Electronic Supplementary Information

Highly selective reduction of nitroarenes by iron(0) nanoparticles in water

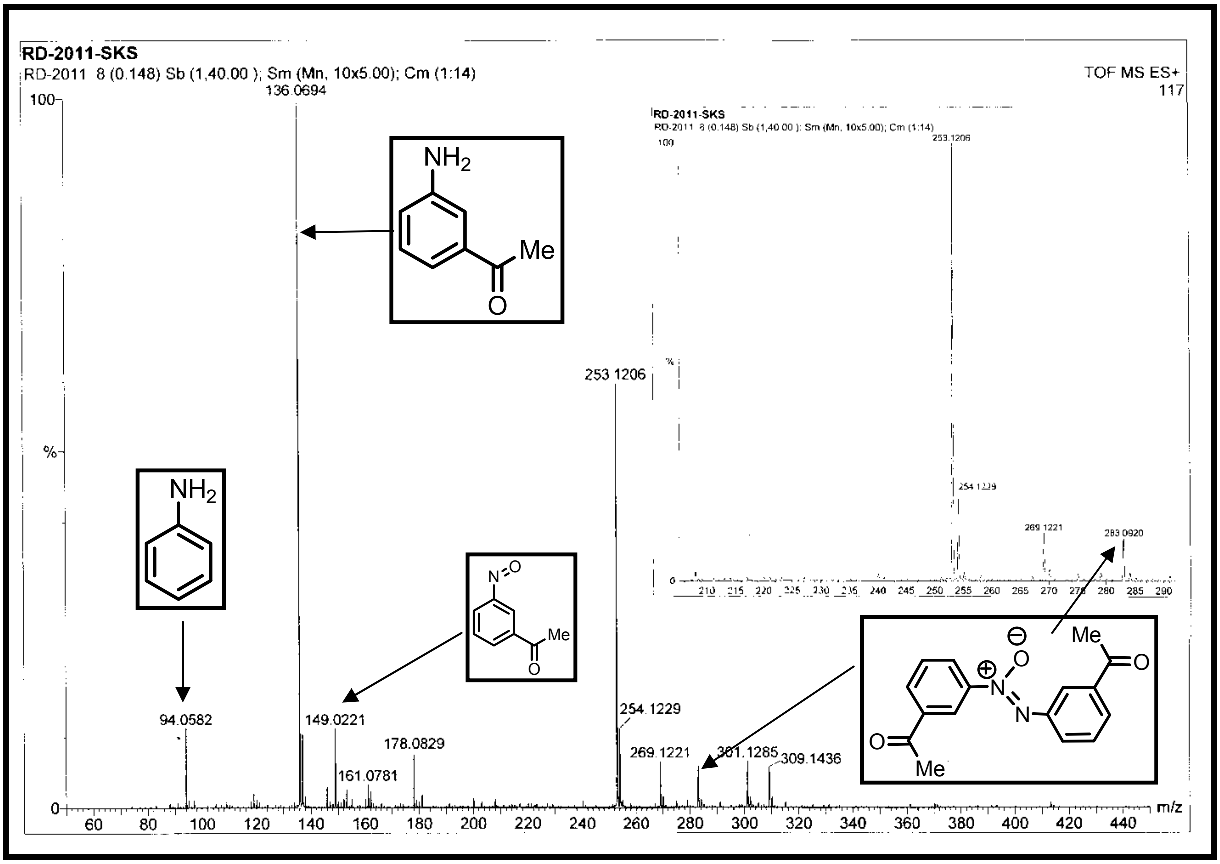
*Raju Dey, Nirmalya Mukherjee, Sabir Ahammed, and Brindaban C. Ranu**

General experimental procedure for reduction of nitroarenes

Sodium borohydride (200 mg, 5 mmol) was added to a stirring solution of ferrous sulphate-hepta-hydrate [$FeSO_4$, $7H_2O$] (834 mg, 3 mmol) and citric acid (55 mg, 0.25 mmol) in water (100 mL) and vigorous stirring was continued for 3-4 minutes. When the solution settled down the water layer was decanted. The residual black solid material of Fe nanoparticles was further washed with water two times to be ready for use in nitro reduction. The nitro compound (1 mmol) was added to these Fe nanoparticles in water (5 mL) in the same pot under constant stirring at room temperature under argon. After completion of reaction (TLC) the mixture was extracted with ethyl acetate (3 X 10 mL) (all Fe-species remained around stirring bar). Evaporation of solvent and purification by short column chromatography provided the pure product which was properly characterized by spectroscopic (IR, ^{1}H NMR, and ^{13}C NMR) data.

Sample preparation and recording of HRMS at intermediate stage of reaction

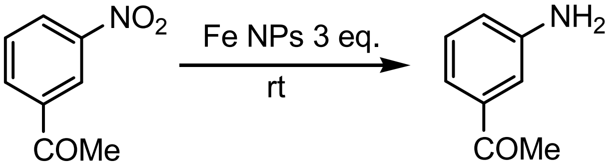
After 1 h of reaction an aliquot of reaction mixture (1 mL) was taken out and extracted with ethyl acetate (3 X 3 mL). After evaporation of solvent the crude material was subjected to HRMS and the spectra was recorded.



Heterogeneity Test

A mixture of Fe NPs (3 mmol), 3-acetylnitrobenzene (165 mg, 1 mmol) in water (5 mL) was stirred at room temperature for 1 h. At this stage (60% conversion by ^{1}H NMR), the catalyst was filtered off and the experiment was continued with the filtrate for another 1 h. There was no increase in the product concentration, as determined by the ^{1}H NMR analysis.

Table The role of solvent in the reduction process.



entry	solvent	time (h)	yield (%)[a]
1	Toluene	4	10
2	CH_2Cl_2	4	8
3	DMF	4	7
4	Water	2	99
5	Methanol	2	10

Yields are based on the ^{1}H-NMR spectra of the crude product.

Characterization of Fe(0) nanoparticles

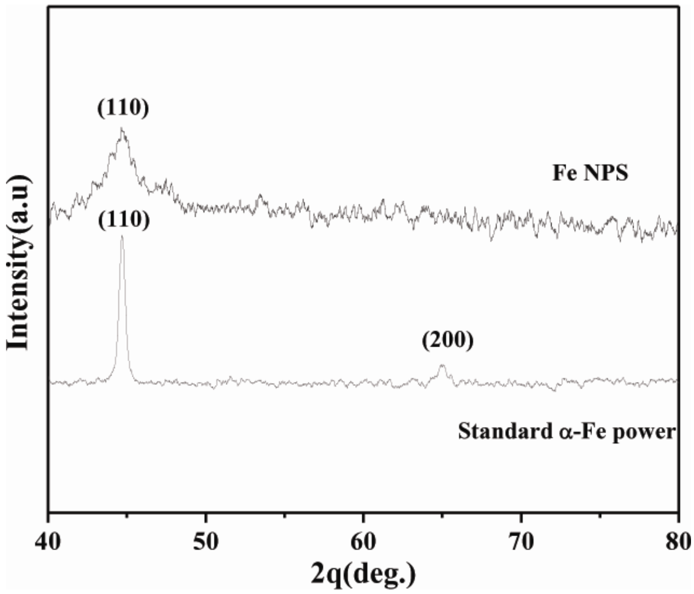


Fig. 1. XRD spectra of Fe(0) NPS

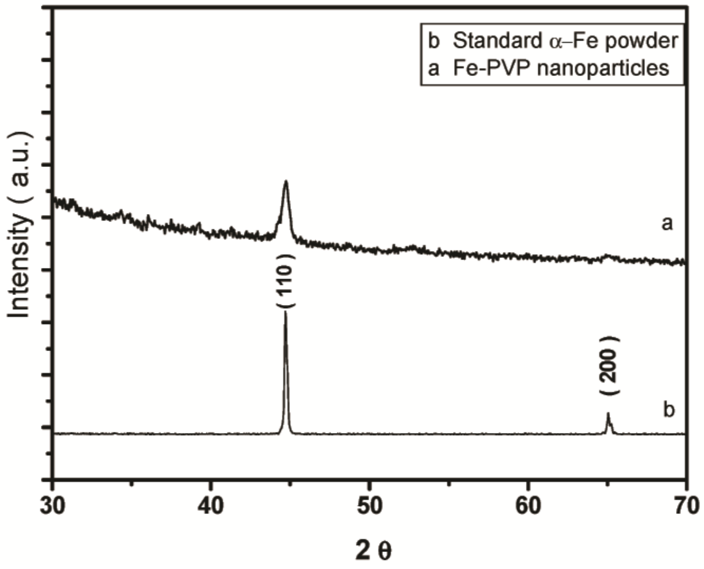


Fig. 2. XRD spectra of Fe(0) nanoparticles reported by Guo et al, *Langmuir* 2006, *22*, 7867

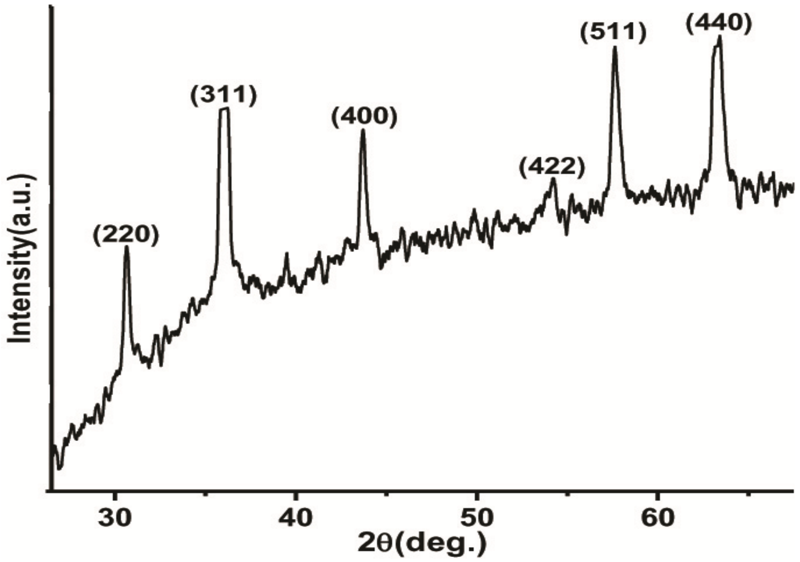


Fig. 3. XRD of end rasidue of Fe after reaction

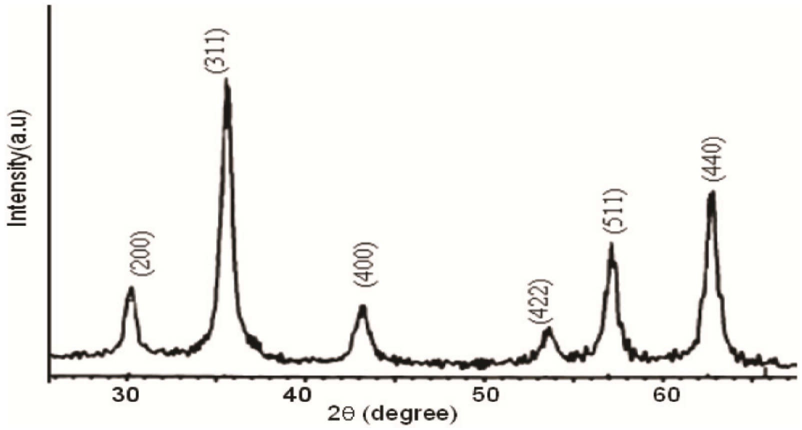


Fig. 4. XRD spectra of Fe_3O_4 nanoparticles reported by Gui-yin Li, Yu-ren Jiang, Ke-long Huang, Ping Ding, Jie Chen, J. Alloys & Comp. 466 (2008) 451–456

Fig. 4. shows X-ray diffraction patterns of the authentic Fe_3O_4 nanopaticles. The XRD peaks of our Fe end residue (Fig. 3.) are compared with those of standard one in JCPDS file (PDF No. 65-3107). A series of characteristic peaks at $2\theta = 30.17°$, 35.46°, 43.38°, 53.69°, 57.23°, and 62.77°, which correspond to (220), (311), (400), (422), (511), and (440) Bragg reflection, respectively in Fig. 3. agree with standard magnetite (Fe_3O_4) XRD patterns in Fig. 4. The Fe_3O_4 nanoparticles are of cubic spinel structure.

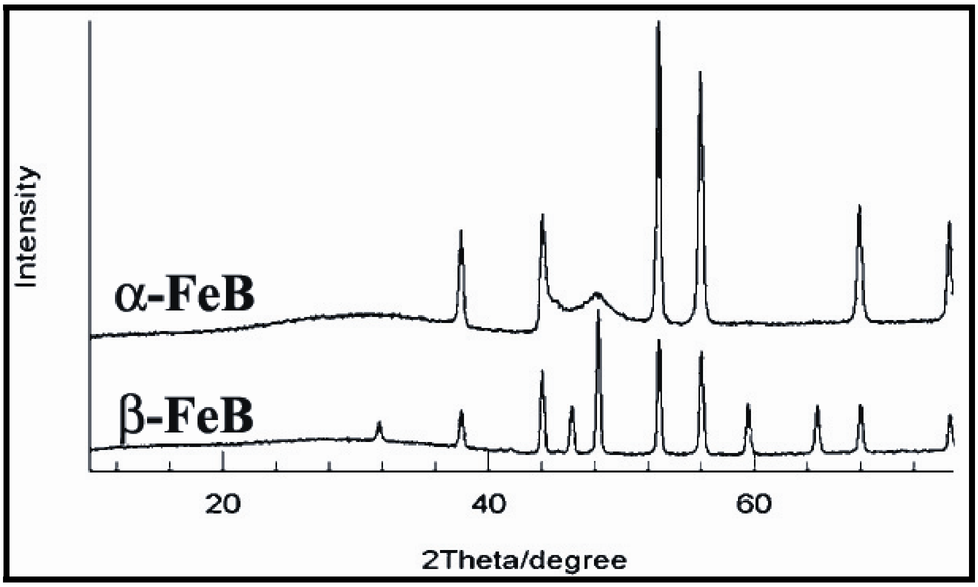


Fig. 5. X-ray diffraction patterns of the α-FeB and β-FeB

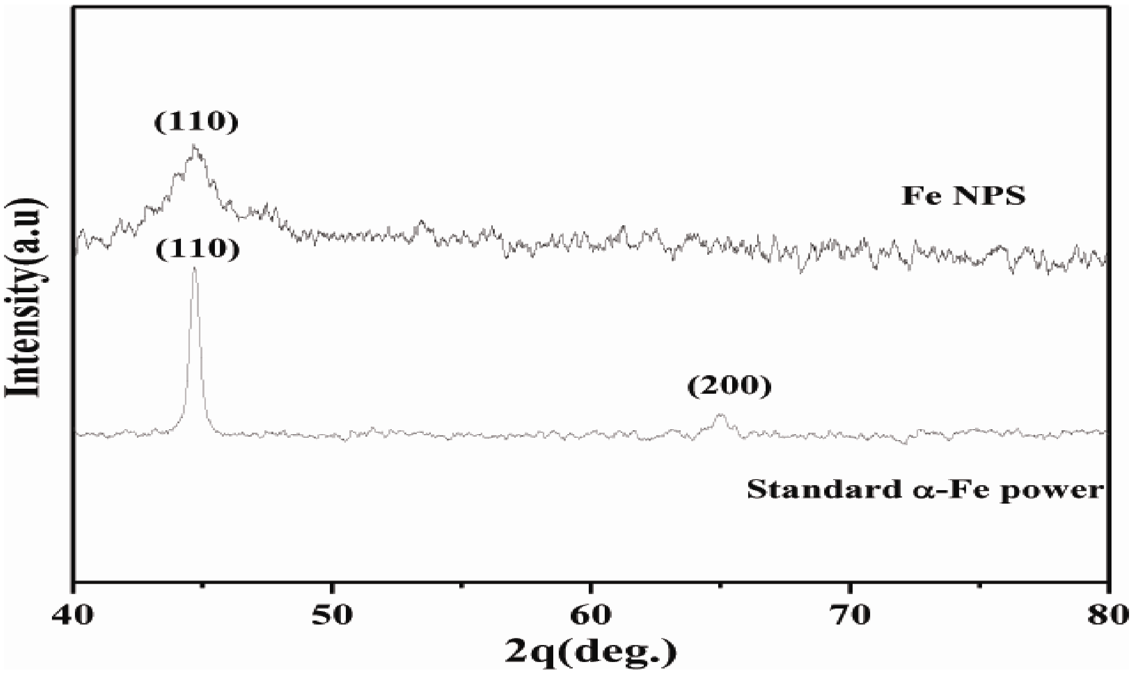


Fig. 1. XRD spectra of our Fe(0) NPS

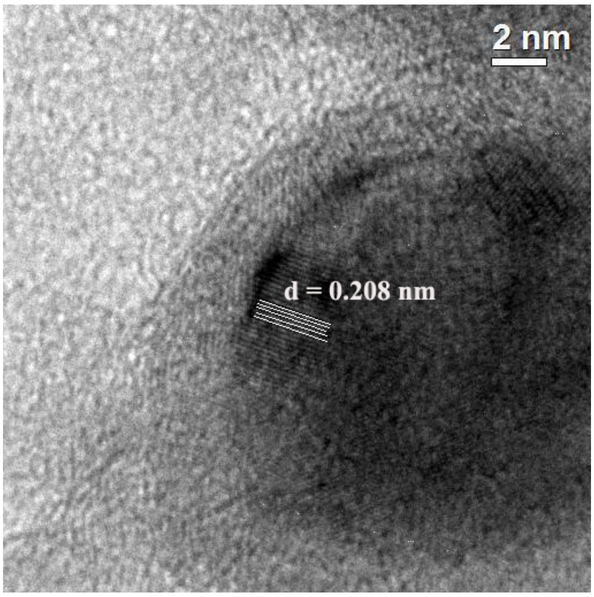


Fig. 6. HR-TEM image of Fe(0) NPs

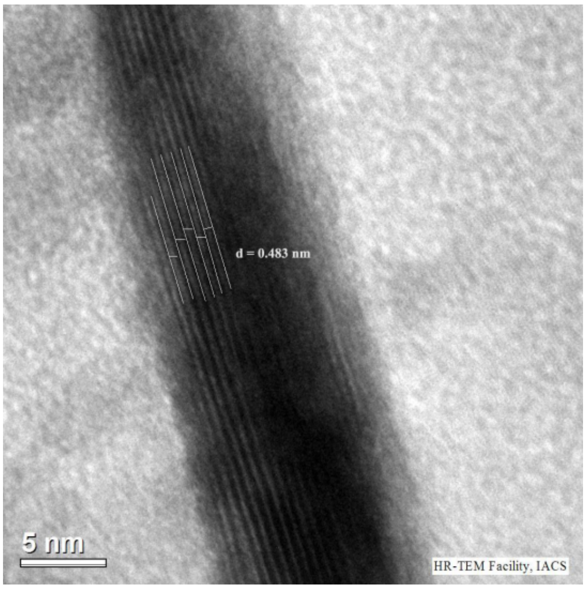


Fig. 7. HR-TEM image of of end rasidue of Fe (Fe_3O_4) after reaction

Fe(0) NPs: From XRD value it was found that prepared Fe(0) NPs is BBC (Body Center Cube)
Lattice d spacing value = 0.208 nm (found form HRTEM)
Standard d spacing value = 2.03 nm (JCPDS data base)
Lattice parameter = 2.94 (from calculation)
Lattice parameter = 2.87 (JCPDS data base)

Fe_3O_4 NPs: From XRD value it was found that prepared Fe(0) NPs is FCC (Body Center Cube)
d spacing value = 0.483 nm (found form HRTEM)
Standard d spacing value = 0.484 nm (JCPDS data base)
Lattice parameter = 8.36 (from calculation)
Lattice parameter = 8.38 (JCPDS data base)

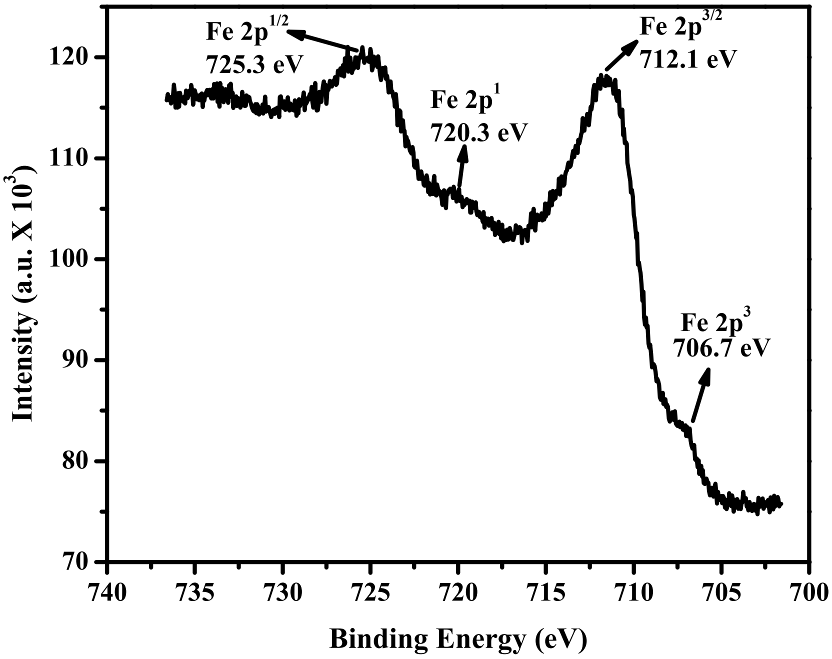


Fig. 8. XPS spectra for Fe(0) NPs

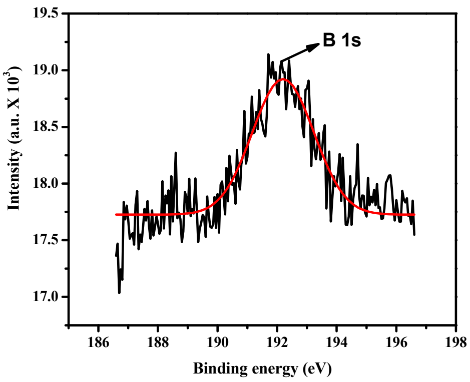


Fig. 9. The XPS spectra of our Fe(0) in the B 1s core level

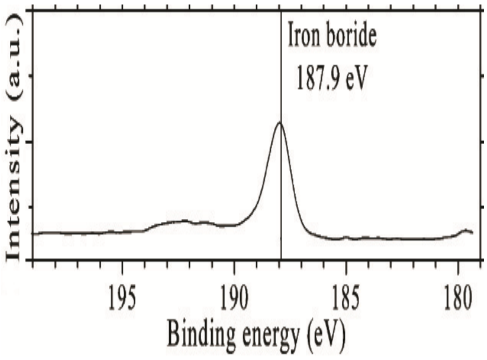
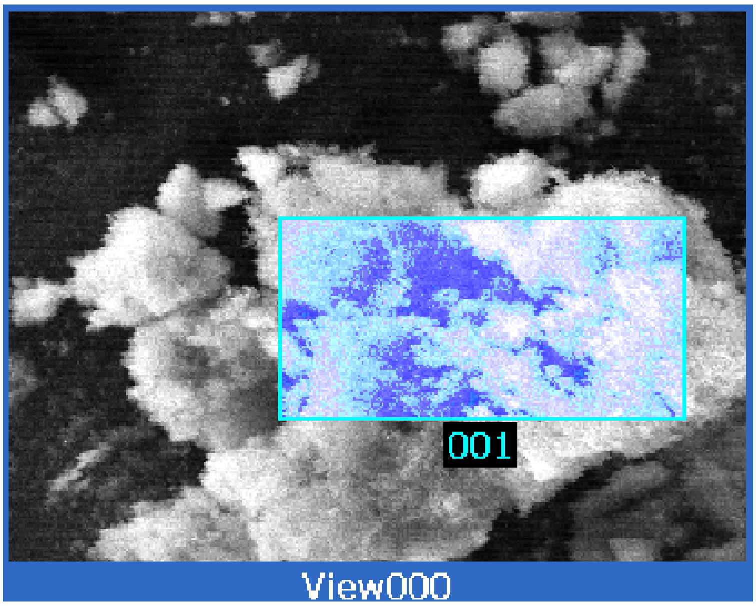
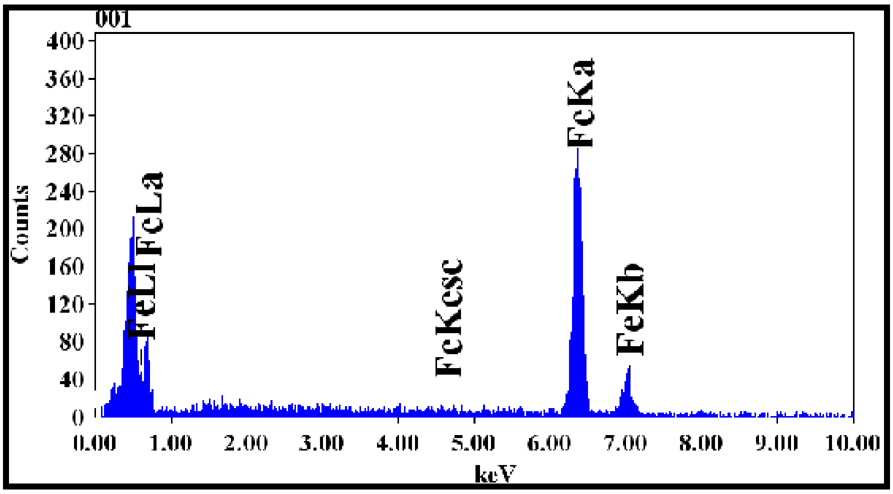


Fig. 10. The XPS spectra on the B 1s core level of FeB reported by Dehlinger et al. Surf. Coat. Technol. (2003) 331–337

EDX experiment with Fe(0) NPS



SEM Image of Fe(0) NPS

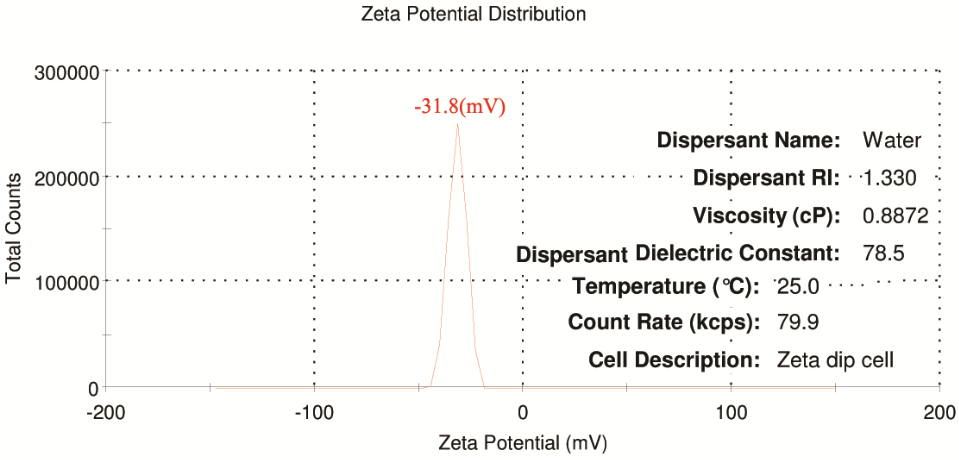


EDX spectra of selected area of Fe(0) NPS

Ele...	Cl...	(keV)	mass%	Error%	At%
Fe K		6.398	100.00	1.36	100.00
Total			100.00		100.00

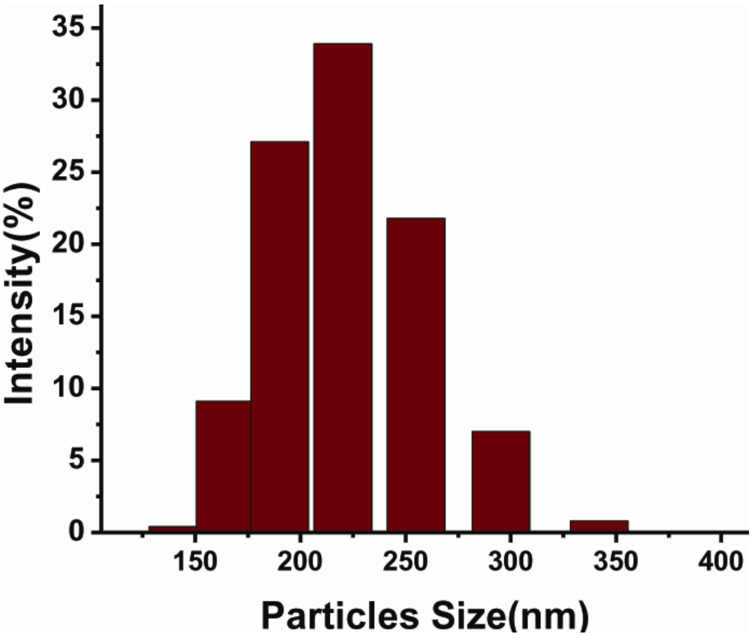
Composition of the material

Zeta potential value of Fe NPS:



Dispersant Name: Water
Dispersant RI: 1.330
Viscosity (cP): 0.8872
Dispersant Dielectric Constant: 78.5
Temperature (℃): 25.0
Count Rate (kcps): 79.9
Cell Description: Zeta dip cell

Particles size distribution of Fe NPS (DLS exp):

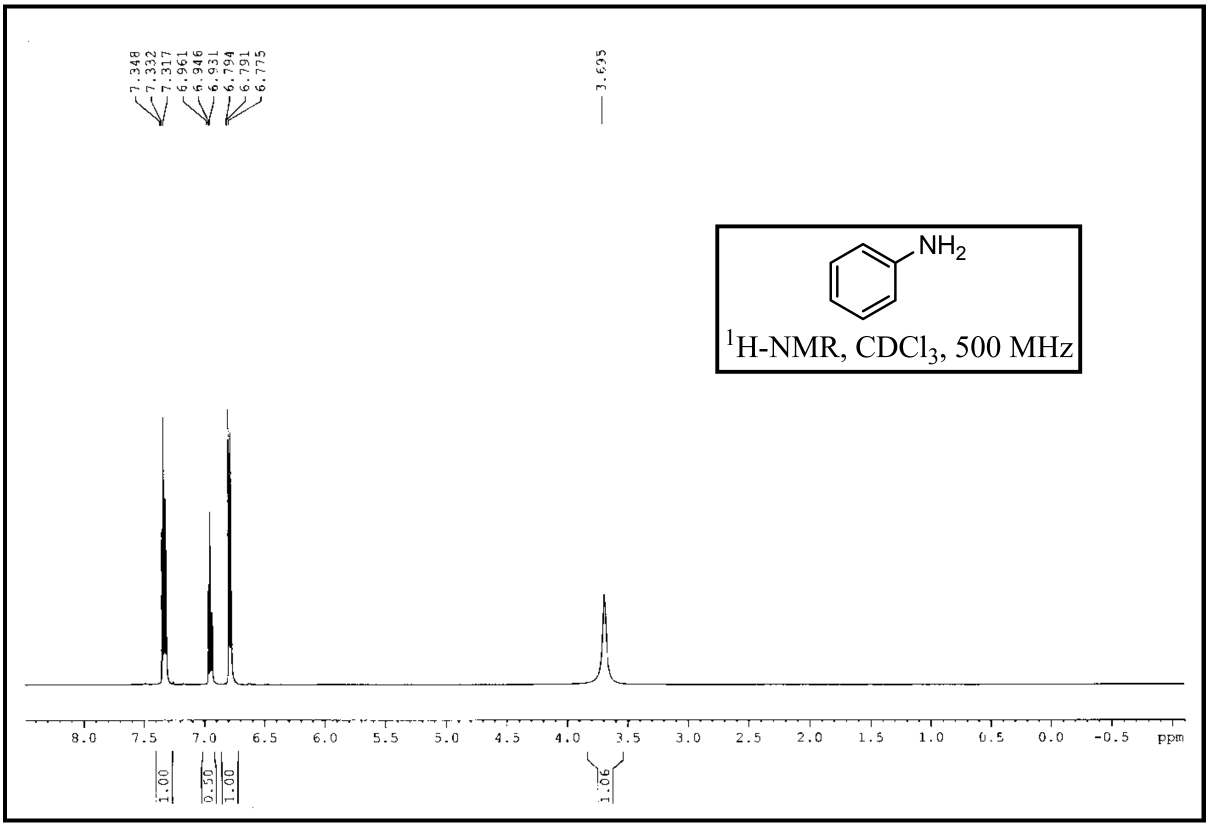


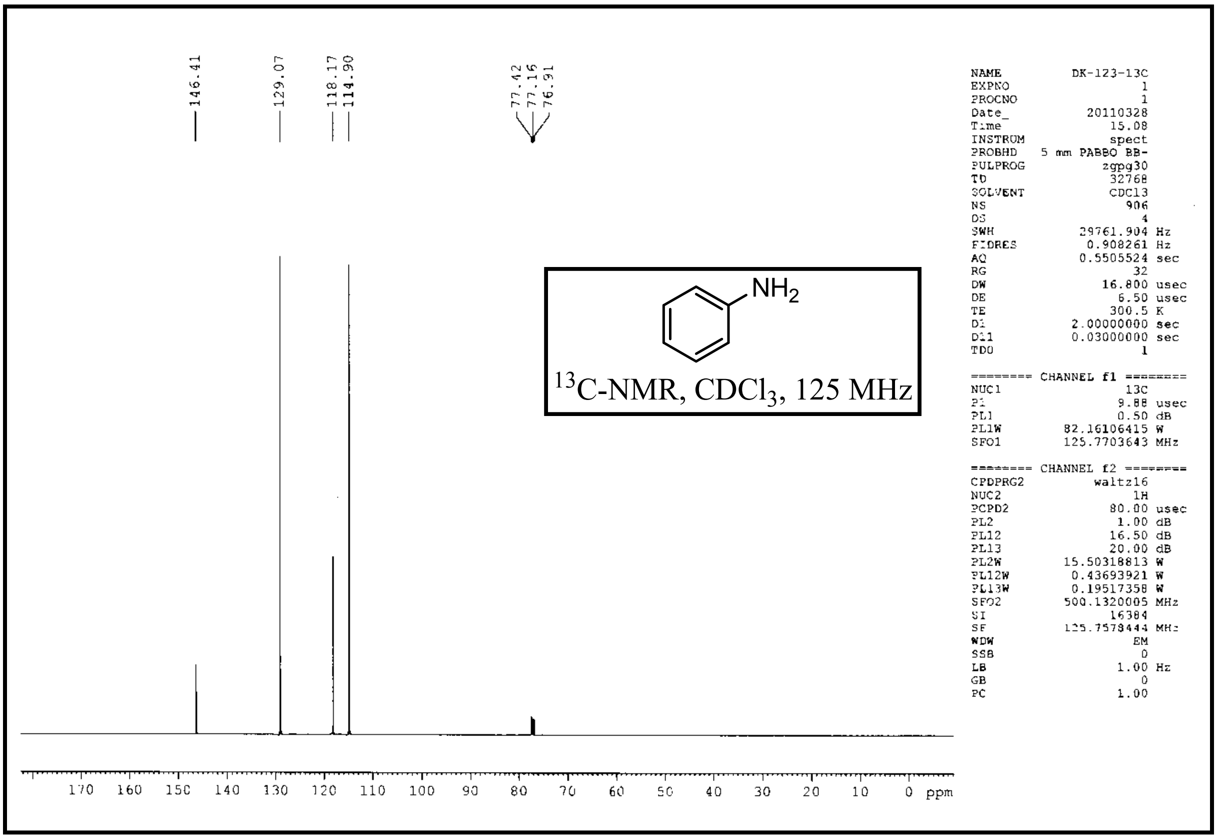
Characterization data of new compounds (Table 1, entries 18 and 19)

3-Aminocinnamyl acetate (Table 1, entry 18): Yellow gummy liquid, IR (Neat): 775, 966, 1024, 1247, 1456, 1600, 1732, 2929, 3032, 3367 cm^{-1}; ^{1}H NMR (CDCl$_3$, 500MHz) δ 2.09 (s, 3H), 4.70 (d, *J*=7 Hz, 2H), 6.21-6.26 (m, 1H), 6.54-6.60 (m, 2H), 6.71 (s, 1H), 6.79 (d, *J*=7.5Hz, 1H), 7.10 (t, *J*=7.5Hz, 1H); ^{13}C NMR(CDCl$_3$, 125 MHz) δ 21.1, 65.2, 113.2, 115.1, 117.4, 123.1, 129.6, 134.5, 137.4, 146.7, 170.9;
HRMS calcd for $C_{11}H_{14}NO_2[M+H]^+$: 192.1025; found: 192.1019.

3-((E)-3-(Phenylselanyl)prop-1-enyl)benzenamine (Table 1, entry 19): Yellow gummy liquid, IR (Neat): 690, 736, 777, 960, 1477, 1579, 1599, 1616, 2852, 2926, 3028, 3367 cm^{-1}; ^{1}H-NMR (CDCl$_3$, 500MHz) δ 3.68 (d, *J*=7.5 Hz, 2H), 6.17 (d, *J*=7.5, 2H), 6.25-6.31 (m, 1H), 6.54-6.56 (m, 1H), 6.61(s, 1H), 6.69 (d, *J*=7.5Hz, 1H), 7.07 (t, *J*=7.5Hz, 1H), 7.24-7.26 (m, 3H), 7.51-7.53 (m, 2H); ^{13}C-NMR (CDCl$_3$, 125 MHz) δ 30.8, 112.9, 114.6, 117.2, 125.9, 127.4, 129.1, 129.5, 132.4, 133.9, 138.1, 146.6; HRMS calcd for $C_{15}H_{16}NSe[M+H]^+$: 290.0448; found: 290.0469.

Copies of ^{1}H NMR and ^{13}C NMR spectra of all products in Table 1





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PROCNO                 1
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PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   906
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    32
DW                16.800 usec
DE                  6.50 usec
TE                 300.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7578444 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
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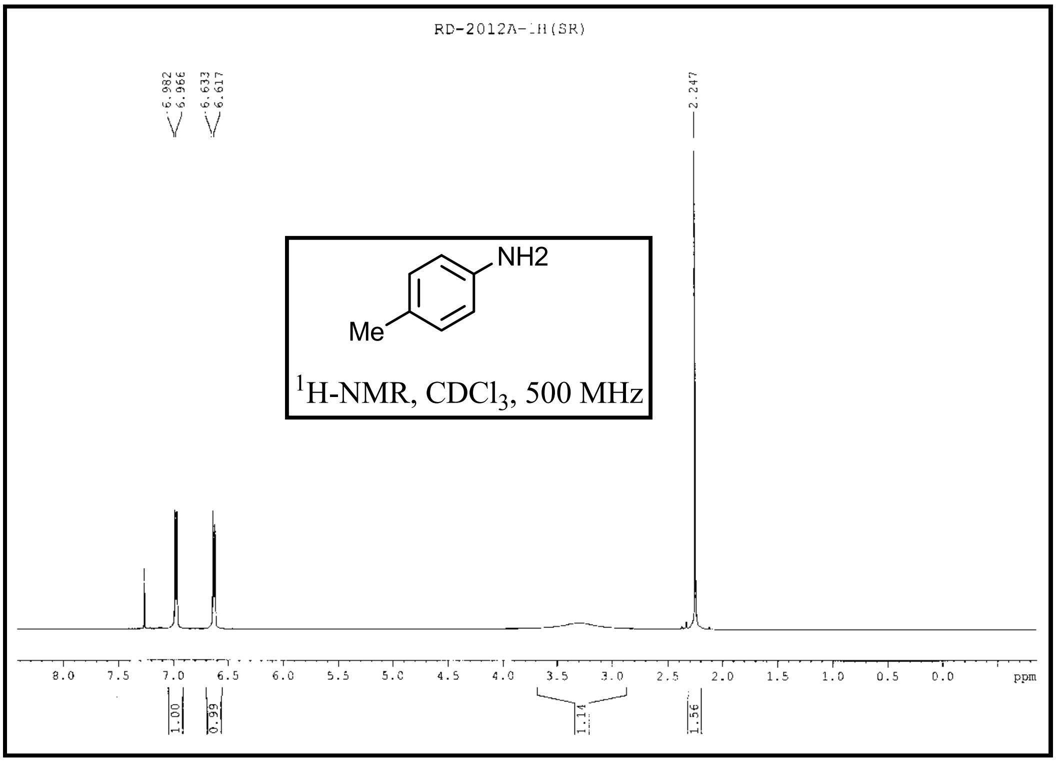
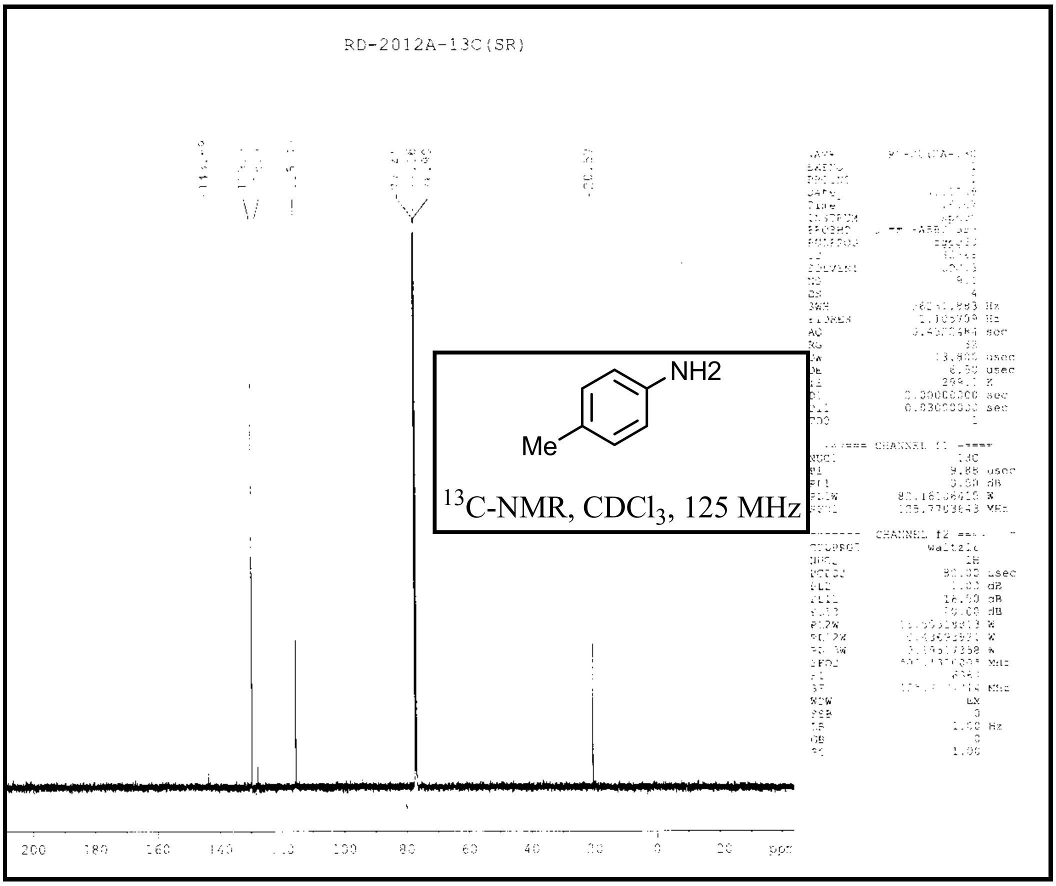
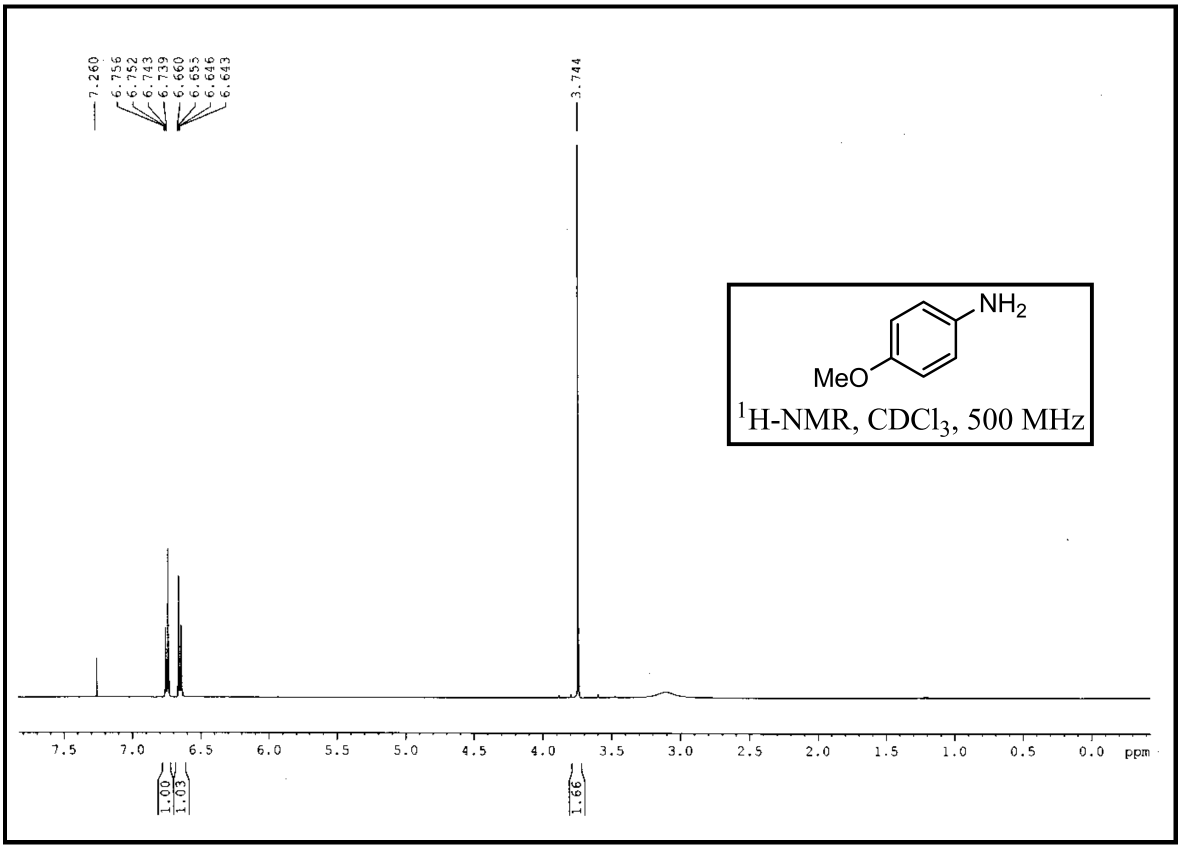
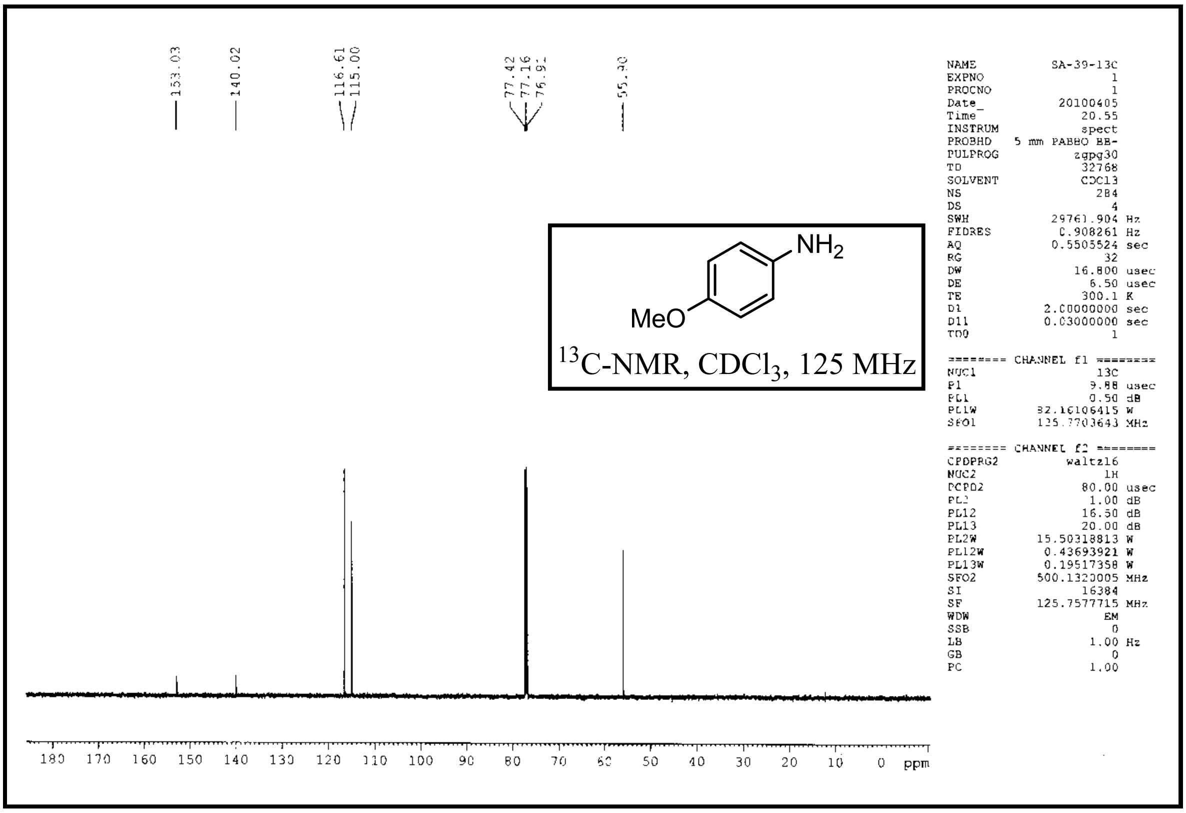

RD-2012A-1H(SR)
6.982
6.966
6.633
6.617
2.247
NH2
Me
^{1}H-NMR, CDCl$_3$, 500 MHz
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
0.99
1.14
1.56


RD-2012A-13C(SR)
NH2
Me
^{13}C-NMR, CDCl$_3$, 125 MHz
200 180 160 140 120 100 80 60 40 20 0 -20 ppm





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TD                   32768
SOLVENT              CDCl3
NS                     284
DS                       4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                      32
DW                  16.800 usec
DE                    6.50 usec
TE                   300.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577715 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
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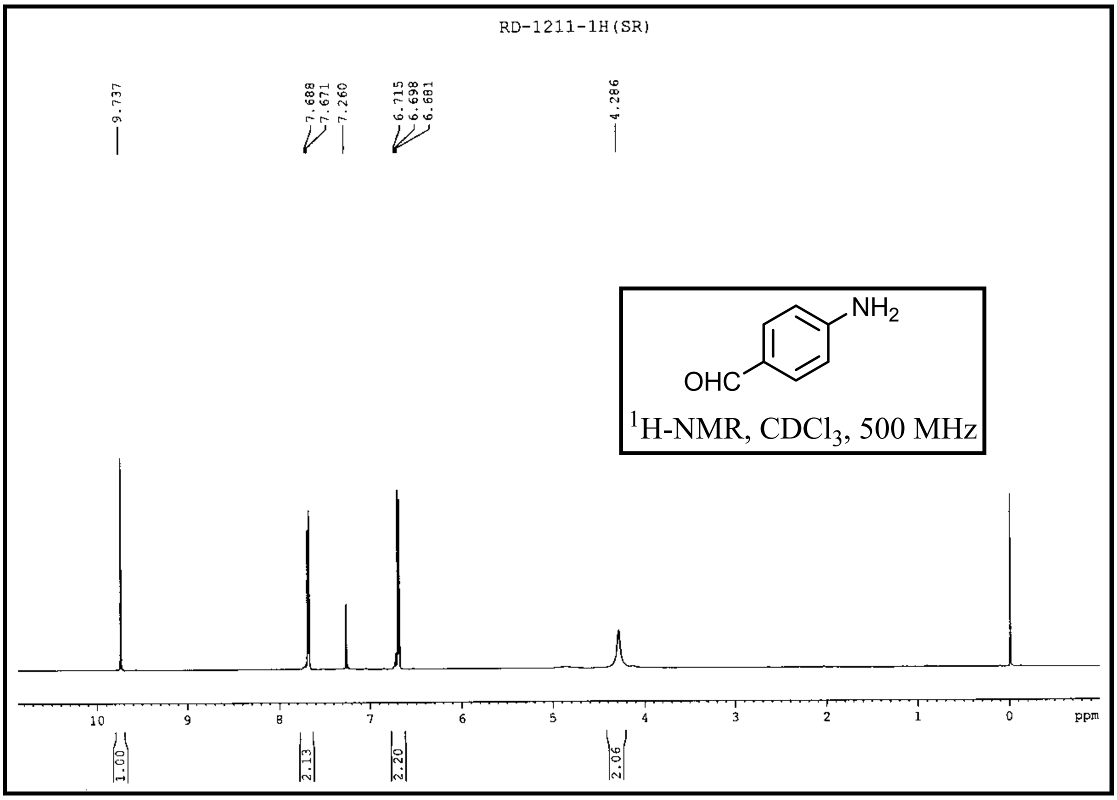

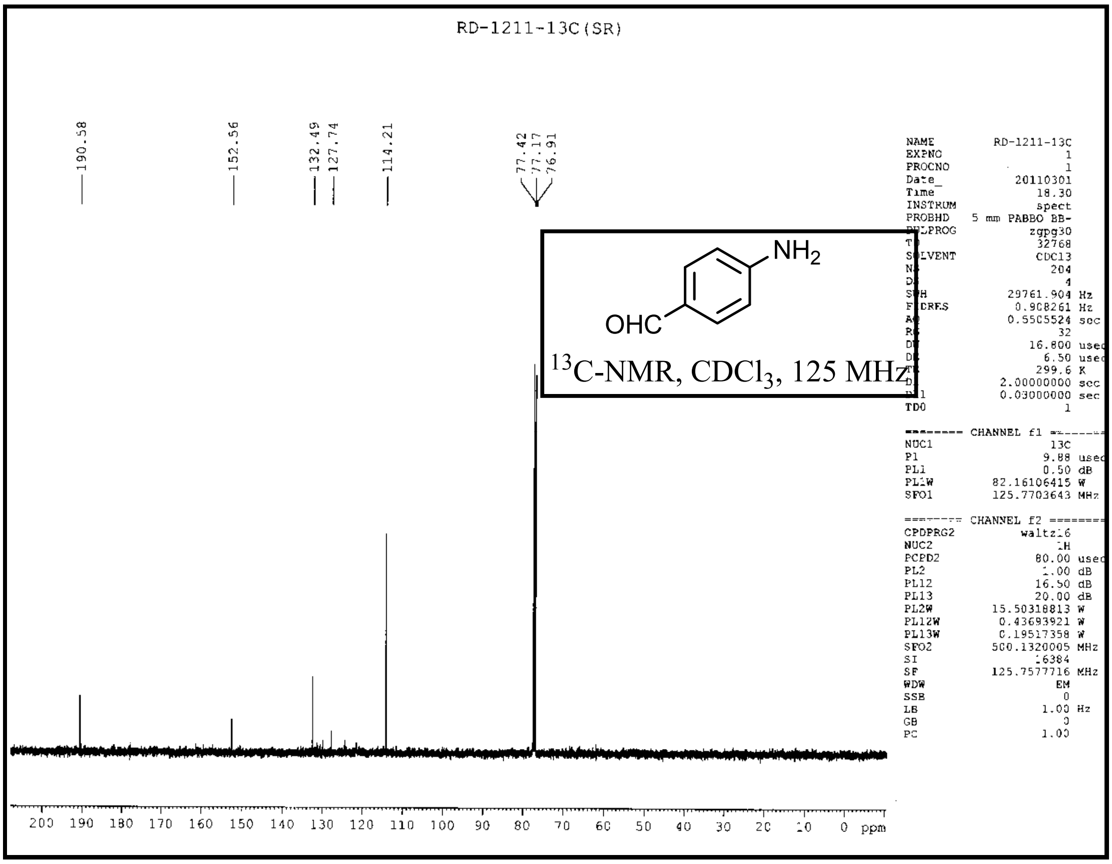

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  LPROG              zgpg30
T                     32768
S LVENT               CDC13
N                       204
D                         4
S H              29761.904 Hz
F DRES            0.908261 Hz
A                0.5505524 sec
R                        32
D                    16.800 use
D                      6.50 use
T                     299.6 K
D                2.00000000 sec
  1              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.88 use
PL1                    0.50 dB
PL1W            82.16106415 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 use
PL2                    1.00 dB
PL12                  16.50 dB
PL13                  20.00 dB
PL2W            15.50318813 W
PL12W            0.43693921 W
PL13W            0.19517358 W
SFO2            500.1320005 MHz
SI                    16384
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WDW                      EM
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LB                     1.00 Hz
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PC                     1.00
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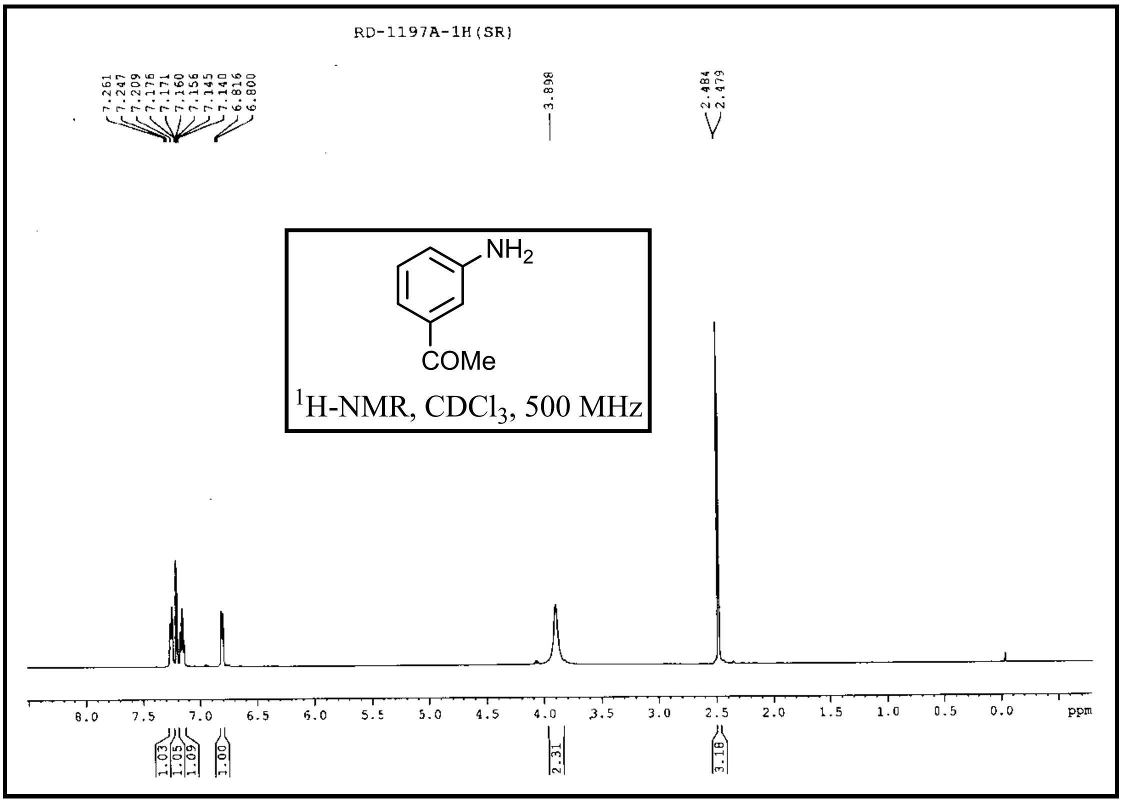

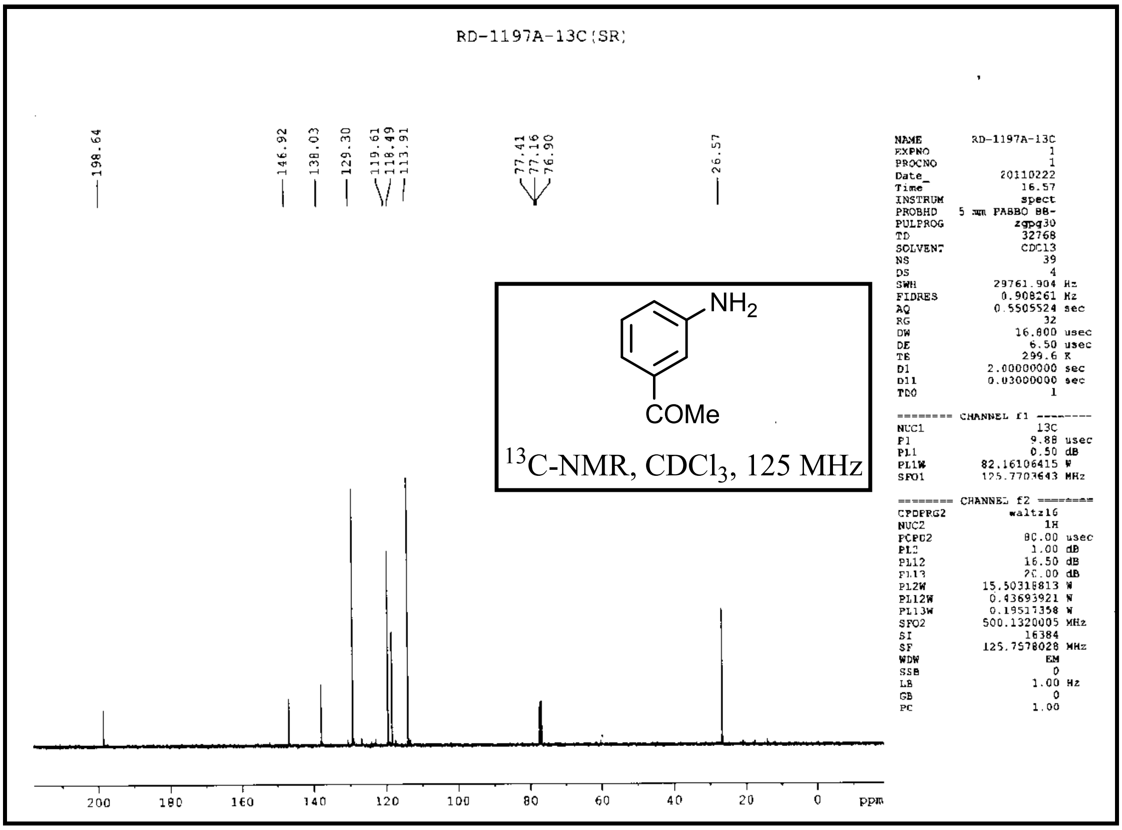

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PROCNO                   1
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TD                  32768
SOLVENT             CDCl3
NS                     39
DS                      4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              0.5505524 sec
RG                     32
DW                 16.800 usec
DE                   6.50 usec
TE                  299.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 --------
NUC1                  13C
P1                   9.88 usec
PL1                  0.50 dB
PL1W          82.16106415 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                16.50 dB
PL13                20.00 dB
PL2W          15.50318813 W
PL12W          0.43693921 W
PL13W          0.19517358 W
SFO2          500.1320005 MHz
SI                  16384
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WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
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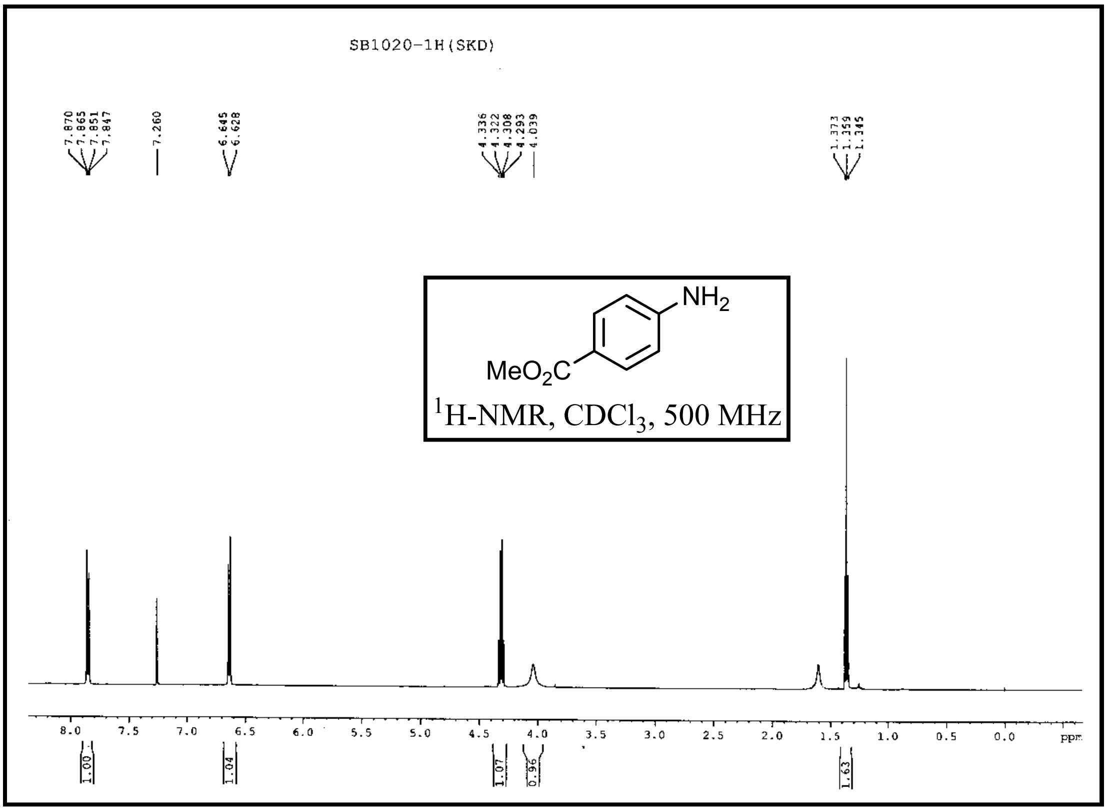

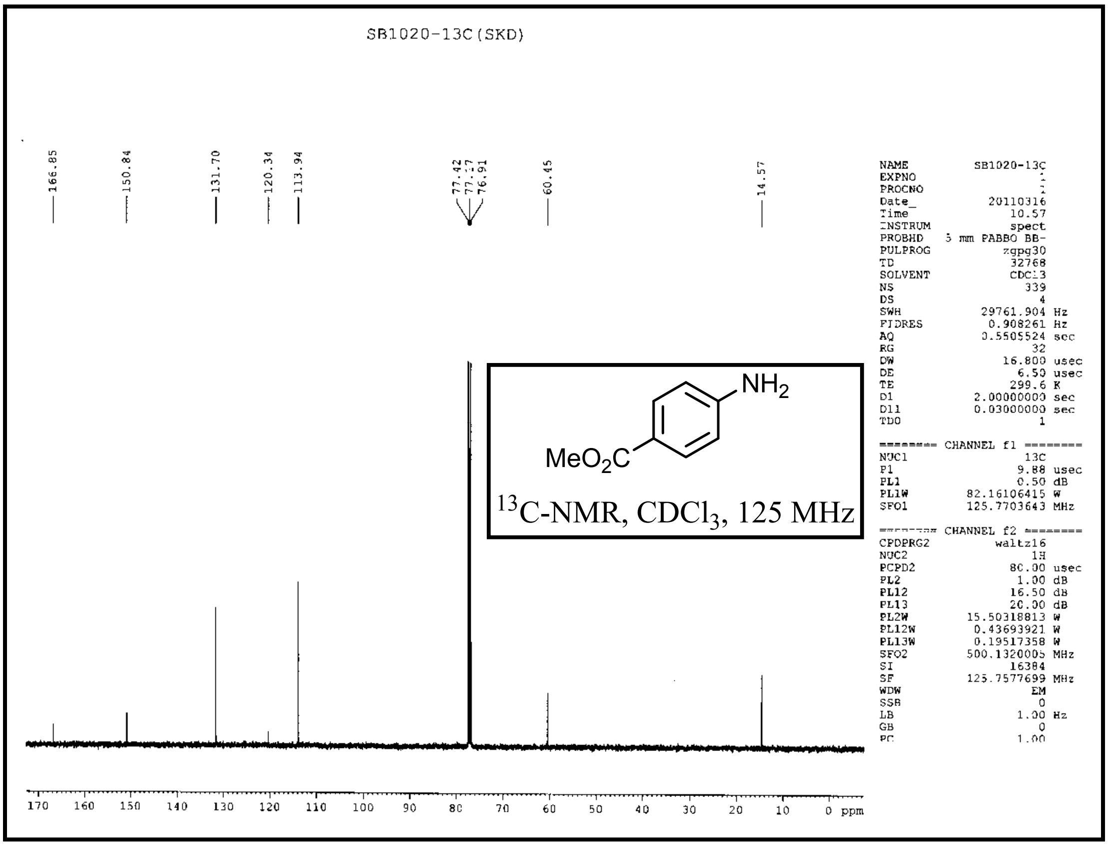

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DE                  6.50 usec
TE                 299.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577699 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
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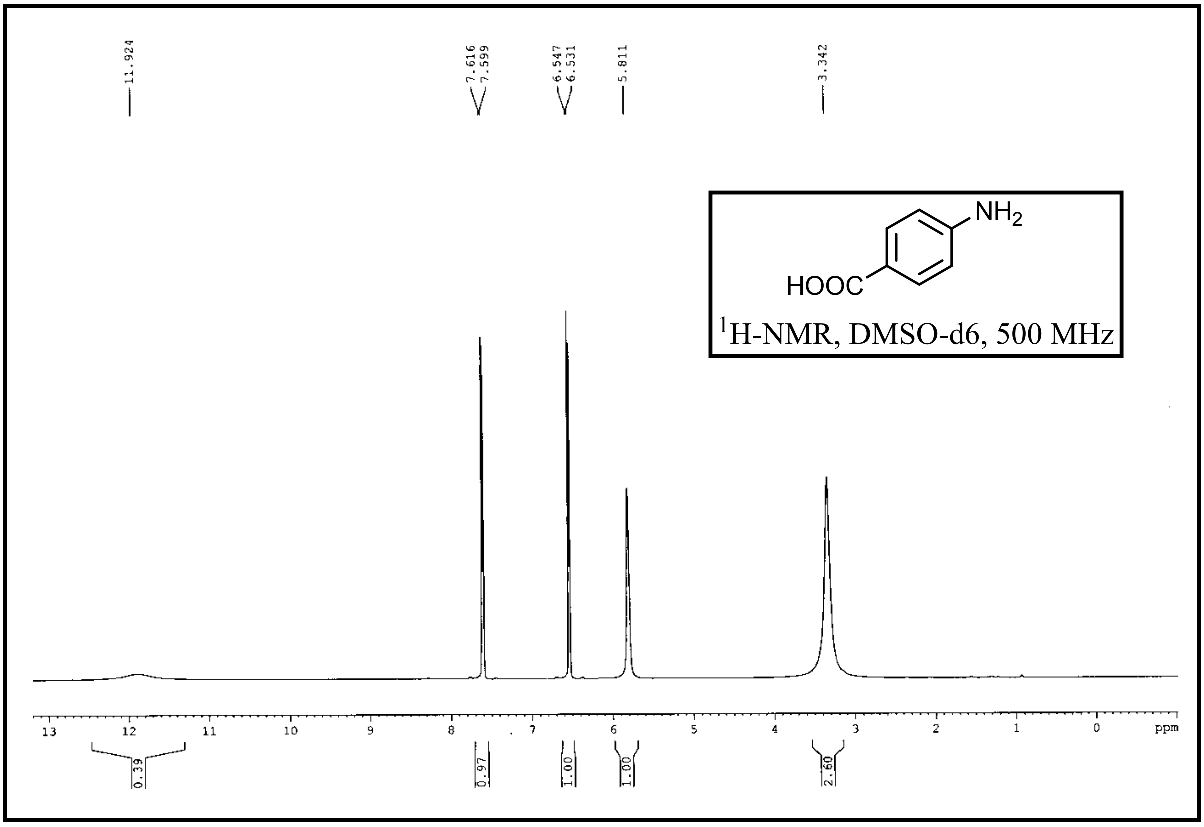
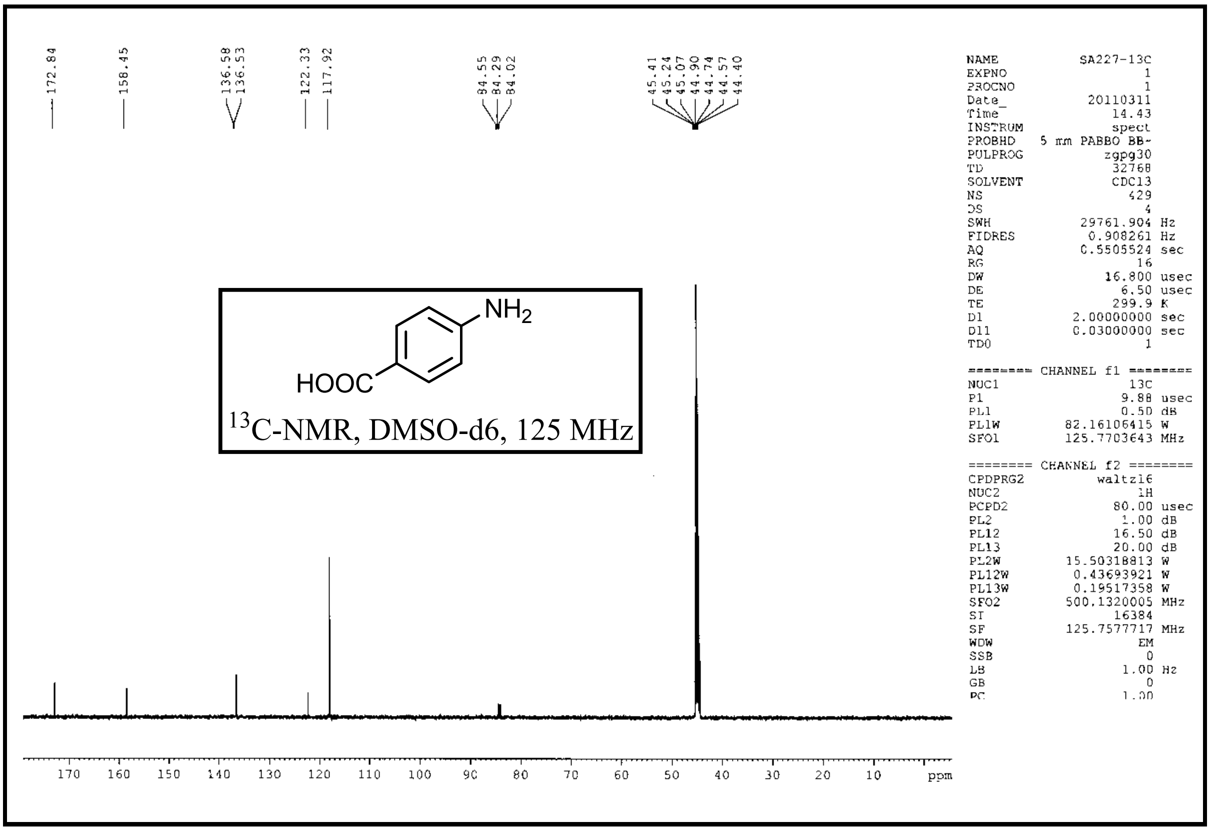


^{1}H-NMR, DMSO-d6, 500 MHz



^{13}C-NMR, DMSO-d6, 125 MHz

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PROCNO                   1
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TD                   32768
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NS                     429
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FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                      16
DW                  16.800 usec
DE                    6.50 usec
TE                   299.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577717 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
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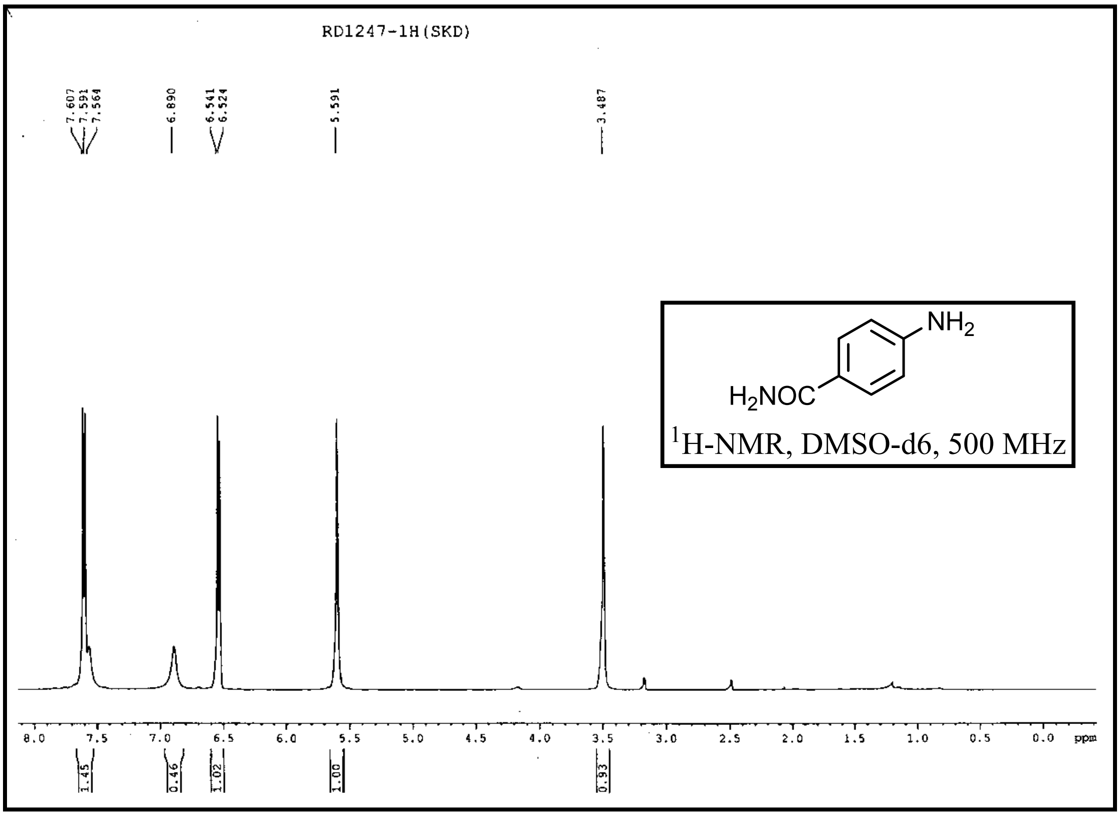
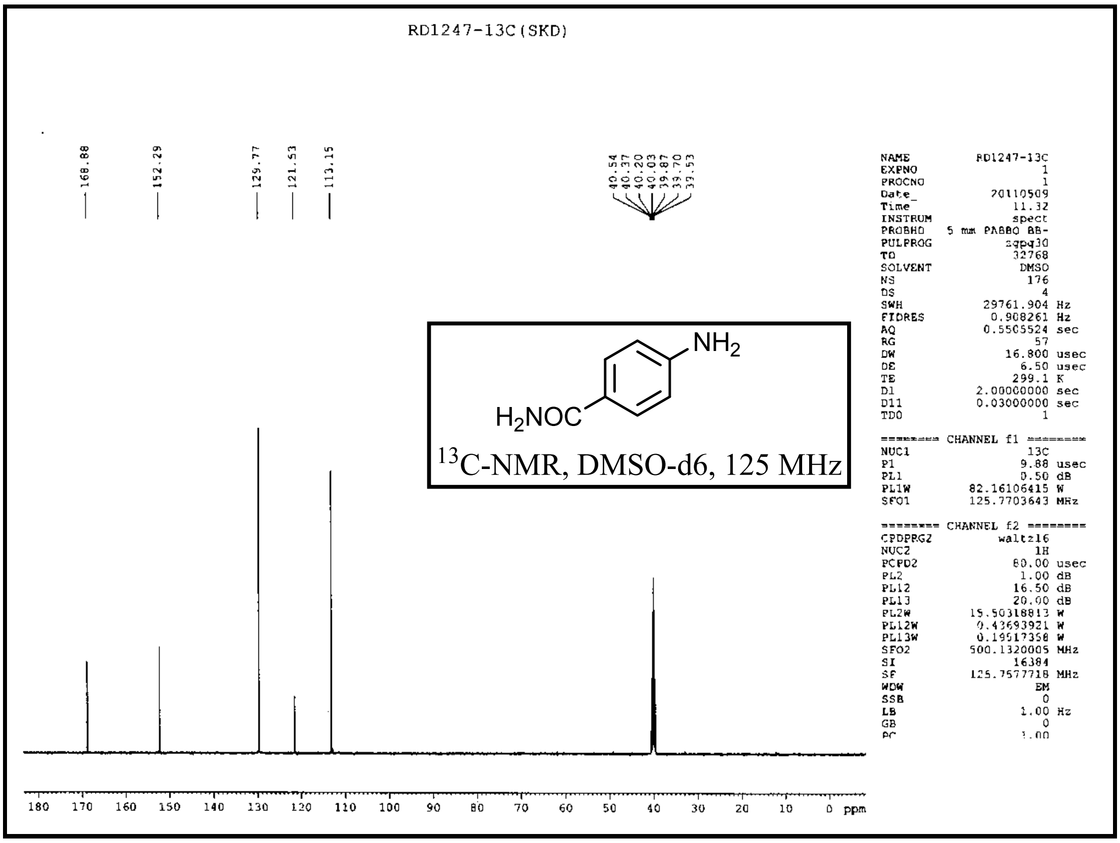
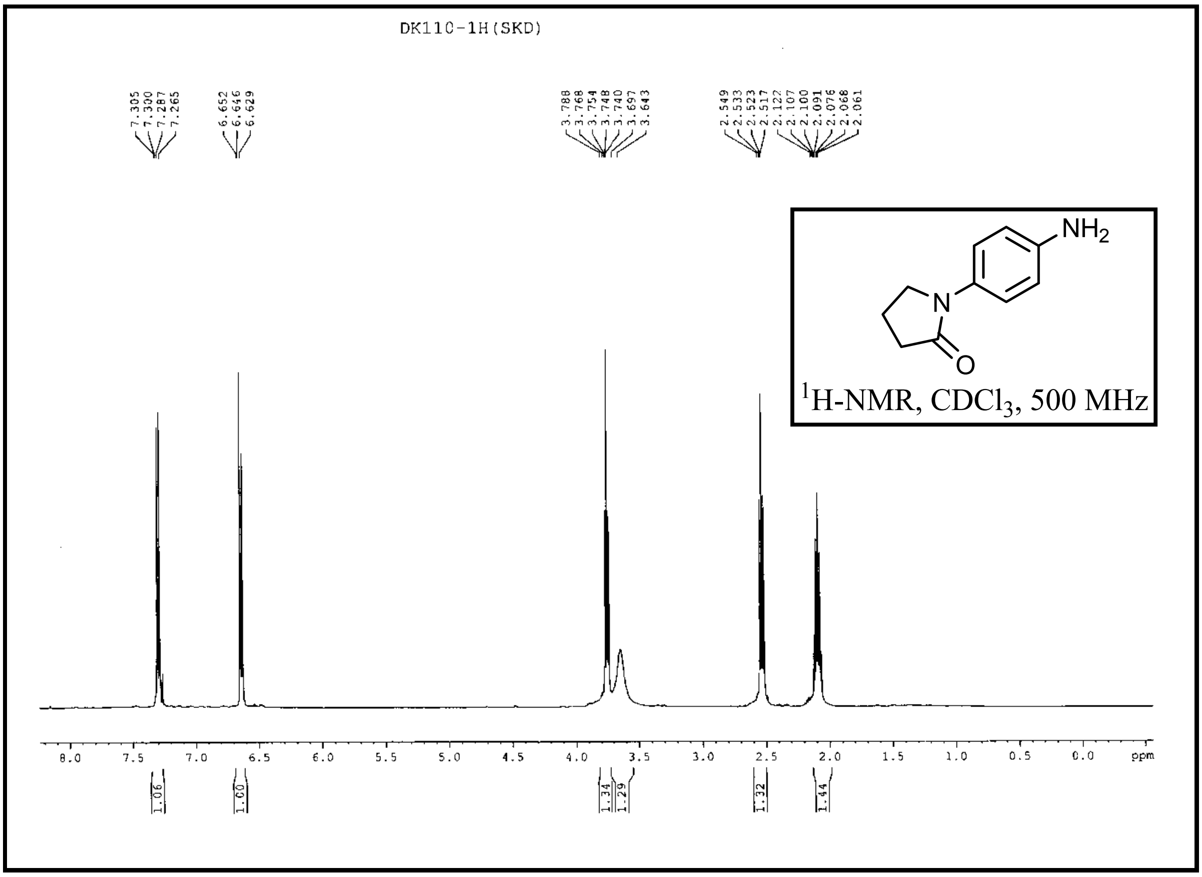


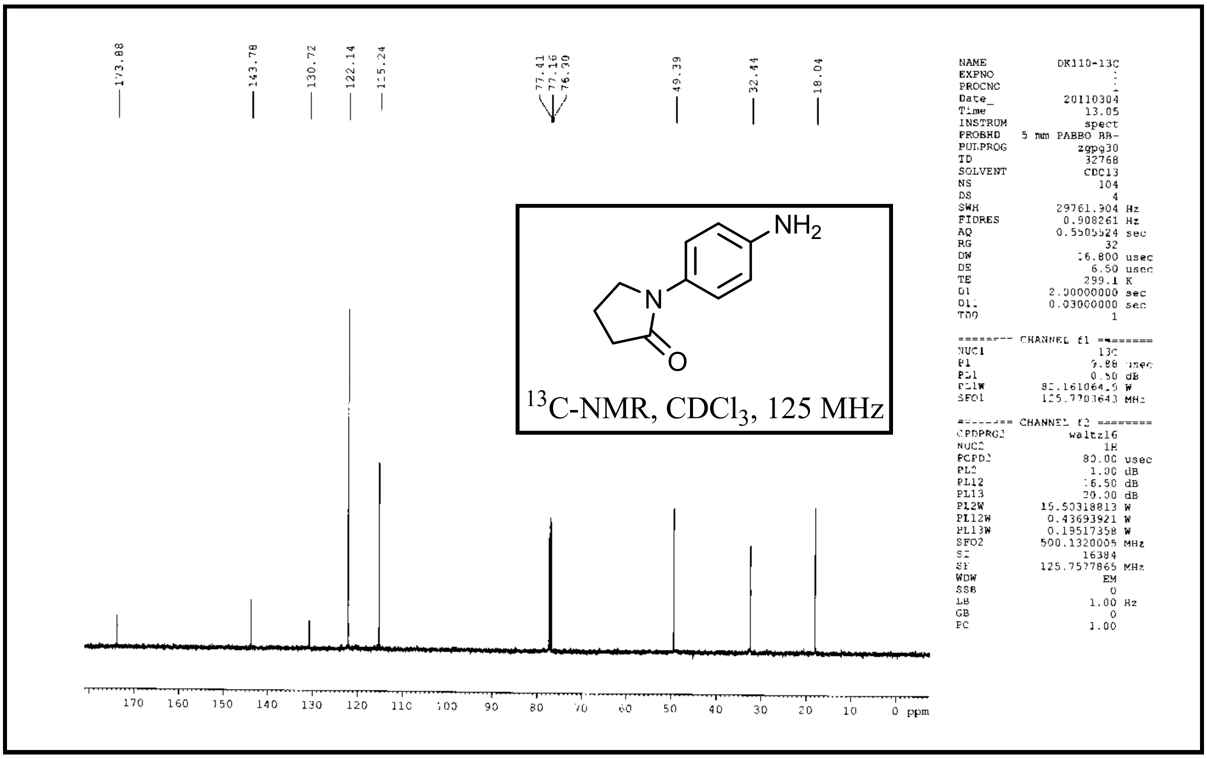
^{1}H-NMR, DMSO-d6, 500 MHz



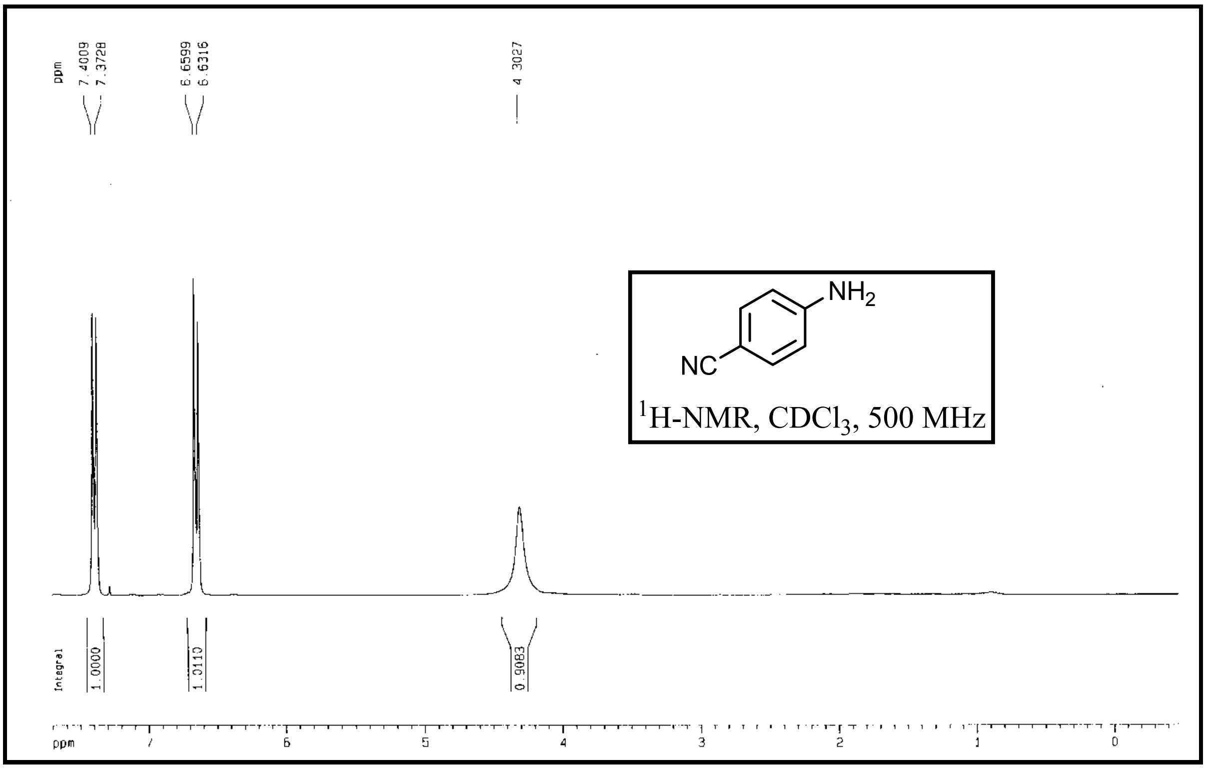
^{13}C-NMR, DMSO-d6, 125 MHz

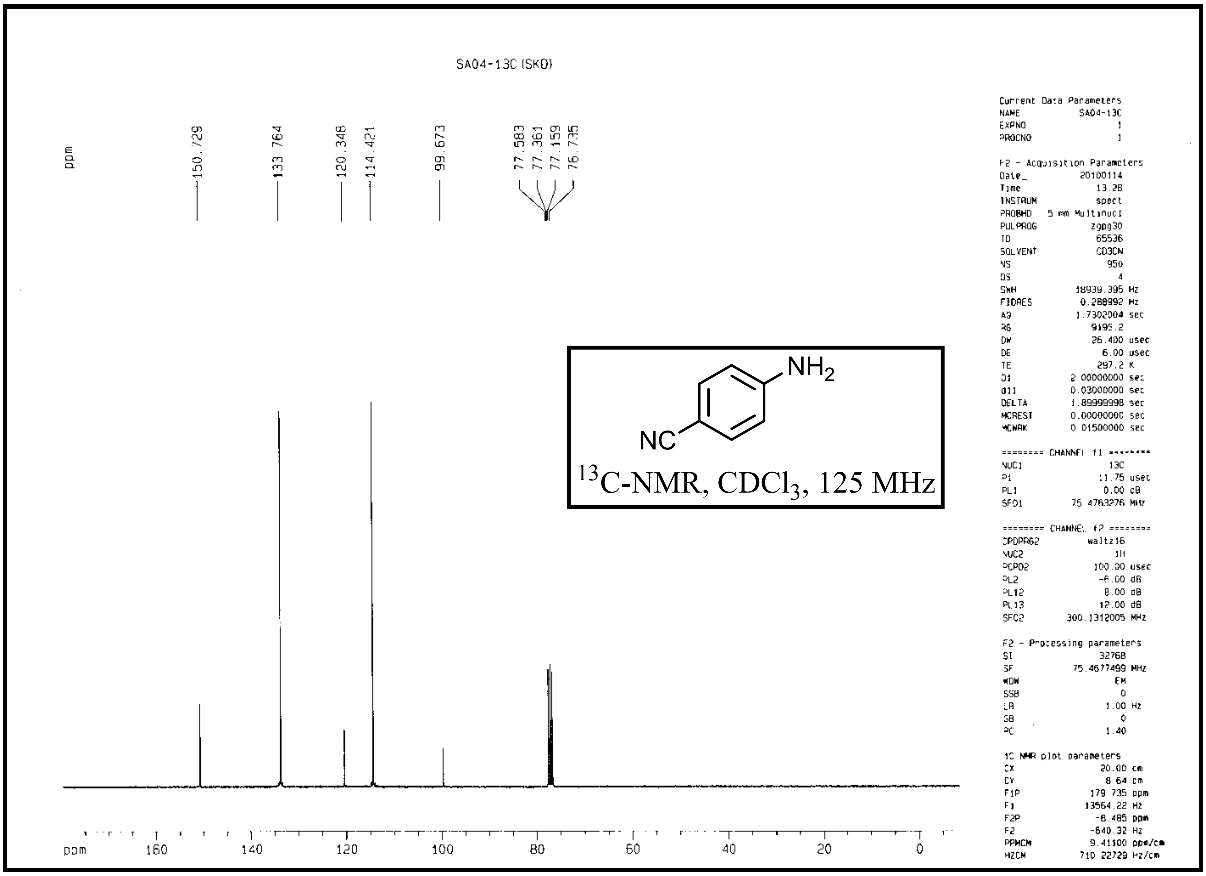
```
NAME             RD1247-13C
EXPNO                     1
PROCNO                    1
Date_              20110509
Time                  11.32
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    32768
SOLVENT                DMSO
NS                      176
DS                        4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                       57
DW                   16.800 usec
DE                     6.50 usec
TE                    299.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.88 usec
PL1                    0.50 dB
PL1W            82.16106415 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  16.50 dB
PL13                  20.00 dB
PL2W            15.50318813 W
PL12W            0.43693921 W
PL13W            0.19517358 W
SFO2            500.1320005 MHz
SI                    16384
SF              125.7577718 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```



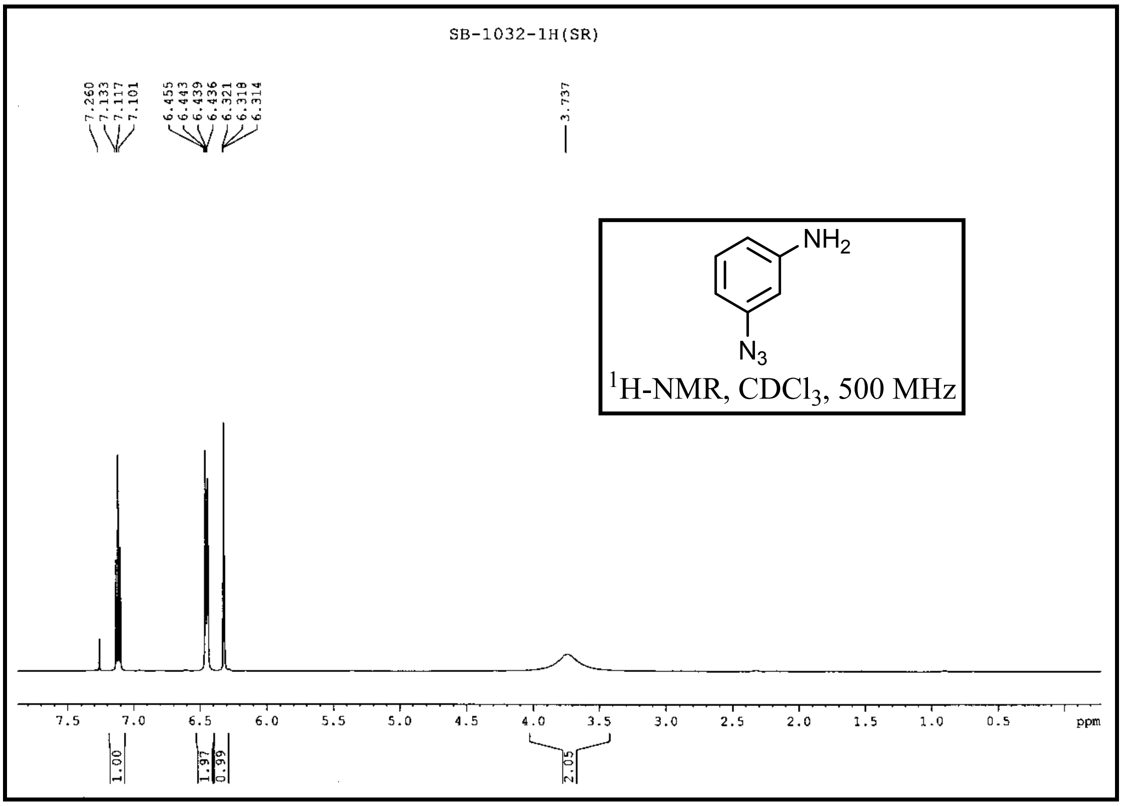


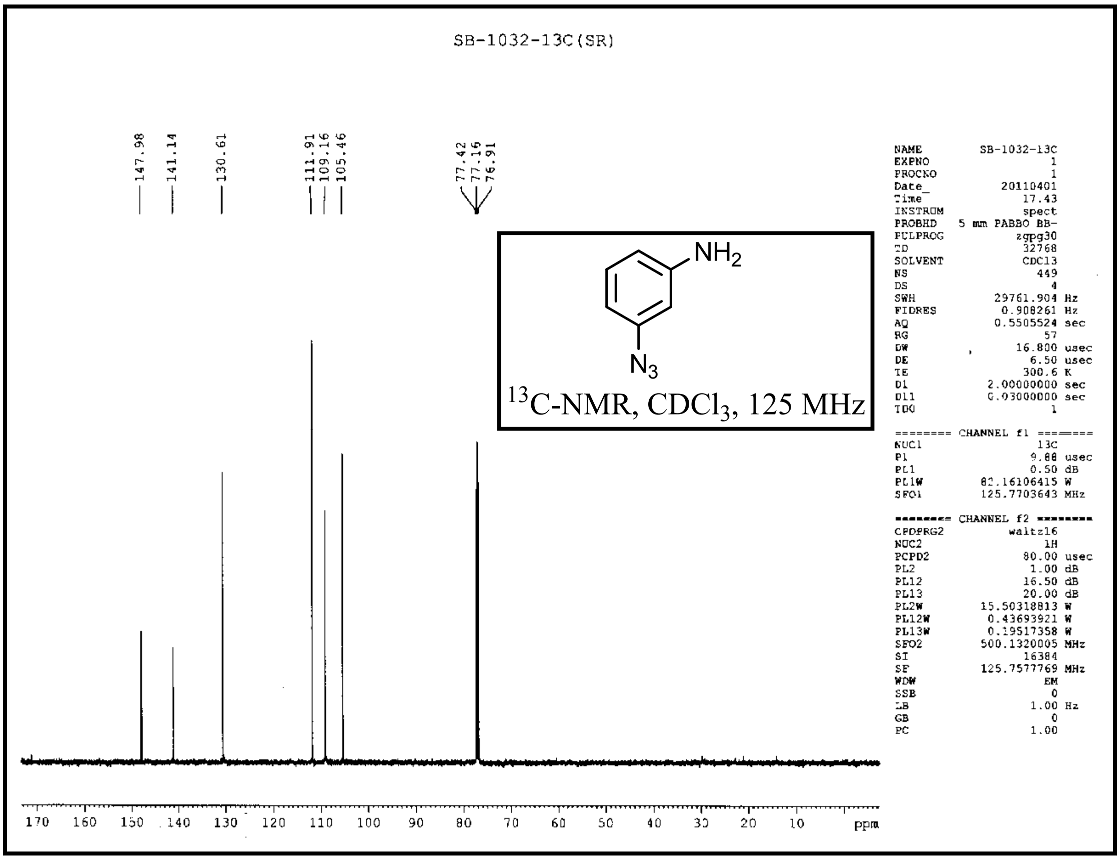
```
NAME              DK110-13C
EXPNO                     1
PROCNO                    1
Date_              20110304
Time                  13.05
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      104
DS                        4
SWH               29761.904 Hz
FIDRES             0.908261 Hz
AQ                0.5505524 sec
RG                       32
DW                   16.800 usec
DE                     6.50 usec
TE                    299.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.88 usec
PL1                    0.50 dB
PL1W             82.1610645 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  16.50 dB
PL13                  20.00 dB
PL2W            15.50318813 W
PL12W            0.43693921 W
PL13W            0.19517358 W
SFO2            500.1320005 MHz
SI                    16384
SF              125.7577865 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```



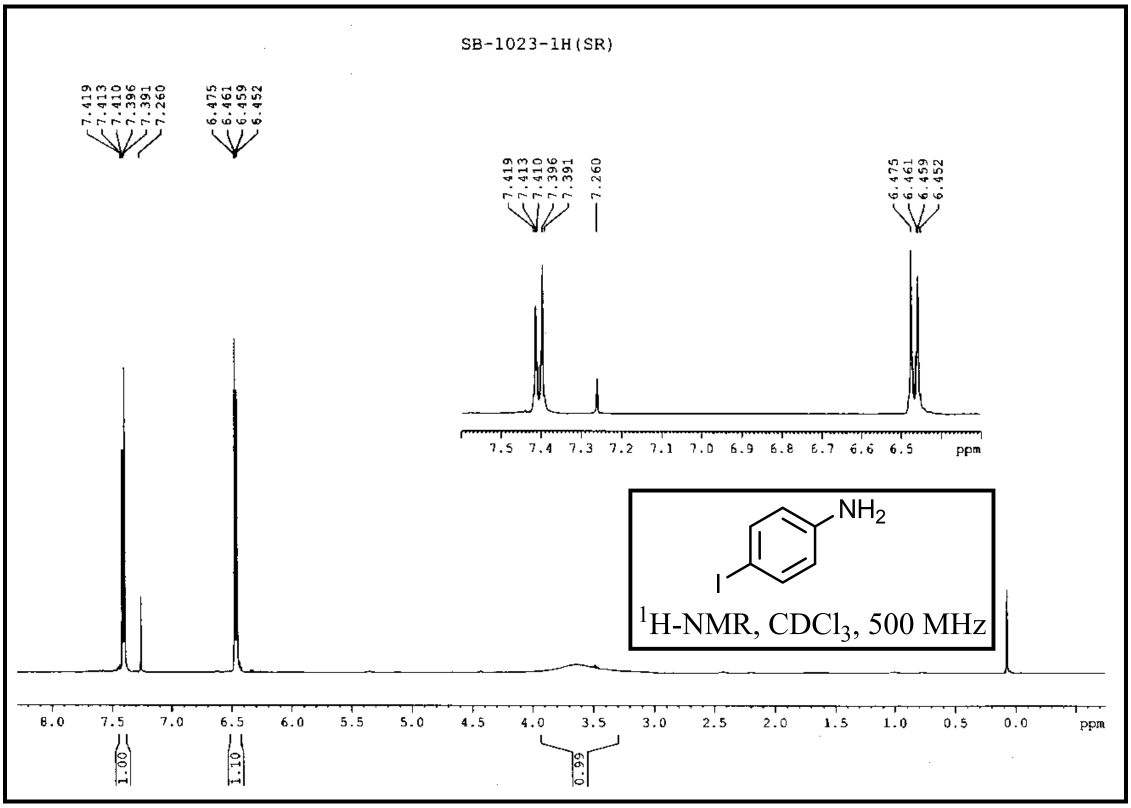


```
Current Data Parameters
NAME             SA04-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100114
Time                13.28
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                  65536
SOLVENT             CD3CN
NS                    950
DS                      4
SWH            18939.395 Hz
FIDRES          0.288992 Hz
AQ             1.7302004 sec
RG                 9195.2
DW                 26.400 usec
DE                   6.00 usec
TE                  297.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.75 usec
PL1                  0.00 dB
SFO1           75.4763276 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -6.00 dB
PL12                 8.00 dB
PL13                12.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF             75.4677499 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   8.64 cm
F1P               179.735 ppm
F1               13564.22 Hz
F2P                -8.485 ppm
F2                -640.32 Hz
PPMCM             9.41100 ppm/cm
HZCM            710.22729 Hz/cm
```

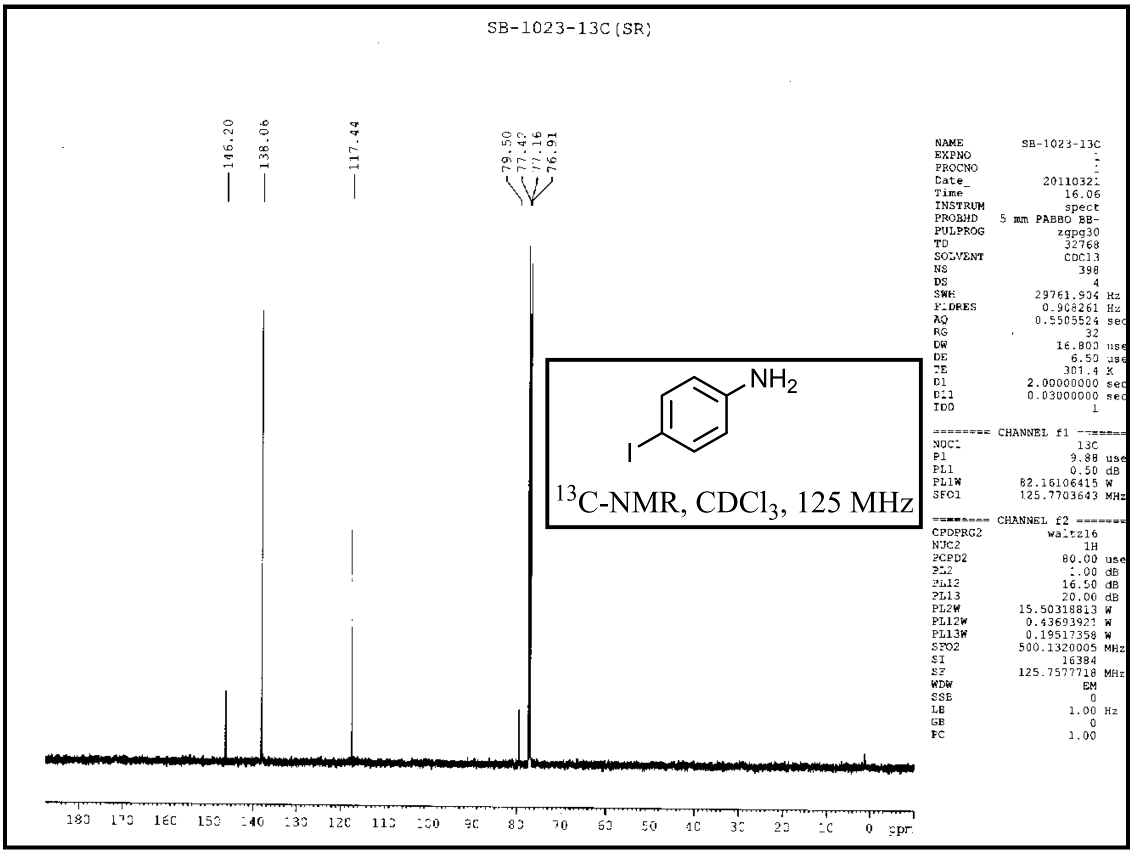




```
NAME          SB-1032-13C
EXPNO                   1
PROCNO                  1
Date_            20110401
Time                17.43
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    449
DS                      4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                     57
DW                 16.800 usec
DE                   6.50 usec
TE                  300.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.88 usec
PL1                  0.50 dB
PL1W          82.16106415 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                16.50 dB
PL13                20.00 dB
PL2W          15.50318813 W
PL12W          0.43693921 W
PL13W          0.19517358 W
SFO2          500.1320005 MHz
SI                  16384
SF            125.7577769 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

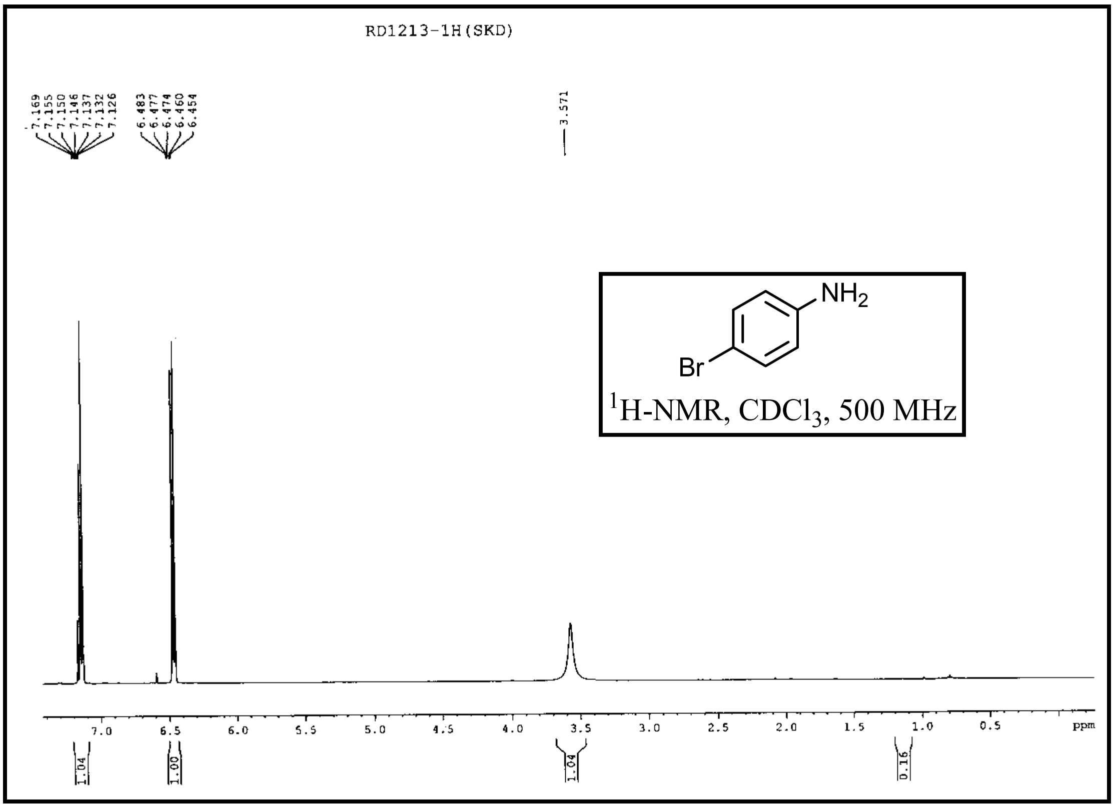


^{1}H-NMR, $CDCl_3$, 500 MHz

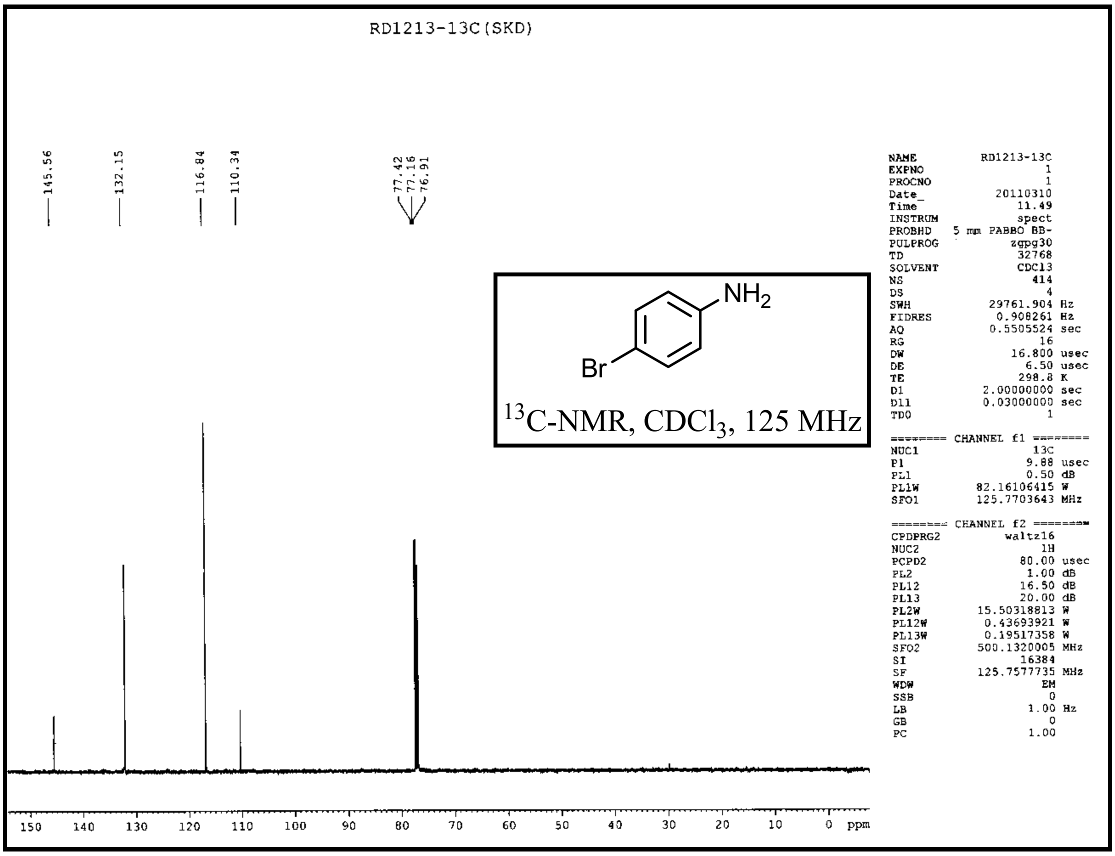


```
NAME            SB-1023-13C
EXPNO                     1
PROCNO                    1
Date_              20110321
Time                  16.06
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      398
DS                        4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                       32
DW                   16.800 use
DE                     6.50 use
TE                    301.4 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.88 use
PL1                    0.50 dB
PL1W            82.16106415 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 use
PL2                    1.00 dB
PL12                  16.50 dB
PL13                  20.00 dB
PL2W            15.50318813 W
PL12W            0.43693921 W
PL13W            0.19517358 W
SFO2            500.1320005 MHz
SI                    16384
SF              125.7577718 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```

^{13}C-NMR, $CDCl_3$, 125 MHz

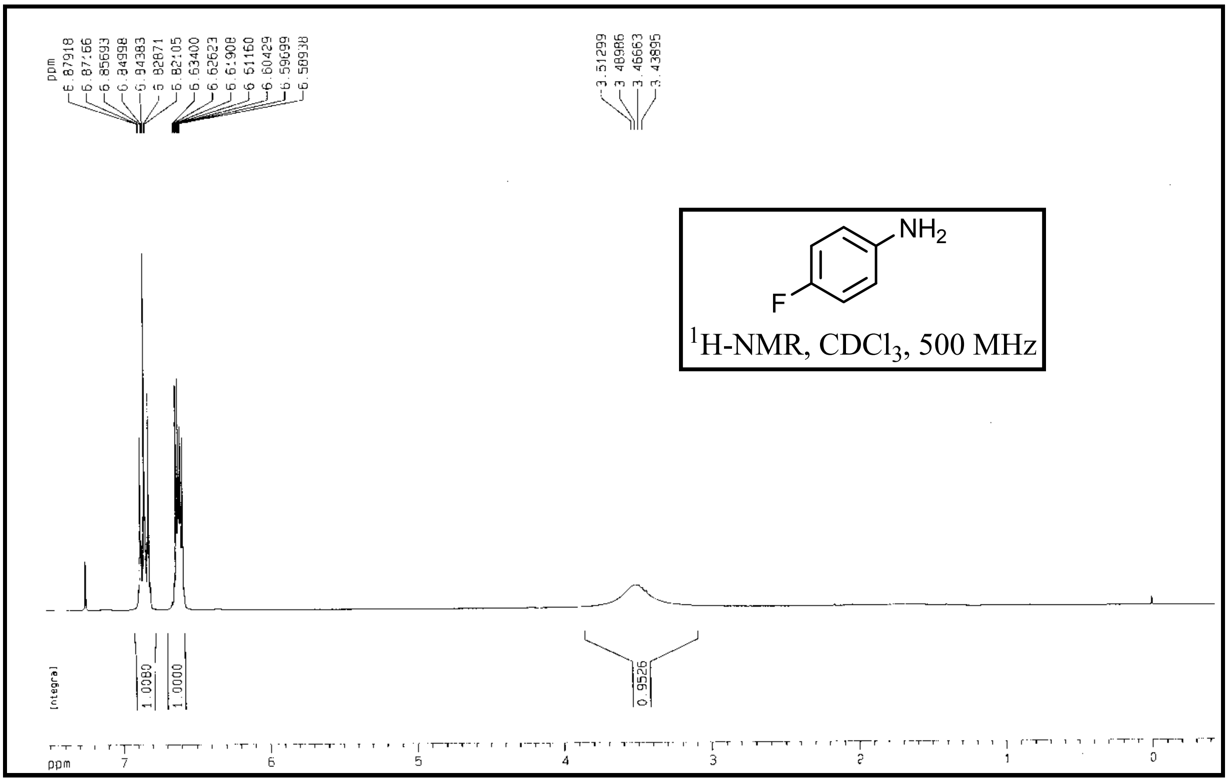


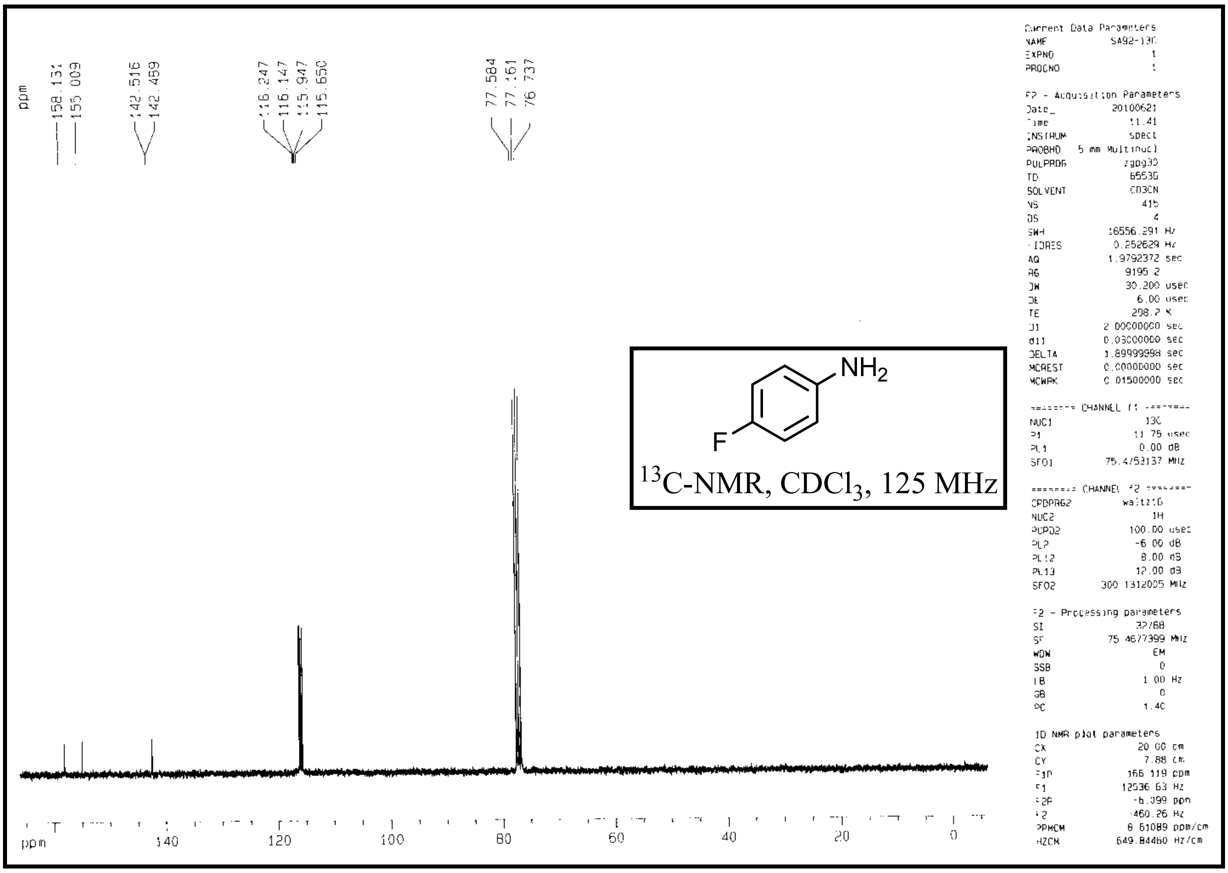
^{1}H-NMR, $CDCl_3$, 500 MHz



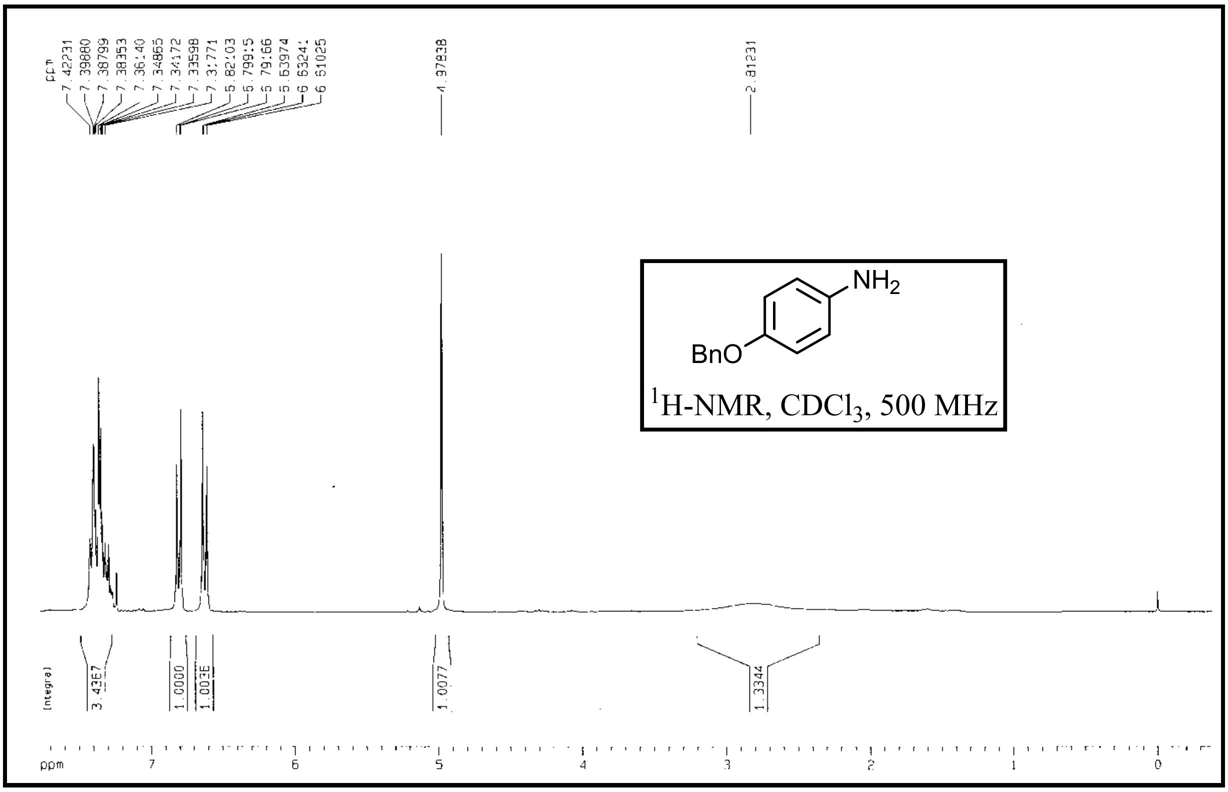
^{13}C-NMR, $CDCl_3$, 125 MHz

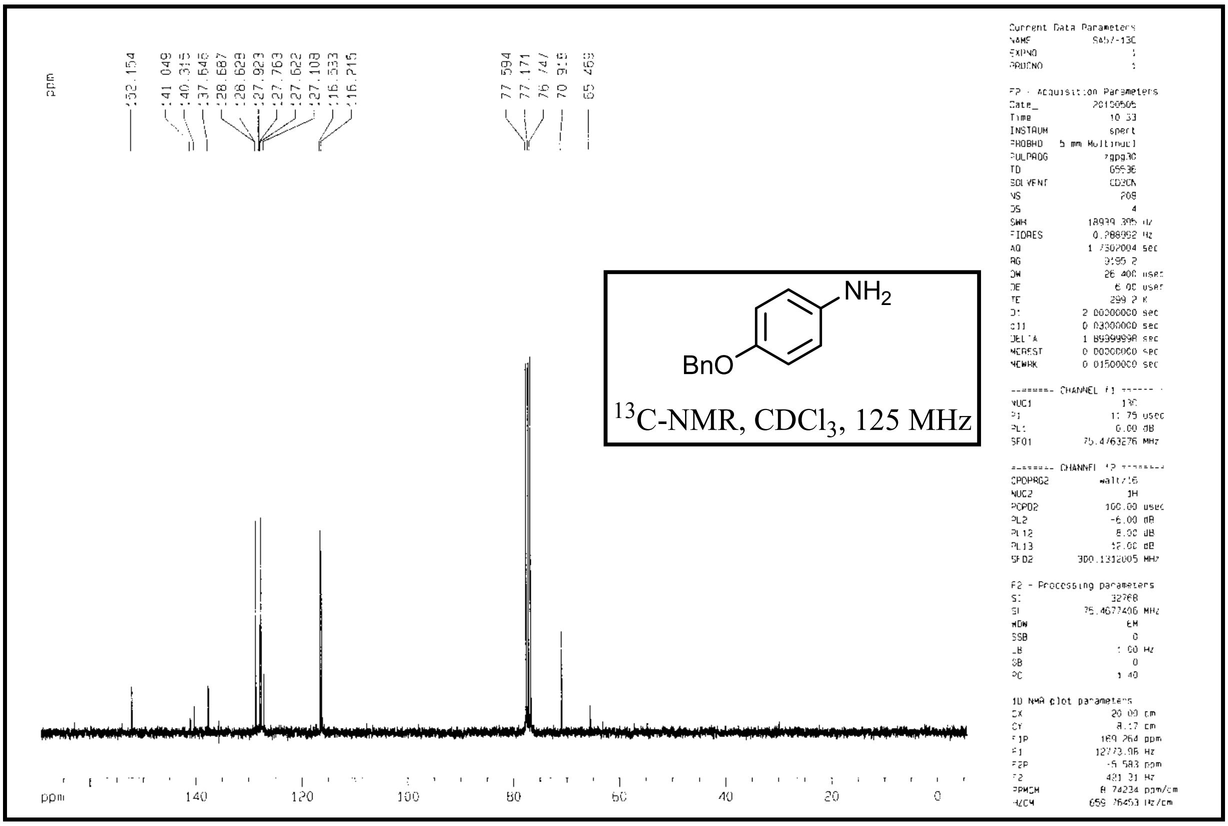
```
NAME          RD1213-13C
EXPNO                  1
PROCNO                 1
Date_           20110310
Time               11.49
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   414
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    16
DW                16.800 usec
DE                  6.50 usec
TE                 298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577735 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```



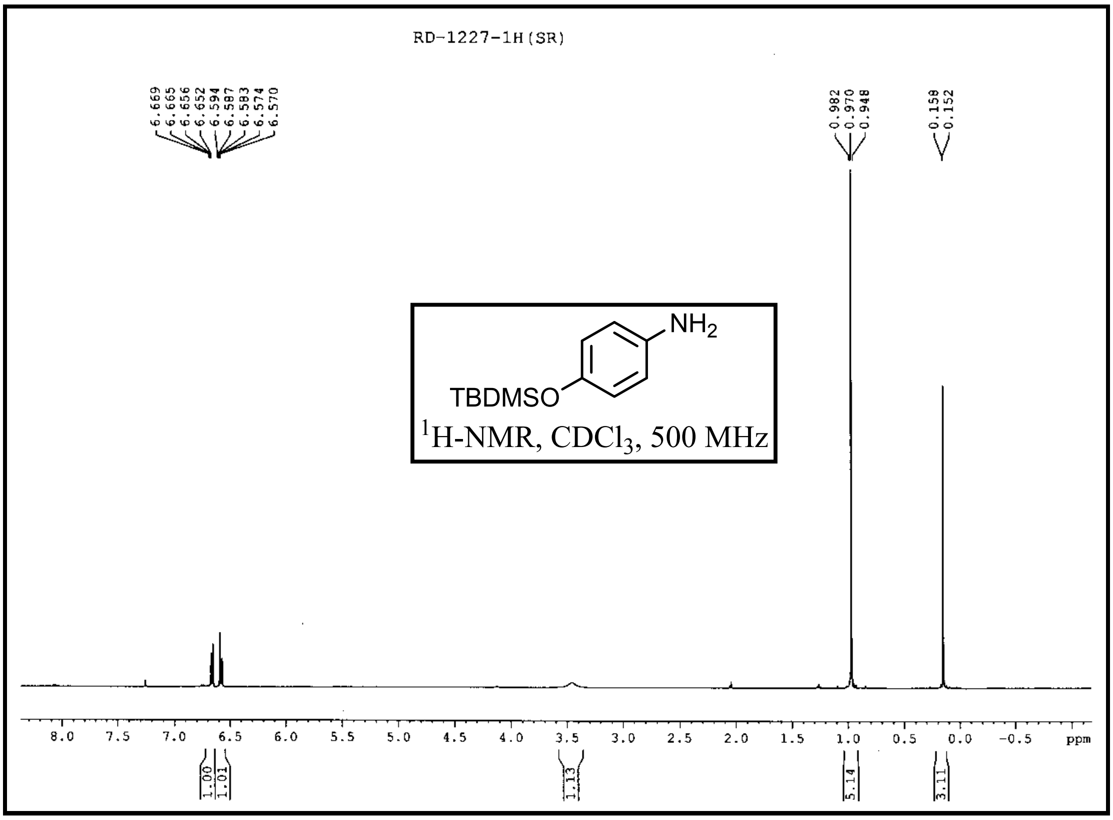


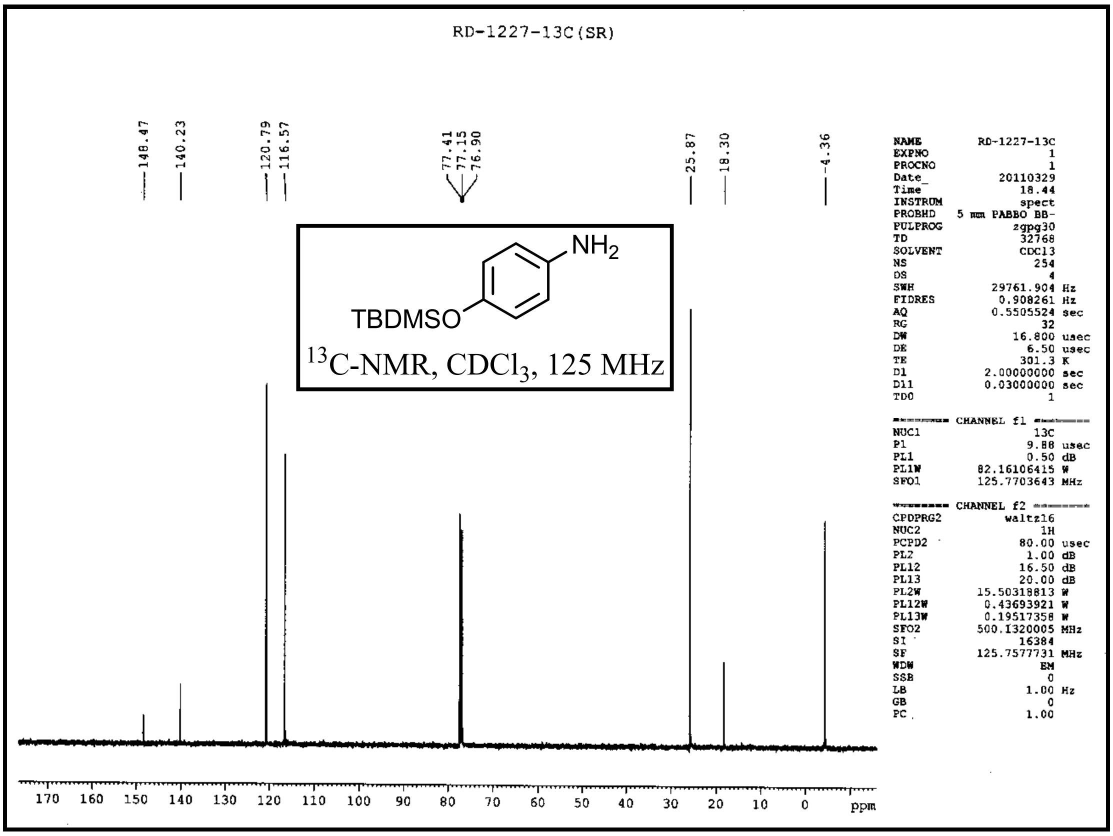
```
Current Data Parameters
NAME            SA92-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100621
Time               11.41
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                 65536
SOLVENT            CD3CN
NS                   415
DS                     4
SWH            16556.291 Hz
FIDRES          0.252629 Hz
AQ             1.9792372 sec
AG                9195.2
DW                30.200 usec
DE                  6.00 usec
TE                 298.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1                 0.00 dB
SFO1         75.4753137 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12                8.00 dB
PL13               12.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677399 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  7.88 cm
F1P              166.119 ppm
F1              12536.63 Hz
F2P               -6.099 ppm
F2               -460.26 Hz
PPMCM            8.61089 ppm/cm
HZCM           649.84460 Hz/cm
```



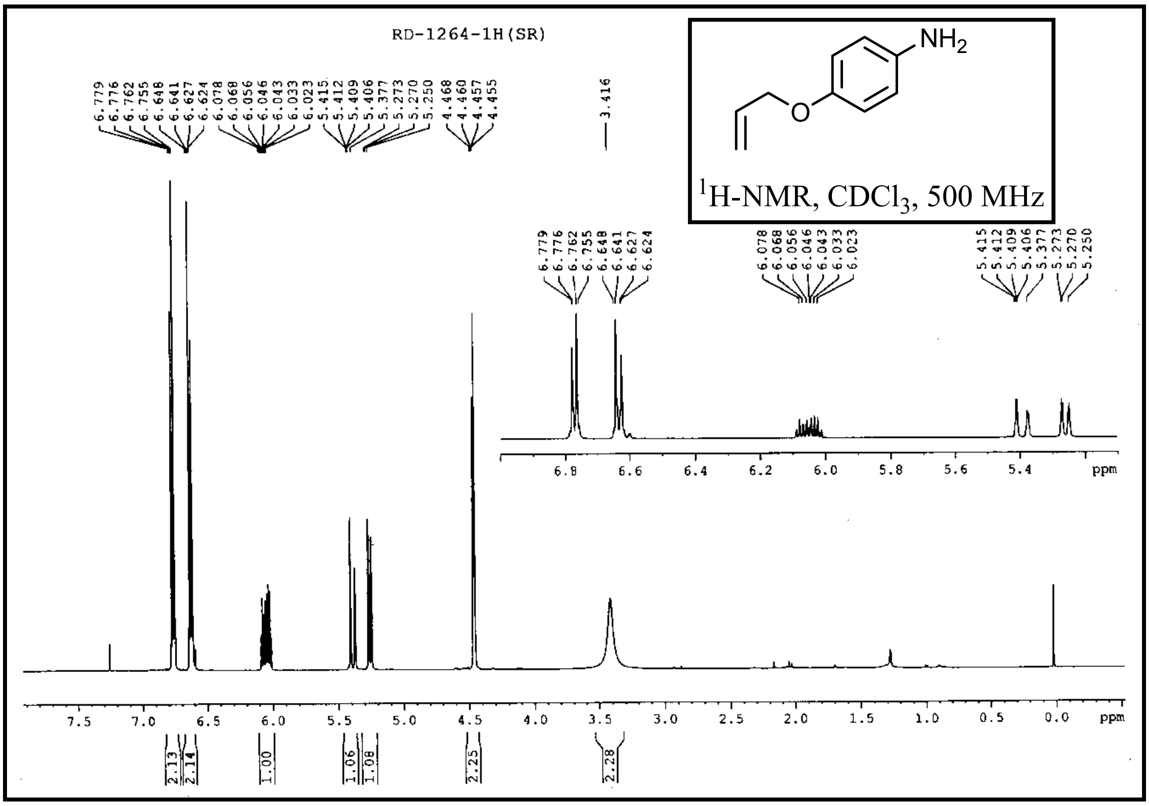


```
Current Data Parameters
NAME             SA57-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150505
Time                10 33
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CD3CN
NS                   208
DS                     4
SWH            18939.395 Hz
FIDRES          0.288992 Hz
AQ             1.7302004 sec
RG                9195.2
DW                26.400 usec
DE                  6.00 usec
TE                 299.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1                 0.00 dB
SFO1          75.4763276 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12                8.00 dB
PL13               12.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677406 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  8.17 cm
F1P              169.264 ppm
F1              12773.96 Hz
F2P               -5.583 ppm
F2                421.31 Hz
PPMCM            8.74234 ppm/cm
HZCM           659.76453 Hz/cm
```

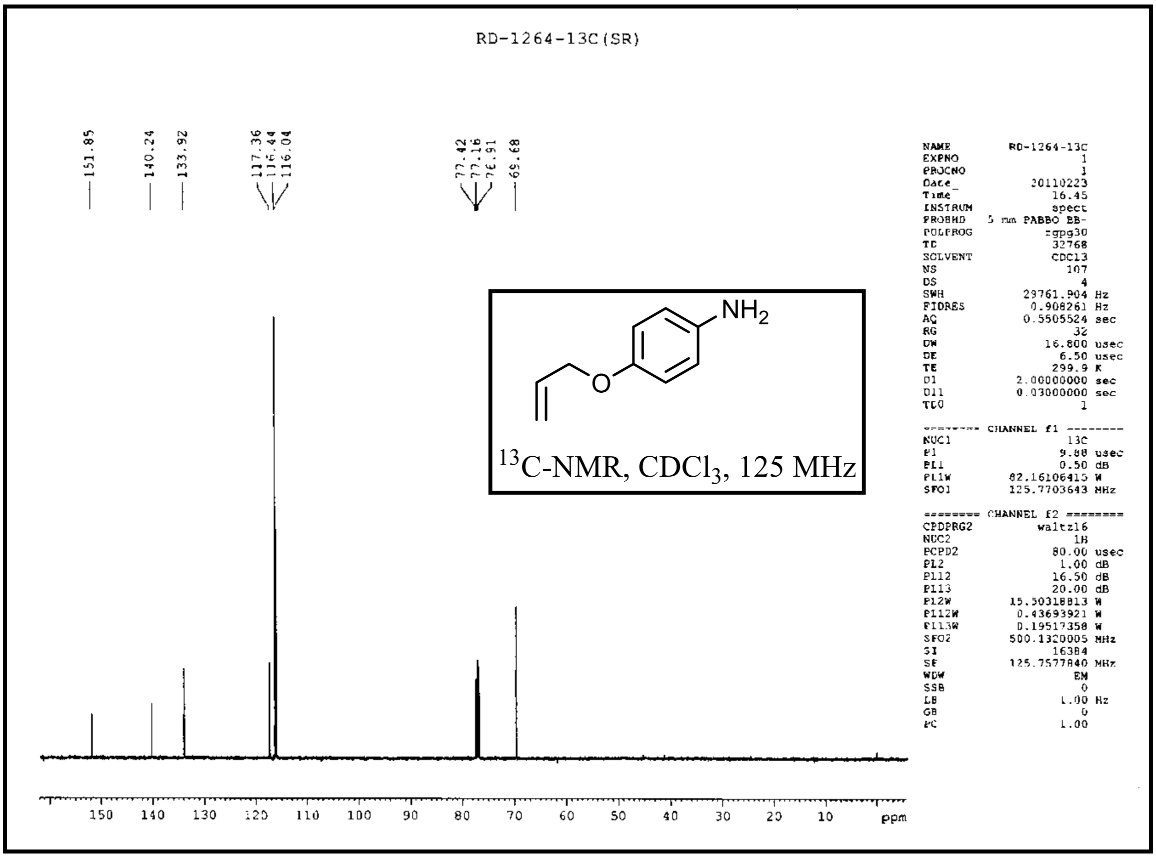




```
NAME          RD-1227-13C
EXPNO                   1
PROCNO                  1
Date_            20110329
Time                18.44
INSTRUM             spect
PROBHD     5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   254
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    32
DW                16.800 usec
DE                  6.50 usec
TE                 301.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577731 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

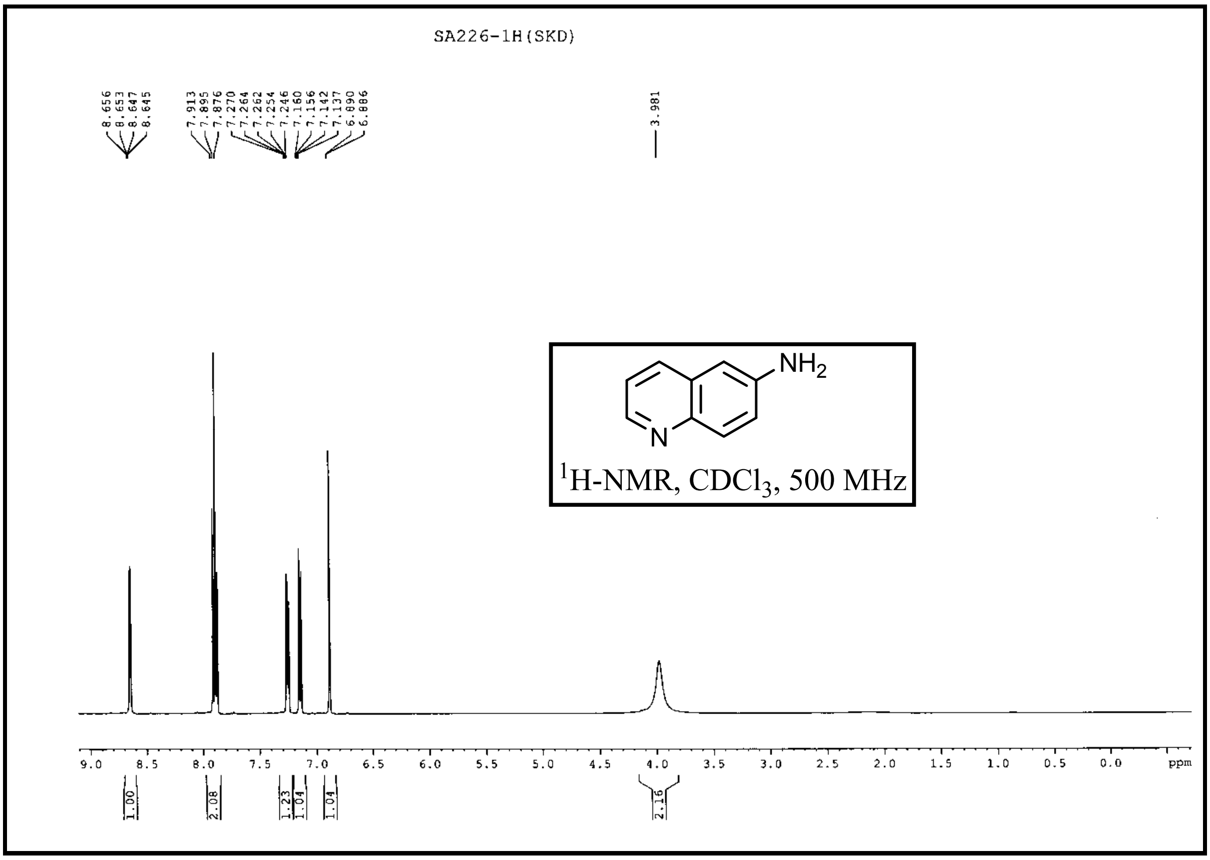


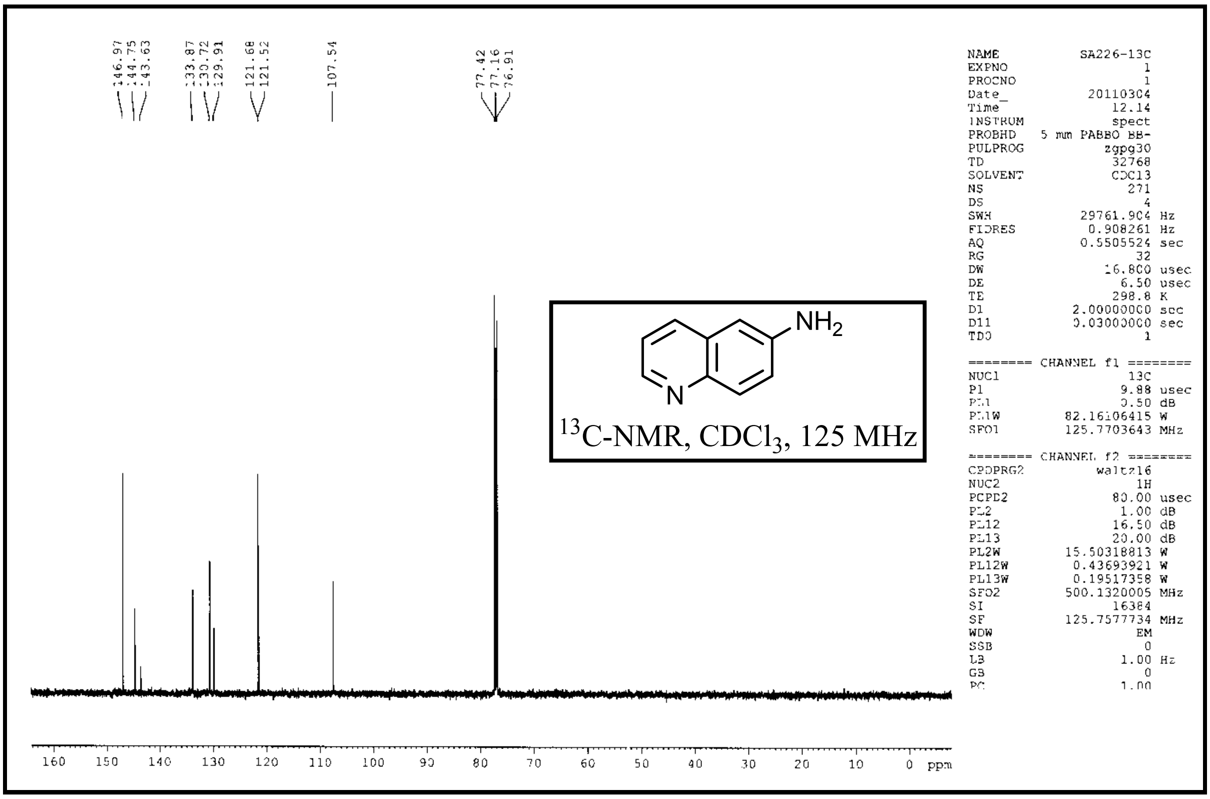
^{1}H-NMR, $CDCl_3$, 500 MHz



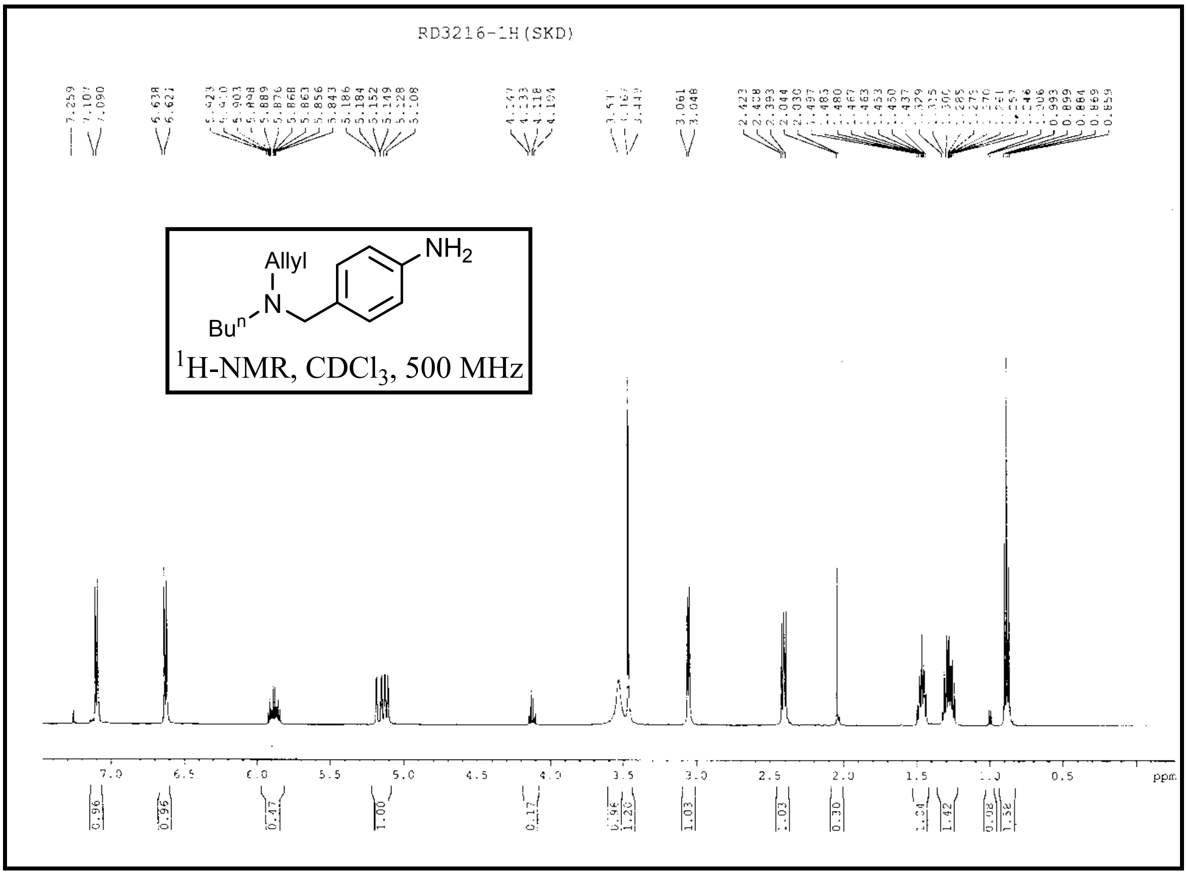
^{13}C-NMR, $CDCl_3$, 125 MHz

```
NAME           RD-1264-13C
EXPNO                    1
PROCNO                   1
Date_             20110223
Time                 16.45
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     107
DS                       4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                      32
DW                  16.800 usec
DE                    6.50 usec
TE                   299.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577840 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```

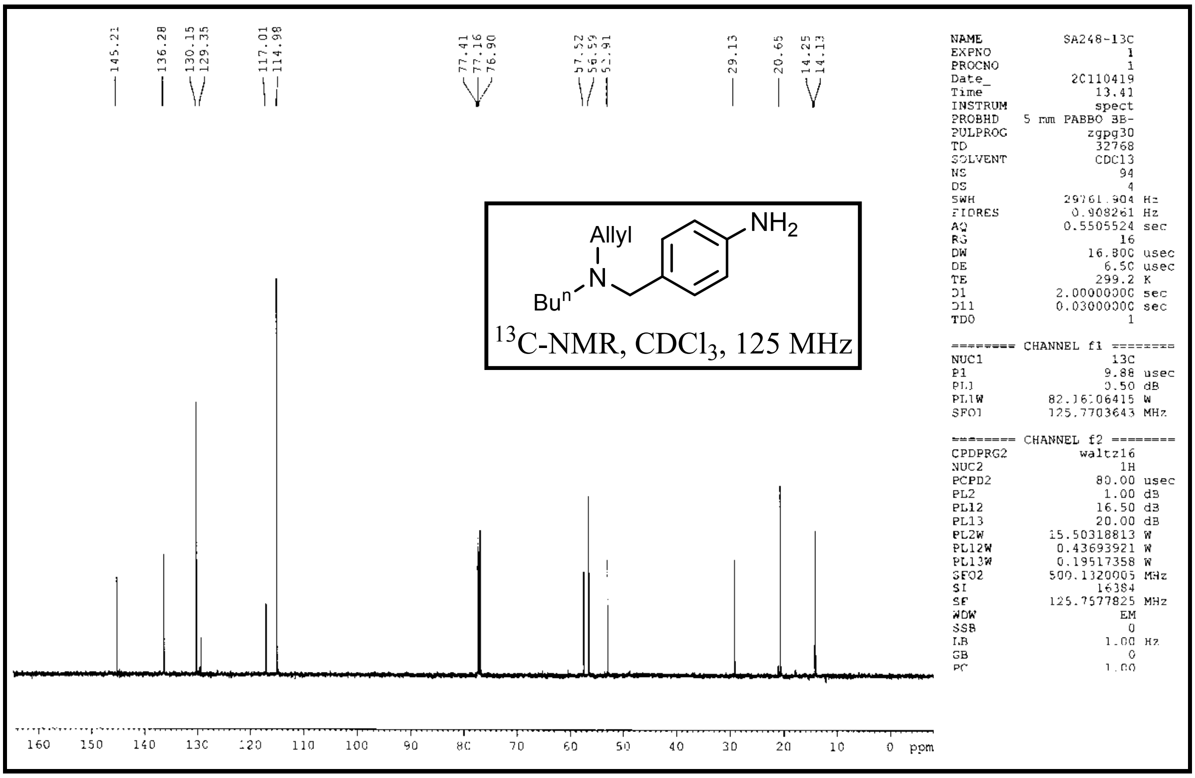




```
NAME              SA226-13C
EXPNO                     1
PROCNO                    1
Date_              20110304
Time                  12.14
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      271
DS                        4
SWH               29761.904 Hz
FIDRES             0.908261 Hz
AQ                0.5505524 sec
RG                       32
DW                   16.800 usec
DE                     6.50 usec
TE                    298.8 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.88 usec
PL1                    0.50 dB
PL1W            82.16106415 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  16.50 dB
PL13                  20.00 dB
PL2W            15.50318813 W
PL12W            0.43693921 W
PL13W            0.19517358 W
SFO2            500.1320005 MHz
SI                    16384
SF              125.7577734 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```

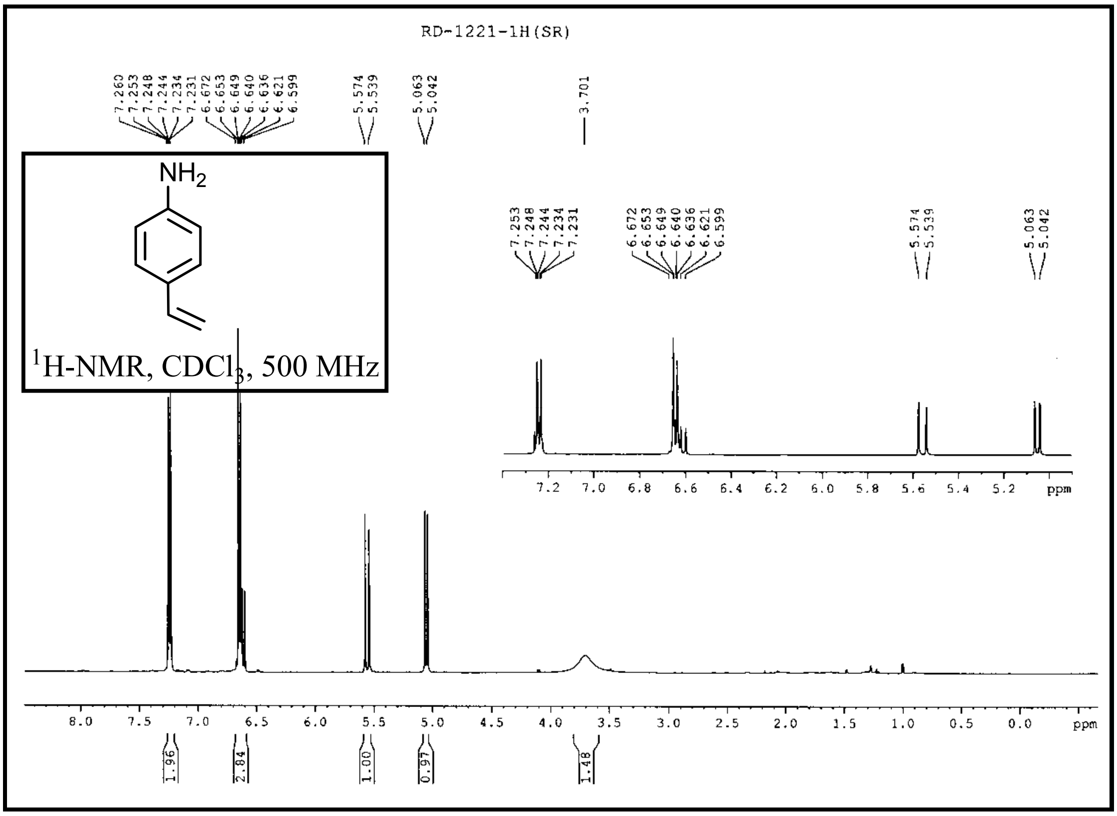


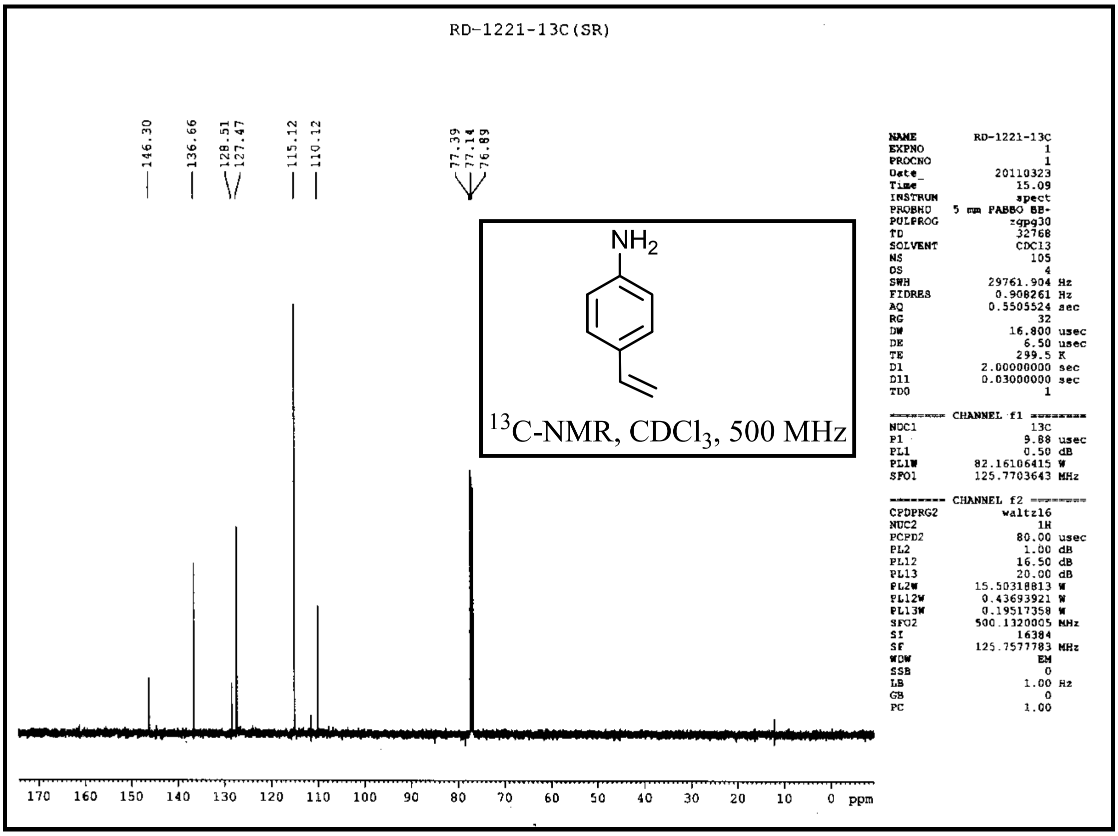
^{1}H-NMR, $CDCl_3$, 500 MHz



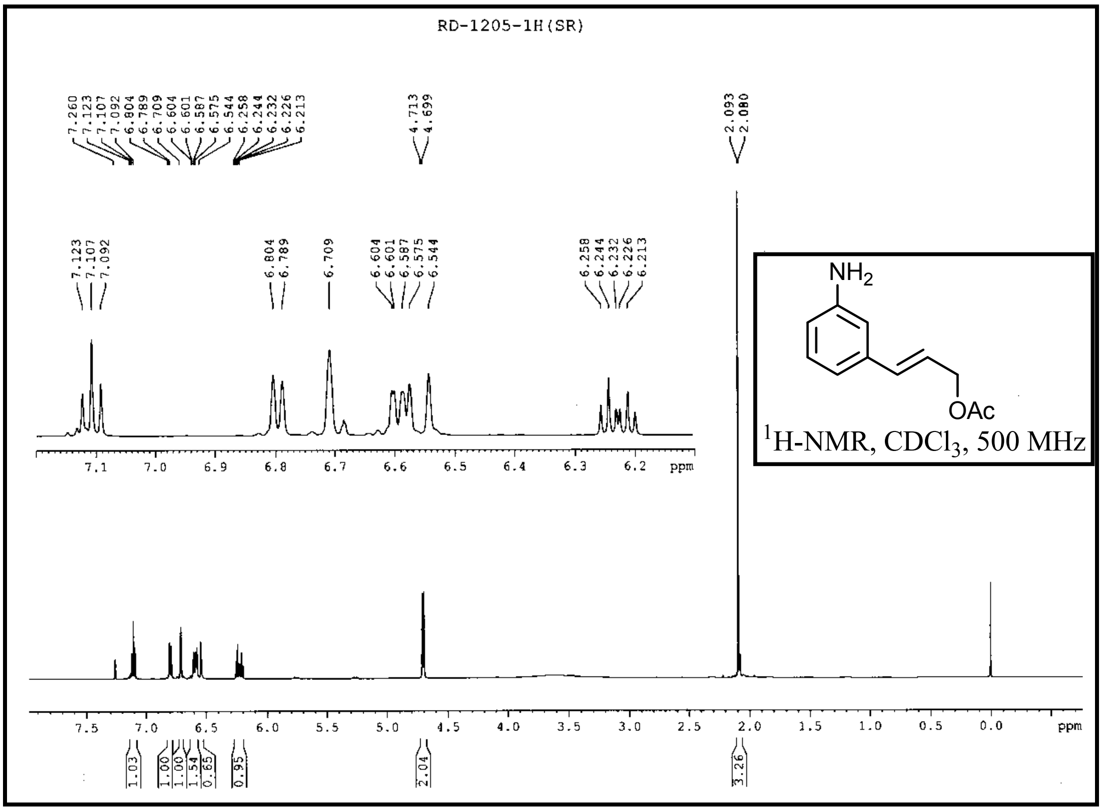
^{13}C-NMR, $CDCl_3$, 125 MHz

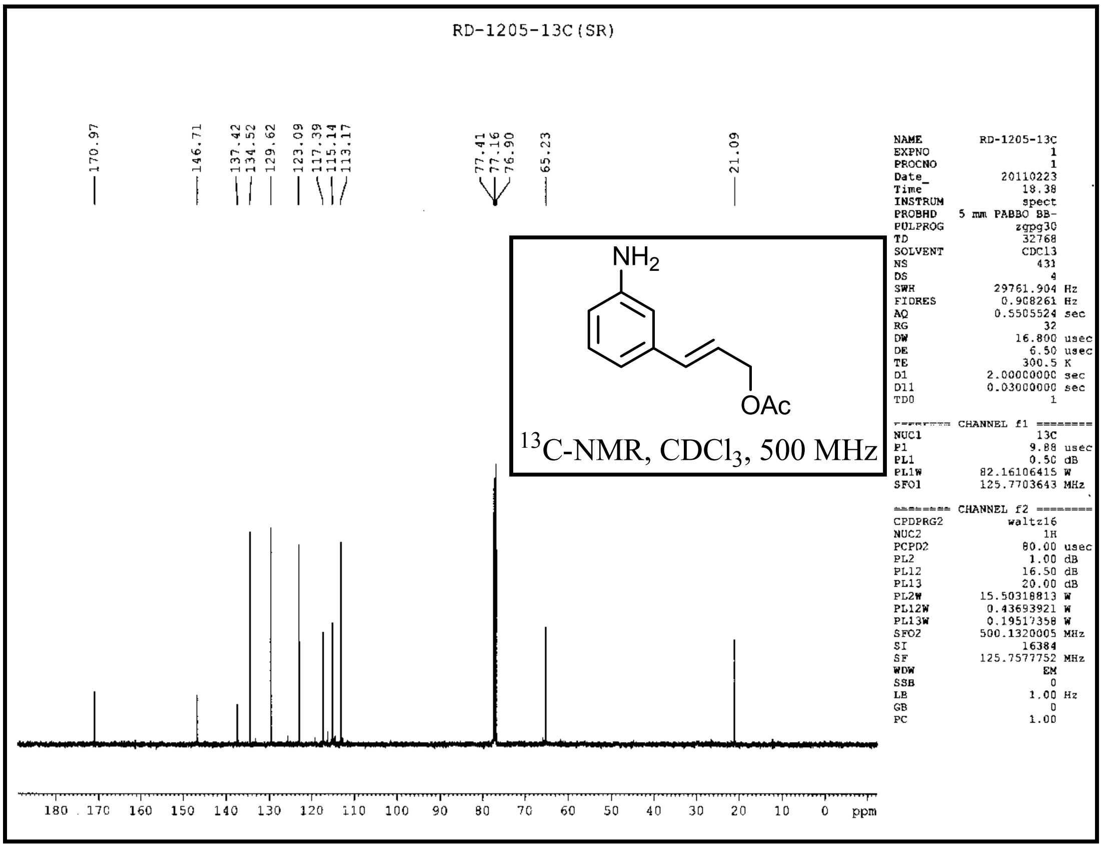
```
NAME             SA248-13C
EXPNO                    1
PROCNO                   1
Date_             20110419
Time                 13.41
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      94
DS                       4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                      16
DW                  16.800 usec
DE                    6.50 usec
TE                   299.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577825 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```



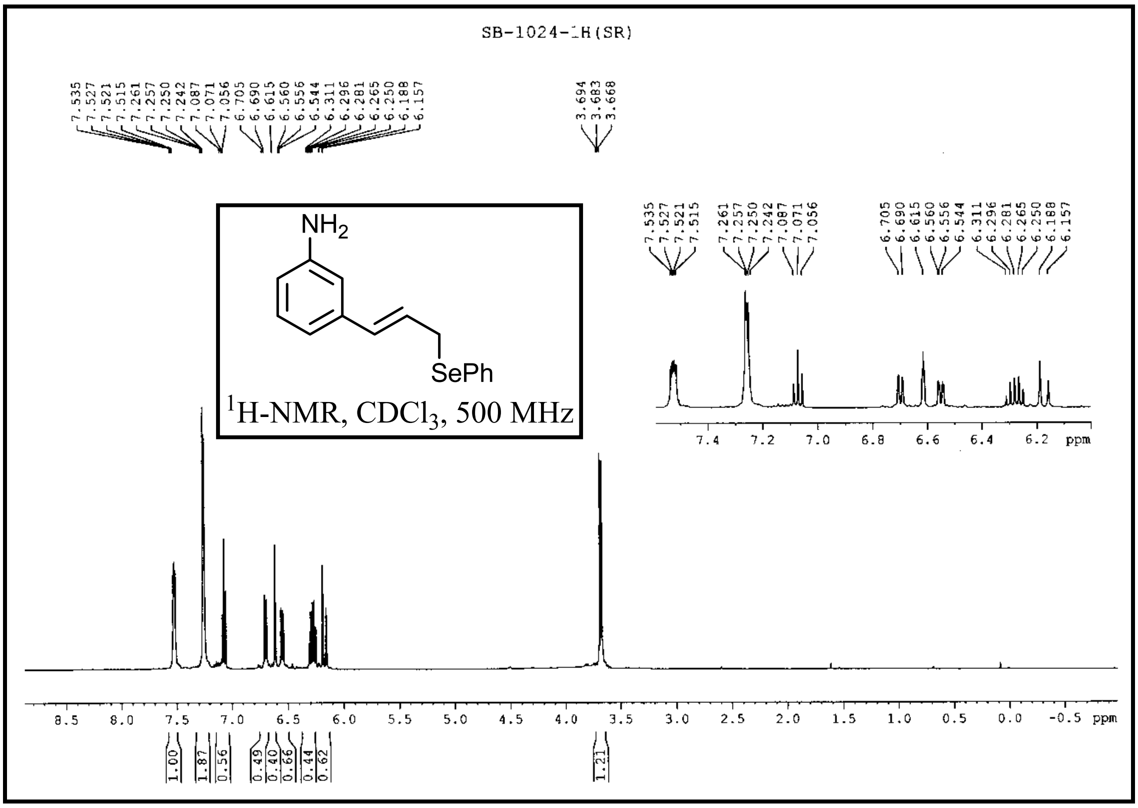


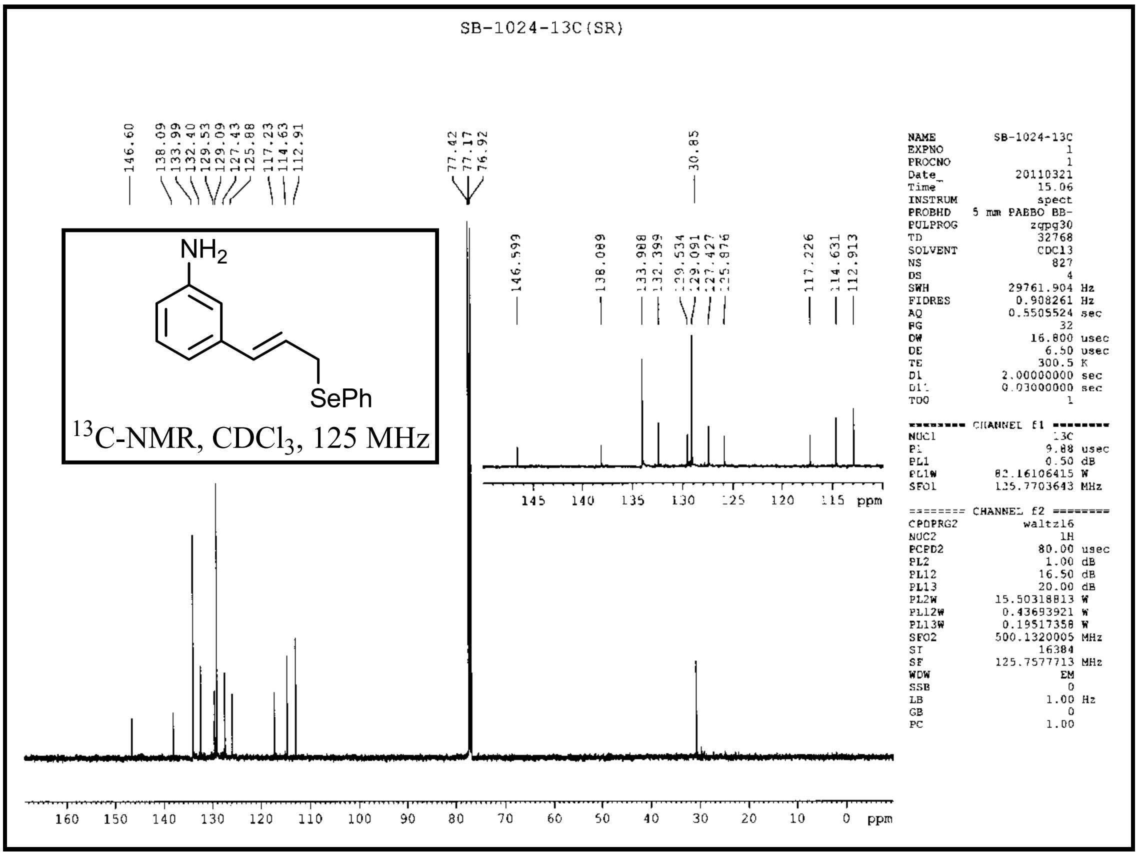
```
NAME           RD-1221-13C
EXPNO                    1
PROCNO                   1
Date_             20110323
Time                 15.09
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     105
DS                       4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              0.5505524 sec
RG                      32
DW                  16.800 usec
DE                    6.50 usec
TE                   299.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577783 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```



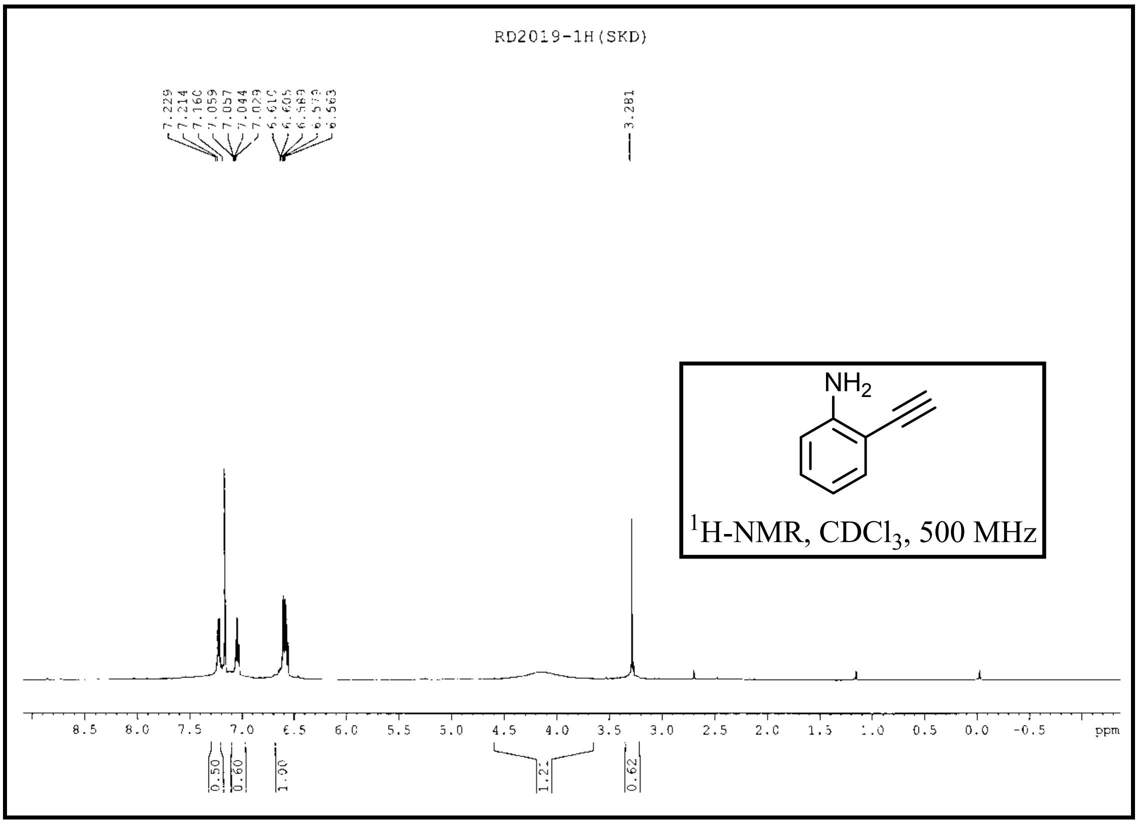


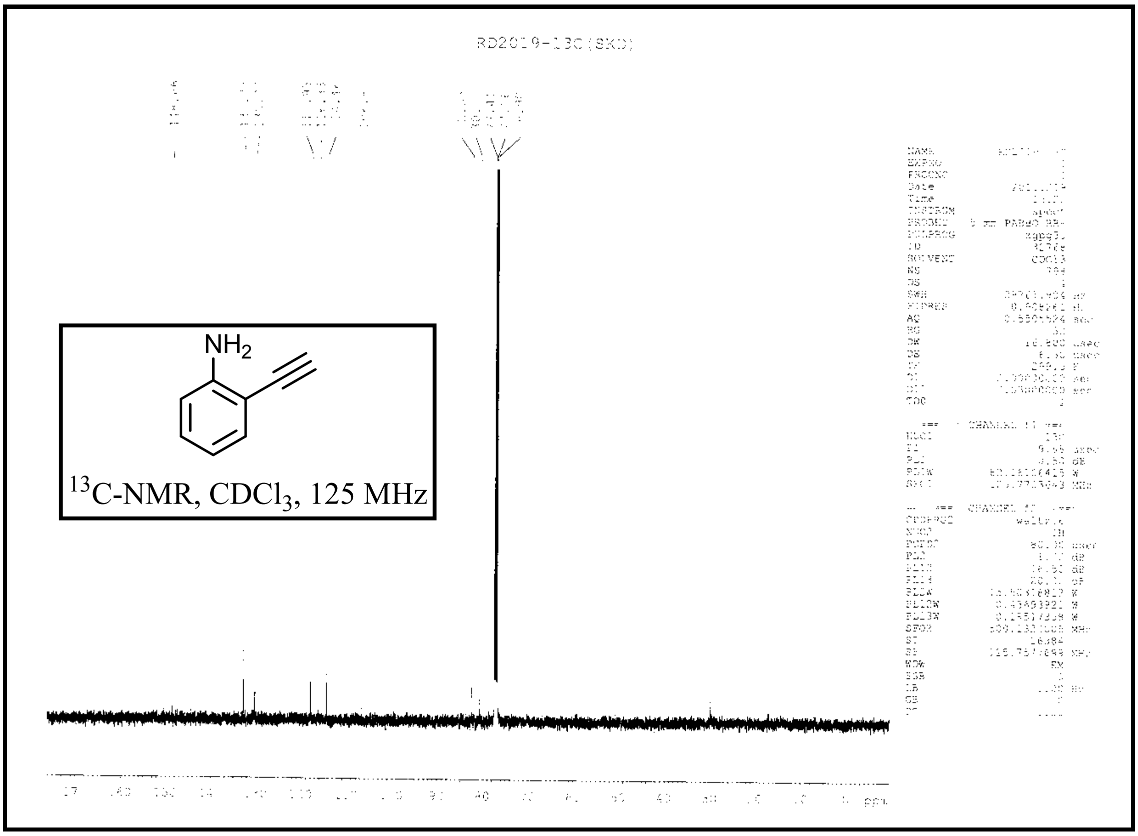
```
NAME         RD-1205-13C
EXPNO                  1
PROCNO                 1
Date_           20110223
Time               18.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   431
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    32
DW                16.800 usec
DE                  6.50 usec
TE                 300.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577752 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

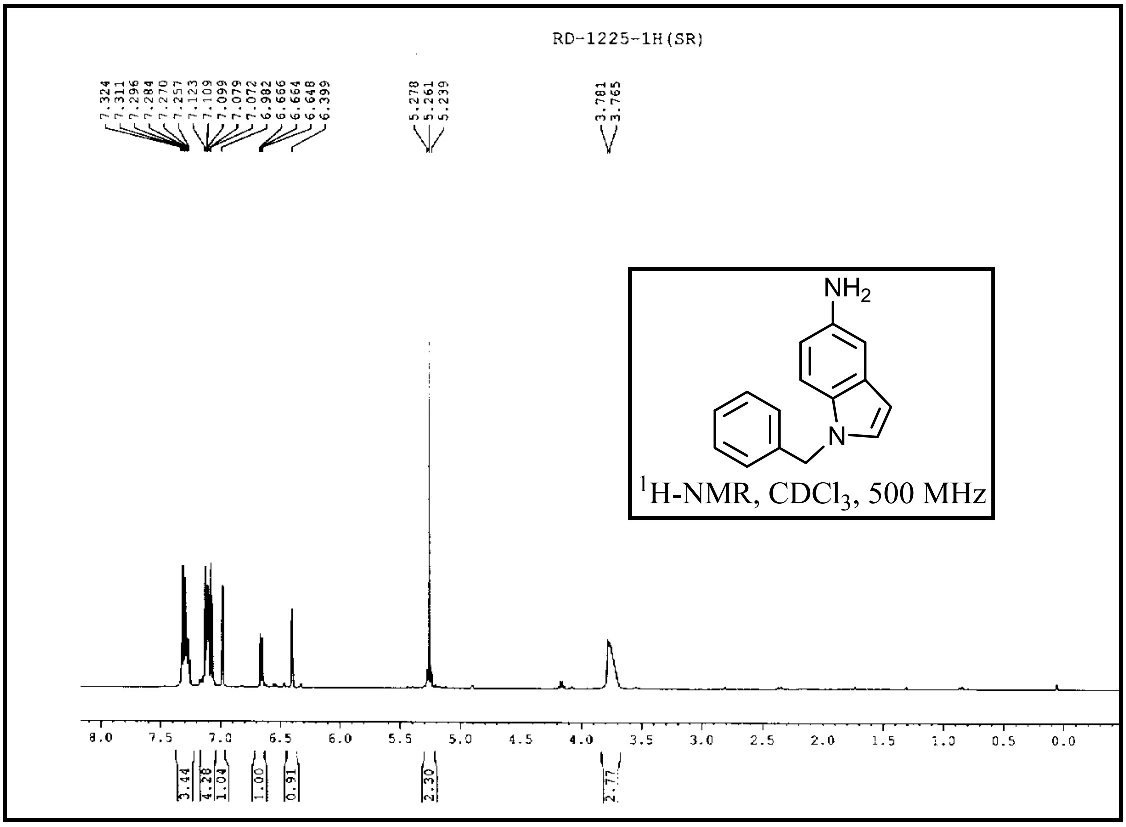


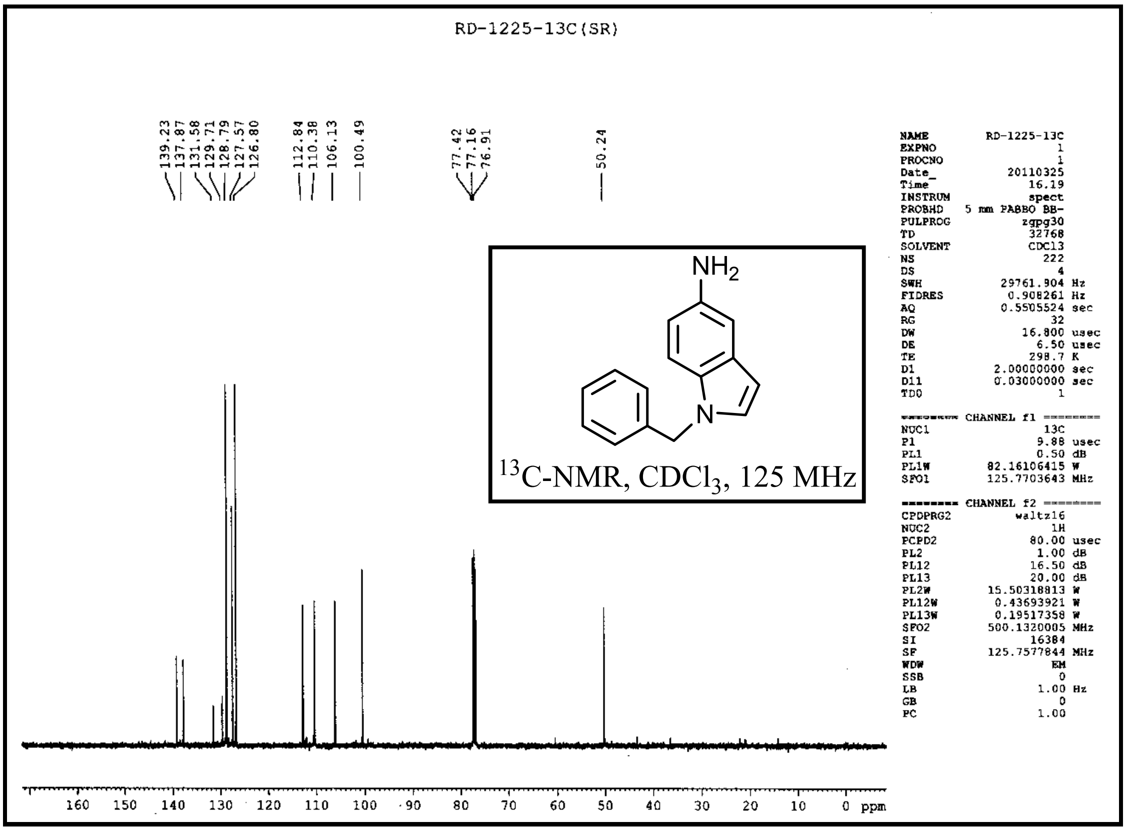


```
NAME           SB-1024-13C
EXPNO                    1
PROCNO                   1
Date_             20110321
Time                 15.06
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     827
DS                       4
SWH              29761.904 Hz
FIDRES            0.908261 Hz
AQ               0.5505524 sec
RG                      32
DW                  16.800 usec
DE                    6.50 usec
TE                   300.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577713 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```

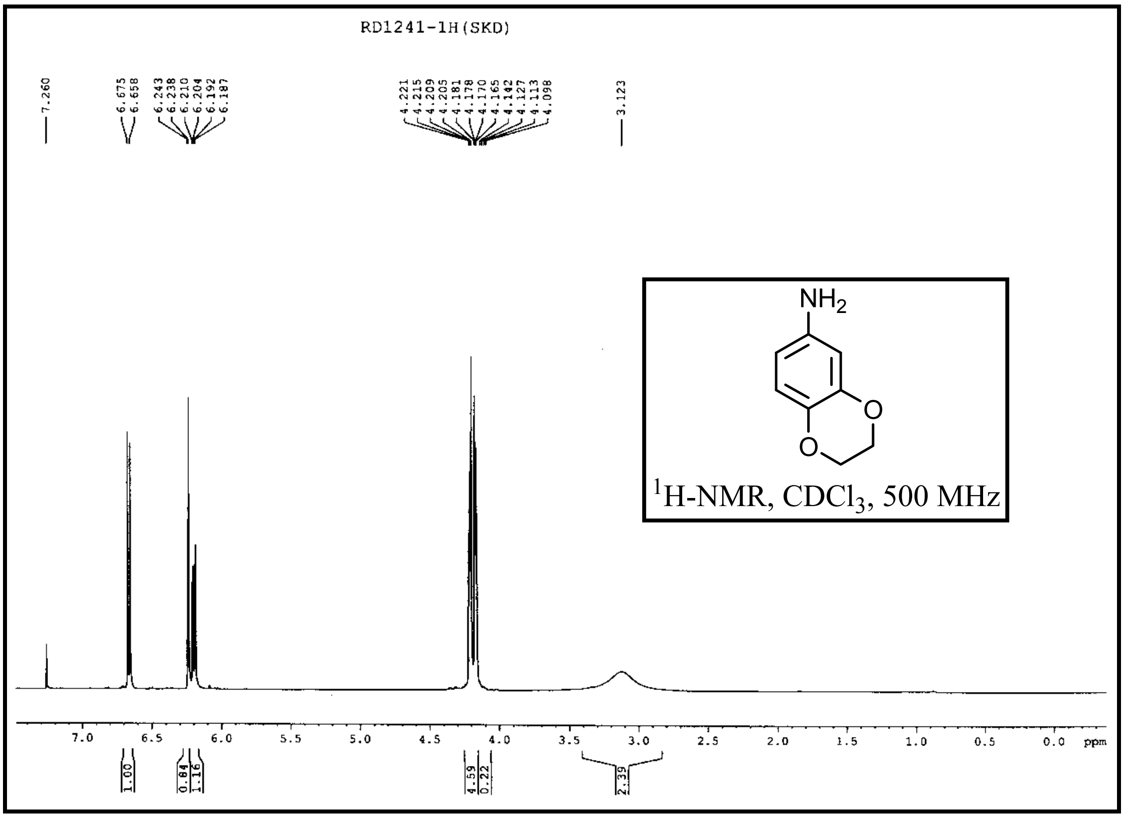

RD2019-1H(SKD)
7.229
7.214
7.160
7.059
7.057
7.044
7.029
6.610
6.605
6.589
6.579
6.563
3.281
NH2
1H-NMR, CDCl3, 500 MHz
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
0.50
0.60
1.00
1.21
0.62


RD2019-13C(SKD)
NH2
13C-NMR, CDCl3, 125 MHz
ppm

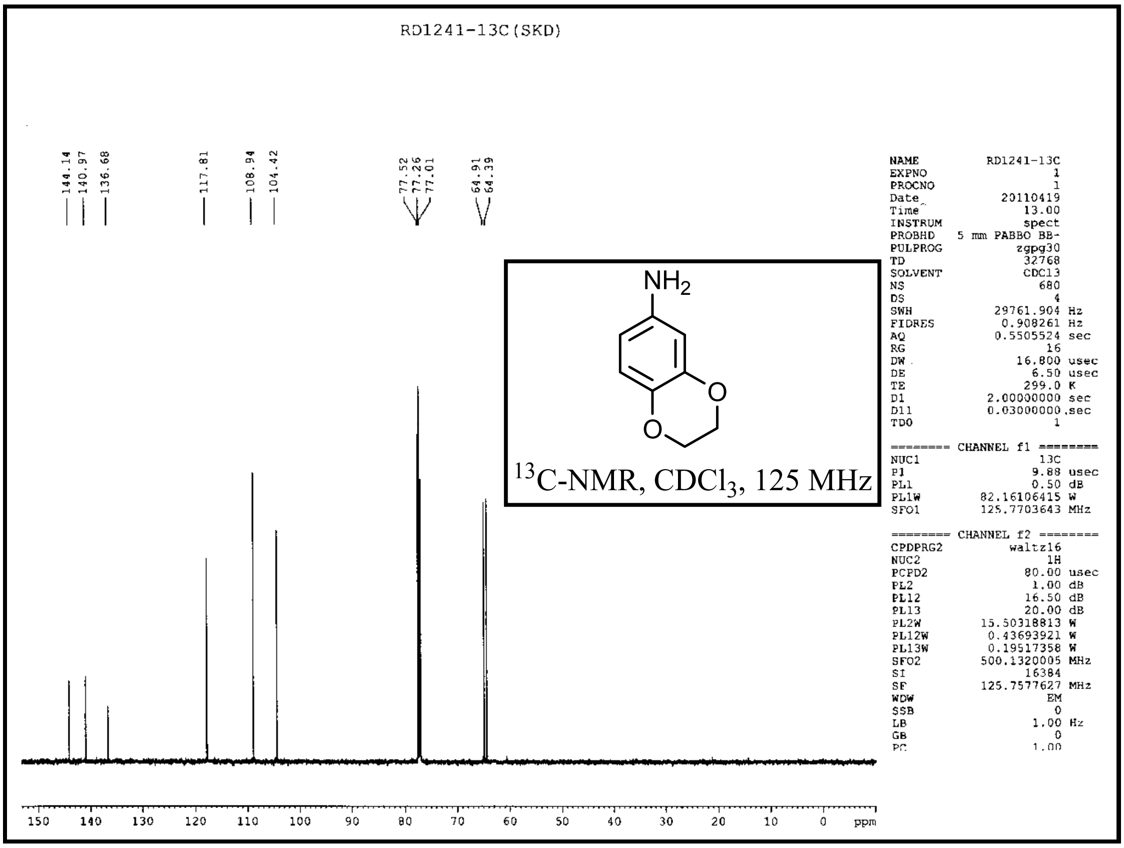




```
NAME          RD-1225-13C
EXPNO                   1
PROCNO                  1
Date_            20110325
Time                16.19
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   222
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    32
DW                16.800 usec
DE                  6.50 usec
TE                 298.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577844 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

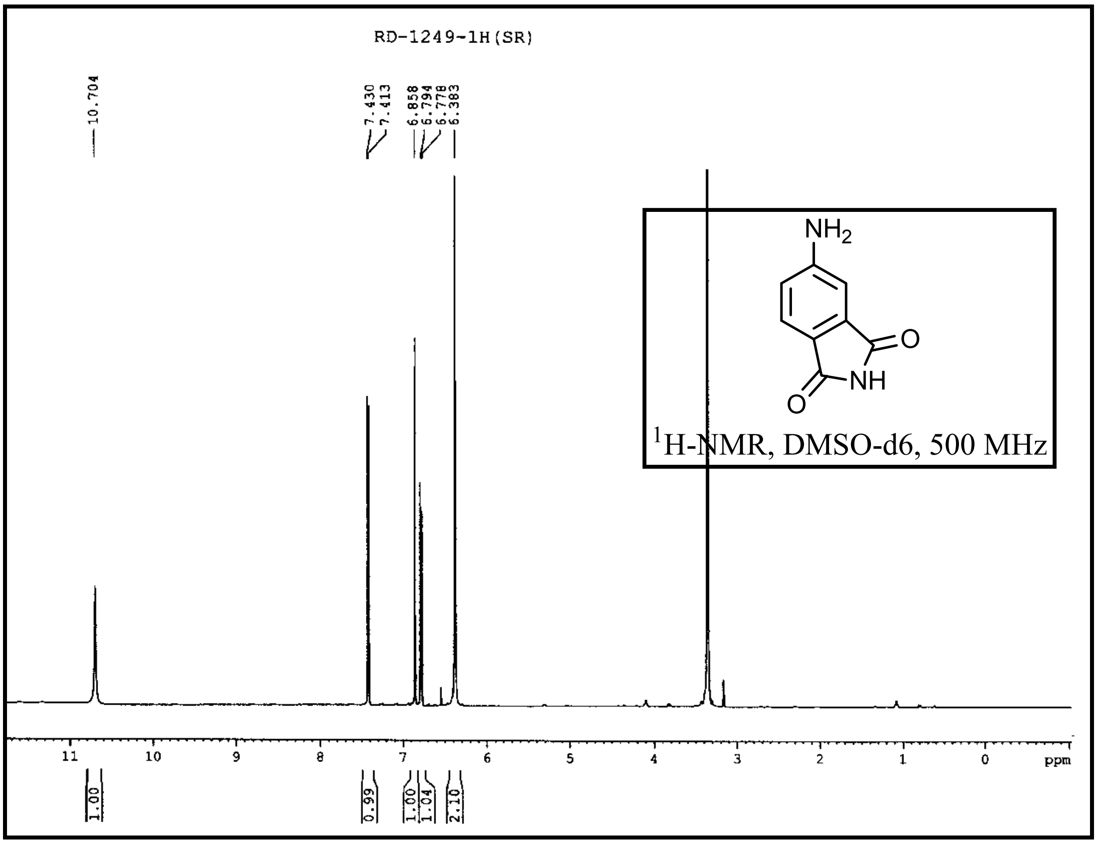


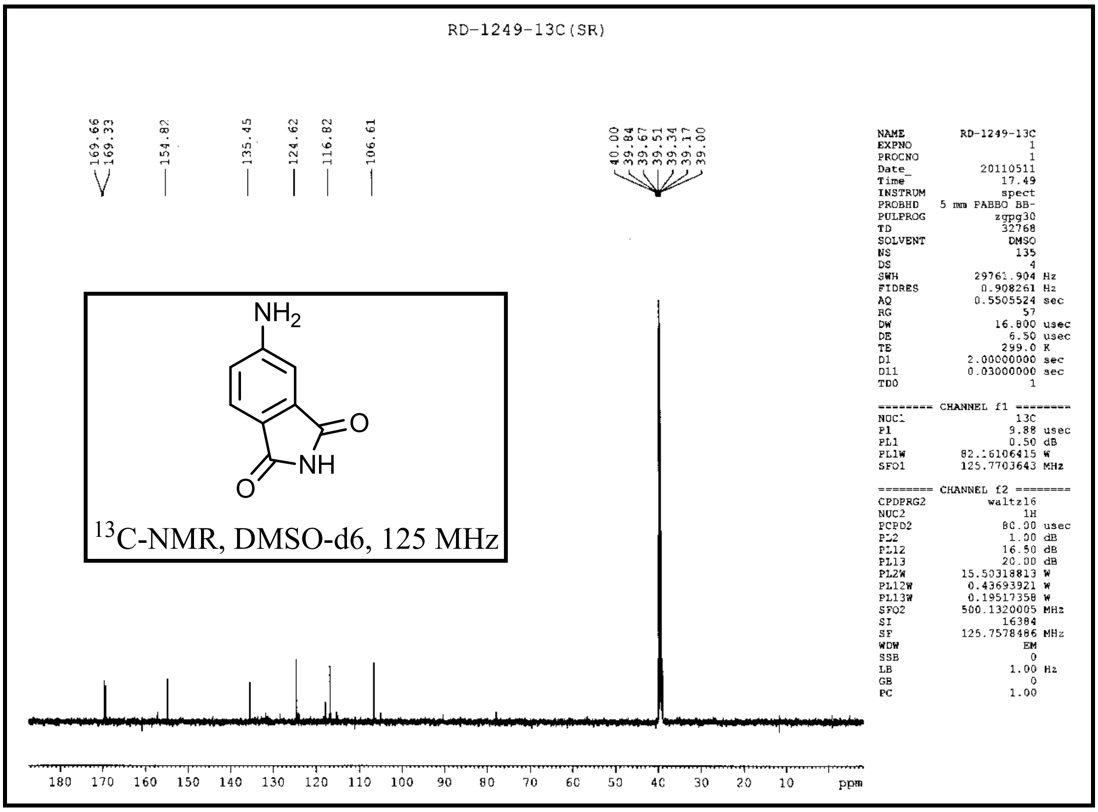
^{1}H-NMR, $CDCl_3$, 500 MHz



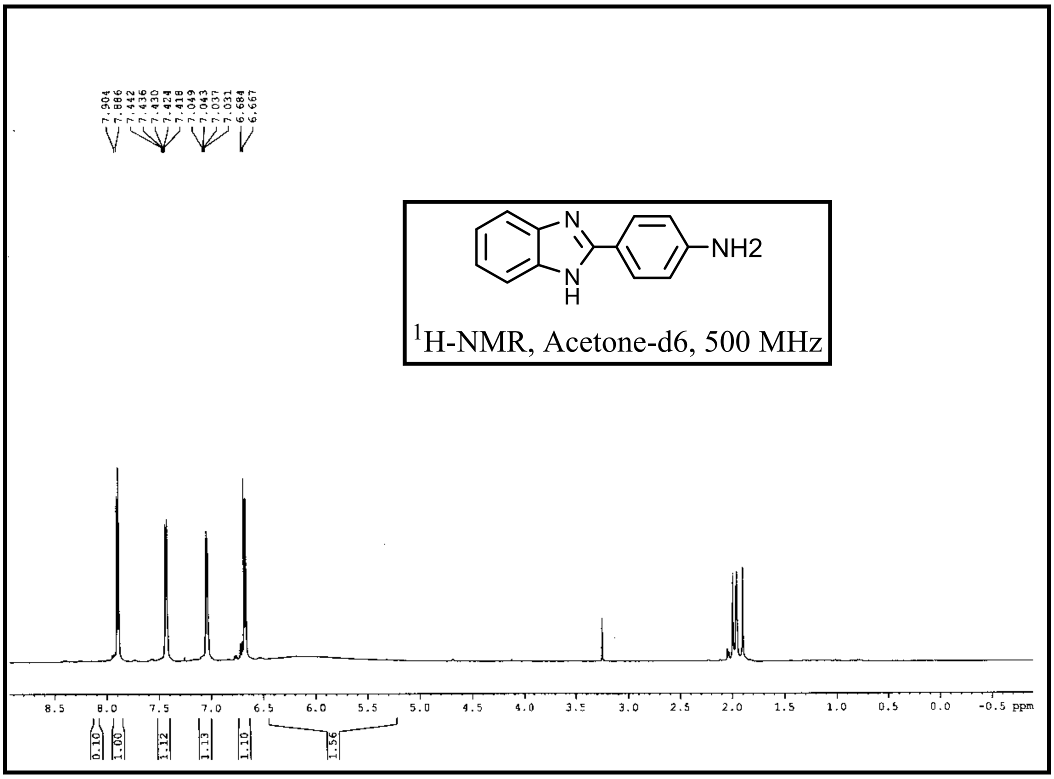
^{13}C-NMR, $CDCl_3$, 125 MHz

```
NAME           RD1241-13C
EXPNO                   1
PROCNO                  1
Date_            20110419
Time                13.00
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT            CDC13
NS                   680
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             0.5505524 sec
RG                    16
DW                16.800 usec
DE                  6.50 usec
TE                 299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.88 usec
PL1                 0.50 dB
PL1W         82.16106415 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.50 dB
PL13               20.00 dB
PL2W         15.50318813 W
PL12W         0.43693921 W
PL13W         0.19517358 W
SFO2         500.1320005 MHz
SI                 16384
SF           125.7577627 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```

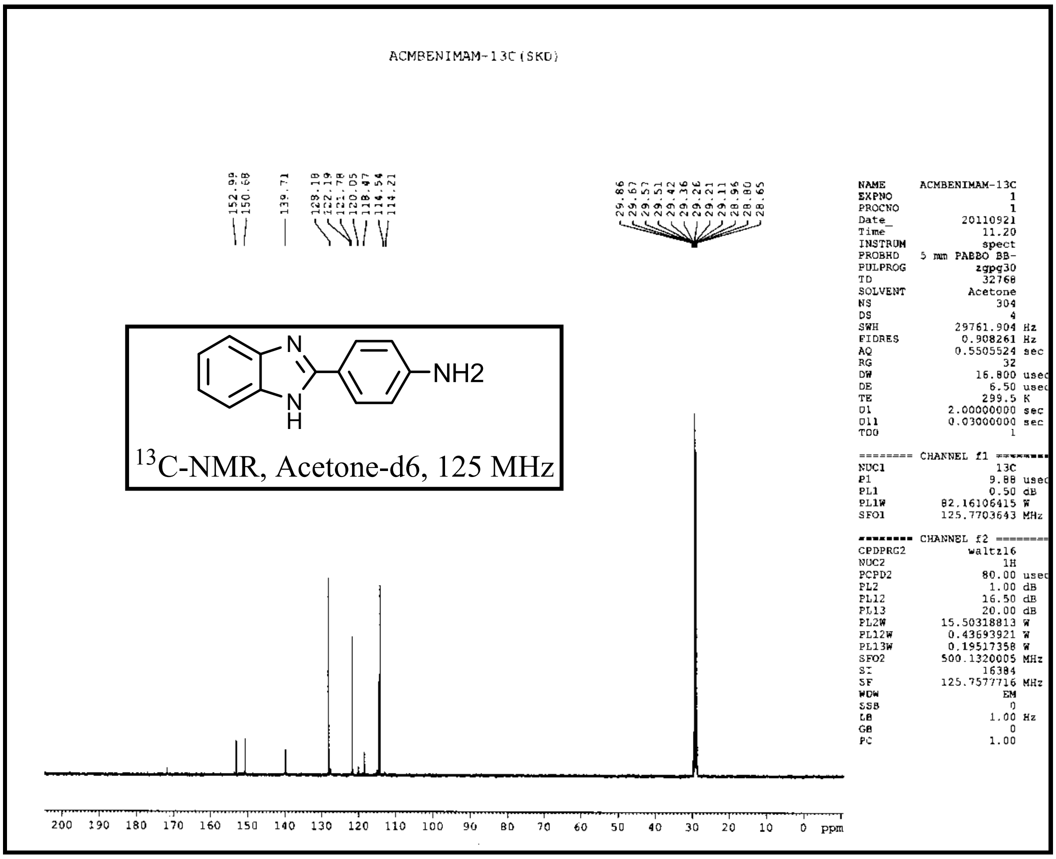




```
NAME          RD-1249-13C
EXPNO                   1
PROCNO                  1
Date_            20110511
Time                17.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  32768
SOLVENT              DMSO
NS                    135
DS                      4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              0.5505524 sec
RG                     57
DW                 16.800 usec
DE                   6.50 usec
TE                  299.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.88 usec
PL1                  0.50 dB
PL1W          82.16106415 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                16.50 dB
PL13                20.00 dB
PL2W          15.50318813 W
PL12W          0.43693921 W
PL13W          0.19517358 W
SFO2          500.1320005 MHz
SI                  16384
SF            125.7578486 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```



^{1}H-NMR, Acetone-d6, 500 MHz



^{13}C-NMR, Acetone-d6, 125 MHz

```
NAME        ACMBENIMAM-13C
EXPNO                    1
PROCNO                   1
Date_             20110921
Time                 11.20
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG            zgpg30
TD                   32768
SOLVENT            Acetone
NS                     304
DS                       4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              0.5505524 sec
RG                      32
DW                  16.800 usec
DE                    6.50 usec
TE                   299.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.88 usec
PL1                   0.50 dB
PL1W           82.16106415 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 16.50 dB
PL13                 20.00 dB
PL2W           15.50318813 W
PL12W           0.43693921 W
PL13W           0.19517358 W
SFO2           500.1320005 MHz
SI                   16384
SF             125.7577716 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```