

Supporting Information

Synthesis of Fluorescent 2,3,5,6-Tetra(alkynyl)pyridines by Site-Selective Sonogashira-Reactions of 2,3,5,6-Tetrachloropyridines

Peter Ehlers,^{a,b} Andranik Petrosyan,^{a,b,d} Antje Neubauer,^c Timo Bröse,^a Stefan Lochbrunner,^c
Tariel V. Ghochikyan,^d Ashot S. Saghyan,^{d,e} Peter Langer*^{a,b}

^a Institut für Chemie, Universität Rostock, Albert Einstein Str. 3a, 18059 Rostock, Germany

^b Leibniz-Institut für Katalyse an der Universität Rostock e.V., Albert Einstein Str. 29a, 18059 Rostock, Germany

^c Institut für Physik, Universität Rostock, Universitätsplatz 3, 18051 Rostock, Germany

^d Faculty of Chemistry, Yerevan State University, Alex Manoogian 1, 0025 Yerevan, Armenia

^e Scientific and Production Center "Armbiotechnology" of NAS RA, Gyurjyan Str. 14, 0056 Yerevan, Armenia

E-Mail: peter.langer@uni-rostock.de

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General Information

All reactions were carried out under Argon atmosphere. Unless otherwise cited, all chemicals are commercially available and were used without further purification. Column chromatography was performed using Silica gel (0.043-0.06 mm).

NMR data were recorded on 300 and 400 MHz spectrometers. ^{13}C and ^1H NMR spectra were referenced to signals of deuterium-solvents and residual protonated solvents, respectively. Peak characterization: s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet of doublet, q = quartet, quint = quintet, sext = sextet, sept = septet.

Infrared Spectra were recorded with ATR sampling technique for solids as well as liquids. Signal characterization: w = weak, m = medium, s = strong.

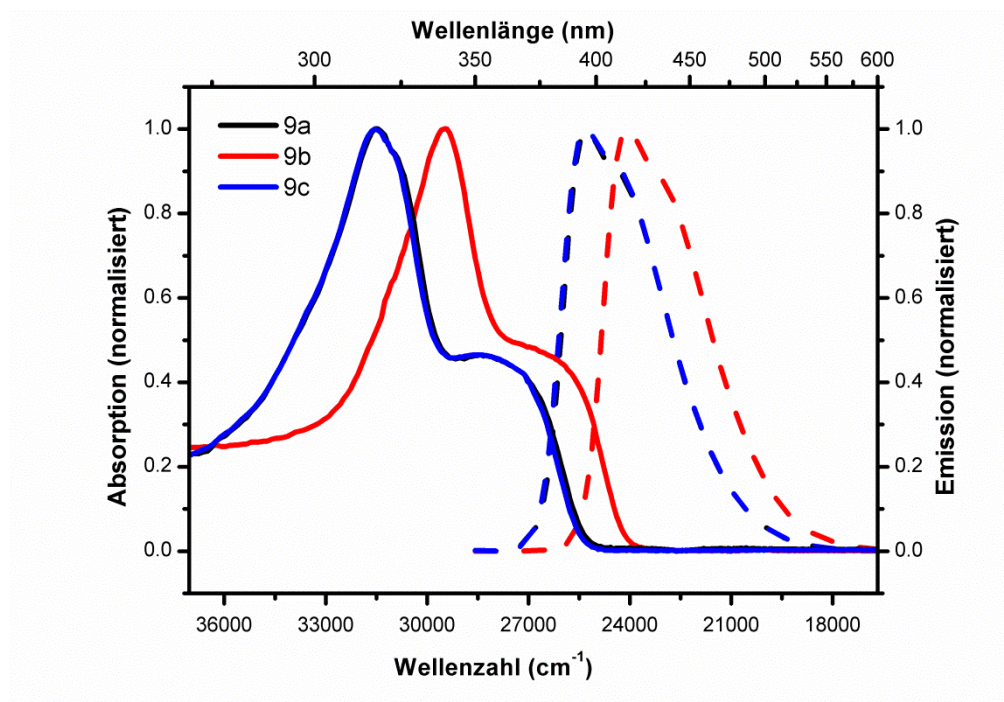
Gas-chromatography-mass-analysis was carried out with a Mass Selective Detector (EI) and HP-5 capillary column using helium carrier gas. Only the measurements with an average deviation from the theoretical mass of $\pm 2\text{mDa}$ were accounted as correct for the High Resolution MS (HRMS).

For the absorption spectra dichloromethane was used as solvent and a concentration of $5 \times 10^{-6}\text{ M}$ was applied for the quantitative determination of the molar extinction coefficient. A solution of quinine sulphate in 0.05 M sulphuric acid was applied as standard for the measurement of the fluorescence quantum yields ($\phi_f = 0.52$).¹ Sample and standard were excited with an absorbance of ~ 0.1 in air-equilibrated solution.

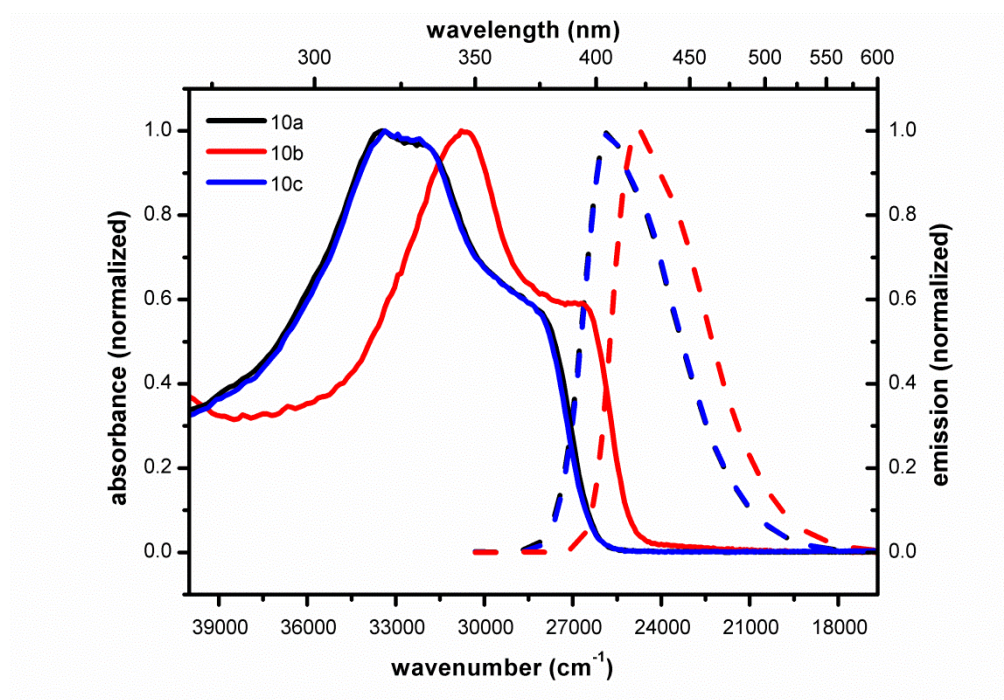
¹ Meech, S. R., Phillips, D. J. *Photochem.* **1983**, 23, 193-217

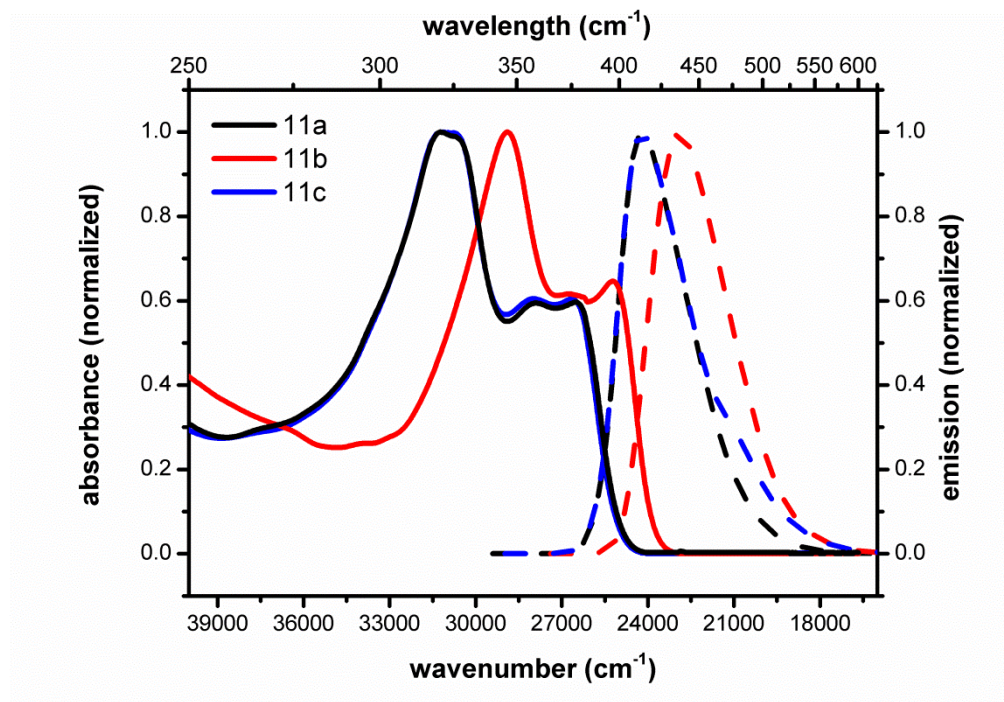
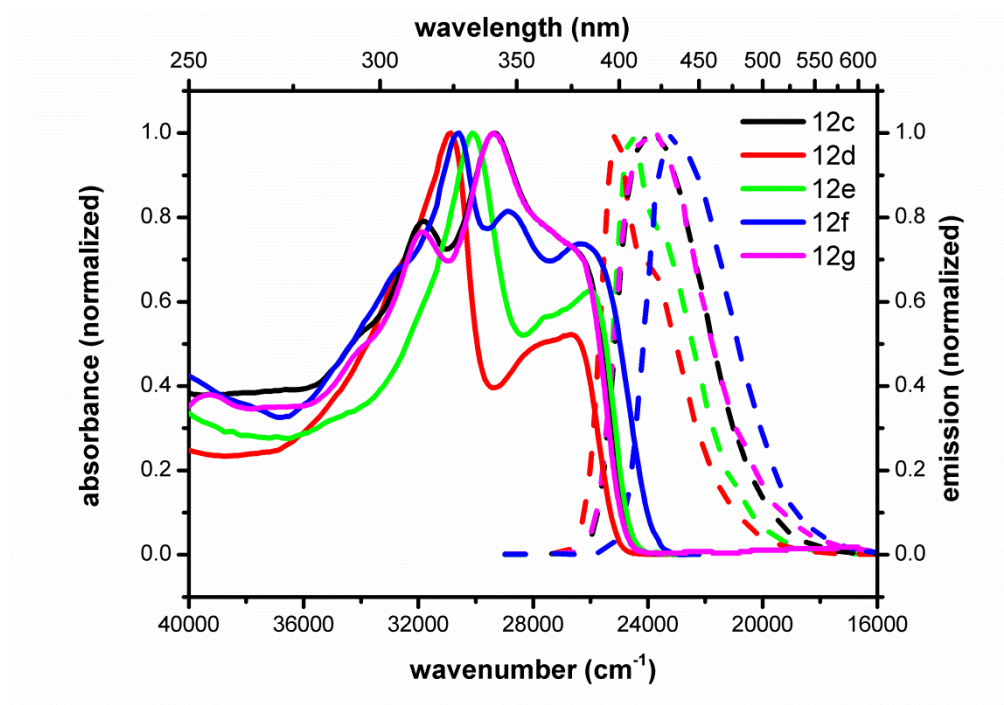
UV/Vis and Fluorescence spectra

S1 – Comparison of different 4-isopropoxy-2,3,5,6-tetraalkynylpyridines



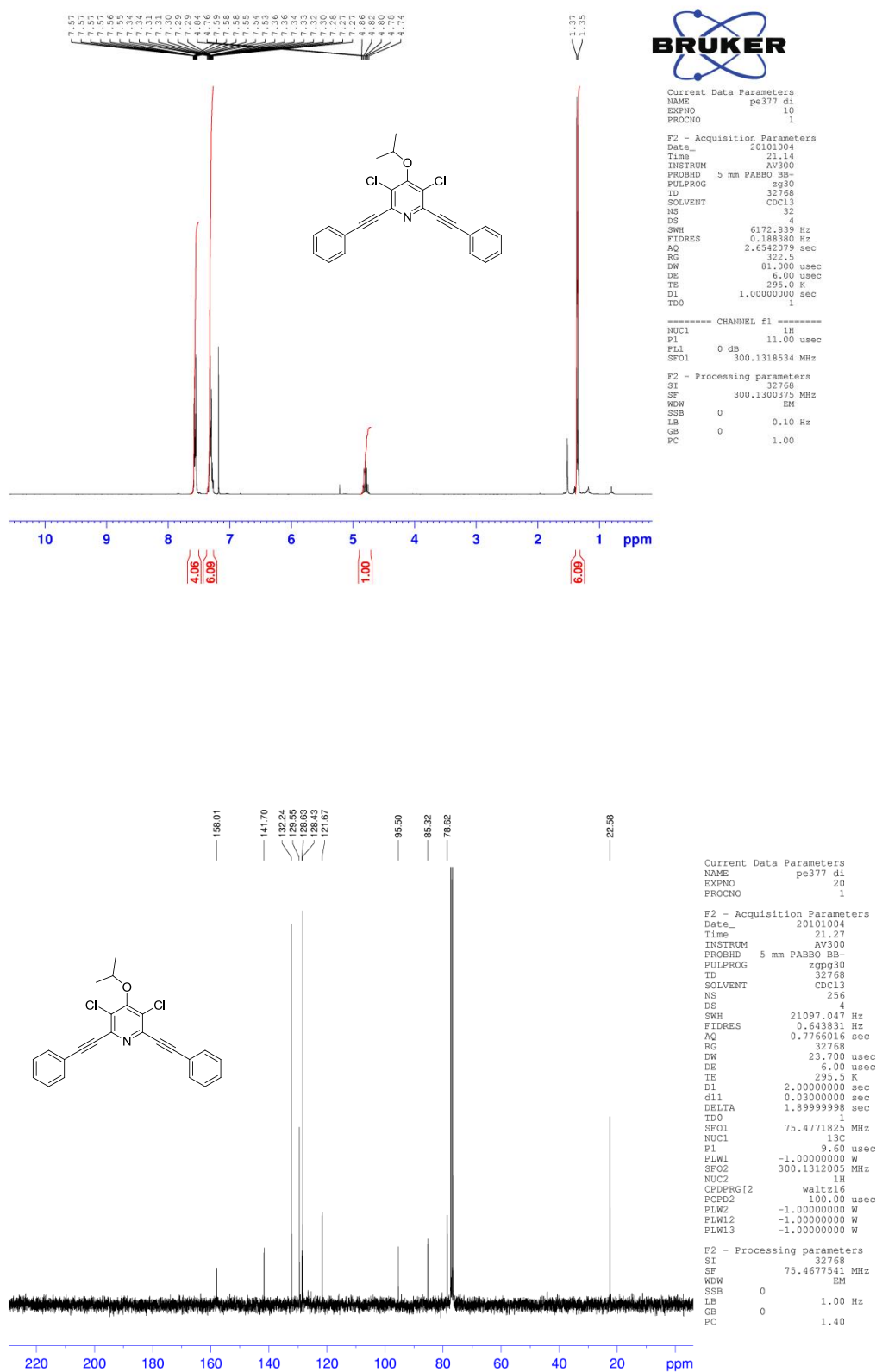
S2– Comparison of different 4-(4-methoxyphenyl)-2,3,5,6-tetraalkynylpyridines

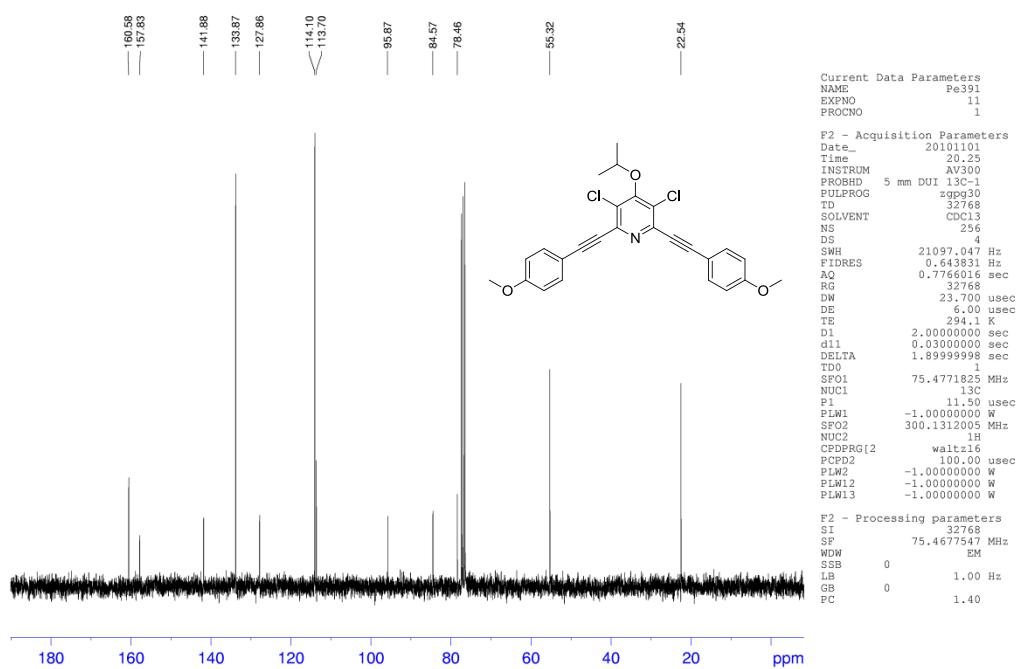
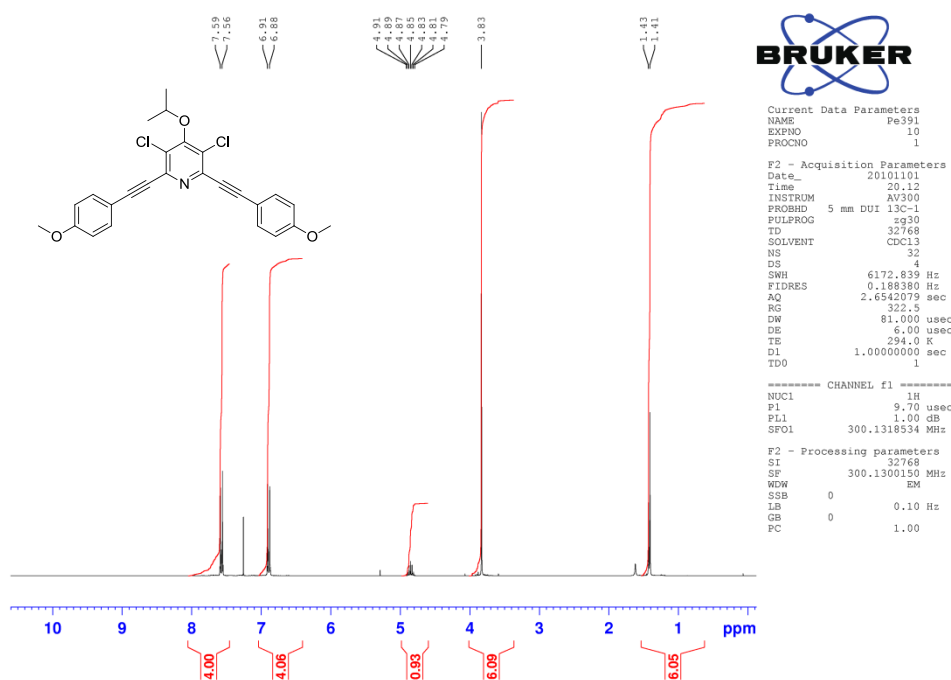


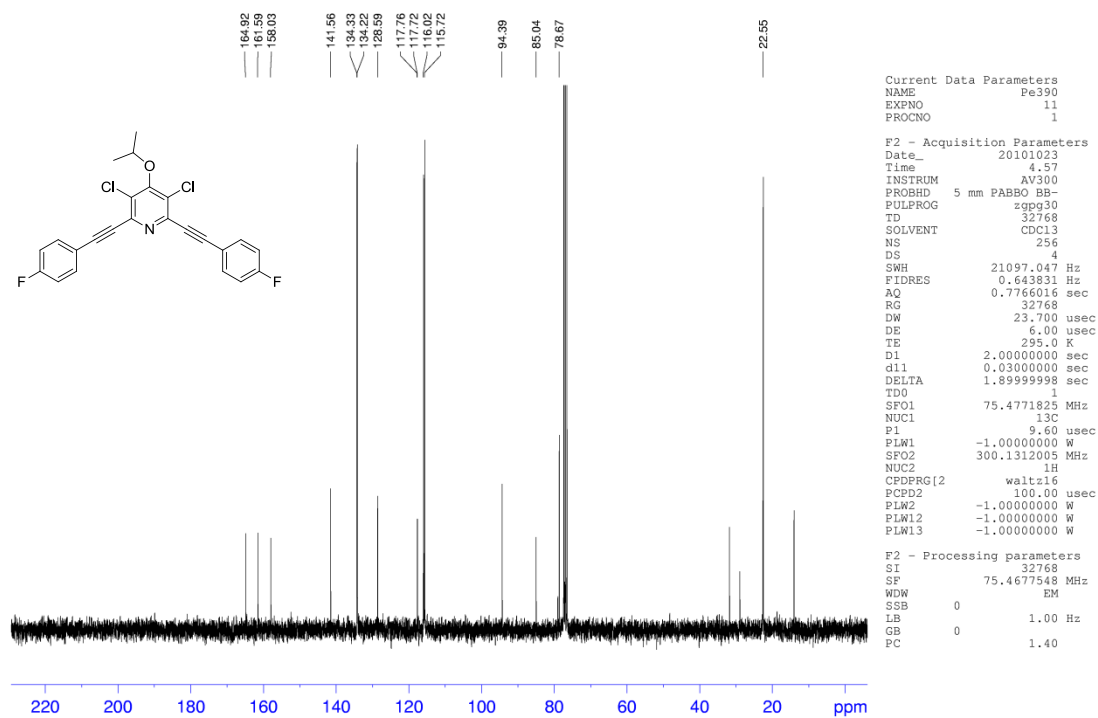
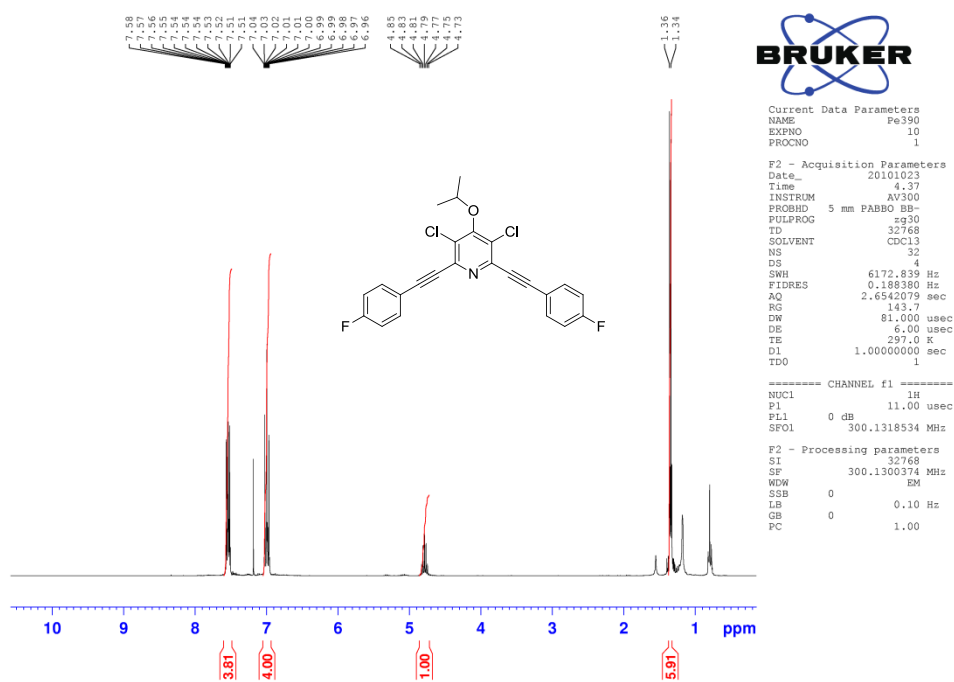
S3 – Comparison of different 4-(4-trifluoromethylphenyl)-2,3,5,6-tetraalkynylpyridines**S4 – Comparison of 2,3,5,6-tetraalkynylpyridines containing different alkynyl moieties**

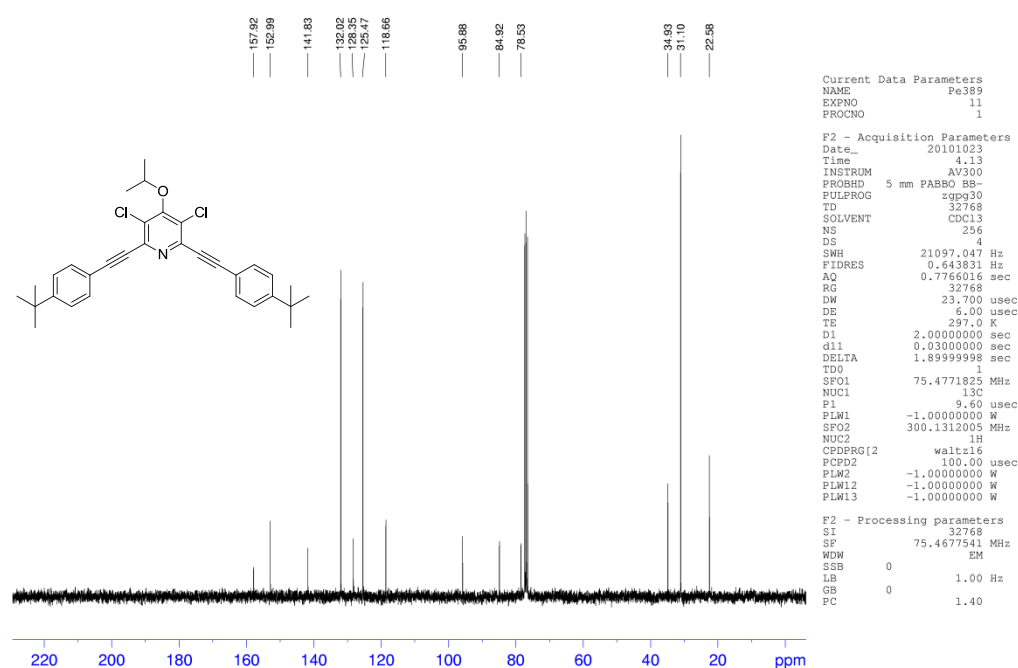
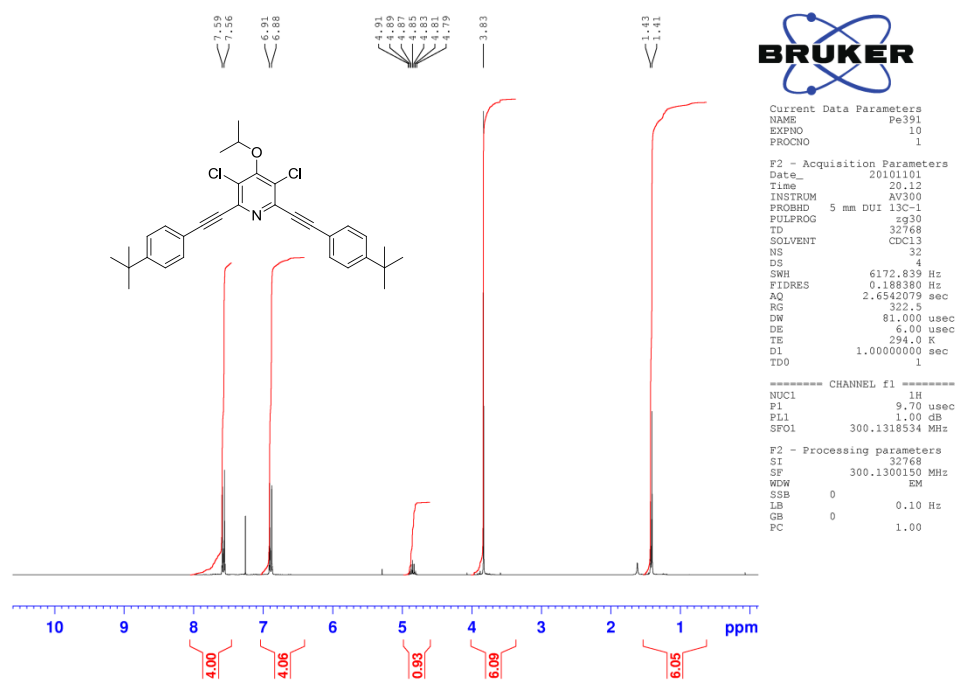
^1H - and ^{13}C -NMR spectra

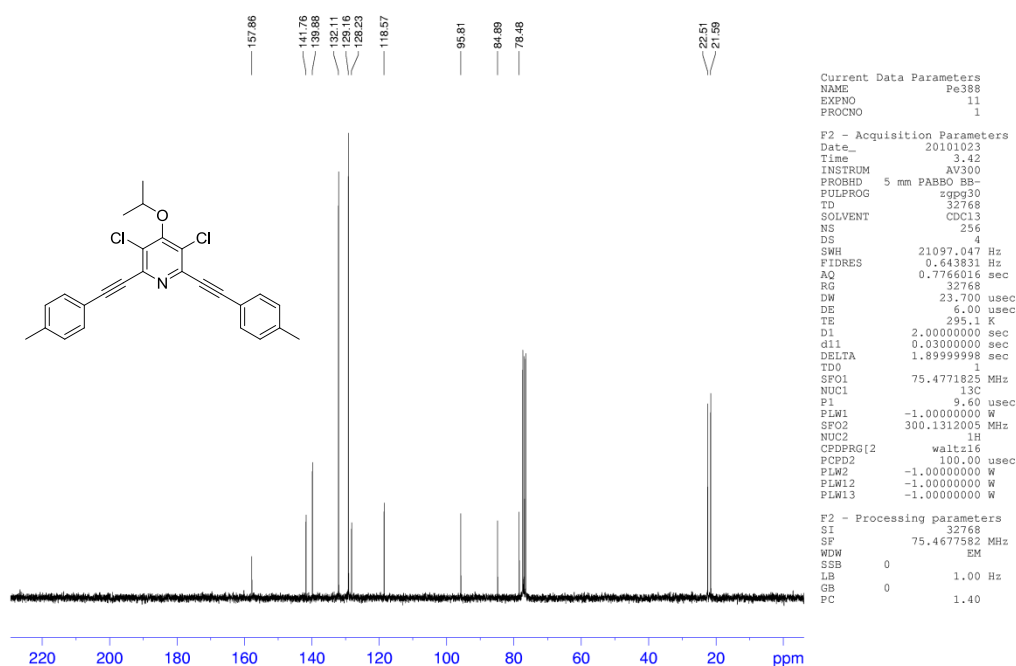
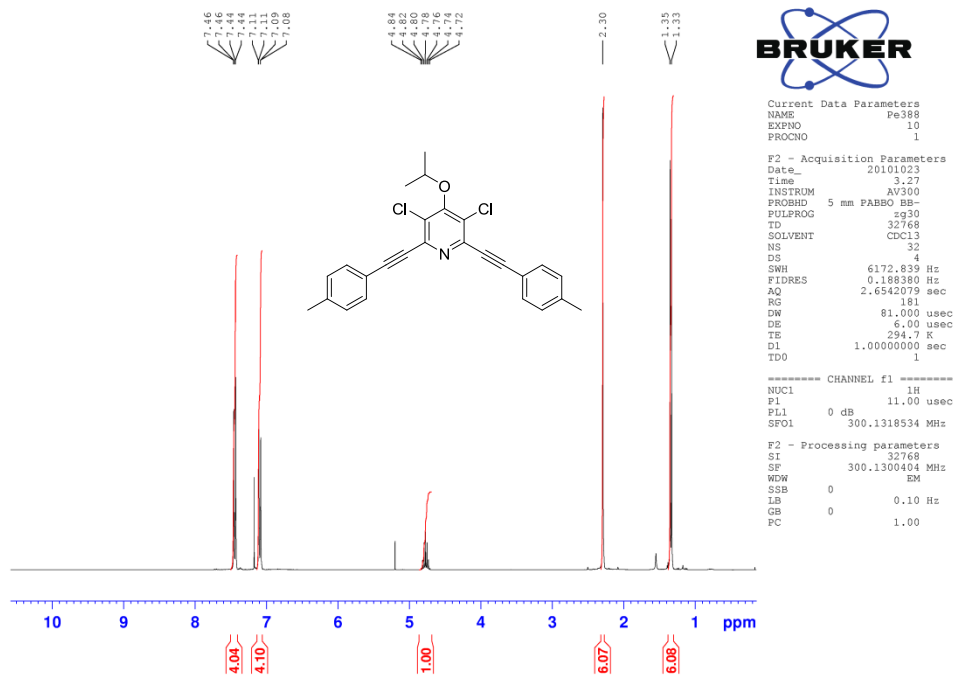
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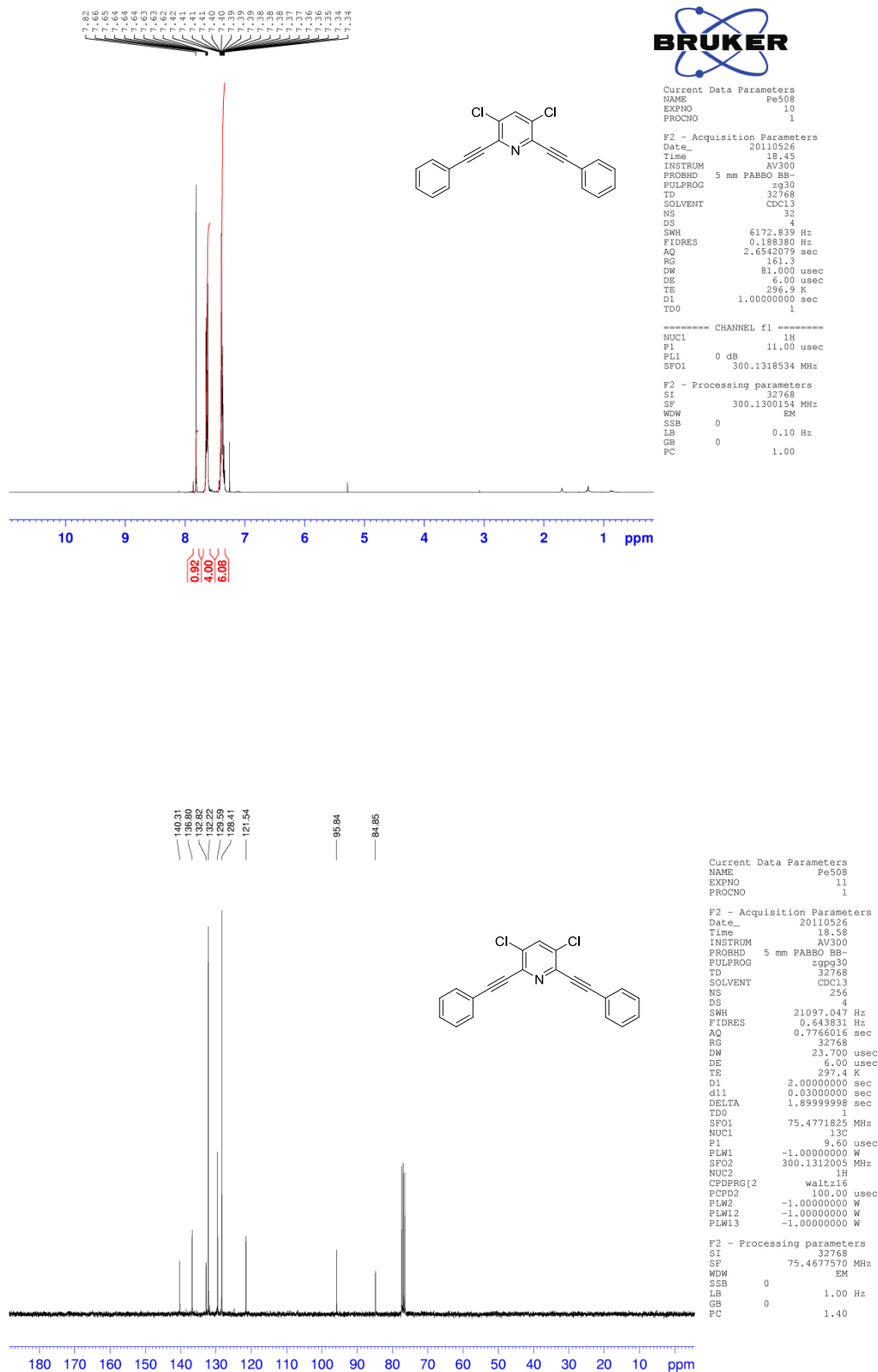


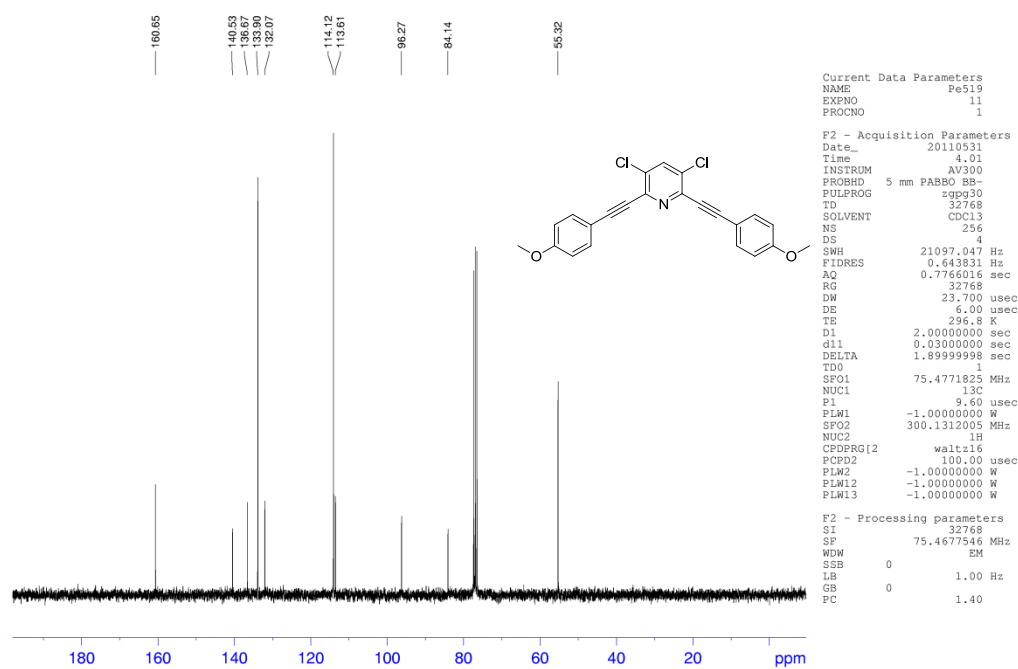
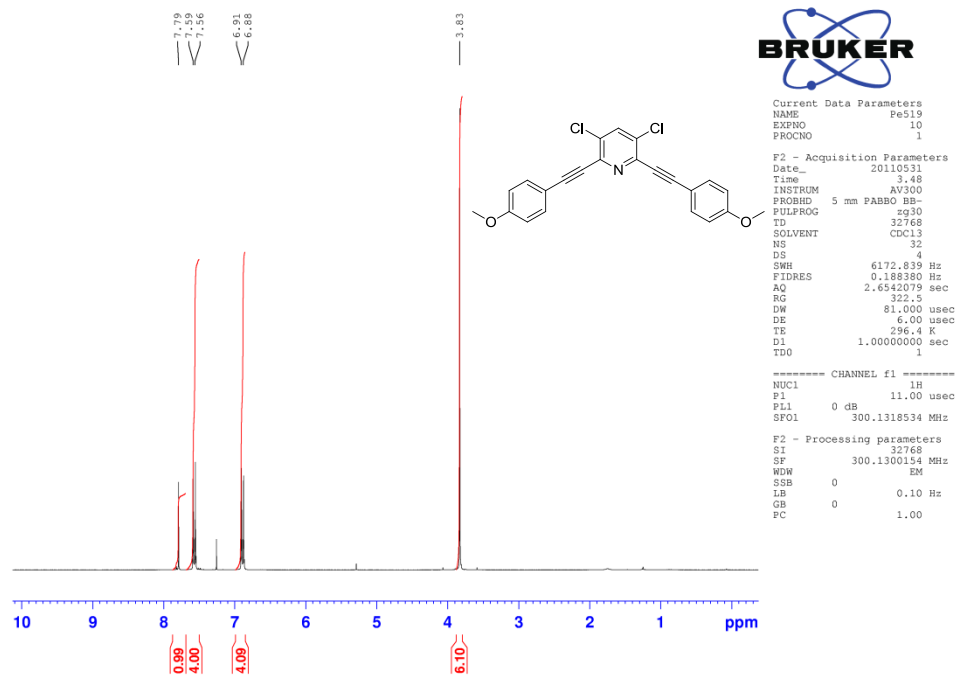
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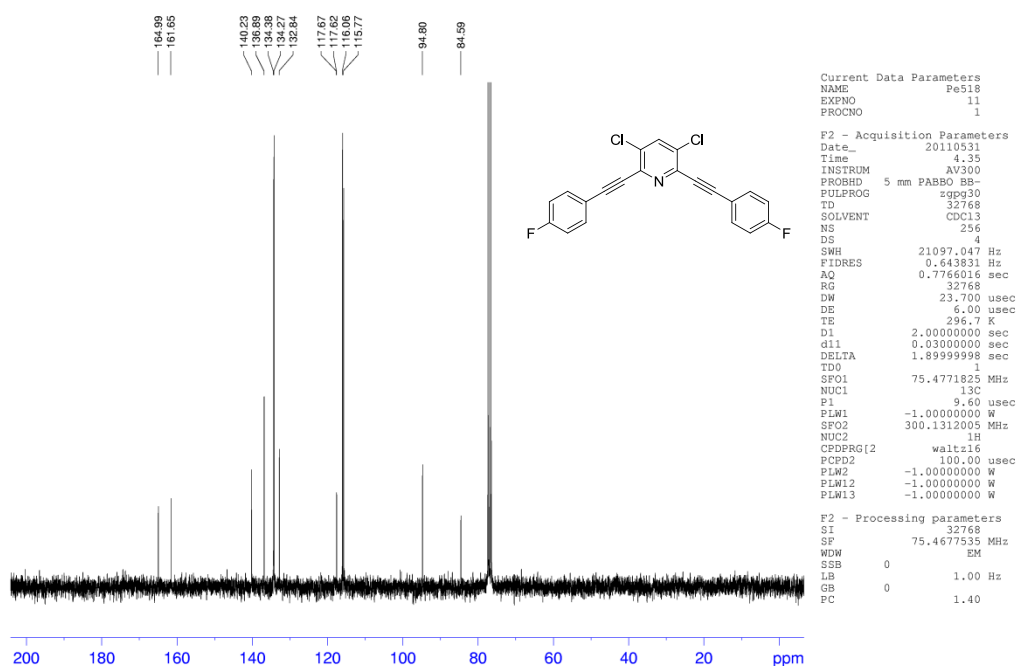
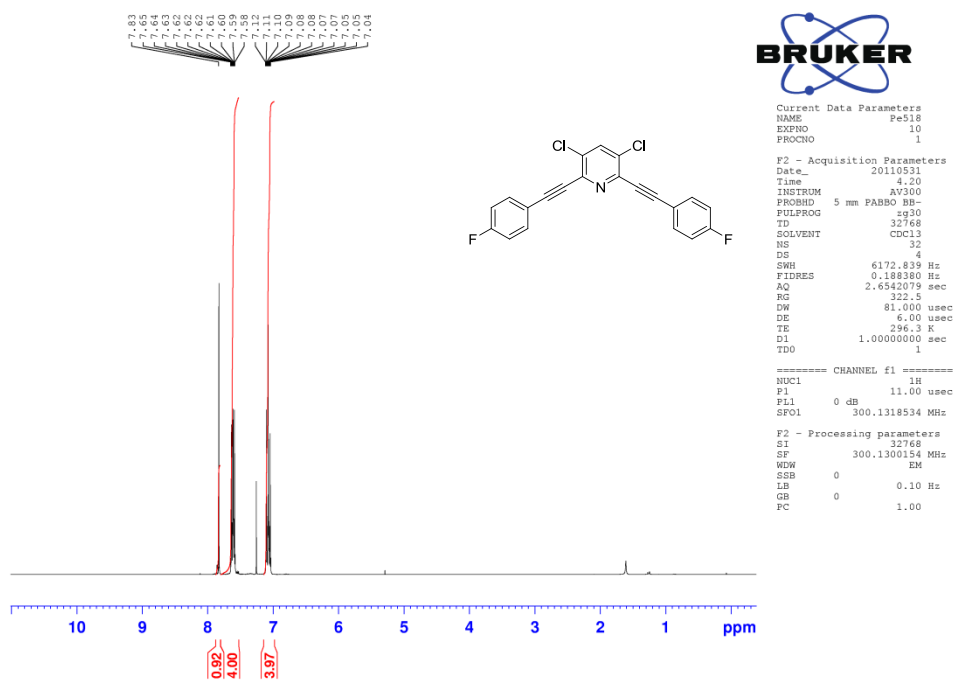
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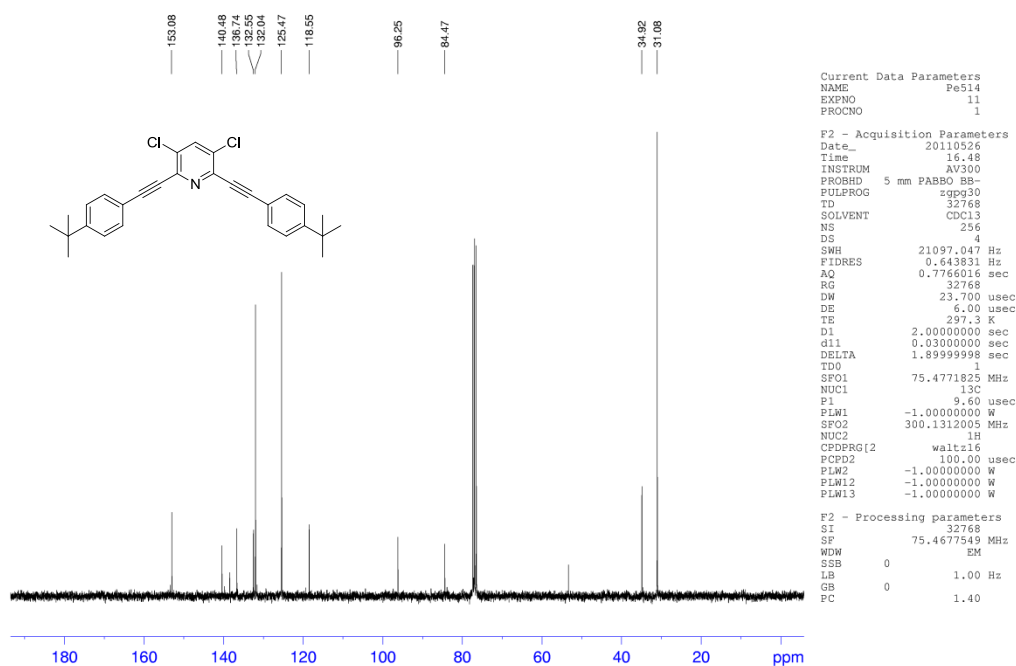
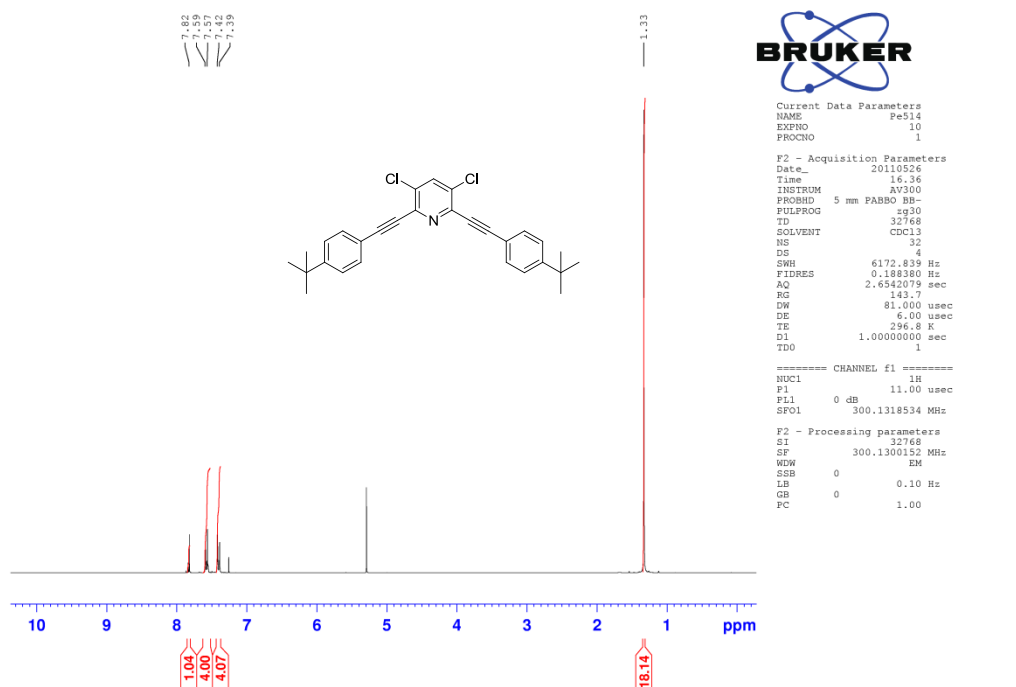
¹H- and ¹³C-NMR spectra of 2d

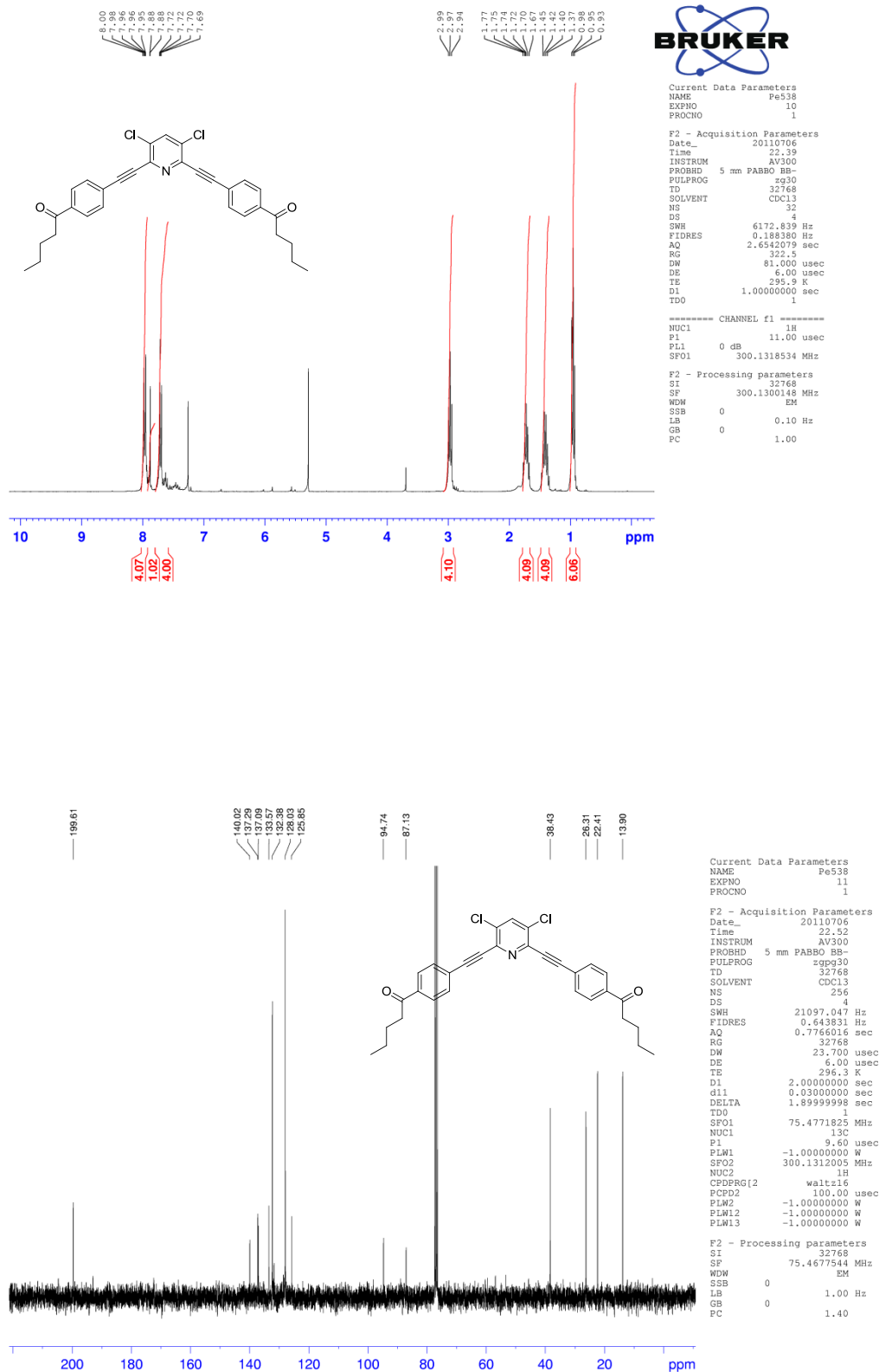
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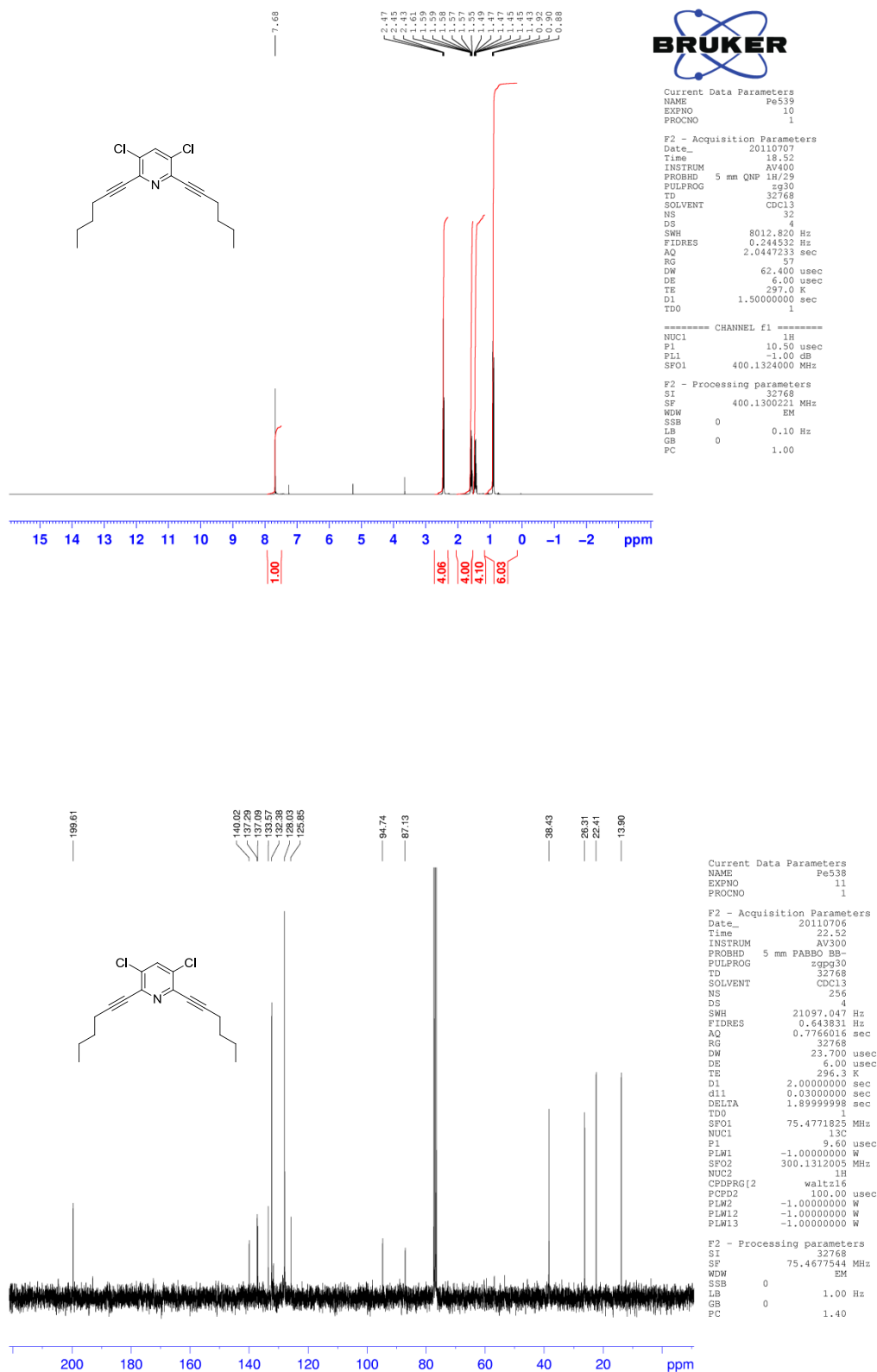
¹H- and ¹³C-NMR spectra of 4a

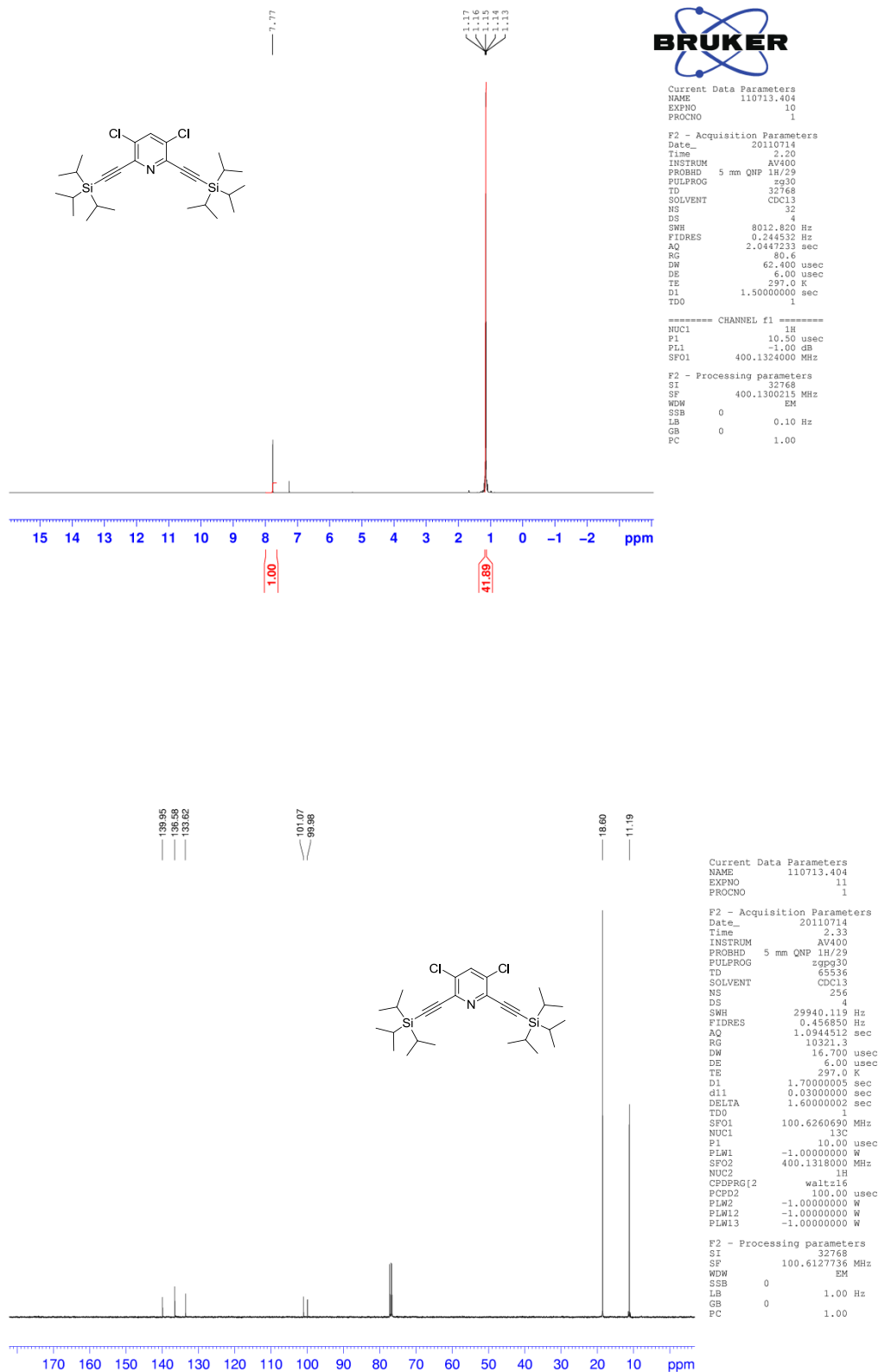
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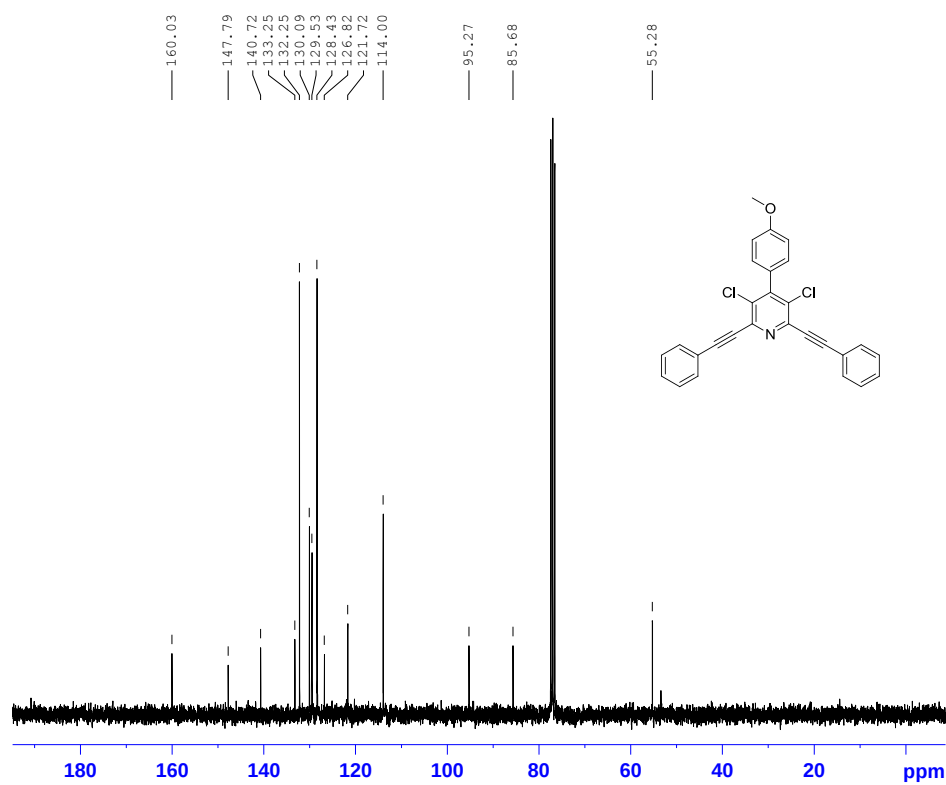
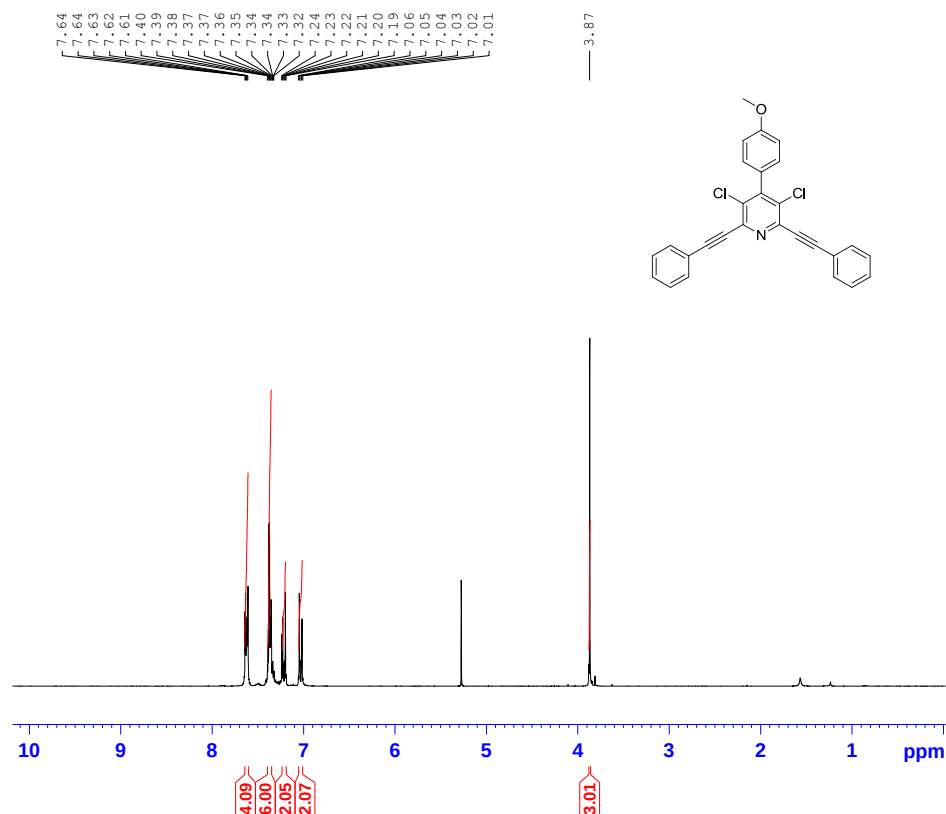
¹H- and ¹³C-NMR spectra of 4c

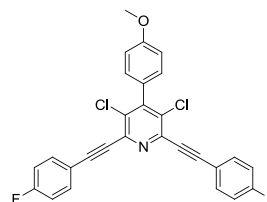
¹H- and ¹³C-NMR spectra of 4d

¹H- and ¹³C-NMR spectra of 4e

¹H- and ¹³C-NMR spectra of 4f

¹H- and ¹³C-NMR spectra of 4g

¹H- and ¹³C-NMR spectra of 6a



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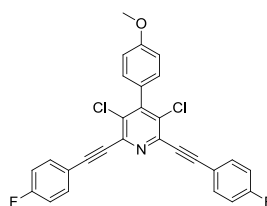
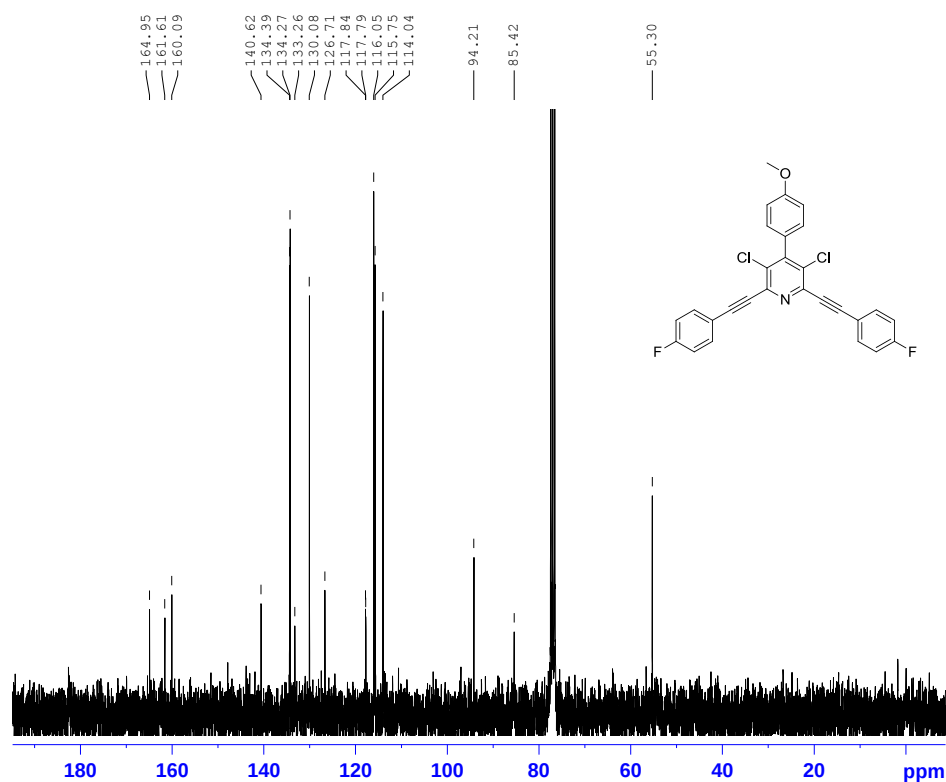
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PL1                 0 dB
SFO1                300.1318534 MHz
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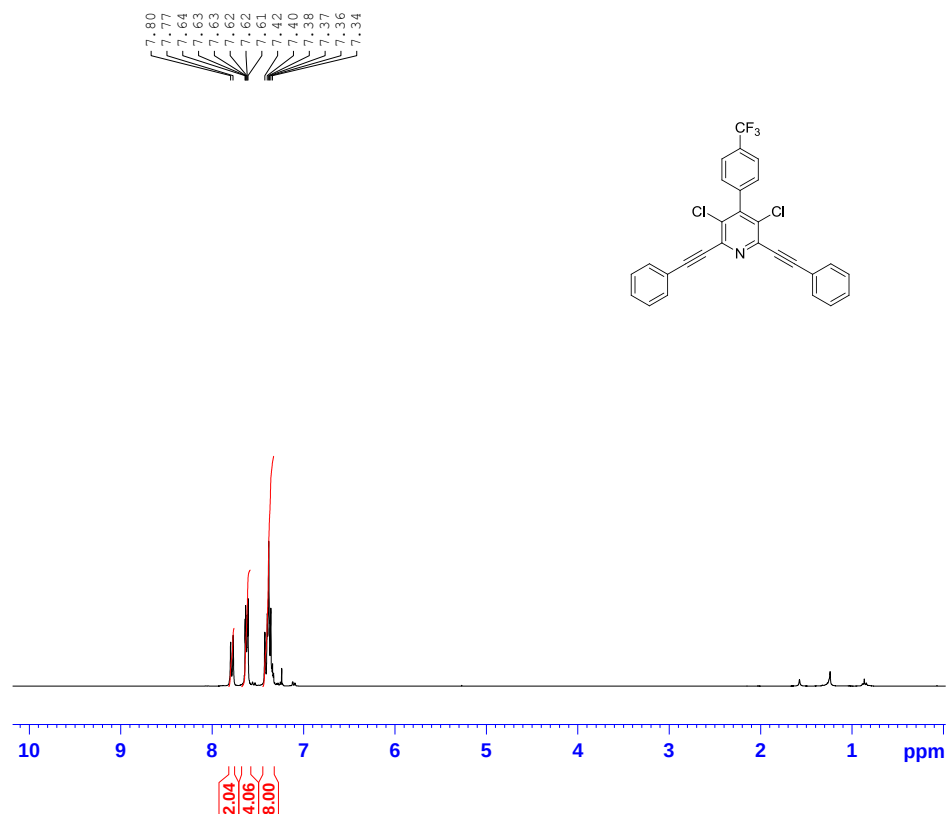
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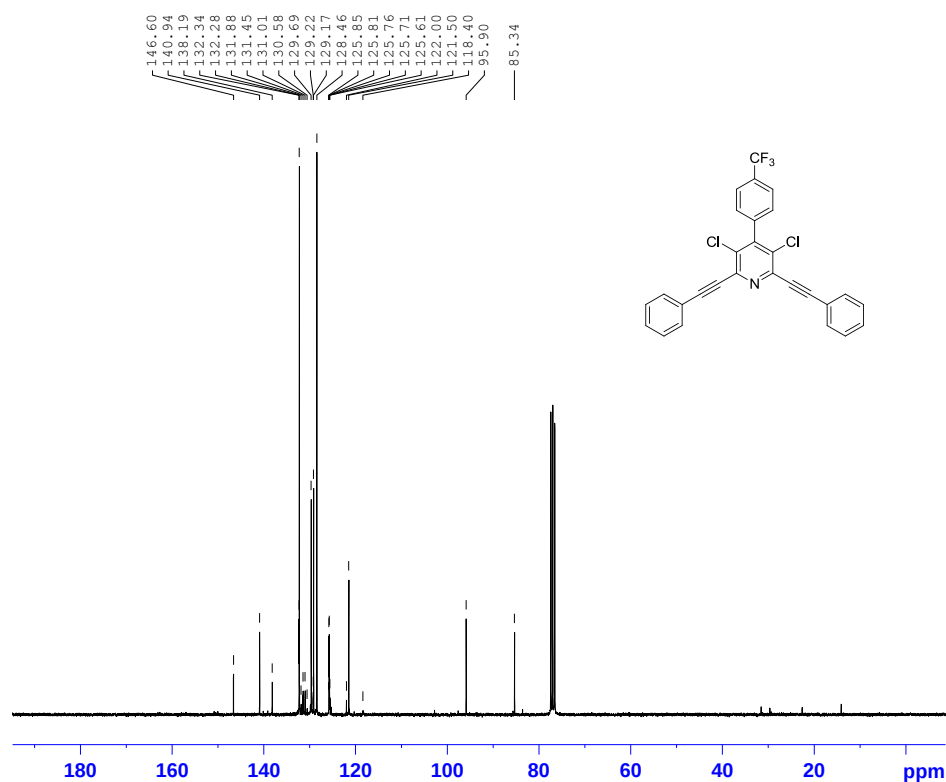
¹H- and ¹³C-NMR spectra of 8a

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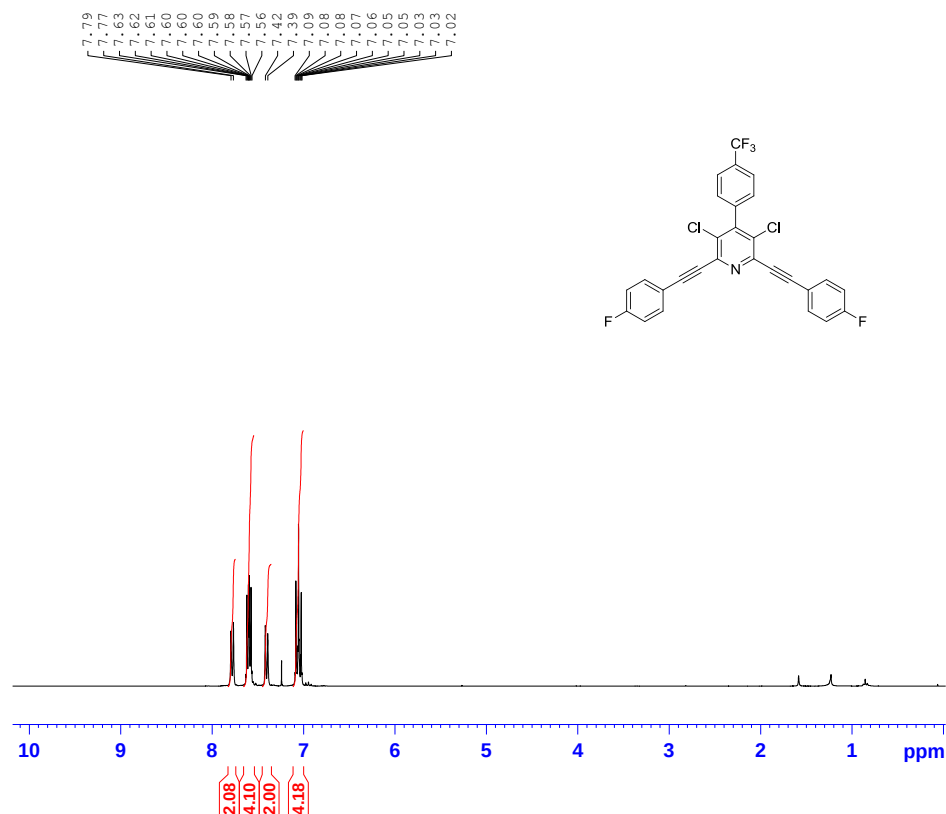
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NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
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PCPD2 100.00 usec
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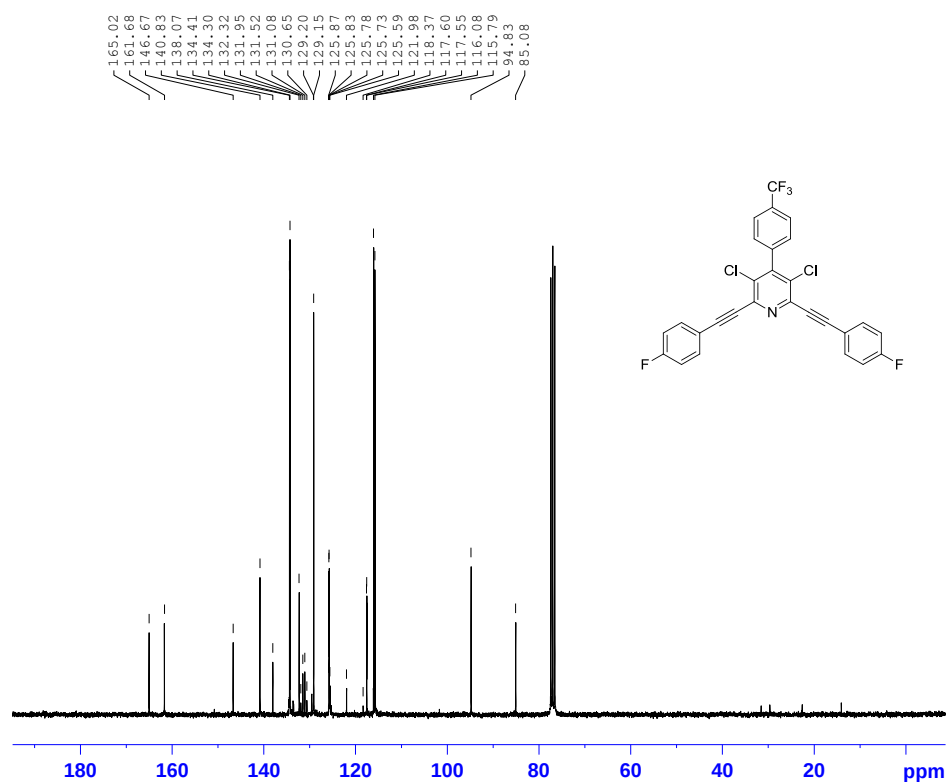
¹H- and ¹³C-NMR spectra of 8b

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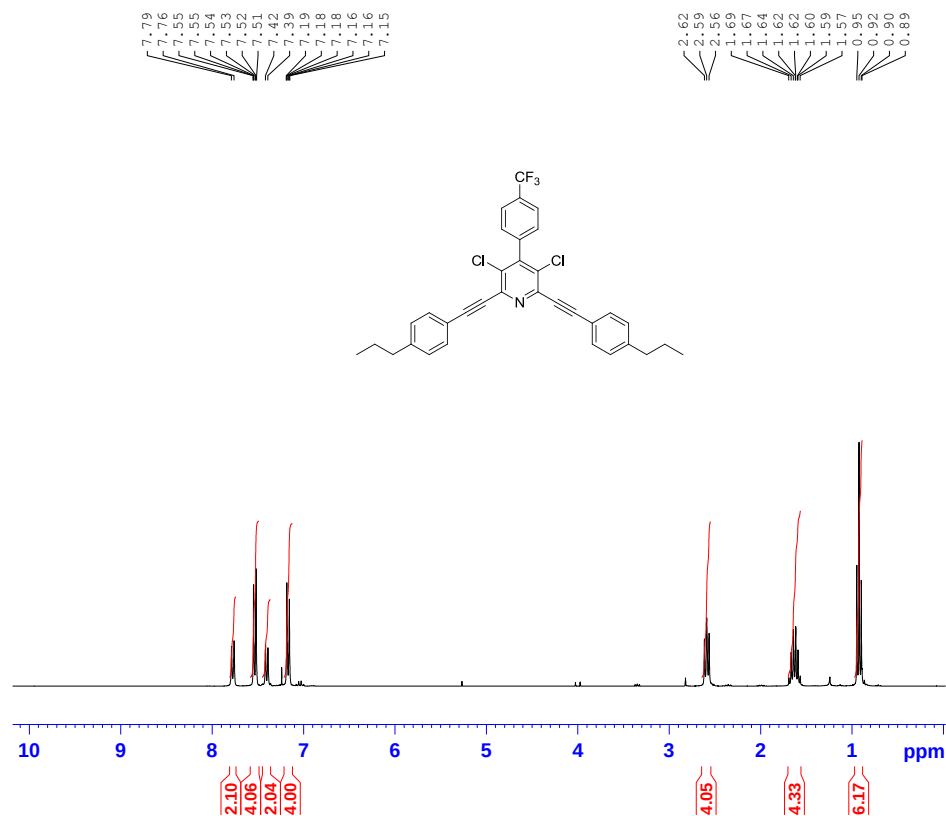
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Current Data Parameters
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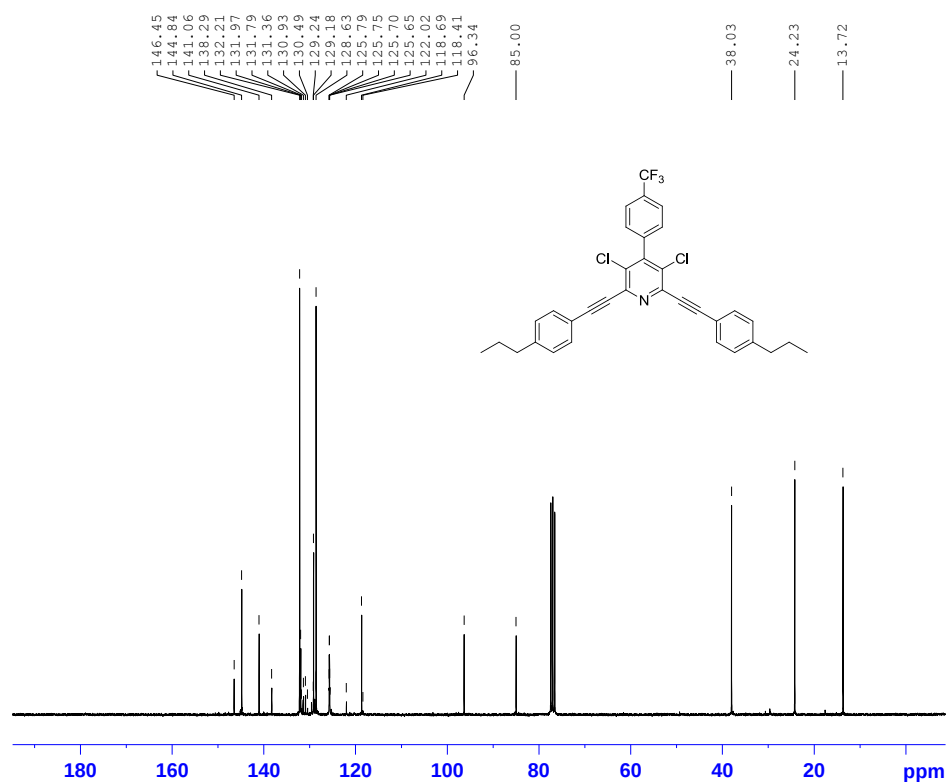
¹H- and ¹³C-NMR spectra of 8c

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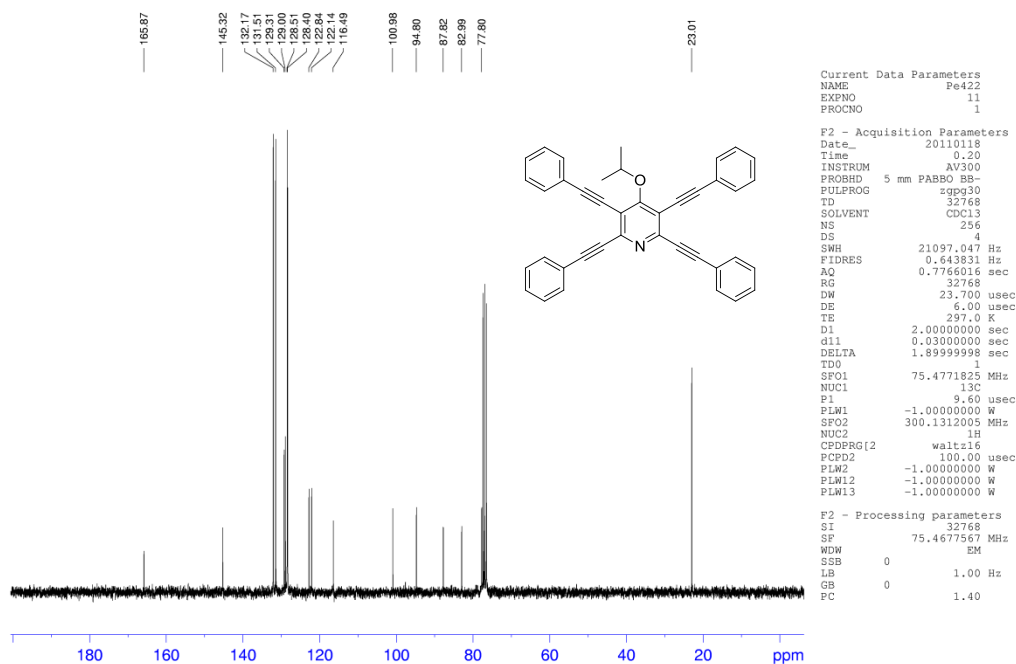
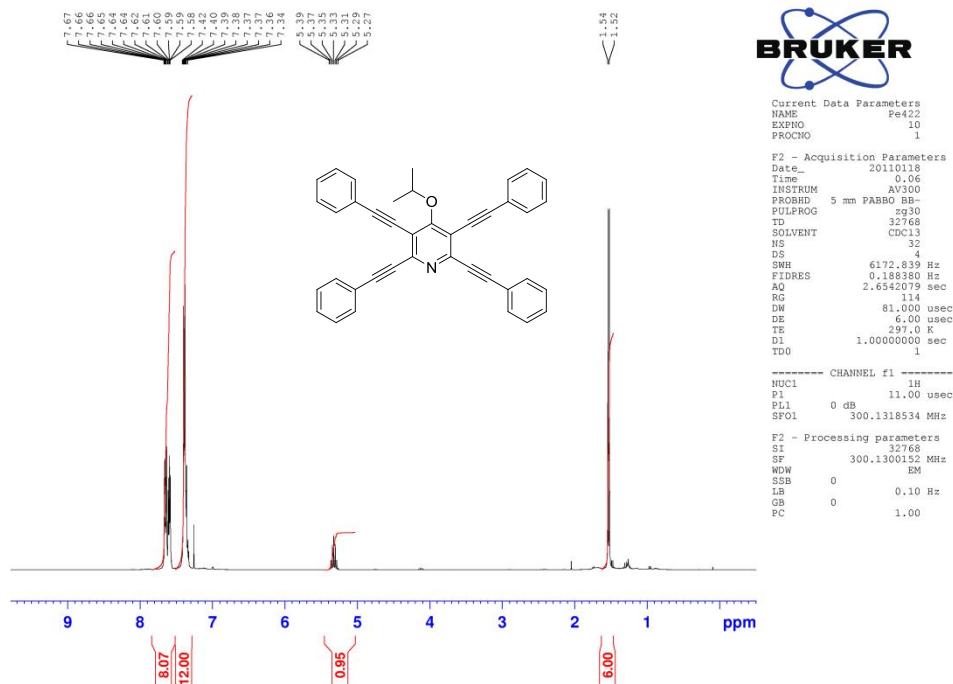
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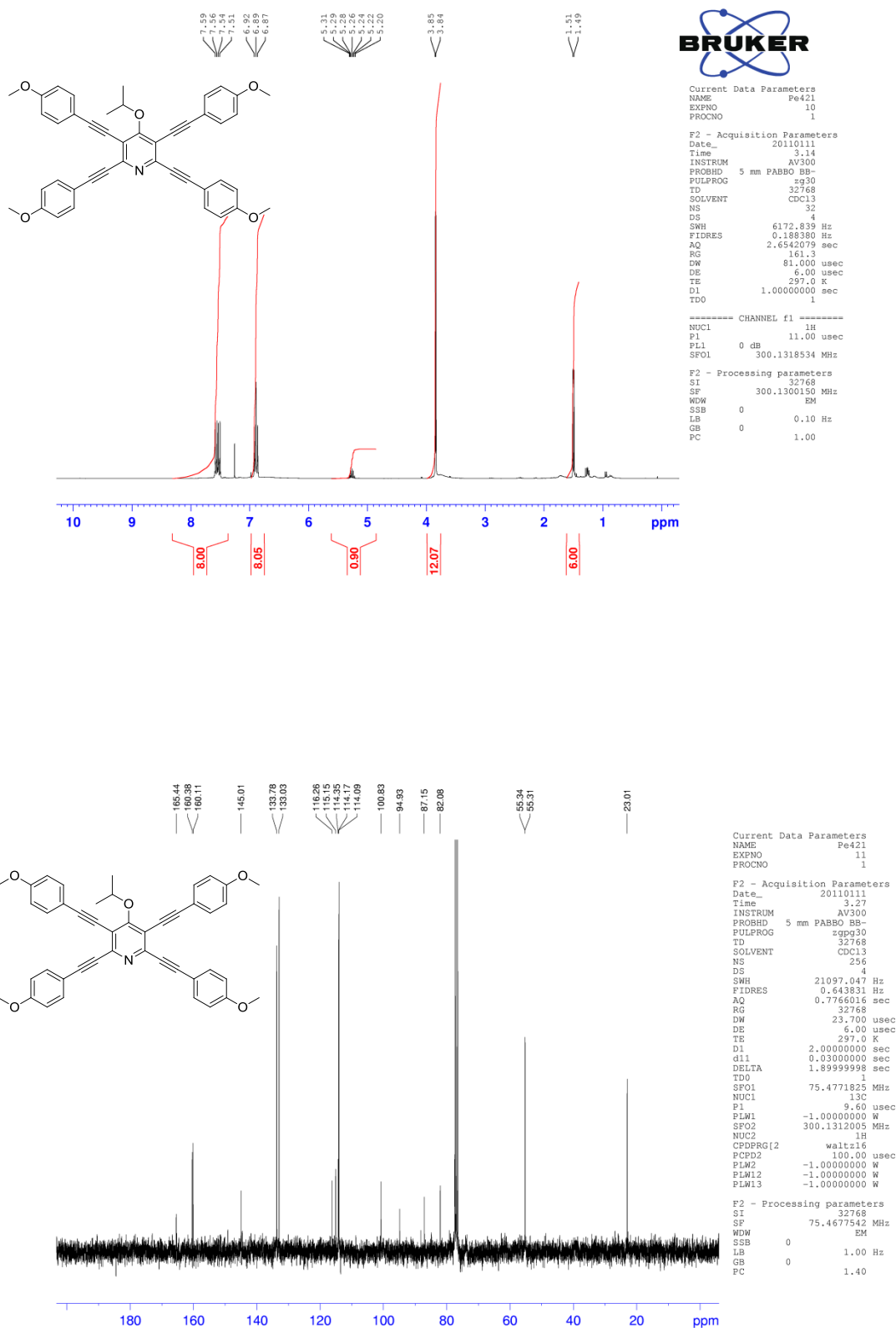


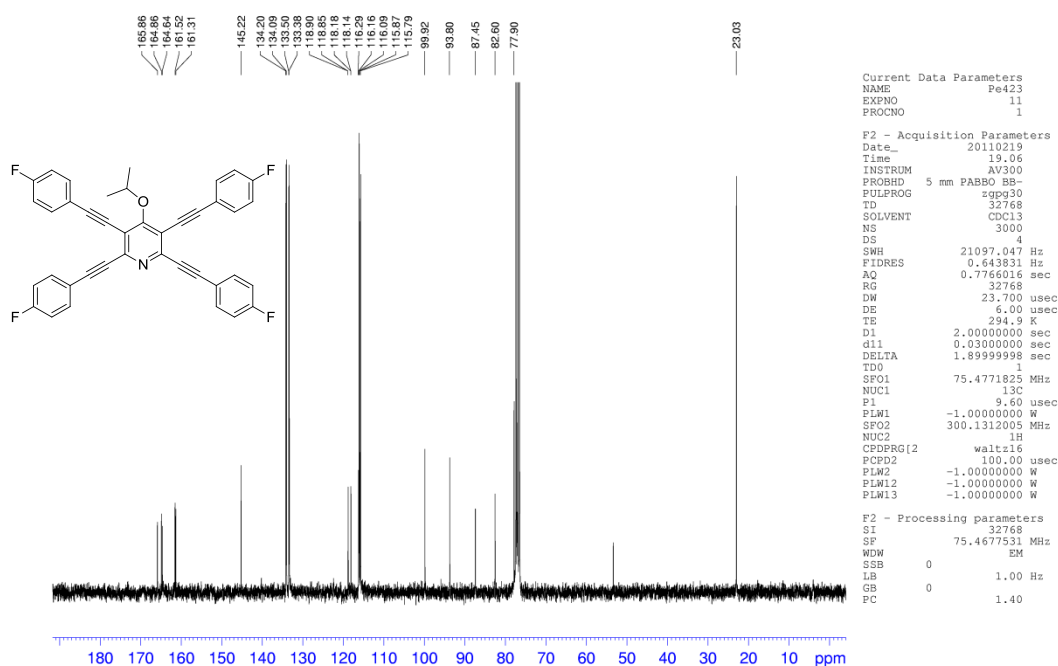
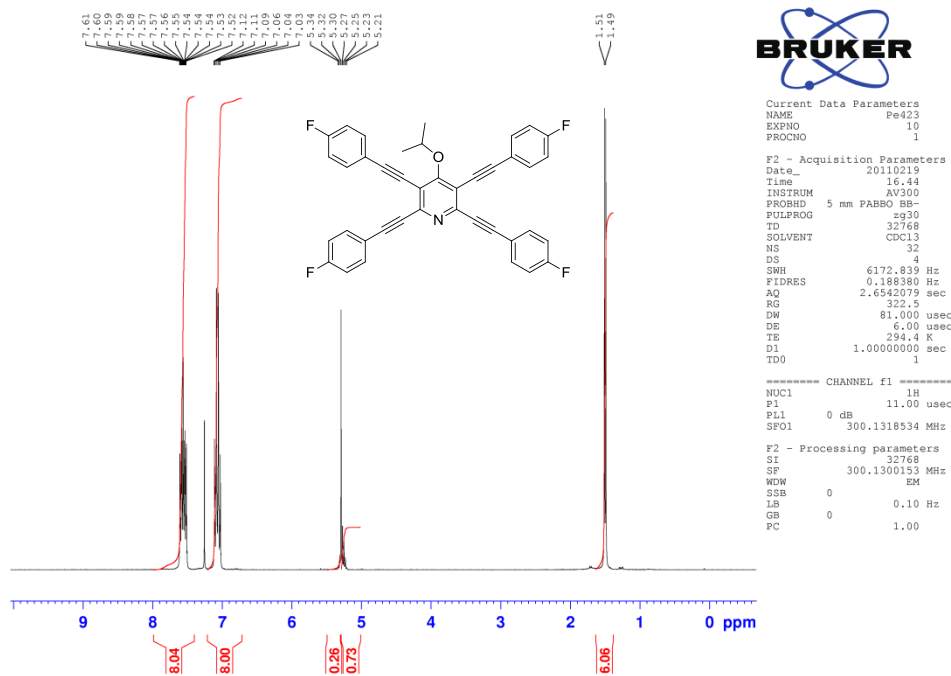
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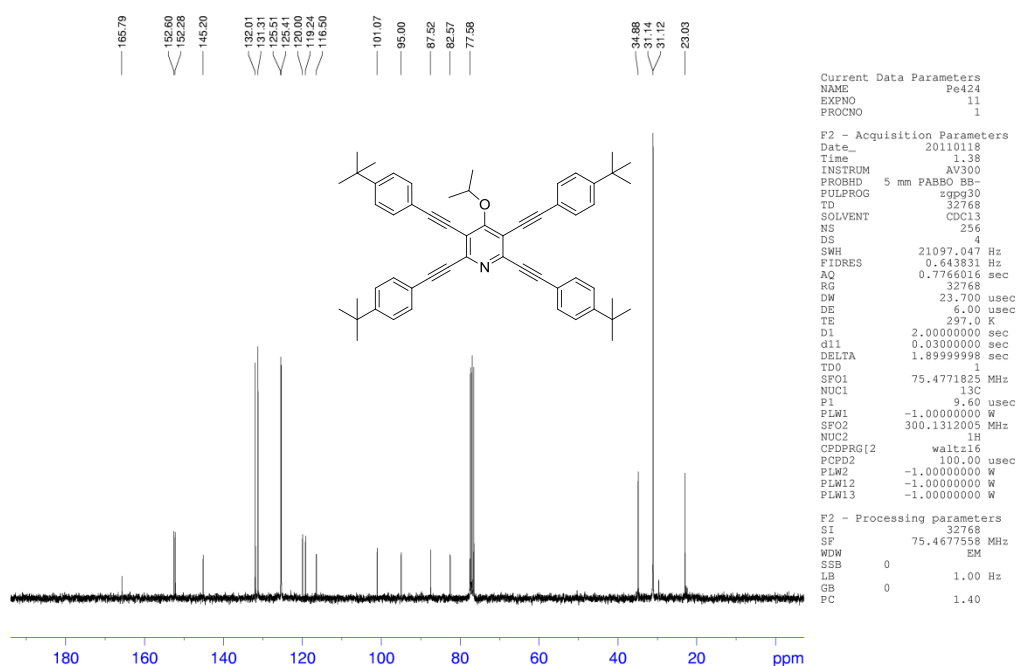
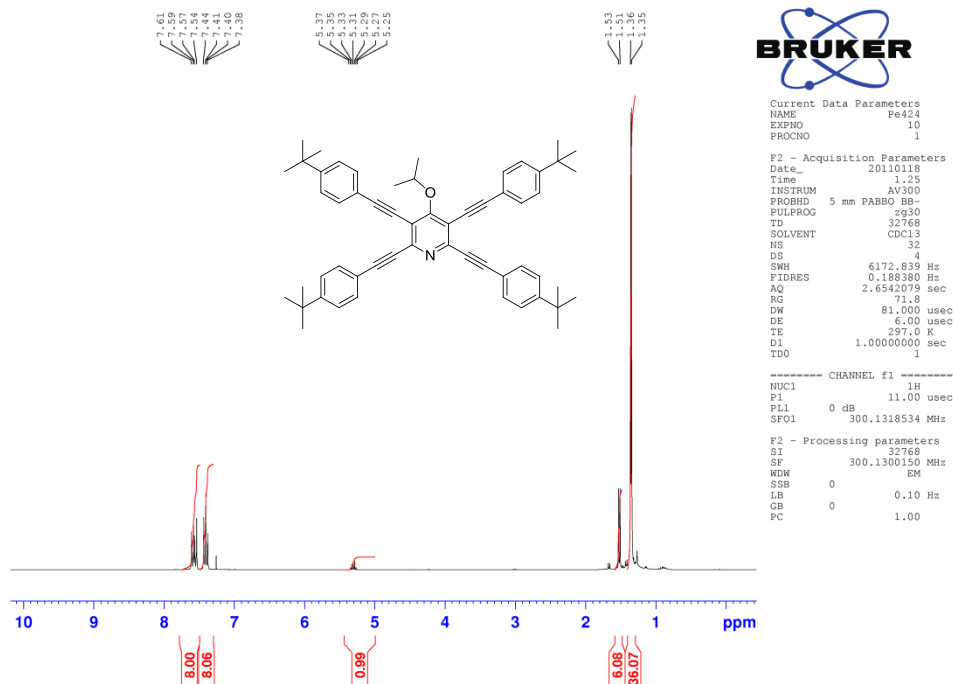
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FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
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NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
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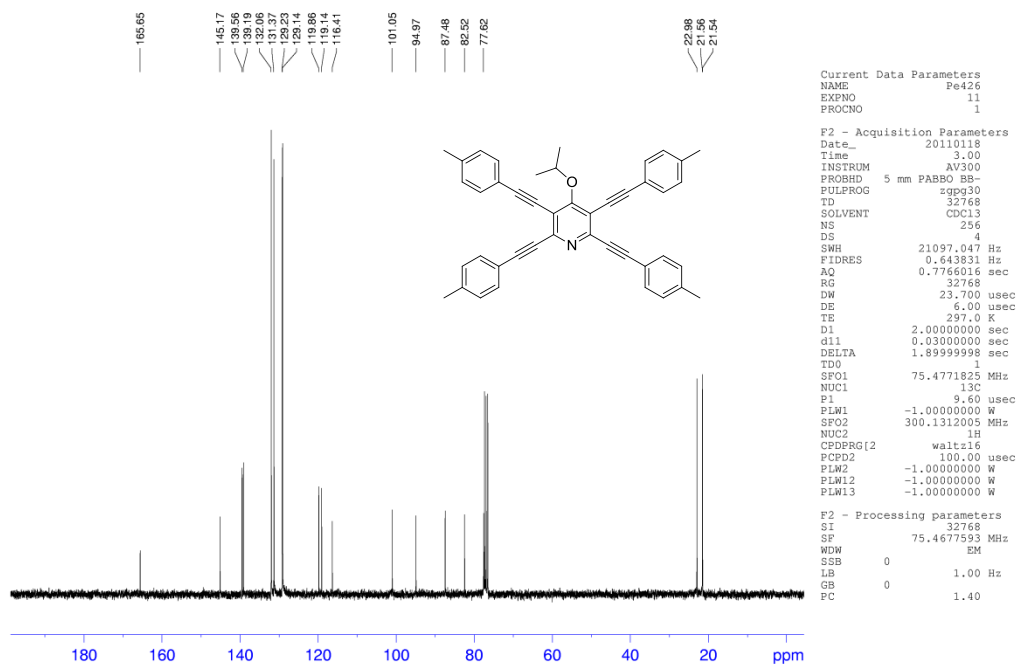
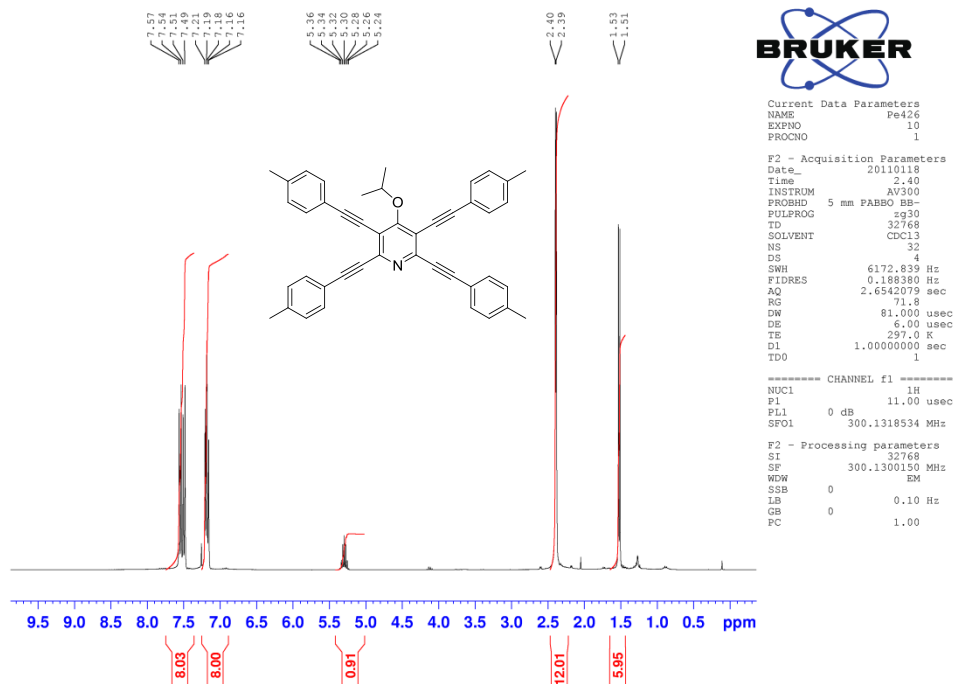
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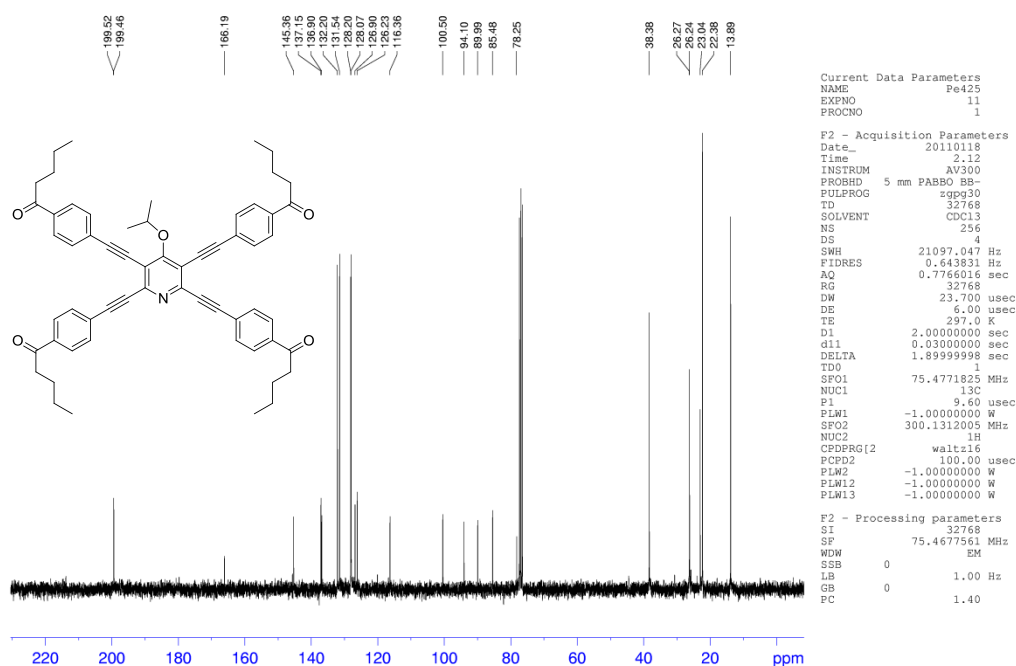
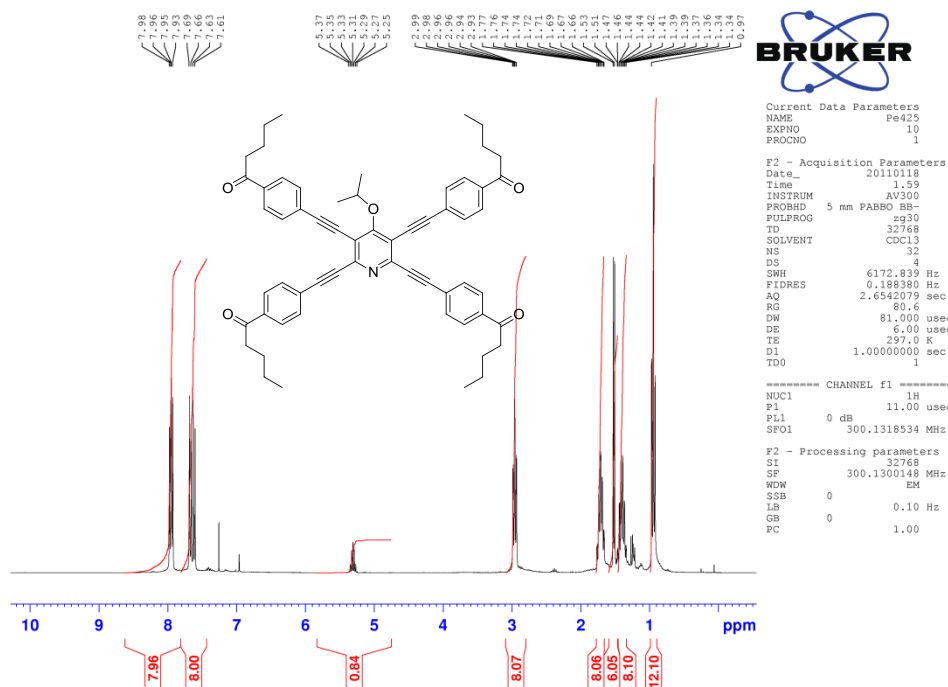
¹H- and ¹³C-NMR spectra of 9a

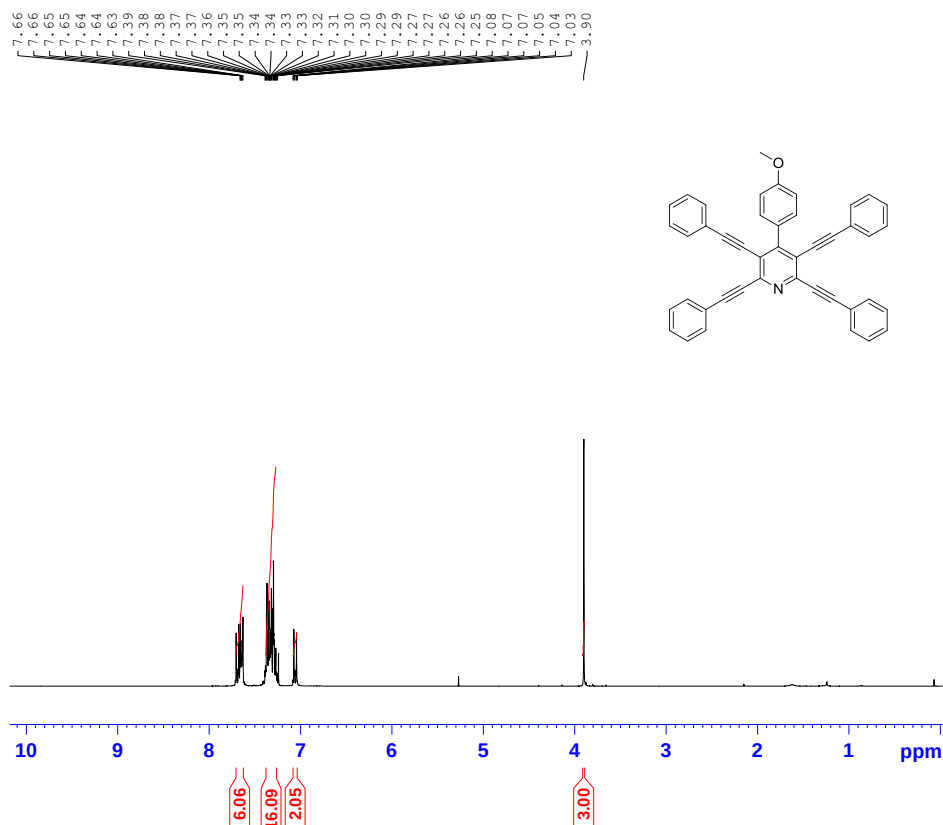
¹H- and ¹³C-NMR spectra of 9b

¹H- and ¹³C-NMR spectra of 9c

¹H- and ¹³C-NMR spectra of 9d

¹H- and ¹³C-NMR spectra of 9e

¹H- and ¹³C-NMR spectra of 9f

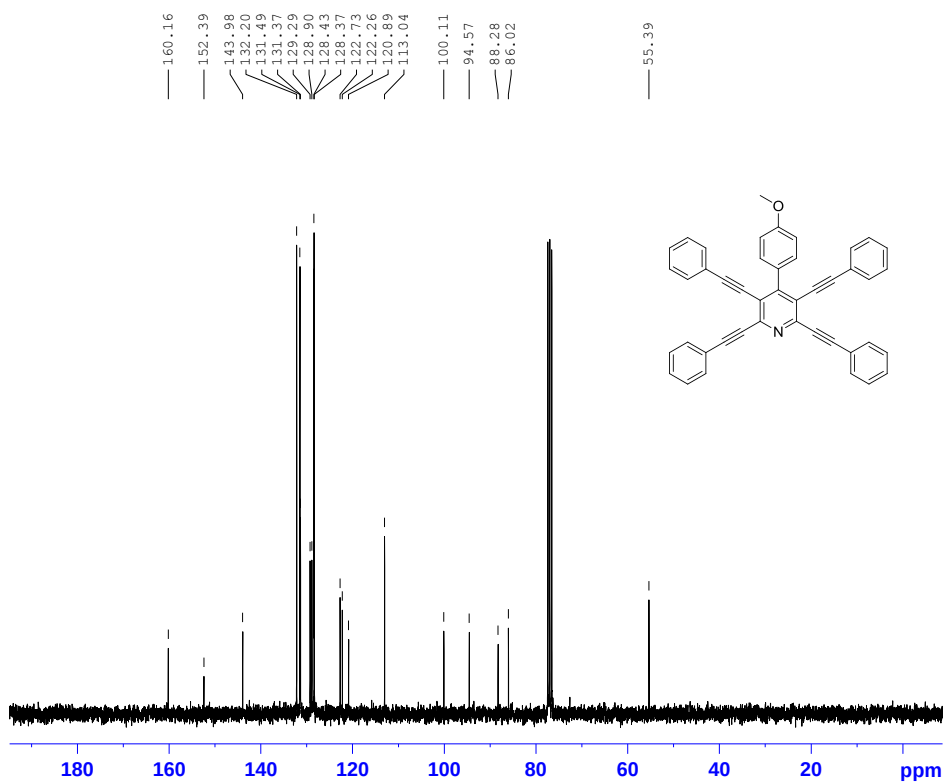
¹H- and ¹³C-NMR spectra of 10a

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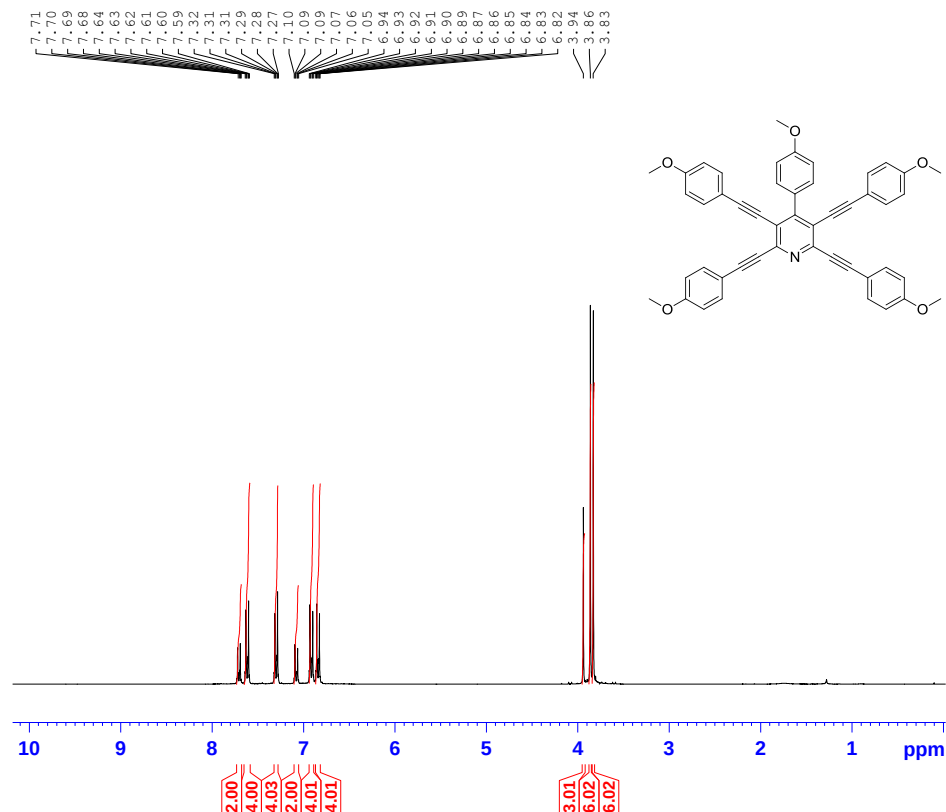
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 DW 23.700 usec
 DE 6.00 usec
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 D1 2.00000000 sec
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 TD0 1
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 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677554 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

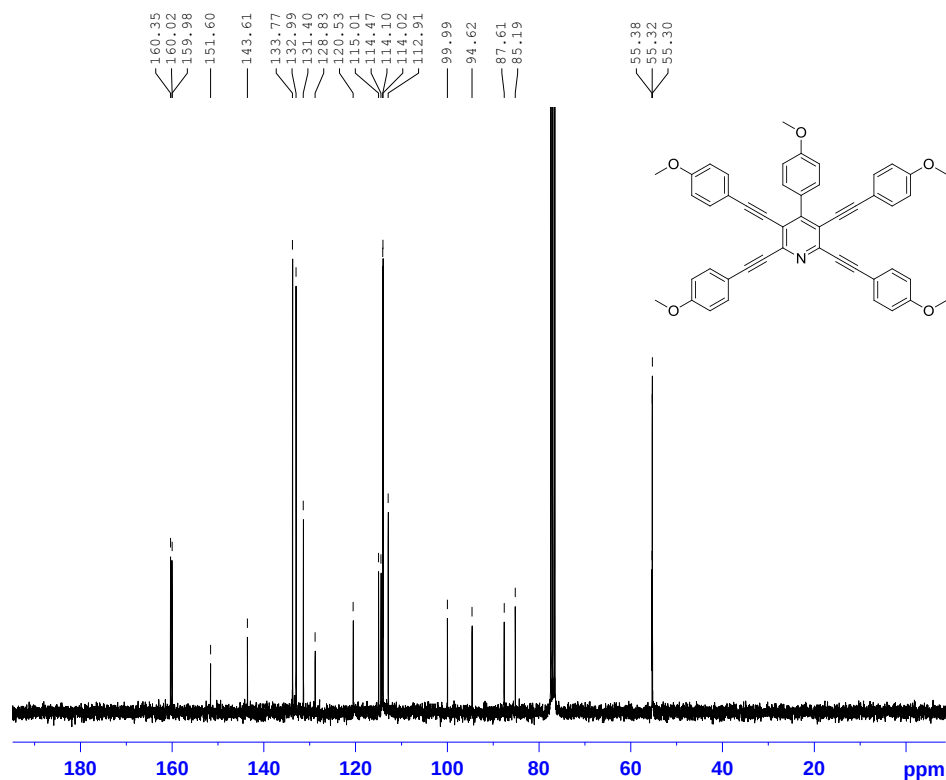
¹H- and ¹³C-NMR spectra of 10b

Current Data Parameters
 NAME AP-40
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120117
 Time_ 8.04
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 294.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300078 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

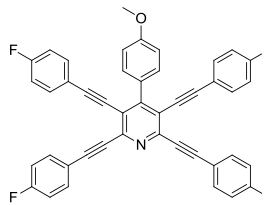
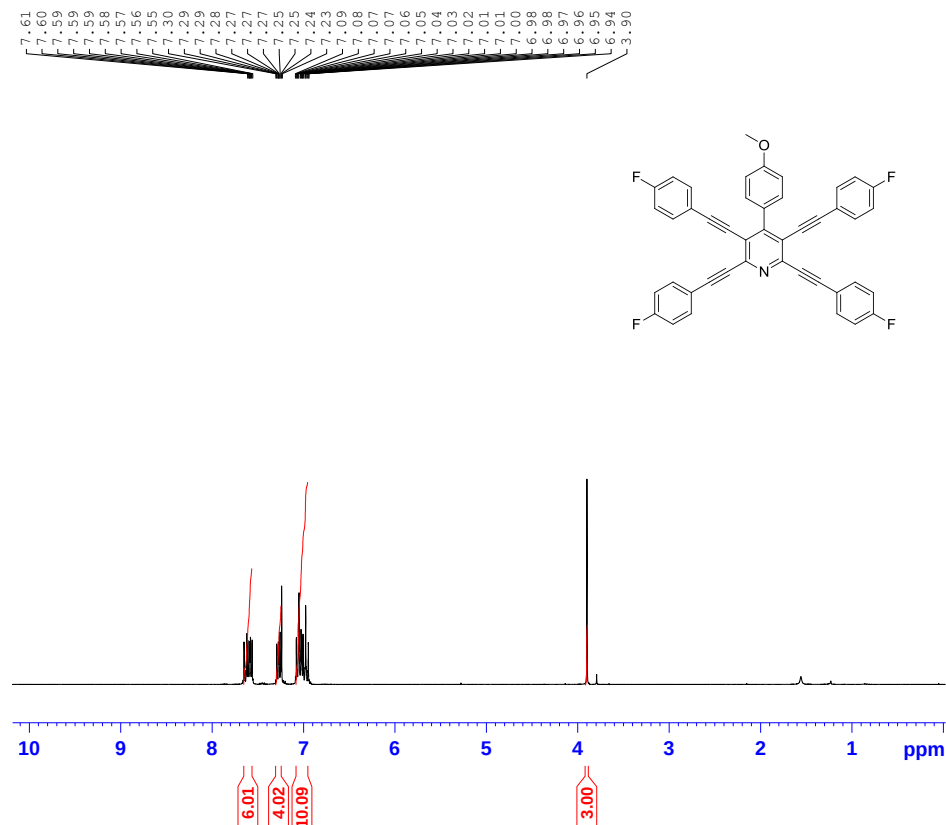


Current Data Parameters
 NAME AP-40
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120117
 Time_ 8.54
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 294.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999999 sec
 TD0 1
 SF01 75.4771825 MHz
 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SF02 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 FCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677547 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

^1H - and ^{13}C -NMR spectra of 10c

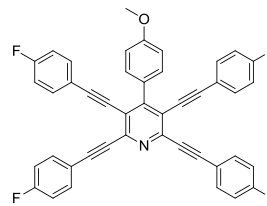
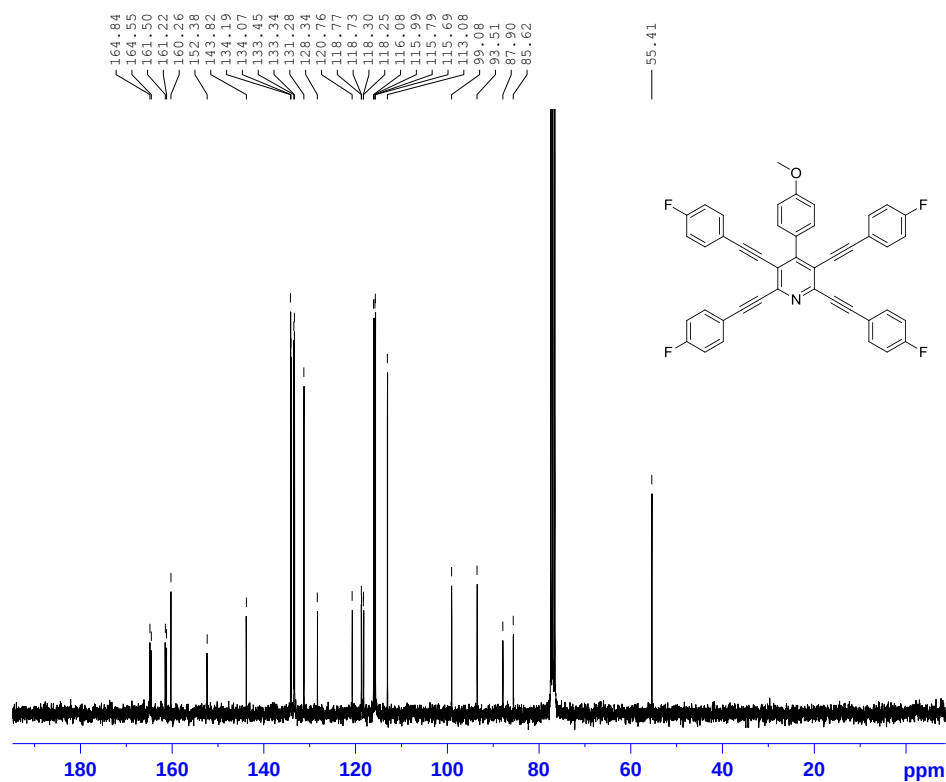


```
Current Data Parameters
NAME                AP-32
EXPNO                10
PROCNO              1
```

```
F2 - Acquisition Parameters
Date_                20111221
Time_                22.40
INSTRUM              AV300
PROBHD      5 mm PABBO BB-
PULPROG              zg30
TD                   32768
SOLVENT              CDCl3
NS                     32
DS                     4
SWH                   6172.839  Hz
FIDRES                0.188380  Hz
AQ                   2.6542079  sec
RG                     574.7
DE                   81.000000  usec
TE                     6.00  usec
TF                     294.4  K
D1                   1.00000000  sec
TD0                     1
```

```
===== CHANNEL f1 =====
NUC1                1H
P1                  11.00 usec
PL1                 0 dB
SFO1                300.1318534 MHz
```

```
F2 - Processing parameters
SI                32768
SF                300.1300214 MHz
WDW               EM
SSB               0
LB                0.10 Hz
GB                0
PC                1.00
```



```
Current Data Parameters
NAME                AP-32
EXPNO                11
PROCNO              1
```

```

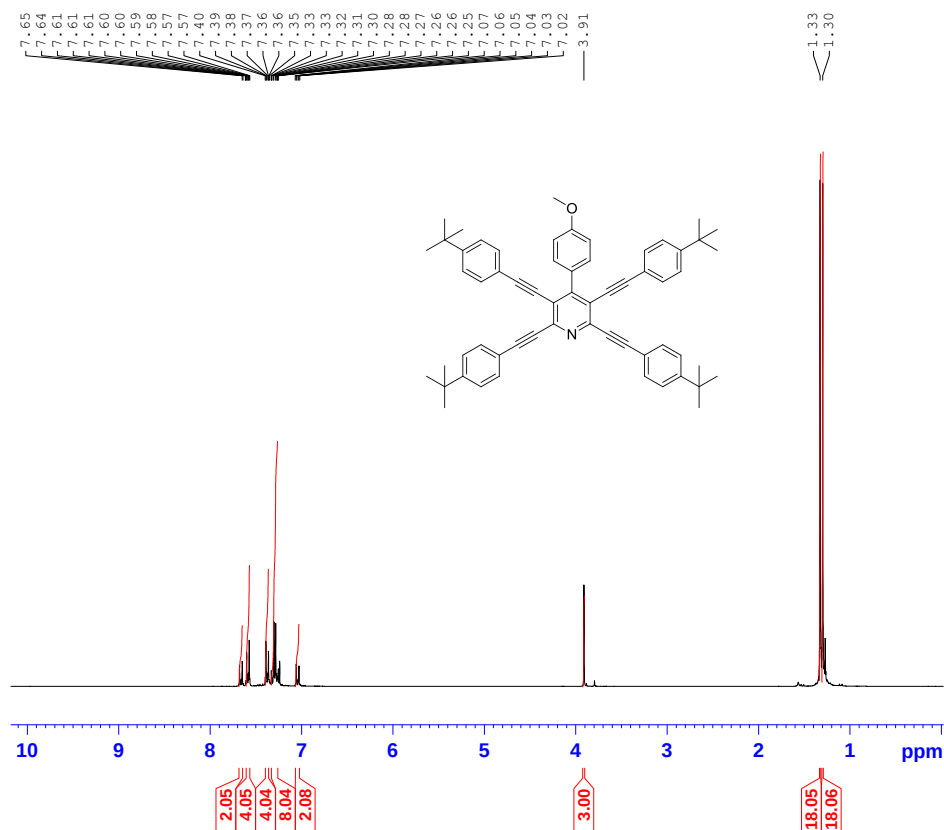
F2 - Acquisition Parameters
Date_      20120717
Time_      15.31
INSTRUM    AV300
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD          32768
SOLVENT    CDCl3
NS          3000
DS          4
SWH         21097.047 Hz
FIDRES     0.643831 Hz
AQ          0.07766016 sec
RG          32768
DE         23.700 usec
WE         6.00 usec
TE          296.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA      1.89999998 sec
TD0         1
SF01       75.4771825 MHz
NUC1        13C
P1          9.60 usec
PLW1       -1.00000000 W
SFO2       300.1312005 MHz
SPQC2      1h
NUC2        13C
PCPD2      waltz16
PLW2       100.00 usec
PLW2       -1.00000000 W
PLW12      -1.00000000 W
PLW13      -1.00000000 W

```

```

F2 - Processing parameters
SI                      32768
SF                      75.4677534 MHz
WDW                      EM
SSB                      0
LB                      1.00 Hz
GB                      0
PC                      1.40

```

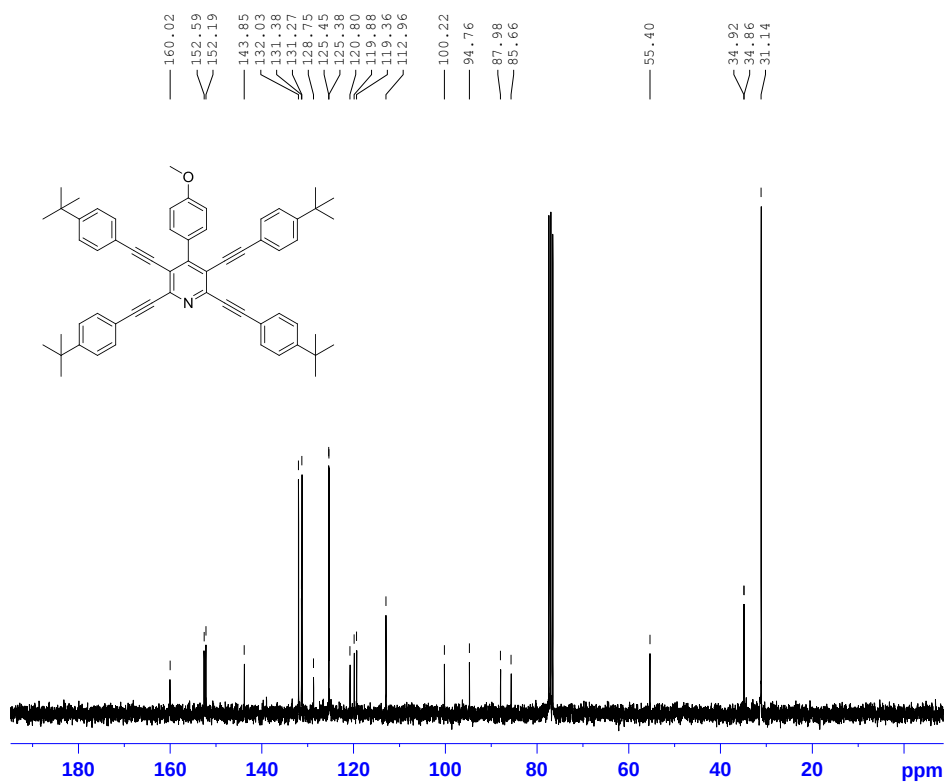

¹H- and ¹³C-NMR spectra of 10d

Current Data Parameters
NAME AP-37
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120107
Time 2.06
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542079 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0 dB
SFO1 300.1318534 MHz

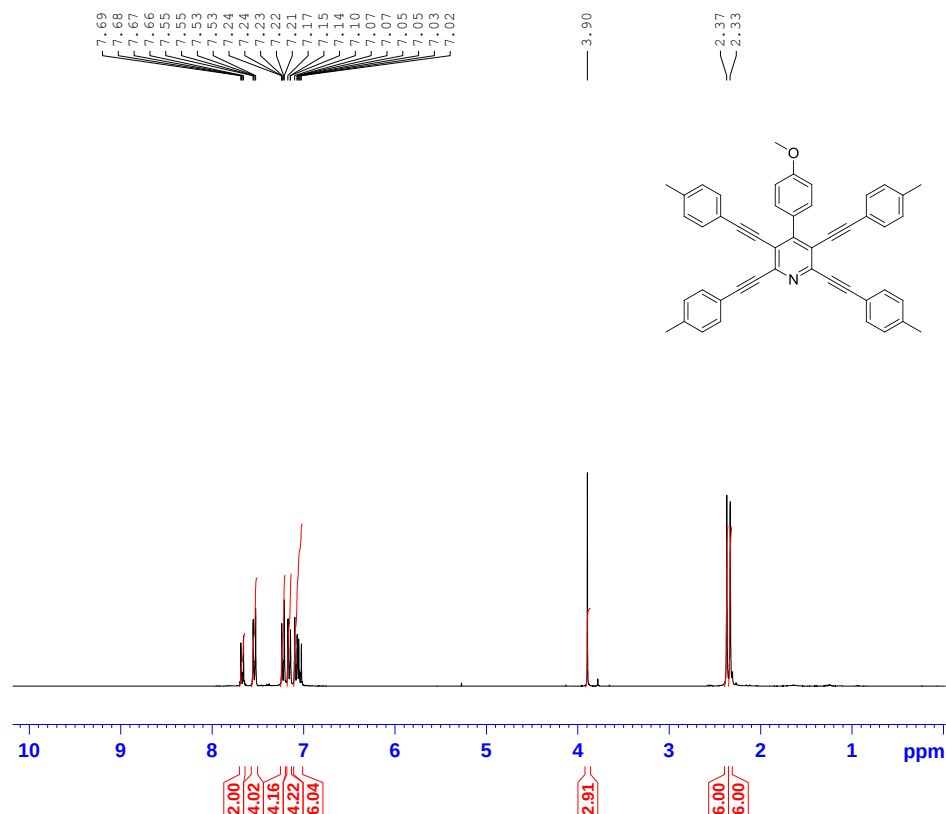
F2 - Processing parameters
SI 32768
SF 300.1300214 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



Current Data Parameters
NAME AP-37
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120107
Time 2.19
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4771825 MHz
NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

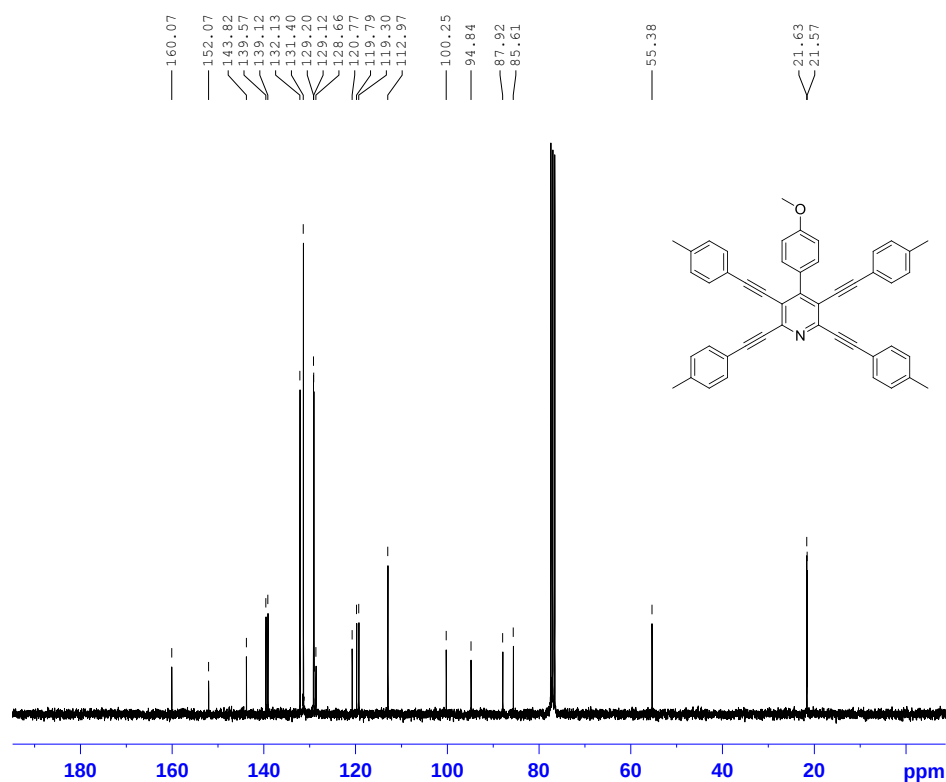
¹H- and ¹³C-NMR spectra of 10e

Current Data Parameters
NAME AP-45
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120124
Time 19.49
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542079 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0 dB
SF01 300.1318534 MHz

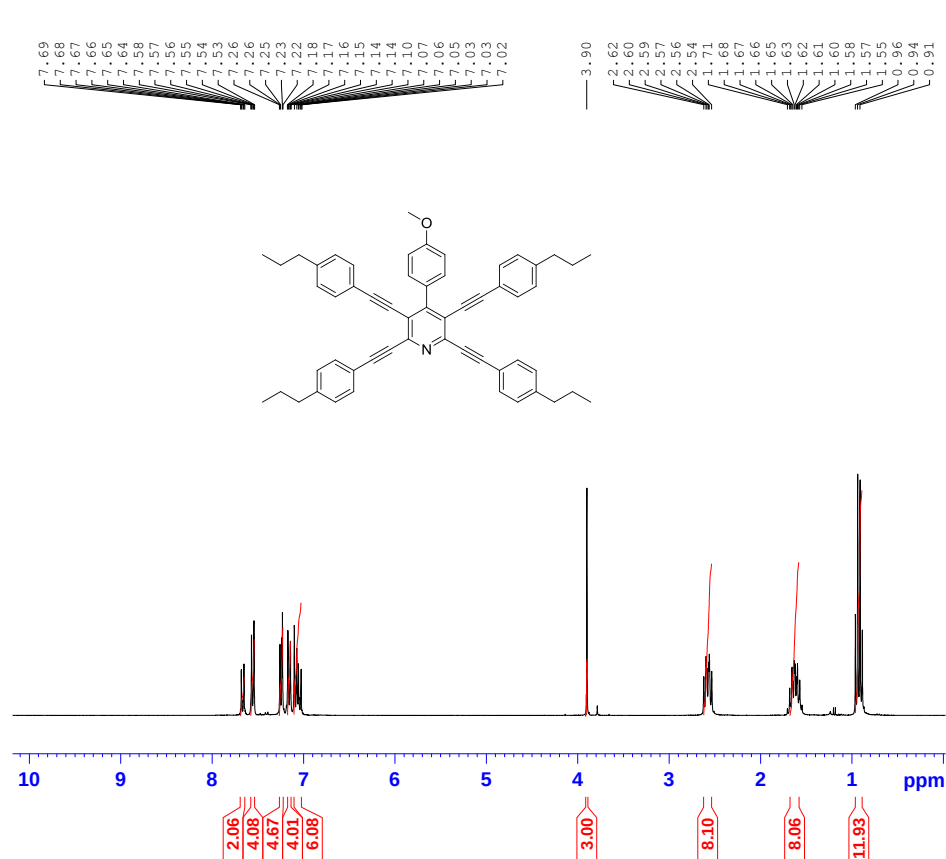
F2 - Processing parameters
SI 32768
SF 300.1300211 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



Current Data Parameters
NAME AP-45
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120723
Time 23.24
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SF01 75.4771825 MHz
NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SF02 300.1312005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

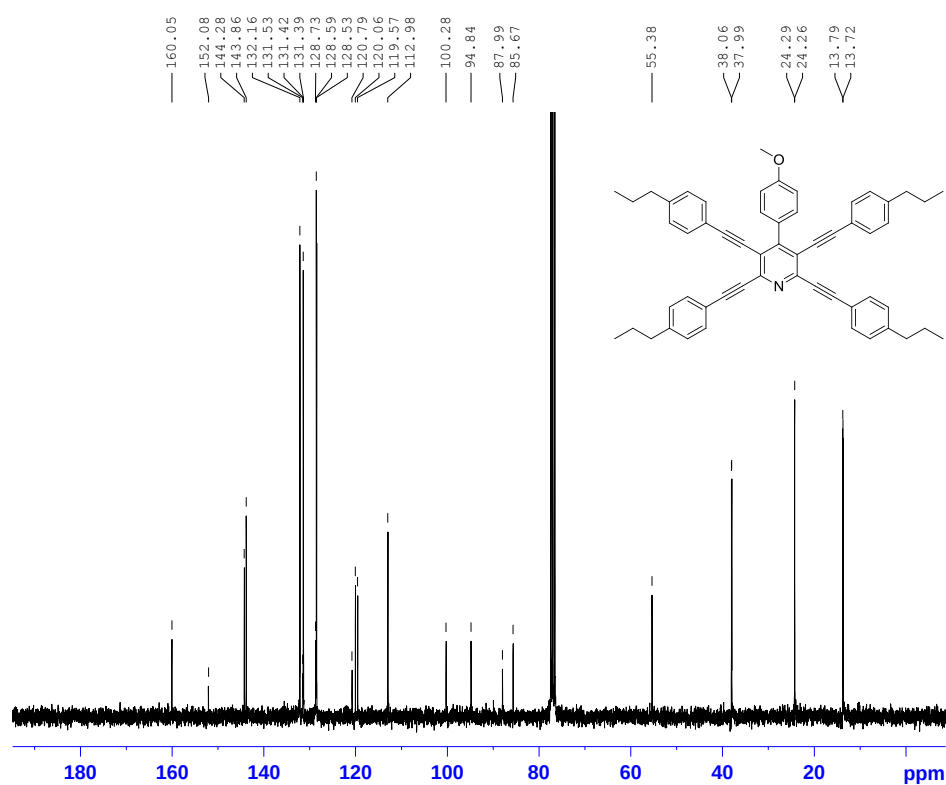
¹H- and ¹³C-NMR spectra of 10f

Current Data Parameters
 NAME AP-44
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120124
 Time_ 20.21
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 294.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SFO1 300.1318534 MHz

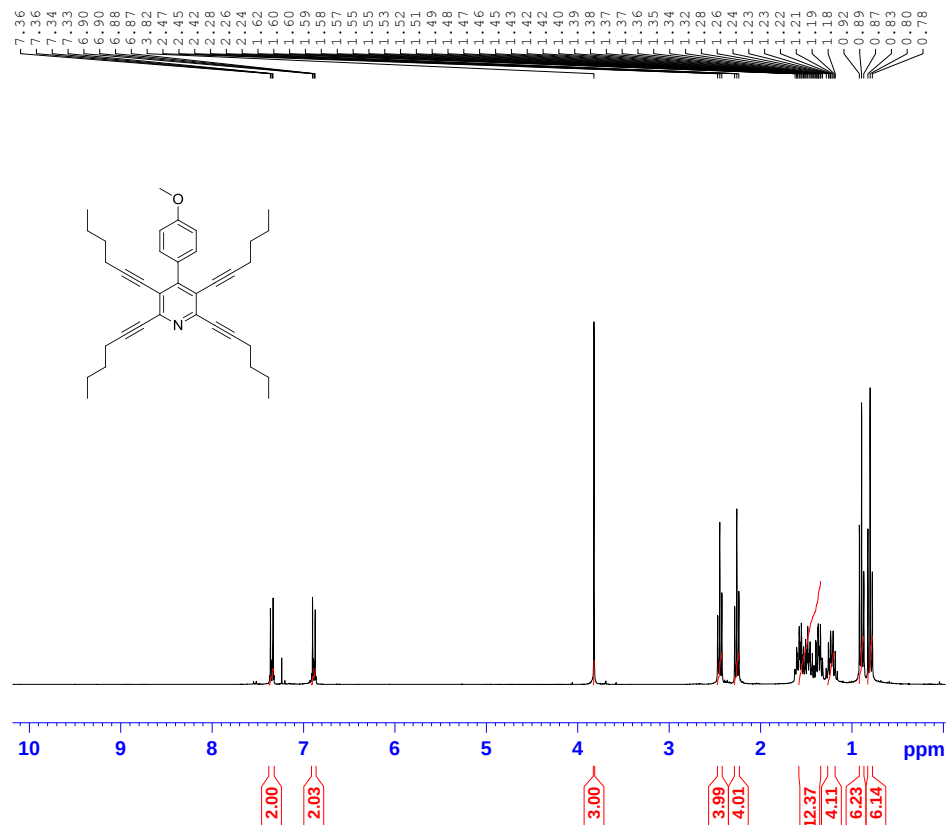
F2 - Processing parameters
 SI 32768
 SF 300.1300214 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME AP-44
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120723
 Time_ 21.38
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1000
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 296.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SFO1 75.4771825 MHz
 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677541 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

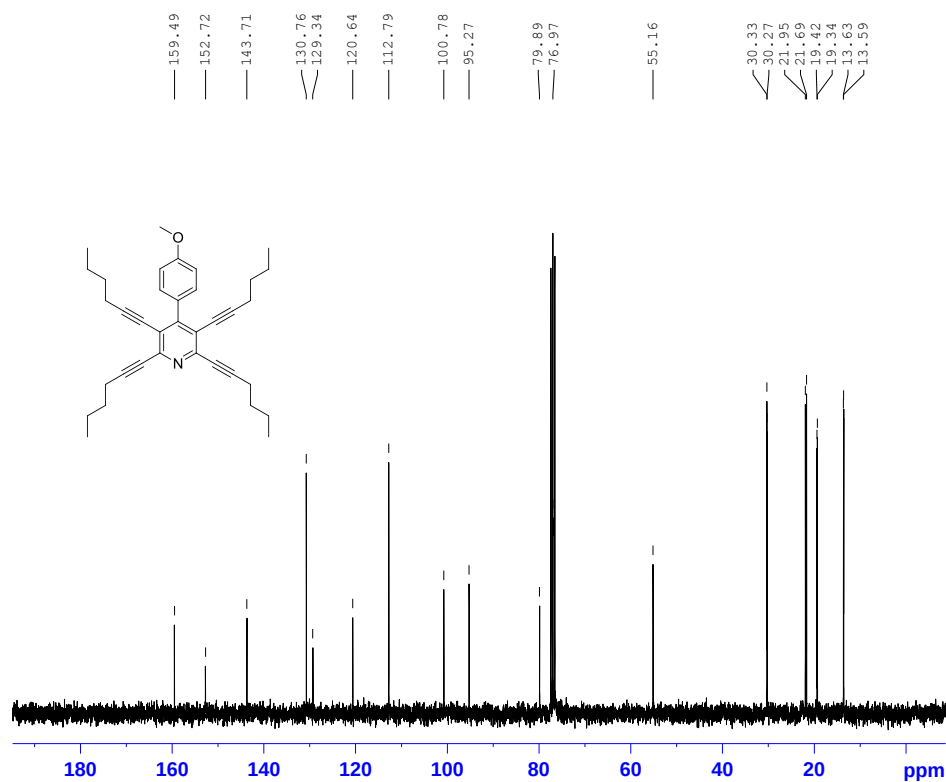
^1H - and ^{13}C -NMR spectra of 10g

Current Data Parameters
 NAME AP-39
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120107
 Time_ 1.04
 INSTRUM AV300
 PROBH 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 101.6
 DW 81.000 usec
 DE 6.00 usec
 TE 293.9 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ^1H
 P1 11.00 usec
 PL1 0 dB
 SF01 300.1318534 MHz

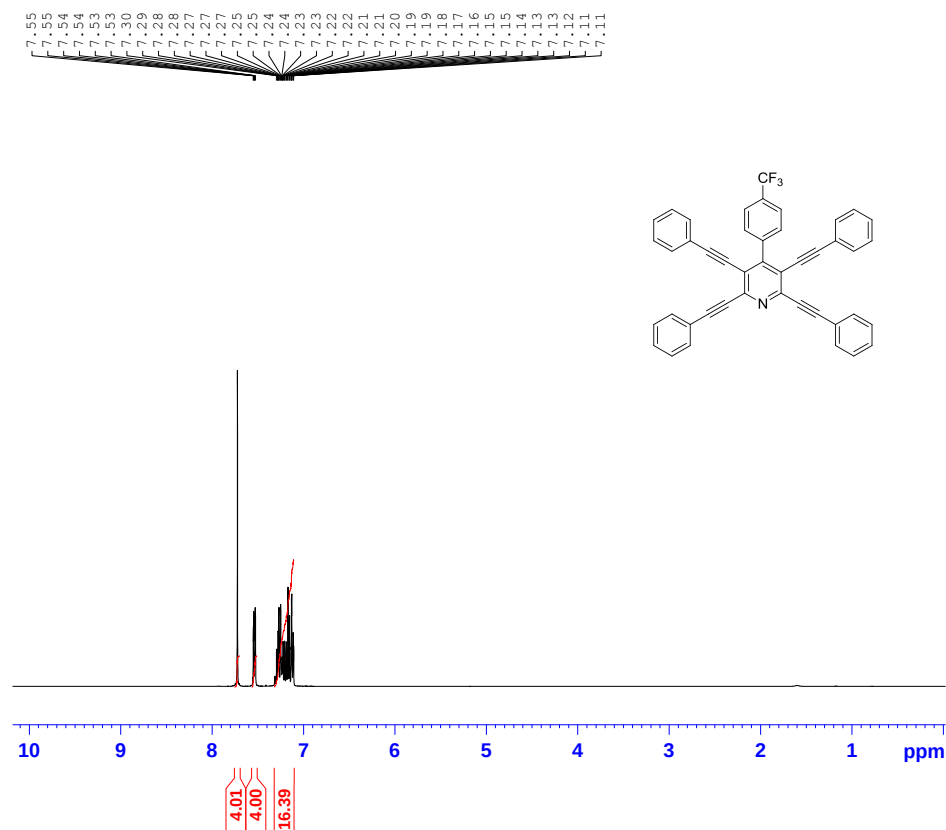
F2 - Processing parameters
 SI 32768
 SF 300.1300215 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME AP-39
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120107
 Time_ 1.19
 INSTRUM AV300
 PROBH 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 294.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SF01 75.4771825 MHz
 NUC1 ^{13}C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SF02 300.1312005 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677541 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

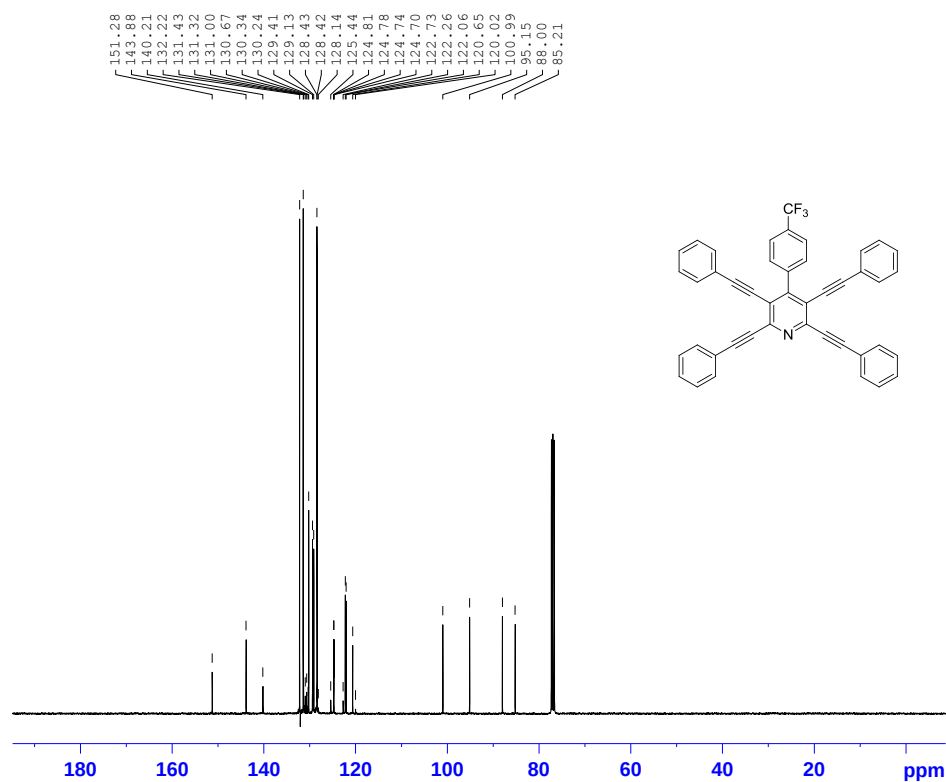
¹H- and ¹³C-NMR spectra of 11a

Current Data Parameters
NAME AP-214
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121210
Time_ 15.18
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 297.0 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 -1.00 dB
PLW 12.26963711 W
SFO1 400.1324000 MHz

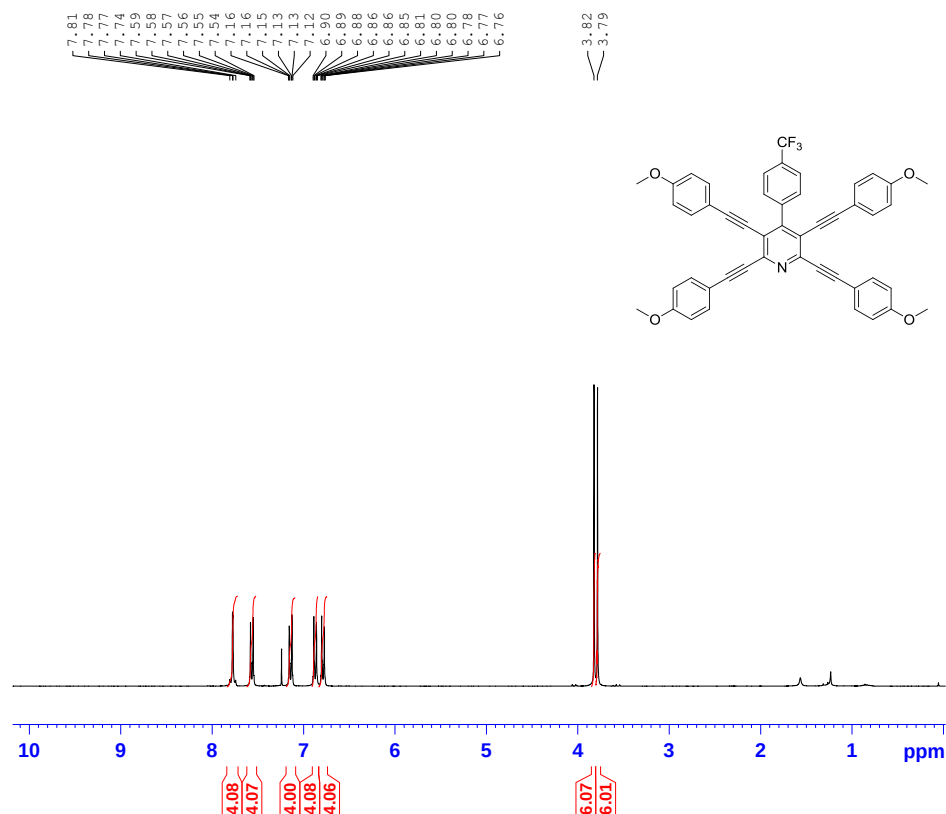
F2 - Processing parameters
SI 32768
SF 400.1300641 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



Current Data Parameters
NAME AP-214
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121210
Time_ 20.54
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7000
DS 4
SWH 29940.119 Hz
FIDRES 0.456850 Hz
AQ 1.0944512 sec
RG 1149.4
DW 16.700 usec
DE 6.00 usec
TE 297.0 K
D1 1.70000005 sec
d11 0.03000000 sec
DELTA 1.60000002 sec
TD0 1
SFO1 100.6260690 MHz
NUC1 13C
P1 10.00 usec
PLW1 -1.00000000 W
SFO2 400.1318000 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 100.6127788 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

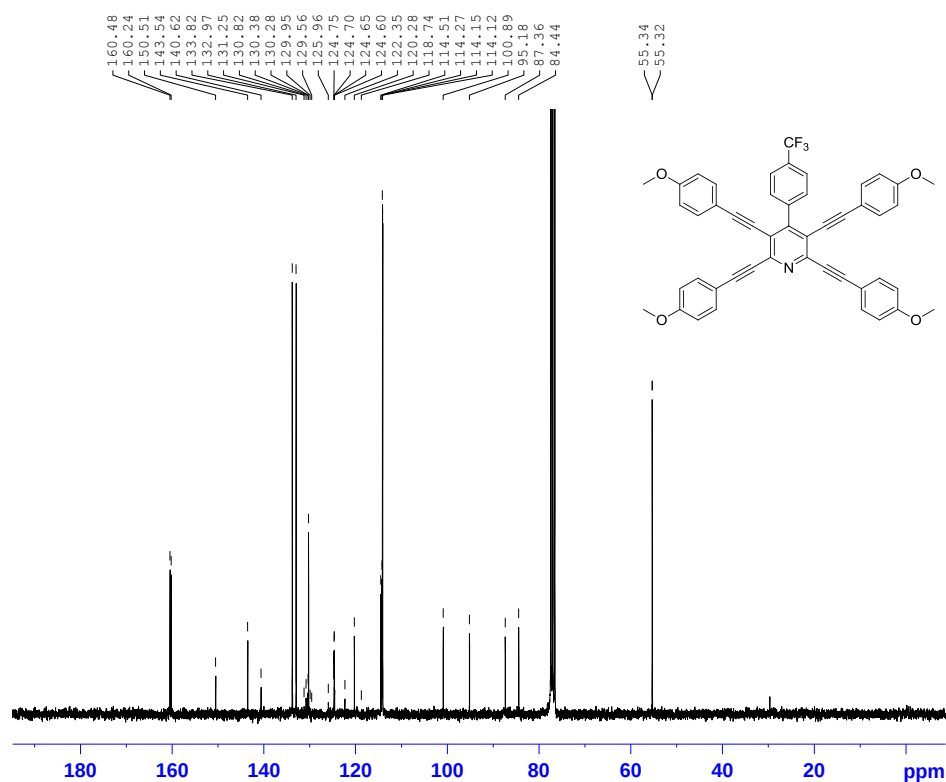
¹H- and ¹³C-NMR spectra of 11b

Current Data Parameters
 NAME AP-213
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121027
 Time 19.18
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 294.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SF01 300.1318534 MHz

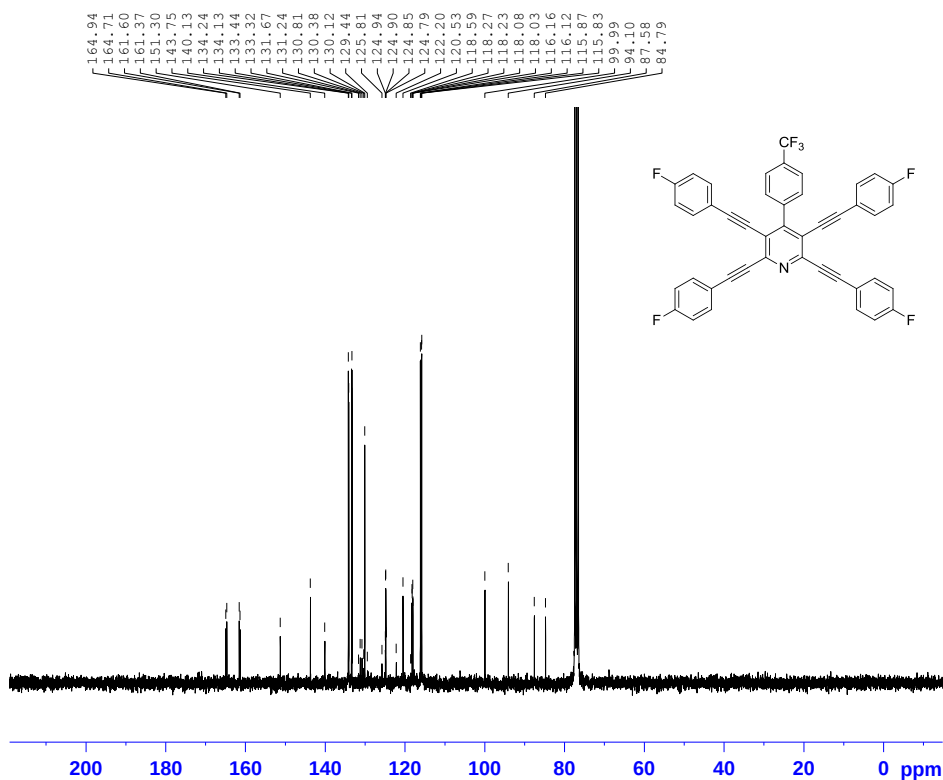
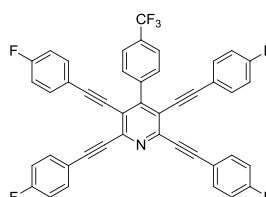
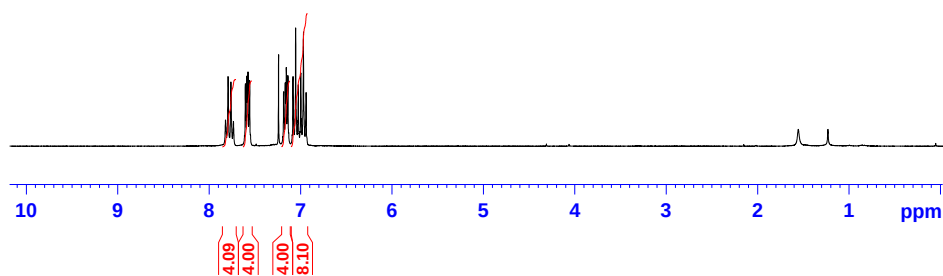
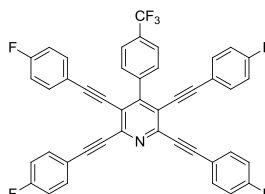
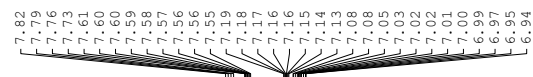
F2 - Processing parameters
 SI 32768
 SF 300.1300214 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME AP-213
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121027
 Time 22.28
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 4000
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 294.6 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SF01 75.4771825 MHz
 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SF02 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 FCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677538 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H- and ¹³C-NMR spectra of 11c

Current Data Parameters
NAME AP-222
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121114
Time 1.07
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542079 sec
RG 512
DW 81.000 usec
DE 6.00 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0 dB
SFO1 300.1318534 MHz

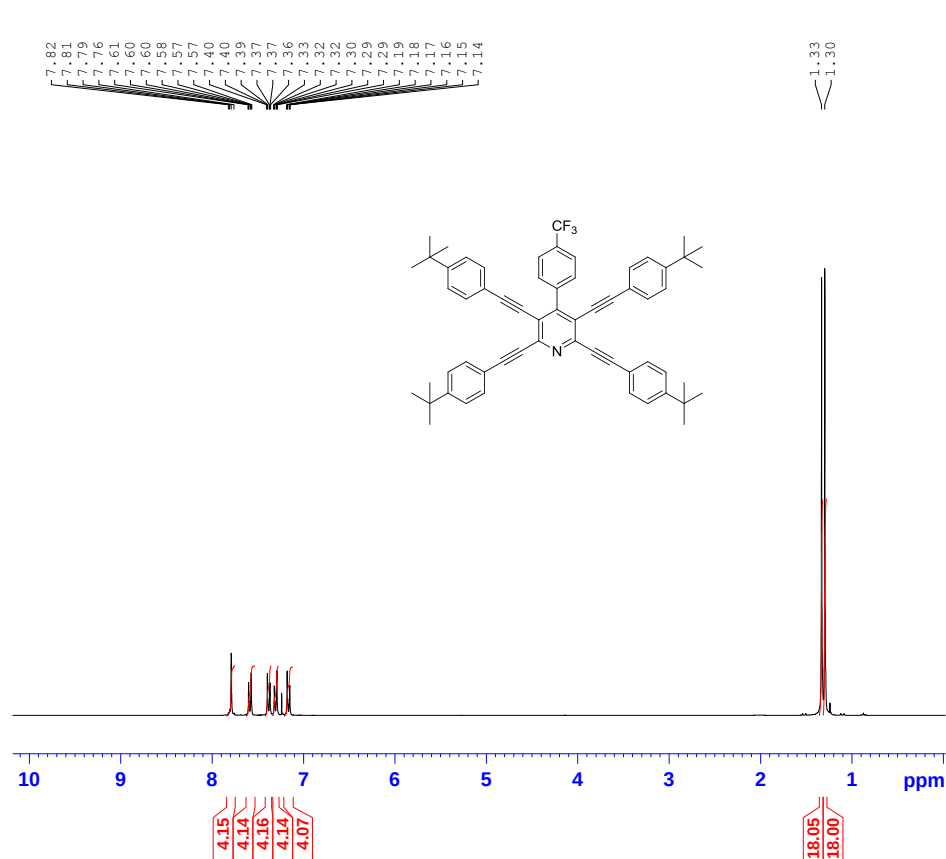
F2 - Processing parameters
SI 32768
SF 300.1300214 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



Current Data Parameters
NAME AP-222
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121114
Time 6.00
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4771825 MHz
NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

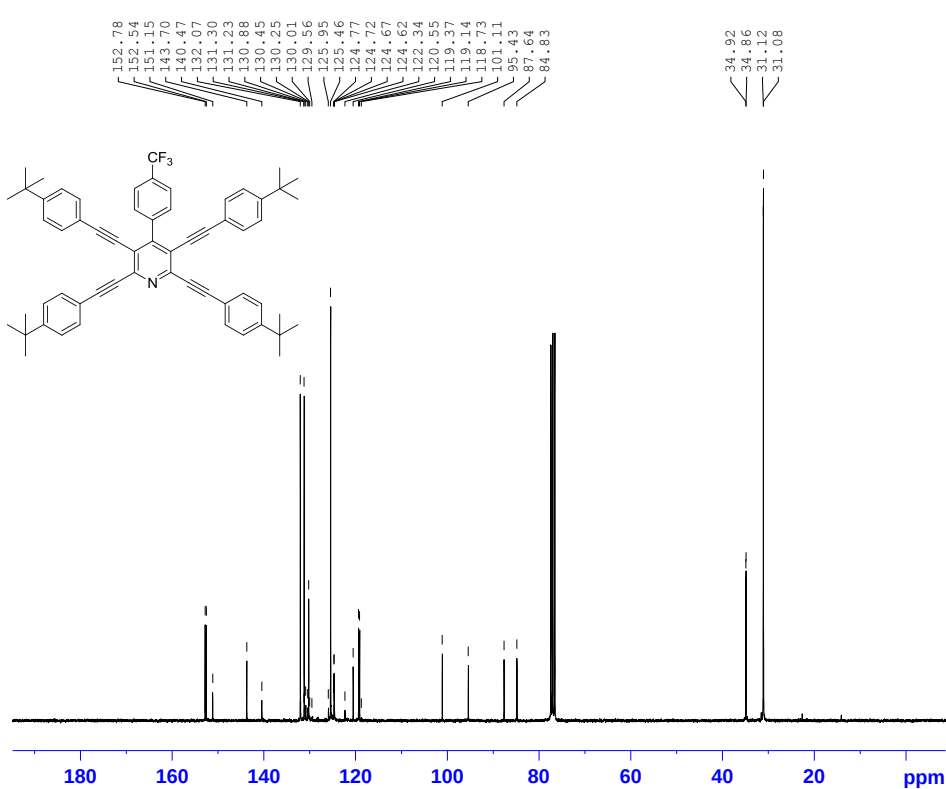
¹H- and ¹³C-NMR spectra of 11d

Current Data Parameters
 NAME AP-211
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121216
 Time_ 19.36
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 101.6
 DW 81.000 usec
 DE 6.00 usec
 TE 293.6 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SFO1 300.1318534 MHz

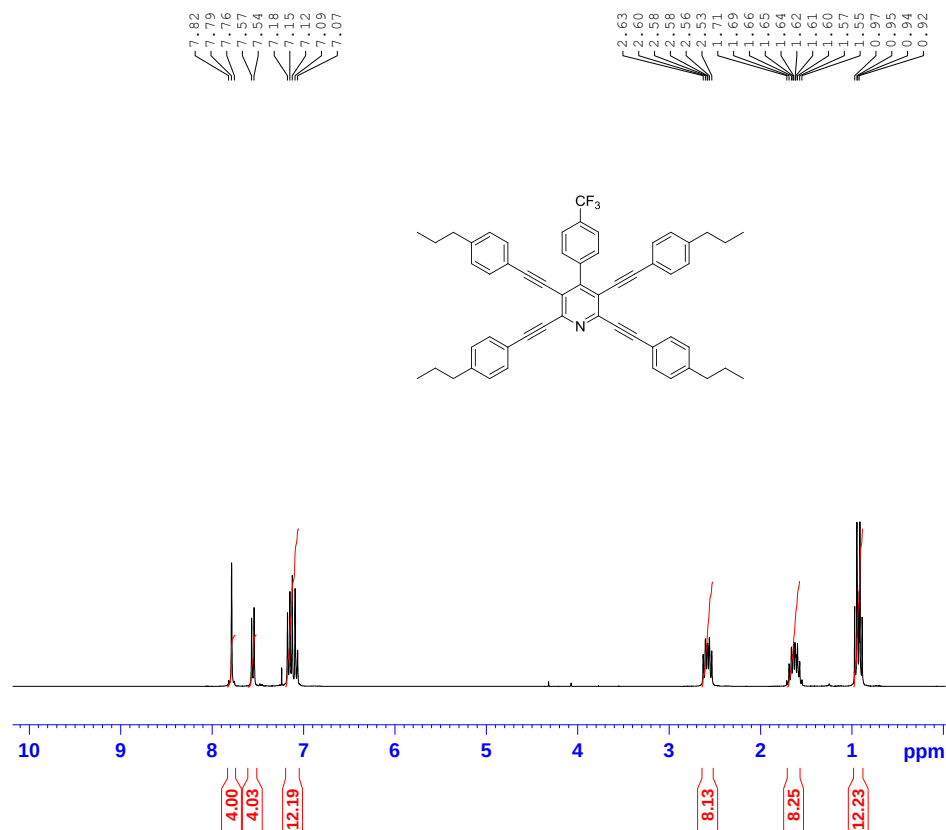
F2 - Processing parameters
 SI 32768
 SF 300.1300214 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME AP-211
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121217
 Time_ 0.22
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 294.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SFO1 75.4771825 MHz
 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SFO2 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677553 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

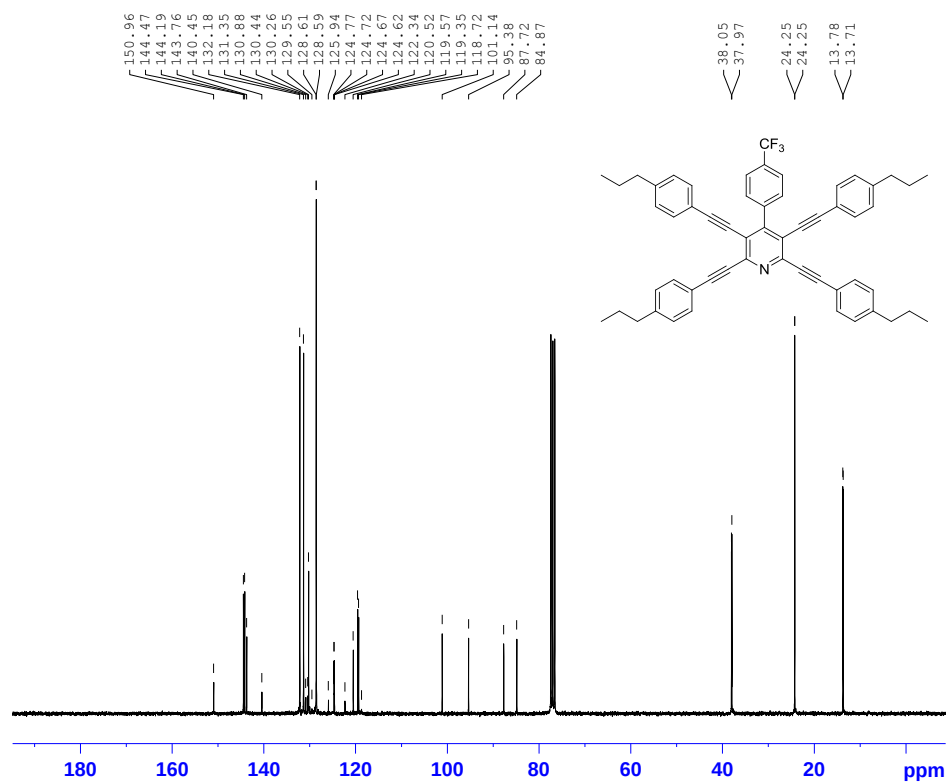
¹H- and ¹³C-NMR spectra of 11e

Current Data Parameters
NAME AP-217
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121103
Time_ 8.38
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542079 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹H
P1 11.00 usec
PL1 0 dB
SFO1 300.1318534 MHz

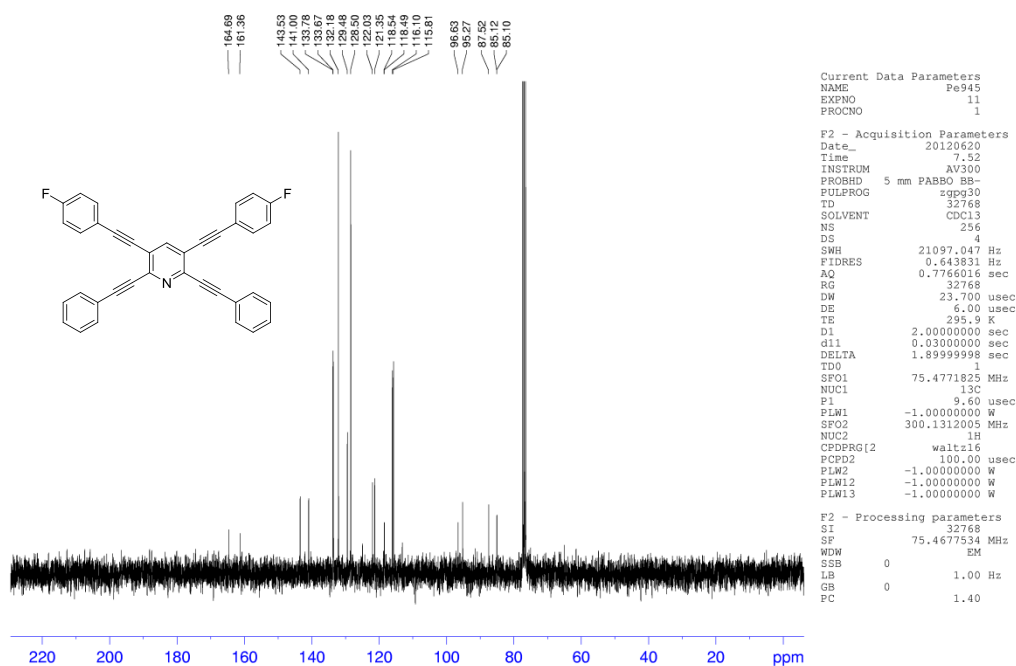
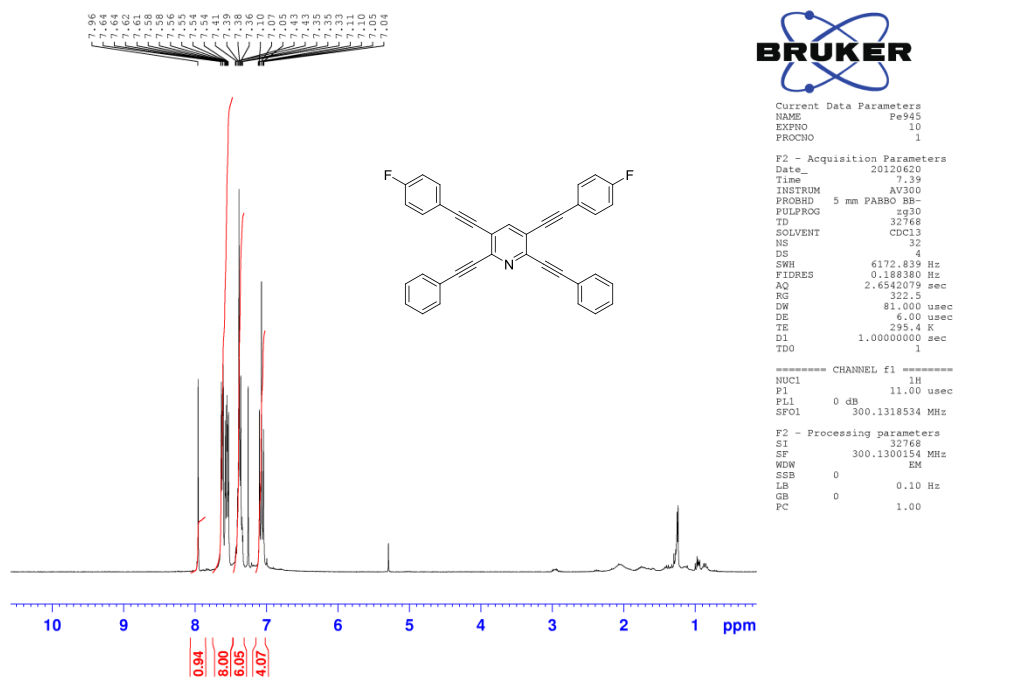
F2 - Processing parameters
SI 32768
SF 300.1300213 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

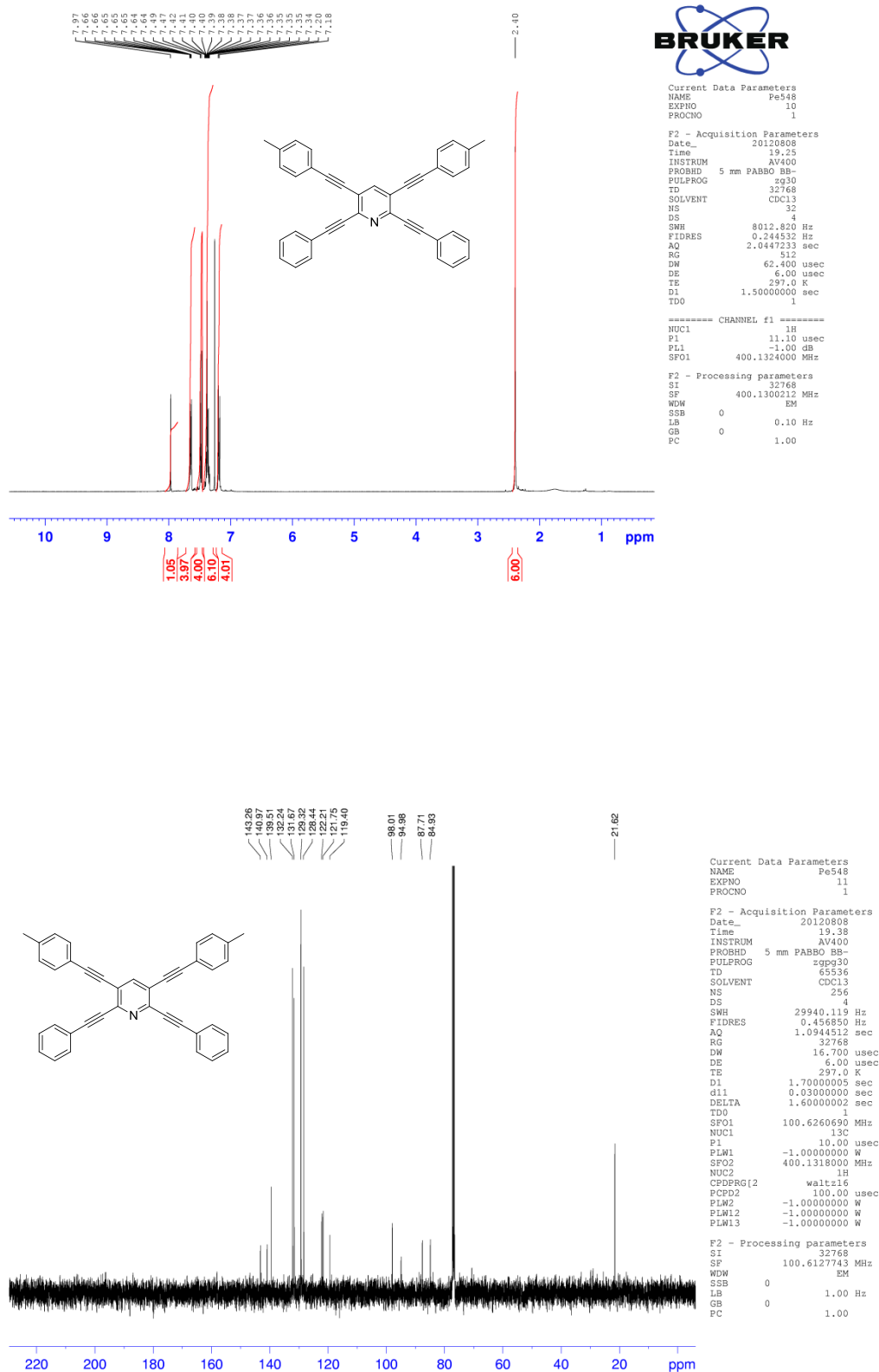


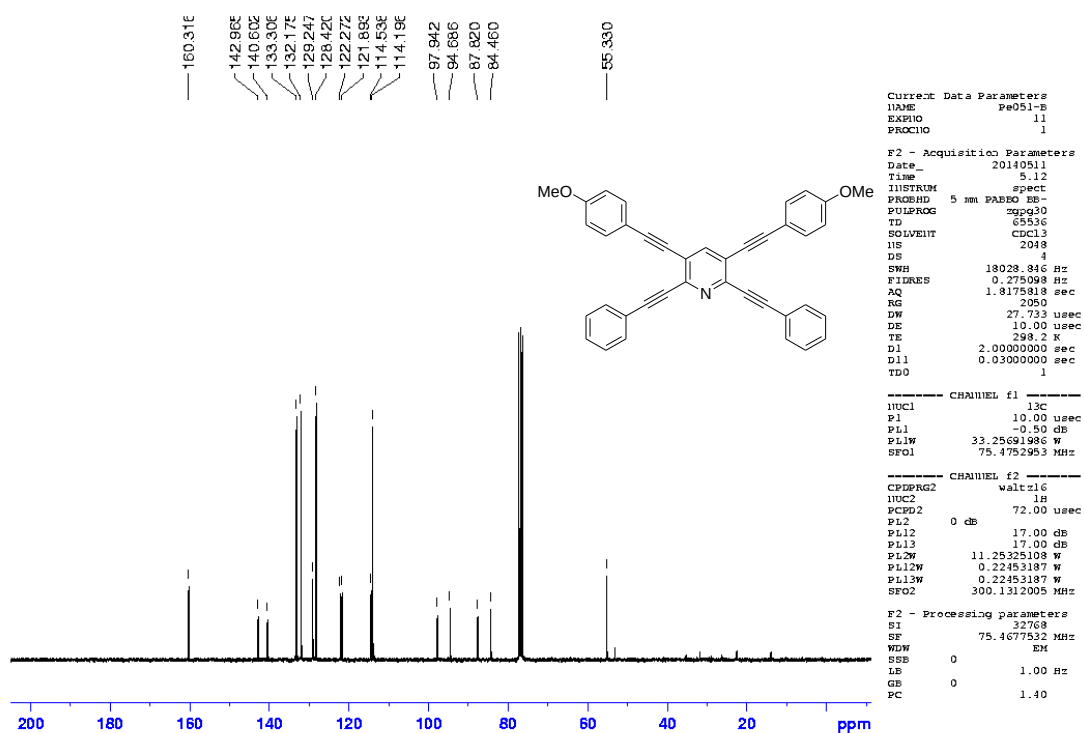
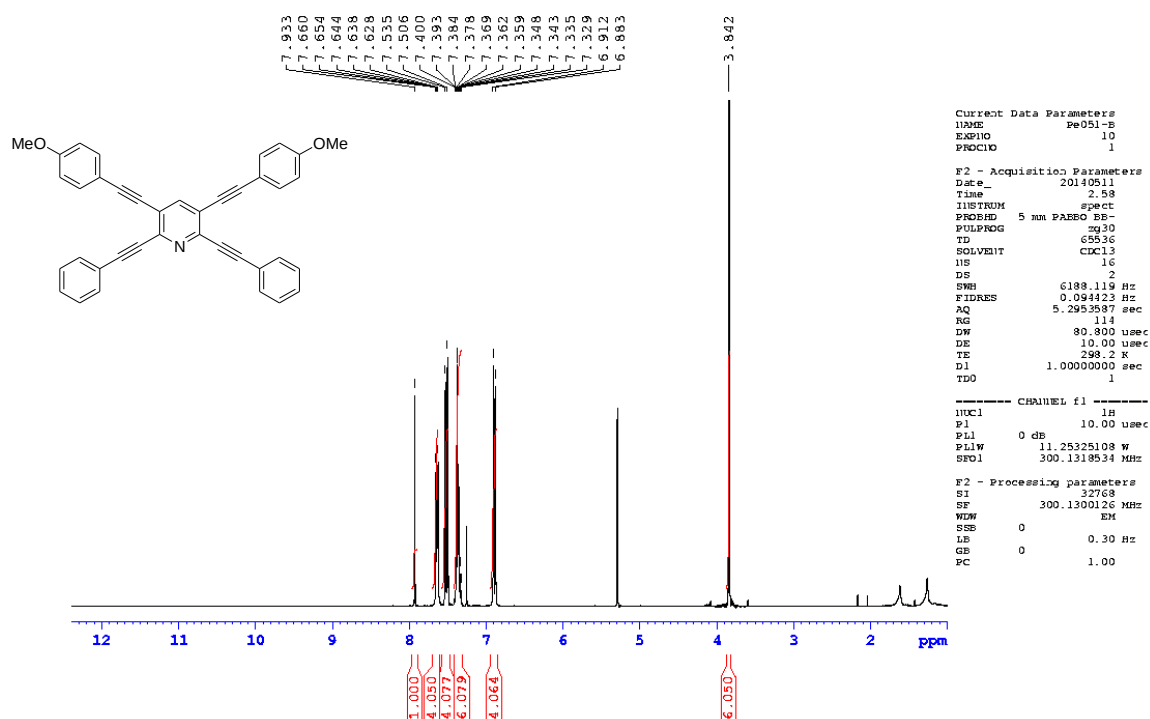
Current Data Parameters
NAME AP-217
EXPNO 13
PROCNO 1

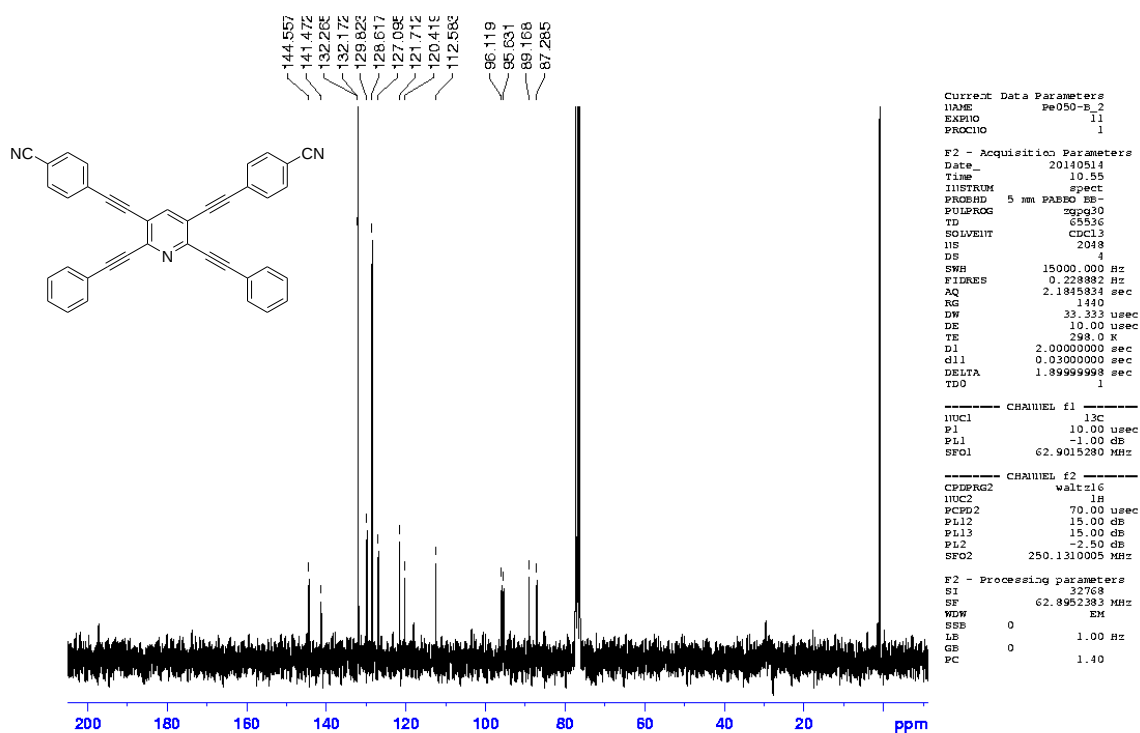
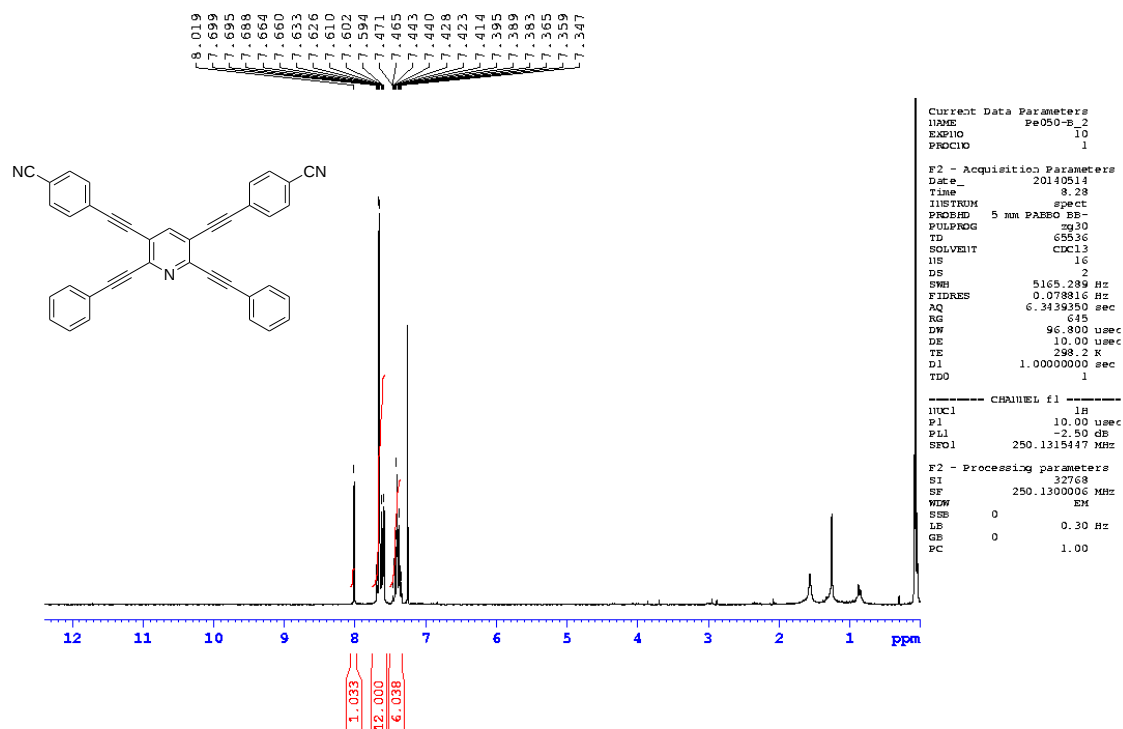
F2 - Acquisition Parameters
Date_ 20121103
Time_ 12.03
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 4000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4771825 MHz
NUC1 ¹³C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 ¹H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

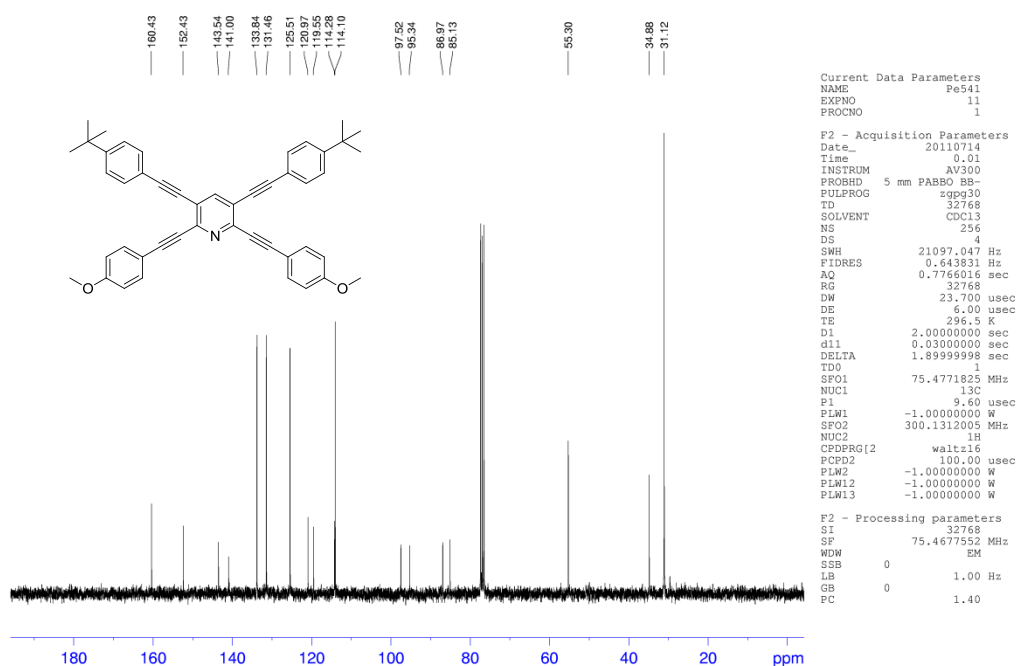
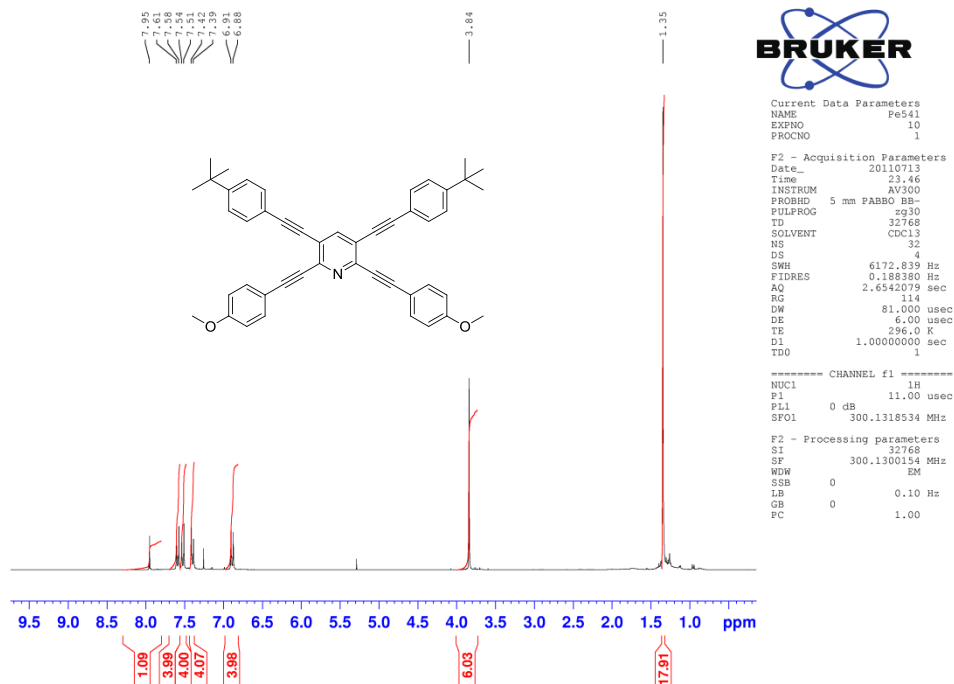
F2 - Processing parameters
SI 32768
SF 75.4677555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

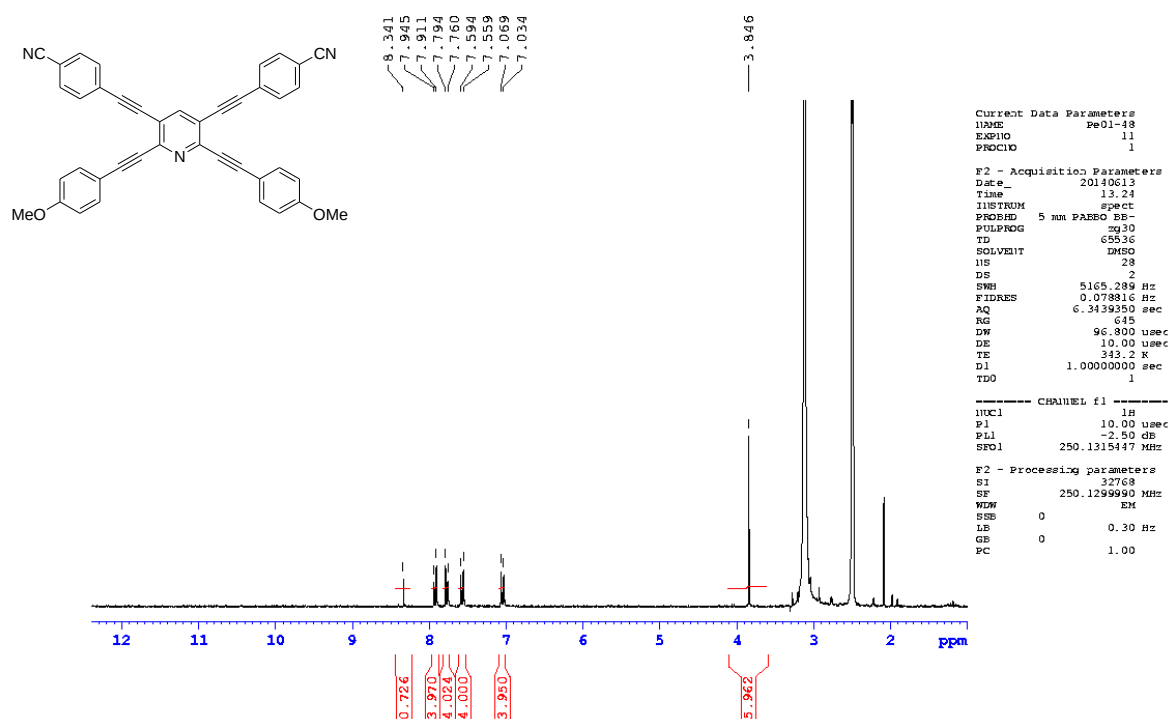
¹H- and ¹³C-NMR spectra of 12a

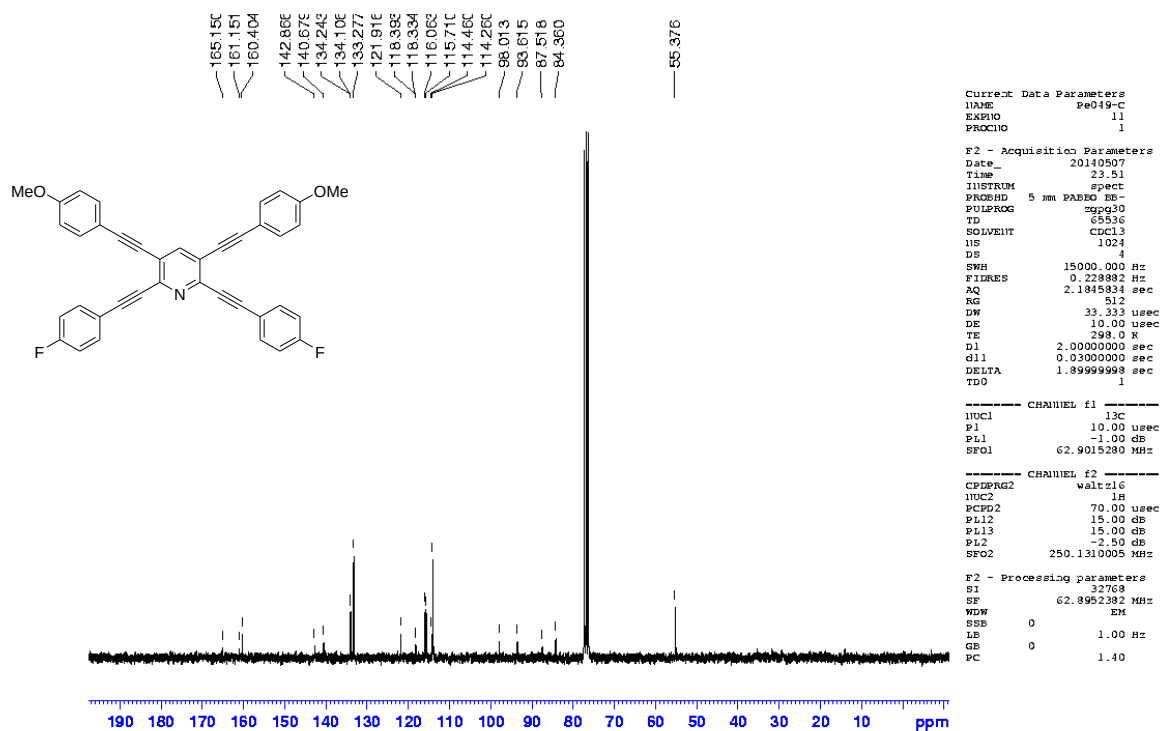
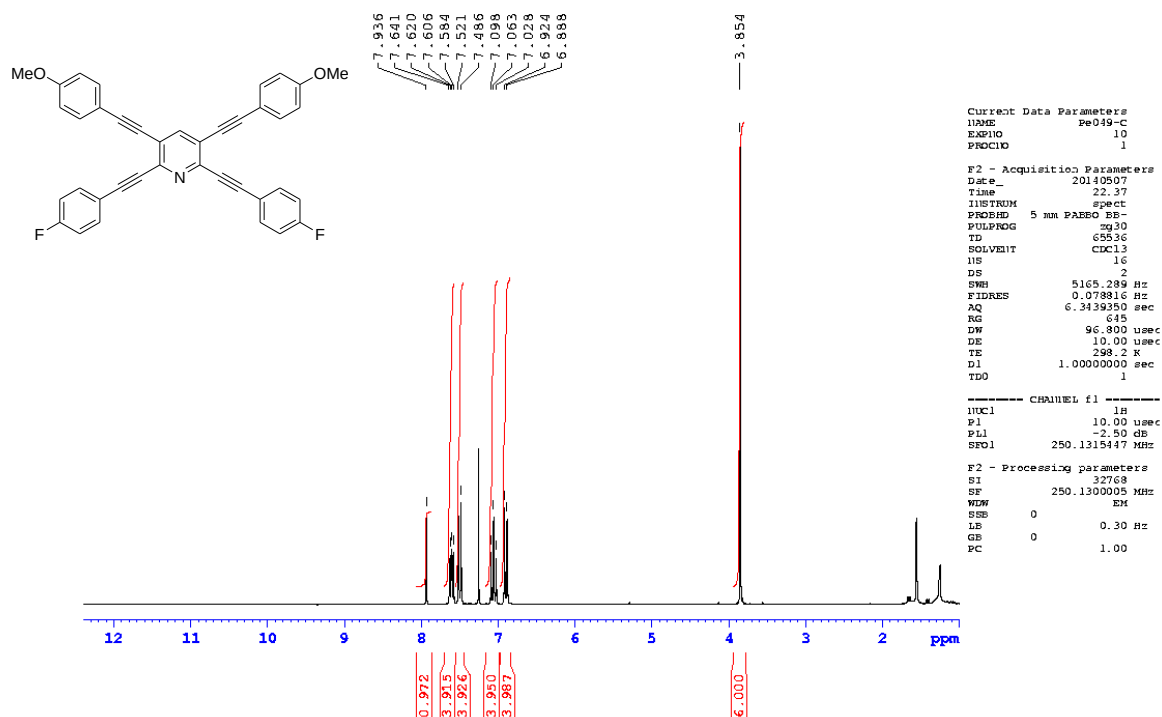
¹H- and ¹³C-NMR spectra of 12b

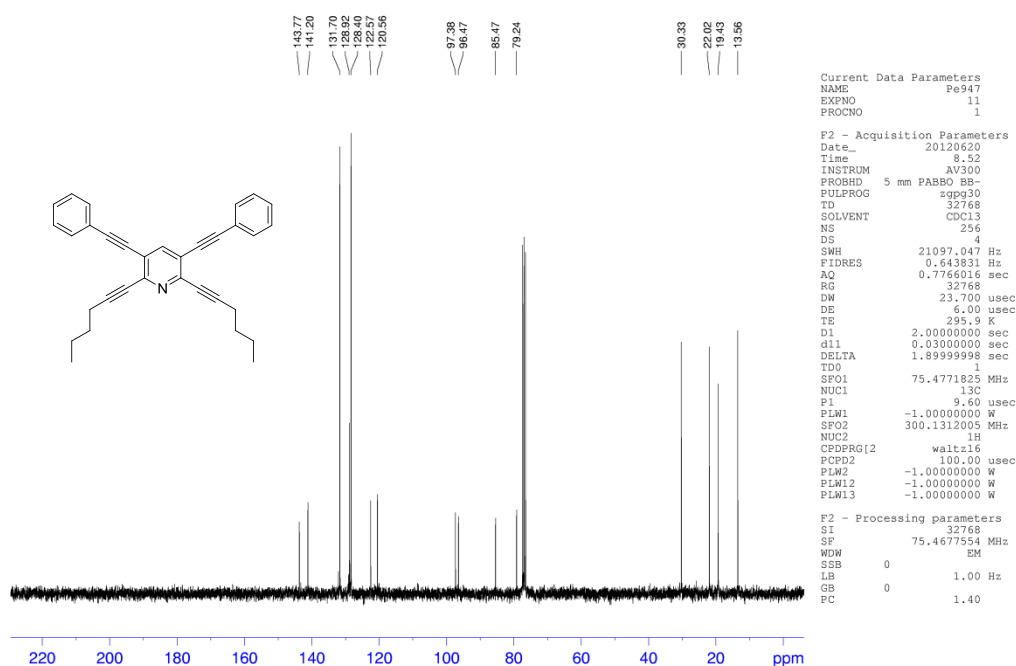
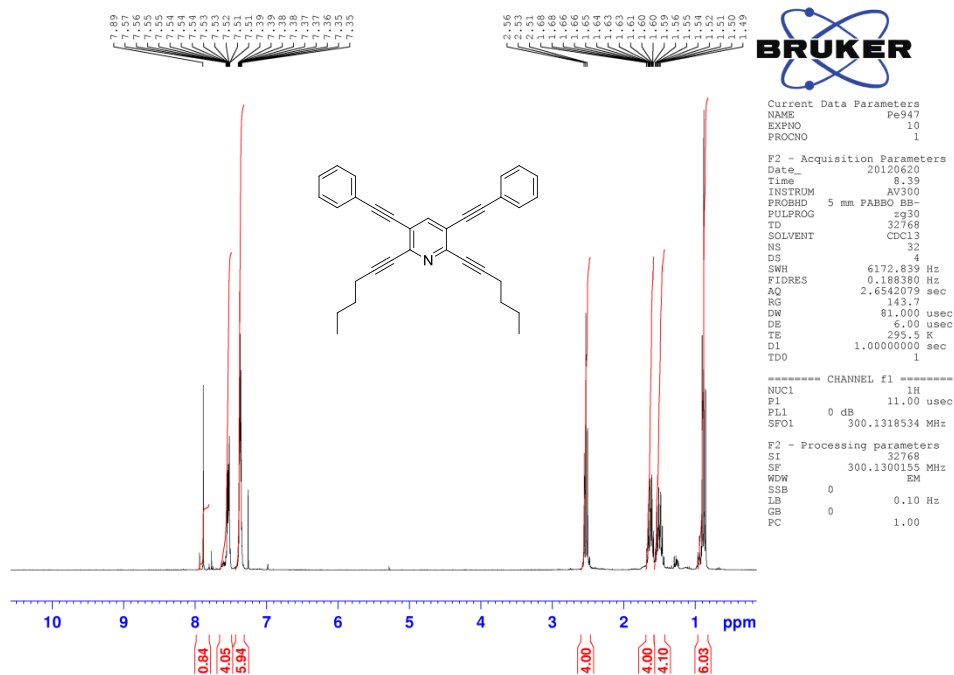
¹H- and ¹³C-NMR spectra of 12c

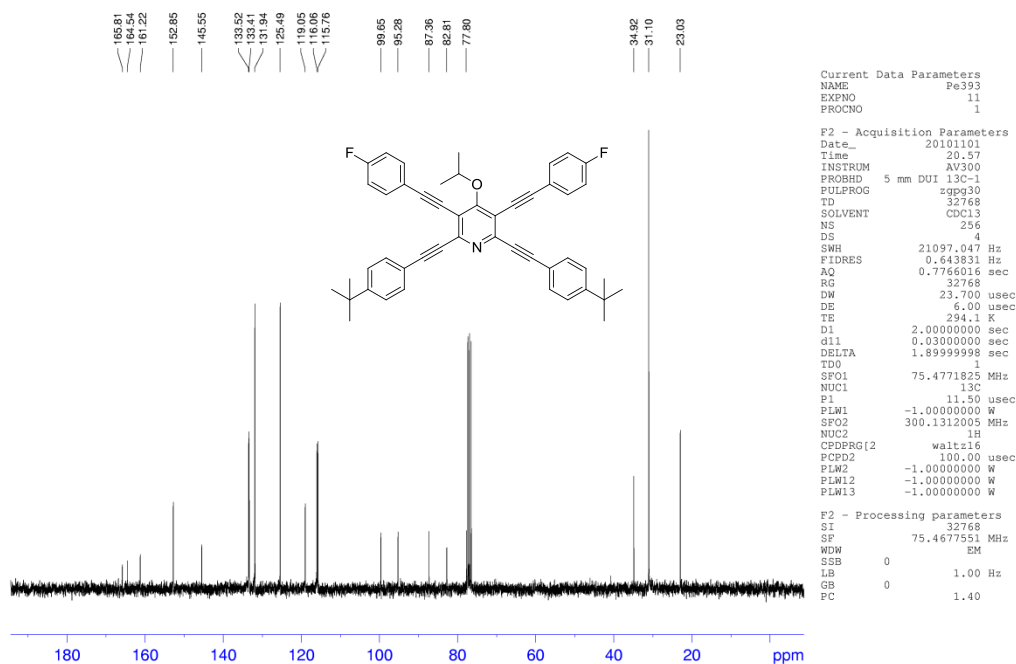
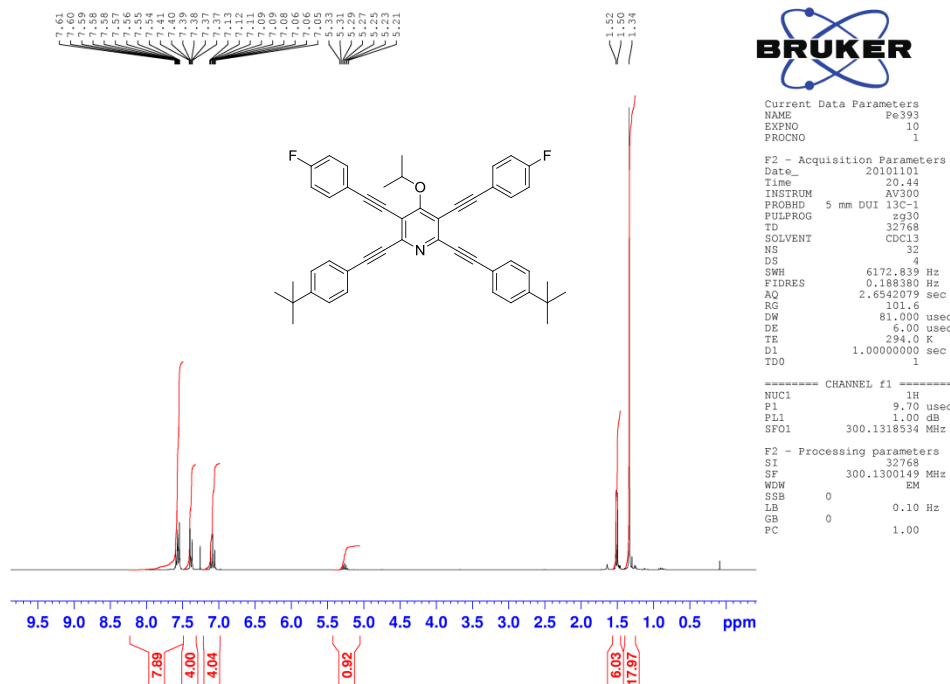
¹H- and ¹³C-NMR spectra of 12d

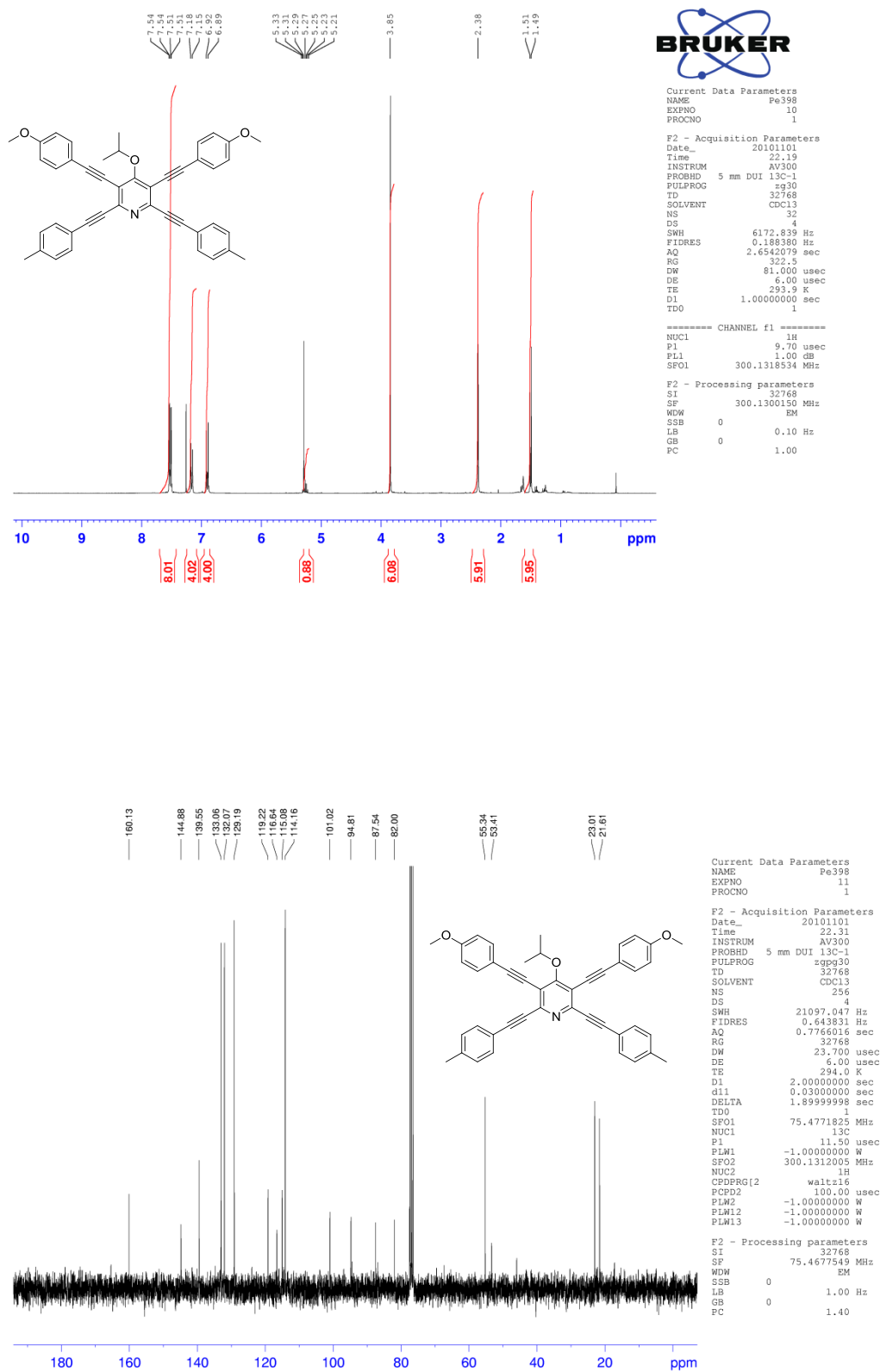
¹H- and ¹³C-NMR spectra of 12e

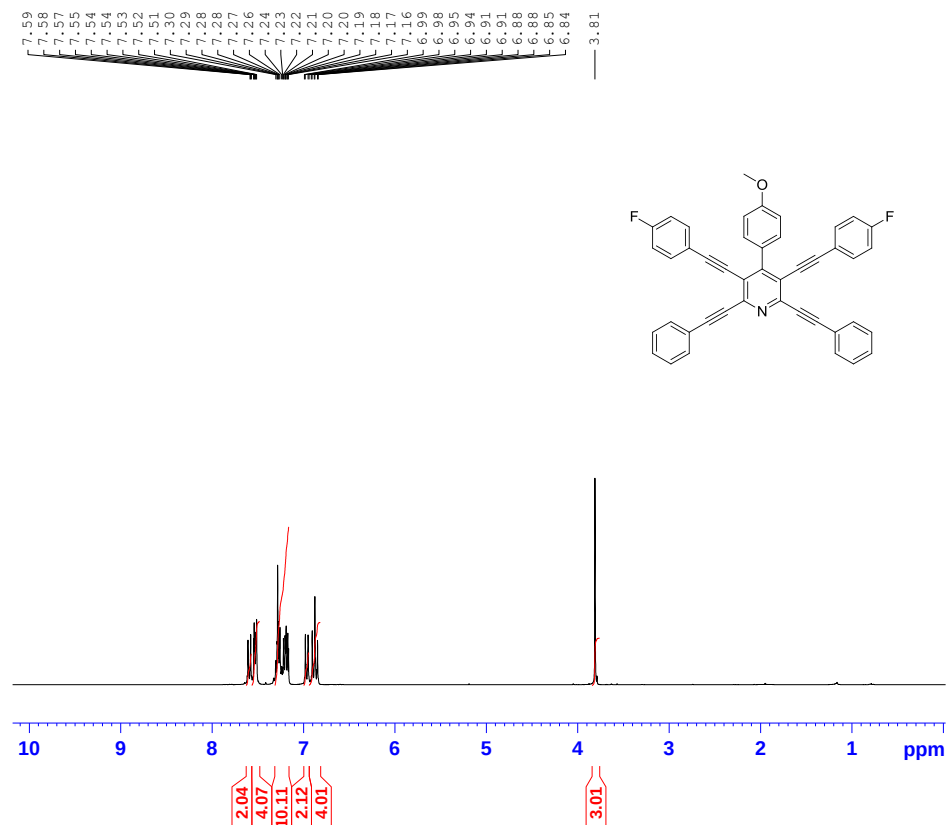
¹H-NMR spectra of 12f at 70°C

¹H- and ¹³C-NMR spectra of 12g

¹H- and ¹³C-NMR spectra of 12h

¹H- and ¹³C-NMR spectra of 13a

¹H- and ¹³C-NMR spectra of 13b

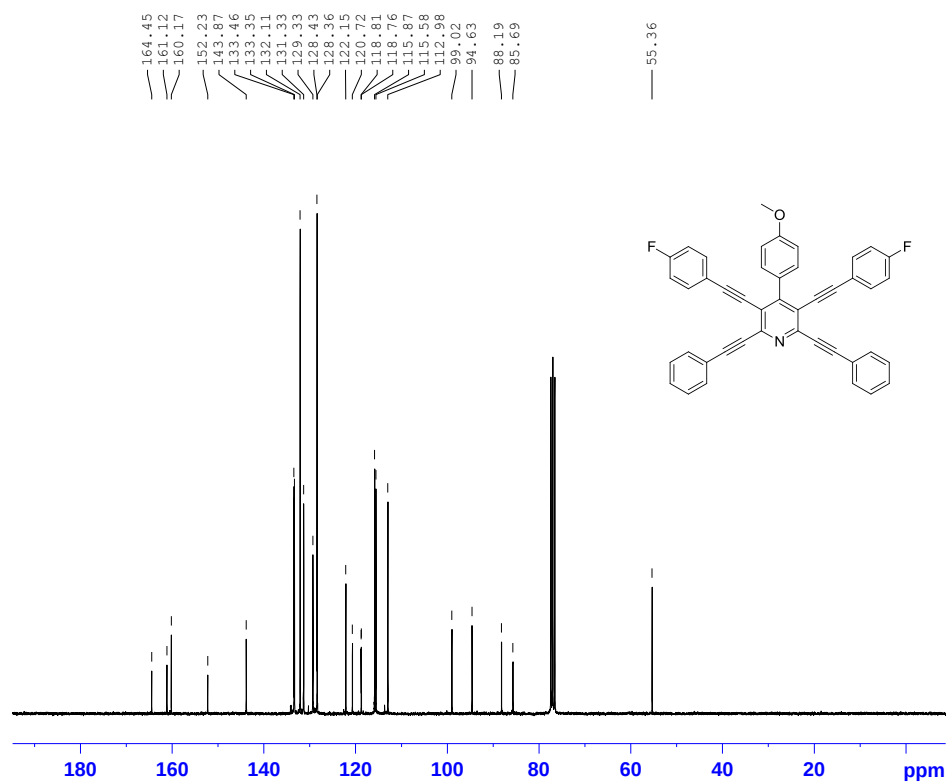
¹H- and ¹³C-NMR spectra of 14

Current Data Parameters
NAME AP-258
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121207
Time_ 4.03
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542079 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0 dB
SFO1 300.1318534 MHz

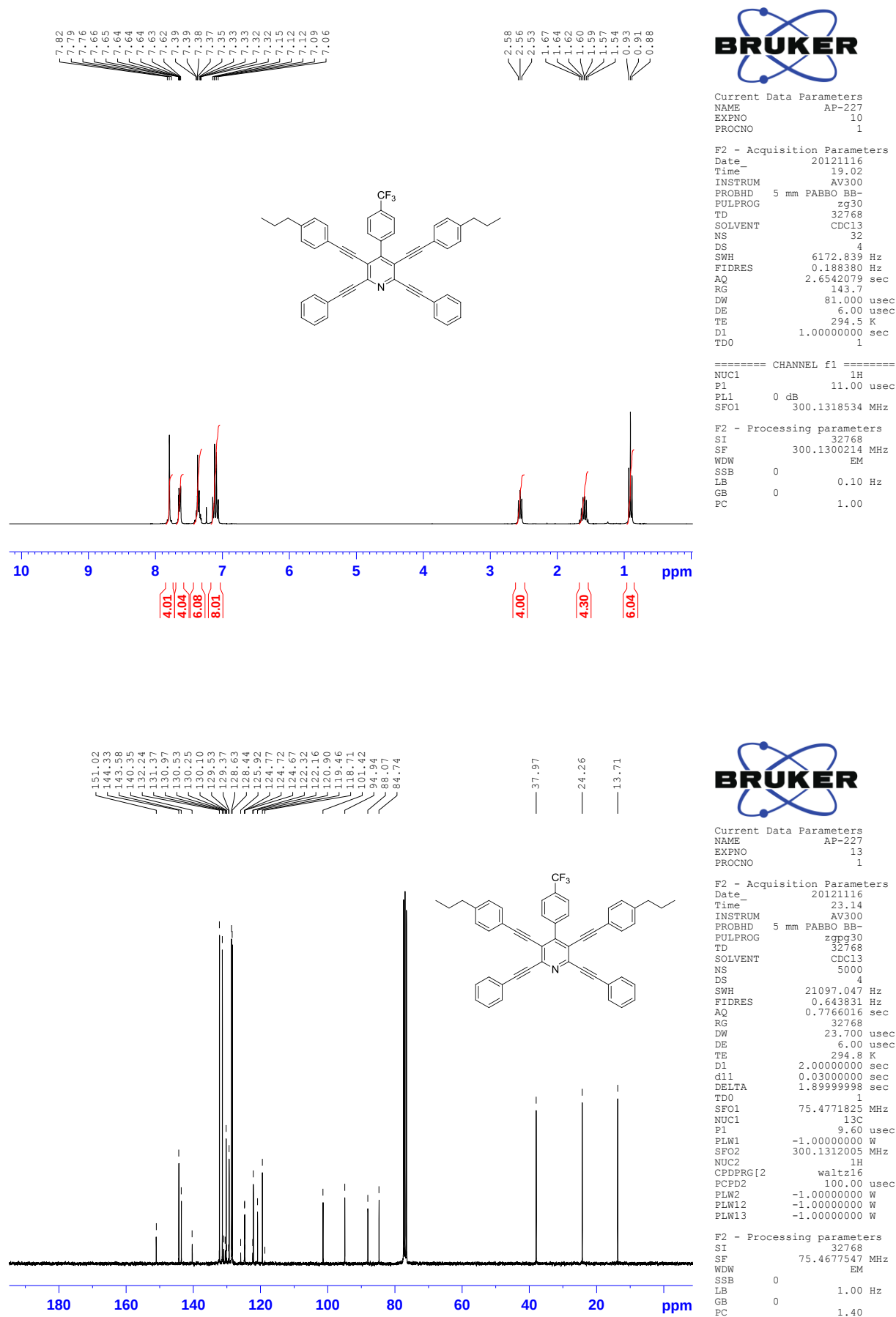
F2 - Processing parameters
SI 32768
SF 300.1300441 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

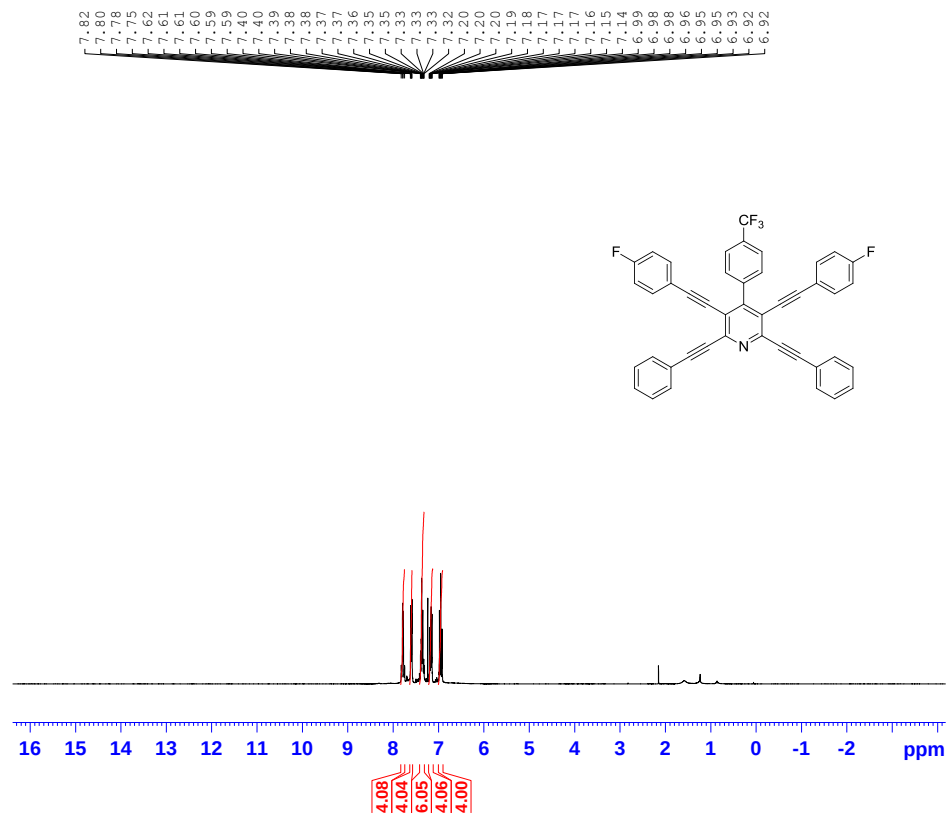


Current Data Parameters
NAME AP-258
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121207
Time_ 8.00
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 5000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766016 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 293.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4771825 MHz
NUC1 13C
P1 9.60 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 75.4677573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H- and ¹³C-NMR spectra of 15a

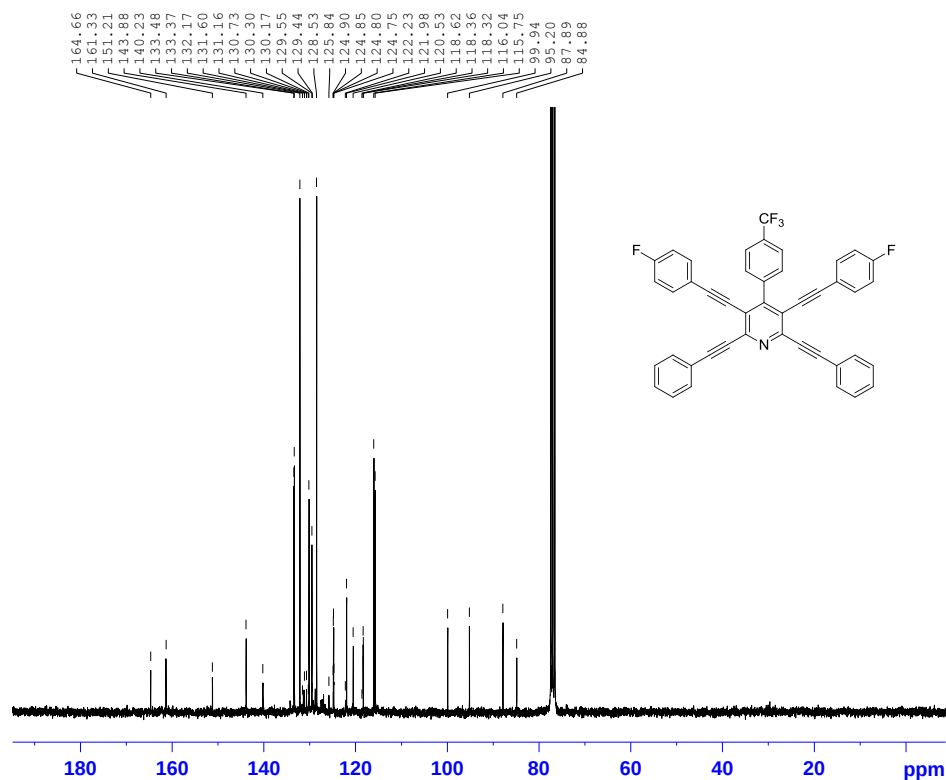
¹H- and ¹³C-NMR spectra of 15b

Current Data Parameters
 NAME AP-229
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121128
 Time_ 3.26
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 294.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300214 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

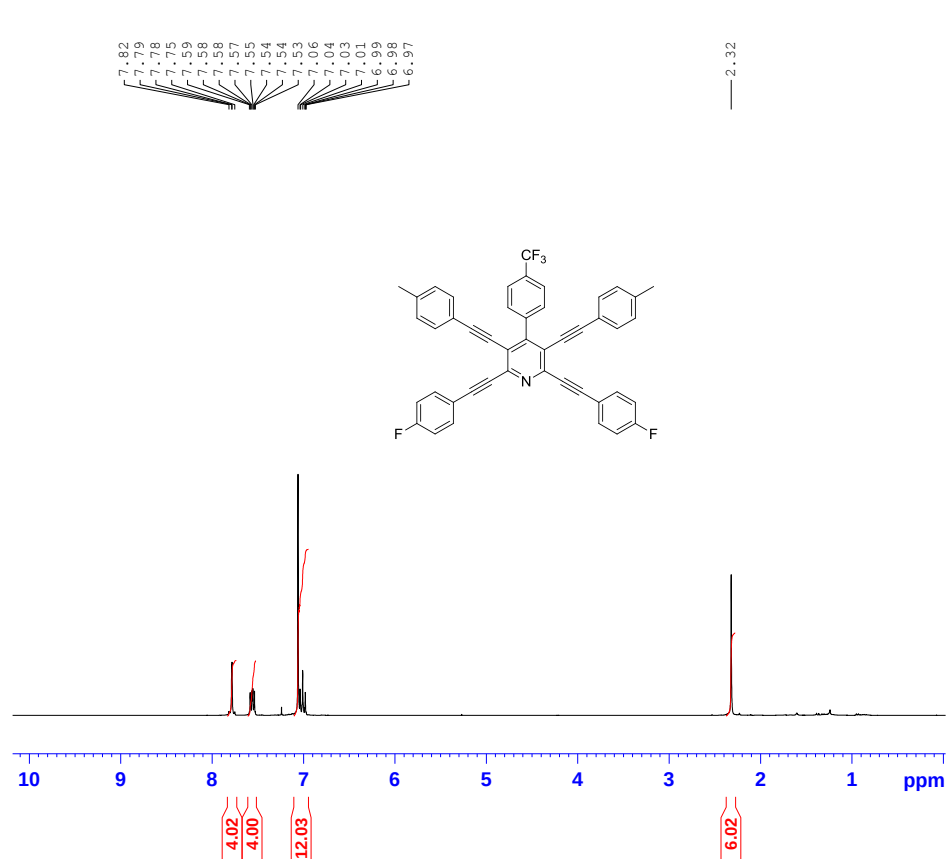


Current Data Parameters
 NAME AP-229
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121128
 Time_ 7.38
 INSTRUM AV300
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl₃
 NS 5000
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 294.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SF01 75.4771825 MHz

NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SF02 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 FCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677534 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

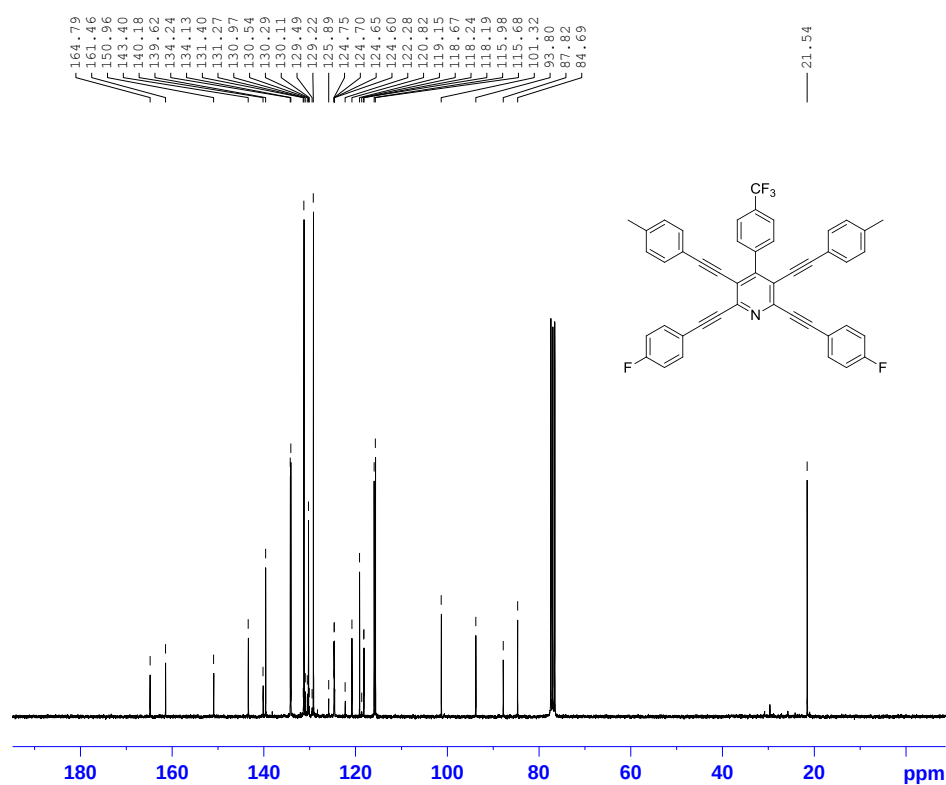
¹H- and ¹³C-NMR spectra of 15c

Current Data Parameters
 NAME AP-260
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121205
 Time_ 4.27
 INSTRUM AV300
 PROBD 5 mm PABBO BB-
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542079 sec
 RG 128
 DW 81.000 usec
 DE 6.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.00 usec
 PL1 0 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300214 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME AP-260
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121205
 Time_ 8.34
 INSTRUM AV300
 PROBD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 5000
 DS 4
 SWH 21097.047 Hz
 FIDRES 0.643831 Hz
 AQ 0.7766016 sec
 RG 32768
 DW 23.700 usec
 DE 6.00 usec
 TE 297.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1
 SF01 75.4771825 MHz
 NUC1 13C
 P1 9.60 usec
 PLW1 -1.00000000 W
 SF02 300.1312005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 PLW2 -1.00000000 W
 PLW12 -1.00000000 W
 PLW13 -1.00000000 W

F2 - Processing parameters
 SI 32768
 SF 75.4677556 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40