



## Supporting Information

for

### **Diastereoselective synthesis of highly substituted cyclohexanones and tetrahydrochromene-4-ones via conjugate addition of curcumins to arylidenemalonates**

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### **Copies of NMR spectra of all new compounds**

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INN-DN-349-AS-I-1H

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PROCNO 1

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PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
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DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 181  
DW 60.800 usec  
DE 6.50 usec  
TE 298.4 K  
D1 1.00000000 sec  
TD0 1

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PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

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SF 400.1300098 MHz  
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LB 0.30 Hz  
GB 0  
PC 1.00

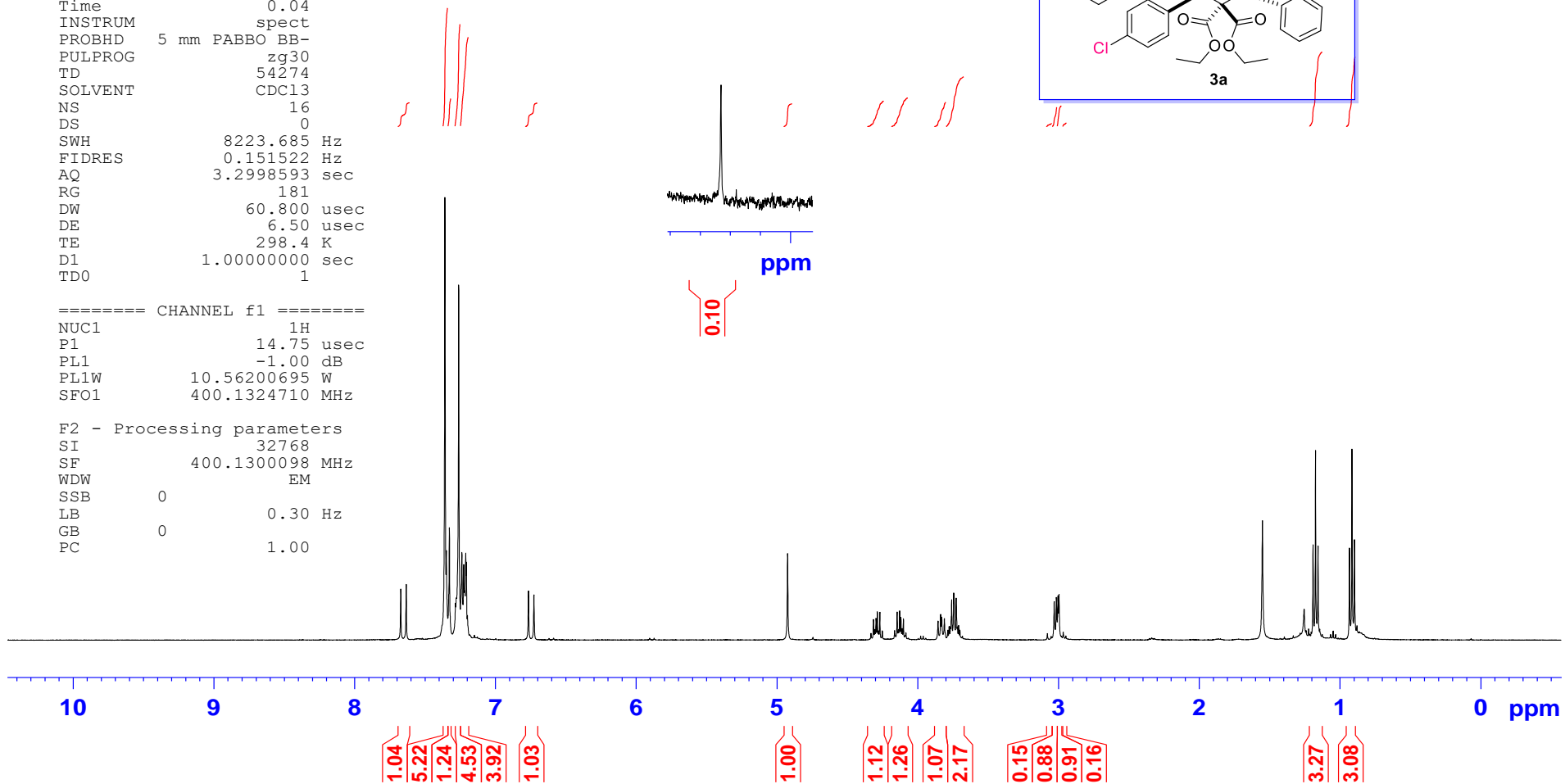
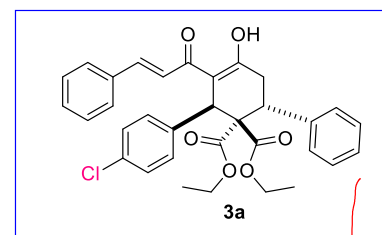


Fig S1. <sup>1</sup>H NMR Spectrum of 3a

INN-DN-349-13C

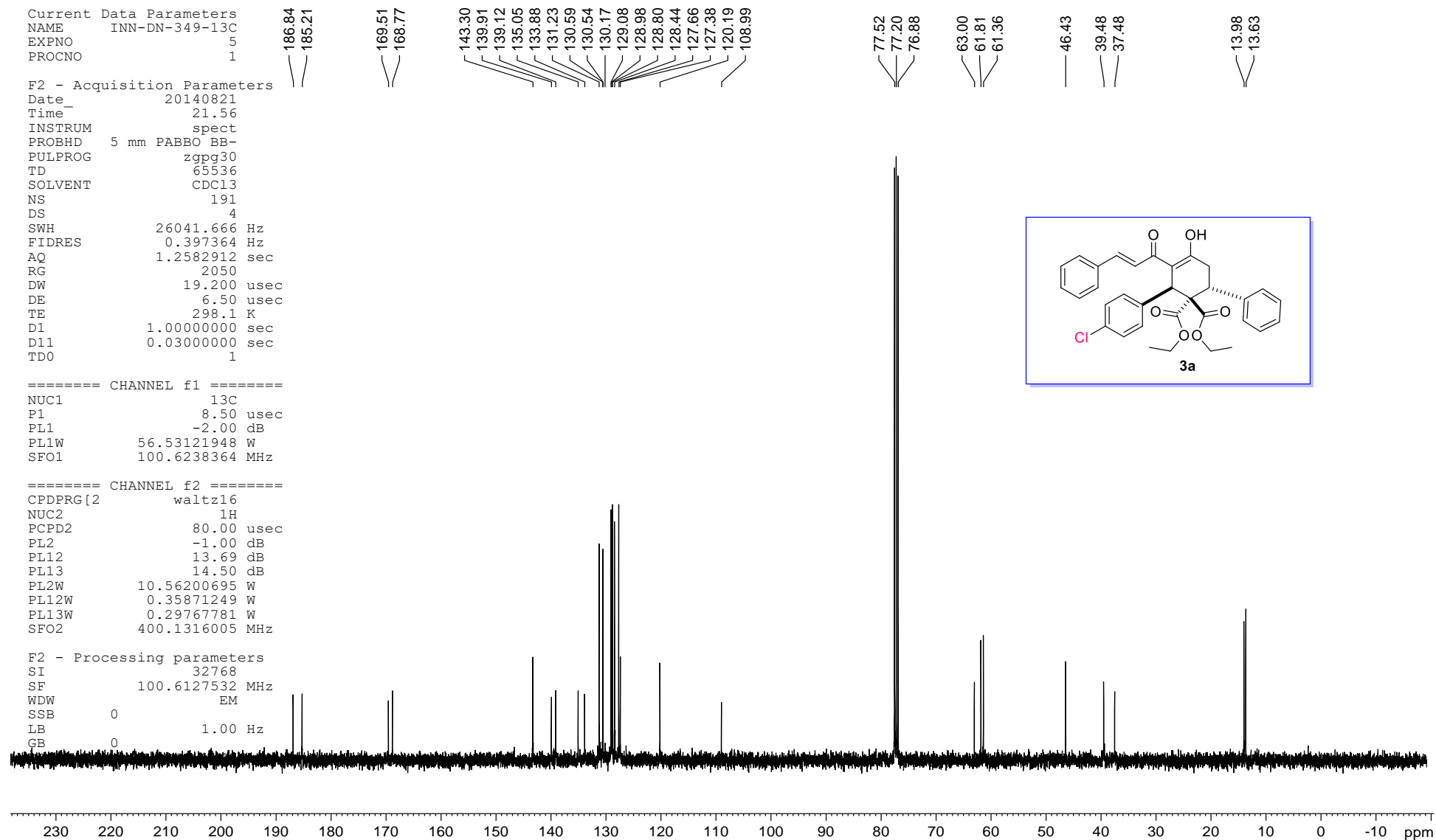


Fig S2. <sup>13</sup>C NMR Spectrum of 3a

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PROCNO 1

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Time\_ 23.45  
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PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 15  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 295.6 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.00000000 W

F2 - Processing parameters  
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WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

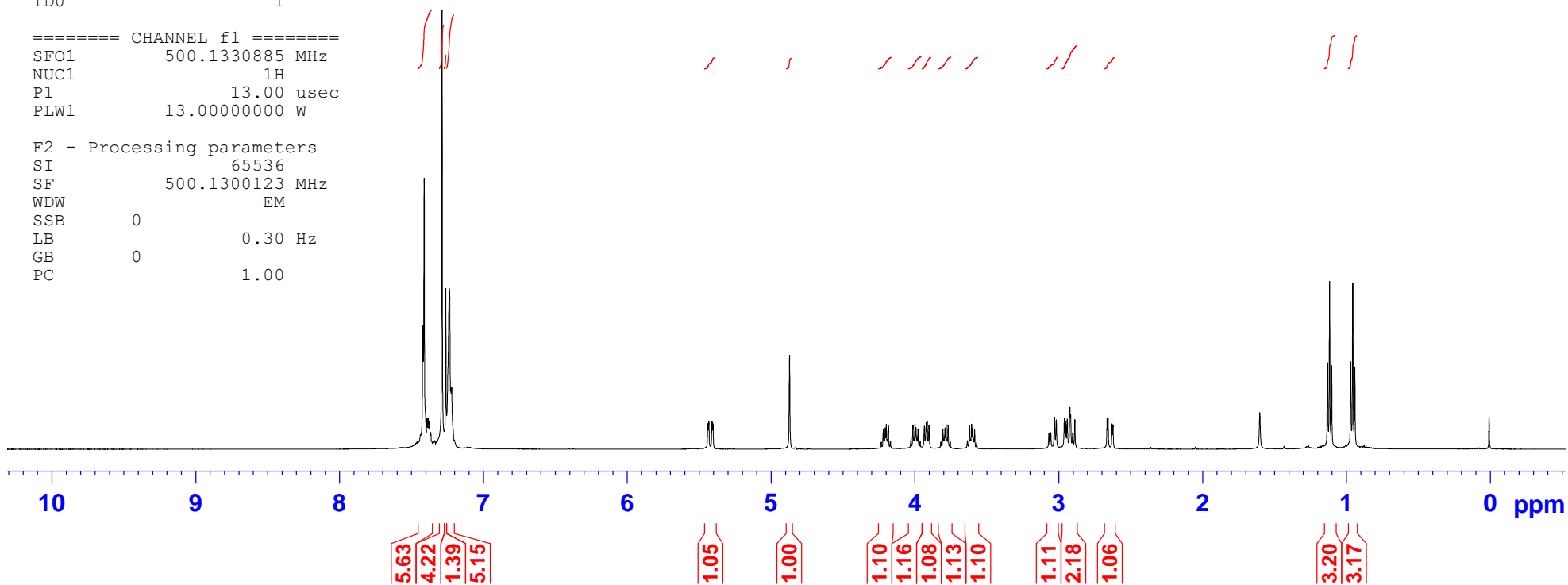
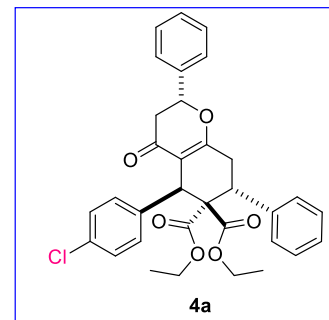


Fig S3. <sup>1</sup>H NMR spectrum of 4a

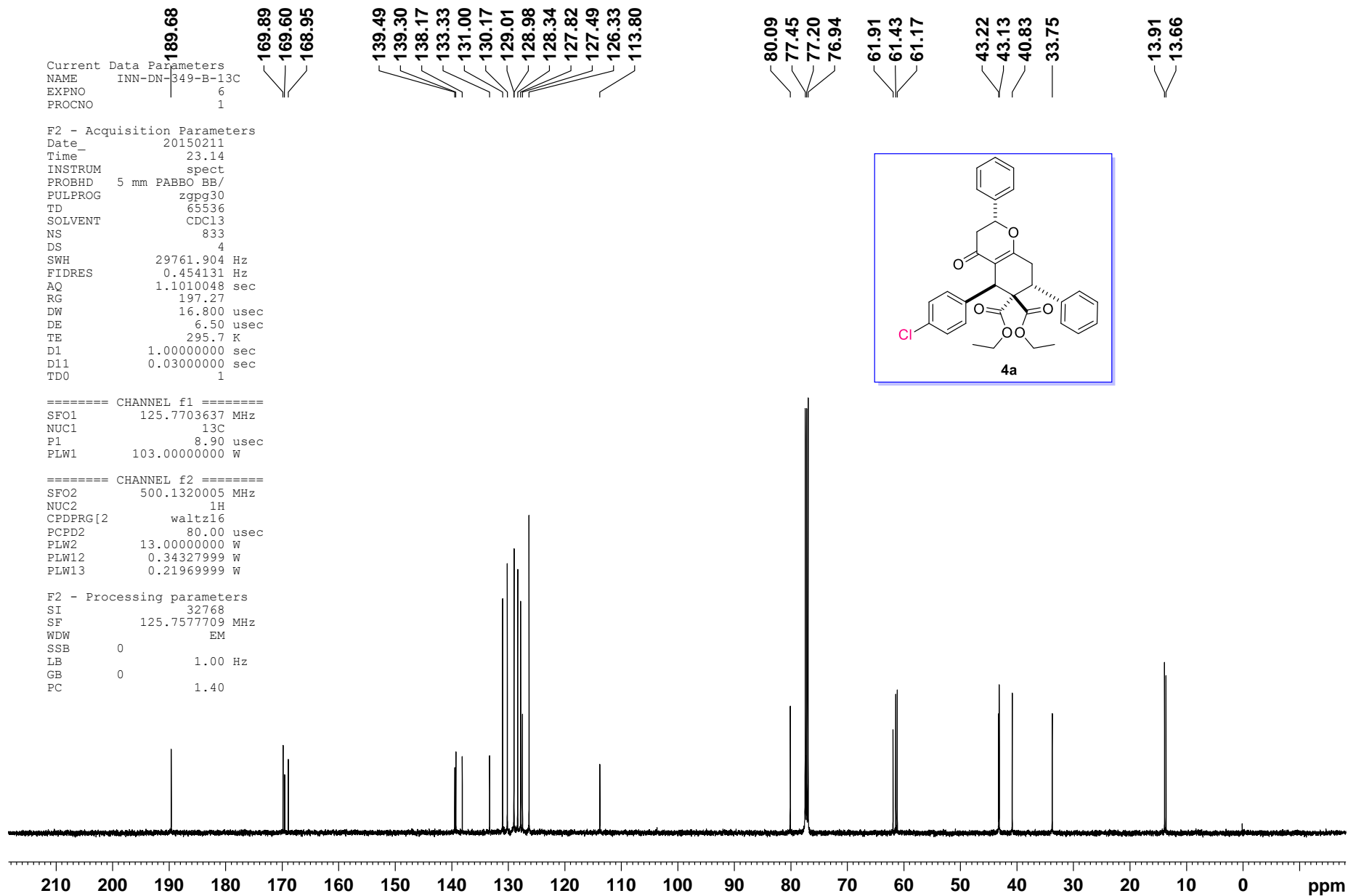


Fig S4. <sup>13</sup>C NMR spectrum of 4a

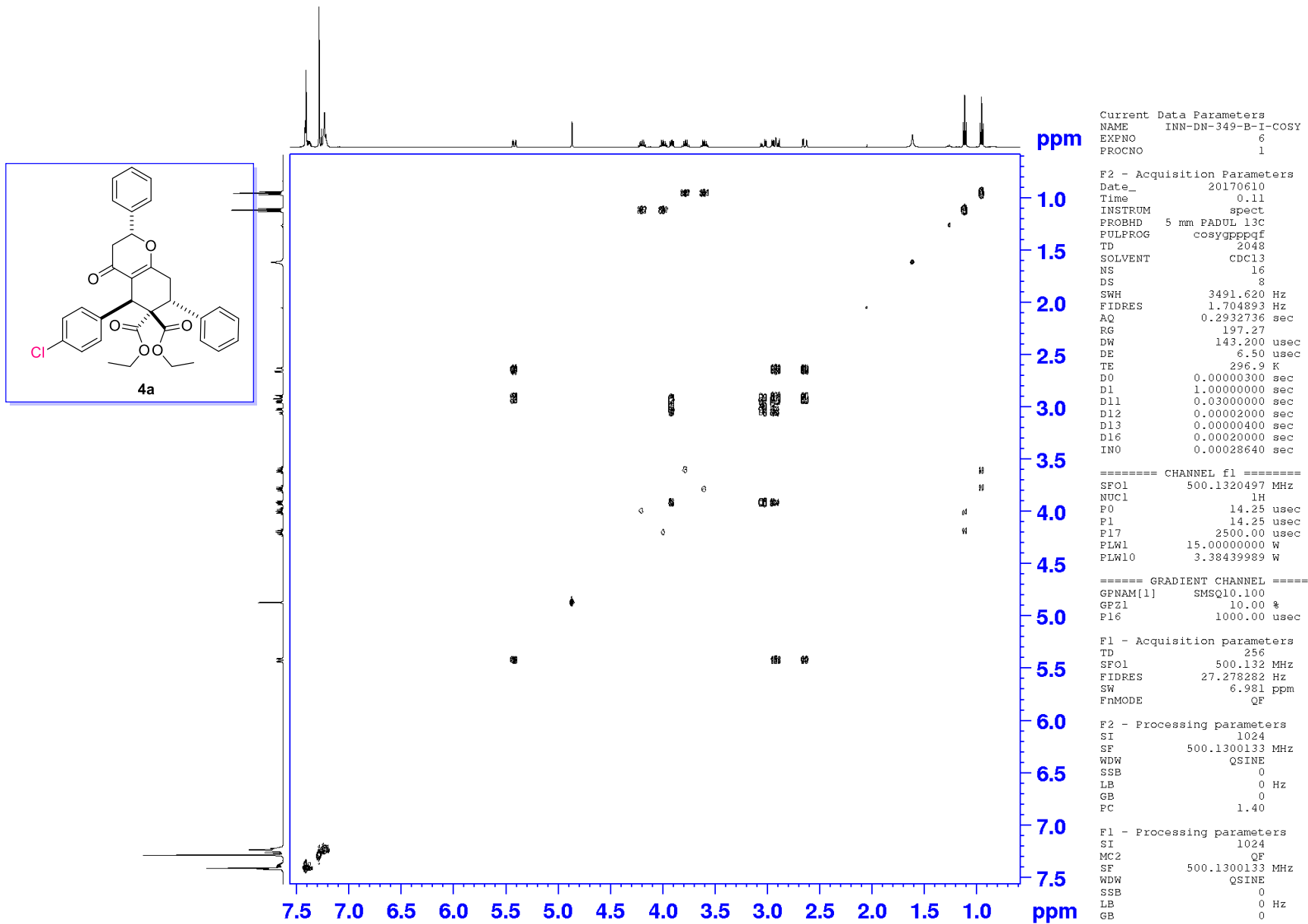


Fig S5.  $^1\text{H}$  -  $^1\text{H}$  COSY NMR Spectrum of 4a



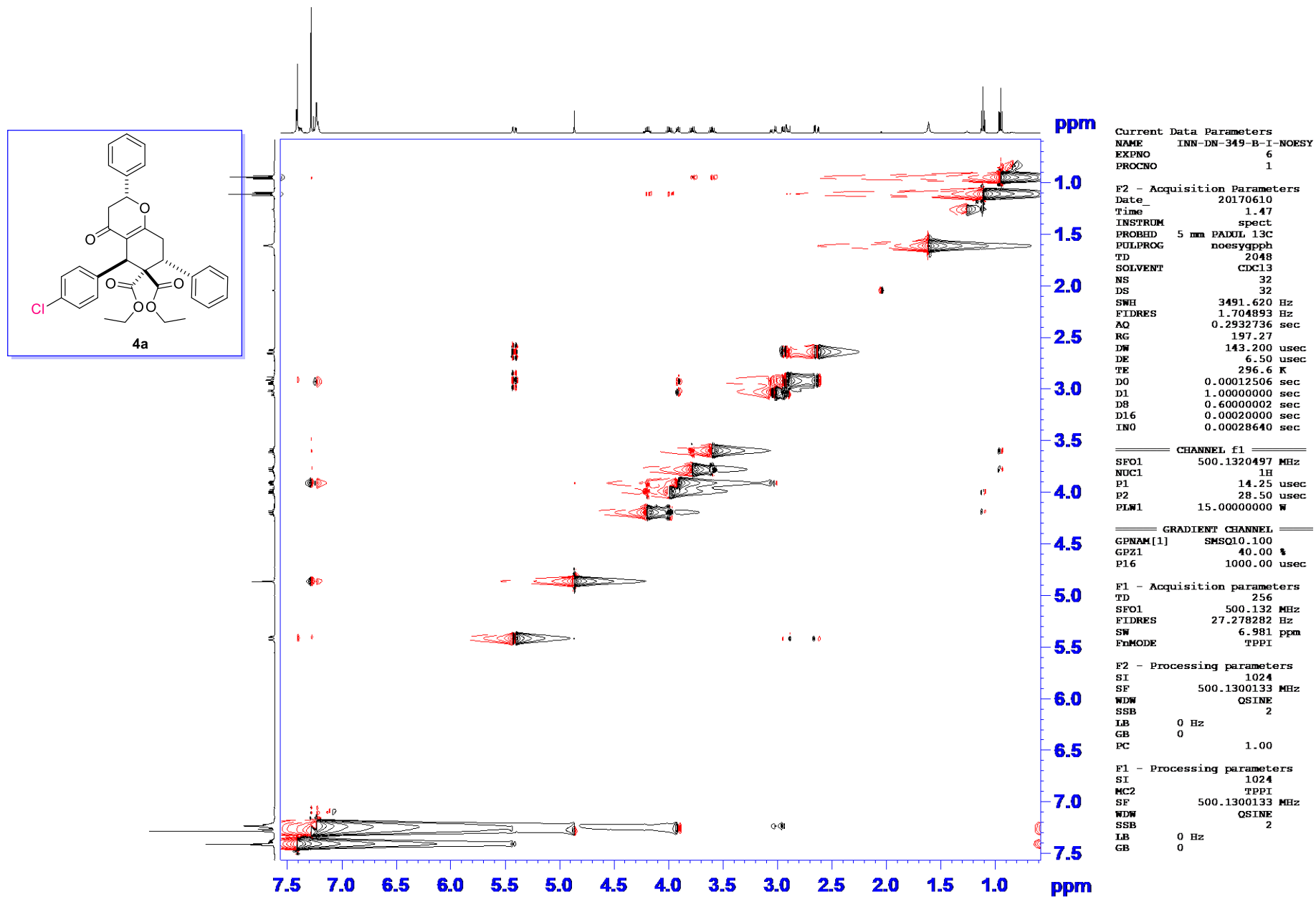


Fig S6.  $^1\text{H}$  -  $^1\text{H}$  NOESY NMR Spectrum of 4a

INN-ART-23-A2-1H

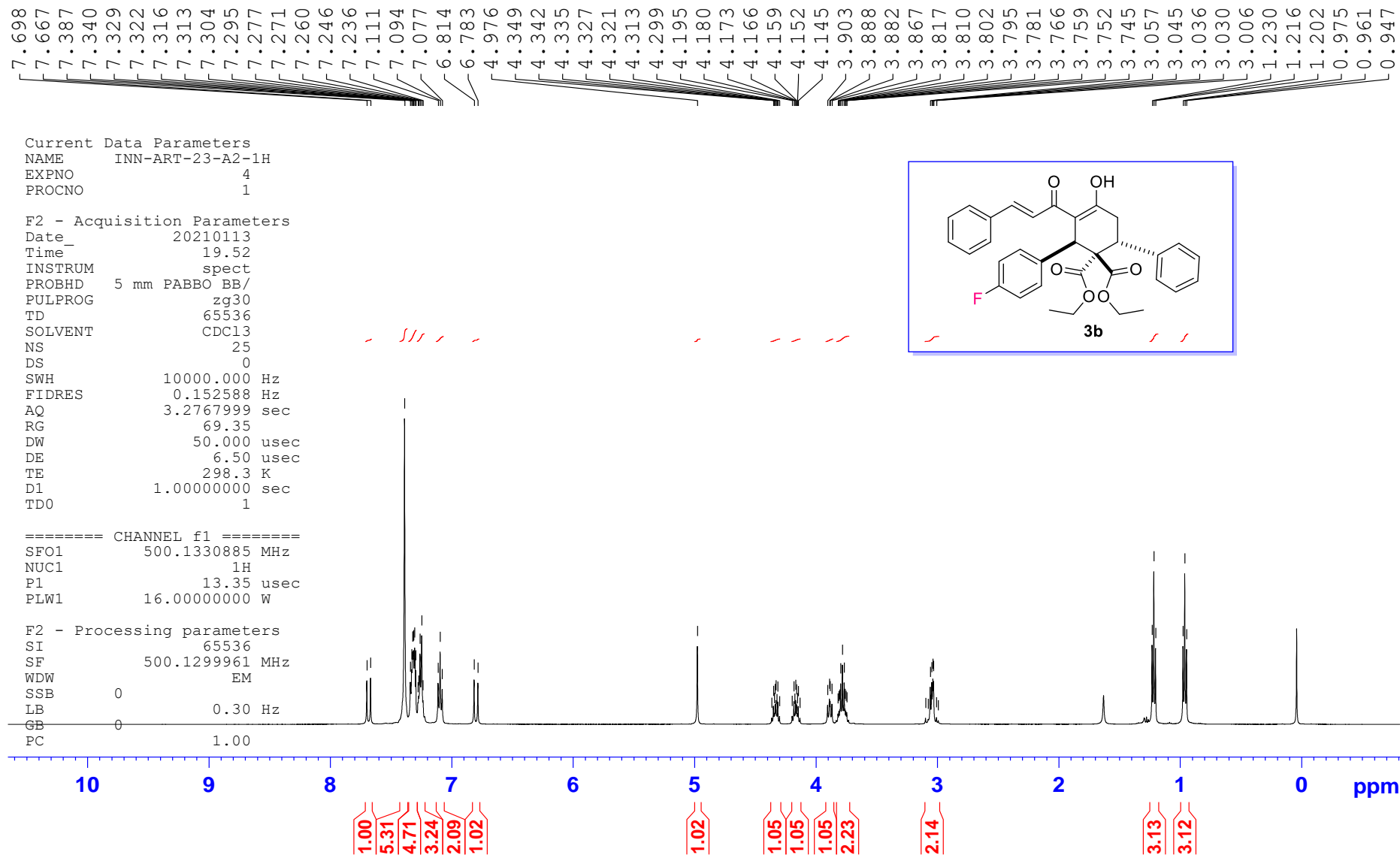
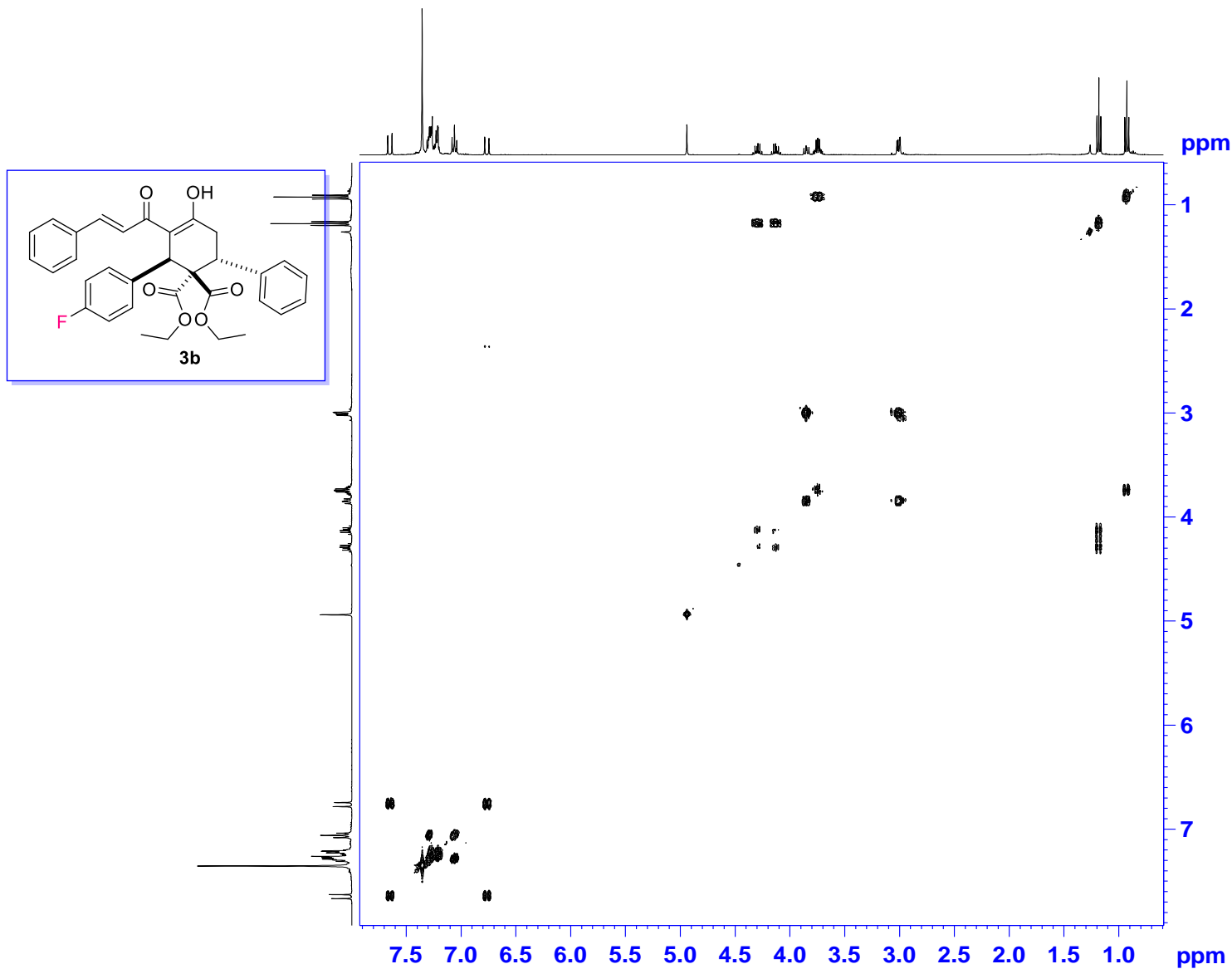


Fig S7. <sup>1</sup>H NMR Spectrum of 3b



Current Data Parameters  
 NAME INN-DN-Ph-Ph-F-COSY  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20170327  
 Time\_ 17.50  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG cosygpgf  
 TD 2048  
 SOLVENT CDCl3  
 NS 16  
 DS 16  
 SWH 2934.272 Hz  
 FIDRES 1.432750 Hz  
 AQ 0.3489792 sec  
 RG 256  
 DW 170.400 usec  
 DE 6.50 usec  
 TE 298.2 K  
 D0 0.00000300 sec  
 D1 1.00000000 sec  
 D13 0.00000400 sec  
 D16 0.00020000 sec  
 IN0 0.00034080 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P0 14.75 usec  
 P1 14.75 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SF01 400.1317136 MHz

===== GRADIENT CHANNEL =====  
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 GPZ1 10.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
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 SF01 400.1317 MHz  
 FIDRES 22.924109 Hz  
 SW 7.333 ppm  
 FnmODE QF

F2 - Processing parameters  
 SI 2048  
 SF 400.1300100 MHz  
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 SSB 0  
 LB 0 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 512  
 MC2 QF  
 SF 400.1300100 MHz  
 WDW SINE  
 SSB 0  
 LB 0 Hz  
 GB 0

Fig S8.  $^1\text{H}$ - $^1\text{H}$  COSY NMR Spectrum of **3b**

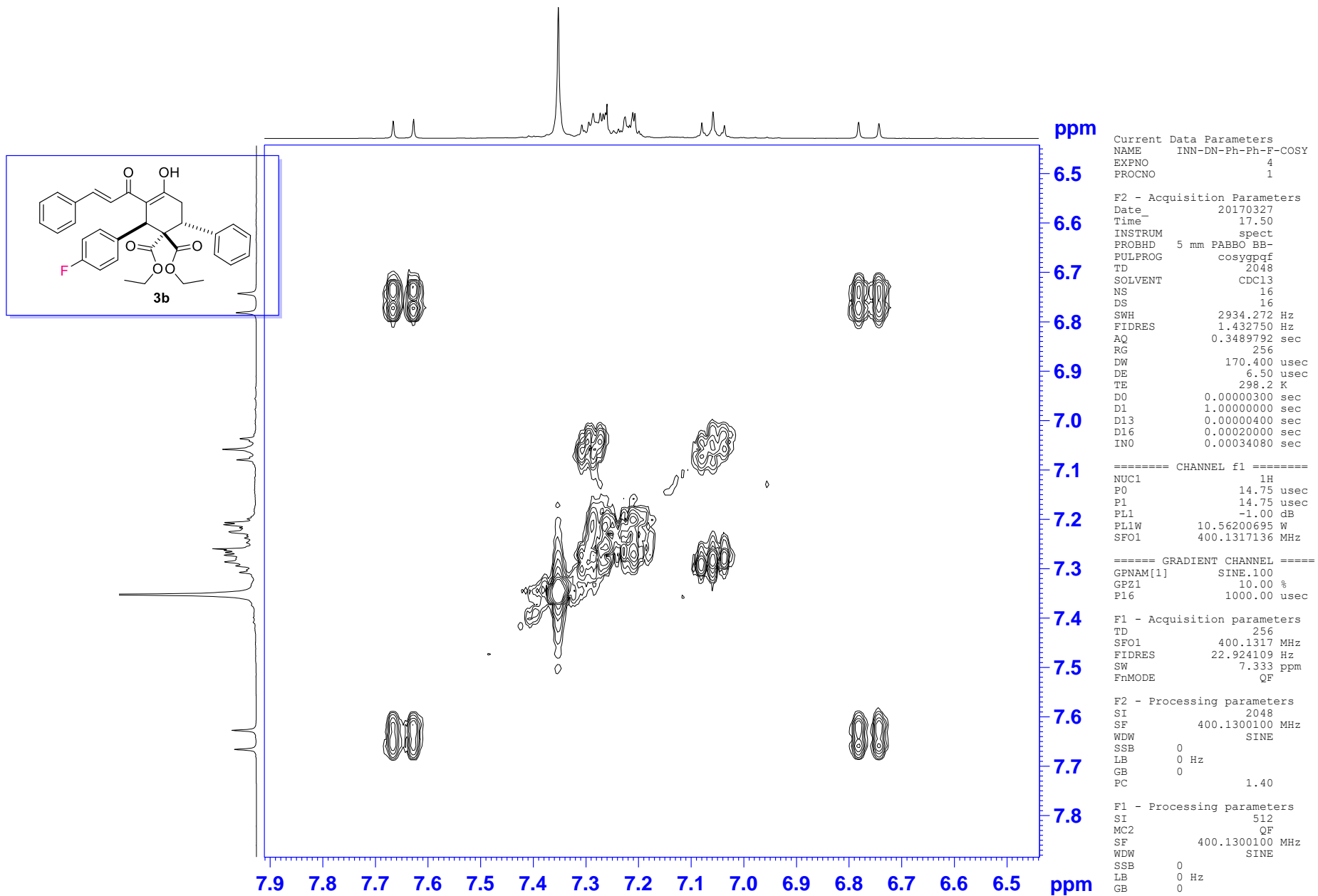
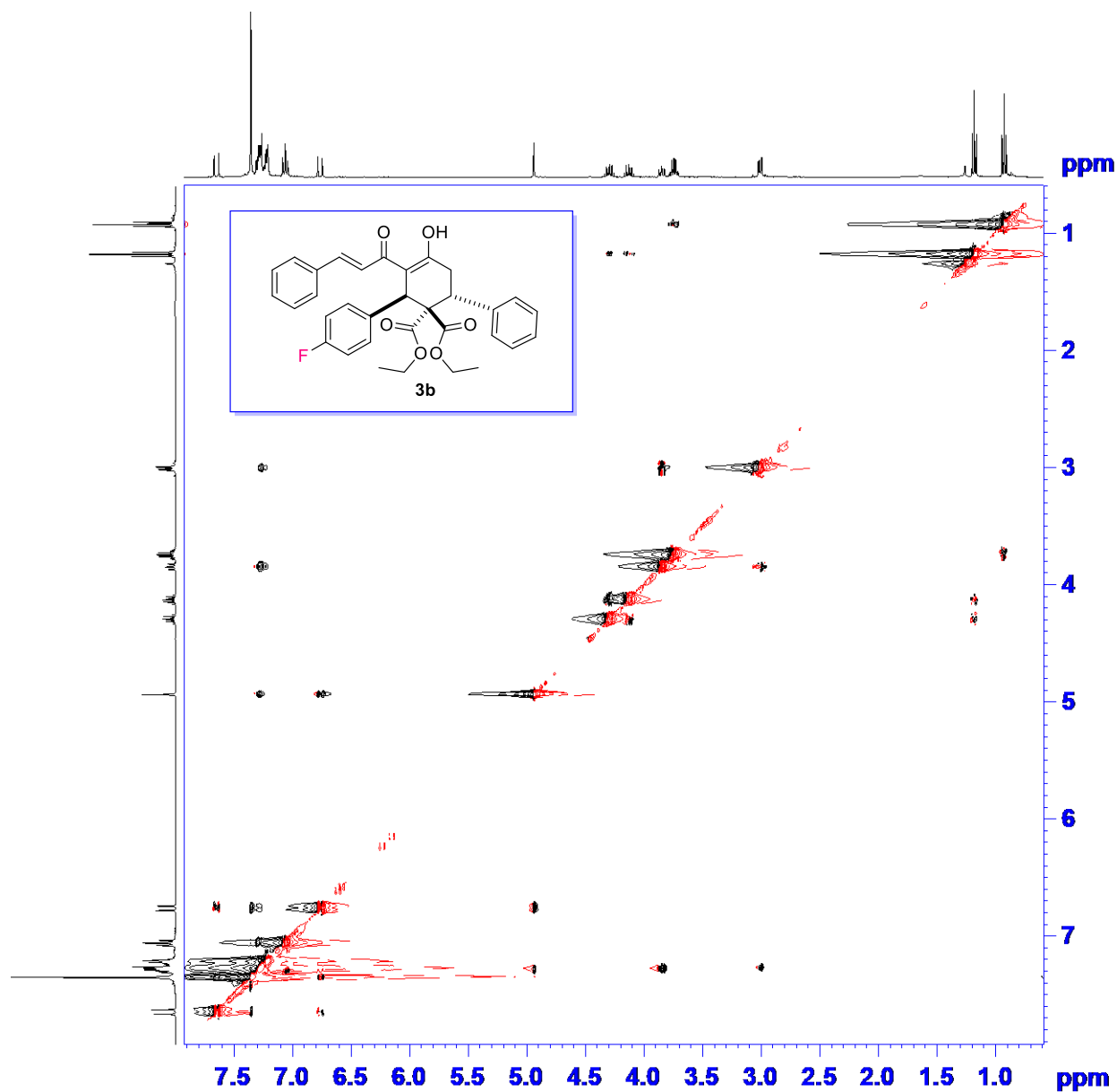


Fig S9.  $^1\text{H}$  -  $^1\text{H}$  COSY NMR Spectrum (expansion) of 3b



Current Data Parameters  
 NAME INN-DN-Ph-Ph-F-NOESY  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20170327  
 Time 19.26  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG noesyph  
 TD 2048  
 SOLVENT CDC13  
 NS 16  
 DS 16  
 SWH 2934.272 Hz  
 FIDRES 1.432750 Hz  
 AQ 0.3489792 sec  
 RG 57  
 DW 170.400 usec  
 DE 6.50 usec  
 TE 297.4 K  
 D0 0.00015162 sec  
 D1 1.00000000 sec  
 D8 0.60000002 sec  
 INO 0.00034080 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.75 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SF01 400.1317136 MHz

F1 - Acquisition parameters  
 TD 512  
 SF01 400.1317 MHz  
 FIDRES 11.462054 Hz  
 SW 7.333 ppm  
 F1MODE States-TPPI

F2 - Processing parameters  
 SI 2048  
 SF 400.1300100 MHz  
 WDW QSINE  
 SSB 2  
 LB 0 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 512  
 MC2 States-TPPI  
 SF 400.1300100 MHz  
 WDW QSINE  
 SSB 2  
 LB 0 Hz  
 GB 0

Fig S10. <sup>1</sup>H -<sup>1</sup>H NOESY NMR Spectrum of 3b

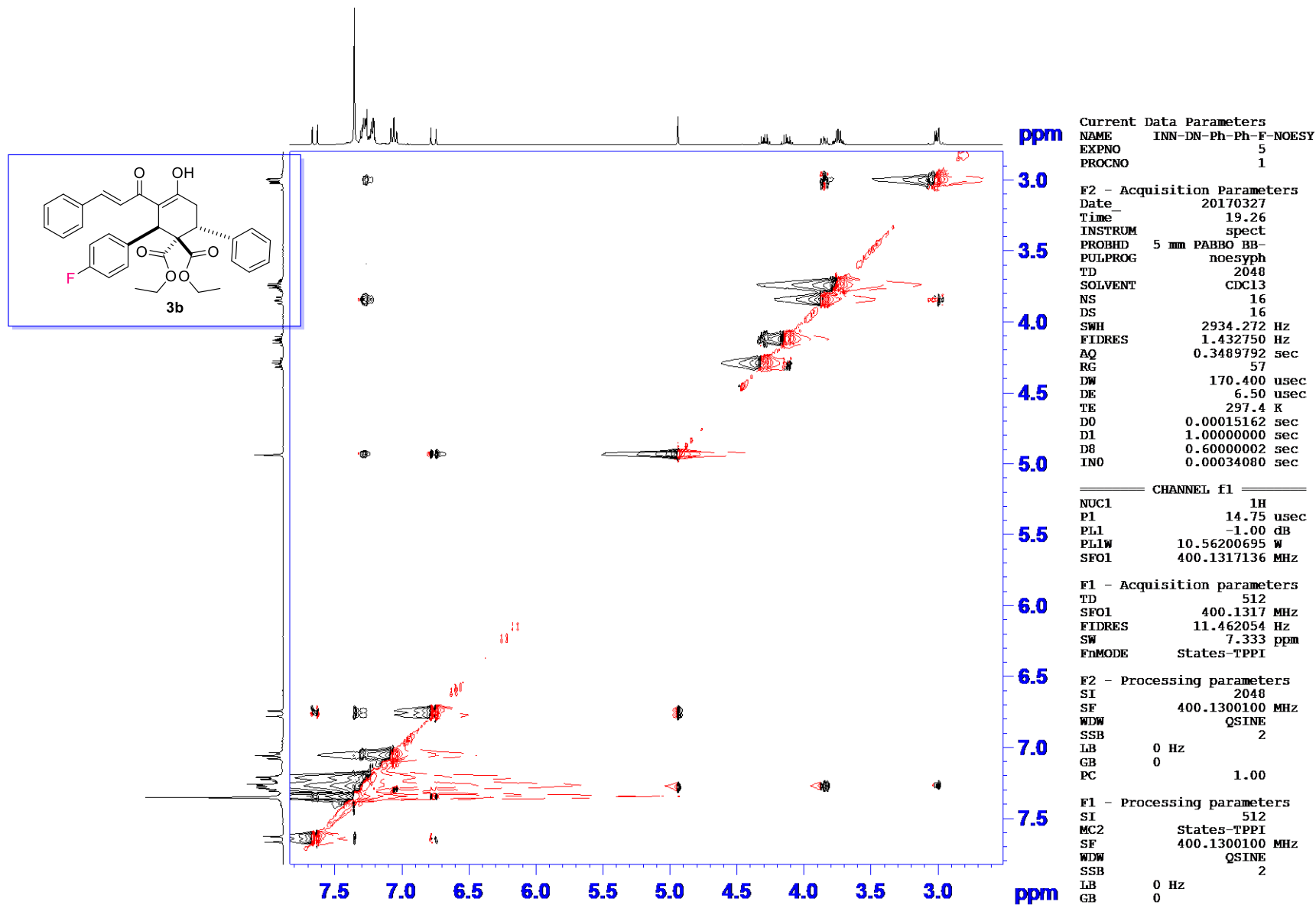


Fig S11.  $^1\text{H}$ - $^1\text{H}$  NOESY NMR Spectrum (expansion) of 3b

```

Current Data Parameters
NAME      INN-ART-289A2-13C
EXPNO     5
PROCNO    1
F2 - Acquisition Parameters
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Time      19.55
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS        232
DS        0
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010048 sec
RG        197.27
DW        16.800 usec
DE        6.50 usec
TE        298.8 K
D1        1.00000000 sec
D11       0.03000000 sec
TD0       1

```

```

===== CHANNEL f1 =====
SFO1     125.7703637 MHz
NUC1     13C
P1       8.90 usec
PLW1     103.00000000 W

```

```

===== CHANNEL f2 =====
SFO2     500.1320005 MHz
NUC2     1H
CPDPRG[2] waltz16
PCPD2    80.00 usec
PLW2     16.00000000 W
PLW12    0.44556001 W
PLW13    0.22411001 W

```

```

F2 - Processing parameters
SI       32768
SF       125.7577692 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40

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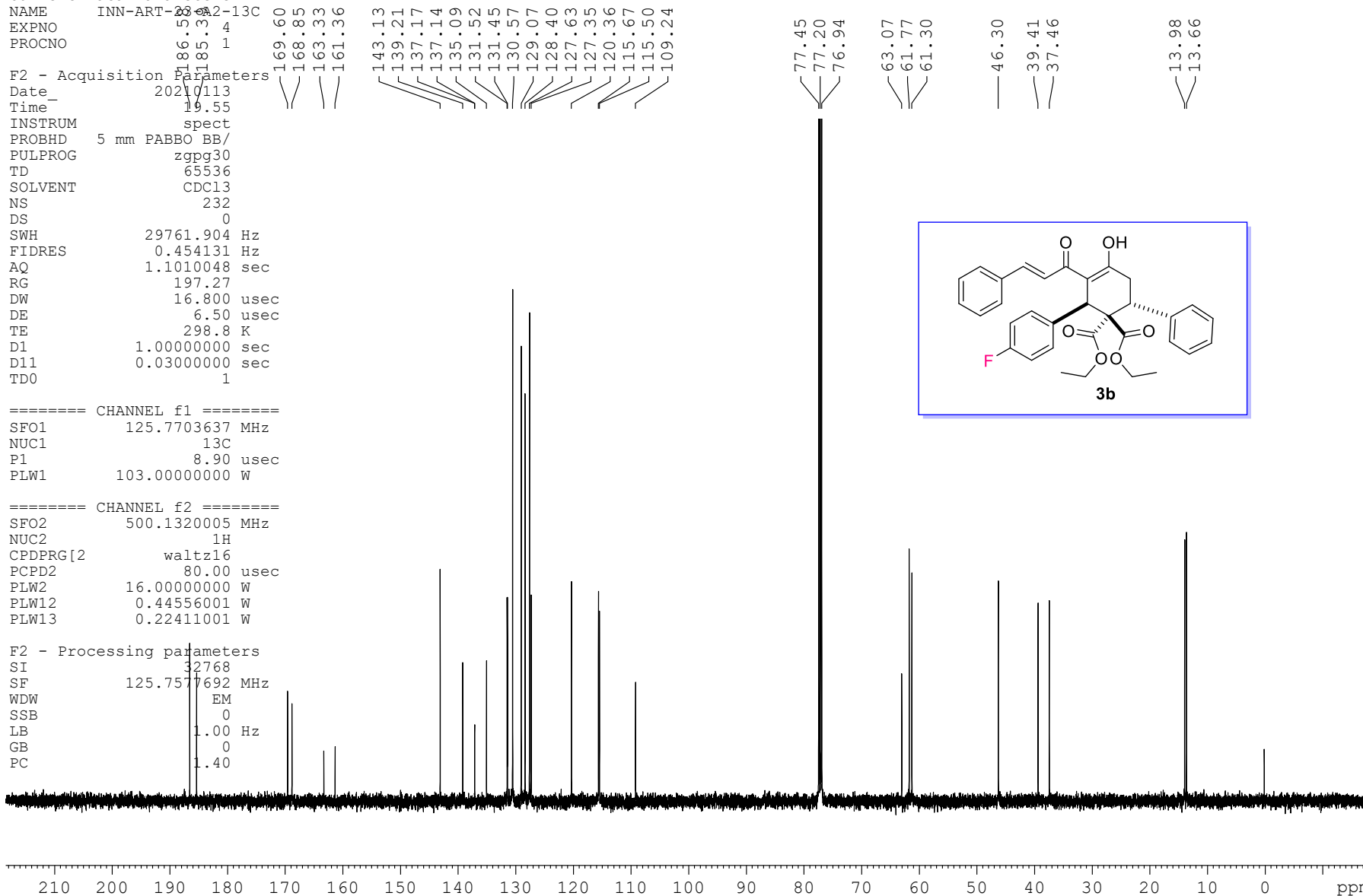
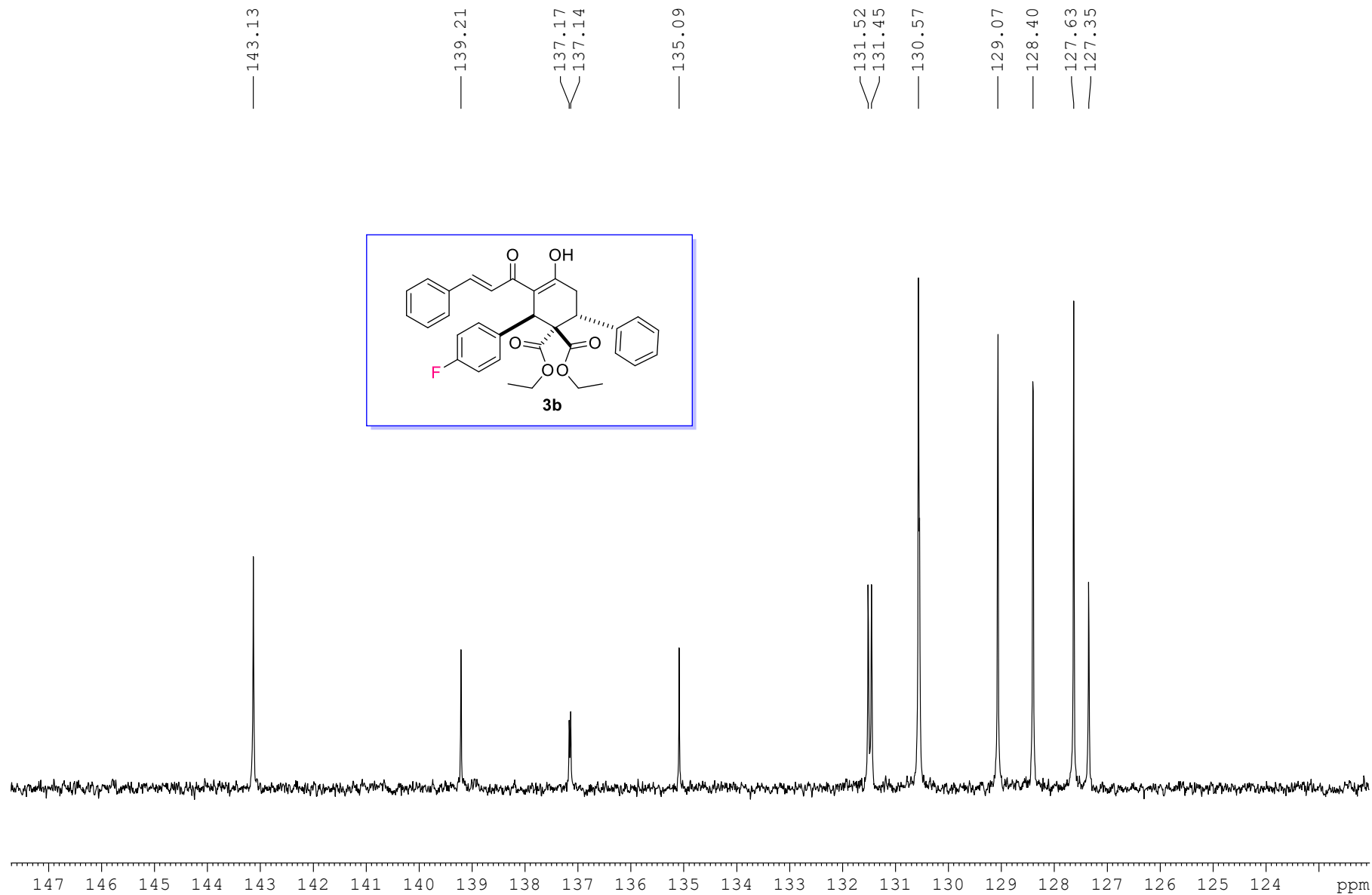


Fig S12. <sup>13</sup>C NMR Spectrum of 3b



**Fig S13. <sup>13</sup>C NMR Spectrum (expansion) of 3b**



```

Current Data Parameters
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PROCNO    1
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PULPROG   jmod
TD         65536
SOLVENT   CDCl3
NS         102
DS         0
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010048 sec
RG         197.27
DW         16.800 usec
DE         6.50 usec
TE         300.7 K
CNST2     145.0000000
CNST11    1.0000000
D1         1.00000000 sec
D20       0.00689655 sec
TD0        1

```

```

===== CHANNEL f1 =====
SFO1      125.7703643 MHz
NUC1       13C
P1         8.90 usec
P2         17.80 usec
PLW1      103.00000000 W

```

```

===== CHANNEL f2 =====
SFO2      500.1320005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2     80.00 usec
PLW2      16.00000000 W
PLW12     0.44556001 W

```

```

F2 - Processing parameters
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SF         125.7577685 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40

```

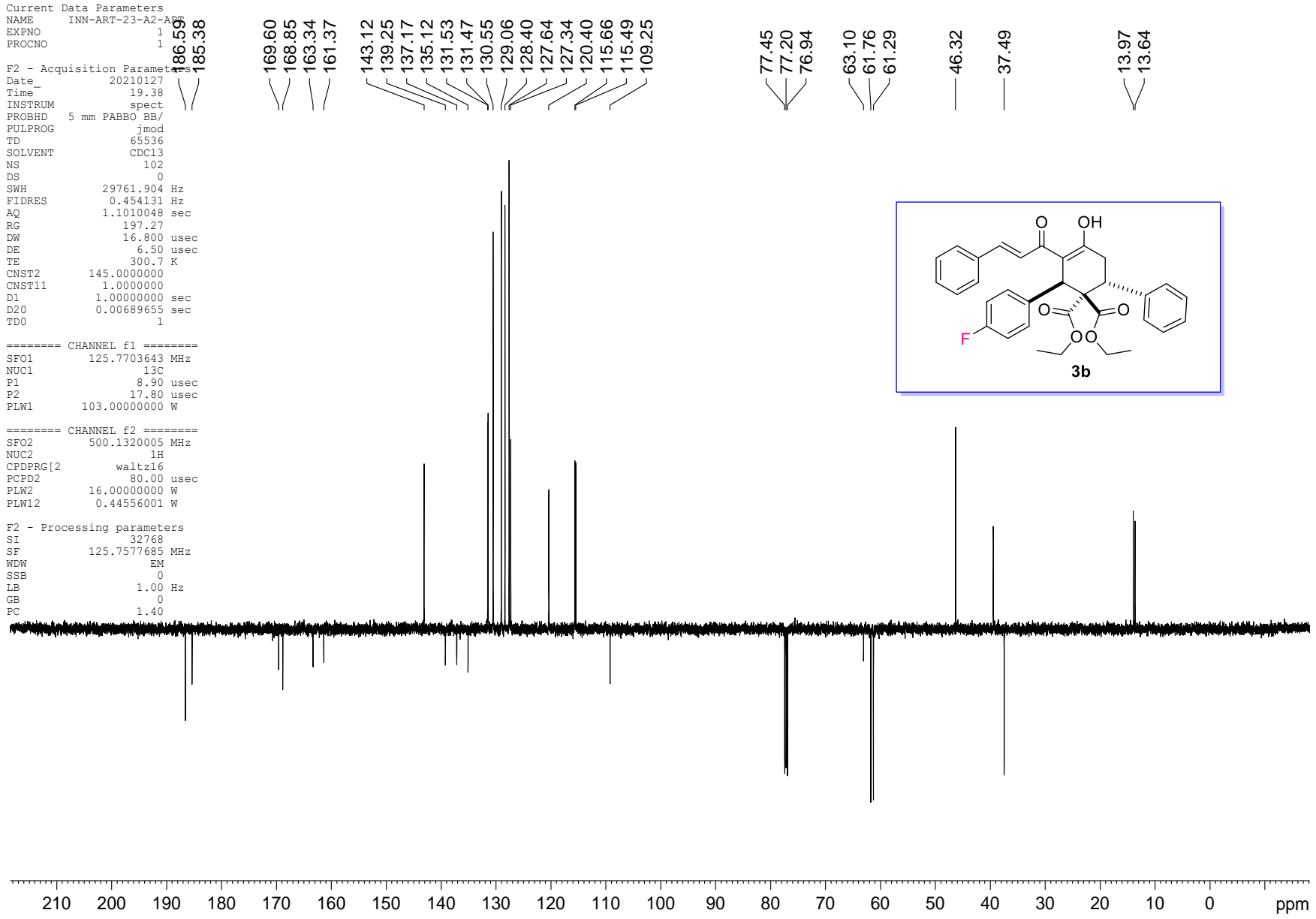


Fig S14. <sup>13</sup>C-APT Spectrum of 3b

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 PROCNO 1

F2 - Acquisition Parameters  
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 Time\_ 11.56  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgflgn  
 TD 131072  
 SOLVENT CDCl3  
 NS 105  
 DS 0  
 SWH 138888.891 Hz  
 FIDRES 1.059638 Hz  
 AQ 0.4718592 sec  
 RG 197.27  
 DW 3.600 usec  
 DE 6.50 usec  
 TE 299.1 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 =====  
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 NUC1 19F  
 P1 19.75 usec  
 PLW1 55.00000000 W

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 LB 0.30 Hz  
 GB 0  
 PC 1.00

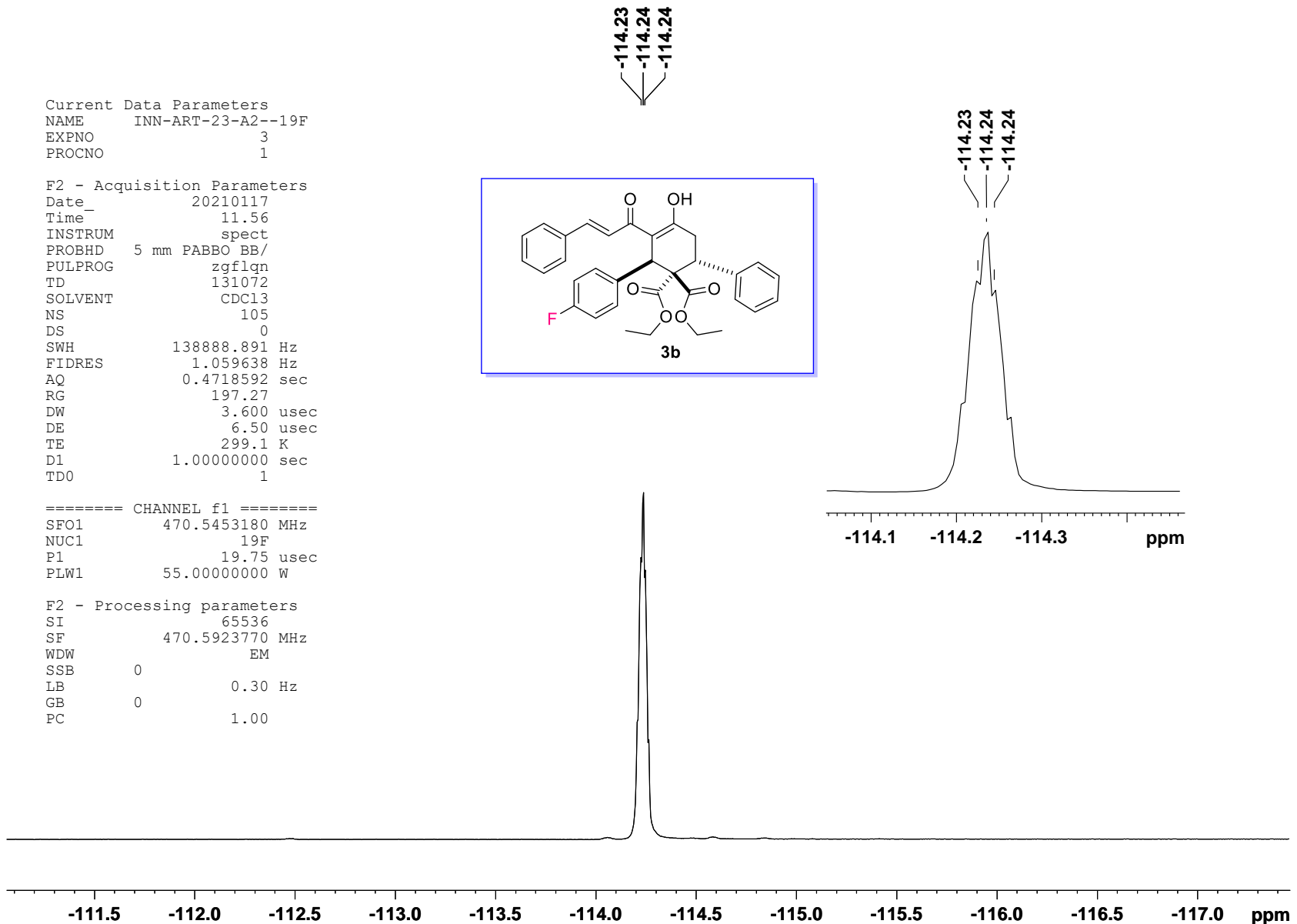
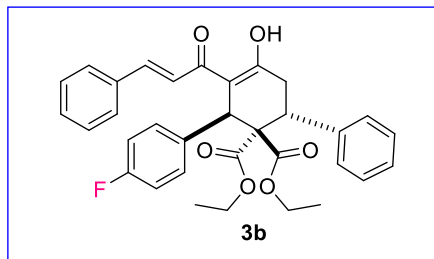


Fig S15. <sup>19</sup>F NMR Spectrum of 3b

INN-ART-23B52-1H

7.419  
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7.388  
7.380  
7.371  
7.328  
7.317  
7.311  
7.301  
7.259  
7.242  
7.230  
7.216  
7.022  
7.005  
6.988  
5.441  
5.434  
5.412  
5.406  
4.883  
4.225  
4.211  
4.204  
4.189  
4.019  
4.004  
3.997  
3.983  
3.945  
3.932  
3.925  
3.912  
3.798  
3.784  
3.777  
3.762  
3.626  
3.611  
3.604  
3.590  
3.004  
2.991  
2.962  
2.948  
2.942  
2.919  
2.914  
2.885  
2.665  
2.658  
2.631  
2.624  
1.132  
1.118  
1.104  
0.973  
0.959  
0.945

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PROCNO 1

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SOLVENT CDCl3  
NS 7  
DS 0  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
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RG 80.35  
DW 33.333 usec  
DE 6.50 usec  
TE 299.2 K  
D1 1.00000000 sec  
TDO 1

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NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

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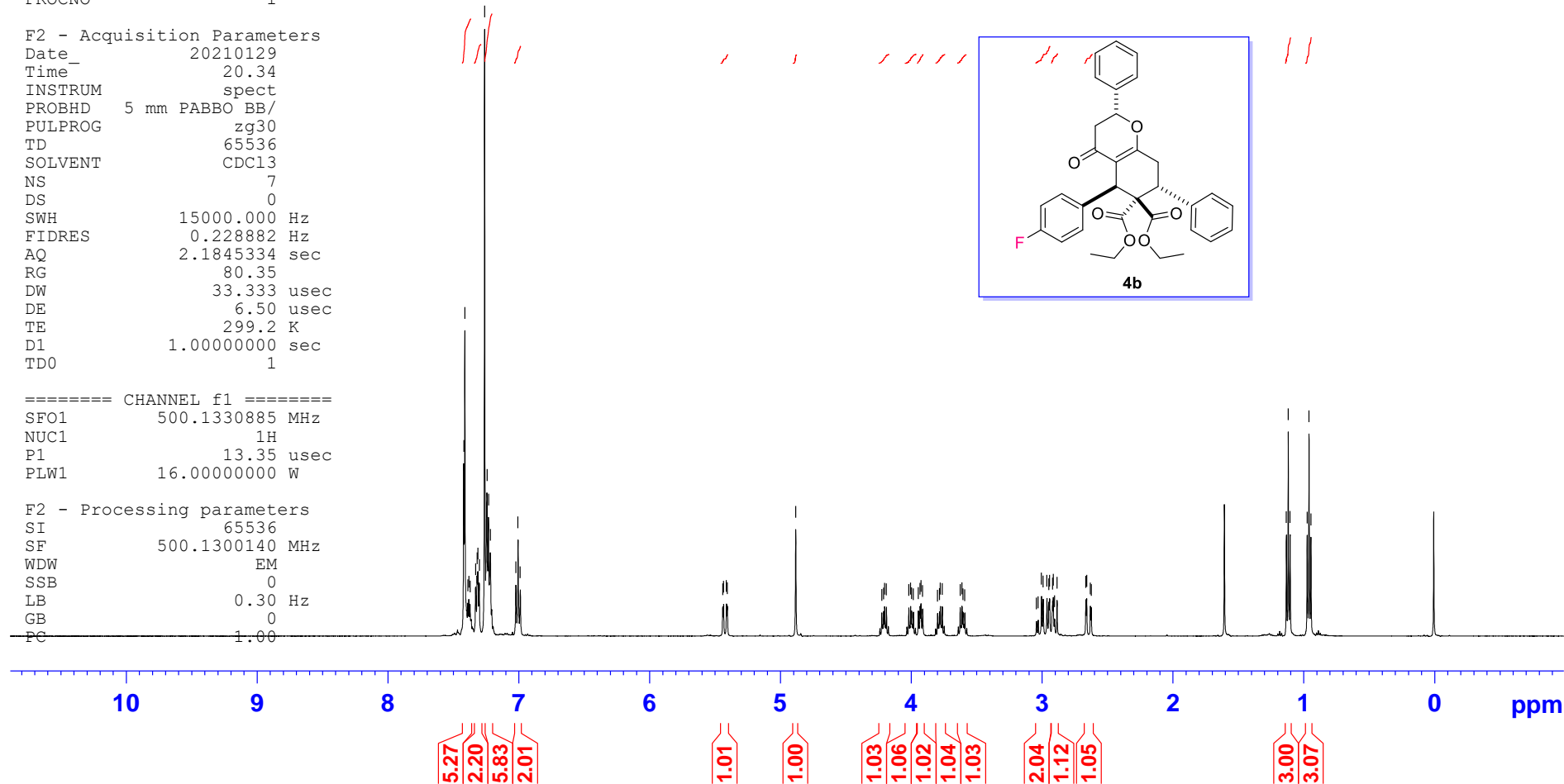
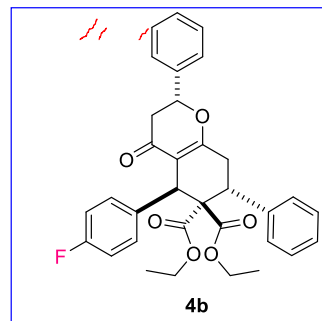


Fig S16. <sup>1</sup>H NMR spectrum of 4b

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TD 65536  
SOLVENT CDCl3  
NS 228  
DS 4  
SWH 23809.523 Hz  
FIDRES 0.726609 Hz  
AQ 1.3762560 sec  
RG 101  
DW 21.000 usec  
DE 6.50 usec  
TE 298.4 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1  
SFO1 100.6655806 MHz  
NUC1 13C  
P0 2.67 usec  
P1 8.00 usec  
PLW1 98.44999695 W  
SFO2 400.3016012 MHz  
NUC2 1H  
CPDPRG[2] waltz65  
PCPD2 90.00 usec  
PLW2 21.61000061 W  
PLW12 0.17073999 W  
PLW13 0.08588200 W

F2 - Processing parameters  
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SF 100.6555210 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

139.34  
138.03  
136.23  
136.19  
131.03  
130.95  
130.03  
128.76  
127.57  
127.23  
126.12  
114.94  
114.73  
113.81

79.86

61.81  
61.19  
60.90

43.04  
42.88  
40.50

33.58

13.71  
13.48

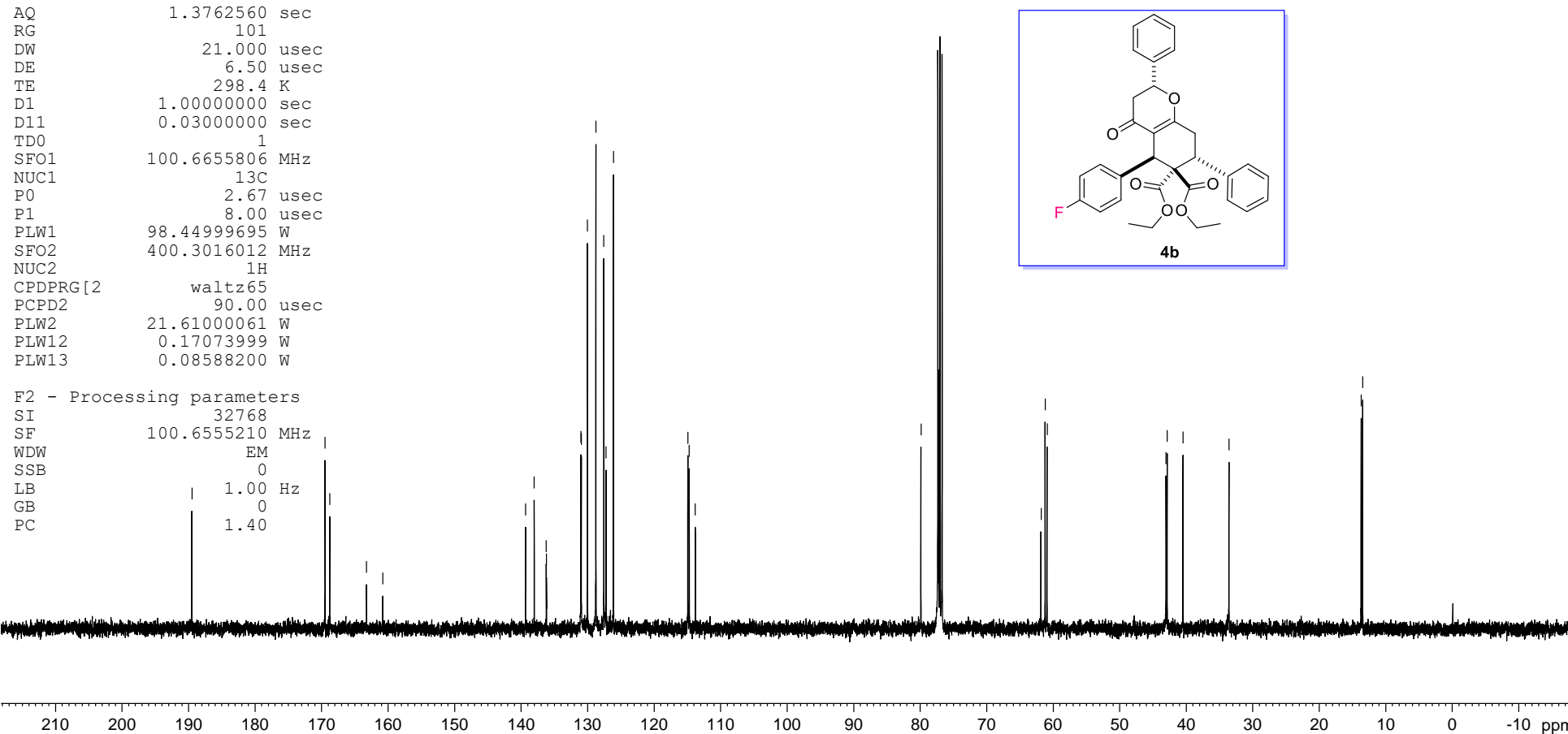
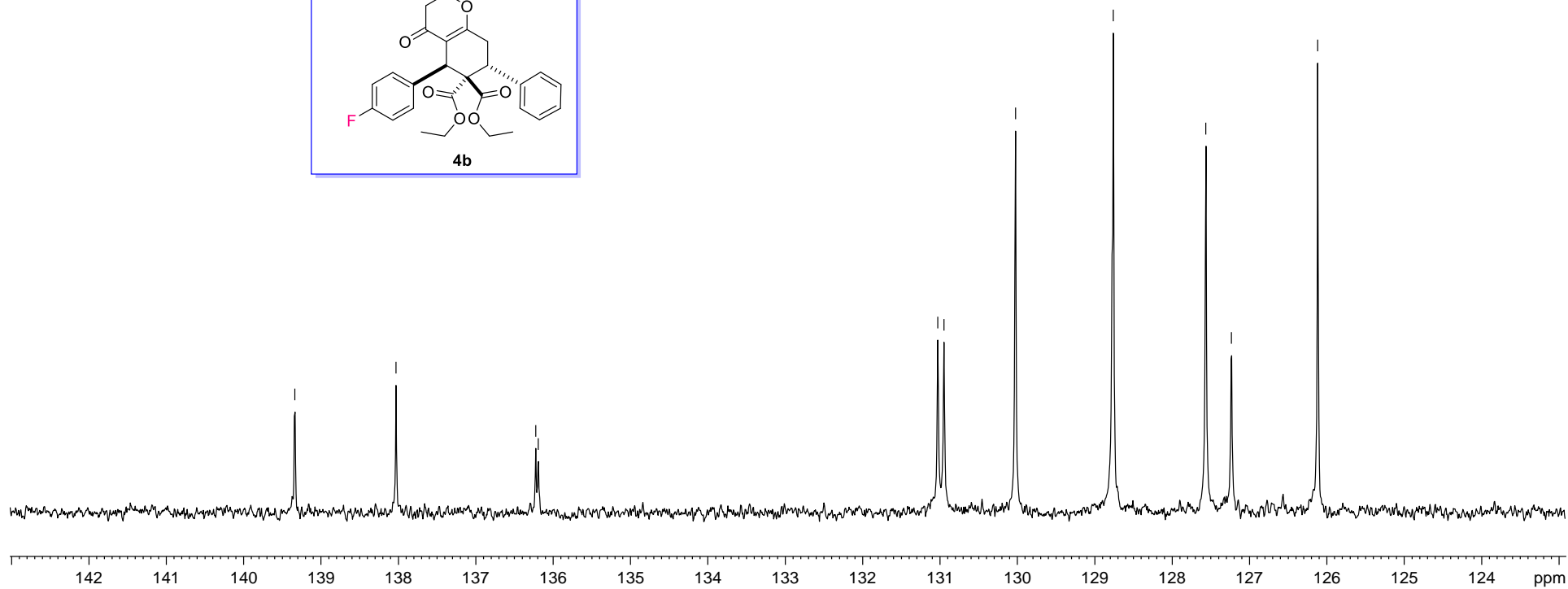
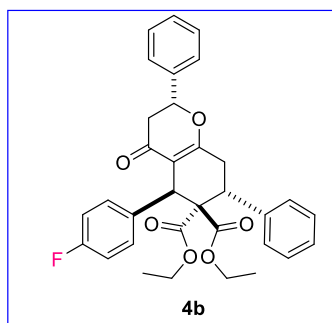


Fig S17. <sup>13</sup>C NMR spectrum of 4b

— 139.34  
— 138.03  
136.23  
136.19  
131.03  
130.95  
— 130.03  
— 128.76  
— 127.57  
— 127.23  
— 126.12



**Fig S18. <sup>13</sup>C NMR spectrum (expansion) of 4b**

Current Data Parameters  
 NAME INN-ART-23B52-APT  
 EXPNO 14  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20210201  
 Time\_ 12.21 h  
 INSTRUM Avance  
 PROBHD Z163739\_0237 (  
 PULPROG jmod  
 TD 65536  
 SOLVENT CDC13  
 NS 61  
 DS 4  
 SWH 23809.523 Hz  
 FIDRES 0.726609 Hz  
 AQ 1.3762560 sec  
 RG 101  
 DW 21.000 usec  
 DE 6.50 usec  
 TE 298.3 K  
 CNST2 145.0000000  
 CNST11 1.0000000  
 D1 2.00000000 sec  
 D20 0.00689655 sec  
 TD0 1  
 SFO1 100.6655806 MHz  
 NUC1 13C  
 P1 8.00 usec  
 P2 16.00 usec  
 PLW1 98.44999695 W  
 SFO2 400.3016012 MHz  
 NUC2 1H  
 PCPD2 90.00 usec  
 PLW2 21.61000061 W  
 PLW12 0.17073999 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6555001 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

139.55  
 138.24  
 136.43  
 136.40  
 131.24  
 131.16  
 130.23  
 128.97  
 127.77  
 127.44  
 115.14  
 114.93  
 114.02

77.52  
 77.20  
 76.88

62.02  
 61.39  
 61.10

43.25

33.79

13.91  
 13.68

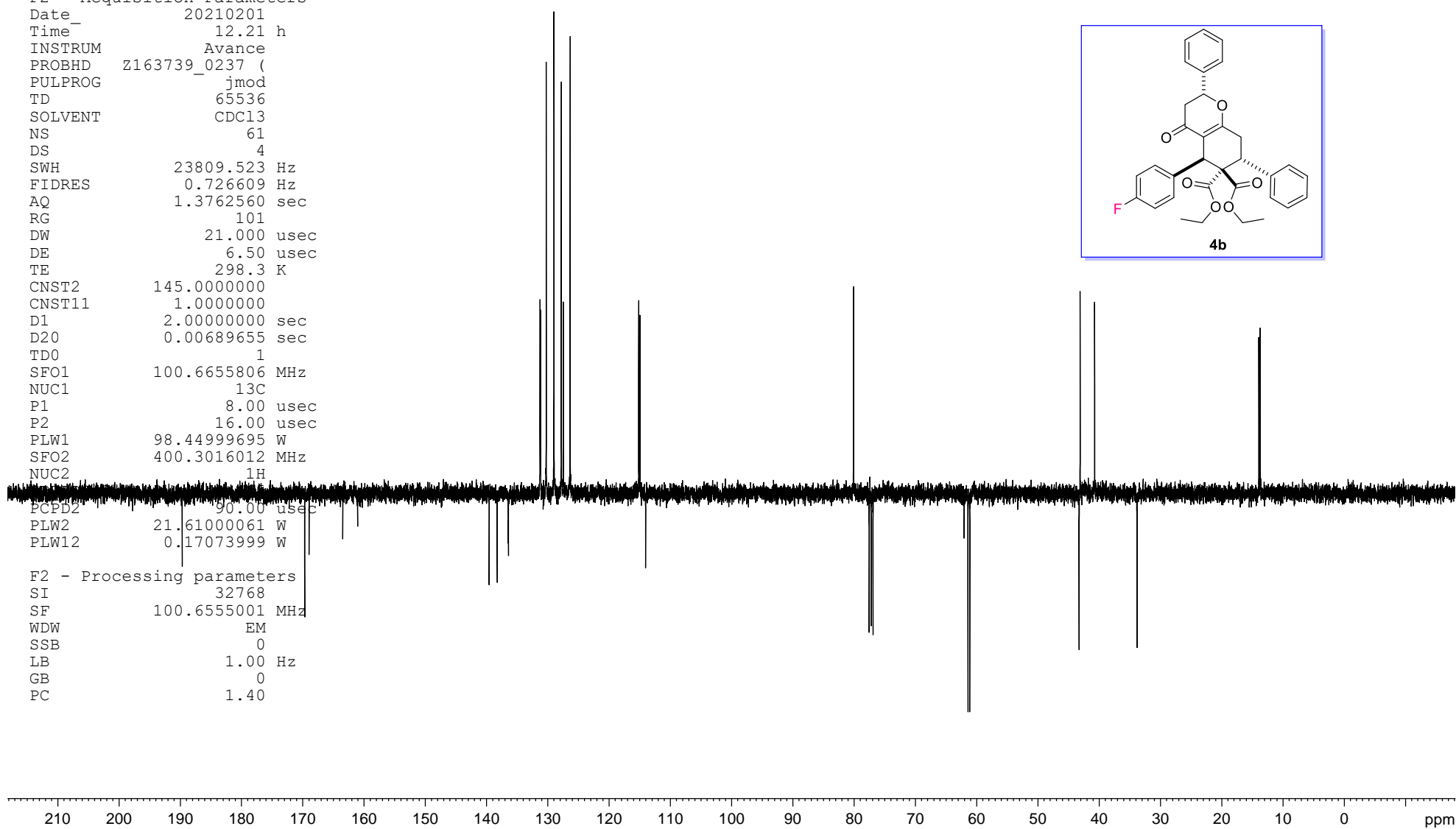


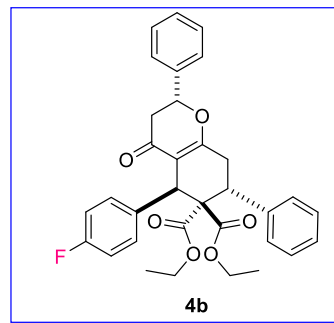
Fig S19. <sup>13</sup>C-APT spectrum of 4b

Current Data Parameters  
NAME INN-ART-25B-52-19F  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210211  
Time\_ 18.06  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgflqn  
TD 131072  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 113636.367 Hz  
FIDRES 0.866977 Hz  
AQ 0.5767168 sec  
RG 197.27  
DW 4.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 470.5453180 MHz  
NUC1 19F  
P1 19.75 usec  
PLW1 55.00000000 W

F2 - Processing parameters  
SI 65536  
SF 470.5923770 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



-115.37  
-115.38  
-115.39  
-115.40  
-115.41  
-115.43

-115.37  
-115.38  
-115.39  
-115.40  
-115.41  
-115.43

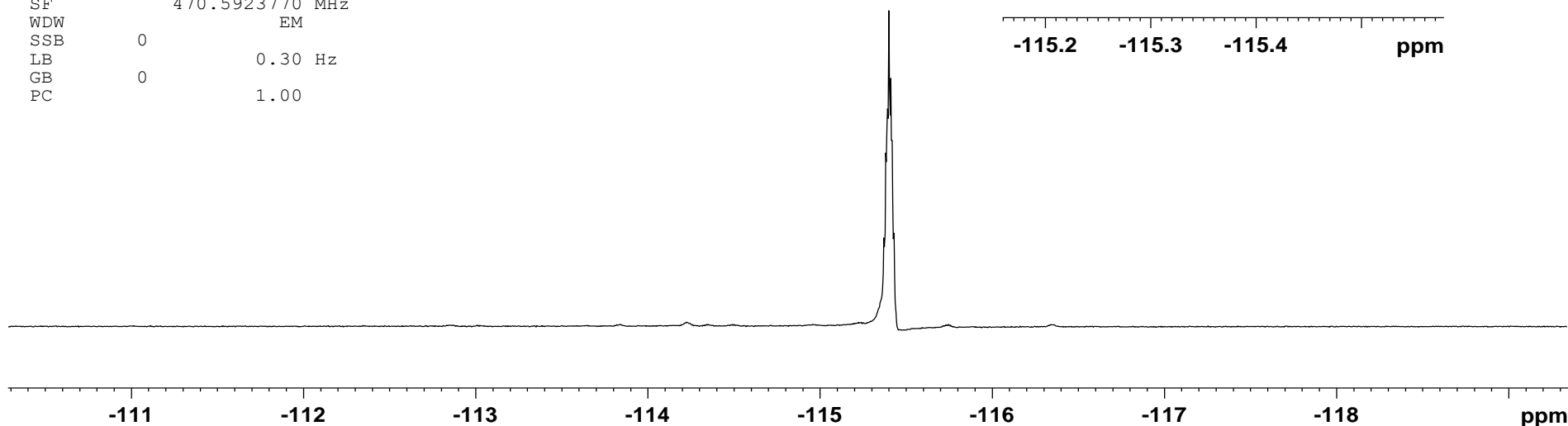


Fig S20.  $^{19}\text{F}$  NMR spectrum of 4b

Current Data Parameters  
NAME INN-DN-PH-PH-BR-A-1H  
EXPNO 9  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20150711  
Time 19.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 7  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1340013 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300100 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

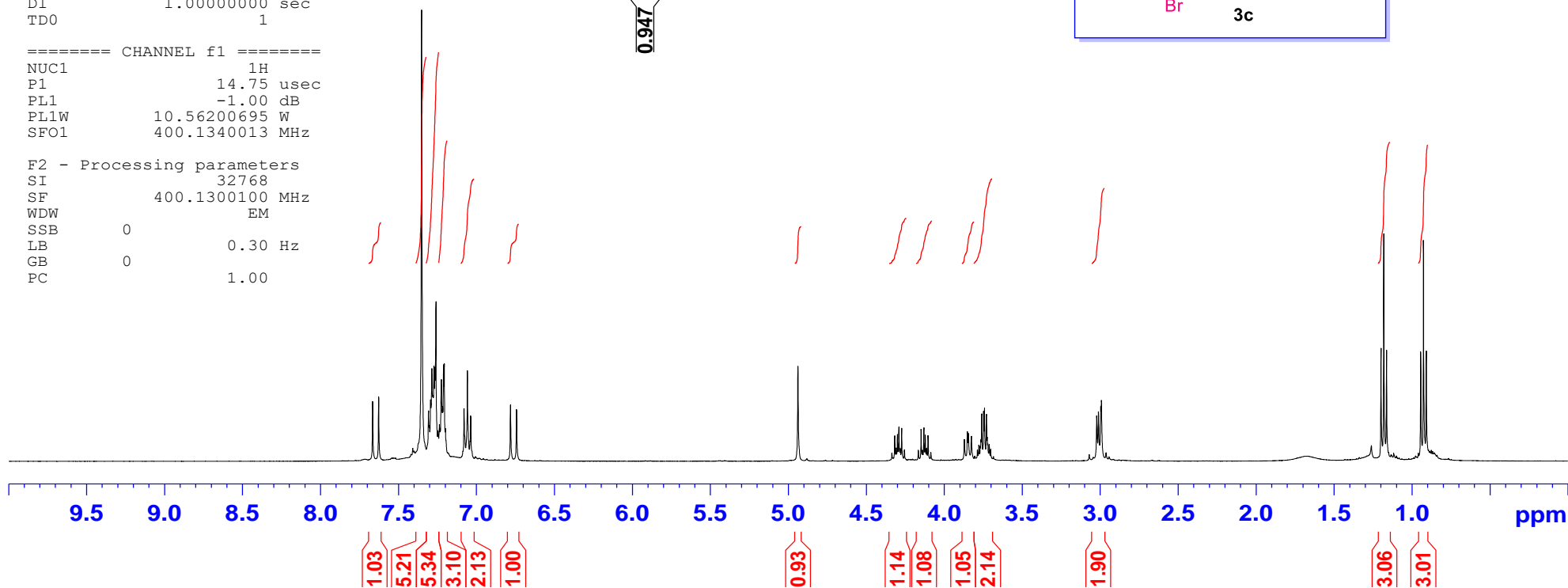
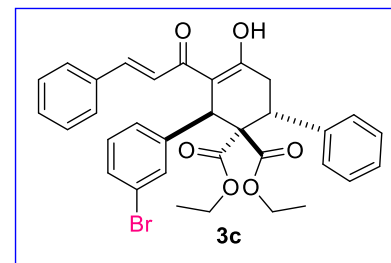
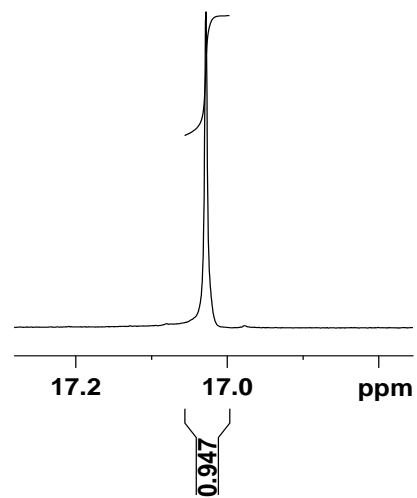


Fig S21. <sup>1</sup>H NMR Spectrum of 3c



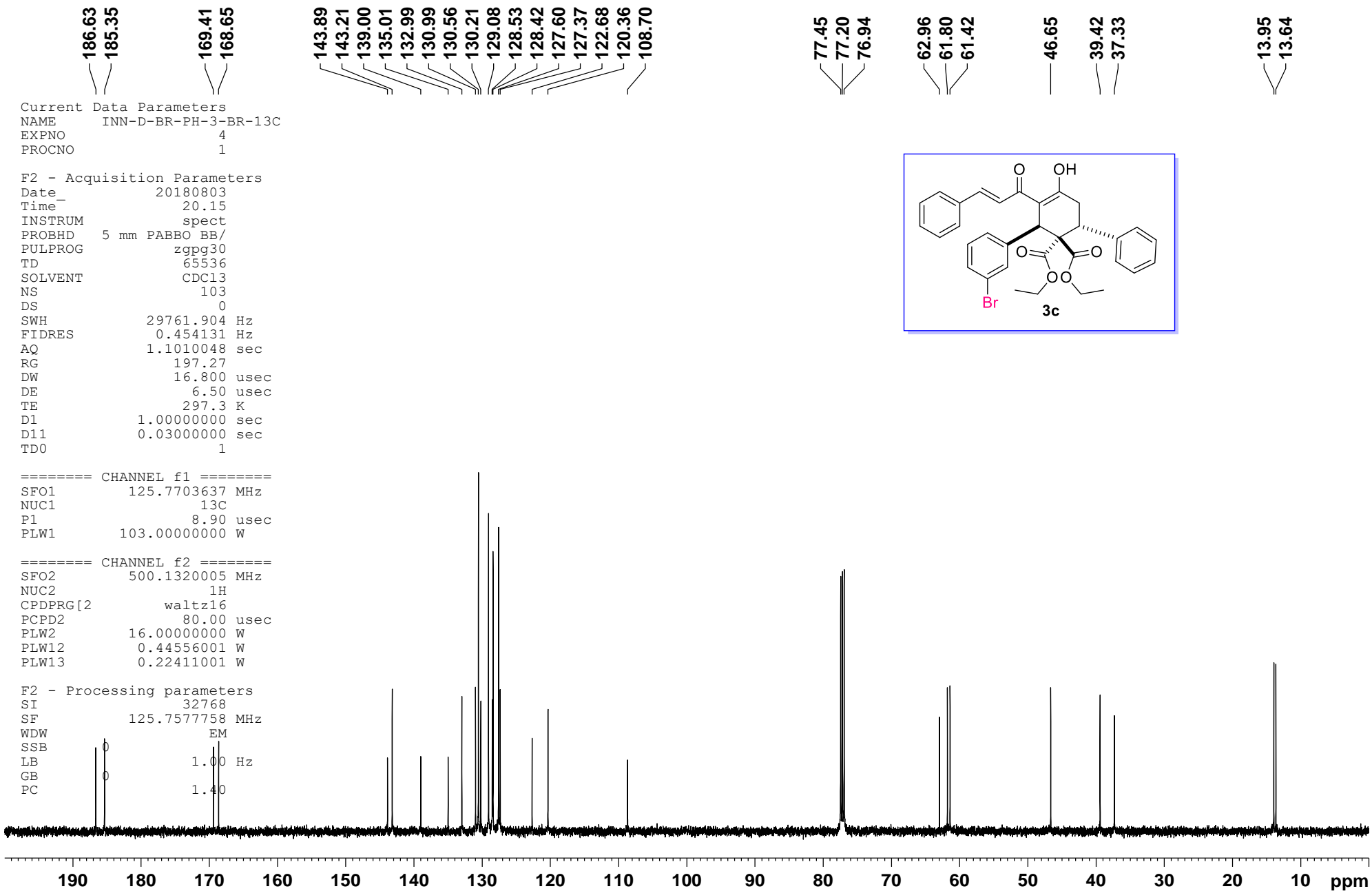


Fig S22. <sup>13</sup>C NMR spectrum of 3c

Current Data Parameters  
NAME INN-DN-PH-PH-BR-B-1H  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20150711  
Time\_ 19.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 228  
DW 60.800 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

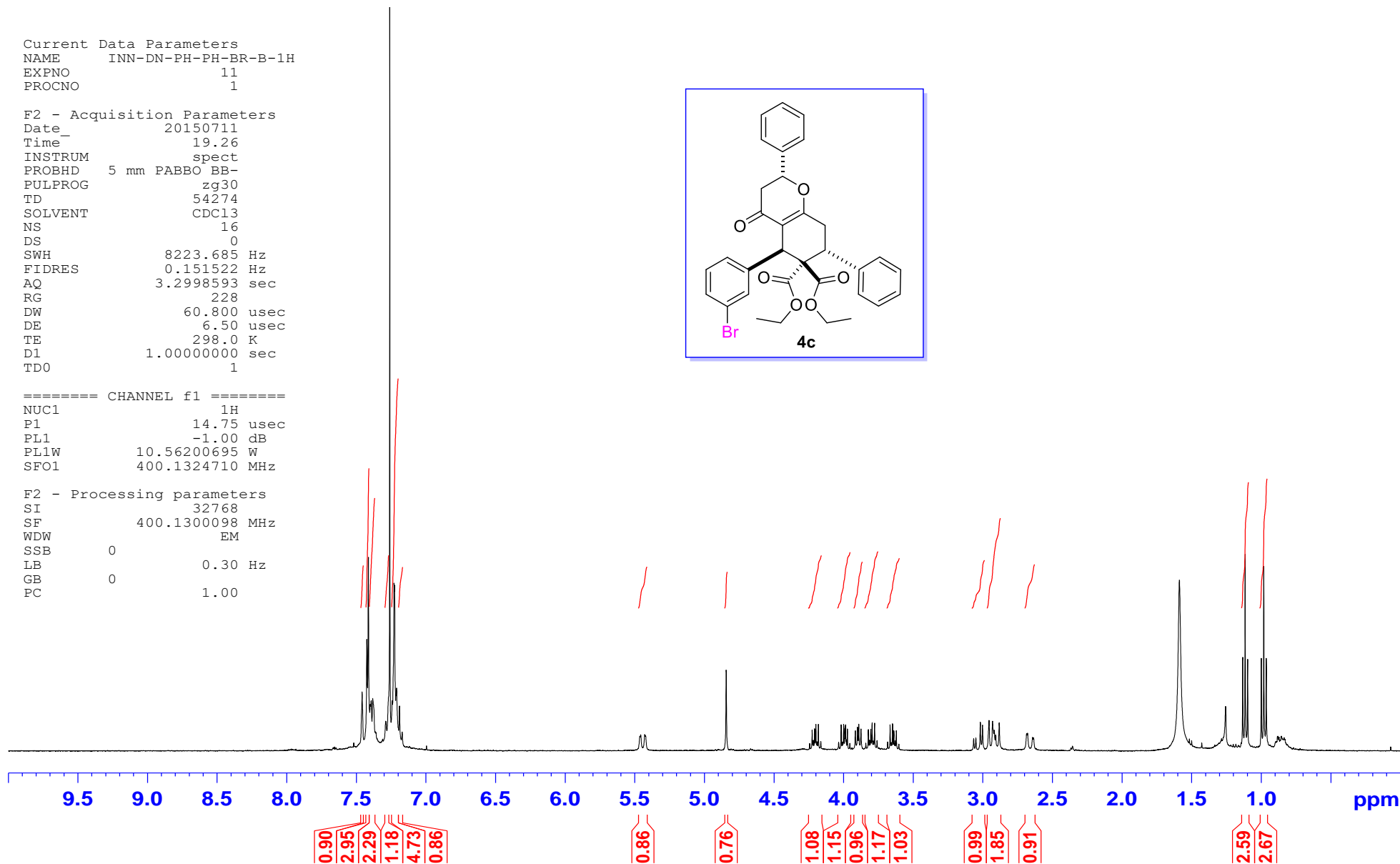
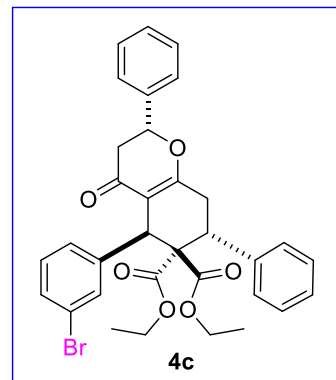


Fig S23. <sup>1</sup>H NMR Spectrum of 4c

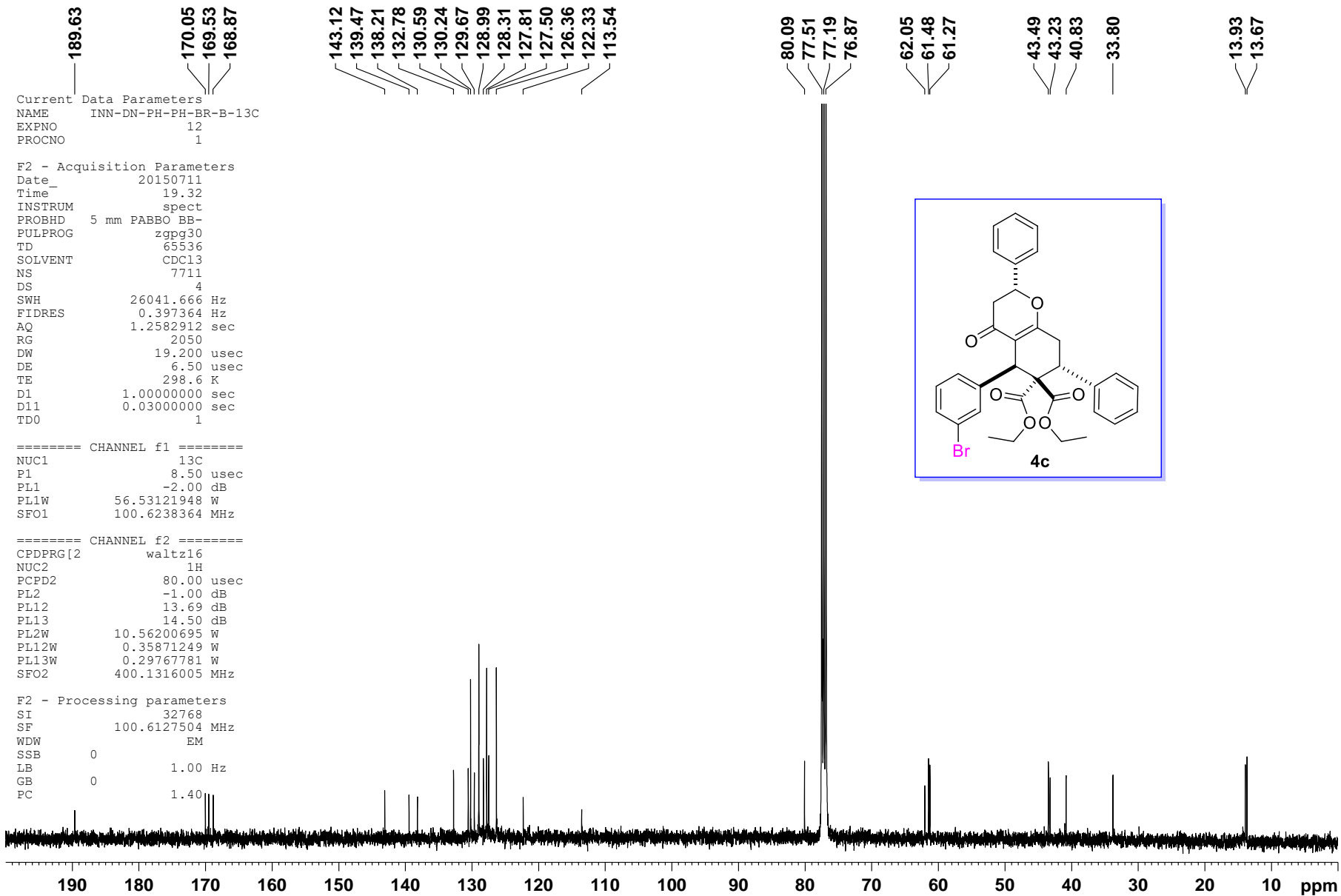


Fig S24. <sup>13</sup>C NMR Spectrum of 4c

Current Data Parameters  
NAME INN-DN-PH-PH-ME-1-1H  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20170528  
Time\_ 10.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 299.6 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1340013 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300243 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

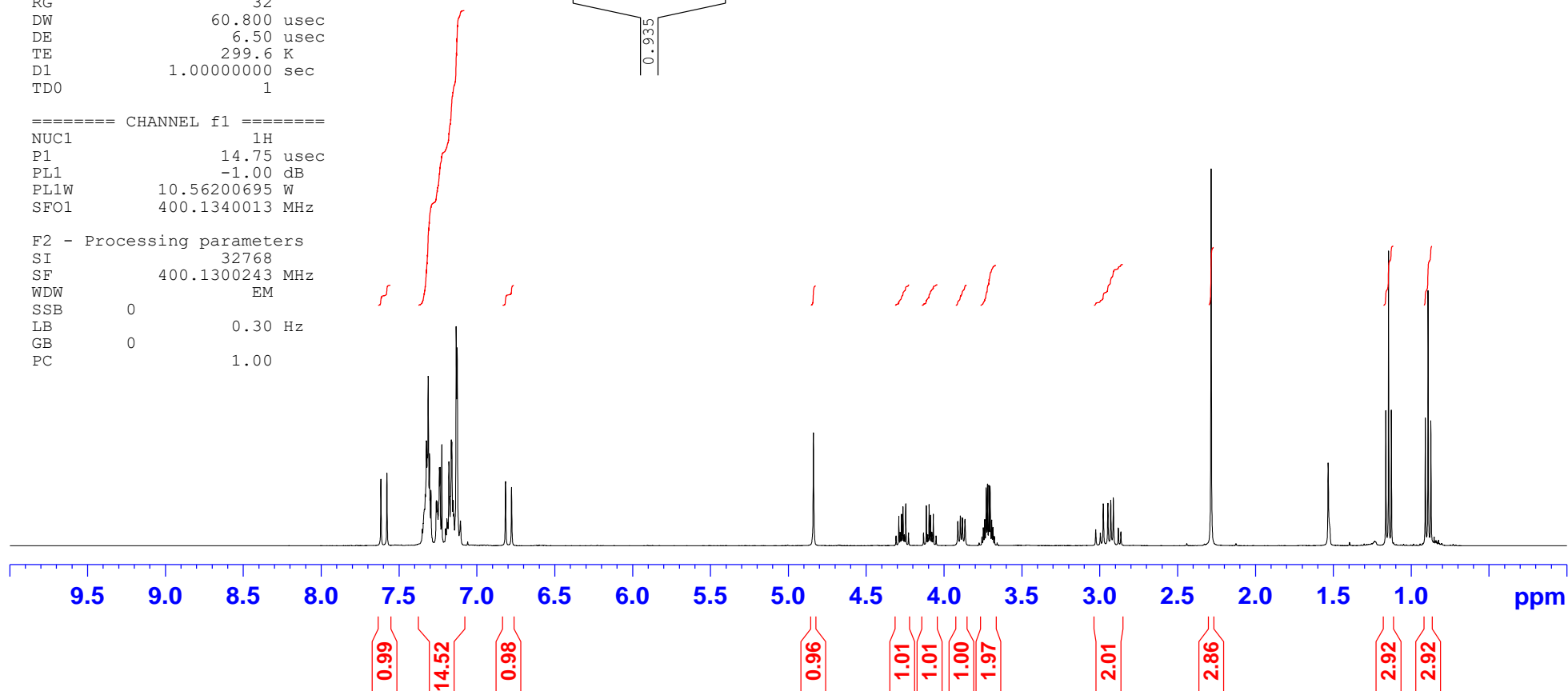
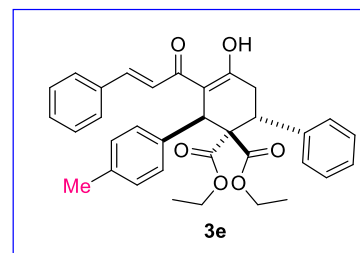
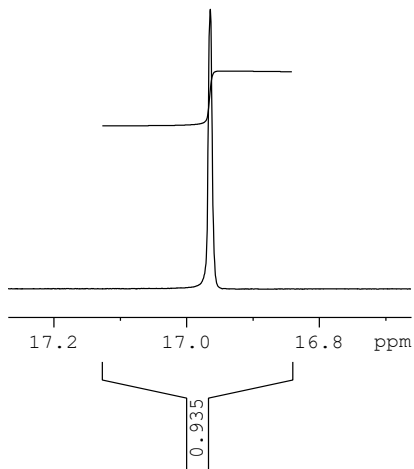


Fig S25. <sup>1</sup>H NMR Spectrum of 3e

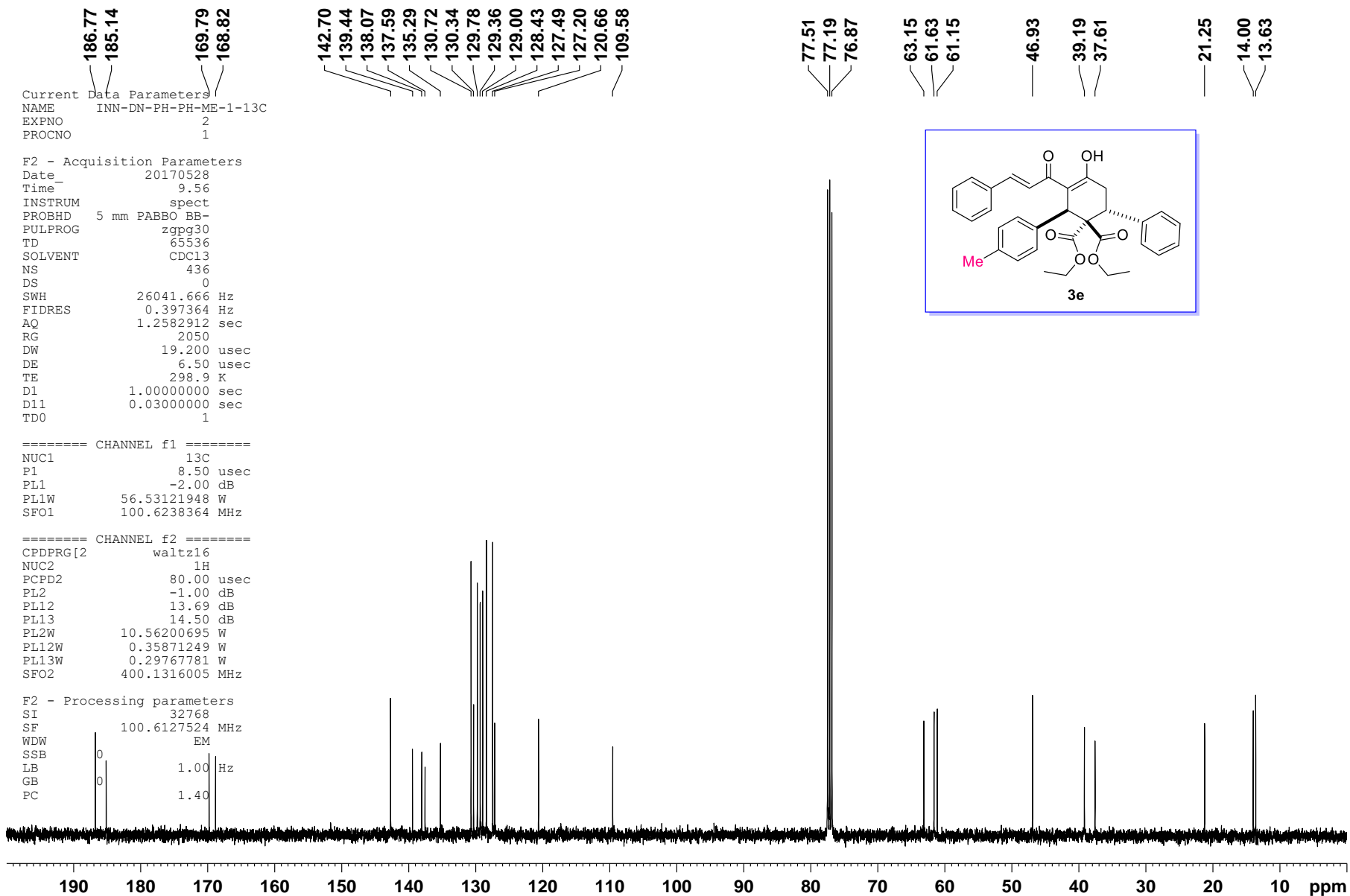
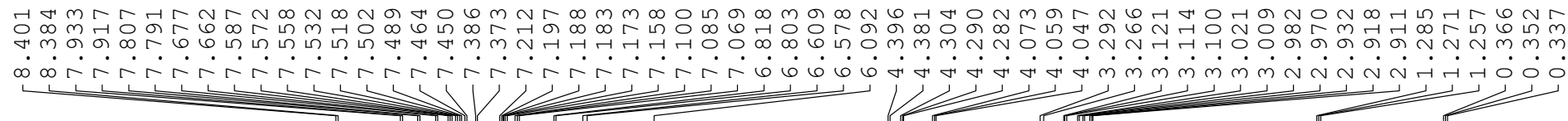


Fig S26. <sup>13</sup>C NMR Spectrum of 3e

INN-ART-36A2-1H



Current Data Parameters  
NAME INN-ART-36A2-1H  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210223  
Time 16.44  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 25  
DS 0  
SWH 12500.000 Hz  
FIDRES 0.190735 Hz  
AQ 2.6214399 sec  
RG 106.54  
DW 40.000 usec  
DE 6.50 usec  
TE 302.7 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1350013 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300131 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

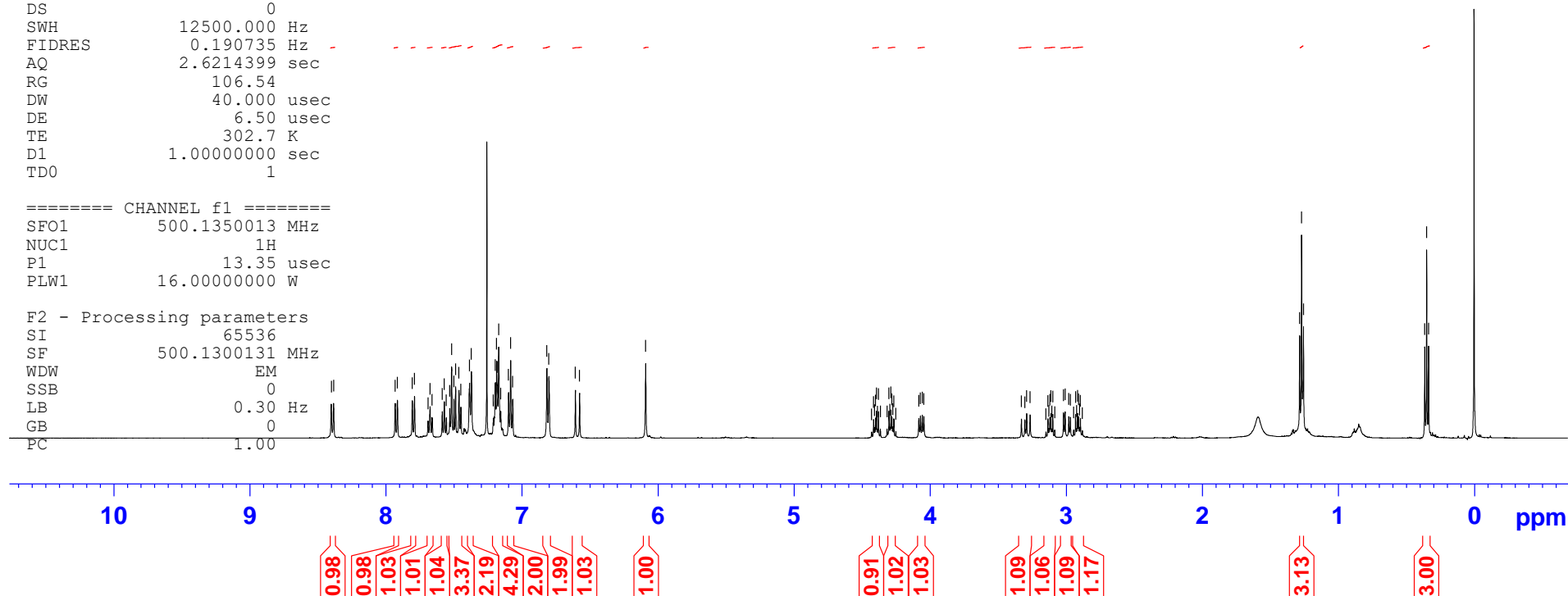
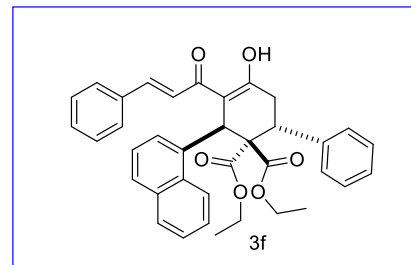


Fig S27. <sup>1</sup>H NMR spectrum of 3f

INN-ART-36A2-COSY

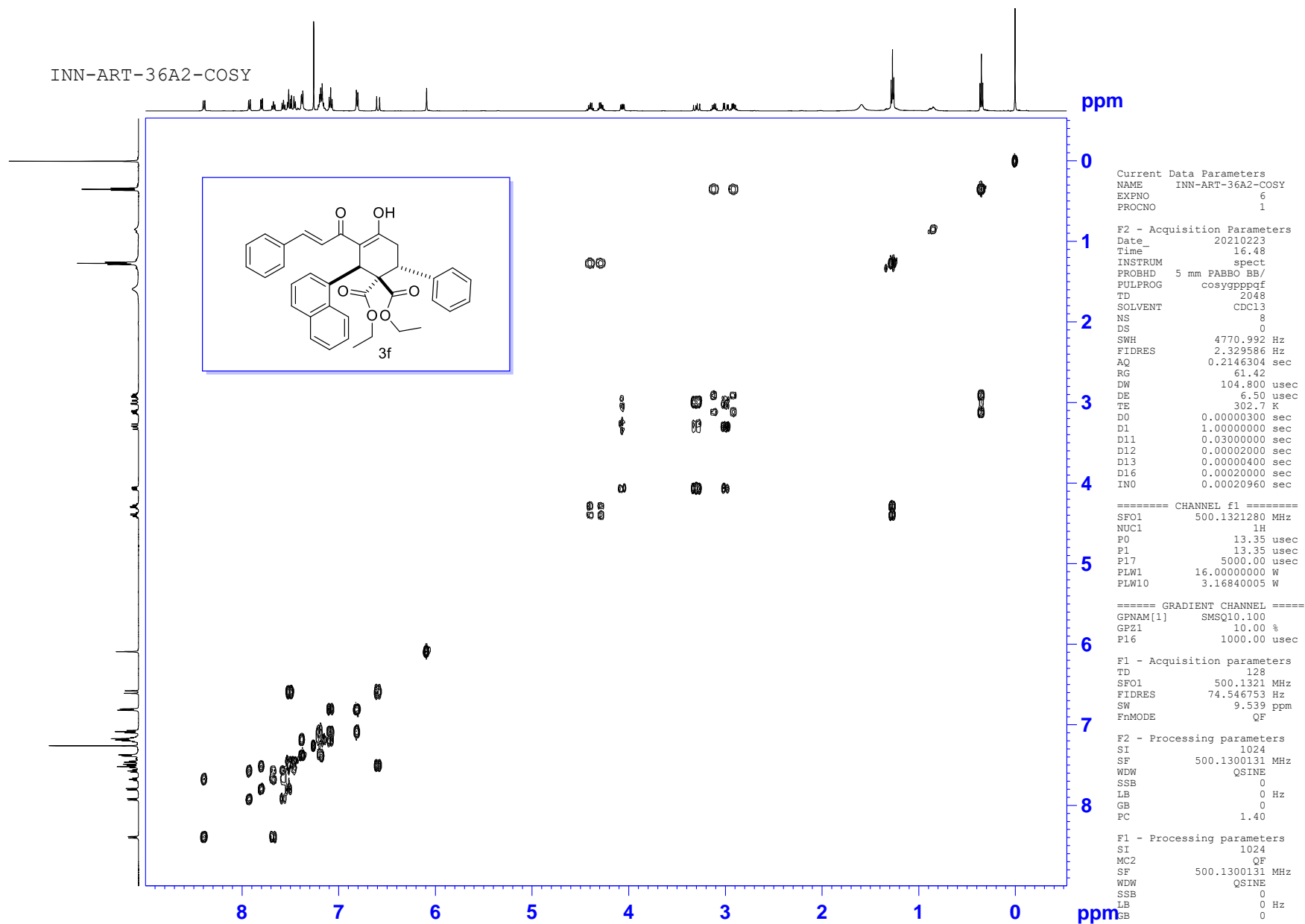


Fig S28. <sup>1</sup>H-<sup>1</sup>H COSY NMR Spectrum of 3f

INN-ART-36-A2-13C

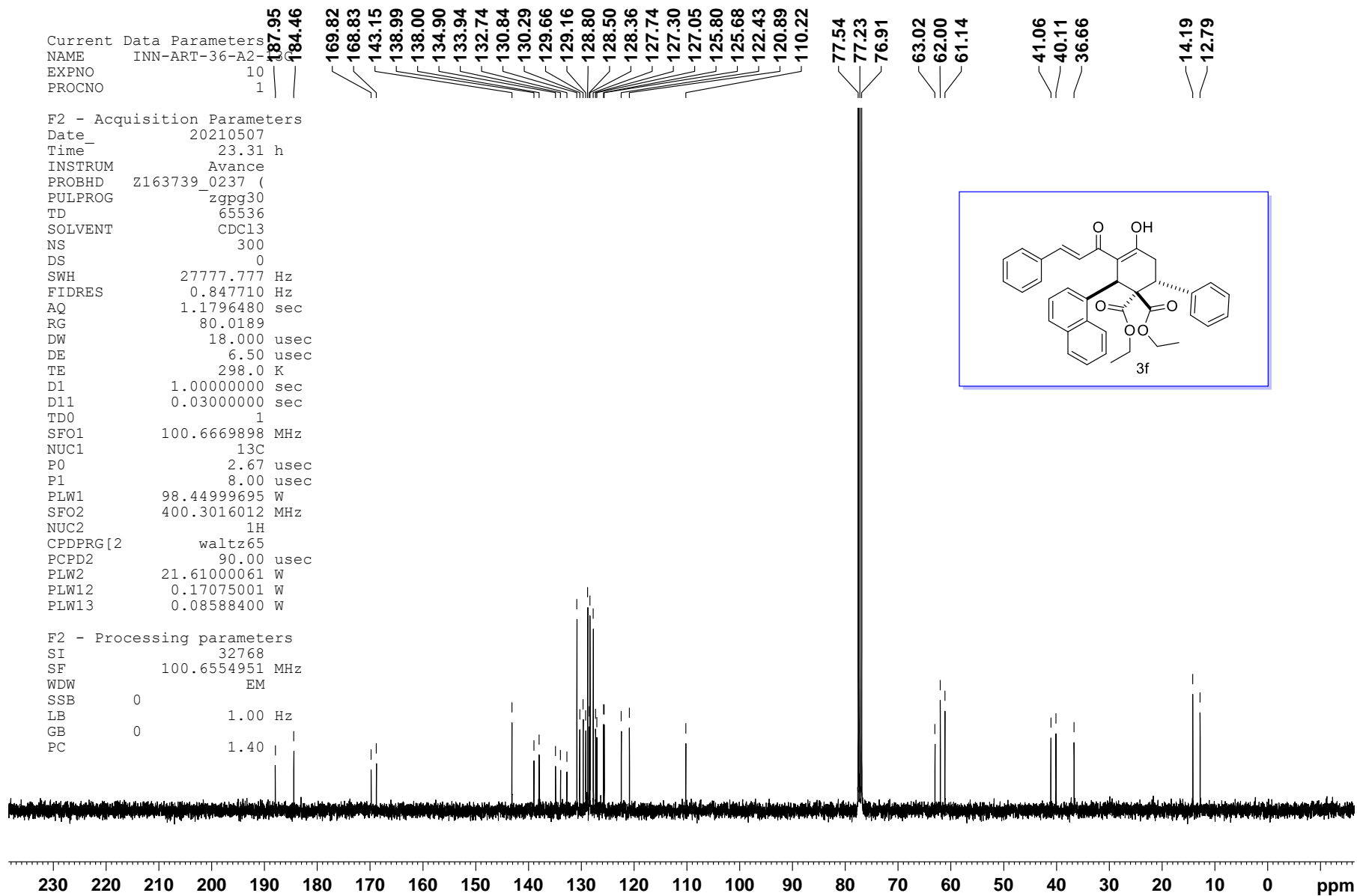


Fig S29. <sup>13</sup>C NMR spectrum of 3f



INN-ART-36-A2-13C

— 143.15

— 138.99

— 138.00

— 134.90

— 133.94

— 132.74

— 130.84

— 130.29

— 129.66

— 129.16

— 128.80

— 128.50

— 128.36

— 127.74

— 127.30

— 127.05

— 125.80

— 125.68

— 122.43

— 120.89

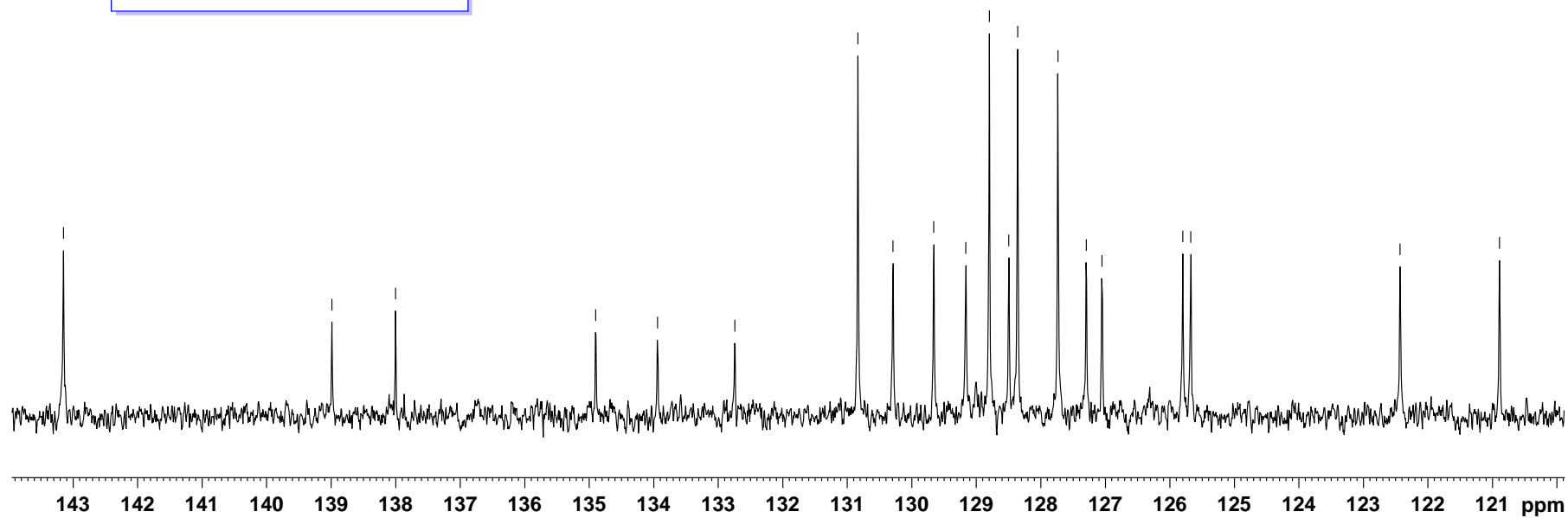
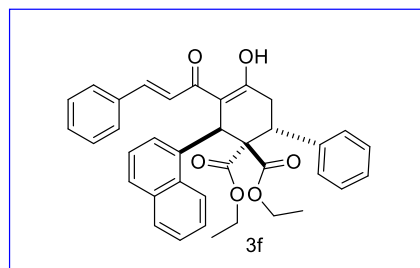


Fig S30.  $^{13}\text{C}$  NMR spectrum (expansion) of 3f

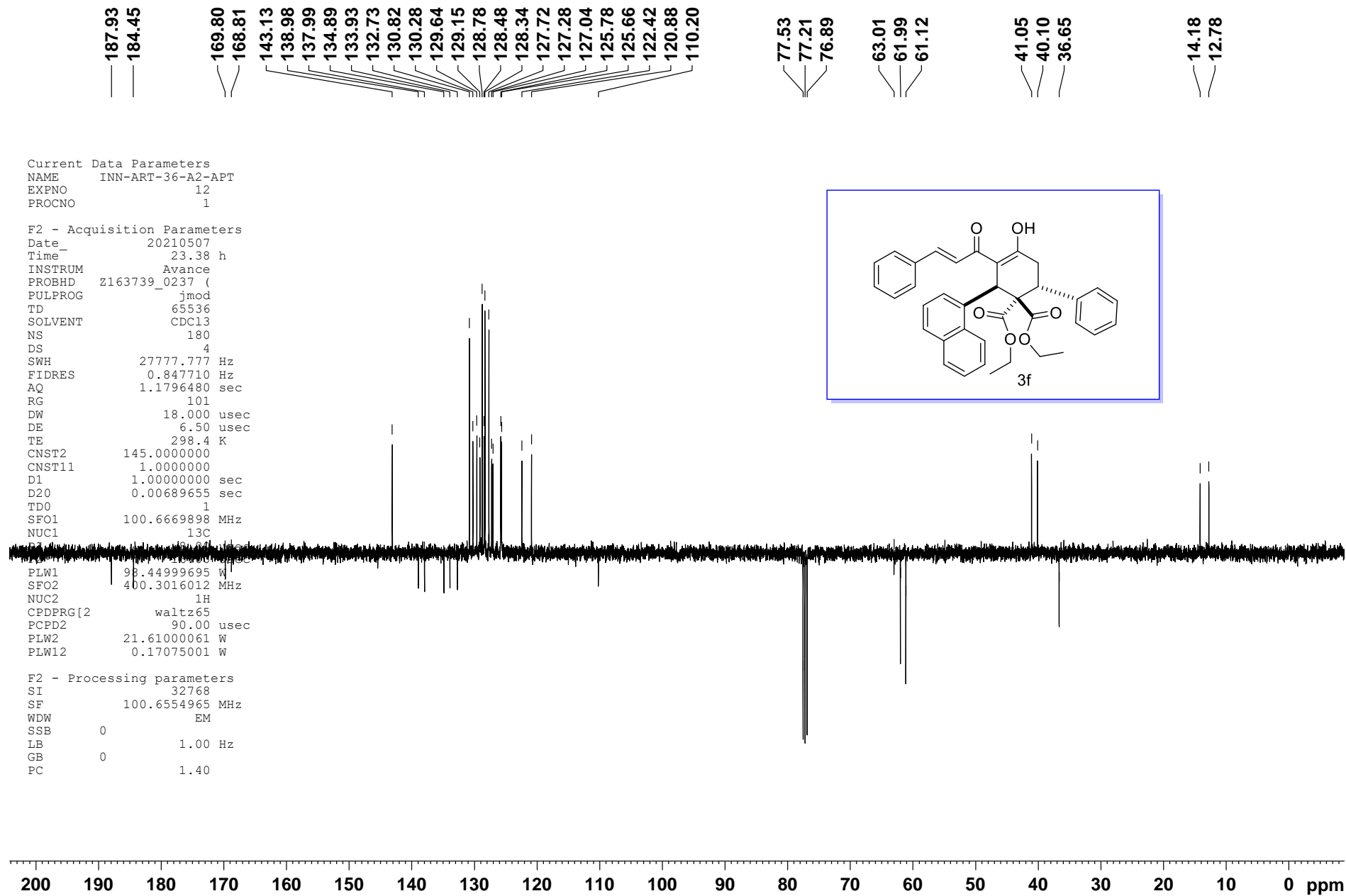


Fig S31. <sup>13</sup>C-APT spectrum of 3f

INN-ART-24A1-1H

7.731  
7.700  
7.473  
7.466  
7.459  
7.455  
7.405  
7.402  
7.377  
7.370  
7.364  
7.347  
7.334  
7.260  
7.241  
7.225  
7.222  
6.852  
6.821  
6.365  
6.361  
6.359  
6.355  
6.237  
6.231  
5.019  
4.283  
4.269  
4.261  
4.247  
4.124  
4.110  
4.103  
4.089  
4.013  
4.001  
3.989  
3.977  
3.934  
3.920  
3.906  
3.892  
3.071  
3.047  
3.033  
3.009  
2.915  
2.903  
2.877  
2.864  
1.181  
1.167  
1.153  
1.039  
1.025  
1.010

Current Data Parameters  
NAME INN-ART-24A1-1H  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210122  
Time\_ 20.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 9  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 80.35  
DW 50.000 usec  
DE 6.50 usec  
TE 296.3 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300131 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

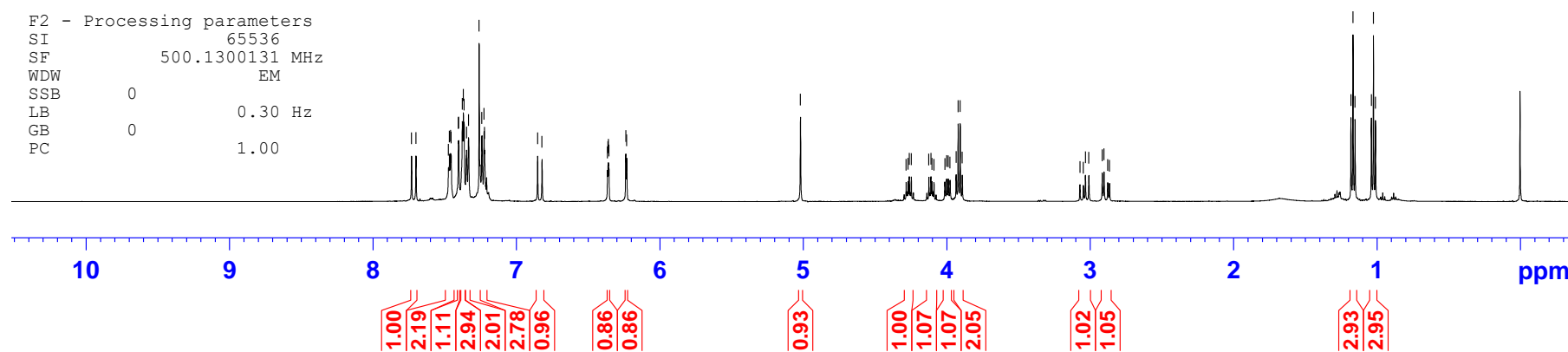
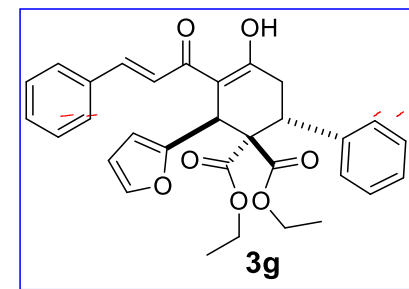


Fig S32. <sup>1</sup>H NMR spectrum of 3g

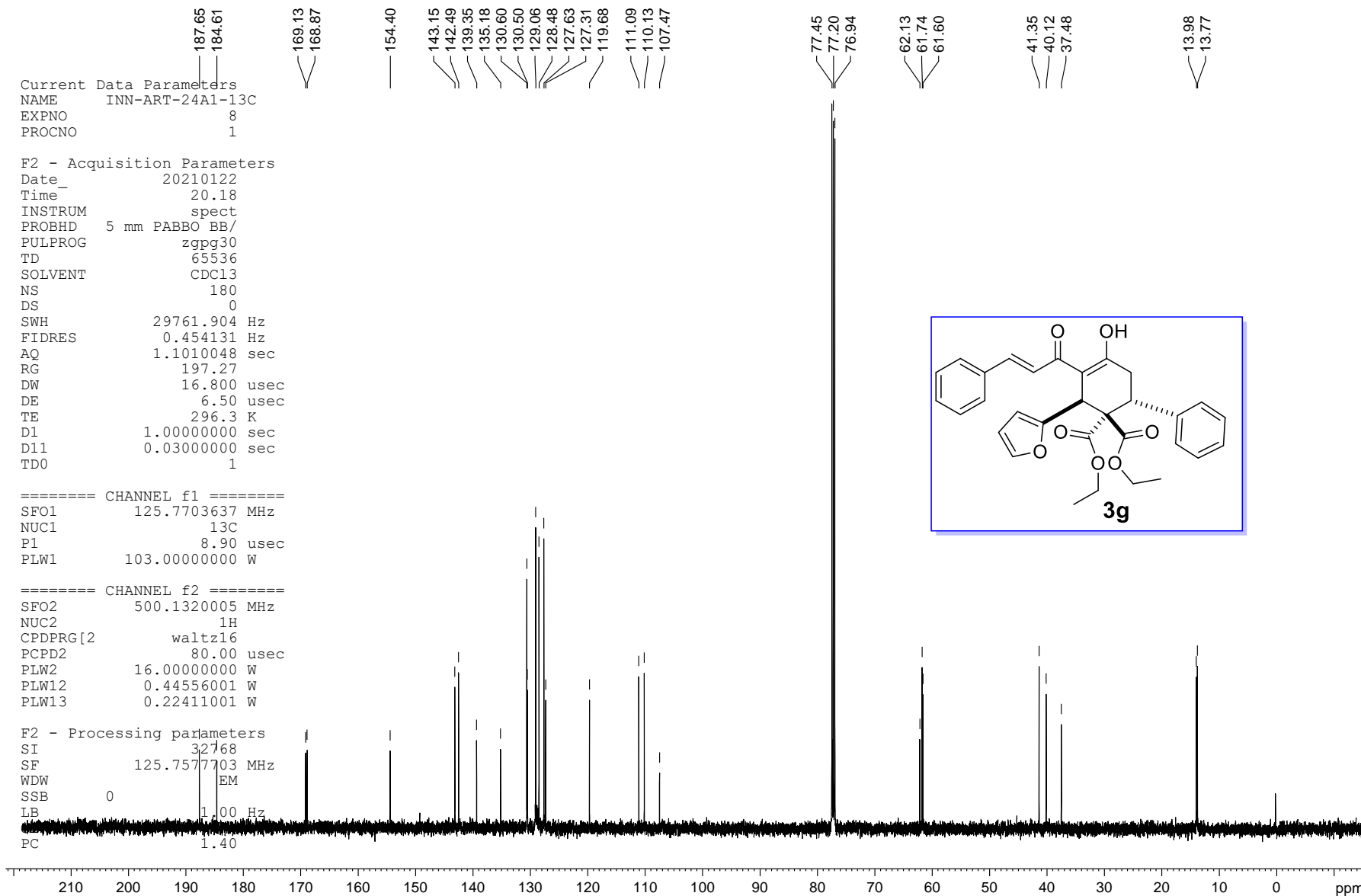


Fig S33. <sup>13</sup>C NMR spectrum of 3g

Current Data Parameters  
NAME INN-DN-PH-ME-PH-CL-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20150711  
Time 5.47  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 19  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 299.3 K  
D1 1.00000000 sec  
TD0 1

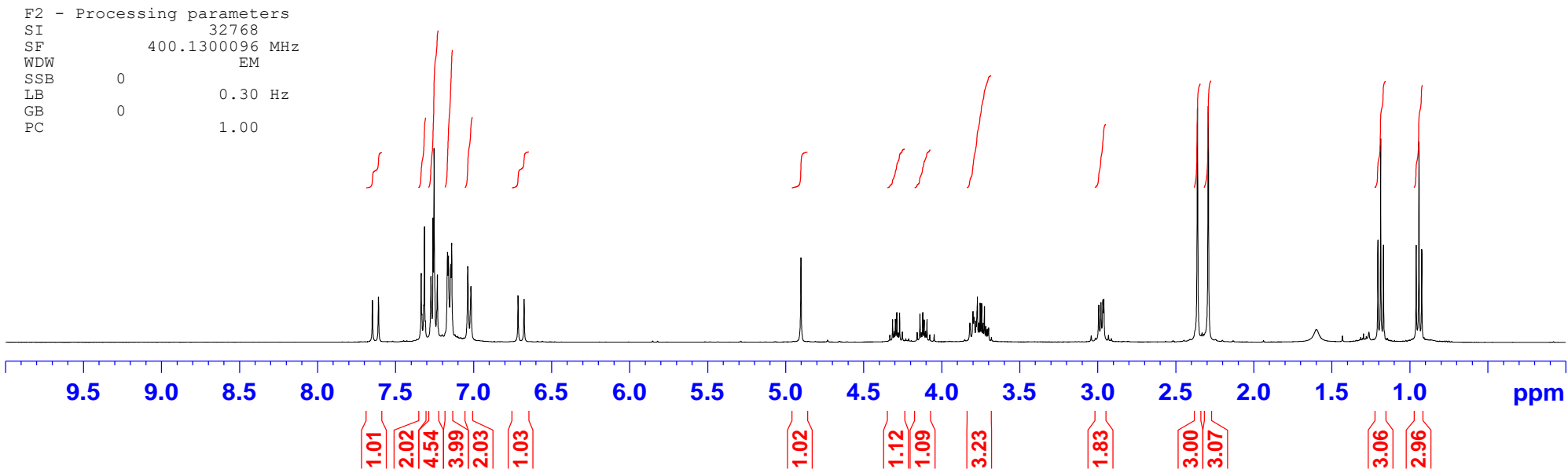
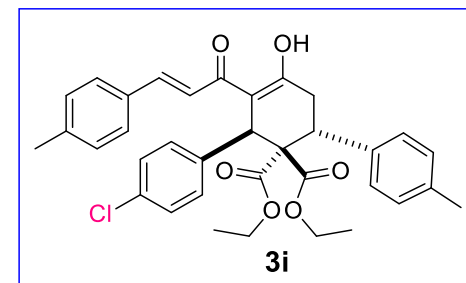
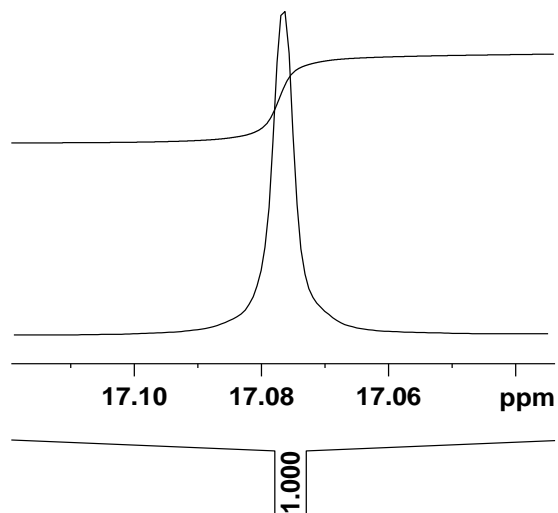


Fig S34. <sup>1</sup>H NMR spectrum of 3i

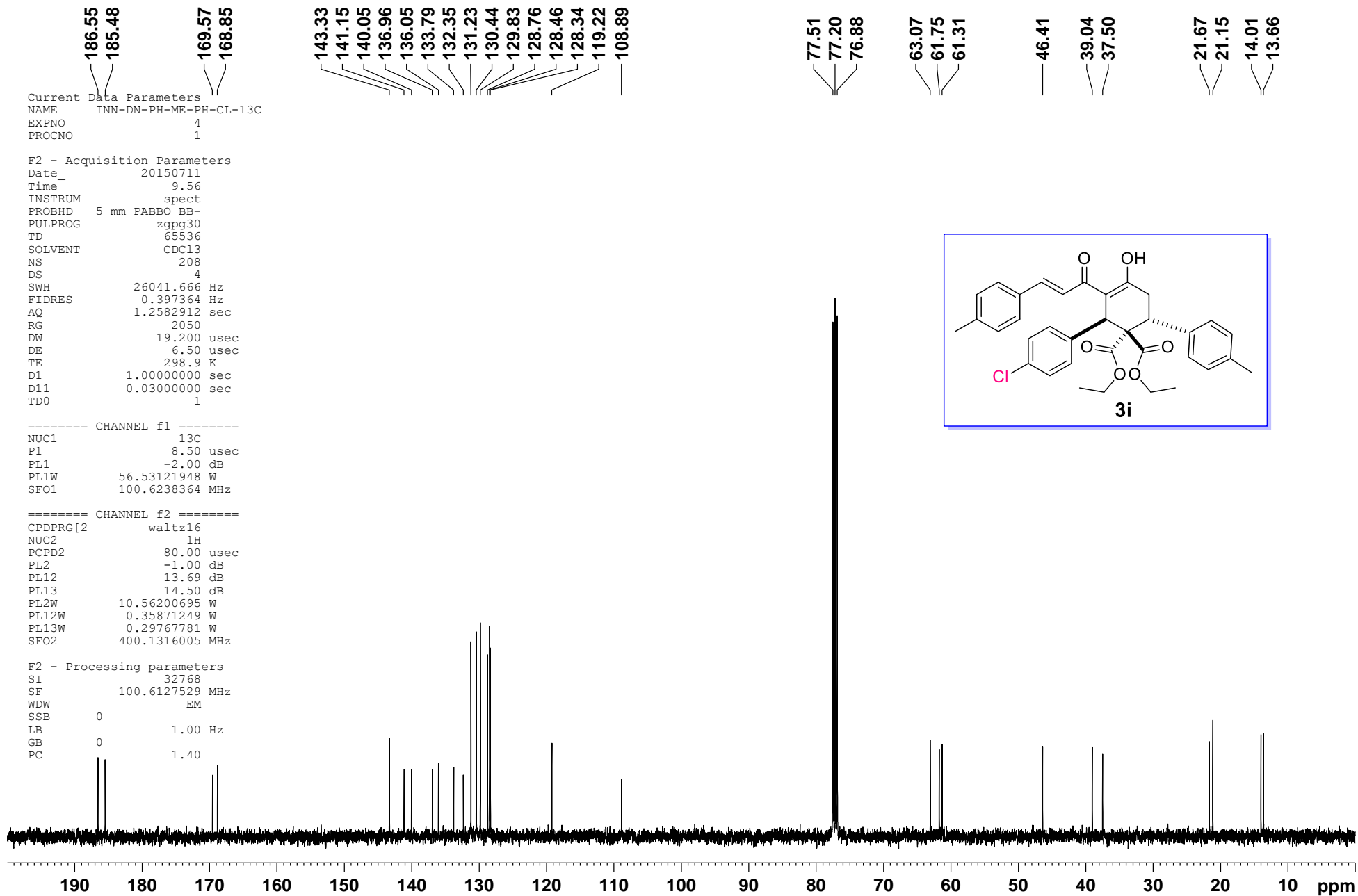


Fig S35. <sup>13</sup>C NMR spectrum of 3i

Current Data Parameters  
NAME INN-DN-PH-BR-PH-CL-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20150711  
Time\_ 5.51  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 13  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 299.4 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1340013 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300100 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

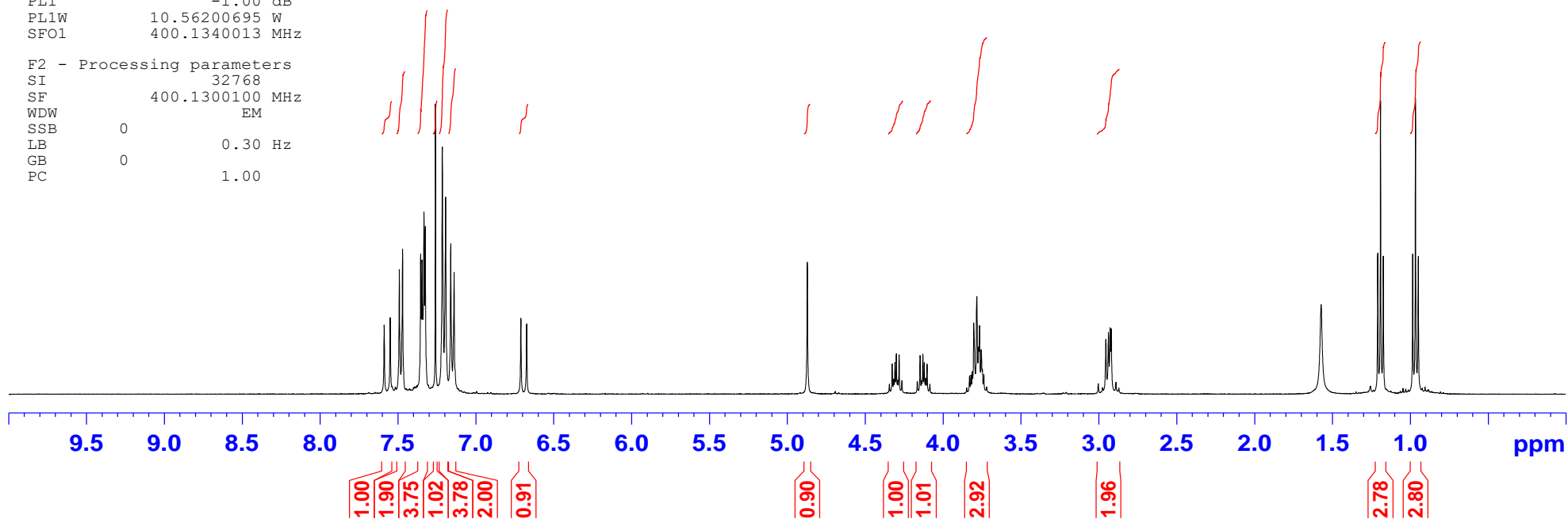
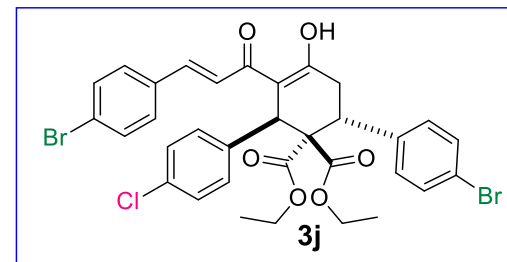
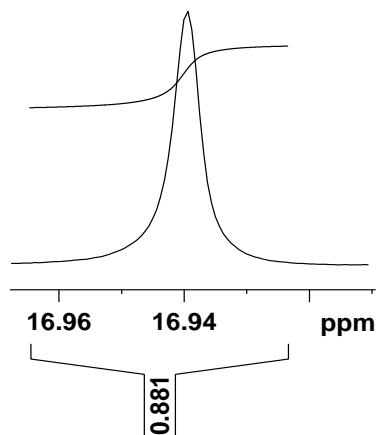


Fig S36. <sup>1</sup>H NMR spectrum of 3j

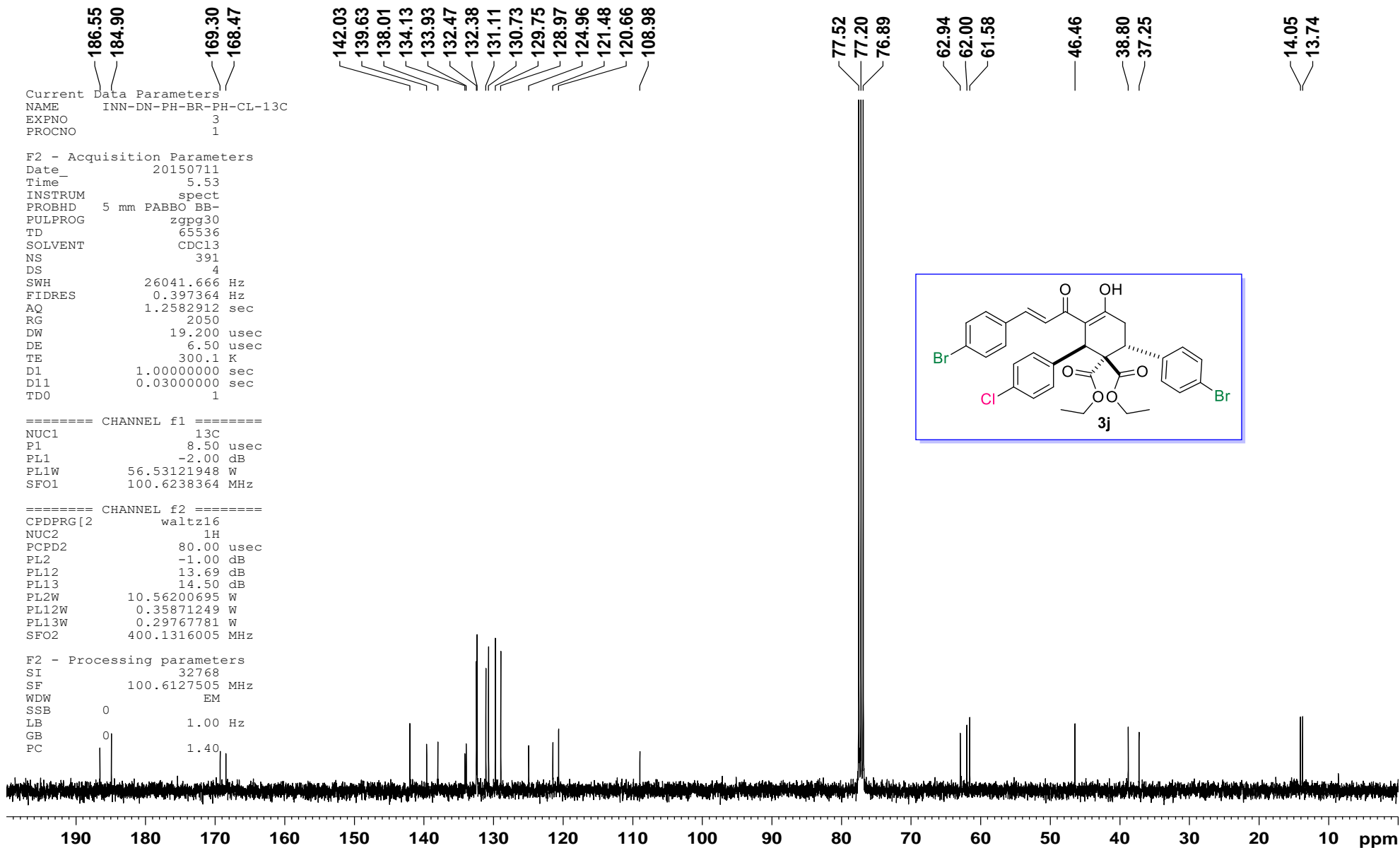


Fig S37. <sup>13</sup>C NMR Spectrum of 3j



INN-ART-41A3-1H

8.538  
8.500  
8.106  
8.100  
8.083  
7.898  
7.875  
7.861  
7.855  
7.830  
7.812  
7.807  
7.730  
7.710  
7.615  
7.597  
7.551  
7.543  
7.535  
7.526  
7.520  
7.481  
7.473  
7.464  
7.453  
7.446  
7.441  
7.423  
7.401  
7.395  
7.375  
7.364  
7.355  
7.260  
6.935  
6.897  
5.283  
4.892  
4.882  
4.232  
4.214  
4.131  
4.113  
4.104  
3.507  
3.488  
3.469  
3.387  
3.371  
3.339  
3.323  
3.139  
3.114  
1.087  
1.069  
1.051  
0.542  
0.524  
0.506

Current Data Parameters  
NAME INN-ART-41A3-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210224  
Time\_ 1.47  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDC13  
NS 15  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 326.0 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300098 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

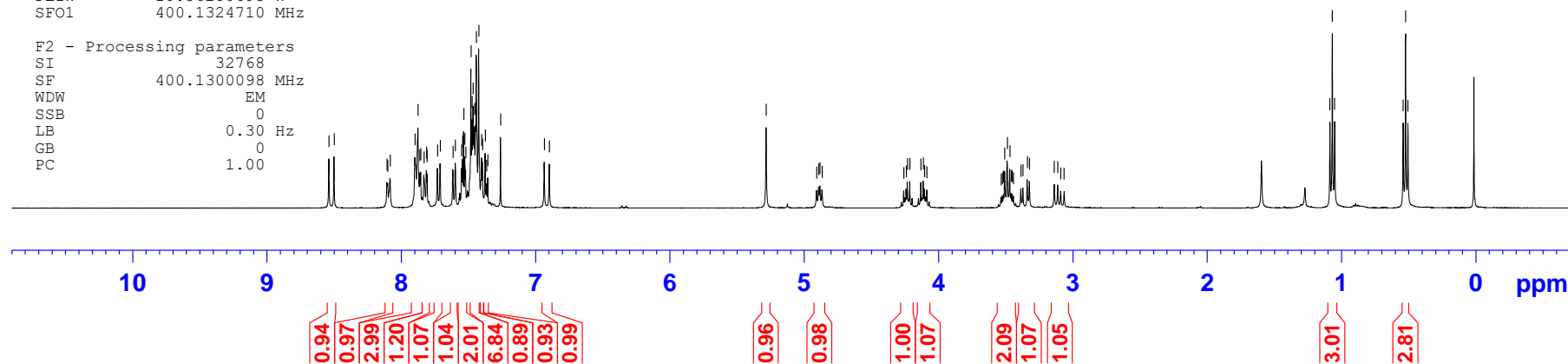
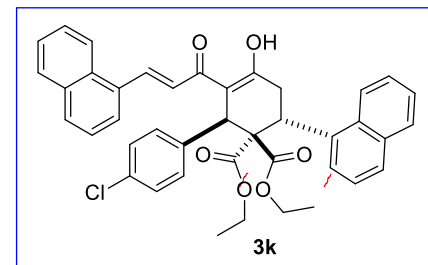


Fig S38. <sup>1</sup>H NMR spectrum of 3k

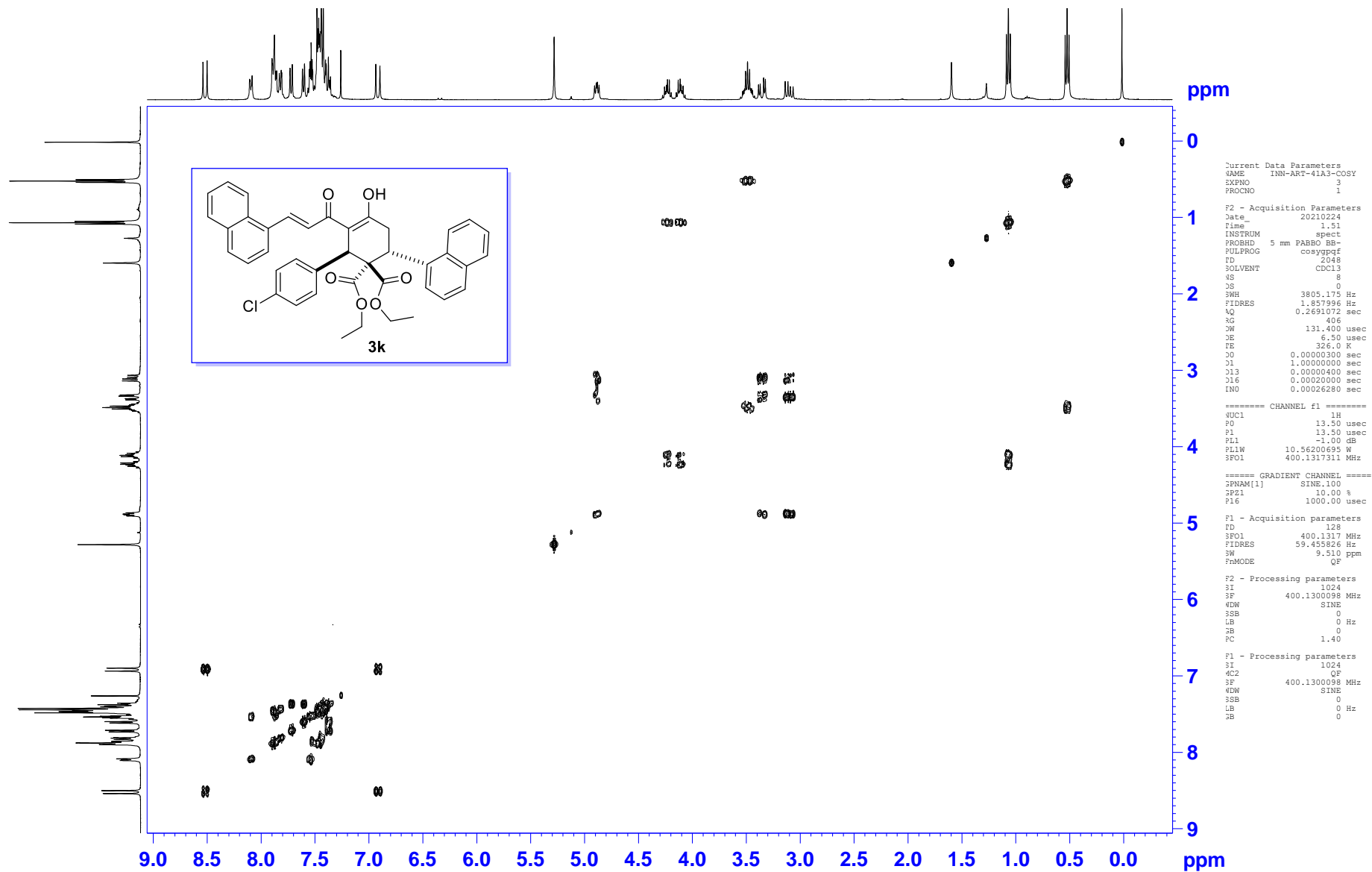


Fig S39.  $^1\text{H}$ - $^1\text{H}$  COSY NMR spectrum of 3k

INN-ART-41-A3-13C

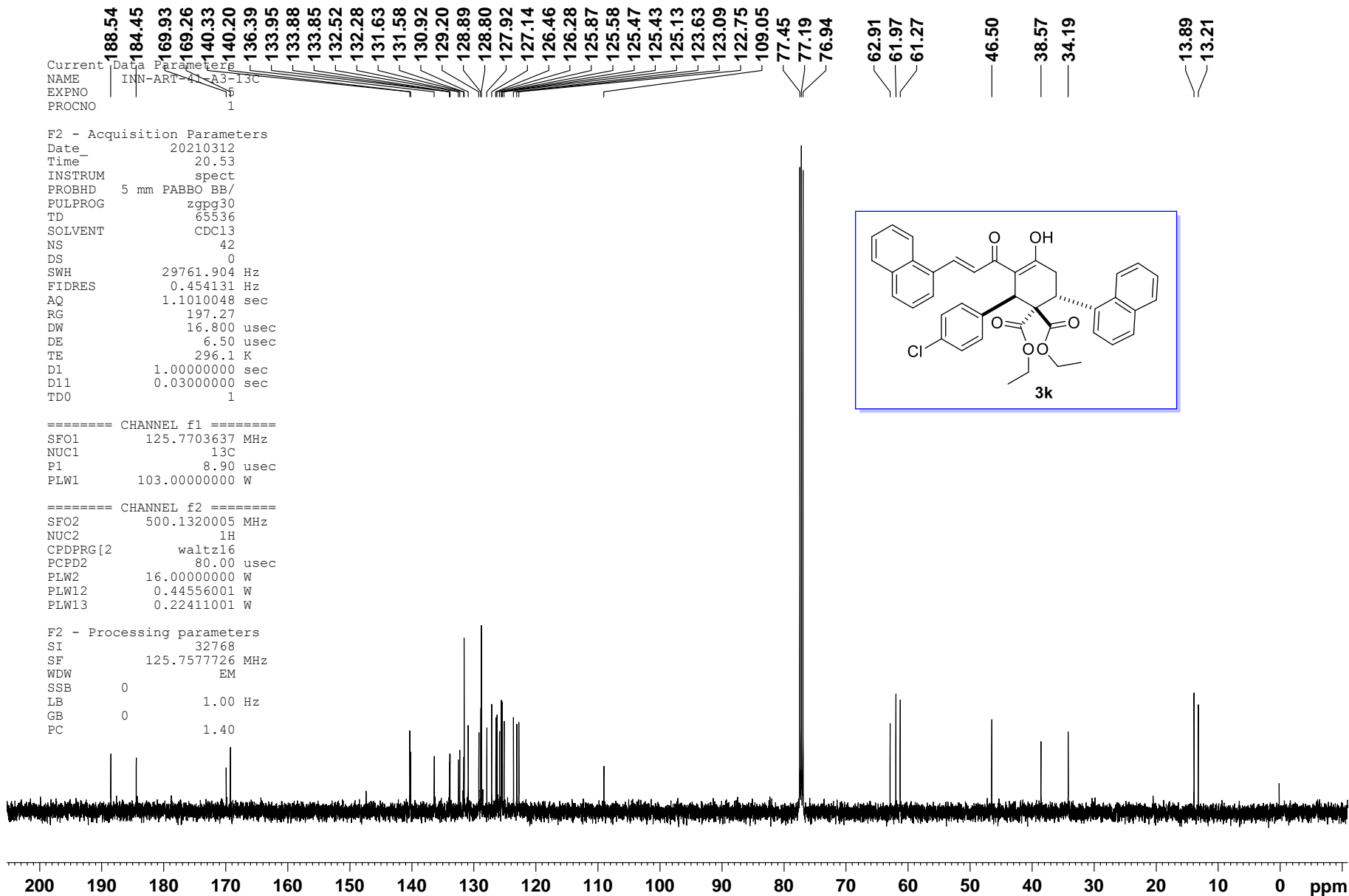


Fig S40. <sup>13</sup>C NMR spectrum of 3k

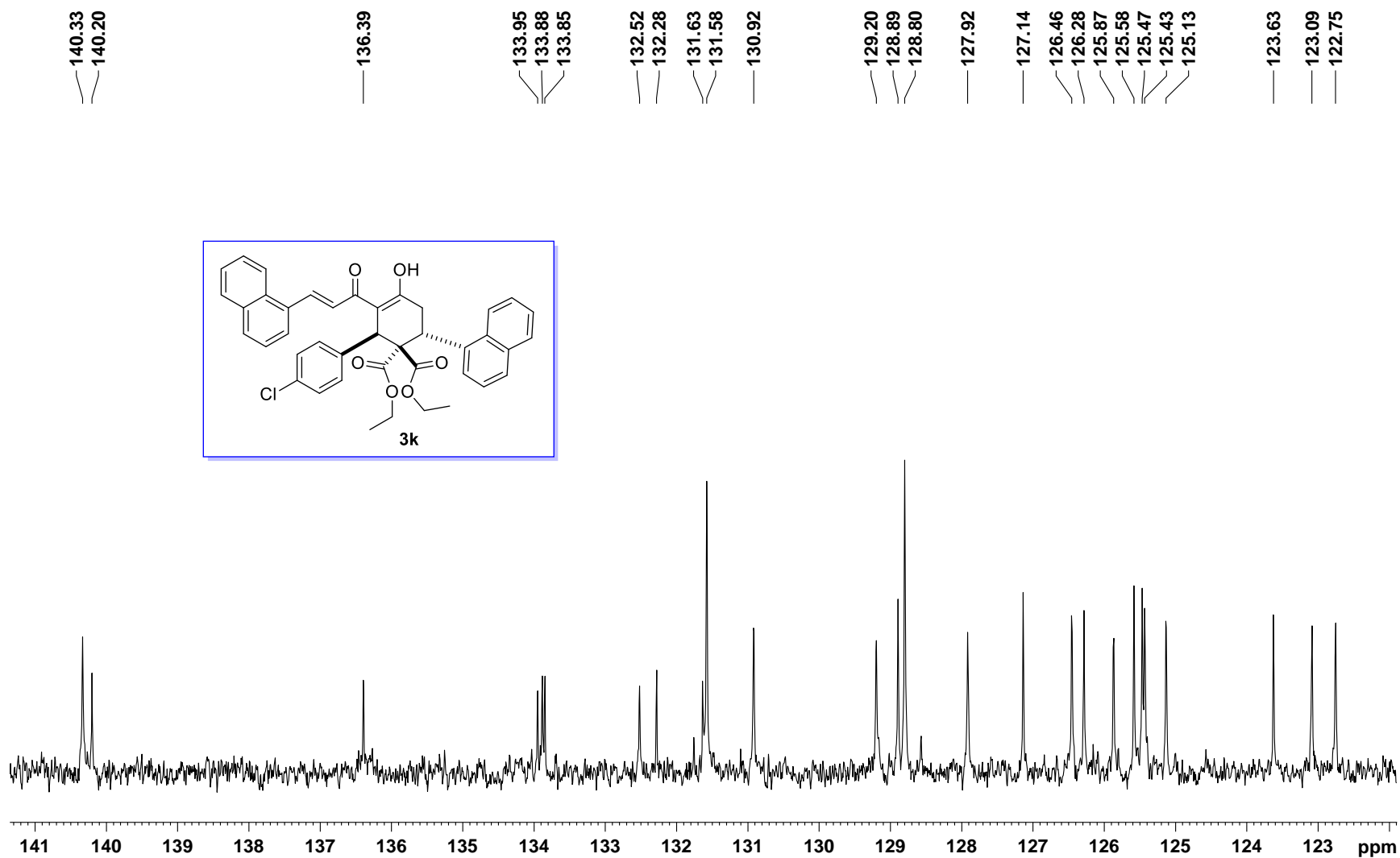
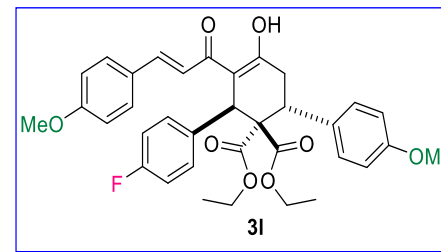
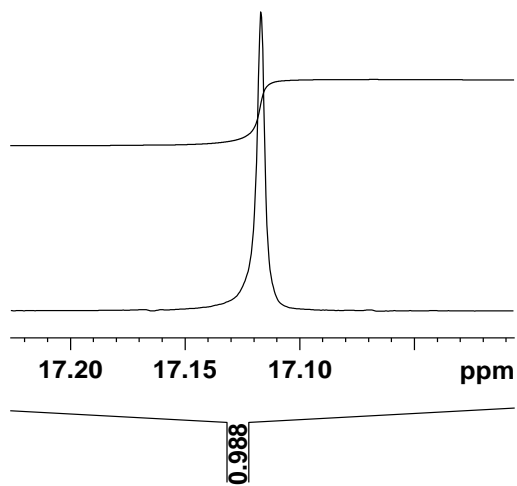


Fig S41. <sup>13</sup>C NMR spectrum expansion of 3k

Current Data Parameters  
NAME INN-DN-PH-OME-PH-F-A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20171110  
Time\_ 12.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 10  
DS 0  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845334 sec  
RG 30.72  
DW 33.333 usec  
DE 6.50 usec  
TE 299.7 K  
D1 1.00000000 sec  
TD0 1



==== CHANNEL f1 =====  
SFO1 500.1360016 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters

SI 65536  
SF 500.1300135 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

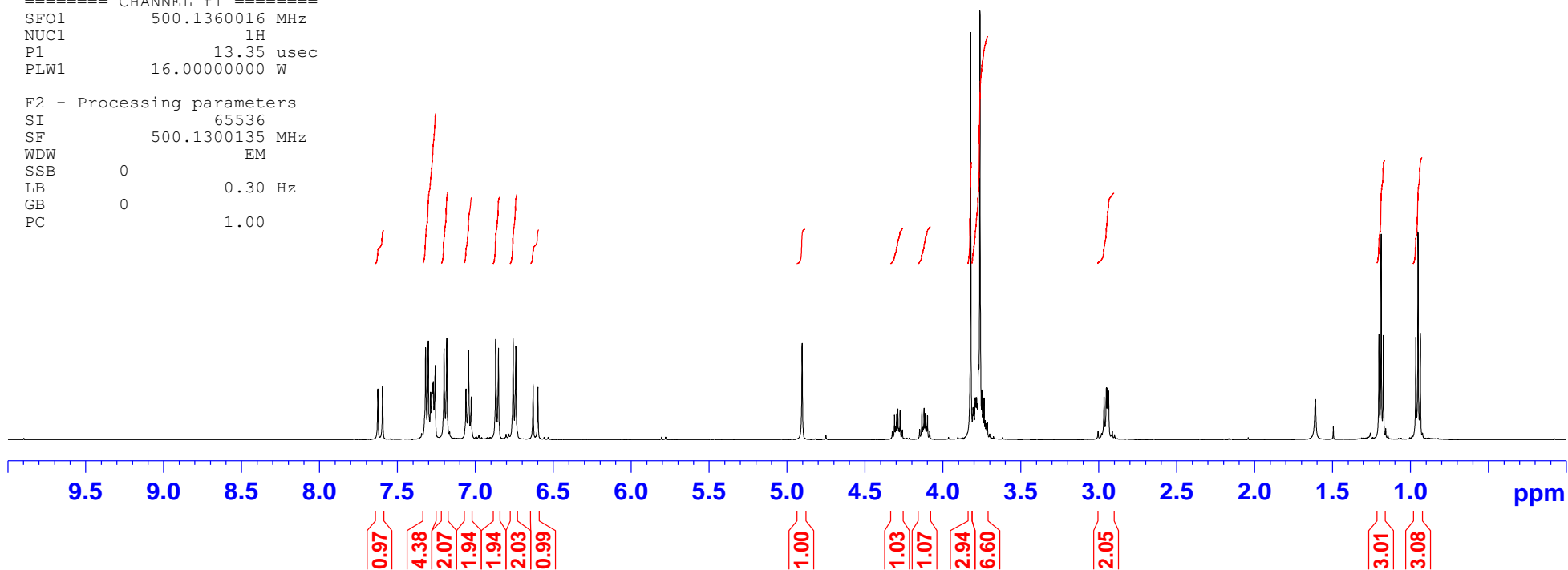


Fig S42. <sup>1</sup>H NMR Spectrum of 31

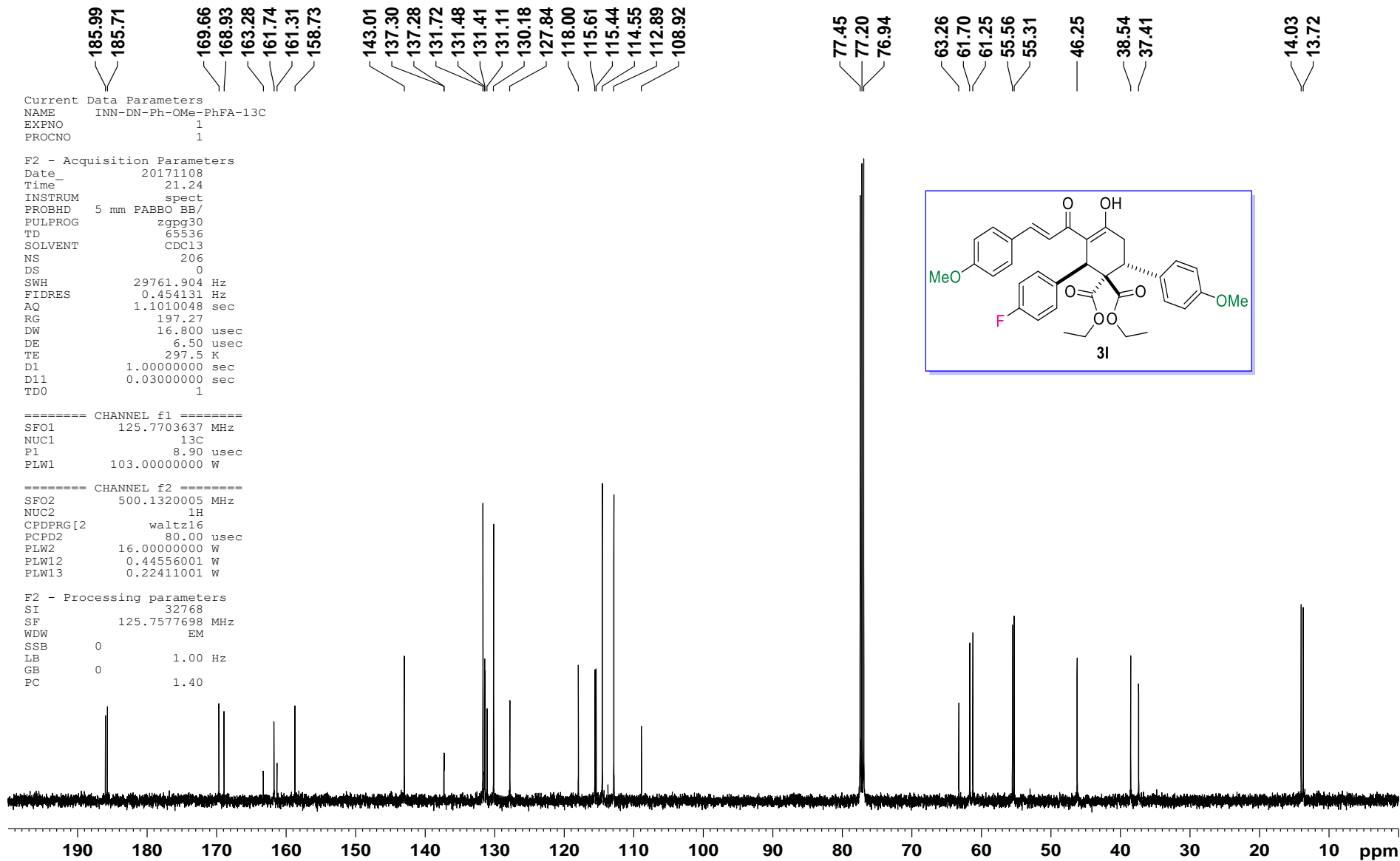


Fig S43. <sup>13</sup>C NMR Spectrum of 3I

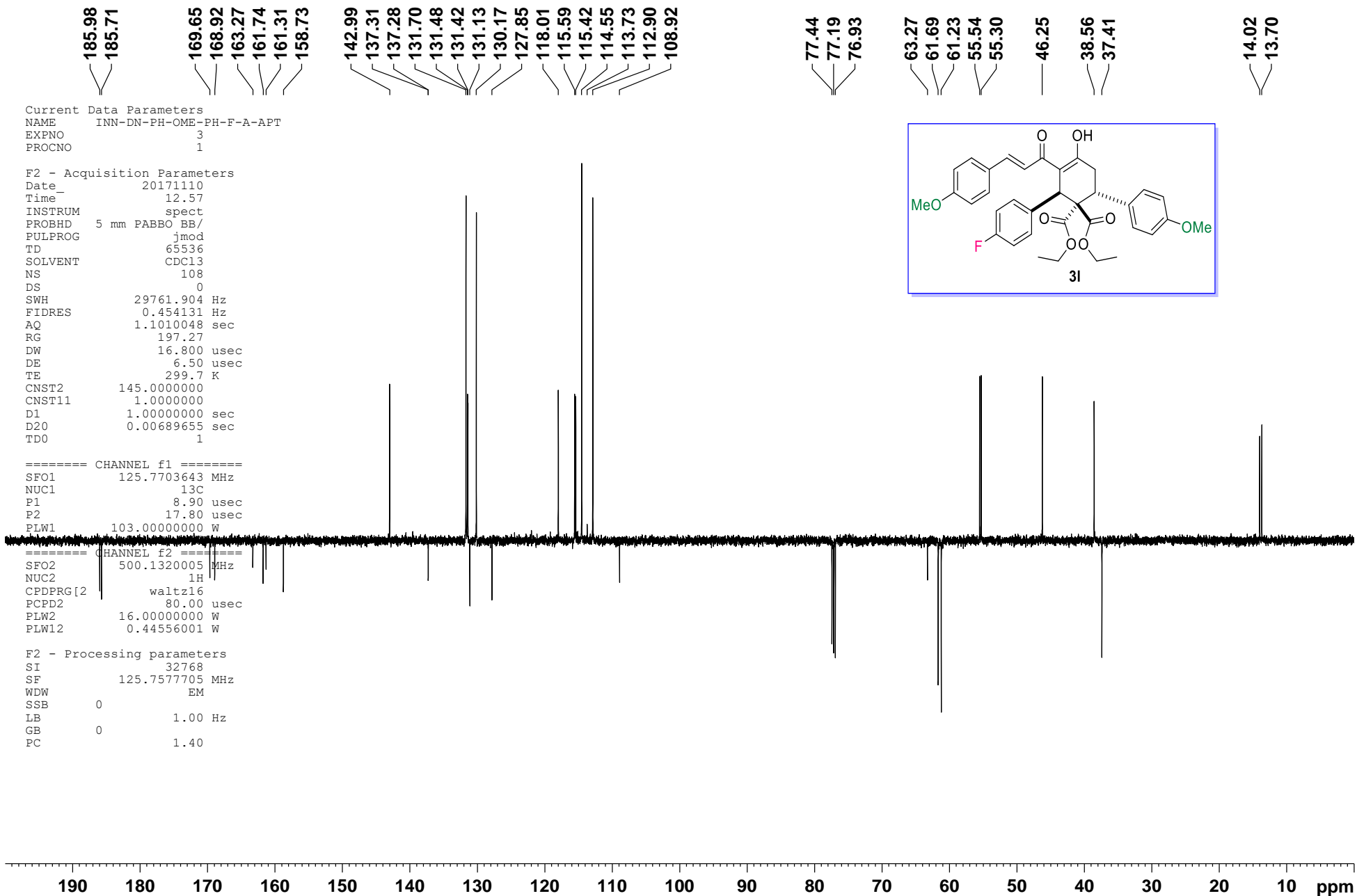


Fig S44. APT spectrum of 31

Current Data Parameters  
NAME INN-ART-45-A1-19F  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210508  
Time\_ 9.46  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgflqn  
TD 131072  
SOLVENT CDCl3  
NS 30  
DS 0  
SWH 113636.367 Hz  
FIDRES 0.866977 Hz  
AQ 0.5767168 sec  
RG 119.07  
DW 4.400 usec  
DE 6.50 usec  
TE 297.7 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 470.5453180 MHz  
NUC1 19F  
P1 19.75 usec  
PLW1 55.00000000 W

F2 - Processing parameters  
SI 65536  
SF 470.5923770 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

-114.40  
-114.41

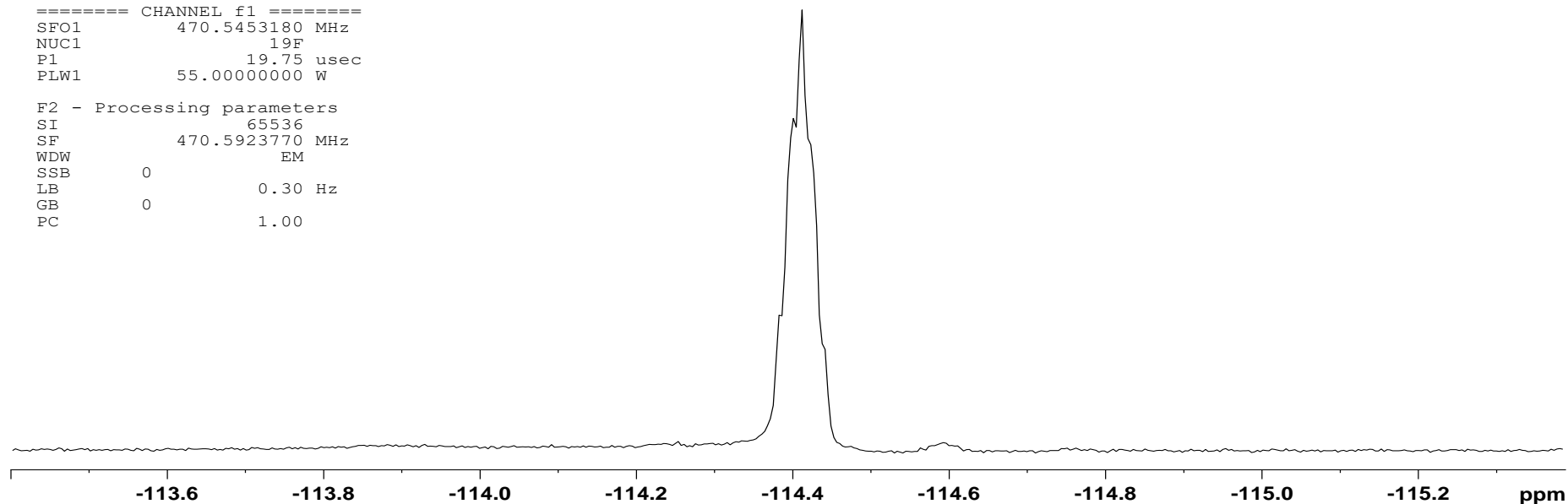
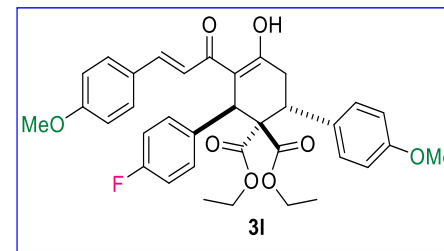


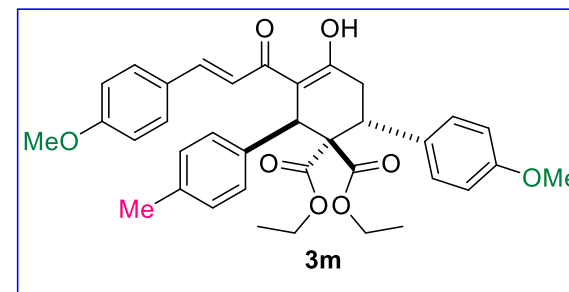
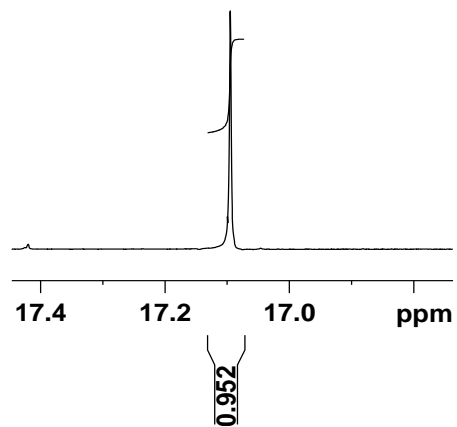
Fig S45. <sup>19</sup>F NMR spectrum of 31



Current Data Parameters  
NAME INN-DN-PH-OME-PH-ME-A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20171110  
Time\_ 12.53  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 11  
DS 0  
SWH 15000.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845334 sec  
RG 30.72  
DW 33.333 usec  
DE 6.50 usec  
TE 299.7 K  
D1 1.00000000 sec  
TD0 1



==== CHANNEL f1 =====  
SFO1 500.1360016 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters

SI 65536  
SF 500.1300130 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

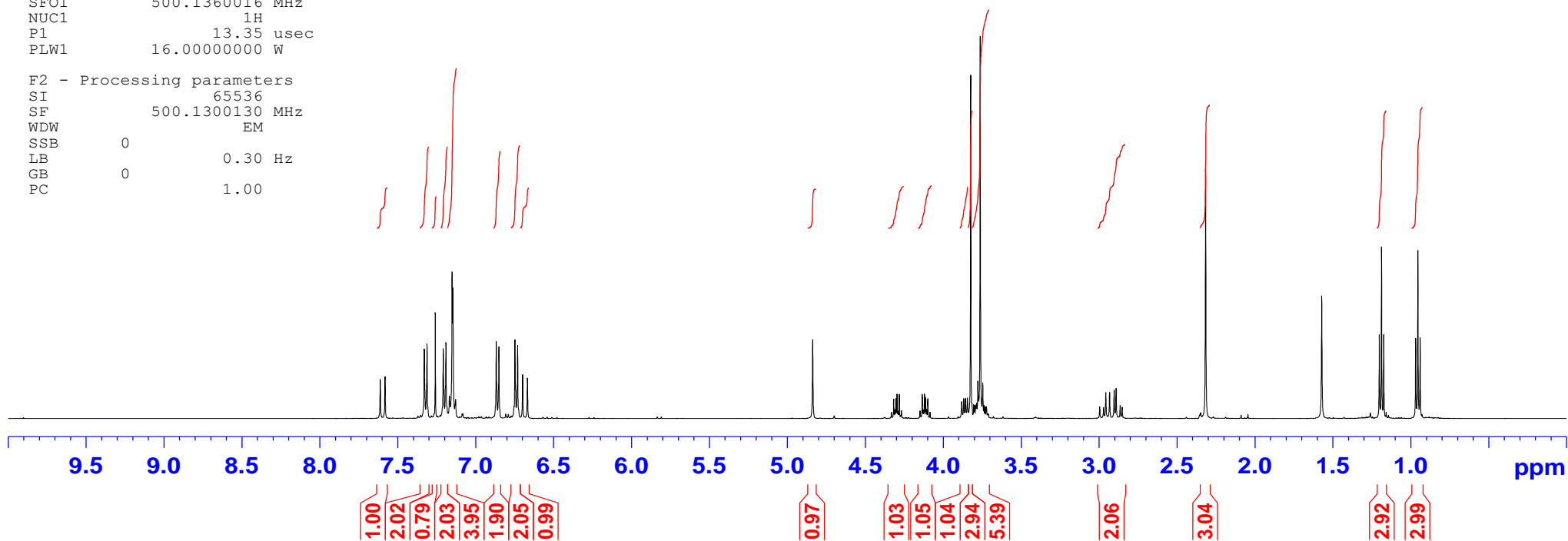


Fig S46. <sup>1</sup>H NMR Spectrum of 3m

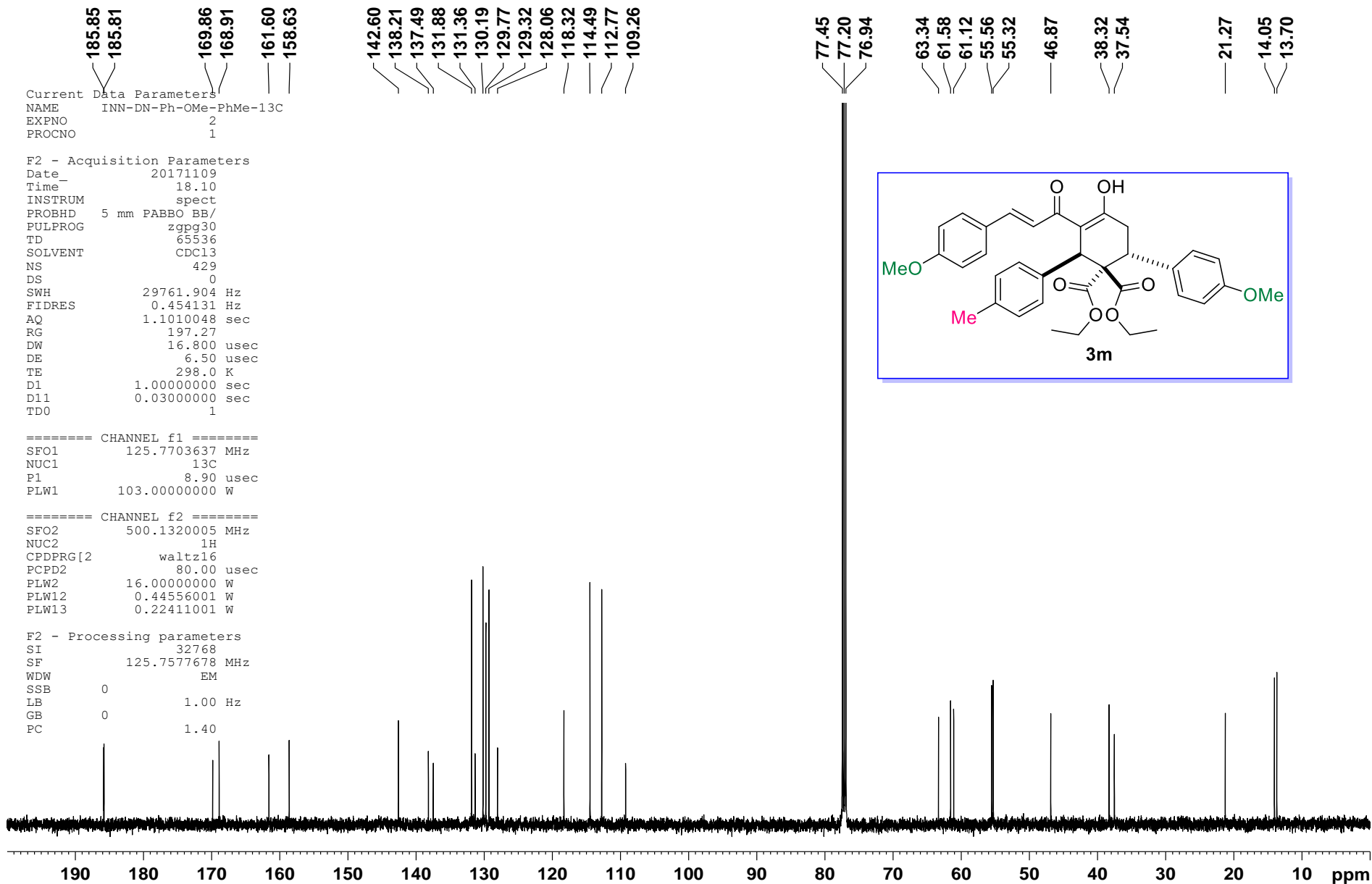


Fig S47. <sup>13</sup>C NMR Spectrum of 3m

7.349  
7.327  
7.258  
7.200  
7.181  
7.159  
7.115  
7.096  
6.940  
6.918  
6.748  
6.726  
5.362  
5.355  
5.327  
5.320  
4.802  
4.258  
4.248  
4.240  
4.231  
4.222  
4.213  
4.022  
4.004  
3.995  
3.986  
3.977  
3.948  
3.930  
3.905  
3.819  
3.793  
3.784  
3.756  
3.676  
3.658  
3.640  
3.632  
3.623  
3.614  
2.958  
2.922  
2.917  
2.879  
2.864  
2.853  
2.847  
2.598  
2.591  
2.556  
2.548  
2.315  
1.153  
1.135  
1.117  
1.009  
0.991  
0.973

Current Data Parameters  
NAME INN-DN-PH-OME-PH-ME-B-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20171110  
Time\_ 11.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 6  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 297.4 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300110 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

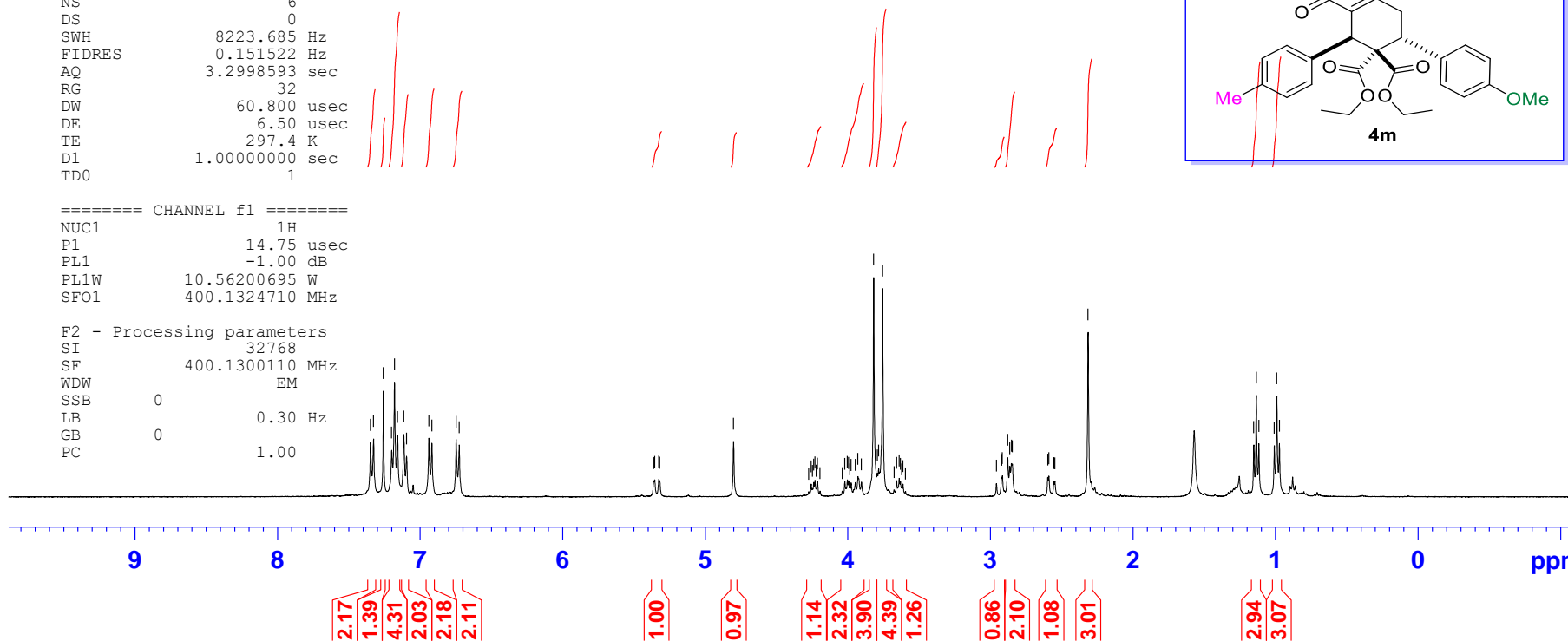


Fig S48. <sup>1</sup>H NMR Spectrum of 4m

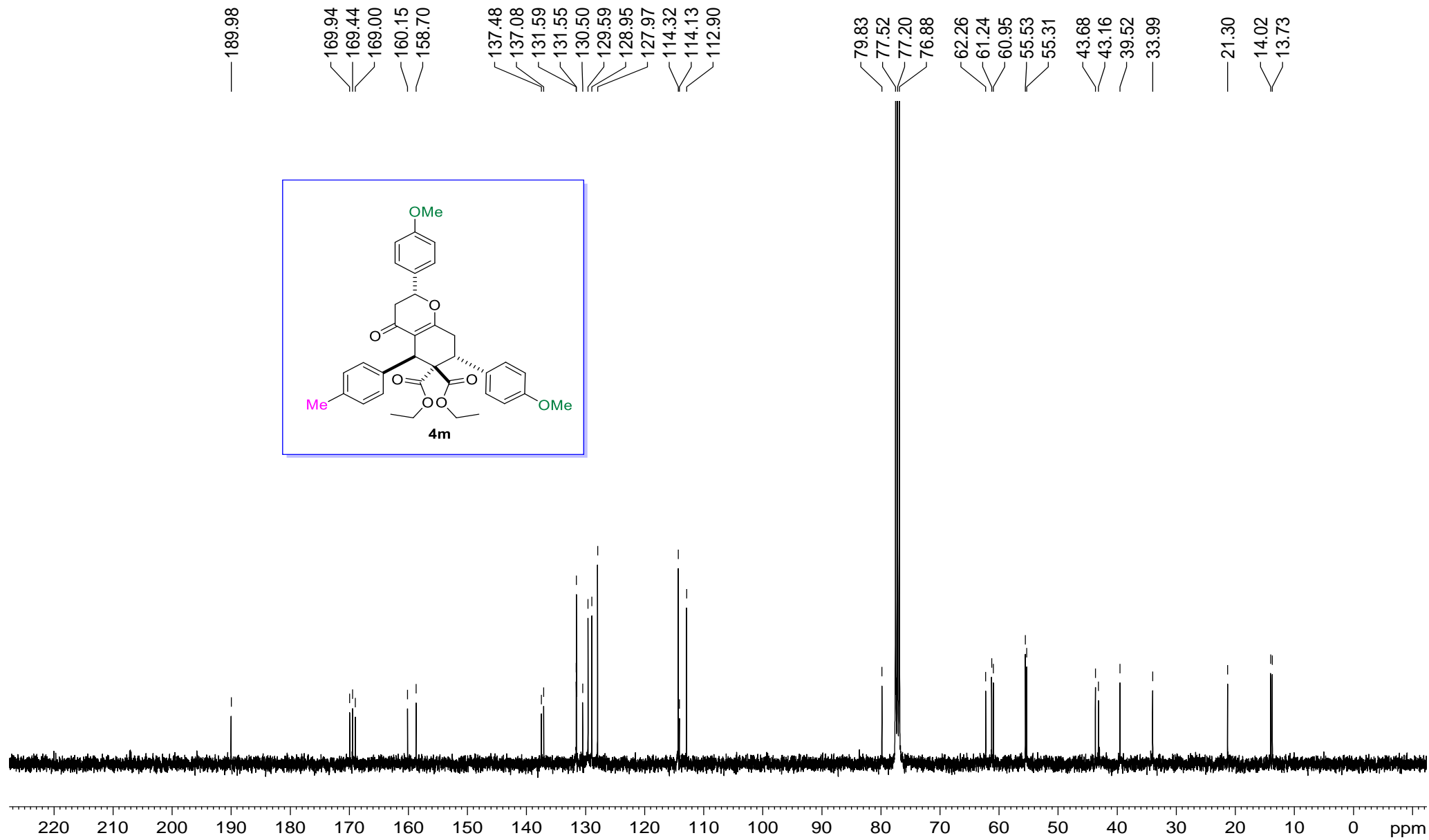


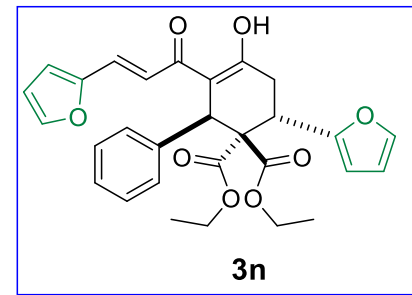
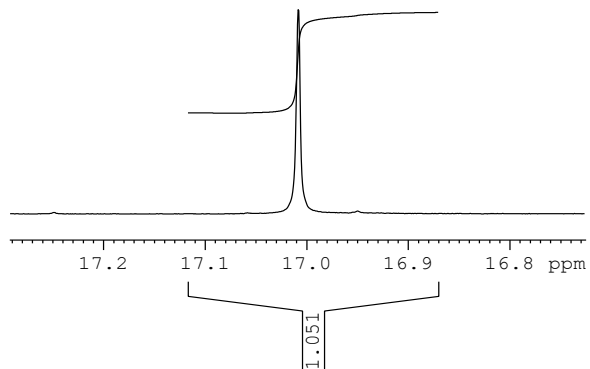
Fig S49. <sup>13</sup>C NMR Spectrum of 4m

```

Current Data Parameters
NAME      INN-DN-FUR-PH-1H
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20150707
Time      19.13
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         54274
SOLVENT   CDC13
NS         7
DS         0
SWH       8223.685 Hz
FIDRES    0.151522 Hz
AQ         3.2998593 sec
RG         161
DW         60.800 usec
DE         6.50 usec
TE         297.4 K
D1         1.00000000 sec
TDO        1

```



```

===== CHANNEL f1 =====
NUC1      1H
P1         14.75 usec
PL1        -1.00 dB
PL1W       10.56200695 W
SFO1       400.1340013 MHz

```

```

F2 - Processing parameters
SI         32768
SF         400.1300097 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```

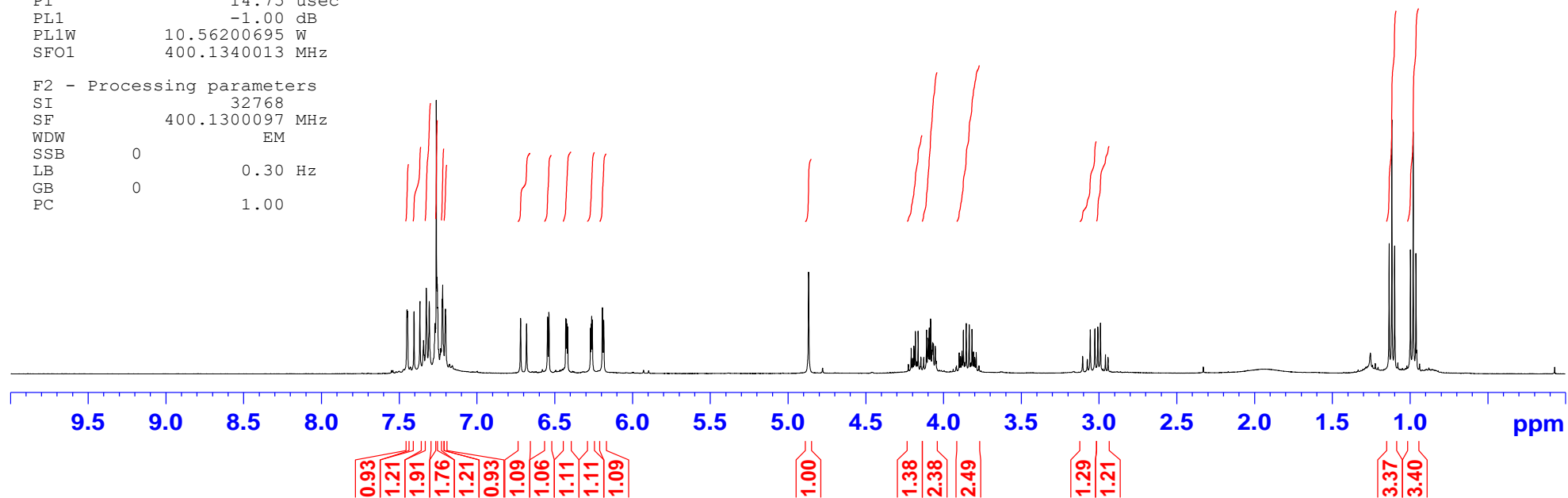


Fig S50. <sup>1</sup>H NMR Spectrum of 3n

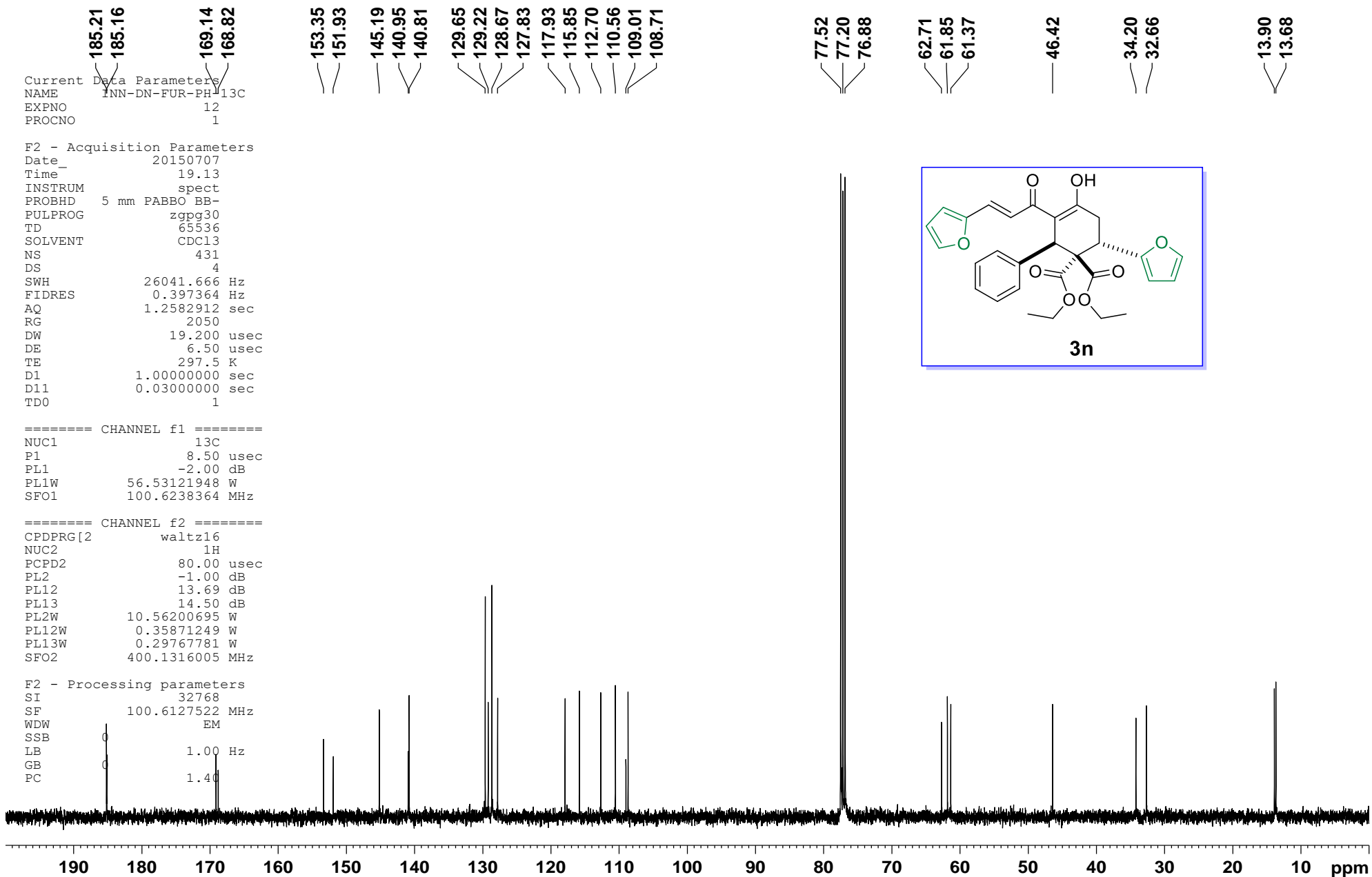


Fig S51. <sup>13</sup>C NMR Spectrum of 3n