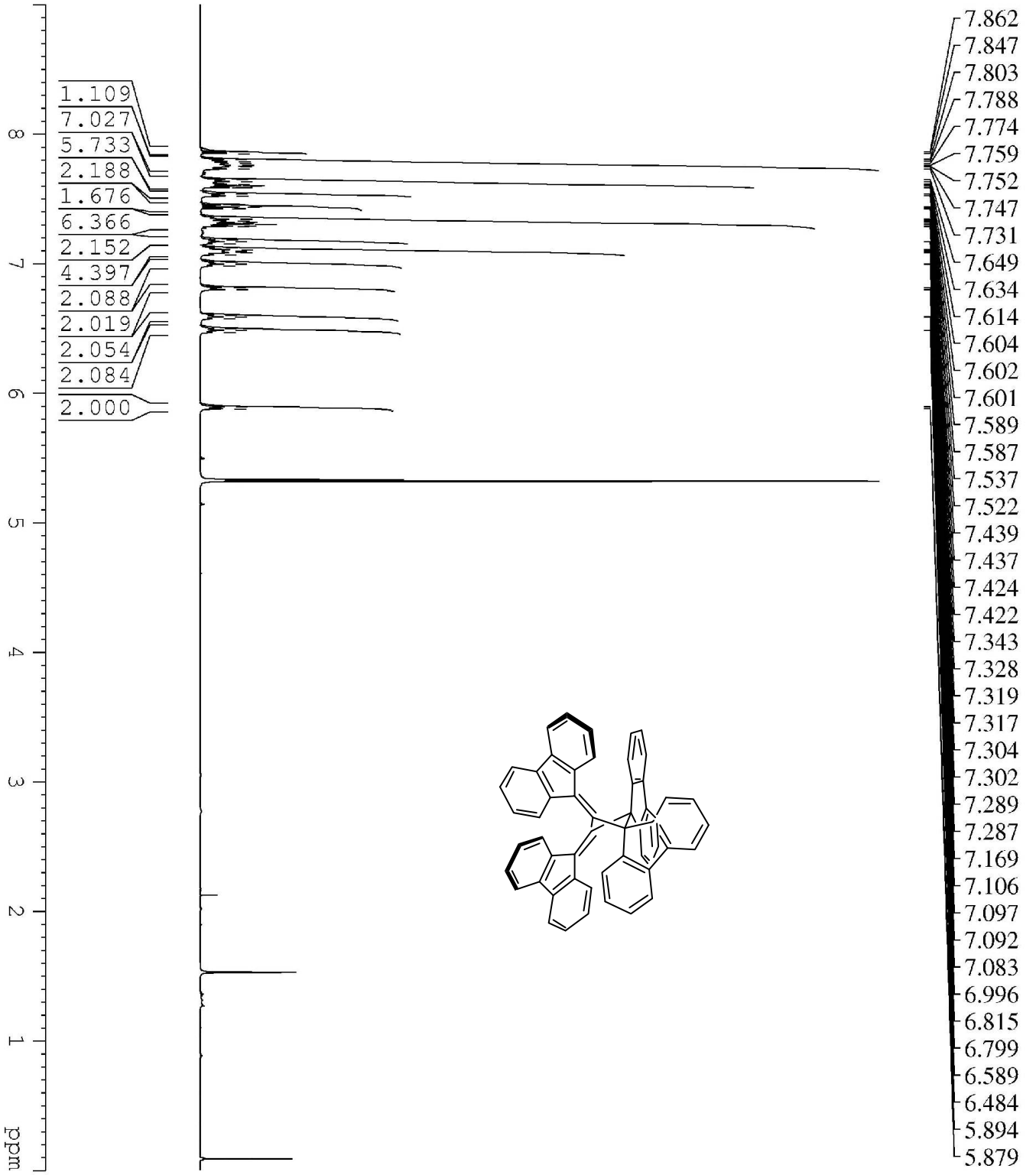
Electronic Supplementary Material (ESI) for Dalton Transactions

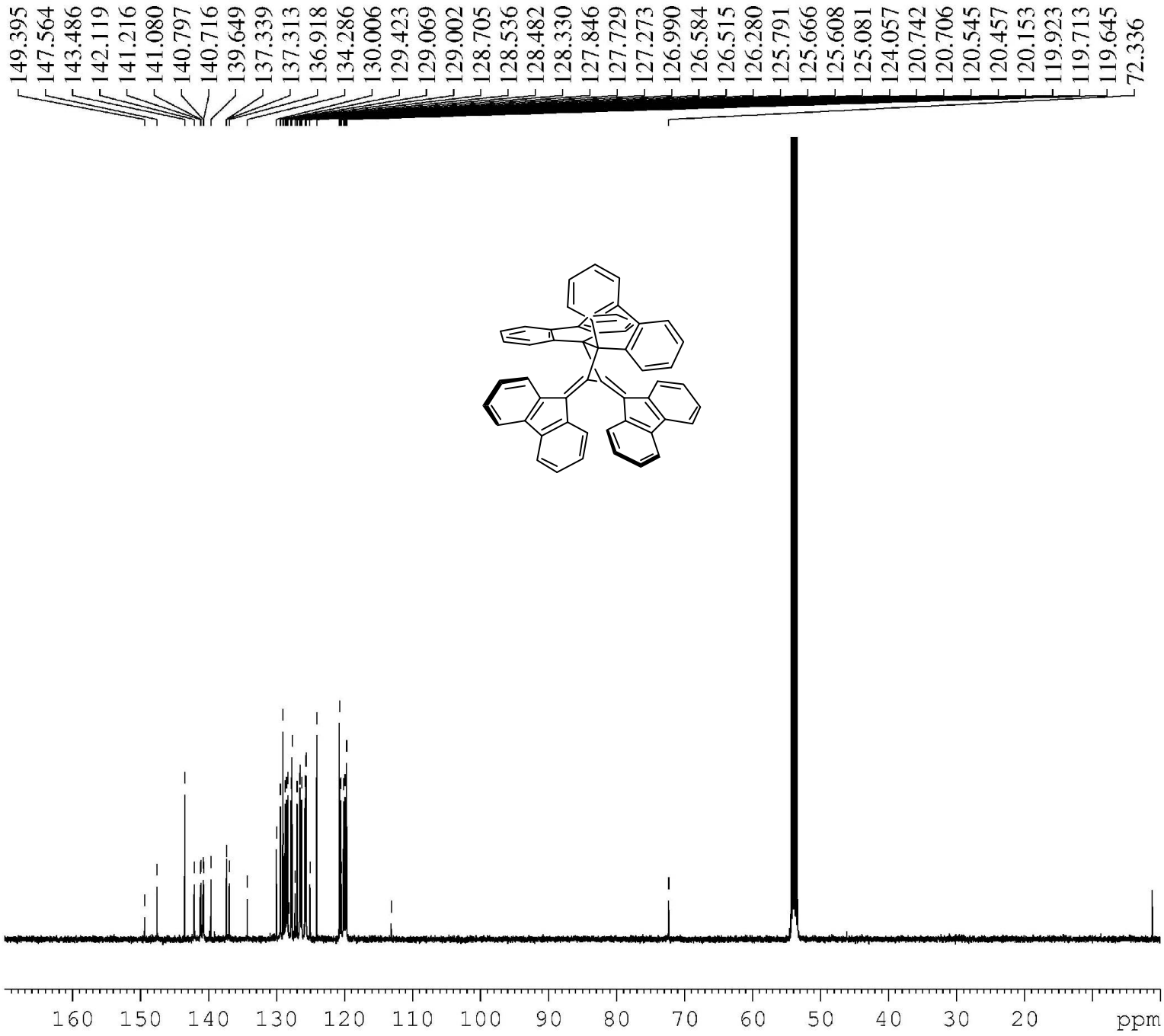
Synthesis and reactivity of electron poor allenes: formation of completely organic frustrated Lewis pairs

David Palomas, Sigrid Holle, Blanca Inés, Hans Bruns, Richard Goddard and Manuel Alcarazo

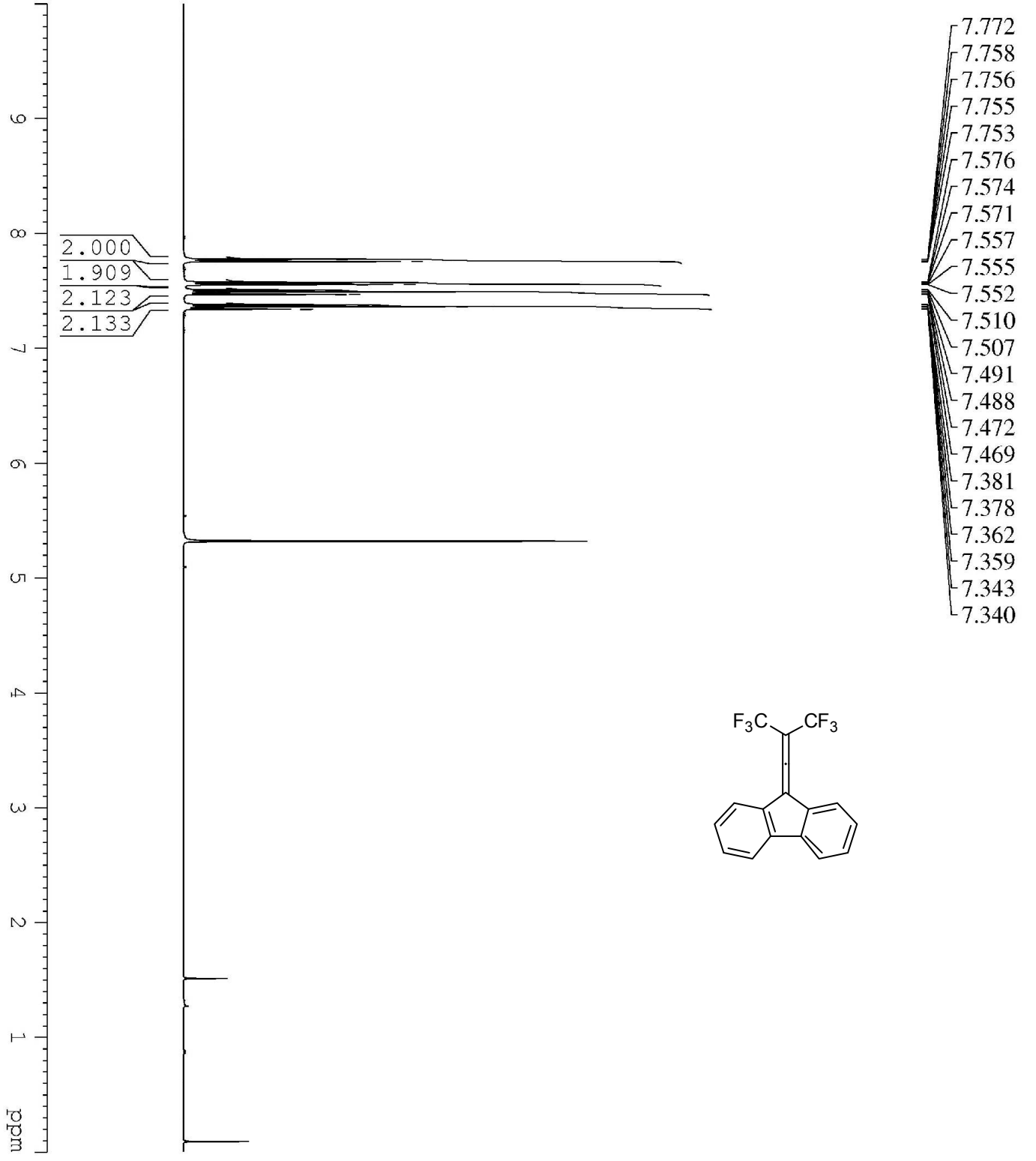
SUPPLEMENTARY INFORMATION



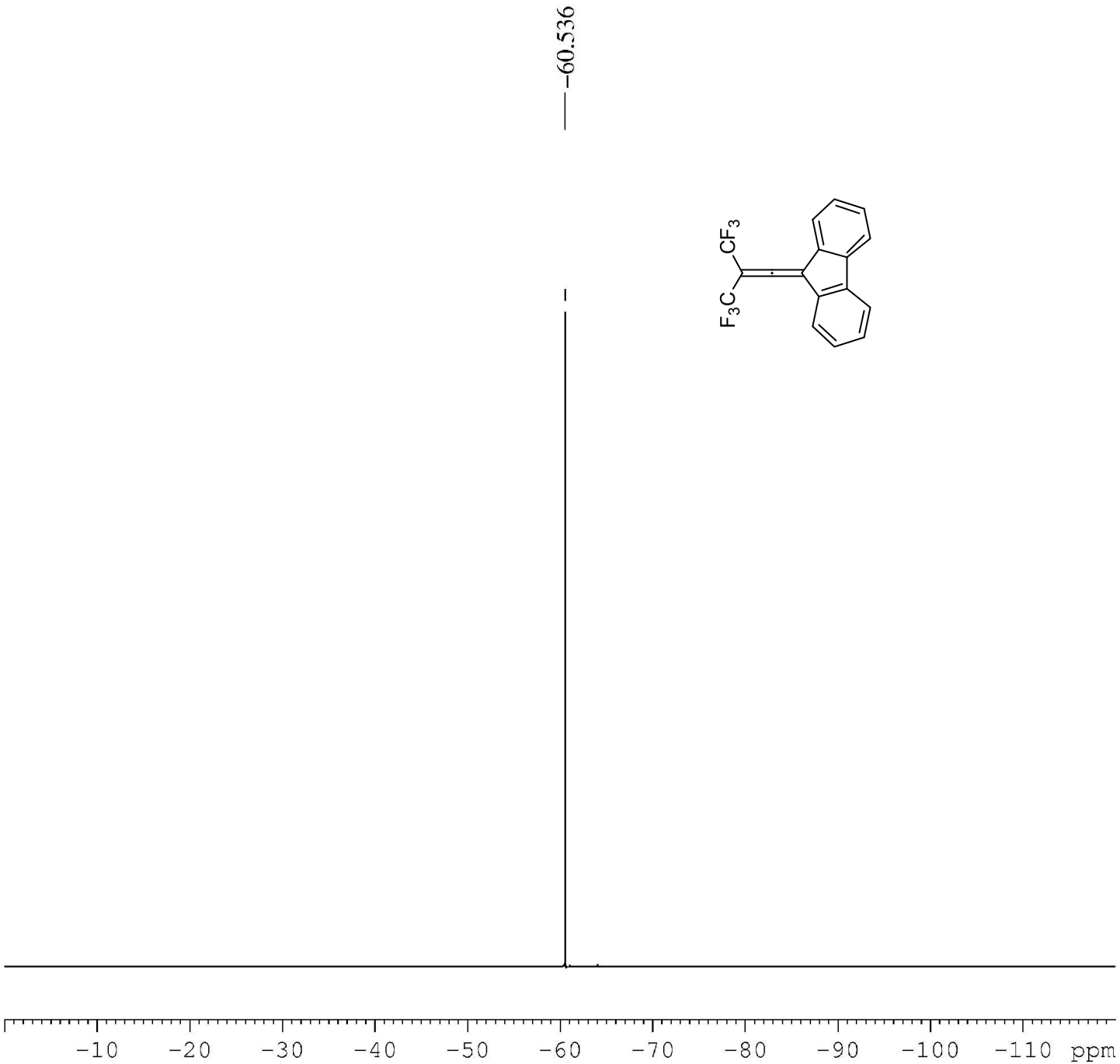

```
NAME                                                compound 2
EXPNO                                                       10
PROCNO                                                       1
Date_                                             20120116
Time                                                   14.21
INSTRUM                                                 spect
PROBHD                                  5 mm PABBO BB-
PULPROG                                                 zg30
TD                                                     32762
SOLVENT                                             CD2Cl2
NS                                                         32
DS                                                          2
SWH                                              8012.820 Hz
FIDRES                                           0.244577 Hz
AQ                                               2.0443988 sec
RG                                                        256
DW                                               62.400 usec
DE                                               10.00 usec
TE                                               296.0 K
D1                                               2.00000000 sec

======== CHANNEL f1 ========
NUC1                                                      1H
P1                                               10.00 usec
SI                                                     32768
SF                                          499.8700245 MHz
WDW                                                    EM
SSB                                                     0
LB                                               0.30 Hz
GB                                                      0
PC                                               2.00
```



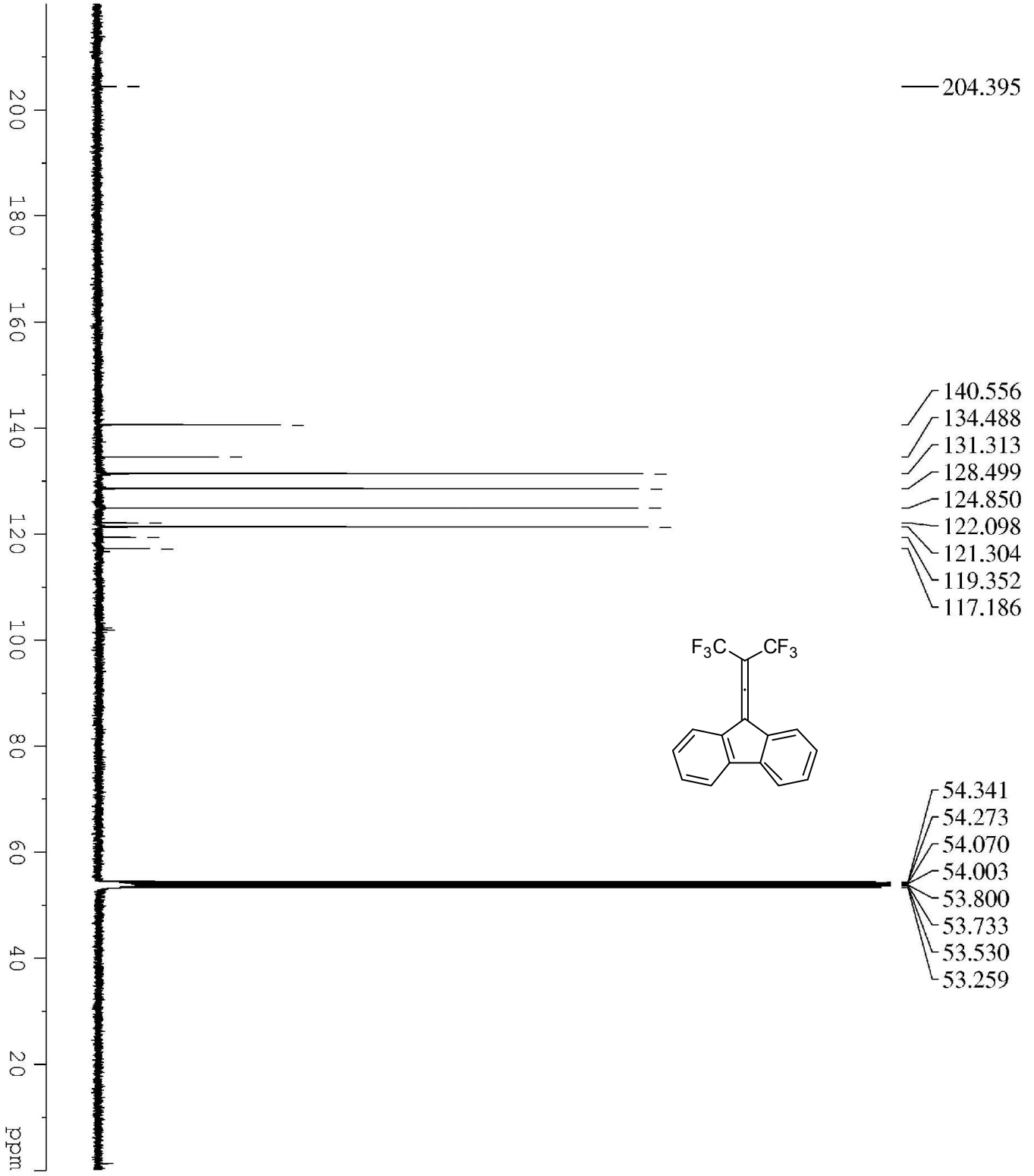
NAME	compound 2
EXPNO	12
PROCNO	1
Date_	20120117
Time	8.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgdc30
TD	65536
SOLVENT	CD2C12
NS	70000
DS	128
SWH	39062.500 Hz
FIDRES	0.596046 Hz
AQ	0.8389108 sec
RG	2050
DW	12.800 usec
DE	10.00 usec
TE	296.0 K
D1	0.01000000 sec
D11	0.03000000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
SI	65536
SF	125.6923648 MHz
WDW	EM
SSB	0
LB	0.80 Hz
GB	0
PC	1.40



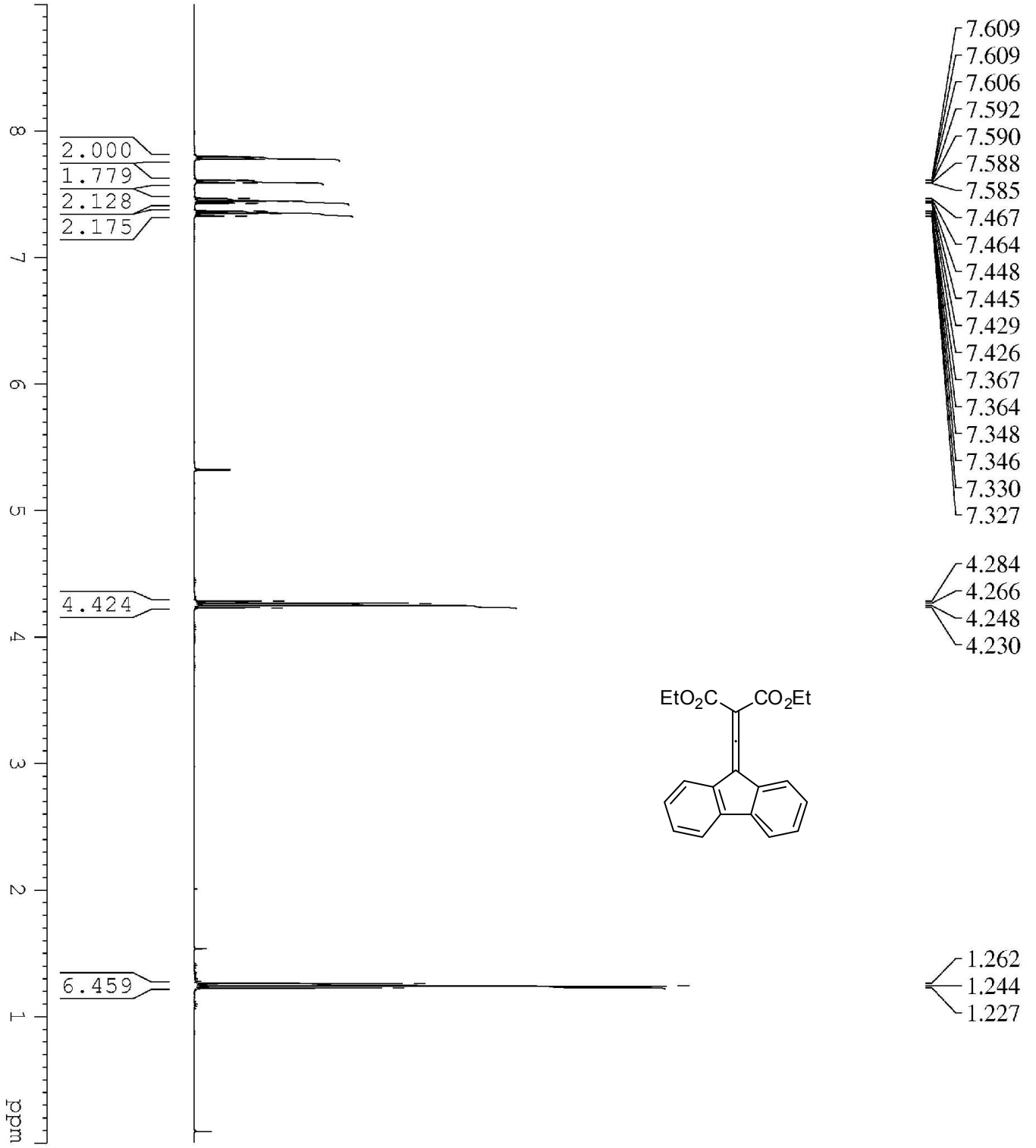

```
NAME                                  compound 5
EXPNO                                        10
PROCNO                                        1
Date_                               20100708
Time                                     8.47
INSTRUM                                  spect
PROBHD                 5 mm PABBO BB-
PULPROG                                   zg30
TD                                       32768
SOLVENT                                CD2C12
NS                                          64
DS                                           2
SWH                                 6578.947 Hz
FIDRES                              0.200774 Hz
AQ                                  2.4904180 sec
RG                                         456.1
DW                                  76.000 usec
DE                                  10.50 usec
TE                                  302.0 K
D1                                  0.01000000 sec
TD0                                          1

======== CHANNEL f1 ========
NUC1                                       1H
P1                                   9.50 usec
PL1                                  -3.00 dB
SFO1                                400.1324710 MHz
SI                                       32768
SF                                  400.1300151 MHz
WDW                                        EM
SSB                                         0
LB                                   0.30 Hz
GB                                          0
PC                                   1.00
```



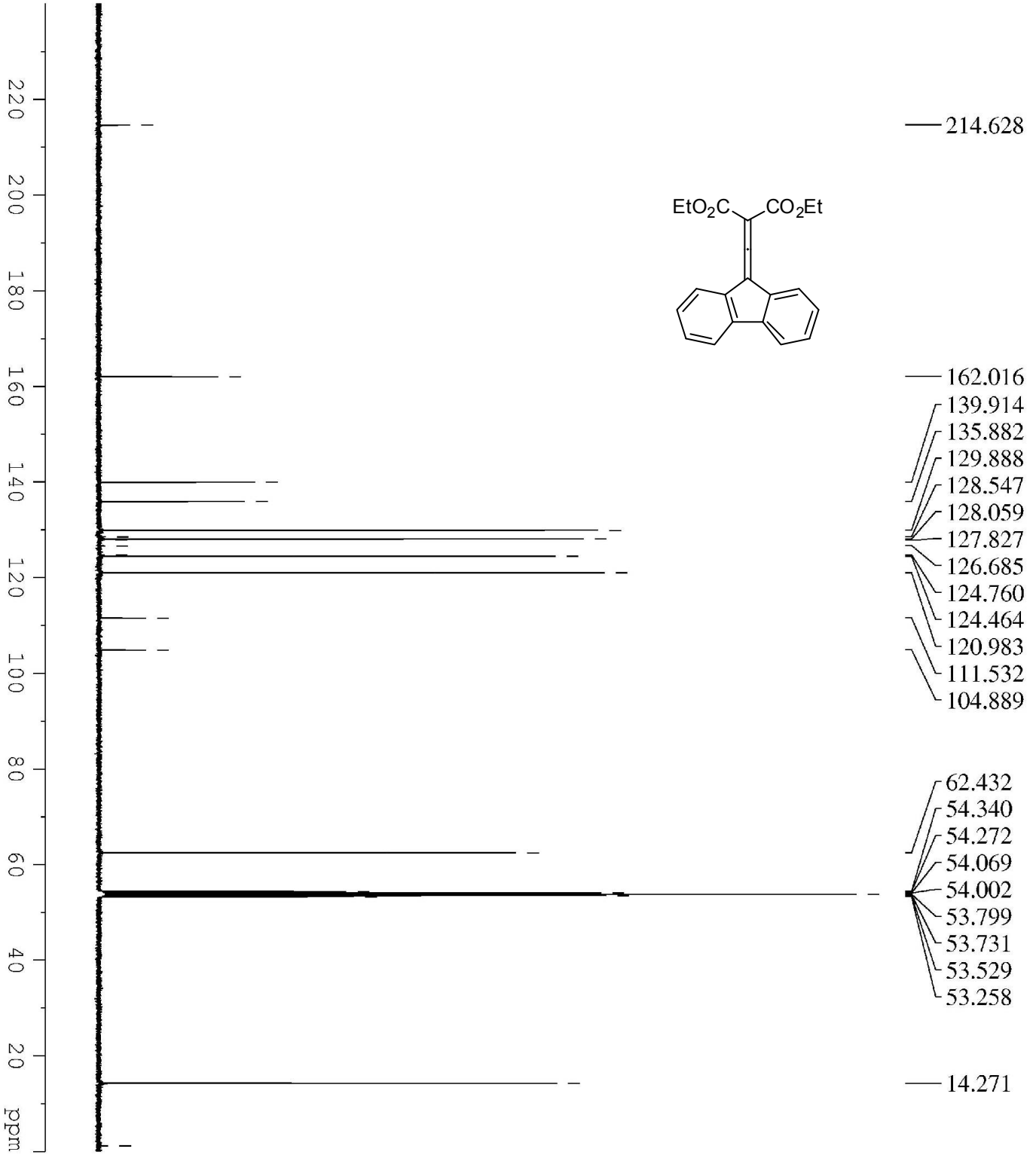
NAME	compound 5
EXPNO	11
PROCNO	1
Date_	20100708
Time	8.55
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgfhig30qn.ph
TD	131072
SOLVENT	CD2Cl2
NS	256
DS	64
SWH	75187.969 Hz
FIDRES	0.573639 Hz
AQ	0.8716788 sec
RG	2298.8
DW	6.650 usec
DE	10.00 usec
TE	302.0 K
D1	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	19F
P1	9.50 usec
PL1	-5.00 dB
SFO1	376.4682341 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	72.00 usec
PL2	-3.00 dB
PL12	16.00 dB
SFO2	400.1324704 MHz
SI	65536
SF	376.4983540 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00



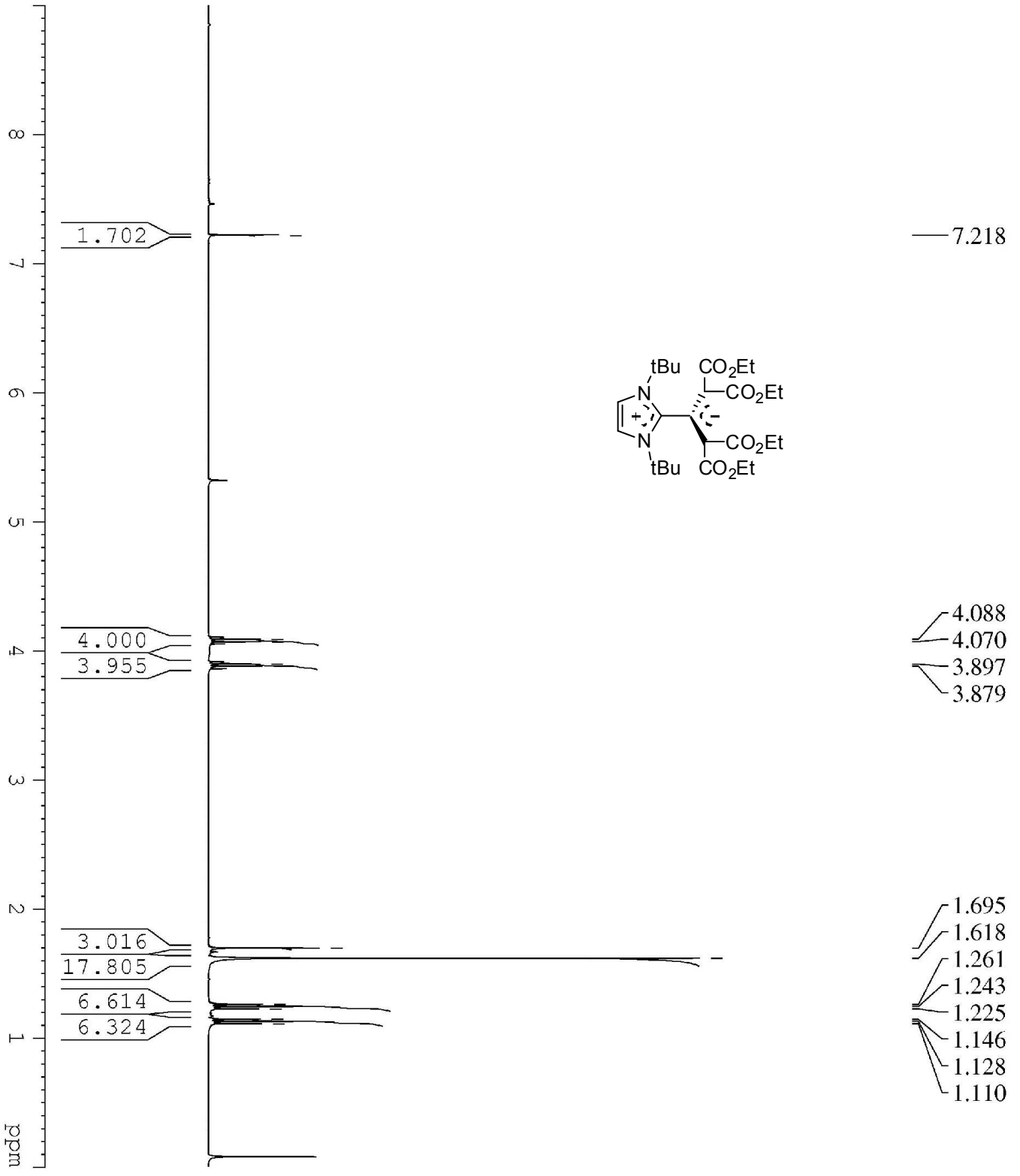

```
NAME                                                compound 5
EXPNO                                                       17
PROCNO                                                       1
Date_                                             20100709
Time                                                 13.06
INSTRUM                                               spect
PROBHD                                 5 mm PABBO BB-
PULPROG                                              zgdc
TD                                                  65536
SOLVENT                                           CD2Cl2
NS                                                  32000
DS                                                    128
SWH                                          31250.000 Hz
FIDRES                                        0.476837 Hz
AQ                                            1.0486259 sec
RG                                              23170.5
DW                                            16.000 usec
DE                                            10.50 usec
TE                                               302.0 K
D1                                            0.01000000 sec
D11                                           0.03000000 sec
TD0                                                     1

======== CHANNEL f1 ========
NUC1                                                  13C
P1                                             1.35 usec
PL1                                              0.00 dB
SFO1                                  100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                                          waltz16
NUC2                                                  1H
PCPD2                                          72.00 usec
PL2                                             -3.00 dB
PL12                                            16.00 dB
SFO2                                  400.1324710 MHz
SI                                                  65536
SF                                    100.6127259 MHz
WDW                                                    EM
SSB                                                     0
LB                                            0.80 Hz
GB                                                      0
PC                                                   1.00
```



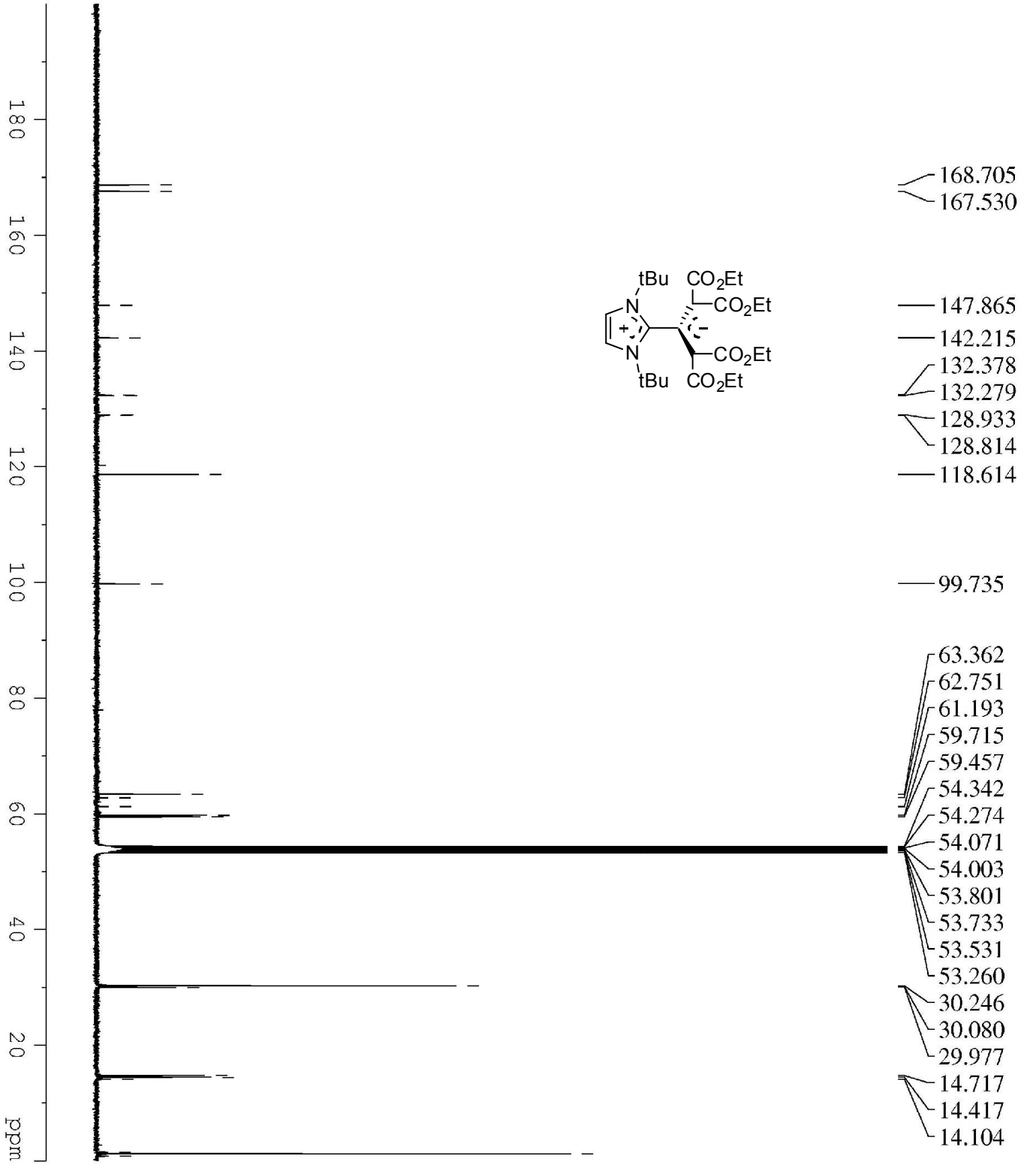

```
NAME                                  compound 6
EXPNO                                        10
PROCNO                                        1
Date_                               20100512
Time                                    12.54
INSTRUM                                 spect
PROBHD                 5 mm PABBO BB-
PULPROG                                  zg30
TD                                      32768
SOLVENT                              CD2Cl2
NS                                          64
DS                                           2
SWH                                 6578.947 Hz
FIDRES                                0.200774 Hz
AQ                                  2.4904180 sec
RG                                        161.3
DW                                     76.000 usec
DE                                     10.50 usec
TE                                     302.0 K
D1                                  0.01000000 sec
TD0                                          1

======== CHANNEL f1 ========
NUC1                                       1H
P1                                      9.50 usec
PL1                                     -3.00 dB
SFO1                              400.1324710 MHz
SI                                       32768
SF                                400.1300148 MHz
WDW                                         no
SSB                                          0
LB                                        0.00 Hz
GB                                           0
PC                                        1.00
```



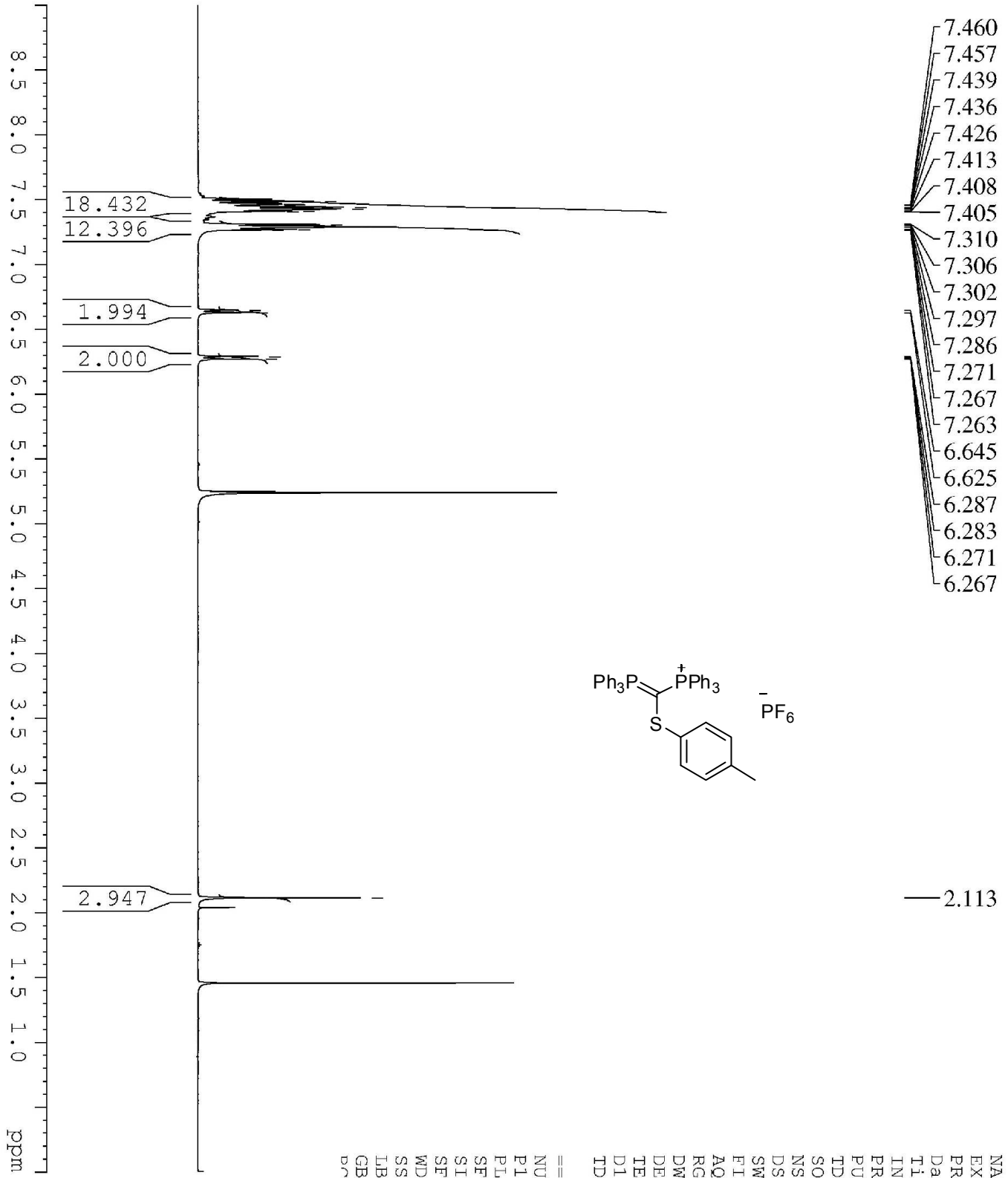

```
NAME                                  compound 6
EXPNO                                        17
PROCNO                                        1
Date_                                  20100515
Time                                       4.03
INSTRUM                                   spect
PROBHD                 5 mm PABBO BB-
PULPROG                                    zgdc
TD                                        65536
SOLVENT                                  CD2Cl2
NS                                         9000
DS                                          128
SWH                                  31250.000 Hz
FIDRES                                0.476837 Hz
AQ                                    1.0486259 sec
RG                                       14596.5
DW                                     16.000 usec
DE                                     10.50 usec
TE                                       302.0 K
D1                                    0.01000000 sec
D11                                   0.03000000 sec
TD0                                             1

======== CHANNEL f1 ========
NUC1                                         13C
P1                                      1.95 usec
PL1                                     0.00 dB
SFO1                          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                                  waltz16
NUC2                                          1H
PCPD2                                   72.00 usec
PL2                                     -3.00 dB
PL12                                    16.00 dB
SFO2                          400.1324710 MHz
SI                                        65536
SF                            100.6127275 MHz
WDW                                          EM
SSB                                           0
LB                                      0.80 Hz
GB                                            0
PC                                         1.00
```



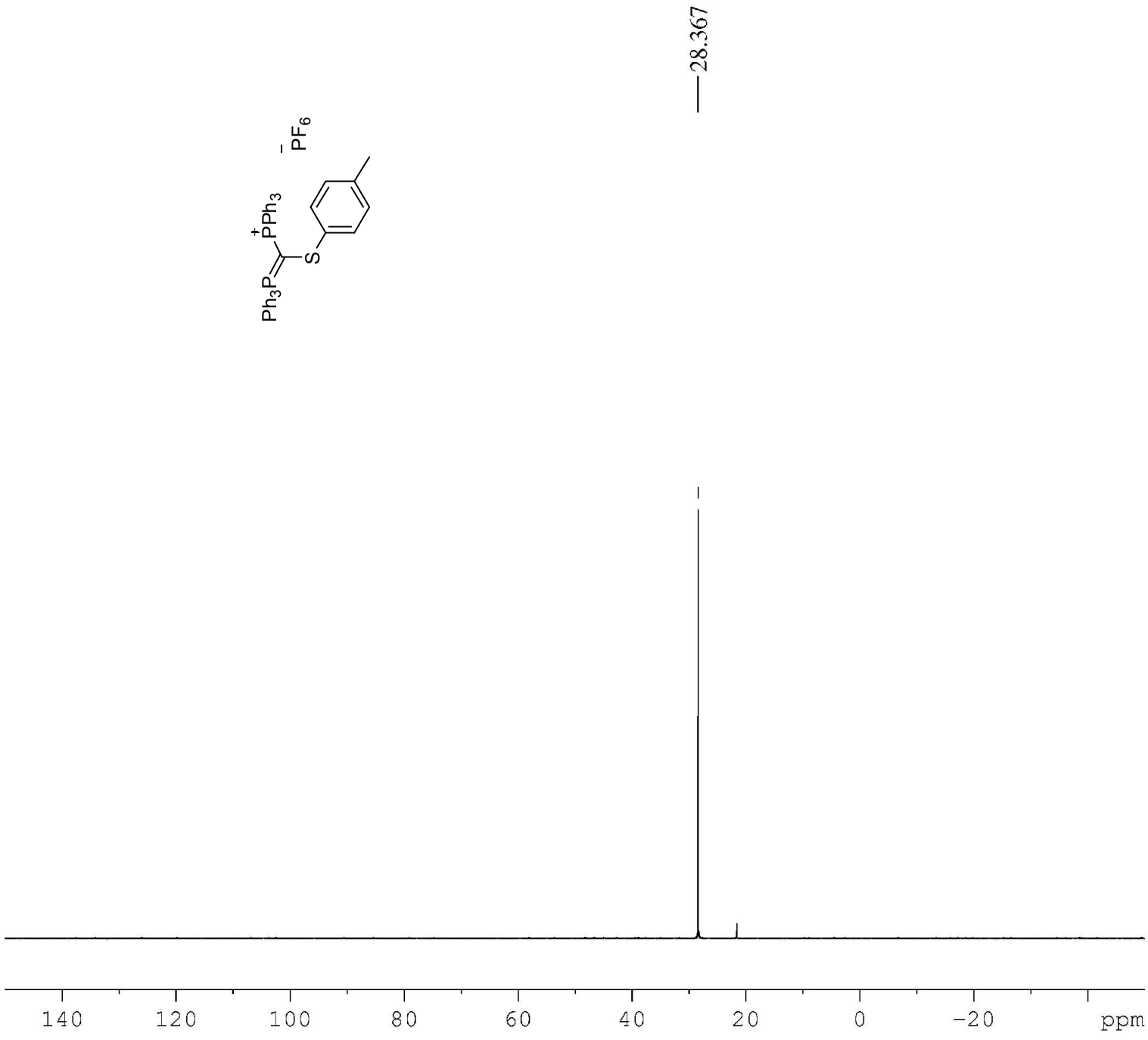

```
NAME                 compound 18_2
EXPNO                          10
PROCNO                          1
Date_                    20100602
Time                        15.23
INSTRUM                      av400
PROBHD     5 mm BBO BB-1H
PULPROG                      zg30
TD                          65536
SOLVENT                  CD2Cl2
NS                              32
DS                               8
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                           161.3
DW                          60.400 use
DE                           6.50 use
TE                          299.8 K
D1                     0.00300000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          9.25 use
PL1                          0.00 dB
SFO1                 400.1324710 MHz
SI                           32768
SF                   400.1300150 MHz
WDW                             EM
SSB                              0
LB                            0.30 Hz
GB                               0
PC                            2.00
```



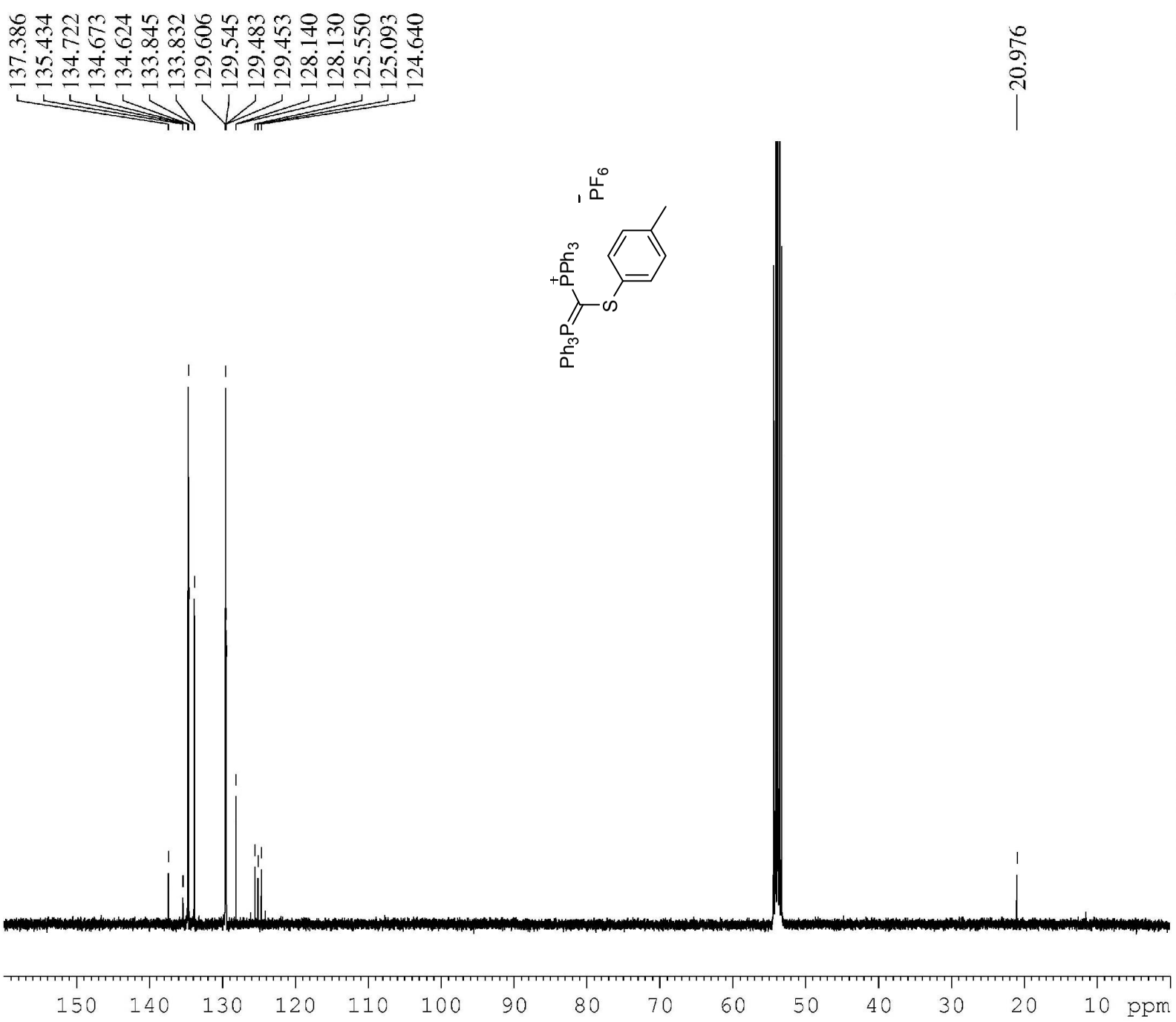

```
NAME                                                compound 18
EXPNO                                                       17
PROCNO                                                       1
Date_                                             20100503
Time                                                 7.18
INSTRUM                                                  spect
PROBHD                                 5 mm PABBO BB-
PULPROG                                                  zgdc
TD                                                      65536
SOLVENT                                                 CD2Cl2
NS                                                      69388
DS                                                        128
SWH                                              31250.000 Hz
FIDRES                                               0.476837 Hz
AQ                                               1.0486259 sec
RG                                                    14596.5
DW                                                   16.000 usec
DE                                                   10.50 usec
TE                                                   302.0 K
D1                                               0.01000000 sec
D11                                              0.03000000 sec
TD0                                                         1

======== CHANNEL f1 ========
NUC1                                                      13C
P1                                                   1.60 usec
PL1                                                  0.00 dB
SFO1                                          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                                              waltz16
NUC2                                                      1H
PCPD2                                                72.00 usec
PL2                                                  -3.00 dB
PL12                                                 16.00 dB
SFO2                                          400.1324710 MHz
SI                                                      65536
SF                                            100.6127264 MHz
WDW                                                      EM
SSB                                                       0
LB                                                   0.80 Hz
GB                                                        0
PC                                                   1.00
```



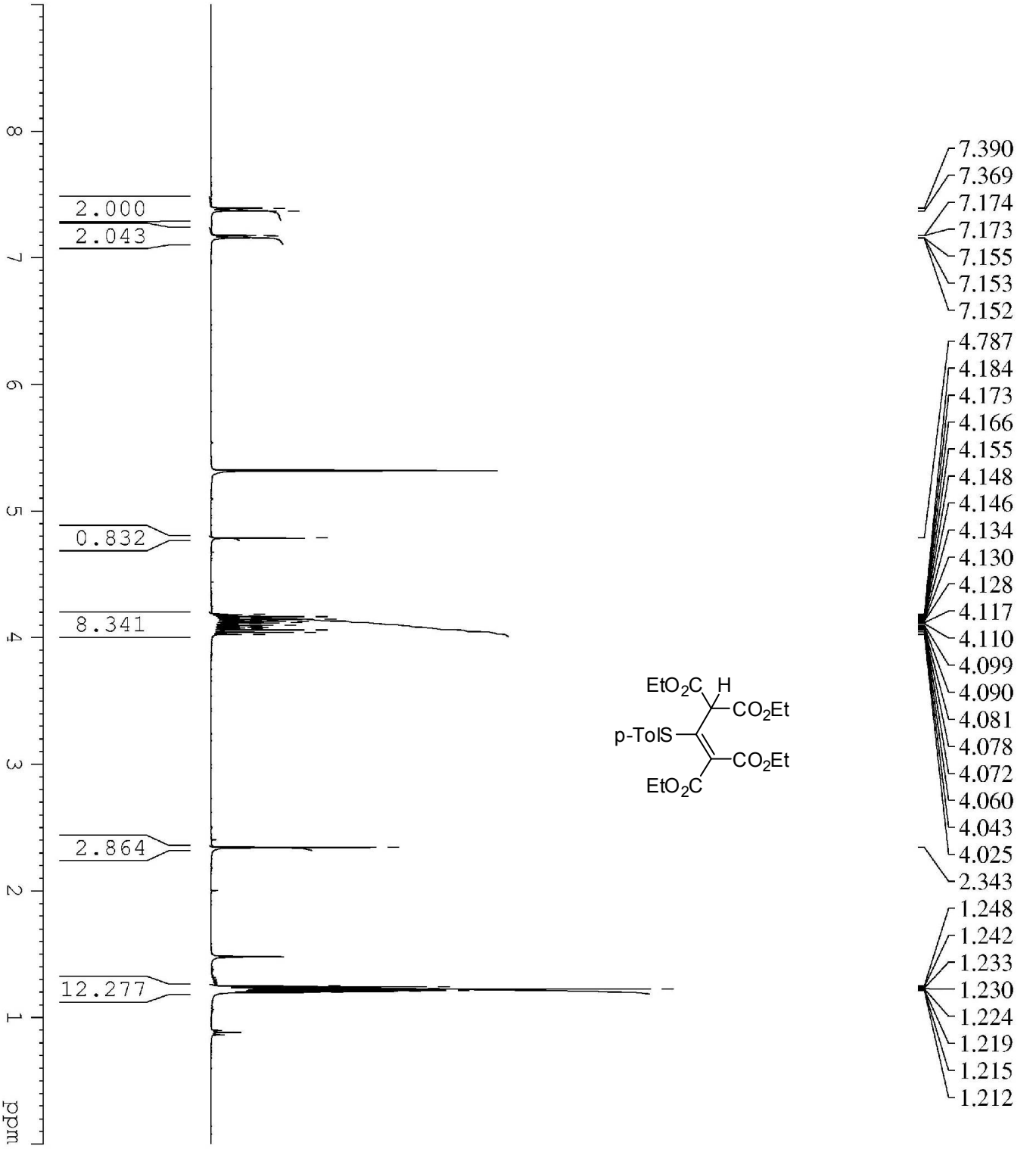

```
NAME                 compound 28
EXPNO                        10
PROCNO                        1
Date_               20100611
Time                     4.08
INSTRUM                av400
PROBHD     5 mm BBO BB-1H
PULPROG                   zg30
TD                       65536
SOLVENT              CD2Cl2
NS                           32
DS                            8
SWH                 8278.146 Hz
FIDRES                 0.126314 Hz
AQ                  3.9584243 sec
RG                          256
DW                       60.400 usec
DE                        6.50 usec
TE                      299.0 K
D1                    0.00300000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        9.25 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz
SI                       32768
SF                  400.1300484 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        2.00
```



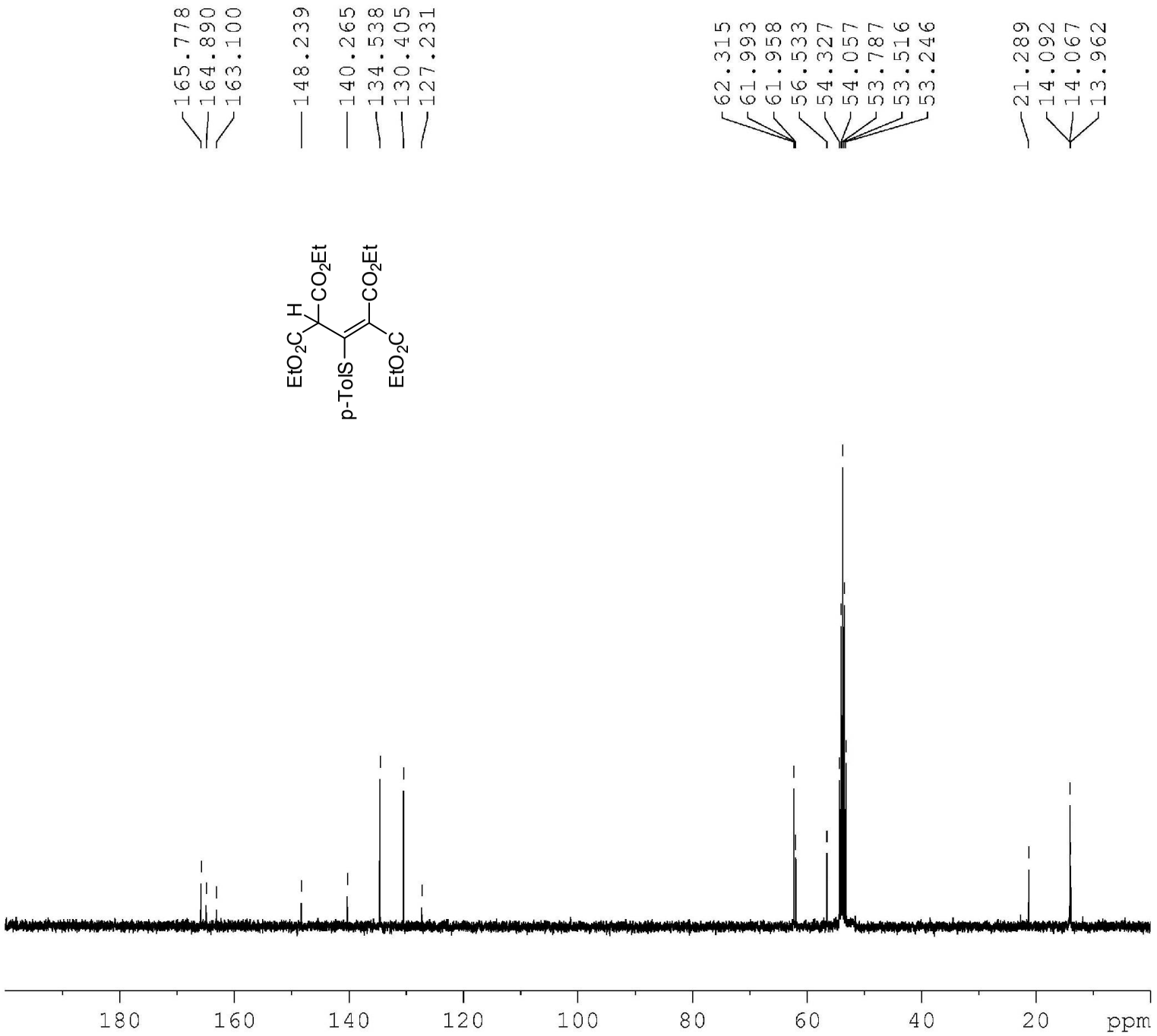
NAME	compound 28	
EXPNO	11	
PROCNO	1	
Date_	20100611	
Time	4.21	
INSTRUM	av400	
PROBHD	5 mm BBO BB-1H	
PULPROG	zgdc30	
TD	65536	
SOLVENT	CD2C12	
NS	1000	
DS	4	
SWH	58139.535	Hz
FIDRES	0.887139	Hz
AQ	0.5636596	sec
RG	18390.4	
DW	8.600	usec
DE	6.50	usec
TE	299.4	K
D1	0.03000000	sec
d11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	31P	
P1	7.04	usec
PL1	5.00	dB
SFO1	161.9883609	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	0.00	dB
PL12	19.76	dB
SFO2	400.1324710	MHz
SI	32768	
SF	161.9754439	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	



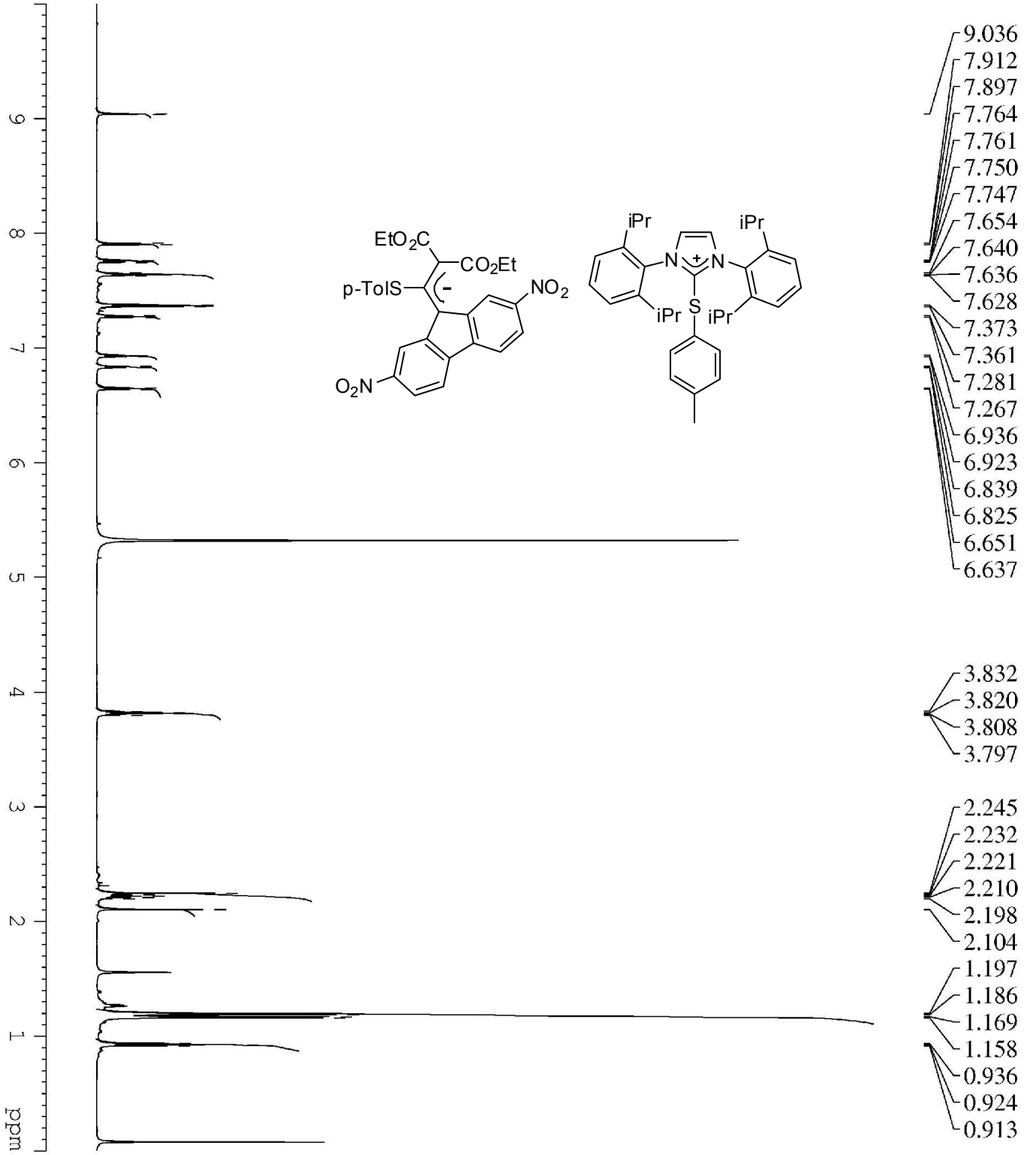
NAME	compound 28_2
EXPNO	10
PROCNO	1
Date_	20120119
Time	16.15
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zgdc30
TD	65536
SOLVENT	CD2C12
NS	45000
DS	12
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	20642.5
DW	16.000 use
DE	10.50 use
TE	300.0 K
D1	0.10000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	14.75 use
PL1	-2.00 dB
SFO1	100.6278609 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	72.00 use
PL2	-3.00 dB
PL12	16.00 dB
SFO2	400.1324710 MHz
SI	65536
SF	100.6127232 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40



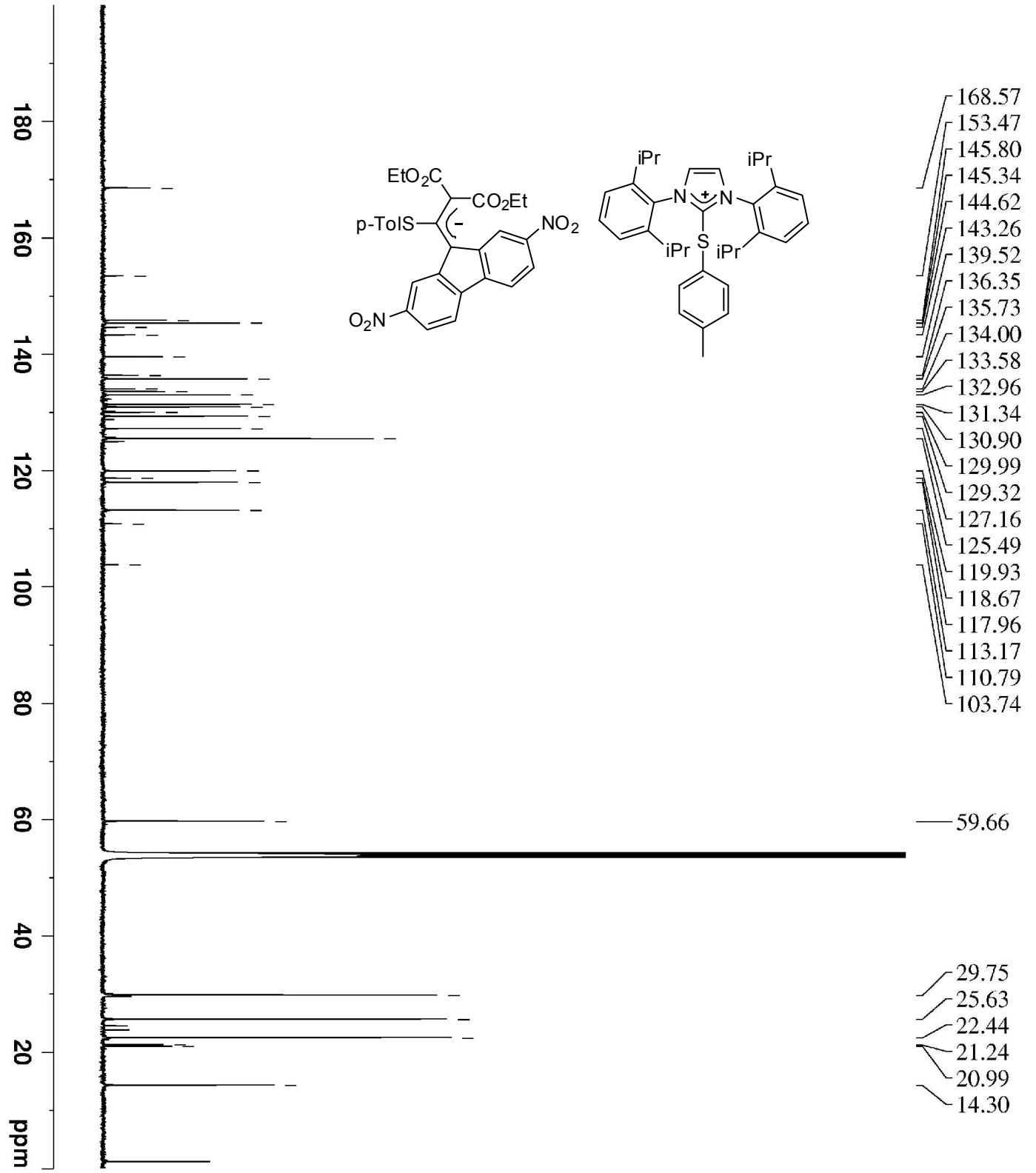

```
NAME                                  compound 29
EXPNO                                         10
PROCNO                                         1
Date_                                   20100817
Time                                       23.18
INSTRUM                                    av400
PROBHD                  5 mm BBO BB-1H
PULPROG                                    zg30
TD                                        65536
SOLVENT                                  CD2Cl2
NS                                            32
DS                                             8
SWH                                   8278.146 Hz
FIDRES                                0.126314 Hz
AQ                                    3.9584243 sec
RG                                        228.1
DW                                    60.400 usec
DE                                     6.50 usec
TE                                     298.6 K
D1                                    0.00300000 sec
TD0                                            1

======== CHANNEL f1 ========
NUC1                                         1H
P1                                     9.25 usec
PL1                                    0.00 dB
SFO1                          400.1324710 MHz
SI                                        32768
SF                            400.1300150 MHz
WDW                                           no
SSB                                            0
LB                                      0.00 Hz
GB                                             0
PC                                          8.00
```



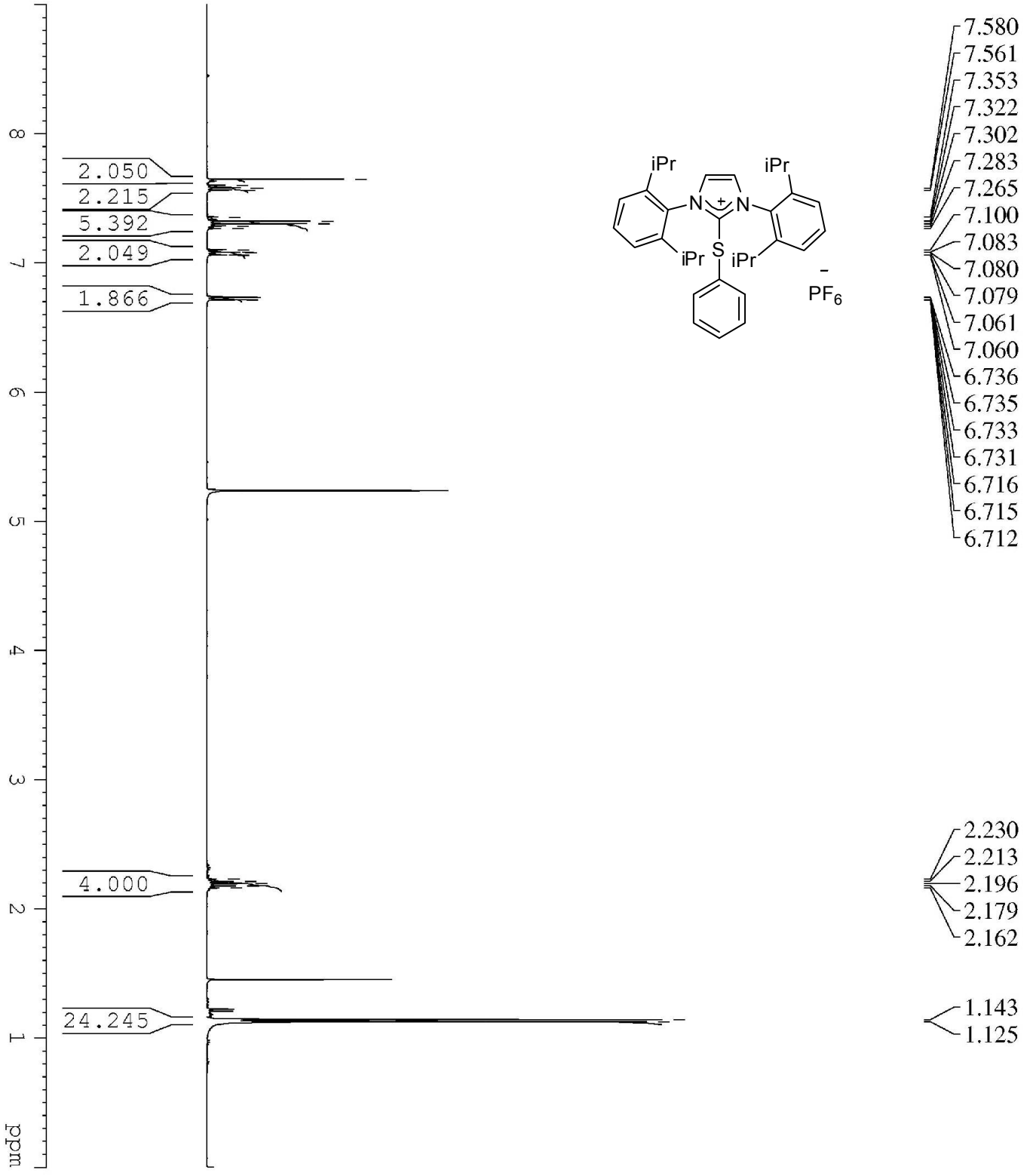
NAME	compound 29
EXPNO	11
PROCNO	1
Date_	20100817
Time	23.47
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc30
TD	65536
SOLVENT	CD2Cl2
NS	1500
DS	2
SWH	33112.582 Hz
FIDRES	0.505258 Hz
AQ	0.9896436 sec
RG	18390.4
DW	15.100 usec
DE	8.00 usec
TE	299.1 K
D1	0.03000000 sec
d11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.94 usec
PL1	5.00 dB
SFO1	100.6242789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	19.76 dB
SFO2	400.1324710 MHz
SI	32768
SF	100.6127306 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00



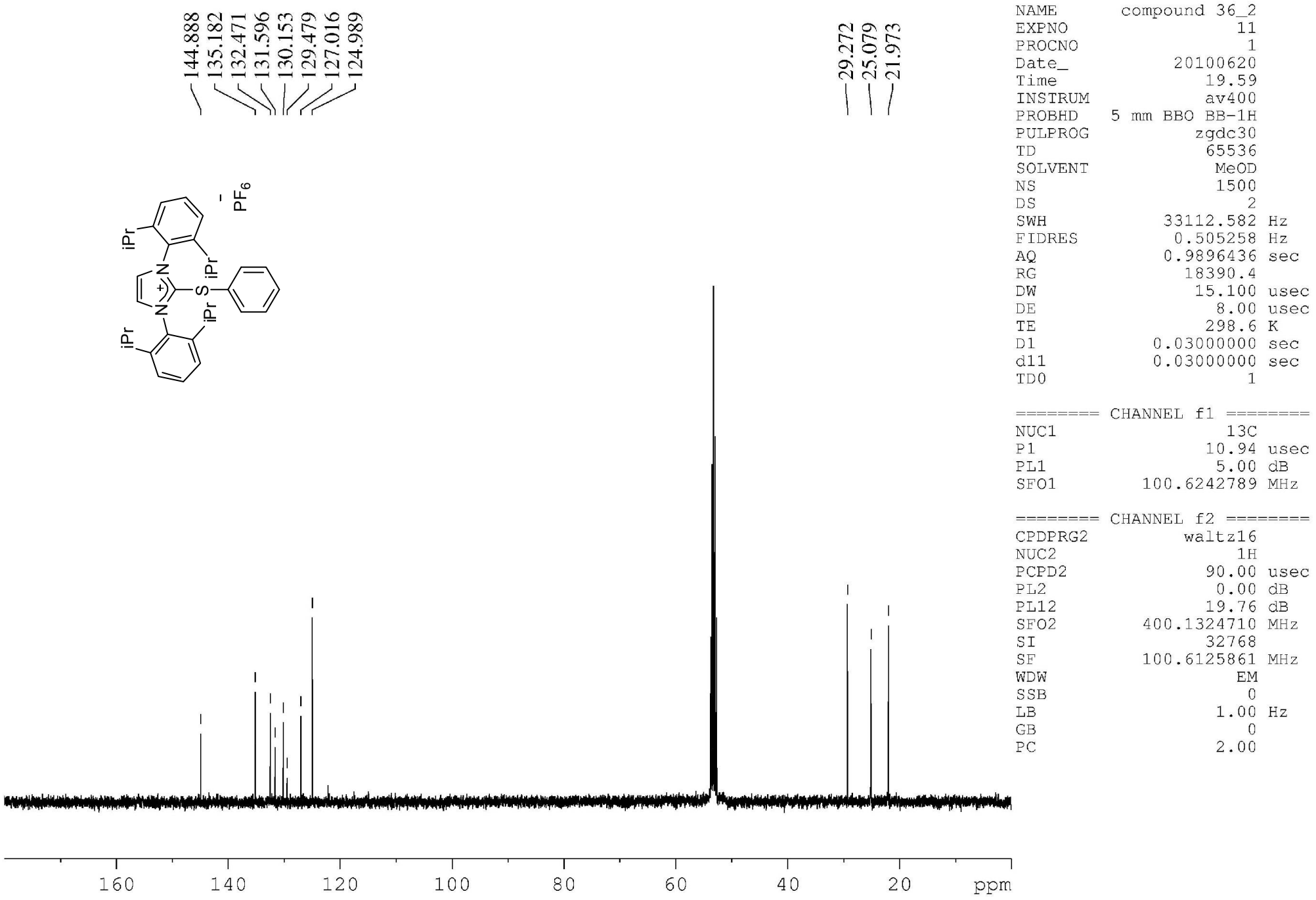

```
NAME                 compound 34
EXPNO                        10
PROCNO                        1
Date_                  20100819
Time                       14.22
INSTRUM                   av600
PROBHD     5 mm CPTCI 1H-
PULPROG                    zg30
TD                        65536
SOLVENT                CD2C12
NS                            32
DS                             2
SWH                  12019.230 Hz
FIDRES                0.183399 Hz
AQ                    2.7263477 sec
RG                         11.3
DW                       41.600 usec
DE                       10.00 usec
TE                       290.5 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                         8.50 usec
PL1                        4.20 dB
PL1W                5.30020905 W
SFO1                600.2234813 MHz
SI                        65536
SF                  600.2200215 MHz
WDW                          no
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.00
```



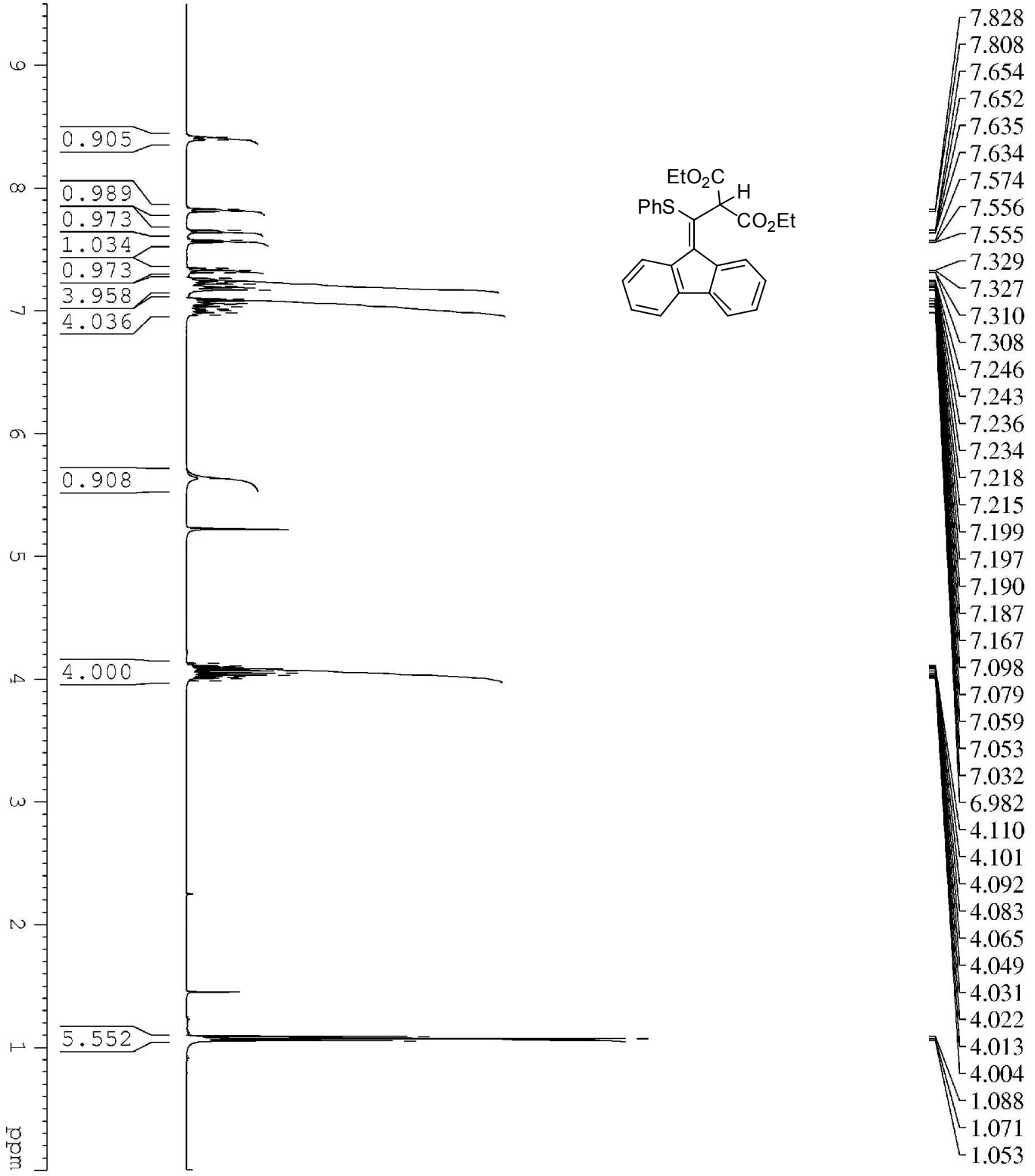

```
NAME                                                compound 34
EXPNO                                                       11
PROCNO                                                       1
Date_                                             20100819
Time                                                  16.43
INSTRUM                                                av600
PROBHD                                  5 mm CPTCI 1H-
PULPROG                                               zgdc30
TD                                                     90904
SOLVENT                                             CD2Cl2
NS                                                     7277
DS                                                      128
SWH                                            45454.547 Hz
FIDRES                                             0.500028 Hz
AQ                                             0.9999940 sec
RG                                                      512
DW                                                 11.000 usec
DE                                                 50.78 usec
TE                                                 290.5 K
D1                                             0.10000000 sec
D11                                            0.03000000 sec
TD0                                                     1

======== CHANNEL f1 ========
NUC1                                                   13C
P1                                                 11.00 usec
PL1                                                -1.00 dB
PL1W                                   109.73103333 W
SFO1                                   150.9405316 MHz

======== CHANNEL f2 ========
CPDPRG2                                            waltz65
NUC2                                                   1H
PCPD2                                              70.00 usec
PL2                                                4.20 dB
PL12                                               22.51 dB
PL2W                                   5.30020905 W
PL12W                                  0.07821552 W
SFO2                                   600.2224009 MHz
SI                                                 65536
SF                                     150.9253905 MHz
WDW                                                   EM
SSB                                                    0
LB                                                 2.00 Hz
GB                                                     0
PC                                                 1.00
```



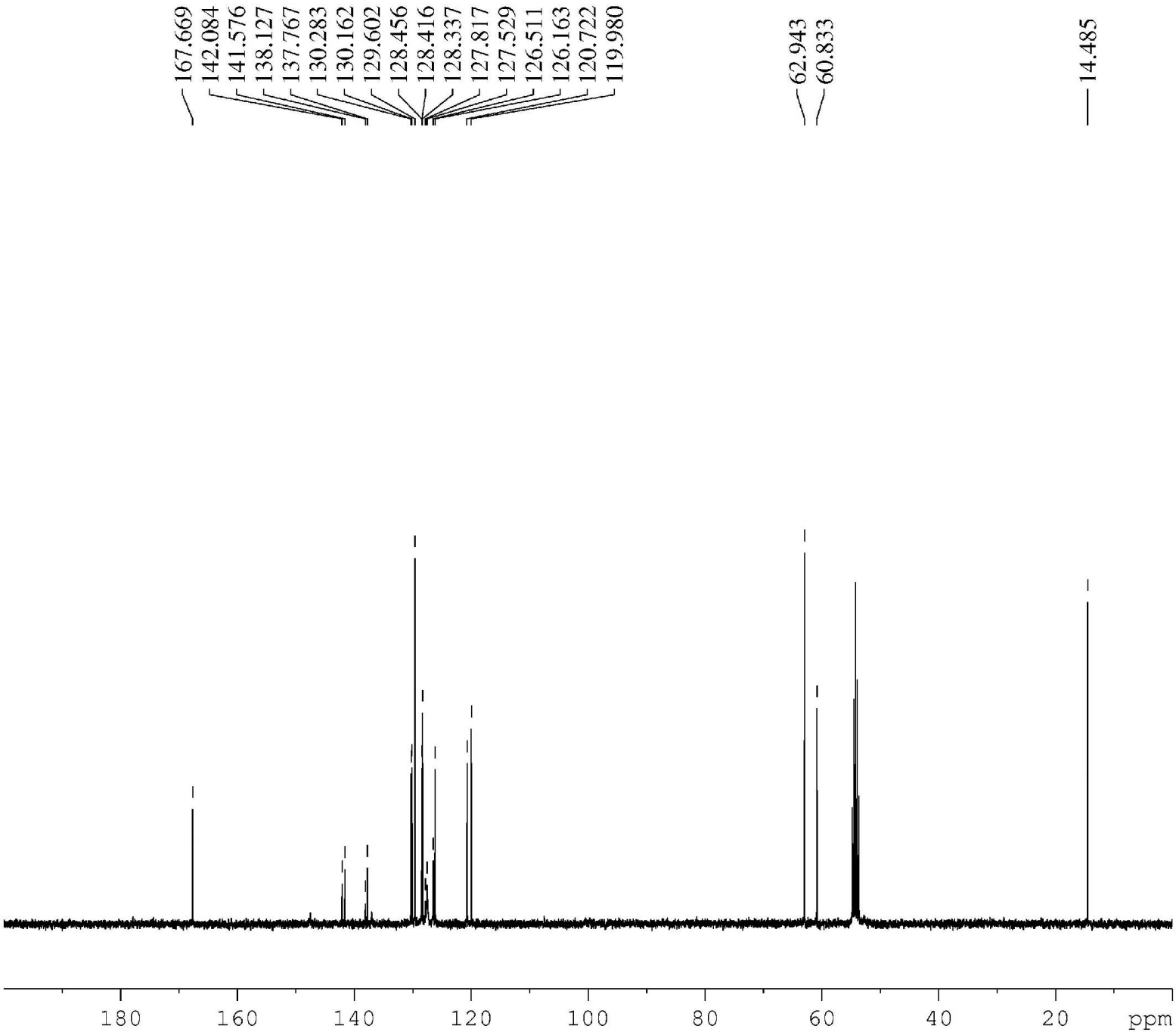

```
NAME                 compound 36
EXPNO                        10
PROCNO                        1
Date_                  20100613
Time                      18.18
INSTRUM                   av400
PROBHD 5 mm BBO BB-1H
PULPROG                   zg30
TD                       65536
SOLVENT                 CD2Cl2
NS                           32
DS                            8
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                         203.2
DW                    60.400 use
DE                     6.50 use
TE                      298.2 K
D1                   0.00300000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        1H
P1                    9.25 use
PL1                    0.00 dB
SFO1          400.1324710 MHz
SI                        32768
SF            400.1300476 MHz
WDW                          no
SSB                           0
LB                     0.00 Hz
GB                            0
PC                         2.00
```



NAME	compound 36_2
EXPNO	11
PROCNO	1
Date_	20100620
Time	19.59
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc30
TD	65536
SOLVENT	MeOD
NS	1500
DS	2
SWH	33112.582 Hz
FIDRES	0.505258 Hz
AQ	0.9896436 sec
RG	18390.4
DW	15.100 usec
DE	8.00 usec
TE	298.6 K
D1	0.03000000 sec
d11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.94 usec
PL1	5.00 dB
SFO1	100.6242789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	19.76 dB
SFO2	400.1324710 MHz
SI	32768
SF	100.6125861 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00




```
NAME                 compound 37
EXPNO                        10
PROCNO                        1
Date_                  20100613
Time                      18.39
INSTRUM                   av400
PROBHD     5 mm BBO BB-1H
PULPROG                   zg30
TD                       65536
SOLVENT              CD2Cl2
NS                           32
DS                            8
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                          114
DW                   60.400 use
DE                    6.50 use
TE                   298.2 K
D1                   0.00300000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                    9.25 use
PL1                    0.00 dB
SFO1          400.1324710 MHz
SI                        32768
SF            400.1300553 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    2.00
```



NAME	compound 37
EXPNO	11
PROCNO	1
Date_	20100613
Time	19.08
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc30
TD	65536
SOLVENT	CD2C12
NS	1500
DS	2
SWH	33112.582 Hz
FIDRES	0.505258 Hz
AQ	0.9896436 sec
RG	18390.4
DW	15.100 usec
DE	8.00 usec
TE	298.6 K
D1	0.03000000 sec
d11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.94 usec
PL1	5.00 dB
SFO1	100.6242789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	19.76 dB
SFO2	400.1324710 MHz
SI	32768
SF	100.6126888 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

Table 1. Crystal data and structure refinement for compound 2.

Identification code	6906	
Empirical formula	$C_{54}H_{32} \cdot CH_2Cl_2$	
Color	orange	
Formula weight	765.72 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca, (no. 61)	
Unit cell dimensions	a = 18.3571(17) Å	α= 90°.
	b = 17.9992(19) Å	β= 90°.
	c = 23.152(4) Å	γ = 90°.
Volume	7649.8(17) Å^3	
Z	8	
Density (calculated)	1.330 Mg · m^{-3}	
Absorption coefficient	0.210 mm^{-1}	
F(000)	3184 e	
Crystal size	0.512 x 0.114 x 0.095 mm^3	
θ range for data collection	2.64 to 33.20°.	
Index ranges	-16 ≤ h ≤ 28, -27≤ k ≤ 26, -35≤ l ≤ 35	
Reflections collected	66746	
Independent reflections	14624 [R_{int} = 0.0821]	
Reflections with I>2σ(I)	8578	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.98	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14624 / 0 / 514	
Goodness-of-fit on F^2	1.046	
Final R indices [I>2σ(I)]	R_1 = 0.0651	wR^2 = 0.1191
R indices (all data)	R_1 = 0.1348	wR^2 = 0.1437
Largest diff. peak and hole	0.528 and -0.595 e · Å^{-3}	

Table 2. Crystal data and structure refinement of compound 5.

Identification code	7053	
Empirical formula	$C_{17} H_8 F_6$	
Color	colourless	
Formula weight	326.23 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	**$Pna2_1$, (no. 33)**	
Unit cell dimensions	a = 7.266(2) Å	α= 90°.
	b = 20.145(6) Å	β= 90°.
	c = 9.725(3) Å	γ = 90°.
Volume	1423.4(7) Å^3	
Z	4	
Density (calculated)	1.522 Mg · m^{-3}	
Absorption coefficient	0.143 mm^{-1}	
F(000)	656 e	
Crystal size	0.09 x 0.026 x 0.026 mm^3	
θ range for data collection	2.33 to 26.44°.	
Index ranges	$-9 \le h \le 8$, $-25 \le k \le 25$, $-12 \le l \le 12$	
Reflections collected	25958	
Independent reflections	2869 [R_{int} = 0.0786]	
Reflections with I>2σ(I)	2559	
Completeness to θ = 26.44°	98.6 %	
Absorption correction	Gaussian	
Max. and min. transmission	1.00 and 0.99	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2869 / 1 / 208	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2σ(I)]	R_1 = 0.0643	wR^2 = 0.1737
R indices (all data)	R_1 = 0.0719	wR^2 = 0.1819
Absolute structure parameter	0.4(13)	
Largest diff. peak and hole	0.874 and -0.363 e · Å^{-3}	

Table 3. Crystal data and structure refinement of compound 6.

Identification code	6971	
Empirical formula	$C_{21}H_{18}O_4$	
Color	colourless	
Formula weight	334.35 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/c$, (no. 14)**	
Unit cell dimensions	a = 9.5811(7) Å	α= 90°.
	b = 20.9360(19) Å	β= 115.659(6)°.
	c = 9.7491(7) Å	γ = 90°.
Volume	1762.7(2) Å^3	
Z	4	
Density (calculated)	1.260 Mg · m^{-3}	
Absorption coefficient	0.087 mm^{-1}	
F(000)	704 e	
Crystal size	0.30 x 0.12 x 0.07 mm^3	
θ range for data collection	2.67 to 33.17°.	
Index ranges	-14 ≤ h ≤ 14, -28≤ k ≤ 32, -14≤ l ≤ 14	
Reflections collected	36463	
Independent reflections	6720 [R_{int} = 0.0835]	
Reflections with I>2σ(I)	4666	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.97	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6720 / 0 / 228	
Goodness-of-fit on F^2	1.194	
Final R indices [I>2σ(I)]	R_1 = 0.0656	wR^2 = 0.1703
R indices (all data)	R_1 = 0.1006	wR^2 = 0.1922
Largest diff. peak and hole	0.440 and -0.470 e · Å^{-3}	

Table 4. Crystal data and structure refinement for compound 8.

Identification code	6979sadabs	
Empirical formula	$C_{21}H_{16}F_2O_4$	
Color	colourless	
Formula weight	370.34 g · mol^{-1}	
Temperature	100 K	
Wavelength	1.54178 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/n$, (no. 14)**	
Unit cell dimensions	a = 4.5285(2) Å	α= 90°.
	b = 45.944(2) Å	β= 101.410(3)°.
	c = 8.2961(4) Å	γ = 90°.
Volume	1691.96(14) Å^3	
Z	4	
Density (calculated)	1.454 Mg · m^{-3}	
Absorption coefficient	0.972 mm^{-1}	
F(000)	768 e	
Crystal size	0.35 x 0.04 x 0.04 mm^3	
θ range for data collection	1.92 to 54.79°.	
Index ranges	-4 ≤ h ≤ 4, -48≤ k ≤ 48, -8≤ l ≤ 8	
Reflections collected	18616	
Independent reflections	2071 [R_{int} = 0.0651]	
Reflections with I>2σ(I)	1705	
Completeness to θ = 54.79°	98.1 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.97 and 0.83	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2071 / 0 / 246	
Goodness-of-fit on F^2	1.056	
Final R indices [I>2σ(I)]	R_1 = 0.0544	wR^2 = 0.1368
R indices (all data)	R_1 = 0.0671	wR^2 = 0.1465
Largest diff. peak and hole	0.376 and -0.317 e · Å^{-3}	

Table 5. Crystal data and structure refinement for compound 17.

Identification code	6946	
Empirical formula	$C_{14}H_{12}F_{12}N_2$	
Color	yellow	
Formula weight	436.26 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/n$, (no. 14)**	
Unit cell dimensions	a = 14.0303(8) Å	α= 90°.
	b = 8.0790(8) Å	β= 111.802(4)°.
	c = 15.6424(12) Å	γ = 90°.
Volume	1646.3(2) Å^3	
Z	4	
Density (calculated)	1.760 Mg · m^{-3}	
Absorption coefficient	0.201 mm^{-1}	
F(000)	872 e	
Crystal size	0.29 x 0.27 x 0.14 mm^3	
θ range for data collection	2.80 to 33.23°.	
Index ranges	-21 ≤ h ≤ 21, -12≤ k ≤ 12, -24≤ l ≤ 24	
Reflections collected	33671	
Independent reflections	6308 [R_{int} = 0.0422]	
Reflections with I>2σ(I)	4676	
Completeness to θ = 30.00°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.97398 and 0.95608	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6308 / 0 / 257	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2σ(I)]	R_1 = 0.0557	wR^2 = 0.1597
R indices (all data)	R_1 = 0.0786	wR^2 = 0.1772
Largest diff. peak and hole	0.732 and -0.643 e · Å^{-3}	

Table 6. Crystal data and structure refinement of compound 18.

Identification code	6966	
Empirical formula	$C_{26}H_{40}N_2O_8$	
Color	yellow	
Formula weight	508.60 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**C 2/c, (no. 15)**	
Unit cell dimensions	a = 10.3691(5) Å	α= 90°.
	b = 16.1875(9) Å	β= 95.356(4)°.
	c = 15.8390(10) Å	γ = 90°.
Volume	2647.0(3) Å^3	
Z	4	
Density (calculated)	1.276 Mg · m^{-3}	
Absorption coefficient	0.094 mm^{-1}	
F(000)	1096 e	
Crystal size	0.40 x 0.26 x 0.17 mm^3	
θ range for data collection	2.76 to 36.10°.	
Index ranges	-17 ≤ h ≤ 17, -26≤ k ≤ 26, -26≤ l ≤ 26	
Reflections collected	48455	
Independent reflections	6305 [R_{int} = 0.0334]	
Reflections with I>2σ(I)	5257	
Completeness to θ = 27.50°	99.8 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.97	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6305 / 0 / 169	
Goodness-of-fit on F^2	1.024	
Final R indices [I>2σ(I)]	R_1 = 0.0360	wR^2 = 0.0919
R indices (all data)	R_1 = 0.0476	wR^2 = 0.0979
Largest diff. peak and hole	0.499 and -0.270 e · Å^{-3}	

Table 7. Crystal data and structure refinement for compound 19.

Identification code	6975	
Empirical formula	$C_{32}H_{38}N_2O_4$	
Color	red	
Formula weight	514.64 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	ORTHORHOMBIC	
Space group	**Pbca, (no. 61)**	
Unit cell dimensions	a = 13.7855(13) Å	α= 90°.
	b = 18.3094(12) Å	β= 90°.
	c = 21.682(2) Å	γ = 90°.
Volume	5472.7(9) $Å^3$	
Z	8	
Density (calculated)	1.249 Mg· m^{-3}	
Absorption coefficient	0.082 mm^{-1}	
F(000)	2208 e	
Crystal size	0.38 x 0.21 x 0.18 mm^3	
θ range for data collection	2.64 to 33.18°.	
Index ranges	$-17 \le h \le 21$, $-28 \le k \le 28$, $-33 \le l \le 33$	
Reflections collected	80356	
Independent reflections	10457 [R_{int} = 0.0601]	
Reflections with I>2σ(I)	8250	
Completeness to θ = 27.50°	99.8 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.98	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10457 / 0 / 351	
Goodness-of-fit on F^2	1.077	
Final R indices [I>2σ(I)]	R_1 = 0.0465	wR^2 = 0.1162
R indices (all data)	R_1 = 0.0669	wR^2 = 0.1296
Largest diff. peak and hole	0.604 and -0.499 e · $Å^{-3}$	

Table 8. Crystal data and structure refinement of compound 20.

Identification code	7008	
Empirical formula	$C_{32}H_{36}F_2N_2O_4$	
Color	red	
Formula weight	550.63 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	ORTHORHOMBIC	
Space group	**Pbca, (no. 61)**	
Unit cell dimensions	a = 13.9352(18) Å	α= 90°.
	b = 18.6325(14) Å	β= 90°.
	c = 21.797(3) Å	γ = 90°.
Volume	5659.6(11) Å^3	
Z	8	
Density (calculated)	1.292 Mg · m^{-3}	
Absorption coefficient	0.094 mm^{-1}	
F(000)	2336 e	
Crystal size	0.31 x 0.21 x 0.14 mm^3	
θ range for data collection	2.61 to 33.22°.	
Index ranges	-21 ≤ h ≤ 21, -28≤ k ≤ 28, -33≤ l ≤ 33	
Reflections collected	96395	
Independent reflections	10840 [R_{int} = 0.0717]	
Reflections with I>2σ(I)	7534	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.98	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10840 / 0 / 369	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2σ(I)]	R_1 = 0.0514	wR^2 = 0.1081
R indices (all data)	R_1 = 0.0881	wR^2 = 0.1245
Largest diff. peak and hole	0.463 and -0.322 e · Å^{-3}	

Table 9. Crystal data and structure refinement of compound 28.

Identification code	7000	
Empirical formula	$C_{44}H_{37}F_6P_3S$	
Color	colourless	
Formula weight	804.71 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	TRICLINIC	
Space group	**$P\bar{1}$, (no. 2)**	
Unit cell dimensions	a = 10.4125(4) Å	α= 98.106(5)°.
	b = 12.2494(14) Å	β= 97.846(5)°.
	c = 16.5886(7) Å	γ = 110.584(5)°.
Volume	1921.0(2) Å^3	
Z	2	
Density (calculated)	1.391 Mg · m^{-3}	
Absorption coefficient	0.271 mm^{-1}	
F(000)	832 e	
Crystal size	0.14 x 0.12 x 0.07 mm^3	
θ range for data collection	2.69 to 30.07°.	
Index ranges	-14 ≤ h ≤ 14, -16≤ k ≤ 17, -23≤ l ≤ 23	
Reflections collected	26184	
Independent reflections	10957 [R_{int} = 0.0600]	
Reflections with I>2σ(I)	7538	
Completeness to θ = 27.50°	98.8 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.98 and 0.97	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10957 / 0 / 488	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2σ(I)]	R_1 = 0.0565	wR^2 = 0.1275
R indices (all data)	R_1 = 0.0948	wR^2 = 0.1464
Largest diff. peak and hole	0.404 and -0.732 e · Å^{-3}	

Table 10. Crystal data and structure refinement of compound 39.

Identification code	7080	
Empirical formula	$C_{28}H_{24}F_2O_4S$	
Color	yellow	
Formula weight	494.53 g · mol^{-1}	
Temperature	200 K	
Wavelength	0.71073 Å	
Crystal system	TRICLINIC	
Space group	**P$\bar{1}$, (no. 2)**	
Unit cell dimensions	a = 7.9304(17) Å	α= 98.06(2)°.
	b = 12.5572(16) Å	β= 104.24(2)°.
	c = 13.327(4) Å	γ = 105.687(12)°.
Volume	1207.7(5) Å^3	
Z	2	
Density (calculated)	1.360 Mg · m^{-3}	
Absorption coefficient	0.182 mm^{-1}	
F(000)	516 e	
Crystal size	0.27 x 0.23 x 0.17 mm^3	
θ range for data collection	2.61 to 28.16°.	
Index ranges	-10 ≤ h ≤ 10, -16≤ k ≤ 16, -17≤ l ≤ 17	
Reflections collected	21411	
Independent reflections	5879 [R_{int} = 0.0333]	
Reflections with I>2σ(I)	4668	
Completeness to θ = 28.16°	99.1 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.97 and 0.95	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5879 / 0 / 327	
Goodness-of-fit on F^2	1.052	
Final R indices [I>2σ(I)]	R_1 = 0.0452	wR^2 = 0.1106
R indices (all data)	R_1 = 0.0610	wR^2 = 0.1207
Largest diff. peak and hole	0.339 and -0.252 e · Å^{-3}	