

Synthesis of Biologically Active Pyridoimidazole/Imidazobenzothiazole

Annulated Polyheterocycles using Cyanuric Chloride in Water

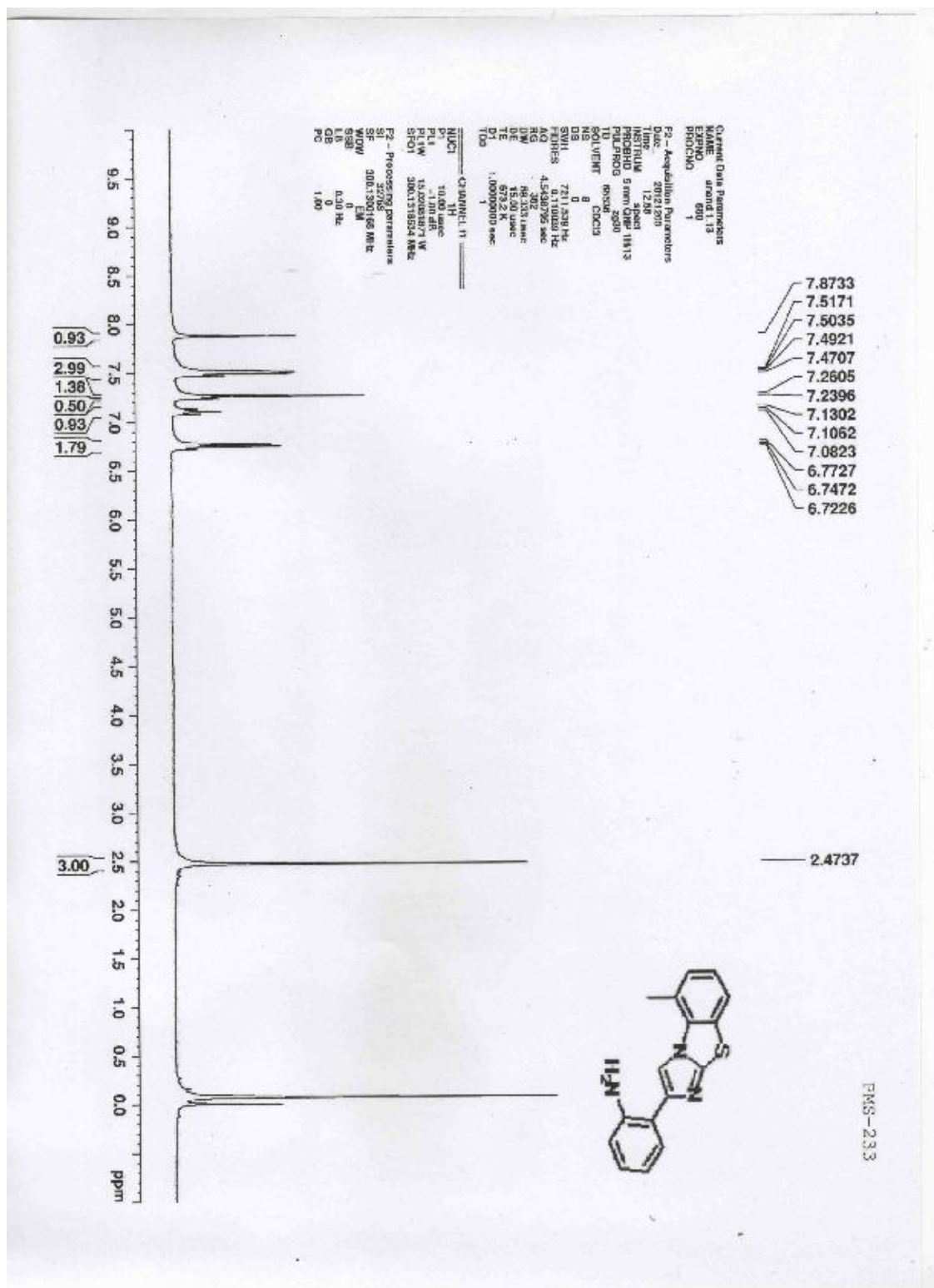
Anand Kumar Pandey,^a Rashmi Sharma,^a Awantika Singh,^b Sanjeev Shukla,^b Kumkum Srivastava,^c Sunil K. Puri,^c Brijesh Kumar,^b and Prem M. S. Chauhan^{*a}

^aMedicinal and Process Chemistry Division, ^bSophisticated Analytical Instrument Facility, ^cDivision of Parasitology, CSIR-Central Drug Research Institute, Lucknow, 226031, India

^{*}Corresponding Author: Phone No: 0522-2771940, Extn: 4659, 4660, Fax: 91-522-2771941; Email: premsc58@hotmail.com, prem_chauhan_2000@yahoo.com

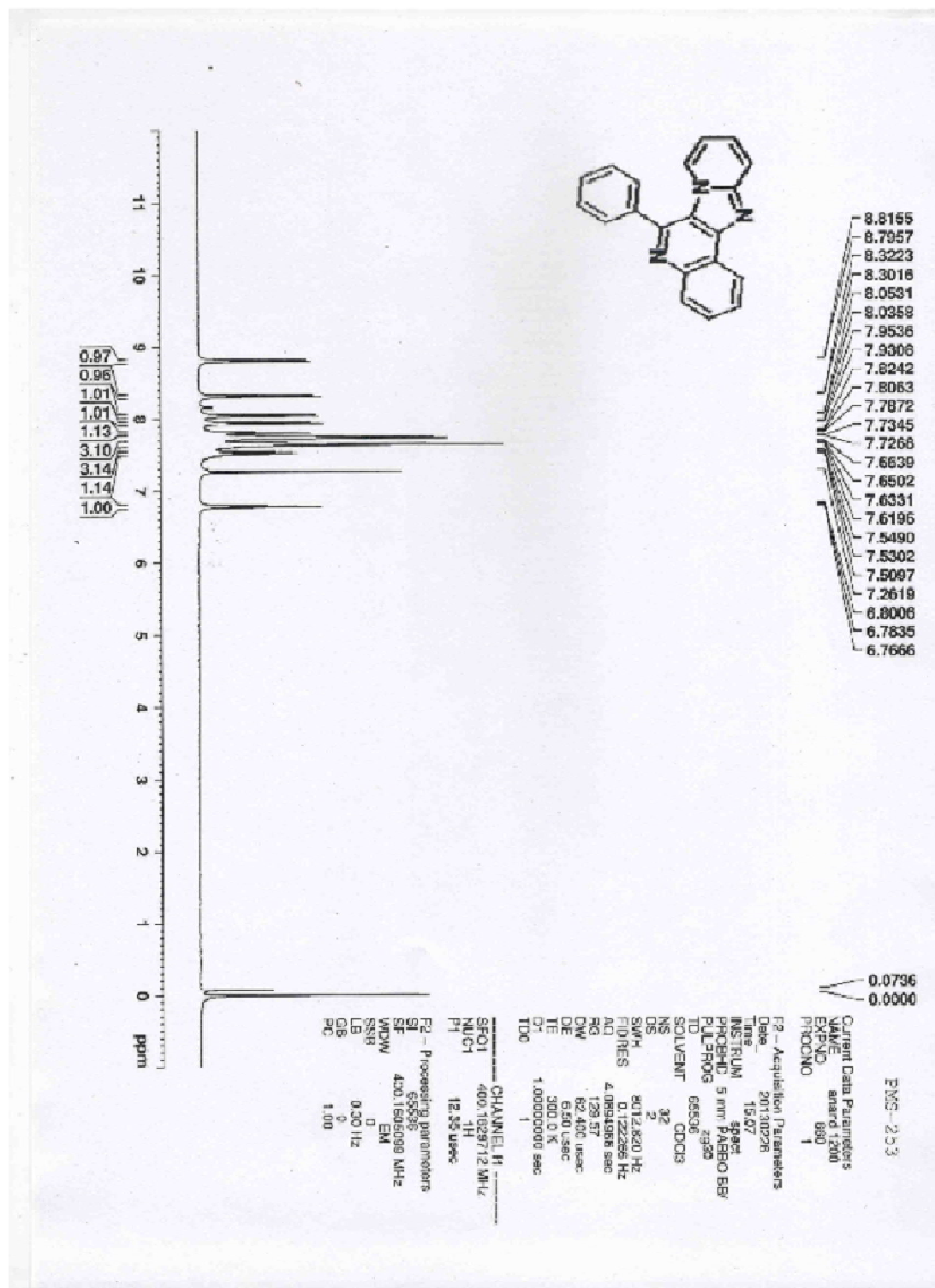
Contents:	Page No.
Scan copies of spectra (¹ H NMR and ¹³ C NMR)	S2-S76
Note for deuterated solvents peaks	S77

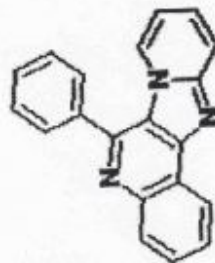
2-(3-methyl-imidazo[2,1-b]-benzothiazol-6-yl)-phenylamine (20):





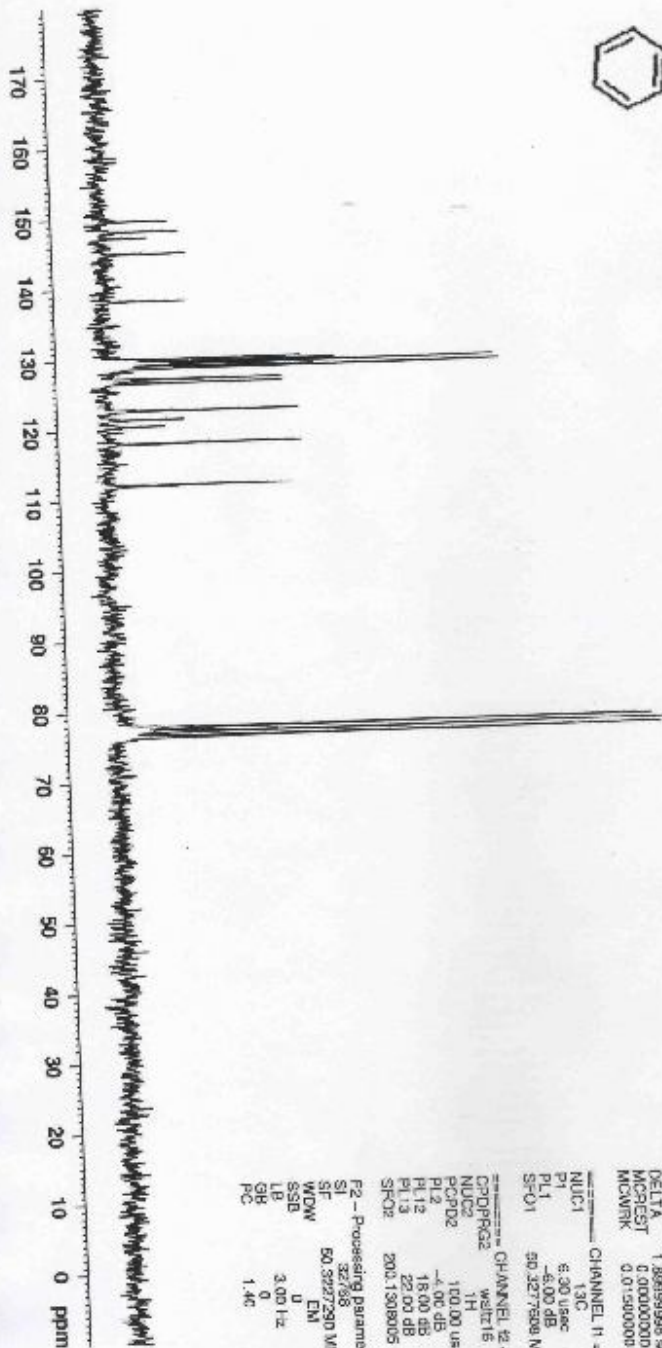
6-Phenyl-5,6b,11-triaza-benzo[a]fluorene (9a).





149.64
148.21
147.29
145.00
138.28
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



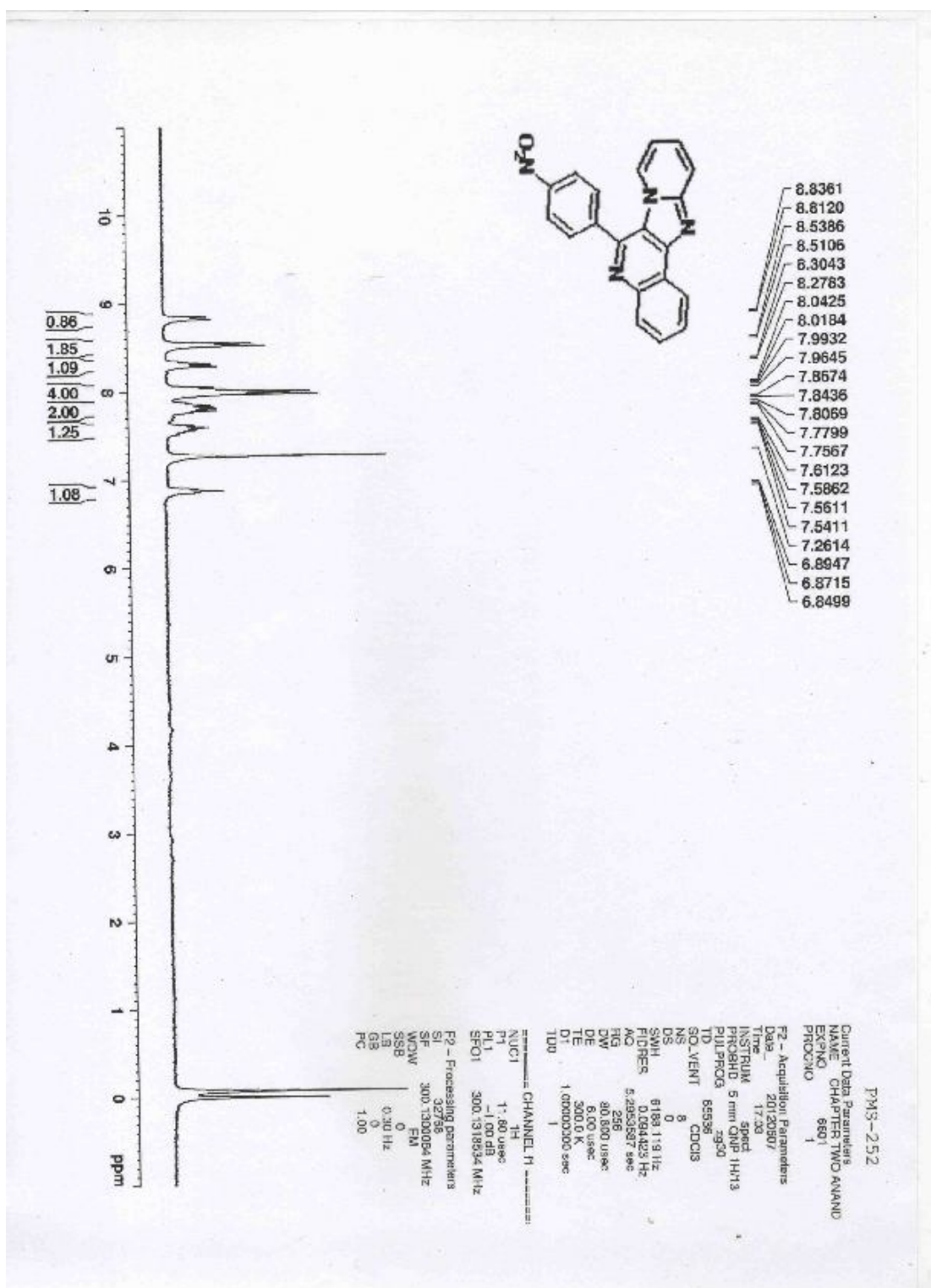
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EXPNO 610
PROCNO 1
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INSTRUM spect
PROBHD 5 mm QNP 130C
PULPROG zgpg30
TD 65536
FIDRES 0.000000
SOLVENT CDCl3
NS 387
DS 4
SWH 11390.407 Hz
FIDRES 0.162959 Hz
AQ 2.7329011 sec
RG 71.8
DW 41.700 usec
DE 6.00 usec
TE 300 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.36959986 sec
MCREST 0.0000000 sec
MCWIRK 0.01500000 sec

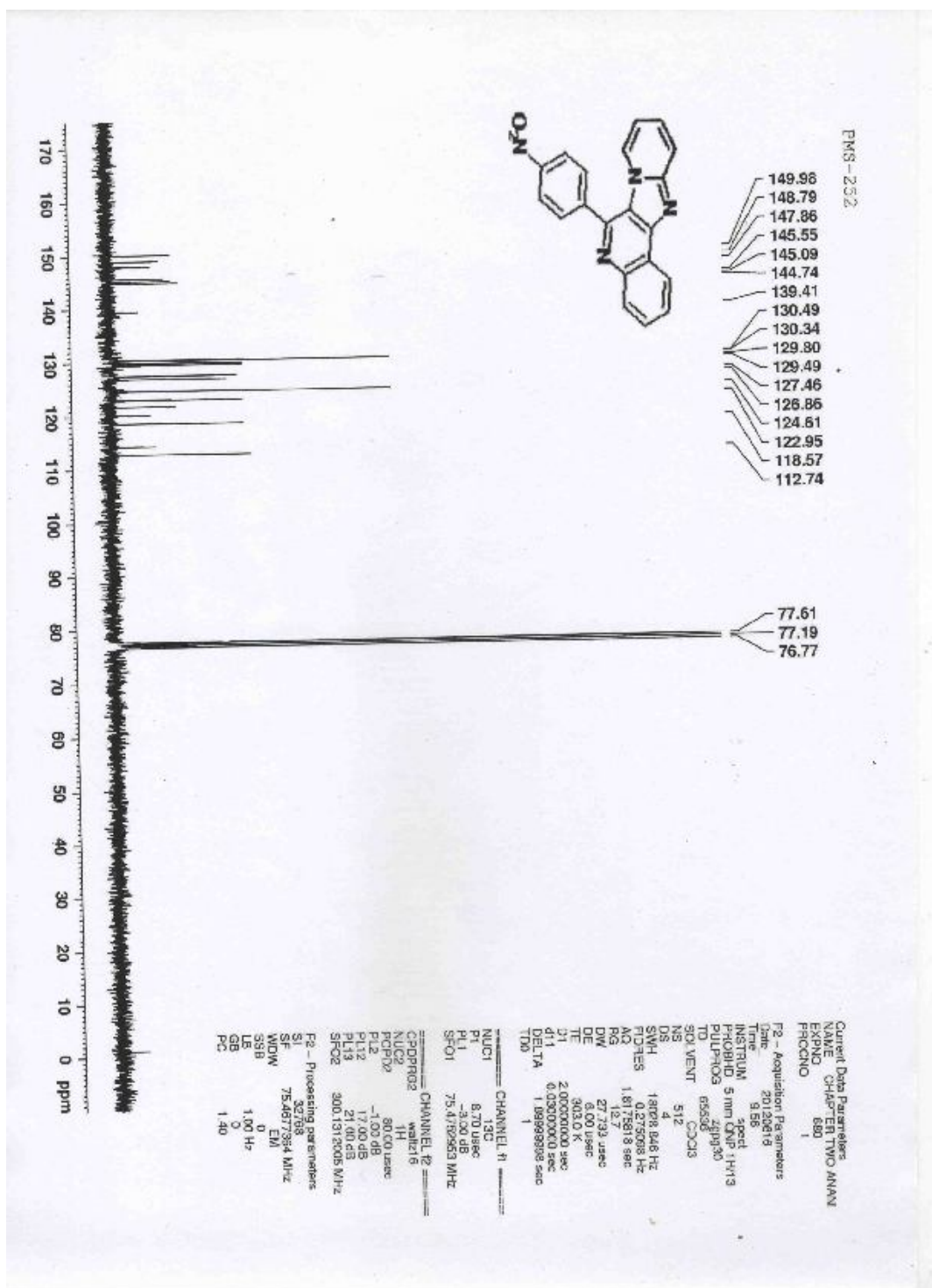
===== CHANNEL f1 =====
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.2675000 MHz

===== 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

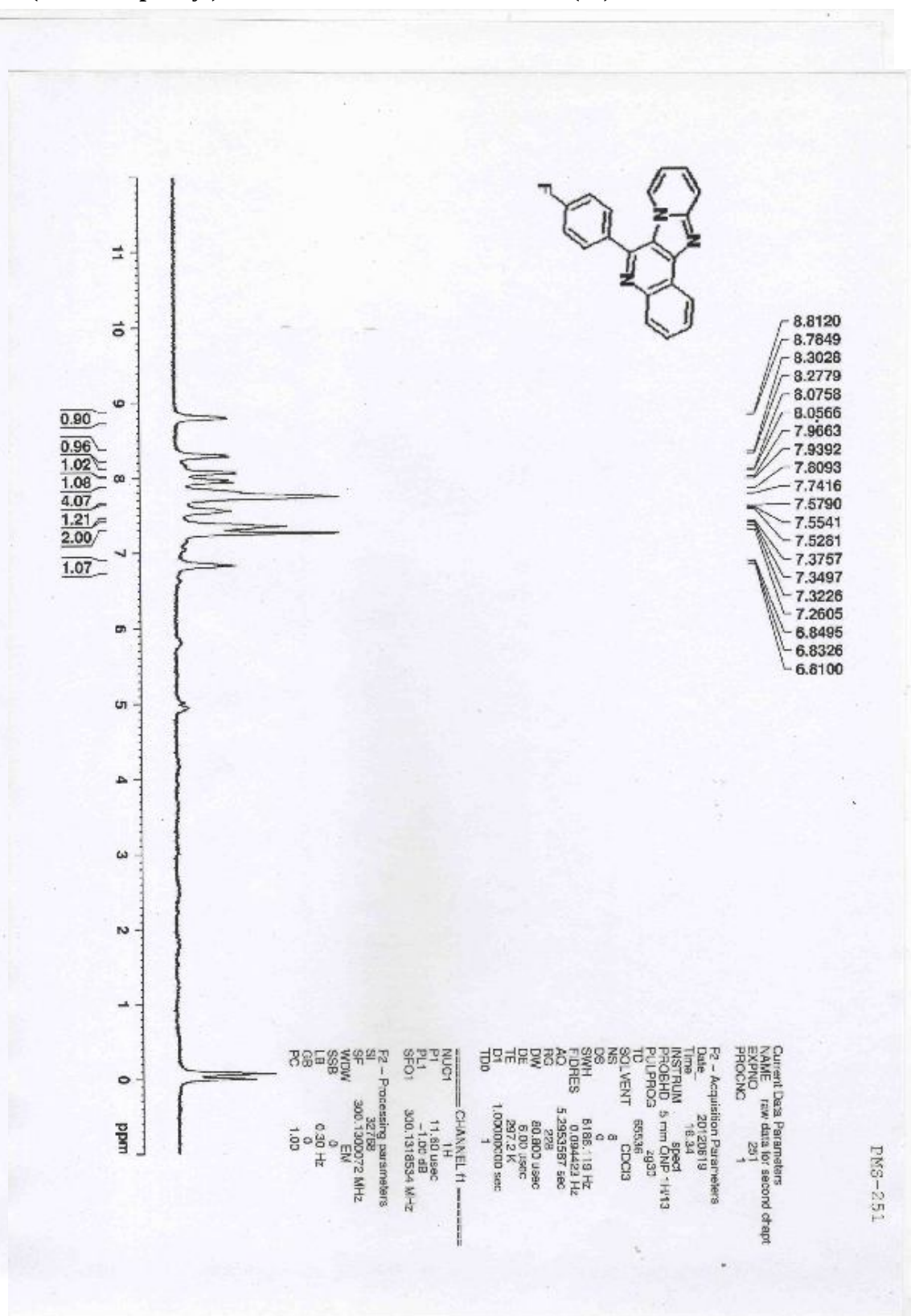
F2 - Processing parameters
SI 32768
SF 503.27290 MHz
WDW EN
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

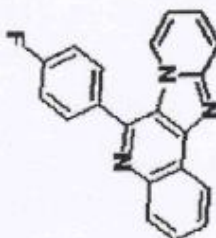
6-(4-Nitro-phenyl)-5,6b,11-triaza-benzo[a]fluorine (9b):



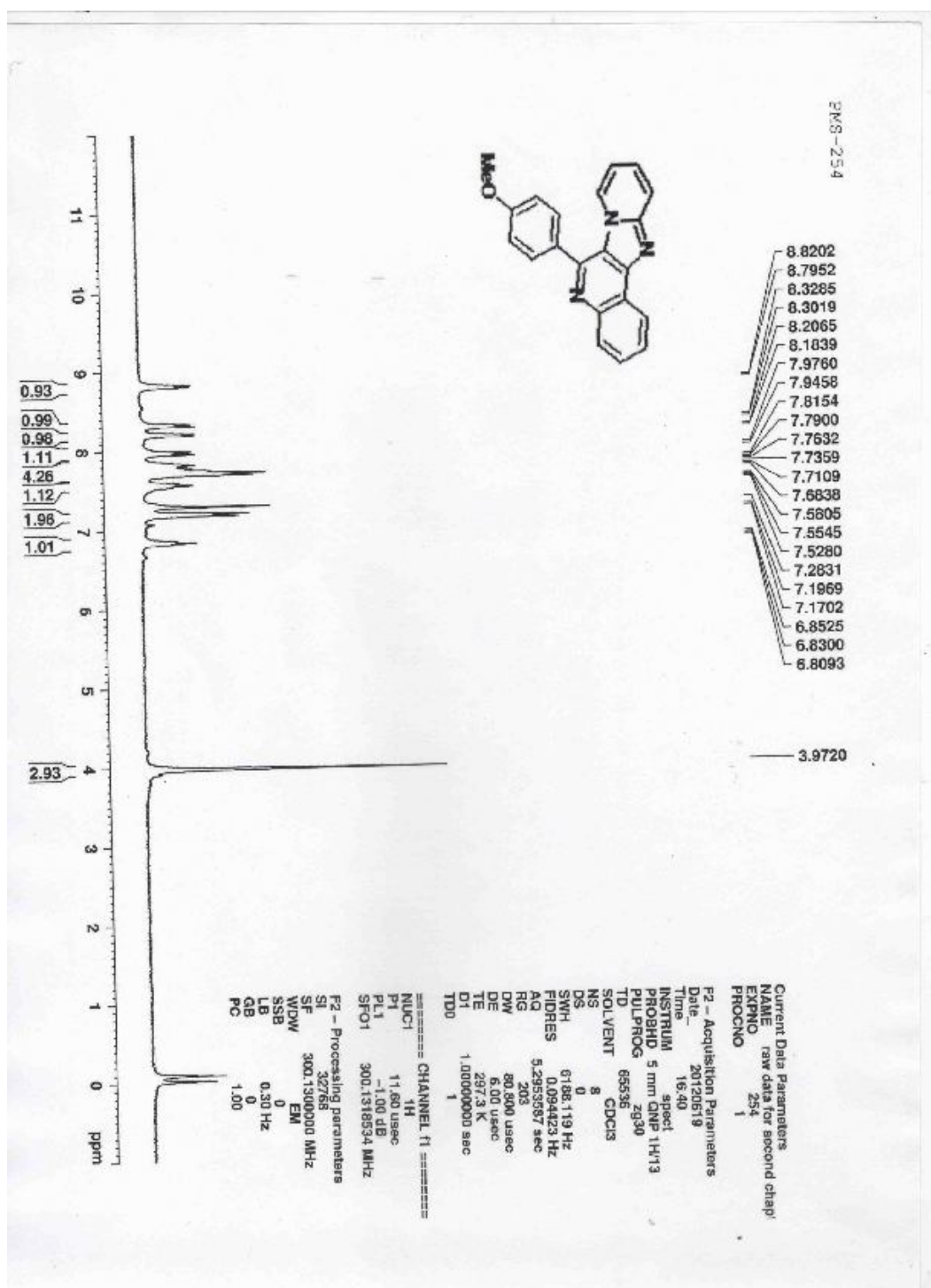


6-(4-Fluoro-phenyl)-5,6b,11-triaza-benzo[a]fluorene (9c):

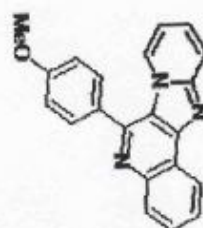


[illegible]

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



PMS-254



- 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
- 118.10
- 114.82
- 112.07

- 77.81
- 77.17
- 76.54

55.63



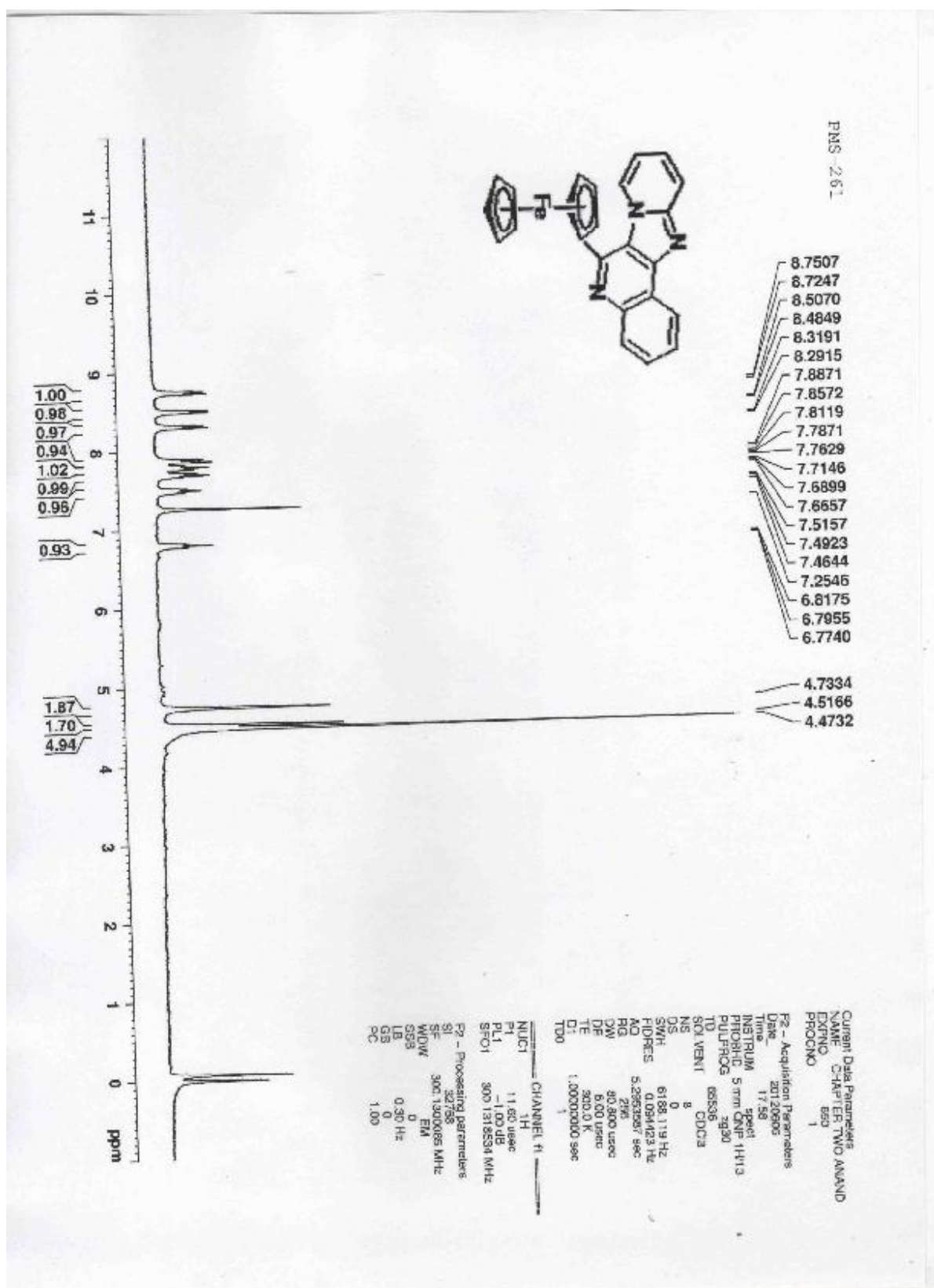
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EXPNO 2542
PROCNO 1
F2 - Acquisition Parameters
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Time 15:47
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SFOH 11990.407 Hz
FIDRES 0.162959 Hz
AQ 2.7329011 sec
RG 40.3
RW 41.700 usec
DE 16.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DE 1.68989906 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

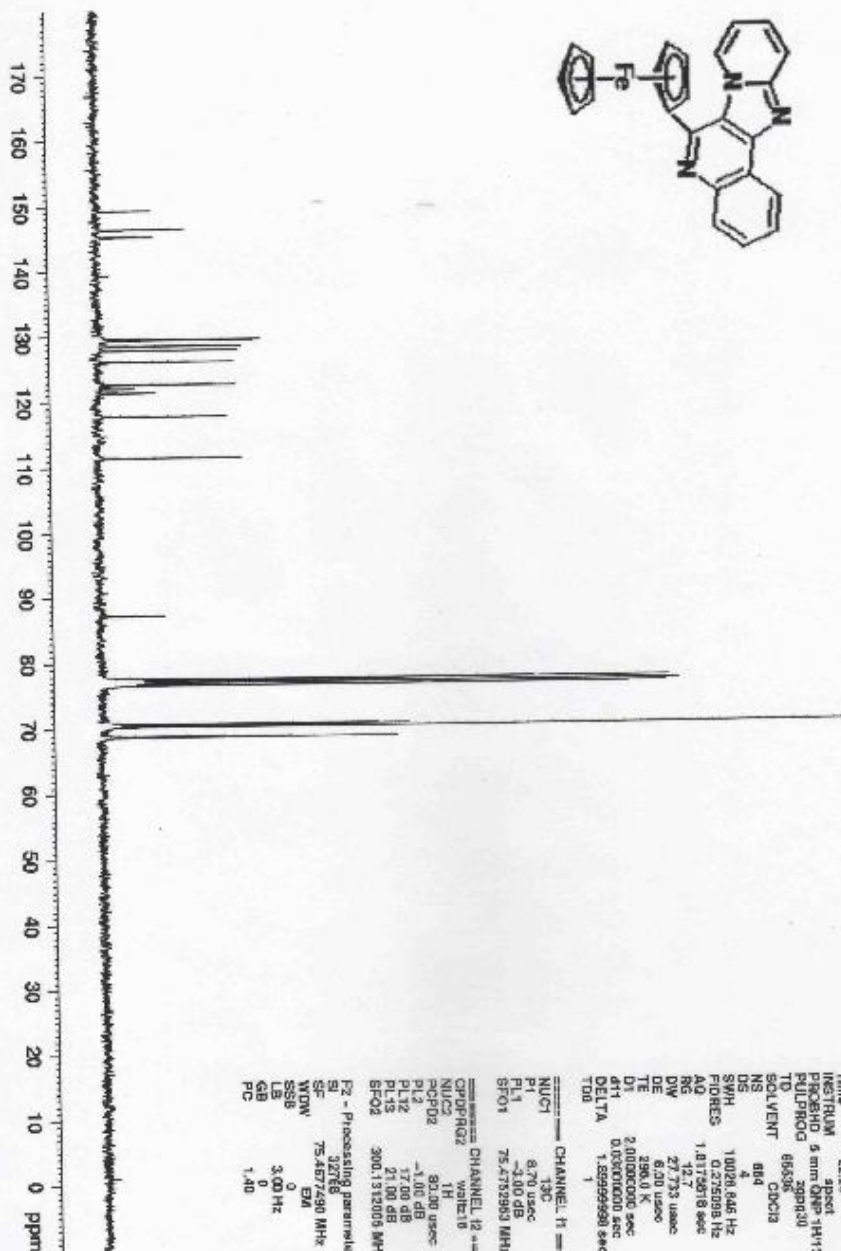
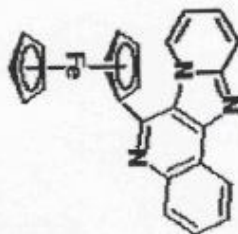
CHANNEL f1
NUC1 13C
P1 0.30 usec
PL1 -6.00 dB
SFO1 50.3277906 MHz

CHANNEL f2
CPDPRG2 1452176
NUC2 1H
P2 1.00 usec
PL2 -4.00 dB
SFO2 500.1360000 MHz

F2 - Processing parameters
SI 32768
SF 503277224 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

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





Current Data Parameters	
NAME	new data for second chapter
EXPNO	2612
PROCNO	1

F2 - Acquisition Parameters
Date_ 20120612
Time_ 22:38

INSTRUM aspect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30

FORMULA	4449000
TD	65536
SOLVENT	CDC12

MS	004
DS	4
SWH	10028.046 Hz

FIDRES	0.275098 Hz
AQ	1.0173618 sec
RG	12.7

27.733 usac
6.00 usac
199.0 K

DT1	2.00000000 sec
DT4	0.00000000 sec
DT1TA	1.00000000 sec

DELIA
TDO

```
===== CHANNEL 7 =====
NUC1      13C
P1         8.70 usec
```

PL1 -3.00 dB
GFO1 75.4752963 MHz

CHANNEL 12
CPDPRG2 wait:10
NICS 1H

PCPD2	82.00 usec
PL2	-1.00 dB
PL3	17.00 dB

PL13 21.00 dB
BFO2 300.1312006 MHz

P2 - Processing parameter
51 32766

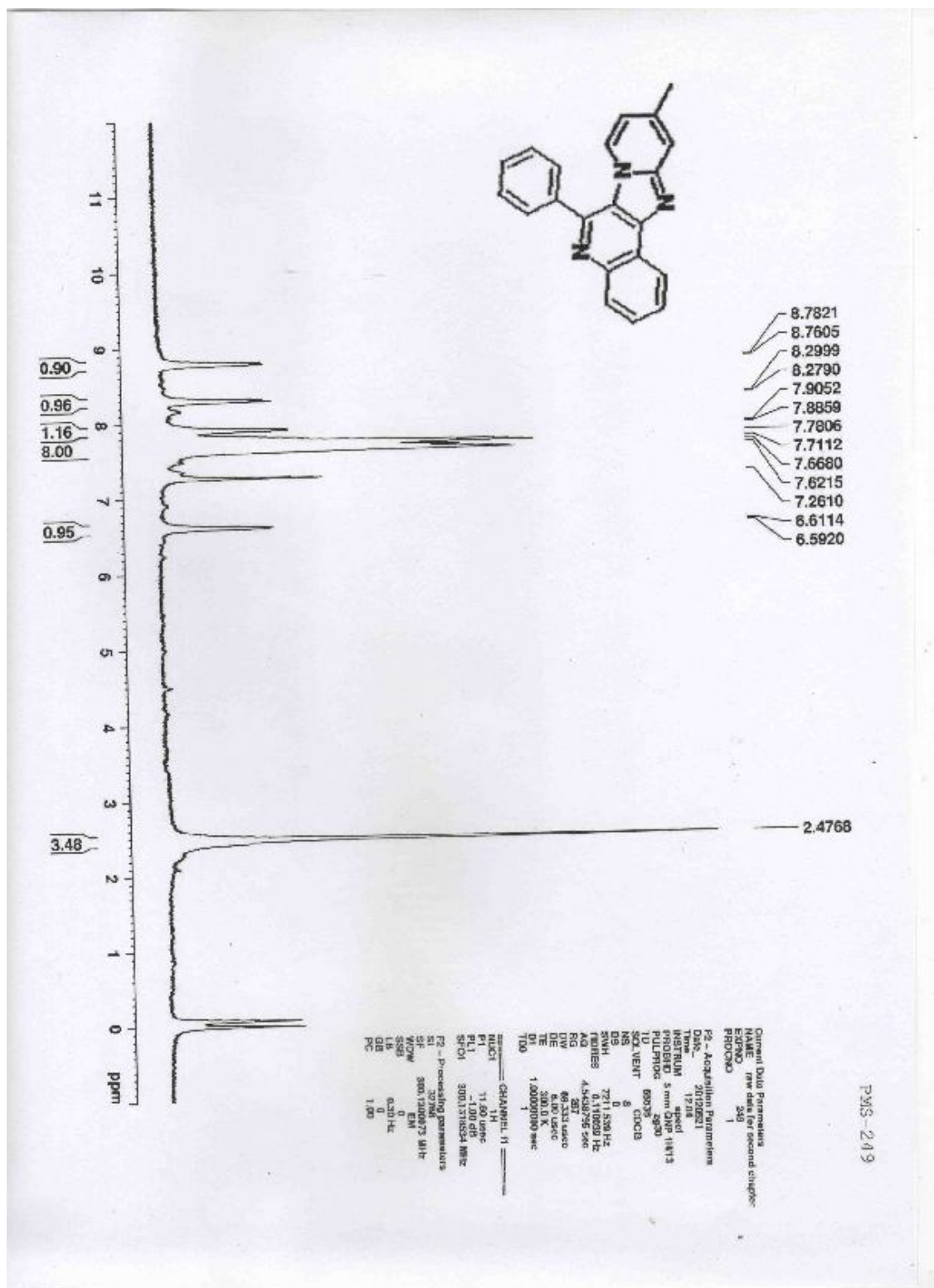
SP 75.467490 MHz
WCDN EA
660B 0

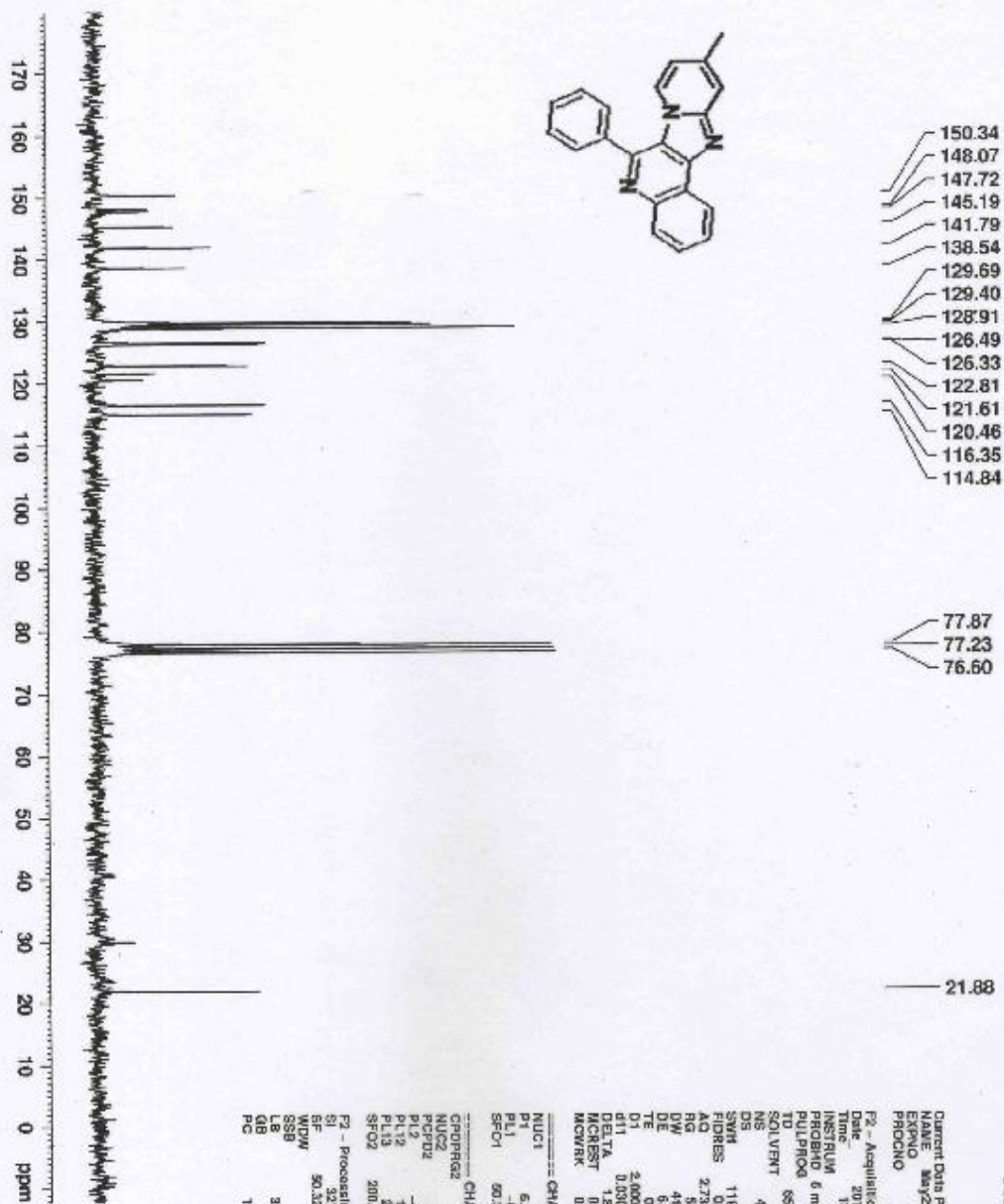
300 Hz
0
1.40

SECRET

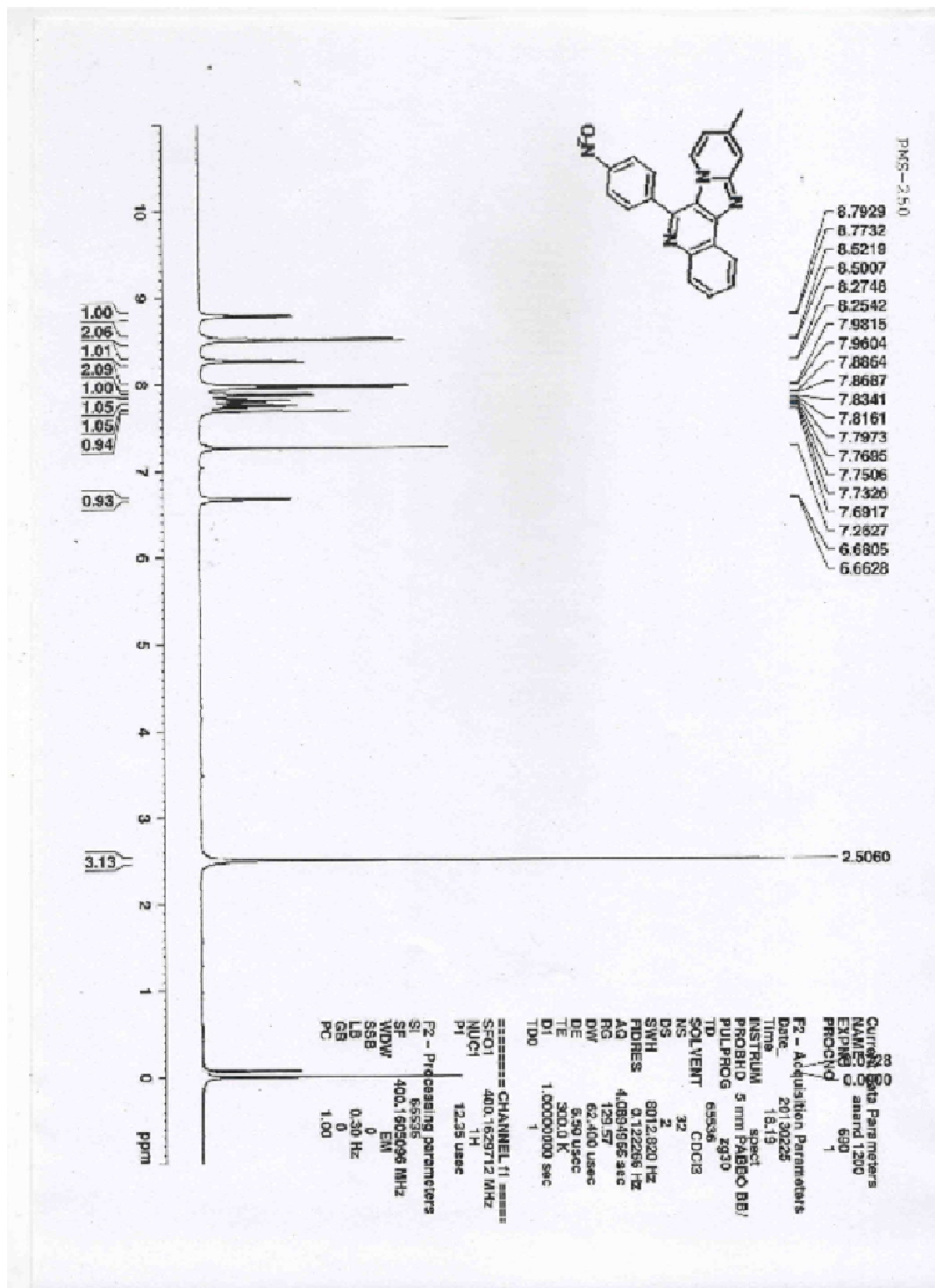
0 10 0 ppm

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

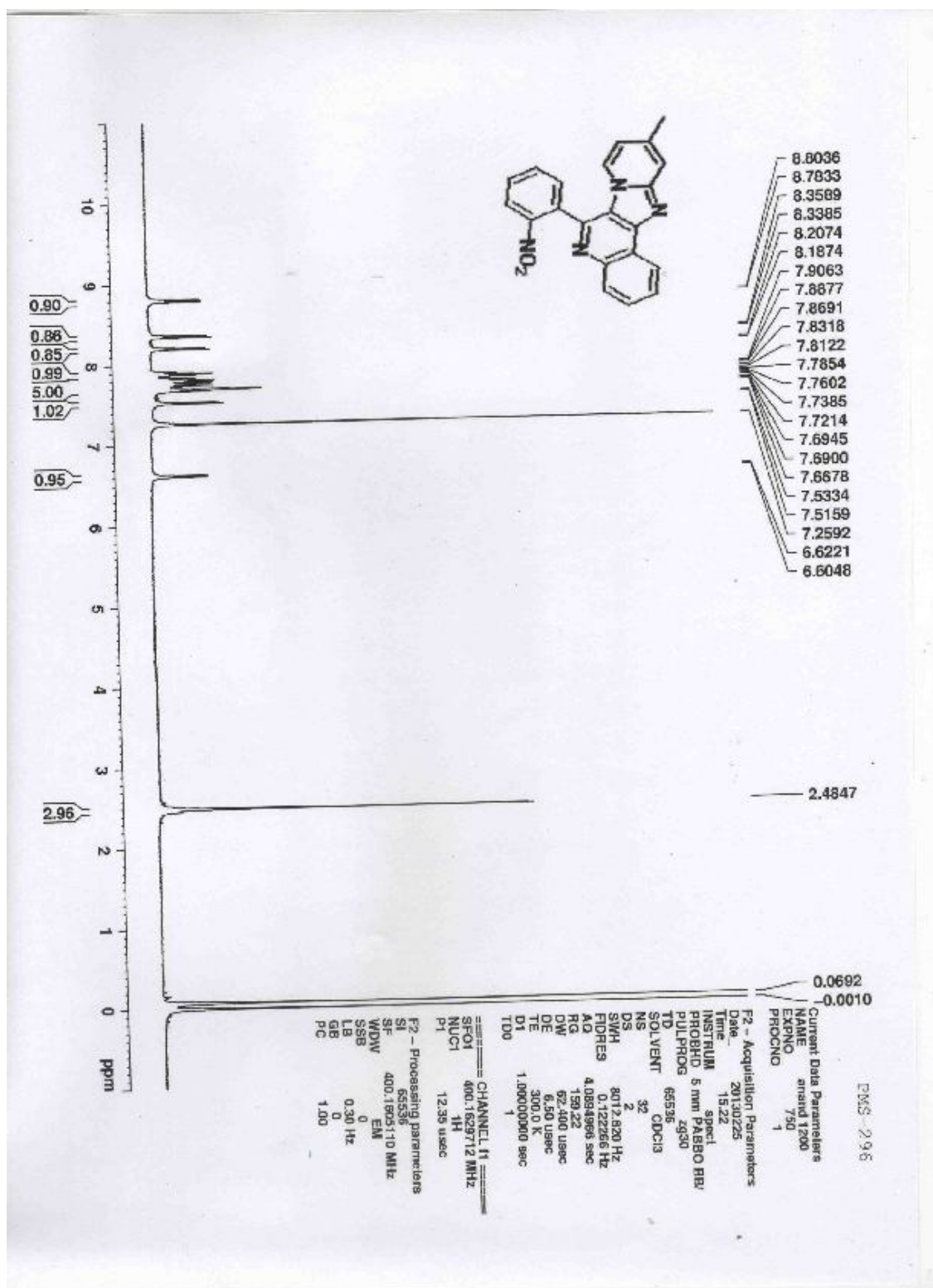


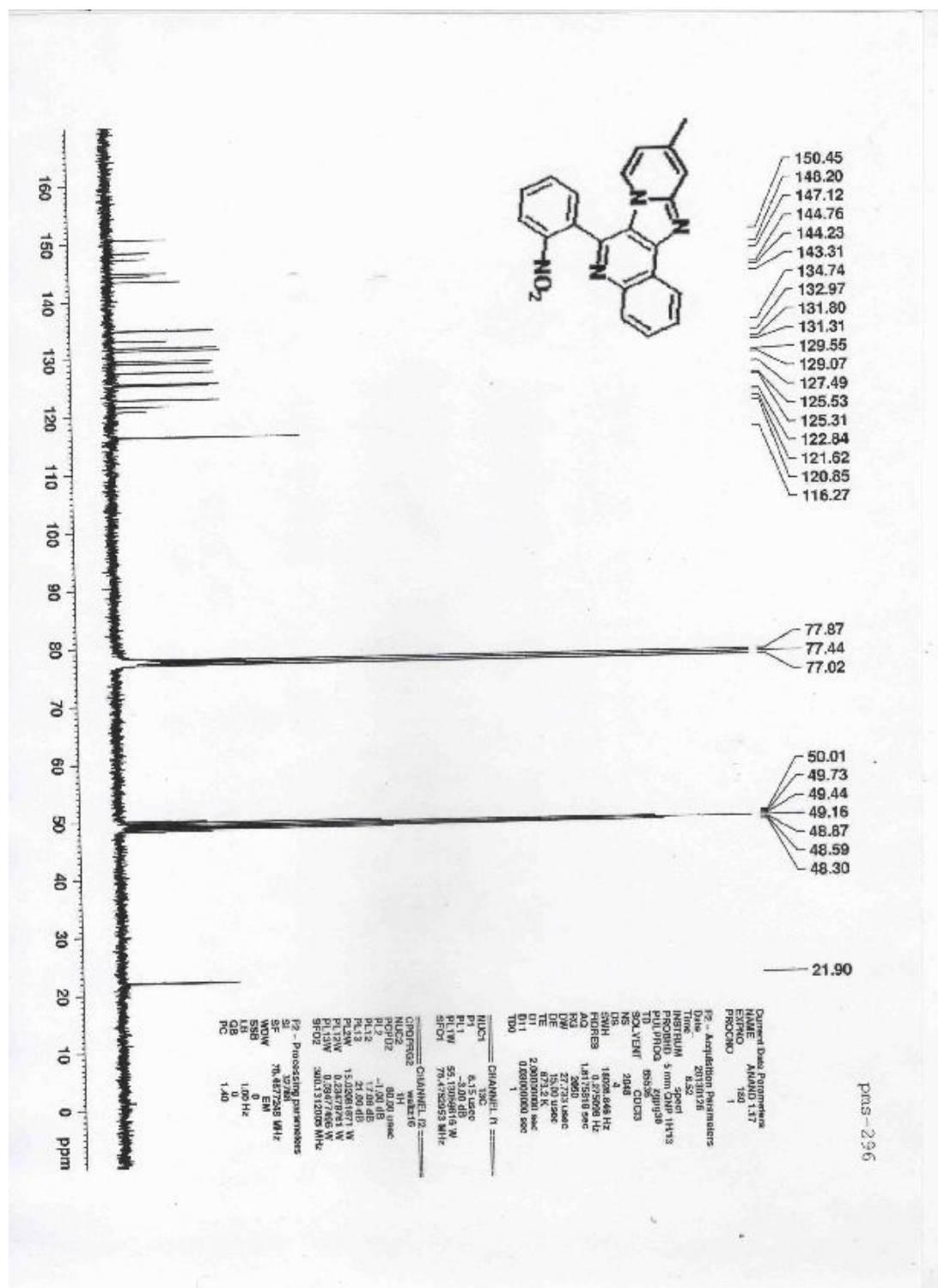


6-(4-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9g):

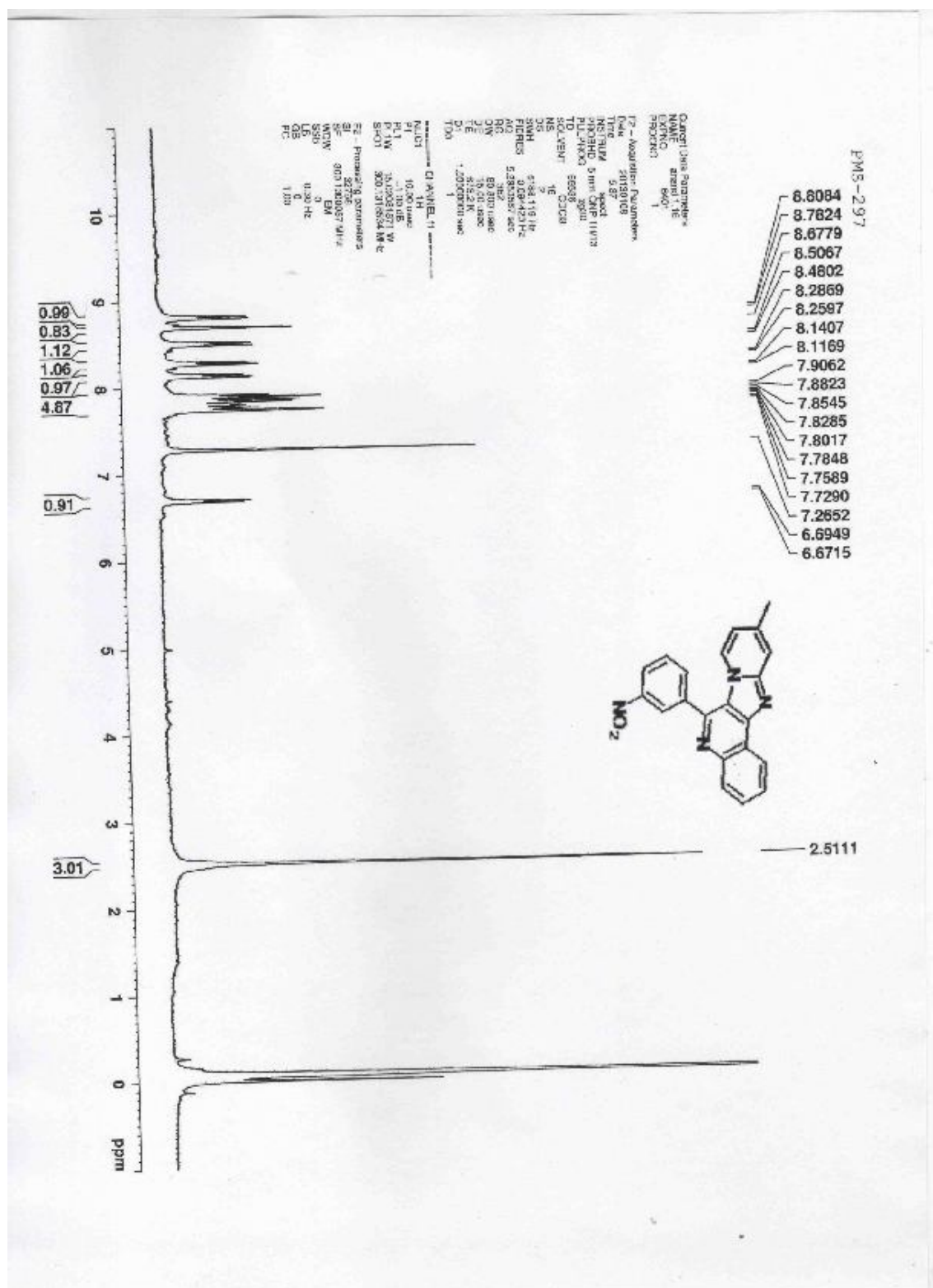


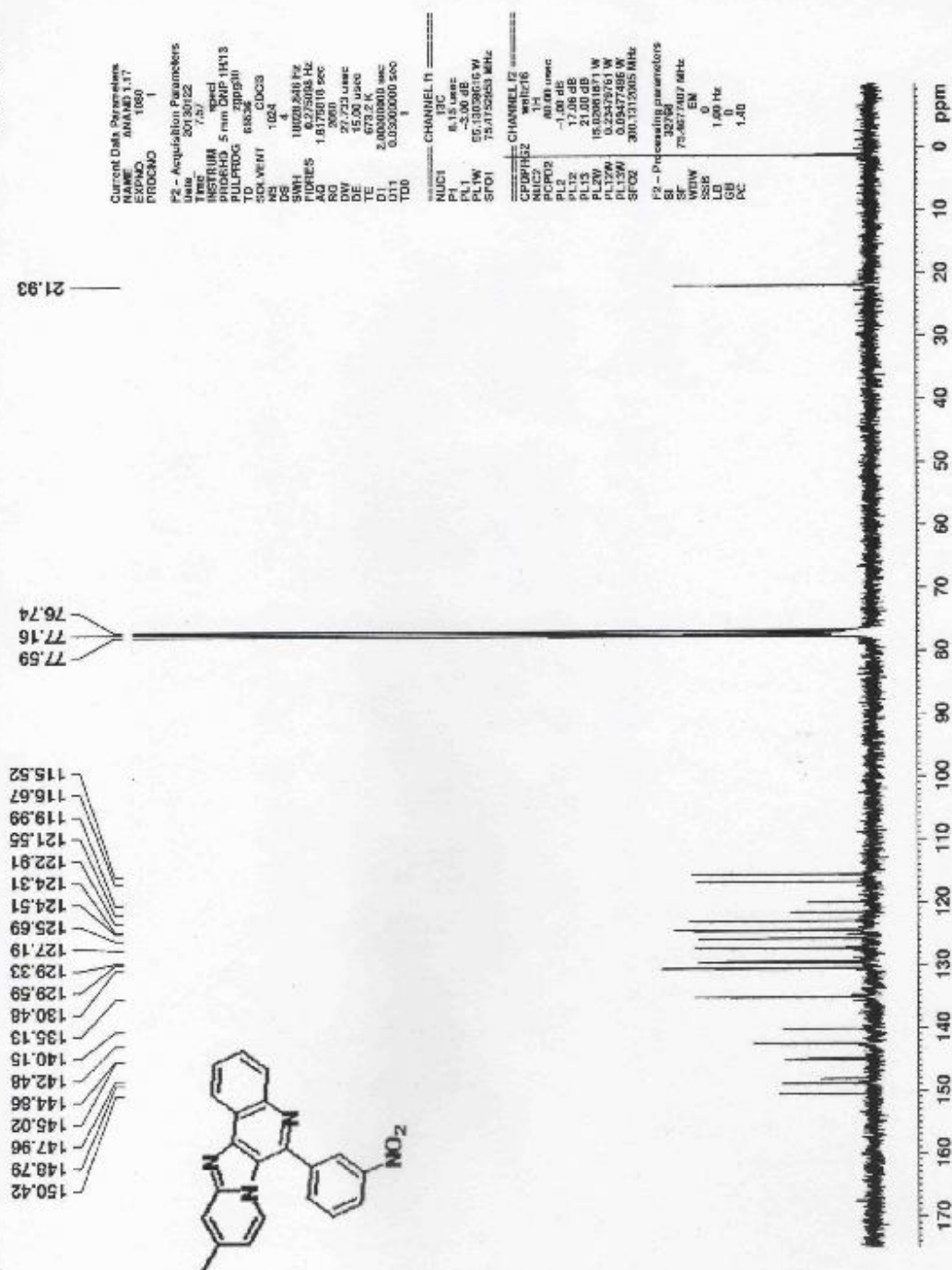
6-(2-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9h):



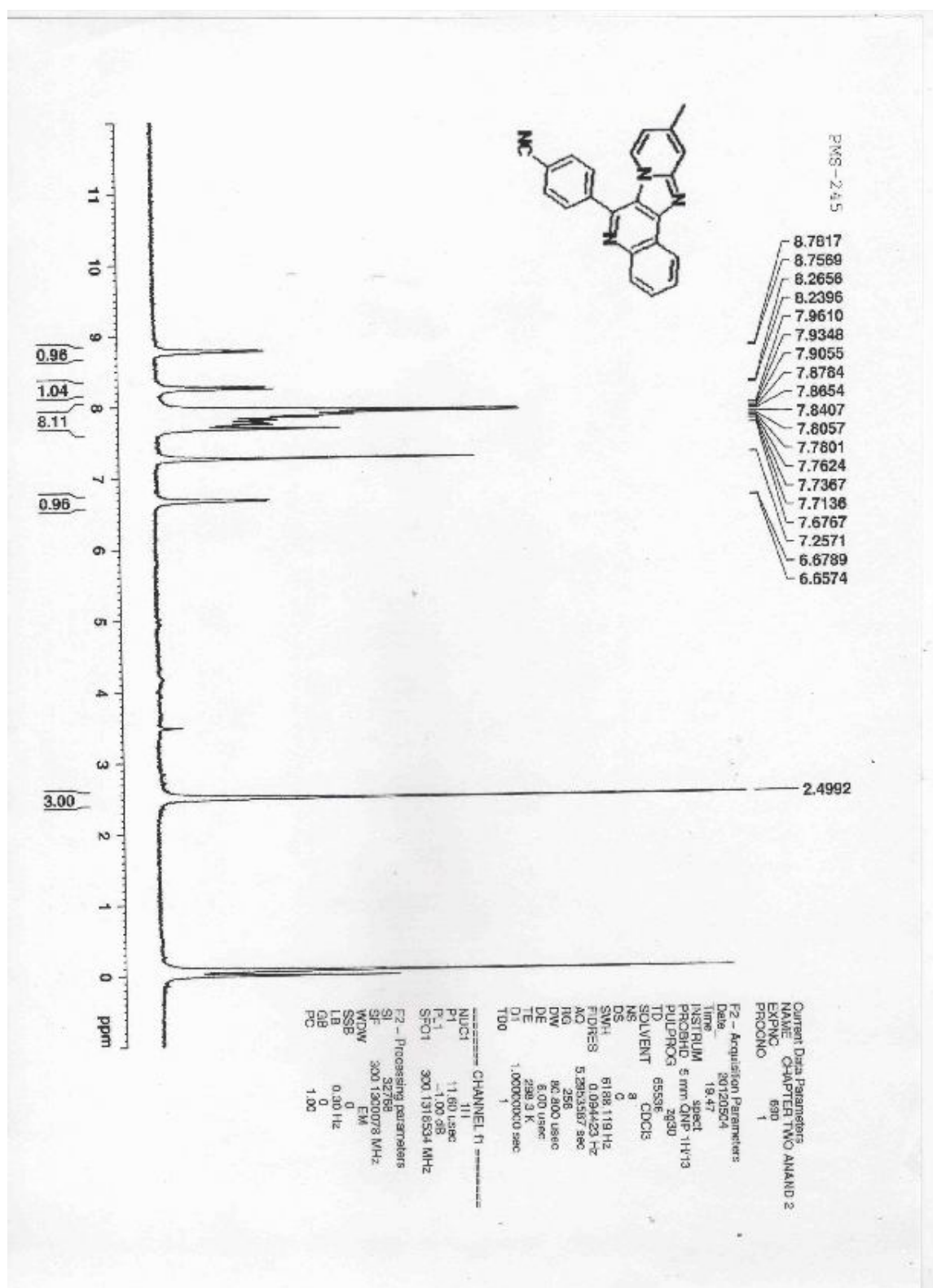


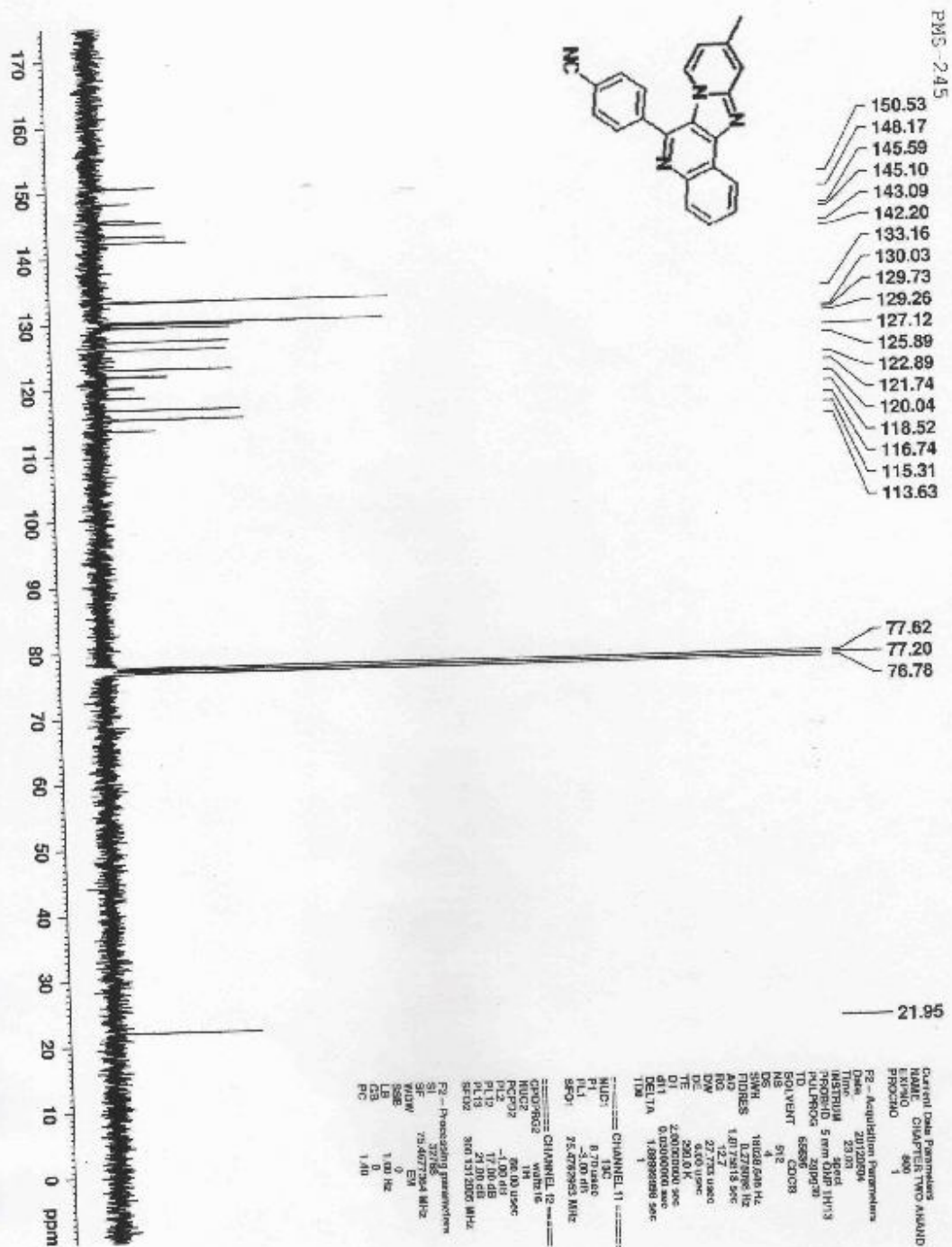
6-(3-Nitro-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9i):



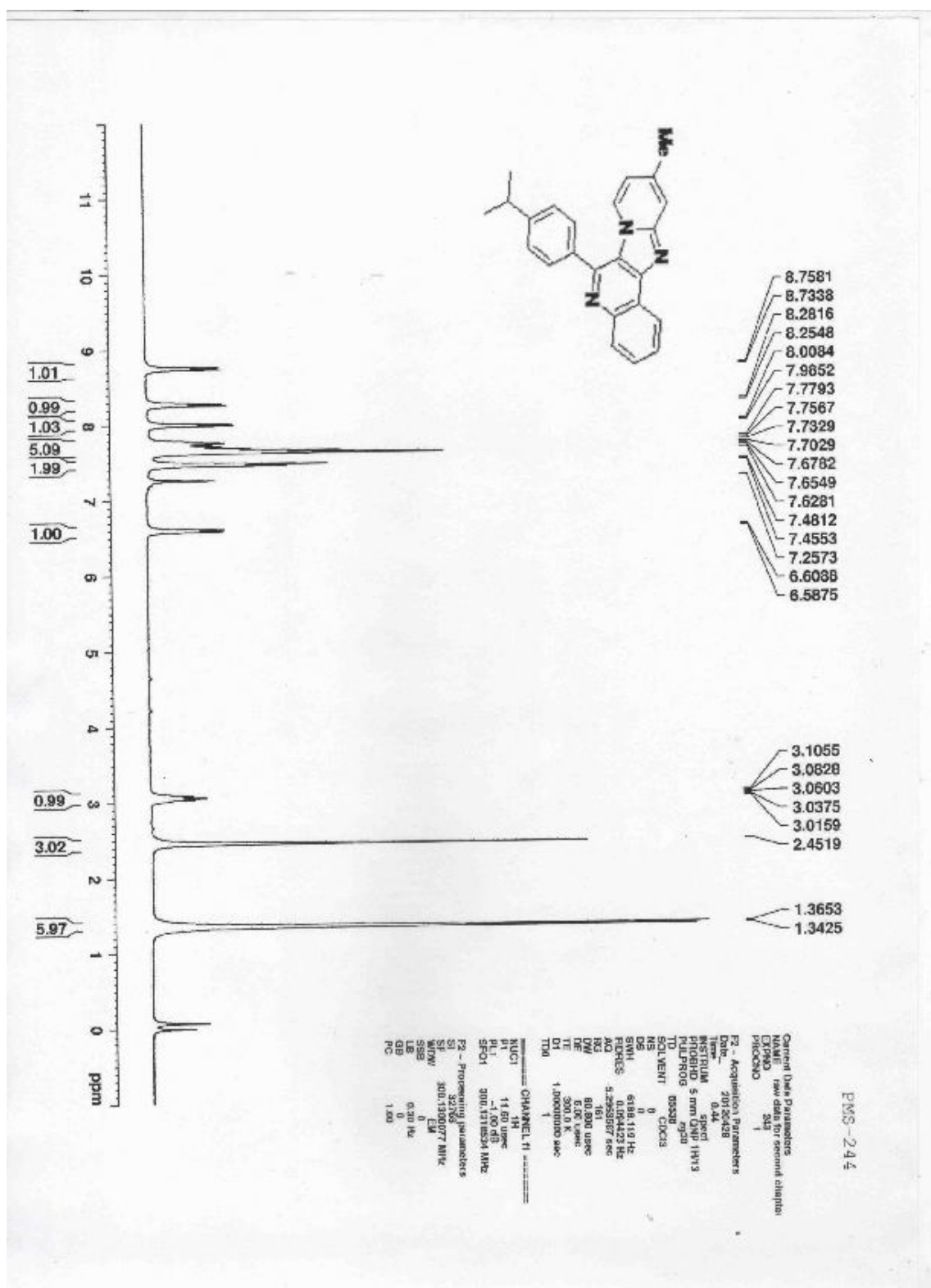


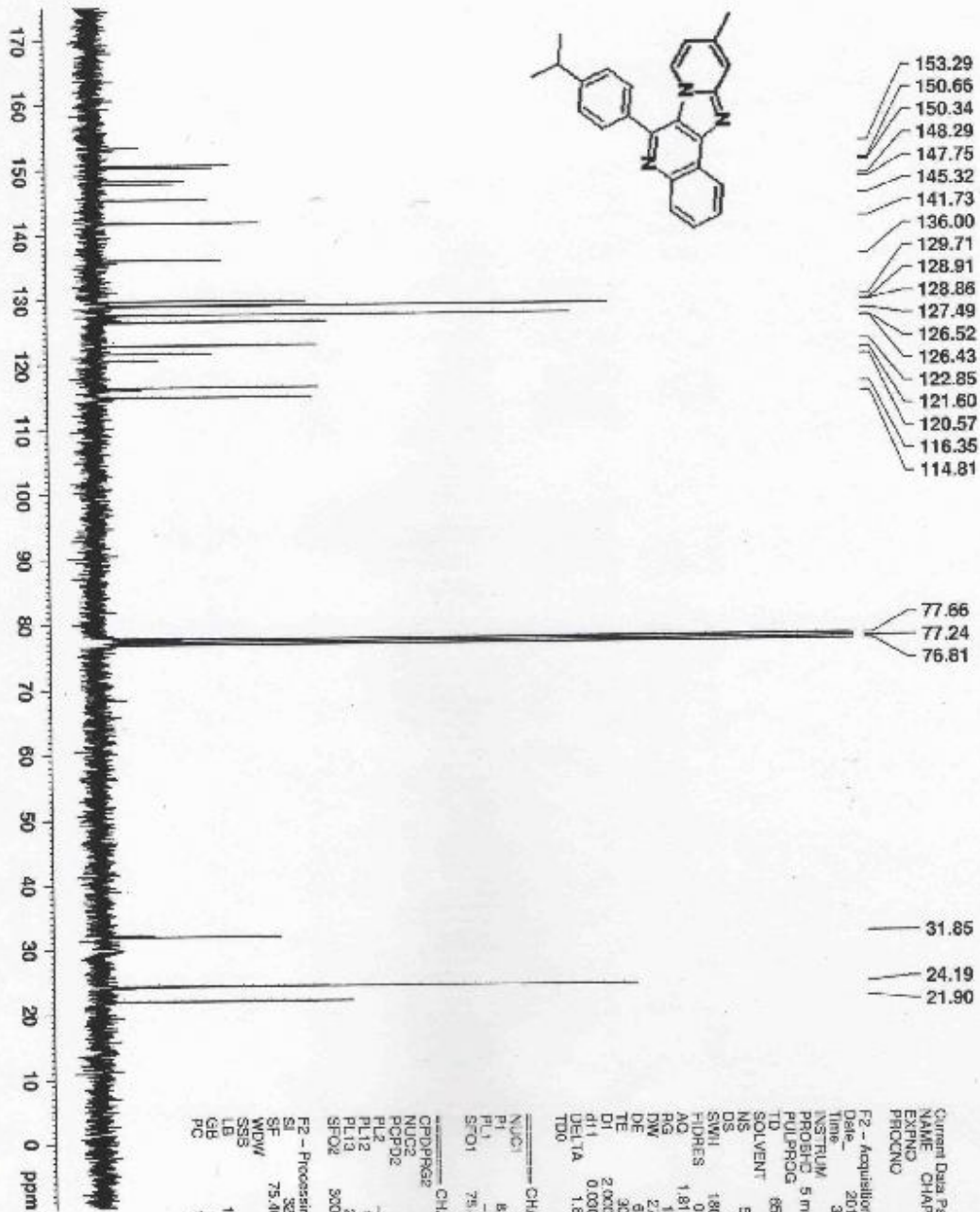
6-(4-Cyano-Phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9j):





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





PMS-244

Current Data Parameters
NAME: CHAPTER TWO.ANP
EXPNO: 800
PROCNO: 1

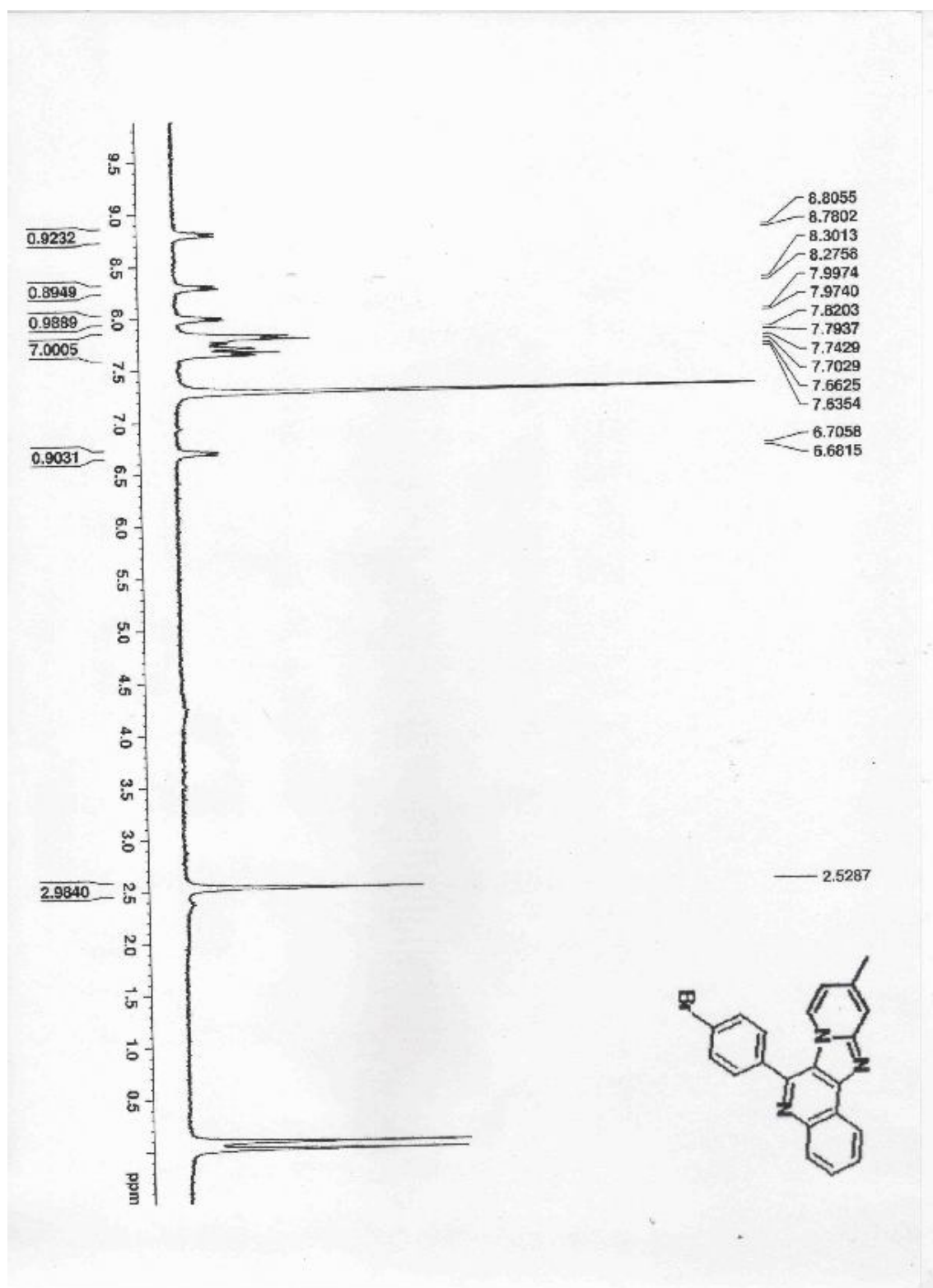
F2 - Acquisition Parameters
Date_: 20120428
Time: 3.55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 4
SWH: 18028.846 Hz
FIDRES: 0.273096 Hz
AQ: 1.8175918 sec
RG: 12.7
RW: 27.733 usec
DE: 5.00 usec
TE: 300.0 K
D1: 2.00000000 sec
d11: 0.00000000 sec
DELTA: 1.89999986 sec
TD0: 1

CHANNEL f1
NUC1: 13C
P1: 8.70 usec
PL1: -3.00 dB
SFO1: 75.4752853 MHz

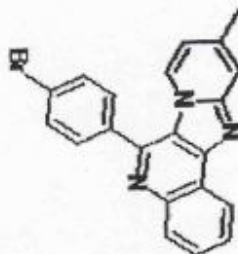
CHANNEL f2
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -1.00 dB
PL12: 17.00 dB
PL13: 21.00 dB
SFO2: 500.1312006 MHz

F2 - Processing parameters
SI: 32768
SF: 75.4677303 MHz
WDW: EM
SSS: 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

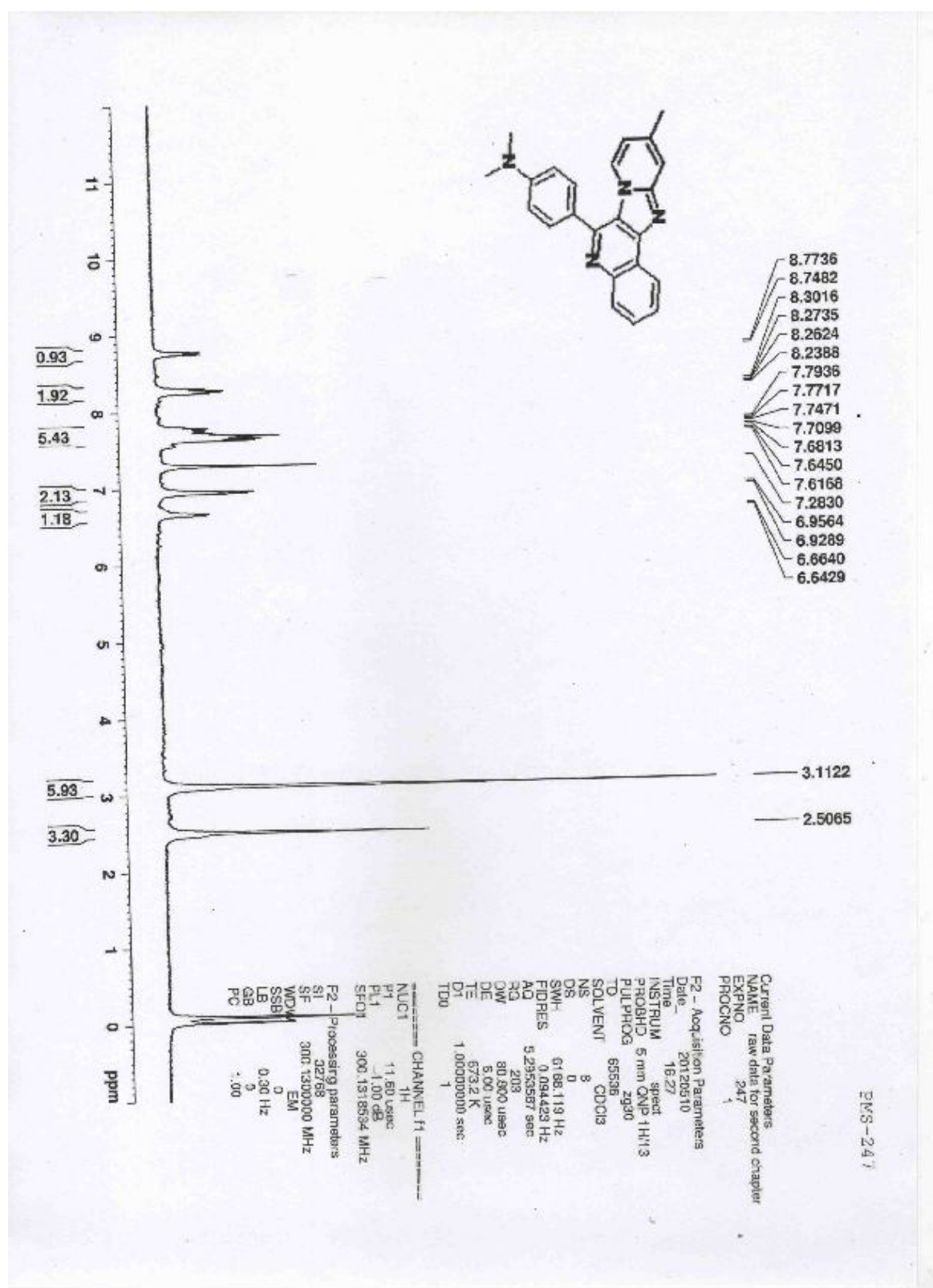
77.51
77.08
76.66

21.74

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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 10063.146 Hz
FIDRES 0.27358 Hz
AQ 1.01238 sec
RG 132
DQ 57.733 usec
DE 5.00 usec
TE 300.1 K
D1 2.00000000 sec
d11 0.05000000 sec
DELTA 1.20999999 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 76.4735053 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312603 MHz
F3 - Processing parameters
SI 32768
SF 75.4777471 MHz
WDW EM
SSB 0
GB 0
PC 1.20

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



PWS 247

Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 830
PROCNO 1

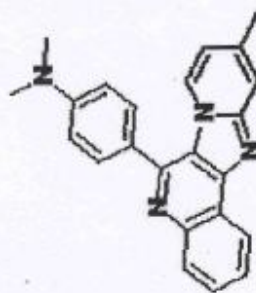
F2 - Acquisition Parameters

Date 20121119
Time 13:35
INSTRUM spect
PROBHD 5 mm Dual 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 11930.407 Hz
FIDRES 0.182619 Hz
AQ 2.7256011 sec
RG 508
DW 41.700 usec
DE 6.00 usec
TE 0.31 K
D1 2.00000000 sec
d11 0.05000000 sec
DELTA 1.80000000 sec
INOCST 1.00000000 sec
INOCNK 0.01500000 sec

CHANNEL 1
NUC1 13C
P1 6.30 usec
PL -6.00 dB
SFO1 50.3277608 MHz

CHANNEL 2
CPURGE wait16
NUC2 1H
P2 130.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz

F2 - Processing parameters
SI 32768
SF 50.3277227 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



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

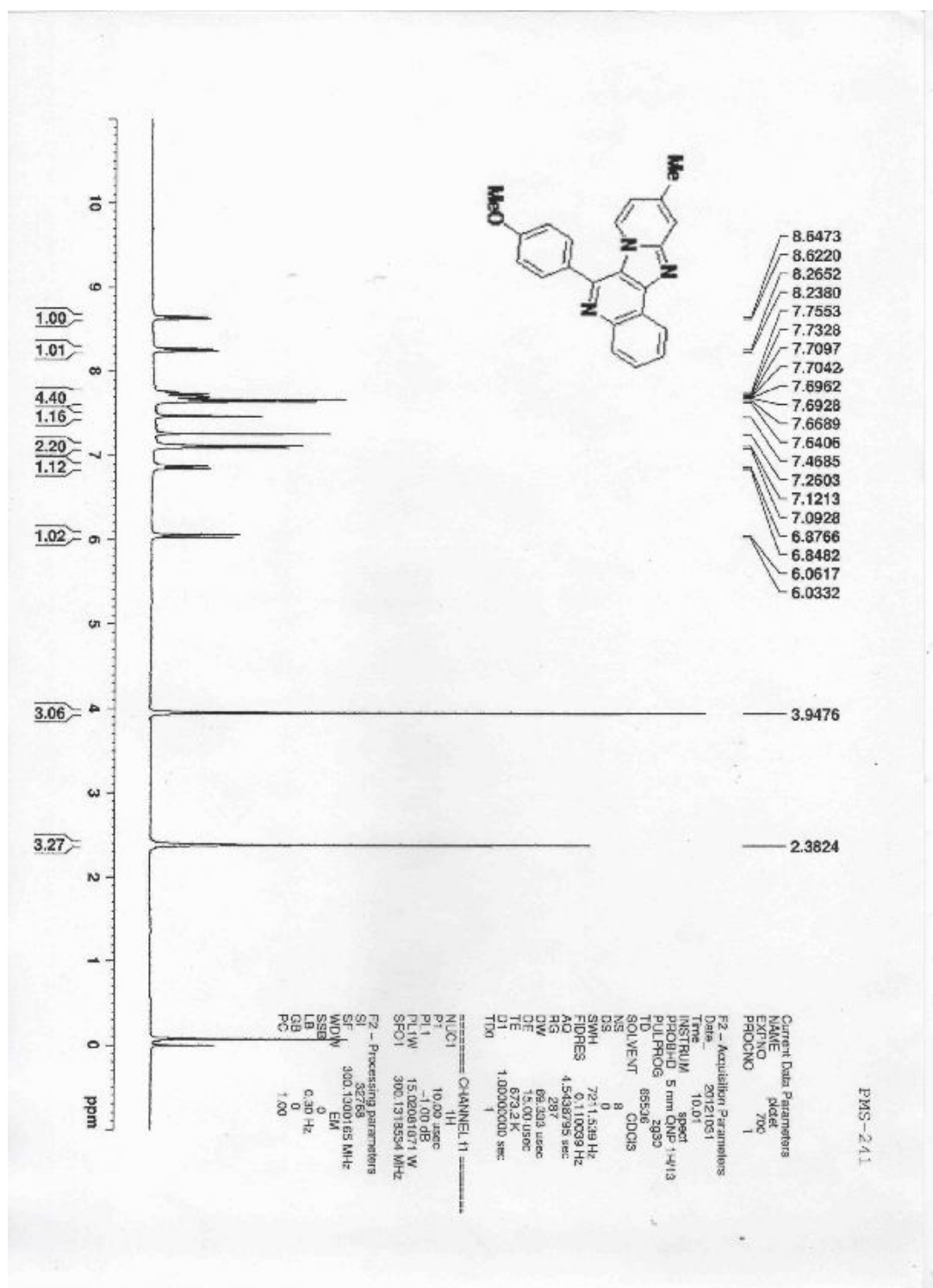
77.85
77.21
76.58

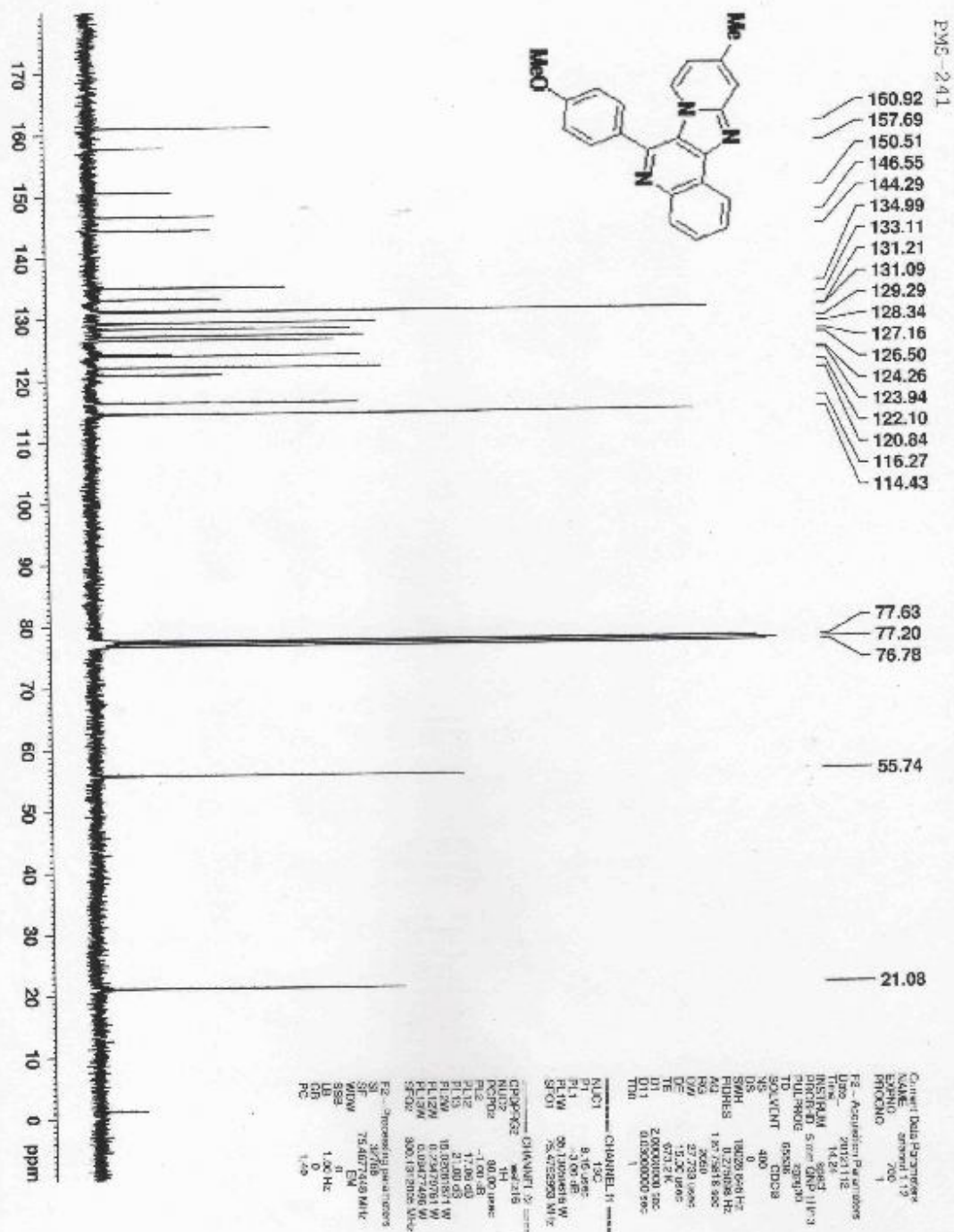
40.54

21.83

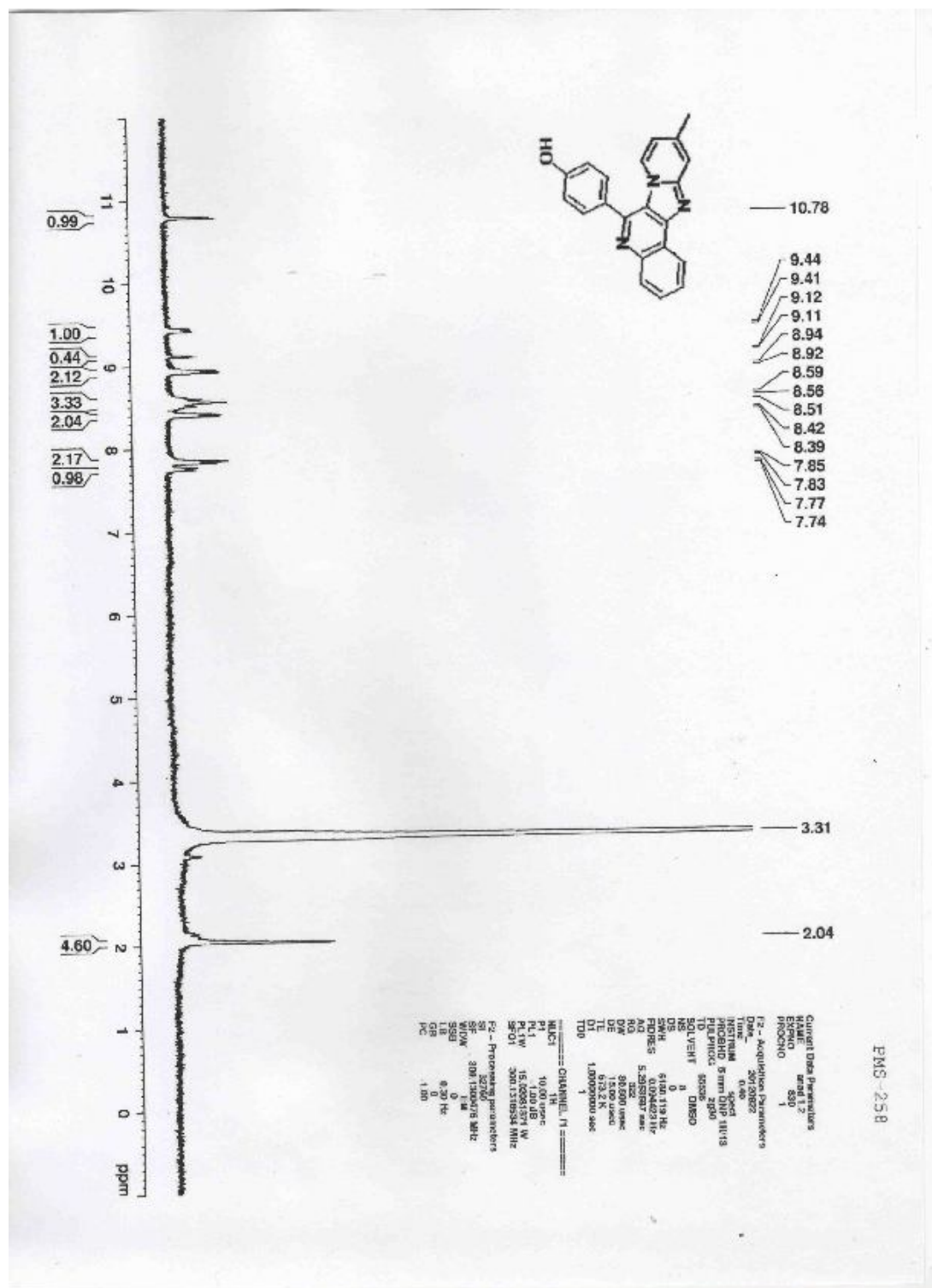
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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

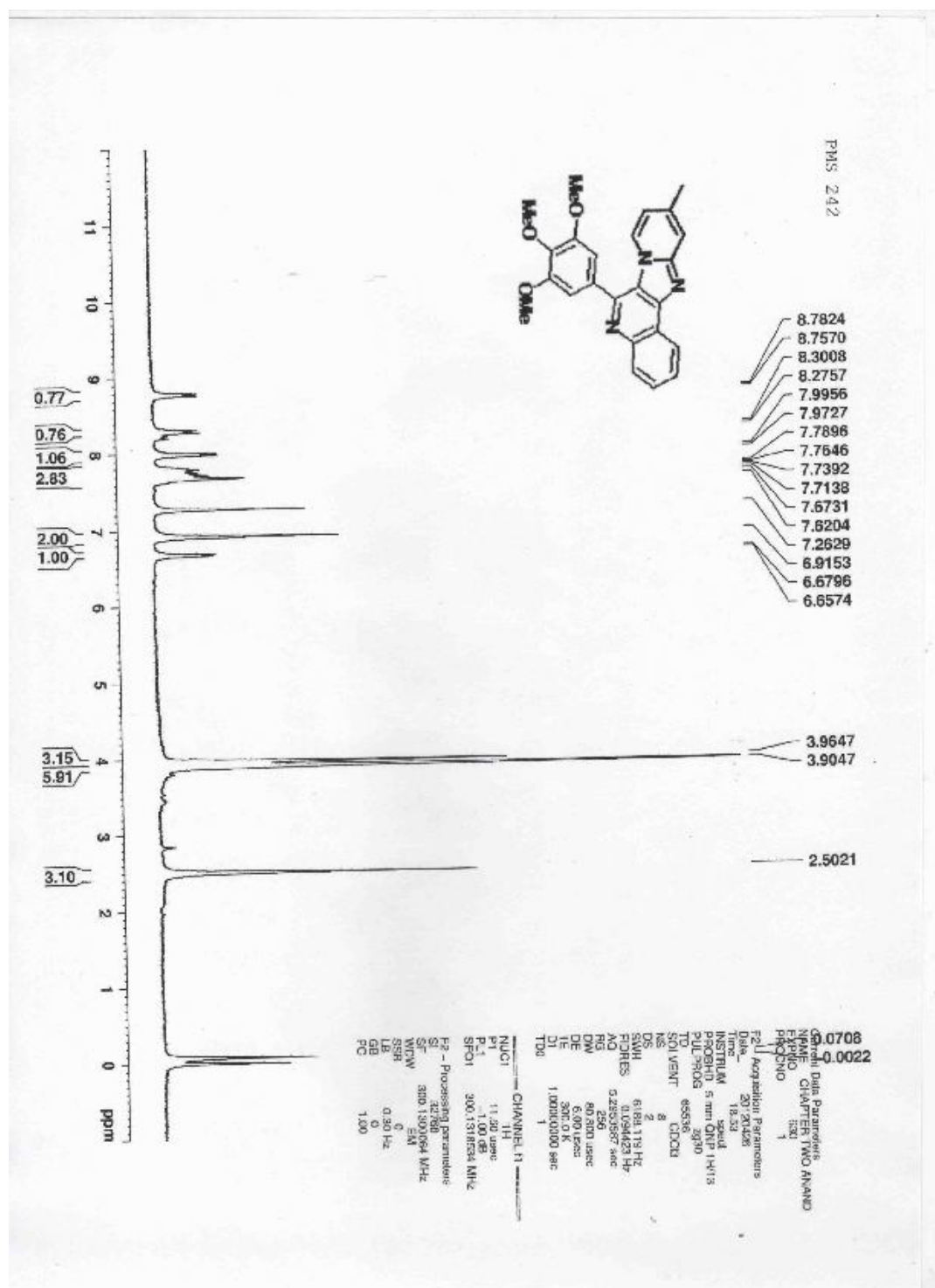




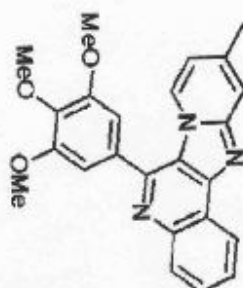
4-(9-Methyl-5,6b,11-triaza-benzo[a]fluoren-6-yl)-phenol (9o):



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
114.68
105.87

77.94
77.30
76.67

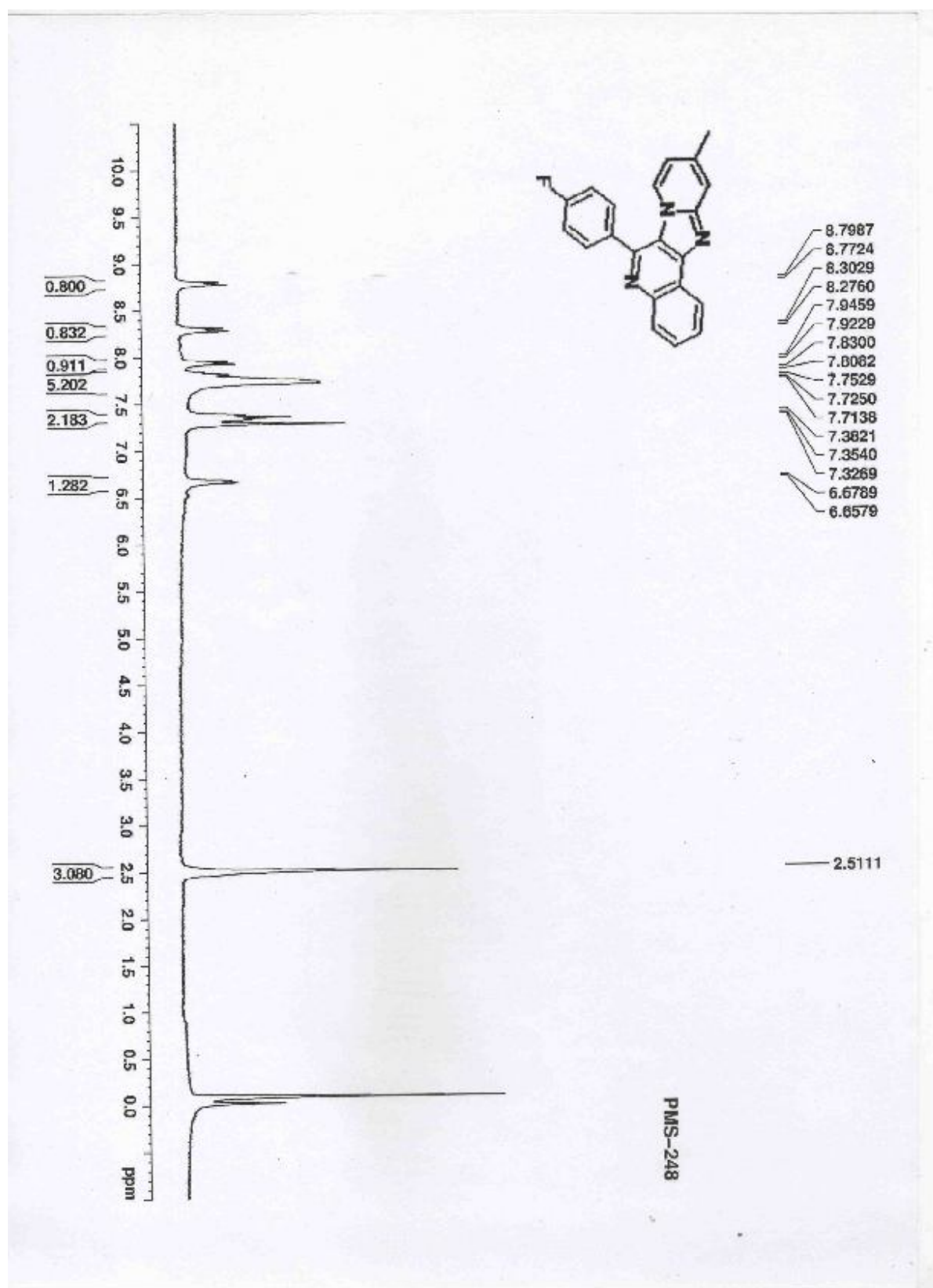
61.21
56.53

21.90

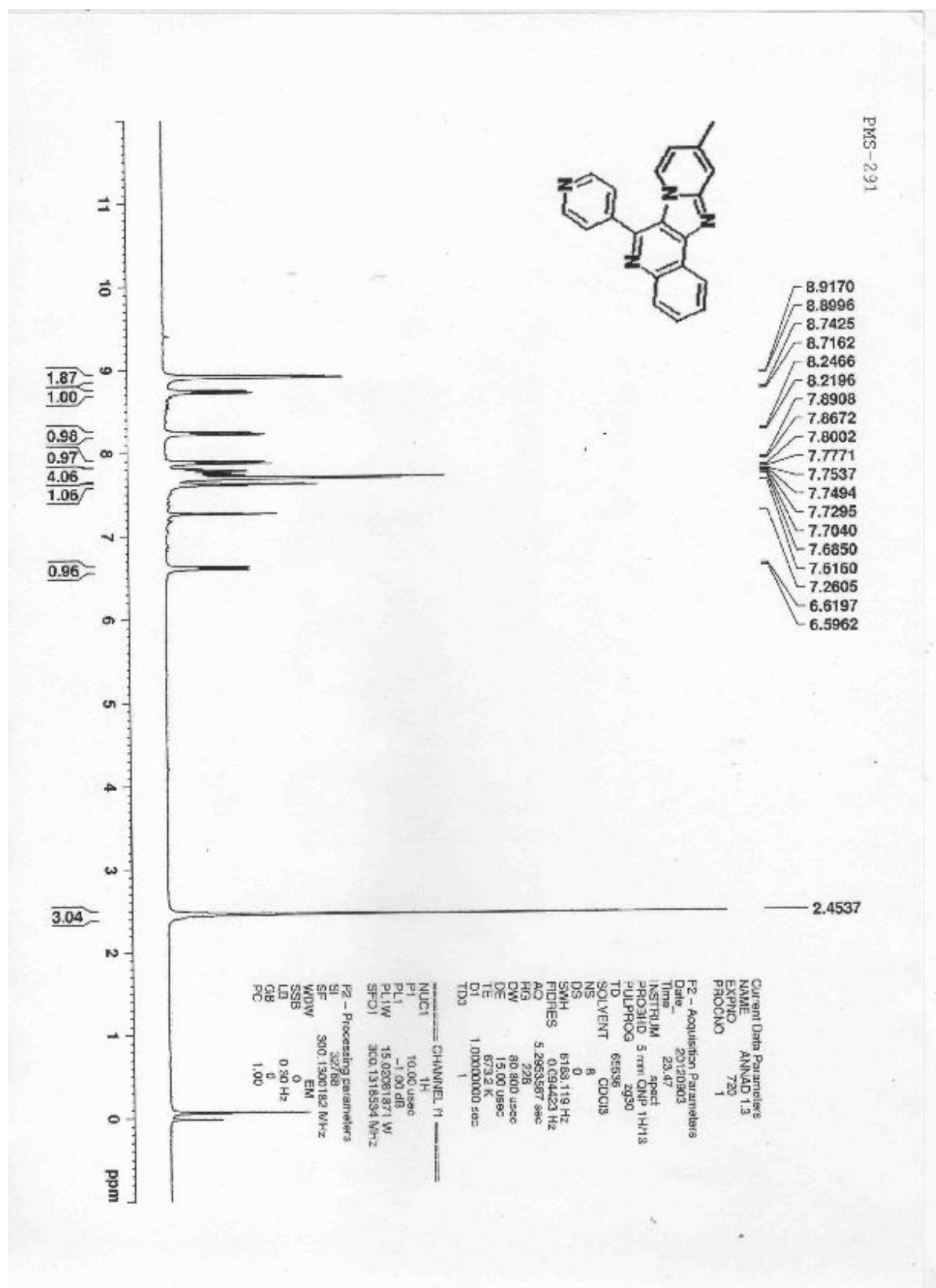
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
NAME: ACQ0-2017-07-02
EXPNO: 610
PROCNO: 1
F2 - Acquisition Parameters
Date_ : 20170702
Time : 11.01
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 168
DS: 4
SWH: 11883.407 Hz
FIDRES: 0.700559 Hz
AQ: 2.733911 s
RG: 327.5
CQ: 41.700 usec
DE: 6.00 usec
TE: 0.0 K
D1: 2.00000000 sec
d11: 0.02000000 sec
DELTA: 1.80000000 sec
NUC1: 13C
NUC2: 1H
P1: 6.00 sec
PL1: 0.00 dB
SFO1: 90.00125 MHz
SFO2: 400.1460000 MHz
CHANNEL F2
NUC1: 13C
P1: 6.00 sec
PL1: 0.00 dB
SFO1: 90.00125 MHz
SFO2: 400.1460000 MHz
CHANNEL F1
NUC1: 1H
P1: 0.00 sec
PL1: 0.00 dB
SFO1: 400.1460000 MHz
SFO2: 90.00125 MHz
CHANNEL F2
NAME: ACQ0-2017-07-02
EXPNO: 610
PROCNO: 1
F2 - Processing Parameters
SI: 32768
SF: 90.0012500 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

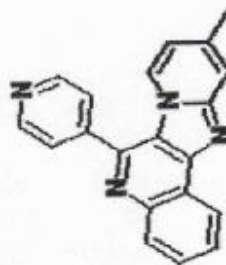
6-(4-Fluoro-phenyl)-9-methyl-5,6b,11-triaza-benzo[a]fluorene (9q):



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



FMS-291



- 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

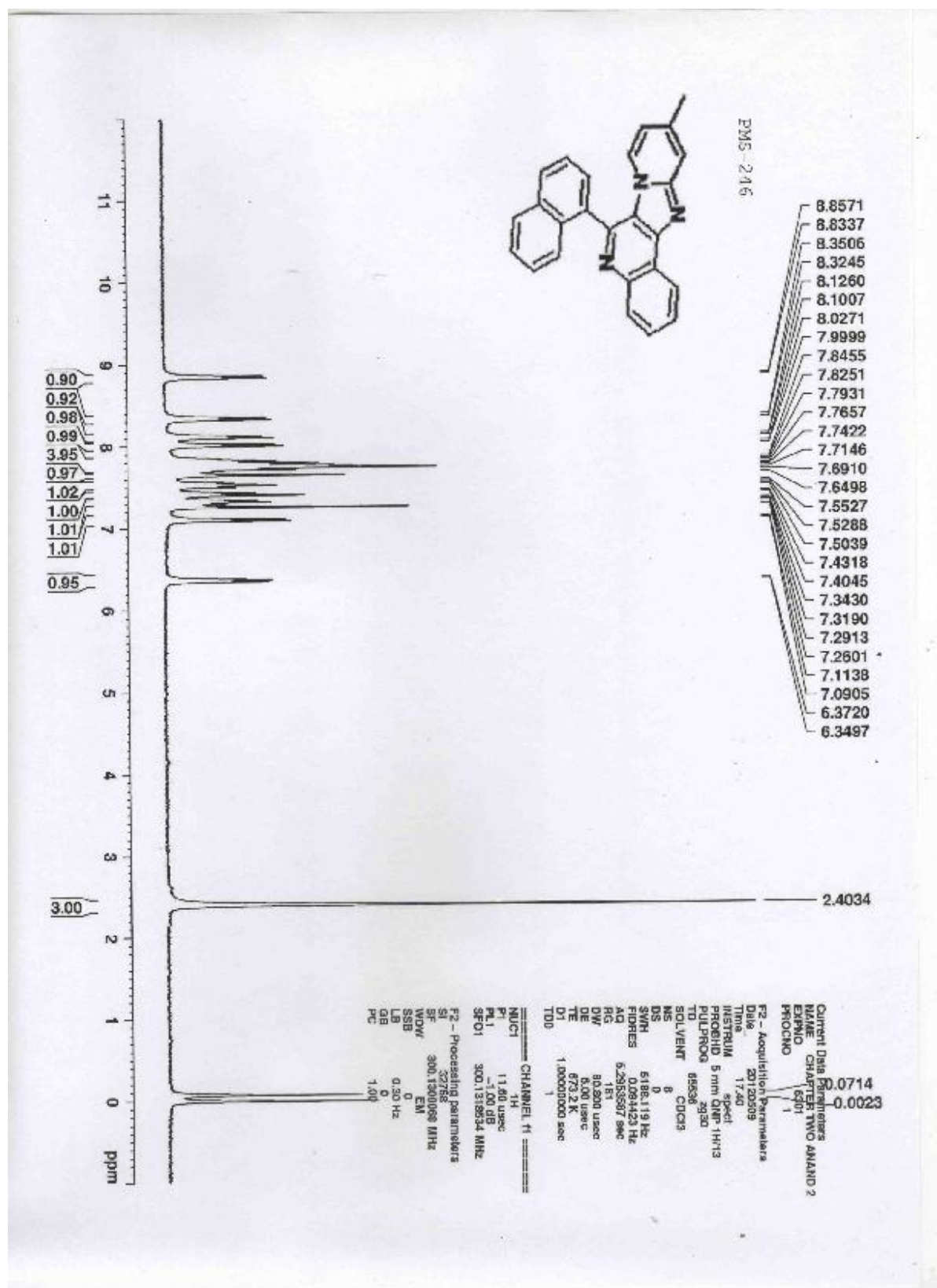
29.69

21.72

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Date: 10/20/2000
 Name: AMMO 13
 PIANO: 601
 PROCNO: 1
 PP - Acoustic Parameters
 TMR: 2.000000
 TMR2: 3.59
 INSTRUM: spect
 PCOBHD: 5mm QNP-1H/3
 PULPROG: zgpg30
 SFO: 600.131500
 SOLVENT: DMSO
 NS: 512
 DS: 4
 SWH: 18028.240 Hz
 FIDRES: 0.270000 Hz
 AQ: 1.480000 s
 RG: 327.68
 DW: 27.728 usec
 DE: 15.00 usec
 TE: 300.2 K
 D1: 2.0000000 sec
 D11: 0.0500000 sec
 TDO: 1
 CHANNEL 1:
 NUCL1: 13C
 P1: 8.15 usec
 PL1: 0.00 dB
 PL12: 19.00 dB
 PL1W: 16.1000000 W
 SFO1: 75.4732933 MHz
 CHANNEL 2:
 NUCL2: 1H
 P2: 1H
 PCPD2: 50.00 usec
 PL2: -1.00 dB
 PL12: 17.00 dB
 PL13: 17.00 dB
 PL14: 15.00 dB
 PL15: 15.00 dB
 PL16: 0.00 dB
 PL17: 0.00 dB
 PL18: 0.00 dB
 PL19: 0.00 dB
 SFO2: 300.1312005 MHz
 EQ - Proton Parameters
 SFO: 300.1312005 MHz
 SE: 75.4677480 MHz
 WCNV: 1.02
 SSF5: 0
 LE: 1.00 Hz
 CG: 1.00
 FG: 1.00

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

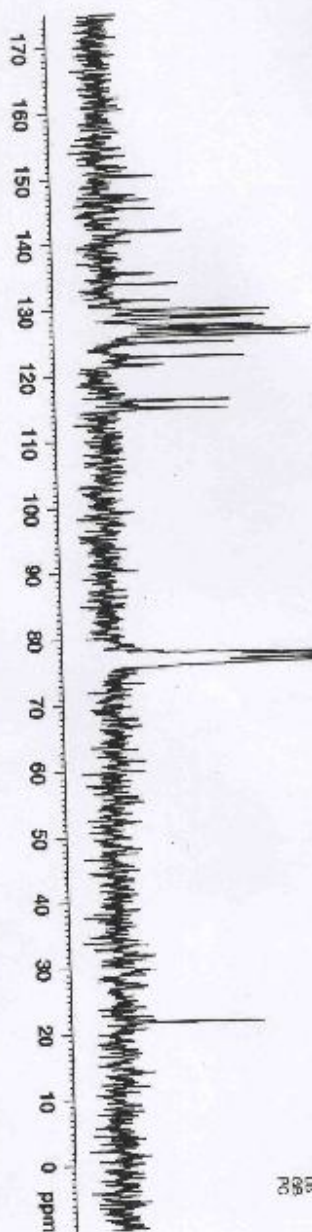
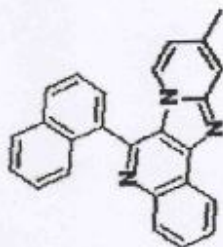


PMS-246

- 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

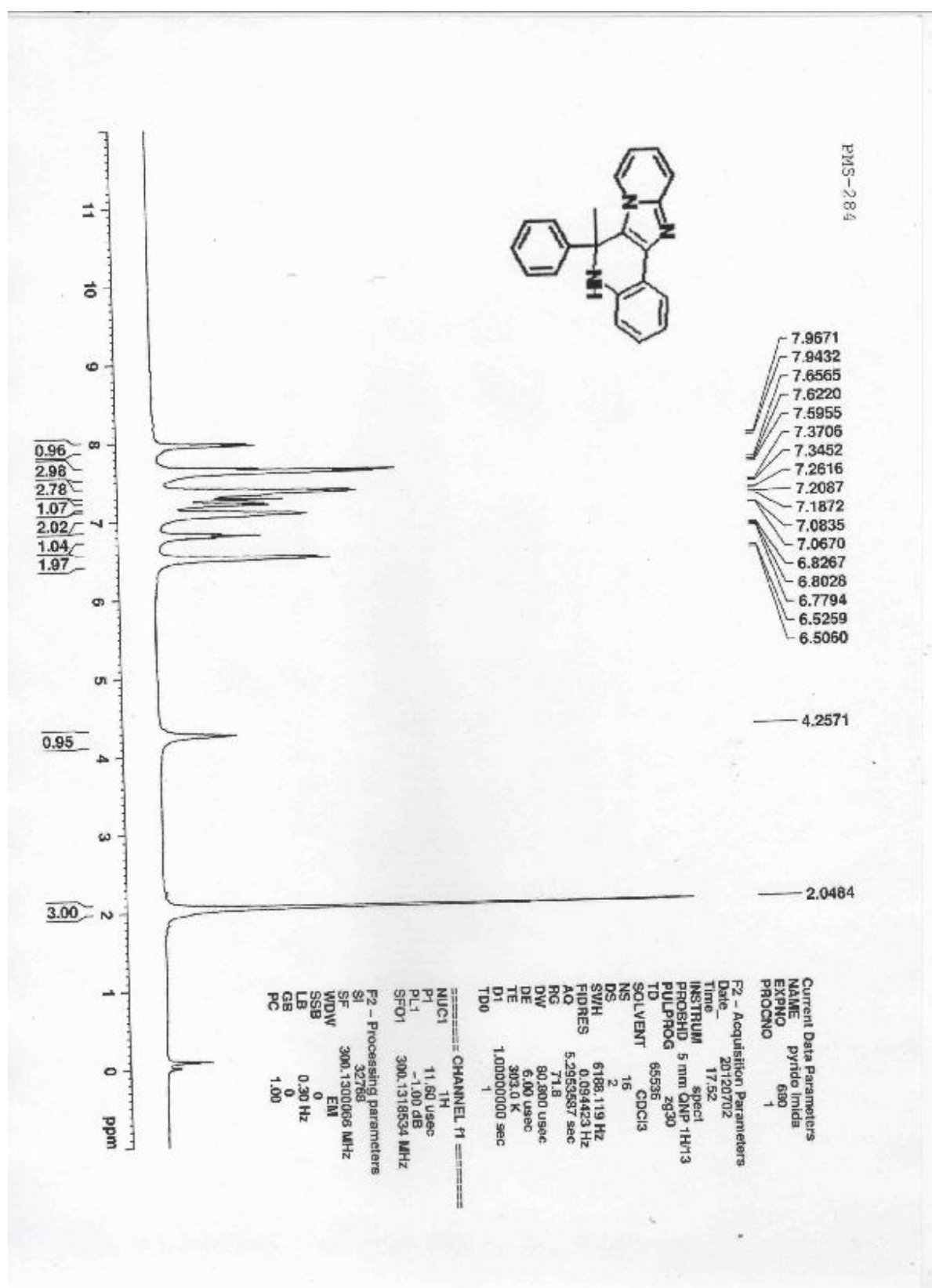
- 77.67
- 77.03
- 76.40

21.70



Current Data Parameters
 NAME: 2406-2012-DP12
 EXPNO: 333
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 2012/10/7
 Time: 15.31
 INSTRUM: spect
 PROTD: 5 cm BBO 15C
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 664
 DS: 4
 SWH: 11990.400 Hz
 FIDRES: 0.18259 Hz
 AQ: 2.732011 sec
 RG: 384
 DWD: 4.700 Hz
 DE: 6.00 Hz
 TE: 300.2 K
 D1: 2.00000000 sec
 e11: 0.00000000 sec
 DELTA: 1.80000000 sec
 ACQ-INT: 100000000 Hz
 ACQ-BW: 0.00000000 Hz
 CHANNEL f1 13C
 NUC1: 13C
 P1: 6.50 Hz
 PL1: -9.00 dB
 SFO1: 50.327000 MHz
 CHANNEL f2 1H
 C-DEPRG: waltz16
 NUC2: 1H
 P2PDC2: 100.00 Hz
 PL2: -4.00 dB
 PL12: 19.00 dB
 PL13: 22.00 dB
 SFO2: 200.130000 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 50.327290 MHz
 WDW: EM
 SSB: 0
 LB: 3.00 Hz
 GB: 0
 PC: 1.40

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



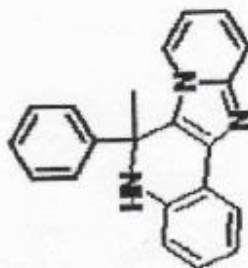
PMS-284

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

Current Data Parameters
NAME ANDIE
EXPNO 620
PROCNO 1

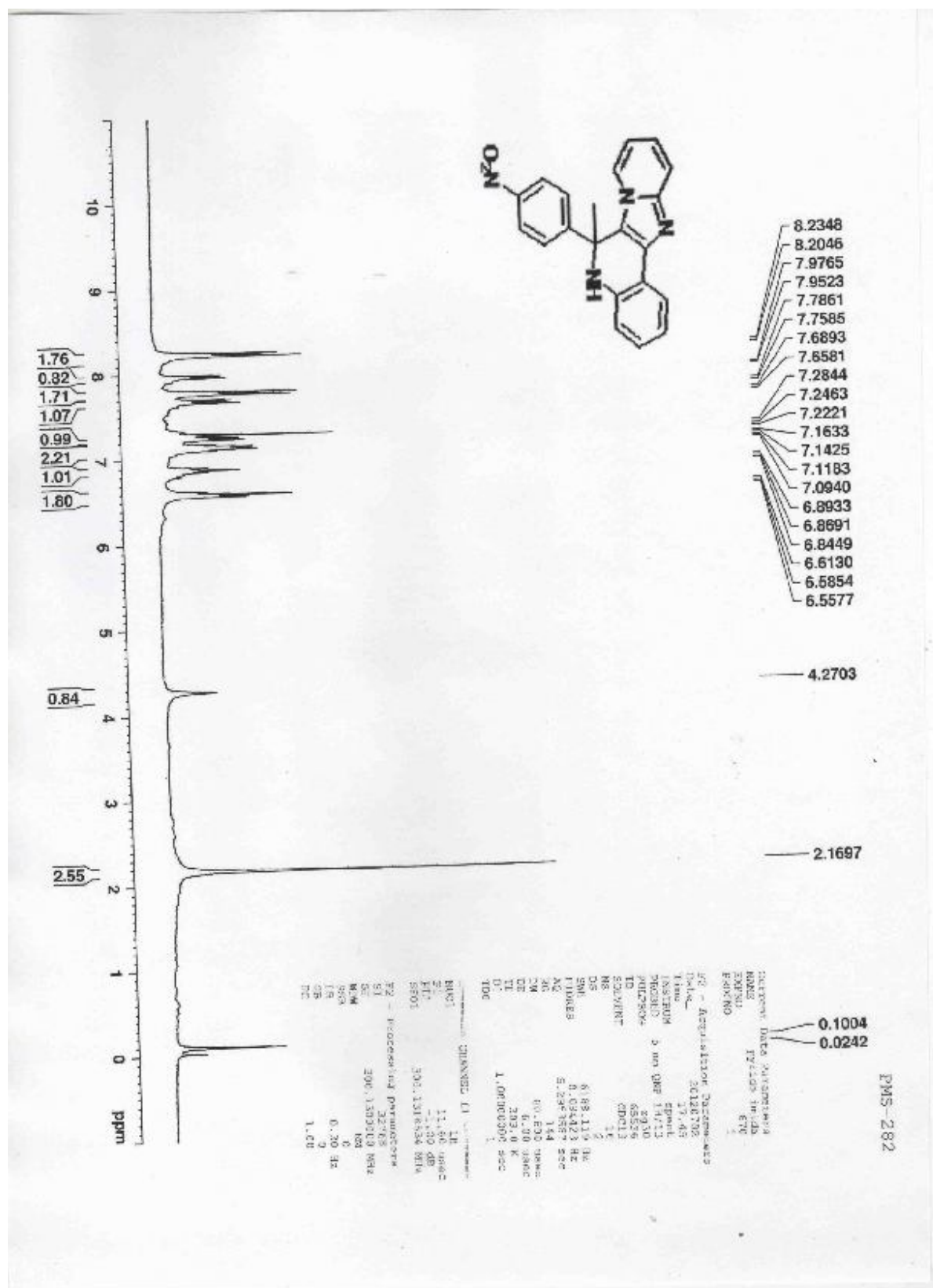
F2 - Acquisition Parameters
Date_ 20120710
Time 15:25
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 11580.407 Hz
FIDRES 0.18295 Hz
AQ 2.732901 sec
RG 71.8
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.88989898 sec
MCREST 0.00000000 sec
MCWET 0.01500000 sec

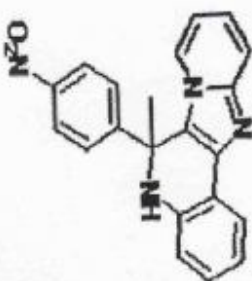
CHANNEL F1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.327690 MHz

CHANNEL P2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.130800 MHz

F2 - Processing parameters
SI 32768
SF 50.327690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

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






 84.77
 64.11
 83.45

- 63.30

- 46.02
 - 45.60
 - 45.18
 - 44.77
 - 44.35
 - 43.93
 - 43.51

— 30.99

```
Current Data Parameters
NAME      ANDF
EXTNO     640
PROCNO    1
```

F2 - Accelerator Parameter
Date 20/20/12

File	Speed
INSTAL.M	6mm Dual 1900
PROG.BD	6mm Dual 1900

PULL-THROU	26900.30
ID	6528.96
POURMENT	CHOCOL

6/2
4

SWH 71990.407 Hz
FIDNES 0.162369 Hz

702	45.3
703	41.700 USDD
704	

6.00 uspc
0.0 K

U1
d11
DELTA
1.00000000 000
1.00000000 000

MACFEST	0.0000000
MACFET	0.0150000
MACFEX	0.0150000

CHINA
NUC-30

P1 6.30 JPM:
P1 1 -6.00 :R
SEC: 20 98775371 M

CHANNEL 2 -

CPD/PRES	SWANTZ 15
NUC2	TH
PRC02	THO GO USE

PI 2	-4.00 DB
PI 12	18.00 DB

PL 13 22.00.05
SFC2 2001-200305 N

1-2 - Processing parameters
51 32706

511.527.280 WIT
CM
WDM
0
CAR

300 Hz
0
GB

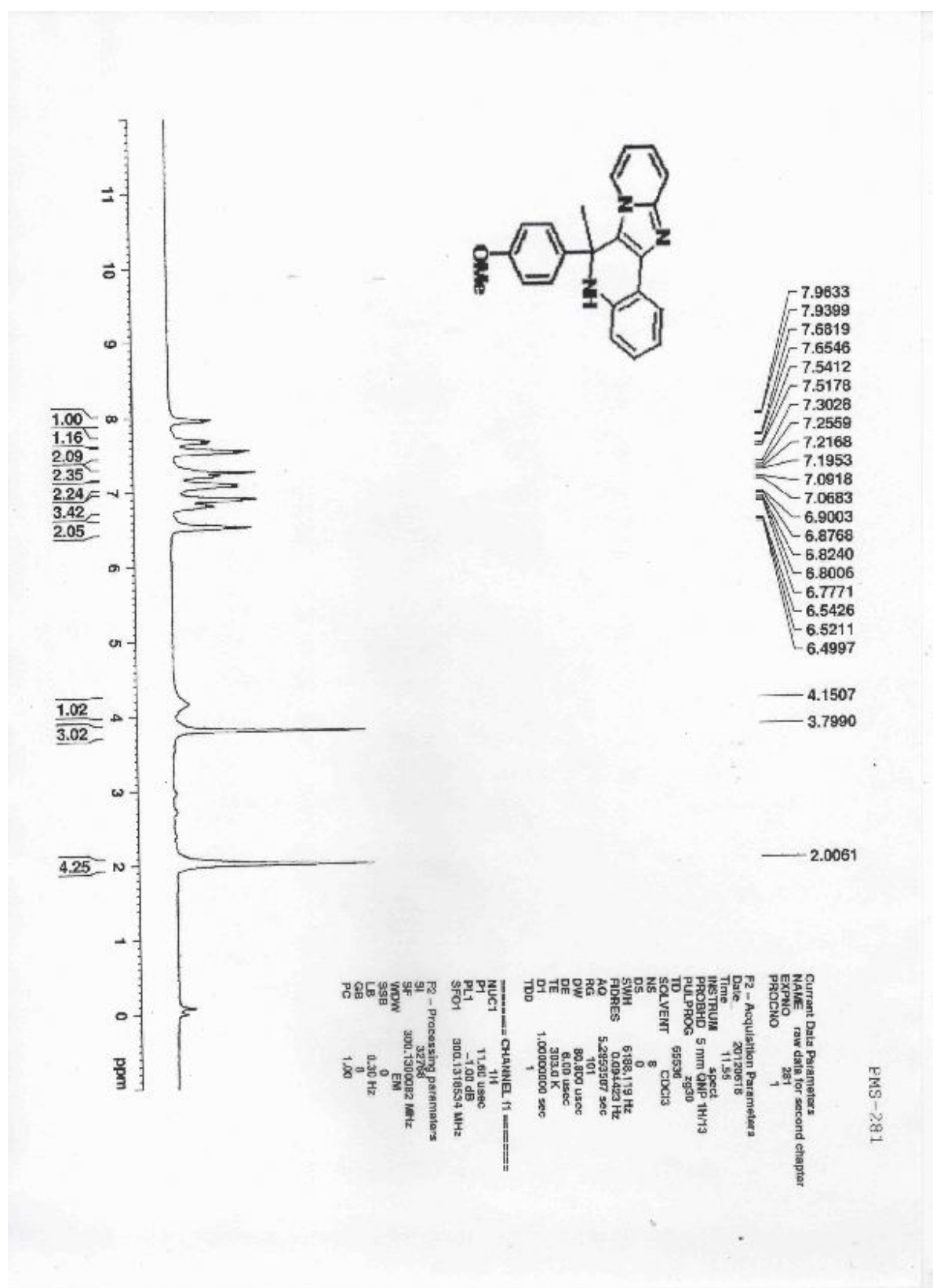
PC 1.40

Handwritten signature

10 0 ppm

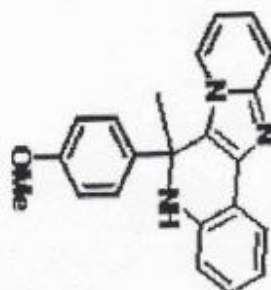
MLC1	CHANNEL 30	8.200 MHz
PL1		90.827650 MHz
SFO1		20.827650 MHz
CORPREG	CHANNEL 30	8.200 MHz
NULC2		8.200 MHz
PL202		90.827650 MHz
PL12		20.827650 MHz
PL13		20.827650 MHz
PL14		20.827650 MHz
PL15		20.827650 MHz
PL16		20.827650 MHz
PL17		20.827650 MHz
PL18		20.827650 MHz
PL19		20.827650 MHz
PL20		20.827650 MHz
PL21		20.827650 MHz
PL22		20.827650 MHz
PL23		20.827650 MHz
PL24		20.827650 MHz
PL25		20.827650 MHz
PL26		20.827650 MHz
PL27		20.827650 MHz
PL28		20.827650 MHz
PL29		20.827650 MHz
PL30		20.827650 MHz
PL31		20.827650 MHz
PL32		20.827650 MHz
PL33		20.827650 MHz
PL34		20.827650 MHz
PL35		20.827650 MHz
PL36		20.827650 MHz
PL37		20.827650 MHz
PL38		20.827650 MHz
PL39		20.827650 MHz
PL40		20.827650 MHz
PL41		20.827650 MHz
PL42		20.827650 MHz
PL43		20.827650 MHz
PL44		20.827650 MHz
PL45		20.827650 MHz
PL46		20.827650 MHz
PL47		20.827650 MHz
PL48		20.827650 MHz
PL49		20.827650 MHz
PL50		20.827650 MHz
PL51		20.827650 MHz
PL52		20.827650 MHz
PL53		20.827650 MHz
PL54		20.827650 MHz
PL55		20.827650 MHz
PL56		20.827650 MHz
PL57		20.827650 MHz
PL58		20.827650 MHz
PL59		20.827650 MHz
PL60		20.827650 MHz
PL61		20.827650 MHz
PL62		20.827650 MHz
PL63		20.827650 MHz
PL64		20.827650 MHz
PL65		20.827650 MHz
PL66		20.827650 MHz
PL67		20.827650 MHz
PL68		20.827650 MHz
PL69		20.827650 MHz
PL70		20.827650 MHz
PL71		20.827650 MHz
PL72		20.827650 MHz
PL73		20.827650 MHz
PL74		20.827650 MHz
PL75		20.827650 MHz
PL76		20.827650 MHz
PL77		20.827650 MHz
PL78		20.827650 MHz
PL79		20.827650 MHz
PL80		20.827650 MHz
PL81		20.827650 MHz
PL82		20.827650 MHz
PL83		20.827650 MHz
PL84		20.827650 MHz
PL85		20.827650 MHz
PL86		20.827650 MHz
PL87		20.827650 MHz
PL88		20.827650 MHz
PL89		20.827650 MHz
PL90		20.827650 MHz
PL91		20.827650 MHz
PL92		20.827650 MHz
PL93		20.827650 MHz
PL94		20.827650 MHz
PL95		20.827650 MHz
PL96		20.827650 MHz
PL97		20.827650 MHz
PL98		20.827650 MHz
PL99		20.827650 MHz
PL100		20.827650 MHz
PL101		20.827650 MHz
PL102		20.827650 MHz
PL103		20.827650 MHz
PL104		20.827650 MHz
PL105		20.827650 MHz
PL106		20.827650 MHz
PL107		20.827650 MHz
PL108		20.827650 MHz
PL109		20.827650 MHz
PL110		20.827650 MHz
PL111		20.827650 MHz
PL112		20.827650 MHz
PL113		20.827650 MHz
PL114		20.827650 MHz
PL115		20.827650 MHz
PL1		

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



PMS-281

- 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

Current Data Parameters
NAME: 20121226-0792
EXPNO: 640
PROCNO: 1

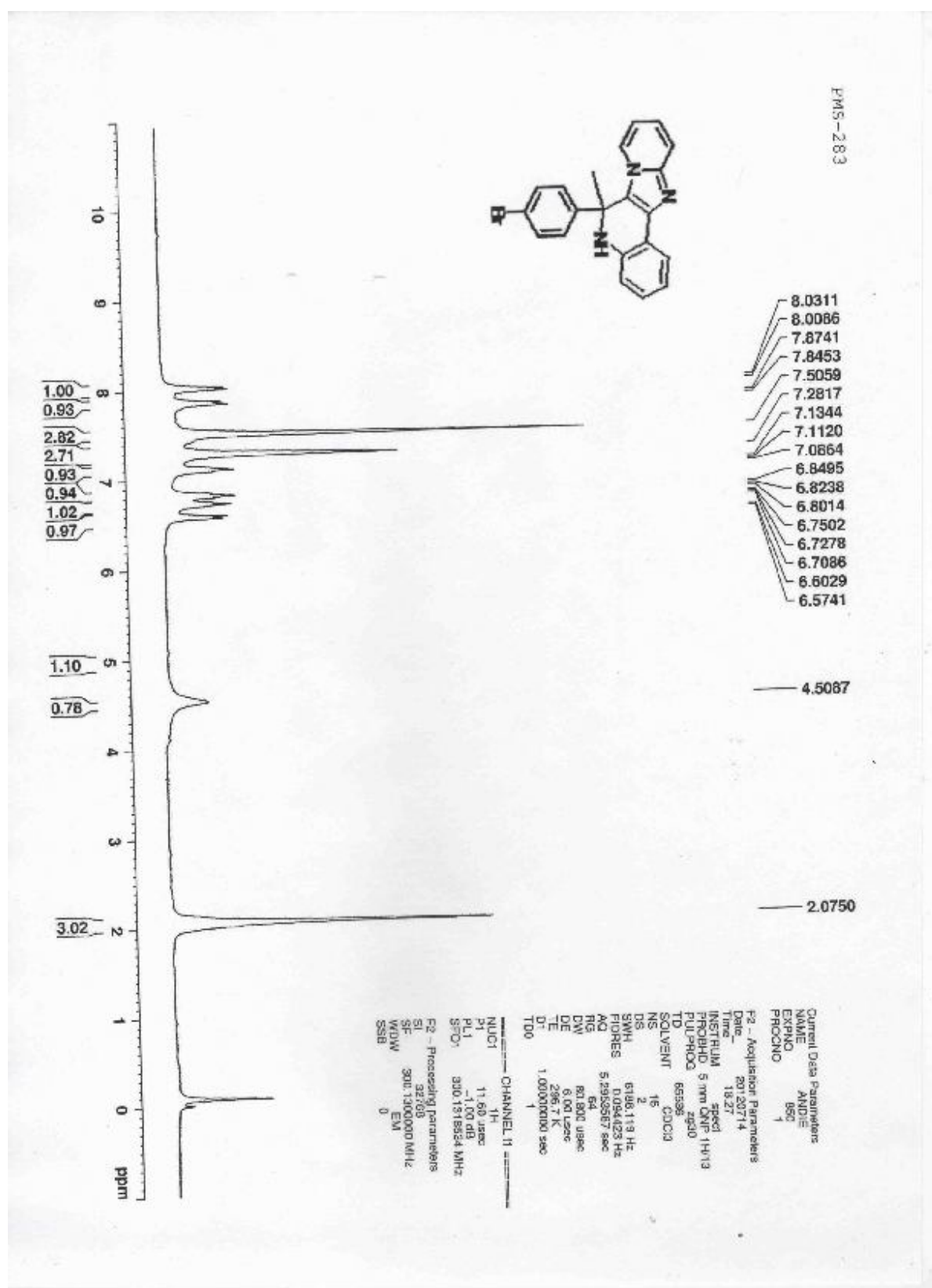
F2 - Acquisition Parameters
Date_ : 20121226
Time : 15:51
INSTRUM : spect
PROBHD : 5 mm DSS (1H/13C)
PULPROG : zgpg30
TD : 65536
SOLVENT : DMSO
NS : 417
DS : 4
SWH : 11980.07 Hz
FIDRES : 0.102346 Hz
AQ : 2.732011 sec
RG : 64
HG : 41.700 usec
WE : 18.00 usec
TE : 300.2 K
D1 : 2.0000000 sec
d11 : 0.0500000 sec
DELTA : 1.8000000 sec
MCPROG : 0.0000000 sec
MCWPRG : 0.0150000 sec

CHANNEL f1
NUC1 : 13C
P1 : 6.00 usec
PL1 : 0.00 dB
SFO : 50.3217600 MHz

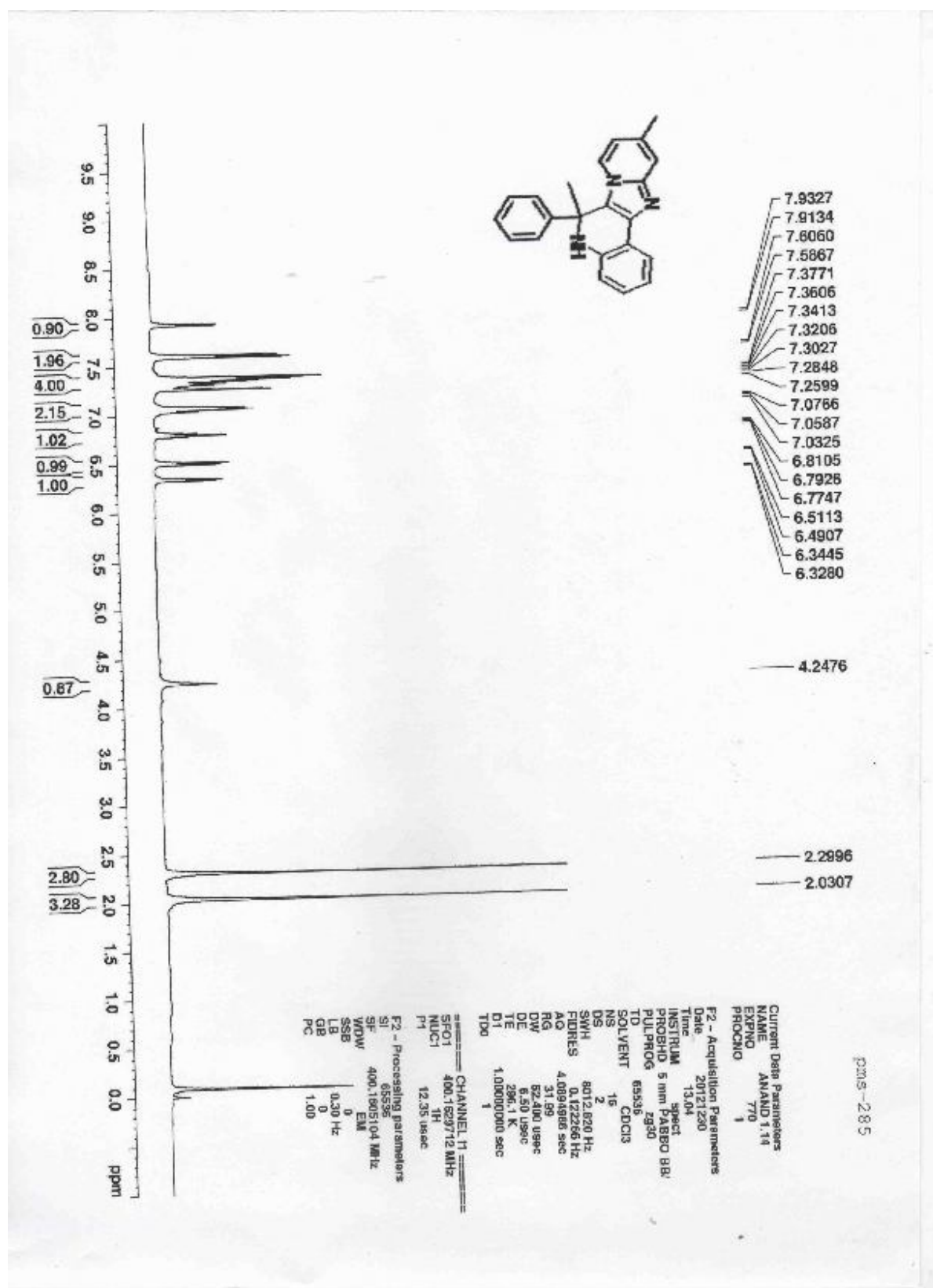
CHANNEL f2
NUC2 : 1H
P2 : 12.00 usec
PL2 : 0.00 dB
SFO2 : 200.1360000 MHz

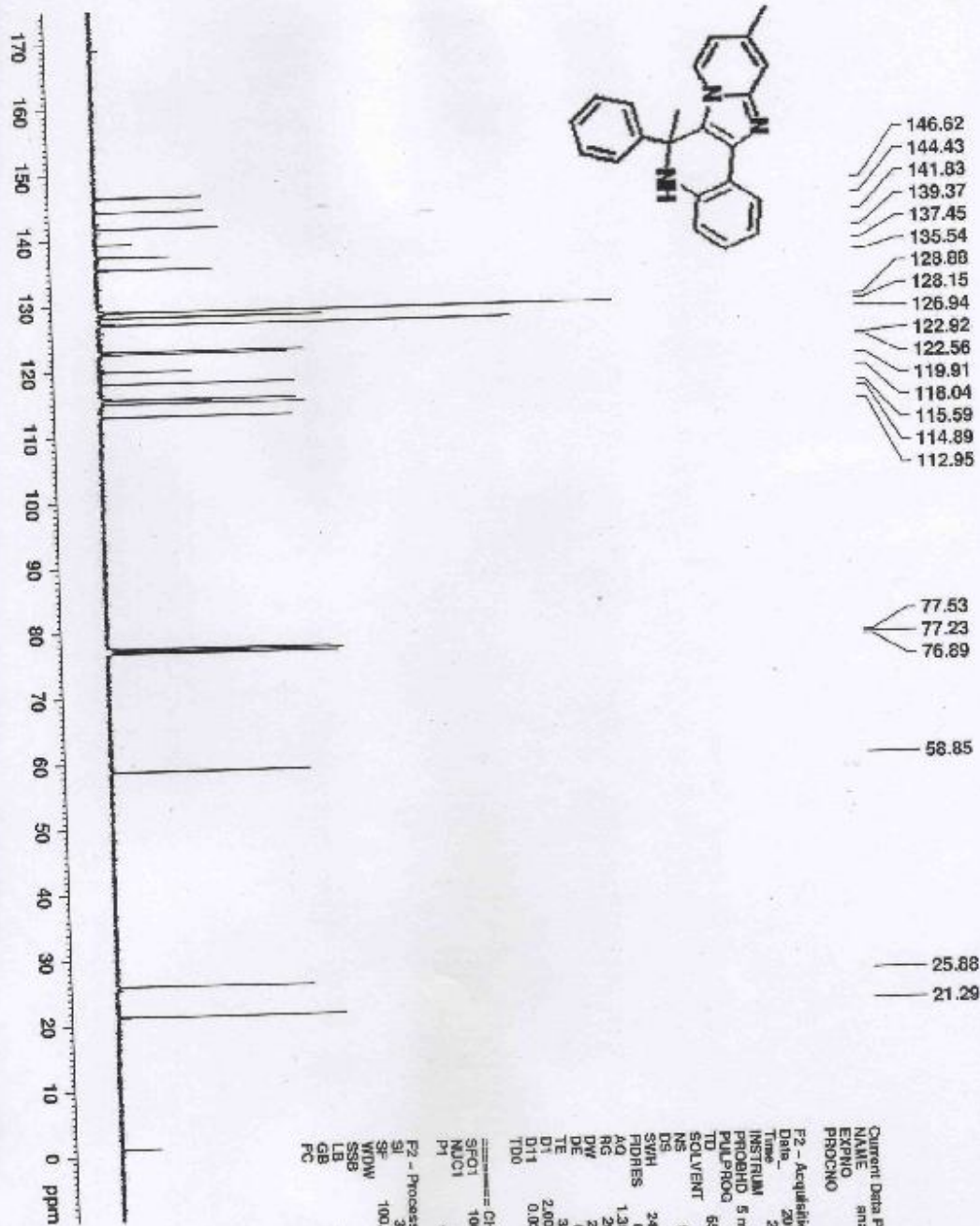
Processing parameters
SI : 32768
SF : 50.3217600 MHz
WDW : EM
SSB : 0
LB : 3.00 Hz
GB : 0
PC : 1.40

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):





Current Data Parameters
NAME: amad 1.18
EXPNO: 649
PROCNO: 1

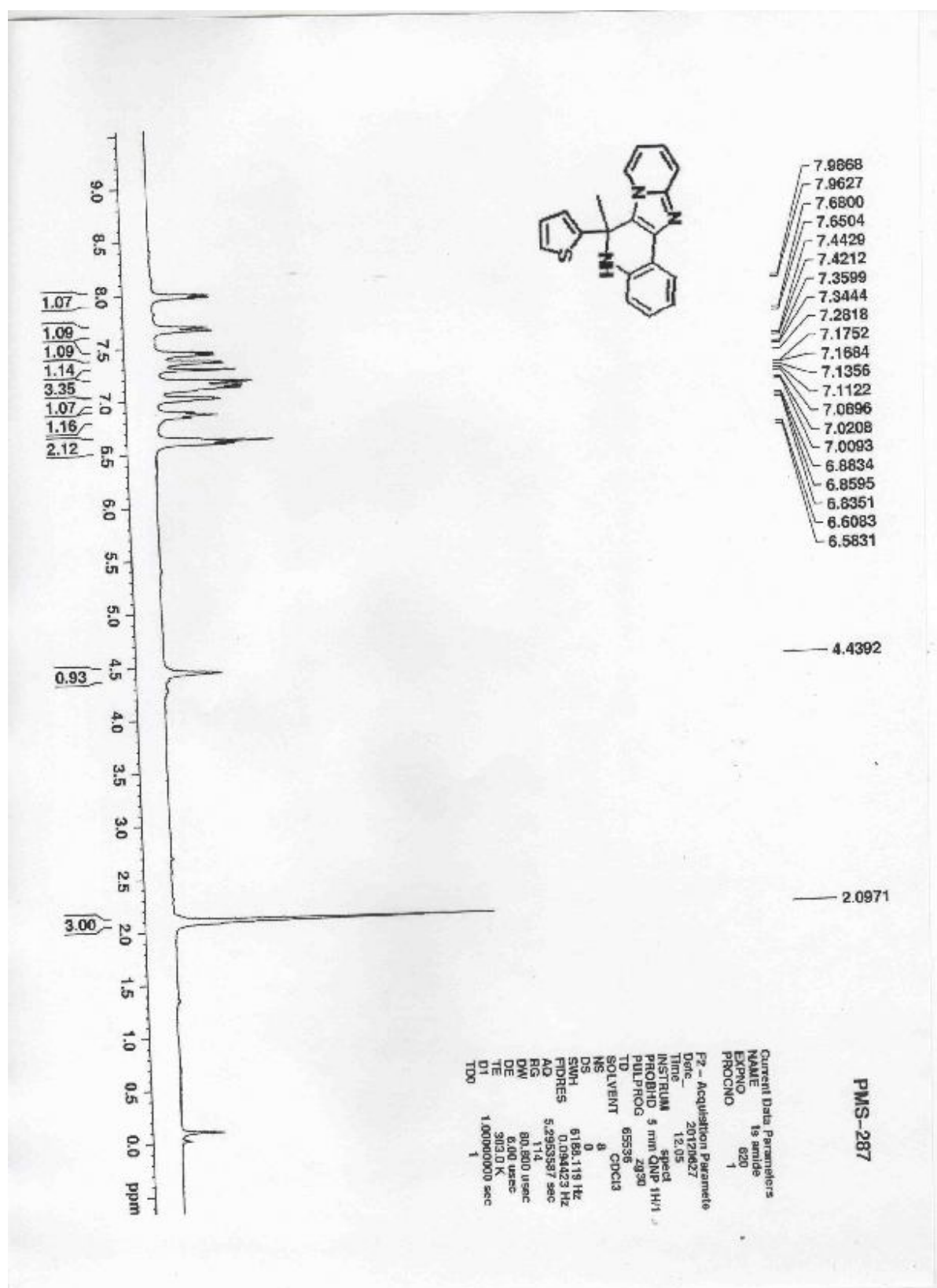
F2 - Acquisition Parameters
Date_: 20130102
Time: 21.49
INSTRUM: spect
PROBHD: 5 mm F4BBO BE
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 4
SVH: 24023.461 Hz
FIDRES: 0.366788 Hz
AQ: 1.3637988 sec
RG: 201.48
DW: 20.800 usec
DE: 6.50 usec
TE: 300.0 K
D1: 2.00000000 sec
D11: 0.00000000 sec
TD0: 1

===== CHANNEL f1 =====
SFO1: 100.620093 MHz
NUC1: 13C
P1: 8.20 usec

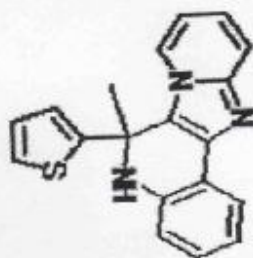
F2 - Processing parameters
SI: 32768
SF: 100.620093 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

PMG-285

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



PM5-287

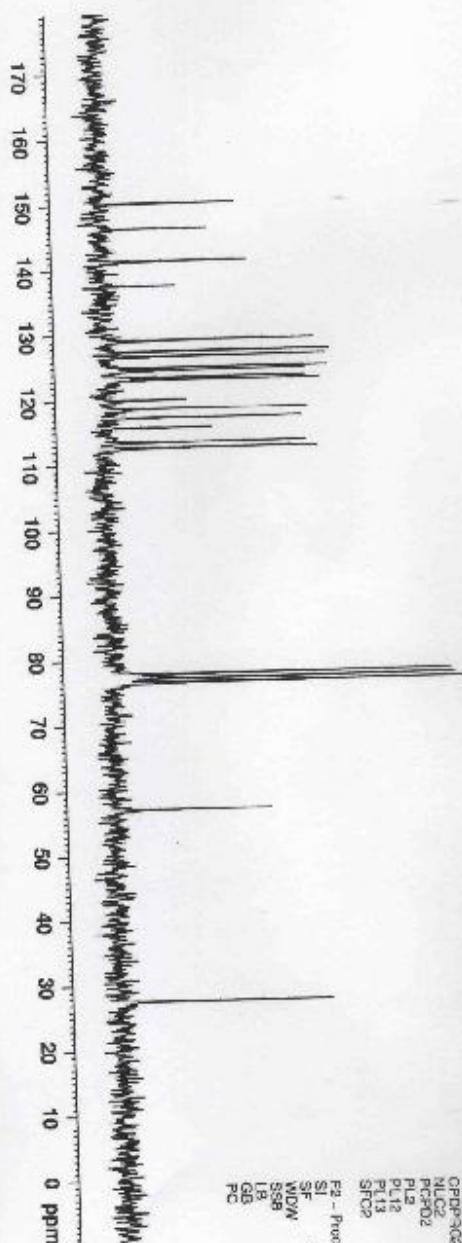


150.16
146.21
141.23
137.42
129.05
127.31
126.57
124.85
124.42
123.37
122.98
119.88
118.53
117.16
115.63
113.33
112.44

77.77
77.13
76.49

56.98

27.42



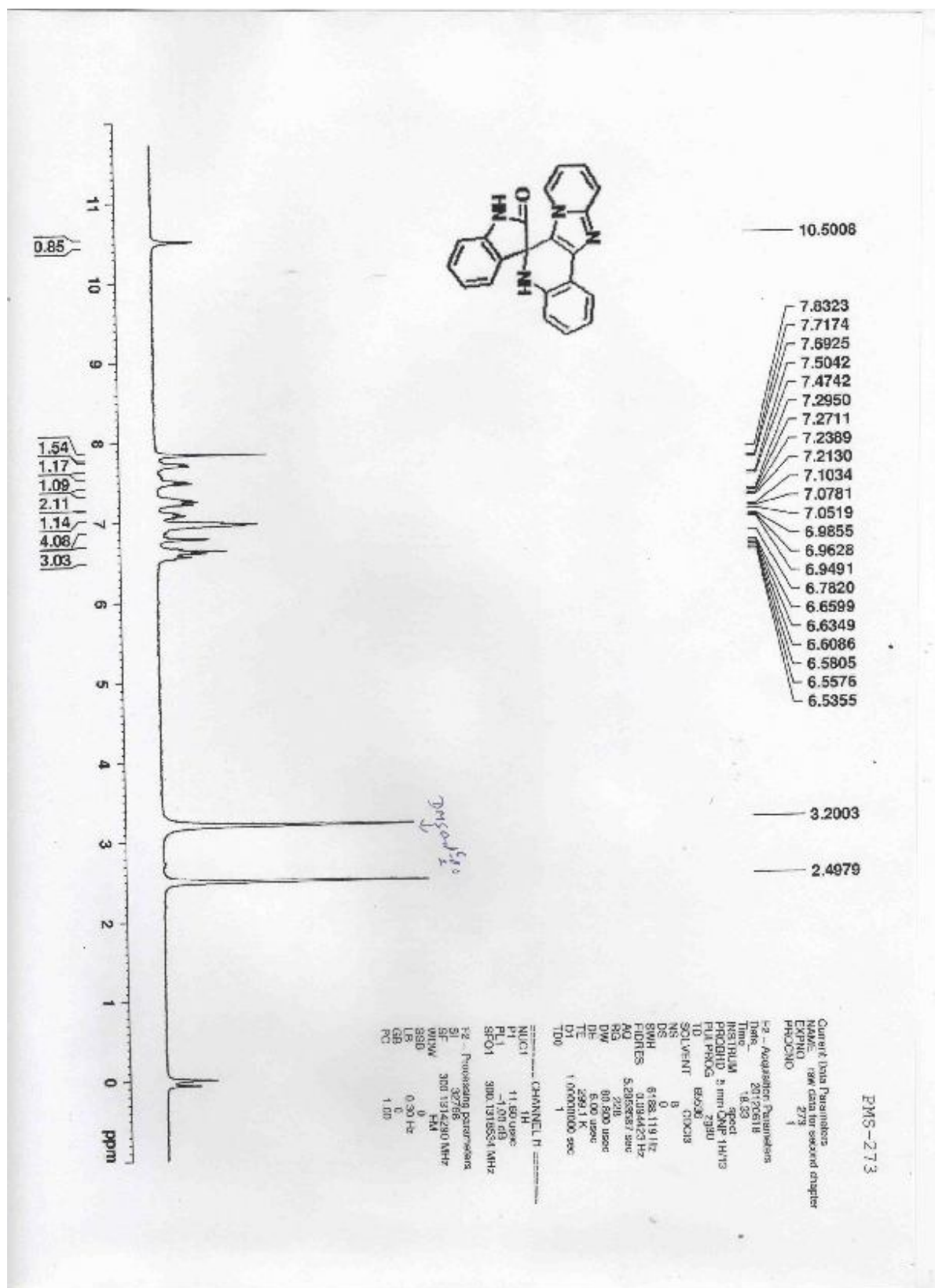
Current Data Parameters
NAME: Jm28-2012-CPXZ
EXPNO: 630
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20121227
Time: 12.42
INSTRUM: spect
PROBHD: 5 mm Dual 13C/
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 122
DS: 4
SWH: 11980.407 Hz
FIDRES: 0.16295 Hz
AQ: 2.732911 sec
RG: 808
RG: 41.700 usec
DE: 6.00 usec
TE: 300.2 K
D1: 2.00000000 sec
DELTA: 1.00000000 sec
DELTA: 0.00000000 sec
MCHRGST: 0.01500000 sec
MCMARK: 0.01500000 sec

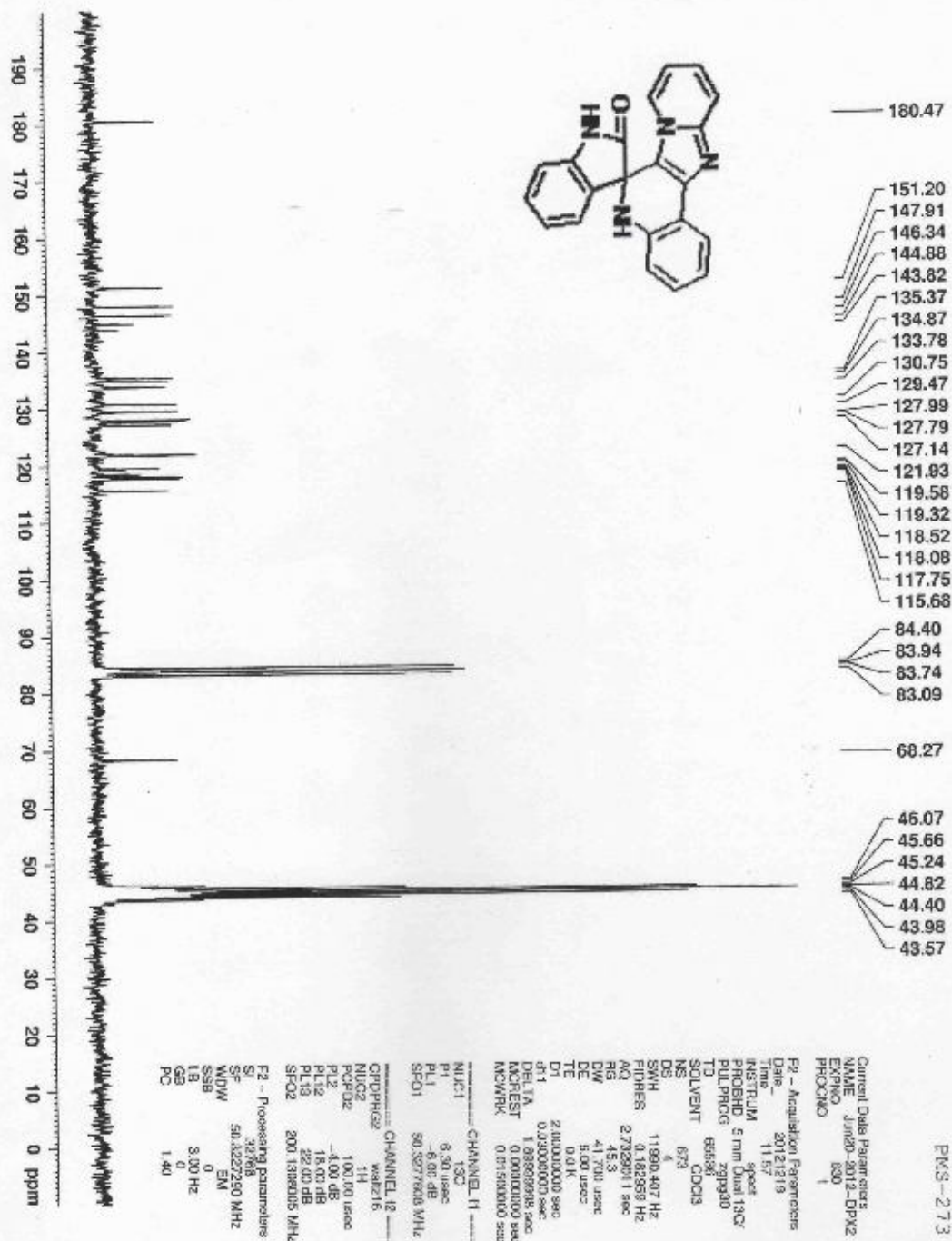
===== CHANNEL f1 =====
NUC1: 13C
P1: 6.30 usec
PL1: 6.00 dB
SFO1: 50.327000 MHz

===== CHANNEL f2 =====
CPDPRG2: WALT16
PULPROG: zgpg30
NUC2: 1H
P2: 100.00 usec
PL2: -4.00 dB
PL12: 18.00 dB
PL13: 22.00 dB
SFO2: 200.136000 MHz

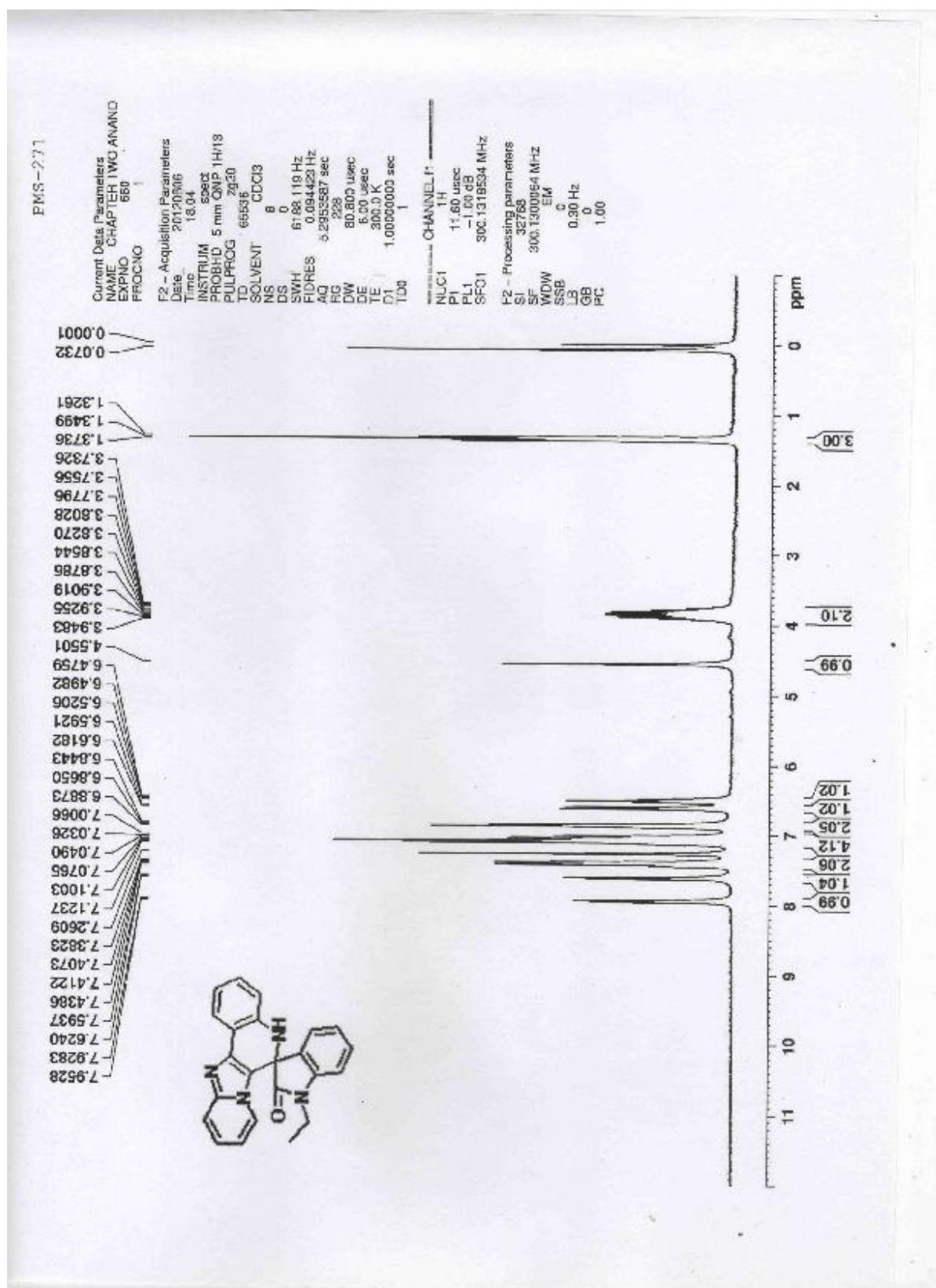
F2 - Processing parameters
SI: 32768
SF: 50.327290 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.60

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



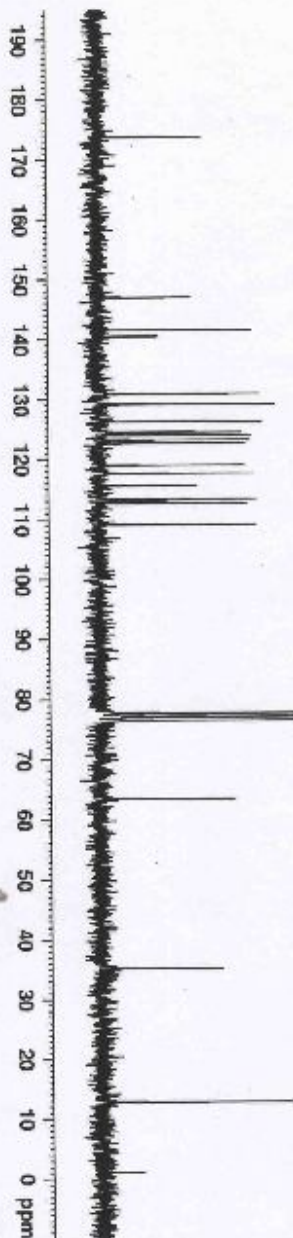
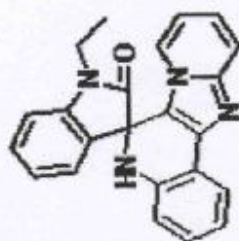


6-Spiro-indol-2(N-ethyl)-one-5,6-dihydro-5,6b,11-triaza-benzo[a]fluorine (13b):



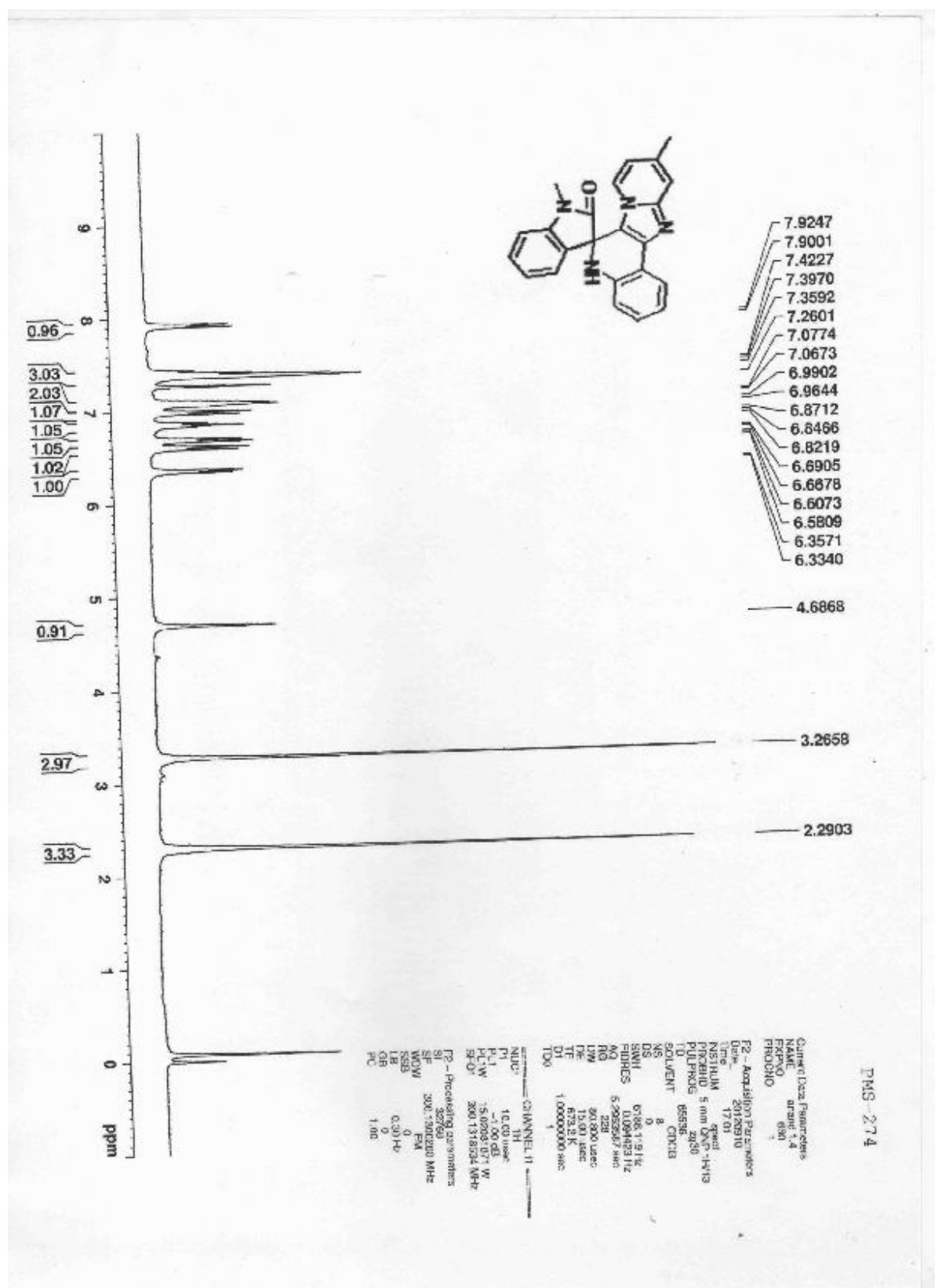
PMS-271

- 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



Current Data Parameters
NAME Jun-1-2012 07:42
EXPNO 640
PROCNO 1
F2 - Acquisition Parameters
Date_ 2012-07-13
Time 14:53
INSTRUM spect
PROBHD 5mm Duet 100
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 11890.437 Hz
FIDRES 0.182558 Hz
AQ 2.732801 sec
RG 90.5
DM 41.760 usec
TE 300.2 K
DELT 0.00000000 sec
DELTA 0.00000000 sec
ACQRES 1.00000000 sec
MCPRIN 0.01500000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 8.30 usec
PL1 -5.00 dB
SFO 50.257608 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 1.00 usec
PL2 0.00 dB
PL12 0.00 dB
PL13 20.00 dB
SFO2 200.136005 MHz
F2 - Processing parameters
SI 32768
SF 50.327290 MHz
WDW EN
SSB 0
GB 0
PC 1.50

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



Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 650
PROCNO 1

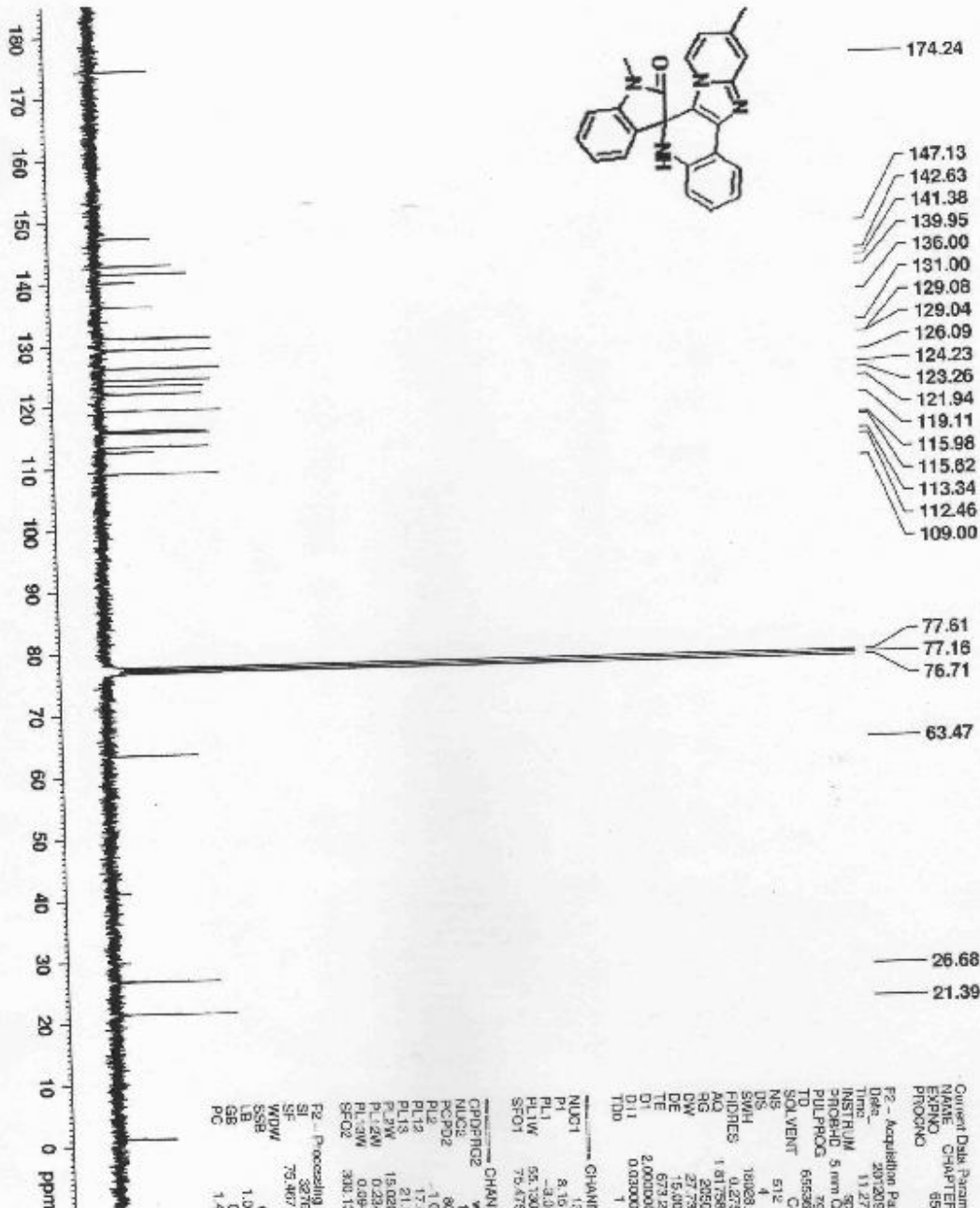
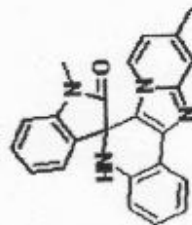
F2 - Acquisition Parameters

Date_ 20020913
Time 11:27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.845 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
TDB 1

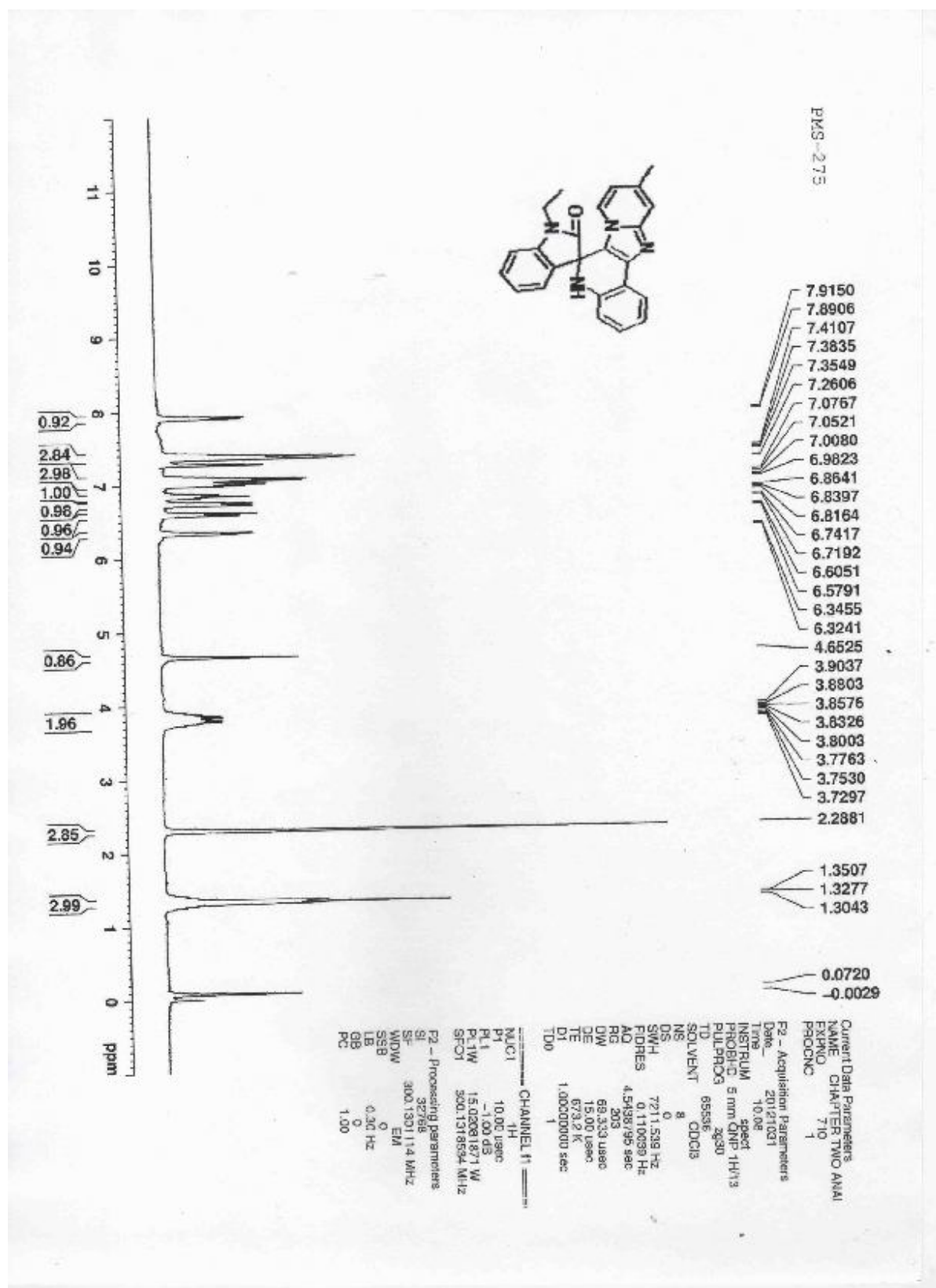
CHANNEL 1
NUC1 13C
P1 8.15 usec
PL1 -3.00 dB
PL1W 55.13056515 W
SFO1 75.4782503 MHz

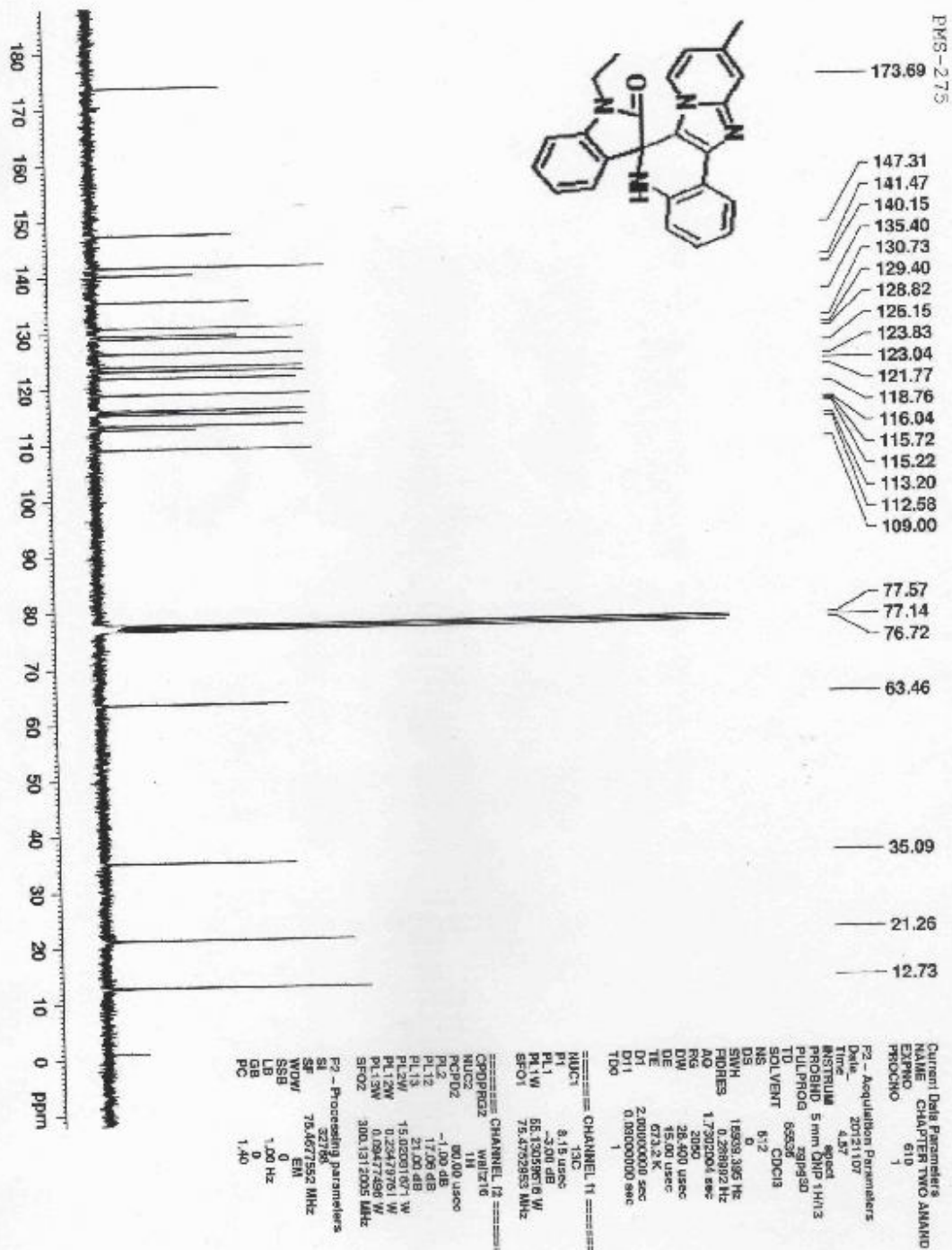
CHANNEL 2
CPDPRG2 MZET16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL2W 17.00 dB
PL13 21.00 dB
P13W 15.02501871 W
P13W 0.23473761 W
P13W 0.09477456 W
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4671437 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

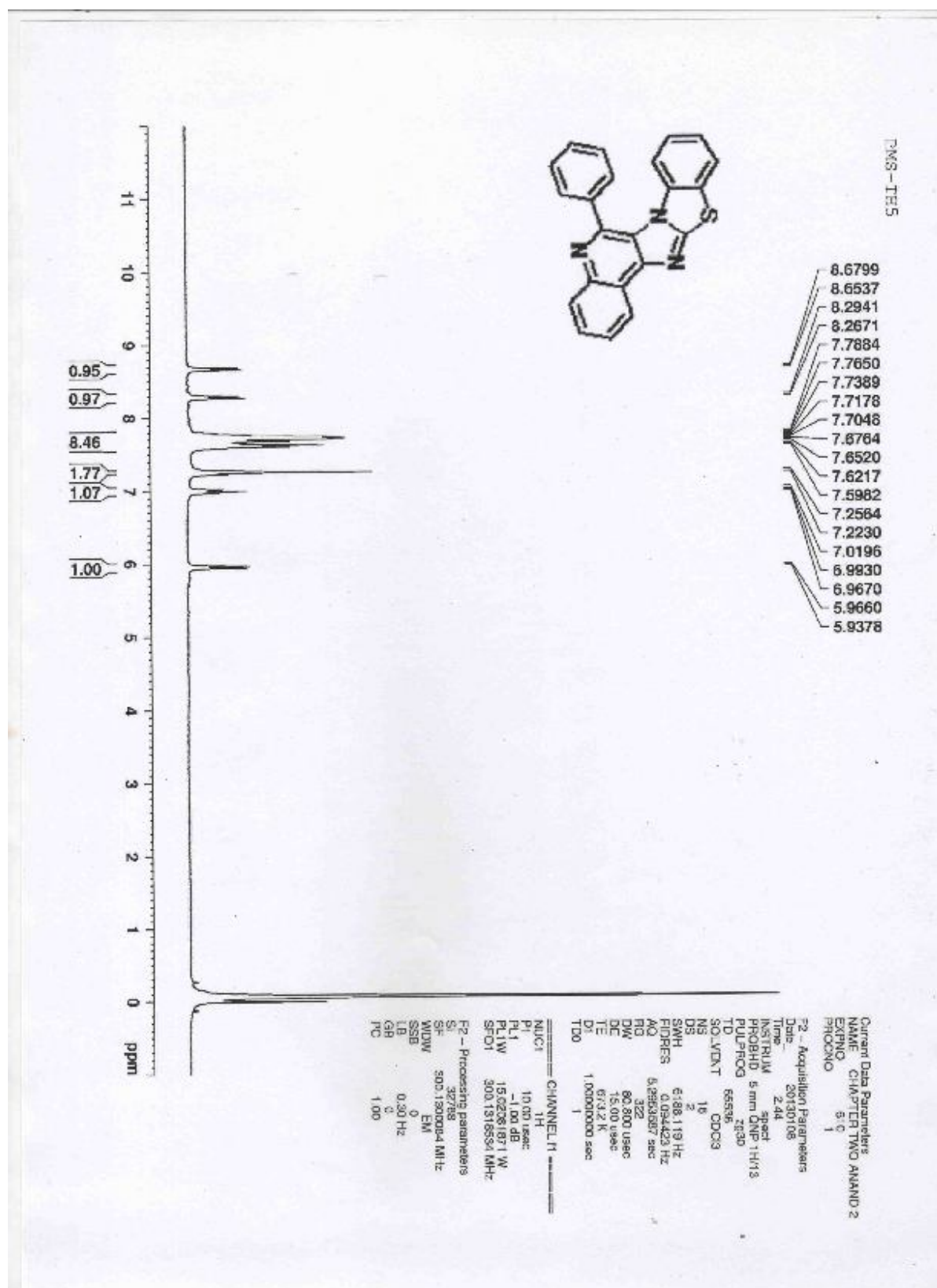


6-Spiro-indol-2(N-ethyl)-one-5,6-dihydro-9-methyl-5,6b,11-triaza-benzo[a]fluorine (13d):





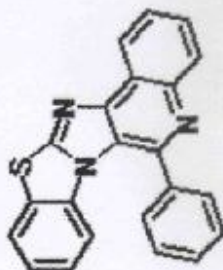
6-(Phenyl)-(11-S)-5,6b,12-triaza-benzo[a]fluorine (21a):



PMS-TR5

150.04
150.85
146.91
144.45
140.84
133.46
129.82
129.76
129.58
129.34
129.21
128.66
126.93
126.34
124.93
124.05
122.28
121.07
116.53

77.64
77.22
76.80



Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 65
PROCNO 1

F2 - Acquisition Parameters

Date_ 20130110
Time 0.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
P1/PRG3 75130
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18428.846 Hz
FIDRES 0.275028 Hz
AQ 1.8175916 sec
RG 2060
DW 27.733 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

CHANNEL F1

NUC1 13C
P1 8.11 usec
PL1 -2.00 dB
PL1W 55.14028613 W
SFO1 75.4753053 MHz

CHANNEL F2

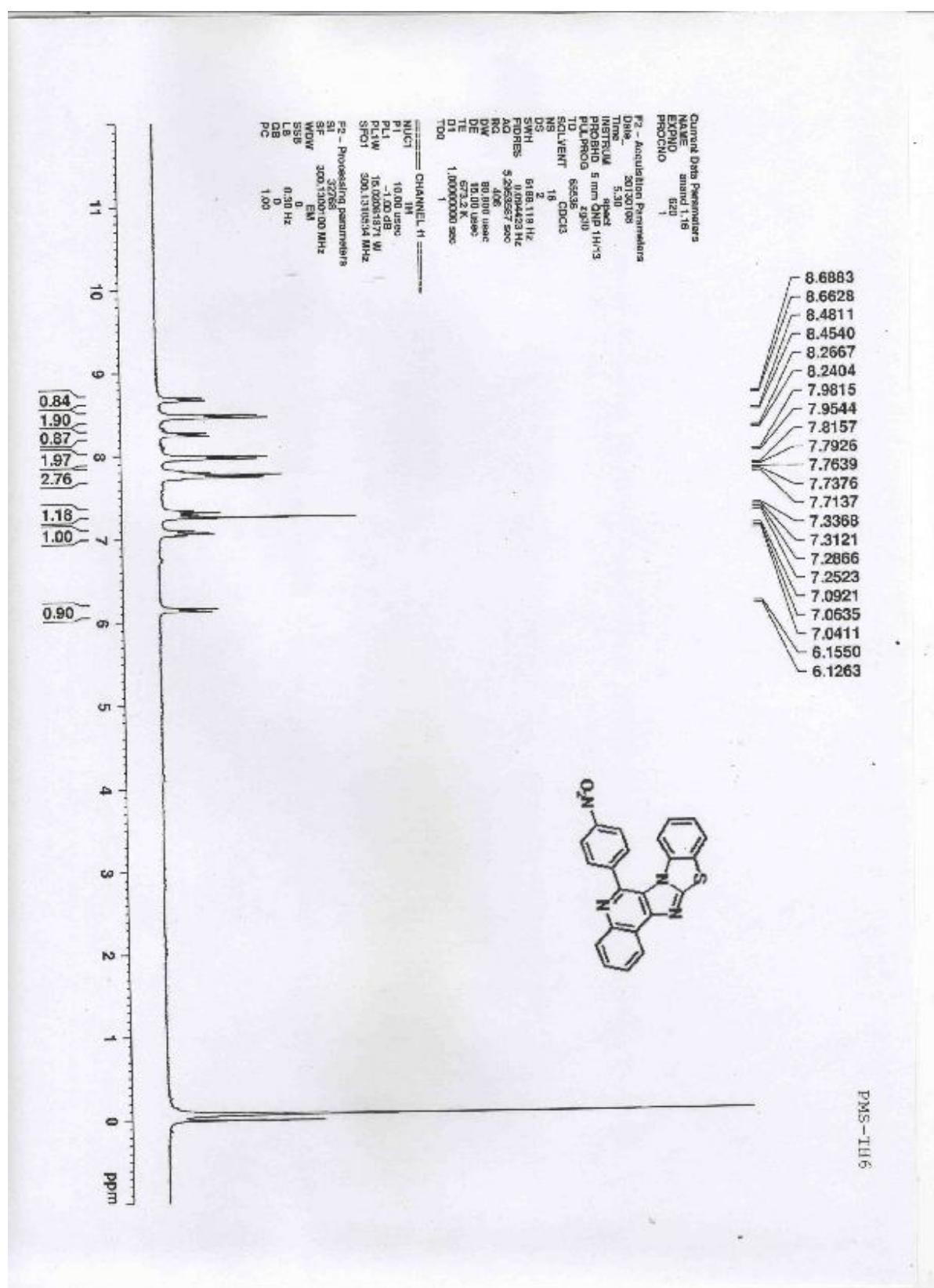
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 15.02067871 W
PL12W 0.23475761 W
PL13W 0.09477496 W
SFO2 300.1313005 MHz

F2 - Processing parameters

SF 300.1313005 MHz
WDW 2048
SSB 0
GB 0
PC 1.40

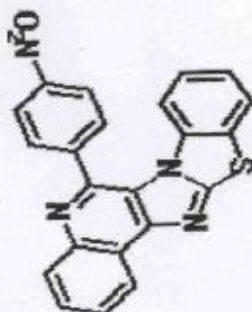
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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



PMS-TH6

158.57
151.47
148.88
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

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

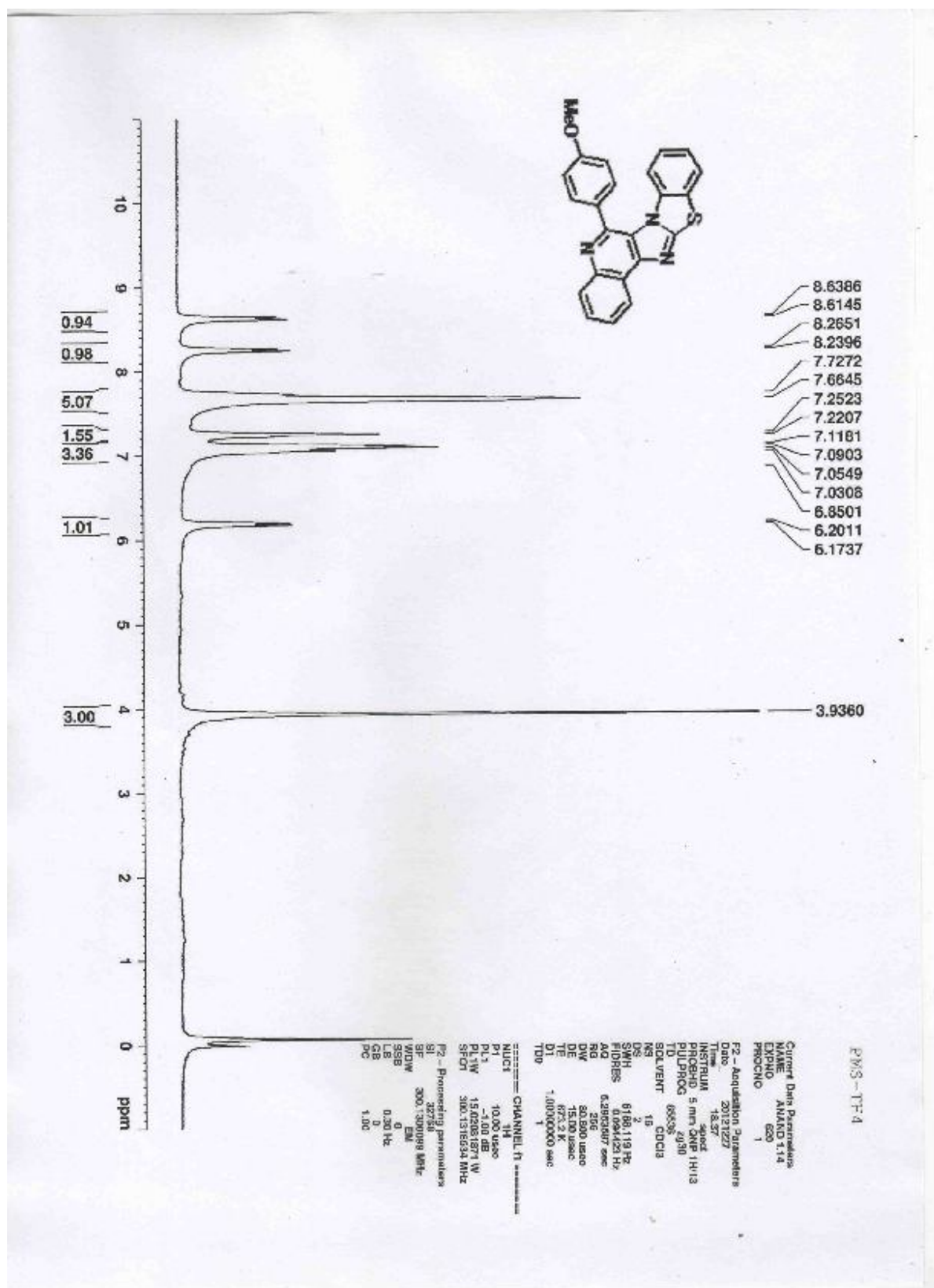
Current Data Parameters
NAME CHAPTER TWO ANAND
EXPNO 819
PROCNO 1
F2 Acquisition Parameters
Date_ 20181116
Time 17:13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18023.046 Hz
FIDRES 0.275088 Hz
AQ 1.8175816 sec
RG 2050
DE 27.734 L/sec
DW 15.00 L/sec
TE 300.2 K
D1 2.0000000 sec
D11 0.2500000 sec
TD0 1

CHANNEL 1
NUC1 13C
P1 8.15 L/sec
PL1 -3.00 dB
PL1W 55.1300000 W
SFO1 75.475903 MHz

CHANNEL 2
CPCPRG2 waltz16
NUC2 1H
PCPD2 80.00 L/sec
PL2 -1.00 dB
PL12 17.06 dB
PL13 21.00 dB
PL2W 16.0200000 W
PL12W 0.2347749 W
PL13W 0.0347749 W
SFO2 300.1372005 MHz

F2 - Processing parameters
SI 32768
SF 754.677383 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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



EMS-III4

Current Date: 10/10/80
NAME: LAYNO
EXPNO: 100
PROCNO: 100

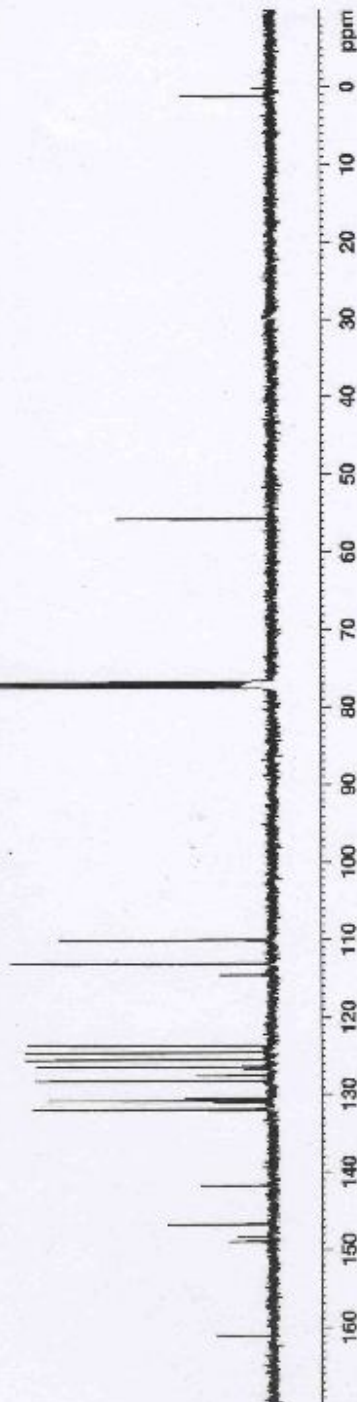
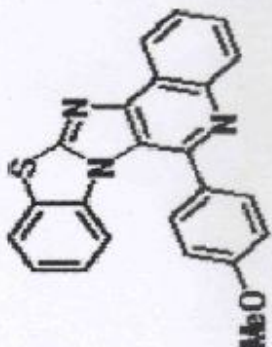
FS - Acquire only
Time: 15.00
Date: 10/10/80
INSTRUM: PULPROG
PULPROG: 5 mm PABIC BR
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 4
SWH: 24022.461 Hz
FIDRES: 0.388746 Hz
AQ: 1.353 900 sec
RG: 201.40
INW: 20.000 kHz
DE: 5.50 kHz
TE: 300.0 K
D1: 2.00000000 sec
D11: 0.00000000 sec
D12: 0.00000000 sec

CHANNEL F1
SF01: 100.6204683 MHz
NUC1: 13C
P1: 0.20 sec
F2 - Processing parameters
SI: 32768
SF: 100.6204683 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

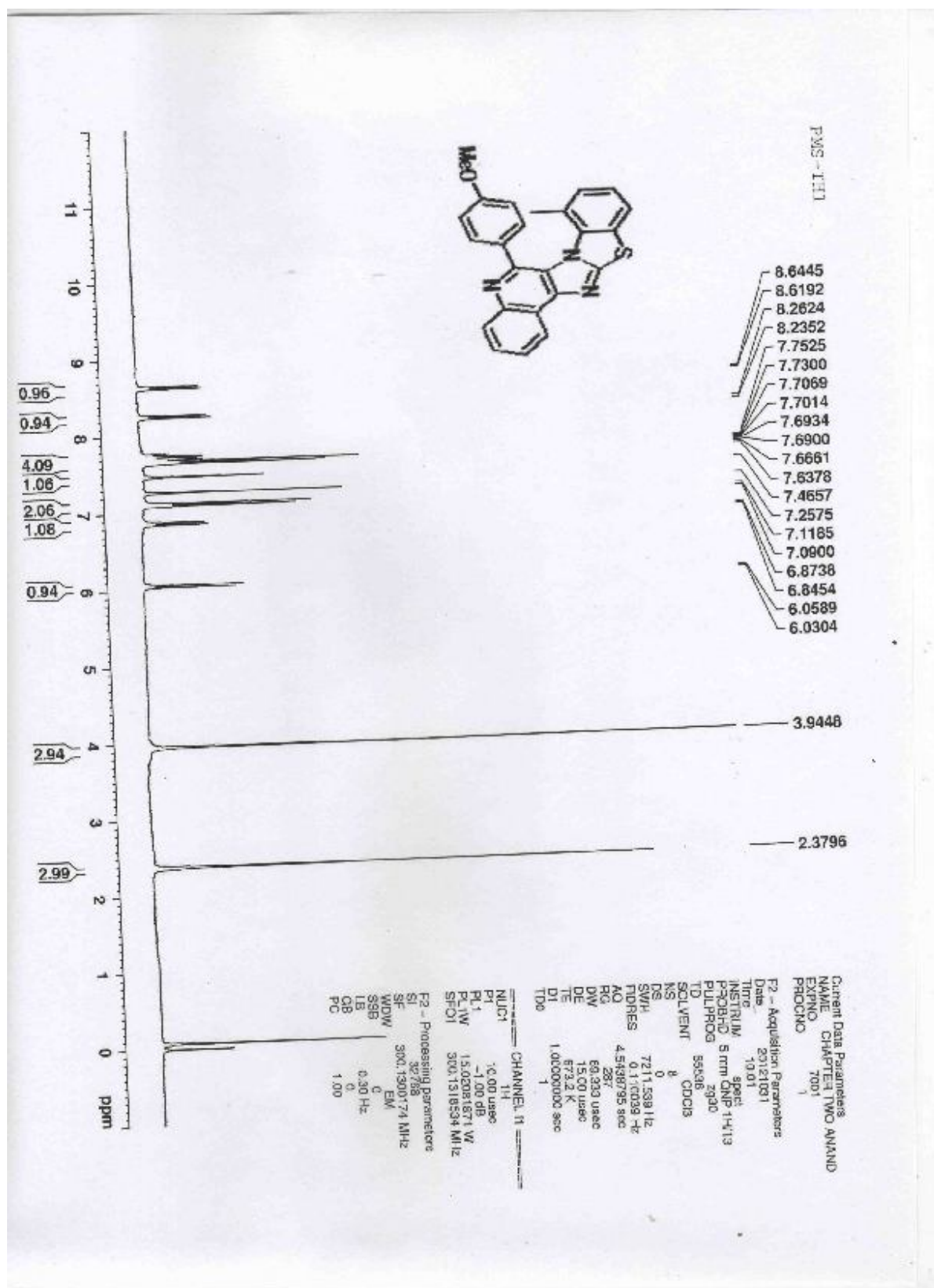
55.85

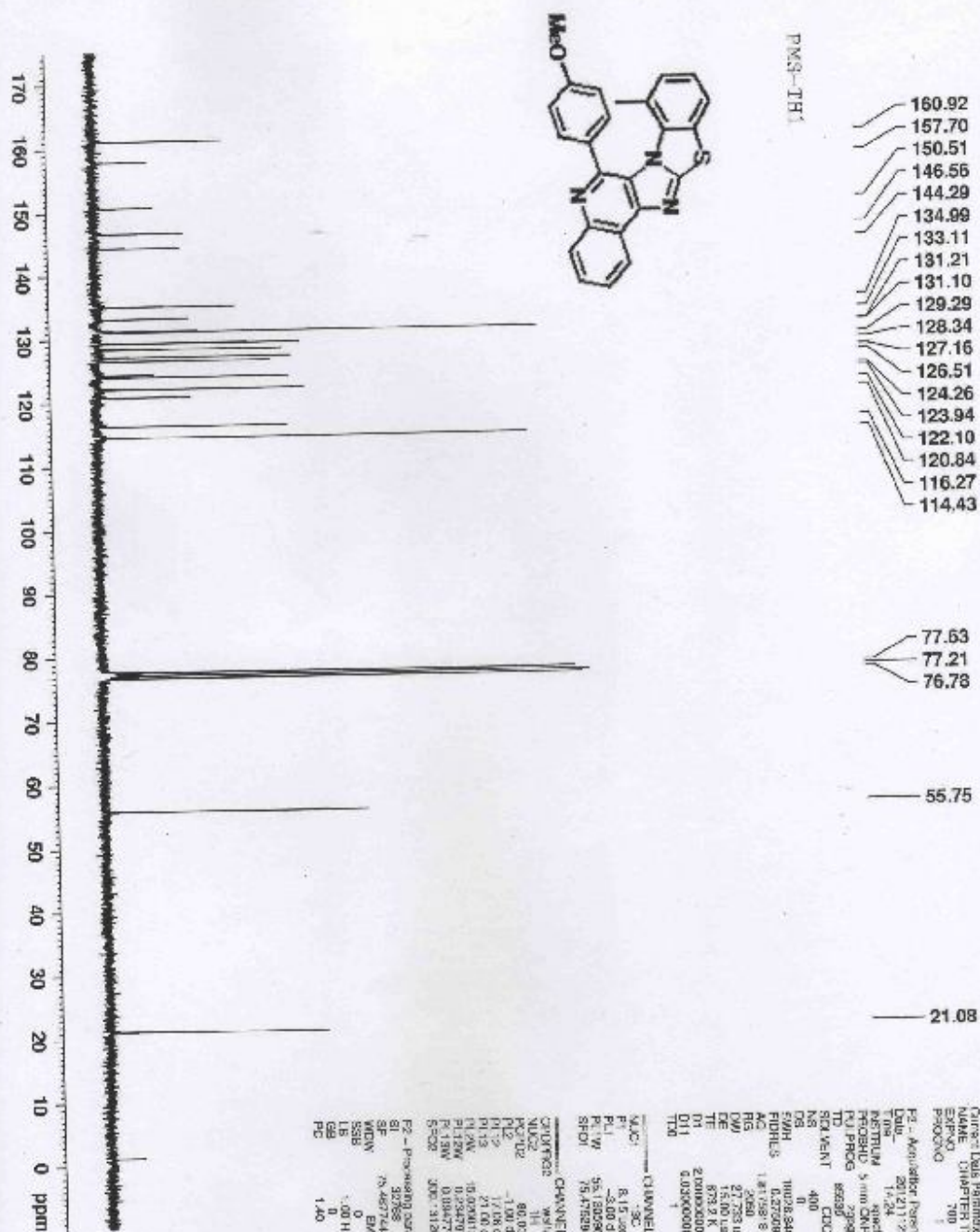
76.86
77.17
77.54

110.10
113.13
114.59
123.71
124.56
125.50
126.49
127.57
128.29
130.55
130.84
131.15
131.96
132.07
141.79
146.72
148.33
148.90
161.04

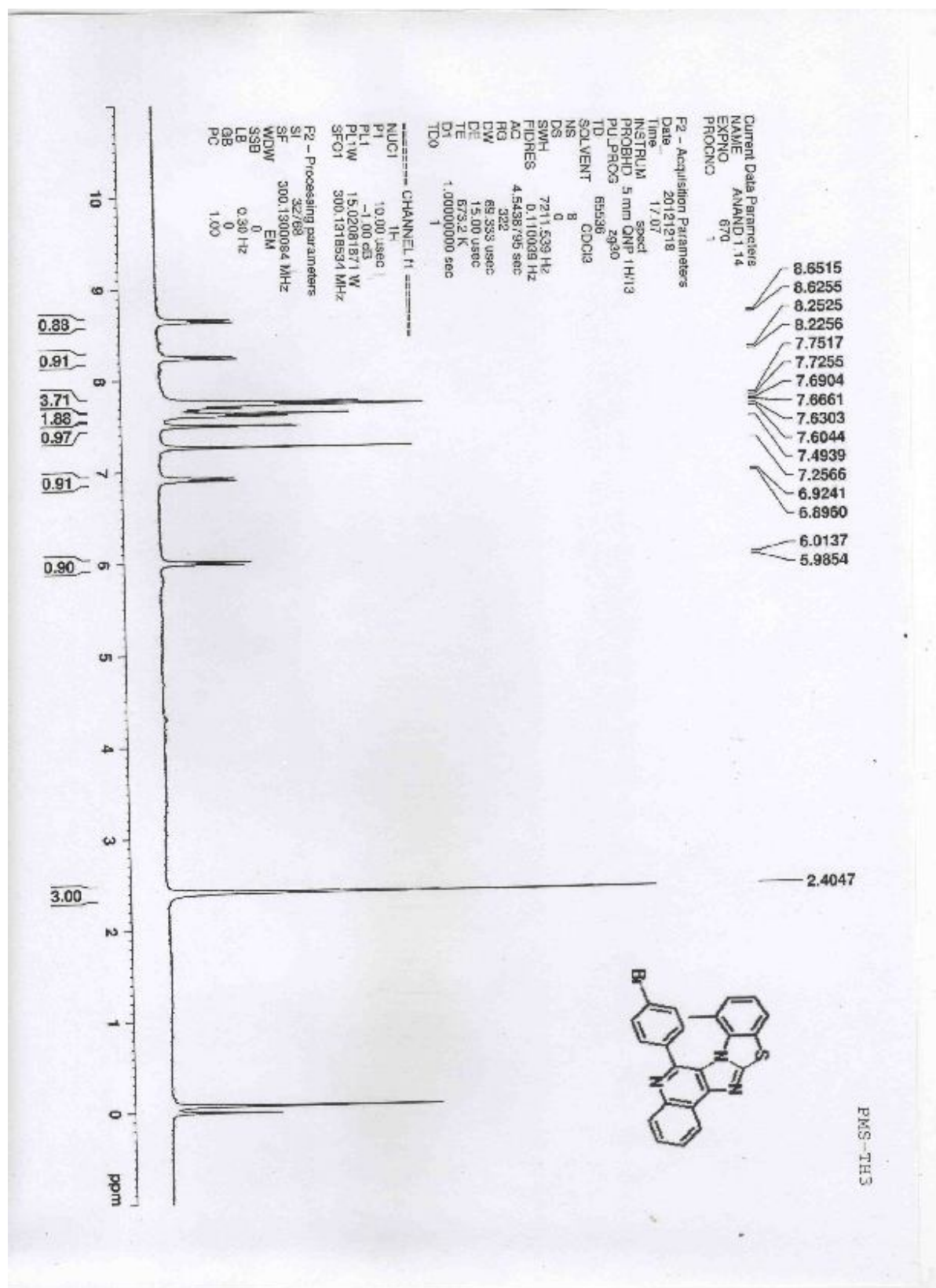


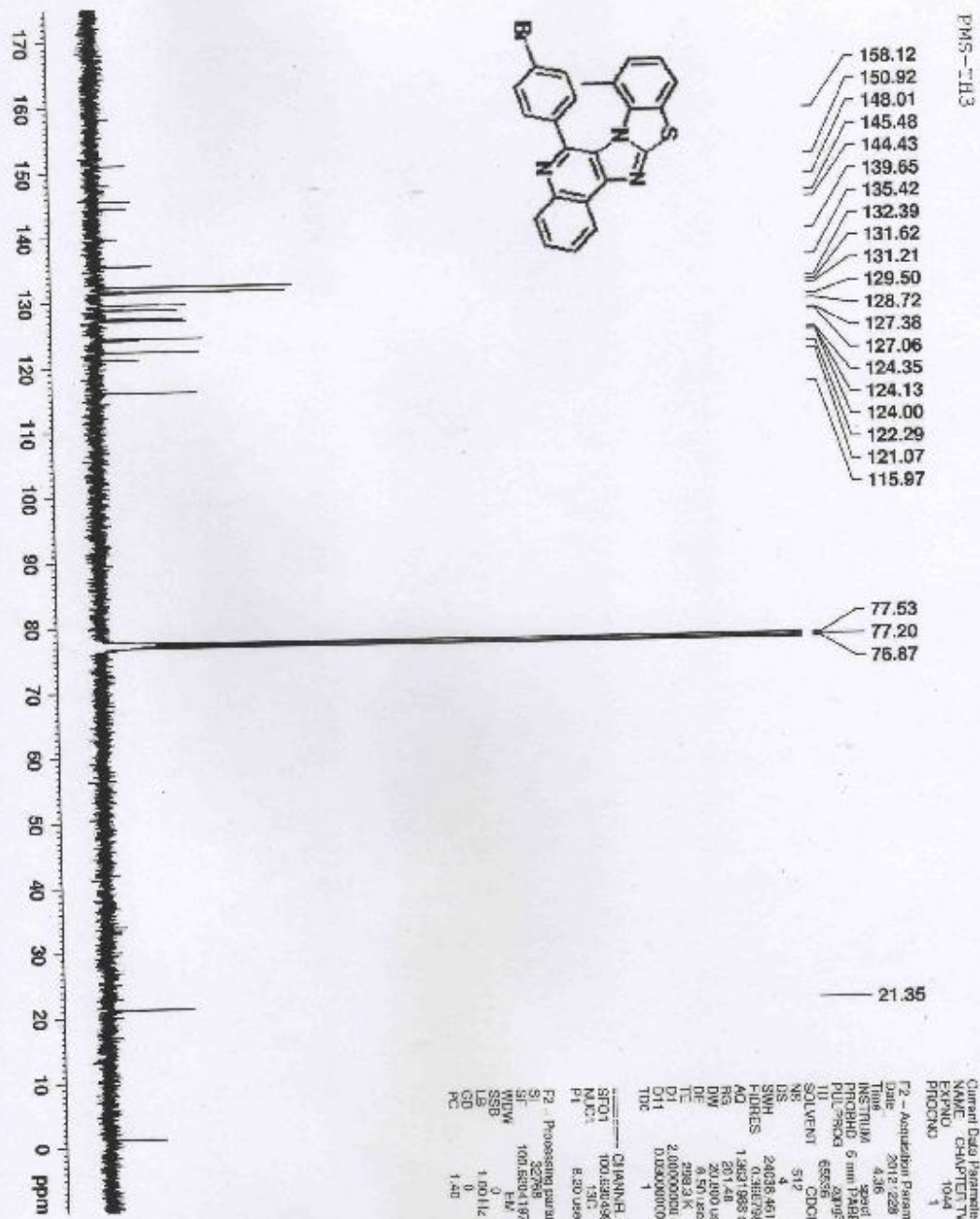
6-(4-Methoxy-Phenyl)-7-methyl-(11-S)-5,6b,12-triaza-benzo[a]fluorine (21d):





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





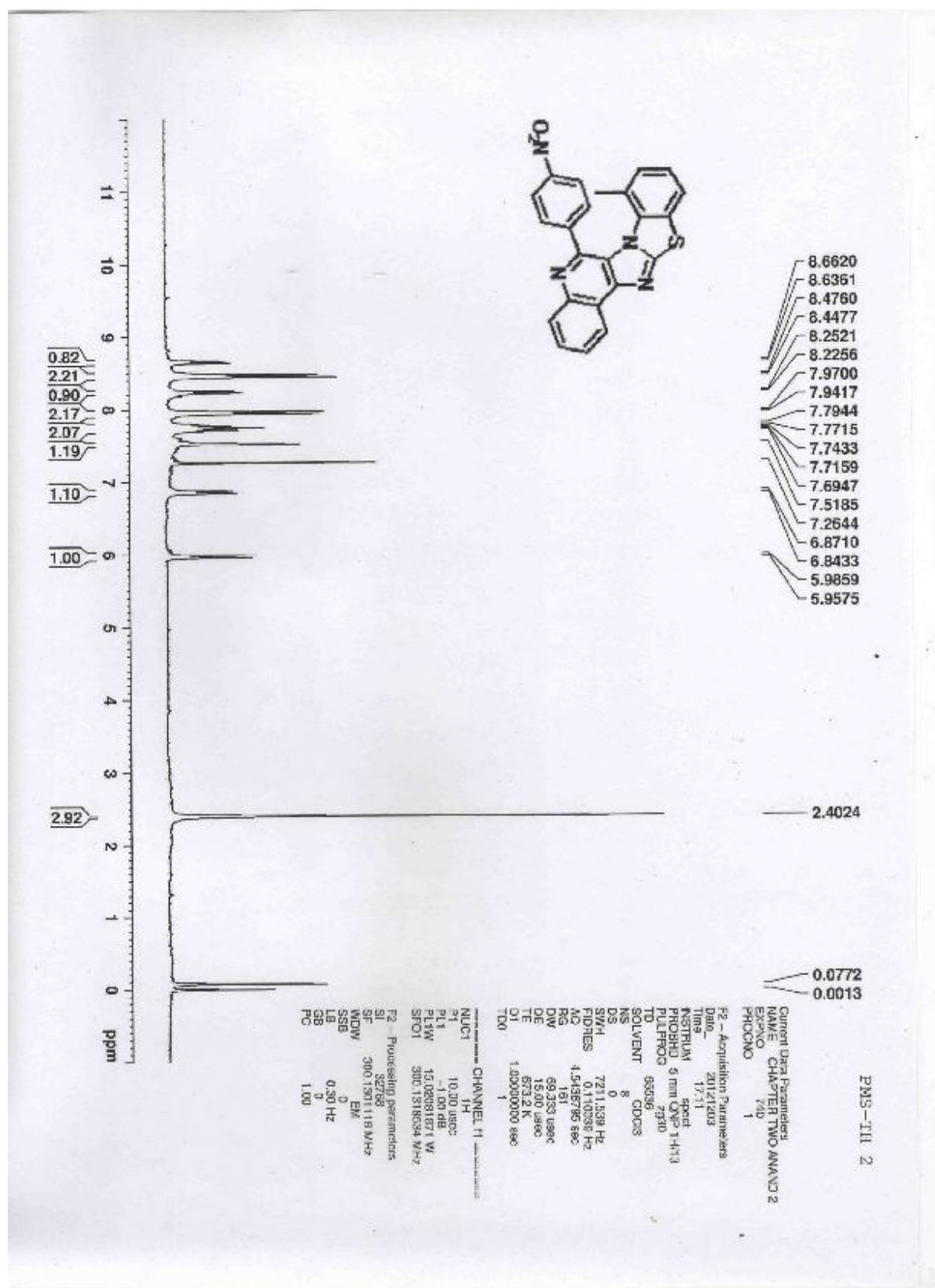
Current Data Parameters
NAME: CHAPLET TWO ANAND
EXPNO: 1044
PROCNO: 1

F2 - Acquisition Parameters
Date_ 2012-228
Time 4.36
INSTRUM spect
PROBHD 5 mm TAIABO BBO
PULPROG zgpg30
TD 65536
SOLVENT dms
SOLVENT 512
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.367798 Hz
AQ 1.3631988 sec
RG 201.48
DW 20.000 usec
DE 9.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD 1

===== CHANNEL f1 =====
SFO1 100.620493 MHz
NUC1 13C
P1 8.30 usec

F2 - Processing parameters
SI 32768
SF 100.620497 MHz
WDW 0 Hz
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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



Current Data Parameters
NAME CHAPTER TWO ANAND 2
EXPNO 5559
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121210
Time 11.51

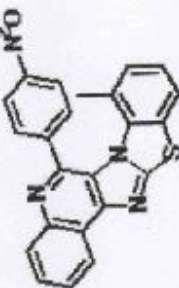
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3

NS 4
DS 1802H 148 Hz
SWH 0.275095 Hz
FIDRES 1.3175018 sec
AQ 2024
RG 27.735 usec
DE 15.00 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO

CHNCL 11
NUC1 13C
P1 8.15 usec
PL1 3.00 dB
PL1W 55.13000000 W
SFO1 75.470253 MHz

CHNCL 12
NUC2 1H
P2 80.00 usec
PL2 -1.00 dB
PL12 17.05 dB
PL13 21.00 dB
PL14 15.00 dB
PL15 0.23479761 W
PL16 0.09477498 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



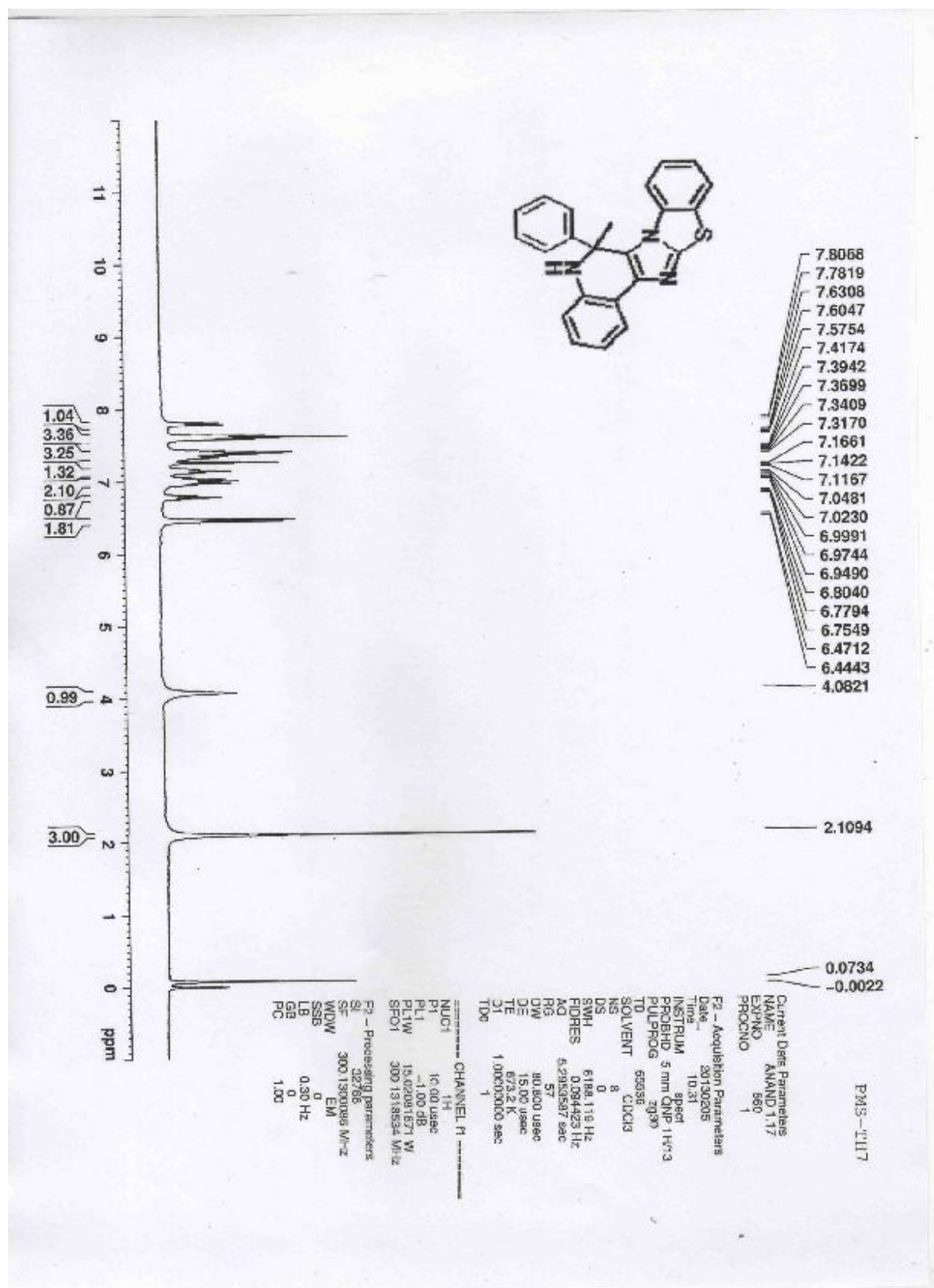
- 158.38
- 151.07
- 148.66
- 146.72
- 144.31
- 143.86
- 135.62
- 131.03
- 130.80
- 129.49
- 129.42
- 128.89
- 127.44
- 127.24
- 124.50
- 124.10
- 123.35
- 122.25
- 121.05
- 115.37

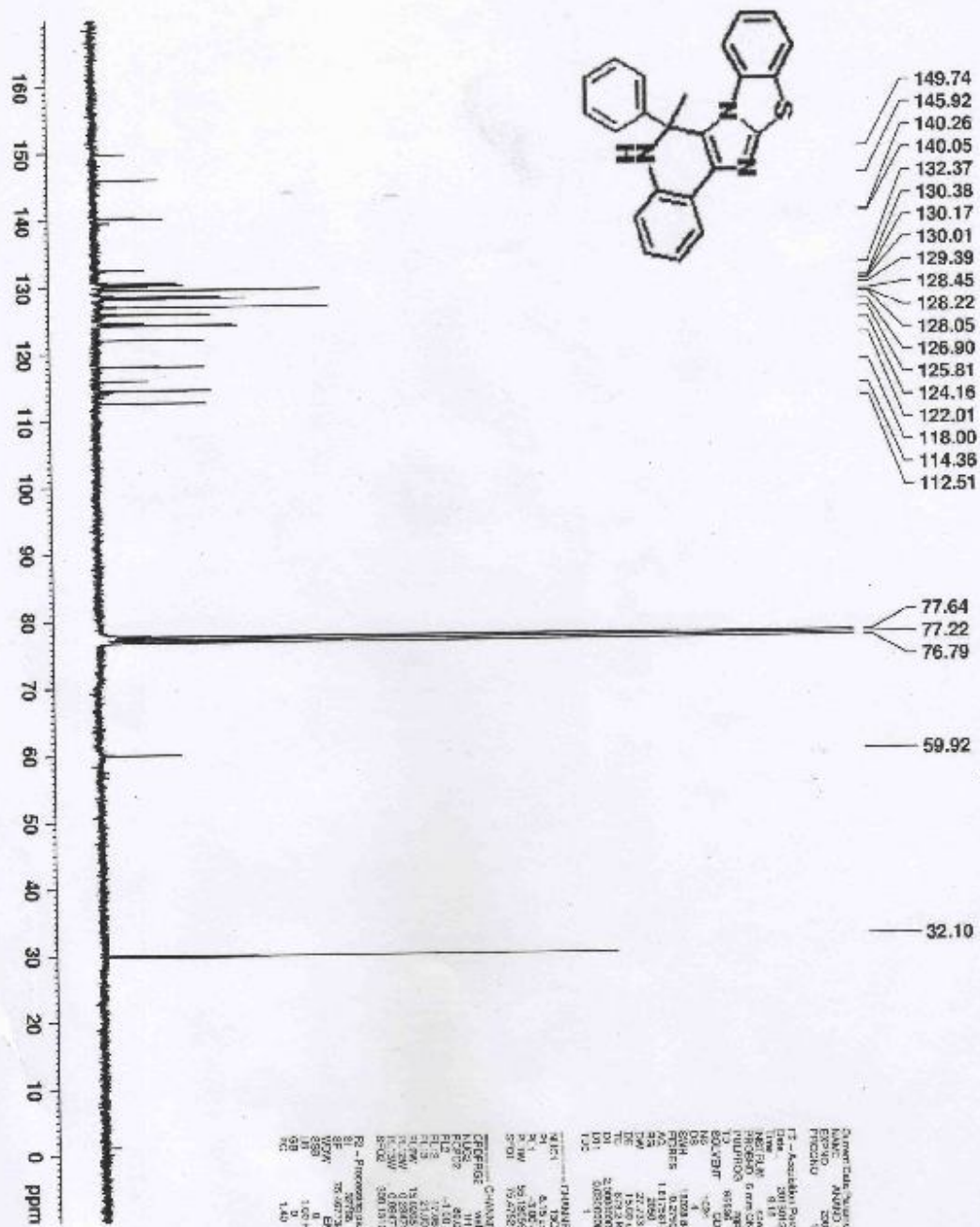
- 77.59
- 77.14
- 76.74

21.11

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):





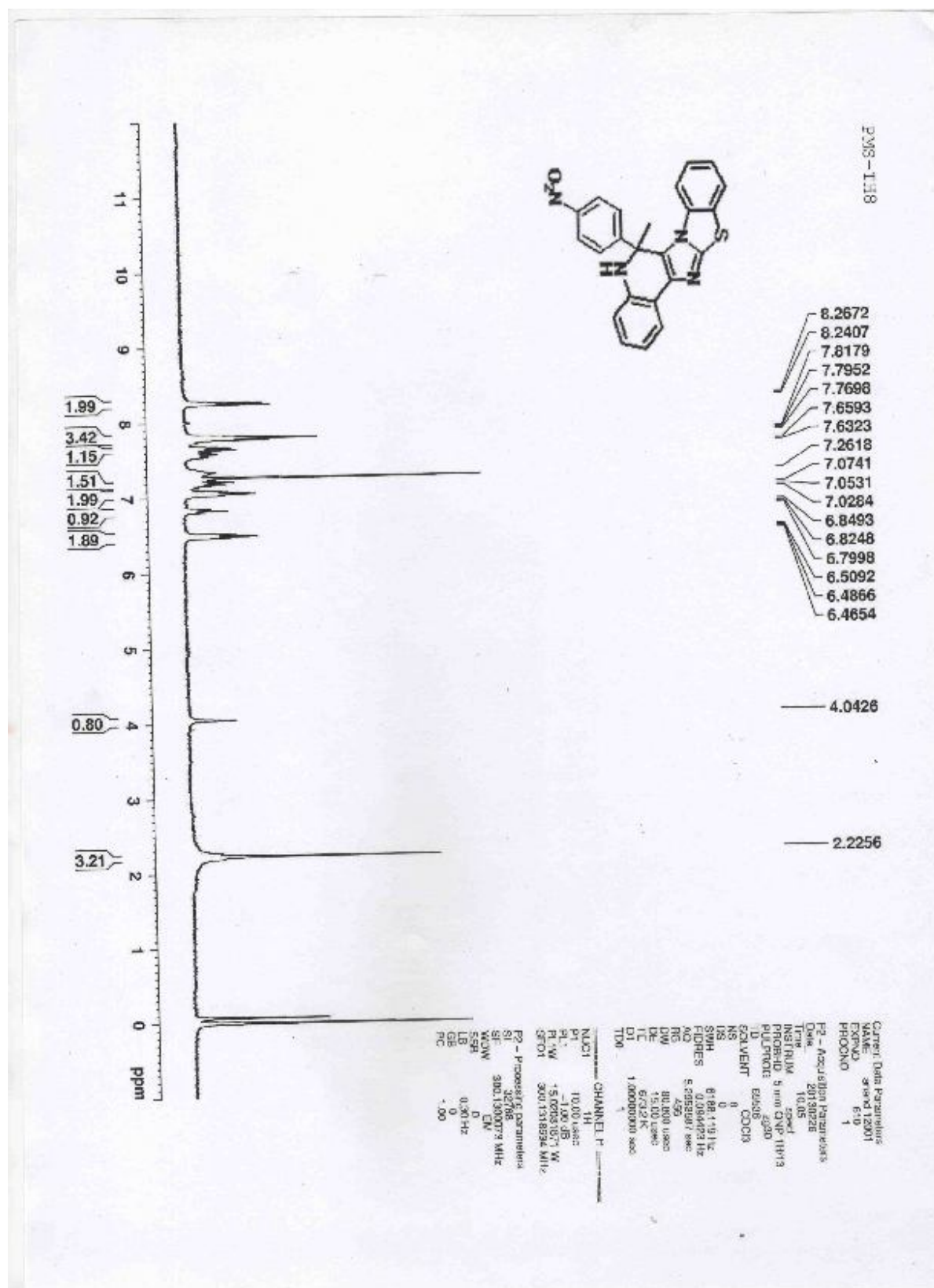
pmh-th7

Chemical structure: Cc1c2ccccc2[nH]1c3ccccc3n4c5ccccc5sc43

¹³C NMR spectrum (ppm) peaks (from top to bottom):

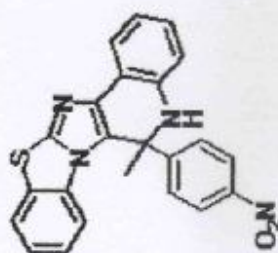
- 149.74
- 145.92
- 140.26
- 140.05
- 132.37
- 130.38
- 130.17
- 130.01
- 129.39
- 128.45
- 128.22
- 128.05
- 126.90
- 125.81
- 124.16
- 122.01
- 118.00
- 114.36
- 112.51
- 77.64
- 77.22
- 76.79
- 59.92
- 32.10

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



pmw-018

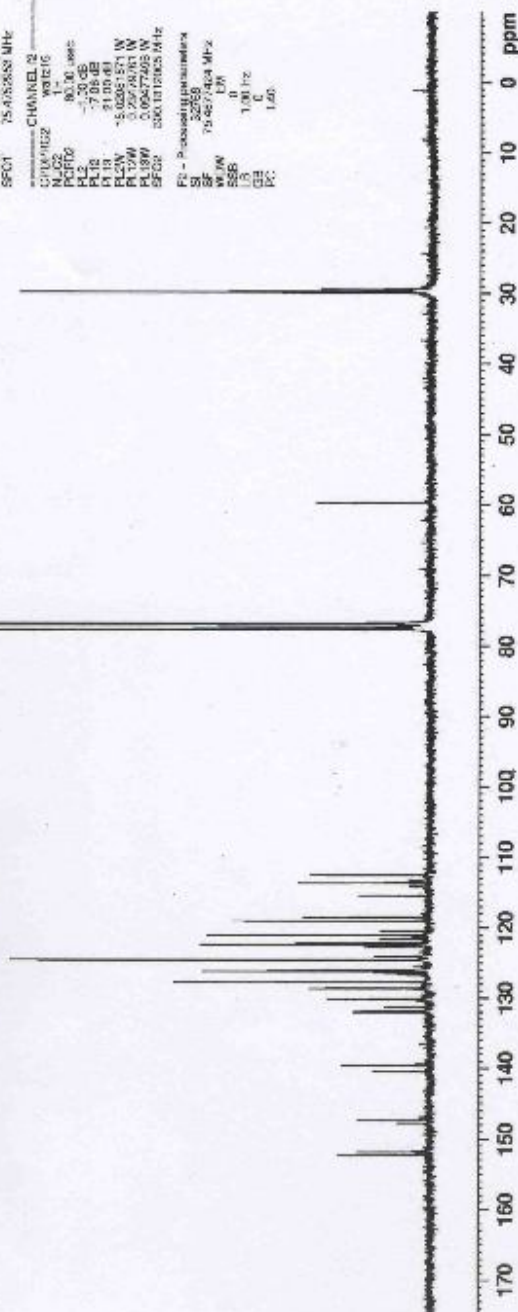
152.27
151.78
147.35
140.44
139.63
131.91
130.10
128.56
127.70
126.11
125.99
124.56
122.34
122.17
121.04
118.97
118.46
113.69
112.57



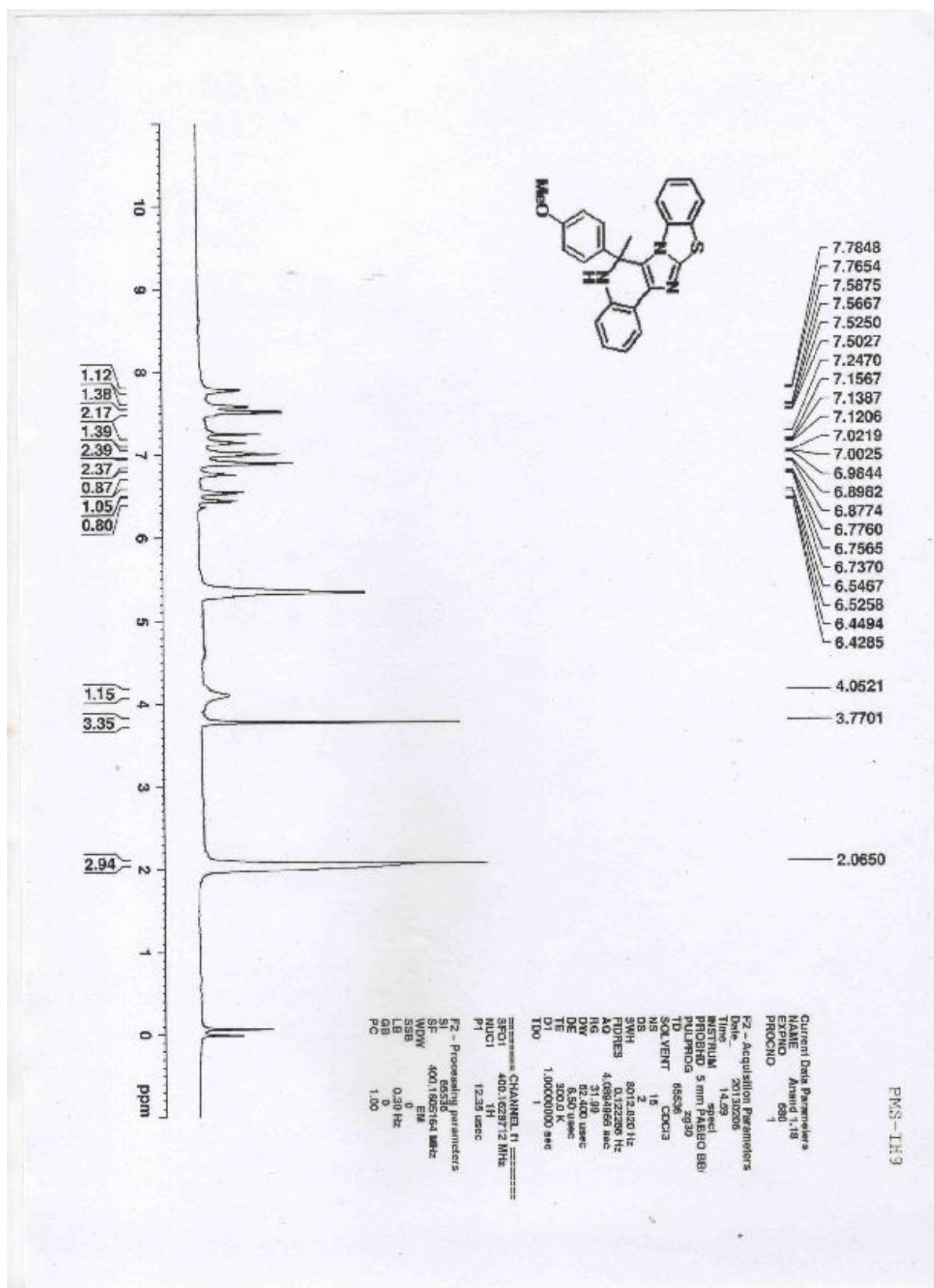
77.63
77.20
76.78
59.68
32.05

Current Data Parameters
NAME: PMW-018
EXPNO: 1
PROCNO: 1
P2 - Acquisition Parameters
Date_: 20130125
Time: 11:13
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 4
SWH: 18038.848 Hz
FIDRES: 0.270030 Hz
AQ: 1.87258 Hz sec
RG: 272.0
DQ: 2.000000 sec
UL: 13.00 sec
TE: 693.2 K
D1: 2.0000000 sec
D11: 0.0500000 sec
D12: 0.0500000 sec

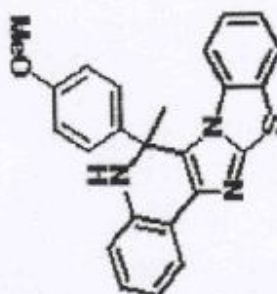
===== CHANNEL f1 =====
NUC1: 13C
P1: 8.12 sec
PL1: 0 dB
PL1W: 0.0000000 W
SFO1: 125.7611535 MHz
===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 0.0100000 sec
PL2: 0 dB
PL2W: 0.0000000 W
PL3: 0 dB
PL3W: 0.0000000 W
PL4: 0 dB
PL4W: 0.0000000 W
SFO2: 500.1311535 MHz
===== CHANNEL f3 =====
SI: 32768
SF: 75.4671174 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00



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



PMS-2H9



- 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
- 115.81
- 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: pcdet
EXPNO: 630
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20130712
Time: 11.56
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 512
DS: 0
SWH: 18029.846 Hz
FIDRES: 0.275698 Hz
AQ: 1.8175618 sec
RG: 2050
RW: 27.735 usec
DE: 19.04 usec
TE: 673.2 K
D1: 2.0000000 sec
D11: 0.05000000 sec
TD0:

CHANNEL F1
NUC1: 13C
P1: 8.15 usec
PL1: -3.00 dB
PL1W: 55.3056615 W
SFO1: 75.4752993 MHz

CHANNEL F2
NUC2: 1H
P2: 100.00 usec
PL2: -1.00 dB
PL2W: 17.06 dB
PL13: 21.00 dB
PL12W: 15.07091671 W
PL13W: 0.23473751 W
SFO2: 300.1312005 MHz

F2 - Processing parameters
SF: 75.4677396 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 $\text{DMSO-}d_6$ at δ 2.48-2.51 (ppm) were observed in respective ^1H -NMR spectrum and at δ 77.15-77.32 (ppm) for CDCl_3 and δ 39.45-39.55 (ppm) for $\text{DMSO-}d_6$ were observed in respective ^{13}C -NMR spectrum.