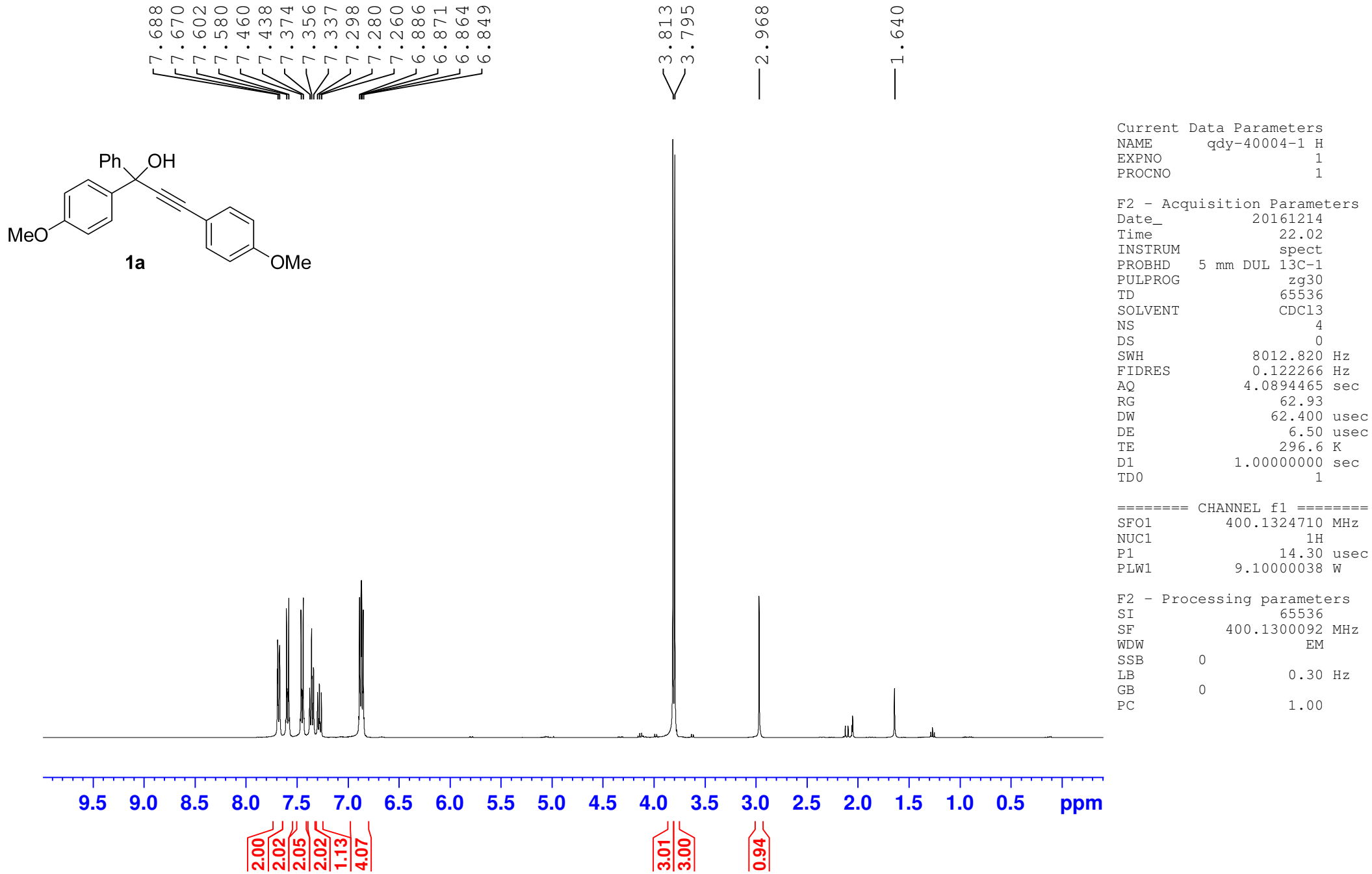
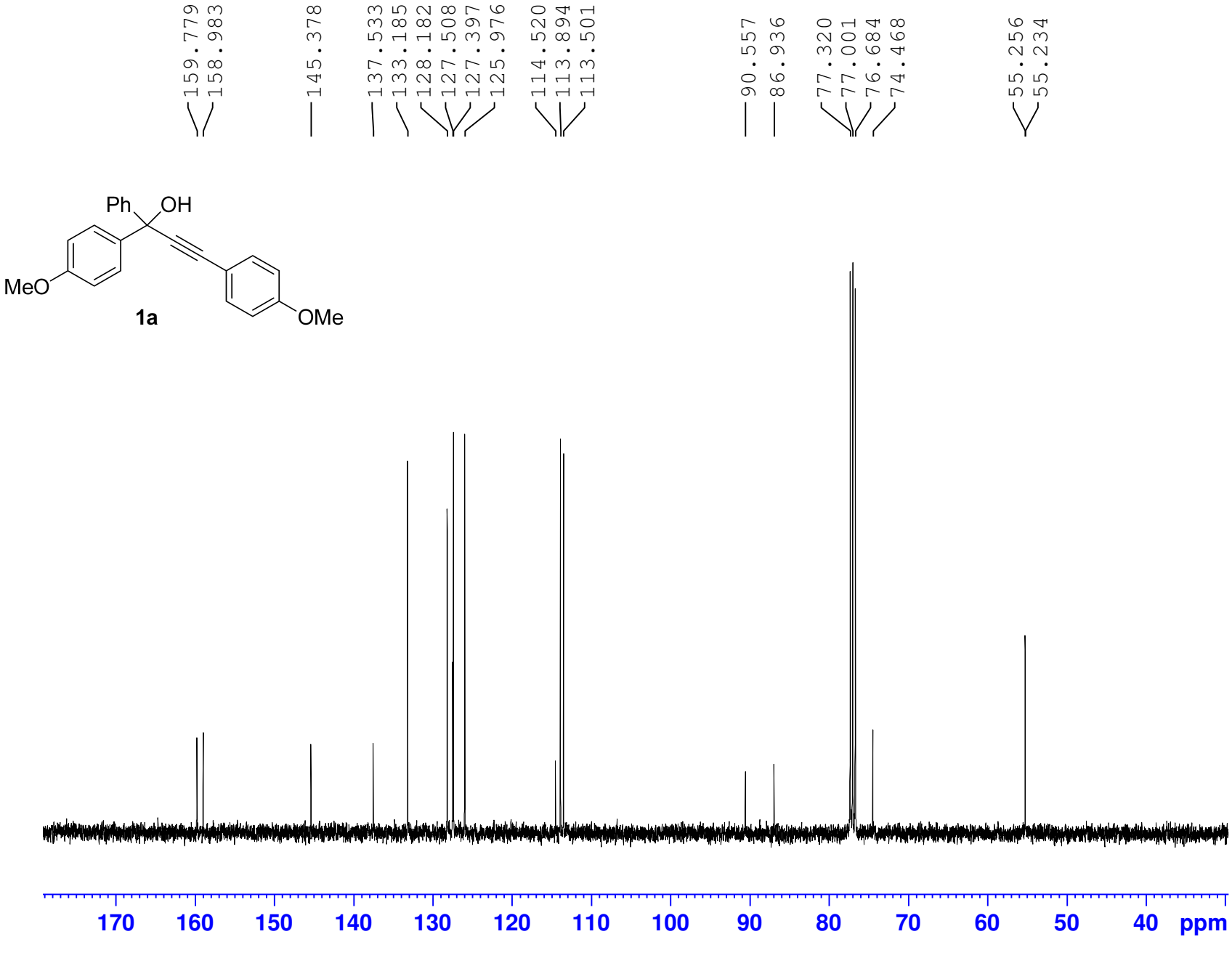
File Name: Supplementary Information
Description: Supplementary Figures, Supplementary Tables, Supplementary Notes, Supplementary Methods and Supplementary References



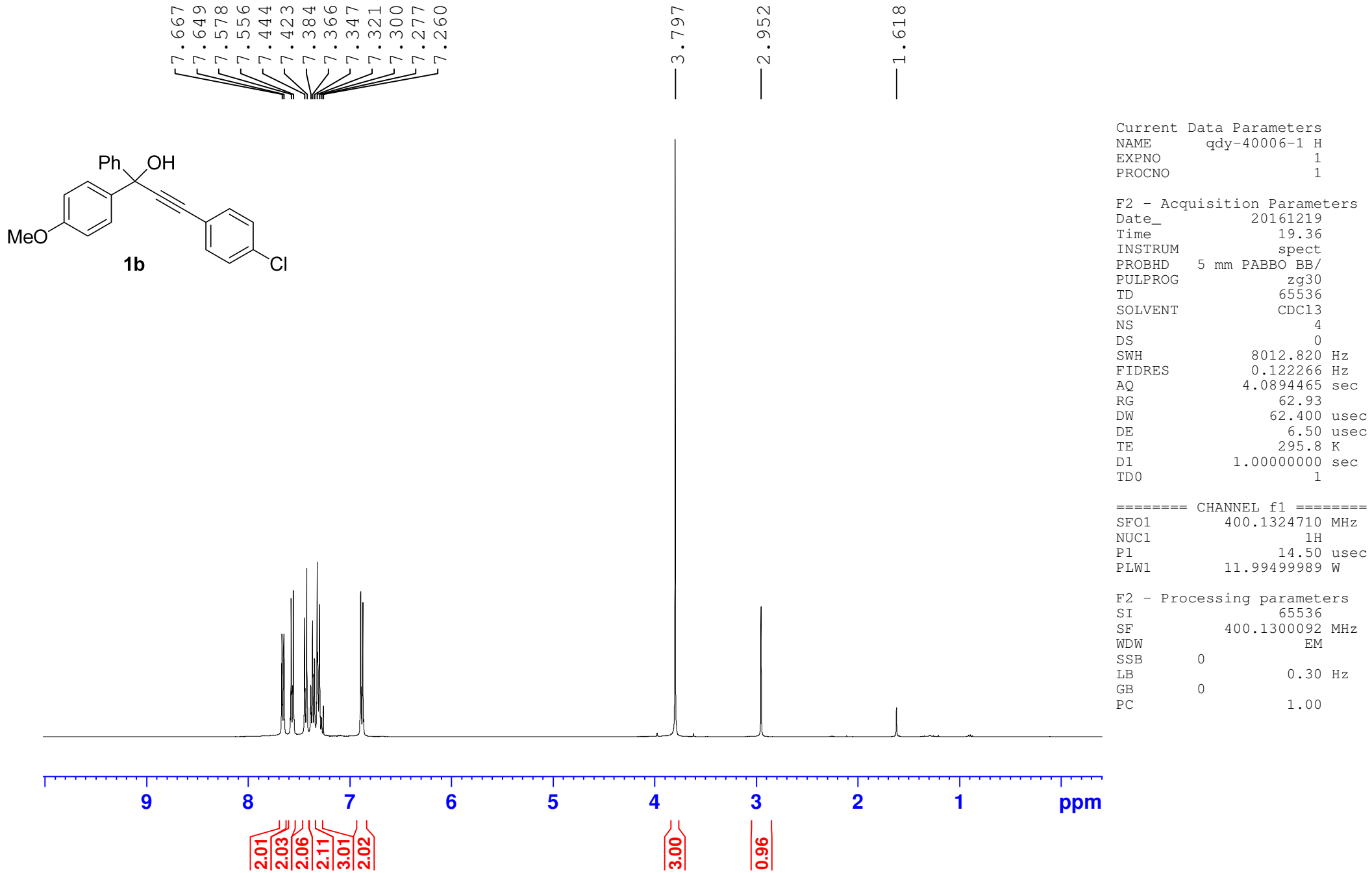

```
Current Data Parameters
NAME      qdy-40004-1 H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161214
Time              22.02
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                62.93
DW               62.400 usec
DE                6.50 usec
TE                296.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                14.30 usec
PLW1          9.10000038 W

F2 - Processing parameters
SI                65536
SF           400.1300092 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 1. ^{1}H NMR spectrum for 1a



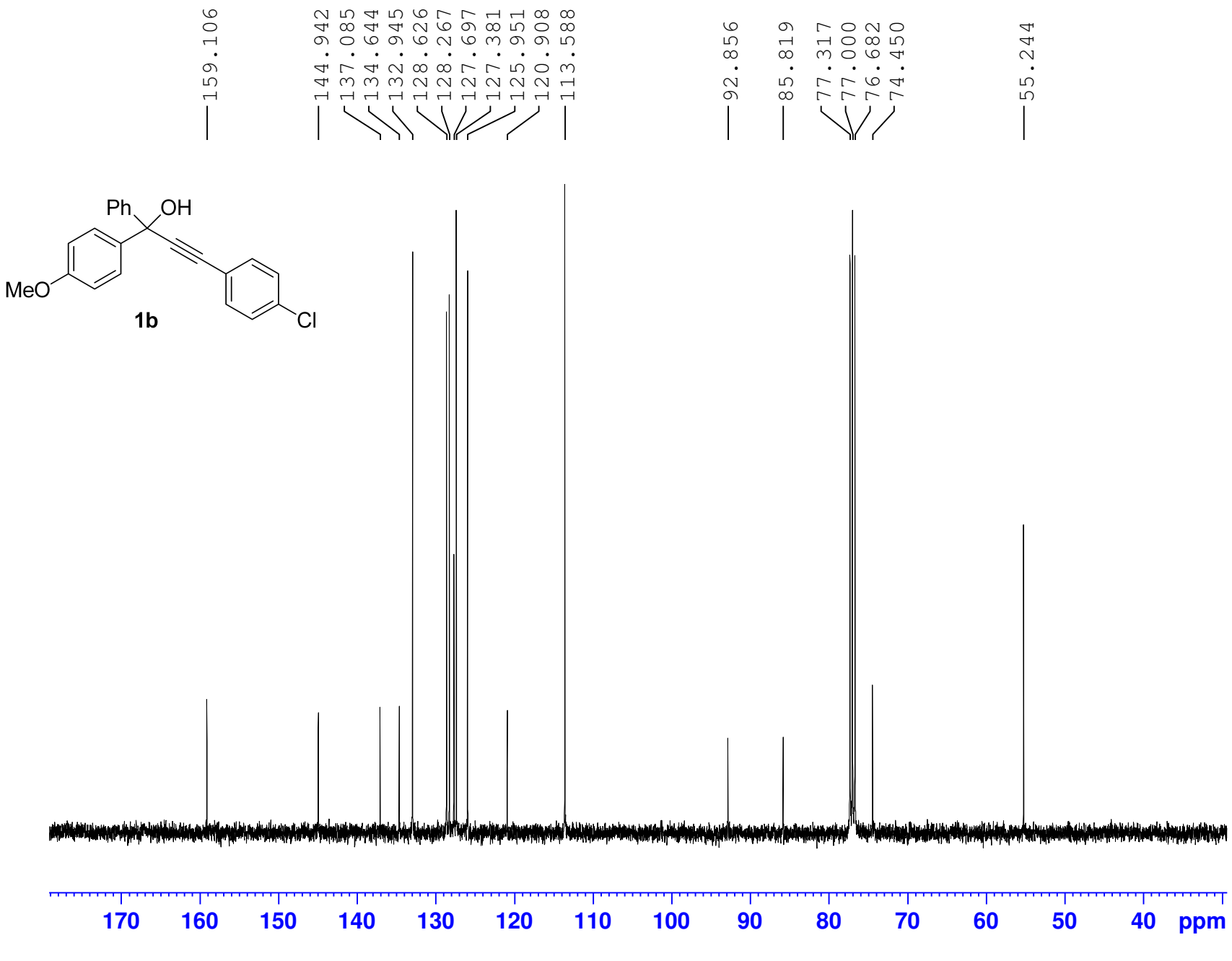

```
Current Data Parameters
NAME       qdy-40004-1 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161214
Time              22.05
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   46
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.60 usec
PLW1         31.98900032 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          9.10000038 W
PLW12         0.24608000 W
PLW13         0.19933000 W

F2 - Processing parameters
SI                 32768
SF           100.6127766 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 2. ^{13}C NMR spectrum for 1a



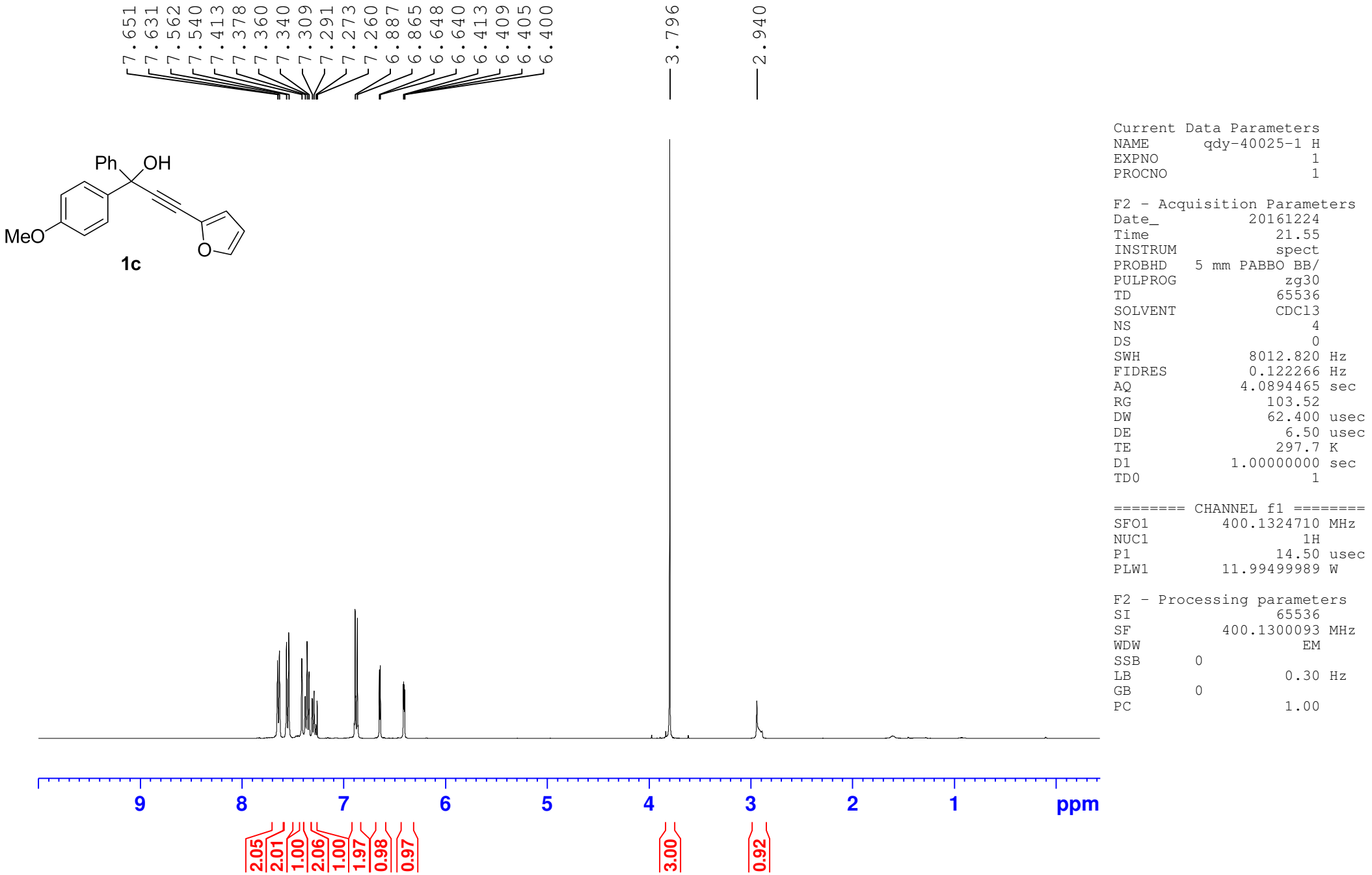

```
Current Data Parameters
NAME      qdy-40006-1 H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161219
Time              19.36
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                62.93
DW               62.400 usec
DE                6.50 usec
TE                295.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1300092 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 3. ^{1}H NMR spectrum for 1b



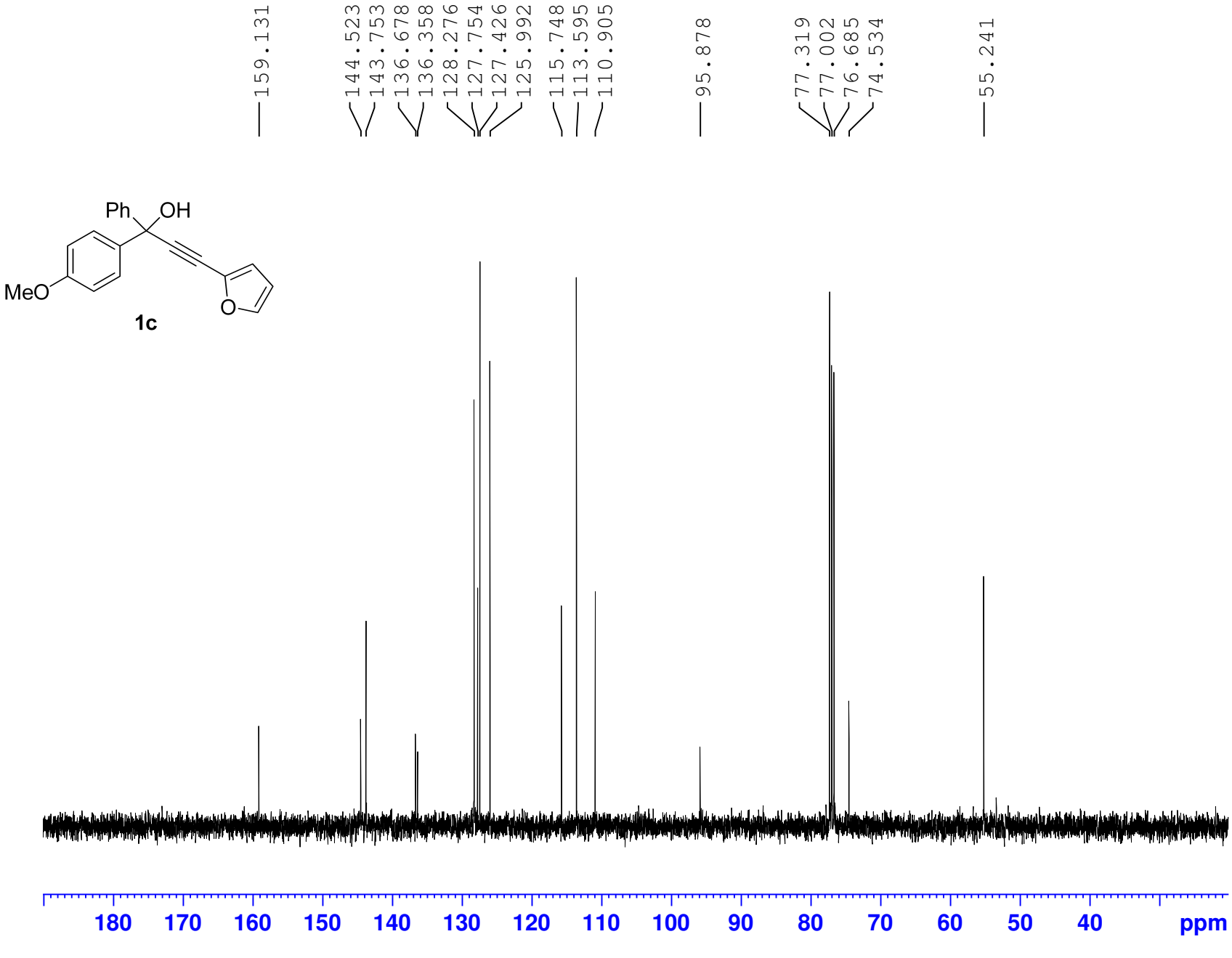

```
Current Data Parameters
NAME      qdy-40006-1  C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161219
Time               19.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   92
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127766 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 4. ^{13}C NMR spectrum for 1b



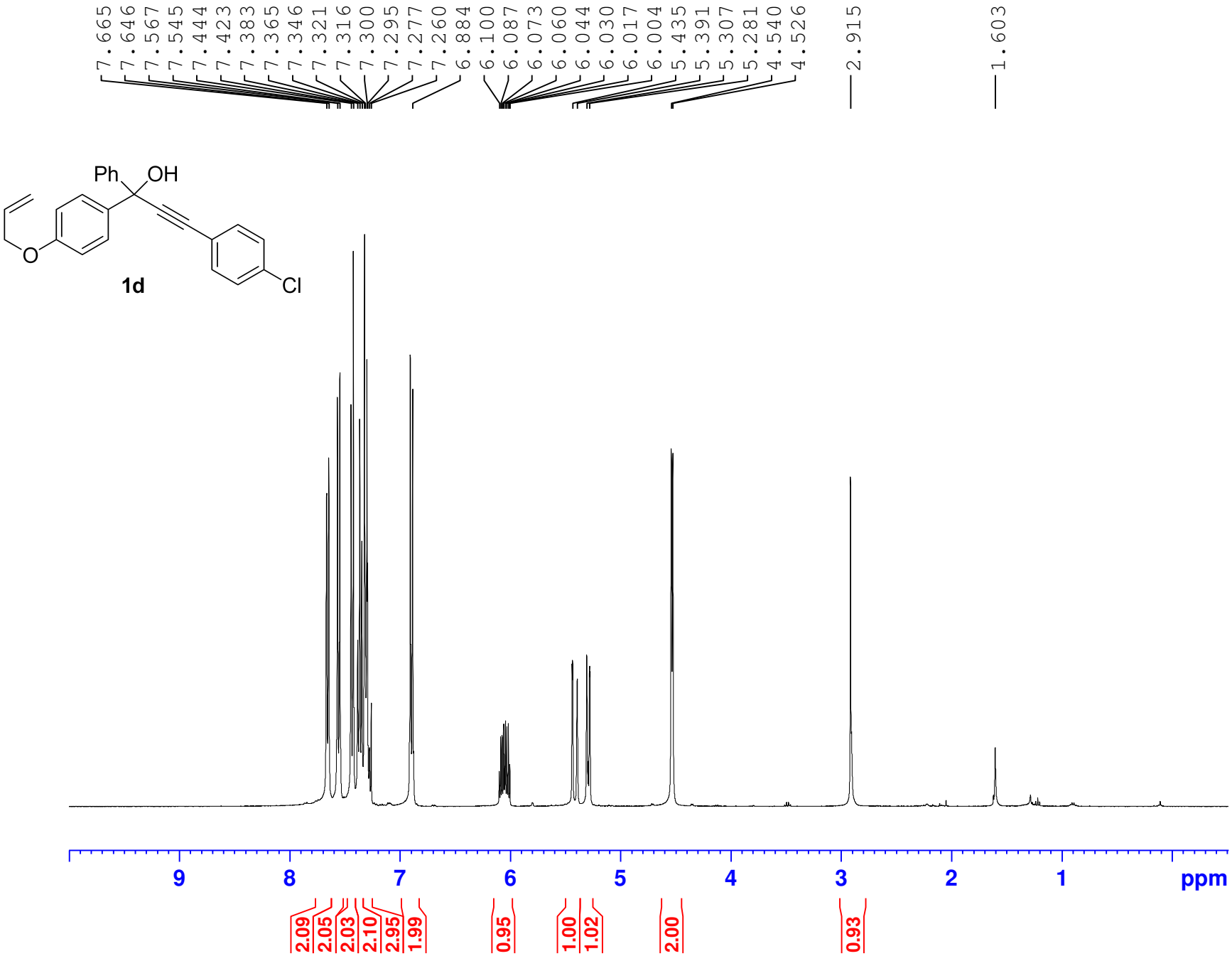

```
Current Data Parameters
NAME      qdy-40025-1 H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20161224
Time               21.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 103.52
DW                 62.400 usec
DE                  6.50 usec
TE                 297.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300093 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 5. ^{1}H NMR spectrum for 1c



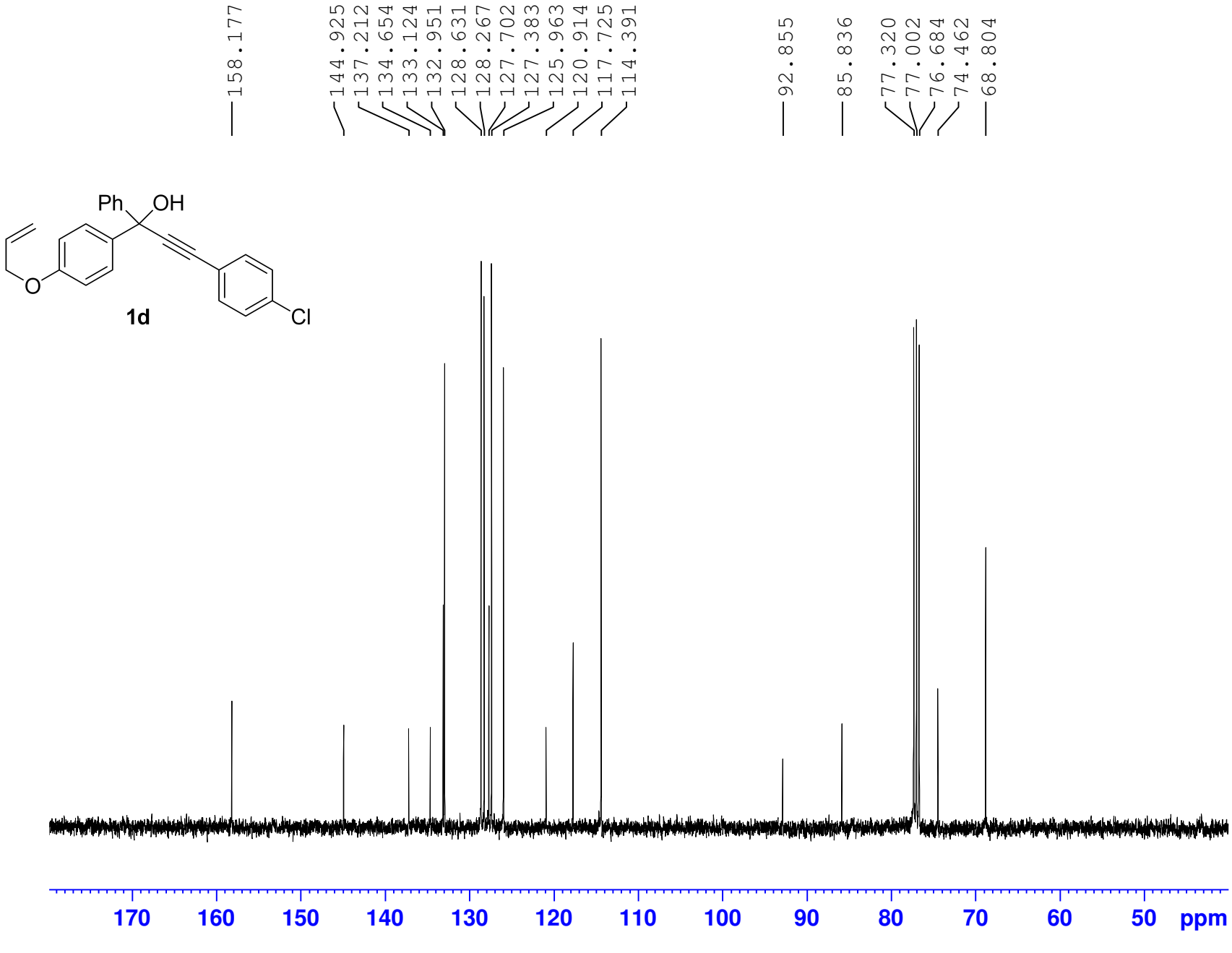

```
Current Data Parameters
NAME      qdy-40025-1 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161224
Time              16.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   29
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           100.6127773 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 6. ^{13}C NMR spectrum for 1c



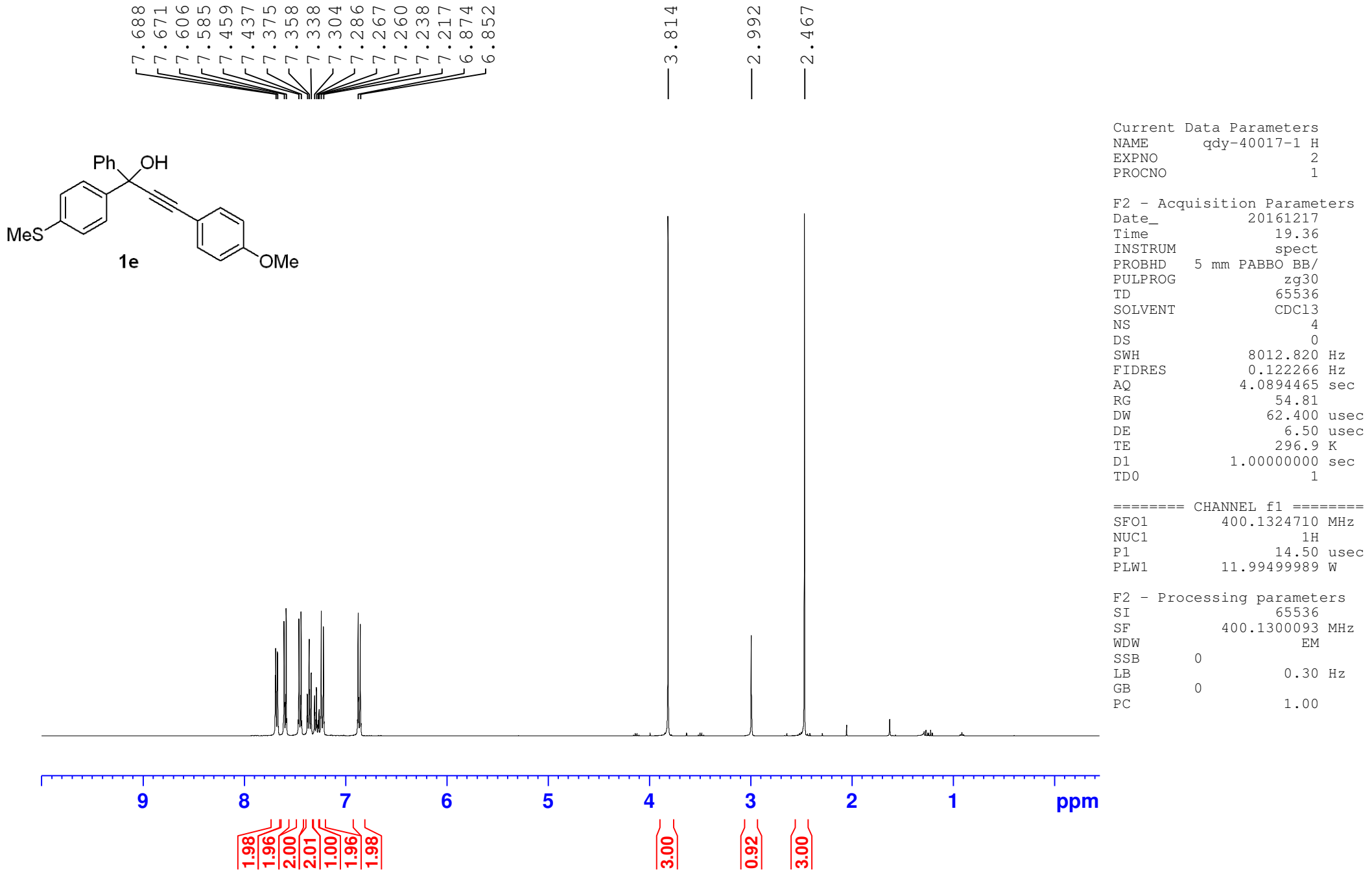

```
Current Data Parameters
NAME       qdy-40021-2 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161221
Time              16.54
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                54.81
DW               62.400 usec
DE                6.50 usec
TE                297.4 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300093 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 7. ^{1}H NMR spectrum for 1d

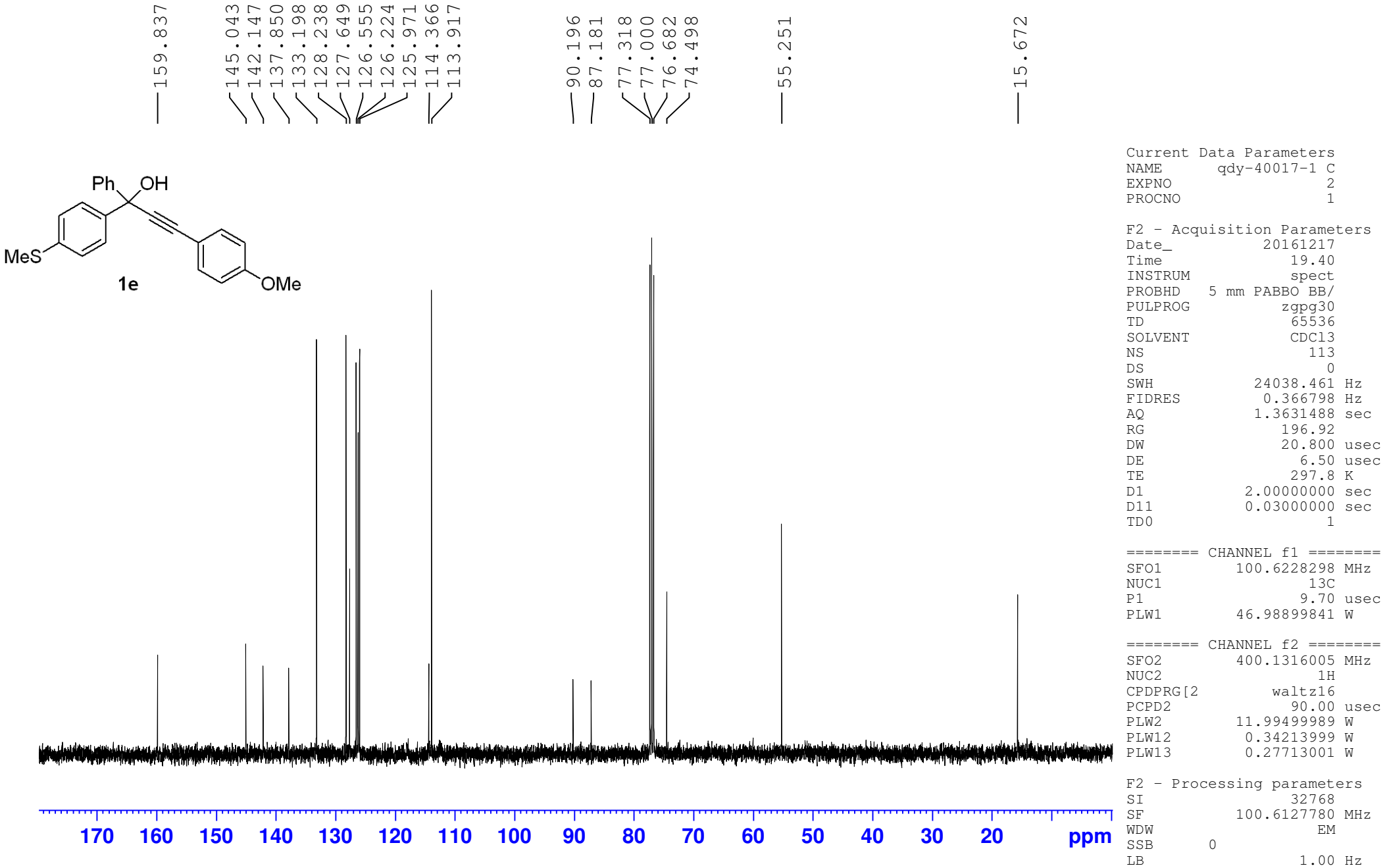



```
Current Data Parameters
NAME      qdy-40021-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161221
Time              16.56
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  71
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127759 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 8. ^{13}C NMR spectrum for 1d

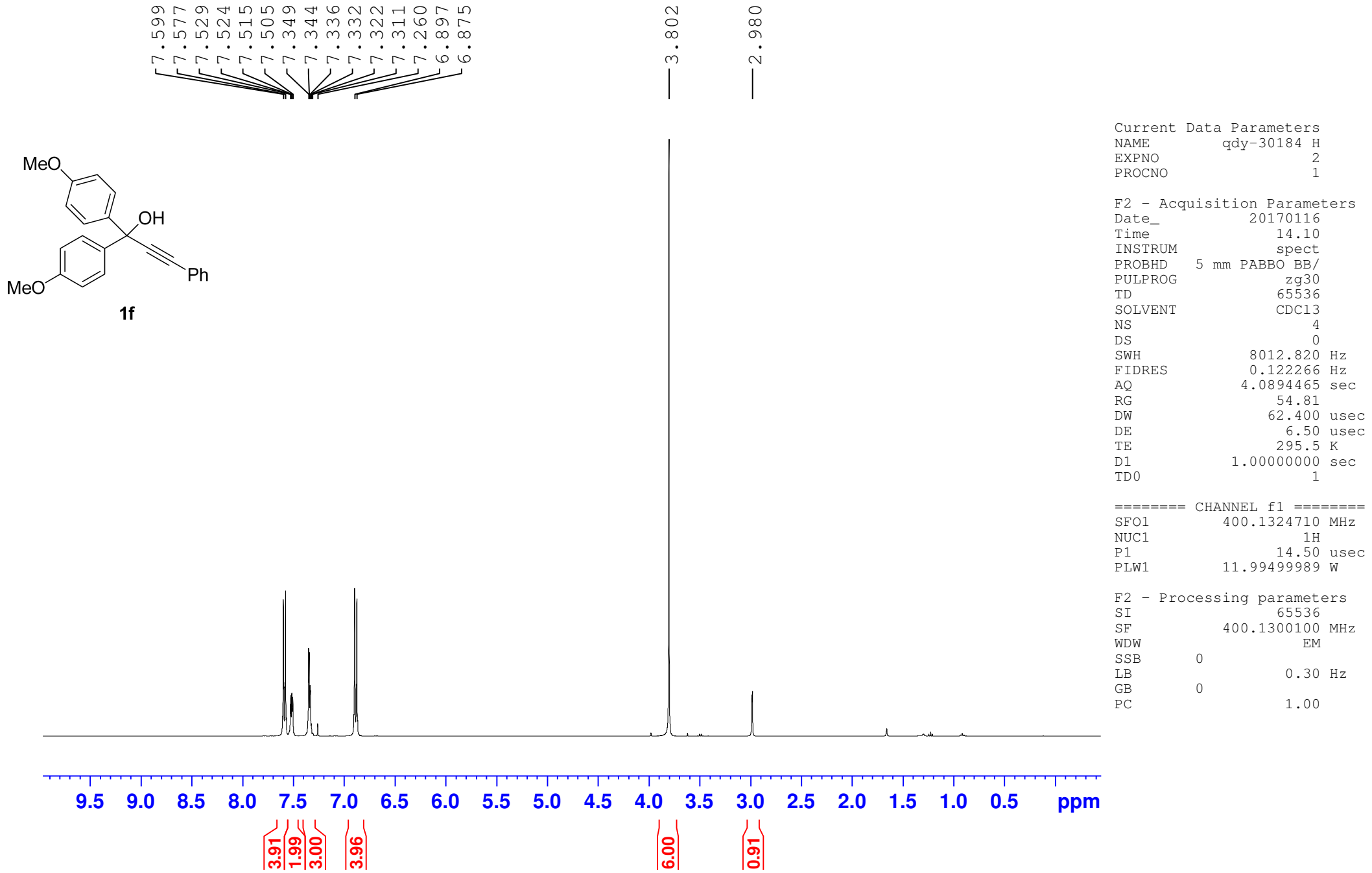


Supplementary Figure 9. ^{1}H NMR spectrum for 1e

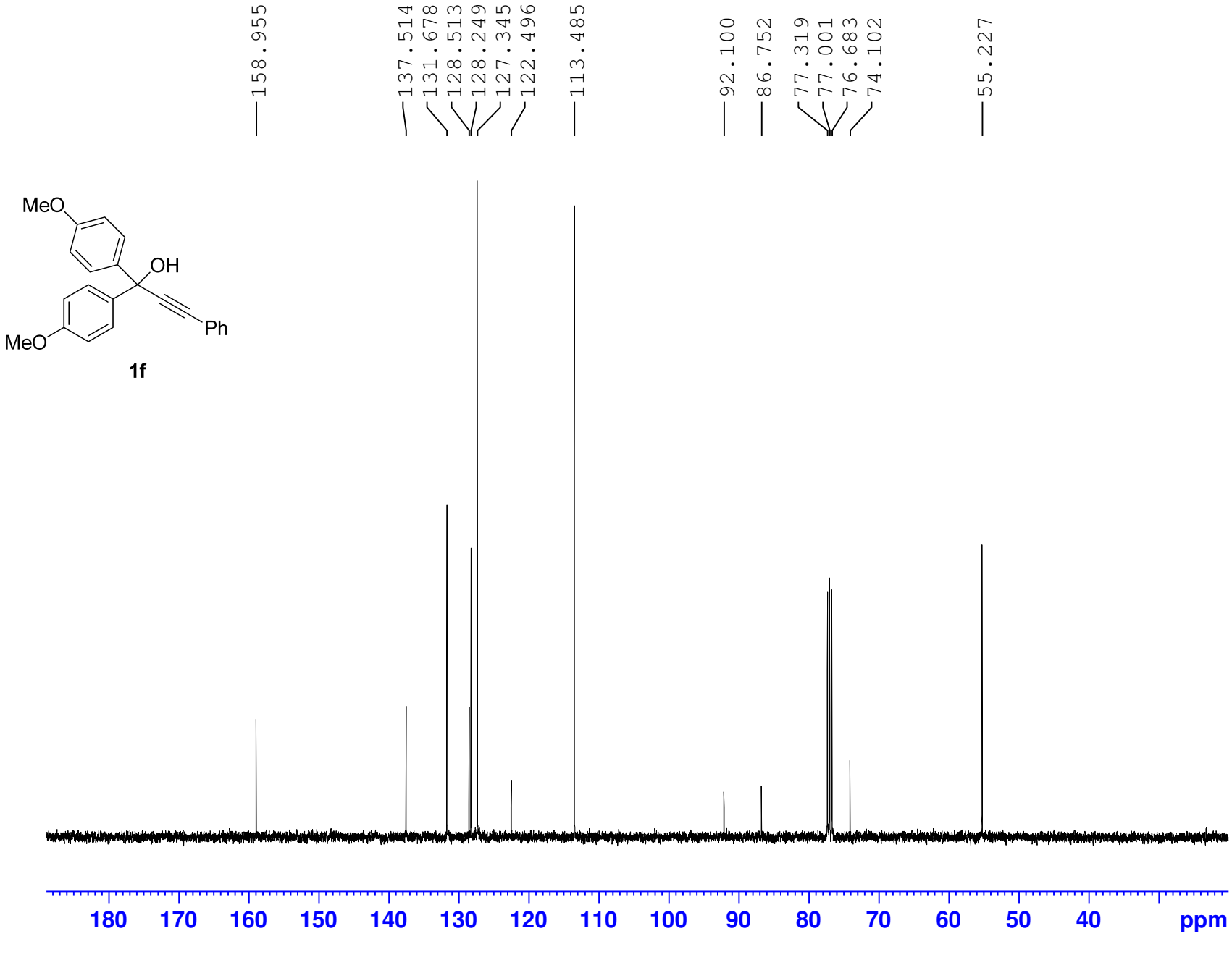



```
Current Data Parameters
NAME      qdy-40017-1 C
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20161217
Time              19.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 113
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                297.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127780 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

Supplementary Figure 10. ^{13}C NMR spectrum for 1e

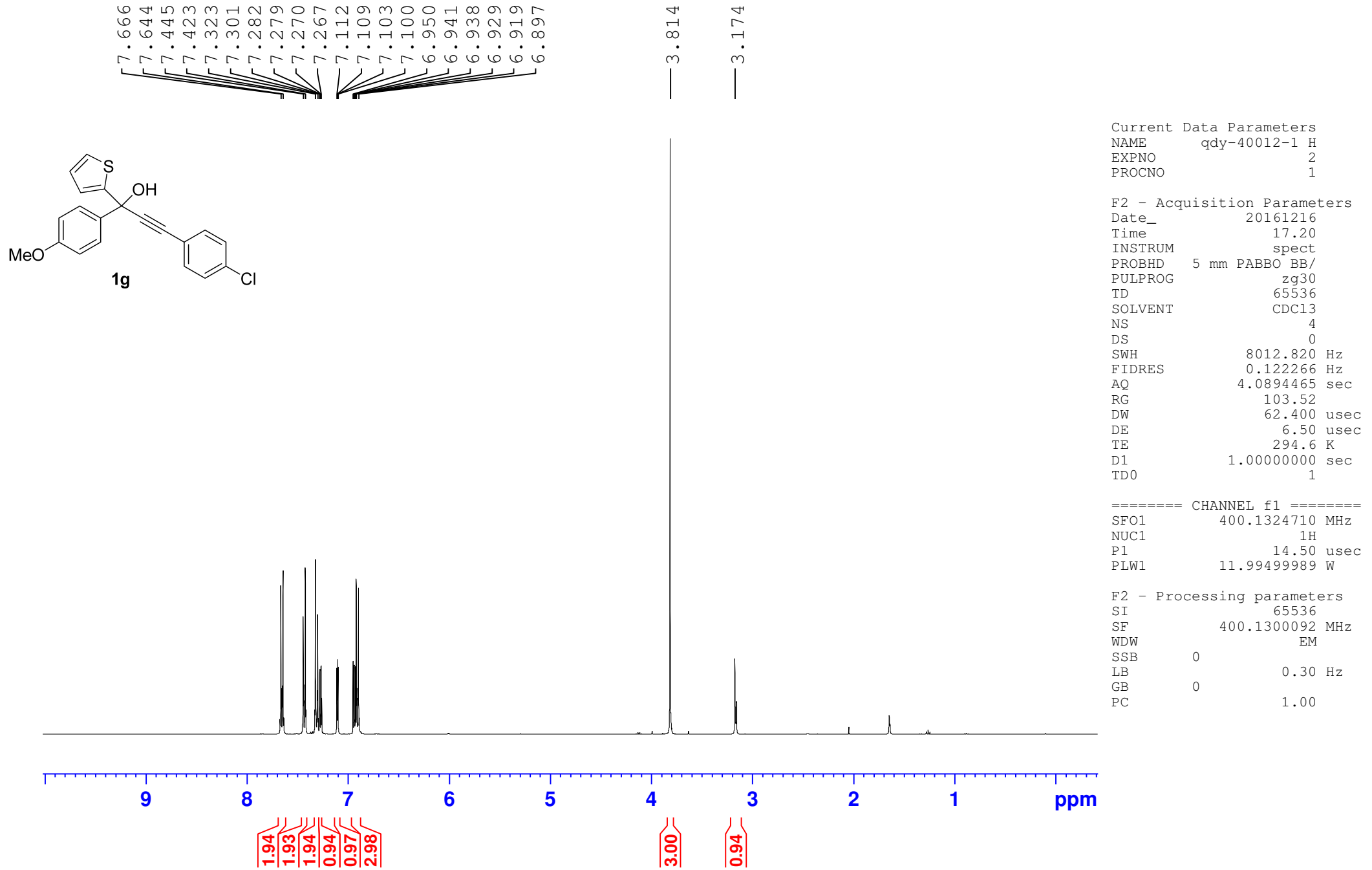


Supplementary Figure 11. ^{1}H NMR spectrum for 1f



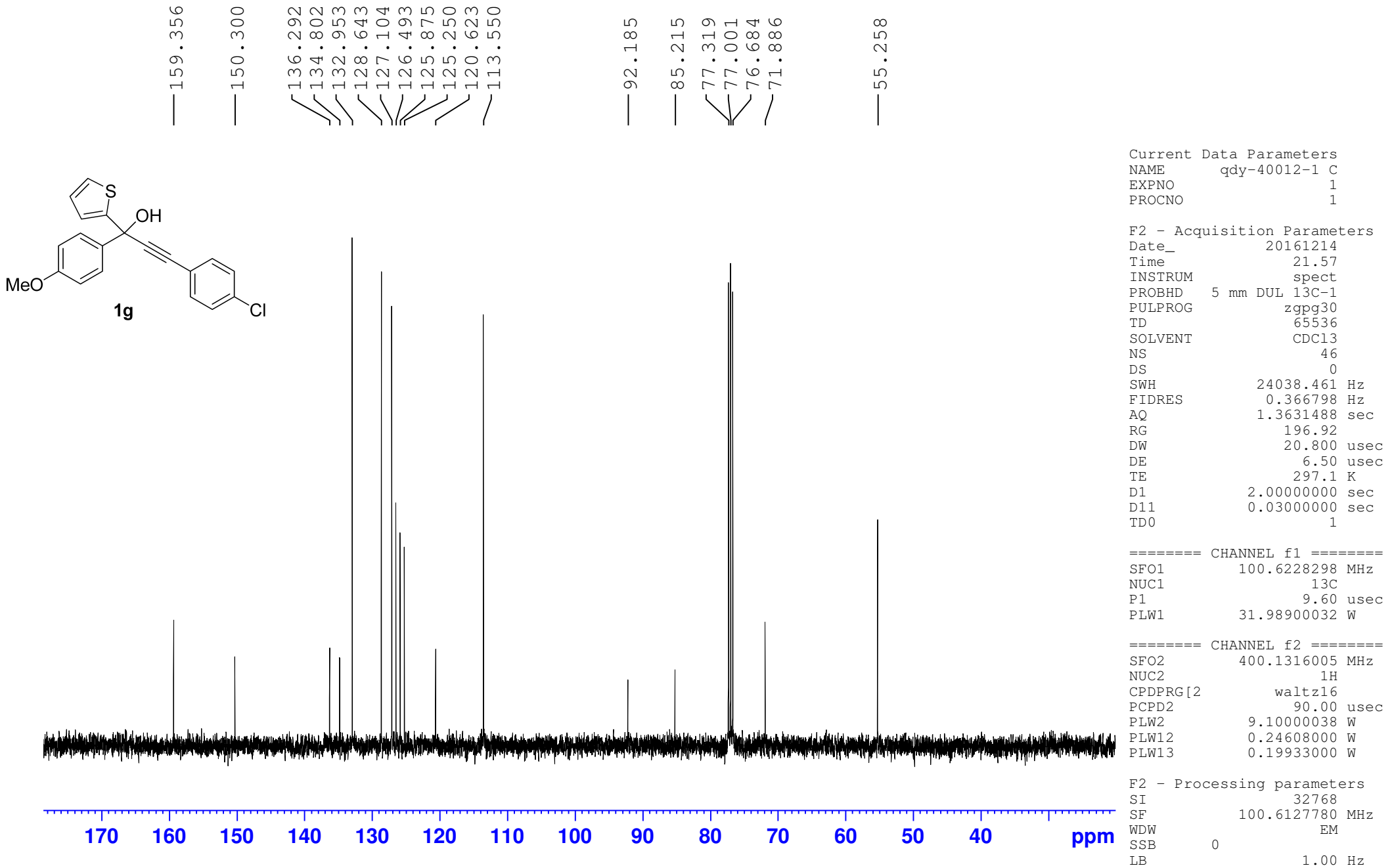

```
Current Data Parameters
NAME         qdy-30184 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170116
Time               14.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   60
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127788 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 12. ^{13}C NMR spectrum for 1f



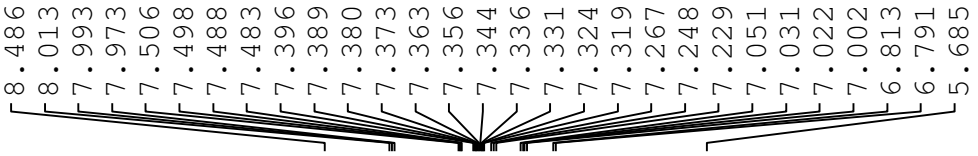

```
Current Data Parameters
NAME      qdy-40012-1 H
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20161216
Time              17.20
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                103.52
DW                62.400 usec
DE                 6.50 usec
TE                294.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300092 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

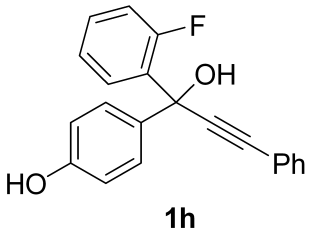
Supplementary Figure 13. ^{1}H NMR spectrum for 1g



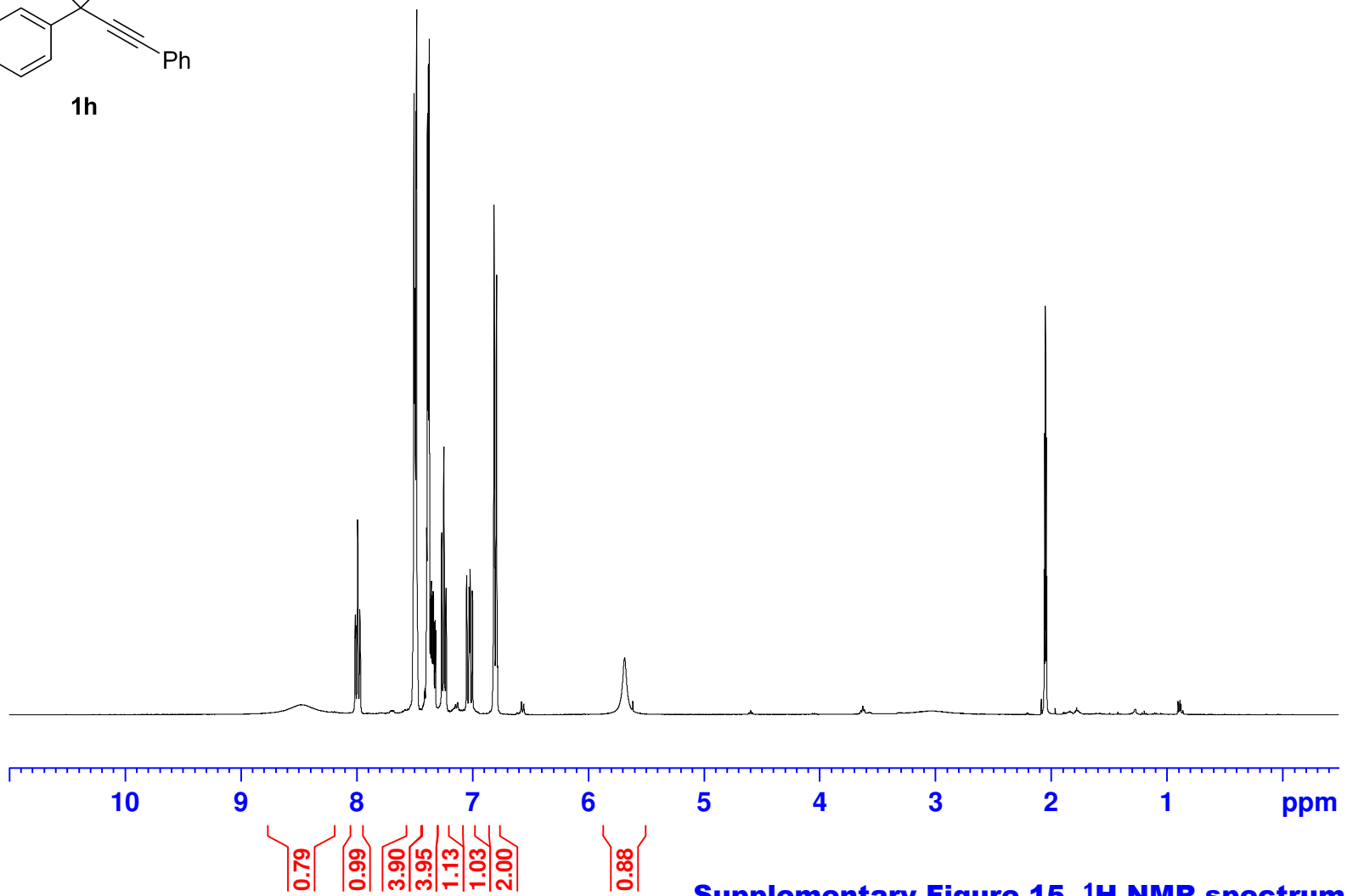

```
Current Data Parameters
NAME      qdy-40012-1 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20161214
Time              21.57
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   46
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.60 usec
PLW1         31.98900032 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          9.10000038 W
PLW12         0.24608000 W
PLW13         0.19933000 W

F2 - Processing parameters
SI                 32768
SF           100.6127780 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 14. ^{13}C NMR spectrum for 1g



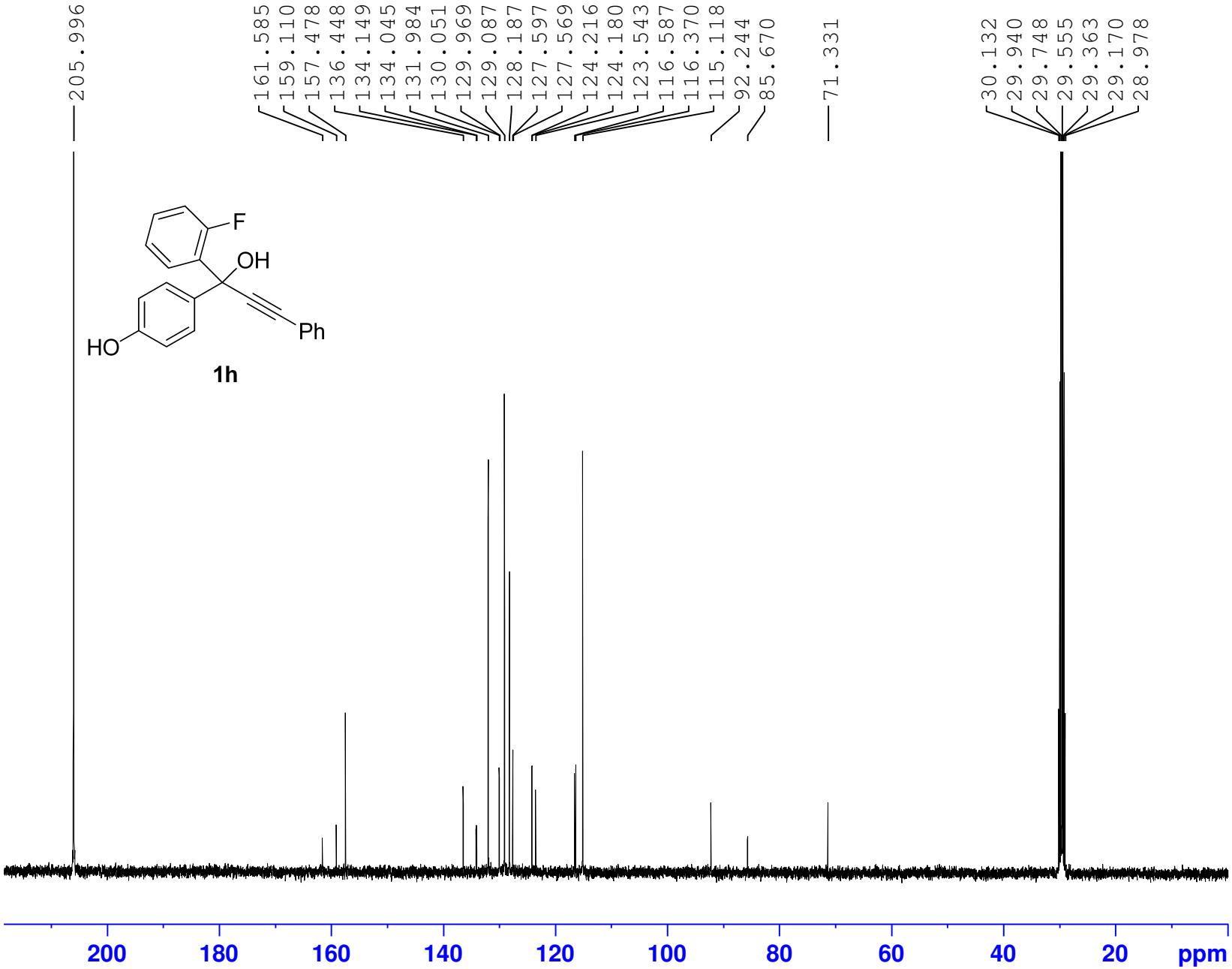


1h




```
Current Data Parameters
NAME         qdy-20089 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160324
Time               19.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 70.97
DW                62.400 usec
DE                 6.50 usec
TE                296.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 15. ^{1}H NMR spectrum for 1h



Current Data Parameters
NAME qdy-20089 C
EXPNO 1
PROCNO 1

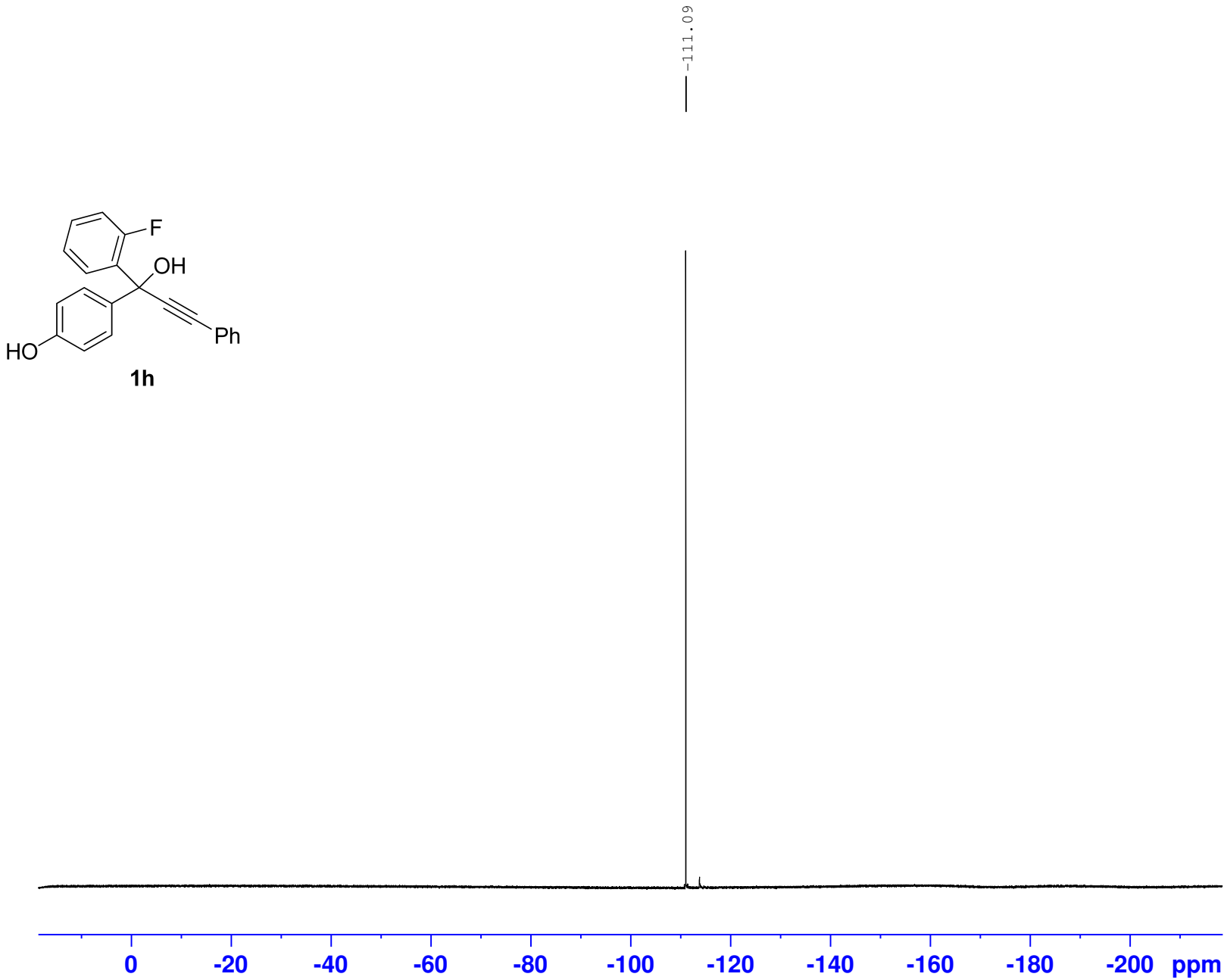
F2 - Acquisition Parameters
Date_ 20160324
Time 19.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 206
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

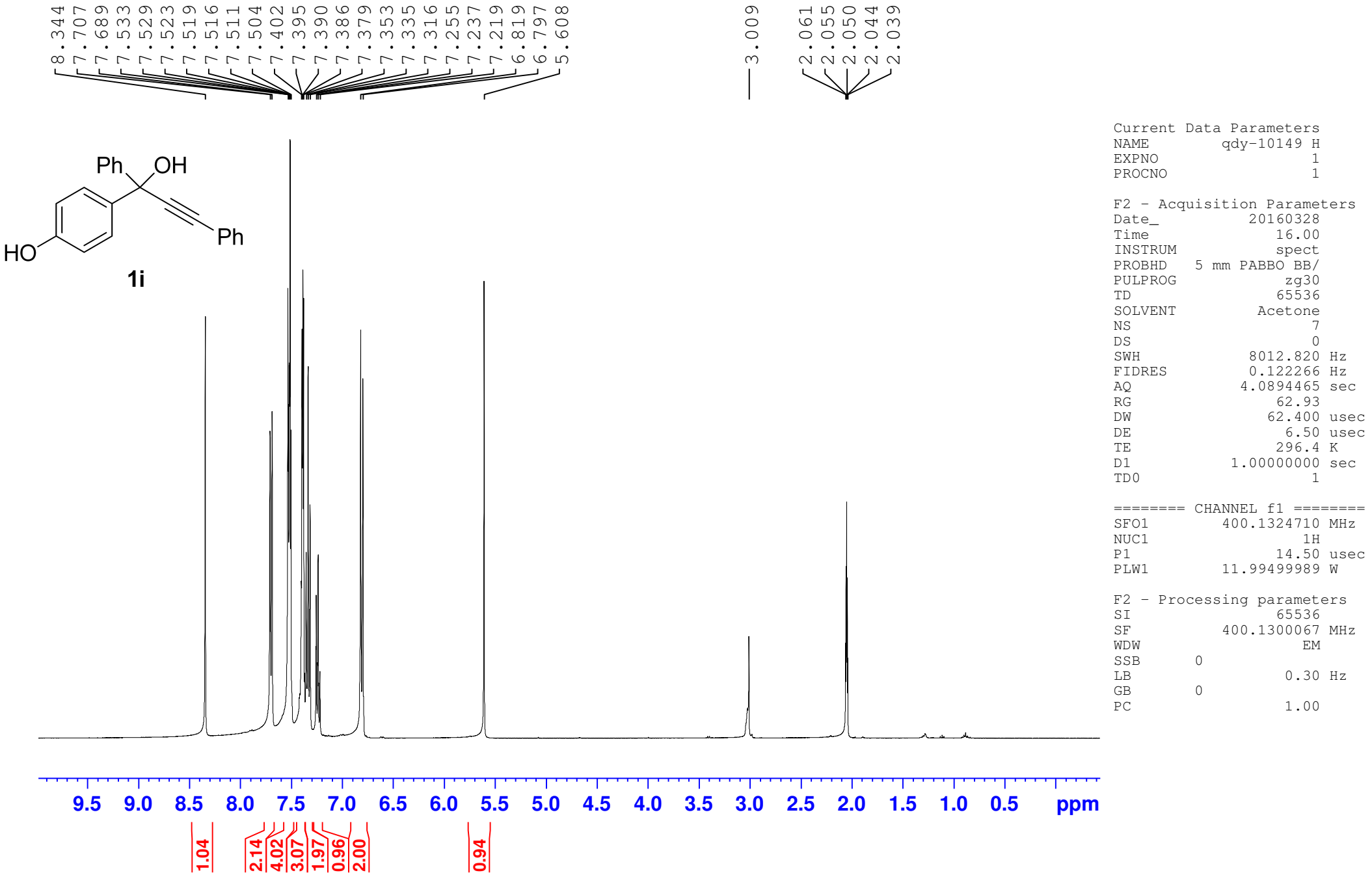
F2 - Processing parameters
SI 32768
SF 100.6127115 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 16. ^{13}C NMR spectrum for 1h



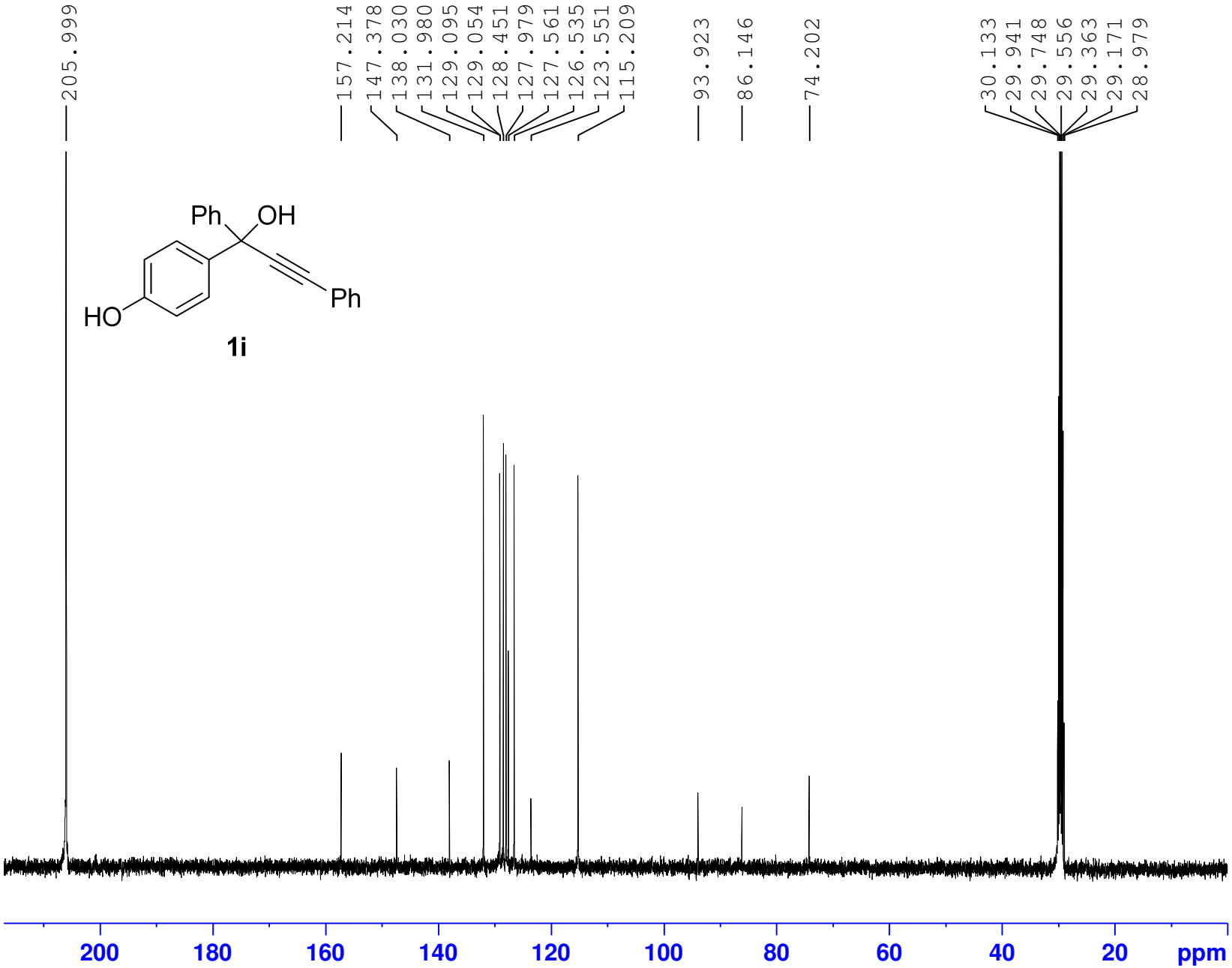

```
Current Data Parameters
NAME         qdy-20089 F
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160324
Time               19.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgflqn
TD               131072
SOLVENT         Acetone
NS                    16
DS                     0
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ             0.7340032 sec
RG                196.92
DW                5.600 usec
DE                6.50 usec
TE                296.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         376.4607164 MHz
NUC1                 19F
P1                14.70 usec
PLW1         15.99600029 W

F2 - Processing parameters
SI                 65536
SF           376.4983660 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 17. ^{19}F NMR spectrum for 1h




```
Current Data Parameters
NAME         qdy-10149 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               16.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     7
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 62.93
DW                62.400 usec
DE                 6.50 usec
TE                296.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 18. ^{1}H NMR spectrum for 1i



Current Data Parameters
NAME qdy-10149 C
EXPNO 1
PROCNO 1

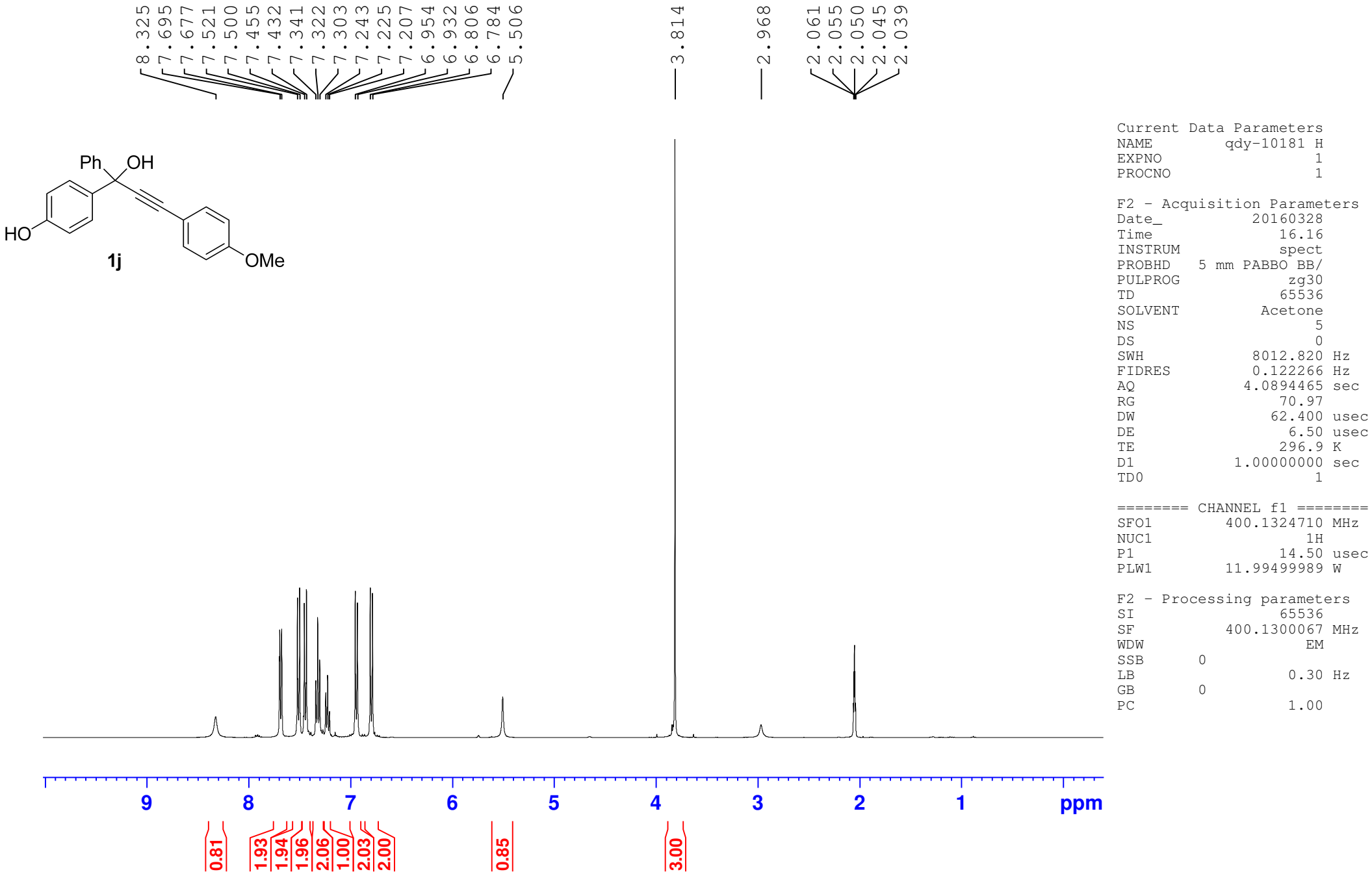
F2 - Acquisition Parameters
Date_ 20160328
Time 16.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 78
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

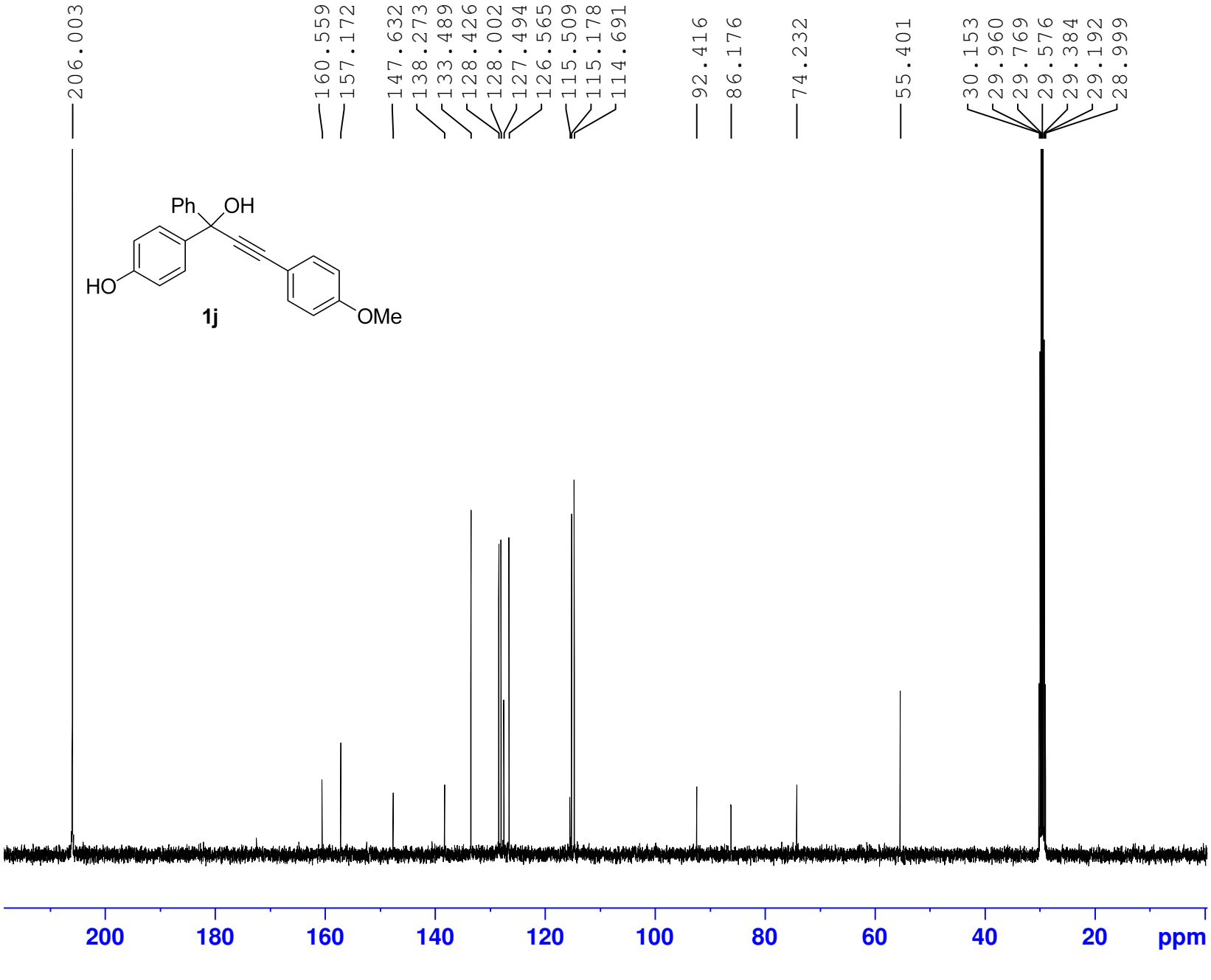
F2 - Processing parameters
SI 32768
SF 100.6127125 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 19. ^{13}C NMR spectrum for 1i



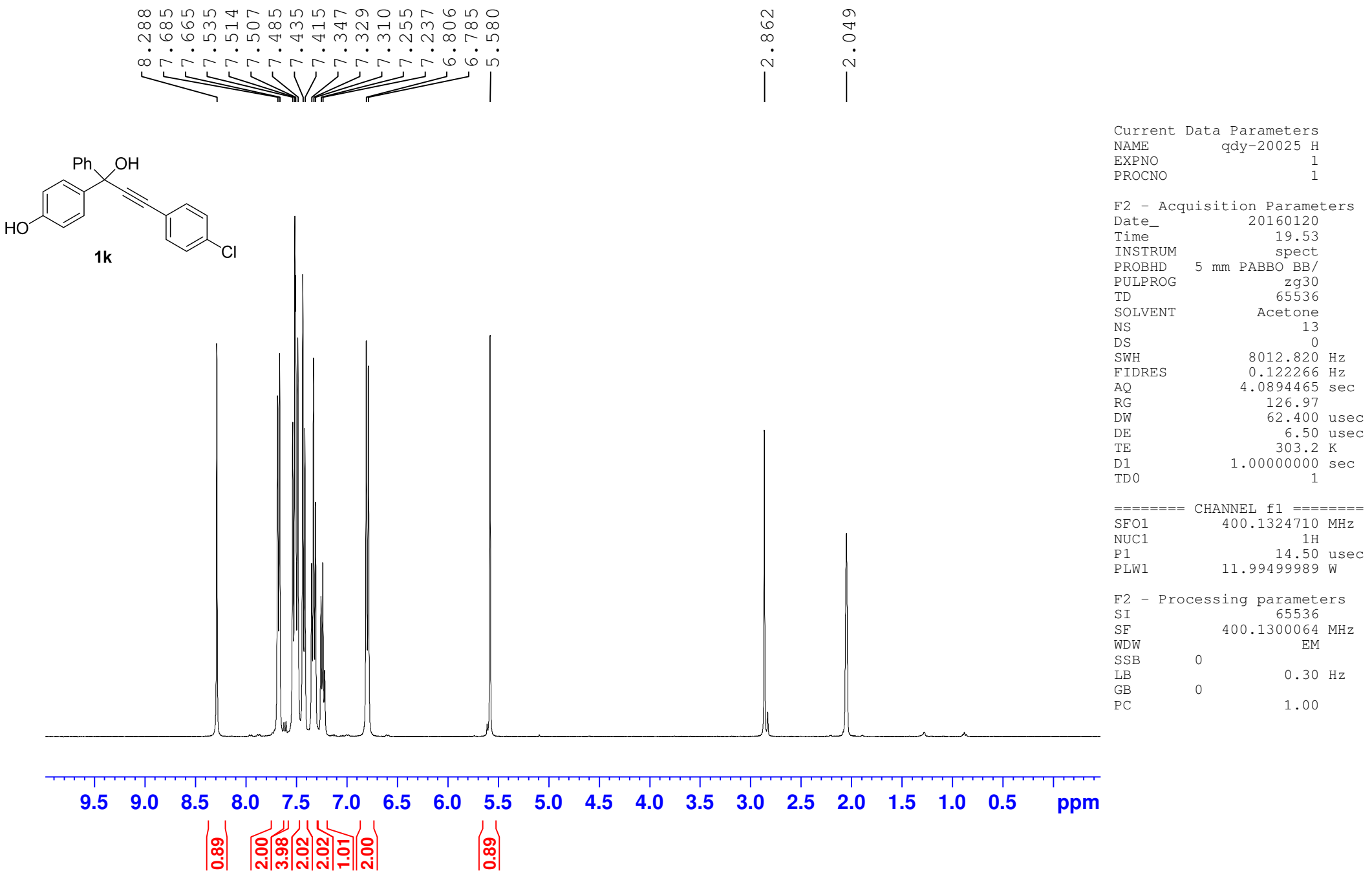

```
Current Data Parameters
NAME         qdy-10181 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               16.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     5
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 70.97
DW                62.400 usec
DE                 6.50 usec
TE                296.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300067 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 20. ^{1}H NMR spectrum for 1j



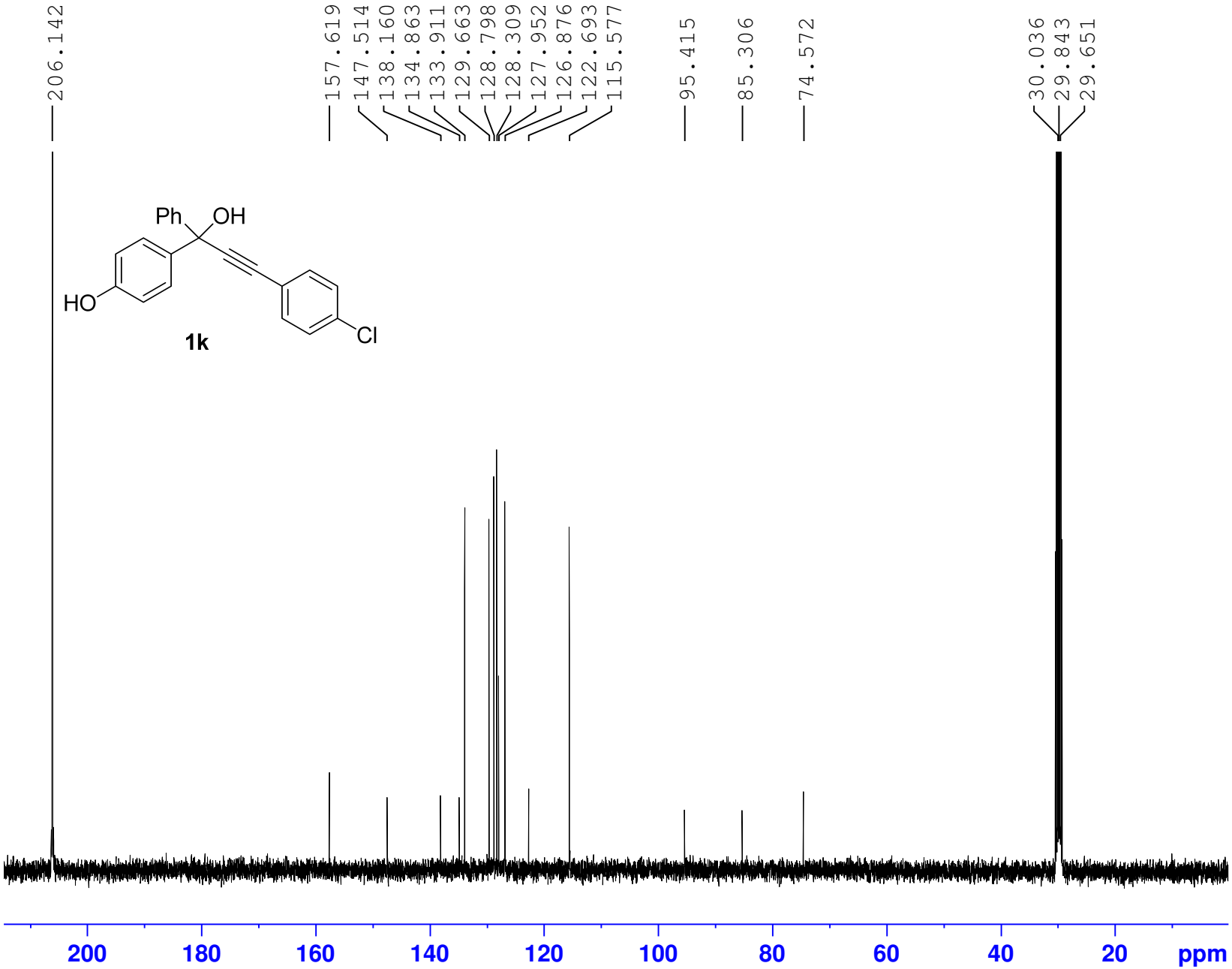

```
Current Data Parameters
NAME         qdy-10181 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               16.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                    50
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127095 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 21. ^{13}C NMR spectrum for 1j




```
Current Data Parameters
NAME         qdy-20025 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160120
Time               19.53
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    13
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                126.97
DW                62.400 usec
DE                 6.50 usec
TE                303.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300064 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 22. ^{1}H NMR spectrum for 1k



Current Data Parameters
NAME qdy-20025 C
EXPNO 1
PROCNO 1

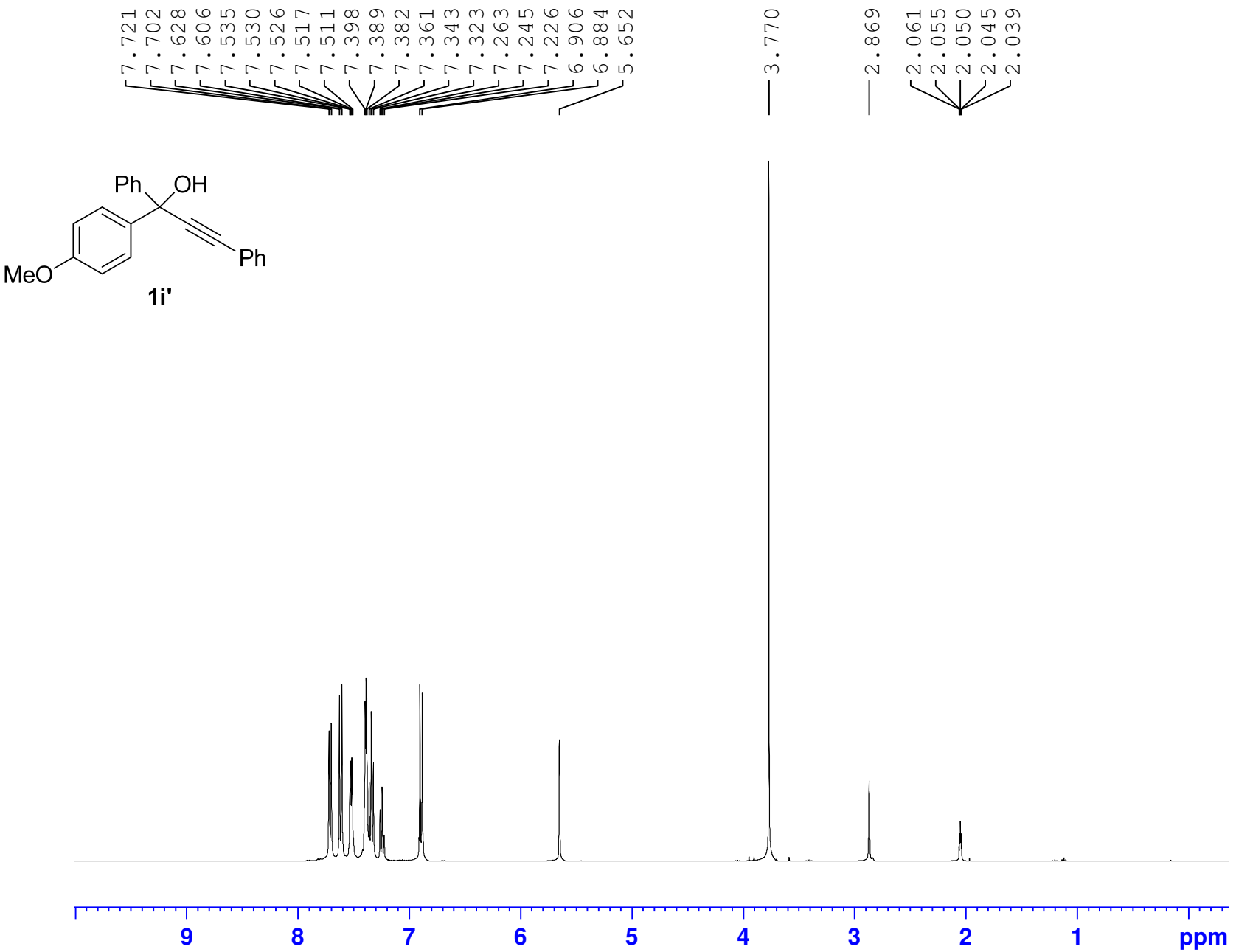
F2 - Acquisition Parameters
Date_ 20160120
Time 19.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 101
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 303.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

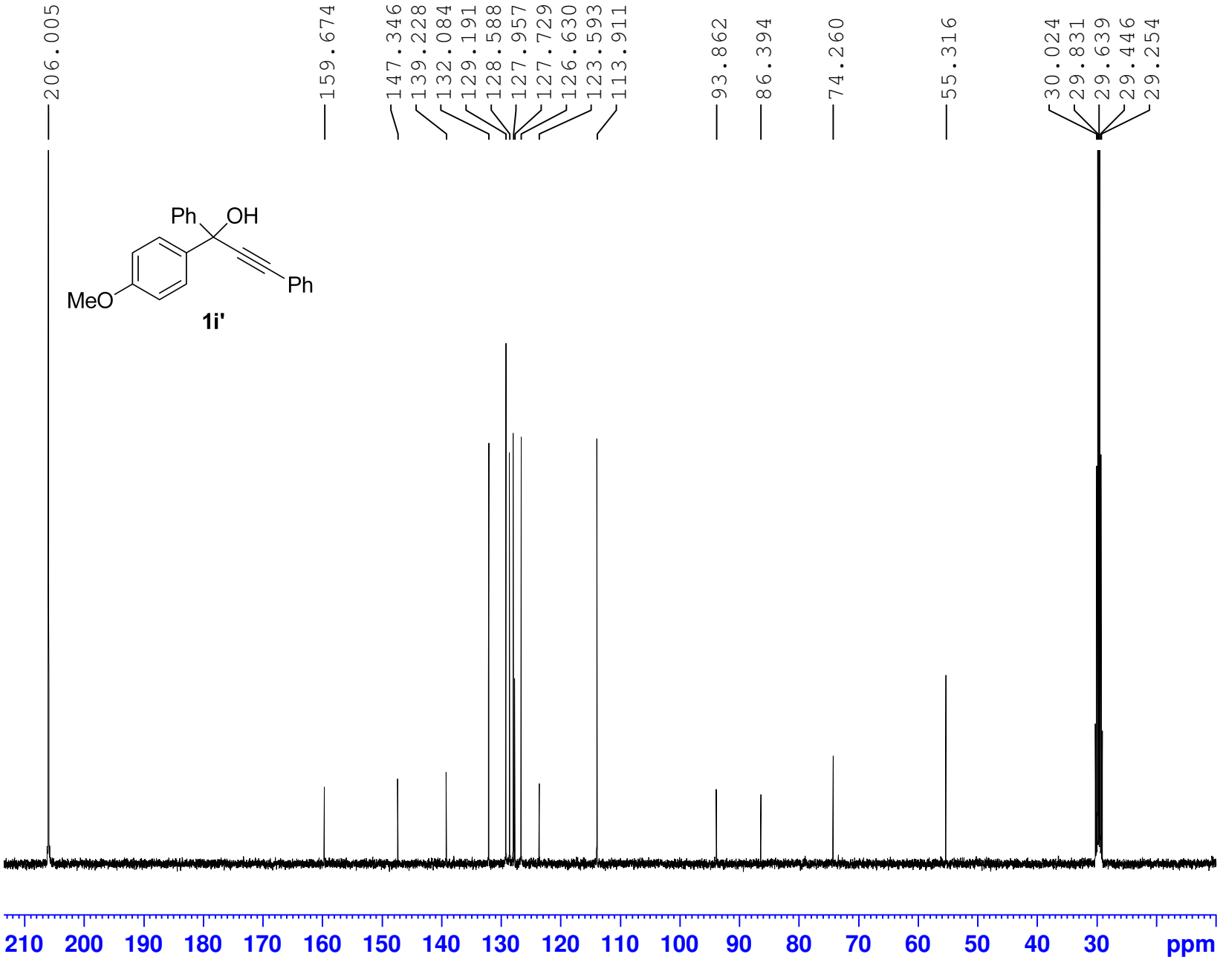
F2 - Processing parameters
SI 32768
SF 100.6126768 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 23. ^{13}C NMR spectrum for 1k



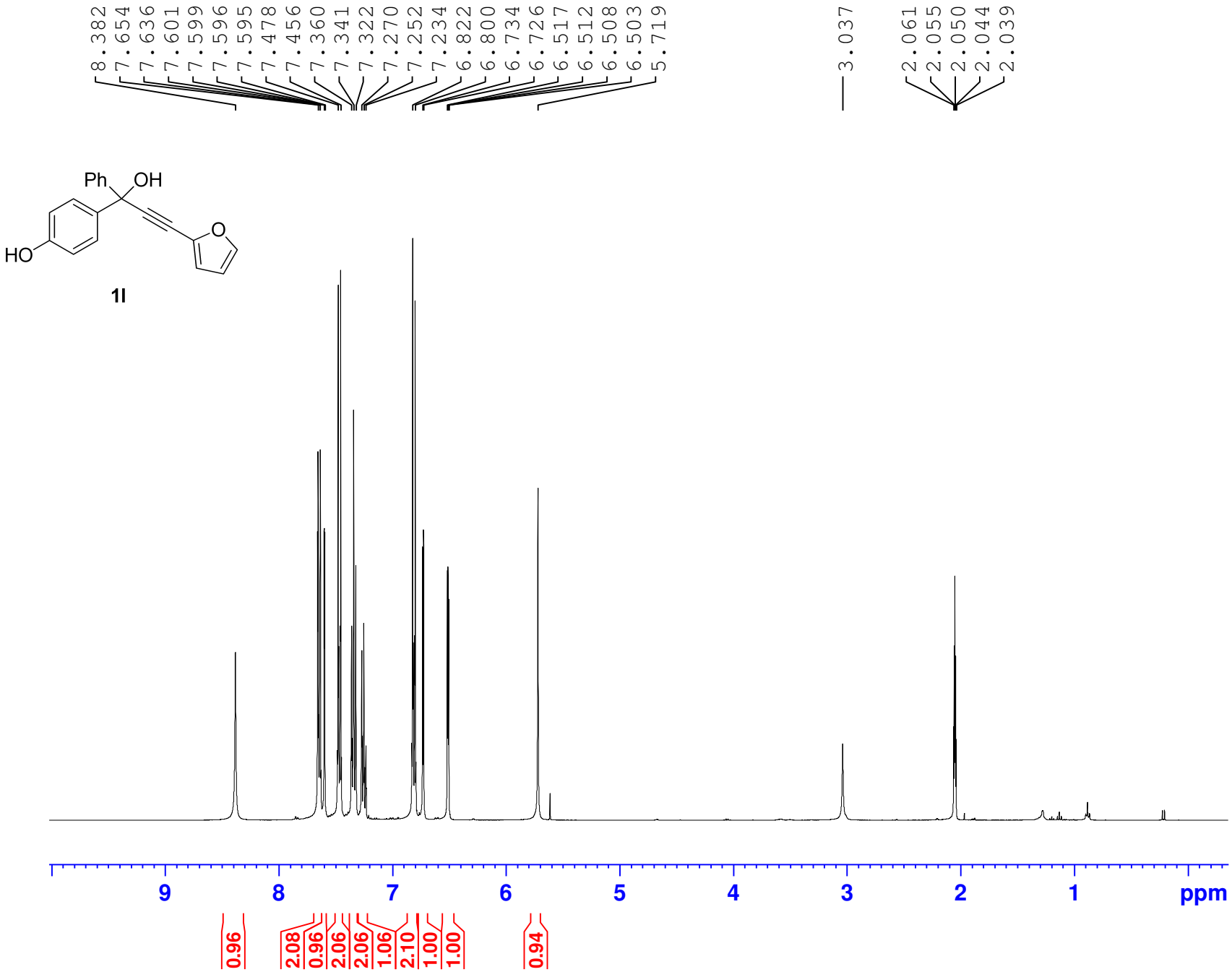

```
Current Data Parameters
NAME         qdy-10173 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160529
Time              21.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                31.55
DW               62.400 usec
DE                6.50 usec
TE                300.4 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                   65536
SF           400.1300066 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 24. ^{1}H NMR spectrum for 1i'



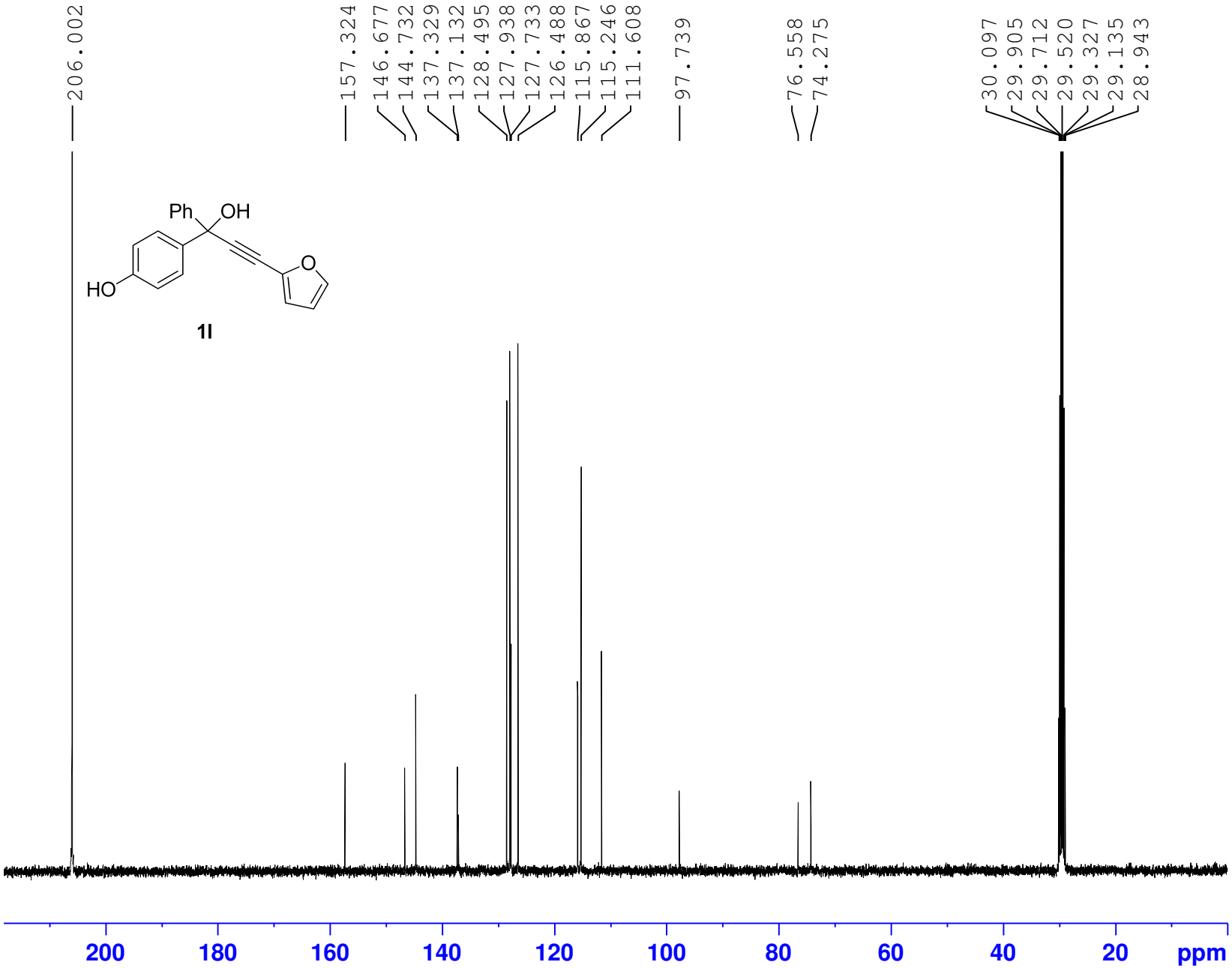

```
Current Data Parameters
NAME         qdy-10173 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160529
Time              21.02
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  106
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127034 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 25. ^{13}C NMR spectrum for 1i'



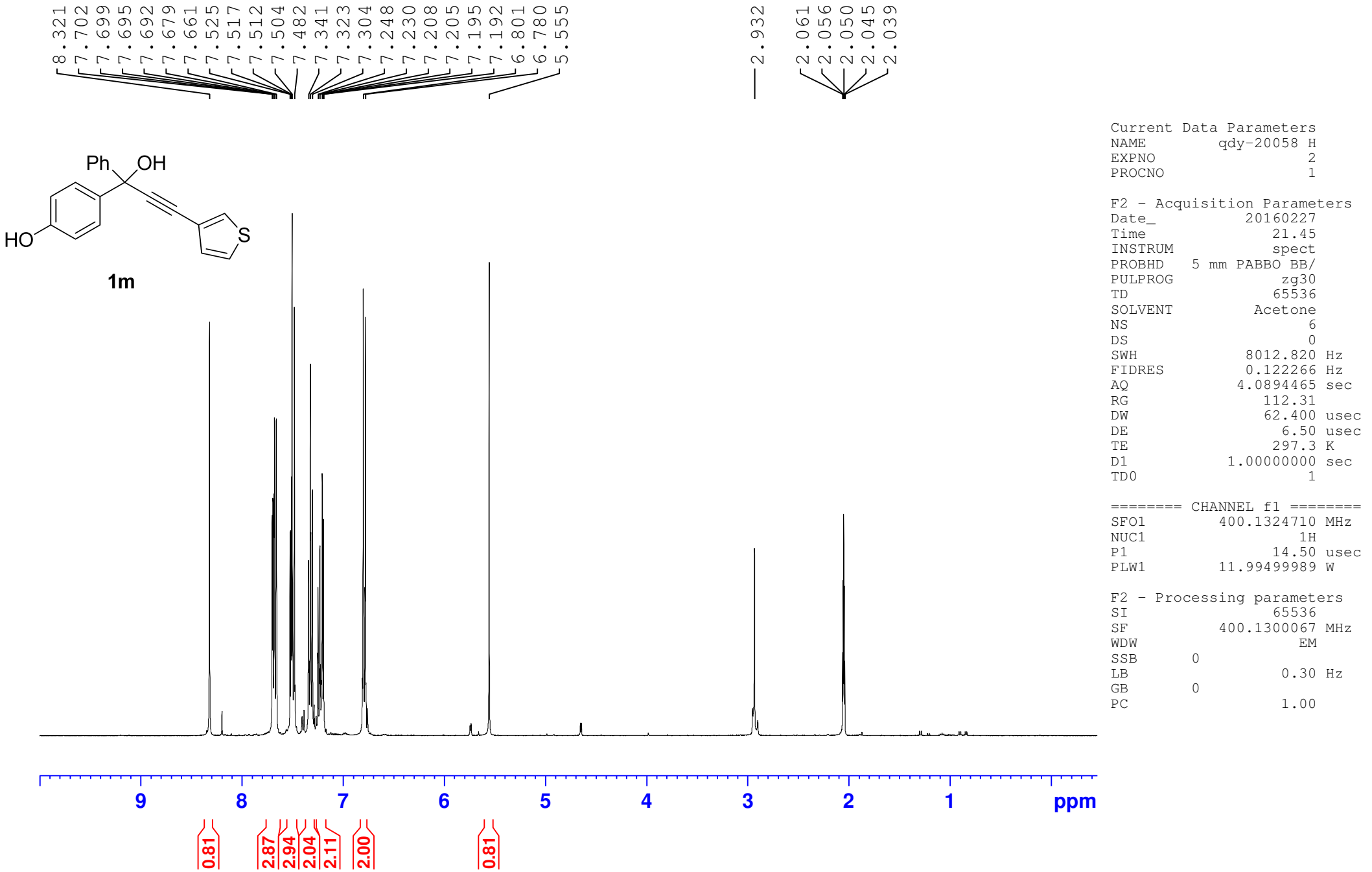

```
Current Data Parameters
NAME      qdy-wcw-1019 H
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160326
Time              14.03
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                   16
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                54.81
DW               62.400 usec
DE                6.50 usec
TE                296.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1300066 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 26. ^{1}H NMR spectrum for 1l



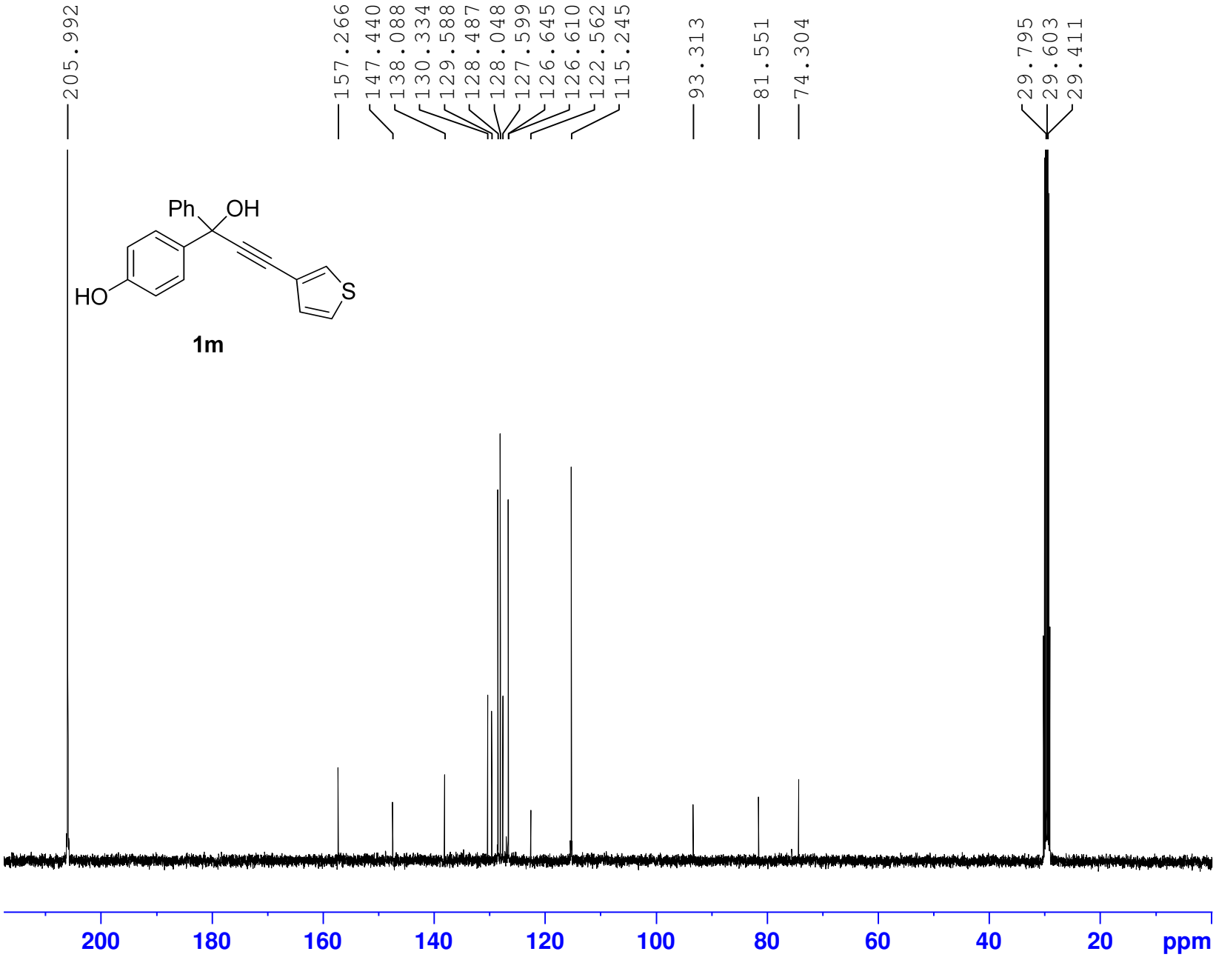

```
Current Data Parameters
NAME      qdy-wcw-1019 C
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160326
Time              14.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                  101
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           100.6127162 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 27. ^{13}C NMR spectrum for 1l



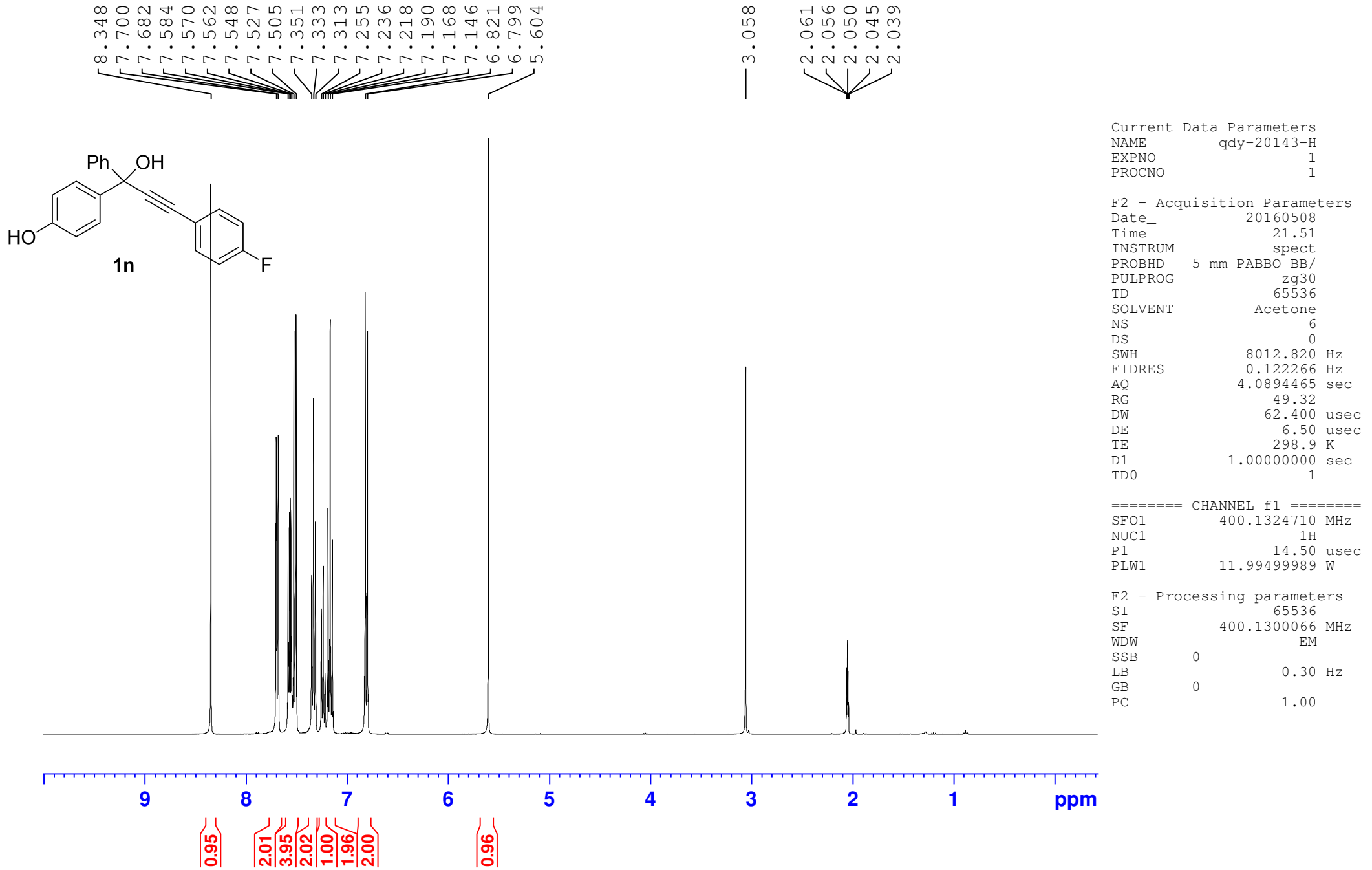

```
Current Data Parameters
NAME         qdy-20058 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160227
Time               21.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                112.31
DW                62.400 usec
DE                 6.50 usec
TE                297.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300067 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 28. ^{1}H NMR spectrum for 1m



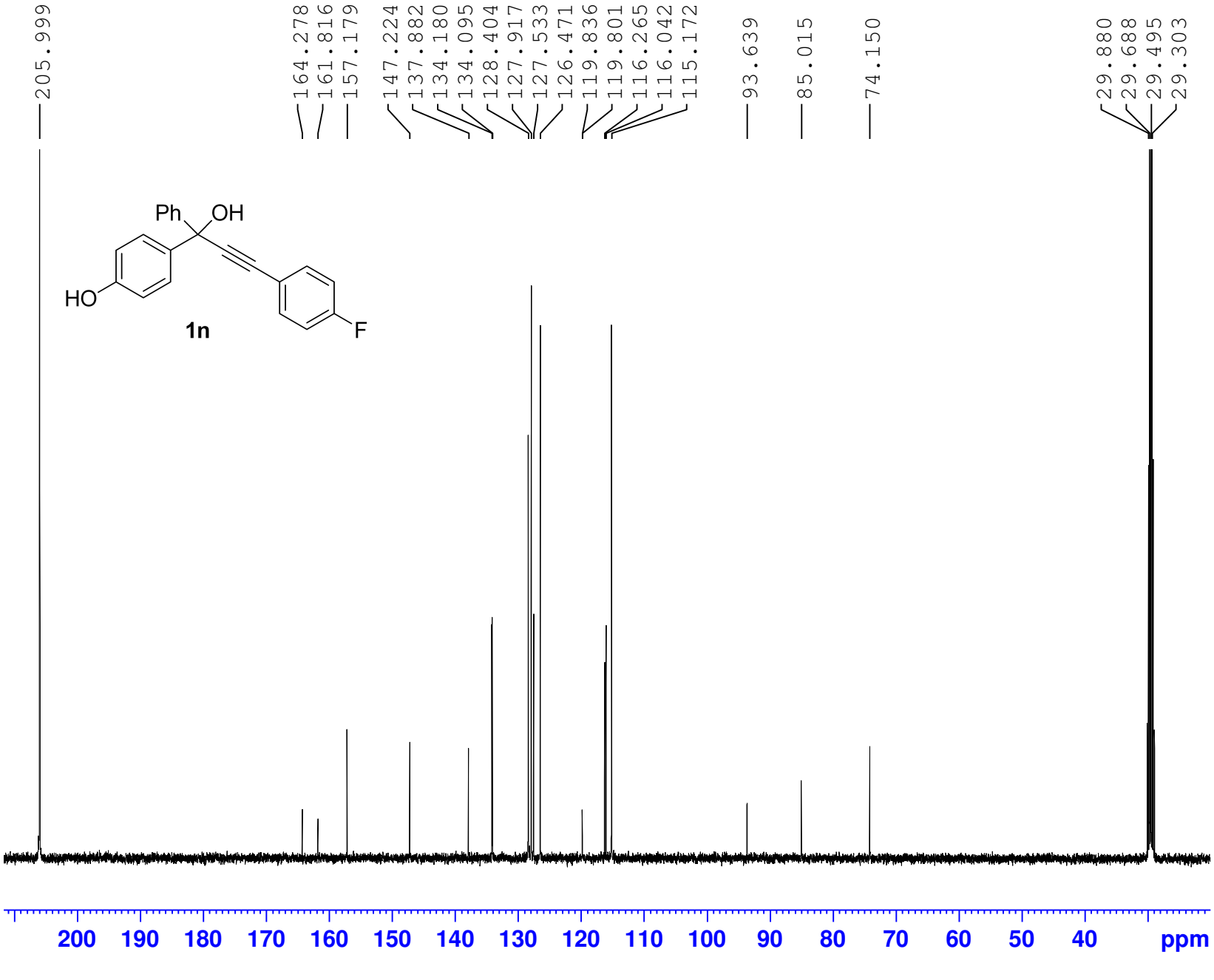

```
Current Data Parameters
NAME         qdy-20058 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160227
Time               16.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   159
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                  9.70 usec
PLW1          46.98899841 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          11.99499989 W
PLW12          0.34213999 W
PLW13          0.27713001 W

F2 - Processing parameters
SI                 32768
SF            100.6127052 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 29. ^{13}C NMR spectrum for 1m



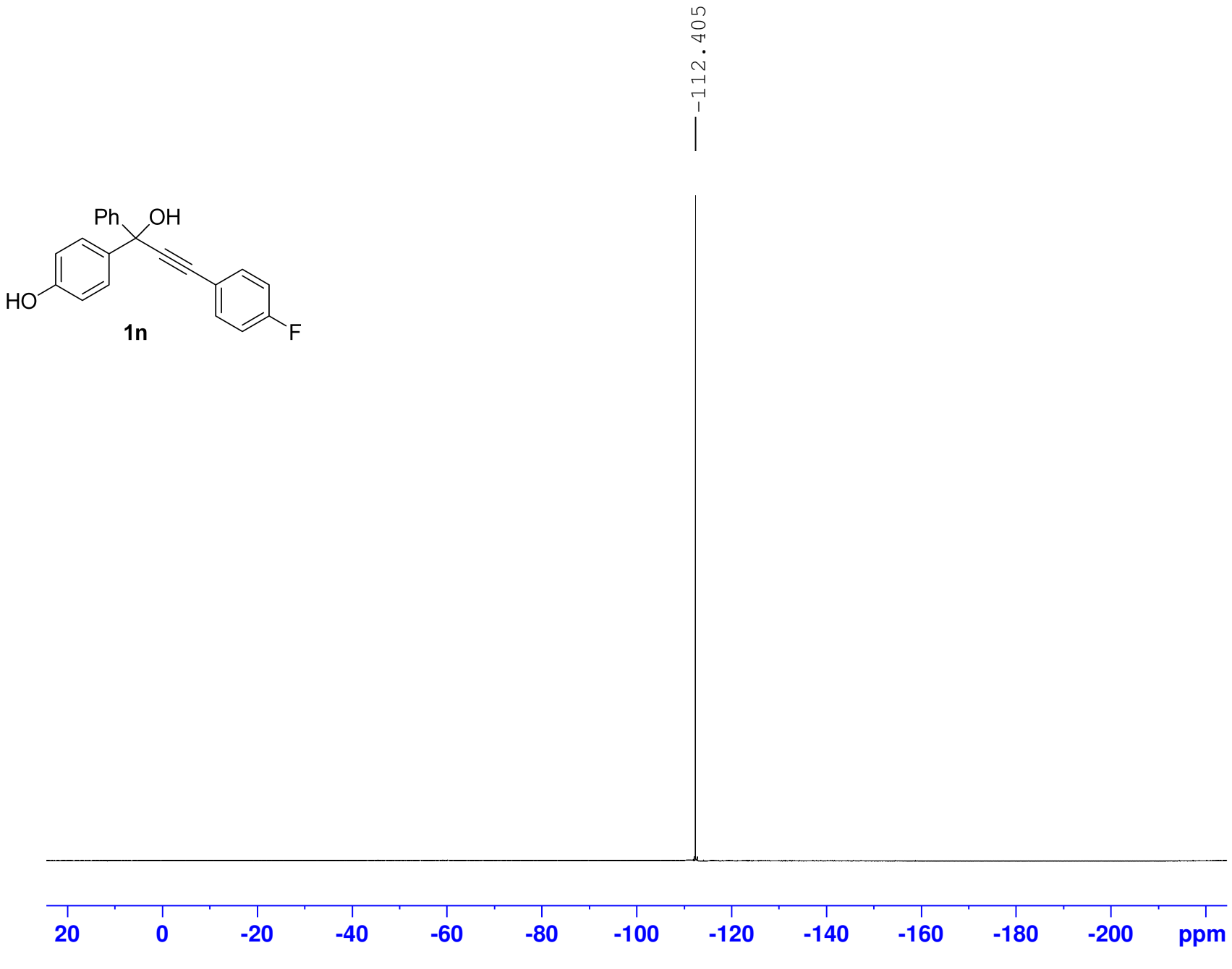

```
Current Data Parameters
NAME         qdy-20143-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160508
Time               21.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 49.32
DW                62.400 usec
DE                 6.50 usec
TE                298.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300066 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 30. ^{1}H NMR spectrum for 1n



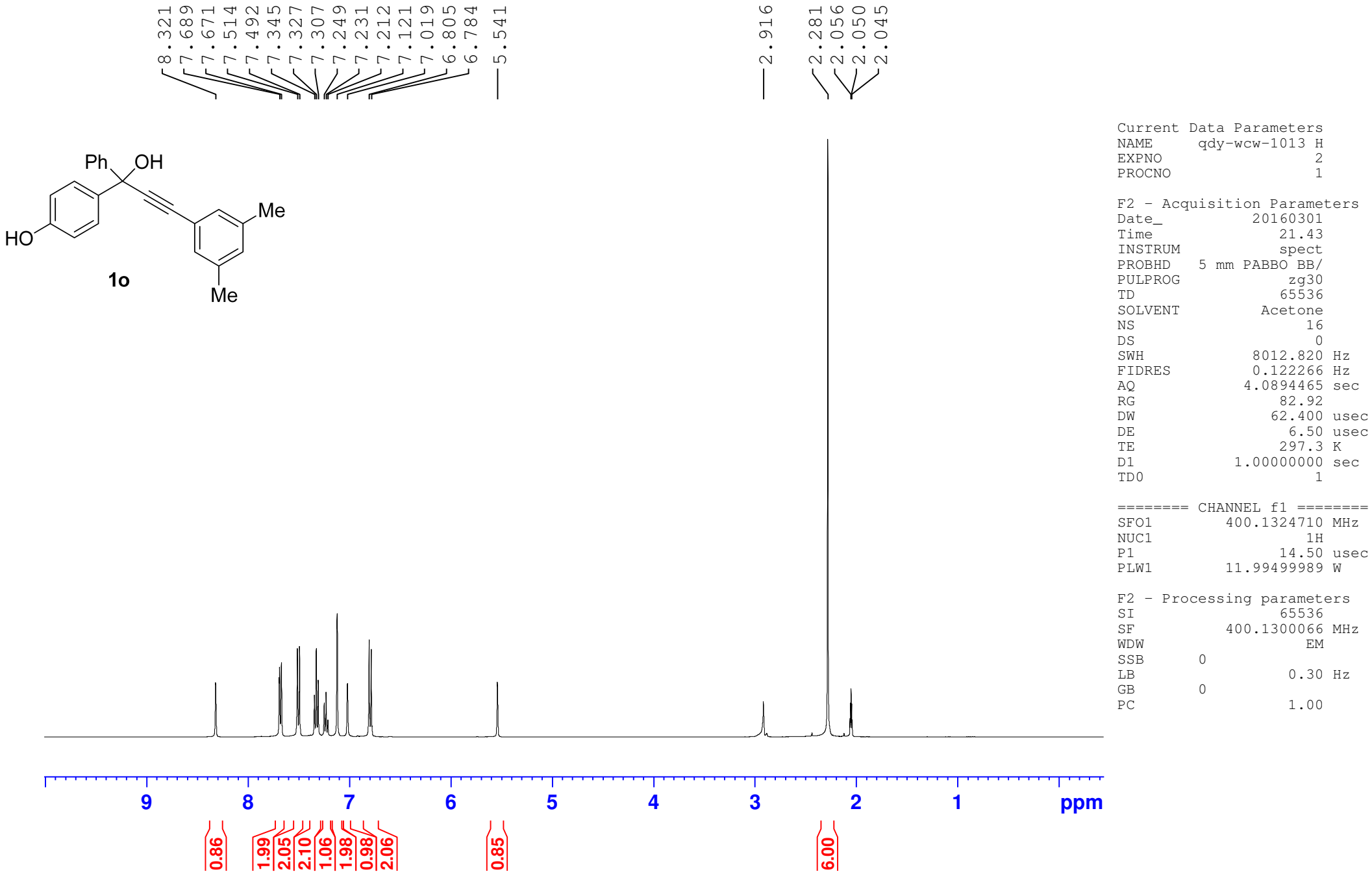

```
Current Data Parameters
NAME         qdy-20143-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160508
Time               21.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                   80
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127184 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 31. ^{13}C NMR spectrum for 1n




```
Current Data Parameters
NAME         qdy-20143-F
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160508
Time               22.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                     4
DS                     0
SWH            93750.000 Hz
FIDRES          1.430511 Hz
AQ             0.3495253 sec
RG                196.92
DW                5.333 usec
DE                 6.50 usec
TE                299.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         376.4607162 MHz
NUC1                  19F
P1                14.70 usec
PLW1         15.99600029 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           376.4983660 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 32. ^{19}F NMR spectrum for 1n



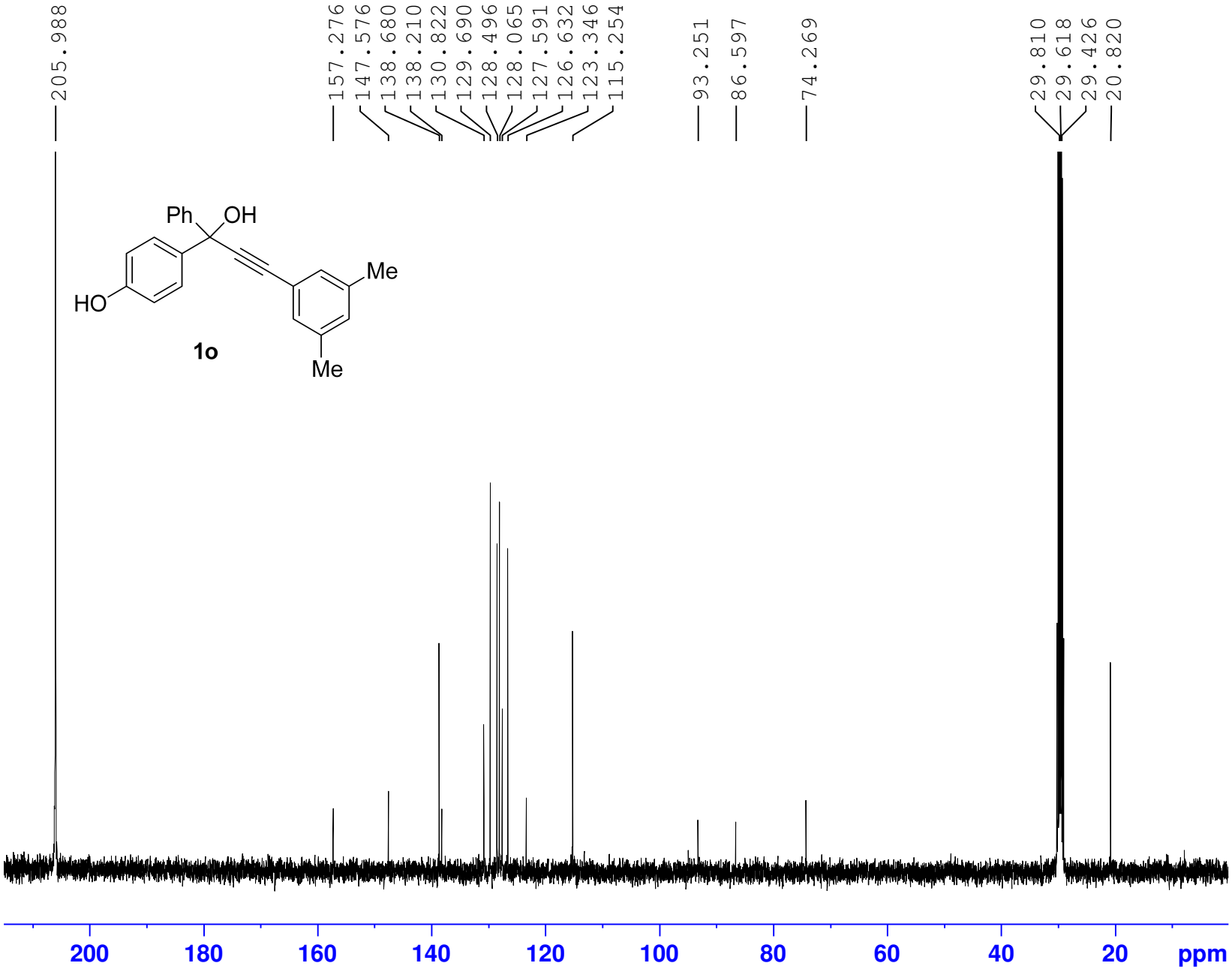
Current Data Parameters
NAME qdy-wcw-1013 H
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160301
Time 21.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

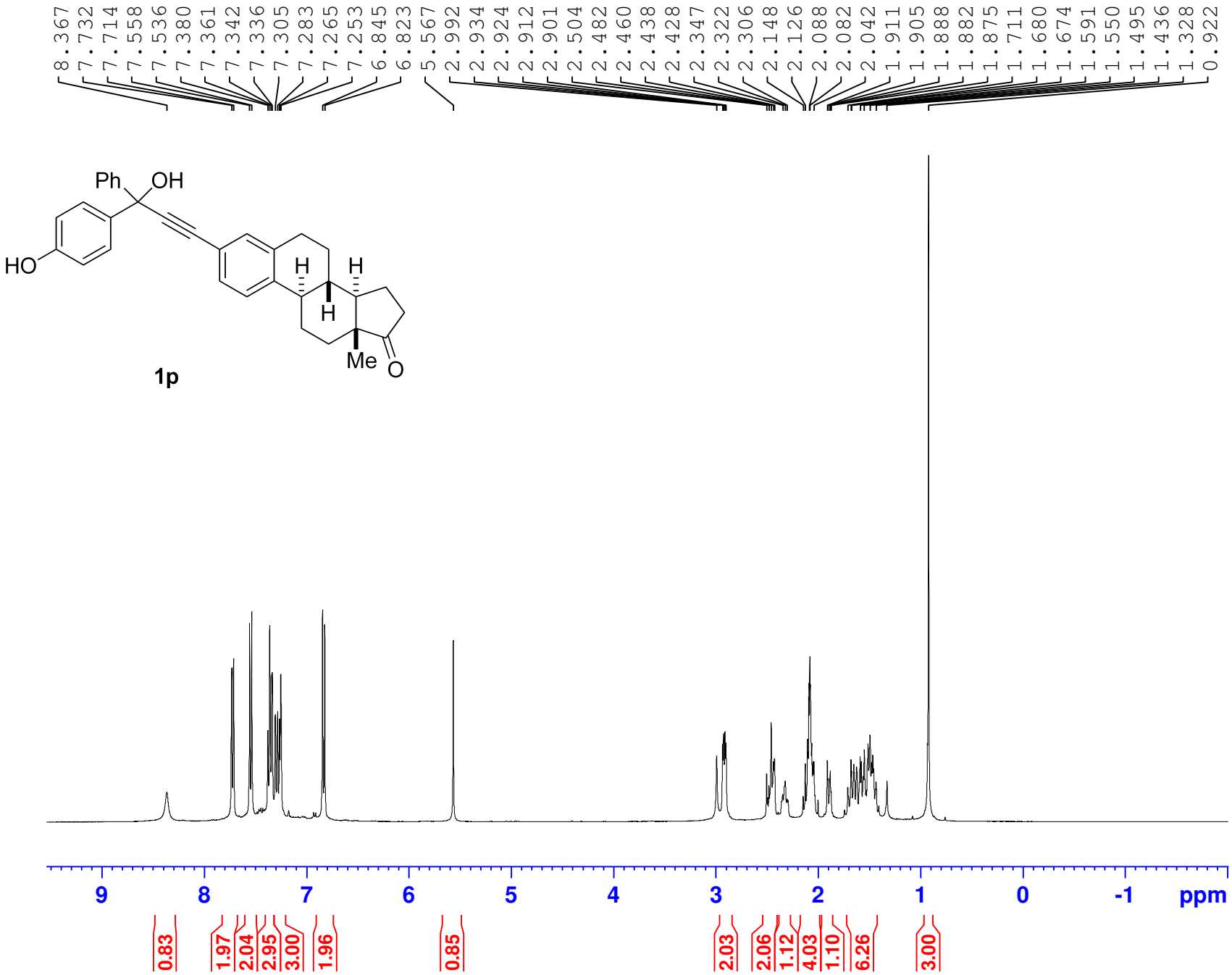
F2 - Processing parameters
SI 65536
SF 400.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 33. ^{1}H NMR spectrum for 1o



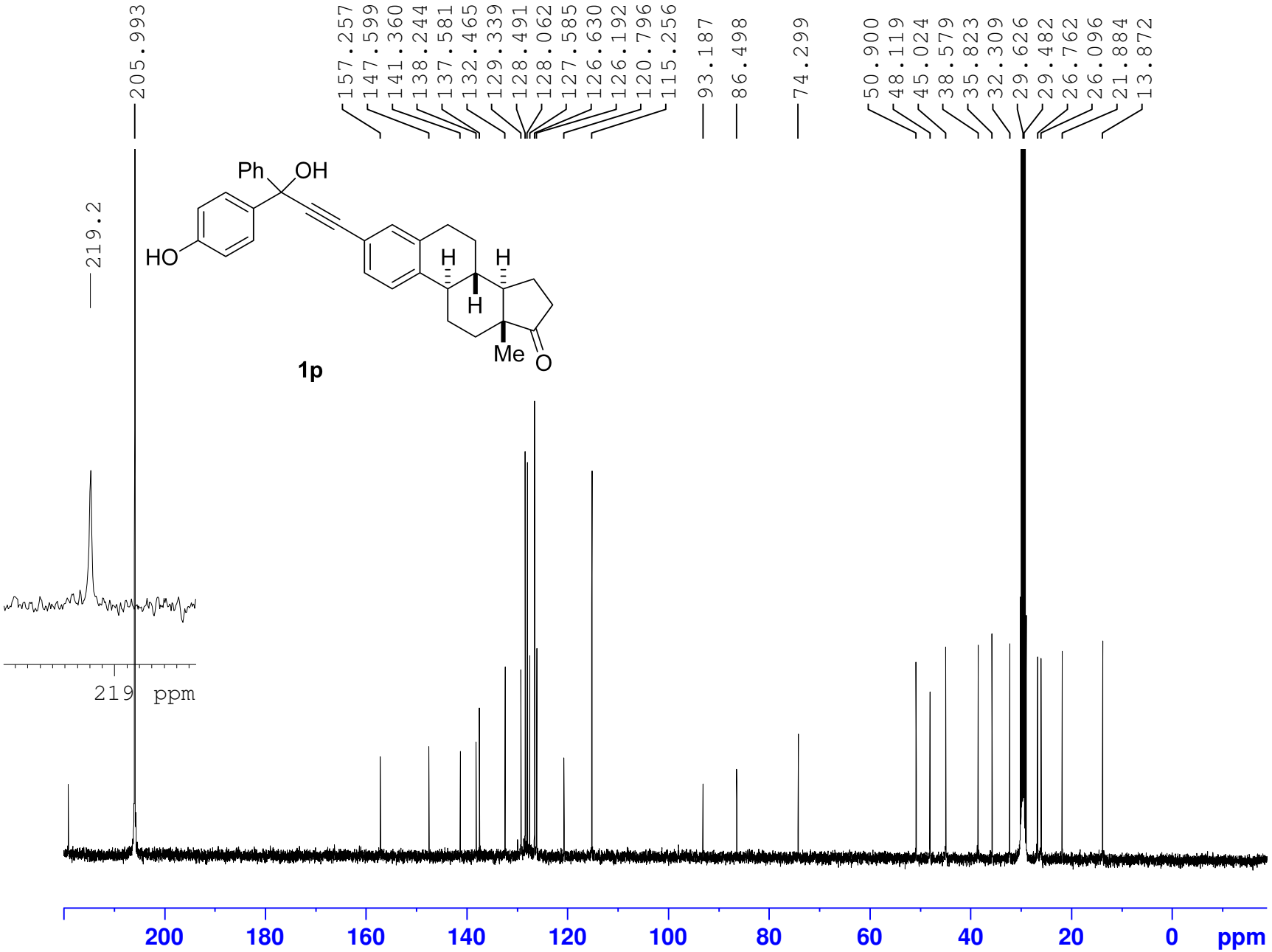

```
Current Data Parameters
NAME      qdy-wcw-1013 C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160301
Time              21.36
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                   81
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           100.6127029 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 34. ^{13}C NMR spectrum for 1o



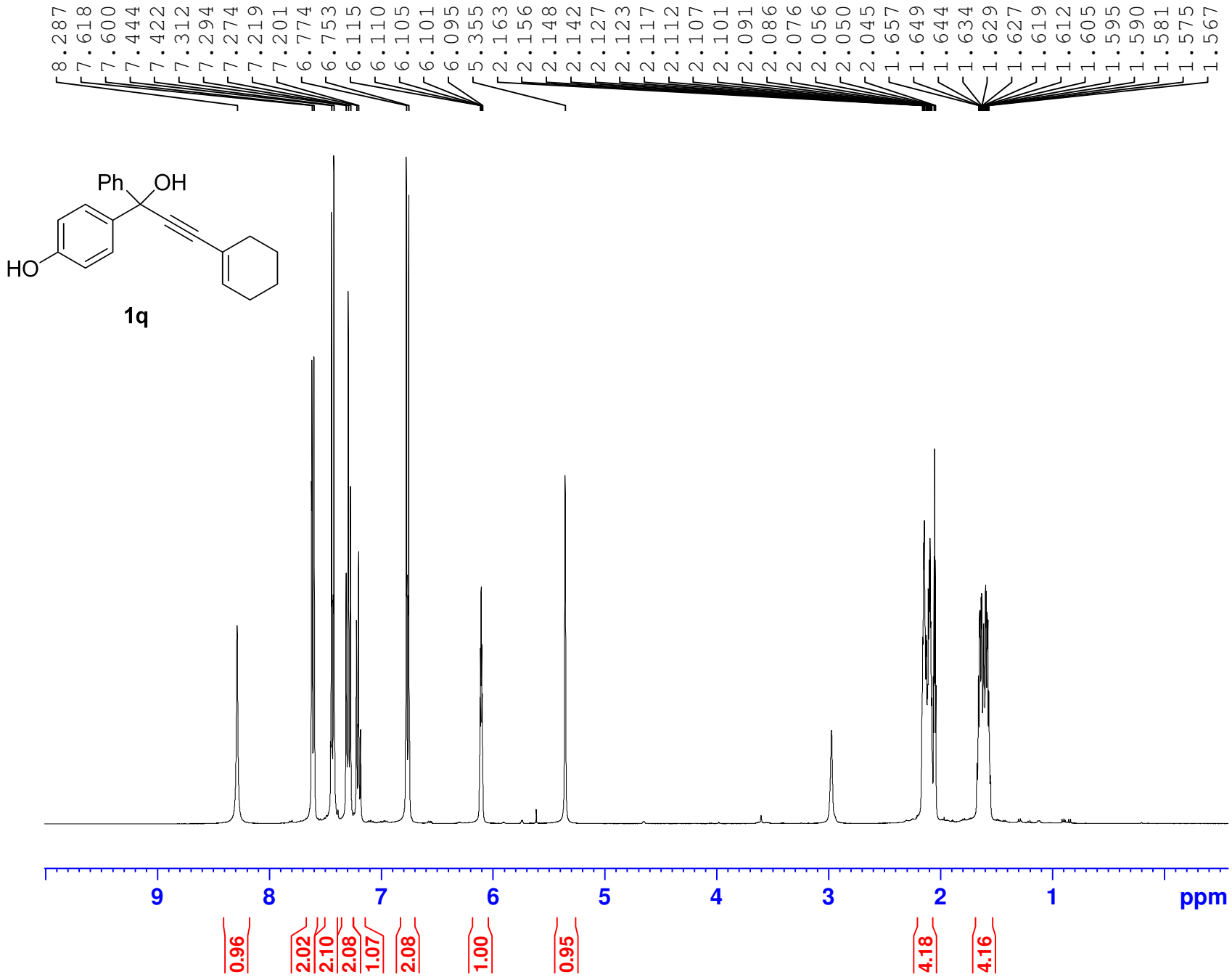

```
Current Data Parameters
NAME       qdy-20110-2 H
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160415
Time              23.03
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                   12
DS                    0
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                49.32
DW               62.400 usec
DE                6.50 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1299916 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 35. ^{1}H NMR spectrum for 1p



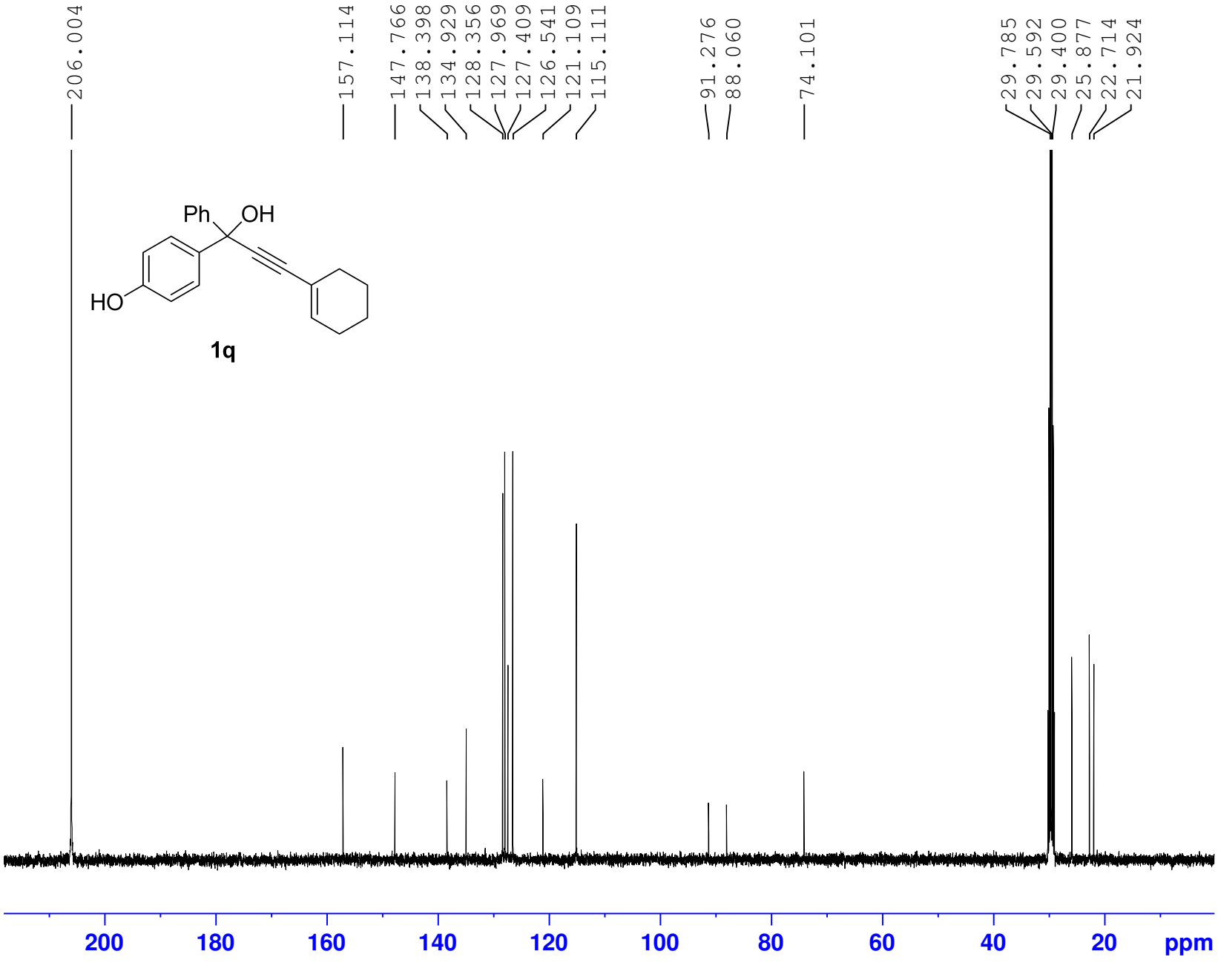

```
Current Data Parameters
NAME      qdy-20110-2 C
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_           20160415
Time              23.10
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  268
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127042 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 36. ^{13}C NMR spectrum for 1p




```
Current Data Parameters
NAME         qdy-20018 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               16.34
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 70.97
DW                62.400 usec
DE                 6.50 usec
TE                296.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300066 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 37. ^{1}H NMR spectrum for 1q



Current Data Parameters
NAME qdy-10181 H
EXPNO 2
PROCNO 1

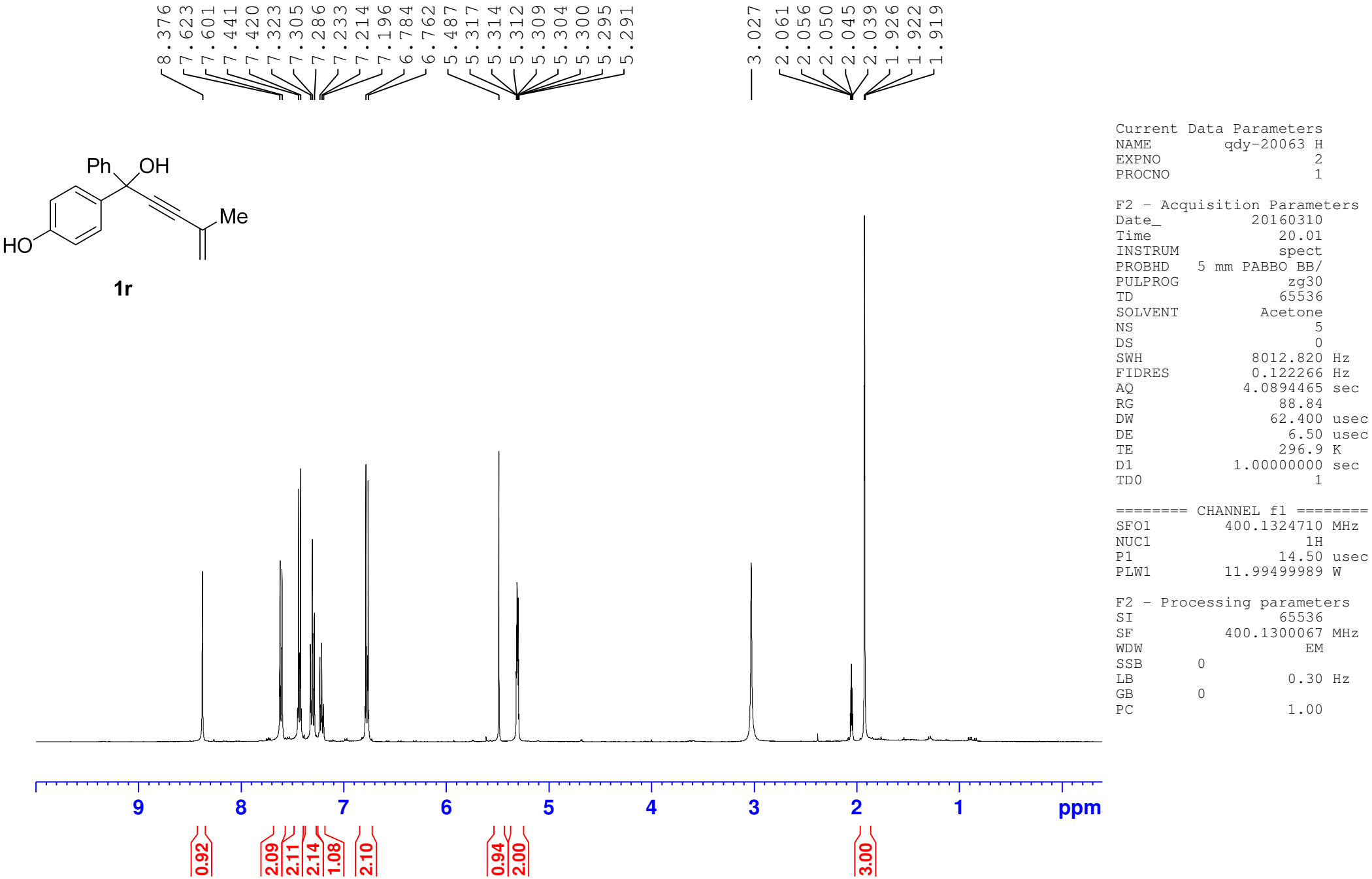
F2 - Acquisition Parameters
Date_ 20160328
Time 16.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 84
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

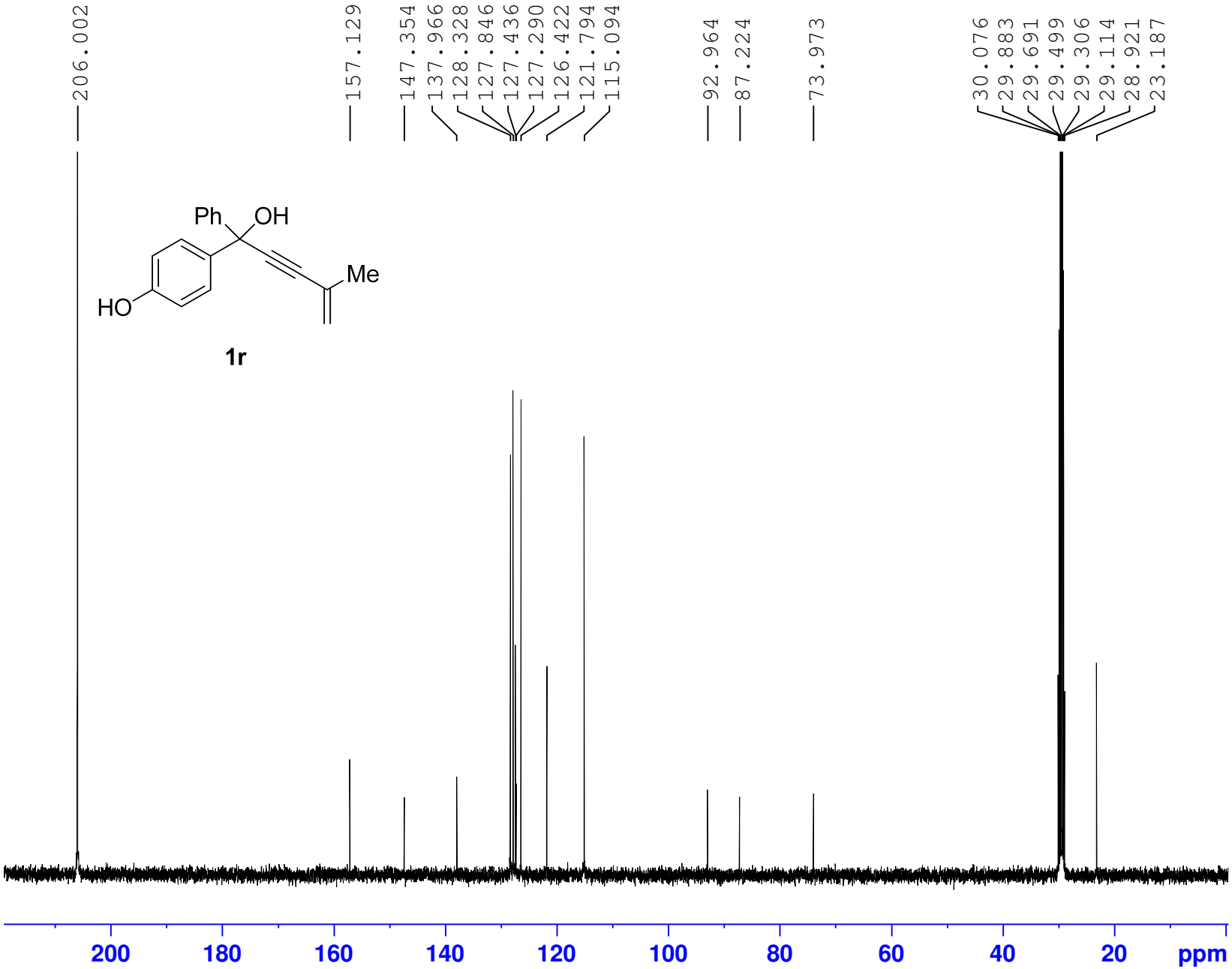
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127072 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 38. ^{13}C NMR spectrum for 1q

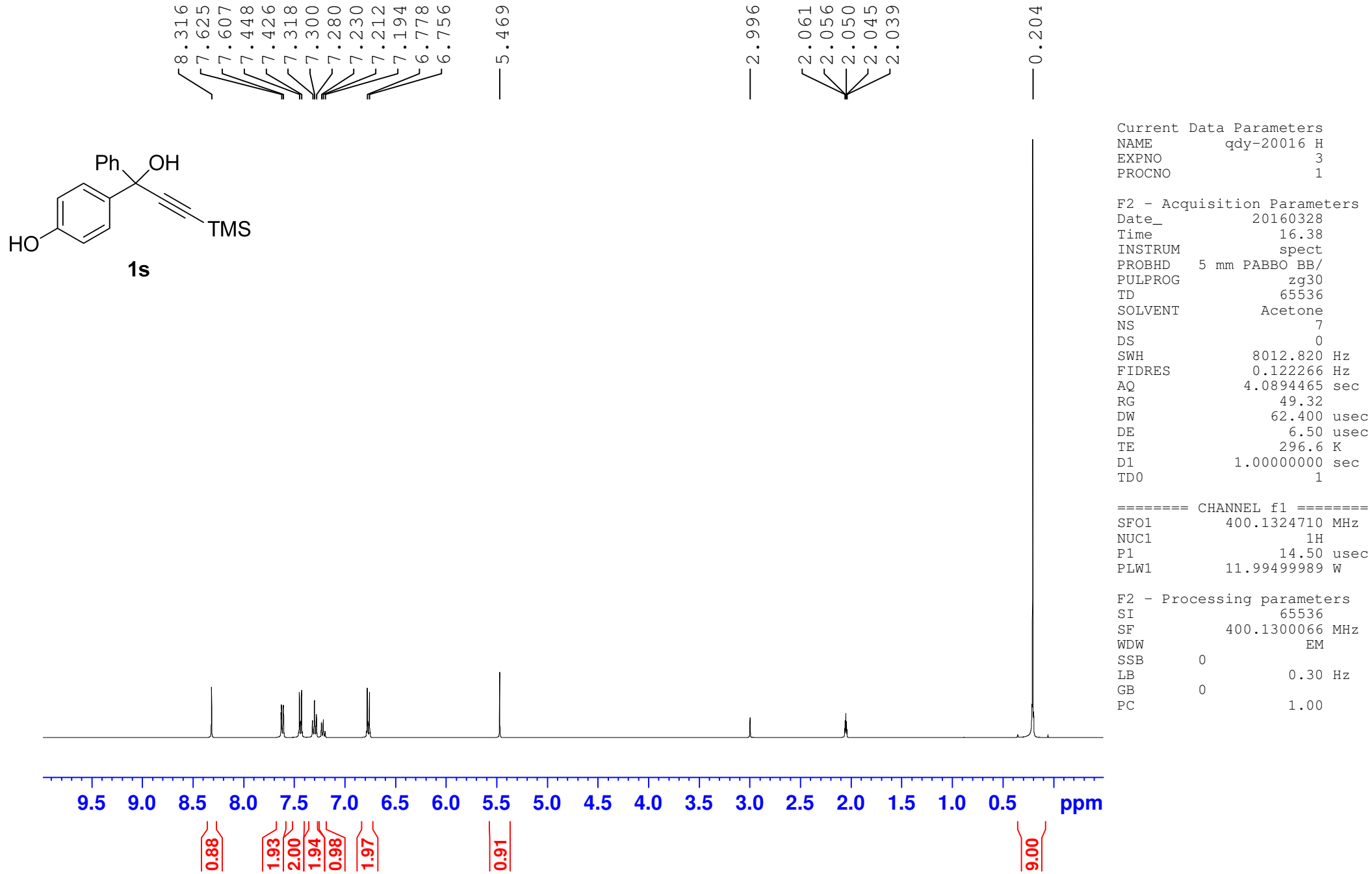


Supplementary Figure 39. ^{1}H NMR spectrum for 1r

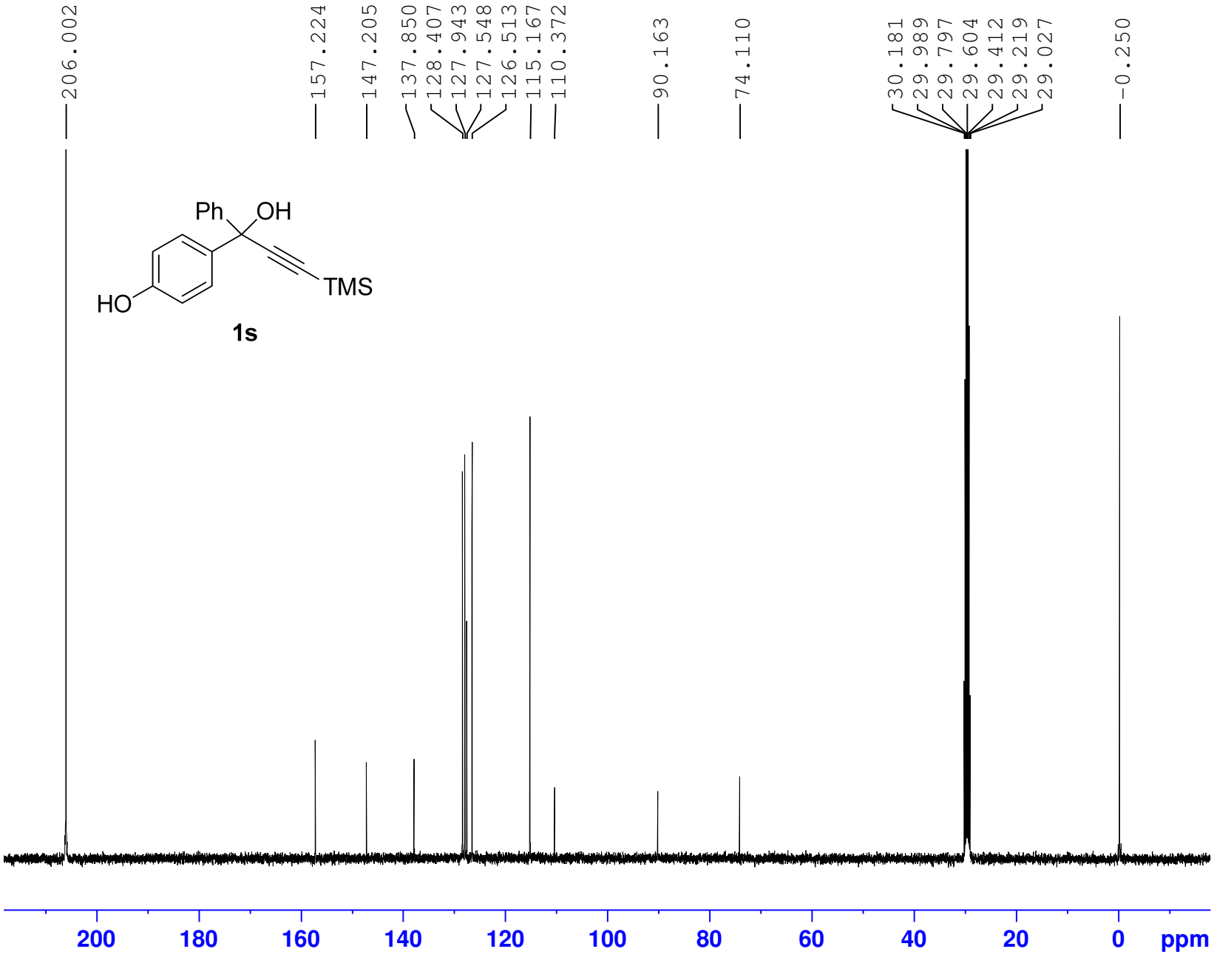



```
Current Data Parameters
NAME         qdy-20063 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160310
Time               19.53
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  143
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127169 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 40. ^{13}C NMR spectrum for 1r



Supplementary Figure 41. ^{1}H NMR spectrum for 1s



Current Data Parameters
NAME qdy-20016 C
EXPNO 3
PROCNO 1

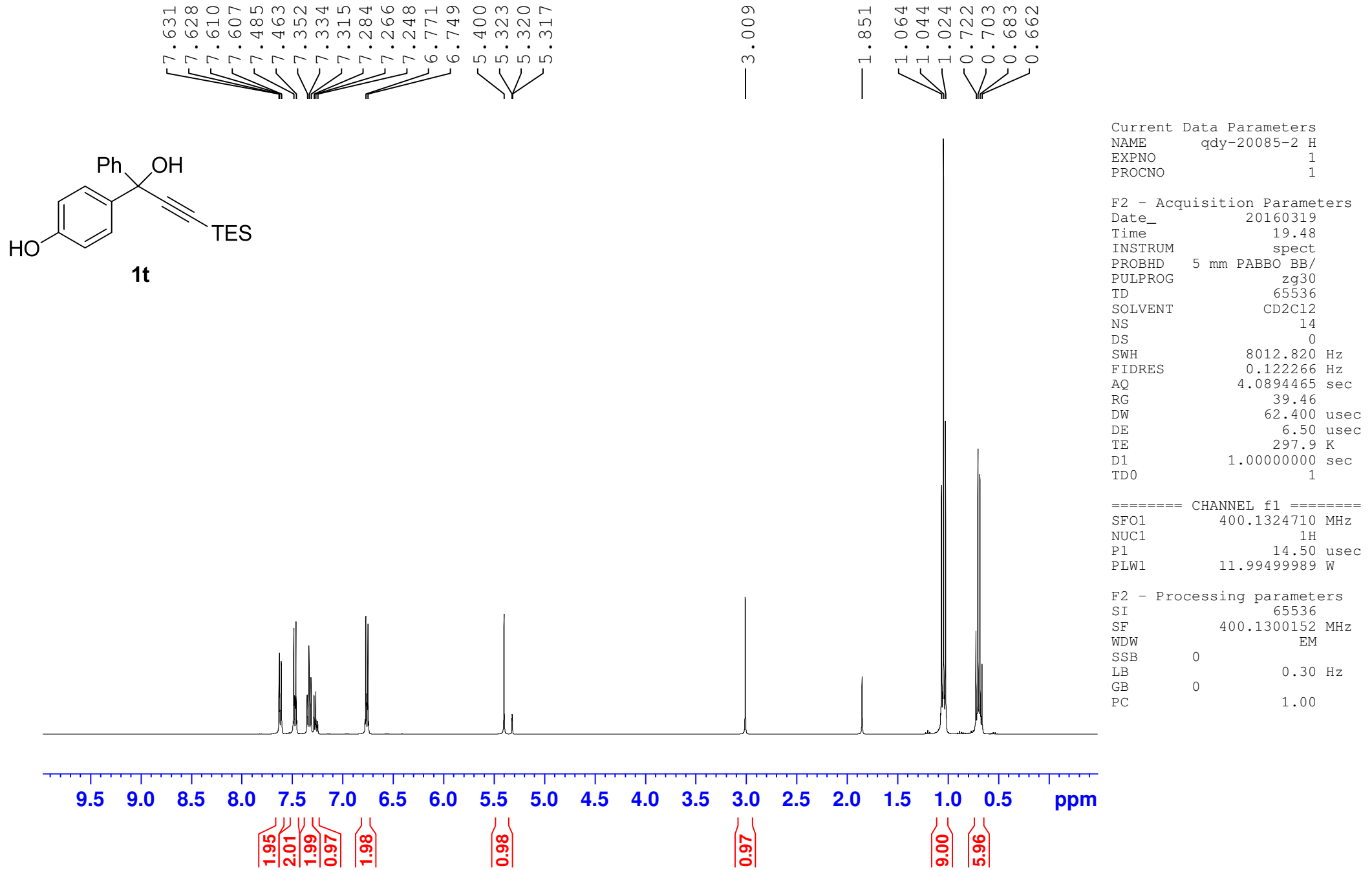
F2 - Acquisition Parameters
Date_ 20160328
Time 19.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 119
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

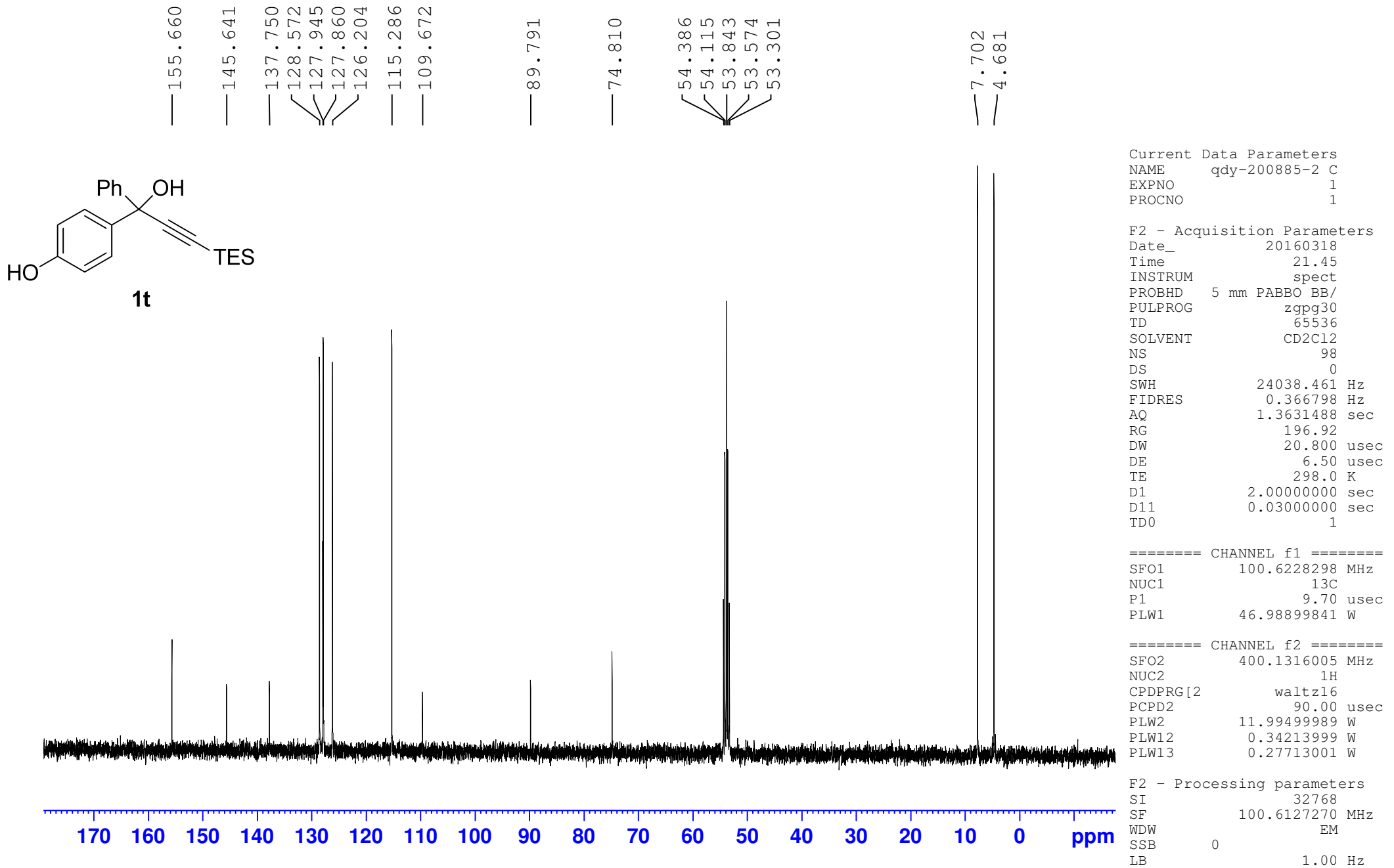
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127064 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 42. ^{13}C NMR spectrum for 1s



Supplementary Figure 43. ^{1}H NMR spectrum for 1t



Current Data Parameters
NAME qdy-200885-2 C
EXPNO 1
PROCNO 1

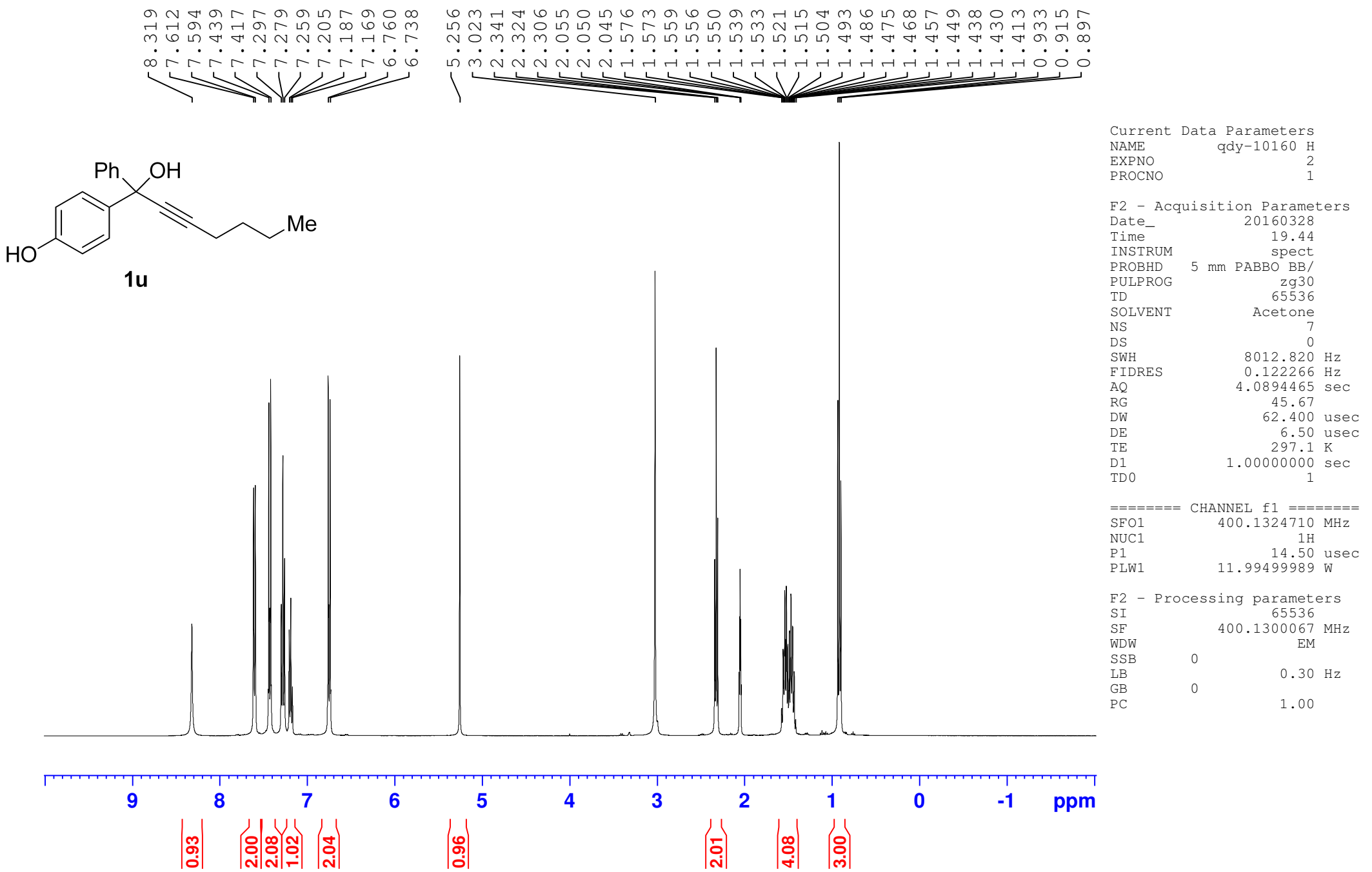
F2 - Acquisition Parameters
Date_ 20160318
Time 21.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 98
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

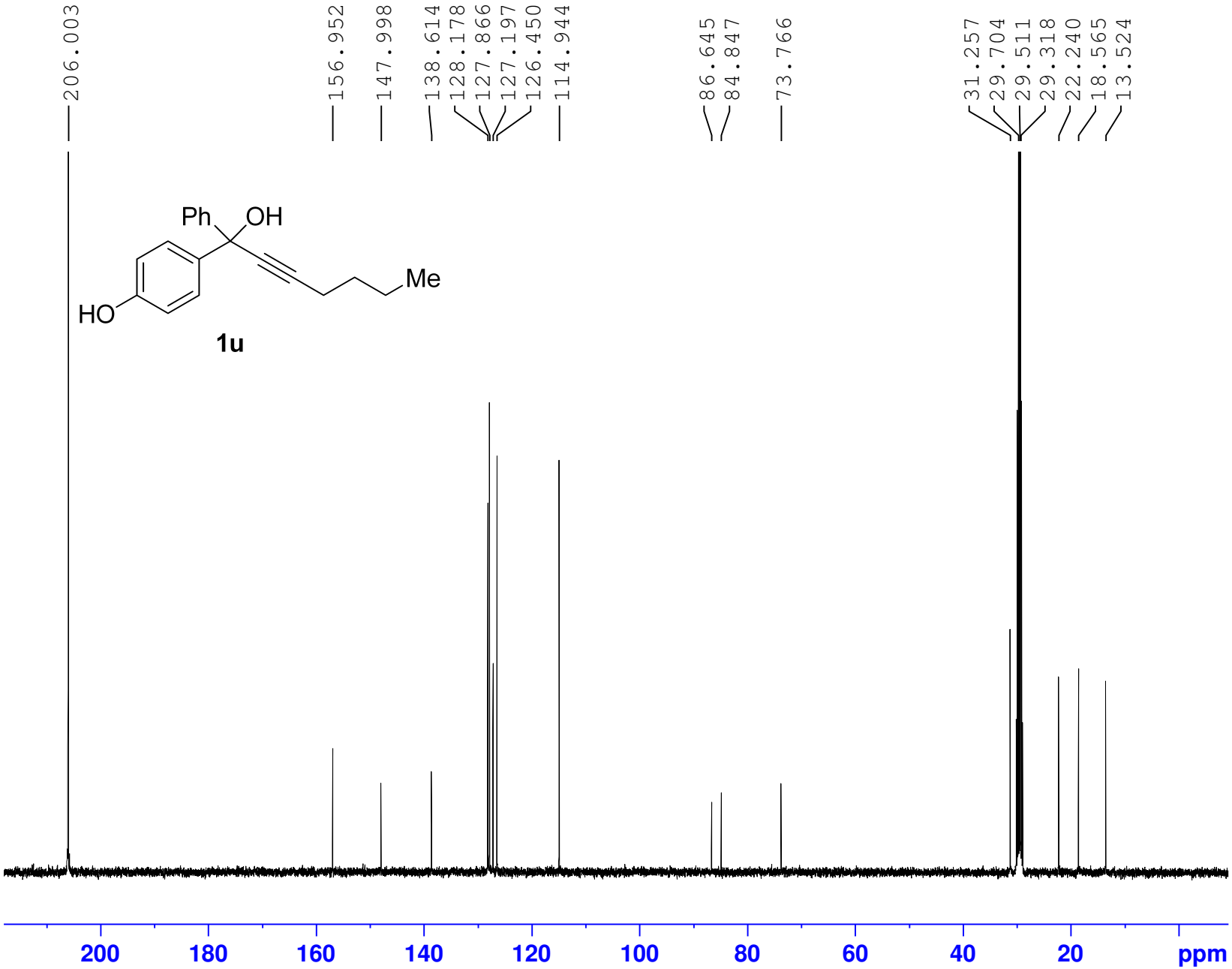
F2 - Processing parameters
SI 32768
SF 100.6127270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 44. ^{13}C NMR spectrum for 1t



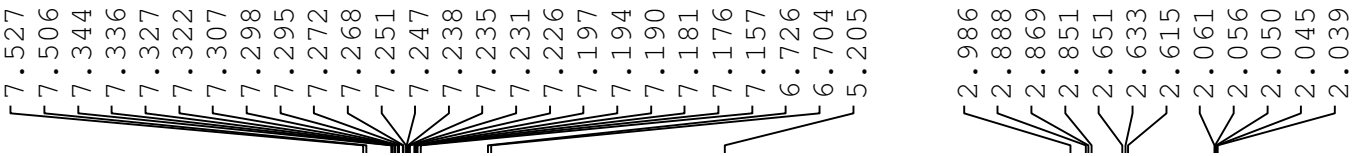

```
Current Data Parameters
NAME         qdy-10160 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               19.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     7
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 45.67
DW                62.400 usec
DE                 6.50 usec
TE                297.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300067 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

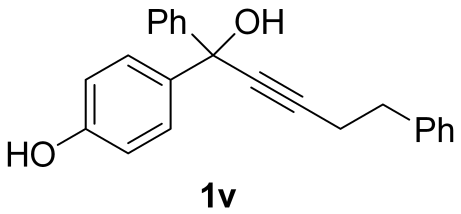
Supplementary Figure 45. ^{1}H NMR spectrum for 1u



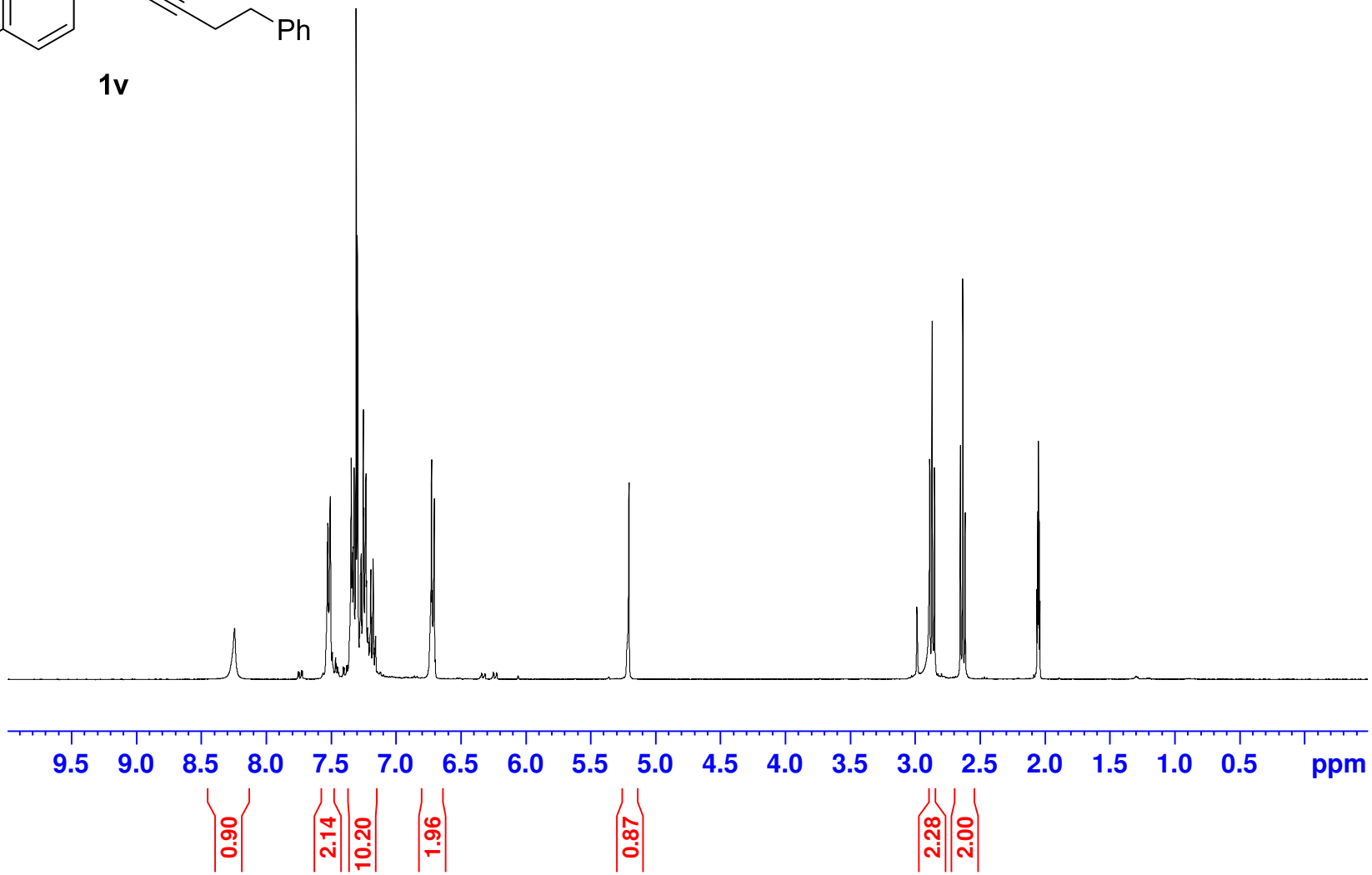

```
Current Data Parameters
NAME         qdy-10160 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               19.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   132
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                  9.70 usec
PLW1          46.98899841 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          11.99499989 W
PLW12          0.34213999 W
PLW13          0.27713001 W

F2 - Processing parameters
SI                 32768
SF            100.6127155 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 46. ^{13}C NMR spectrum for 1u



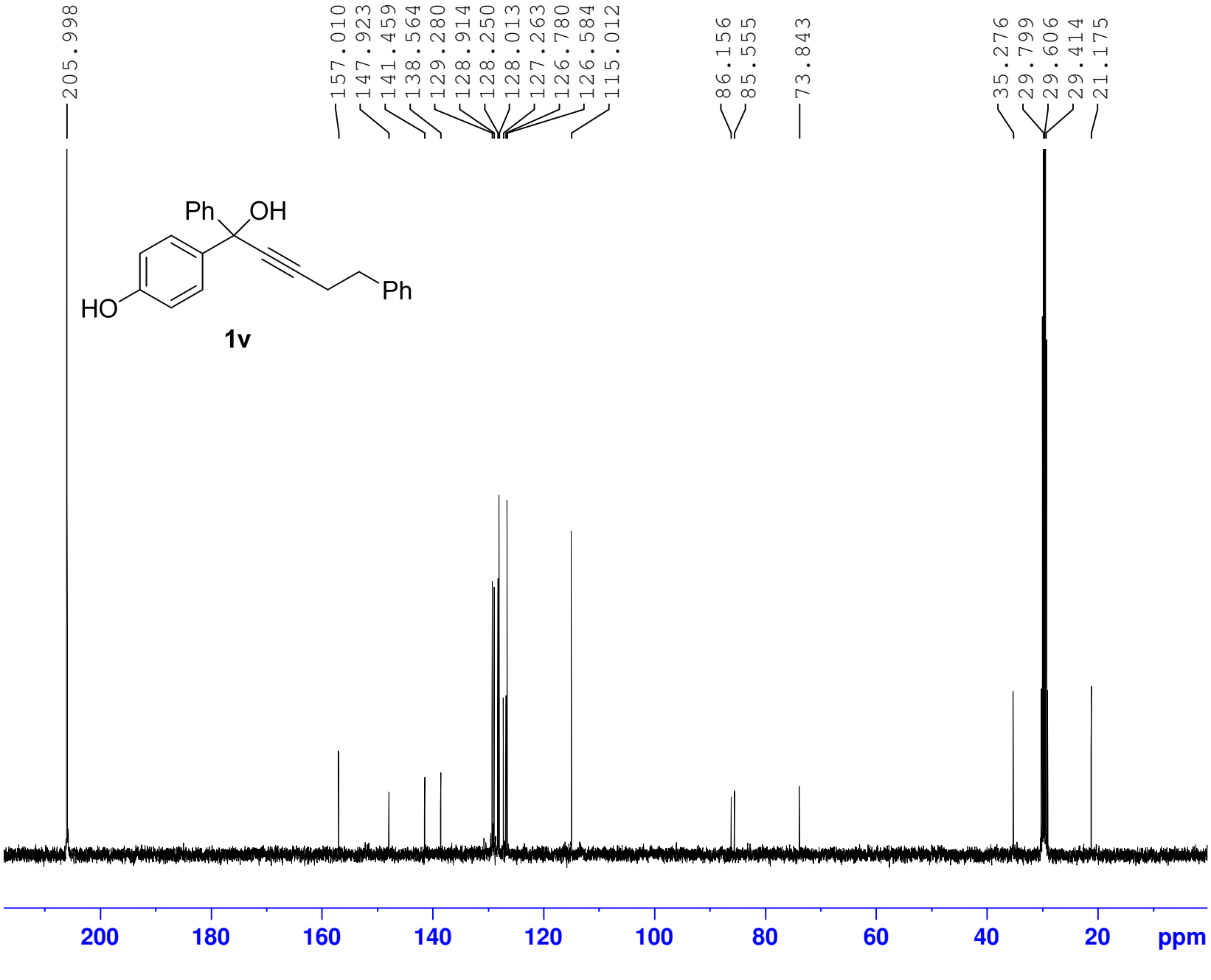


1v



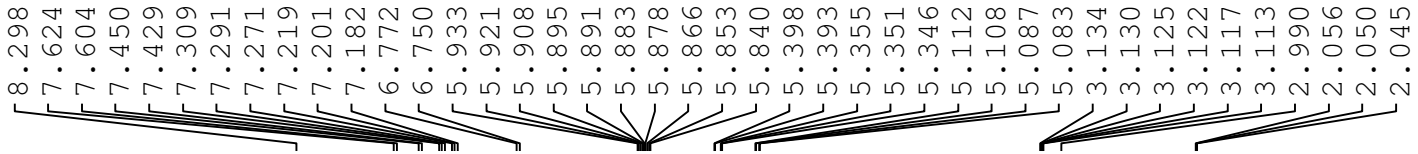

```
Current Data Parameters
NAME         qdy-20116 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160418
Time              21.43
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                103.52
DW                62.400 usec
DE                 6.50 usec
TE                296.9 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300067 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

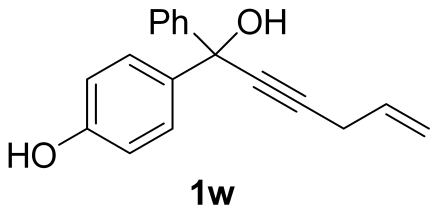
Supplementary Figure 47. ^{1}H NMR spectrum for 1v



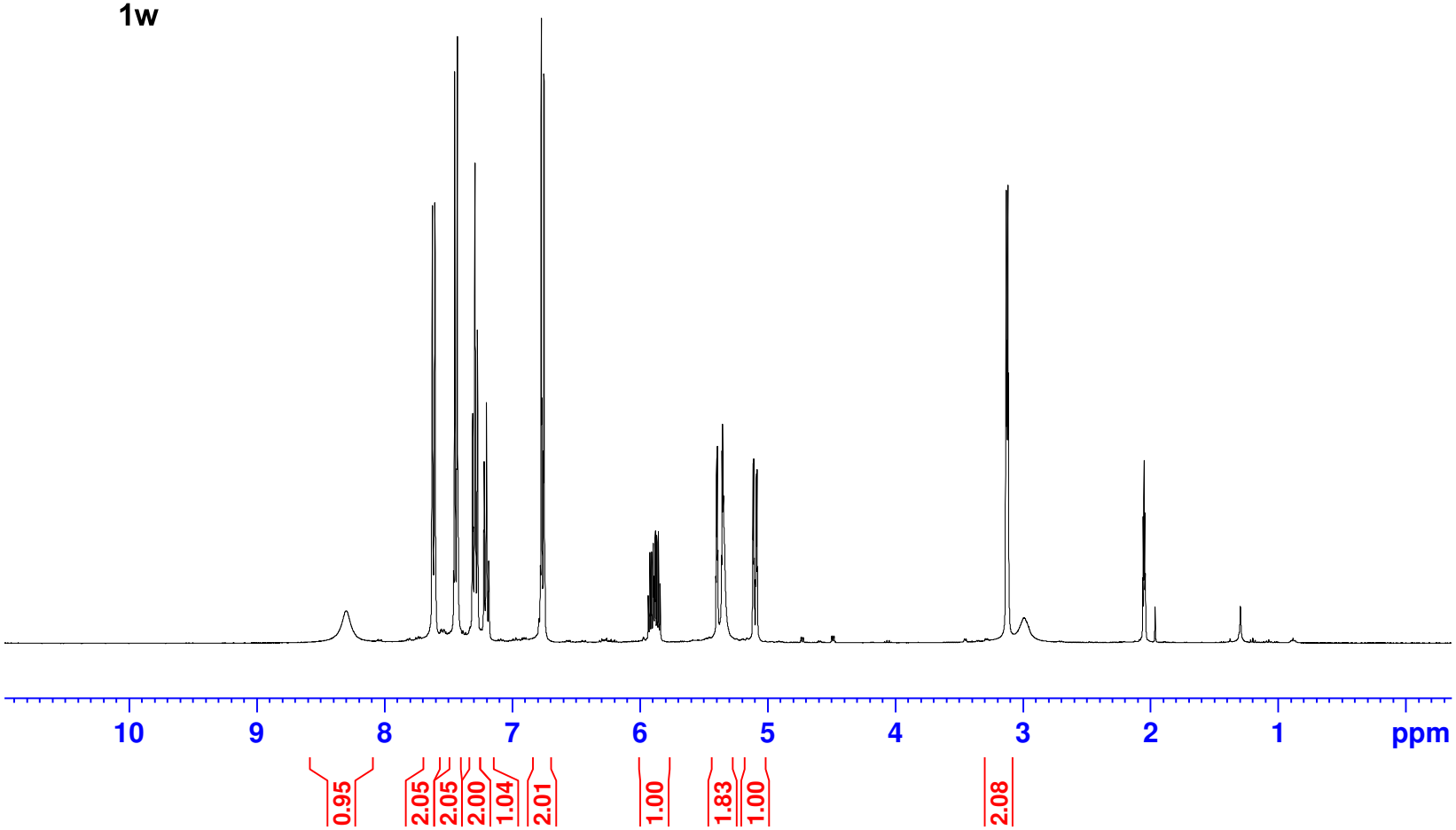

```
Current Data Parameters
NAME         qdy-20116 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160418
Time               19.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   139
DS                     0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                 32768
SF          100.6127051 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 48. ^{13}C NMR spectrum for 1v



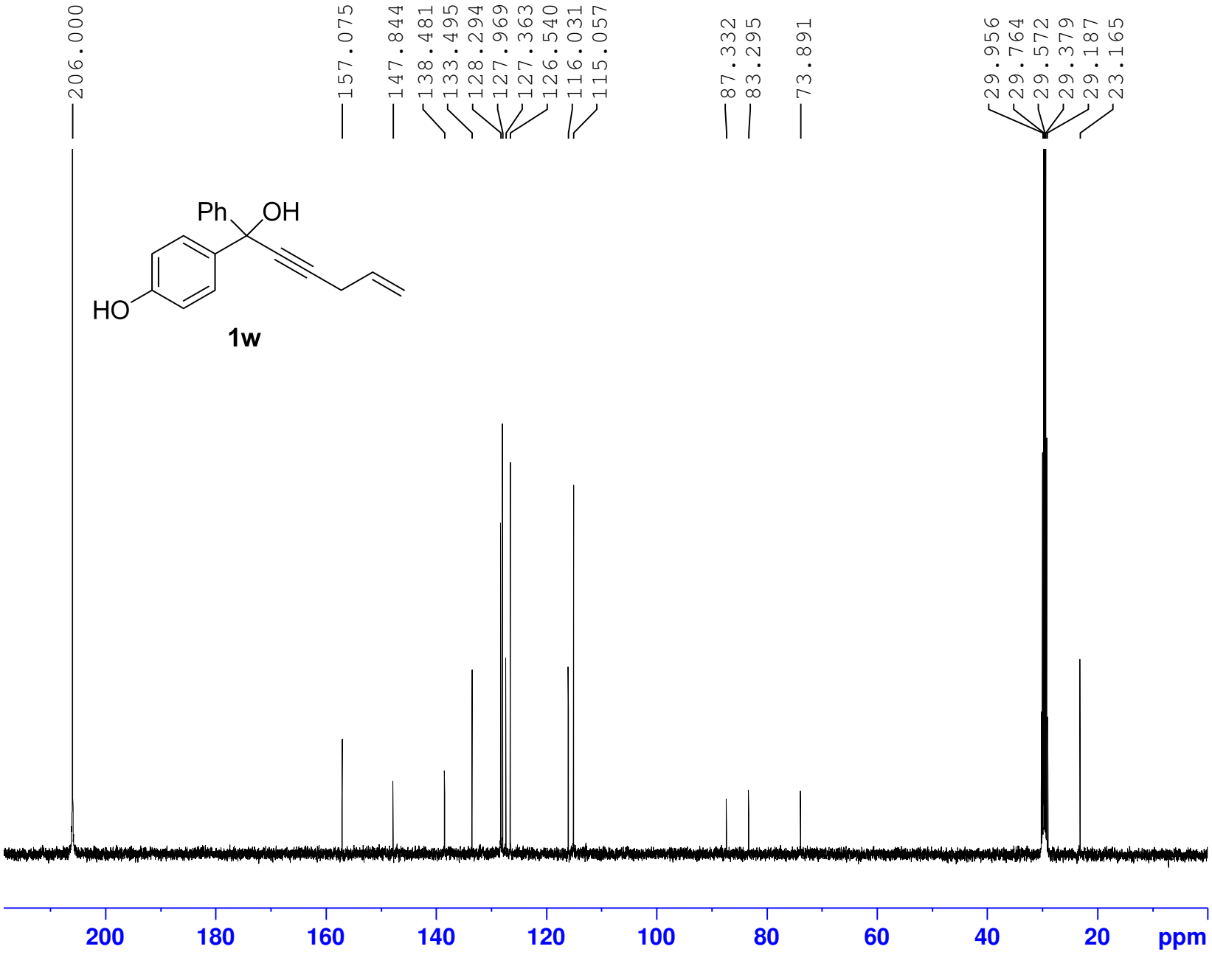


1w



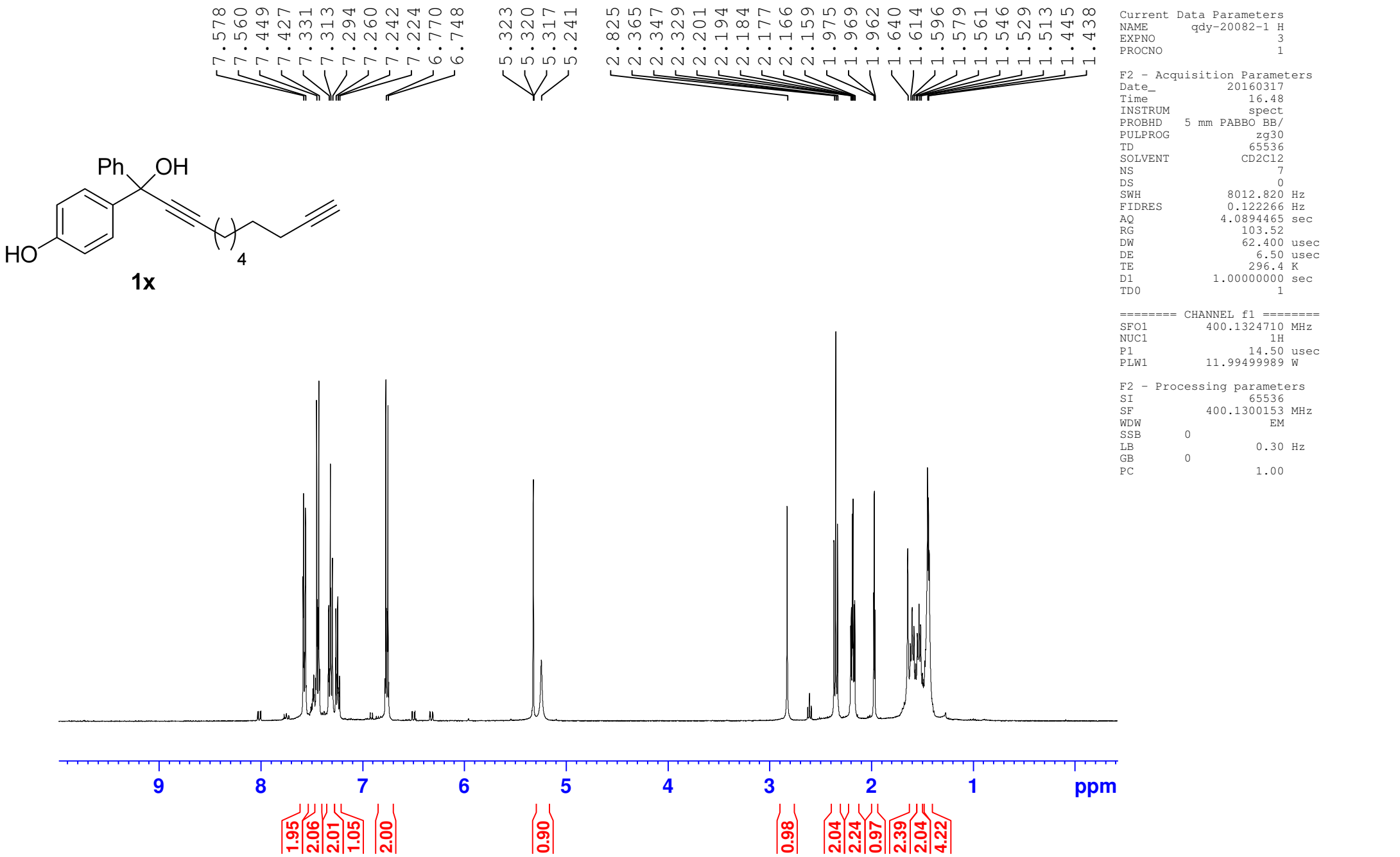

```
Current Data Parameters
NAME       qdy-20114-c H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160416
Time               19.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     5
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 54.81
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300066 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 49. ^{1}H NMR spectrum for 1w

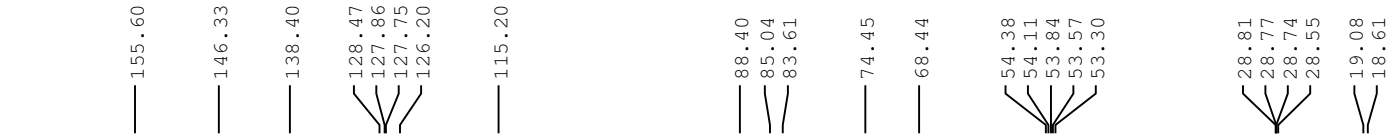


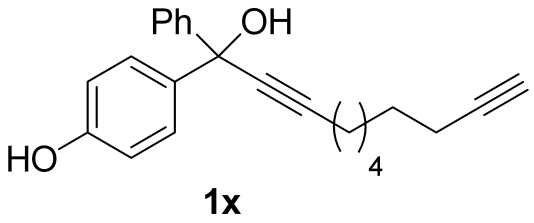

```
Current Data Parameters
NAME      qdy-20114-c C
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20160416
Time              19.20
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                   86
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127088 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 50. ^{13}C NMR spectrum for 1w



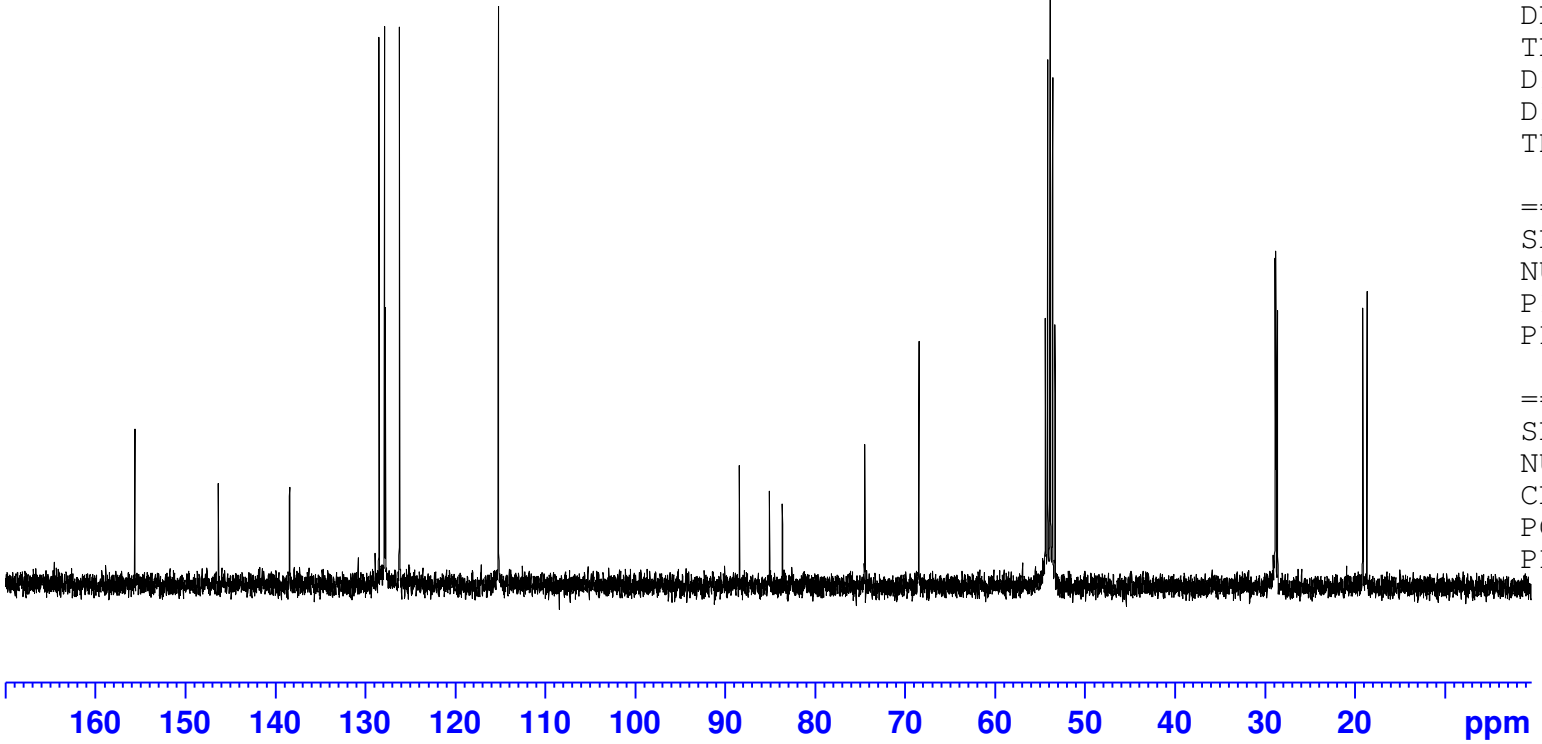
Supplementary Figure 51. ^{1}H NMR spectrum for 1x



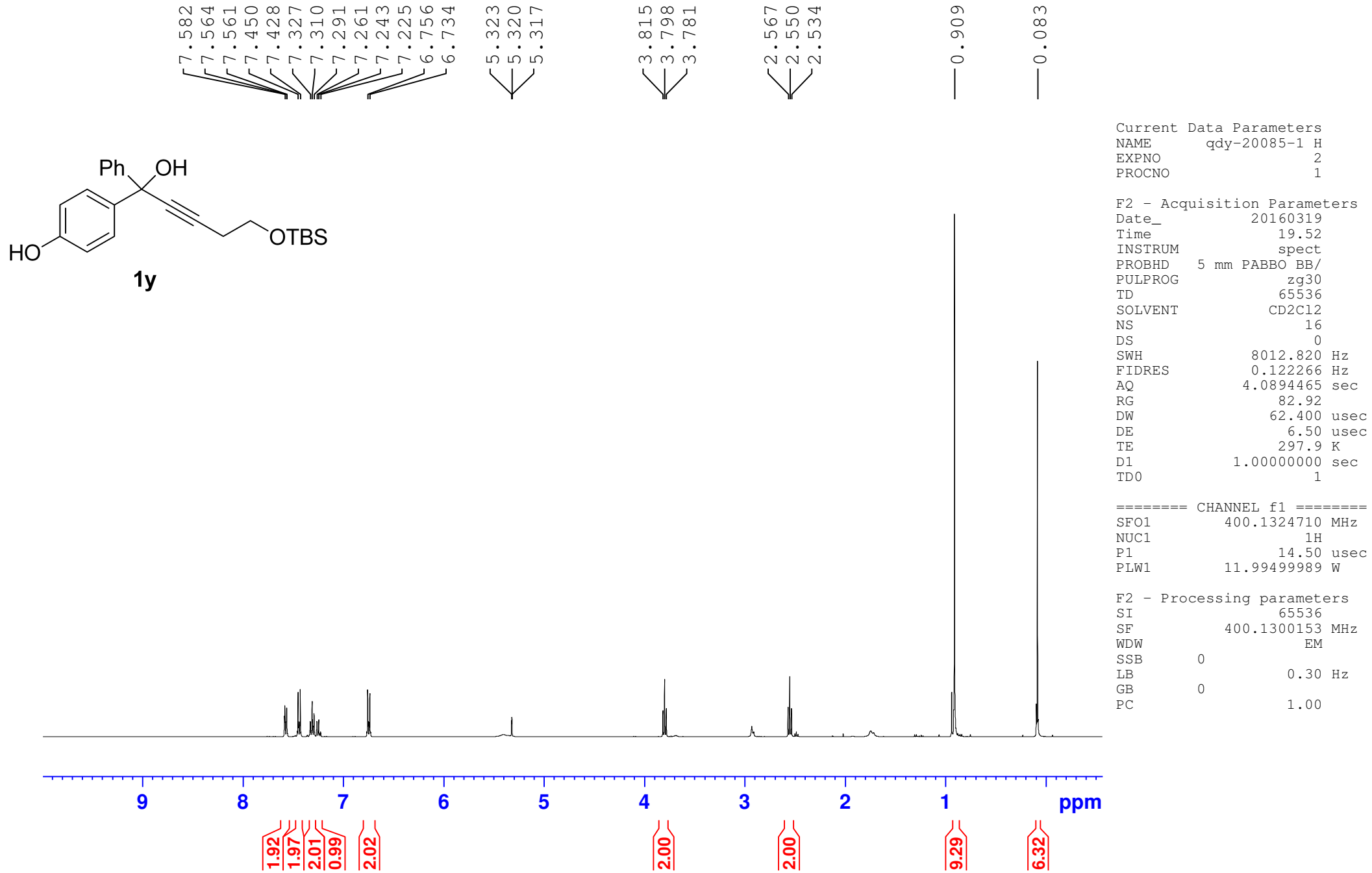


1x

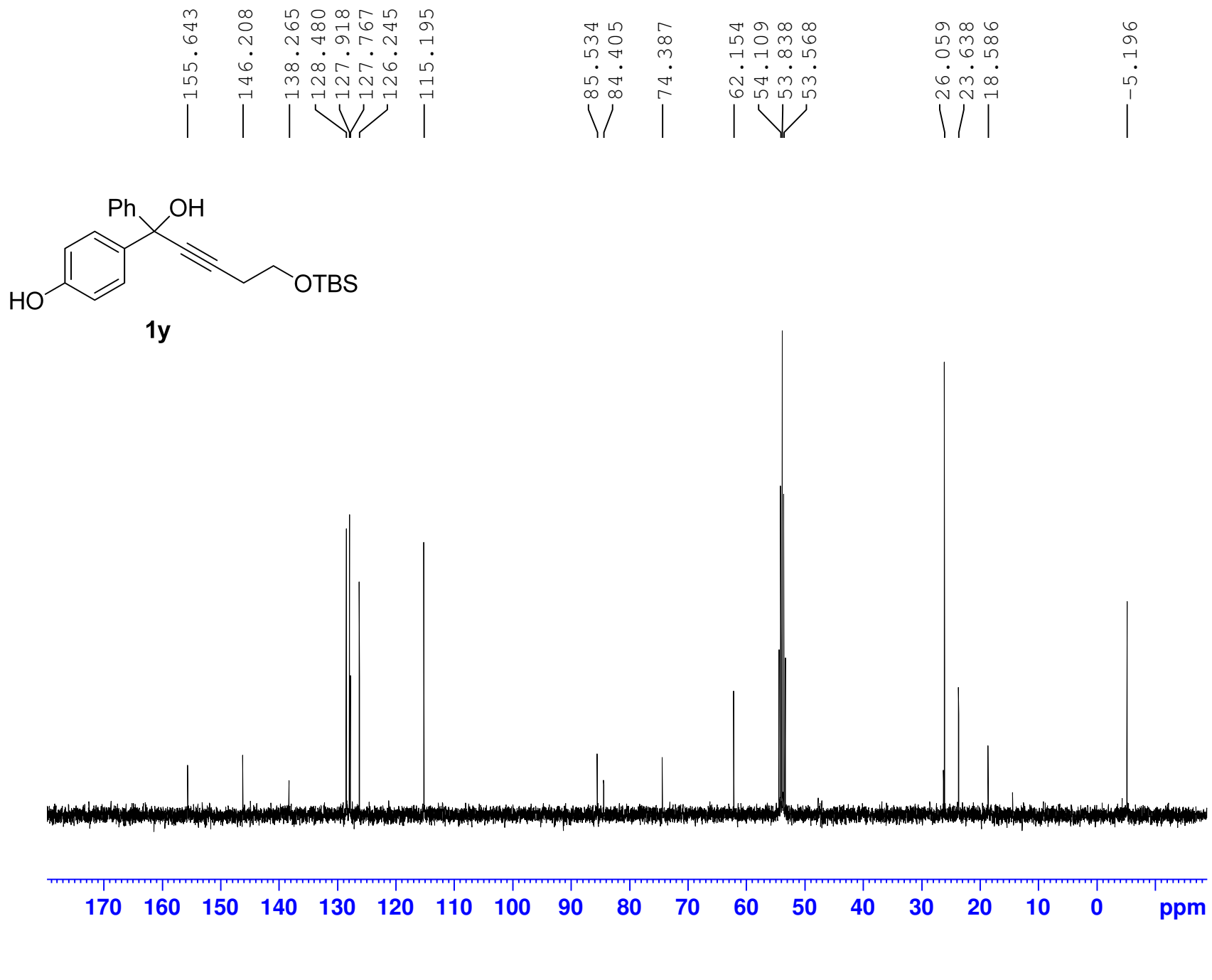
Supplementary Figure 52. ^{13}C NMR spectrum for 1x




```
Current Data Parameters
NAME       qdy-20082-2 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Paramete
Date_           20160316
Time               21.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          CD2Cl2
NS                   116
DS                     0
SWH            24038.461 H
FIDRES          0.366798 H
AQ              1.3631488 s
RG                 196.92
DW                 20.800 u
DE                  6.50 u
TE                 297.5 K
D1            2.00000000 s
D11           0.03000000 s
TD0                    1

======== CHANNEL f1 =====
SFO1         100.6228298 M
NUC1                13C
P1                 9.70 u
PLW1         46.98899841 W

======== CHANNEL f2 =====
SFO2         400.1316005 M
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 u
PLW2         11.99499989 W
```

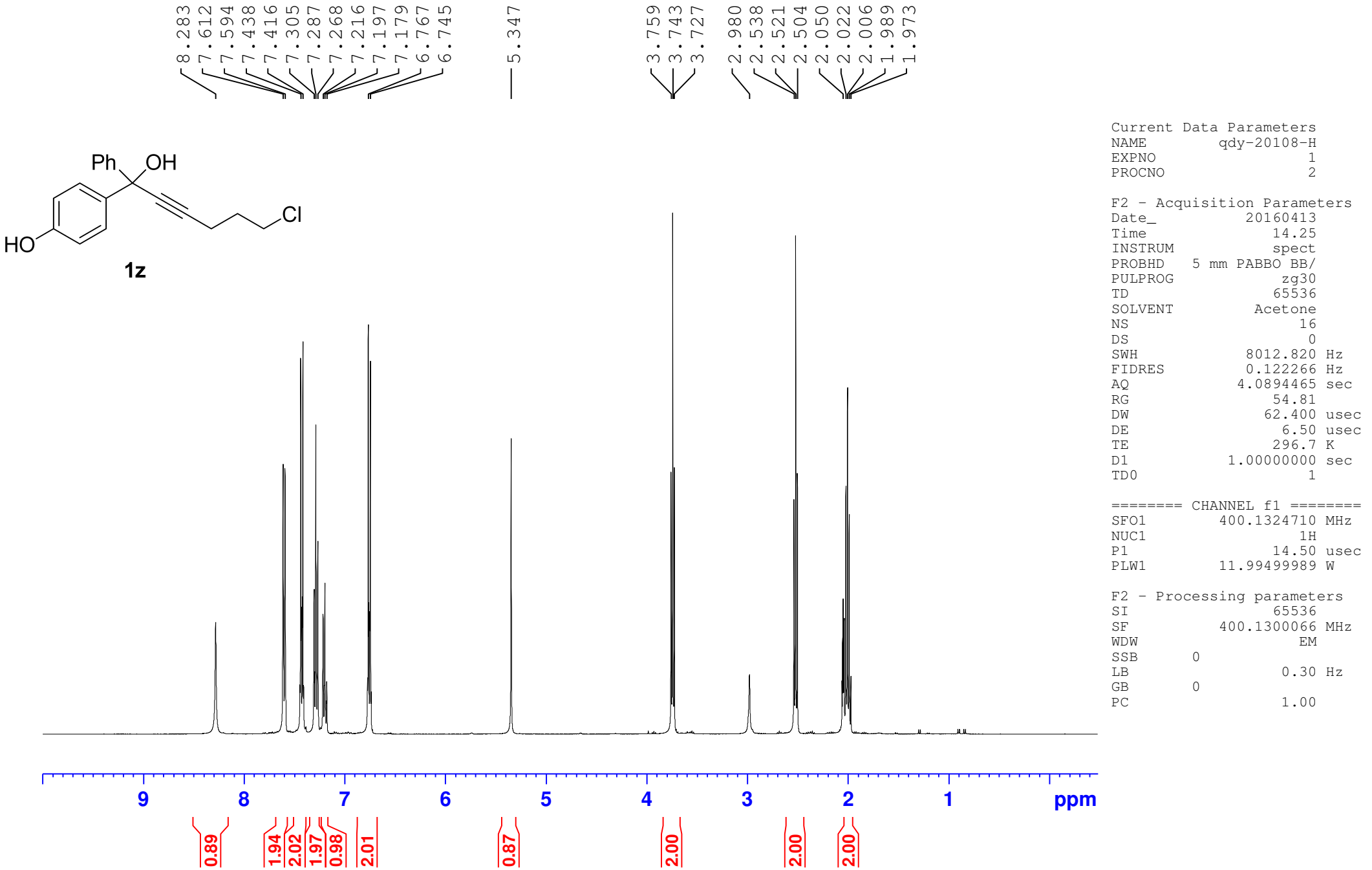


Supplementary Figure 53. ^{1}H NMR spectrum for 1y




```
Current Data Parameters
NAME       qdy-20085-1 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160317
Time               16.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                    74
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127277 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 54. ^{13}C NMR spectrum for 1y



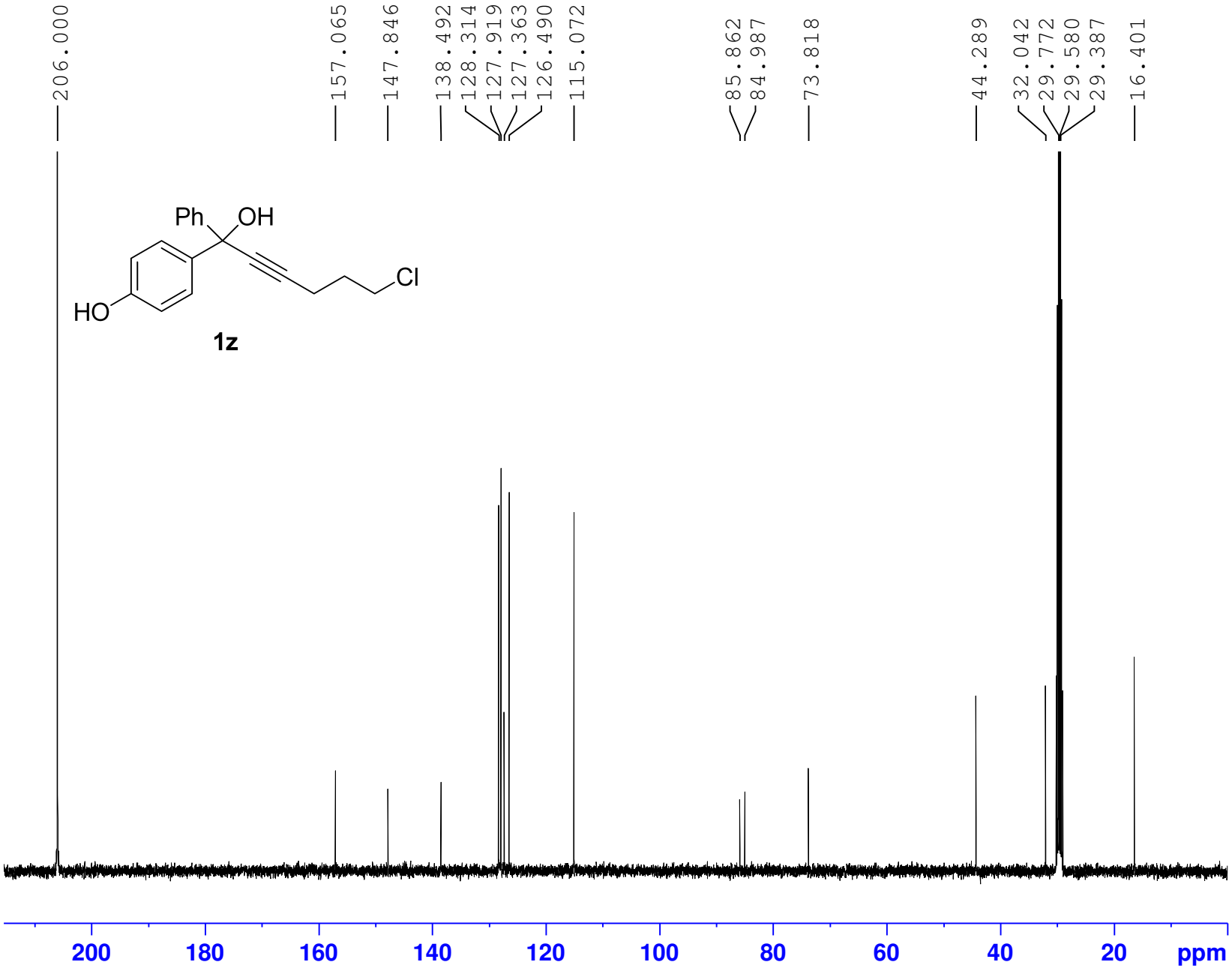
Current Data Parameters
NAME qdy-20108-H
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters
Date_ 20160413
Time 14.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

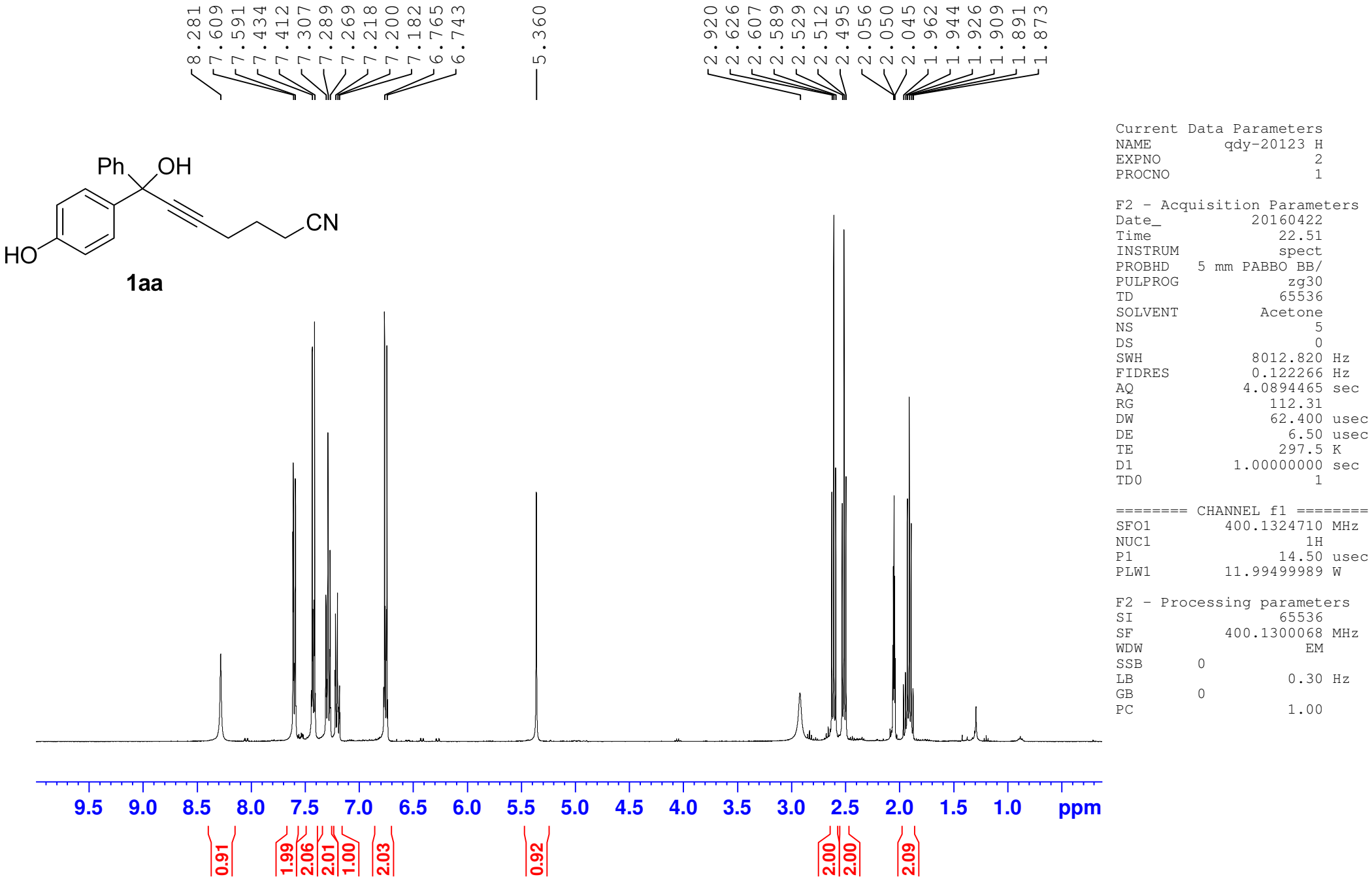
F2 - Processing parameters
SI 65536
SF 400.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 55. ^{1}H NMR spectrum for 1z



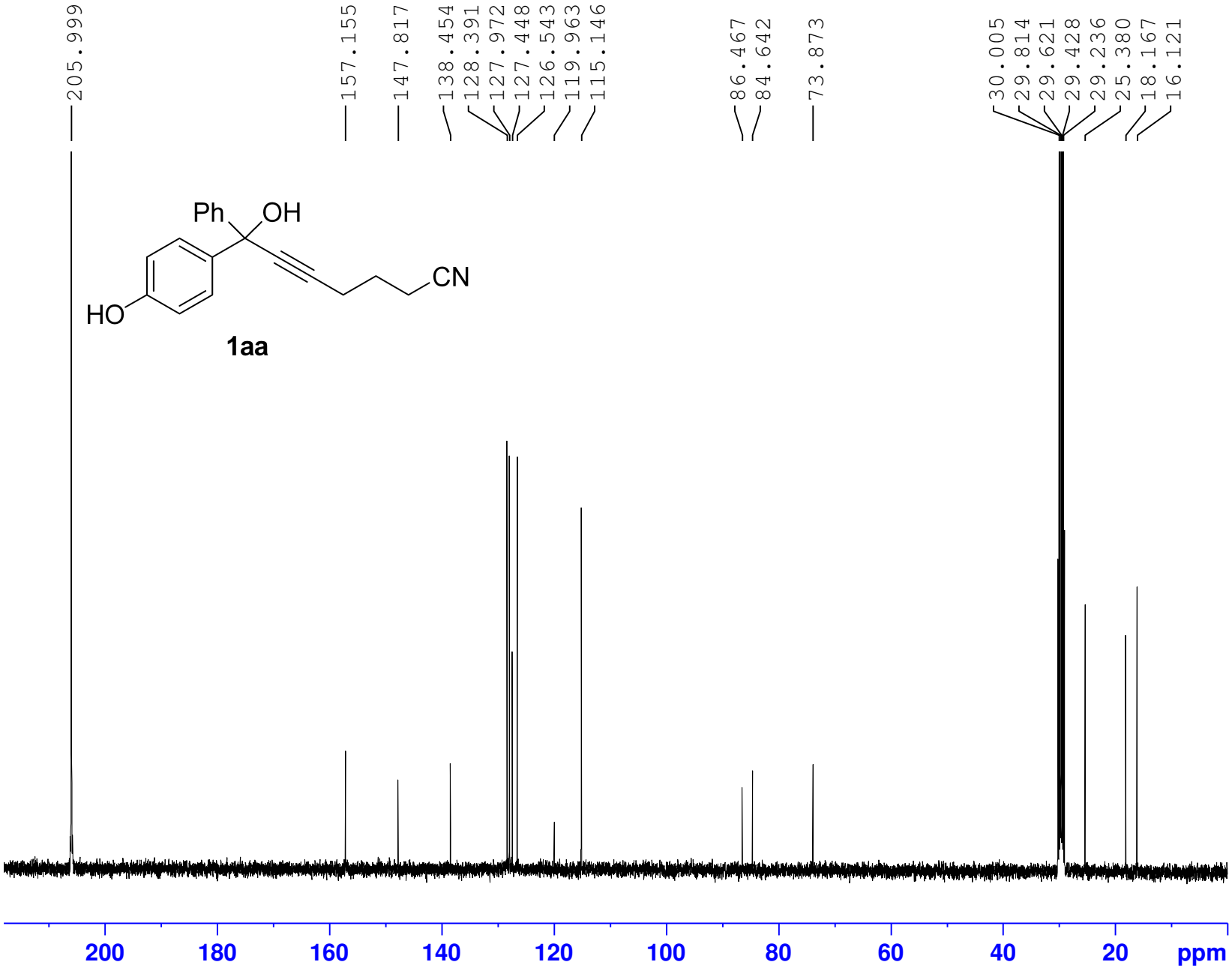

```
Current Data Parameters
NAME         qdy-20108 C
EXPNO                  1
PROCNO                 2

F2 - Acquisition Parameters
Date_           20160412
Time               21.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                    91
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127086 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

Supplementary Figure 56. ^{13}C NMR spectrum for 1z



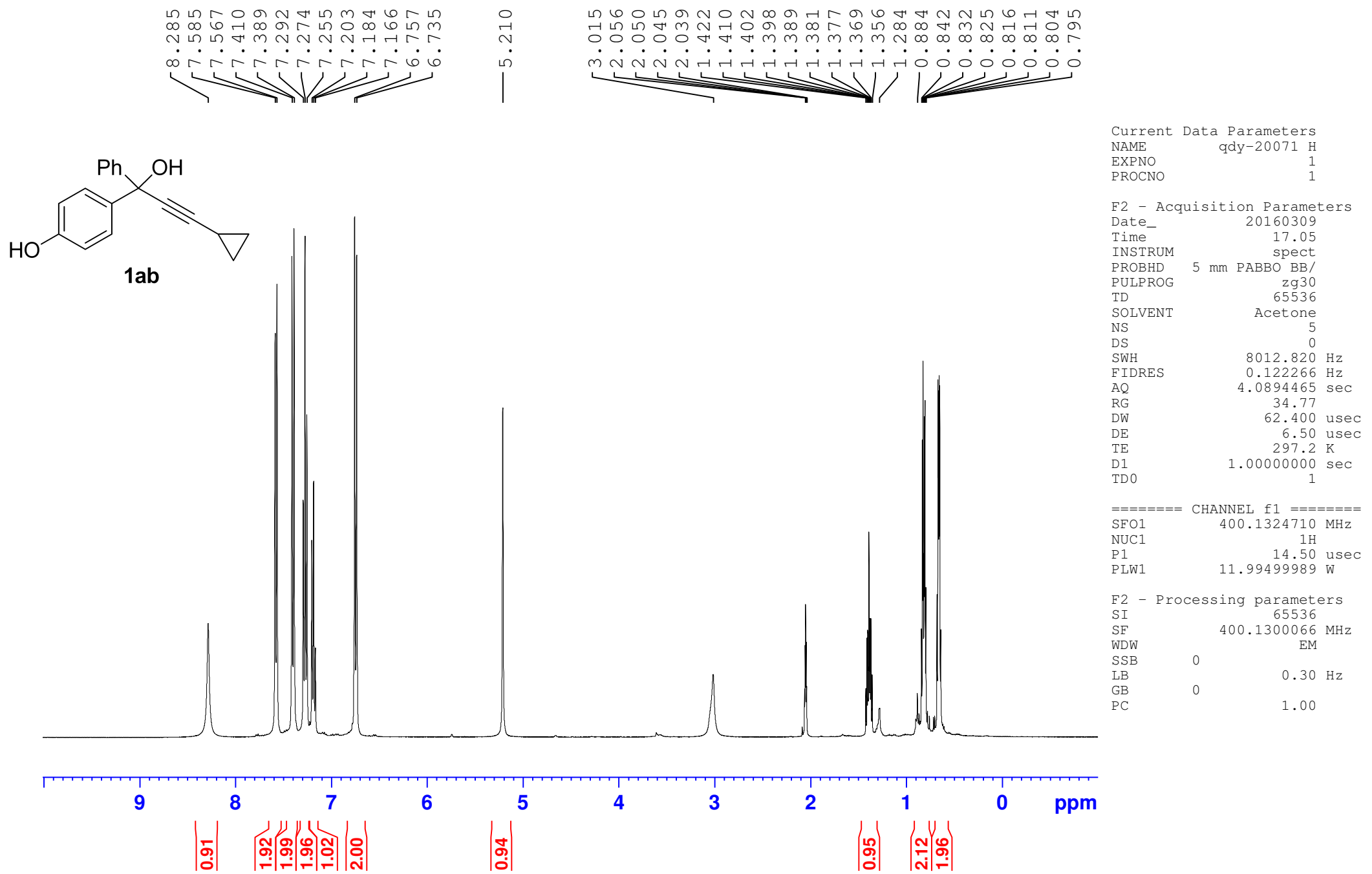

```
Current Data Parameters
NAME         qdy-20123 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160422
Time               22.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     5
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                112.31
DW                62.400 usec
DE                 6.50 usec
TE                297.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300068 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 57. ^{1}H NMR spectrum for 1aa




```
Current Data Parameters
NAME         qdy-20123 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160422
Time               22.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   200
DS                     0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                 32768
SF          100.6127022 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 58. ^{13}C NMR spectrum for 1aa



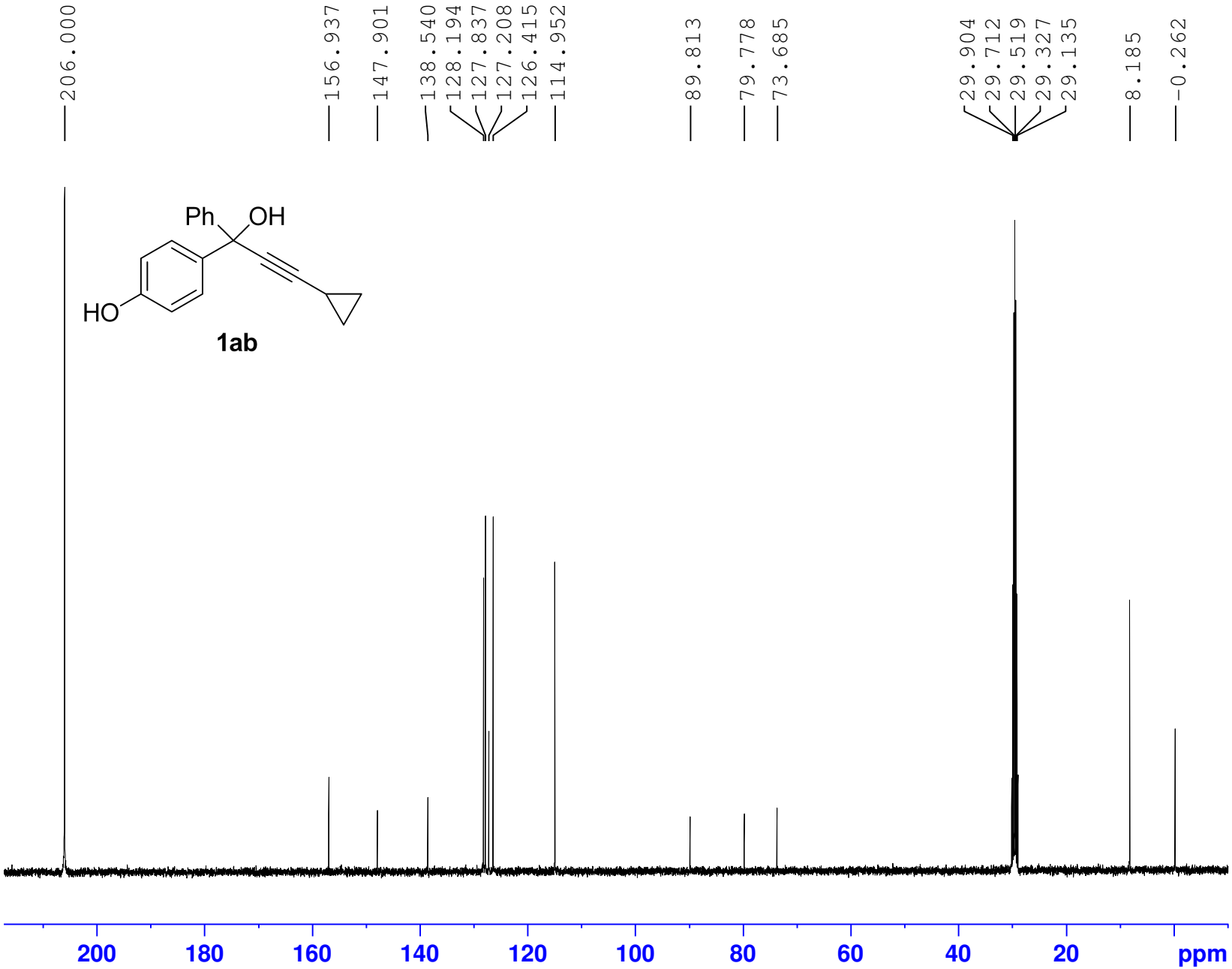
Current Data Parameters
NAME qdy-20071 H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160309
Time 17.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 5
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 34.77
DW 62.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

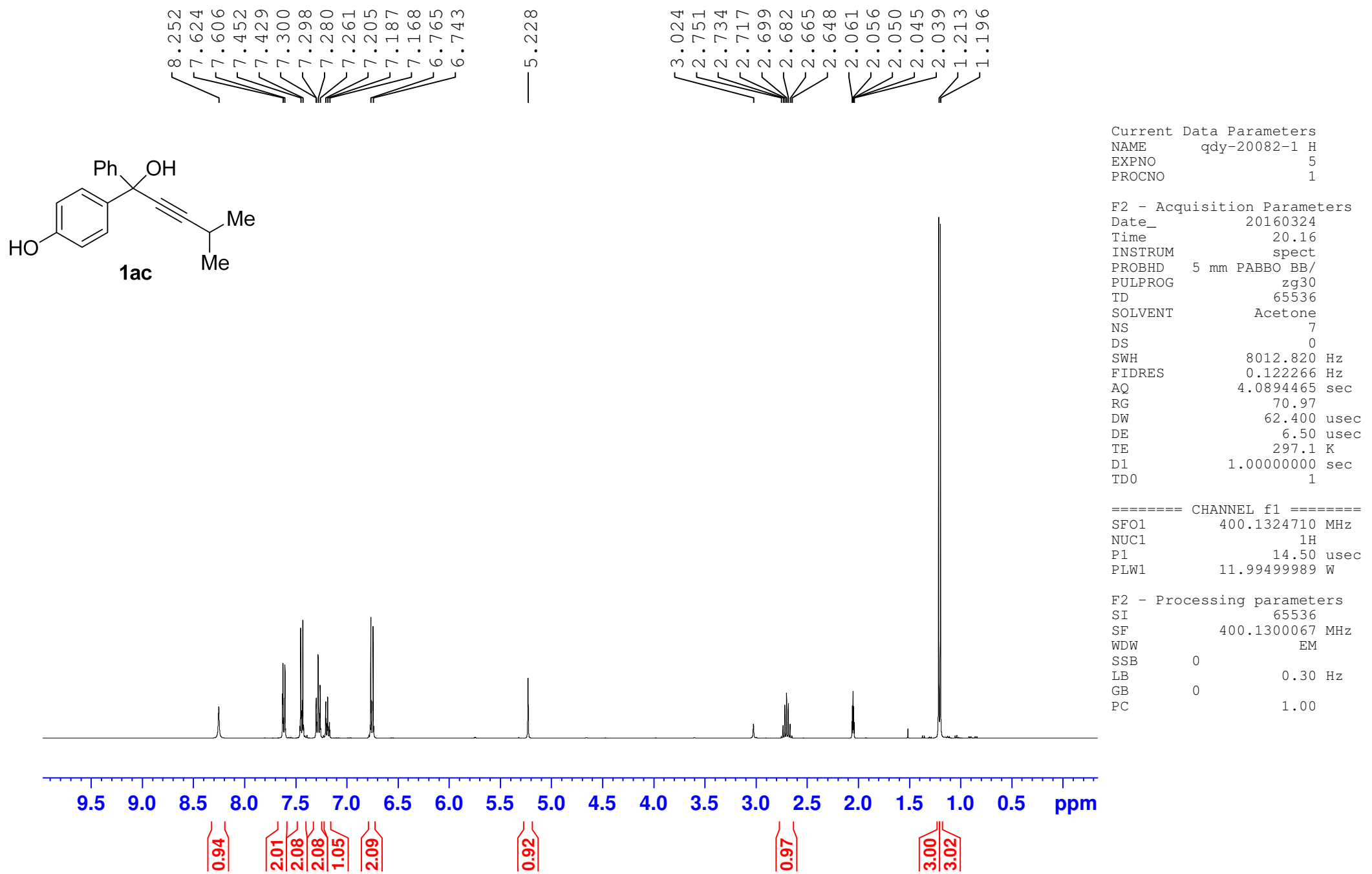
F2 - Processing parameters
SI 65536
SF 400.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 59. ^{1}H NMR spectrum for 1ab



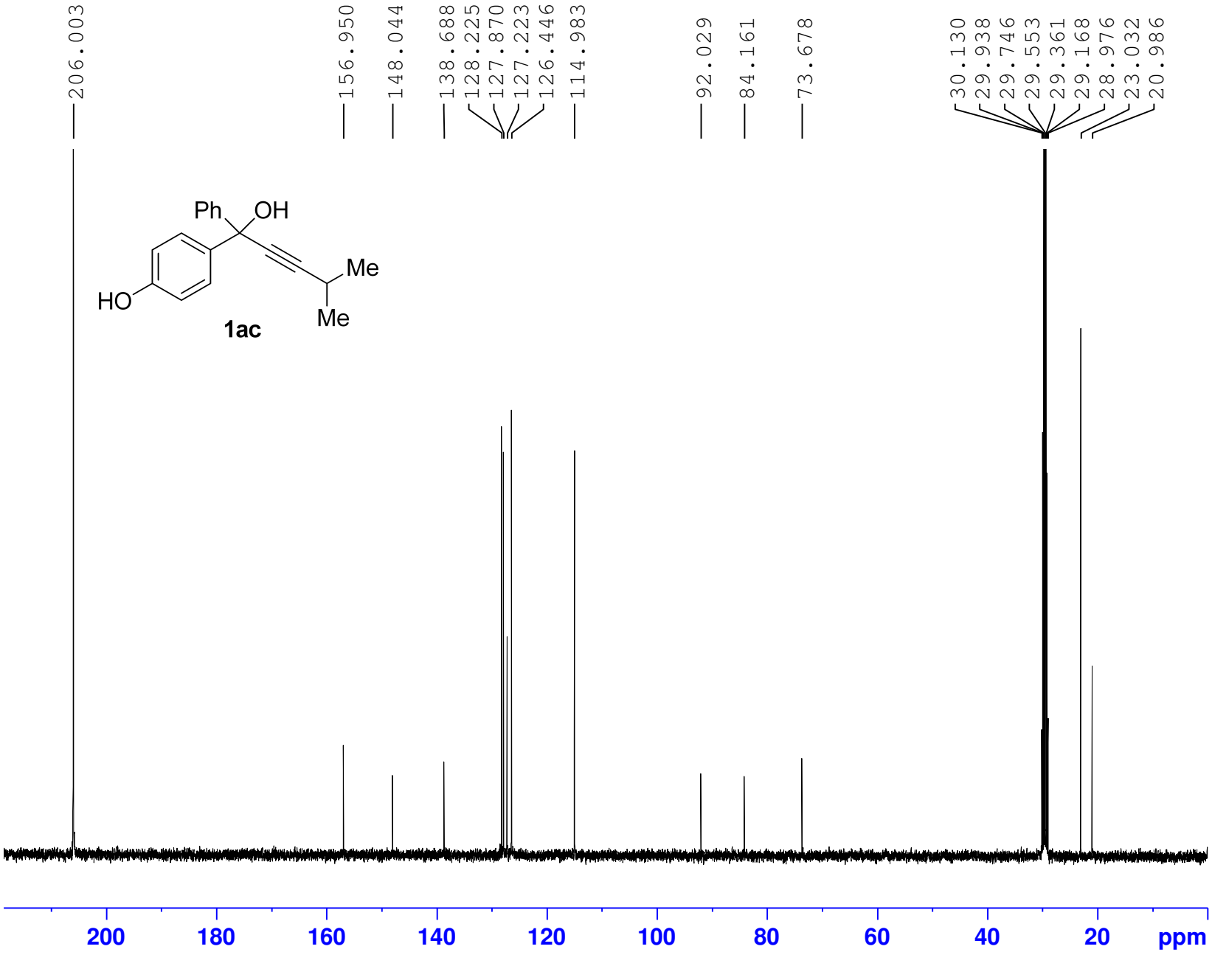

```
Current Data Parameters
NAME         qdy-20071 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160309
Time               17.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                    96
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                  9.70 usec
PLW1          46.98899841 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          11.99499989 W
PLW12          0.34213999 W
PLW13          0.27713001 W

F2 - Processing parameters
SI                 32768
SF            100.6127162 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 60. ^{13}C NMR spectrum for 1ab



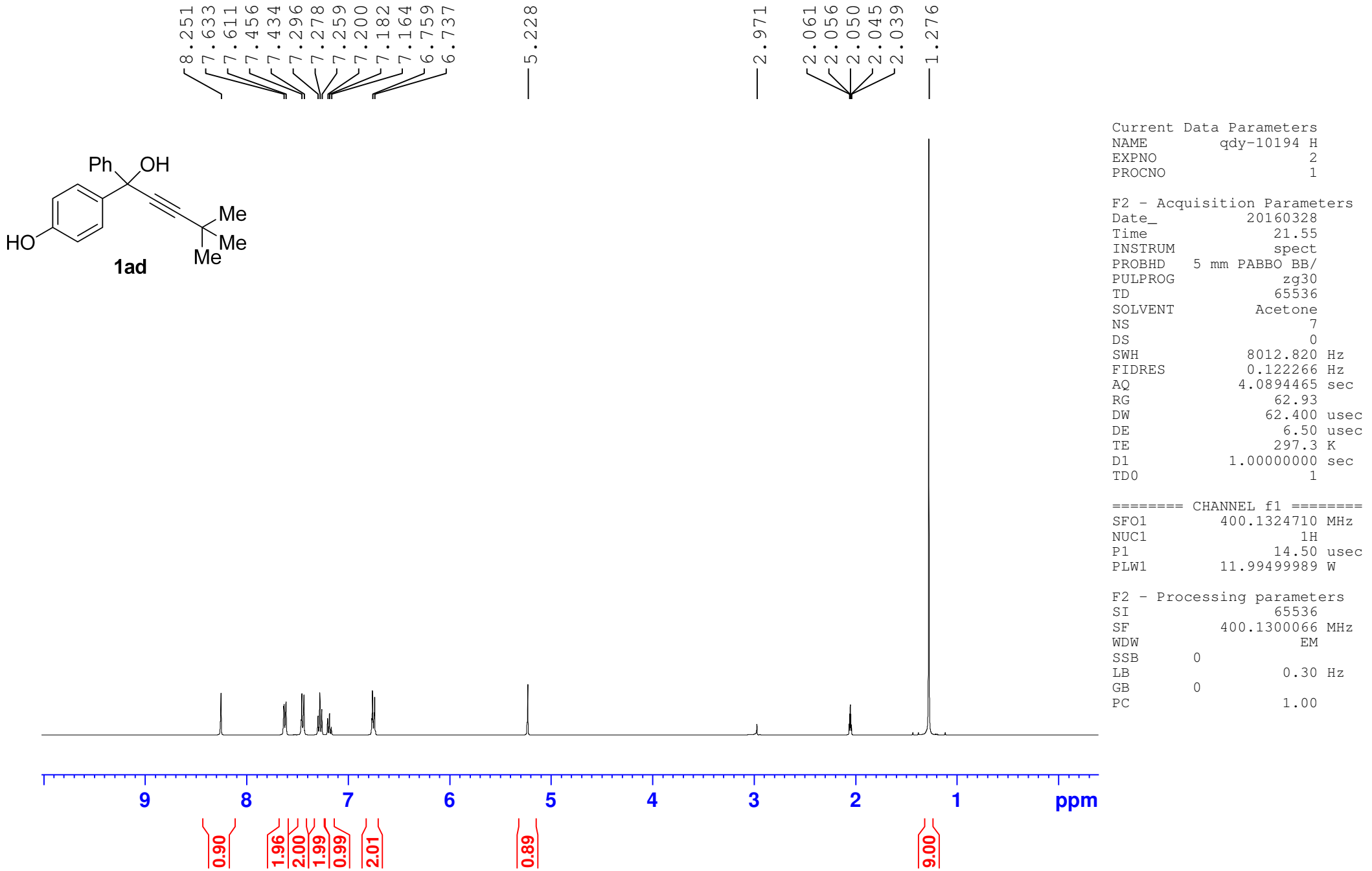

```
Current Data Parameters
NAME       qdy-20082-1 H
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_           20160324
Time              20.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    7
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                70.97
DW               62.400 usec
DE                6.50 usec
TE                297.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                    65536
SF           400.1300067 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 61. ^{1}H NMR spectrum for 1ac

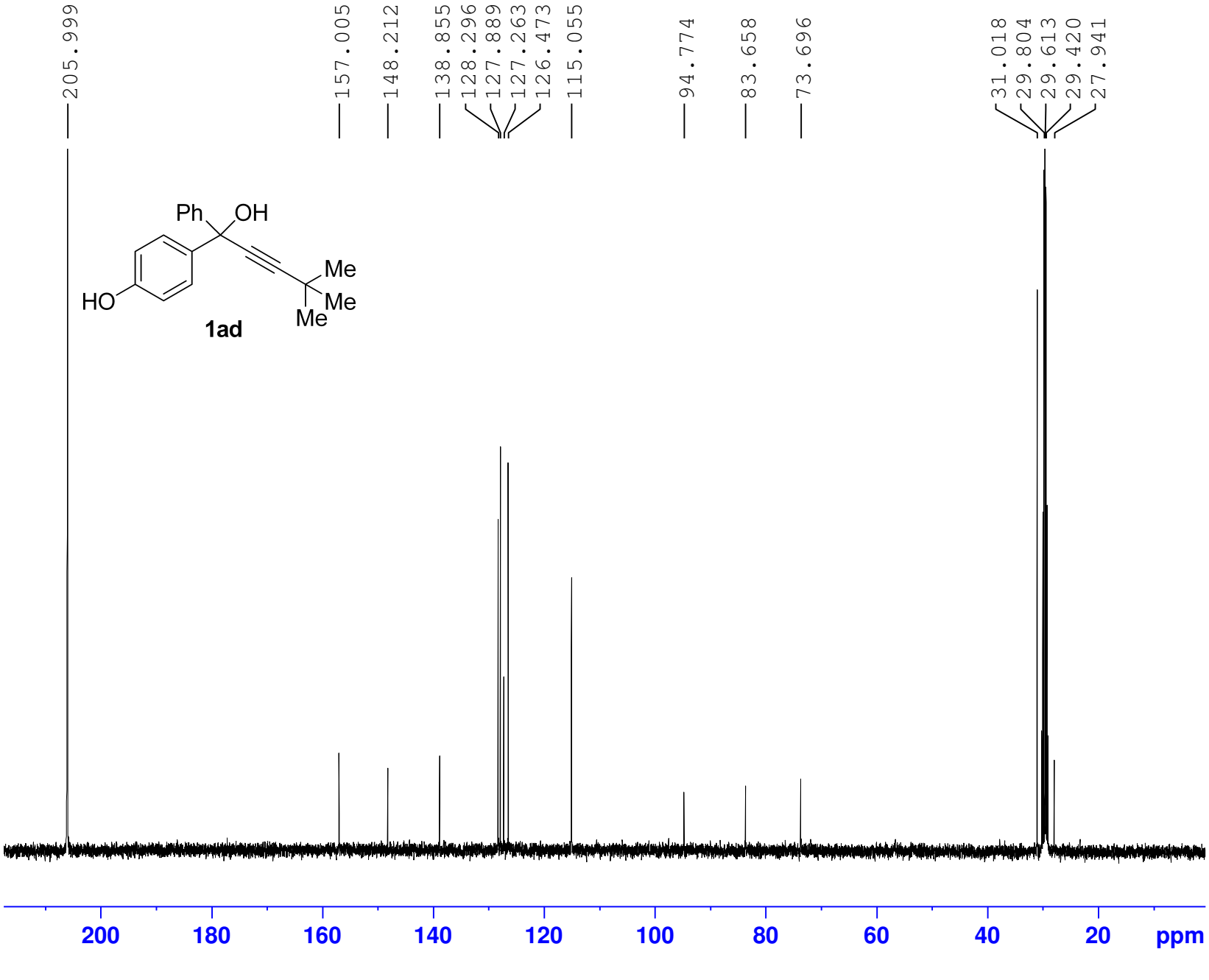



```
Current Data Parameters
NAME      qdy-20082-1 C
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20160324
Time             20.07
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                 108
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE               297.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127125 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

Supplementary Figure 62. ^{13}C NMR spectrum for 1ac

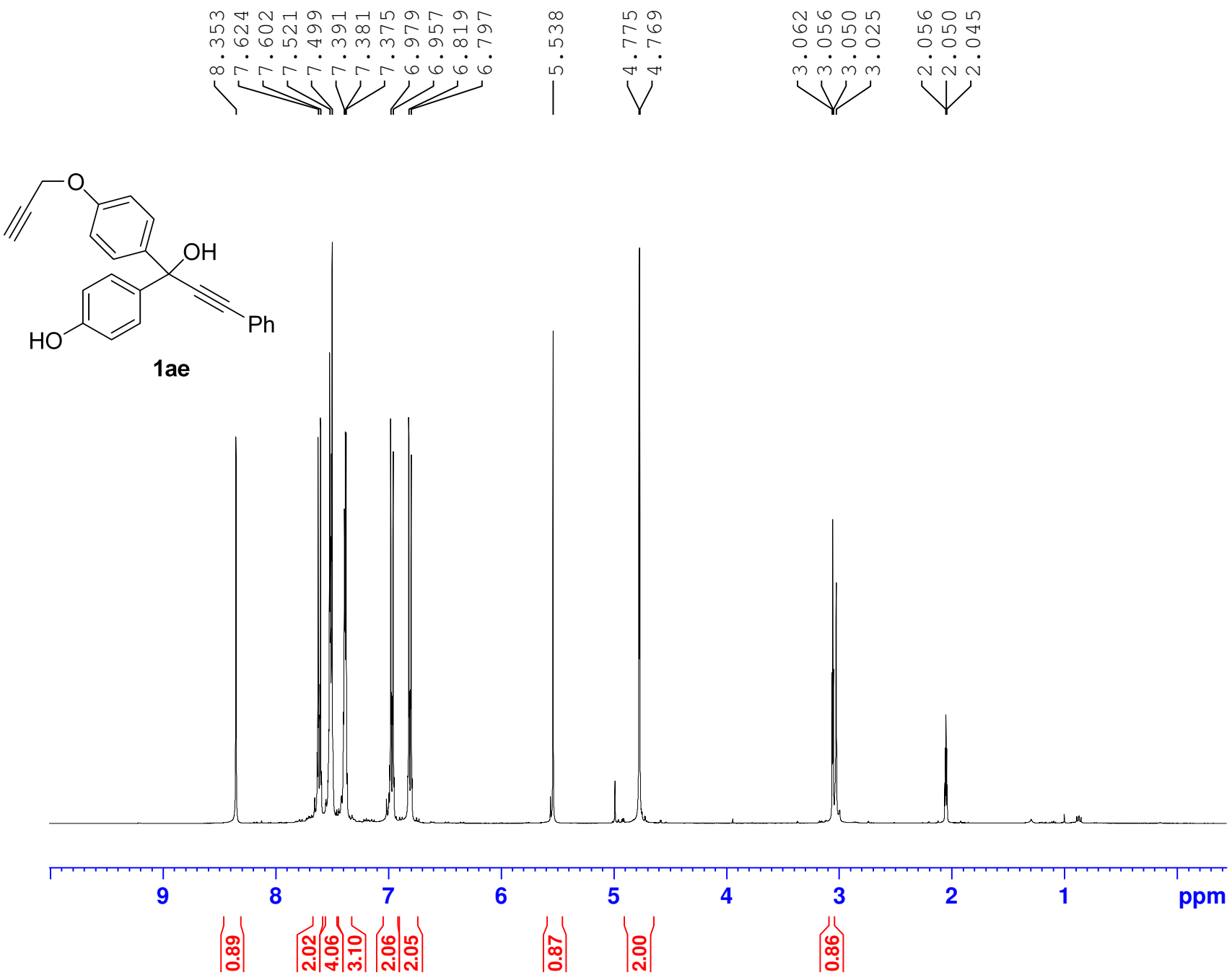


Supplementary Figure 63. ^{1}H NMR spectrum for 1ad



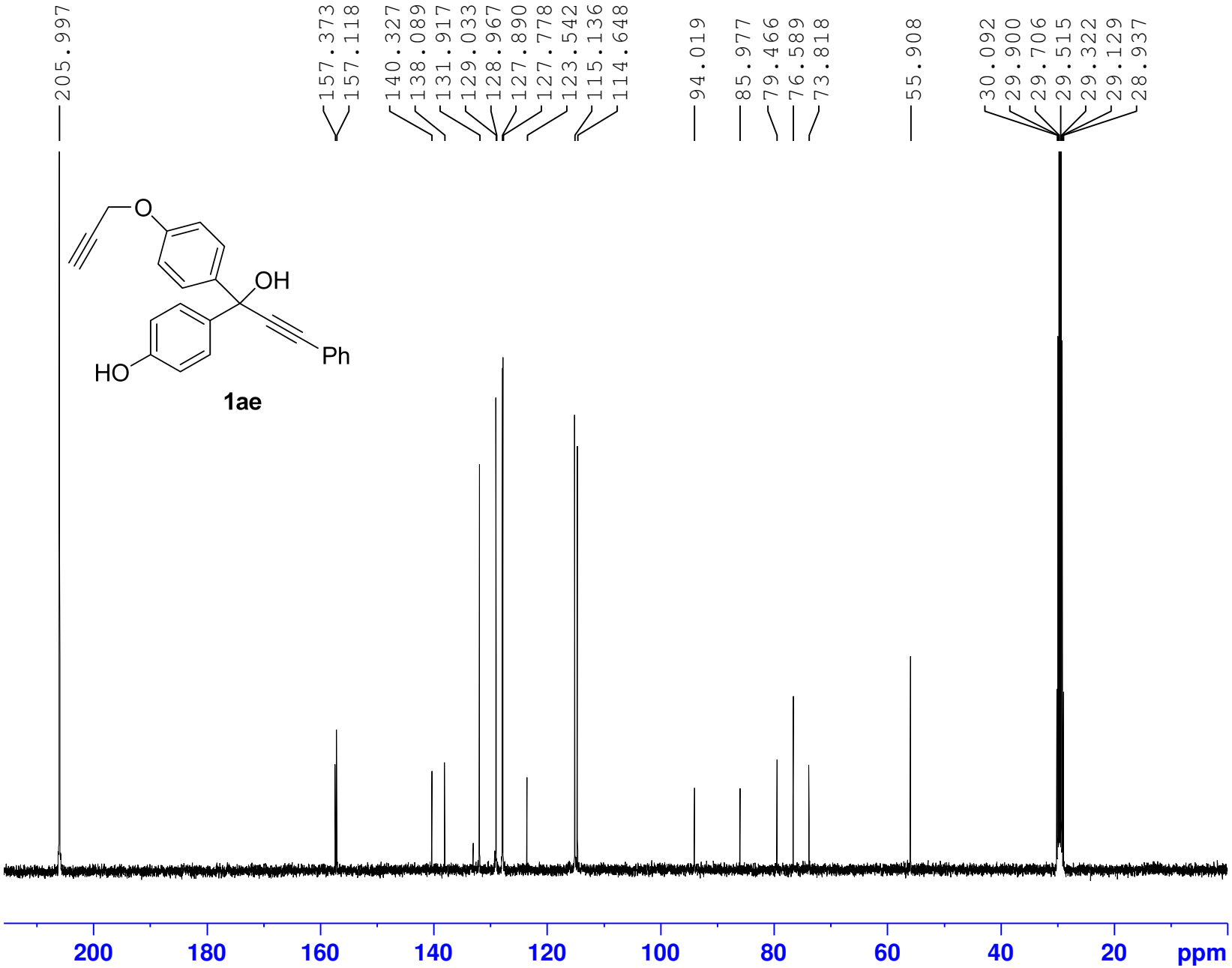

```
Current Data Parameters
NAME         qdy-10194 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160328
Time               22.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  130
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127049 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 64. ^{13}C NMR spectrum for 1ad



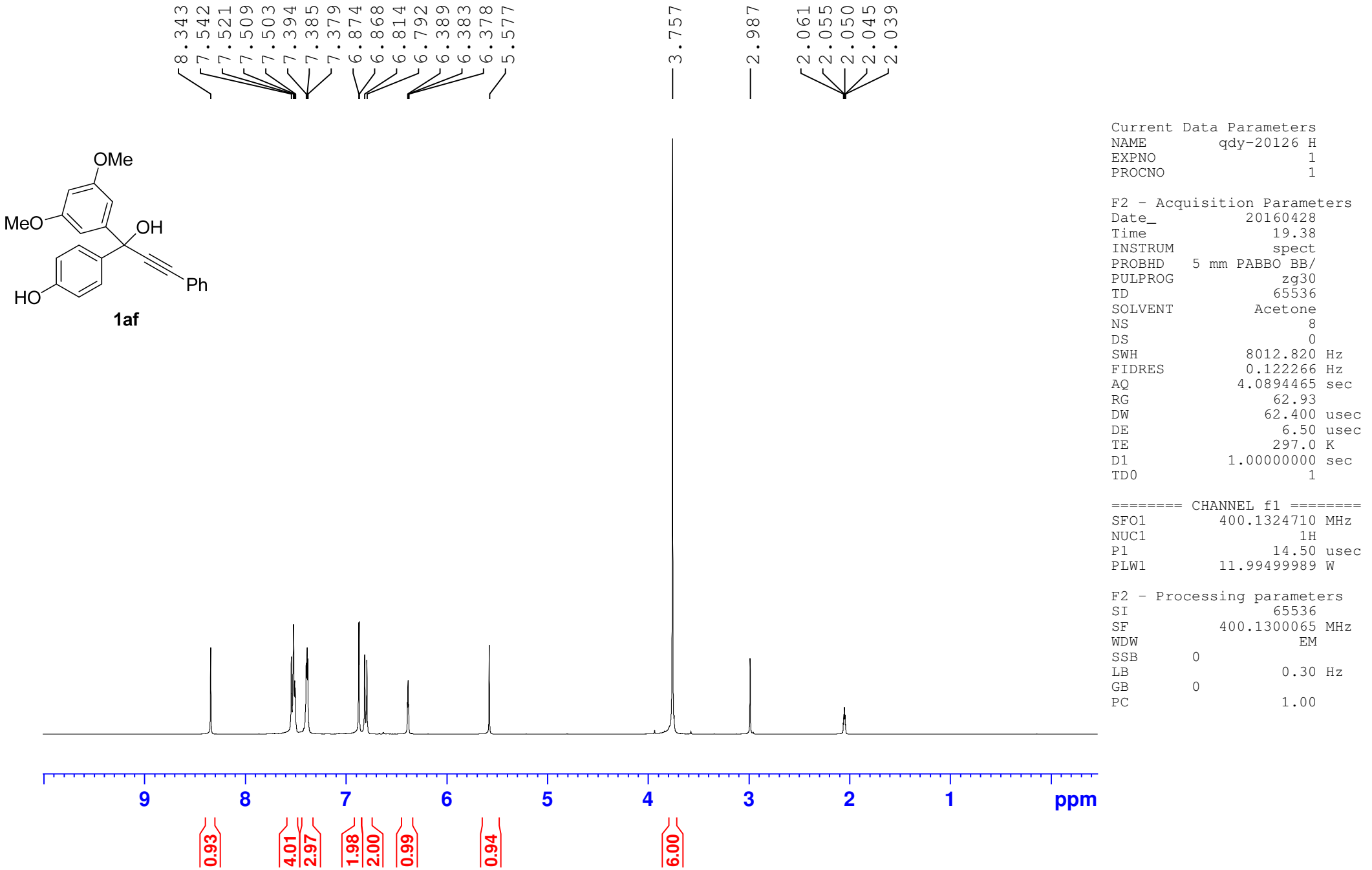

```
Current Data Parameters
NAME         qdy-20068 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160310
Time               19.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    12
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 49.32
DW                62.400 usec
DE                 6.50 usec
TE                296.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300066 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 65. ^{1}H NMR spectrum for 1ae




```
Current Data Parameters
NAME         qdy-20068 C
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160310
Time               19.46
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  115
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127177 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

Supplementary Figure 66. ^{13}C NMR spectrum for 1ae



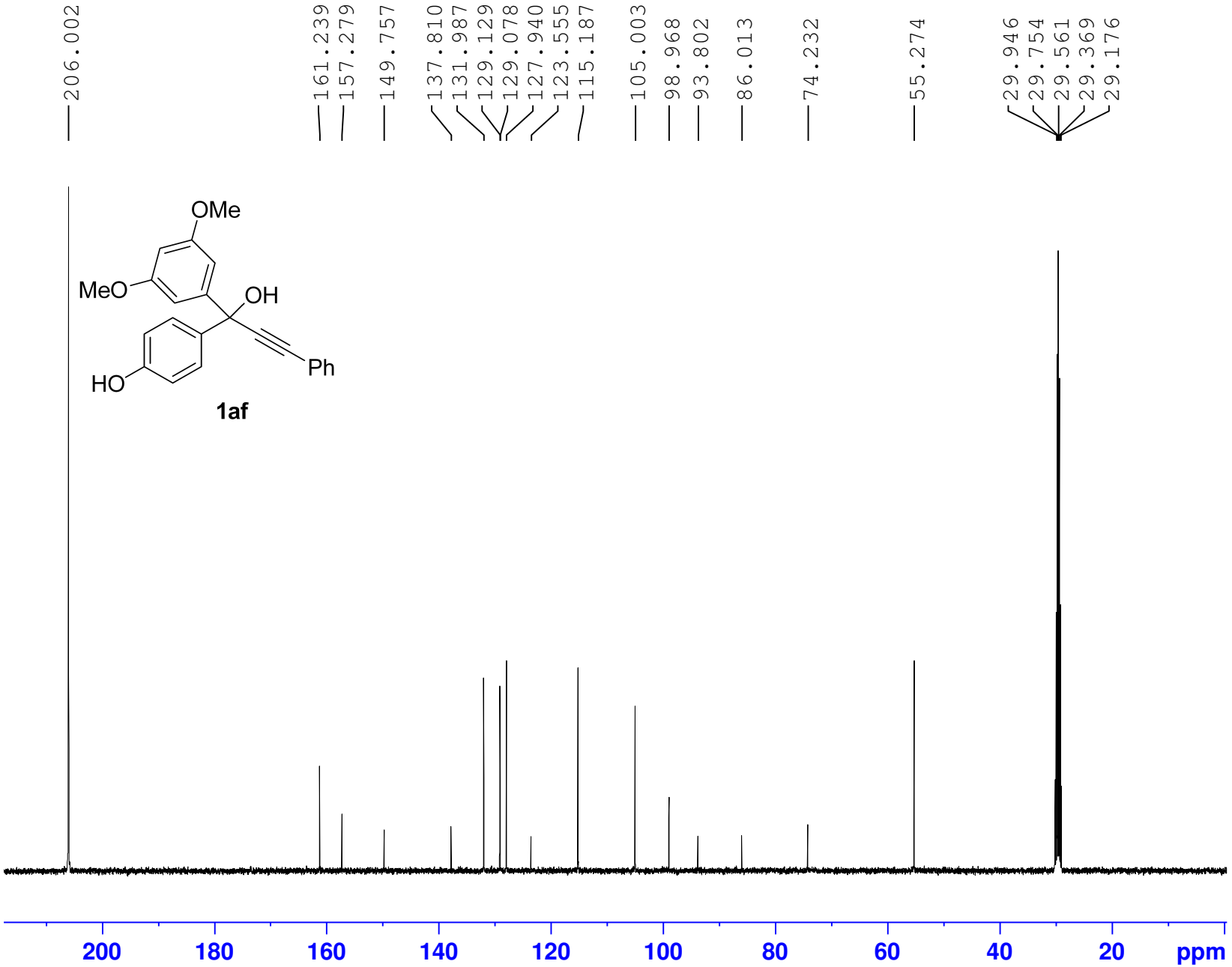
Current Data Parameters
NAME qdy-20126 H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160428
Time 19.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

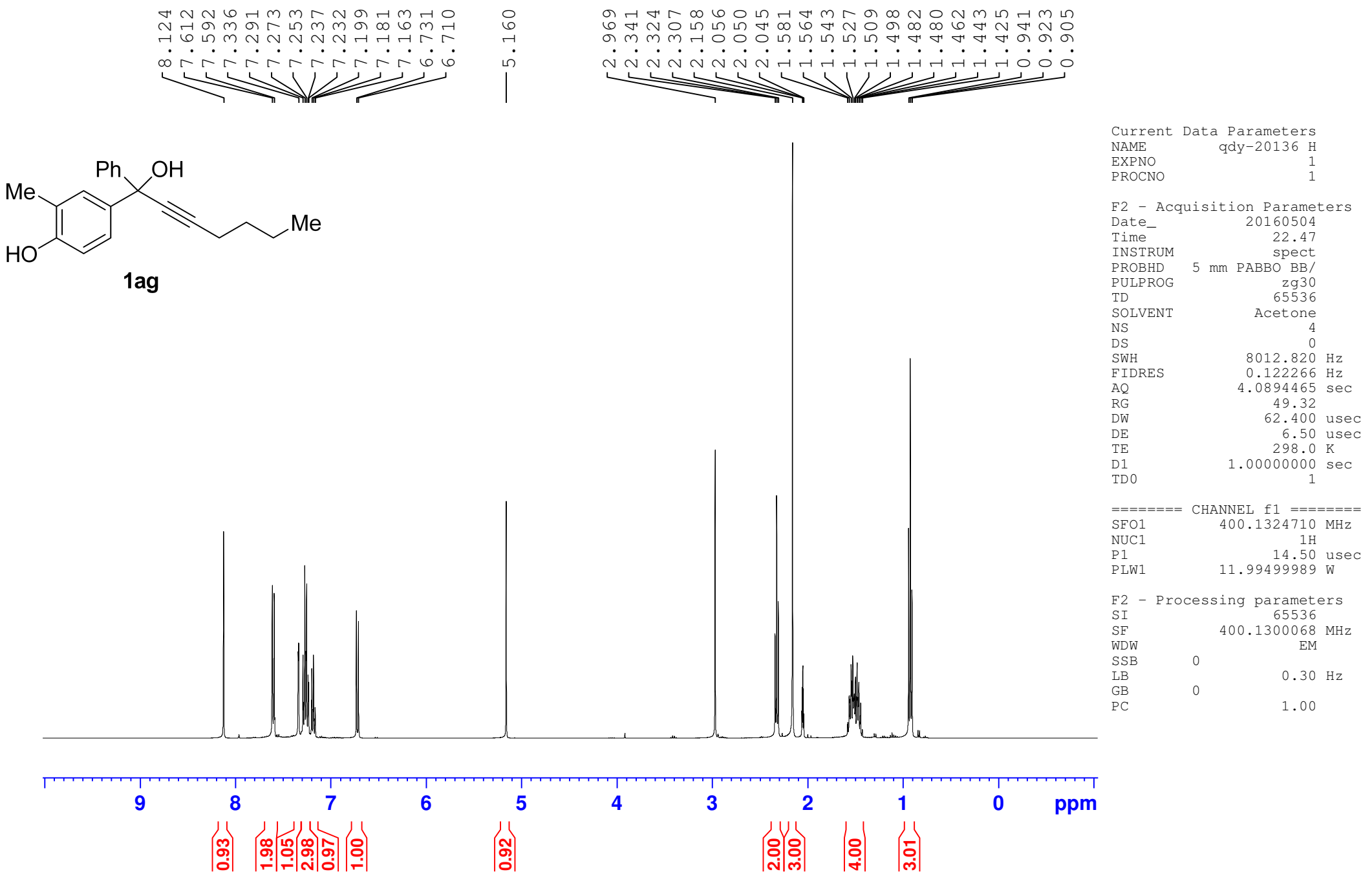
F2 - Processing parameters
SI 65536
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 67. ^{1}H NMR spectrum for 1af



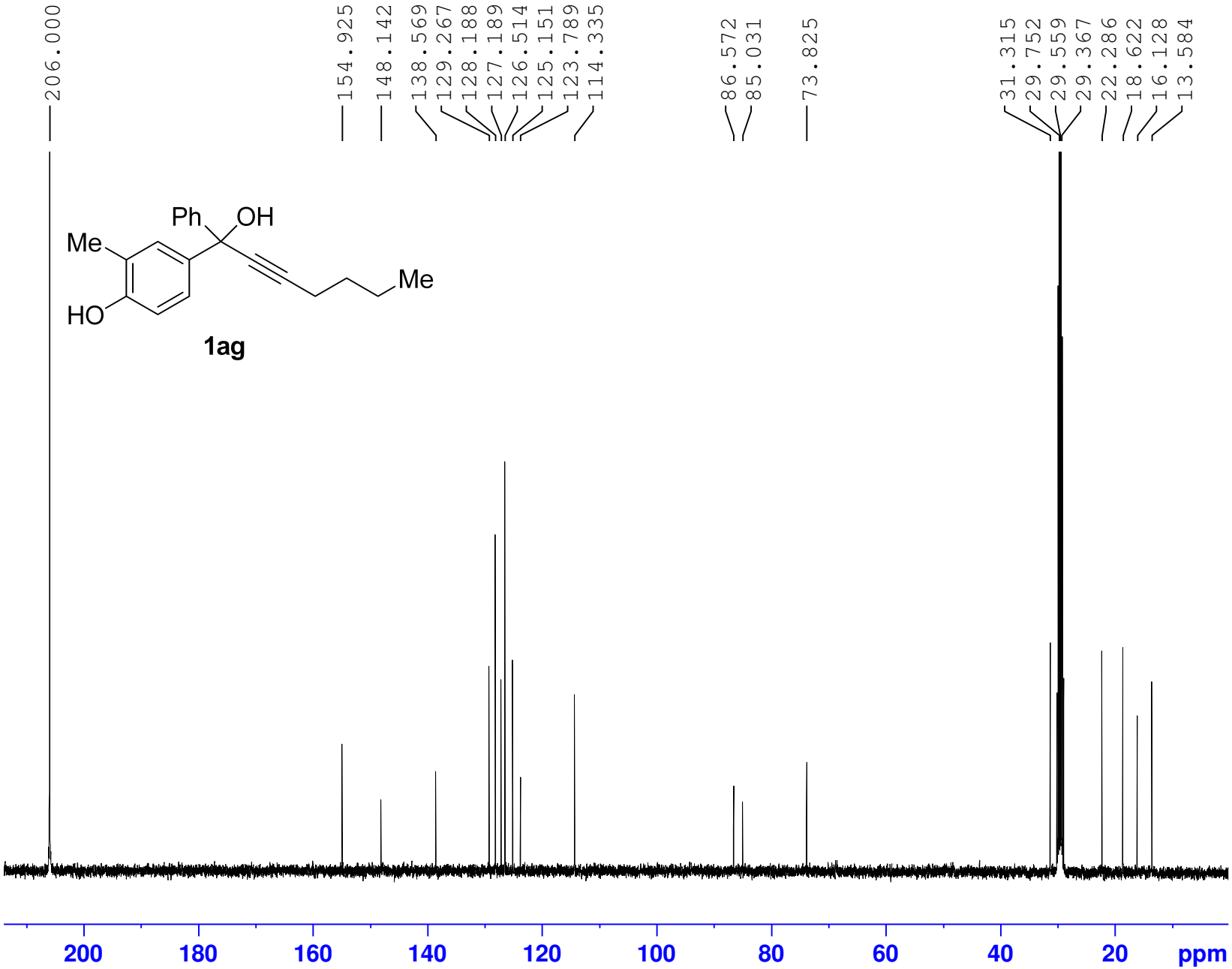

```
Current Data Parameters
NAME         qdy-20126 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160428
Time               19.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   105
DS                     0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE               297.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127103 MHz
WDW                  EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

Supplementary Figure 68. ^{13}C NMR spectrum for 1af



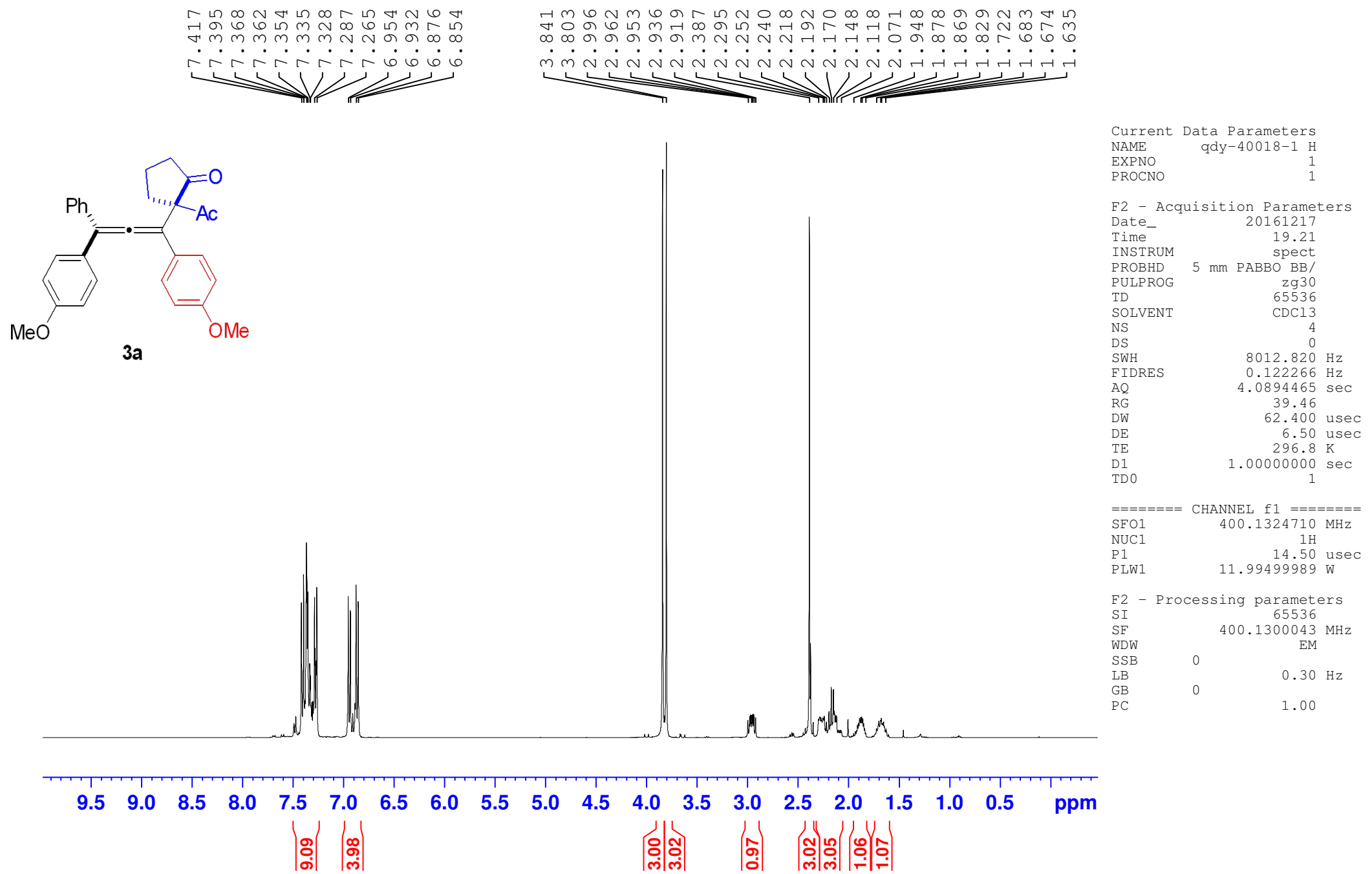

```
Current Data Parameters
NAME         qdy-20136 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160504
Time               22.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 49.32
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300068 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 69. ^{1}H NMR spectrum for 1ag

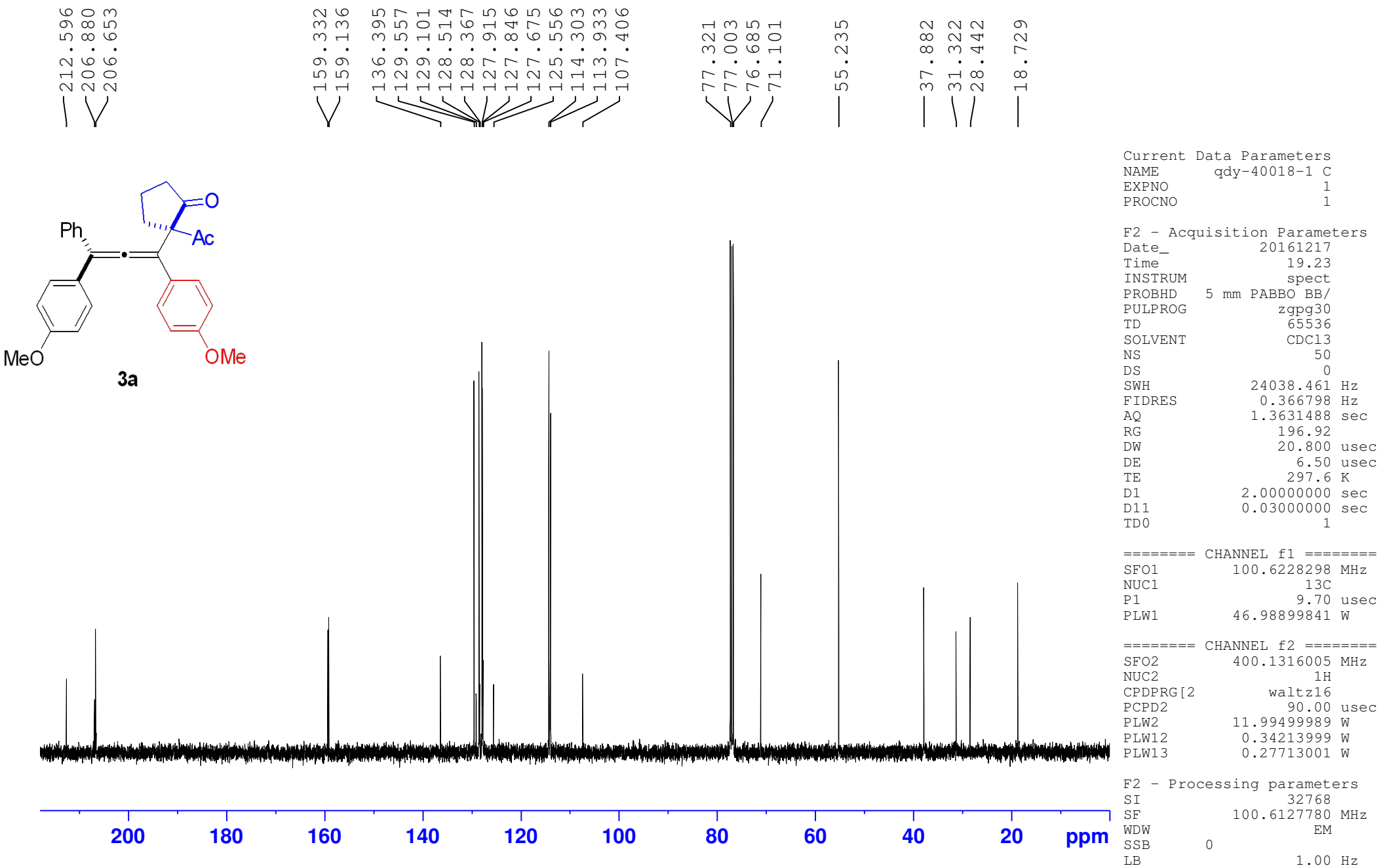



```
Current Data Parameters
NAME         qdy-20136 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160504
Time               22.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                    95
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127103 MHz
WDW                  EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

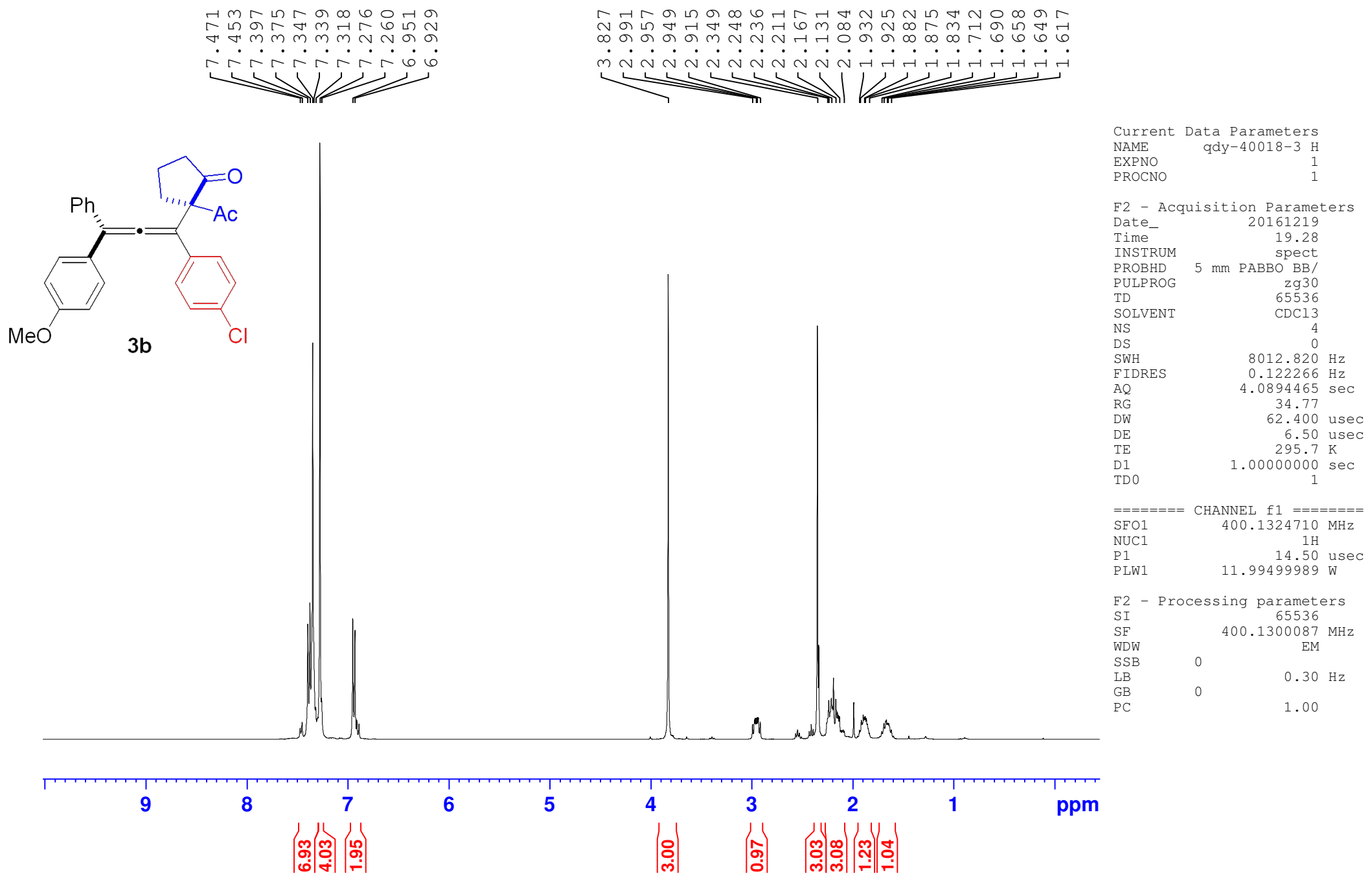
Supplementary Figure 70. ^{13}C NMR spectrum for 1ag



Supplementary Figure 71. ^{1}H NMR spectrum for 3a

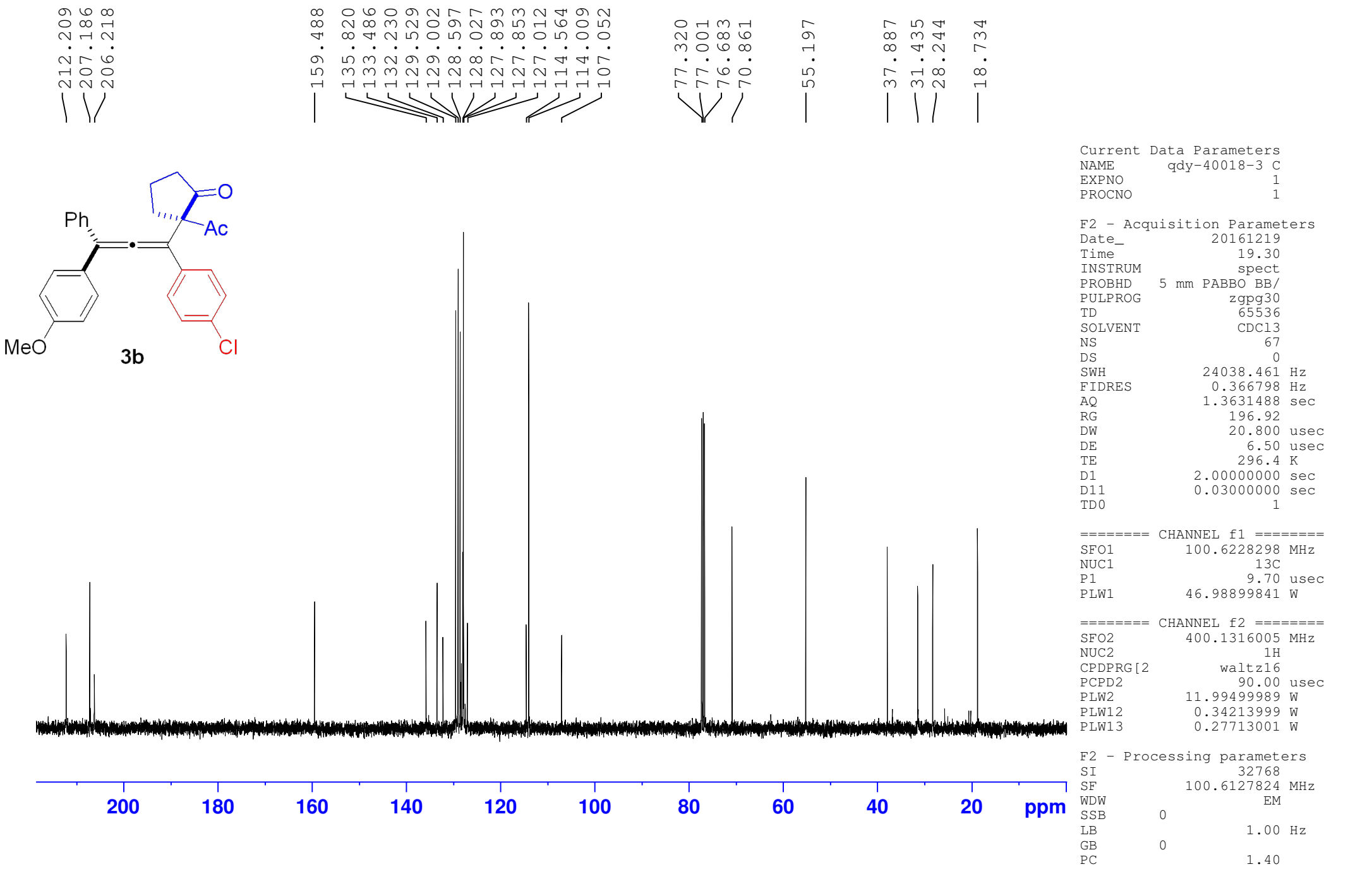


Supplementary Figure 72. ^{1}H NMR spectrum for 3a

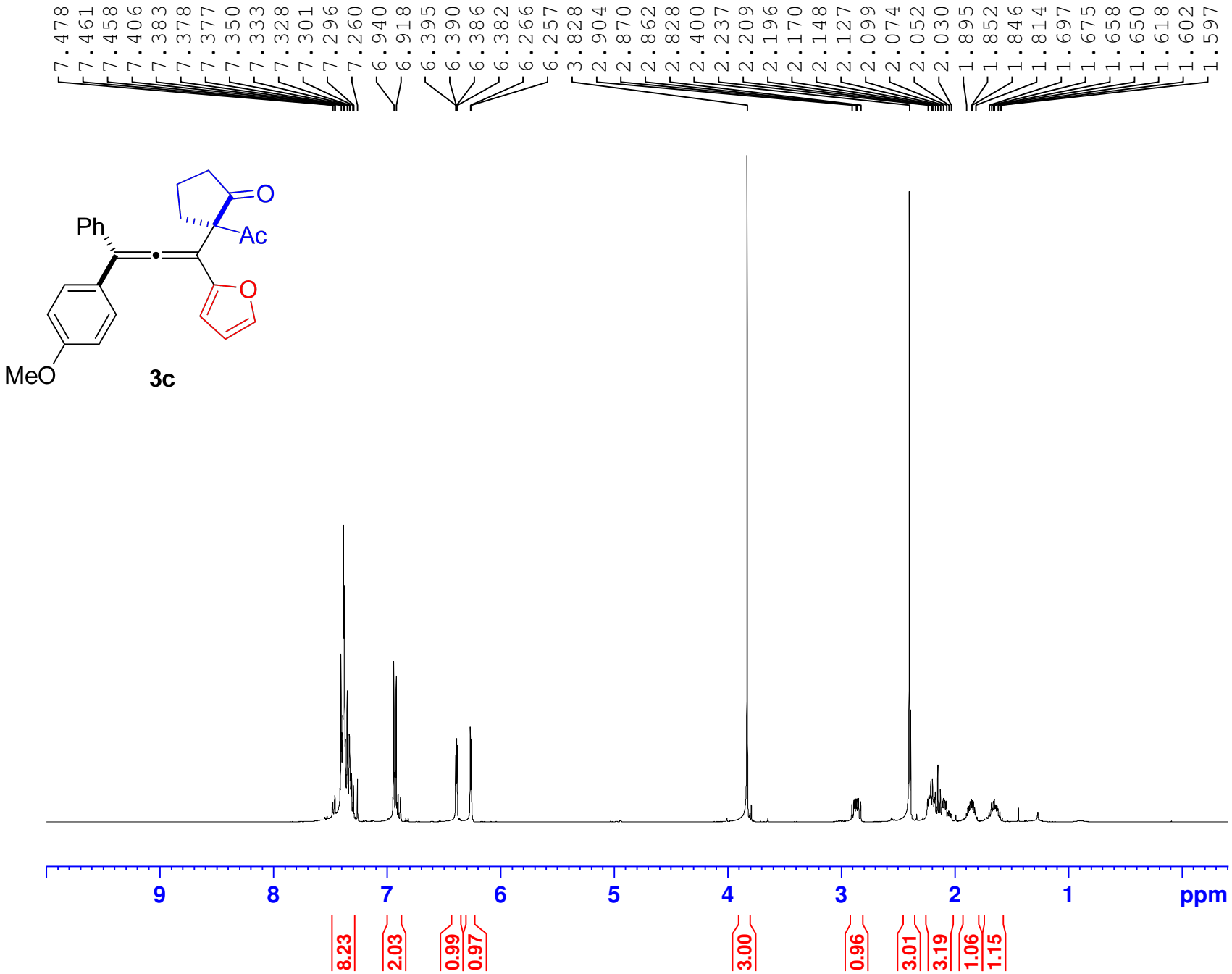



```
Current Data Parameters
NAME       qdy-40018-3 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161219
Time              19.28
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                34.77
DW               62.400 usec
DE                6.50 usec
TE                295.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300087 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 73. ^{1}H NMR spectrum for 3b

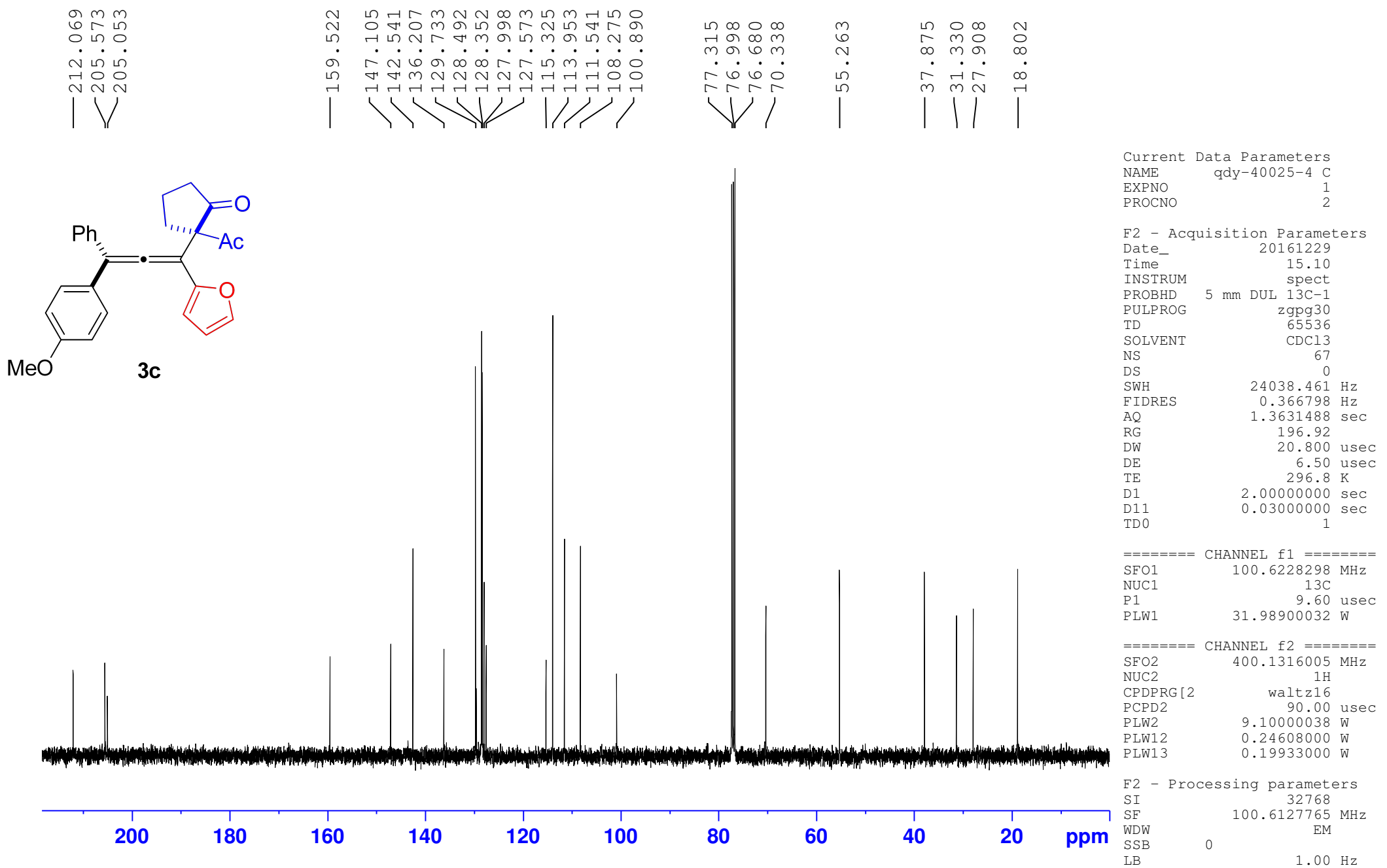


Supplementary Figure 74. ^{13}C NMR spectrum for 3b



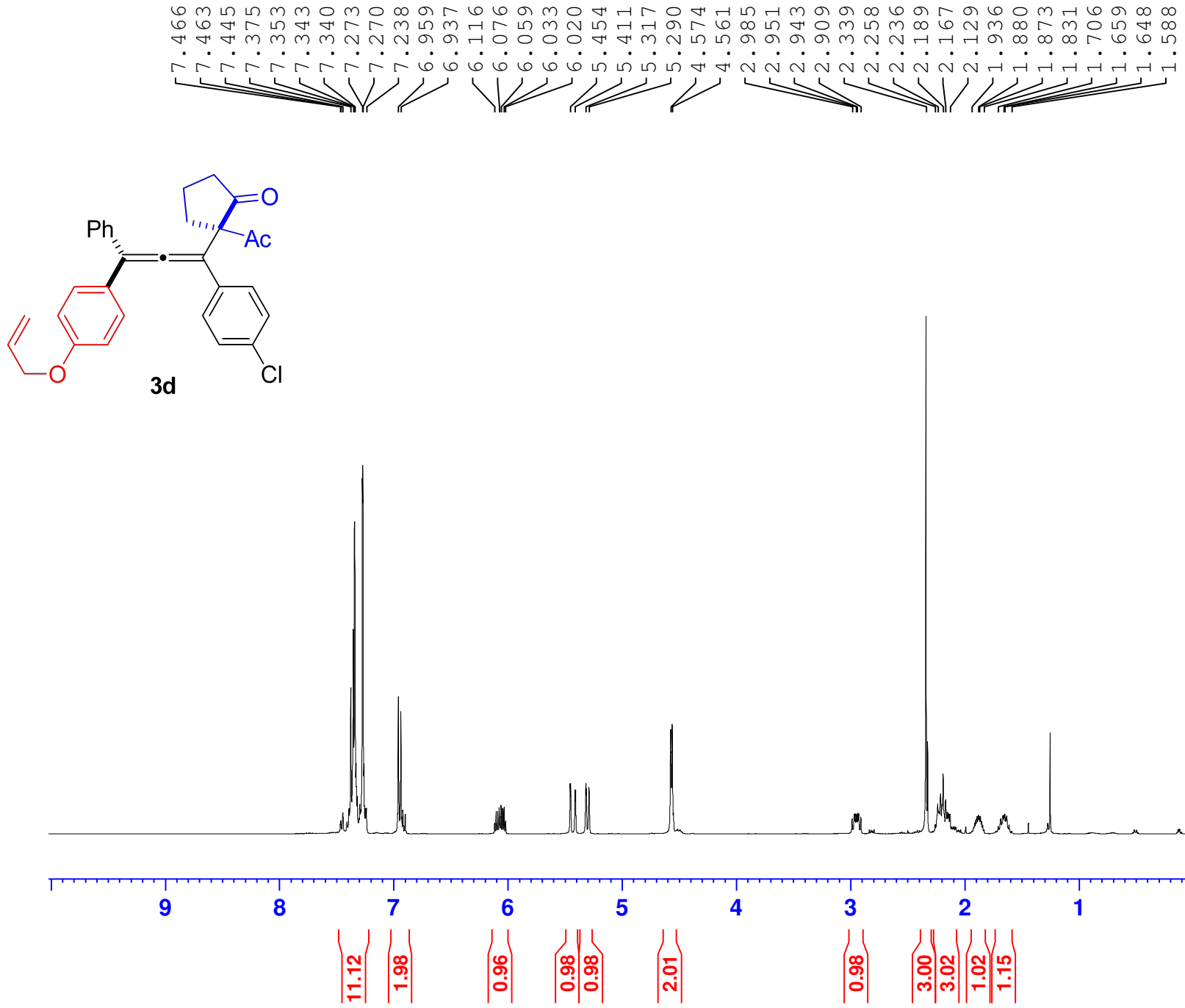

```
Current Data Parameters
NAME         qdy-40025 H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161229
Time               15.05
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 49.32
DW                62.400 usec
DE                 6.50 usec
TE                296.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                14.30 usec
PLW1          9.10000038 W

F2 - Processing parameters
SI                 65536
SF           400.1300090 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 75. ^{1}H NMR spectrum for 3c



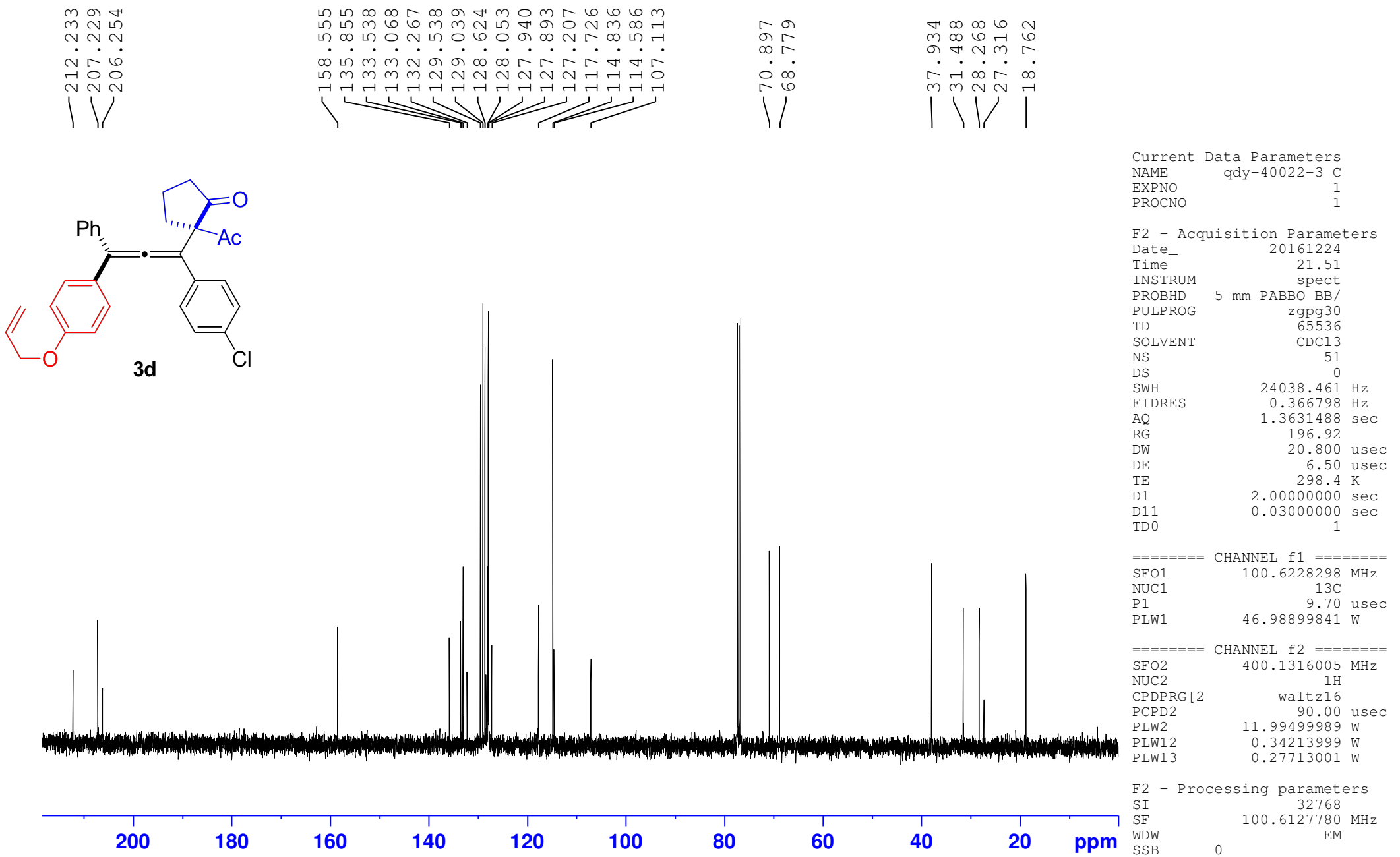

```
Current Data Parameters
NAME      qdy-40025-4 C
EXPNO                1
PROCNO               2

F2 - Acquisition Parameters
Date_          20161229
Time              15.10
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   67
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.60 usec
PLW1         31.98900032 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2          9.10000038 W
PLW12         0.24608000 W
PLW13         0.19933000 W

F2 - Processing parameters
SI                 32768
SF           100.6127765 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 76. ^{13}C NMR spectrum for 3c




```
Current Data Parameters
NAME       qdy-40022-3 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161224
Time              21.48
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                45.67
DW               62.400 usec
DE                6.50 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300091 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 77. ^{1}H NMR spectrum for 3d



Current Data Parameters
NAME qdy-40022-3 C
EXPNO 1
PROCNO 1

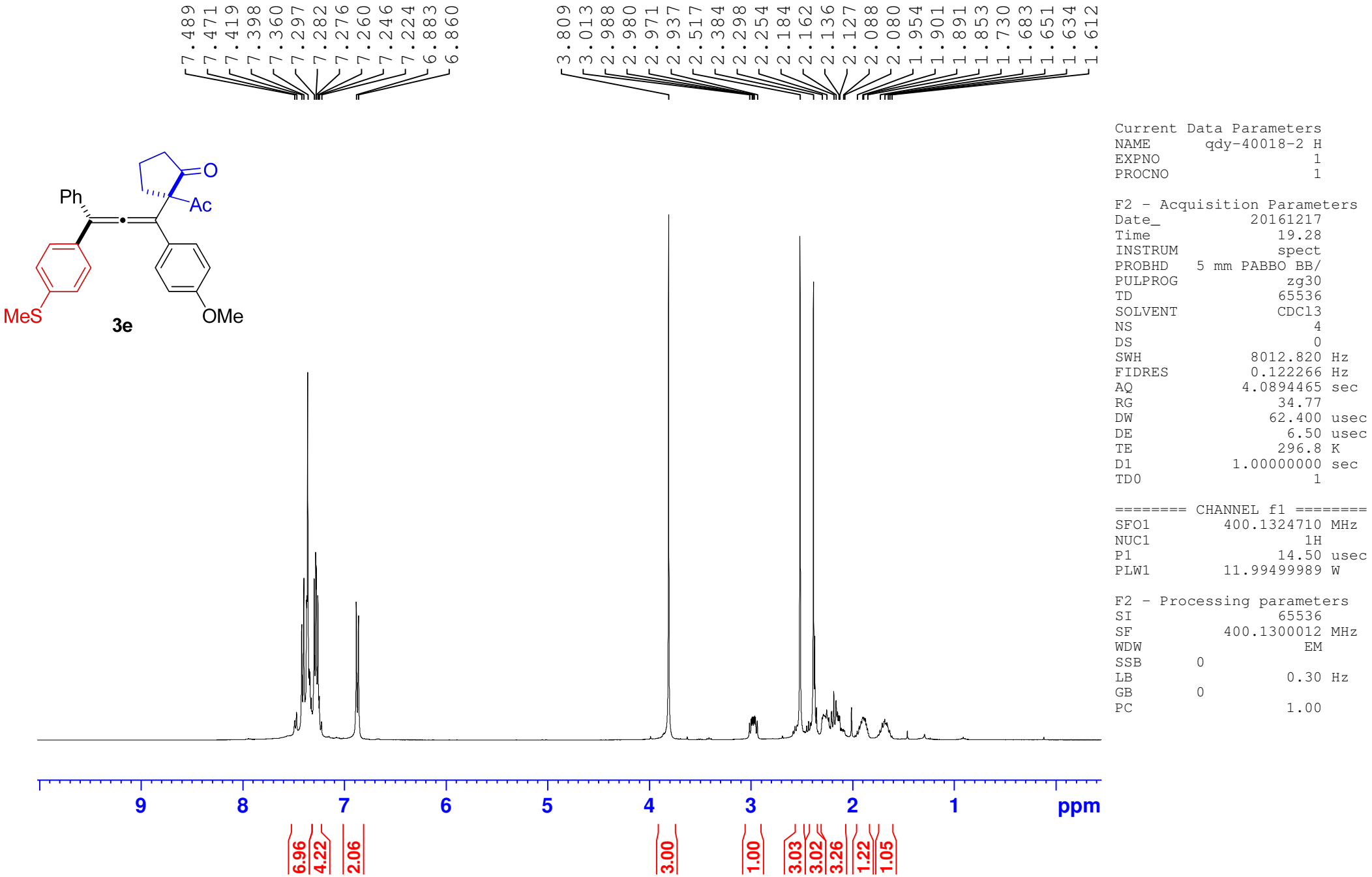
F2 - Acquisition Parameters
Date_ 20161224
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

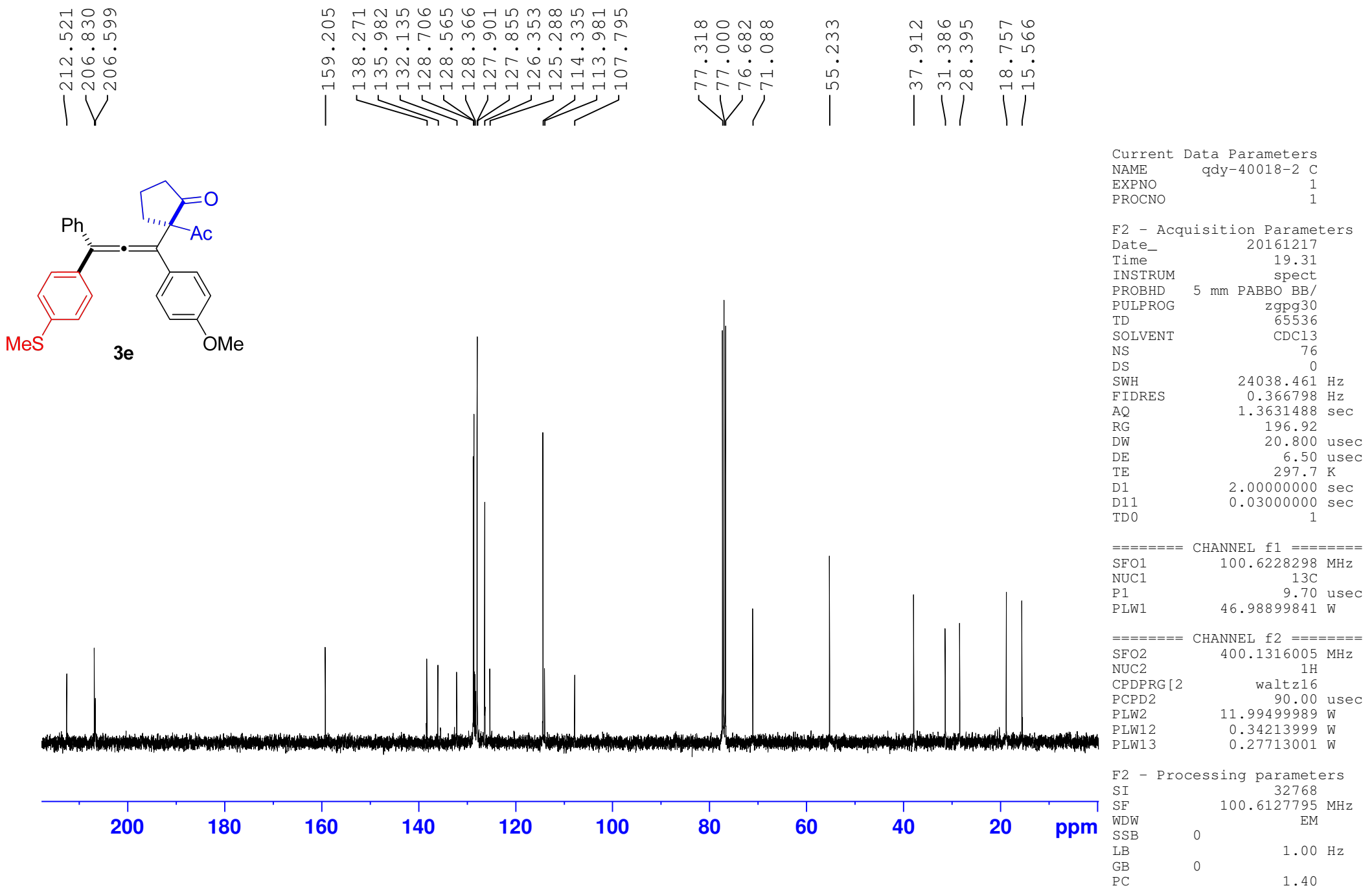
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 78. ^{13}C NMR spectrum for 3d

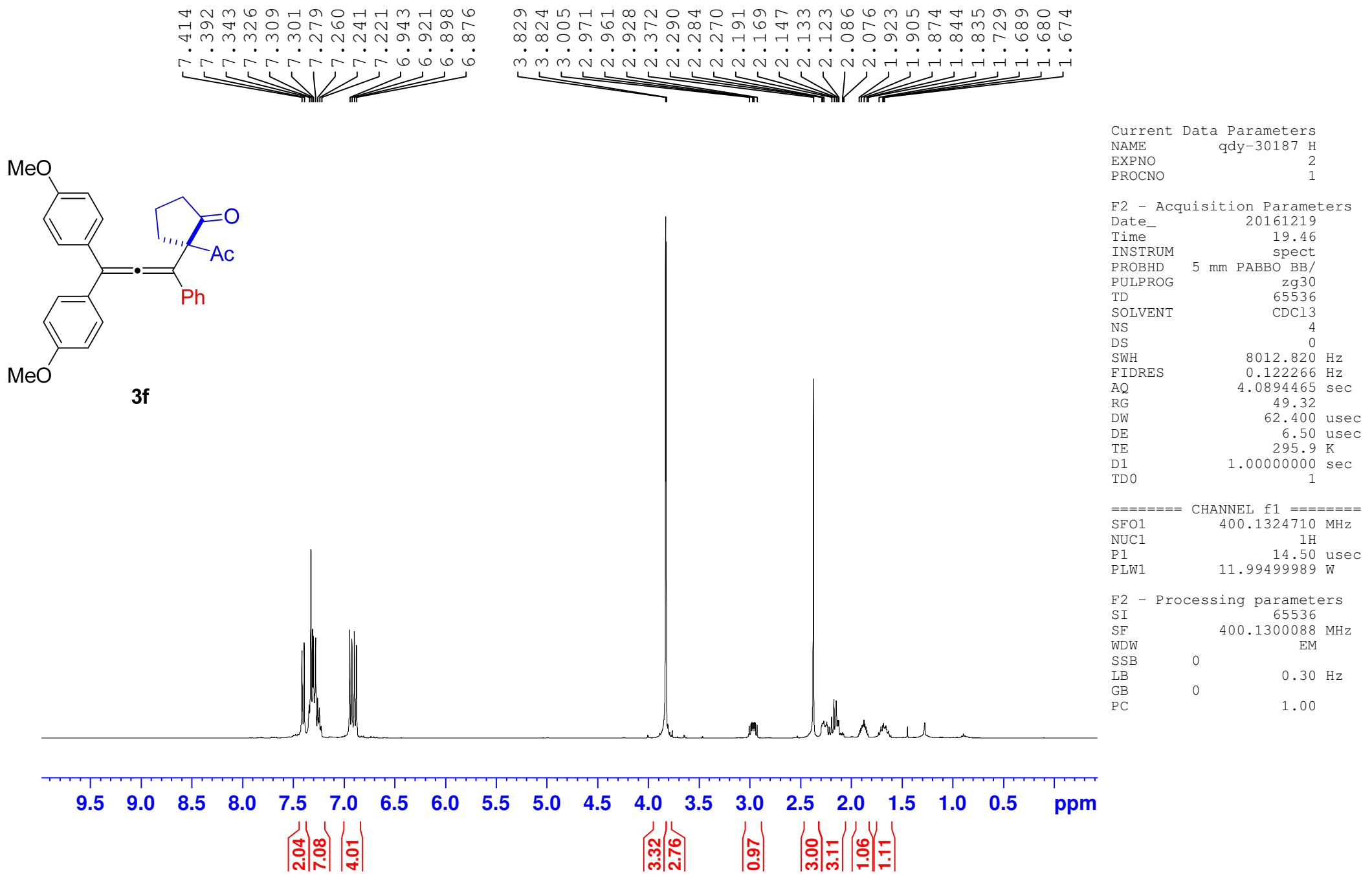



```
Current Data Parameters
NAME       qdy-40018-2 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161217
Time              19.28
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                34.77
DW               62.400 usec
DE                6.50 usec
TE                296.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300012 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

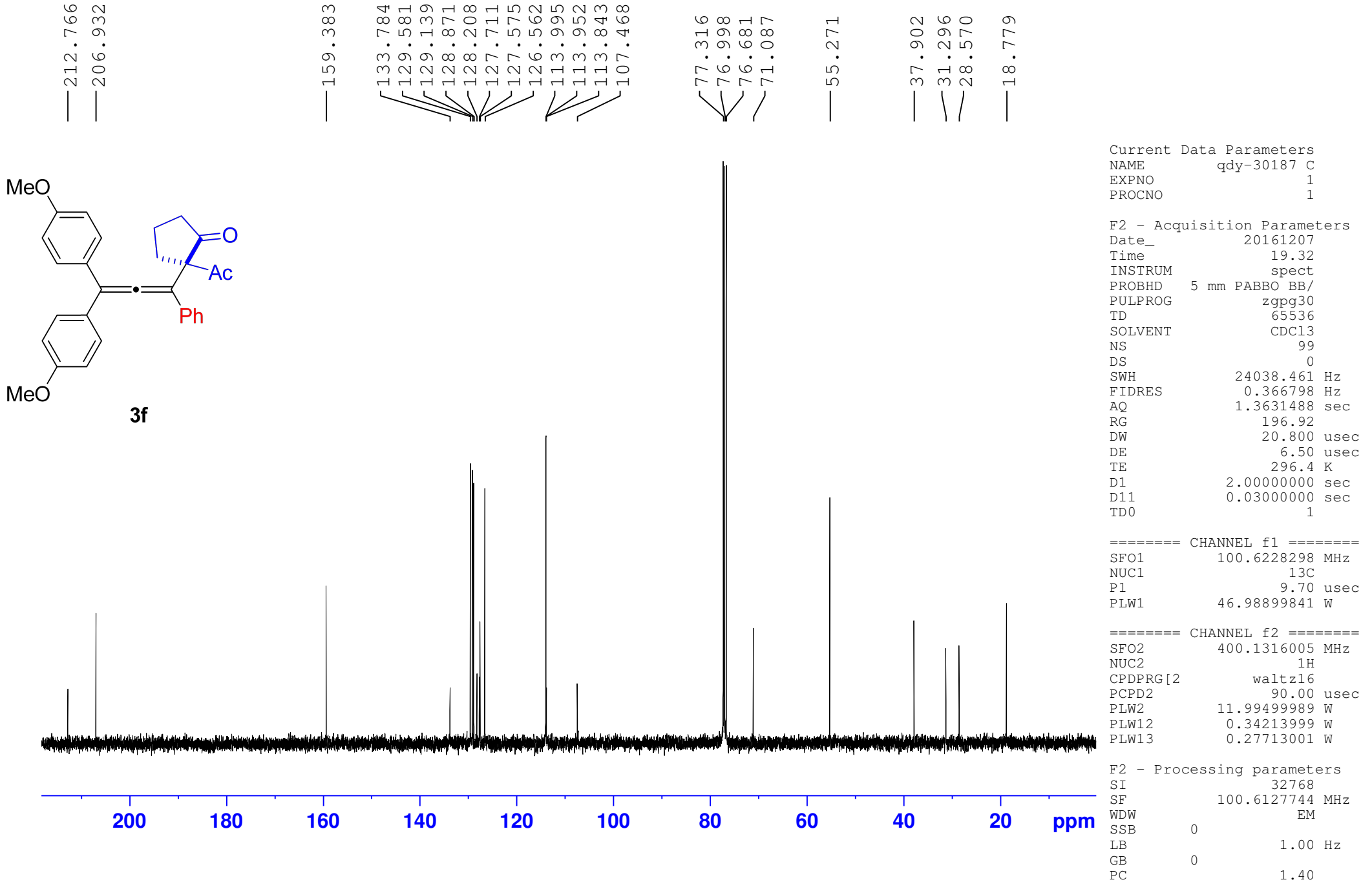
Supplementary Figure 79. ^{1}H NMR spectrum for 3e



Supplementary Figure 80. ^{13}C NMR spectrum for 3e

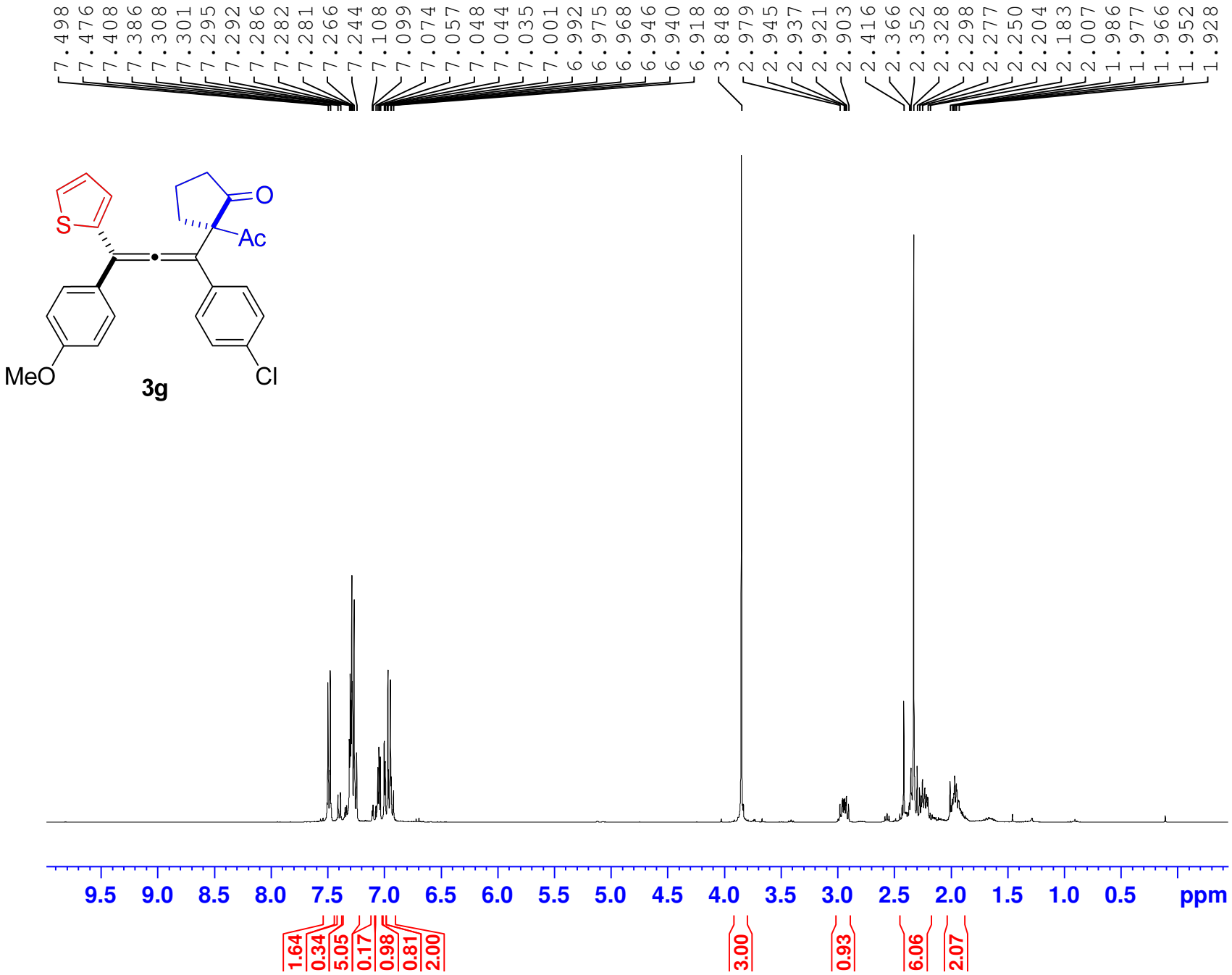


Supplementary Figure 81. ^{1}H NMR spectrum for 3f



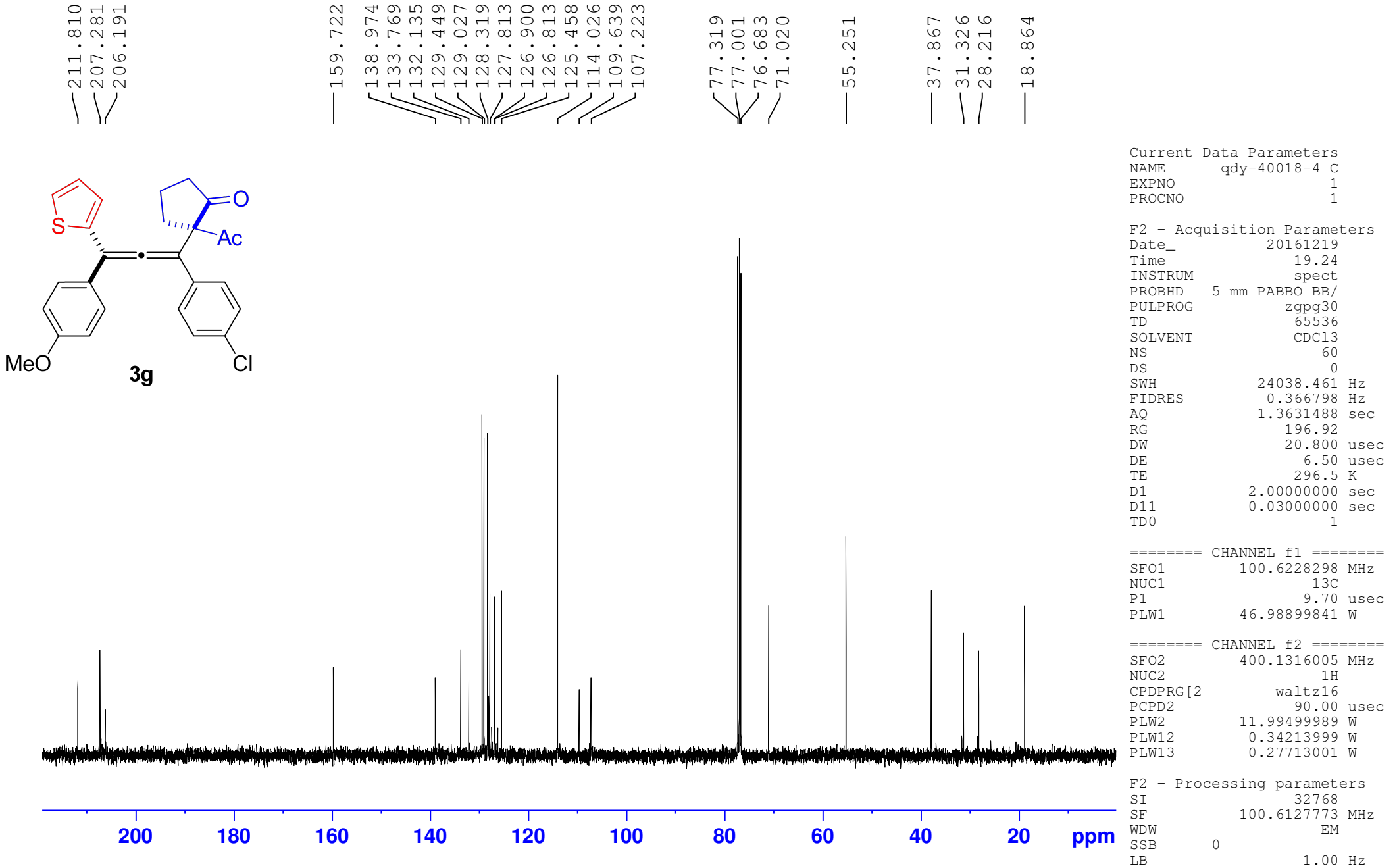

```
Current Data Parameters
NAME         qdy-30187 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161207
Time               19.32
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   99
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127744 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 82. ^{13}C NMR spectrum for 3f



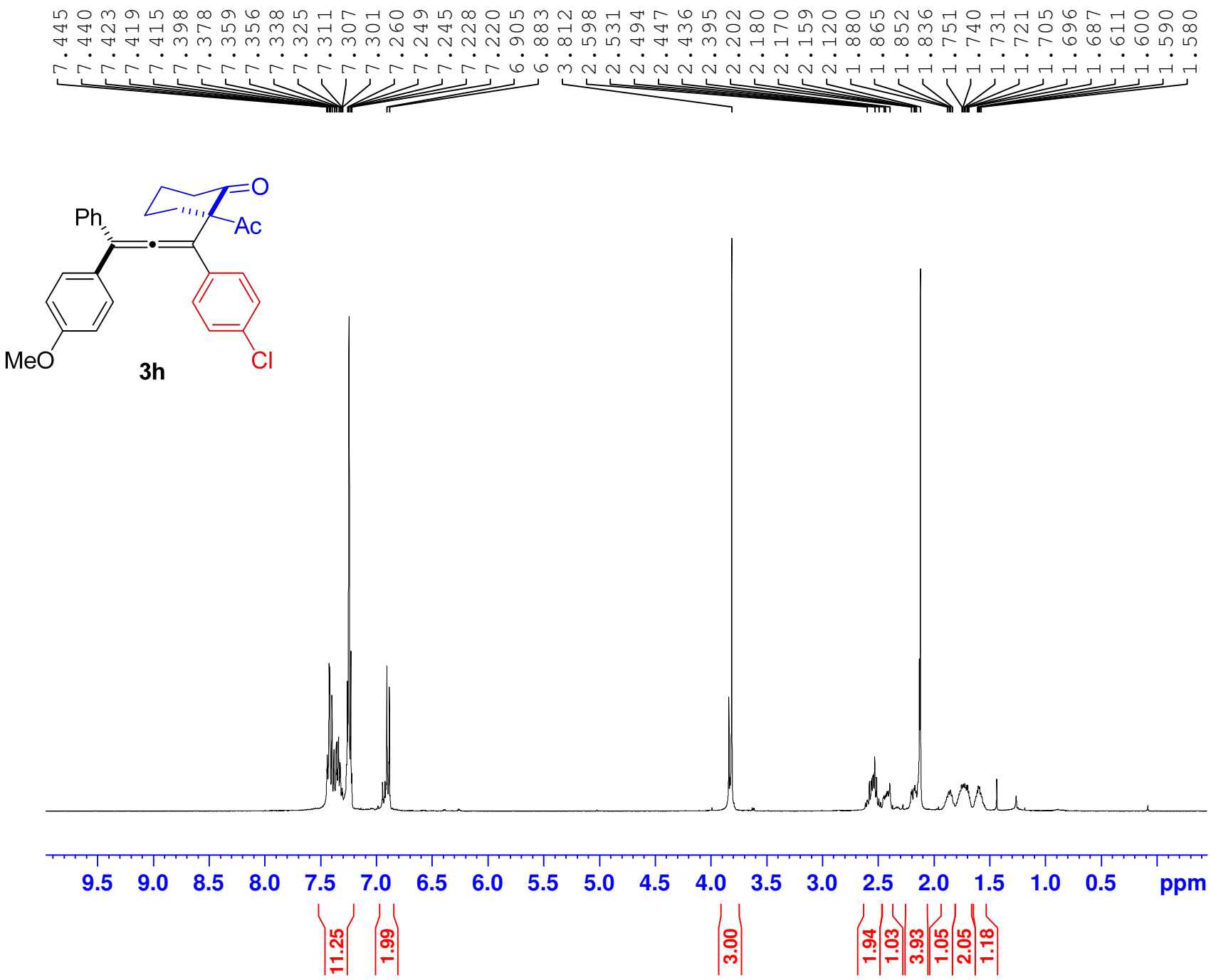

```
Current Data Parameters
NAME       qdy-40018-4 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161219
Time              19.21
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                45.67
DW               62.400 usec
DE                6.50 usec
TE                295.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300009 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 83. ^{1}H NMR spectrum for 3g



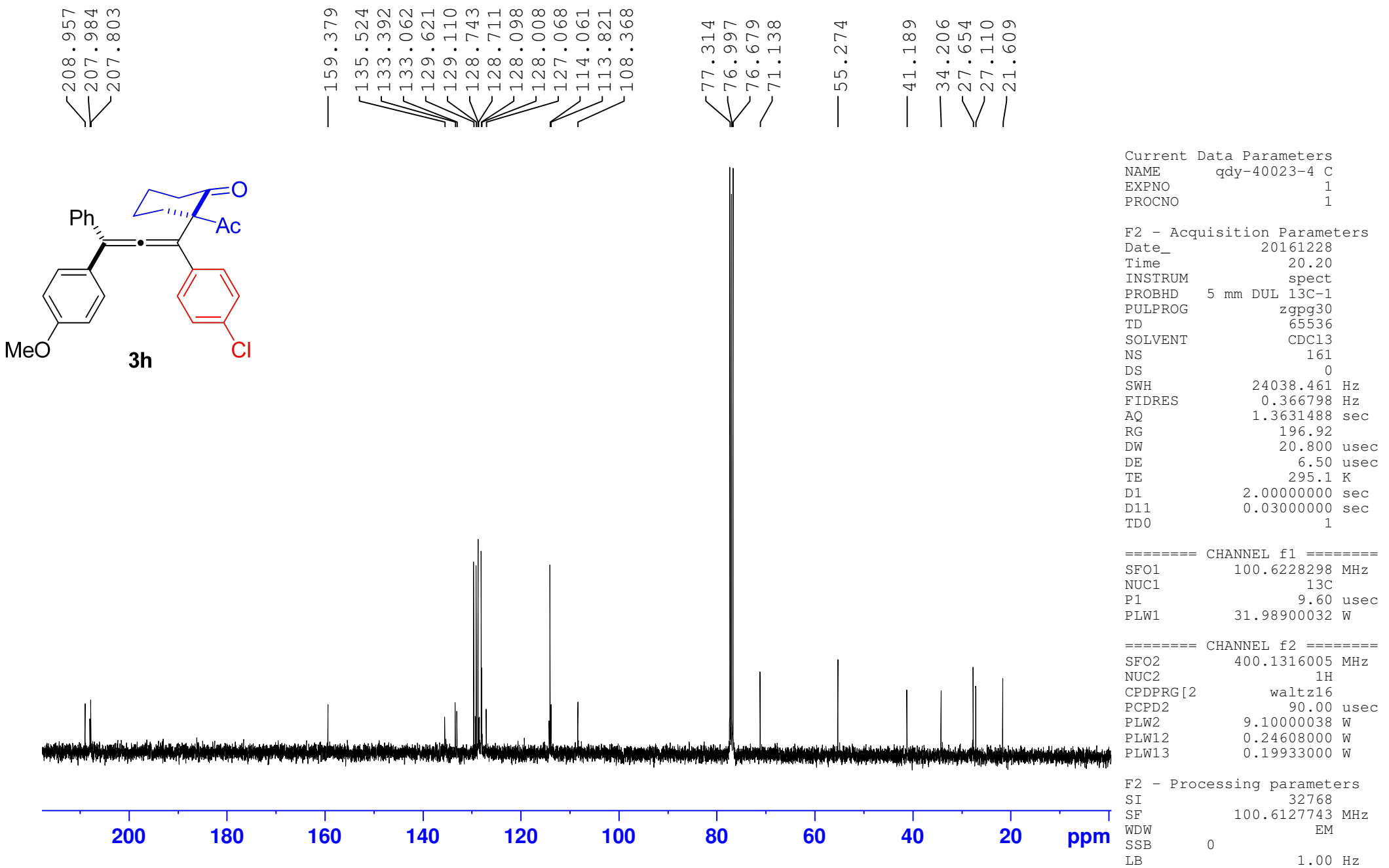

```
Current Data Parameters
NAME       qdy-40018-4 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161219
Time              19.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   60
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127773 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 84. ^{13}C NMR spectrum for 3g

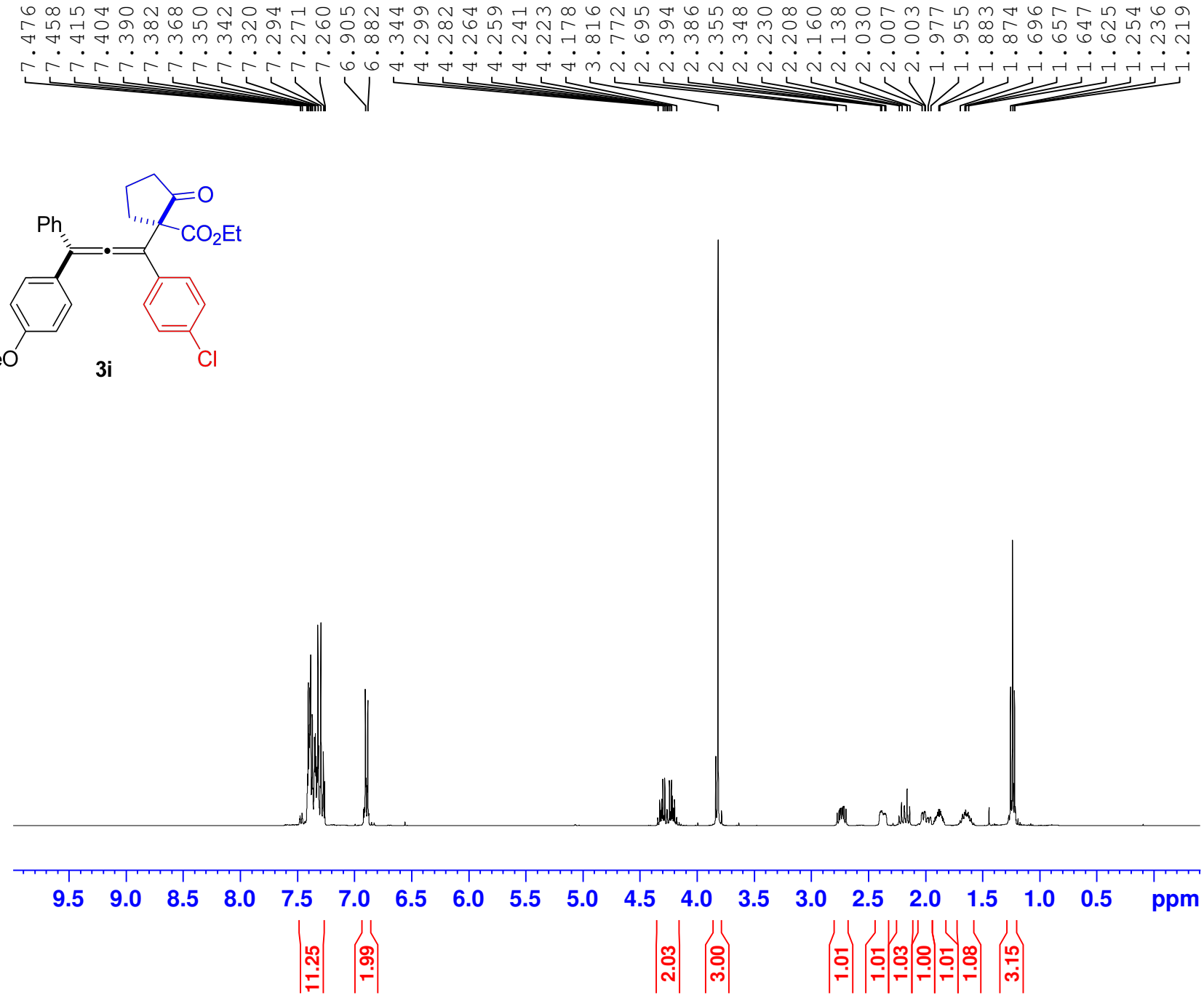



```
Current Data Parameters
NAME       qdy-40023-4 H
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20161228
Time              20.29
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                126.97
DW                62.400 usec
DE                 6.50 usec
TE                295.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.30 usec
PLW1         9.10000038 W

F2 - Processing parameters
SI                65536
SF          400.1300091 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 85. ^{1}H NMR spectrum for 3h

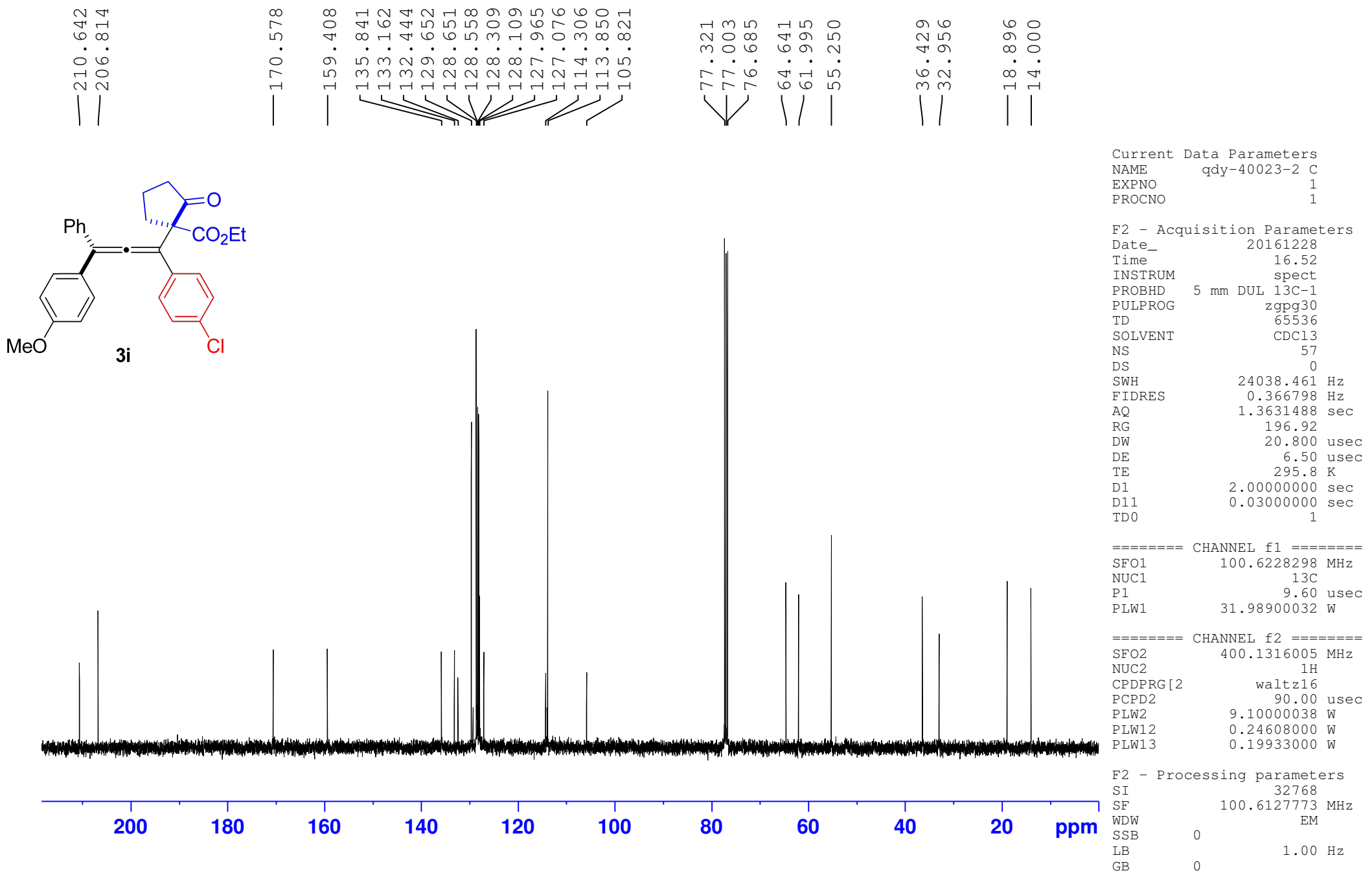


Supplementary Figure 86. ^{13}C NMR spectrum for 3h

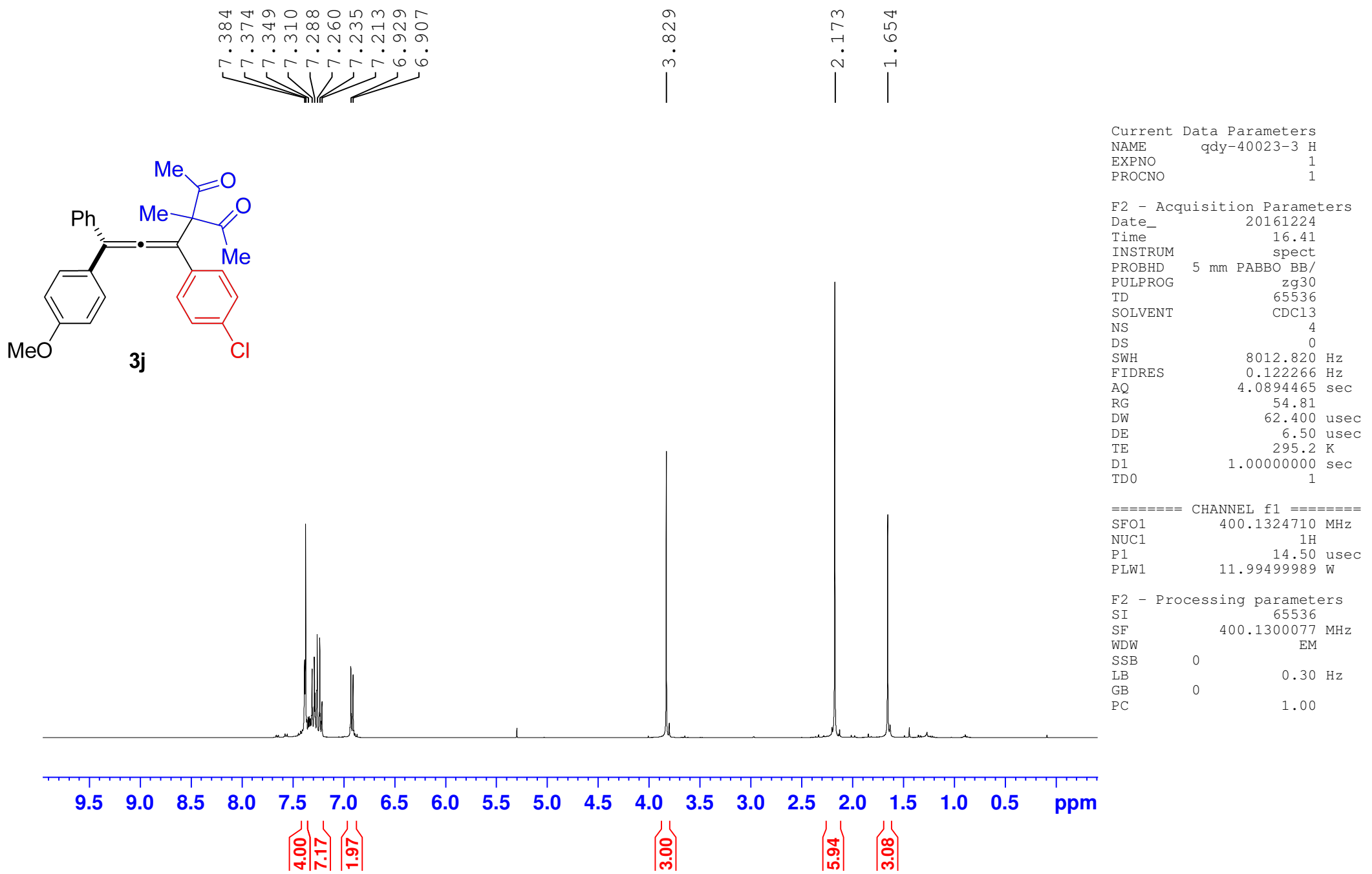



```
Current Data Parameters
NAME       qdy-40023-2 H
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20161228
Time              20.11
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                49.32
DW               62.400 usec
DE                6.50 usec
TE                294.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.30 usec
PLW1         9.10000038 W

F2 - Processing parameters
SI                65536
SF          400.1300089 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 87. ^{1}H NMR spectrum for 3i

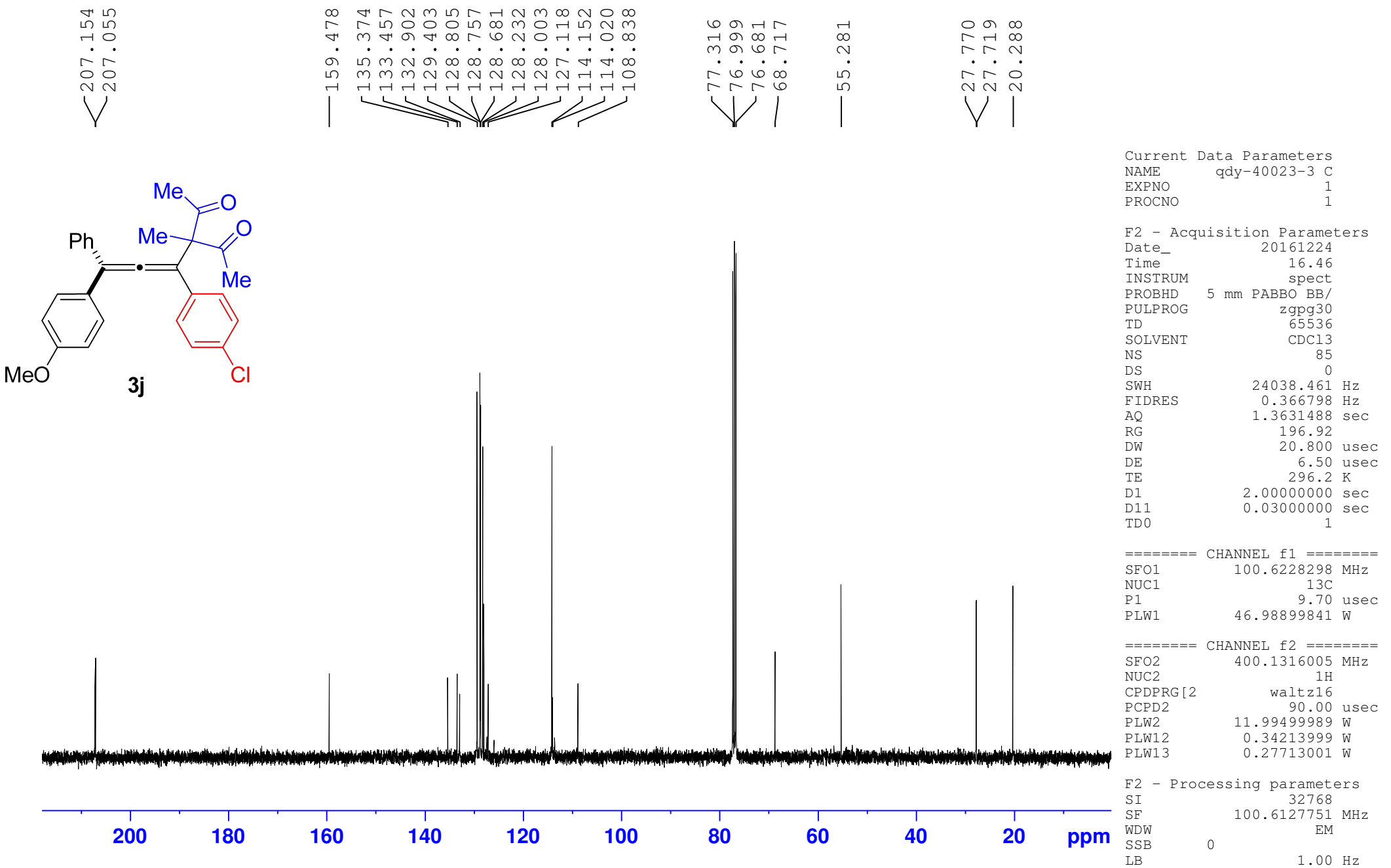


Supplementary Figure 88. ^{13}C NMR spectrum for 3i

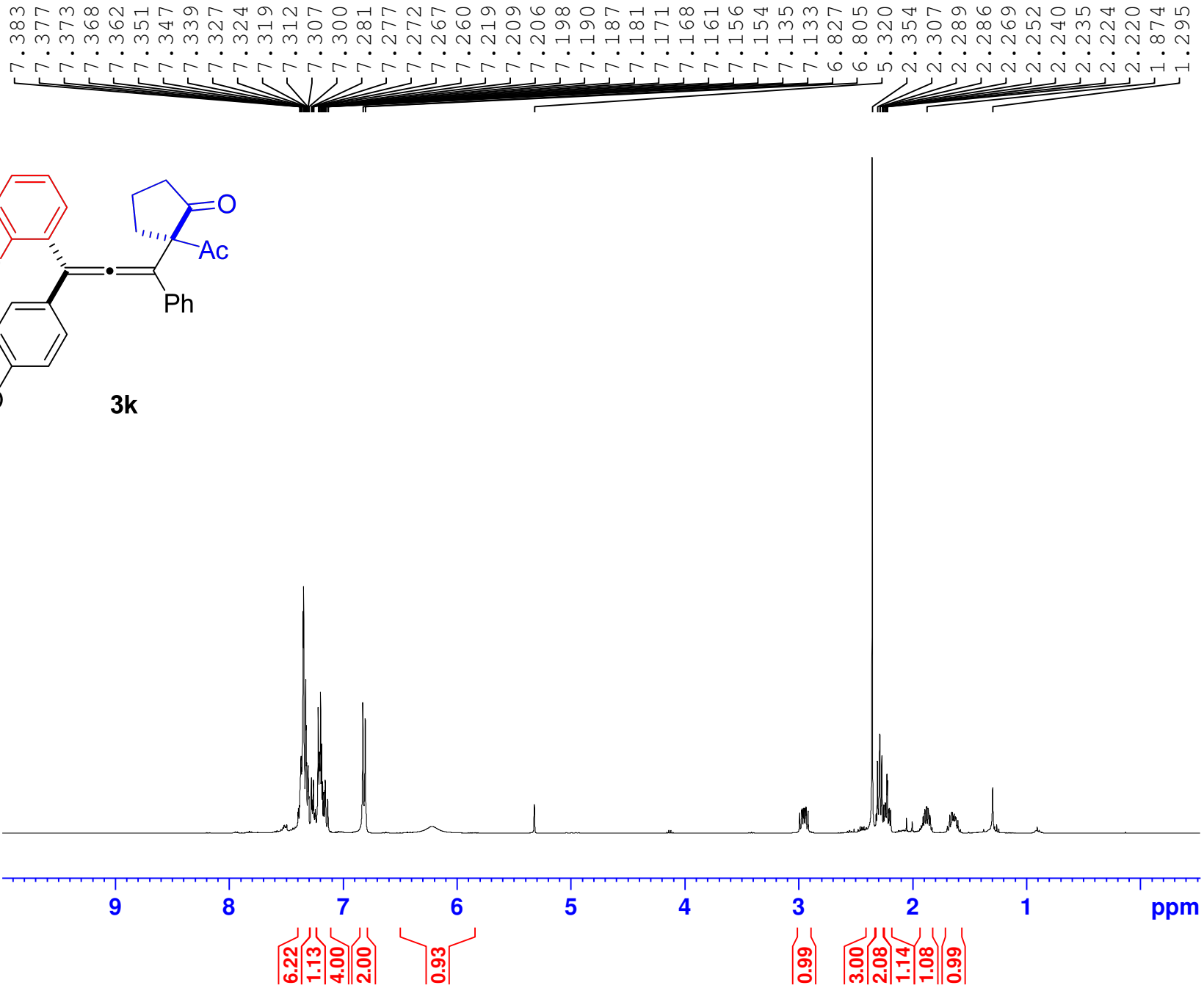



```
Current Data Parameters
NAME       qdy-40023-3 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161224
Time              16.41
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                54.81
DW               62.400 usec
DE                6.50 usec
TE                295.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300077 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 89. ^{1}H NMR spectrum for 3j

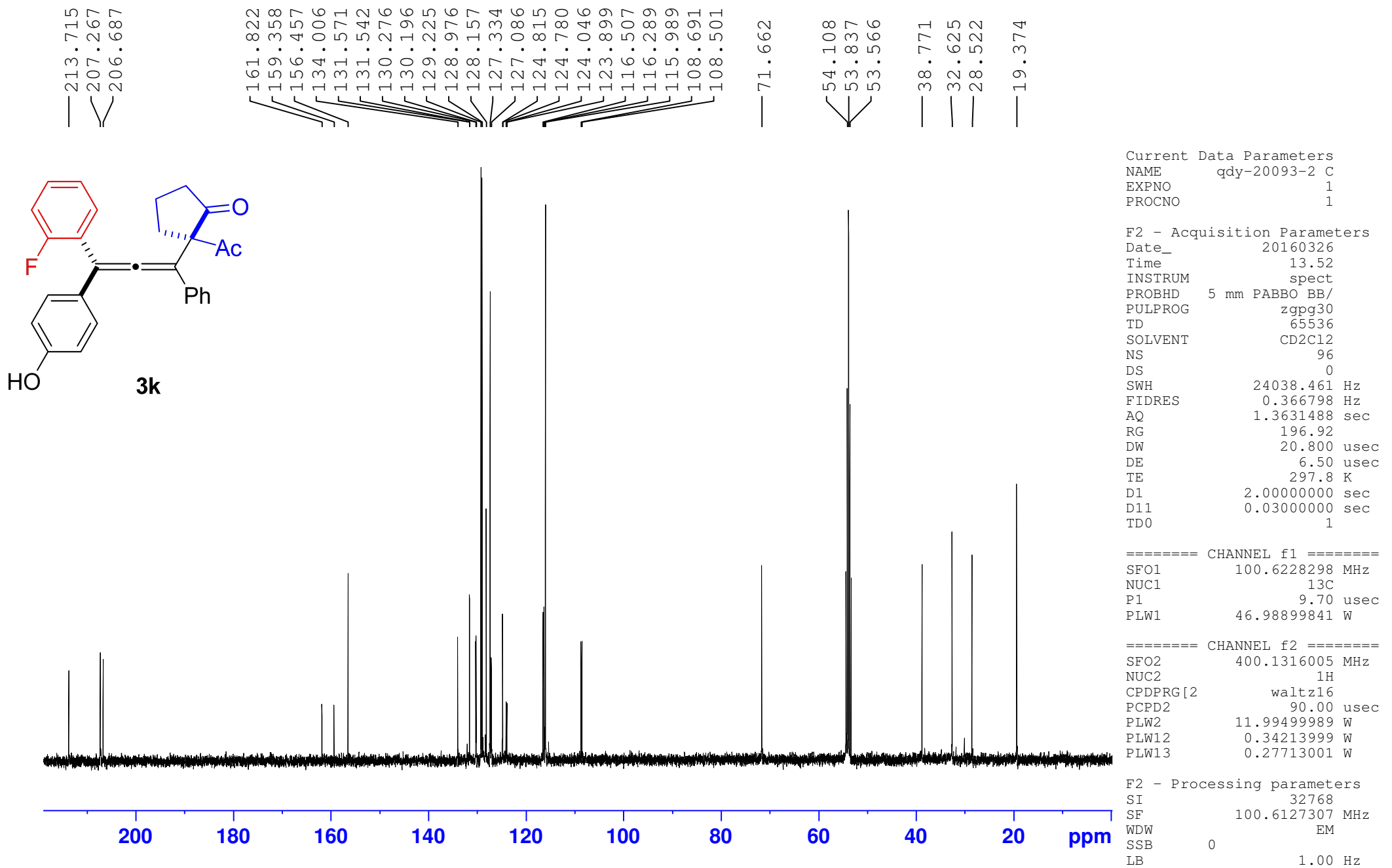


Supplementary Figure 90. ^{13}C NMR spectrum for 3j



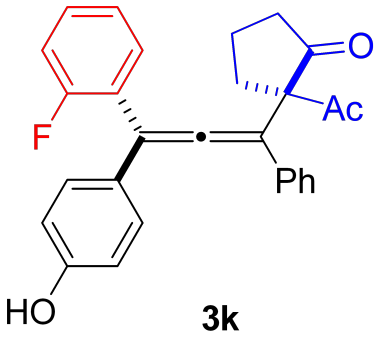

```
Current Data Parameters
NAME       qdy-20093-2 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160326
Time              13.46
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    5
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                34.77
DW               62.400 usec
DE                6.50 usec
TE                296.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 91. ^{1}H NMR spectrum for 3k

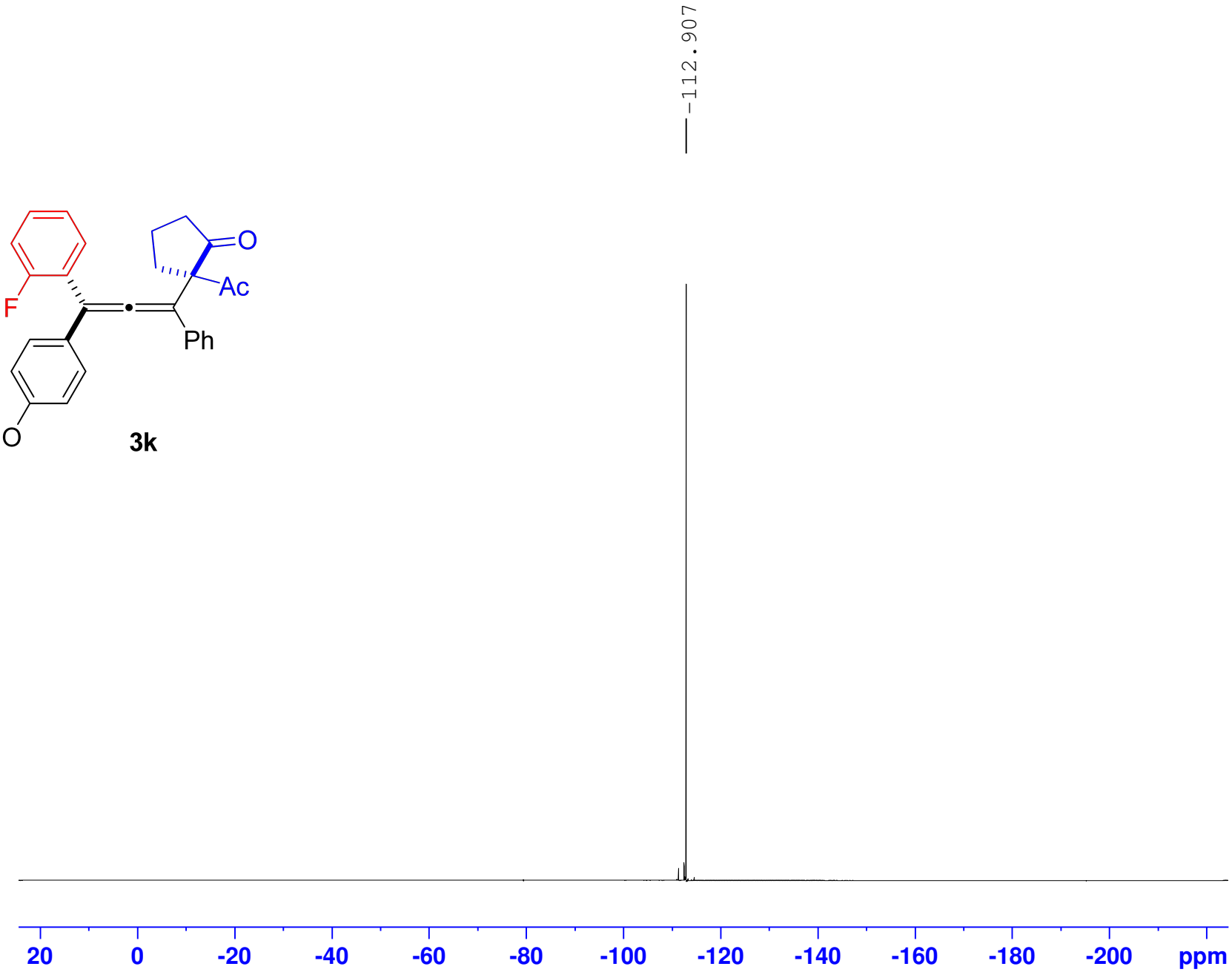



```
Current Data Parameters
NAME       qdy-20093-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160326
Time              13.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                   96
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           100.6127307 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 92. ^{13}C NMR spectrum for 3k

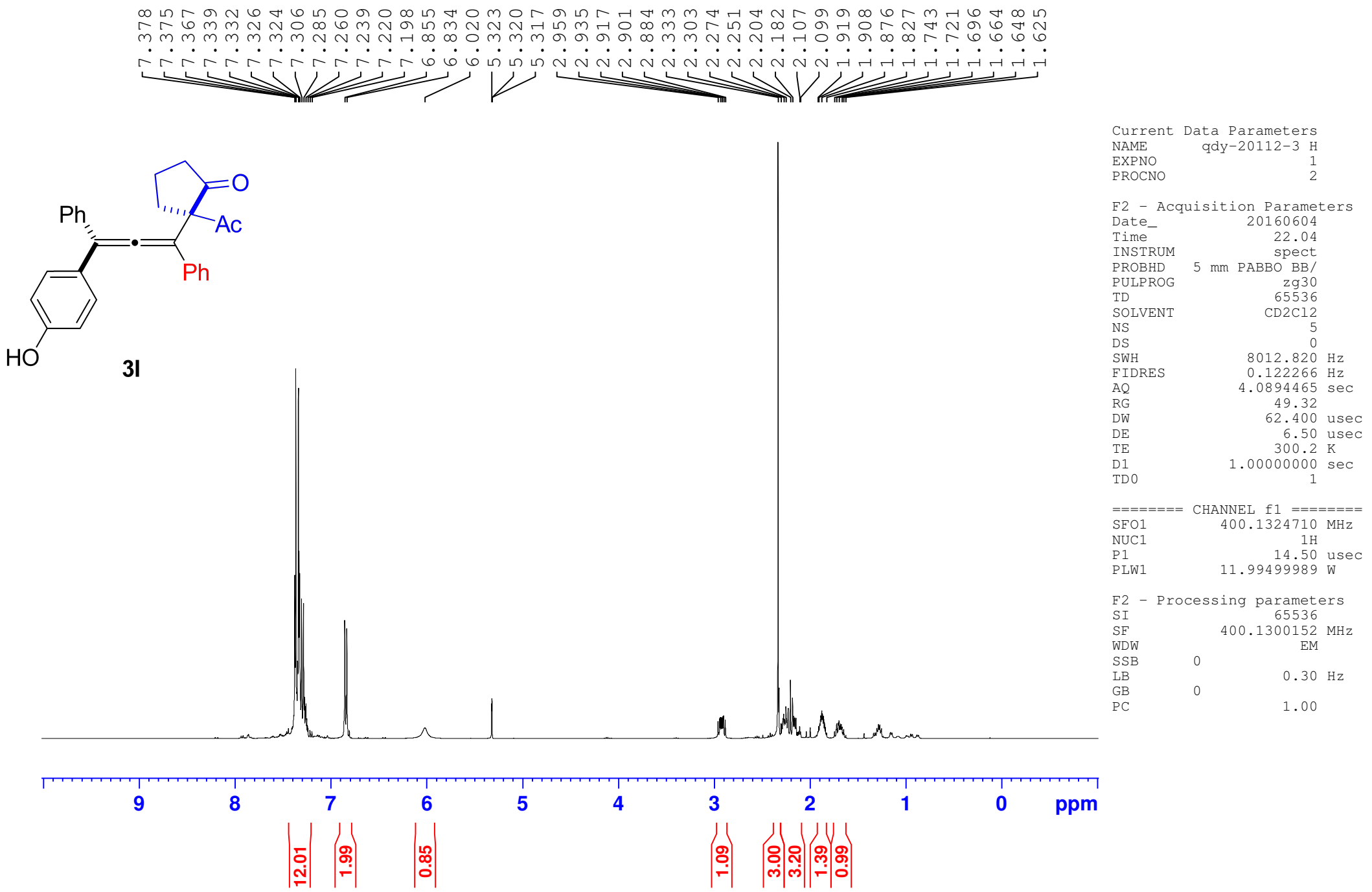


3k



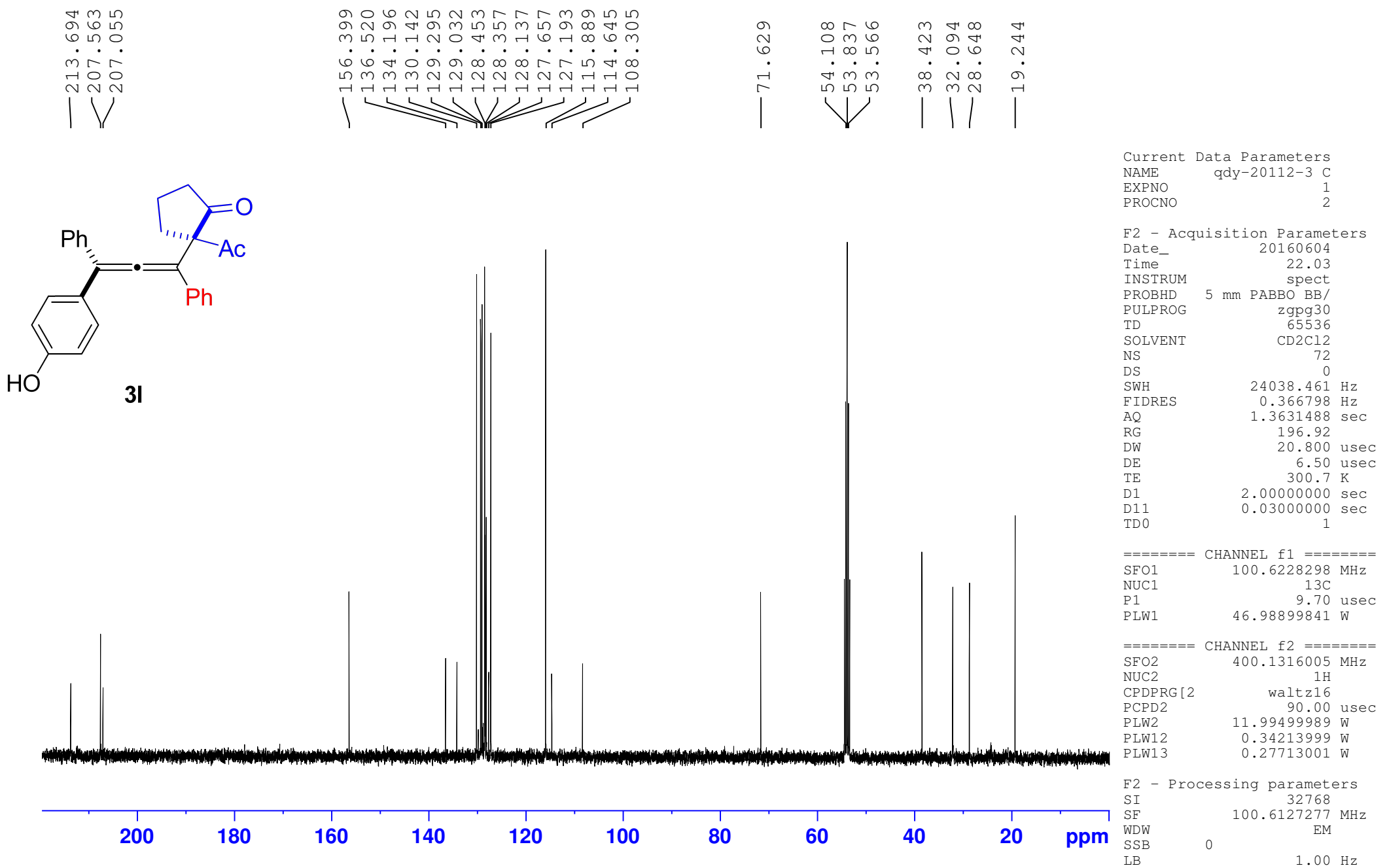

```
Current Data Parameters
NAME       qdy-20093-2 F
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160326
Time              13.56
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                   35
DS                    2
SWH           93750.000 Hz
FIDRES         1.430511 Hz
AQ            0.3495253 sec
RG               196.92
DW                5.333 usec
DE                6.50 usec
TE                297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         376.4607162 MHz
NUC1                19F
P1                14.70 usec
PLW1         15.99600029 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           376.4983660 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 93. ^{19}F NMR spectrum for 3k

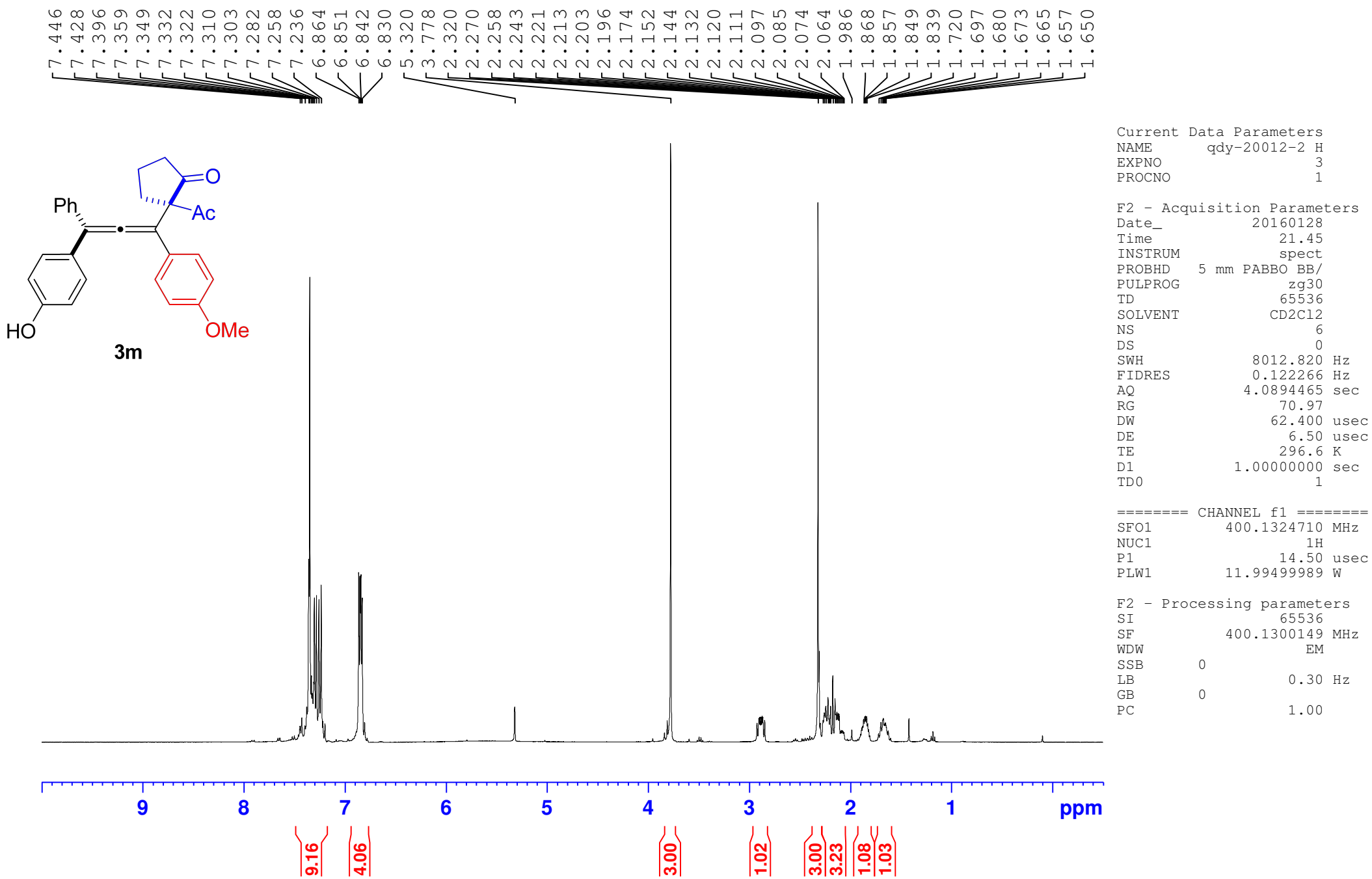



```
Current Data Parameters
NAME       qdy-20112-3 H
EXPNO                 1
PROCNO                2

F2 - Acquisition Parameters
Date_          20160604
Time              22.04
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    5
DS                    0
SWH            8012.820 Hz
FIDRES            0.122266 Hz
AQ             4.0894465 sec
RG                49.32
DW                62.400 usec
DE                 6.50 usec
TE                300.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 94. ^{1}H NMR spectrum for 3l

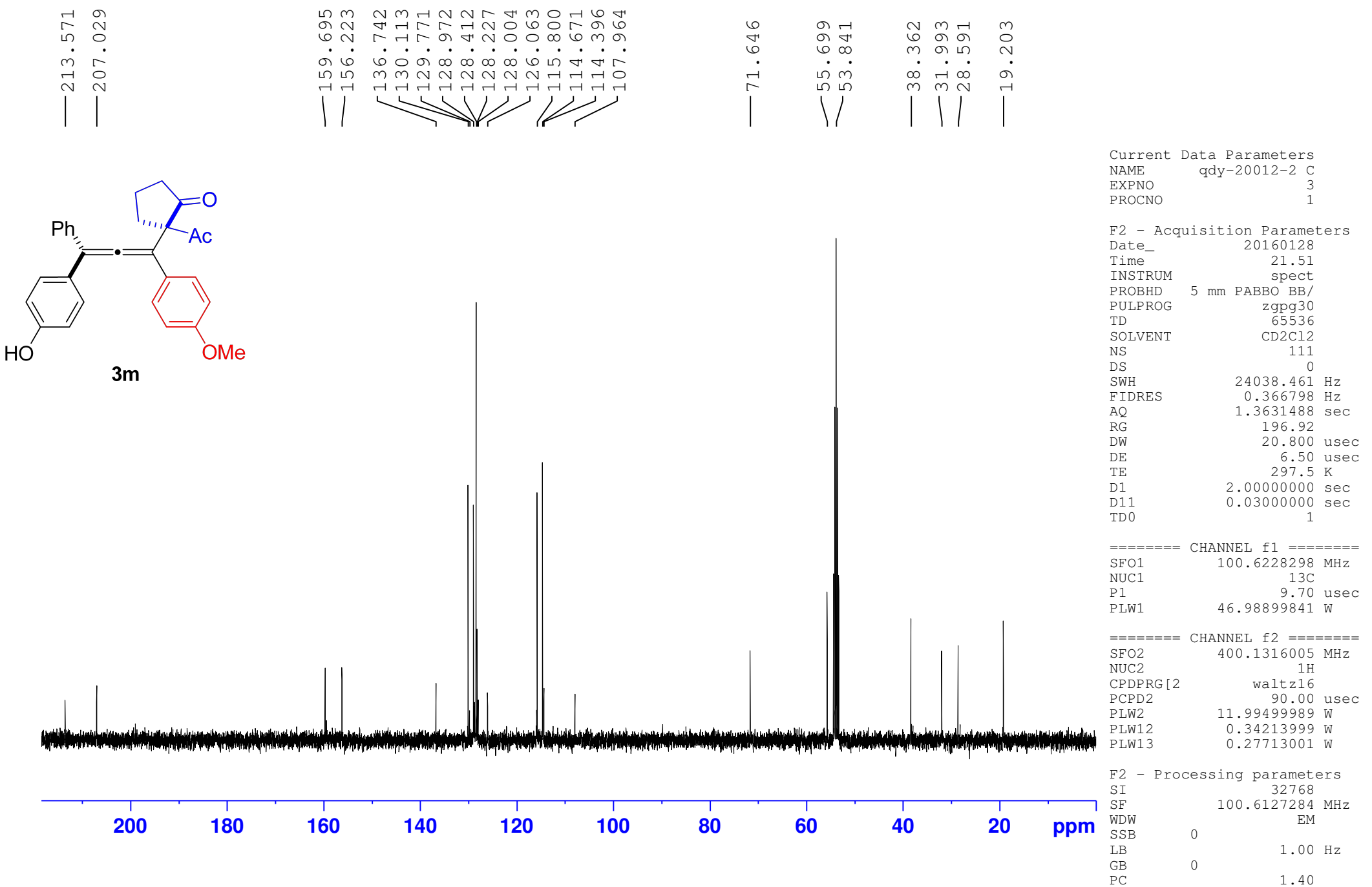


Supplementary Figure 95. ^{13}C NMR spectrum for 3l

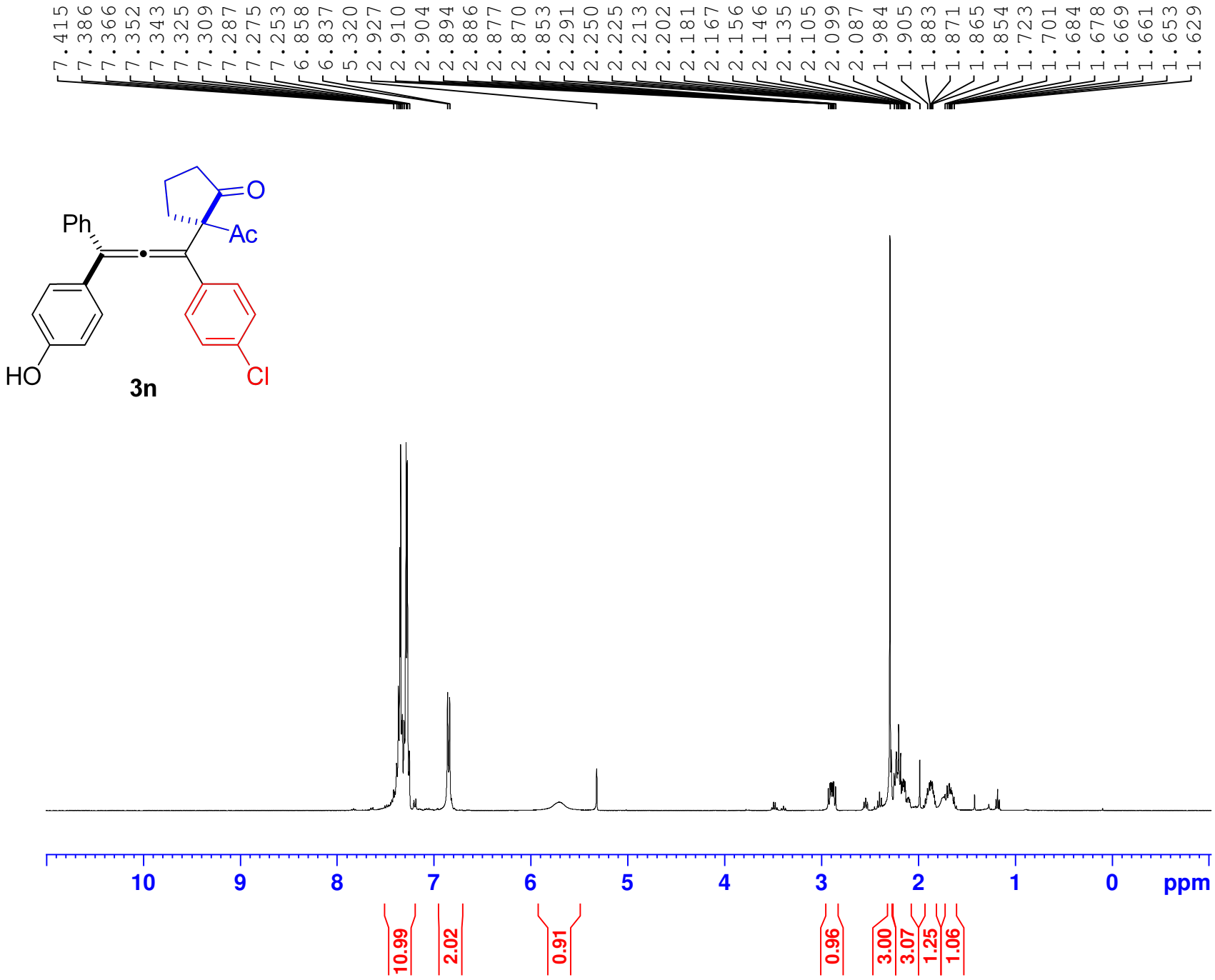



```
Current Data Parameters
NAME       qdy-20012-2 H
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160128
Time              21.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                70.97
DW                62.400 usec
DE                 6.50 usec
TE                296.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300149 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 96. ^{1}H NMR spectrum for 3m

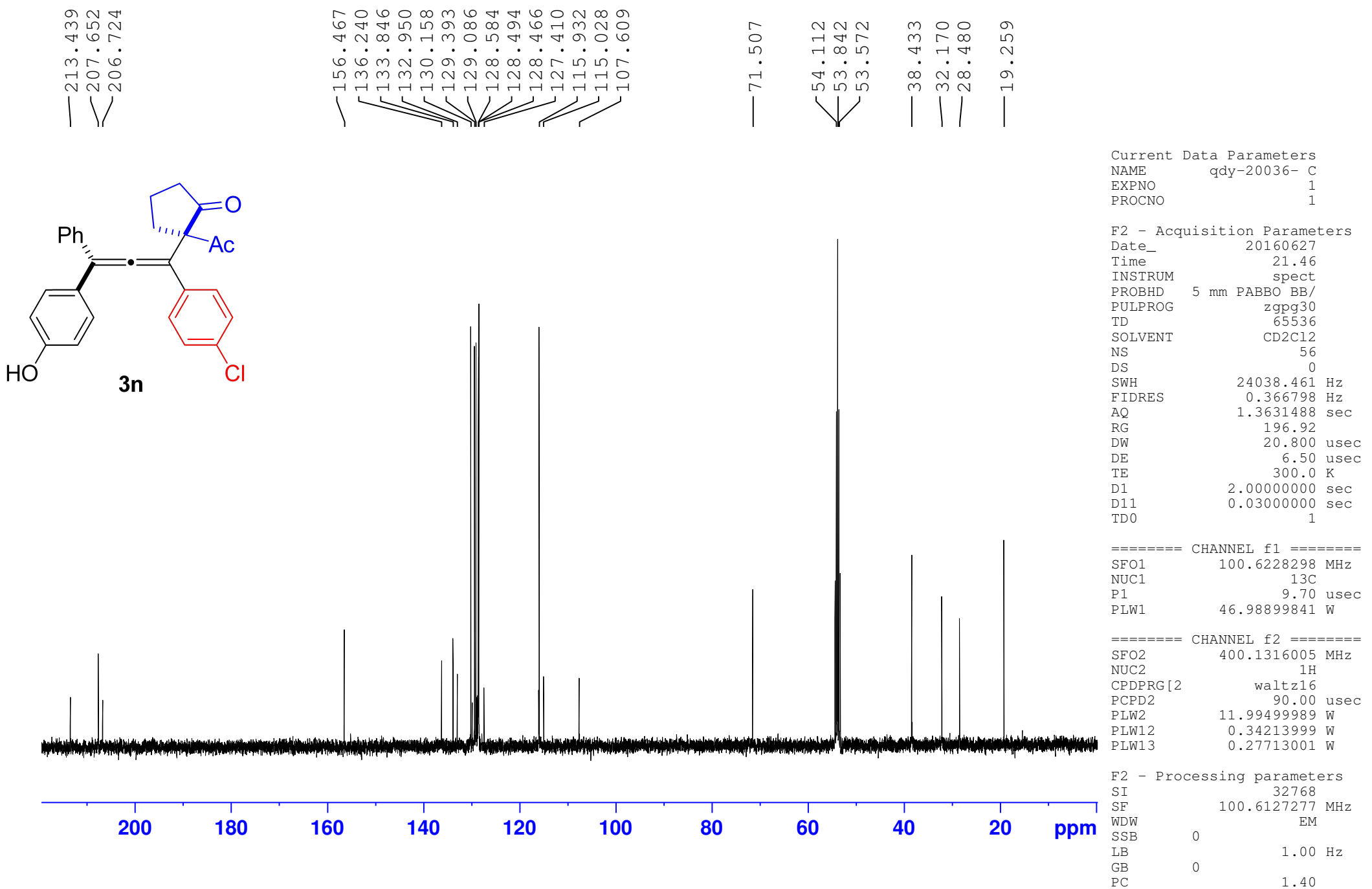


Supplementary Figure 97. ^{13}C NMR spectrum for 3m

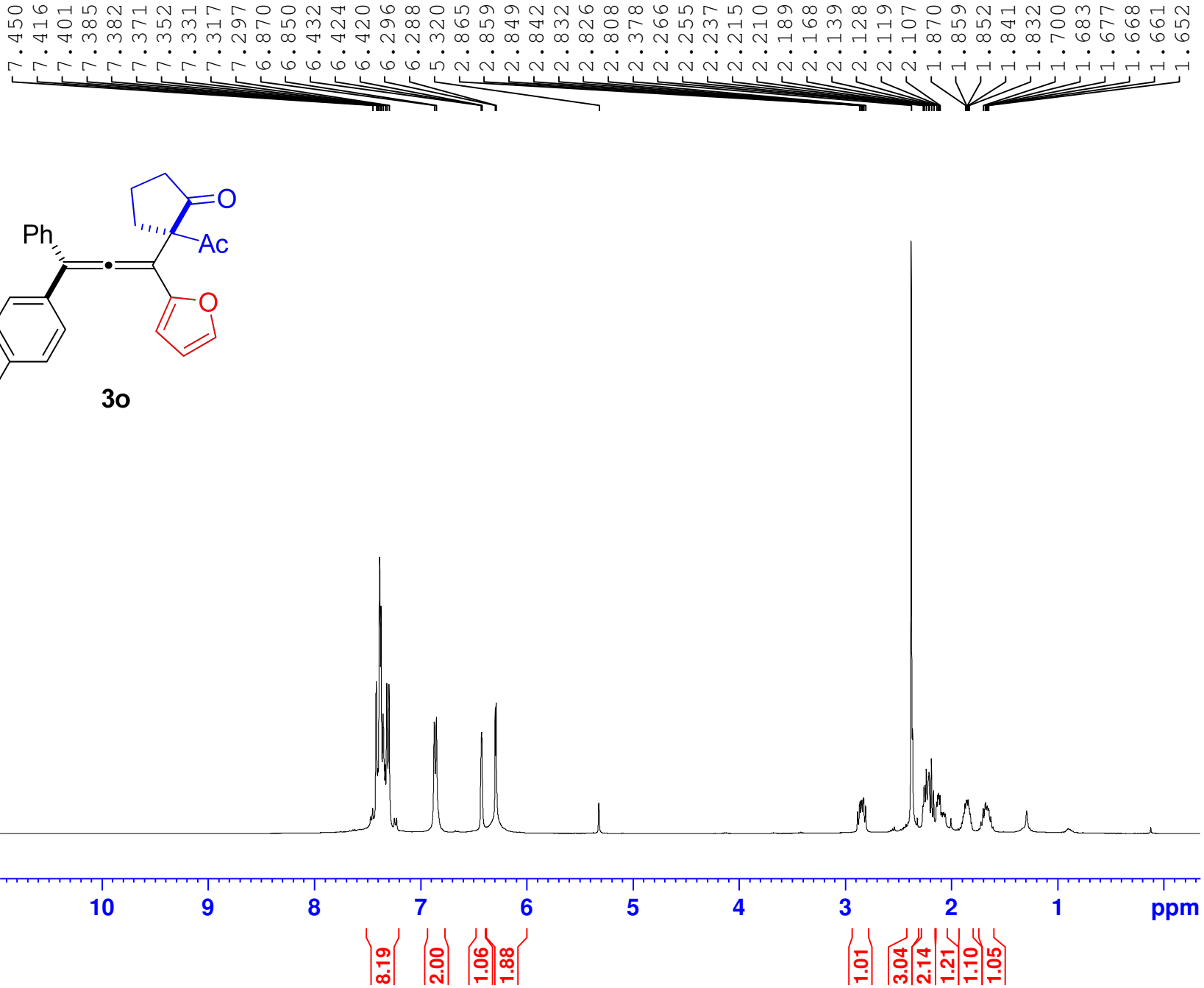



```
Current Data Parameters
NAME         qdy-20036 H
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160128
Time               22.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT          CD2Cl2
NS                     6
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 88.84
DW                62.400 usec
DE                 6.50 usec
TE                297.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                 65536
SF           400.1300154 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Figure 98. ^{1}H NMR spectrum for 3n

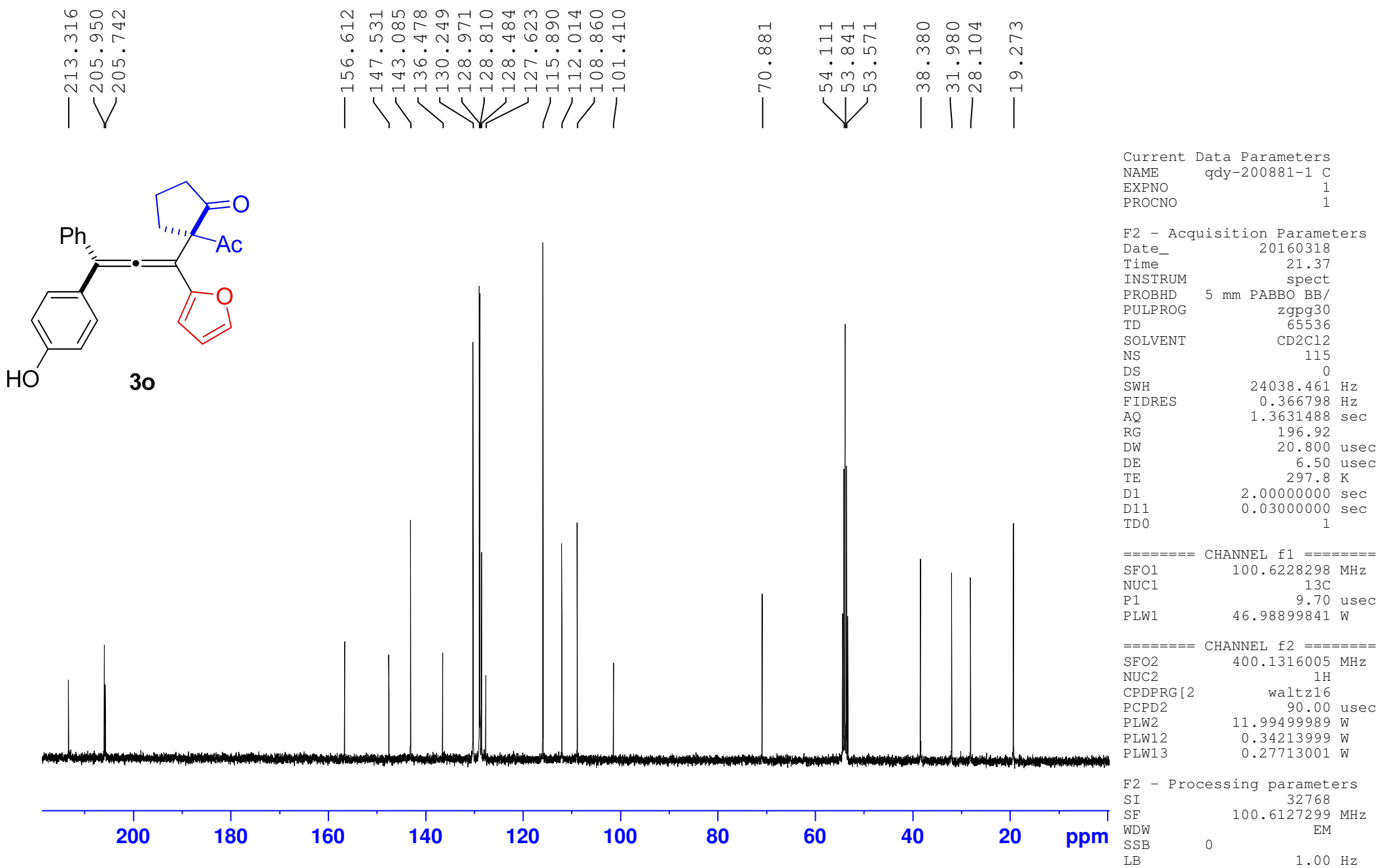


Supplementary Figure 99. ^{13}C NMR spectrum for 3n




```
Current Data Parameters
NAME      qdy-200881-1 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160318
Time              21.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                   10
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                34.77
DW               62.400 usec
DE                6.50 usec
TE                296.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                  1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1300149 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 100. ^{1}H NMR spectrum for 3o



Current Data Parameters
NAME qdy-200881-1 C
EXPNO 1
PROCNO 1

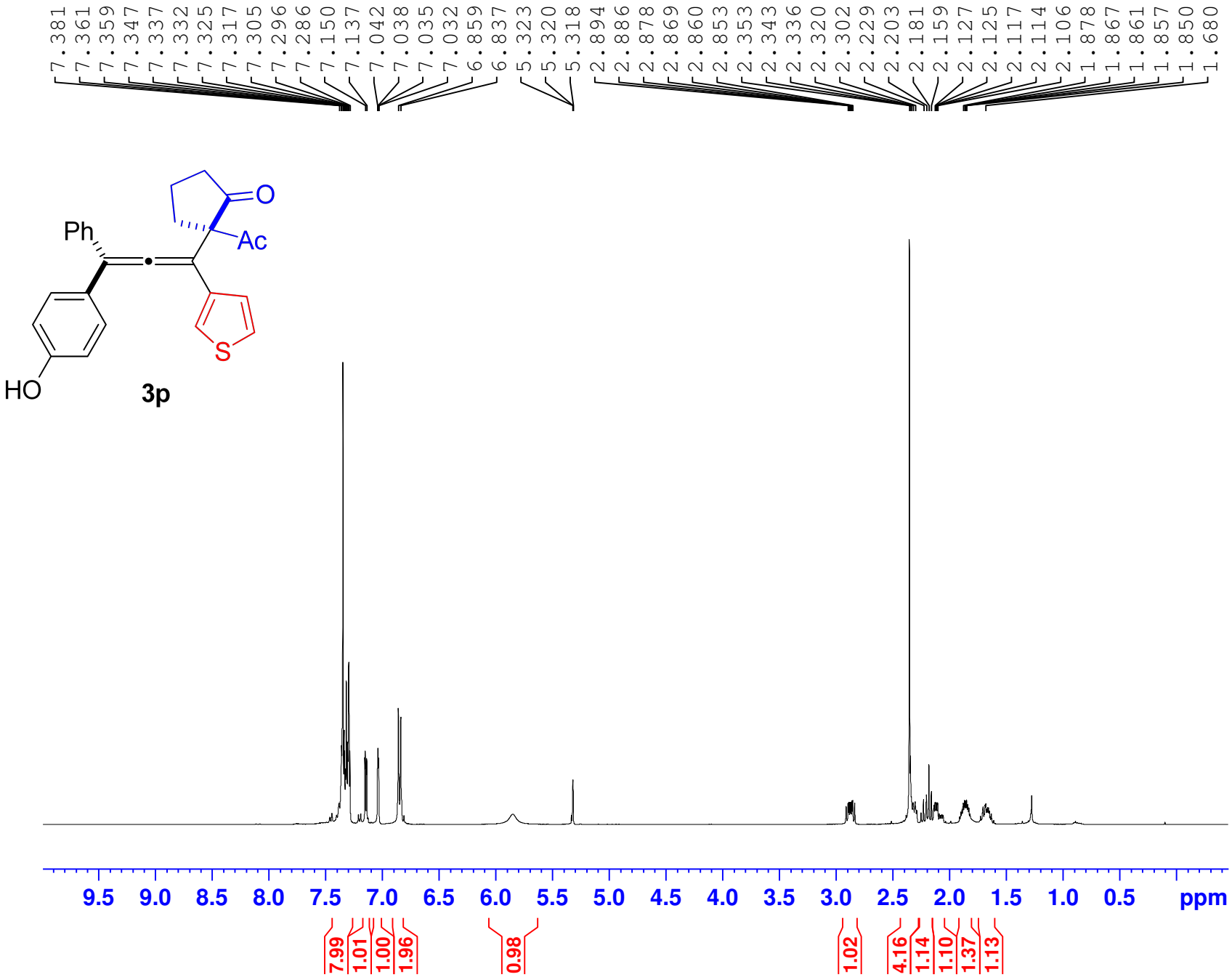
F2 - Acquisition Parameters
Date_ 20160318
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 115
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

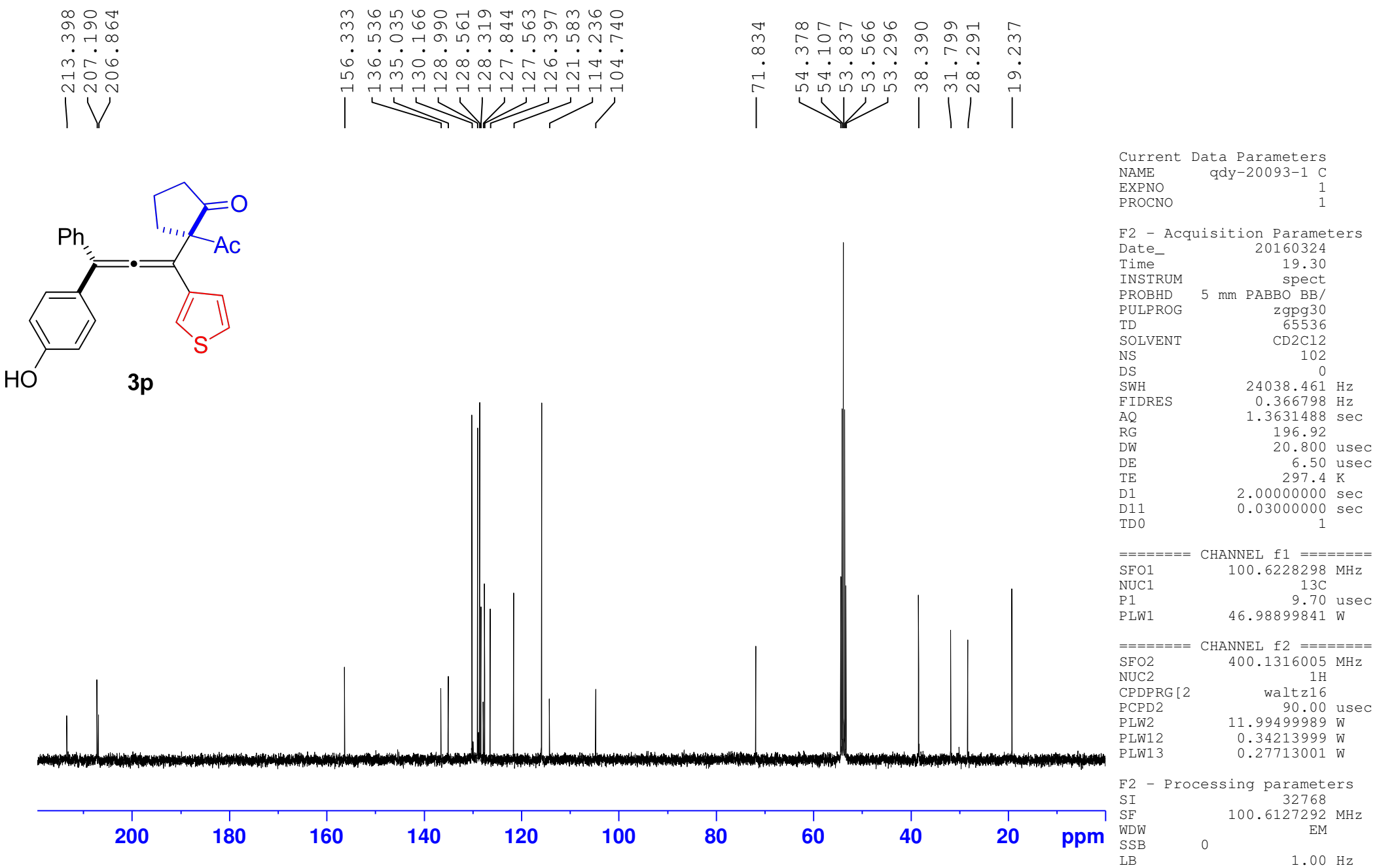
F2 - Processing parameters
SI 32768
SF 100.6127299 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 101. ^{13}C NMR spectrum for 3o




```
Current Data Parameters
NAME       qdy-20093-1 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160324
Time              19.25
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                54.81
DW                62.400 usec
DE                 6.50 usec
TE                296.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 102. ^{1}H NMR spectrum for 3p



Current Data Parameters
NAME qdy-20093-1 C
EXPNO 1
PROCNO 1

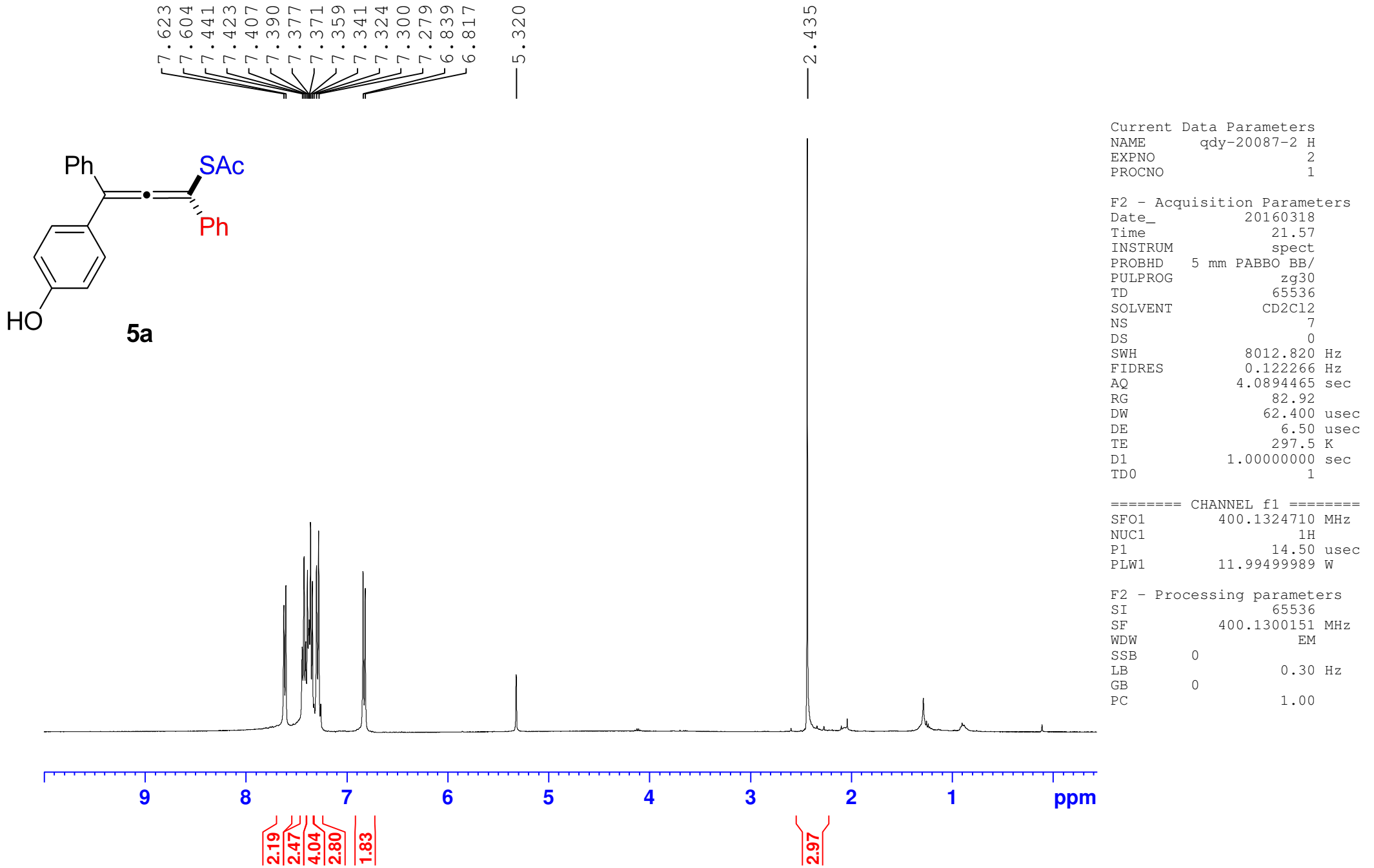
F2 - Acquisition Parameters
Date_ 20160324
Time 19.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 102
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

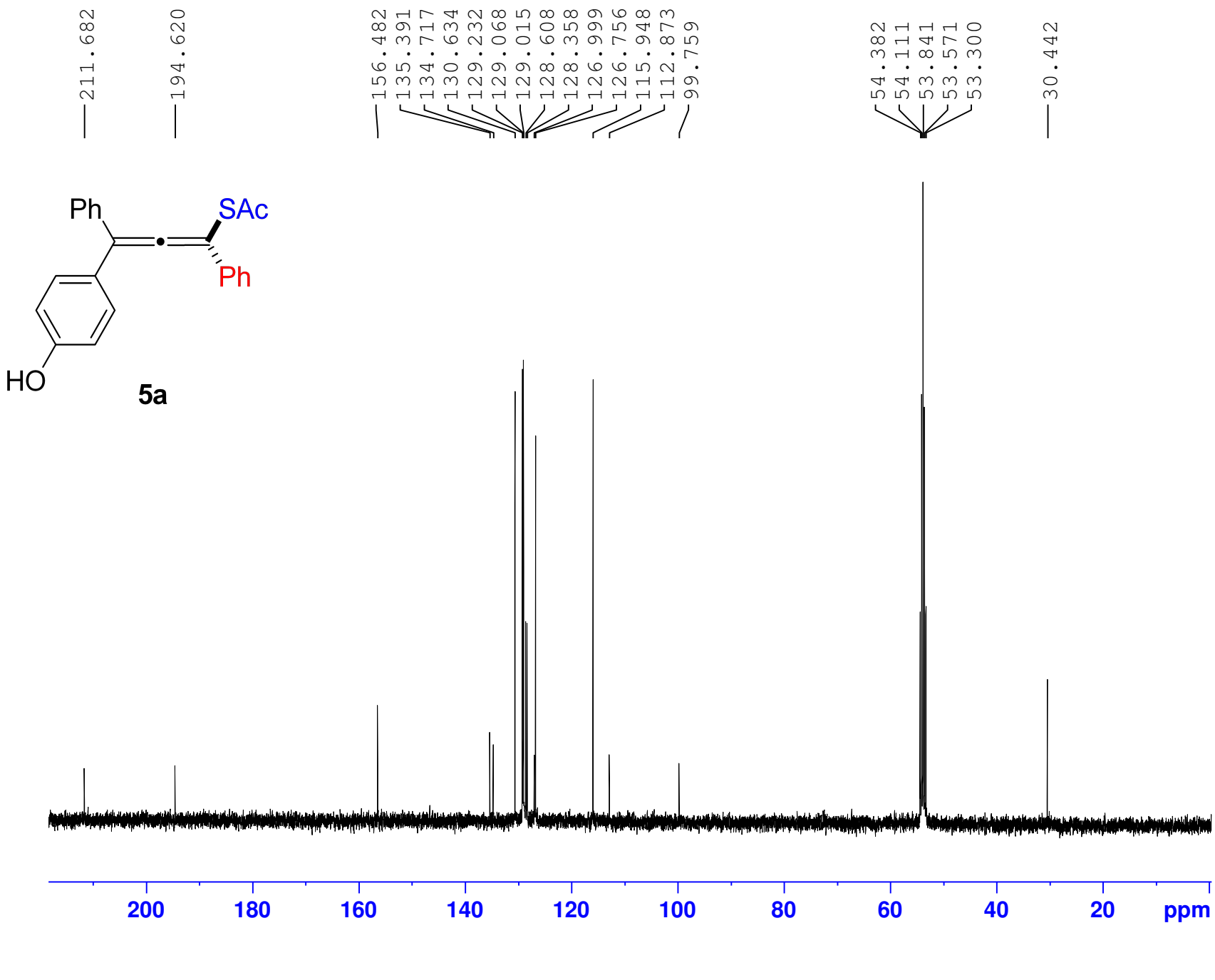
F2 - Processing parameters
SI 32768
SF 100.6127292 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Supplementary Figure 103. ^{13}C NMR spectrum for 3p



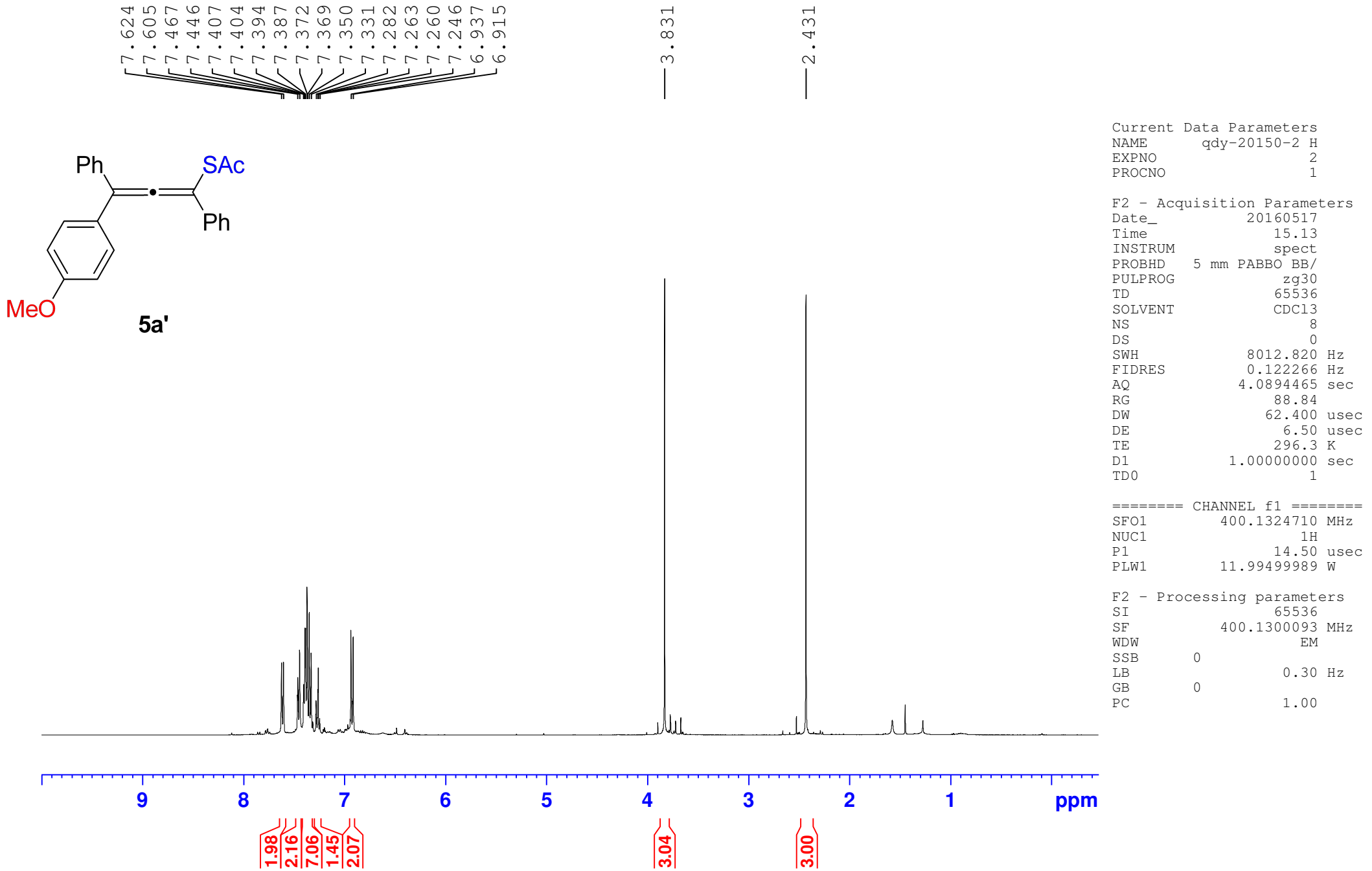

```
Current Data Parameters
NAME       qdy-20087-2 H
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160318
Time              21.57
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    7
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                82.92
DW                62.400 usec
DE                 6.50 usec
TE                297.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300151 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 104. ^{1}H NMR spectrum for 5a

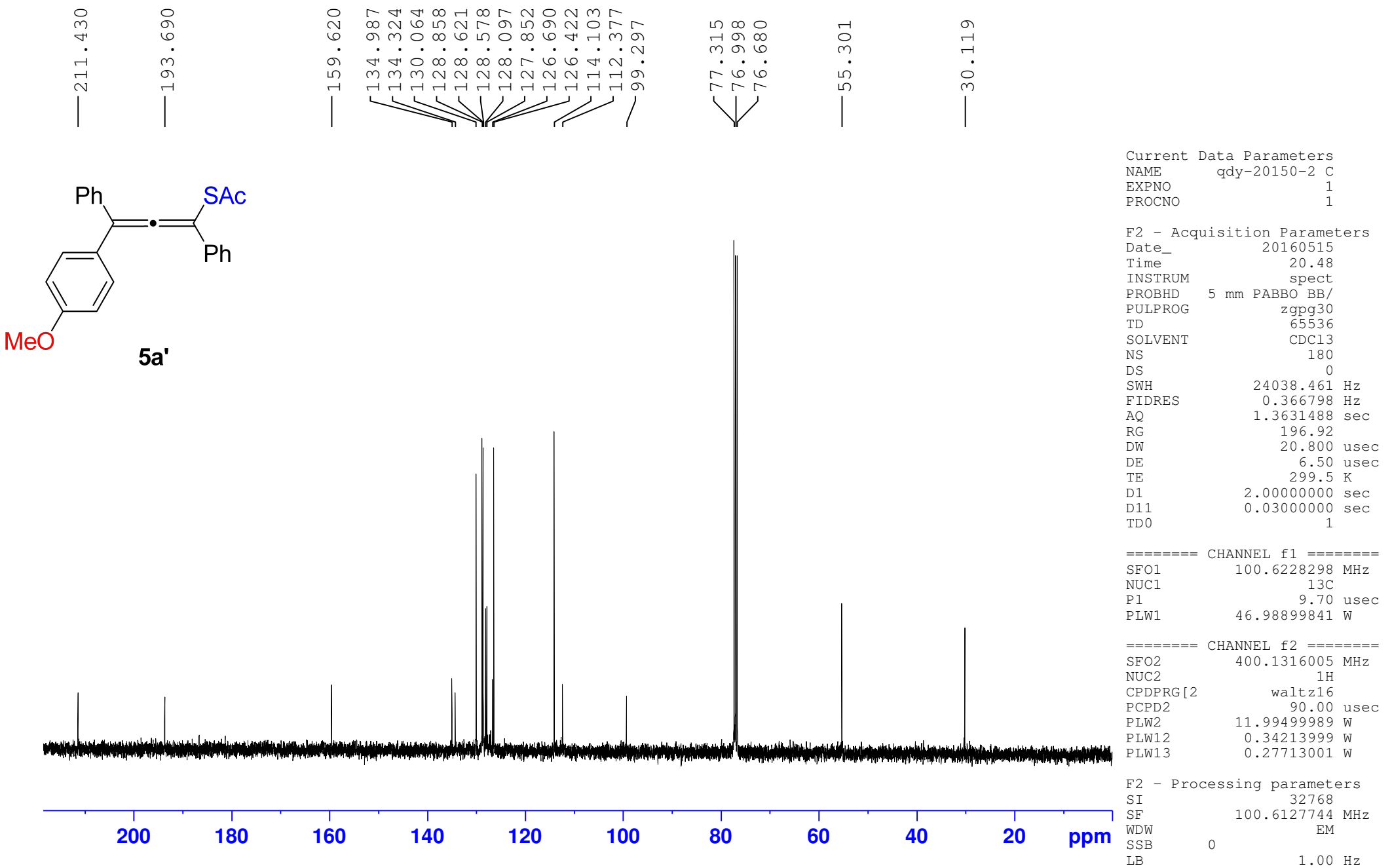



```
Current Data Parameters
NAME       qdy-20087-2 C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160318
Time              22.04
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                  168
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127277 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 105. ^{13}C NMR spectrum for 5a

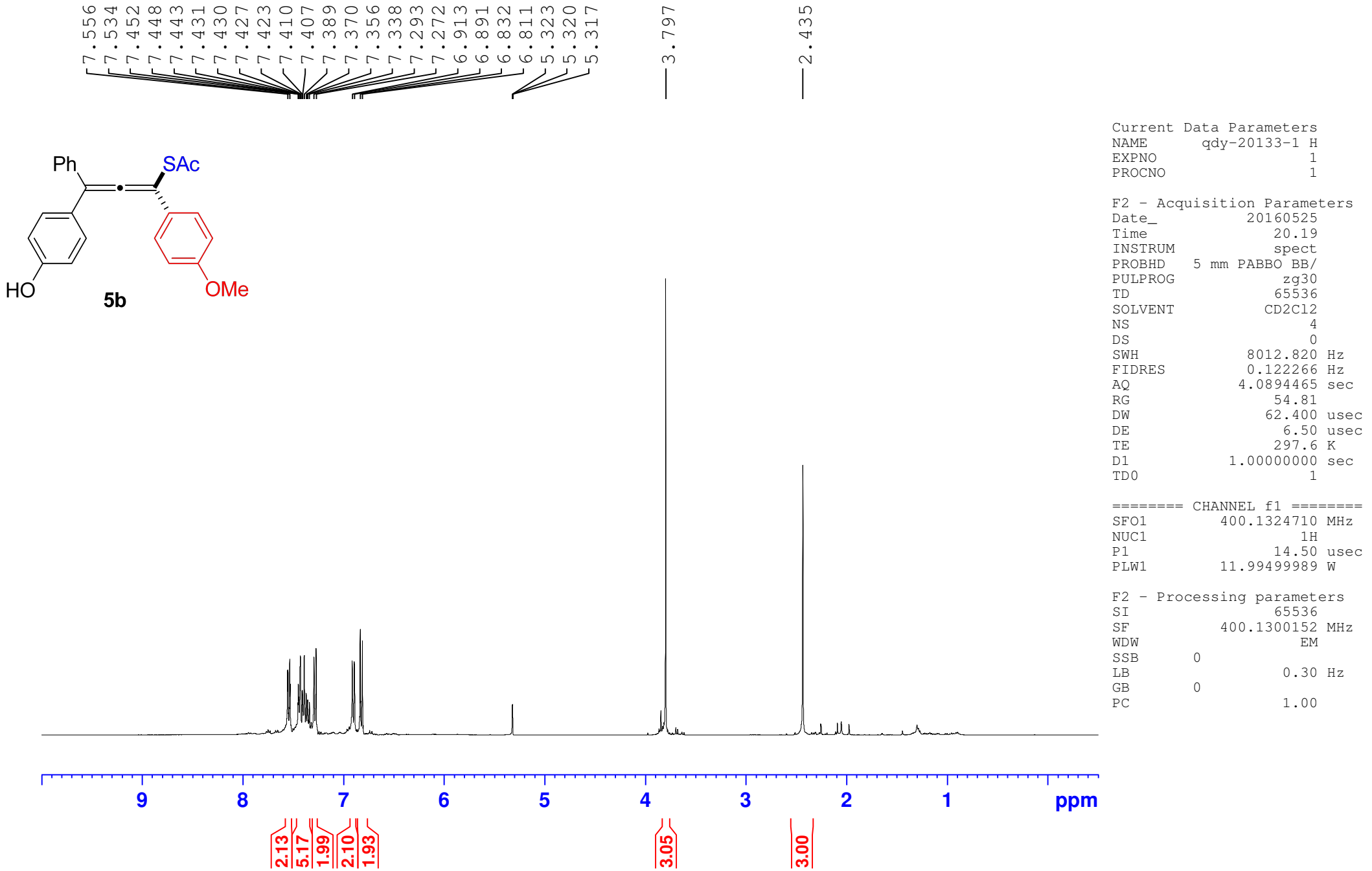


Supplementary Figure 106. ^{1}H NMR spectrum for 5a'




```
Current Data Parameters
NAME      qdy-20150-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160515
Time             20.48
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 180
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                299.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127744 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 107. ^{13}C NMR spectrum for 5a'



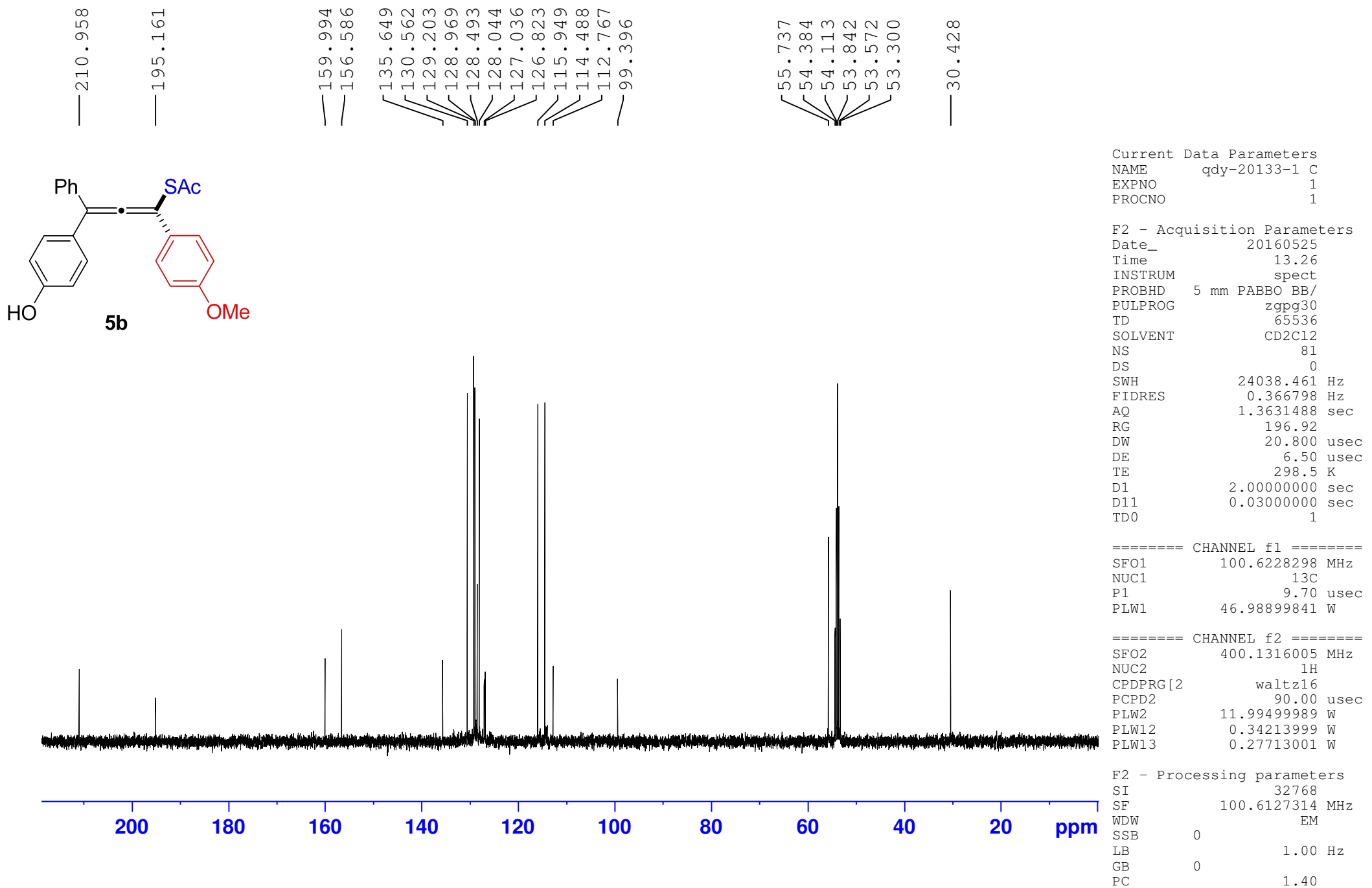
Current Data Parameters
NAME qdy-20133-1 H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160525
Time 20.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

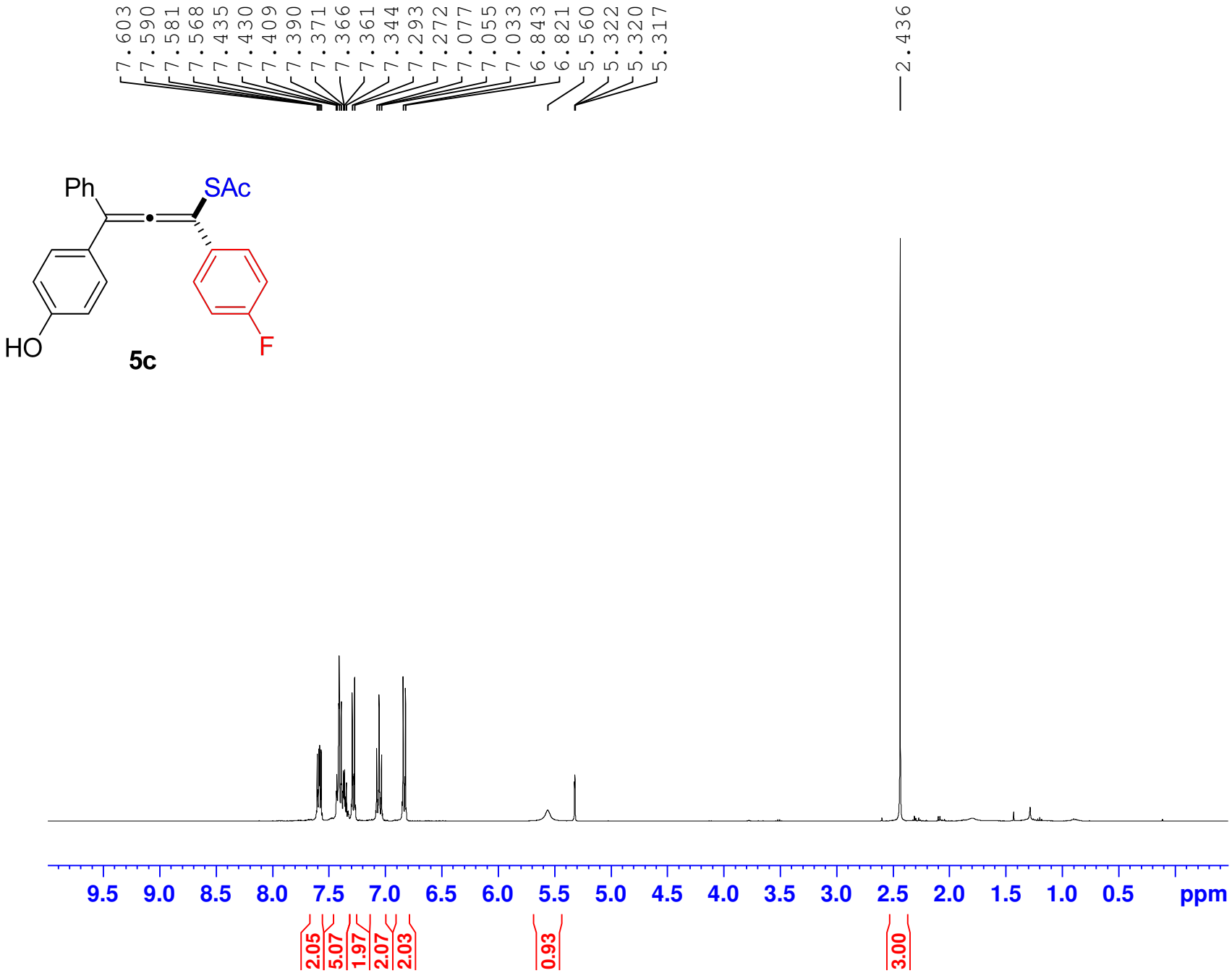
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

F2 - Processing parameters
SI 65536
SF 400.1300152 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

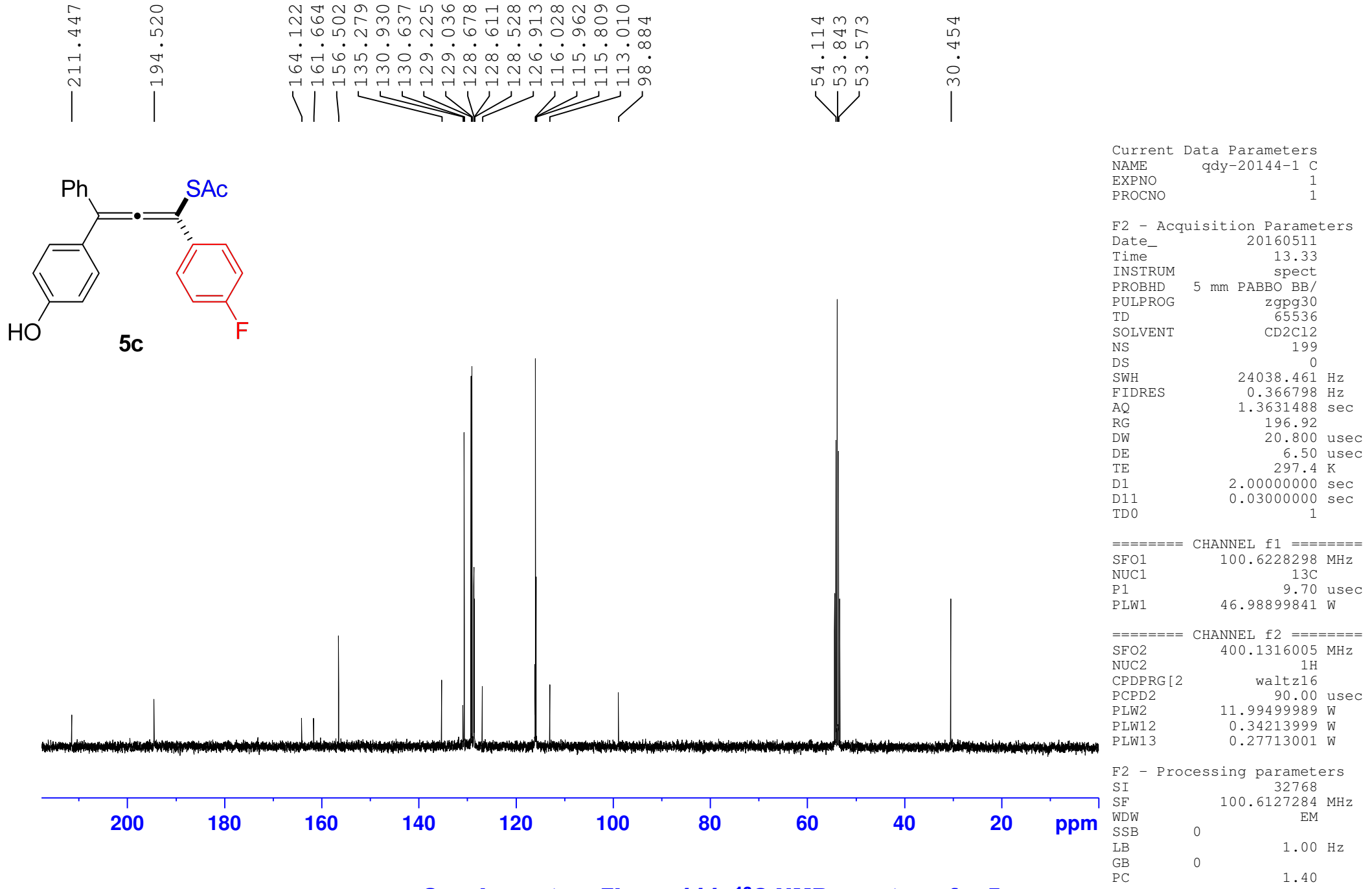
Supplementary Figure 108. ^{1}H NMR spectrum for 5b



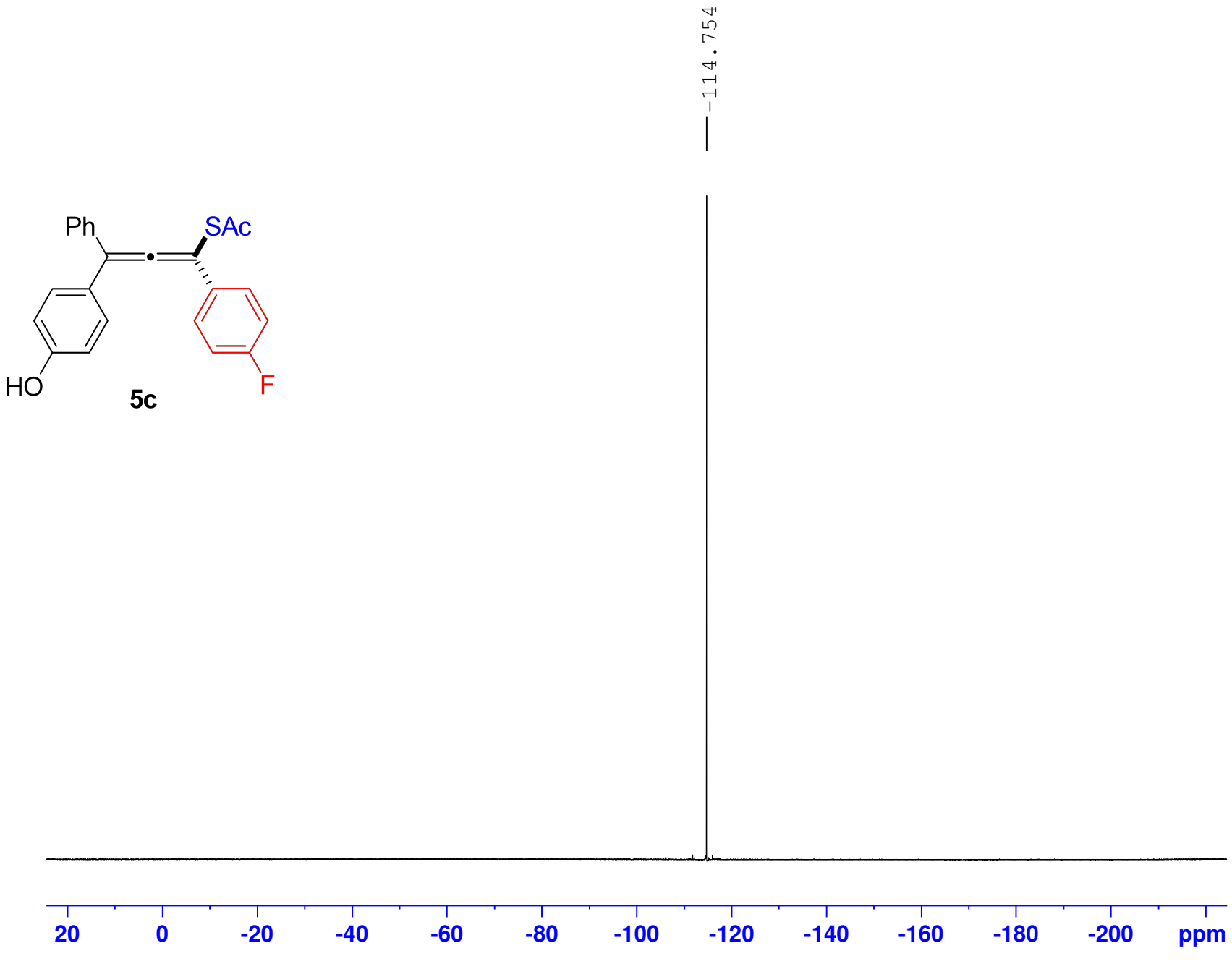
Supplementary Figure 109. ^{13}C NMR spectrum for 5b



Supplementary Figure 110. ^{1}H NMR spectrum for 5c

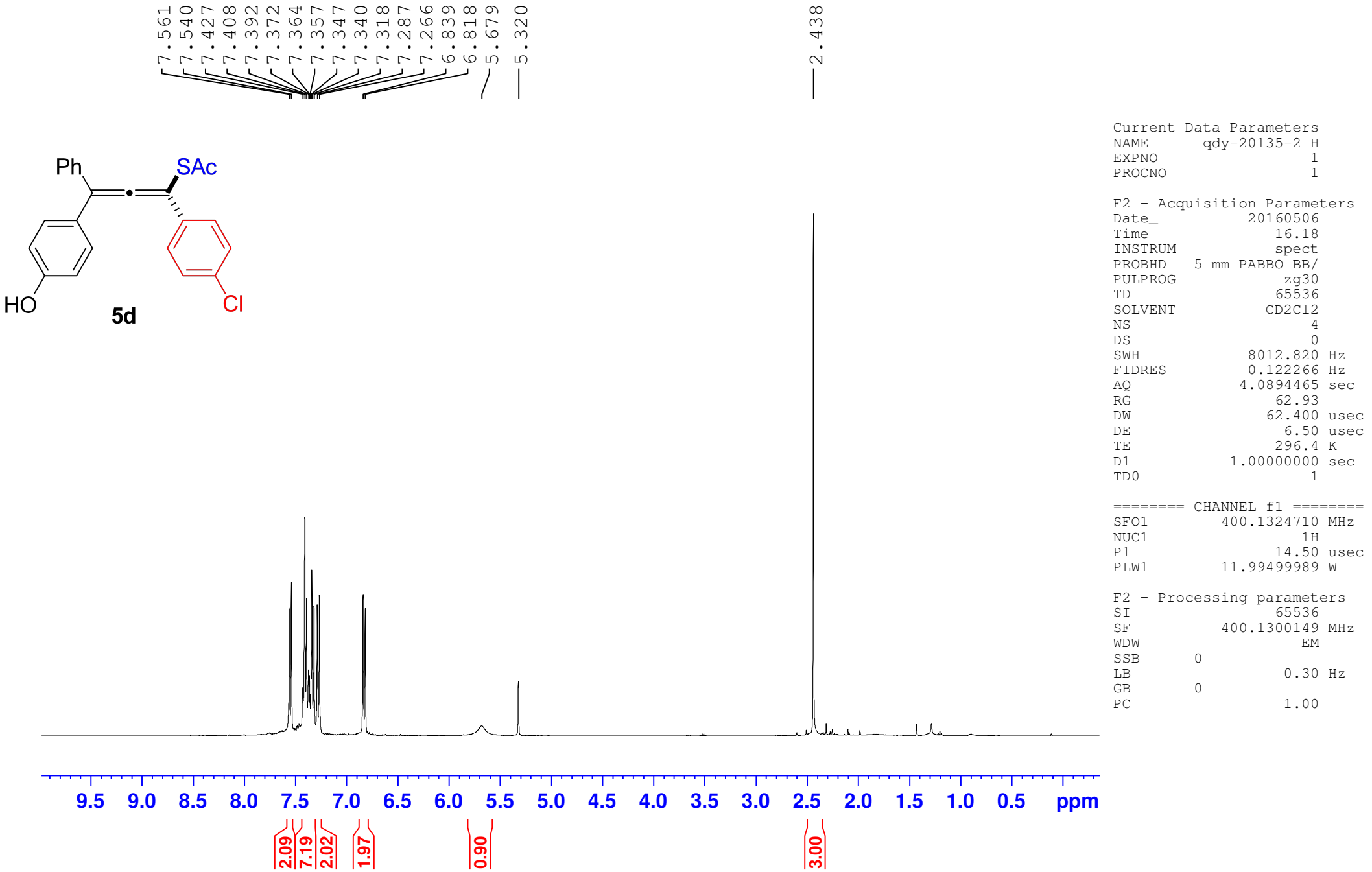


Supplementary Figure 111. ^{13}C NMR spectrum for 5c



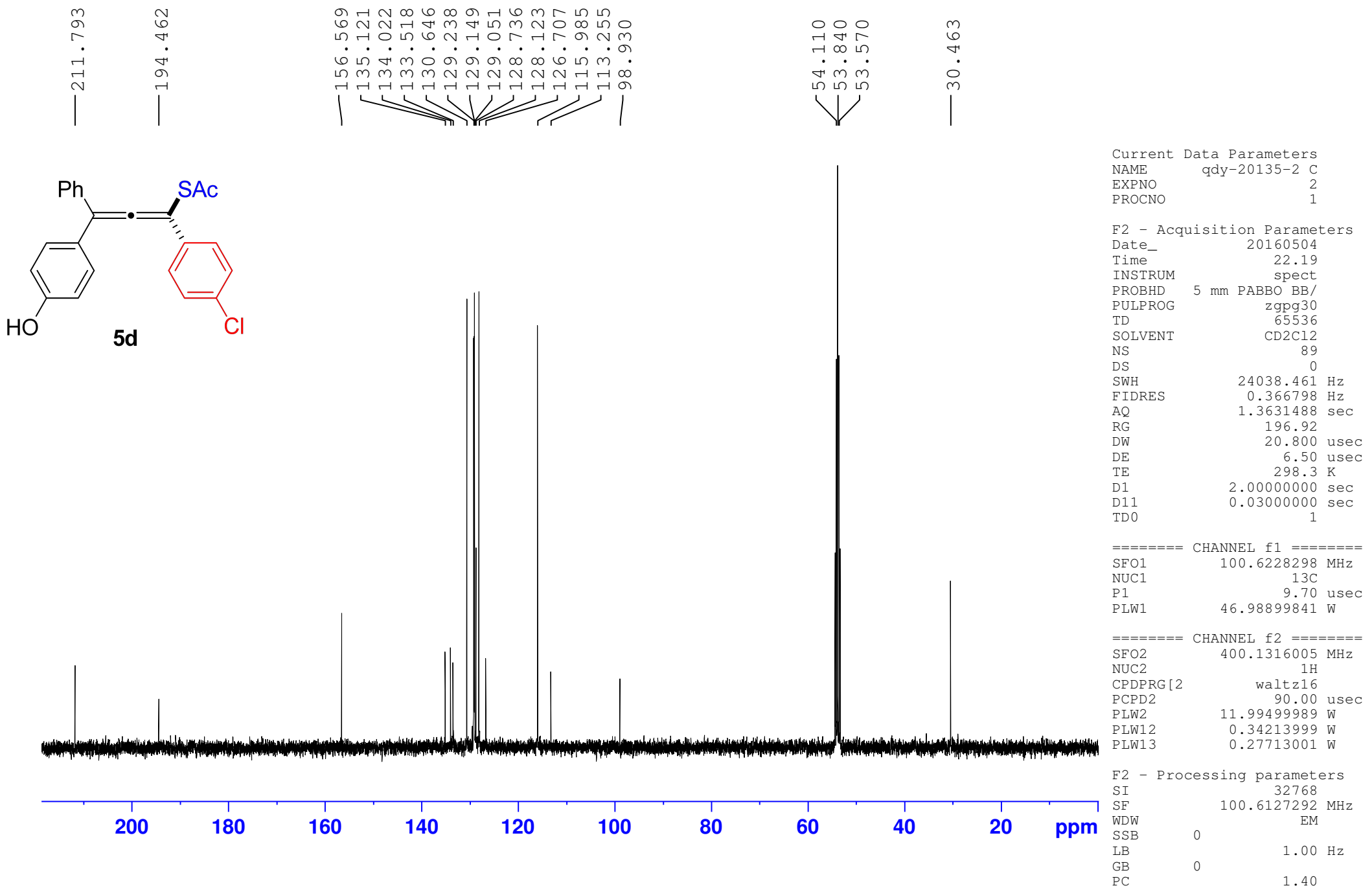

```
Current Data Parameters
NAME      qdy-20144-1 F
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20160511
Time               13.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                    4
DS                    0
SWH           93750.000 Hz
FIDRES         1.430511 Hz
AQ            0.3495253 sec
RG               196.92
DW                5.333 usec
DE                6.50 usec
TE               296.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         376.4607162 MHz
NUC1                19F
P1                14.70 usec
PLW1         15.99600029 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           376.4983660 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 112. ^{19}F NMR spectrum for 5c

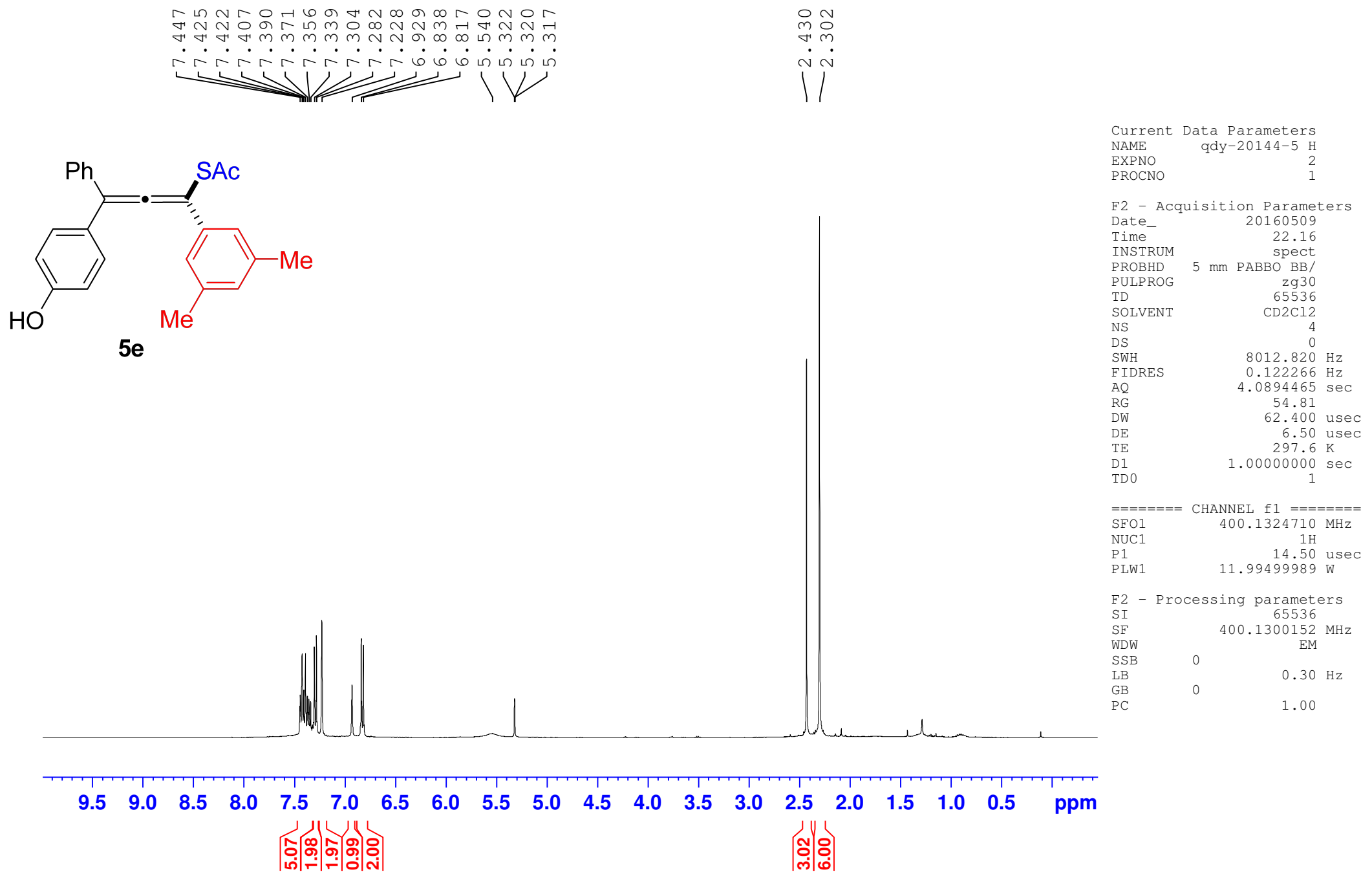



```
Current Data Parameters
NAME       qdy-20135-2 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160506
Time              16.18
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CD2Cl2
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                62.93
DW                62.400 usec
DE                 6.50 usec
TE                296.4 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300149 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

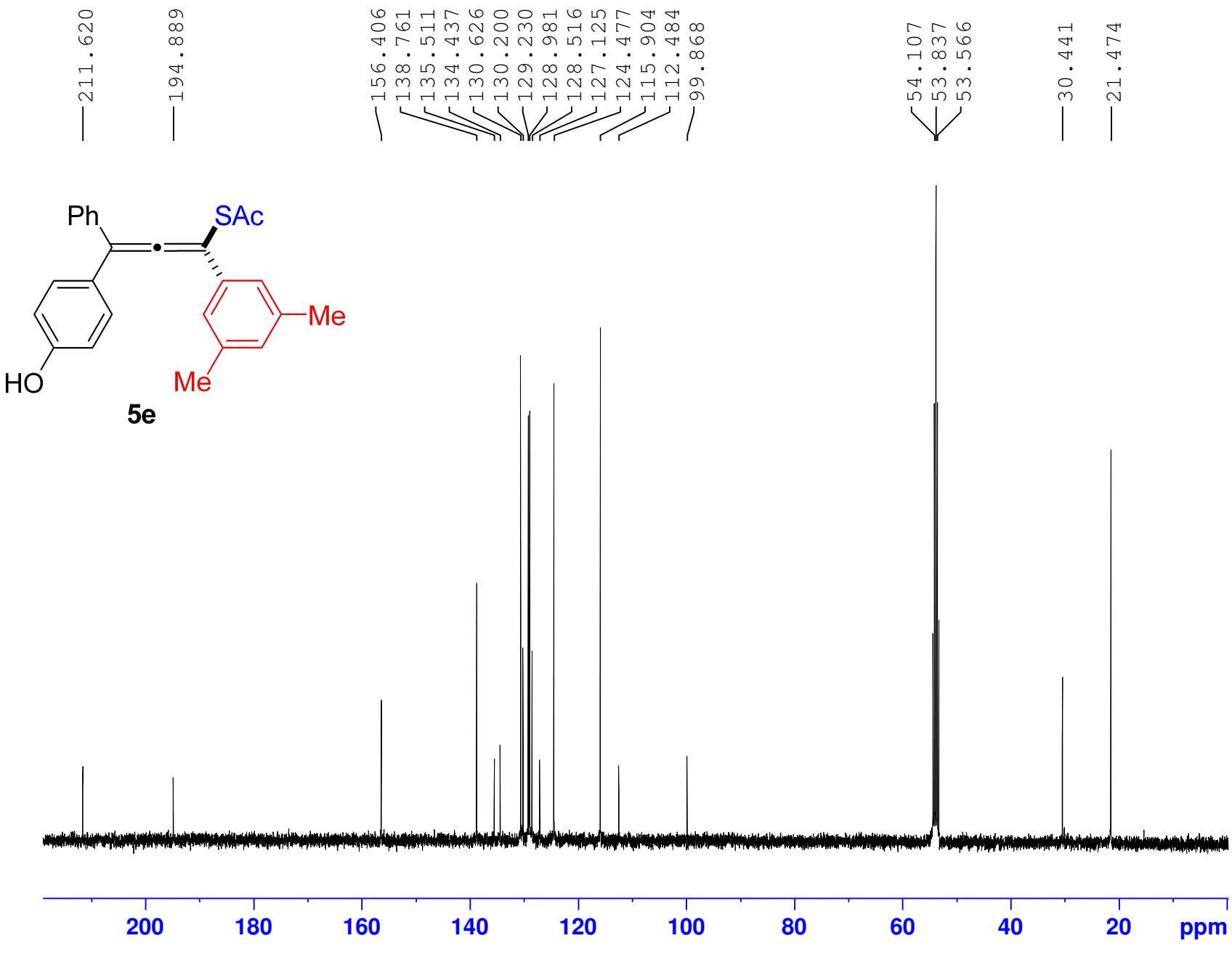
Supplementary Figure 113. ^{1}H NMR spectrum for 5d



Supplementary Figure 114. ^{13}C NMR spectrum for 5d

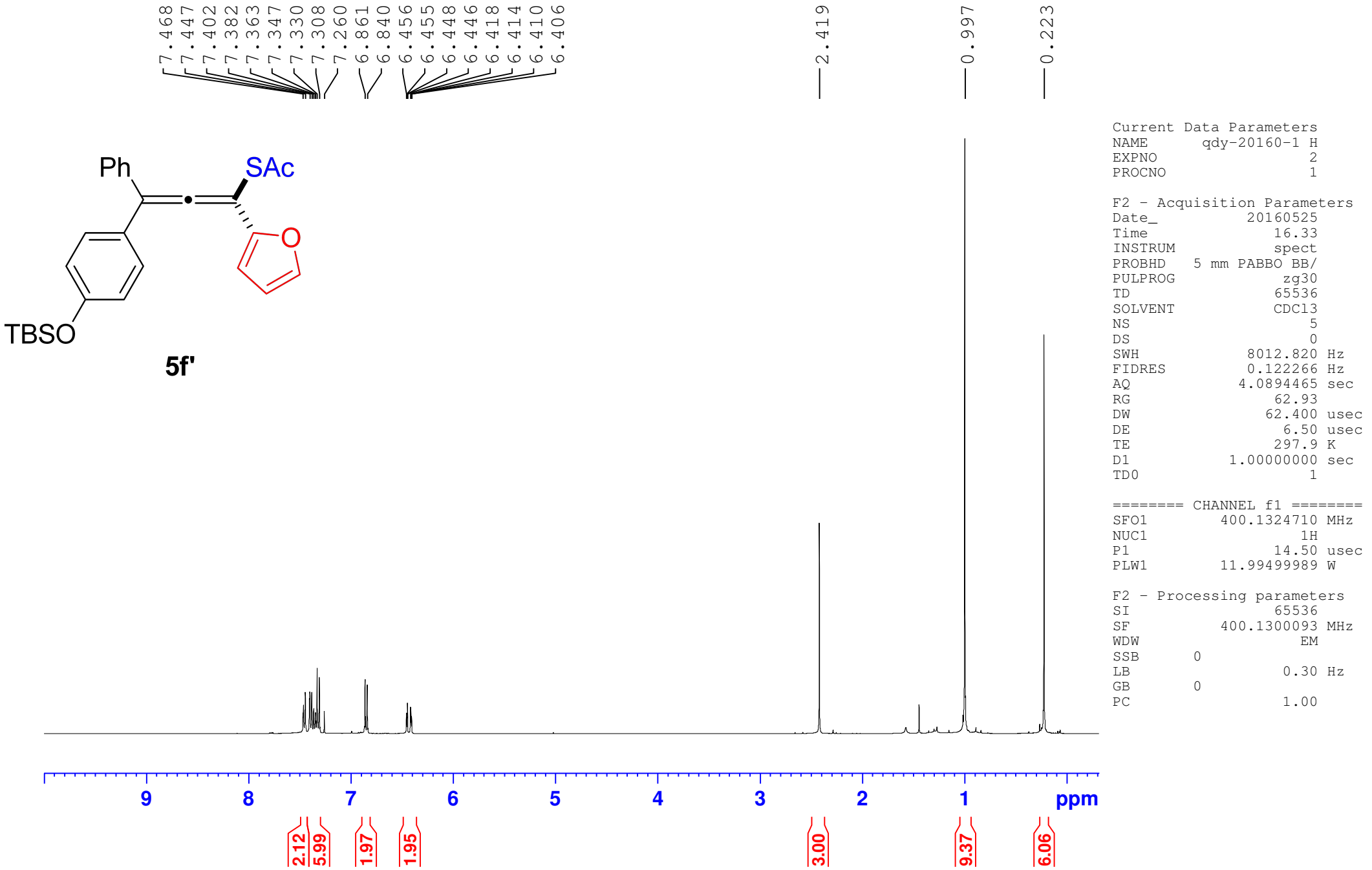


Supplementary Figure 115. ^{1}H NMR spectrum for 5e

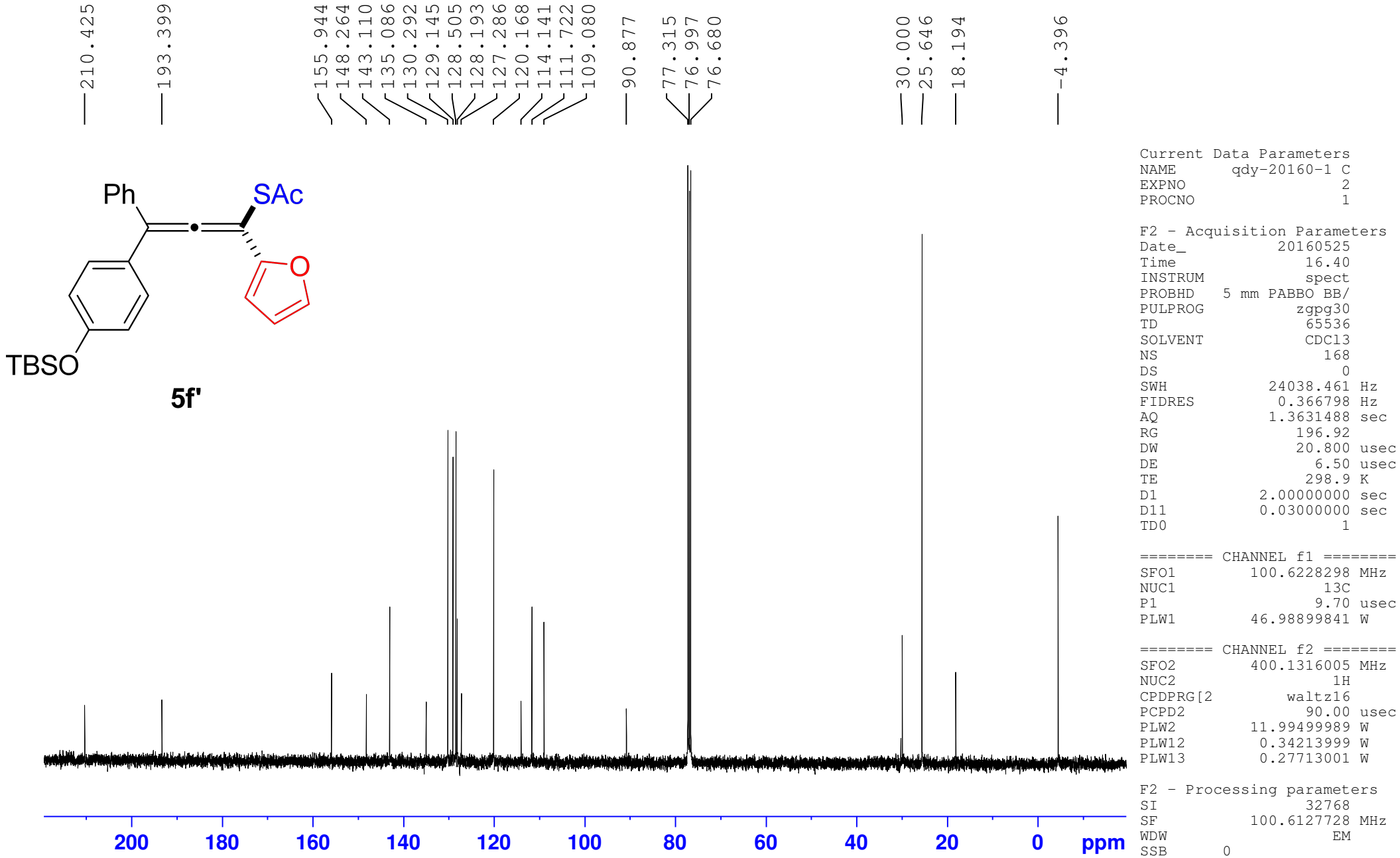



```
Current Data Parameters
NAME       qdy-20144-5 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160509
Time              20.02
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                  176
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127292 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 116. ^{13}C NMR spectrum for 5e

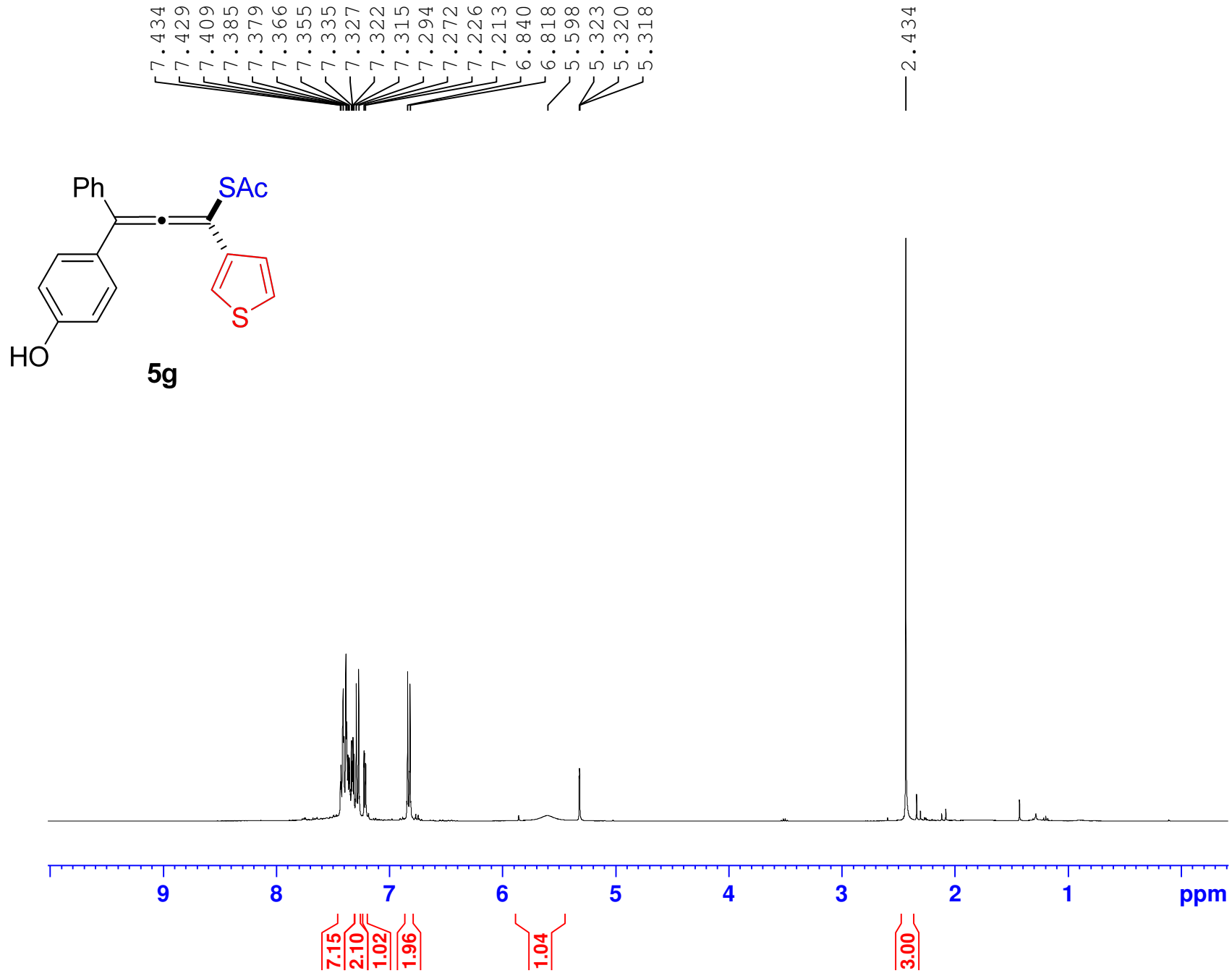


Supplementary Figure 117. ^{1}H NMR spectrum for 5f'

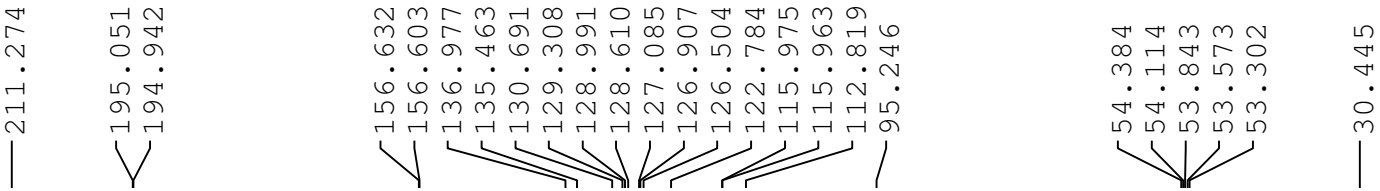


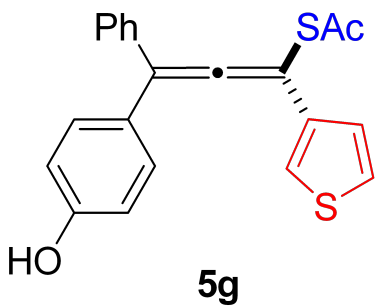

```
Current Data Parameters
NAME       qdy-20160-1 C
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20160525
Time              16.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  168
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127728 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 118. ^{13}C NMR spectrum for 5f'

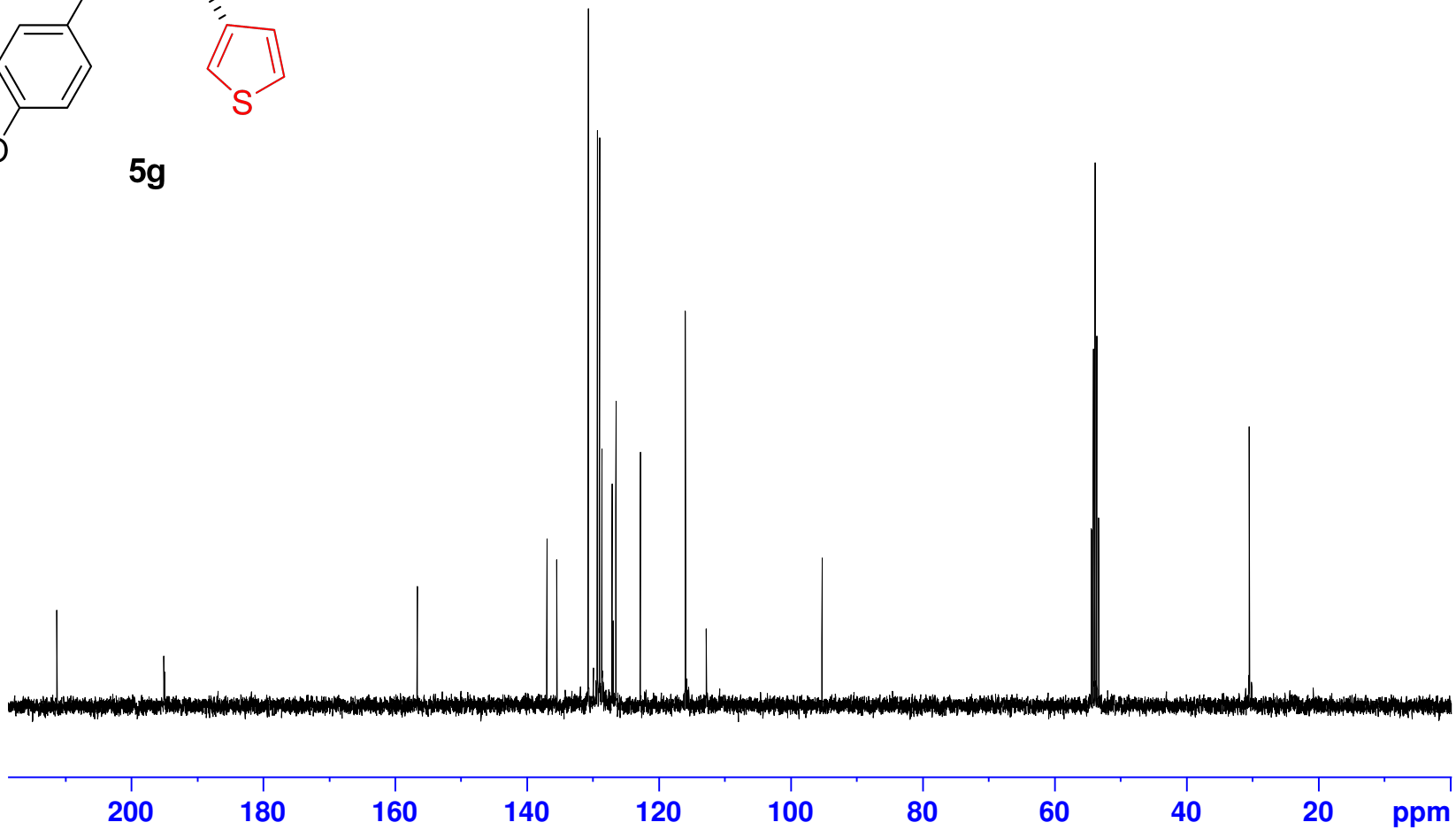


Supplementary Figure 119. ^{1}H NMR spectrum for 5g



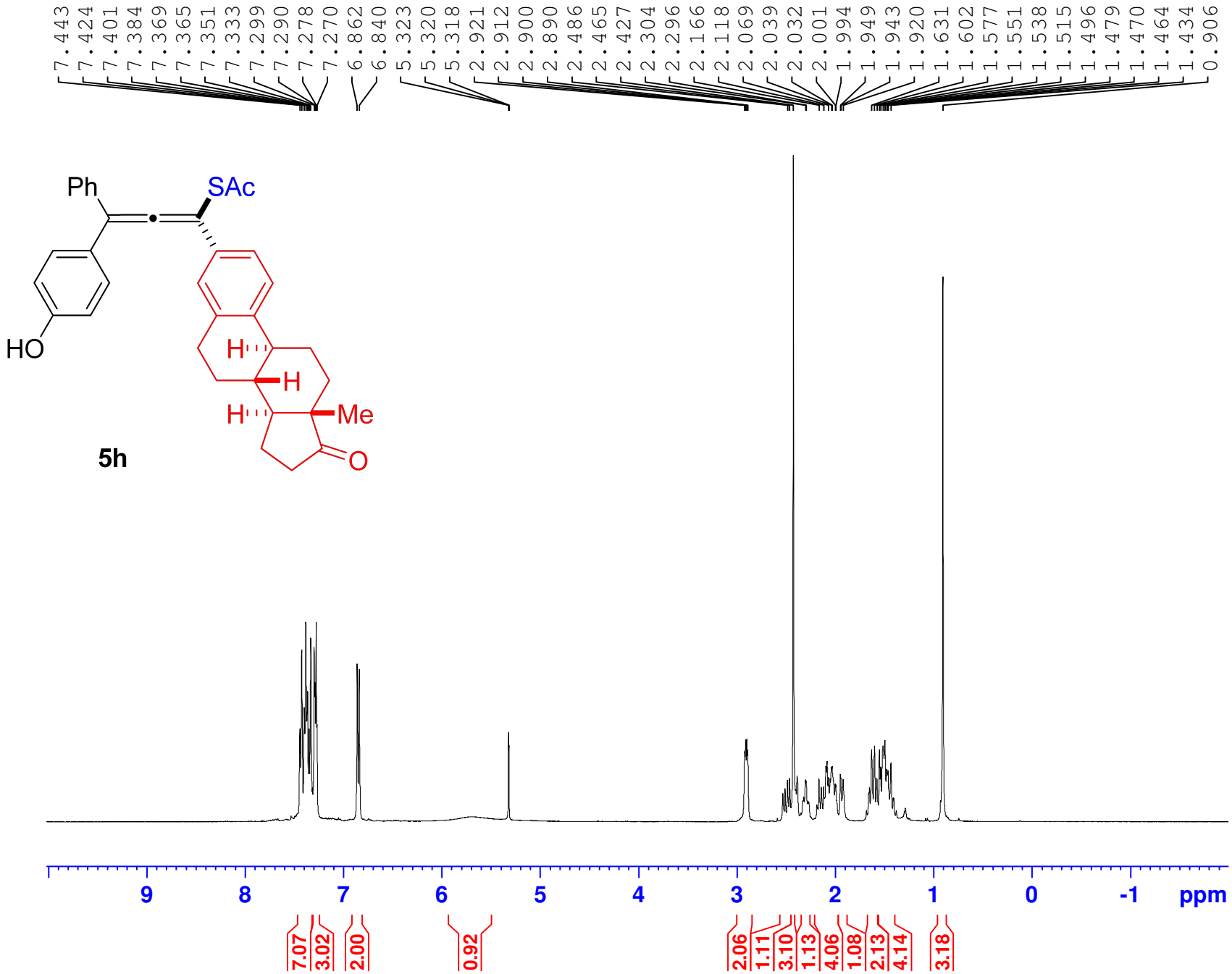


5g



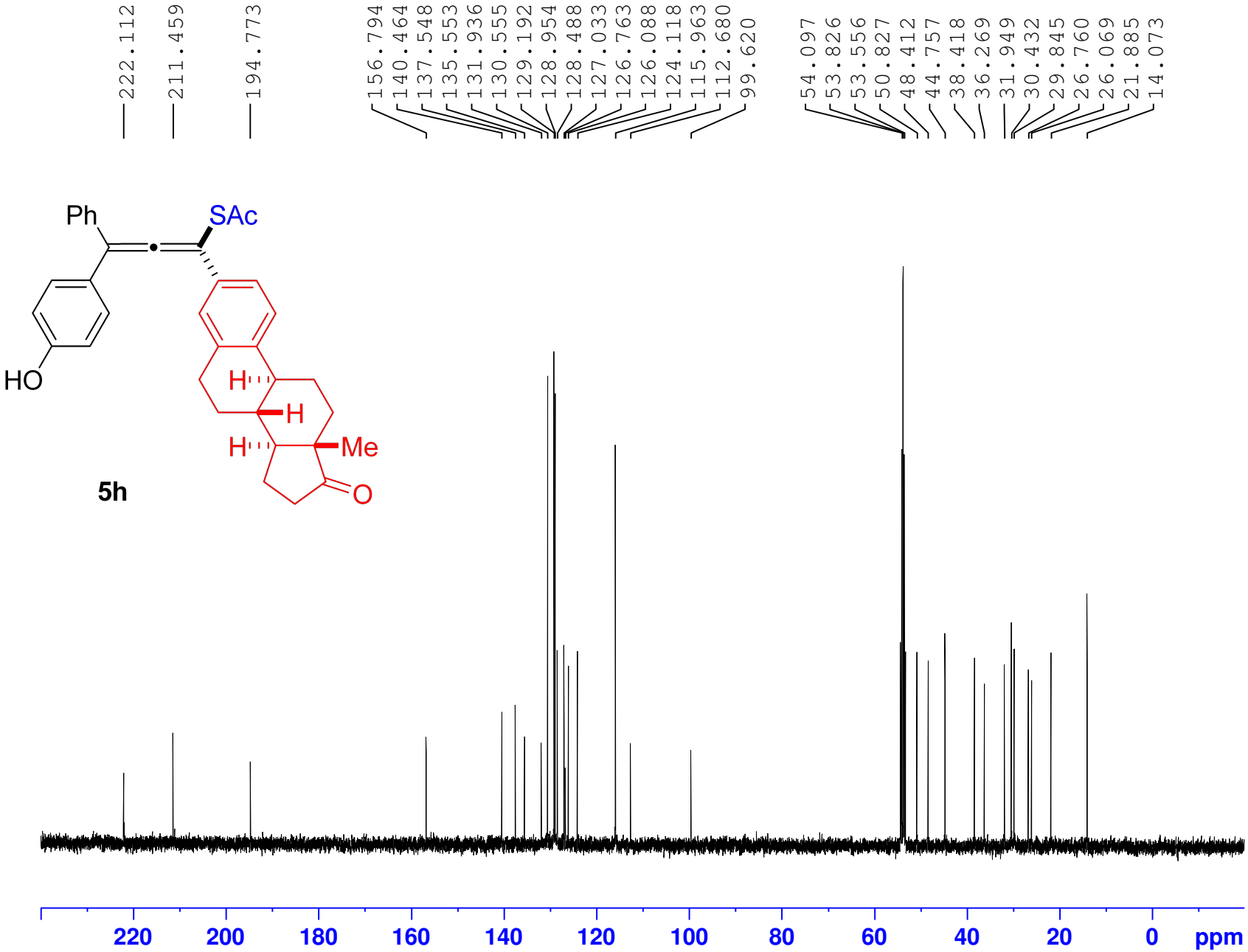

```
Current Data Parameters
NAME      qdy-20115-1 C
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20160414
Time              19.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CD2Cl2
NS                 102
DS                   2
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE               297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127321 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 120. ^{13}C NMR spectrum for 5g



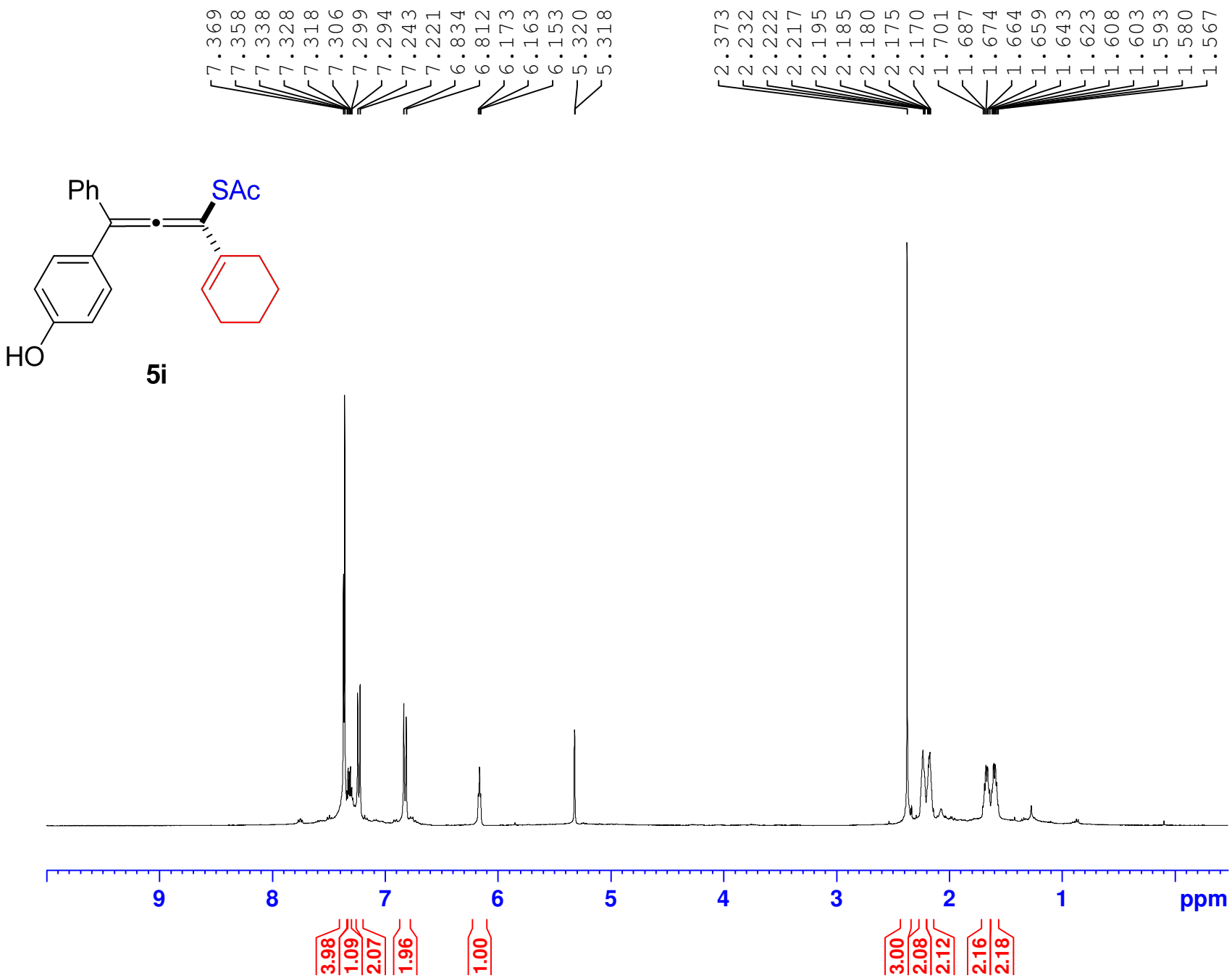

```
Current Data Parameters
NAME       qdy-20117-3 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160421
Time              19.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                54.81
DW                62.400 usec
DE                 6.50 usec
TE                297.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300151 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 121. ^{1}H NMR spectrum for 5h



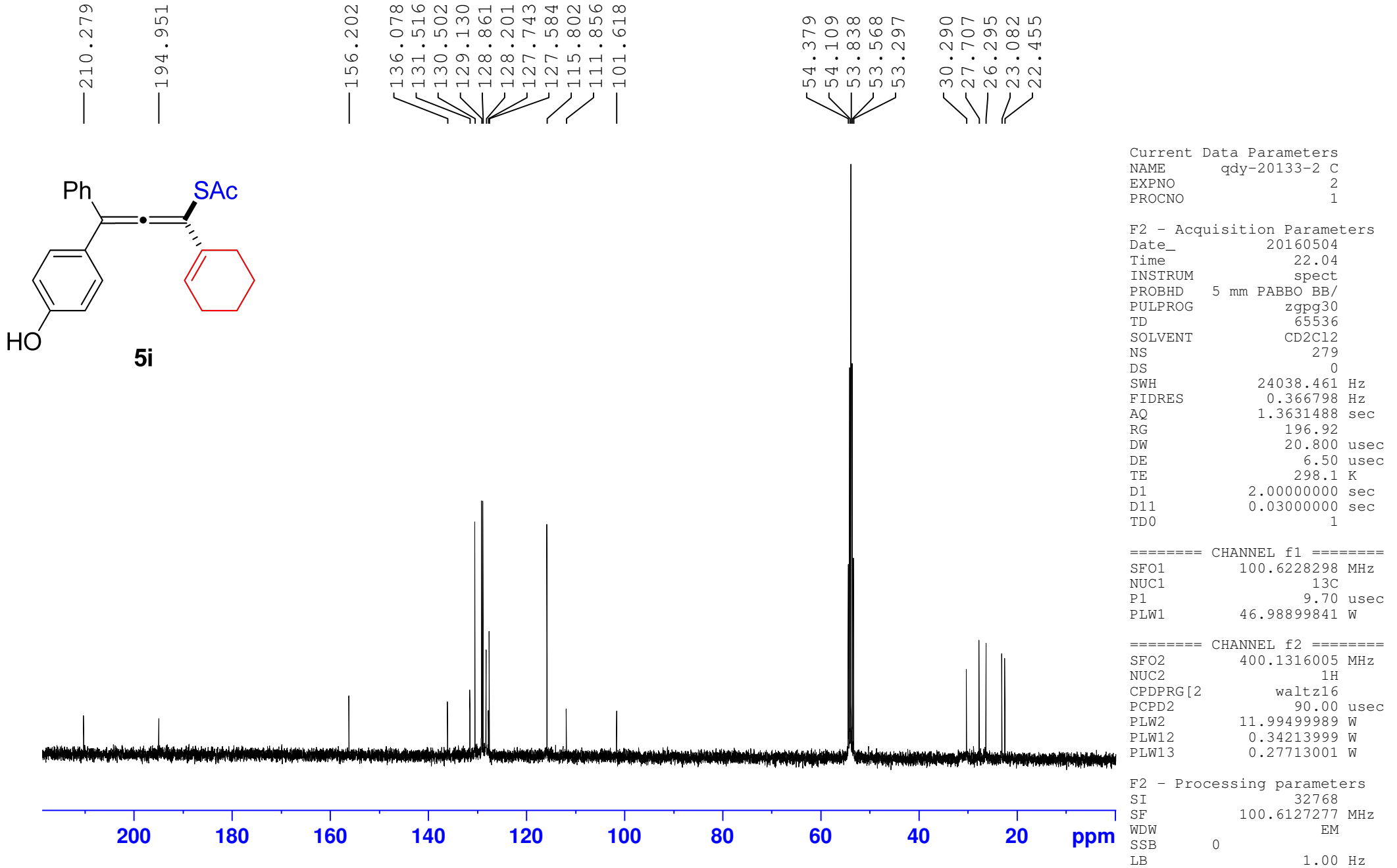

```
Current Data Parameters
NAME      qdy-20117-3 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160420
Time              19.42
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT         CD2Cl2
NS                 194
DS                   2
SWH          29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG               196.92
DW               16.800 usec
DE                6.50 usec
TE                297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228303 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127317 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 122. ^{13}C NMR spectrum for 5h

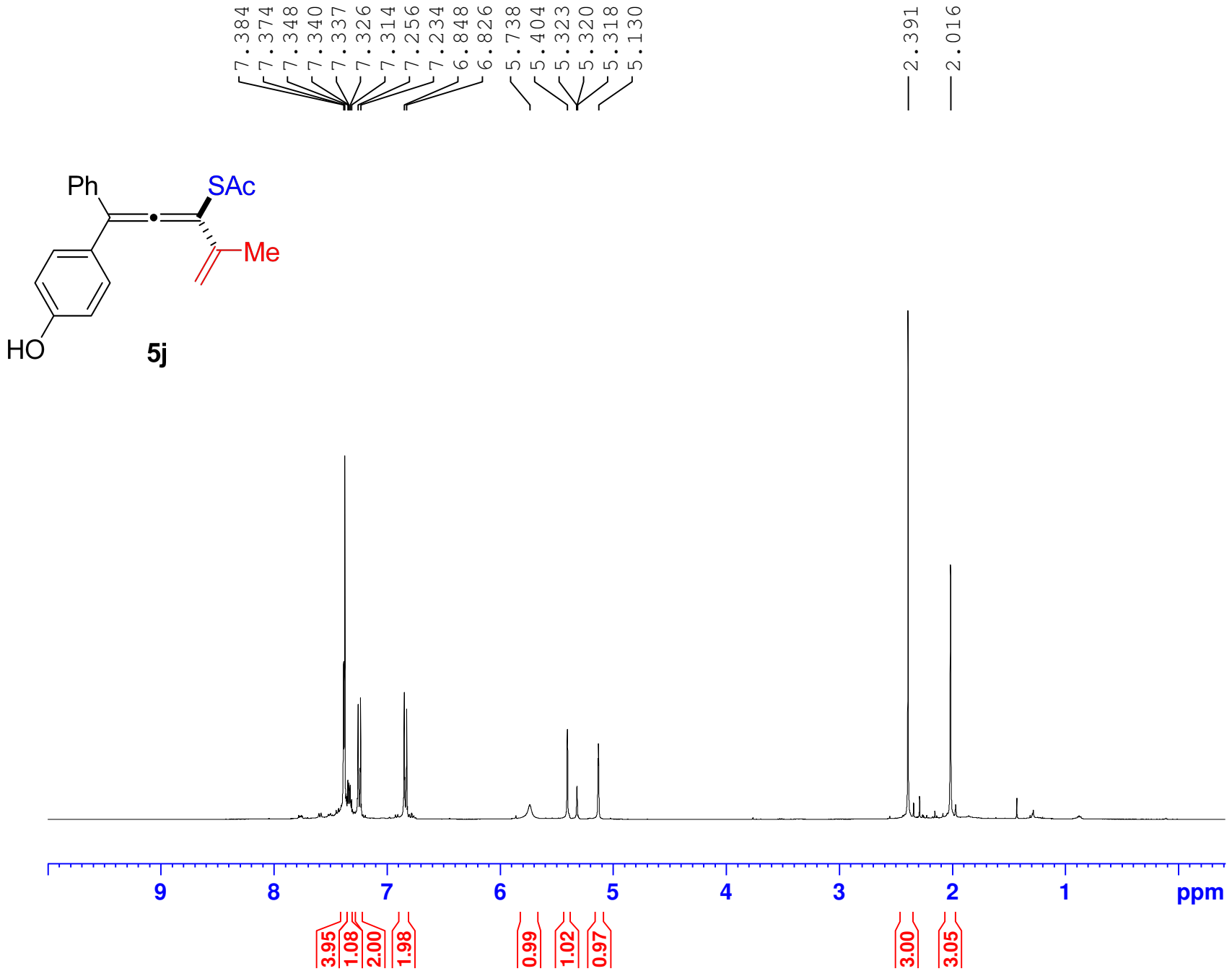



```
Current Data Parameters
NAME       qdy-20133-2 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160504
Time              21.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CD2Cl2
NS                    9
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                88.84
DW               62.400 usec
DE                6.50 usec
TE                296.9 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 123. ^{1}H NMR spectrum for 5i

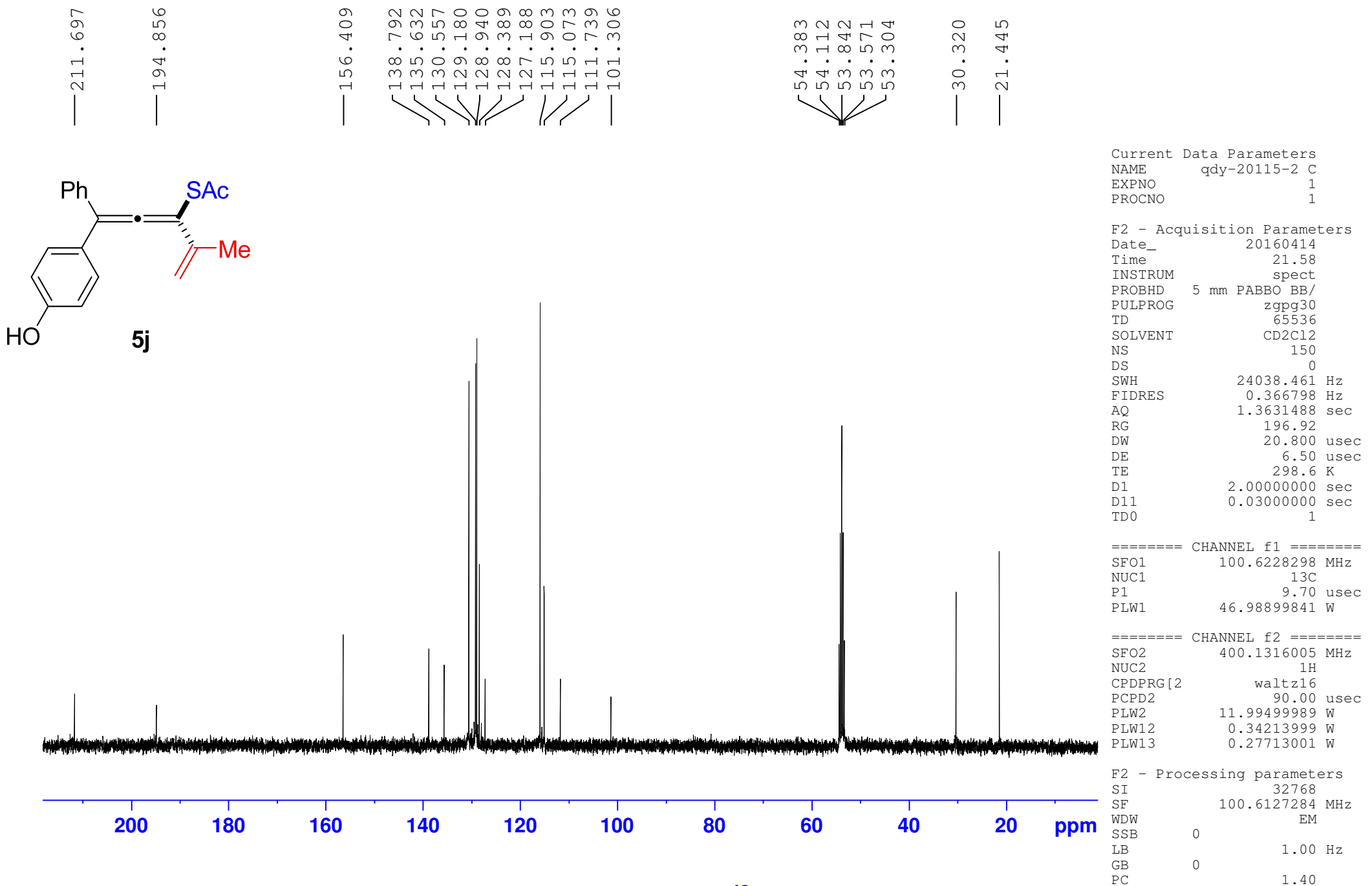


Supplementary Figure 124. ^{13}C NMR spectrum for 5i



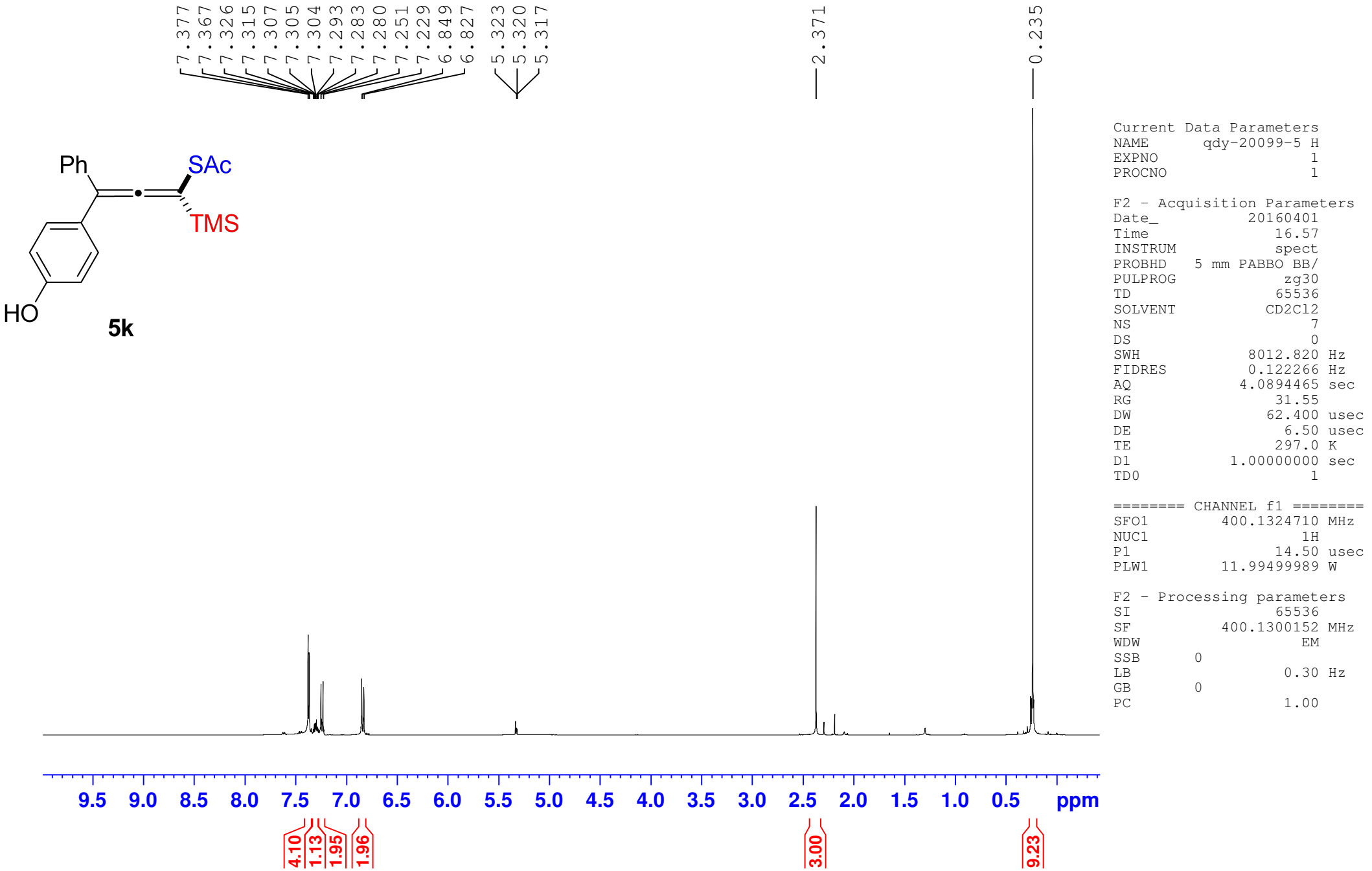

```
Current Data Parameters
NAME       qdy-20115-2 H
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20160416
Time              19.31
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                   11
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                62.93
DW               62.400 usec
DE                6.50 usec
TE                298.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300151 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 125. ^{1}H NMR spectrum for 5j



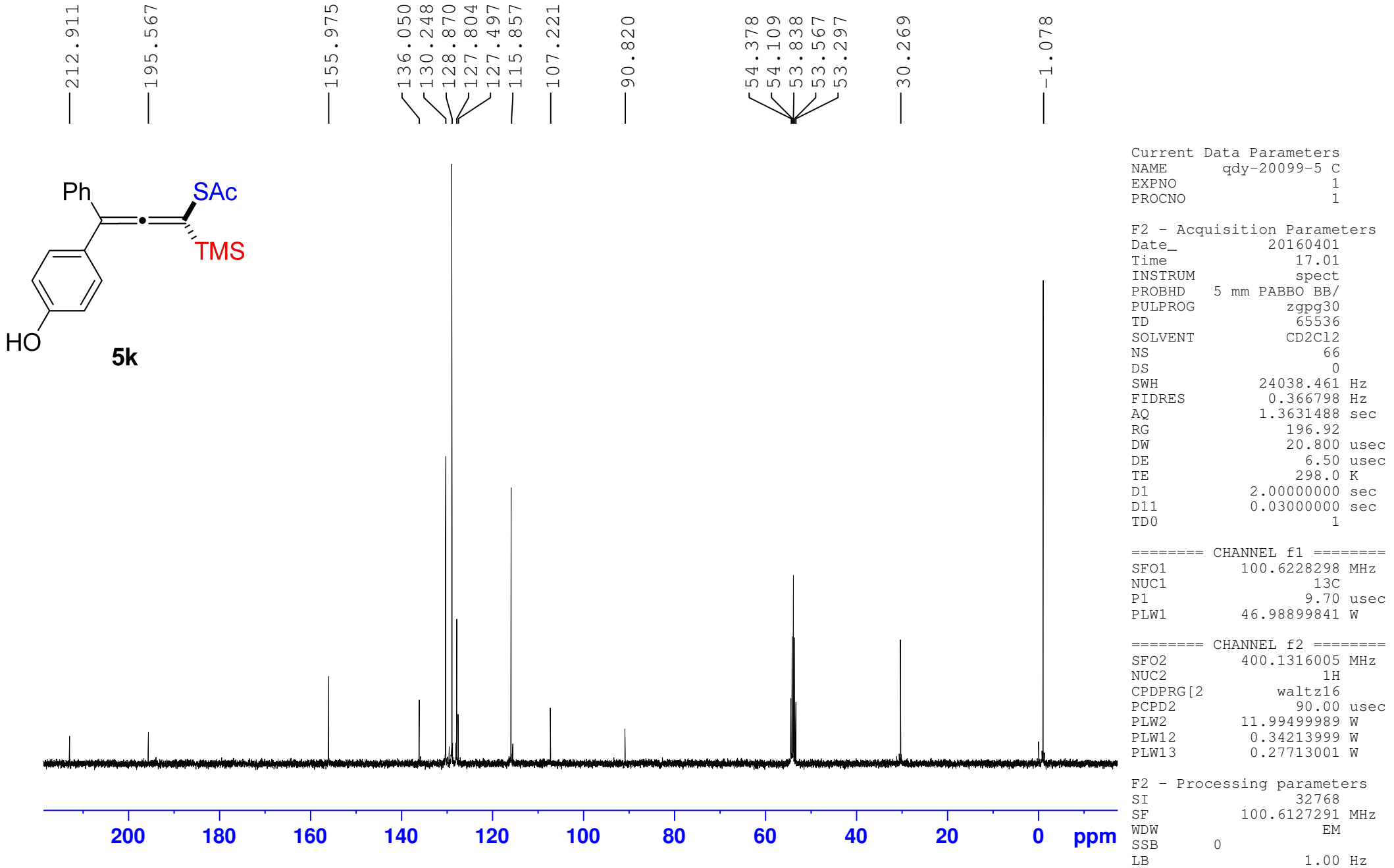

```
Current Data Parameters
NAME       qdy-20115-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160414
Time              21.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                  150
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127284 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 126. ^{13}C NMR spectrum for 5j



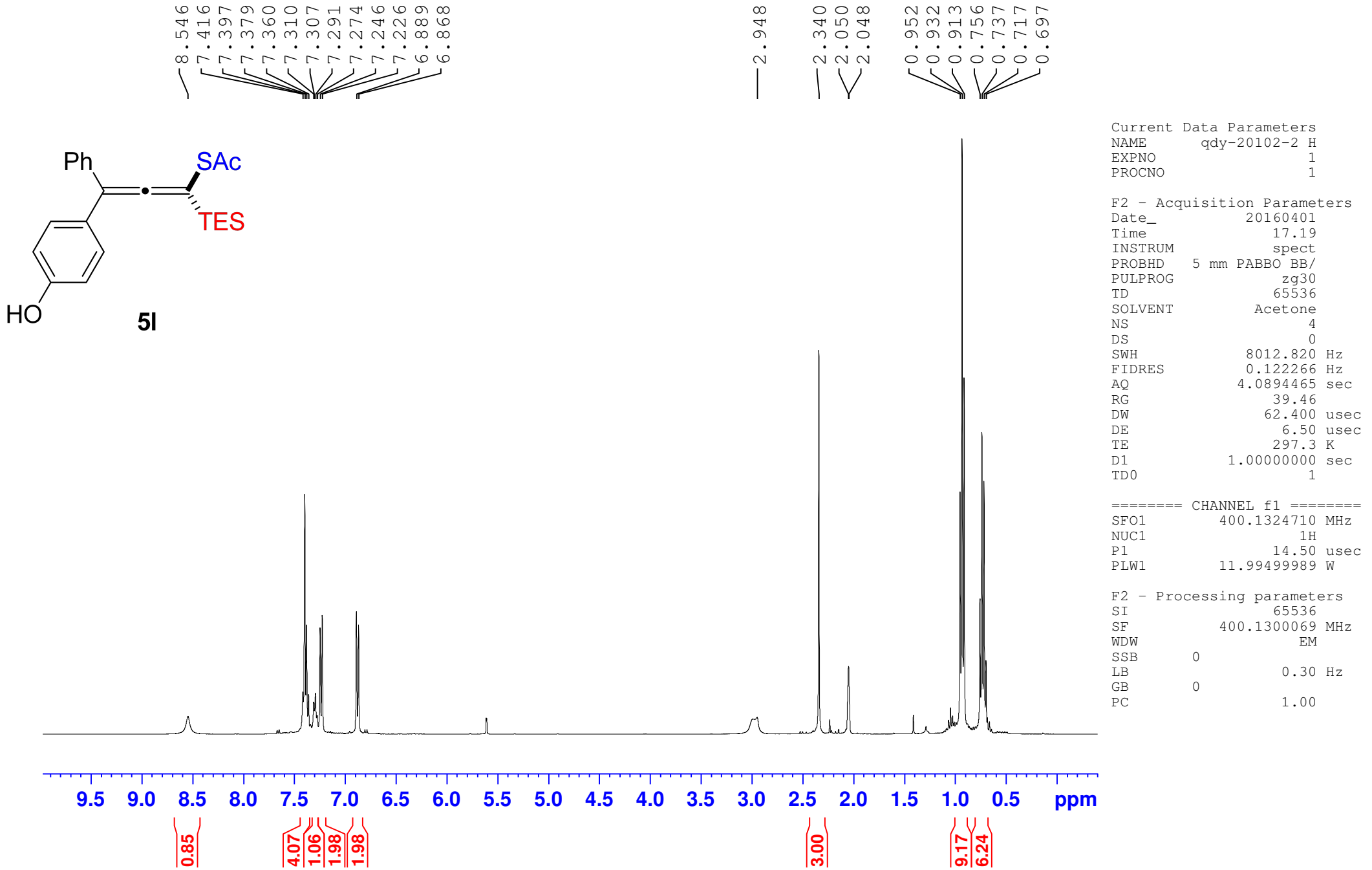

```
Current Data Parameters
NAME       qdy-20099-5 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160401
Time              16.57
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CD2Cl2
NS                    7
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                31.55
DW               62.400 usec
DE                6.50 usec
TE                297.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 127. ^{1}H NMR spectrum for 5k

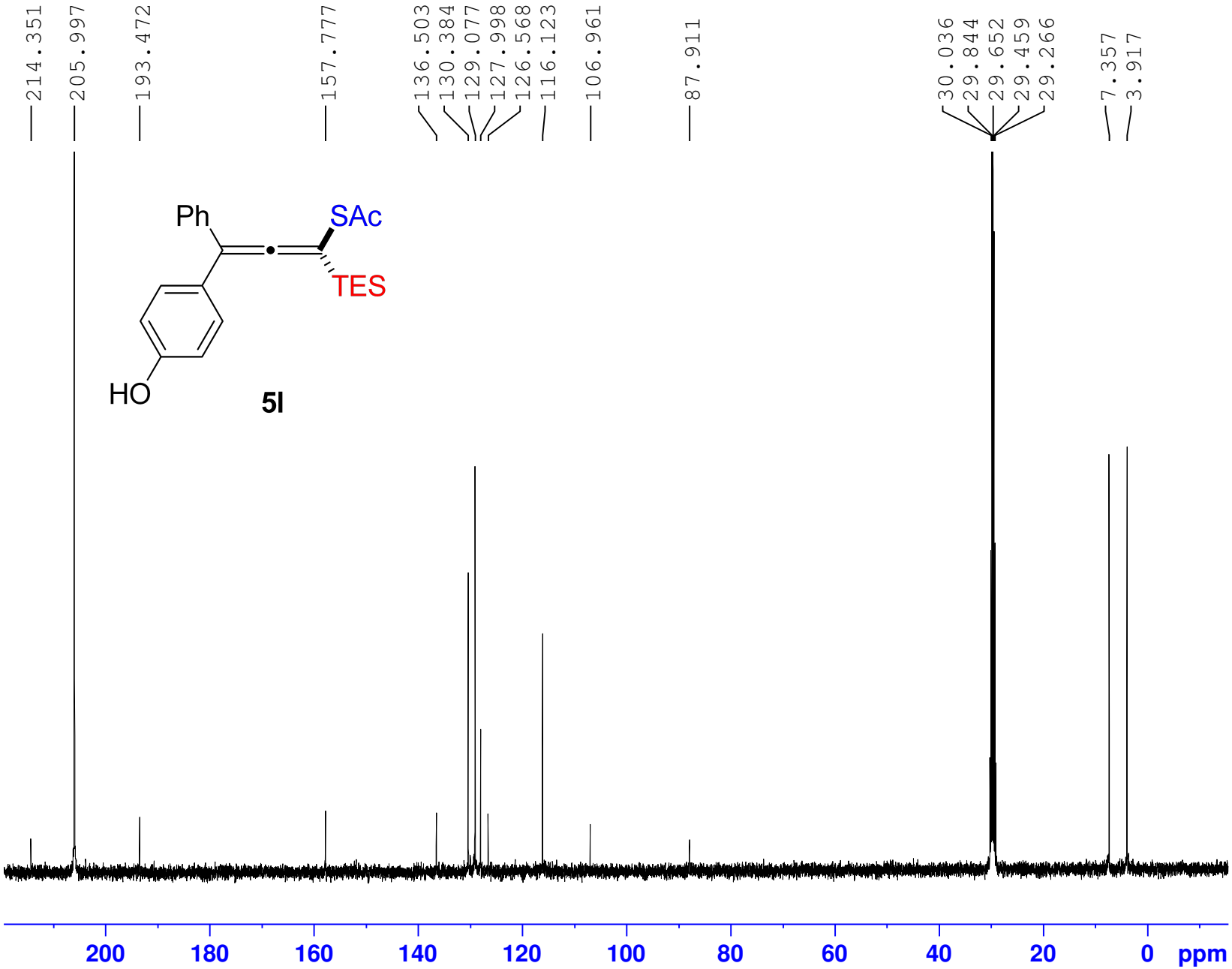



```
Current Data Parameters
NAME       qdy-20099-5 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160401
Time              17.01
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                   66
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127291 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 128. ^{13}C NMR spectrum for 5k

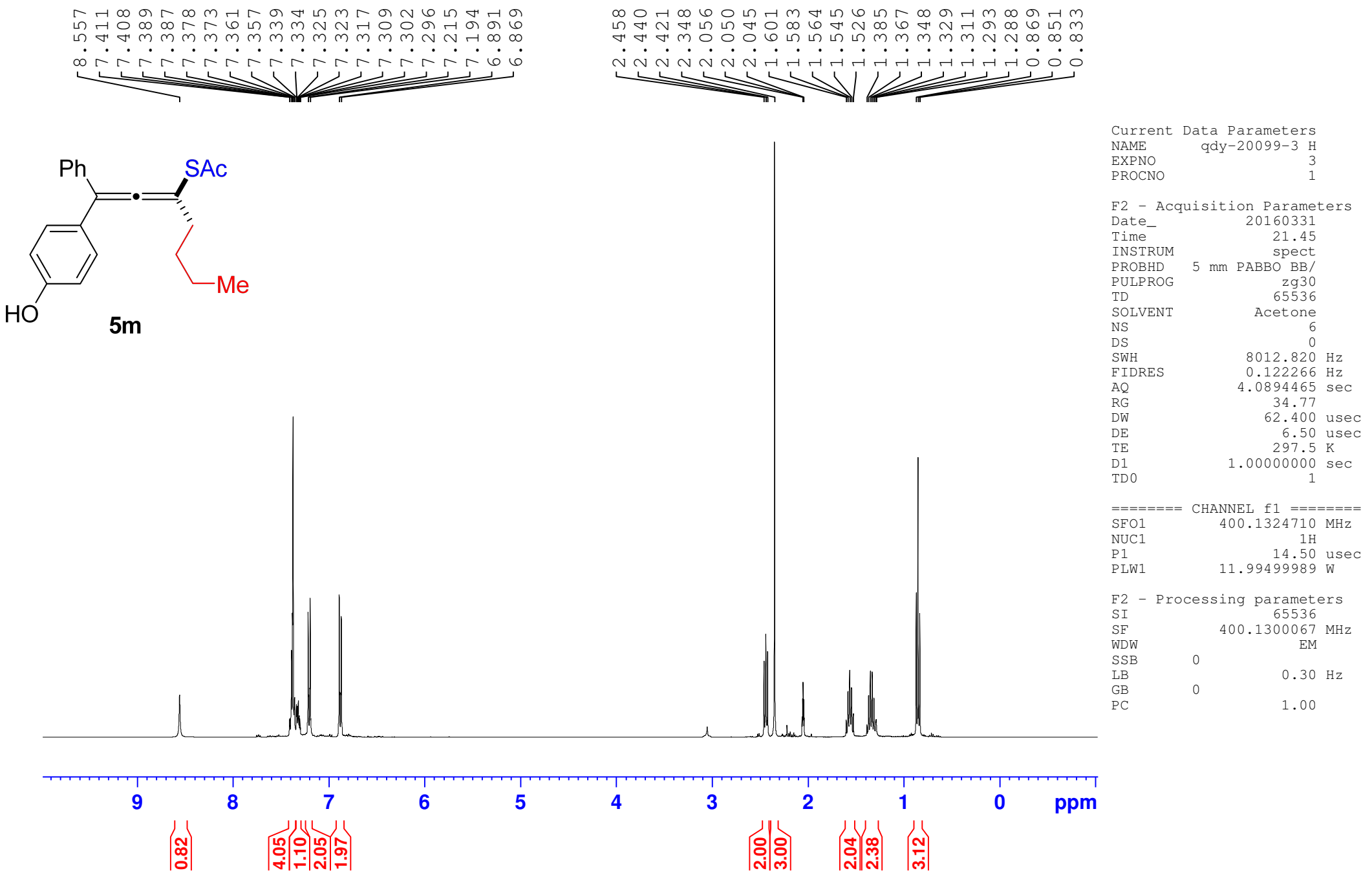


Supplementary Figure 129. ^{1}H NMR spectrum for 5l



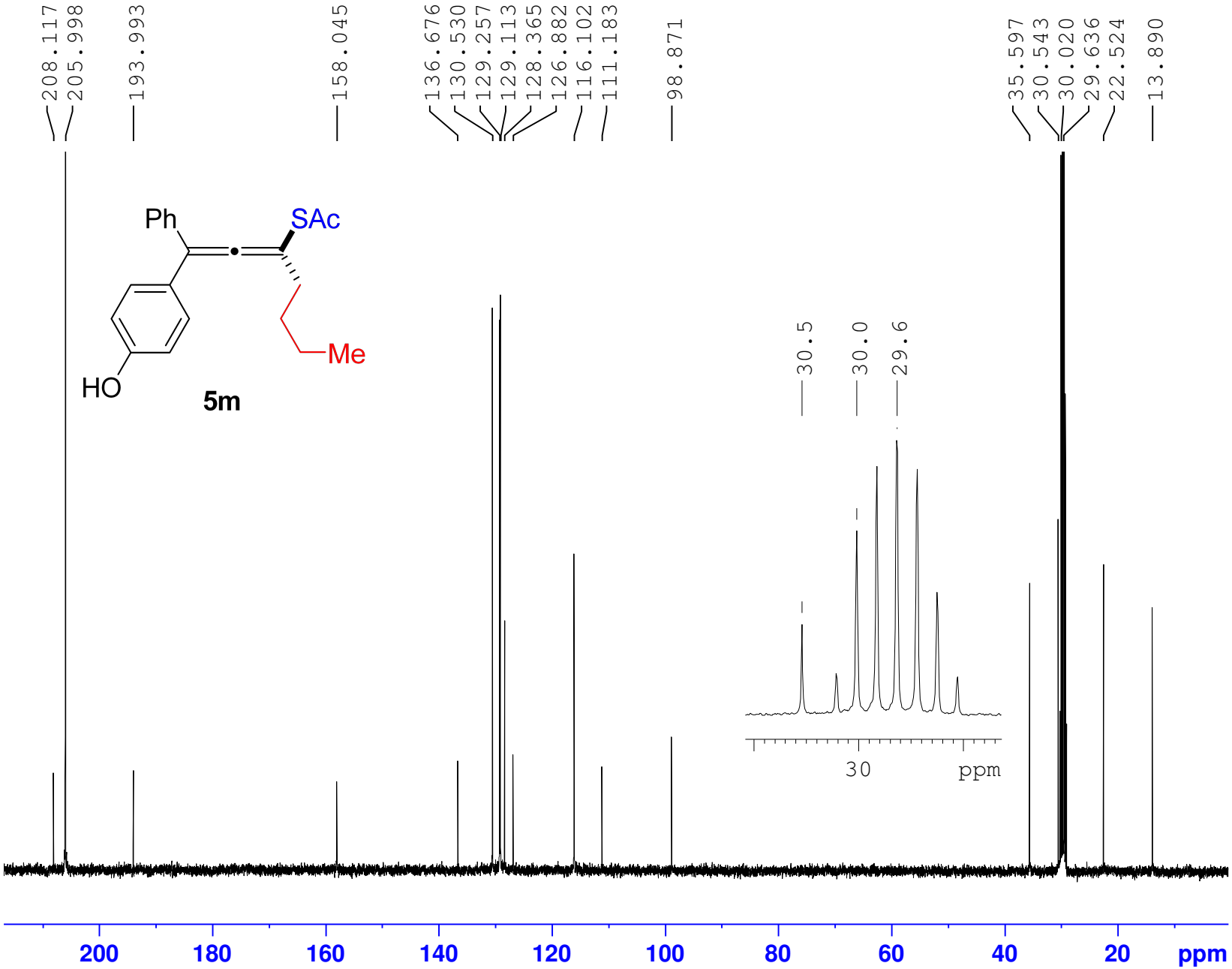

```
Current Data Parameters
NAME      qdy-20102-2 C
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20160402
Time             14.01
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                 150
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127007 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 130. ^{13}C NMR spectrum for 5l



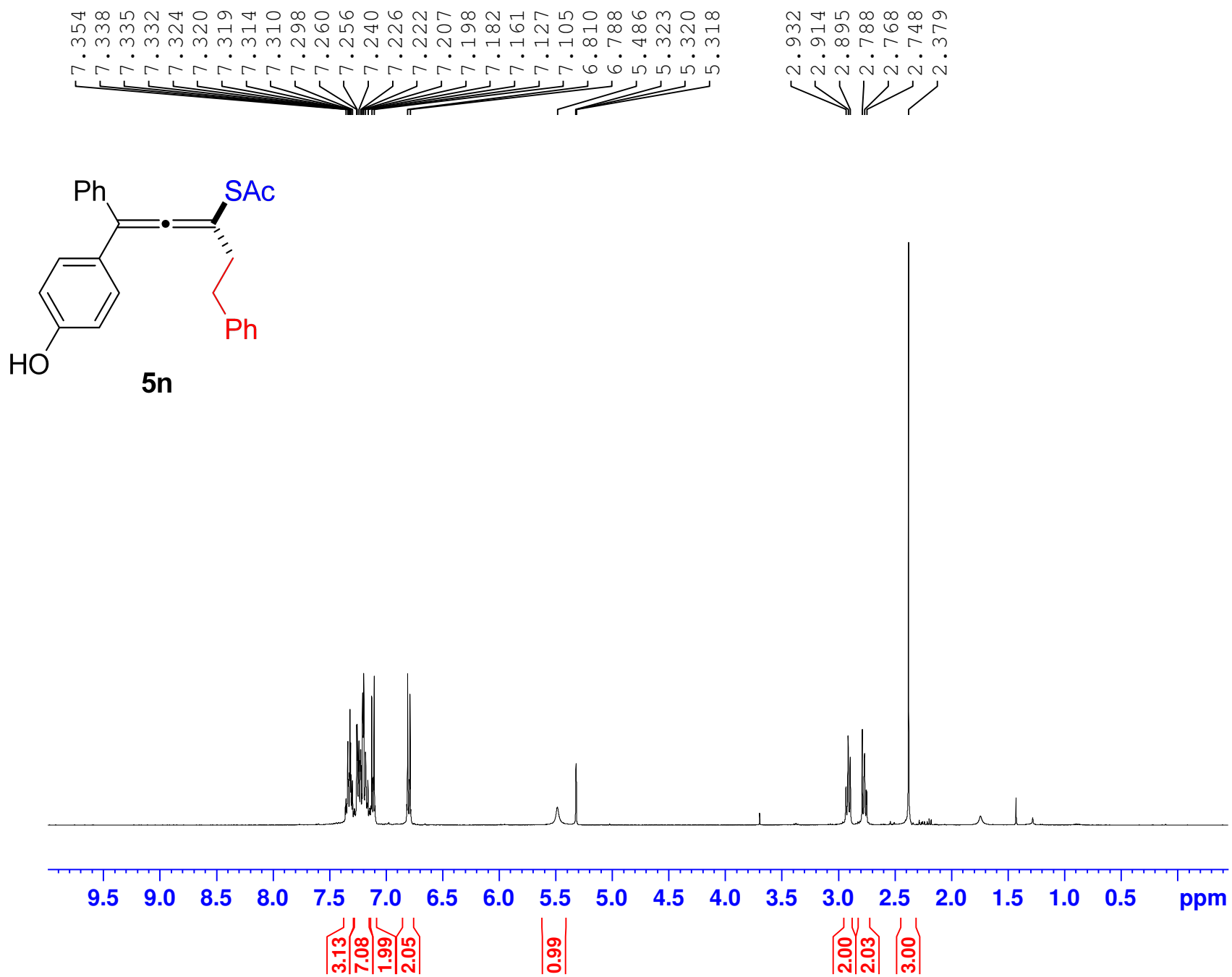

```
Current Data Parameters
NAME       qdy-20099-3 H
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20160331
Time              21.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                34.77
DW               62.400 usec
DE                6.50 usec
TE                297.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300067 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 131. ^{1}H NMR spectrum for 5m

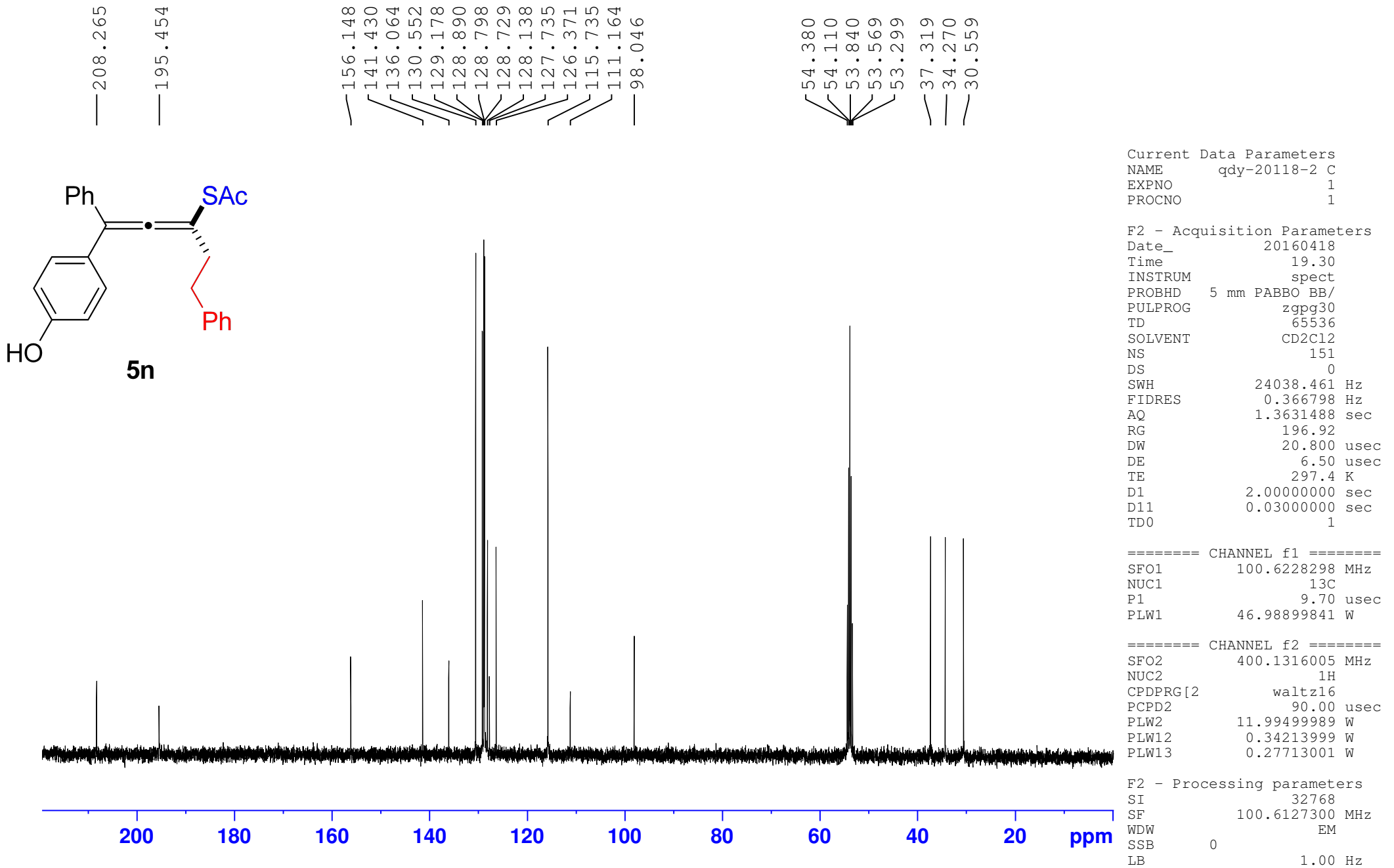



```
Current Data Parameters
NAME       qdy-20099-3 C
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160331
Time              21.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                   99
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127044 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 132. ^{13}C NMR spectrum for 5m

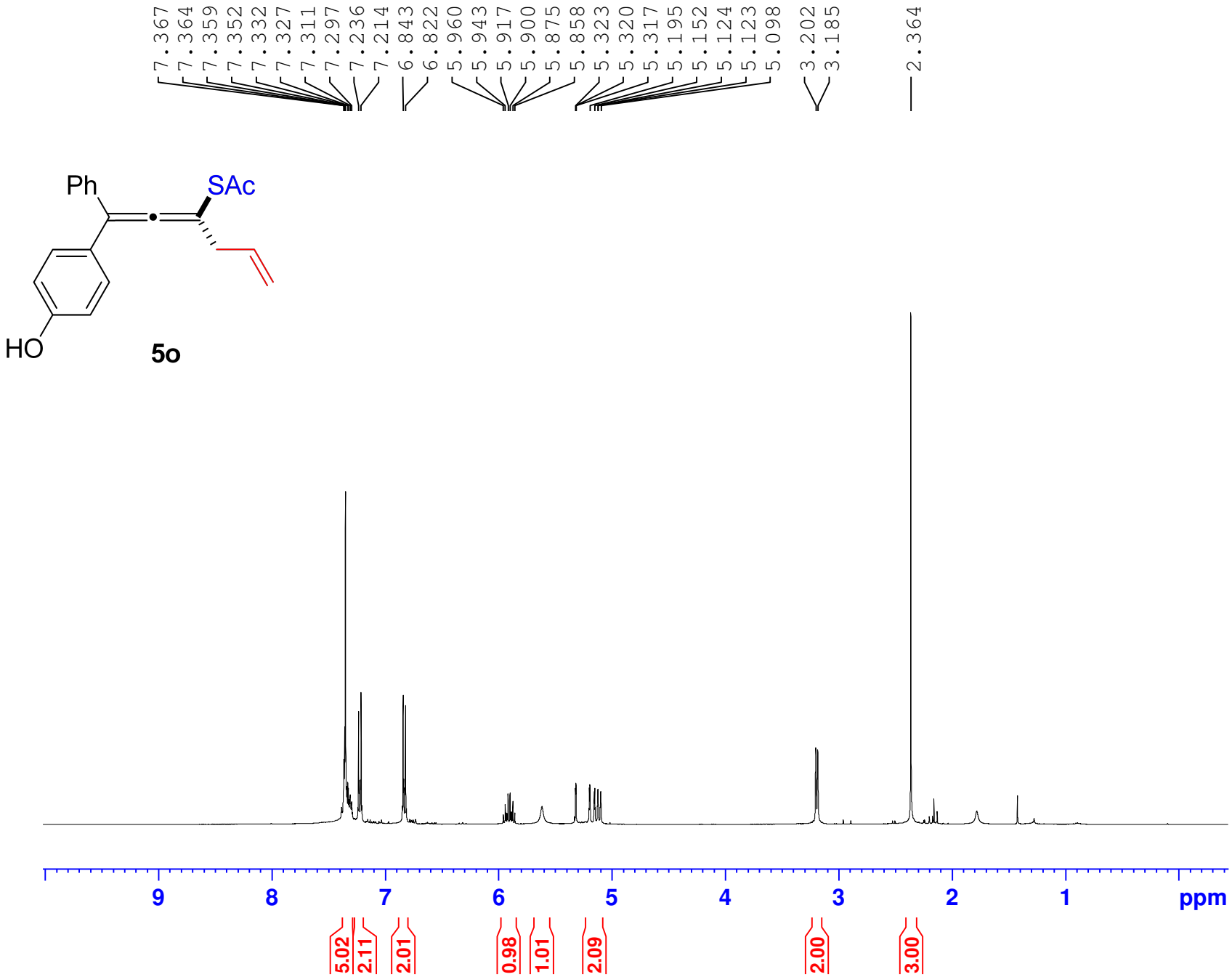


Supplementary Figure 133. ^{1}H NMR spectrum for 5n



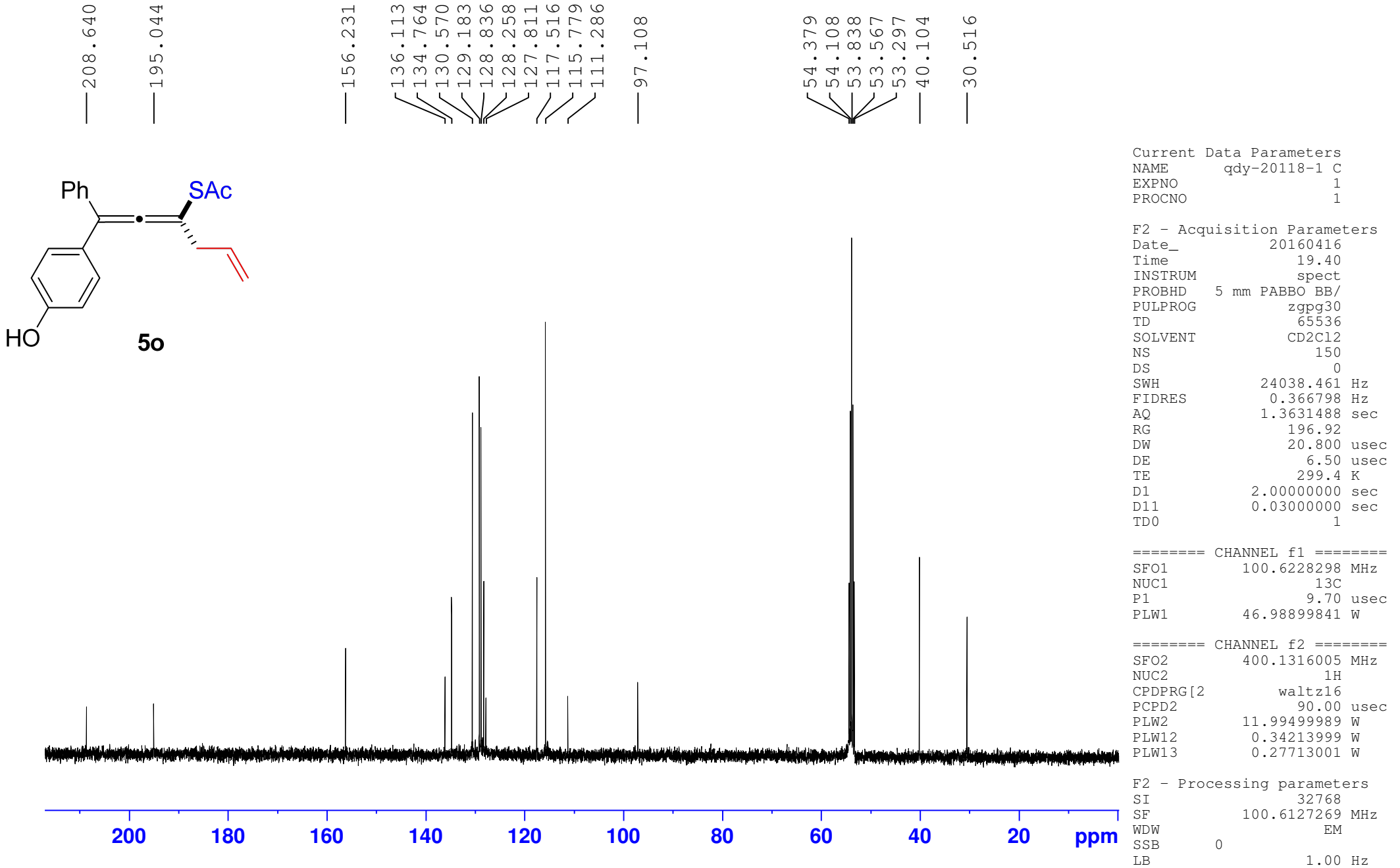

```
Current Data Parameters
NAME      qdy-20118-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160418
Time              19.30
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT         CD2Cl2
NS                 151
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                297.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127300 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 134. ^{13}C NMR spectrum for 5n

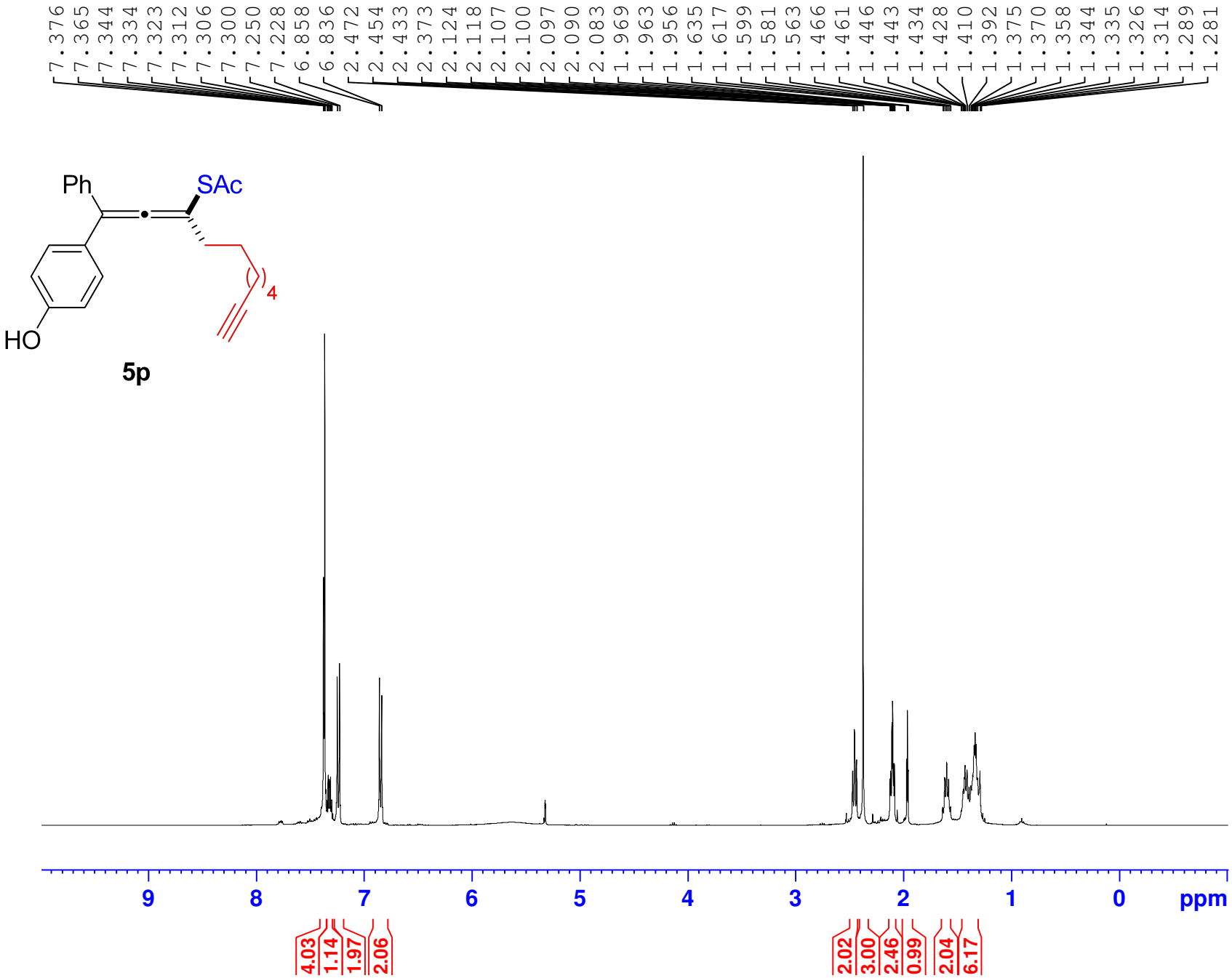



```
Current Data Parameters
NAME       qdy-20118-1 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160416
Time              19.35
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    7
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                70.97
DW               62.400 usec
DE                6.50 usec
TE                298.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300153 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 135. ^{1}H NMR spectrum for 5o

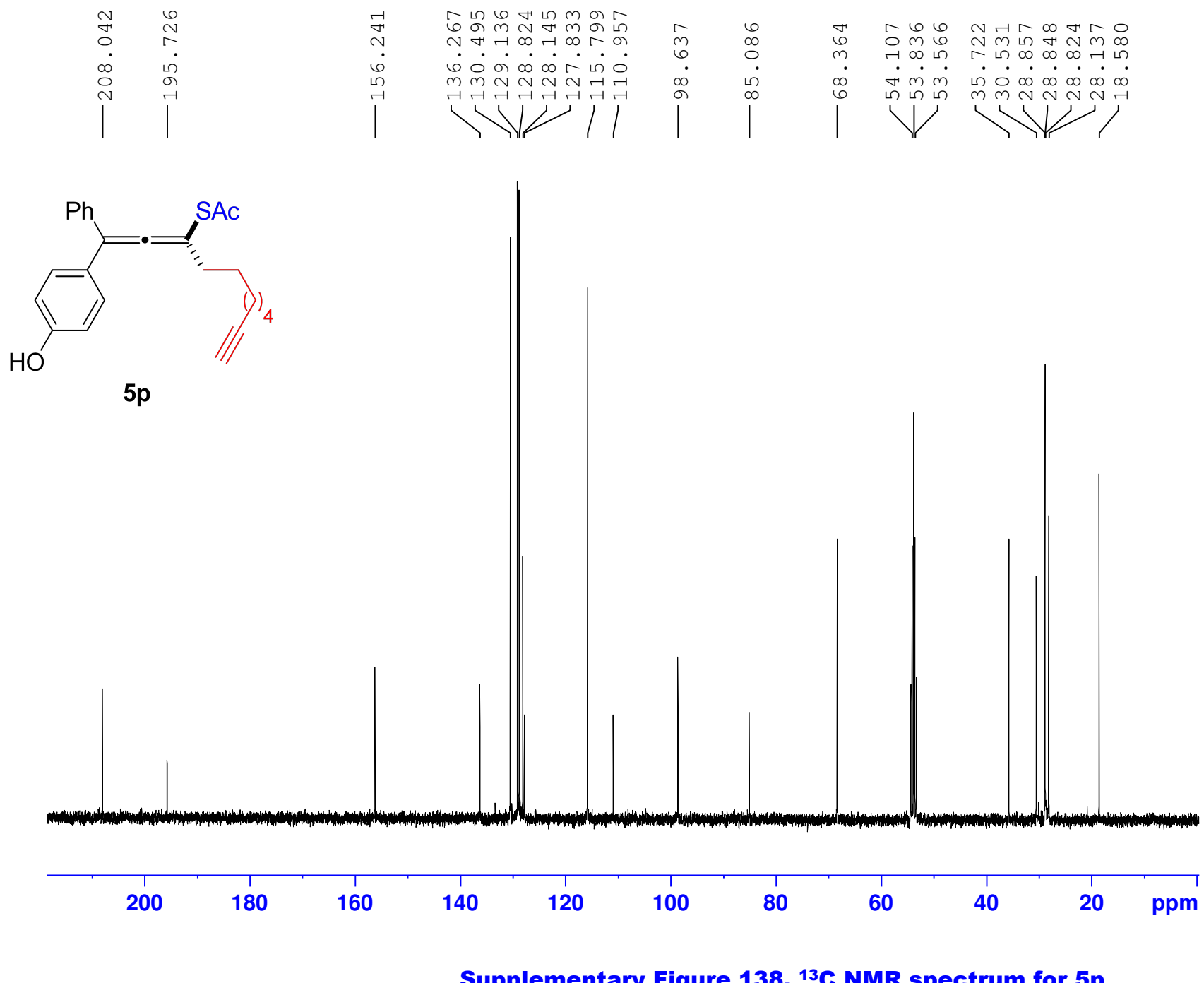


Supplementary Figure 136. ^{13}C NMR spectrum for 5o



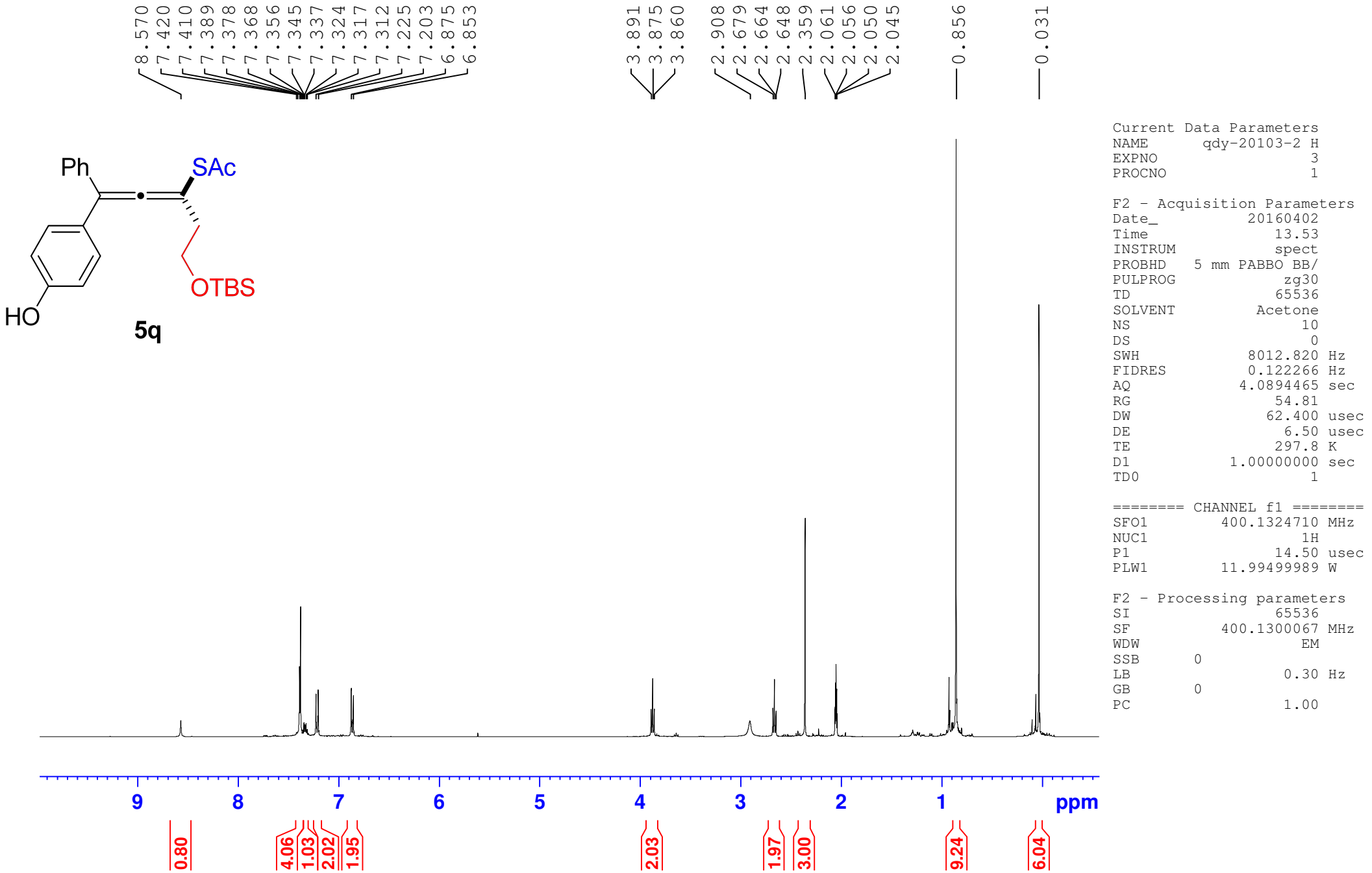

```
Current Data Parameters
NAME       qdy-20099-4 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160331
Time              13.27
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    6
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                31.55
DW               62.400 usec
DE                6.50 usec
TE                297.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 137. ^{1}H NMR spectrum for 5p

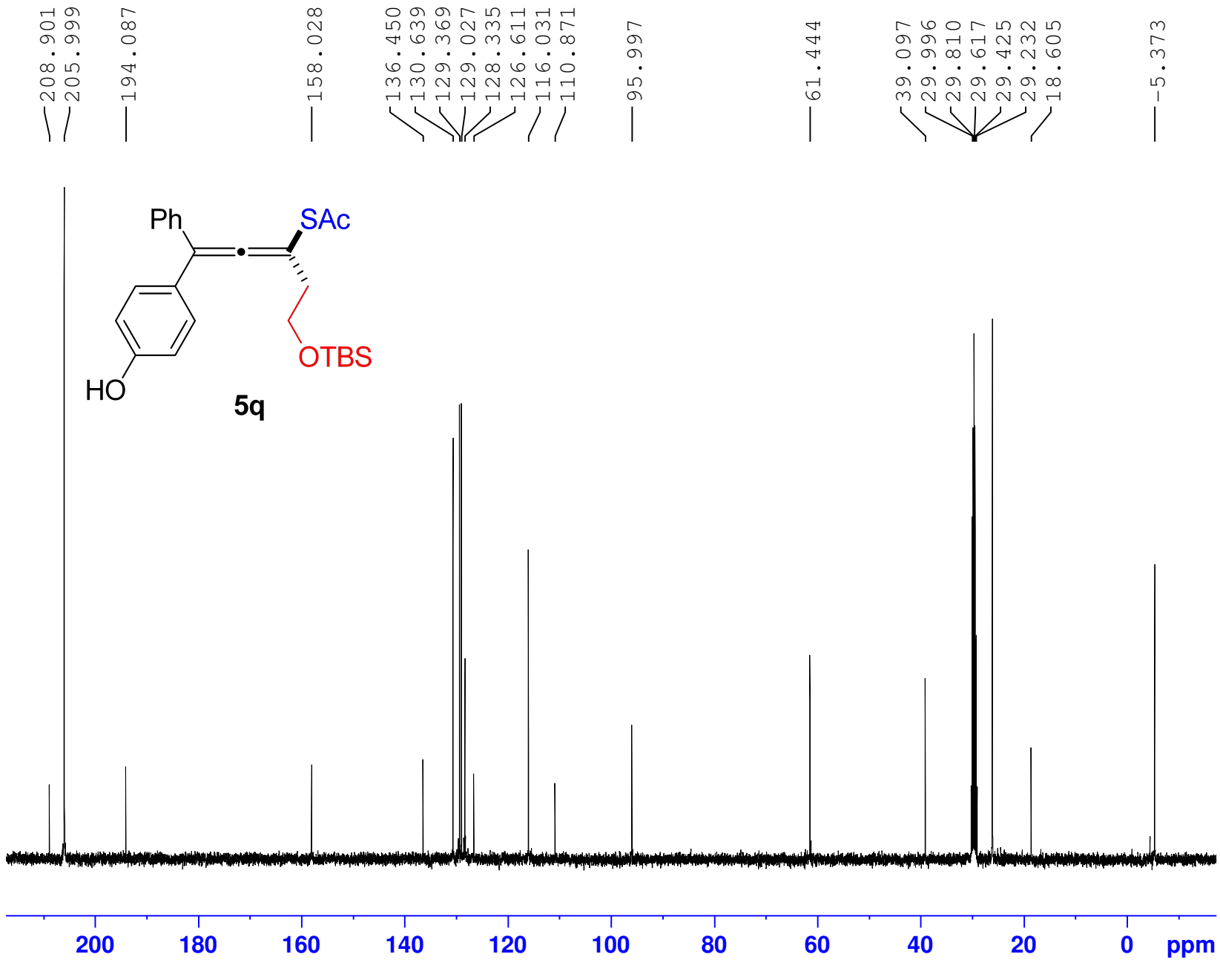



```
Current Data Parameters
NAME       qdy-20099-4 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160331
Time              13.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT         CD2Cl2
NS                 83
DS                  0
SWH         24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631488 sec
RG              196.92
DW              20.800 usec
DE               6.50 usec
TE              298.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127299 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 138. ^{13}C NMR spectrum for 5p

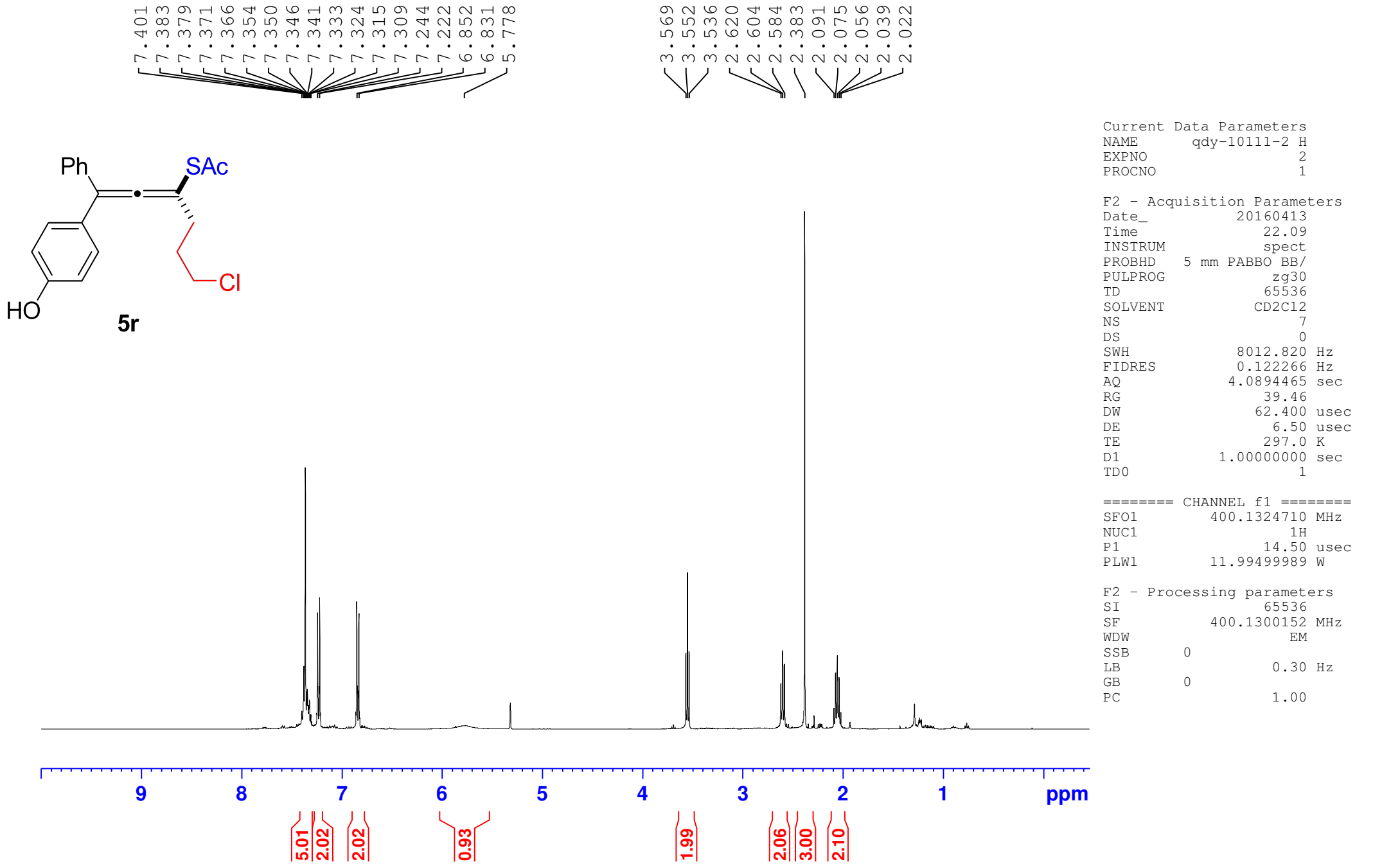


Supplementary Figure 139. ^{1}H NMR spectrum for 5q

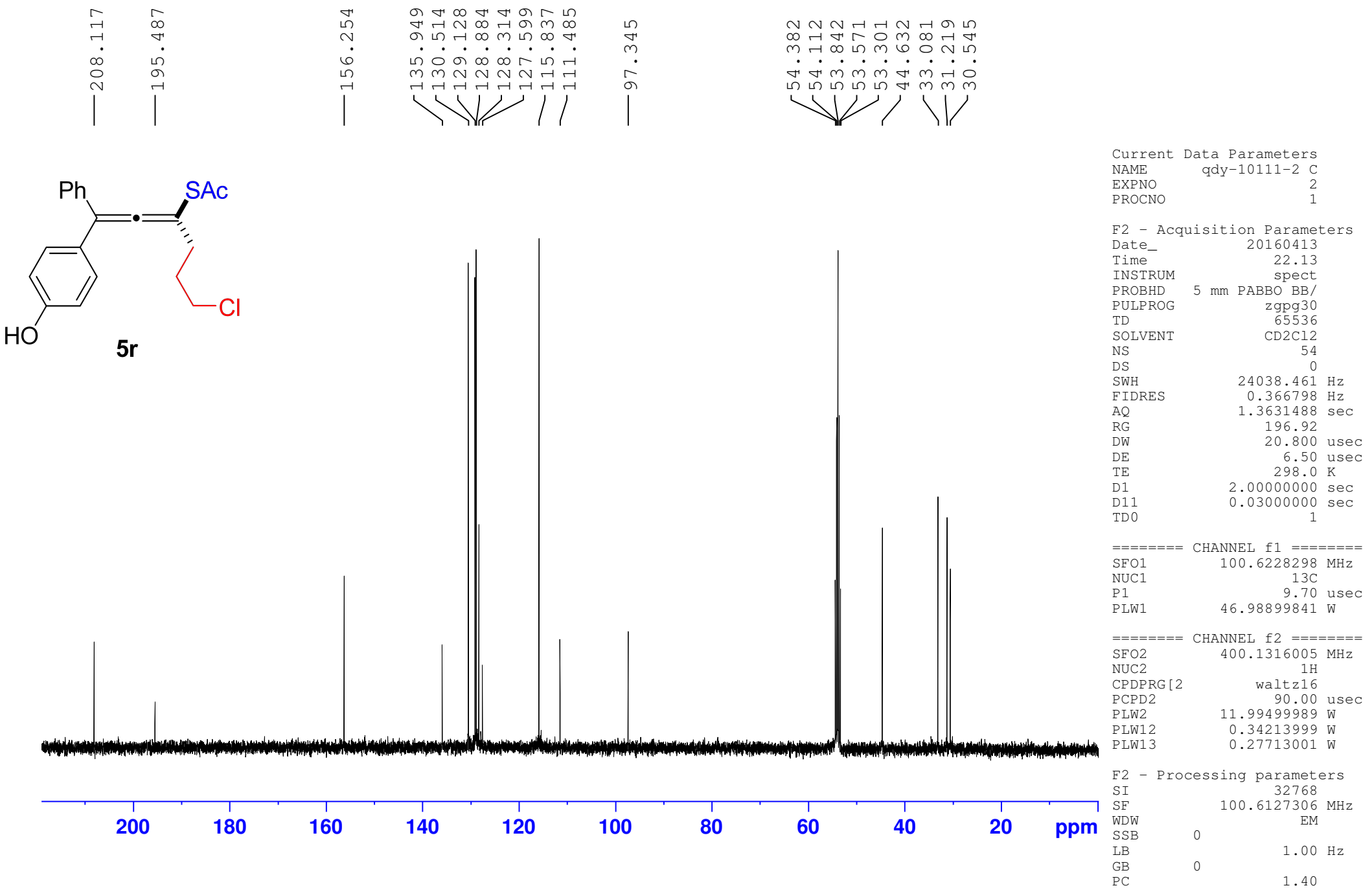



```
Current Data Parameters
NAME       qdy-20103-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160401
Time              21.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                   45
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127109 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

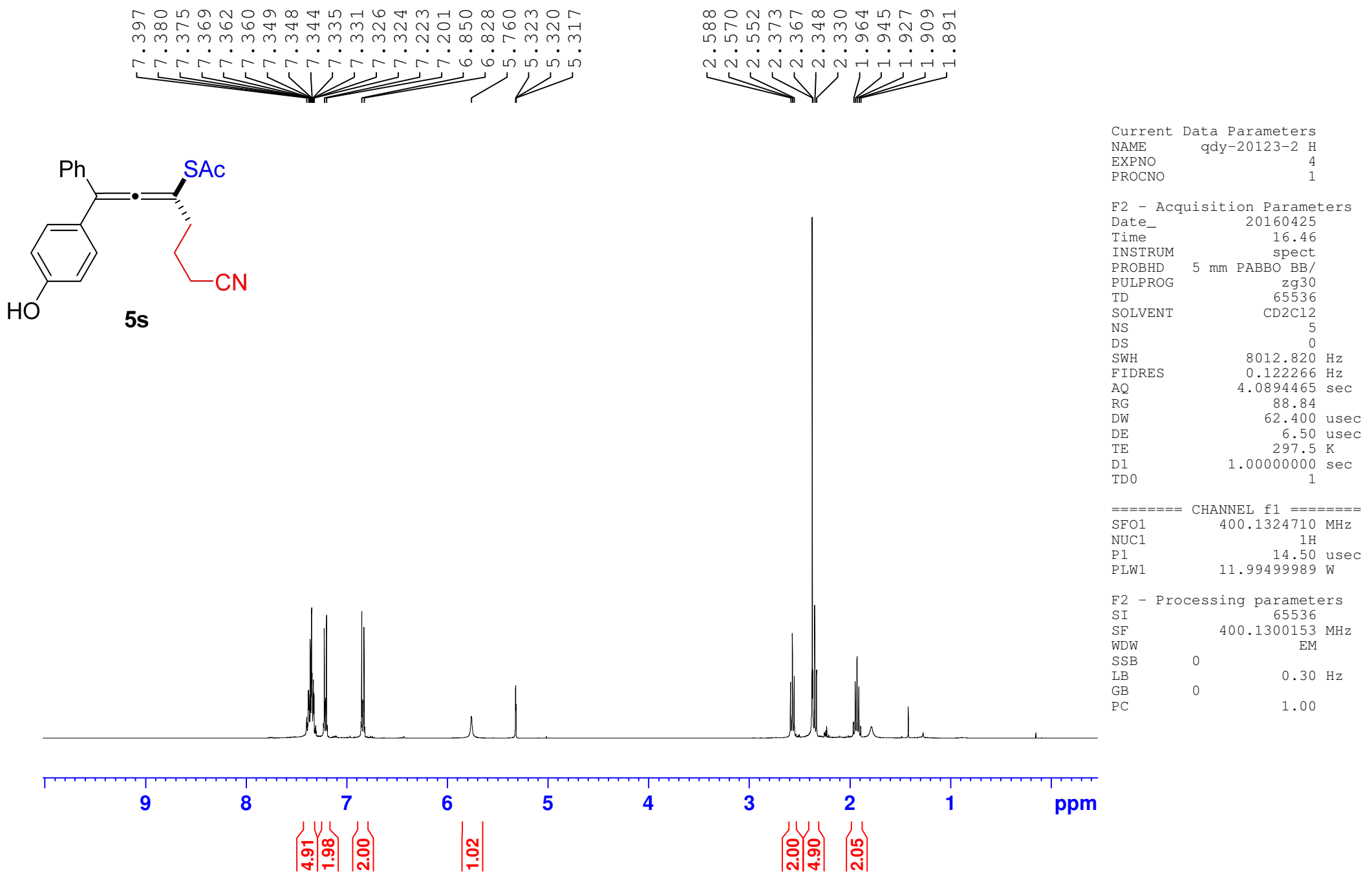
Supplementary Figure 140. ^{13}C NMR spectrum for 5q



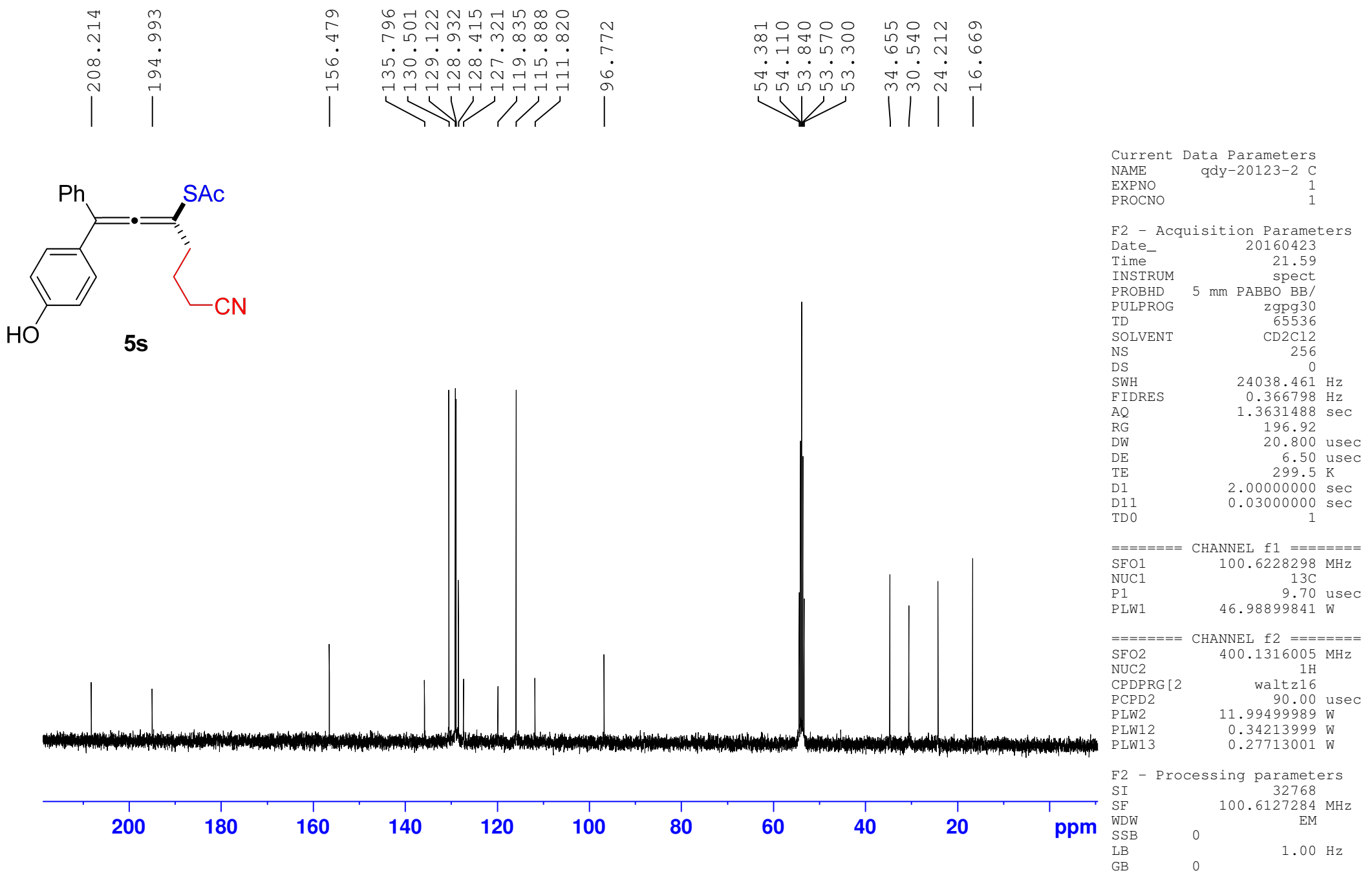
Supplementary Figure 141. ^{1}H NMR spectrum for 5r



Supplementary Figure 142. ^{13}C NMR spectrum for 5r

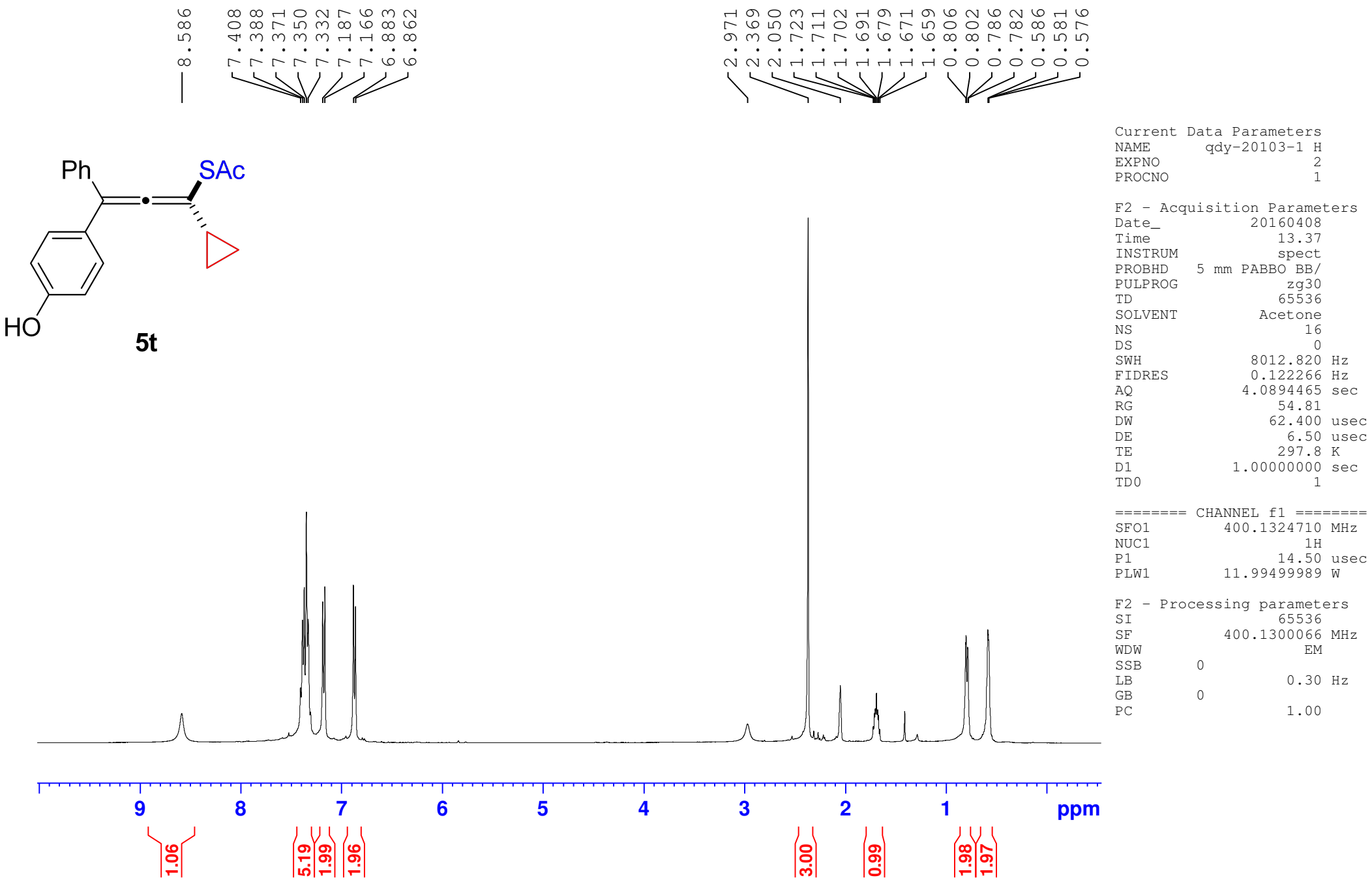


Supplementary Figure 143. ^{1}H NMR spectrum for 5s

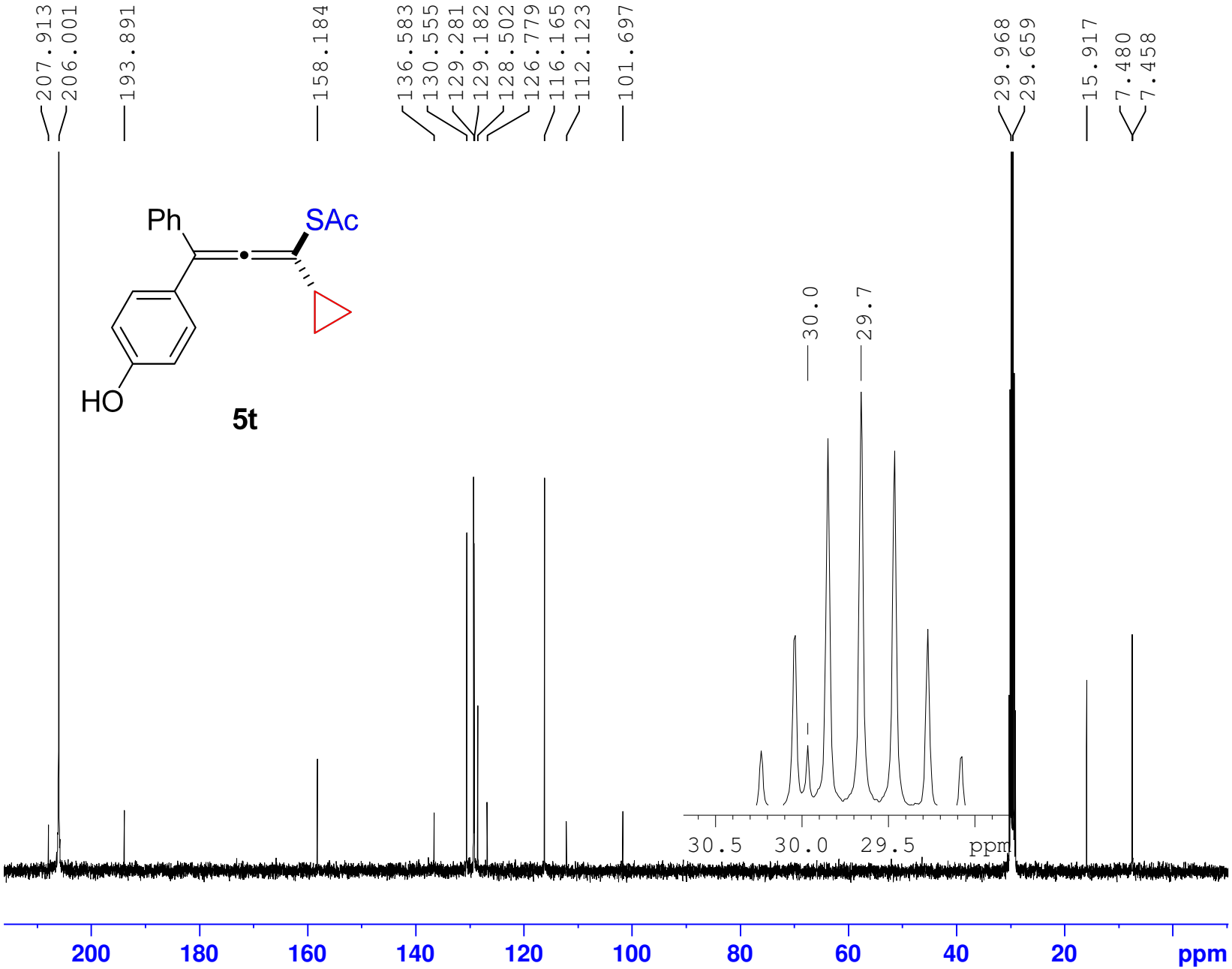



```
Current Data Parameters
NAME       qdy-20123-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160423
Time              21.59
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         CD2Cl2
NS                  256
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127284 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 144. ^{13}C NMR spectrum for 5s

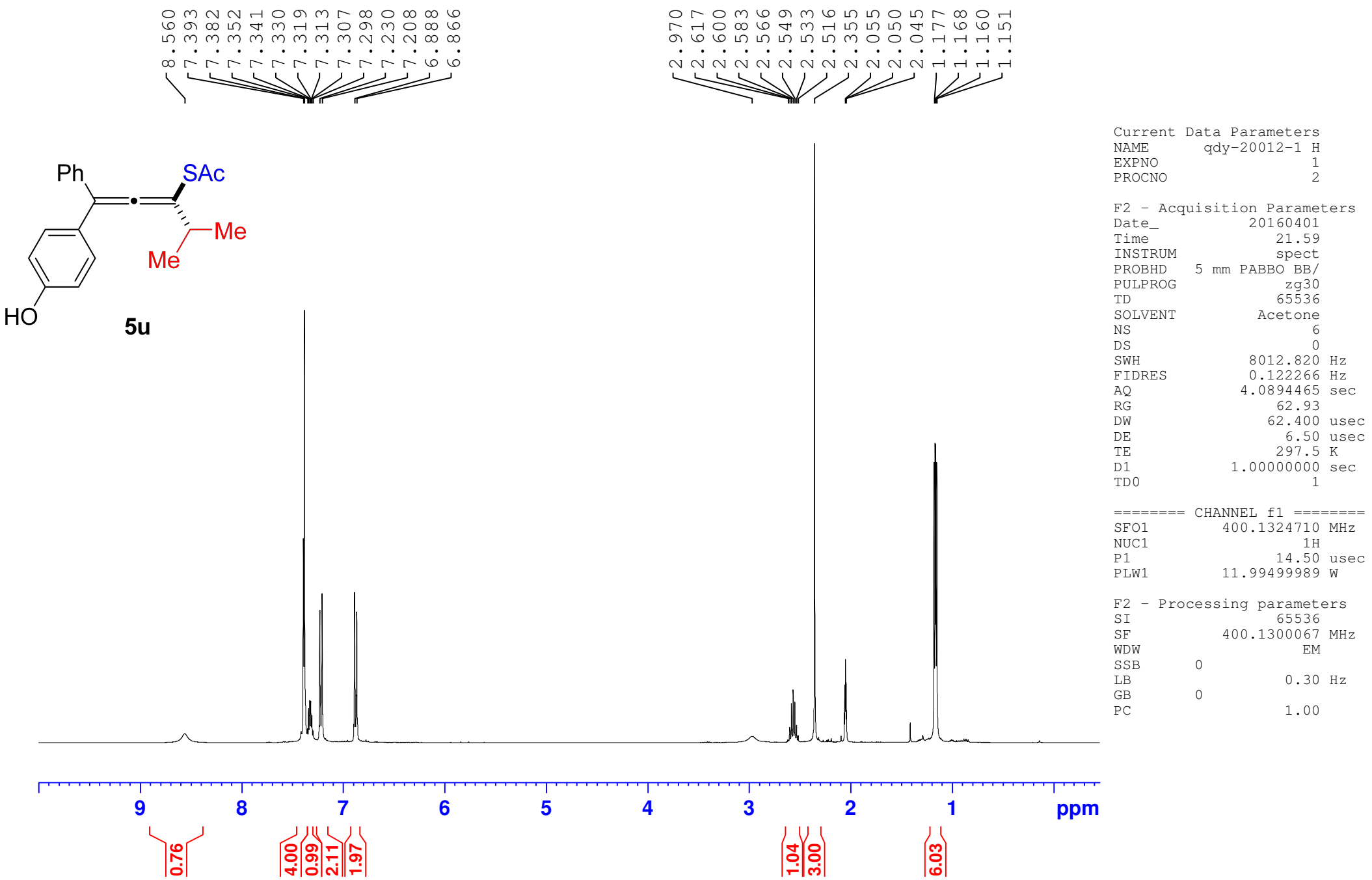


Supplementary Figure 145. ^{1}H NMR spectrum for 5t



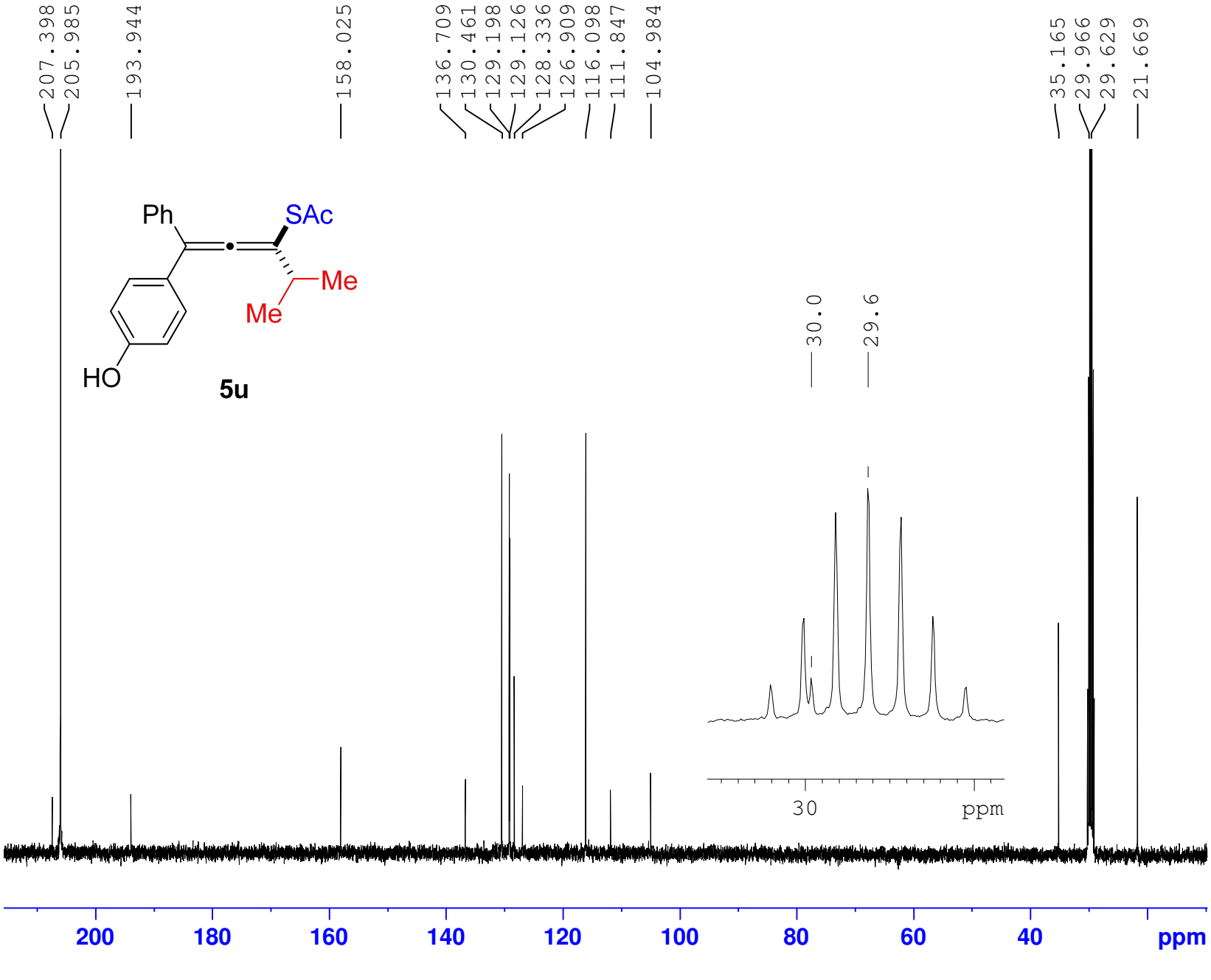

```
Current Data Parameters
NAME       qdy-20103-1 C
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160408
Time               13.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  116
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6126993 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 146. ^{13}C NMR spectrum for 5t



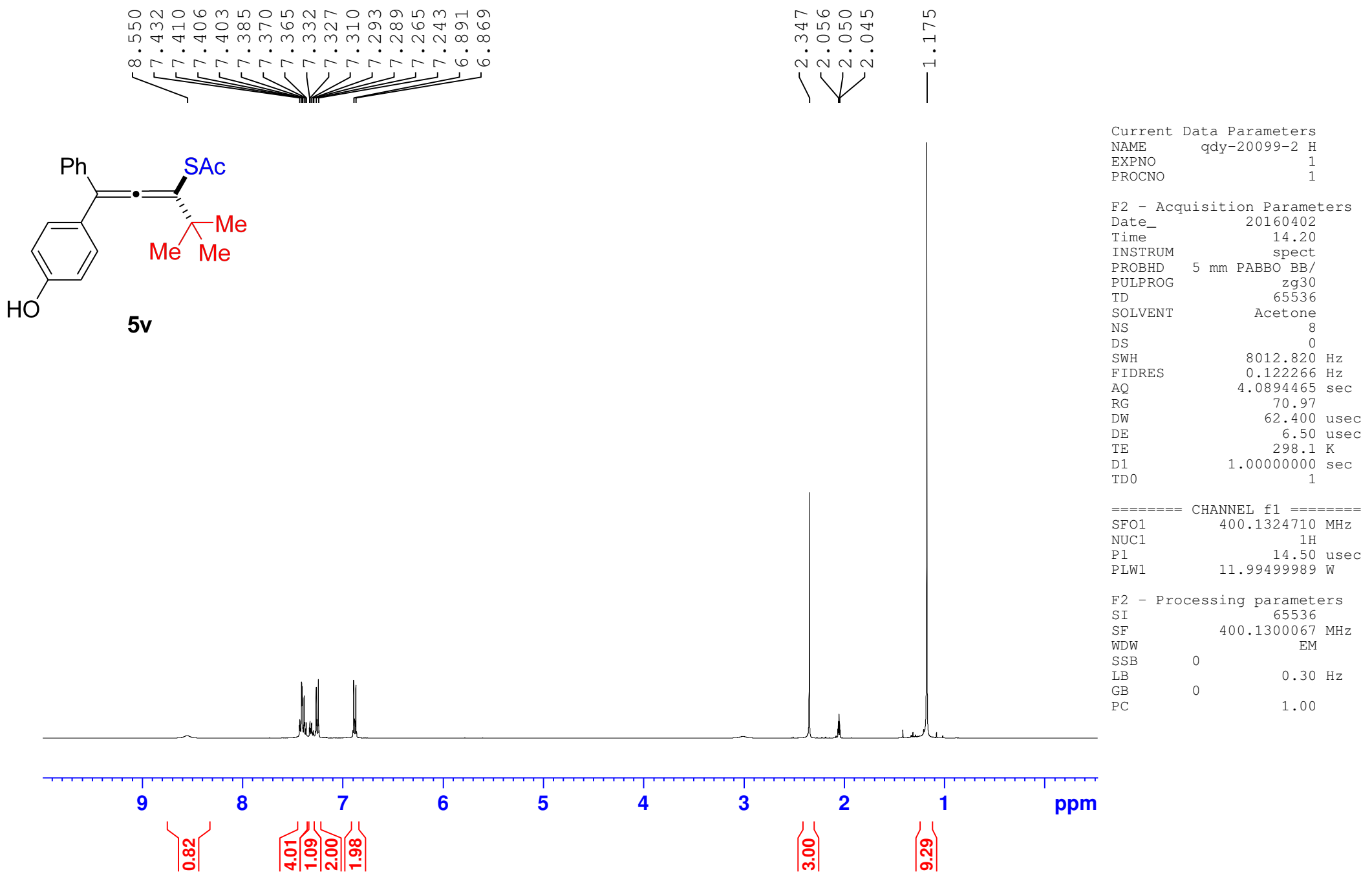

```
Current Data Parameters
NAME       qdy-20012-1 H
EXPNO                 1
PROCNO                2

F2 - Acquisition Parameters
Date_          20160401
Time             21.59
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT        Acetone
NS                   6
DS                   0
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               62.93
DW              62.400 usec
DE               6.50 usec
TE              297.5 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1300067 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 147. ^{1}H NMR spectrum for 5u

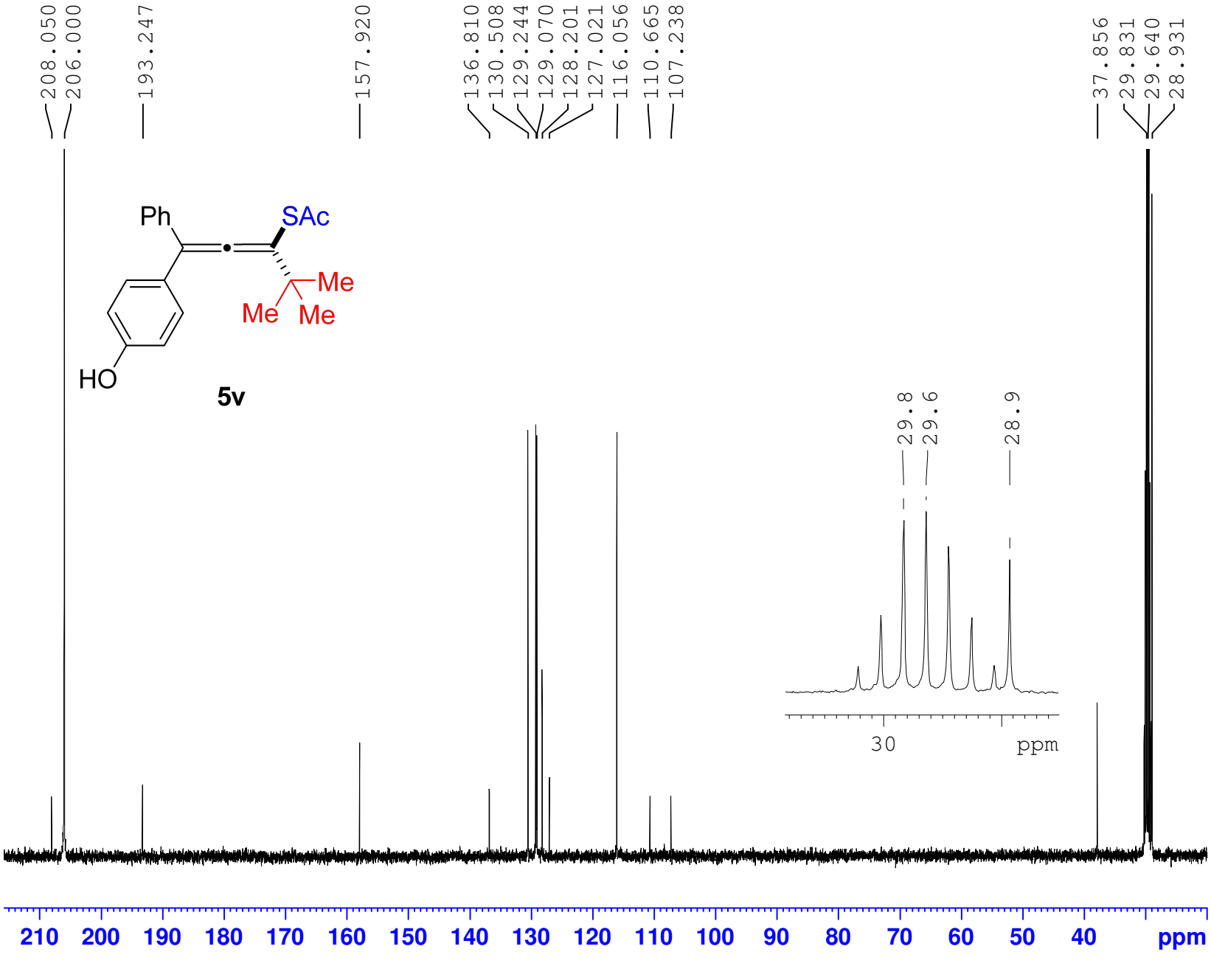



```
Current Data Parameters
NAME       qdy-20012-1 C
EXPNO                  1
PROCNO                 2

F2 - Acquisition Parameters
Date_           20160401
Time              21.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                   60
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE               297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127029 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 148. ^{13}C NMR spectrum for 5u

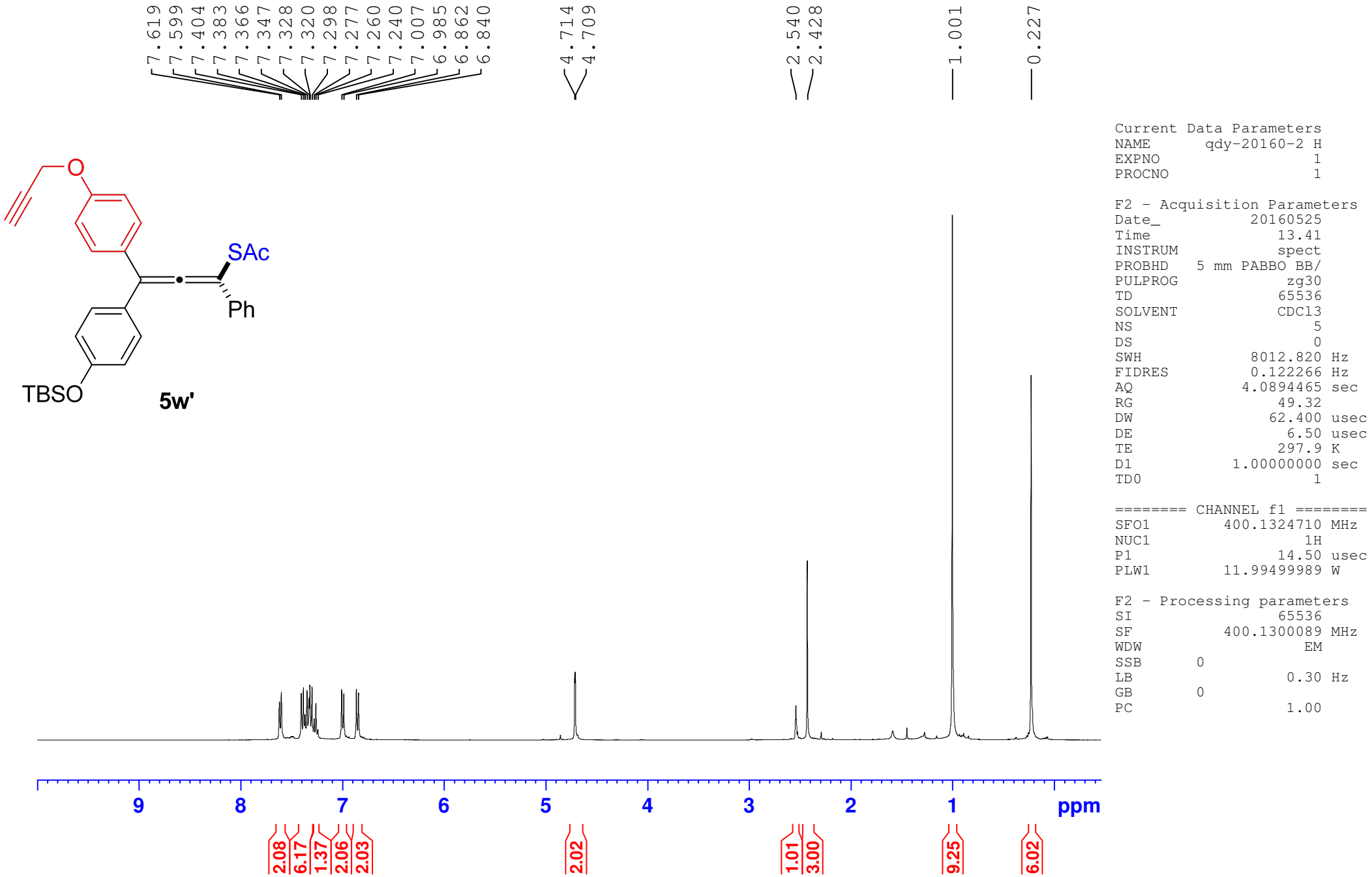


Supplementary Figure 149. ^{1}H NMR spectrum for 5v

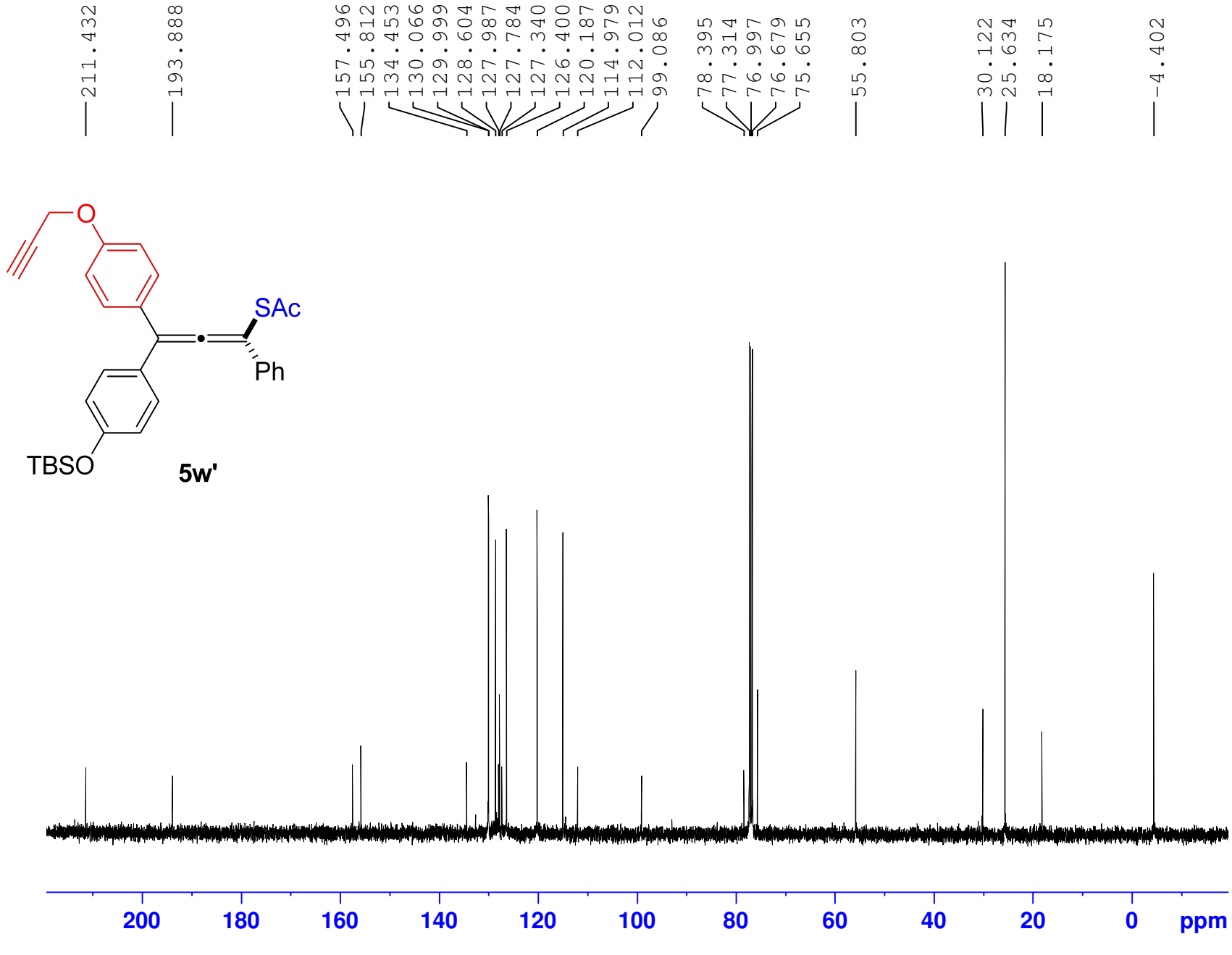



```
Current Data Parameters
NAME       qdy-20099-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160402
Time              14.14
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         Acetone
NS                  130
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127029 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 150. ^{13}C NMR spectrum for 5v

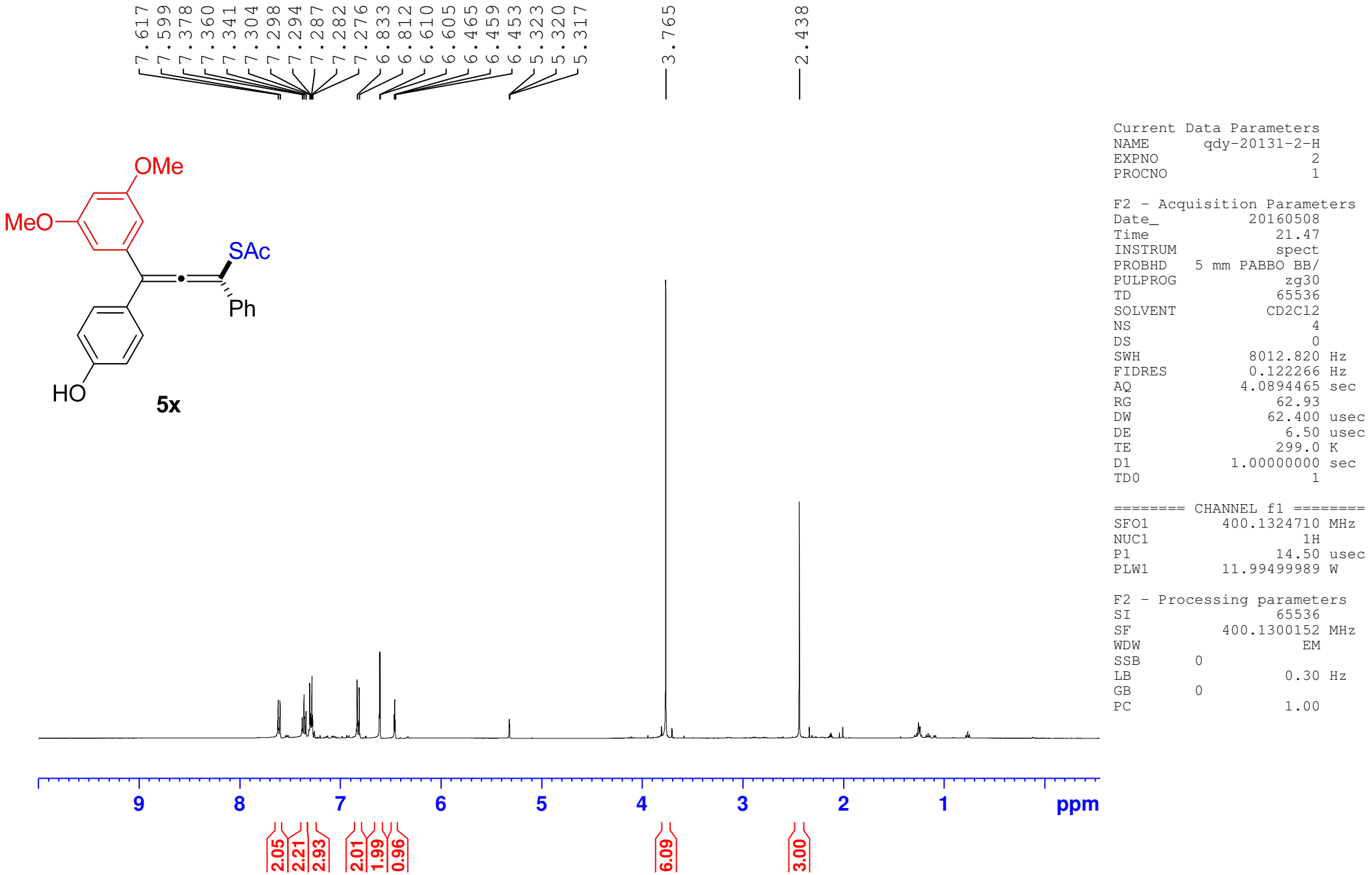


Supplementary Figure 151. ^{1}H NMR spectrum for 5w'

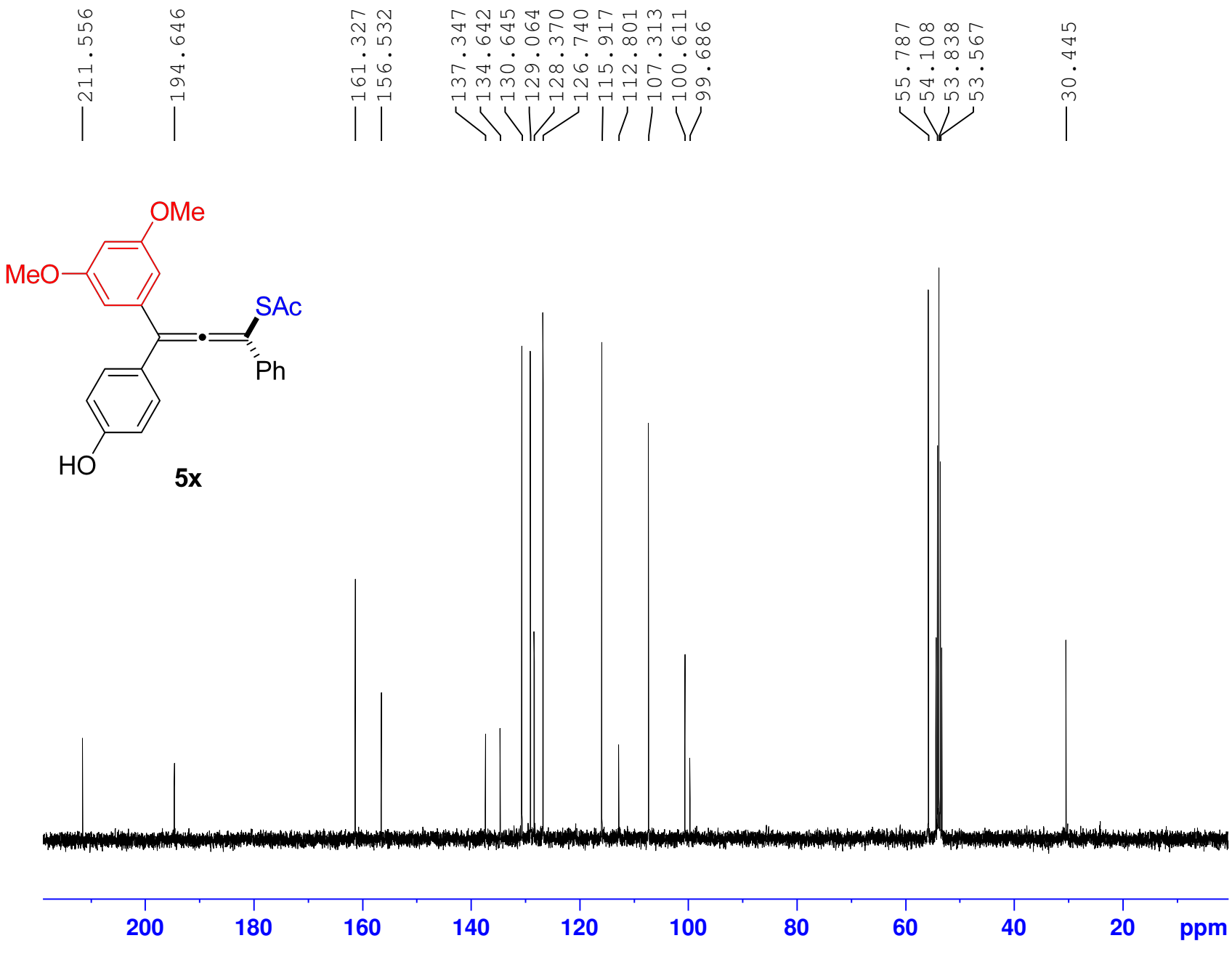



```
Current Data Parameters
NAME      qdy-20160-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160525
Time              13.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 126
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                299.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127744 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 152. ^{13}C NMR spectrum for 5w'

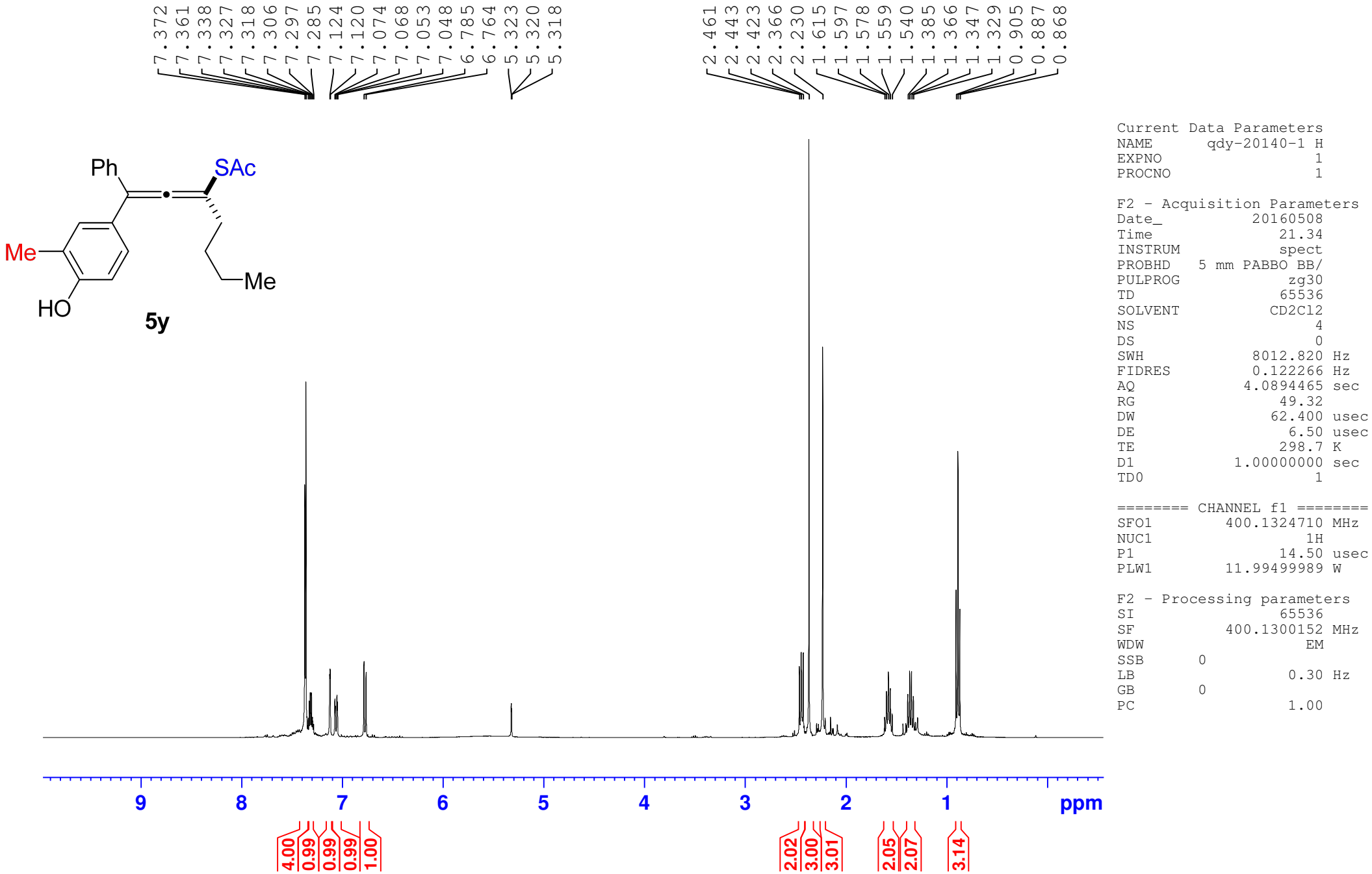


Supplementary Figure 153. ^{1}H NMR spectrum for 5x



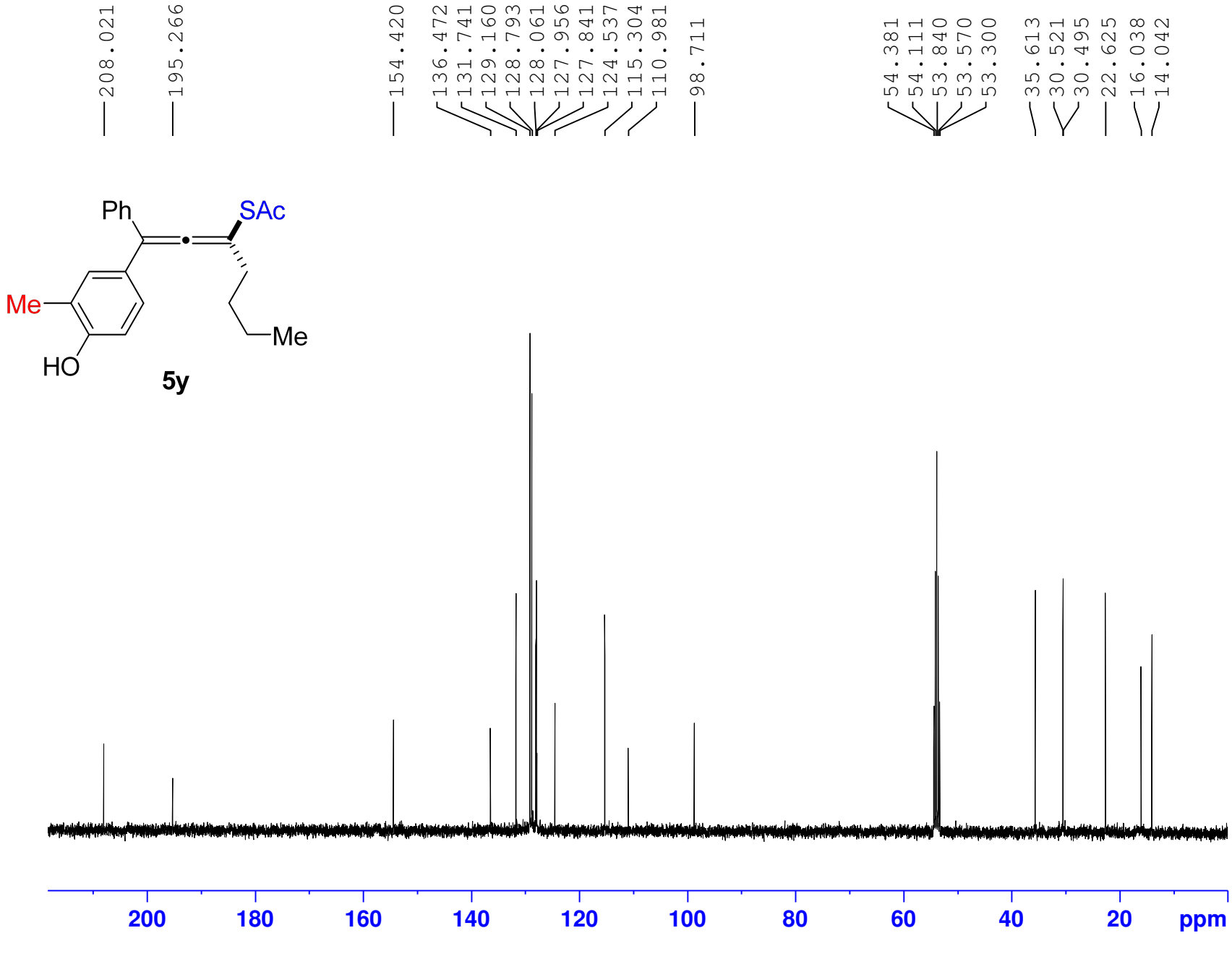

```
Current Data Parameters
NAME      qdy-20131-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160429
Time              19.23
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CD2Cl2
NS                 188
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE               297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1               13C
P1                9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                32768
SF          100.6127299 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 154. ^{13}C NMR spectrum for 5x



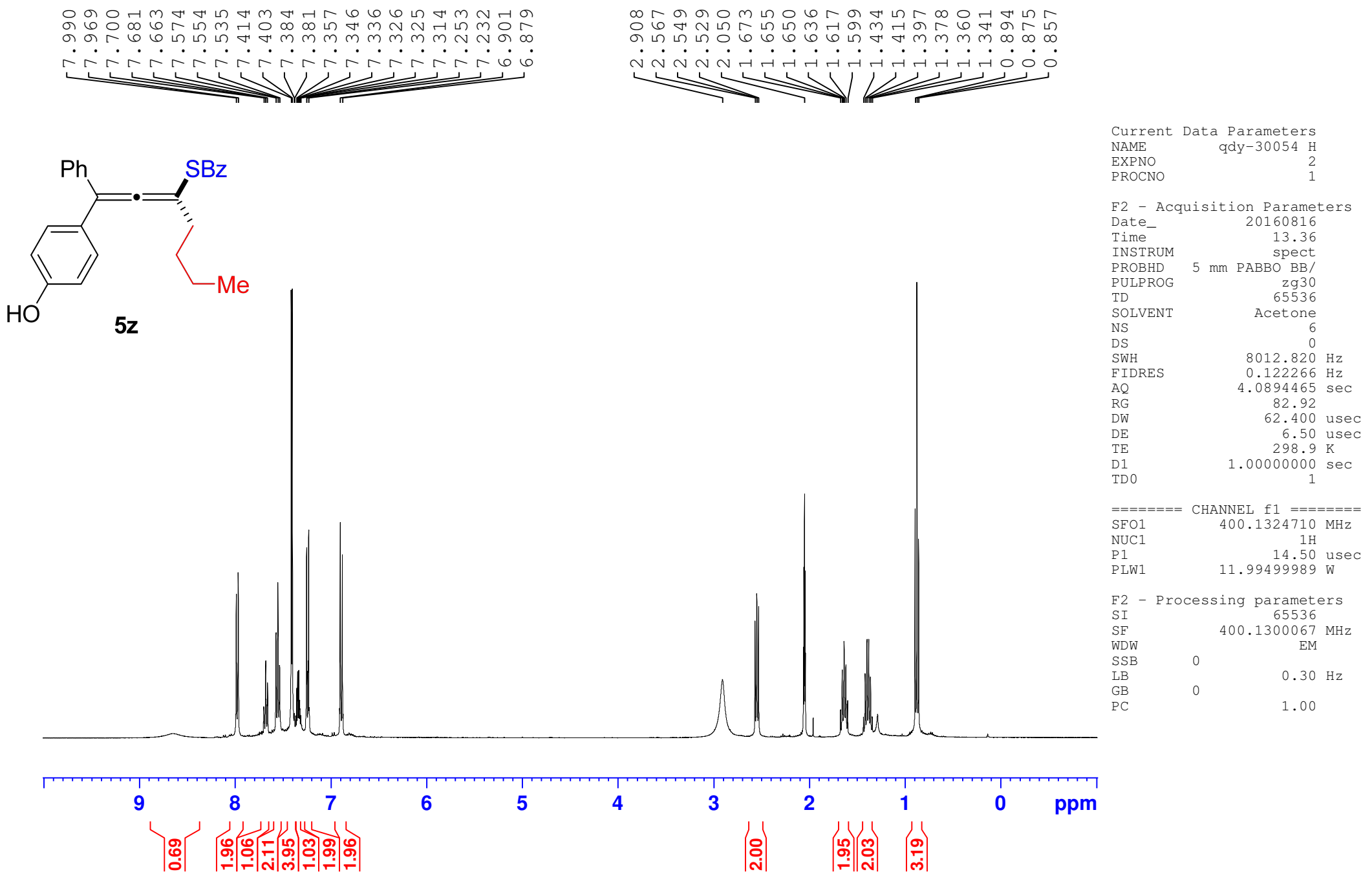

```
Current Data Parameters
NAME       qdy-20140-1 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160508
Time              21.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CD2Cl2
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                49.32
DW               62.400 usec
DE                6.50 usec
TE                298.7 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300152 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 155. ^{1}H NMR spectrum for 5y




```
Current Data Parameters
NAME       qdy-20140-1 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160508
Time              21.39
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT         CD2Cl2
NS                  149
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127262 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 156. ^{13}C NMR spectrum for 5y



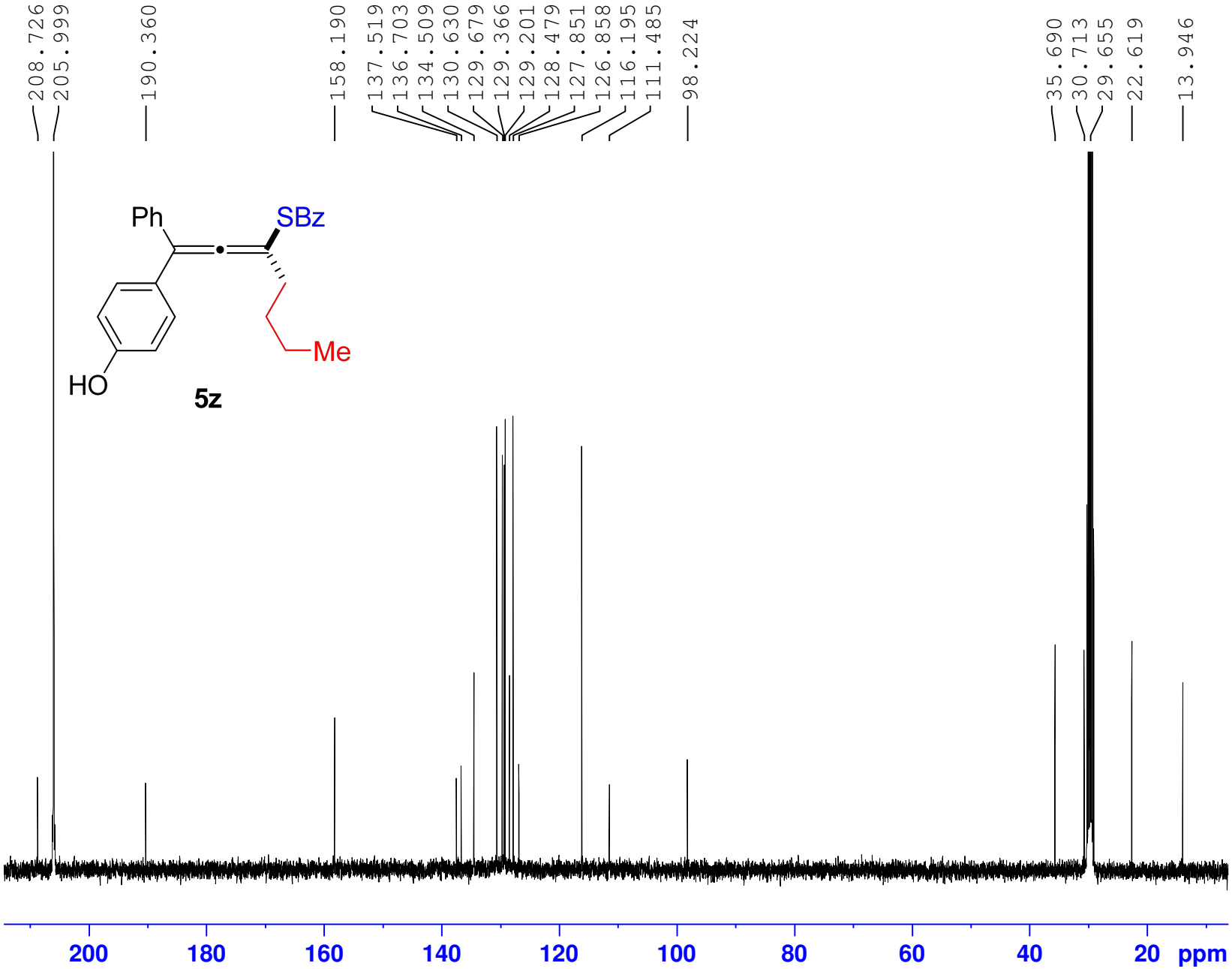
Current Data Parameters
NAME qdy-30054 H
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160816
Time 13.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 6
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

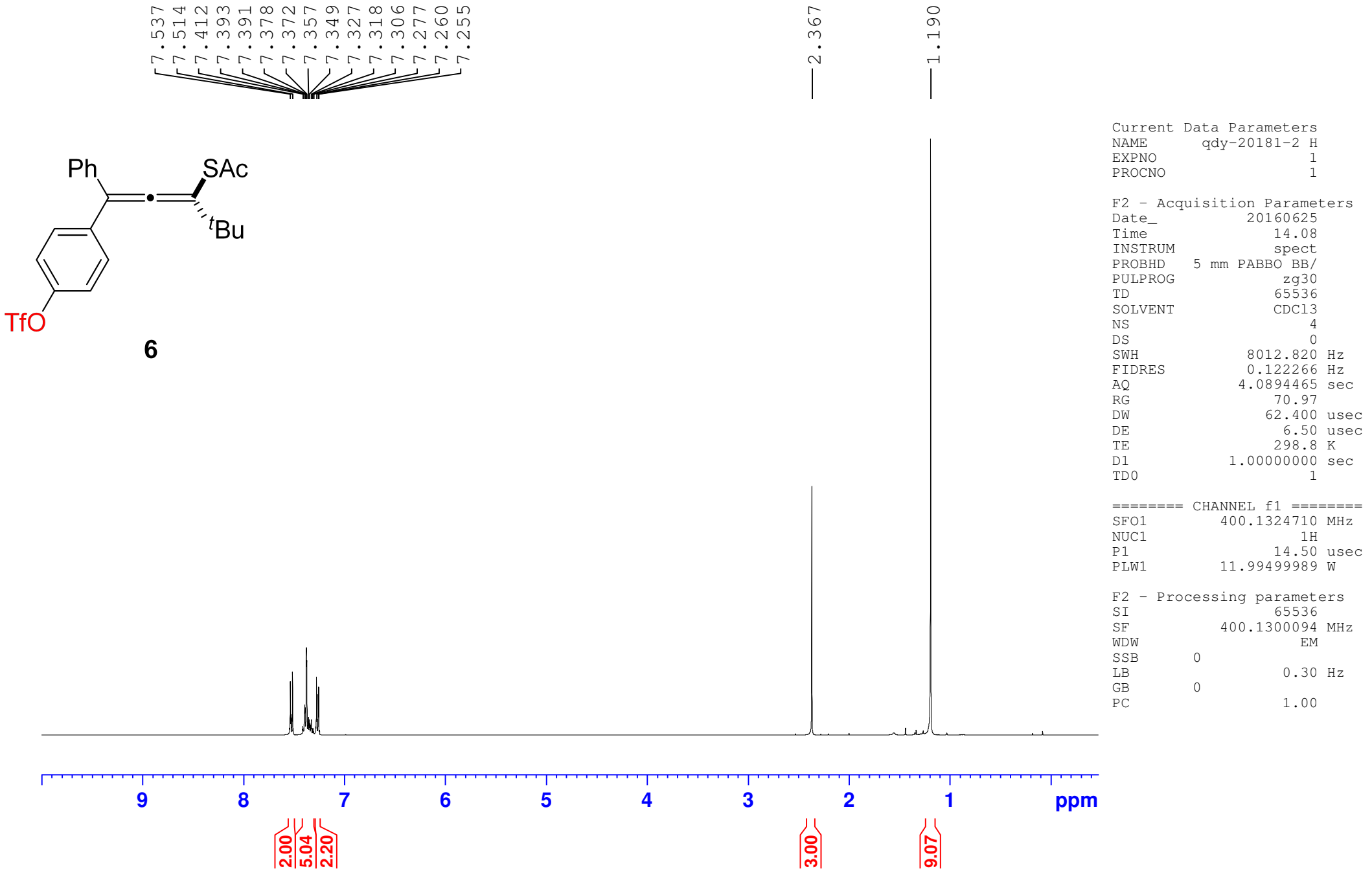
F2 - Processing parameters
SI 65536
SF 400.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 157. ^{1}H NMR spectrum for 5z

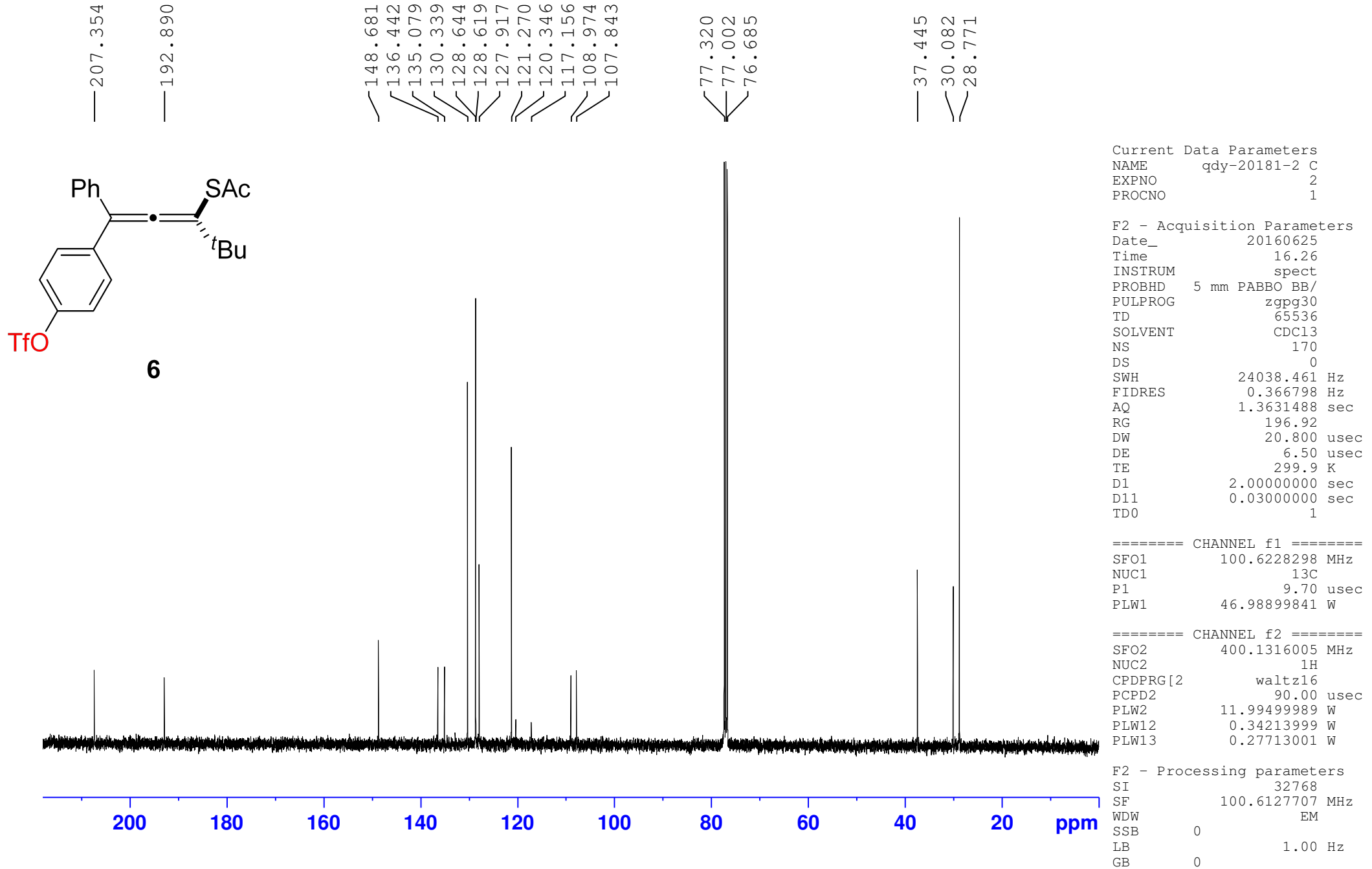



```
Current Data Parameters
NAME         qdy-30054 C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160815
Time               21.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   244
DS                     0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               196.92
DW               20.800 usec
DE                6.50 usec
TE                300.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1        46.98899841 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        11.99499989 W
PLW12        0.34213999 W
PLW13        0.27713001 W

F2 - Processing parameters
SI                 32768
SF          100.6126978 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

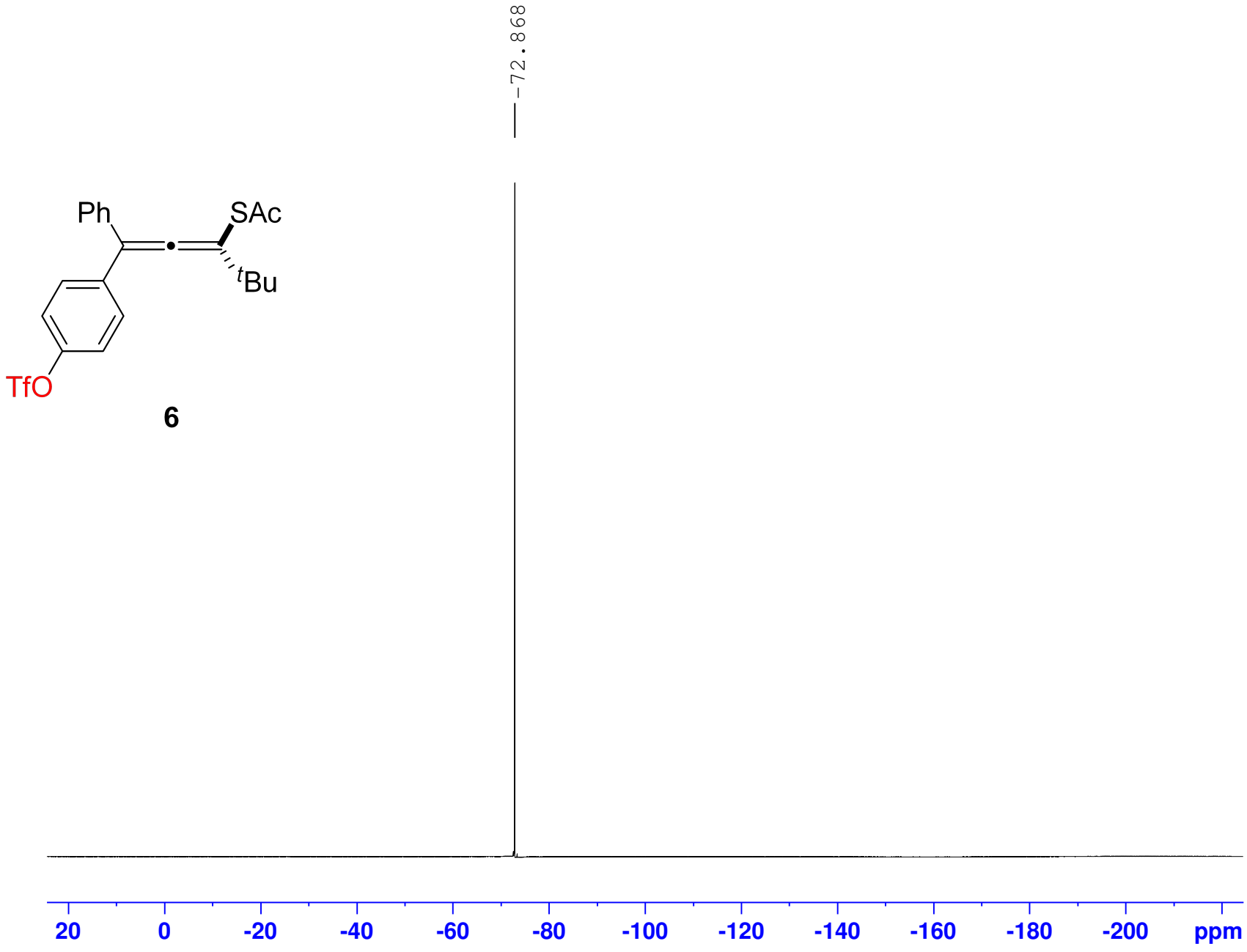
Supplementary Figure 158. ^{13}C NMR spectrum for 5z



Supplementary Figure 159. ^{1}H NMR spectrum for 6

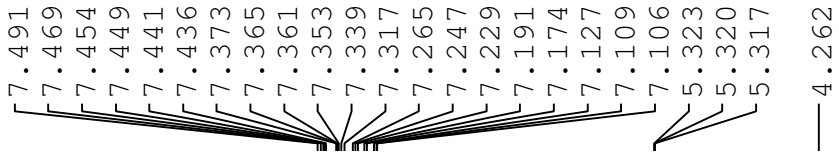


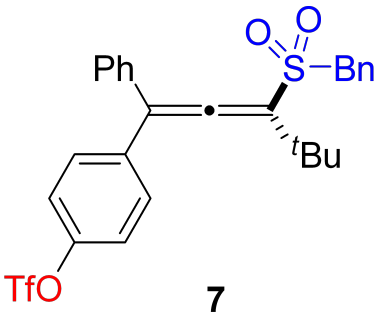
Supplementary Figure 160. ^{13}C NMR spectrum for 6



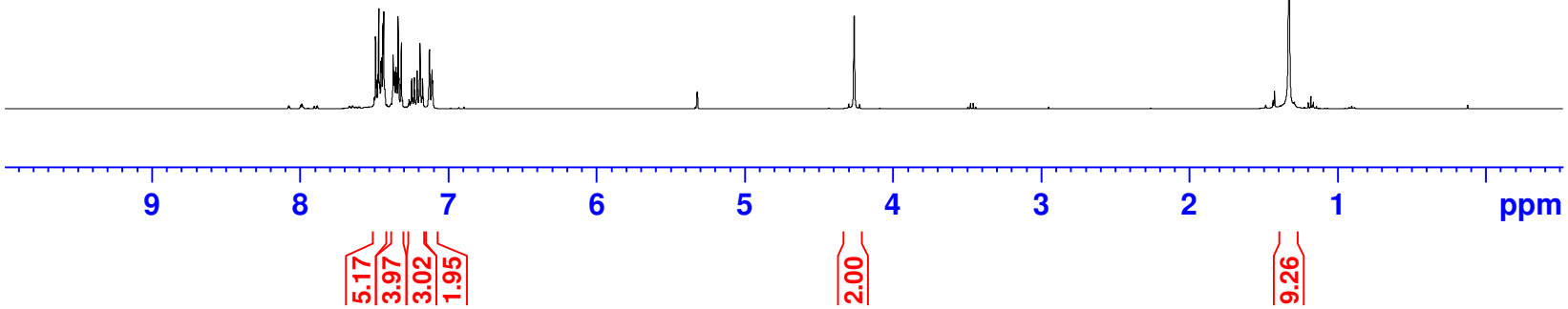

```
Current Data Parameters
NAME      qdy-20181-2 F
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20160625
Time              16.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH           93750.000 Hz
FIDRES         1.430511 Hz
AQ            0.3495253 sec
RG               196.92
DW                5.333 usec
DE                6.50 usec
TE                299.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         376.4607162 MHz
NUC1                19F
P1                14.70 usec
PLW1         15.99600029 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           376.4983660 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 161. ^{19}F NMR spectrum for 6



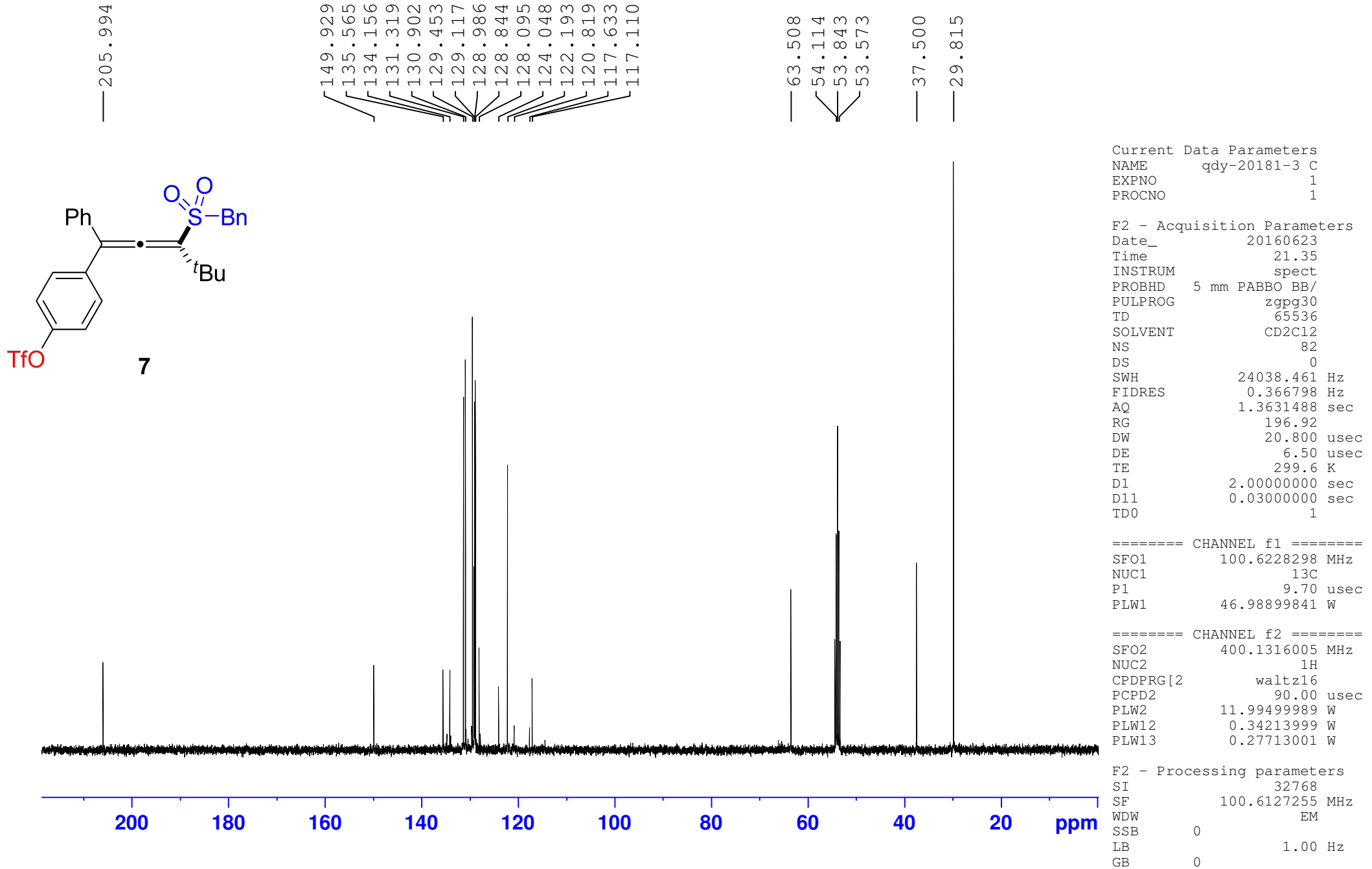


7

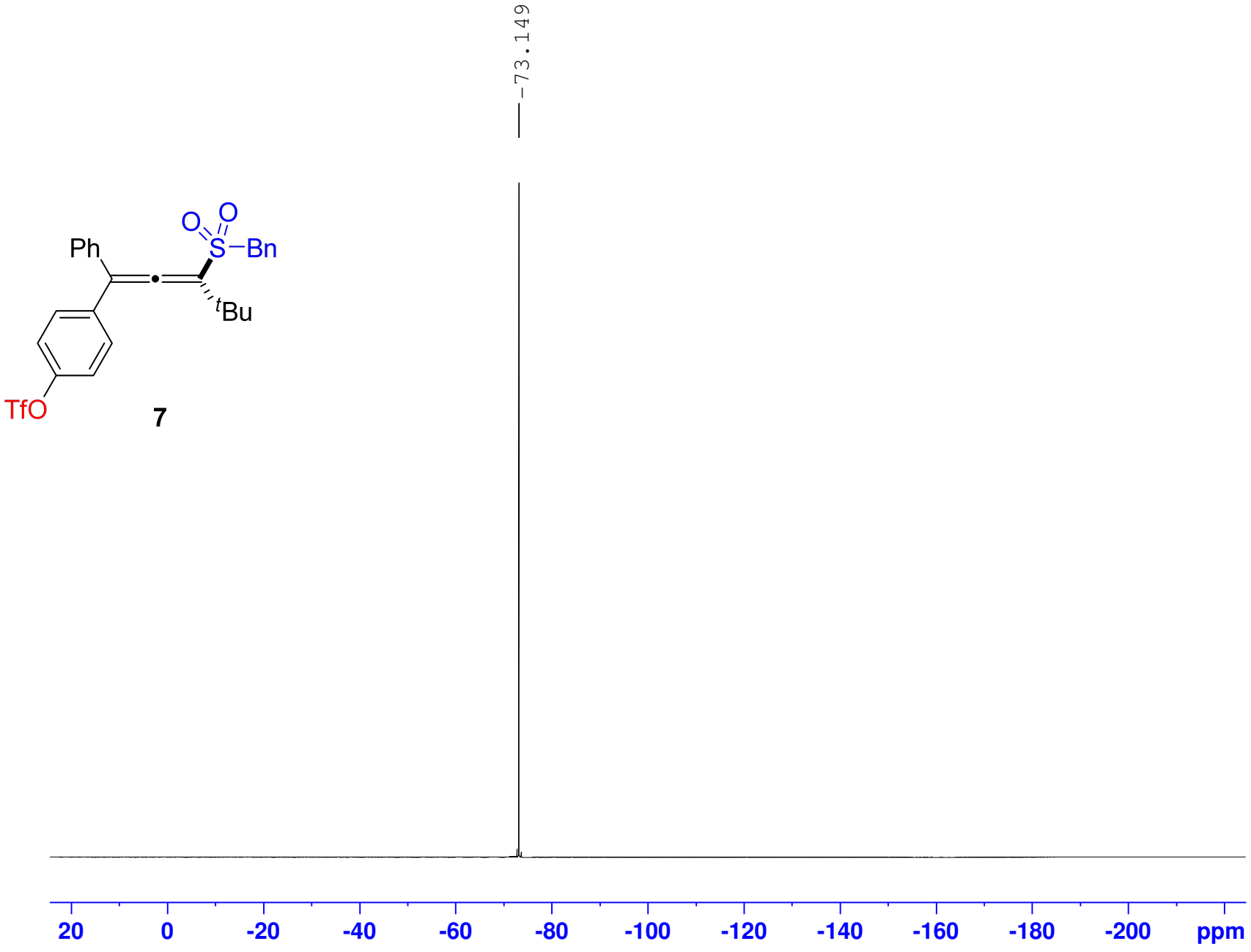



```
Current Data Parameters
NAME       qdy-20181-3 H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20160623
Time              21.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT        CD2Cl2
NS                   4
DS                   0
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               34.77
DW               62.400 usec
DE                6.50 usec
TE                298.7 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.50 usec
PLW1        11.99499989 W

F2 - Processing parameters
SI                65536
SF          400.1300152 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 162. ^{1}H NMR spectrum for 7

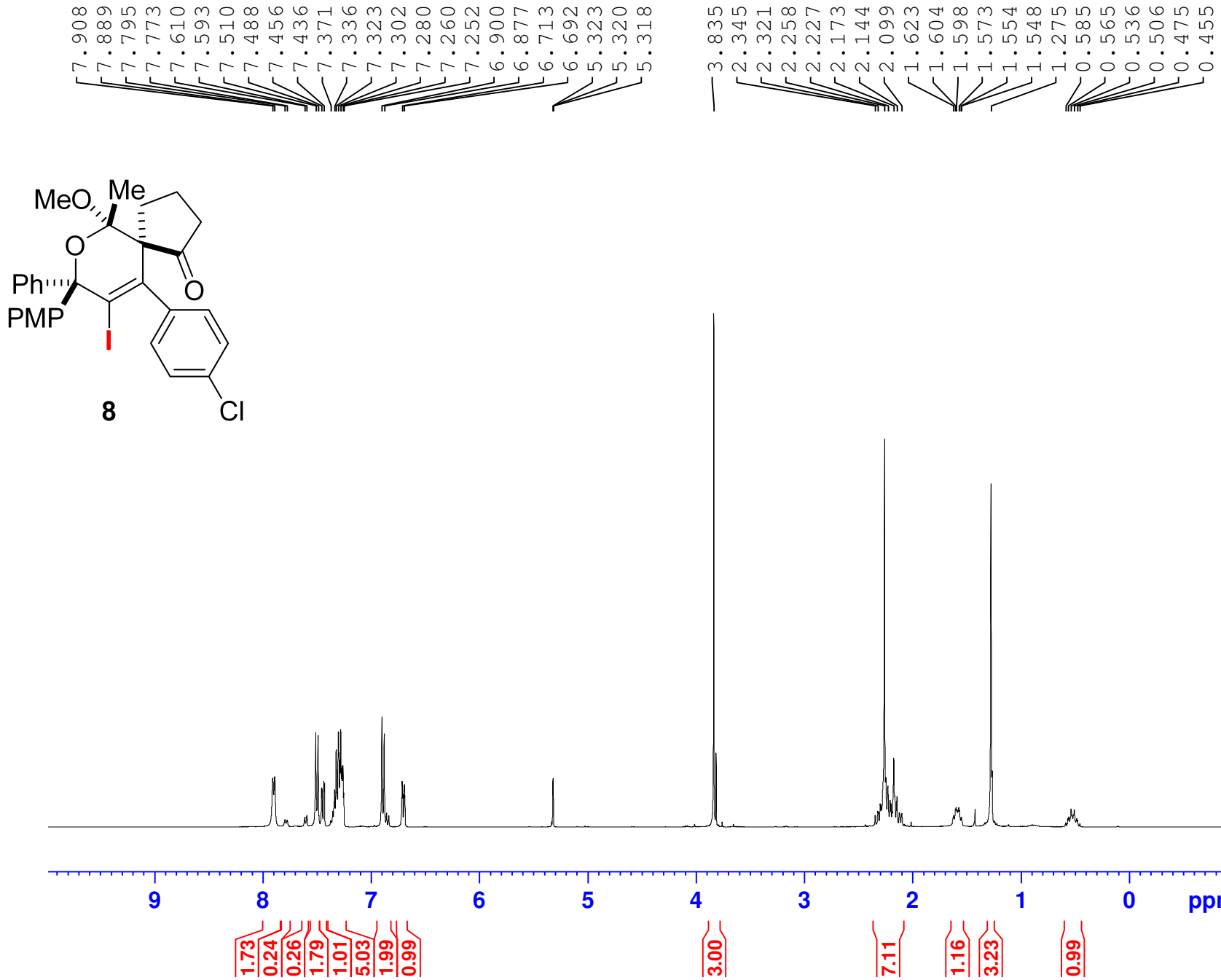


Supplementary Figure 163. ^{13}C NMR spectrum for 7



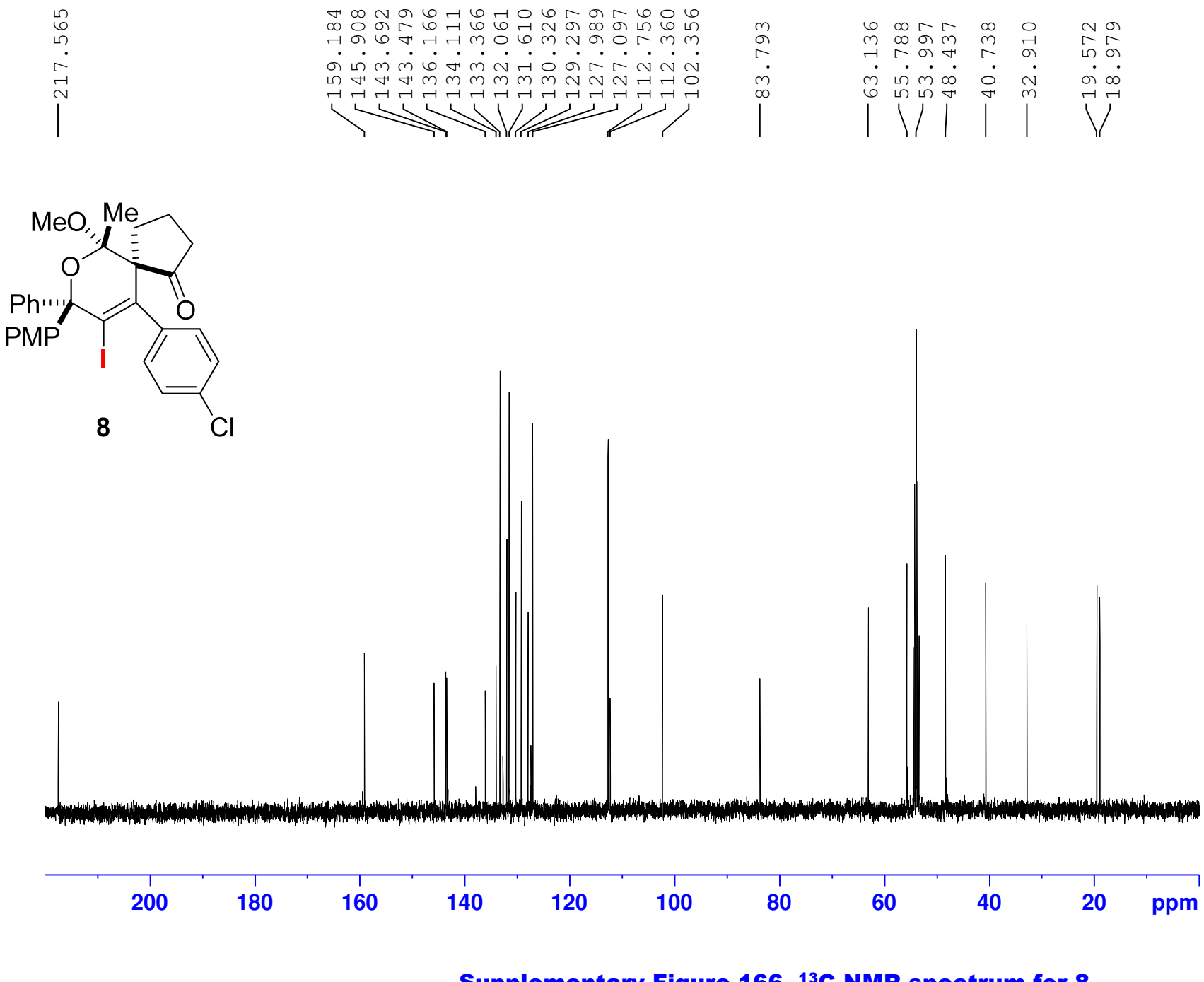

```
Current Data Parameters
NAME      qdy-20181-3 F
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20160623
Time              21.39
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                    4
DS                    0
SWH           93750.000 Hz
FIDRES         1.430511 Hz
AQ            0.3495253 sec
RG               196.92
DW                5.333 usec
DE                6.50 usec
TE                299.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         376.4607162 MHz
NUC1                19F
P1                14.70 usec
PLW1         15.99600029 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                32768
SF           376.4983660 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 164. ^{19}F NMR spectrum for 7



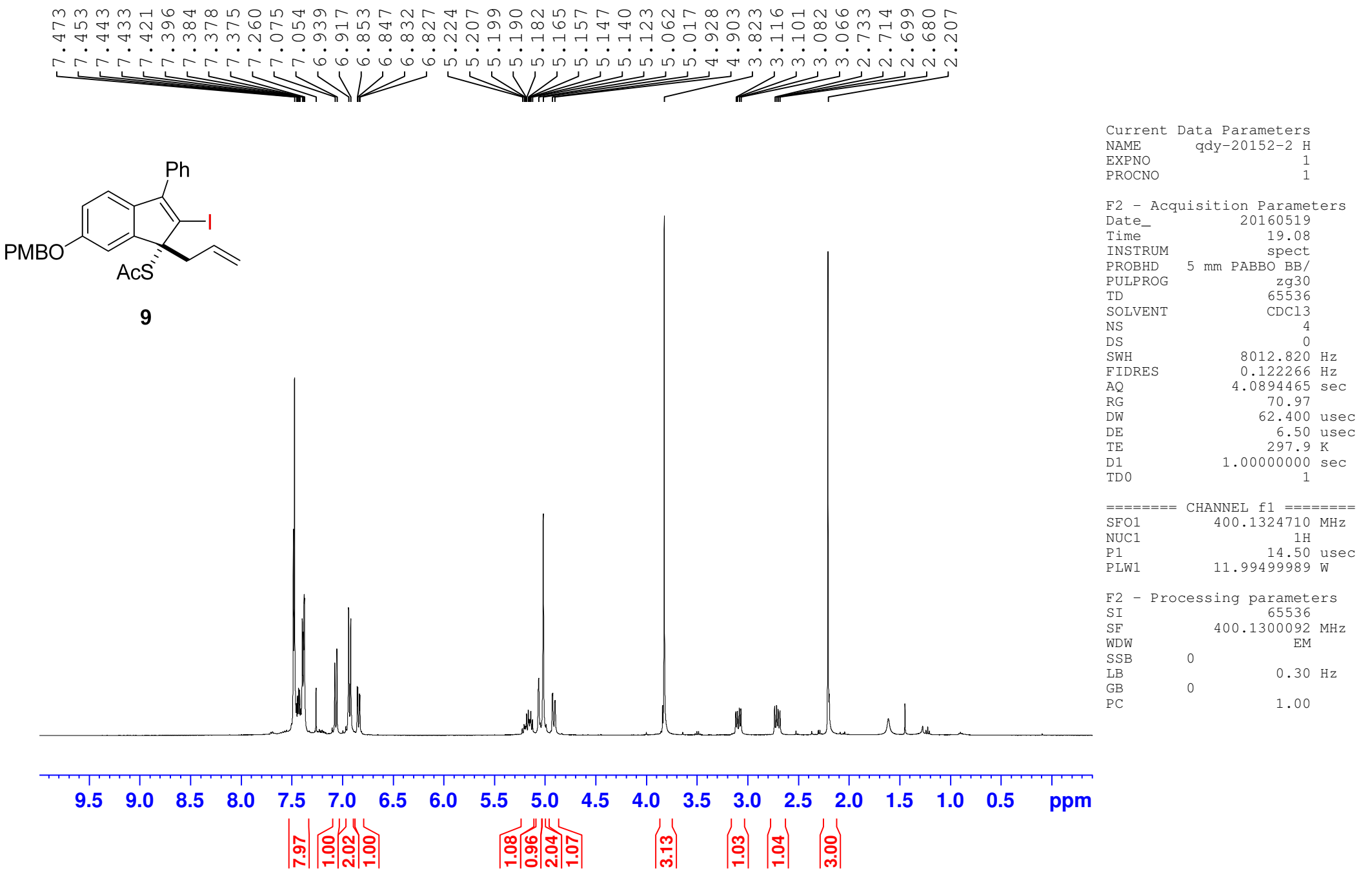

```
Current Data Parameters
NAME       qdy-40038-2 H
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20170114
Time              20.10
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT          CD2Cl2
NS                    4
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                49.32
DW                62.400 usec
DE                 6.50 usec
TE                296.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                14.50 usec
PLW1         11.99499989 W

F2 - Processing parameters
SI                65536
SF           400.1300157 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Supplementary Figure 165. ^{1}H NMR spectrum for 8

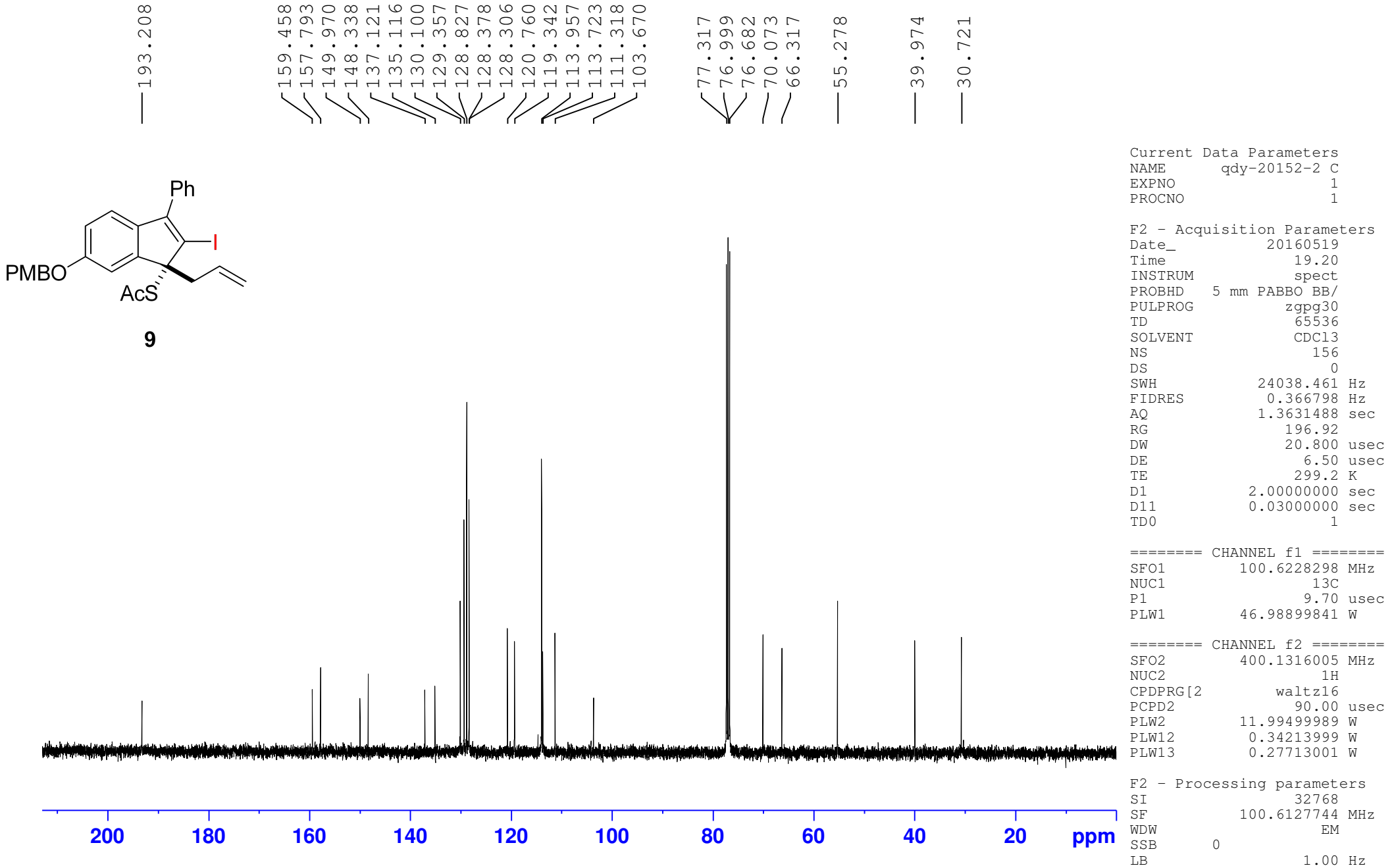



```
Current Data Parameters
NAME       qdy-40038-1 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170115
Time              17.36
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT          CD2Cl2
NS                   74
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                296.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127153 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Figure 166. ^{13}C NMR spectrum for 8

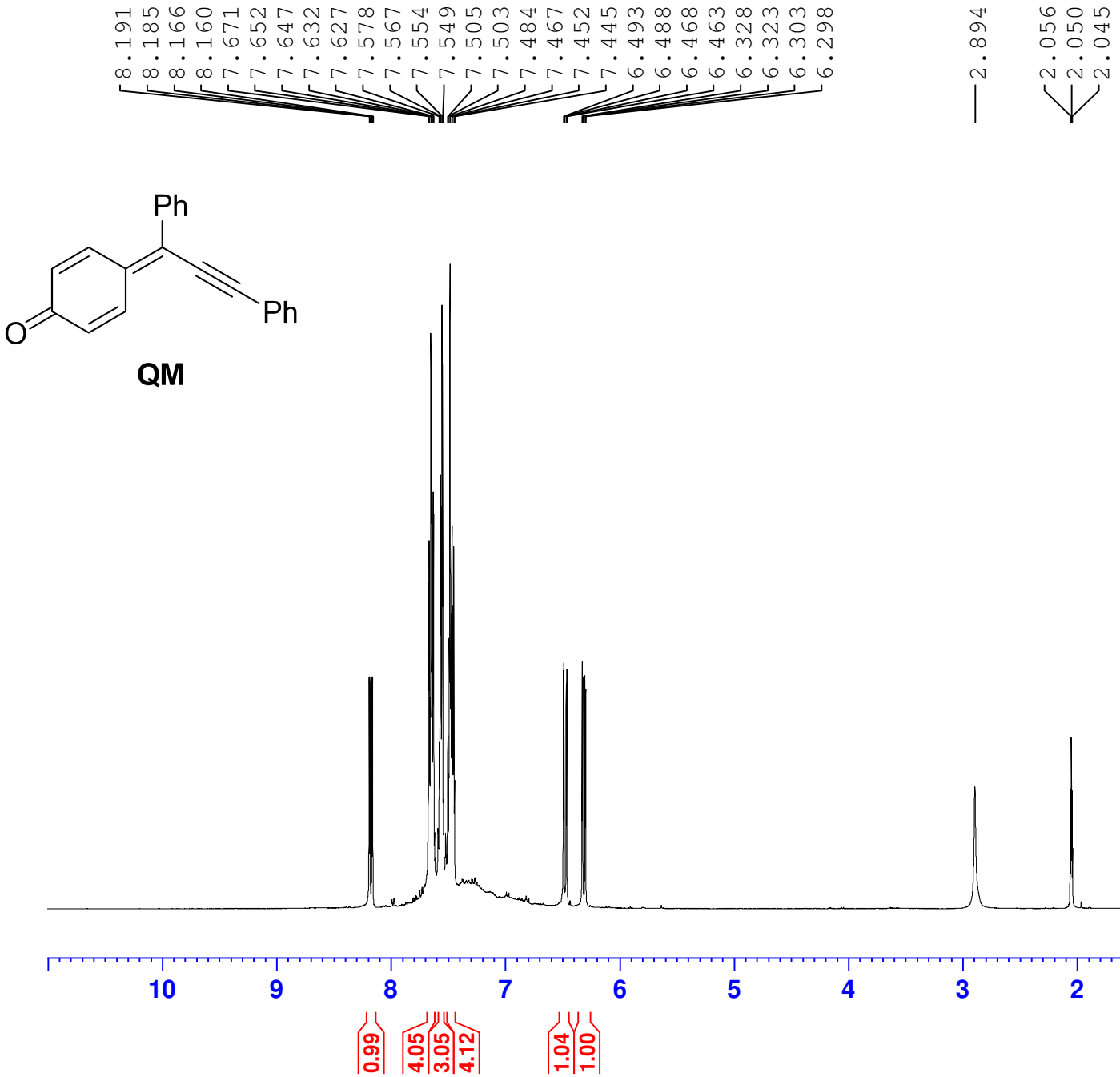


Supplementary Figure 167. ^{1}H NMR spectrum for 9




```
Current Data Parameters
NAME       qdy-20152-2 C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160519
Time              19.20
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  156
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6127744 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 168. ^{13}C NMR spectrum for 9



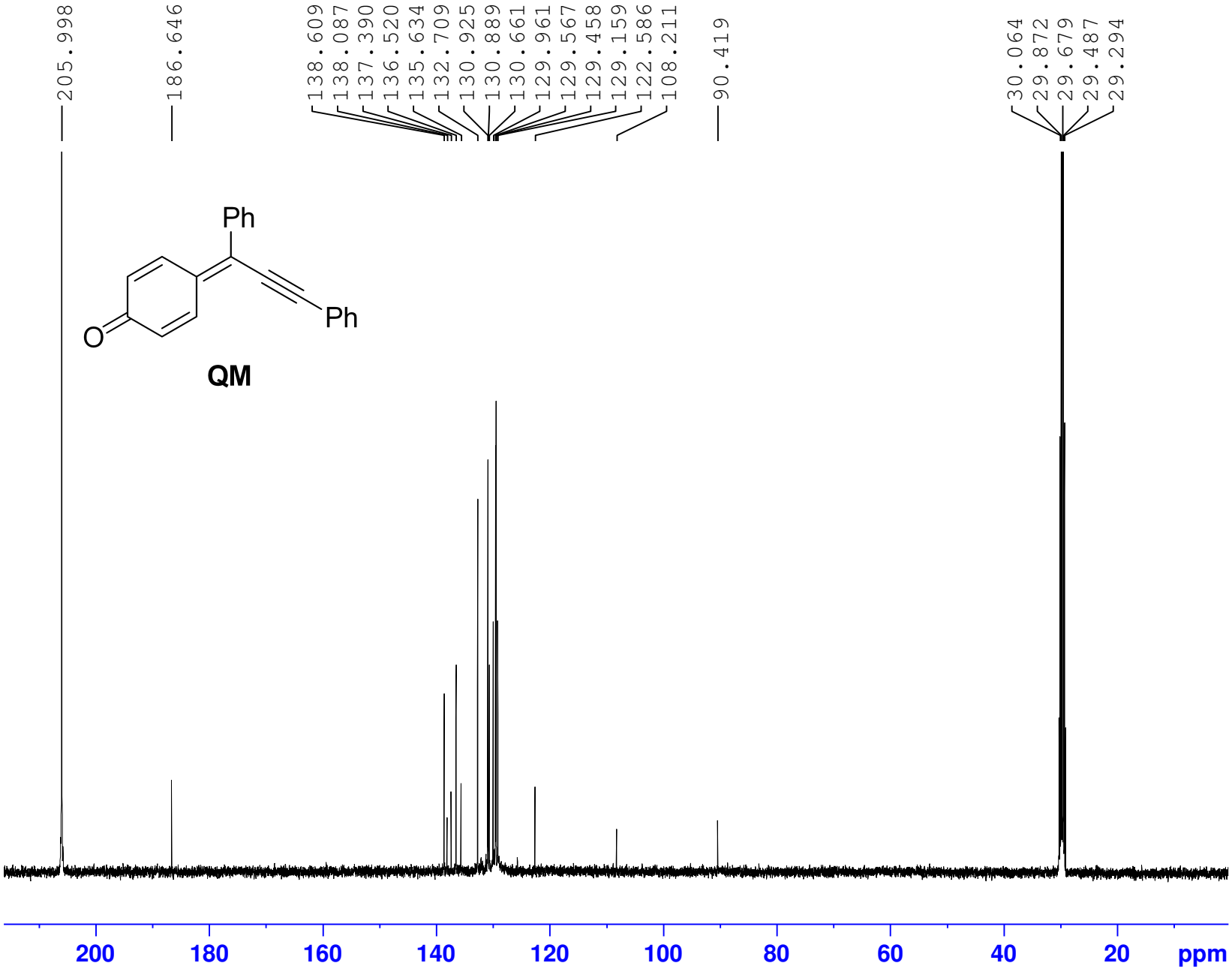
Current Data Parameters
NAME qdy-20167-2 H
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160607
Time 15.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

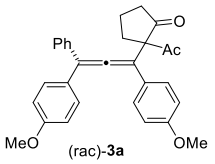
F2 - Processing parameters
SI 65536
SF 400.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Supplementary Figure 169. ^{1}H NMR spectrum for QM



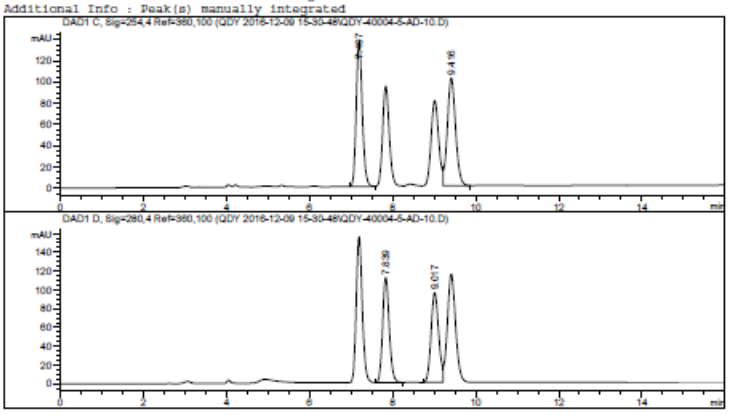

```
Current Data Parameters
NAME      qdy-20167-2 C
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20160607
Time               16.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  191
DS                    0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                 6.50 usec
TE                297.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.70 usec
PLW1         46.98899841 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         11.99499989 W
PLW12         0.34213999 W
PLW13         0.27713001 W

F2 - Processing parameters
SI                 32768
SF           100.6126984 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Figure 170. ^{13}C NMR spectrum for QM



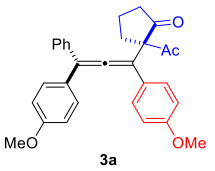
(rac)-**3a**

```
Acq. Operator   :                                   Seq. Line :  22
Acq. Instrument : Instrument 1                      Location : Vial 35
Injection Date  : 12/10/2016 12:38:52 AM               Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-09 15-30-48\AD-10-30.M
Last changed    : 11/30/2015 12:57:24 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/17/2016 10:34:06 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



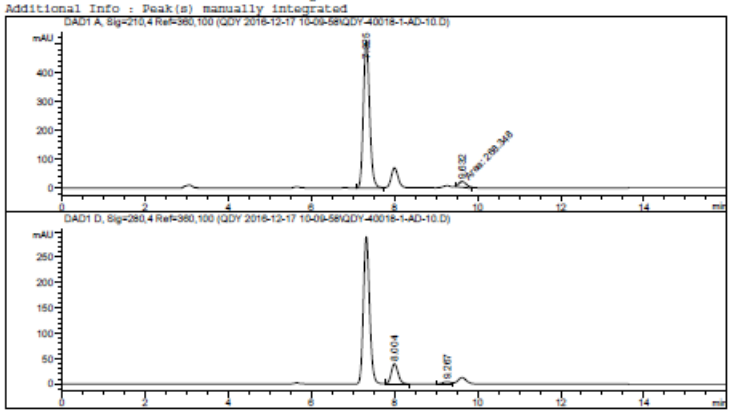

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   7.197 BB    0.1562 1393.25073  137.51768  49.9470
   2   9.416 VB    0.2099 1396.20593  101.89838  50.0530

Totals :                  2789.45667  239.41606
```

```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   7.839 BB    0.1686 1232.49475  111.78729  50.5441
   2   9.017 BV    0.1956 1205.95996   95.34112  49.4559

Totals :                  2438.45471  207.12841

=====================================================================
                      *** End of Report ***
```



3a

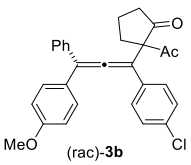
```
Acq. Operator   :                                   Seq. Line :  18
Acq. Instrument : Instrument 1                      Location : Vial 31
Injection Date  : 12/17/2016 6:13:43 PM                Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-17 10-09-58\AD-10-25.M
Last changed    : 12/17/2016 6:33:07 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/17/2016 10:21:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   7.325 BV    0.1588 5284.21094  510.25525  95.1671
   2   9.632 MM    0.2169  268.34750   20.61820   4.8329

Totals :                  5552.55844  530.87345
```

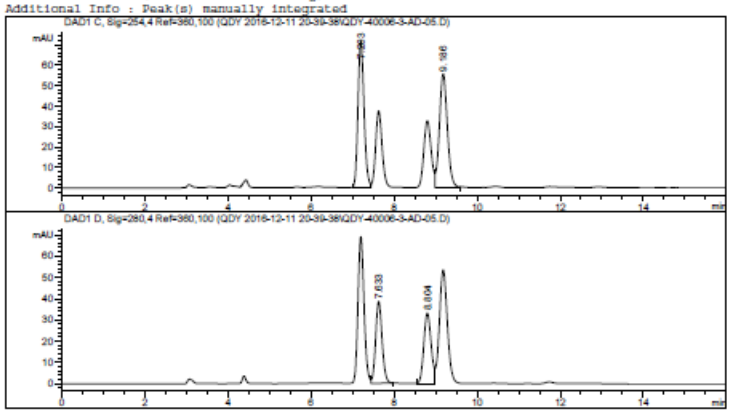
```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.004 BB    0.1768  453.72427   39.86567  89.5725
   2   9.267 BV    0.1940   52.81998    4.21946  10.4275

Totals :                   506.54425   44.08513

=====================================================================
                      *** End of Report ***
```

Supplementary Figure 171. HPLC spectrum for 3a



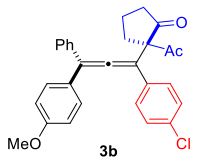
(rac)-**3b**

```
Acq. Operator   :                          Seq. Line :  8
Acq. Instrument : Instrument 1              Location : Vial 32
Injection Date  : 12/11/2016 11:13:09 PM       Inj :  1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-11 20-39-38\AD-05-20.M
Last changed    : 10/14/2015 3:51:32 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/17/2016 10:34:06 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



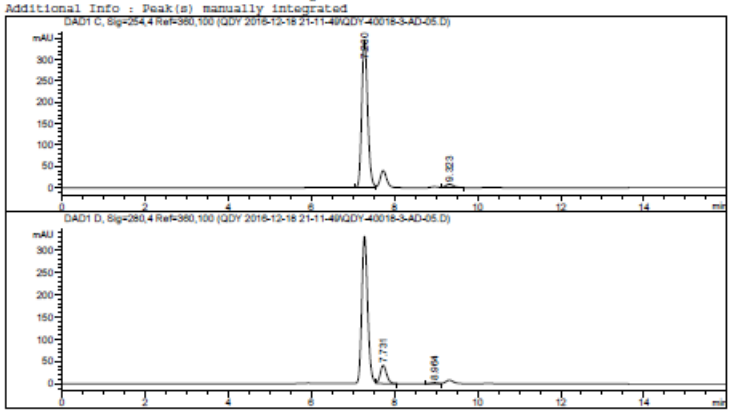

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.203 BV    0.1562  727.00128   71.74940  49.5600
   2   9.186 VB    0.2056  739.91150   55.46935  50.4400

Totals :                   1466.91278  127.21876
```

```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.633 VB    0.1661  415.61536   38.45949  50.6287
   2   8.804 BV    0.1910  405.29398   33.07433  49.3713

Totals :                   820.90933   71.53382

                        *** End of Report ***
```



3b

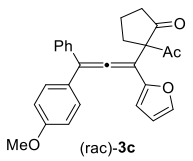
```
Acq. Operator   :                          Seq. Line :  2
Acq. Instrument : Instrument 1              Location : Vial 33
Injection Date  : 12/18/2016 9:26:08 PM        Inj :  1
                                          Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-18 21-11-49\AD-05-20.M
Last changed    : 12/18/2016 9:25:14 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-05-30-1.3.M
Last changed    : 12/18/2016 9:48:04 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.280 BV    0.1504 3380.13574  344.81693  96.7568
   2   9.323 VB    0.2039  113.29745    8.47690   3.2432

Totals :                   3493.43320  353.29382
```

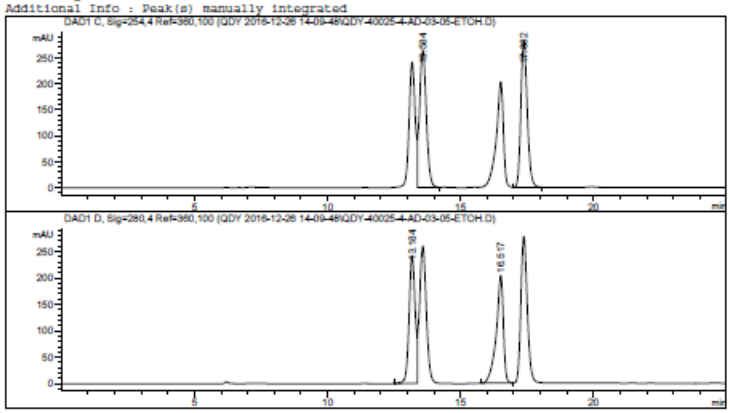
```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.731 VB    0.1628  441.41266   41.25728  94.1989
   2   8.964 BV    0.1809   27.18377    2.31690   5.8011

Totals :                   468.59643   43.57418

                        *** End of Report ***
```

Supplementary Figure 172. HPLC spectrum for 3b



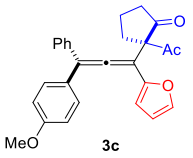
(rac)-**3c**

```
Acq. Operator   :                                   Seq. Line :   4
Acq. Instrument : Instrument 1                       Location : Vial 31
Injection Date  : 12/26/2016 3:06:26 PM                 Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-26 14-09-48\ETOH-AD-03-25-0.5.M
Last changed    : 12/21/2016 2:33:56 PM
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/25/2016 12:31:15 AM
Additional Info : Peak(s) manually integrated
```



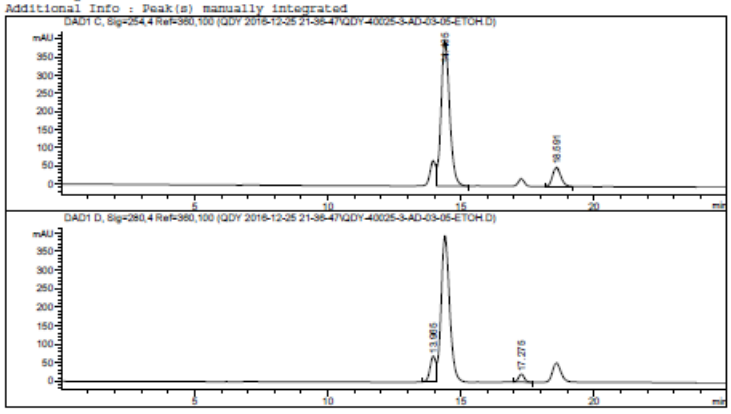

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.584 VB    0.2722 4725.58398  264.86414  49.9491
   2  17.382 VB    0.2570 4735.20996  283.24130  50.0509

Totals :                  9460.79395  548.10544
```

```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.184 BV    0.2422 3813.76855  241.50697  50.1114
   2  16.517 BV    0.2696 3796.81274  203.57631  49.8886
```



3c

```
Acq. Operator   :                                   Seq. Line :   2
Acq. Instrument : Instrument 1                       Location : Vial 31
Injection Date  : 12/25/2016 10:00:57 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-25 21-36-47\ETOH-AD-03-25-0.5.M
Last changed    : 12/25/2016 10:12:41 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/25/2016 12:31:15 AM
Additional Info : Peak(s) manually integrated
```

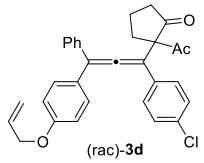



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.405 VB    0.3393 8968.72949  403.54016  88.5462
   2  18.591 BB    0.3383 1160.14307   53.21543  11.4538

Totals :                  1.01289e4   456.75559
```

```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.965 BV    0.2313 1063.85193   70.00453  76.9423
   2  17.275 BB    0.2337  318.80972   21.17364  23.0577

Totals :                  1382.66165   91.17817
```

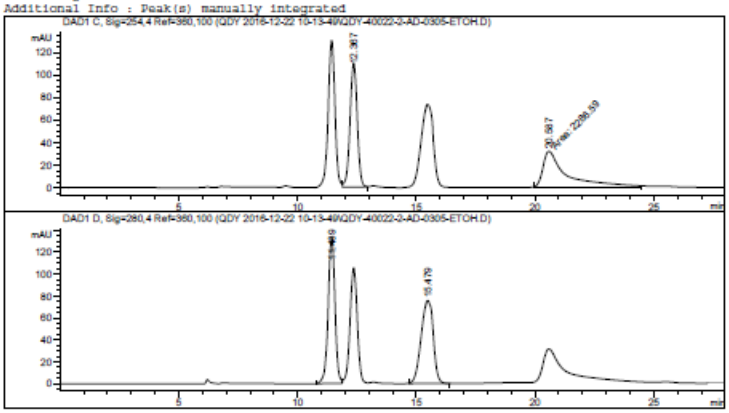
```
                        *** End of Report ***
```

Supplementary Figure 173. HPLC spectrum for 3c



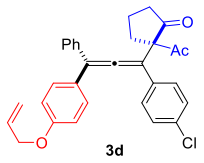
(rac)-**3d**

```
Acq. Operator   :                                   Seq. Line :   2
Acq. Instrument : Instrument 1                       Location : Vial 33
Injection Date  : 12/22/2016 10:42:58 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-22 10-13-49\ETOH-AD-03-25-0.5.M
Last changed    : 12/22/2016 11:03:45 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-03-25-07.M
Last changed    : 12/21/2016 7:43:26 PM
Additional Info : Peak(s) manually integrated
```



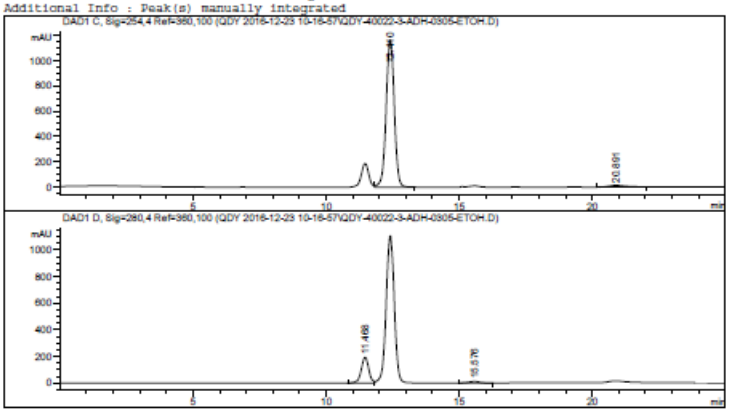

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]      [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  12.367 VB    0.3293 2336.37280  110.21536  50.5384
   2  20.587 MM    1.1878 2286.59106   32.08462  49.4616

Totals :                  4622.96387  142.29998
```

```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]      [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  11.439 BV    0.3084 2670.96265  134.09711  49.8320
   2  15.479 BB    0.5162 2688.97021   75.73504  50.1680

Totals :                  5359.93286  209.83215

                        *** End of Report ***
```



3d

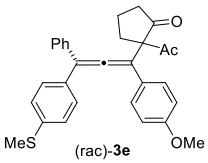
```
Acq. Operator   :                                   Seq. Line :   7
Acq. Instrument : Instrument 1                       Location : Vial 33
Injection Date  : 12/23/2016 12:06:26 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-23 10-16-57\ETOH-AD-03-25-0.5.M
Last changed    : 12/21/2016 2:33:56 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-03-25-07.M
Last changed    : 12/23/2016 1:47:07 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]      [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  12.410 VB    0.3327 2.48662e4  1166.35120  97.6844
   2  20.891 BB    0.6552  589.45648   13.50646   2.3156

Totals :                  2.54557e4  1179.85765
```

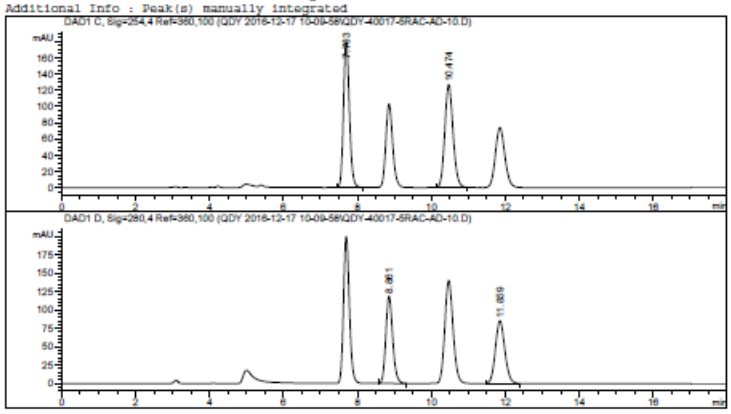
```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]      [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  11.468 BV    0.3041 3771.59863  192.94066  92.3225
   2  15.576 BB    0.3789  313.64468   11.61888   7.6775

Totals :                  4085.24332  204.55954

                        *** End of Report ***
```

Supplementary Figure 174. HPLC spectrum for 3d



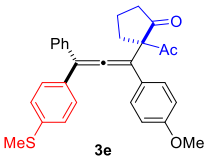
(rac)-**3e**

```
Acq. Operator   :                                   Seq. Line :   3
Acq. Instrument : Instrument 1                       Location : Vial 35
Injection Date  : 12/17/2016 11:07:14 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-17 10-09-58\AD-10-25.M
Last changed    : 12/17/2016 11:22:08 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/17/2016 10:28:54 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



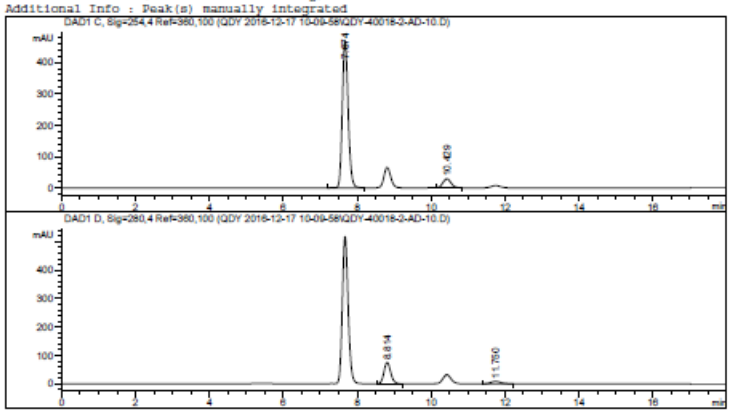

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.703 BB    0.1708 1999.58411  181.13736  50.3226
   2  10.474 BB    0.2418 1973.94446  126.68971  49.6774

Totals :                  3973.52856  307.82707
```

```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.861 BB    0.1986 1530.18848  118.56132  50.2966
   2  11.859 BB    0.2754 1512.13953   85.07885  49.7034

Totals :                  3042.32800  203.64017

                        *** End of Report ***
```



3e

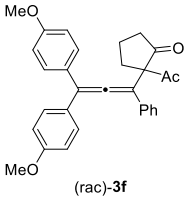
```
Acq. Operator   :                                   Seq. Line :  19
Acq. Instrument : Instrument 1                       Location : Vial 32
Injection Date  : 12/17/2016 6:34:55 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-17 10-09-58\AD-10-25.M
Last changed    : 12/17/2016 6:33:07 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/17/2016 10:28:54 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.674 BB    0.1701 5147.08936  468.59857  91.9216
   2  10.429 BB    0.2391  452.34695   29.13834   8.0784

Totals :                  5599.43631  497.73691
```

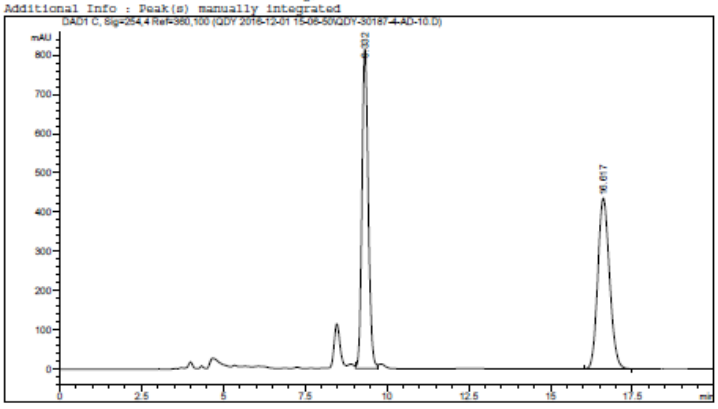
```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.814 BB    0.1962  954.48505   75.15132  85.9938
   2  11.750 BB    0.2766  155.46130    8.61222  14.0062

Totals :                  1109.94635   83.76354

                        *** End of Report ***
```

Supplementary Figure 175. HPLC spectrum for 3e

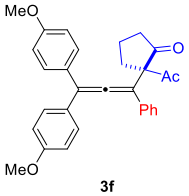


(rac)-**3f**

```
Acq. Operator   :                                   Seq. Line :  22
Acq. Instrument : Instrument 1                      Location : Vial 34
Injection Date  : 12/1/2016 11:43:21 PM                Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-01 15-06-50\AD-10-30.M
Last changed    : 12/1/2016 10:27:40 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 1/1/2017 3:00:17 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

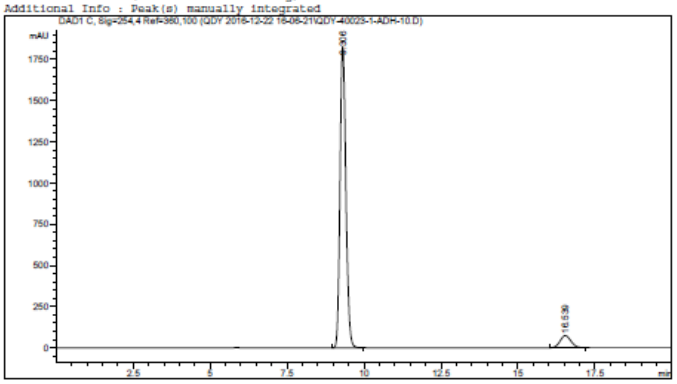



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.332 VV    0.2081 1.09001e4   814.87524  50.0876
   2  16.617 BB    0.3873 1.08620e4   435.09927  49.9124

Totals :                  2.17621e4  1249.97452

                          *** End of Report ***
```



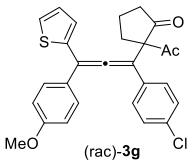
3f

```
Acq. Operator   :                                   Seq. Line :  24
Acq. Instrument : Instrument 1                      Location : Vial 31
Injection Date  : 12/22/2016 11:57:21 PM               Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-22 16-06-21\AD-10-25.M
Last changed    : 9/12/2015 7:30:37 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-03-25-07.M
Last changed    : 12/22/2016 7:51:24 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



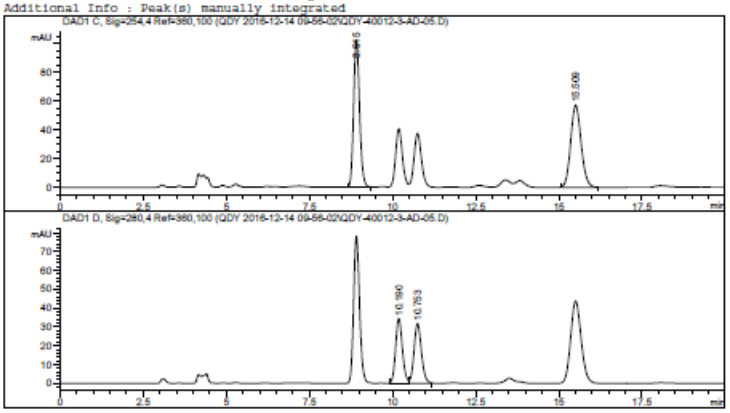

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.306 BB    0.2050 2.42926e4  1828.79016  92.9066
   2  16.539 BB    0.3852 1854.72717   74.82449   7.0934

Totals :                  2.61473e4  1903.61465

                          *** End of Report ***
```

Supplementary Figure 176. HPLC spectrum for 3f



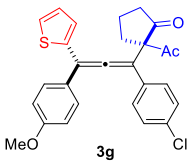
(rac)-**3g**

```
Acq. Operator   :                                  Seq. Line :  19
Acq. Instrument : Instrument 1                     Location : Vial 33
Injection Date  : 12/14/2016 5:19:42 PM               Inj :  1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-14 09-56-02\AD-05-20.M
Last changed    : 12/14/2016 5:18:48 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/18/2016 9:24:07 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



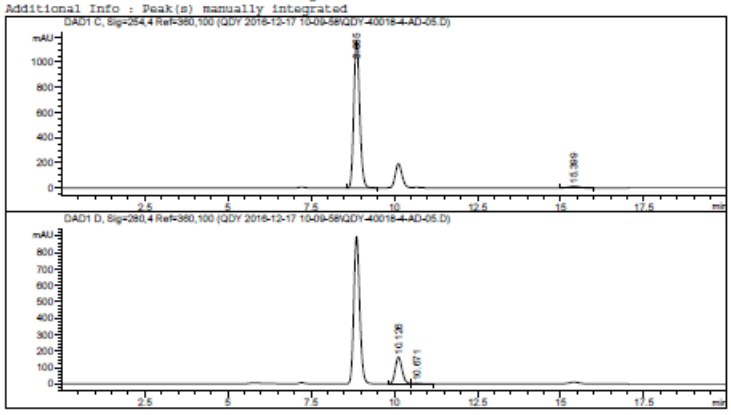

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.915 BB    0.1908 1271.38049  102.41248  50.2509
   2  15.509 BB    0.3401 1258.68274   57.34032  49.7491

Totals :                  2530.06323  159.75280
```

```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  10.190 BV    0.2195  487.97296   34.39703  49.8834
   2  10.753 VB    0.2379  490.25323   31.79630  50.1166

Totals :                   978.22620   66.19333

                        *** End of Report ***
```



3g

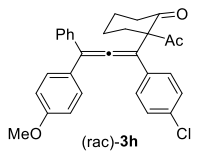
```
Acq. Operator   :                                  Seq. Line :  41
Acq. Instrument : Instrument 1                     Location : Vial 34
Injection Date  : 12/18/2016 2:53:23 AM               Inj :  1
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-17 10-09-58\AD-05-20.M
Last changed    : 12/18/2016 2:30:04 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-50-30.M
Last changed    : 12/18/2016 9:40:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.865 BB    0.1898 1.44274e4  1170.38037  97.9690
   2  15.399 BB    0.3363  299.08856   13.72236   2.0310

Totals :                  1.47265e4  1184.10273
```

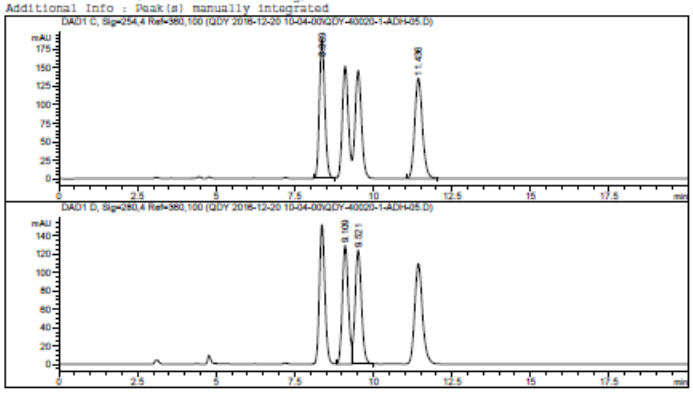
```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  10.126 BV    0.2185 2298.65723  163.05170  97.5052
   2  10.671 VB    0.2264   58.81377    3.98117   2.4948

Totals :                  2357.47100  167.03287

                        *** End of Report ***
```

Supplementary Figure 177. HPLC spectrum for 3g



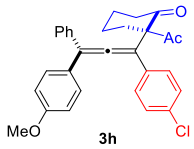
(rac)-**3h**

```
Acq. Operator   :                                   Seq. Line :   9
Acq. Instrument : Instrument 1                      Location : Vial 31
Injection Date  : 12/20/2016 1:59:46 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-20 10-04-00\AD-05-20.M
Last changed    : 12/20/2016 1:58:54 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/26/2016 1:11:51 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



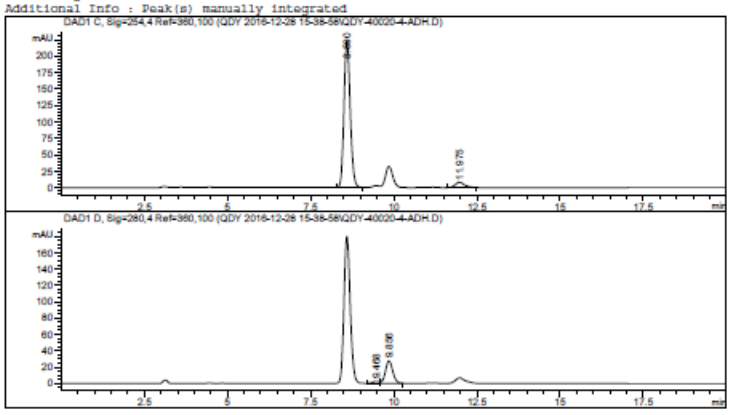

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.369 BB    0.1877 2288.36597  188.41090  49.7870
   2  11.436 BB    0.2625 2307.94678  135.61908  50.2130

Totals :                  4596.31274  324.02998
```

```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   9.109 BV    0.2034 1703.65442  129.56859  49.2211
   2   9.521 VB    0.2189 1757.57336  124.31697  50.7789

Totals :                  3461.22778  253.88556

                        *** End of Report ***
```



3h

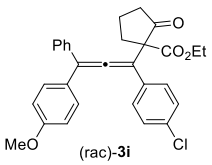
```
Acq. Operator   :                                   Seq. Line :   2
Acq. Instrument : Instrument 1                      Location : Vial 34
Injection Date  : 12/28/2016 3:53:13 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 6.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-28 15-38-58\AD-05-20.M
Last changed    : 12/28/2016 3:52:19 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/25/2016 12:31:15 AM
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.590 BB    0.1994 2898.87183  223.33513  94.8538
   2  11.975 BB    0.2967  157.27544    8.02446   5.1462

Totals :                  3056.14726  231.35959
```

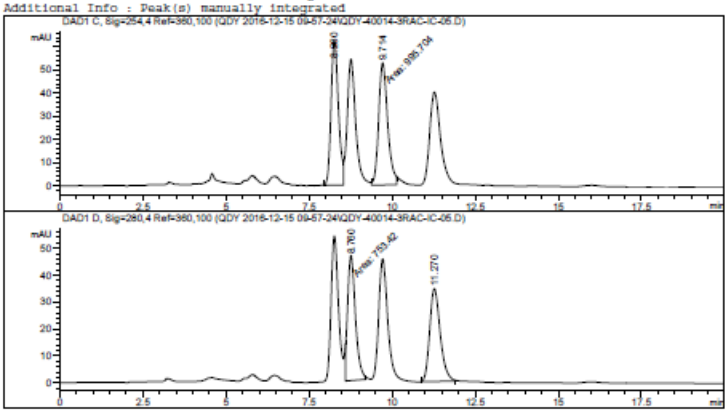
```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   9.468 BV    0.1947   30.80071    2.44990   6.8343
   2   9.856 VB    0.2345  419.87592   27.75845  93.1657

Totals :                   450.67662   30.20835

                        *** End of Report ***
```

Supplementary Figure 178. HPLC spectrum for 3h



(rac)-**3i**

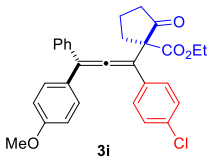
```
Acq. Operator   :                                   Seq. Line :  41
Acq. Instrument : Instrument 1                      Location : Vial 34
Injection Date  : 12/16/2016 1:40:39 AM                 Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-15 09-57-24\IC-05-30.M
Last changed    : 8/23/2012 1:59:23 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-03-25-07.M
Last changed    : 12/23/2016 11:10:32 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.260 BV    0.2355  966.61340   62.83598  49.2588
   2   9.714 MF    0.3144  995.70392   52.78615  50.7412

Totals :                   1962.31732  115.62213
```

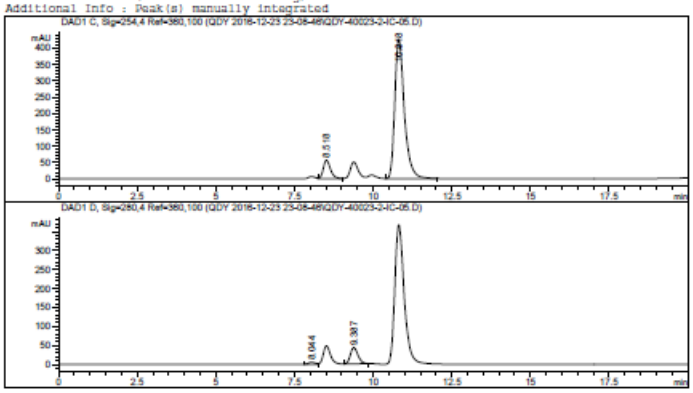
```
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.760 FM    0.2696  753.42029   46.57753  49.8034
   2  11.270 BB    0.3366  759.36938   34.52636  50.1966

Totals :                   1512.78967   81.10389
```

```
                        *** End of Report ***
```



3i

```
Acq. Operator   :                                   Seq. Line :  6
Acq. Instrument : Instrument 1                      Location : Vial 31
Injection Date  : 12/24/2016 12:20:45 AM                Inj :  1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-23 23-08-46\IC-05-30.M
Last changed    : 12/24/2016 12:19:53 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-03-25-07.M
Last changed    : 12/24/2016 10:24:47 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

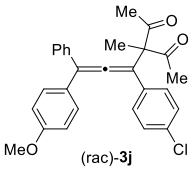



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.518 VB    0.2444  920.95679   57.03419   9.2614
   2  10.818 BB    0.3211 9023.05469  426.03329  90.7386

Totals :                   9944.01147  483.06748
```

```
Signal 2: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.044 BV    0.2093   74.39173    5.51589   9.3557
   2   9.387 BB    0.2595  720.75873   43.01805  90.6443

Totals :                   795.15046   48.53394
```

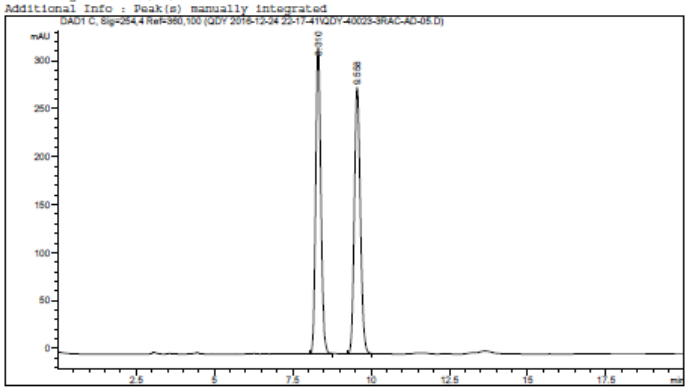
```
                        *** End of Report ***
```

Supplementary Figure 179. HPLC spectrum for 3i

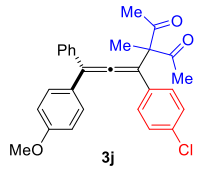


(rac)-**3j**

```
Acq. Operator   :                                   Seq. Line :   6
Acq. Instrument : Instrument 1                      Location : Vial 33
Injection Date  : 12/24/2016 11:58:45 PM               Inj :   1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-24 22-17-41\AD-05-20.M
Last changed    : 12/24/2016 11:57:53 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/25/2016 12:31:15 AM
Additional Info : Peak(s) manually integrated
```

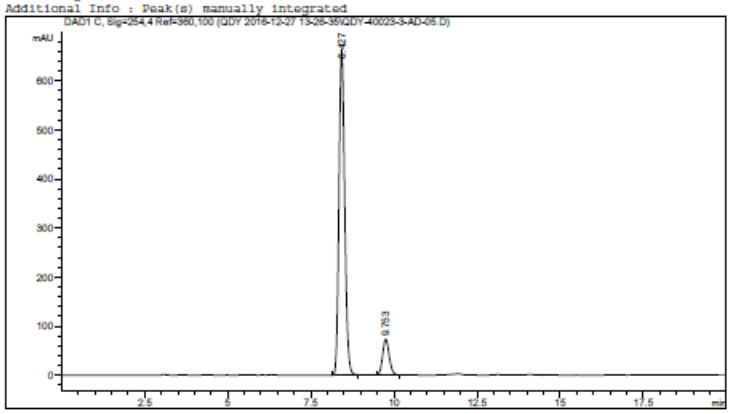



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.310 BB    0.1792 3708.74731  320.02621  49.9828
   2   9.558 BB    0.2079 3711.30029  277.69928  50.0172

Totals :                  7420.04761  597.72549

                       *** End of Report ***
```



3j

```
Acq. Operator   :                                   Seq. Line :   7
Acq. Instrument : Instrument 1                      Location : Vial 33
Injection Date  : 12/27/2016 4:04:03 PM                Inj :   1
                                                    Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-12-27 13-26-35\AD-05-20.M
Last changed    : 12/27/2016 4:03:11 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 12/25/2016 12:31:15 AM
Additional Info : Peak(s) manually integrated
```

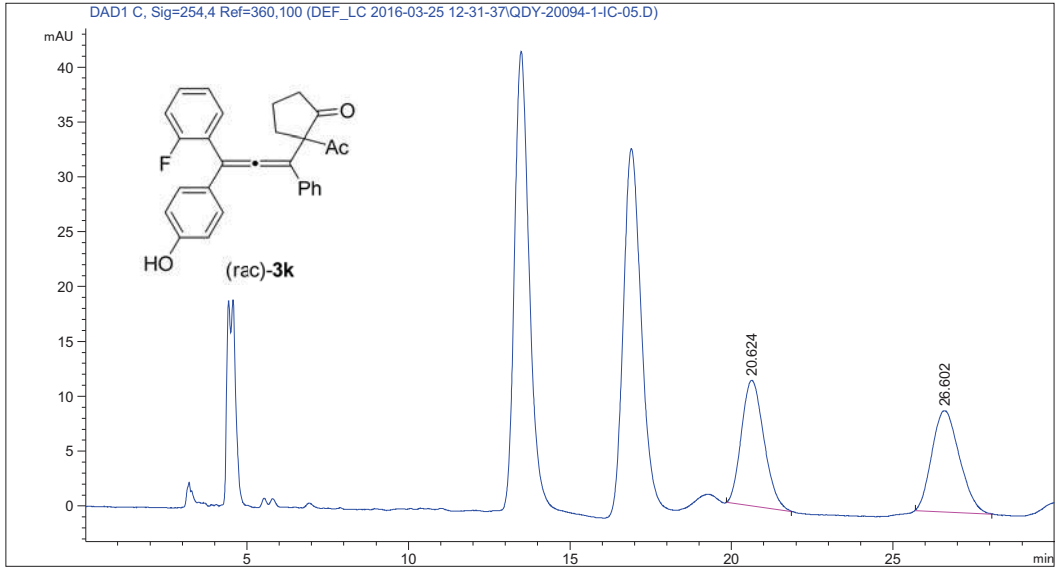



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.427 BB    0.1858 7953.15088  663.68365  88.7915
   2   9.753 BB    0.2127 1003.95966   72.87911  11.2085

Totals :                  8957.11053  736.56277

                       *** End of Report ***
```

Supplementary Figure 180. HPLC spectrum for 3j

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-25 12-31-37\QDY-20094-1-IC-05.D
Sample Name:

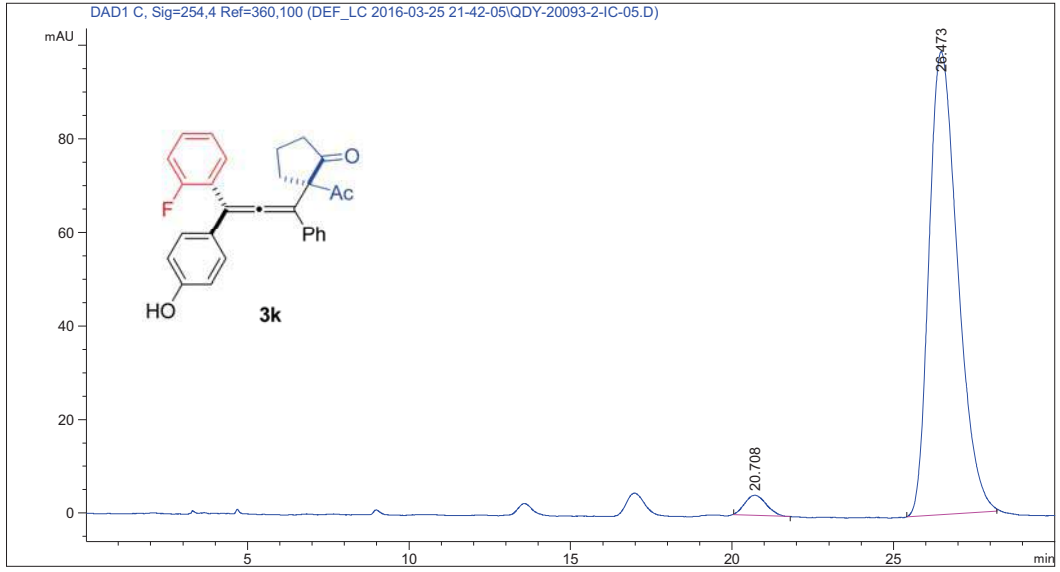
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   8
Acq. Instrument : Instrument 1                    Location : Vial 36
Injection Date  : 3/25/2016 2:13:03 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-25 12-31-37\IC-05-30.M
Last changed    : 3/25/2016 2:12:13 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:34:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.624 BB    0.6286  556.81903   11.46449  50.2834
   2  26.602 BB    0.7284  550.54340    9.25036  49.7166

Totals :                  1107.36243   20.71485
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-25 21-42-05\QDY-20093-2-IC-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 3/25/2016 9:55:53 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-25 21-42-05\IC-05-30.M
Last changed    : 3/25/2016 9:55:02 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:34:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

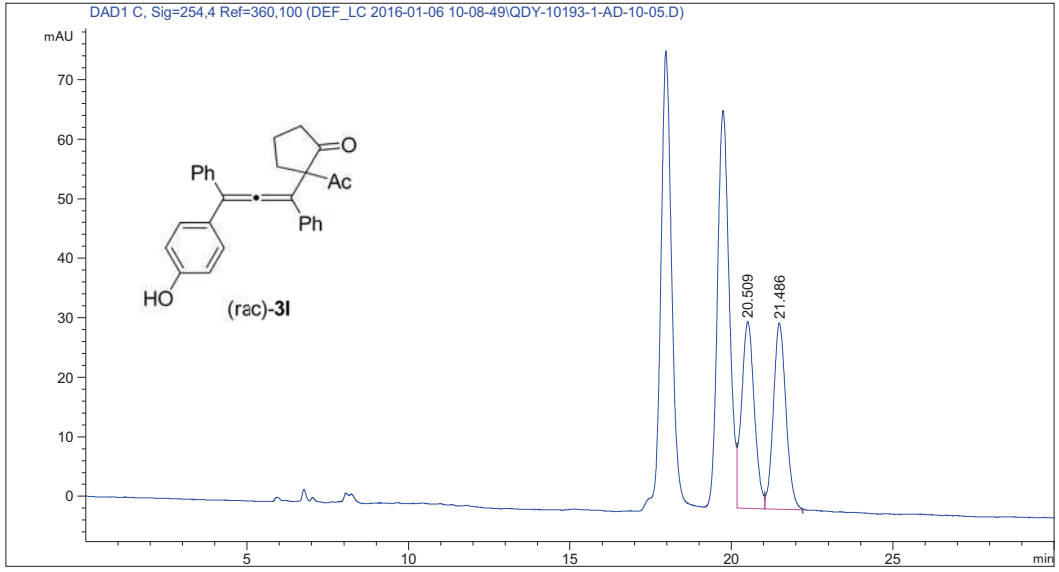



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.708 BB    0.5561  198.82310    4.29505   3.1520
   2  26.473 BB    0.9478 6108.96680   99.00853  96.8480

Totals :                  6307.78990  103.30358
```

Supplementary Figure 181. HPLC spectrum for 3k

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-06 10-08-49\QDY-10193-1-AD-10-05.D
Sample Name:

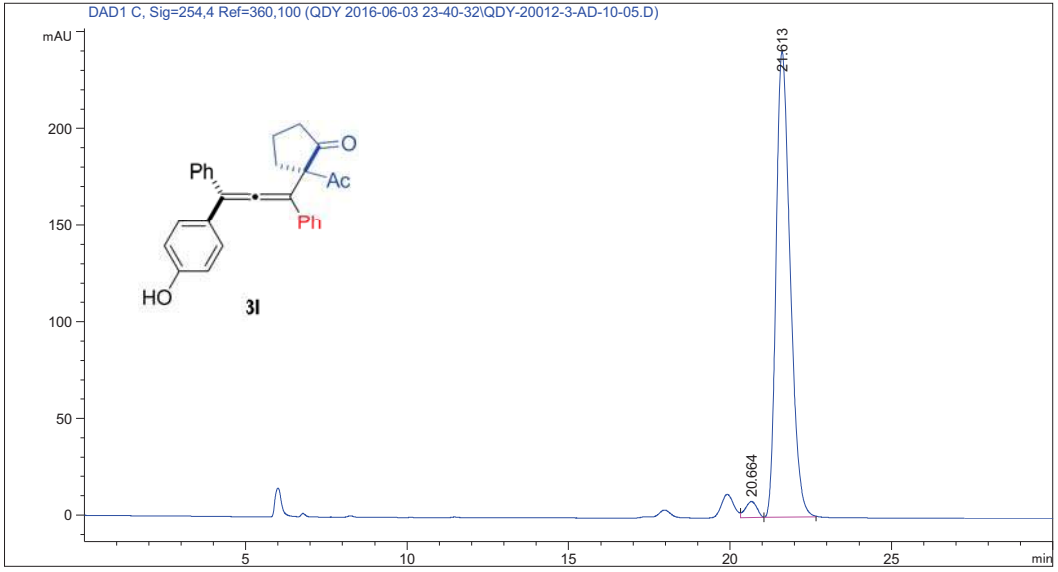
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  11
Acq. Instrument : Instrument 1                    Location : Vial 34
Injection Date  : 1/6/2016 1:13:34 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-06 10-08-49\AD-10-30-0.5.M
Last changed    : 1/6/2016 1:12:39 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:08:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.509 VV    0.4218  864.40710   31.34101  50.3231
   2  21.486 VB    0.4203  853.30853   31.28021  49.6769

Totals :                  1717.71564   62.62122
```


Data File C:\CHEM32\1\DATA\QDY 2016-06-03 23-40-32\QDY-20012-3-AD-10-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 6/3/2016 11:54:26 PM                Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-06-03 23-40-32\AD-10-30-0.5.M
Last changed    : 6/3/2016 11:53:33 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:08:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

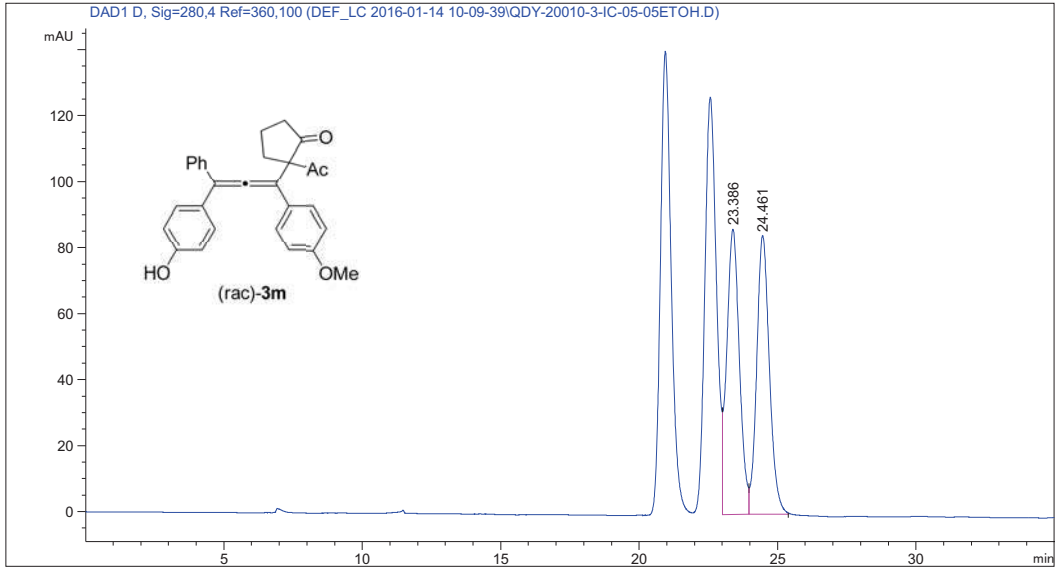



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.664 VV    0.3936  206.00653    8.29917   2.7387
   2  21.613 VB    0.4703 7315.97168  240.87839  97.2613

Totals :                  7521.97821  249.17756
```

Supplementary Figure 182. HPLC spectrum for 3l

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-14 10-09-39\QDY-20010-3-IC-05-05ETOH.D
Sample Name:

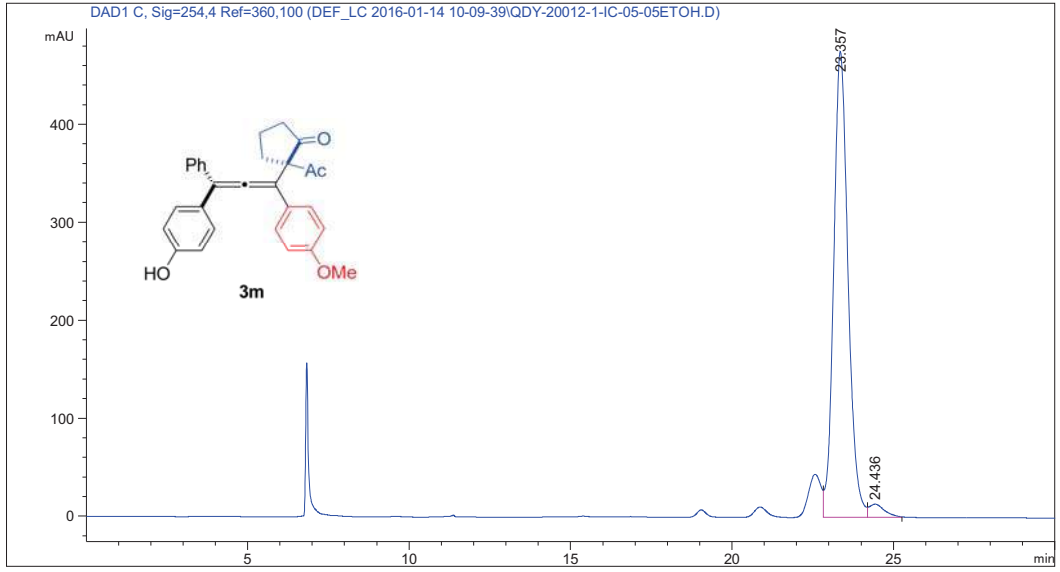
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  14
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 1/14/2016 2:42:09 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-14 10-09-39\ETOH-IC-05-30-05.M
Last changed    : 1/14/2016 3:13:23 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:08:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.386 VV    0.4855 2753.98926   86.47826  50.5775
   2  24.461 VB    0.4898 2691.10376   84.43309  49.4225

Totals :                  5445.09302  170.91135
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-14 10-09-39\QDY-20012-1-IC-05-05ETOH.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  15
Acq. Instrument : Instrument 1                     Location : Vial 35
Injection Date  : 1/14/2016 3:18:15 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-14 10-09-39\ETOH-IC-05-30-05.M
Last changed    : 1/14/2016 3:45:28 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:08:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

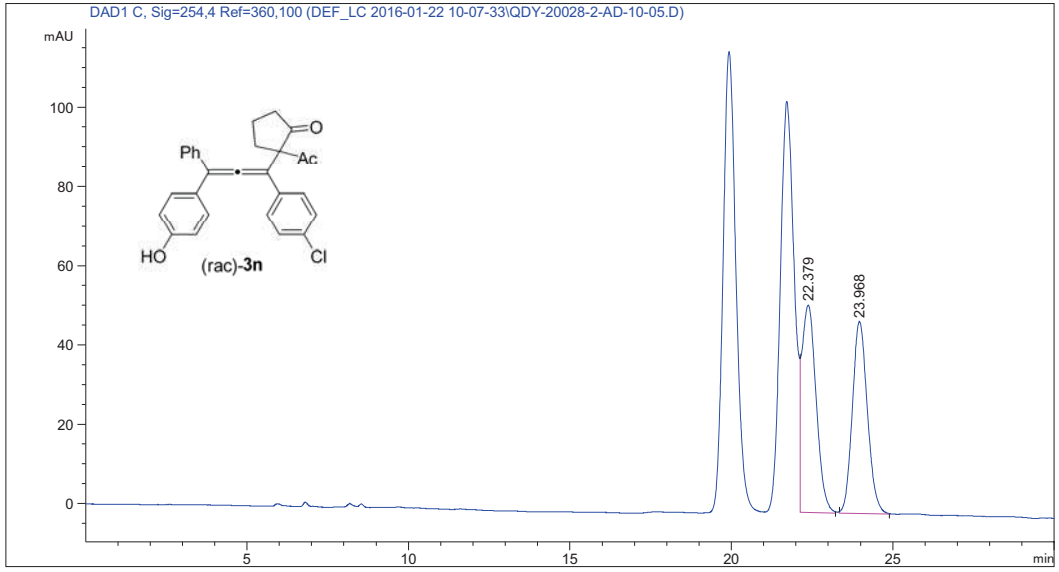



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.357 VV    0.4742 1.46803e4   475.52399  97.0404
   2  24.436 VB    0.4846  447.73767   13.37484   2.9596

Totals :                  1.51281e4   488.89882
```

Supplementary Figure 183. HPLC spectrum for 3m

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-22 10-07-33\QDY-20028-2-AD-10-05.D
Sample Name:

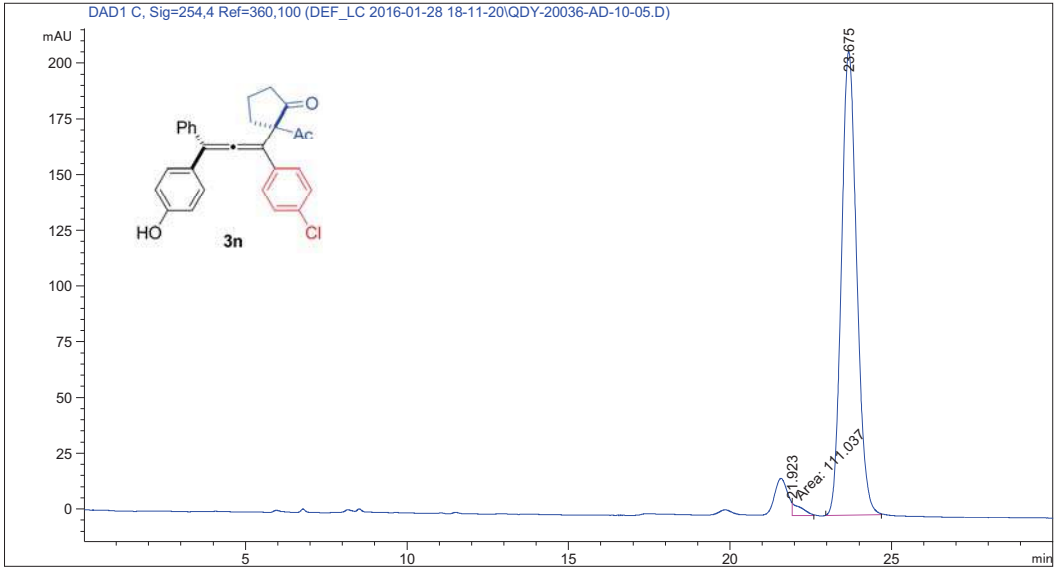
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  31
Acq. Instrument : Instrument 1                    Location : Vial 33
Injection Date  : 1/22/2016 7:46:01 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-22 10-07-33\AD-10-30-0.5.M
Last changed    : 1/7/2016 11:07:32 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:13:09 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  22.379 VB    0.4605 1617.20544   52.33952  50.8224
   2  23.968 BB    0.5012 1564.86450   48.39814  49.1776

Totals :                  3182.06995  100.73766


==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-28 18-11-20\QDY-20036-AD-10-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 1/28/2016 6:22:19 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-28 18-11-20\AD-10-30-0.5.M
Last changed    : 1/28/2016 6:22:31 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:13:09 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

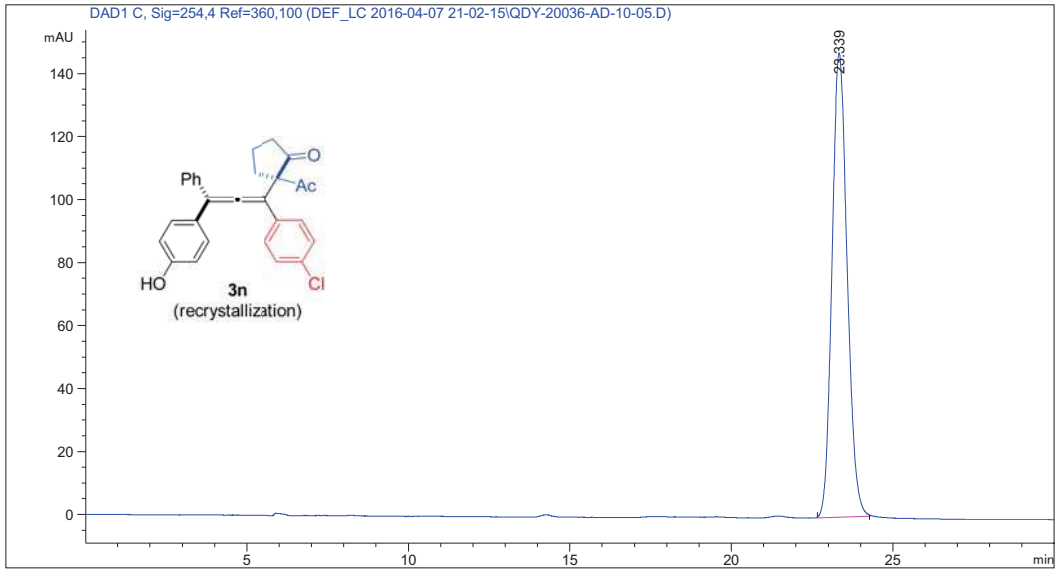



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  21.923 FM    0.3323  111.03651    5.56921   1.5975
   2  23.675 BB    0.5077 6839.57959  207.98744  98.4025

Totals :                  6950.61610  213.55665
```

Supplementary Figure 184. HPLC spectrum for 3n

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-07 21-02-15\QDY-20036-AD-10-05.D
Sample Name:

```
==================================================================
Acq. Operator   :                                Seq. Line :  29
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 4/8/2016 9:11:23 AM                  Inj :   1
                                                 Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-07 21-02-15\AD-10-30-0.5.M
Last changed    : 1/7/2016 11:07:32 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:13:09 PM
                  (modified after loading)
```

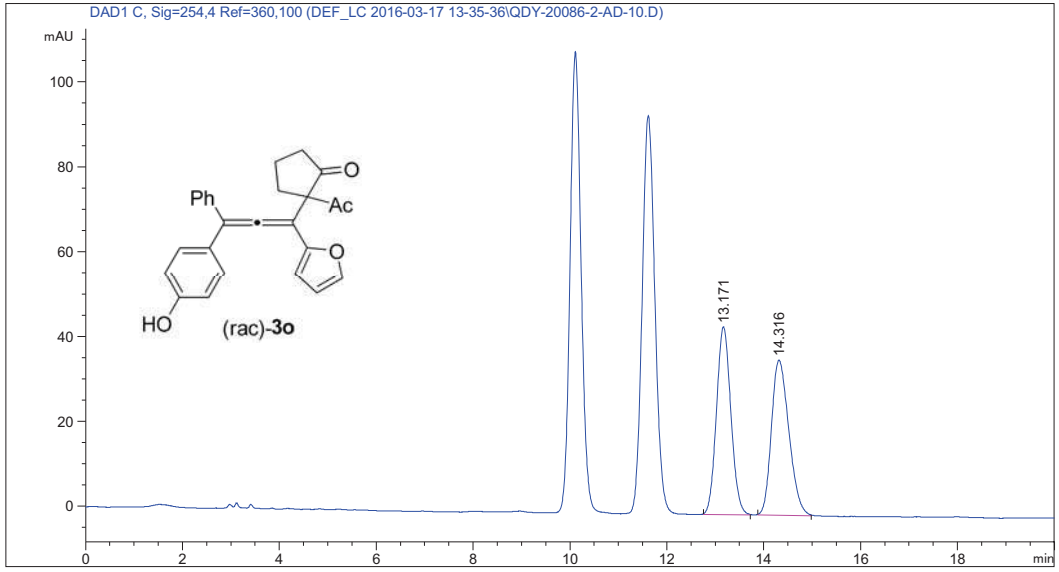



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.339 BB    0.5087 4807.56592  147.34752 100.0000

Totals :                  4807.56592  147.34752

==================================================================
                        *** End of Report ***
```

Supplementary Figure 185. HPLC spectrum for 3n'

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\QDY-20086-2-AD-10.D
Sample Name:

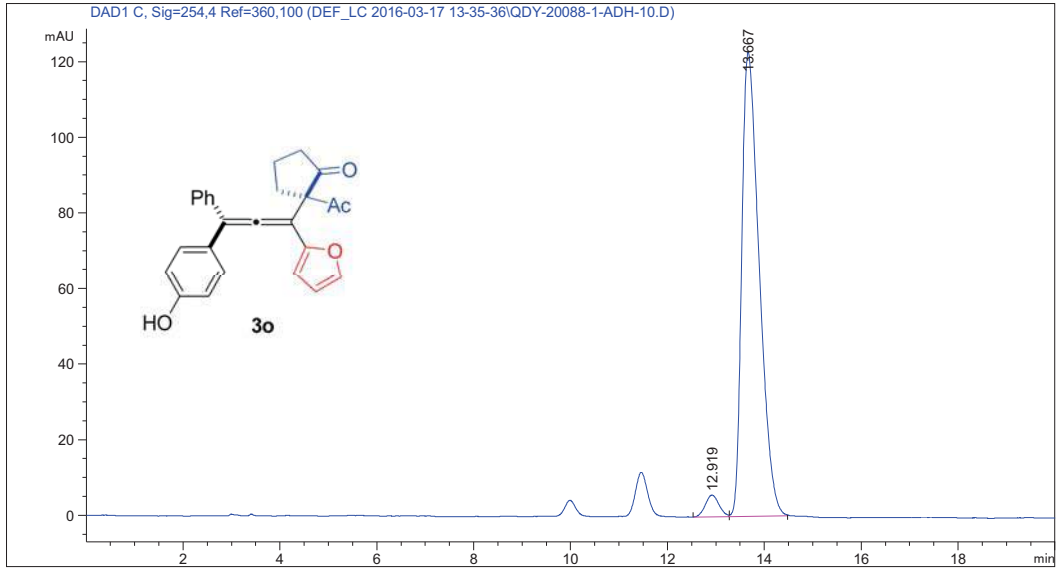
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  13
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 3/17/2016 5:12:23 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\AD-10-30.M
Last changed    : 11/30/2015 12:57:24 PM
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:26:25 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.171 BB    0.3233  915.31964   44.26314  50.0637
   2  14.316 BB    0.3834  912.99078   36.54956  49.9363

Totals :                  1828.31042   80.81270

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\QDY-20088-1-ADH-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  70
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 3/18/2016 4:46:31 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\AD-10-20.M
Last changed    : 11/28/2014 10:15:17 AM
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:08:47 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

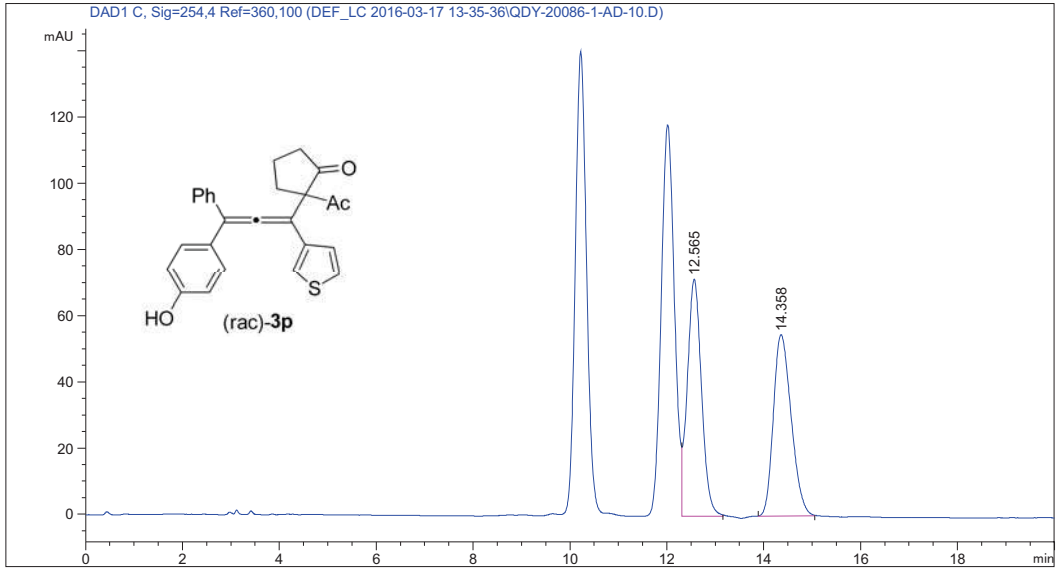



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.919 BV    0.3037  111.90915    5.73329   3.5141
   2  13.667 VB    0.3818 3072.70459  122.87263  96.4859

Totals :                  3184.61374  128.60593

==========================================================================
```

Supplementary Figure 186. HPLC spectrum for 3o

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\QDY-20086-1-AD-10.D
Sample Name:

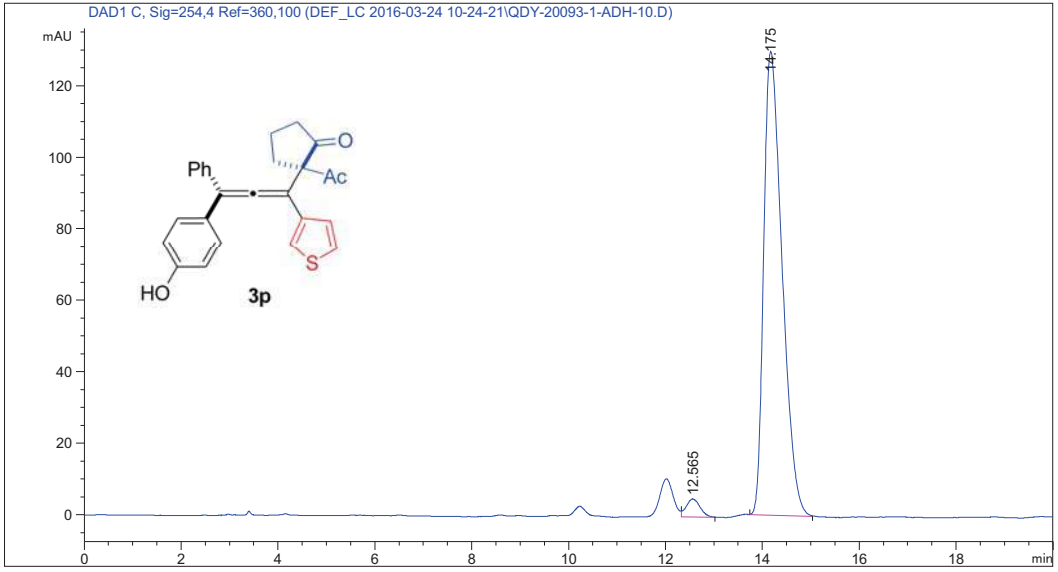
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  16
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 3/17/2016 6:19:13 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\AD-10-30.M
Last changed    : 11/30/2015 12:57:24 PM
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:26:25 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.565 VB    0.3114 1455.71436   71.54522  50.8861
   2  14.358 BB    0.4009 1405.01794   54.84630  49.1139

Totals :                  2860.73230  126.39152

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-24 10-24-21\QDY-20093-1-ADH-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   9
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 3/24/2016 12:34:12 PM                Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-24 10-24-21\AD-10-30.M
Last changed    : 3/24/2016 12:40:12 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-15-30.M
Last changed    : 8/20/2016 4:26:25 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

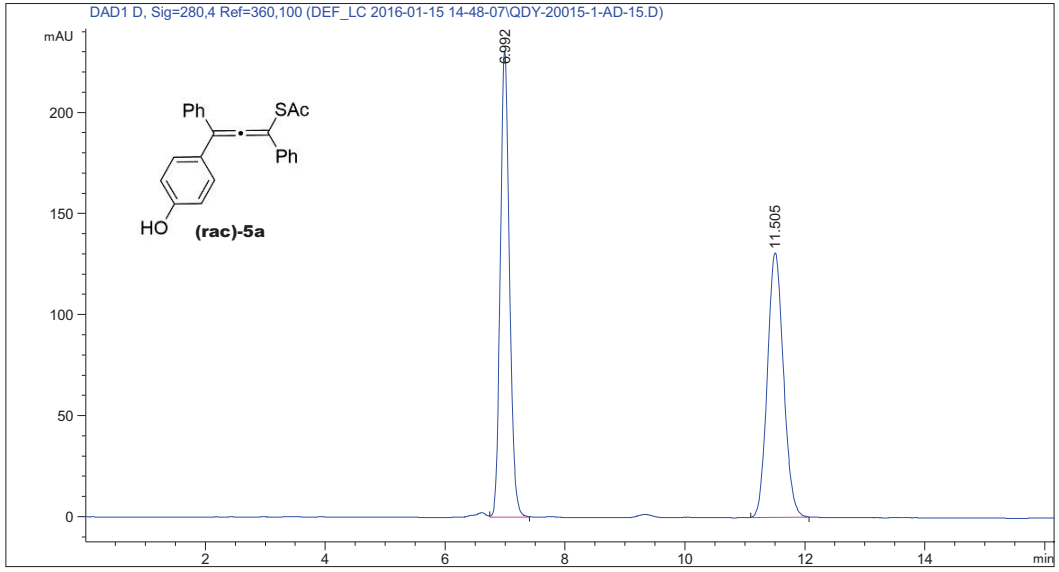



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.565 VB    0.3027  102.38339    5.08985   2.9131
   2  14.175 BB    0.4070 3412.21973  129.67111  97.0869

Totals :                  3514.60312  134.76096
```

Supplementary Figure 187. HPLC spectrum for 3p

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-15 14-48-07\QDY-20015-1-AD-15.D
Sample Name:

Acq. Operator : Seq. Line : 14
Acq. Instrument : Instrument 1 Location : Vial 31
Injection Date : 1/15/2016 7:04:56 PM Inj : 1
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-01-15 14-48-07\AD-15-30.M
Last changed : 1/15/2016 7:21:04 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed : 6/25/2016 10:45:20 PM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

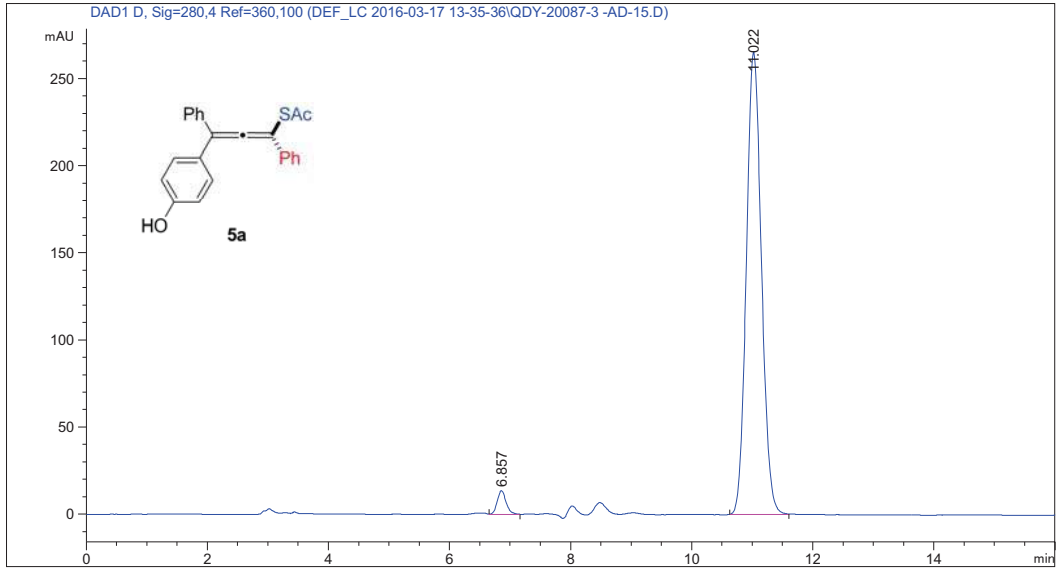
Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.992	VB	0.1642	2452.11304	230.39255	50.0793
2	11.505	BB	0.2897	2444.35181	130.97171	49.9207

Totals : 4896.46484 361.36426

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\QDY-20087-3 -AD-15.D
Sample Name:

Acq. Operator : Seq. Line : 72
Acq. Instrument : Instrument 1 Location : Vial 39
Injection Date : 3/18/2016 5:15:48 PM Inj : 1
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-03-17 13-35-36\AD-15-22.M
Last changed : 9/15/2015 8:34:07 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-3030.M
Last changed : 6/15/2016 2:43:08 PM
(modified after loading)
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=280,4 Ref=360,100

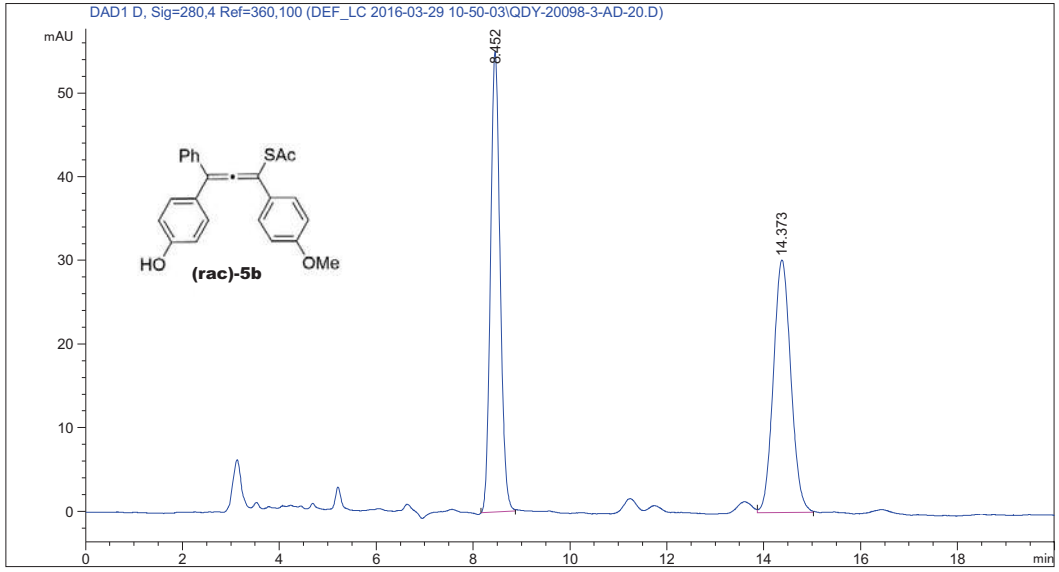
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.857	VB	0.1572	138.61012	13.56842	2.9376
2	11.022	BB	0.2691	4579.83252	265.64444	97.0624

Totals : 4718.44264 279.21286

Supplementary Figure 188. HPLC spectrum for 5a

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-29 10-50-03\QDY-20098-3-AD-20.D
Sample Name:

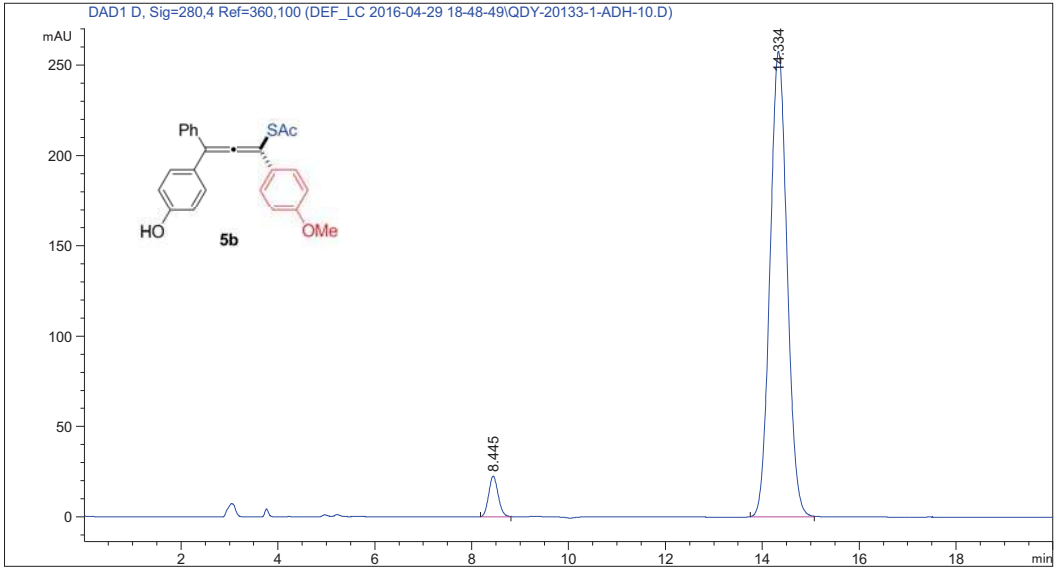
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  17
Acq. Instrument : Instrument 1                     Location : Vial 33
Injection Date  : 3/29/2016 4:38:58 PM                  Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-29 10-50-03\AD-20-30.M
Last changed    : 9/14/2015 3:31:48 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:16:02 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.452 BB    0.2145  756.95715   55.01791  50.0821
   2  14.373 VB    0.3875  754.47668   30.19891  49.9179

Totals :                  1511.43384   85.21682

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\QDY-20133-1-ADH-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  12
Acq. Instrument : Instrument 1                     Location : Vial 35
Injection Date  : 4/29/2016 10:34:54 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\AD-20-20.M
Last changed    : 4/29/2016 10:34:02 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:13:23 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

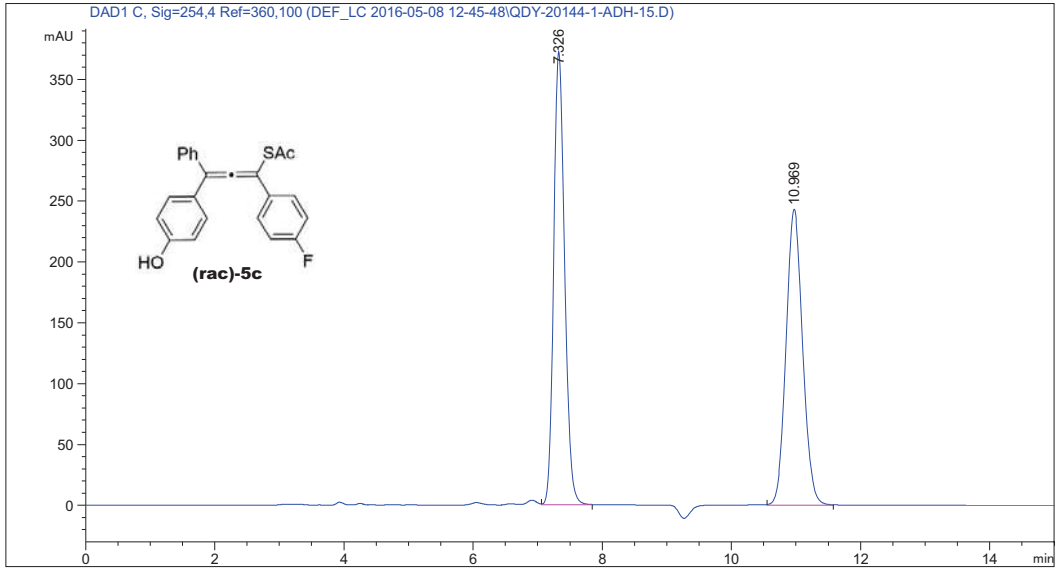



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.445 BB    0.2113  307.34174   22.50527   4.6406
   2  14.334 BB    0.3800 6315.47900  257.61475  95.3594

Totals :                  6622.82074  280.12002
```

Supplementary Figure 189. HPLC spectrum for 5b

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-08 12-45-48\QDY-20144-1-ADH-15.D
Sample Name:

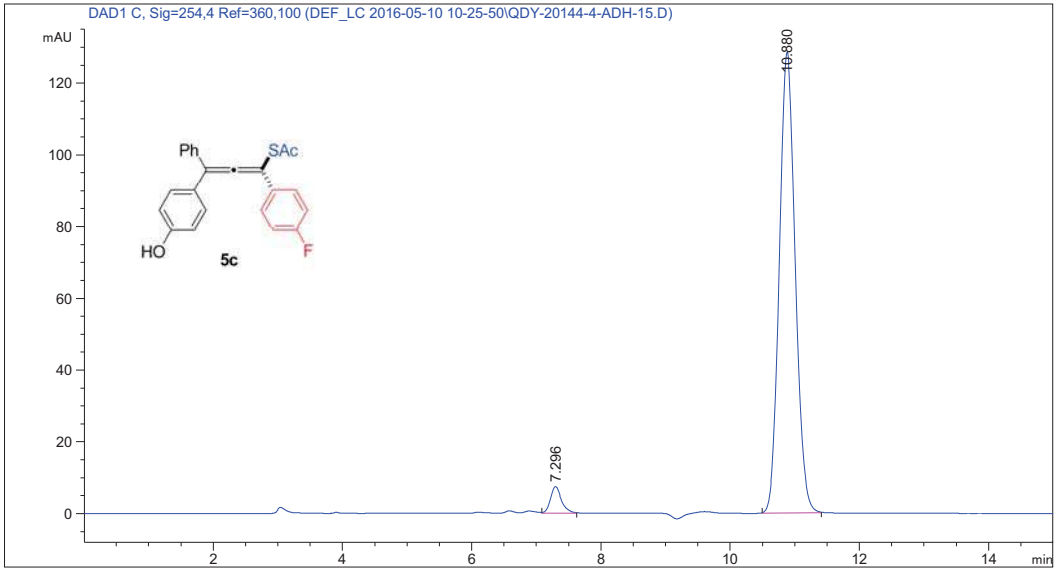
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  33
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 5/9/2016 12:25:00 AM                  Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-05-08 12-45-48\AD-15-22.M
Last changed    : 9/15/2015 8:34:07 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:57:24 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.326 VB    0.1828 4366.85938  372.40533  50.2199
   2  10.969 BB    0.2757 4328.62256  243.13440  49.7801

Totals :                  8695.48193  615.53973

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-10 10-25-50\QDY-20144-4-ADH-15.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  21
Acq. Instrument : Instrument 1                     Location : Vial 33
Injection Date  : 5/10/2016 6:18:08 PM                  Inj :  1
                                                  Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-05-10 10-25-50\AD-15-22.M
Last changed    : 5/10/2016 6:24:54 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:55:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

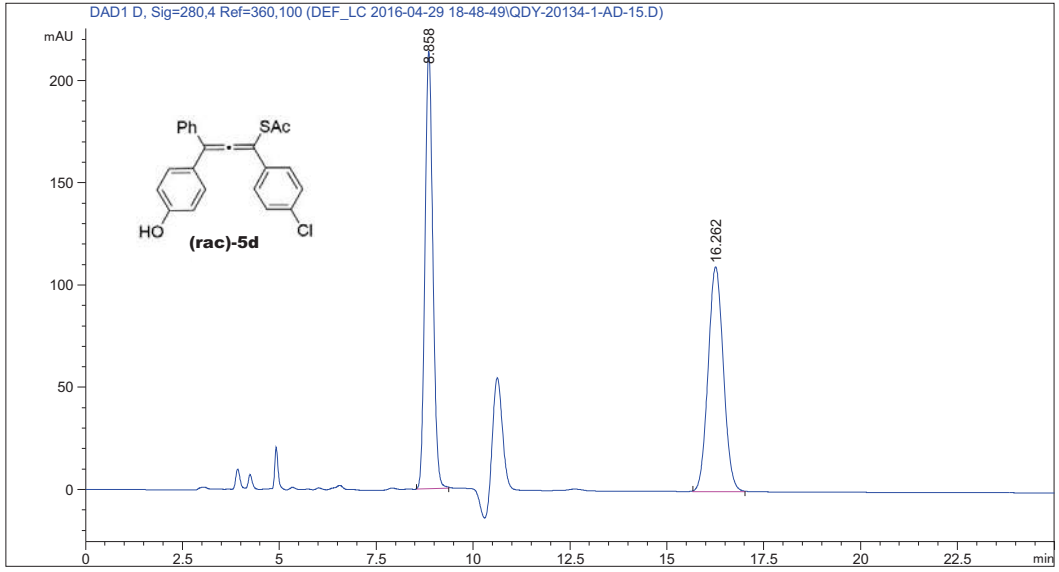



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.296 BB    0.1765   87.03292   7.43725   3.8065
   2  10.880 BB    0.2657 2199.37378  128.49953  96.1935

Totals :                  2286.40670  135.93677
```


Supplementary Figure 190. HPLC spectrum for 5c

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\QDY-20134-1-AD-15.D
Sample Name:

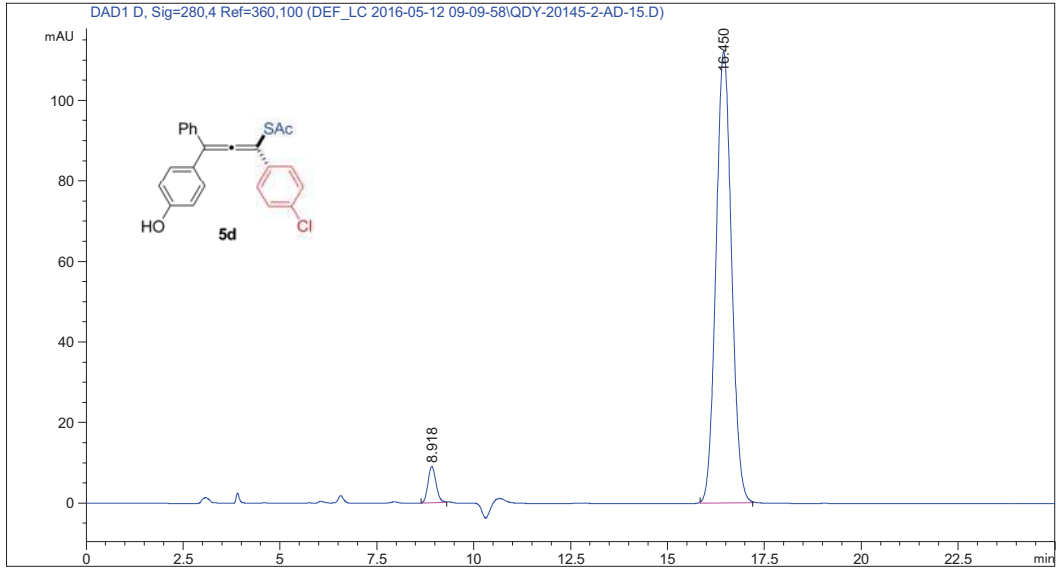
```
==================================================================
Acq. Operator   :                          Seq. Line :  18
Acq. Instrument : Instrument 1              Location : Vial 31
Injection Date  : 4/30/2016 12:42:59 AM          Inj :  1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\AD-15-30.M
Last changed    : 4/30/2016 1:07:01 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:00:17 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.858 BB    0.2209 3023.58618  213.92877  50.0682
   2  16.262 BB    0.4280 3015.34619  109.93711  49.9318

Totals :                  6038.93237  323.86588
```

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-12 09-09-58\QDY-20145-2-AD-15.D
Sample Name:

```
==================================================================
Acq. Operator   :                          Seq. Line :  10
Acq. Instrument : Instrument 1              Location : Vial 31
Injection Date  : 5/12/2016 11:40:46 AM          Inj :  1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-05-12 09-09-58\AD-15-30.M
Last changed    : 5/12/2016 11:39:53 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:00:17 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

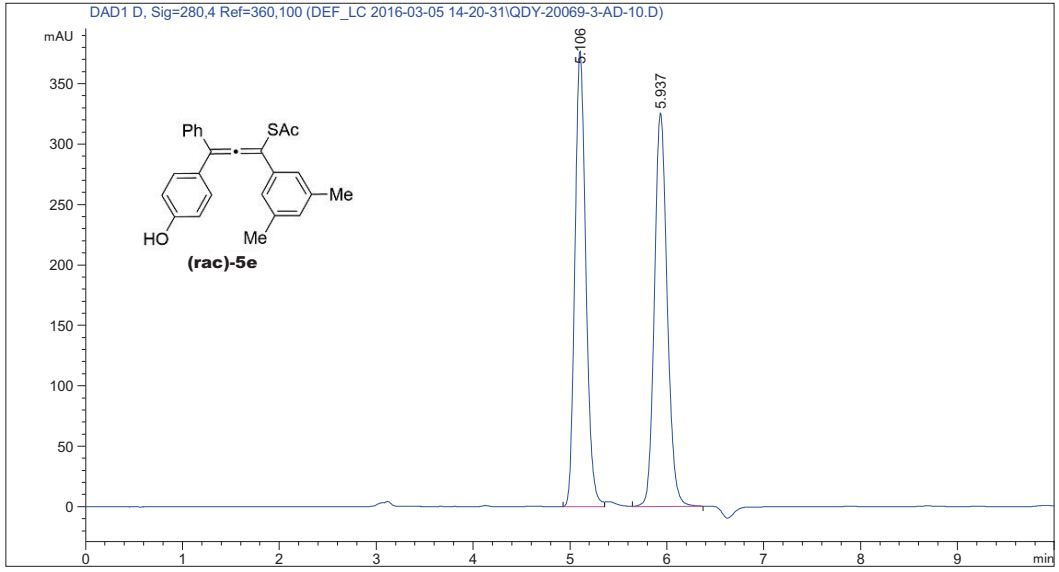



```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.918 BB    0.2243  130.28934    9.02971   4.0040
   2  16.450 BB    0.4331 3123.68799  112.10854  95.9960

Totals :                  3253.97733  121.13826
```

Supplementary Figure 191. HPLC spectrum for 5d

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\QDY-20069-3-AD-10.D
Sample Name:

Acq. Operator : Seq. Line : 16
Acq. Instrument : Instrument 1 Location : Vial 33
Injection Date : 3/5/2016 7:28:45 PM Inj : 1
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\AD-10-20.M
Last changed : 3/5/2016 7:11:35 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed : 6/26/2016 2:31:40 PM
(modified after loading)
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

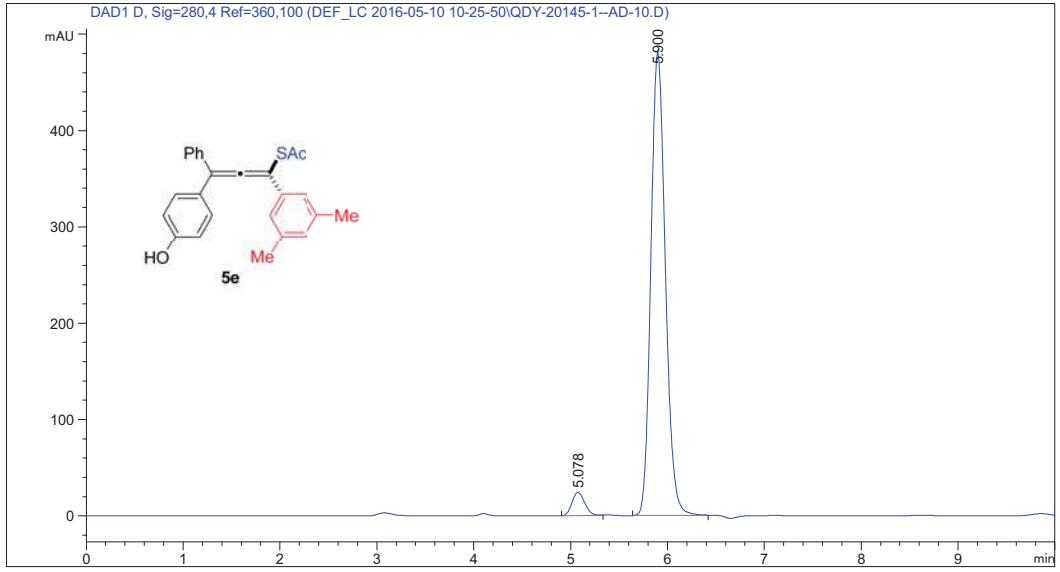
Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.106	BV	0.1192	2947.91675	377.19528	49.7389
2	5.937	VB	0.1407	2978.87012	325.63467	50.2611

Totals : 5926.78687 702.82996

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-10 10-25-50\QDY-20145-1--AD-10.D
Sample Name:

Acq. Operator : Seq. Line : 87
Acq. Instrument : Instrument 1 Location : Vial 31
Injection Date : 5/11/2016 6:35:56 PM Inj : 1
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-05-10 10-25-50\AD-10-20.M
Last changed : 11/28/2014 10:15:17 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed : 6/26/2016 2:31:40 PM
(modified after loading)
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=280,4 Ref=360,100

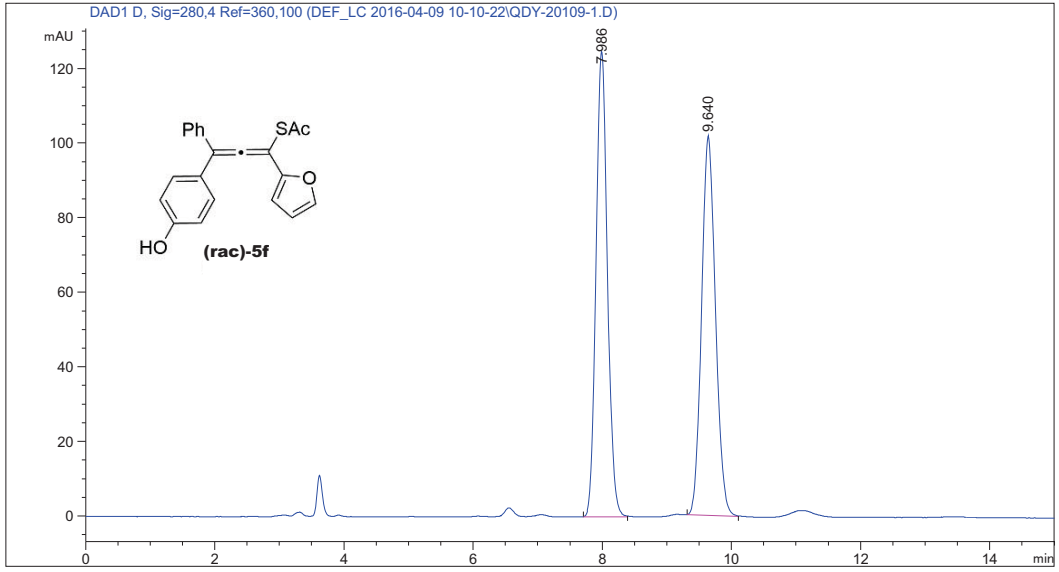
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.078	BB	0.1423	219.88640	24.14296	4.2105
2	5.900	BB	0.1612	5002.39453	481.36807	95.7895

Totals : 5222.28093 505.51103

Supplementary Figure 192. HPLC spectrum for 5e

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-09 10-10-22\QDY-20109-1.D
Sample Name:

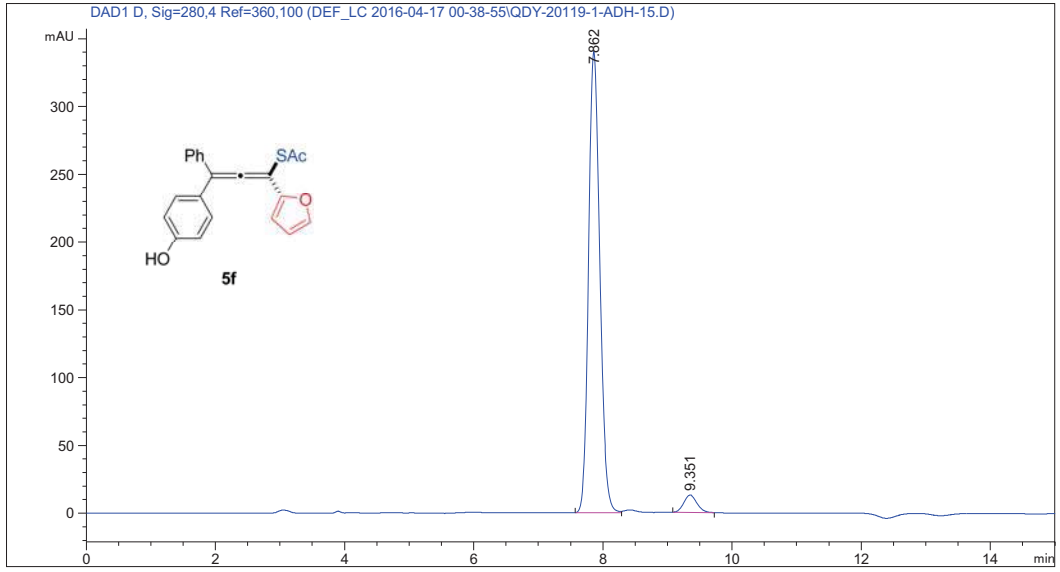
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 4/9/2016 10:24:09 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-09 10-10-22\AD-15-30.M
Last changed    : 4/9/2016 10:50:56 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:19:48 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.986 BB    0.1911 1530.46655  124.77222  50.0747
   2   9.640 BB    0.2329 1525.90210  101.77819  49.9253

Totals :                  3056.36865  226.55042
```

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-17 00-38-55\QDY-20119-1-ADH-15.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   4
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 4/17/2016 1:13:12 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-17 00-38-55\AD-15-22.M
Last changed    : 9/15/2015 8:34:07 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:19:48 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

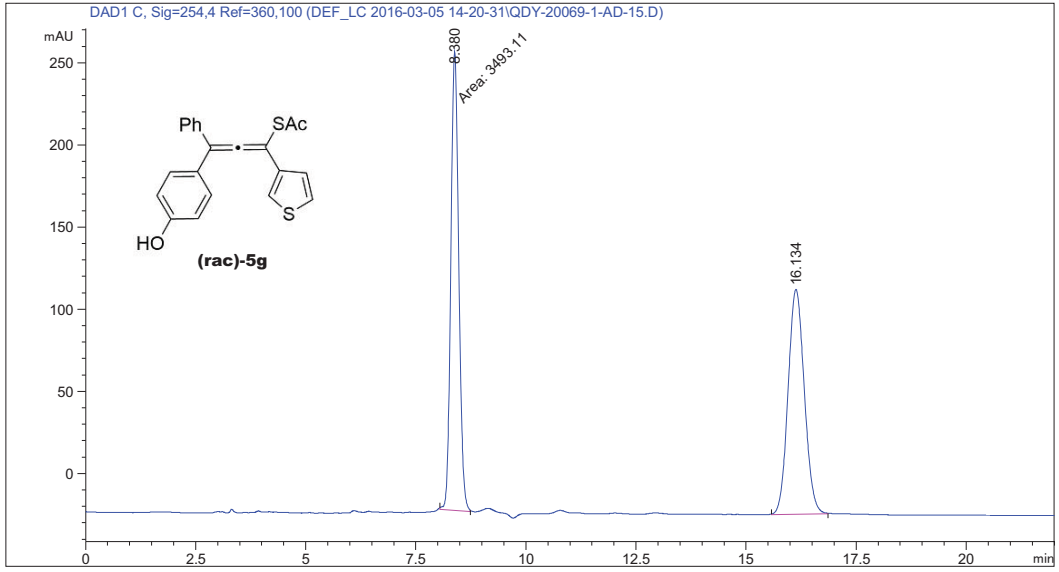



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.862 BB    0.1866 4096.49463  340.03119  95.6900
   2   9.351 BB    0.2203  184.51270   12.94315   4.3100

Totals :                  4281.00732  352.97434

==========================================================================
```

Supplementary Figure 193. HPLC spectrum for 5f

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\QDY-20069-1-AD-15.D
Sample Name:

Acq. Operator	:	Seq. Line	: 6
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 3/5/2016 3:54:17 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\AD-15-22.M		
Last changed	: 3/5/2016 4:11:52 PM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 9:27:49 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

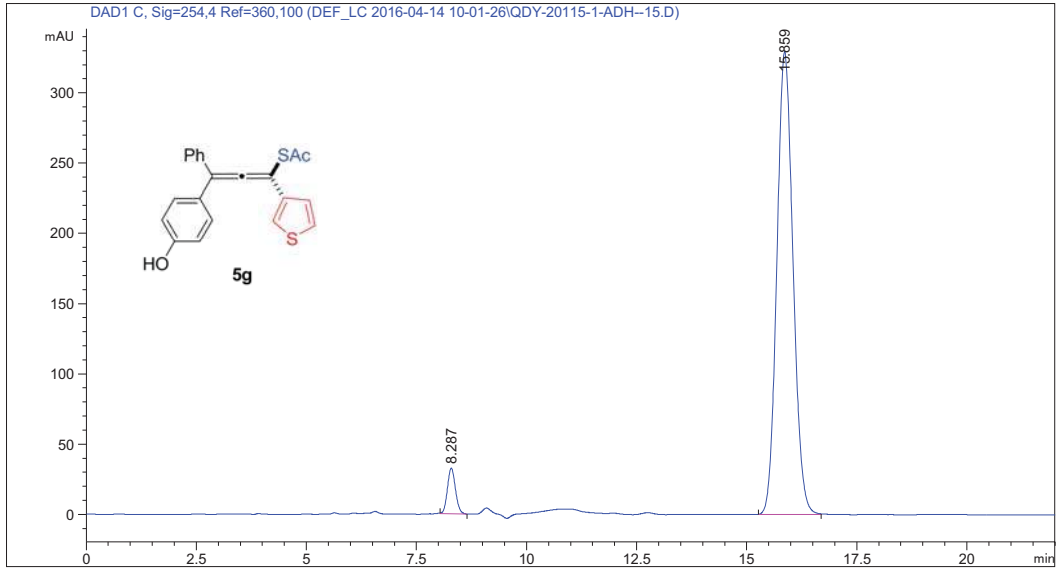
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.380	MM	0.2083	3493.10522	279.45023	49.9186
2	16.134	BB	0.4008	3504.49414	136.82832	50.0814
Totals :				6997.59937	416.27855	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-14 10-01-26\QDY-20115-1-ADH--15.D
Sample Name:

Acq. Operator	:	Seq. Line	: 35
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 4/14/2016 9:48:14 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-14 10-01-26\AD-15-22.M		
Last changed	: 9/15/2015 8:34:07 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 9:27:49 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

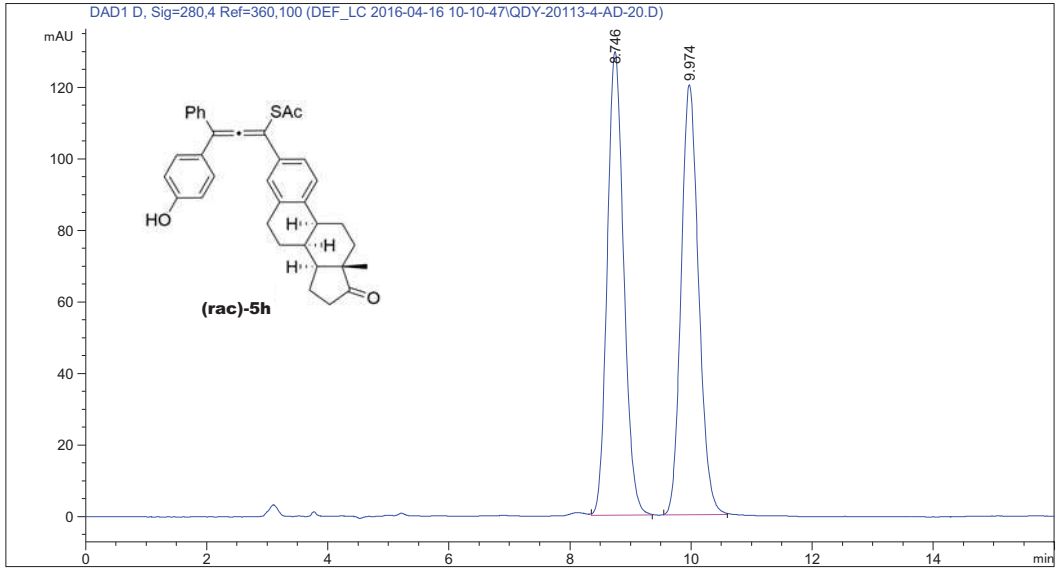
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.287	BB	0.1976	412.45316	32.60120	4.6671
2	15.859	BB	0.3966	8424.96777	329.19797	95.3329
Totals :				8837.42093	361.79917	

Supplementary Figure 194. HPLC spectrum for 5g

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-16 10-10-47\QDY-20113-4-AD-20.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   7
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 4/16/2016 11:48:40 AM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-16 10-10-47\AD-20-20.M
Last changed    : 7/4/2015 10:27:19 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:40:34 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

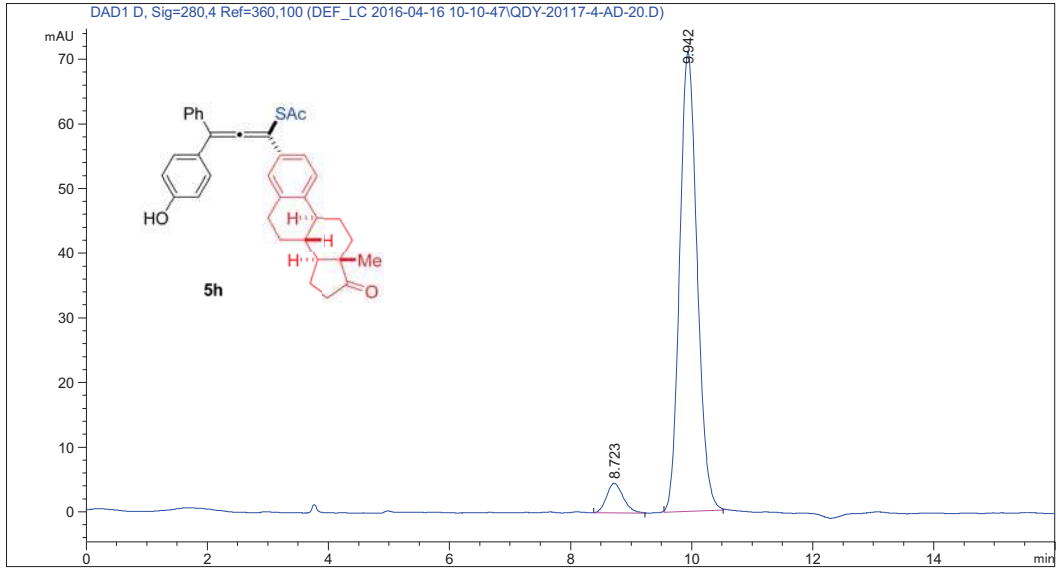



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.746 BB    0.2927 2451.85254  129.59225  50.4080
   2   9.974 BB    0.3102 2412.16650  120.15545  49.5920

Totals :                  4864.01904  249.74770

==========================================================================
```

Instrument 1 6/26/2016 8:43:09 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-16 10-10-47\QDY-20117-4-AD-20.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   6
Acq. Instrument : Instrument 1                    Location : Vial 34
Injection Date  : 4/16/2016 11:27:34 AM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-16 10-10-47\AD-20-20.M
Last changed    : 7/4/2015 10:27:19 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:40:34 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



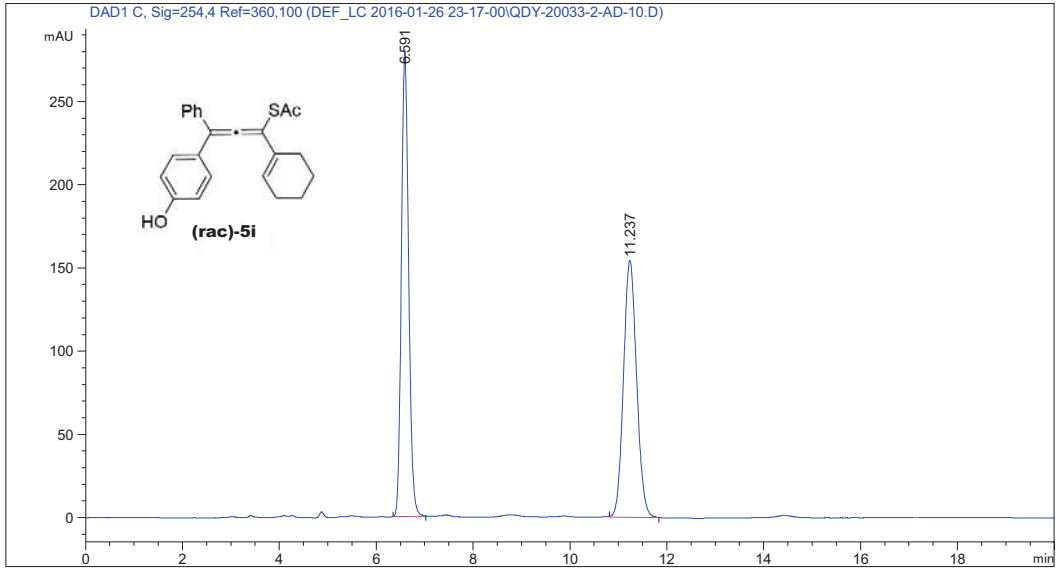

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.723 BB    0.2887   86.59442   4.57787   5.7274
   2   9.942 BB    0.3099 1425.34900  71.10870  94.2726

Totals :                  1511.94342  75.68657

==========================================================================
```

Supplementary Figure 195. HPLC spectrum for 5h

Instrument 1 6/26/2016 8:40:39 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-26 23-17-00\QDY-20033-2-AD-10.D
Sample Name:

Acq. Operator	:	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 1/27/2016 12:12:58 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-01-26 23-17-00\AD-10-30.M		
Last changed	: 11/30/2015 12:57:24 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/27/2016 11:28:15 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

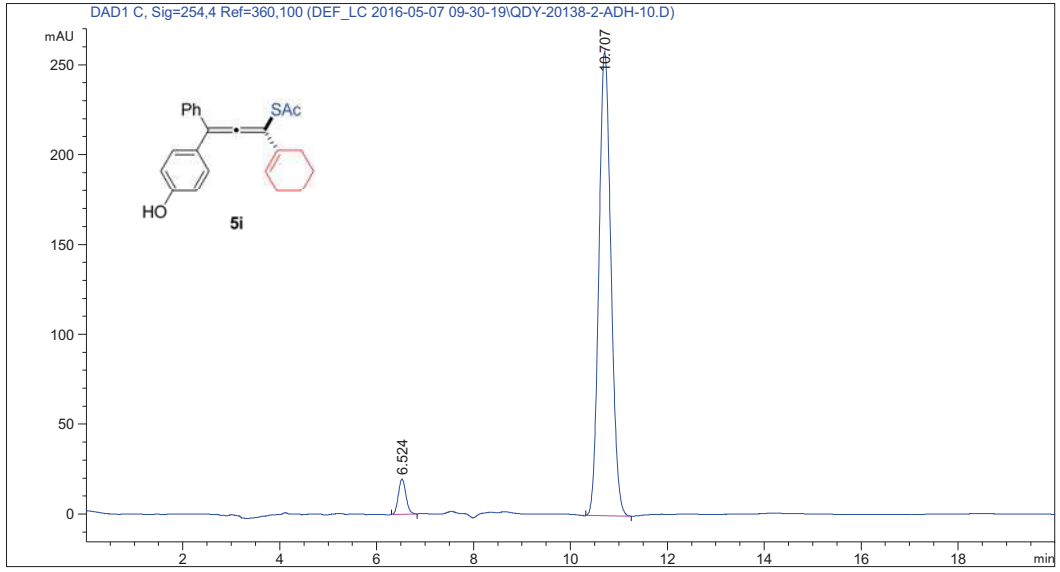
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.591	BB	0.1619	2919.46143	279.40152	50.0987
2	11.237	BB	0.2918	2907.95361	154.31596	49.9013
Totals :				5827.41504	433.71748	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-07 09-30-19\QDY-20138-2-ADH-10.D
Sample Name:

Acq. Operator	:	Seq. Line	: 45
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 5/7/2016 11:46:26 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-05-07 09-30-19\AD-10-20.M		
Last changed	: 11/28/2014 10:15:17 AM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 2:13:09 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

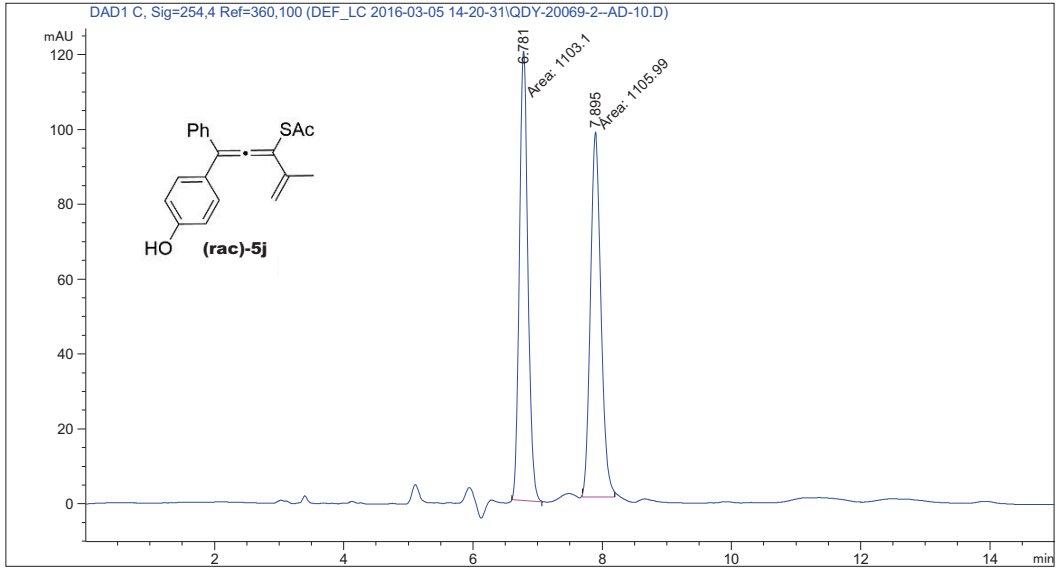
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.524	BB	0.1698	211.17703	19.58147	4.5187
2	10.707	BB	0.2676	4462.21826	258.18164	95.4813
Totals :				4673.39529	277.76311	

Supplementary Figure 196. HPLC spectrum for 5i

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\QDY-20069-2--AD-10.D
Sample Name:

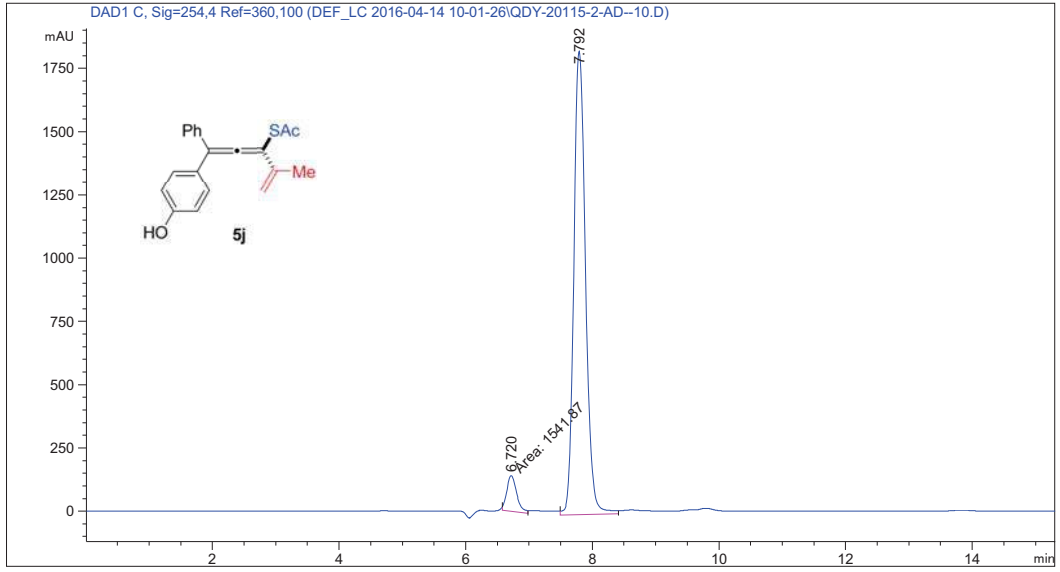
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  15
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 3/5/2016 7:12:41 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\AD-10-20.M
Last changed    : 3/5/2016 7:11:35 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 9:35:27 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.781 MM    0.1531 1103.10168  120.10813  49.9346
   2   7.895 MM    0.1890 1105.98950   97.53231  50.0654

Totals :                  2209.09119  217.64044
```

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-14 10-01-26\QDY-20115-2-AD--10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  31
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 4/14/2016 8:33:53 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-14 10-01-26\AD-10-25.M
Last changed    : 4/14/2016 8:49:15 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/28/2016 12:41:50 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

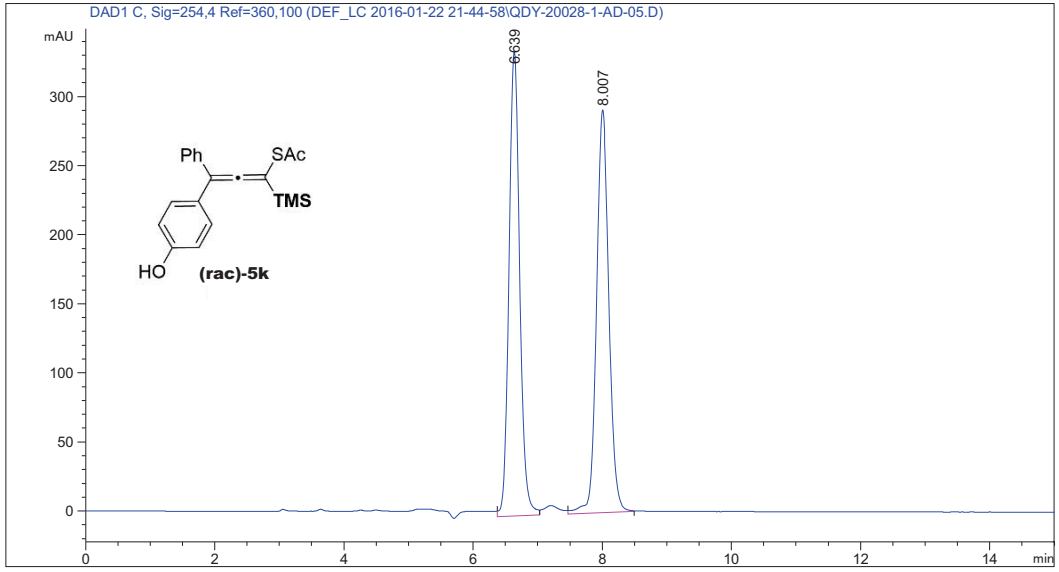



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.720 MM    0.1825 1541.86816  140.83981   6.1453
   2   7.792 BB    0.2001 2.35485e4  1830.42786  93.8547

Totals :                  2.50904e4  1971.26767
```

Supplementary Figure 197. HPLC spectrum for 5j

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-22 21-44-58\QDY-20028-1-AD-05.D
Sample Name:

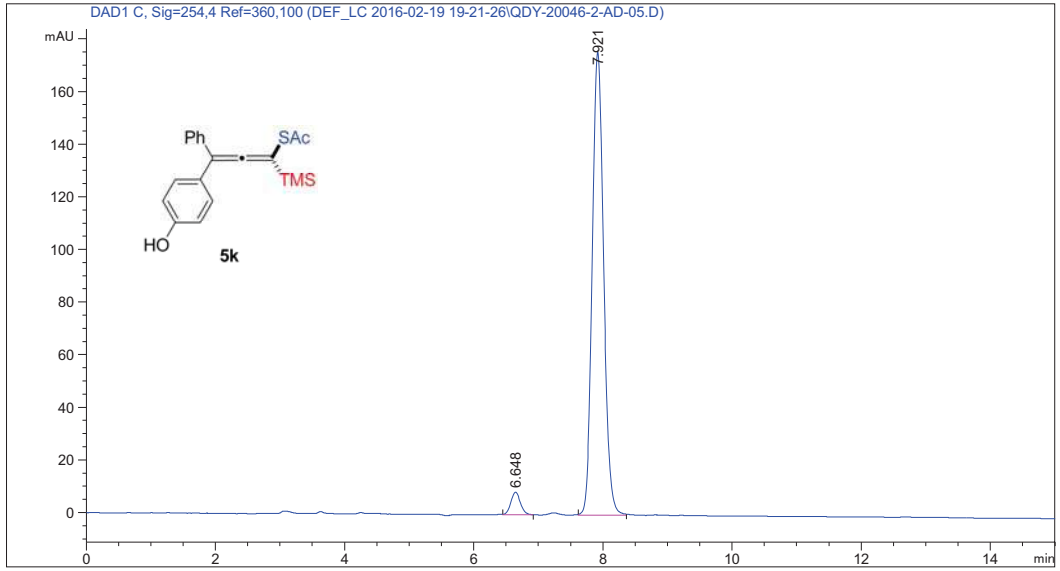
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   7
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 1/22/2016 11:18:59 PM                Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-22 21-44-58\AD-05-20.M
Last changed    : 10/14/2015 3:51:32 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:05:13 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.639 BV    0.1785 3878.38232  336.35043  50.0397
   2   8.007 VB    0.2049 3872.23242  291.63898  49.9603

Totals :                  7750.61475  627.98941

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-02-19 19-21-26\QDY-20046-2-AD-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  10
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 2/19/2016 9:04:18 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-02-19 19-21-26\AD-05-15.M
Last changed    : 7/8/2015 4:31:16 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:05:13 PM
                  (modified after loading)
```

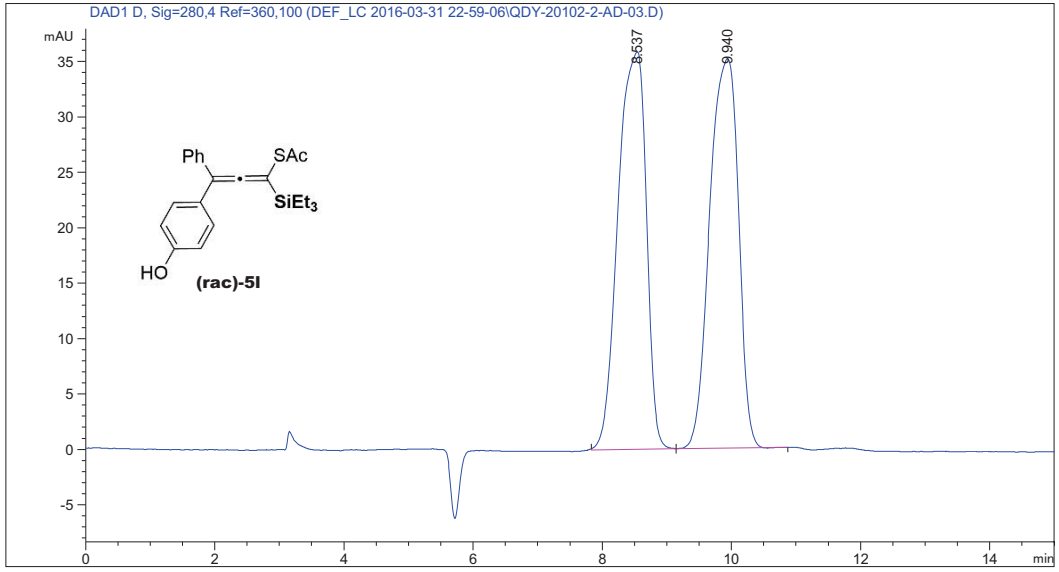



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.648 BB    0.1604   88.50684    8.57913   3.9644
   2   7.921 BB    0.1882 2144.02930  175.93285  96.0356

Totals :                  2232.53613  184.51197

==========================================================================
                        *** End of Report ***
```


Supplementary Figure 198. HPLC spectrum for 5k

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-31 22-59-06\QDY-20102-2-AD-03.D
Sample Name:

Acq. Operator	:	Seq. Line	: 7
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 4/1/2016 1:05:56 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-03-31 22-59-06\AD-03-30.M		
Last changed	: 1/30/2015 10:17:06 AM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 3:55:25 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

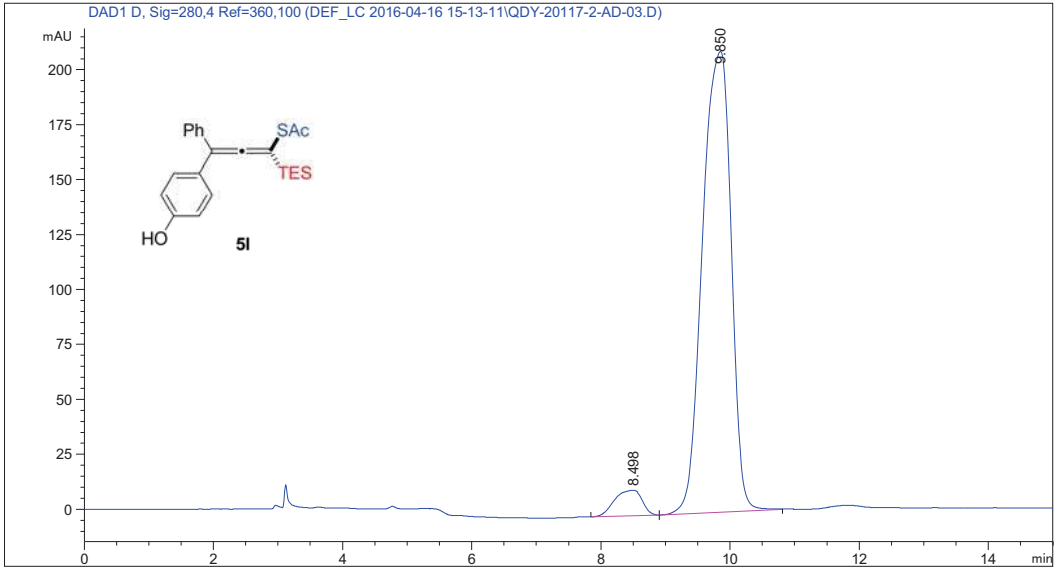
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.537	BV	0.4946	1090.32153	35.86992	50.1032
2	9.940	VB	0.5005	1085.82886	35.13862	49.8968
Totals :				2176.15039	71.00854	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-16 15-13-11\QDY-20117-2-AD-03.D
Sample Name:

Acq. Operator	:	Seq. Line	: 7
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 4/16/2016 5:09:50 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-16 15-13-11\AD-03-20.M		
Last changed	: 4/16/2016 5:22:49 PM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 3:55:25 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

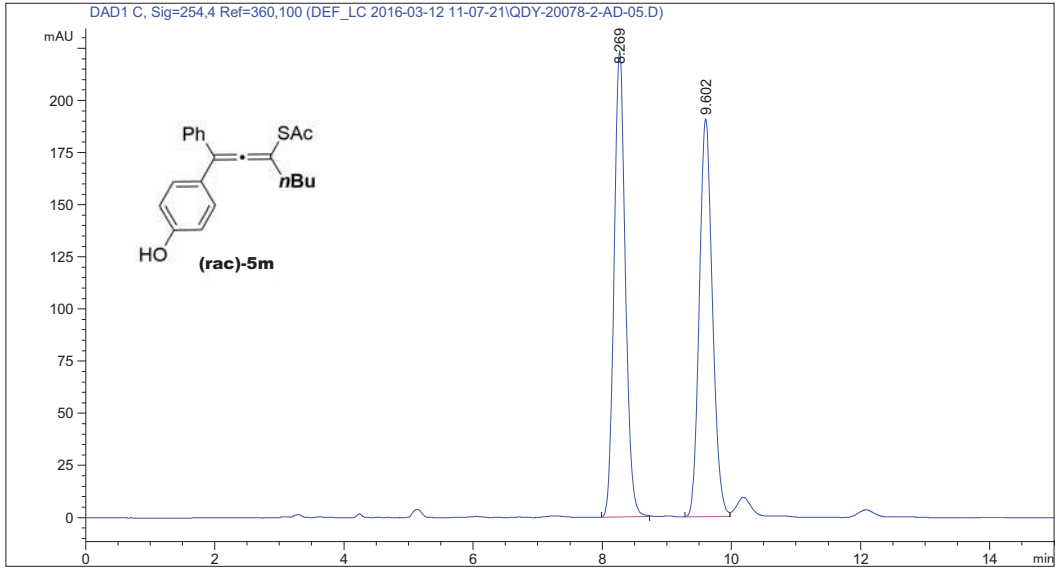
Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.498	BV	0.4241	341.95050	11.59381	5.0289
2	9.850	VB	0.5034	6457.77686	209.60730	94.9711
Totals :				6799.72736	221.20111	

Supplementary Figure 199. HPLC spectrum for 5l

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-12 11-07-21\QDY-20078-2-AD-05.D
Sample Name:

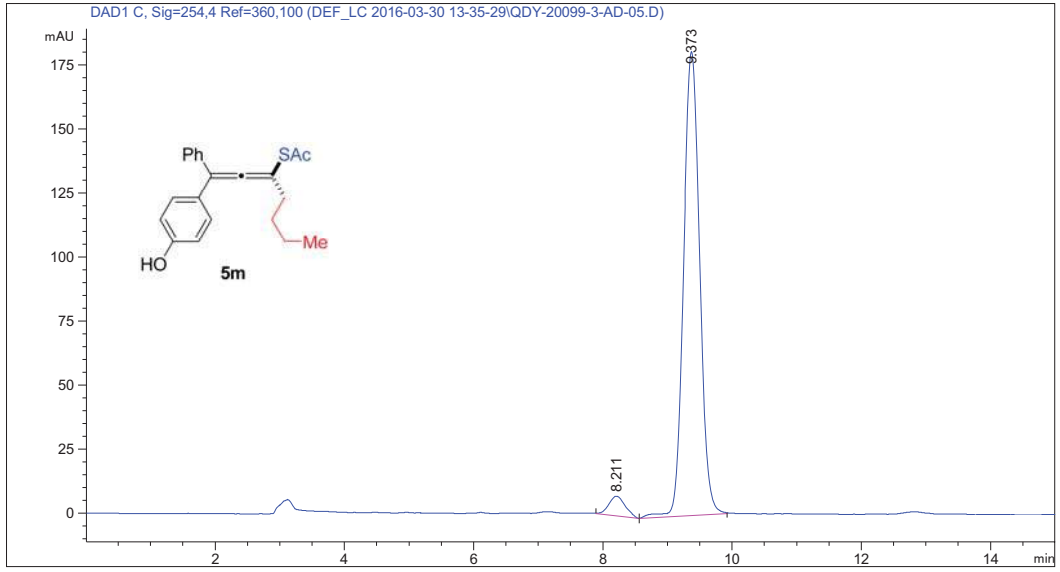
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   4
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 3/12/2016 12:03:15 PM              Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-12 11-07-21\AD-05-30.M
Last changed    : 3/12/2016 12:20:26 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 3:10:06 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.269 BB    0.1876 2708.51978  223.12842  50.1121
   2   9.602 BV    0.2190 2696.39819  190.68352  49.8879

Totals :                  5404.91797  413.81194
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-30 13-35-29\QDY-20099-3-AD-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  13
Acq. Instrument : Instrument 1                    Location : Vial 33
Injection Date  : 3/30/2016 6:42:27 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-30 13-35-29\AD-05-15.M
Last changed    : 7/8/2015 4:31:16 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:50:44 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

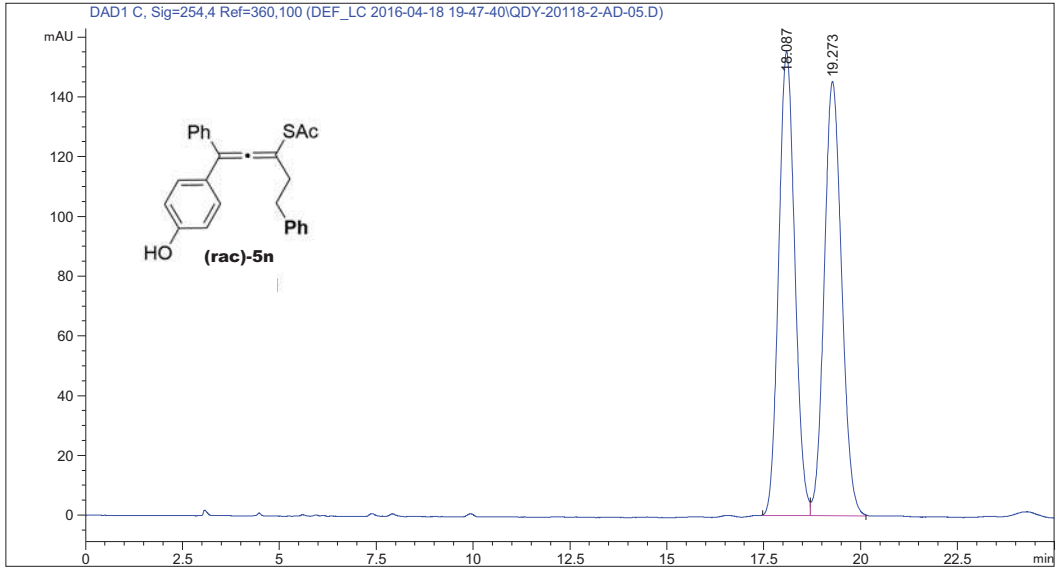



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.211 BV    0.2769  136.74849    7.71228   4.1759
   2   9.373 VB    0.2720 3137.98779  181.20929  95.8241

Totals :                  3274.73628  188.92157

==========================================================================
```

Supplementary Figure 200. HPLC spectrum for 5m

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-18 19-47-40\QDY-20118-2-AD-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 24
Acq. Instrument	: Instrument 1	Location	: Vial 35
Injection Date	: 4/19/2016 2:32:40 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-18 19-47-40\AD-05-30.M		
Last changed	: 4/19/2016 2:31:49 AM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 4:03:06 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

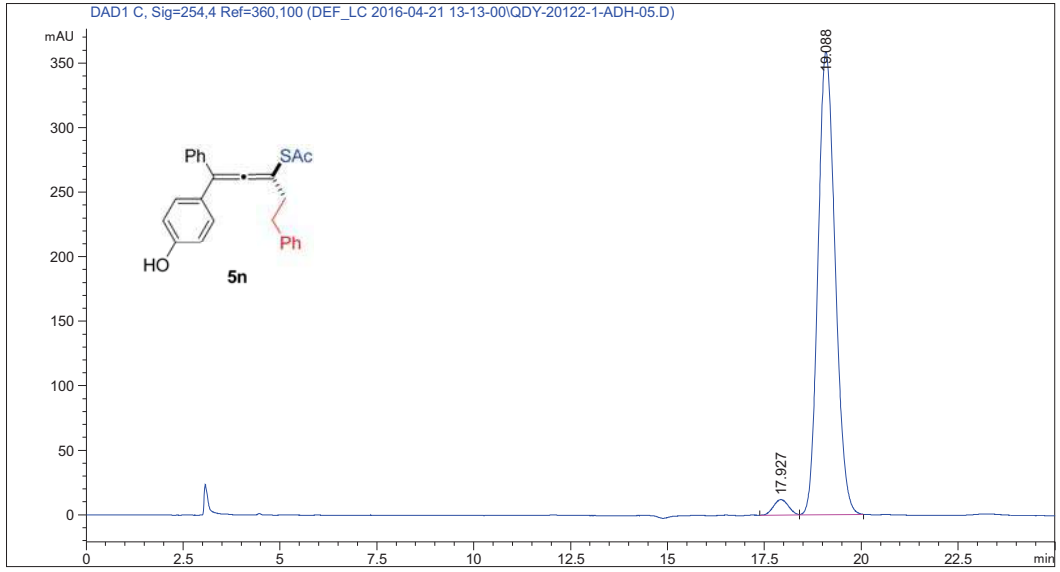
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.087	BV	0.4539	4498.68213	155.34293	49.6694
2	19.273	VB	0.4899	4558.57422	145.34494	50.3306
Totals :				9057.25635	300.68787	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-21 13-13-00\QDY-20122-1-ADH-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 35
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 4/22/2016 12:24:04 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-21 13-13-00\AD-05-30.M		
Last changed	: 8/31/2012 5:22:01 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 4:03:06 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

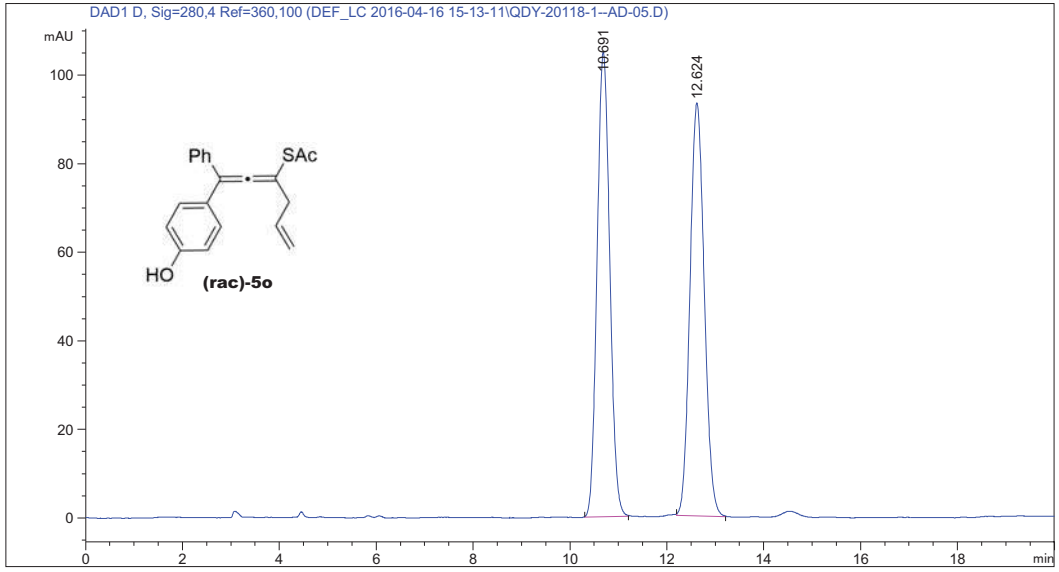
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.927	BV	0.4259	323.51538	12.02499	2.8466
2	19.088	VB	0.4830	1.10415e4	358.84393	97.1534
Totals :				1.13650e4	370.86892	

Supplementary Figure 201. HPLC spectrum for 5n

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-16 15-13-11\QDY-20118-1--AD-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 21
Acq. Instrument	: Instrument 1	Location	: Vial 34
Injection Date	: 4/16/2016 9:20:06 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-16 15-13-11\AD-05-20.M		
Last changed	: 10/14/2015 3:51:32 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 1:41:03 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

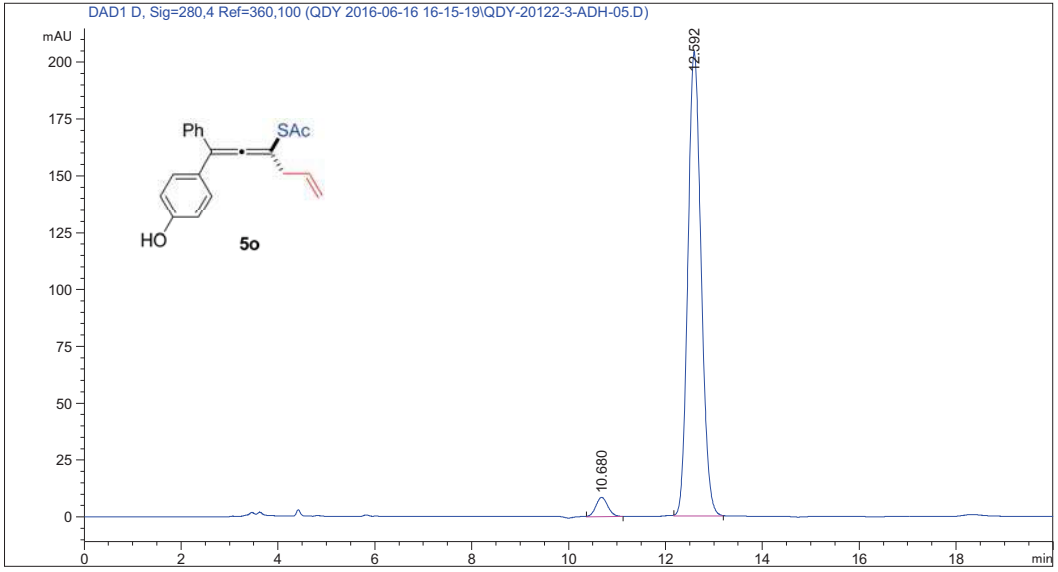
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.691	BB	0.2858	1906.98474	105.06792	49.9276
2	12.624	BB	0.3212	1912.51782	93.29216	50.0724
Totals :				3819.50256	198.36008	

Data File C:\CHEM32\1\DATA\QDY 2016-06-16 16-15-19\QDY-20122-3-ADH-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 20
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 6/16/2016 10:44:01 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\QDY 2016-06-16 16-15-19\AD-05-20.M		
Last changed	: 6/16/2016 10:43:09 PM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 1:41:03 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

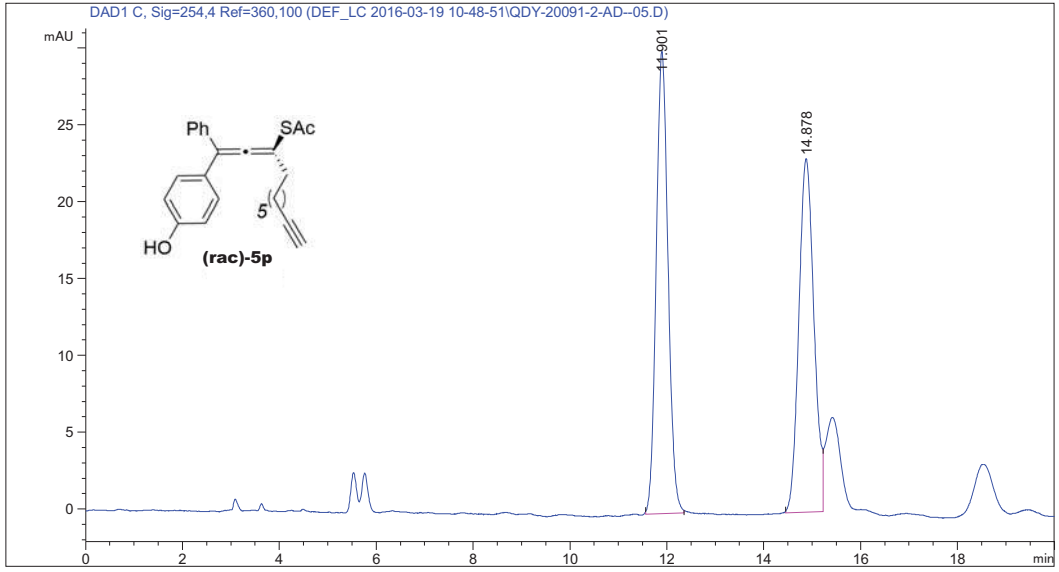
Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.680	BB	0.2657	145.52939	8.50029	3.6314
2	12.592	BB	0.2946	3862.04248	204.24312	96.3686
Totals :				4007.57187	212.74341	

Supplementary Figure 202. HPLC spectrum for 5o

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-19 10-48-51\QDY-20091-2-AD--05.D
Sample Name:

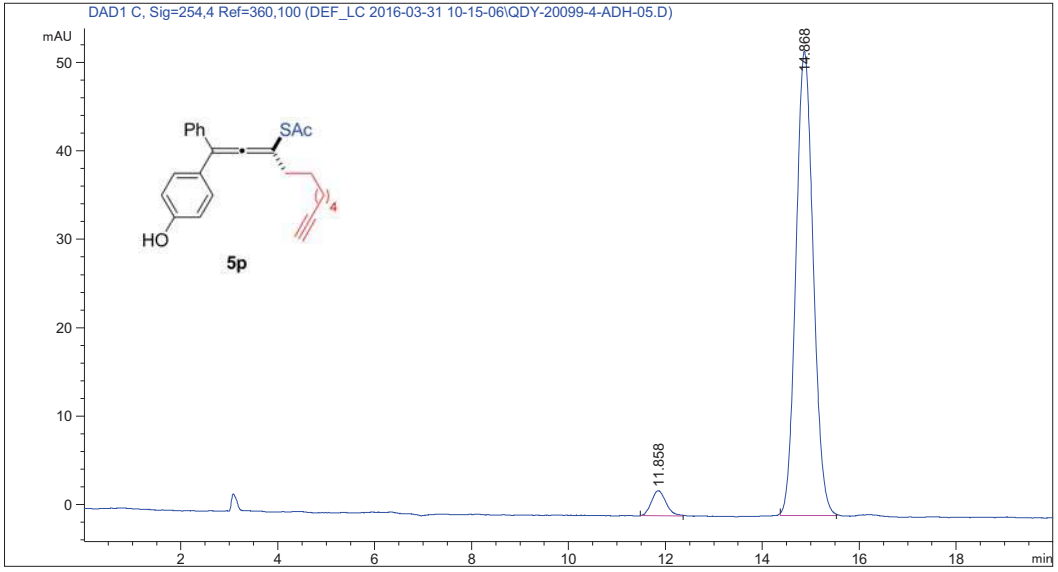
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  32
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 3/19/2016 10:32:01 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-19 10-48-51\AD-05-30.M
Last changed    : 3/19/2016 10:31:10 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:35:21 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.901 BB    0.2634  508.70099   30.07041  50.4435
   2  14.878 BV    0.3393  499.75665   23.01589  49.5565

Totals :                  1008.45764   53.08629
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-31 10-15-06\QDY-20099-4-ADH-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  5
Acq. Instrument : Instrument 1                     Location : Vial 34
Injection Date  : 3/31/2016 11:24:51 AM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-31 10-15-06\AD-05-20.M
Last changed    : 3/31/2016 11:27:42 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:32:29 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

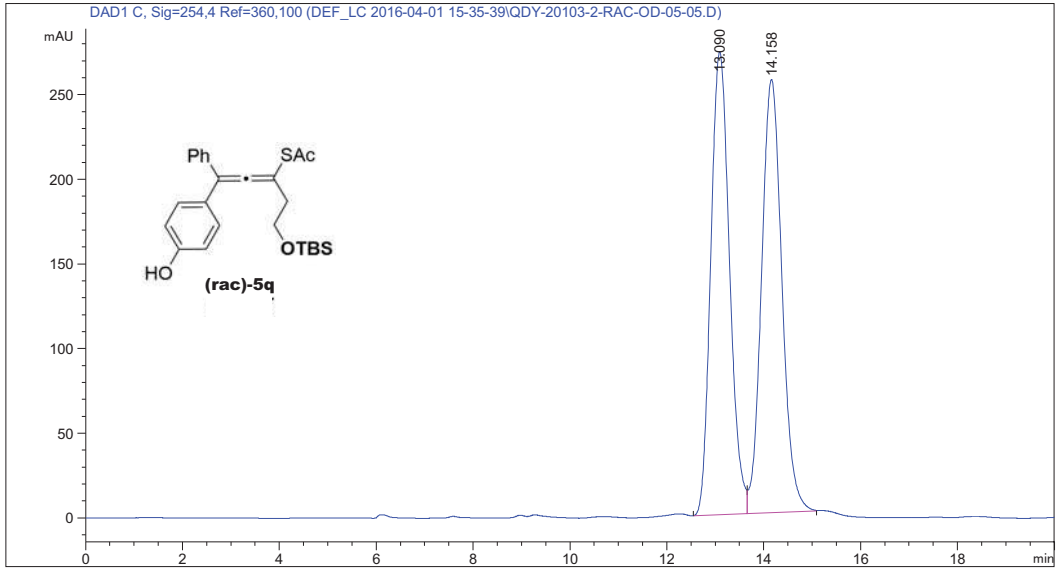



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.858 BB    0.2872   58.77607    2.81997   4.3129
   2  14.868 BB    0.3859 1304.01526   52.48608  95.6871

Totals :                  1362.79133   55.30606
```

Supplementary Figure 203. HPLC spectrum for 5p

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\QDY-20103-2-RAC-OD-05-05.D
Sample Name:

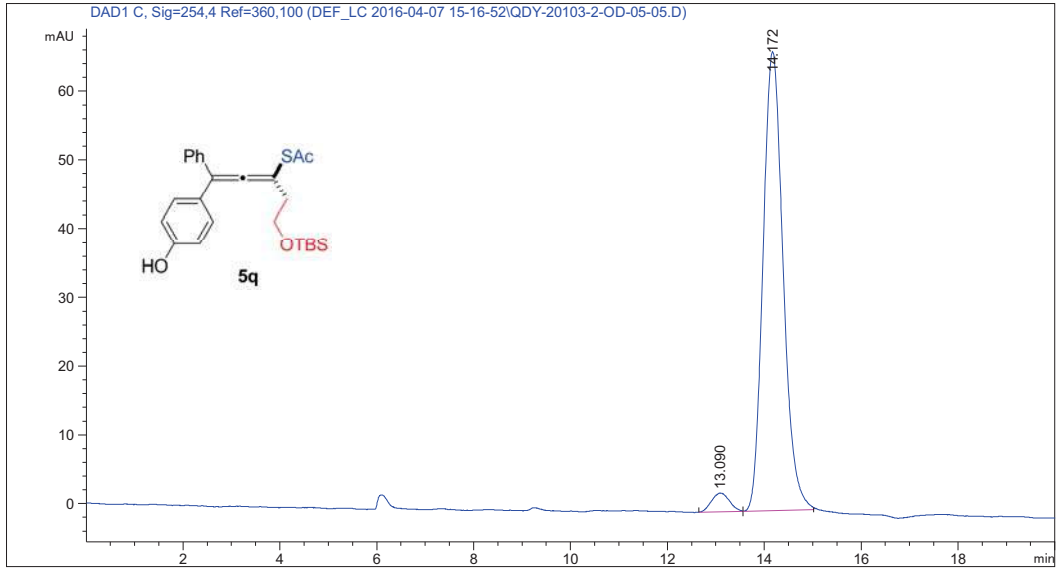
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  85
Acq. Instrument : Instrument 1                    Location : Vial 33
Injection Date  : 4/2/2016 10:04:16 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\OD-05-40-0.5.M
Last changed    : 12/11/2014 6:35:45 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 4:11:48 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.090 BV    0.4200 7359.00879  273.43948  49.6634
   2  14.158 VB    0.4540 7458.77197  255.94975  50.3366

Totals :                  1.48178e4   529.38924

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-07 15-16-52\QDY-20103-2-OD-05-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 4/7/2016 3:30:20 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-07 15-16-52\OD-05-20-0.5.M
Last changed    : 4/7/2016 10:27:36 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 4:09:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

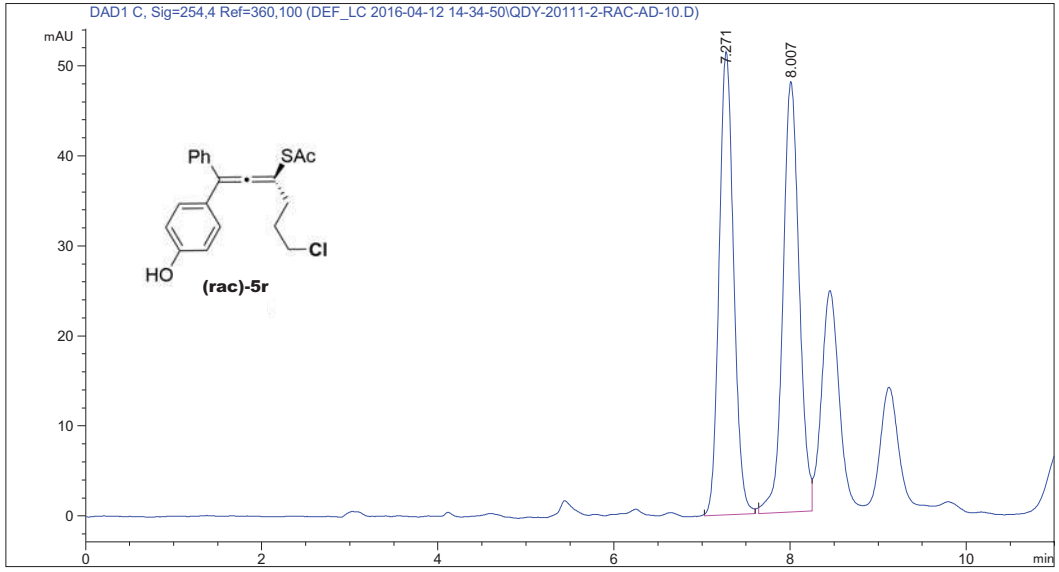



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.090 BV    0.3273   69.24229    2.71682   3.4859
   2  14.172 VB    0.4450 1917.12439   66.75624  96.5141

Totals :                  1986.36668   69.47306

==========================================================================
```

Supplementary Figure 204. HPLC spectrum for 5q

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-12 14-34-50\QDY-20111-2-RAC-AD-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   9
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 4/12/2016 5:37:31 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-12 14-34-50\AD-10-20.M
Last changed    : 11/28/2014 10:15:17 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 4:17:43 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

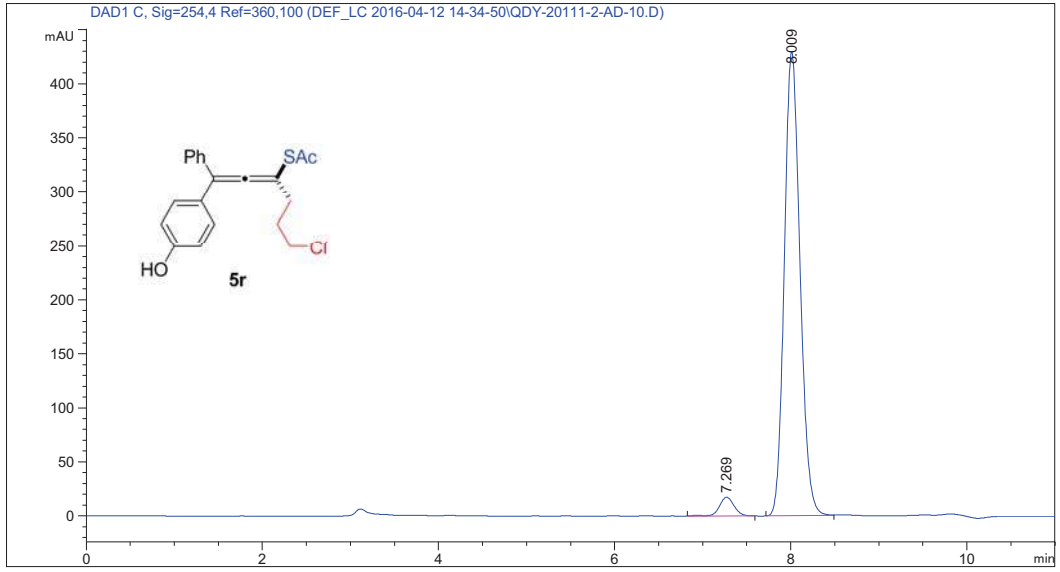



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.271 BB    0.1783  592.31207   51.44891  49.0472
   2   8.007 BV    0.1981  615.32520   47.82840  50.9528

Totals :                  1207.63727   99.27731

==========================================================================
```

Instrument 1 6/26/2016 4:18:33 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-12 14-34-50\QDY-20111-2-AD-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   8
Acq. Instrument : Instrument 1                    Location : Vial 32
Injection Date  : 4/12/2016 5:16:25 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-12 14-34-50\AD-10-20.M
Last changed    : 11/28/2014 10:15:17 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 4:17:43 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



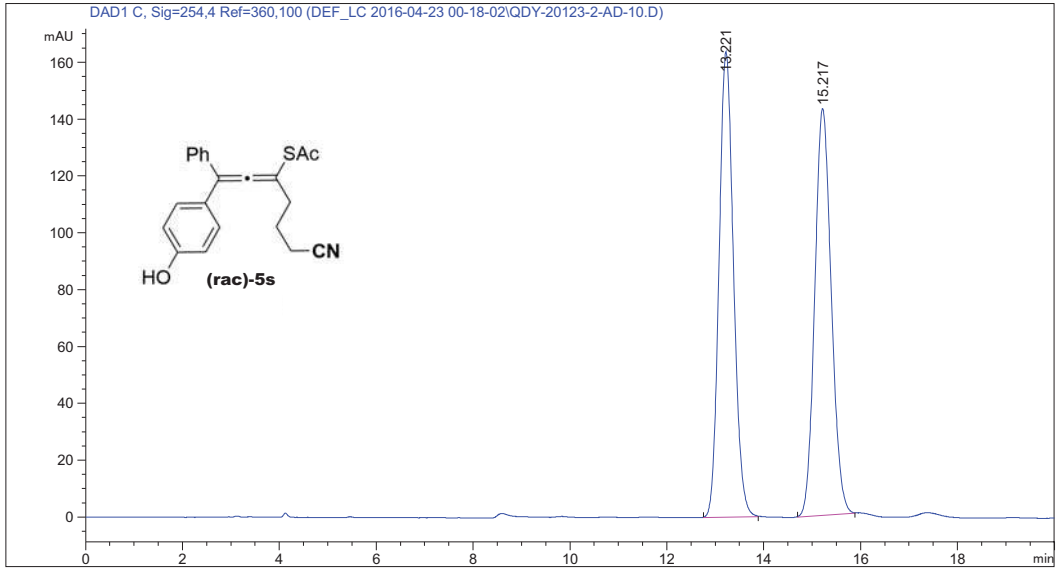

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.269 BB    0.1810  204.44051   17.41126   3.7023
   2   8.009 BB    0.1925 5317.61865  429.46994  96.2977

Totals :                  5522.05916  446.88120

==========================================================================
```

Supplementary Figure 205. HPLC spectrum for 5r

Instrument 1 6/26/2016 4:20:35 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-23 00-18-02\QDY-20123-2-AD-10.D
Sample Name:

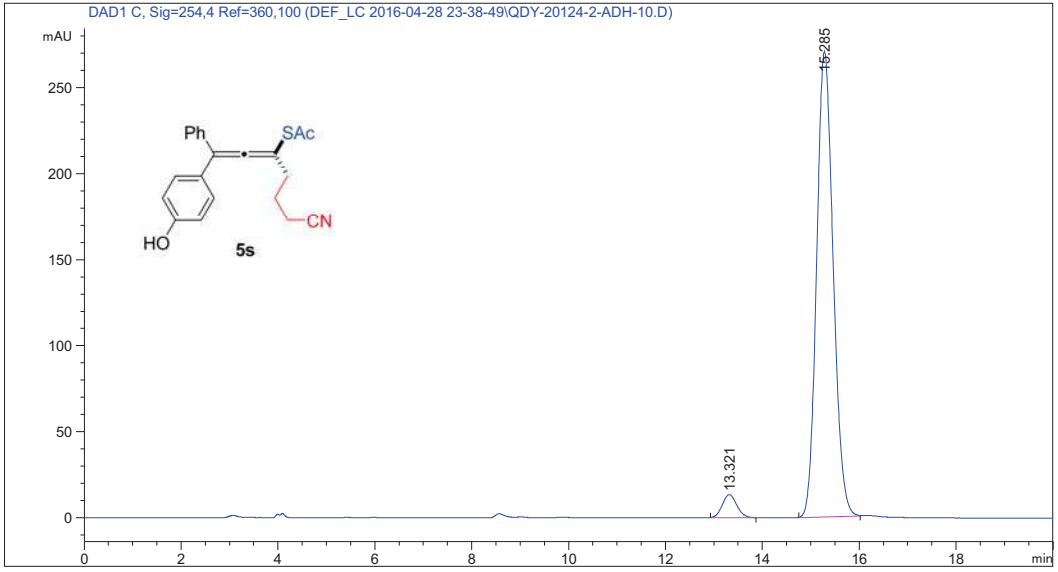
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   7
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 4/23/2016 2:07:44 AM                Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-23 00-18-02\AD-10-30.M
Last changed    : 11/30/2015 12:57:24 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 1:41:03 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.221 BB    0.3219 3394.72974  163.70085  50.0480
   2  15.217 BB    0.3682 3388.21924  143.13855  49.9520

Totals :                  6782.94897  306.83940

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-28 23-38-49\QDY-20124-2-ADH-10.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                     Location : Vial 34
Injection Date  : 4/28/2016 11:51:09 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-28 23-38-49\AD-10-25.M
Last changed    : 4/28/2016 11:50:16 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\IC-20-70.M
Last changed    : 6/18/2016 10:41:44 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

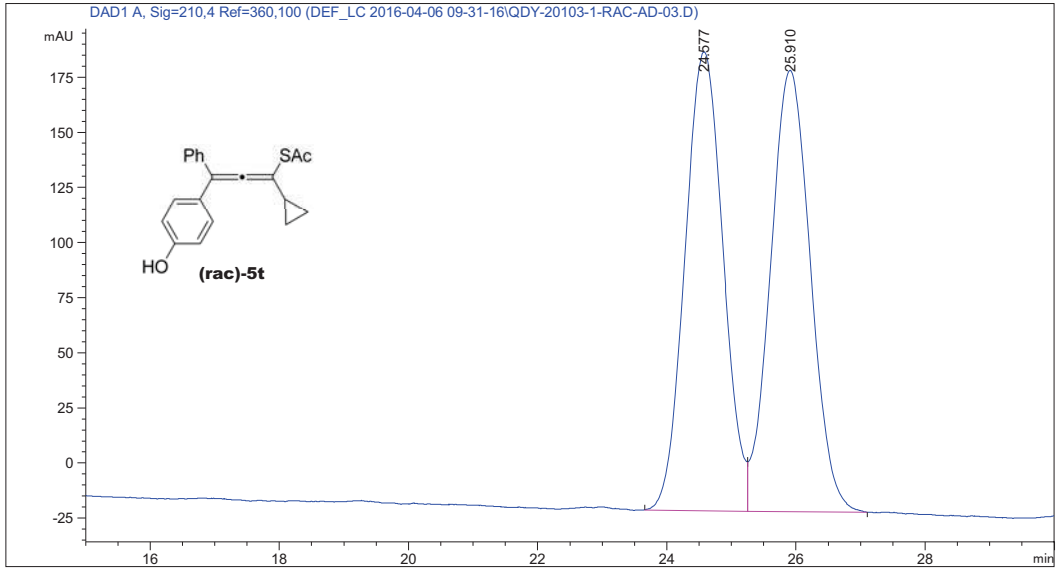



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.321 BB    0.3241   282.51279  13.50160   4.2193
   2  15.285 BB    0.3685 6413.16797  270.58994  95.7807

Totals :                  6695.68076  284.09153
```


Supplementary Figure 206. HPLC spectrum for 5s

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-06 09-31-16\QDY-20103-1-RAC-AD-03.D
Sample Name:

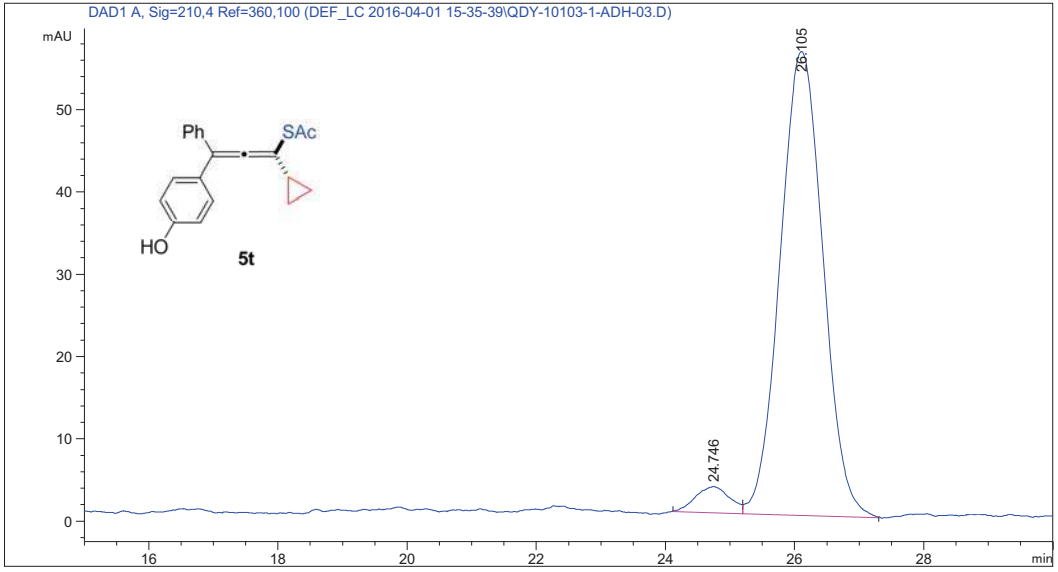
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  45
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 4/6/2016 11:40:19 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-06 09-31-16\AD-03-30.M
Last changed    : 1/30/2015 10:17:06 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/28/2016 11:26:02 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.577 BV    0.6475 8555.16602  208.15483  49.7271
   2  25.910 VB    0.6621 8649.06543  200.16136  50.2729

Totals :                  1.72042e4   408.31619

==========================================================================
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\QDY-10103-1-ADH-03.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   6
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 4/1/2016 4:59:28 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 6.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\AD-03-30.M
Last changed    : 1/30/2015 10:17:06 AM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/28/2016 11:20:17 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

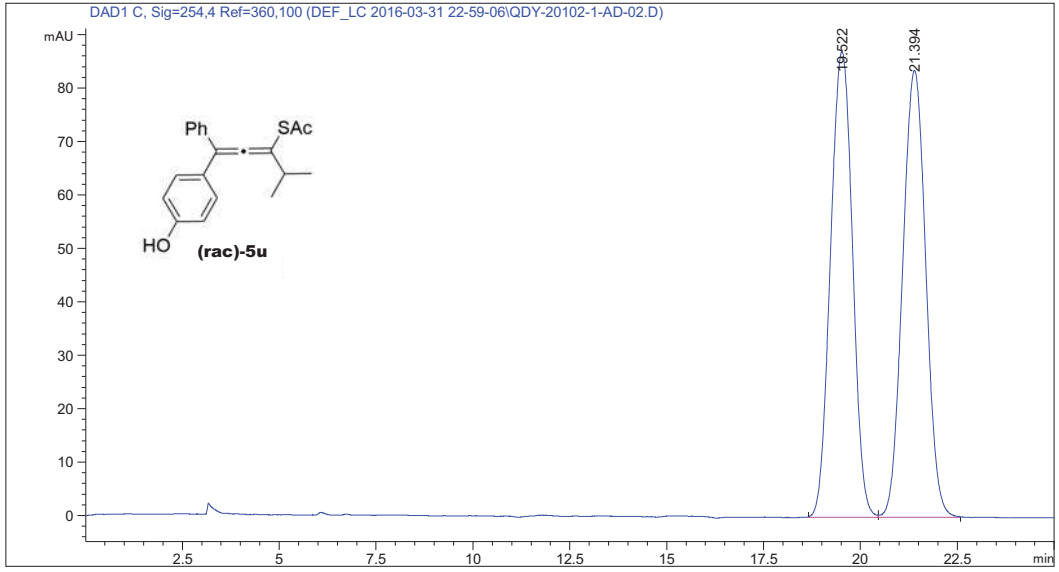



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.746 BV    0.4412  117.73360    3.20055   4.3359
   2  26.105 VB    0.6777 2597.57642   56.36382  95.6641

Totals :                  2715.31002   59.56437
```

Supplementary Figure 207. HPLC spectrum for 5t

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-31 22-59-06\QDY-20102-1-AD-02.D
Sample Name:

Acq. Operator	:	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 4/1/2016 12:08:16 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-03-31 22-59-06\AD-02-20.M		
Last changed	: 4/1/2016 12:19:44 AM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 4:00:03 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

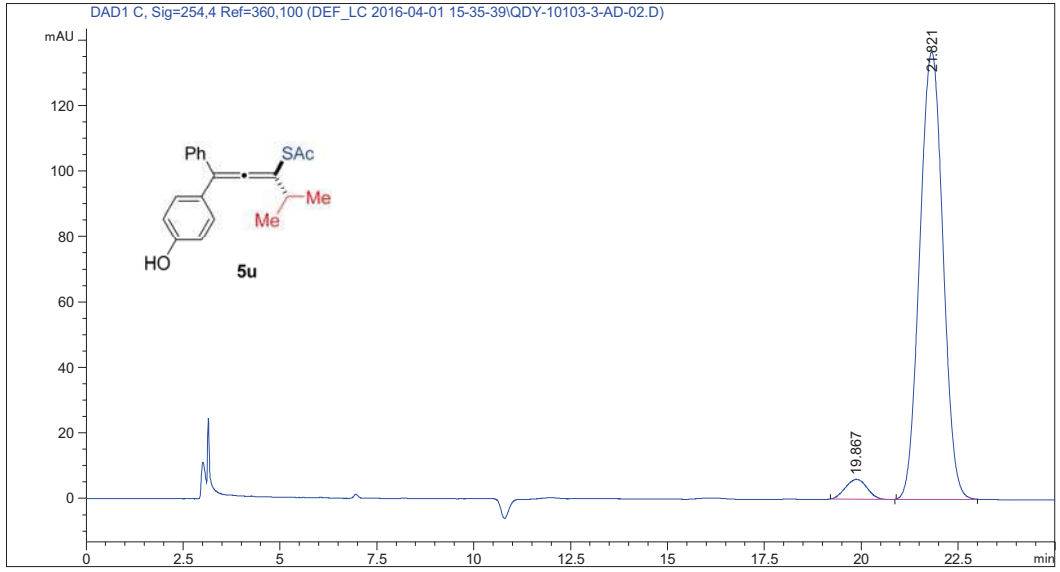
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.522	BV	0.6306	3440.79224	87.14542	49.7928
2	21.394	VB	0.6621	3469.42822	83.61929	50.2072
Totals :				6910.22046	170.76472	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\QDY-10103-3-AD-02.D
Sample Name:

Acq. Operator	:	Seq. Line	: 55
Acq. Instrument	: Instrument 1	Location	: Vial 35
Injection Date	: 4/2/2016 12:03:22 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-04-01 15-35-39\AD-02-30.M		
Last changed	: 2/4/2015 1:17:54 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 4:03:06 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

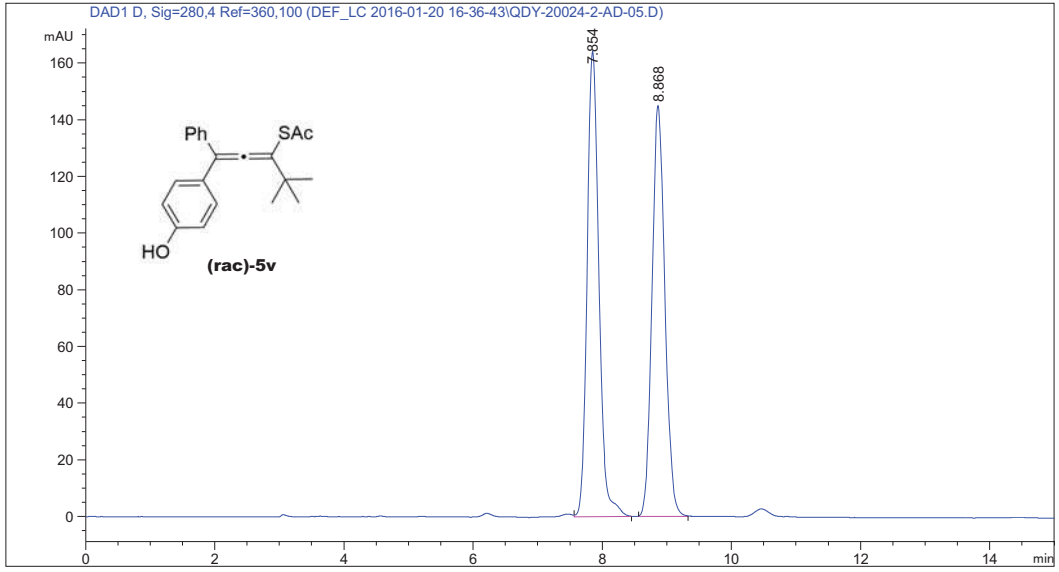
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.867	BB	0.5267	232.61073	6.04875	3.9310
2	21.821	BB	0.6612	5684.76416	136.70180	96.0690
Totals :				5917.37489	142.75055	

Supplementary Figure 208. HPLC spectrum for 5u

Data File C:\CHEM32\1\DATA\DEF_LC 2016-01-20 16-36-43\QDY-20024-2-AD-05.D
Sample Name:

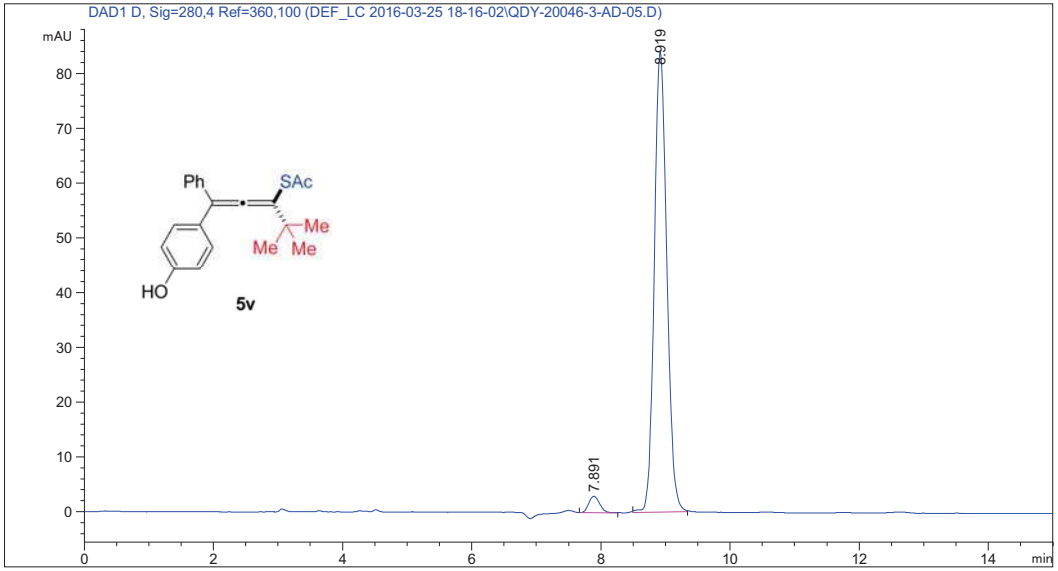
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   6
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 1/20/2016 5:56:11 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-01-20 16-36-43\AD-05-30.M
Last changed    : 1/20/2016 5:55:21 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:39:22 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.854 VB    0.1955 2075.83936  164.15967  50.7349
   2   8.868 BB    0.2163 2015.70105  144.91257  49.2651

Totals :                  4091.54041  309.07224
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-25 18-16-02\QDY-20046-3-AD-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                     Location : Vial 31
Injection Date  : 3/25/2016 6:29:38 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-03-25 18-16-02\AD-05-20.M
Last changed    : 10/14/2015 3:51:32 PM
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 2:39:22 PM
                  (modified after loading)
```



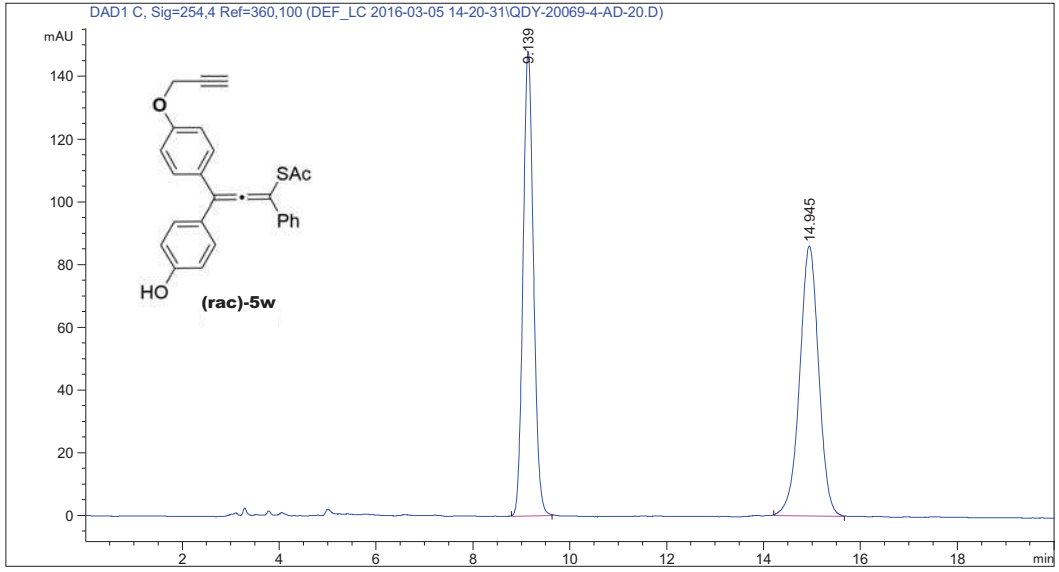

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=280,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.891 BB    0.1840   35.07615    3.00973   3.0115
   2   8.919 BB    0.2069 1129.64929   83.97672  96.9885

Totals :                  1164.72544   86.98645

==========================================================================
                        *** End of Report ***
```


Supplementary Figure 209. HPLC spectrum for 5v

Data File C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\QDY-20069-4-AD-20.D
Sample Name:

Acq. Operator	:	Seq. Line	: 19
Acq. Instrument	: Instrument 1	Location	: Vial 34
Injection Date	: 3/5/2016 8:13:02 PM	Inj	: 1
		Inj Volume	: 5.000 µl

Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-03-05 14-20-31\AD-20-30.M
Last changed : 3/5/2016 8:29:34 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed : 6/26/2016 2:50:44 PM
(modified after loading)
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

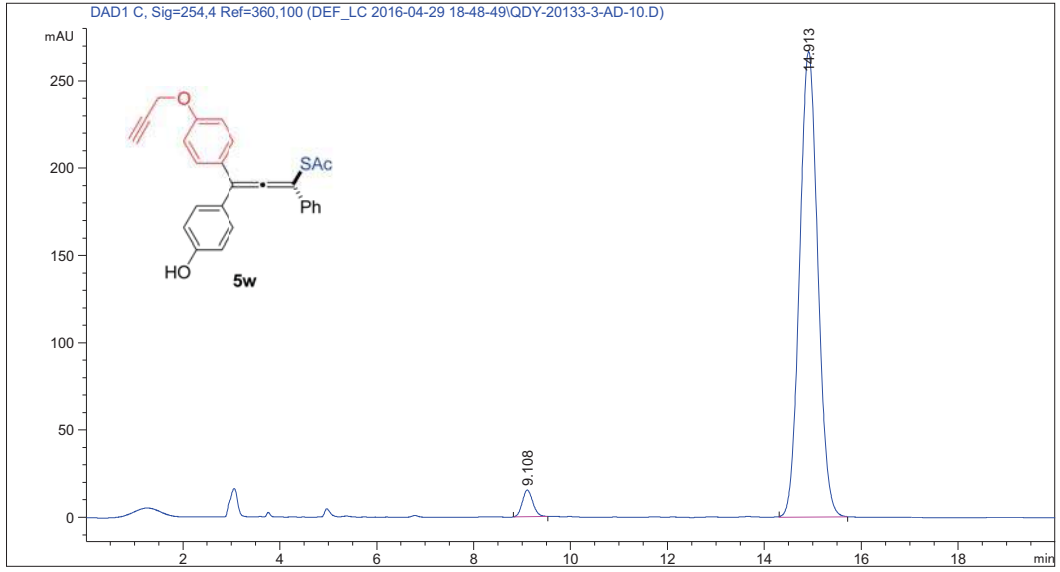
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.139	BB	0.2304	2214.57837	148.15285	48.5121
2	14.945	BB	0.4185	2350.42090	86.10274	51.4879
Totals :				4564.99927	234.25558	

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\QDY-20133-3-AD-10.D
Sample Name:

Acq. Operator	:	Seq. Line	: 14
Acq. Instrument	: Instrument 1	Location	: Vial 36
Injection Date	: 4/29/2016 11:17:11 PM	Inj	: 1
		Inj Volume	: 5.000 µl

Acq. Method : C:\CHEM32\1\DATA\DEF_LC 2016-04-29 18-48-49\AD-20-20.M
Last changed : 4/29/2016 10:34:02 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed : 6/26/2016 2:50:44 PM
(modified after loading)
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

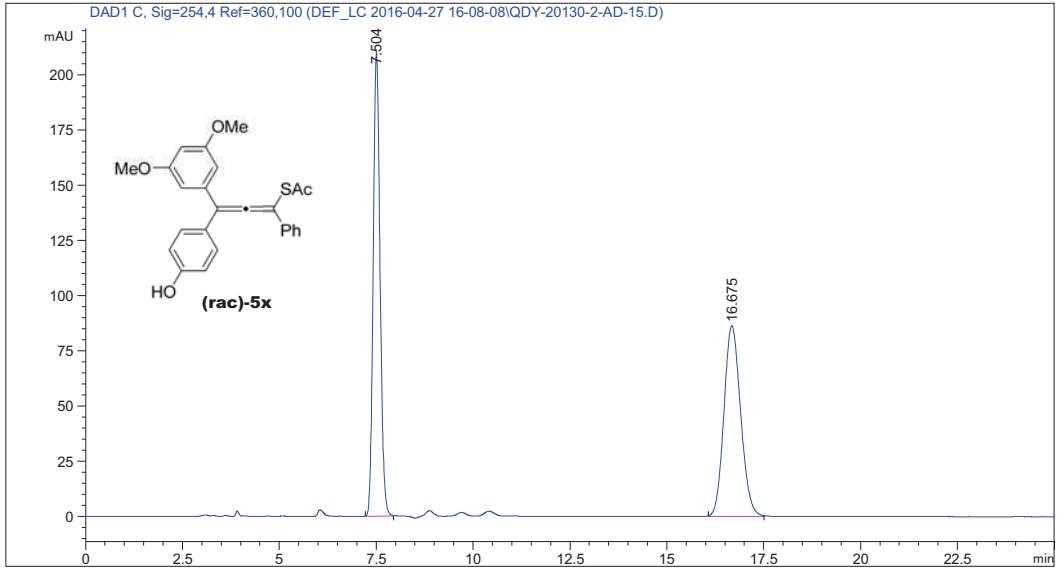
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.108	BB	0.2353	233.07611	15.33749	3.2413
2	14.913	BB	0.4063	6957.69434	266.73419	96.7587
Totals :				7190.77045	282.07168	

Supplementary Figure 210. HPLC spectrum for 5w

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-27 16-08-08\QDY-20130-2-AD-15.D
Sample Name:

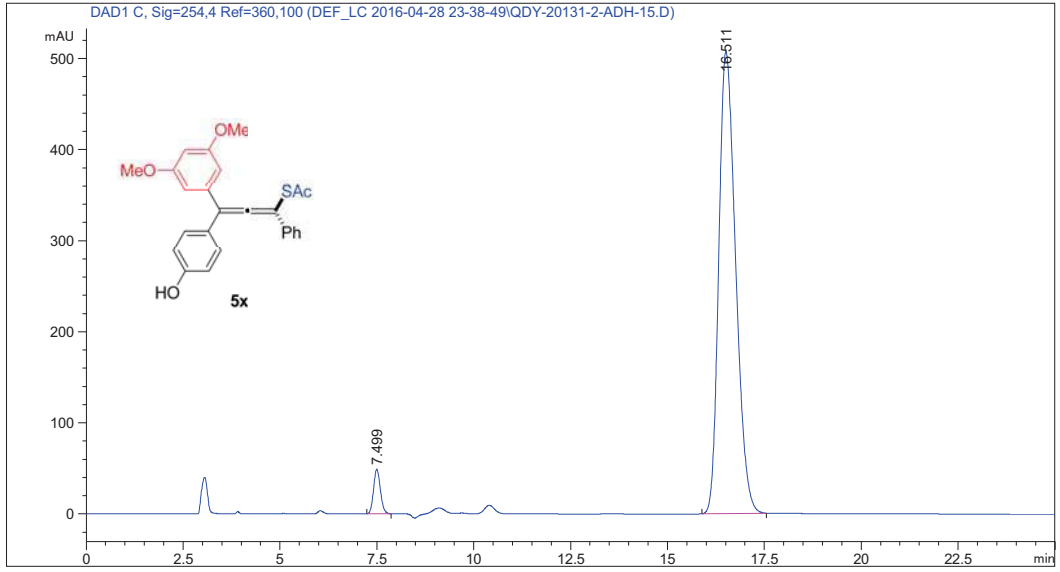
```
==========================================================================
Acq. Operator   :                                 Seq. Line :  27
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 4/28/2016 2:11:46 AM                  Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-27 16-08-08\AD-15-30.M
Last changed    : 4/28/2016 1:39:09 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 3:48:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.504 BB    0.1895 2587.34521  210.38361  50.0512
   2  16.675 BB    0.4629 2582.05249   86.33402  49.9488

Totals :                  5169.39771  296.71763
```

Data File C:\CHEM32\1\DATA\DEF_LC 2016-04-28 23-38-49\QDY-20131-2-ADH-15.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  7
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 4/29/2016 1:42:50 AM                  Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-04-28 23-38-49\AD-15-30.M
Last changed    : 4/29/2016 1:41:58 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 3:48:19 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

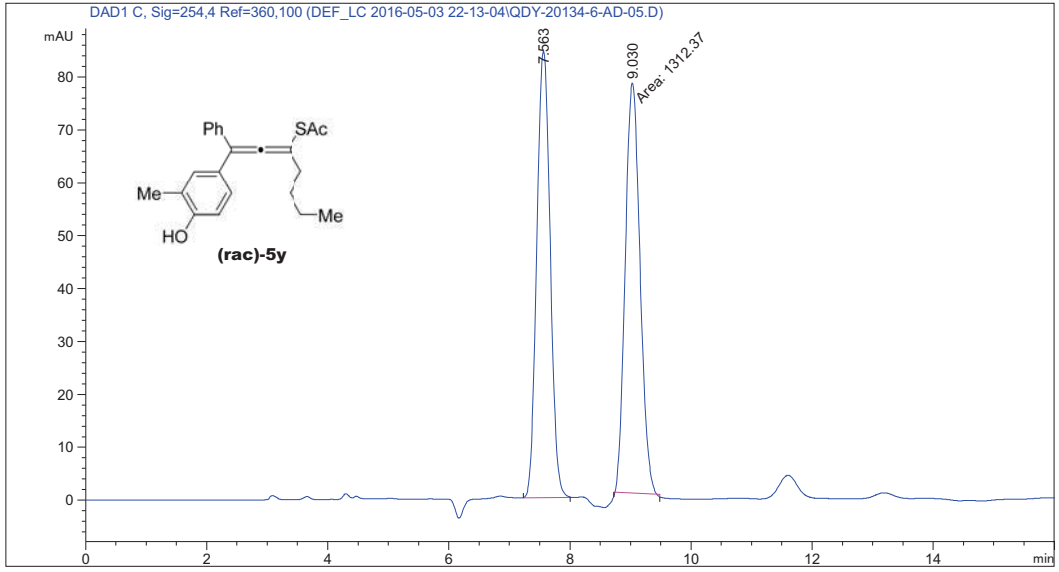



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.499 BB    0.1892  601.64008   49.02268   3.7736
   2  16.511 BB    0.4687 1.53417e4   507.45258  96.2264

Totals :                  1.59434e4   556.47525
```

Supplementary Figure 211. HPLC spectrum for 5x

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-03 22-13-04\QDY-20134-6-AD-05.D
Sample Name:

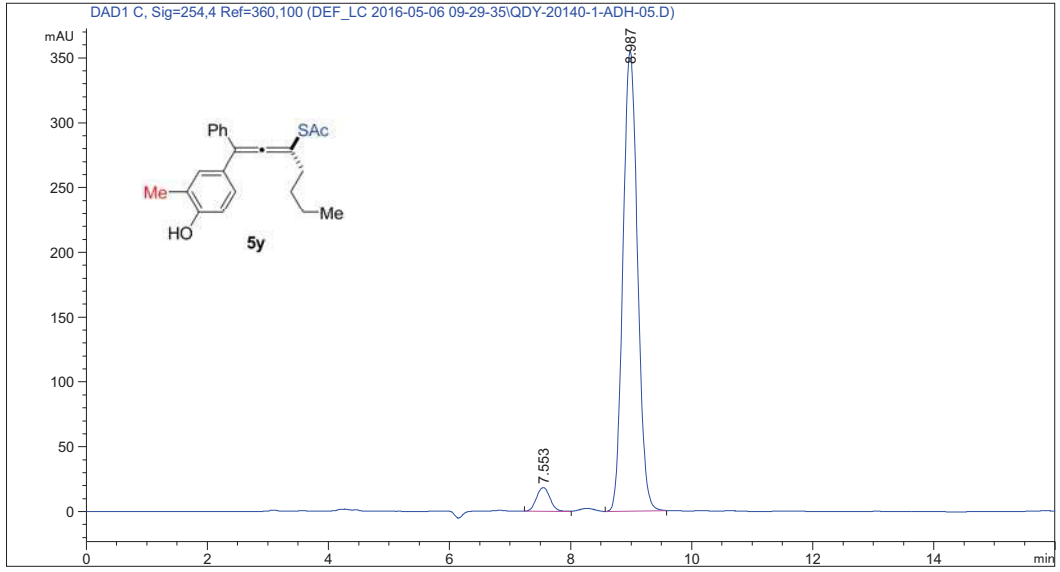
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   9
Acq. Instrument : Instrument 1                    Location : Vial 33
Injection Date  : 5/4/2016 12:44:24 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-05-03 22-13-04\AD-05-20.M
Last changed    : 5/4/2016 12:43:31 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:40:34 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.563 BB    0.2457 1298.89539   84.33715  49.7420
   2   9.030 MM    0.2822 1312.37000   77.51844  50.2580

Totals :                  2611.26538  161.85559
```


Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-06 09-29-35\QDY-20140-1-ADH-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :  23
Acq. Instrument : Instrument 1                    Location : Vial 31
Injection Date  : 5/6/2016 4:22:16 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEF_LC 2016-05-06 09-29-35\AD-05-20.M
Last changed    : 5/6/2016 4:21:24 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 8:40:34 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

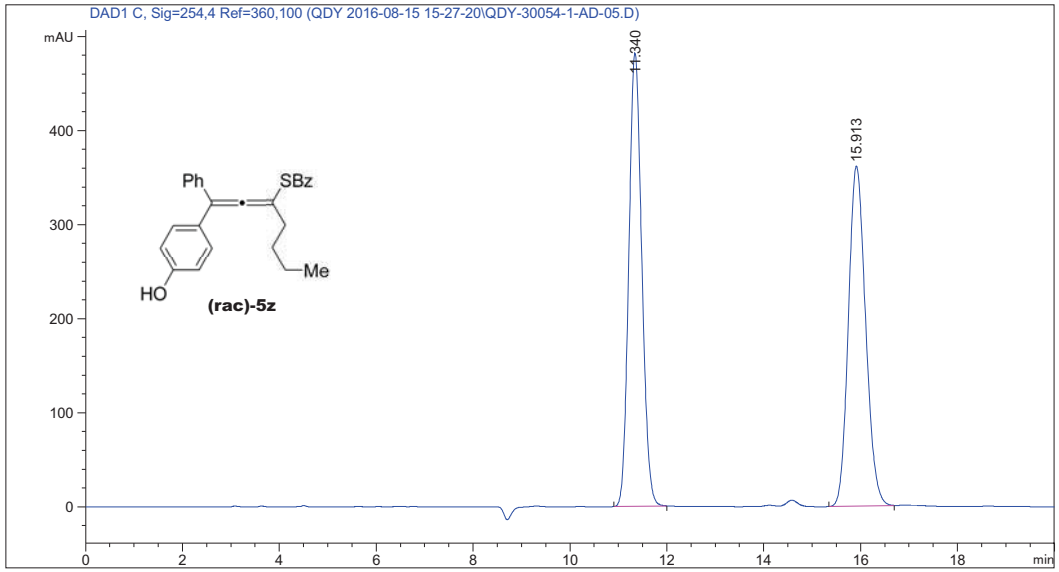



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.553 BB    0.2492  285.28354   18.37008   4.5947
   2   8.987 VB    0.2628 5923.73438  354.80585  95.4053

Totals :                  6209.01791  373.17592
```

Supplementary Figure 212. HPLC spectrum for 5y

Data File C:\CHEM32\1\DATA\QDY 2016-08-15 15-27-20\QDY-30054-1-AD-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 22
Acq. Instrument	: Instrument 1	Location	: Vial 33
Injection Date	: 8/15/2016 10:28:34 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\QDY 2016-08-15 15-27-20\AD-05-20.M		
Last changed	: 10/14/2015 3:51:32 PM		
Analysis Method	: C:\CHEM32\1\METHODS\OD-2.5-30.M		
Last changed	: 8/6/2016 3:47:03 PM		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

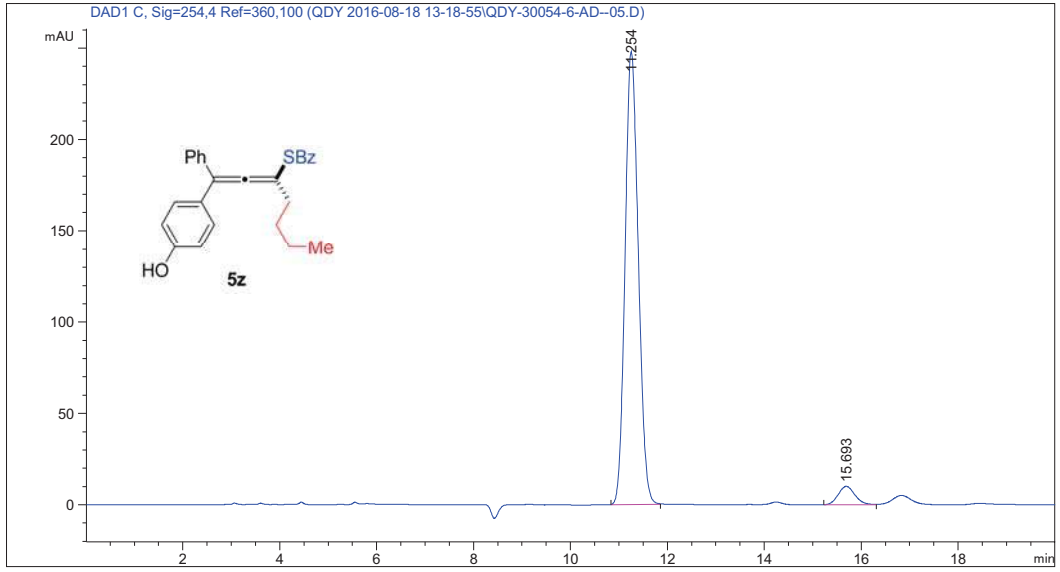
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.340	BB	0.2887	8956.43457	482.23285	50.1377
2	15.913	BB	0.3834	8907.23926	361.53693	49.8623
Totals :				1.78637e4	843.76978	

*** End of Report ***

Data File C:\CHEM32\1\DATA\QDY 2016-08-18 13-18-55\QDY-30054-6-AD--05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 29
Acq. Instrument	: Instrument 1	Location	: Vial 33
Injection Date	: 8/18/2016 10:32:40 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\QDY 2016-08-18 13-18-55\AD-05-20.M		
Last changed	: 10/14/2015 3:51:32 PM		
Analysis Method	: C:\CHEM32\1\METHODS\IC-15-30.M		
Last changed	: 8/18/2016 11:16:41 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

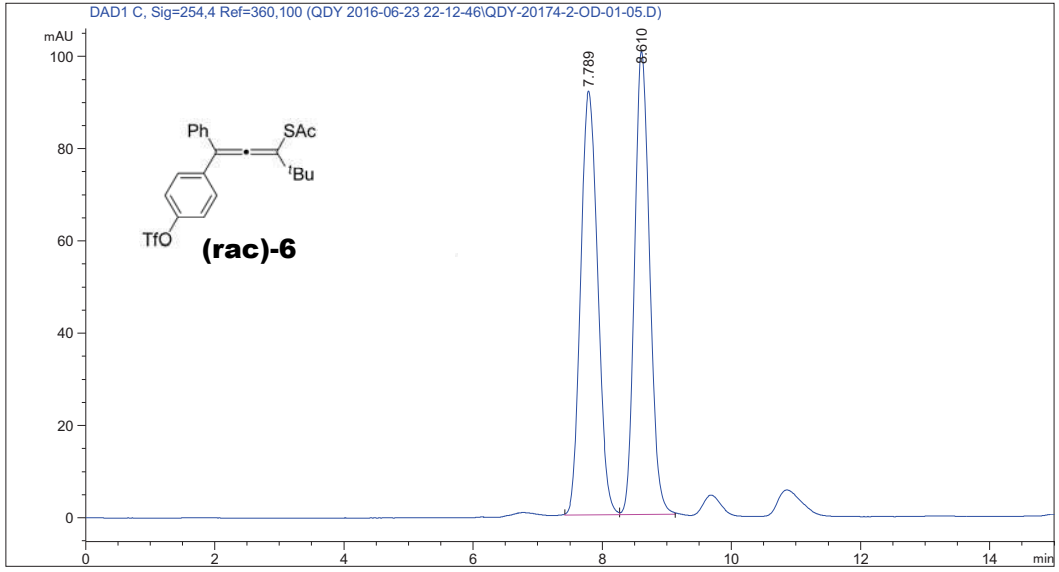
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.254	BB	0.2970	4696.32715	247.93793	95.0618
2	15.693	BB	0.3739	243.95964	10.09740	4.9382
Totals :				4940.28679	258.03533	

Supplementary Figure 213. HPLC spectrum for 5z

Data File C:\CHEM32\1\DATA\QDY 2016-06-23 22-12-46\QDY-20174-2-OD-01-05.D
Sample Name:

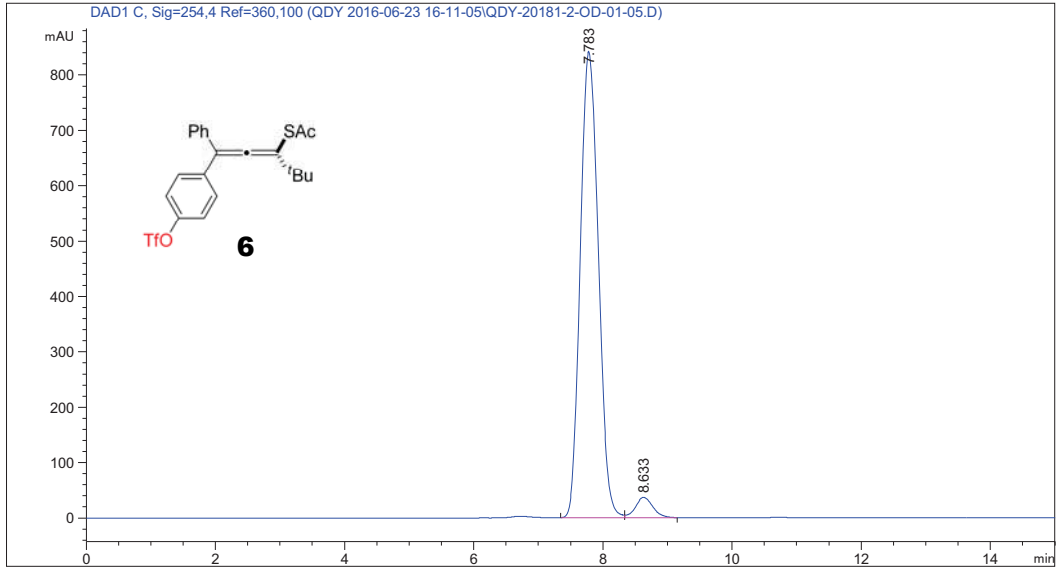
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   7
Acq. Instrument : Instrument 1                     Location : Vial 33
Injection Date  : 6/23/2016 11:51:23 PM                Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-06-23 22-12-46\OD-01-20-0.5.M
Last changed    : 6/23/2016 11:50:31 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-25-90.M
Last changed    : 6/24/2016 12:31:56 AM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.789 BV    0.2921 1686.98352   91.91465  50.7609
   2   8.610 VB    0.2561 1636.40552  100.41180  49.2391

Totals :                  3323.38904  192.32645
```


Data File C:\CHEM32\1\DATA\QDY 2016-06-23 16-11-05\QDY-20181-2-OD-01-05.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   4
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 6/23/2016 5:07:25 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-06-23 16-11-05\OD-01-20-0.5.M
Last changed    : 6/23/2016 5:06:32 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-25-90.M
Last changed    : 6/23/2016 11:05:26 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

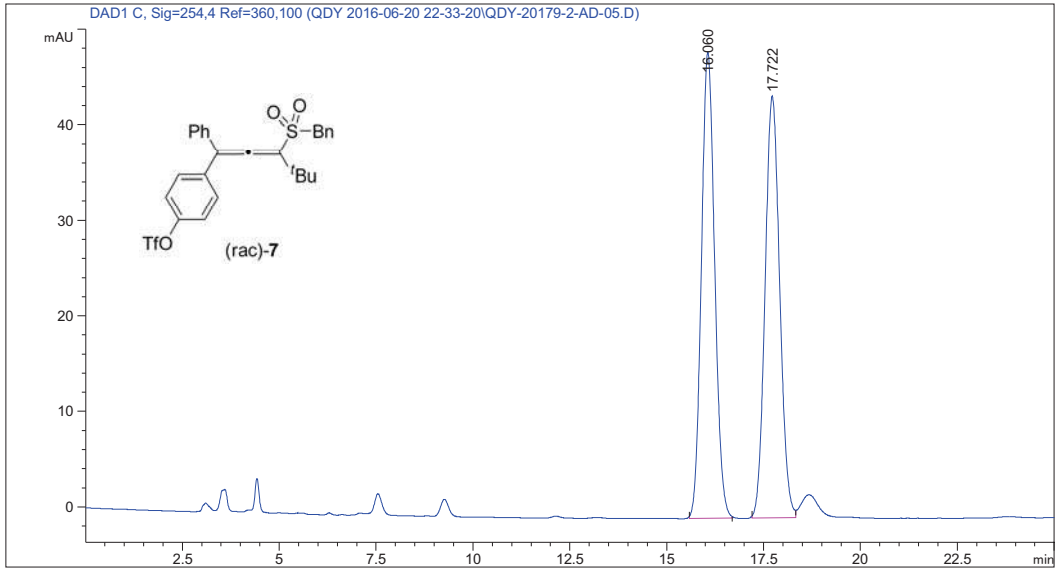



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.783 BV    0.3130 1.63889e4   842.21082  95.9034
   2   8.633 VB    0.2896  700.05945   36.86123   4.0966

Totals :                  1.70890e4   879.07204
```

Supplementary Figure 214. HPLC spectrum for 6

Data File C:\CHEM32\1\DATA\QDY 2016-06-20 22-33-20\QDY-20179-2-AD-05.D
Sample Name:

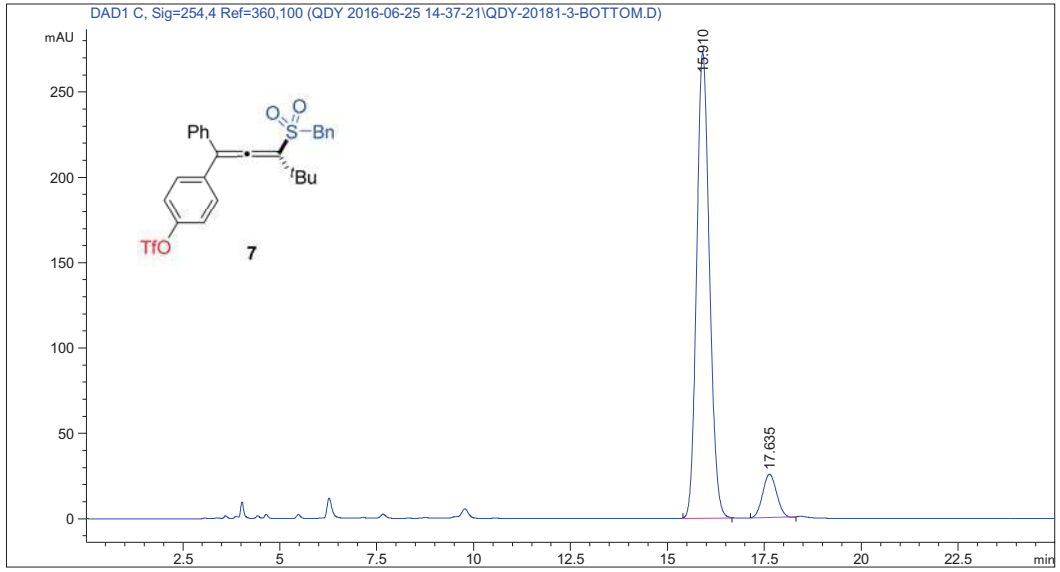
```
==========================================================================
Acq. Operator   :                                 Seq. Line :   2
Acq. Instrument : Instrument 1                     Location : Vial 33
Injection Date  : 6/20/2016 10:47:22 PM                Inj :   1
                                                  Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 10.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-06-20 22-33-20\AD-05-30.M
Last changed    : 6/20/2016 10:59:56 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 1:59:23 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.060 BB    0.3694 1159.83057   48.78622  50.0884
   2  17.722 BV    0.4073 1155.73547   44.15915  49.9116

Totals :                  2315.56604   92.94537
```

Data File C:\CHEM32\1\DATA\QDY 2016-06-25 14-37-21\QDY-20181-3-BOTTOM.D
Sample Name:

```
==========================================================================
Acq. Operator   :                                 Seq. Line :   3
Acq. Instrument : Instrument 1                     Location : Vial 32
Injection Date  : 6/25/2016 3:17:25 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2016-06-25 14-37-21\AD-05-30.M
Last changed    : 6/25/2016 2:57:48 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AD-01-10-2.0.M
Last changed    : 6/26/2016 1:59:23 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

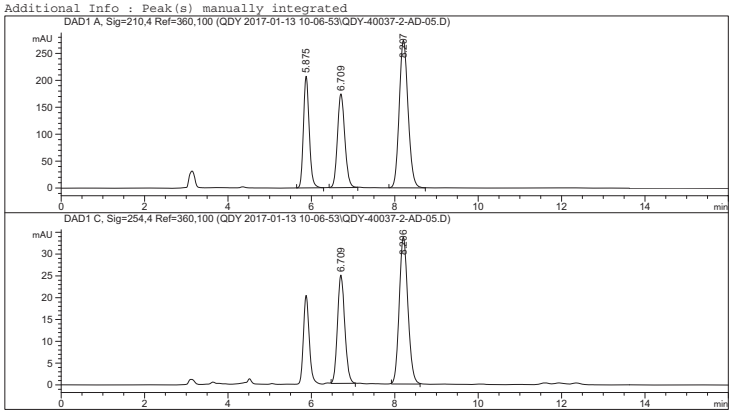



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.910 BB    0.3581 6275.77734  273.06549  90.8933
   2  17.635 BB    0.3896  628.77716   25.33145   9.1067

Totals :                  6904.55450  298.39694
```

Supplementary Figure 215. HPLC spectrum for 7

Data File C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\QDY-40037-2-AD-05.D
Sample Name:

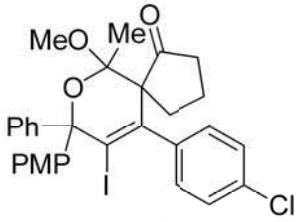
```
==================================================================
Acq. Operator   :                          Seq. Line :  12
Acq. Instrument : Instrument 1              Location : Vial 31
Injection Date  : 1/13/2017 2:11:06 PM          Inj :  1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\AD-05-20.M
Last changed    : 1/13/2017 2:10:15 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 1/15/2017 4:11:48 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.875 BB    0.1481 1992.61841  207.29070  24.4290
   2   6.709 BB    0.1912 2136.81909  174.07649  26.1968
   3   8.207 BB    0.2275 4027.34155  273.93179  49.3742

Totals :                  8156.77905  655.29898
```


Data File C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\QDY-40037-2-AD-05.D
Sample Name:

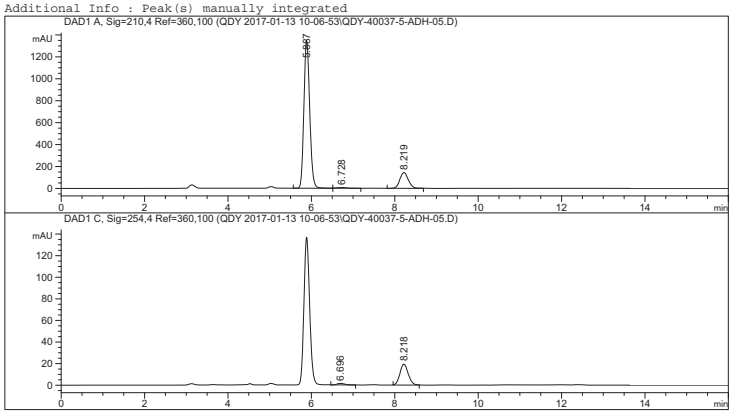
```
Signal 2: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.709 BB    0.1883  302.87158   24.83160  38.4254
   2   8.206 BB    0.2240  485.33475   33.69499  61.5746

Totals :                  788.20633   58.52659

==================================================================
                      *** End of Report ***
```



(rac)-8

Data File C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\QDY-40037-5-ADH-05.D
Sample Name:

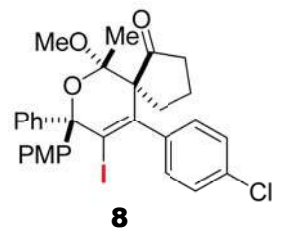
```
==================================================================
Acq. Operator   :                          Seq. Line :  32
Acq. Instrument : Instrument 1              Location : Vial 33
Injection Date  : 1/13/2017 8:13:46 PM          Inj :  1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\AD-05-20.M
Last changed    : 10/14/2015 3:51:32 PM
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 1/15/2017 4:11:48 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.887 BB    0.1505 1.29937e4  1347.09326  85.8763
   2   6.728 BB    0.2030  110.11534    8.28580   0.7278
   3   8.219 VB    0.2175 2026.89929  144.63719  13.3959

Totals :                  1.51307e4  1500.01626
```


Data File C:\CHEM32\1\DATA\QDY 2017-01-13 10-06-53\QDY-40037-5-ADH-05.D
Sample Name:

```
Signal 2: DAD1 C, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.696 BB    0.2009   17.99112    1.42895   6.2794
   2   8.218 BB    0.2132  268.51770   19.43753  93.7206

Totals :                  286.50882   20.86649

==================================================================
                      *** End of Report ***
```

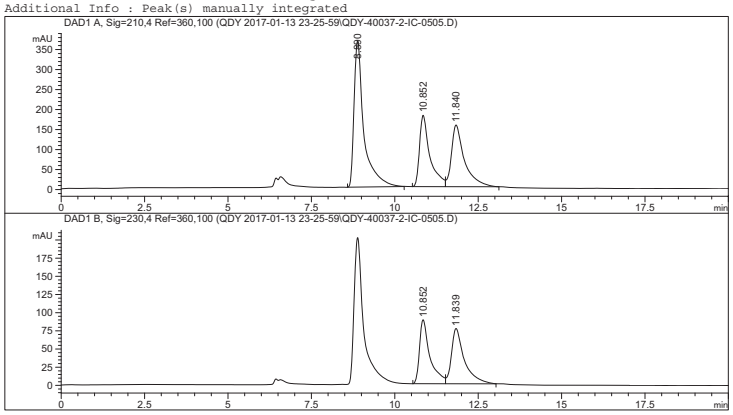


8

Supplementary Figure 216. HPLC spectrum for 8 (AD)

Data File C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\QDY-40037-2-IC-0505.D
Sample Name:

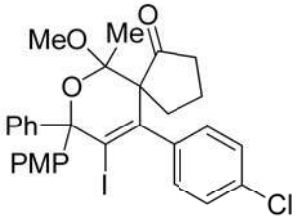
```
==================================================================
Acq. Operator   :                          Seq. Line :   2
Acq. Instrument : Instrument 1              Location : Vial 31
Injection Date  : 1/13/2017 11:40:24 PM          Inj :   1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\IC-05-25-05.M
Last changed    : 1/13/2017 11:22:04 PM
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 1/15/2017 4:07:21 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.890 BB    0.3003 7469.63574  365.63831  48.1861
   2  10.852 BV    0.3130 3867.48804  178.32393  24.9489
   3  11.840 VB    0.3852 4164.50977  154.16713  26.8650

Totals :                  1.55016e4   698.12936
```


Data File C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\QDY-40037-2-IC-0505.D
Sample Name:

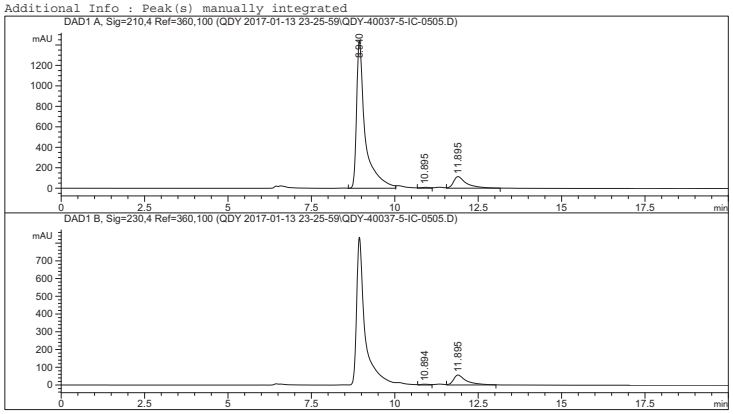
```
Signal 2: DAD1 B, Sig=230,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  10.852 BV    0.3127 1905.97717   87.99619  48.2916
   2  11.839 VB    0.3832 2040.83240   76.01822  51.7084

Totals :                  3946.80957  164.01440


==================================================================
                      *** End of Report ***
```



(rac)-8

Data File C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\QDY-40037-5-IC-0505.D
Sample Name:

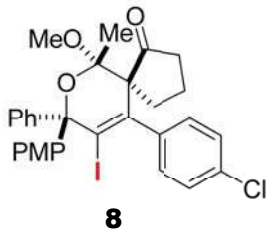
```
==================================================================
Acq. Operator   :                          Seq. Line :   3
Acq. Instrument : Instrument 1              Location : Vial 33
Injection Date  : 1/14/2017 12:06:40 AM          Inj :   1
                                          Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\IC-05-25-05.M
Last changed    : 1/13/2017 11:22:04 PM
Analysis Method : C:\CHEM32\1\METHODS\ETOH-IC-01-25-05.M
Last changed    : 1/15/2017 4:07:21 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.940 VV    0.2556 2.59539e4  1444.38599  88.9591
   2  10.895 VV    0.2718  167.76729    8.98649   0.5750
   3  11.895 VB    0.3894 3053.43042  113.65852  10.4659

Totals :                  2.91751e4  1567.03100
```


Data File C:\CHEM32\1\DATA\QDY 2017-01-13 23-25-59\QDY-40037-5-IC-0505.D
Sample Name:

```
Signal 2: DAD1 B, Sig=230,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  10.894 BV    0.2714   84.35708    4.52643   5.3166
   2  11.895 VB    0.3883 1502.31970   56.11365  94.6834

Totals :                  1586.67678   60.64008


==================================================================
                      *** End of Report ***
```

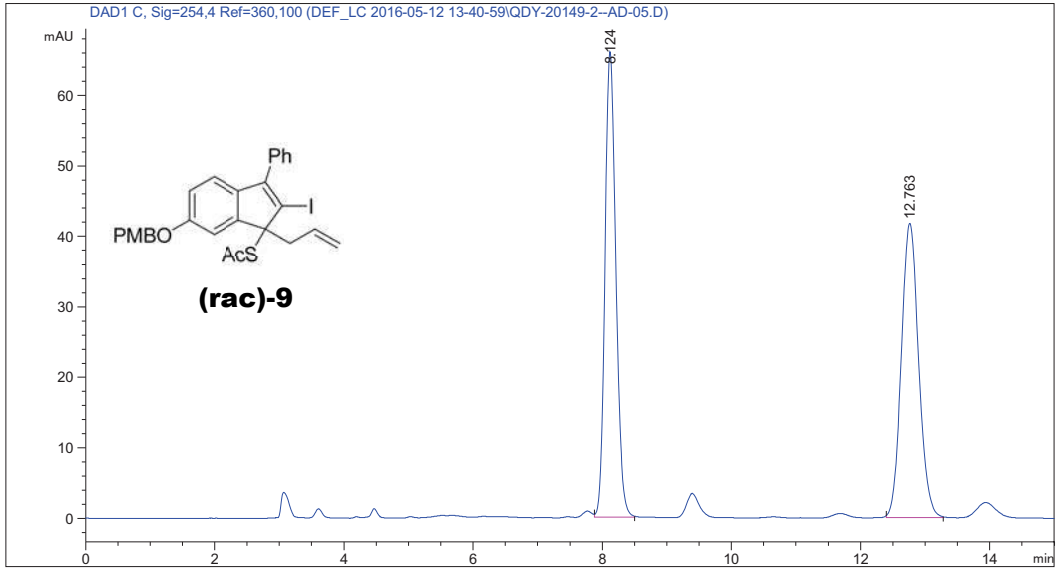


8

Supplementary Figure 217. HPLC spectrum for 8 (IC)

Data File C:\CHEM32\1\DATA\DEF_LC 2016-05-12 13-40-59\QDY-20149-2--AD-05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 38
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 5/13/2016 1:51:46 AM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEF_LC 2016-05-12 13-40-59\AD-05-20.M		
Last changed	: 10/14/2015 3:51:32 PM		
Analysis Method	: C:\CHEM32\1\METHODS\AD-01-10-2.0.M		
Last changed	: 6/26/2016 2:05:13 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

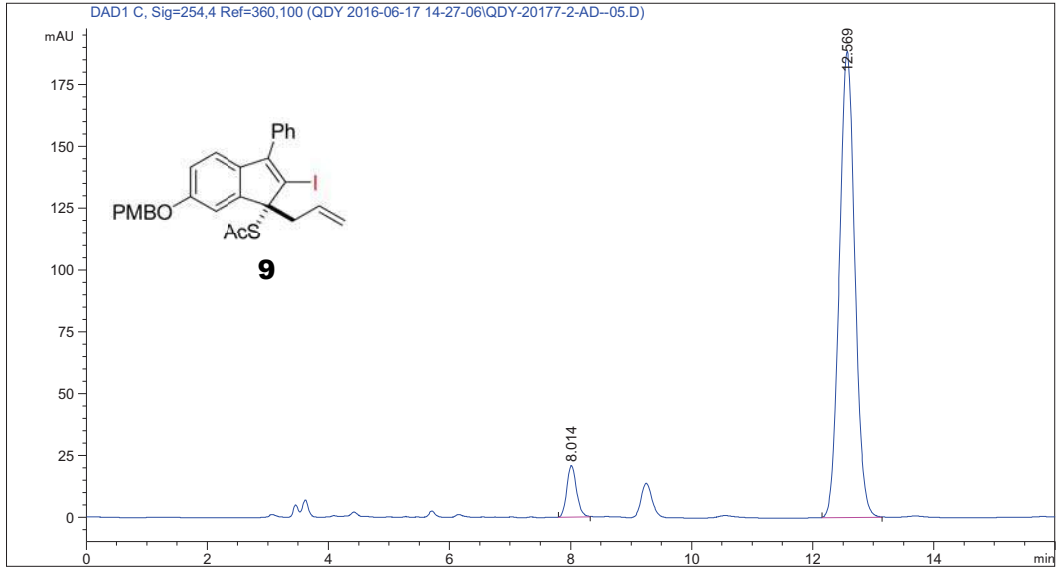
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.124	VB	0.1801	770.64471	66.04201	49.8542
2	12.763	BB	0.2865	775.15118	41.77250	50.1458
Totals :				1545.79590	107.81450	

Data File C:\CHEM32\1\DATA\QDY 2016-06-17 14-27-06\QDY-20177-2-AD--05.D
Sample Name:

Acq. Operator	:	Seq. Line	: 19
Acq. Instrument	: Instrument 1	Location	: Vial 32
Injection Date	: 6/17/2016 9:56:27 PM	Inj	: 1
		Inj Volume	: 5.000 µl
Acq. Method	: C:\CHEM32\1\DATA\QDY 2016-06-17 14-27-06\AD-05-30.M		
Last changed	: 6/17/2016 10:20:15 PM (modified after loading)		
Analysis Method	: C:\CHEM32\1\METHODS\IC-20-70.M		
Last changed	: 6/17/2016 10:40:01 PM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

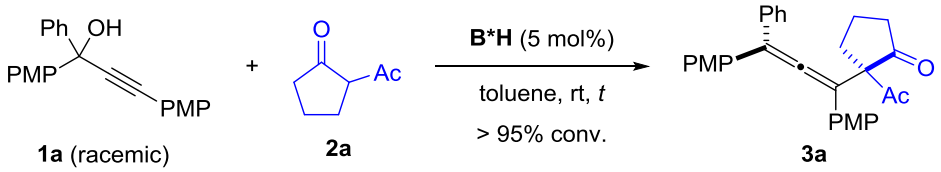
Use Multiplier & Dilution Factor with ISTDs

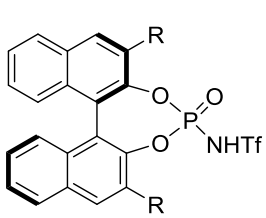
Signal 1: DAD1 C, Sig=254,4 Ref=360,100

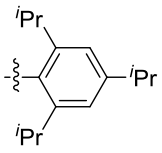
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.014	BB	0.1721	233.15421	20.90103	6.5009
2	12.569	BB	0.2756	3353.31763	188.42543	93.4991
Totals :				3586.47183	209.32646	

Supplementary Figure 218. HPLC spectrum for 9

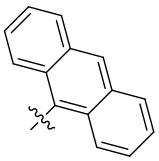
Supplementary Table 1. Evaluation of Brønsted acid in asymmetric allene synthesis of propargylic alcohol **1** and **2**.[a]



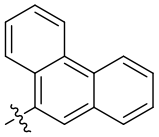




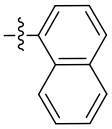
A1: 0.1 h,
80% *ee*, 2.5:1 d.r.



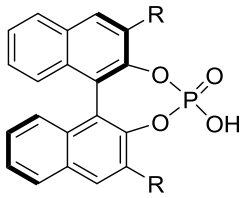
A3: 0.2 h
52% *ee*, 1.4:1 d.r.

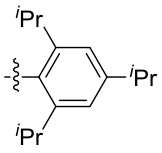


A4: 0.2 h
71% *ee*, 1.8:1 d.r.

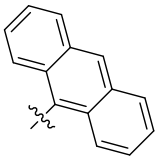


A5: 0.3 h
12% *ee*, 1.7:1 d.r.

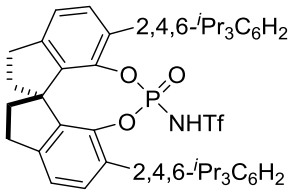




A2: 0.1-12 h
no reaction



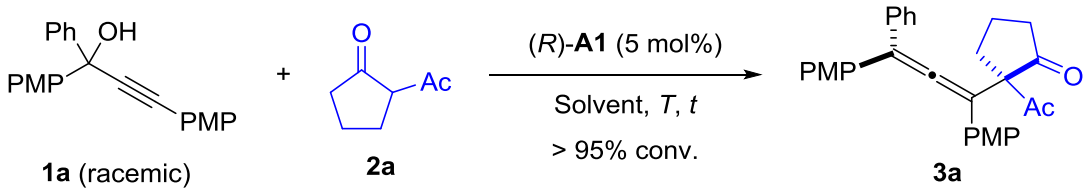
A7: 0.1-12 h
no reaction



B1: 0.3 h
15% *ee*, 1.7:1 d.r.

[a] Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), **B*H** (2.5 μmol), toluene (1.0 mL). The d.r. was determined by ^{1}H NMR analysis. The *ee* was determined by HPLC with a chiral stationary phase. PMP = (*p*-MeO)C$_6$H$_4$.

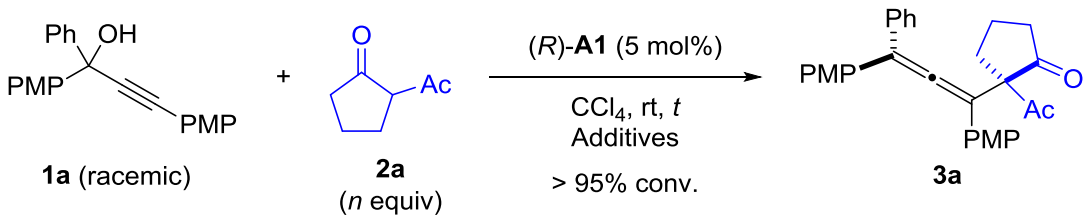
Supplementary Table 2. Solvent and temperature effect on asymmetric allene synthesis of propargylic alcohol **1** and **2**.[a]



Entry	Solvent	*T* [°C]	*t* [h]	d.r.[b]	*ee* [%][c]
1	toluene	25	0.1	2.5:1	80
2	xylene	25	0.2	2.5:1	73
3	PhF	25	0.2	2.4:1	84
4	CH_2Cl_2	25	0.1	1.6:1	73
5	DCE	25	0.1	1.4:1	72
6	$CHCl_3$	25	0.5	1.6:1	76
7	CCl_4	25	0.1	4.6:1	85
8	Et_2O	25	12	2.8:1	84
9	MTBE	25	12	2.0:1	81
10	CH_3CN	25	0.5	1:1	7
11	hexane	25	3	2.1:1	65
12	cyclohexane	25	1	3.5:1	80
13	hexane/CCl_4 (1:1)	25	0.3	4.3:1	83
14	cyclohexane/CCl_4 (1:1)	25	0.3	4.6:1	84
15	cyclohexane/CCl_4 (3:1)	25	0.5	4.6:1	83
16	cyclohexane/CCl_4 (1:3)	25	0.2	4.6:1	84
17	CCl_4	0	1.5	5.3:1	88
18	CCl_4	−20	3	6.1:1	90[d]

[a] Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), **A1** (2.5 μmol), solvent (1.0 mL). [b] Determined by ^{1}H NMR analysis. [c] Determined by HPLC with a chiral stationary phase. [d] **3a** was obtained in 92% isolated yield. PMP = 4-$MeOC_6H_4$.

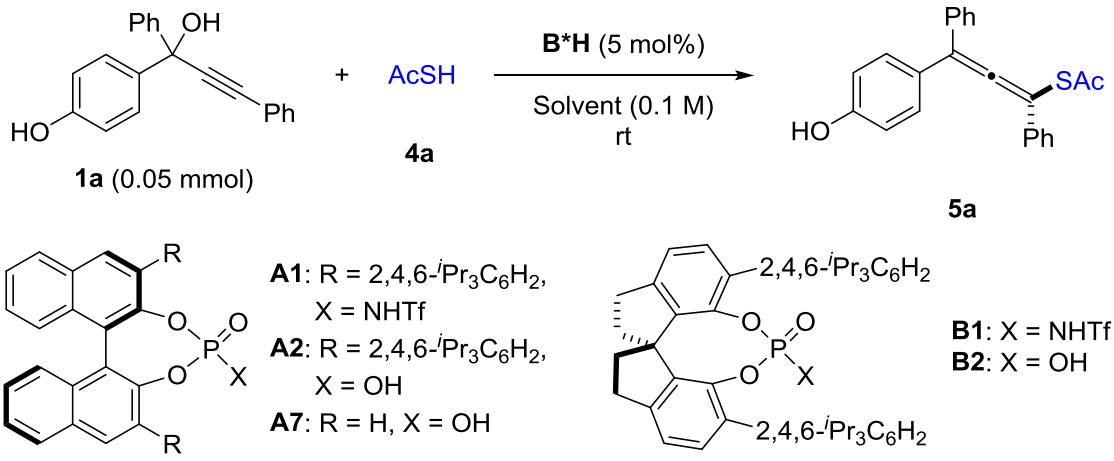
Supplementary Table 3. Other parameters on asymmetric allene synthesis of propargylic alcohol **1** and **2**.[a]



Entry	*n*	Additives	*t* [h]	d.r.[b]	*ee* [%][c]
1	1.1	-	0.3	4.2:1	84
2	1.5	-	0.1	4.6:1	85
3	2.0	-	0.1	4.3:1	85
4[d]	1.5	-	0.1	4.4:1	85
5	1.5	4Å MS (15 mg)	24	4.7:1	85
6	1.5	3Å MS (15 mg)	12	4.7:1	85
7	1.5	$MgSO_4$ (15 mg)	0.1	4.6:1	85

[a] Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), **A1** (2.5 μmol), solvent (1.0 mL). [b] Determined by ^1H NMR analysis. [c] Determined by HPLC with a chiral stationary phase. [d] c = 0.1 M. PMP = 4-$MeOC_6H_4$.

Supplementary Table 4. Reaction optimization on the asymmetric allene synthesis of propargylic alcohol **1** and **4**.

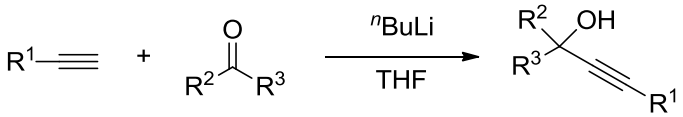


Entry	**B*H**	Solvent	*t* [h]	Yield [%][a]	*ee* [%][a]
1	(*R*)-**A1**	CCl_4	0.5	87	46
2	(*R*)-**A2**	CCl_4	18	85	60
3	(*R*)-**A7**	CCl_4	1.5	65	20
4	(*S*)-**B1**	CCl_4	0.3	83	85
5	(*S*)-**B1**	DCM	0.3	73	81
6	(*S*)-**B2**	DCM	24	51	75
7	(*R*)-**A1**	DCM	0.5	90	43
8	(*S*)-**B1**	Et_2O	12	<5	-
9	(*S*)-**B1**	toluene	1	93	85
10[b]	(*S*)-**B1**	toluene	5	87	89
11[c]	(*S*)-**B1**	toluene	12	<5	-
12[b,d]	(*S*)-**B1**	toluene	6	85	90
13[b,d,e]	(*S*)-**B1**	toluene	12	84	94

[a] Yield was determined by ^{1}H NMR analysis of the crude mixture. Ee was determined by HPLC with a chiral stationary phase. [b] Run with 3Å molecular sieves (MS, 15 mg). [c] Run 4Å MS (15 mg). [d] c = 0.05 M. [e] Run at –5 °C.

Supplementary Methods

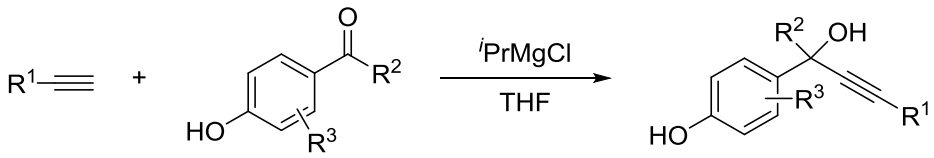
General Procedure A



S1 **S2** **1**

At –78 °C under N_2, to an oven-dried flask charged with a solution of the terminal alkyne **S1** (7.5 mmol, 1.5 equiv) in dry THF (10 mL) was added ^{n}BuLi (7.7 mmol, 3.2 mL, 2.4 M in hexane) dropwise. The reaction was stirred for 1 h at the same temperature and then a solution of the corresponding ketone **S2** (5.0 mmol, 1.0 equiv) in THF (5 mL) was added via syringe. The reaction mixture was then gradually warmed up to room temperature. Upon completion (~3 h), the reaction mixture was cooled to 0 °C and a saturated aqueous NH_4Cl solution (10 mL) was added dropwise. The organic layer was separated. The aqueous layer was extracted with ethyl acetate (3×20 mL). The combined organic layers were washed with brine (50 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel chromatography to afford the pure alcohol **1**.

General Procedure B

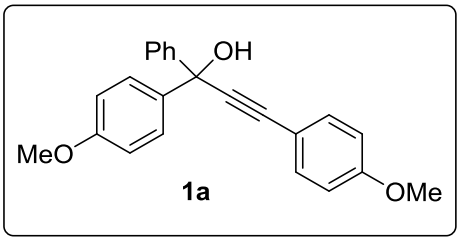


S1 **S3** **1**

Under N_2, to an oven-dried flask charged with a solution of the alkyne **S1** (12.5 mmol, 2.5 equiv) in dry THF (30 mL) was added ^{i}PrMgCl (12.6 mmol, 6.3 mL, 2.0 M in THF) dropwise at 0 °C. After addition, the mixture was warmed to room temperature and stirred at the same temperature for 1 hour. Next, the reaction mixture was cooled to 0 °C, and a solution of the ketone **S3** (5.0 mmol) in THF (5 mL)

was added dropwise. The reaction mixture was then warmed to 60 °C and stirred at the same temperature. The reaction progress was monitored by thin layer chromatography. Upon completion (6-12 h), the reaction mixture was cooled to 0 °C and slowly treated with a saturated aqueous NH_4Cl solution (10 mL). The layers were separated. The aqueous layer was extracted with EtOAc (3×30 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography to afford the pure alcohol **1**.

1,3-bis(4-Methoxyphenyl)-1-phenylprop-2-yn-1-ol (1a) was prepared as pale yellow oil according to the General Procedure A (purification by flash column chromatography: 10→20% EtOAc in hexanes, 96% yield).



1a

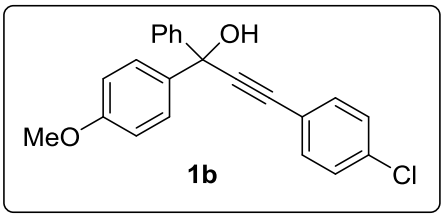
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 7.2 Hz, 2H), 7.59 (d, J = 8.8 Hz, 2H), 7.45 (d, J = 8.8 Hz, 2H), 7.36 (t, J = 7.2 Hz, 2H), 7.28 (t, J = 7.2 Hz, 1H), 6.88 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 3.80 (s, 3H), 2.97 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 159.0, 145.4, 137.5, 133.2, 128.2, 127.5, 127.4, 126.0, 114.5, 113.9, 113.5, 90.6, 86.9, 74.5, 55.3, 55.2 ppm.

IR (thin film) 3450, 2951, 1603, 1507, 1238, 1169, 1021, 827, 661 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{20}O_3$ [M^+]: 344.1412, Found: 344.1420.

3-(4-Chlorophenyl)-1-(4-methoxyphenyl)-1-phenylprop-2-yn-1-ol (1b) as colourless oil according to the General Procedure A (purification by flash column chromatography: 10 →20% EtOAc in hexanes, 81% yield).



1b

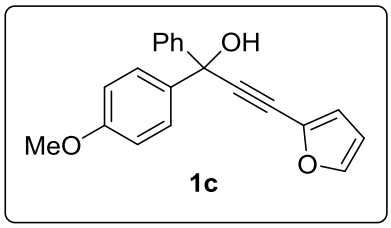
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 7.2 Hz, 2H), 7.57 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.37 (t, J = 7.2 Hz, 2H), 7.32-7.27 (m, 3H), 6.88 (d, J = 8.8 Hz, 2H), 3.80 (s, 3H), 2.95 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 145.0, 137.1, 134.6, 133.0, 128.6, 128.3, 127.7, 127.4, 126.0, 120.9, 113.6, 92.9, 85.8, 74.5, 55.2 ppm.

IR (thin film) 3440, 2933, 1607, 1488, 1246, 1169, 1030, 827, 697 cm^{-1}.

HRMS (CI+) Calcd for $C_{22}H_{17}{}^{35}ClO_2$ [M^+]: 348.0917, Found: 348.0926.

3-(Furan-2-yl)-1-(4-methoxyphenyl)-1-phenylprop-2-yn-1-ol (1c) as brown oil according to the General Procedure A (purification by flash column chromatography: 10→20% EtOAc in hexanes, 81% yield).



1c

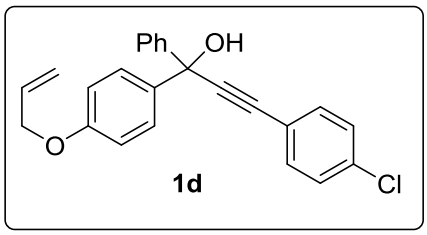
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 8.0 Hz, 2H), 7.55 (d, J = 8.8 Hz, 2H), 7.41 (s, 1H), 7.36 (t, J = 7.2 Hz, 2H), 7.30 (d, J = 7.2 Hz, 1H), 6.87 (d, J = 8.8 Hz, 2H), 6.65-6.64 (m, 1H), 6.42-6.39 (m, 1H), 3.80 (s, 3H), 2.94 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 144.5, 143.8, 136.7, 136.4, 128.3, 127.8, 127.4, 126.0, 115.8, 113.6, 110.9, 95.9, 77.3, 74.5, 55.2 ppm.

IR (thin film) 3418, 2934, 1606, 1508, 1247, 1171, 989, 829, 704 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{16}O_3$ [M^+]: 304.1099, Found: 304.1112.

1-(4-(Allyloxy)phenyl)-3-(4-chlorophenyl)-1-phenylprop-2-yn-1-ol (1d) as colourless oil according to the General Procedure A (purification by flash column chromatography: 10→20% EtOAc in hexanes, 85% yield).



1d

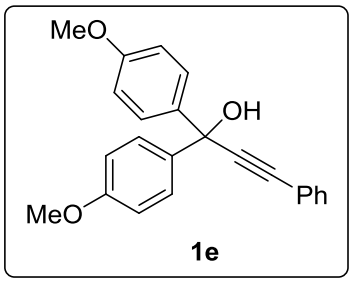
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 7.2 Hz, 2H), 7.56 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.37 (t, J = 7.2 Hz, 2H), 7.33-7.27 (m, 3H), 6.88 (d, J = 8.8 Hz, 2H), 6.11-5.99 (m, 1H), 5.41 (d, J = 17.6 Hz, 1H), 5.30 (d, J = 10.4 Hz, 1H), 4.53 (d, J = 5.2 Hz, 2H), 2.92 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 144.9, 137.2, 134.7, 133.1, 133.0, 128.6, 128.3, 127.7, 127.4, 126.0, 120.9, 117.7, 114.4, 92.9, 85.8, 74.5, 68.8 ppm.

IR (thin film) 3429, 2960, 1606, 1488, 1243, 1170, 1091, 986, 827, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{24}H_{19}{}^{35}ClO_2$ [M^+]: 374.1074, Found: 374.1080.

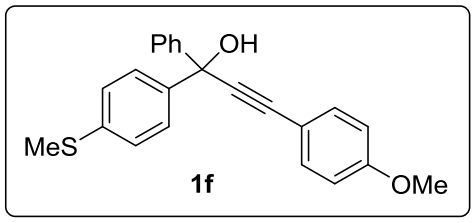
1,3-bis(4-Methoxyphenyl)-1-phenylprop-2-yn-1-ol (1a) was prepared as pale yellow oil according to the literature procedure [1] (purification by flash column chromatography: 10→20% EtOAc in hexanes, 96% yield).



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 8.8 Hz, 4H), 7.53-7.50 (m, 2H), 7.35-7.31 (m, 3H), 6.89 (d, J = 8.8 Hz, 4H), 3.80 (s, 6H), 2.98 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 137.5, 131.7, 128.5, 128.3, 127.3, 122.5, 113.5, 92.1, 86.8, 74.1, 55.2 ppm.

3-(4-Methoxyphenyl)-1-(4-(methylthio)phenyl)-1-phenylprop-2-yn-1-ol (1f) as brown oil according to the General Procedure A (purification by flash column chromatography: 10→20% EtOAc in hexanes, 87% yield).



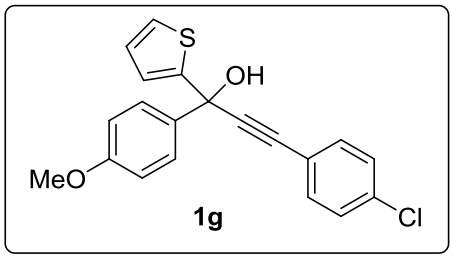
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 7.2 Hz, 2H), 7.60 (d, J = 8.8 Hz, 2H), 7.45 (d, J = 8.8 Hz, 2H), 7.36 (t, J = 7.2 Hz, 2H), 7.29 (t, J = 7.2 Hz, 1H), 7.23 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 2.99 (br s, 1H), 3.47 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 145.0, 142.2, 137.9, 133.2, 128.2, 127.7, 126.6, 126.2, 126.0, 114.4, 113.9, 90.2, 87.2, 74.5, 55.3, 15.2 ppm.

IR (thin film) 3432, 2919, 1604, 1508, 1245, 1170, 1028, 829, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{20}O_2S$ [M^+]: 360.1184, Found: 360.1196.

3-(4-Chlorophenyl)-1-(4-methoxyphenyl)-1-(thiophen-2-yl)prop-2-yn-1-ol (1g) as brown oil according to the General Procedure A (purification by flash column chromatography: 10→20% EtOAc in hexanes, 70% yield).



1g

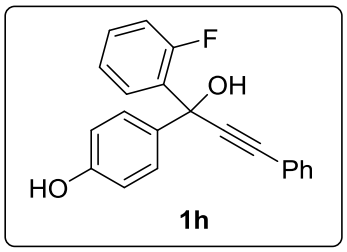
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.31 (d, J = 8.8 Hz, 2H), 7.29-7.26 (m, 1H), 7.12-1.09 (m, 1H), 6.96-6.92 (m, 1H), 6.91 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 3.17 (br s, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 150.3, 136.3, 134.8, 133.0, 128.6, 127.1, 126.5, 125.9, 125.3, 120.6, 113.6, 92.2, 85.2, 71.9, 55.3 ppm.

IR (thin film) 3418, 2933, 1606, 1507, 1247, 1171, 1031, 826, 702 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{15}{}^{35}ClO_2S$ [M^+]: 354.0481, Found: 354.0491.

4-(1-(2-Fluorophenyl)-1-hydroxy-3-phenylprop-2-yn-1-yl)phenol (1h) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 93% yield).



1h

^{1}H NMR (400 MHz, acetone-d_6) δ 8.49 (br s, 1H), 7.99 (t, J = 8.0 Hz, 1H), 7.51-7.48 (m, 4H), 7.40-7.31 (m, 4H), 7.25 (t, J = 7.6 Hz, 1H), 7.06-6.98 (m, 1H), 6.80 (d, J = 8.8 Hz, 2H), 5.69(br s, 1H) ppm.

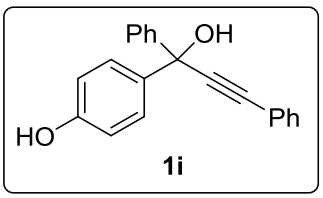
^{13}C NMR (100 MHz, acetone-d_6) δ 160.3 (d, $J_{C\text{-}F}$ = 247 Hz), 157.5, 136.5, 134.1 (d, $J_{C\text{-}F}$ = 11 Hz), 132.0, 130.0 (d, $J_{C\text{-}F}$ = 8 Hz), 129.1, 128.2 (2C), 127.6 (d, $J_{C\text{-}F}$ = 3 Hz), 124.2 (d, $J_{C\text{-}F}$ = 4 Hz), 123.5, 116.5 (d, $J_{C\text{-}F}$ = 22 Hz), 115.1, 92.2, 85.7, 71.3 ppm.

^{19}F NMR (376 MHz, acetone-d_6) δ –112.1 ppm.

IR (thin film) 3383, 1613, 1512, 1453, 1224, 1170, 989, 837, 757 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{15}FO_2$ [M^+]: 318.1056, Found: 318.1072.

4-(1-Hydroxy-1,3-diphenylprop-2-yn-1-yl)phenol (1i) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 87% yield).



1i

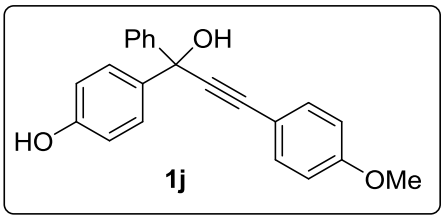
^{1}H NMR (400 MHz, acetone-d_6) δ 8.34 (br s, 1H), 7.70 (d, J = 8.4 Hz, 2H), 7.58-7.50 (m, 4H), 7.41-7.37 (m, 3H), 7.34 (t, J = 7.2 Hz, 2H), 7.24 (t, J = 7.2 Hz, 1H), 6.81 (d, J = 8.8 Hz, 2H), 5.61 (br s, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.2, 147.4, 138.0, 132.0, 129.1, 129.0, 128.5, 128.0, 127.6, 126.5, 123.6, 115.2, 93.9, 86.2, 74.2 ppm.

IR (thin film) 3382, 1598, 1511, 1446, 1236, 1170, 986, 836, 756 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{16}O_2$ [M$^+$]: 300.1150, Found: 300.1152.

4-(1-Hydroxy-3-(4-methoxyphenyl)-1-phenylprop-2-yn-1-yl)phenol (1j) was prepared as red solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 85% yield).



1j

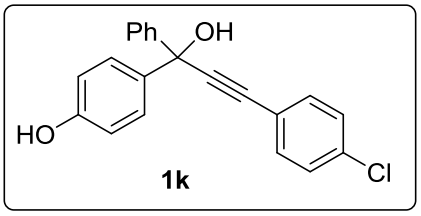
^{1}H NMR (400 MHz, acetone-d_6) δ 8.33 (br s, 1H), 7.69 (d, J = 7.2 Hz, 2H), 7.51 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 8.8 Hz, 2H), 7.32 (t, J = 7.2 Hz, 2H), 7.23 (t, J = 7.2 Hz, 1H), 6.94 (d, J = 7.2 Hz, 2H), 6.80 (d, J = 8.8 Hz, 2H), 5.51 (br s, 1H), 3.81 (s, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 160.6, 157.2, 147.6, 138.3, 133.5, 128.4, 128.0, 127.5, 126.6, 115.5, 115.2, 114.7, 92.4, 86.2, 74.2, 55.4 ppm.

IR (thin film) 3283, 2976, 1606, 1510, 1448, 1249, 1170, 1043, 833, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{22}H_{18}O_3$ [M$^+$]: 330.1256, Found: 330.1255.

4-(3-(4-Chlorophenyl)-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1k) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 95% yield).



1k

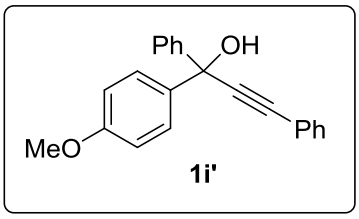
^{1}H NMR (400 MHz, acetone-d_6) δ 8.29 (br s, 1H), 7.68 (d, J = 8.0 Hz, 2H), 7.51 (t, J = 8.4 Hz, 4H), 7.43 (d, J = 8.0 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 7.24 (d, J = 7.6 Hz, 1H), 6.80 (d, J = 7.6 Hz, 2H), 5.58 (br s, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.6, 147.5, 138.1, 134.9, 133.9, 129.7, 128.8, 128.3, 128.0, 126.7, 122.7, 115.6, 95.4, 85.3, 74.6 ppm.

IR (thin film) 3289, 1511, 1490, 1170, 982, 830, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{15}{}^{35}ClO_2$ [M$^+$]: 334.0761, Found: 334.0762.

1-(4-Methoxyphenyl)-1,3-diphenylprop-2-yn-1-ol (1i') was prepared as yellow solid in 92% yield according to the literature procedure[2] (purification by flash column chromatography: 10→20% EtOAc in hexanes).

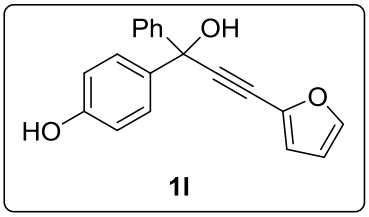


1i'

^{1}H NMR (400 MHz, acetone-d_6) δ 7.71 (d, J = 7.6 Hz, 2H), 7.62 (d, J = 8.8, 2H), 7.54–7.50 (m, 2H), 7.41-7.31 (m, 5H), 7.25 (t, J = 7.6 Hz, 1H), 6.90 (d, J = 8.8 Hz, 2H), 5.65 (s, 1H), 3.77 (s, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 159.7, 147.3, 139.2, 132.1, 129.2, 128.6 (2C), 128.0, 127.7, 126.6, 123.6, 113.9, 93.9, 86.4, 74.3, 55.3 ppm.

4-(3-(Furan-2-yl)-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1l) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 75% yield).



1l

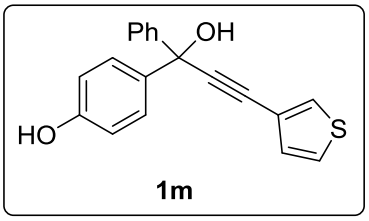
^{1}H NMR (400 MHz, acetone-d_6) δ 8.38 (br s, 1H), 7.65 (d, J = 7.2 Hz, 2H), 7.61-7.59 (m, 1H), 7.47 (d, J = 8.8 Hz, 2H), 7.34 (t, J = 7.6 Hz, 2H), 7.25 (t, J = 7.2 Hz, 1H), 6.81 (d, J = 8.8 Hz, 2H), 6.74-6.72 (m, 1H), 6.52-6.49 (m, 1H), 5.72 (br s, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.3, 146.7, 144.7, 137.3, 137.1, 128.5, 127.9, 127.7, 126.5, 115.9, 115.3, 111.6, 97.7, 76.6, 74.3 ppm.

IR (thin film) 3330, 1613, 1511, 1448, 1213, 1171, 1050, 834, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{19}H_{14}O_3$ [M$^+$]: 290.0943, Found: 290.0952.

4-(1-Hydroxy-1-phenyl-3-(thiophen-3-yl)prop-2-yn-1-yl)phenol (1m) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→ 30% EtOAc in hexanes, 83% yield).



1m

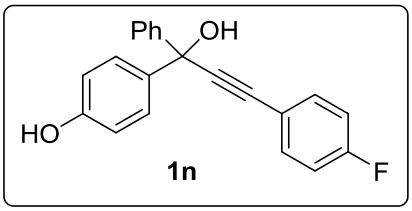
^{1}H NMR (400 MHz, acetone-d_6) δ 8.32 (br s, 1H), 7.71-7.65 (m, 3H), 7.53-7.47 (m, 3H), 7.32 (t, J = 7.6 Hz, 2H), 7.25-7.18 (m, 2H), 6.79 (d, J = 8.4 Hz, 2H), 5.56 (br s, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.3, 147.4, 138.1, 130.3, 129.6, 128.5, 128.1, 127.6, 126.7, 126.6, 122.6, 115.2, 93.3, 81.6, 74.3 ppm.

IR (thin film) 3379, 2884, 1613, 1511, 1358, 1167, 1099, 784, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{19}H_{14}O_2S$ [M$^+$]: 306.0715, Found: 306.0718.

4-(3-(4-Fluorophenyl)-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1n) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→ 30% EtOAc in hexanes, 82% yield).



1n

^{1}H NMR (400 MHz, acetone-d_6) δ 8.35 (br s, 1H), 7.69 (d, J = 7.2 Hz, 2H), 7.59-7.50 (m, 4H), 7.33 (t, J = 7.2 Hz, 2H), 7.23 (t, J = 7.2 Hz, 1H), 7.17 (t, J = 8.8 Hz, 2H), 6.81 (d, J = 8.8 Hz, 2H), 5.60 (br s, 1H) ppm.

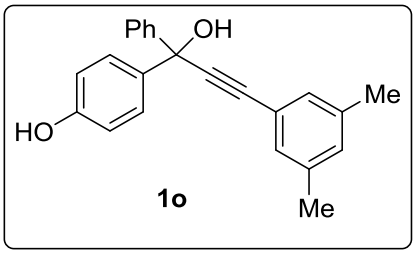
^{13}C NMR (100 MHz, acetone-d_6) δ 163.1 (d, $J_{C\text{-}F}$ = 246 Hz), 157.2, 147.2, 137.9, 134.1 (d, $J_{C\text{-}F}$ = 8.5 Hz), 128.4, 127.9, 127.5, 126.5, 119.8 (d, $J_{C\text{-}F}$ = 3.5 Hz), 116.2 (d, $J_{C\text{-}F}$ = 22.3 Hz), 115.2, 93.7, 85.0, 74.2 ppm.

^{19}F NMR (376 MHz, acetone-d_6) δ -112.4 ppm.

IR (thin film) 3288, 2978, 1600, 1509, 1449, 1230, 1170, 987, 835, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{15}FO_2$ [M$^+$]: 318.1056, Found: 318.1058.

4-(3-(3,5-Dimethylphenyl)-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1o) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 87% yield).



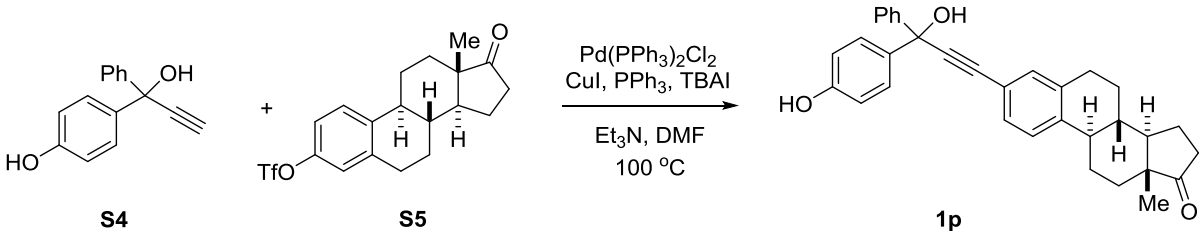
1o

^{1}H NMR (400 MHz, acetone-d_6) δ 8.32 (br s, 1H), 7.68 (d, J = 7.2 Hz, 2H), 7.50 (d, J = 8.8 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 7.23 (t, J = 7.6 Hz, 1H), 7.12 (s, 2H), 7.02 (s, 1H), 6.80 (d, J = 8.4 Hz, 2H), 5.54 (br s, 1H), 2.28 (s, 6H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.3, 147.6, 138.7, 138.2, 130.8, 129.7, 128.5, 128.1, 127.6, 126.6, 123.4, 115.3, 93.3, 86.6, 74.3, 20.8 ppm.

IR (thin film) 3285, 2977, 1598, 1511, 1449, 1167, 982, 833, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{20}O_2$ [M$^+$]: 328.1463, Found: 328.1470.



S4 **S5** **1p**

(8*R*,9*S*,13*S*,14*S*)-3-(3-Hydroxy-3-(4-hydroxyphenyl)-3-phenylprop-1-yn-1-yl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[a]phenanthren-17-one (1p) was synthesized according to the literature procedure. [3] An oven-dried Schlenk tube was charged with triflate **S5**[4] (2.6 g, 6.5 mmol, 1.3 equiv), $Pd(PPh_3)_2Cl_2$ (351.6 mg, 0.5 mmol, 0.1 equiv), PPh_3 (164.9 mg, 0.65 mmol, 0.13 equiv), and CuI (49.4 mg, 0.25 mmol, 0.05 equiv). Then anhydrous DMF (5 mL) and anhydrous Et_3N (5 mL) were added under N_2. After degassing, the alkyne **S4** (1.1g, 5.0 mmol) was added, and the reaction mixture was stirred at 95 °C for 24 h. Then, the reaction mixture was cooled to room temperature, diluted with Et_2O, and washed with brine. The layers were separated and the organic layer was dried over Na_2SO_4, filtered, and concentrated.

The crude product was purified by flash column chromatography (10→30% EtOAc in hexanes) to afford the pure alkyne **1p** as a red solid (0.92 g, 39% yield).

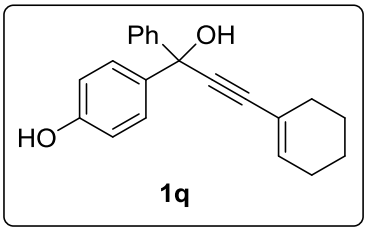
^{1}H NMR (400 MHz, acetone-d_6) δ 8.37 (br s, 1H), 7.72 (d, J = 7.2 Hz, 2H), 7.55 (d, J = 8.8 Hz, 2H), 7.39-7.33 (m, 3H), 7.31-7.25 (m, 3H), 6.83 (d, J = 8.8 Hz, 1H), 5.57 (br s, 1H), 2.94-2.90 (m, 2H), 2.51-2.41 (m, 2H), 2.35-2.30 (m, 1H), 2.15-1.98 (m, 4H), 1.92-1.87 (m, 1H), 1.72-1.38 (m, 6H), 0.92 (s, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 219.2, 157.3, 147.6, 141.4, 138.2, 137.6, 132.5, 129.3, 128.5, 128.1, 127.6, 126.6, 126.2, 120.8, 115.3, 93.2, 86.5, 74.3, 50.9, 48.1, 45.0, 38.6, 35.8, 32.3, 29.5, 26.8, 26.1, 21.9, 13.9 ppm.

IR (thin film) 3396, 2932, 1724, 1614, 1511, 1264, 1056, 833, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{33}H_{32}O_3$ [M$^+$]: 476.2351, Found: 476.2334.

4-(3-(Cyclohex-1-en-1-yl)-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1q) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes) in 93% yield.



1q

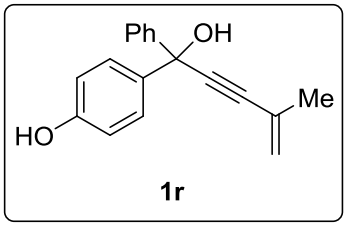
^{1}H NMR (400 MHz, acetone-d_6) δ 8.29 (br s, 1H), 7.61 (d, J = 7.2 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.29 (t, J = 7.2 Hz, 2H), 7.20 (t, J = 7.2 Hz, 1H), 6.76 (d, J = 7.6 Hz, 2H), 6.12-6.09 (m, 1H), 5.36 (br s, 1H), 2.17-2.07 (m, 4H), 1.66-1.56 (m, 4H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.1, 147.8, 138.4, 134.9, 128.4, 128.0, 127.4, 126.6, 121.1, 115.1, 91.3, 88.1, 74.1, 25.9, 22.7, 21.9 ppm.

IR (thin film) 3396, 2931, 1614, 1511, 1448, 1227, 1166, 833, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{20}O_2$ [M$^+$]: 304.1463, Found: 304.1477.

4-(1-Hydroxy-4-methyl-1-phenylpent-4-en-2-yn-1-yl)phenol (1r) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes) in 86% yield.



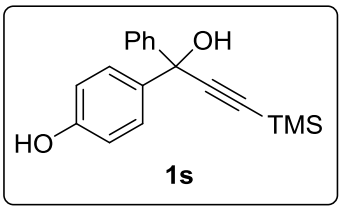
^{1}H NMR (400 MHz, acetone-d_6) δ 8.38 (br s, 1H), 7.61 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.31 (t, J = 7.6 Hz, 2H), 7.21 (t, J = 7.6 Hz, 1H), 6.77 (d, J = 8.8 Hz, 2H), 5.49 (br s, 1H), 5.32-5.28 (m, 2H), 1.92 (t, J = 1.6 Hz, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.1, 147.4, 138.0, 128.3, 127.9, 127.4, 127.3, 126.4, 121.8, 115.1, 93.0, 87.2, 74.0, 23.2 ppm.

IR (thin film) 3384, 1613, 1511, 1448, 1237, 1172, 996, 837, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{16}O_2$ [M$^+$]: 264.1150, Found: 264.1153.

4-(1-Hydroxy-1-phenyl-3-(trimethylsilyl)prop-2-yn-1-yl)phenol (1s) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes) in 92% yield.



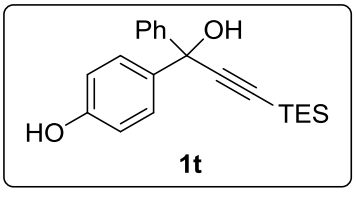
^{1}H NMR (400 MHz, acetone-d_6) δ 8.32 (br s, 1H), 7.62 (d, J = 7.2 Hz, 2H), 7.44 (d, J = 8.8 Hz, 2H), 7.30 (t, J = 7.2 Hz, 2H), 7.21 (t, J = 7.2 Hz, 1H), 6.77 (d, J = 8.8 Hz, 2H), 5.47 (br s, 1H), 0.20 (s, 9H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 155.2, 147.2, 137.9, 128.4, 127.9, 127.6, 126.5, 115.2, 110.4, 90.2, 74.1, -0.3 ppm.

IR (thin film) 3355, 2974, 2168, 1613, 1512, 1449, 1251, 1059, 842, 766 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{20}O_2Si$ [M$^+$]: 296.1233, Found: 296.1237.

4-(1-Hydroxy-1-phenyl-3-(triethylsilyl)prop-2-yn-1-yl)phenol (1t) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes) in 50% yield.



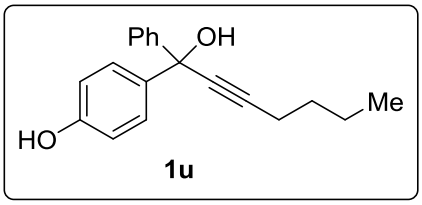
^{1}H NMR (400 MHz, $CDCl_2$) δ 7.62 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.8 Hz, 2H), 7.33 (t, J = 7.2 Hz, 2H), 7.27 (t, J = 7.2 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 5.40 (br s, 1H), 1.04 (t, J = 8.0 Hz, 9H), 0.69 (q, J = 8.0 Hz, 6H) ppm.

^{13}C NMR (100 MHz, $CDCl_2$) δ 155.7, 145.6, 137.8, 128.6, 127.94, 127.86, 126.2, 115.3, 109.7, 89.8, 74.8, 7.7, 4.7 ppm.

IR (thin film) 3321, 2956, 1598, 1511, 1450, 1172, 1004, 835, 728 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{26}O_2Si$ [M^+]: 338.1702, Found: 338.1714.

4-(1-Hydroxy-1-phenylhept-2-yn-1-yl)phenol (1u) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes) in 89% yield.



1u

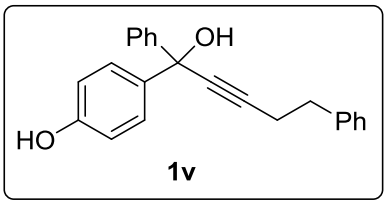
^{1}H NMR (400 MHz, acetone-d_6) δ 8.32 (br s, 1H), 7.60 (d, J = 7.2 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.28 (t, J = 7.2 Hz, 2H), 7.18 (t, J = 7.2 Hz, 1H), 6.75 (d, J = 8.8 Hz, 2H), 5.26 (br s, 1H), 2.32 (t, J = 7.2 Hz, 2H), 2.06-1.41 (m, 4H), 0.92 (t, J = 7.2 Hz, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.0, 148.0, 138.6, 128.2, 127.9, 127.2, 126.5, 114.9, 86.6, 84.9, 73.8, 31.3, 22.2, 18.6, 13.5 ppm.

IR (thin film) 3385, 2958, 1614, 1511, 1449, 1171, 1001, 829, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{19}H_{20}O_2$ [M^+]: 280.1463, Found: 280.1466.

4-(1-Hydroxy-1,5-diphenylpent-2-yn-1-yl)phenol (1v) was prepared as brown oil according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 67% yield).



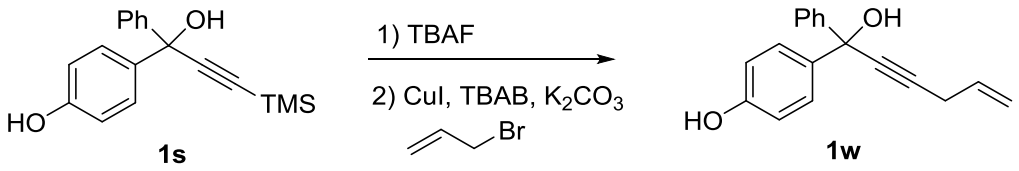
1v

^{1}H NMR (400 MHz, acetone-d_6) δ 8.29 (br s, 1H), 7.52 (d, J = 8.4 Hz, 2H), 7.35-7.15 (m, 10H), 6.72 (d, J = 8.8 Hz, 2H), 5.21 (br s, 1H), 2.87 (t, J = 7.2 Hz, 2H), 2.63 (t, J = 7.2 Hz, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.0, 147.9, 141.5, 138.6, 129.3, 128.9, 128.3, 128.0, 127.3, 126.8, 126.6, 115.0, 86.2, 85.6, 73.8, 35.3, 21.2 ppm.

IR (thin film) 3340, 2977, 1611, 1511, 1449, 1171, 1018, 832, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{20}O_2$ [M^+]: 328.1463, Found: 328.1452.



4-(1-Hydroxy-1-phenylhex-5-en-2-yn-1-yl)phenol (1w) was prepared from alcohol **1s** according to the literature procedure.[5] At room temperature, to a stirred solution of alcohol **1s** (3.0 g, 10.0 mmol) in THF (20 mL) was added a solution of tetrabutylammonium fluoride (3.1 g, 12.0 mmol, 1.2 equiv) in THF (12 mL) dropwise. The mixture was stirred for 1 h and then concentrated to evaporate most of the THF solvent. The residue was taken up in diethyl ether and water (1:1, 20 ml). The resulting layers were separated and the aqueous layer was extracted with diethyl ether (3 × 20 mL). The combined organic layers were dried, filtered, and concentrated. The residue was purified by silica gel column chromatography (20→30% EtOAc in hexanes) to give the terminal alkyne (1.95 g, 87% yield) as pale brown solid. Under N_2, to a stirred solution of the above terminal alkyne (1.12 g, 5.0 mmol) in dry DMF (5 mL) were sequentially added K_2CO_3 (0.97 g, 7.0 mmol), tetrabutylammonium bromide (241 mg, 0.75 mmol), and CuI (48 mg, 0.25 mmol) at room temperature. Allyl bromide (0.65 mL, 7.5 mmol) was added 15 min later. Next, the reaction mixture was stirred for 24 h before it was poured into water and extracted with ether (20 mL×3). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentration. The residue was purified by silica gel flash column chromatography (10→20% EtOAc in hexanes) to give the pure **1w** (1.13 g, 66% yield) as pale yellow solid.

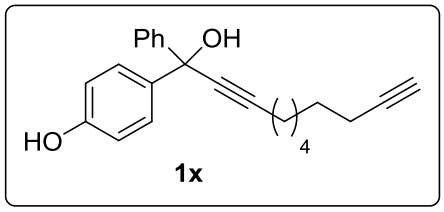
^{1}H NMR (400 MHz, acetone-d_6) δ 8.30 (br s, 1H), 7.61 (d, J = 8.0 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 7.29 (t, J = 7.6 Hz, 2H), 7.20 (t, J = 7.6 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 5.94-5.83 (m, 1H), 5.41-5.30 (m, 1H), 5.36 (br s, 1H), 5.12-5.07 (m, 1H), 3.14-2.98 (m, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.1, 147.8, 138.5, 133.5, 128.3, 128.0, 127.4, 126.5, 116.0, 115.1, 87.3, 83.3, 73.9, 23.2 ppm.

IR (thin film) 3380, 1612, 1511, 1448, 1233, 1172, 994, 830, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{16}O_2$ [M^+]: 264.1150, Found: 264.1140.

4-(1-Hydroxy-1-phenylundeca-2,10-diyn-1-yl)phenol (1x) was prepared as brown oil according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 78% yield).



1x

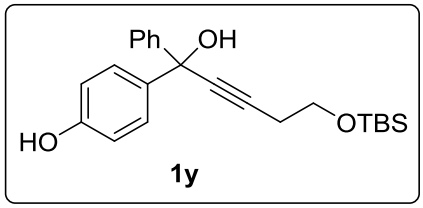
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.57 (d, J = 7.2 Hz, 2H), 7.44 (d, J = 8.8 Hz, 2H), 7.31 (t, J = 7.2 Hz, 2H), 7.24 (t, J = 7.2 Hz, 1H), 6.76 (t, J = 8.8 Hz, 2H), 5.24 (br s, 1H), 2.83 (br s, 1H), 2.35 (t, J = 7.2 Hz, 2H), 2.21-2.15 (m, 2H), 1.98-1.96 (m, 1H), 1.65-1.38 (m, 8H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 155.6, 146.3, 138.4, 128.5, 127.9, 127.8, 126.2, 115.2, 88.4, 85.0, 83.6, 74.5, 68.4, 28.81, 28.77, 28.7, 28.6, 19.1, 18.6 ppm.

IR (thin film) 3384, 3296, 2935, 1611, 1511, 1447, 1231, 1171, 831, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{24}O_2$ [M^+]: 332.1776, Found: 332.1774.

4-(5-((*tert*-Butyldimethylsilyl)oxy)-1-hydroxy-1-phenylpent-2-yn-1-yl)phenol (1y) was prepared as yellow oil according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 75% yield).



1y

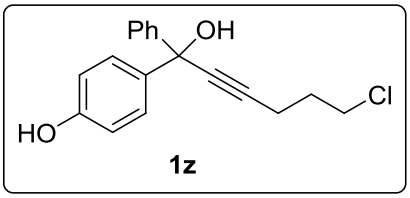
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.57 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.8 Hz, 2H), 7.31 (t, J = 7.6 Hz, 2H), 7.24 (t, J = 7.2 Hz, 1H), 6.75 (t, J = 8.8 Hz, 2H), 3.80 (t, J = 6.8 Hz, 2H), 2.55 (t, J = 6.8 Hz, 2H), 0.91 (s, 9H), 0.08 (s, 6H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 155.6, 146.2, 138.3, 128.5, 127.9, 127.8, 126.2, 115.2, 85.5, 84.4, 74.4, 62.2, 26.1, 23.6, 18.6, –5.2 ppm.

IR (thin film) 3363, 2929, 1613, 1511, 1448, 1254, 1105, 834, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{30}O_3Si$ [M^+]: 382.1964, Found: 382.1962.

4-(6-Chloro-1-hydroxy-1-phenylhex-2-yn-1-yl)phenol (1z) was prepared as pale yellow solid according to the General Procedure A (purification by flash column chromatography: 20→30% EtOAc in hexanes, 98% yield).

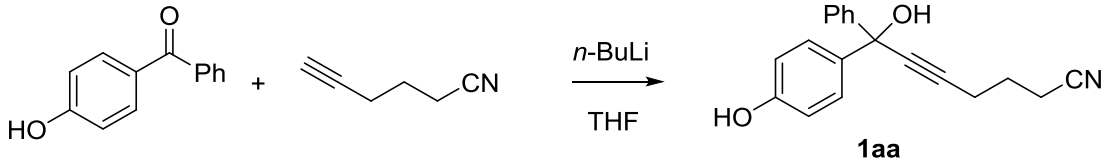


^{1}H NMR (400 MHz, acetone-d_6) δ 8.28 (br s, 1H), 7.60 (d, J = 7.2 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 7.29 (t, J = 7.2 Hz, 2H), 7.20 (t, J = 7.2 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 5.35 (br s, 1H), 3.74 (t, J = 6.4 Hz, 2H), 2.52 (t, J = 6.8 Hz, 2H), 2.00 (q, J = 6.4 Hz, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.0, 147.8, 138.4, 128.3, 127.9, 127.3, 126.4, 115.0, 85.8, 84.9, 73.8, 44.2, 32.0, 16.3 ppm.

IR (thin film) 3388, 2961, 1613, 1511, 1448, 1234, 1172, 1016, 831, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{17}{}^{35}ClO_2$ [M$^+$]: 300.0917, Found: 300.0913.



7-Hydroxy-7-(4-hydroxyphenyl)-7-phenylhept-5-ynenitrile (1aa). At –78 °C under N_2, to an oven-dried flask charged with a solution of hex-5-ynenitrile (1.3 mL, 12.5 mmol) in dry THF (50 mL) was added ^{n}BuLi (12.5 mmol, 5.2 mL, 2.4 M in hexane) dropwise. The reaction was stirred for 1 h at the same temperature and then a solution of the ketone (1.0 g, 5.0 mmol) in THF (3 mL) was added via syringe. The reaction mixture was then warmed to 60 °C and stirred overnight. Upon completion (~12 h), the reaction mixture was cooled to 0 °C. A saturated aqueous NH_4Cl solution (20 mL) was added dropwise. The organic layer was separated. The aqueous layer was extracted with Et_2O (3×20 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel chromatography to afford the pure alcohol **1aa** as brown oil (20→30% EtOAc in hexanes, 0.87 g, 60% yield).

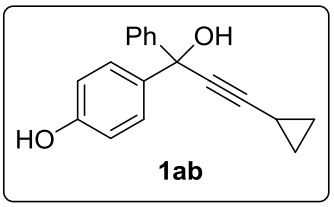
^{1}H NMR (400 MHz, acetone-d_6) δ 8.28 (br s, 1H), 7.60 (d, J = 7.2 Hz, 2H), 7.42 (d, J = 8.8 Hz, 2H), 7.29 (t, J = 7.6 Hz, 2H), 7.20 (t, J = 7.2 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 5.36 (br s, 1H), 2.61 (t, J = 7.2 Hz, 2H), 2.51 (t, J = 6.8 Hz, 2H), 1.97-1.87 (m, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.2, 147.8, 138.5, 128.4, 128.0, 127.4, 126.5, 120.0, 115.2, 86.5, 84.6, 73.9, 25.4, 18.2, 16.1 ppm.

HRMS (CI+) Calcd for $C_{19}H_{18}NO_2$ [M+H$^+$]: 292.1338, Found: 292.1335.

IR (thin film) 3385, 2977, 2250, 1609, 1512, 1446, 1372, 1230, 835, 701 cm^{-1}.

4-(3-Cyclopropyl-1-hydroxy-1-phenylprop-2-yn-1-yl)phenol (1ab) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 88% yield).



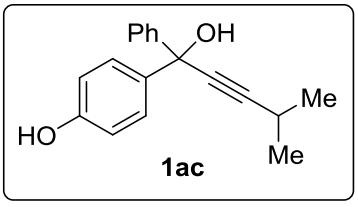
^{1}H NMR (400 MHz, acetone-d_6) δ 8.29 (br s, 1H), 7.58 (d, J = 7.2 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H), 7.27 (t, J = 7.2 Hz, 2H), 7.18 (t, J = 7.2 Hz, 1H), 6.75 (d, J = 8.8 Hz, 2H), 5.21 (br s, 1H), 1.42-1.35 (m, 1H), 0.85-0.75 (m, 2H), 0.58-0.68 (m, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 156.9, 147.9, 138.5, 128.2, 127.8, 127.2, 126.4, 115.0, 89.8, 79.8, 73.7, 8.2, -0.3 ppm.

IR (thin film) 3355, 2236, 1613, 1511, 1448, 1235, 1171, 994, 836, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{16}O_2$ [M$^+$]: 264.1150, Found: 264.1150.

4-(1-Hydroxy-4-methyl-1-phenylpent-2-yn-1-yl)phenol (1ac) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 96% yield).



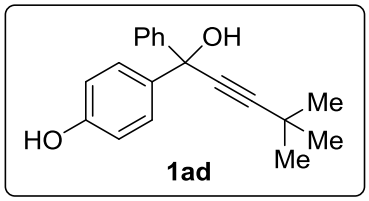
^{1}H NMR (400 MHz, acetone-d_6) δ 8.25 (br s, 1H), 7.62 (d, J = 7.2 Hz, 2H), 7.44 (d, J = 8.8 Hz, 2H), 7.28 (t, J = 7.6 Hz, 2H), 7.19 (t, J = 7.6 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 5.23 (br s, 1H), 2.76-2.64 (m, 1H), 1.21 (s, 3H), 1.20 (s, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.0, 148.0, 138.7, 128.2, 127.9, 127.2, 126.5, 115.0, 92.0, 84.2, 73.7, 23.0, 21.0 ppm.

IR (thin film) 3388, 2971, 1613, 1511, 1448, 1235, 1169, 994, 835, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{18}H_{18}O_2$ [M$^+$]: 266.1307, Found: 266.1308.

4-(1-Hydroxy-4,4-dimethyl-1-phenylpent-2-yn-1-yl)phenol (1ad) was prepared as white solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 86% yield).



1ad

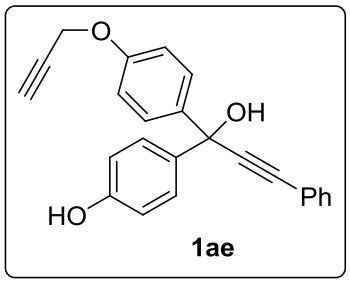
^{1}H NMR (400 MHz, acetone-d_6) δ 8.25 (br s, 1H), 7.62 (d, J = 8.8 Hz, 2H), 7.45 (d, J = 8.4 Hz, 2H), 7.28 (t, J = 7.2 Hz, 2H), 7.18 (t, J = 7.2 Hz, 1H), 6.75 (d, J = 8.8 Hz, 2H), 5.23 (br s, 1H), 1.28 (s, 9H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.0, 148.2, 138.9, 128.3, 127.9, 127.3, 126.5, 115.1, 94.8, 83.7, 73.7, 31.0, 27.9 ppm.

IR (thin film) 3407, 2970, 1614, 1511, 1449, 1264, 1171, 1002, 832, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{19}H_{20}O_2$ [M$^+$]: 280.1463, Found: 280.1458.

4-(1-Hydroxy-3-phenyl-1-(4-(prop-2-yn-1-yloxy)phenyl)prop-2-yn-1-yl)phenol (1ae) was prepared as brown oil according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 75% yield).



1ae

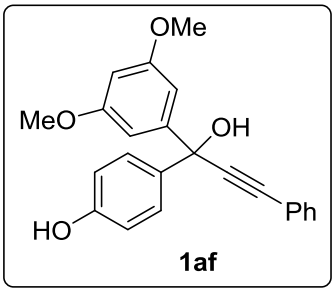
^{1}H NMR (400 MHz, acetone-d_6) δ 8.36 (br s, 1H), 7.61 (d, J = 8.8 Hz, 2H), 7.51 (d, J = 8.8 Hz, 4H), 7.40-7.37 (m, 3H), 6.97 (d, J = 8.8 Hz, 2H), 6.81 (d, J = 8.8 Hz, 2H), 5.54 (br s, 1H), 4.77 (d, J = 2.4 Hz, 2H), 3.06 (t, J = 2.4 Hz, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 157.4, 157.1, 140.3, 138.1, 131.9, 129.03, 128.97, 127.9, 127.8, 123.5, 115.1, 114.7, 94.0, 86.0, 79.5, 76.6, 73.8, 55.9 ppm.

IR (thin film) 3391, 3290, 1597, 1508, 1225, 1170, 1026, 836, 691 cm^{-1}.

HRMS (CI+) Calcd for $C_{24}H_{18}O_3$ [M$^+$]: 354.1256, Found: 354.1245.

4-(1-(3,5-Dimethoxyphenyl)-1-hydroxy-3-phenylprop-2-yn-1-yl)phenol (1af) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 85% yield).



1af

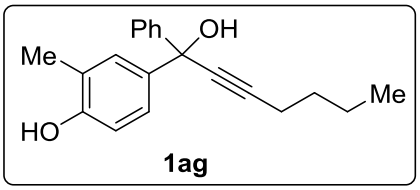
^{1}H NMR (400 MHz, acetone-d_6) δ 8.34 (br s, 1H), 7.55-7.49 (m, 4H), 7.40-7.37 (m, 3H), 6.88-6.86 (m, 2H), 6.80 (d, J = 8.8 Hz, 2H), 6.39-6.37 (m, 1H), 5.58 (br s, 1H), 3.76 (s, 6H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 161.2, 157.3, 149.8, 137.8, 132.0, 129.13, 129.08, 127.9, 123.6, 115.2, 105.0, 99.0, 93.8, 86.0, 74.2, 55.3 ppm.

IR (thin film) 3417, 2974, 1597, 1511, 1458, 1204, 1155, 1052, 837, 759, 691 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{20}O_4$ [M$^+$]: 360.1362, Found: 360.1372.

4-(1-Hydroxy-1-phenylhept-2-yn-1-yl)-2-methylphenol (1ag) was prepared as pale yellow solid according to the General Procedure B (purification by flash column chromatography: 20→30% EtOAc in hexanes, 89% yield).



1ag

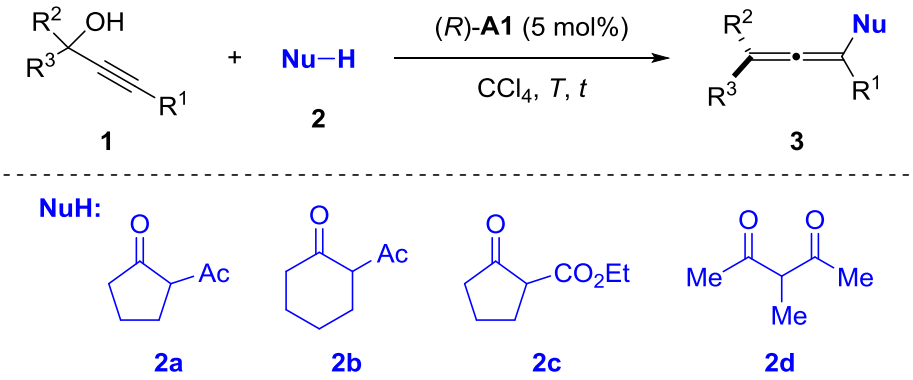
^{1}H NMR (400 MHz, acetone-d_6) δ 8.12 (br s, 1H), 7.60 (d, J = 8.8 Hz, 2H), 7.34 (s, 1H), 7.30-7.23 (m, 3H), 7.18 (t, J = 7.2 Hz, 1H), 6.72 (d, J = 8.4 Hz, 1H), 5.16 (br s, 1H), 2.32 (d, J = 6.8 Hz, 2H), 2.16 (s, 3H), 1.59-1.42 (m, 4H), 0.92 (t, J = 7.2 Hz, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 154.9, 148.1, 138.6, 129.3, 128.2, 127.2, 126.5, 125.2, 123.8, 114.3, 86.6, 85.0, 73.8, 31.3, 22.3, 18.6, 16.1, 13.6 ppm.

IR (thin film) 3386, 2957, 1610, 1505, 1449, 1260, 1114, 1012, 819, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{22}O_2$ [M$^+$]: 294.1620, Found: 294.1610.

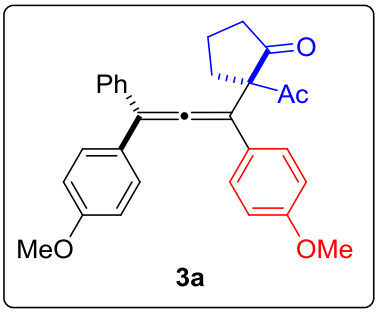
General Procedure C



At room temperature, an oven-dried 10-mL vial was charged with a mixture of the alcohol substrate **1** (0.2 mmol), the carbon nucleophile **2** (0.3 mmol, 1.5 equiv), and CCl_4 (3.8 mL). The reaction mixture was cooled to –20 °C (for **3a-j**) or 0 °C (for **3k-p**) unless otherwise specified, and a solution of the catalyst (*R*)-**A1** (10 μmol, 5 mol%) in CCl_4 (0.2 mL) was added in one portion. The reaction mixture was stirred at the same temperature. The progress was monitored by thin layer chromatography. Upon completion, the crude mixture was directly subjected to silica gel flash chromatography to afford the pure product **3**.

Note: All the racemic products (used for HPLC reference in determining the ee value), were prepared using the same reaction with the simplest racemic phosphoric acid catalyst or TsOH.

(*R*)-2-Acetyl-2-((*R*)-1,3-bis(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)cyclope



ntan-1-one (3a) was prepared as pale yellow oil from propargylic alcohol **1a** (0.2 mmol, 68.9 mg) and **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column chromatography: 5-10%

EtOAc in hexanes, 80.6 mg, 89% yield, 6:1 d.r., 90% ee).

$[\alpha]_D^{25}$: +218.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.3 min (major), 9.6 min (minor).

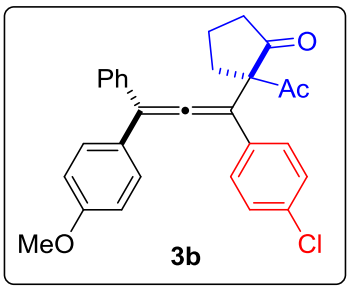
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.28 (m, 9H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 3.80 (s, 3H), 3.01-2.91 (m, 1H), 2.39 (s, 3H), 2.31-2.06 (m, 3H), 1.95-1.82 (m, 1H), 1.73-1.63 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.6, 206.9, 206.7, 159.3, 159.1, 136.4, 129.6, 129.1, 128.5, 128.4, 127.9, 127.8, 127.7, 125.6, 114.3, 113.9, 107.4, 71.1, 55.2 (2C), 37.9, 31.3, 28.4, 18.7 ppm.

IR (thin film) 2955, 1701, 1605, 1507, 1245, 1030, 906, 832, 730 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{28}O_4$ [M^+]: 452.1988, Found: 452.1998.

(*R*)-2-Acetyl-2-((*R*)-1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-phenylpropa-1,2-die



3b

n-1-yl)cyclopentan-1-one (3b) was prepared as pale yellow foam from propargylic alcohol **1b** (0.2 mmol, 69.6 mg) **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 81.6 mg, 90% yield, 7:1 d.r., 94% ee).

$[\alpha]_D^{25}$: +236.0 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.3 min (major), 9.3 min (minor).

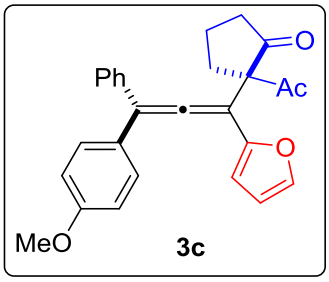
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.31 (m, 7H), 7.31-7.26 (m, 4H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 3.01-2.91 (m, 1H), 2.35 (s, 3H), 2.25-2.12 (m, 3H), 1.94-1.82 (m, 1H), 1.72-1.61 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.2, 207.2, 206.2, 159.5, 135.8, 133.5, 132.2, 129.5, 129.0, 128.6, 128.0, 127.9, 127.8, 127.0, 114.6, 114.0, 107.1, 70.9, 55.2, 37.9, 31.4, 28.2, 18.7 ppm.

IR (thin film) 2960, 1702, 1605, 1508, 1246, 1031, 906, 830, 697 cm^{-1}.

HRMS (CI+) Calcd for $C_{29}H_{25}{}^{35}ClO_3$ [M^+]: 456.1492, Found: 456.1482.

(*R*)-2-Acetyl-2-((*S*)-1-(furan-2-yl)-3-(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)



cyclopentan-1-one (3c) was prepared as pale yellow foam from propargylic alcohol **1c** (0.2 mmol, 60.8 mg) **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 75.8 mg, 92% yield, 7:1 d.r., 77% ee).

$[\alpha]_D{}^{25}$: +167.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 3% EtOH in hexanes; 0.5 mL/min; retention times: 14.4 min (major), 18.6 min (minor).

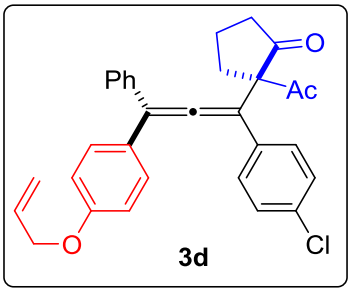
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.27 (m, 8H), 6.93 (d, *J* = 8.8 Hz, 2H), 6.40-6.38 (m, 1H), 6.27-6.25 (m, 1H), 3.83 (s, 3H), 2.91-2.82 (m, 1H), 2.40 (s, 3H), 2.25-2.05 (m, 3H), 1.91-1.81 (m, 1H), 1.70-1.59 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.1, 205.6, 205.1, 159.5, 147.1, 142.5, 136.2, 129.7, 128.5, 128.4, 128.0, 127.6, 115.3, 114.0, 111.5, 108.3, 100.9, 70.4, 55.3, 37.9, 31.3, 27.9, 18.8 ppm.

IR (thin film) 2961, 1704, 1604, 1508, 1246, 1028, 904, 833, 696 cm^{-1}.

HRMS (CI+) Calcd for $C_{27}H_{24}O_4$ [M^+]: 412.1675, Found: 412.1674.

(*R*)-2-Acetyl-2-((*R*)-3-(4-(allyloxy)phenyl)-1-(4-chlorophenyl)-3-phenylpropa-1,2-di



en-1-yl)cyclopentan-1-one (3d) was prepared as pale yellow foam from propargylic alcohol **1d** (0.2 mmol, 74.8 mg) **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column

chromatography: 5-10% EtOAc in hexanes, 90.6 mg, 94% yield, 6:1 d.r., 92% ee). $[\alpha]_D^{25}$: +156.6 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 3% EtOH in hexanes; 0.5 mL/min; retention times: 12.4 min (major), 20.9 min (minor).

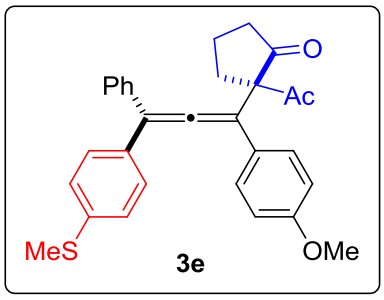
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.23 (m, 11H), 6.95 (d, J = 8.8 Hz, 2H), 6.12-6.01 (m, 1H), 5.43 (d, J = 17.2 Hz, 1H), 5.30 (d, J = 10.8 Hz, 1H), 4.57 (d, J = 5.2 Hz, 2H), 2.99-2.90 (m, 1H), 2.34 (s, 3H), 2.27-2.12 (m, 3H), 1.94-1.82 (m, 1H), 1.71-1.59 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.2, 207.2, 206.3, 158.6, 135.9, 133.5, 133.1, 132.3, 129.5, 129.0, 128.6, 128.1, 127.94, 127.89, 127.2, 117.7, 114.8, 114.6, 107.1, 70.9, 68.8, 37.9, 31.5, 28.3, 18.8 ppm.

IR (thin film) 2967, 1702, 1604, 1507, 1243, 1175, 1010, 915, 830, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{31}H_{27}{}^{35}ClO_3$ [M^+]: 482.1649, Found: 482.1635.

(*R*)-2-Acetyl-2-((*R*)-1-(4-methoxyphenyl)-3-(4-(methylthio)phenyl)-3-phenylpropa-1,2-dien-1-yl)cyclopentan-1-one (3e) was prepared as pale yellow oil from propargylic alcohol **1e** (0.2 mmol, 72.1 mg) **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 89.9 mg, 96% yield, 6:1 d.r., 84% ee).



3e

$[\alpha]_D^{25}$: +213.8 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.7 min (major), 10.4 min (minor).

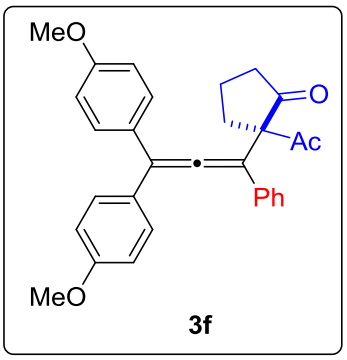
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.30 (m, 7H), 7.30-7.21 (m, 4H), 6.87 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 3.02-2.93 (m, 1H), 2.52 (s, 3H), 2.38 (s, 3H), 2.31-2.07 (m, 3H), 1.96-1.84 (m, 1H), 1.74-1.61 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.5, 206.8, 206.6, 159.2, 138.3, 136.0, 132.1, 128.7, 128.6, 128.4, 127.9, 127.8, 126.4, 125.3, 114.3, 114.0, 107.8, 71.1, 55.2, 37.9, 31.4, 28.4, 18.8, 15.6 ppm.

IR (thin film) 2957, 1702, 1604, 1509, 1248, 1029, 906, 831, 730 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{28}O_3S$ [M^+]: 468.1759, Found: 468.1773.

(*R*)-2-Acetyl-2-(3,3-bis(4-methoxyphenyl)-1-phenylpropa-1,2-dien-1-yl)cyclopentan



3f

-1-one (3e) was prepared as pale yellow oil from propargylic alcohol **1f** (0.2 mmol, 68.9 mg) and **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (2 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 88.5 mg, 98% yield, 86% ee).

$[\alpha]_D^{25}$: +179.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 9.3 min (major), 16.5 min (minor).

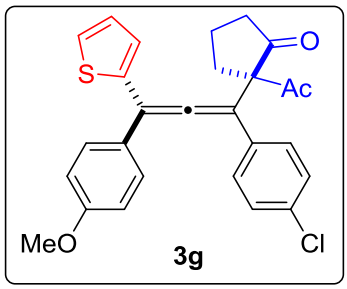
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.8 Hz, 2H), 7.35-7.21 (m, 7H), 6.93 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 3.82 (s, 3H), 3.01-2.92 (m, 1H), 2.37 (s, 3H), 2.31-2.07 (m, 3H), 1.93-1.83 (m, 1H), 1.74-1.67 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 212.8, 206.9 (2C), 159.4 (2C), 133.8, 129.6, 129.1, 128.9, 128.2, 127.7, 127.6, 126.6, 114.0, 113.9, 113.8, 107.5, 71.1, 55.3 (2C), 37.9, 31.3, 28.6, 18.8 ppm.

IR (thin film) 2956, 1702, 1605, 1507, 1244, 1172, 1029, 832, 696 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{28}O_4$ [M^+]: 452.1988, Found: 452.1989.

(*R*)-2-Acetyl-2-((S)-1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-(thiophen-2-yl)propa



3g

-1,2-dien-1-yl)cyclopentan-1-one (3g) was prepared as yellow oil from propargylic alcohol **1g** (0.2 mmol, 70.7 mg) **2a** (0.3 mmol, 36.3 μL) according to General Procedure **C** (3 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 78.6 mg, 85% yield, 5:1 d.r., 96% ee). $[\alpha]_D^{25}$: +189.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.9 min (major), 15.4 min (minor).

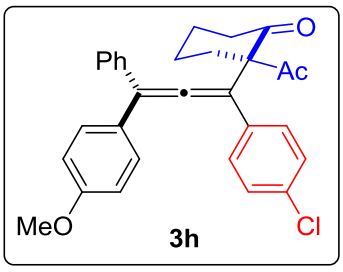
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.8 Hz, 2H), 7.31-7.24 (m, 5H), 7.11-6.91 (m, 4H), 3.85 (s, 3H), 2.98-2.90 (m, 1H), 2.42-2.18 (m, 3H), 2.33 (s, 3H), 2.01-1.89 (m, 2H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.8, 207.3, 206.2, 159.7, 139.0, 133.8, 132.1, 129.5, 129.0, 128.3, 127.8, 126.9, 126.8, 125.5, 114.0 109.6, 107.2, 71.0, 55.3, 37.9, 31.3, 28.2, 18.9 ppm.

IR (thin film) 2958, 1702, 1605, 1508, 1247, 1030, 911, 828, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{27}H_{23}{}^{35}ClO_3S$ [M^+]: 462.1056, Found: 462.1052.

(*R*)-2-Acetyl-2-((*R*)-1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-phenylpropa-1,2-die



3h

n-1-yl)cyclohexan-1-one (3h) was prepared as pale yellow foam from propargylic alcohol **1b** (0.2 mmol, 69.6 mg) and **2b** (0.3 mmol, 39.0 μL) with 200 mg anhydrous $MgSO_4$ according to General Procedure **C** (rt, 36 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 58.3 mg, 62% yield, 6:1 d.r., 90% ee).

$[\alpha]_D^{25}$: +41.9 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.6 min (major), 12.0 min (minor).

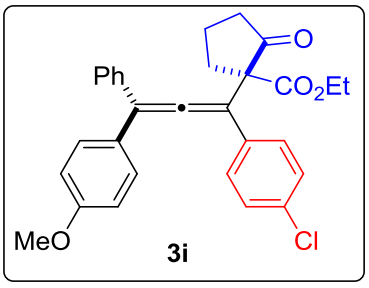
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.21 (m, 11H), 6.89 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 2.61-2.49 (m, 2H), 2.49-2.39 (m, 1H), 2.21-2.10 (m, 1H), 2.12 (s, 3H), 1.89-1.83 (m, 1H), 1.82-1.68 (m, 2H), 1.68-1.56 (m, 1H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 209.0, 208.0, 207.8, 159.4, 135.5, 133.4, 133.1, 129.6, 129.1, 128.74, 128.71, 128.1, 128.0, 127.1, 114.1, 113.8, 108.4, 71.1, 55.3, 41.2, 34.2, 27.7, 27.1, 21.6 ppm.

IR (thin film) 2939, 1699, 1604, 1508, 1246, 1175, 1030, 905, 831, 729 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{27}{}^{35}ClO_3$ [M^+]: 470.1649, Found: 470.1651.

Ethyl (*S*)-1-((*R*)-1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)-2-oxocyclopentane-1-carboxylate (3i) was prepared as pale yellow foam from propargylic alcohol **1b** (0.2 mmol, 69.6 mg) and **2c** (0.3 mmol, 44.5 μL) according to General Procedure **C** (-15 °C, 24 h, purified by flash column chromatography: 10% EtOAc and 20% CH_2Cl_2 in hexanes, 87.5 mg, 90% yield, 10:1 d.r., 82% ee).



3i

$[\alpha]_D^{25}$: +156.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK IC column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.5 min (minor), 10.8 min (major).

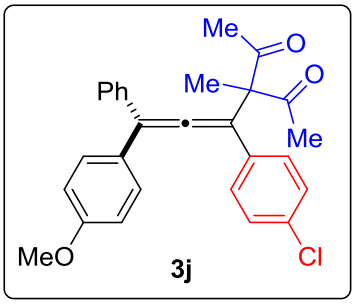
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.26 (m, 11H), 6.89 (d, *J* = 8.8 Hz, 2H), 4.35-4.17 (m, 2H), 3.82 (s, 3H), 2.78-2.69 (m, 1H), 2.40-2.34 (m, 1H), 2.23-2.13 (m, 1H), 2.04-1.95 (m, 1H), 1.95-1.87 (m, 1H), 1.70-1.62 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 210.6, 206.8, 170.6, 159.4, 135.8, 133.2, 132.4, 129.7, 128.7, 128.6, 128.3, 128.1, 128.0, 127.1, 114.3, 113.9, 105.8, 64.6, 62.0, 55.3, 36.4, 33.0, 18.9, 14.0 ppm.

IR (thin film) 2977, 1718, 1605, 1509, 1246, 1176, 1029, 906, 831, 699 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{27}{}^{35}ClO_3$ [M^+]: 486.1598, Found: 486.1581.

(*R*)-3-(1-(4-Chlorophenyl)-3-(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)-3-methylpentane-2,4-dione (3j) was prepared as pale yellow foam from propargylic alcohol **1b** (0.2 mmol, 69.6 mg) and **2d** (0.3 mmol, 34.9 μL) according to General Procedure **C** (0 °C, 10 h, purified by flash column chromatography: 5-10% EtOAc in hexanes, 72.1 mg, 81% yield, 78% ee).



3j

$[\alpha]_D{}^{25}$: +74.5 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.4 min (major), 9.8 min (minor).

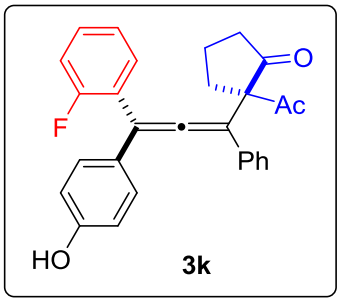
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.37 (m, 4H), 7.37-7.21 (m, 7H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 2.17 (s, 6H), 1.65 (m, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.2, 207.1 (2C), 159.5, 135.4, 133.5, 132.9, 129.4, 128.8, 128.76, 128.68, 128.2, 128.0, 127.1, 114.2, 114.0, 108.8, 68.7, 55.3, 27.8, 27.7, 20.3 ppm.

IR (thin film) 2933, 1701, 1604, 1508, 1247, 1090, 906, 831, 730 cm^{-1}.

HRMS (CI+) Calcd for $C_{28}H_{25}{}^{35}ClO_3$ [M^+]: 444.1492, Found: 444.1495.

(*R*)-2-Acetyl-2-((*S*)-3-(2-fluorophenyl)-3-(4-hydroxyphenyl)-1-phenylpropa-1,2-dien-1-yl)cyclopentan-1-one (3k) was prepared as pale yellow solid from propargylic alcohol **1h** (0.2 mmol, 63.8 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 65.3 mg, 75% yield, 21:1 d.r., 94% ee).



$[\alpha]_D^{25}$: +103.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK IC column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 20.7 min (major), 26.5 min (minor).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.39-7.31 (m, 6H), δ 7.31-7.21 (m, 1H), δ 7.21-7.13 (m, 4H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.23 (s br, 1H), 2.98-2.91 (m, 1H), 2.35 (s, 3H), 2.31-2.25 (m, 2H), 2.25-2.18 (m, 1H), 1.91-1.82 (m, 1H), 1.71-1.59 (m, 1H) ppm.

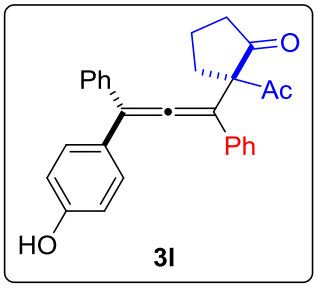
^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.7, 207.3, 206.7, 160.6 (d, $J_{C\text{-}F}$ = 123.2 Hz), 156.5, 134.0, 131.6 (d, $J_{C\text{-}F}$ = 3 Hz), 130.2 (d, $J_{C\text{-}F}$ = 8 Hz), 129.2, 129.0, 128.2, 127.3, 127.1, 124.8 (d, $J_{C\text{-}F}$ = 3 Hz), 124.0 (d, $J_{C\text{-}F}$ = 15 Hz), 116.4 (d, $J_{C\text{-}F}$ = 22 Hz), 116.0, 108.7, 108.5, 71.7, 38.8, 32.6, 28.5, 19.4 ppm.

^{19}F NMR (376 MHz, CD_2Cl_2) δ -112.9 ppm.

IR (thin film) 3569, 2950, 1665, 1508, 1259, 1103, 832 cm^{-1}.

HRMS (CI+) Calcd for $C_{28}H_{23}FO_3$ [M^+]: 426.1631, Found: 426.1629.

(*R*)-2-Acetyl-2-((*R*)-3-(4-hydroxyphenyl)-1,3-diphenylpropa-1,2-dien-1-yl)cyclopentan-1-one (3l) was prepared as pale yellow solid from propargylic alcohol **1i** (0.2 mmol, 60.1 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 69.5 mg, 85% yield, 16:1 d.r., 94% ee).



$[\alpha]_D^{25}$: +217.9 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 0.5 mL/min; retention times: 20.7 min (minor), 21.6 min (major).

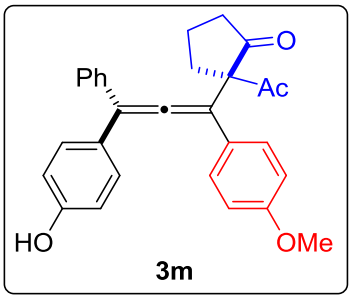
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.38-7.19 (m, 12H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.02 (br s, 1H), 2.96-2.87 (m, 1H), 2.33 (s, 3H), 2.31-2.17 (m, 3H), 1.93-1.82 (m, 1H), 1.75-1.62 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.7, 207.6, 207.1, 156.4, 136.5, 134.2, 130.1, 129.3, 129.0, 128.5, 128.4, 128.1, 127.7, 127.2, 115.9, 114.7, 108.3, 71.6, 38.4, 32.1, 28.7, 19.2 ppm.

IR (thin film) 3435, 1703, 1610, 1511, 1216, 763, 696 cm^{-1}.

HRMS (CI+) Calcd for $C_{28}H_{24}O_3$ [M^+]: 408.1725, Found: 408.1720.

(*R*)-2-Acetyl-2-((*R*)-3-(4-hydroxyphenyl)-1-(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)cyclopentan-1-one (3m) was prepared as pale yellow solid from propargylic alcohol **1j** (0.2 mmol, 66.1 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 71.0 mg, 81% yield, 10:1 d.r., 94% ee).



3m

$[\alpha]_D^{25}$: +170.7 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK IC column; 5% EtOH in hexanes; 0.5 mL/min; retention times: 23.4 min (major), 24.4 min (minor).

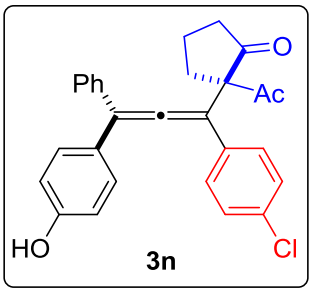
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.46-7.17 (m, 9H), 6.87-6.83 (m, 4H), 3.78 (s, 3H), 2.93-2.83 (m, 1H), 2.32 (s, 3H), 2.28-2.05 (m, 3H), 1.99-1.83 (m, 1H), 1.73-1.59 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.6, 207.0 (2C), 159.7, 156.2, 136.7, 130.1, 129.8, 129.0, 128.4, 128.2, 128.0, 126.1, 115.8, 114.7, 114.4, 108.0, 71.7, 55.7, 38.4, 32.0, 28.6, 19.2 ppm.

IR (thin film) 3362, 1695, 1607, 1508, 1355, 1252, 1178, 831, 754 cm^{-1}.

HRMS (CI+) Calcd for $C_{29}H_{26}O_4$ [M^+]: 438.1831, Found: 438.1815.

(*R*)-2-Acetyl-2-((*R*)-1-(4-chlorophenyl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl)cyclopentan-1-one (3n) was prepared as pale yellow solid from propargylic alcohol **1k** (0.2 mmol, 67.3 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 84.2 mg, 94% yield, 13:1 d.r., 97% ee).



$[\alpha]_D^{25}$: +253.3 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 0.5 mL/min; retention times: 21.9 min (minor), 23.7 min (major).

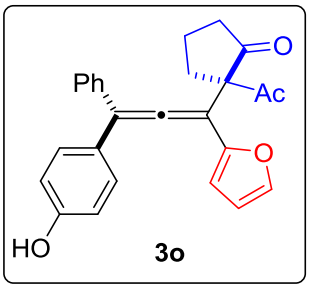
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.41-7.21 (m, 11H), 6.85 (d, *J* = 8.4 Hz, 2H), 5.77 (br s, 1H), 2.93-2.85 (m, 1H), 2.29 (s, 3H), 2.26-2.09 (m, 3H), 1.92-1.79 (m, 1H), 1.70-1.59 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.4, 207.7, 206.7, 156.5, 136.2, 133.8, 133.0, 130.2, 129.4, 129.1, 128.6, 128.49, 128.47, 127.4, 115.9, 115.0, 114.4, 107.6, 71.5, 38.4, 32.2, 28.5, 19.3 ppm.

IR (thin film) 3584, 1704, 1610, 1511, 1353, 1215, 1172, 834, 697 cm^{-1}.

HRMS (CI+) Calcd for $C_{28}H_{23}{}^{35}ClO_3$ [M^+]: 442.1336, Found: 442.1322.

(*R*)-2-Acetyl-2-((*S*)-1-(furan-2-yl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl) cyclopentan-1-one (3o) was prepared as white solid from propargylic alcohol **1l** (0.2 mmol, 58.1 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 71.7 mg, 90% yield, 12:1 d.r., 93% ee).



$[\alpha]_D^{25}$: +177.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 12.9 min (minor), 13.7 min (major).

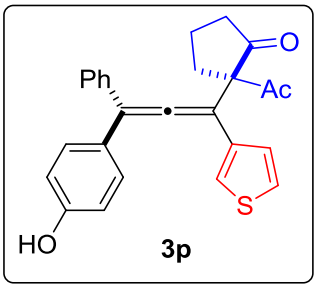
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.46-7.16 (m, 8H), 6.86 (d, *J* = 8.0 Hz, 2H), 6.42 (s, 1H), 6.35-6.15 (m, 2H), 2.91-2.78 (m, 1H), 2.38 (s, 3H), 2.27-2.16 (m, 2H), 2.15-2.05 (m, 1H), 1.91-1.79 (m, 1H), 1.71-1.59 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.3, 206.0, 205.7, 156.6, 147.5, 143.1, 136.5, 130.3, 129.0, 128.8, 128.5, 127.6, 115.9, 112.0, 108.9, 101.4, 70.9, 38.4, 32.0, 28.1, 19.3 ppm.

IR (thin film) 3393, 3022, 2968, 1702, 1507, 1217, 1019, 830, 755 cm^{-1}.

HRMS (CI+) Calcd for $C_{26}H_{22}O_4$ [M^+]: 398.1518, Found: 398.1515.

(*R*)-2-Acetyl-2-((*S*)-3-(4-hydroxyphenyl)-3-phenyl-1-(thiophen-3-yl)propa-1,2-dien-1-yl)cyclopentan-1-one (3p) was prepared as pale yellow solid from propargylic alcohol **1m** (0.2 mmol, 61.3 mg) according to General Procedure **C** (purified by flash column chromatography: 10-20% EtOAc in hexanes, 71.1 mg, 86% yield, 13:1 dr, 94% ee).



3p

$[\alpha]_D^{25}$: +185.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 12.6 min (minor), 14.2 min (major).

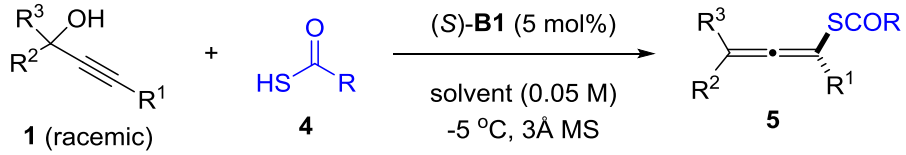
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.52-7.28 (m, 8H), 7.14 (d, *J* = 5.2 Hz, 1H), 7.05-7.03 (m, 1H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.85 (s br, 1H), 2.91-2.81 (m, 1H), 2.36-2.29 (m, 1H), 2.35 (s, 3H), 2.29-2.16 (m, 1H), 2.16-2.10 (m, 1H), 1.88-1.68 (m, 1H), 1.71-1.61 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 213.4, 207.2, 206.9, 156.3, 136.5, 135.0, 130.2, 130.0, 128.6, 128.3, 127.8, 127.6, 126.4, 121.6, 114.2, 104.7, 71.8, 38.4, 31.8, 28.3, 19.2 ppm.

IR (thin film) 3437, 1697, 1508, 1214, 757 cm^{-1}.

HRMS (CI+) Calcd for $C_{26}H_{22}O_3S$ [M^+]: 414.1290, Found: 414.1292.

General Procedure D



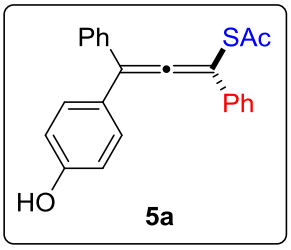
R = Me (**4a**), R = Ph (**4b**)

At room temperature, an oven-dried 10-mL vial was charged with a mixture of the alcohol substrate **1** (0.3 mmol), 3Å molecular sieves (90 mg), and the solvent (6.0 mL, toluene for **5a-g** and DCM for others, or noted otherwise). The reaction mixture was cooled to –5 °C, and a solution of the catalyst (*S*)-**B1**[6] (0.015 mmol, 5 mol%) in the same solvent (0.2 mL) and thioacetic acid **4a** (0.6 mmol, 42.9 uL) were added in one

portion. The reaction progress was monitored by thin layer chromatography. Upon completion, the reaction mixture was directly subjected to silica gel column flash chromatography (the column was pretreated with 10% AcOH in hexanes) to afford the desired product **5**.

Note: All the racemic products (used for HPLC reference in ee determination), were prepared using the same reaction with the simplest racemic phosphoric acid catalyst.

(*R*)-*S*-(3-(4-Hydroxyphenyl)-1,3-diphenylpropa-1,2-dien-1-yl) ethanethioate (5a)



was prepared as brown oil from substrate **1i** (0.3 mmol, 90.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 88.1 mg, 82% yield, 94% ee).

$[\alpha]_D^{25}$: +12.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 6.9 min (minor), 11.0 min (major).

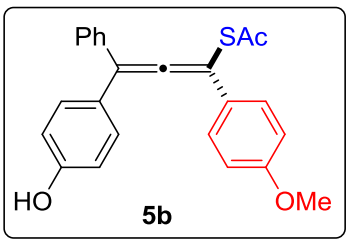
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.61 (d, *J* = 7.6 Hz, 2H), 7.45-7.32 (m, 7H), 7.29 (d, *J* = 8.0 Hz, 3H), 6.83 (d, *J* = 8.8 Hz, 2H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.7, 194.6, 156.5, 135.4, 134.7, 130.6, 129.2, 129.1, 129.0, 128.6, 128.4, 127.0, 126.8, 116.0, 112.9, 99.8, 30.4 ppm.

IR (thin film) 3443, 3064, 2925, 1683, 1504, 1267, 1117, 834, 760 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{18}O_2S$ [M^+]: 358.1028, Found: 358.1036.

(*R*)-*S*-(3-(4-Hydroxyphenyl)-1-(4-methoxyphenyl)-3-phenylpropa-1,2-dien-1-yl)eth anethioate ethanethioate (5b) was prepared as brown oil from substrate **1j** (0.3 mmol, 99.2 mg) according to the General Procedure D (purified by flash column chromatography: 20% EtOAc and 1% AcOH in hexanes, 87.3 mg, 75% yield, 91% ee).



$[\alpha]_D^{25}$: –17.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 20% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.5 min (minor), 14.3 min (major).

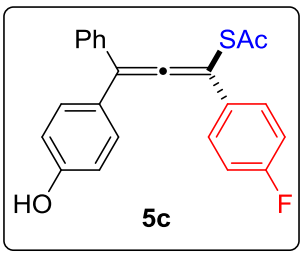
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.55 (d, *J* = 8.8 Hz, 2H), 7.46-7.33 (m, 5H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 3H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.0, 195.2, 160.0, 156.6, 135.7, 130.6, 129.2, 129.0, 128.5, 128.0, 127.0, 126.8, 116.0, 114.5, 112.8, 99.4, 55.7, 30.4 ppm.

IR (thin film) 3405, 3065, 1702, 1606, 1509, 1250, 1112, 833, 769 cm^{-1}.

HRMS (CI+) Calcd for $C_{24}H_{20}O_3S$ [M^+]: 388.1133, Found: 388.1145.

(*R*)-*S*-(1-(4-Fluorophenyl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl) ethan ethioate (5c) was prepared as brown oil from substrate **1n** (0.3 mmol, 95.4 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 93.6 mg, 83% yield, 92% ee).



5c

$[\alpha]_D^{25}$: +8.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.3 min (minor), 10.9 min (major).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.61-7.56 (m, 2H), 7.44-7.34 (m, 5H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 5.56 (br s, 1H), 2.44 (s, 3H) ppm.

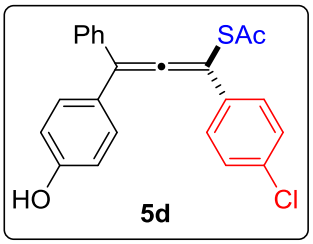
^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.5, 194.5, 162.9 (d, $J_{C\text{-}F}$ = 246 Hz), 156.5, 135.3, 130.9, 130.6, 129.2, 129.0, 128.6 (d, $J_{C\text{-}F}$ = 6.7 Hz), 128.5, 126.9, 115.9 (d, $J_{C\text{-}F}$ = 21.9 Hz), 115.8, 113.0, 98.9, 30.5 ppm.

^{19}F NMR (376 MHz, CD_2Cl_2) δ -114.8 ppm.

IR (thin film) 3402, 3028, 2927, 1691, 1505, 1226, 1118, 835 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{17}FO_2S$ [M^+]: 376.0933, Found: 376.0934

(*R*)-*S*-(1-(4-Chlorophenyl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl) ethan ethioate (5d) was prepared as brown oil from substrate **1k** (0.3 mmol, 100.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 106.1 mg, 90% yield, 92% ee).



$[\alpha]_D^{25}$: +5.3 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.9 min (minor), 16.5 min (major).

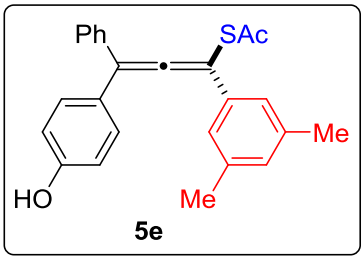
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.43-7.31 (m, 7H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 5.63 (br s, 1H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.8, 194.5, 156.6, 135.1, 134.0, 133.5, 130.6, 129.2, 129.15, 129.05, 128.7, 128.1, 126.7, 116.0, 113.3, 98.9, 30.5 ppm.

IR (thin film) 3395, 1682, 1609, 1510, 1218, 1172, 1012, 830, 769 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{17}{}^{35}ClO_2S$ [M^+]: 392.0638, Found: 392.0649

(*R*)-*S*-(1-(3,5-Dimethylphenyl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl)et hanethioate (5e) was prepared as brown oil from substrate **1o** (0.3 mmol, 98.4 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 107.3 mg, 93% yield, 92% ee).



$[\alpha]_D^{25}$: +13.8 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 5.1 min (minor), 5.9 min (major).

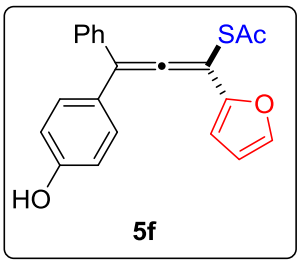
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.45-7.33 (m, 5H), 7.29 (d, *J* = 8.8 Hz, 2H), 7.23 (s, 2H), 6.93 (s, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 5.54 (br s, 1H), 2.43 (s, 3H), 2.30 (s, 6H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.6, 194.9, 156.4, 138.8, 135.5, 134.4, 130.6, 130.2, 129.2, 129.0, 128.5, 127.1, 124.5, 115.9, 112.5, 99.9, 30.4, 21.5 ppm.

IR (thin film) 3387, 3029, 2924, 1684, 1508, 1267, 1116, 839, 758 cm^{-1}.

HRMS (CI+) Calcd for $C_{25}H_{22}O_2S$ [M^+]: 386.1341, Found: 386.1346.

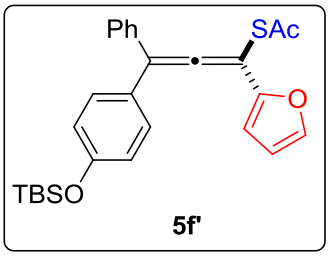
(*R*)-*S*-(3-(4-Hydroxyphenyl)-3-phenyl-1-(thiophen-3-yl)propa-1,2-dien-1-yl)ethanethioate ethanethioate (5f) was prepared as brown oil from substrate **1l** (0.3 mmol, 87.1 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 78.3 mg, 75% yield, 91% ee).



5f

$[\alpha]_D^{25}$: +9.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.9 min (major), 9.4 min (minor).

The pure **5f** is relatively unstable, but it can be stored as a solution in DCM or $CHCl_3$ in the refrigerator. However, its TBS-protected derivative **5f'** is stable. Therefore, the characterization of **5f'** is provided. At 0 °C, to a solution of **5f** (0.2 mmol, 69.6 mg) in CH_2Cl_2 (1 mL) were added 2,6-lutidine (83.2 uL, 0.72 mmol, 3.6 equiv) and a solution of TBSOTf (99.3 uL, 0.44 mmol, 2.2 equiv) in CH_2Cl_2 (0.2 mL) sequentially. The reaction was stirred at 0 °C for 2.5 h and then warmed to room temperature. The reaction mixture was directly subjected silica gel column chromatography (eluent: 3% Et_2O in hexanes, pale yellow oil, 0.62 g, quant. yield).



5f'

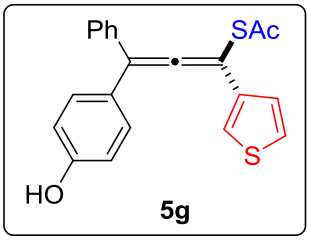
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.4 Hz, 2H), 7.41-7.30 (m, 6H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.46-6.40 (m, 2H), 2.42 (s, 3H), 1.00 (s, 9H), 0.22 (s, 6H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 210.4, 193.4, 155.9, 148.3, 143.1, 135.1, 130.3, 129.1, 128.5, 128.2, 127.3, 120.2, 114.1, 111.7, 109.1, 90.9, 30.0, 25.7, 18.2, 4.4 ppm.

IR (thin film) 3292, 2930, 1705, 1603, 1507, 1262, 1119, 910, 838 cm^{-1}.

HRMS (CI+) Calcd for $C_{27}H_{30}O_3SSi$ [M^+]: 462.1685, Found: 462.1692.

(*R*)-*S*-(3-(4-Hydroxyphenyl)-3-phenyl-1-(thiophen-3-yl)propa-1,2-dien-1-yl) ethanethioate ethanethioate (5g) was prepared as brown oil from substrate **1m** (0.3 mmol, 91.8 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 65.7 mg, 88% yield, 90% ee).



$[\alpha]_D^{25}$: +8.0 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.3 min (minor), 16.0 min (major).

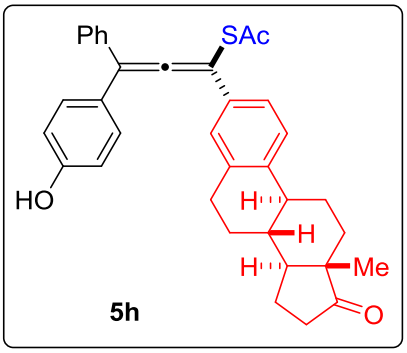
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.44-7.31 (m, 7H), 7.28 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 5.2 Hz, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 5.60 (br s, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.3, 195.1, 156.6, 137.0, 135.5, 130.7, 129.3, 129.0, 128.6, 127.1, 126.9, 126.5, 122.8, 116.0, 112.8, 95.2, 30.4 ppm.

IR (thin film) 3405, 3027, 1683, 1509, 1222, 1118, 841 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{16}O_2S_2$ [M^+]: 364.0592, Found: 364.0581

S-((*R*)-3-(4-Hydroxyphenyl)-1-((8*S*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)-3-phenylpropa-1,2-dien-1-yl) ethanethioate (5h) was prepared as red oil from substrate **1p** (0.2 mmol, 95.3 mg) according to the General Procedure D (purified by flash column chromatography: 20% EtOAc and 1% AcOH in hexanes, 81.3 mg, 76% yield, 89% de).



$[\alpha]_D^{25}$: +86.6 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 20% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.7 min (minor), 9.9 min (major).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.45-7.35 (m, 7H), 7.34-7.26 (m, 3H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.61 (br s, 1H), 2.93-2.83 (m, 2H), 2.55-2.45 (m, 1H), 2.43 (s, 3H), 2.31-2.21 (m, 1H),

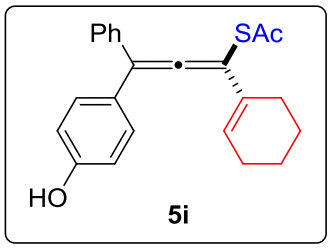
217-1.99 (m, 4H), 1.95-1.91 (m, 1H), 1.64-1.51 (m, 2H), 1.51-1.38 (m, 4H), 0.91 (m, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 222.1, 211.5, 194.8, 156.8, 140.5, 137.6, 135.6, 131.9, 130.6, 129.2, 129.0, 128.5, 127.0, 126.8, 126.1, 124.1, 116.0, 112.7, 99.6, 50.8, 48.4, 44.8, 38.4, 36.3, 32.0, 30.4, 29.9, 26.8, 26.1, 21.9, 14.1 ppm.

IR (thin film) 3401, 2925, 1721, 1511, 1269, 1117, 838 cm^{-1}.

HRMS (CI+) Calcd for $C_{35}H_{35}O_3S$ [$M+H^+$]: 535.2307, Found: 535.2337.

(*R*)-*S*-(1-(Cyclohex-1-en-1-yl)-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl) ethanethioate (5i) was prepared as brown oil from substrate **1q** (0.3 mmol, 91.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 76.1 mg, 70% yield, 91% ee).



5i

$[\alpha]_D^{25}$: +27.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 6.5 min (minor), 10.7 min (major).

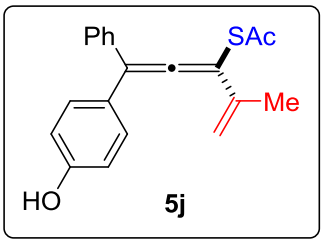
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.38-7.28 (m, 5H), 7.23 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.18-6.14 (m, 1H), 2.37 (s, 3H), 2.24-2.21 (m, 2H), 2.21-2.16 (m, 2H), 1.71-1.61 (m, 2H), 1.61-1.56 (m, 2H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 210.3, 195.0, 156.2, 136.1, 131.5, 130.5, 129.1, 128.9, 128.2, 127.7, 127.6, 115.8, 111.9, 101.6, 30.3, 27.7, 26.3, 23.1, 22.5 ppm.

IR (thin film) 3446, 3027, 2930, 1681, 1507, 1268, 1119, 832, 757 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{22}O_2S$ [M^+]: 362.1341, Found: 362.1353.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-4-methyl-1-phenylpenta-1,2,4-trien-3-yl) ethanethioate (5j) was prepared as brown oil from substrate **1r** (0.3 mmol, 79.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 69.6 mg, 72% yield, 88% ee).



$[\alpha]_D^{25}$: –33.4 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 6.7 min (minor), 7.8 min (major).

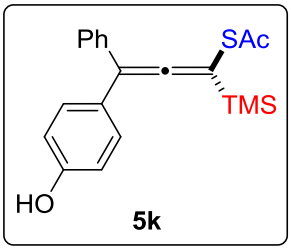
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.39-7.33 (m, 4H), 7.33-7.31 (m, 1H), 7.25 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.74 (br s, 1H), 5.40 (s, 1H), 5.13 (s, 1H), 2.39 (s, 3H), 2.02 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.7, 194.9, 156.4, 138.8, 135.6, 130.6, 129.2, 128.9, 128.4, 127.2, 115.9, 115.1, 111.7, 101.3, 30.3, 21.5 ppm.

IR (thin film) 3403, 1681, 1510, 1269, 1122, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{18}O_2S$ [M^+]: 322.1028, Found: 322.1016.

(*R*)-*S*-(3-(4-Hydroxyphenyl)-3-phenyl-1-(trimethylsilyl)propa-1,2-dien-1-yl) ethanethioate (5k) was prepared as brown oil from substrate **1s** (0.3 mmol, 89.1 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 74.4 mg, 70% yield, 92% ee).



$[\alpha]_D^{25}$: –31.9 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALCEL column; AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 6.6 min (minor), 7.9 min (major).

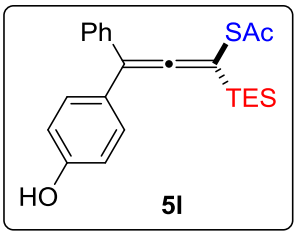
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.37 (d, *J* = 8.0 Hz, 4H), 7.33-7.27 (m, 1H), 7.24 (d, *J* = 8.8 Hz, 3H), 6.84 (d, *J* = 8.8 Hz, 2H), 2.37 (s, 3H), 0.24 (s, 9H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 212.9, 195.6, 160.0, 136.1, 130.3, 128.9, 127.8 (2C), 127.5, 115.9, 107.2, 90.8, 30.3, –1.1 ppm.

IR (thin film) 3538, 1657, 1506, 1251, 1130, 834, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{22}O_2SSi$ [M^+]: 354.1110, Found: 354.1107.

(*R*)-*S*-(3-(4-Hydroxyphenyl)-3-phenyl-1-(triethylsilyl)propa-1,2-dien-1-yl) ethanethioate (5l) was prepared as brown oil from substrate **1t** (0.3 mmol, 101.4 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 107.4 mg, 90% yield, 90% ee).



$[\alpha]_D^{25}$: –28.5 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: AD-H column; 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.5 min (minor), 9.9 min (major).

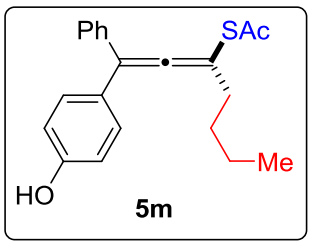
^{1}H NMR (400 MHz, acetone-d_6) δ 8.55 (br s, 1H), 7.43-7.35 (m, 4H), 7.32-7.27 (m, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 2.34 (s, 3H), 0.93 (t, *J* = 8.0 Hz, 9H), 0.73 (q, *J* = 8.0 Hz, 6H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 214.4, 193.5, 157.8, 136.5, 130.4, 129.1, 128.0 (2C), 126.6, 116.1, 107.0, 87.9, 30.0, 7.4, 3.9 ppm.

IR (thin film) 3447, 2955, 1662, 1507, 1227, 1119, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{23}H_{28}O_2SSi$ [M^+]: 396.1579, Found: 396.1574.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-1-phenylhepta-1,2-dien-3-yl) ethanethioate (5m) was prepared as brown oil from substrate **1u** (0.3 mmol, 84.0 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 96.2 mg, 95% yield, 92% ee).



$[\alpha]_D^{25}$: –13.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.2 min (minor), 9.4 min (major).

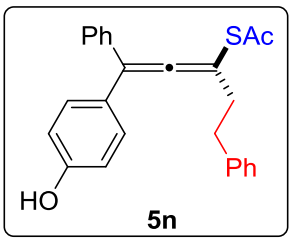
^{1}H NMR (400 MHz, acetone-d_6) δ 8.56 (br s, 1H), 7.42-7.35 (m, 4H), 7.35-7.29 (m, 1H), 7.21 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 2.44 (t, *J* = 7.2 Hz, 2H), 2.35 (s, 3H), 1.61-1.52 (m, 2H), 1.39-1.28 (m, 2H), 0.85 (t, *J* = 7.2 Hz, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 208.1, 194.0, 158.0, 136.7, 130.5, 129.3, 129.1, 128.4, 126.9, 116.1, 111.2, 98.9, 35.6, 30.5, 30.0, 22.5, 13.9 ppm.

IR (thin film) 3445, 3029, 2927, 1674, 1507, 1220, 1115, 832, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{22}O_2S$ [M^+]: 338.1341, Found: 338.1338.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-1,5-diphenylpenta-1,2-dien-3-yl) ethanethioate (5n)



5n

was prepared as brown oil from substrate **1v** (0.3 mmol, 98.4 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 113.6 mg, 98% yield, 94% ee).

$[\alpha]_D^{25}$: –20.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 17.9 min (minor), 19.1 min (major).

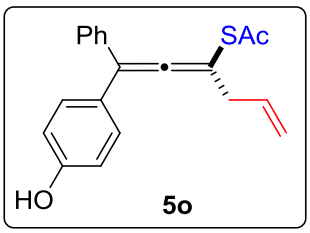
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.36-7.29 (m, 3H), 7.27-7.16 (m, 7H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 5.49 (br s, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.77 (t, *J* = 8.0 Hz, 2H), 2.38 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.3, 195.5, 156.1, 141.4, 136.1, 130.6, 129.2, 128.9, 128.8, 128.7, 128.1, 127.7, 126.4, 115.7, 111.2, 98.1, 37.3, 34.3, 30.6 ppm.

IR (thin film) 3405, 1674, 1510, 1267, 1115, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{25}H_{22}O_2S$ [M^+]: 386.1341, Found: 386.1331.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-1-phenylhexa-1,2,5-trien-3-yl) ethanethioate (5o) was



5o

prepared as brown oil from propargylic substrate **1w** (0.3 mmol, 79.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 90.2 mg, 92% yield, 93% ee).

$[\alpha]_D^{25}$: –17.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 10.7 min (minor), 12.6 min (major).

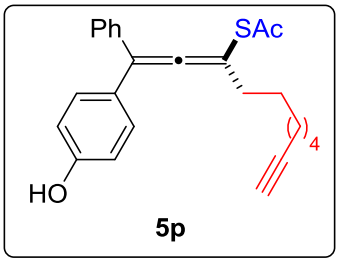
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.37-7.28 (m, 5H), 7.23 (d, J = 8.8 Hz, 2H), 6.83 (d, J = 8.8 Hz, 2H), 5.97-5.87 (m, 1H), 5.62 (br s, 1H), 5.20-5.09 (m, 2H), 3.19 (d, J = 6.8 Hz, 2H), 2.36 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.6, 195.0, 156.2, 136.1, 134.8, 130.6, 129.2, 128.8, 128.3, 127.8, 117.5, 115.8, 111.3, 97.1, 40.1, 30.5 ppm.

IR (thin film) 3315, 3078, 1675, 1510, 1269, 1116, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{18}O_2S$ [M^+]: 322.1028, Found: 322.1017.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-1-phenylundeca-1,2-dien-10-yn-3-yl) ethanethioate (5p)



5p

was prepared as brown oil from substrate **1x** (0.3 mmol, 99.8 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 75.5 mg, 64% yield, 91% ee).

$[\alpha]_D^{25}$: –17.7 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 11.9 min (minor), 14.9 min (major).

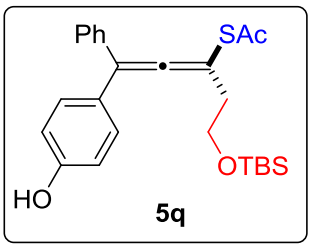
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.42-7.34 (m, 4H), 7.34-7.27 (m, 1H), 7.24 (d, J = 8.8 Hz, 2H), 6.85 (d, J = 8.8 Hz, 2H), 2.45 (t, J = 7.2 Hz, 2H), 2.37 (s, 3H), 2.13-2.08 (3, 2H), 1.97-1.95 (m, 1H), 1.64-1.54 (m, 2H), 1.47-1.27 (m, 6H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.0, 195.7, 156.2, 136.3, 130.5, 129.1, 128.8, 128.2, 127.8, 115.8, 111.0, 98.6, 85.1, 68.4, 35.7, 30.5, 28.86, 28.85, 28.8, 28.1, 18.6 ppm.

IR (thin film) 3629, 3295, 2932, 1683, 1508, 1267, 1118, 833, 769 cm^{-1}.

HRMS (CI+) Calcd for $C_{25}H_{26}O_2S$ [M^+]: 390.1654, Found: 390.1671.

(*R*)-*S*-(5-((Tert-butyldimethylsilyl)oxy)-1-(4-hydroxyphenyl)-1-phenylpenta-1,2-dien-3-yl) ethanethioate (5q) was prepared as brown oil from substrate **1y** (0.3 mmol, 114.6 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 108.6 mg, 82% yield, 93% ee).



$[\alpha]_D^{25}$: –16.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALCEL OD-H column; 5% *i*-PrOH in hexanes; 0.5 mL/min; retention times: 13.1 min (minor), 14.2 min (major).

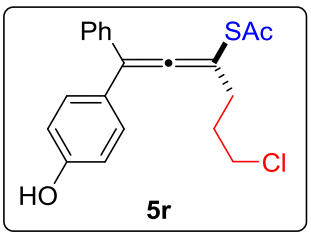
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.35 (d, *J* = 8.0 Hz, 4H), 7.34-7.27 (m, 1H), 7.24 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 3.84 (t, *J* = 6.4 Hz, 2H), 2.66 (t, *J* = 6.4 Hz, 2H), 2.36 (s, 3H), 0.86 (s, 9H), 0.03 (s, 6H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.8, 195.2, 156.2, 136.1, 130.7, 129.3, 128.8, 128.2, 127.8, 115.8, 110.7, 95.8, 61.5, 39.0, 30.5, 26.1, 18.6, –5.2 ppm.

IR (thin film) 3340, 2939, 1698, 1508, 1259, 1103, 835 cm^{-1}.

HRMS (CI+) Calcd for $C_{25}H_{32}O_3SSi$ [M^+]: 440.1841, Found: 440.1831.

(*R*)-*S*-(6-Chloro-1-(4-hydroxyphenyl)-1-phenylhexa-1,2-dien-3-yl) ethanethioate (5r)



was prepared as brown oil from substrate **1z** (0.3 mmol, 90.3 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 103.2 mg, 96% yield, 93% ee).

$[\alpha]_D^{25}$: –24.9 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.3 min (minor), 8.0 min (major).

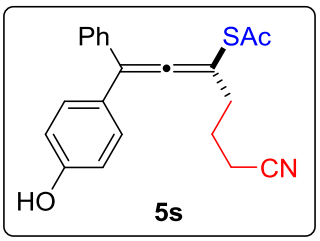
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.41-7.30 (m, 5H), 7.23 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.78 (br s, 1H), 3.55 (t, *J* = 6.8 Hz, 2H), 2.60 (t, *J* = 6.8 Hz, 2H), 2.38 (s, 3H), 2.10-2.02 (m, 2H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.1, 195.5, 156.3, 136.0, 130.5, 129.1, 128.9, 128.3, 127.6, 115.8, 111.5, 97.3, 44.6, 33.1, 31.2, 30.6 ppm.

IR (thin film) 3421, 3029, 2926, 1675, 1511, 1269, 1118, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{19}{}^{35}ClO_2S$ [M^+]: 358.0794, Found: 358.0779.

(*R*)-*S*-(6-Cyano-1-(4-hydroxyphenyl)-1-phenylhexa-1,2-dien-3-yl) ethanethioate (5s)



was prepared as brown oil from substrate **1aa** (0.3 mmol, 87.5 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 81.6 mg, 78% yield, 92% ee).

$[\alpha]_D^{25}$: –10.7 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 13.3 min (minor), 15.3 min (major).

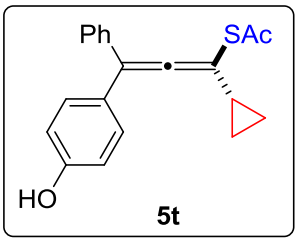
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.41-7.30 (m, 5H), 7.21 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.87 (br s, 1H), 2.57 (t, *J* = 7.2 Hz, 2H), 2.76 (s, 3H), 2.35 (t, *J* = 7.2 Hz, 2H), 1.97-1.89 (m, 2H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.2, 195.0, 156.5, 135.8, 130.5, 129.1, 128.9, 128.4, 127.3, 119.8, 115.9, 111.8, 96.8, 34.7, 30.5, 24.2, 16.7 ppm.

IR (thin film) 3227, 1610, 1511, 1269, 1114, 771 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{19}NO_2S$ [M^+]: 349.1136, Found: 349.1135.

(*R*)-*S*-(1-Cyclopropyl-3-(4-hydroxyphenyl)-3-phenylpropa-1,2-dien-1-yl) et



hanethioate (5t) was prepared as brown oil from substrate **1ab** (0.3 mmol, 79.2 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 81.9 mg, 85% yield, 92% ee).

$[\alpha]_D^{25}$: –44.3 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H; 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 24.8 min (minor), 26.1 min (major).

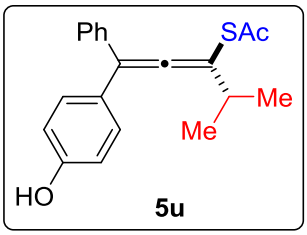
^{1}H NMR (400 MHz, acetone-d_6) δ 8.59 (br s, 1H), 7.42-7.30 (m, 5H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 2.37 (s, 3H), 1.73-1.65 (m, 1H), 0.82-0.78 (m, 2H), 0.59-0.49 (m, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 207.9, 193.9, 158.2, 136.6, 130.6, 129.3, 129.2, 128.5, 126.8, 116.2, 112.1, 101.7, 30.0, 15.9, 7.48, 7.46 ppm.

IR (thin film) 3418, 3015, 2963, 1691, 1507, 1266, 1119, 832, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{18}O_2S$ [M$^+$]: 322.1028, Found: 322.1016.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-4-methyl-1-phenylpenta-1,2-dien-3-yl) ethanethioate



(5u) was prepared as brown oil from substrate **1ac** (0.3 mmol, 79.8 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 87.8 mg, 90% yield, 92% ee).

$[\alpha]_D^{25}$: –12.1 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 2% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 19.9 min (minor), 21.8 min (major).

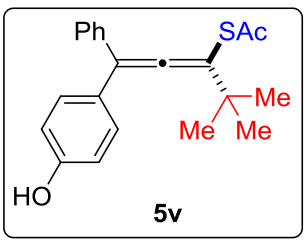
^{1}H NMR (400 MHz, acetone-d_6) δ 8.56 (br s, 1H), 7.48-7.38 (m, 4H), 7.38-7.28 (m, 1H), 7.22 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 2.98-2.51 (m, 1H), 2.36 (s, 3H), 1.18-1.15 (m, 6H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 207.4, 194.0, 158.0, 136.7, 130.5, 129.2, 129.1, 128.3, 126.9, 116.1, 111.9, 105.0, 35.2, 30.0, 21.7 ppm.

IR (thin film) 3415, 2965, 1671, 1508, 1222, 1115, 832, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{20}H_{20}O_2S$ [M$^+$]: 324.1184, Found: 324.1183.

(*R*)-*S*-(1-(4-Hydroxyphenyl)-4,4-dimethyl-1-phenylpenta-1,2-dien-3-yl) ethanethioate (5v) was prepared as brown oil from substrate **1ad** (0.3 mmol, 84.5 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 90.2 mg, 89% yield, 94% ee).



$[\alpha]_D^{25}$: –35.2 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.9 min (minor), 8.9 min (major).

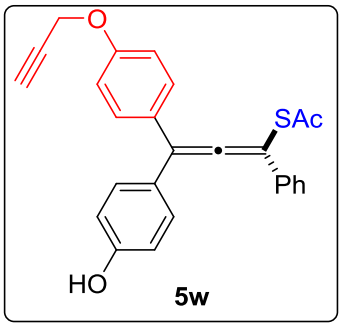
^{1}H NMR (400 MHz, acetone-d_6) δ 8.55 (br s, 1H), 7.44-7.36 (m, 4H), 7.34-7.28 (m, 1H), 7.25 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 2.35 (s, 3H), 1.18 (s, 9H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 208.1, 193.3, 157.9, 136.8, 130.5, 129.2, 129.1, 128.2, 127.0, 116.1, 110.7, 107.2, 37.9, 29.8, 28.9 ppm.

IR (thin film) 3446, 2966, 1674, 1508, 1222, 1118, 759 cm^{-1}.

HRMS (CI+) Calcd for $C_{21}H_{22}O_2S$ [M$^+$]: 338.1341, Found: 338.1357.

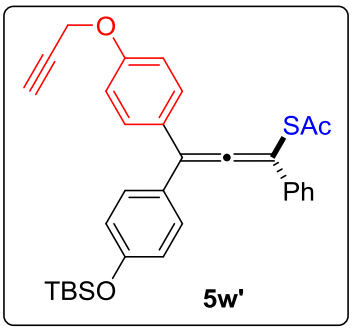
(*S*)-S-(3-(4-Hydroxyphenyl)-1-phenyl-3-(4-(prop-2-yn-1-yloxy)phenyl)propa-1,2-dien-1-yl) ethanethioate (5w) was prepared as brown oil from substrate **1ae** (0.3 mmol, 106.2 mg) according to the General Procedure D (purified by flash column chromatography: 20% EtOAc and 1% AcOH in hexanes, 62.8 mg, 51% yield, 93% ee).



5w

$[\alpha]_D^{25}$: –24.1 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 20% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 9.1 min (minor), 14.9 min (major).

The pure form of **5w** is relatively unstable, but it can be stored as a solution in DCM or $CHCl_3$ in the refrigerator. However, its TBS-protected derivative **5w'** is stable. Therefore, the characterization of **5w'** is provided.



5w'

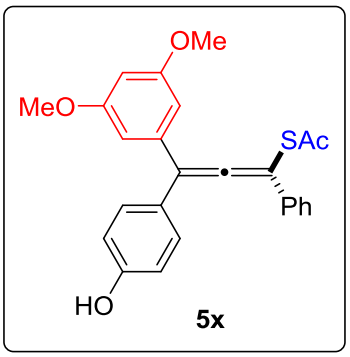
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 8.0 Hz, 2H), 7.41-7.23 (m, 7H), 6.99 (d, J = 8.8 Hz, 2H), 6.85 (d, J = 8.8 Hz, 2H), 4.71 (s, 2H), 2.54 (s, 1H), 2.43 (s, 3H), 1.0 (s, 9H), 0.23 (s, 6H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.4, 193.9, 157.5, 155.8, 134.5, 130.1, 130.0, 128.6, 128.0, 127.8, 127.3, 126.4, 120.2, 115.0, 112.0, 99.1, 78.4, 75.7, 55.8, 30.1, 25.6, 18.2, 4.4 ppm.

IR (thin film) 3443, 3294, 2930, 1705, 1603, 1507, 1261, 1029, 909, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{32}H_{34}O_3SSi$ [M$^+$]: 526.1998, Found: 526.2009.

(*S*)-*S*-(3-(3,5-Dimethoxyphenyl)-3-(4-hydroxyphenyl)-1-phenylpropa-1,2-dien-1-yl) ethanethioate (5x) was prepared as brown oil from substrate **1af** (0.3 mmol, 108.1 mg) according to the General Procedure D (purified by flash column chromatography: 20% EtOAc and 1% AcOH in hexanes, 80.3 mg, 64% yield, 92% ee).



5x

$[\alpha]_D^{25}$: –47.3 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 15% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.5 min (minor), 16.5 min (major).

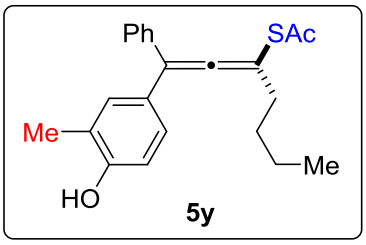
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.61 (d, *J* = 7.2 Hz, 2H), 7.36 (t, *J* = 7.2 Hz, 2H), 7.31-7.27 (m, 3H), 6.82 (d, *J* = 8.4 Hz, 2H), 6.61-6.05 (m, 2H), 6.47-6.45 (m, 2H), 3.77 (s, 6H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 211.6, 194.7, 161.3, 156.5, 137.4, 134.6, 130.7, 129.1, 128.4 (2C), 126.7, 115.9, 112.8, 107.3, 100.6, 99.7, 55.8, 30.5 ppm.

IR (thin film) 3474, 2961, 1680, 1593, 1511, 1204, 1155, 837 cm^{-1}.

HRMS (CI+) Calcd for $C_{25}H_{22}O_4S$ [M^+]: 418.1239, Found: 418.1237.

(*R*)-*S*-(1-(4-Hydroxy-3-methylphenyl)-1-phenylhepta-1,2-dien-3-yl) ethanethioate (5y) was prepared as brown oil from substrate **1ag** (0.3 mmol, 88.4 mg) according to the General Procedure D (purified by flash column chromatography: 10% EtOAc and 1% AcOH in hexanes, 98.3 mg, 93% yield, 91% ee).



5y

$[\alpha]_D^{25}$: –22.0 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 7.6 min (minor), 9.0 min (major).

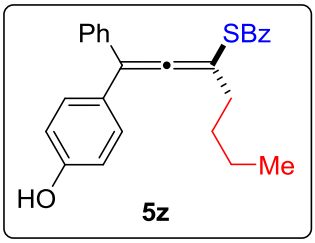
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.37 (d, *J* = 8.4 Hz, 4H), 7.34-7.28 (m, 1H), 7.13-7.12 (m, 1H), 7.08-7.05 (m, 1H), 6.77 (d, *J* = 8.4 Hz, 1H), 2.44 (t, *J* = 7.6 Hz, 2H), 2.37 (s, 3H), 2.23 (s, 3H), 1.62-1.53 (m, 2H), 1.36 (q, *J* = 7.6 Hz, 2H), 0.89 (t, *J* = 7.6 Hz, 3H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 208.0, 195.3, 154.4, 136.5, 131.7, 129.2, 128.8, 128.1, 128.0, 127.8, 124.5, 115.3, 111.0, 98.7, 35.6, 30.52, 30.50, 22.6, 16.0, 14.0 ppm.

IR (thin film) 3400, 3023, 2928, 1685, 1503, 1278, 1116, 954, 824, 758 cm^{-1}.

HRMS (CI+) Calcd for $C_{22}H_{24}O_2S$ [M^+]: 352.1497, Found: 352.1501.

(*R*)-S-(1-(4-Hydroxyphenyl)-1-phenylhepta-1,2-dien-3-yl) benzothioate (5z) was



prepared as pale yellow solid from substrate **1u** (0.3 mmol, 84.5 mg) according to the General Procedure D (catalyst (*R*)-**A7** instead of catalyst (*S*)-**B1** and thiobenzoic acid instead of thioacetic acid, purified by flash column chromatography: 8% EtOAc and 2% AcOH in hexanes, 97.2 mg, 81% yield, 90% ee). $[\alpha]_D^{25}$: +27.5 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 11.3 min (major), 15.9 min (minor).

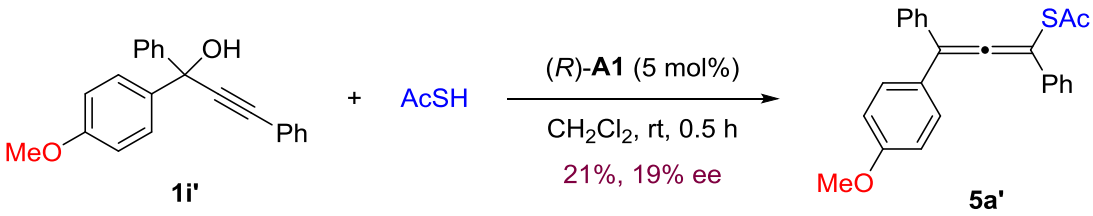
^{1}H NMR (400 MHz, acetone-d_6) δ 8.15 (br s, 1H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 2H), 7.42-7.34 (m, 4H), 7.34-7.31 (m, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 2.55 (t, *J* = 7.6 Hz, 2H), 1.68-1.59 (m, 2H), 1.44-1.33 (m, 2H), 0.88 (t, *J* = 7.6 Hz, 3H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 208.7, 190.4, 158.2, 137.5, 136.7, 134.5, 130.6, 129.7, 129.4, 129.2, 128.5, 127.9, 126.9, 116.2, 111.5, 98.2, 35.7, 30.7, 22.6, 14.0 ppm.

IR (thin film) 3416, 2929, 1669, 1510, 1206, 904, 767 cm^{-1}.

HRMS (CI+) Calcd for $C_{26}H_{24}O_2S$ [M^+]: 400.1497, Found: 400.1505.

Supplementary Note 1. Mechanistic Study



***S*-(3-(4-methoxyphenyl)-1,3-diphenylpropa-1,2-dien-1-yl) ethanethioate (5a')** was prepared as red oil from propargylic alcohol **1i'** (94.3 mg, 0.3 mmol) according to the General Procedure D (with CH_2Cl_2 as solvent, and without 3Å MS, room temperature for 0.5 h (21% ^{1}H NMR yield) or 72 h (full conversion), purified by flash column chromatography: 10% EtOAc in hexanes, 74.8 mg, 67% yield, 19% ee).

$[\alpha]_D^{25}$: +4.2 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.5 min (major), 10.0 min (minor).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.41-7.33 (m, 7H), 7.29-7.24 (m, 1H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.4, 193.7, 159.6, 135.0, 134.3, 120.1, 128.9, 128.6, 128.1, 127.9, 126.7, 126.4, 114.1, 112.4, 99.3, 55.3, 30.1 ppm.

IR (thin film) 3057, 2932, 1605, 1509, 1444, 1250, 1178, 1034, 832, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{24}H_{20}O_2S$ [M^+]: 372.1184, Found: 372.1182.

4-(1,3-Diphenylprop-2-yn-1-ylidene)cyclohexa-2,5-dienone (QM). To a solution of substrate **1i** (300.4 mg, 1.0 mmol) in CCl_4 (10 mL) were added 3Å MS (300 mg) and (*rac*)-**A7** (17.4 mg, 0.05 mmol) or (*S*)-**B1** (42.5 mg, 0.05 mmol). The reaction was stirred at room temperature for 1-2 h to give *para*-quinone methide **QM** as a red solid

(purified by flash column chromatography: 5% EtOAc in hexanes, 211.8 mg, 75% yield for **A7**; 104.5 mg, 37% yield for **B1**).

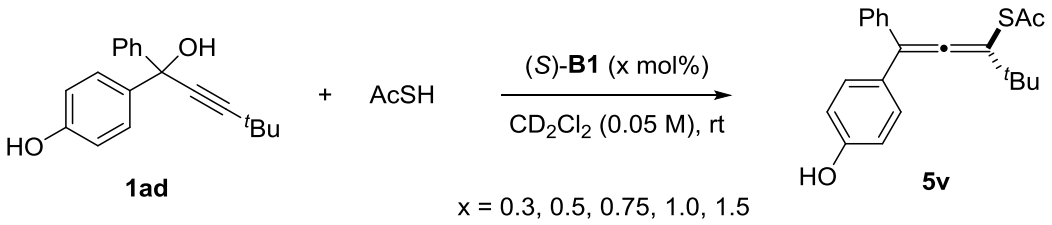
^{1}H NMR (400 MHz, acetone-d_6) δ 8.18 (dd, J = 2.4 Hz, J = 10 Hz, 1H), 7.68-7.62 (m, 4H), 7.58-7.52 (m, 3H), 7.51-7.44 (m, 4H), 6.48 (dd, J = 2.0 Hz, J = 10 Hz, 1H), 6.32 (dd, J = 2.0 Hz, J = 10 Hz, 1H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ 186.7, 138.6, 138.1, 137.4, 136.5, 135.6, 132.7, 130.93, 130.89, 130.7, 130.0, 129.6, 129.5, 129.2, 122.6, 108.2, 90.4 ppm.

IR (thin film) 2976, 2180, 1625, 1501, 1444, 1166, 864, 698 cm^{-1}.

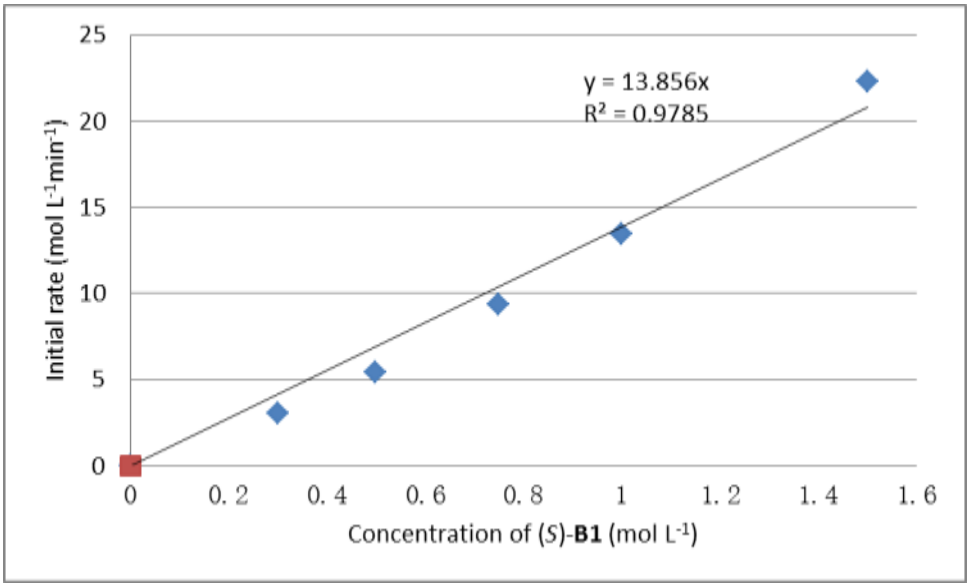
HRMS (CI+) Calcd for $C_{21}H_{14}O$ [M^+]: 282.1045, Found: 282.1058.

Kinetic Study



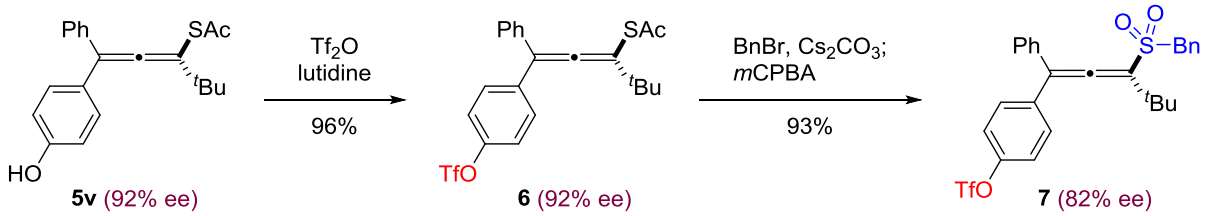
At room temperature, to an oven-dried 4-mL vial charged with a solution of propargylic alcohol **1ad** (14 mg, 0.05 mmol, 1.0 equiv.) in CD_2Cl_2 (1.0 mL) was added a solution of the catalyst (*S*)-**B1** (1st run: 0.13 mg, 0.15 μmol, 0.3 mol%; 2nd run: 0.21 mg, 0.25 μmol, 0.5 mol%; 3rd run: 0.32 mg, 0.38 μmol, 0.75 mol%; 4th run: 0.43 mg, 0.5 μmol, 1.0 mol%; 5th run: 0.64 mg, 0.75 μmol, 1.5 mol%) and thioacetic acid (7.2 uL, 0.1 mmol, 2.0 equiv.) in CD_2Cl_2 (50 uL). The homogeneous reaction mixture was transferred to an NMR tube. The progress of the five reactions was monitored by ^{1}H NMR every 2 min.

As shown in Supplementary Figure 219, a linear relationship between the initial rates and catalyst loading was observed, indicating 1st-order kinetics in catalyst.



Supplementary Figure 219. First-order kinetics in catalyst.

Supplementary Note 2. Product Derivatizations



(*R*)-*S*-(4,4-Dimethyl-1-phenyl-1-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)penta-1, 2-dien-3-yl) ethanethioate (6). At 0 °C, to a solution of **5v** (338.2 mg, 1.0 mmol) in CH_2Cl_2 (10 mL) were added 2,6-lutidine (419.3 uL, 3.6 mmol, 3.6 equiv) and a solution of Tf_2O (370.1 uL, 2.2 mmol, 2.2 equiv) in CH_2Cl_2 (0.5 mL) sequentially. The reaction mixture was stirred at 0 °C for 10 min and then allowed to warm to room temperature. The mixture was concentrated, and the residue was purified by silica gel column chromatography (eluent: 3% Et_2O in hexanes, colorless oil, 451.7 mg, 96 yield, 92% ee).

$[\alpha]_D^{25}$: +22.8 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALCEL OD-H column; 1% *i*-PrOH in hexanes; 0.5 mL/min; retention times: 7.8 min (major), 8.6 min (minor).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 8.8 Hz, 2H), 7.42-7.31 (m, 5H), 7.27 (d, J = 8.8 Hz, 2H), 2.37 (s, 3H), 1.19 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.4, 192.9, 148.7, 136.4, 135.1, 130.3, 128.64, 128.62, 127.9, 121.3, 118.8 (d, $J_{C\text{-}F}$ = 319 Hz), 109.0, 107.8, 37.4, 30.1, 28.8 ppm.

^{19}F NMR (376 MHz, $CDCl_3$) δ –72.9 ppm.

IR (thin film) 3521, 2967, 2882, 1708, 1426, 1214, 1142, 887 cm^{-1}.

HRMS (CI+) Calcd for $C_{22}H_{25}F_3NO_4S_2$ [M+NH_4^+]: 488.1177, Found: 488.1164.

(*R*)-4-(3-(Benzylsulfonyl)-4,4-dimethyl-1-phenylpenta-1,2-dien-1-yl)phenyltrifluoromethanesulfonate (7). At room temperature, to a stirred solution of Cs_2CO_3 (117.6 mg, 0.36 mmol, 1.2 equiv) in MeOH (6.0 mL) was added a solution of **6** (141.2 mg, 0.3

mmol, 1.0 equiv) and benzyl bromide (71.3 uL, 0.6 mmol, 2.0 equiv) in MeOH (0.3 mL) dropwise. The mixture was stirred for 20 min. Next, Et_2O (10 mL) was added, and the mixture was filtered through a short pad of silica gel, which was washed with Et_2O (20 mL). The filtrate was concentrated to the crude product as a pale green oil, which was used for the next step without further purification.

At room temperature, the above oil was dissolved in CH_2Cl_2 (6 mL) and then treated with *m*CPBA (151.9 mg, 0.66 mmol, 75 wt%, 2.2 equiv). The reaction mixture was stirred for 5 min before a saturated aqueous $NaHCO_3$ solution (10 mL) was added. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3×15 mL). The combined organic layers were washed with a saturated aqueous Na_2SO_3 solution, a saturated aqueous $NaHCO_3$ solution, and brine, dried over Na_2SO_4, and concentrated. The residue was purified by silica gel flash column chromatography (5% Et_2O in hexanes) to give pure sulfone **7** (153.6 mg, 93% yield, 82% ee).

$[\alpha]_D^{25}$: –44.8 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 15.9 min (major), 17.6 min (minor).

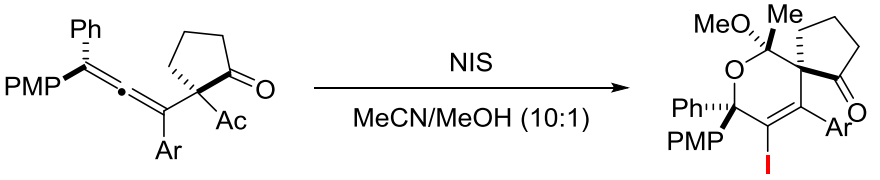
^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.50-7.43 (m, 5H), 7.38-7.31 (m, 4H), 7.27-7.17 (m, 3H), 7.13-7.10 (m, 2H), 4.26 (s, 2H), 1.33 (s, 9H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 206.0, 149.9, 135.6, 134.2, 131.3, 130.9, 129.5, 129.1, 129.0, 128.8, 128.1, 124.1, 122.2, 119.2 (d, $J_{C\text{-}F}$ = 319 Hz), 117.1 (2C), 63.5, 37.5, 29.8 ppm.

^{19}F NMR (376 MHz, CD_2Cl_2) δ –73.2 ppm.

IR (thin film) 3444, 2970, 1934, 1498, 1425, 1313, 1214, 1141, 1016, 887, 698 cm^{-1}.

HRMS (CI+) Calcd for $C_{27}H_{29}F_3NO_5S_2$ [M+NH_4^+]: 568.1439, Found: 568.1470.



3b
(7:1 d.r., 94% *ee*)

8
(7:1 d.r., 93% *ee*)

PMP = *p*-(MeO)C_6H_4, Ar = *p*-ClC_6H_4

(5*R*,6*S*,8*R*)-10-(4-Chlorophenyl)-9-iodo-6-methoxy-8-(4-methoxyphenyl)-6-methyl-8-phenyl-7-oxaspiro[4.5]dec-9-en-1-one (8). Under N_2, the tetrasubstituted allene **3b** (91.2 mg, 0.2 mmol, 1 equiv) was dissolved in anhydrous MeCN/MeOH (10:1, 4 mL) and then *N*-iodosuccinimide (54.0 mg, 0.24mmol, 1.2 equiv) was added. After stirring at room temperature for 5 h, the reaction mixture was subjected to flash chromatography to give the desired product **8** as a white foam (eluent: 10% EtOAc in hexanes, 105.7 mg, 86% yield, 7:1 d.r., 93% ee).

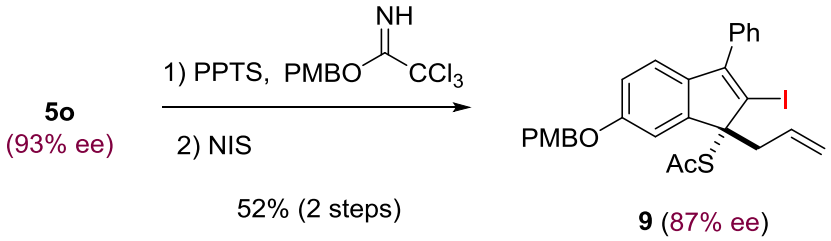
$[\alpha]_D^{25}$: –26.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 5.9 min (major), 8.2 min (minior). Daicel CHIRALPAK IC; 5% *i*-PrOH in hexanes; 0.5 mL/min; retention times: 8.9 (major), 11.9 min (minior).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.90 (d, *J* = 7.6 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.38-7.25 (m, 5H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.70 (d, *J* = 8.8 Hz, 1H), 3.84 (s, 3H), 2.35-2.08 (m, 4H), 2.26 (s, 3H), 1.63-1.54 (m, 1H), 1.28 (s, 3H), 0.59-0.45 (m, 1H) ppm.

^{13}C NMR (100 MHz, CD_2Cl_2) δ 217.6, 159.2, 145.9, 143.7, 136.2, 134.1, 133.4, 132.1, 131.6, 130.3, 129.3, 128.0, 127.1, 112.8, 112.3, 102.4, 83.8, 63.1, 55.8, 48.4, 40.8, 32.9, 19.6, 19.0 ppm.

IR (thin film) 2963, 1736, 1608, 1509, 1376, 1249, 1140, 998, 829, 700 cm^{-1}.

HRMS (CI+) Calcd for $C_{30}H_{28}{}^{35}ClIO_4$ [M^+]: 614.0721, Found: 614.0725.



(*R*)-*S*-(1-Allyl-2-iodo-6-((4-methoxybenzyl)oxy)-3-phenyl-1H-inden-1-yl) ethanethioate (9). At room temperature, to a solution of **5o** (64.5 mg, 0.2 mmol) and *p*-methoxybenzyl trichloroacetimidate (PMBTCA)[7] in CCl_4 (2 mL) was added PPTS (25.1 mg, 0.1 mmol, 0.5 equiv). The mixture was stirred for 18 h, and then a saturated aqueous solution of $NaHCO_3$ (2 mL) was added. The layers were separated, and the aqueous layer was extracted with ethyl acetate (2 mL×3). The combined organic layers were concentrated. The residue was dissolved in DCM and then triturated with hexanes. The resulting mixture was filtered and the filtrate was concentrated. The residue was purified by silica gel column chromatography (eluent: 10% DCM and 1% Et_2O in hexanes) to give a pale yellow oil (75% yield based on ^1H NMR).

To a solution of *N*-iodosuccinimide (40.5 mg, 0.18 mmol, 1.2 equiv) in nitromethane (1 mL) was added a solution of the above oil (0.15 mmol) in nitromethane (0.5 mL). The mixture was stirred at room temperature for 30 min, and then a saturated solution of sodium thiosulfate (1 mL) was added to quench the reaction. The mixture was extracted with ethyl acetate (2×20 mL). The combined organics layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated. The residue was purified by flash chromatography (eluent: 10% DCM and 1% Et_2O in hexanes) to get the iodide **9** as a colorless oil (59.2 mg, 52% yield, 87% ee).

$[\alpha]_D^{25}$: –31.1 (*c* = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 8.0 min (minor), 12.6 min (major).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.37 (m, 8H), 7.06 (d, J = 8.4 Hz, 1H), 6.93 (d, J = 8.8 Hz, 2H), 6.84 (d, J = 8.8 Hz, 1H), 5.23-5.12 (m, 1H), 5.06 (s, 1H), 5.02 (s, 2H), 4.92 (d, J = 10 Hz, 1H), 3.82 (s, 3H), 3.12-3.06 (m, 1H), 2.74-2.67 (m, 1H), 2.21 (s, 3H) ppm.

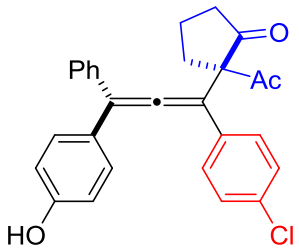
^{13}C NMR (100 MHz, $CDCl_3$) δ 193.2, 159.5, 157.8, 150.0, 148.3, 137.1, 135.1, 130.1, 129.4, 128.8, 128.4, 128.3 (2C), 120.8, 119.3, 114.0, 113.7, 111.3, 103.7, 70.1, 66.3, 55.3, 40.0, 30.7 ppm.

IR (thin film) 3445, 1688, 1514, 1424, 1248, 1140, 1018, 824, 699 cm^{-1}.

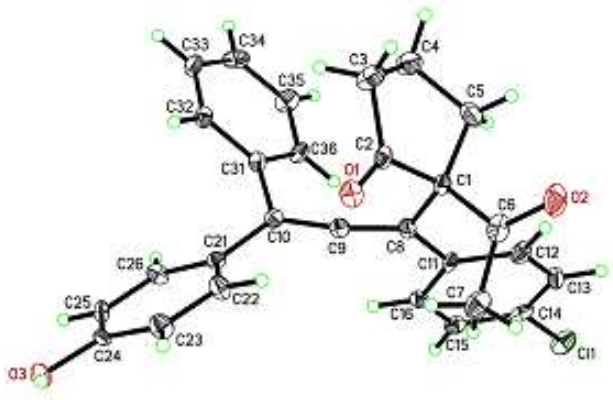
HRMS (CI+) Calcd for $C_{28}H_{25}IO_3S$ [M^+]: 568.0569, Found: 568.0558.

Supplementary Note 3. Determination of the Absolute Stereochemistry

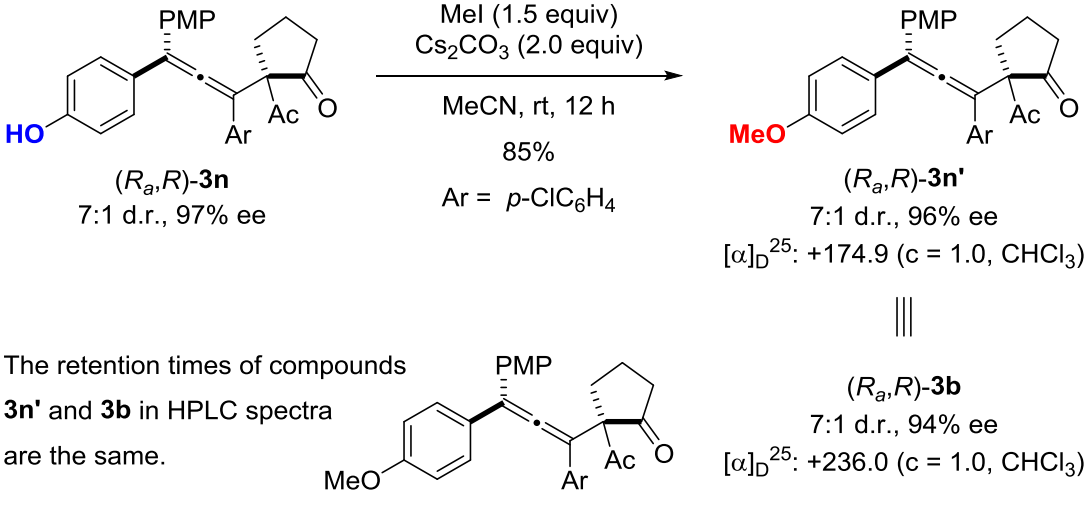
(1) The absolute stereochemistry of product **3n** was determined by X-ray diffraction (shown below). The X-ray data of **3n** have been deposited at the Cambridge Crystallographic Data Center (CCDC 1498348). The stereochemistry of other products was assumed by analogy.



3n

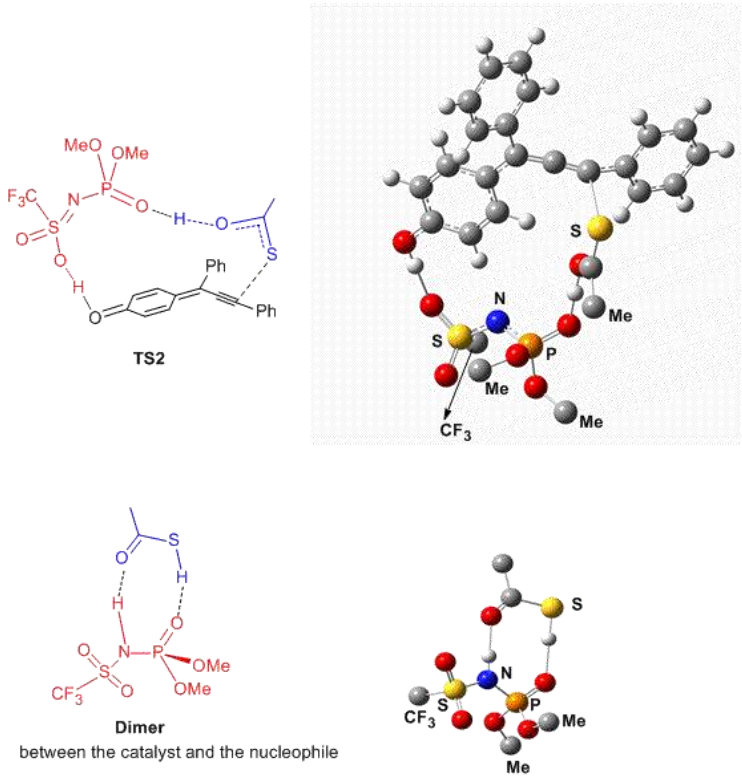


(2) The absolute stereochemistry of product **3b** was determined by comparing the optical rotation and HPLC spectra with that of **3n′** (the methyl-protected derivative of **3n**).



Supplementary Note 4. DFT Calculations

Using DFT calculations on a model system (Figure S1), we were able to locate the important transition state (**TS2**, as discussed in the main text) featuring a nucleophilic attack leading to the C-S bond formation. This transition state lies above the energy reference point (the substrate + a dimer formed between the catalyst and the nucleophile) by 11.4 kcal/mol in free energy, which is consistent with the experimental observations that the reaction can take place smoothly at room temperature. It is also consistent with the kinetic study and the proposed mechanism that only one catalyst molecule is sufficient to activate both of the reaction partners through the bifunctional model.



Supplementary Figure 220. The structures calculated for TS2 and the dimer formed between the catalyst and nucleophile.

In the DFT calculations, the Becke three-parameter hybrid functional (B3LYP) method was employed.[8-10] The effective core potentials (ECPs) of Lanl2dz were used to describe S and P atoms,[11] with polarization functions for P (ζ_d =0.387) and S (ζ_d

=0.503) being added.[12] The two H atoms, which were related to prototropic, were described by the 6-31G** basis set. [13] The 6-31g* basis set was used for all remaining atoms. [14,15] Frequency calculations were performed to ensure that a transition state has only one imaginary frequency and the local minimum has no imaginary frequency. Intrinsic reaction coordinates (IRC) [16,17] were run to ensure that transitions states connect the relevant minima. All the calculations were performed in the Gaussian 09 program. [18]

Cartesian Coordinates for **TS2**, the dimer, and the substrate:

TS2

S 2.593479 -2.070699 1.888858
S -4.259545 1.204648 -0.379173
P -3.127975 -1.107498 0.958979
O -1.480505 4.052451 -0.822059
C -0.300446 1.973829 -0.538138
C 0.909955 1.337247 -0.377785
C 2.134534 2.057259 -0.341916
C 2.073609 3.467074 -0.515740
C 0.866279 4.114674 -0.681041
C -0.342557 3.380109 -0.681789
H -1.222387 1.401401 -0.544359
H 0.924772 0.259383 -0.255531
H 2.991539 4.043691 -0.546892
H 0.818028 5.189280 -0.824347
C 3.381548 1.348362 -0.167519
C 4.642376 2.029028 0.218228
C 4.652894 3.030844 1.206997
C 5.863400 1.638847 -0.362142
C 5.851015 3.627893 1.595026
H 3.724295 3.314584 1.691108
C 7.056203 2.247654 0.018413
H 5.861557 0.868228 -1.127082
C 7.054145 3.243786 0.998775
H 5.844668 4.389712 2.369460
H 7.988611 1.946083 -0.450546
H 7.985892 3.715044 1.298950
C 3.436027 -0.004556 -0.348746
C 3.414341 -1.250568 -0.417756
C 3.646753 -2.497872 -1.091485
C 2.575356 -3.250187 -1.611592
C 4.967534 -2.952209 -1.273027
C 2.829329 -4.424625 -2.312313
H 1.560078 -2.906761 -1.445029
C 5.210577 -4.128549 -1.977736
H 5.789069 -2.374153 -0.861269
C 4.144005 -4.865674 -2.498656
H 2.000584 -4.999625 -2.715498
H 6.231685 -4.471391 -2.118898
H 4.335601 -5.784352 -3.046248
C 0.912002 -2.200349 1.719188
C 0.071542 -2.630026 2.898920
H 0.685415 -2.812536 3.780569
H -0.679663 -1.860978 3.112712
H -0.477191 -3.541855 2.634601
O 0.312698 -1.946097 0.607670
N -3.101790 0.151327 -0.057500
O -3.641031 2.453866 -0.911447
O -5.334501 1.333120 0.625692
C -5.082762 0.492416 -1.912002
O -2.813845 -0.660057 2.503306
O -4.622349 -1.735905 0.981974
O -2.095079 -2.195504 0.653037
F -6.054183 1.317420 -2.316378
F -4.187617 0.356726 -2.895452
F -5.612487 -0.704724 -1.638032
C -4.864851 -2.941072 1.725755

H -4.658491 -2.788208 2.790340
H -5.921333 -3.176315 1.586099
H -4.245211 -3.757544 1.344283
C -3.473382 0.478029 3.089560
H -3.211137 0.464680 4.149384
H -3.115693 1.404094 2.630500
H -4.556869 0.415987 2.965748
H -2.262212 3.444266 -0.818069
H -0.793463 -2.040695 0.637366

Dimer

S 1.427189 -0.975729 0.926824
S -4.151019 0.554918 -0.235269
P 0.147815 1.606739 -0.105825
N 0.155994 -0.019856 0.394482
O 0.842003 -2.097356 1.666482
O 2.496416 -0.119171 1.449527
C 2.080374 -1.720044 -0.677208
O 1.124027 2.441006 0.866520
O 1.025964 1.574703 -1.455523
O -1.262215 2.101171 -0.201140
F 2.952249 -2.680633 -0.365504
F 1.067148 -2.244591 -1.369642
F 2.688135 -0.788924 -1.409841
C 0.736064 2.699111 2.231087
H 1.333897 3.549548 2.562240
H -0.327843 2.945005 2.292621
H 0.967230 1.824415 2.844130
C 1.313066 2.807127 -2.147713
H 1.788301 2.519990 -3.086563
H 0.389886 3.357705 -2.351299
H 1.996155 3.421280 -1.554839
C -3.496564 -1.099881 -0.019684
O -2.313185 -1.357502 0.126018
C -4.546338 -2.190872 -0.065281
H -5.531121 -1.832317 0.245281
H -4.623629 -2.558803 -1.095813
H -4.224579 -3.019449 0.570672
H -0.750622 -0.515196 0.322998
H -2.937045 1.190579 -0.225691

Substrate

O -4.538811 -3.757969 -0.044859
C -2.261254 -3.114073 0.130015
C -1.348996 -2.115267 0.127202
C -1.732703 -0.721982 -0.029844
C -3.144839 -0.449905 -0.238920
C -4.067045 -1.441542 -0.250775
C -3.699778 -2.853196 -0.049351
H -1.971341 -4.151521 0.269321
H -0.294691 -2.333184 0.268727
H -3.450939 0.574114 -0.423235
H -5.118517 -1.237165 -0.430653
C -0.771871 0.284576 -0.013558
C -1.092774 1.739733 -0.011956
C -2.043368 2.275739 0.874641
C -0.403231 2.615049 -0.869497
C -2.313351 3.644116 0.883903
H -2.550112 1.619116 1.575027
C -0.683067 3.979605 -0.866303
H 0.345548 2.213476 -1.545753
C -1.639944 4.499203 0.009731
H -3.044734 4.042133 1.581921
H -0.151833 4.639372 -1.546897
H -1.852335 5.564738 0.016609
C 0.608411 -0.024600 0.006911
C 1.811607 -0.229220 0.018562
C 3.212019 -0.470750 0.036078
C 4.118157 0.598938 0.198438
C 3.717795 -1.780153 -0.107633
C 5.488971 0.360278 0.215326
H 3.731113 1.606842 0.311354
C 5.090464 -2.008089 -0.087925
H 3.023692 -2.605144 -0.234393
C 5.979340 -0.941170 0.072818
H 6.177981 1.190795 0.341177
H 5.469100 -3.020291 -0.198848
H 7.050289 -1.123259 0.087336

Supplementary References

1. Gabbutt, C. D., Heron, B. M., Kilner, C. & Kolla, S. B. The influence of a 1,1-diarylvinyl moiety on the photochromism of naphthopyrans. *Org. Biomol. Chem.* **8**, 4874–4883 (2010).
2. Wei, L.-M., Wei, L.-L., Pan, W.-B. & Wu, M.-J. Synthesis of tetraarylallenes via palladium-catalyzed addition-Elimination reactions of 1,1,3-triaryl-2-propyn-1-ols with aryl iodides. *Synlett* 2219–2223 (2005).
3. Zhu, S., Niljianskul, N. & Buchwald, S. L. A direct approach to amines with remote stereocentres by enantioselective CuH-catalysed reductive relay hydroamination . *Nat. Chem*. **8**, 144–150 (2016).
4. van der Born, D., Sewing, C., Herscheid, J. D. M., Windhorst, A. D., Orru, R. V. A. & Vugts, D. J. A universal procedure for the [^{18}F]trifluoromethylation of aryl iodides and aryl boronic acids with highly improved specific activity. *Angew. Chem. Int. Ed*. **53**, 11046–11050 (2014).
5. Buzas, A. & Gagosz, F. Gold(I) catalyzed isomerization of 5-en-2-yn-1-yl acetates: an efficient access to acetoxy bicyclo[3.1.0]hexenes and 2-cycloalken-1-ones. *J. Am. Chem. Soc.* **128**, 12614–12615 (2006).
6. Hong, X., Küçük, H. B., Maji, M. S., Yang, Y.-F., Rueping, M. & Houk, K. N. Mechanism and selectivity of *N*-triflylphosphoramide catalyzed (3+2) cycloaddition between hydrazones and alkenes. *J. Am. Chem. Soc.* **136**, 13769–13780 (2014).
7. Kato, T., Sato, T., Kashiwagi, Y. & Hosokawa, S. Synthetic studies on Aculeximycin: synthesis of C24–C40 segment by Kobayashi aldolization and epoxide rearrangements. *Org. Lett*. **17**, 2274–2277 (2015).
8. Becke, A. D. Density-functional thermochemistry. III. The role of exact exchange. *J. Chem. Phys.* **98**, 5648–5652 (1993).
9. Lee, C., Yang, W. & Parr, R. G. Development of the Colle-Salvetti correlation-energy formula into a functional of the electron density. *Phys. Rev. B*. **37**, 785-787 (1988).
10. Stephens, P. J., Devlin, F. J., Chabalowski, C. & Frisch, M. J. Ab initio calculation of vibrational absorption and circular dichroism spectra using density functional force fields. *J. Phys. Chem.* **98**, 11623-11627 (1994).
11. Wadt, W. R. & Hay, P. J. Ab initio effective core potentials for molecular calculations. Potentials for main group elements Na to Bi. *J. Chem. Phys.* ***82***, 284-298 (1985).
12. Höllwarth, A., Böhme, M., Dapprich, S., Ehlers, A. W., Gobbi, A., Jonas, V., Köhler, K., Stegmann, R. & Frenking, G. A set of d-polarization functions for pseudo-potential basis sets of the main group elements Al-Bi and f-type polarization functions for Zn, Cd, Hg. *Chem. Phys. Lett*. **208**, 237-240 (1993).

13. Hariharan, P. C. & Pople, J. A. The influence of polarization functions on molecular orbital hydrogenation energies. *Theor. Chim. Acta.* **28**, 213-222 (1973).
14. Petersson, A., Bennett, A., Tensfeldt, T. G., Al-Laham, M. A., Shirley, W. A. & Mantzaris, J. A complete basis set model chemistry. I. The total energies of closed-shell atoms and hydrides of the first-row elements. *J. Chem. Phys.* **89**, 2193-2218 (1988).
15. Petersson, G. A. & Al-Laham, M. A. A complete basis set model chemistry. II. Open-shell systems and the total energies of the first-row atoms. *J. Chem. Phys.* **94**, 6081-6090 (1991).
16. Fukui, K. The path of chemical reactions-the IRC approach. *Acc. Chem. Res.* **14**, 363-368 (1981).
17. Fukui, K. Formulation of the reaction coordinate. *J. Phys. Chem.* **74**, 4161-4163 (1970).
18. Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Montgomery, J. A., Jr., Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, M. J., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C., Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, Ö., Foresman, J. B., Ortiz, J. V., Cioslowski, J. & Fox, D. J. Gaussian 09, Revision D.01, Gaussian, Inc., Wallingford, CT, 2009.