Electronic Supplementary Material (ESI) for RSC Advances.


# Supplementary Information

# Synthesis of Benzofulvenes through Chemoselective Sonogashira, Barluenga Couplings of ortho Ethynyl-*N*-Tosylhydrazones and Cycloisomerization

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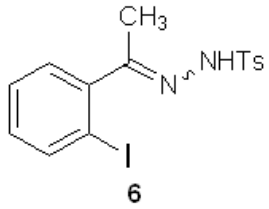
## CONTENTS

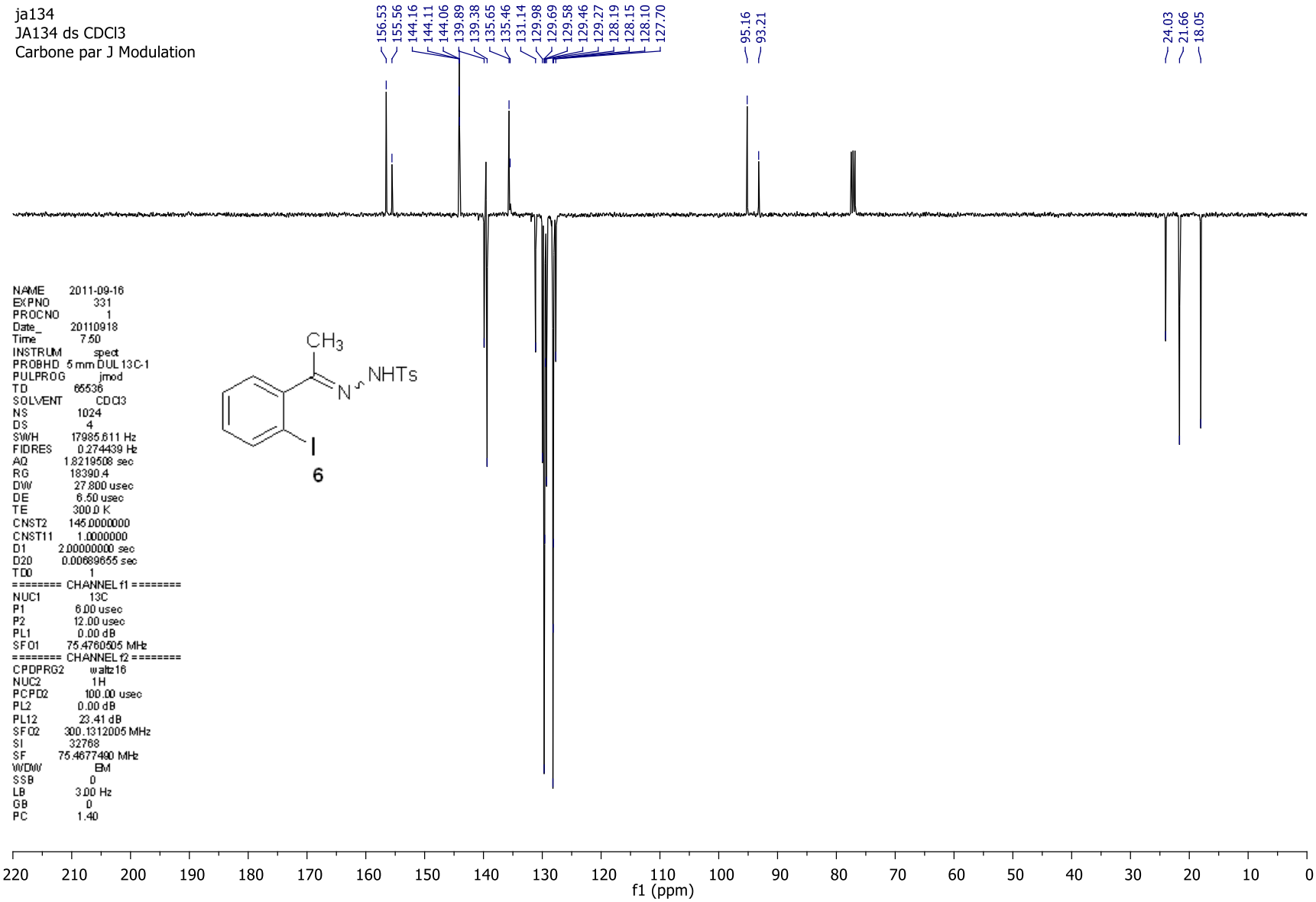
ja134
JA134 ds CDCl3
PROTON

NAME 2011-09-16
EXPNO 50
PROCNO 1
Date_ 20110916
Time 10.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




6

ja134
JA134 ds CDCl3
Carbone par J Modulation

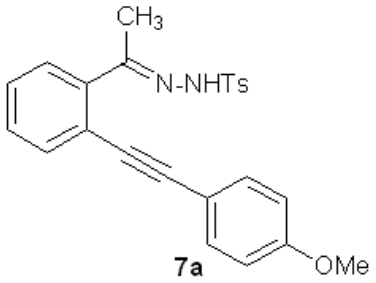



NAME 2011-09-16
EXPNO 331
PROCNO 1
Date_ 20110918
Time 7.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

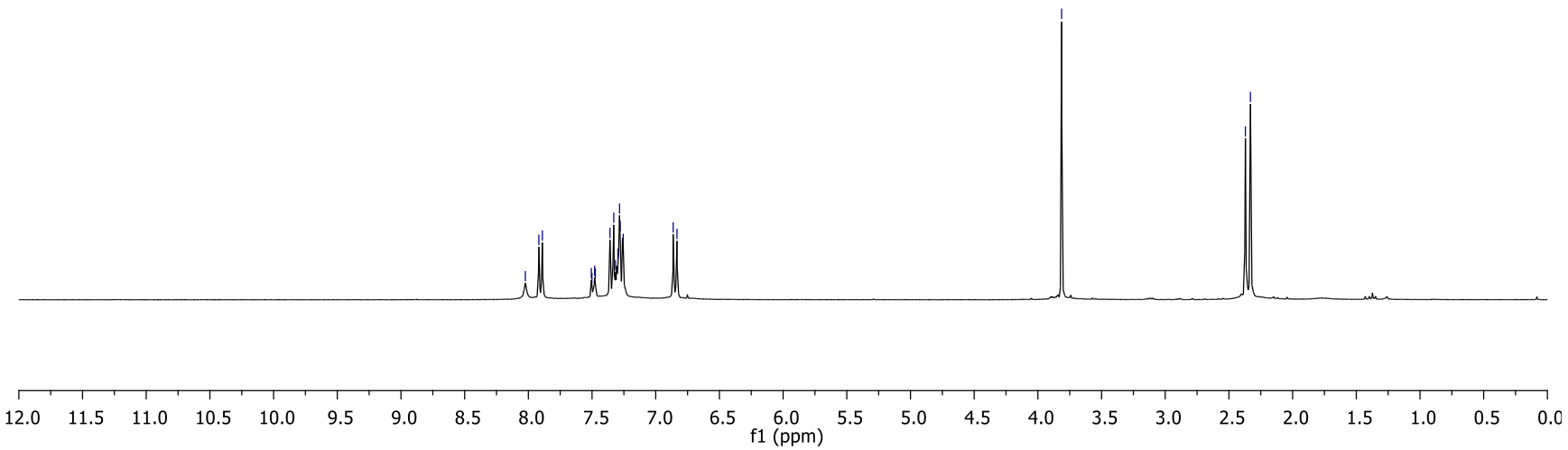
2012-02-23
JA257
Proton.4 CDCl3 D:\\ chit 55

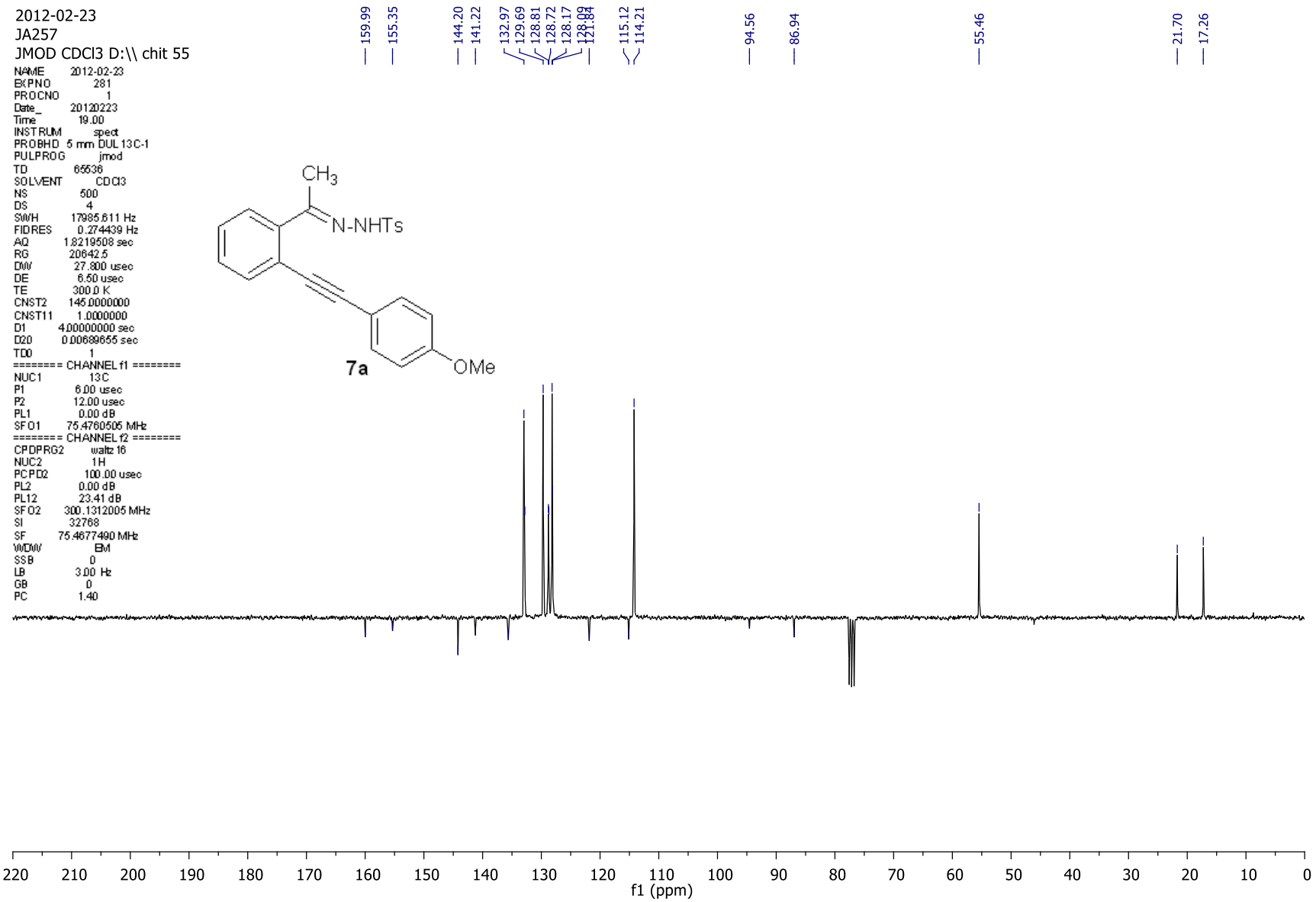
8.02 7.92 7.89 7.36 7.33 7.28 7.26 6.86 6.83 3.81 2.37 2.33

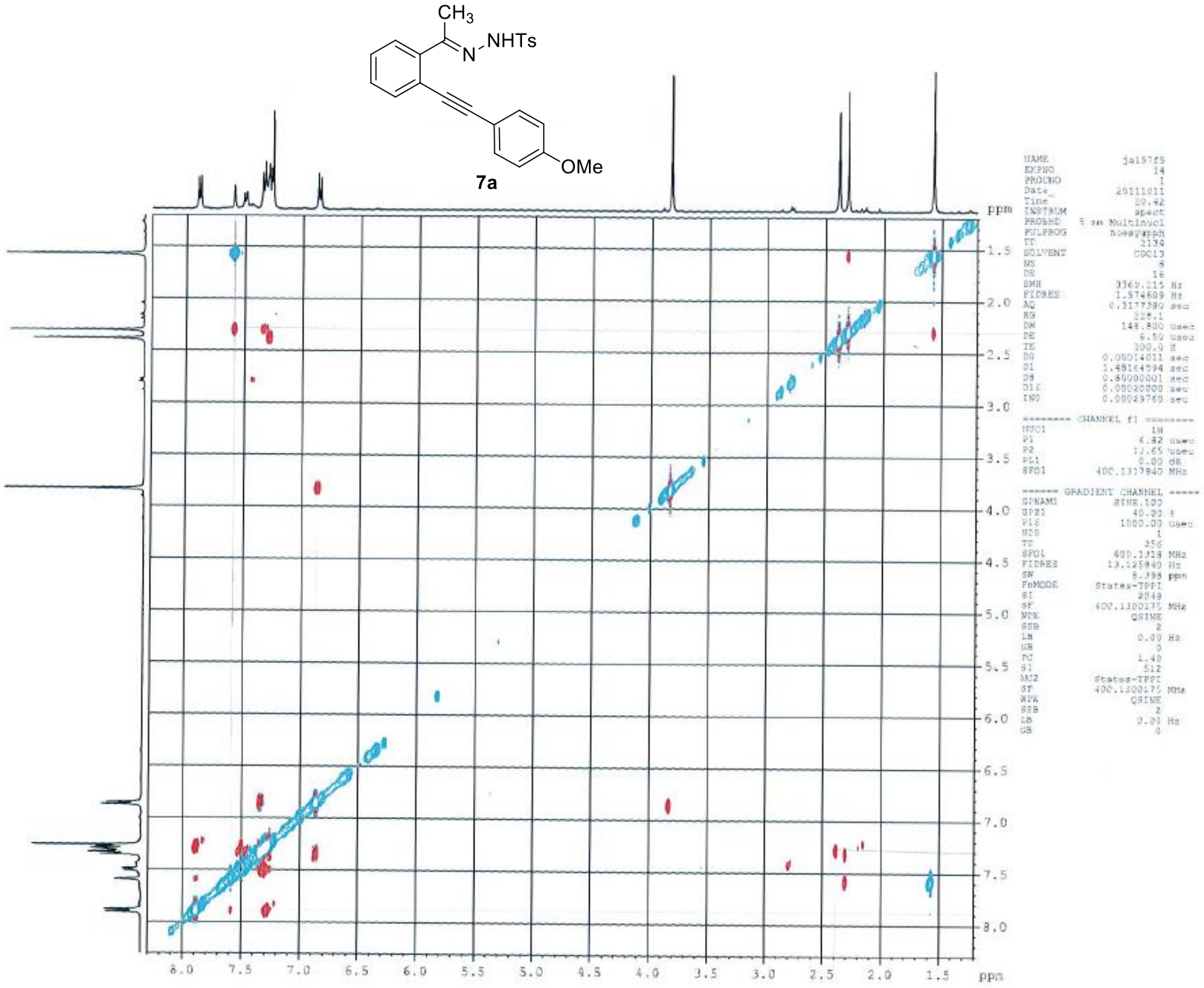
NAME 2012-02-23
EXPNO 280
PROCNO 1
Date_ 20120223
Time 18.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 812.7
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300283 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




**7a**




2012-02-23
JA257
JMOD CDCl3 D:\\ chit 55
159.99
155.35
144.20
141.22
132.97
129.69
128.81
128.72
128.17
115.12
114.21
94.56
86.94
55.46
21.70
17.26
NAME 2012-02-23
EXPNO 281
PROCNO 1
Date_ 20120223
Time 19.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
N-NHTs
7a
OMe
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

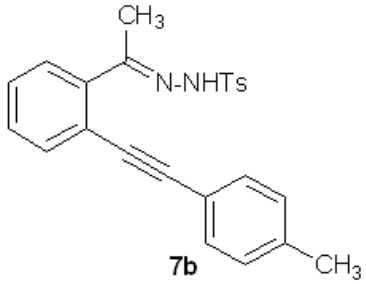

CH3
NHTs
N
OMe
7a
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
NAME ja19f5
EXPNO 14
PROCNO 1
Date_ 20111011
Time 19.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG noesygpph
TD 2134
SOLVENT CDCl3
NS 8
DS 16
SWH 3369.115 Hz
FIDRES 1.574609 Hz
AQ 0.3177380 sec
RG 328.1
DW 148.800 usec
DE 6.50 usec
TE 300.0 K
D0 0.00014011 sec
D1 1.48164594 sec
D8 0.40000001 sec
D16 0.00020000 sec
IN0 0.00023769 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.82 usec
P2 13.65 usec
PL1 0.00 dB
SFO1 400.1317940 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPZ1 40.00 %
PL6 1000.00 usec
NSP 1
TP 256
SFO1 400.1318 MHz
FIDRES 13.125840 Hz
SW 8.398 ppm
FnMODE States-TPPI
SI 2048
SF 400.1300175 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 512
MC2 States-TPPI
SF 400.1300175 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

ja279
JA279 dans CDCl3
PROTON

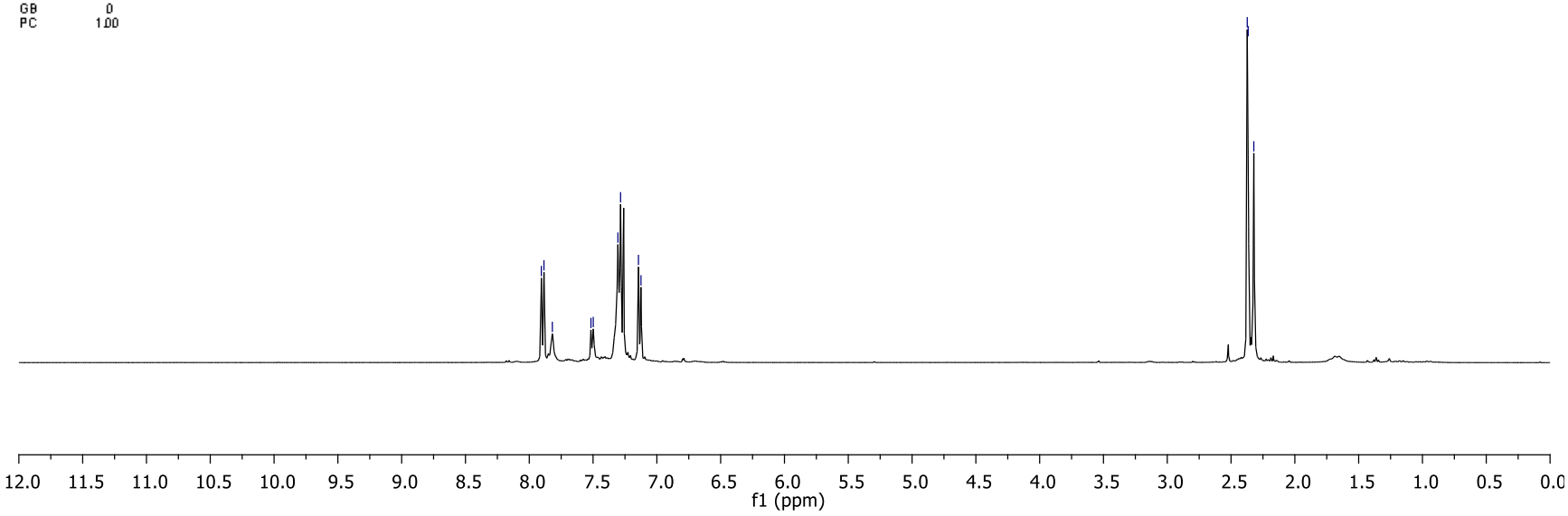
7.91
7.88
7.82
7.52
7.50
7.31
7.28
7.14
7.12

2.37
2.37
2.32

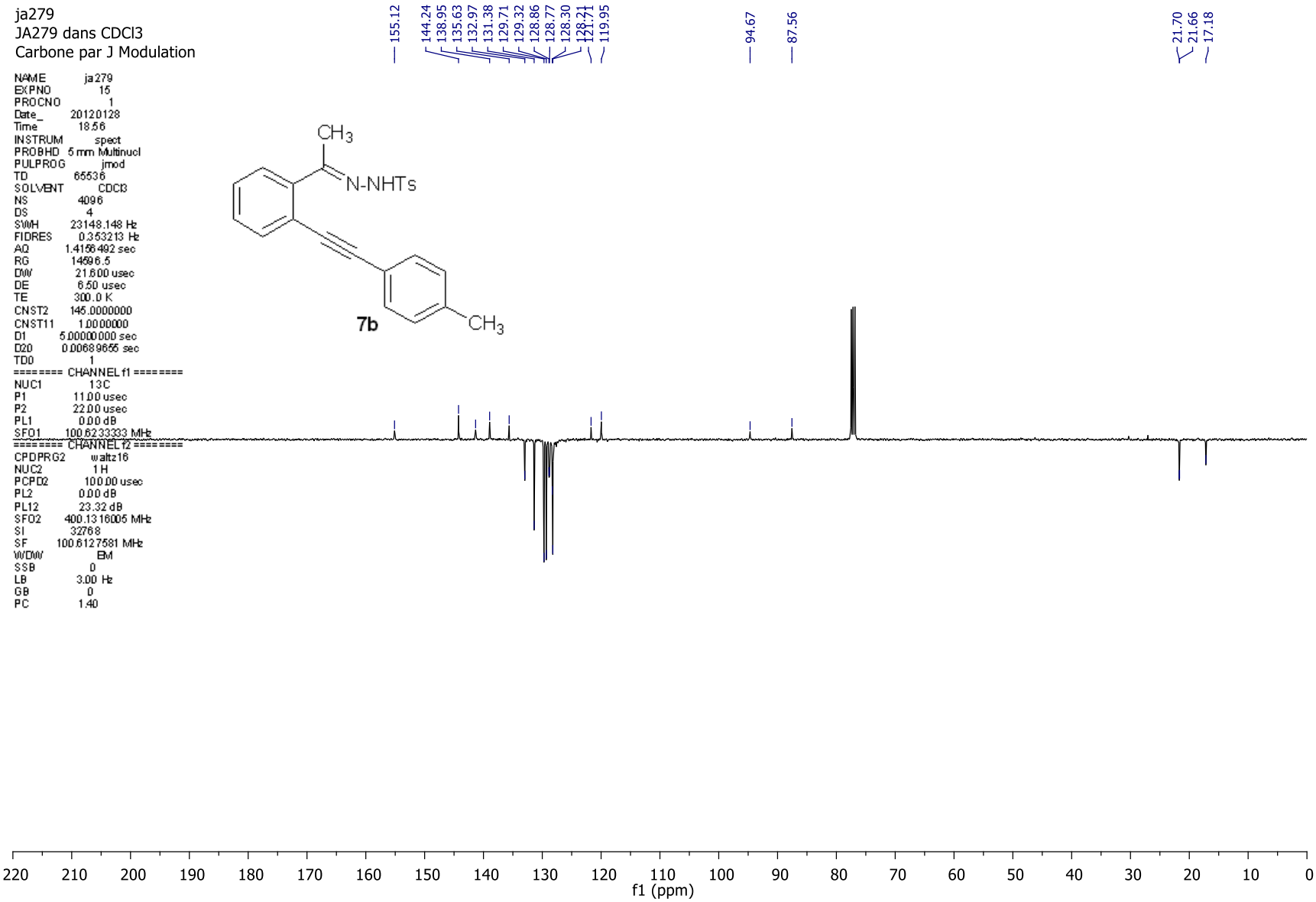
NAME ja279
EXPNO 10
PROCNO 1
Date_ 20120128
Time 8.35
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715922 sec
RG 90.5
DW 60.600 usec
DE 6.50 usec
TE 300.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.82 usec
PL1 0.00 dB
SFO1 400.1336012 MHz
SI 32768
SF 400.1300181 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



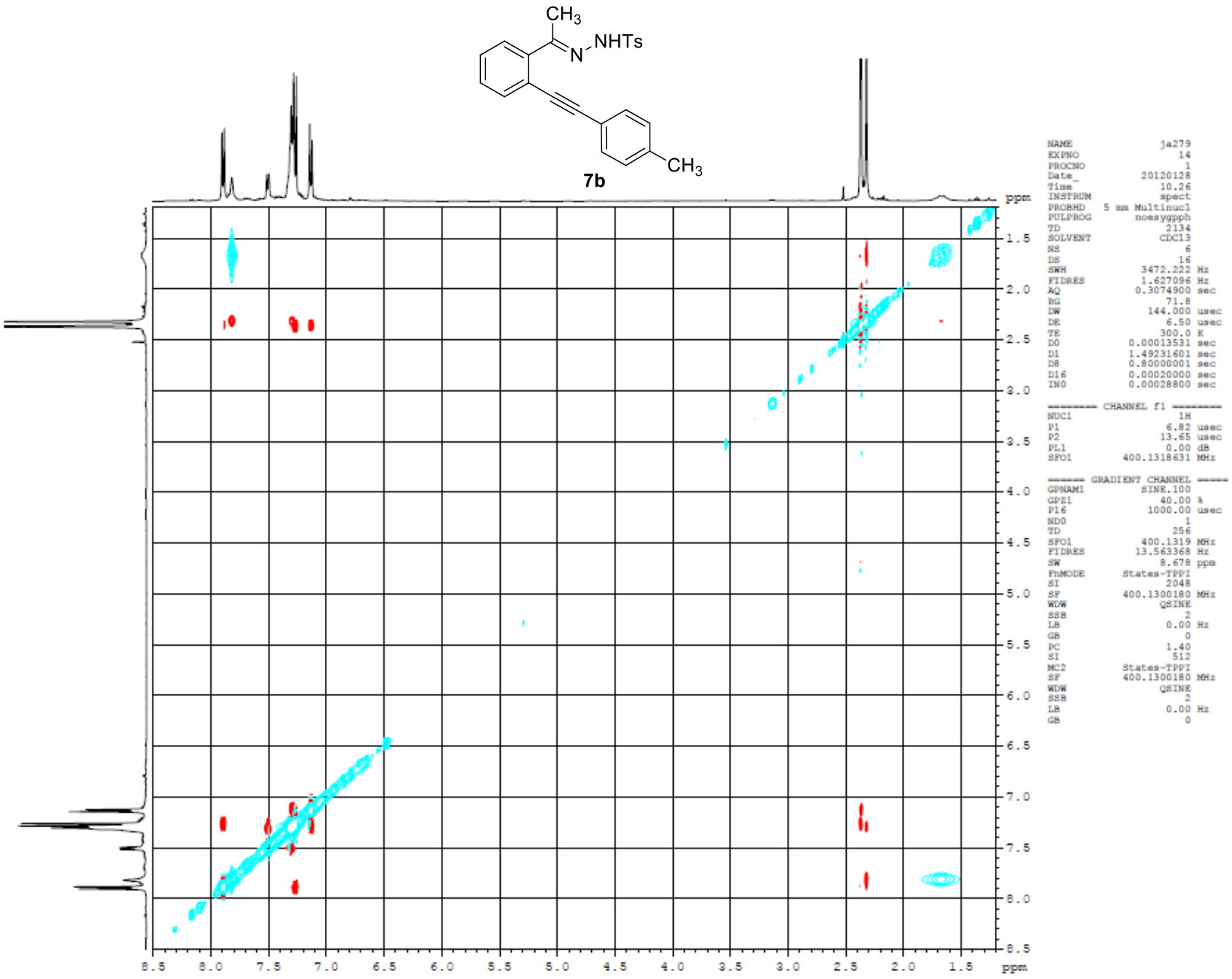
7b



ja279
JA279 dans CDCl3
Carbone par J Modulation




NAME ja279
EXPNO 15
PROCNO 1
Date_ 20120128
Time 18.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156492 sec
RG 14596.5
DW 21.600 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 5.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
P2 22.00 usec
PL1 0.00 dB
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.32 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127581 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40


CH3
NHTs
N
CH3
7b
ppm
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
NAME ja279
EXPNO 14
PROCNO 1
Date_ 20120128
Time 10.26
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG noesygpph
TD 2134
SOLVENT CDCl3
NS 6
DS 16
SWH 3472.222 Hz
FIDRES 1.627096 Hz
AQ 0.3074900 sec
RG 71.8
DW 144.000 usec
DE 6.50 usec
TE 300.0 K
D0 0.00013531 sec
D1 1.49231601 sec
D8 0.80000001 sec
D16 0.00020000 sec
IN0 0.00028800 sec
CHANNEL f1
NUC1 1H
P1 6.82 usec
P2 13.65 usec
PL1 0.00 dB
SFO1 400.1318631 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPZ1 40.00 %
P16 1000.00 usec
ND0 1
TD 256
SFO1 400.1319 MHz
FIDRES 13.563368 Hz
SW 8.678 ppm
FnMODE States-TPPI
SI 2048
SF 400.1300180 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 512
MC2 States-TPPI
SF 400.1300180 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

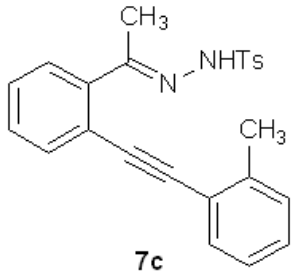
2012-04-20
JA277 DMSO
Proton.4 DMSO D:\\chit 20

10.63

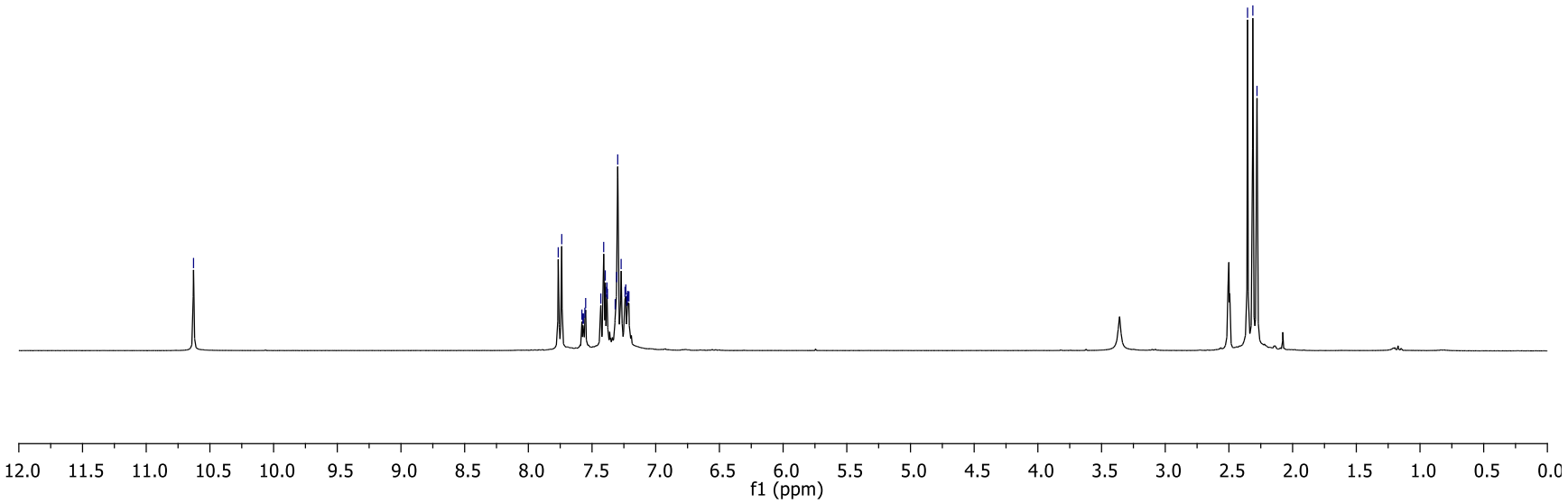
7.77 7.74 7.58 7.57 7.57 7.56 7.55 7.43 7.41 7.40 7.39 7.38 7.38 7.32 7.31 7.30 7.27 7.24 7.24 7.23 7.22 7.22 7.21

2.35 2.31 2.28

NAME 2012-04-20
EXPNO 230
PROCNO 1
Date_ 20120423
Time 8.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




**7c**

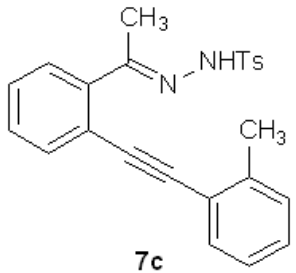


2012-04-20
JA277 DMSO
JMOD DMSO D:\\ chit 20

155.22 143.15 141.64 139.55 136.33 132.64 131.61 129.57 129.34 128.87 128.73 128.65 128.13 127.30 125.82 121.92 120.52

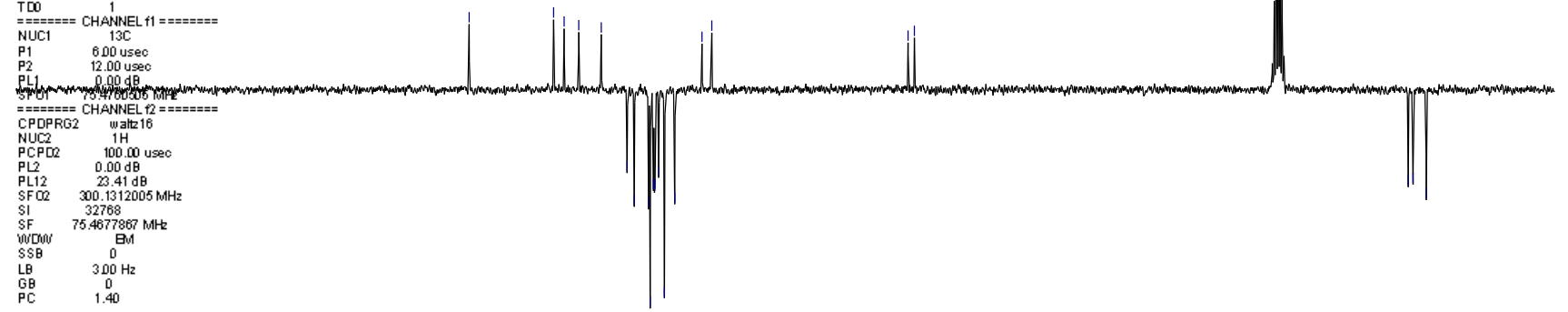
92.45 91.55

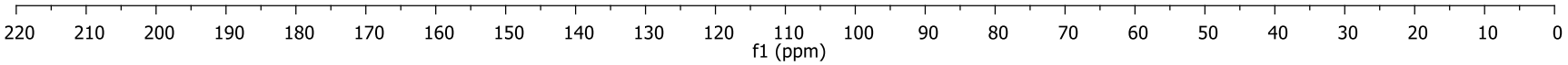
20.92 20.23 18.35




**7c**

EXPNO 231
PROCNO 1
Date_ 20120423
Time 8.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 83
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4766805 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

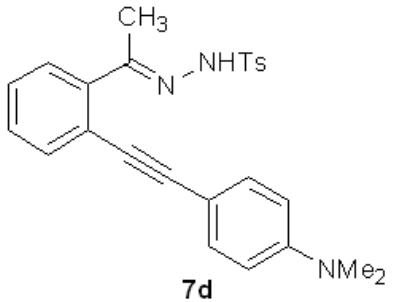




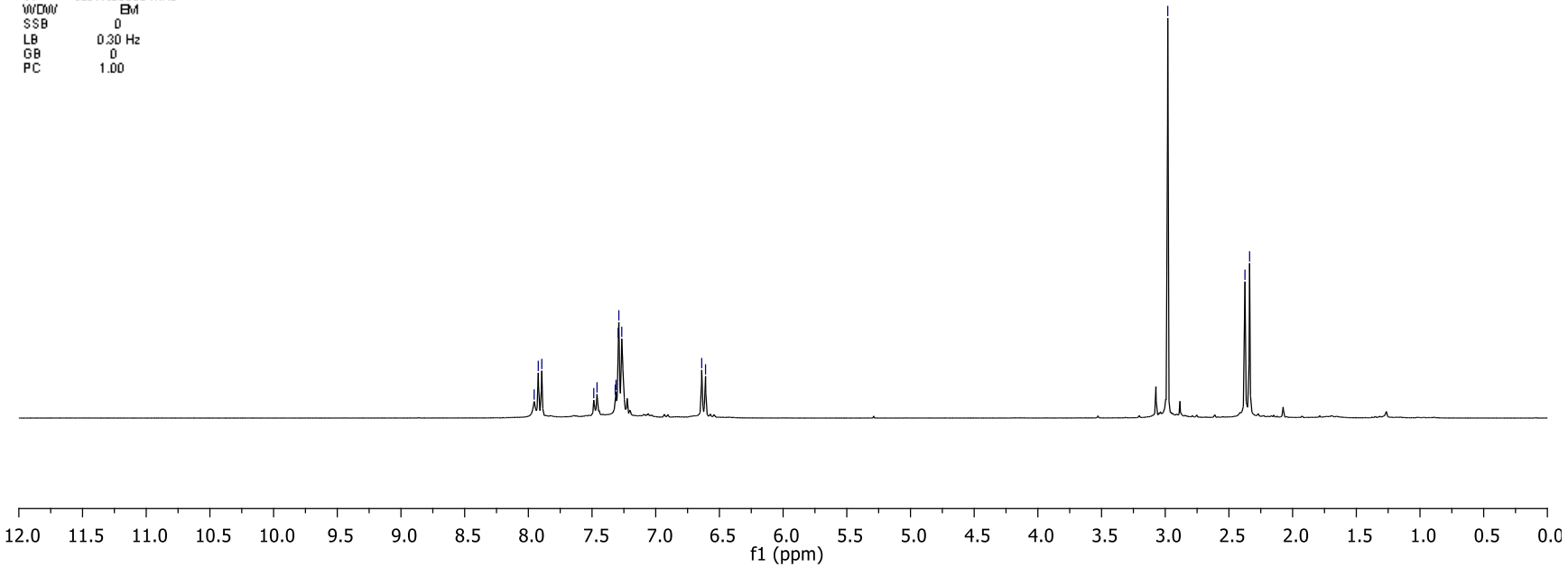
2012-02-15
JA301F4
Proton.4 CDCl3 D:\\ chit 40

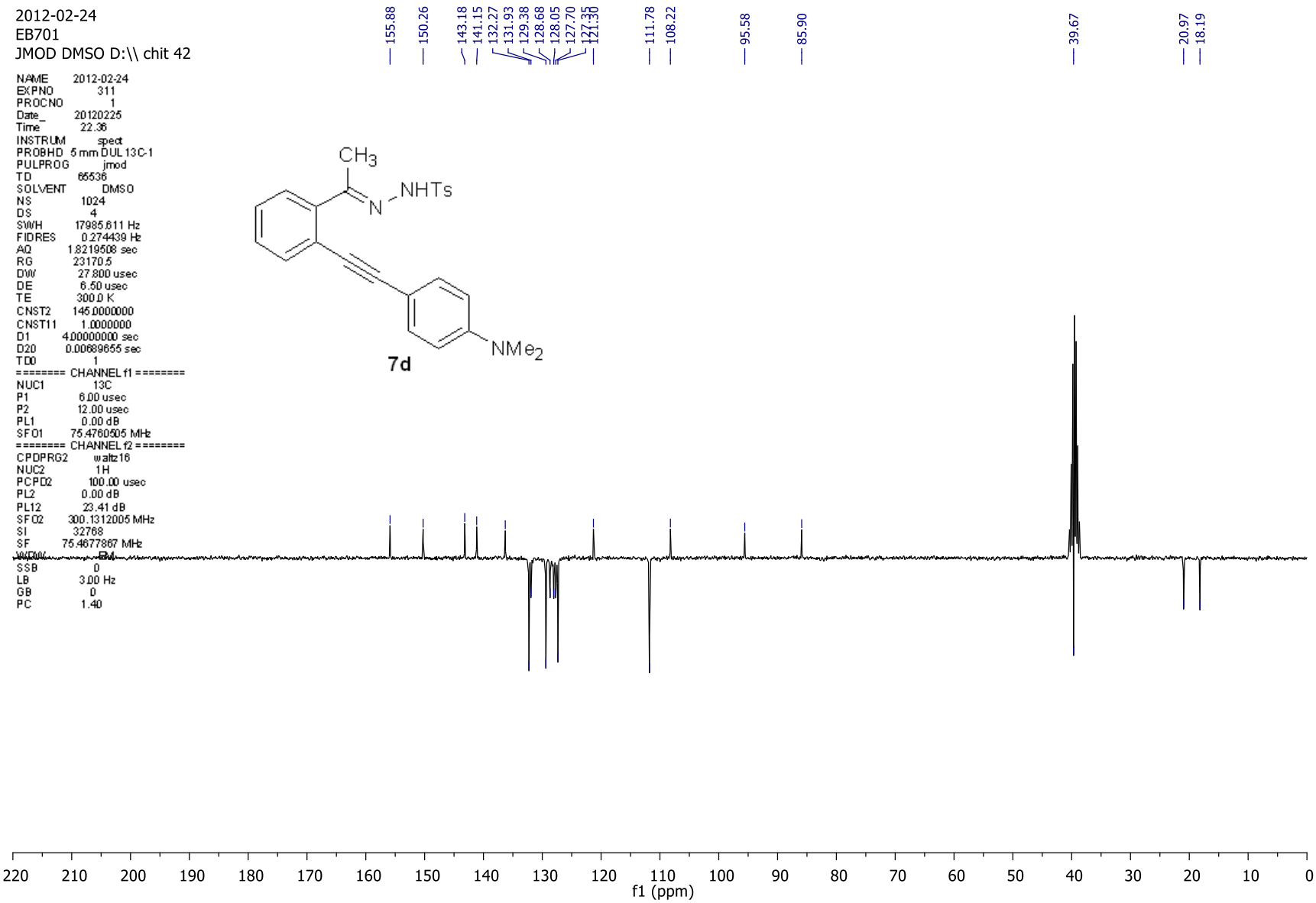
7.95
7.92
7.89
7.46
7.32
7.31
7.30
7.29
7.27
6.64
6.61
2.98
2.37
2.34

NAME 2012-02-15
EXPNO 320
PROCNO 1
Date_ 20120216
Time 6.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




**7d**

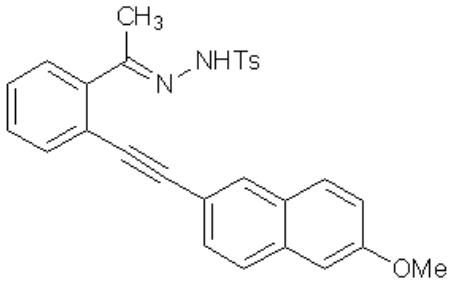



2012-02-24
EB701
JMOD DMSO D:\\ chit 42
155.88
150.26
143.18
141.15
132.27
131.93
129.38
128.68
128.05
127.70
127.35
121.30
111.78
108.22
95.58
85.90
39.67
20.97
18.19
NAME 2012-02-24
EXPNO 311
PROCNO 1
Date_ 20120225
Time 22.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 23170.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
NHTs
N
NMe2
7d
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

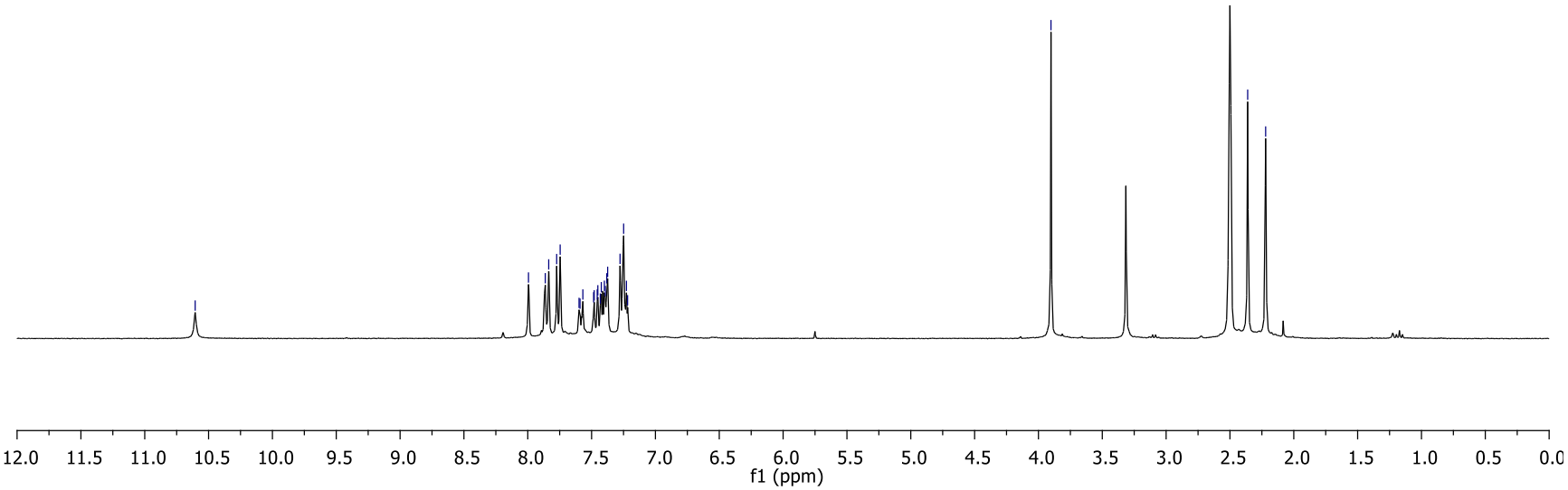
2012-02-24
JA296
Proton.4 DMSO D:\\ chit 36

10.60
7.99
7.86
7.84
7.77
7.75
7.60
7.59
7.57
7.48
7.48
7.46
7.45
7.43
7.42
7.40
7.39
7.38
7.37
7.28
7.25
7.23
7.22
3.90
2.36
2.22

NAME 2012-02-24
EXPNO 200
PROCNO 1
Date_ 20120224
Time 11.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



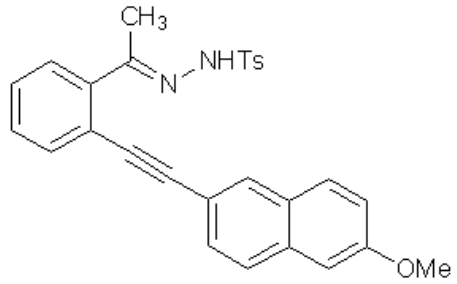

**7e**



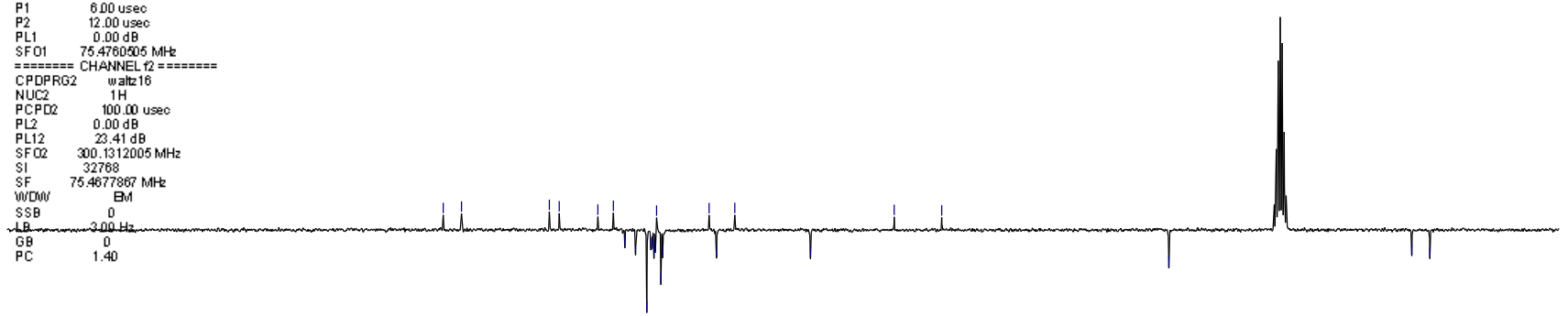
2012-02-24
JA296
JMOD DMSO D:\\ chit 36

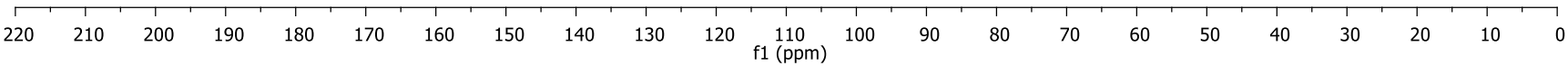
158.18
155.59
143.14
141.74
130.92
129.33
128.31
128.11
127.32
120.49
119.44
116.85
106.12
94.25
87.51
55.31
20.87
18.31

NAME 2012-02-24
EXPNO 301
PROCNO 1
Date_ 20120225
Time 14.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



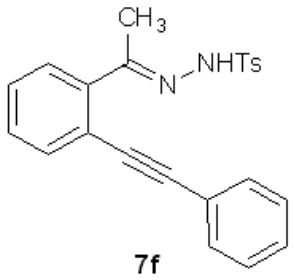

**7 e**



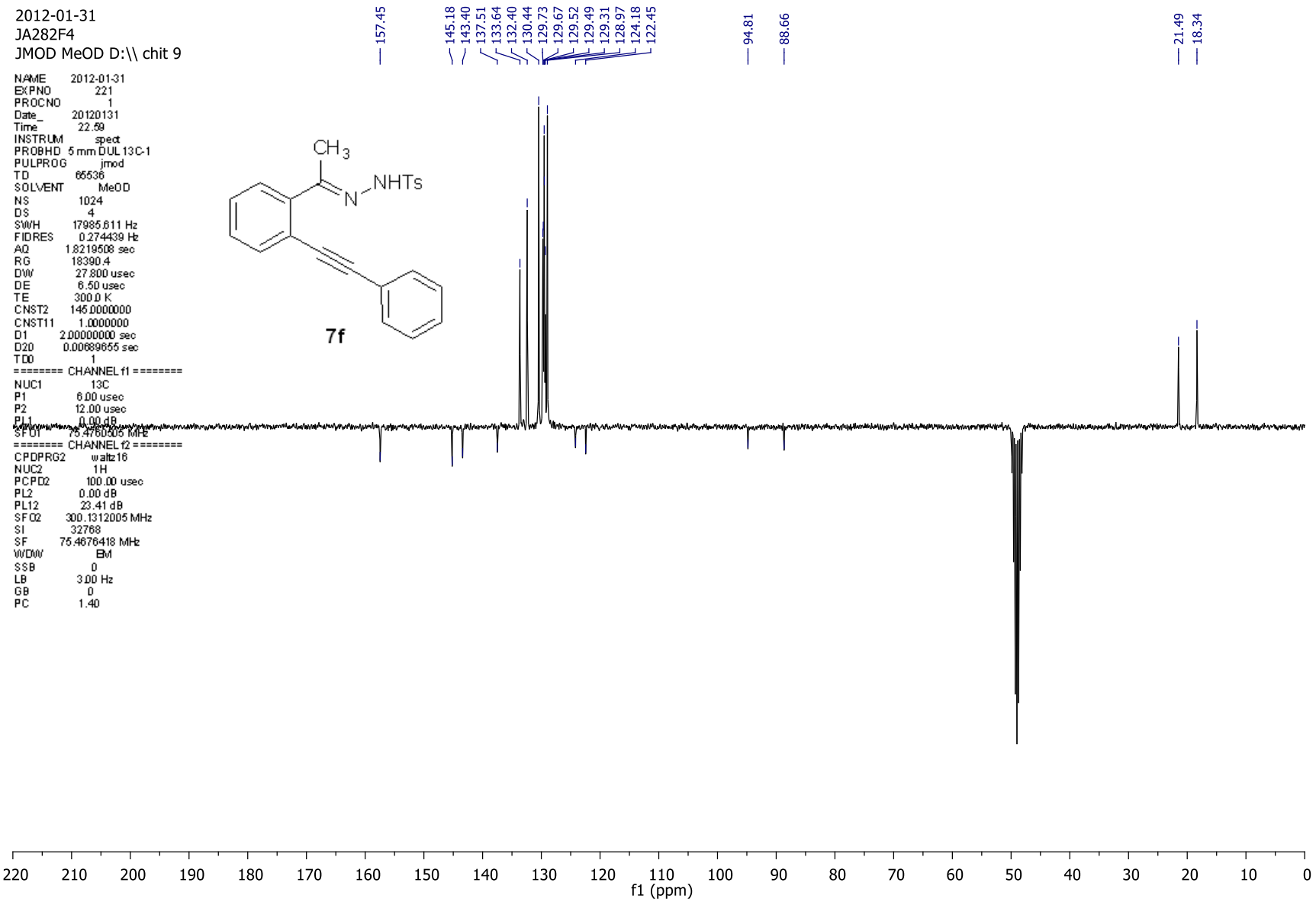


2012-01-31
JA282F4
Proton.4 MeOD D:\\ chit 22

| | |
|---|---|
| NAME | 2012-01-31 |
| EXPNO | 70 |
| PROCNO | 1 |
| Date_ | 20120131 |
| Time | 13.16 |
| INSTRUM | spect |
| PROBHD | 5 mm DUL 13C-1 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | MeOD |
| NS | 4 |
| DS | 2 |
| SWH | 6172.839 Hz |
| FIDRES | 0.094190 Hz |
| AQ | 5.3084660 sec |
| RG | 256 |
| DW | 81.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 6.75 usec |
| PL1 | 0.00 dB |
| SFO1 | 300.1318534 MHz |
| SI | 32768 |
| SF | 300.1300000 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




**7f**

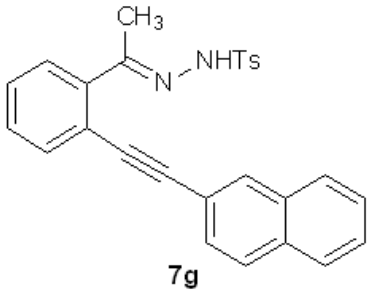

2012-01-31
JA282F4
JMOD MeOD D:\\ chit 9
NAME 2012-01-31
EXPNO 221
PROCNO 1
Date_ 20120131
Time 22.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT MeOD
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4676418 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
N
NHTs
7f
157.45
145.18
143.40
137.51
133.64
132.40
130.44
129.73
129.67
129.52
129.49
129.31
128.97
124.18
122.45
94.81
88.66
21.49
18.34
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2012-01-31
JA286F4
Proton.4 CDCl3 D:\\ chit 21

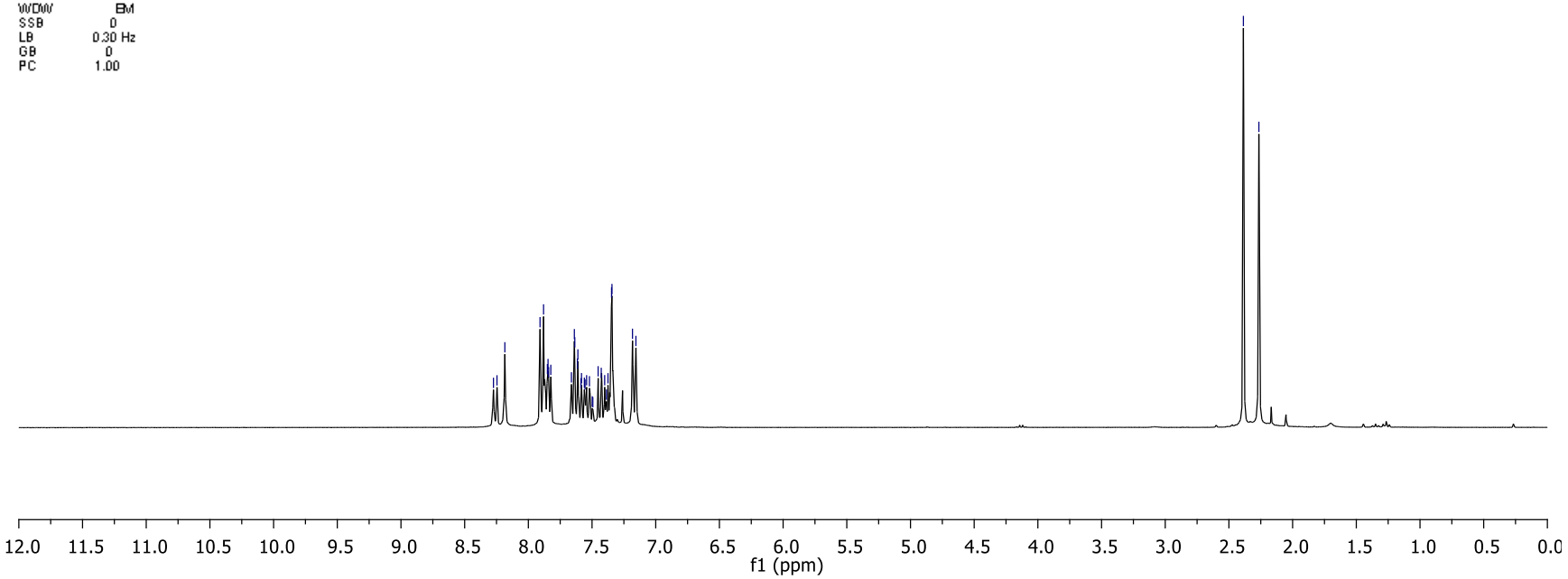
8.27 8.25 8.18 7.91 7.88 7.85 7.85 7.84 7.82 7.66 7.64 7.64 7.61 7.61 7.59 7.58 7.56 7.55 7.54 7.52 7.50 7.49 7.45 7.43 7.40 7.39 7.37 7.35 7.34 7.18 7.16

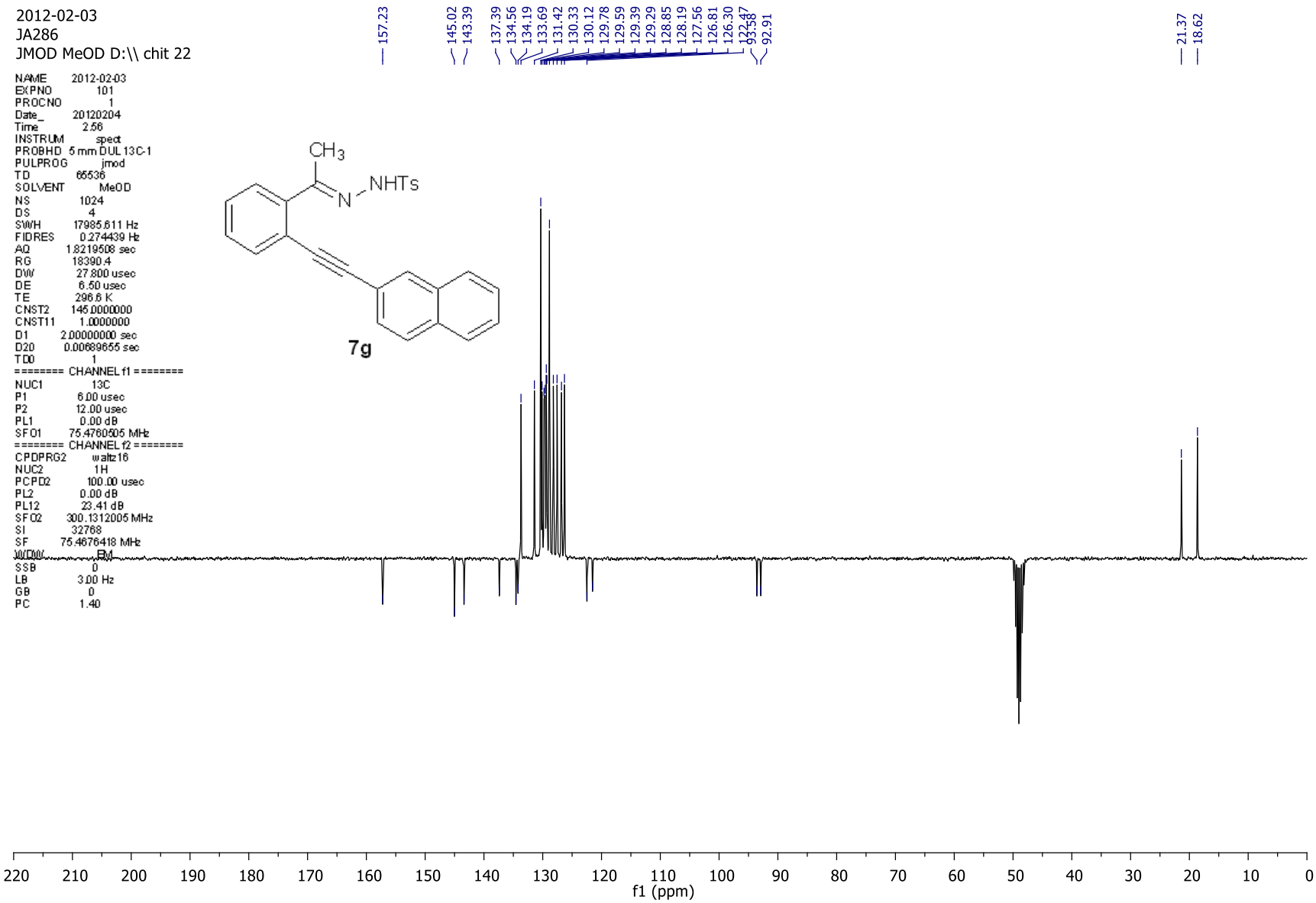
2.39 2.26

```
NAME        2012-01-31
EXPNO           240
PROCNO            1
Date_       20120201
Time            7.08
INSTRUM         spect
PROBHD  5 mm DUL 13C-1
PULPROG         zg30
TD             65536
SOLVENT         CDCl3
NS                4
DS                2
SWH          6172.839 Hz
FIDRES       0.094190 Hz
AQ           5.3084660 sec
RG              161.3
DW             81.000 usec
DE              6.50 usec
TE             300.0 K
D1          1.00000000 sec
TD0               1
======== CHANNEL f1 ========
NUC1              1H
P1              6.75 usec
PL1             0.00 dB
SFO1       300.1318534 MHz
SI             32768
SF         300.1300000 MHz
WDW               EM
SSB               0
LB              0.30 Hz
GB                0
PC              1.00
```




**7g**




2012-02-03
JA286
JMOD MeOD D:\\ chit 22
157.23
145.02
143.39
137.39
134.56
134.19
133.69
131.42
130.33
130.12
129.78
129.59
129.39
129.29
128.85
128.19
127.56
126.81
126.30
122.47
93.58
92.91
21.37
18.62
NAME 2012-02-03
EXPNO 101
PROCNO 1
Date_ 20120204
Time 2.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT MeOD
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.50 usec
TE 296.6 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4676418 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
NHTs
N
7g
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

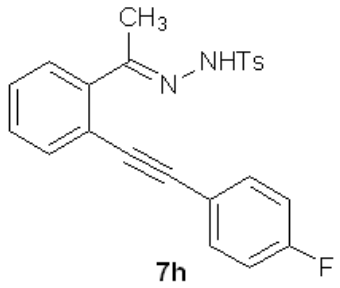
2012-05-08
JA304 DMSO
Proton.4 DMSO D:\\ chit 14

10.59

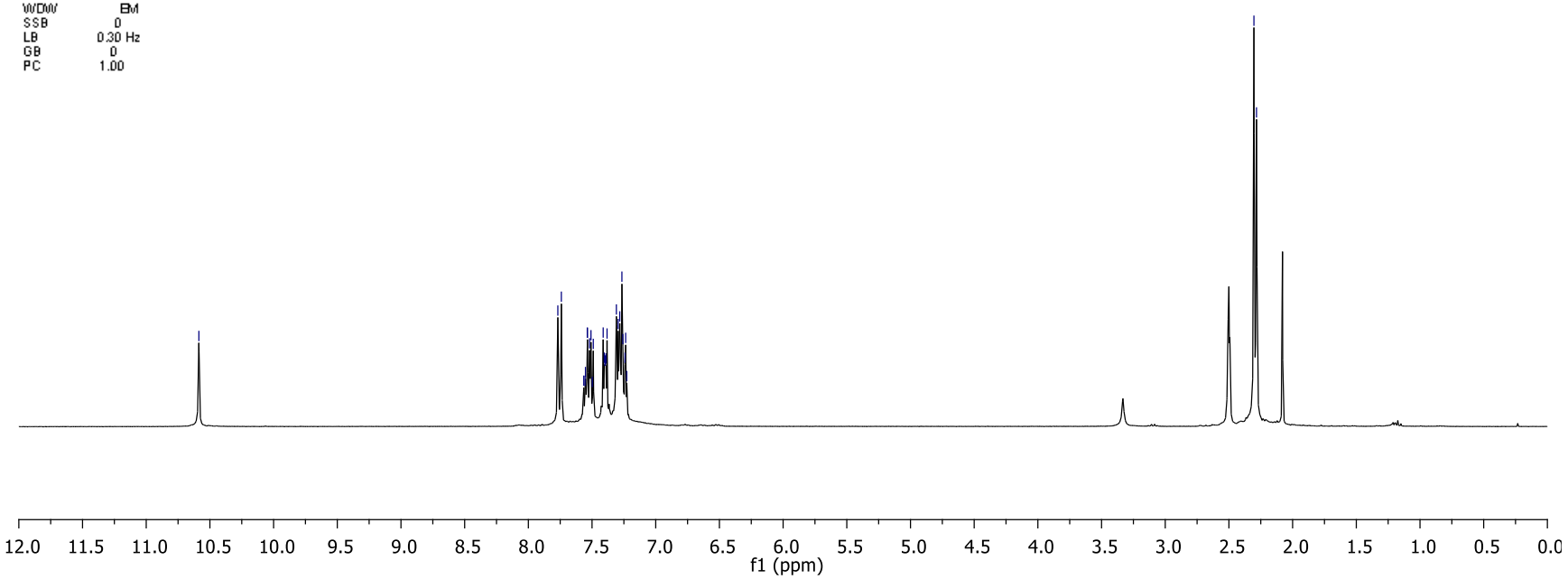
7.77 7.74 7.56 7.55 7.55 7.54 7.54 7.52 7.51 7.50 7.49 7.41 7.40 7.40 7.40 7.39 7.38 7.31 7.30 7.28 7.27 7.26 7.24 7.24 7.23

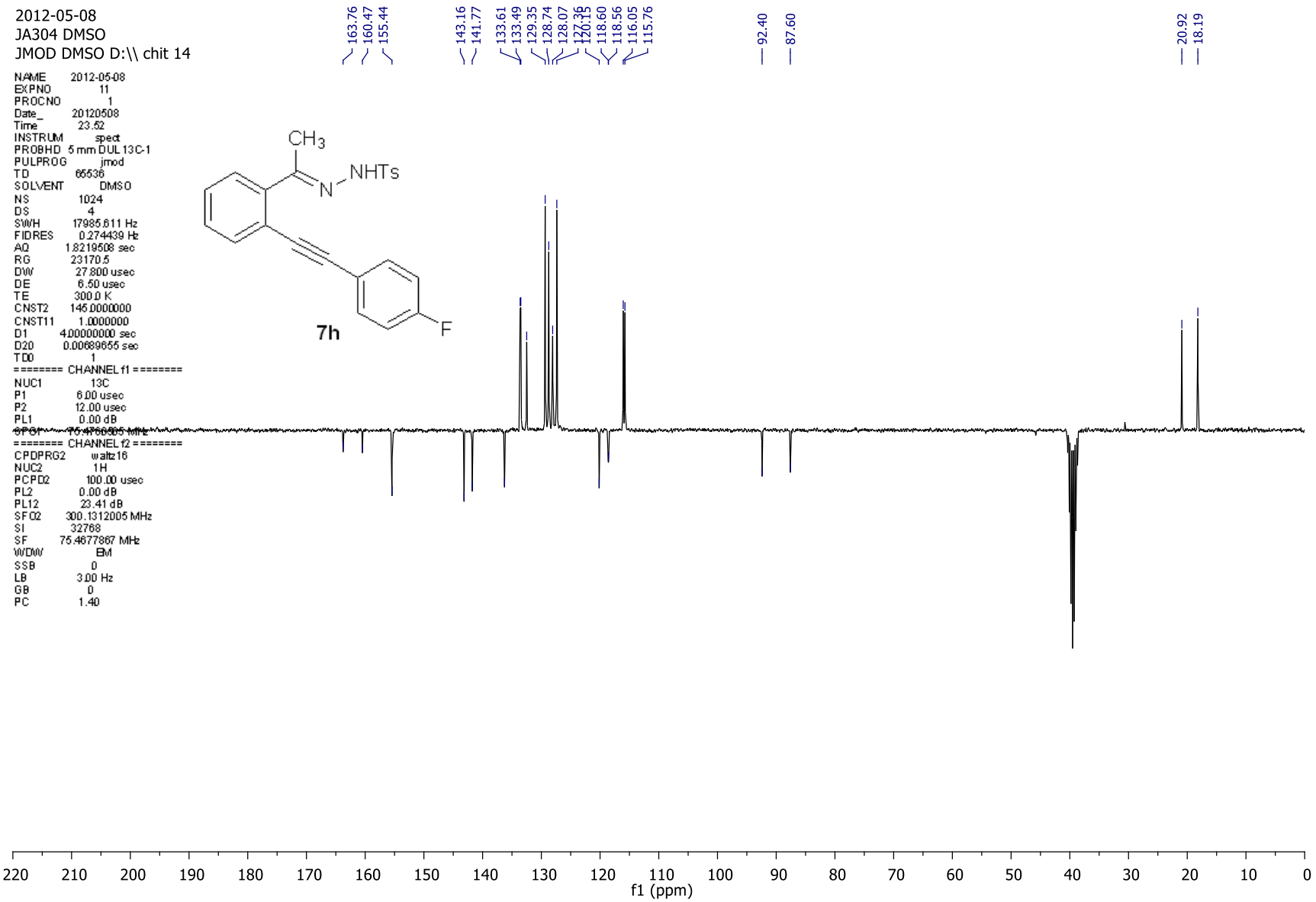
2.30 2.28

| | |
|---|---|
| NAME | 2012-05-08 |
| EXPNO | 10 |
| PROCNO | 1 |
| Date_ | 20120508 |
| Time | 22.10 |
| INSTRUM | spect |
| PROBHD | 5 mm DUL 13C-1 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | DMSO |
| NS | 4 |
| DS | 2 |
| SWH | 6172.839 Hz |
| FIDRES | 0.094190 Hz |
| AQ | 5.3084660 sec |
| RG | 256 |
| DW | 81.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 6.75 usec |
| PL1 | 0.00 dB |
| SFO1 | 300.1318534 MHz |
| SI | 32768 |
| SF | 300.1300710 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

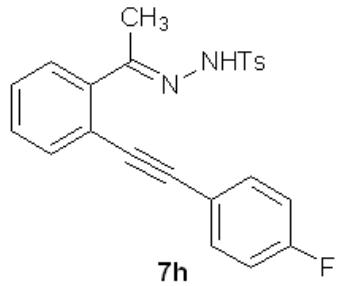



**7h**




2012-05-08
JA304 DMSO
JMOD DMSO D:\\ chit 14
163.76
160.47
155.44
143.16
141.77
133.61
133.49
129.35
128.74
128.07
118.60
118.56
116.05
115.76
92.40
87.60
20.92
18.19
NAME 2012-05-08
EXPNO 11
PROCNO 1
Date_ 20120508
Time 23.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 23170.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
NHTs
N
F
7h
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2012.03.12
JA304 DMSO
F19CPD DMSO v biocis 21

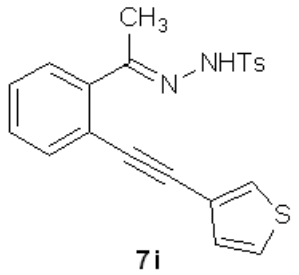



**7h**

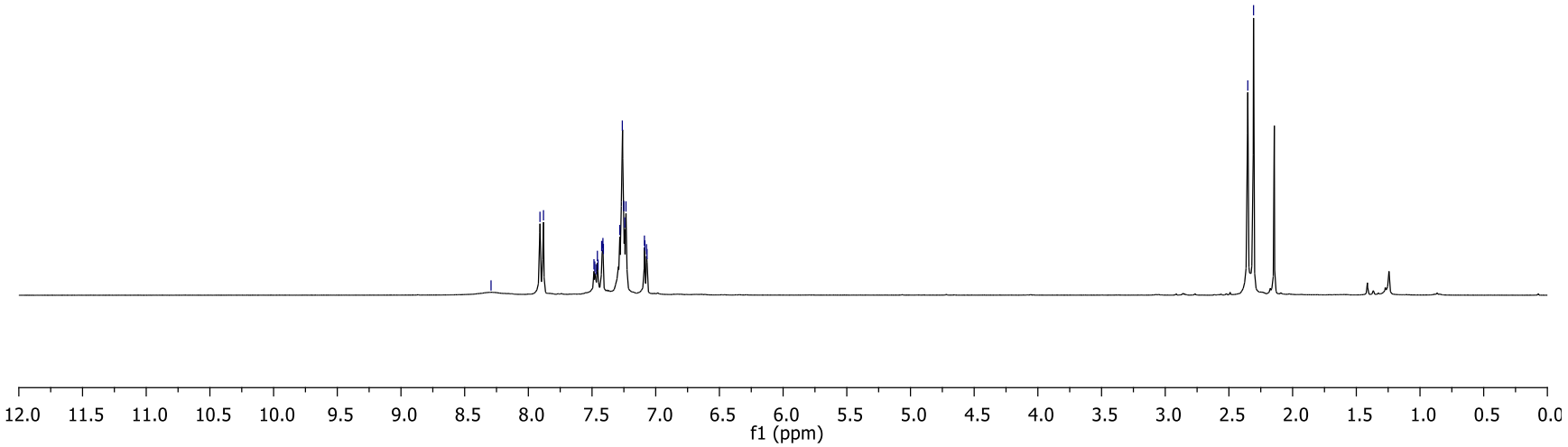
2014-11-07
JA1589P
Proton.4 CDCl3 D:\\ chit 59

8.29 | 7.91 | 7.88 | 7.48 | 7.48 | 7.47 | 7.46 | 7.45 | 7.42 | 7.42 | 7.41 | 7.41 | 7.28 | 7.26 | 7.25 | 7.24 | 7.23 | 7.09 | 7.08 | 7.07 | 7.07 | 2.35 | 2.31

```
NAME        2014-11-07
EXPNO             350
PROCNO              1
Date_        20141107
Time             9.02
INSTRUM          spect
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   4
DS                   2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                 114
DW              81.000 usec
DE               6.50 usec
TE              300.0 K
D1         1.00000000 sec
TD0                  1
======== CHANNEL f1 ========
NUC1               1H
P1               6.75 usec
PL1              0.00 dB
SFO1        300.1318534 MHz
SI               32768
SF          300.1300000 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC                1.00
```



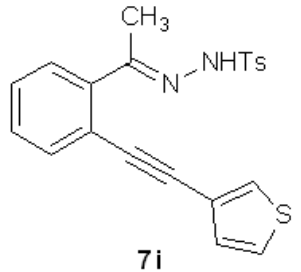

**7i**



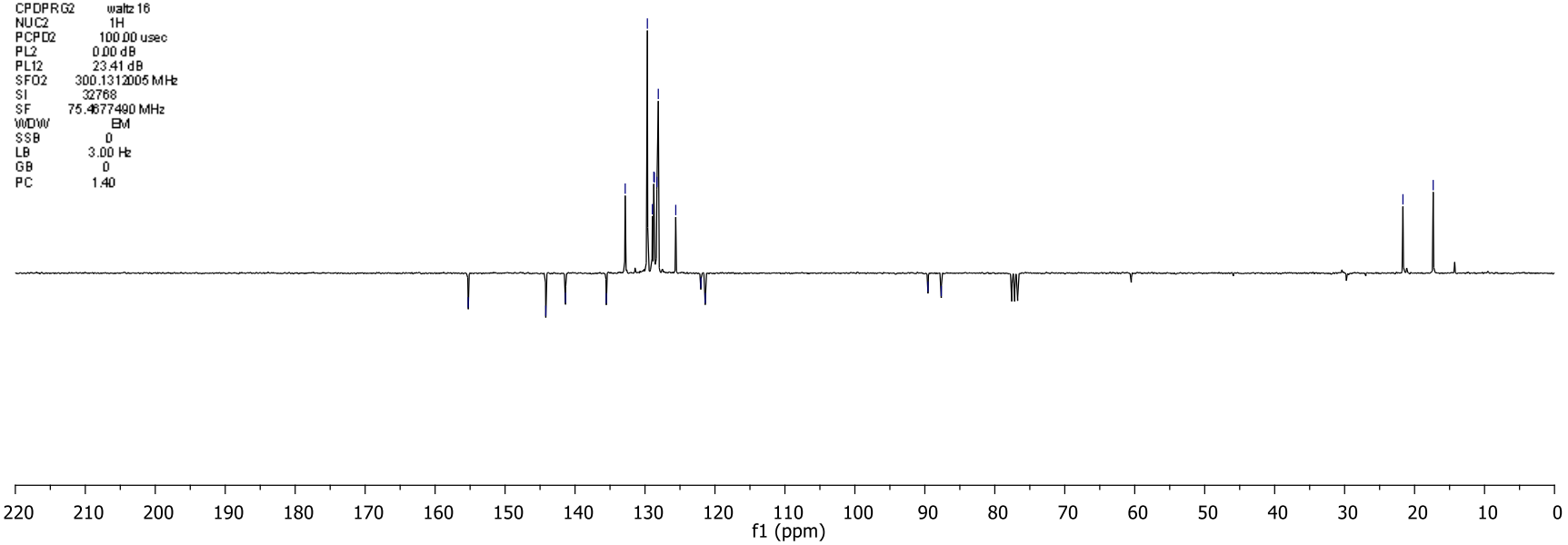
2014-11-06
JA1590P
JMOD CDCl3 D:\\ chit 58

155.26
144.18
141.36
135.53
132.83
129.66
128.94
128.74
128.66
128.30
128.10
125.60
122.02
121.39
89.55
87.65
21.66
17.34

NAME 2012-11-06
EXPNO 290
PROCNO 1
Date_ 20141106
Time 9.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

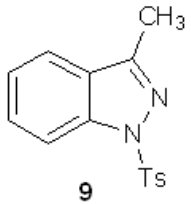



**7i**

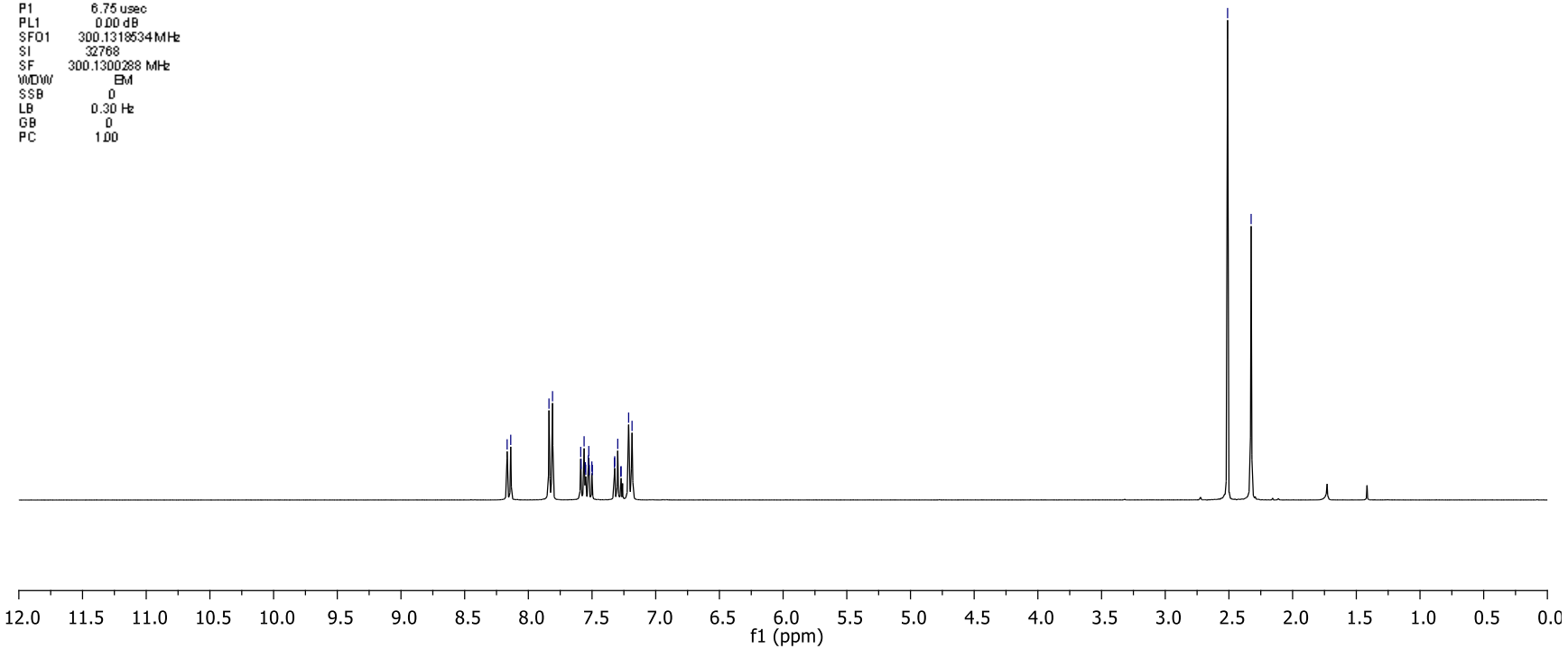


2014-09-05
JA237 RECRIST
Proton.4 CDCl3 D:\\ chit 24


| | |
|---|---|
| NAME | 2014-09-05 |
| EXPNO | 180 |
| PROCNO | 1 |
| Date_ | 20140906 |
| Time | 4.56 |
| INSTRUM | spect |
| PROBHD | 5 mm DUL 13C-1 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 4 |
| DS | 2 |
| SWH | 6172.839 Hz |
| FIDRES | 0.094190 Hz |
| AQ | 5.3084660 sec |
| RG | 256 |
| DW | 81.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 6.75 usec |
| PL1 | 0.00 dB |
| SFO1 | 300.1318534 MHz |
| SI | 32768 |
| SF | 300.1300288 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |



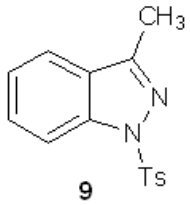

**9**



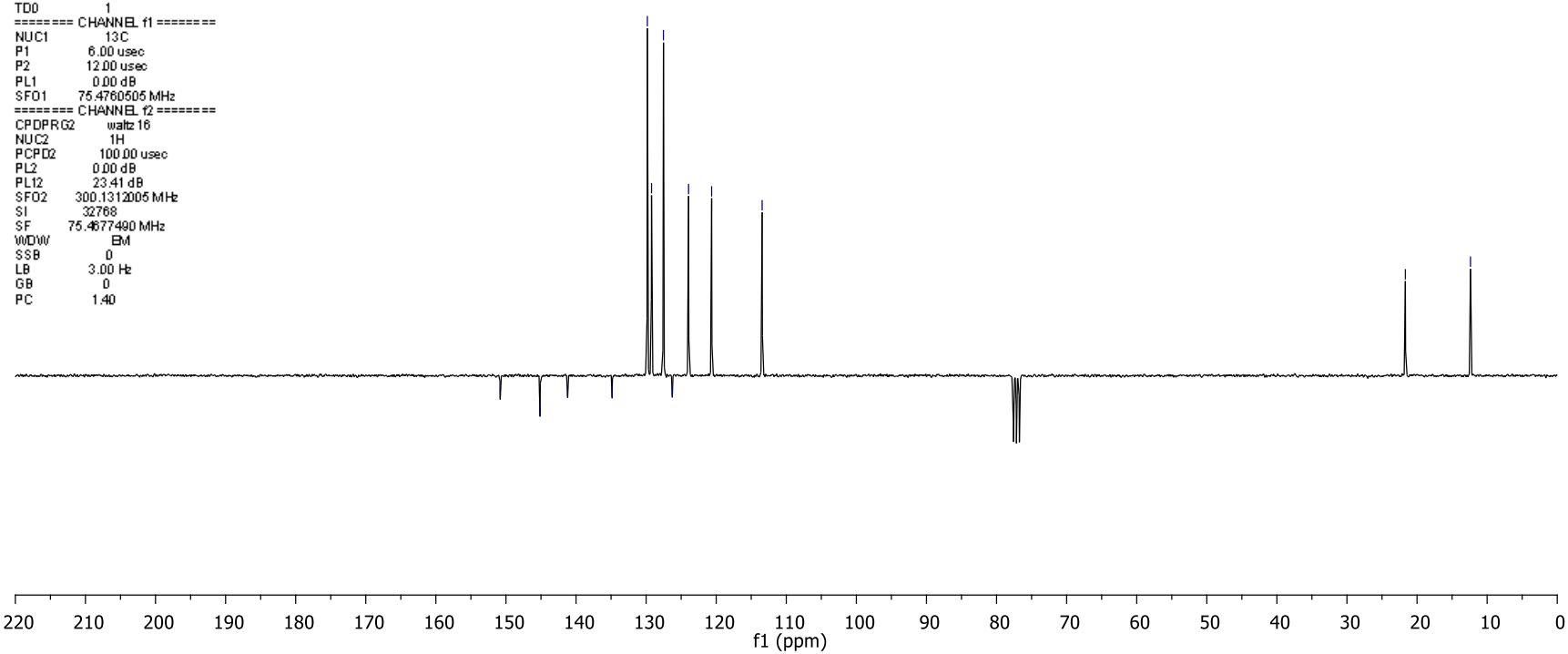
2014-09-05
JA237 RECRIST
JMOD CDCl3 D:\\ chit 24

150.82 145.14 141.19 134.85 129.82 129.23 127.53 126.26 123.94 120.65 113.44 21.67 12.37

NAME 2014-09-05
EXPNO 181
PROCNO 1
Date_ 20140906
Time 13.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



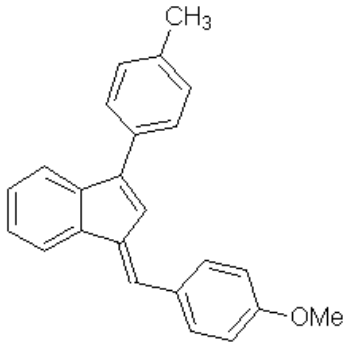

9



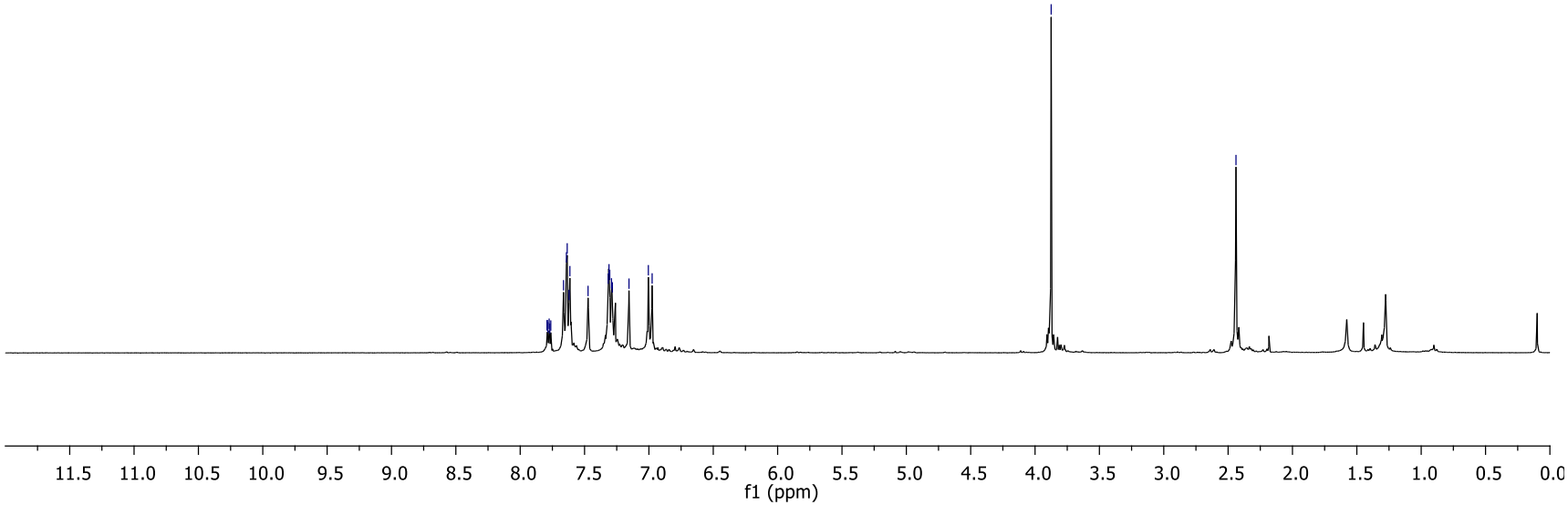
2014-10-17
JA1581 Prepa
Proton.4 CDCl3 D:\\ chit 40

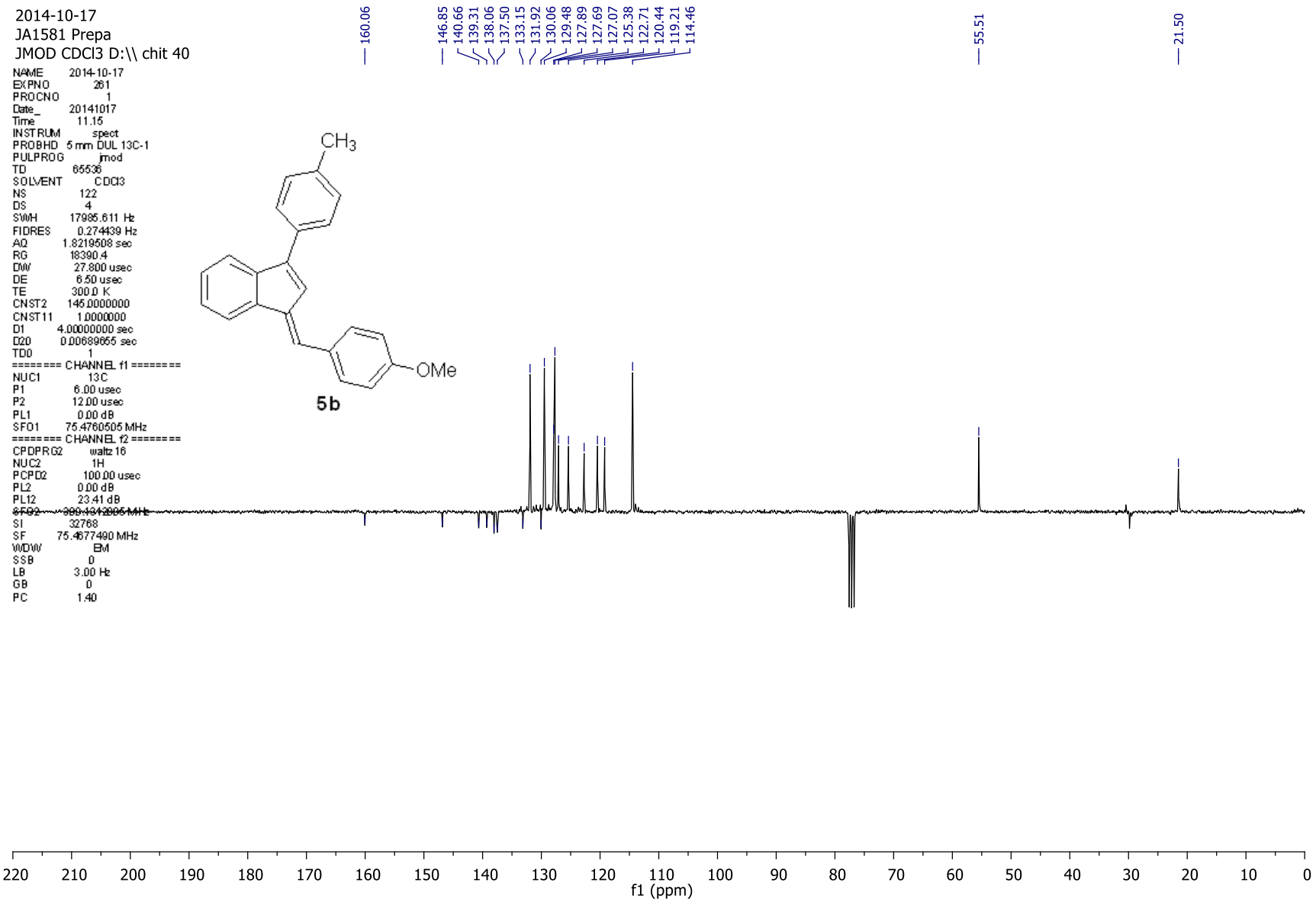
7.79 7.79 7.78 7.78 7.77 7.76 7.66 7.64 7.63 7.62 7.61 7.47 7.32 7.31 7.30 7.29 7.28 7.15 7.00 6.97 — 3.87 — 2.44

| | |
|---|---|
| NAME | 2014-10-17 |
| EXPNO | 260 |
| PROCNO | 1 |
| Date_ | 20141017 |
| Time | 11.04 |
| INSTRUM | spect |
| PROBHD | 5 mm DUL 13C-1 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 4 |
| DS | 2 |
| SWH | 6172.839 Hz |
| FIDRES | 0.094190 Hz |
| AQ | 5.3084660 sec |
| RG | 362 |
| DW | 81.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 6.75 usec |
| PL1 | 0.00 dB |
| SFO1 | 300.1318534 MHz |
| SI | 32768 |
| SF | 300.1300114 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




**5b**



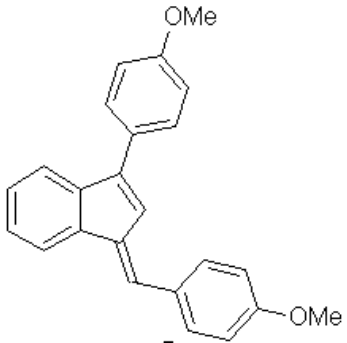

2014-10-17
JA1581 Prepa
JMOD CDCl3 D:\\ chit 40
160.06
146.85
140.66
139.31
138.06
137.50
133.15
131.92
130.06
129.48
127.89
127.69
127.07
125.38
122.71
120.44
119.21
114.46
55.51
21.50
NAME 2014-10-17
EXPNO 261
PROCNO 1
Date_ 20141017
Time 11.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 122
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 399.4342005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
CH3
OMe
5b
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2014-10-23
JA1583Prepa
Proton.4 CDCl3 D:\\ chit 56

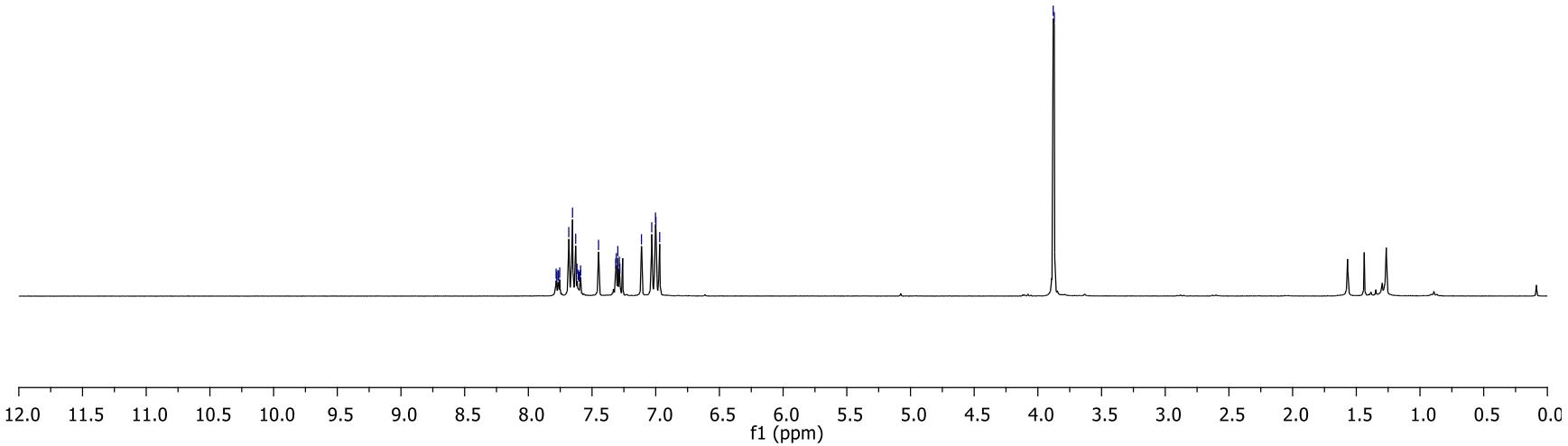
7.78 7.78 7.77 7.76 7.75 7.68 7.65 7.63 7.62 7.61 7.60 7.59 7.59 7.45 7.31 7.31 7.30 7.29 7.28 7.11 7.03 7.00 7.00 6.97

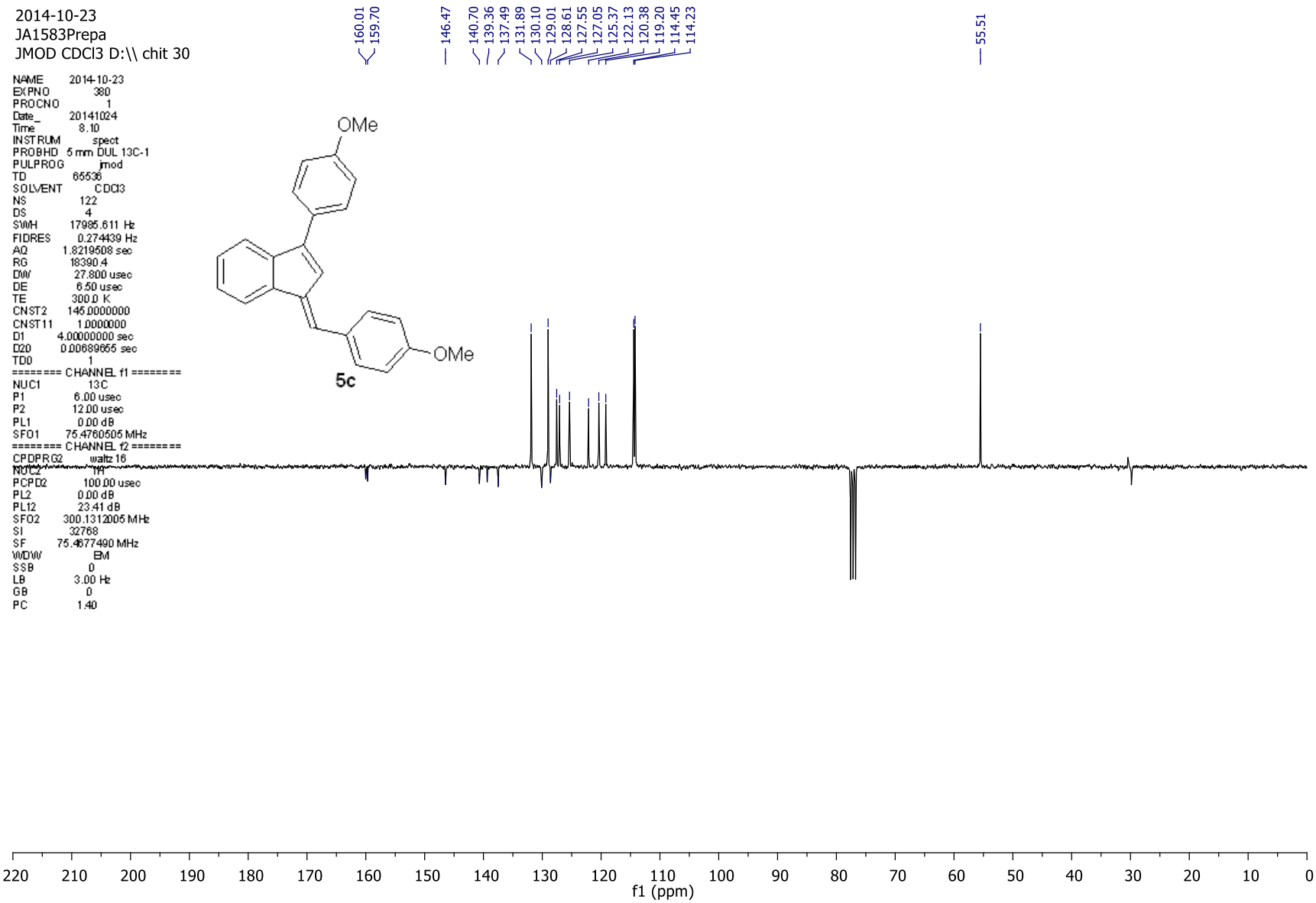
3.88 3.87

| | |
|---|---|
| NAME | 2014-10-23 |
| EXPNO | 110 |
| PROCNO | 1 |
| Date_ | 20141023 |
| Time | 12.35 |
| INSTRUM | spect |
| PROBHD | 5 mm DUL 13C-1 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 4 |
| DS | 2 |
| SWH | 6172.839 Hz |
| FIDRES | 0.094190 Hz |
| AQ | 5.3084660 sec |
| RG | 512 |
| DW | 81.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 6.75 usec |
| PL1 | 0.00 dB |
| SFO1 | 300.1318534 MHz |
| SI | 32768 |
| SF | 300.1300549 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




**5c**



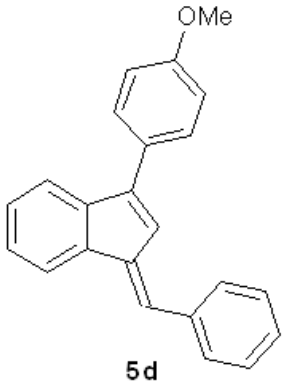

2014-10-23
JA1583Prepa
JMOD CDCl3 D:\\ chit 30
160.01
159.70
146.47
140.70
139.36
137.49
131.89
130.10
129.01
128.61
127.55
127.05
125.37
122.13
120.38
119.20
114.45
114.23
55.51
NAME 2014-10-23
EXPNO 380
PROCNO 1
Date_ 20141024
Time 8.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 122
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OMe
OMe
5c
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2014-11-20
JA1587
Proton.4 CDCl3 D:\\ chit 16

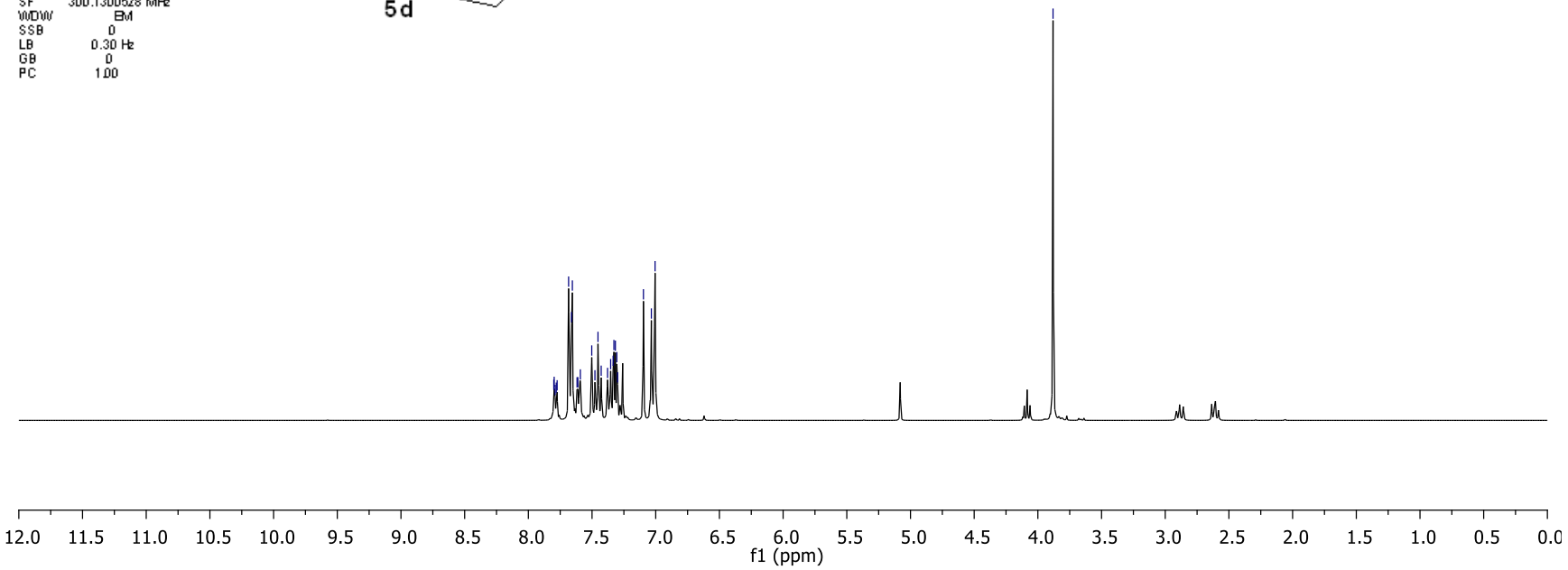
7.80 7.80 7.79 7.78 7.77 7.68 7.65 7.62 7.61 7.59 7.50 7.48 7.45 7.43 7.38 7.35 7.33 7.33 7.32 7.31 7.30 7.10 7.03 7.01

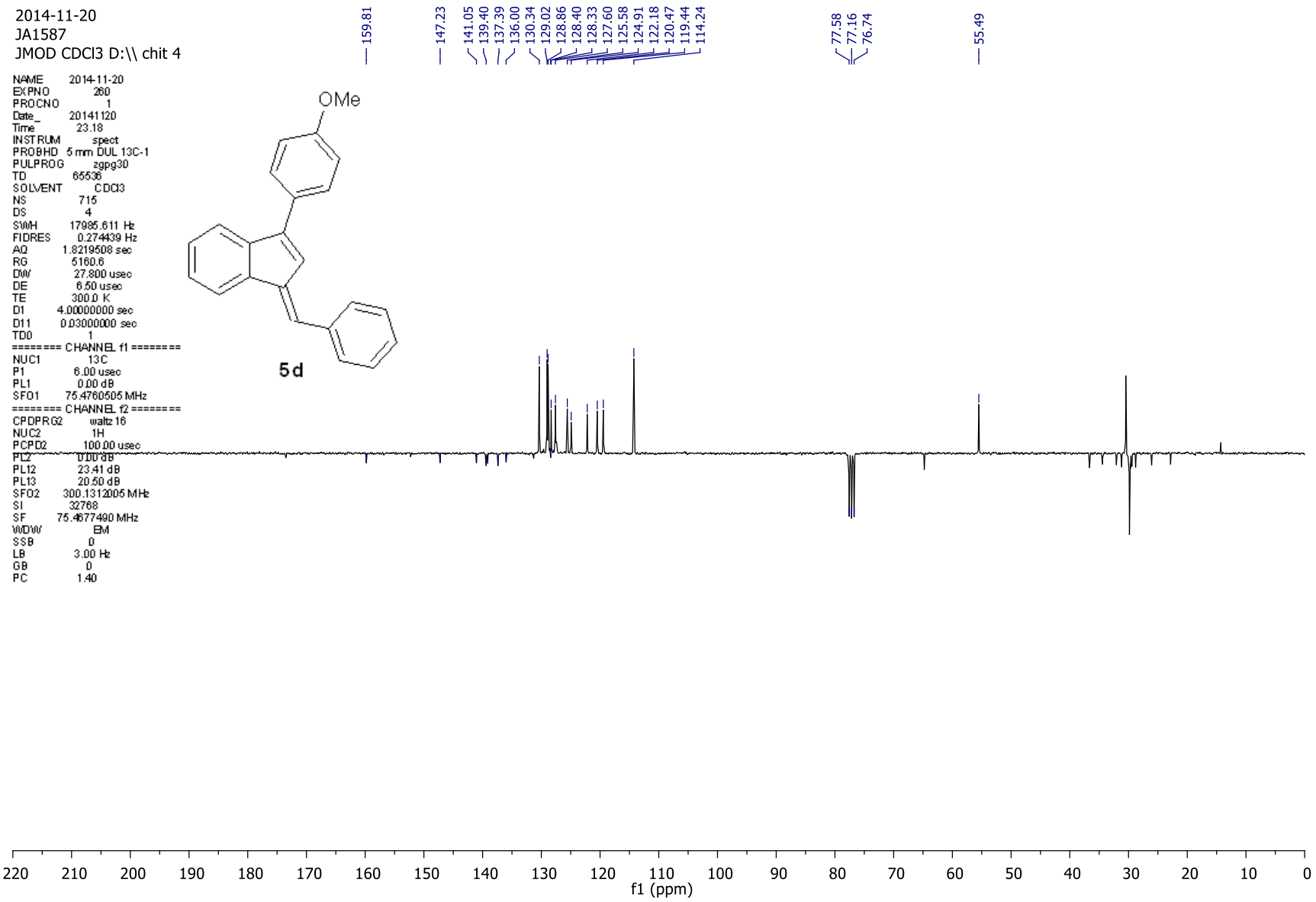
3.88

NAME 2014-11-20
EXPNO 70
PROCNO 1
Date_ 20141120
Time 11.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.75 usec
PL1 0.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300528 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



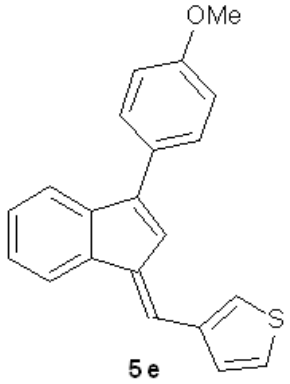

**5d**



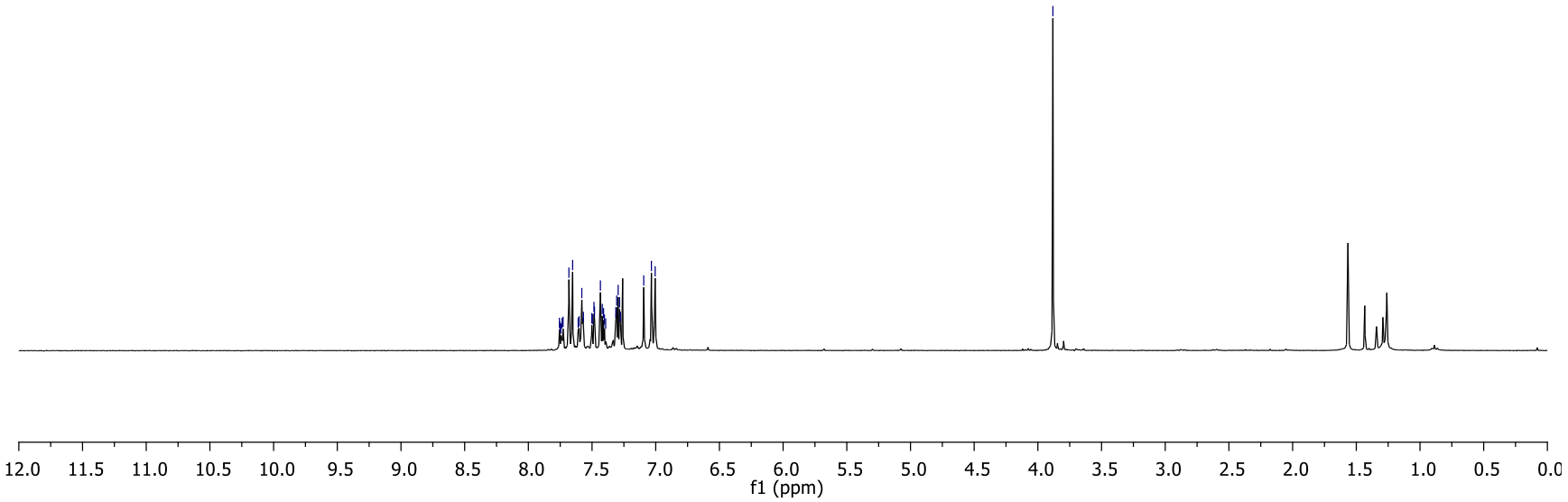

2014-11-20
JA1587
JMOD CDCl3 D:\\ chit 4
159.81
147.23
141.05
139.40
137.39
136.00
130.34
129.02
128.86
128.40
128.33
127.60
125.58
124.91
122.18
120.47
119.44
114.24
77.58
77.16
76.74
55.49
NAME 2014-11-20
EXPNO 260
PROCNO 1
Date_ 20141120
Time 23.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 715
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 5160.6
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 4.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
PL13 20.50 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OMe
5d
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2014-11-14
JA1590
Proton.4 CDCl3 D:\\ chit 43


```
NAME        2014-11-14
EXPNO          280
PROCNO           1
Date_       20141115
Time            2.33
INSTRUM         spect
PROBHD  5 mm DUL 13C-1
PULPROG         zg30
TD             65536
SOLVENT         CDCl3
NS                 4
DS                 2
SWH         6172.839 Hz
FIDRES      0.094190 Hz
AQ          5.3084660 sec
RG               256
DW            81.000 usec
DE             6.50 usec
TE            300.0 K
D1          1.00000000 sec
TD0                1
======== CHANNEL f1 ========
NUC1              1H
P1             6.75 usec
PL1            0.00 dB
SFO1       300.1318534 MHz
SI             32768
SF         300.1299704 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```



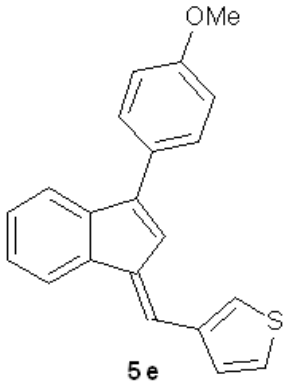

**5 e**



2014-11-14
JA1590
JMOD CDCl3 D:\\ chit 43

159.79 146.60 140.95 139.31 139.08 138.04 129.03 128.71 128.44 127.24 126.95 126.55 125.51 122.03 121.17 120.56 119.28 114.26 55.51

NAME 2014-11-14
EXPNO 281
PROCNO 1
Date_ 20141115
Time 2.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
P2 12.00 usec
PL1 0.00 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.41 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40




5 e