Supporting Information

A diastereoselective synthesis of pyrano fused coumarins via organocatalytic three-component reaction

Somayeh Ahadi, Mahdi Zolghadr, Hamid Reza Khavasi and Ayoob Bazgir*
Department of Chemistry, Shahid Beheshti University G. C., P. O. Box 19396-4716, Tehran, Iran
a_bazgir@sbu.ac.ir

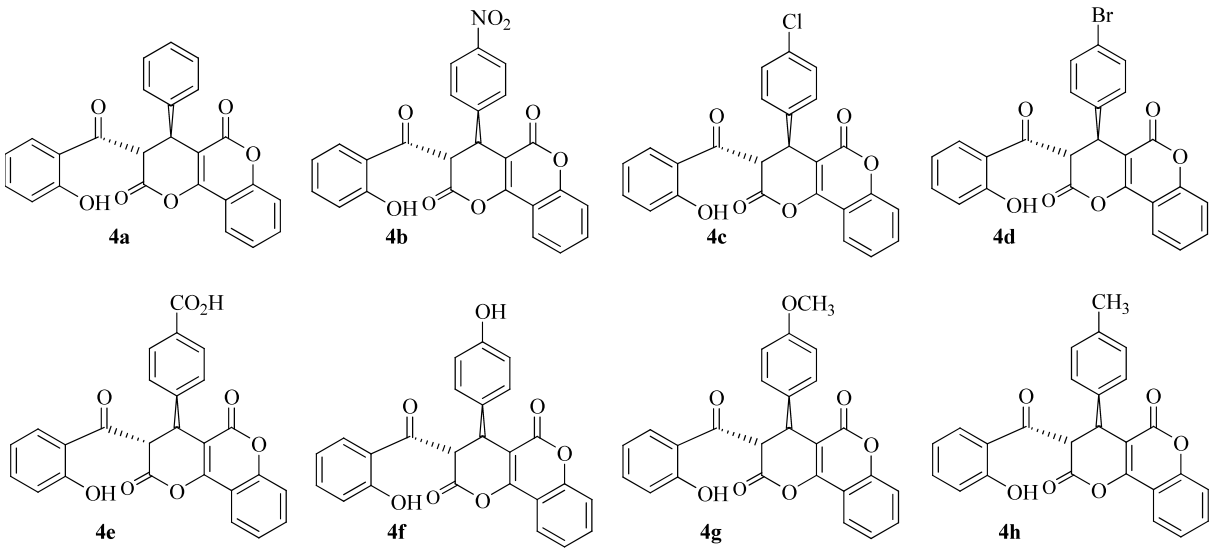
Table of Contents

Experimental Section

General remarks:

Melting points were determined on a melting point apparatus and are uncorrected. IR spectra were taken with a FT/IR spectrophotometer. ^{1}H NMR spectra were recorded on a 300 MHz spectrometer. Chemical shifts are expressed in parts per million downfield from tetramethylsilane as an internal standard. ^{13}C NMR spectra were recorded on a 70 MHz spectrometer using broadband proton decoupling. Chemical shifts are expressed in parts per million using the middle resonance of DMSO as an internal standard. Elemental analyses for C, H and N performed using a Heraeus CHN–O– Rapid analyzer.

All chemicals were purchased from Merck or Aldrich and were used without further purification. Known 3-bromo-4-hydroxycoumarin was prepared by the procedure reported previously (Kotharkar, S. A.; Shinde, D. B. *Bioorg. Med. Chem. Lett*. **2006**, 16, 6181–6184).



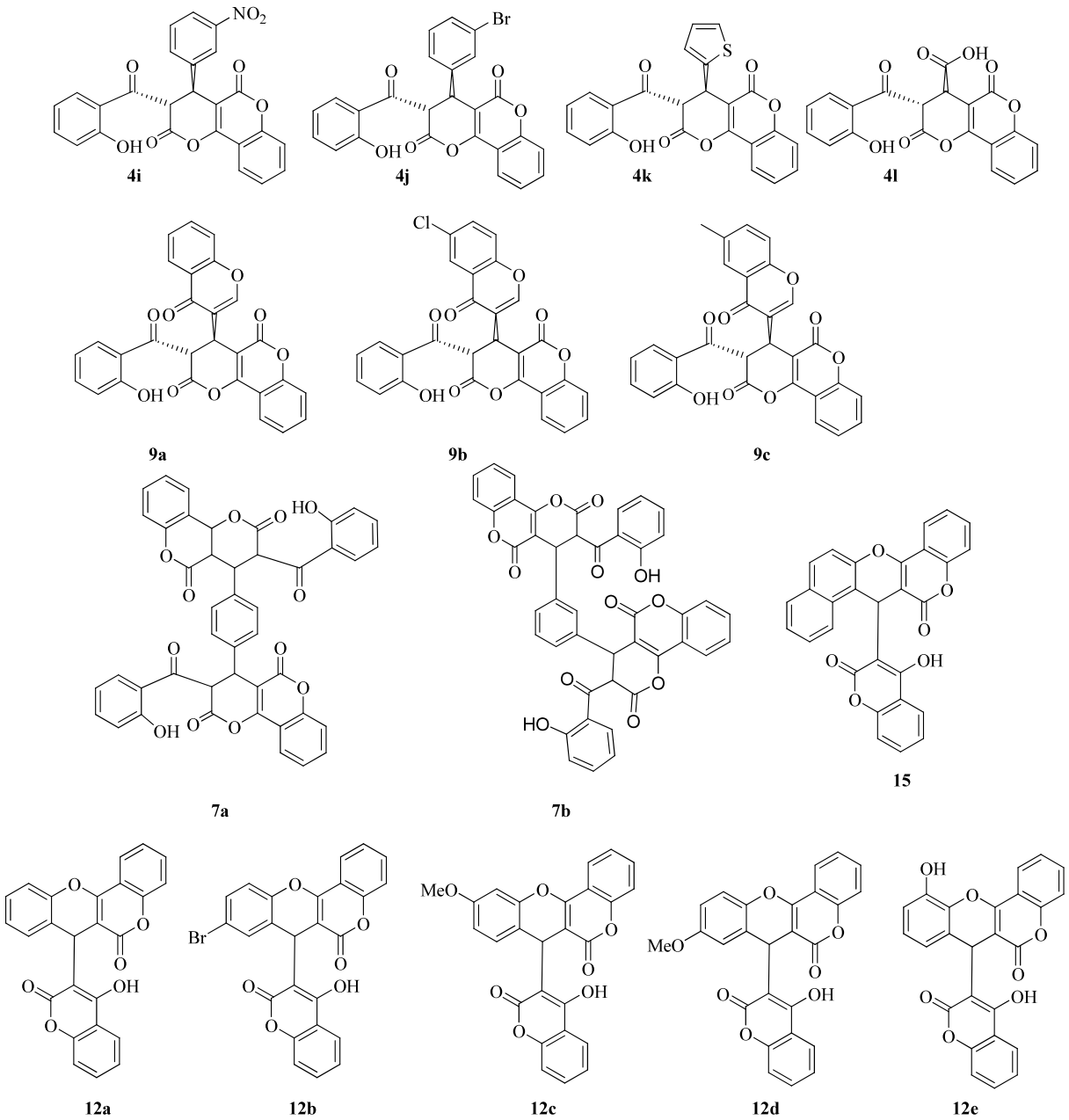


FIGURE 1: Structure of all products

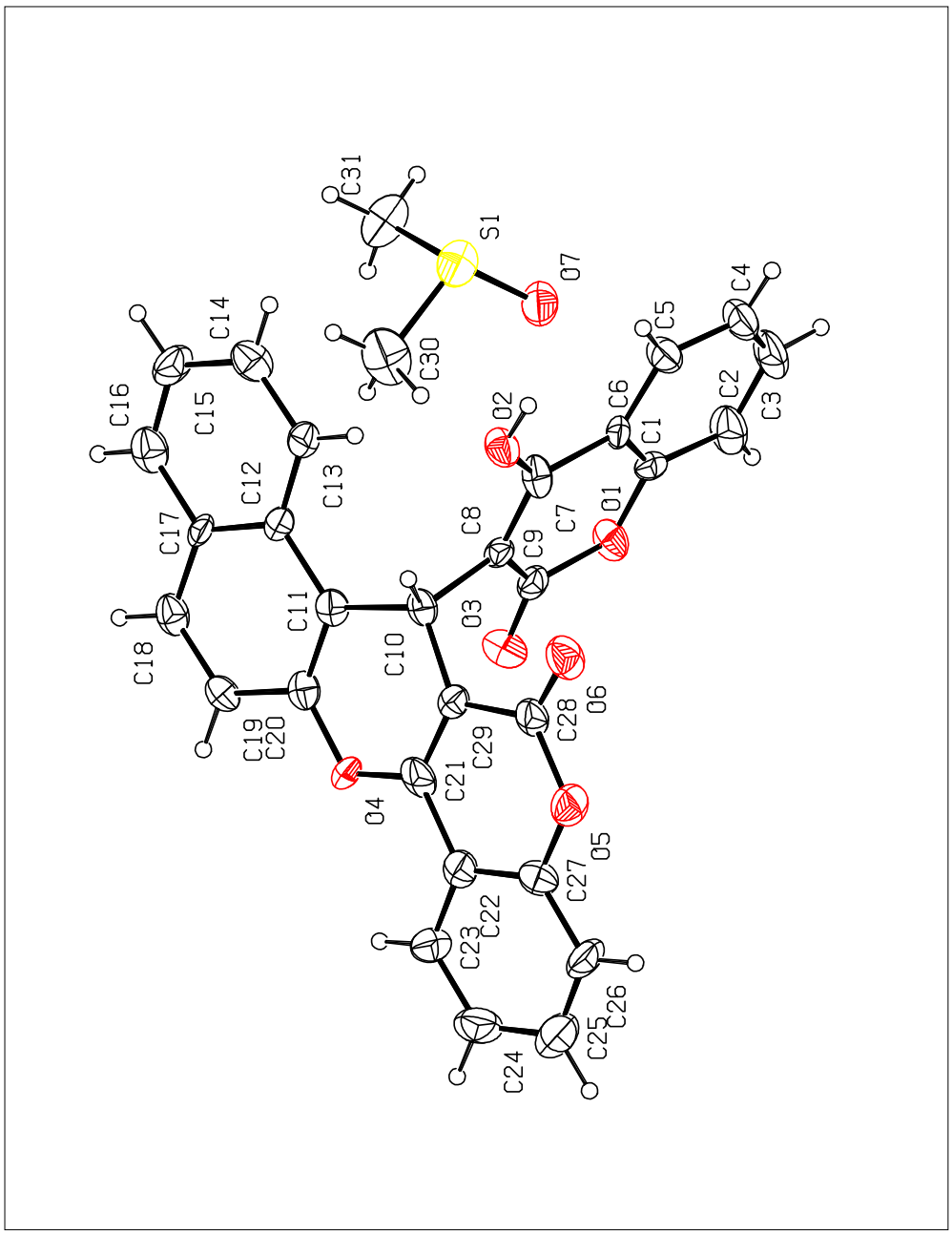
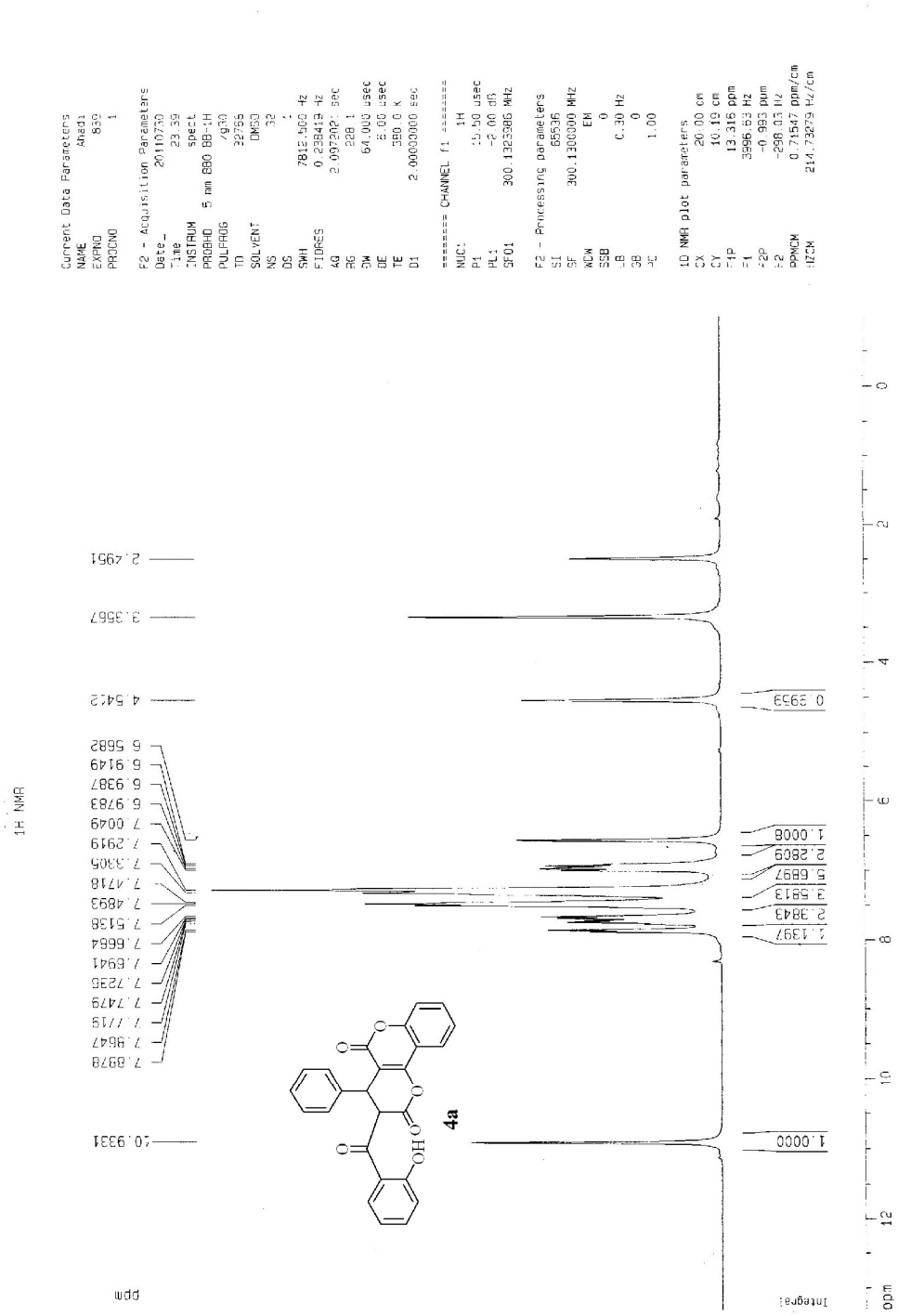
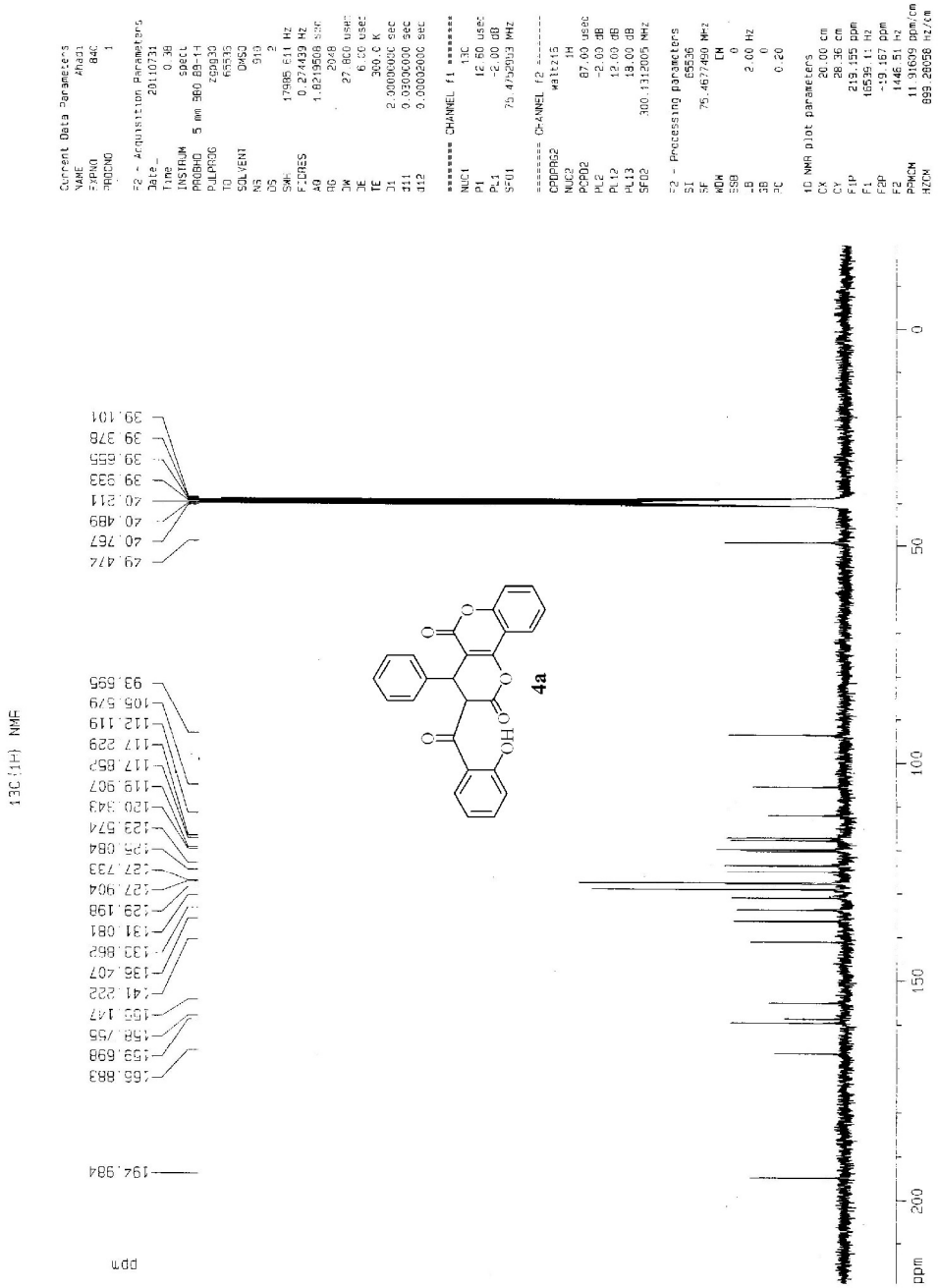
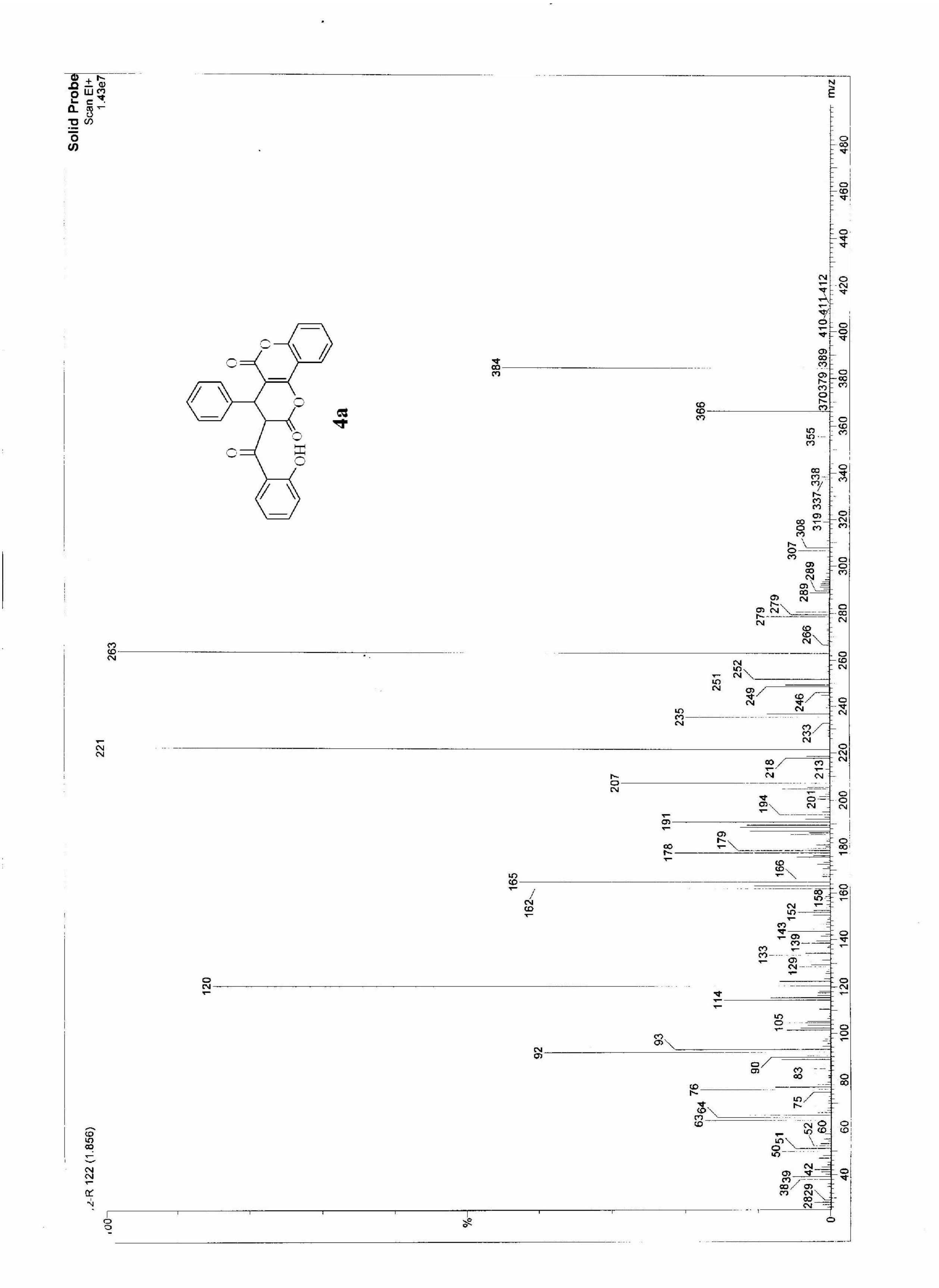
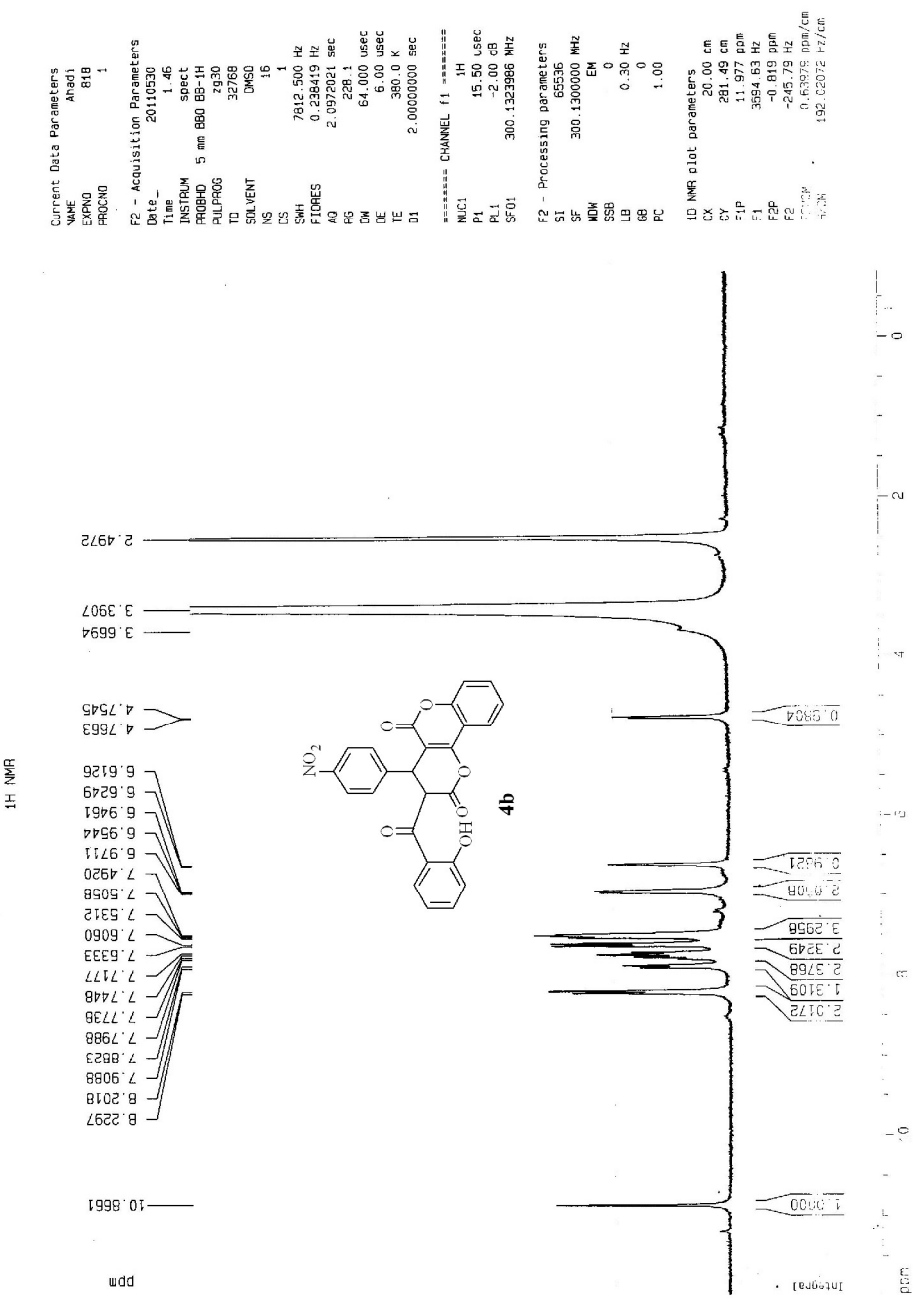


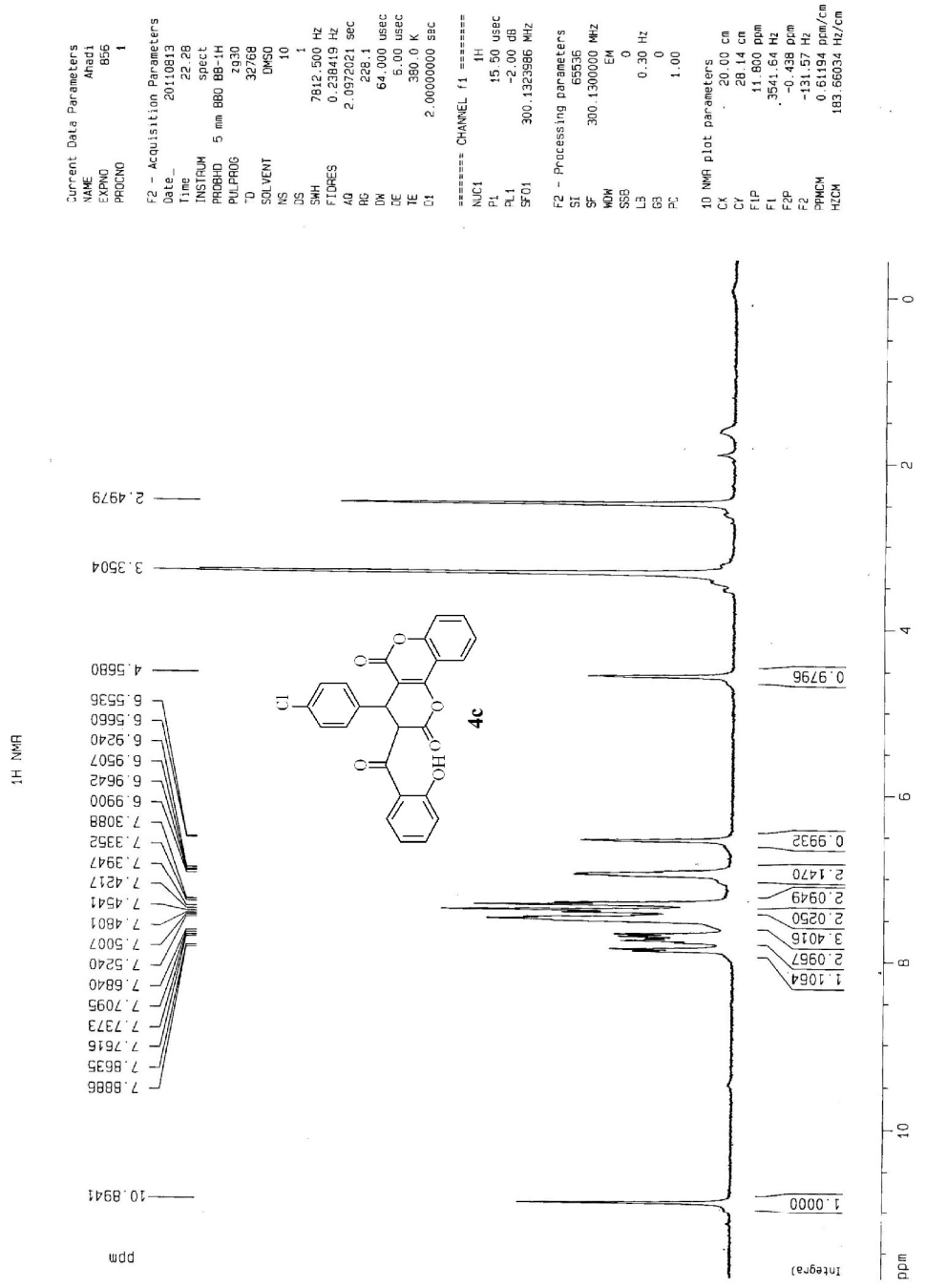
FIGURE 2. ORTEP diagram of product **15**

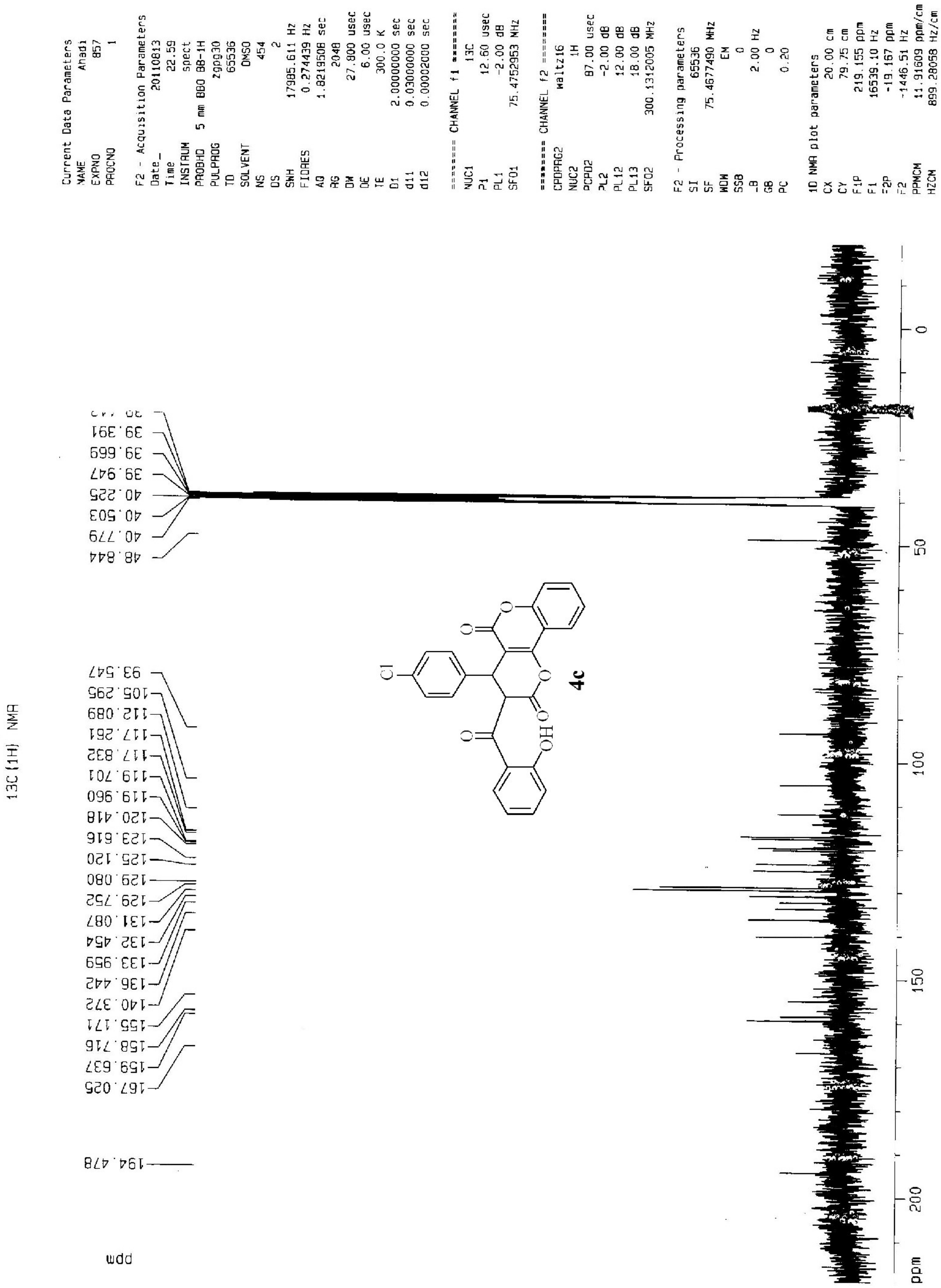

1H NMR
4a
OH
O
Current Data Parameters
NAME Ahad1
EXPNO 830
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110730
Time 23.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 4
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972320 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 350.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.19 cm
F1P 13.316 ppm
F1 3996.63 Hz
F2P -0.993 ppm
F2 -298.03 Hz
PPMCM 0.71547 ppm/cm
HZCM 214.73279 Hz/cm
ppm
Integral
2.4961
3.3567
4.5412
5.5682
6.9149
6.9387
6.9783
7.0049
7.2919
7.3305
7.4718
7.4893
7.5138
7.6684
7.6941
7.7236
7.7479
7.7719
7.9647
7.9878
10.9331
0.9959
1.0008
2.2809
5.6897
3.5813
2.3843
2.1397
1.0000

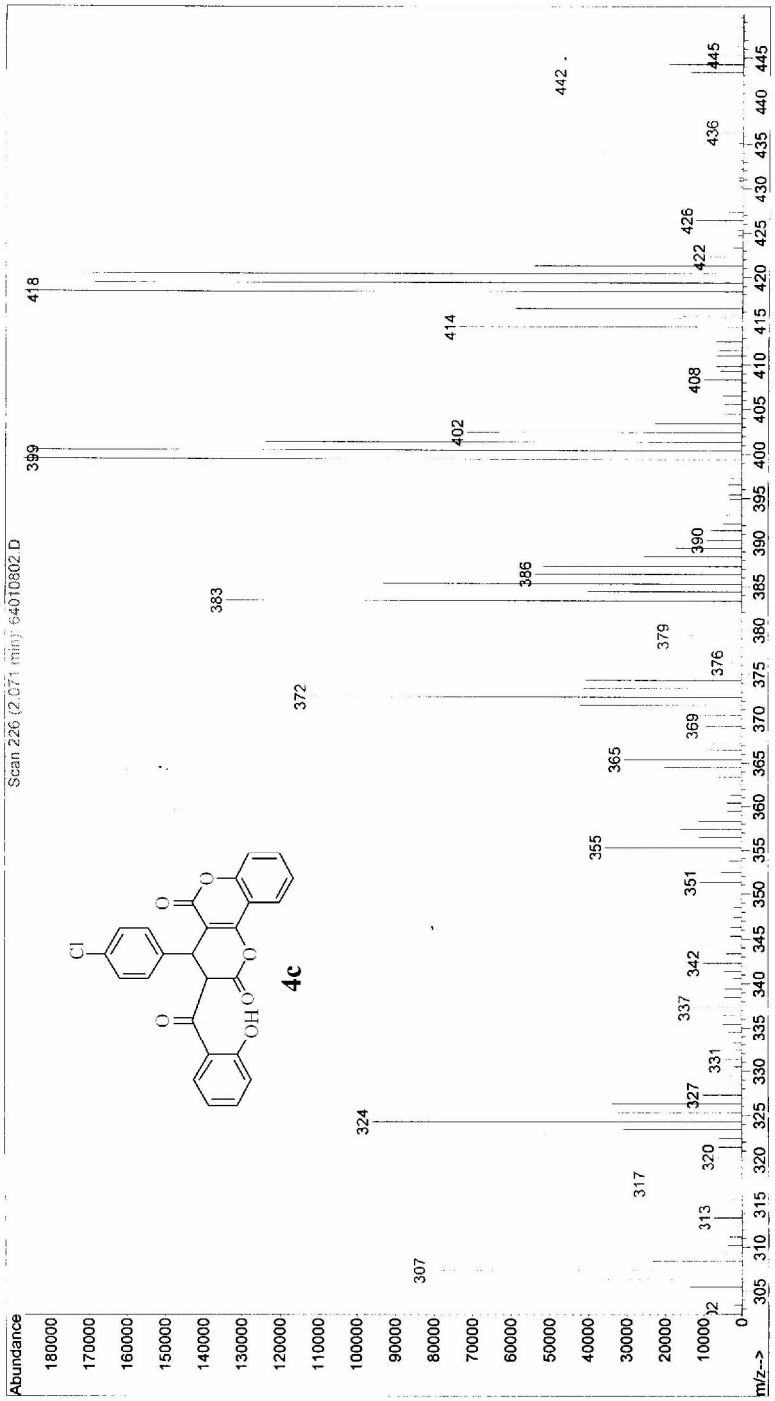

Current Data Parameters
NAME Anasi
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110731
Time 0.38
INSTRUM spect
PROBHD 5 mm BBO BB-H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 910
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.860 usec
DE 6.50 usec
TE 360.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752093 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 28.36 cm
F1P 219.155 ppm
F1 16539.11 Hz
F2P -19.167 ppm
F2 1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm
13C{1H} NMF
4a
OH
ppm
0
50
100
150
200
39.104
39.378
39.655
39.933
40.211
40.489
40.767
40.474
93.695
105.579
112.119
117.229
117.852
119.907
120.343
123.574
125.084
127.733
127.904
129.198
131.081
133.862
136.407
141.222
155.147
158.755
159.698
166.883
194.984

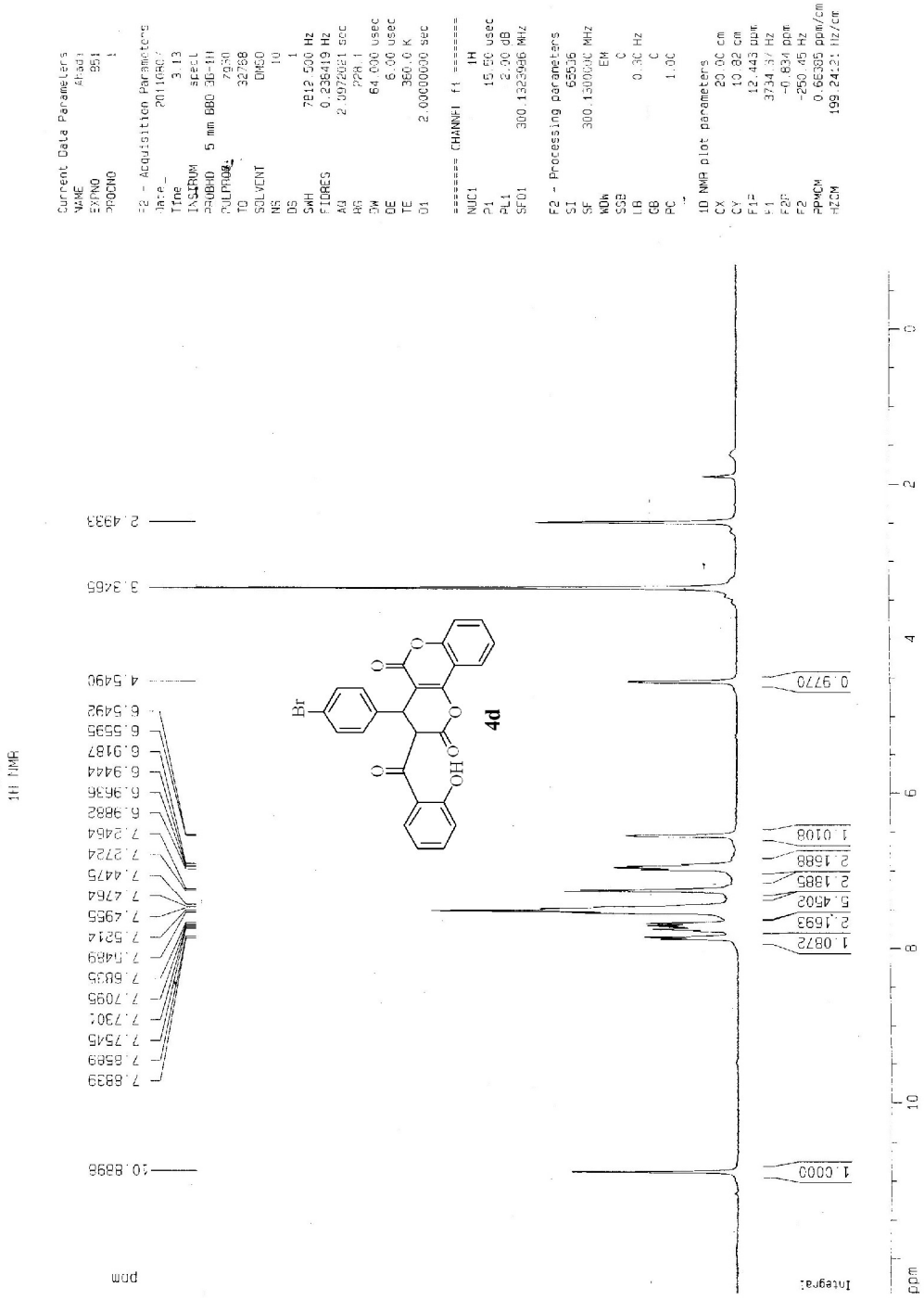


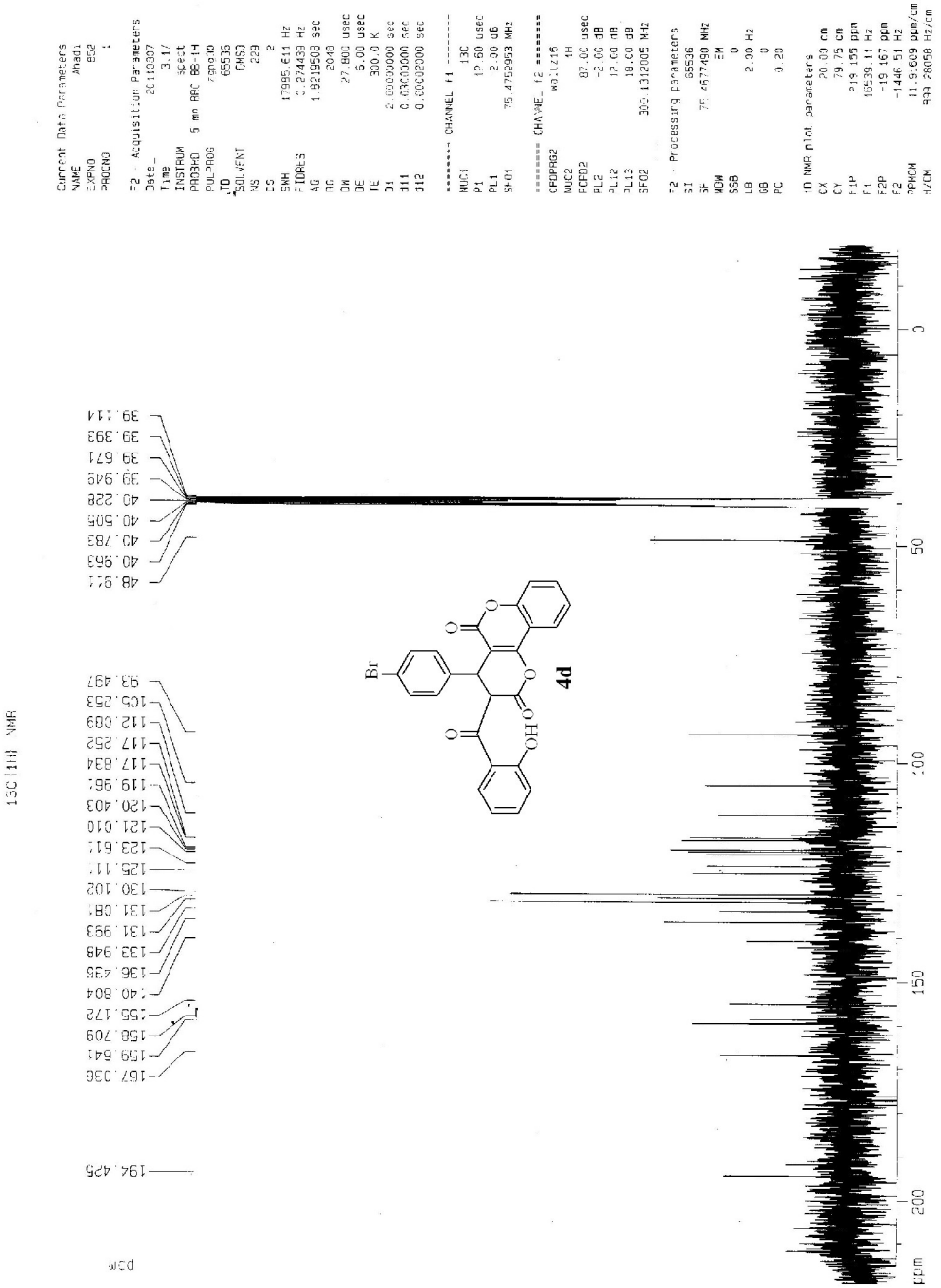

1H NMR
4b
ppm

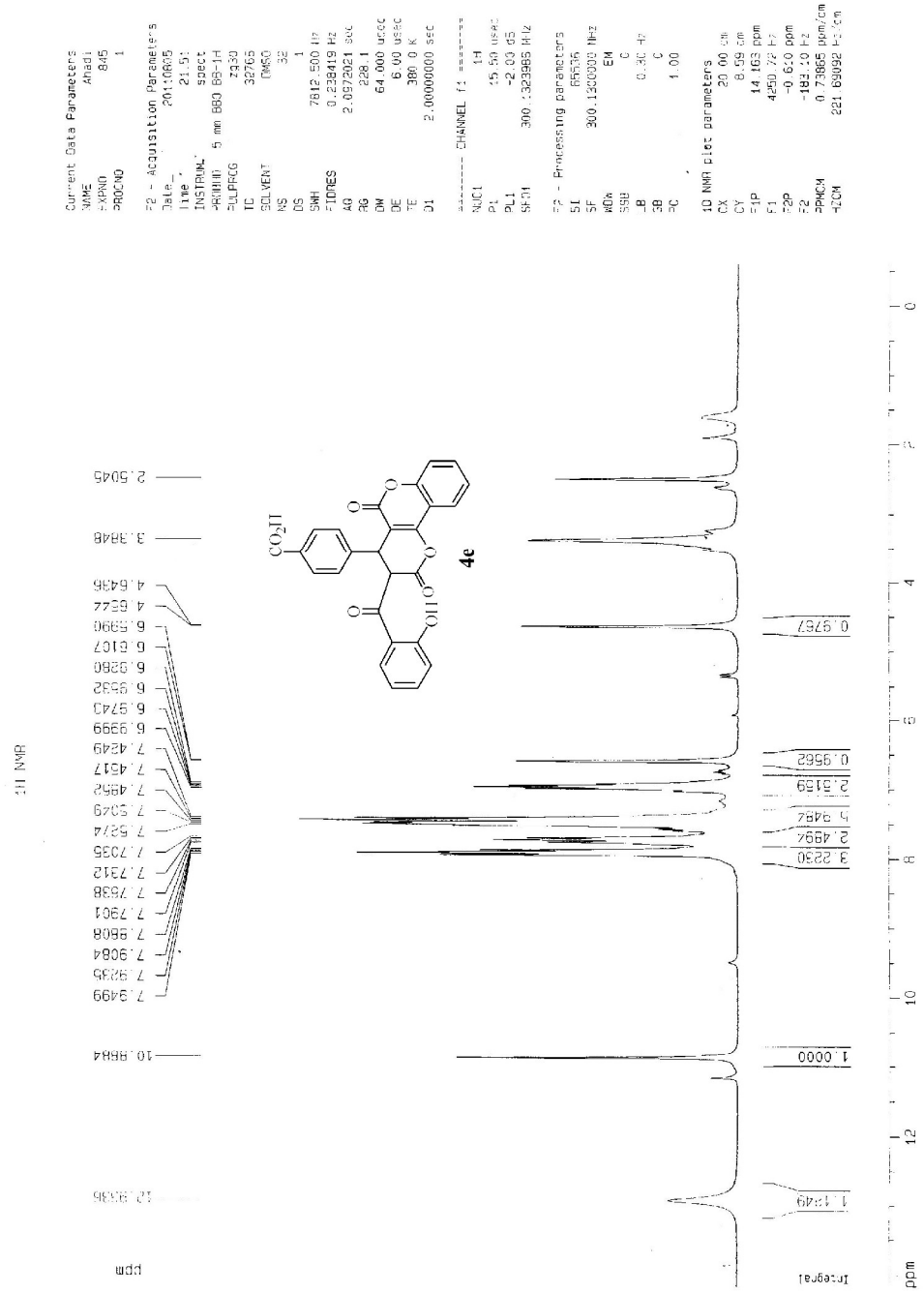

1H NMR
4c
Cl
O
OH
2.4979
3.3504
4.5680
6.5536
6.5660
6.9240
6.9507
6.9642
6.9900
7.3088
7.3352
7.3947
7.4217
7.4541
7.4801
7.5007
7.5240
7.6840
7.7095
7.7373
7.7616
7.8635
7.8886
10.8941
0.9796
0.9932
2.1470
2.0949
2.0250
3.4016
2.0957
1.1064
1.0000
Integral
ppm

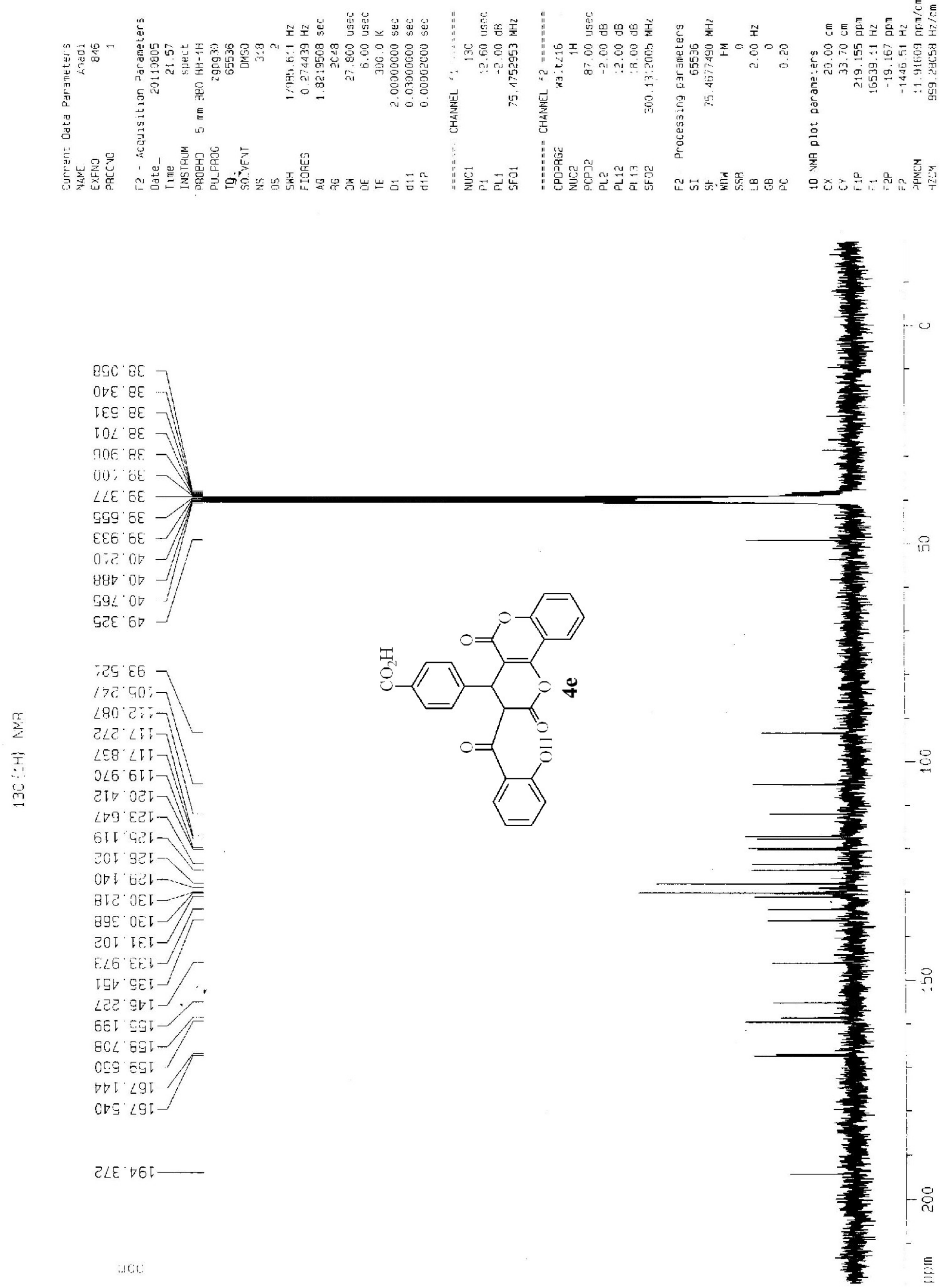

13C {1H} NMR
194.478
167.025
159.637
158.716
155.171
140.372
136.442
133.959
132.454
131.087
129.752
129.080
125.120
123.616
120.418
119.960
119.701
117.832
117.261
112.089
105.295
93.547
48.844
40.779
40.503
40.225
39.947
39.669
39.391
Cl
O
O
O
O
OH
O
4c
ppm
200
150
100
50
0
Current Data Parameters
NAME Ahadi
EXPNO 857
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110813
Time 22.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 454
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 79.75 cm
F1P 219.155 ppm
F1 16539.10 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm

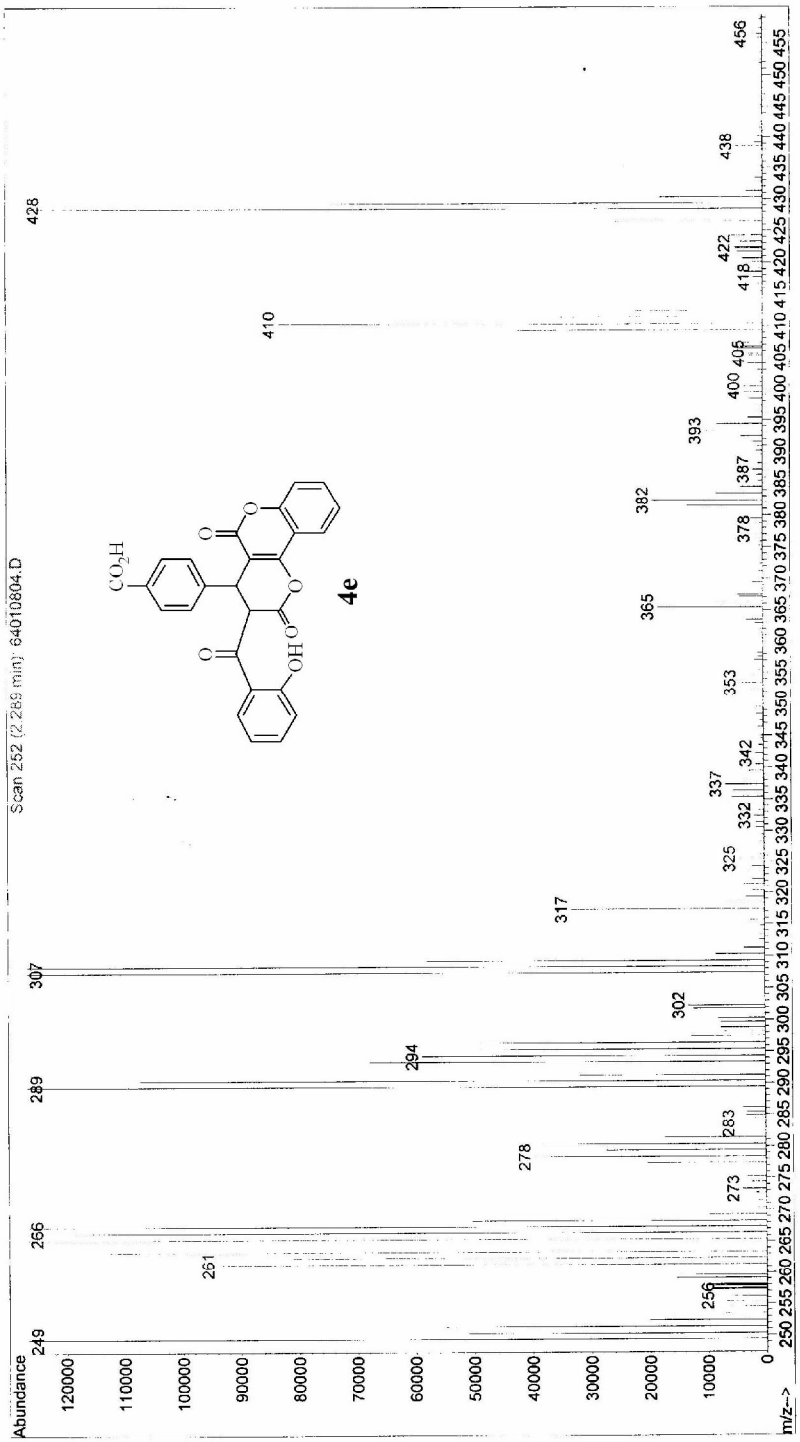

Scan 226 (2.071 min): 64010802.D
Abundance
m/z-->
4c
Cl
O
OH

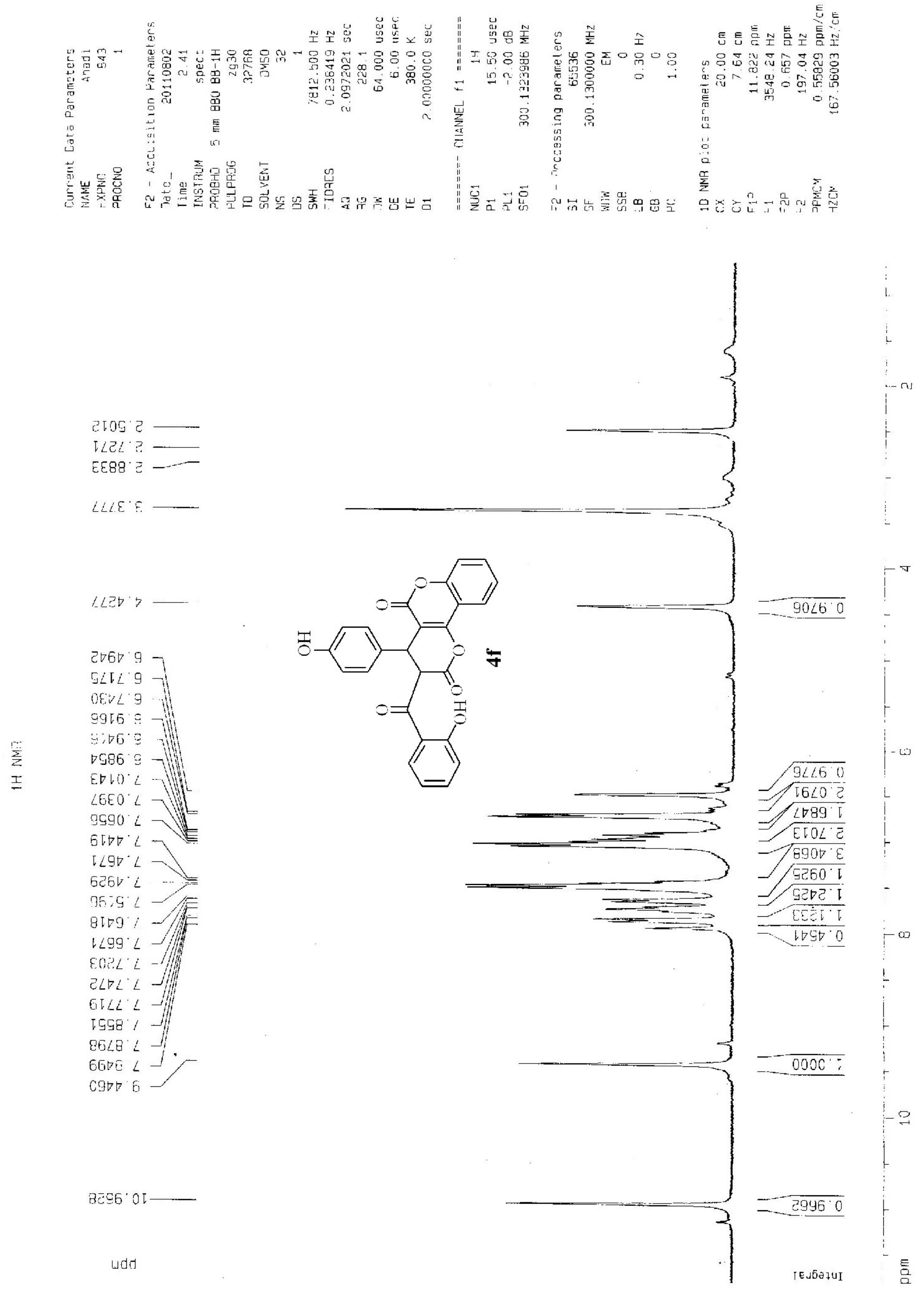

4d
Br
O
OH
2.4833
3.3465
4.5490
6.5492
6.5596
6.9187
6.9444
6.9636
6.9882
7.2464
7.2724
7.4475
7.4764
7.4955
7.5214
7.5469
7.6035
7.7095
7.7301
7.7545
7.8589
7.8839
10.8896
0.9770
1.0108
2.1588
2.1885
5.4602
2.1693
1.0872
1.0000
1H NMR
ppm
Integral

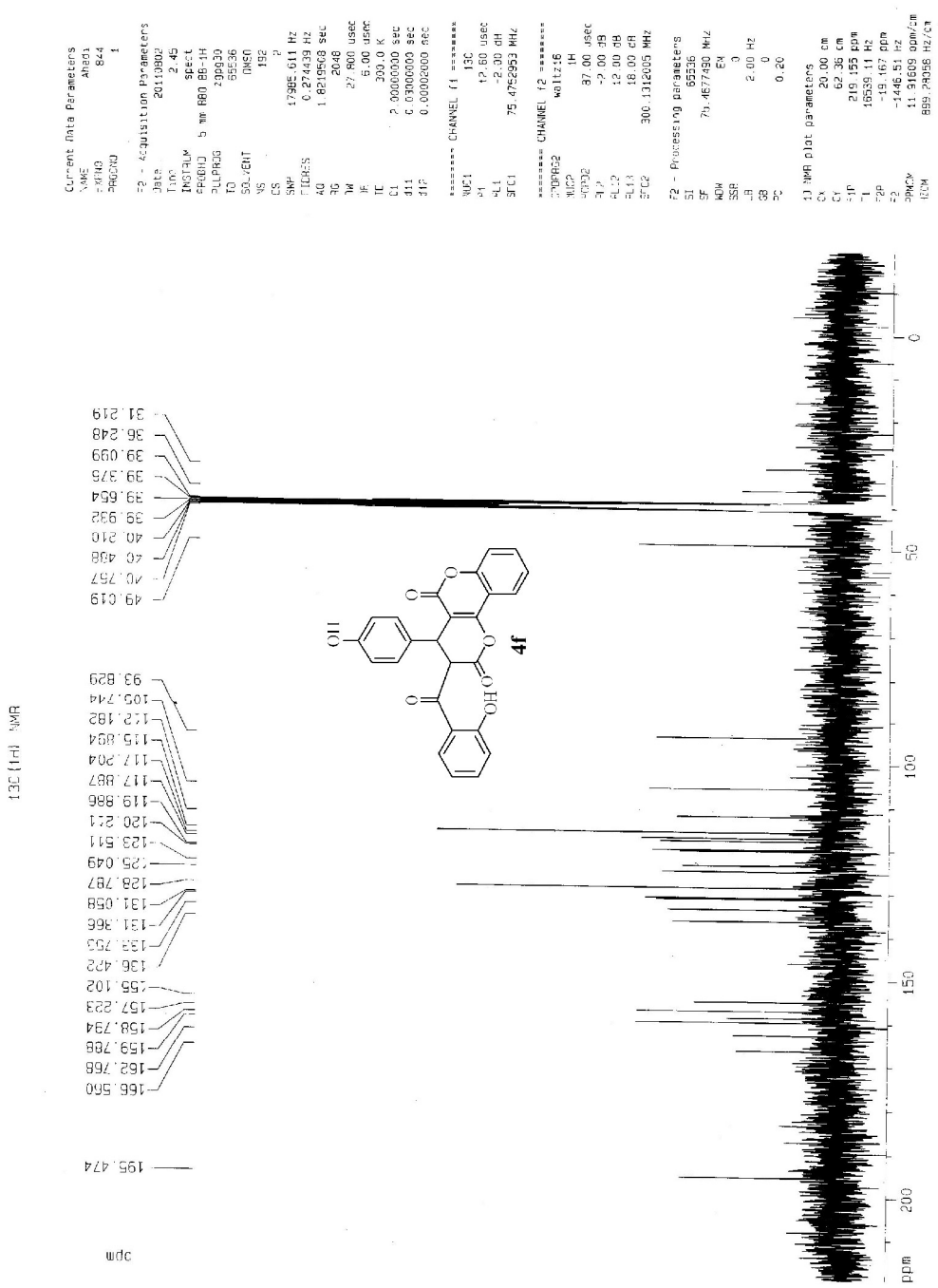

13C{1H} NMR
4d
Br
O
OH
ppm

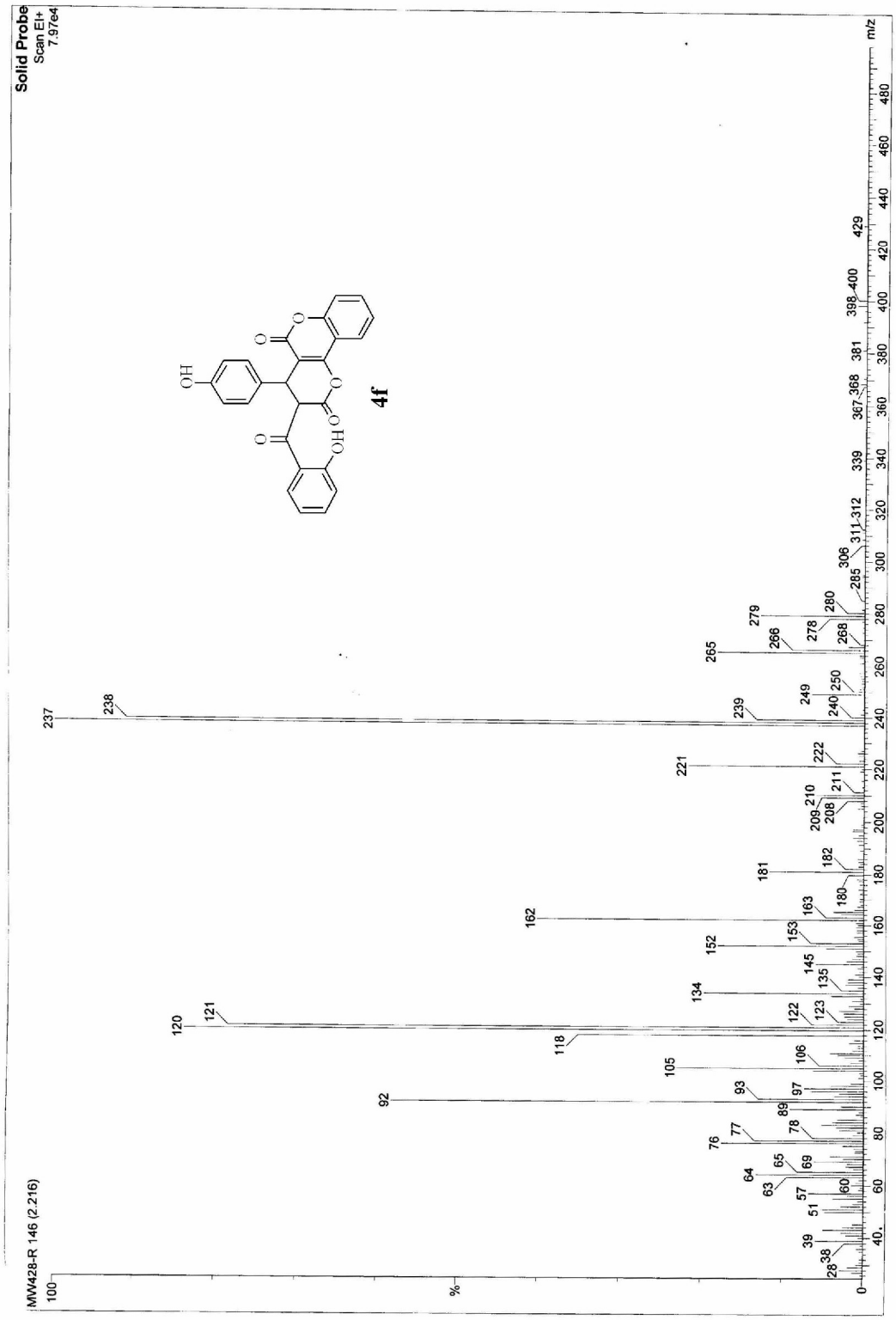

4e
2.5045
3.3848
4.6436
4.6524
6.5990
6.6107
6.9280
6.9582
6.9740
6.9999
7.4249
7.4517
7.4952
7.5049
7.5274
7.7035
7.7312
7.7638
7.7901
7.8808
7.9084
7.9235
7.9499
10.8884
0.9787
0.9562
2.3159
5.3482
2.4892
3.2230
1.0000
1.1249
ppm

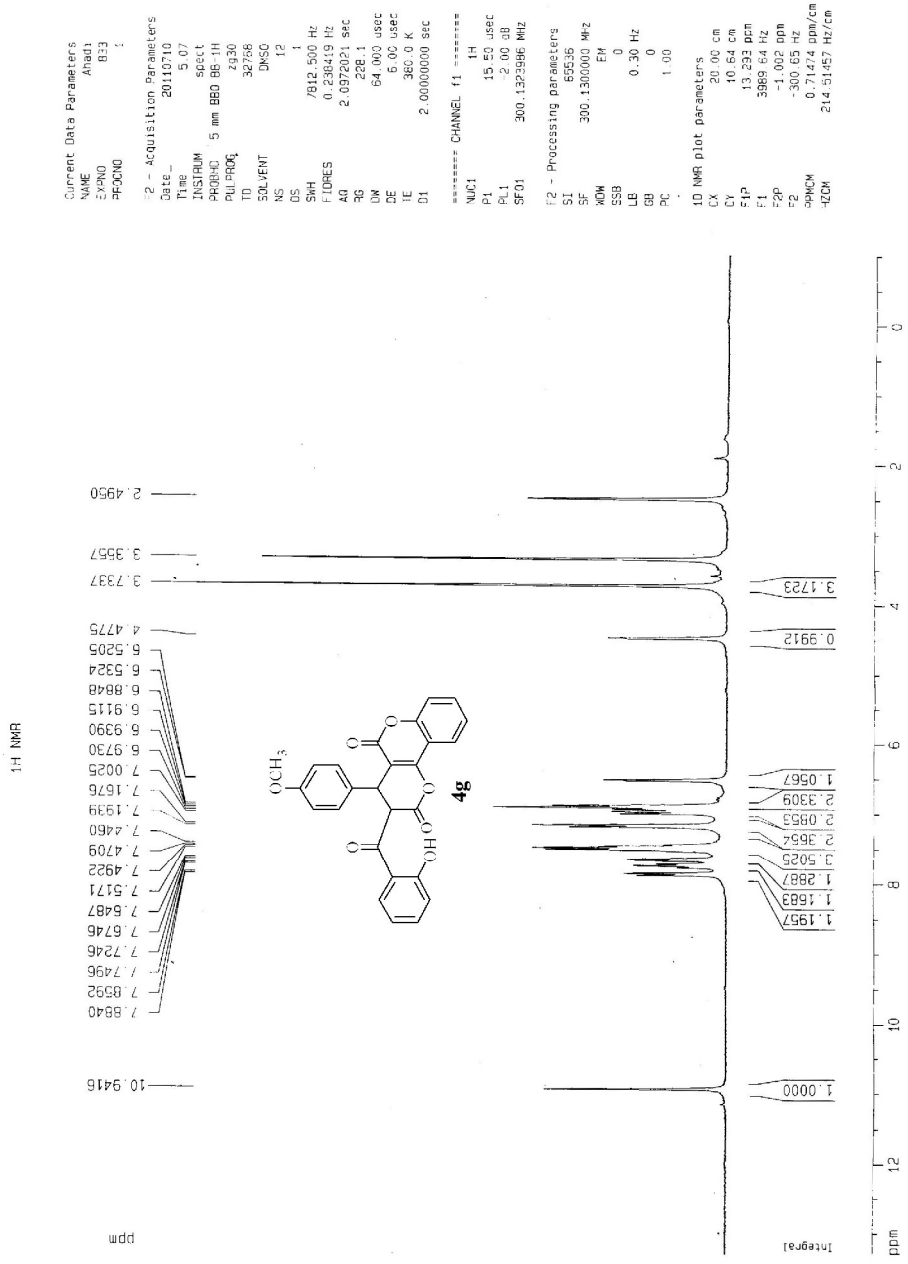


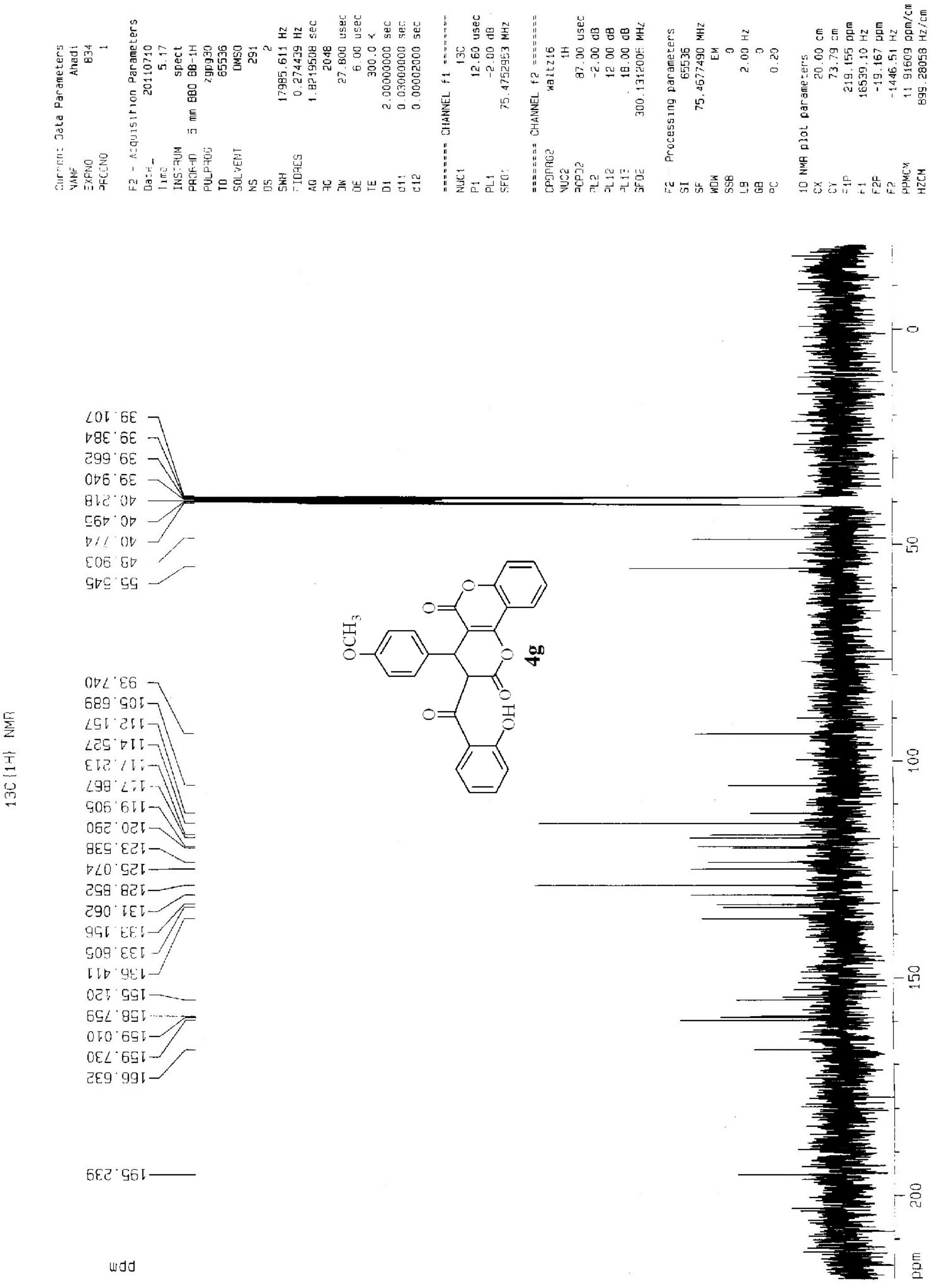


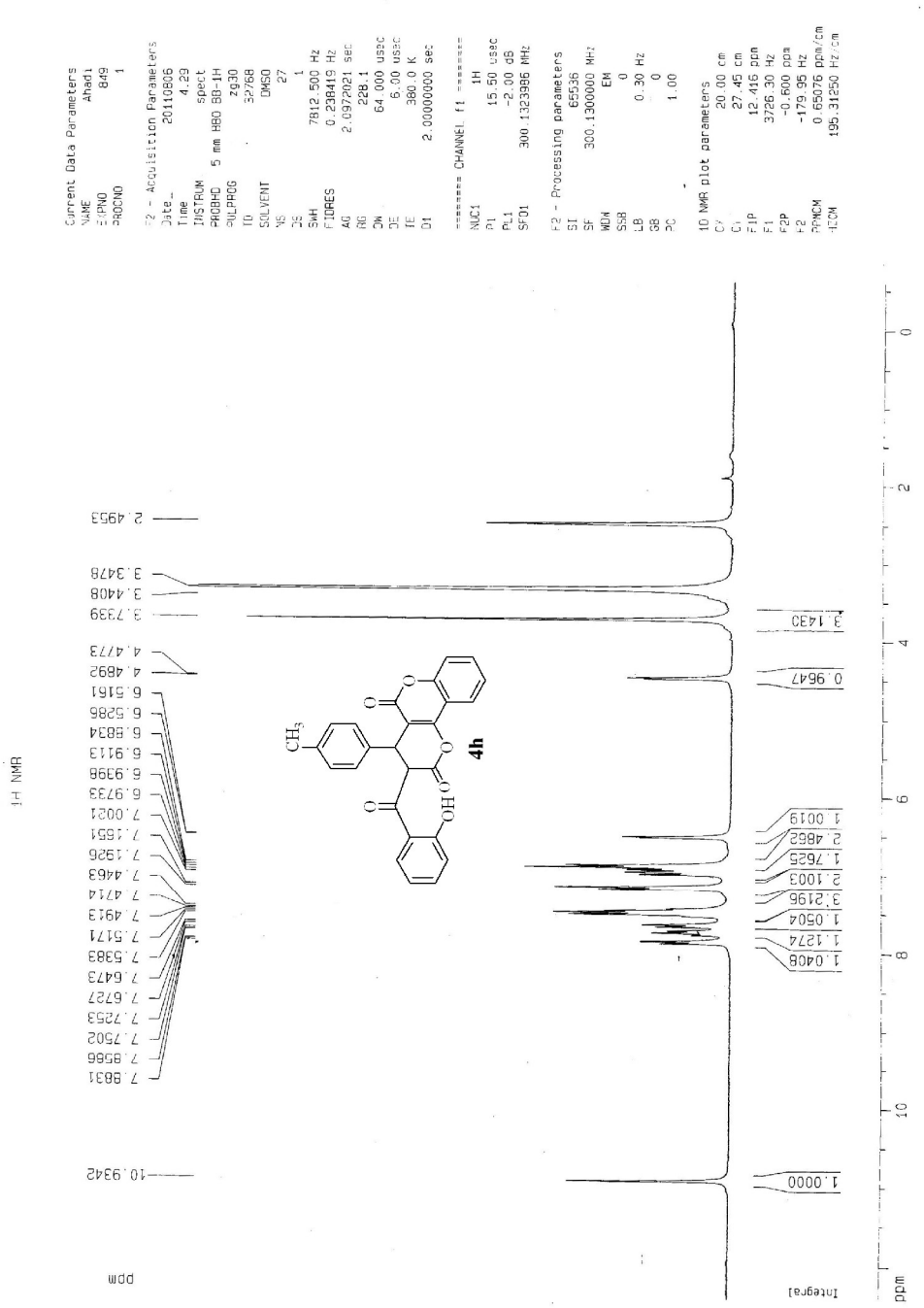

1H NMR
4f
Current Data Parameters
NAME Anadi
EXPNO 543
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110802
Time 2.41
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 7.64 cm
F1P 11.822 ppm
F1 3548.24 Hz
F2P 0.657 ppm
F2 197.04 Hz
PPMCM 0.55829 ppm/cm
HZCM 167.56003 Hz/cm
2.5012
2.7271
2.8833
3.3777
4.4277
6.4942
6.7175
6.7430
6.9166
6.9416
6.9654
7.0143
7.0397
7.0656
7.4419
7.4671
7.4929
7.5190
7.6418
7.6671
7.7203
7.7472
7.7719
7.8551
7.8798
7.9499
9.4463
10.9628
0.9706
0.9776
2.0791
1.6847
2.7013
3.4068
1.0925
1.2425
1.1233
0.4541
1.0000
0.9662
Integral
ppm

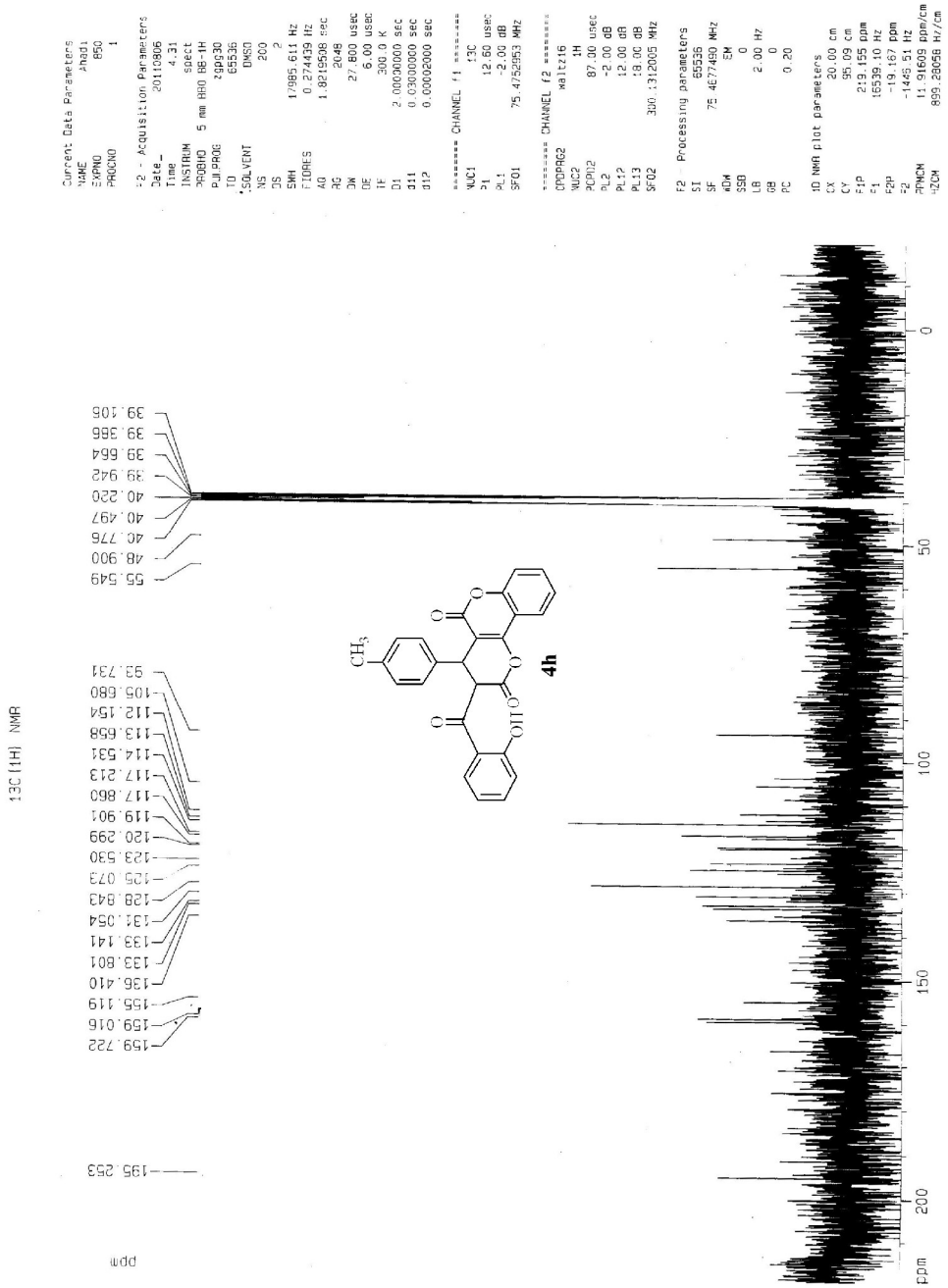

13C{1H} NMR
4f
ppm

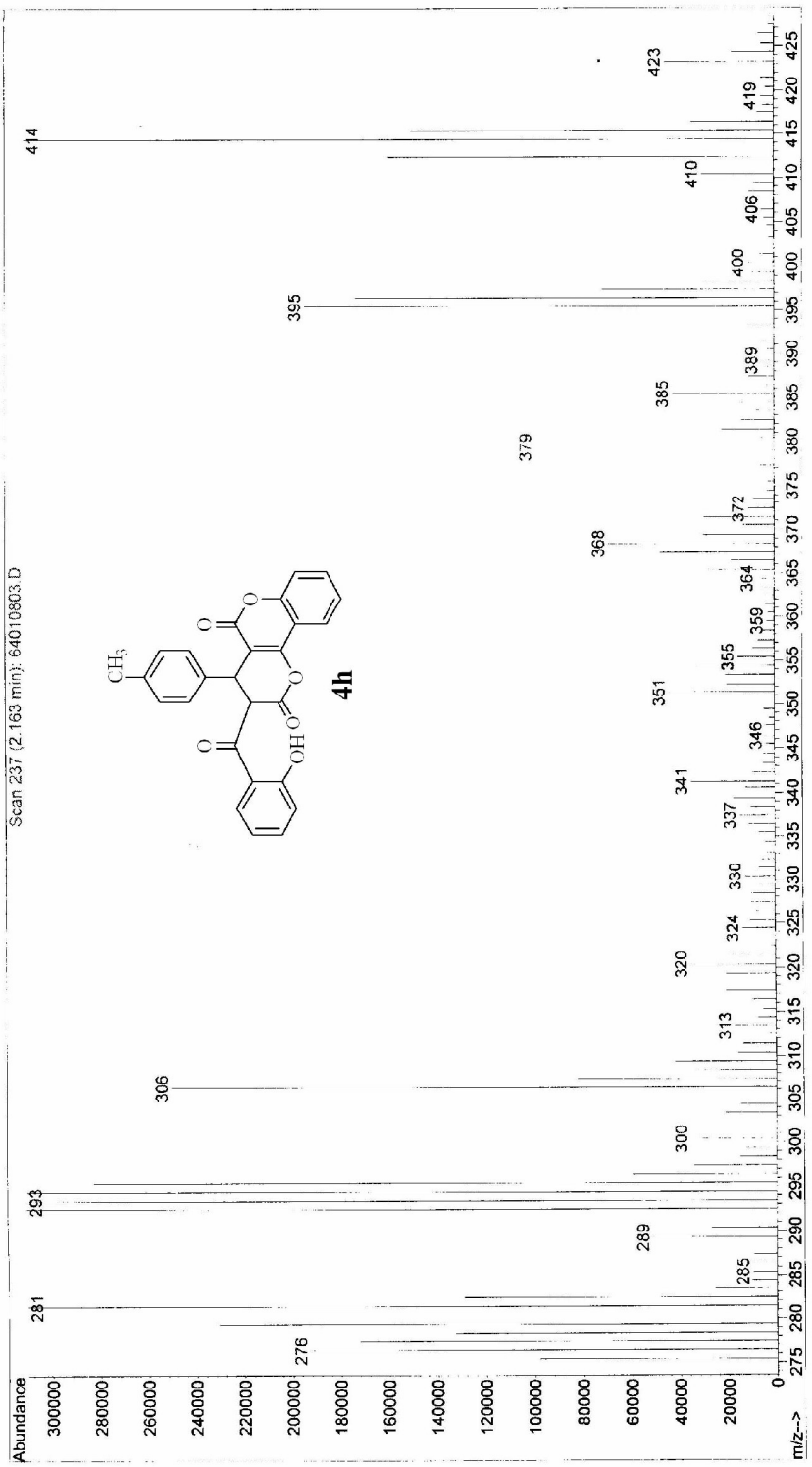

Solid Probe
Scan EI+
7.97e4
MW428-R 146 (2.216)
100
%
0
m/z
4f
OH
OH

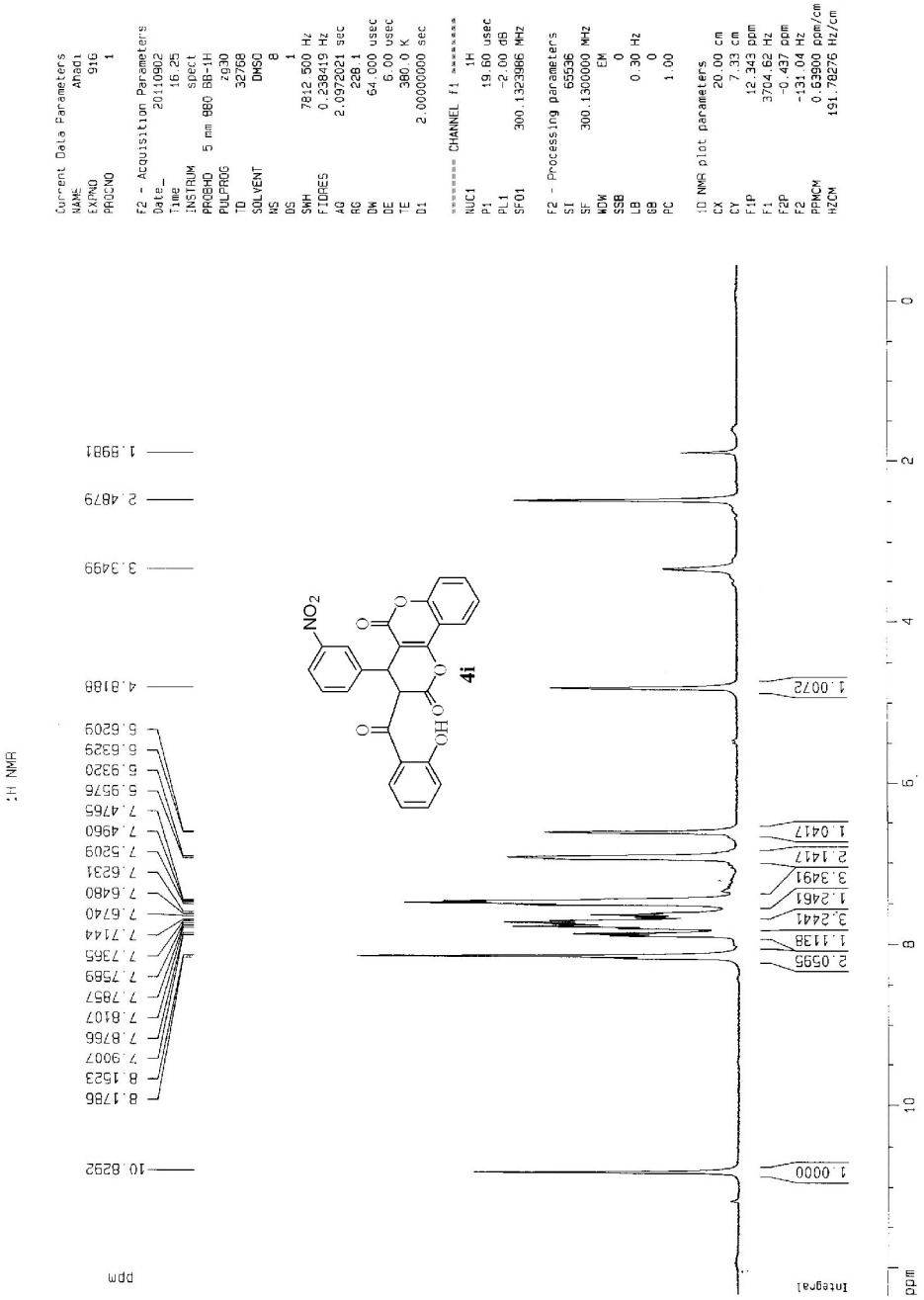

1H NMR
4g
OCH3
OH
2.4950
3.3657
3.7337
4.4775
6.5205
6.5324
6.8848
6.9115
6.9390
6.9730
7.0025
7.1676
7.1939
7.4460
7.4709
7.4922
7.5171
7.5487
7.5746
7.7246
7.7496
7.8592
7.8840
10.9416
3.1723
0.9912
1.0567
2.3309
2.0853
2.3654
3.5025
1.2887
1.1683
1.1957
1.0000
Integral
ppm

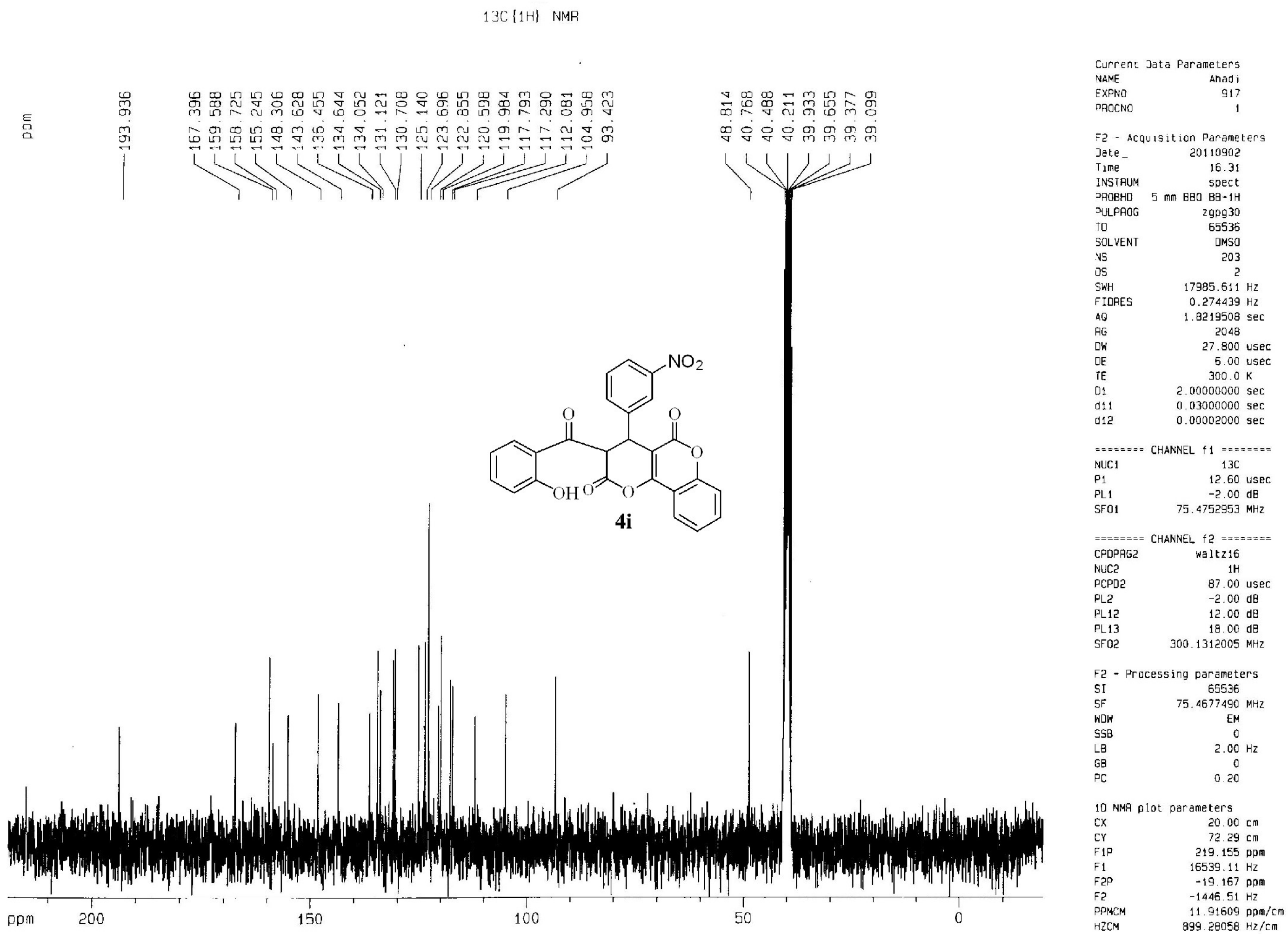

13C{1H} NMR
4g
OCH3
OH
39.107
39.384
39.662
39.940
40.218
40.495
40.774
49.903
55.545
93.740
105.689
112.157
114.527
117.213
117.867
119.905
120.290
123.538
125.074
128.852
131.062
133.156
133.805
136.411
155.120
158.759
159.010
159.730
166.632
195.239
ppm

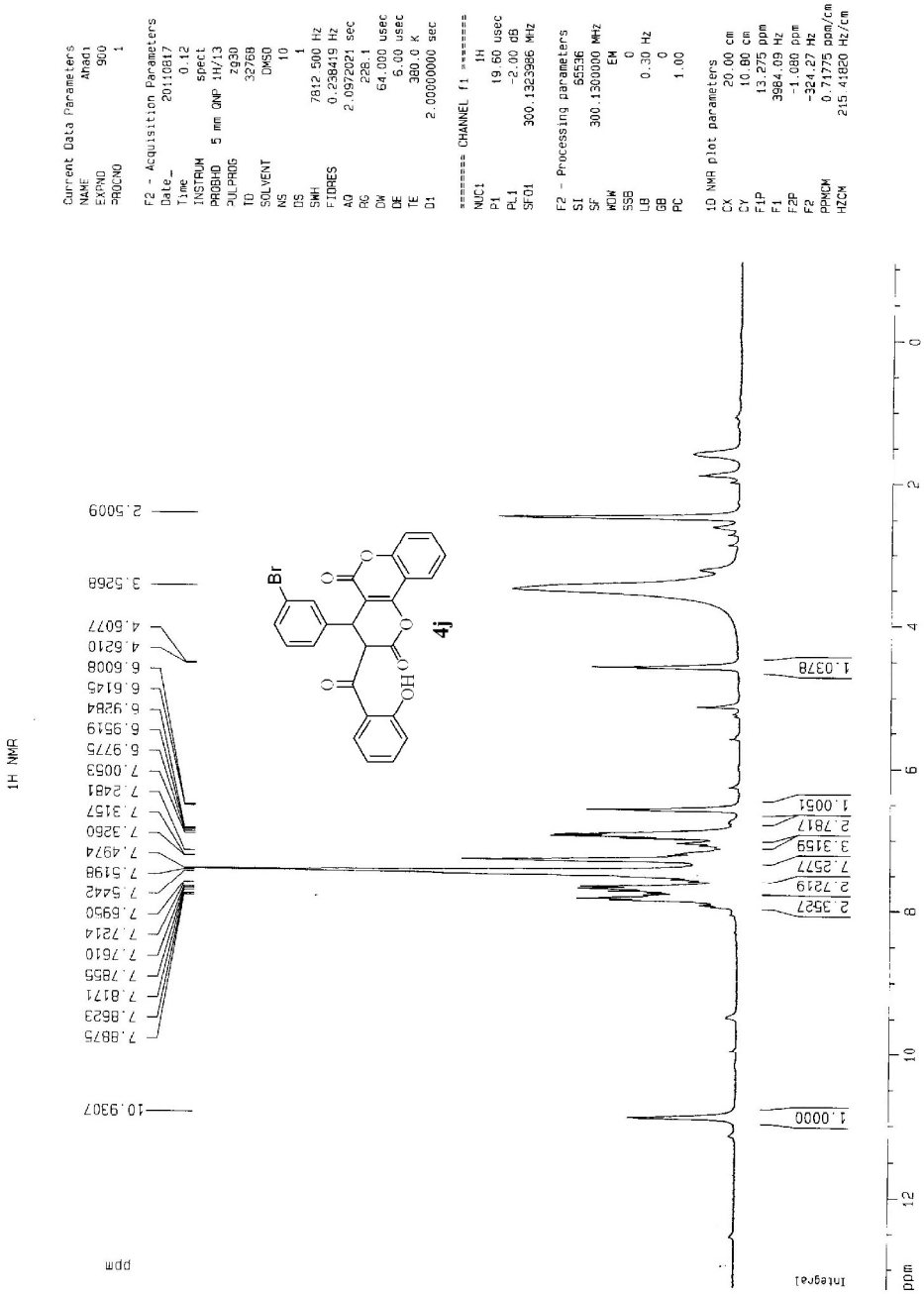

1H NMR
4h
ppm
Integral

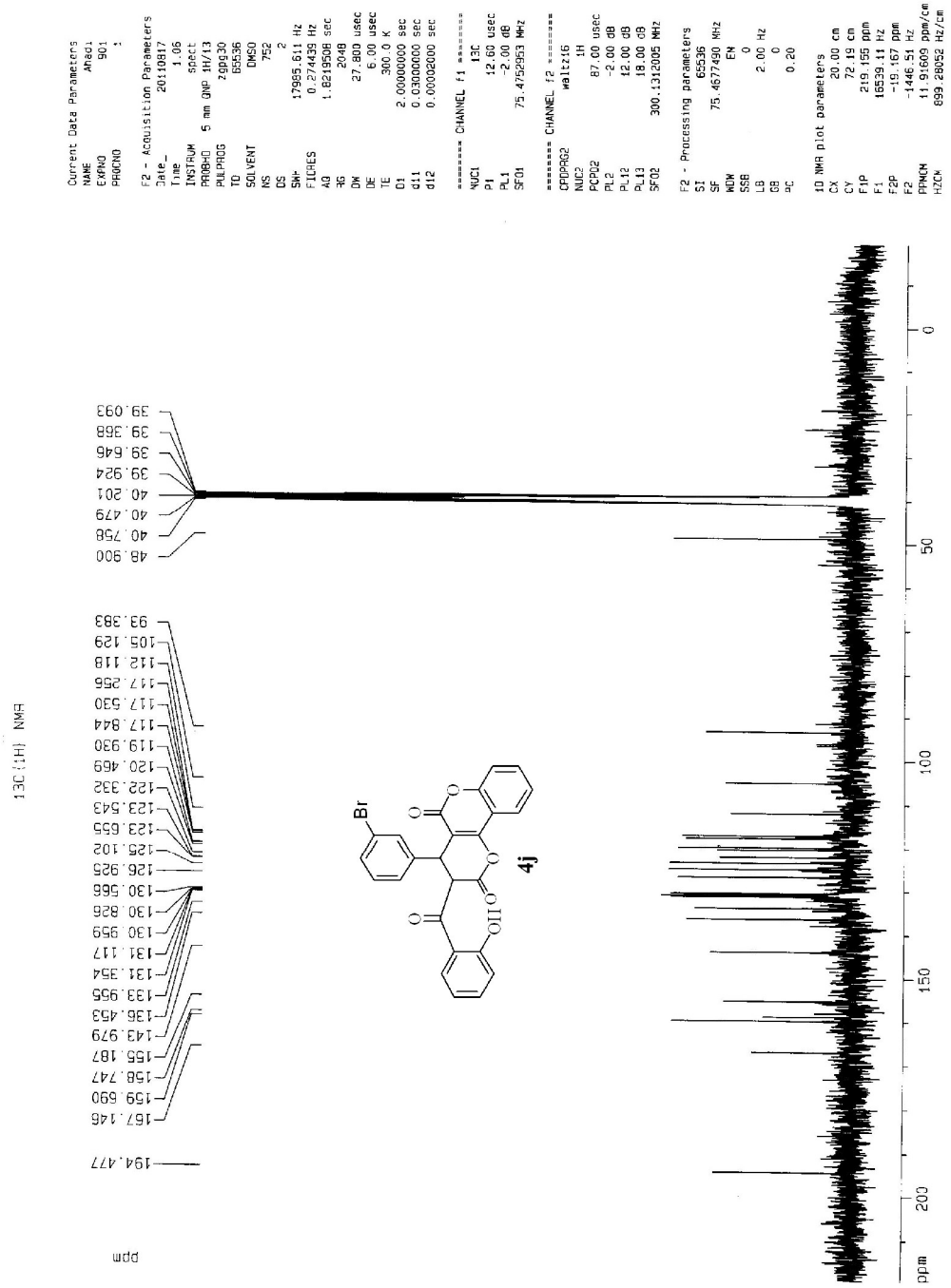

4h
13C{1H} NMR
ppm

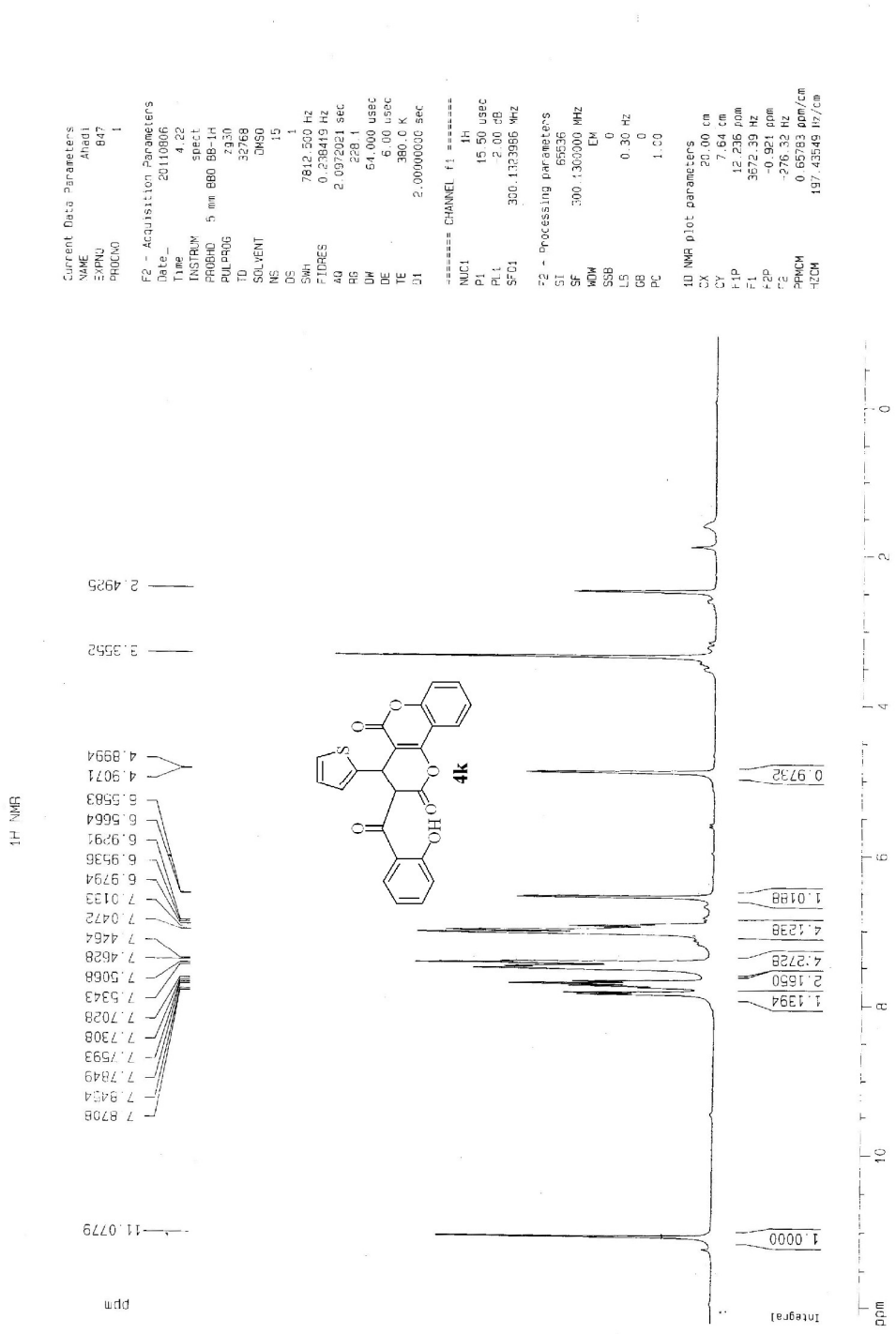


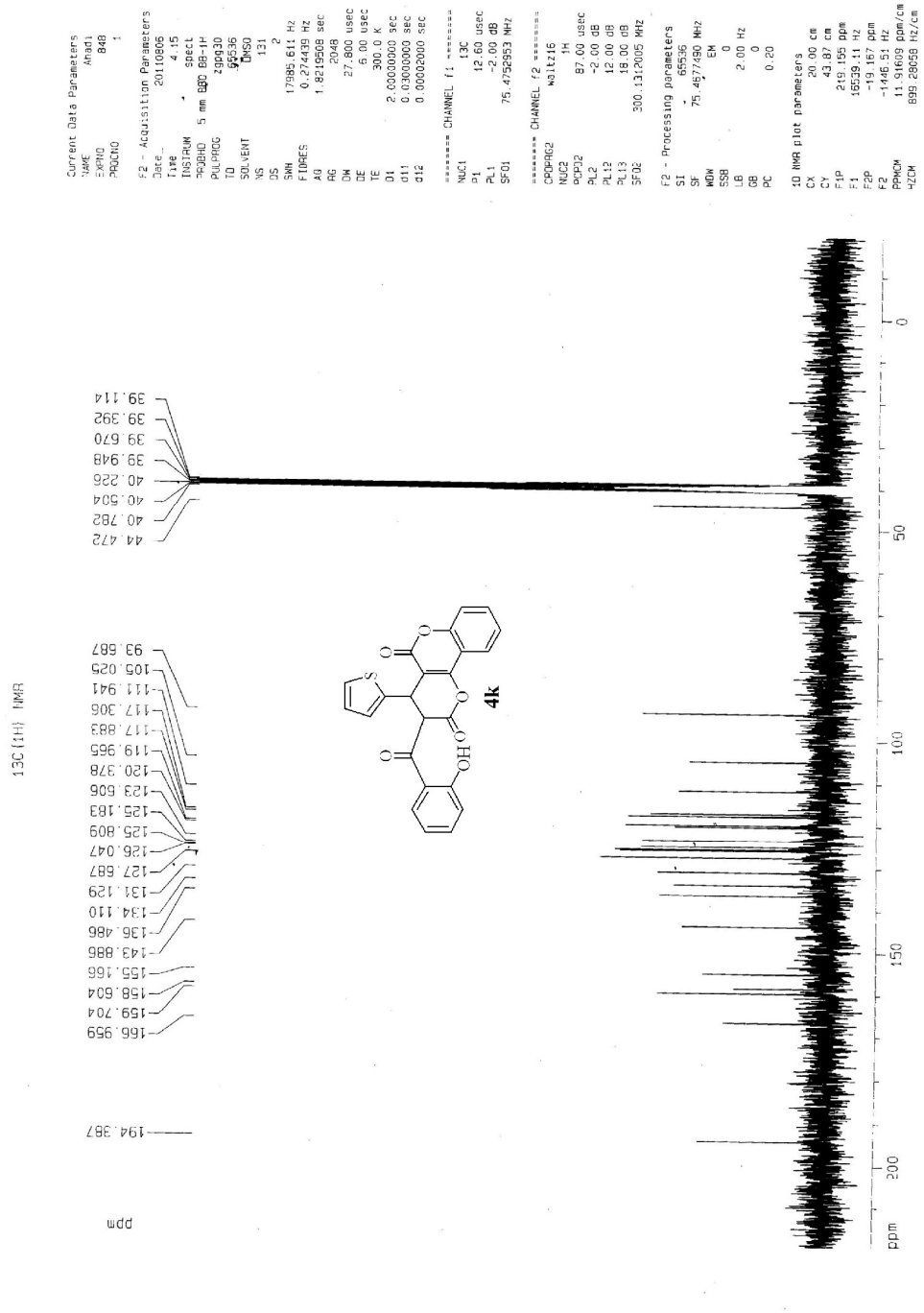


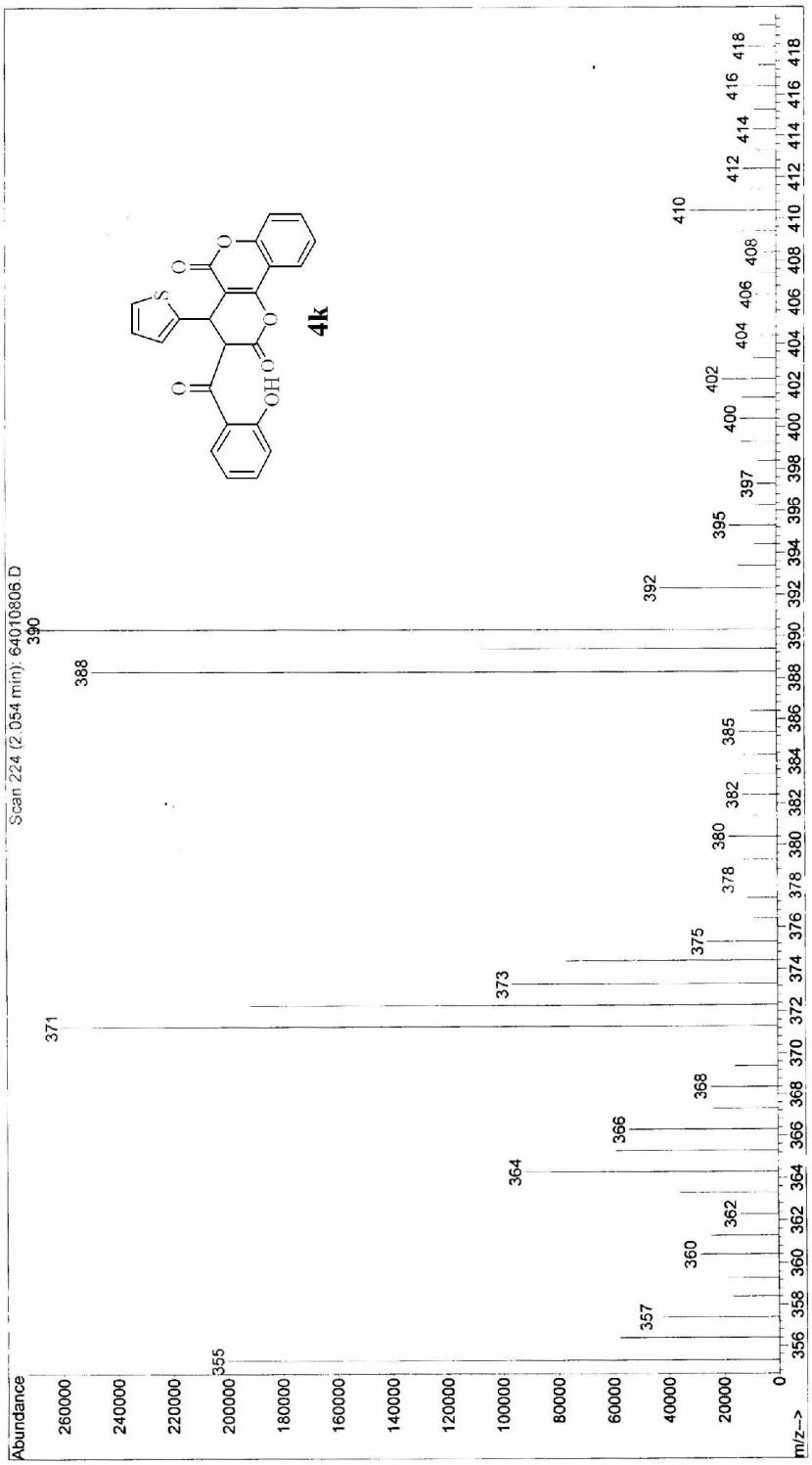

13C{1H} NMR
193.936
167.396
159.588
158.725
155.245
148.306
143.628
136.455
134.644
134.052
131.121
130.708
125.140
123.696
122.855
120.598
119.984
117.793
117.290
112.081
104.958
93.423
48.814
40.768
40.488
40.211
39.933
39.655
39.377
39.099
NO2
OH
4i
ppm 200 150 100 50 0
Current Data Parameters
NAME Ahadi
EXPNO 917
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110902
Time 16.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 203
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 72.29 cm
F1P 219.155 ppm
F1 16539.11 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm

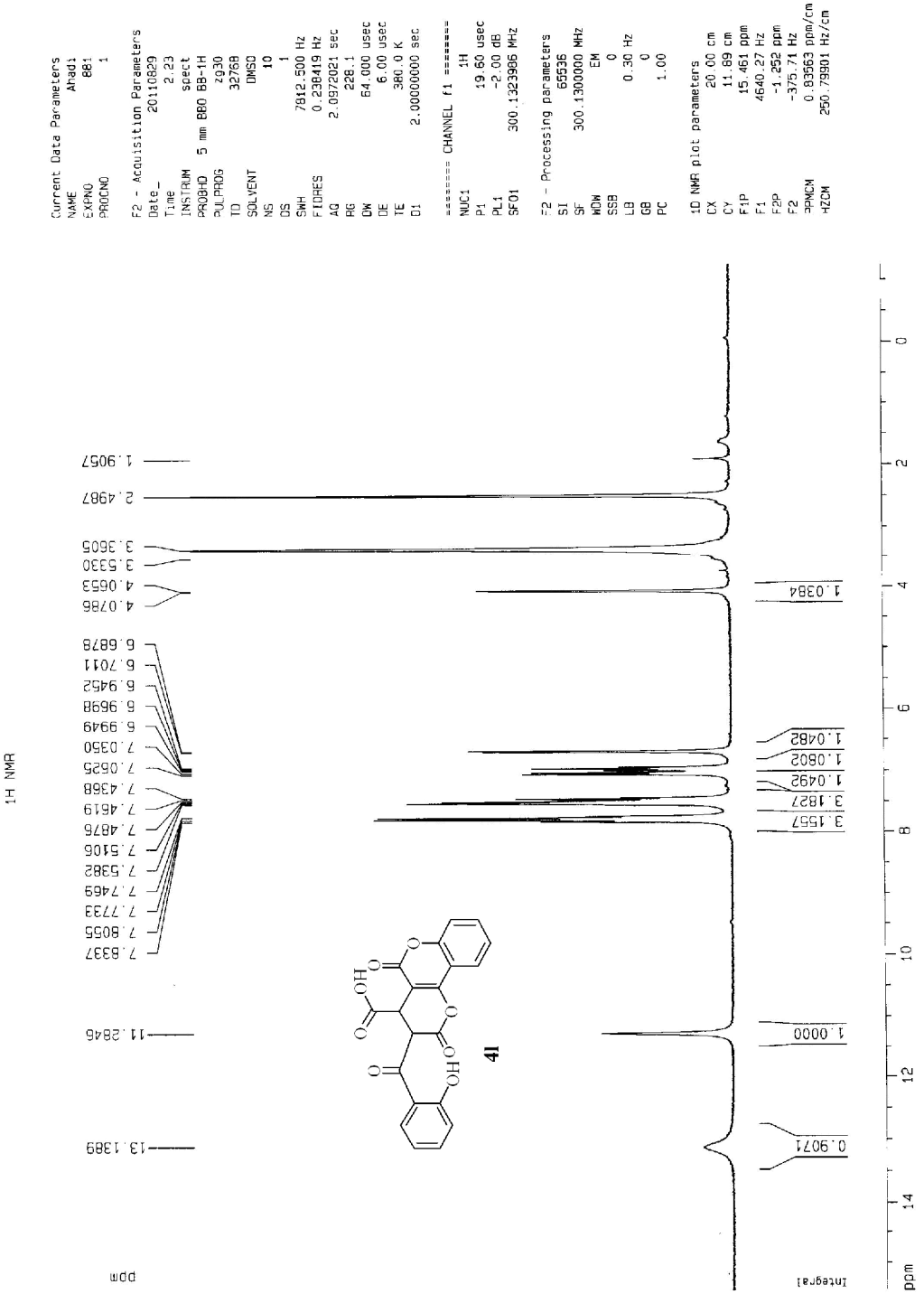

1H NMR
Current Data Parameters
NAME Ahad1
EXPNO 900
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110817
Time 0.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.80 cm
F1P 13.275 ppm
F1 3984.09 Hz
F2P -1.080 ppm
F2 -324.27 Hz
PPMCM 0.71775 ppm/cm
HZCM 215.41820 Hz/cm
Br
O
OH
4j
10.9307
7.8875
7.8629
7.8171
7.7855
7.7610
7.7214
7.6950
7.5442
7.5198
7.4974
7.3260
7.3157
7.2481
7.0053
6.9775
6.9519
6.9284
6.6145
6.6008
4.6210
4.6077
3.5268
2.5009
1.0000
3.3527
2.7219
2.7577
2.3159
2.7817
1.0051
1.0378
Integral
ppm
12
10
8
6
4
2
0

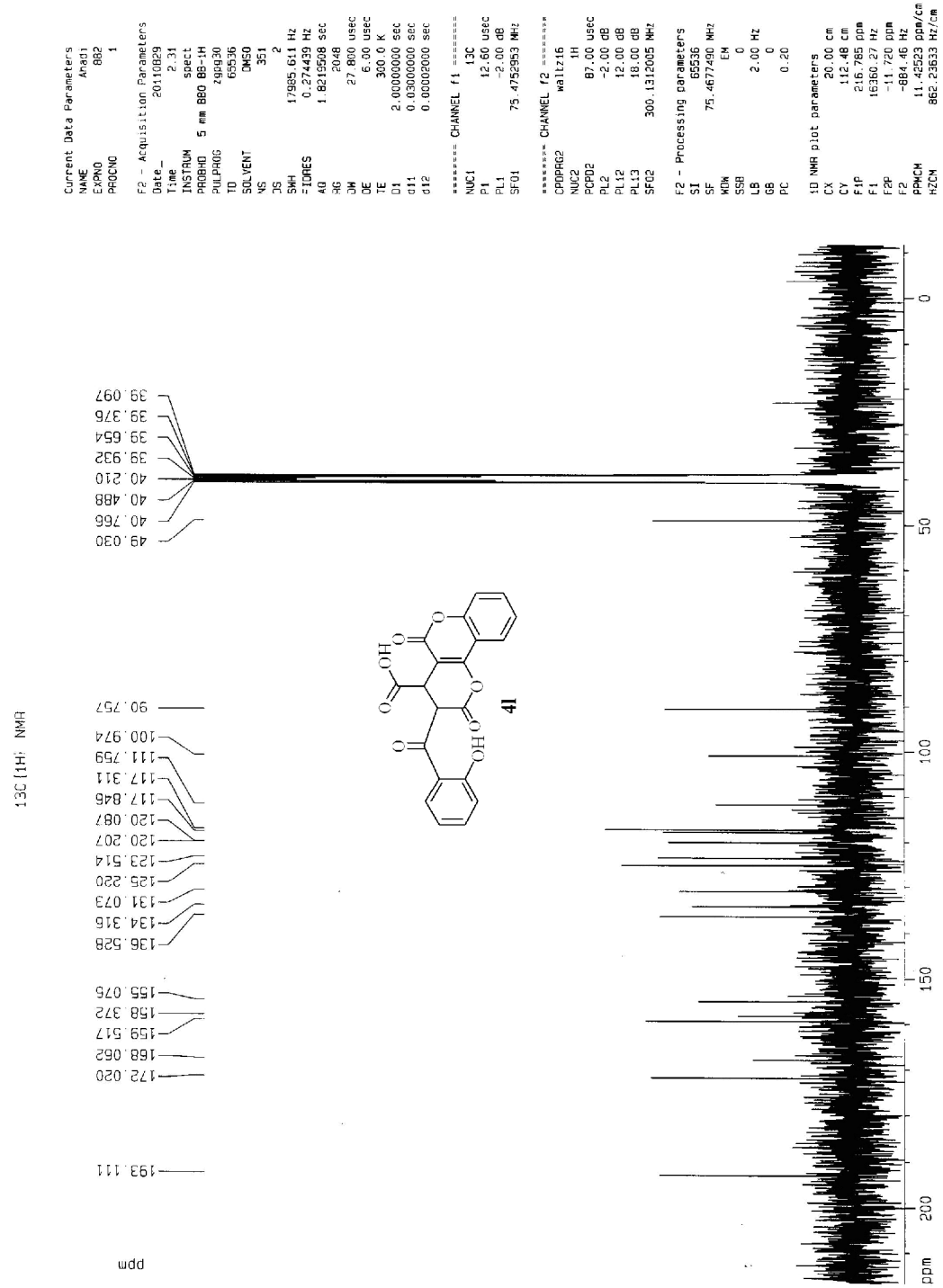

13C{1H} NMR
4j
Br
O
O
O
O
O
O
ppm
0
50
100
150
200
39.093
39.368
39.646
39.924
40.201
40.479
40.758
48.900
93.383
105.129
112.118
117.256
117.530
117.844
119.930
120.469
122.332
123.543
123.655
125.102
126.925
130.566
130.826
130.959
131.117
131.354
133.955
136.453
143.979
155.187
158.747
159.690
167.146
194.477

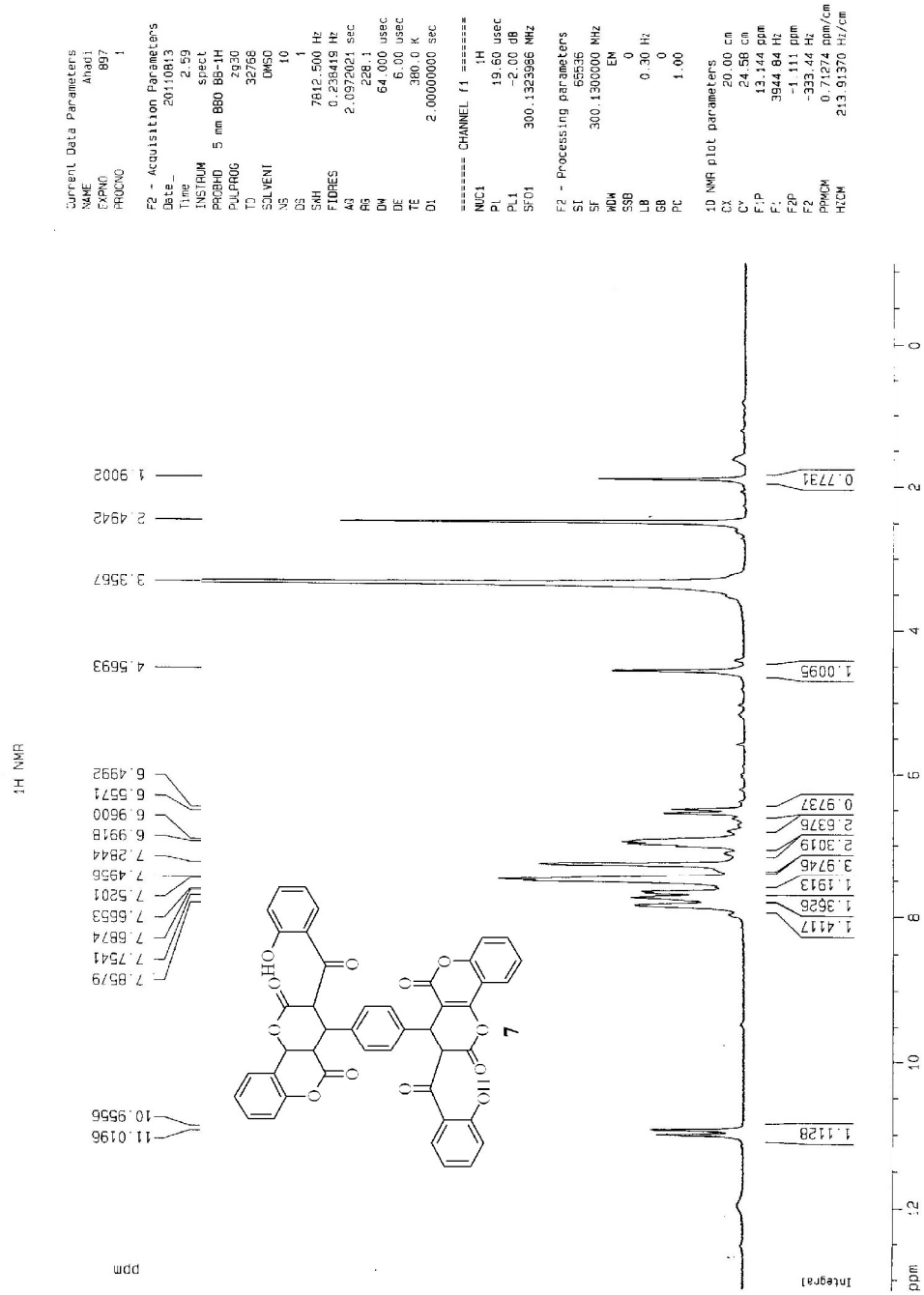

Current Data Parameters
NAME Ahadi
EXPNO 847
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110806
Time 4.22
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG 2g30
TD 32768
SOLVENT DMSO
NS 15
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 2.00 dB
SFO1 300.1333966 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 7.64 cm
F1P 12.236 ppm
F1 3672.39 Hz
F2P -0.921 ppm
F2 -276.32 Hz
PPMCM 0.65783 ppm/cm
HZCM 197.43549 Hz/cm
4k
1H NMR
ppm
Integral
2.4925
3.3552
4.8994
4.9071
5.5583
6.5664
6.9291
6.9536
6.9794
7.0133
7.0472
7.4464
7.4628
7.5068
7.5343
7.7028
7.7308
7.7593
7.7849
7.8424
7.8708
11.0779
0.9732
1.0188
4.1238
4.2728
2.1650
1.1394
1.0000

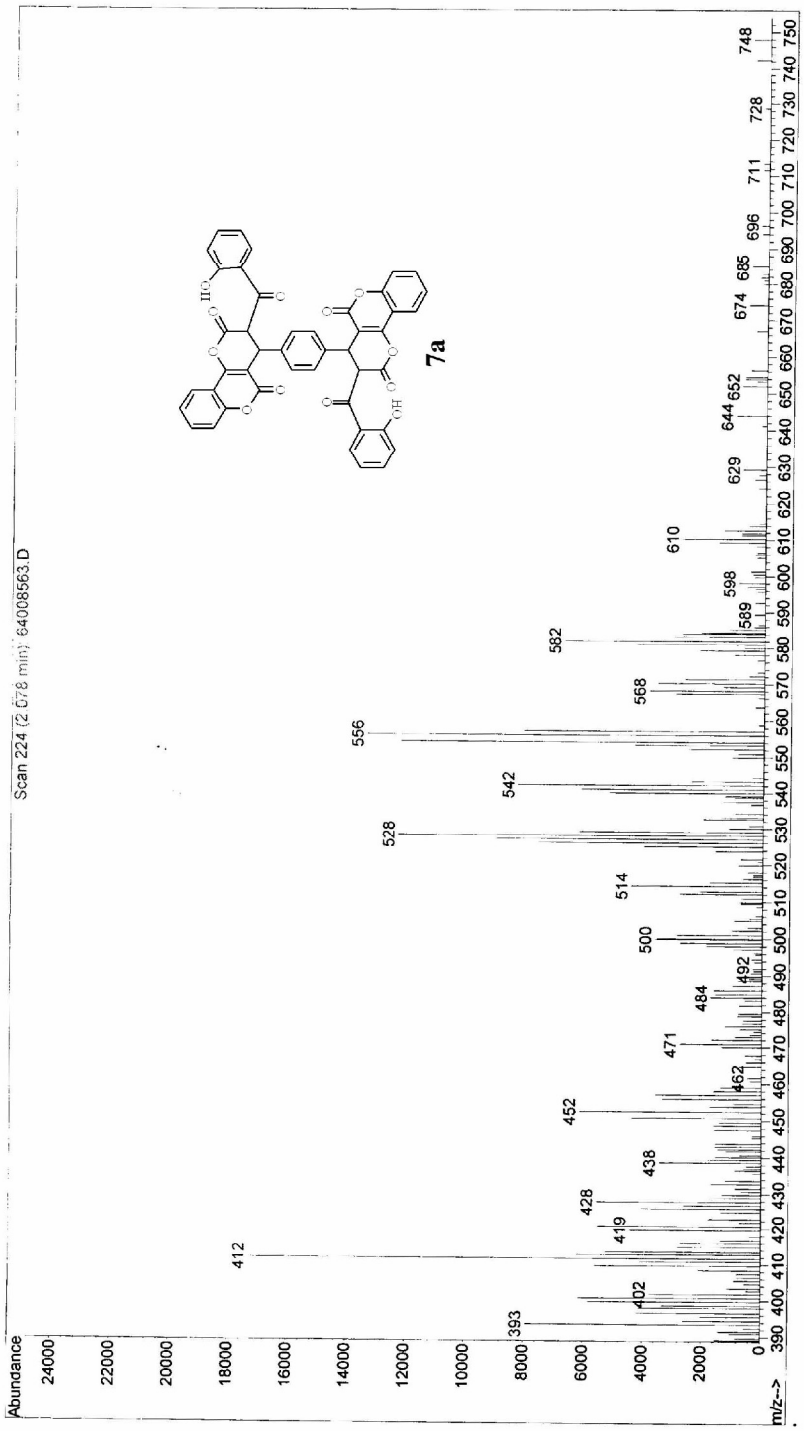

4k
13C{1H} NMR
ppm

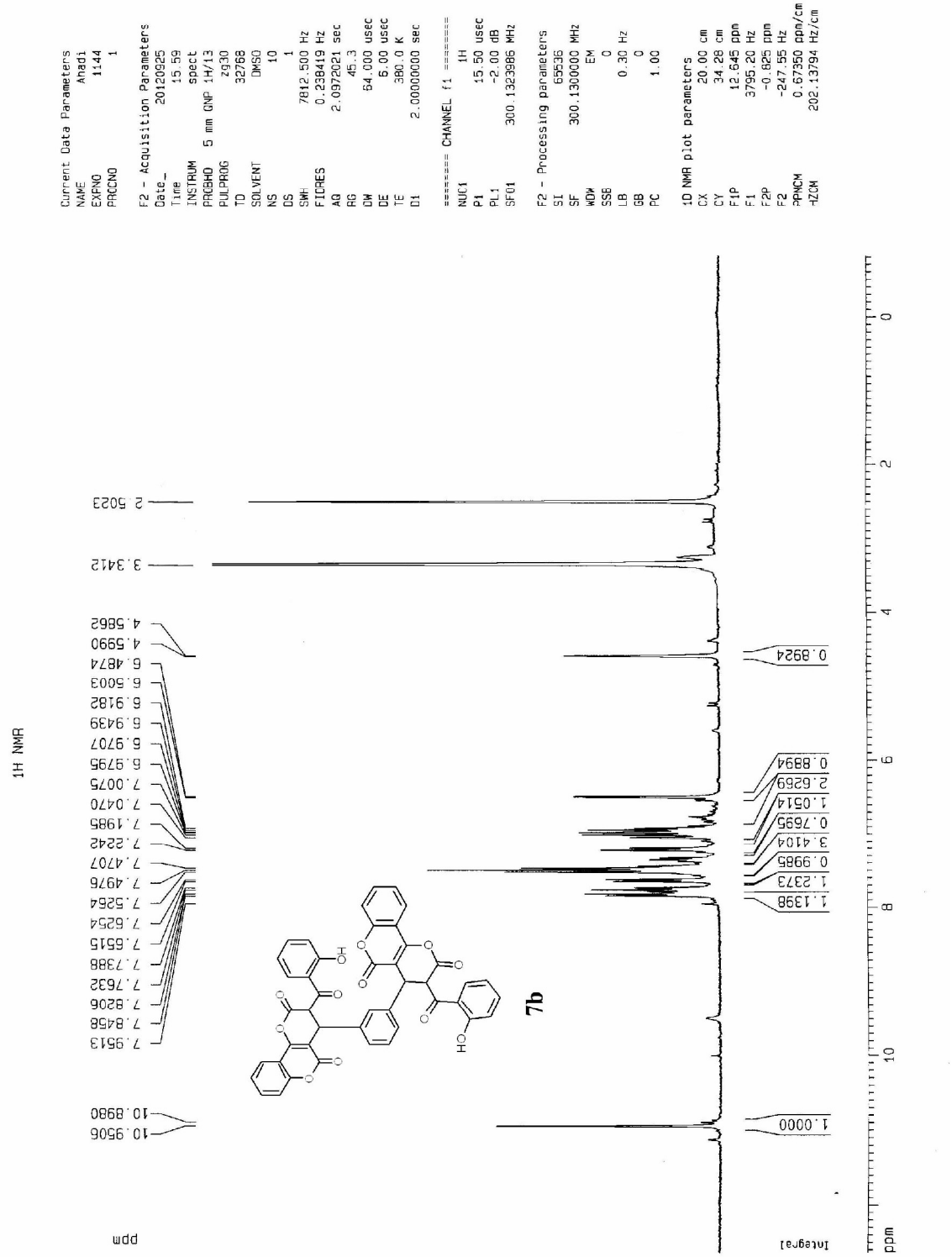


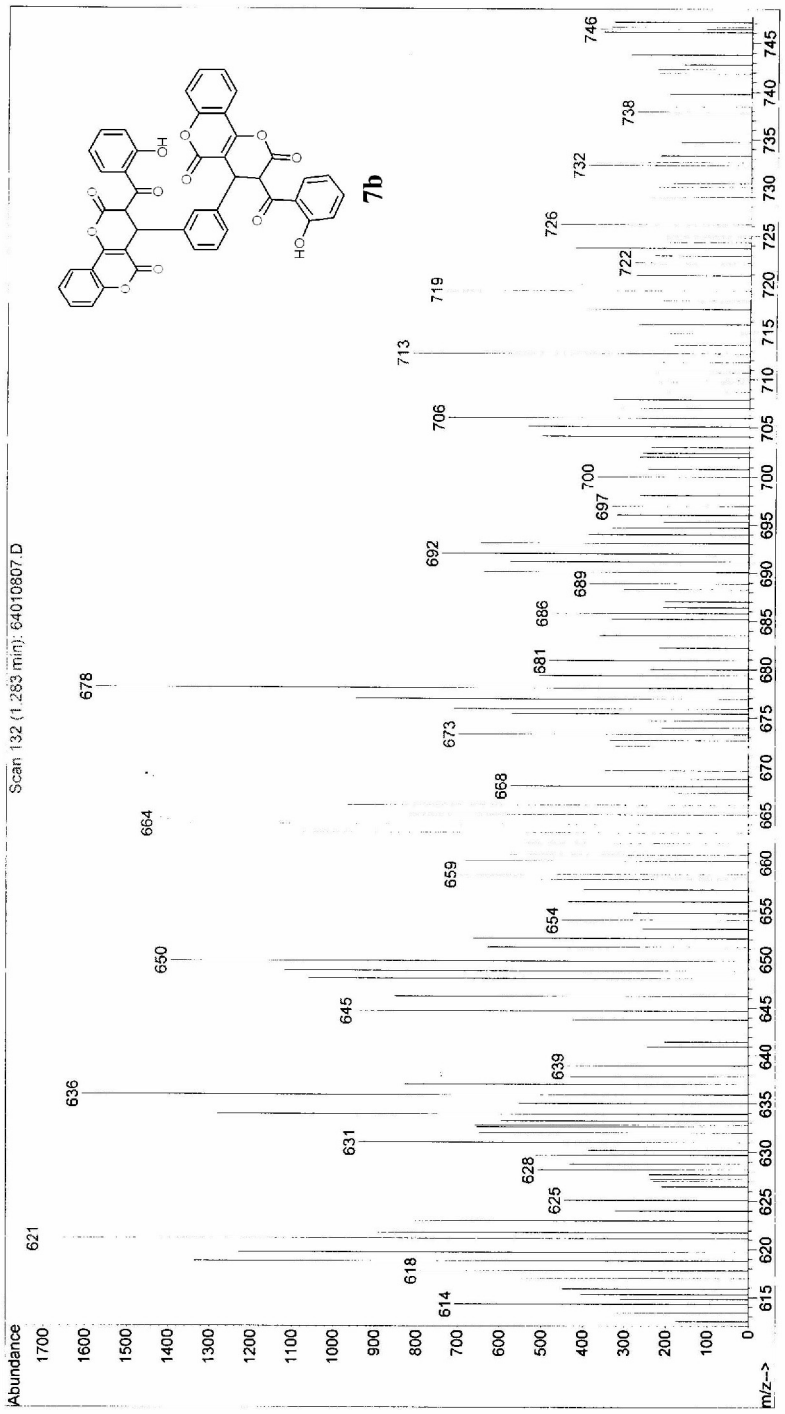

Current Data Parameters
NAME Ahadi
EXPNO 881
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110829
Time 2.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG 2930
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 11.89 cm
F1P 15.461 ppm
F1 4640.27 Hz
F2P -1.252 ppm
F2 -375.71 Hz
PPMCM 0.83563 ppm/cm
HZCM 250.79301 Hz/cm
1H NMR
ppm
Integral
1.9057
2.4987
3.3605
3.5330
4.0653
4.0786
6.6878
6.7011
6.9452
6.9698
6.9949
7.0350
7.0625
7.4368
7.4619
7.4876
7.5106
7.5382
7.7469
7.7733
7.8055
7.8337
11.2846
13.1389
1.0384
1.0482
1.0802
1.0492
3.1827
3.1557
1.0000
0.9071
41

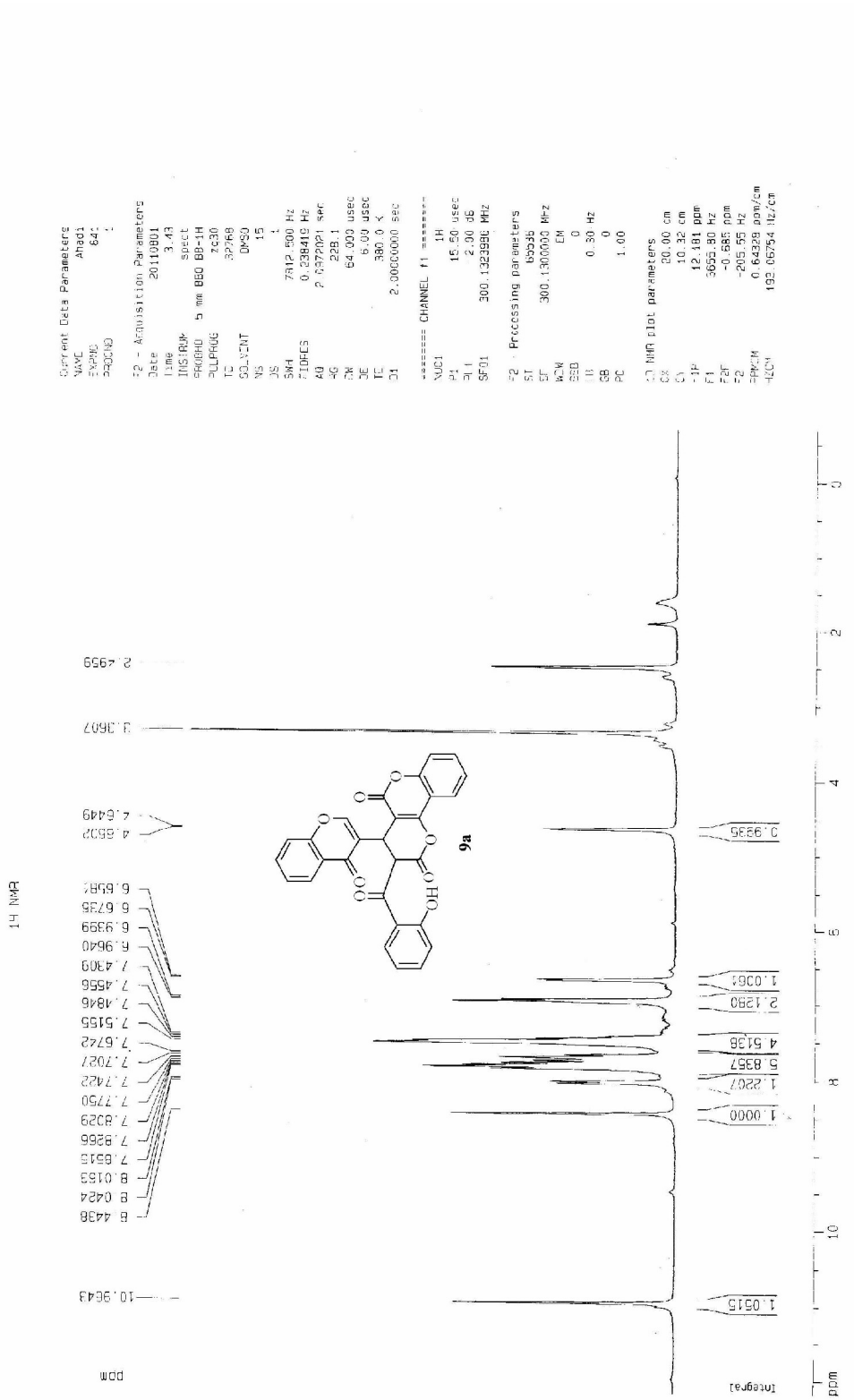

Current Data Parameters
NAME Anadi
EXPNO 882
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110829
Time 2.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 351
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 112.48 cm
F1P 216.785 ppm
F1 16360.27 Hz
F2P -11.720 ppm
F2 -884.46 Hz
PPMCM 11.42523 ppm/cm
HZCM 862.23633 Hz/cm
13C{1H} NMR
41
ppm
0
50
100
150
200
39.097
39.376
39.654
39.932
40.210
40.488
40.766
49.030
90.757
100.974
111.759
117.311
117.846
120.087
120.207
123.514
125.220
131.073
134.316
136.528
155.076
158.372
159.517
168.062
172.020
193.111

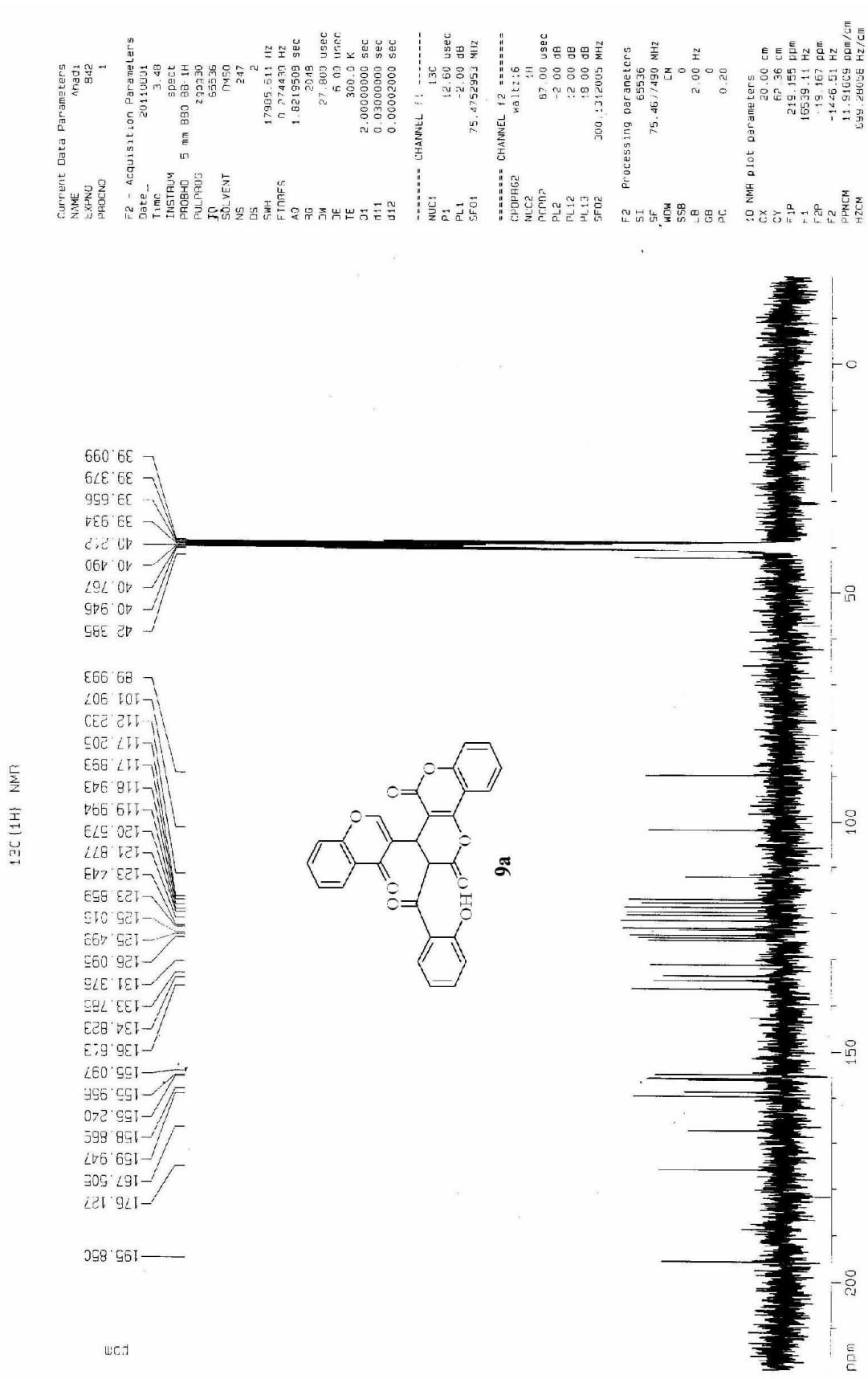


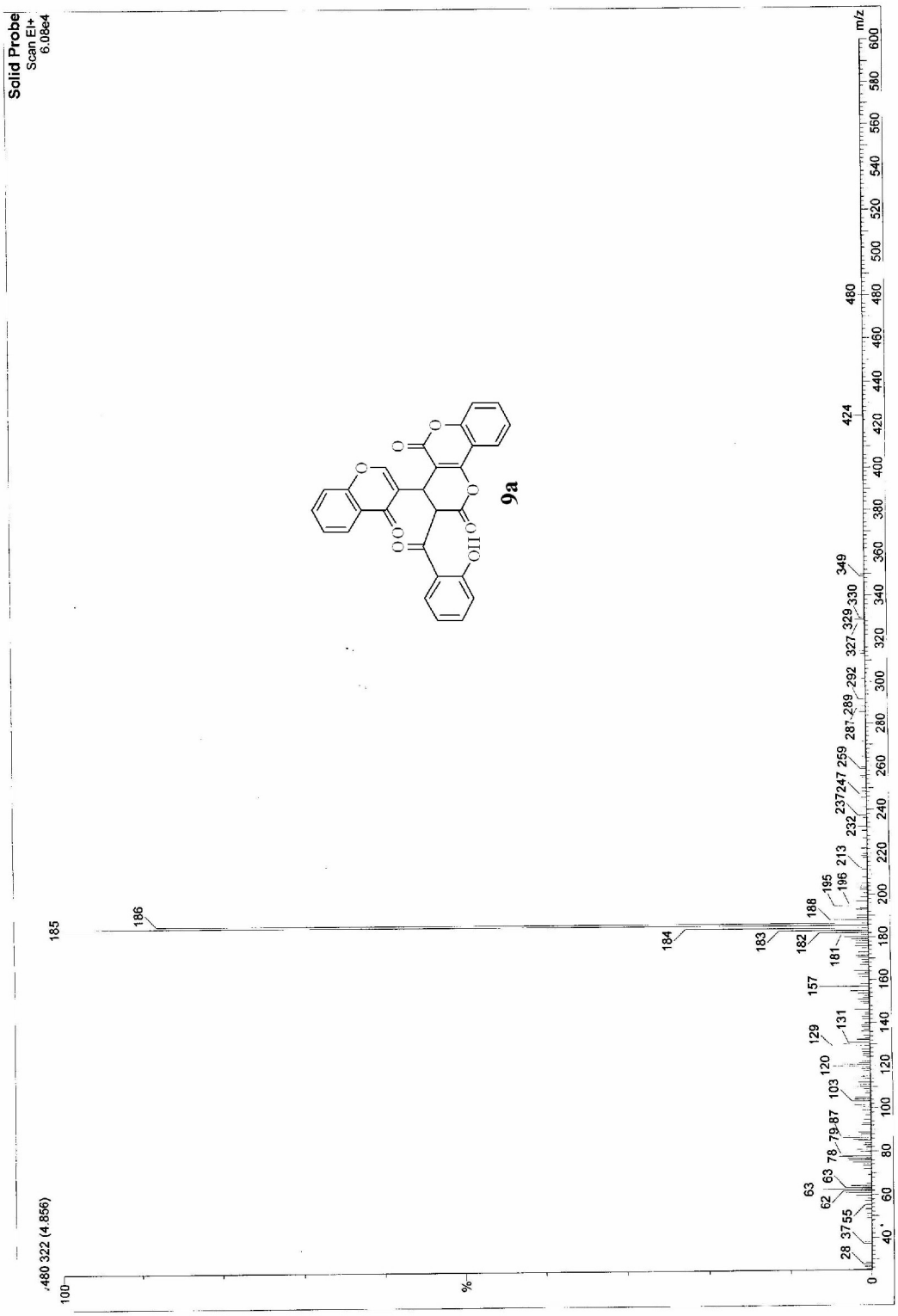

Scan 224 (2.678 min): 64008563.D
Abundance
m/z-->
7a

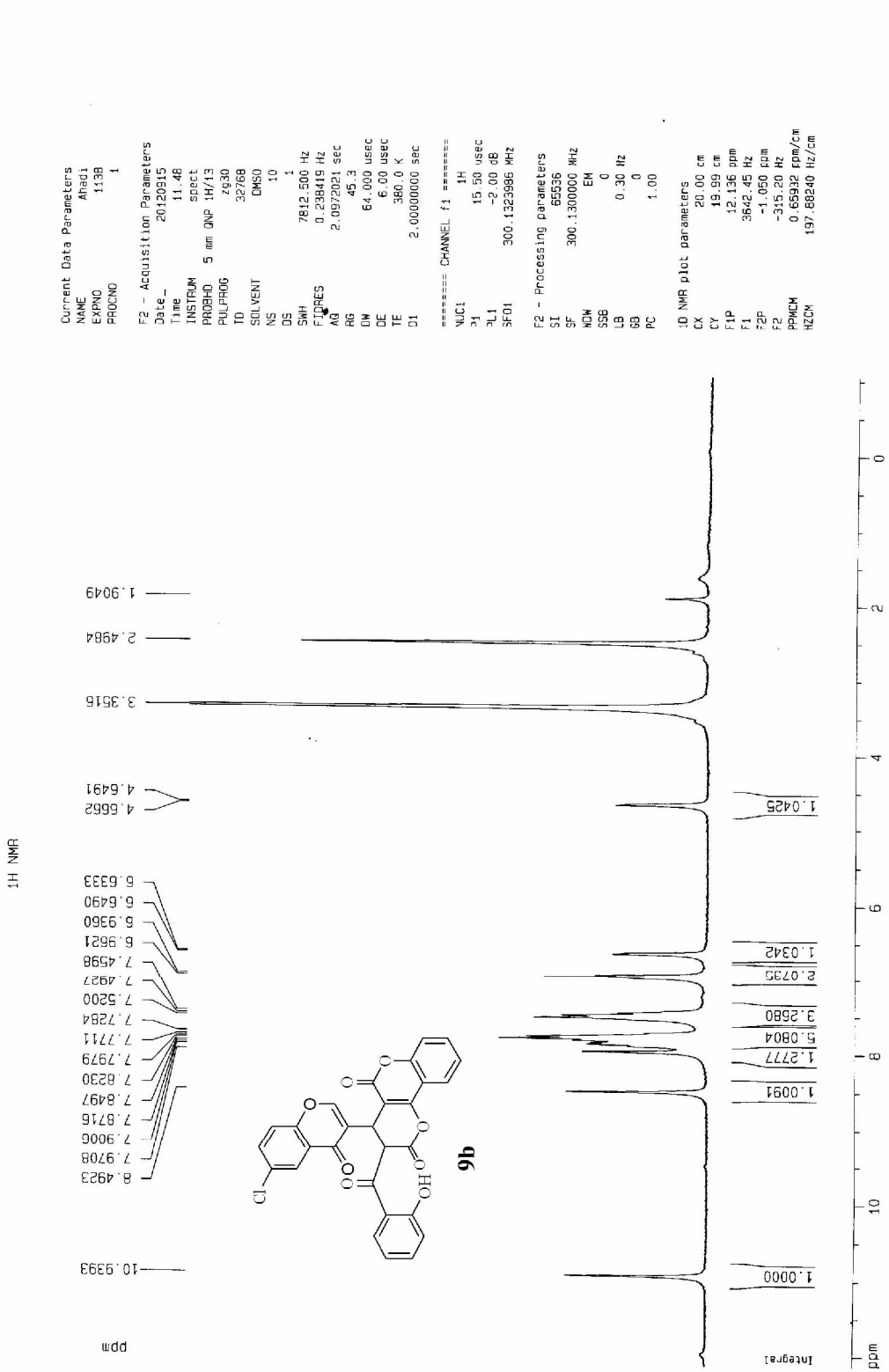


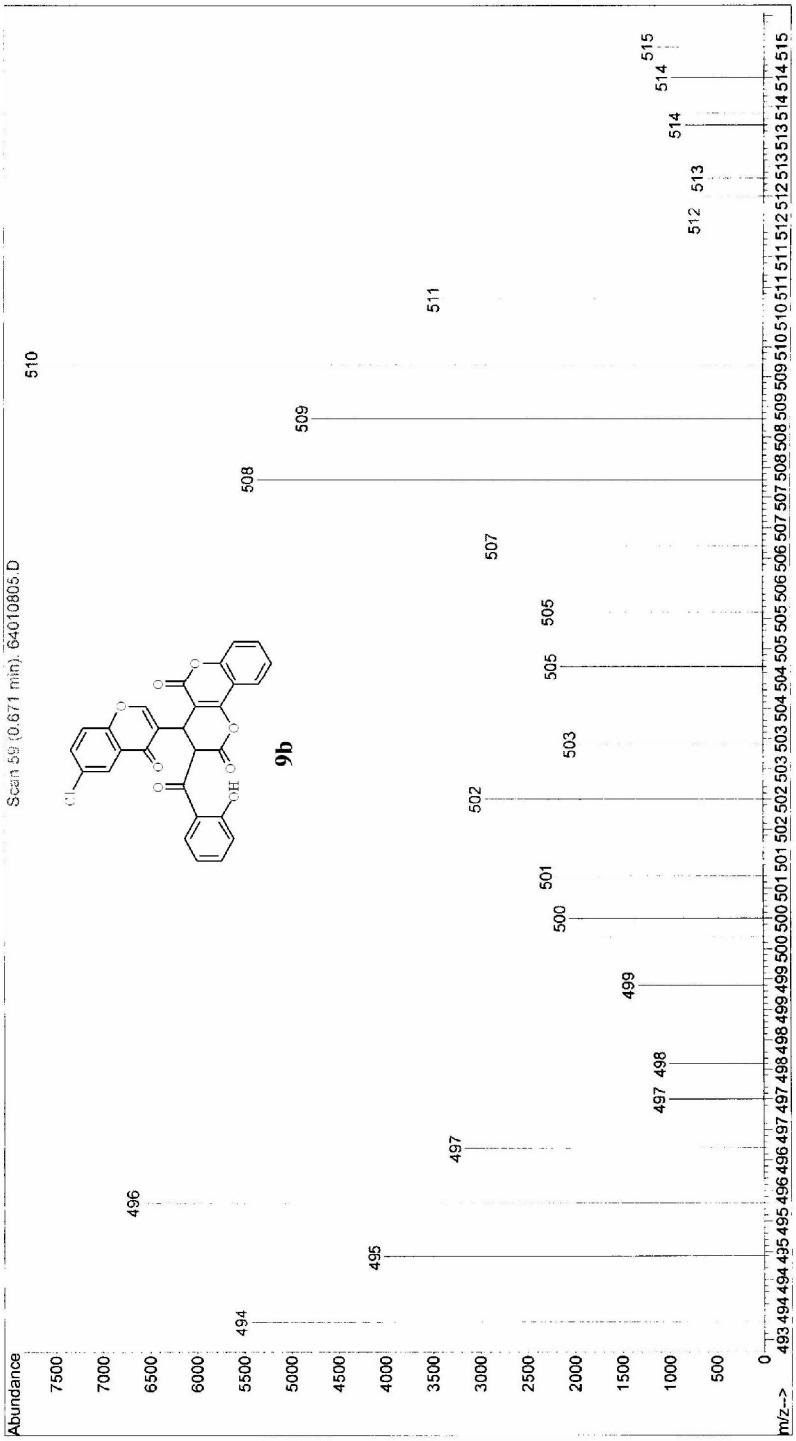


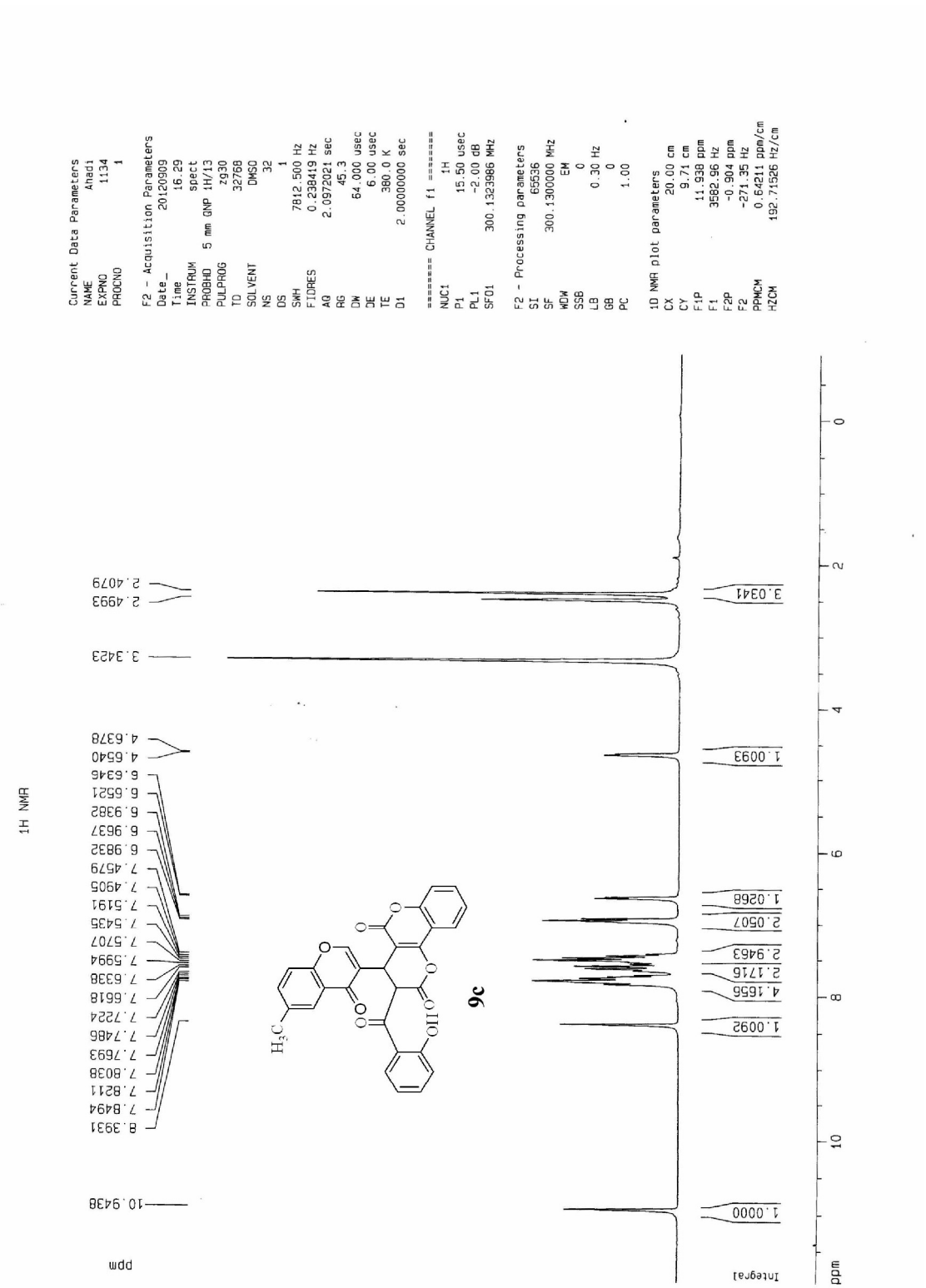

1H NMR
9a
ppm
Integral

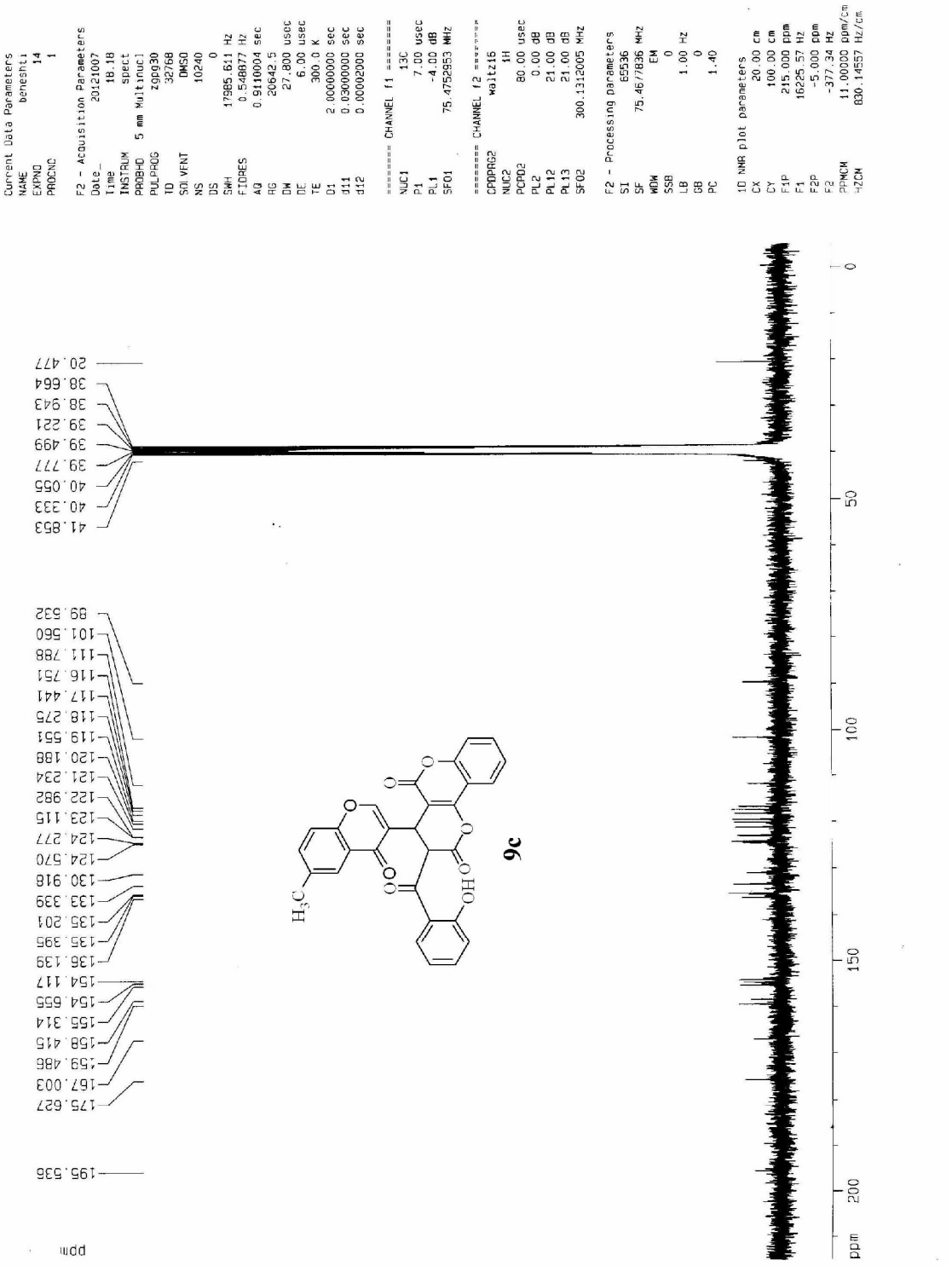

13C (1H) NMR
9a
ppm

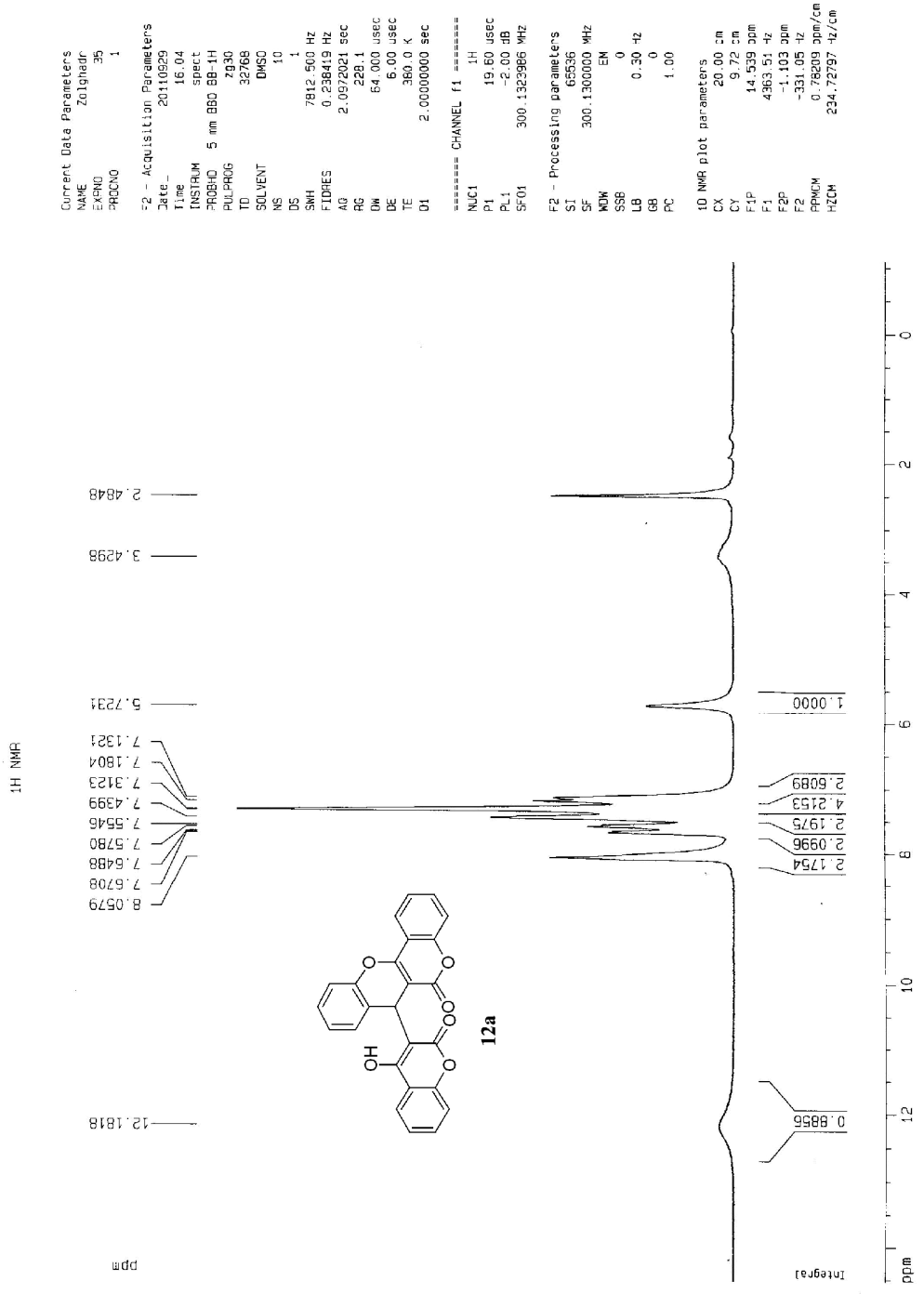

Solid Probe
Scan EI+
6.08e4
9a
m/z
%
480 322 (4.856)

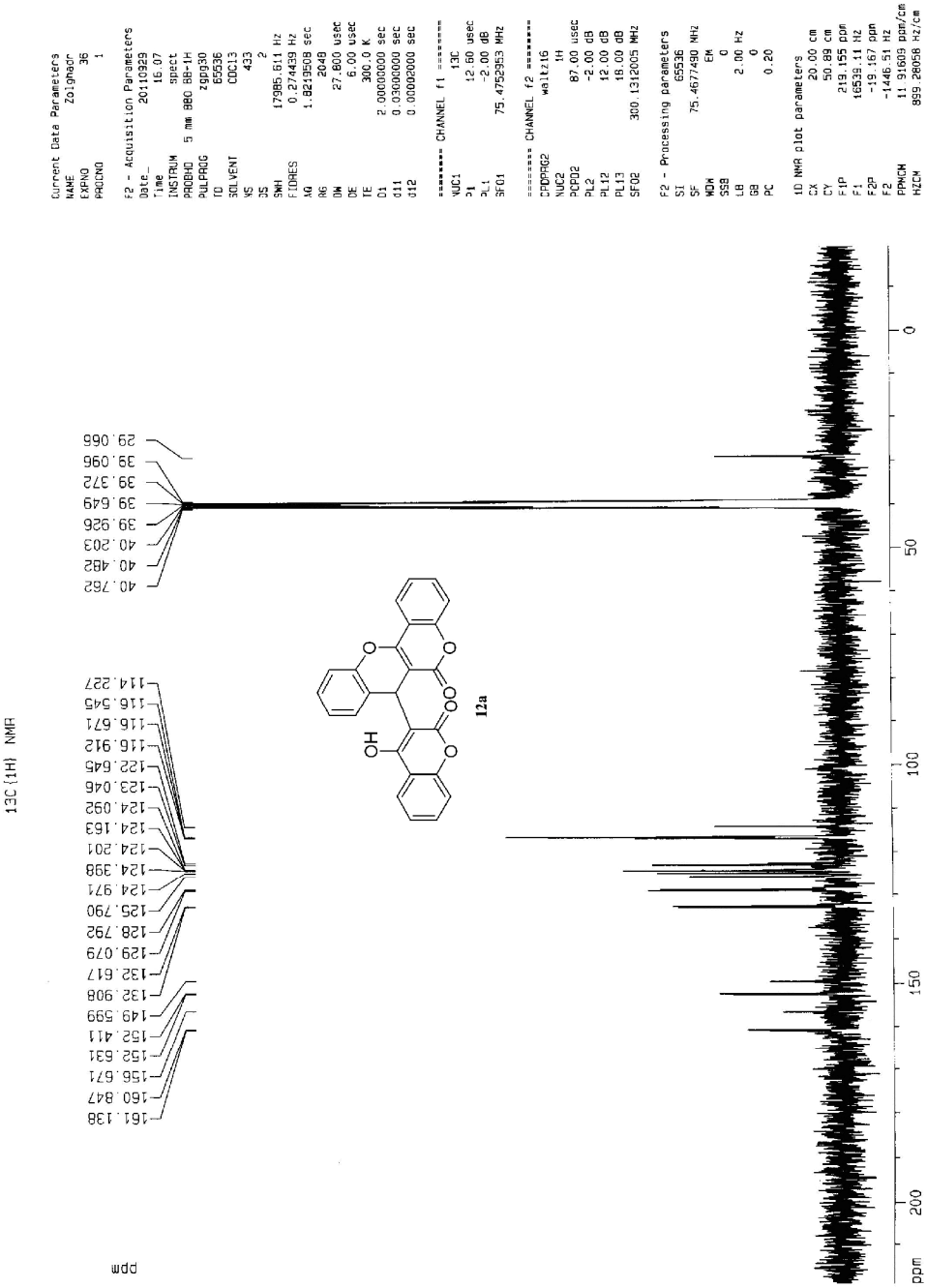

1H NMR
Current Data Parameters
NAME Ahad1
EXPNO 1138
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120915
Time 11.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 45.3
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 19.99 cm
F1P 12.136 ppm
F1 3642.45 Hz
F2P -1.050 ppm
F2 -315.20 Hz
PPMCM 0.65932 ppm/cm
HZCM 197.88240 Hz/cm
10.9393
8.4923
8.0097
7.9708
7.9006
7.8716
7.8497
7.8230
7.7979
7.7711
7.7284
7.5200
7.4927
7.4598
6.9821
6.9360
6.6490
6.6333
4.6562
4.6491
3.3516
2.4984
1.9049
1.0000
1.0091
1.2777
5.0804
3.2680
1.0342
2.0735
1.0425
Integral
ppm
9b
Cl
OH
O

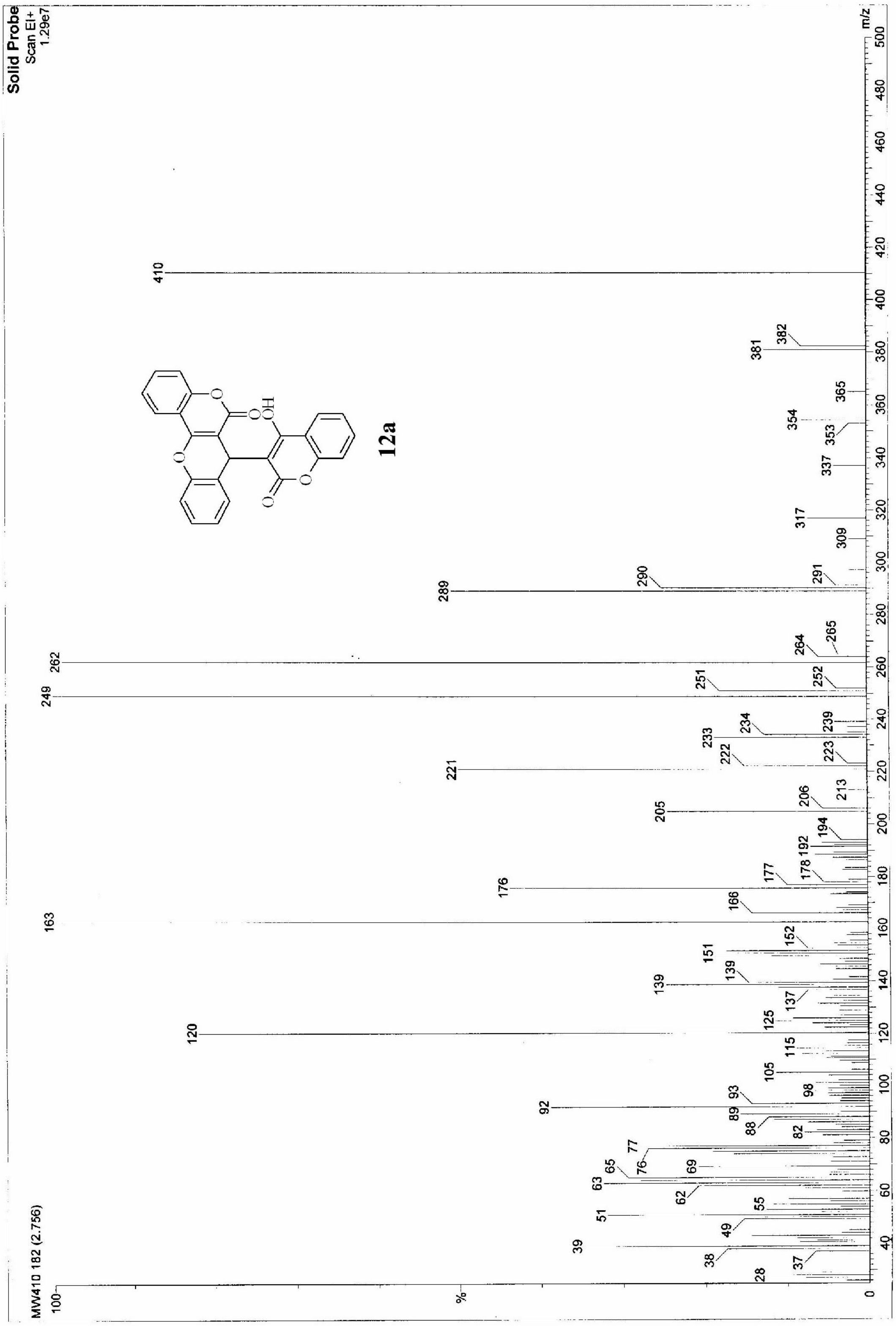

Scan 59 (0.671 min): 6401080S.D
Abundance
m/z-->
9b
Cl
OH
O


1H NMR
Current Data Parameters
NAME Ahadi
EXPNO 1134
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120909
Time 16.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 45.3
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.71 cm
F1P 11.938 ppm
F1 3582.96 Hz
F2P -0.904 ppm
F2 -271.35 Hz
PPMCM 0.64211 ppm/cm
HZCM 192.71526 Hz/cm
10.9438
8.3931
7.8494
7.8211
7.8038
7.7693
7.7486
7.7224
7.6618
7.6338
7.5994
7.5707
7.5435
7.5191
7.4905
7.4579
6.9832
6.9637
6.9382
6.6521
6.6346
4.6540
4.6378
3.3423
2.4993
2.4079
1.0000
4.1656
1.0092
2.9463
2.1716
2.0507
1.0268
1.0093
3.0341
Integral
ppm
9c

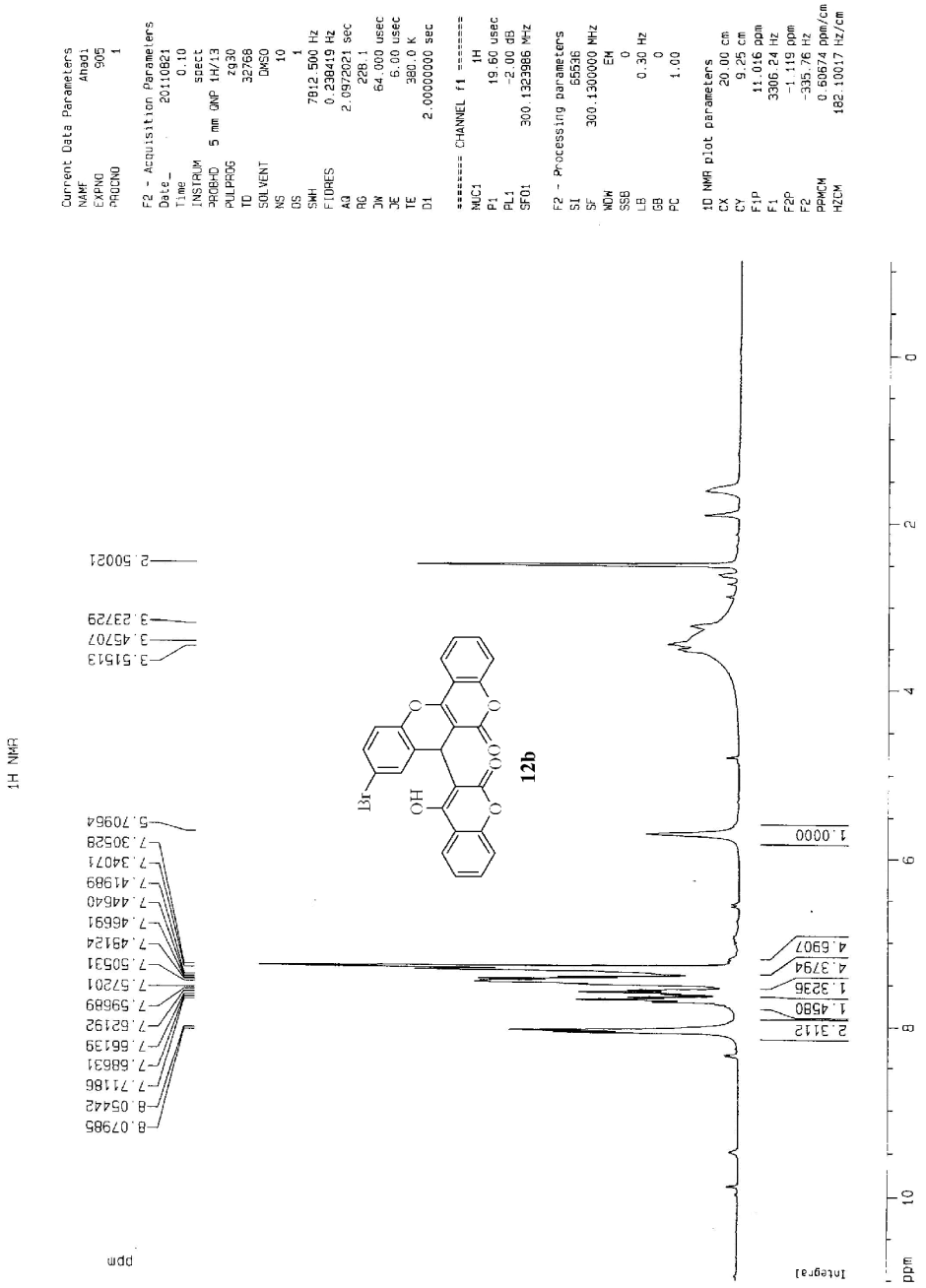


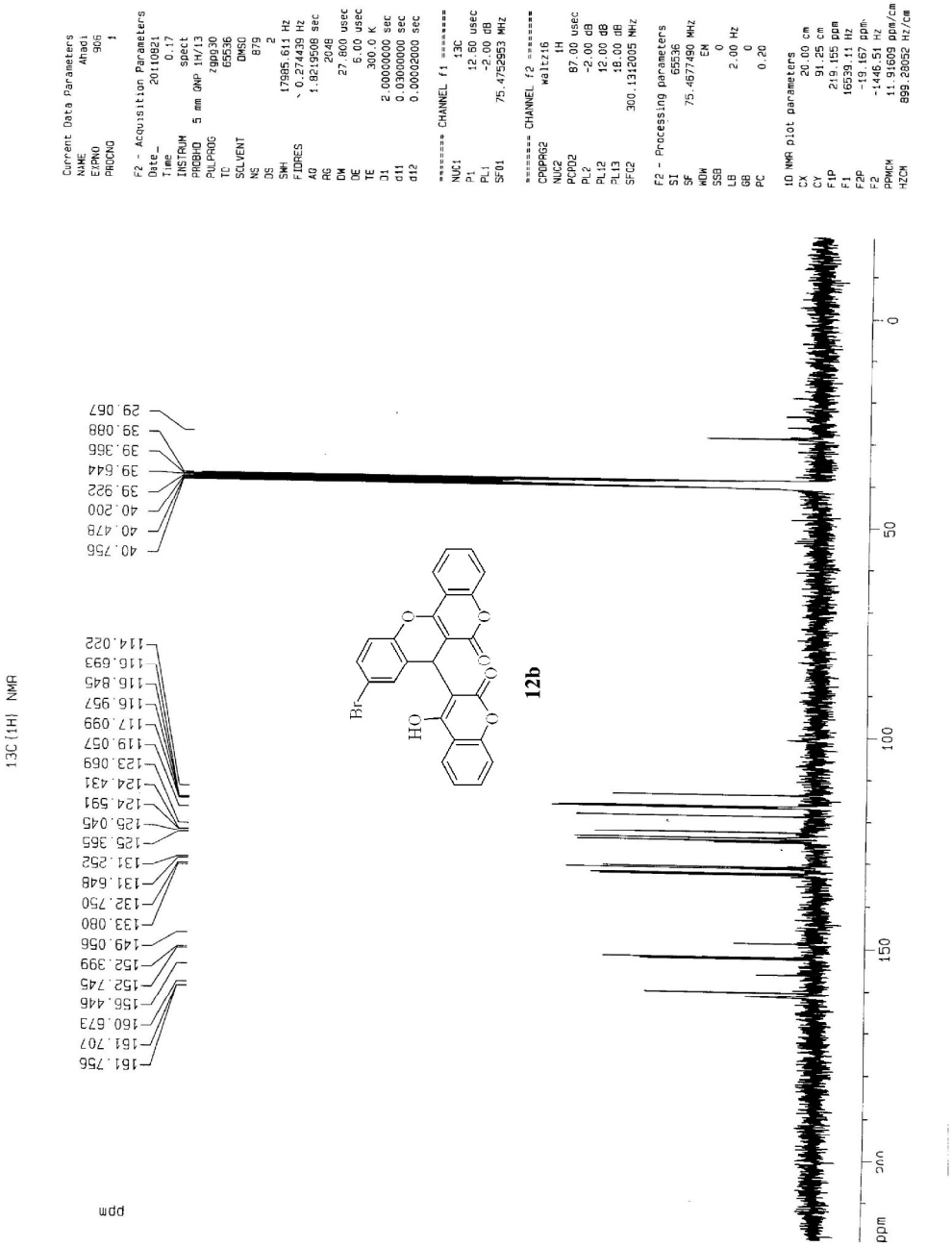

Current Data Parameters
NAME Zolghadr
EXPNO 35
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110929
Time 16.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 360.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.72 cm
F1P 14.539 ppm
F1 4363.51 Hz
F2P -1.103 ppm
F2 -331.05 Hz
PPMCM 0.78209 ppm/cm
HZCM 234.72797 Hz/cm
1H NMR
12.1818
8.0579
7.6708
7.6488
7.5780
7.5546
7.4399
7.3123
7.1804
7.1321
5.7231
3.4298
2.4848
1.0000
2.6089
4.2153
2.1975
2.0996
2.1754
0.8856
12a
OH
O
ppm
Integral

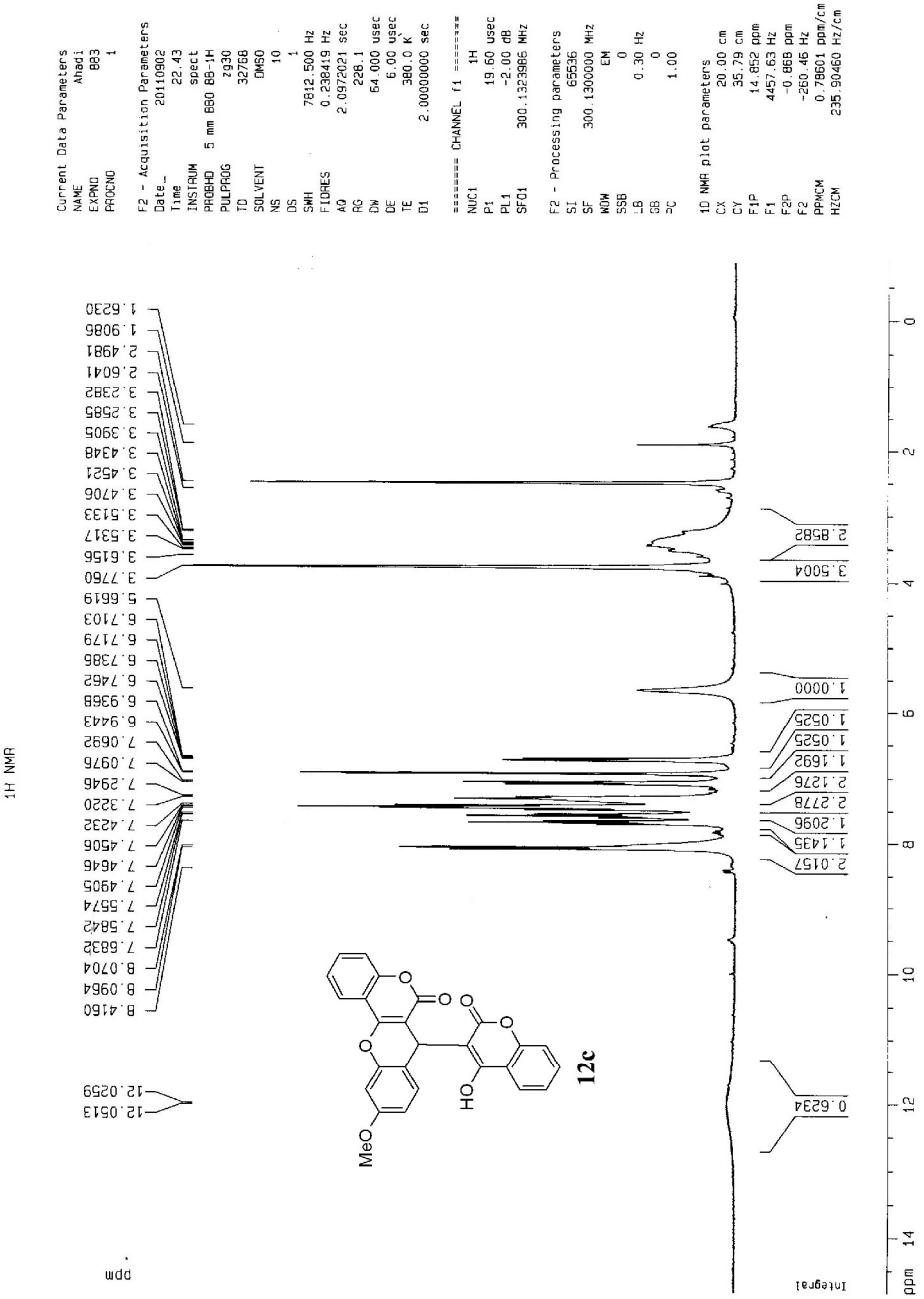

13C{1H} NMR
12a
ppm

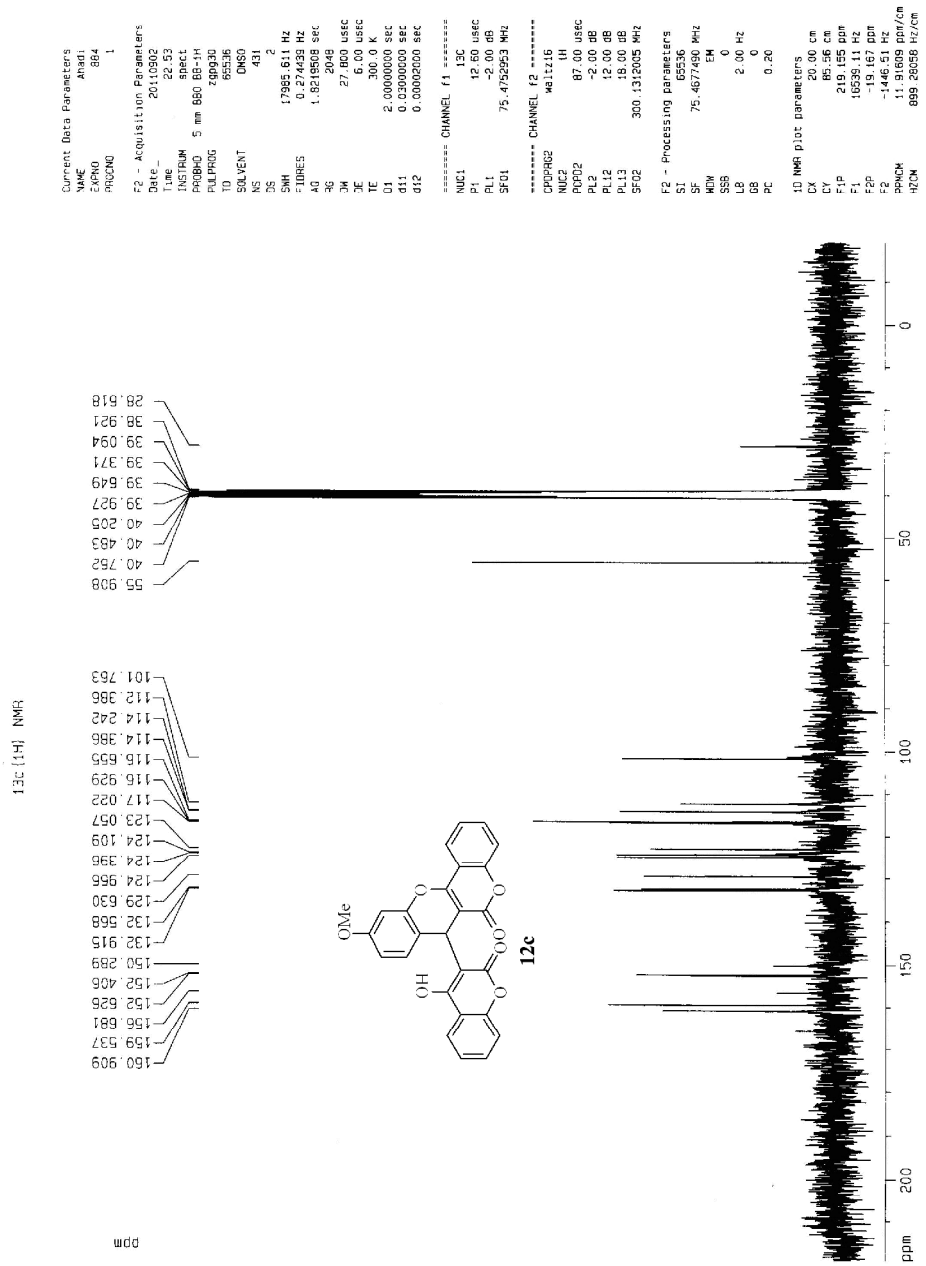

Solid Probe
Scan EI+
1.29e7
MW410 182 (2.756)
100
%
0
m/z
410
382
381
365
354
353
337
317
309
291
290
289
265
264
262
252
251
249
239
234
233
223
222
221
213
206
205
194
192
178
177
176
166
163
152
151
139
137
125
120
115
105
98
93
92
89
88
82
77
76
69
65
63
62
55
51
49
39
38
37
28
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
500

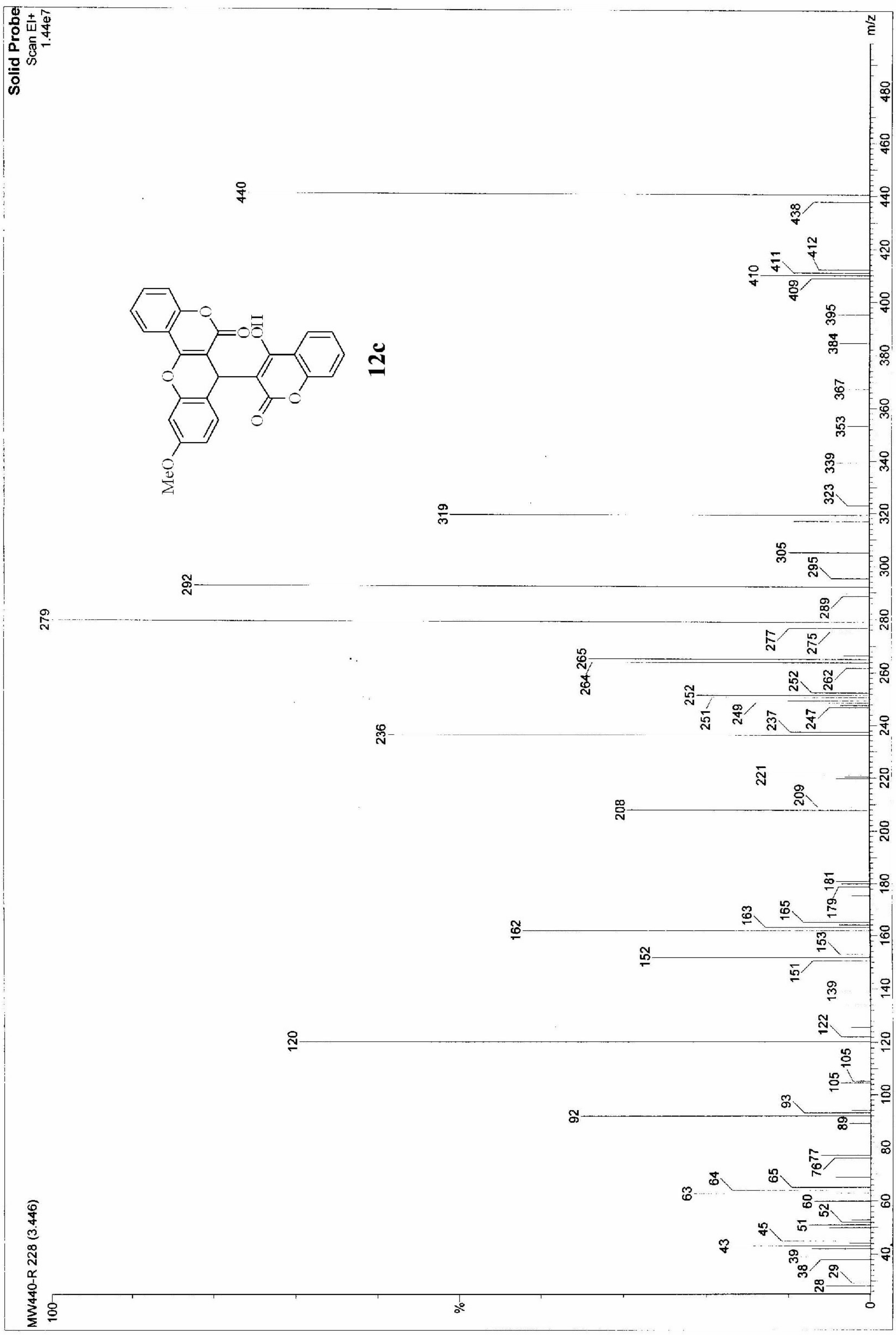
12a

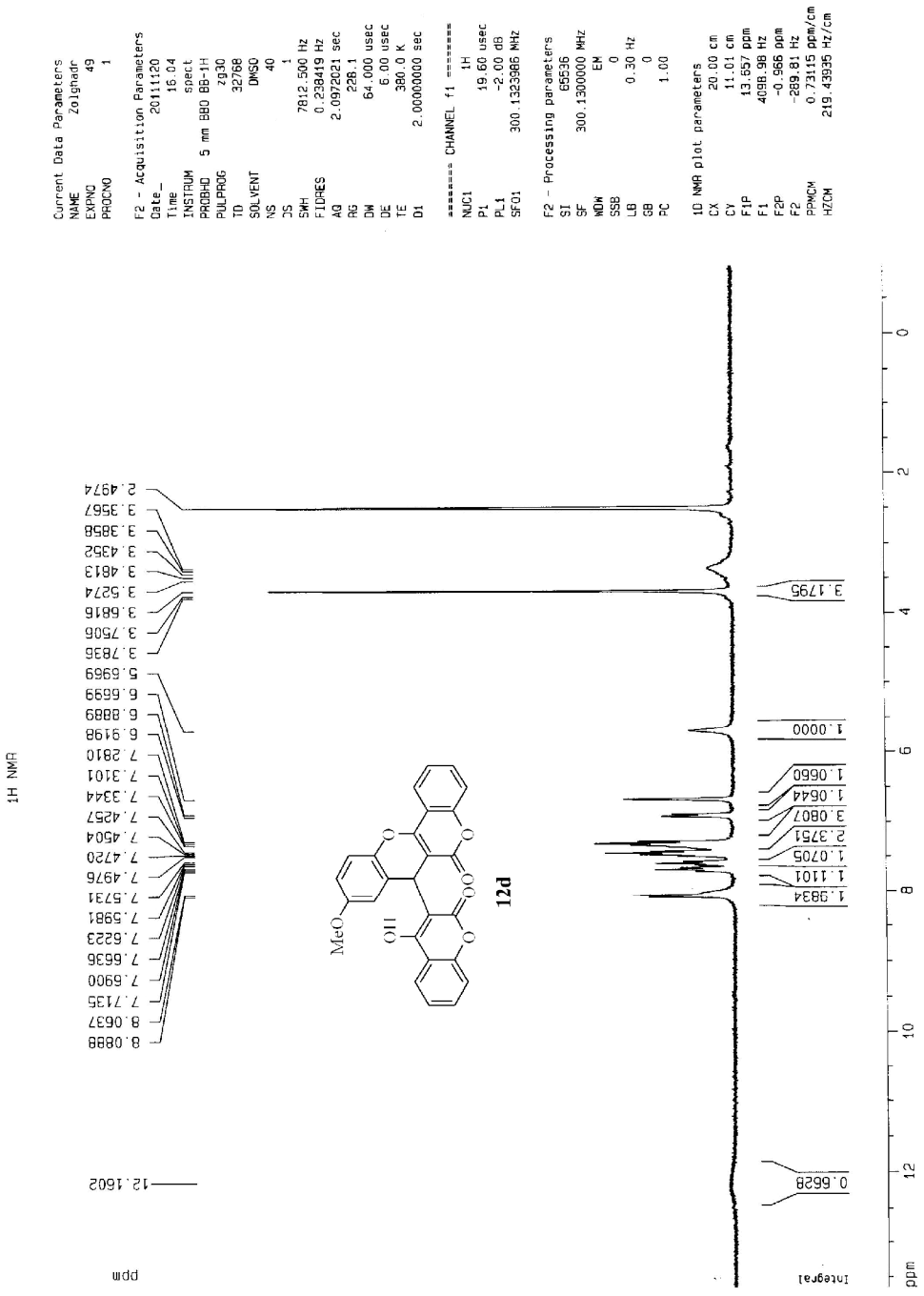

1H NMR
12b
Br
OH
O
ppm
Integral
ppm
Current Data Parameters
NAME Anad1
EXPNO 905
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110821
Time 0.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.25 cm
F1P 11.016 ppm
F1 3306.24 Hz
F2P -1.119 ppm
F2 -335.76 Hz
PPMCM 0.60674 ppm/cm
HZCM 182.10017 Hz/cm
2.50021
3.23729
3.45707
3.51513
5.70964
7.30528
7.34071
7.41989
7.44640
7.46691
7.48124
7.50531
7.57201
7.59689
7.62192
7.66139
7.68631
7.71186
8.05442
8.07985
1.0000
2.3112
1.4580
1.2236
4.3794
4.6907

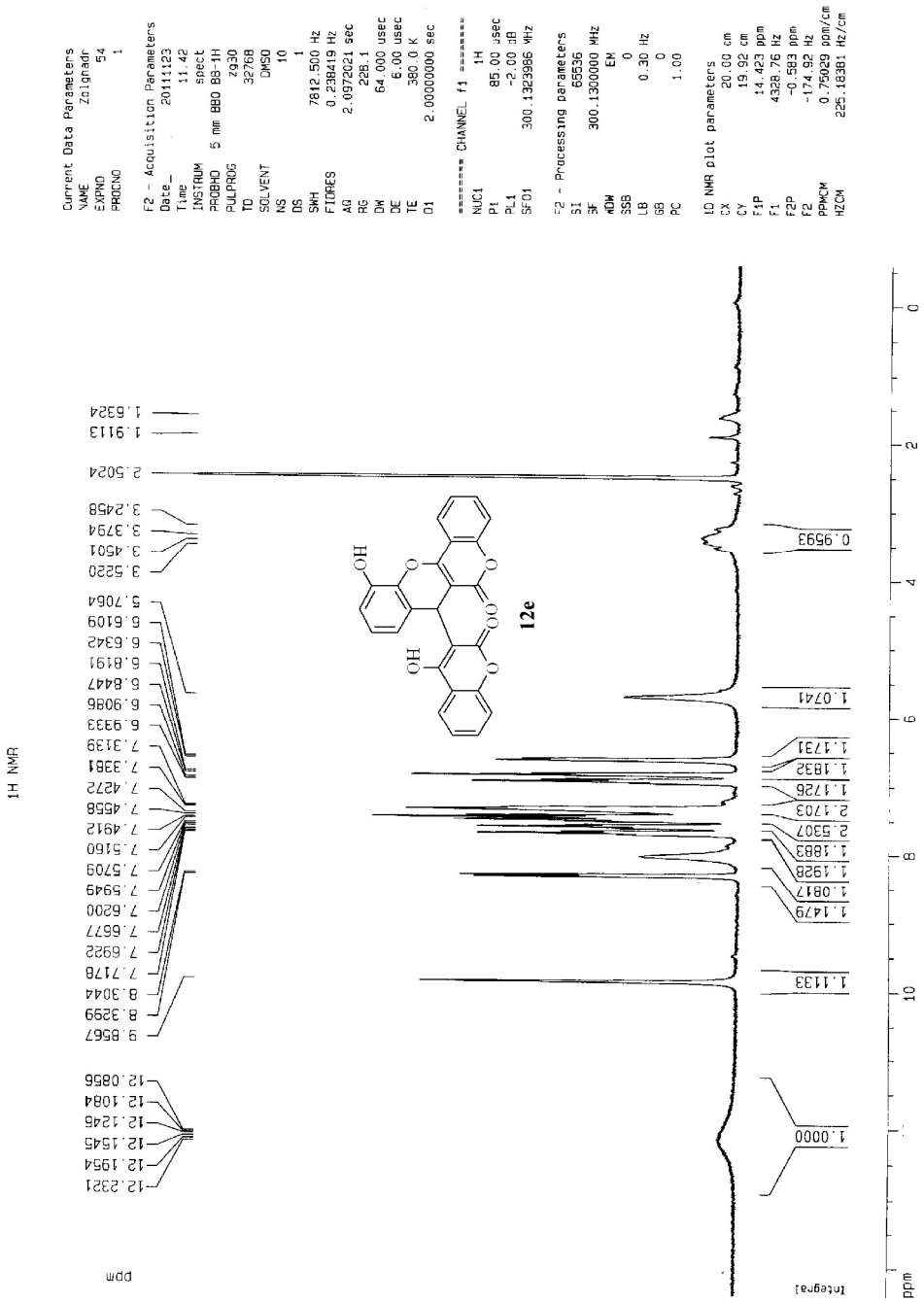

Current Data Parameters
NAME Anadi
EXPNO 906
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110821
Time 0.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 879
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 -2.00 dB
SF01 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 91.25 cm
F1P 219.155 ppm
F1 16539.11 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28052 Hz/cm
29.067
39.088
39.366
39.644
39.922
40.200
40.478
40.756
114.022
116.693
116.845
116.957
117.099
119.057
123.069
124.431
124.591
125.045
125.365
131.252
131.648
132.750
133.080
149.056
152.399
152.745
156.446
160.573
161.707
161.756
13C{1H} NMR
ppm
0
50
100
150
200
Br
HO
O
O
O
O
O
12b

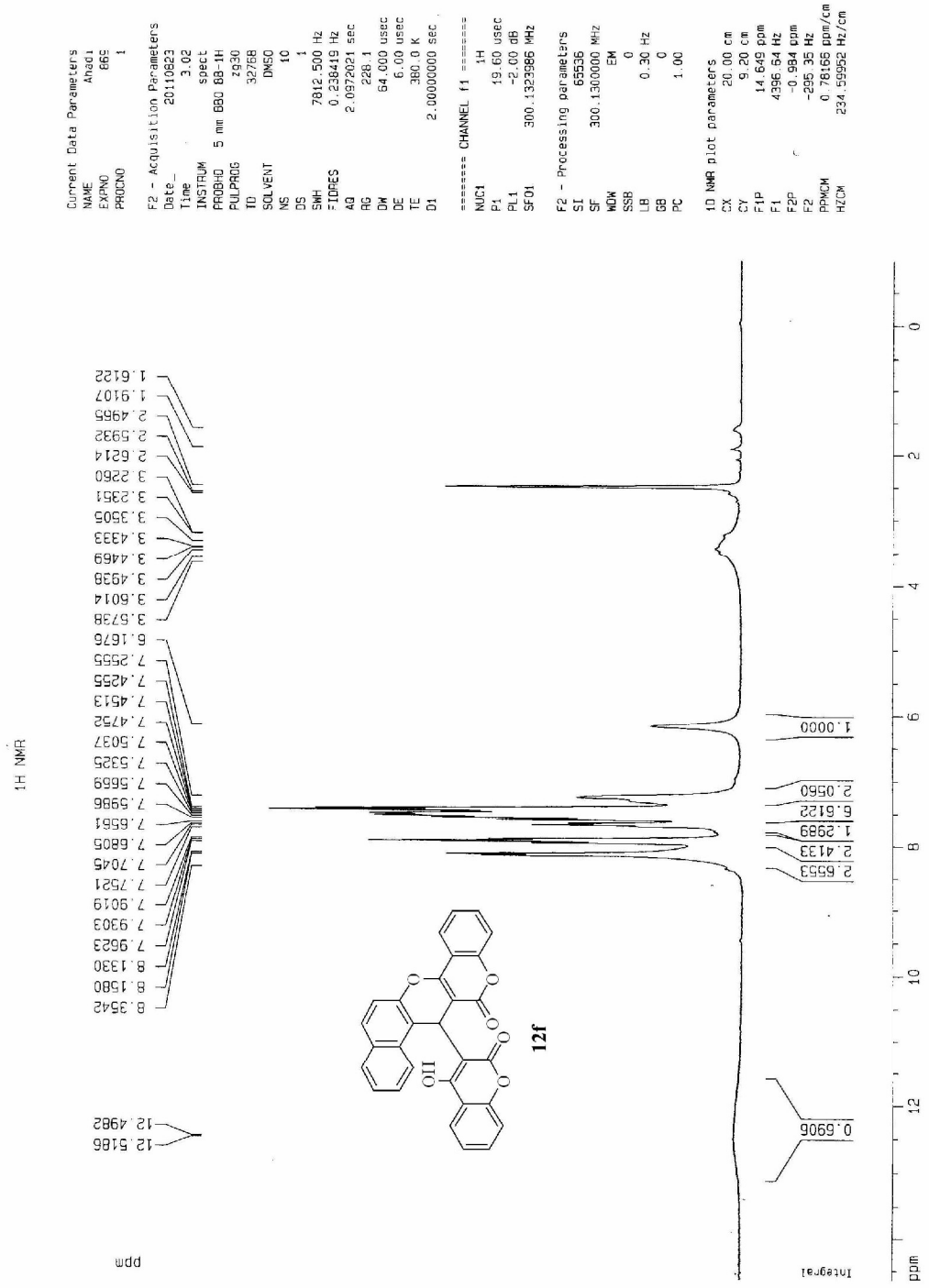


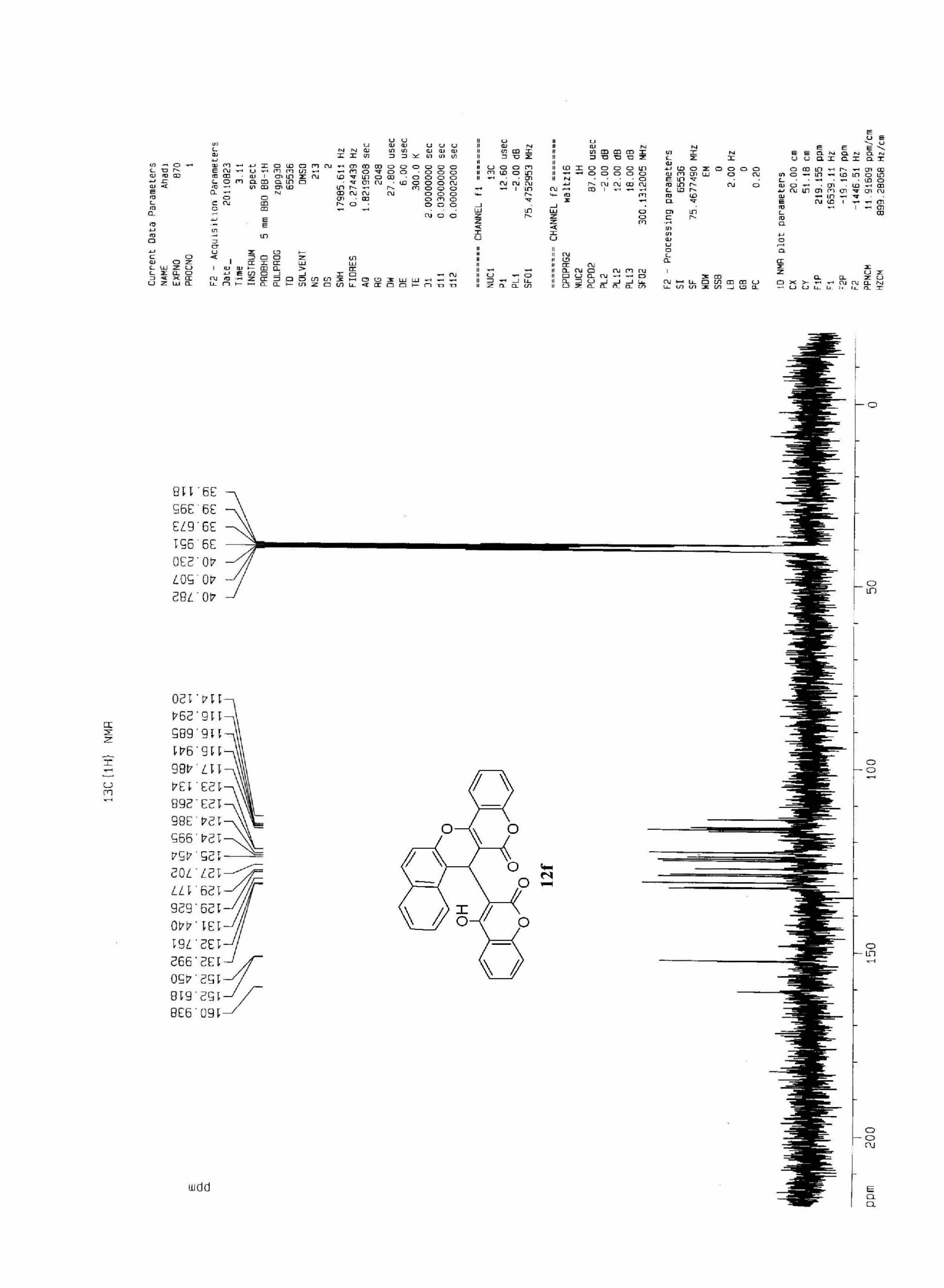

13c {1H} NMR
ppm
28.618
38.921
39.094
39.371
39.649
39.927
40.205
40.483
40.762
55.908
101.763
112.386
114.242
114.386
116.655
116.929
117.022
123.057
124.109
124.396
124.966
129.630
132.568
132.915
150.289
152.406
152.626
156.681
159.537
160.909
OMe
OH
O
O
O
O
O
12c
Current Data Parameters
NAME Amadi
EXPNO 864
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110902
Time 22.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 431
DS 2
SWH 17985.611 Hz
FIDRES 0.274430 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 85.56 cm
F1P 219.155 ppm
F1 16539.11 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm
0
50
100
150
200


MW440-R 228 (3.446)
Solid Probe
Scan EI+
1.44e7
12c
m/z
%


Current Data Parameters
NAME Zolghadr
EXPNO 49
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111120
Time 16.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 40
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323985 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 11.01 cm
F1P 13.657 ppm
F1 4098.98 Hz
F2P -0.966 ppm
F2 -289.81 Hz
PPMCM 0.73115 ppm/cm
HZCM 219.43935 Hz/cm
1H NMR
12d
MeO
OH
ppm
Integral


Current Data Parameters
NAME Zaignadr
EXPNO 54
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111123
Time 11.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.09720211 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 85.00 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 19.92 cm
F1P 14.423 ppm
F1 4328.76 Hz
F2P -0.583 ppm
F2 -174.92 Hz
PPMCM 0.75029 ppm/cm
HZCM 225.18381 Hz/cm
1H NMR
12e
OH
OH
O
O
O
O
O
integral
ppm
1.6324
1.9113
2.5024
3.2458
3.3794
3.4501
3.5220
5.7064
6.6109
6.6342
6.8191
6.8447
6.9086
6.9333
7.3139
7.3381
7.4272
7.4558
7.4912
7.5160
7.5709
7.5949
7.6200
7.6677
7.6922
7.7178
8.3044
8.3299
9.8567
12.0856
12.1084
12.1246
12.1545
12.1954
12.2321
0.9593
1.0741
1.1731
1.1832
1.1926
2.1703
2.5307
1.1883
1.1928
1.0817
1.1479
1.1133
1.0000


Current Data Parameters
NAME Ahad1
EXPNO 685
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110823
Time 3.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 19.60 usec
PL1 -2.00 dB
SFO1 300.1323986 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.20 cm
F1P 14.649 ppm
F1 4396.64 Hz
F2P -0.984 ppm
F2 -295.35 Hz
PPMCM 0.78165 ppm/cm
HZCM 234.59952 Hz/cm
1H NMR
12.5186
12.4982
8.3542
8.1580
8.1330
7.9623
7.9303
7.9019
7.7521
7.7045
7.6805
7.6561
7.5986
7.5659
7.5325
7.5037
7.4752
7.4513
7.4255
7.2555
6.1676
3.5738
3.6014
3.4938
3.4469
3.4333
3.3505
3.2351
3.2260
2.6214
2.5932
2.4965
1.9107
1.6122
1.0000
2.0560
6.6122
1.2989
2.4133
2.6553
0.6906
12f
ppm
Integral


13C {1H} NMR
160.938
152.618
152.450
132.995
132.761
131.440
129.626
129.177
127.702
125.454
124.995
124.386
123.268
123.134
117.486
116.941
116.685
116.294
114.120
40.782
40.507
40.230
39.951
39.673
39.395
39.118
12f
OH
ppm
Current Data Parameters
NAME Ahadi
EXPNO 870
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110823
Time 3.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 213
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.20
1D NMR plot parameters
CX 20.00 cm
CY 51.18 cm
F1P 219.155 ppm
F1 16539.11 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm
200
150
100
50
0
ppm