Supporting Information

Palladium Nanoparticle Catalyzed Hiyama Coupling Reaction of Benzyl Halides

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1. General Information

Unless otherwise stated, all starting materials were obtained from commercial suppliers and used as received. Organic solvents were dried and distilled as described elsewhere.[1]

All moisture and air sensitive reactions were carried out in a oven-dried flask under argon atmosphere. Column chromatography was performed with silica gel 230~400 mesh. All ^{1}H NMR ^{13}C NMR spectra were recorded on a Bruker- Avance DPX300 and Bruker- Avance III DPX500 for a $CDCl_3$ solution and reported in ppm (δ). Transmission electron microscopy (TEM): TEM pictures were taken on a JEM-2011 (JEOL) microscope.

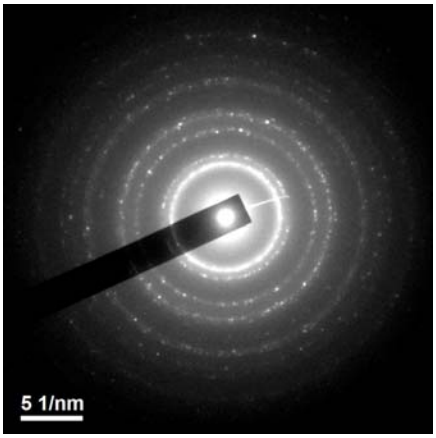
2. Experimental Procedures:

General method of Pd nanoparticle catalyzed Hiyama coupling reaction: Weighed amount of K_2PdCl_4 (13 mg, 0.04 mmol) and PEG 600 (700 mg, 1.16 mol) was heated at 70 °C for 15 min to form the Pd nanoparticle. Then it was cooled and to this suspension of colloidal Pd, aryltrialkoxysilane (2 mmol) and benzyl halide (1 mmol) and tetrabutylammonium fluoride TBAF [1.5 ml 1(M)solution in THF] were added and stirred at 70 °C for the specified time. Then the reaction mixture was allowed to cool at room temperature and THF was removed and extracted with ether and the organic layers were dried over anhydrous Na_2SO_4 and concentrated in vacuum. The residue thus obtained was purified by flash column chromatography on silica gel (230~400 mesh) with either petroleum ether or a mixture of ethyl acetate (2-10%) or as eluent.

Preparation of 4-(triethoxysilyl)toluene:[2] A solution of *n*-BuLi (1.6 M in hexane, 3.1 mL, 5 mmol) was added dropwise (1 drop per second) with stirring to a solution of 4-bromotoluene (855 mg, 0.62 mL, 5 mmol), in Et_2O or THF (7 mL) at room temperature. After 1 h, the solution was cooled to -78 °C and transfered *via* cannula to a stirred solution of tetraethyl orthosilicate (1.56 g, 1.68 mL, 7.50 mmol) in Et_2O or THF (7.0 mL) at -78 °C. After 1 h, the reaction was quenched with H_2O (5 drops) at -78 °C and allowed to warm slowly to room temperature. The crude reaction mixture was then extracted with (3 × 50 mL) Et_2O. The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated in vacuum. Bulb-to-bulb distillation (125 °C, 0.5 Torr) afforded 1.08 g (81%) of 4-(triethoxysilyl)toluene as a colorless oil.

Preparation of Pd Nanoparticles:

Pd Nanoparticle prepared by stirring 14 mg K_2PdCl_4 with 700 mg of PEG-600 for 15 min at 70 ^oC.



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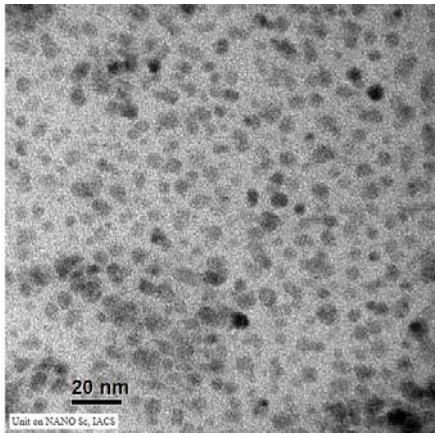
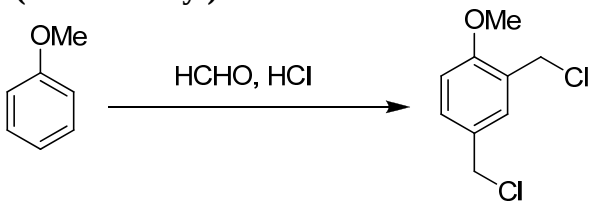


b

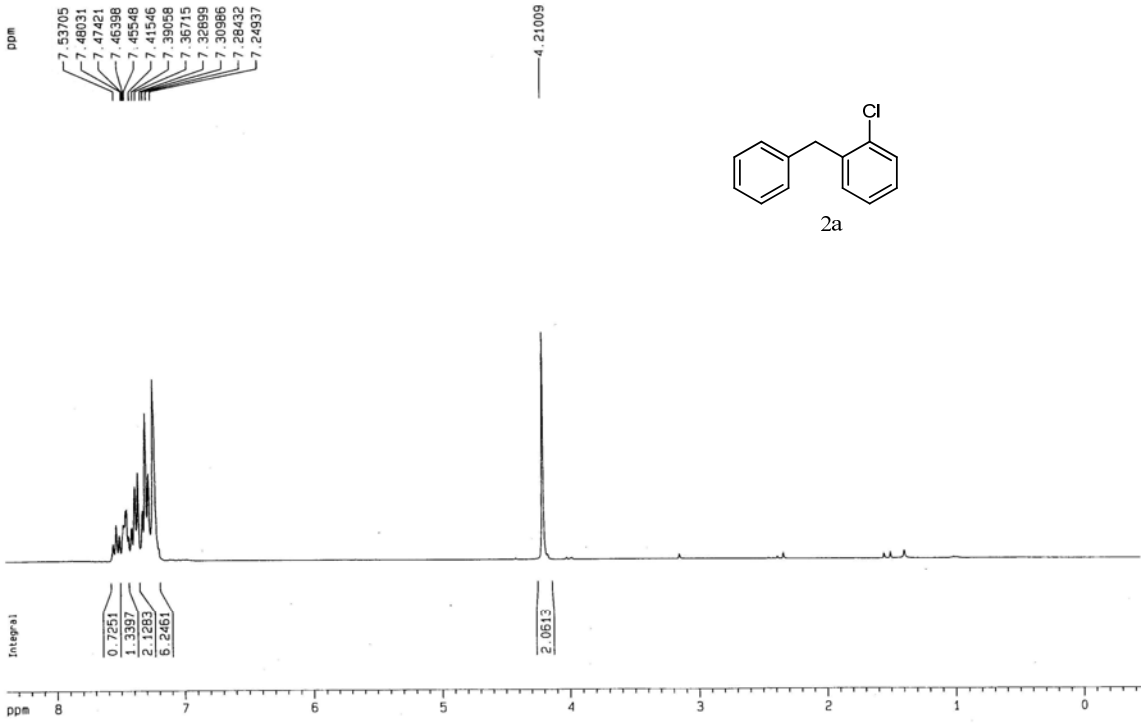
Figure 1. (a) Electron diffraction pattern of palladium nanoparticles. (b) TEM image of the palladium nanoparticles.

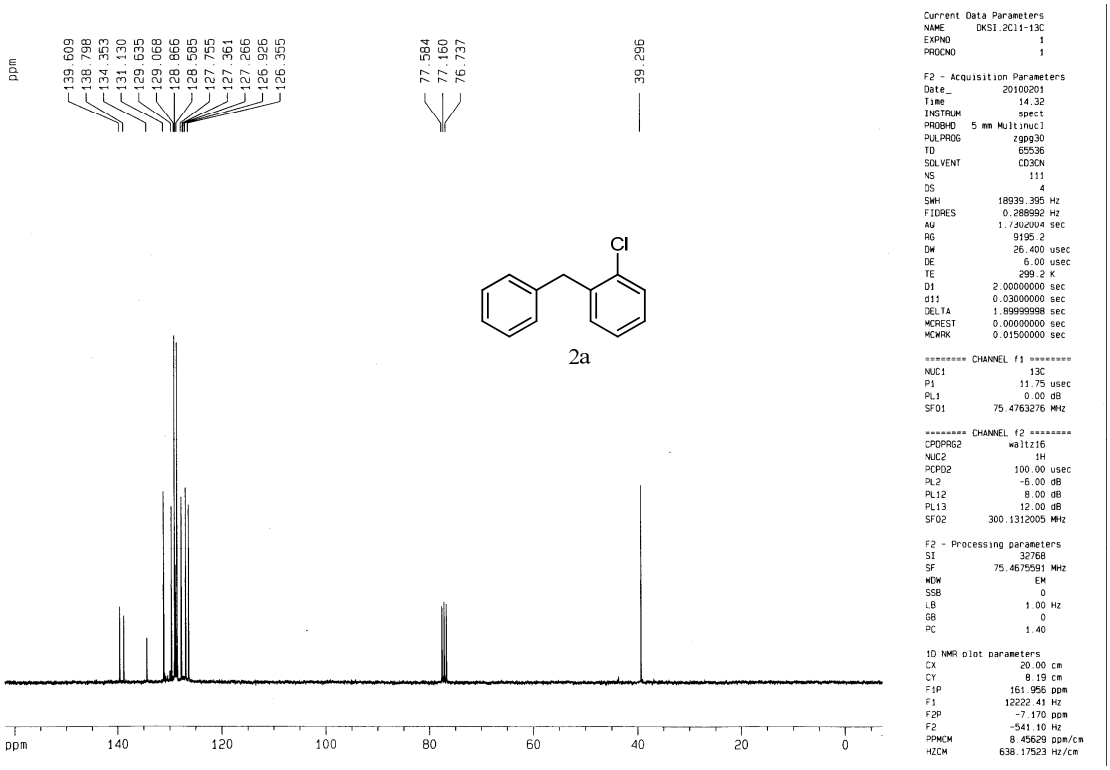
Preparation of 2, 6-di(chloromethyl) anisole:

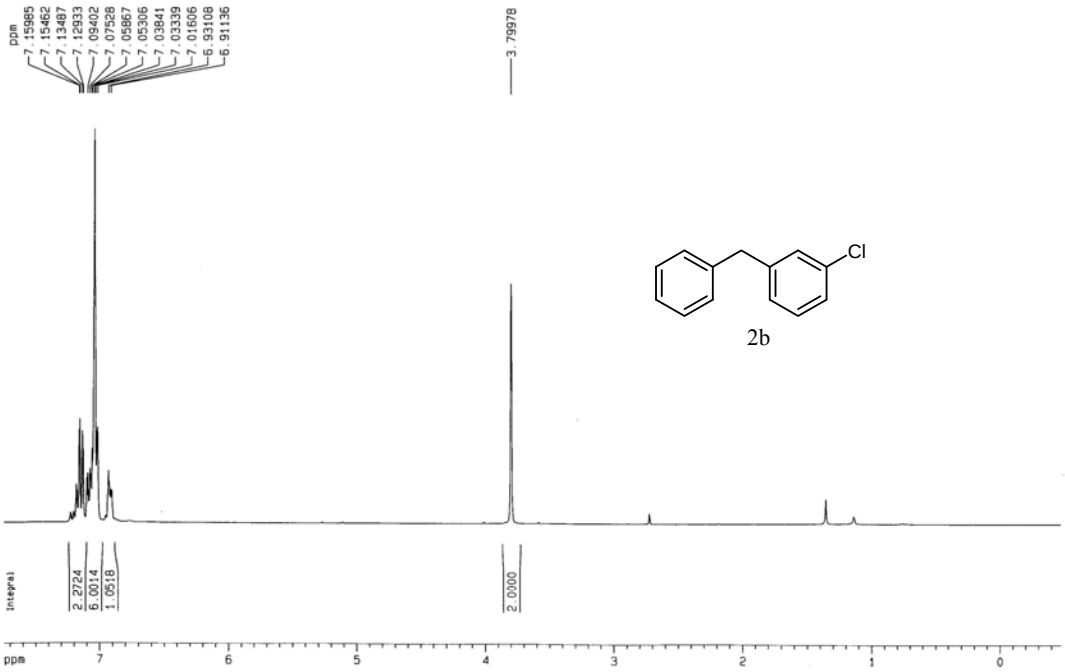


Gaseus HCl was introduced into a mixture of 50 mmol of formaldehyde of 30% strength and 120 mmol of concentrated hydrochloric acid until the mixture saturated therewith.17 mmol (1.84 g) of anisole was then added gradually with stirring. The temperature was kept at 30 °C and the introduction of gas was continued with stirring for about 10 h. Crystalline solids were separated which were filtered with suction and washed with water and dried in air. Then it was recrystallized from hot petroleum ether.(2.70 g, 80%)

3. Figures reproducing ^{1}H and ^{13}C NMR spectra



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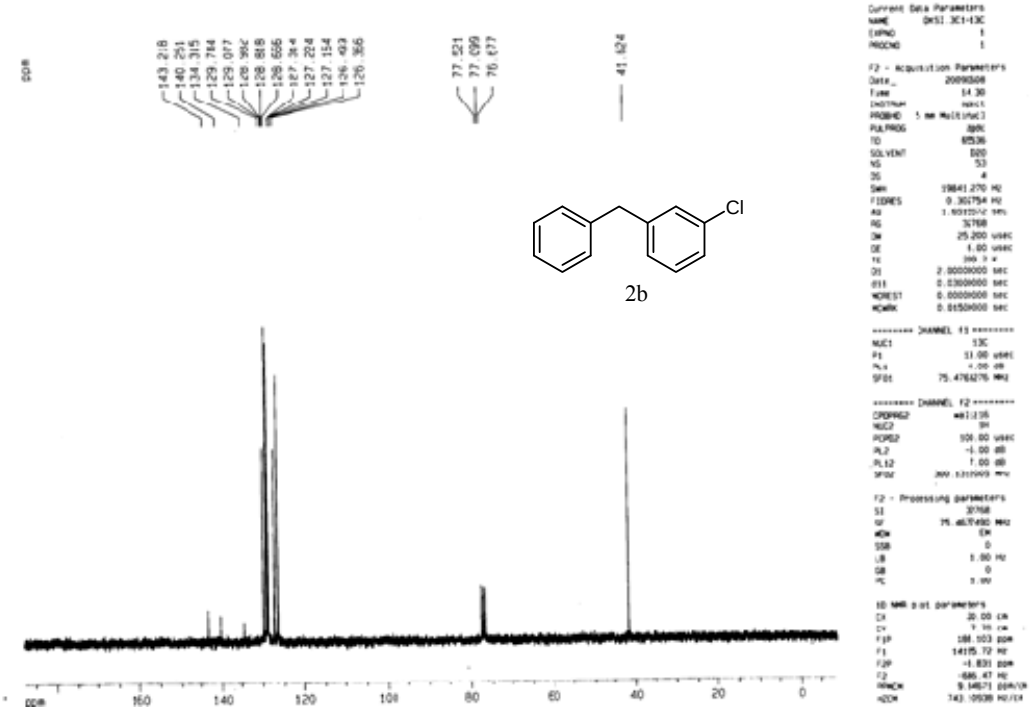
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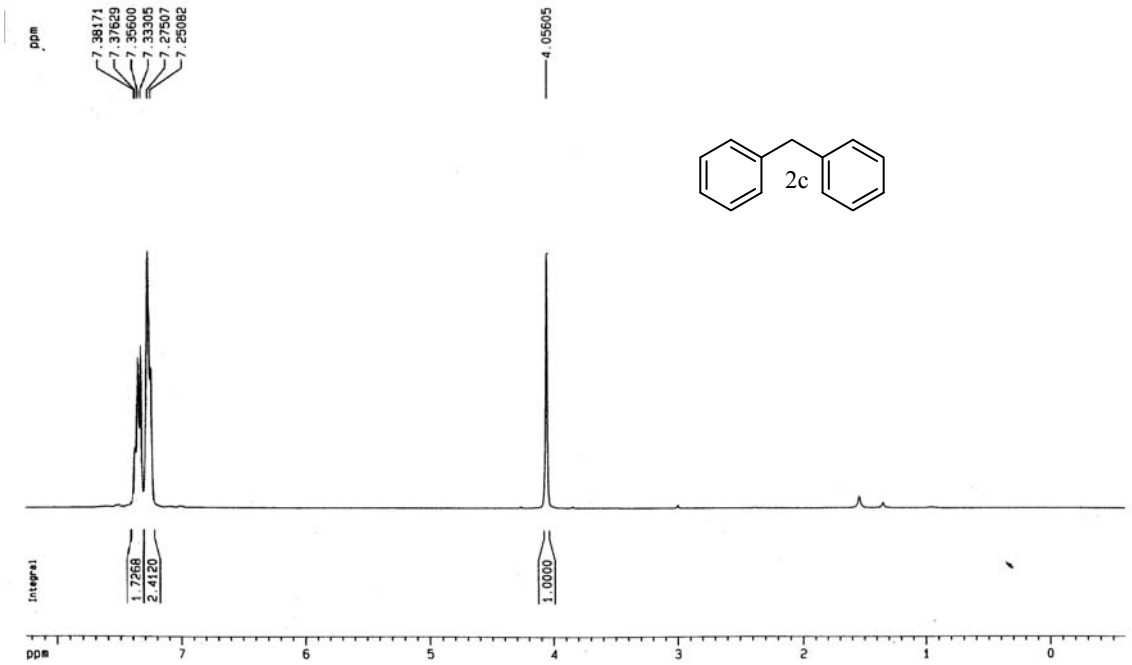

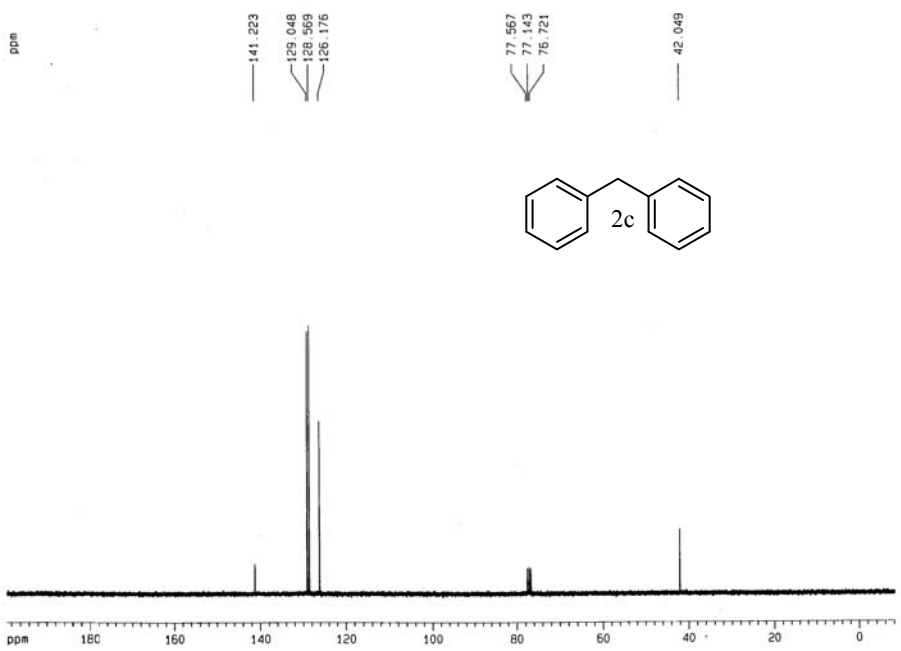


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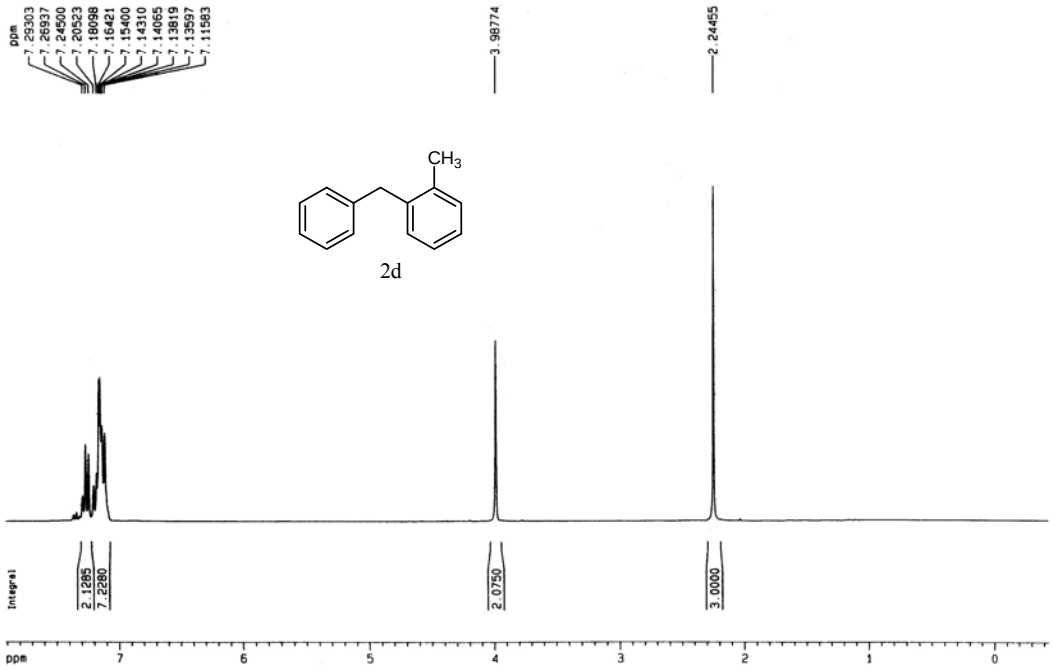

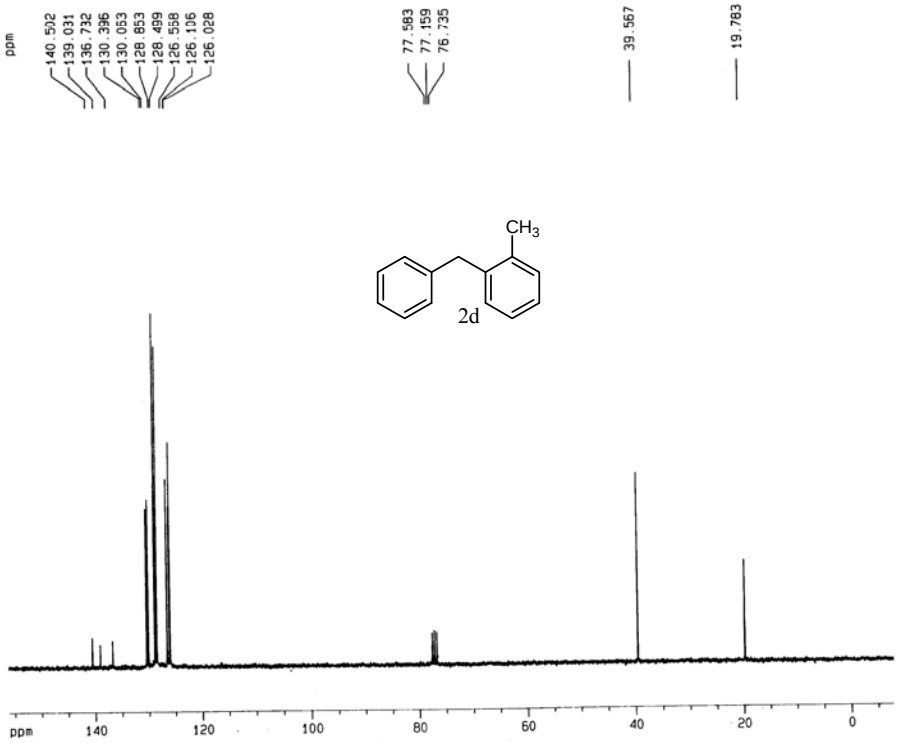


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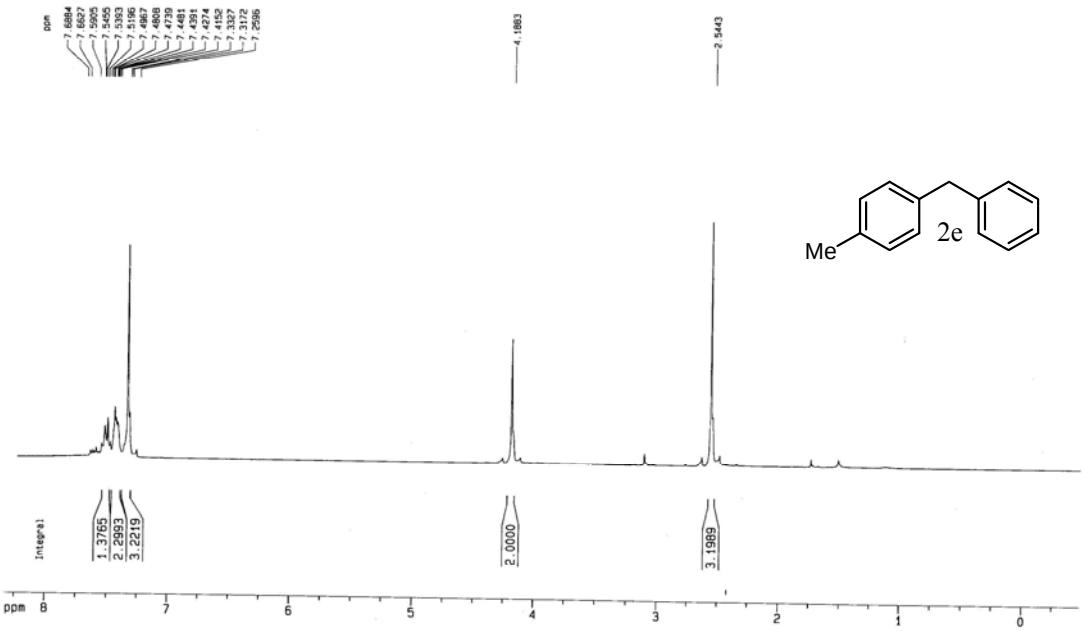
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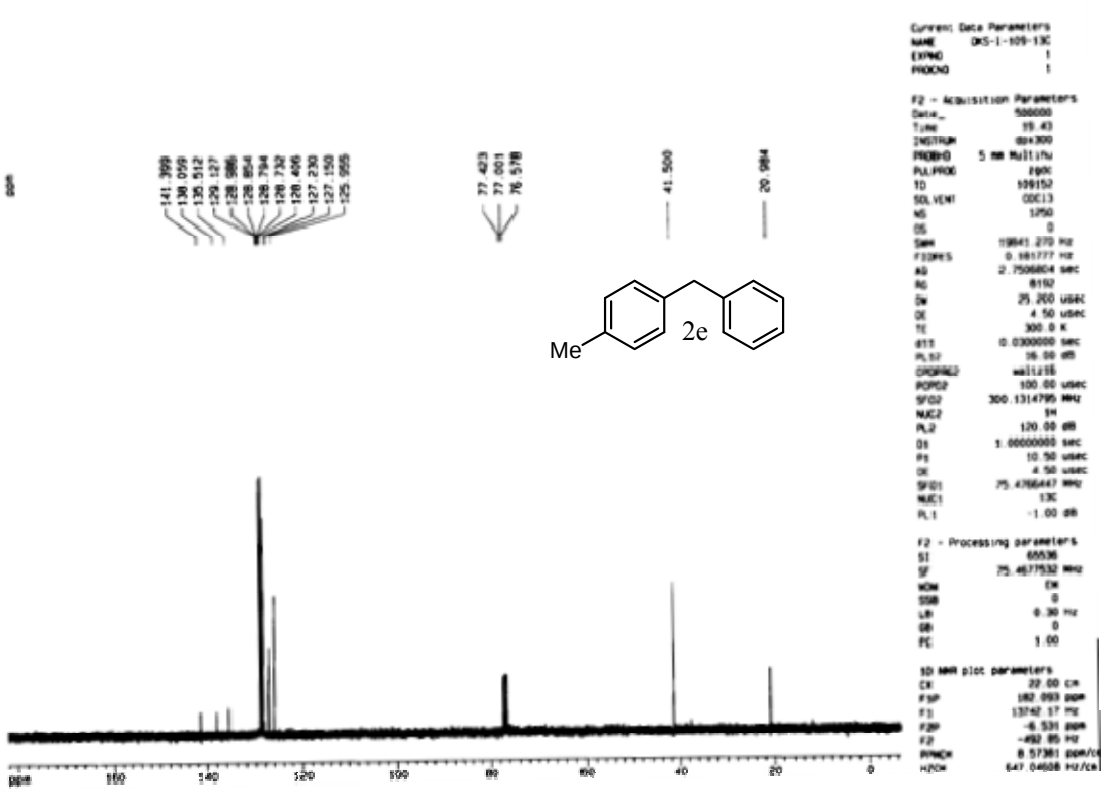
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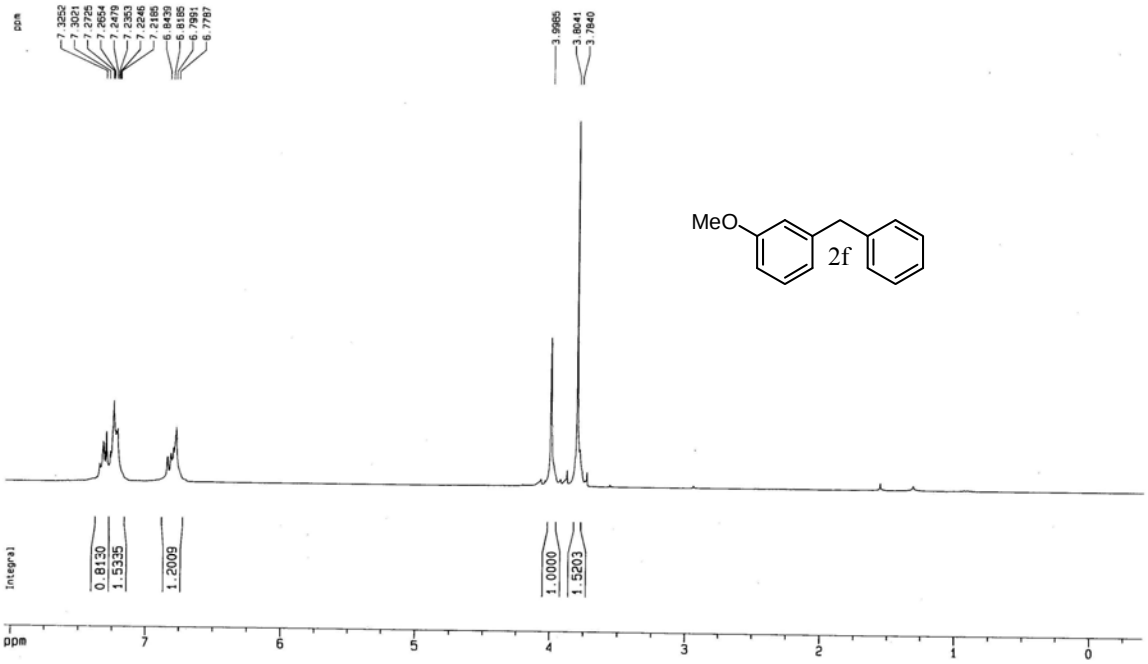
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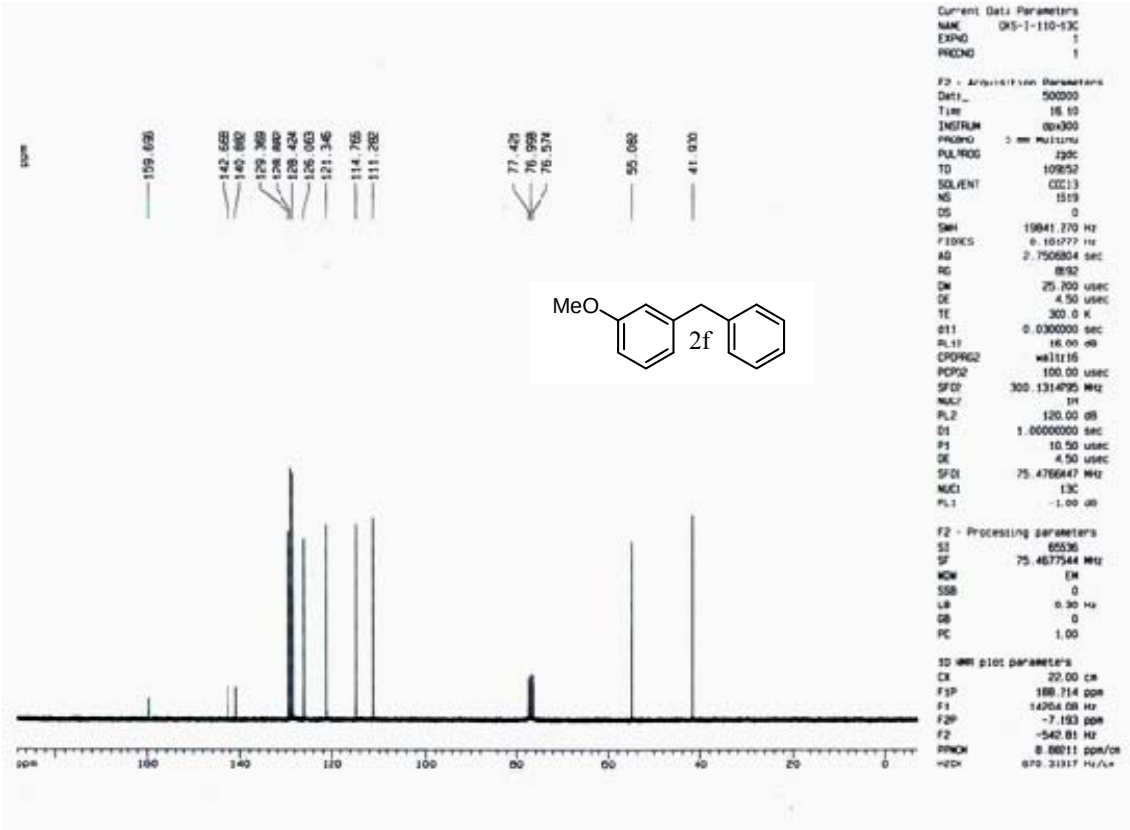
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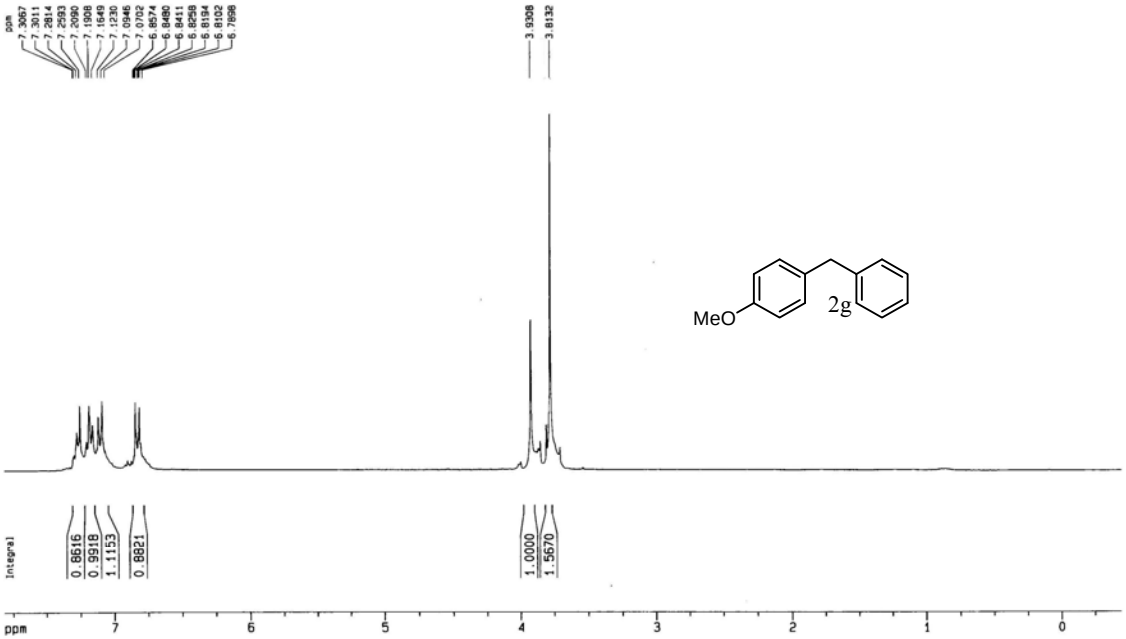
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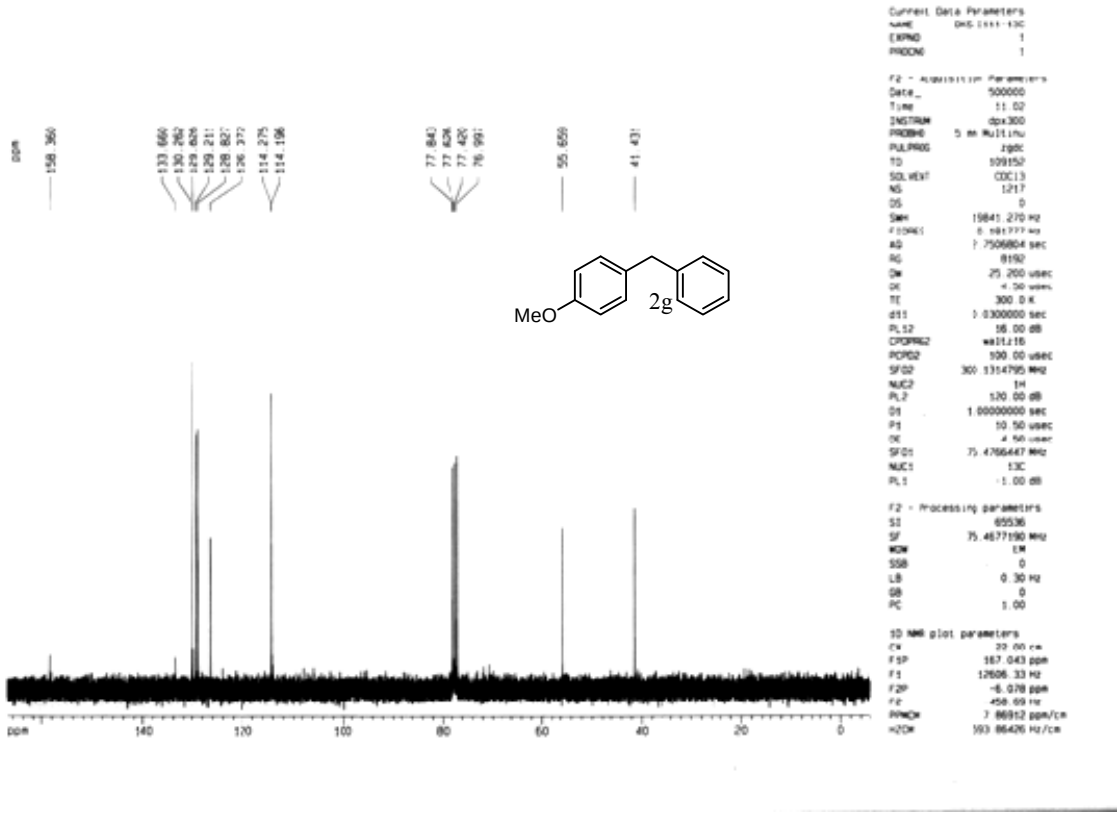
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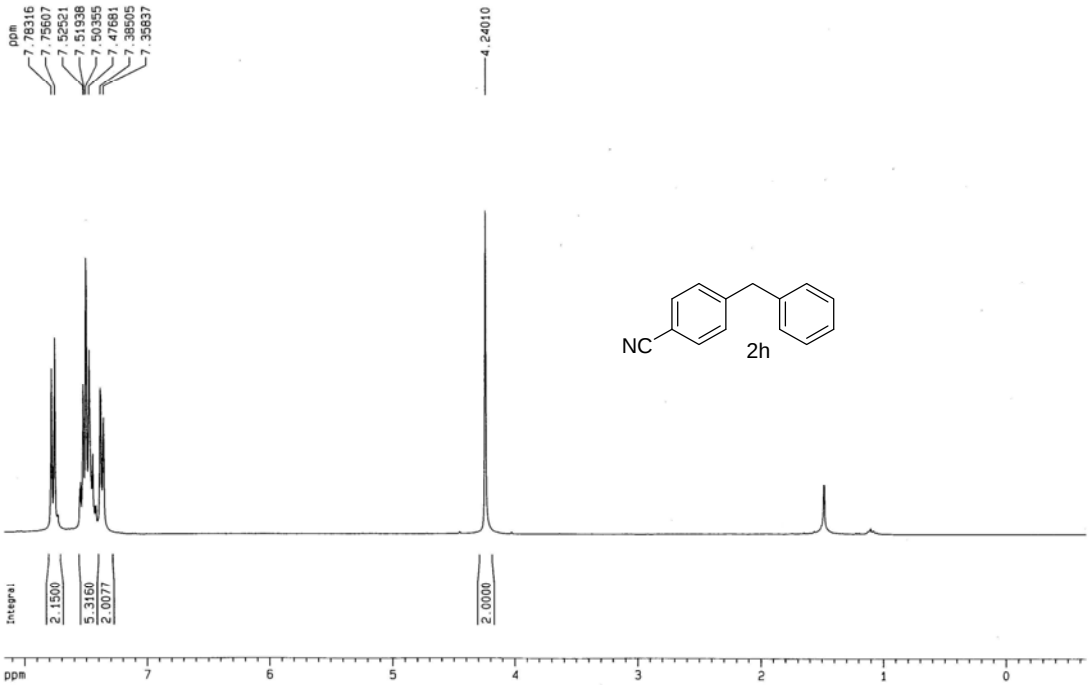

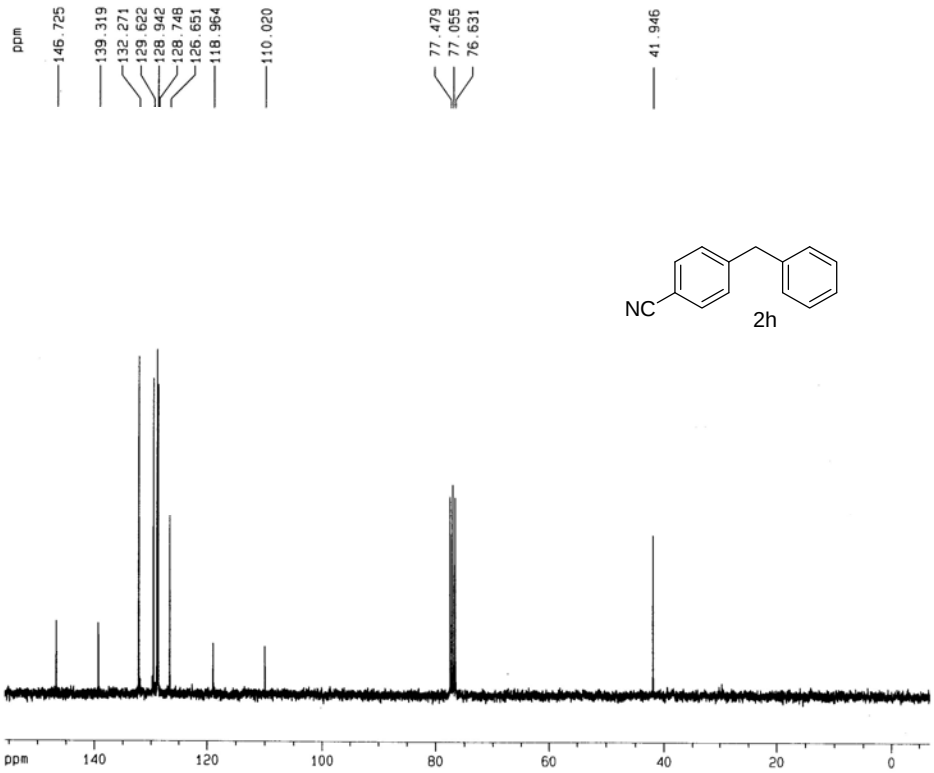


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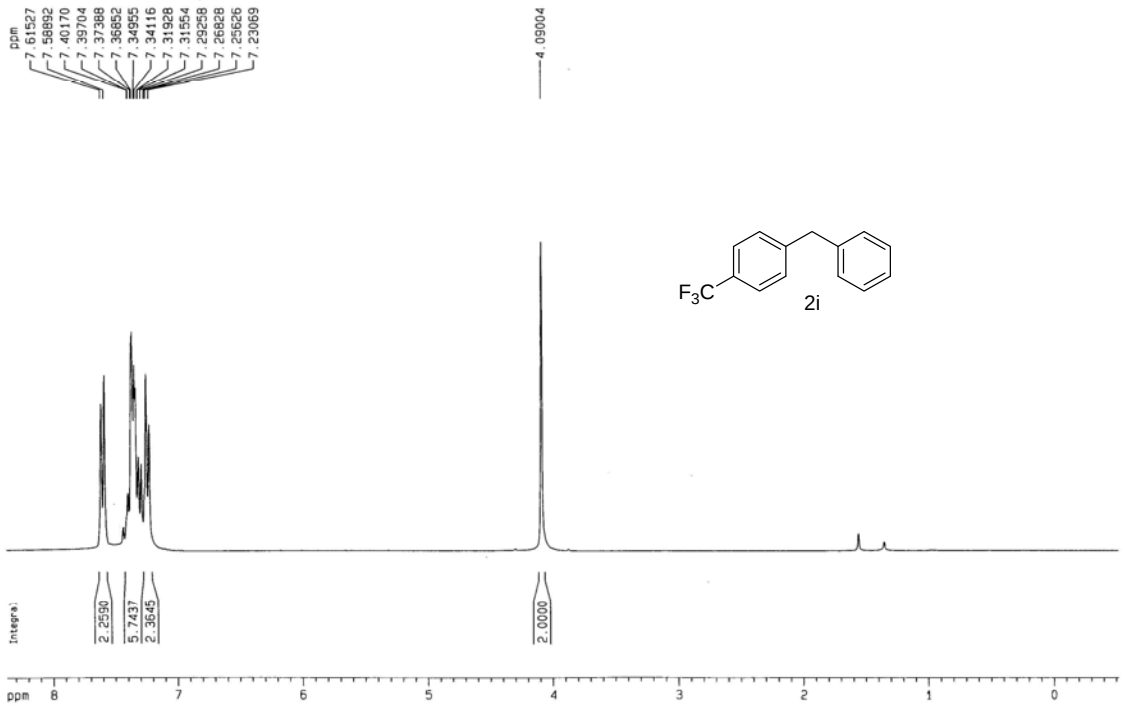

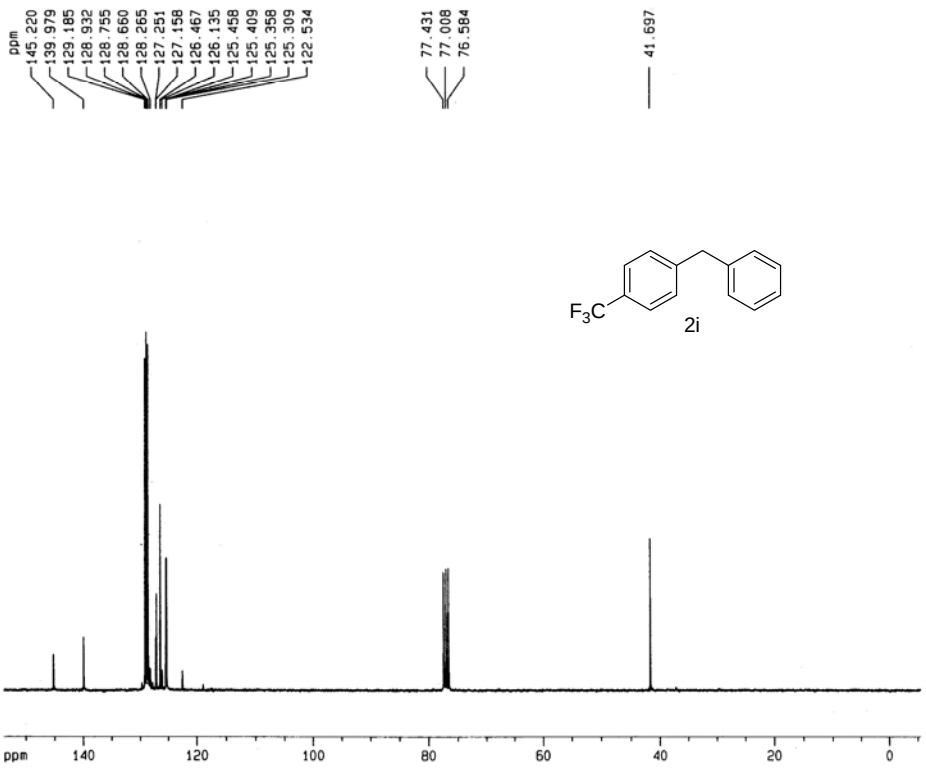


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F2 - Processing parameters
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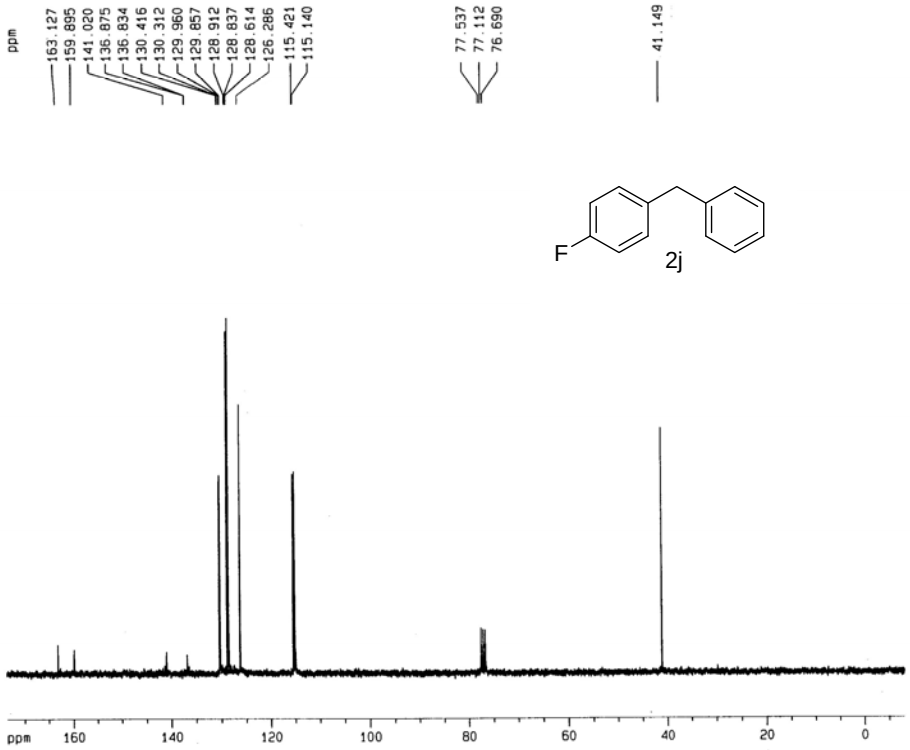
SF             75.4677534 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     7.74 cm
F1P                 153.832 ppm
F1                11609.35 Hz
F2P                  -5.525 ppm
F2                 -416.99 Hz
PPMCM               7.96786 ppm/cm
HZCM              601.31677 Hz/cm
```




```
Current Data Parameters
NAME        OKS.I.FBD-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090831
Time                11.10
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG              zgdc
TD                   65536
SOLVENT              D20
NS                      93
DS                       4
SWH             19841.270 Hz
FIDRES           0.302754 Hz
AQ              1.6515572 sec
RG                   32768
DW                  25.200 usec
DE                   6.00 usec
TE                  299.2 K
D1              2.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

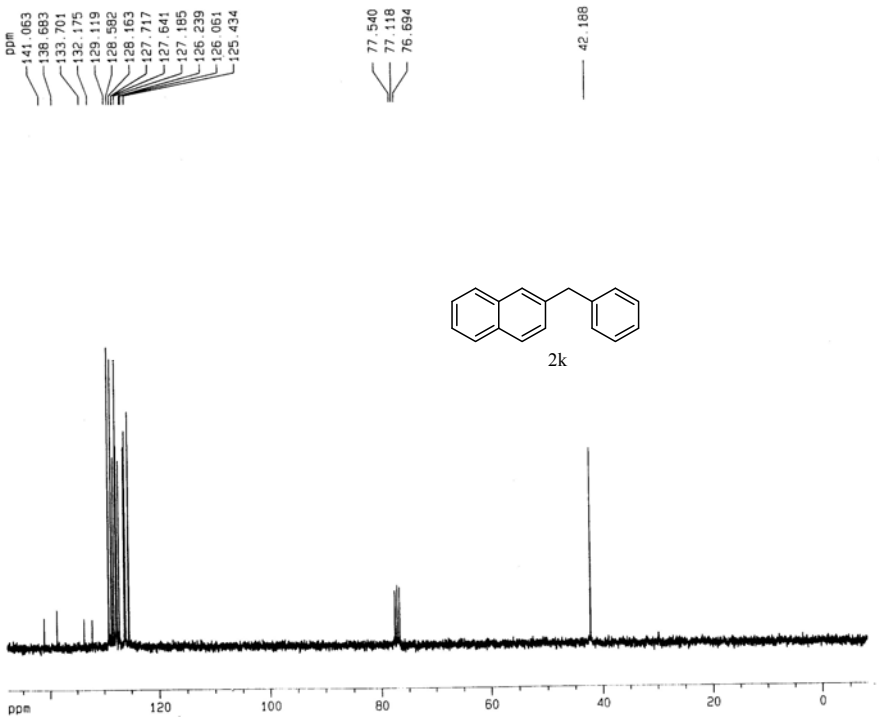
======== CHANNEL f1 ========
NUC1                   13C
P1                   13.00 usec
PL1                   0.00 dB
SFO1            75.4763276 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -6.00 dB
PL12                  7.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.60 cm
CY                    8.02 cm
F1P                173.578 ppm
F1                13099.57 Hz
F2P                 -7.995 ppm
F2                 -603.38 Hz
PPMCM              9.07868 ppm/cm
HZCM             685.14783 Hz/cm
```

2k



2k

```
Current Data Parameters
NAME      DKS.I.Nap-13C
EXPNO                 1
PROCNO                1

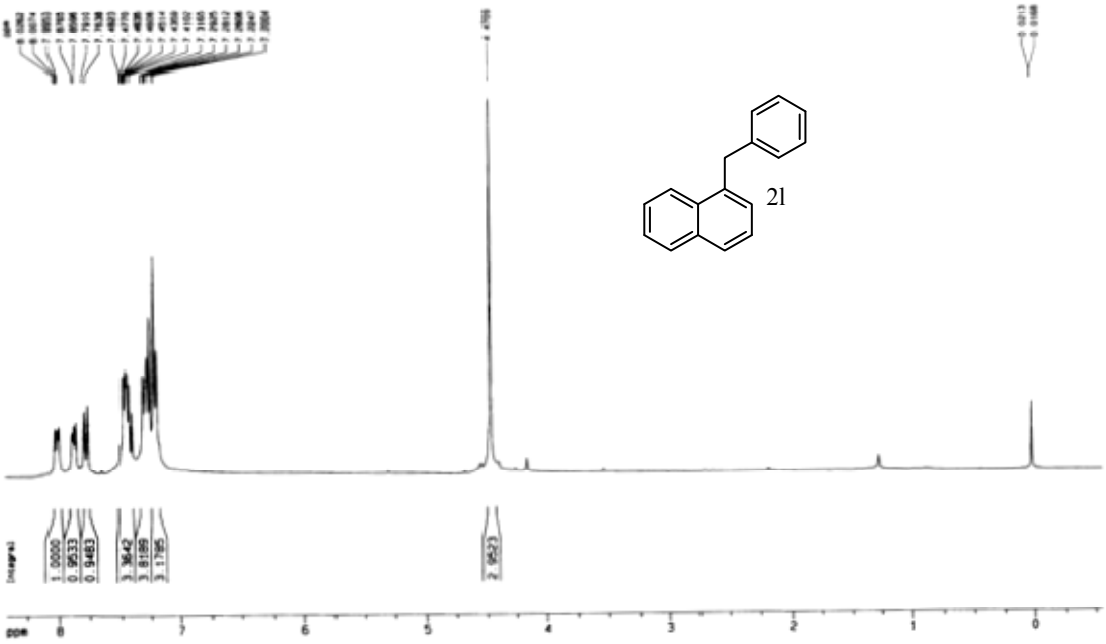
F2 - Acquisition Parameters
Date_          20090616
Time              13.30
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG           zgdc
TD                65536
SOLVENT             D20
NS                   59
DS                    4
SWH           19841.270 Hz
FIDRES         0.302754 Hz
AQ            1.6515572 sec
RG                32768
DW               25.200 usec
DE                6.00 usec
TE               298.2 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

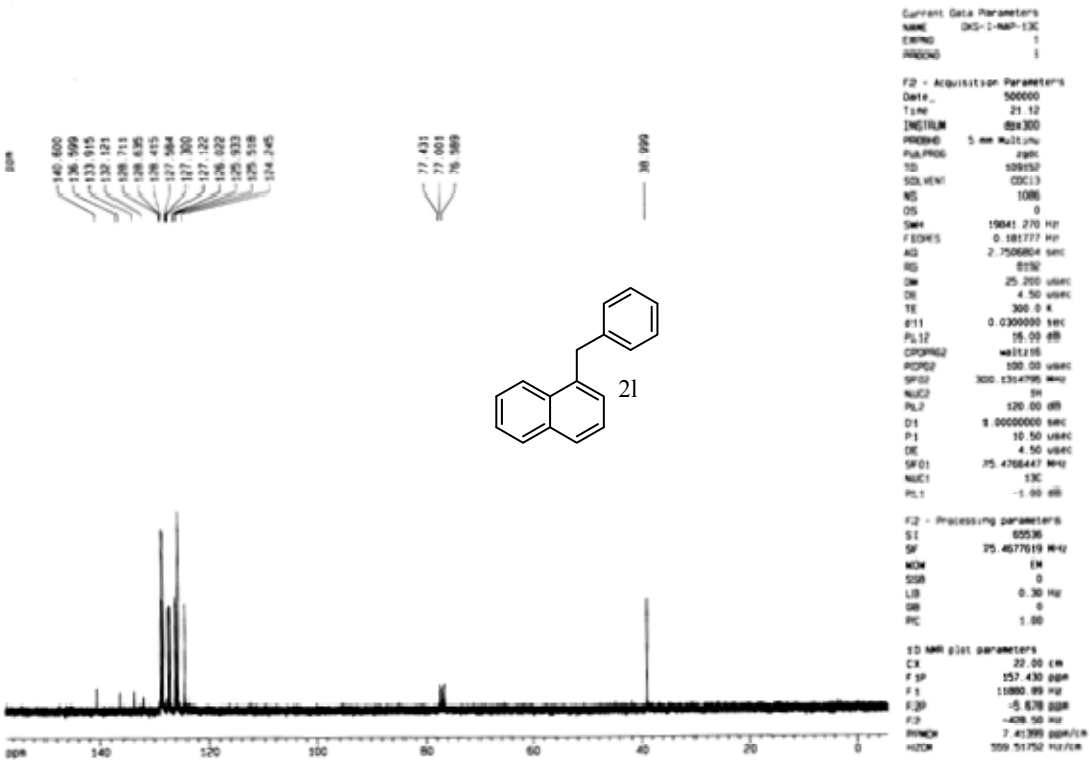
======== CHANNEL f1 ========
NUC1                 13C
P1                13.00 usec
PL1                0.00 dB
SFO1          75.4763276 MHz

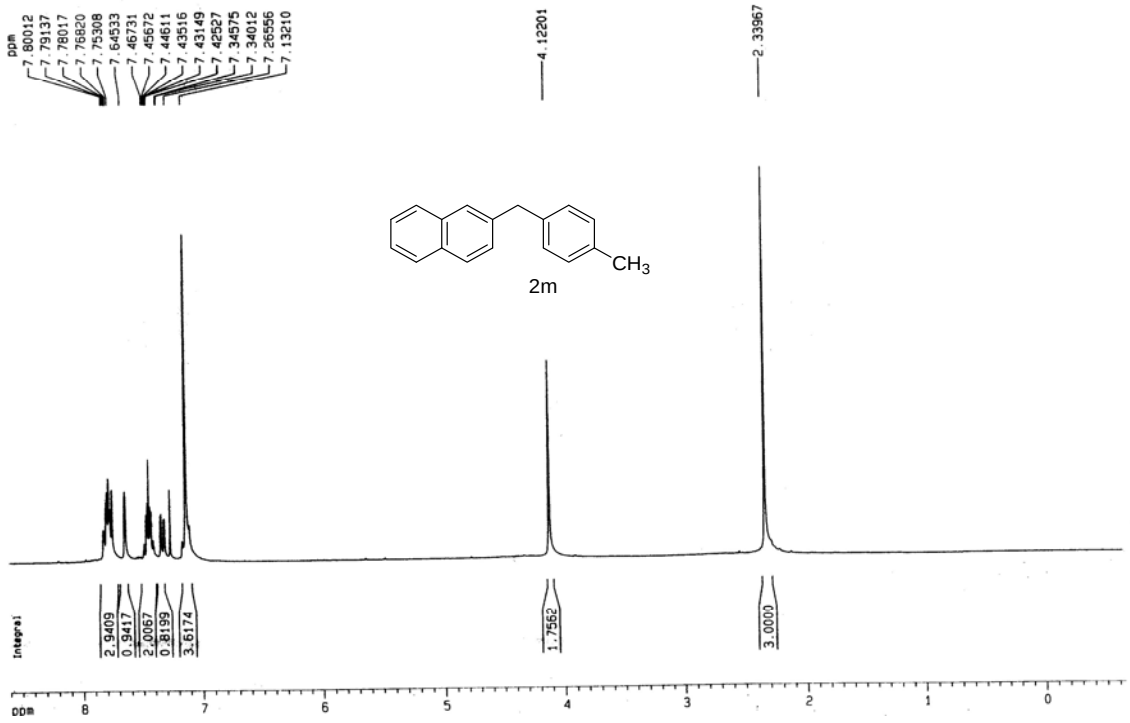
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12               7.00 dB
SFO2         300.1312005 MHz

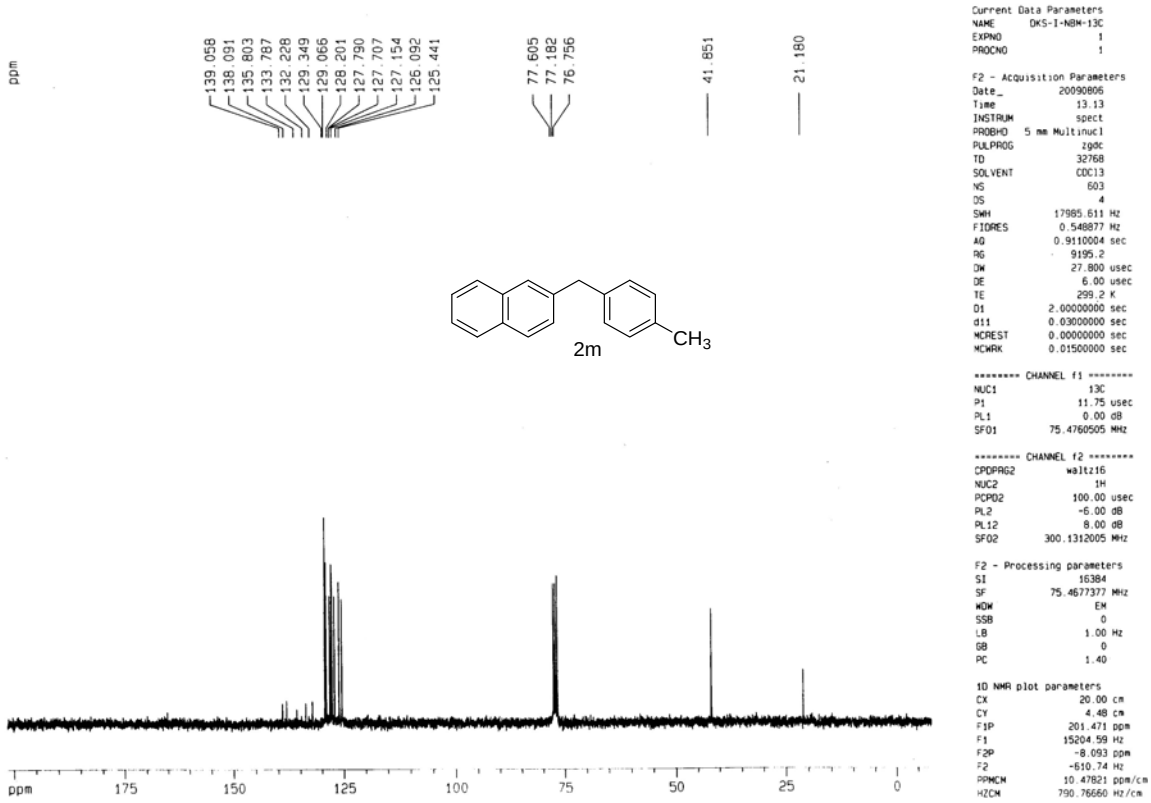
F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 6.99 cm
F1P             147.791 ppm
F1            11153.44 Hz
F2P             -8.082 ppm
F2            -609.96 Hz
PPMCM           7.79366 ppm/cm
HZCM          588.17004 Hz/cm
```


21
ppm
8
7
6
5
4
3
2
1
0


21
ppm
140
120
100
80
60
40
20
0
Current Data Parameters
NAME DKS-I-NAP-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 21.12
INSTRUM dpx300
PROBHD 5 mm Multinu
PULPROG zgdc
TD 109152
SOLVENT CDCl3
NS 1086
DS 0
SWH 19841.270 Hz
FIDRES 0.181777 Hz
AQ 2.7506804 sec
RG 8192
DW 25.200 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 16.00 dB
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1314795 MHz
NUC2 1H
PL2 120.00 dB
D1 9.00000000 sec
P1 10.50 usec
DE 4.50 usec
SFO1 75.4766447 MHz
NUC1 13C
PL1 -1.00 dB
F2 - Processing parameters
SI 65536
SF 75.4677619 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
F1P 157.430 ppm
F1 11880.89 Hz
F2P -5.678 ppm
F2 -428.50 Hz
PPMCM 7.41399 ppm/cm
HZCM 559.51752 Hz/cm






```
Current Data Parameters
NAME        DKS-I-NBM-13C
EXPNO                   1
PROCNO                  1

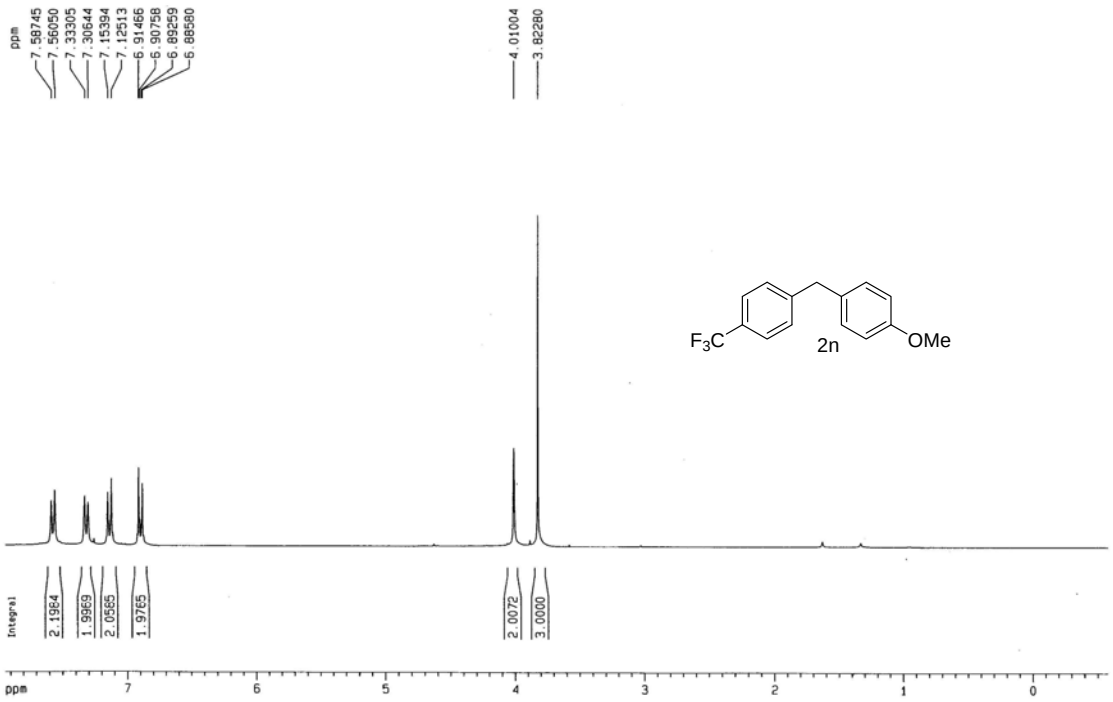
F2 - Acquisition Parameters
Date_             20090806
Time                 13.13
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG              zgdc
TD                   32768
SOLVENT              CDCl3
NS                     603
DS                       4
SWH             17985.611 Hz
FIDRES           0.548877 Hz
AQ              0.9110004 sec
RG                 9195.2
DW                 27.800 usec
DE                  6.00 usec
TE                 299.2 K
D1              2.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

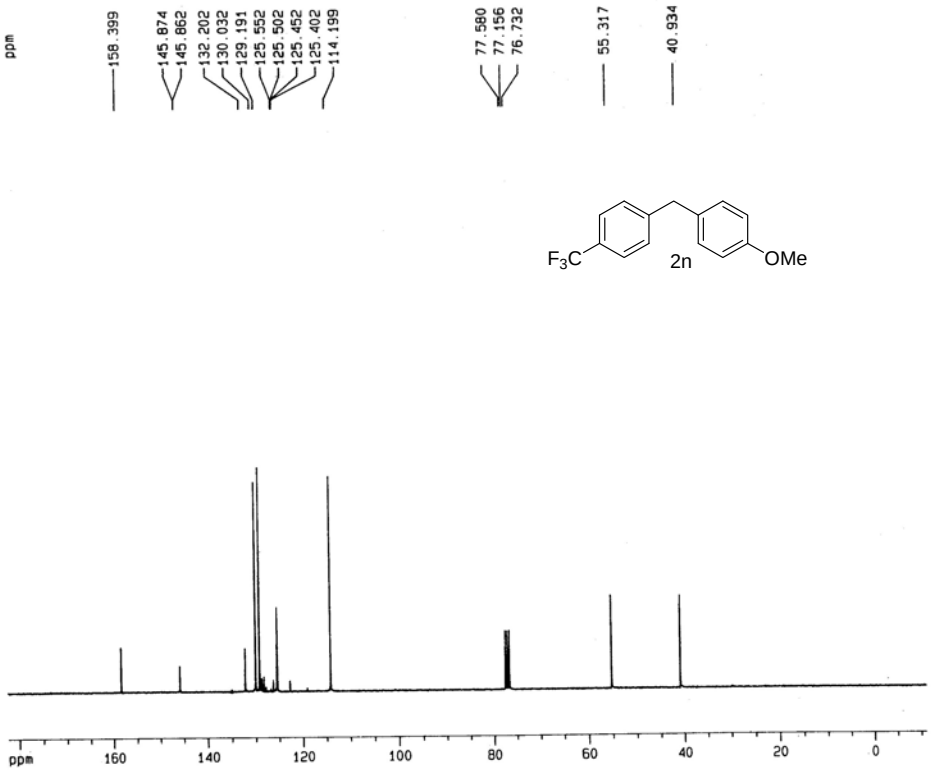
======== CHANNEL f1 ========
NUC1                  13C
P1                  11.75 usec
PL1                  0.00 dB
SFO1            75.4760505 MHz

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

F2 - Processing parameters
SI                    16384
SF              75.4677377 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     4.48 cm
F1P                 201.471 ppm
F1                15204.59 Hz
F2P                  -8.093 ppm
F2                  -610.74 Hz
PPMCM              10.47821 ppm/cm
HZCM              790.76660 Hz/cm
```






```
Current Data Parameters
NAME        DKS-I-CFMB-13C
EXPNO                    1
PROCNO                   1

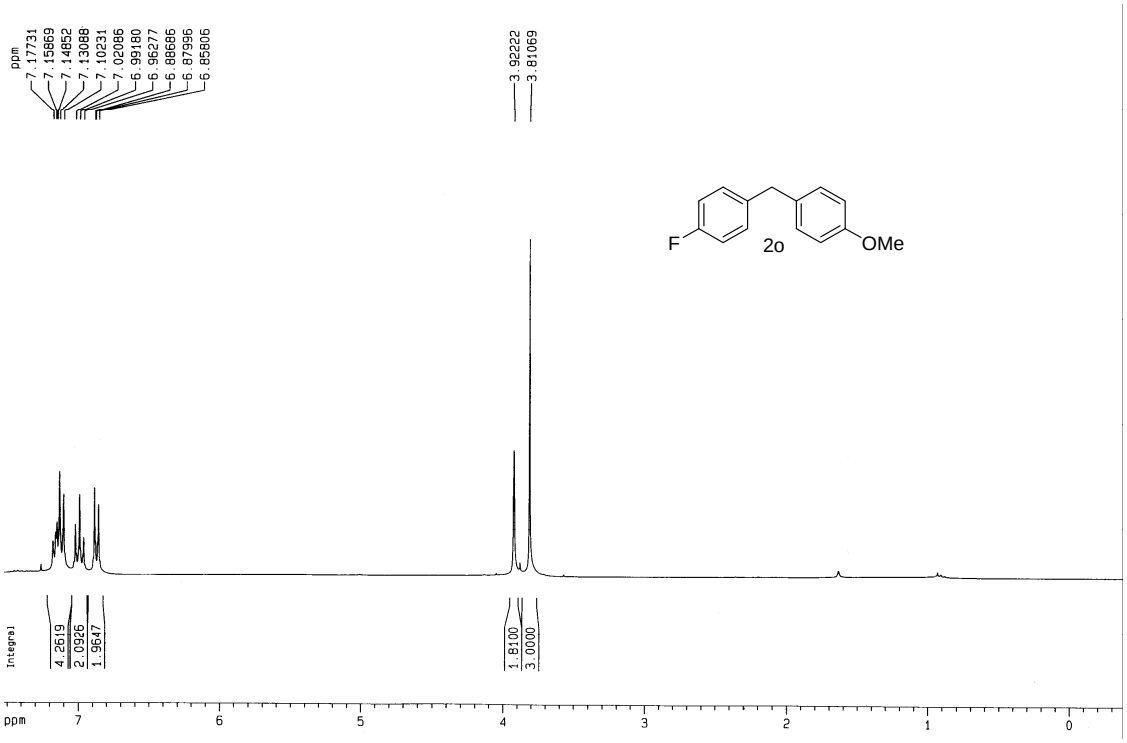
F2 - Acquisition Parameters
Date_             20091015
Time                 18.11
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     588
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ               1.8219508 sec
RG                 10321.3
DW                  27.800 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

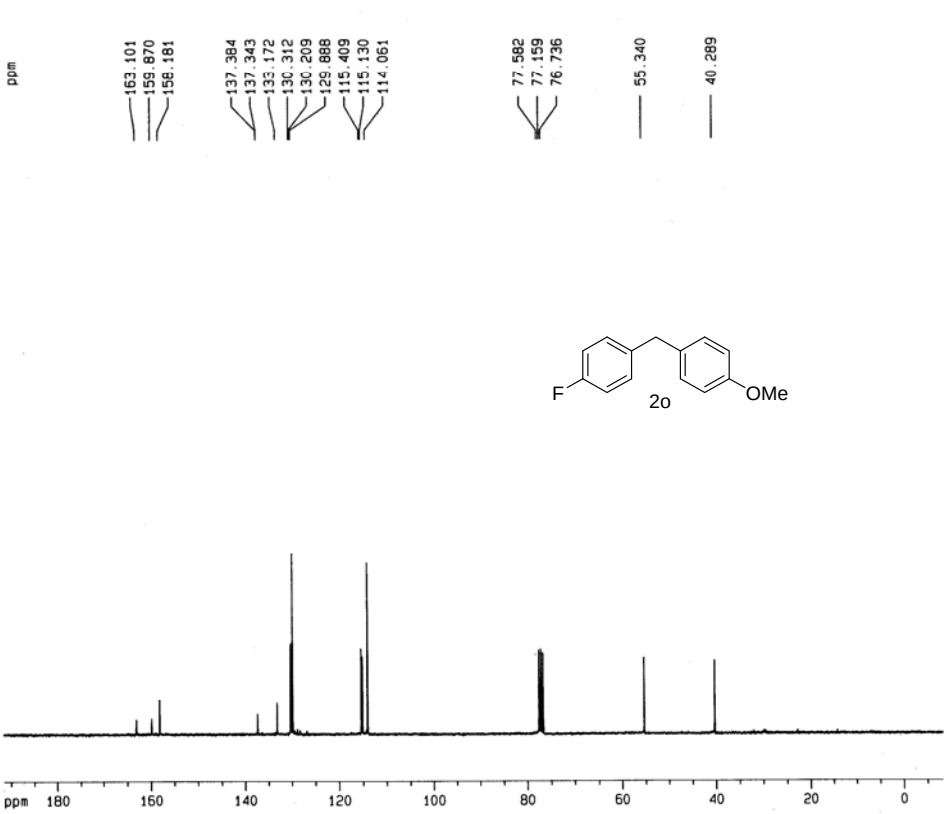
======== CHANNEL f1 ========
NUC1                   13C
P1                   11.75 usec
PL1                   0.00 dB
SFO1            75.4752953 MHz

======== 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              75.4677427 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     4.84 cm
F1P                  182.396 ppm
F1                 13765.02 Hz
F2P                  -11.024 ppm
F2                  -831.99 Hz
PPMCM                9.67103 ppm/cm
HZCM               729.85089 Hz/cm
HZCM               647.30817 Hz/cm
```






```
Current Data Parameters
NAME      DKS-I-2c1MB-13C
EXPNO                   1
PROCNO                  1

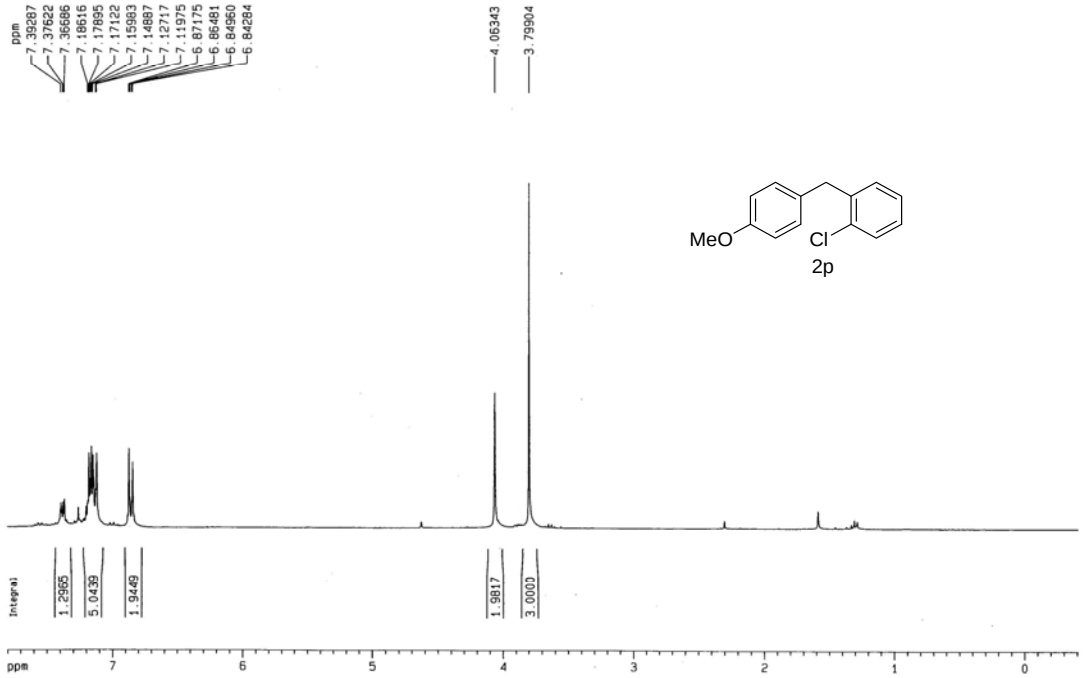
F2 - Acquisition Parameters
Date_            20091023
Time                19.21
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    284
DS                      4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 9195.2
DW                 27.800 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

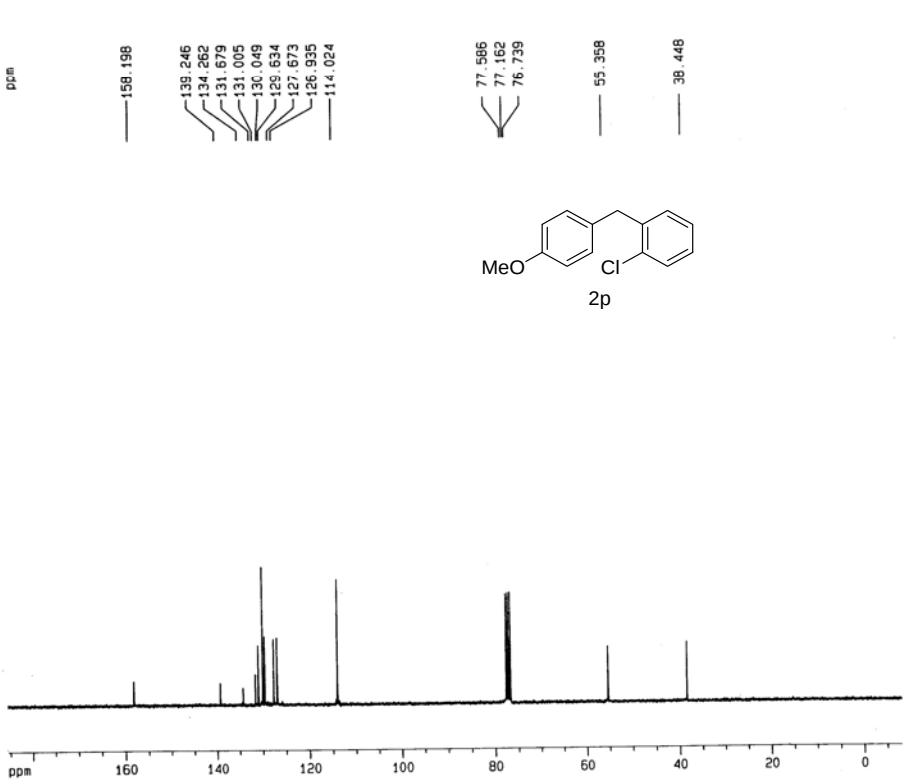
======== CHANNEL f1 ========
NUC1                  13C
P1                  11.75 usec
PL1                  0.00 dB
SFO1           75.4752953 MHz

======== 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             75.4677432 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   3.90 cm
F1P                191.599 ppm
F1               14459.57 Hz
F2P                 -8.153 ppm
F2                 -615.32 Hz
PPMCM              9.98764 ppm/cm
HZCM             753.74481 Hz/cm
```






```
Current Data Parameters
NAME      DKS-I-4FMB-13C
EXPNO                  1
PROCNO                 1

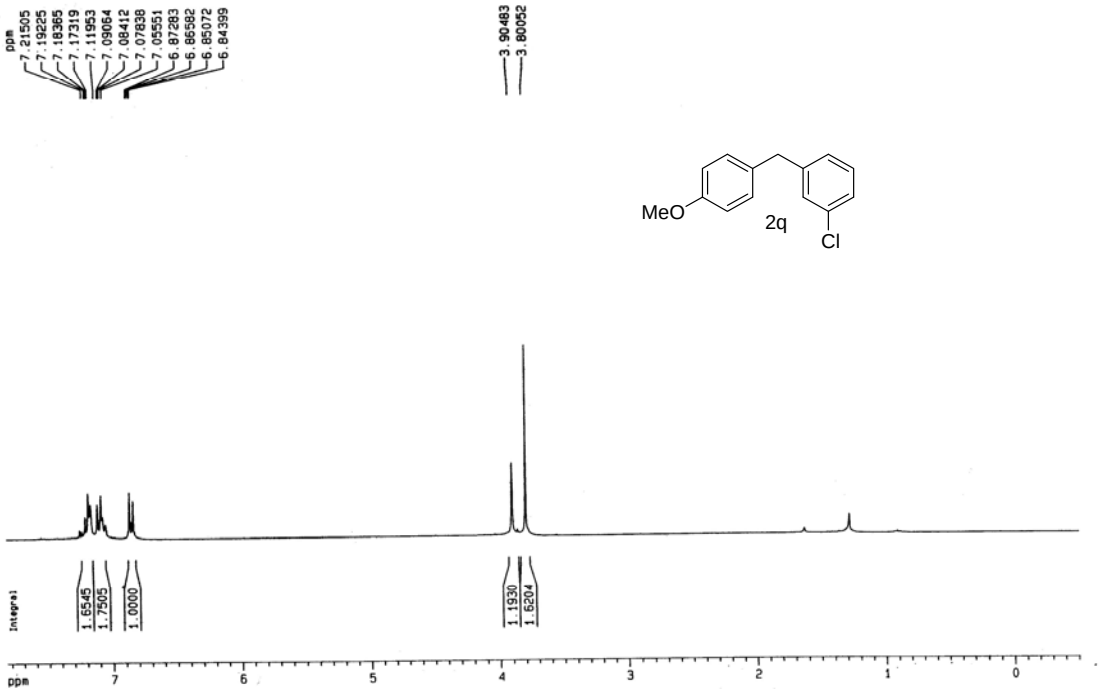
F2 - Acquisition Parameters
Date_           20091023
Time               18.36
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  335
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG               9195.2
DW               27.800 usec
DE                6.00 usec
TE               298.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.90999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

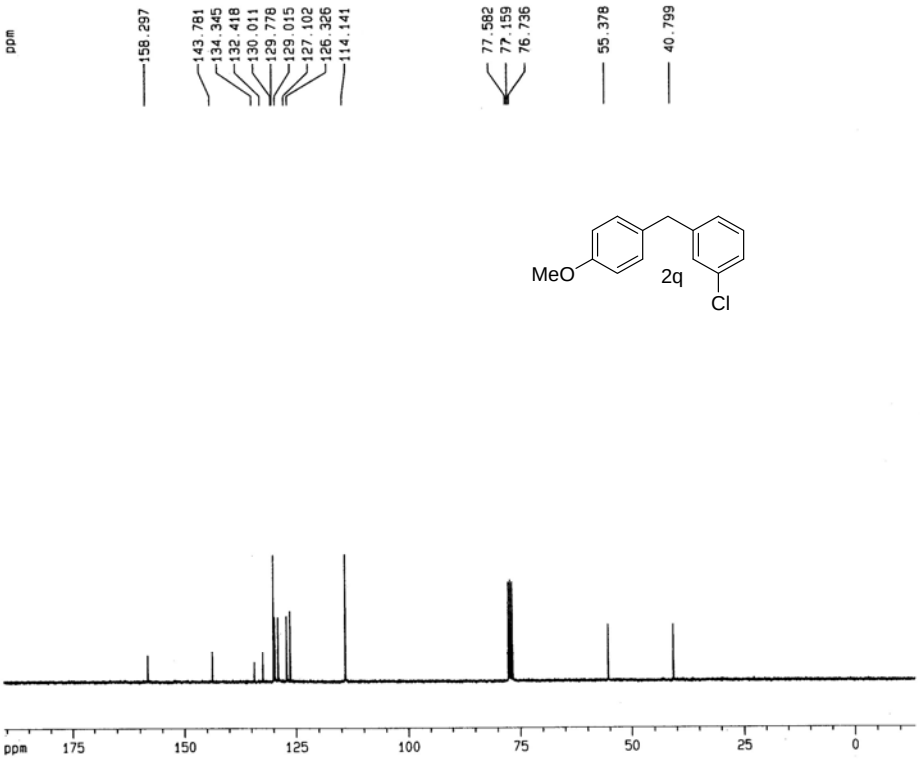
======== CHANNEL f1 ========
NUC1                13C
P1               11.75 usec
PL1               0.00 dB
SFO1         75.4752953 MHz

======== 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           75.4677410 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   3.08 cm
F1P                185.584 ppm
F1               14005.61 Hz
F2P                -8.124 ppm
F2                -613.13 Hz
PPMCM             9.68542 ppm/cm
HZCM            730.93695 Hz/cm
```






```
Current Data Parameters
NAME      DKS-I-c1MB-13C
EXPNO                  1
PROCNO                 1

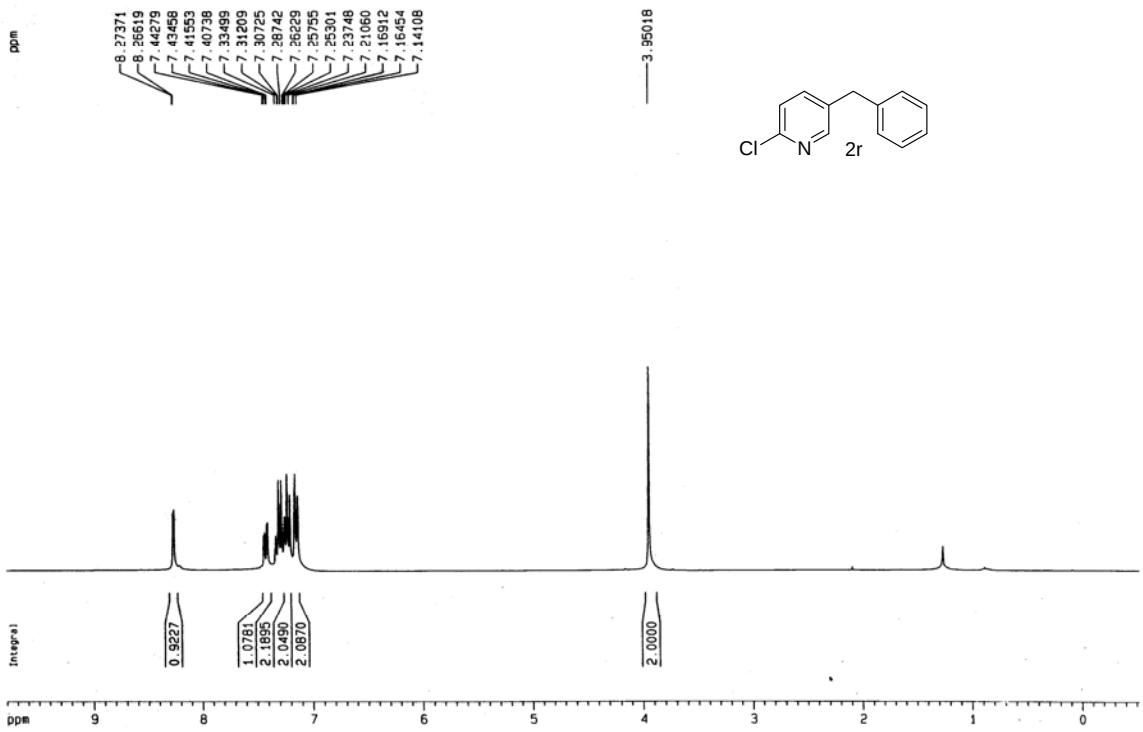
F2 - Acquisition Parameters
Date_           20091020
Time               18.47
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  332
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG              10321.3
DW               27.800 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

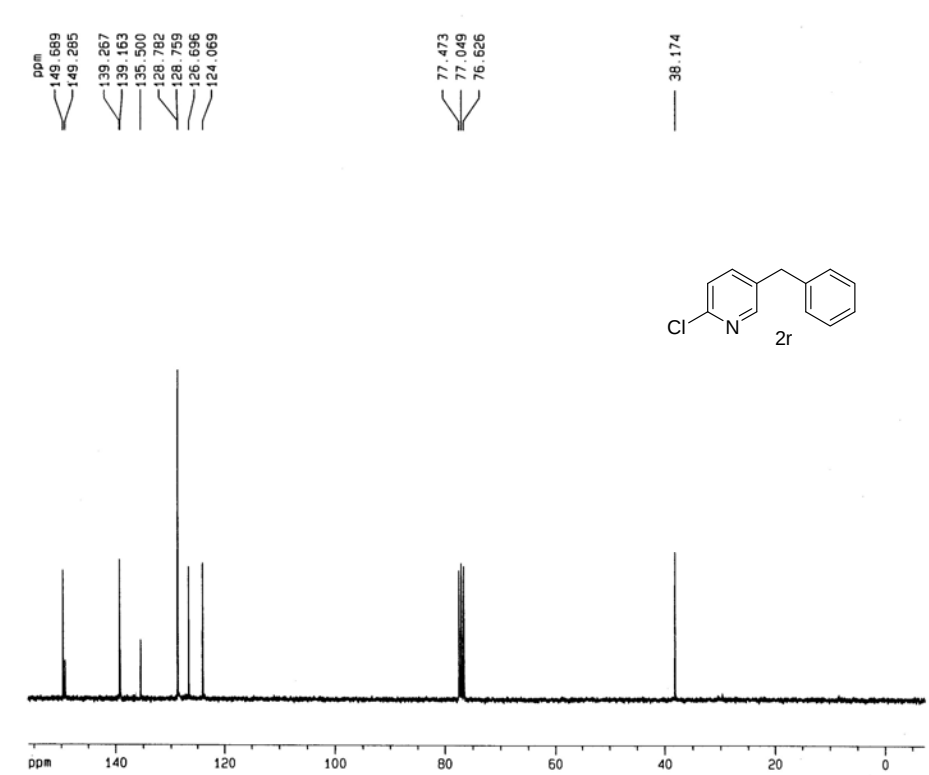
======== CHANNEL f1 ========
NUC1                13C
P1                11.75 usec
PL1               0.00 dB
SFO1         75.4752953 MHz

======== 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           75.4677411 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  2.87 cm
F1P              190.765 ppm
F1             14396.60 Hz
F2P              -13.305 ppm
F2             -1004.13 Hz
PPMCM            10.20351 ppm/cm
HZCM           770.03619 Hz/cm
```






```
Current Data Parameters
NAME        DKS.I.CPB-13C
EXPNO                   1
PROCNO                  1

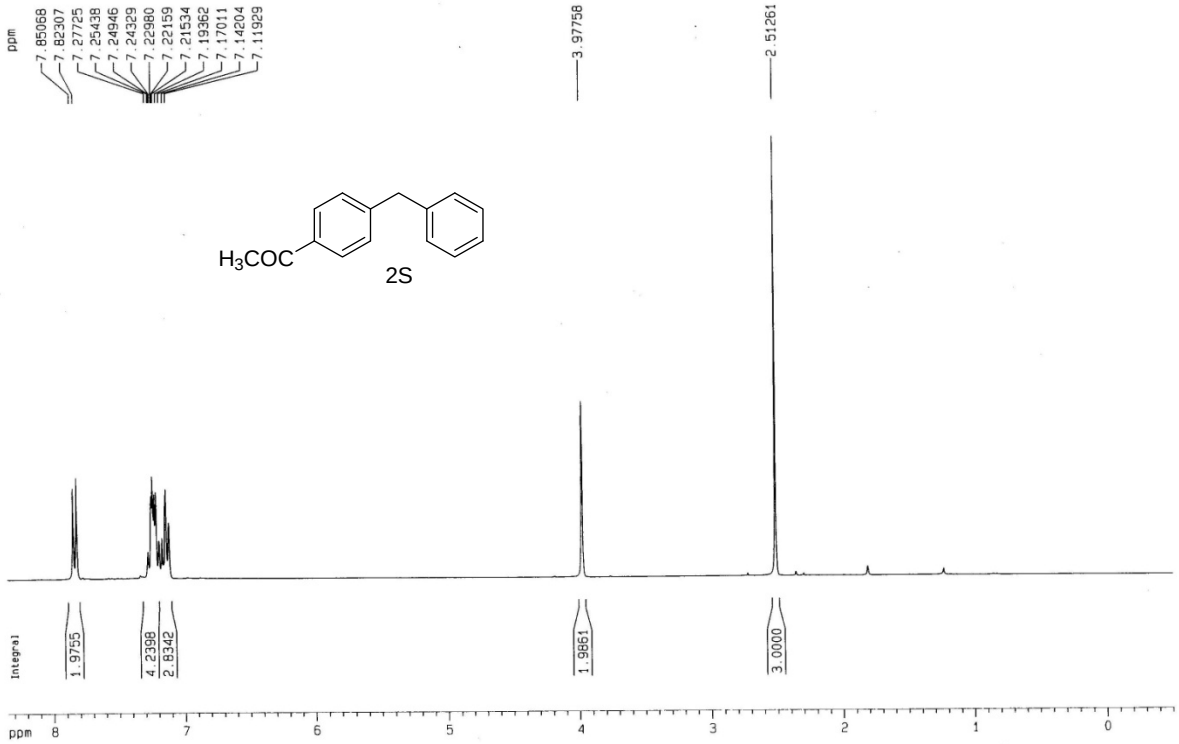
F2 - Acquisition Parameters
Date_             20091009
Time                 12.57
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     172
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ               1.8219508 sec
RG                  9195.2
DW                  27.800 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

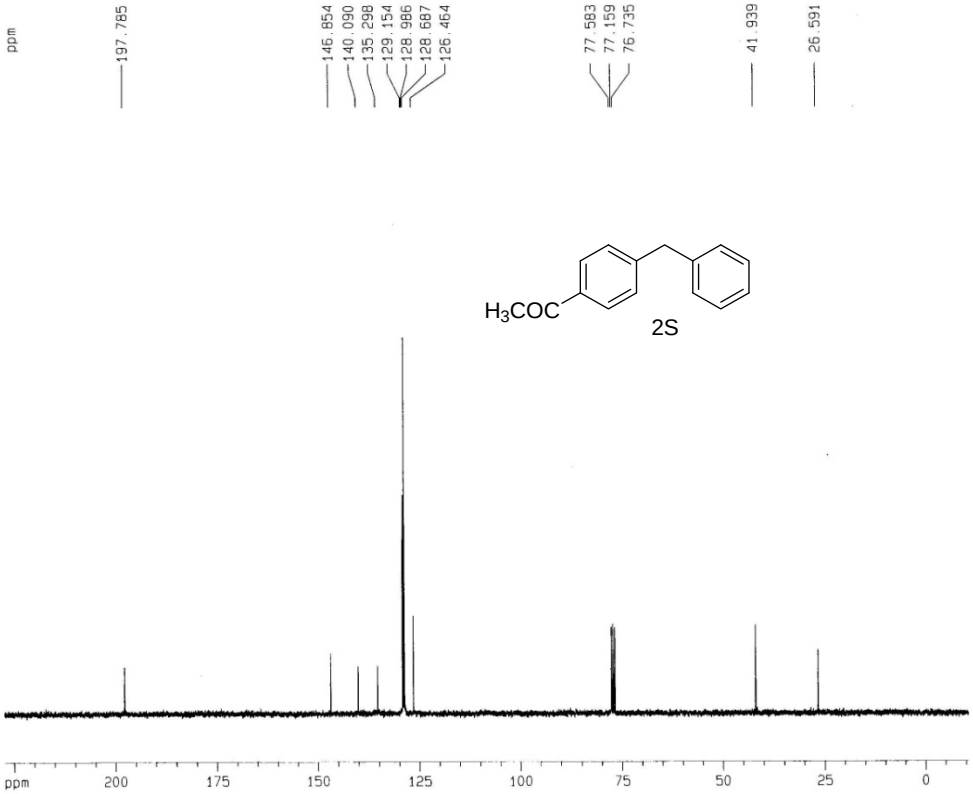
======== CHANNEL f1 ========
NUC1                   13C
P1                   11.75 usec
PL1                   0.00 dB
SFO1            75.4752953 MHz

======== 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              75.4677522 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    7.27 cm
F1P                 156.121 ppm
F1                11782.07 Hz
F2P                 -7.308 ppm
F2                 -551.55 Hz
PPMCM               8.17145 ppm/cm
HZCM              616.68079 Hz/cm
```






```
Current Data Parameters
NAME          DKSI.CO-13C
EXPNO                   1
PROCNO                  1

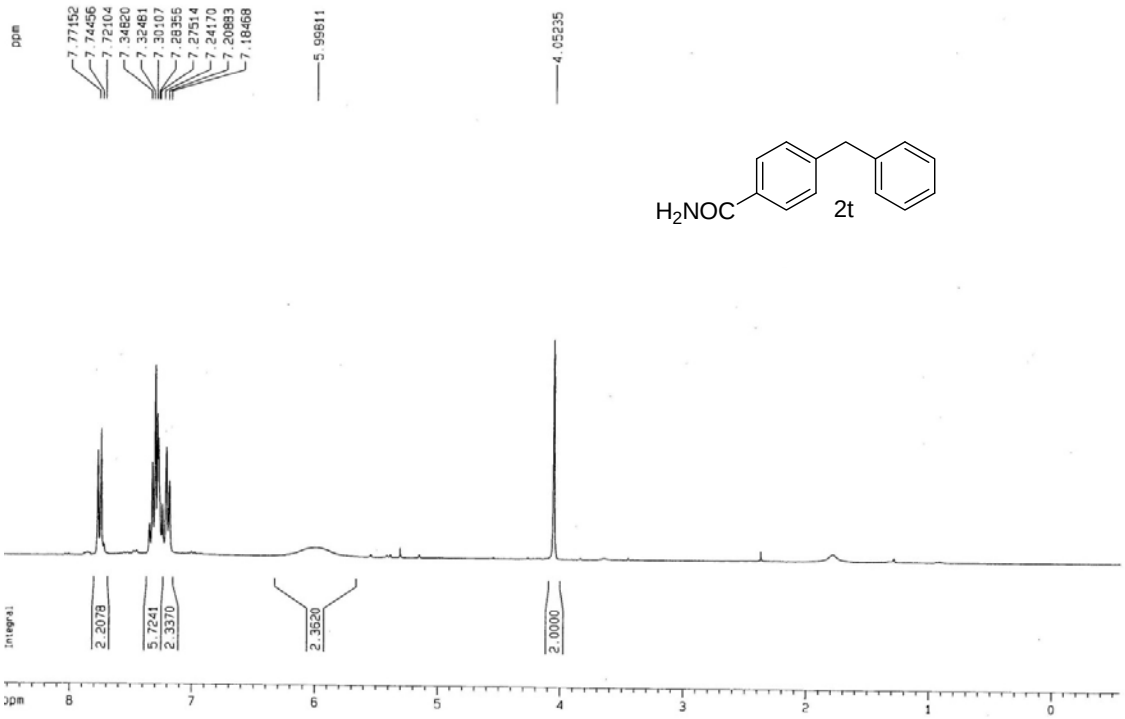
F2 - Acquisition Parameters
Date_            20100405
Time               12.15
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                   33
DS                    4
SWH           18939.395 Hz
FIDRES         0.288992 Hz
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

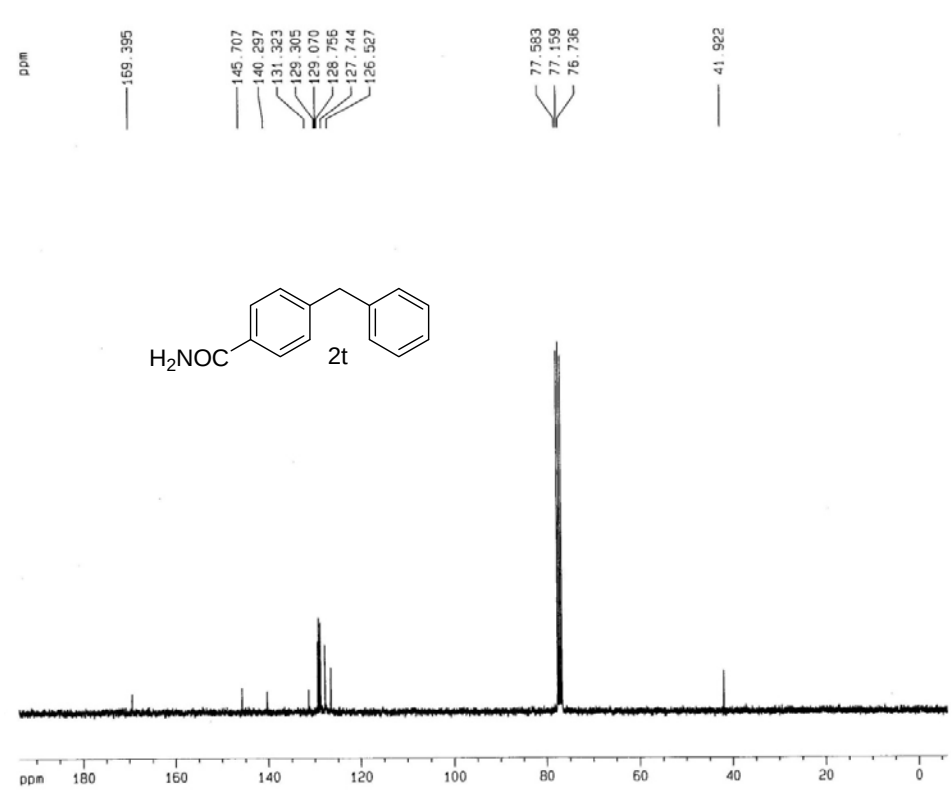
======== CHANNEL f1 ========
NUC1                13C
P1               11.75 usec
PL1               0.00 dB
SFO1         75.4763276 MHz

======== 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          75.4677487 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX               20.00 cm
CY                7.83 cm
F1P             227.541 ppm
F1            17172.01 Hz
F2P            -10.581 ppm
F2             -798.50 Hz
PPMCM          11.90609 ppm/cm
HZCM          898.52545 Hz/cm
```






```
NAME          DKSI.AMB-13C
EXPNO                    1
PROCNO                   1

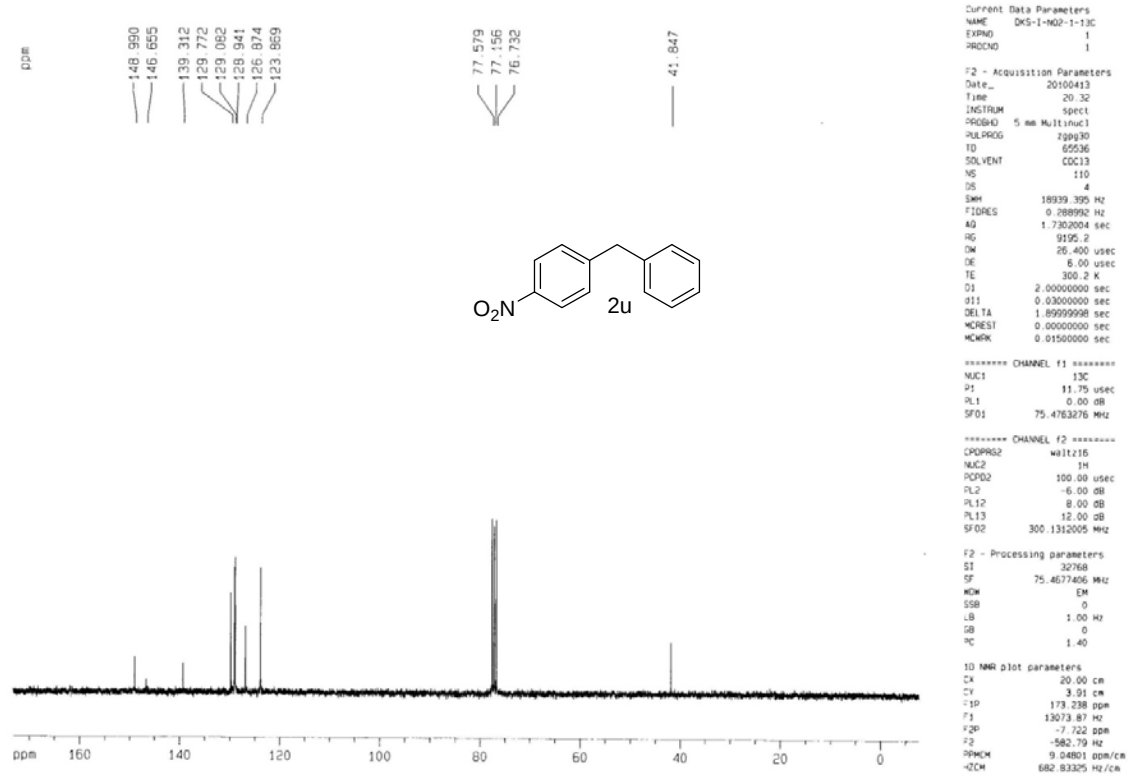
F2 - Acquisition Parameters
Date_            20100412
Time                12.11
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CD3CN
NS                   431
DS                     4
SWH            18939.395 Hz
FIDRES         0.288992 Hz
AQ             1.7302004 sec
RG               9195.2
DW                26.400 usec
DE                  6.00 usec
TE                300.2 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
SF01           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
SF02          300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF             75.4677389 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   7.94 cm
F1P               193.741 ppm
F1               14621.21 Hz
F2P                -6.171 ppm
F2                -465.71 Hz
PPMCM             9.99561 ppm/cm
HZCM            754.34613 Hz/cm
```




```
Current Data Parameters
NAME        DKS-I-N02-1-13C
EXPNO                     1
PROCNO                    1

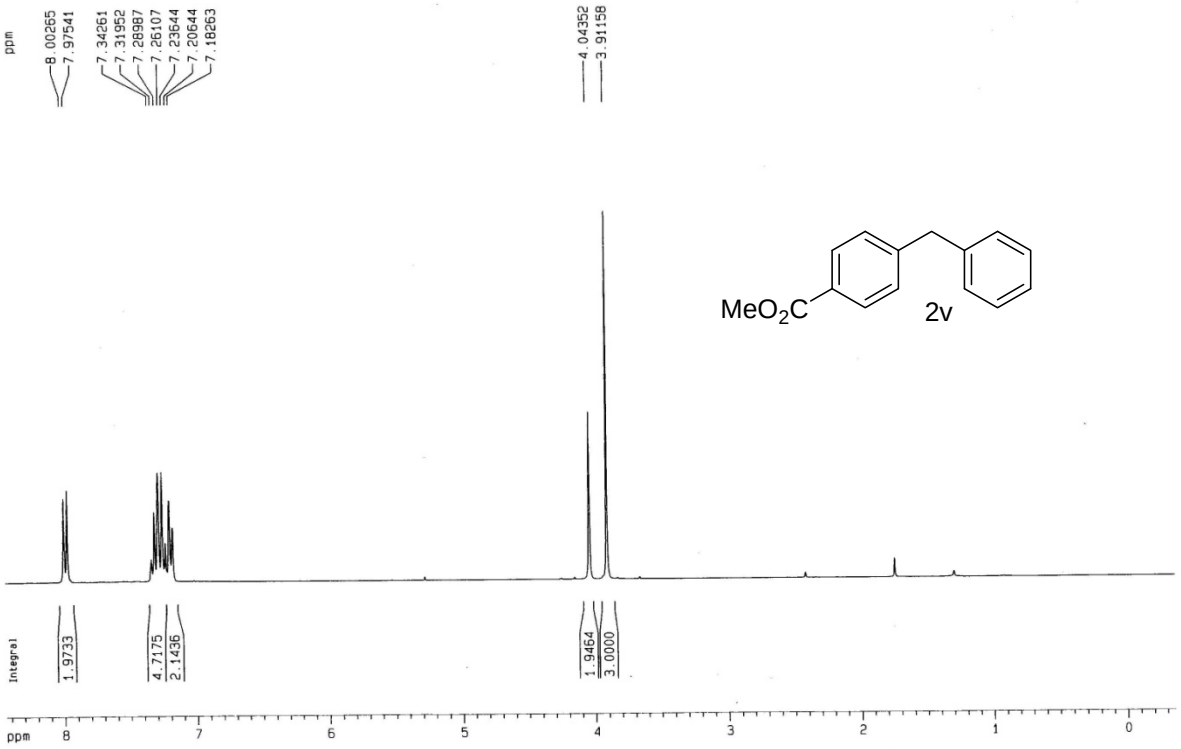
F2 - Acquisition Parameters
Date_             20100413
Time                 20.32
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     110
DS                       4
SWH              18939.395 Hz
FIDRES            0.288992 Hz
AQ                1.7302004 sec
RG                   9195.2
DW                   26.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

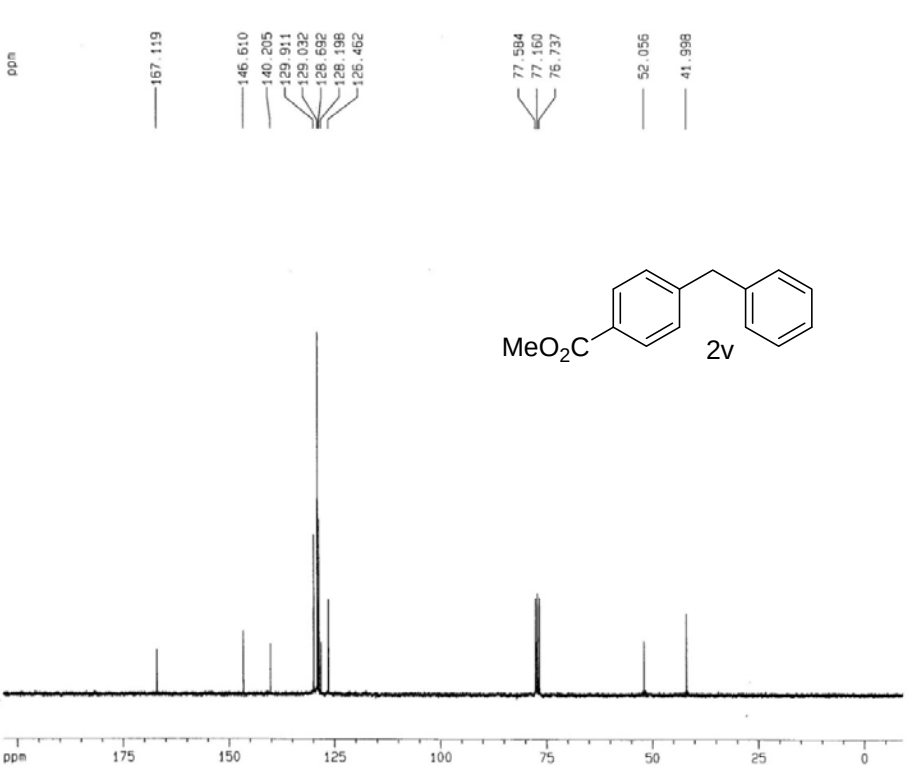
======== CHANNEL f1 ========
NUC1                    13C
P1                    11.75 usec
PL1                    0.00 dB
SFO1             75.4763276 MHz

======== 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                     3.91 cm
F1P                  173.238 ppm
F1                  13073.87 Hz
F2P                   -7.722 ppm
F2                   -582.79 Hz
PPMCM                 9.04801 ppm/cm
HZCM                682.83325 Hz/cm
```






```
Current Data Parameters
NAME        DKSI.ESB-13C
EXPNO                  1
PROCNO                 1

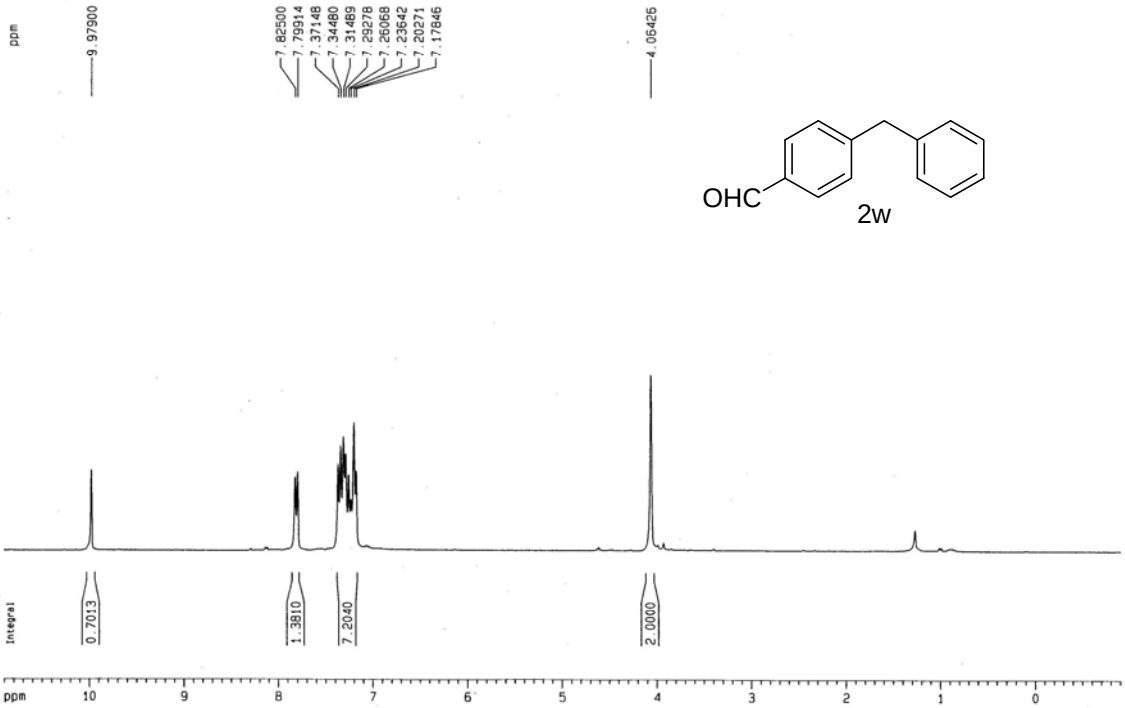
F2 - Acquisition Parameters
Date_           20100407
Time               13.36
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  105
DS                    4
SWH            18939.395 Hz
FIDRES          0.288992 Hz
AQ              1.7302004 sec
RG               9195.2
DW                26.400 usec
DE                 6.00 usec
TE                300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

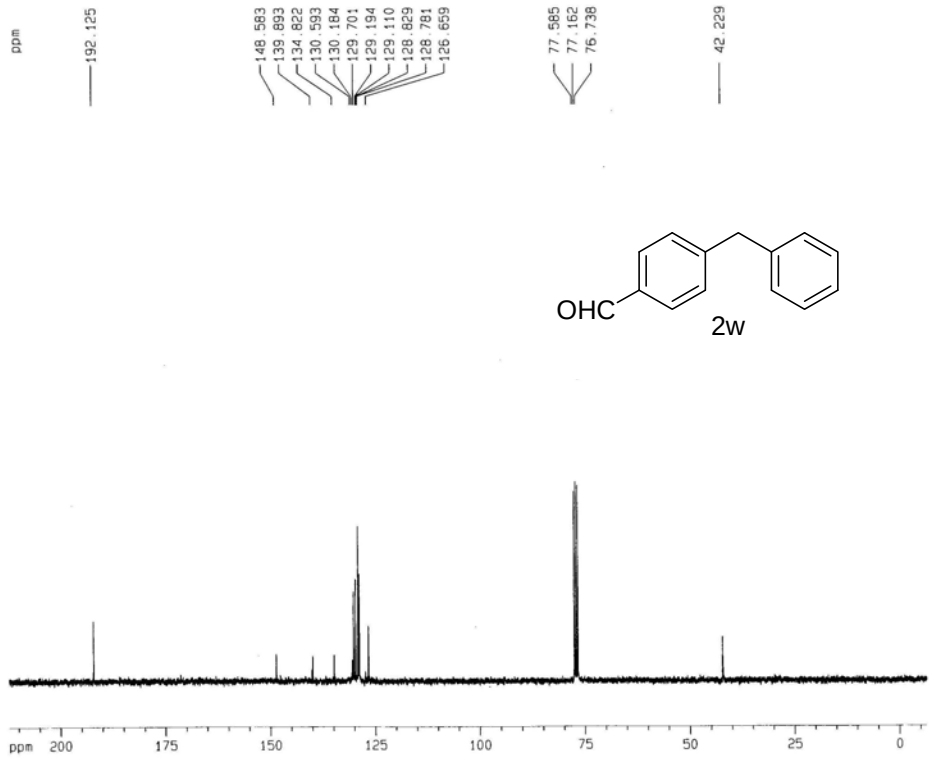
======== CHANNEL f1 ========
NUC1                13C
P1                11.75 usec
PL1                0.00 dB
SFO1          75.4763276 MHz

======== 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            75.4677452 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  8.04 cm
F1P               203.439 ppm
F1              15353.05 Hz
F2P                -9.006 ppm
F2               -679.69 Hz
PPMCM            10.62225 ppm/cm
HZCM            801.63690 Hz/cm
```






```
Current Data Parameters
NAME      DKS-I-CHO-13C
EXPNO              1
PROCNO             1

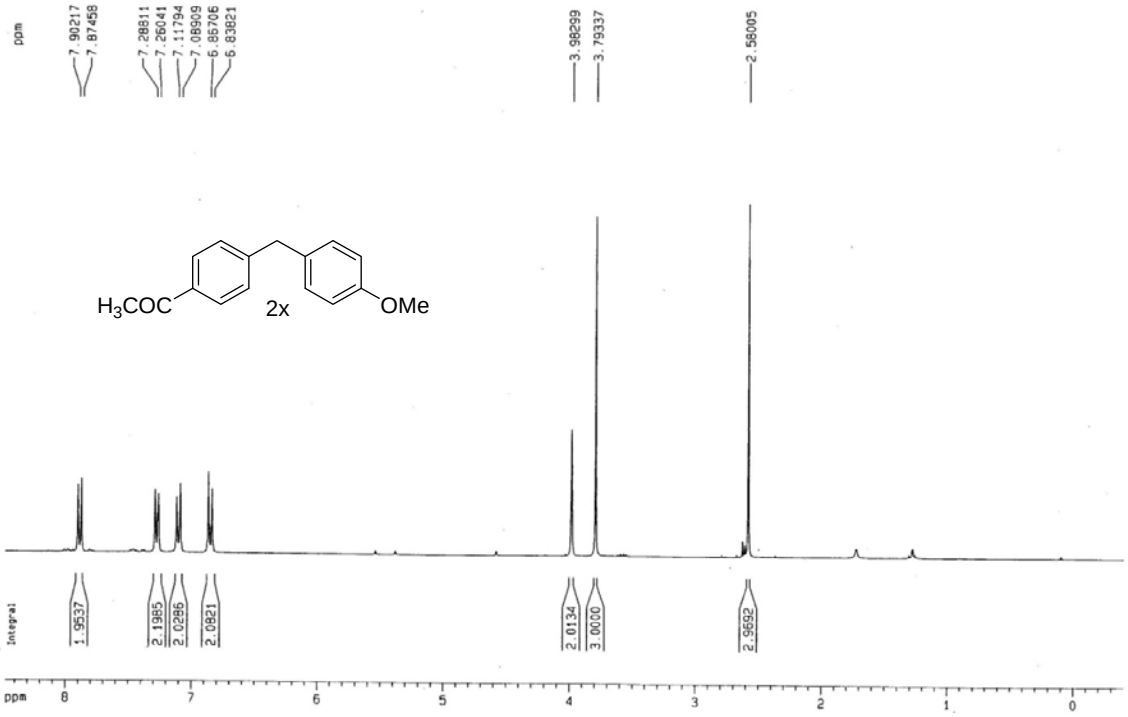
F2 - Acquisition Parameters
Date_        20100421
Time           18.39
INSTRUM        spect
PROBHD   5 mm Multinucl
PULPROG        zgpg30
TD             65536
SOLVENT        CD3CN
NS               340
DS                 4
SWH        18939.395 Hz
FIDRES      0.288992 Hz
AQ         1.7302004 sec
RG            9195.2
DW            26.400 usec
DE             6.00 usec
TE            300.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

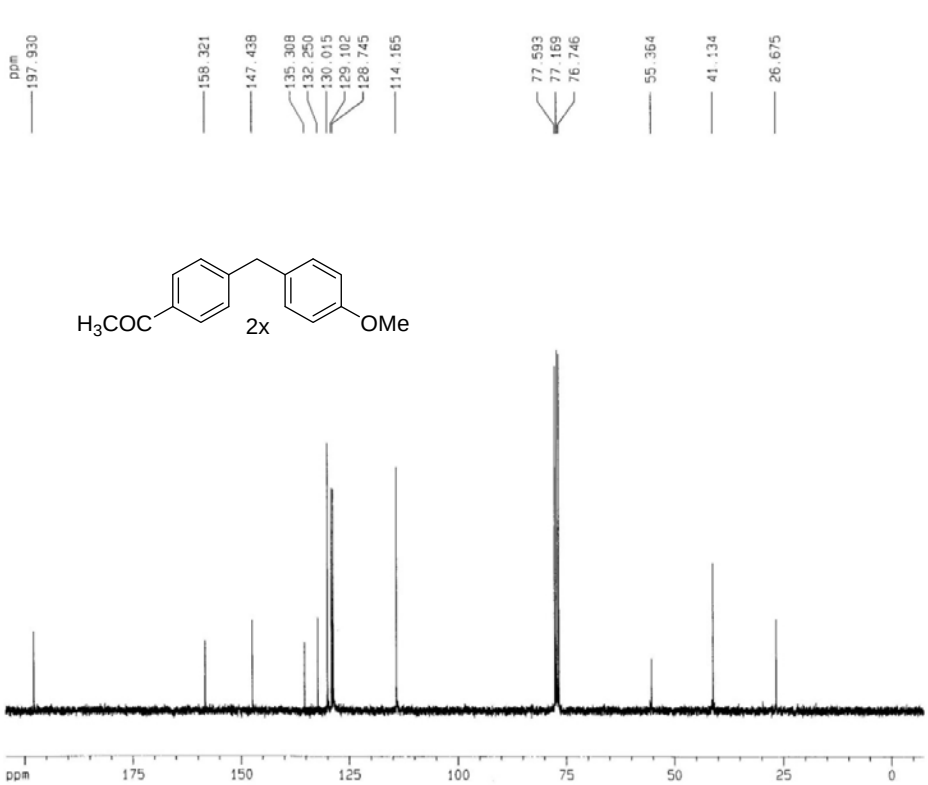
======== CHANNEL f1 ========
NUC1             13C
P1             11.75 usec
PL1             0.00 dB
SFO1       75.4763276 MHz

======== 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       75.4677394 MHz
WDW              EM
SSB               0
LB             1.00 Hz
GB                0
PC             1.40

1D NMR plot parameters
CX            20.00 cm
CY             4.38 cm
F1P          212.380 ppm
F1         16027.82 Hz
F2P         -6.484 ppm
F2          -489.36 Hz
PPMCM      10.94321 ppm/c
HZCM      825.85907 Hz/cm
```






```
Current Data Parameters
NAME        DKSI.MCO-13C
EXPNO                  1
PROCNO                 1

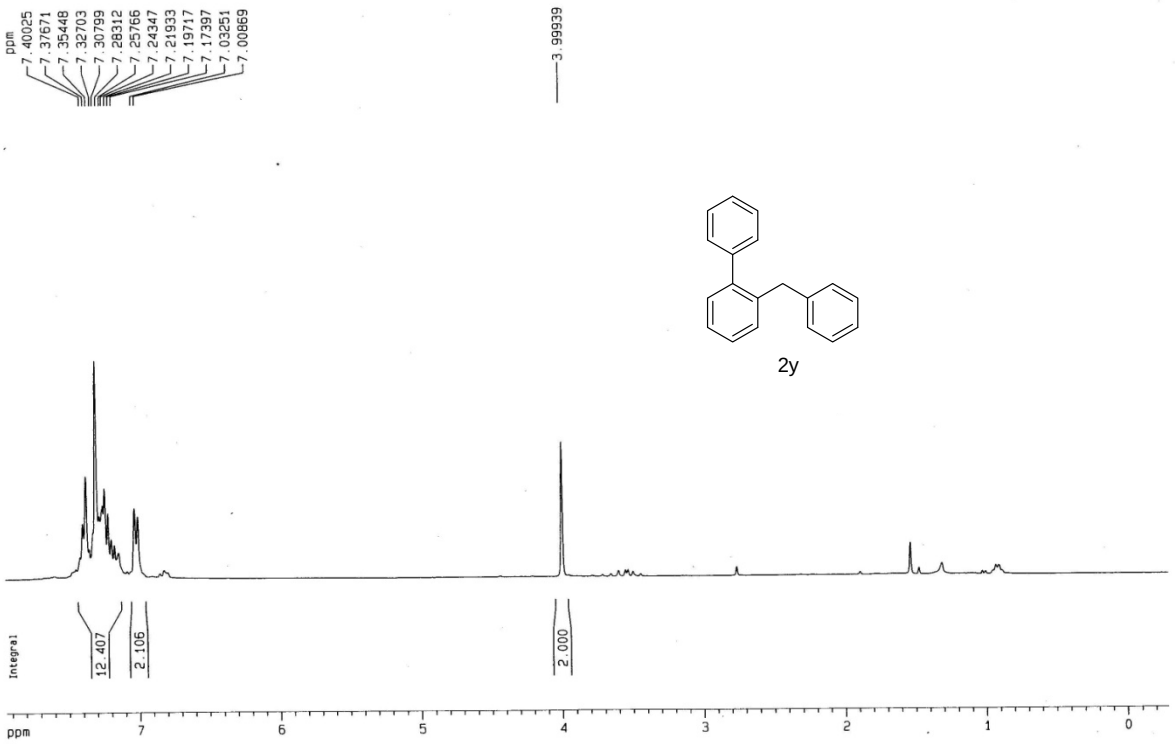
F2 - Acquisition Parameters
Date_           20100420
Time               14.42
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCN
NS                  324
DS                    4
SWH           18939.395 Hz
FIDRES        0.288992 Hz
AQ            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

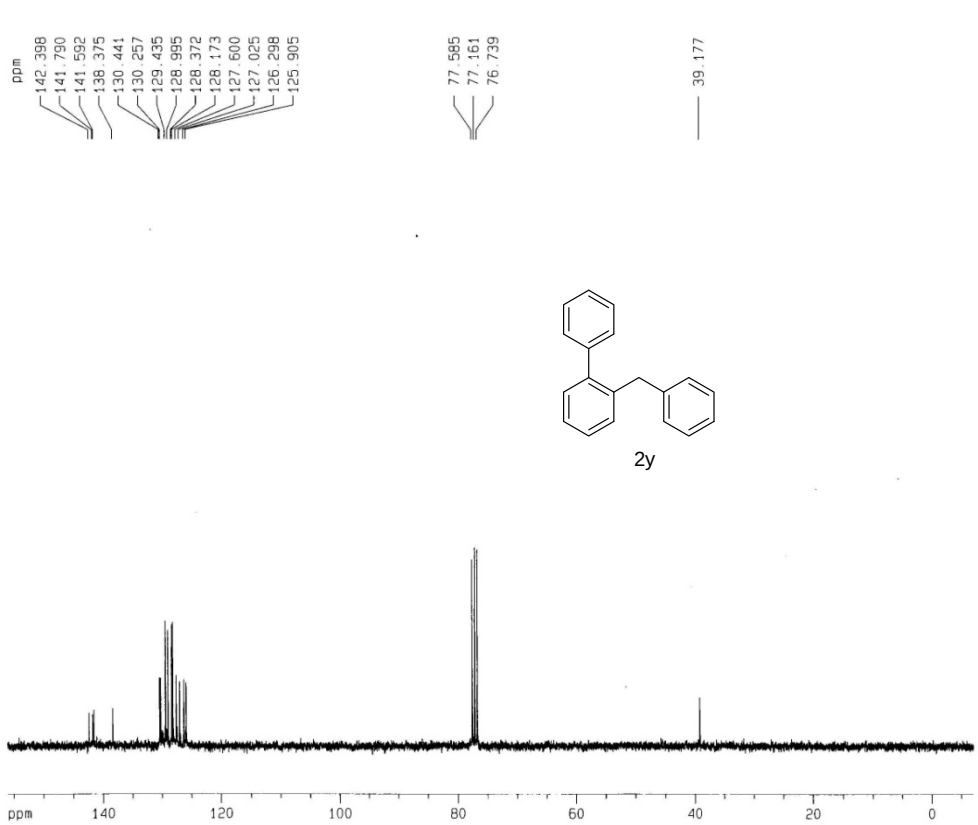
======== CHANNEL f1 ========
NUC1                13C
P1                11.75 usec
PL1                0.00 dB
SFO1         75.4763276 MHz

======== 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                  7.89 cm
F1P               204.417 ppm
F1              15426.88 Hz
F2P                -7.417 ppm
F2               -559.73 Hz
PPMCM           10.59168 ppm/cm
HZCM           799.33020 Hz/cm
```






```
Current Data Parameters
NAME        DKS-I-2PB-13C
EXPNO                   1
PROCNO                  1

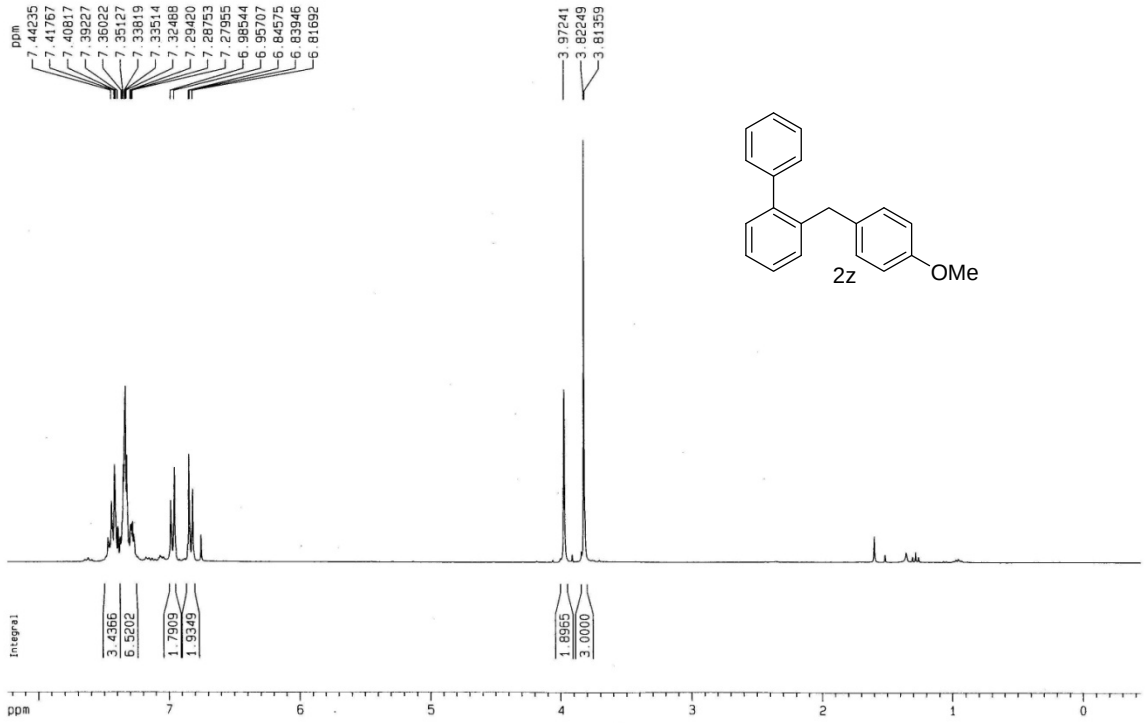
F2 - Acquisition Parameters
Date_            20100416
Time                12.55
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    79
DS                     4
SWH            18939.395 Hz
FIDRES          0.288992 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

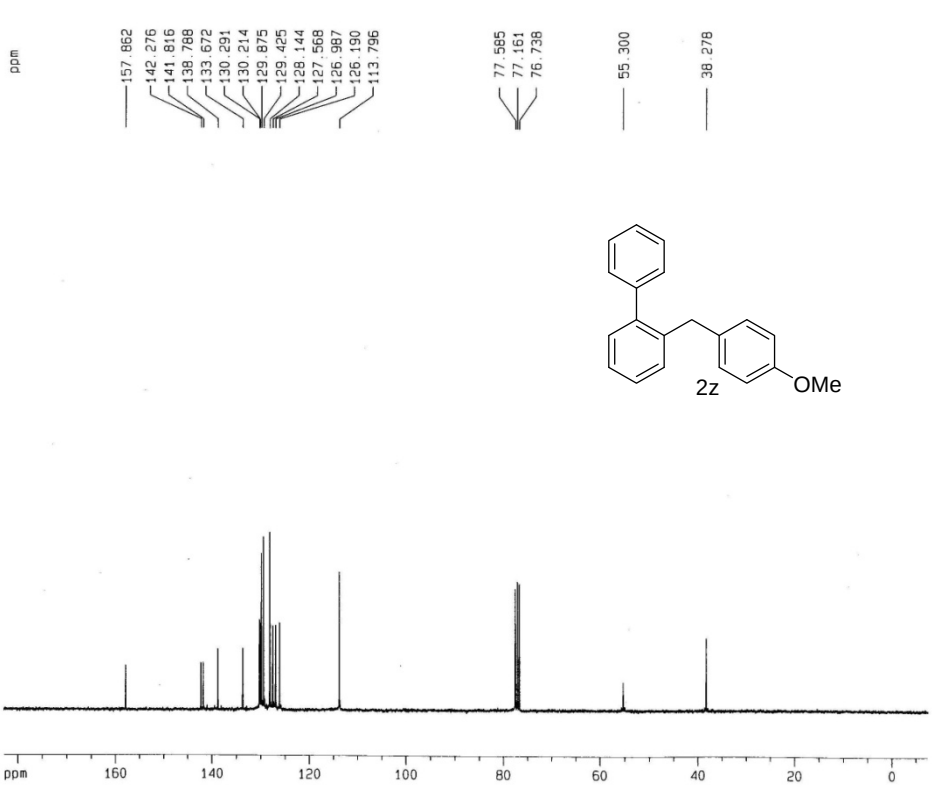
======== CHANNEL f1 ========
NUC1                  13C
P1                  11.75 usec
PL1                  0.00 dB
SFO1           75.4763276 MHz

======== 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             75.4677418 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   4.14 cm
F1P               156.105 ppm
F1               11780.87 Hz
F2P                -7.126 ppm
F2               -537.81 Hz
PPMCM             8.16155 ppm/cm
HZCM            615.93402 Hz/cm
```






```
Current Data Parameters
NAME        DKS-I-SMB-13C
EXPNO               1
PROCNO              1

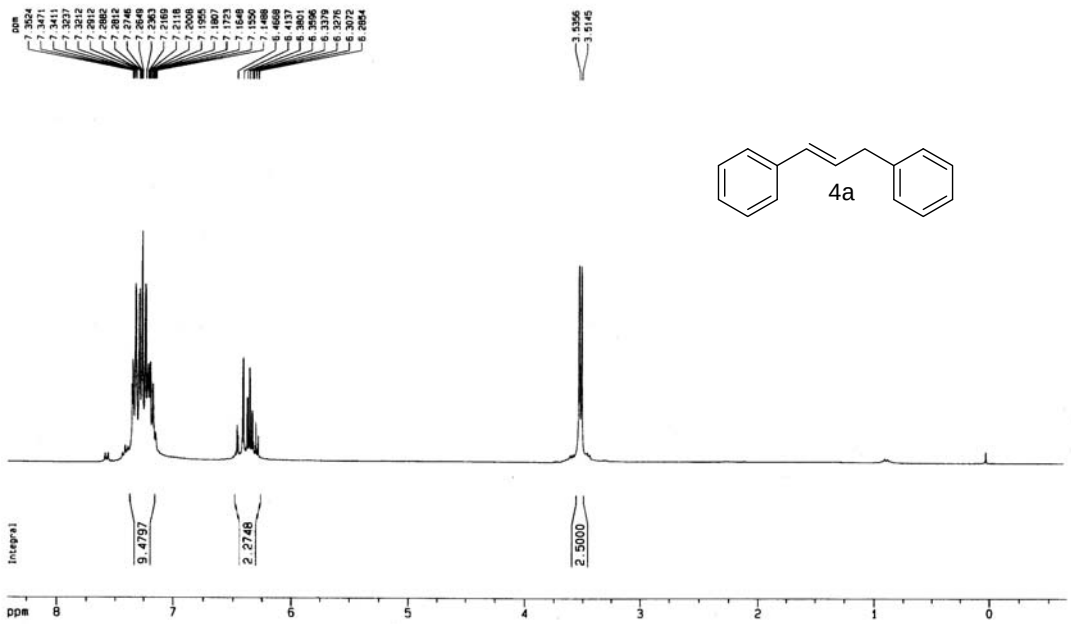
F2 - Acquisition Parameters
Date_          20100419
Time              15.55
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CD3CN
NS                  369
DS                    4
SWH           18939.395 Hz
FIDRES         0.288992 Hz
AQ             1.7302004 sec
RG               9195.2
DW               26.400 usec
DE                6.00 usec
TE               300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

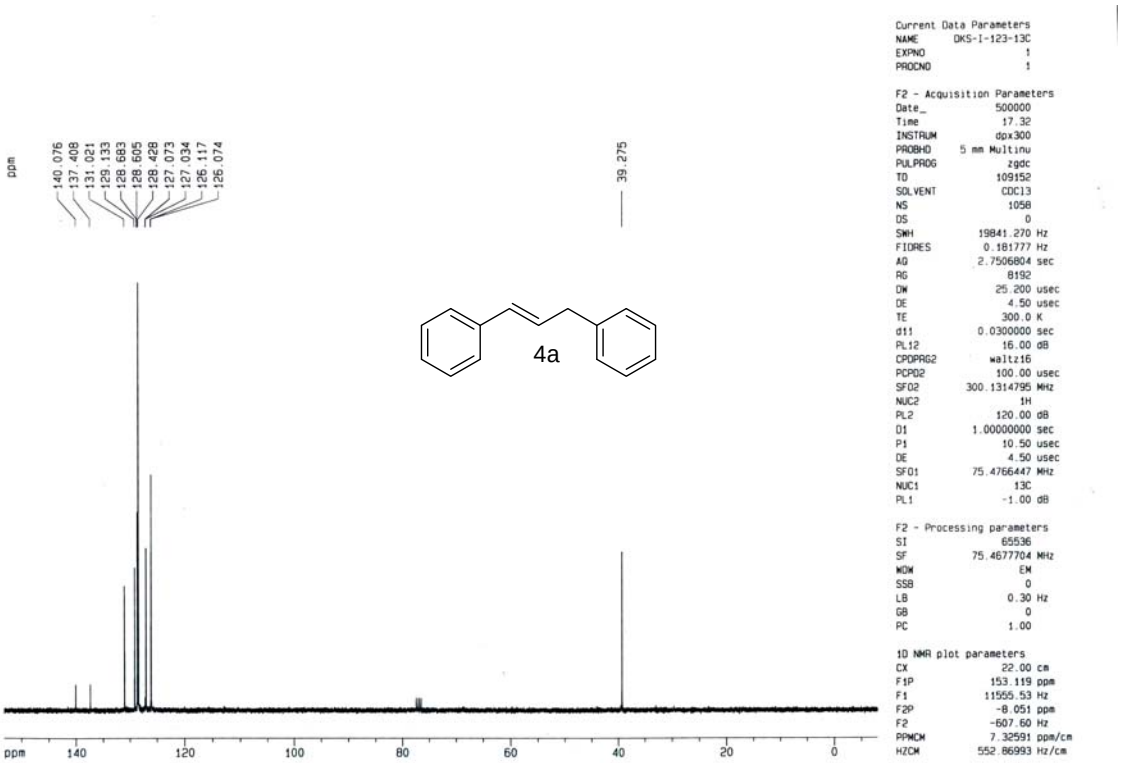
======== CHANNEL f1 ========
NUC1                13C
P1                11.75 usec
PL1                0.00 dB
SFO1          75.4763276 MHz

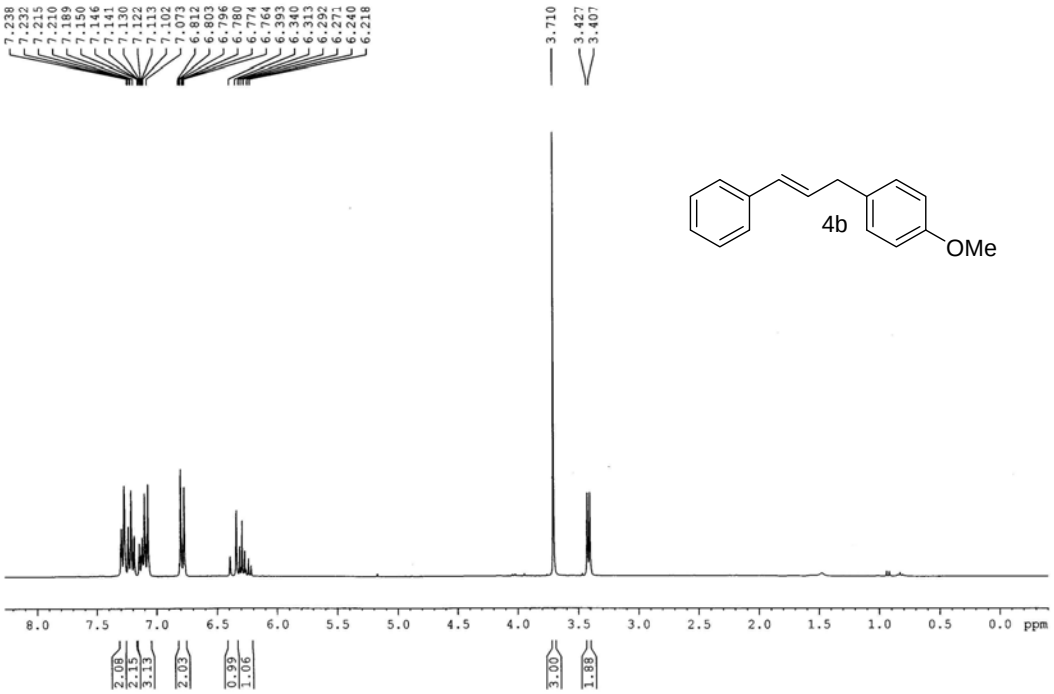
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12               8.00 dB
PL13              12.00 dB
SFO2         300.1312005 MHz

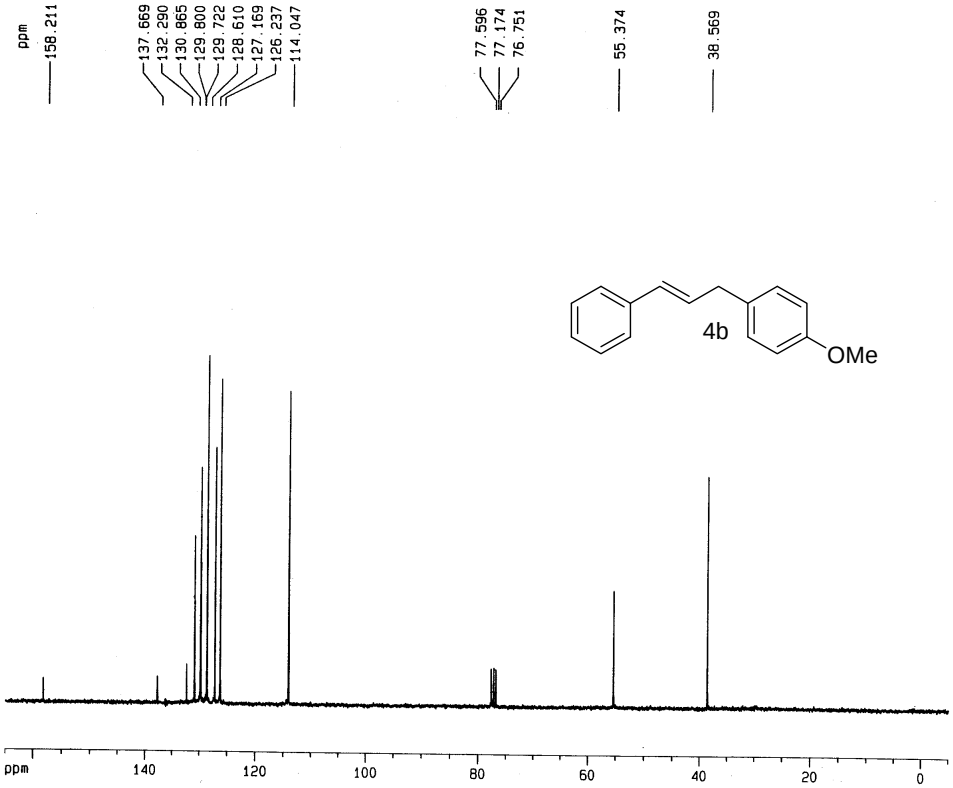
F2 - Processing parameters
SI                 32768
SF            75.4677446 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 3.85 cm
F1P             182.966 ppm
F1            13808.02 Hz
F2P              -7.470 ppm
F2             -563.77 Hz
PPMCM           9.52181 ppm/cm
HZCM          718.58966 Hz/cm
```


ppm
7.3524
7.3471
7.3411
7.3237
7.3212
7.2912
7.2882
7.2812
7.2746
7.2649
7.2383
7.2169
7.2118
7.2008
7.1955
7.1897
7.1723
7.1649
7.1550
7.1488
6.4668
6.4137
6.3861
6.3596
6.3379
6.3276
6.3072
6.2854
3.5356
3.5145
4a
Integral
9.4797
2.2748
2.5000
ppm
8
7
6
5
4
3
2
1
0


ppm
140.076
137.408
131.021
129.133
128.683
128.605
128.428
127.073
127.034
126.117
126.074
39.275
4a
ppm
140
120
100
80
60
40
20
0
Current Data Parameters
NAME DKS-I-123-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 17.32
INSTRUM dpx300
PROBHD 5 mm Multinu
PULPROG zgdc
TD 109152
SOLVENT CDCl3
NS 1058
DS 0
SWH 19841.270 Hz
FIDRES 0.181777 Hz
AQ 2.7506804 sec
RG 8192
DW 25.200 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 16.00 dB
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1314795 MHz
NUC2 1H
PL2 120.00 dB
D1 1.00000000 sec
P1 10.50 usec
DE 4.50 usec
SFO1 75.4766447 MHz
NUC1 13C
PL1 -1.00 dB
F2 - Processing parameters
SI 65536
SF 75.4677704 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
F1P 153.119 ppm
F1 11555.53 Hz
F2P -8.051 ppm
F2 -607.60 Hz
PPMCM 7.32591 ppm/cm
HZCM 552.86993 Hz/cm






```
Current Data Parameters
NAME                 DKSI.MCC-13C
EXPNO                           1
PROCNO                          1

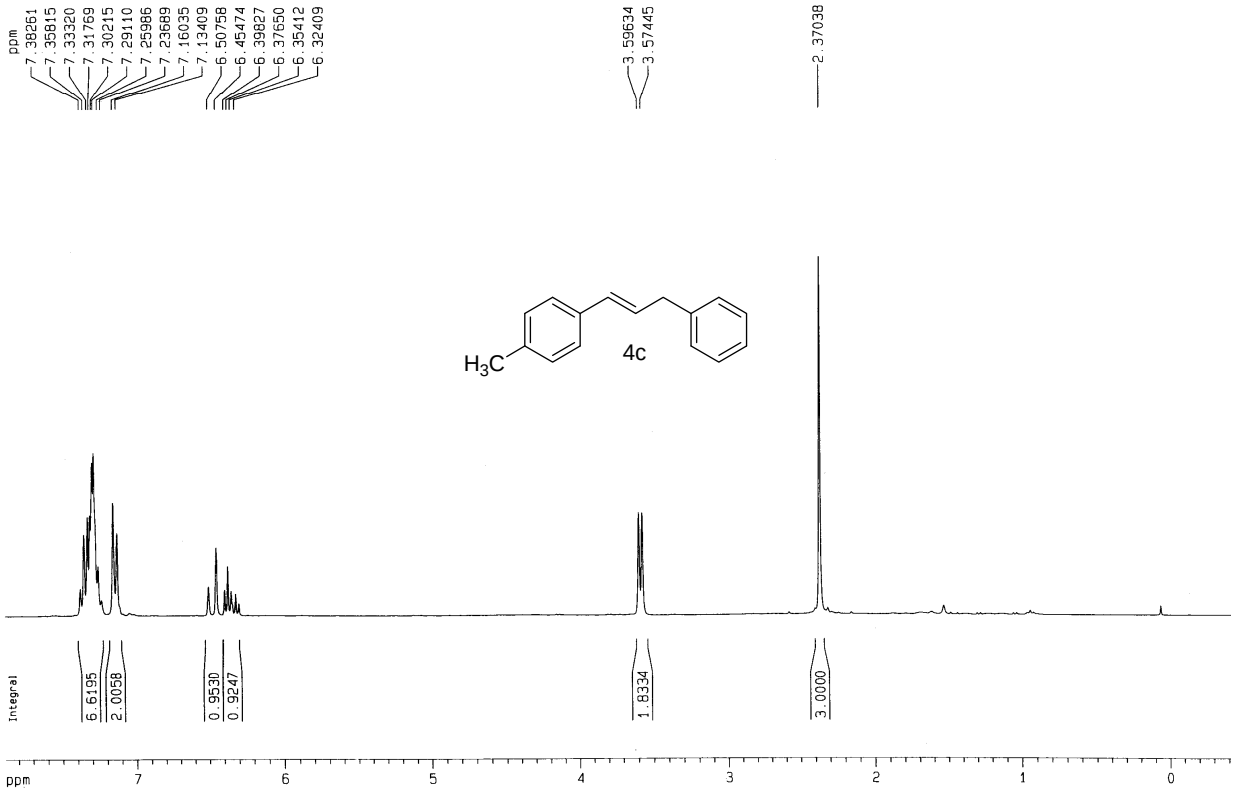
F2 - Acquisition Parameters
Date_             20091218
Time                 14.51
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG              zgdc
TD                   32768
SOLVENT              CDCl3
NS                     146
DS                       4
SWH              17985.611 Hz
FIDRES            0.548877 Hz
AQ                0.9110004 sec
RG                   32768
DW                  27.800 usec
DE                   6.00 usec
TE                  300.2 K
D1               2.00000000 sec
d11              0.03000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

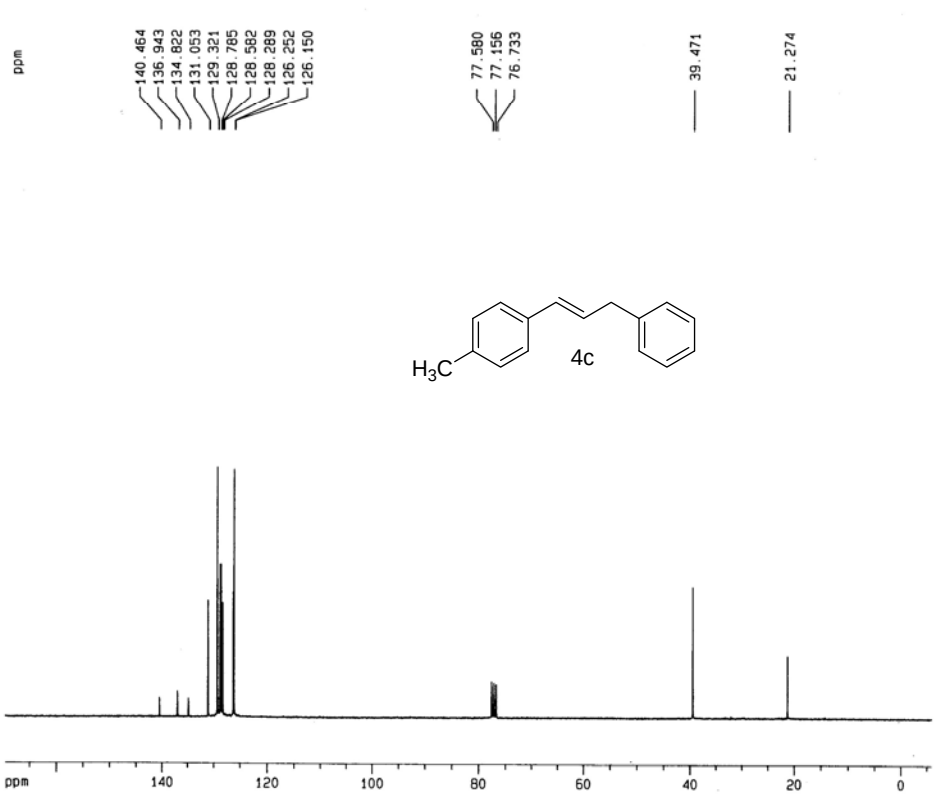
======== CHANNEL f1 ========
NUC1                   13C
P1                   11.75 usec
PL1                   0.00 dB
SFO1            75.4760505 MHz

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

F2 - Processing parameters
SI                    16384
SF               75.4677453 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    7.33 cm
F1P                 165.024 ppm
F1                 12453.97 Hz
F2P                  -5.083 ppm
F2                  -383.58 Hz
PPMCM                8.50533 ppm/cm
HZCM               641.87775 Hz/cm
```






```
Current Data Parameters
NAME        DKS-1-MeCP-13C
EXPNO                    1
PROCNO                   1

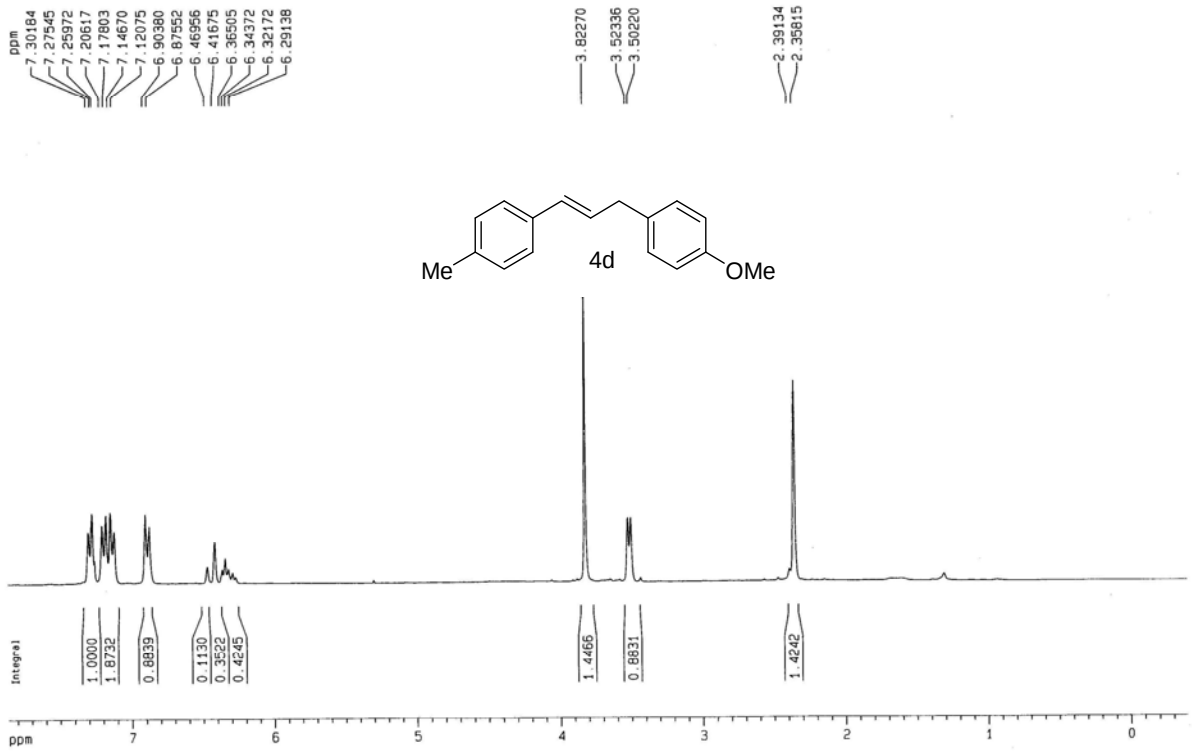
F2 - Acquisition Parameters
Date_            20091229
Time                17.59
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG             zgdc
TD                  32768
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             17985.611 Hz
FIDRES           0.548877 Hz
AQ              0.910004 sec
RG                 10321.3
DW                  27.800 usec
DE                   6.00 usec
TE                  298.2 K
D1             2.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

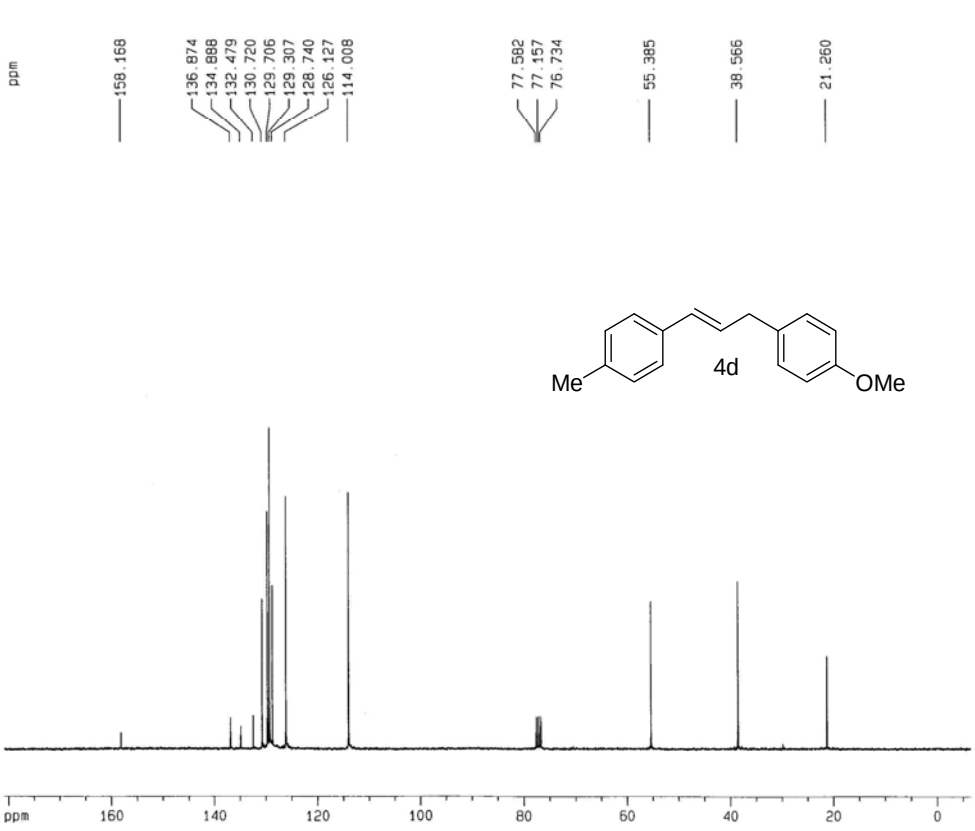
======== CHANNEL f1 ========
NUC1                  13C
P1                  11.75 usec
PL1                  0.00 dB
SFO1            75.4760505 MHz

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

F2 - Processing parameters
SI                   16384
SF              75.4677442 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    5.34 cm
F1P                 169.644 ppm
F1                12802.61 Hz
F2P                  -5.932 ppm
F2                 -447.65 Hz
PPMCM                8.77876 ppm/cm
HZCM               662.51337 Hz/cm
```






```
Current Data Parameters
NAME      DKS-1-MeCM-13C
EXPNO                  1
PROCNO                 1

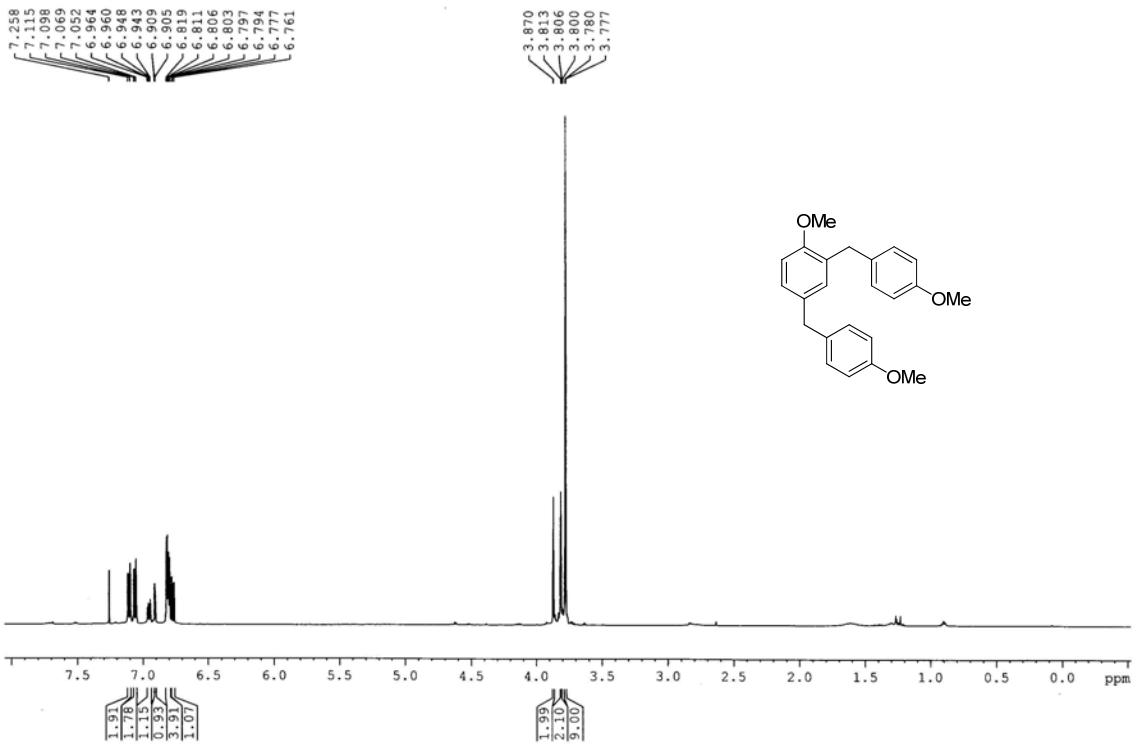
F2 - Acquisition Parameters
Date_           20100111
Time                17.03
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG             zgdc
TD                 32768
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            17985.611 Hz
FIDRES          0.548877 Hz
AQ             0.9110004 sec
RG                 32768
DW                27.800 usec
DE                 6.00 usec
TE                299.2 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

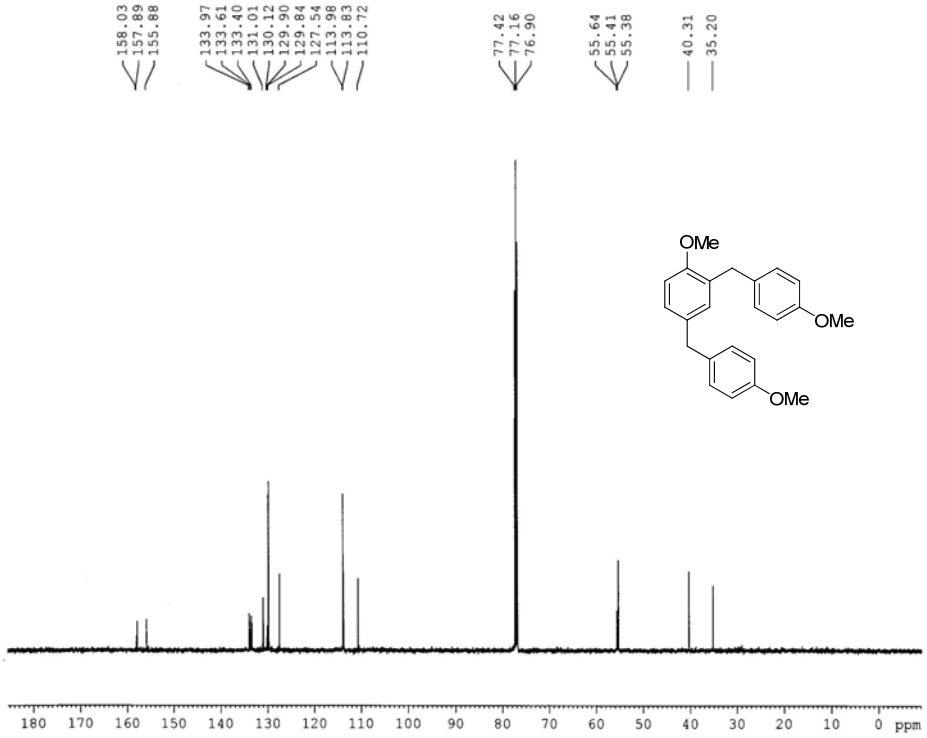
======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1                 0.00 dB
SFO1           75.4760505 MHz

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

F2 - Processing parameters
SI                  16384
SF            75.4677431 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   6.64 cm
F1P               180.883 ppm
F1              13650.86 Hz
F2P                -6.493 ppm
F2               -490.00 Hz
PPMCM             9.36881 ppm/cm
HZCM            707.04303 Hz/cm
```



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TD                  32768
SOLVENT             CDC13
NS                   1024
DS                      4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
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RG                     57
DW                16.800 usec
DE                  6.50 usec
TE                 301.6 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           125.7577697 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
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4. Spectroscopic characterization of the cross-coupling products:

Diphenylmethane (2a): [3] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.05 (s, 2H), 7.25–7.27 (m, 6H), 7.33-7.38 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 42.05, 126.18, 128.57, 129.05, 141.22.

2-Chlorodiphenylmethane (2b): [4] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.21 (s, 2H), 7.25-7.54 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 39.29, 126.36, 126.93, 127.76, 128.59, 129.07, 129.64, 131.13, 134.35, 138.79, 139.61.

3-Chlorodiphenylmethane (2c): [5] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.8(s, 2H), 6.91-6.93 (m, 1H), 7.01-7.09 (m, 6H), 7.13-7.16 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.62, 126.37, 126.43, 127.16, 128.67, 128.98, 129.08, 129.74, 134.31, 140.25, 143.22.

2-Benzyltoluene (2d): [6] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.24 (s, 3H), 3.99 (s, 2H), 7.12–7.21 (m, 7H), 7.24-7.29 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 19.78, 39.57, 126.03, 126.1, 126.57, 128.49, 128.85, 130.06, 130.39, 136.73, 139.03, 140.50.

1-Benzyl-4-methylbenzene (2e): [5] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.54 (s, 3H), 4.19 (s, 2H), 7.26-7.33 (m, 4H), 7.41-7.49 (m, 3H), 7.51-7.59 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.98, 41.5, 125.2, 127.15, 128.79, 128.85, 129.13, 135.51, 138.06, 141.39.

1-Benzyl-3-methoxybenzene (2f):[5] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.80 (s, 3H), 3.99 (s, 2H), 6.78-6.84 (m, 3H), 7.22-7.32 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.93, 55.08, 111.28, 114.77, 121.35, 126.06, 128.42, 128.88, 129.37, 140.88, 142.67, 159.69.

1-Benzyl-4-methoxybenzene (2g):[7] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.81 (s, 3H), 3.93 (s, 2H), 6.79-6.86 (m, 2H), 7.07-7.12 (m, 2H), 7.16-7.20 (m, 3H), 7.26-7.30 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.43, 55.66, 114.28, 126.37, 128.83, 129.21, 129.83, 130.26, 133.66, 158.36.

2-benzylbenzonitrile (2h): [7] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.24 (s, 2H), 7.37 (d, *J*= 8.01 Hz, 2H), 7.48-7.53 (m, 5H), 7.76 (d, *J*= 8.13 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.95, 110.02, 118.96, 126.65, 128.75, 128.94, 129.62, 132.27, 139.32, 146.73

4-Benzyl-1-(trifluoromethyl)benzene (2i):[6] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.09 (s, 2H), 7.23-7.40 (m, 7H), 7.59 (d, *J* = 7.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$):δ 41.69, 124.33 (q, *J* = 270.08 Hz), 125.4 (q, *J* = 3.83 Hz), 126.47, 128.5 (q, *J* = 32.1 Hz), 128.66, 128.93, 129.19, 139.98, 145.22.

1-Benzyl-4-fluorobenzene (2j): [8] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.96 (s, 2H), 6.97 (t, *J* = 8.6 Hz, 2H), 7.12-7.24 (m, 5H), 7.28-7.30 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.15, 115.28 (d, *J*= 21.07 Hz), 126.29, 128.61, 128.91, 130.35 (d, *J*=7.8 Hz), 136.85(d, *J*= 3.08 Hz), 141.02, 161.51(d , *J*= 242.4 Hz).

2-Benzylnaphthalene (2k): [9] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.23 (s, 2H), 7.31-7.41 (m, 6H), 7.51-7.53 (m, 2H), 7.72 (s, 1H), 7.83-7.86 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 42.19, 125.43, 126.06, 126.24, 127.19, 127.64, 127.72, 128.16, 128.58, 129.12, 132.18, 133.70, 138.68, 141.06.

1-Benzylnaphthalene (2l): [5] ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.47 (s, 2H), 7.20-7.22 (m, 3H), 7.26-7.31 (m, 3H), 7.41-7.49 (m, 3H), 7.77 (d, *J* = 8.16 Hz, 1H), 7.86-7.89 (m, 1H), 7.99-8.03 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 38.99, 124.26, 125.52, 125.93, 126.02, 127.12, 127.30, 128.42, 128.64, 128.71, 132.12, 133.92, 136.56, 140.60.

2-*p*-Methylbenzyl naphthalene (2m):[10] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.34 (s, 3H), 4.12 (s, 2H), 7.13- 7.34 (m, 5H), 7.43-7.47 (m, 2H), 7.65-7.80 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.18, 41.85, 125.44, 126.09, 127.15, 127.71, 127.79, 128.20, 129.07, 129.35, 132.23, 133.79, 135.80, 138.09, 139.06.

4-[4-(trifluoromethyl)benzyl]anisole (2n):[6] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.82 (s, 3H), 4.01 (s, 2H), 6.89 (dd, *J* = 6.4 Hz, 2.1Hz, 2H), 7.13 (d, *J* = 8.6 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 7.57 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 40.93, 55.32, 114.19, 124.49 (q, *J* = 270.2 Hz), 125.47 (q, *J* = 3.75 Hz), 128.48 (q, *J* = 32.17 Hz), 129.19, 130.03, 132.20, 145.87, 158.39.

1-(4-fluorobenzyl)-4-methoxybenzene (2o):[11] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.81 (s, 3 H), 3.92 (s, 2 H), 6.86 – 6.89 (m, 2 H), 6.96 – 7.02 (m, 2 H), 7.10 – 7.17 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 40.29, 55.34, 114.06, 115.27(d, *J*= 20.93Hz), 129.89, 130.26(d, *J*= 7.7Hz), 133.17, 137.341, 158.18, 161.49 (d, *J*= 242.33 Hz).

1-(2-chlorobenzyl)-4-methoxybenzene (2p):[11] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.79 (s, 3 H), 4.06 (s, 2 H), 6.84 – 6.87 (m, 2 H), 7.13 – 7.19 (m, 5 H), 7.37 – 7.39 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 38.45, 55.36, 114.02, 126.94, 127.67, 129.63, 130.05, 131.01, 131.68, 134.26, 139.25, 158.19.

1-(3-chlorobenzyl)-4-methoxybenzene (2q): ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.80 (s, 3H), 3.90 (s, 2H), 6.86 (dd, *J*=6.6 Hz, 2.02 Hz, 2H), 7.06-7.12 (m, 3H), 7.17-7.22 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 40.79, 55.39, 114.14, 126.33, 127.10, 129.02, 129.78, 130.01, 132.42, 134.35, 143.78, 158.29

5-Benzyl-2-chloropyridine (2r):[12] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.95 (s, 2H), 7.14 7.33 (m, 6H), 7.42 (dd, *J* =8.17, 2.46 Hz, 1H), 8.25 (d, *J* = 2.25 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 38.17, 124.07, 126.69, 128.76, 128.78, 135.50, 139.16, 139.27, 149.29, 149.69.

4-Benzyl-Acetophenone (2s):[13] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.51 (s, 3H), 3.98 (s, 2H), 7.12-7.19 (m, 3H), 7.21-7.28 (m, 4H), 7.83 (d, *J*=8.28 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.59, 41.94, 126.46, 128.69,128.99, 129.15, 135.29, 140.09, 146.85, 197.79.

4-Benzyl-Benzamide (2t): ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.05(s, 2H), 5.99(br.s, 2H), 7.18-7.24(m, 2H), 7.28-7.35(m, 5H), 7.76(d, *J*=8.08 Hz, 2H) ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.92, 126.53, 127.74, 128.76, 129.07, 129.31, 131.32, 140.29, 145.71, 169.39.

1-Benzyl-4-nitrobenzene (2u): [8] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 4.08 (s, 2H), 7.18 (d, *J* = 7.44, 2H), 7.25-7.35 (m, 5H), 8.14 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ: 41.85, 123.87, 126.87, 128.94, 129.08, 129.77, 139.31, 146.66, 148.99,

Methyl 4-Benzylbenzoate (2v): [14] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 3.91 (s, 3H), 4.04(s, 2H), 7.19 (d, *J* = 7.14 Hz, 2H), 7.24–7.34 (m, 5H), 7.99 (d, *J* = 8.17 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ: 41.99, 52.06,126.46, 128.19, 128.69, 129.03, 129.91, 140.21, 146.61, 167.12.

4–Benzylbenzaldehyde (2w): [13] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 4.06 (s, 2H), 7.18-7.37(m, 7H), 7.81(d, J= 7.8 Hz, 2H), 9.97 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ:192.13, 148.58, 139.89, 134.82, 130.18, 129.70, 129.19, 129.11, 126.66, 42.23.

1-(4-(4-methoxybenzyl)phenyl)ethanone (2x):[15] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 2.58 (s, 3H), 3.79 (s, 3H), 3.98 (s, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 7.10 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ:26.5, 40.9, 55.2, 113.9, 128.6, 128.9, 129.8, 132.1, 135.1, 147.3, 158.1, 197.8.

2-Benzylbiphenyl (2y): [14] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 3.99 (s, 2H), 7.02 (d, *J* = 7.15, 2H), 7.17–7.40 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$): δ:39.18, 125.91, 126.29, 127.03, 127.60, 128.17, 128.37, 128.99, 129.44, 130.26, 130.44, 138.38, 141.59, 141.79, 142.39.

4-(2-Phenylbenzyl)anisole (2z): [6] ^{1}H NMR (300 MHz, $CDCl_3$): δ: 3.82 (s, 3H), 3.97(s, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 6.96 (d, *J* =8.6 Hz, 2H), 7.27–7.44 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ:38.28, 55.3, 113.79, 126.19,126.98, 127.57, 128.14, 129.43, 129.88, 130.21, 130.29, 133.67, 138.79, 141.81, 142.28, 157.86.

(E)-1, 3-diphenylprop-1-ene(4a): [5] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.52 (d, *J*=6.33 Hz, 2H), 6.29-6.47 (m, 2H), 7.15-7.35 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 39.28, 126.07, 126.12, 127.03, 128.43, 128.61, 129.13, 131.02, 137.41, 140.08.

(*E*)-3-(4-Methoxyphenyl)-1-phenylpropene (**4b**): [16] ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.42 (d, *J*=6.18 Hz, 2H), 3.71 (s, 3H), 6.21-6.31 (m, 1H), 6.36 (d, 1H, *J*=15.88 Hz), 6.77-6.81 (m, 2H), 7.07-7.15 (m, 3H), 7.19-7.24 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 38.57, 55.37, 114.05, 126.24, 127.17, 128.61, 129.72, 129.80, 130.87, 132.29, 137.67, 158.21.

1-methyl-4-(E)-3-phenylpro-1-enyl) benzene (4c): [17] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.37 (s, 3H), 3.58 (d, J = 6.57 Hz, 2H), 6.32-6.39 (m, 1H), 6.48 (d, *J* = 15.8 Hz, 1H),7.14 (d, *J*= 7.9 Hz, 2H), 7.23-7.38 (m, 7H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.27, 39.47, 126.15, 126.25, 128.29, 128.58, 128.79, 129.32, 131.05, 134.82, 136.94, 140.46.

1-((*E*)-3-(4-methoxyphenyl)prop-1-enyl)-4-methybenzene(4d): [17] ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.36 (s, 3H), 3.51 (d, *J* = 6.35 Hz, 2H), 3.82 (s, 3H), 6.29-6.36 (m, 1H), 6.44 (d, *J* = 15.84 Hz, 1H), 6.89 (d, *J* = 8.48 Hz, 2H), 7.13 (d, *J* = 7.79 Hz, 2H), 7.19 (d, *J* = 8.44 Hz, 2H), 7.29 (d, *J* = 7.91 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.26, 38.57, 55.39, 114.01, 126.13, 128.74, 129.31, 129.71, 130.72, 132.48, 134.89, 136.87, 158.17.

1-Methoxy-2,4-bis(4-methoxy-benzyl)-benzene: [18] ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.78 (s, 9H), 3.81 (s, 2H), 3.87 (s, 2H), 6.77 (d, *J* = 8.0 Hz, 1H,), 6.79-6.82 (m, 4H), 6.91 (d, *J* = 2.0 Hz, 1H), 6.95 (dd, *J* = 8.5, 2.5 Hz, 1H), 7.05 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 35.20, 40.31, 55.38, 55.41, 55.64, 110.72, 113.83, 113.98, 127.54, 129.84, 129.90, 130.12, 131.01, 133.40, 133.61, 133.97, 155.88, 157.89, 158.03.

5. References:

1. Perrin, D. D.; Armarego, W. L. F. *Purification of Laboratory Chemicals*, 3rd ed; Butterworth- Heinemann: Oxford, 1988.
2. Manoso, A. S.; Ahn, C.; Soheili, A.; Handy, C.J.; Correia, R.; Seganish, W. M.; DeShong, P. *J. Org. Chem.* **2004**, *69*, 8305-8314.
3. Srimani, D; Sarkar A.;*Tetrahedron Lett.* **2008**, *49*, 6304.
4. de Lang R,-J.; van Hooijdonk, M. J.C.M.; Brandsma L. *Tetrahedron* **1998**, *54* 2953.
5. Ebert, G. W.; Pfennig, D. R.; Suchan, S. D.; Donovan Jr. T. A., Aouad, E.; Tehrani, S. S., Gunnersen J. **N.**; Dong, L. *J. Org. Chem.* **1995,** *60***,** 2361.
6. Kuwano, R.; Yokogi. M. *Chem. Commun.*, **2005**, 5899.
7. Amator, M.; Gosmini, C. *Chem. Commun.* **2008**, 5019.
8. Burns, M. J.; Fairlamb, I. J. S.; Kapdi, A. R.; Sehnal, P.; Taylor, R. J. K. *Org. Lett.* **2007**, *9*, 5397.
9. Cho C,-H.; Sun M.; Seo Y,-S.; Kim C,-B.; Park K. *J. Org. Chem.* **2005**, *70*, 1482.
10. Giannangeli, N.; Baiocchi, L. *Tetrahedron* **1980**, *36*, 1381.
11. Sun, G.; Wang Z. *Tetrahedron Lett.* **2008,** 49, 4929.
12. Alacid E.; Nájera C. *Org. Lett.* **2008**, *10*, 5011.
13. Molander, G. A.; Elia. M. D. *J. Org. Chem.* **2006**, *71*, 9198.
14. Kuwano, R.; Yokogi M.*Org. Lett.* **2005**, *7*, 945.
15. Itami, K.; Mineno, M.; Kamei, T.; Yoshida J. *Org. Lett.* **2002**, *4*, 3635.
16. Narahashi, H.; Shimizu, I.; Yamamoto, A. *J. Organomet. Chem.* **2008,** *693*, 283.
17. Tsukamoto, H.; Uchiyama T.; Suzuki T.; Kondo Y. *Org. Biomol. Chem.*, **2008***, 6*, 3005.
18. Flaherty, A.; Trunkfield, A.; Barton W. *Org. Lett.* **2005**, *7*, 4975.