Electronic Supplementary Material (ESI) for RSC Advances.

Synthesis of Biologically Active Pyridoimidazole/Imidazobenzothiazole Annulated Polyheterocycles using Cyanuric Chloride in Water

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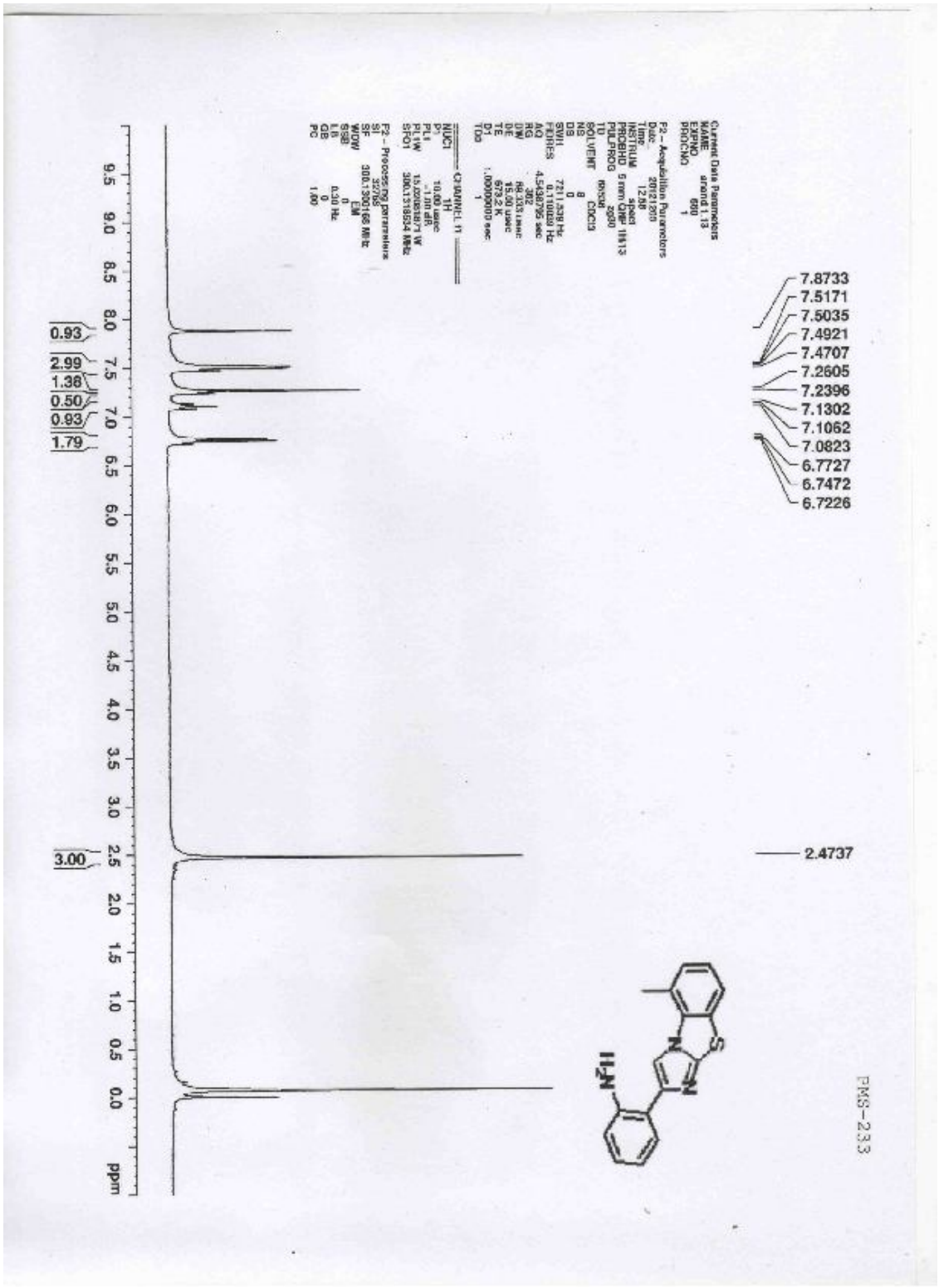
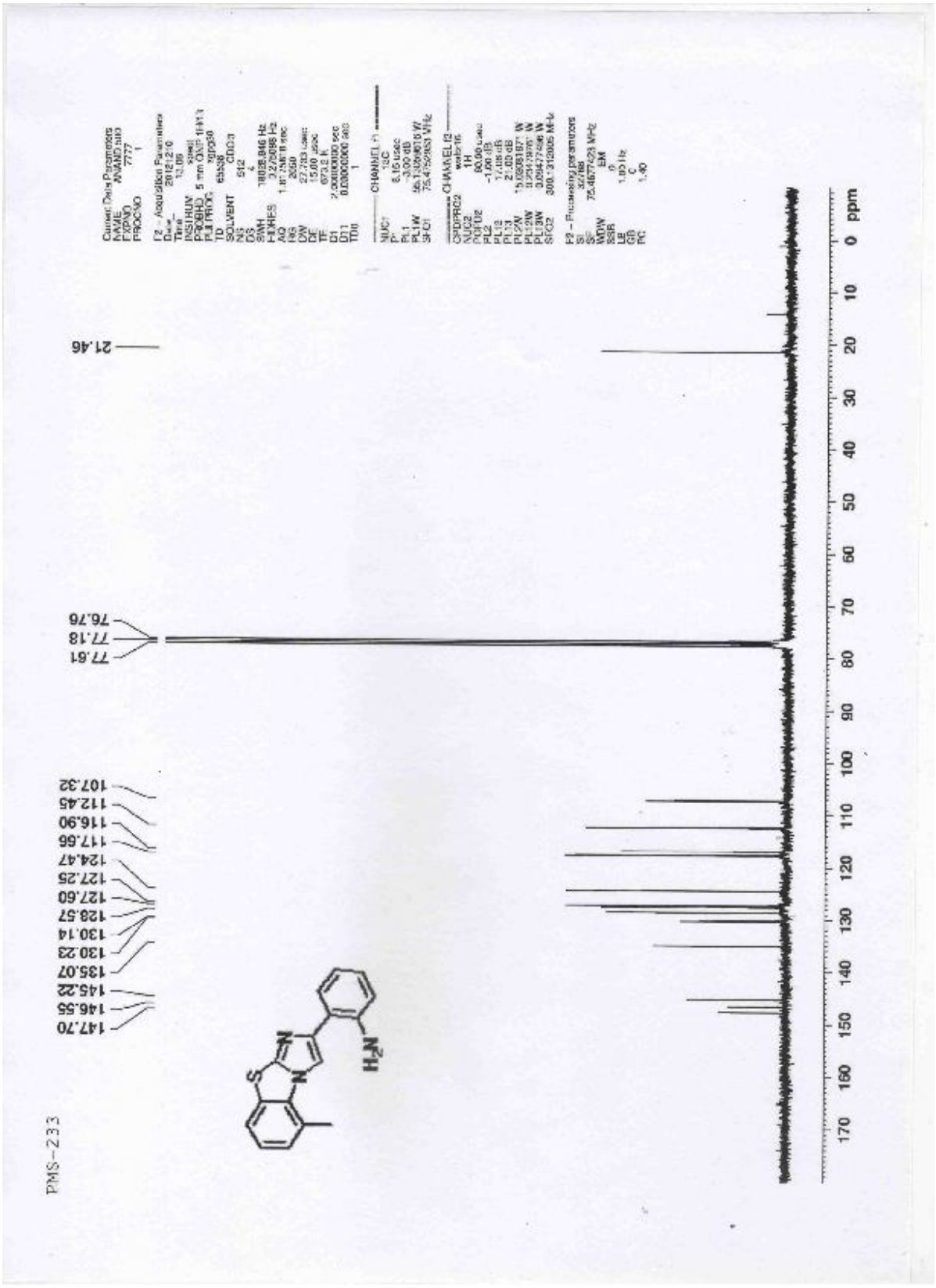
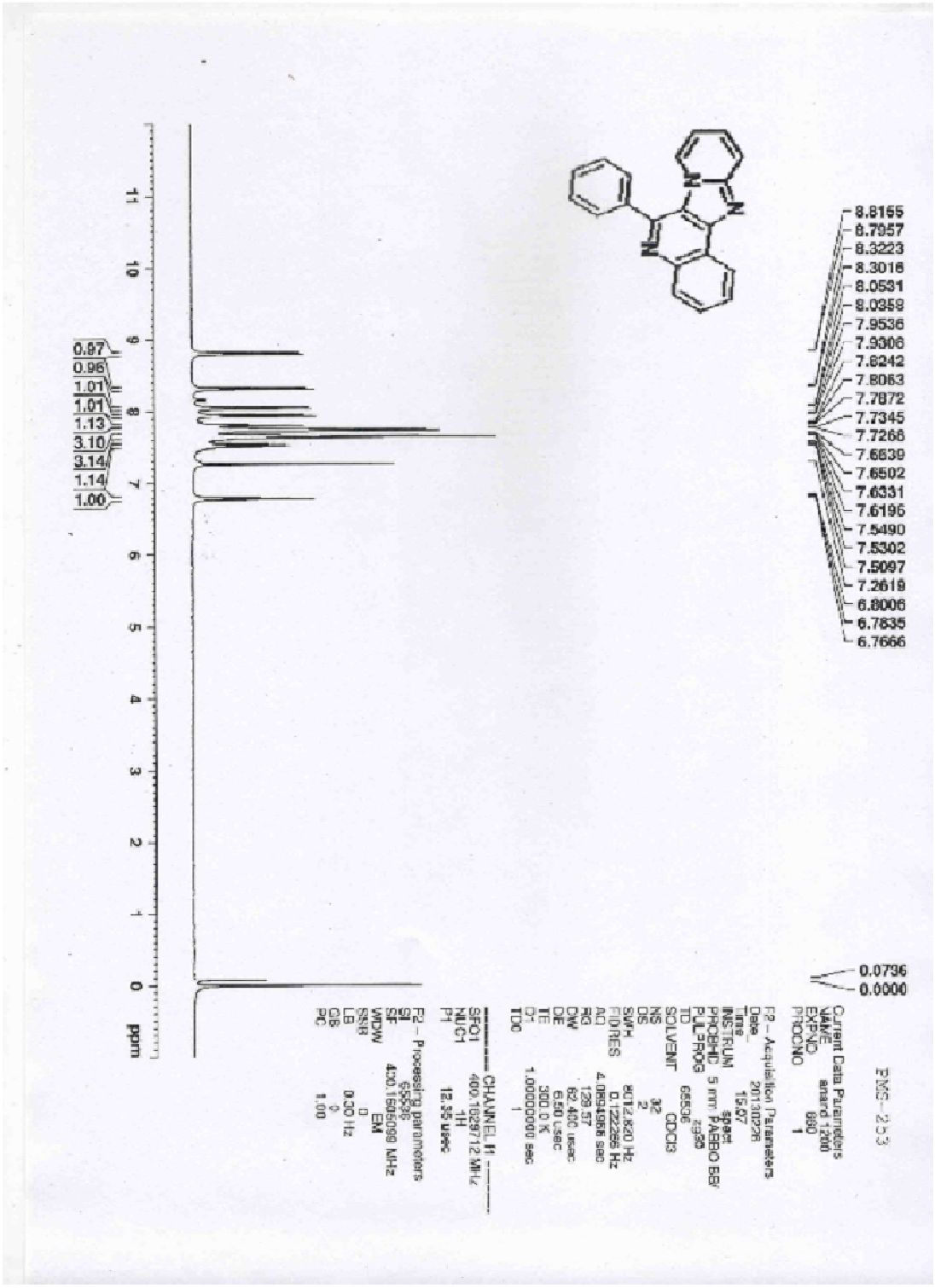
2-(3-methyl-imidazo[2,1-b]-benzothiazol-6-yl)-phenylamine (20):

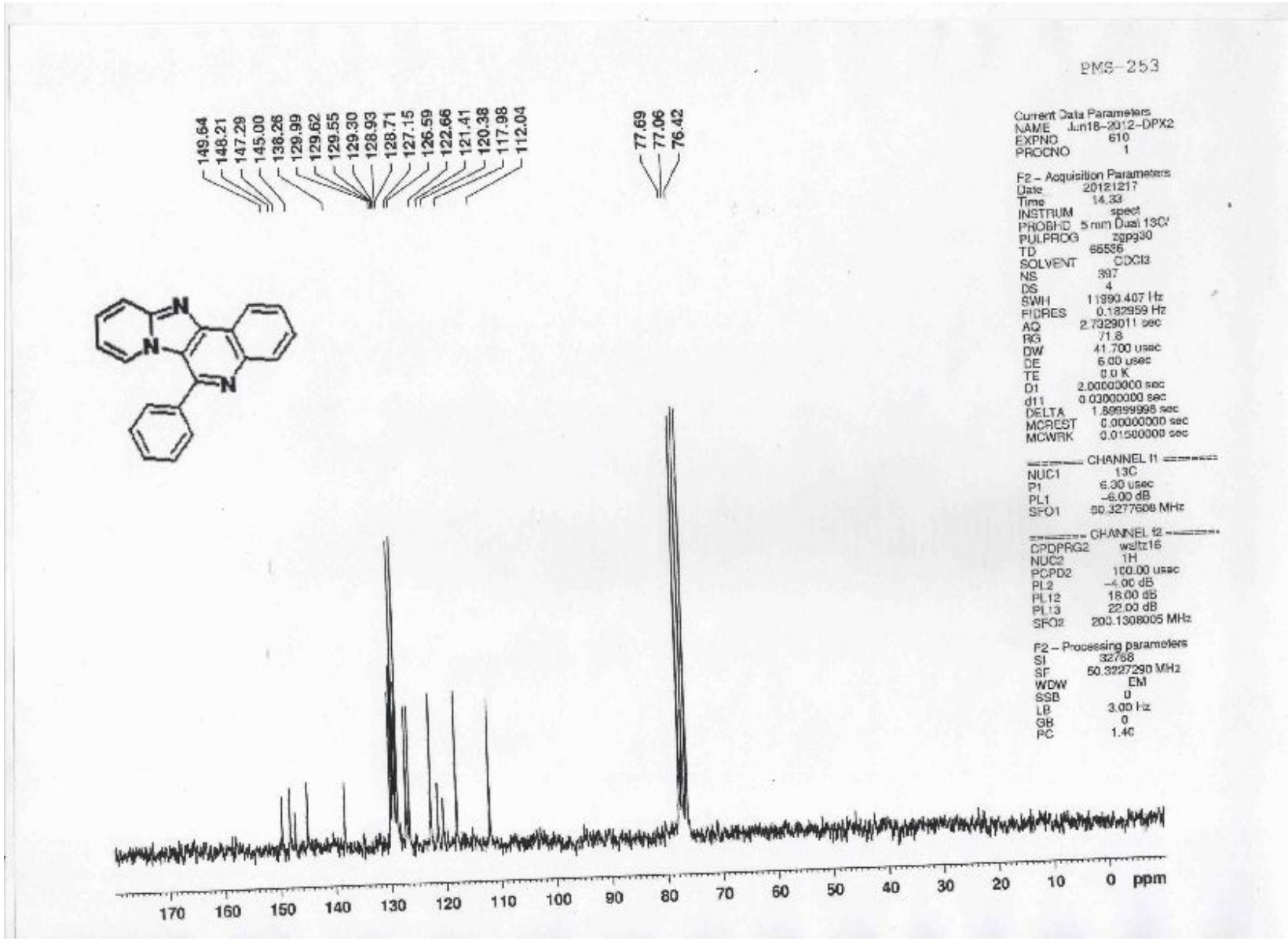



PMS-233
147.70
146.55
145.22
135.07
130.23
130.14
128.57
127.60
127.25
124.47
117.66
116.90
112.45
107.32
77.61
77.18
76.76
21.46
0 ppm

6-Phenyl-5,6b,11-triaza-benzo[a]fluorene (9a).



S4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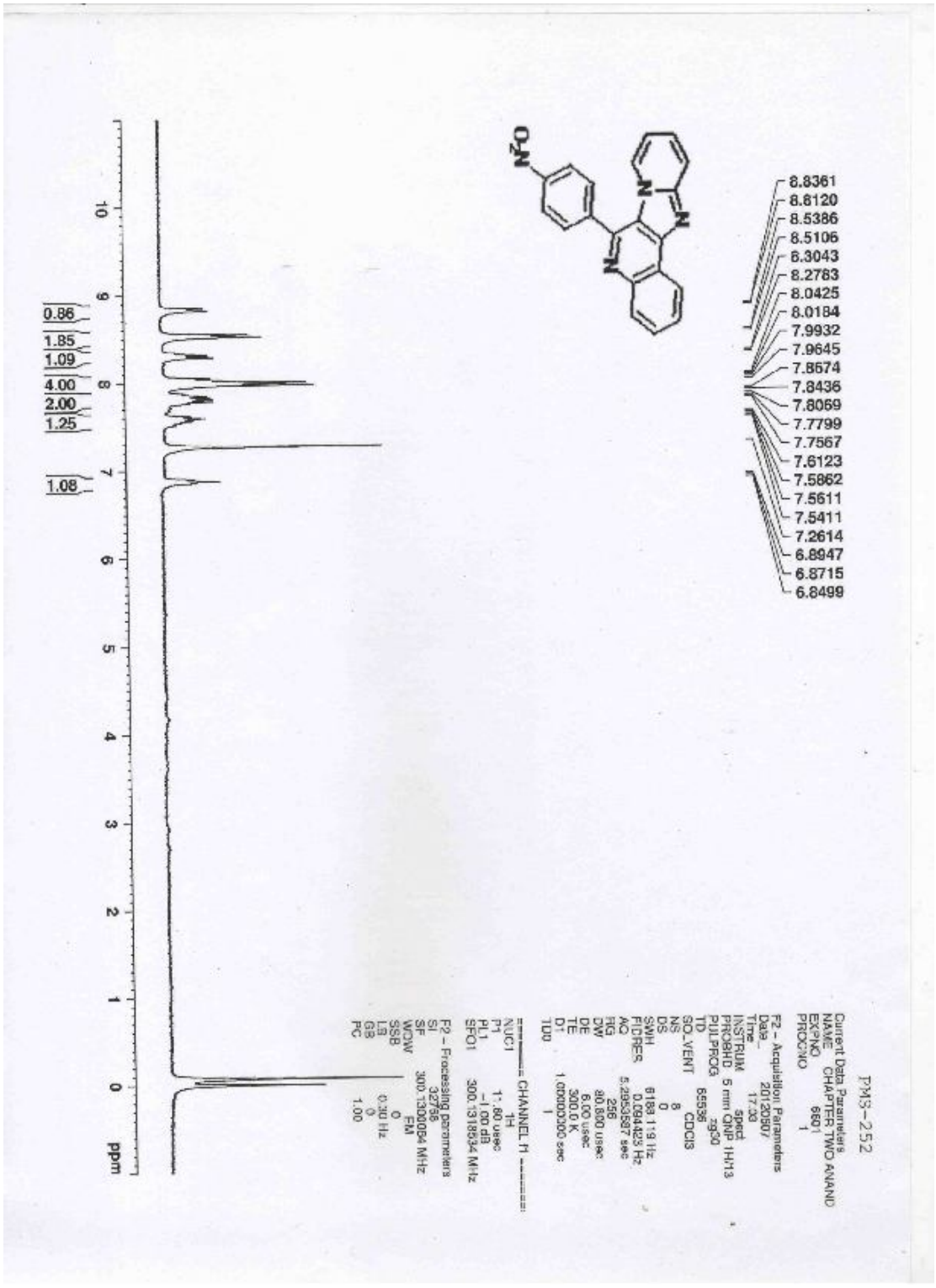
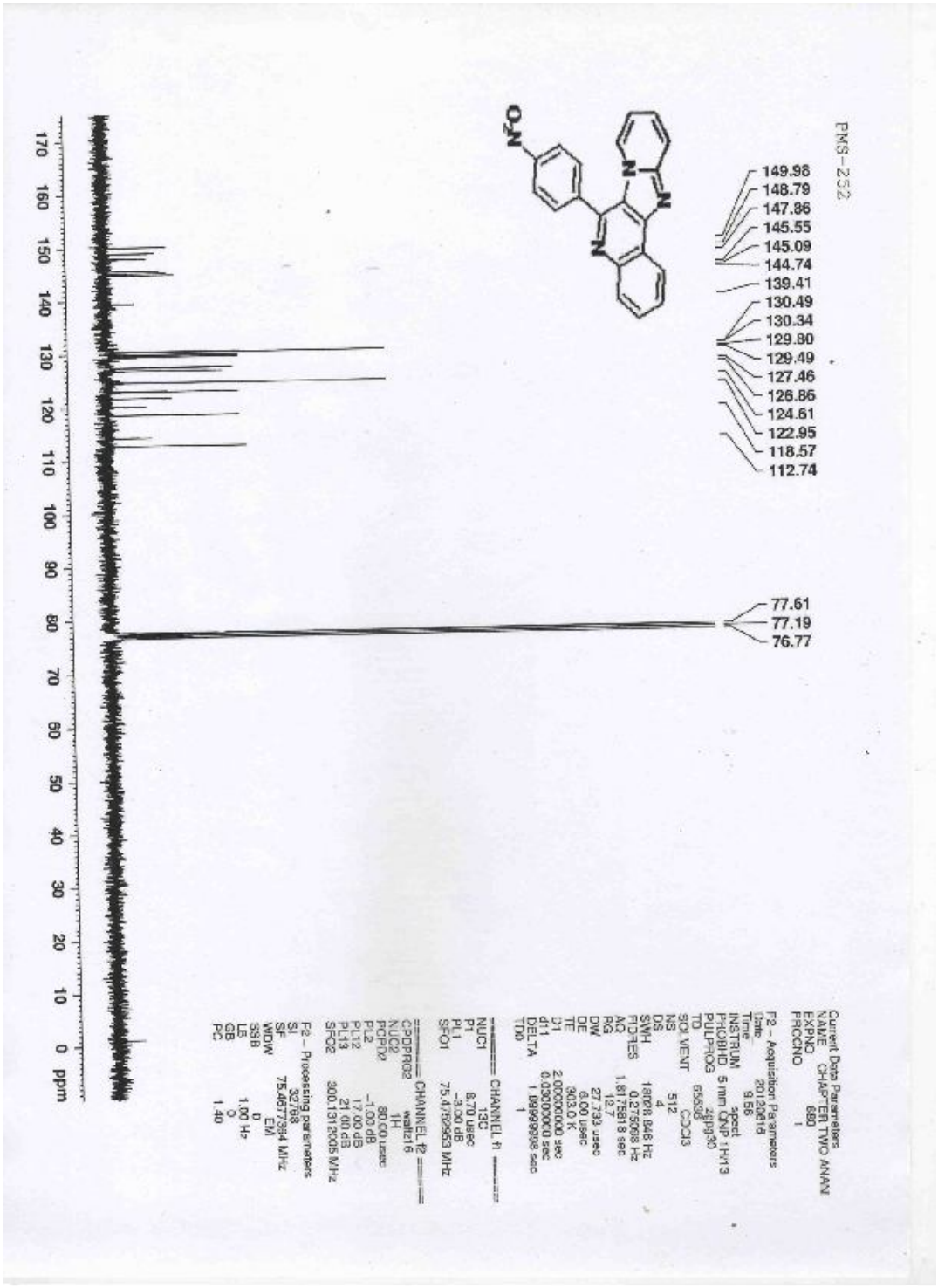
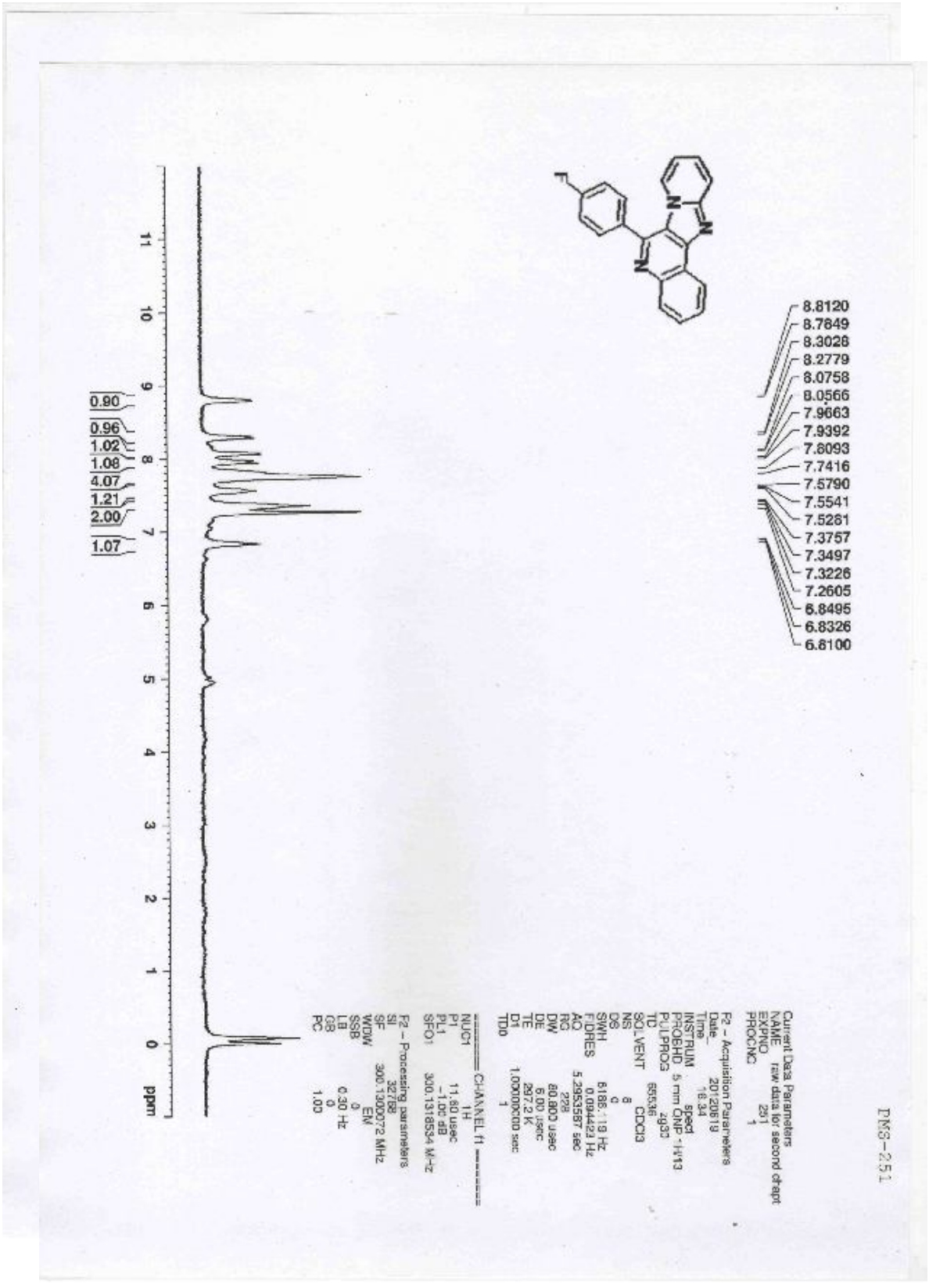
149.64
148.21
147.29
145.00
138.26
129.99
129.62
129.55
129.30
128.93
128.71
127.15
126.59
122.66
121.41
120.38
117.98
112.04
77.69
77.06
76.42
Current Data Parameters
NAME Jun18-2012-DPX2
EXPNO 610
PROCNO 1
F2 - Acquisition Parameters
Date 20121217
Time 14.33
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 397
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7929011 sec
RG 71.8
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

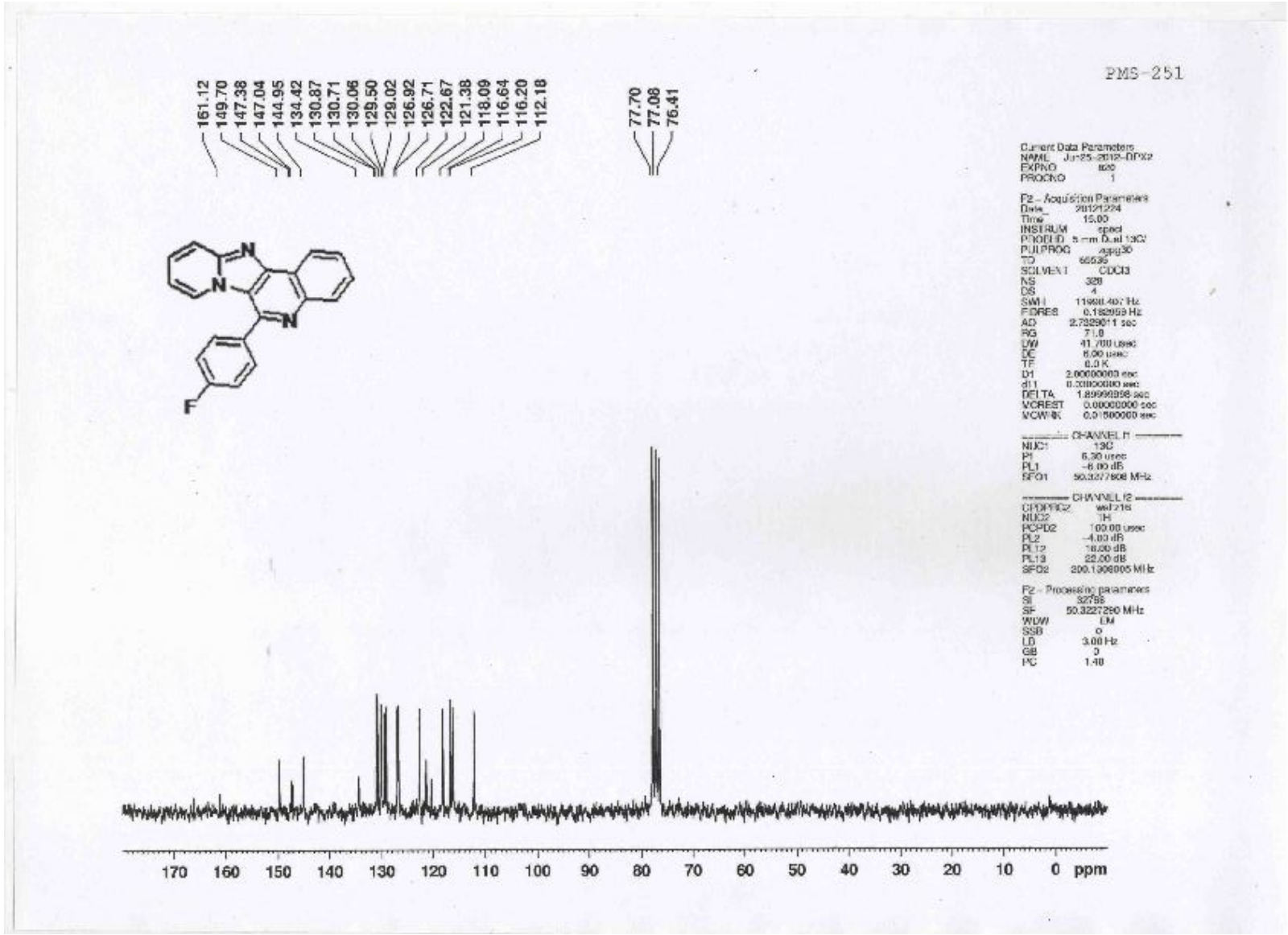
6-(4-Nitro-phenyl)-5,6b,11-triaza-benzo[a]fluorine (9b):



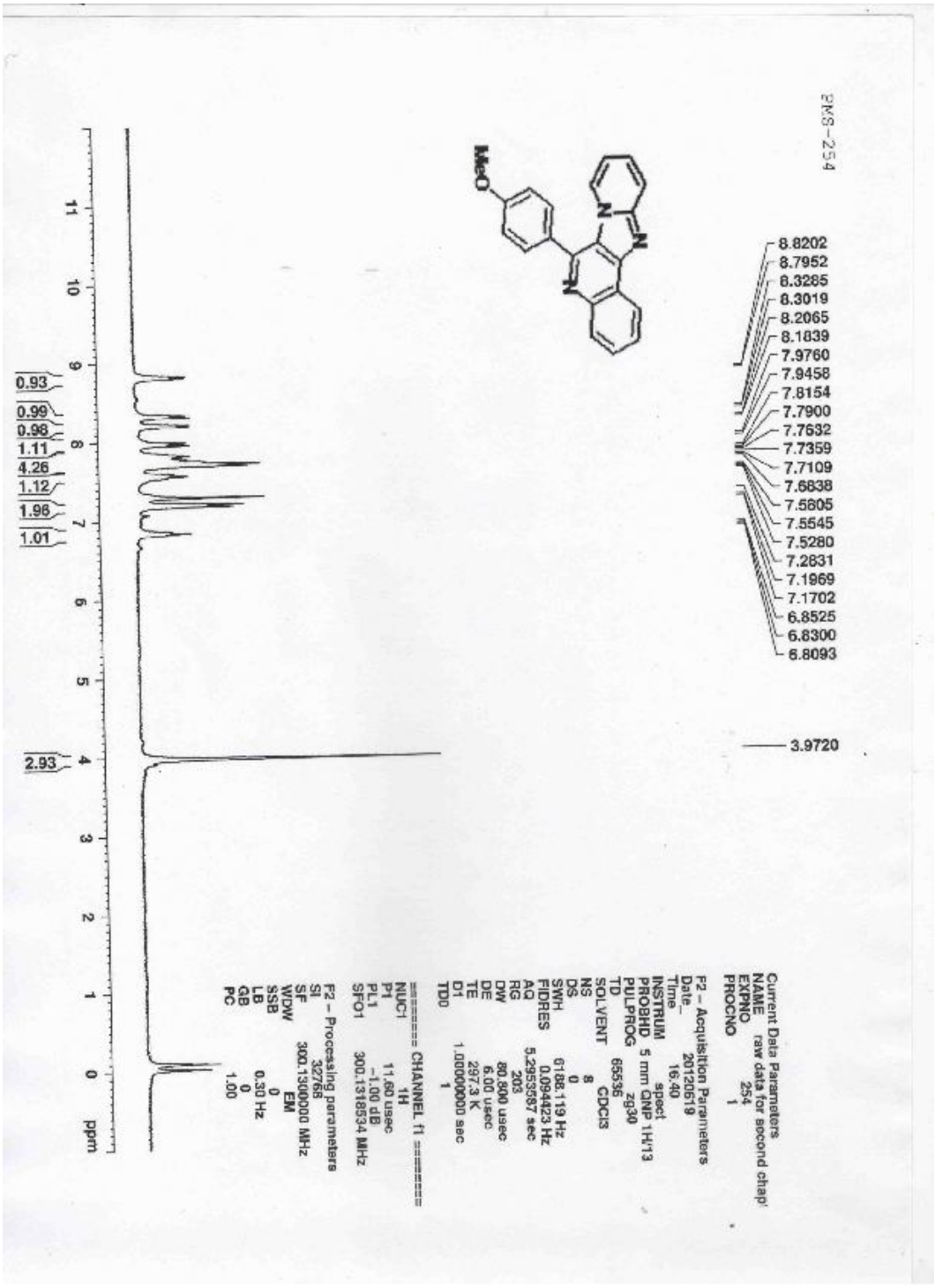

PMS-252
149.96
148.79
147.86
145.55
145.09
144.74
139.41
130.49
130.34
129.80
129.49
127.46
126.86
124.61
122.95
118.57
112.74
77.61
77.19
76.77
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME CHAPTER TWO ANAN
EXPNO 680
PROCNO 1
F2 - Acquisition Parameters
Date 20120618
Time 9.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275068 Hz
AQ 1.8175818 sec
RG 12.7
DW 27.733 usec
DE 6.00 usec
TE 303.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4578353 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4577354 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

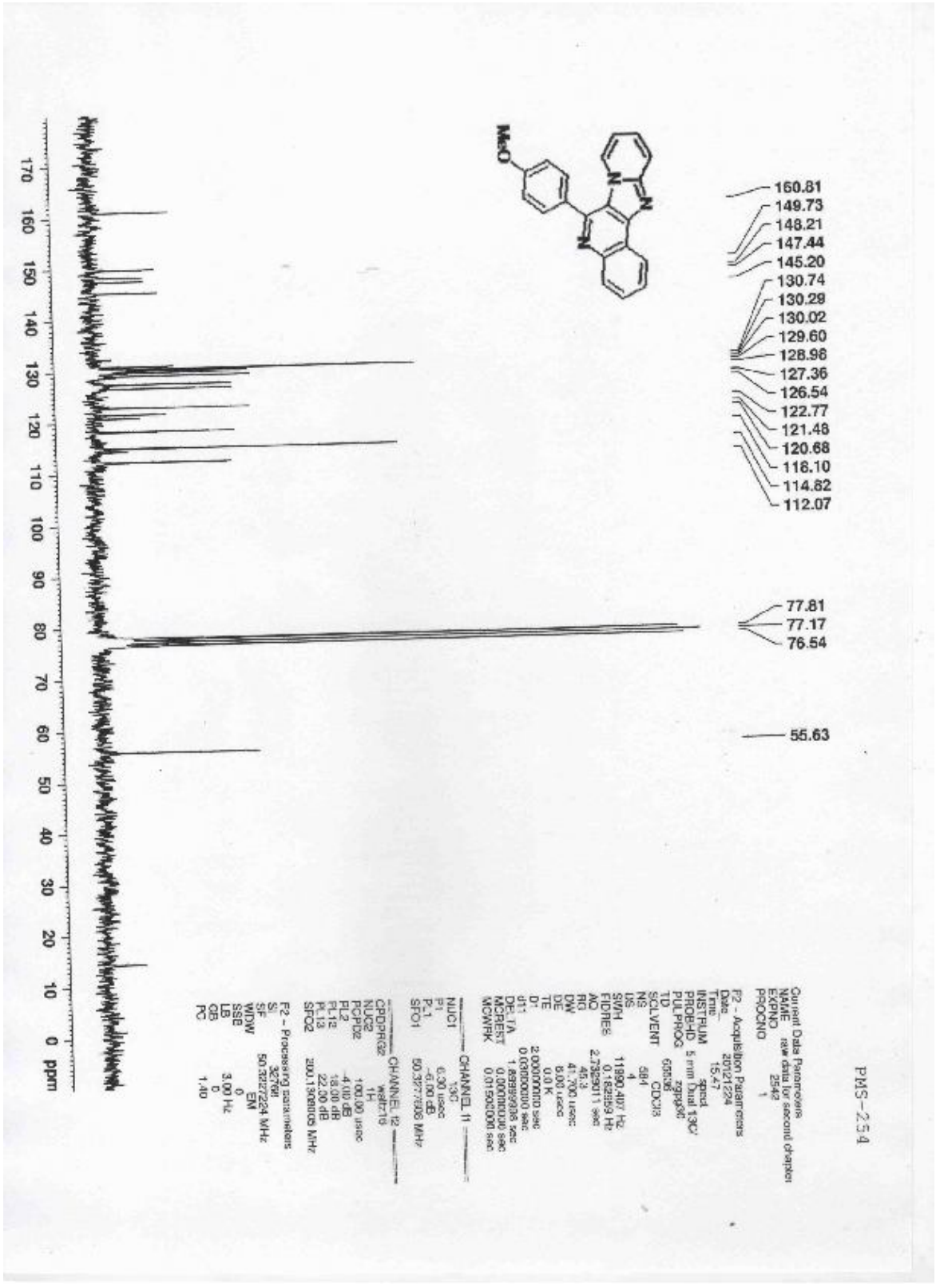
6-(4-Flouro-phenyl)-5,6b,11-triaza-benzo[a]fluorene (9c):



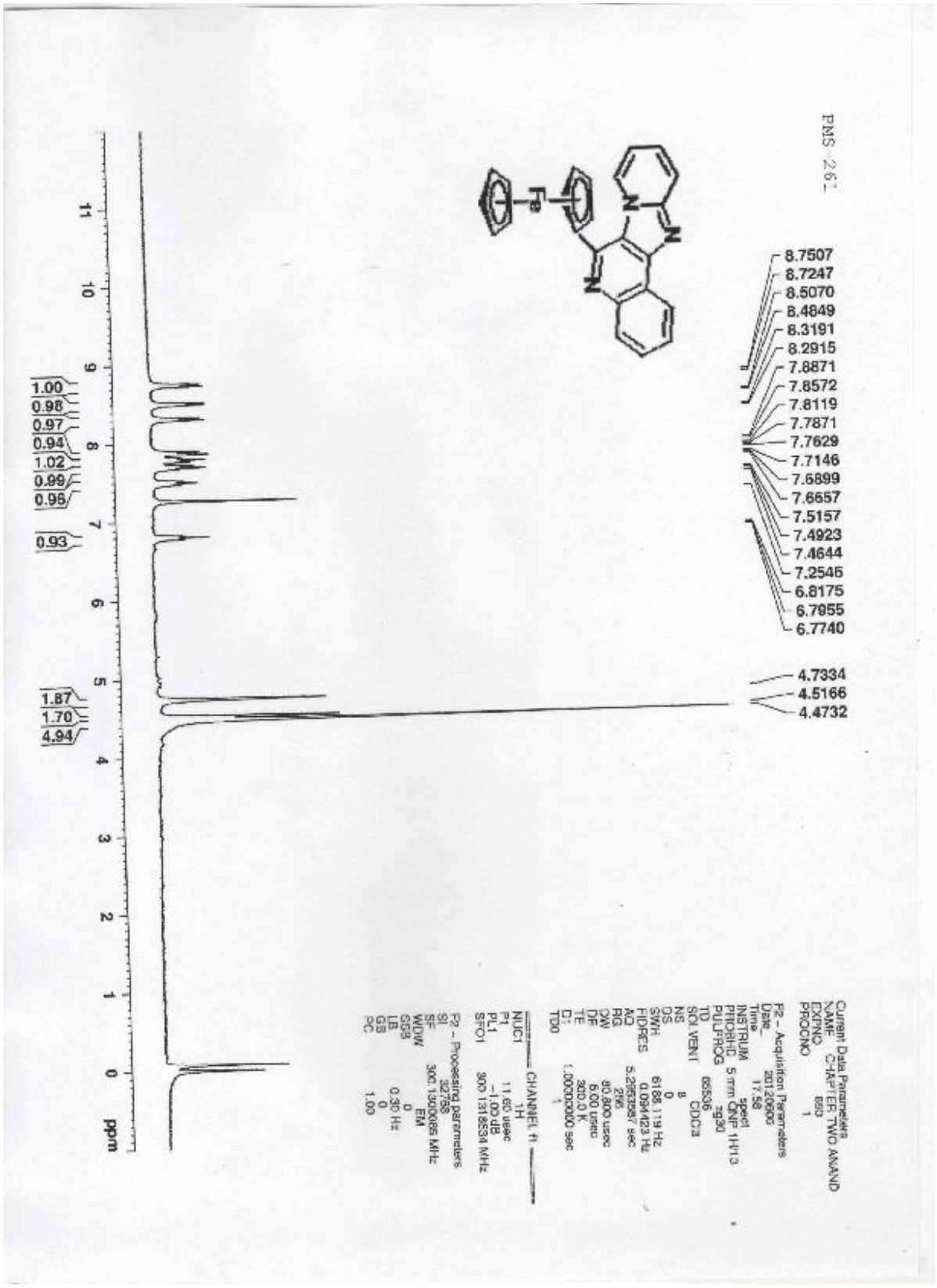

PMS-251
161.12
149.70
147.38
147.04
144.95
134.42
130.87
130.71
130.06
129.50
129.02
126.92
126.71
122.67
121.38
118.09
116.64
116.20
112.18
77.70
77.08
76.41
N
N
N
F
Current Data Parameters
NAME Jun25-2012-DPX2
EXPNO 820
PROCNO 1
F2 - Acquisition Parameters
Date 20121224
Time 15.00
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 328
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.0
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
VCREST 0.00000000 sec
VCWIN 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277808 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 16.00 dB
PL13 22.00 dB
SFO2 200.1300005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227280 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

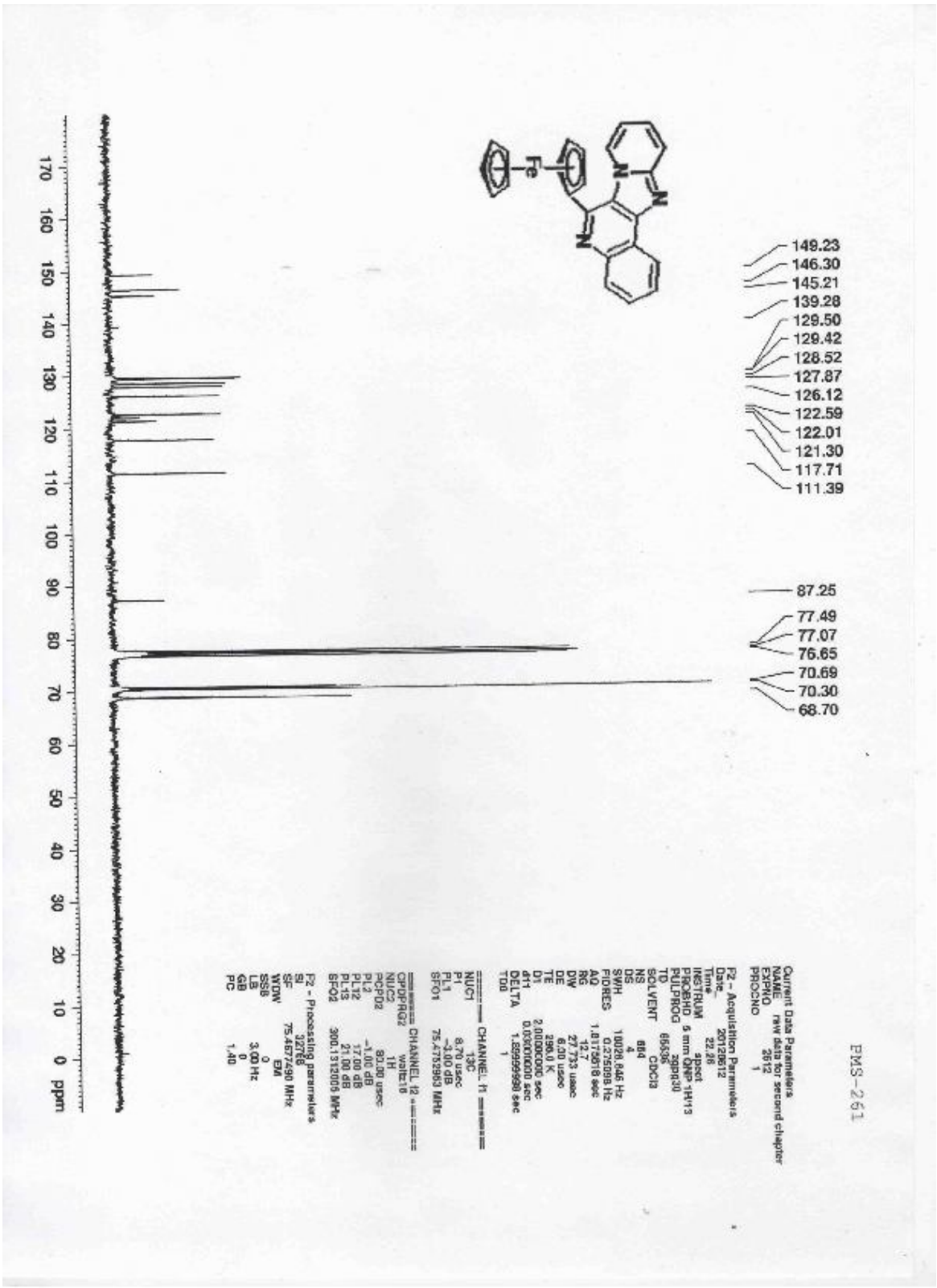
6-(4-Methoxy-phenyl)-5,6b,11-triaza-benzo[a]fluorene (9d):




PMS-254
MeO
160.81
149.73
148.21
147.44
145.20
130.74
130.29
130.02
129.60
128.96
127.36
126.54
122.77
121.48
120.68
116.10
114.82
112.07
77.81
77.17
76.54
55.63
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

6-(Ferrocenyl)-5,6b,11-triaza-benzo[a]fluorene (9e):




EMS-261
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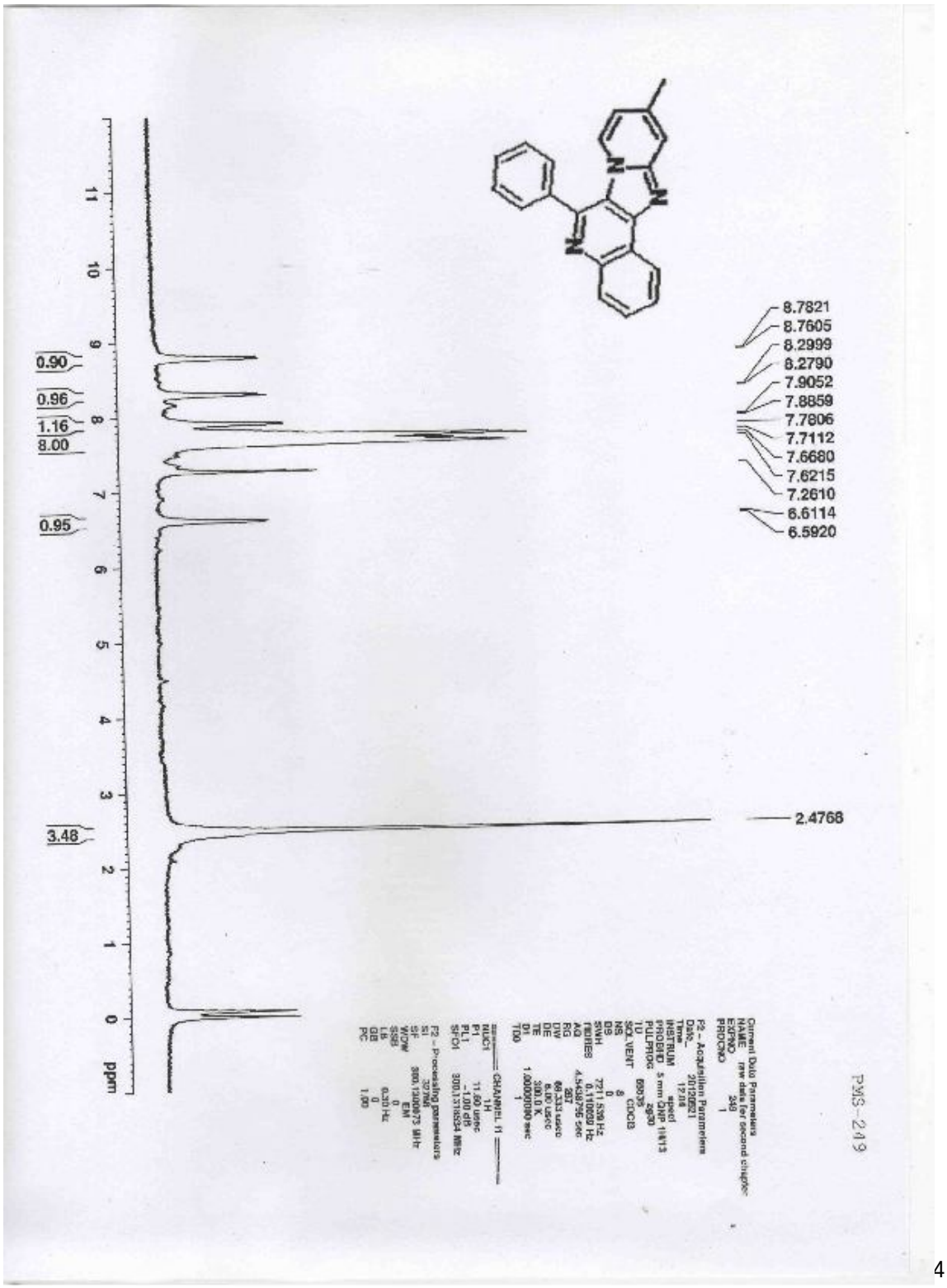
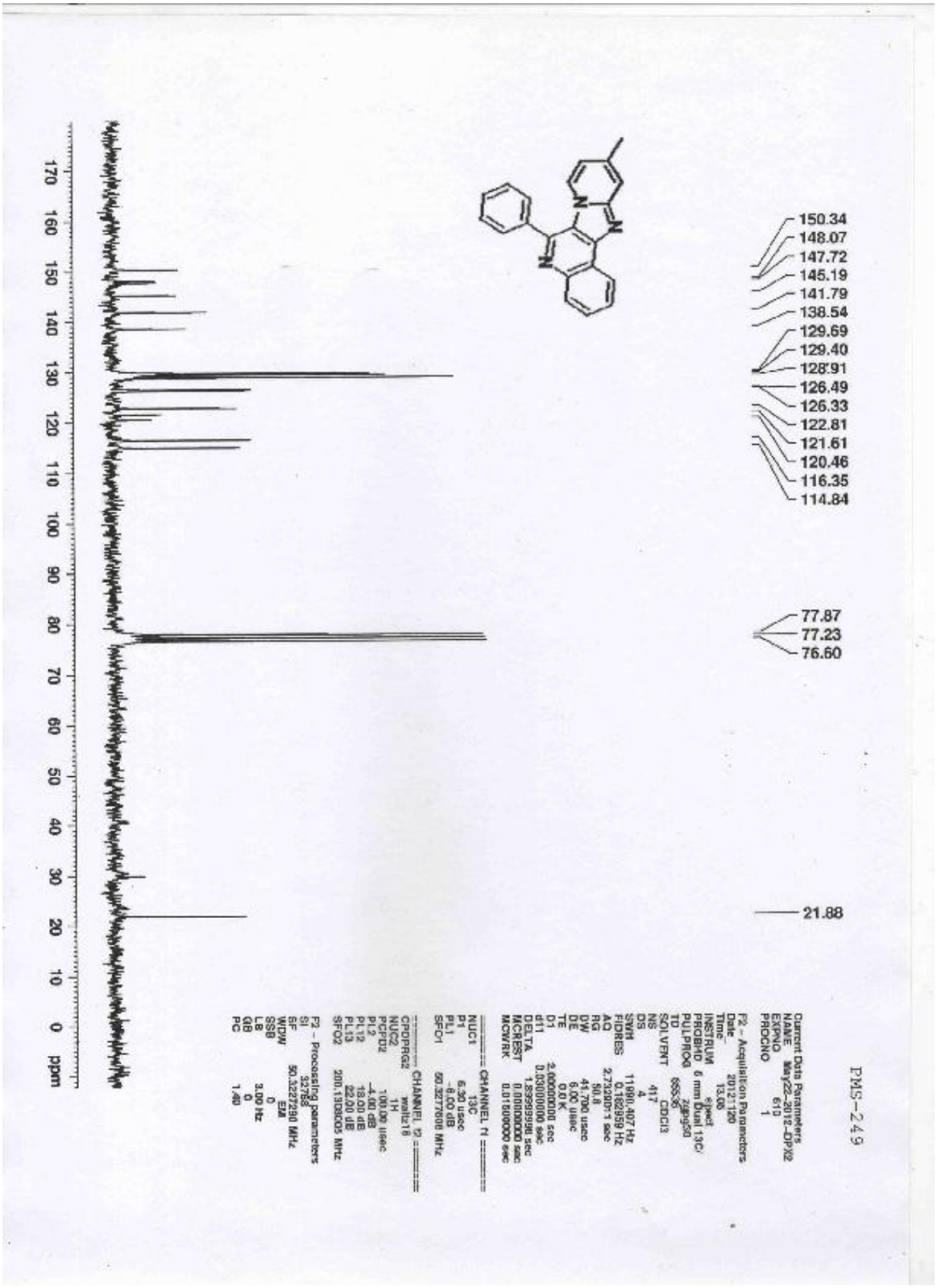
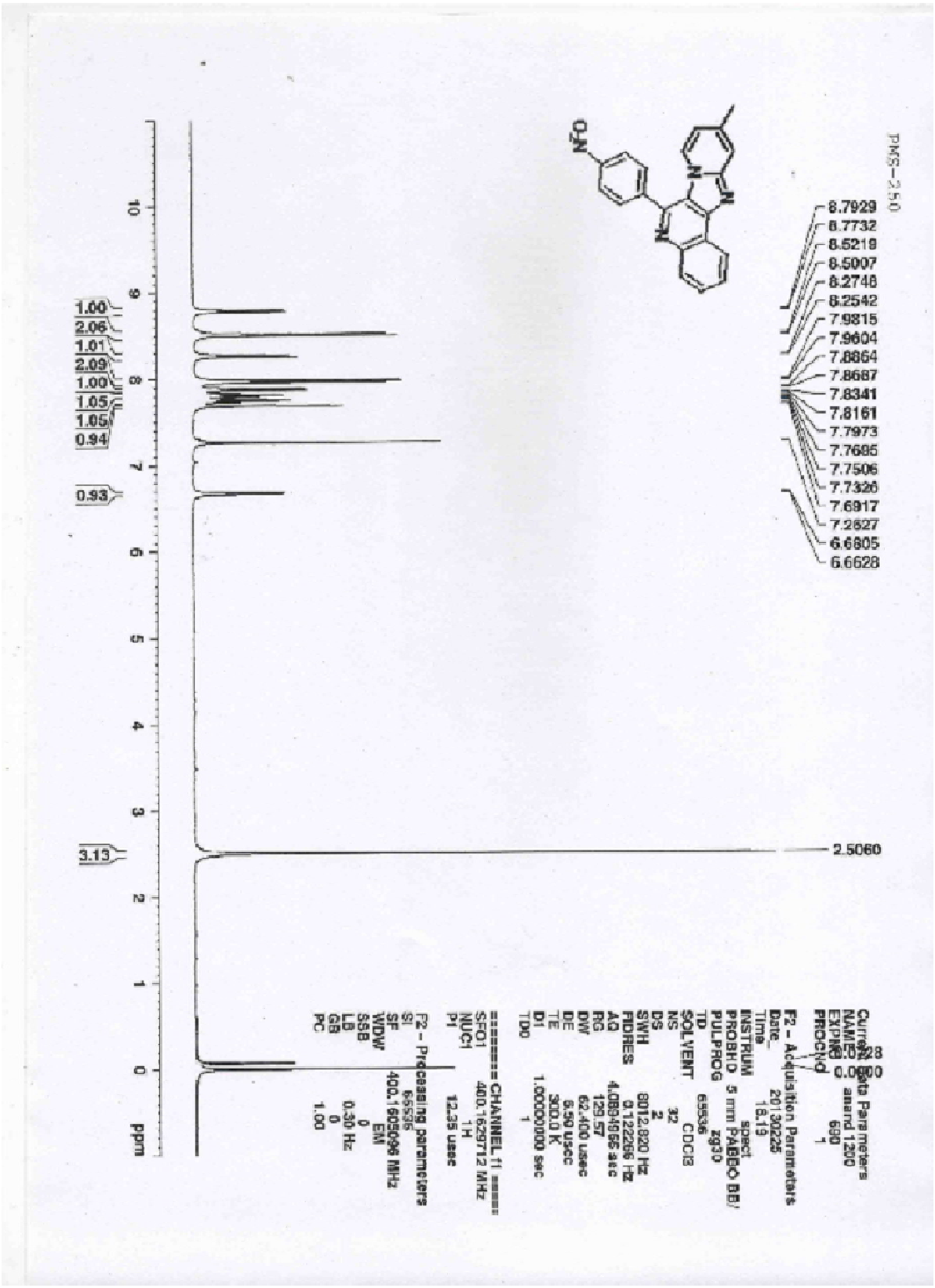
NAME raw data for second chapter
EXPNO 2612
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120612
Time 22.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 884
DS 4
SWH 10028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175618 sec
RG 12.7
DW 27.733 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
149.23
146.30
145.21
139.28
129.50
129.42
128.52
127.87
126.12
122.59
122.01
121.30
117.71
111.39
87.25
77.49
77.07
76.65
70.69
70.30
68.70
Fe
N
N
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

6-(Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9f):

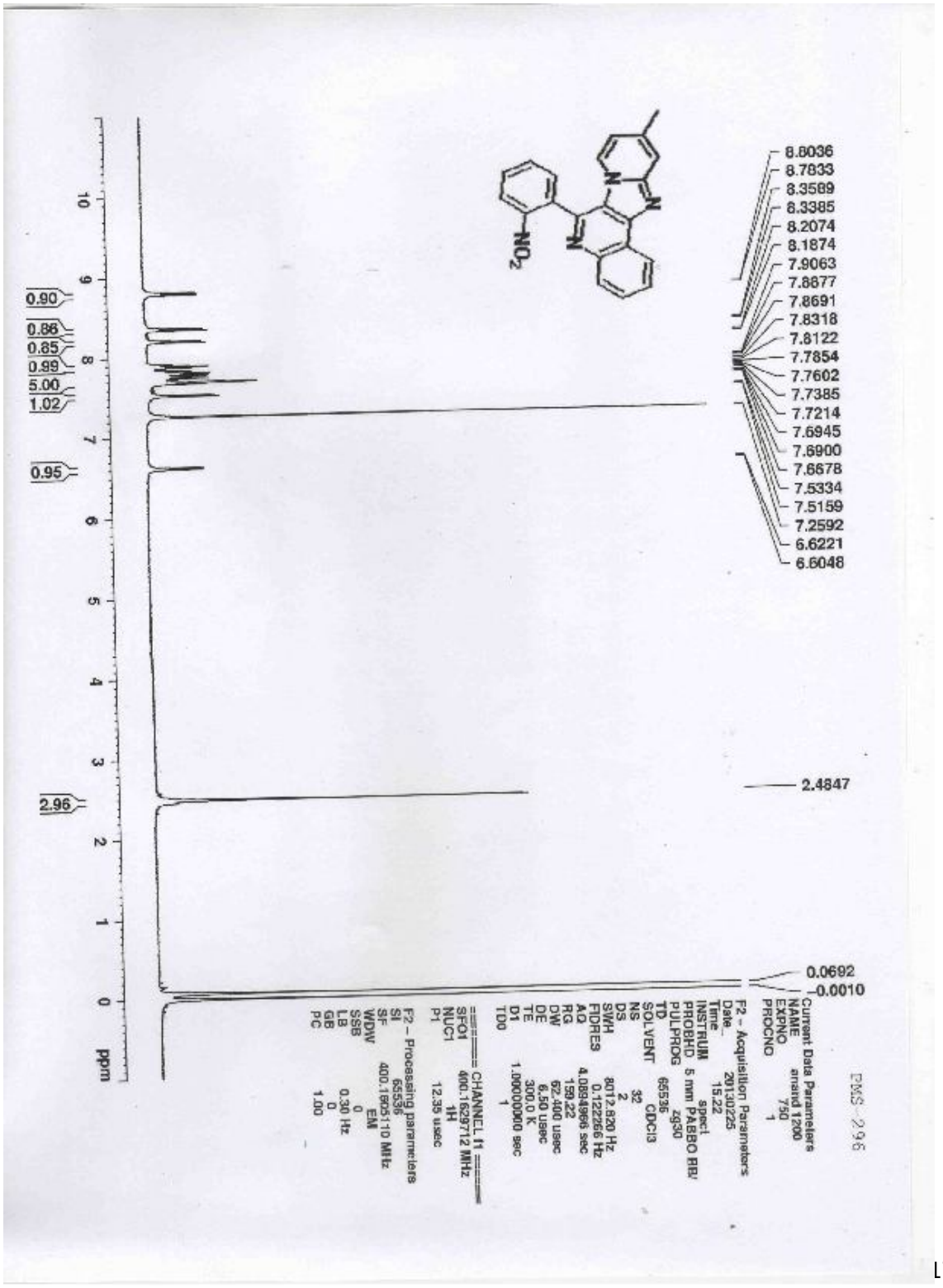



PMS-249
150.34
148.07
147.72
145.19
141.79
138.54
129.69
129.40
128.91
126.49
126.33
122.81
121.61
120.46
116.35
114.84
77.87
77.23
76.60
21.88
Current Data Parameters
NAME May22_2012_DPX2
EXPNO 610
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121120
Time 13.06
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 417
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7328071 sec
RG 50.8
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.83999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277808 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

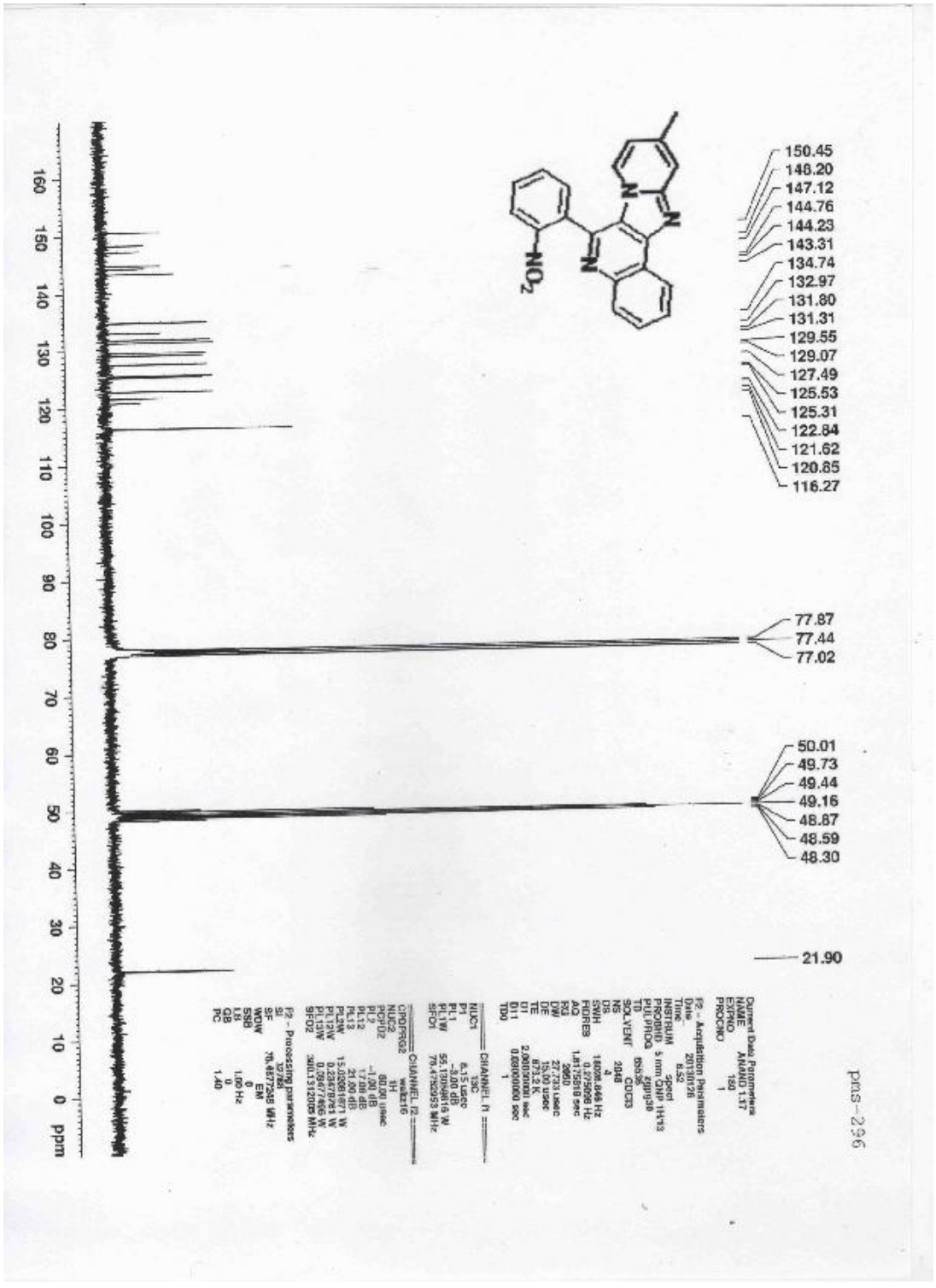
6-(4-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9g):



6-(2-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9h):



L7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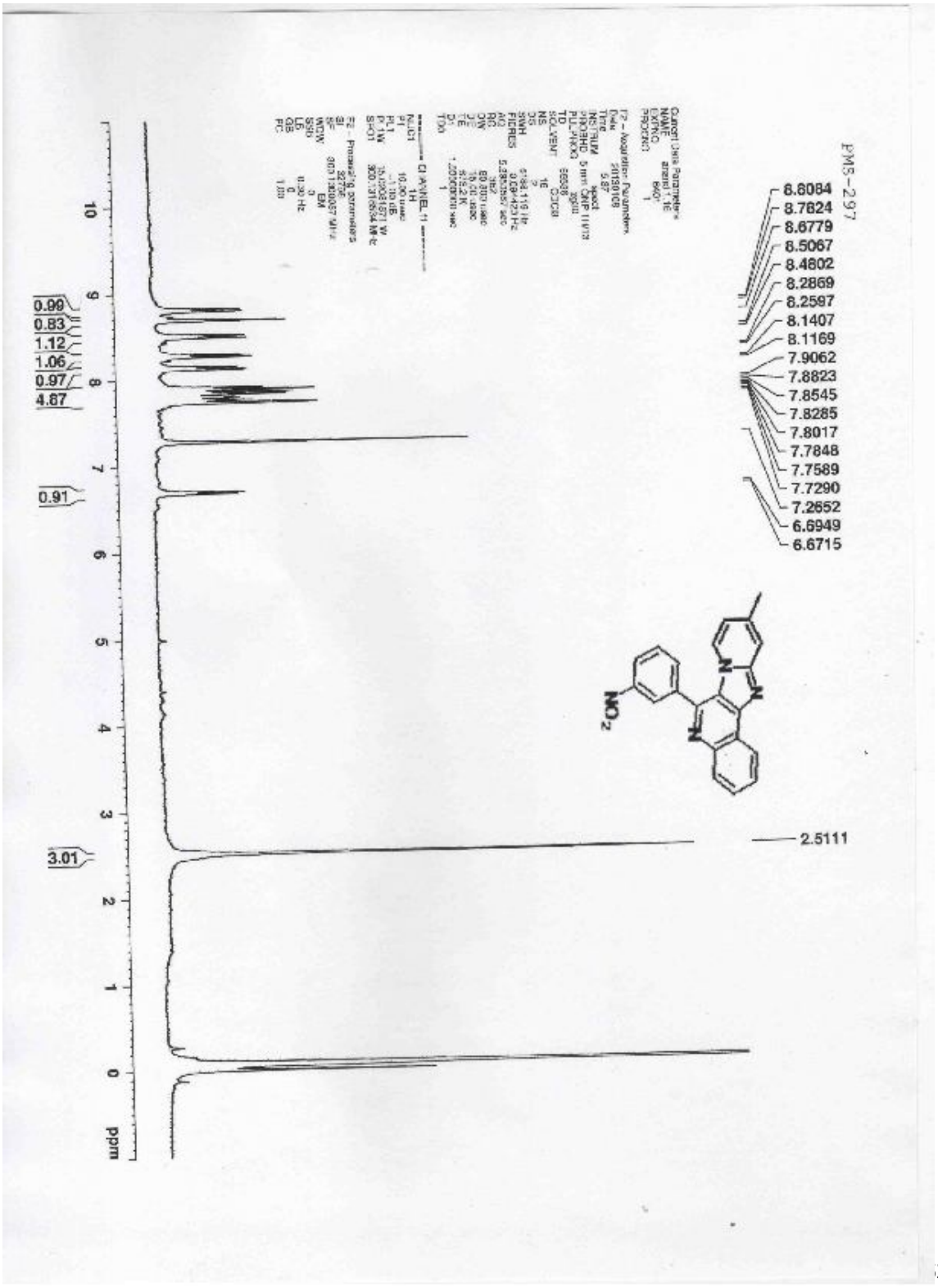
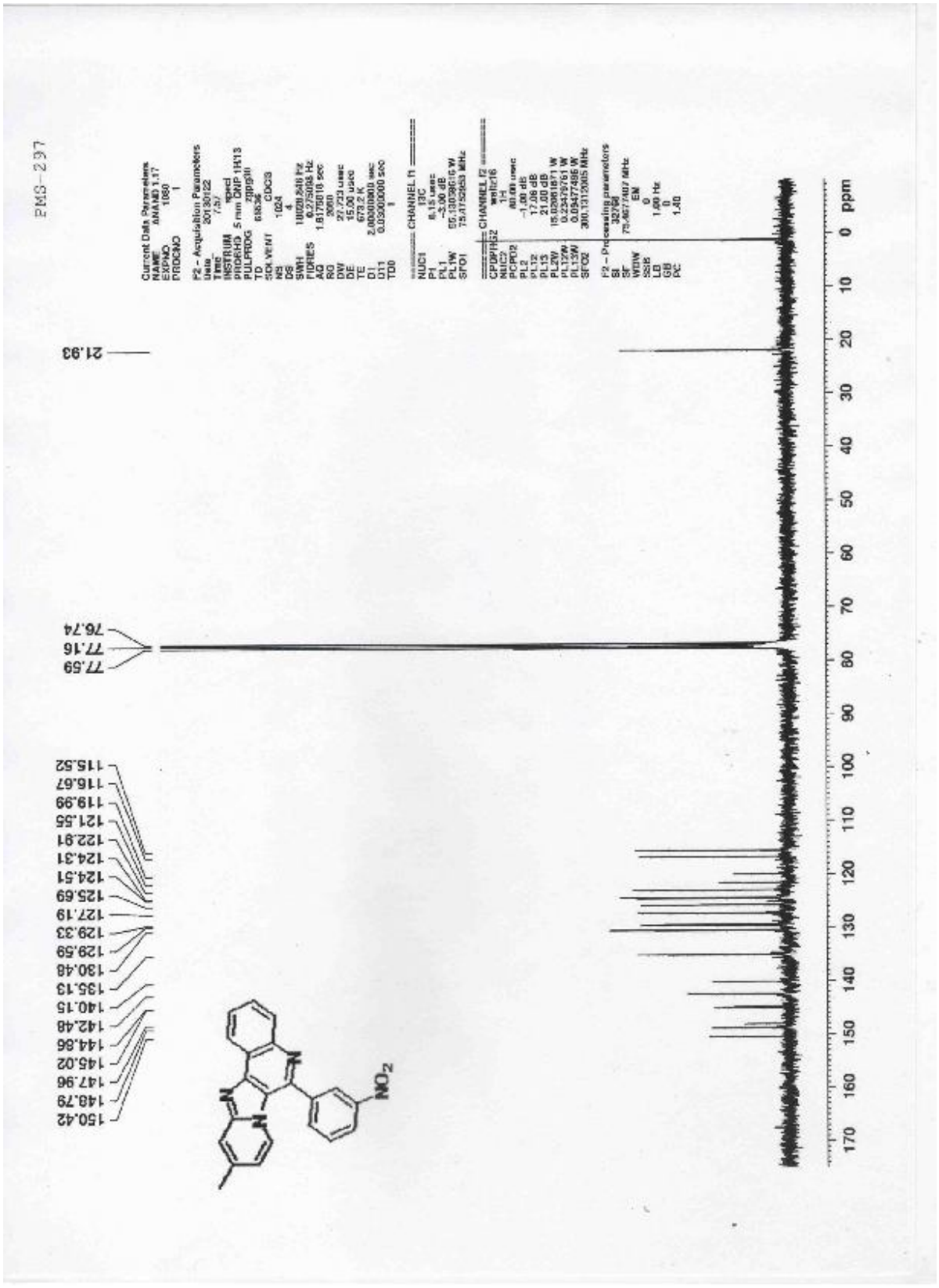
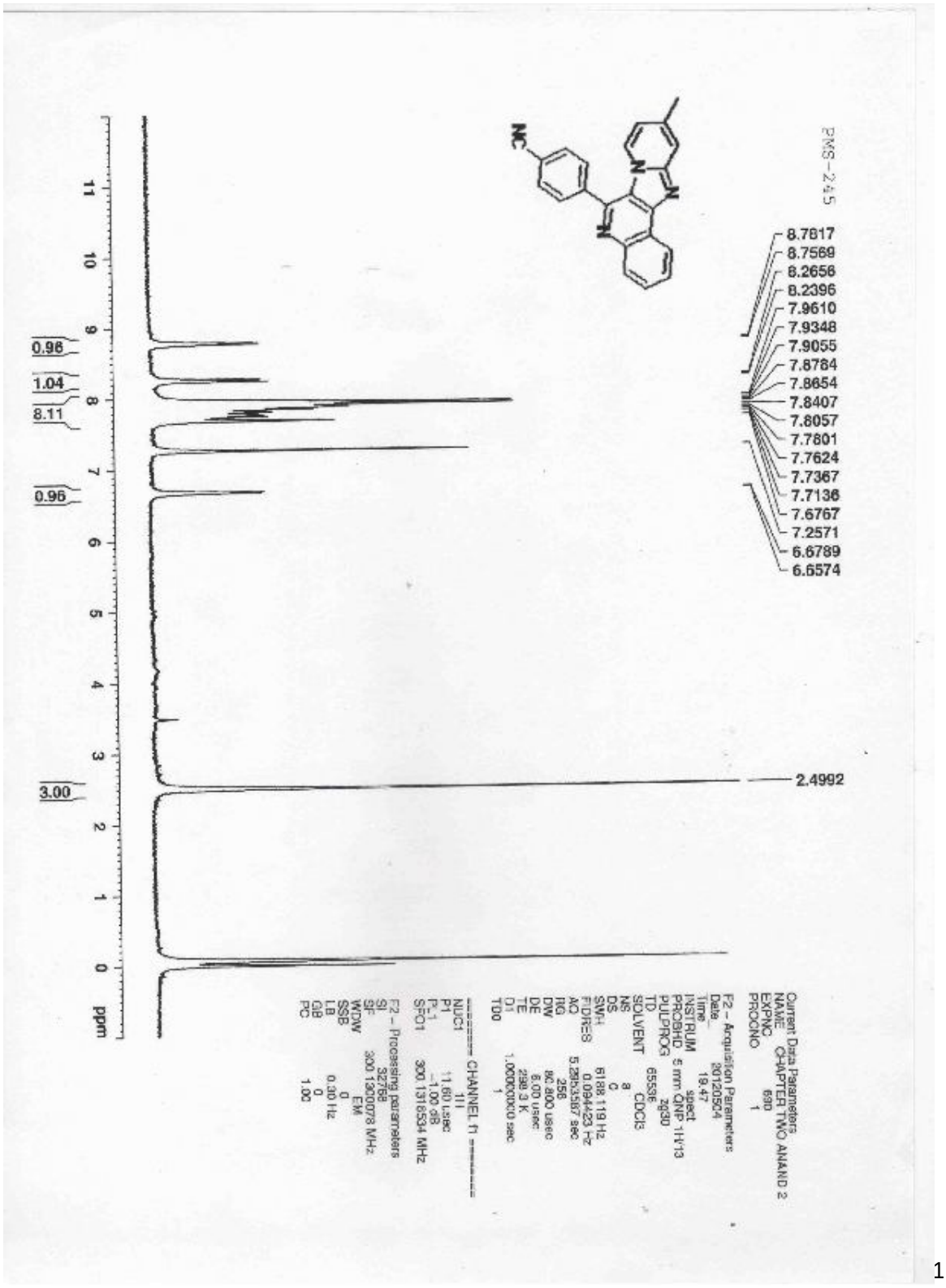
148.20
147.12
144.76
144.23
143.31
134.74
132.97
131.80
131.31
129.55
129.07
127.49
125.53
125.31
122.84
121.62
120.85
116.27
77.87
77.44
77.02
50.01
49.73
49.44
49.16
48.87
48.59
48.30
21.90
pns-296
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

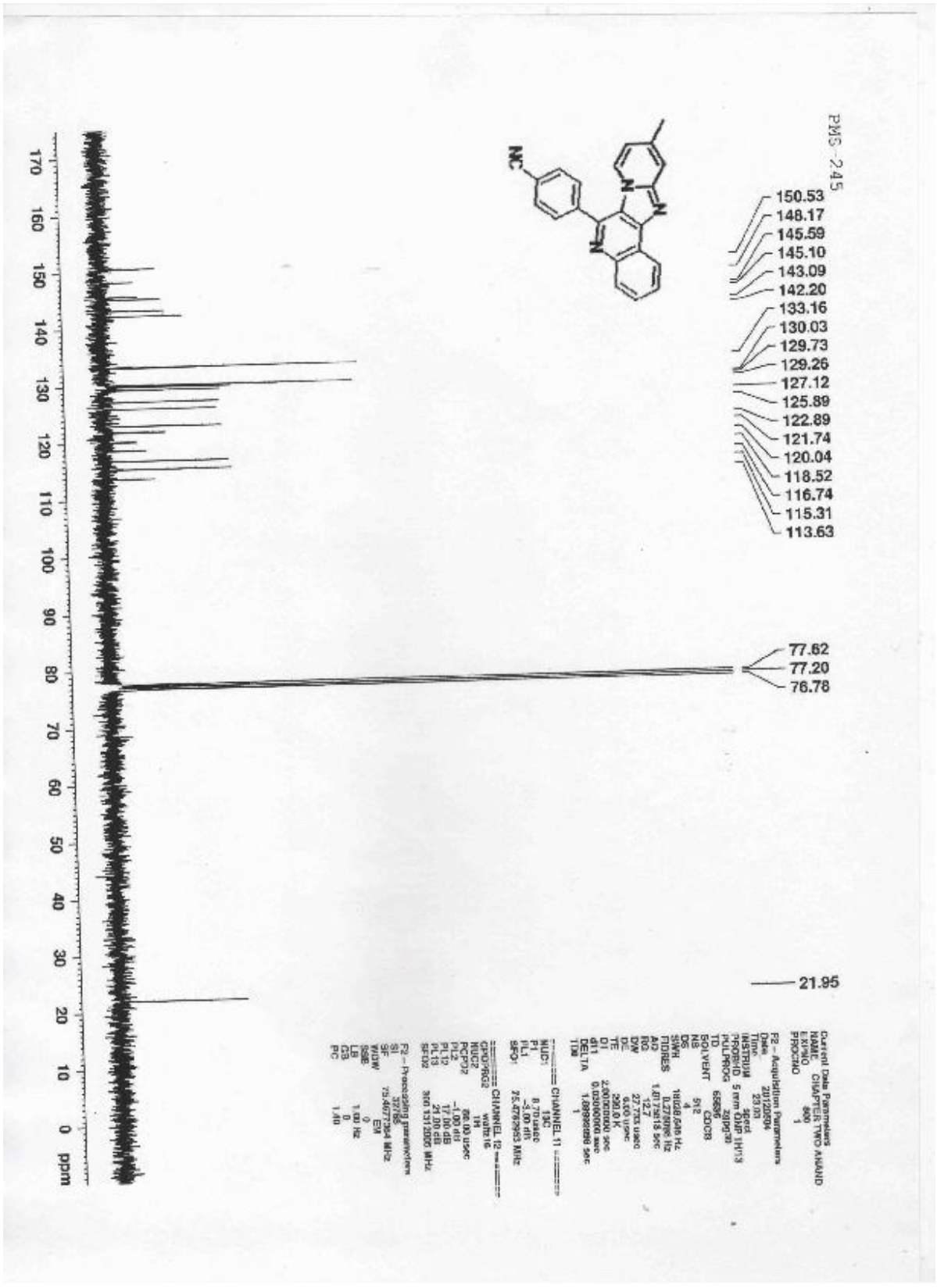
6-(3-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9i):



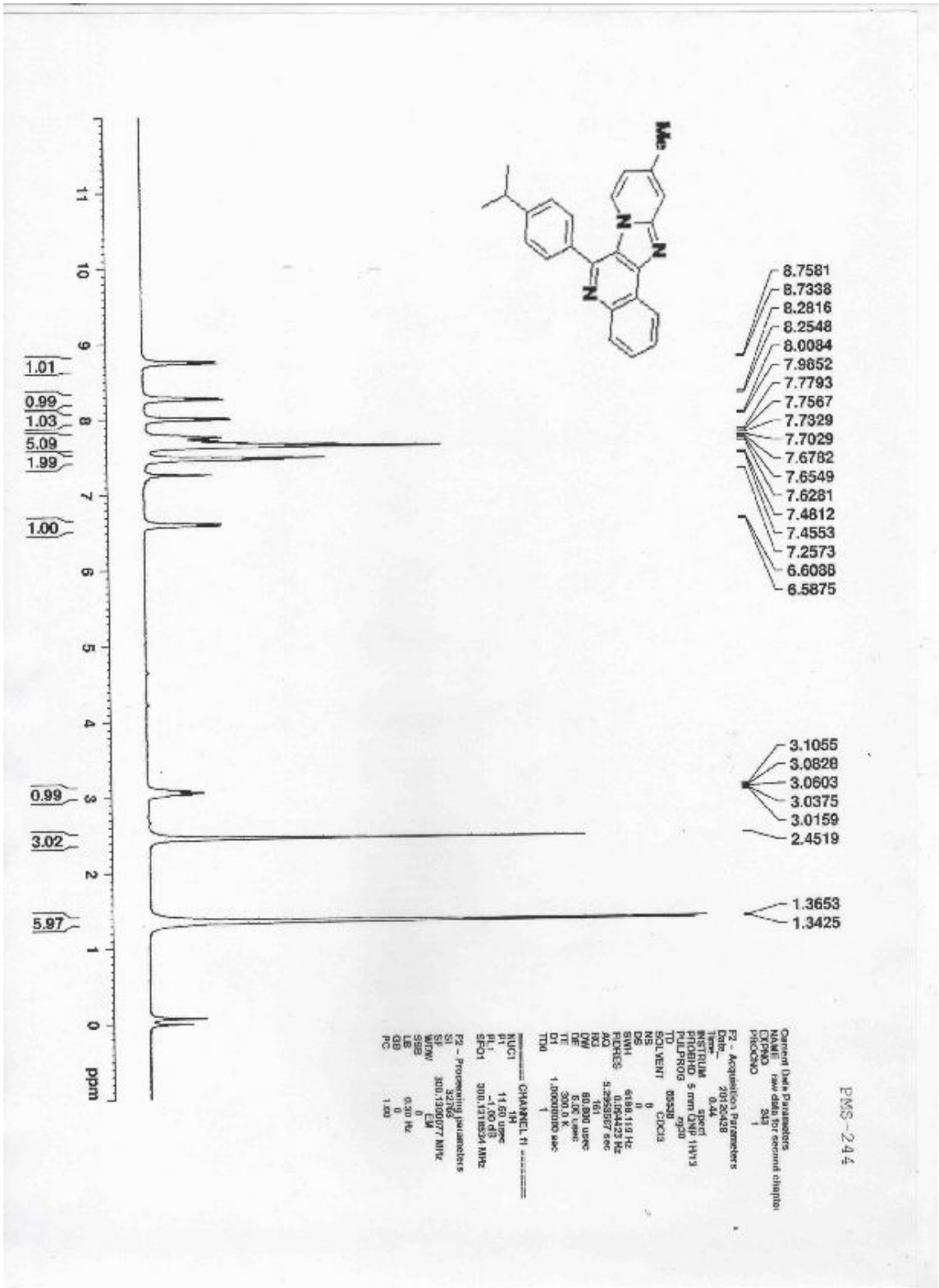

PMS-297
21.93
77.59
77.16
76.74
150.42
148.79
147.96
145.02
144.86
142.48
140.15
135.13
130.48
129.59
129.33
127.19
125.69
124.51
124.31
122.91
121.55
119.99
116.67
115.52
NO2
0 ppm

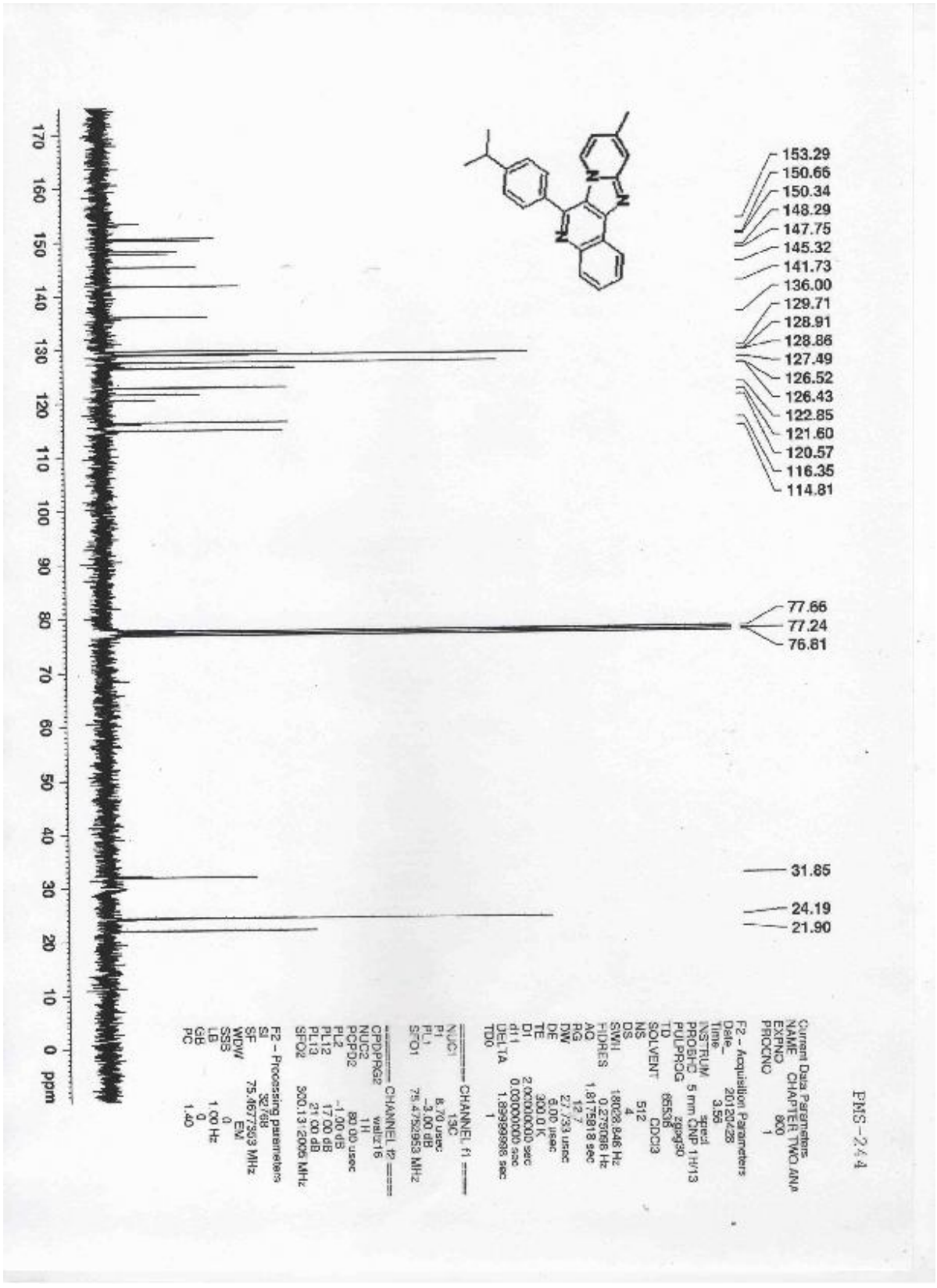
6-(4-Cyano-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9j):



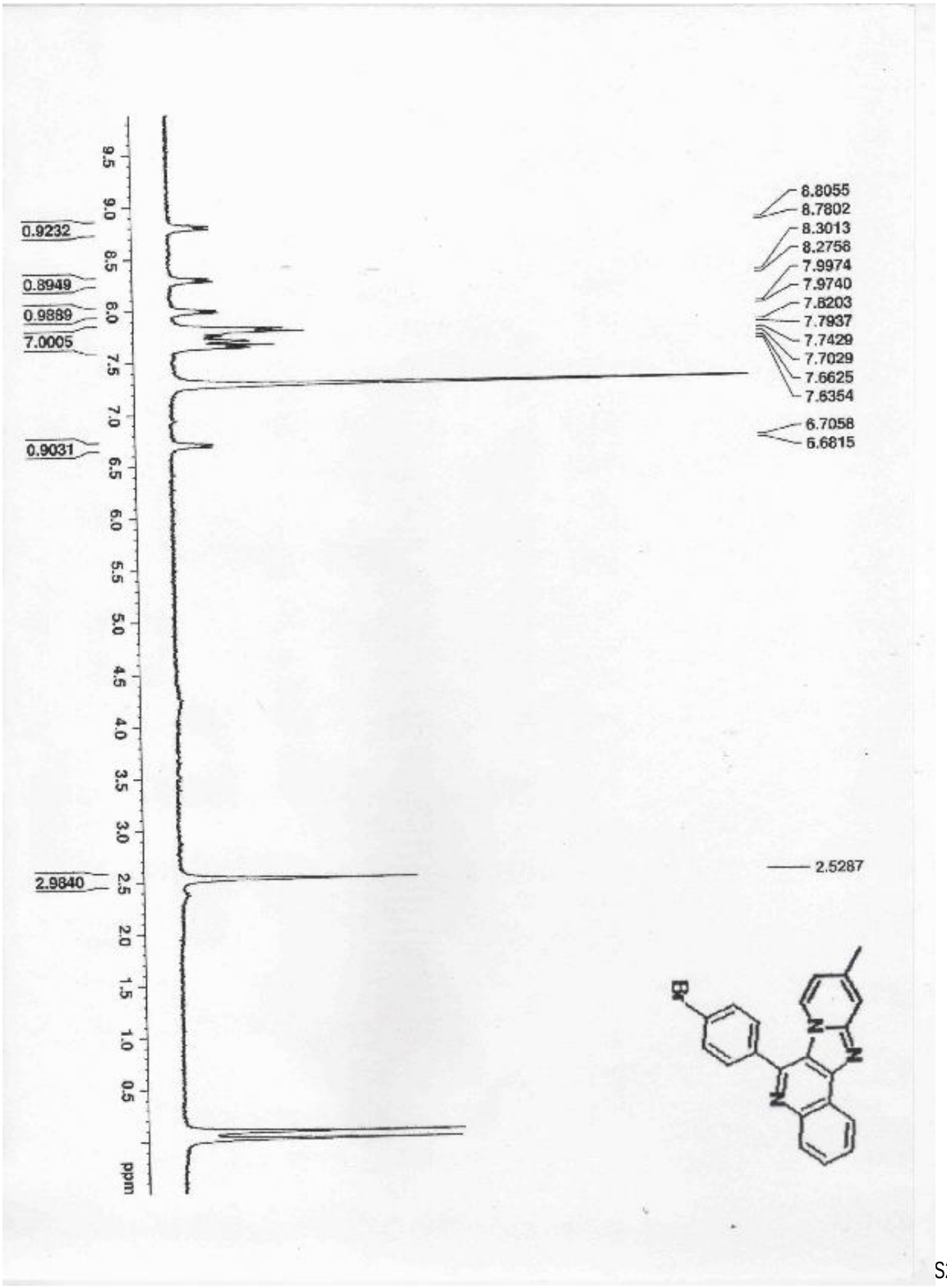

PMS-245
150.53
148.17
145.59
145.10
143.09
142.20
133.16
130.03
129.73
129.26
127.12
125.89
122.89
121.74
120.04
118.52
116.74
115.31
113.63
77.62
77.20
76.78
21.95
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

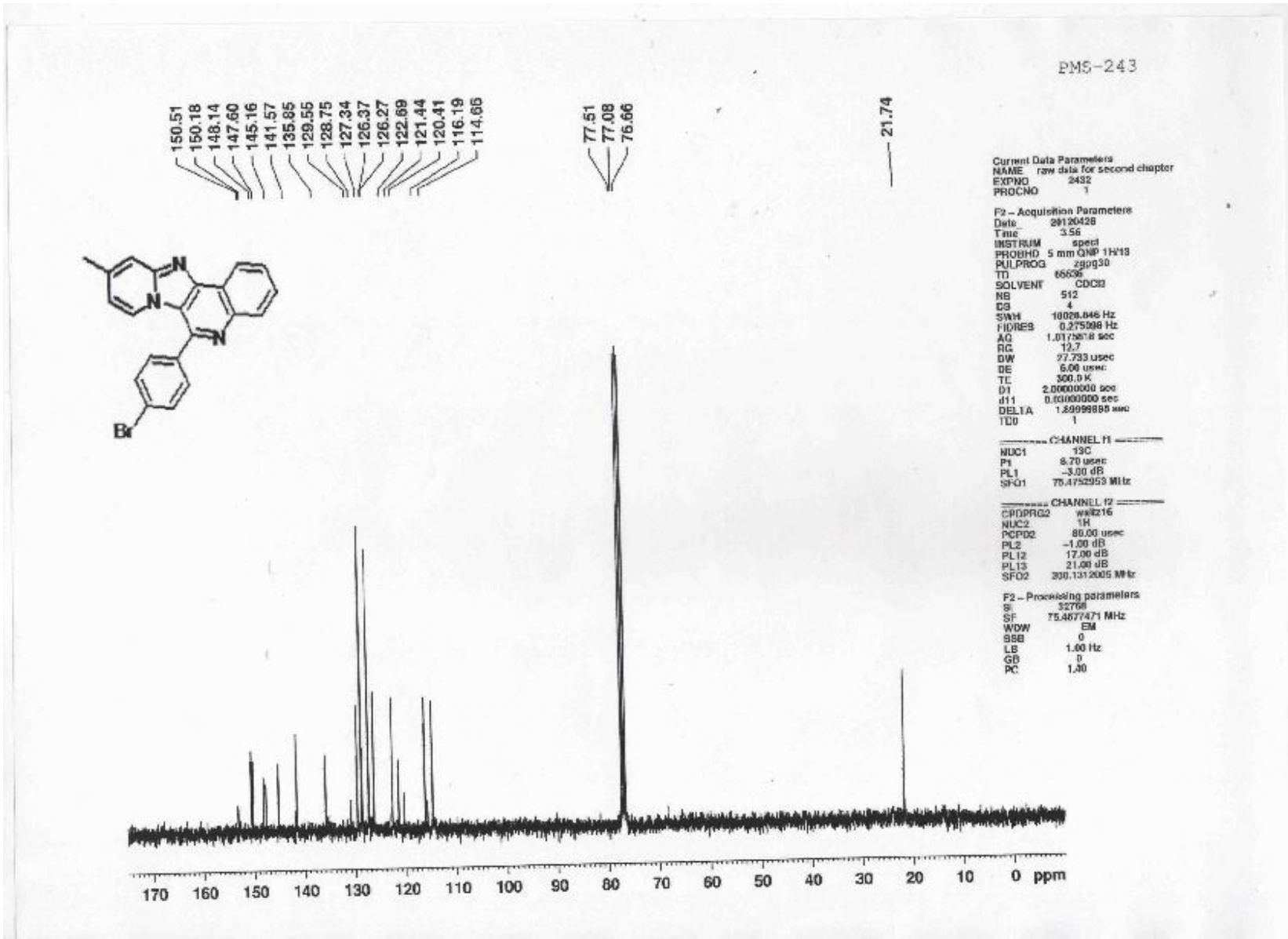
6-(4-Isopropyl-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9k):



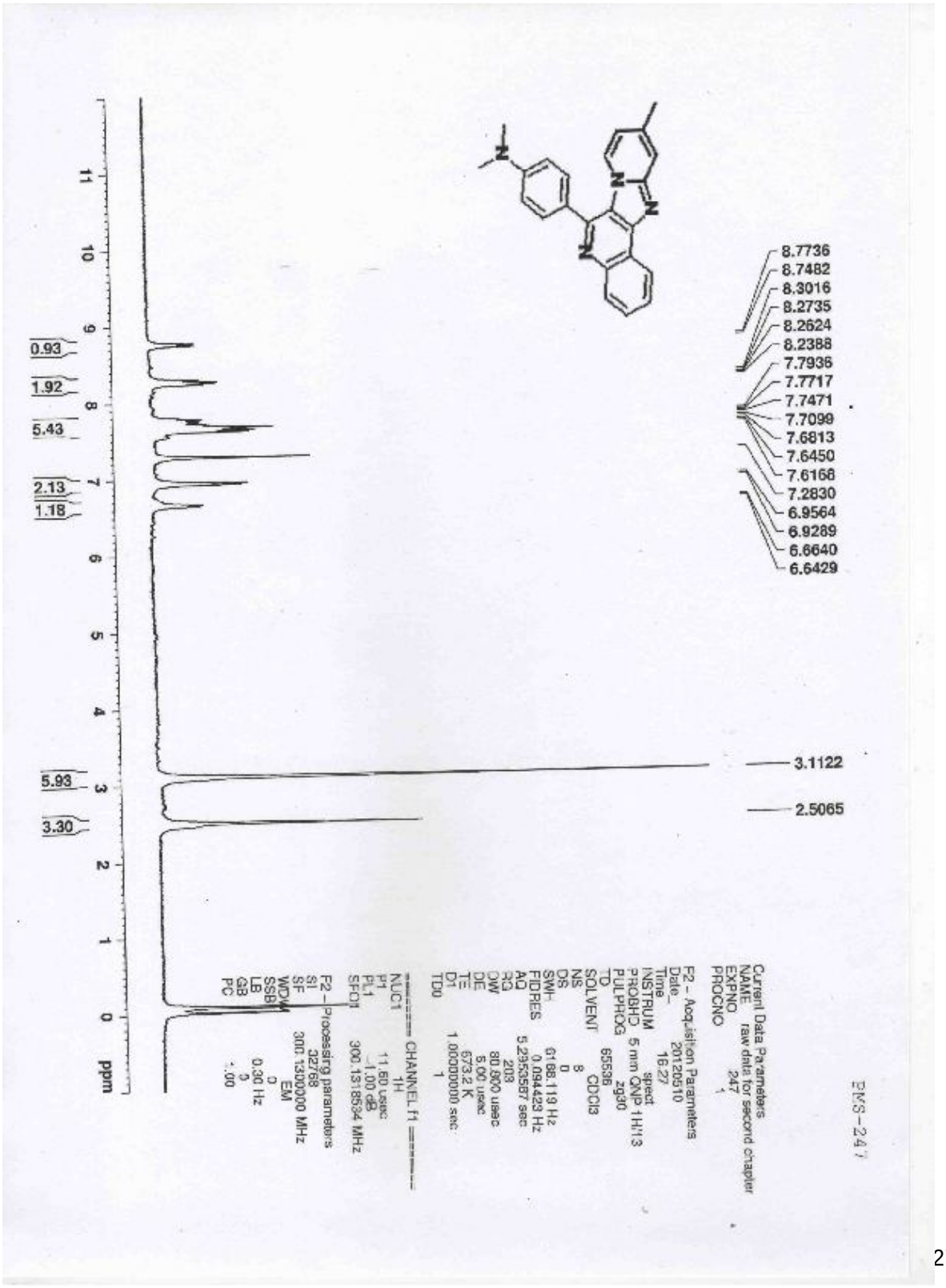

153.29
150.66
150.34
148.29
147.75
145.32
141.73
136.00
129.71
128.91
128.86
127.49
126.52
126.43
122.85
121.60
120.57
116.35
114.81
77.66
77.24
76.81
31.85
24.19
21.90
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
PMS-2-4
Current Data Parameters
NAME CHAPTER TWO ANA
EXPNO 800
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120428
Time 3.56
INSTRUM spect
PROBHD 5 mm CNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275086 Hz
AQ 1.8175818 sec
RG 12.7
DW 27.733 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677903 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

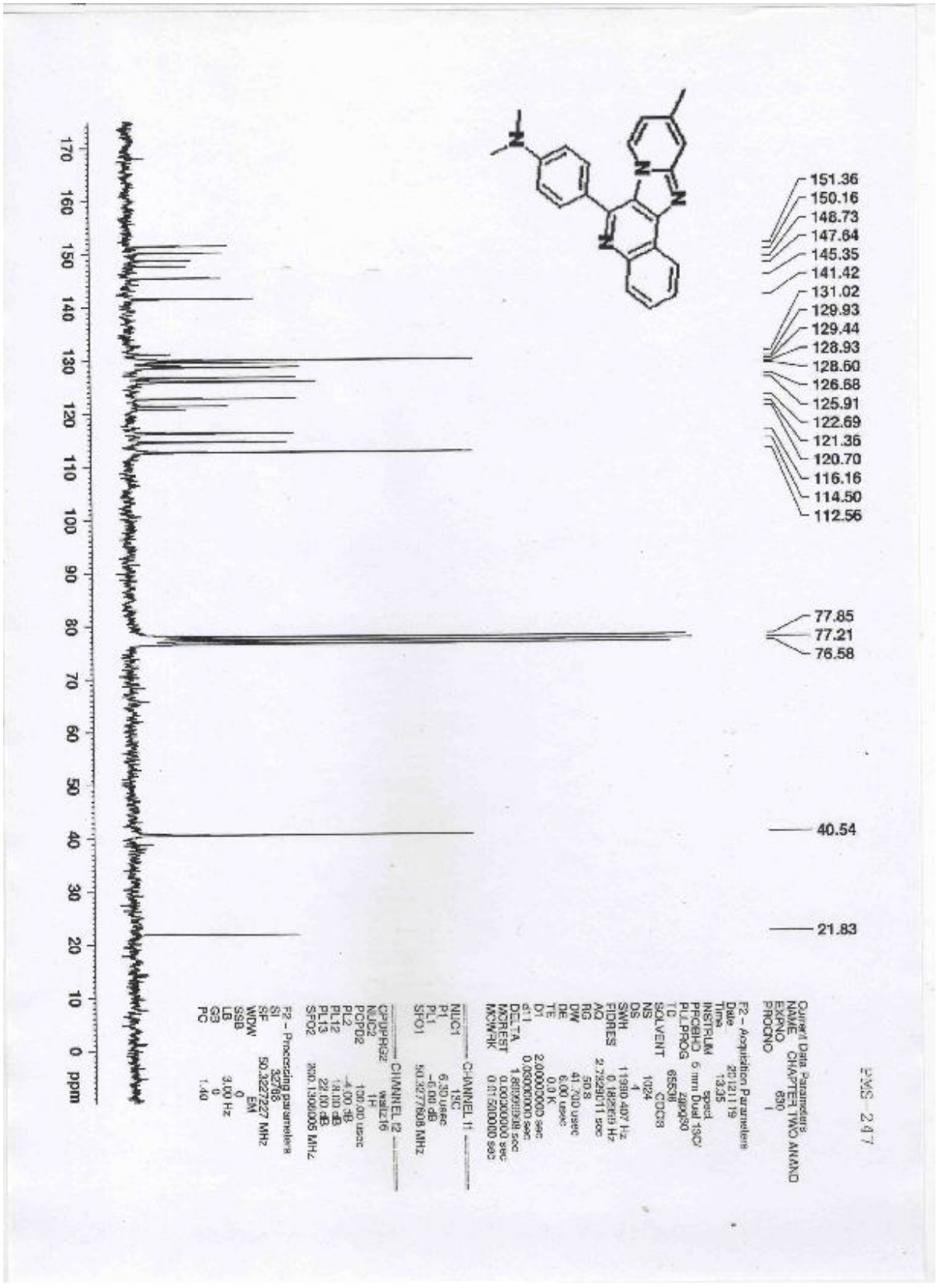
6-(4-Bromo-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9l):



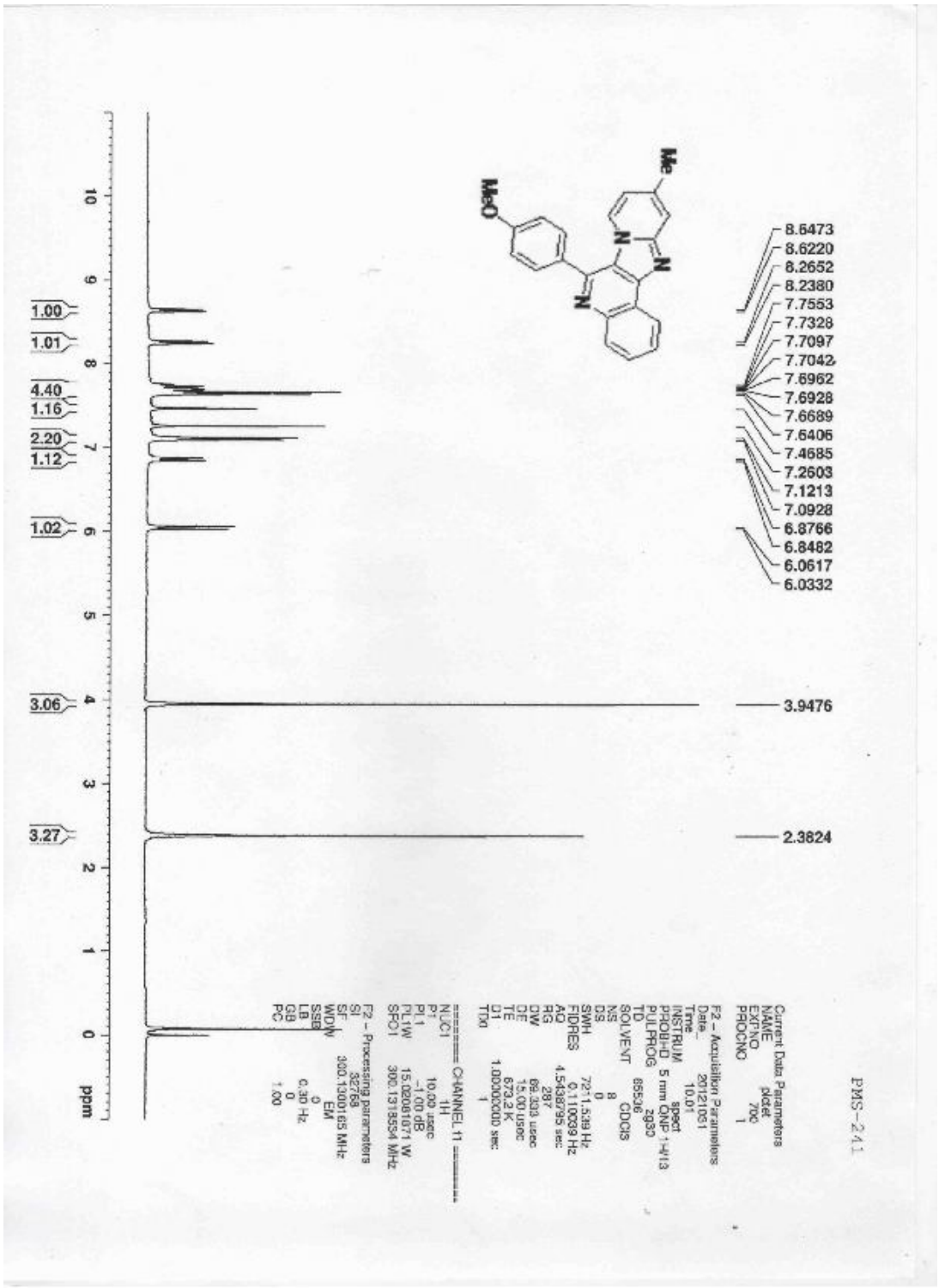

PMS-243
150.51
150.18
148.14
147.60
145.16
141.57
135.85
129.55
128.75
127.34
126.37
126.27
122.69
121.44
120.41
116.19
114.66
77.51
77.08
76.66
21.74
Br
Current Data Parameters
NAME raw data for second chapter
EXPNO 2432
PROCNO 1
F2 - Acquisition Parameters
Date 20120428
Time 3.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175616 sec
RG 12.7
DW 27.733 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999885 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677471 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

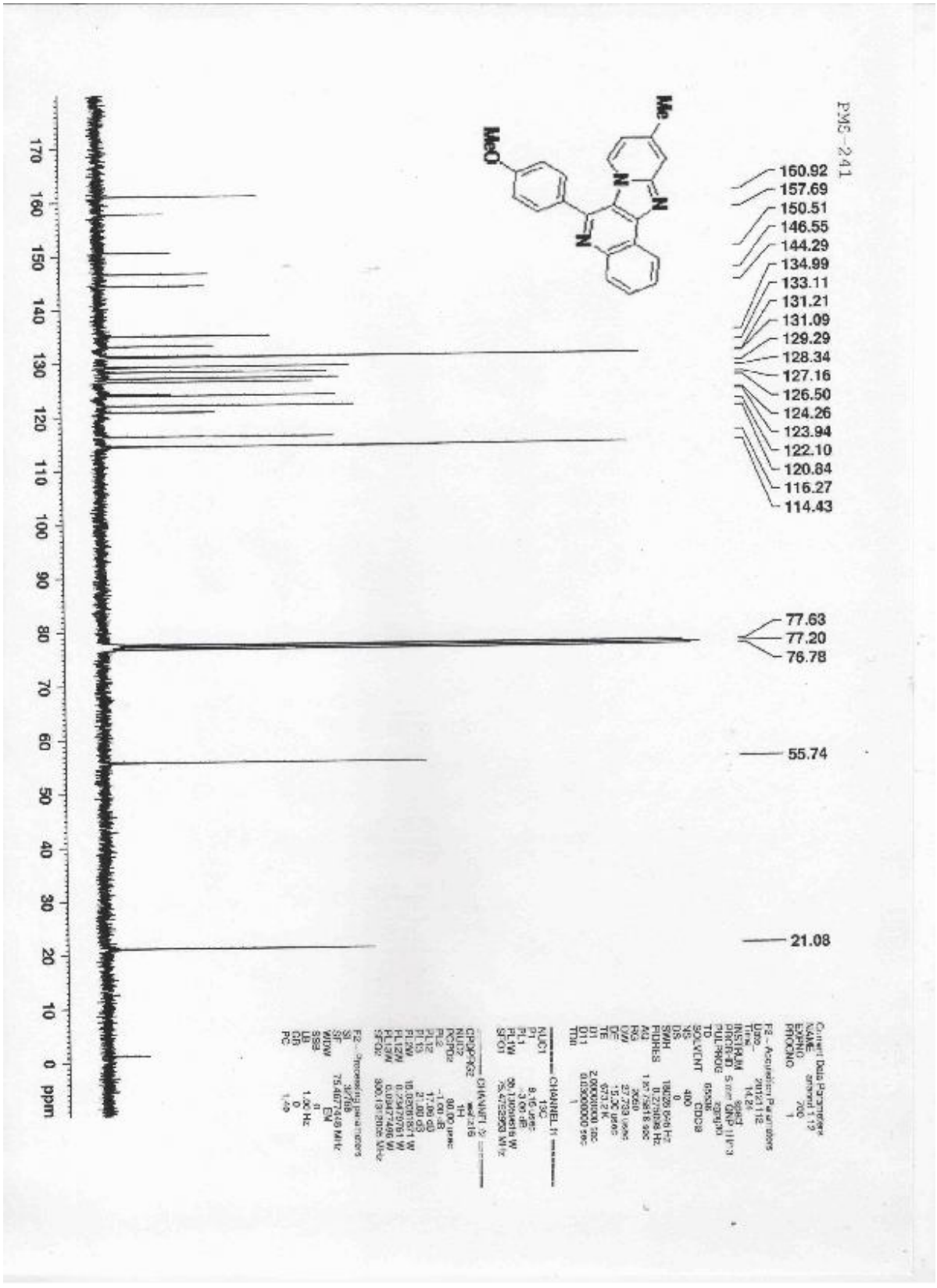
6-(4-N, N-dimethylamino-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9m):



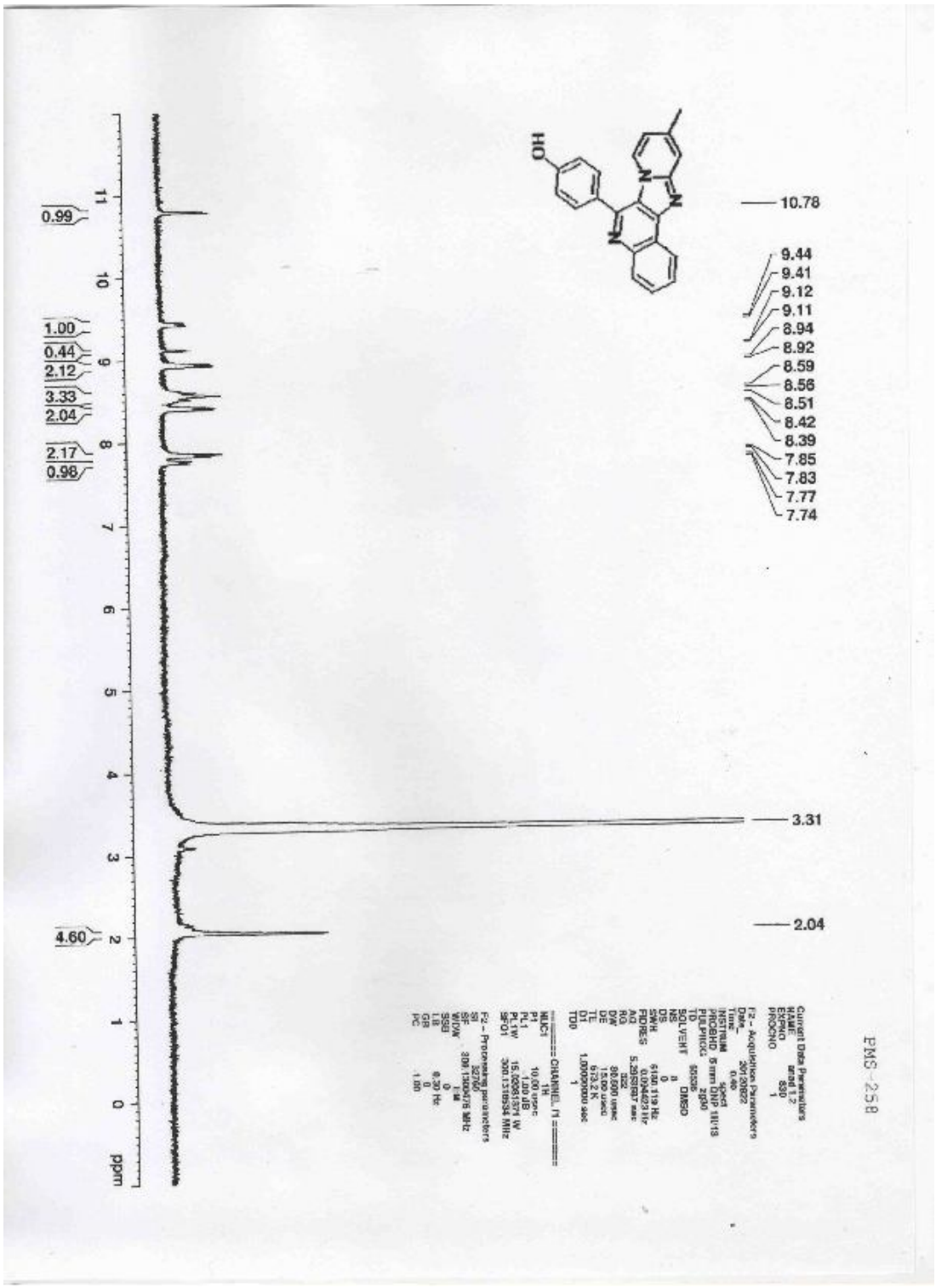

151.36
150.16
148.73
147.64
145.35
141.42
131.02
129.93
129.44
128.93
128.60
126.68
125.91
122.69
121.36
120.70
116.16
114.50
112.56
77.85
77.21
76.58
40.54
21.83
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

6-(4-Methoxy-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9n):

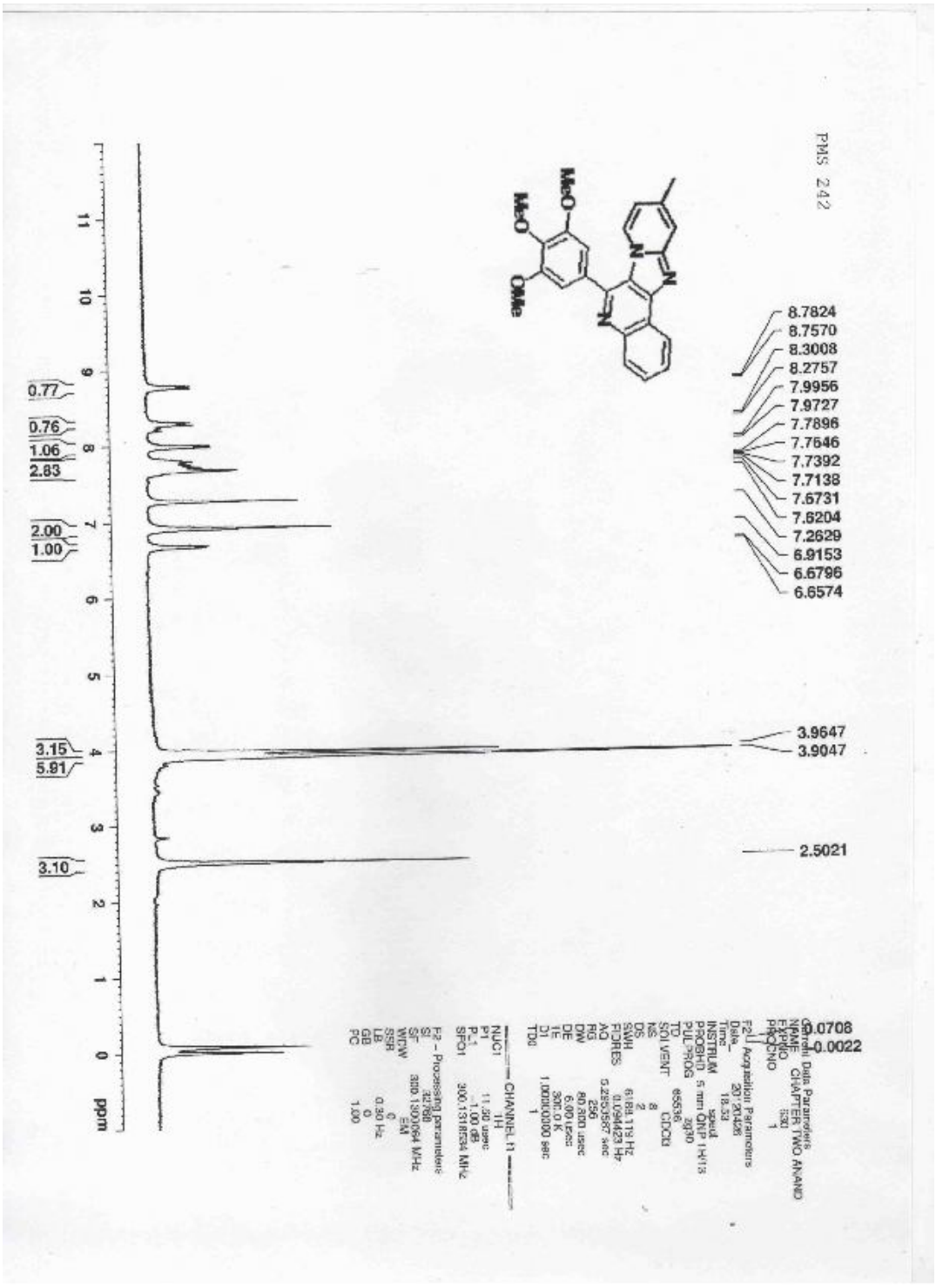


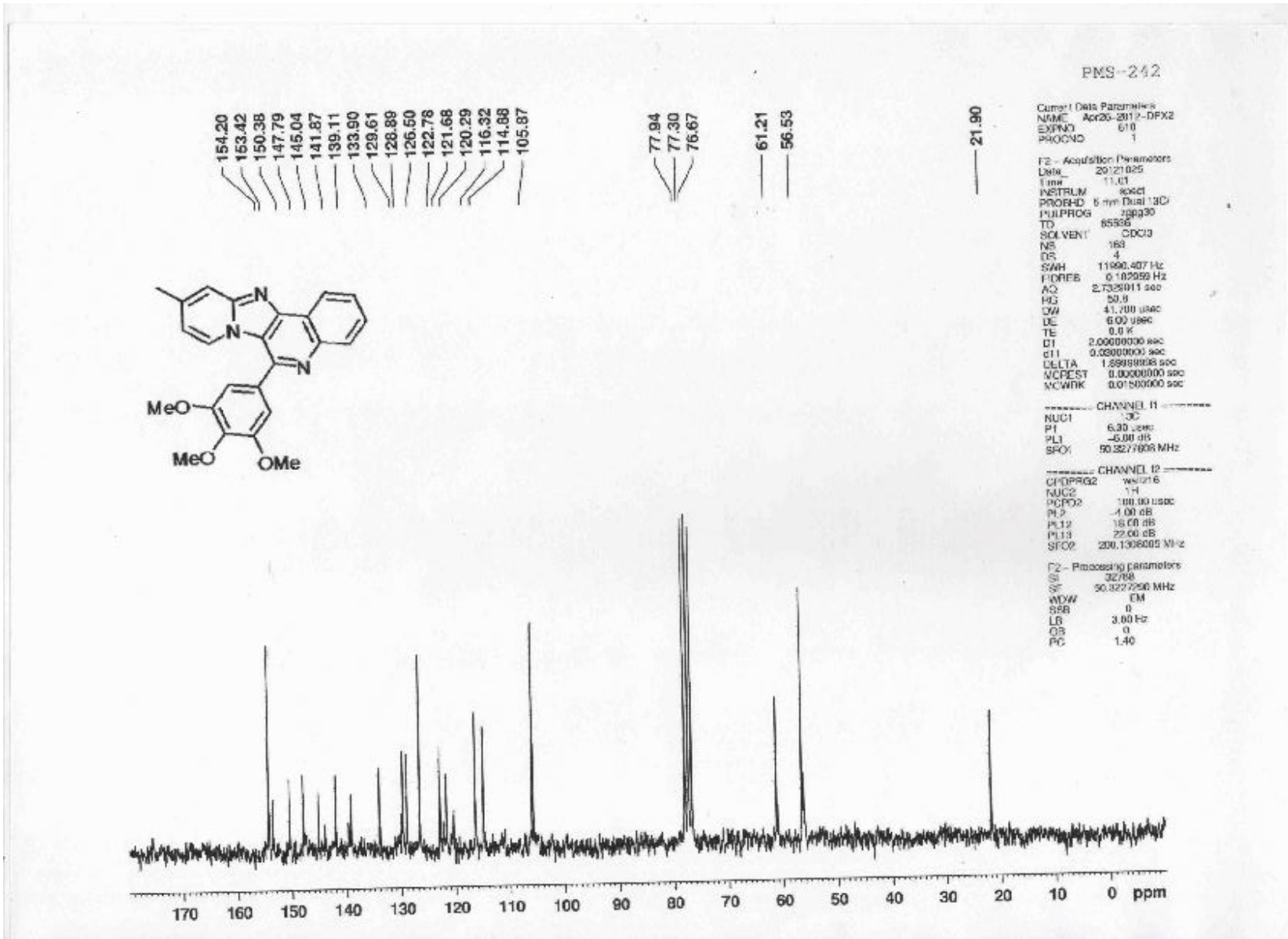

PMS-241
Me
MeO
N
N
N
160.92
157.69
150.51
146.55
144.29
134.99
133.11
131.21
131.09
129.29
128.34
127.16
126.50
124.26
123.94
122.10
120.84
116.27
114.43
77.63
77.20
76.78
55.74
21.08
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4-(9-Methyl-5,6b,11-triaza-benzo[a]fluoren-6-yl)-phenol (9o):

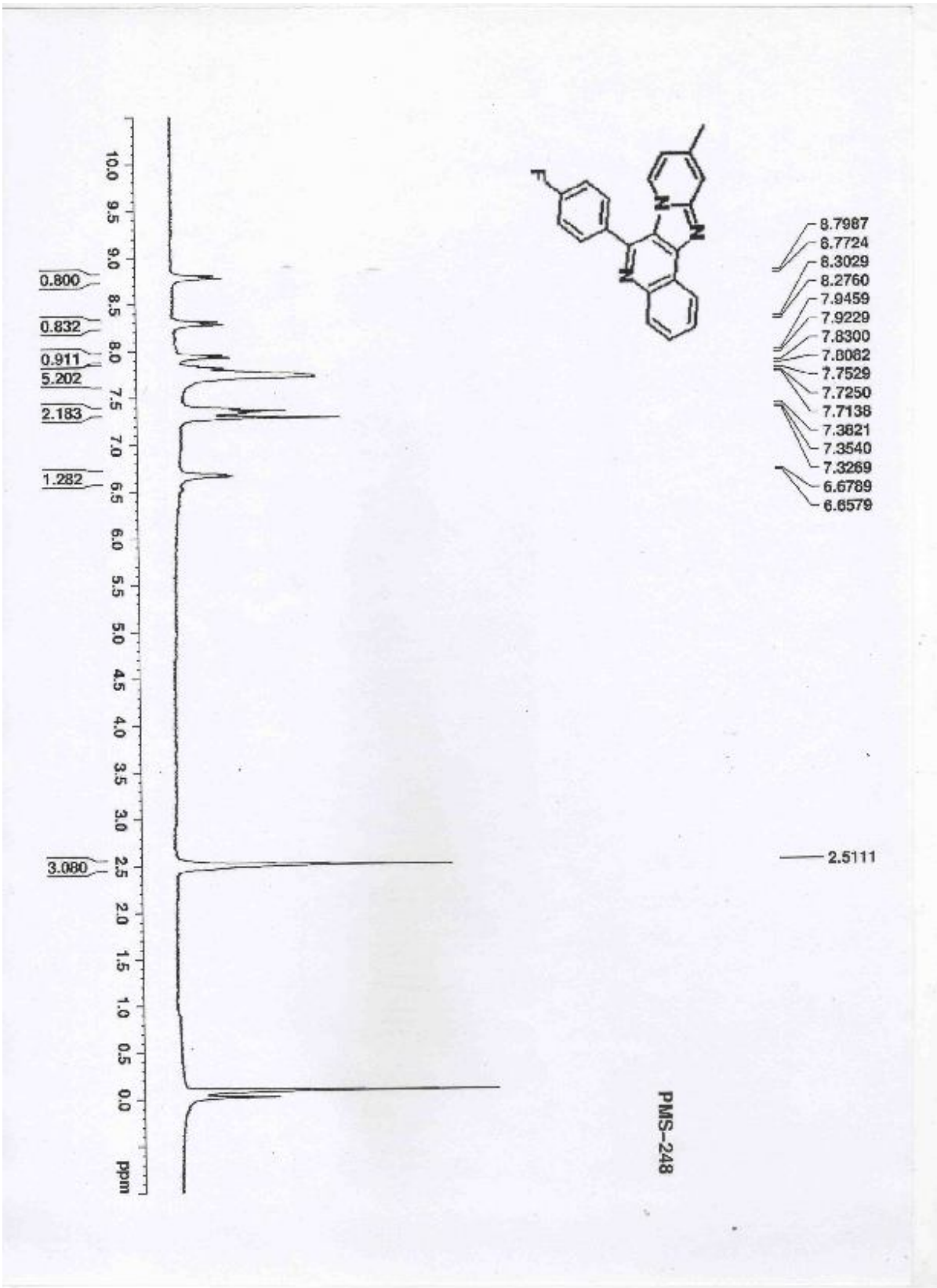


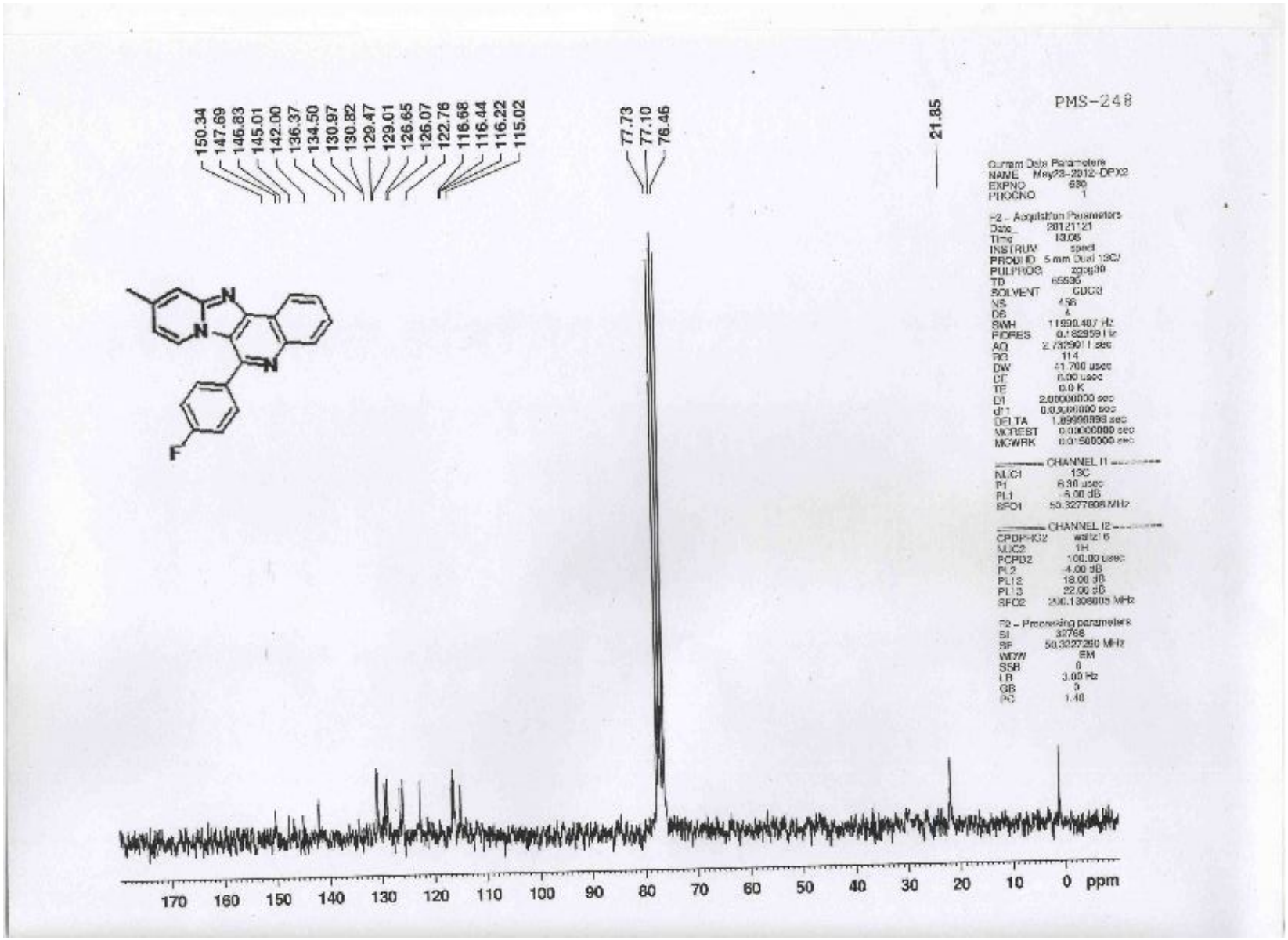
6-(3,4,5-trimethoxy-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9p):



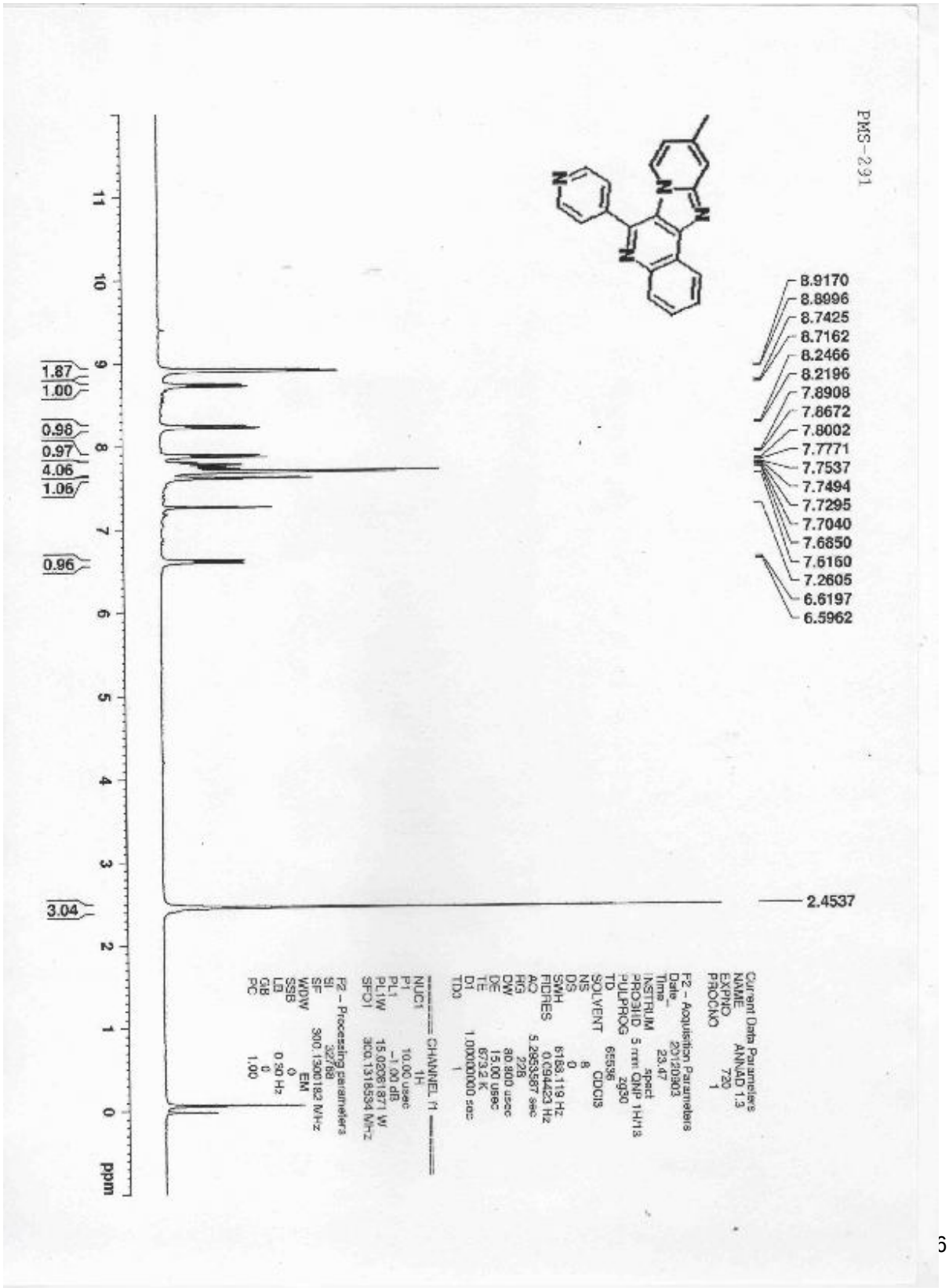

PMS-242
154.20
153.42
150.38
147.79
145.04
141.87
139.11
133.90
129.61
128.89
126.50
122.78
121.68
120.29
116.32
114.88
105.87
77.94
77.30
76.67
61.21
56.53
21.90
MeO
MeO
OMe
N
N
N
Current Data Parameters
NAME Apr26-2012-DPX2
EXPNO 610
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121025
Time 11.01
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7328911 sec
RG 50.8
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
VCPEST 0.00000000 sec
VCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -5.00 dB
SFO1 50.3227608 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 16.06 dB
PL13 22.06 dB
SFO2 200.1308005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

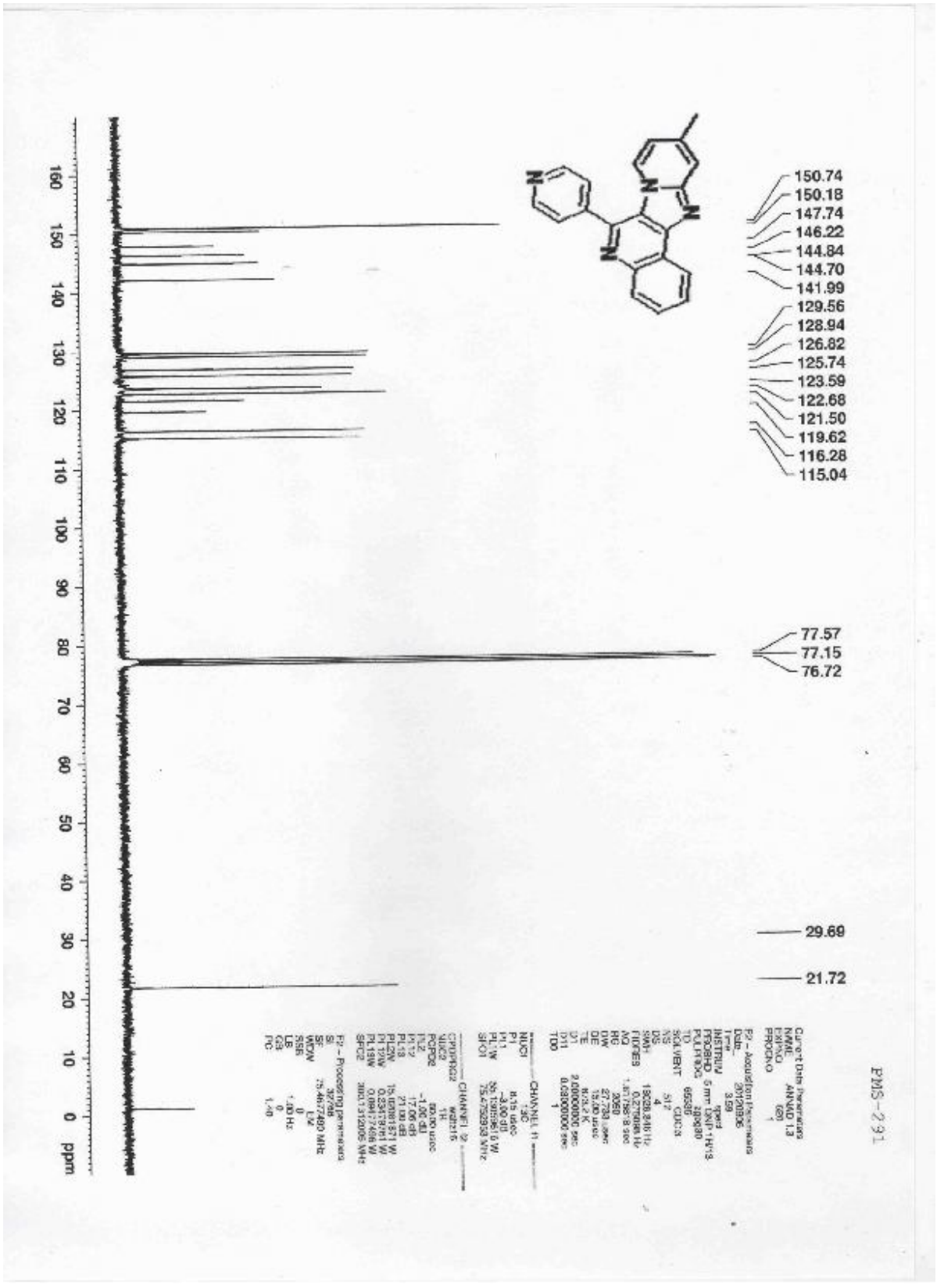
6-(4-Flouro-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9q):



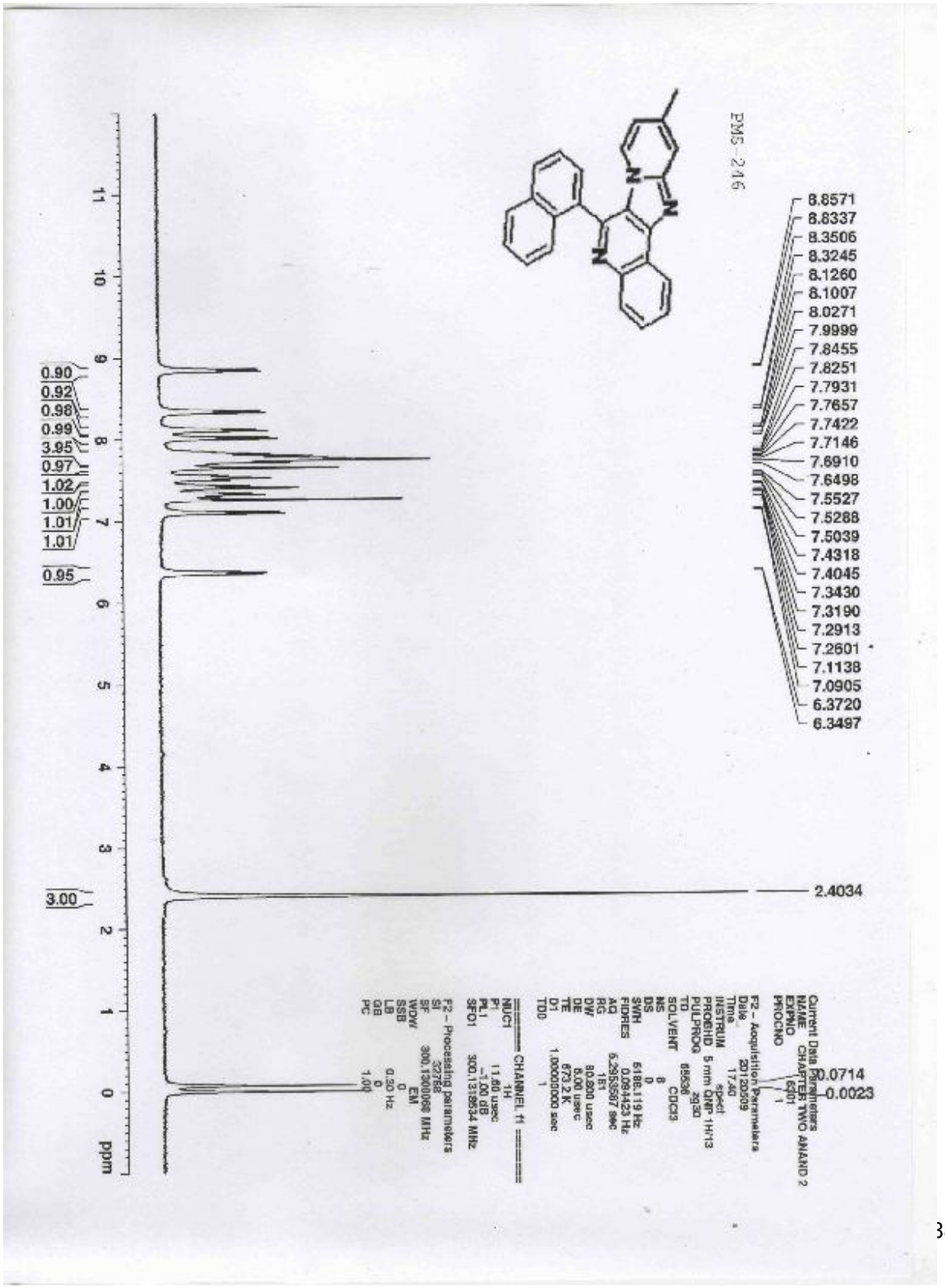

PMS-248
150.34
147.69
146.83
145.01
142.00
136.37
134.50
130.97
130.82
129.47
129.01
126.65
126.07
122.76
116.68
116.44
116.22
115.02
77.73
77.10
76.46
21.85
N
N
N
F
Current Data Parameters
NAME May28-2012-DPX2
EXPNO 630
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121121
Time 13.06
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 456
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 114
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.99999993 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1306005 MHz
F2 - Processing parameters
SI 32768
SF 50.3277250 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

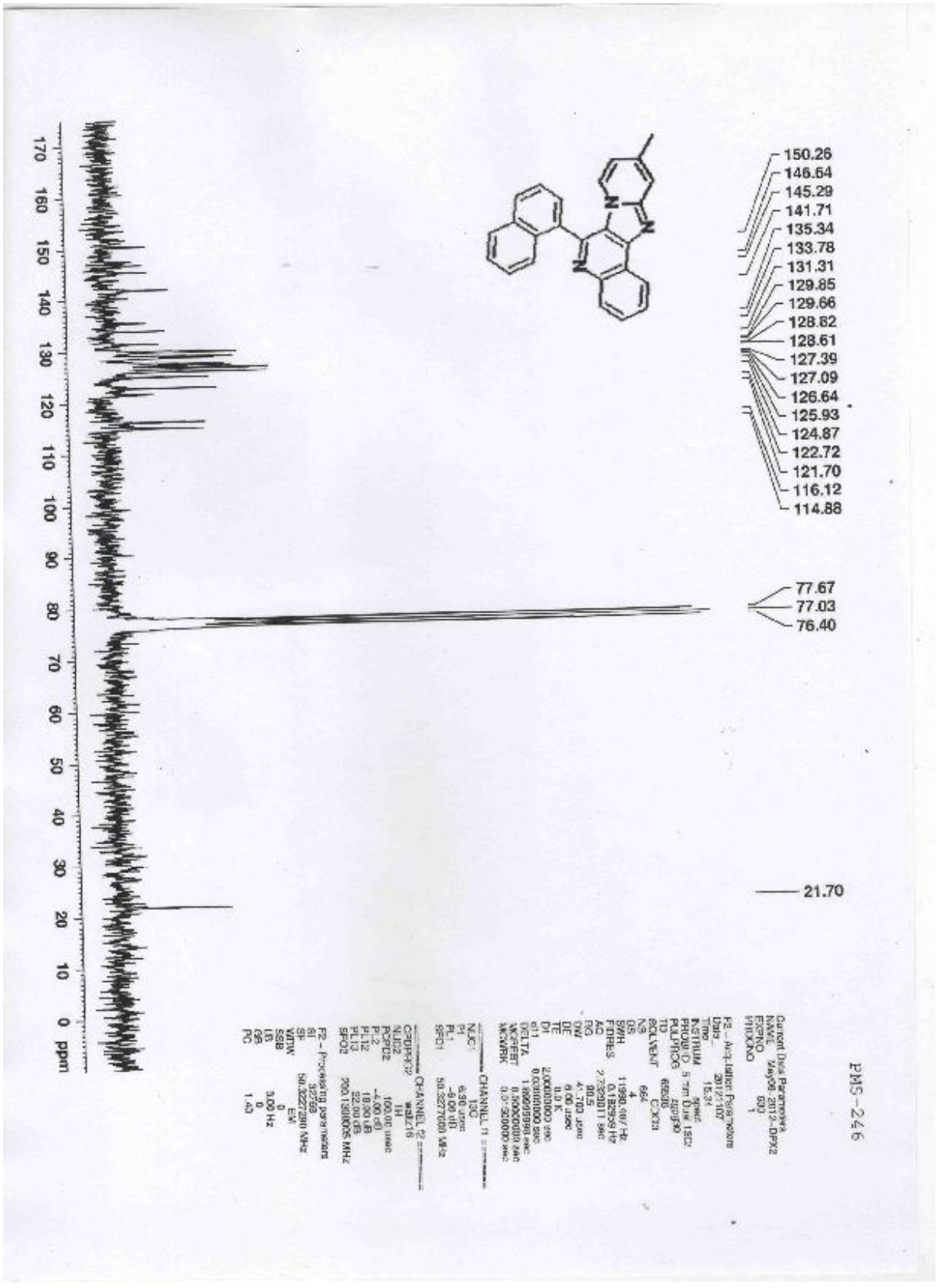
6-(4-Pyridyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9r):



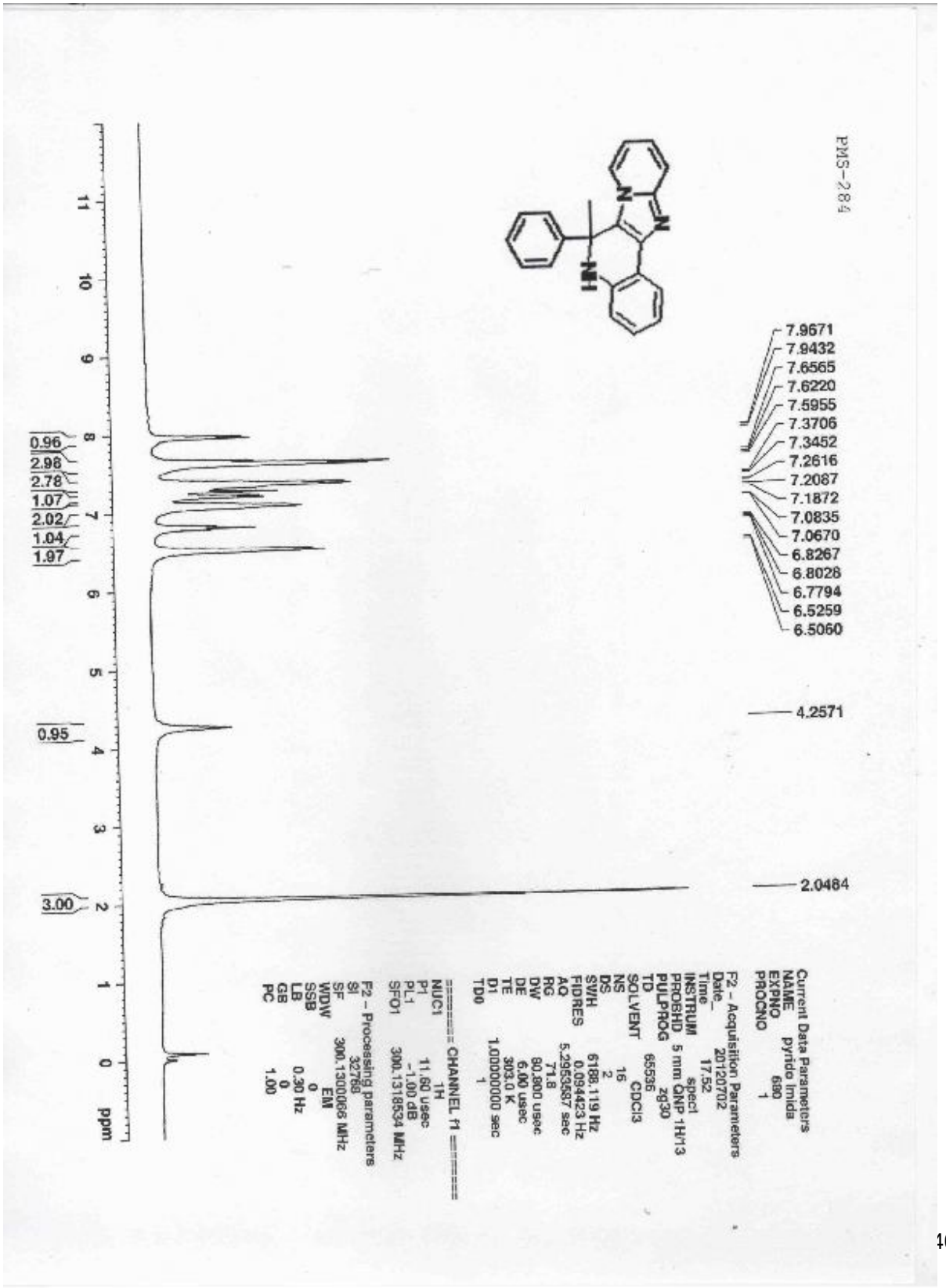

150.74
150.18
147.74
146.22
144.84
144.70
141.99
129.56
128.94
126.82
125.74
123.59
122.68
121.50
119.62
116.28
115.04
77.57
77.15
76.72
29.69
21.72
PMS-231
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

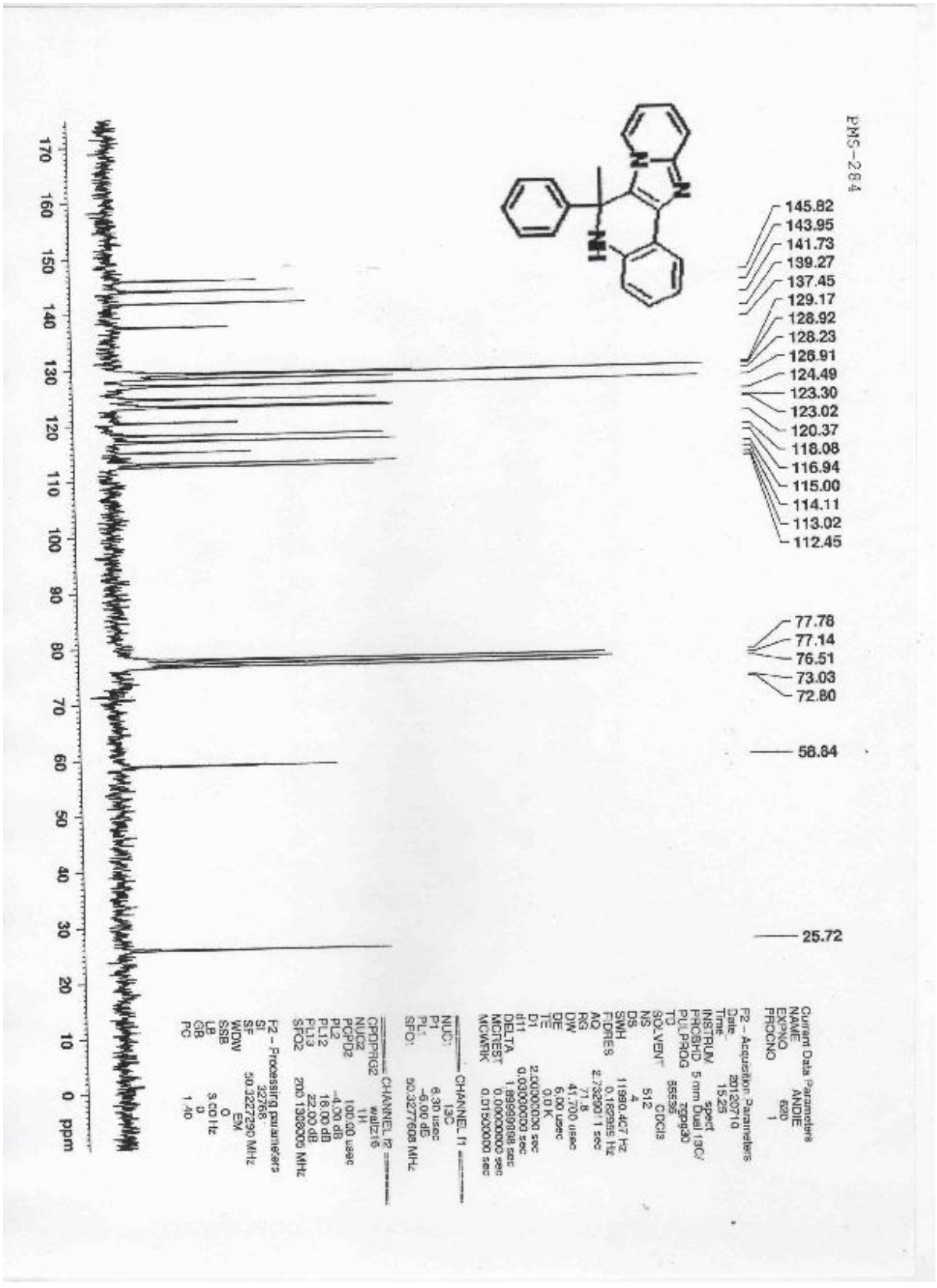
6-(1-Naphthyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9s):



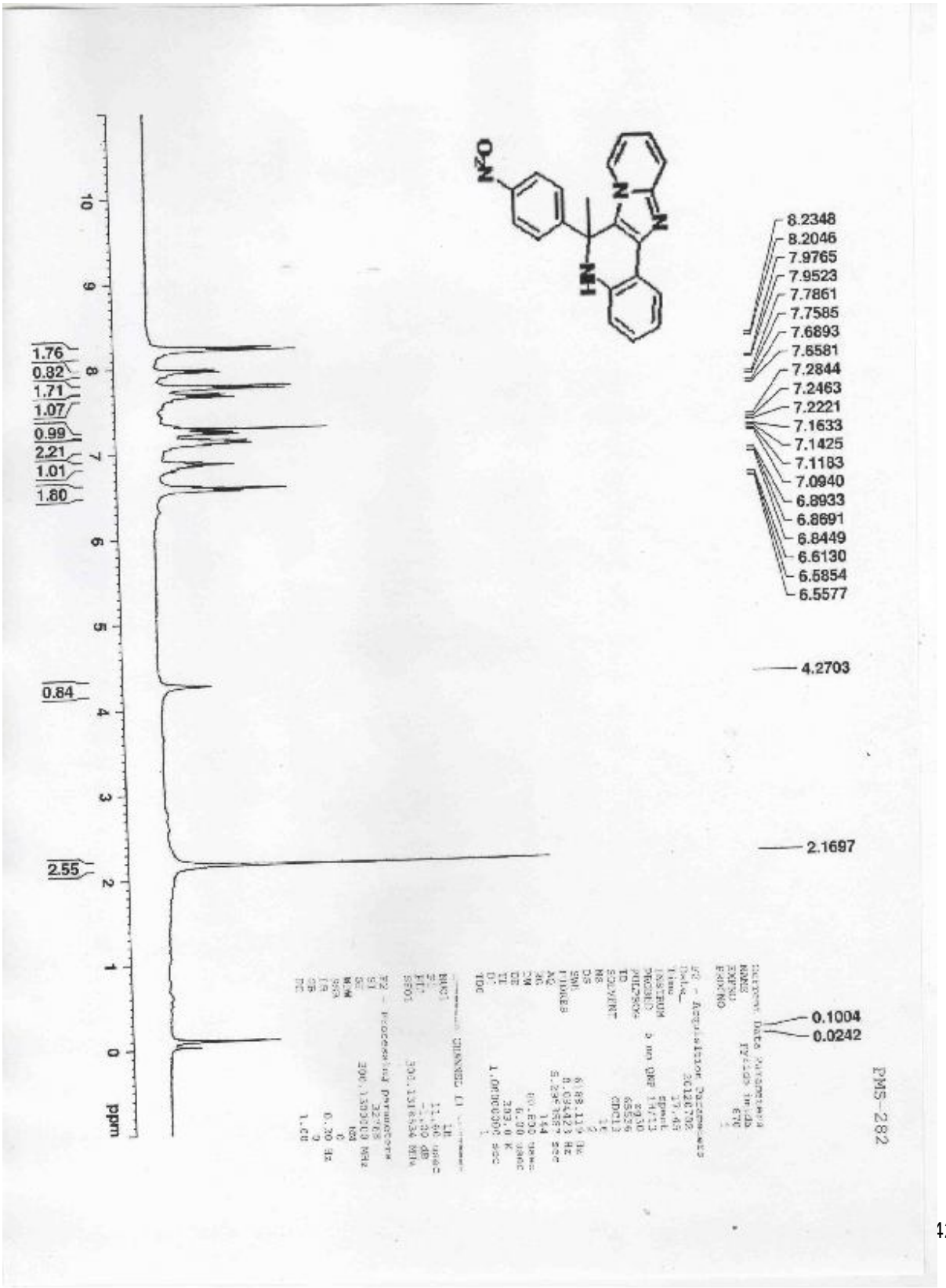

150.26
146.64
145.29
141.71
135.34
133.78
131.31
129.85
129.66
128.62
128.61
127.39
127.09
126.64
125.93
124.87
122.72
121.70
116.12
114.88
77.67
77.03
76.40
21.70
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
PMS-246

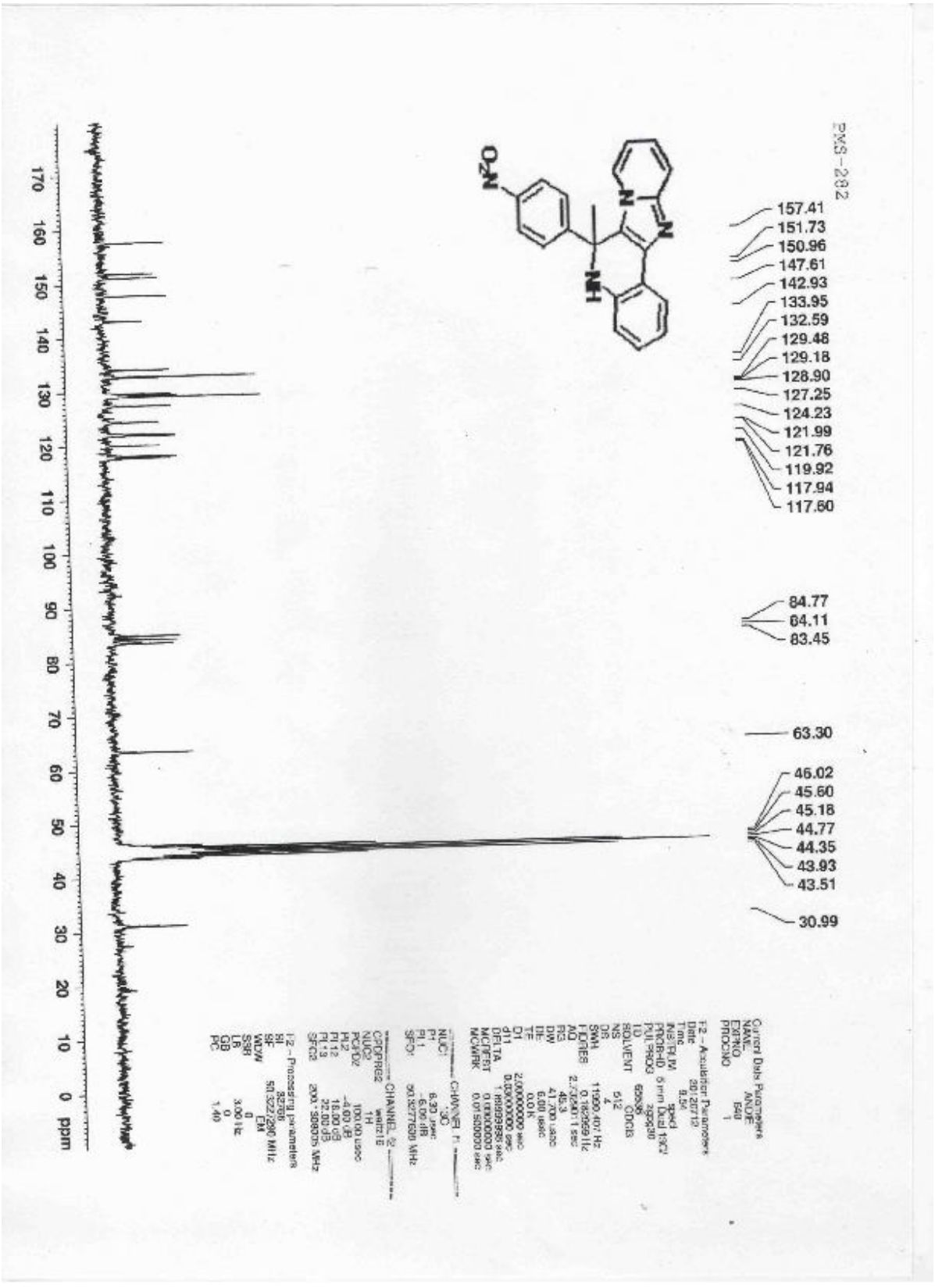
6-Methyl-6-(phenyl)-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorene (11a):



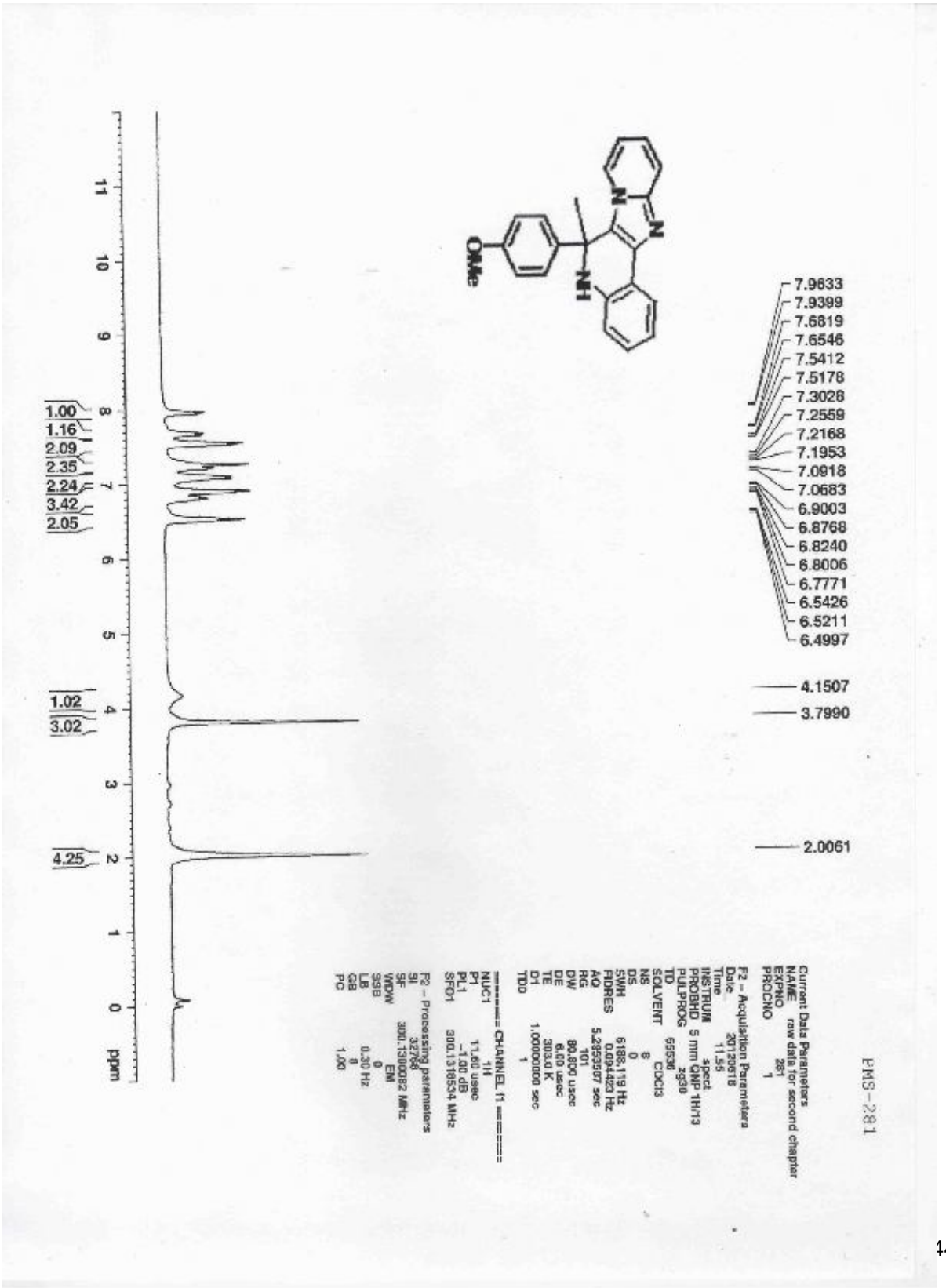

PMS-284
145.82
143.95
141.73
139.27
137.45
129.17
128.92
128.23
126.91
124.49
123.30
123.02
120.37
118.08
116.94
115.00
114.11
113.02
112.45
77.78
77.14
76.51
73.03
72.80
58.84
25.72
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

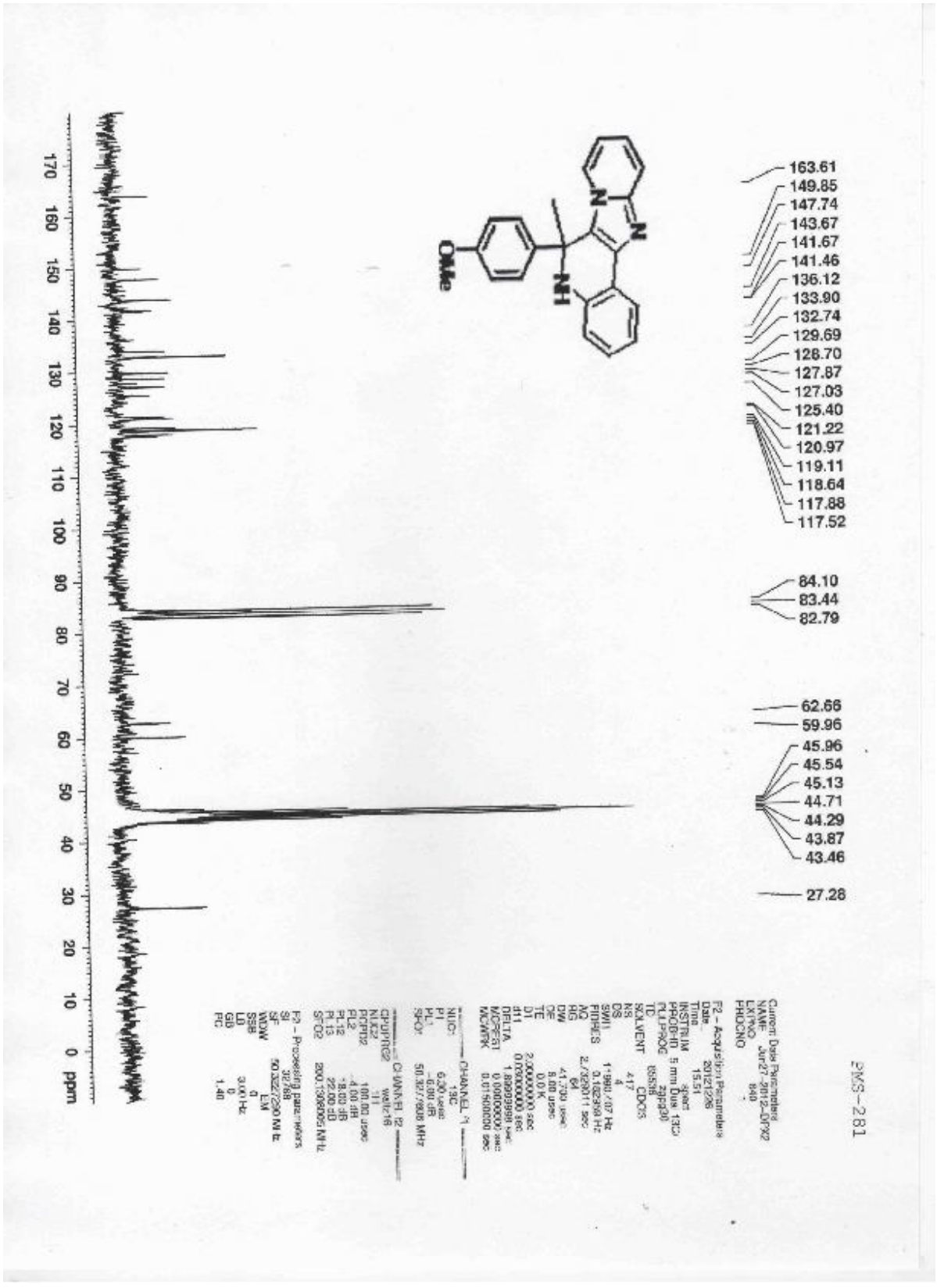
6-Methyl-6-(4-nitro-phenyl)-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorene (11b):



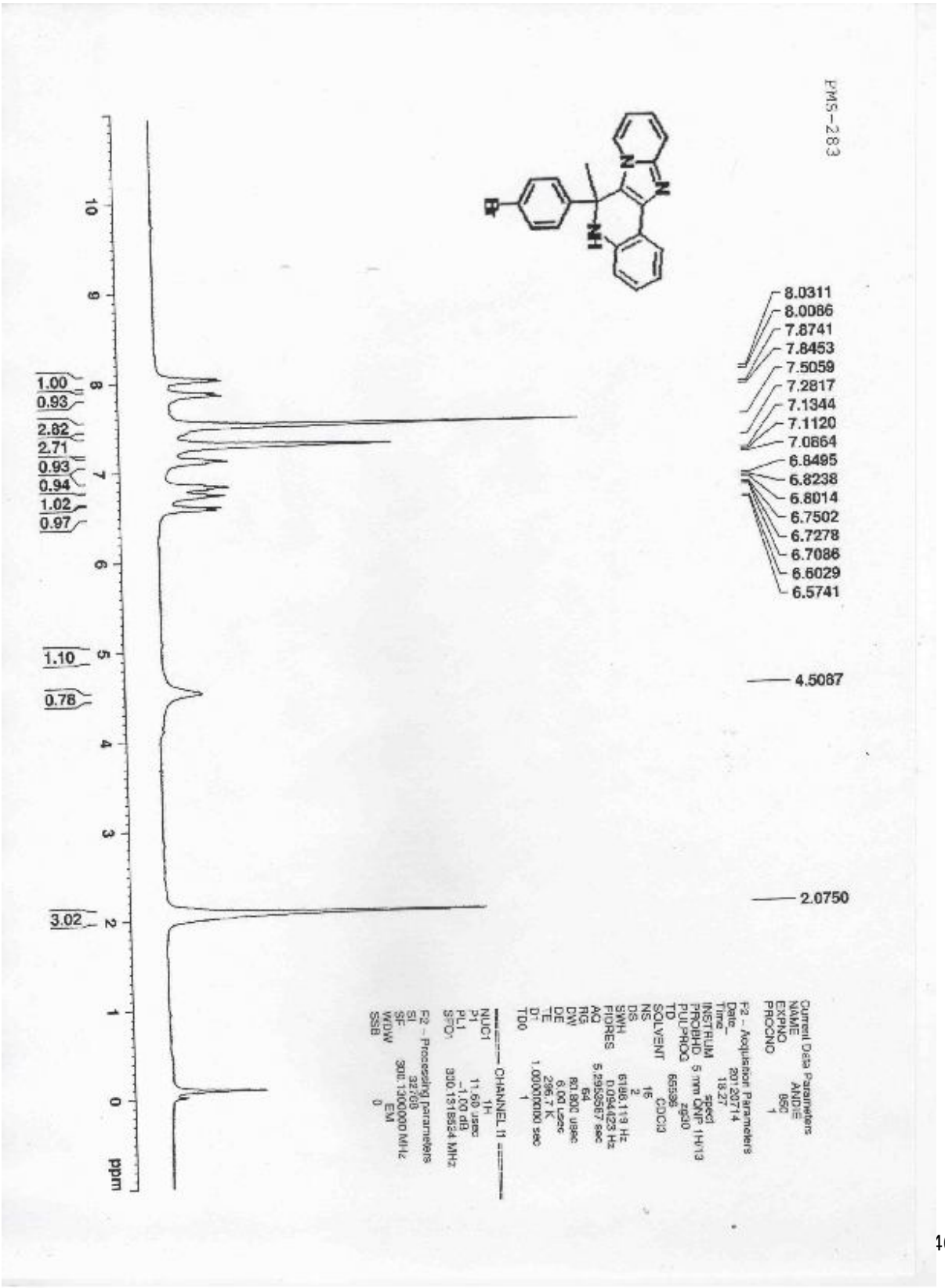

PMS-282
157.41
151.73
150.96
147.61
142.93
133.95
132.59
129.48
129.18
128.90
127.25
124.23
121.99
121.76
119.92
117.94
117.60
84.77
64.11
83.45
63.30
46.02
45.60
45.18
44.77
44.35
43.93
43.51
30.99
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

6-Methyl-6-(4-methoxy-phenyl)-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorene (11c):

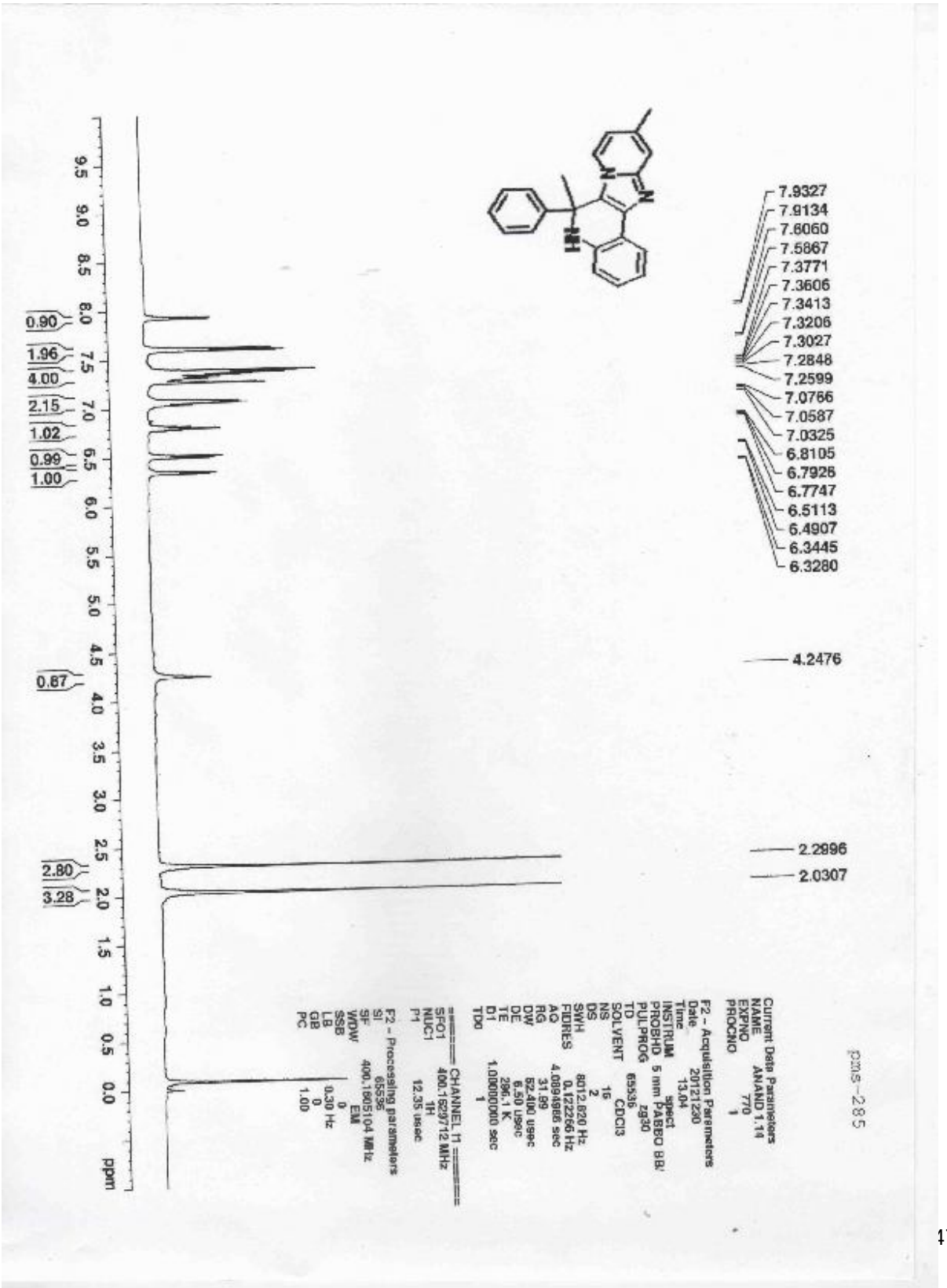


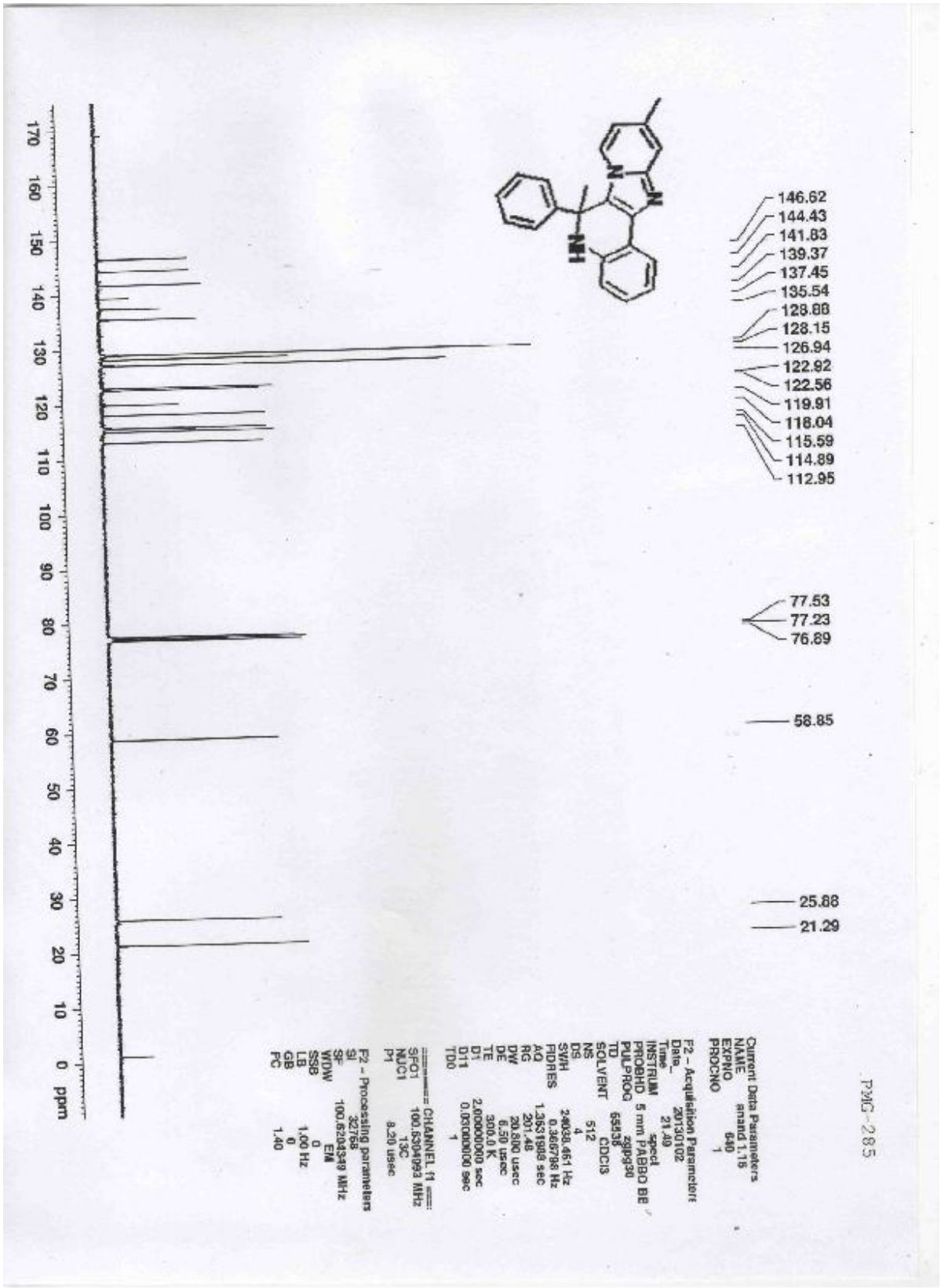

OMe
N
NH
163.61
149.85
147.74
143.67
141.67
141.46
136.12
133.90
132.74
129.69
128.70
127.87
127.03
125.40
121.22
120.97
119.11
118.64
117.88
117.52
84.10
83.44
82.79
62.66
59.96
45.96
45.54
45.13
44.71
44.29
43.87
43.46
27.28
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

6-Methyl-6-(4-bromo-phenyl)-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorene (11d):

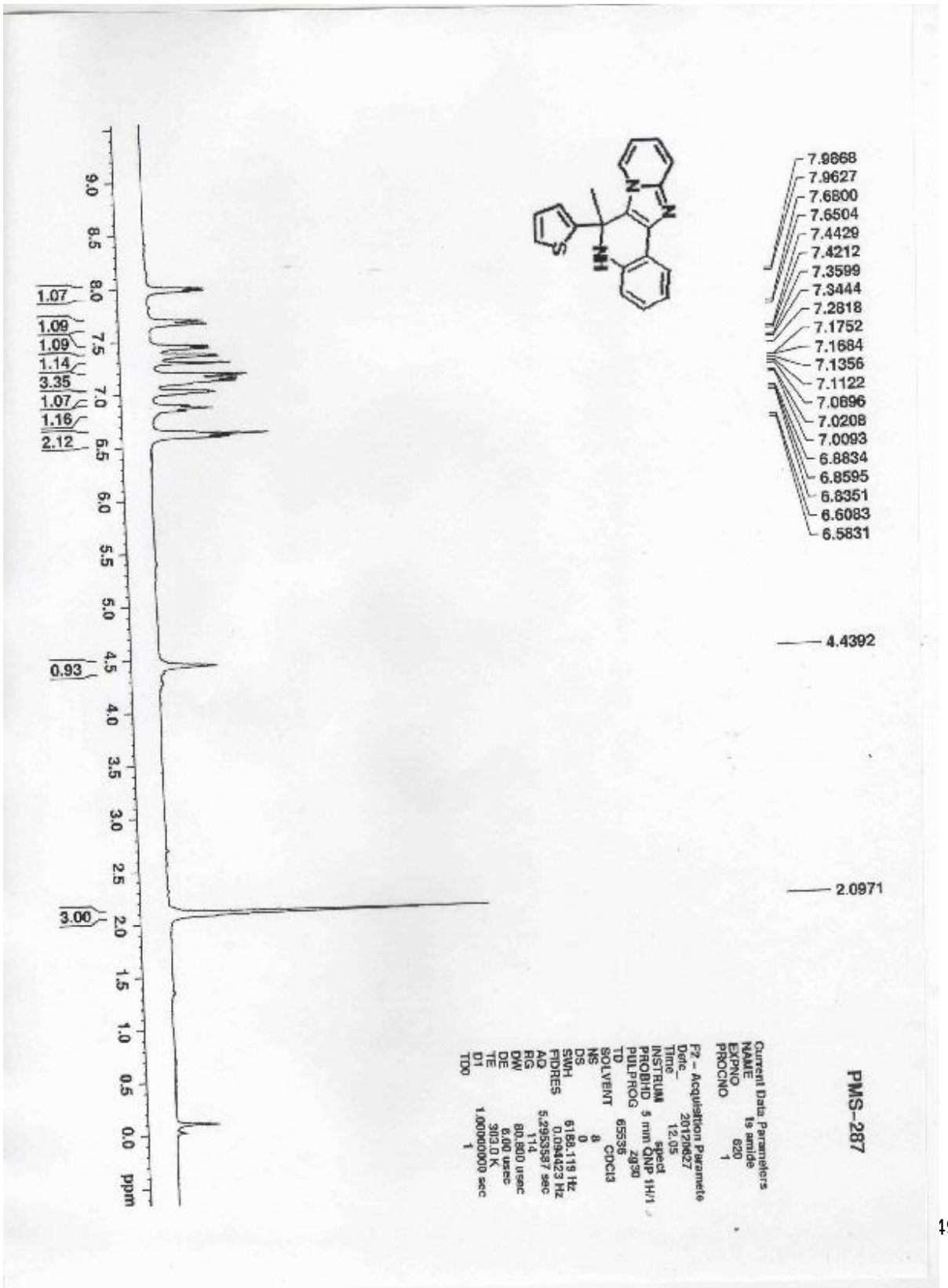


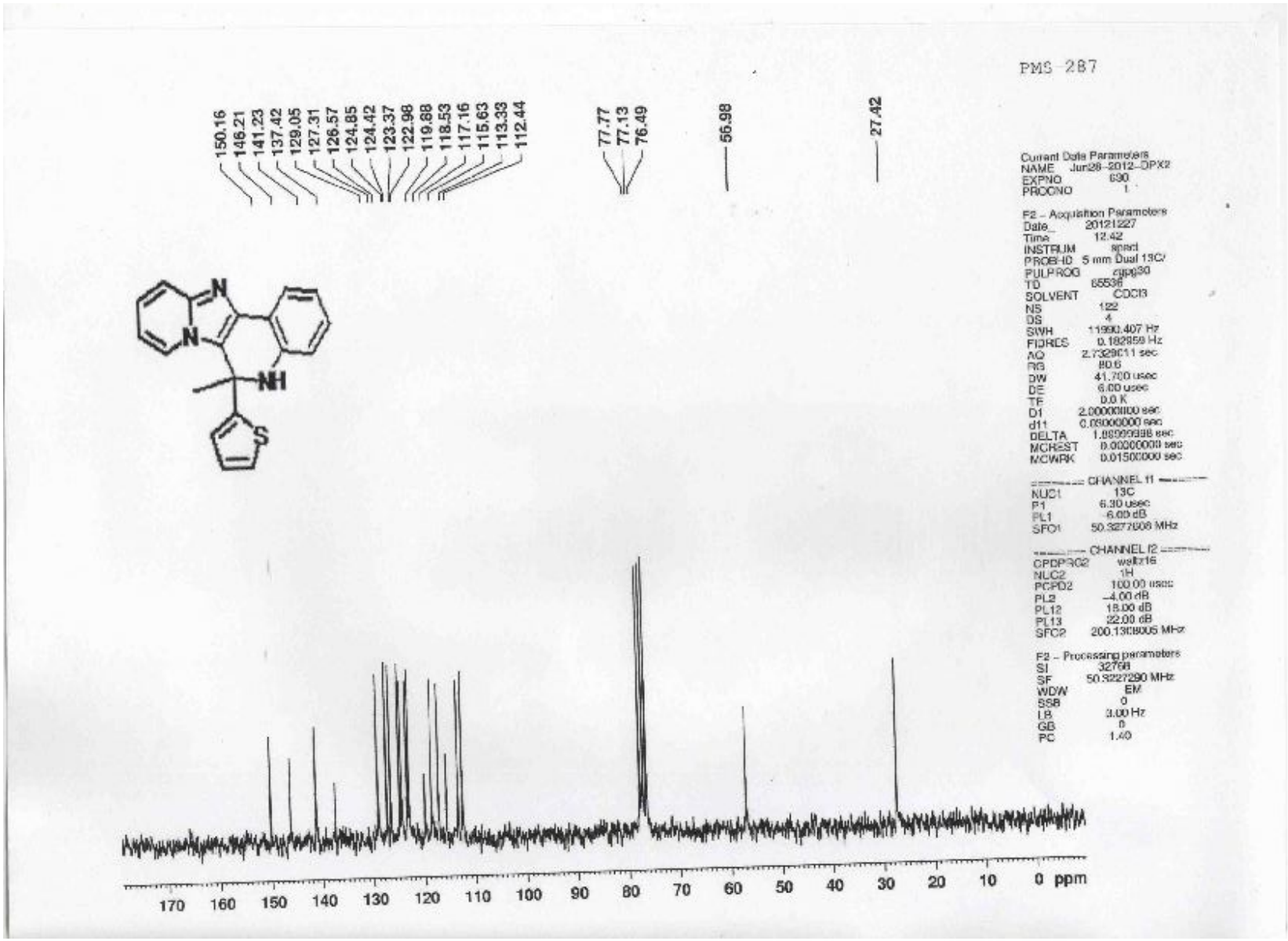
6-Methyl-6-(phenyl)-5,6-dihydro-9-methyl-5,6b,11-triaza-benzo[a]fluorene (11e):



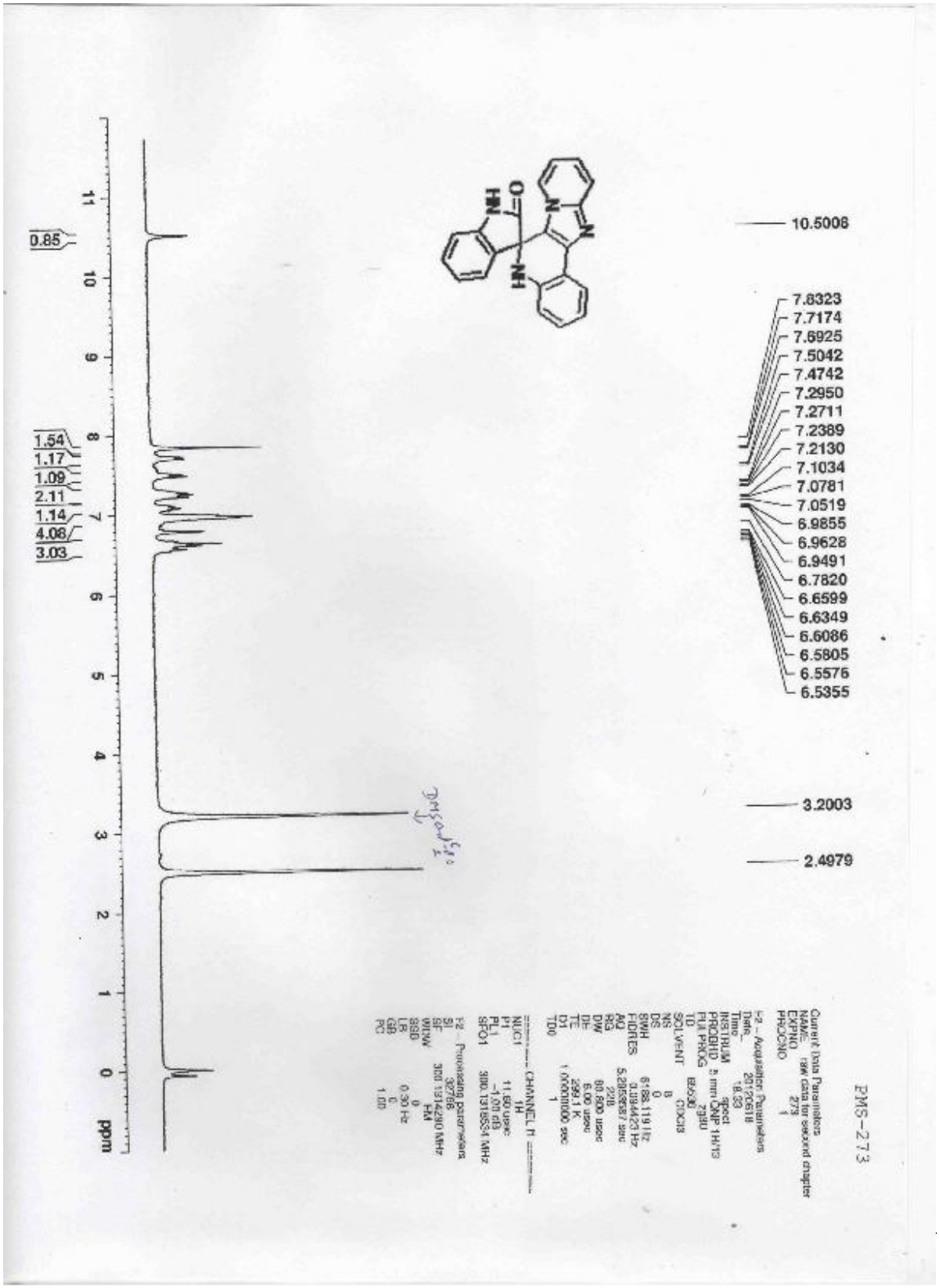

146.62
144.43
141.83
139.37
137.45
135.54
128.88
128.15
126.94
122.92
122.56
119.91
118.04
115.59
114.89
112.95
77.53
77.23
76.89
58.85
25.88
21.29
PMG-285
Current Data Parameters
NAME anand 1.15
EXPNO 640
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130102
Time 21.49
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366788 Hz
AQ 1.3631988 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6304993 MHz
NUC1 13C
P1 9.20 usec
F2 - Processing parameters
SI 32768
SF 100.6204349 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

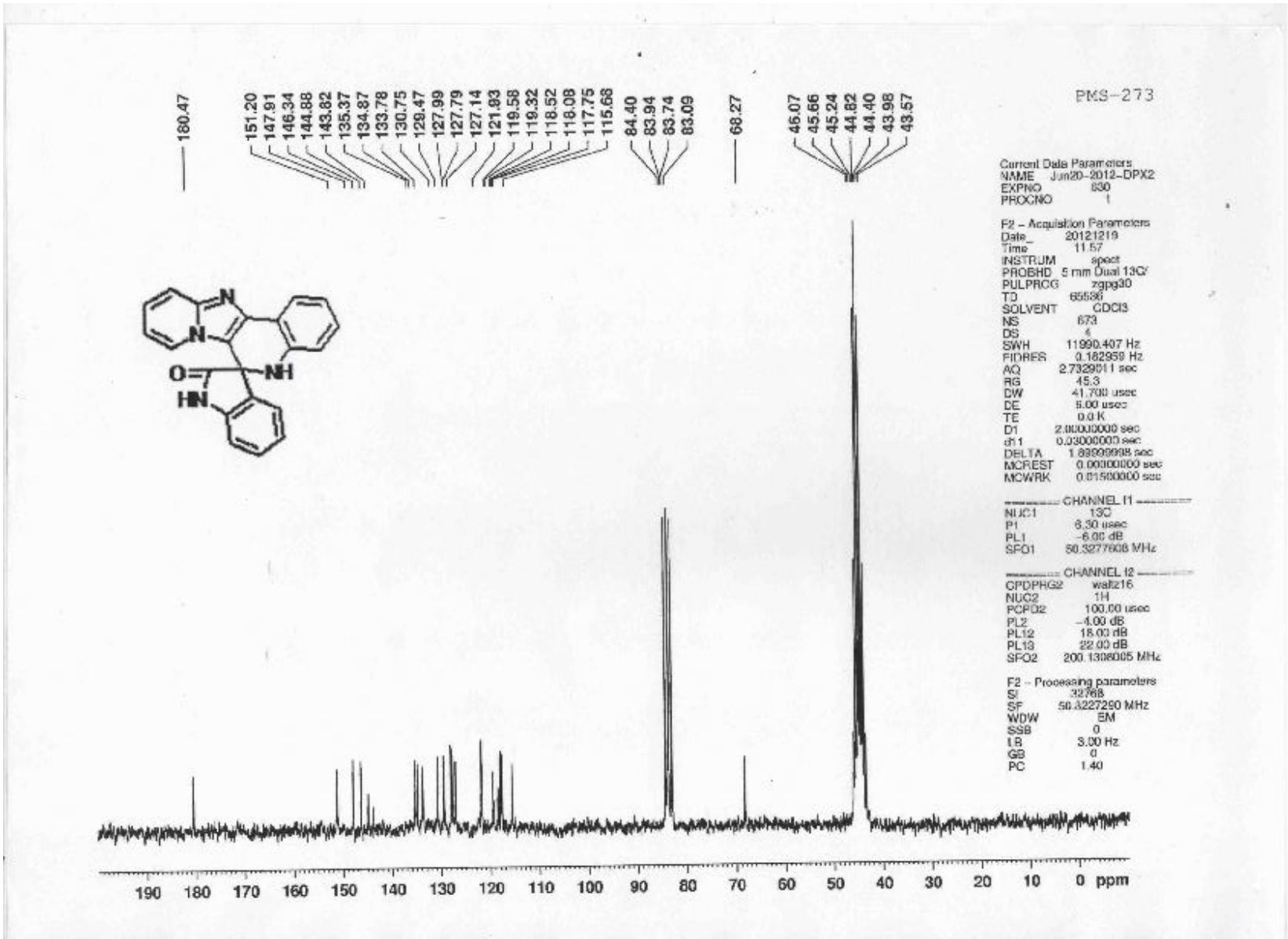
6-Methyl-6-(2-thiophenyl)-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorene (11f):



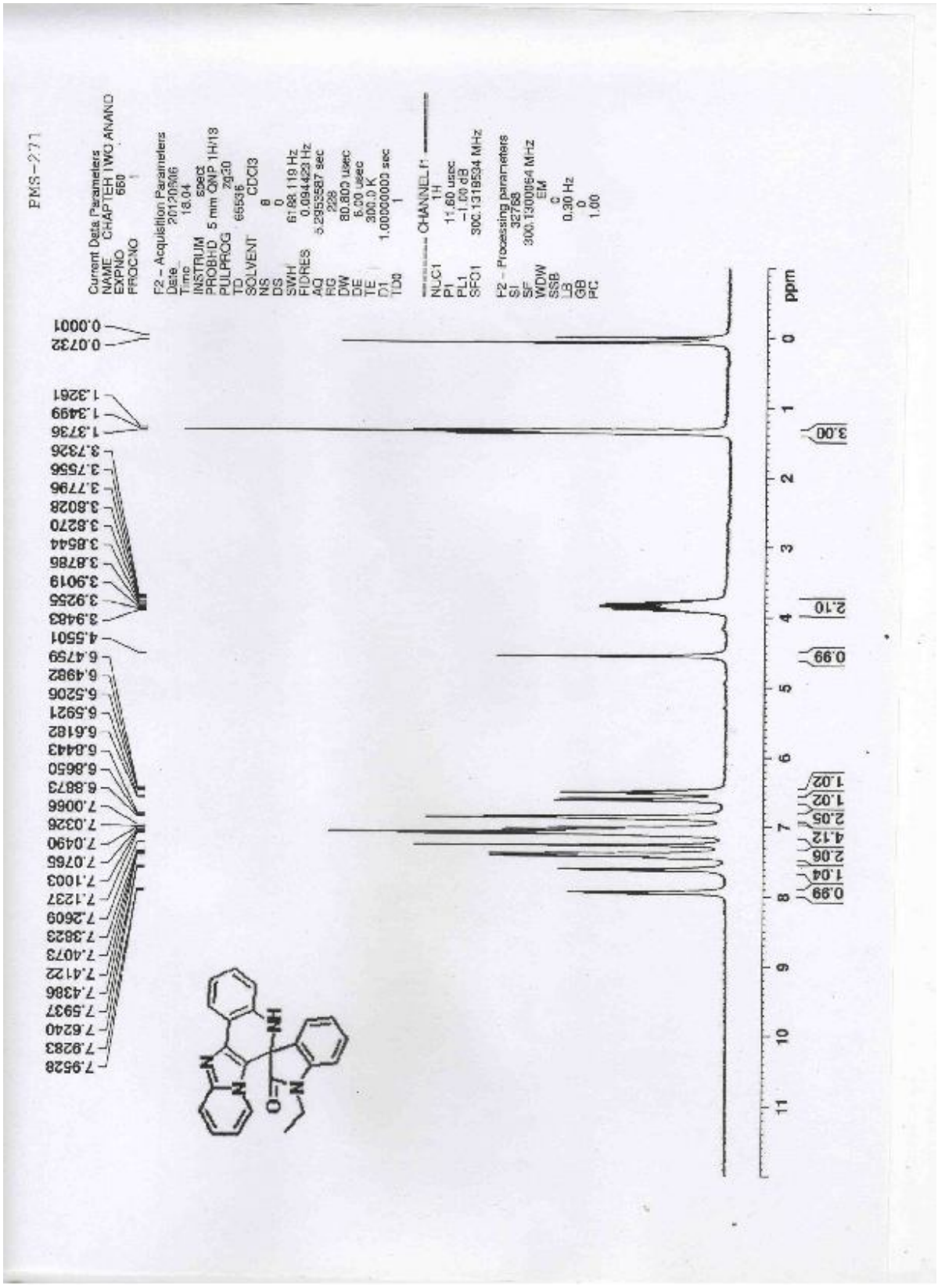

PMS-287
150.16
146.21
141.23
137.42
129.05
127.31
126.57
124.85
124.42
123.37
122.98
119.88
118.53
117.16
115.63
113.33
112.44
77.77
77.13
76.49
55.98
27.42
N
N
NH
S
Current Data Parameters
NAME Jun28-2012-DPX2
EXPNO 630
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121227
Time 12.42
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 122
DS 4
SWH 11990.407 Hz
FIDRES 0.182859 Hz
AQ 2.7329011 sec
RG 80.6
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277006 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

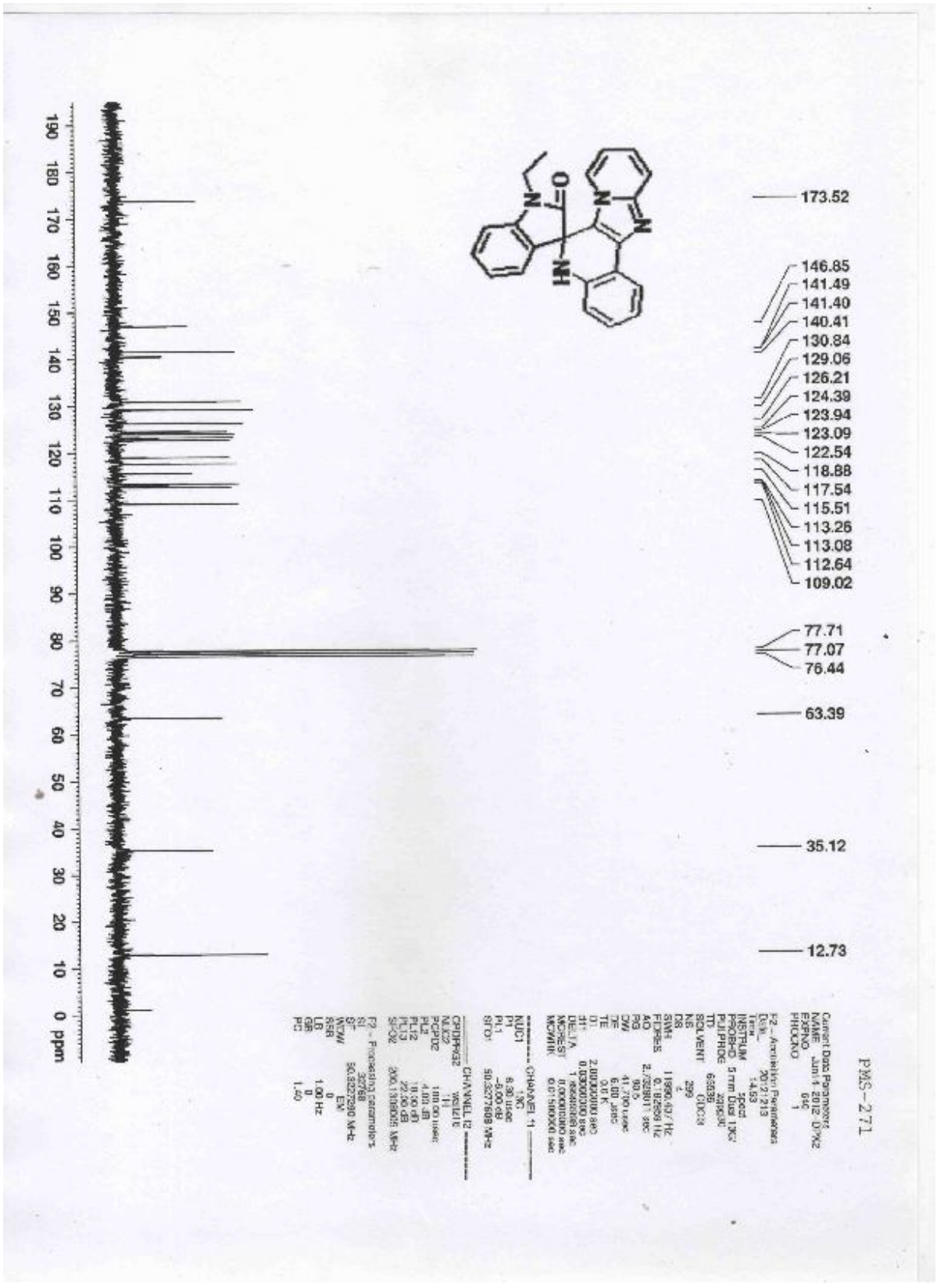
6-Spiro-indol-2(1H)-one-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorine (13a):



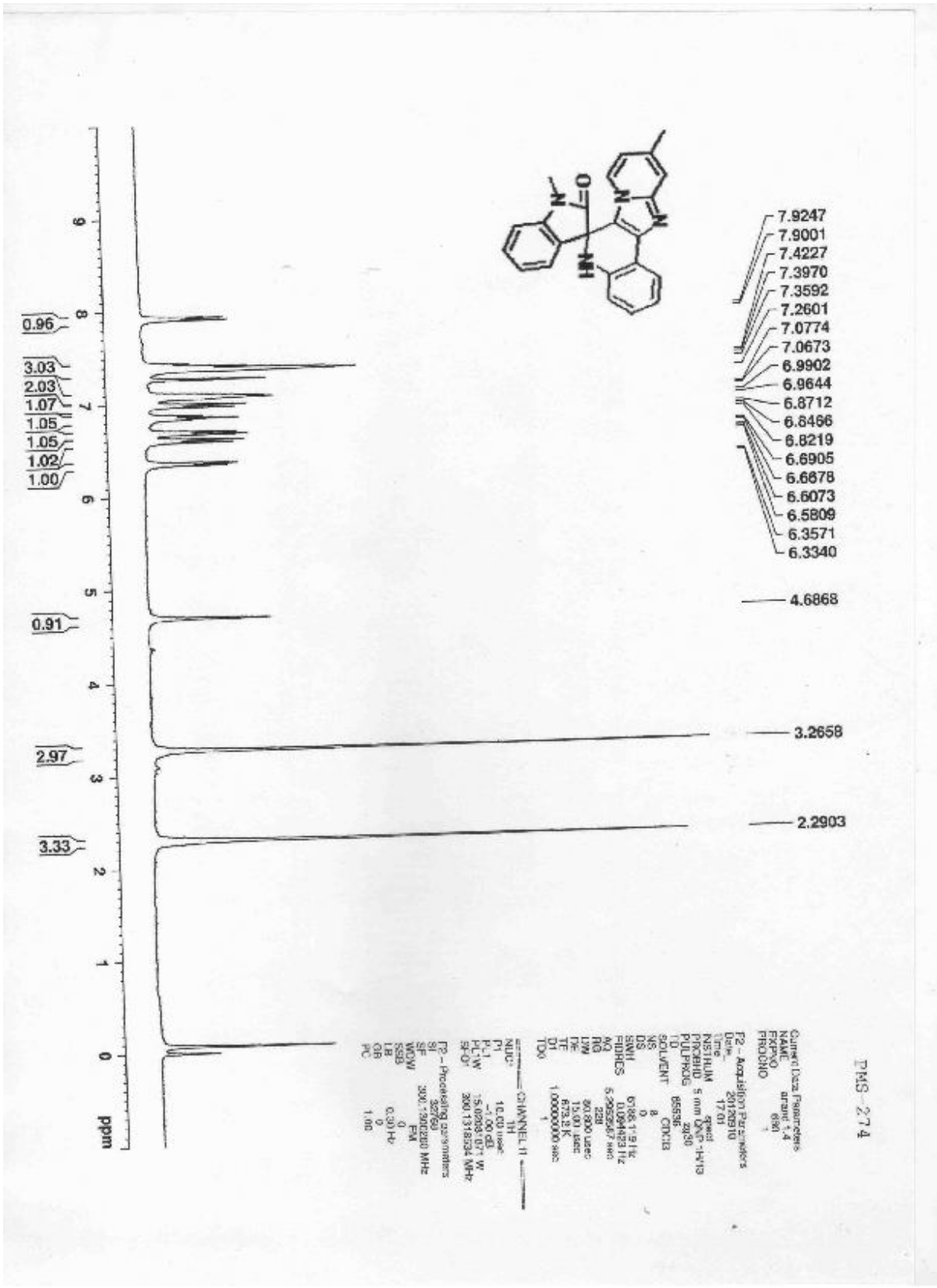

PMS-273
180.47
151.20
147.91
146.34
144.88
143.82
135.37
134.87
133.78
130.75
129.47
127.99
127.79
127.14
121.83
119.58
119.32
118.52
118.08
117.75
115.68
84.40
83.94
83.74
83.09
68.27
46.07
45.66
45.24
44.82
44.40
43.98
43.57
Current Data Parameters
NAME Jan20-2012-DPX2
EXPNO 830
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121219
Time 11.57
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 673
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329911 sec
RG 45.3
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.03 dB
PL13 22.00 dB
SFO2 200.1306005 MHz
F2 - Processing parameters
SI 32768
SF 50.3227290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

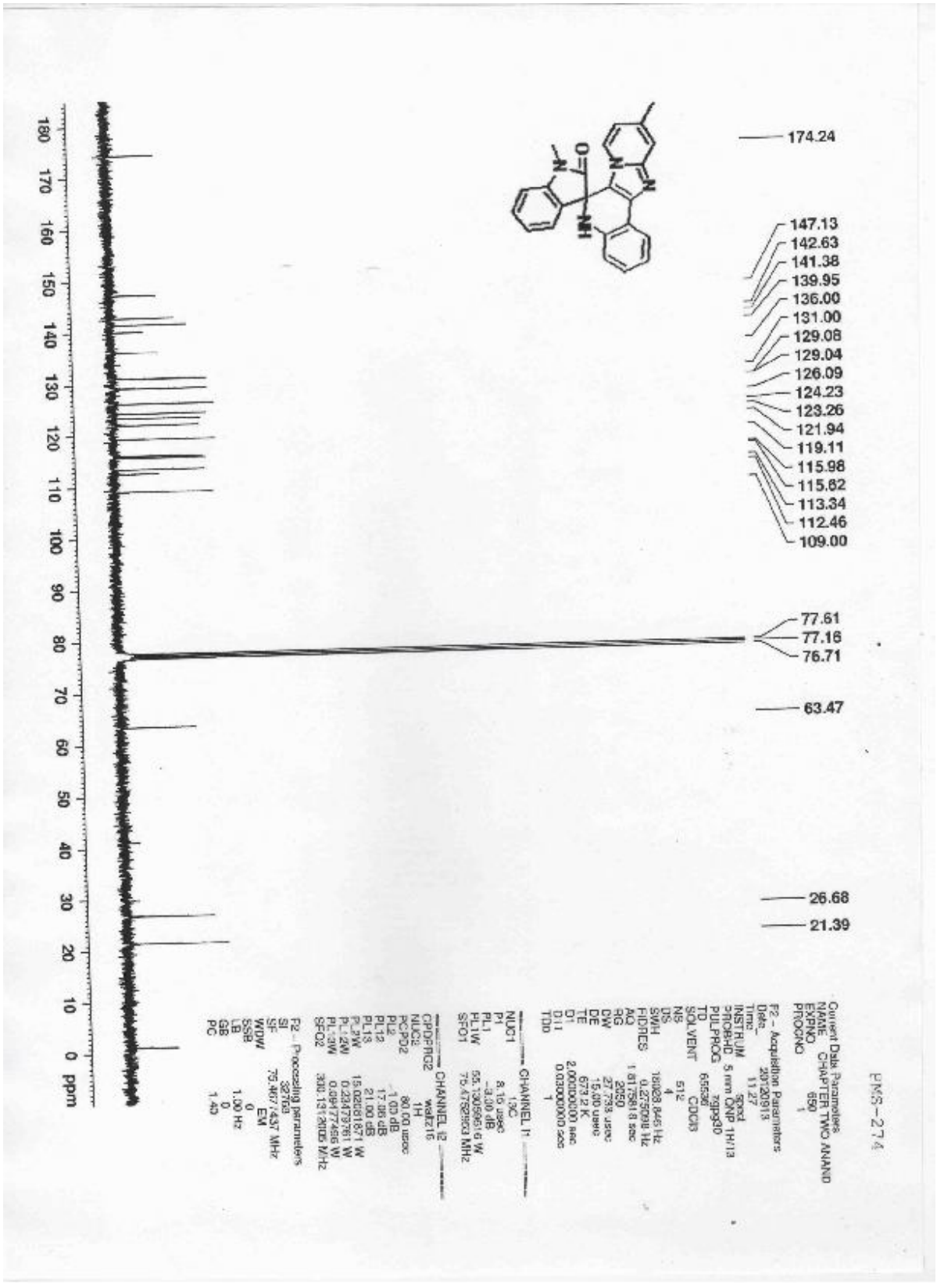
6-Spiro-indol-2(N-ethyl)-one-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorine (13b):



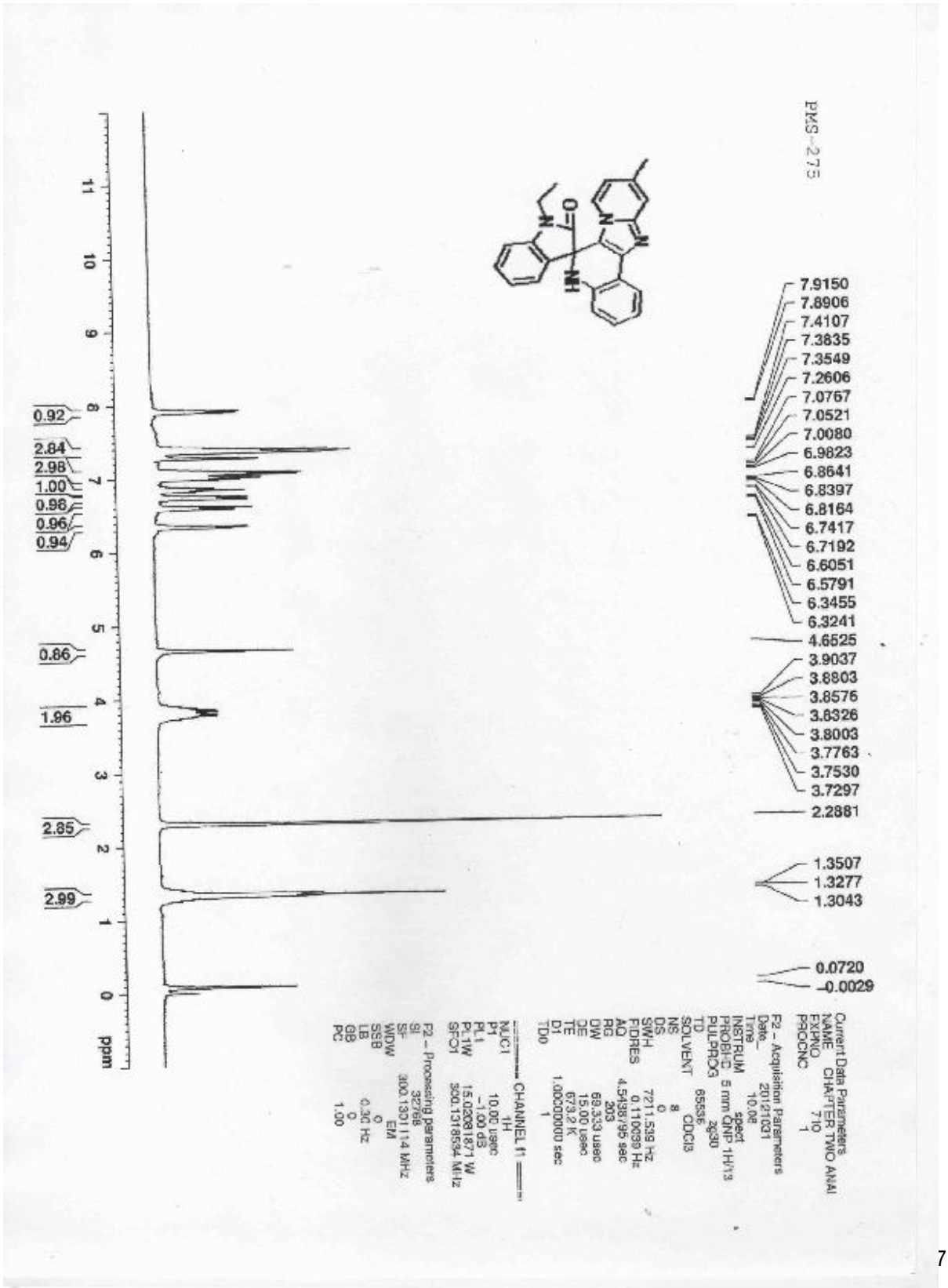

PMS-271
173.52
146.85
141.49
141.40
140.41
130.84
129.06
126.21
124.39
123.94
123.09
122.54
118.88
117.54
115.51
113.26
113.08
112.64
109.02
77.71
77.07
76.44
63.39
35.12
12.73
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

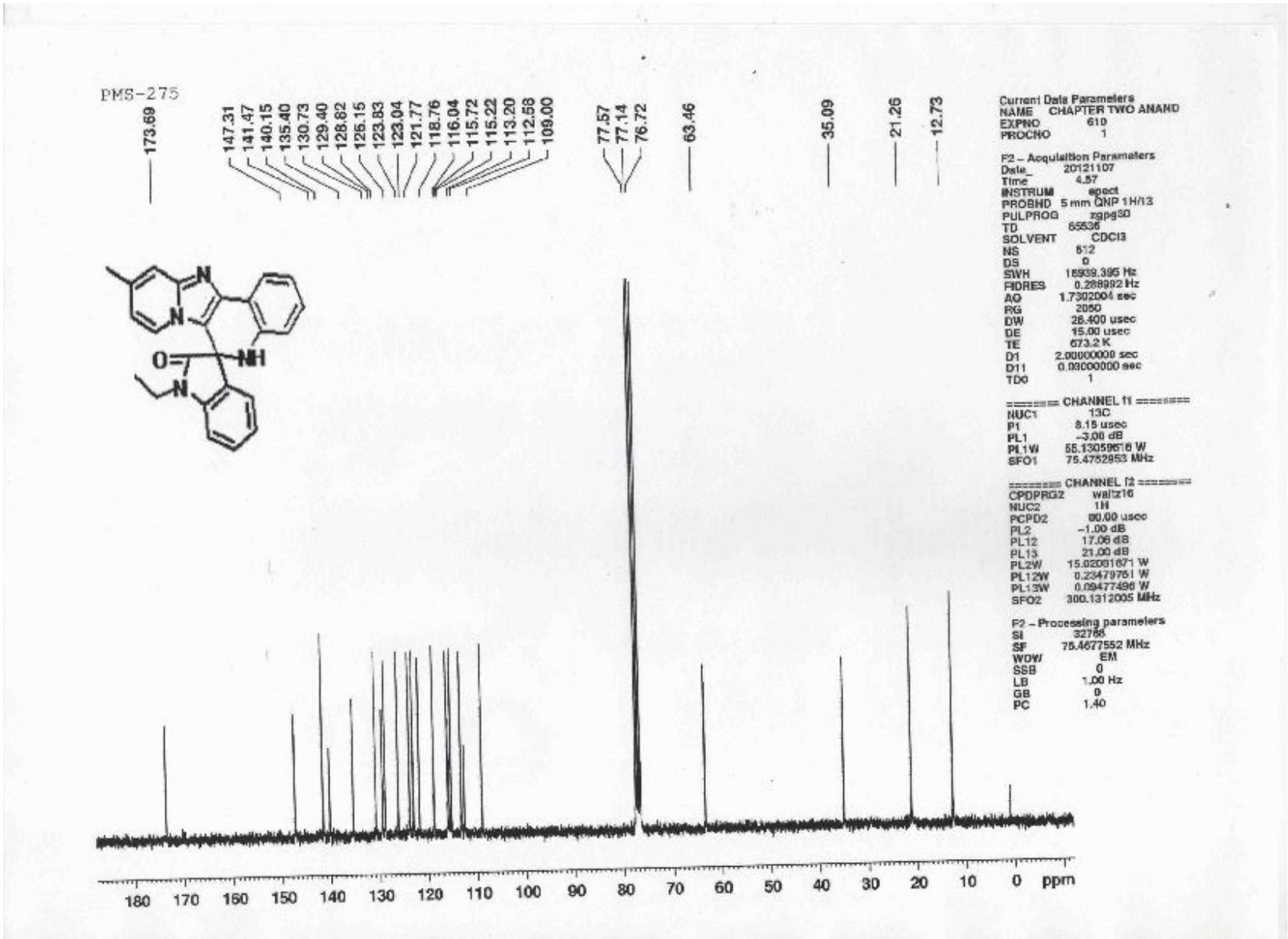
6-Spiro-indol-2(N-methyl)-one-5,6-dihydro-9-methyl-5,6b,11-triaza-benzo[a]fluorine (13c):



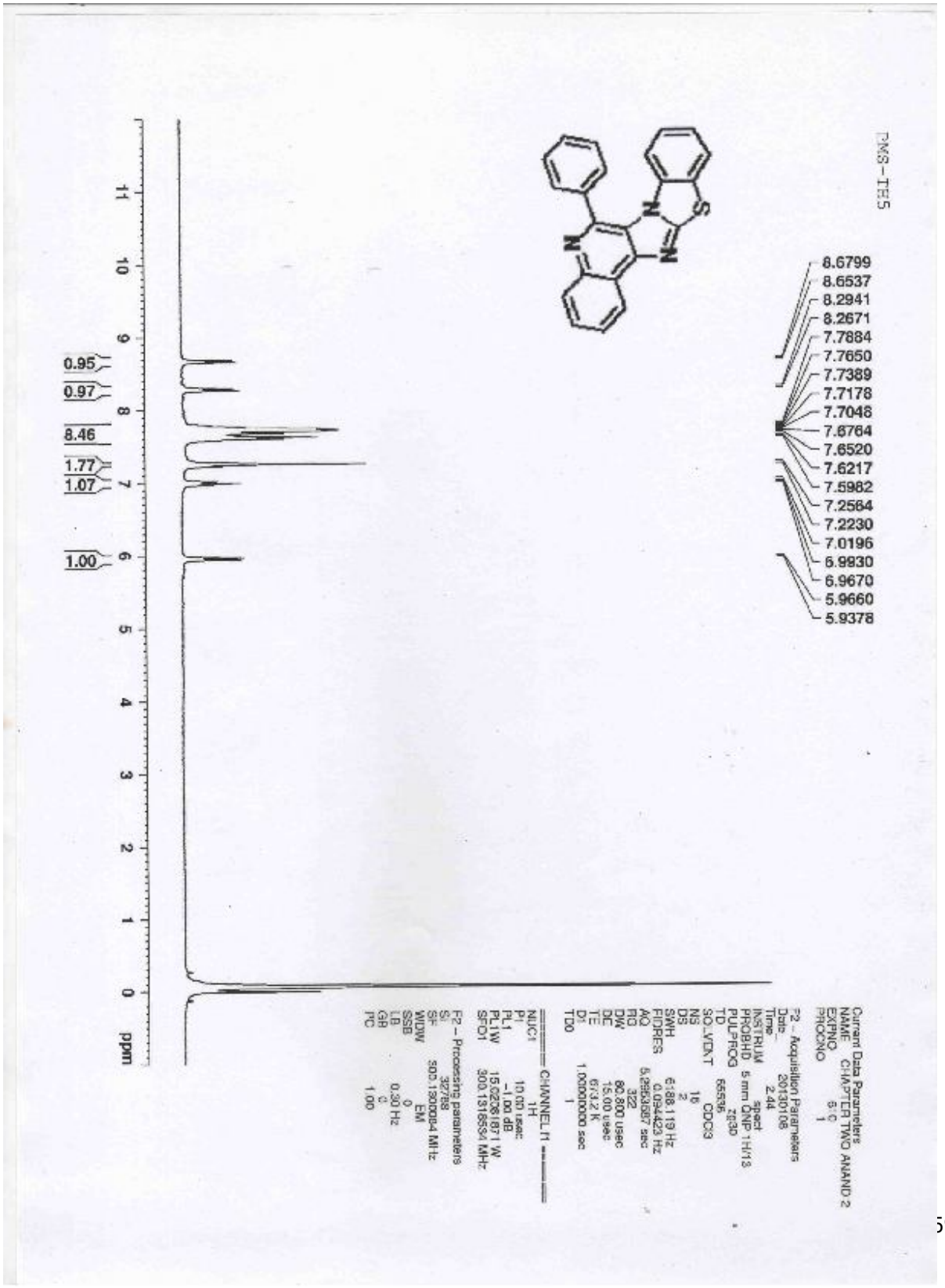

174.24
147.13
142.63
141.38
139.95
136.00
131.00
129.08
129.04
126.09
124.23
123.26
121.94
119.11
115.98
115.82
113.34
112.46
109.00
77.61
77.16
76.71
63.47
26.68
21.39
PMS-274
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 650
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120913
Time 11.27
INSTRUM spect
PROBHD 5 mm CNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275089 Hz
AQ 1.8175618 sec
RG 2050
DW 27.733 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.13056616 W
SFO1 75.4752853 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.38 dB
PL13 21.00 dB
PL2W 15.02818671 W
PL12W 0.23479761 W
PL13W 0.09177466 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677437 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

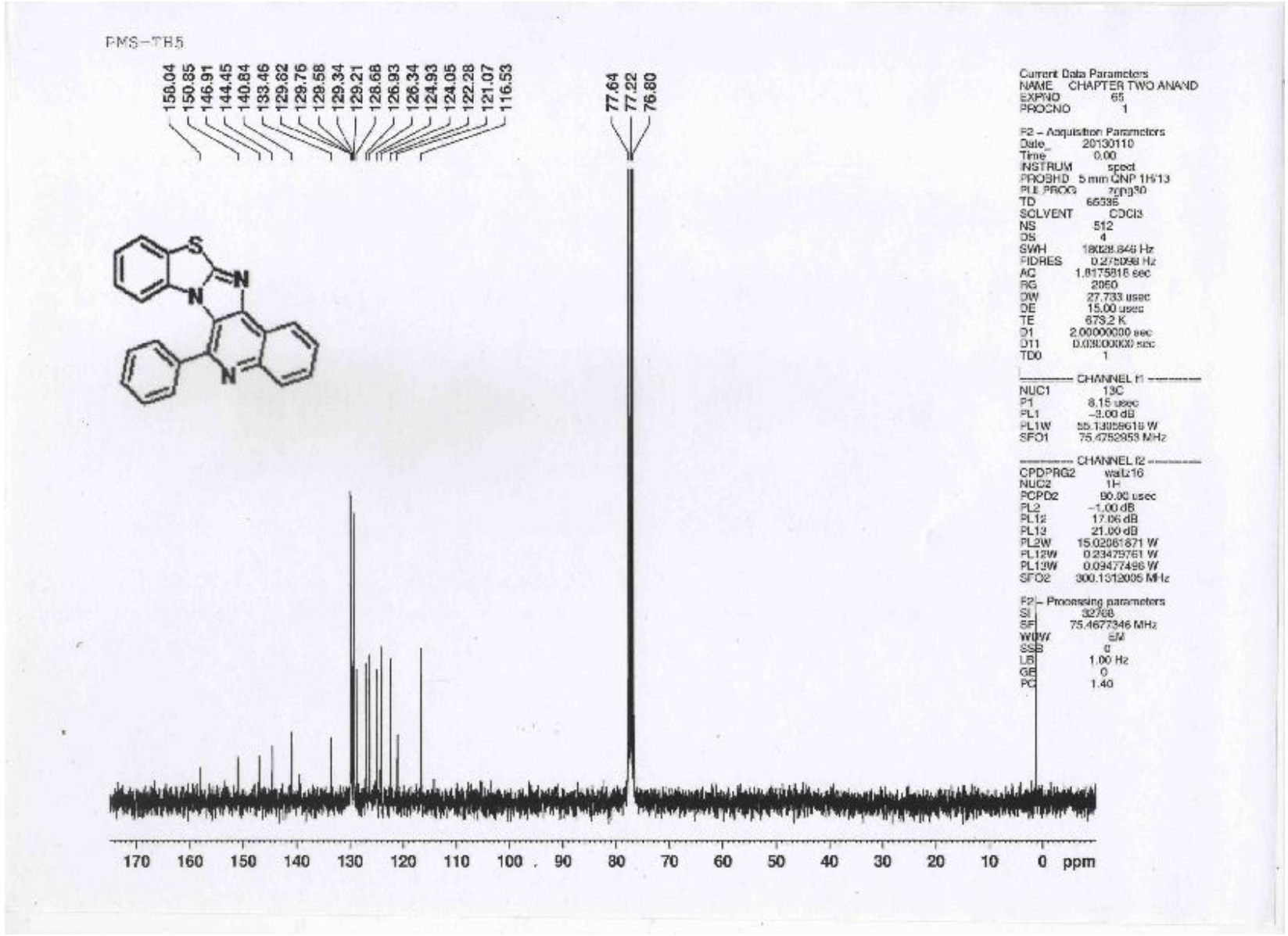
6-Spiro-indol-2(N-ethyl)-one-5,6-dihydro-9-methyl-5,6b,11-triaza-benzo[a]fluorine (13d):



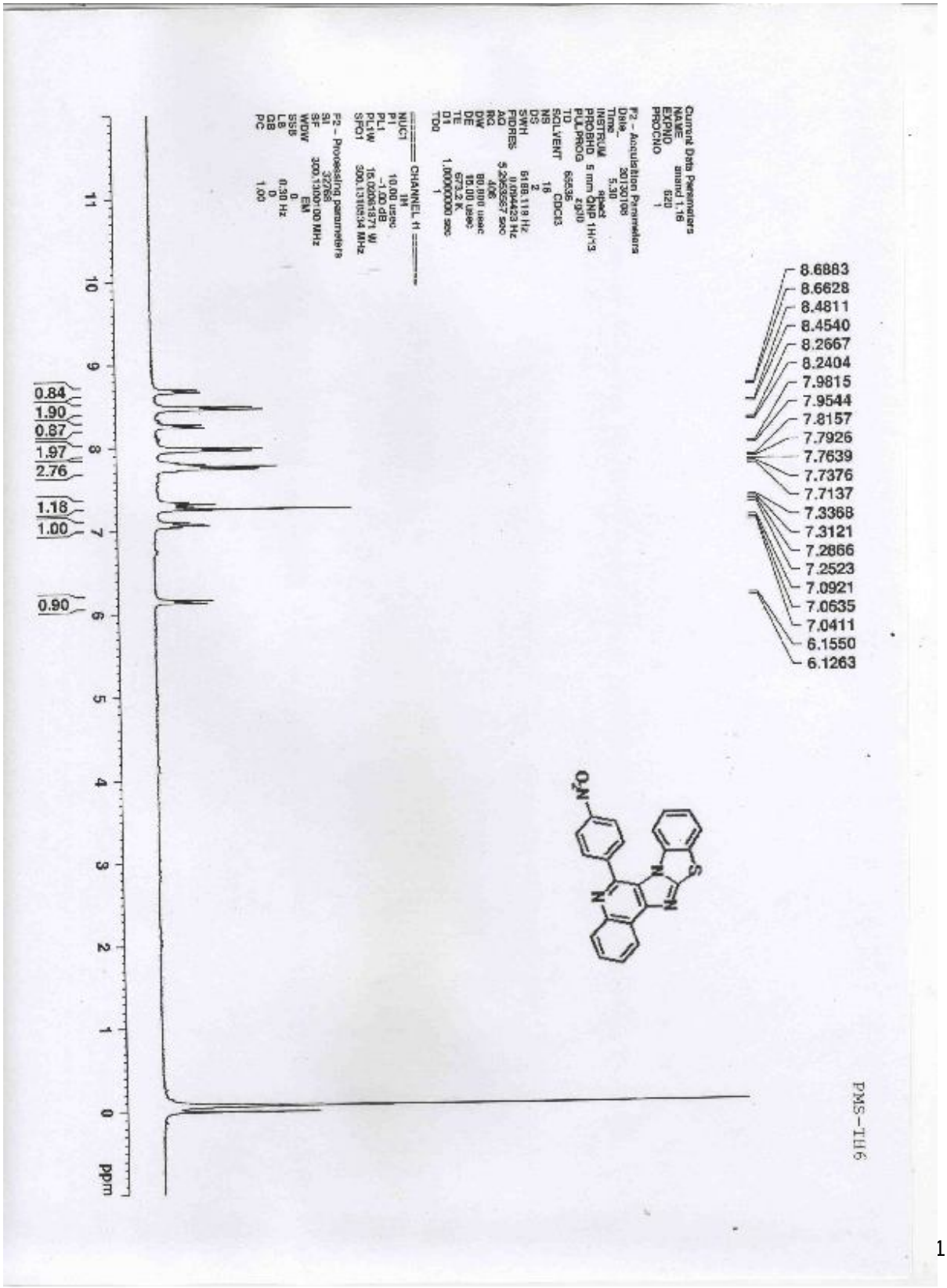

PMS-275
173.69
147.31
141.47
140.15
135.40
130.73
129.40
128.82
125.15
123.83
123.04
121.77
118.76
116.04
115.72
115.22
113.20
112.58
109.00
77.57
77.14
76.72
63.46
35.09
21.26
12.73
O=
NH
N
N
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 610
PROCNO 1
F2 – Acquisition Parameters
Date_ 20121107
Time 4.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 18939.395 Hz
FIDRES 0.288992 Hz
AQ 1.7302004 sec
RG 2050
DW 26.400 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.13059616 W
SFO1 75.4752853 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 15.02001671 W
PL12W 0.23479761 W
PL13W 0.09477496 W
SFO2 300.1312005 MHz
F2 – Processing parameters
SI 32768
SF 75.4677552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

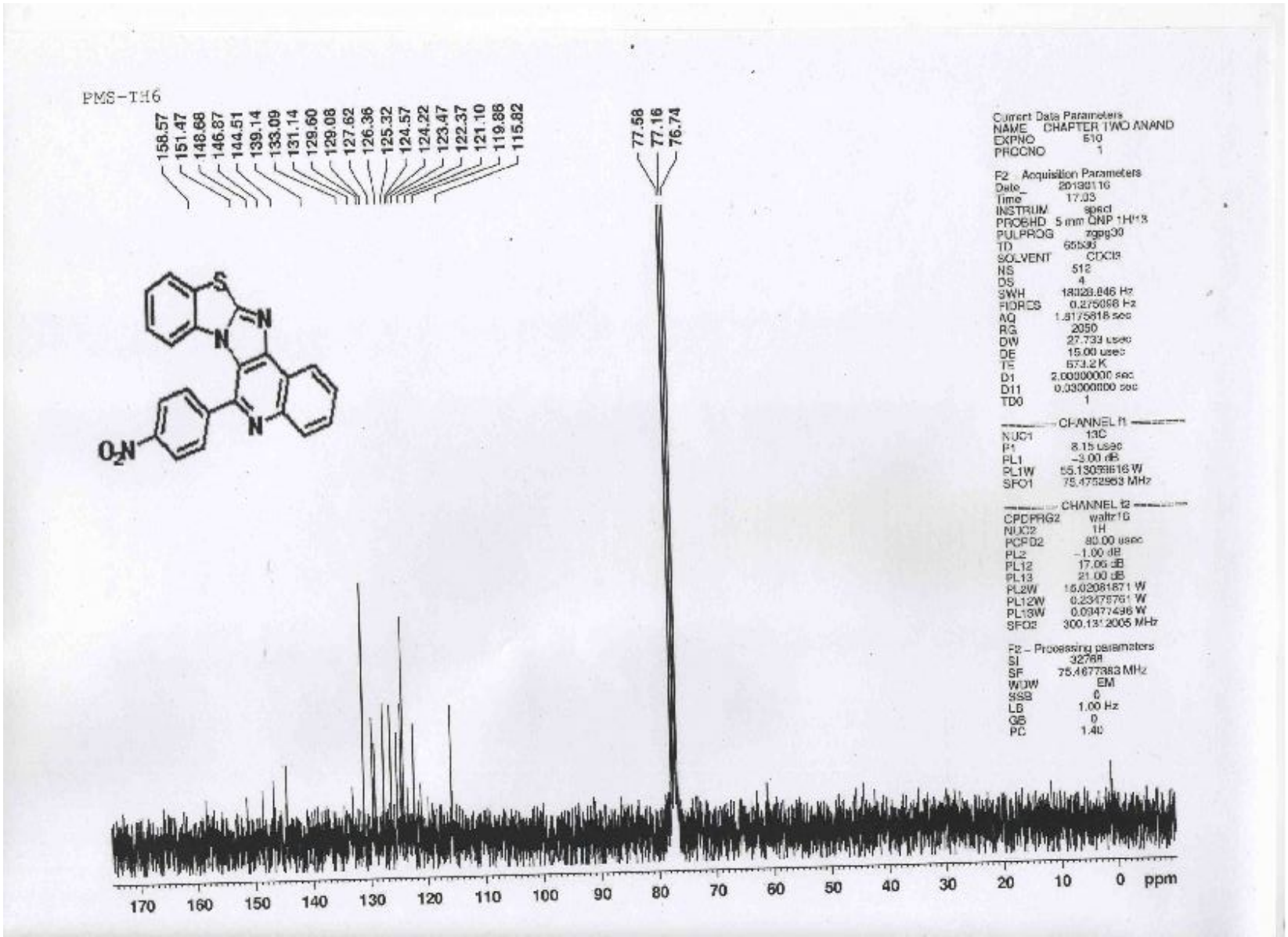
6-(Phenyl)-(11-S)-5,6b,12-triaza-benzo[a]fluorine (21a):



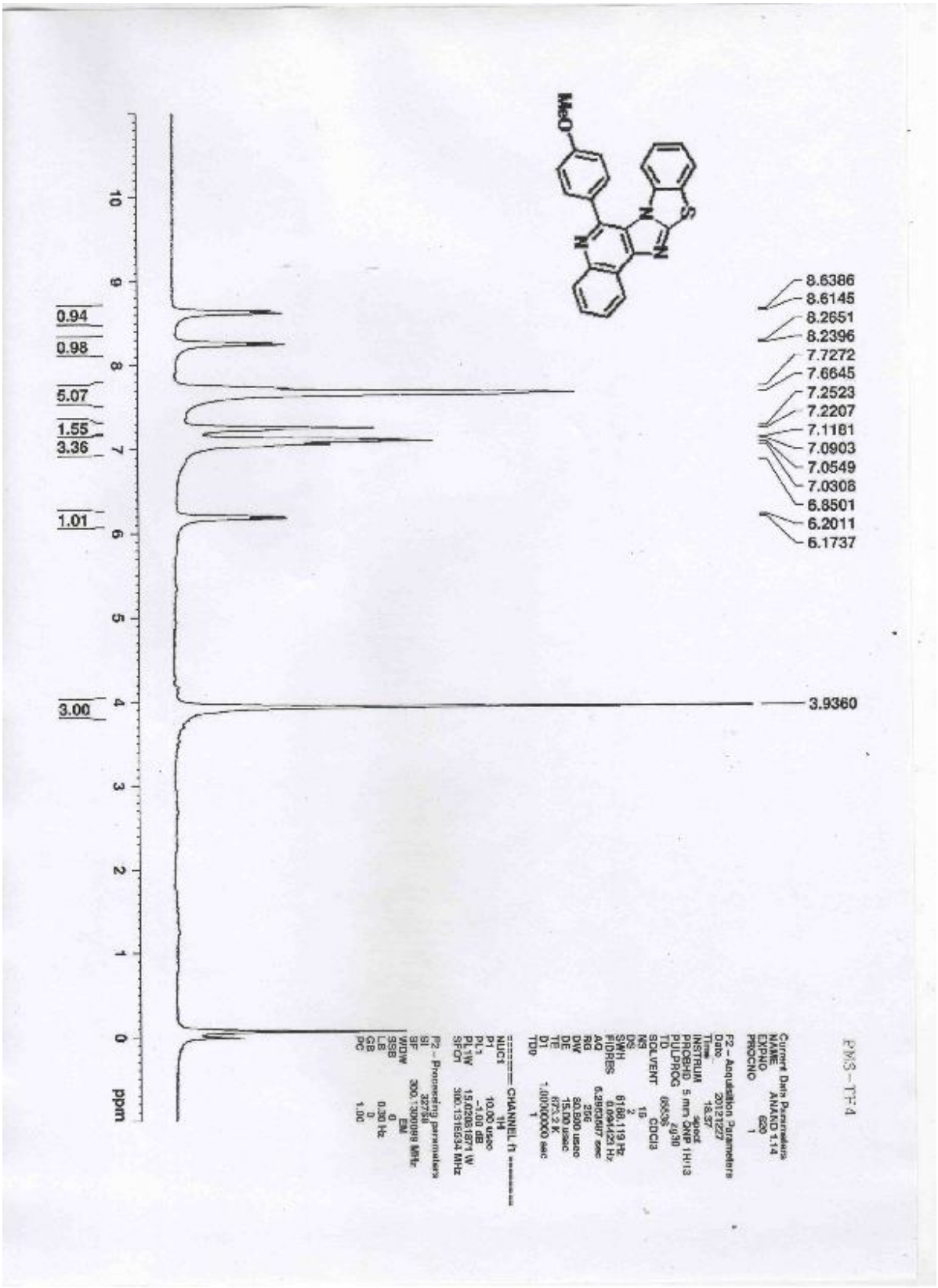

PMS-TH5
150.04
150.85
146.91
144.45
140.84
133.46
129.82
129.76
129.58
129.34
129.21
128.68
126.93
126.34
124.93
124.05
122.28
121.07
116.53
77.64
77.22
76.80
S
N
N
N
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 65
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130110
Time 0.00
INSTRUM spect
PROBHD 5 mm CNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AC 1.8175816 sec
RG 2050
DW 27.733 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.13058615 W
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 15.02061871 W
PL12W 0.23479761 W
PL13W 0.09477496 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677346 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

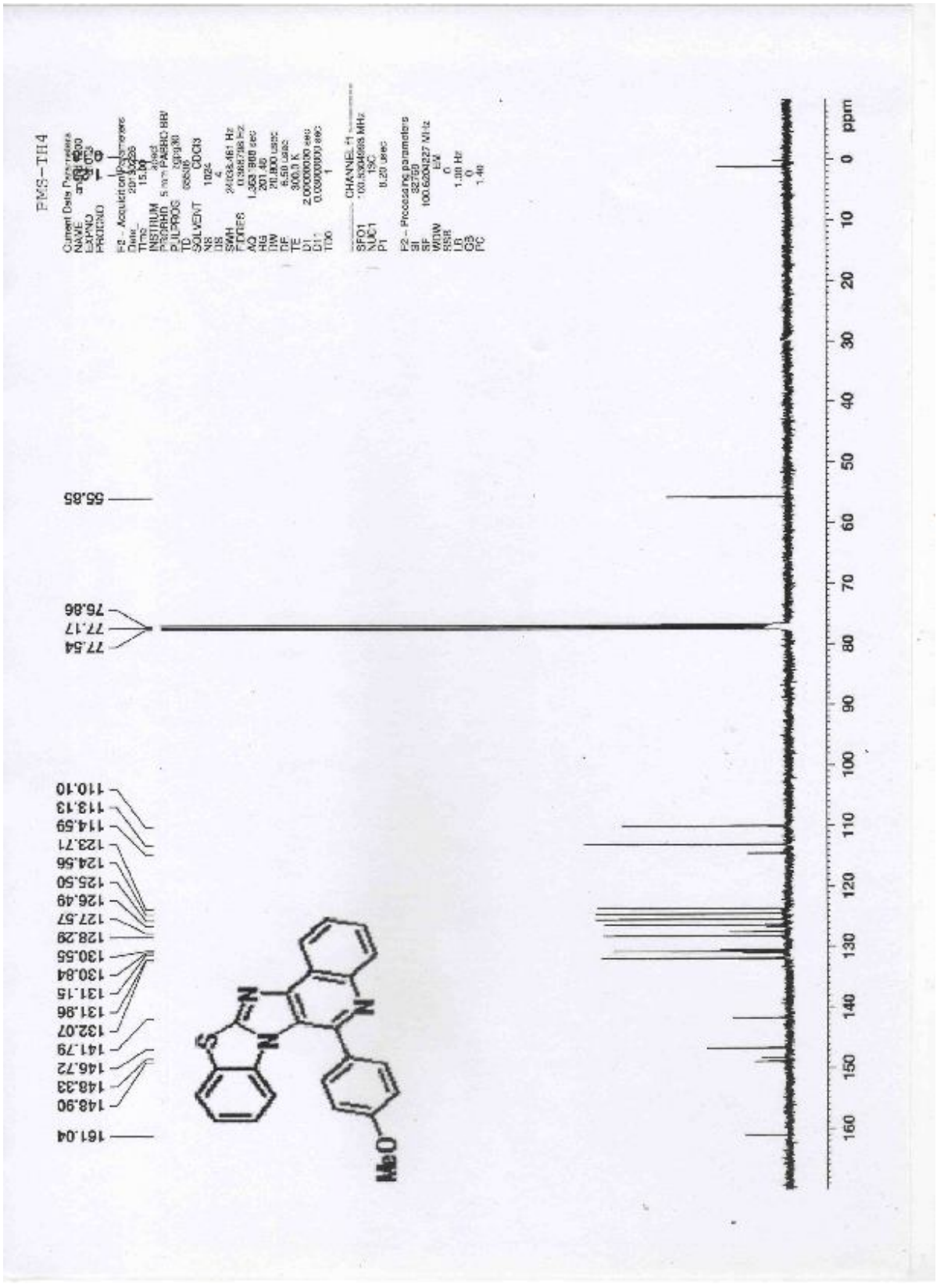
6-(4-nitro-Phenyl)-(11-S)-5,6b,12-triaza-benzo[a]fluorene (21b):



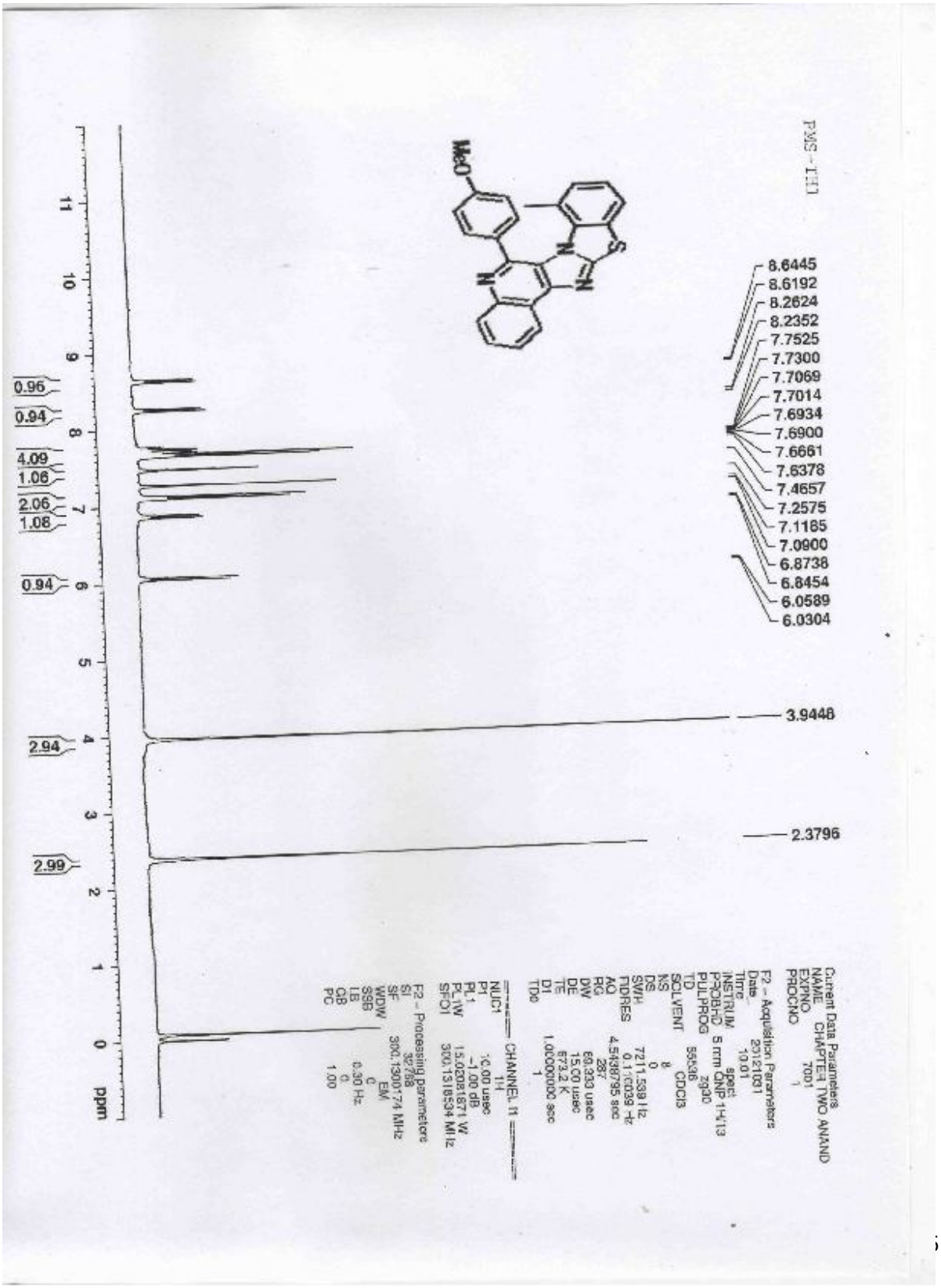

PMS-TH6
158.57
151.47
148.68
146.87
144.51
139.14
133.09
131.14
129.60
129.08
127.62
126.36
125.32
124.57
124.22
123.47
122.37
121.10
119.88
115.82
77.58
77.16
76.74
S
N
N
N
O2N
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 510
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130116
Time 17.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275068 Hz
AQ 1.8175616 sec
RG 2050
DW 27.733 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.13059616 W
SFO1 75.4752963 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 15.02081871 W
PL12W 0.23478761 W
PL13W 0.09477496 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677383 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

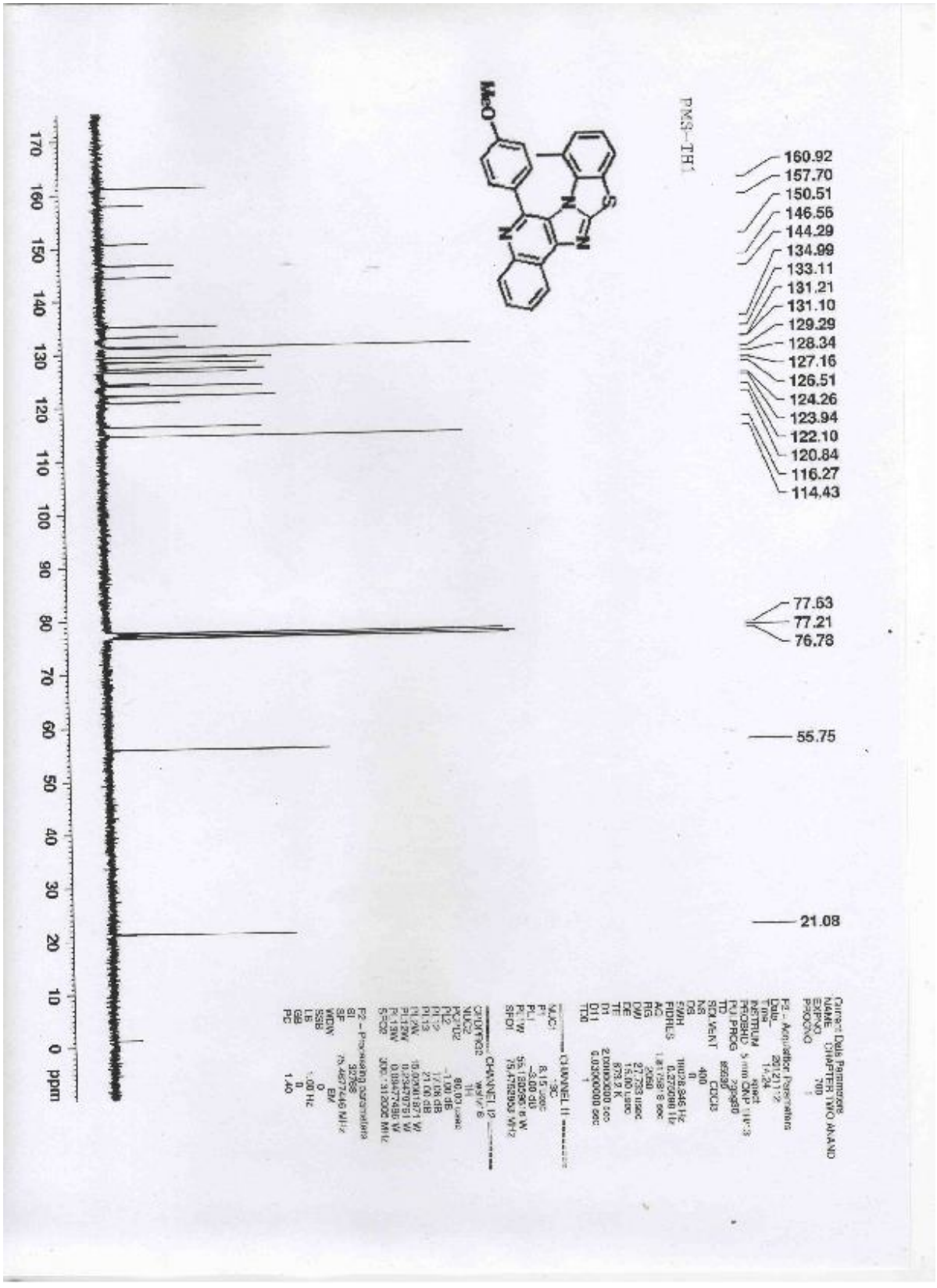
6-(4-Methoxy-Phenyl)-(11-S)-5,6b,12-triaza-benzo[a]fluorene (21c):



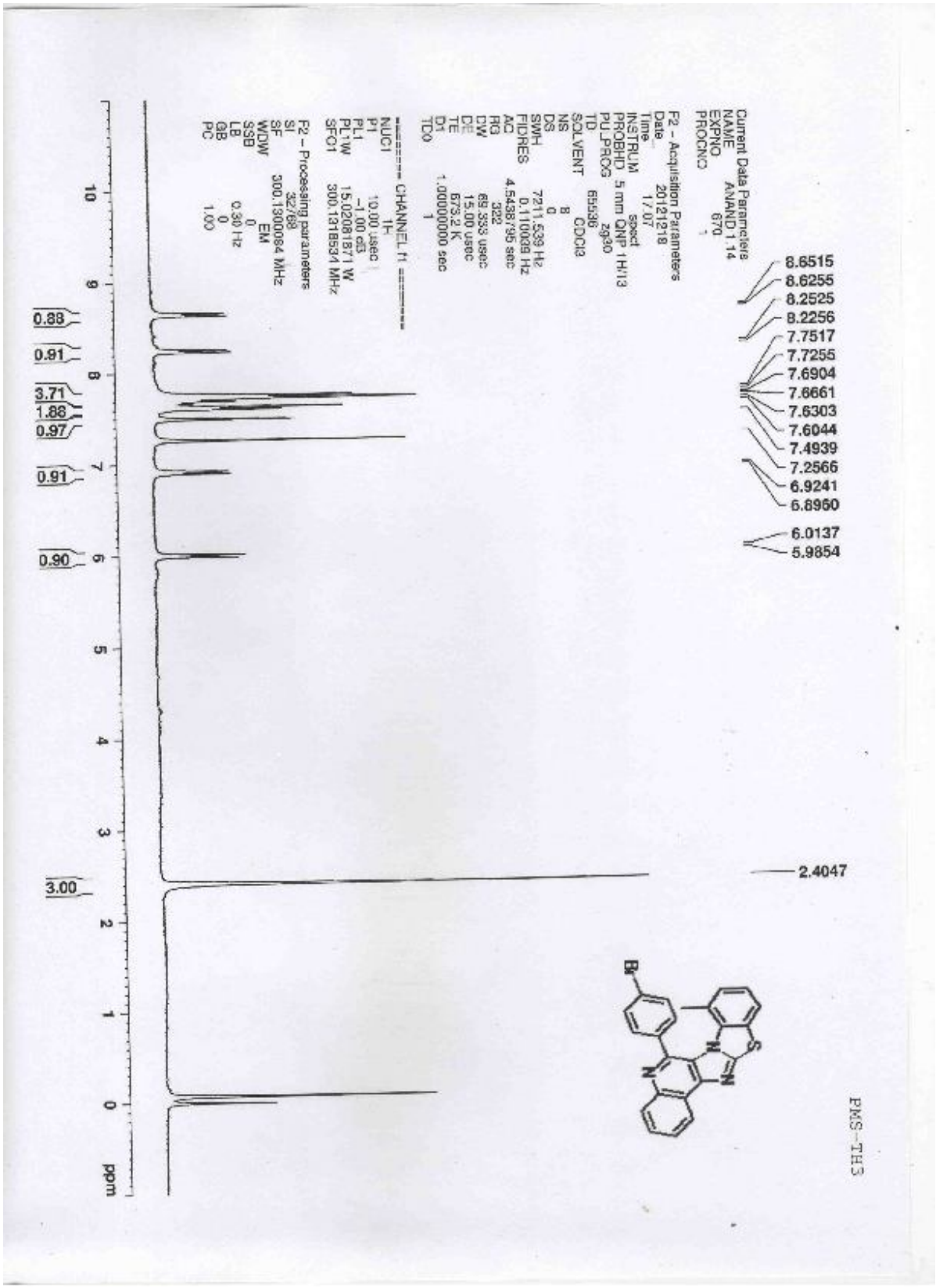

FMS-TH4
55.85
76.86
77.17
77.54
110.10
113.13
114.59
123.71
124.56
125.50
126.49
127.57
128.29
130.55
130.84
131.15
131.86
132.07
141.79
146.72
148.33
148.90
161.04
MeO
ppm

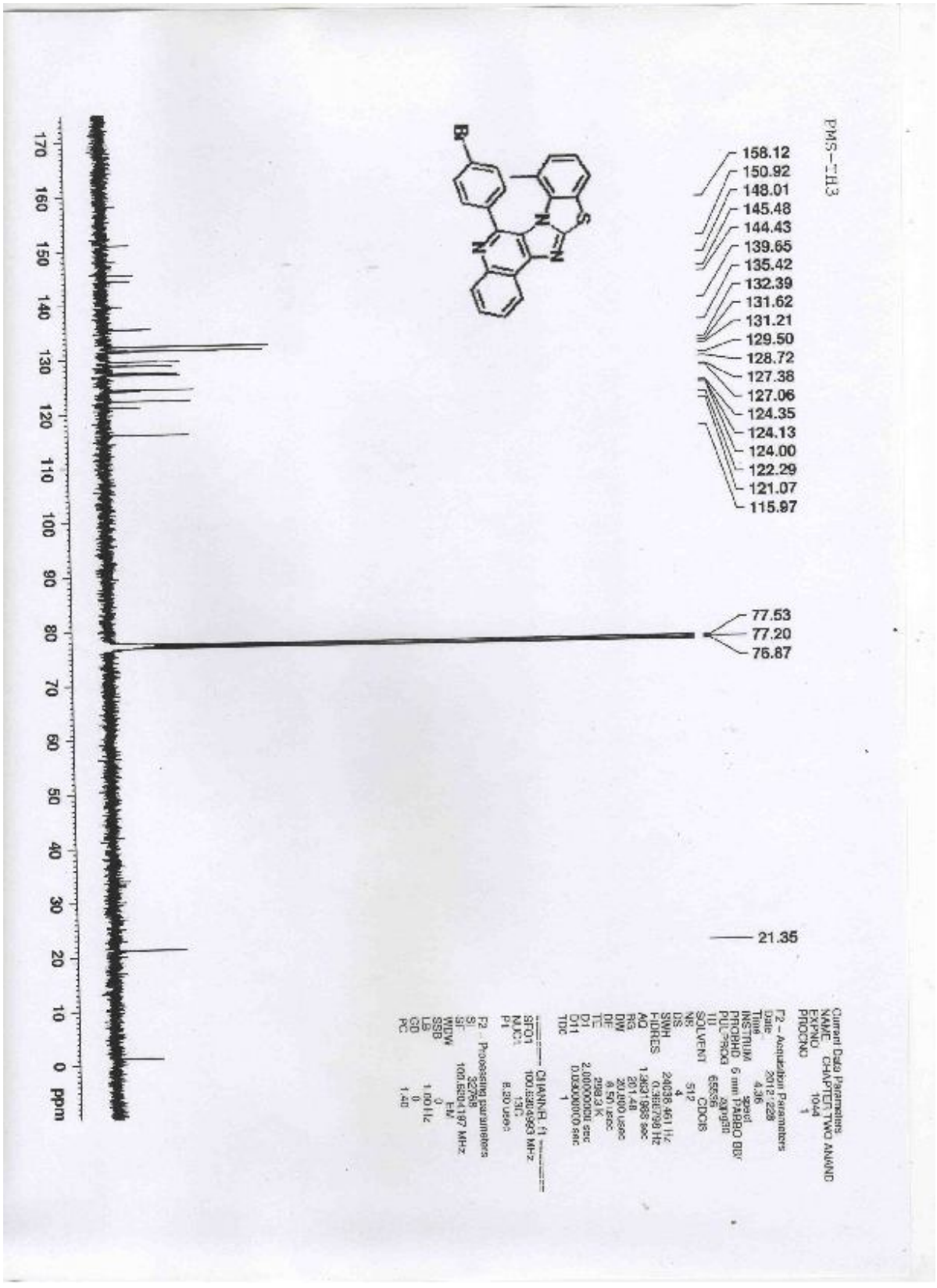
6-(4-Methoxy-Phenyl)-7-methyl-(11-S)-5,6b,12-triaza-benzo[a]fluorine (21d):



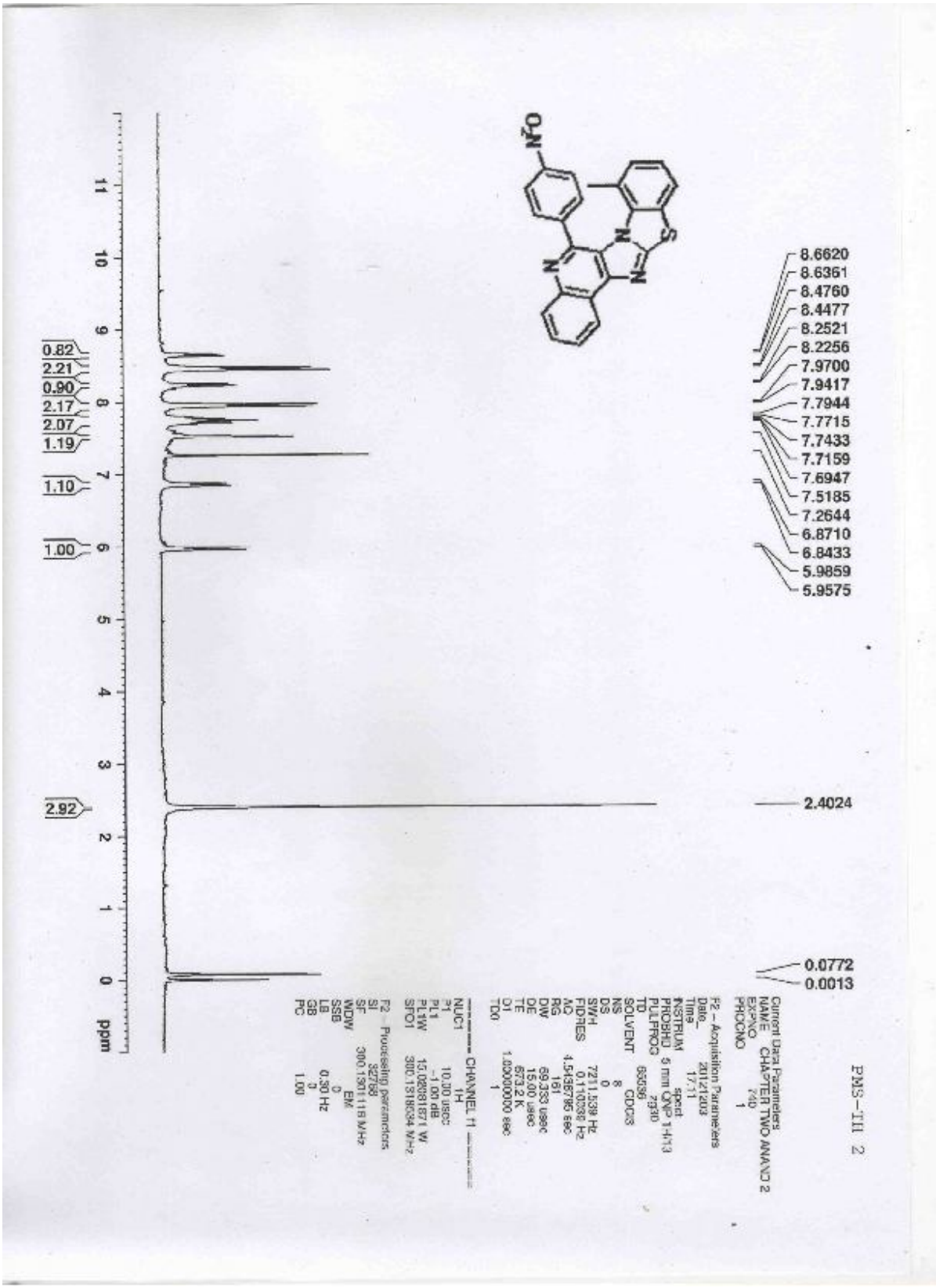

PMS-TH1
MeO
160.92
157.70
150.51
146.56
144.29
134.99
133.11
131.21
131.10
129.29
128.34
127.16
126.51
124.26
123.94
122.10
120.84
116.27
114.43
77.63
77.21
76.78
55.75
21.08
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

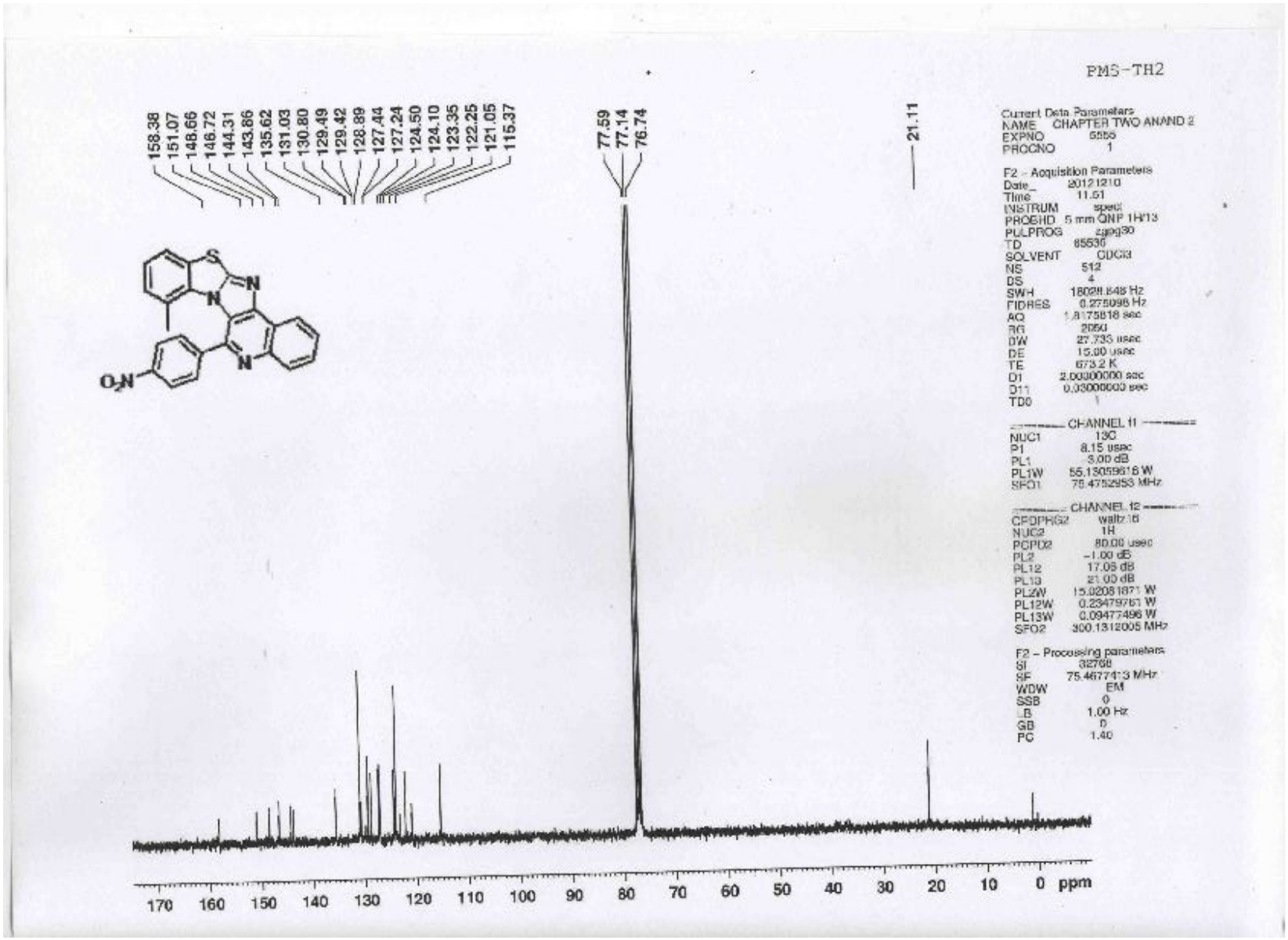
6-(4-Bromo-Phenyl)-7-methyl-(11-S)-5,6b,12-triaza-benzo[a]fluorene (21e):



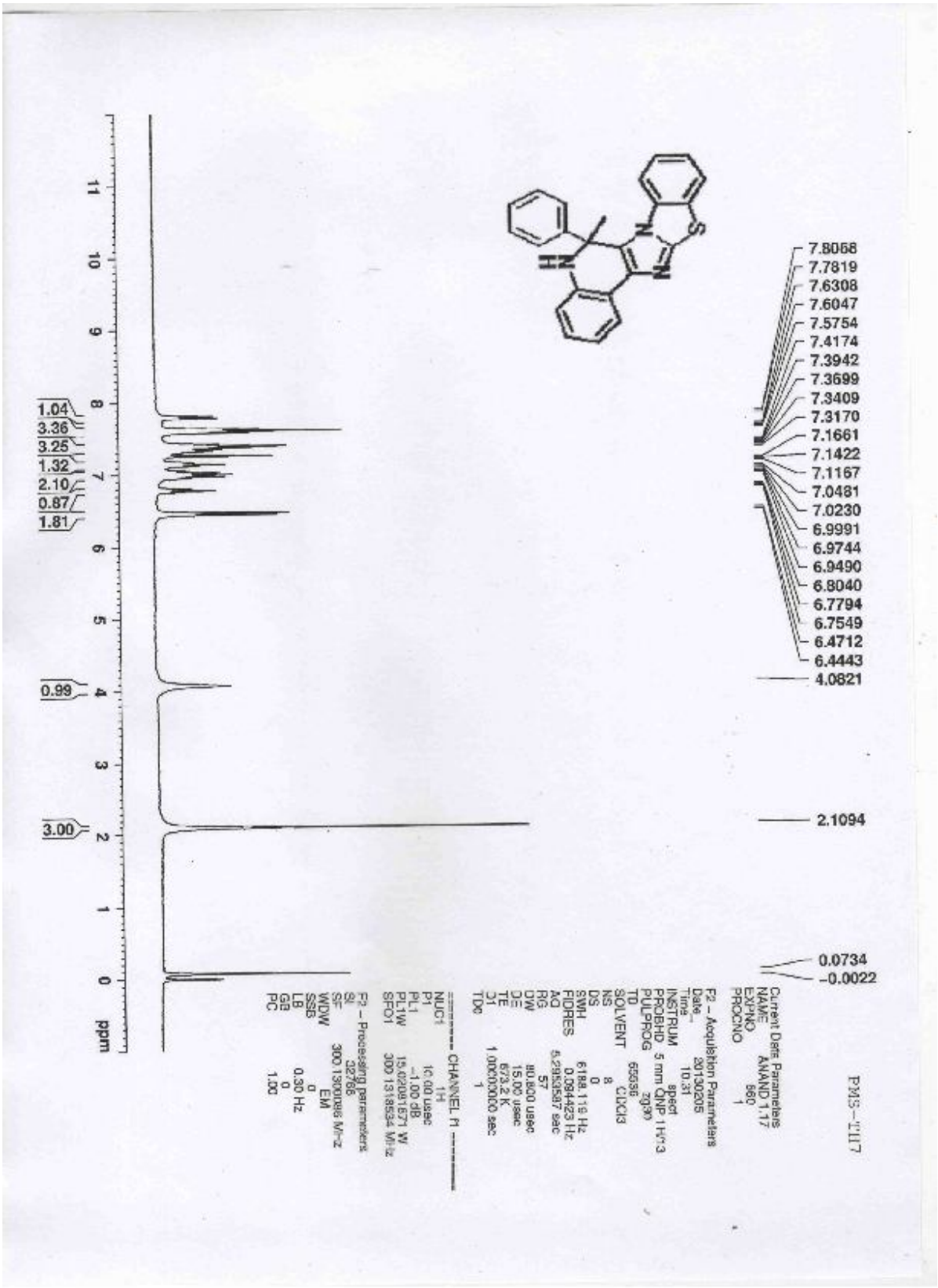

PMS-TH3
Br
158.12
150.92
148.01
145.48
144.43
139.65
135.42
132.39
131.62
131.21
129.50
128.72
127.38
127.06
124.35
124.13
124.00
122.29
121.07
115.97
77.53
77.20
76.87
21.35
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 1044
PROCNO 1
F2 - Acquisition Parameters
Date 20121228
Time 4.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631868 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000009 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6284983 MHz
NUC1 13C
P1 8.50 usec
F2 - Processing parameters
SI 32768
SF 100.6300167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

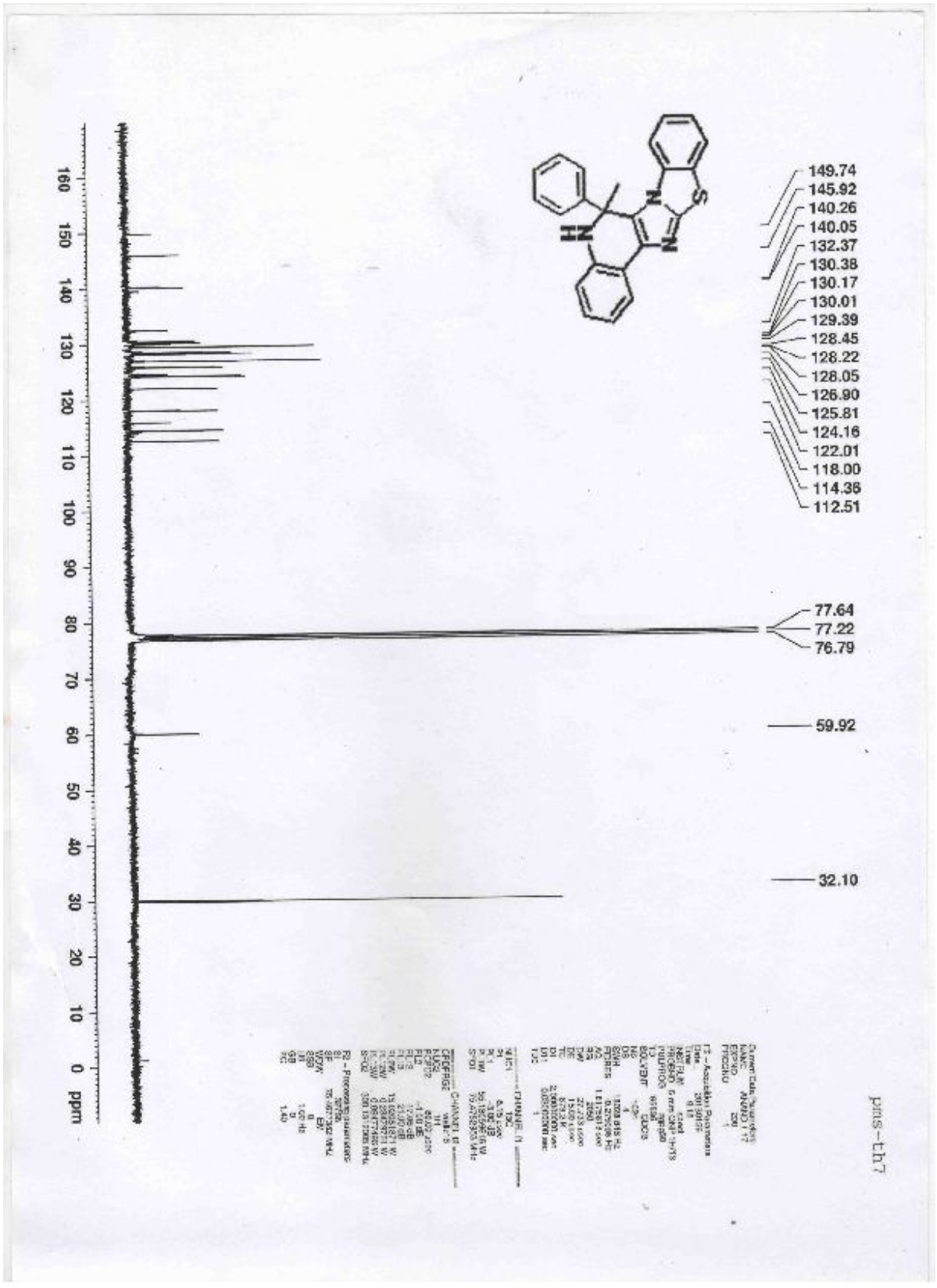
6-(4-Nitro-Phenyl)-7-methyl-(11-S)-5,6b,12-triaza-benzo[a]fluorene (21f):



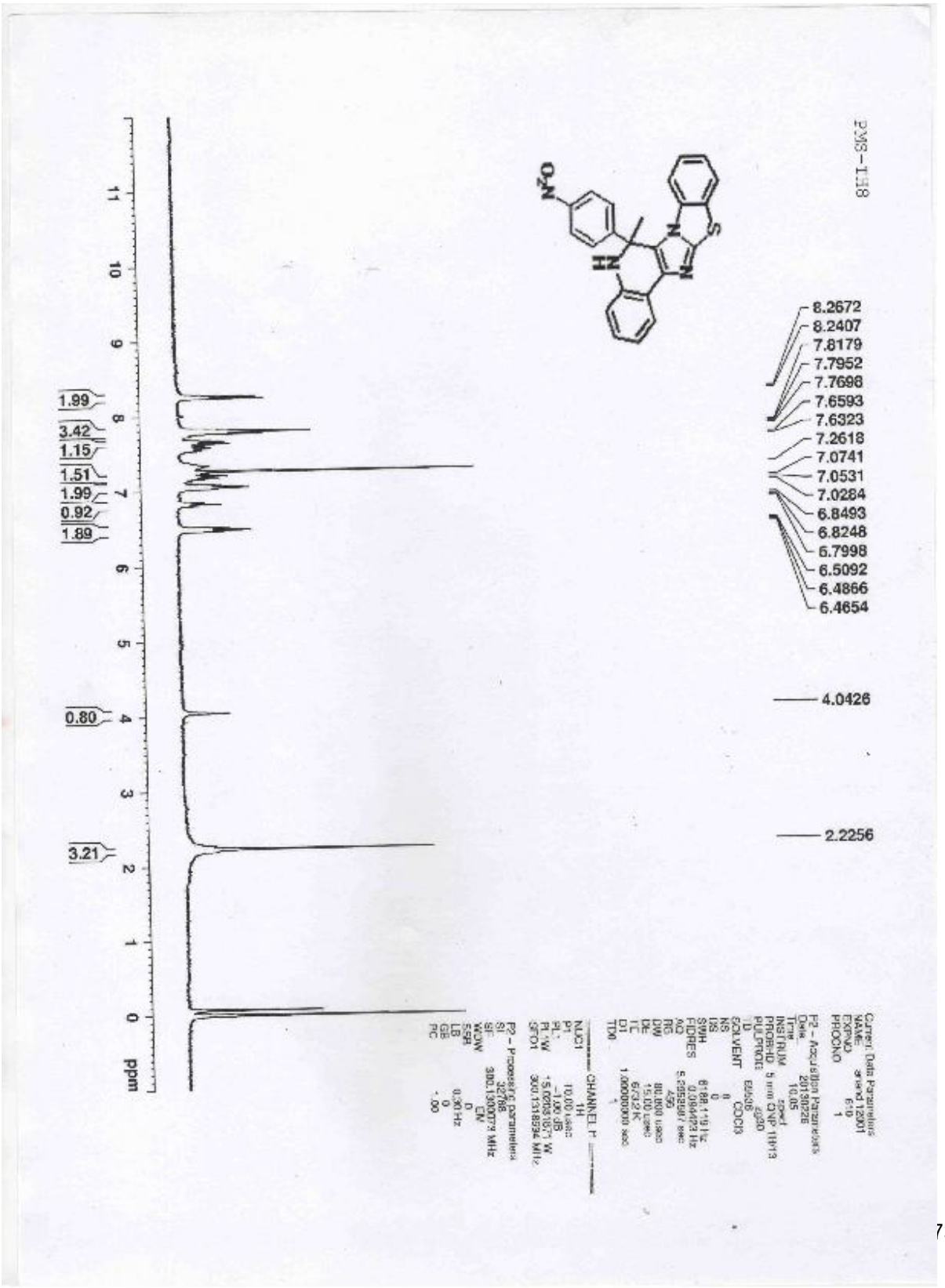

PMS-TH2
158.38
151.07
148.66
146.72
144.31
143.86
135.62
131.03
130.80
129.49
129.42
128.89
127.44
127.24
124.50
124.10
123.35
122.25
121.05
115.37
77.59
77.14
76.74
21.11
Current Data Parameters
NAME CHAPTER TWO ANAND 2
EXPNO 5555
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121210
Time 11.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.848 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.735 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.15 usec
PL1 3.00 dB
PL1W 55.13059616 W
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.05 dB
PL13 21.00 dB
PL2W 15.02081871 W
PL12W 0.23479751 W
PL13W 0.09477496 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677413 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
S
N
N
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

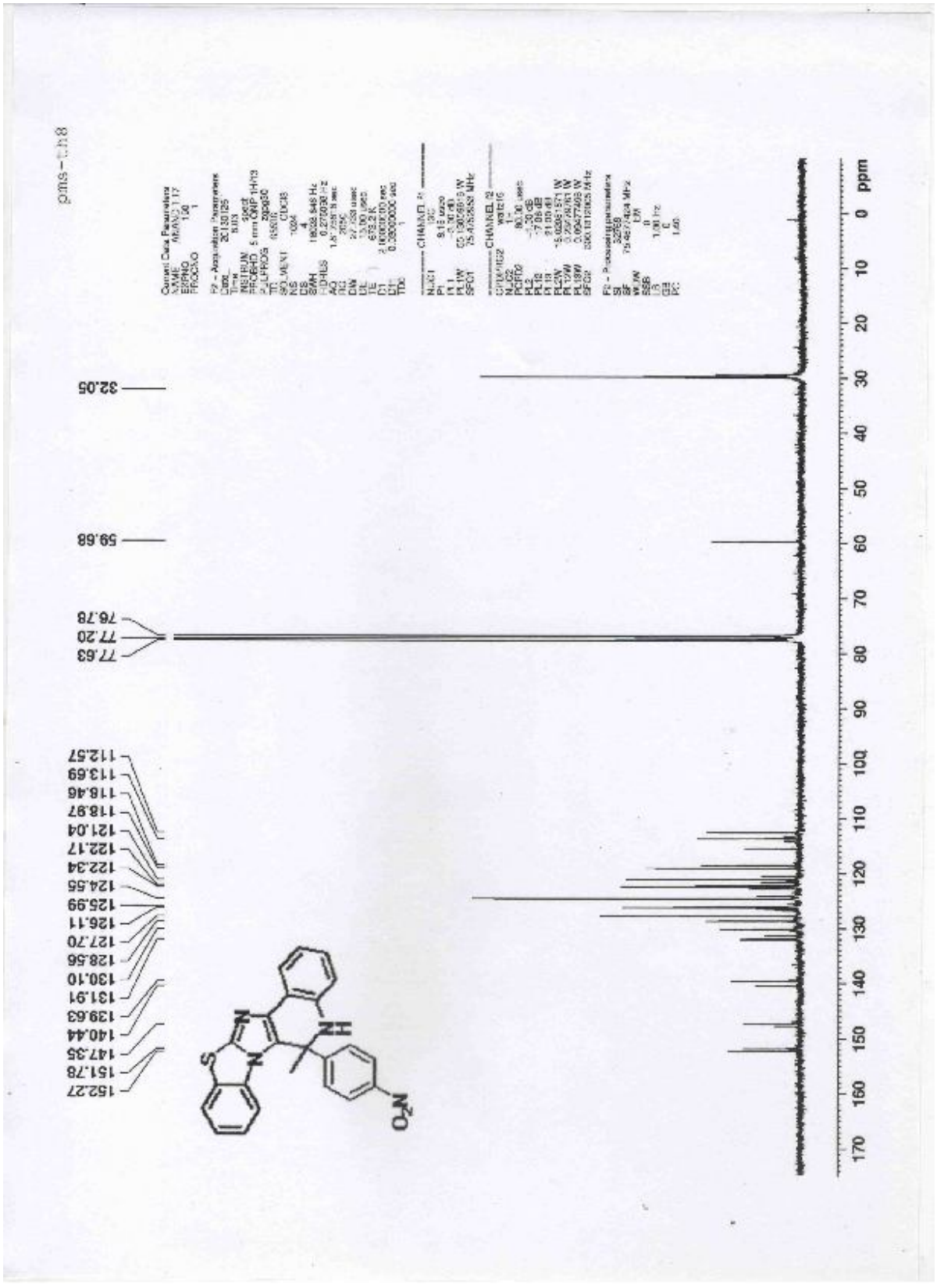
6-Methyl-6-(phenyl)-5,6-dihydro-(11-S)-5,6b,12-triaza-benzo[a]fluorine (22a):



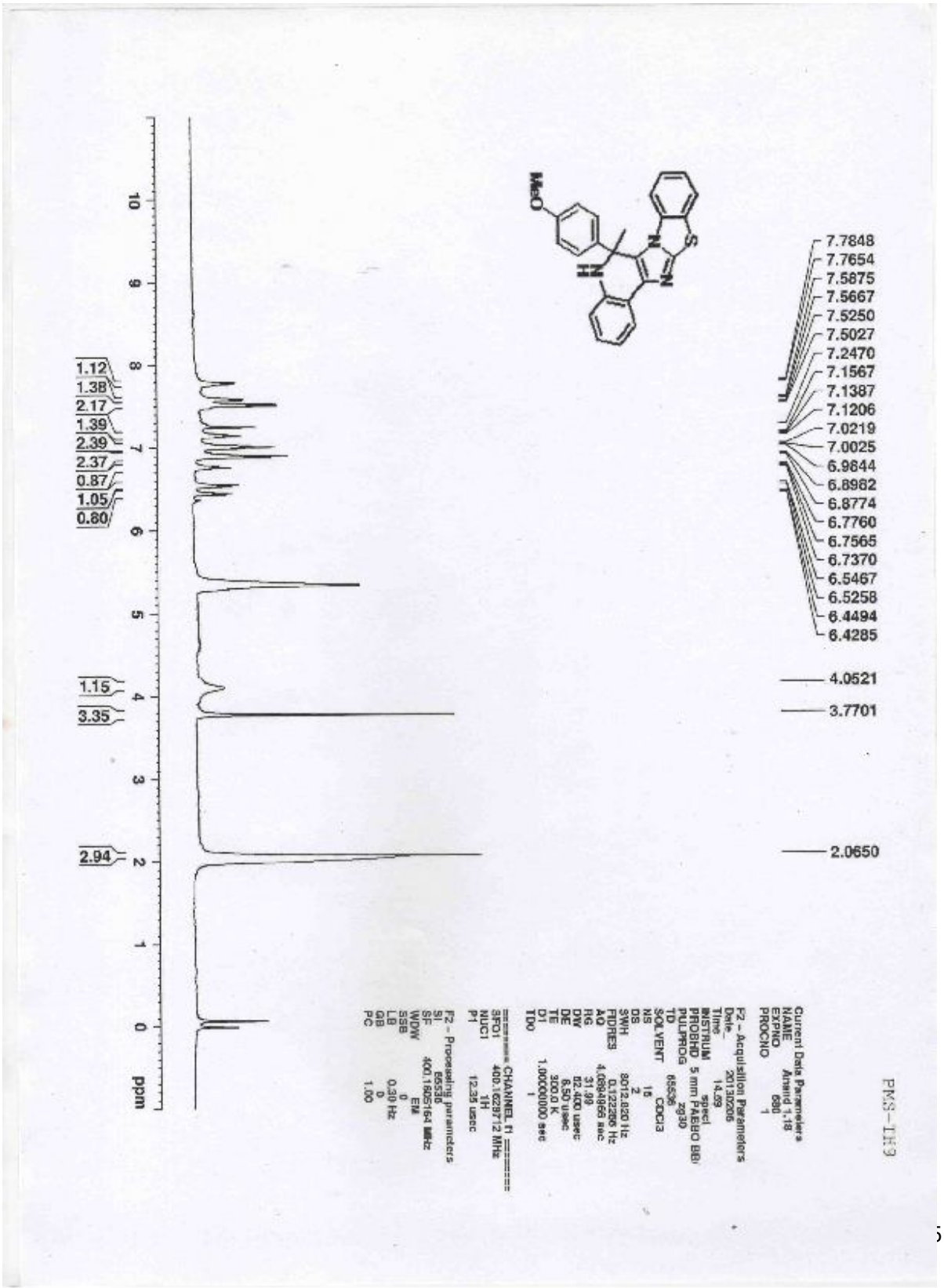

149.74
145.92
140.26
140.05
132.37
130.38
130.17
130.01
129.39
128.45
128.22
128.05
126.90
125.81
124.16
122.01
118.00
114.36
112.51
77.64
77.22
76.79
59.92
32.10
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
pms-th7

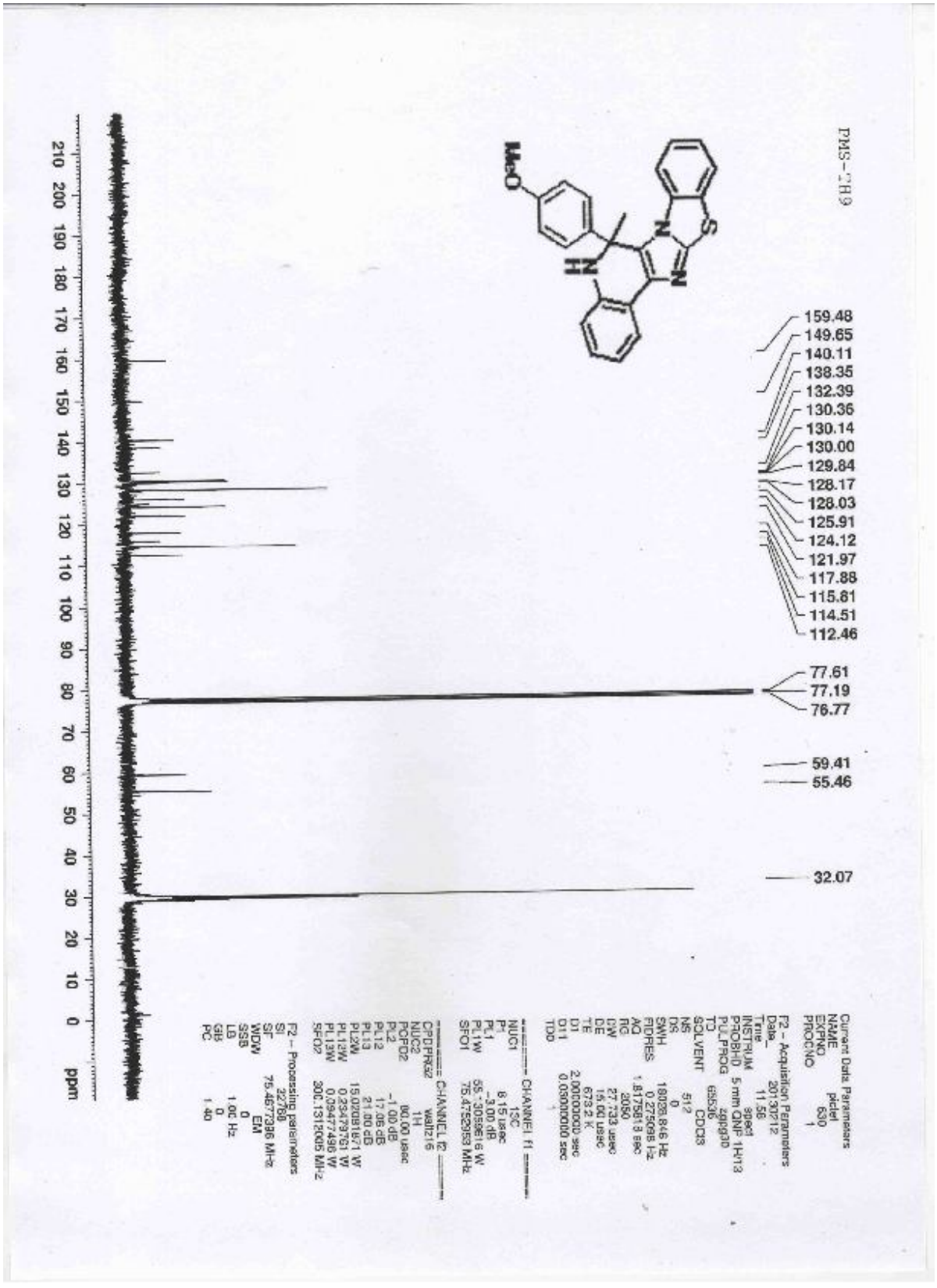
6-Methyl-6-(4-nitro-phenyl)-5,6-dihydro-(11-S)-5,6b,12-triaza-benzo[a]fluorine (22b):




gms-ch8
32.05
59.68
76.78
77.20
77.63
112.57
113.69
118.46
118.97
121.04
122.17
122.34
124.55
125.99
126.11
127.70
128.56
130.10
131.91
139.63
140.44
147.35
151.78
152.27
O2N
ppm

6-Methyl-6-(4-methoxy-phenyl)-5,6-dihydro-(11-S)-5,6b,12-triaza-benzo[a]fluorine (22c):




PMS-2H9
MeO
159.48
149.65
140.11
138.35
132.39
130.36
130.14
130.00
129.84
128.17
128.03
125.91
124.12
121.97
117.88
115.61
114.51
112.46
77.61
77.19
76.77
59.41
55.46
32.07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME picle1
EXPNO 630
PROCNO 1
F2 - Acquisition Parameters
Data_ 20130712
11.56
Time
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 18028.846 Hz
FIDRES 0.275088 Hz
AQ 1.8175813 sec
RG 2050
DW 27.733 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.30559616 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 15.02081671 W
PL12W 0.23479761 W
PL13W 0.09477496 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677386 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Note: The signal of deuterated solvent $CDCl_3$ at δ 7.25-7.29 (ppm) and DMSO-d_6 at δ 2.48-2.51 (ppm) were observed in respective ^{1}H-NMR spectrum and at δ 77.15-77.32 (ppm) for $CDCl_3$ and δ 39.45-39.55 (ppm) for DMSO-d_6 were observed in respective ^{13}C-NMR spectrum.