

Supplementary Material

New Synthesis of Dibenzofulvenes via Palladium-Catalyzed Double Cross-Coupling Reactions

Masaki Shimizu,^{A,B} Ikuhiro Nagao,^A Shin-ichi Kiyomoto,^A and Tamejiro Hiyama^{A,C}

^A Department of Material Chemistry, Graduate School of Engineering, Kyoto University
Kyoto University Katsura, Nishikyo-ku, Kyoto 615-8510, Japan

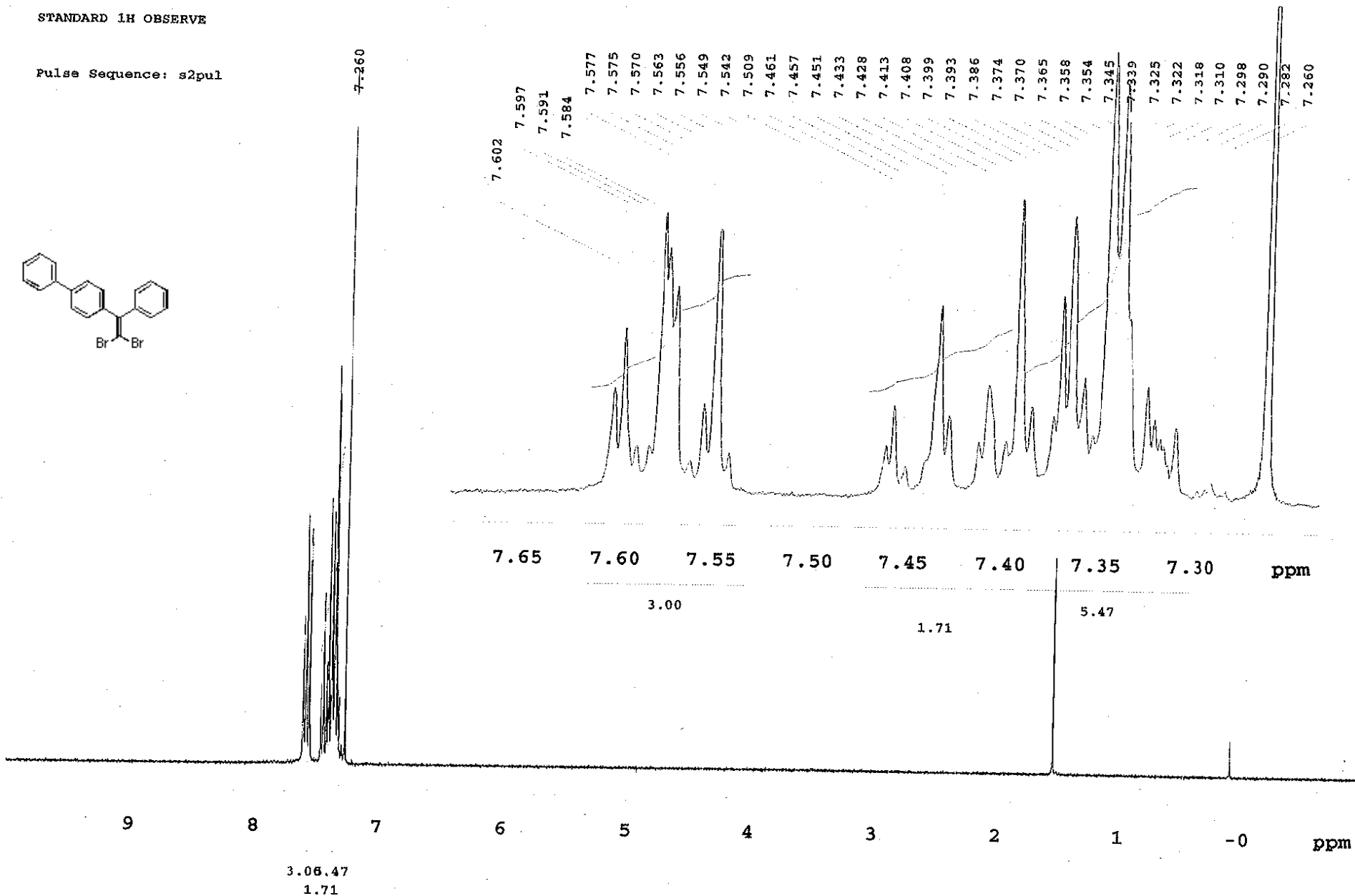
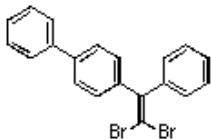
^B Corresponding author. Email: m.shimizu@hs2.ecs.kyoto-u.ac.jp

^C Current address: Research & Development Initiative, Chuo University, 1-13-27, Kasuga, Bunkyo-ku, Tokyo 112-8551, Japan

This material includes ¹H and ¹³C NMR spectra of **8m**, **4b**, **4d**, **4e**, **4j**, **4m**, **4n**, **4o**, **4p**, and **4s**.

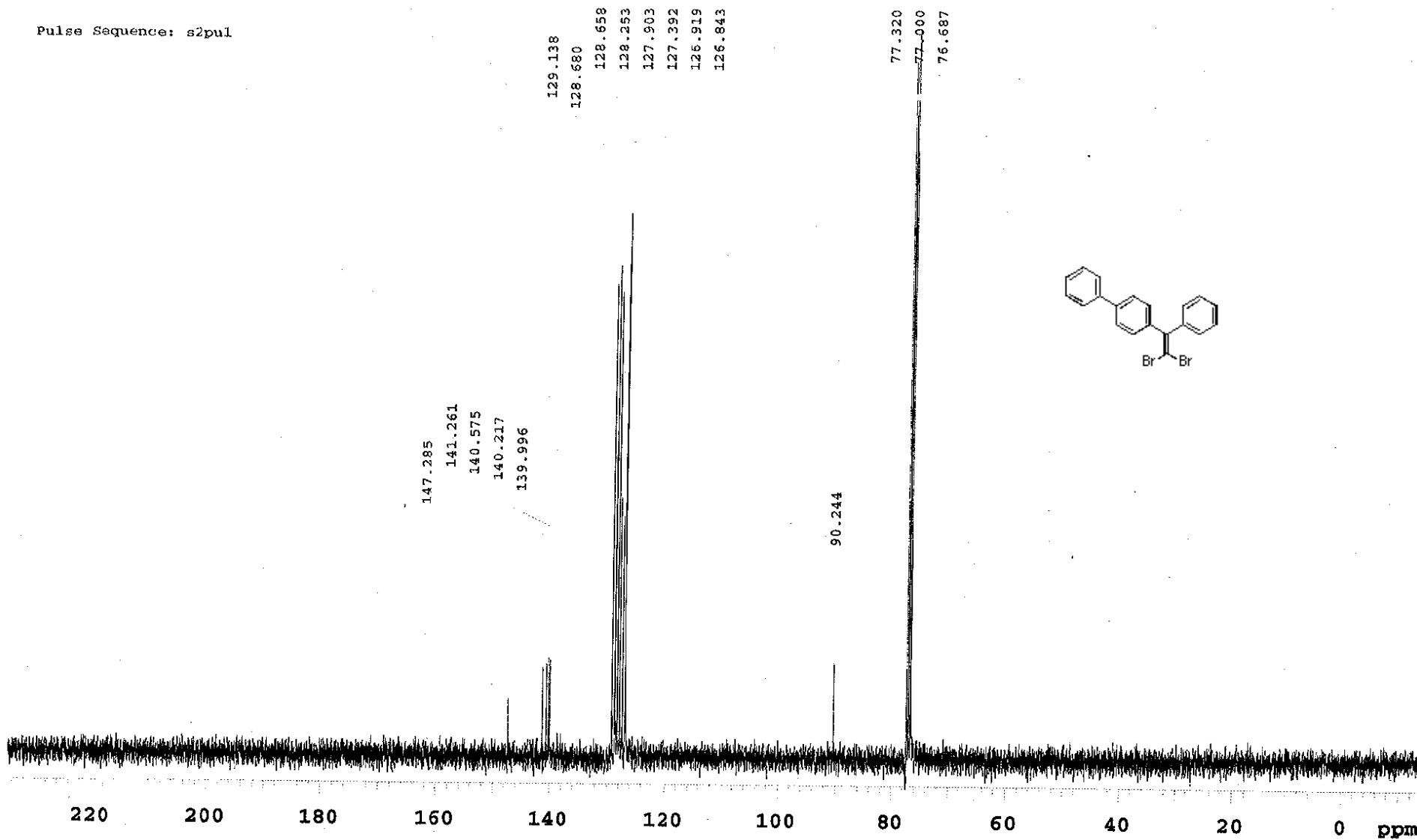
STANDARD 1H OBSERVE

Pulse Sequence: s2pul



13C OBSERVE

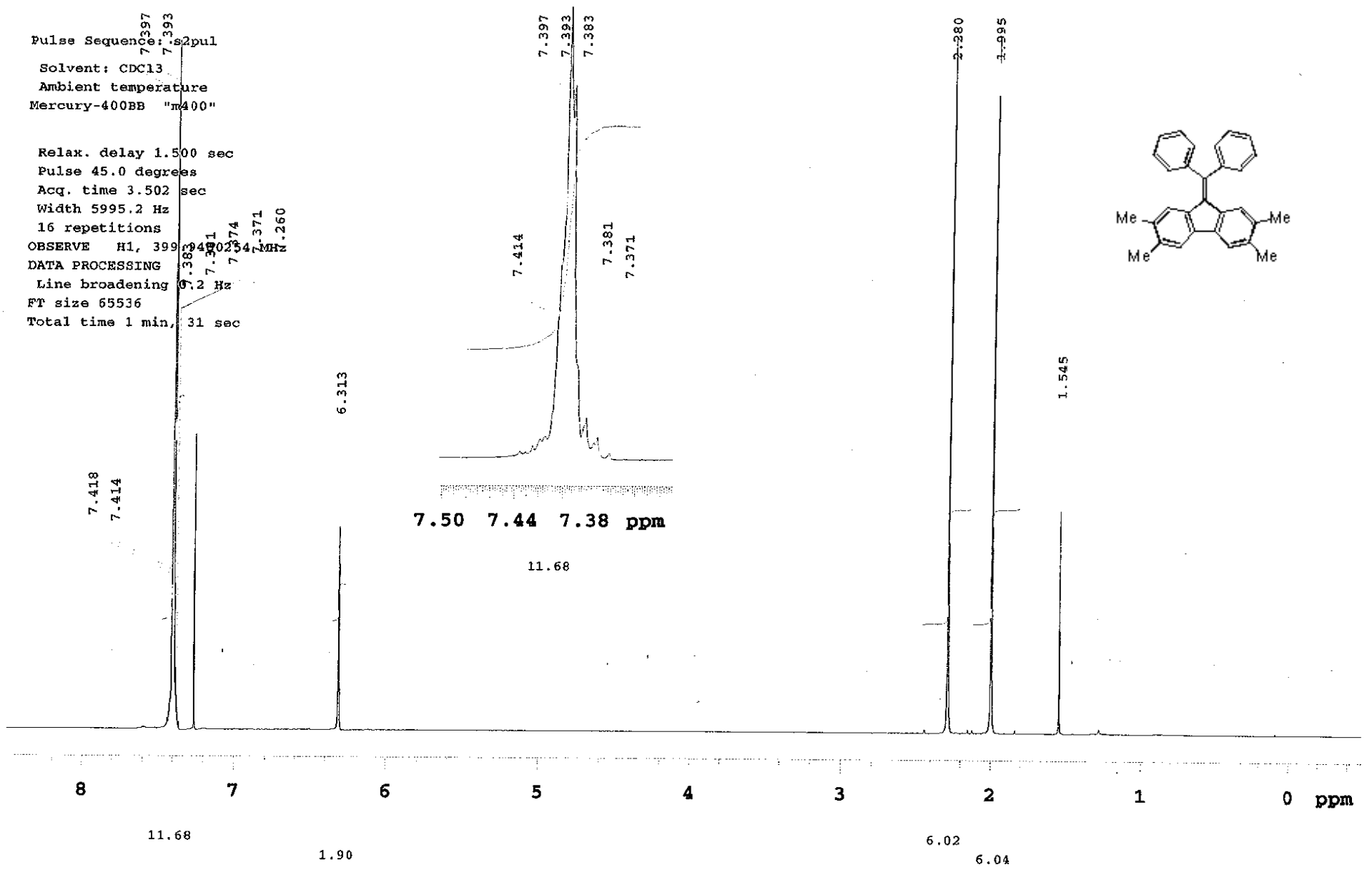
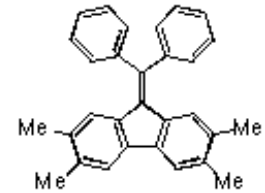
Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

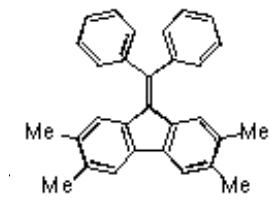
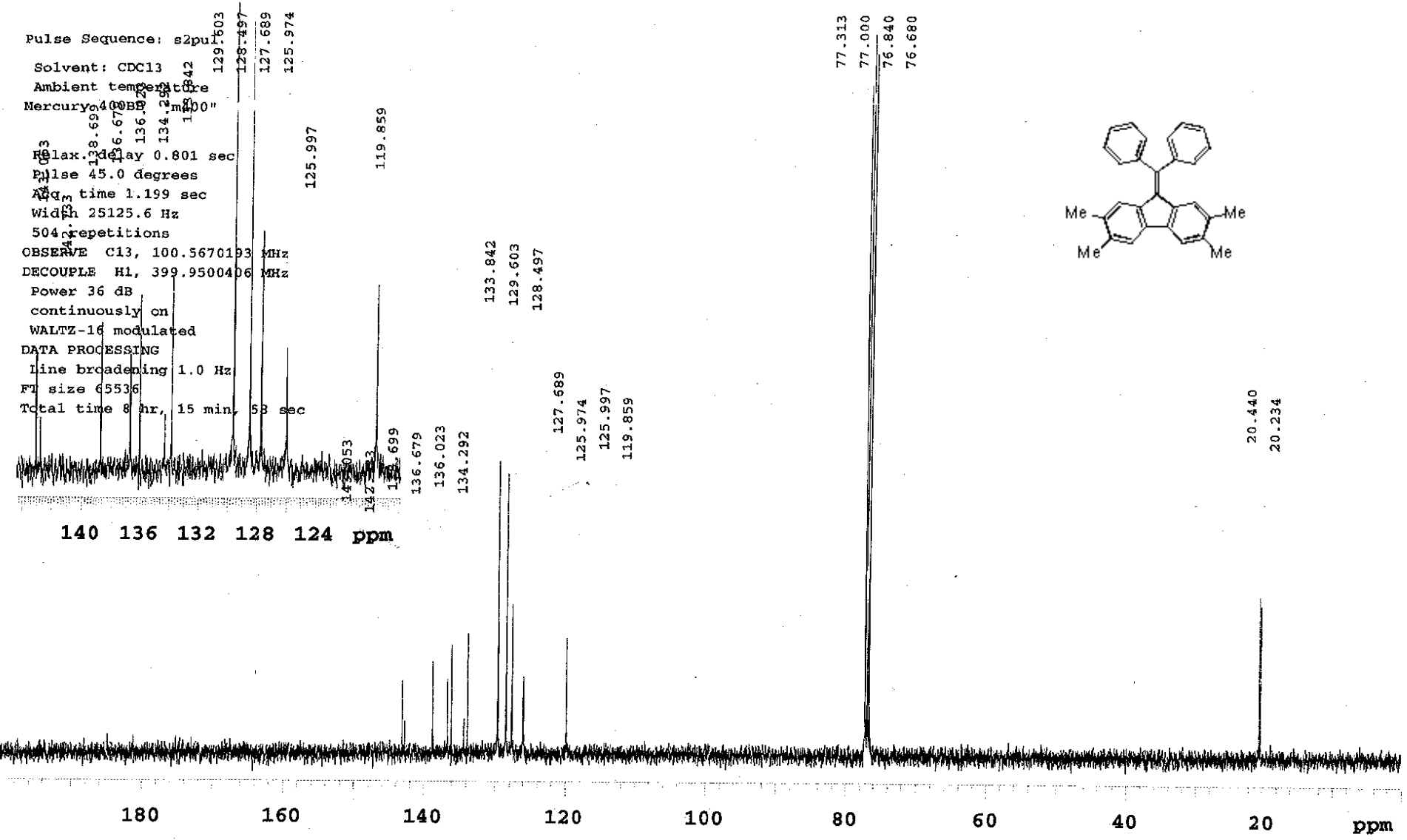
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.131 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 31 sec



13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury 400BB
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
504 repetitions
OBSERVE C13, 100.5670193 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 15 min, 58 sec



13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

3008 repetitions

OBSERVE C13, 100.5670208 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

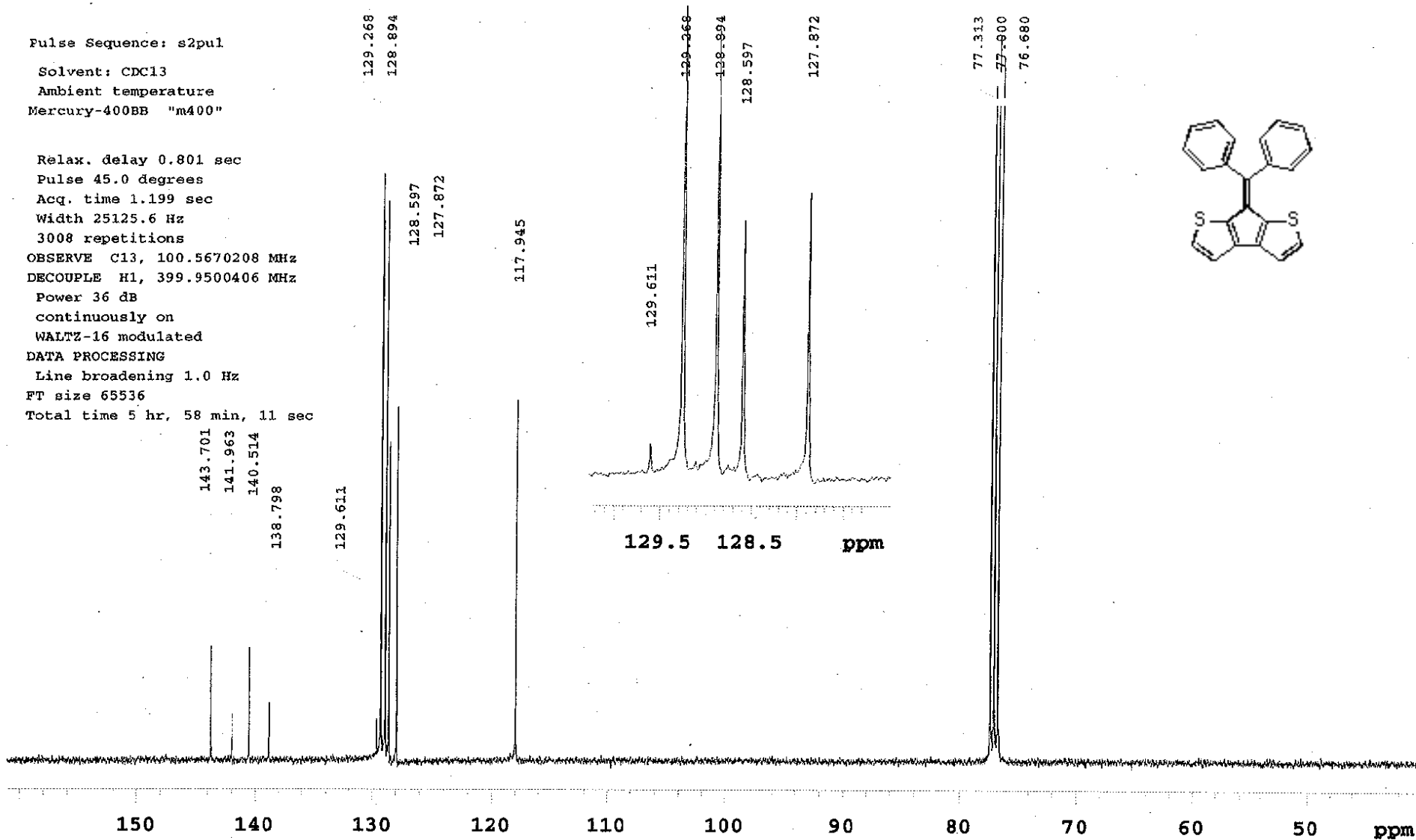
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 5 hr, 58 min, 11 sec



STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

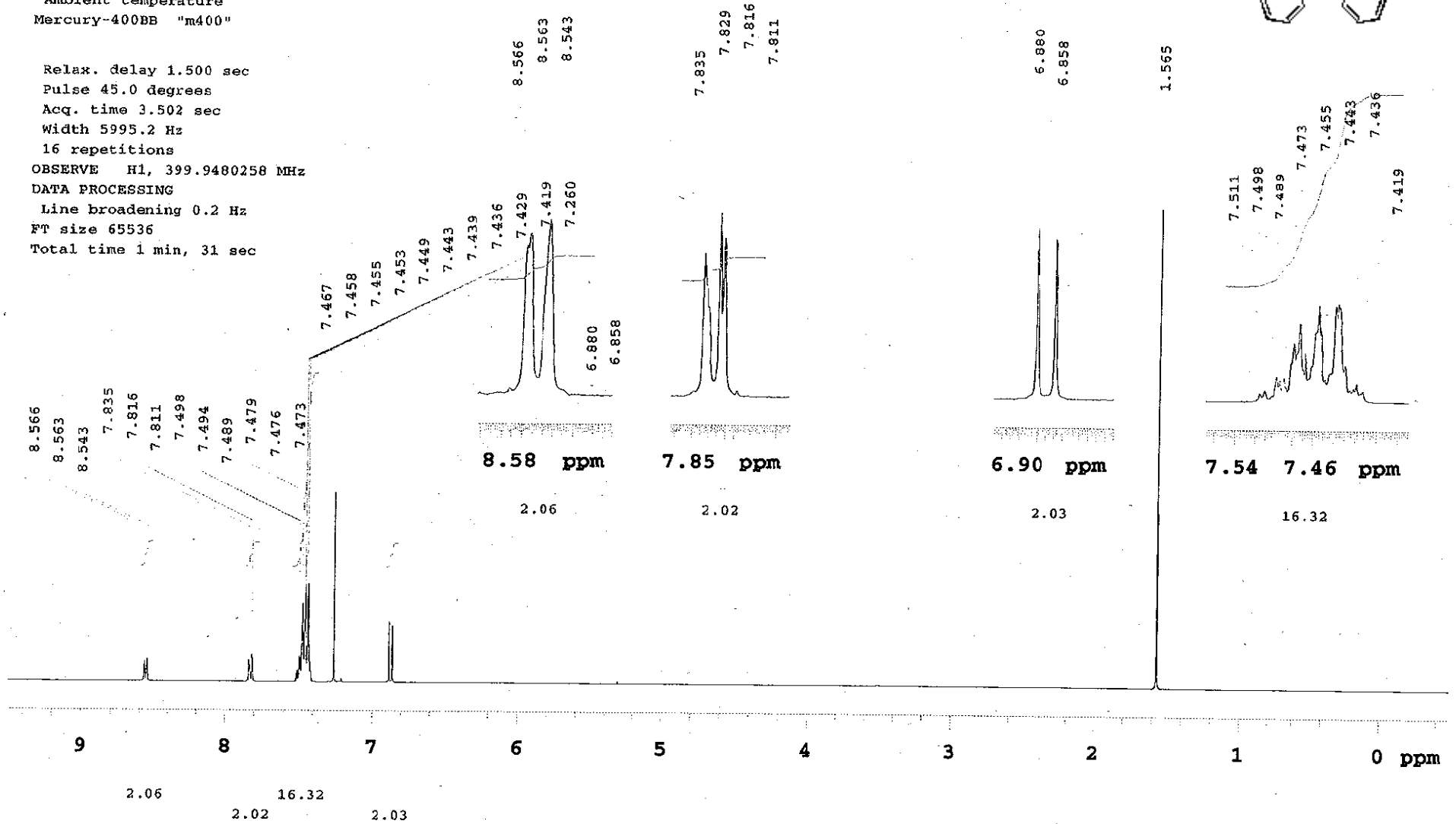
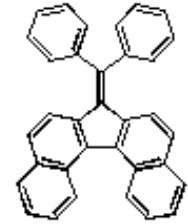
OBSERVE H1, 399.9480258 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 31 sec



13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

200 repetitions

OBSERVE C13, 100.5670216 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously

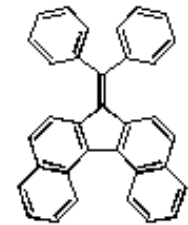
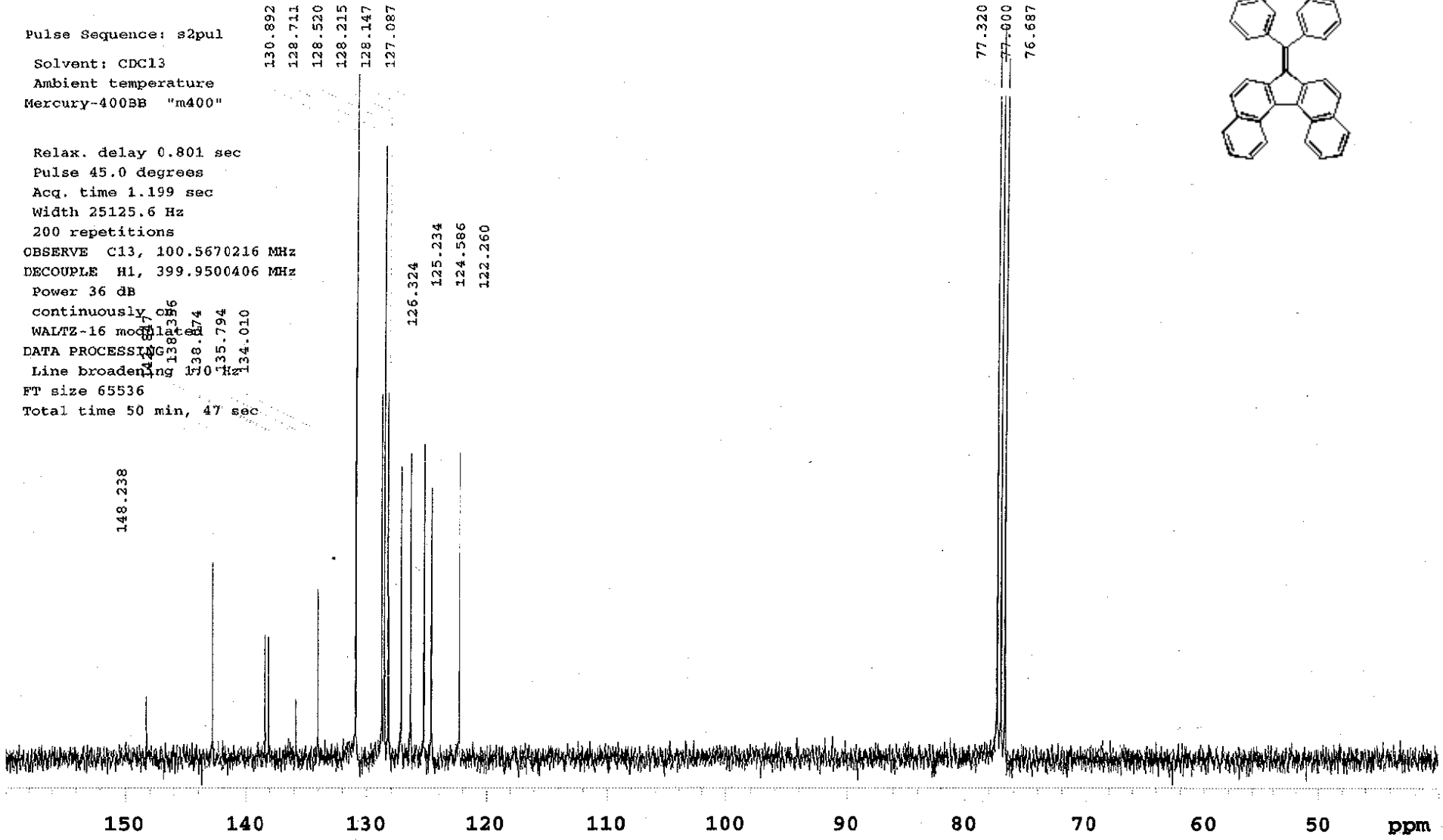
WALTZ-16 mod

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 50 min, 47 sec



13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

224 repetitions

OBSERVE C13, 100.5670216 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

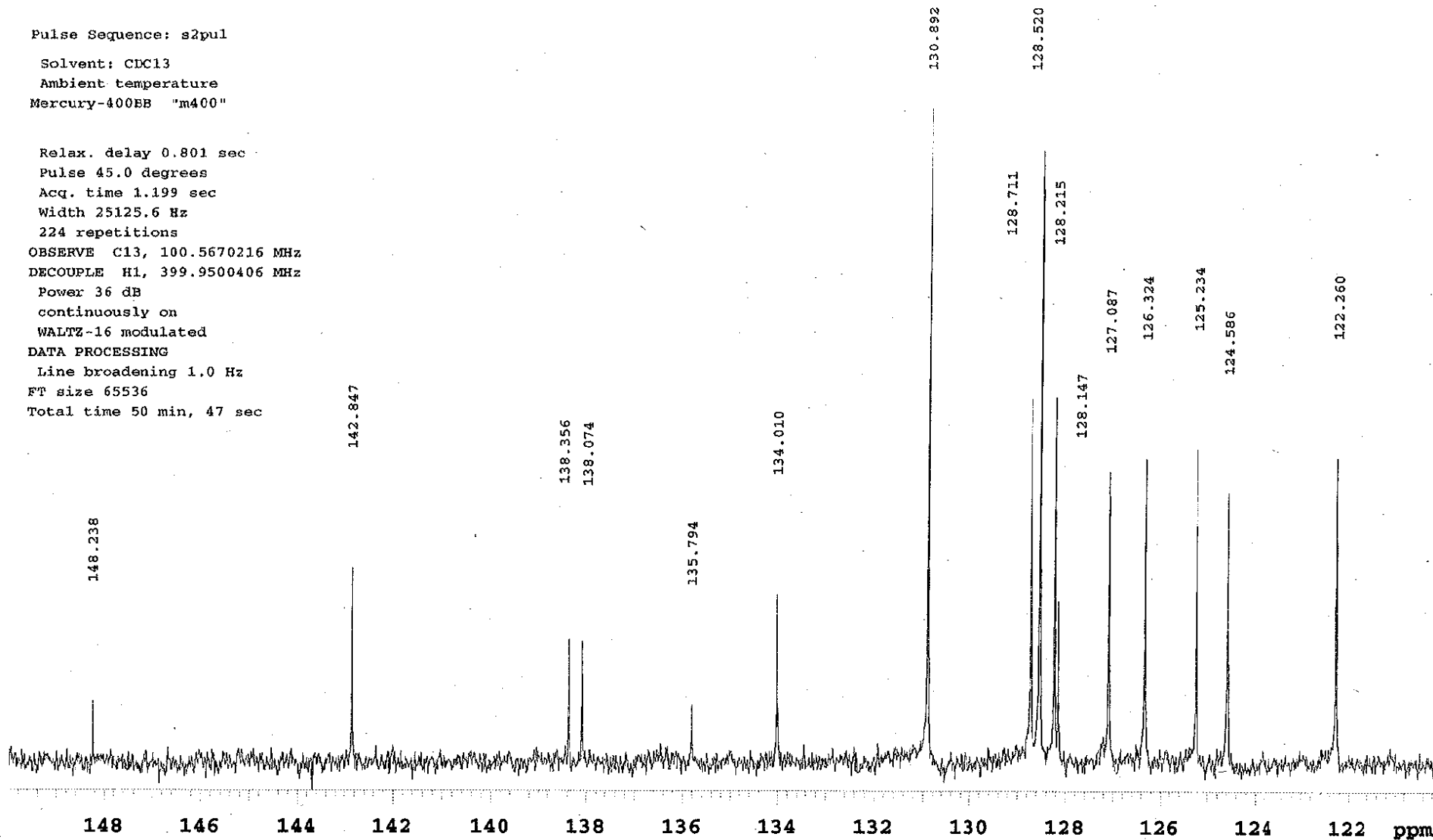
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 50 min, 47 sec



STANDARD 1H OBSERVE

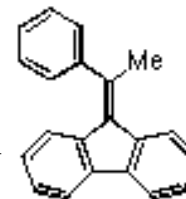
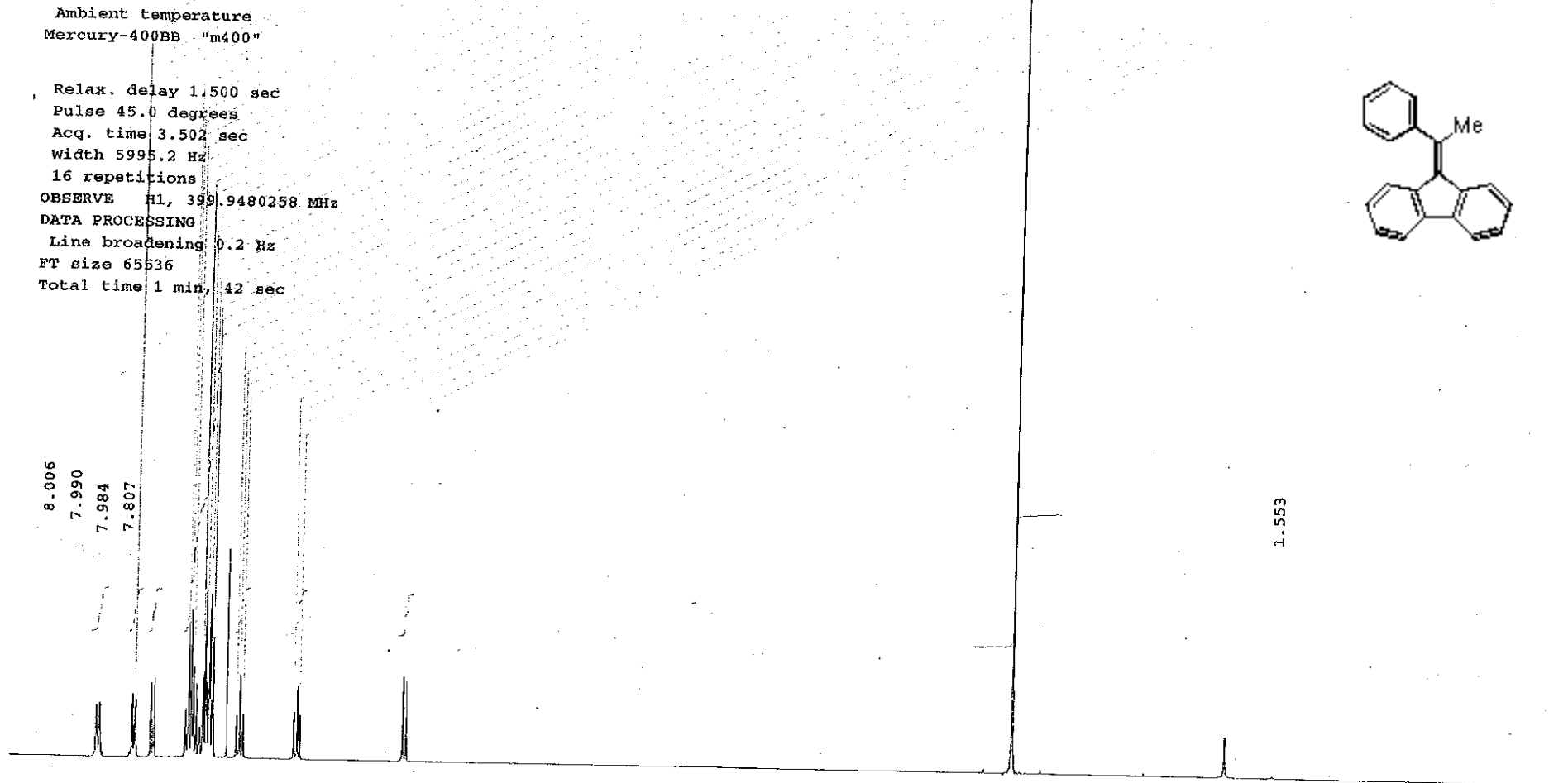
Pulse Sequence
7.801
7.794
7.789
7.786
7.701

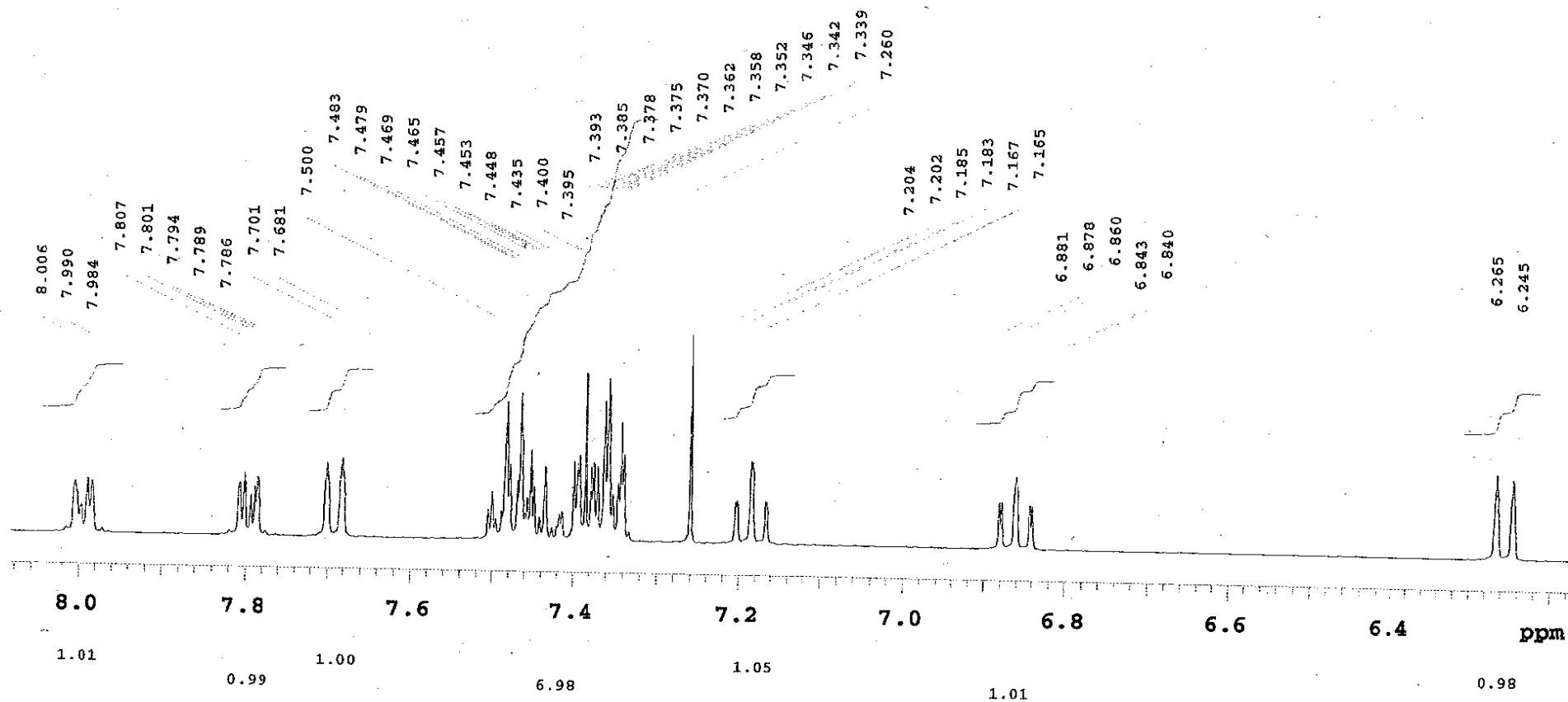
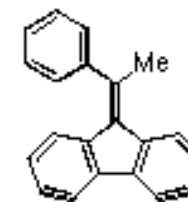
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9480258 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 42 sec

8.006
7.990
7.984
7.807

7.681 7.500 7.483 7.479 7.469 7.465 7.457 7.453 7.448 7.435 7.400 7.395 7.393 7.385 7.378 7.375 7.370 7.362 7.358 7.352 7.346 7.342 7.339 7.260 7.204 7.202 7.185 7.183 7.167 7.165 6.881 6.878 6.860 6.843 6.840 6.265 6.245 2.780





13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

18800 repetitions

OBSERVE C13, 100.5670216 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 29 min, 32 sec

145.232
142.617
140.280
139.441
138.769
138.371
132.889

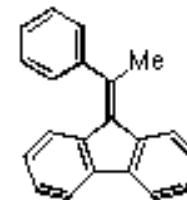
128.909
127.491
127.460
127.140

126.744
126.675
126.256
125.173
124.472
119.432
118.936

77.320
77.206
77.130
77.000
76.809
76.680

25.998

160 140 120 100 80 60 40 20 0 ppm



13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

18800 repetitions

OBSERVE C13, 100.5670216 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

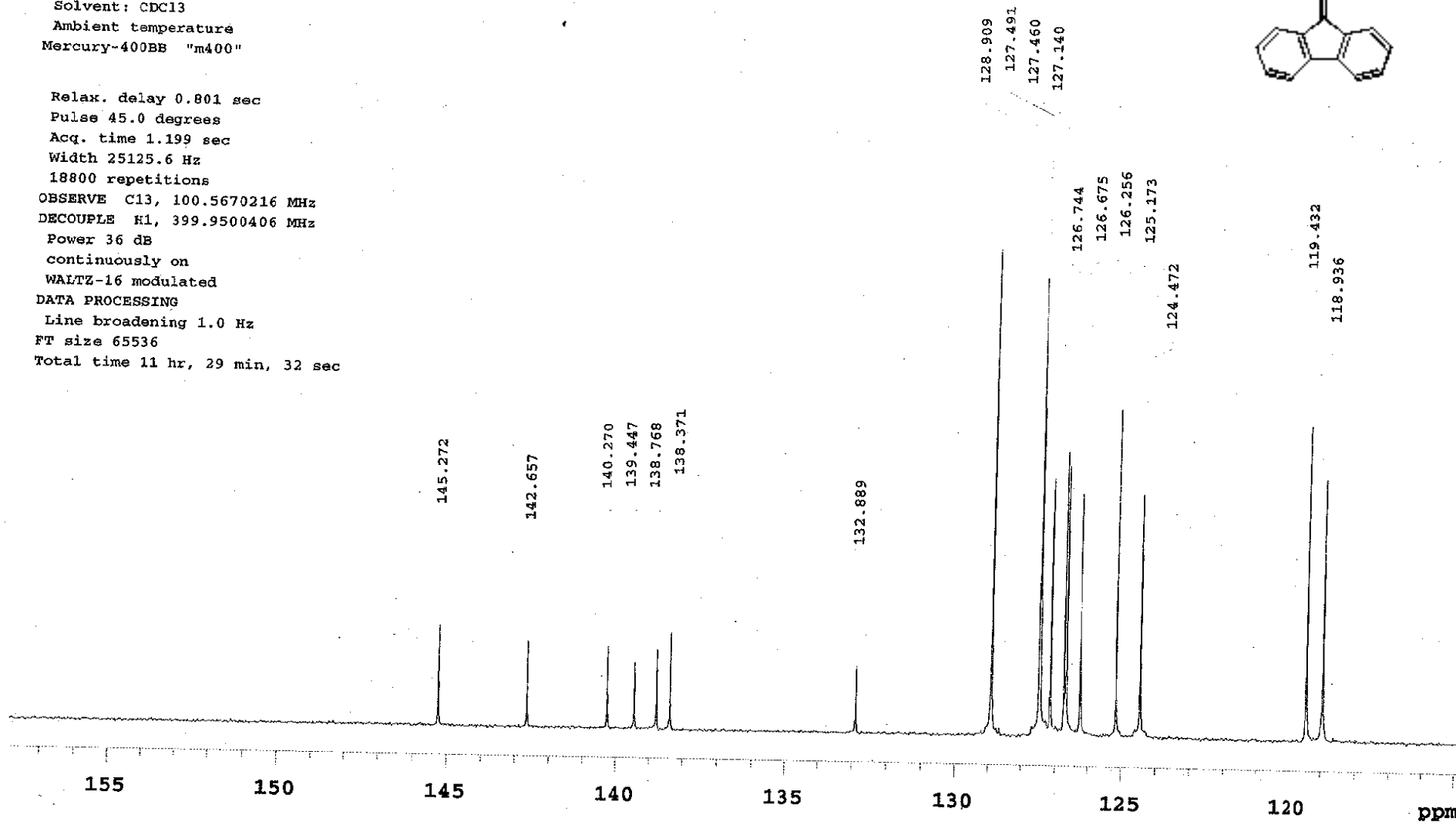
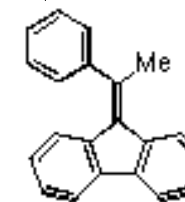
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

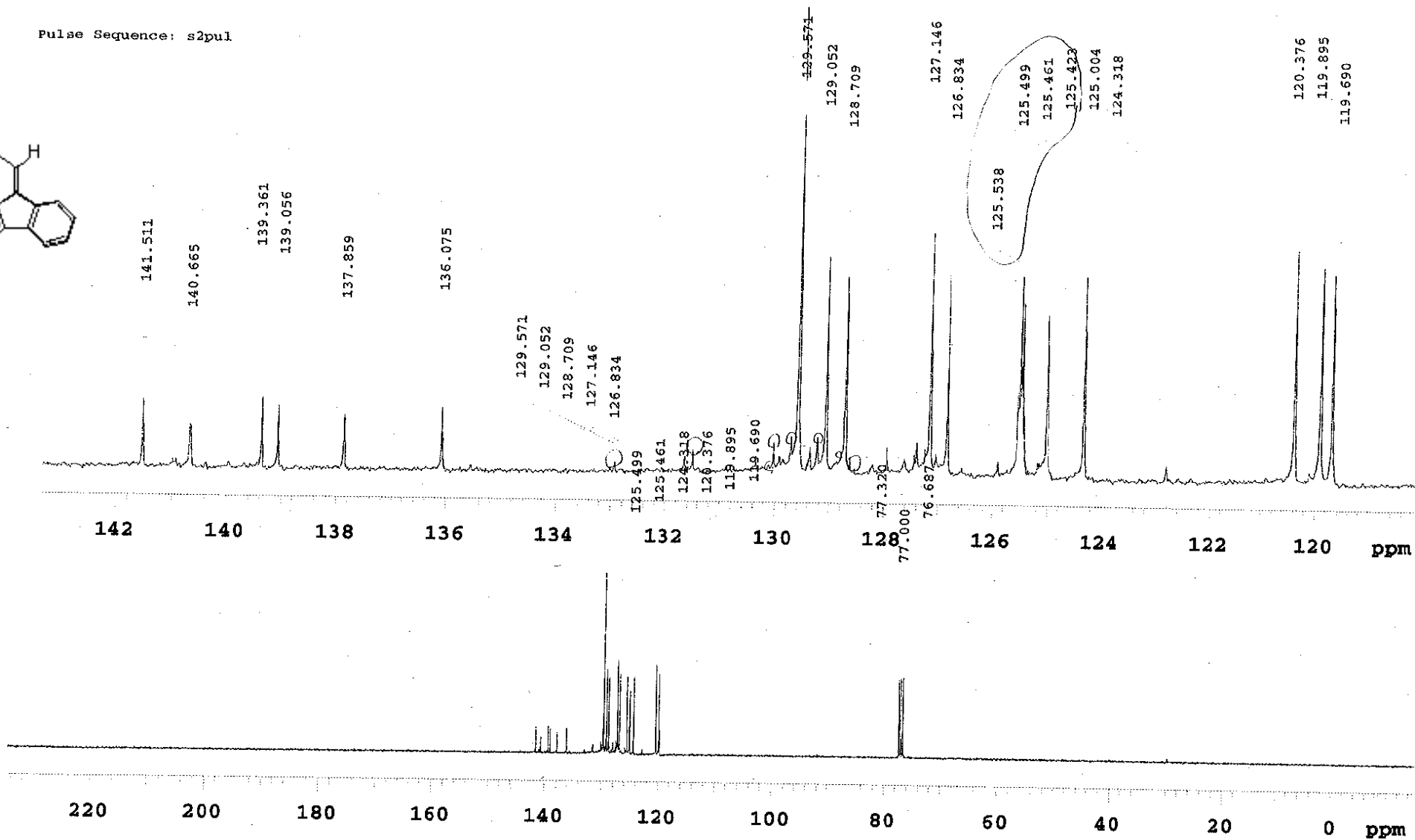
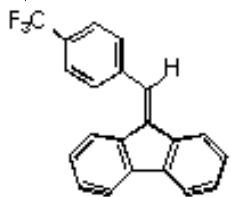
FT size 65536

Total time 11 hr, 29 min, 32 sec



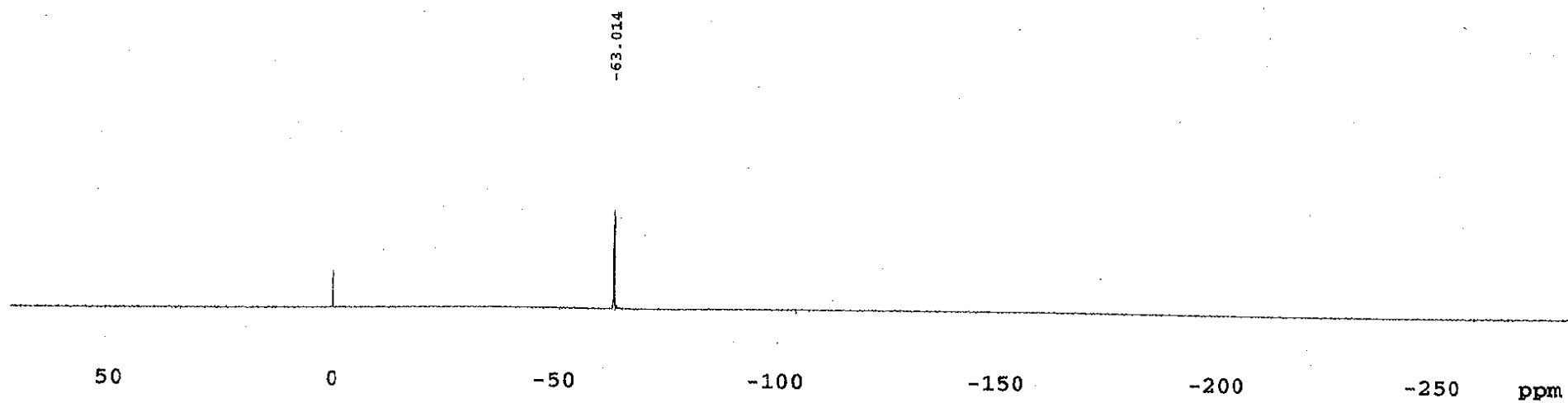
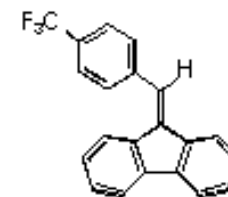
13C OBSERVE

Pulse Sequence: s2pul



FLUORINE SENSITIVITY
0.05% TRIFLUOROTOLUENE

Pulse Sequence: s2pul



STANDARD 1H OBSERVE

Pulse Sequence

8.151

8.147

8.134

8.130

7.779

7.761

7.725

7.720

7.717

7.704

7.699

7.669

7.663

7.649

7.643

7.636

7.511

7.491

7.417

7.401

7.383

7.358

7.355

7.345

8.155

7.339

7.329

7.321

7.318

7.310

7.260

7.079

7.062

7.060

7.044

7.040

3.982

8.4

8.2

8.0

7.8

7.6

7.4

7.2

ppm

2.11

2.19

1.18

2.22

1.19

3.27

1.37

1.08

13

12

11

10

9

8

7

6

5

4

3

2

1

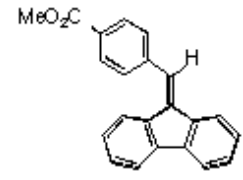
-0

ppm

2.11 2.19 2.22

1.18 1.37 1.08

3.00



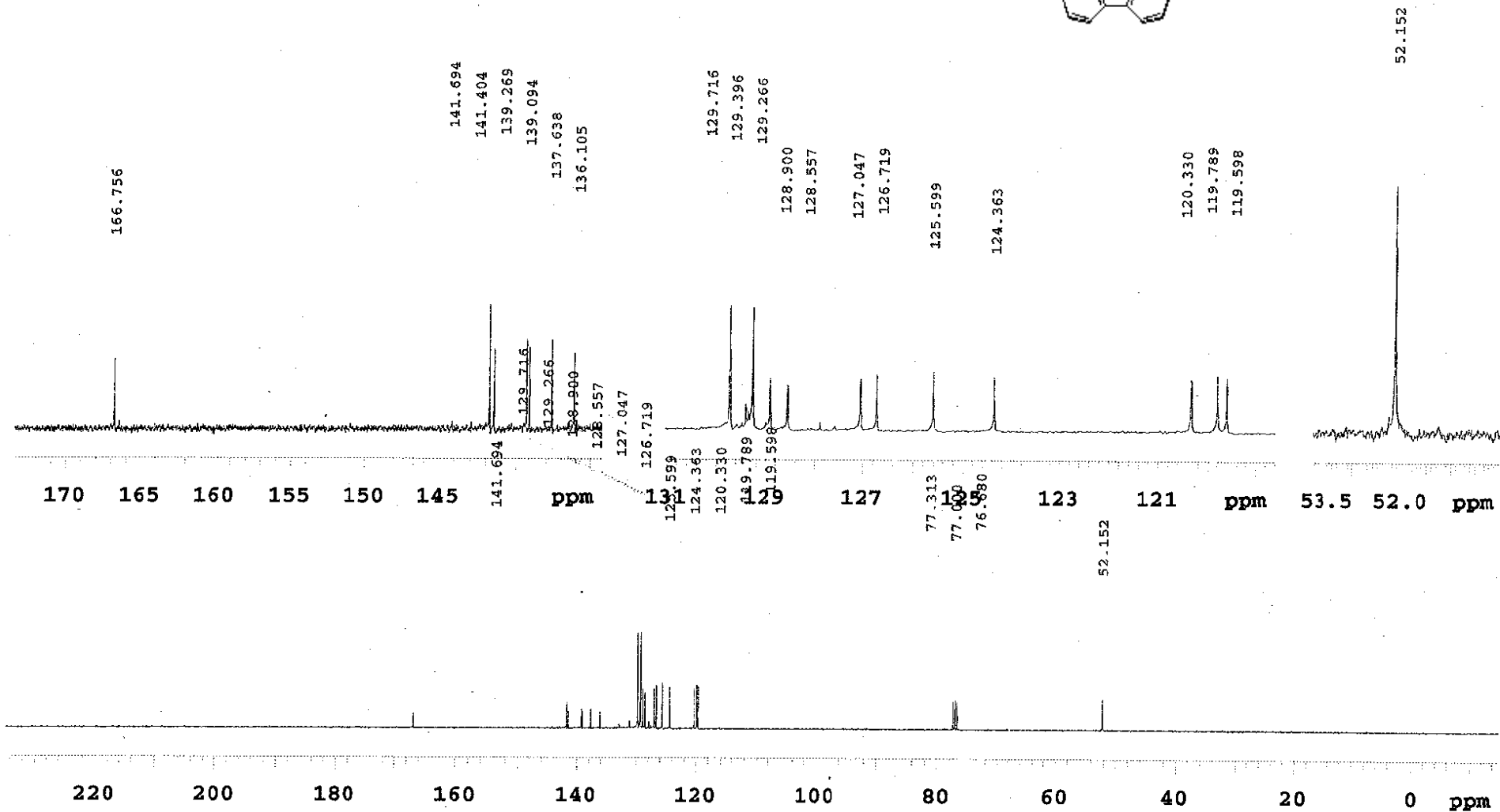
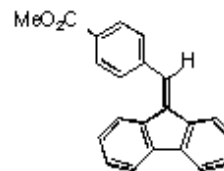
OH/2

7.2H2

2.11 2.19 2.22
1.18 1.37 1.08

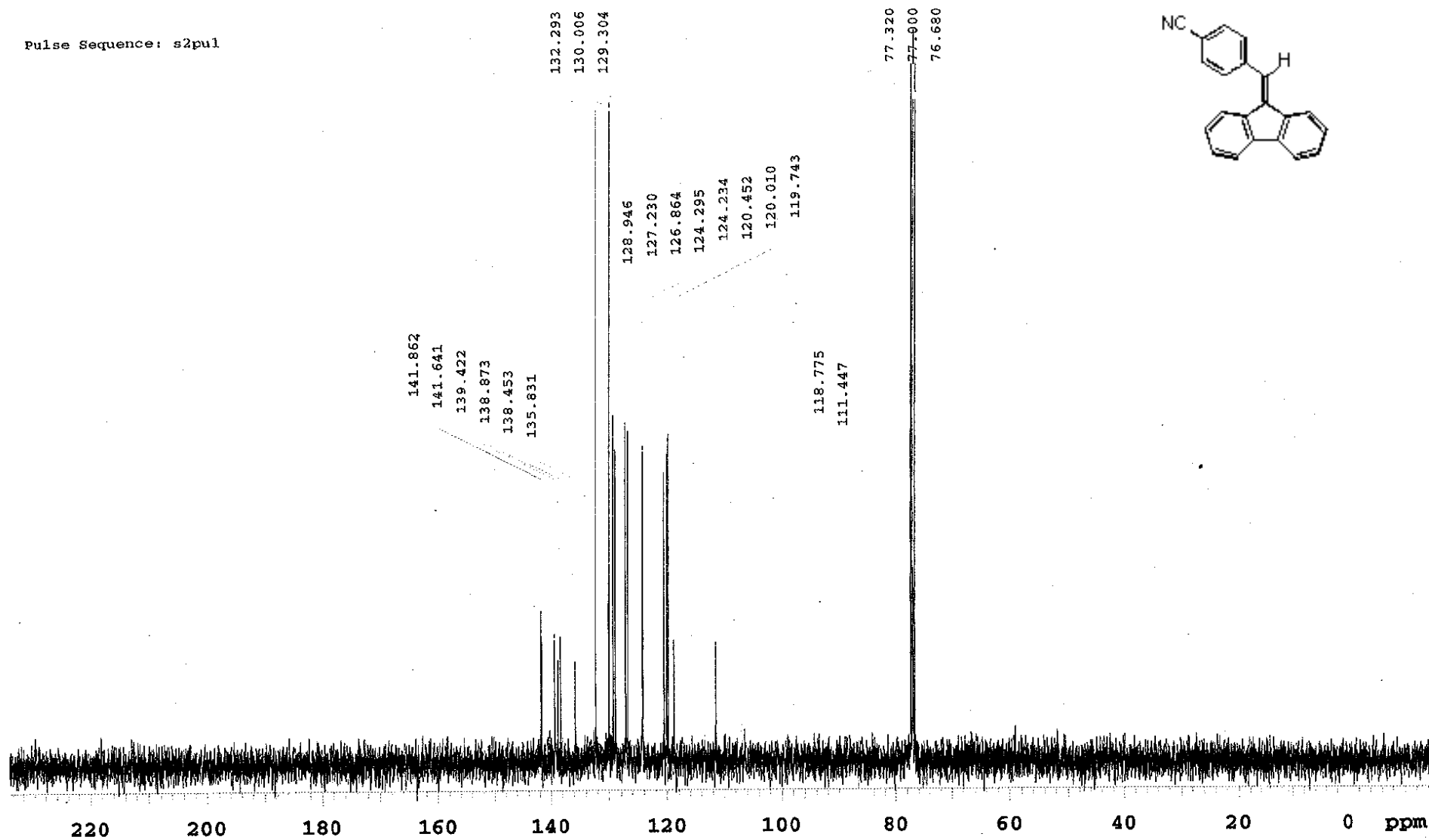
13C OBSERVE

Pulse Sequence: s2pul



13C OBSERVE

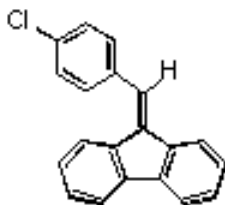
Pulse Sequence: s2pul



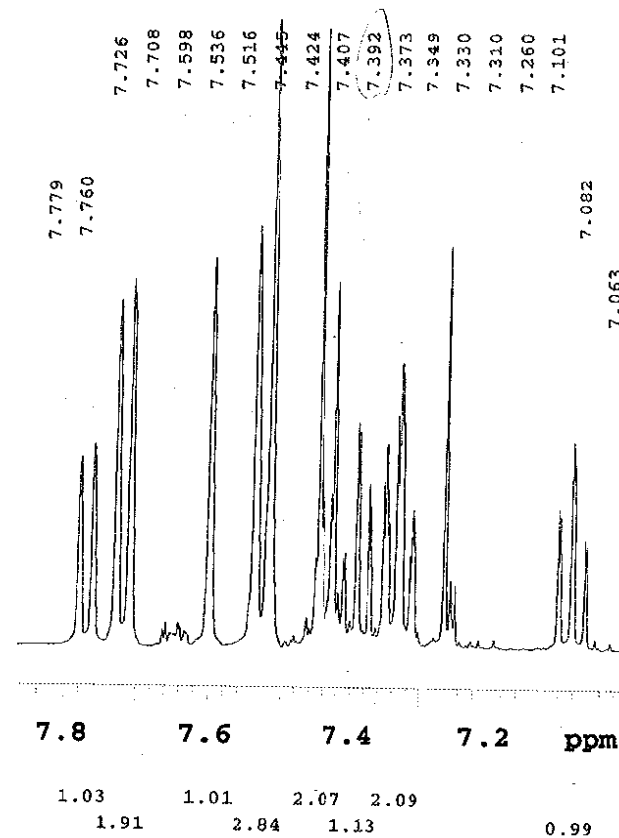
STANDARD01H108ERRURd

Pulse Sequence: s2pul

Pulse Sequence: s2pul



7.598
7.536
7.516
7.445
7.260

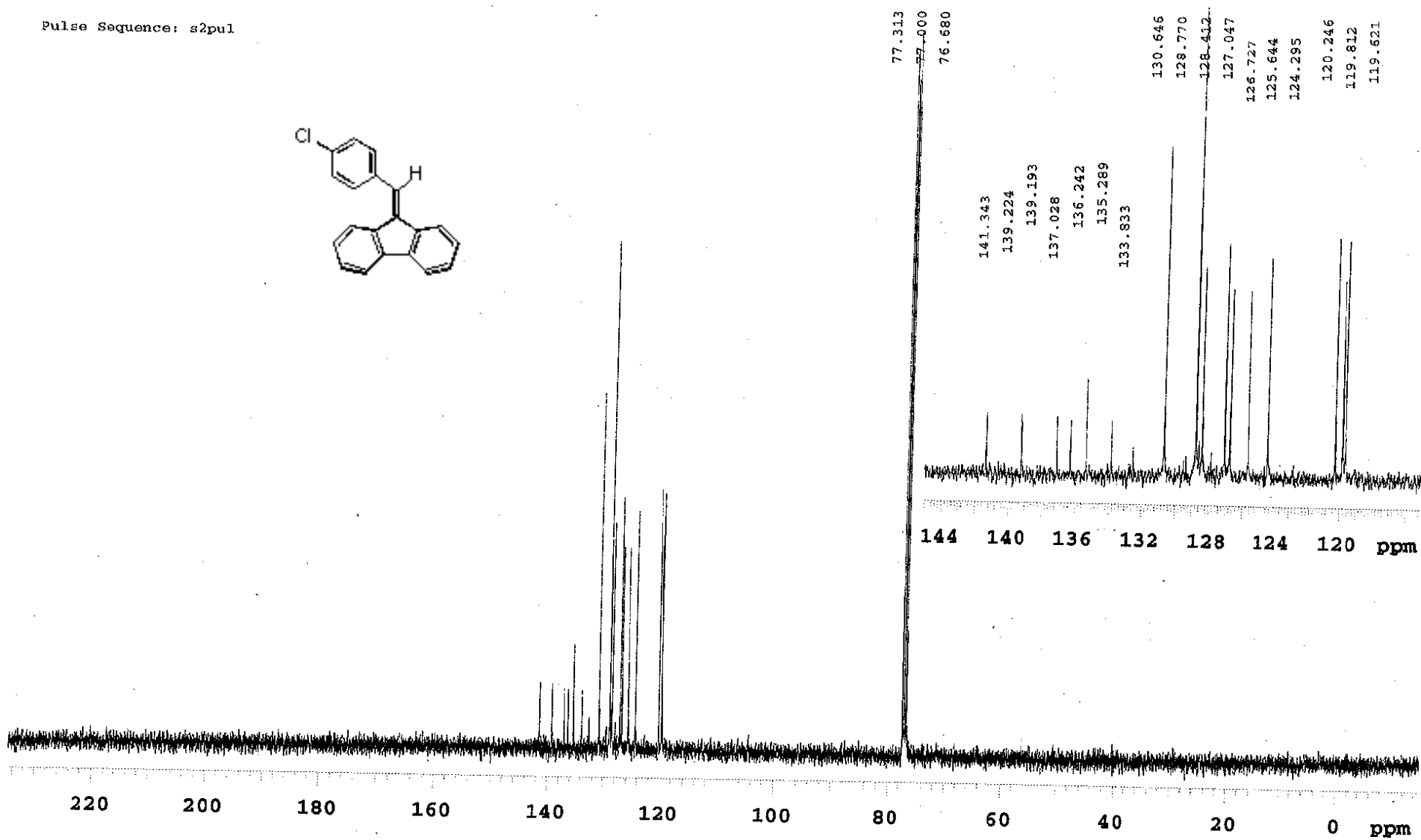
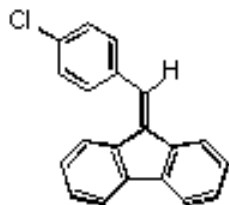


13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm

1.0302010
1.92853.00

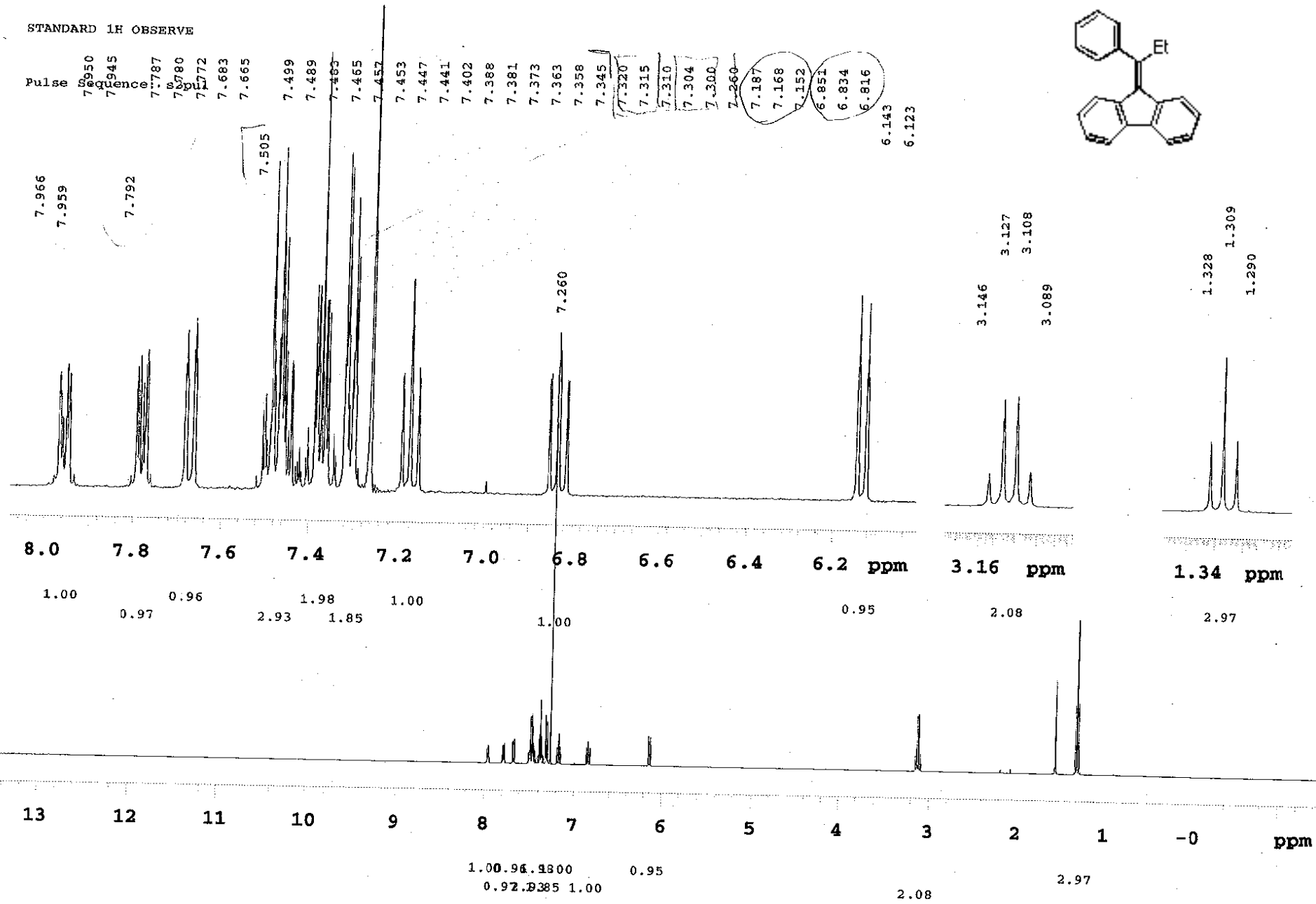
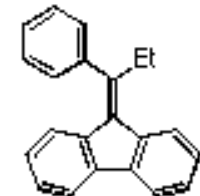
¹³C OBSERVE

Pulse Sequence: s2pu1



STANDARD 1H OBSERVE

Pulse 78950
 sequence 78945
 78787
 78780
 78772
 78683
 78665



13C OBSERVE

Pulse Sequence: s2pul

