO2               300.1312005 MHz
SI                       32768
SF                  75.4677502 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

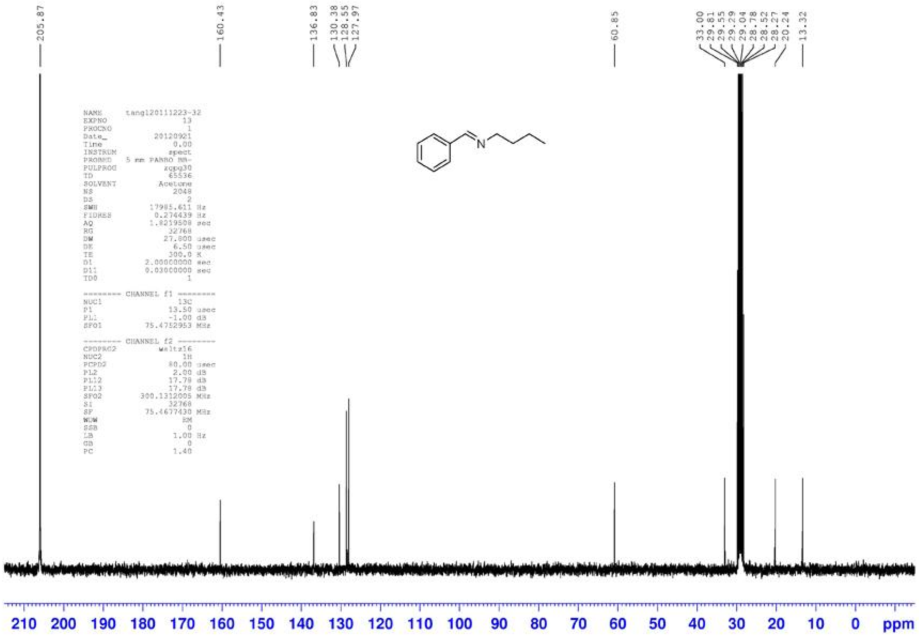


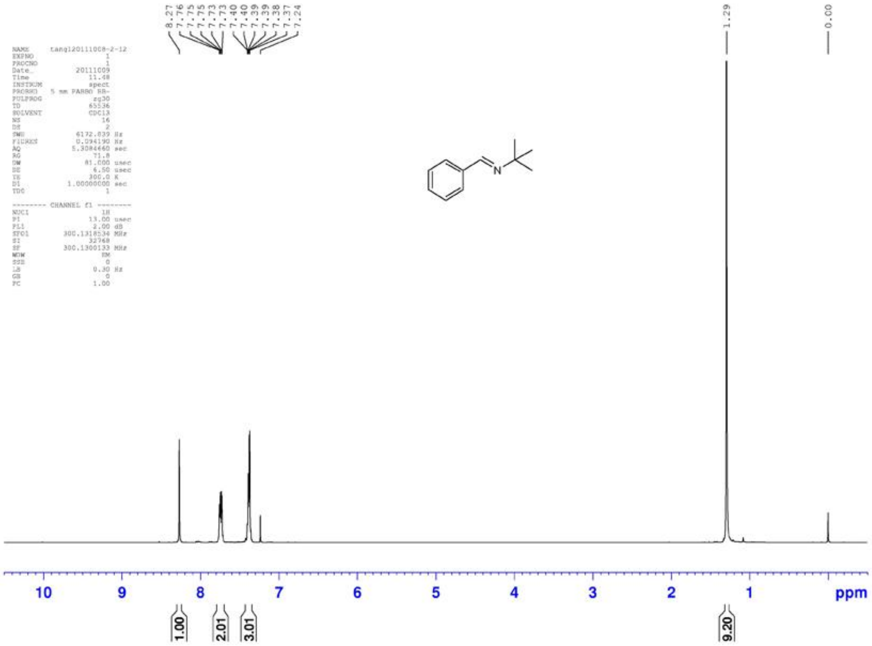
```
NAME     105L12_tang120111225_2
EXPNO                    1
PROCNO                   1
Date_             20120924
Time                 15.40
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT            Acetone
NS                      16
DS                       2
SWH               8250.825 Hz
FIDRES            0.125898 Hz
AQ               3.9715922 sec
RG                    71.8
DW                  60.600 usec
DE                    6.50 usec
TE                   300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                  -1.00 dB
SFO1           400.1324710 MHz
SI                   32768
SF             400.1299889 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

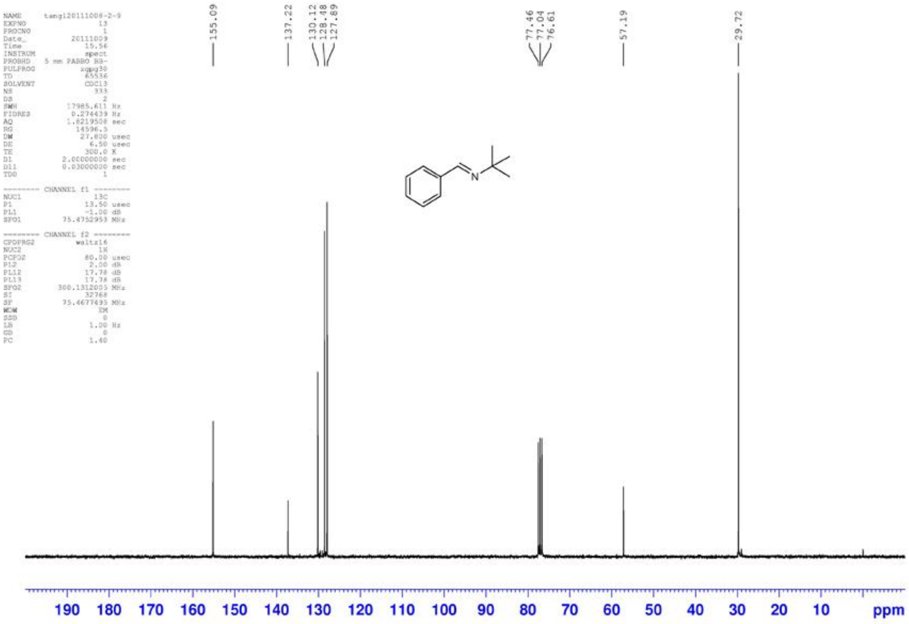


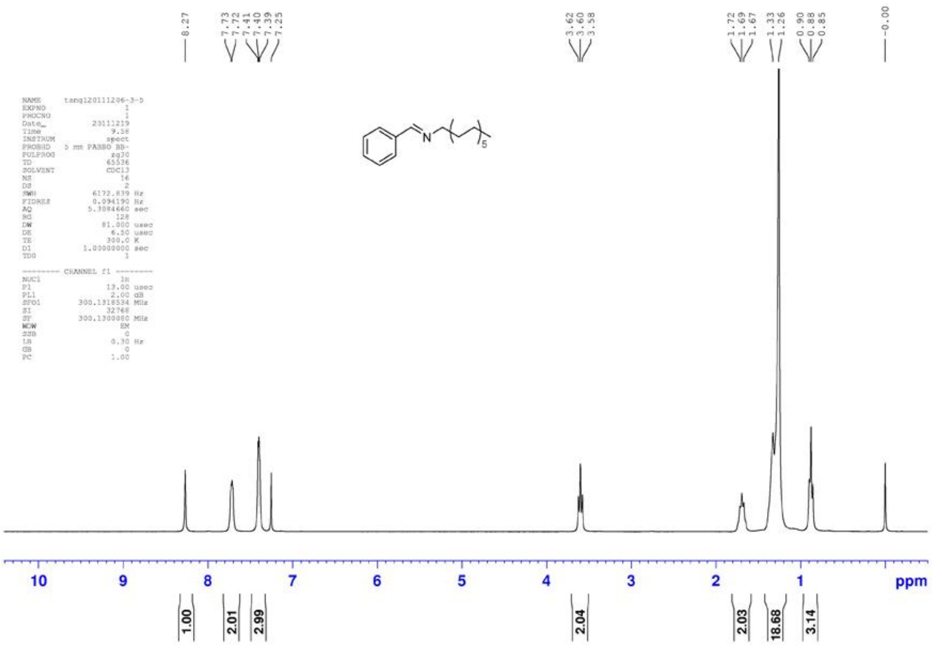
```
NAME     105L12_tang120111225_2
EXPNO                    2
PROCNO                   1
Date_             20120924
Time                 16.42
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3632196 sec
RG                   574.7
DW                  20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.19 usec
PL1                   2.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 15.19 dB
PL13                 14.80 dB
SFO2           400.1316005 MHz
SI                   32768
SF             100.6127546 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

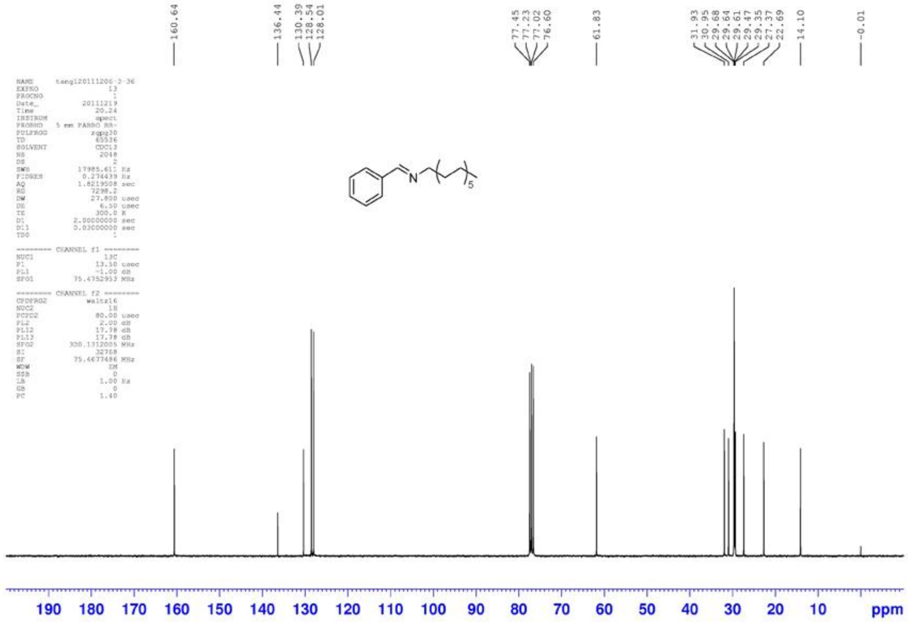

8.361
7.819
7.811
7.800
7.788
7.473
7.464
7.453
3.646
3.623
3.600
2.097
2.090
2.083
1.726
1.702
1.678
1.653
1.630
1.485
1.460
1.435
1.410
1.385
1.361
0.990
0.965
0.941
0.029
NAME tang120111223-1-43
EXPNO 1
PROCNO 1
Date_ 20120920
Time 13.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299918 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.00
1.96
3.02
2.06
2.09
2.29
3.28

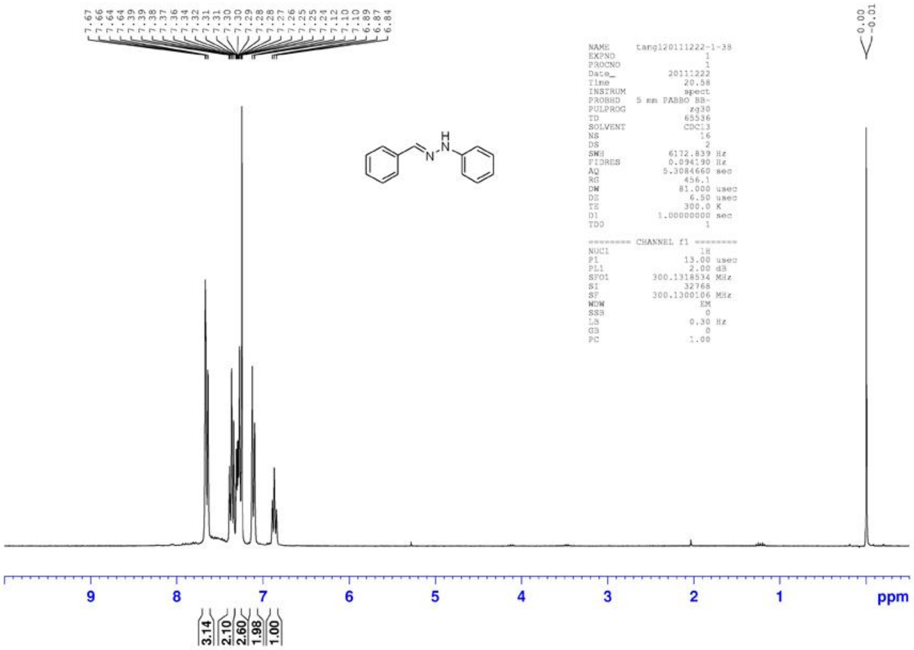

205.87
160.43
136.83
130.38
128.55
127.97
60.85
33.00
29.81
29.55
29.29
29.04
28.78
28.52
28.27
20.24
13.32
NAME tang120111223-32
EXPNO 13
PROCNO 1
Date_ 20120921
Time 0.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 2048
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677430 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

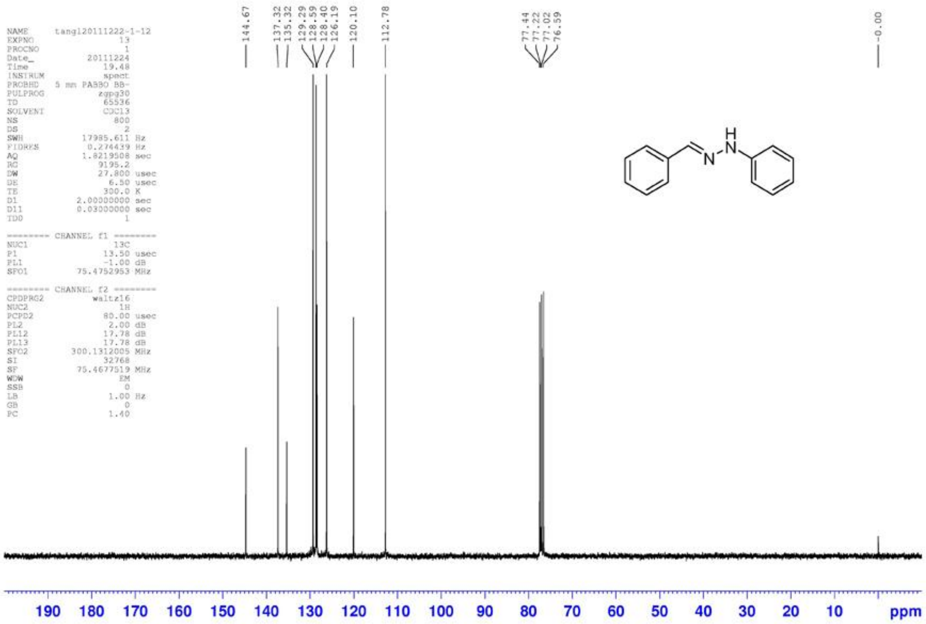

NAME tang120111008-2-12
EXPNO 1
PROCNO 1
Date_ 20111009
Time 11.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.27
7.76
7.75
7.75
7.73
7.73
7.40
7.40
7.39
7.39
7.38
7.37
7.24
1.29
0.00
N
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.01
3.01
9.20


NAME tang120111008-2-9
EXPNO 13
PROCNO 1
Date_ 20111009
Time 15.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 333
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677495 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
155.09
137.22
130.12
128.48
127.89
77.46
77.04
76.61
57.19
29.72
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.27
7.73
7.72
7.41
7.40
7.39
7.25
3.62
3.60
3.58
1.72
1.69
1.67
1.33
1.26
0.90
0.88
0.85
-0.00
NAME tang120111206-3-5
EXPNO 1
PROCNO 1
Date_ 20111219
Time 9.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
5
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.01
2.99
2.04
2.03
18.68
3.14


160.64
136.44
130.39
128.54
128.01
77.45
77.23
77.02
76.60
61.83
31.93
30.95
29.68
29.64
29.61
29.47
29.35
27.37
22.69
14.10
-0.01
NAME tang120111206-3-36
EXPNO 13
PROCNO 1
Date_ 20111219
Time 20.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 7298.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.67
7.66
7.64
7.64
7.39
7.39
7.38
7.37
7.36
7.34
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.25
7.25
7.24
7.12
7.10
7.10
6.89
6.87
6.84
0.00
-0.01
NAME tang120111222-1-38
EXPNO 1
PROCNO 1
Date_ 20111222
Time 20.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 456.1
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
N
9
8
7
6
5
4
3
2
1
ppm
3.14
2.10
2.60
1.98
1.00


144.67
137.32
135.32
129.29
128.59
128.40
126.19
120.10
112.78
77.44
77.22
77.02
76.59
-0.00
NAME tang120111222-1-12
EXPNO 13
PROCNO 1
Date_ 20111224
Time 19.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 9195.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm