Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

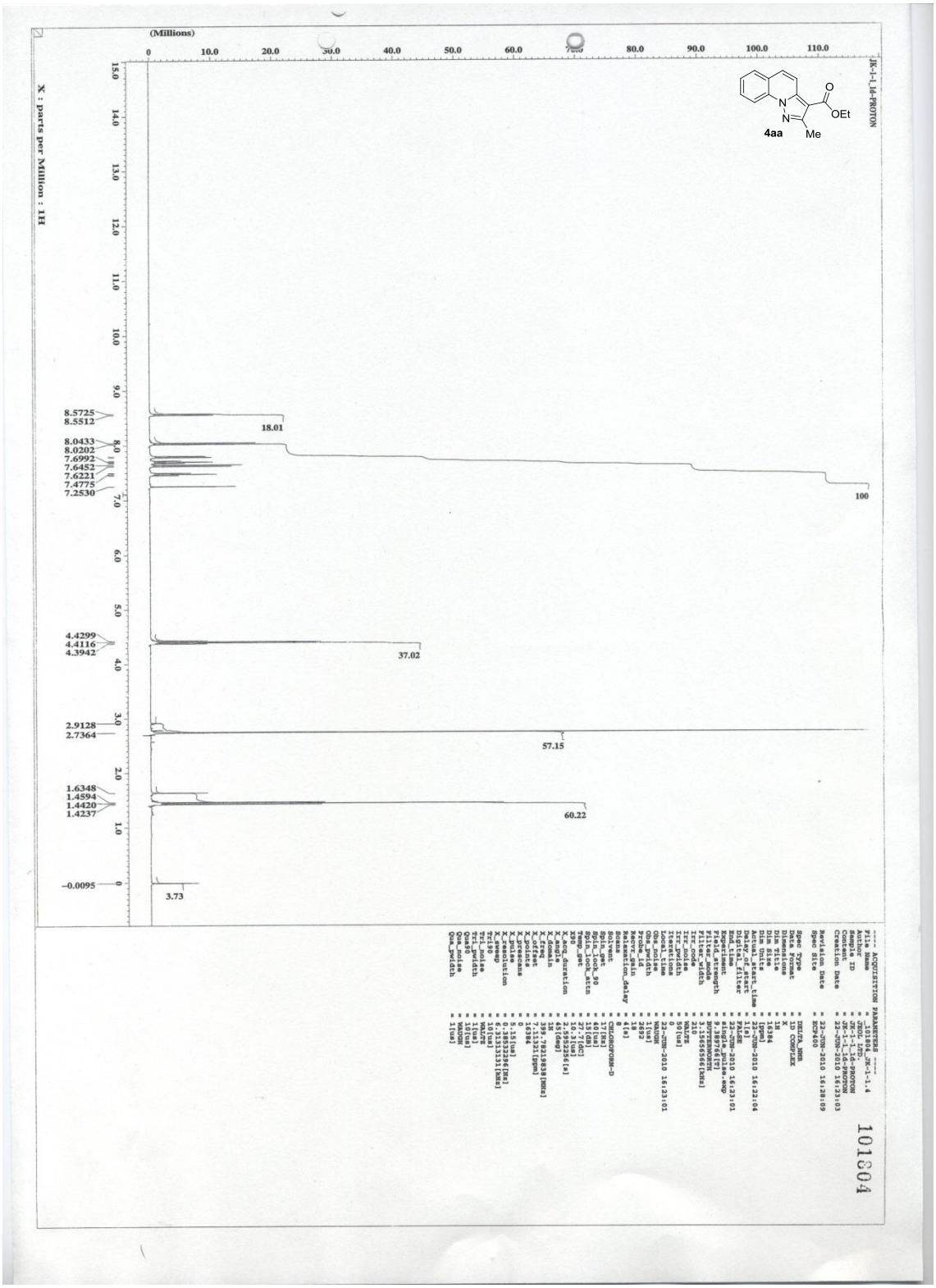
Supplementary Information

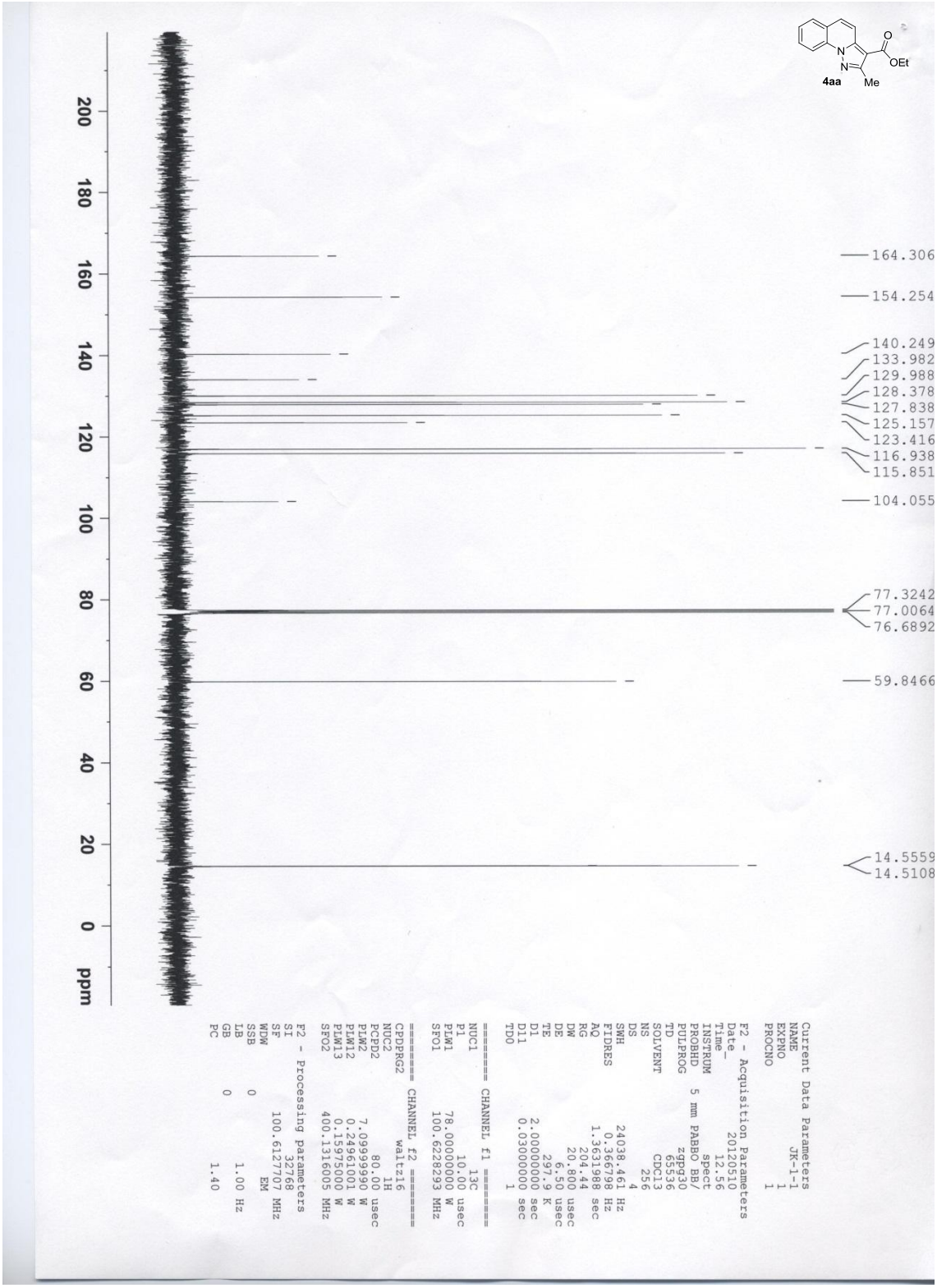
Development of a new cascade reaction for convergent synthesis of pyrazolo[1,5-*a*]quinoline derivatives under transition-metal-free conditions

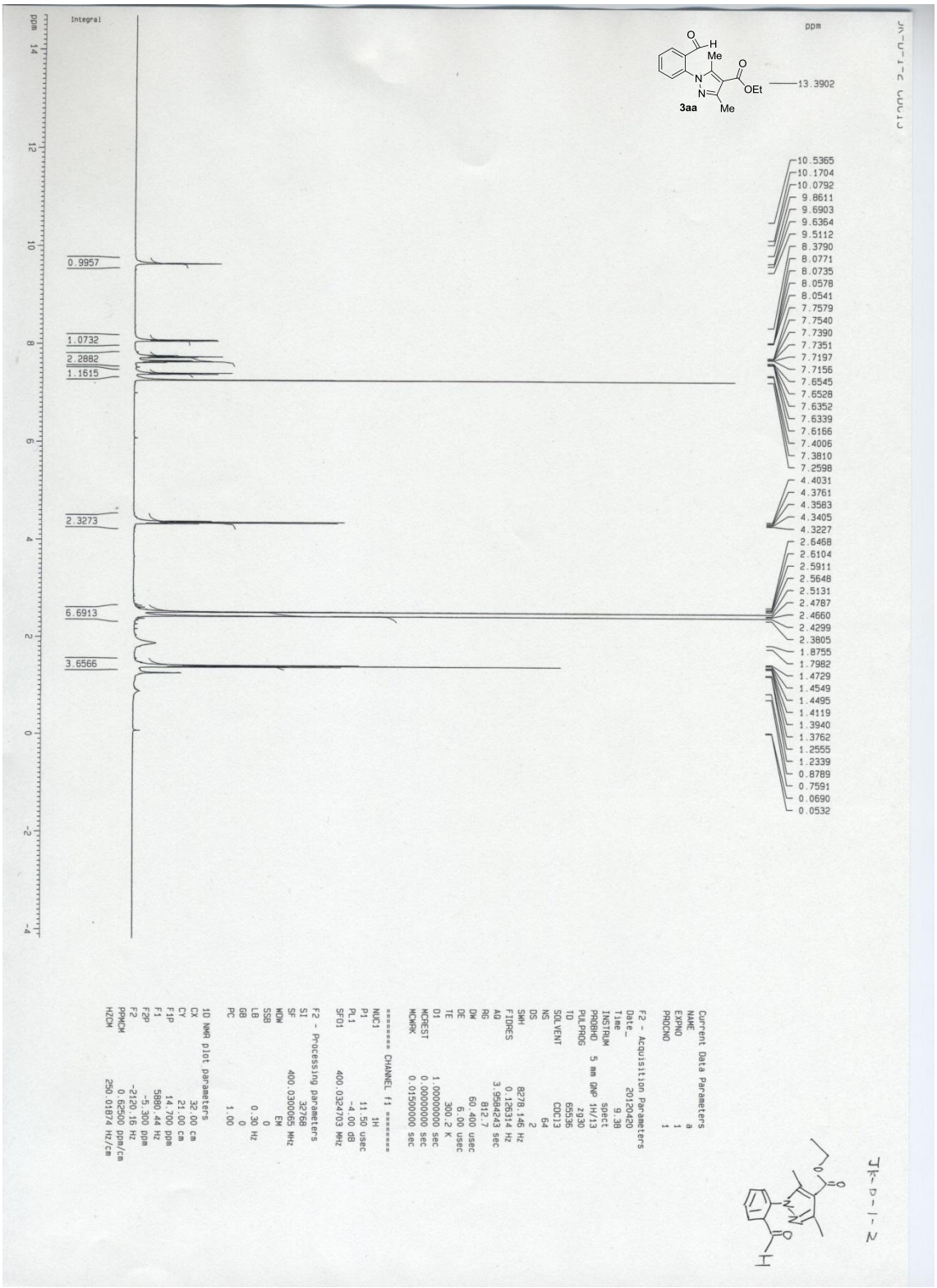
Jun-ya Kato, Hiroshi Aoyama, and Tsutomu Yokomatsu*

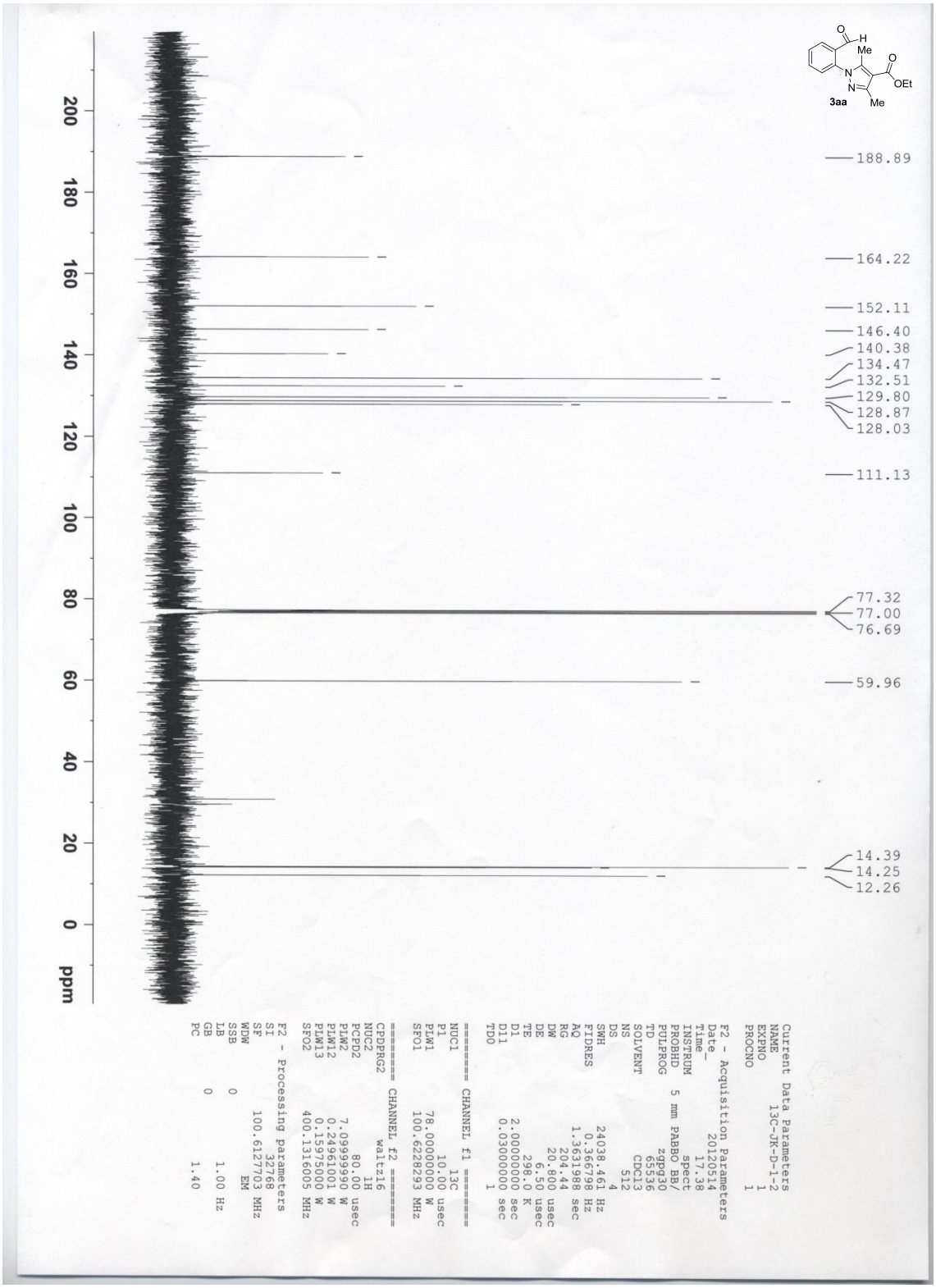
Tokyo University of Pharmacy and Life Sciences, Horinouchi, Hachioji, Tokyo 192-0392, Japan
E-mail: yokomatu@toyaku.ac.jp

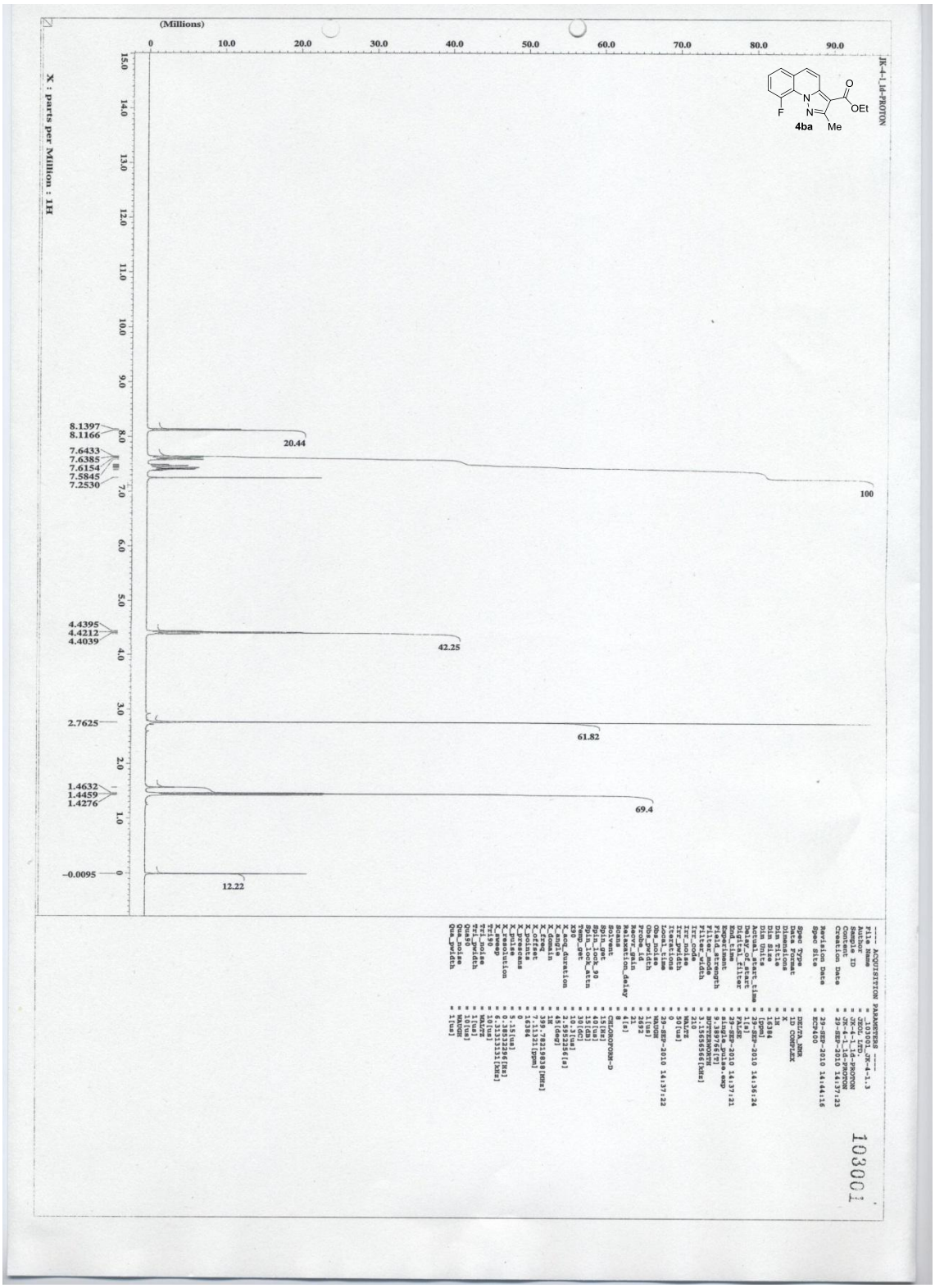
1. NMR spectrum

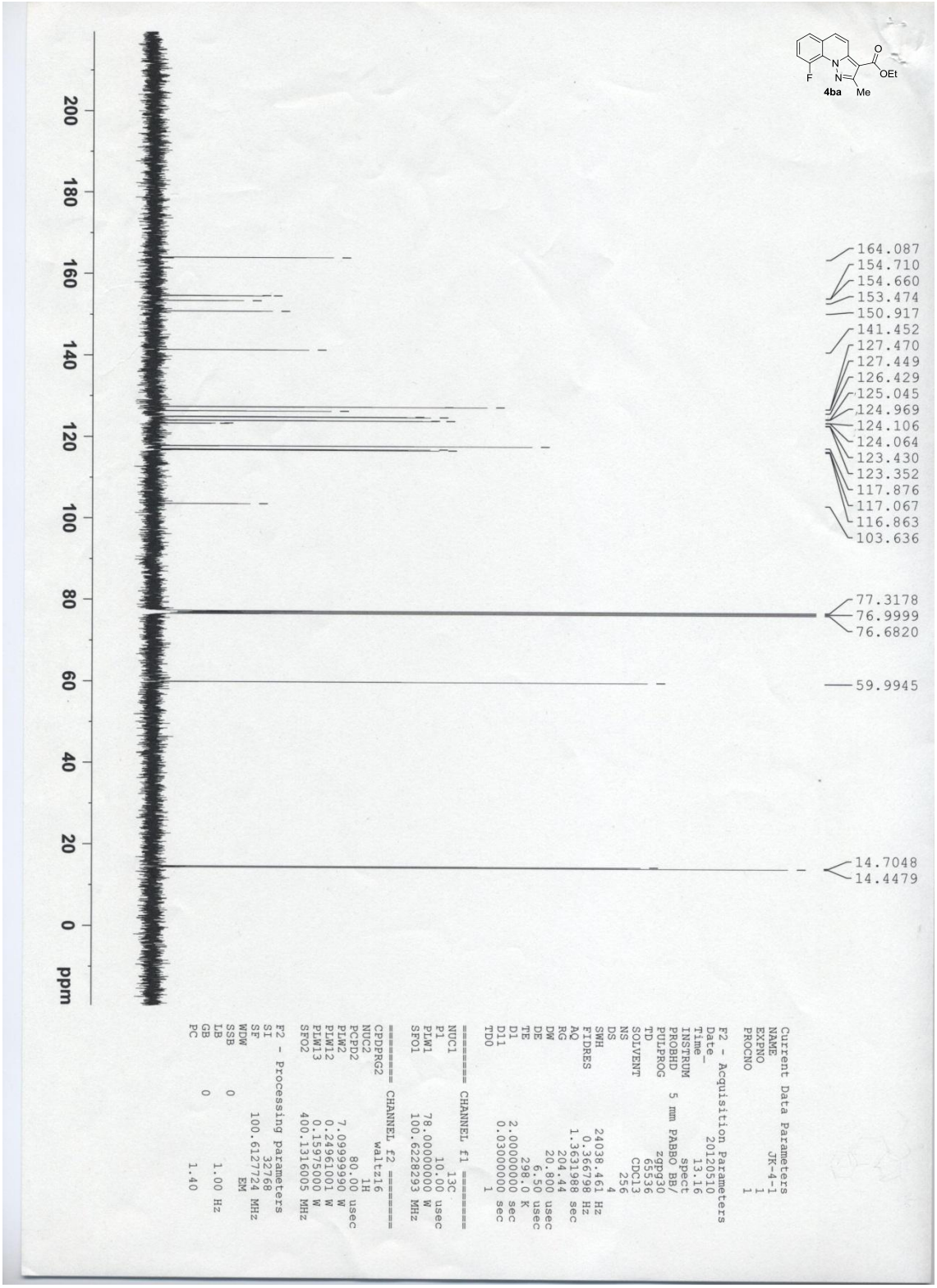


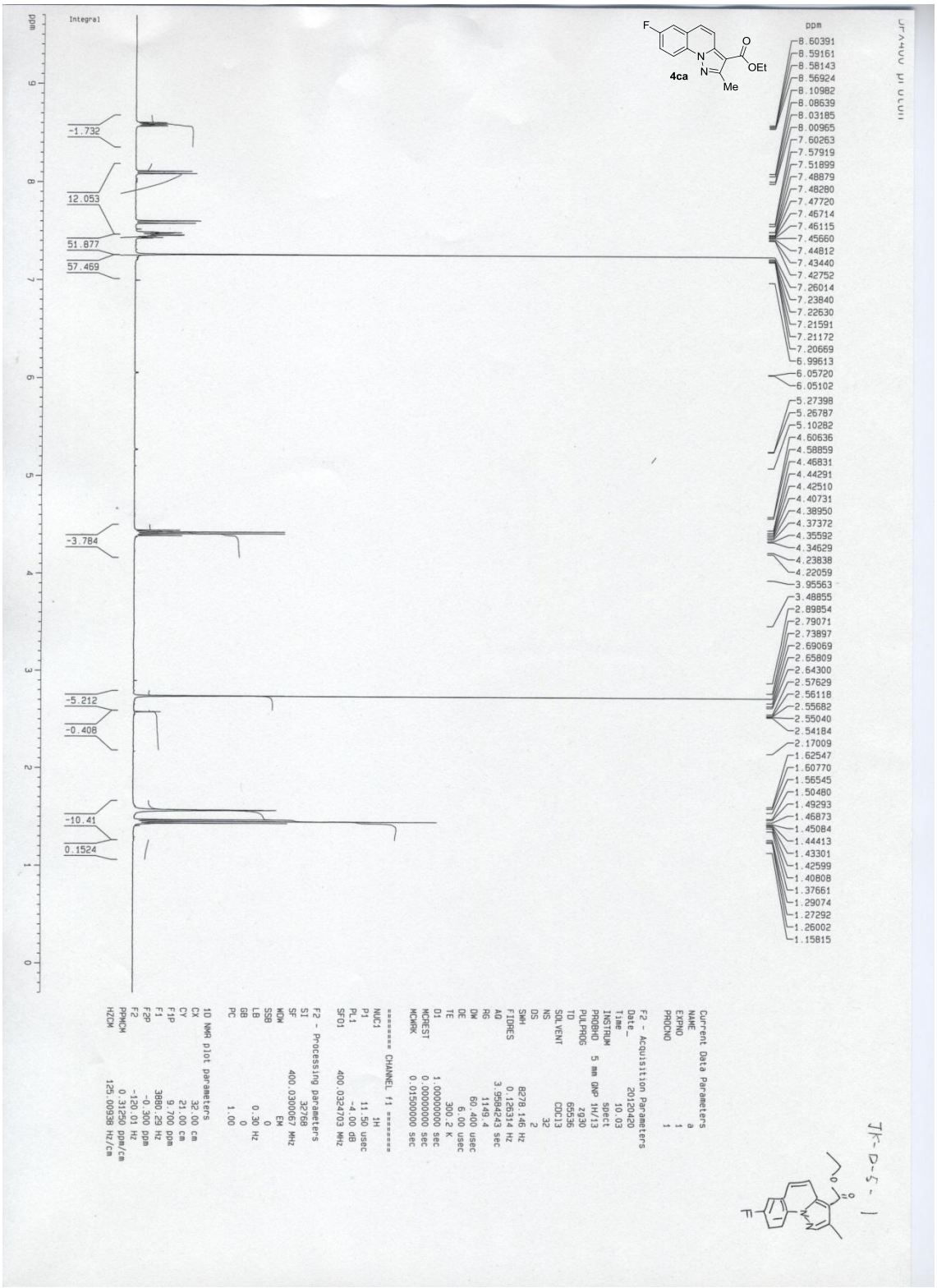


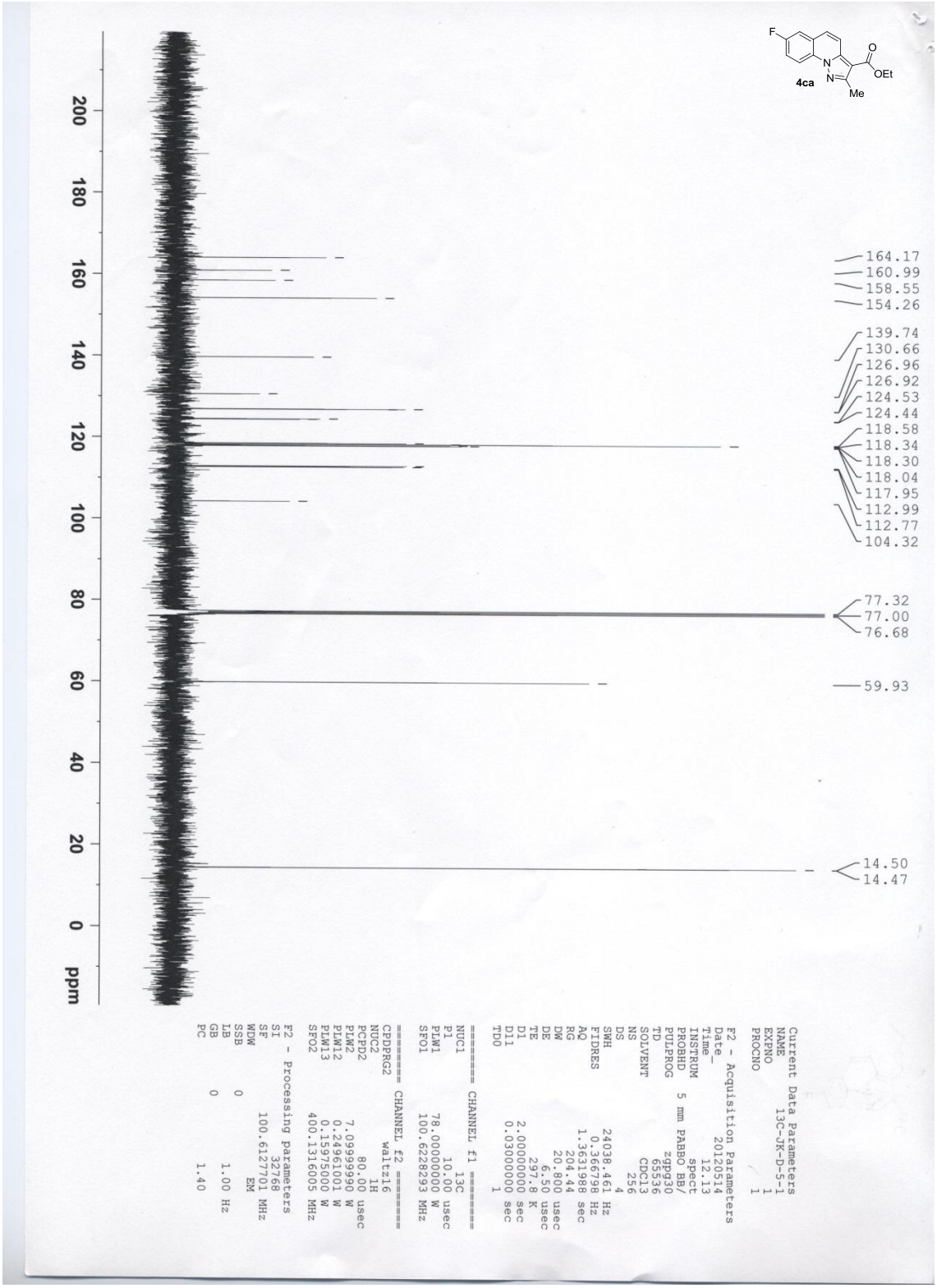


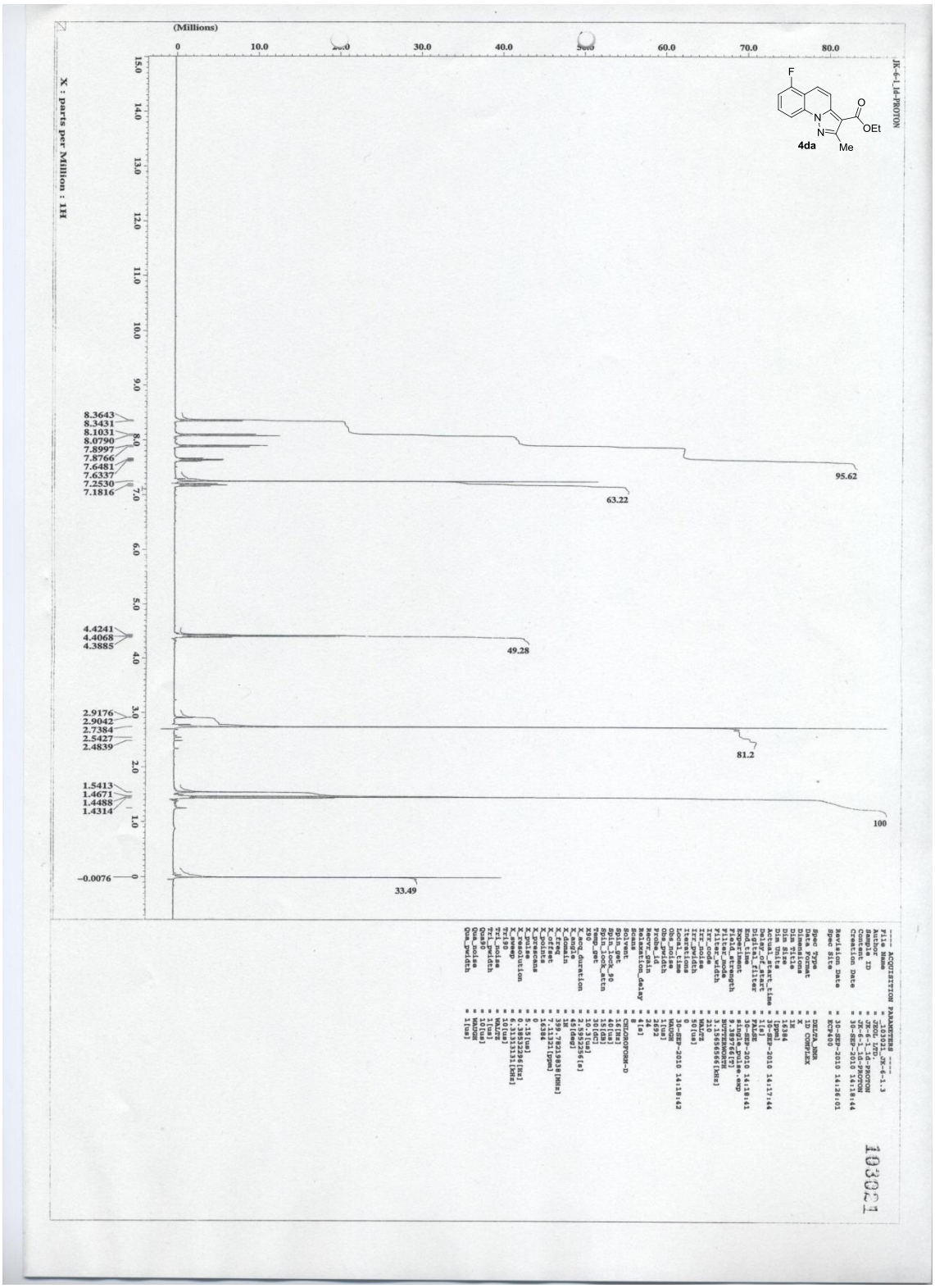

3aa
188.89
164.22
152.11
146.40
140.38
134.47
132.51
129.80
128.87
128.03
111.13
77.32
77.00
76.69
59.96
14.39
14.25
12.26
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-D-1-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120514
Time_ 17.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

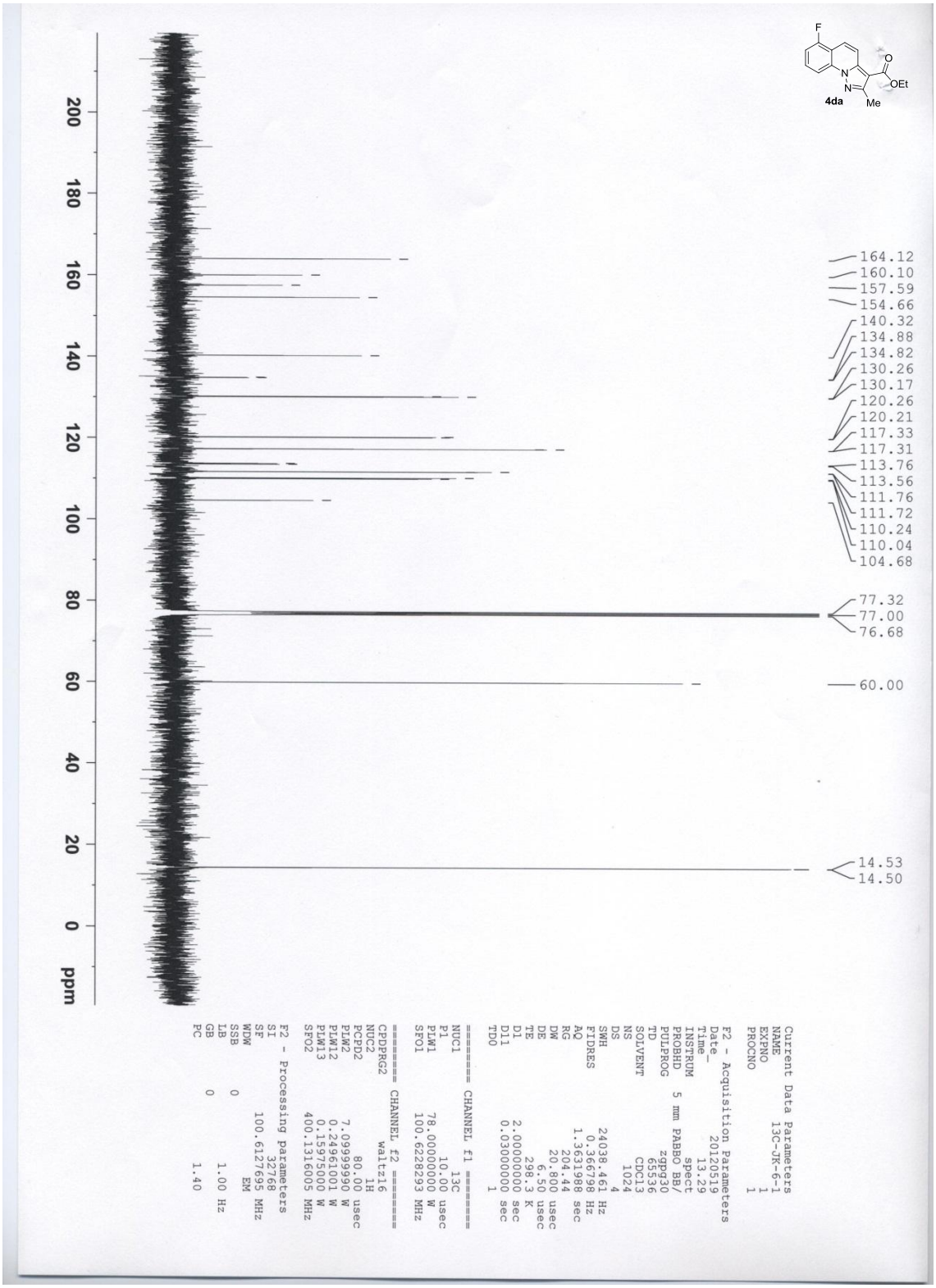

(Millions)
X : parts per Million : 1H
JK-4-1.1H-PROTON
4ba
Me
OEt
F
8.1397
8.1166
7.6433
7.6385
7.6154
7.5845
7.2530
4.4395
4.4212
4.4039
2.7625
1.4632
1.4459
1.4276
-0.0095
20.44
42.25
61.82
69.4
12.22
100
103001

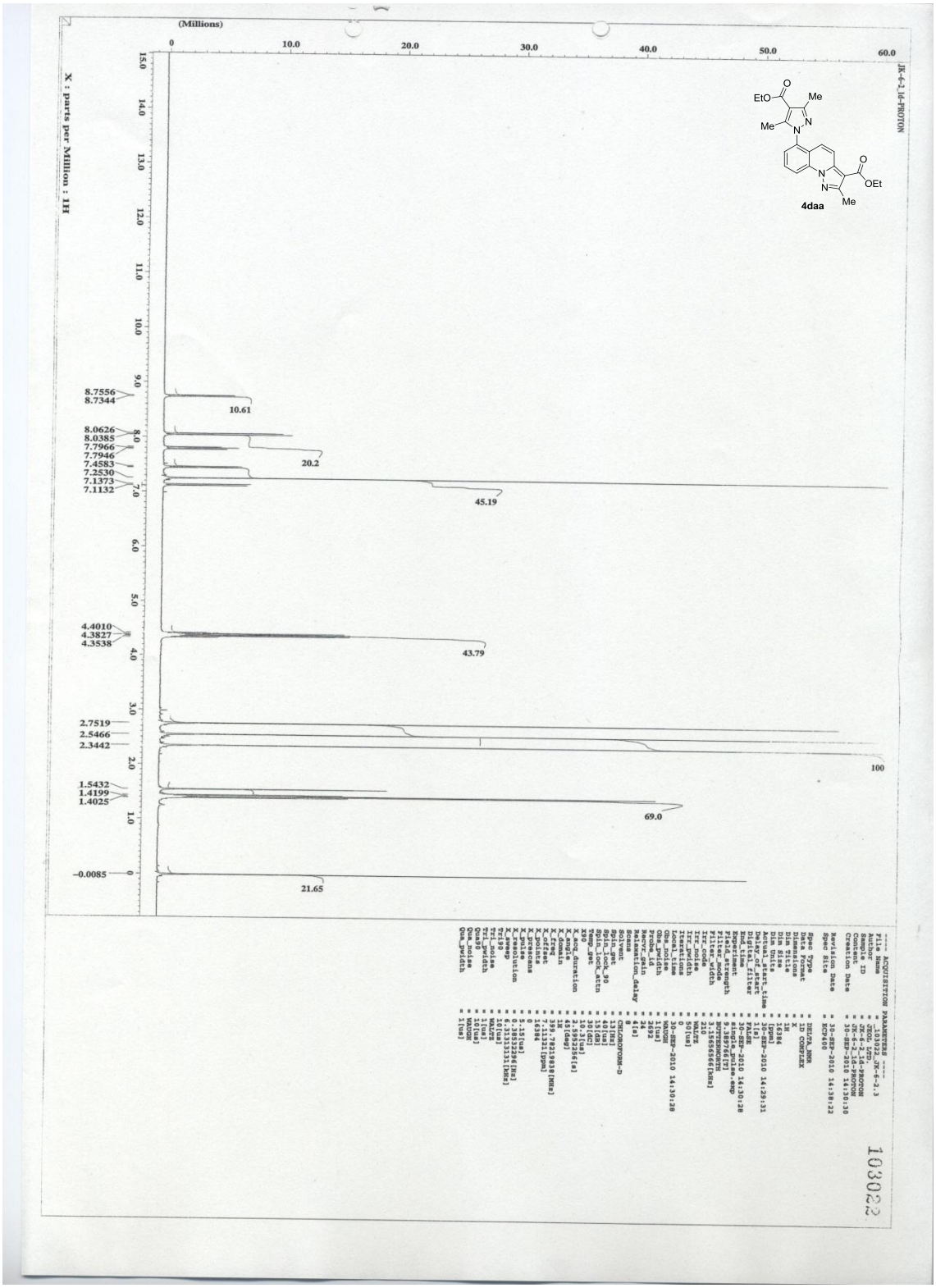

4ba
Me
OEt
O
F
N
164.087
154.710
154.660
153.474
150.917
141.452
127.470
127.449
126.429
125.045
124.969
124.106
124.064
123.430
123.352
117.876
117.067
116.863
103.636
77.3178
76.9999
76.6820
59.9945
14.7048
14.4479
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME JK-4-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120510
Time_ 13.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

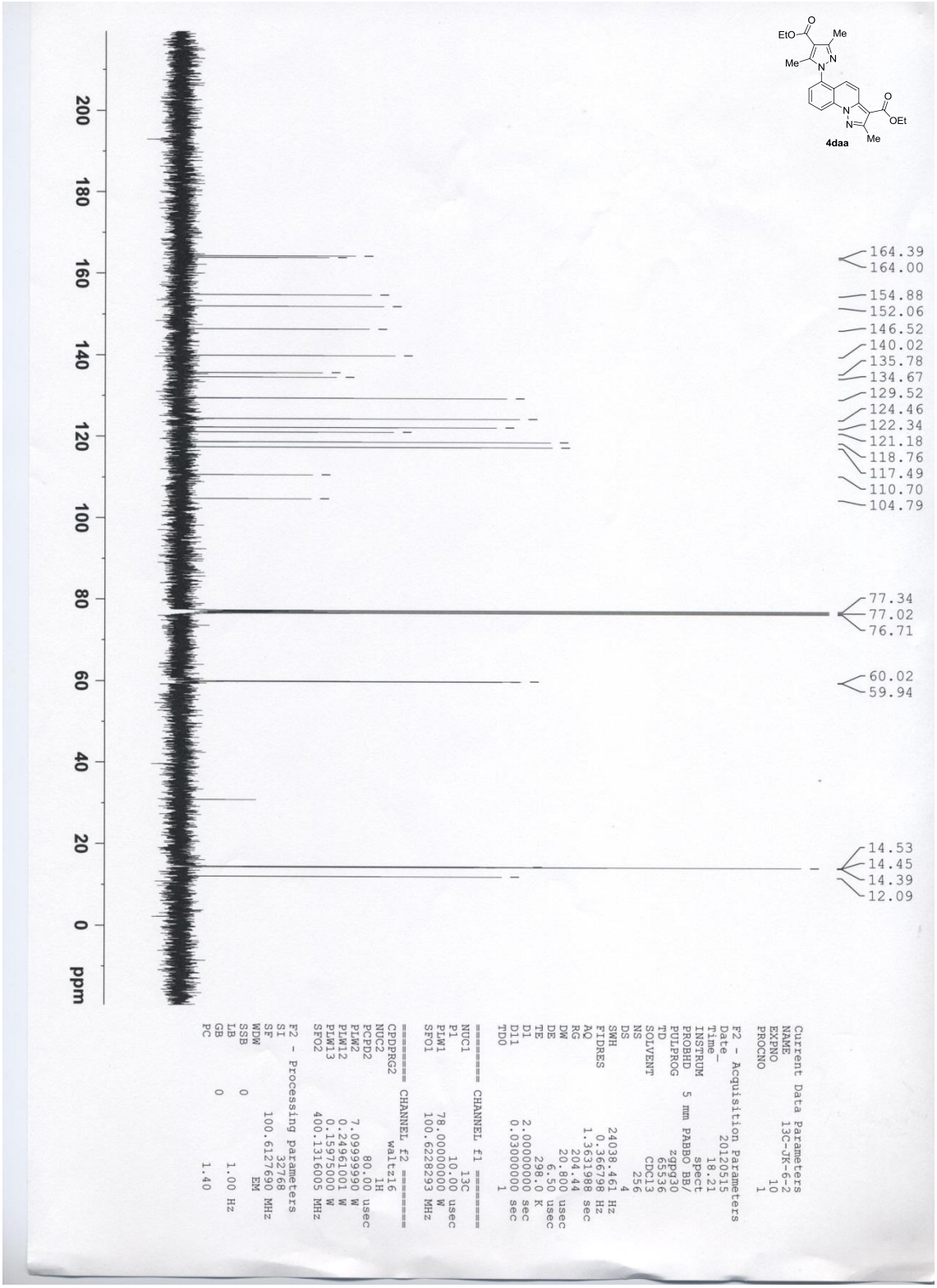


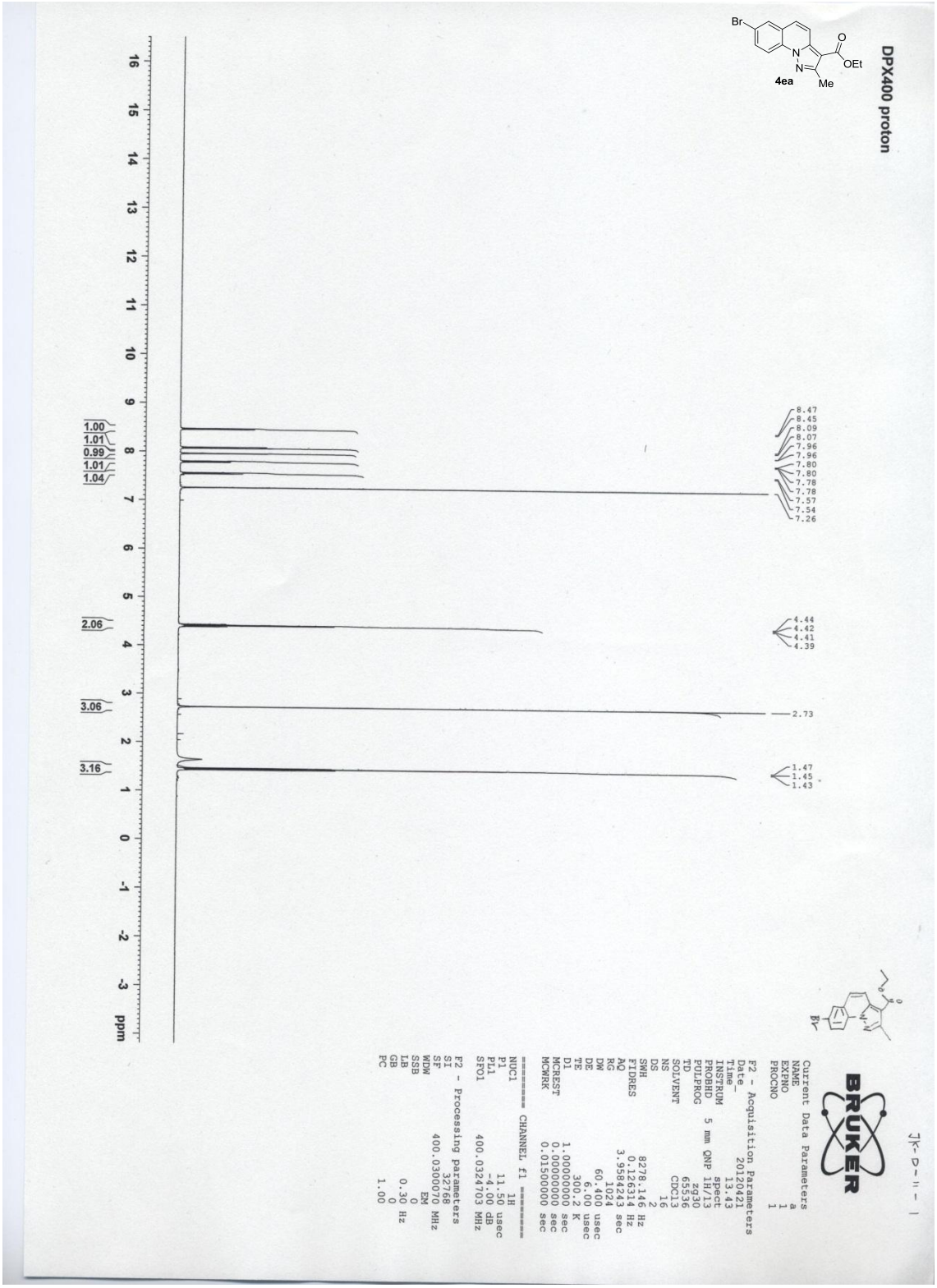

F
O
OEt
N
N
Me
4ca
164.17
160.99
158.55
154.26
139.74
130.66
126.96
126.92
124.53
124.44
118.58
118.34
118.30
118.04
117.95
112.99
112.77
104.32
77.32
77.00
76.68
59.93
14.50
14.47
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-D-5-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120514
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

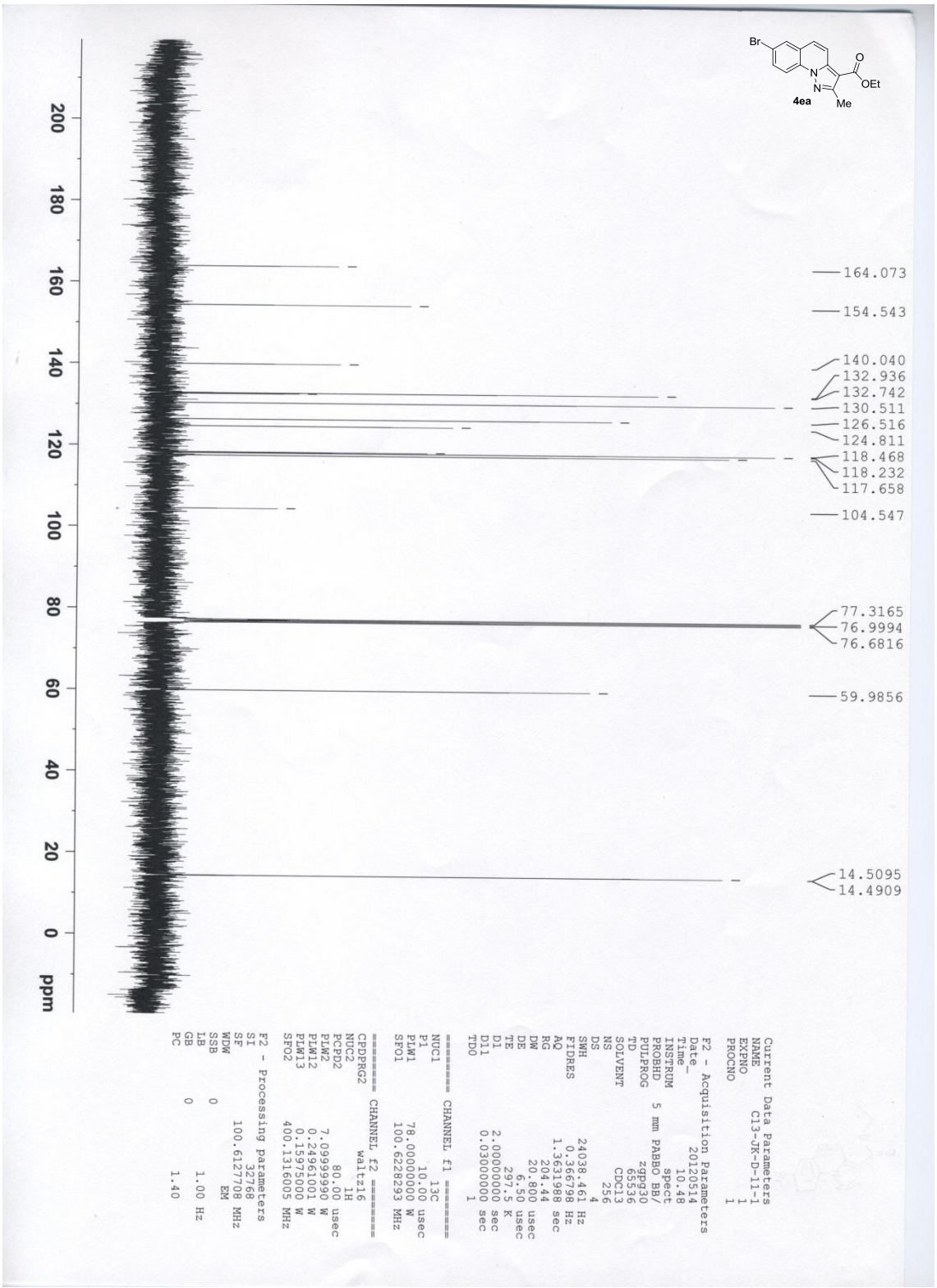

JK-6-1_1H-PROTON
X : parts per Million : 1H
(Millions)
F
O
OEt
N
N
Me
4da
8.3643
8.3431
8.1031
8.0790
7.8997
7.8766
7.6481
7.6337
7.2530
7.1816
4.4241
4.4068
4.3885
2.9176
2.9042
2.7384
2.5427
2.4839
1.5413
1.4671
1.4488
1.4314
-0.0076
95.62
63.22
49.28
81.2
100
33.49

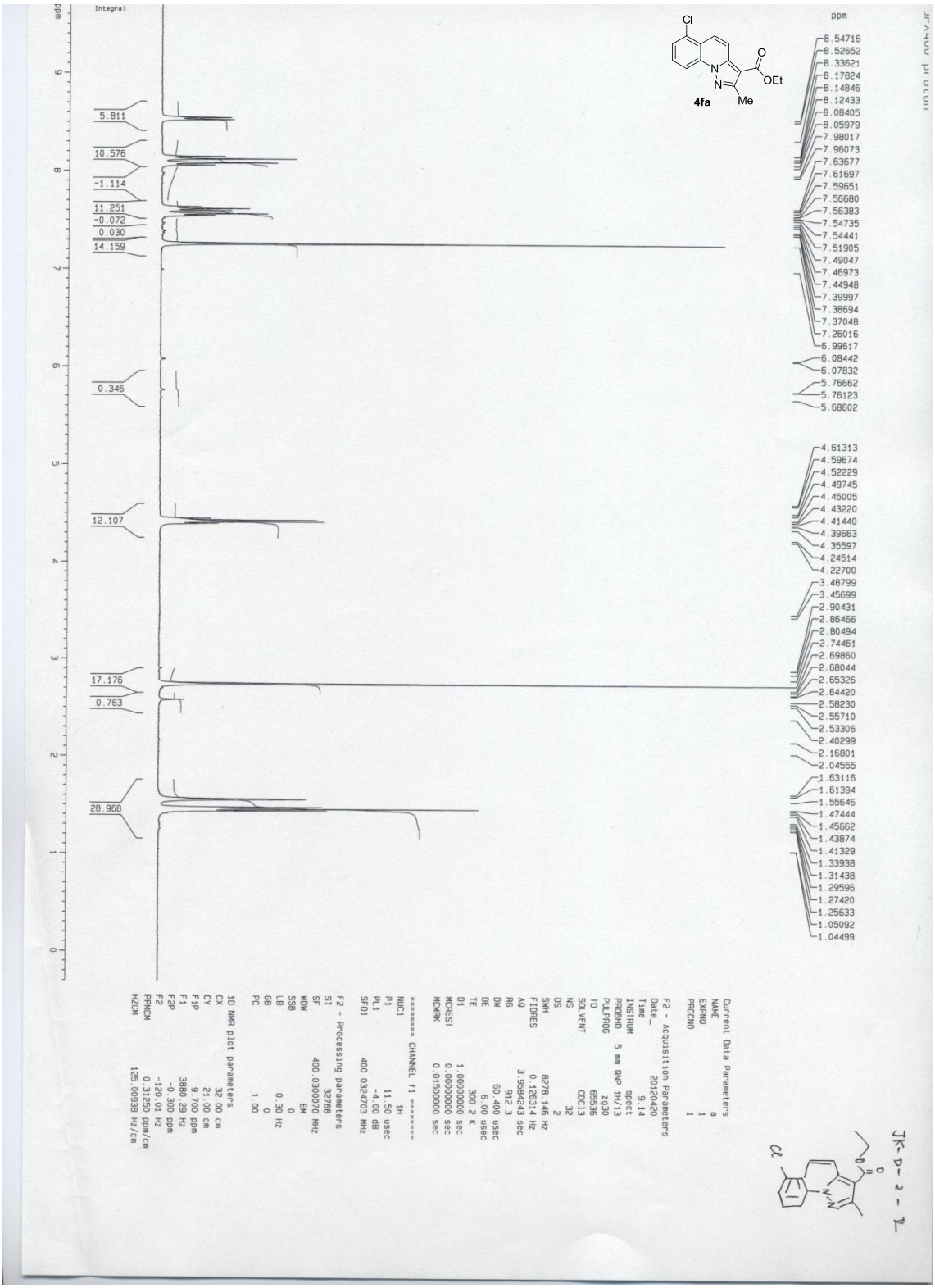

F
O
OEt
N
N
Me
4da
164.12
160.10
157.59
154.66
140.32
134.88
134.82
130.26
130.17
120.26
120.21
117.33
117.31
113.76
113.56
111.76
111.72
110.24
110.04
104.68
77.32
77.00
76.68
60.00
14.53
14.50
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-6-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120519
Time_ 13.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127695 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

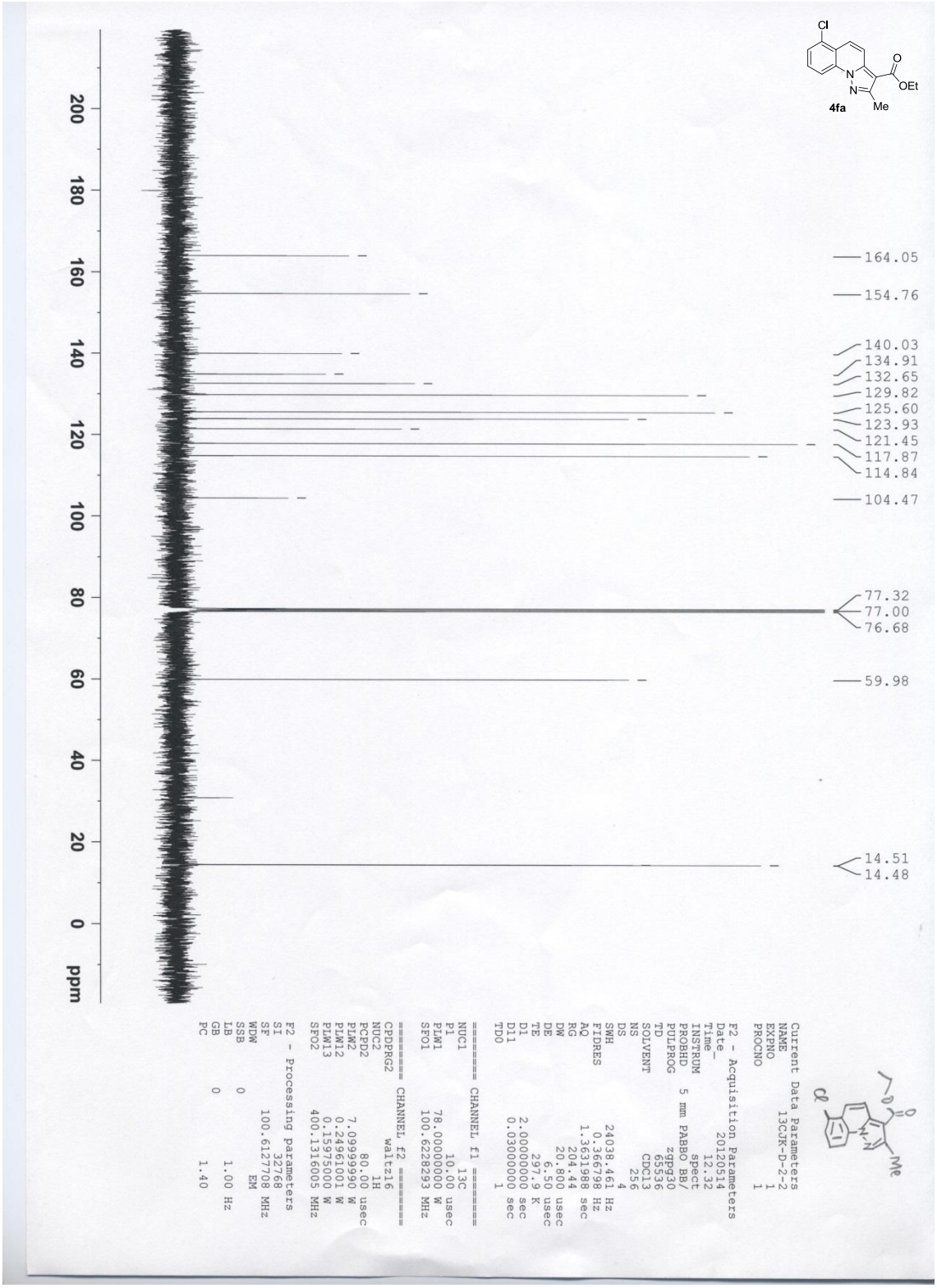

(Millions)
X : parts per Million : 1H
JK-6-2.14-PROTON
4daa
8.7556
8.7344
10.61
8.0626
8.0385
7.7966
7.7946
7.4583
7.2530
7.1373
7.1132
20.2
45.19
4.4010
4.3827
4.3538
43.79
2.7519
2.5466
2.3442
100
1.5432
1.4199
1.4025
69.0
-0.0085
21.65

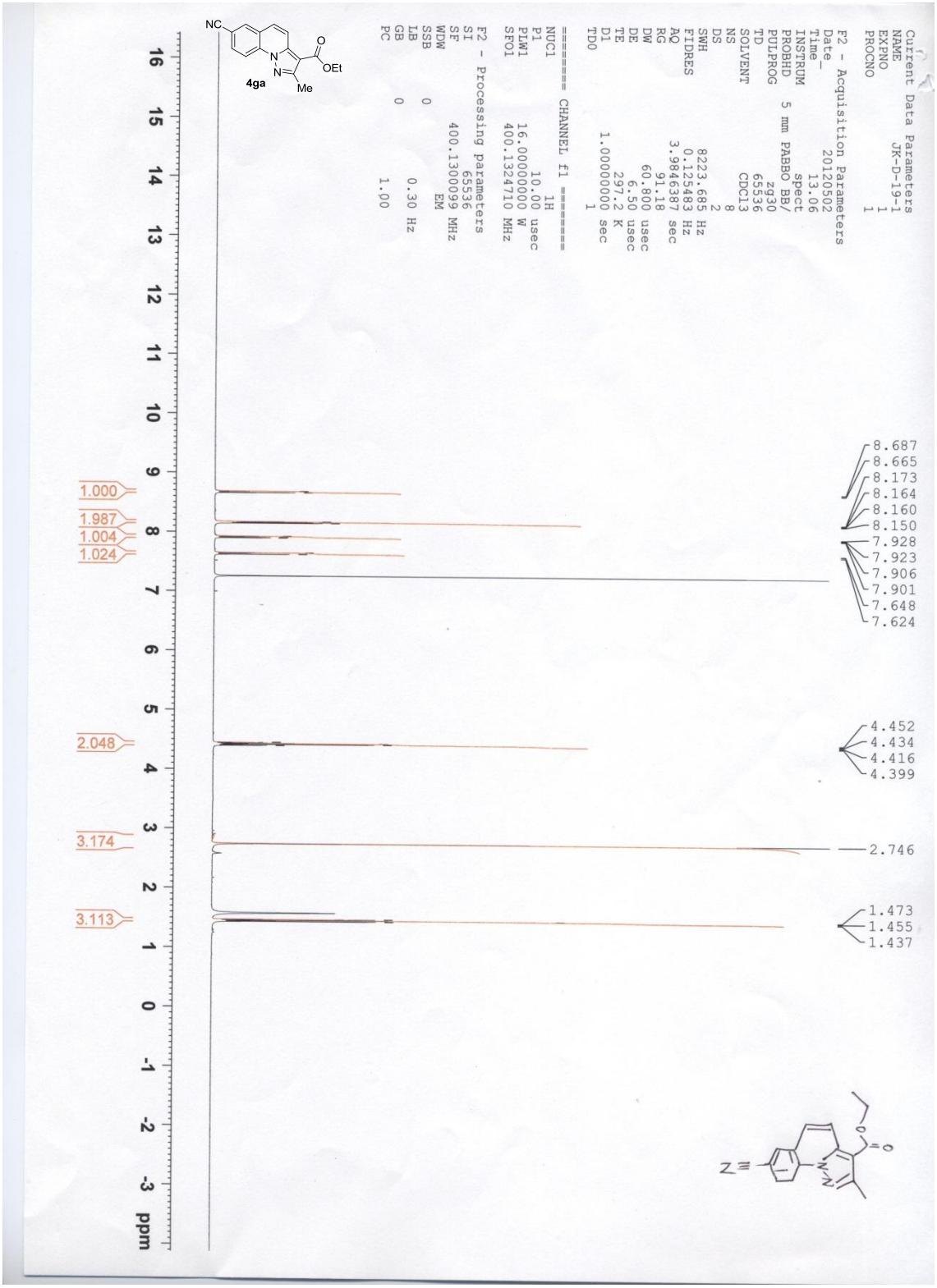


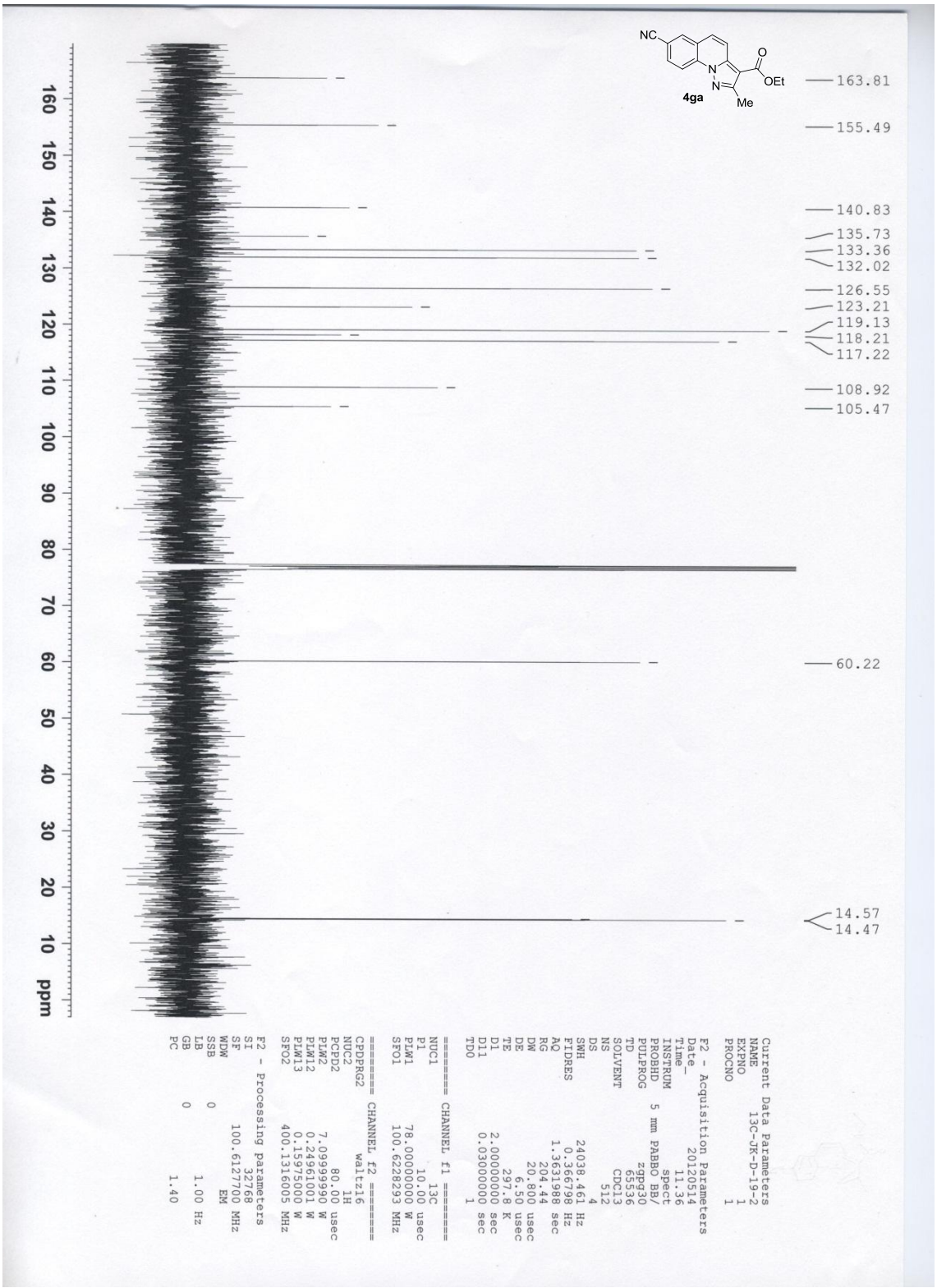

DPX400 proton
4ea
Br
O
OEt
N
N
Me
8.47
8.45
8.09
8.07
7.96
7.96
7.80
7.80
7.78
7.78
7.57
7.54
7.26
4.44
4.42
4.41
4.39
2.73
1.47
1.45
1.43
1.00
1.01
0.99
1.01
1.04
2.06
3.06
3.16
ppm
BRUKER
Current Data Parameters
NAME a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120421
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 1024
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

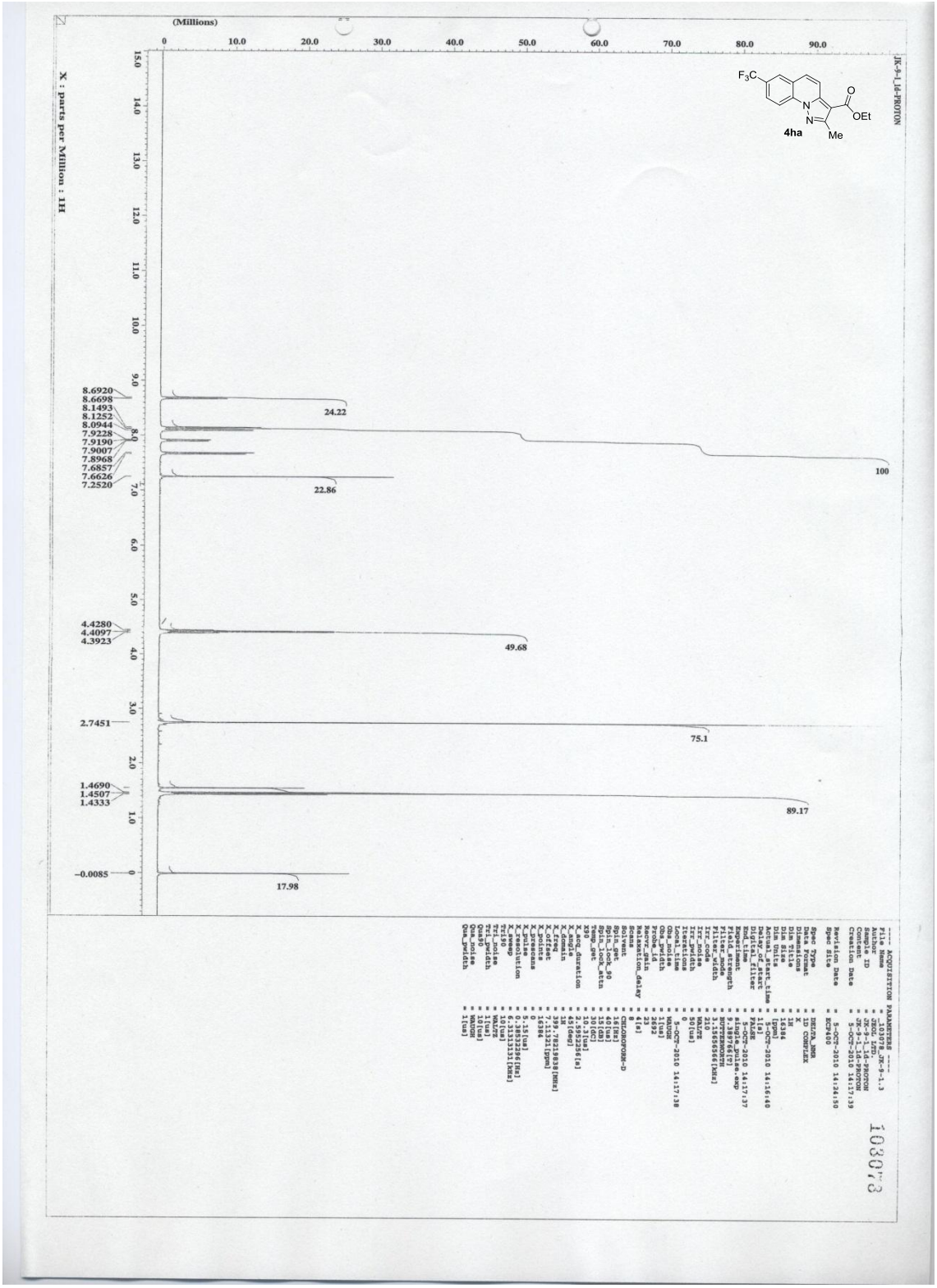


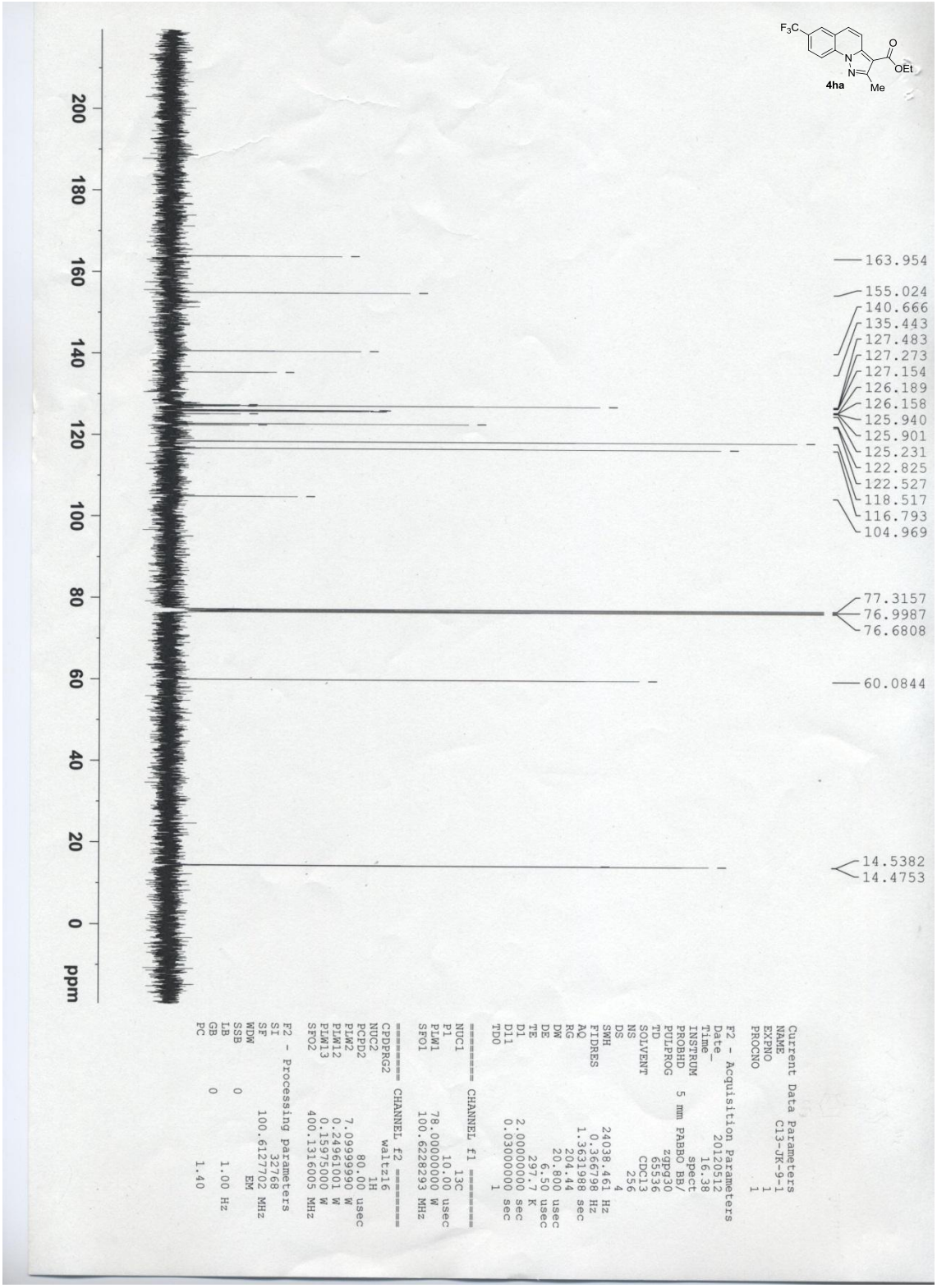


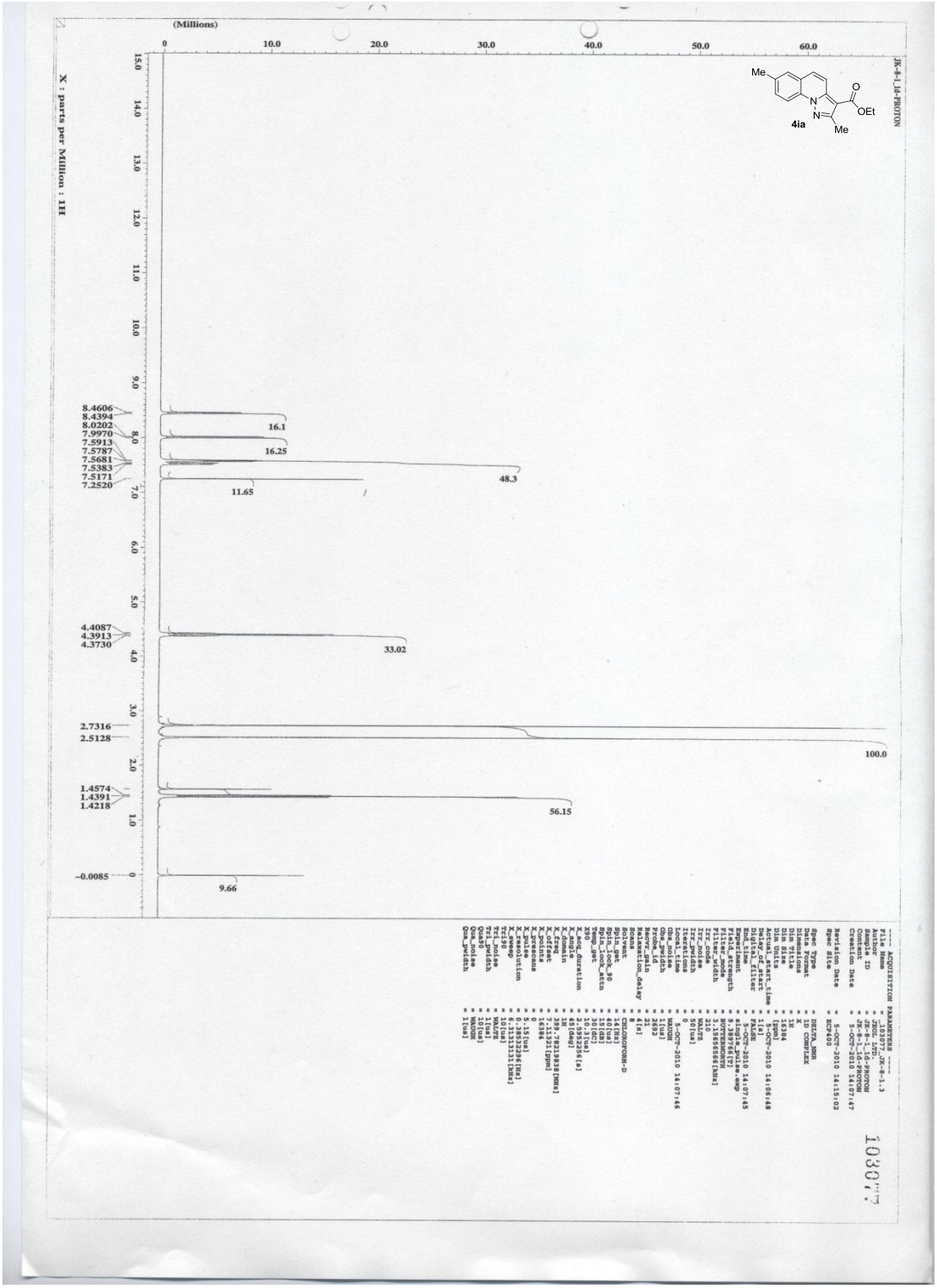


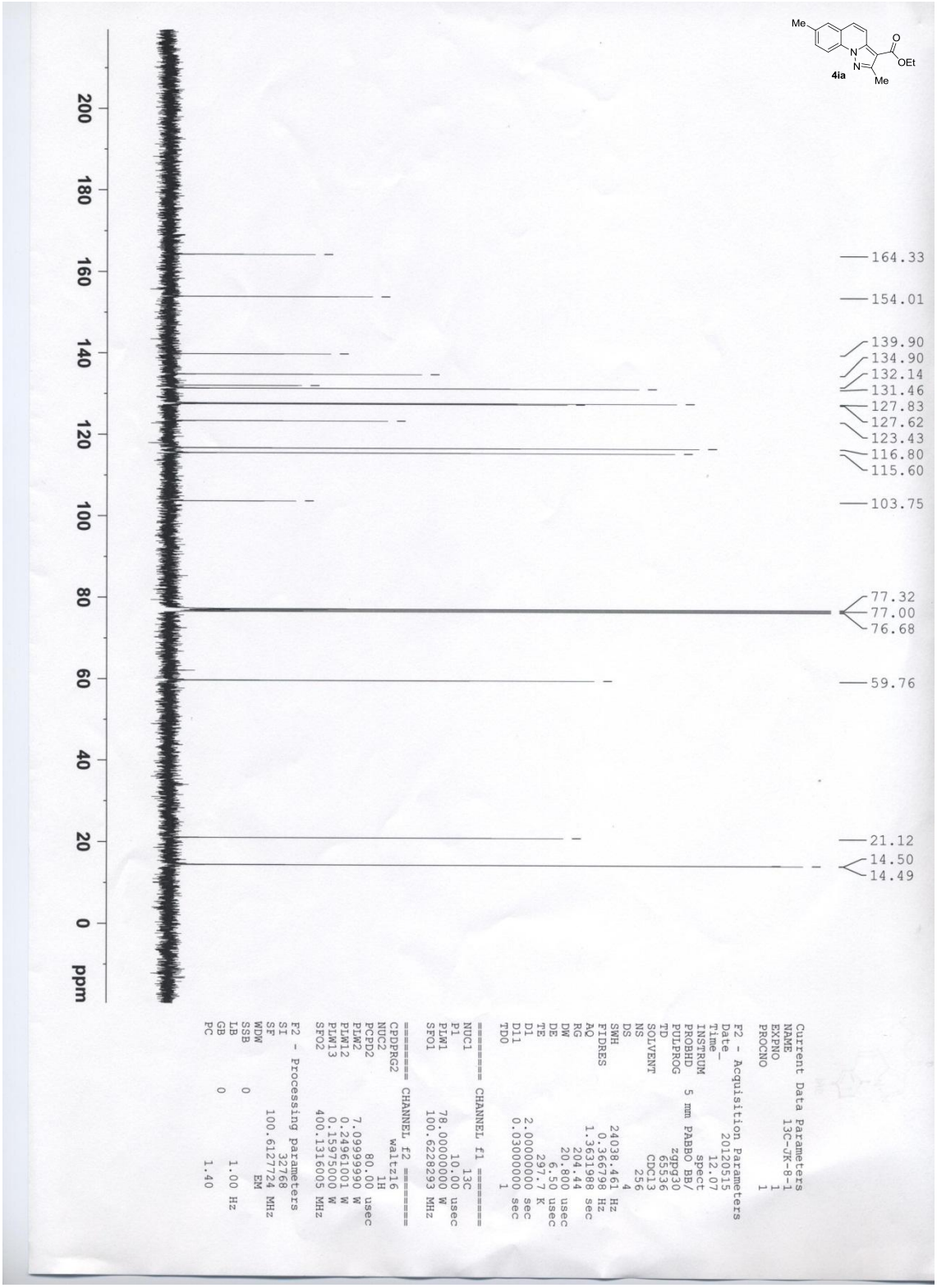

Current Data Parameters
NAME JK-D-19-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120502
Time_ 13.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.18
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4ga
NC
O
OEt
N
N
Me
8.687
8.665
8.173
8.164
8.160
8.150
7.928
7.923
7.906
7.901
7.648
7.624
4.452
4.434
4.416
4.399
2.746
1.473
1.455
1.437
1.000
1.987
1.004
1.024
2.048
3.174
3.113
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm

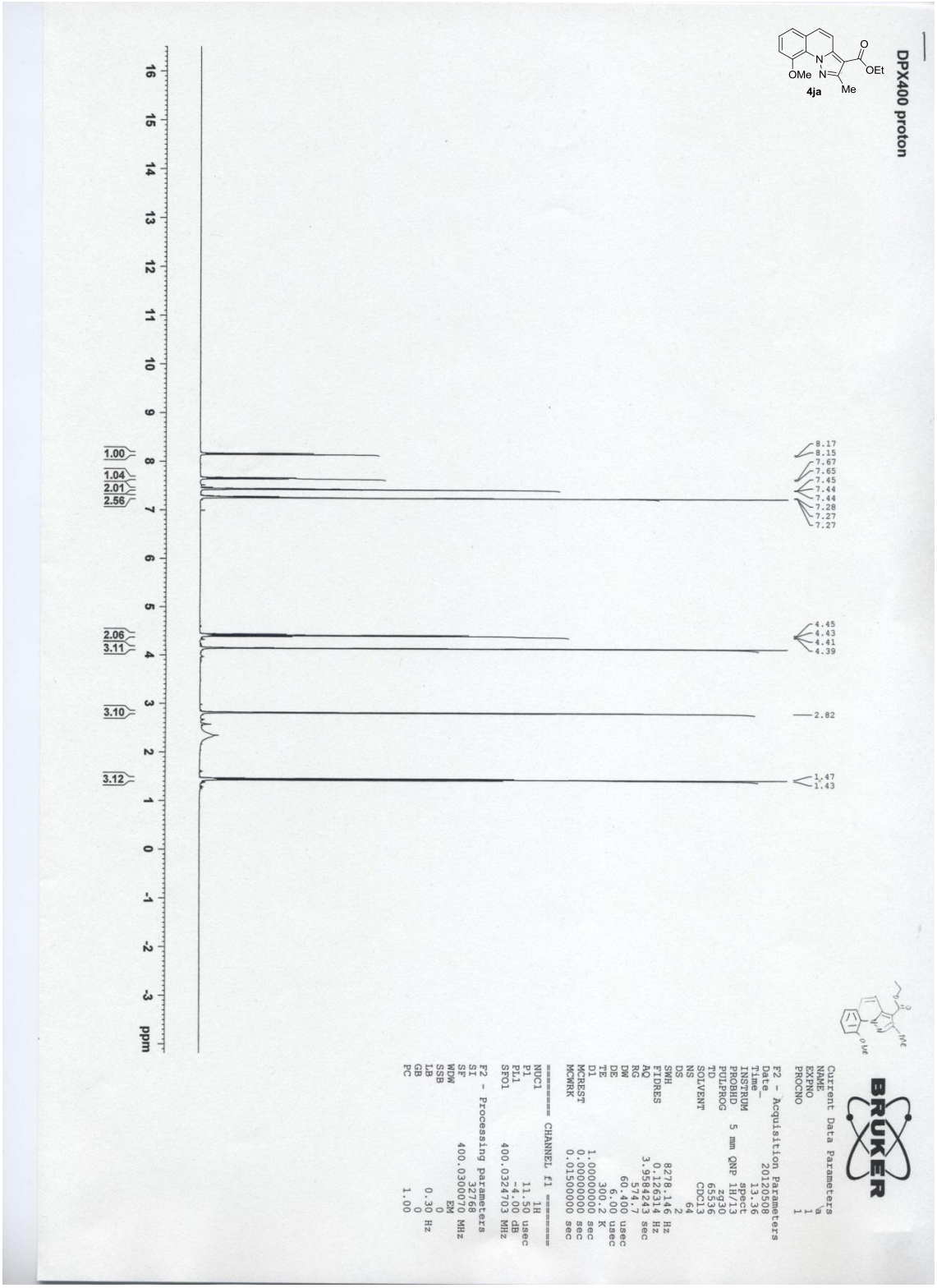


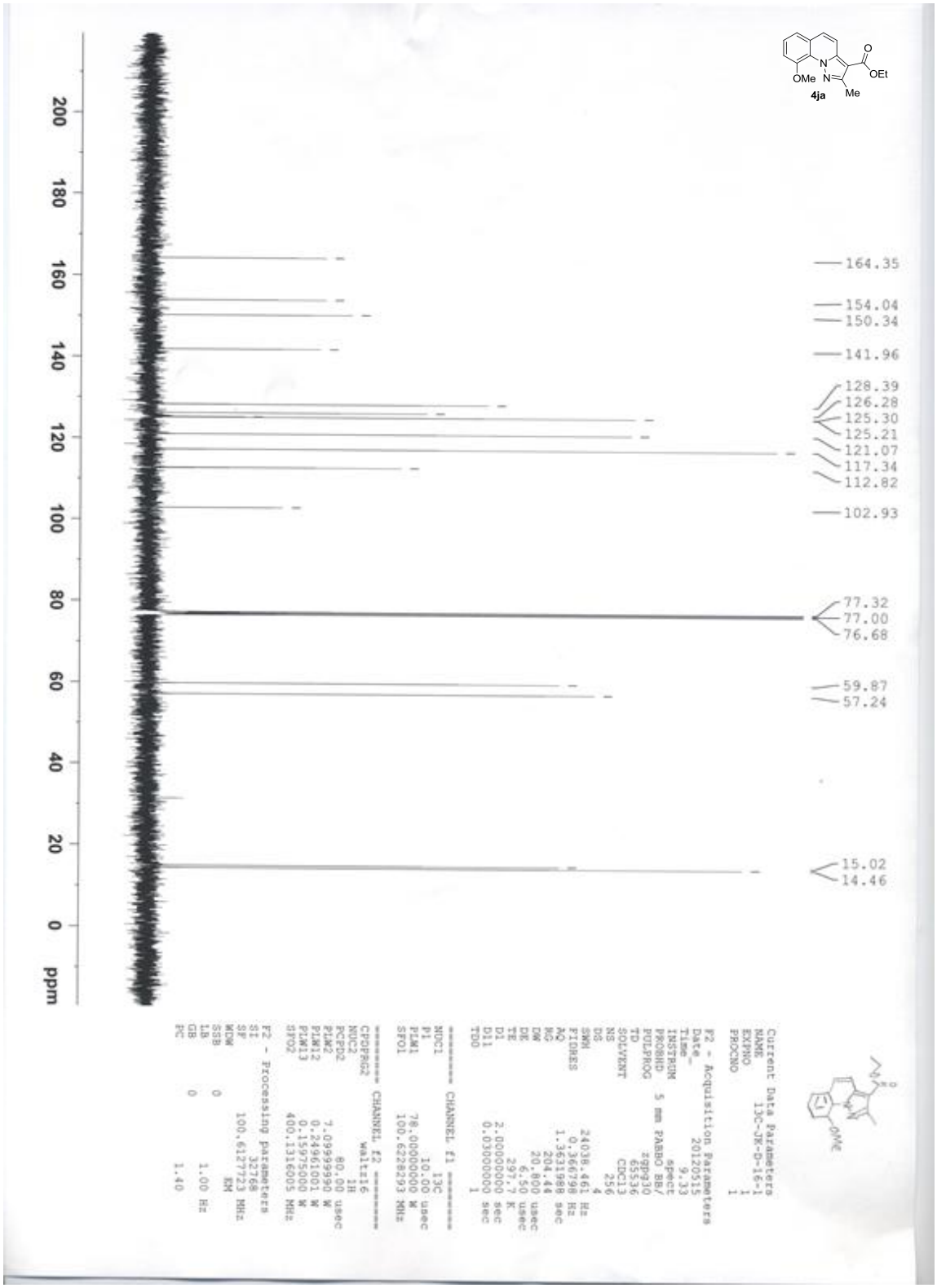

(Millions)
X : parts per Million : 1H
JK-5-1.1d-PROTON
4ha
8.6920
8.6698
8.1493
8.1252
8.0944
7.9228
7.9190
7.9007
7.8968
7.6857
7.6626
7.2520
4.4280
4.4097
4.3923
2.7451
1.4690
1.4507
1.4333
-0.0085
24.22
22.86
100
49.68
75.1
89.17
17.98
ACQUISITION PARAMETERS
108073

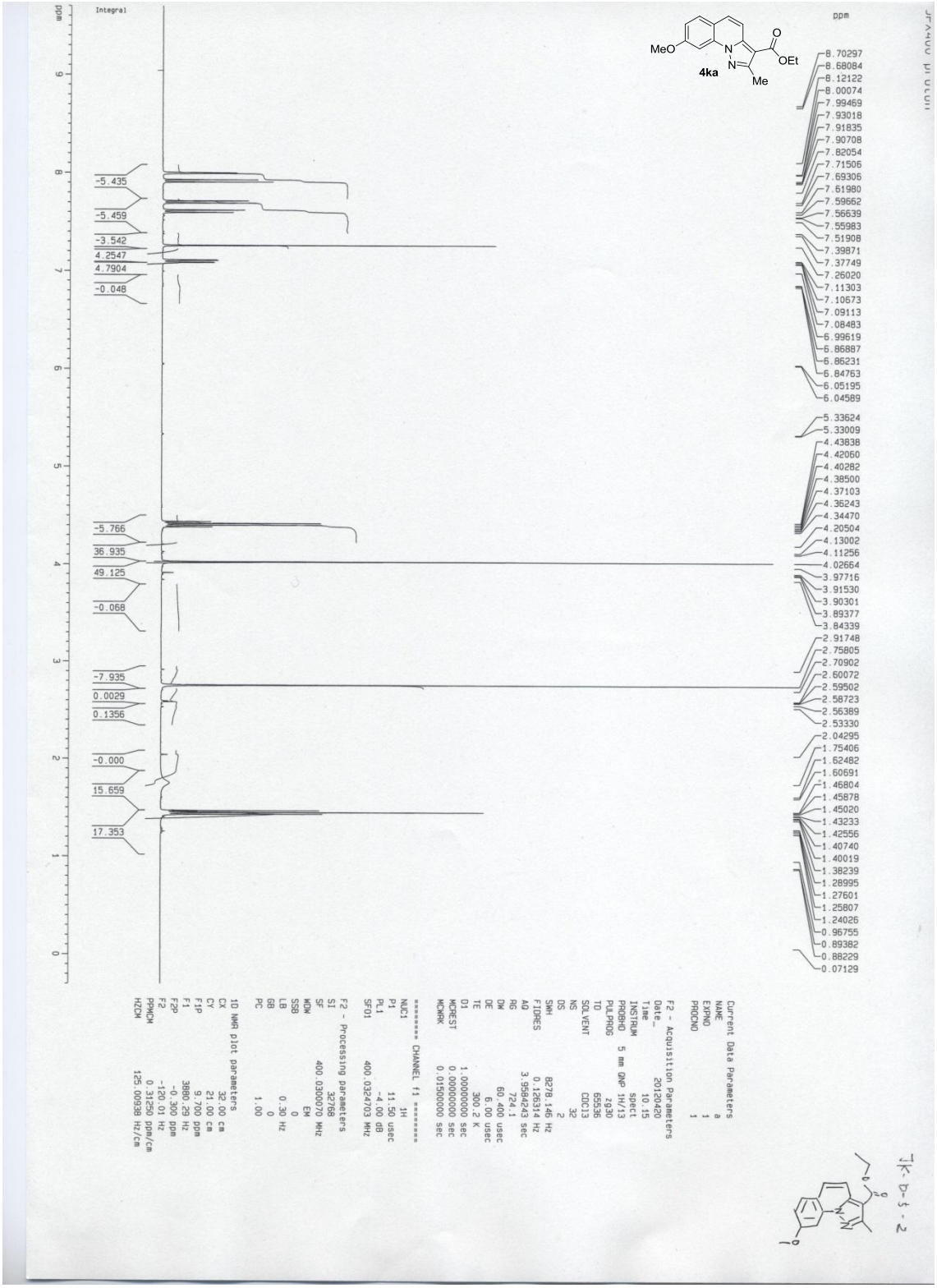

4ha
163.954
155.024
140.666
135.443
127.483
127.273
127.154
126.189
126.158
125.940
125.901
125.231
122.825
122.527
118.517
116.793
104.969
77.3157
76.9987
76.6808
60.0844
14.5382
14.4753
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME C13-JK-9-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120512
Time 16.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127702 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

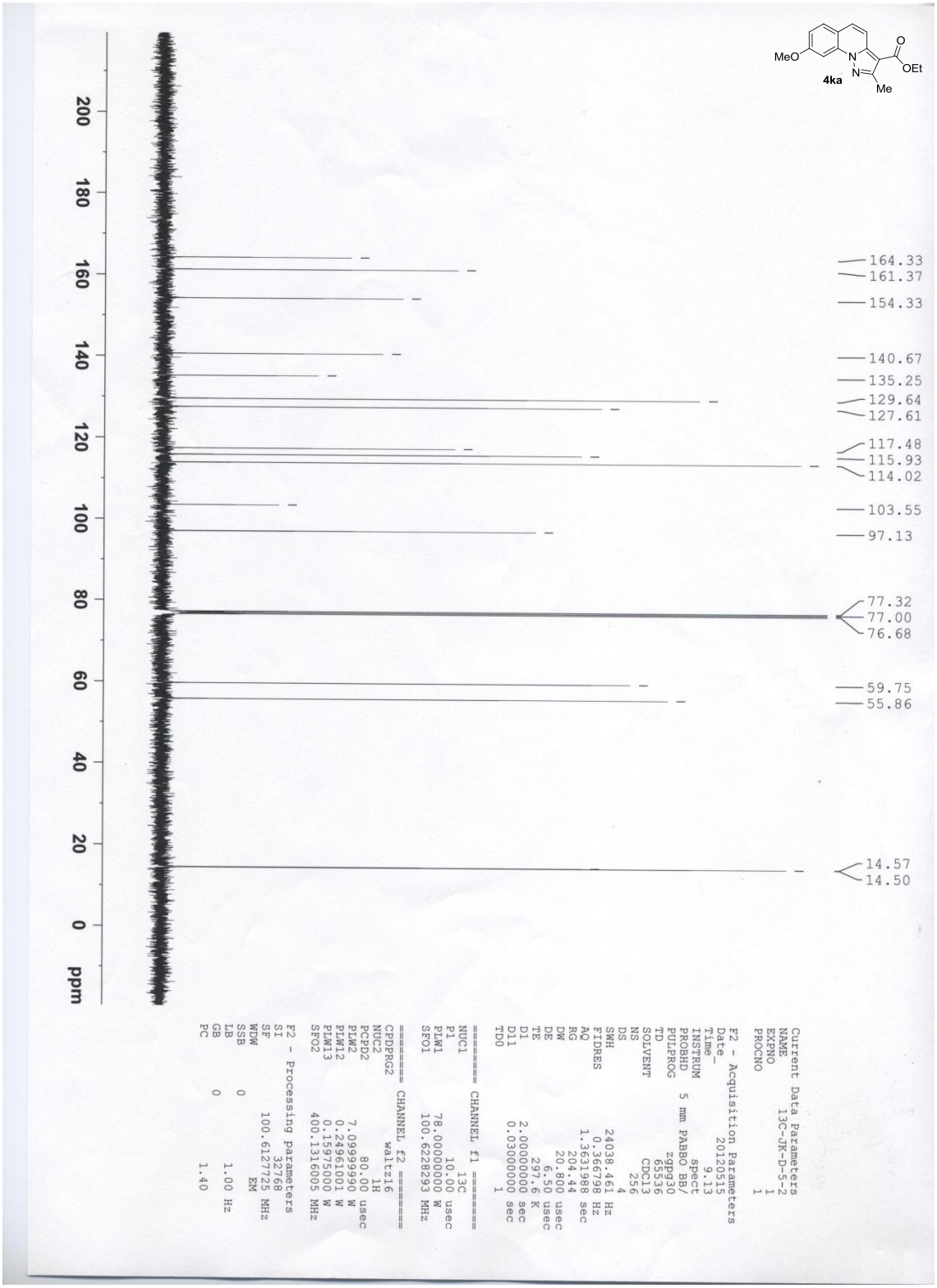


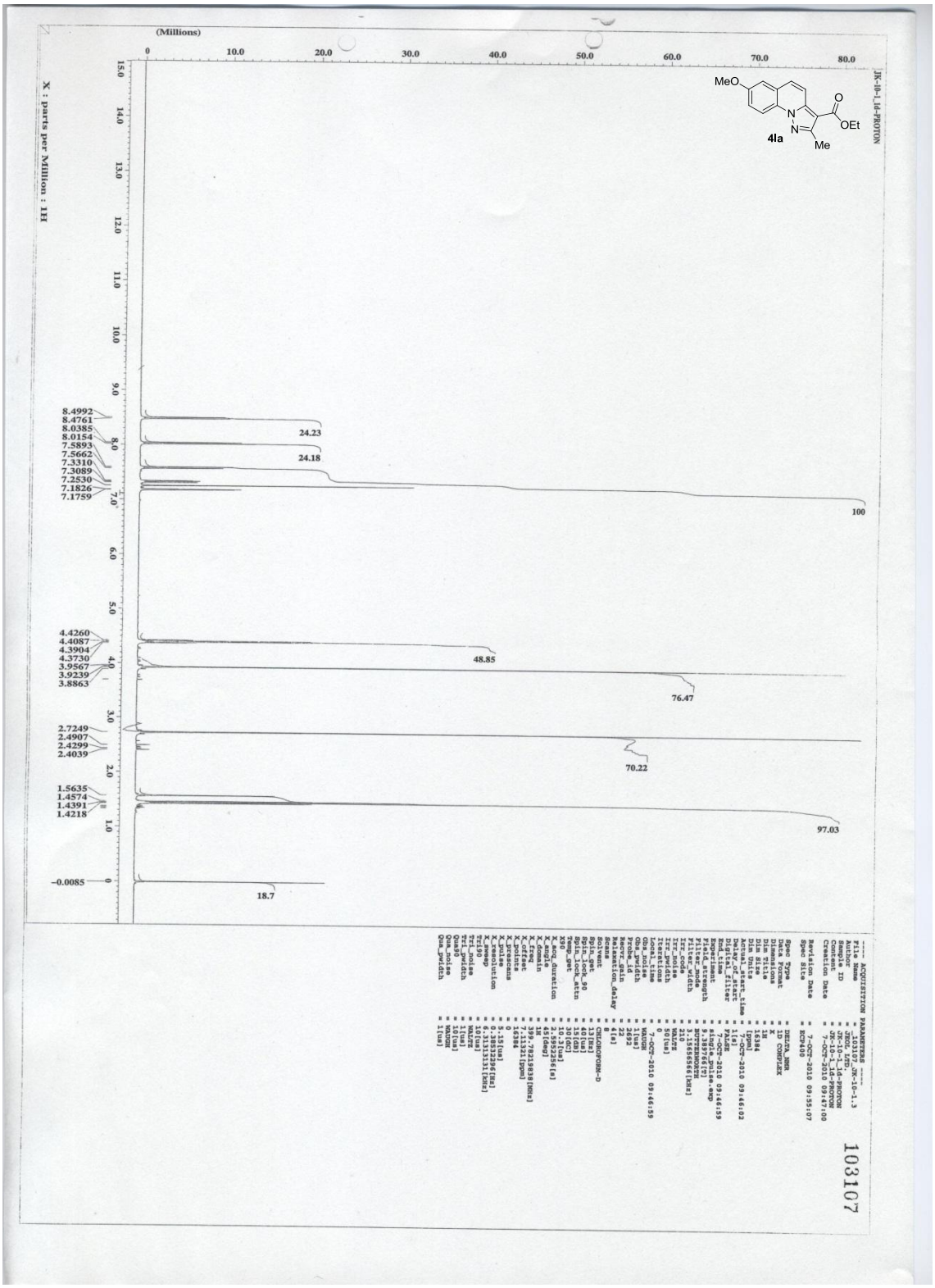

Me
O
OEt
N
N
Me
4ia
164.33
154.01
139.90
134.90
132.14
131.46
127.83
127.62
123.43
116.80
115.60
103.75
77.32
77.00
76.68
59.76
21.12
14.50
14.49
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-8-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120515
Time_ 12.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 78.00000000 W
SFO1 100.628293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.09999990 W
PL12 0.24961001 W
PL13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

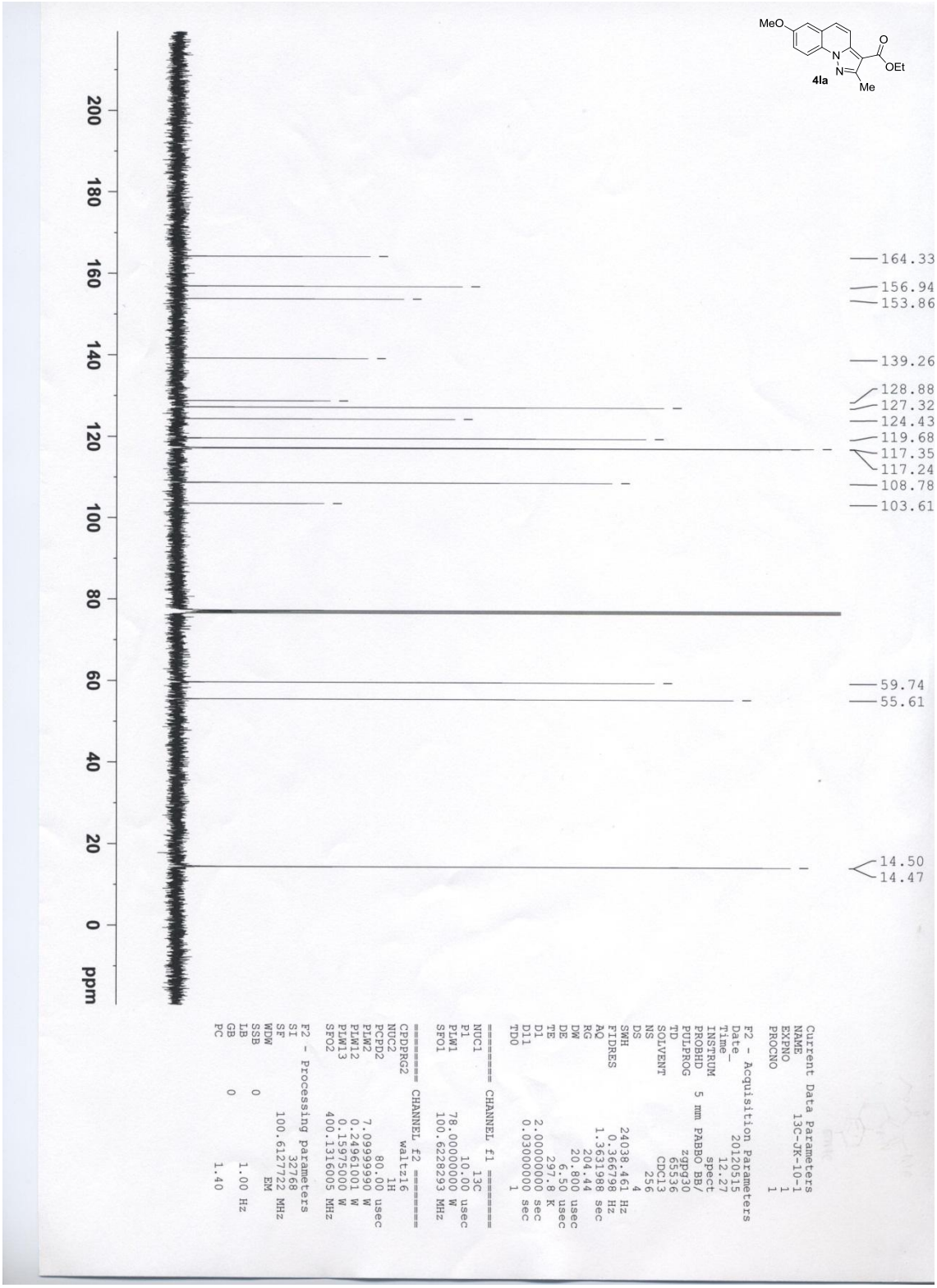

DPX400 proton
4ja
OMe
OEt
Me
8.17
8.15
7.67
7.65
7.45
7.44
7.44
7.28
7.27
7.27
4.45
4.43
4.41
4.39
2.82
1.47
1.43
1.00
1.04
2.01
2.56
2.06
3.11
3.10
3.12
ppm
BRUKER
Current Data Parameters
NAME a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120508
Time 13.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

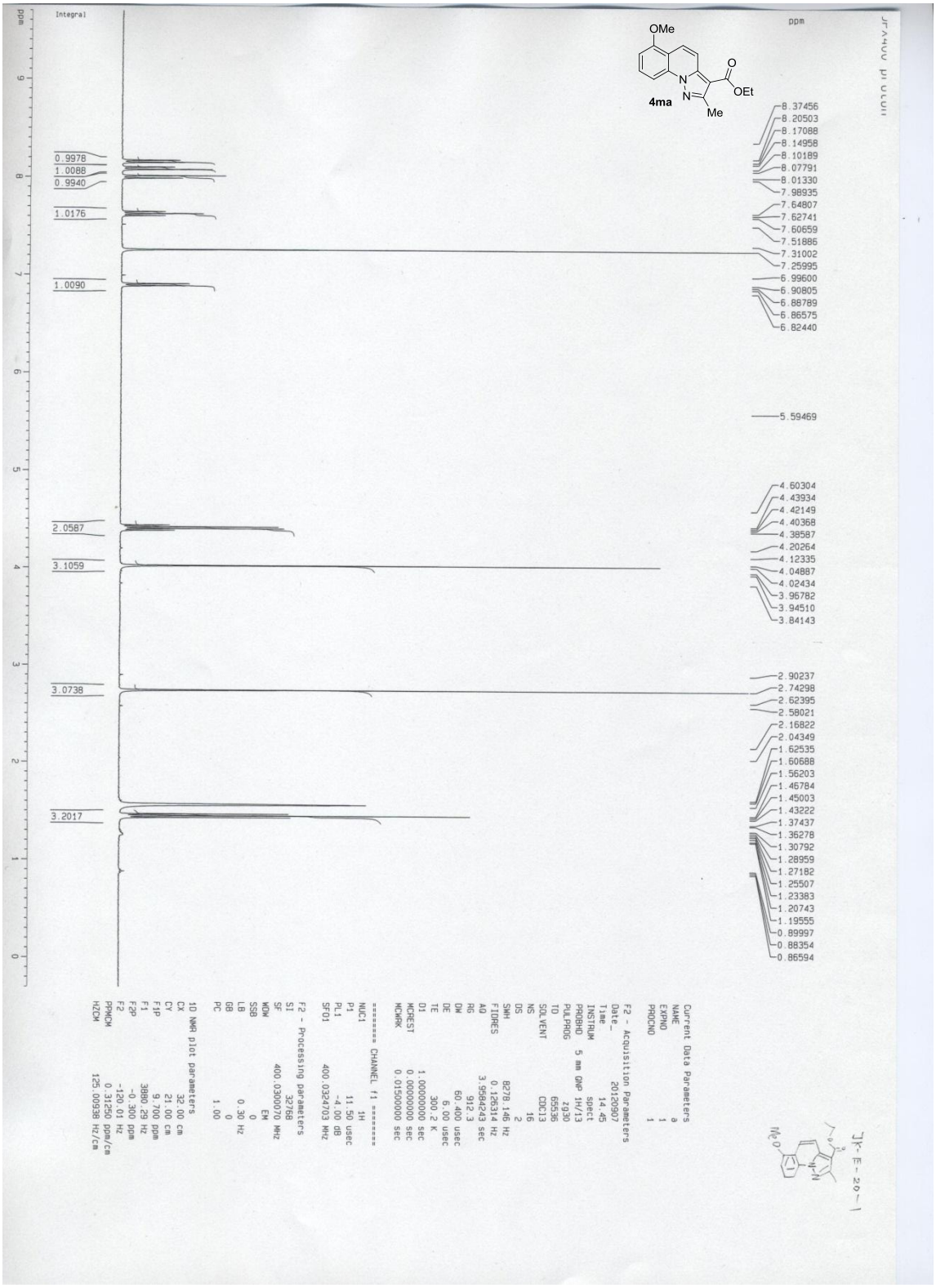

4ja
OMe
OEt
Me
164.35
154.04
150.34
141.96
128.39
126.28
125.30
125.21
121.07
117.34
112.82
102.93
77.32
77.00
76.68
59.87
57.24
15.02
14.46
200
180
160
140
120
100
80
60
40
20
0
ppm

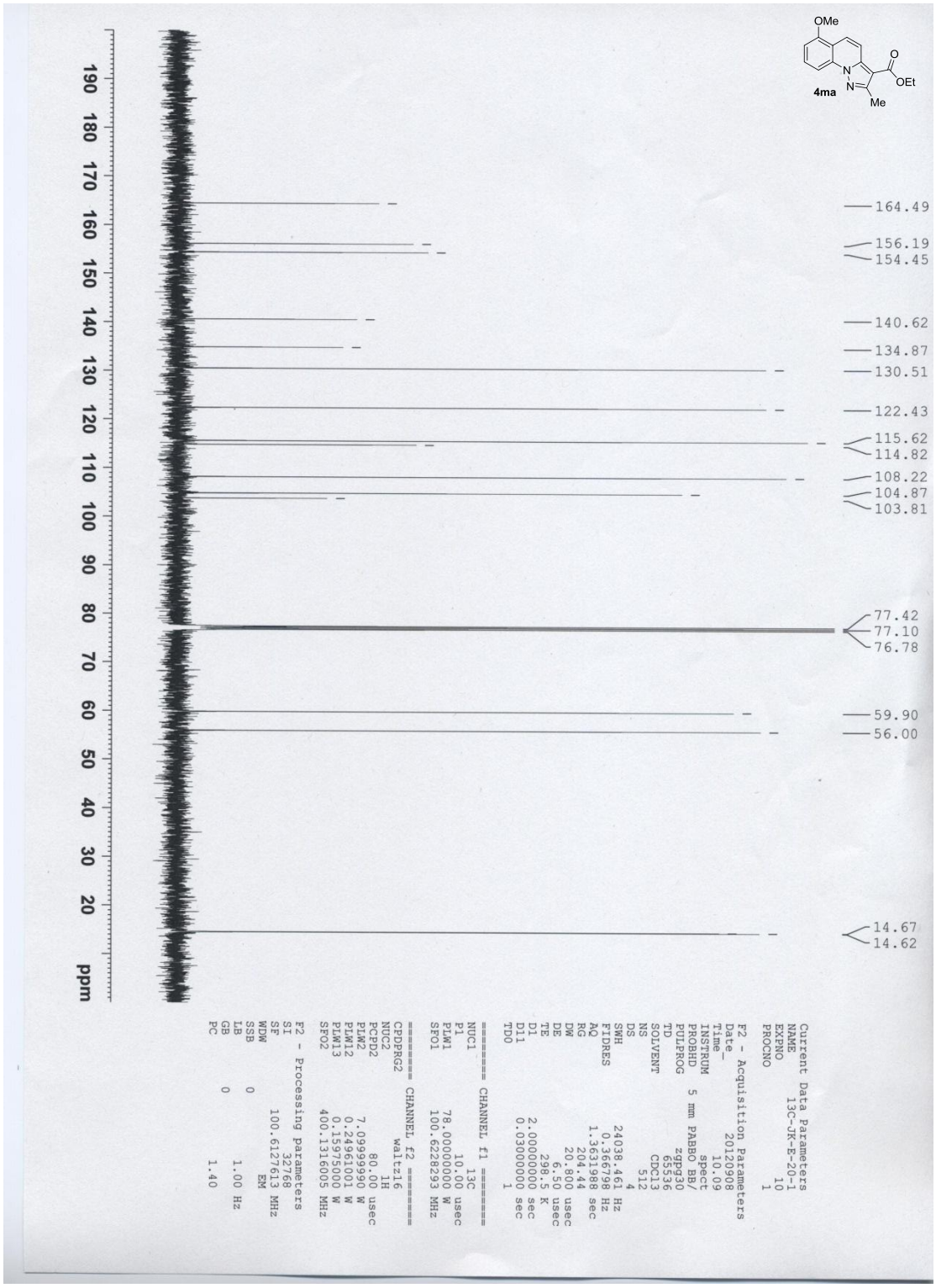

MeO
N
N
O
OEt
Me
4ka
ppm
Integral
-5.435
-5.459
-3.542
4.2547
4.7904
-0.048
-5.766
36.935
49.125
-0.068
-7.935
0.0029
0.1356
-0.000
15.659
17.353
8.70297
8.68084
8.12122
8.00074
7.99469
7.93018
7.91835
7.90708
7.82054
7.71506
7.69306
7.61980
7.59662
7.56639
7.55983
7.51908
7.39871
7.37749
7.26020
7.11303
7.10673
7.09113
7.08483
6.99619
6.86887
6.86231
6.84763
6.05195
6.04589
5.33624
5.33009
4.43838
4.42060
4.40282
4.38500
4.37103
4.36243
4.34470
4.20504
4.13002
4.11256
4.02664
3.97716
3.91530
3.90301
3.89377
3.84339
2.91748
2.75805
2.70902
2.60072
2.59502
2.58723
2.56389
2.53330
2.04295
1.75406
1.62482
1.60691
1.46804
1.45878
1.45020
1.43233
1.42556
1.40740
1.40019
1.38239
1.28995
1.27601
1.25807
1.24026
0.96755
0.89382
0.88229
0.07129
Current Data Parameters
NAME a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120420
Time 10.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 724.1
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm
JK-D-5-2

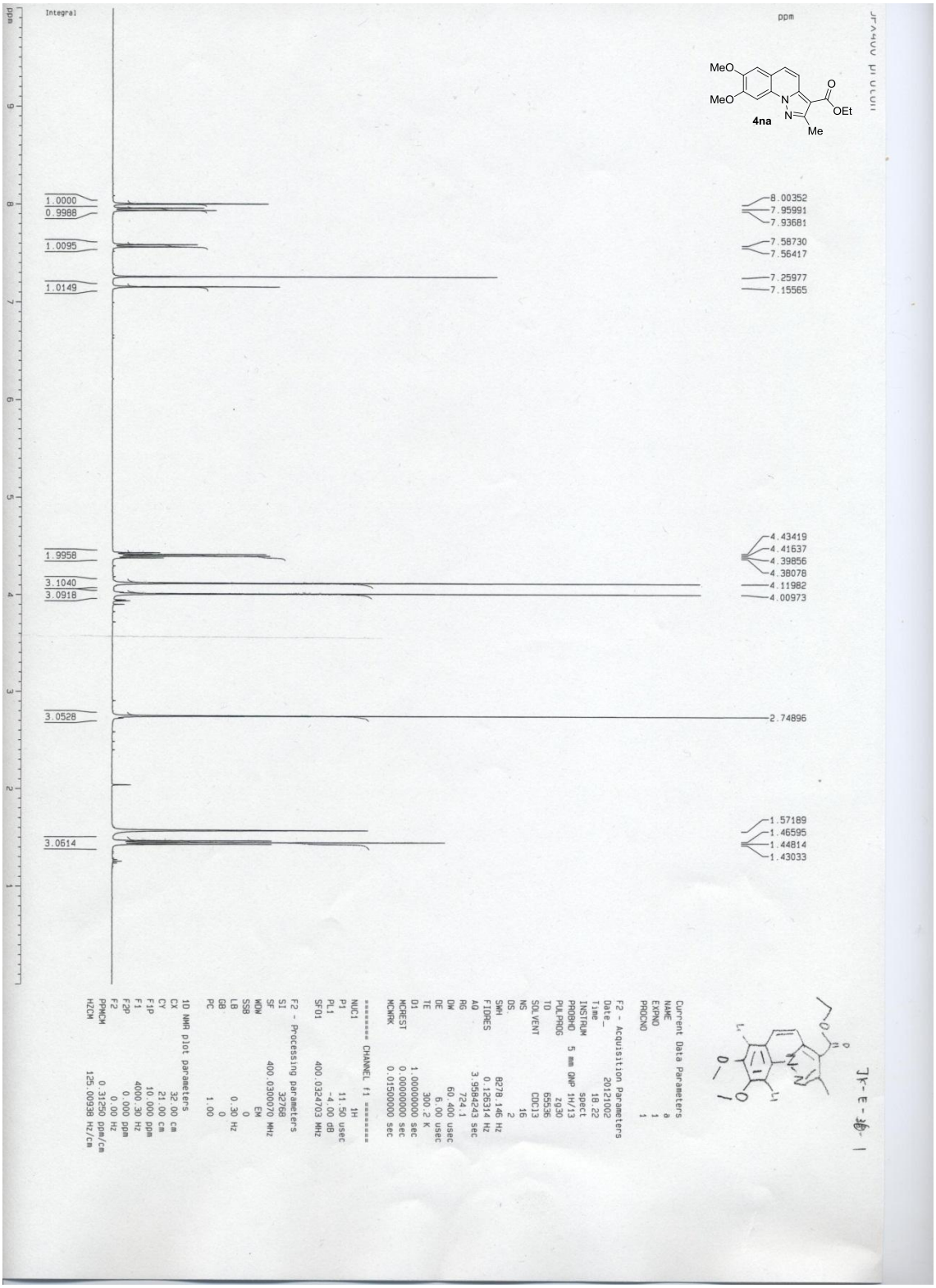


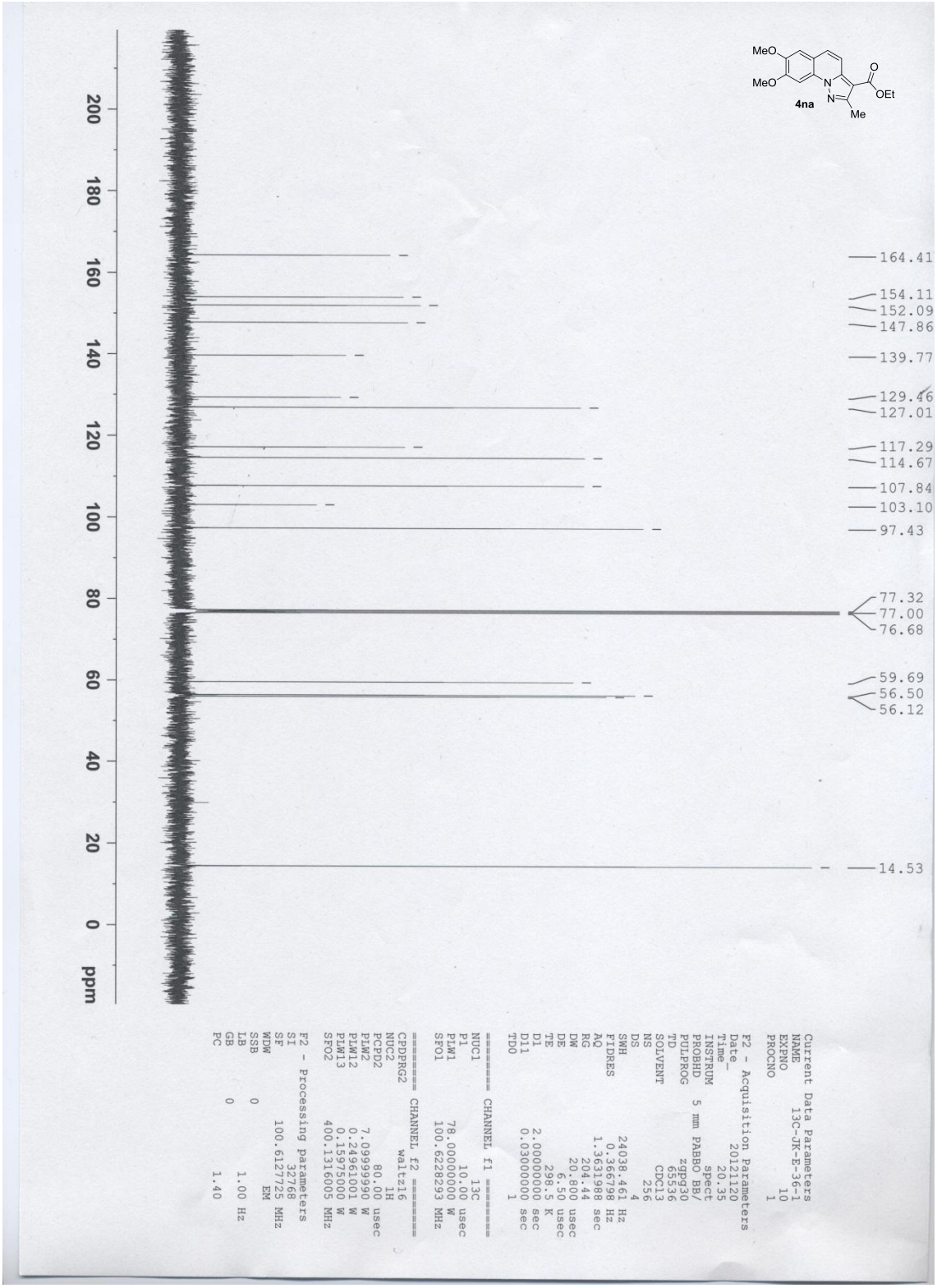

(Millions)
X : parts per Million : 1H
JK-10-1.1d-PROTON
MeO
O
OEt
N
N
Me
4la
8.4992
8.4761
8.0385
8.0154
7.5893
7.5662
7.3310
7.3089
7.2530
7.1826
7.1759
24.23
24.18
100
4.4260
4.4087
4.3904
4.3730
3.9567
3.9239
3.8863
48.85
76.47
2.7249
2.4907
2.4299
2.4039
70.22
1.5635
1.4574
1.4391
1.4218
97.03
-0.0085
18.7
103107

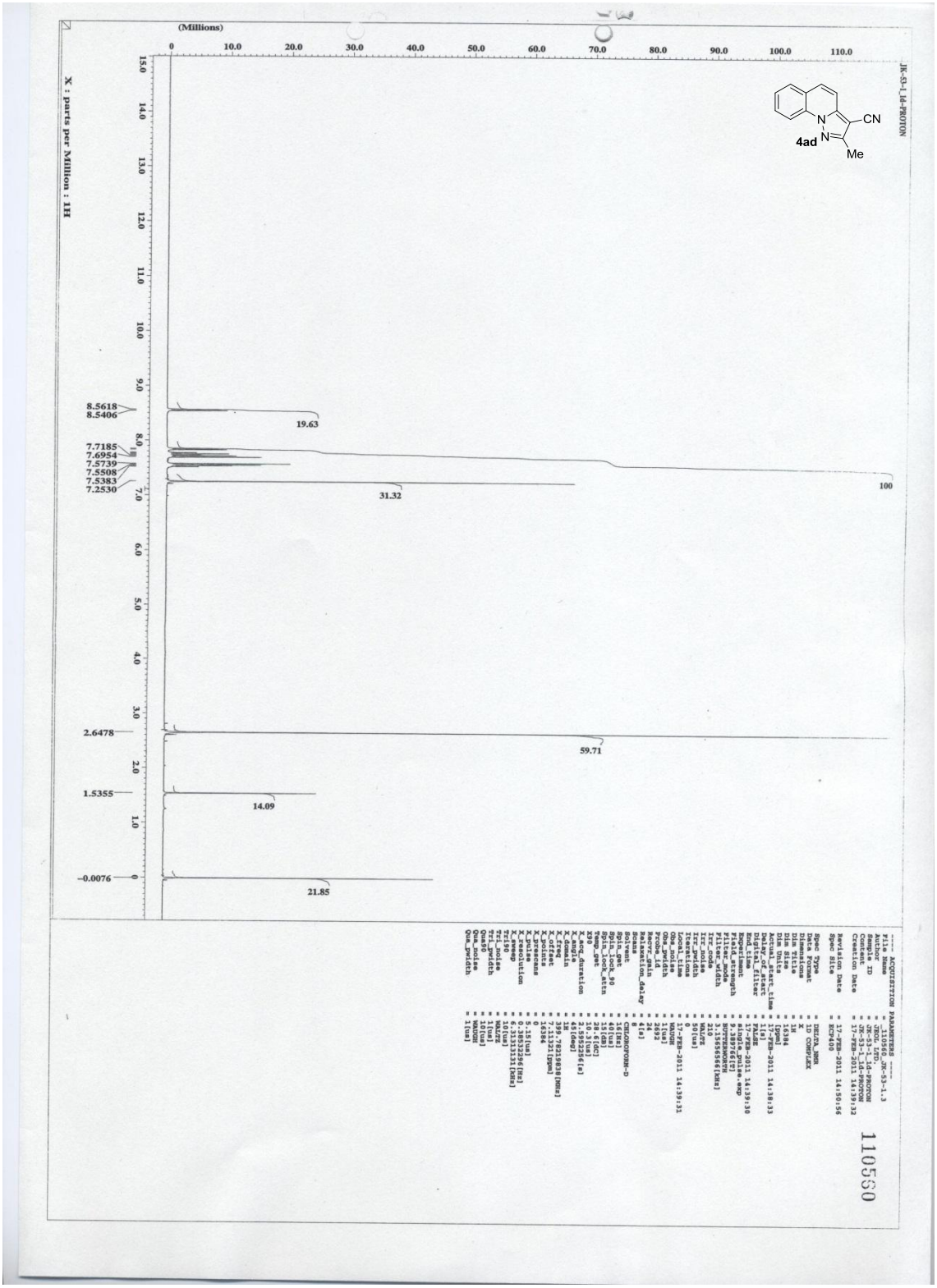


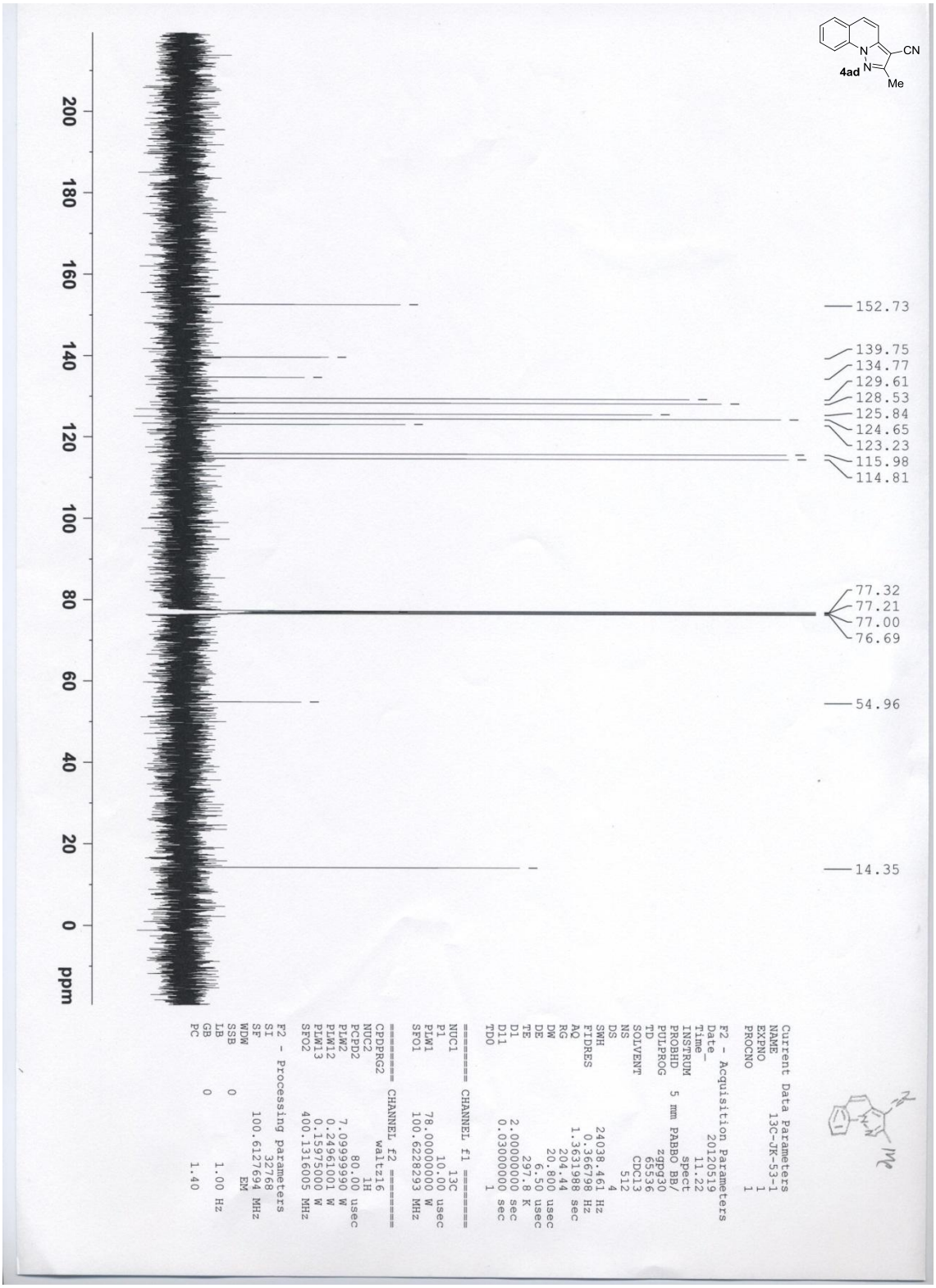

OMe
O
OEt
N
N
Me
4ma
ppm
Integral
0.9978
1.0088
0.9940
1.0176
1.0090
2.0587
3.1059
3.0738
3.2017
8.37456
8.20503
8.17088
8.14958
8.10189
8.07791
8.01330
7.98935
7.64807
7.62741
7.60659
7.51886
7.31002
7.25995
6.99600
6.90805
6.88789
6.86575
6.82440
5.59469
4.60304
4.43934
4.42149
4.40368
4.38587
4.20264
4.12335
4.04887
4.02434
3.96782
3.94510
3.84143
2.90237
2.74298
2.62395
2.58021
2.16822
2.04349
1.62535
1.60688
1.56203
1.46784
1.45003
1.43222
1.37437
1.36278
1.30792
1.28959
1.27182
1.25507
1.23383
1.20743
1.19555
0.89997
0.88354
0.86594
Current Data Parameters
NAME a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120907
Time 14.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 912.3
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.700 ppm
F1 3880.29 Hz
F2P -0.300 ppm
F2 -120.01 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm
JK-E-20-1

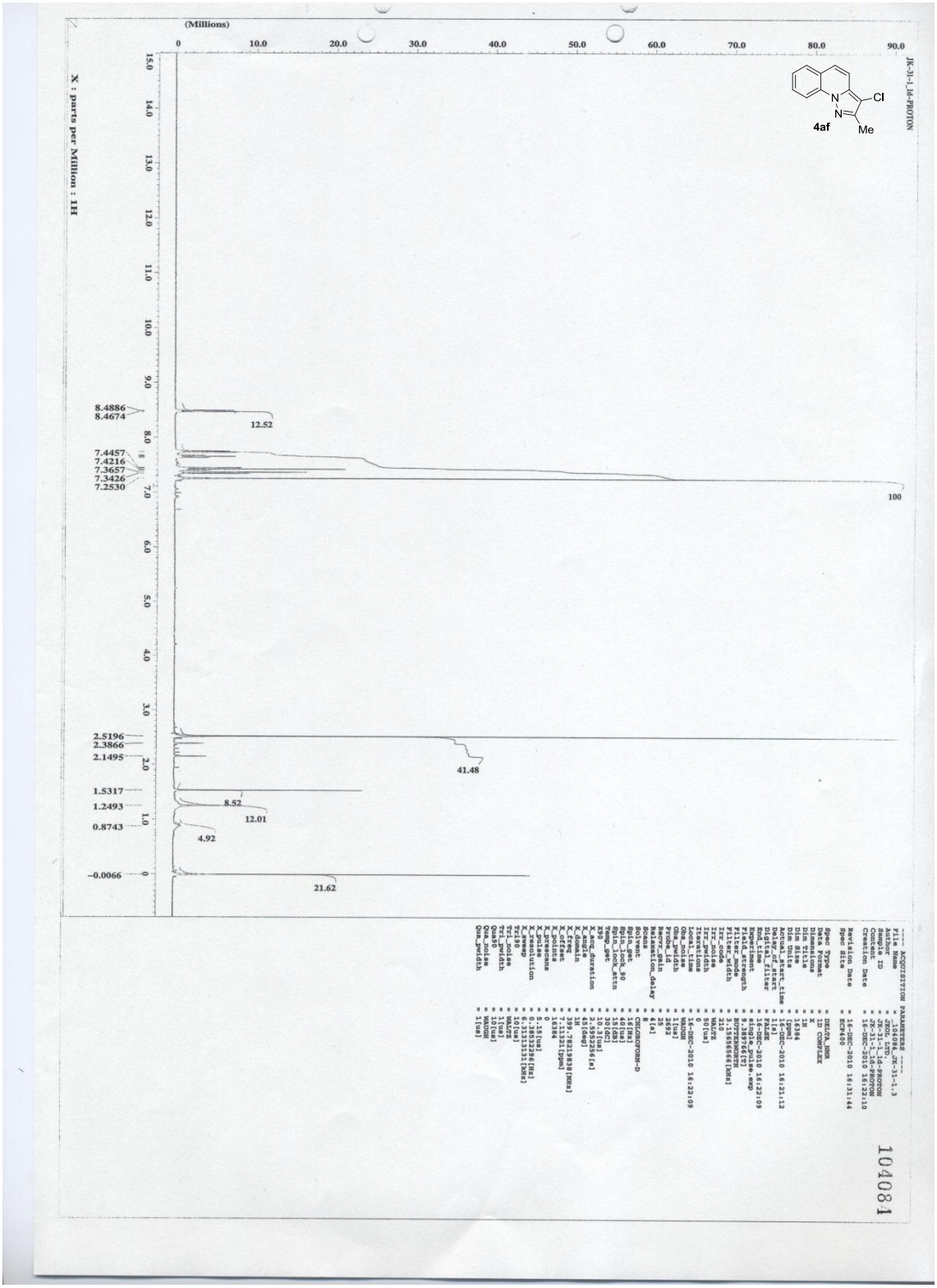

OMe
O
OEt
N
N
Me
4ma
164.49
156.19
154.45
140.62
134.87
130.51
122.43
115.62
114.82
108.22
104.87
103.81
77.42
77.10
76.78
59.90
56.00
14.67
14.62
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME 13C-JK-E-20-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120908
Time_ 10.09
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 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127613 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

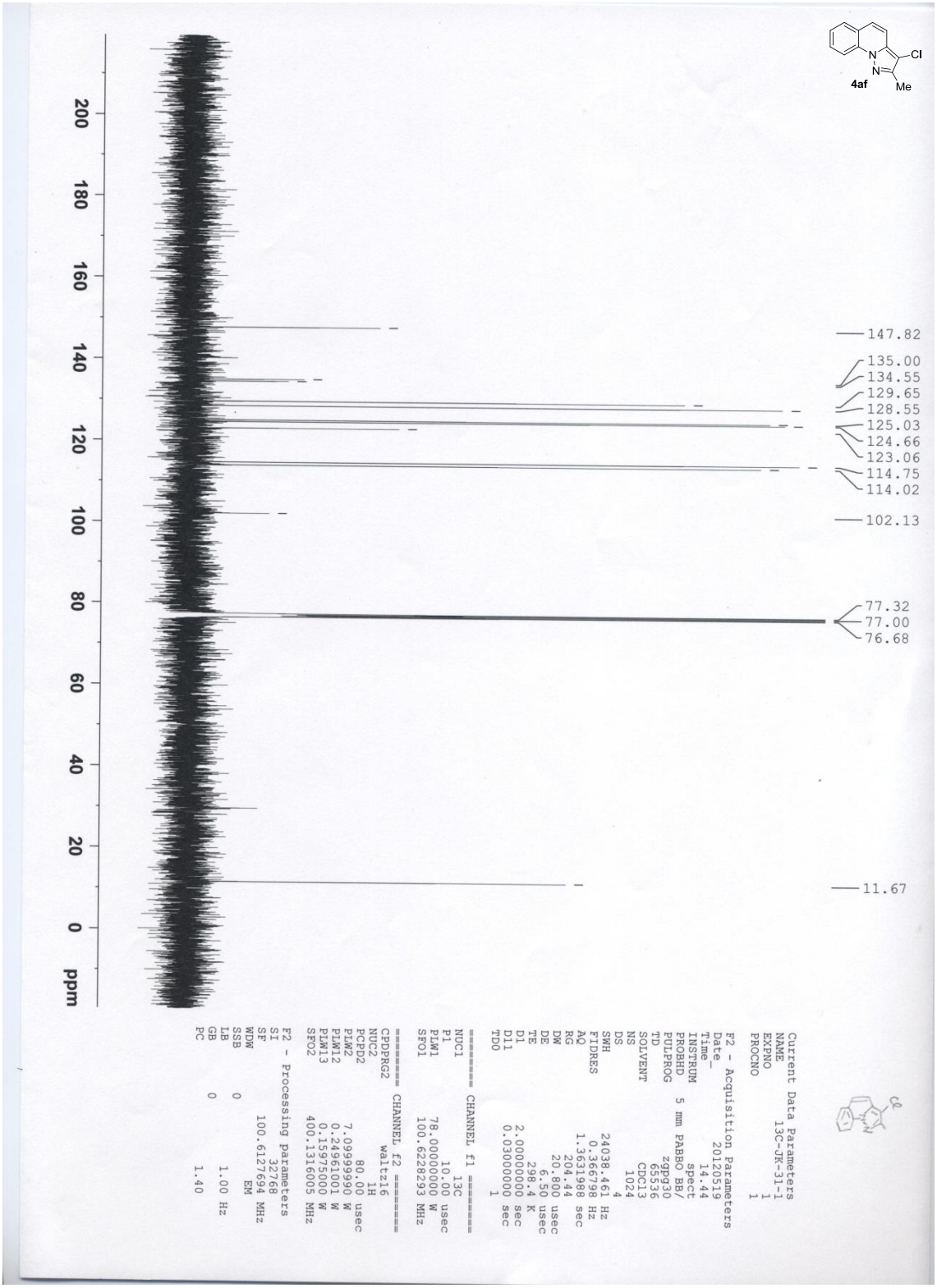

4na
MeO
MeO
N
N
O
OEt
Me
ppm
Integral
1.0000
0.9988
1.0095
1.0149
1.9958
3.1040
3.0918
3.0528
3.0614
8.00352
7.95991
7.93681
7.58730
7.56417
7.25977
7.15565
4.43419
4.41637
4.39856
4.38078
4.11982
4.00973
2.74896
1.57189
1.46595
1.44814
1.43033
Current Data Parameters
NAME a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121002
Time 18 22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 724.1
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz
F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

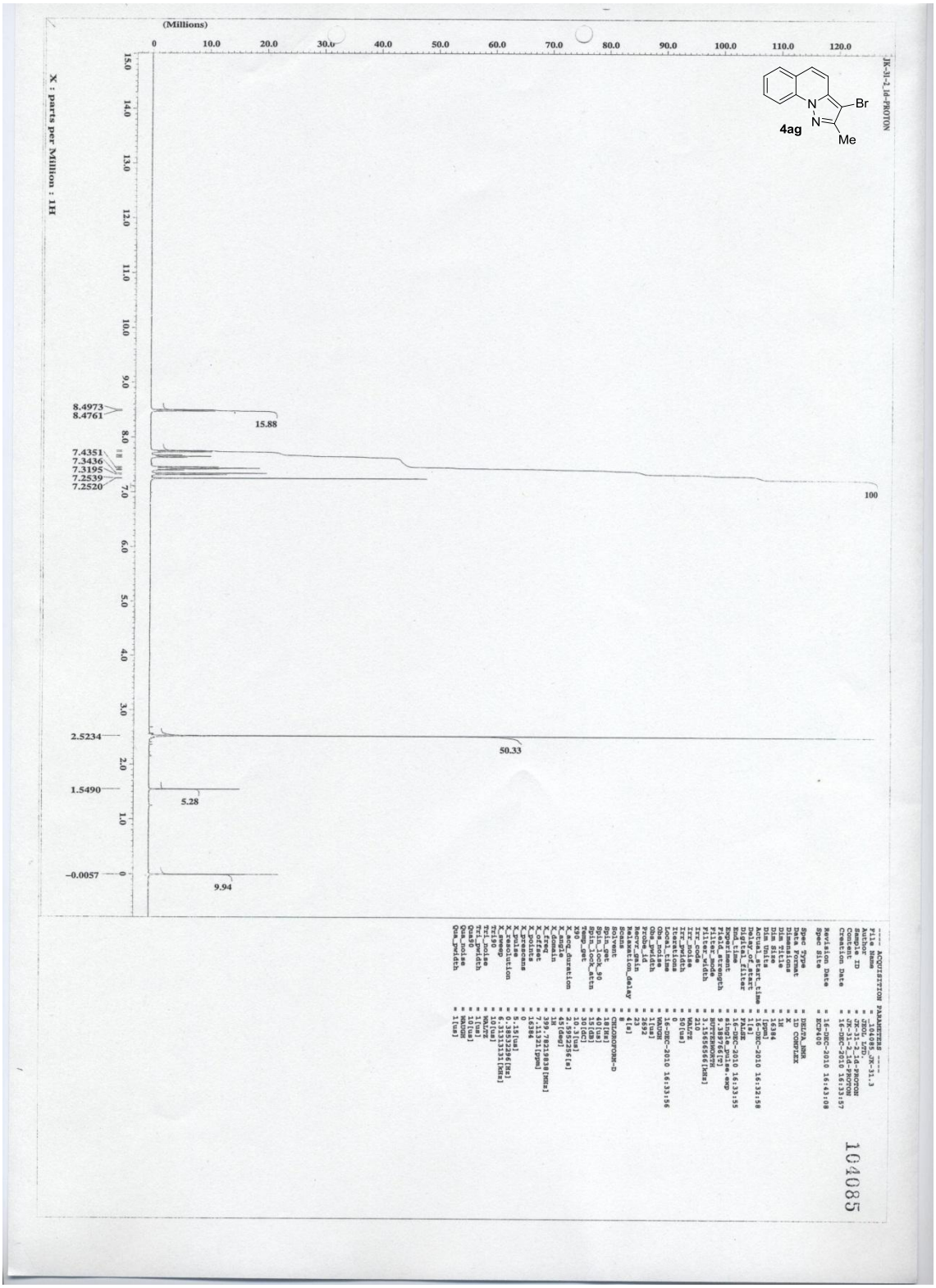

MeO
MeO
O
OEt
N
N
Me
4na
164.41
154.11
152.09
147.86
139.77
129.46
127.01
117.29
114.67
107.84
103.10
97.43
77.32
77.00
76.68
59.69
56.50
56.12
14.53
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-E-36-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121120
Time_ 20.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.622829 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

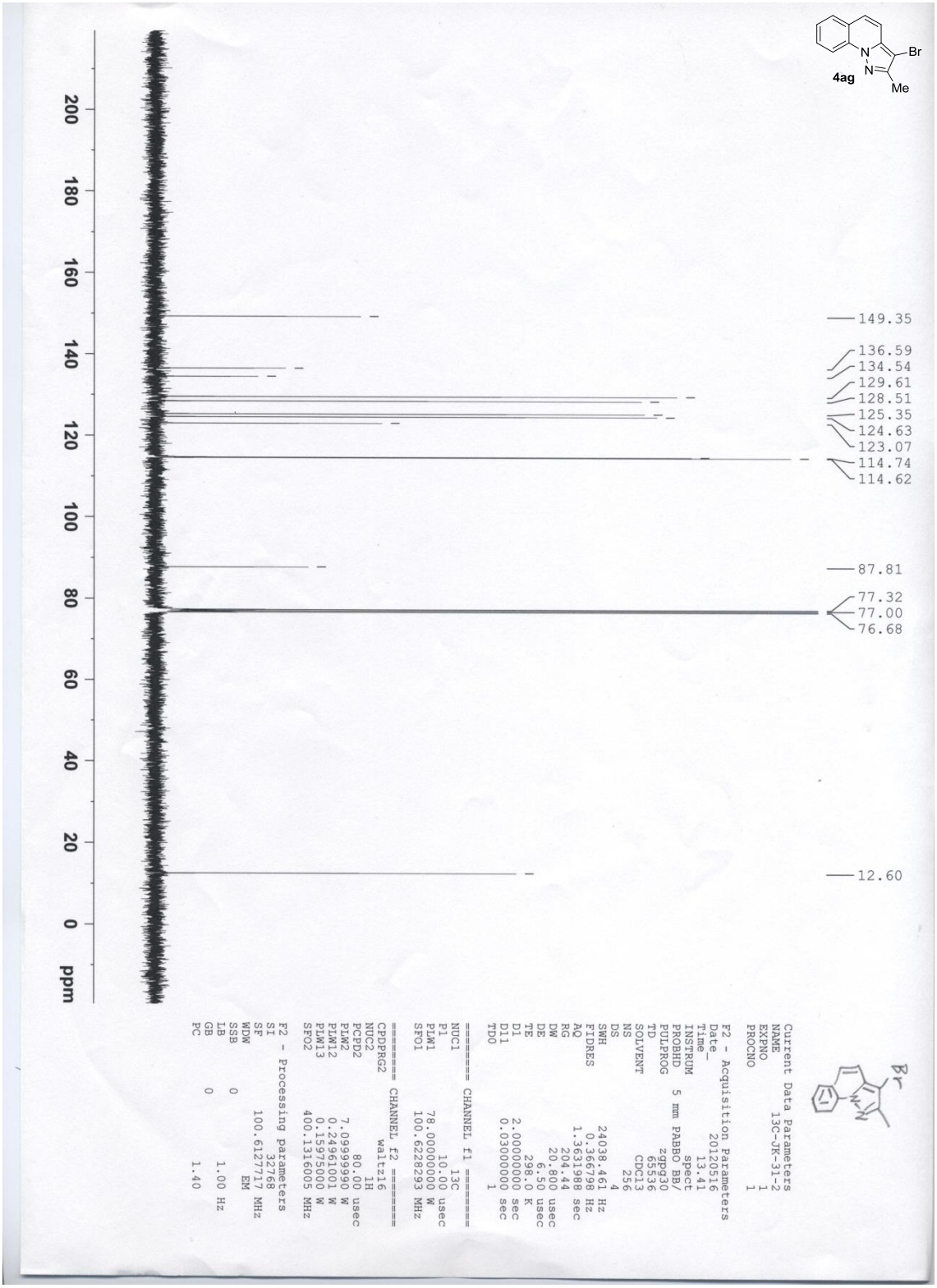

(Millions)
X : parts per Million : 1H
JK-53-1-1d-PROTON
4ad
CN
N
N
Me
8.5618
8.5406
7.7185
7.6954
7.5739
7.5508
7.5383
7.2530
2.6478
1.5355
-0.0076
19.63
31.32
100
59.71
14.09
21.85
110560

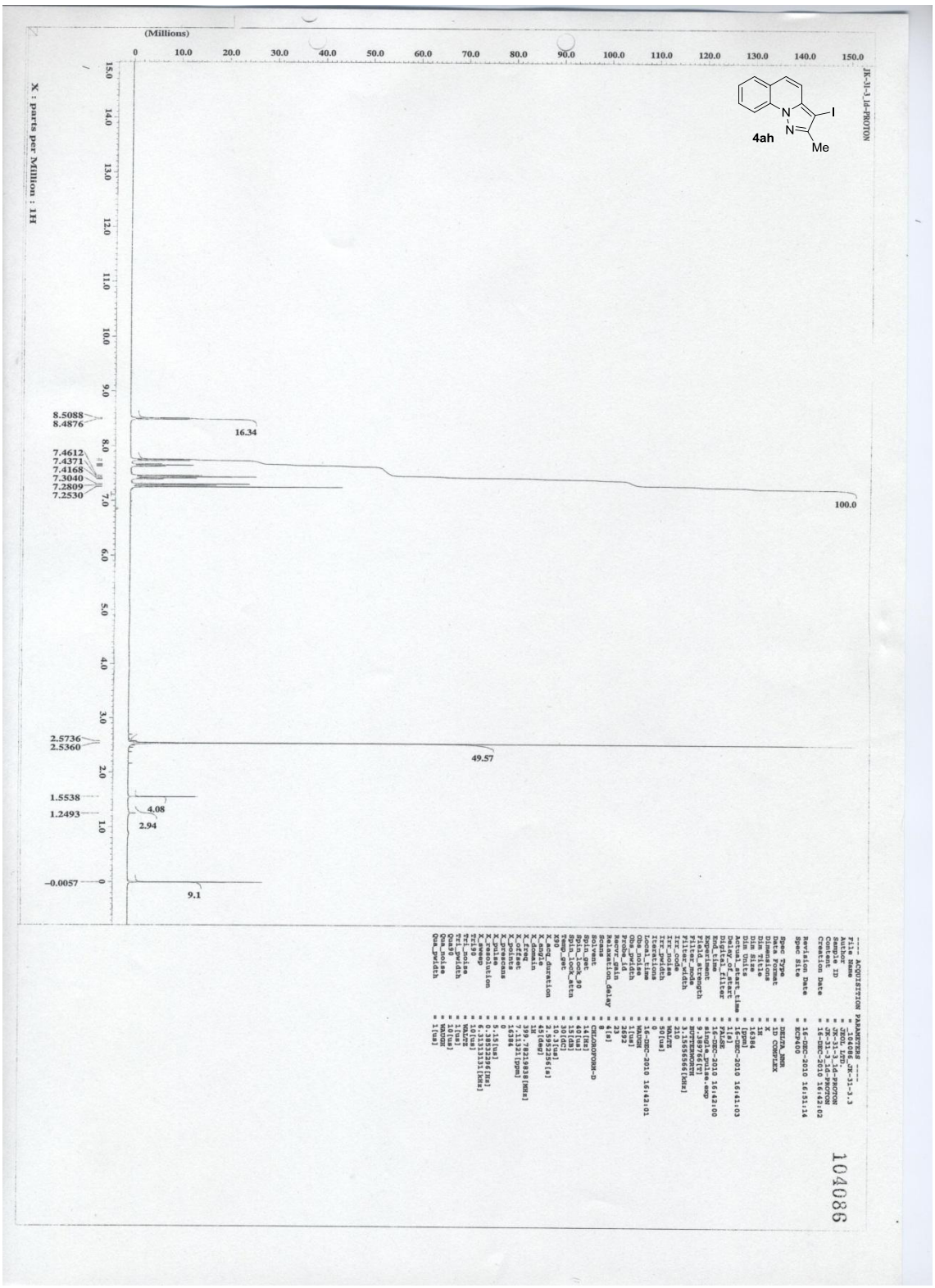


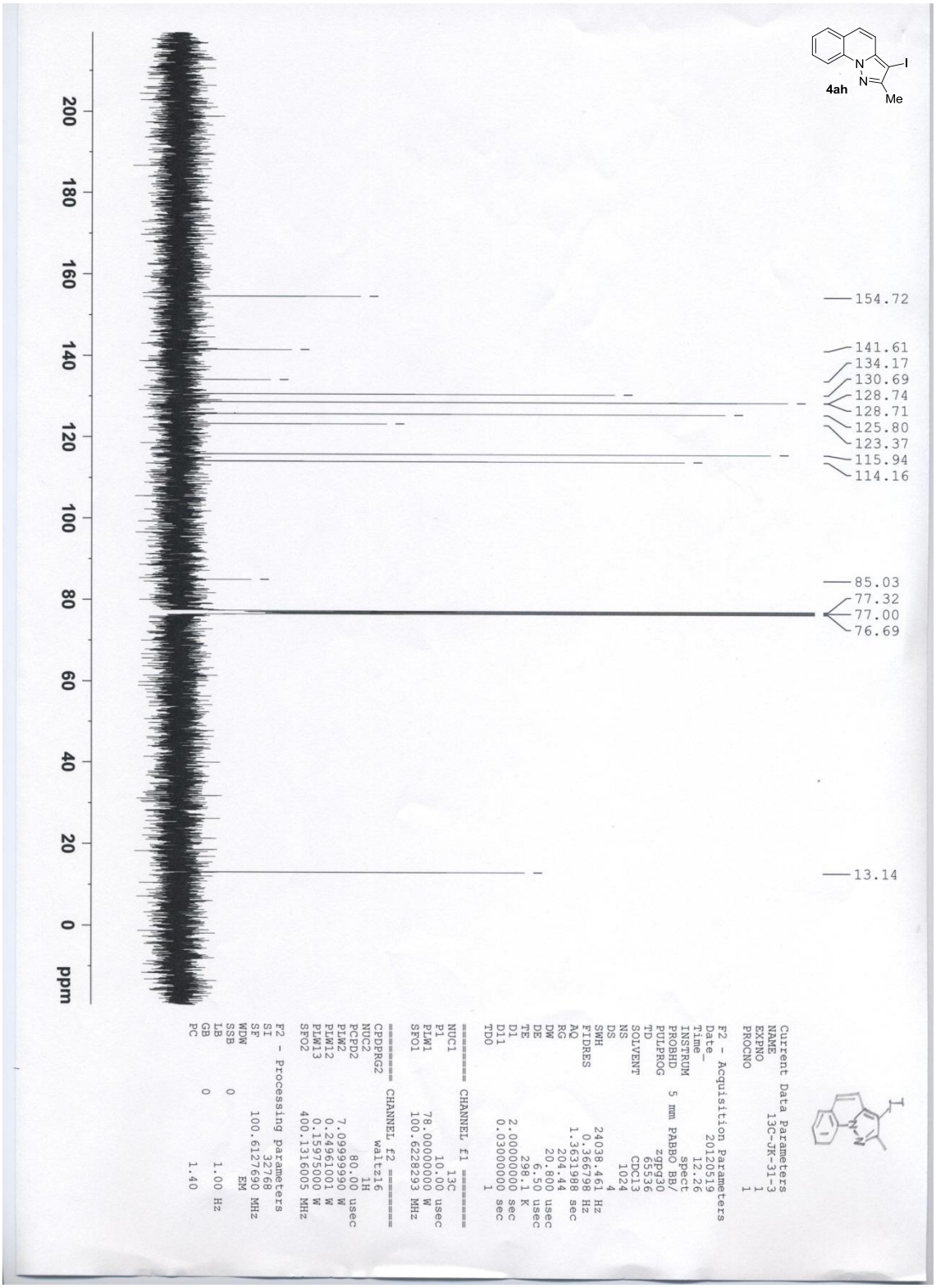

(Millions)
X : parts per Million : 1H
JK-31-1_1H-PROTON
4af
Cl
Me
8.4886
8.4674
7.4457
7.4216
7.3657
7.3426
7.2530
2.5196
2.3866
2.1495
1.5317
1.2493
0.8743
−0.0066
12.52
100
41.48
8.52
12.01
4.92
21.62
104081

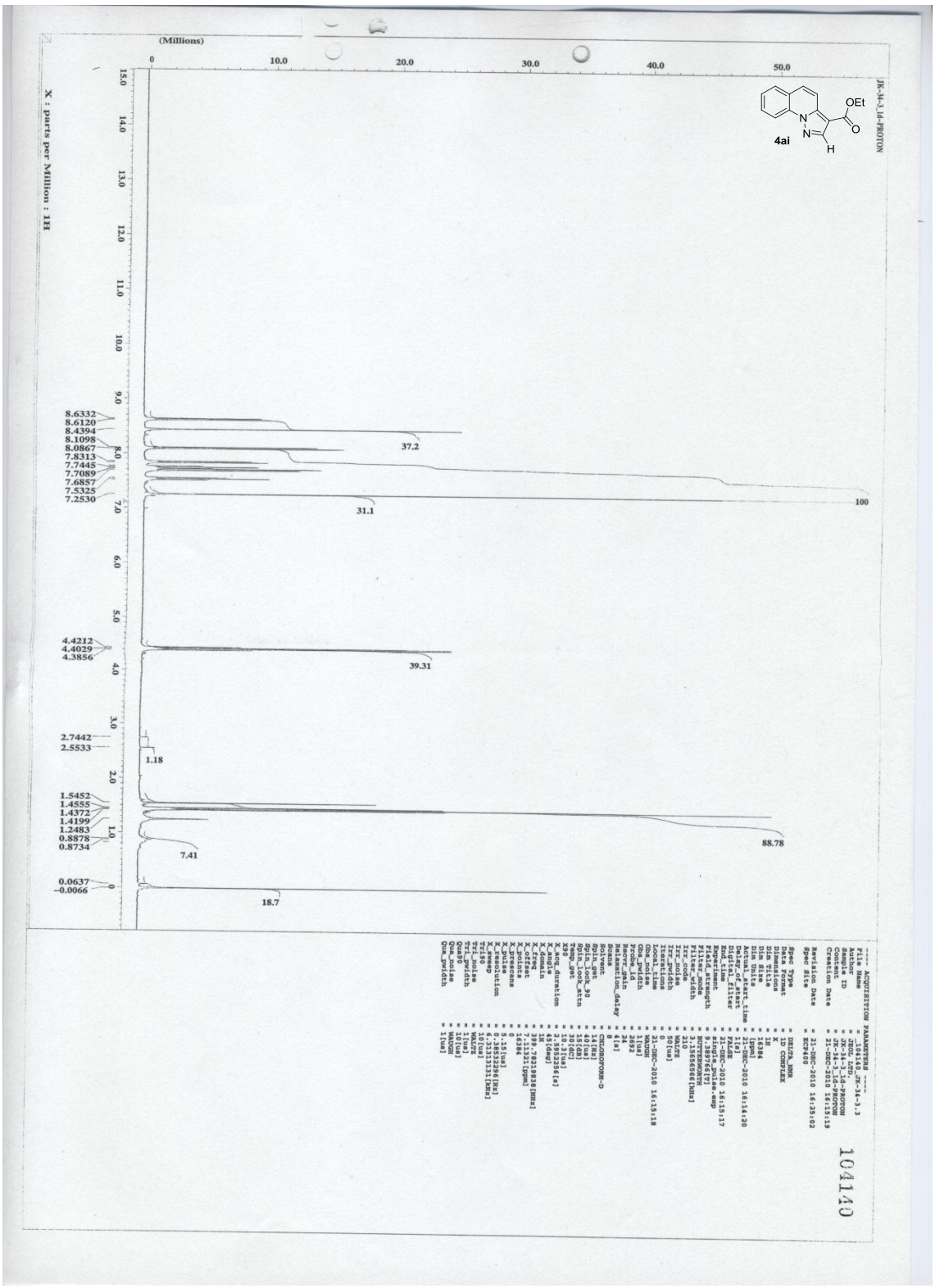


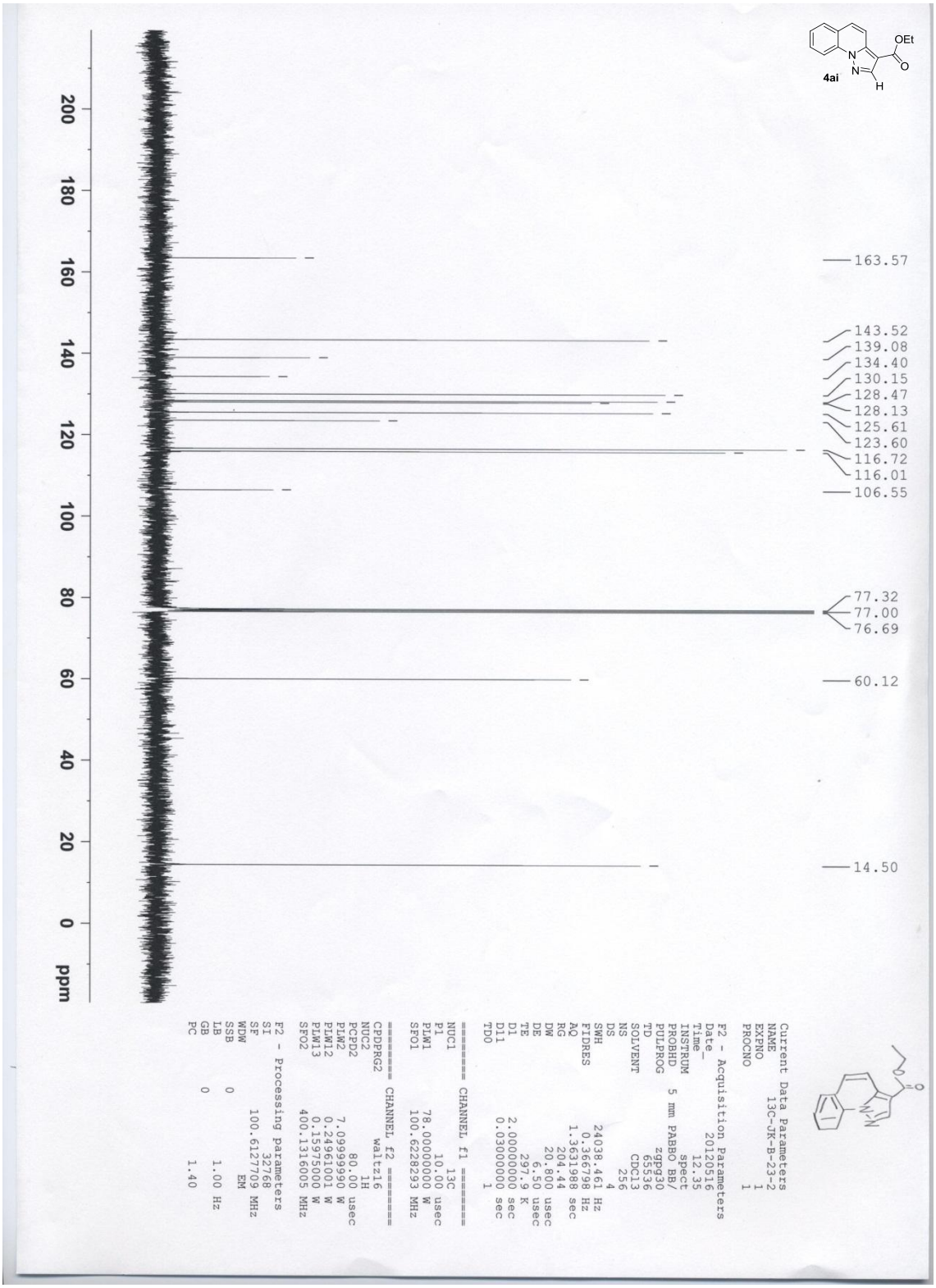

(Millions)
X : parts per Million : 1H
JK-31-2_1d-PROTON
4ag
Br
N
N
Me
8.4973
8.4761
15.88
7.4351
7.3436
7.3195
7.2539
7.2520
100
2.5234
50.33
1.5490
5.28
-0.0057
9.94
104085

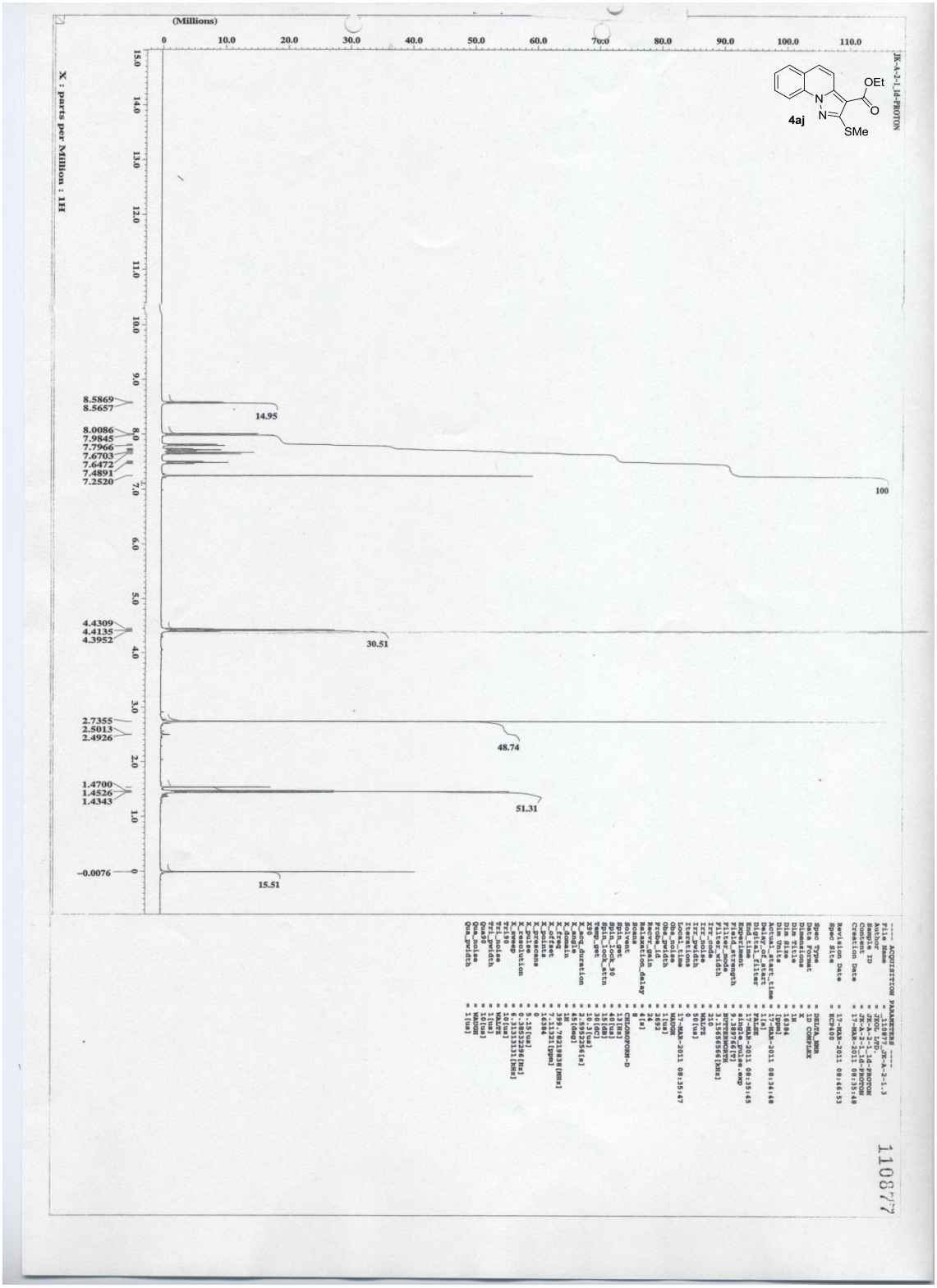


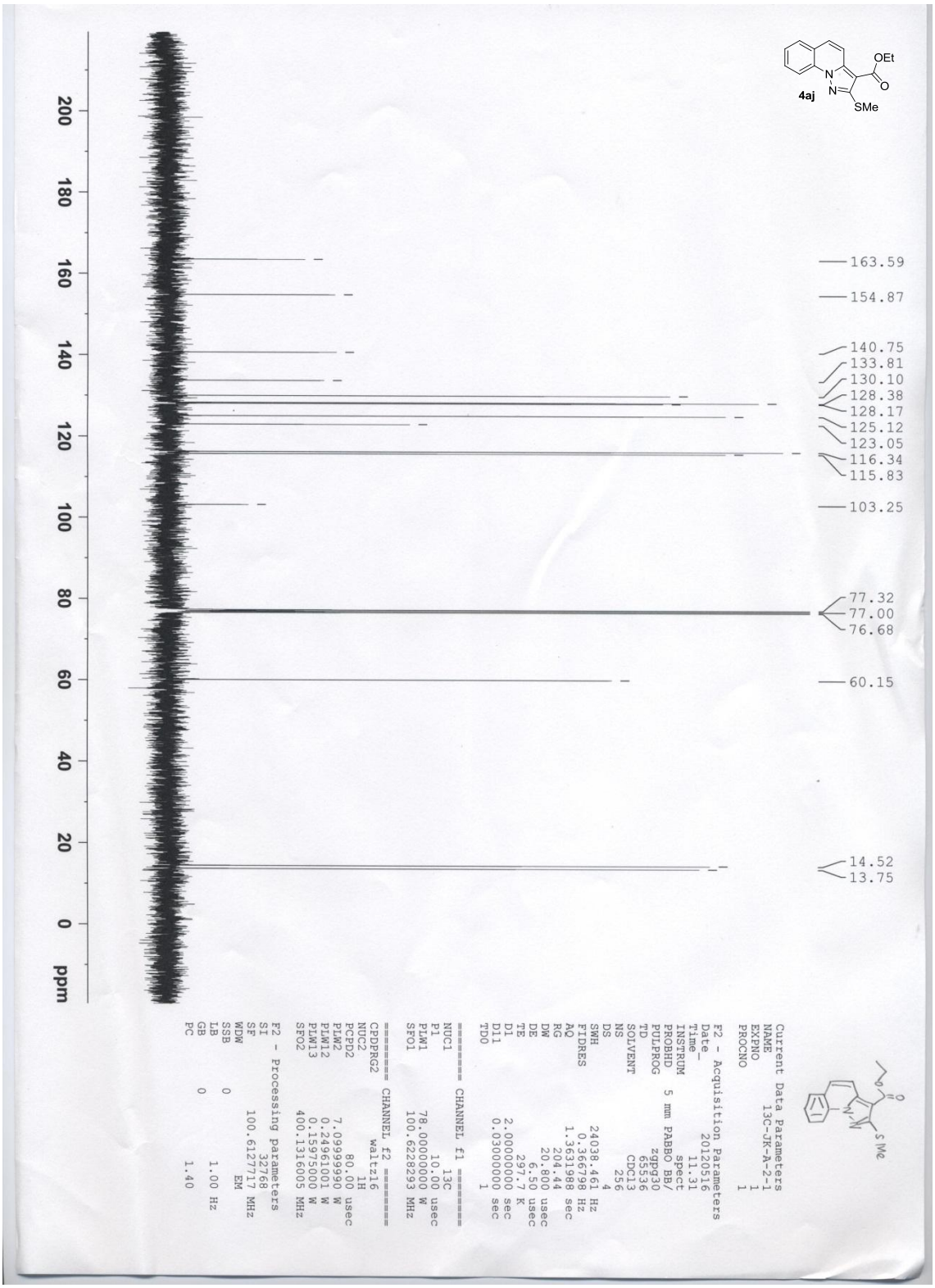

4ah
Me
I
N
N
JK-31-1_16-PROTON
X : parts per Million : 1H
(Millions)
8.5088
8.4876
7.4612
7.4371
7.4168
7.3040
7.2809
7.2530
2.5736
2.5360
1.5538
1.2493
-0.0057
16.34
100.0
49.57
4.08
2.94
9.1
104086

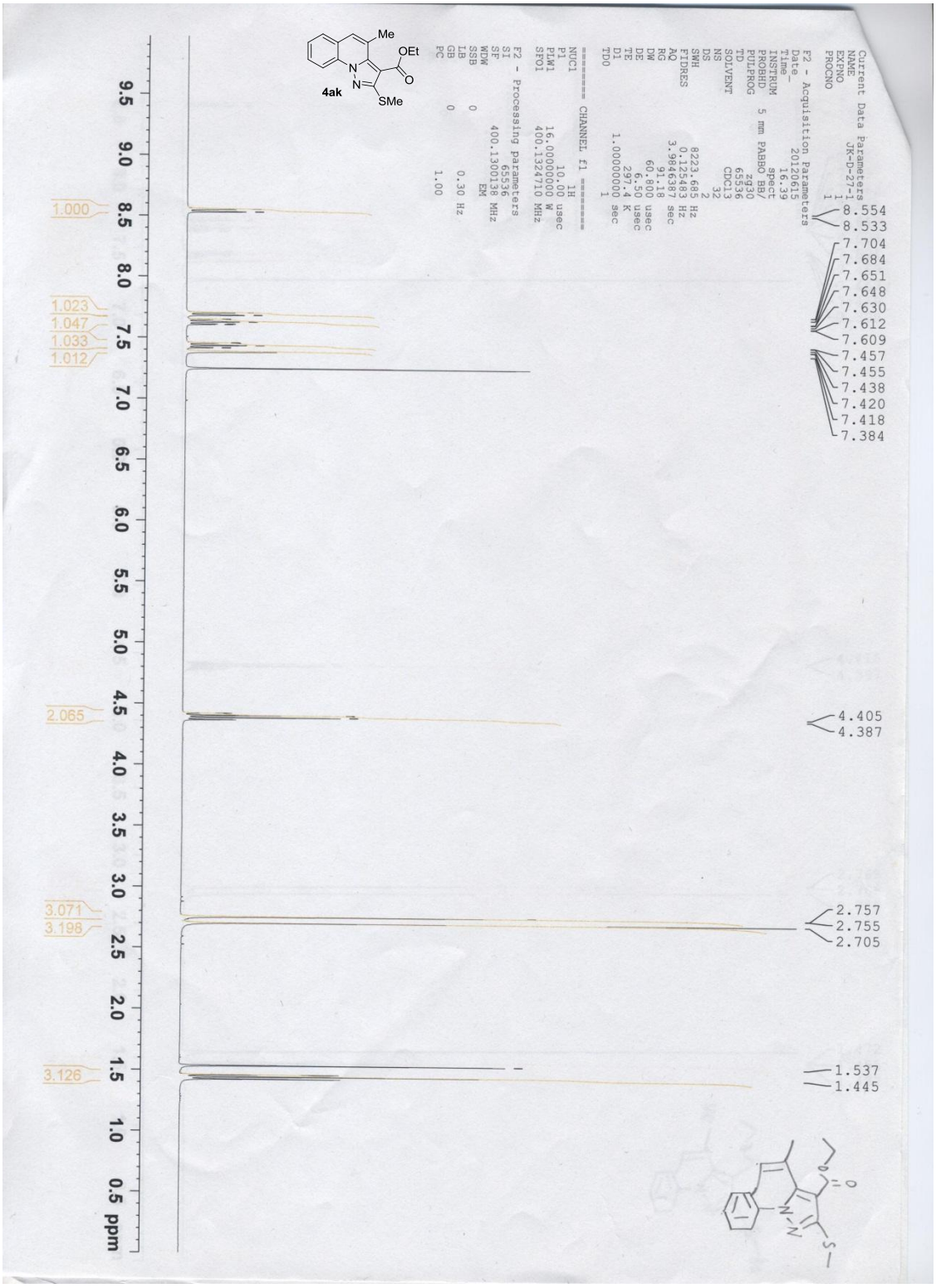

4ah
I
N
N
Me
154.72
141.61
134.17
130.69
128.74
128.71
125.80
123.37
115.94
114.16
85.03
77.32
77.00
76.69
13.14
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-31-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120519
Time_ 12.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

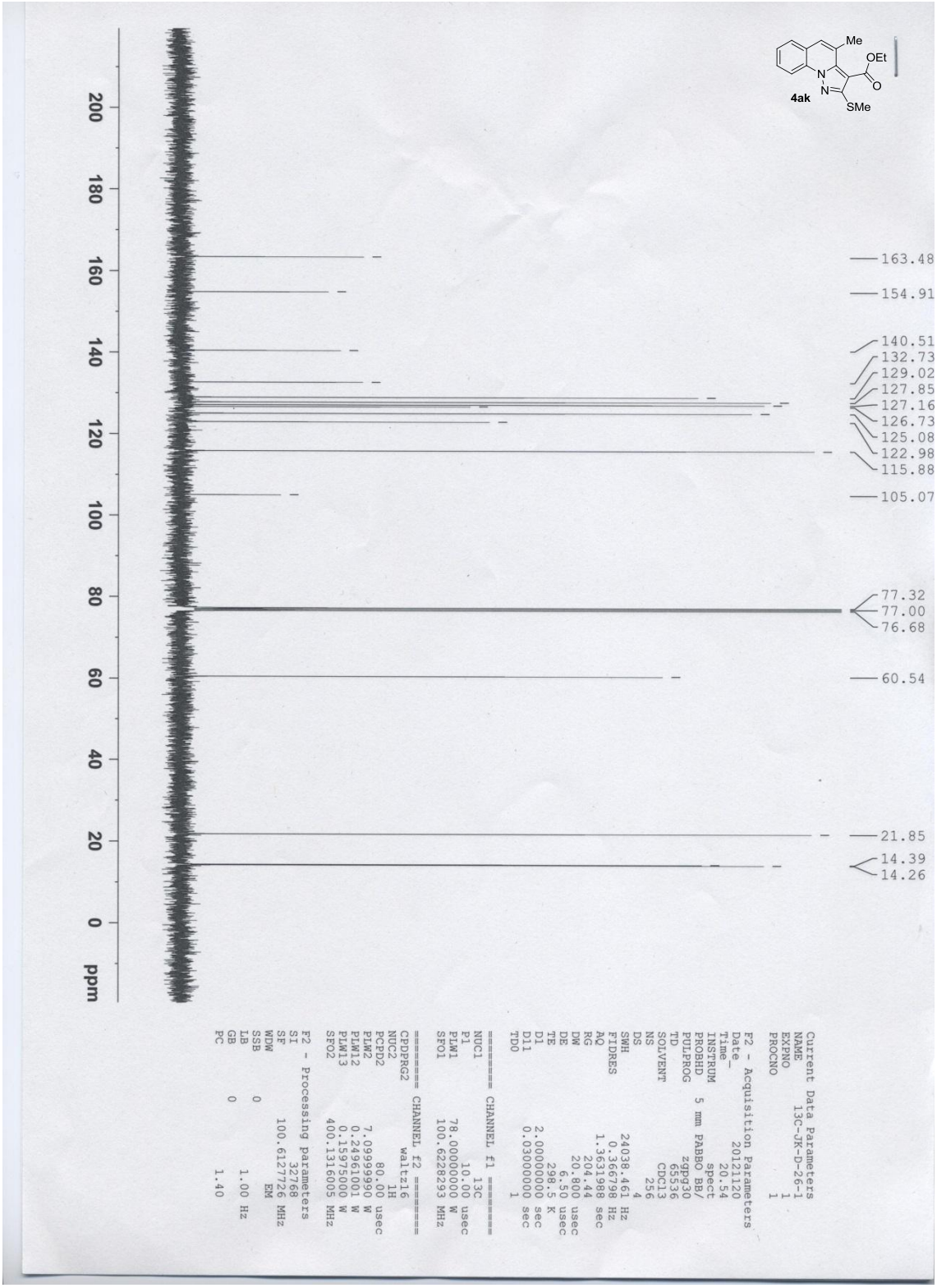


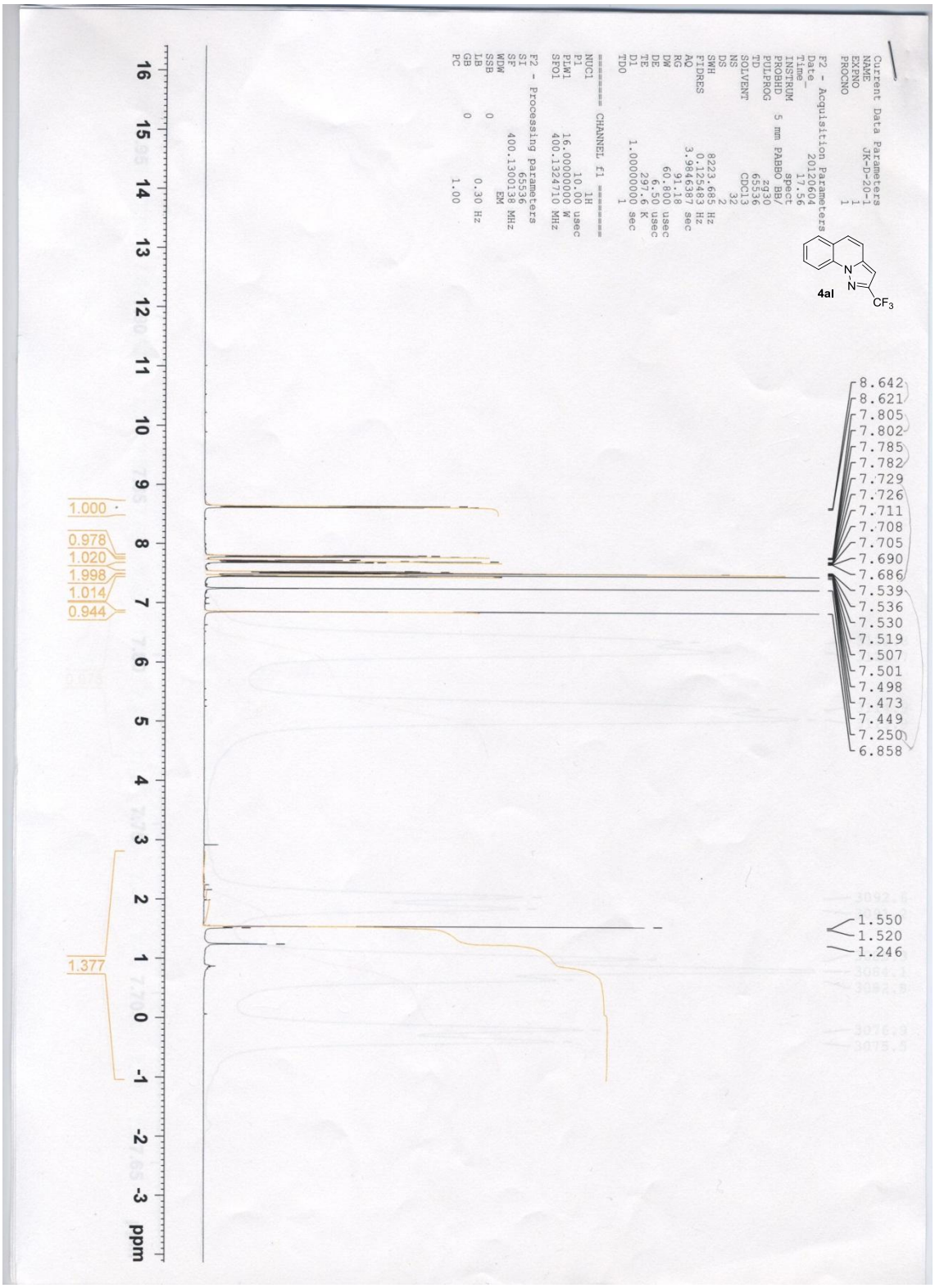


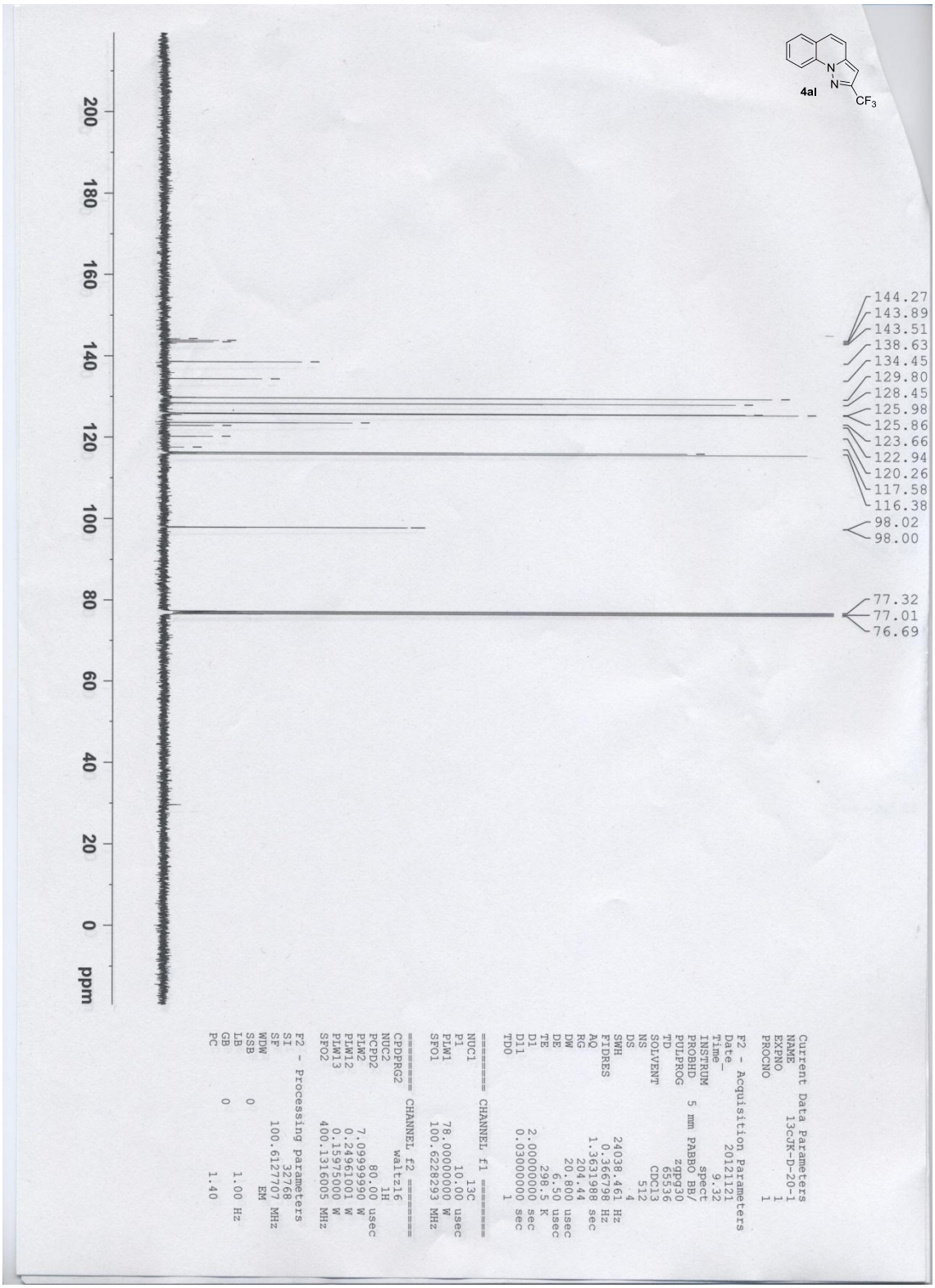


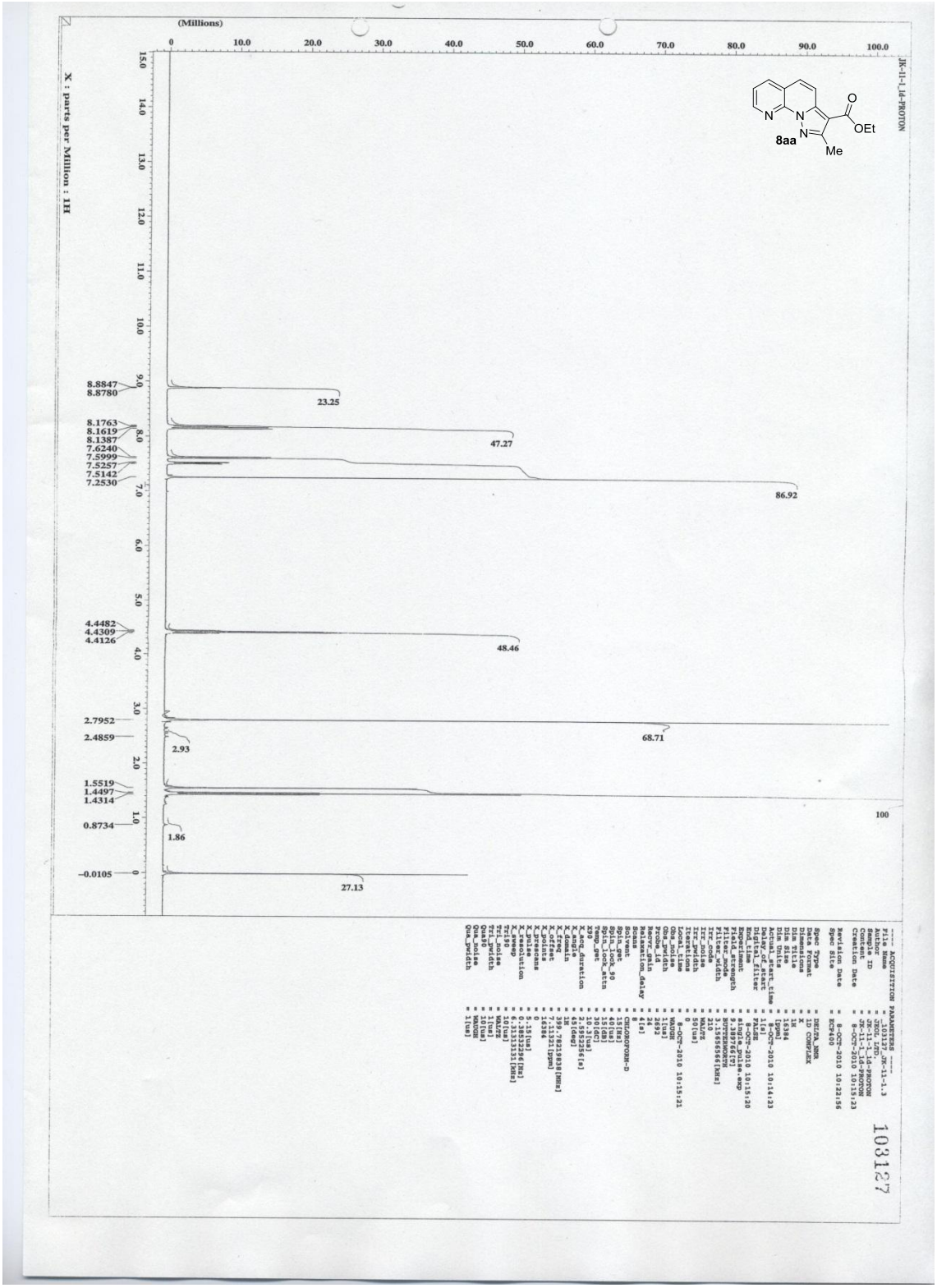

4aj
OEt
O
N
N
SMe
163.59
154.87
140.75
133.81
130.10
128.38
128.17
125.12
123.05
116.34
115.83
103.25
77.32
77.00
76.68
60.15
14.52
13.75
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-A-2-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120516
Time_ 11.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

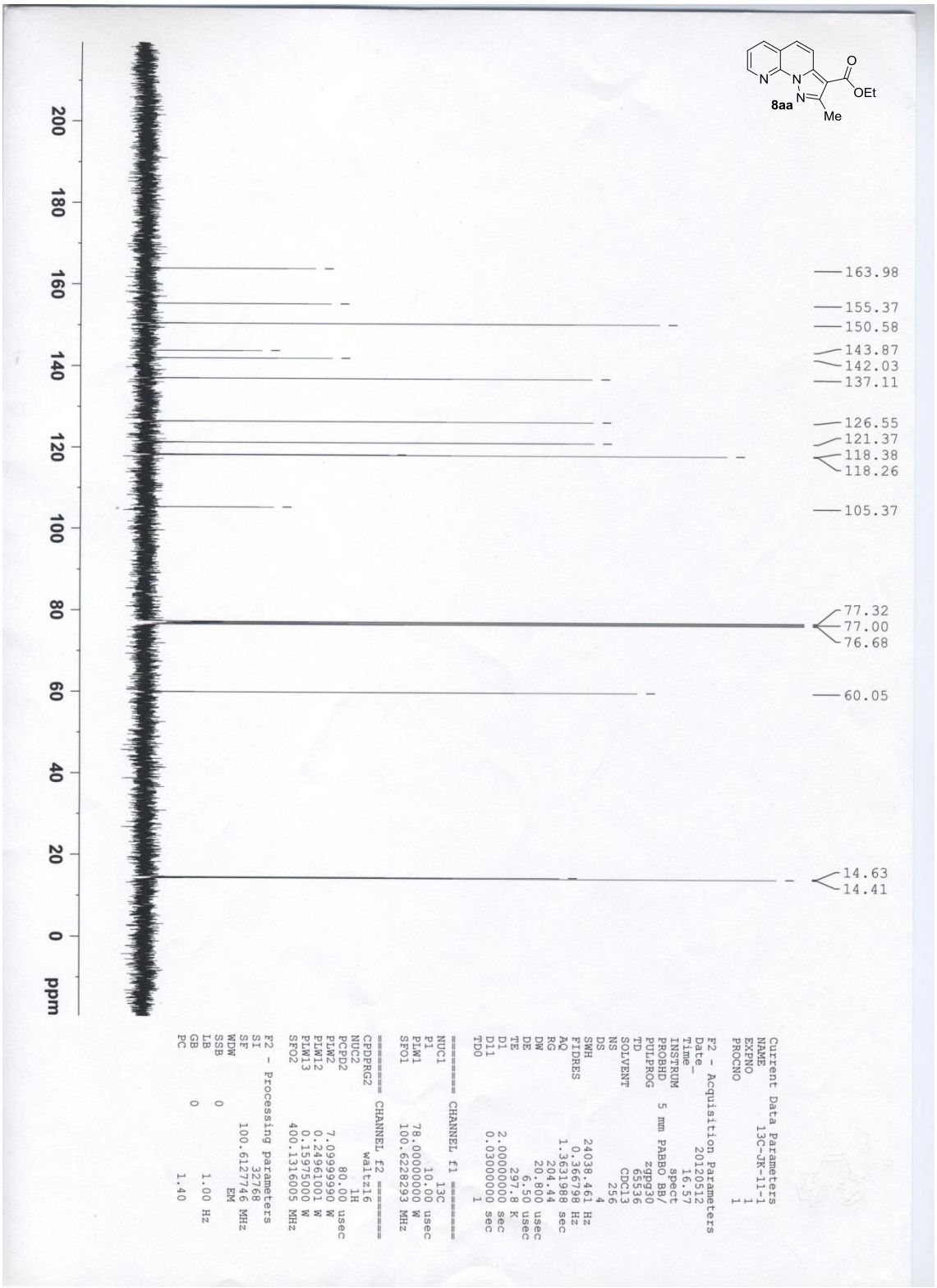

Me
OEt
O
N
N
SMe
4ak
Current Data Parameters
NAME JK-D-27-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120615
Time 16.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.18
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB
PC 1.00
8.554
8.533
7.704
7.684
7.651
7.648
7.630
7.612
7.609
7.457
7.455
7.438
7.420
7.418
7.384
4.405
4.387
2.757
2.755
2.705
1.537
1.445
1.000
1.023
1.047
1.033
1.012
2.065
3.071
3.198
3.126
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

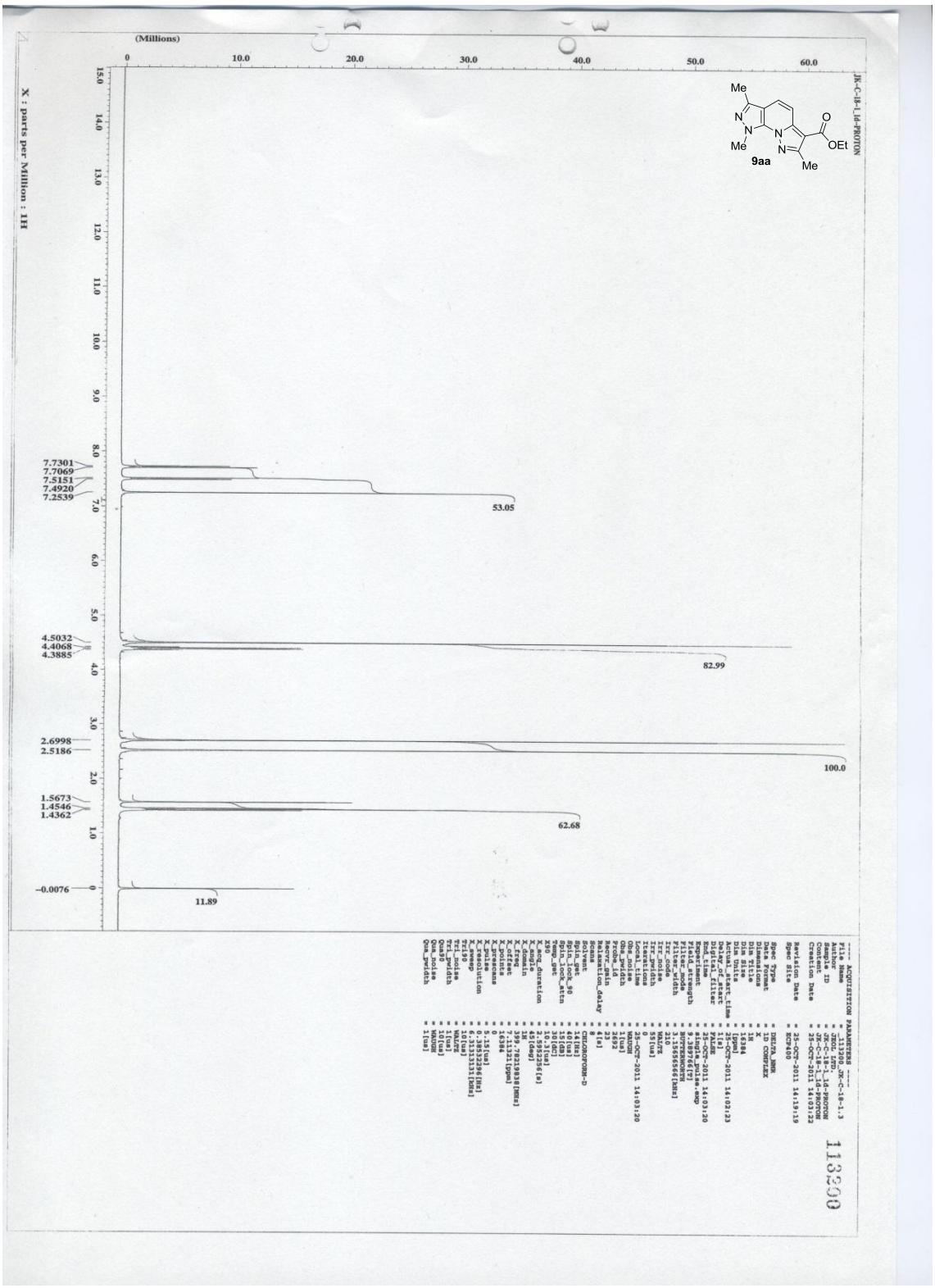


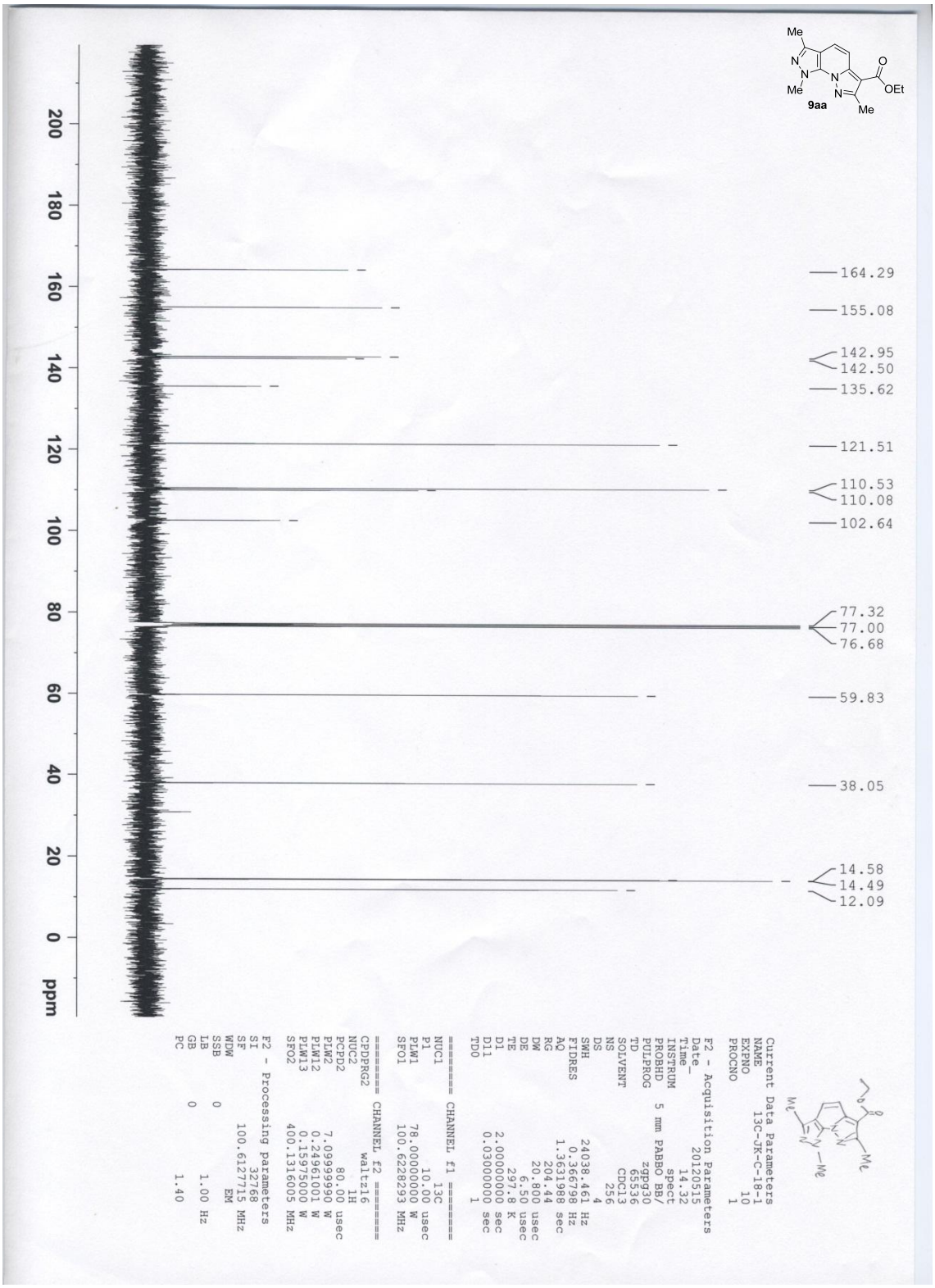

4al
8.642
8.621
7.805
7.802
7.785
7.782
7.729
7.726
7.711
7.708
7.705
7.690
7.686
7.539
7.536
7.530
7.519
7.507
7.501
7.498
7.473
7.449
7.250
6.858
1.550
1.520
1.246
1.000
0.978
1.020
1.998
1.014
0.944
1.377
ppm

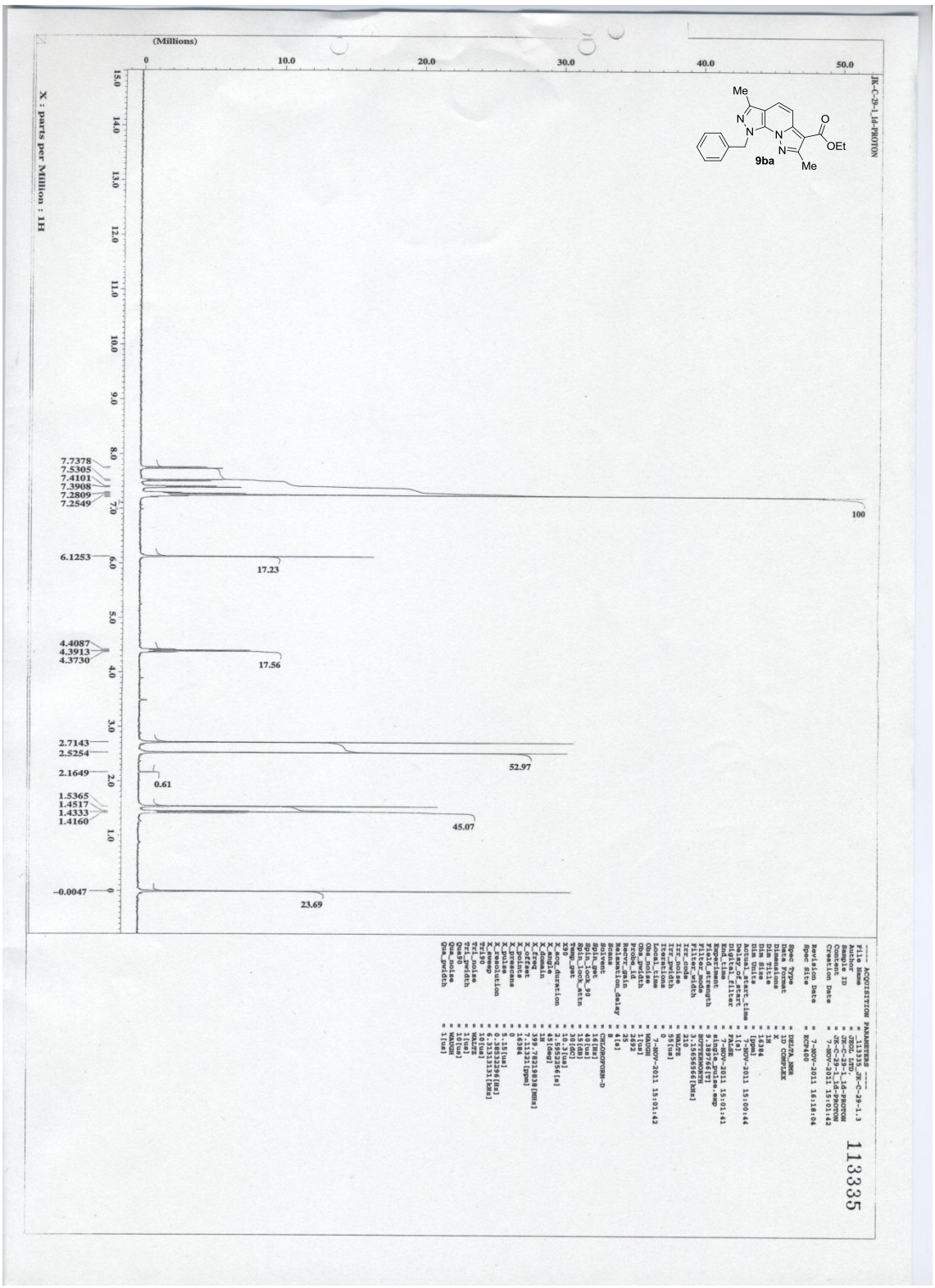

4al
CF3
144.27
143.89
143.51
138.63
134.45
129.80
128.45
125.98
125.86
123.66
122.94
120.26
117.58
116.38
98.02
98.00
77.32
77.01
76.69
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13cJK-D-20-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121121
Time 9.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

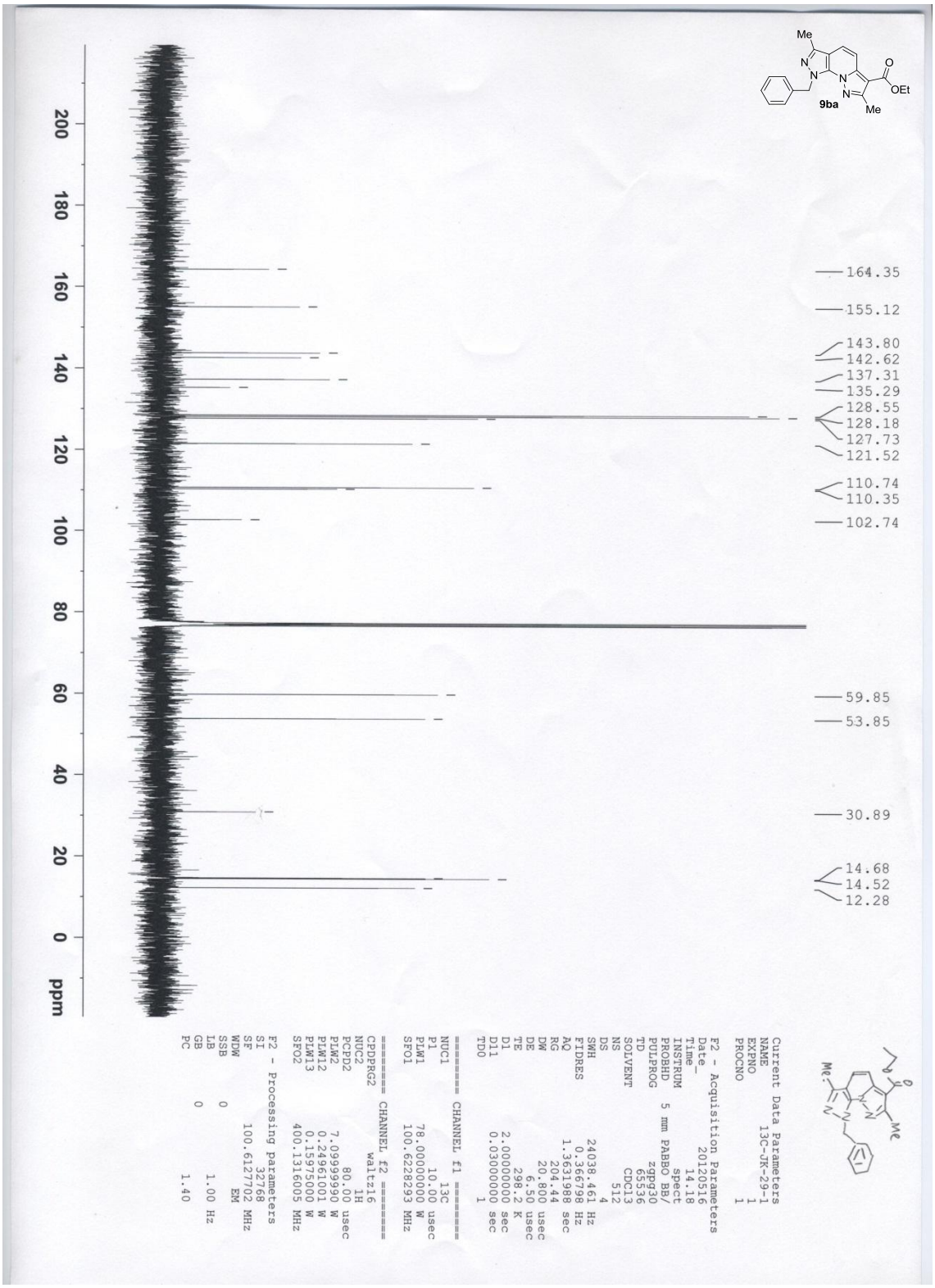

(Millions)
X : parts per Million : 1H
8aa
OEt
Me
8.8847
8.8780
8.1763
8.1619
8.1387
7.6240
7.5999
7.5257
7.5142
7.2530
4.4482
4.4309
4.4126
2.7952
2.4859
1.5519
1.4497
1.4314
0.8734
-0.0105
23.25
47.27
86.92
48.46
2.93
68.71
100
1.86
27.13
103127

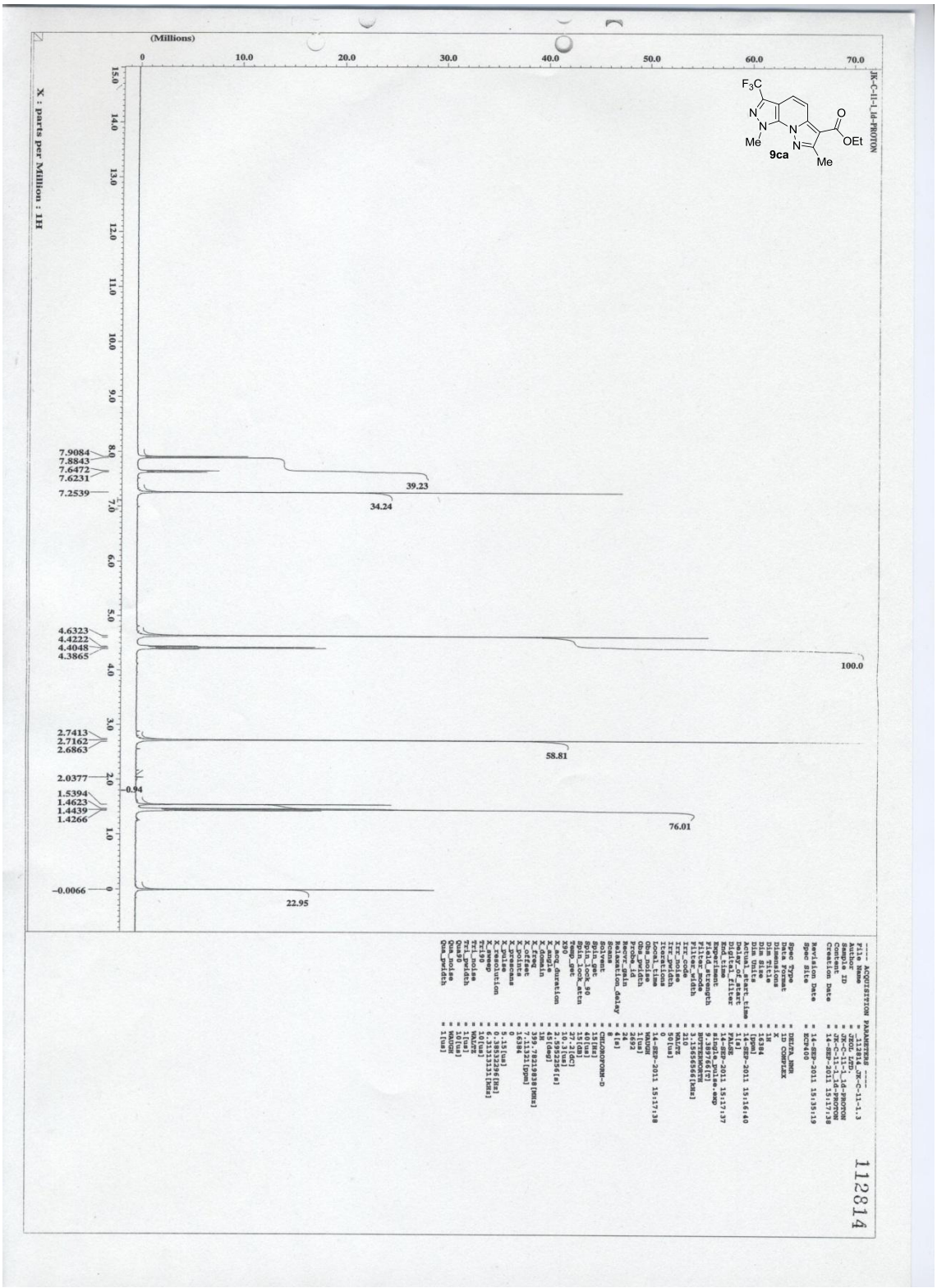

8aa
Me
OEt
163.98
155.37
150.58
143.87
142.03
137.11
126.55
121.37
118.38
118.26
105.37
77.32
77.00
76.68
60.05
14.63
14.41
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-11-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120512
Time_ 16.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127746 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

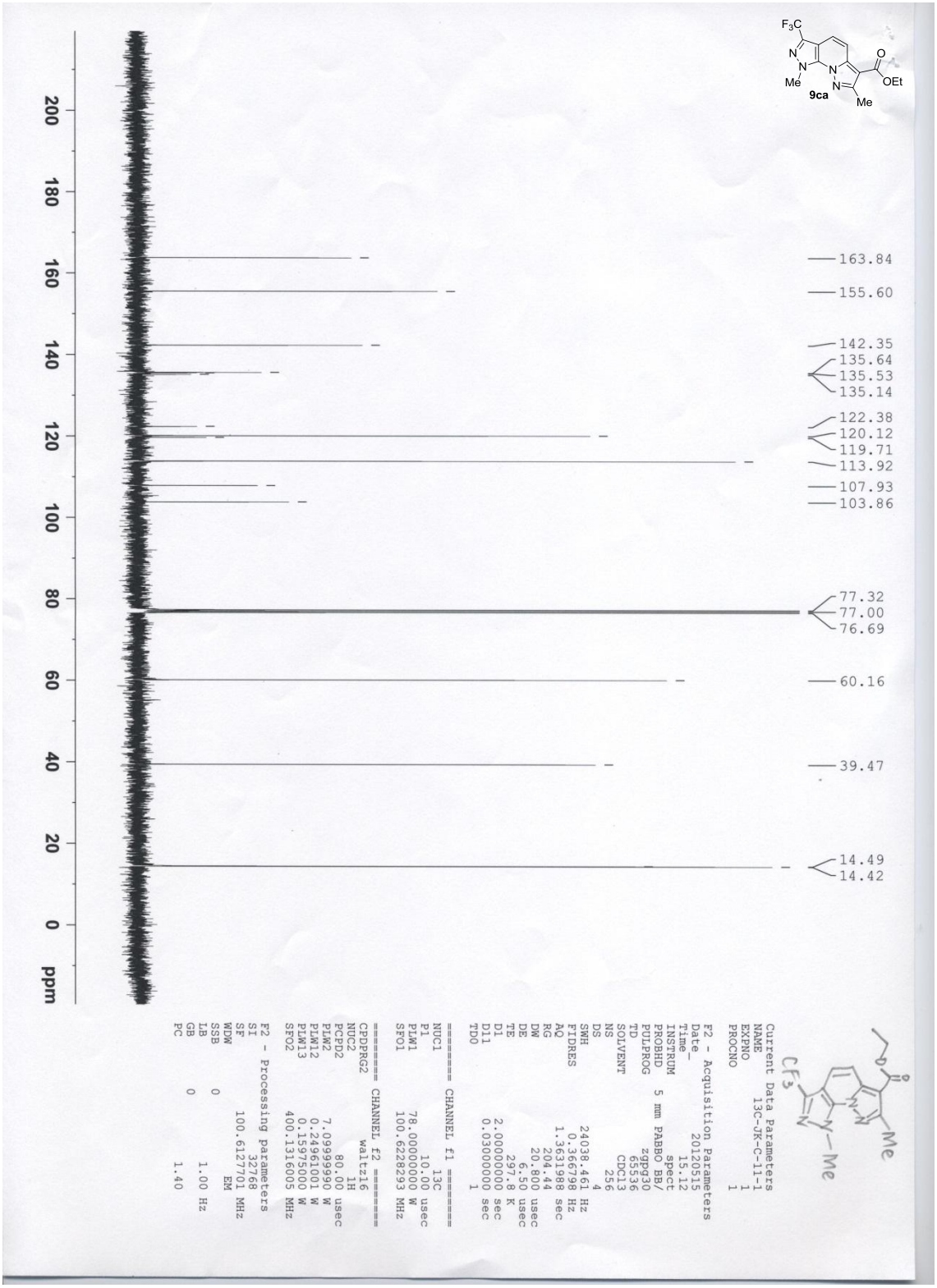

Me
N
N
Me
N
N
O
OEt
Me
9aa
(Millions)
X : parts per Million : 1H
7.7301
7.7069
7.5151
7.4920
7.2539
53.05
4.5032
4.4068
4.3885
82.99
2.6998
2.5186
100.0
1.5673
1.4546
1.4362
62.68
-0.0076
11.89
JK-C-18-1_1d-PROTON
113200


Me
N
N
Me
N
O
OEt
Me
9aa
164.29
155.08
142.95
142.50
135.62
121.51
110.53
110.08
102.64
77.32
77.00
76.68
59.83
38.05
14.58
14.49
12.09
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-C-18-1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120516
Time_ 14.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

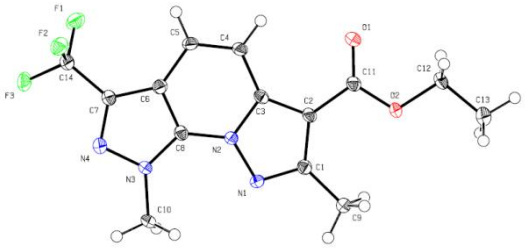

JK-C-2b-1.1d-PROTON
X : parts per Million : 1H
(Millions)
0
10.0
20.0
30.0
40.0
50.0
Me
N
N
N
N
O
OEt
Me
9ba
7.7378
7.5305
7.4101
7.3908
7.2809
7.2549
6.1253
4.4087
4.3913
4.3730
2.7143
2.5254
2.1649
1.5365
1.4517
1.4333
1.4160
-0.0047
100
17.23
17.56
52.97
0.61
45.07
23.69
113335


Me
N
N
N
O
OEt
9ba
Me
164.35
155.12
143.80
142.62
137.31
135.29
128.55
128.18
127.73
121.52
110.74
110.35
102.74
59.85
53.85
30.89
14.68
14.52
12.28
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JR-29-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120516
Time 14.18
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 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127702 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


JK-C-11-1_1d-PROTON
(Millions)
X : parts per Million : 1H
9ca
7.9084
7.8843
7.6472
7.6231
7.2539
4.6323
4.4222
4.4048
4.3865
2.7413
2.7162
2.6863
2.0377
1.5394
1.4623
1.4439
1.4266
-0.0066
39.23
34.24
100.0
58.81
0.94
76.01
22.95
112814


9ca
163.84
155.60
142.35
135.64
135.53
135.14
122.38
120.12
119.71
113.92
107.93
103.86
77.32
77.00
76.69
60.16
39.47
14.49
14.42
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME 13C-JK-C-11-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120515
Time_ 15.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NOC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

2. ORTEP Drawing of 9ca



Identification code	**9ca**	
Empirical formula	C14 H13 F3 N4 O2	
Formula weight	326.28	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 8.0135(7) Å	α= 90°.
	b = 15.0651(13) Å	β= 90.2910(10)°.
	c = 22.7939(19) Å	γ = 90°.
Volume	2751.7(4) Å3	
Z	8	
Density (calculated)	1.575 Mg/m3	
Absorption coefficient	0.136 mm-1	
F(000)	1344	
Crystal size	0.27 x 0.18 x 0.07 mm3	
Theta range for data collection	2.24 to 25.03°.	
Index ranges	-9<=h<=9, -13<=k<=17, -27<=l<=26	
Reflections collected	12930	
Independent reflections	4837 [R(int) = 0.0287]	
Completeness to theta = 25.03°	99.6 %	
Absorption correction	Analytical	
Max. and min. transmission	0.9906 and 0.9643	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	4837 / 0 / 421	
Goodness-of-fit on F2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0400, wR2 = 0.0967	
R indices (all data)	R1 = 0.0501, wR2 = 0.1018	
Largest diff. peak and hole	0.279 and -0.283 e.Å-3	