Electronic Supplementary Material (ESI) for RSC Advances

Solvent free, Phosphine free Pd-Catalyzed Annulations of Aryl Bromides with Diarylacetylenes

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General Information

All chemicals were purchased from commercial suppliers (Aldrich, Merck, Avra and SRL) and used as received. All 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, ^{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). Electrospray ionisation mass spectroscopy (ESI-MS) experiments were carried out on Microtek QtoF Micro YA 263 spectrometer in positive ion ESI mode. Transmission electron microscopy (TEM): TEM pictures were taken on a JEM-2011 (JEOL) microscope.

General Procedure for the Pd-Catalyzed Annulation Reaction:

Weighted amount of K_2PdCl_4 (16 mg, 5 mol%) and PEG 600 (700 mg, 1.16 mmol) was heated at 70 °C for 15 min to form colloidal Pd. Then it was cooled at room temperature, aryl bromide (1 mmol) and diphenylacetylene (2 mmol) and NaOAc (182 mg, 2 eq) were added then stirred at 80 °C for the specified time. Then the reaction mixture was allowed to cool at room temperature, extracted with dichloromethane 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 a mixture of ethyl acetate (2-10%) and petroleum ether as eluent.

Preparation of Pd nanoparticles

Weighted amount of K_2PdCl_4 (16 mg, 5 mol%) and PEG 600 (700 mg, 1.16 mmol) was heated at 70 °C for 15 min to form colloidal Pd.

The nanoparticle formed is characterized by TEM (Figure 1). The average size of the nanoparticles before and after the reaction was 3.8 and 3.4 nm respectively.

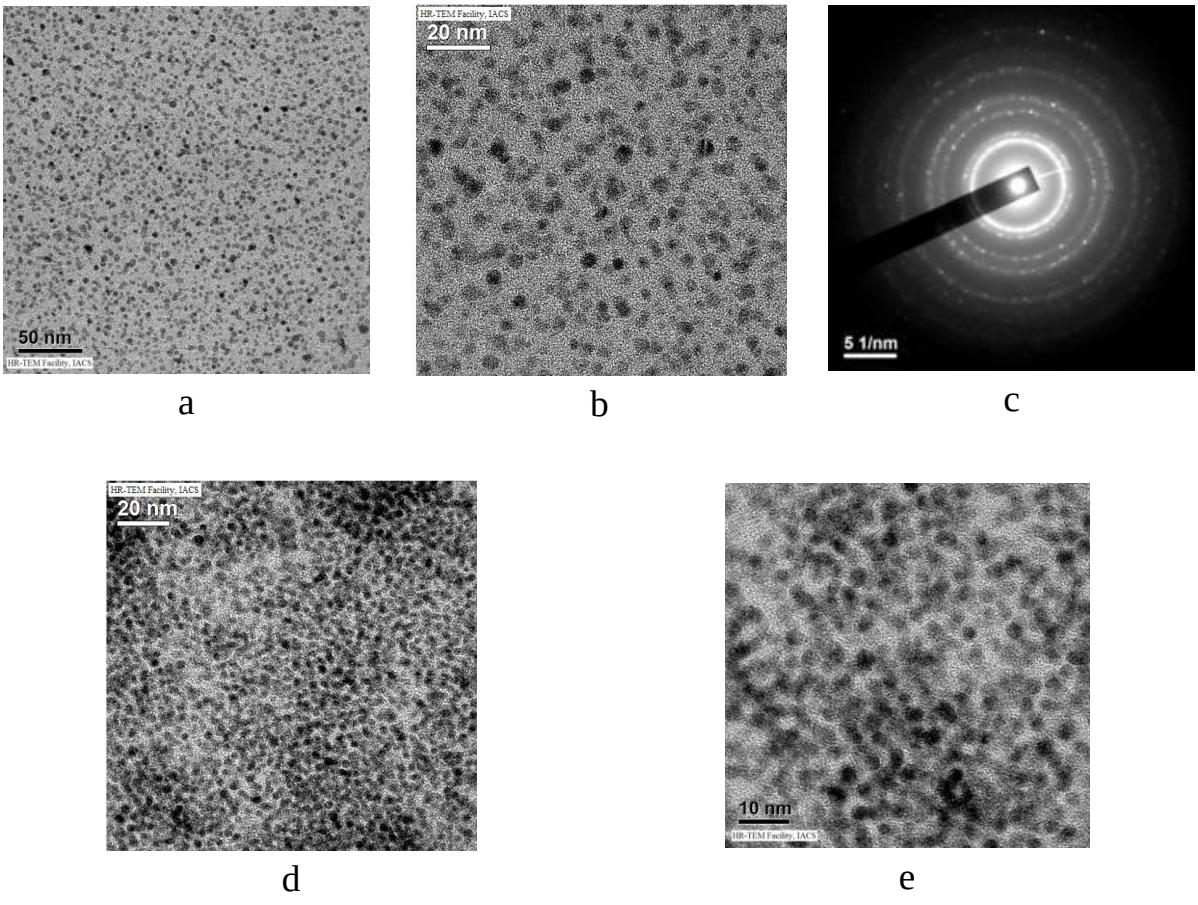
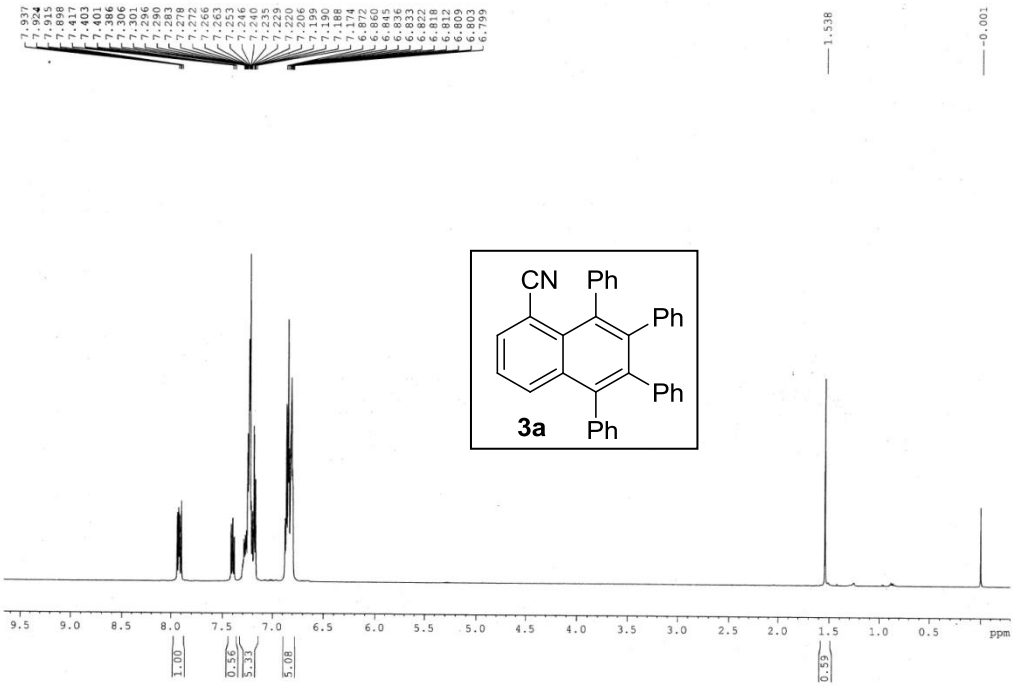
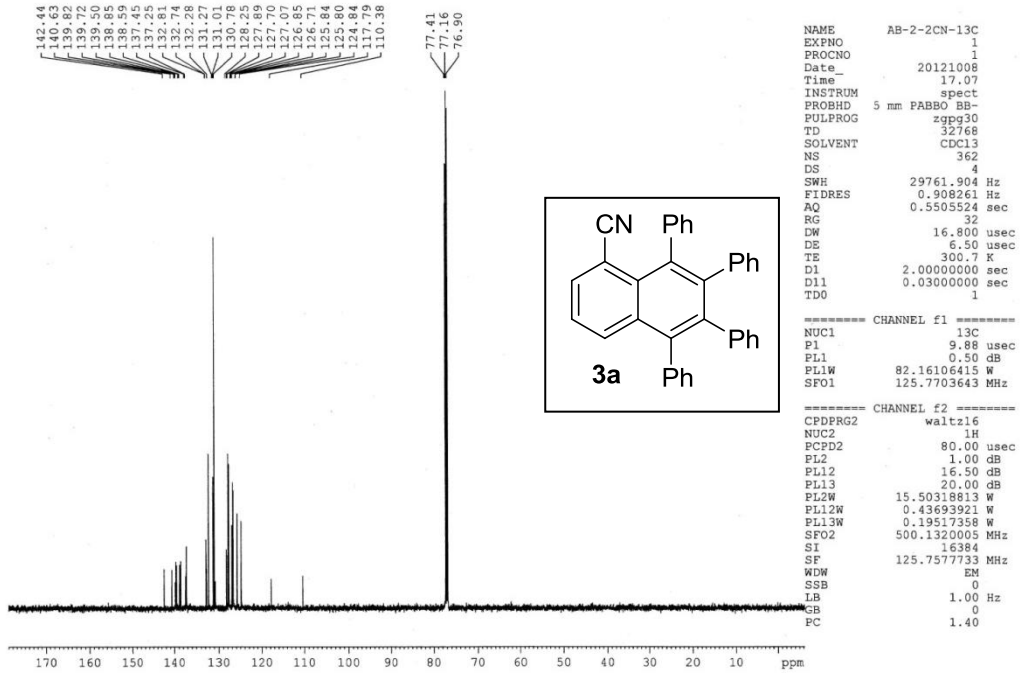


Figure 1. (a) TEM image of Palladium nanoparticles, (b) HRTEM image of Palladium nanoparticles, (c) Electron diffraction pattern of palladium nanoparticles, (d) TEM image of palladium nanoparticles after reaction, (e) HRTEM image of palladium nanoparticles after reaction.

^{1}H and ^{13}C NMR Spectrum for all Compounds





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FIDRES	0.908261 Hz
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DE	6.50 usec
TE	300.7 K
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D11	0.03000000 sec
TD0	1
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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
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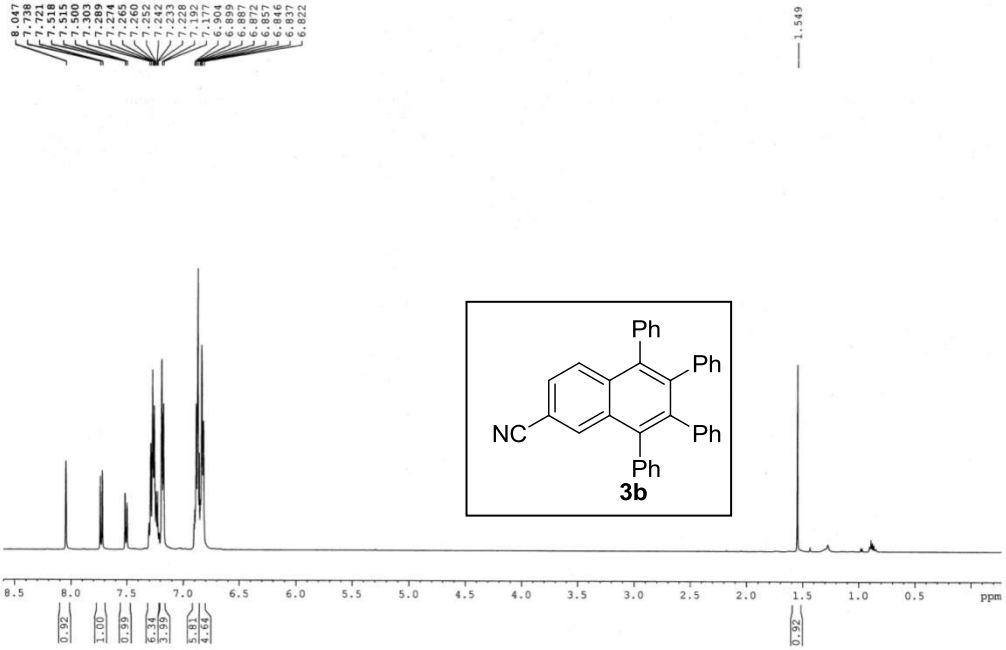

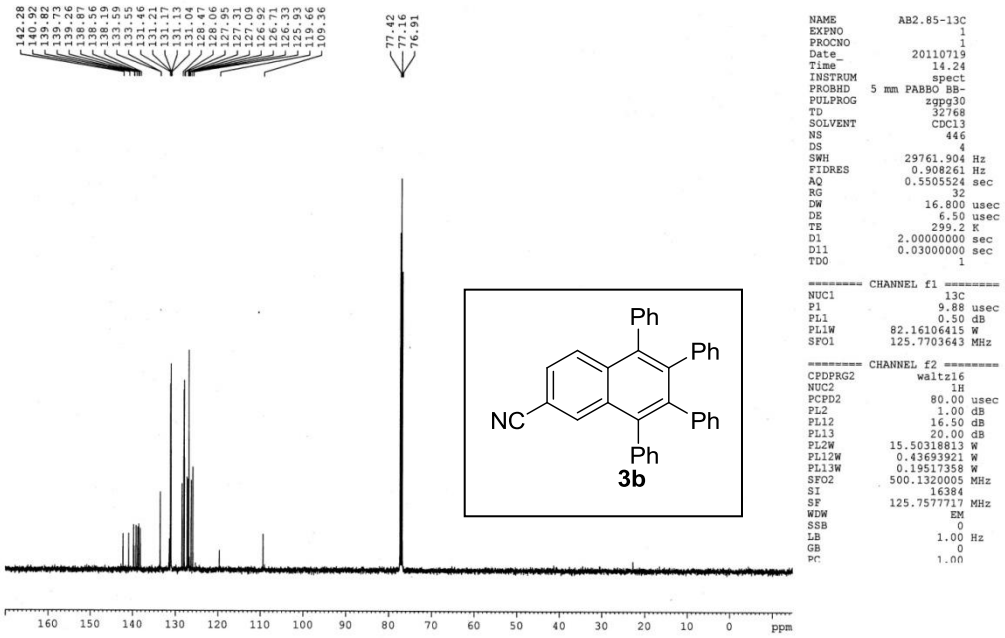

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NUC1	13C
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PL1	0.50 dB
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SFO1	125.7703643 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
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PL12	16.50 dB
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PL13W	0.19517358 W
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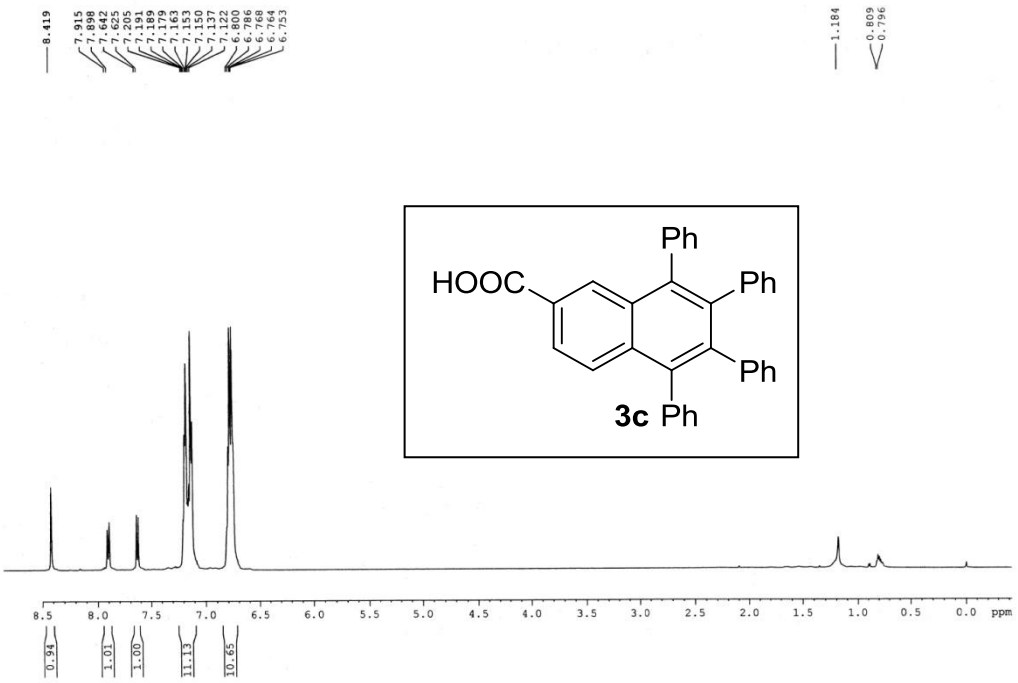

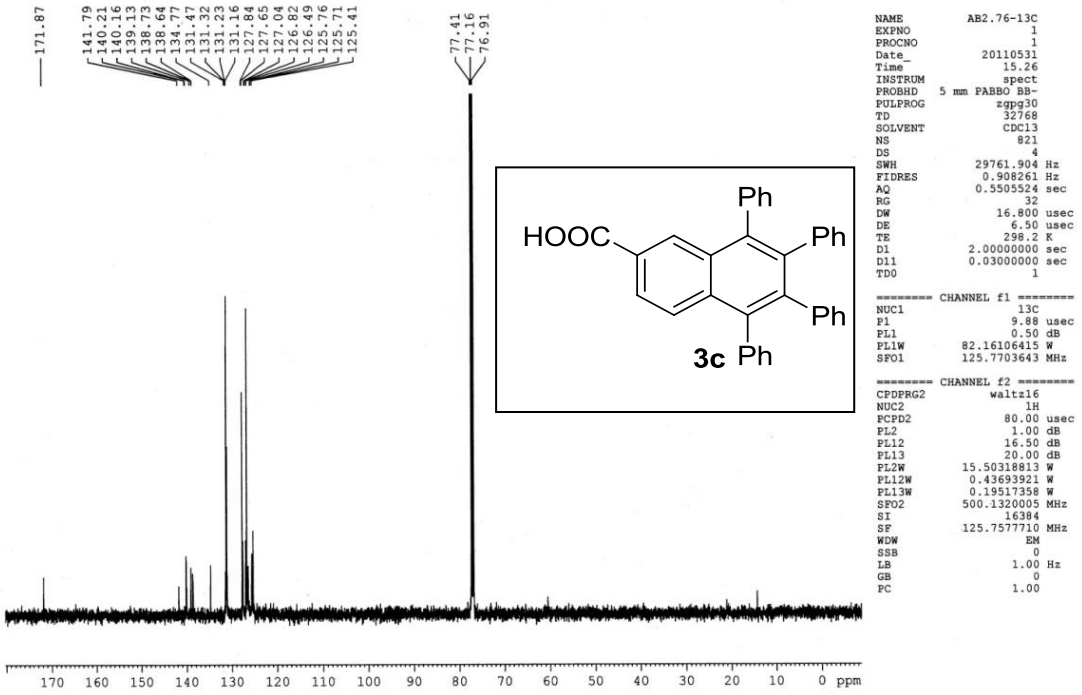

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FIDRES	0.908261 Hz
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RG	32
DW	16.800 usec
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TE	298.2 K
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D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.88 usec
PL1	0.50 dB
PL1W	82.16106415 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
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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
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SI	16384
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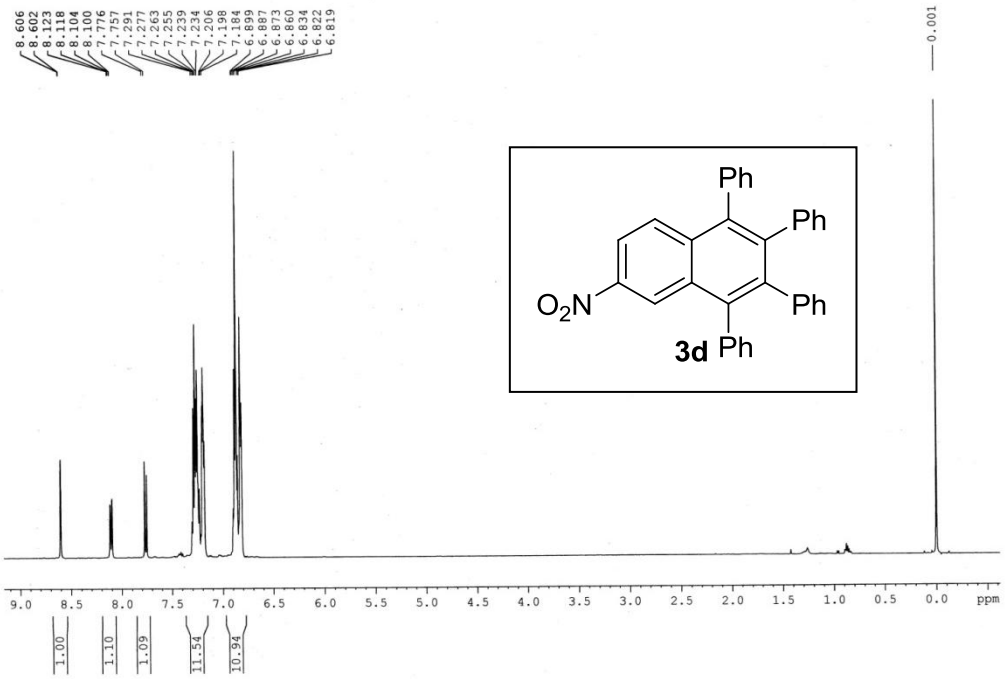

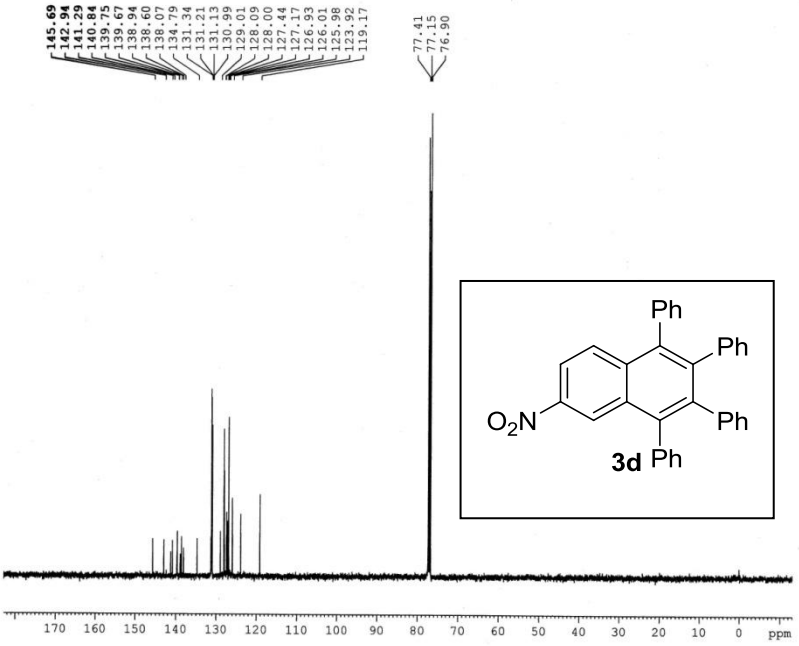

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SOLVENT	CDCl3
NS	563
DS	4
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FIDRES	1.105709 Hz
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RG	32
DW	13.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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NUC1	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
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SI	16384
SF	125.7577715 MHz
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SSB	0
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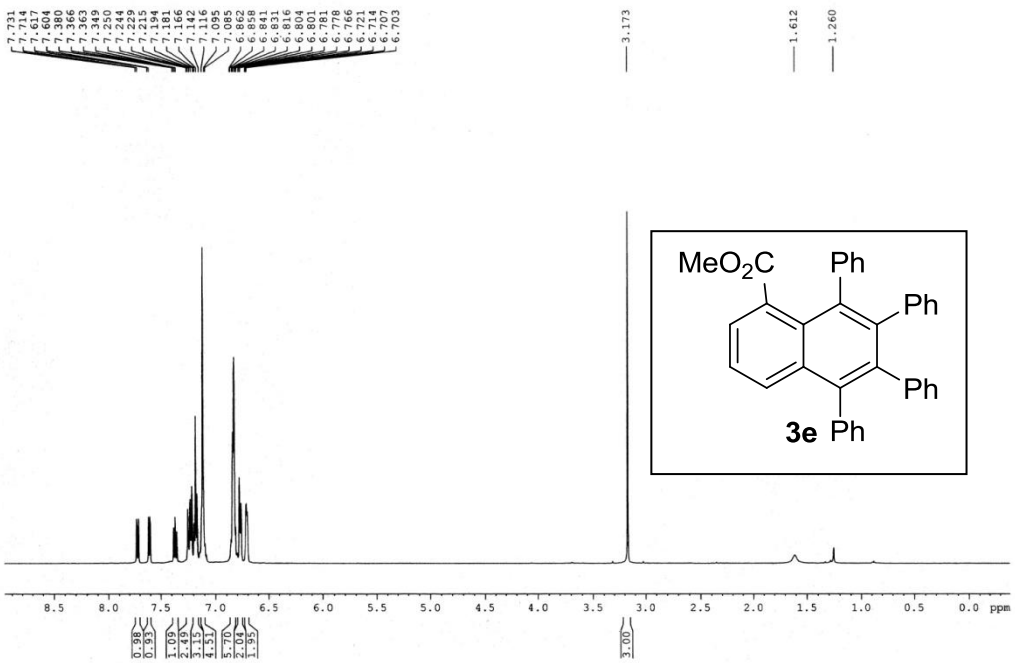

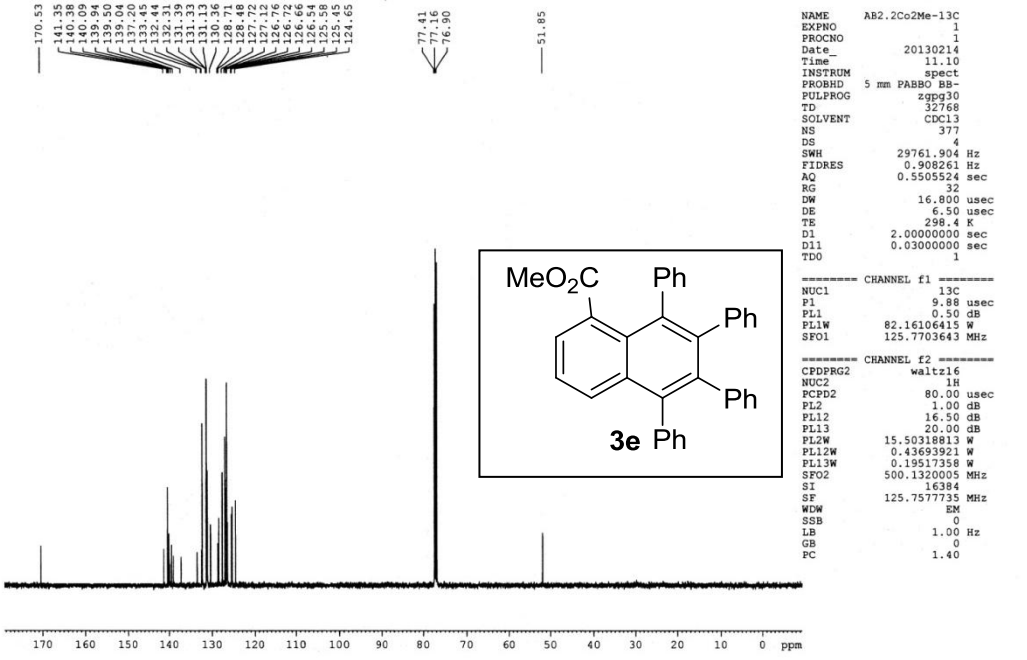

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RG	32
DW	16.800 usec
DE	6.50 usec
TE	298.4 K
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TD0	1
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NUC1	13C
P1	9.88 usec
PL1	0.50 dB
PL1W	82.16106415 W
SFO1	125.7703643 MHz
======== CHANNEL 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
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SI	16384
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LB	1.00 Hz
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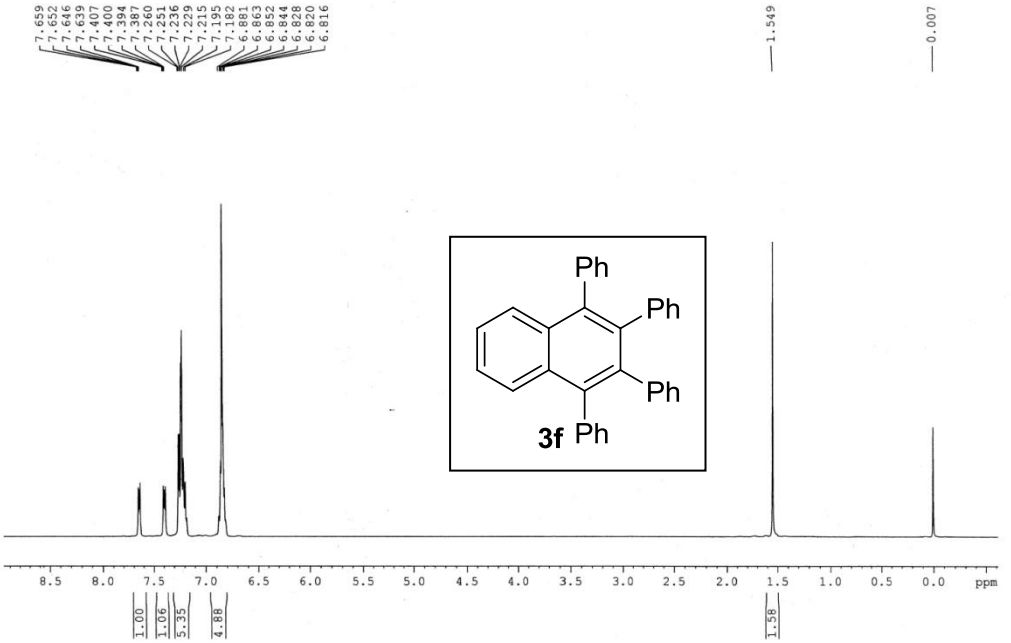
PC	1.40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Ph
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3f
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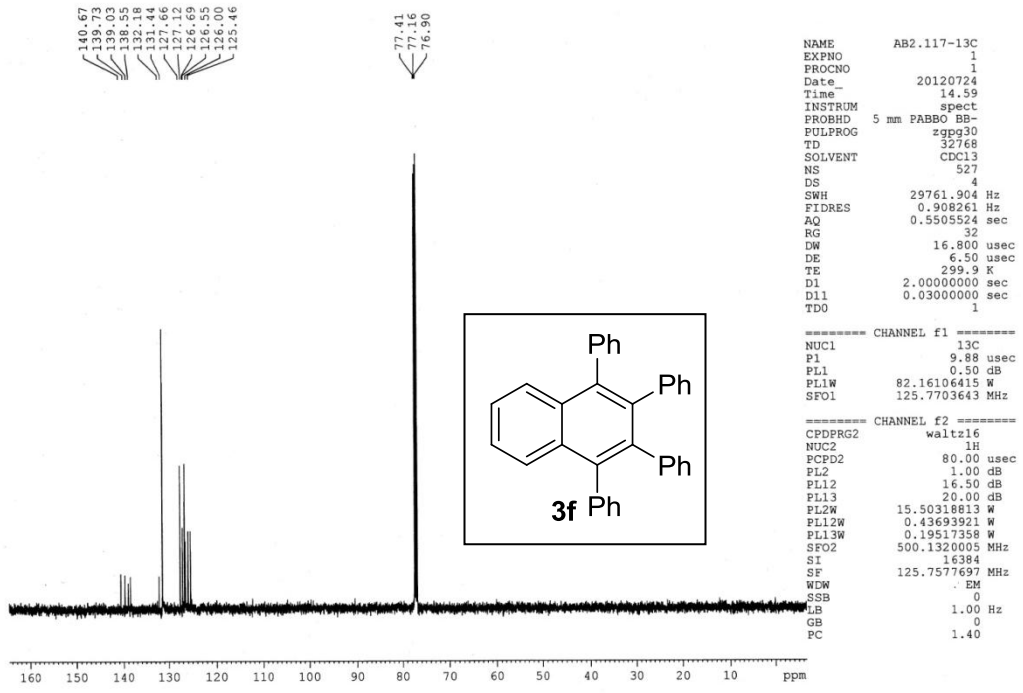
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PROCNO 1
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SOLVENT CDCl3
NS 527
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FIDRES 0.908261 Hz
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RG 32
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DE 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
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SSB 0
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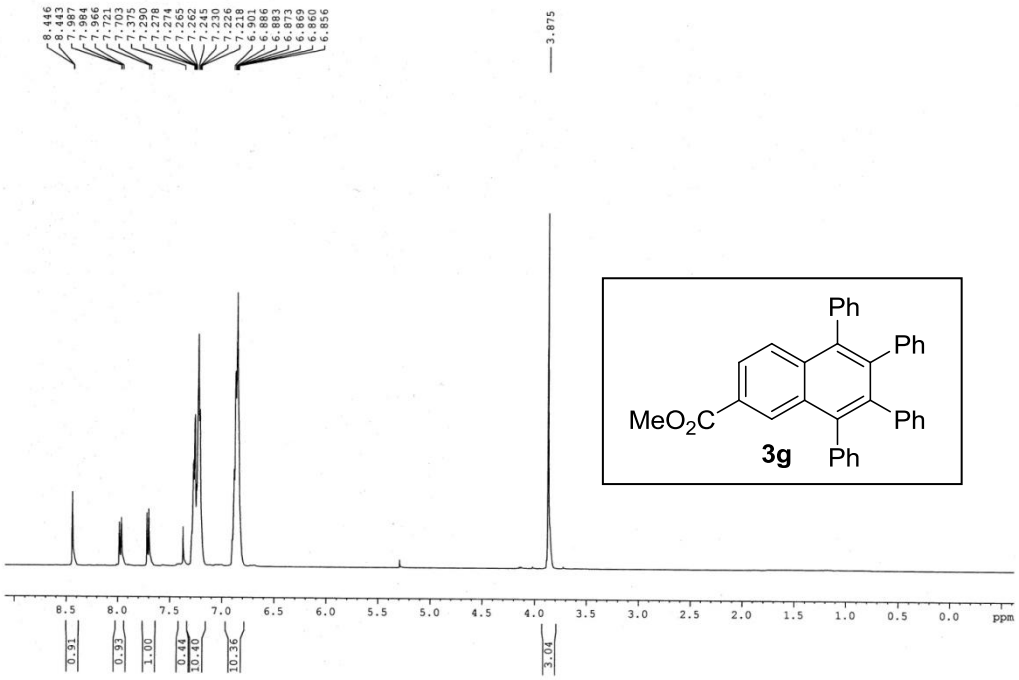
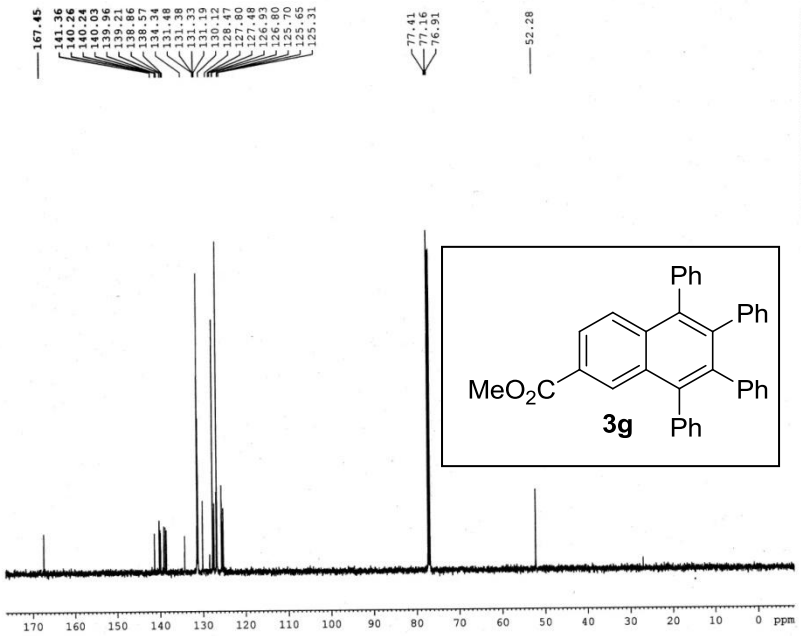
GB 0
PC 1.40





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FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	298.1 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
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SI	16384
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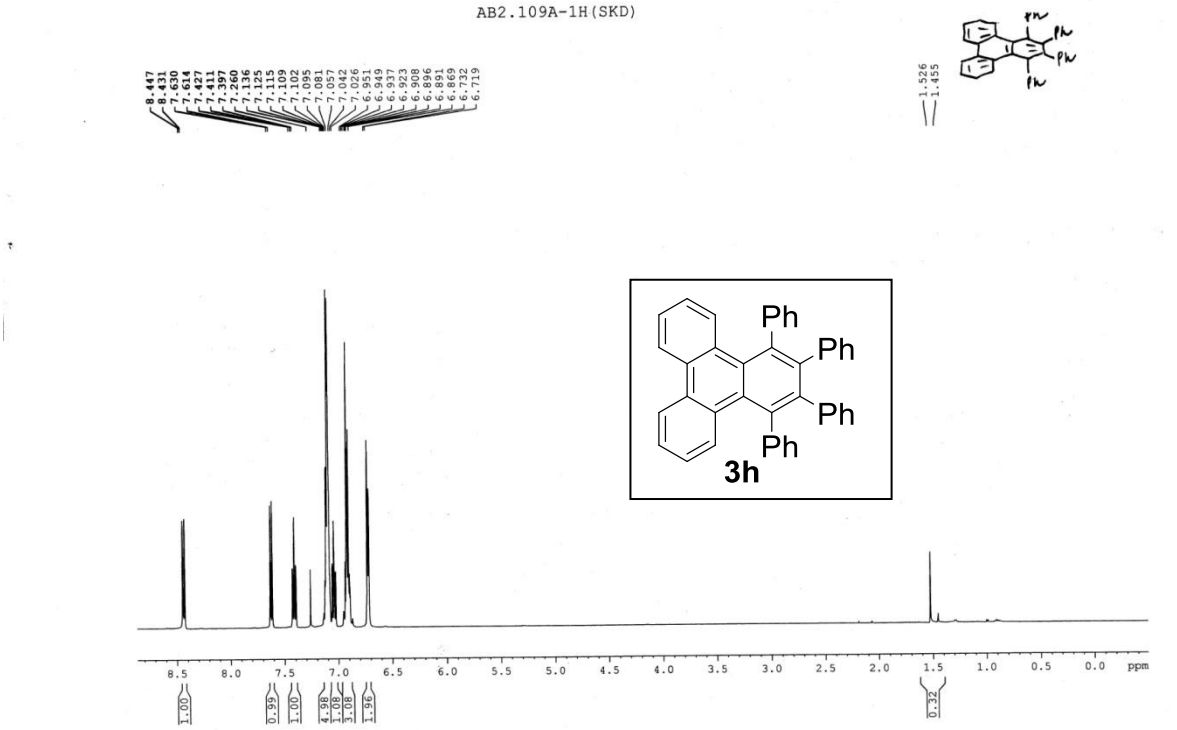
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00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6.896
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Ph
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Ph
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3h
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5.5
5.0
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2.5
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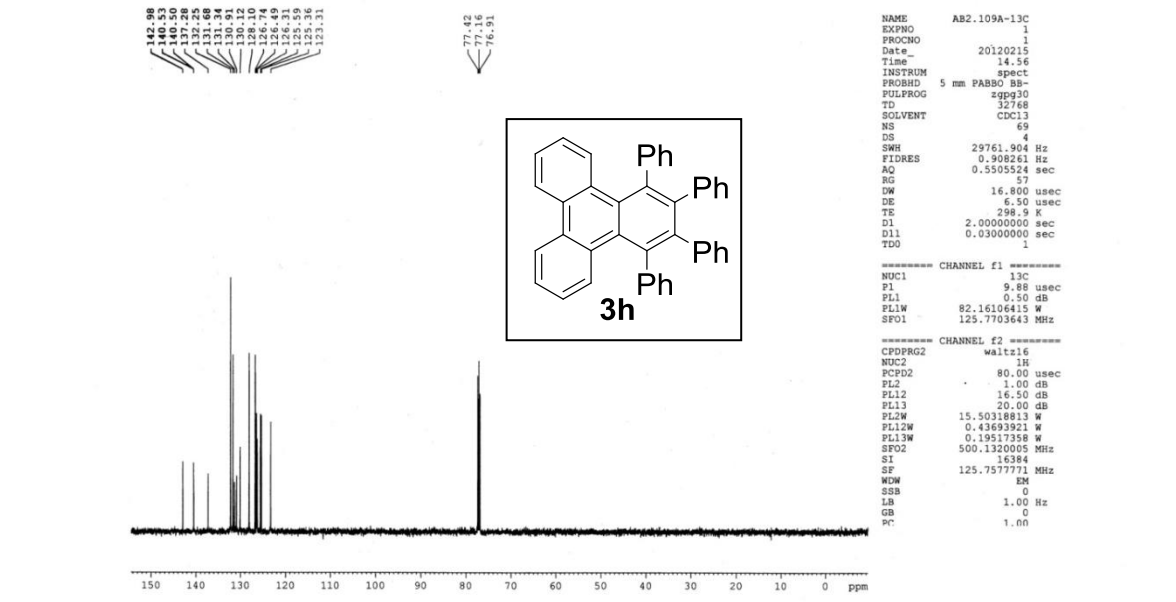
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Ph
Ph
Ph
3h
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120
110
100
90
80
70
60
50
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20
10
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EXPNO 1
PROCNO 1
Date_ 20120215
Time 14.56
INSTRUM spect
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PULPROG zgpg30
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SOLVENT CDCl3
NS 69
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 298.9 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
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SSB 0
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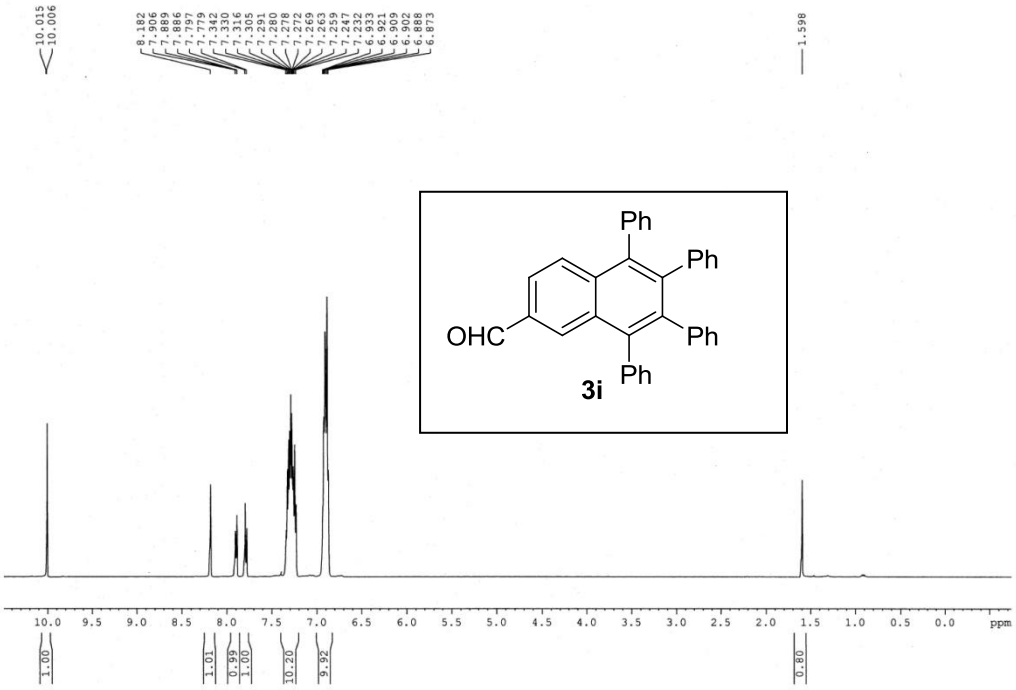
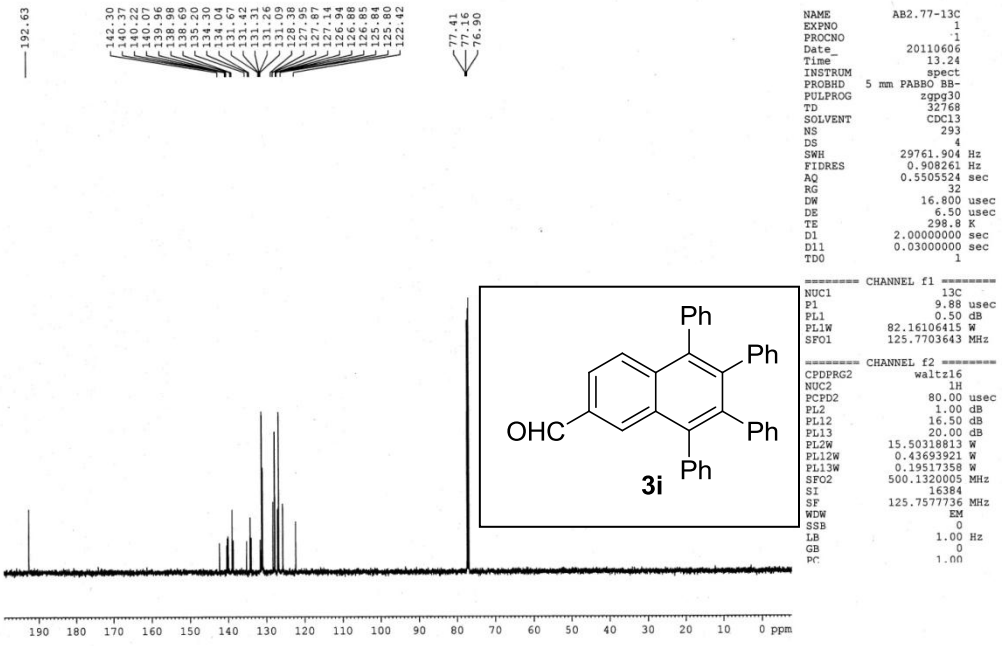
GB 0
PC 1.00





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SOLVENT	CDCl3
NS	293
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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
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WDW	EM
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LB	1.00 Hz
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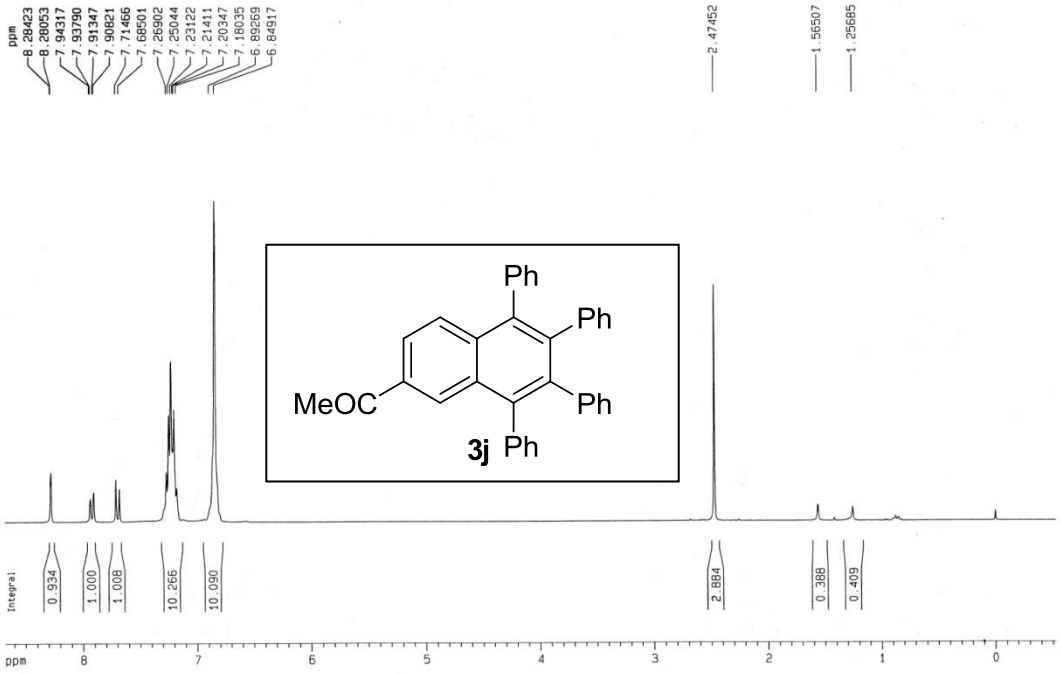
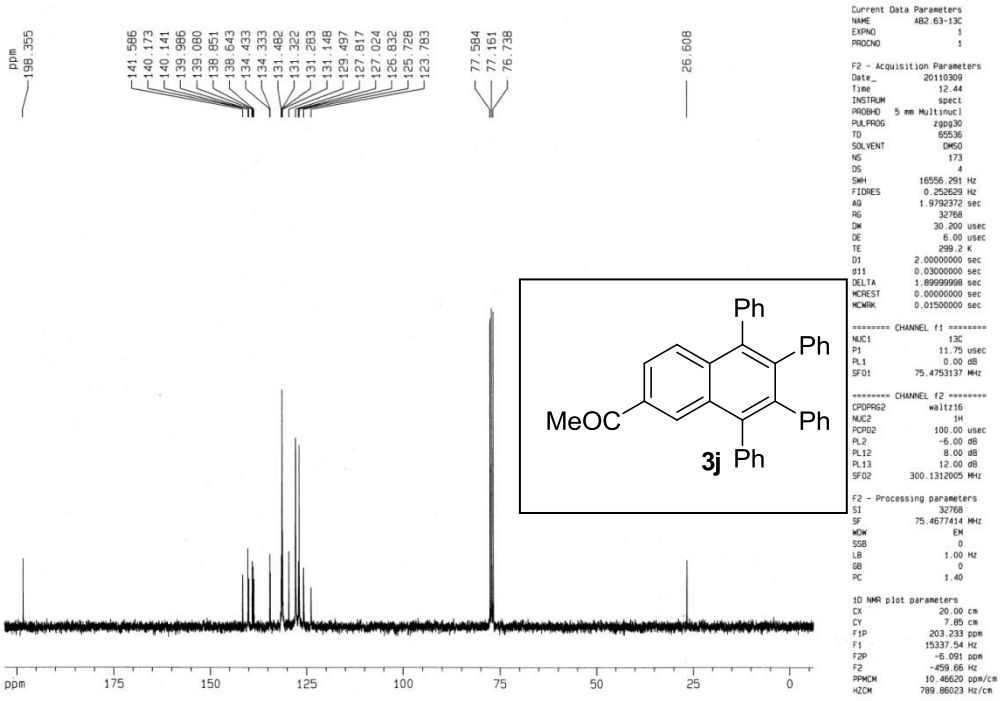
PC	1.00



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PROCNO                         1

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TD                         65536
SOLVENT                    DMSO
NS                           173
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FIDRES                 0.252629 Hz
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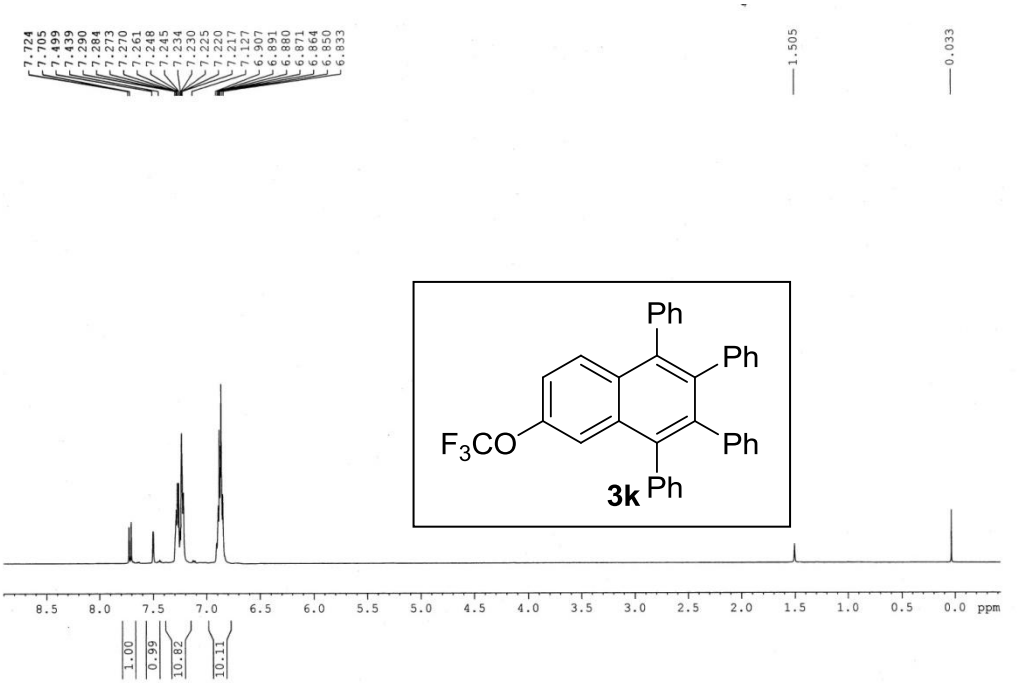
RG                         32768
DW                        30.200 usec
DE                         6.00 usec
TE                        299.2 K
D1                     2.00000000 sec
d11                    0.03000000 sec
DELTA                  1.89999998 sec
MCREST                 0.00000000 sec
MCWRK                  0.01500000 sec

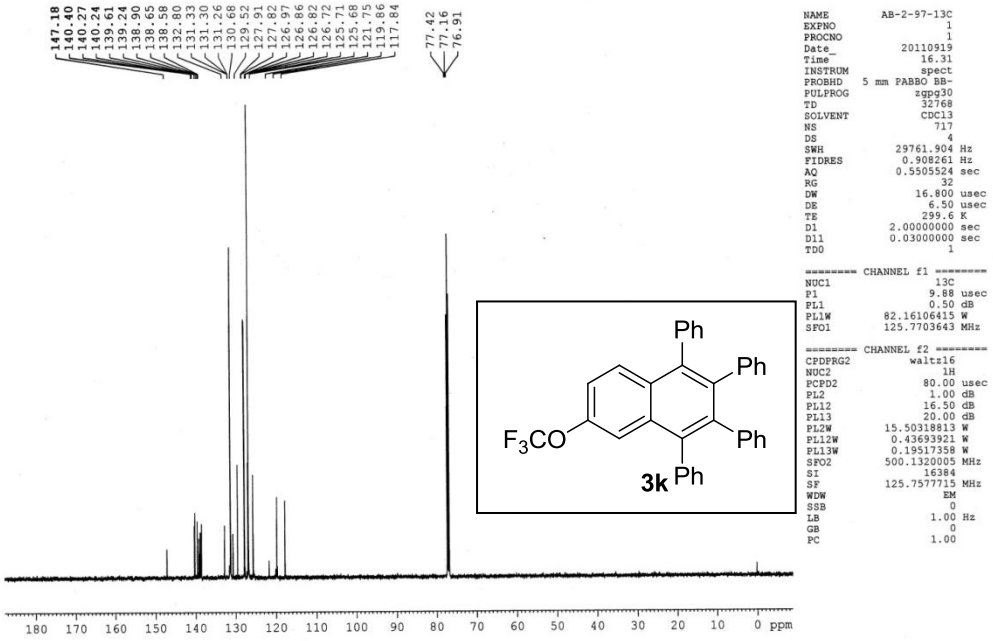
======== CHANNEL f1 ========
NUC1                         13C
P1                         11.75 usec
PL1                         0.00 dB
SFO1                  75.4753137 MHz

======== 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                    75.4677414 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
CY                          7.85 cm
F1P                       203.233 ppm
F1                      15337.54 Hz
F2P                        -6.091 ppm
F2                       -459.66 Hz
PPMCM                  10.46620 ppm/cm
HZCM                   789.86023 Hz/cm
```

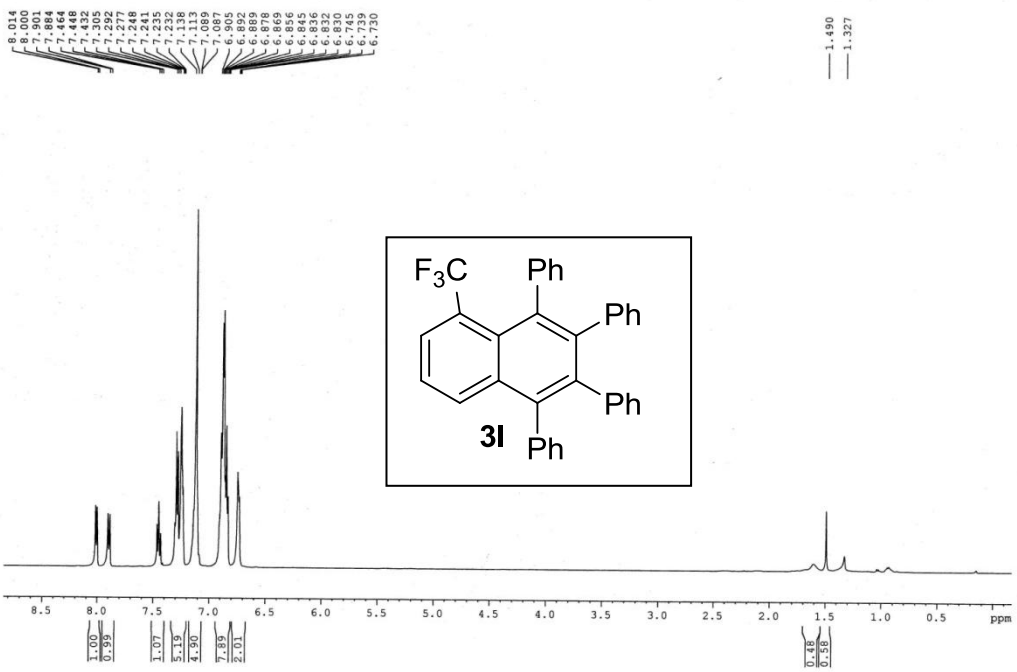


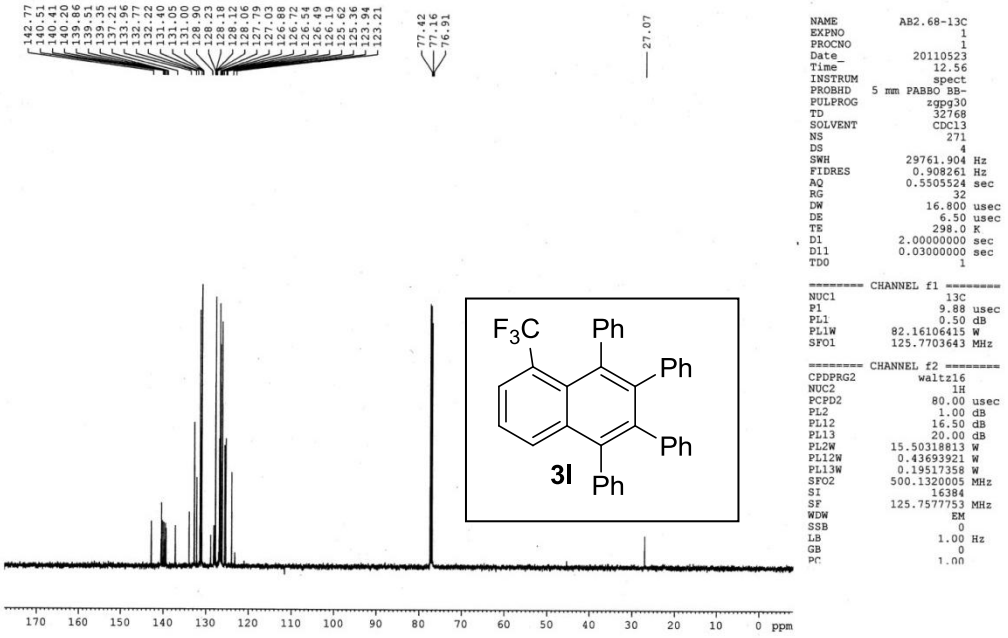



```
NAME                AB-2-97-13C
EXPNO                         1
PROCNO                        1
Date_                  20110919
Time                      16.31
INSTRUM                   spect
PROBHD  5 mm PABBO BB-
PULPROG                zgpg30
TD                         32768
SOLVENT                   CDC13
NS                          717
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
SF                  125.7577715 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                         1.00
```

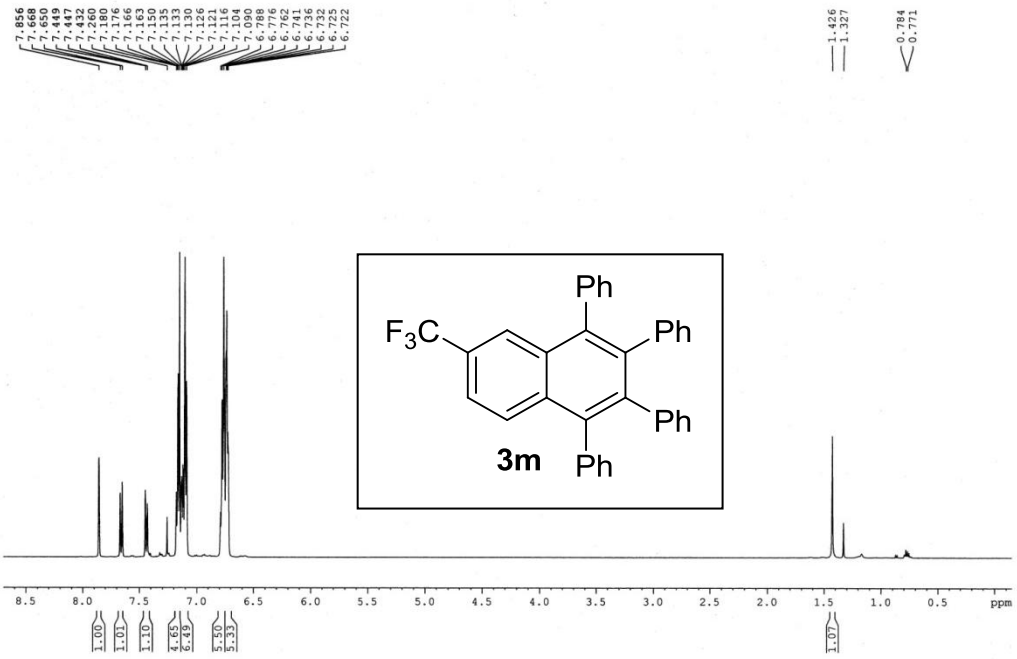


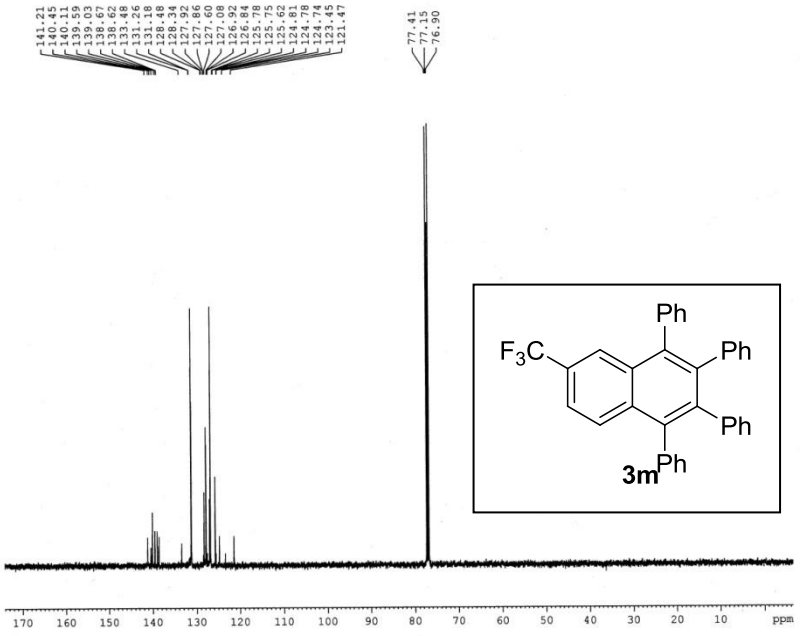



```
NAME                AB2.68-13C
EXPNO                        1
PROCNO                       1
Date_                 20110523
Time                     12.56
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       32768
SOLVENT                  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                       298.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

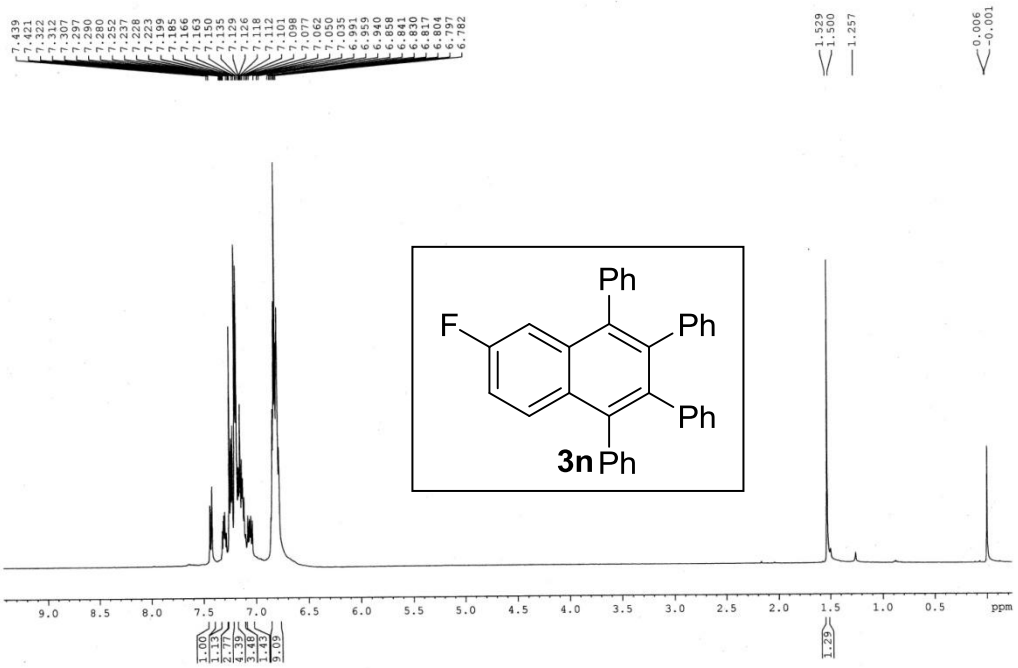
======== CHANNEL f1 ========
NUC1                        13C
P1                         9.88 usec
PL1                        0.50 dB
PL1W                82.16106415 W
SFO1               125.7703643 MHz

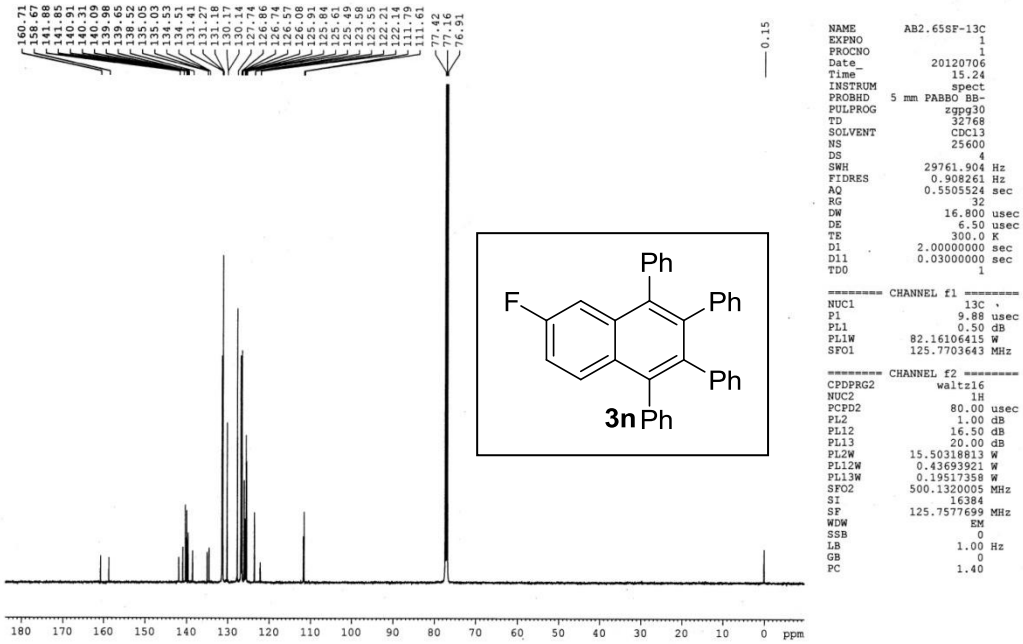
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                        1.00 dB
PL12                      16.50 dB
PL13                      20.00 dB
PL2W                15.50318813 W
PL12W                0.43693921 W
PL13W                0.19517358 W
SFO2               500.1320005 MHz
SI                        16384
SF                 125.7577753 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.00
```



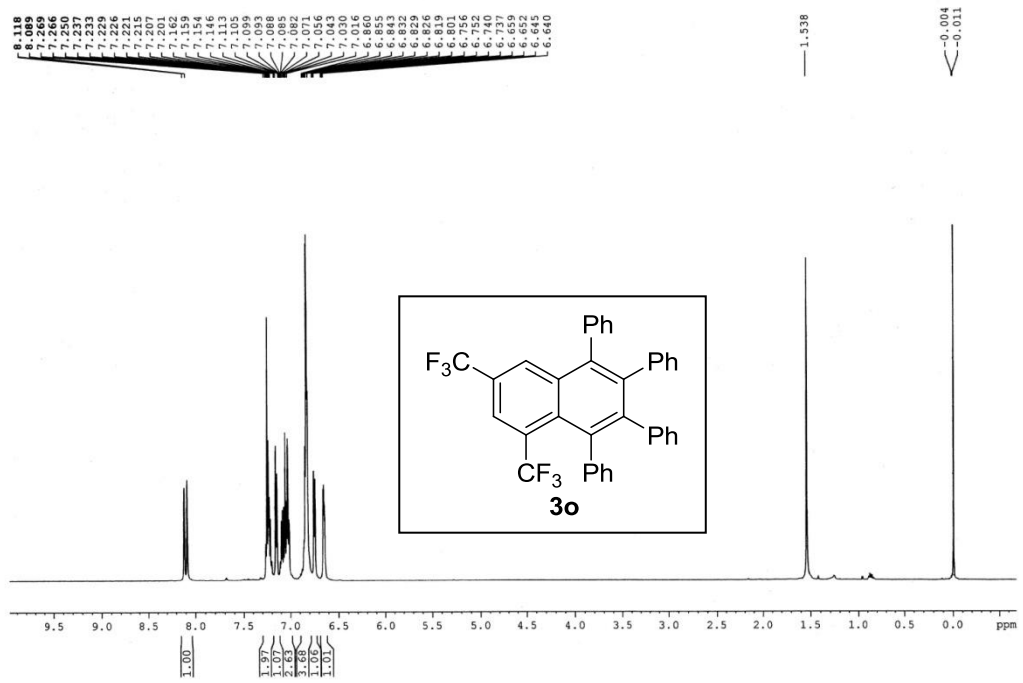


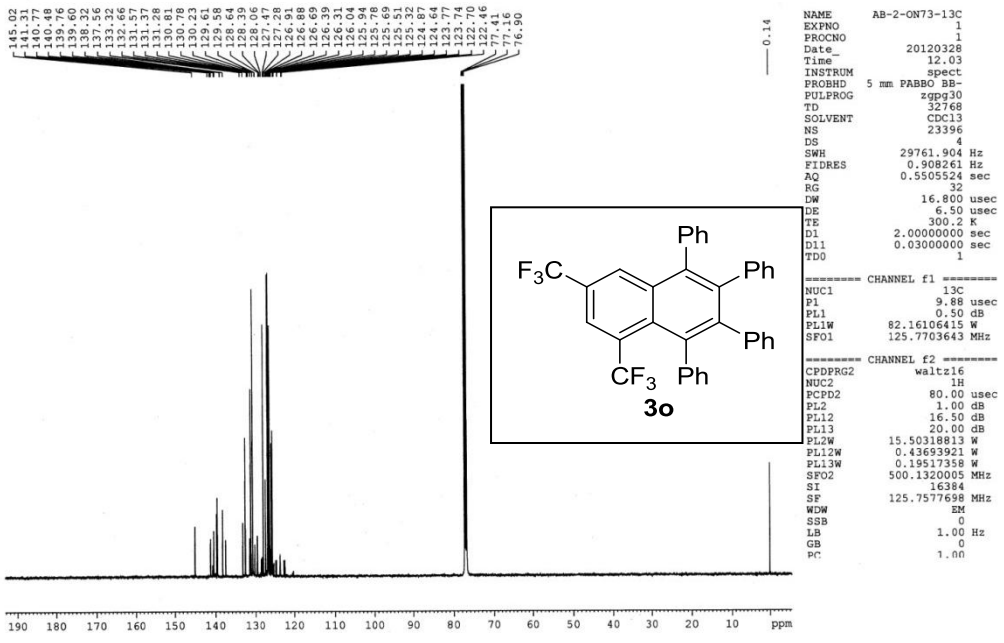
NAME	AB2.69P2-13C
EXPNO	1
PROCNO	1
Date_	20110520
Time	11.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	421
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577716 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

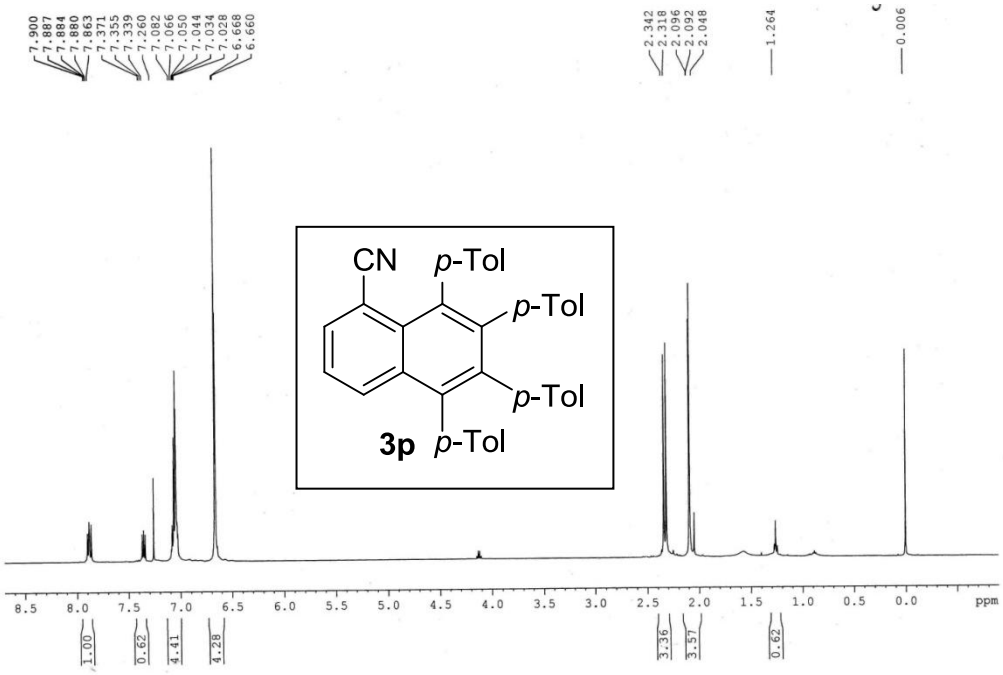


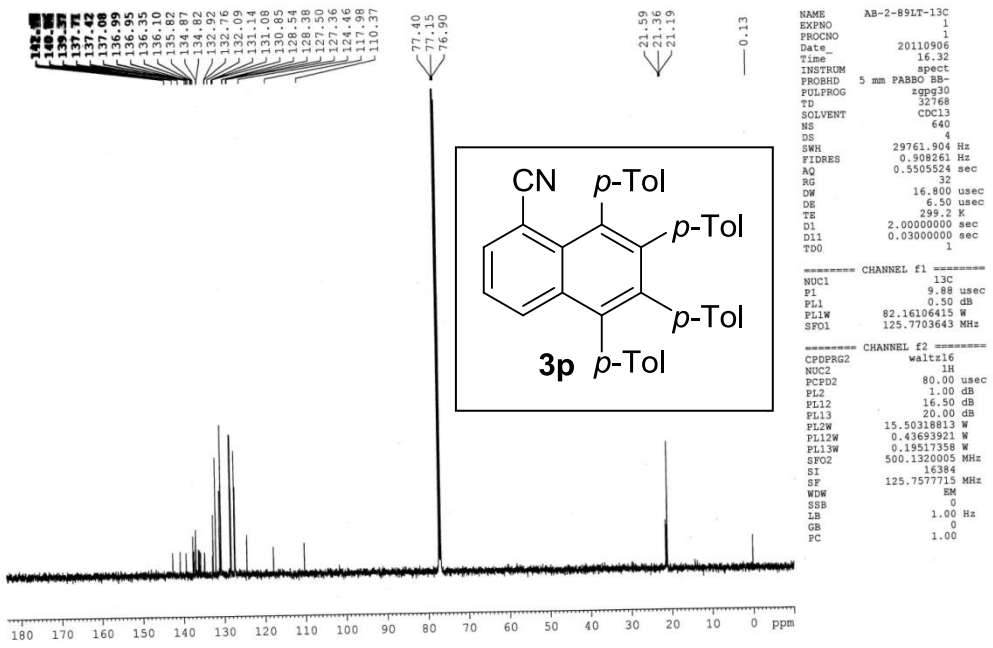


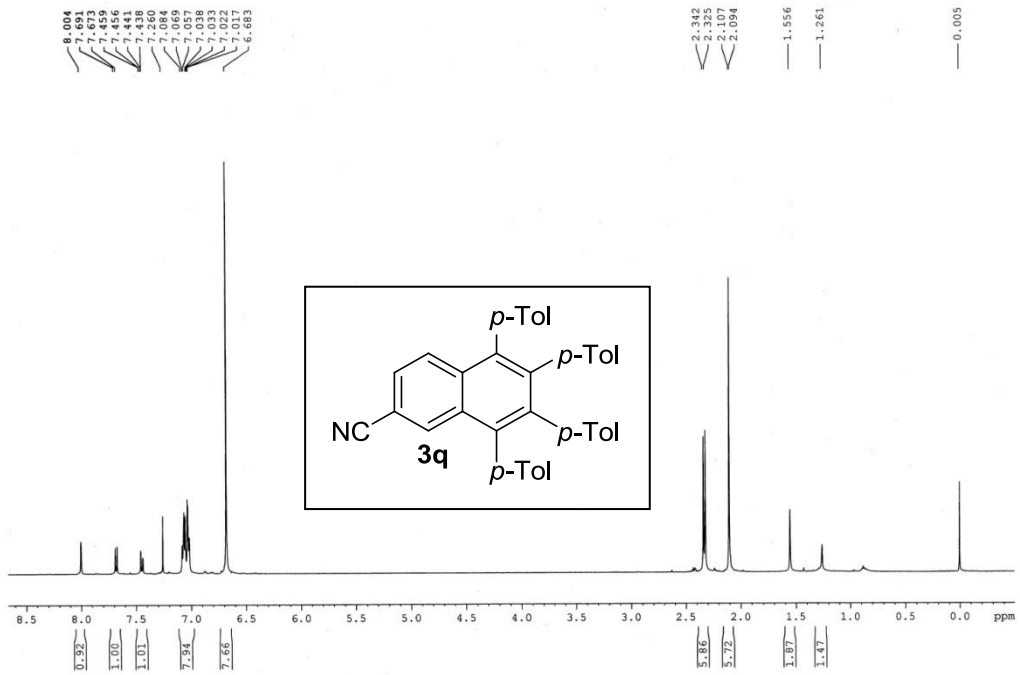
NAME	AB2.65SF-13C
EXPNO	1
PROCNO	1
Date_	20120706
Time	15.24
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	25600
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577699 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

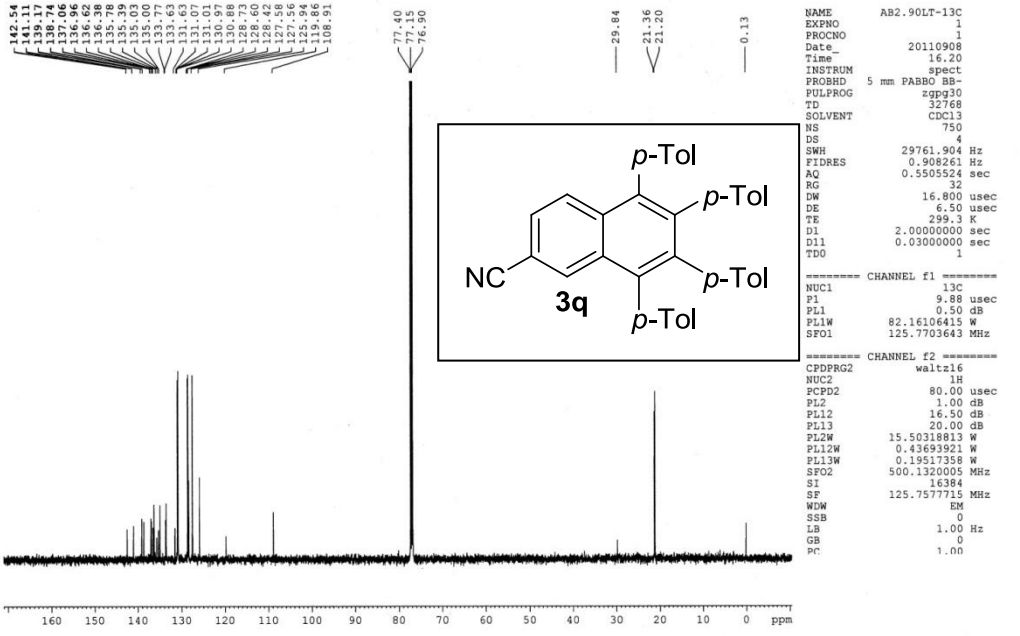

Ph
F3C
Ph
Ph
CF3
Ph
3o
ppm


Ph
F3C
Ph
Ph
CF3
Ph
3o
ppm

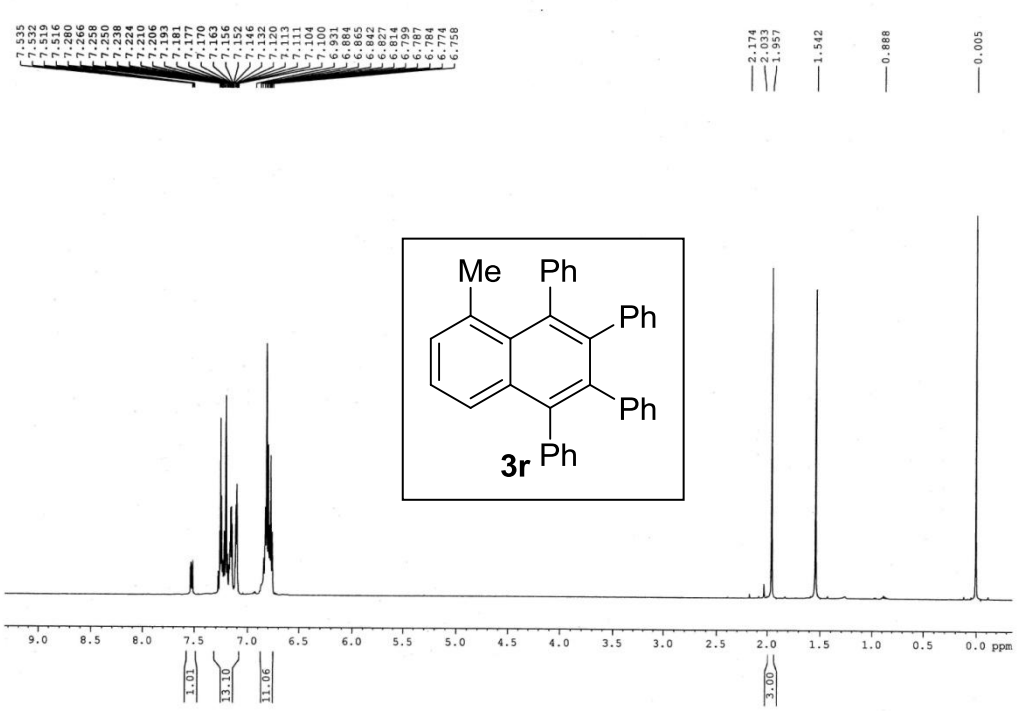

7.900
7.887
7.884
7.880
7.863
7.371
7.355
7.339
7.260
7.082
7.066
7.050
7.044
7.034
7.028
6.668
6.660
2.342
2.318
2.096
2.092
2.048
1.264
0.006
CN
p-Tol
p-Tol
p-Tol
3p
p-Tol
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.62
4.41
4.28
3.36
3.57
0.62

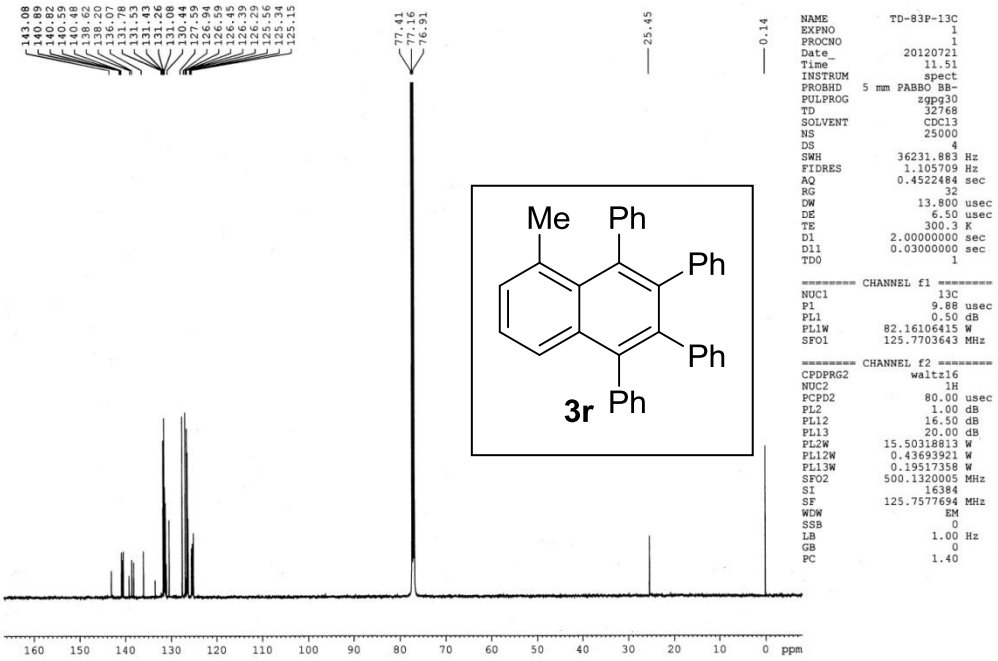

77.40
77.15
76.90
21.59
21.36
21.19
0.13
CN
p-Tol
p-Tol
p-Tol
3p
p-Tol
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
NAME AB-2-89LT-13C
EXPNO 1
PROCNO 1
Date_ 20110906
Time 16.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 640
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 32
DW 16.800 usec
DE 6.50 usec
TE 299.2 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 125.7577715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



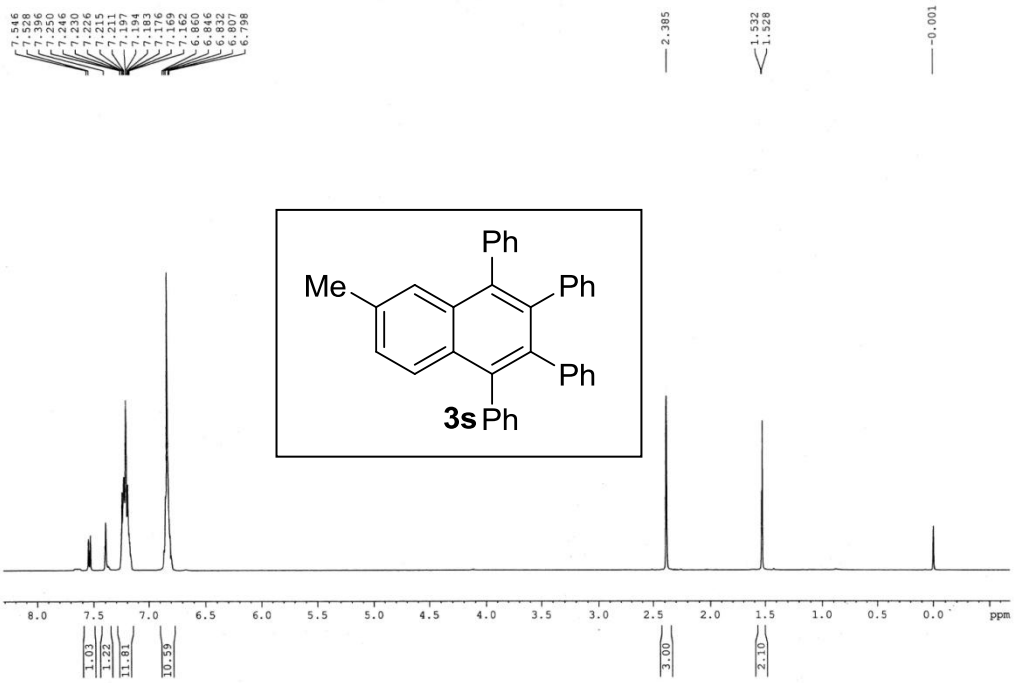


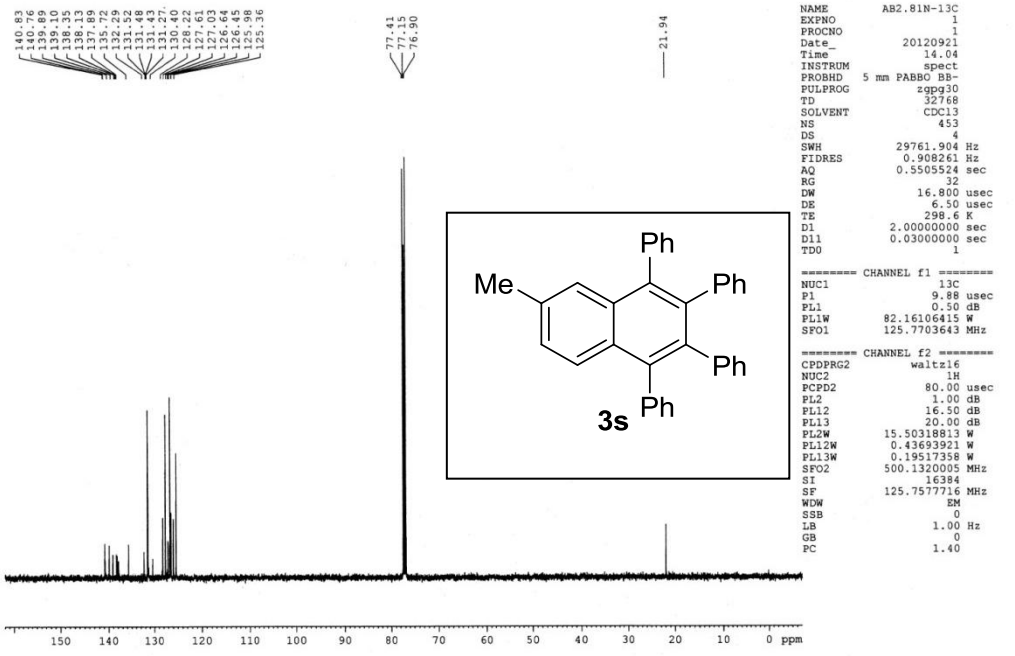
NAME	AB2.90LT-13C
EXPNO	1
PROCNO	1
Date_	20110908
Time	16.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	750
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL 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	125.7577715 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00



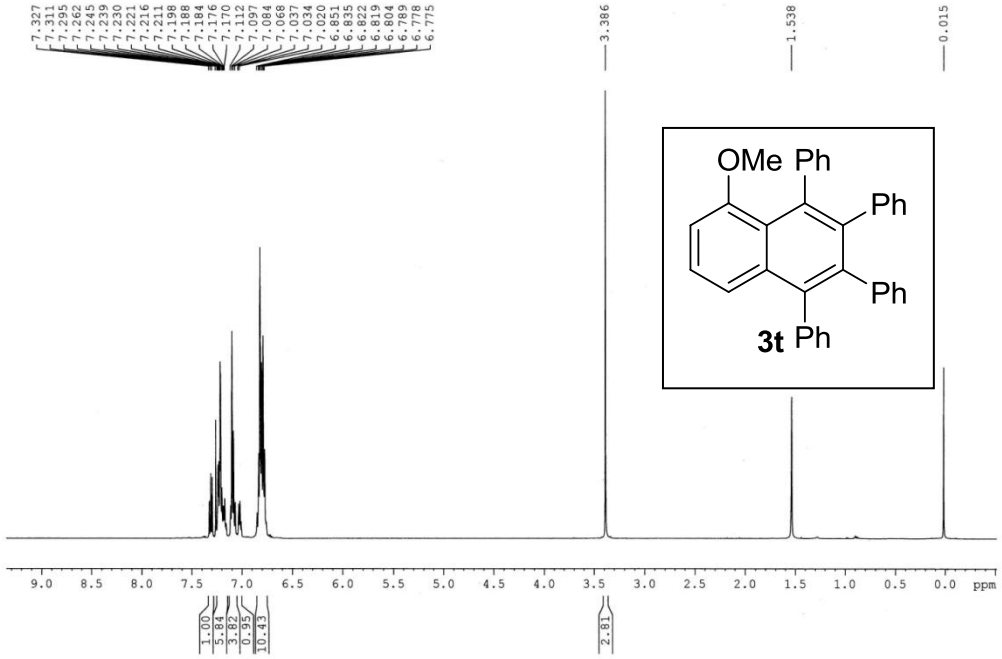


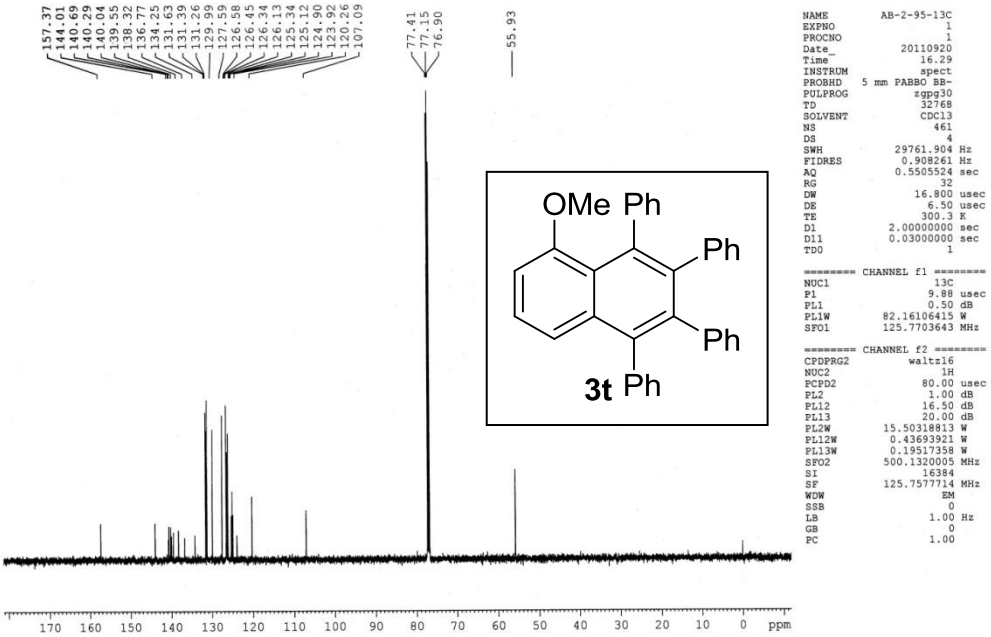
NAME	TD-83P-13C
EXPNO	1
PROCNO	1
Date_	20120721
Time	11.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	25000
DS	4
SWH	36231.883 Hz
FIDRES	1.105709 Hz
AQ	0.4522484 sec
RG	32
DW	13.800 usec
DE	6.50 usec
TE	300.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577694 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



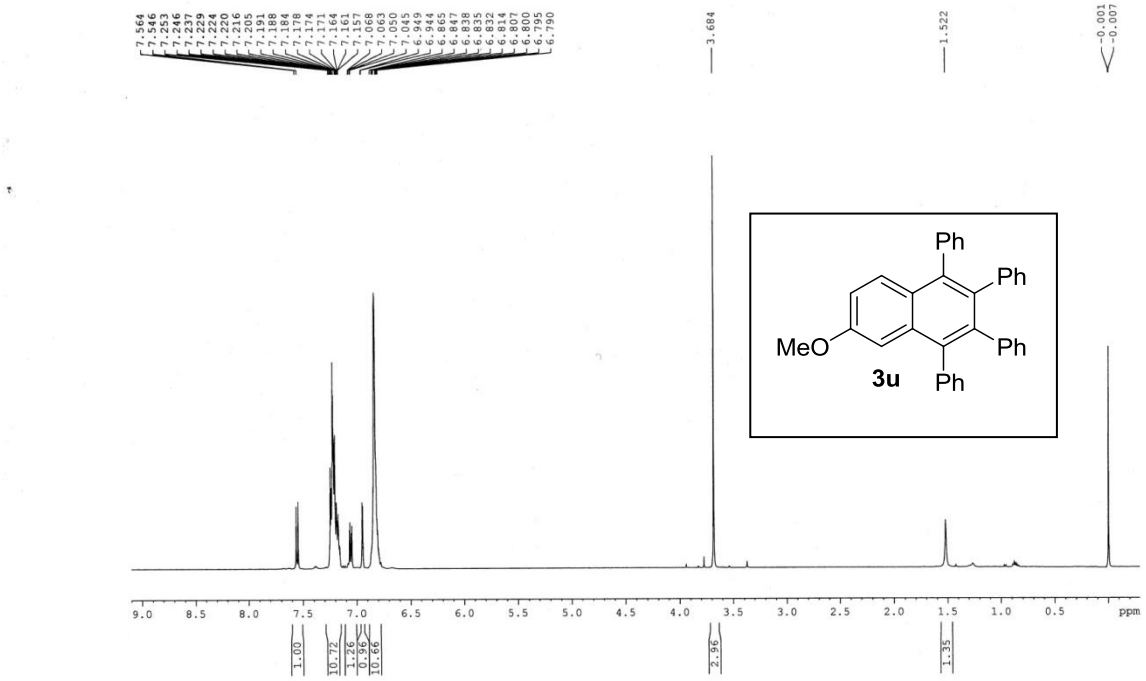


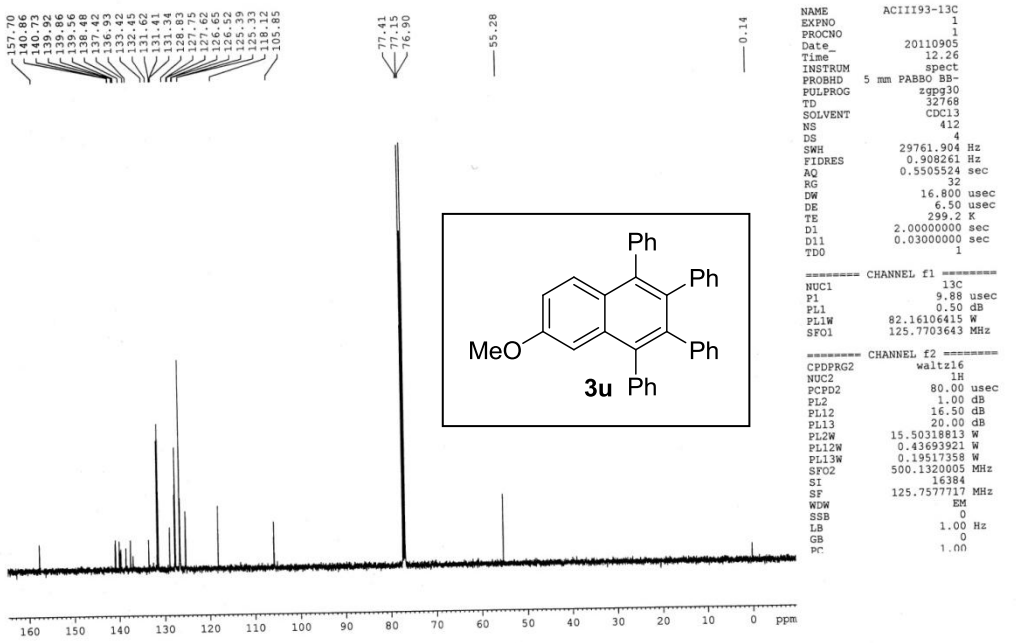
NAME	AB2.81N-13C
EXPNO	1
PROCNO	1
Date_	20120921
Time	14.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	453
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577716 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



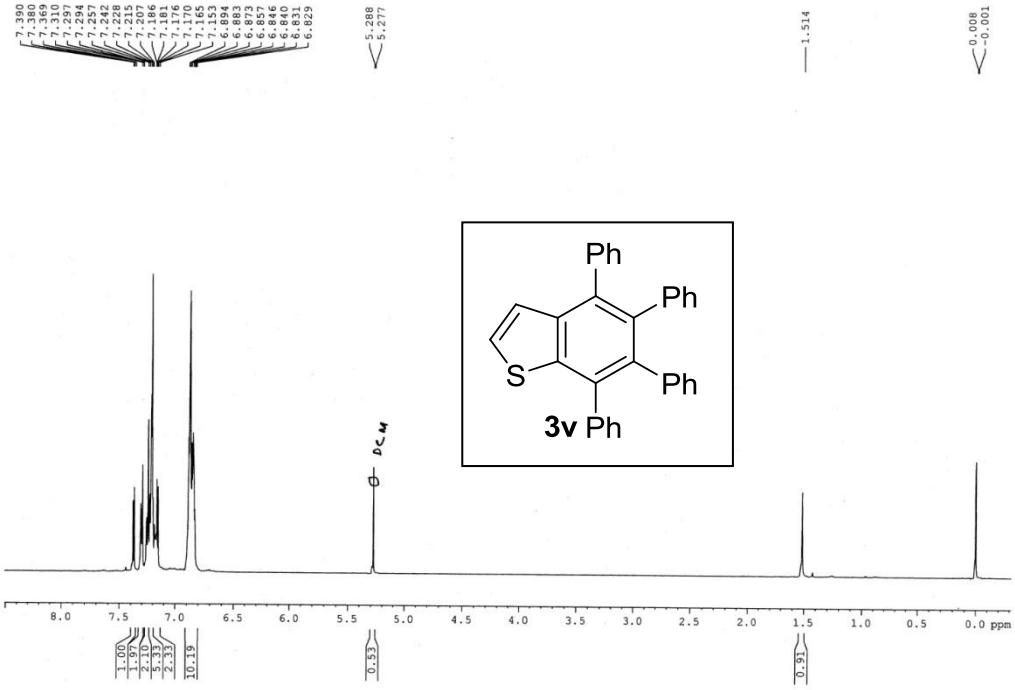


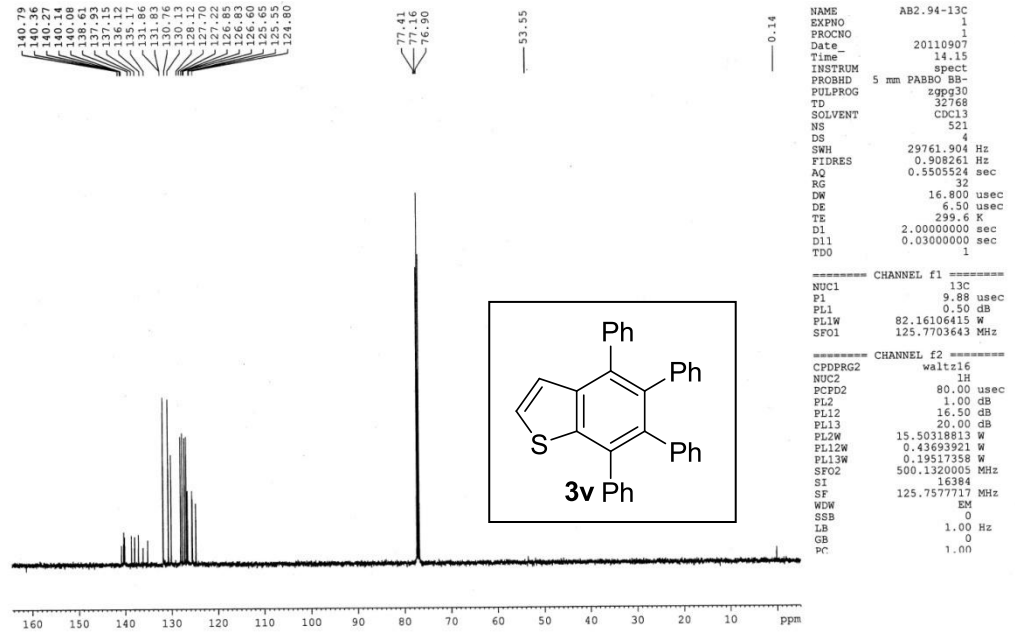
NAME	AB-2-95-13C
EXPNO	1
PROCNO	1
Date_	20110920
Time	16.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	461
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	300.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577714 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00



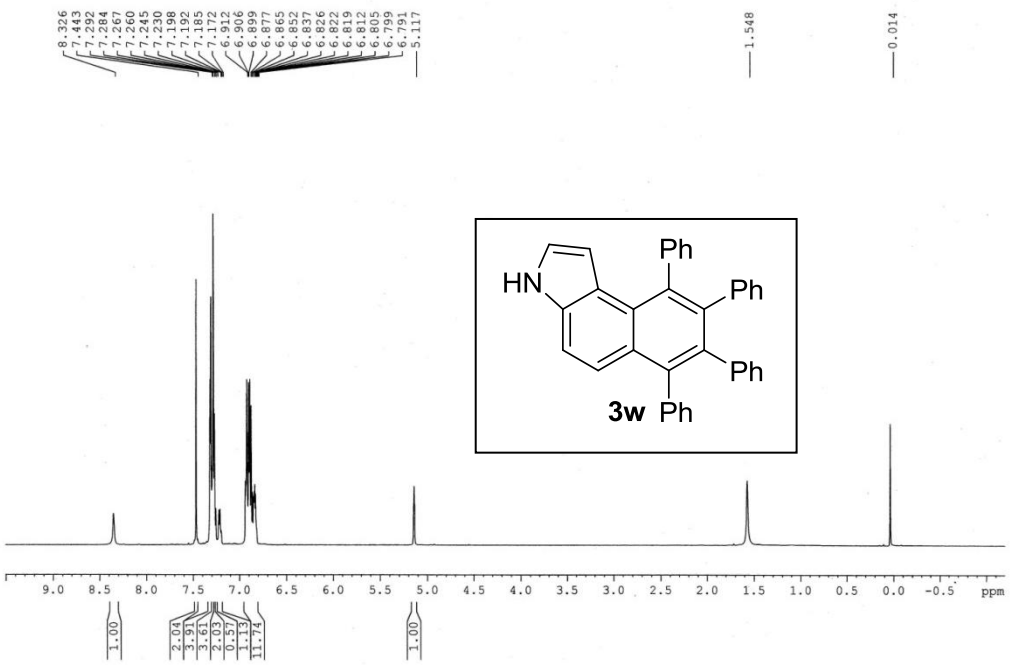


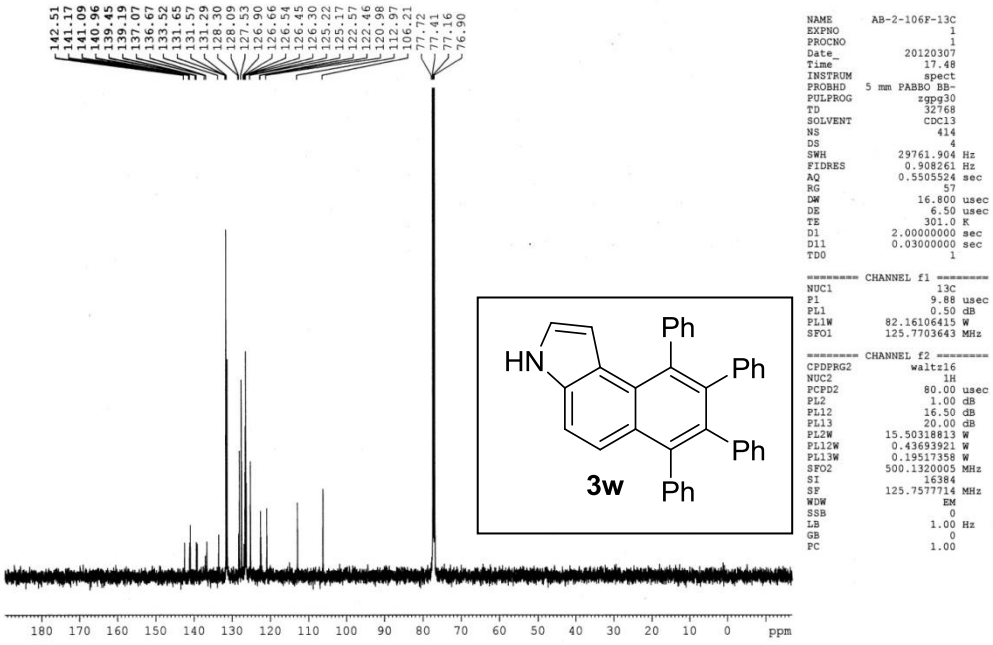
NAME	ACIII93-13C
EXPNO	1
PROCNO	1
Date_	20110905
Time	12.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	412
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	32
DW	16.800 usec
DE	6.50 usec
TE	299.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL 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	125.7577717 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00





NAME	AB2.94-13C
EXPNO	1
PROCNO	1
Date_	20110907
Time	14.15
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	521
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
SF	125.7577717 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00



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PROCNO                      1
Date_            20120307
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PULPROG                 zgpg30
TD                       32768
SOLVENT                 CDCl3
NS                        414
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                      301.0 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                 125.7577714 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.00
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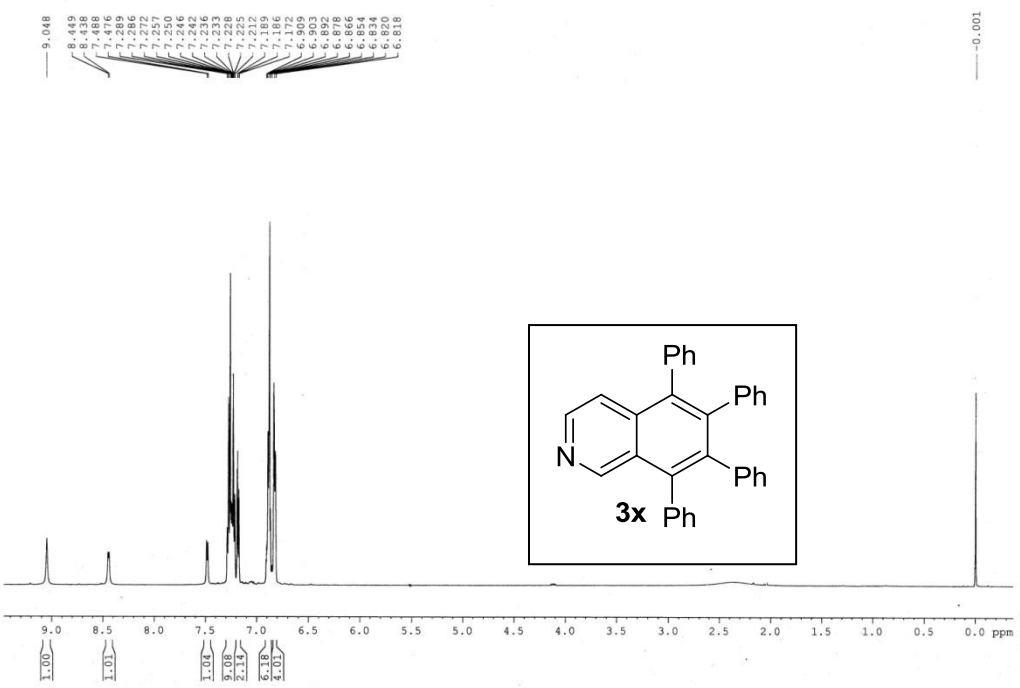
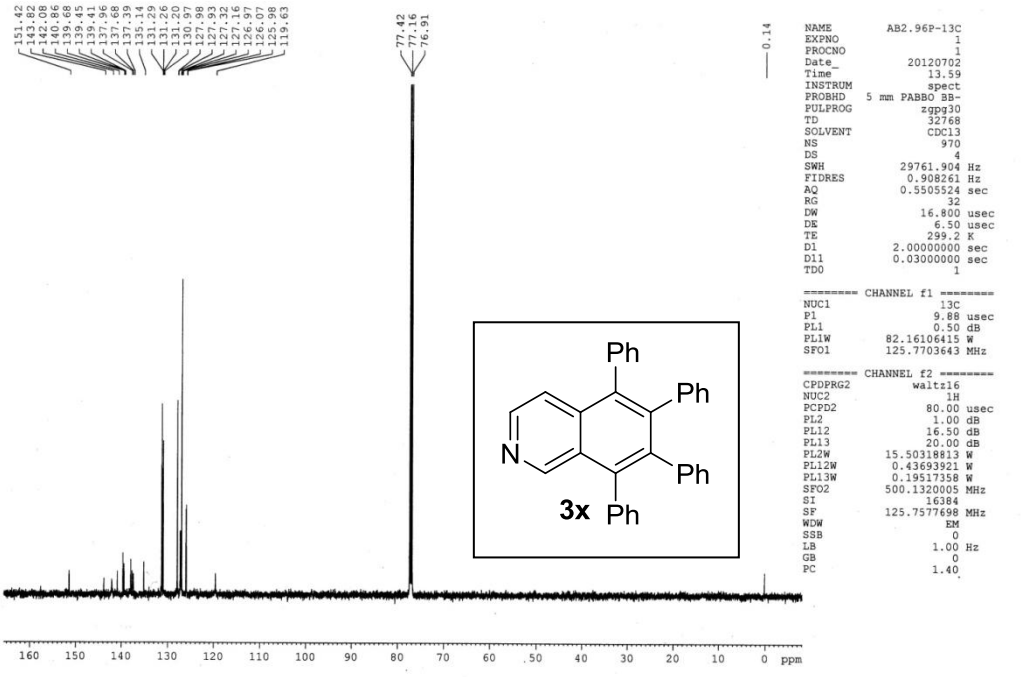

9.048
8.449
8.438
7.488
7.476
7.289
7.286
7.272
7.257
7.250
7.246
7.242
7.236
7.233
7.228
7.225
7.212
7.189
7.186
7.172
6.909
6.903
6.892
6.878
6.866
6.854
6.834
6.820
6.818
-0.001
Ph
Ph
Ph
Ph
N
3x
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.01
1.04
9.08
2.14
6.18
4.01


151.42
143.82
142.08
140.86
139.68
139.45
139.41
137.96
137.68
137.39
135.14
131.29
131.26
131.20
130.97
130.88
127.93
127.32
127.16
126.97
126.07
125.98
119.63
77.42
77.16
76.91
0.14
NAME AB2.96P-13C
EXPNO 1
PROCNO 1
Date_ 20120702
Time 13.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 970
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 299.2 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 125.7577698 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Ph
Ph
Ph
Ph
N
3x
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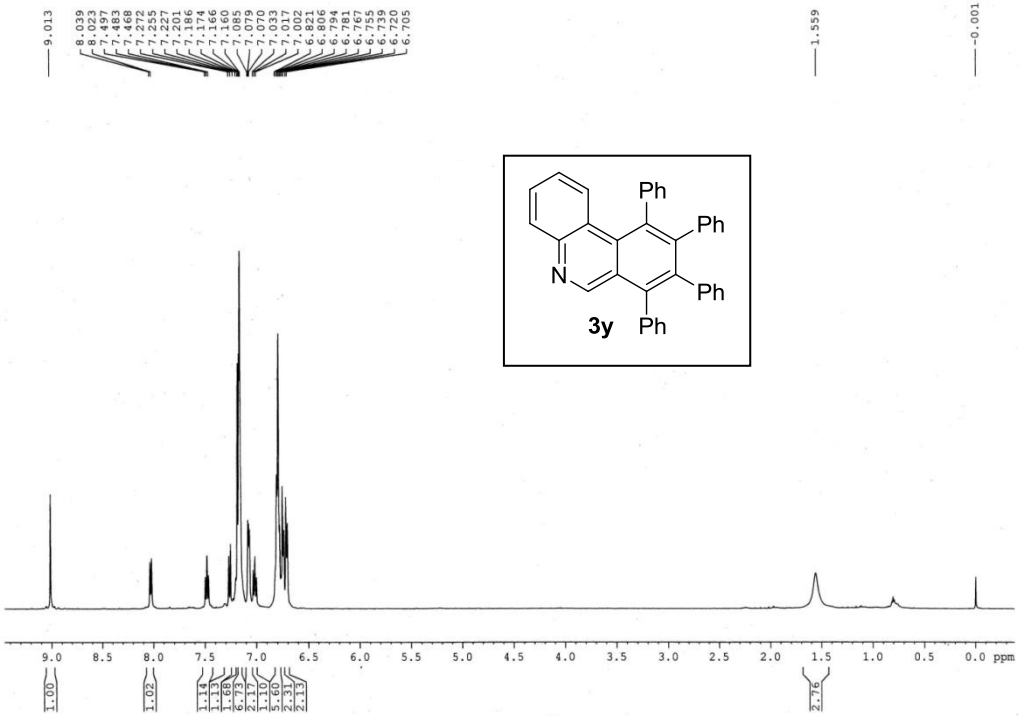

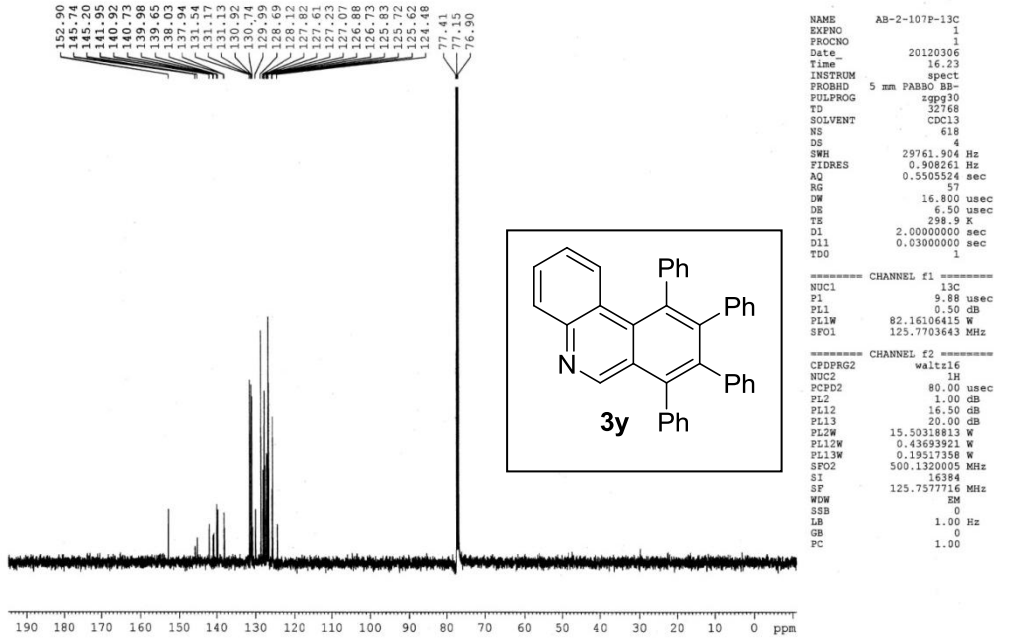

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PROCNO	1
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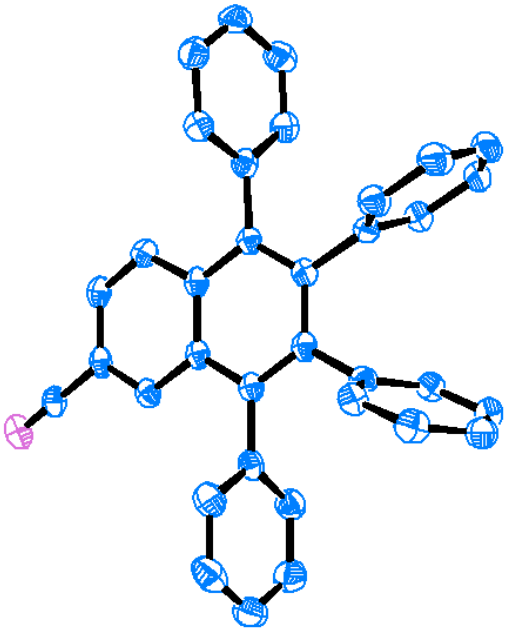
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	618
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	57
DW	16.800 usec
DE	6.50 usec
TE	298.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.88 usec
PL1	0.50 dB
PL1W	82.16106415 W
SFO1	125.7703643 MHz
======== 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	125.7577716 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

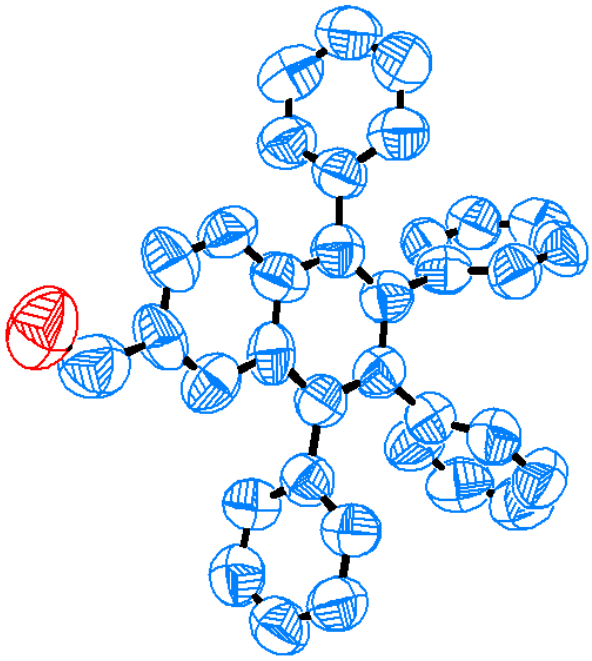
Crystallographic information:

Figure 1: thermal ellipsoid representation of crystal structure of compound **3b**



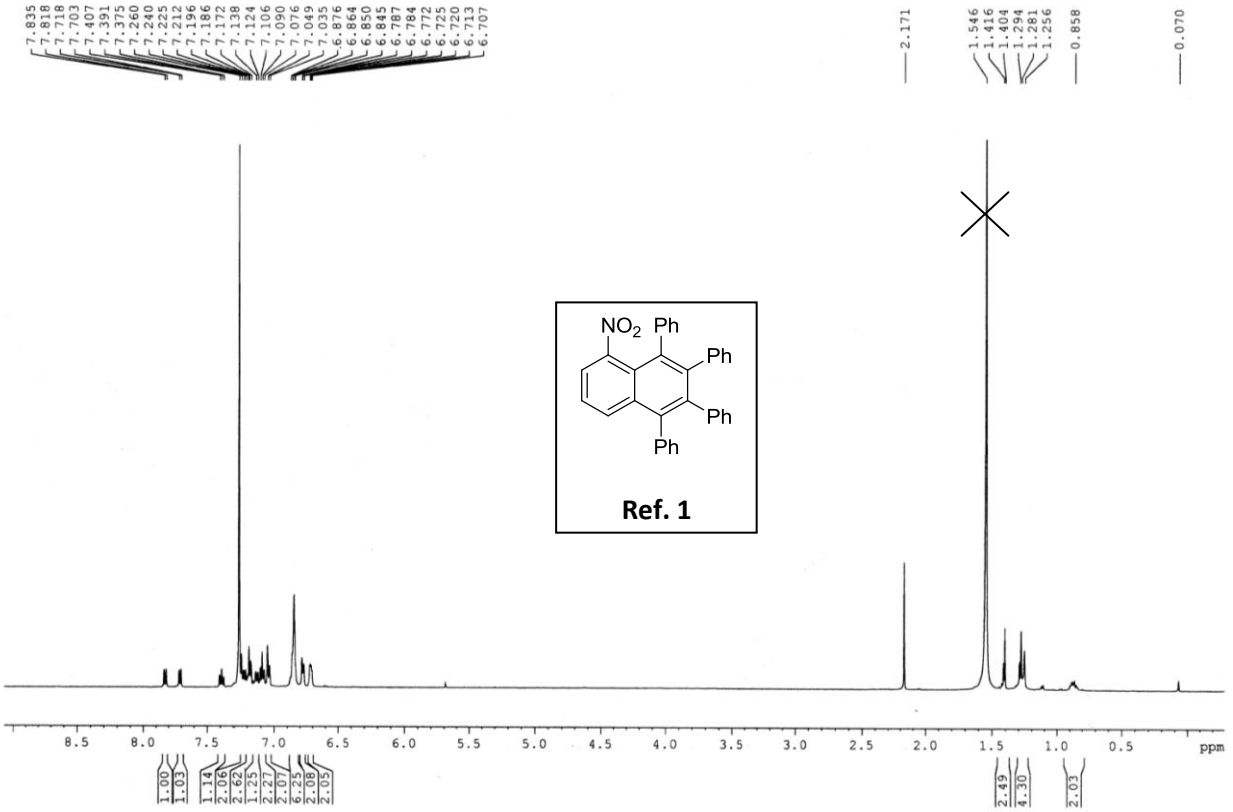
Crystallographic data for compound **3b.** $C_{35}H_{23}N$; Mr = 457.54 $gmol^{-1}$, orthorhombic, space group P_{bca}, a = 6.6655 (8) Å, b = 21.962 (2) Å, c = 32.777 (4) Å, α = 90˚, β = 90˚, γ = 90˚, V = 4798.2 (9) $Å^3$, Z = 8, $\rho_{calcd.}$ = 1.267 gcm^{-3}, μ = 0.073 mm^{-1}, T = 150(2) K, 4029 observed reflections, 325 refined parameters, R_1 = 0.0453 wR_2 = 0.1479 GOF = 1.102.

Figure 2: thermal ellipsoid representation of crystal structure of compound **3i**

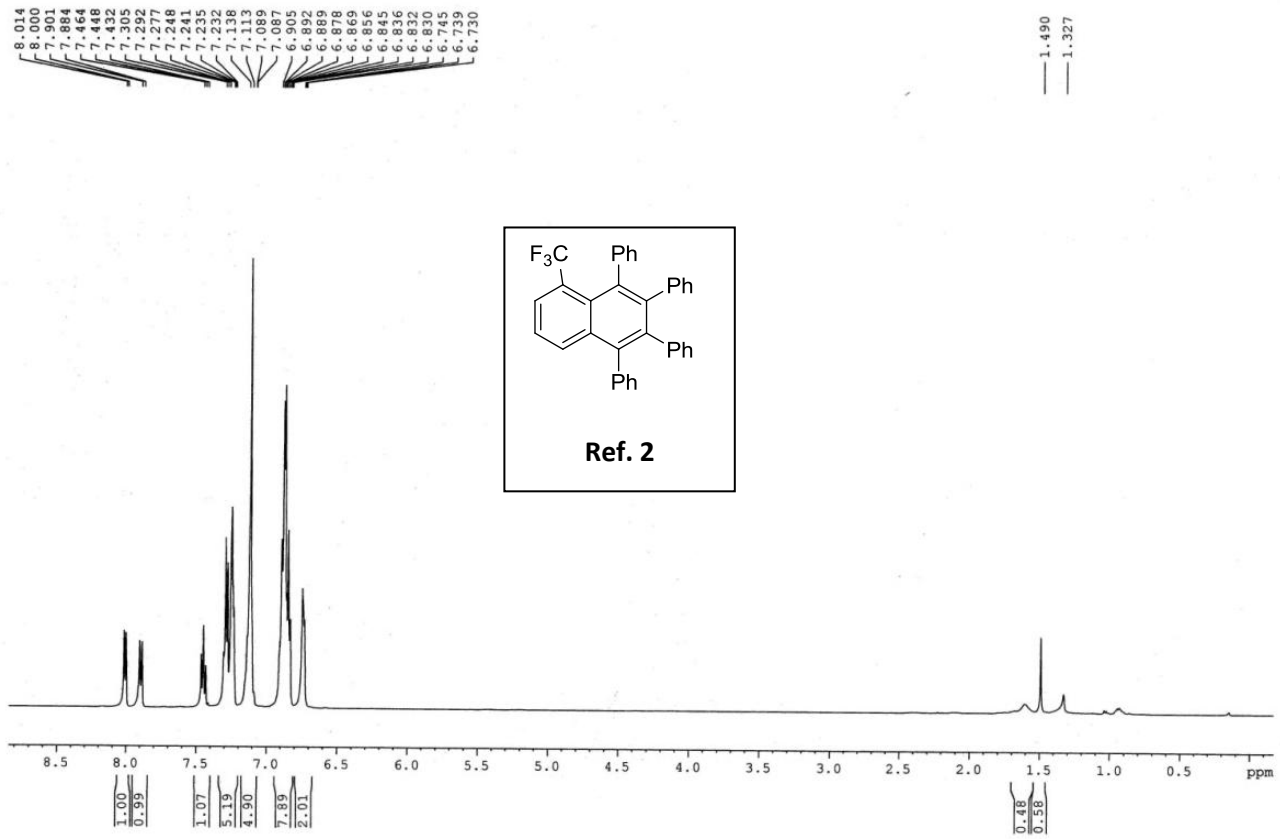


Crystallographic data for compound **3i.** $C_{35}H_{24}O$; Mr = 460.54 $gmol^{-1}$, orthorhombic, space group $P2_12_12_1$, a = 8.242(4) b=15.900(8) c=18.948(10) α = 90˚, β = 90˚, γ = 90˚, V = 2483 (2) $Å^3$, Z = 4, $\rho_{calcd.}$ = 1.232 g cm^{-3}, μ = 0.072 mm^{-1}, T = 150(2) K, 1488 observed reflections, 327 refined parameters, R_1 = 0.0468 wR_2 = 0.1213 GOF = 0.504.

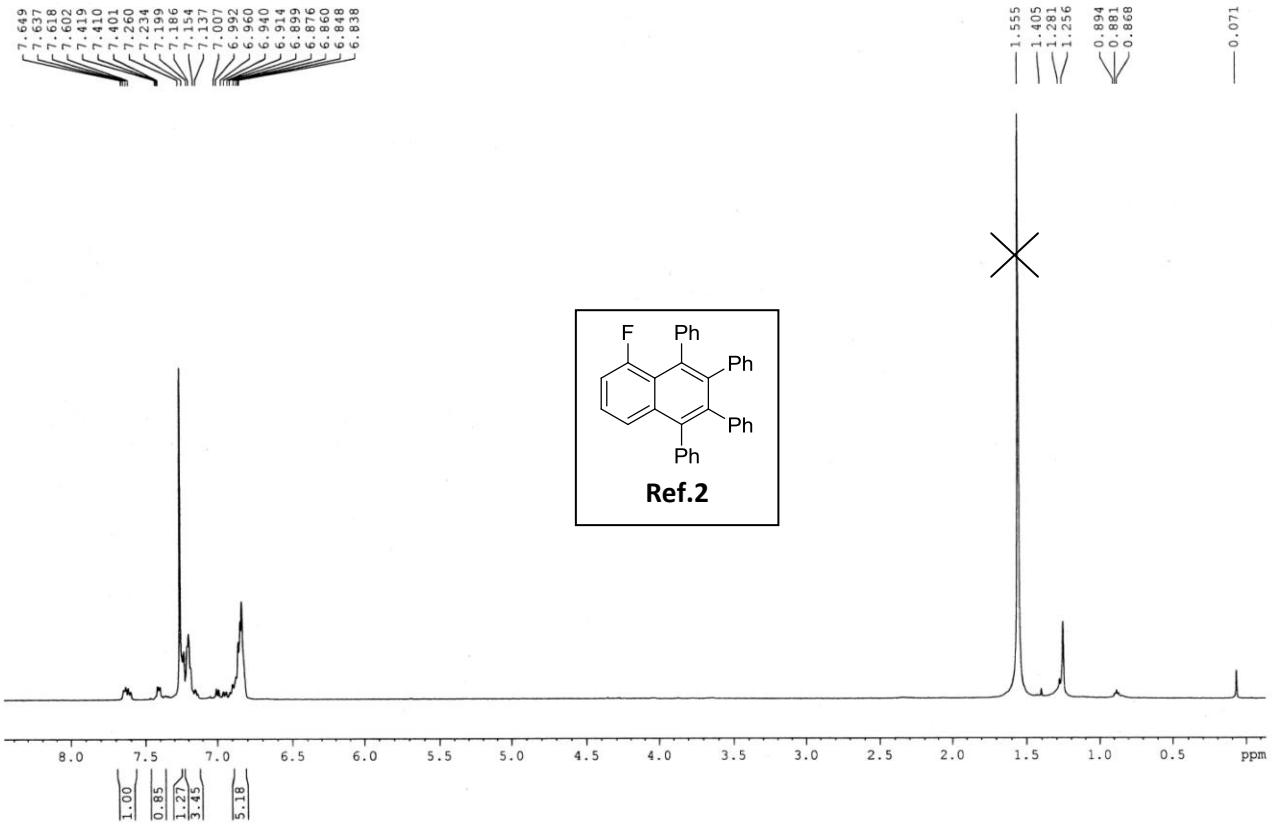
Isomeric products – some representative NMR data :

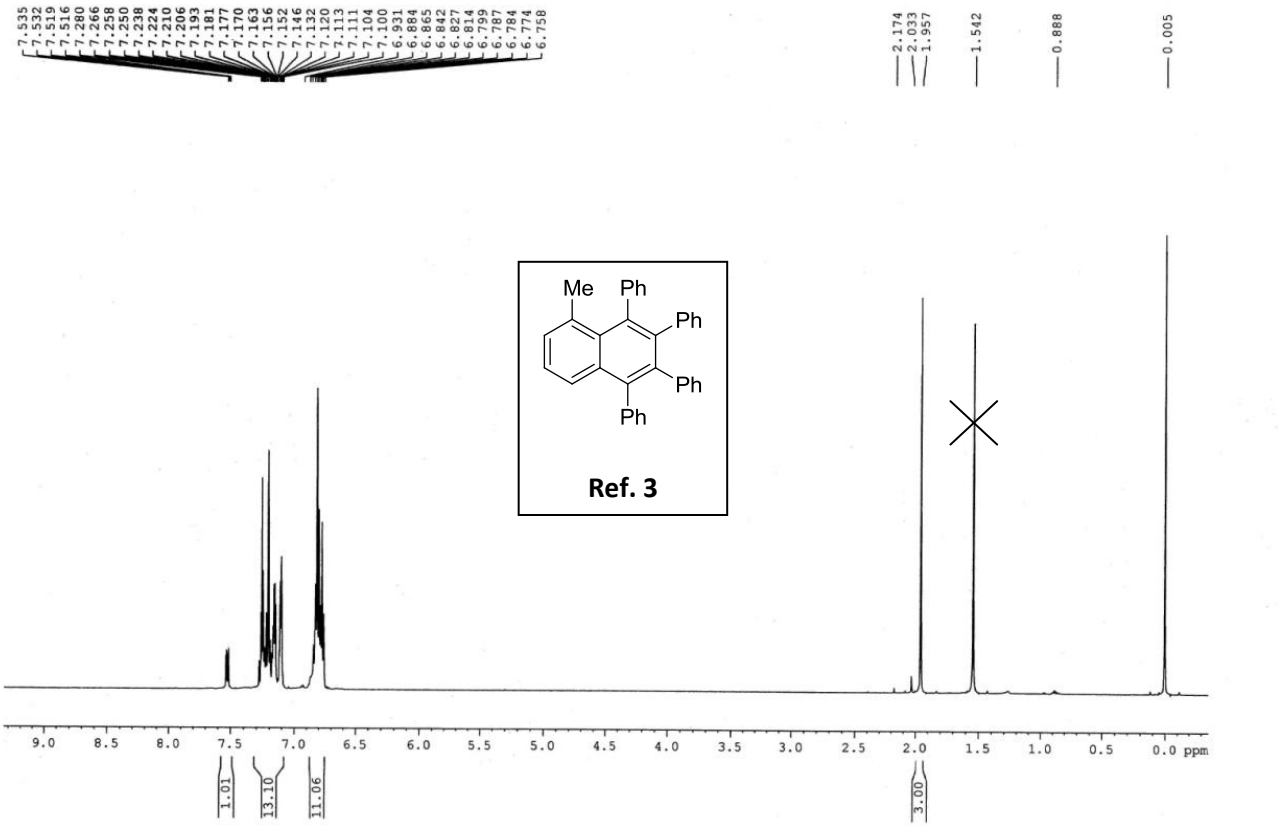


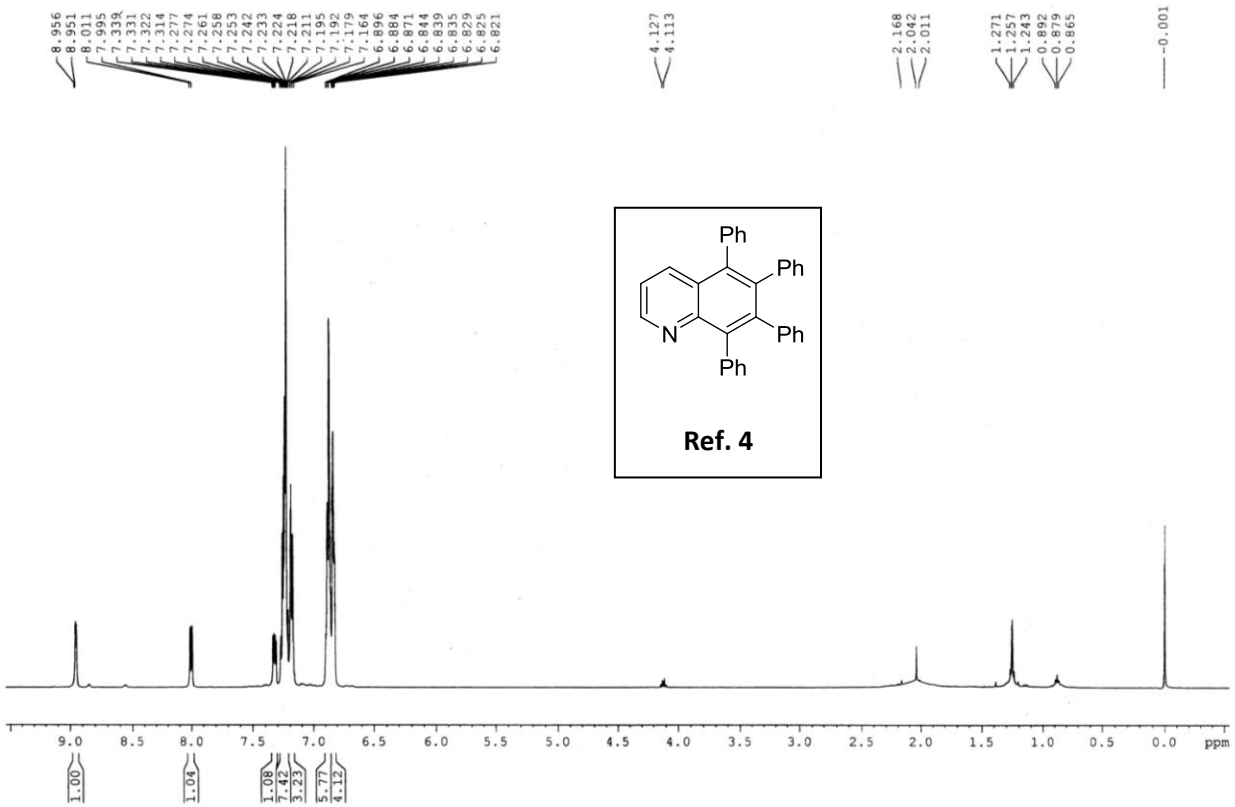
Ref. 1



Ref. 2

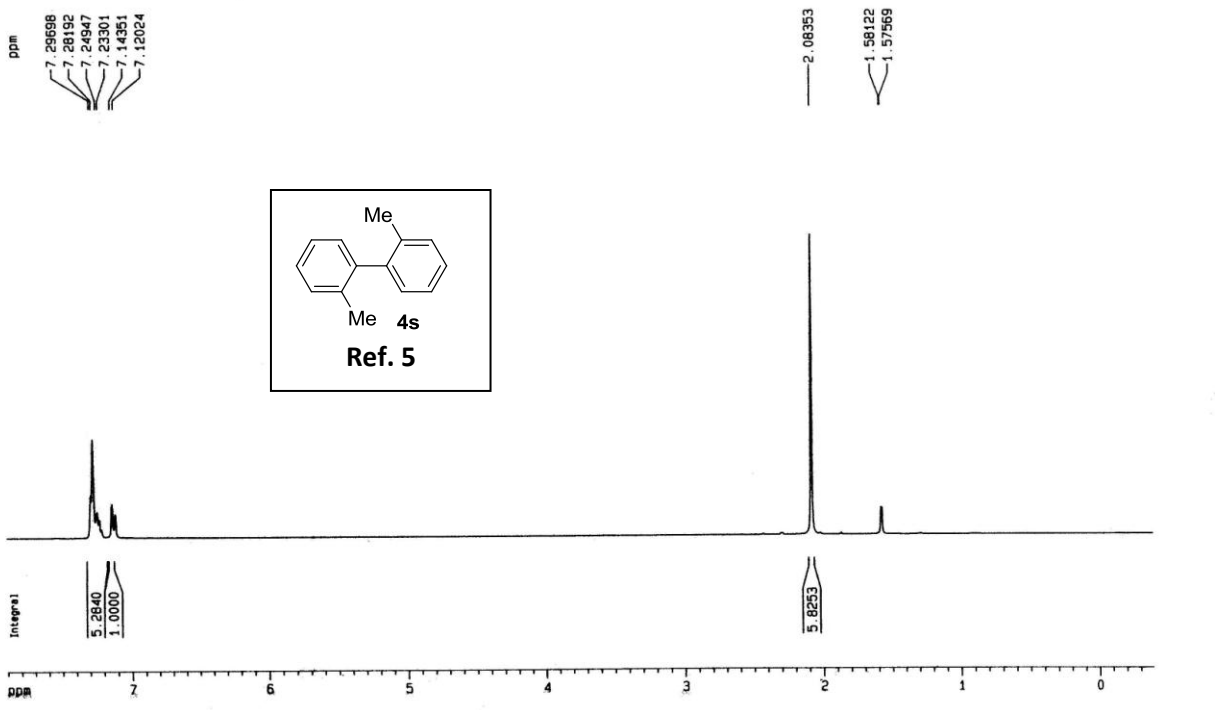


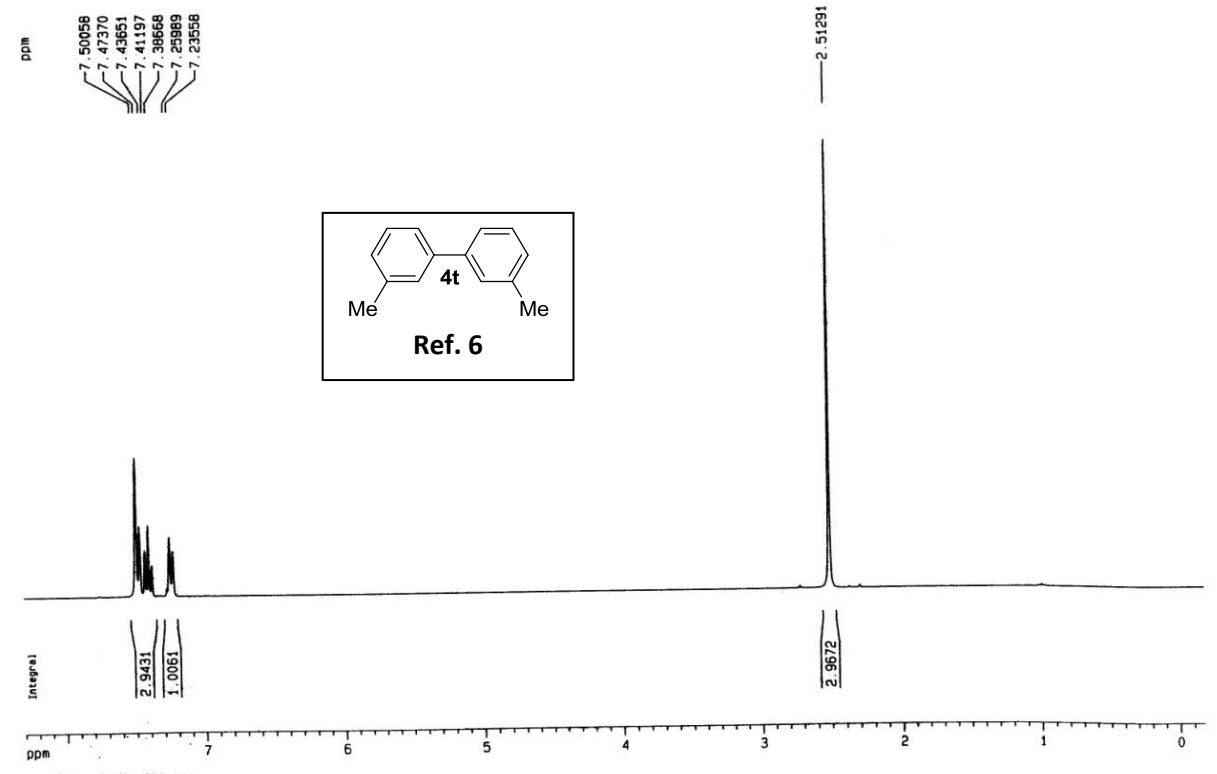


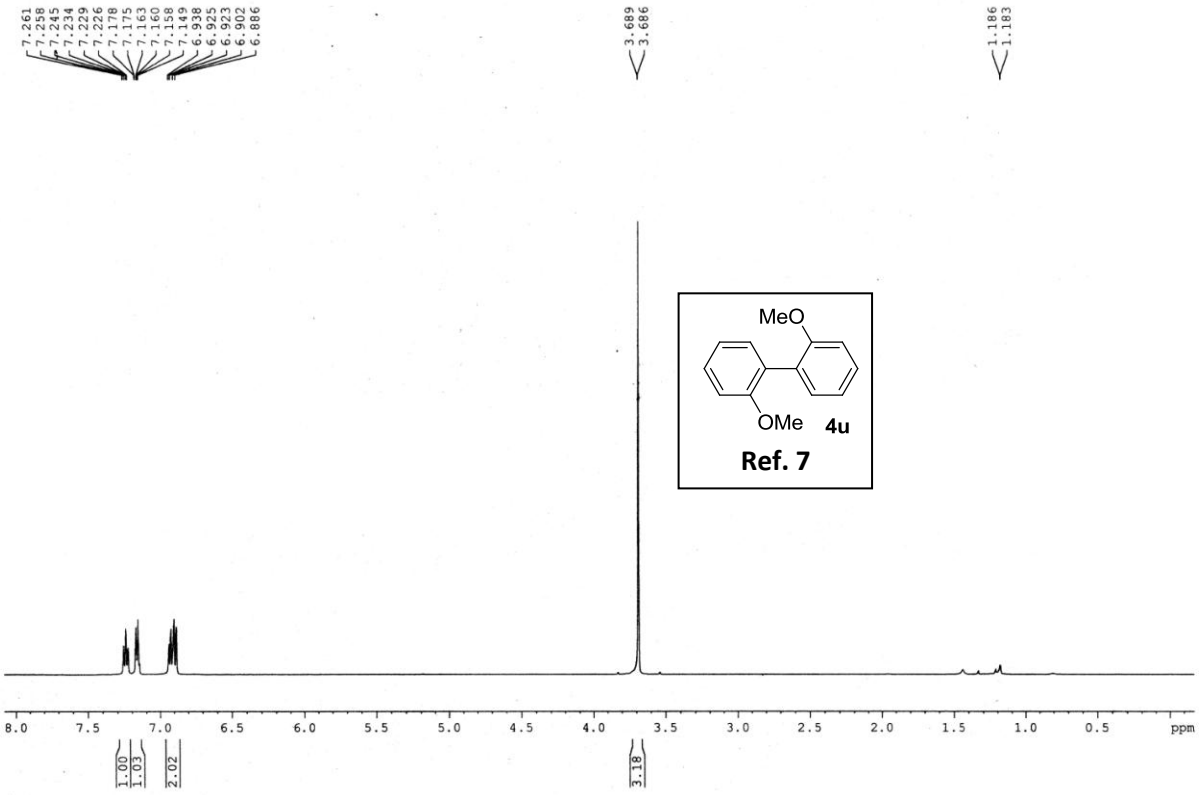


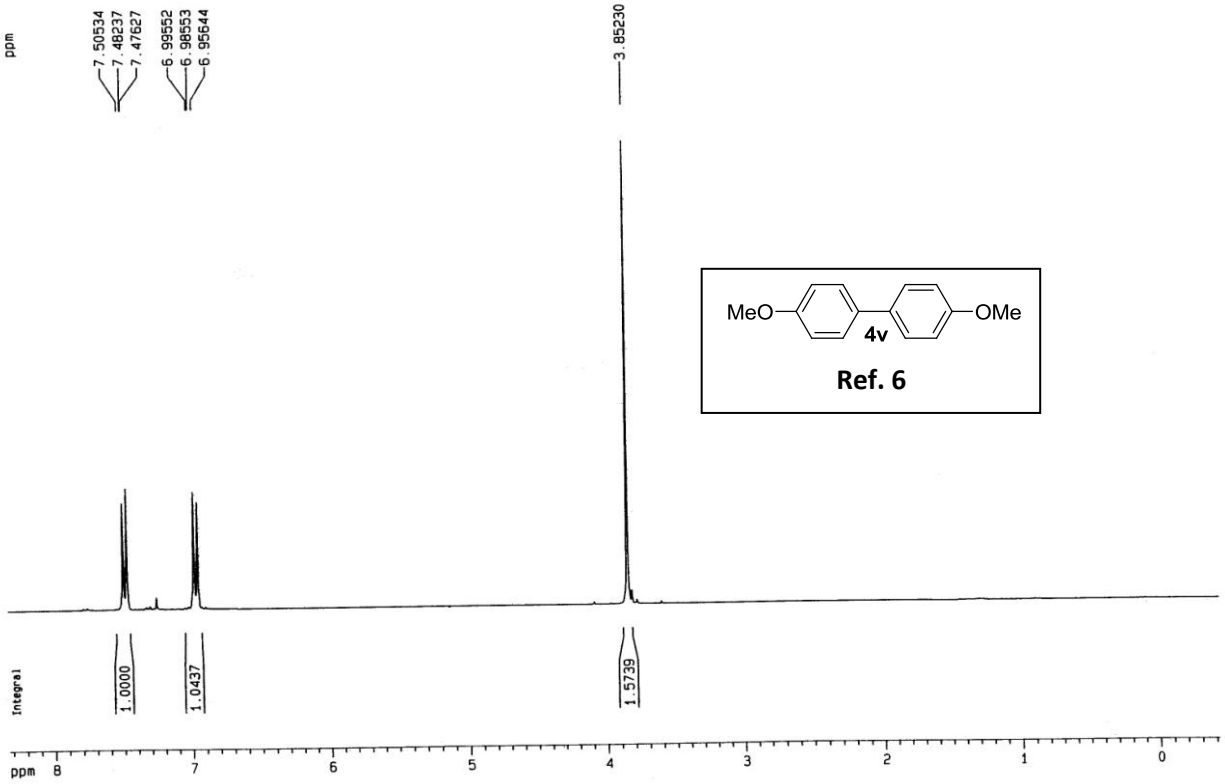
(# X indicates peak due to water in $CDCl_3$)

^{1}H-NMR spectrum of Homo-coupling Products :









References:

Ref. 1. J. I. G. Cadogan, D. M. Smith and J. B. Thomson, *J. Chem. Soc., Perkin Trans. 1*, 1972, 1296-1304.

Ref. 2. B. Baigrie, J. I. G. Cadogan, J. R. Mitchell, A. K. Robertson and J. T. Sharp, *J. Chem. Soc., Perkin Trans. 1*, 1972, 2563-2567.

Ref. 3. T. Fukutani, K. Hirano, T. Satoh and M. Miura, *J. Org. Chem.* 2011, **76**, 2867.

Ref. 4. T. Takahashi, Y. Li, P. Stepnicka, M. Kitamura, Y. Liu, K. Nakajima and M. Kotora, *J. Am. Chem. Soc.*, 2002, **124**, 576.

Ref. 5. Y. Yuan and Y. Bian, *Appl. Organomet. Chem.* 2008, **22**, 15.

Ref. 6. D. Srimani, A. Bej and A. Sarkar, *J. Org. Chem.* 2010, **75**, 4296.

Ref. 7. C. F. Nising, U. K. Schmid, M. Nieger and S. Bräse, *J. Org. Chem.* 2004, **69**, 6830.