Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information Part - II

Conformationally-constrained Indeno[2,1-c]quinolines - A New Class of Anti-mycobacterial agents**

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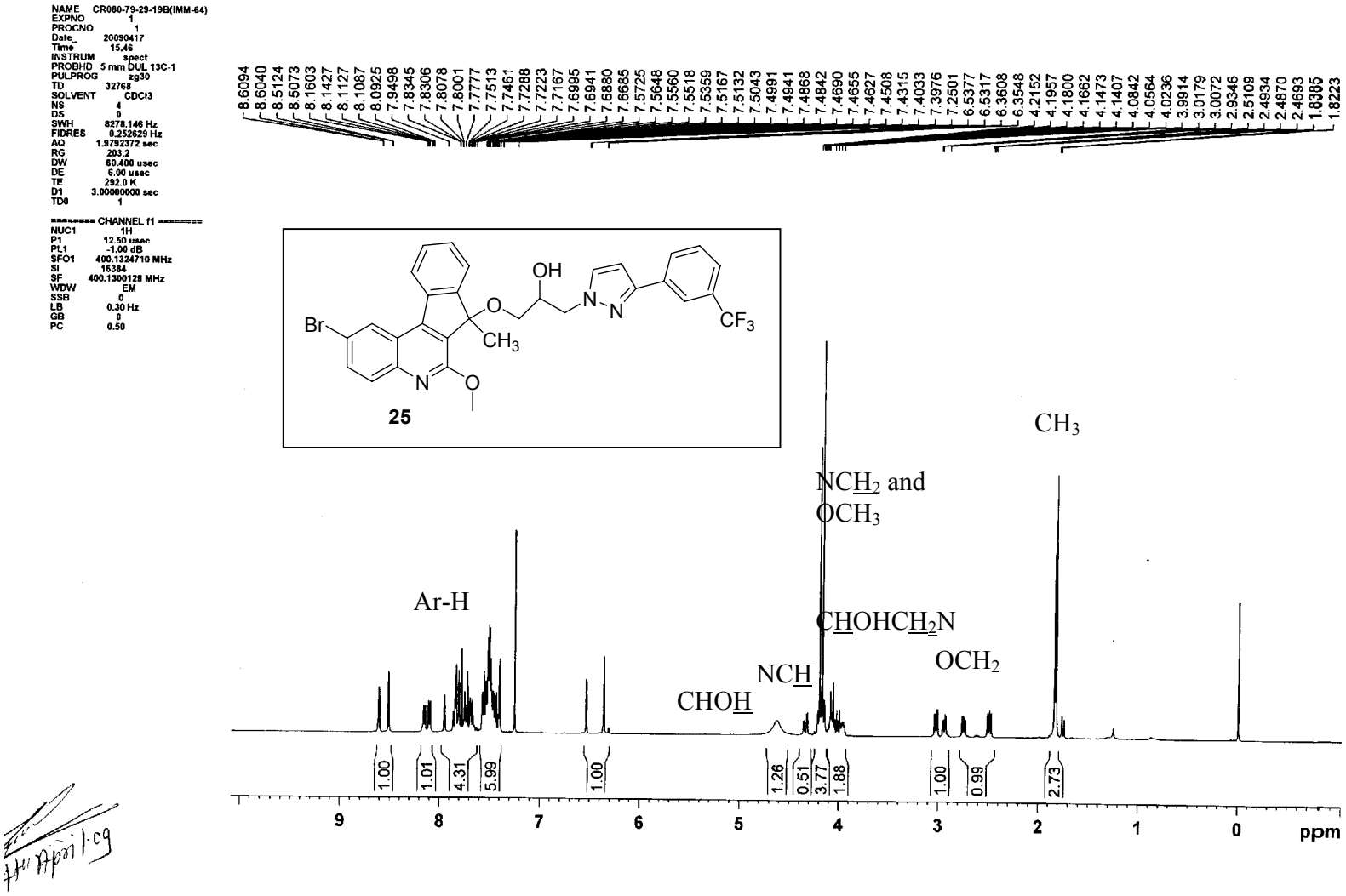
Table T2: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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 292.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
8.6094 8.6040 8.5124 8.5073 8.1603 8.1427 8.1127 8.1087 8.0925 7.9498 7.8345 7.8306 7.8078 7.8001 7.7777 7.7513 7.7461 7.7288 7.7223 7.7167 7.6995 7.6941 7.6880 7.6685 7.5725 7.5648 7.5560 7.5518 7.5359 7.5167 7.5132 7.5043 7.4991 7.4941 7.4868 7.4842 7.4690 7.4655 7.4627 7.4508 7.4315 7.4033 7.3976 7.2501 6.5377 6.5317 6.3608 6.3548 4.2152 4.1957 4.1800 4.1662 4.1473 4.1407 4.0842 4.0564 4.0236 3.9914 3.0179 3.0072 2.9346 2.5109 2.4934 2.4870 2.4693 1.8385 1.8223
Br
O
OH
N
N
CF3
CH3
N
O
25
CH3
NCH2 and
OCH3
Ar-H
CHOHCH2N
OCH2
NCH
CHOH
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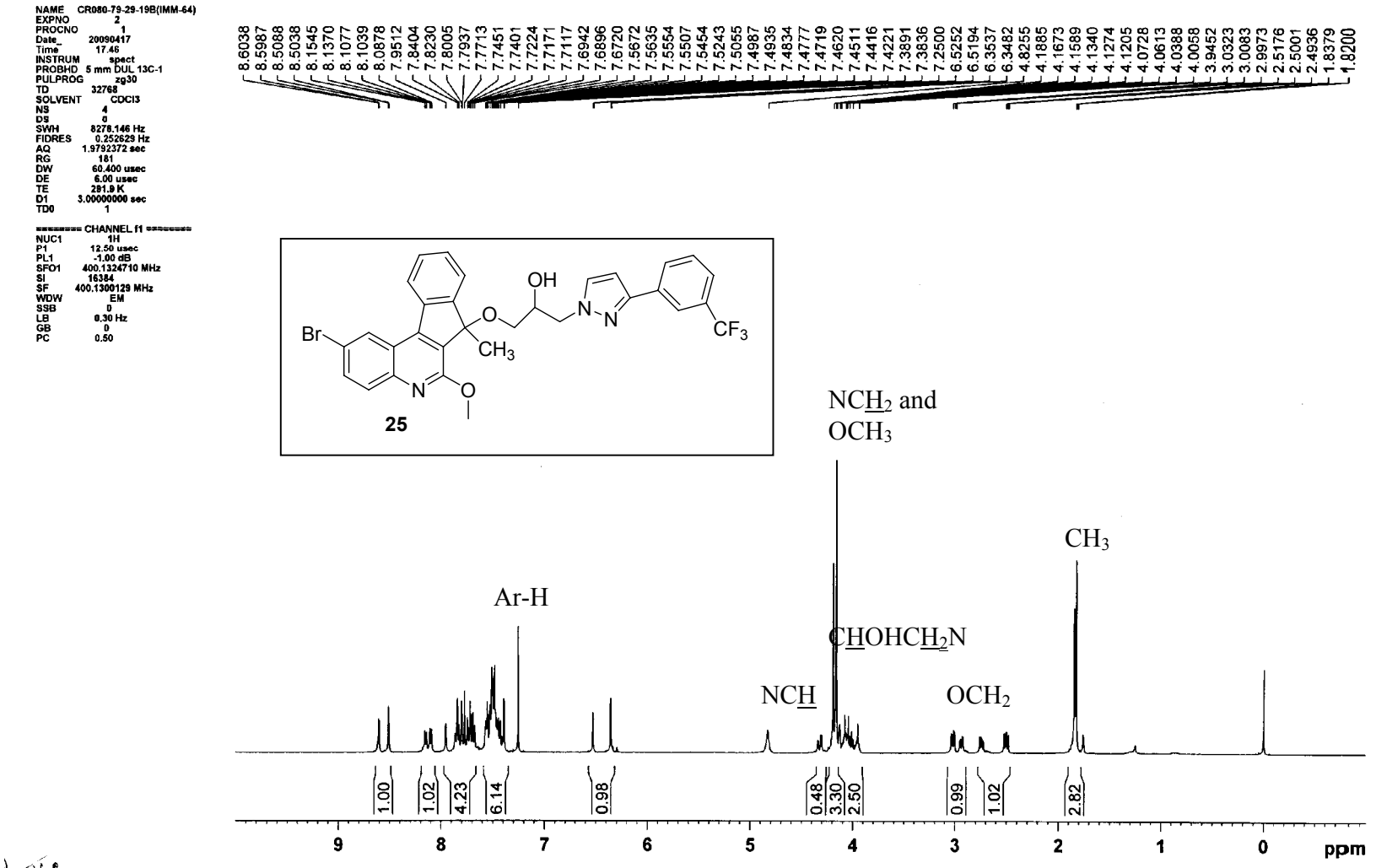
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 291.9 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
8.6038 8.5987 8.5088 8.5038 8.1545 8.1370 8.1077 8.1039 8.0878 7.9512 7.8404 7.8230 7.8005 7.7937 7.7713 7.7451 7.7401 7.7224 7.7171 7.7117 7.6942 7.6896 7.6720 7.5672 7.5635 7.5554 7.5507 7.5454 7.5243 7.5055 7.4987 7.4935 7.4834 7.4777 7.4719 7.4620 7.4511 7.4416 7.4221 7.3891 7.3836 7.2500 6.5252 6.5194 6.3537 6.3482 4.8255 4.1885 4.1673 4.1589 4.1340 4.1274 4.1205 4.0728 4.0613 4.0388 4.0058 3.9452 3.0323 3.0083 2.9973 2.5176 2.5001 2.4936 1.8379 1.8200
Br
O
OH
N
N
CF3
CH3
N
O
25
NCH2 and
OCH3
CH3
Ar-H
CHOHCH2N
NCH
OCH2
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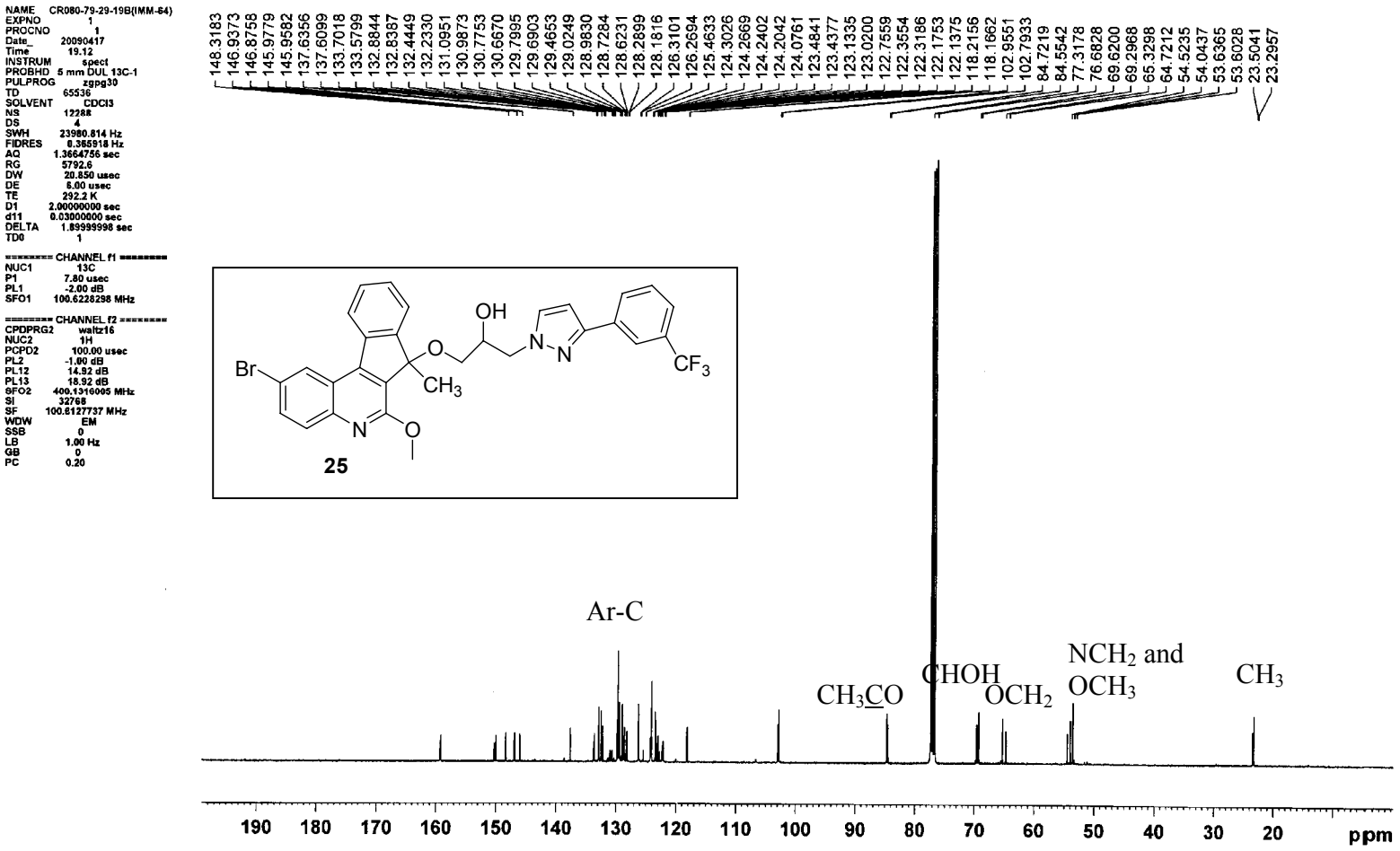
9 8 7 6 5 4 3 2 1 0 ppm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TD 65536
SOLVENT CDCl3
NS 12288
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 292.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
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NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
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146.9373
146.8758
145.9779
145.9582
137.6356
137.6099
133.7018
133.5799
132.8844
132.8387
132.4449
132.2330
131.0951
130.9873
130.7753
130.6670
129.7995
129.6903
129.4653
129.0249
128.9830
128.7284
128.6231
128.2899
128.1816
126.3101
126.2694
125.4633
124.3026
124.2669
124.2402
124.2042
124.0761
123.4841
123.4377
123.1335
123.0200
122.7559
122.3554
122.3186
122.1753
122.1375
118.2156
118.1662
102.9551
102.7933
84.7219
84.5542
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69.6200
69.2968
65.3298
64.7212
54.5235
54.0437
53.6365
53.6028
23.5041
23.2957
Br
OH
O
N
N
CF3
CH3
N
O
25
Ar-C
CH3CO
CHOH
OCH2
NCH2 and
OCH3
CH3
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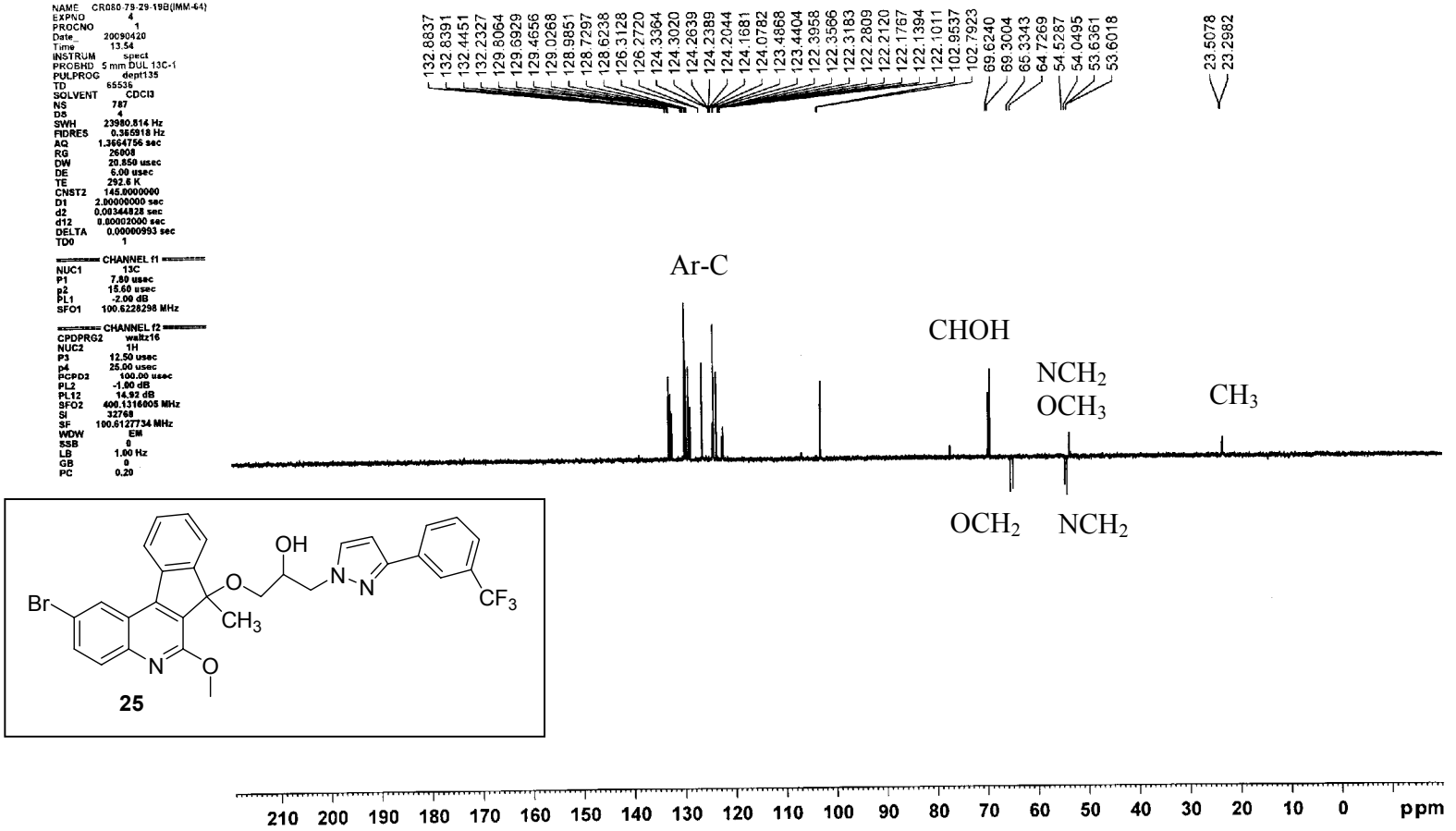
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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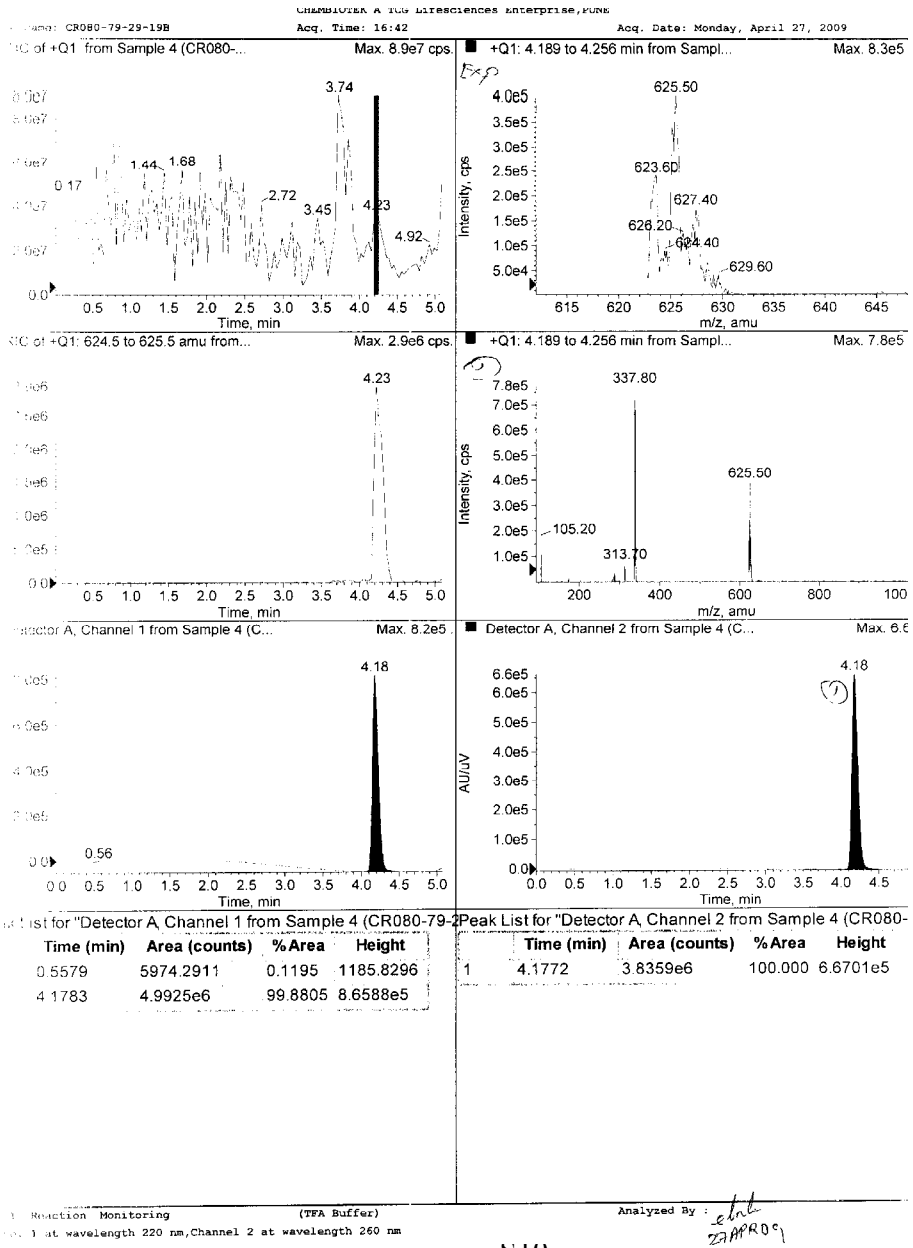
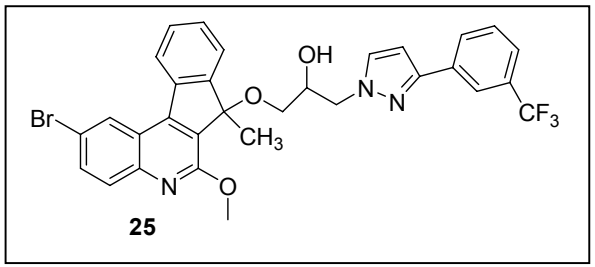
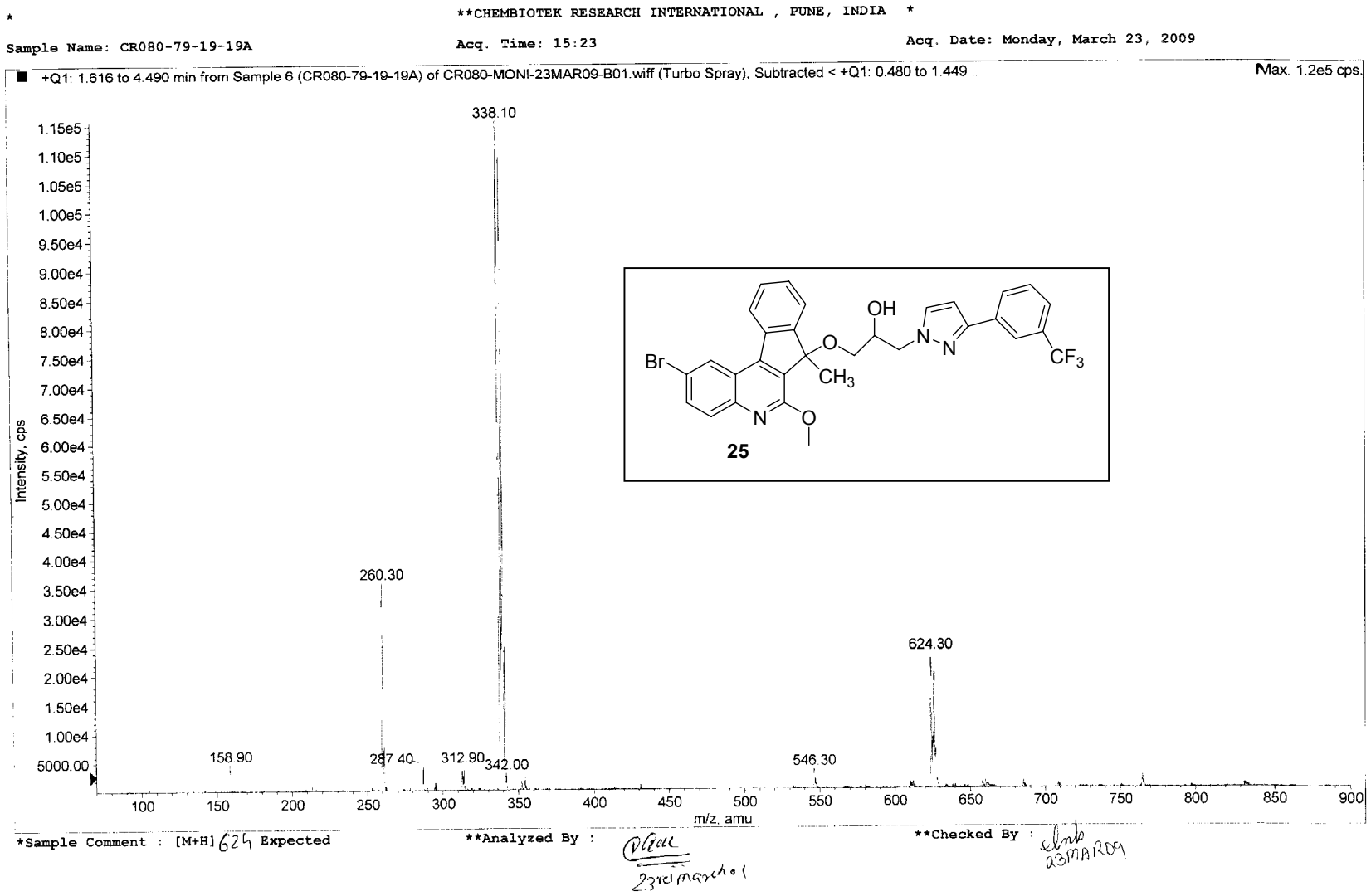
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 787
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 26908
DW 20.850 usec
DE 6.00 usec
TE 292.6 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
132.8837
132.8391
132.4451
132.2327
129.8064
129.6929
129.4656
129.0268
128.9851
128.7297
128.6238
126.3128
126.2720
124.3364
124.3020
124.2639
124.2389
124.2044
124.1681
124.0782
123.4868
123.4404
122.3958
122.3586
122.3183
122.2809
122.2120
122.1767
122.1394
122.1011
102.9537
102.7923
69.6240
69.3004
65.3343
64.7269
54.5287
54.0495
53.6361
53.6018
23.5078
23.2982
Ar-C
CHOH
NCH2
OCH3
CH3
OCH2
NCH2
Br
O
OH
N
N
CF3
CH3
N
O
25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Acq. Time: 16:42
Acq. Date: Monday, April 27, 2009
Max. 8.9e7 cps.
Max. 8.3e5
Max. 2.9e6 cps.
Max. 7.8e5
Max. 8.2e5
Max. 6.6
Time, min
m/z, amu
Intensity, cps
AU/uV
Peak List for "Detector A, Channel 1 from Sample 4 (CR080-79-2
Peak List for "Detector A, Channel 2 from Sample 4 (CR080-
Time (min) Area (counts) %Area Height
0.5579 5974.2911 0.1195 1185.8296
4.1783 4.9925e6 99.8805 8.6588e5
1 4.1772 3.8359e6 100.000 6.6701e5
(TFA Buffer)
Analyzed By :


Br
OH
O
N
N
CF3
CH3
N
O

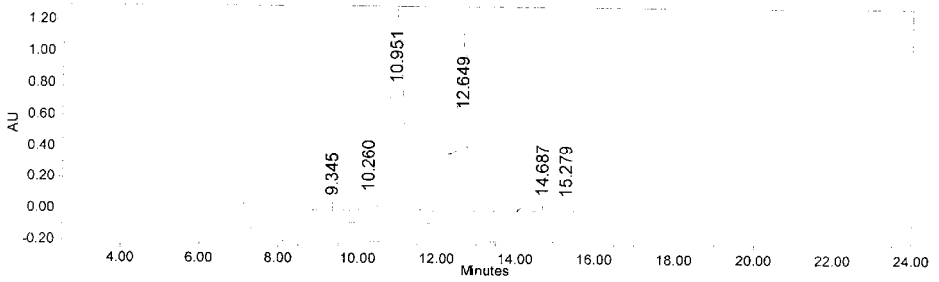
25


* **CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *
Sample Name: CR080-79-19-19A
Acq. Time: 15:23
Acq. Date: Monday, March 23, 2009
+Q1: 1.616 to 4.490 min from Sample 6 (CR080-79-19-19A) of CR080-MONI-23MAR09-B01.wiff (Turbo Spray), Subtracted < +Q1: 0.480 to 1.449 ..
Max. 1.2e5 cps.
Intensity, cps
m/z, amu
338.10
260.30
624.30
158.90
287.40
312.90
342.00
546.30
Br
OH
O
N
N
CH3
CF3
25
*Sample Comment : [M+H] 624 Expected
**Analyzed By :
23rd March 09
**Checked By :
23MAR09

SAMPLE INFORMATION

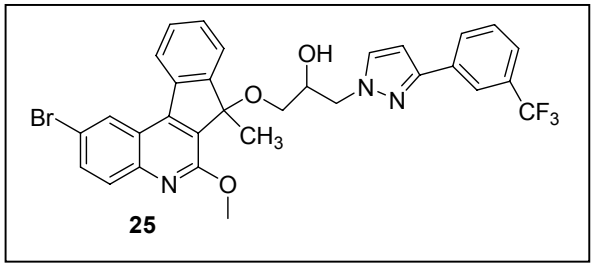
Sample Name	CR080/54/107/107A	Acquired By	Ramudu
Sample Type	Unknown	Sample Set Name	P19NON030
Vial:	2	Acq. Method Set	P19_CR80_ARD25_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_93C_PRO
Injection Volume	10.00 ul	Label	P19090109
Run Time:	30.0 Minutes	Proc. Chanel	[2998 Ch1 200nm@1.2nm]
Date Acquired	2/9/2009 12:31:11 PM IST	Date Processed	2/9/2009 2:29:06 PM IST

Auto-Scaled Chromatogram



Peak Results

	RT	Area	% Area
1	9.345	379386	0.66
2	10.260	284118	0.49
3	10.951	28765596	49.80
4	12.649	27948295	48.39
5	14.687	218052	0.38
6	15.279	165495	0.29

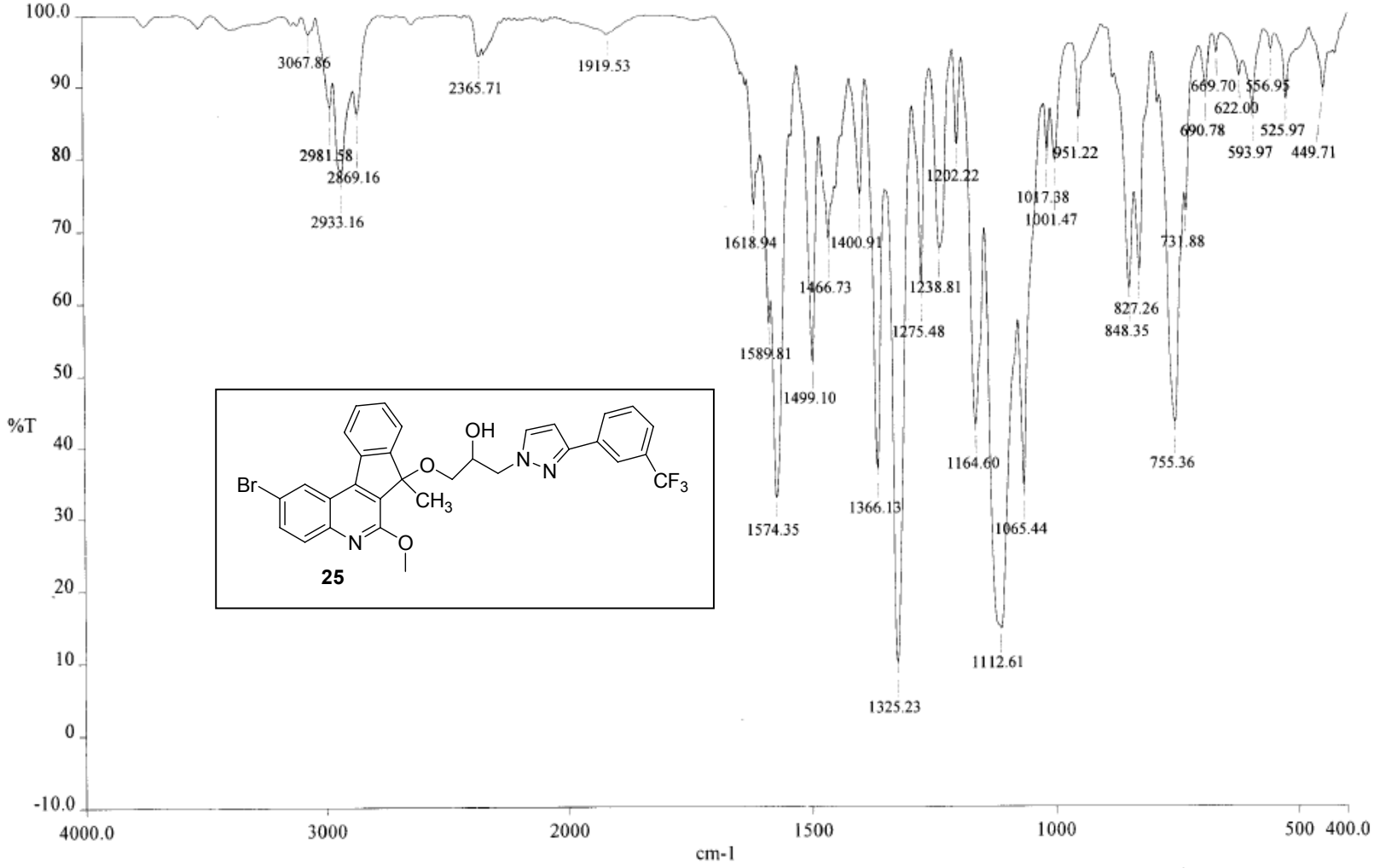


25

Reported by User : Ramudu Kurva (Ramudu)
Report Method : GENERAL_ASSAY_REPORT
Analysed by : 09 FEB 2009

Project Name: HPLC_P19_NON GLP
Date Printed : 2/9/2009
Checked by :

Page: 1 of 1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90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3067.86
2981.58
2869.16
2933.16
2365.71
1919.53
1618.94
1589.81
1574.35
1499.10
1466.73
1400.91
1366.13
1325.23
1275.48
1238.81
1202.22
1164.60
1112.61
1065.44
1017.38
1001.47
951.22
848.35
827.26
755.36
731.88
690.78
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OH
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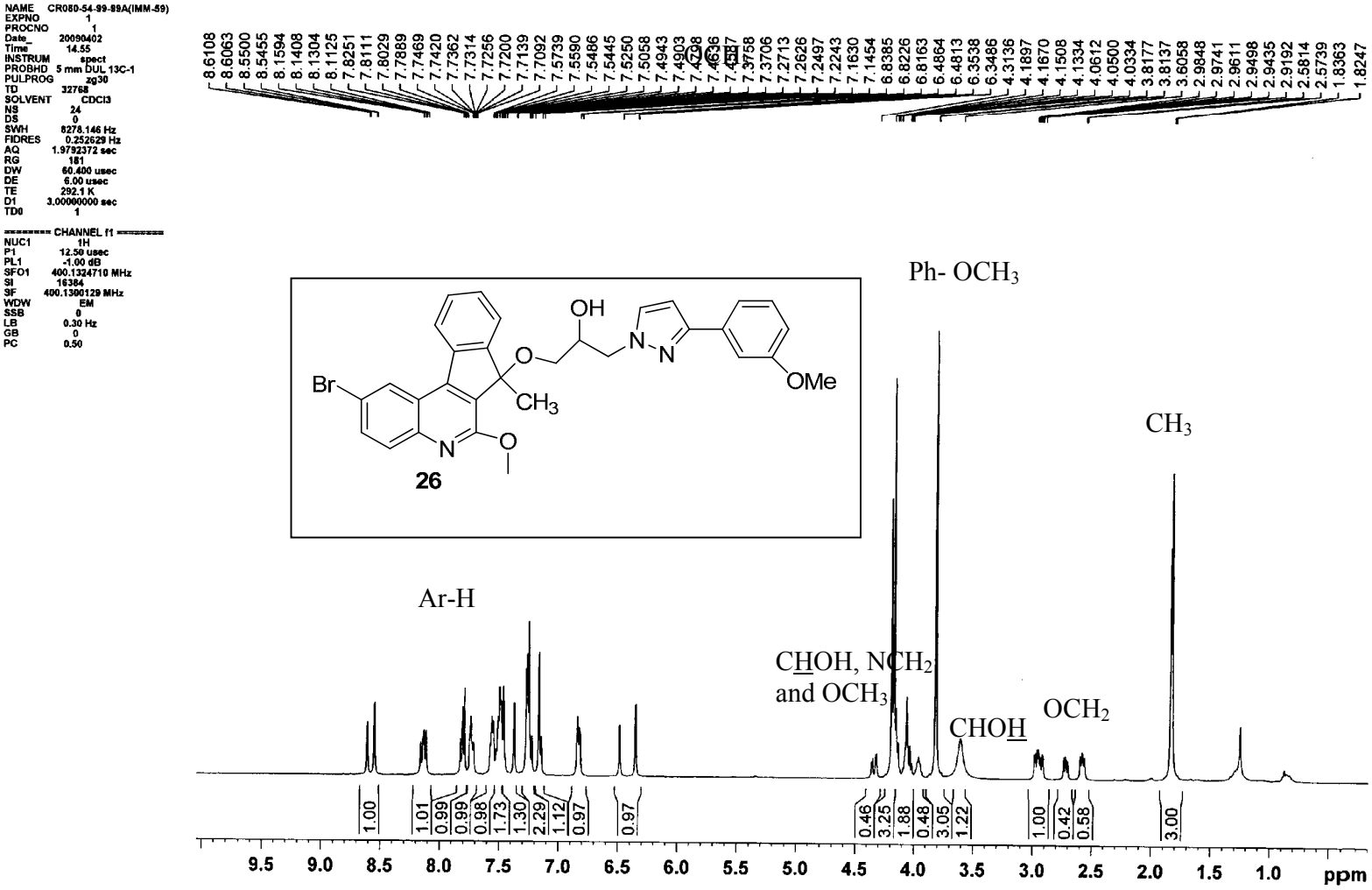
N
CF3
N
O
25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Time 14.55
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PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 292.1 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
Br
OH
N
N
O
OMe
CH3
O
N
O
26
Ph- OCH3
CH3
Ar-H
CHOH, NCH2
and OCH3
CHOH
OCH2
1.00
1.01
0.99
0.99
0.98
1.73
1.30
2.29
1.12
0.97
0.97
0.46
3.25
1.88
0.48
3.05
1.22
1.00
0.42
0.58
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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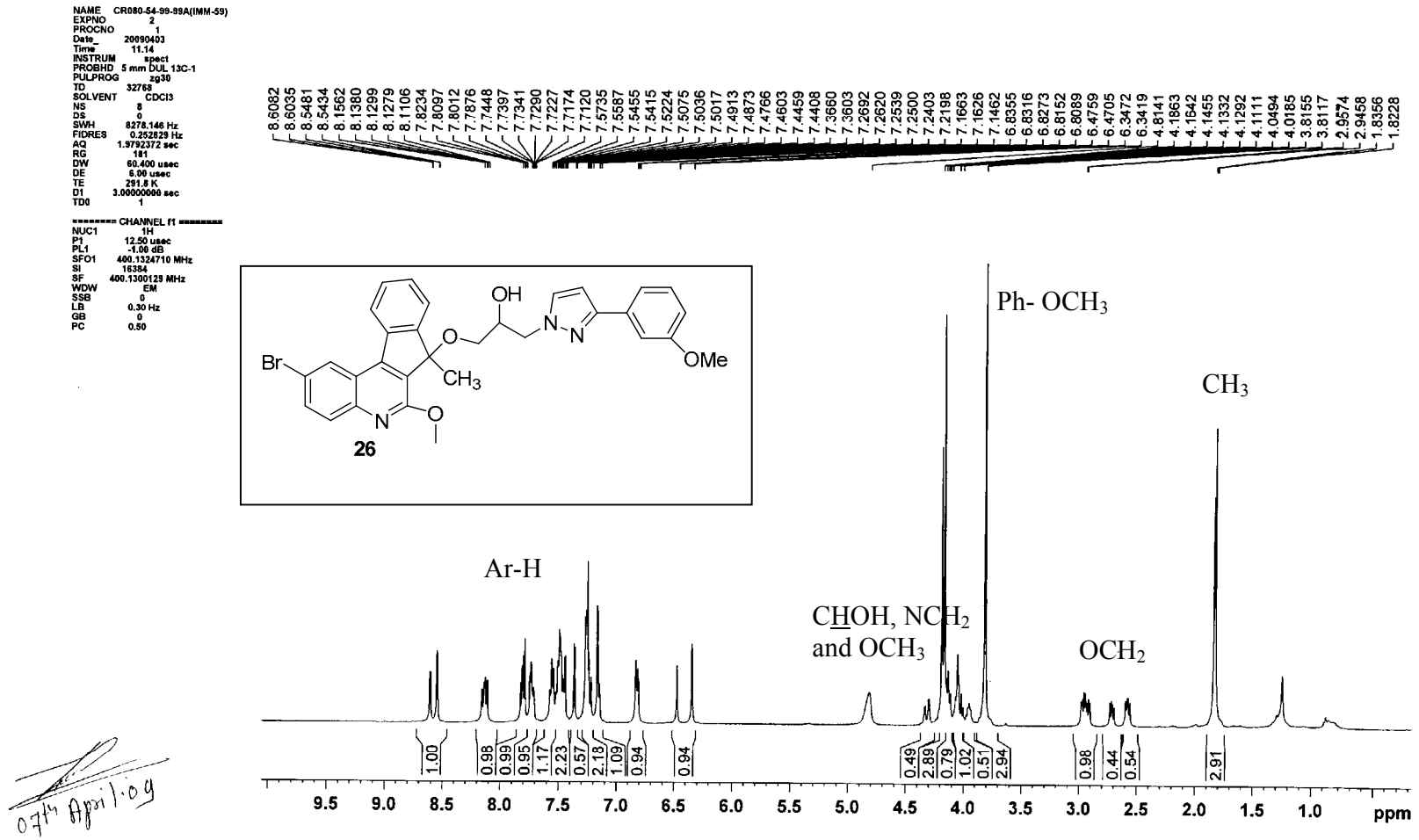
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252829 Hz
AQ 1.9792372 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 291.8 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
8.6082
8.6035
8.5481
8.5434
8.1562
8.1380
8.1299
8.1279
8.1106
7.8234
7.8097
7.8012
7.7876
7.7448
7.7397
7.7341
7.7290
7.7227
7.7174
7.7120
7.5735
7.5587
7.5455
7.5415
7.5224
7.5075
7.5036
7.5017
7.4913
7.4873
7.4766
7.4603
7.4459
7.4408
7.3660
7.3603
7.2692
7.2620
7.2539
7.2500
7.2403
7.2198
7.1663
7.1626
7.1462
6.8355
6.8316
6.8273
6.8152
6.8089
6.4759
6.4705
6.3472
6.3419
4.8141
4.1863
4.1642
4.1455
4.1332
4.1292
4.1111
4.0494
4.0185
3.8155
3.8117
2.9574
2.9458
1.8356
1.8228
Br
OH
O
N
N
OMe
CH3
N
O
26
Ph- OCH3
CH3
Ar-H
CHOH, NCH2
and OCH3
OCH2
1.00
0.98
0.99
0.95
1.17
2.23
0.57
2.18
1.09
0.94
0.94
0.49
2.89
0.79
1.02
0.51
2.94
0.98
0.44
0.54
2.91
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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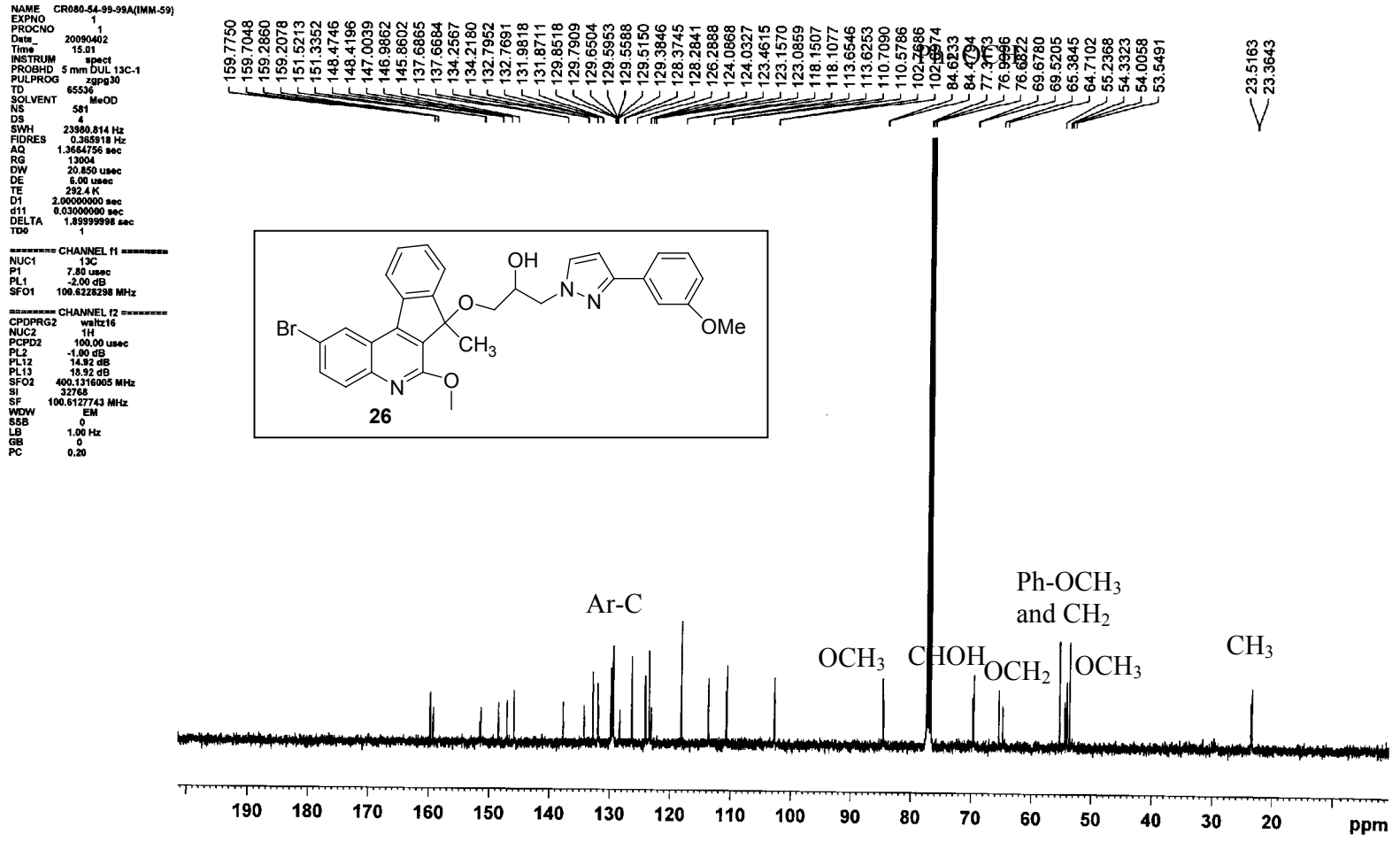
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm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Time 15.01
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PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 581
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654756 sec
RG 13004
DW 20.850 usec
DE 6.00 usec
TE 292.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
159.7750
159.7048
159.2860
159.2078
151.5213
151.3352
148.4746
148.4196
147.0039
146.9862
145.8602
137.6865
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134.2567
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113.6546
113.6253
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110.5786
84.6233
64.7102
55.2368
54.3323
54.0058
53.5491
23.5163
23.3643
Br
OH
O
N
N
OMe
CH3
N
O
26
Ar-C
OCH3
CHOH
OCH2
Ph-OCH3
and CH2
OCH3
CH3
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120
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50
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30
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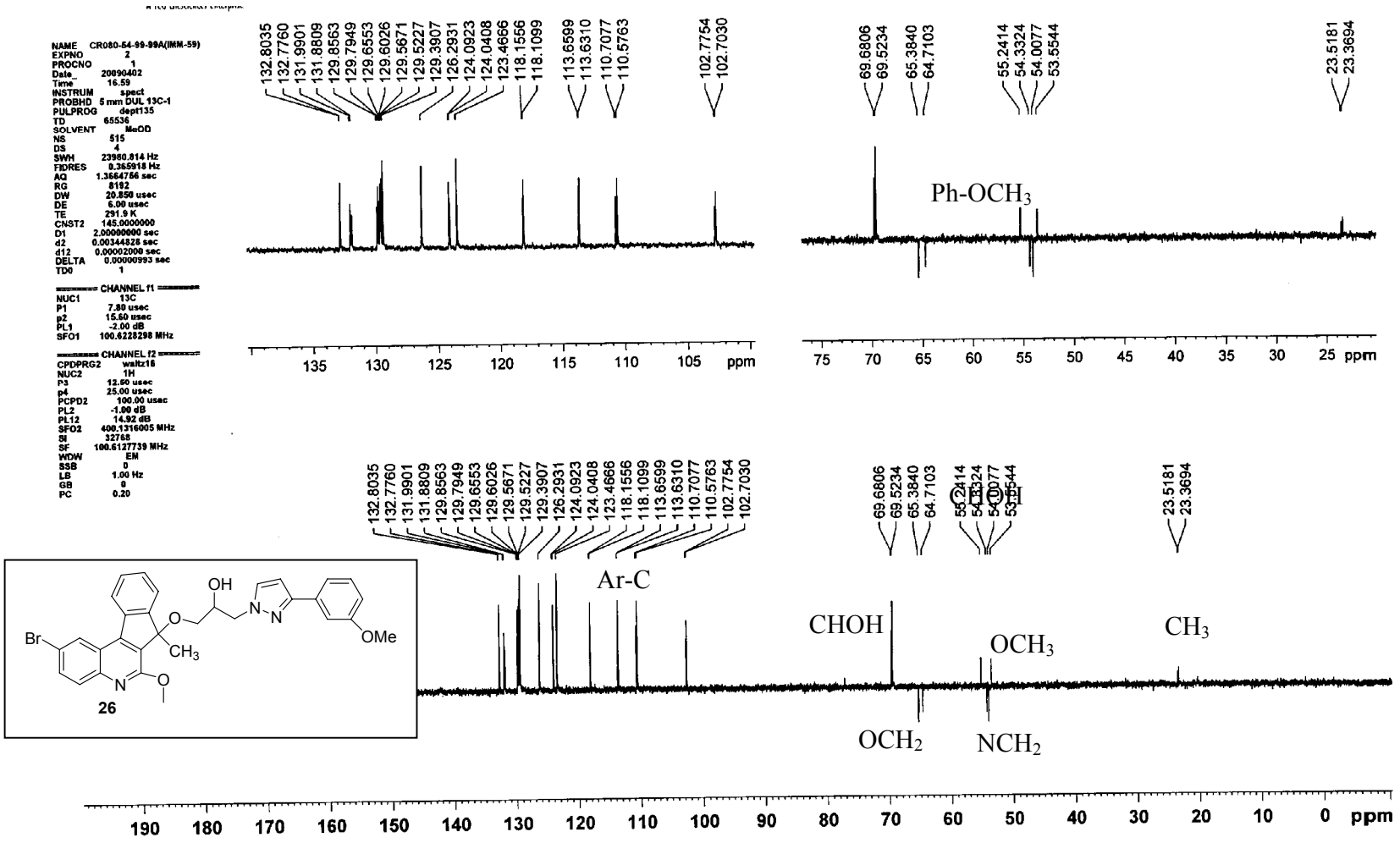
ppm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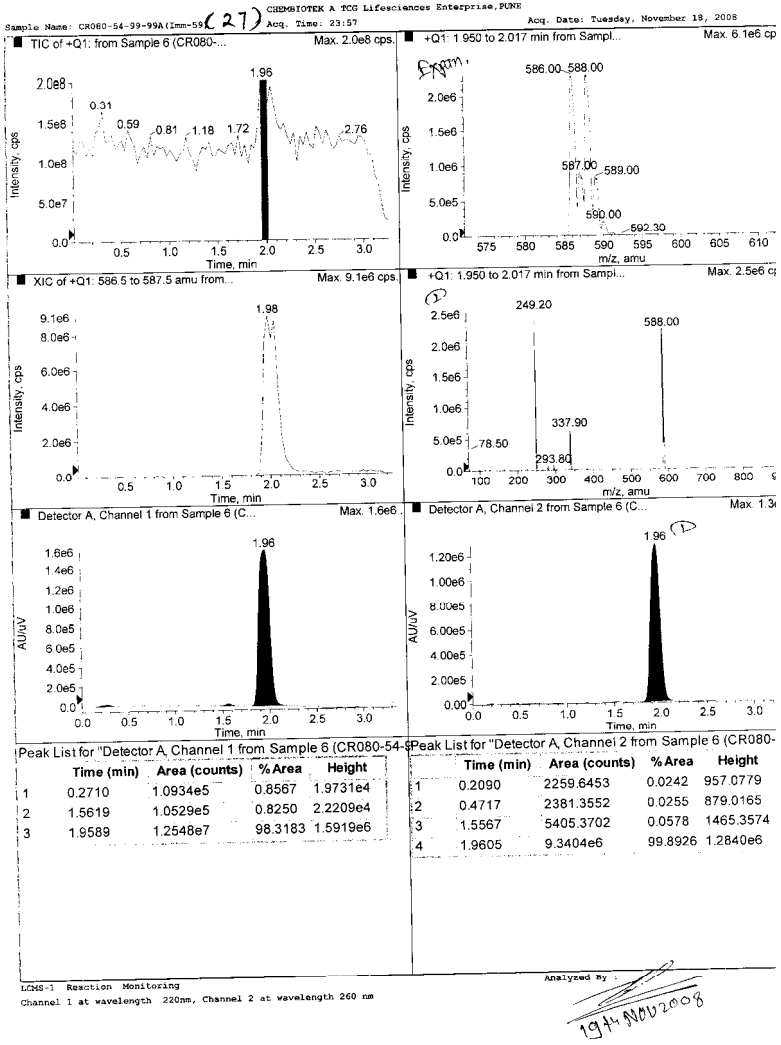
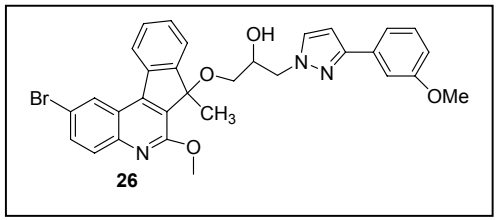
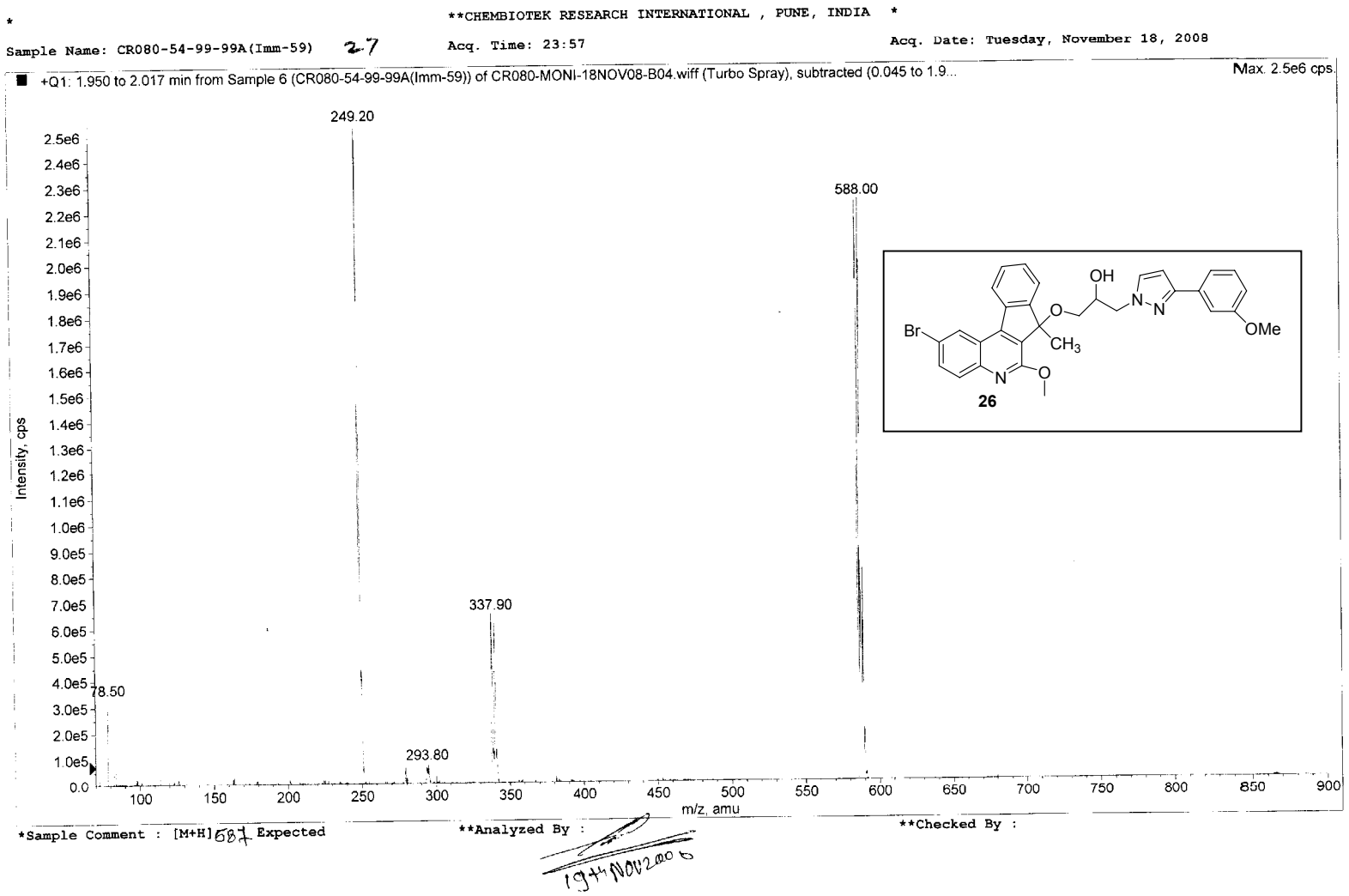
EXPNO 2
PROCNO 1
Date_ 20090402
Time 16.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT MeOD
NS 515
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 8192
DW 20.850 usec
DE 6.00 usec
TE 291.9 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
132.8035
132.7760
131.9901
131.8809
129.8563
129.7949
129.6553
129.6026
129.5671
129.5227
129.3907
126.2931
124.0923
124.0408
123.4666
118.1556
118.1099
113.6599
113.6310
110.7077
110.5763
102.7754
102.7030
69.6806
69.5234
65.3840
64.7103
55.2414
54.3324
54.0077
53.5544
23.5181
23.3694
Ph-OCH3
135 130 125 120 115 110 105 ppm
75 70 65 60 55 50 45 40 35 30 25 ppm
Ar-C
CHOH
OCH3
CH3
OCH2
NCH2
Br
OH
OMe
CH3
26
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Sample Name: CR080-54-99-99A(Imm-59) (27)
CHEMBIOTEK A TCG Lifesciences Enterprise,PUNE
Acq. Time: 23:57
Acq. Date: Tuesday, November 18, 2008
TIC of +Q1: from Sample 6 (CR080-...
Max. 2.0e8 cps.
+Q1: 1.950 to 2.017 min from Sampl...
Max. 6.1e6 cp
XIC of +Q1: 586.5 to 587.5 amu from...
Max. 9.1e6 cps.
+Q1: 1.950 to 2.017 min from Sampl...
Max. 2.5e6 c
Detector A, Channel 1 from Sample 6 (C...
Max. 1.6e6
Detector A, Channel 2 from Sample 6 (C...
Max. 1.3
Intensity, cps
AU/uV
Time, min
m/z, amu
Peak List for "Detector A, Channel 1 from Sample 6 (CR080-54-9
Time (min) Area (counts) %Area Height
1 0.2710 1.0934e5 0.8567 1.9731e4
2 1.5619 1.0529e5 0.8250 2.2209e4
3 1.9589 1.2548e7 98.3183 1.5919e6
Peak List for "Detector A, Channel 2 from Sample 6 (CR080-
Time (min) Area (counts) %Area Height
1 0.2090 2259.6453 0.0242 957.0779
2 0.4717 2381.3552 0.0255 879.0165
3 1.5567 5405.3702 0.0578 1465.3574
4 1.9605 9.3404e6 99.8926 1.2840e6
LCMS-1 Reaction Monitoring
Channel 1 at wavelength 220nm, Channel 2 at wavelength 260 nm
Analyzed By :
19th Nov 2008

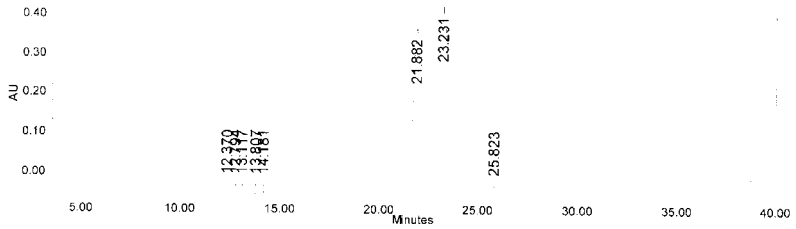

Br
OH
O
N
N
OMe
CH3
N
O
26


**CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *
Sample Name: CR080-54-99-99A(Imm-59) 27
Acq. Time: 23:57
Acq. Date: Tuesday, November 18, 2008
+Q1: 1.950 to 2.017 min from Sample 6 (CR080-54-99-99A(Imm-59)) of CR080-MONI-18NOV08-B04.wiff (Turbo Spray), subtracted (0.045 to 1.9...
Max. 2.5e6 cps.
249.20
588.00
337.90
78.50
293.80
Intensity, cps
m/z, amu
26
*Sample Comment : [M+H]687 Expected
**Analyzed By :
19th NOV 2008
**Checked By :

SAMPLE INFORMATION

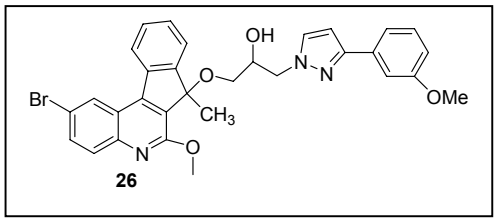
Sample Name	CR080/54/99/99A(IMM-59)	Acquired By	Ramudu
Sample Type	Unknown	Sample Set Name	P19NON034
Vial:	2	Acq. Method Set	P19_CR80_ARD25_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_99A_PRO
Injection Volume	5.00 ul	Label	P19090123
Run Time:	40.0 Minutes	Proc. Chanel	2998 Ch1 225nm@1.2nm
Date Acquired	2/10/2009 1:36:52 PM IST	Date Processed	2/10/2009 3:02:36 PM IST

Auto-Scaled Chromatogram



Peak Results

	RT	Area	% Area
1	12.370	5378	0.02
2	12.794	4439	0.02
3	13.117	8457	0.04
4	13.807	8272	0.04
5	14.181	12264	0.06
6	21.882	10021990	46.03
7	23.231	11694472	53.71
8	25.823	17600	0.08



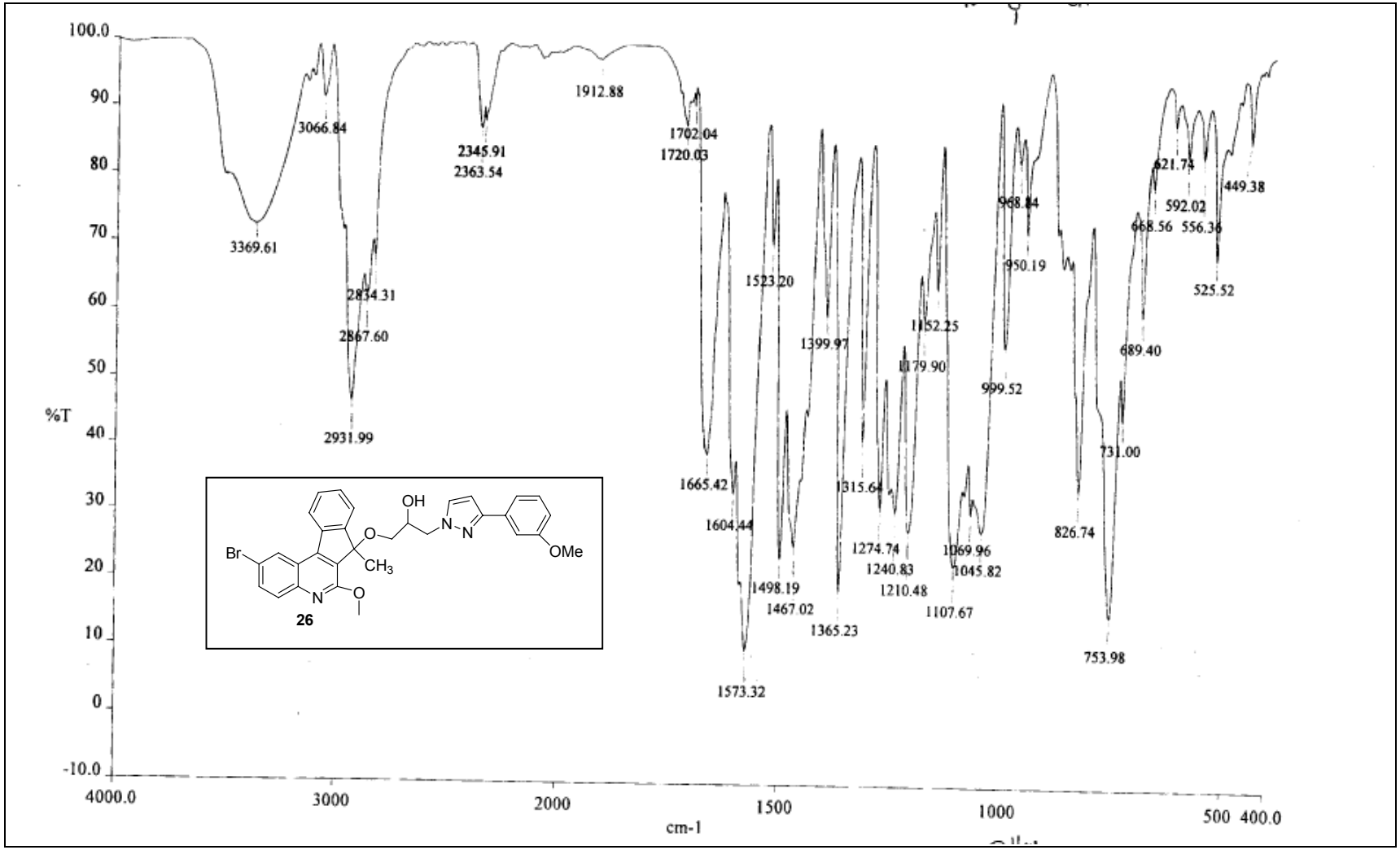
26

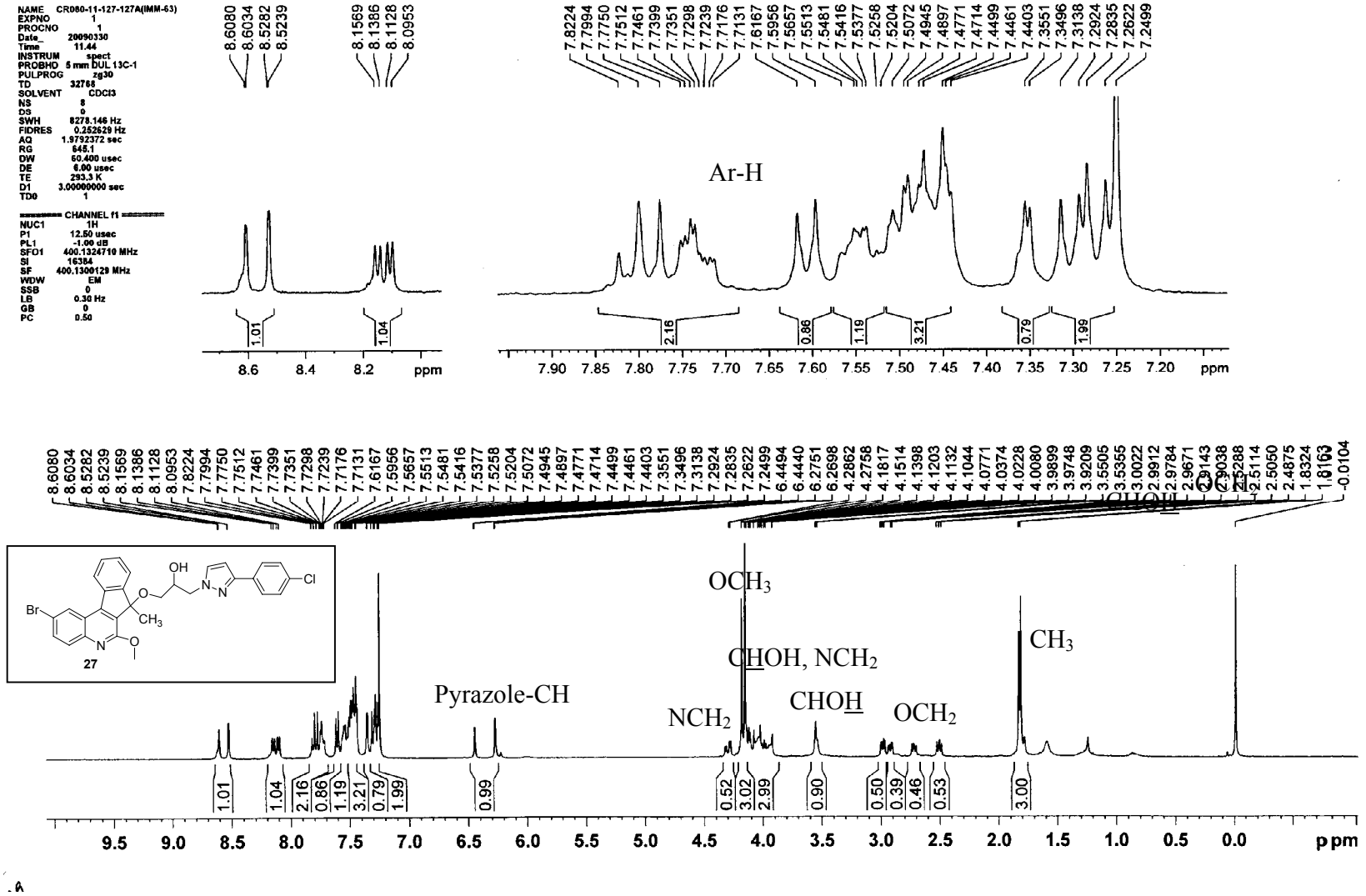
Reported by User : Ramudu Kurva (Ramudu)
Report Method : GENERAL_ASSAY_REPORT
Analysed by :

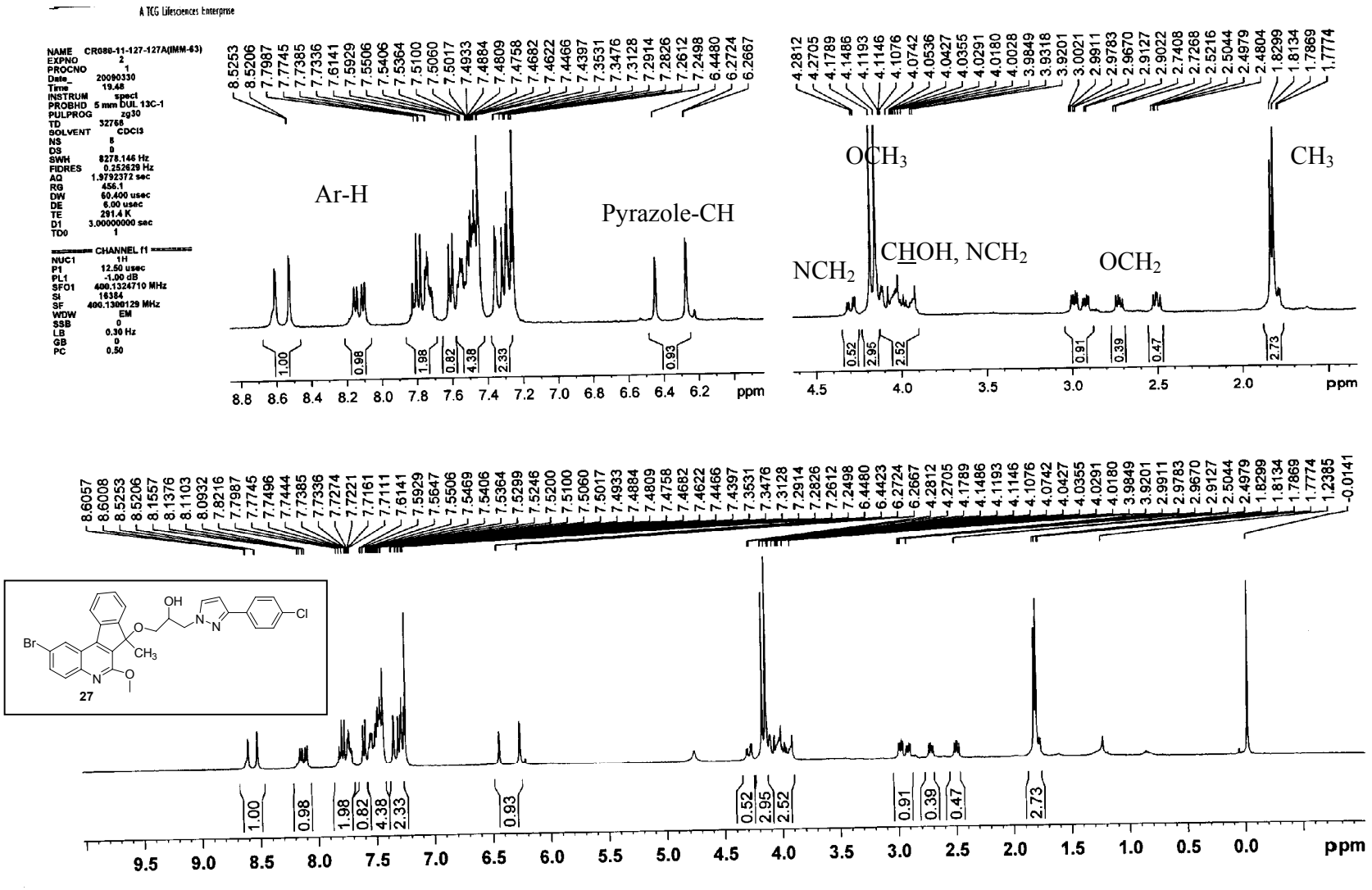
Project Name: HPLC_P19_NON GLP
Date Printed : 2/10/2009
Checked by :

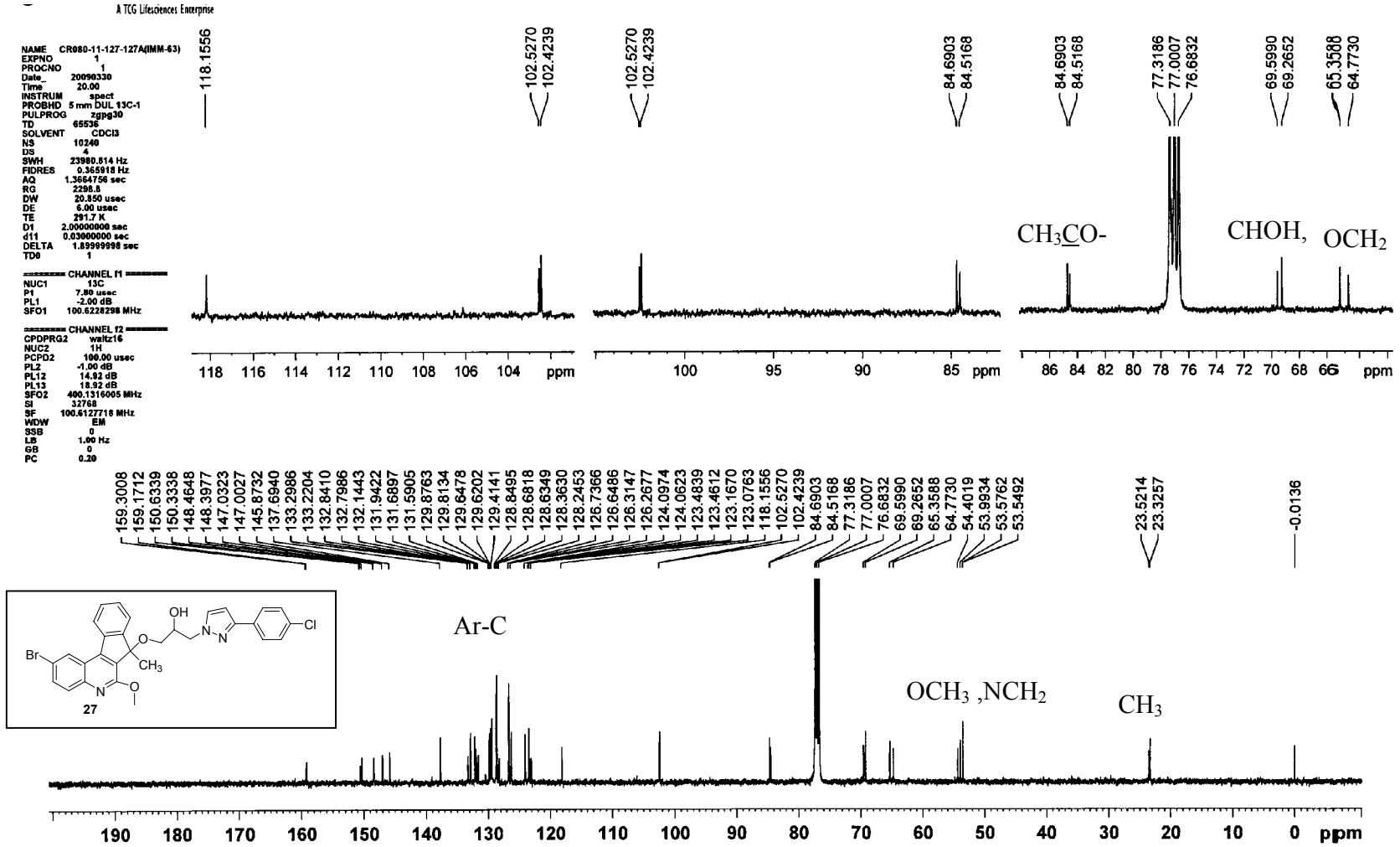
10 FEB 2009

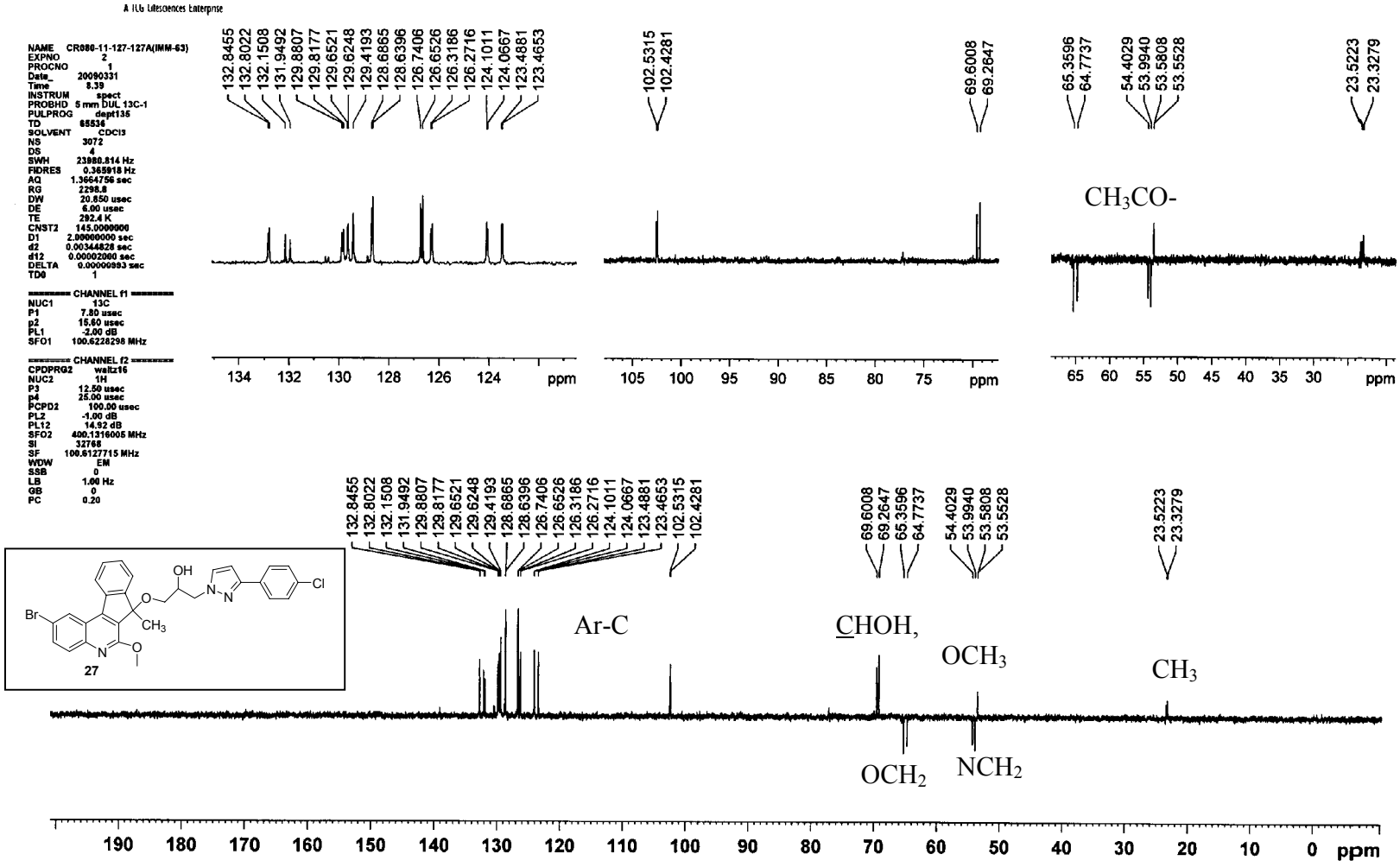
Page: 1 of 1

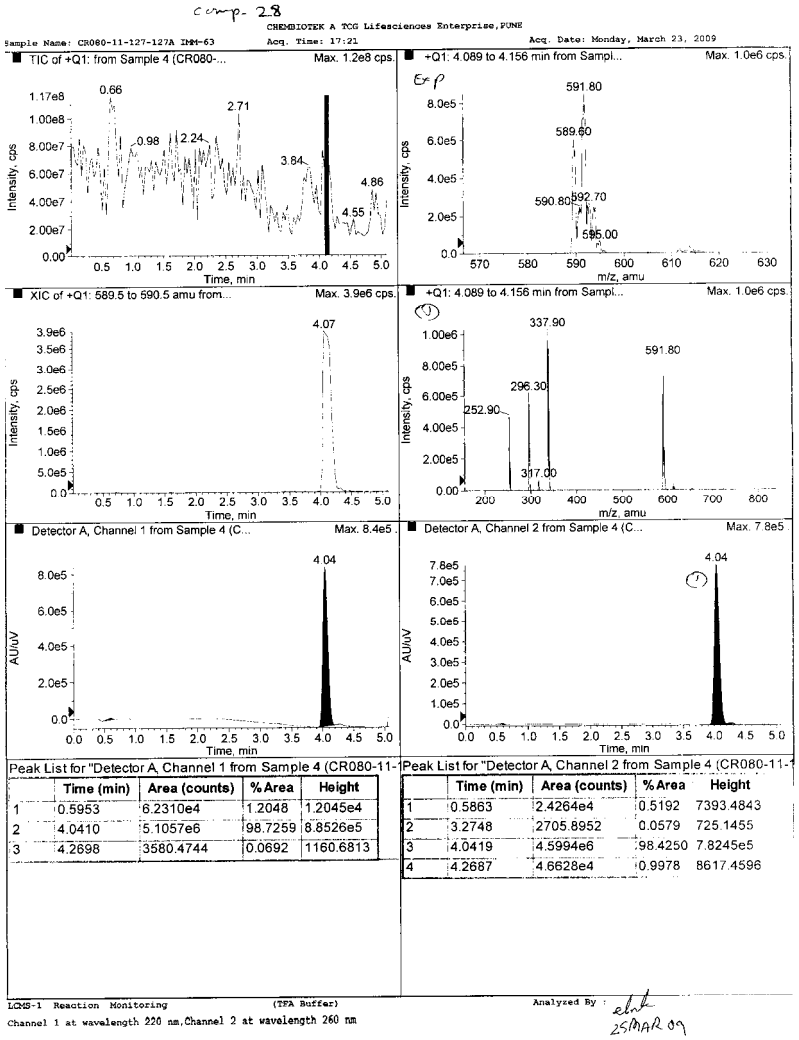

100.0
90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500 400.0
cm-1
3369.61
3066.84
2931.99
2867.60
2834.31
2363.54
2345.91
1912.88
1720.03
1702.04
1665.42
1604.44
1573.32
1523.20
1498.19
1467.02
1399.97
1365.23
1315.64
1274.74
1240.83
1210.48
1179.90
1152.25
1107.67
1069.96
1045.82
999.52
968.84
950.19
826.74
753.98
731.00
689.40
668.56
621.74
592.02
556.36
525.52
449.38
Br
OH
O
N
N
OMe
CH3
N
O
26


NAME CR080-11-127-127A(IMM-63)
EXPNO 1
PROCNO 1
Date_ 20090330
Time 11.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 645.1
DW 60.400 usec
DE 6.00 usec
TE 293.3 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
Ar-H
Pyrazole-CH
NCH2
CHOH, NCH2
CHOH
OCH2
OCH3
CH3
27
ppm


A TCG Lifesciences Enterprise
NAME CR080-11-127-127A(IMM-63)
EXPNO 2
PROCNO 1
Date_ 20090330
Time 19.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 456.1
DW 60.400 usec
DE 6.00 usec
TE 291.4 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
Ar-H
Pyrazole-CH
OCH3
CH3
NCH2
CHOH, NCH2
OCH2
ppm
Br
Cl
OH
N
O
CH3
27

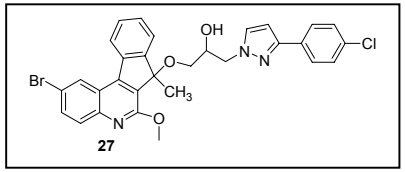

A TCG Lifesciences Enterprise
NAME CR080-11-127-127A(IMM-63)
EXPNO 1
PROCNO 1
Date_ 20090330
Time 20.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10240
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654756 sec
RG 2298.8
DW 20.850 usec
DE 6.00 usec
TE 291.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
118.1556
102.5270
102.4239
84.6903
84.5168
77.3186
77.0007
76.6832
69.5990
69.2652
65.3588
64.7730
CH3CO-
CHOH,
OCH2
ppm
159.3008
159.1712
150.6339
150.3338
148.4648
148.3977
147.0323
147.0027
145.8732
137.6940
133.2986
133.2204
132.8410
132.7986
132.1443
131.9422
131.6897
131.5905
129.8763
129.8134
129.6478
129.6202
129.4141
128.8495
128.6818
128.6349
128.3630
128.2453
126.7366
126.6486
126.3147
126.2677
124.0974
124.0623
123.4839
123.4612
123.1670
123.0763
118.1556
102.5270
102.4239
84.6903
84.5168
77.3186
77.0007
76.6832
69.5990
69.2652
65.3588
64.7730
54.4019
53.9934
53.5762
53.5492
23.5214
23.3257
-0.0136
Ar-C
OCH3 ,NCH2
CH3
ppm
27


A IUG Lifesciences Enterprise
NAME CR080-11-127-127A(IMM-63)
EXPNO 2
PROCNO 1
Date_ 20090331
Time 8.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2298.8
DW 20.850 usec
DE 6.00 usec
TE 292.4 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
132.8455 132.8022 132.1508 131.9492 129.8807 129.8177 129.6521 129.6248 129.4193 128.6865 128.6396 126.7406 126.6526 126.3186 126.2716 124.1011 124.0667 123.4881 123.4653
102.5315 102.4281
69.6008 69.2647
65.3596 64.7737
54.4029 53.9940 53.5808 53.5528
23.5223 23.3279
CH3CO-
134 132 130 128 126 124 ppm
105 100 95 90 85 80 75 ppm
65 60 55 50 45 40 35 30 ppm
OH
Cl
Br
CH3
O
N
27
Ar-C
CHOH,
OCH3
CH3
OCH2
NCH2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



	Time (min)	Area (counts)	% Area	Height
1	0.5953	6.2310e4	1.2048	1.2045e4
2	4.0410	5.1057e6	98.7259	8.8526e5
3	4.2698	3580.4744	0.0692	1160.6813

	Time (min)	Area (counts)	% Area	Height
1	0.5863	2.4264e4	0.5192	7393.4843
2	3.2748	2705.8952	0.0579	725.1455
3	4.0419	4.5994e6	98.4250	7.8245e5
4	4.2687	4.6628e4	0.9978	8617.4596



27

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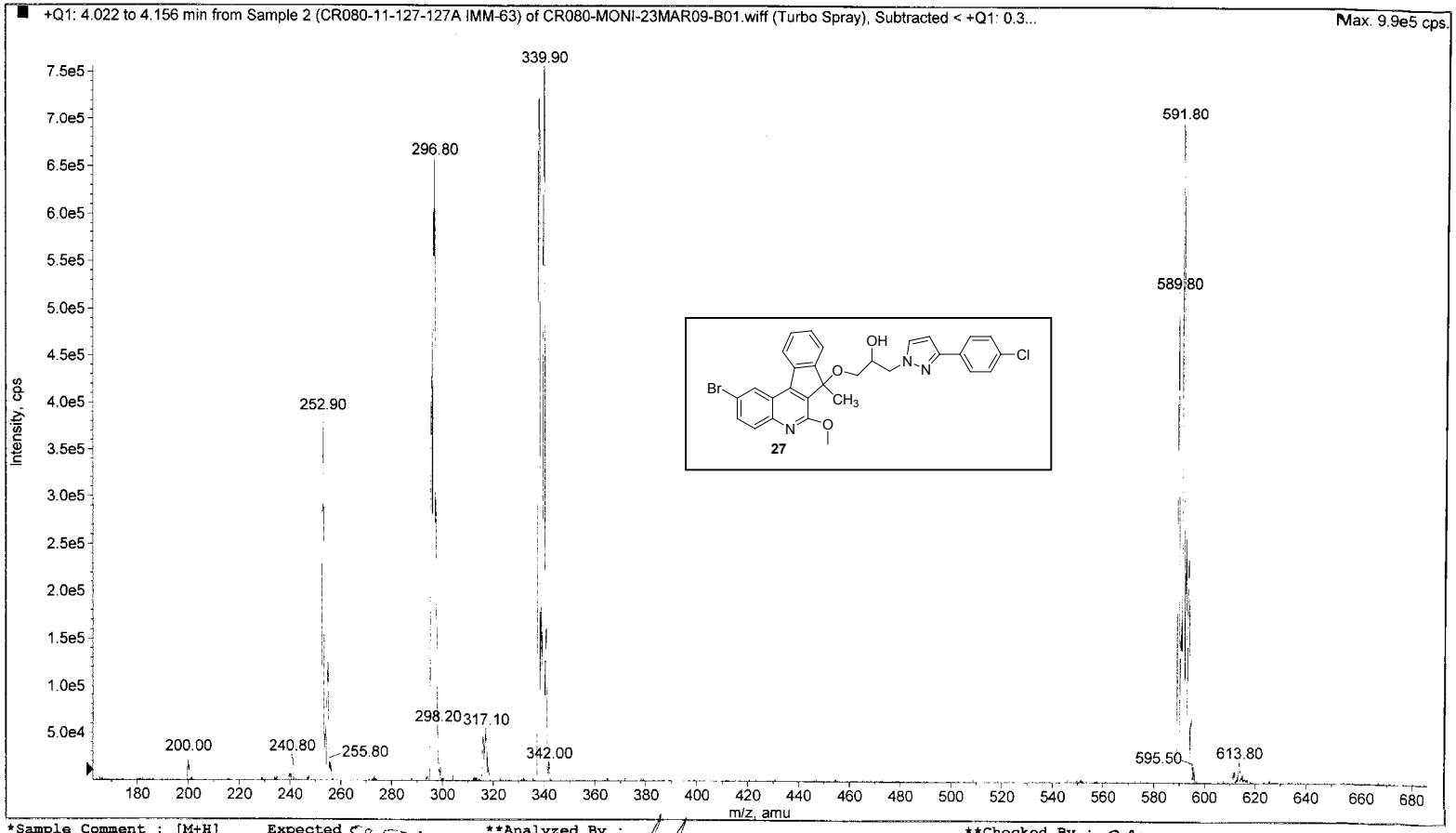
* **CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *

Sample Name: CR080-11-127-127A IMM-63 Acq. Time: 17:09 Acq. Date: Monday, March 23, 2009

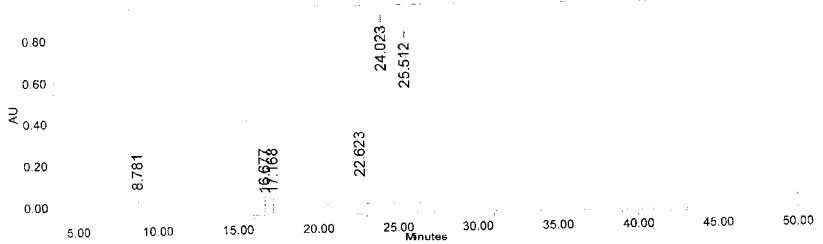


*Sample Comment : [M+H] Expected ~~578~~ 578 **Analyzed By : 23MAR09 **Checked By : 23rd March09

SAMPLE INFORMATION

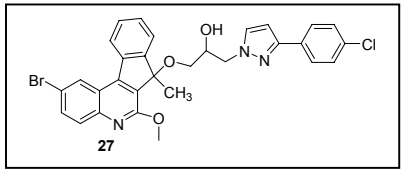
Sample Name	CR080/11//127/127A(IMM-63)	Acquired By	Ramudu
Sample Type	Unknown	Sample Set Name	P19NON039
Vial:	6	Acq. Method Set	P19_CR80_ARD25_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_127A_PRO
Injection Volume	10.00 ul	Label	P19090145
Run Time:	50.0 Minutes	Proc. Chanel	2998 Ch1 245nm@1.2nm
Date Acquired	2/12/2009 11:43:16 AM IST	Date Processed	2/12/2009 2:11:41 PM IST

Auto-Scaled Chromatogram



Peak Results

	RT	Area	% Area
1	8.781	161018	0.37
2	16.677	54771	0.13
3	17.168	41092	0.09
4	22.623	1605958	3.67
5	24.023	21860013	50.00
6	25.512	20001364	45.74



27

Reported by User : Ramudu Kurva (Ramudu)
Report Method : GENERAL_ASSAY_REPORT
Analysed by :
12 FEB 2009

Project Name: HPLC_P19_NON GLP
Date Printed : 2/12/2009
Checked by :

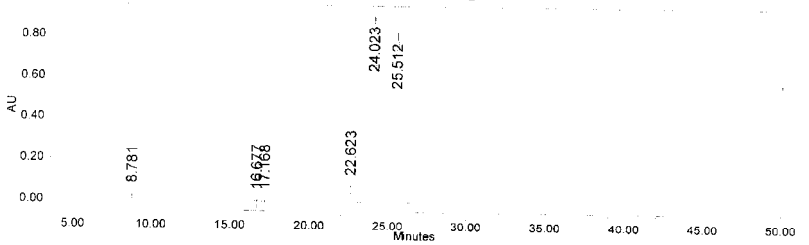
Page: 1 of 1

GENERAL_ASSAY_REPORT

SAMPLE INFORMATION

Sample Name	CR080/11//127/127A(IMM-63)	Acquired By	Ramudu
Sample Type	Unknown	Sample Set Name	P19NON039
Vial:	6	Acq. Method Set	P19_CR80_ARD25_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_127A_PRO
Injection Volume	10.00 ul	Label	P19090145
Run Time:	50.0 Minutes	Proc. Chanel	2998 Ch1 245nm@1.2nm
Date Acquired	2/12/2009 11:43:16 AM IST	Date Processed	2/12/2009 2:11:41 PM IST

Auto-Scaled Chromatogram



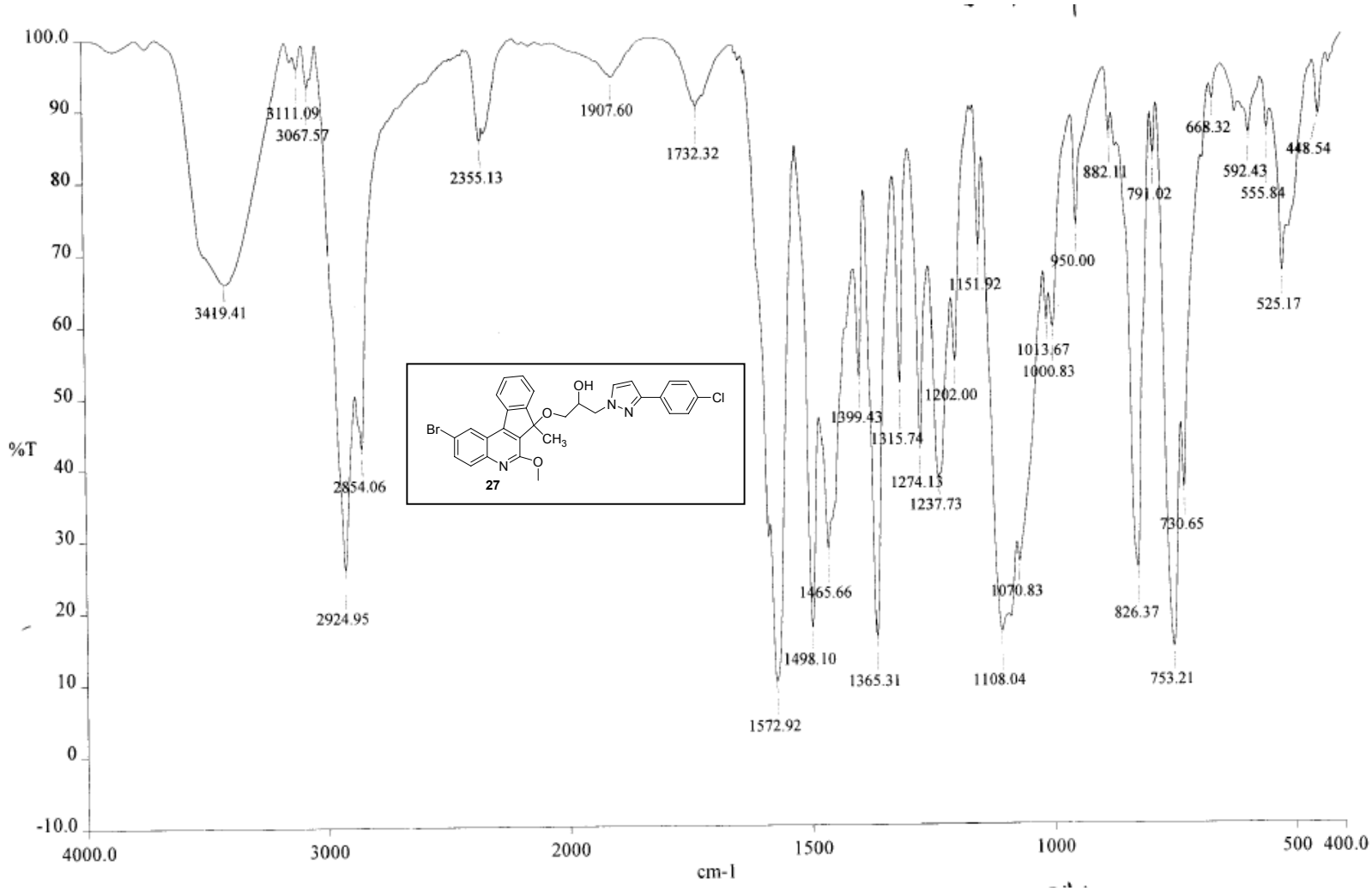
Peak Results

	RT	Area	% Area
1	8.781	161018	0.37
2	16.677	54771	0.13
3	17.168	41092	0.09
4	22.623	1605958	3.67
5	24.023	21860013	50.00
6	25.512	20001364	45.74

Reported by User : Ramudu Kurva (Ramudu)
Report Method : GENERAL_ASSAY_REPORT
Analysed by :
12 FEB 2009

Project Name: HPLC_P19_NON GLP
Date Printed : 2/12/2009
Checked by :

Page: 1 of 1


100.0
90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3419.41
3111.09
3067.57
2924.95
2854.06
2355.13
1907.60
1732.32
1572.92
1498.10
1465.66
1399.43
1365.31
1315.74
1274.13
1237.73
1202.00
1151.92
1108.04
1070.83
1013.67
1000.83
950.00
882.11
826.37
791.02
753.21
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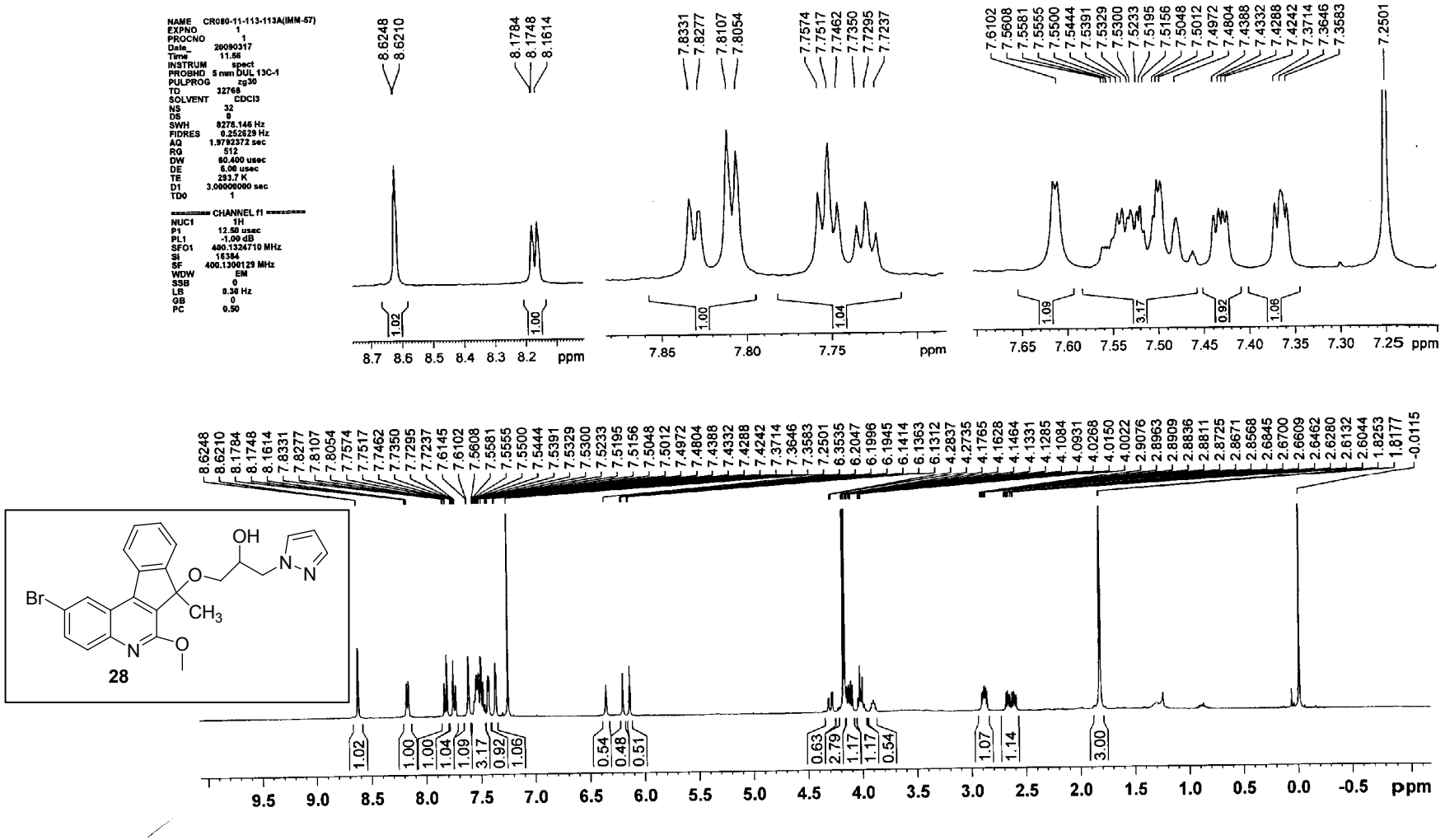
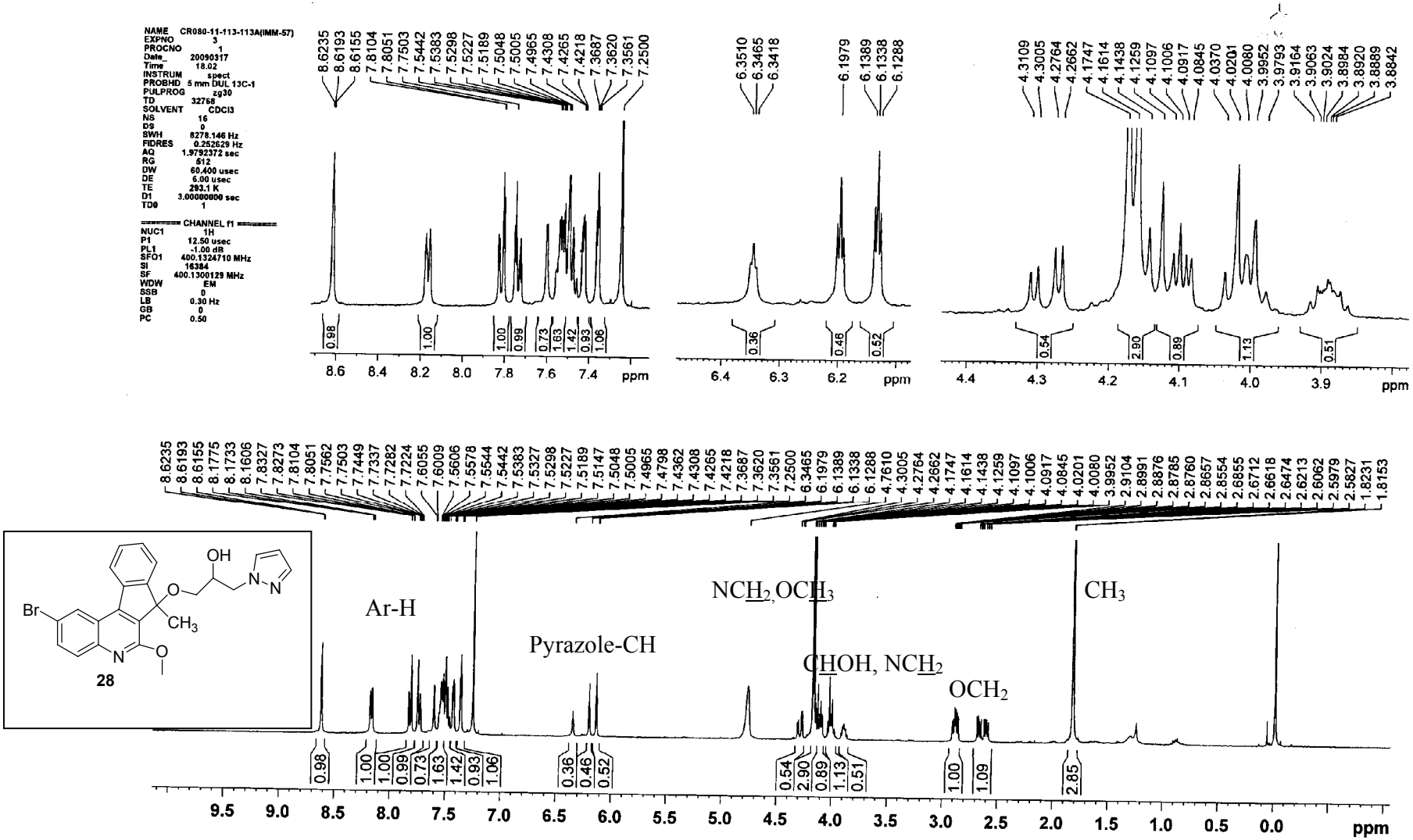
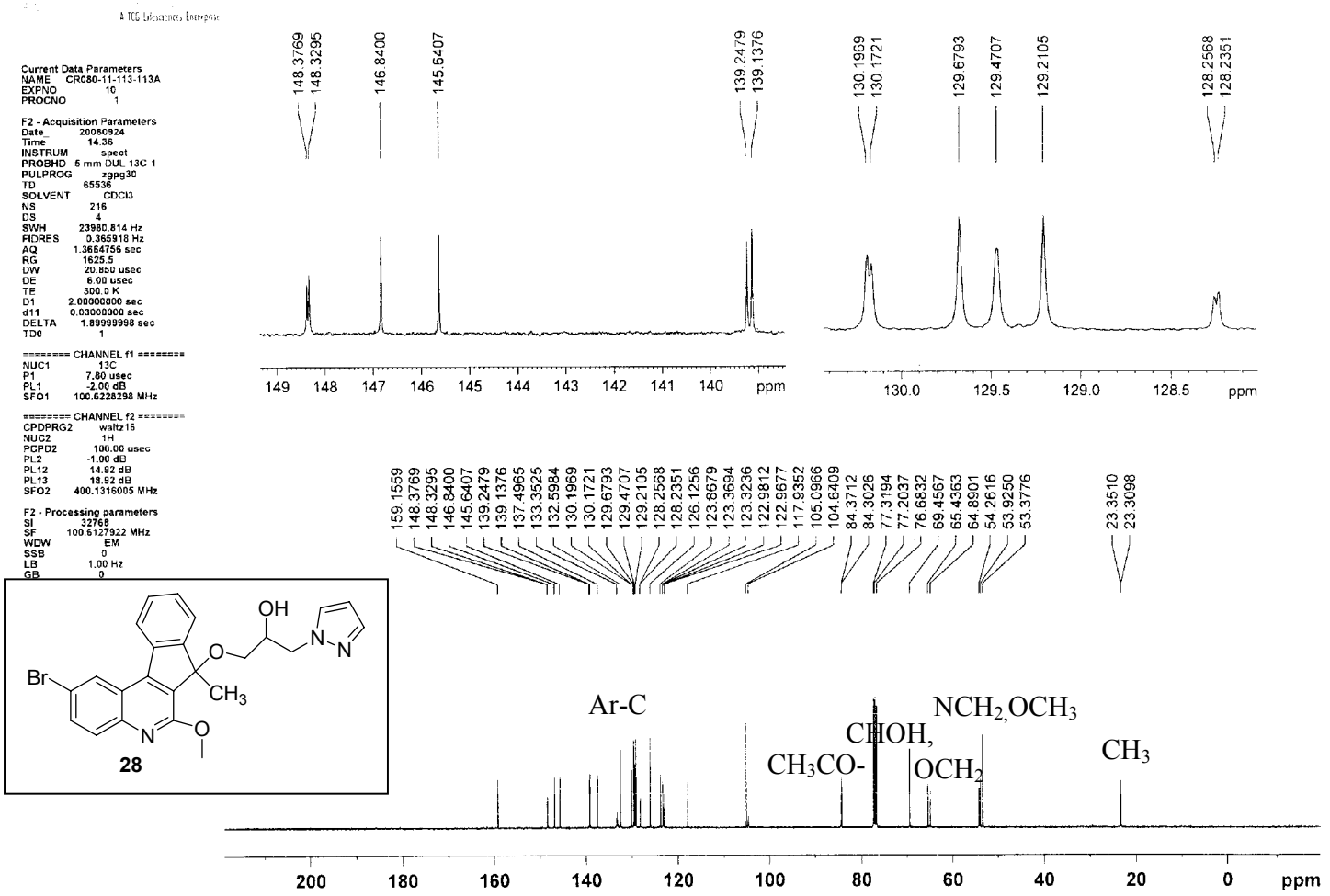
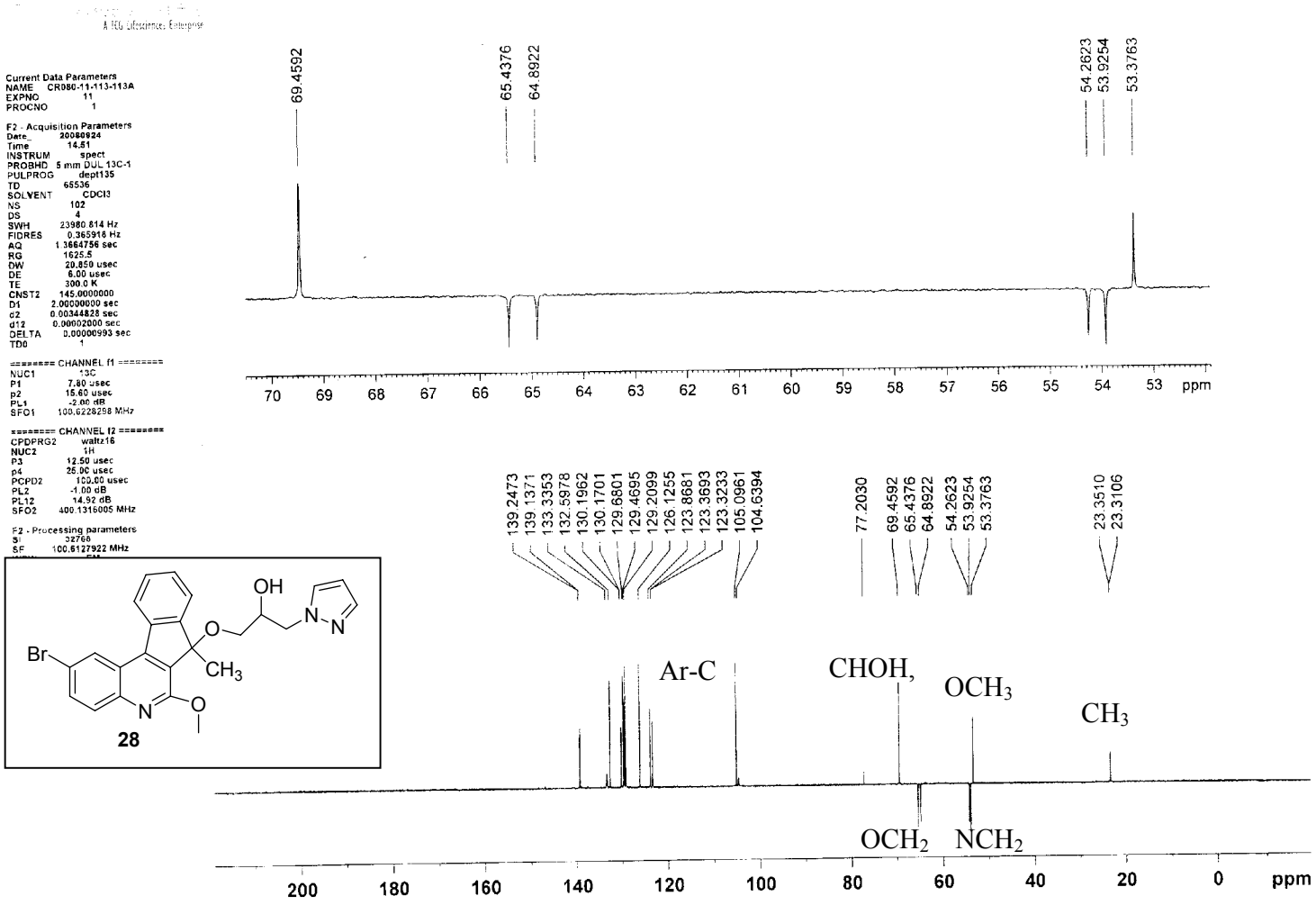
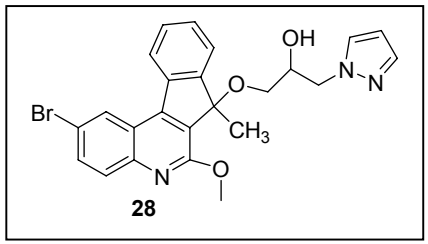
448.54
Br
CH3
O
OH
N
N
Cl
27


NAME CR080-11-113-113A(IMM-57)
EXPNO 1
PROCNO 1
Date_ 20090317
Time 11.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 512
DW 60.400 usec
DE 6.00 usec
TE 293.7 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
OH
N
N
O
Br
CH3
N
O
28
ppm

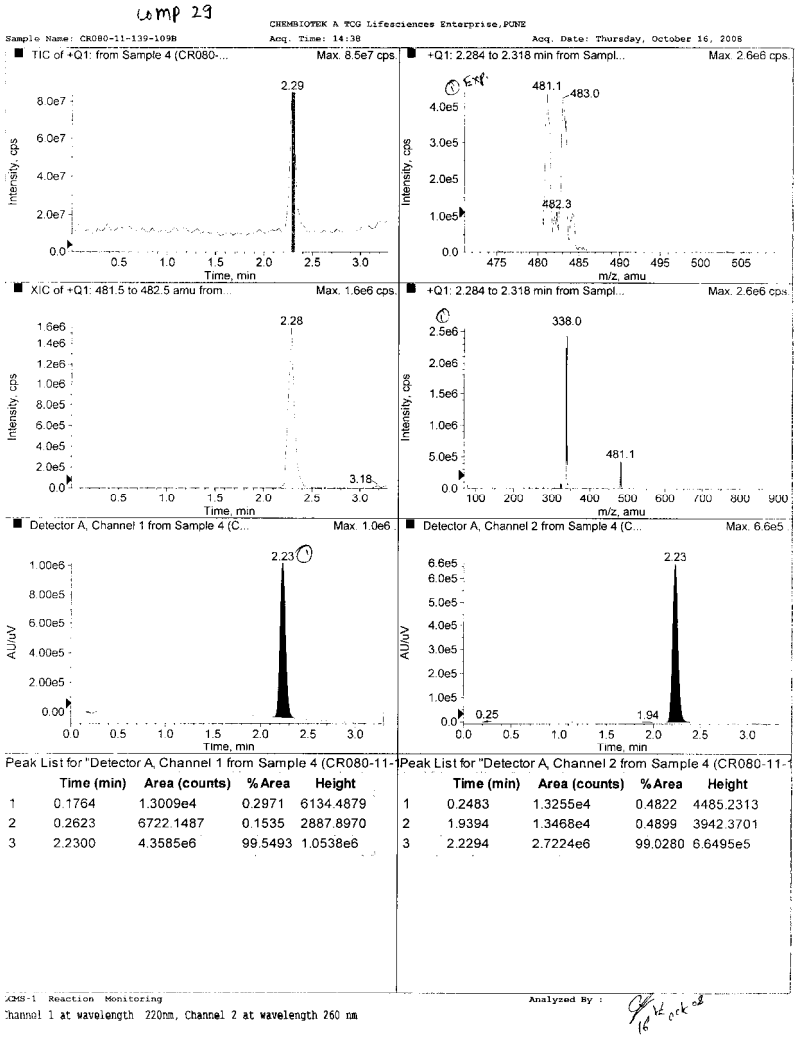

NAME CR080-11-113-113A(IMM-57)
EXPNO 3
PROCNO 1
Date_ 20090317
Time 18.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 512
DW 60.400 usec
DE 6.00 usec
TE 293.1 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
ppm
Br
OH
N
O
CH3
28
Ar-H
Pyrazole-CH
NCH2, OCH3
CHOH, NCH2
OCH2
CH3

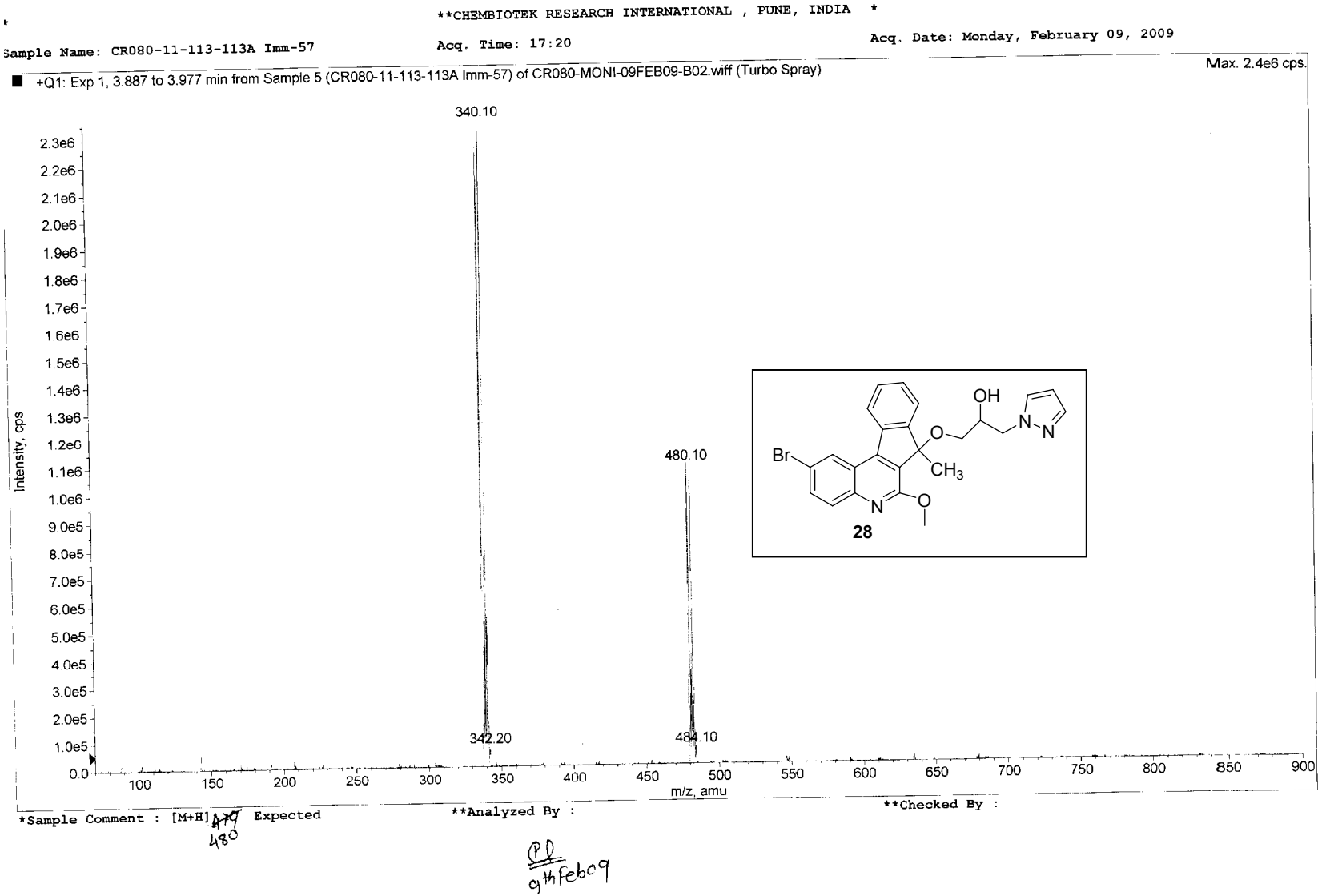

Current Data Parameters
NAME CR080-11-113-113A
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080924
Time 14.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 216
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
148.3769
148.3295
146.8400
145.6407
139.2479
139.1376
130.1969
130.1721
129.6793
129.4707
129.2105
128.2568
128.2351
149 148 147 146 145 144 143 142 141 140 ppm
130.0 129.5 129.0 128.5 ppm
159.1559
148.3769
148.3295
146.8400
145.6407
139.2479
139.1376
137.4965
133.3525
132.5984
130.1969
130.1721
129.6793
129.4707
129.2105
128.2568
128.2351
126.1256
123.8679
123.3694
123.3236
122.9812
122.9677
117.9352
105.0966
104.6409
84.3712
84.3026
77.3194
77.2037
76.6832
69.4567
65.4363
64.8901
54.2616
53.9250
53.3776
23.3510
23.3098
OH
O
N
N
Br
CH3
N
O
28
Ar-C
CH3CO-
CHOH,
OCH2
NCH2,OCH3
CH3
200 180 160 140 120 100 80 60 40 20 0 ppm

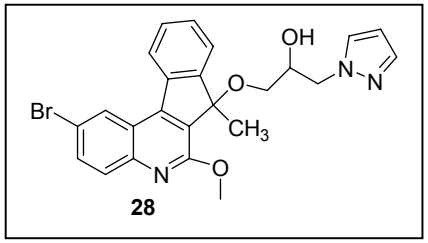

Current Data Parameters
NAME CR080-11-113-113A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080924
Time 14.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 23980.814 Hz
FIDRES 0.365916 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00006993 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127922 MHz
69.4592
65.4376
64.8922
54.2623
53.9254
53.3763
70 69 68 67 66 65 64 63 62 61 60 59 58 57 56 55 54 53 ppm
139.2473
139.1371
133.3353
132.5978
130.1962
130.1701
129.6801
129.4695
129.2099
126.1255
123.8681
123.3693
123.3233
105.0961
104.6394
77.2030
69.4592
65.4376
64.8922
54.2623
53.9254
53.3763
23.3510
23.3106
OH
N
N
O
Br
CH3
N
O
28
Ar-C
CHOH,
OCH3
CH3
OCH2
NCH2
200 180 160 140 120 100 80 60 40 20 0 ppm


OH
N
N
O
Br
CH3
N
O

28


comp 29
CHEMBIOTEK A TCG Lifesciences Enterprise,PUNE
Sample Name: CR080-11-139-109B
Acq. Time: 14:38
Acq. Date: Thursday, October 16, 2008
TIC of +Q1: from Sample 4 (CR080-...
Max. 8.5e7 cps.
2.29
Intensity, cps
Time, min
+Q1: 2.284 to 2.318 min from Sampl...
Max. 2.6e6 cps.
481.1
483.0
482.3
m/z, amu
XIC of +Q1: 481.5 to 482.5 amu from...
Max. 1.6e6 cps.
2.28
3.18
+Q1: 2.284 to 2.318 min from Sampl...
Max. 2.6e6 cps.
338.0
481.1
Detector A, Channel 1 from Sample 4 (C...
Max. 1.0e6
2.23
AU/uV
Detector A, Channel 2 from Sample 4 (C...
Max. 6.6e5
2.23
0.25
1.94
Peak List for "Detector A, Channel 1 from Sample 4 (CR080-11-1
Time (min) Area (counts) %Area Height
1 0.1764 1.3009e4 0.2971 6134.4879
2 0.2623 6722.1487 0.1535 2887.8970
3 2.2300 4.3585e6 99.5493 1.0538e6
Peak List for "Detector A, Channel 2 from Sample 4 (CR080-11-1
Time (min) Area (counts) %Area Height
1 0.2483 1.3255e4 0.4822 4485.2313
2 1.9394 1.3468e4 0.4899 3942.3701
3 2.2294 2.7224e6 99.0280 6.6495e5
XMS-1 Reaction Monitoring
Channel 1 at wavelength 220nm, Channel 2 at wavelength 260 nm
Analyzed By :

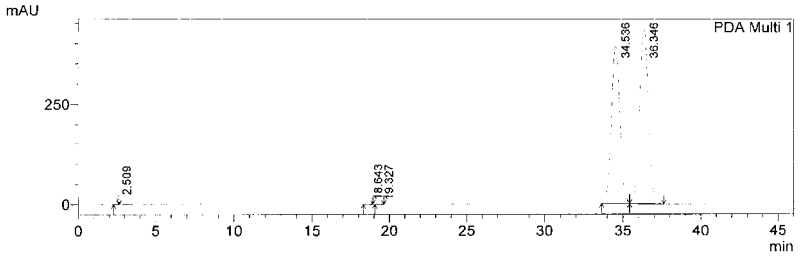

**CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *
Sample Name: CR080-11-113-113A Imm-57
Acq. Time: 17:20
Acq. Date: Monday, February 09, 2009
+Q1: Exp 1, 3.887 to 3.977 min from Sample 5 (CR080-11-113-113A Imm-57) of CR080-MONI-09FEB09-B02.wiff (Turbo Spray)
Max. 2.4e6 cps.
340.10
480.10
342.20
484.10
Intensity, cps
m/z, amu
Br
OH
O
N
N
CH3
O
28
*Sample Comment : [M+H] 479 480 Expected
**Analyzed By :
PD 9th Feb 09
**Checked By :



28

Sample Name : CR080-11-113-113A
Sample ID : CR080-11-113-113A
Column : Gemini C-18 (150 x 4.6 mm)
Vial # : 6
Inj. Volume : 4 uL
Tray # : 1
Acquired by : PANKAJ

Data File Name : 16_10_08_CR080-11-113-113A_05.lcd
Method File Name : CR080_01.lcm
Batch File Name : 16-10-2008.lcb
Data Acquired : 10/16/2008 8:46:00 PM
Data Processed : 10/17/2008 8:35:49 AM
Ref.No.: PAC/1140/65



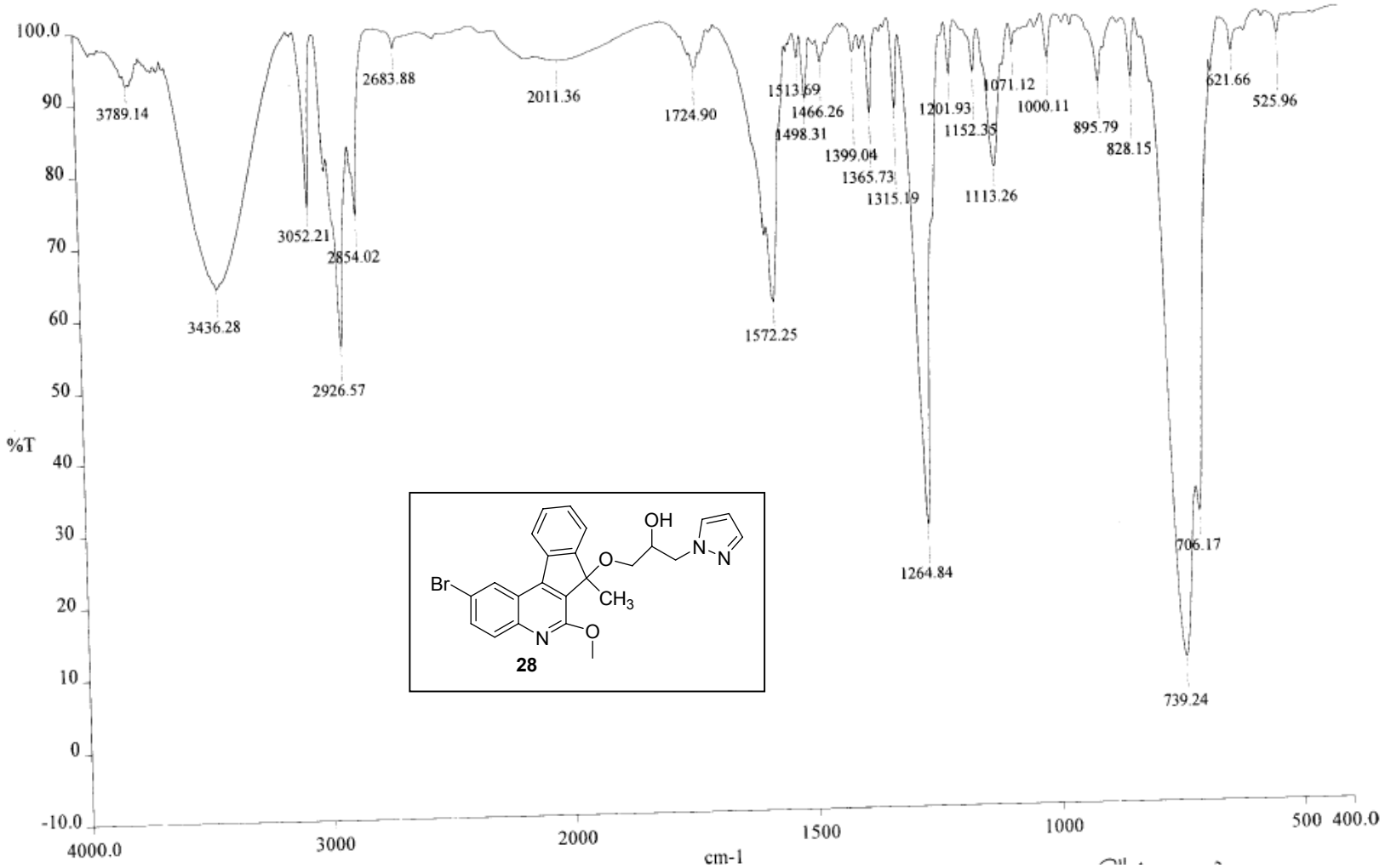
1 PDA Multi 1/227nm 4nm

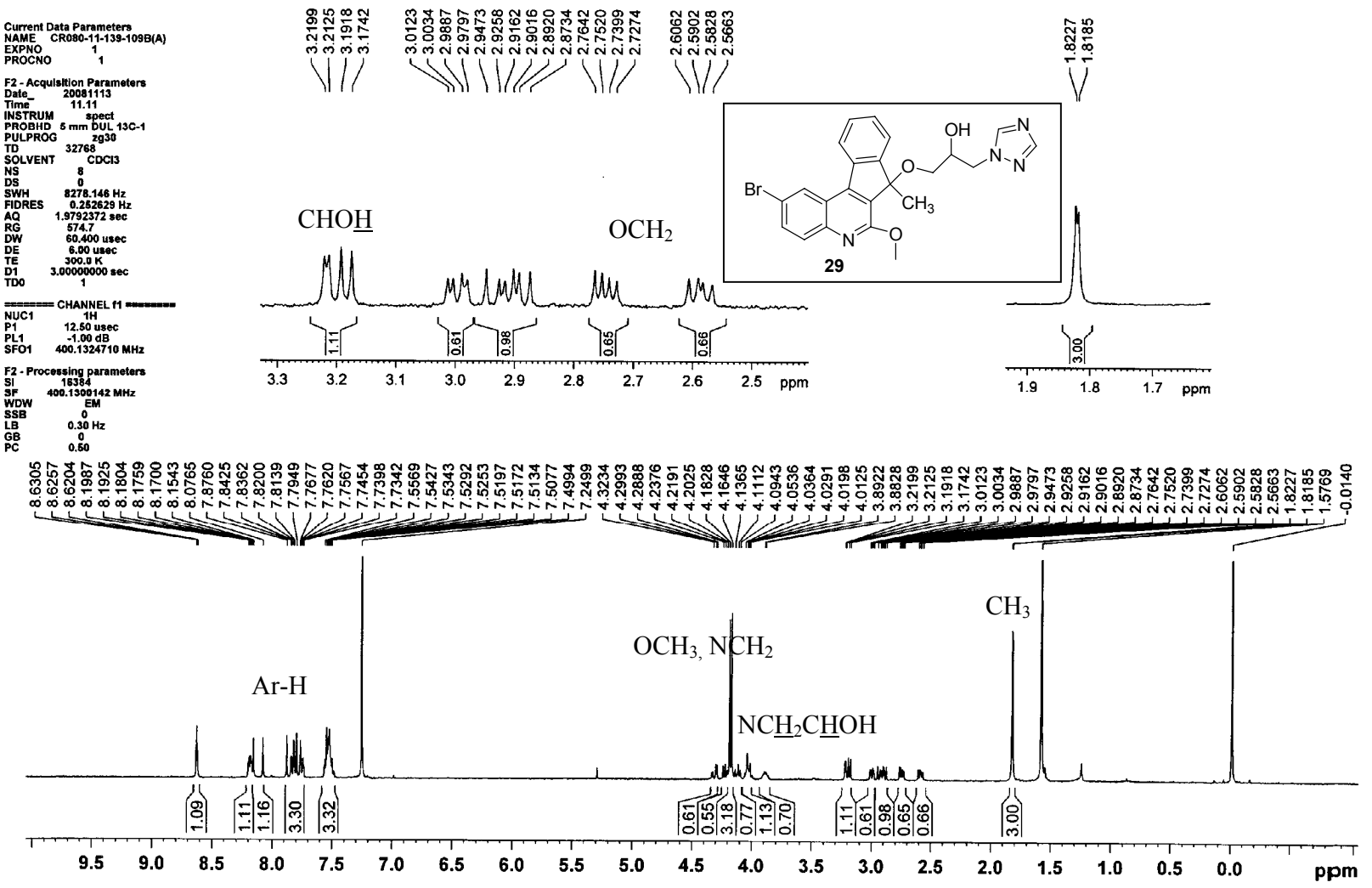
PeakTable

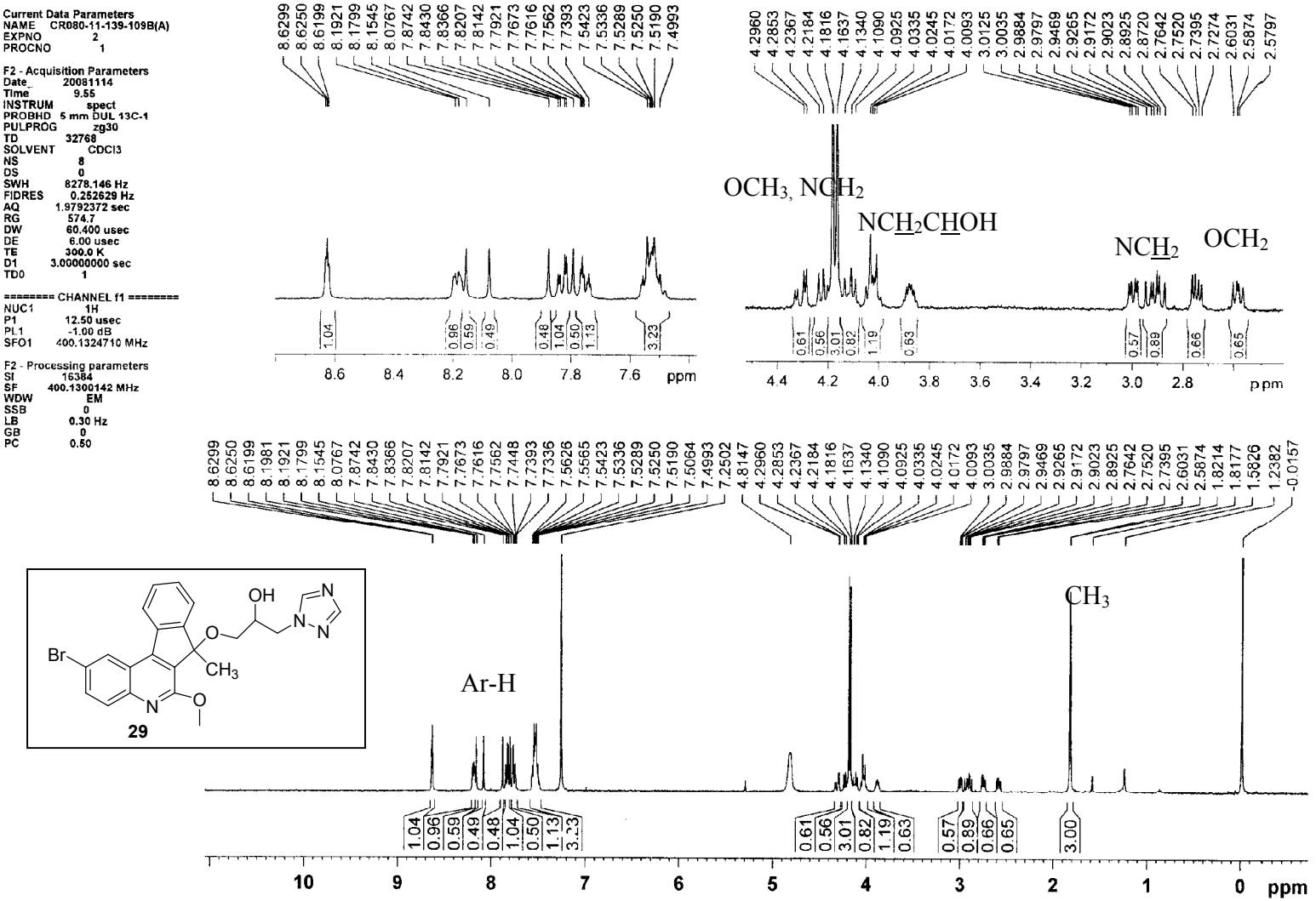
PDA Ch1 227nm 4nm

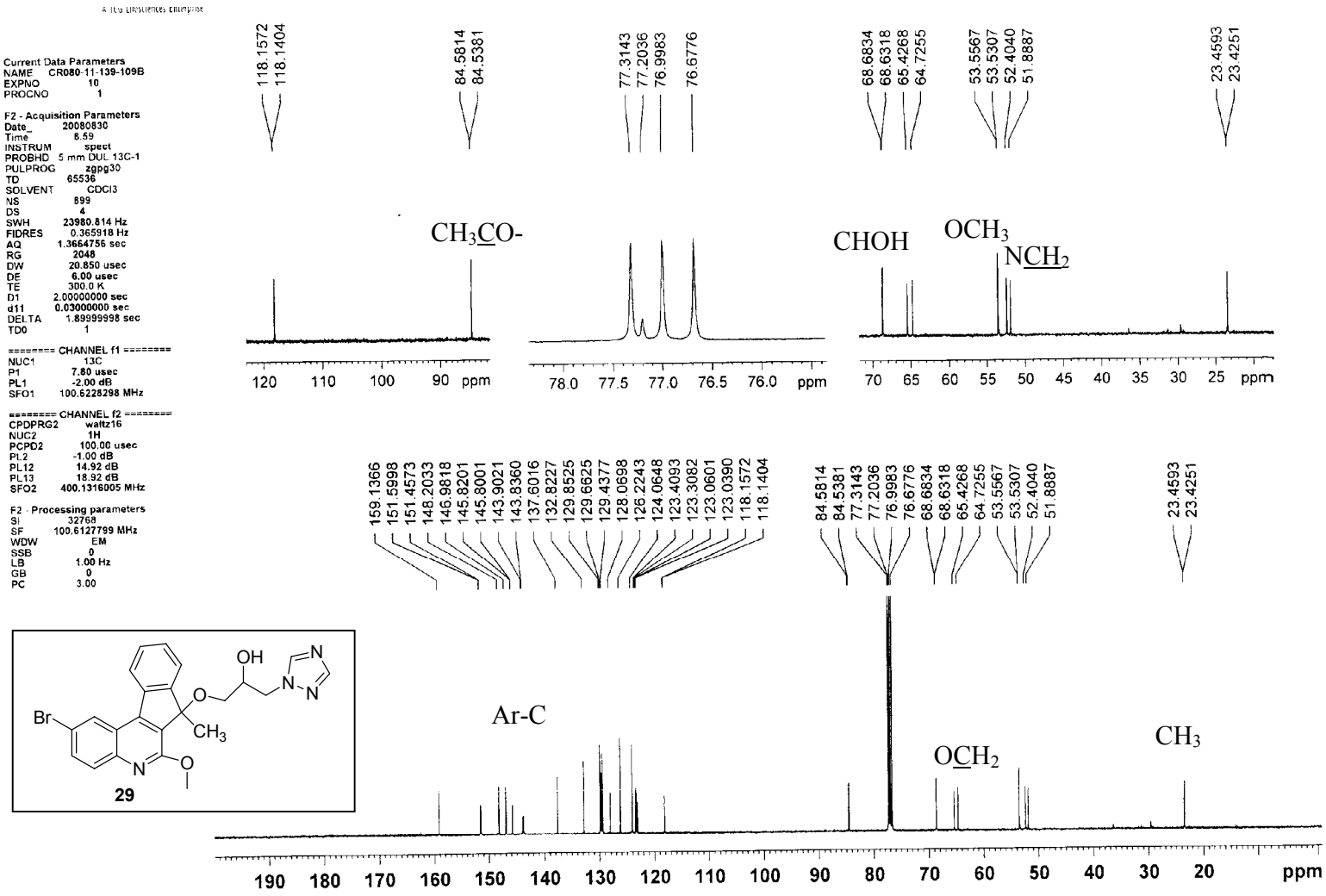
Peak#	Ret. Time	Area	Area %	Height
1	2.509	133246	0.412	15603
2	18.643	43867	0.136	2743
3	19.327	41409	0.128	2579
4	34.536	14730217	45.543	394840
5	36.346	17395006	53.782	436104
Total		32343745	100.000	851869

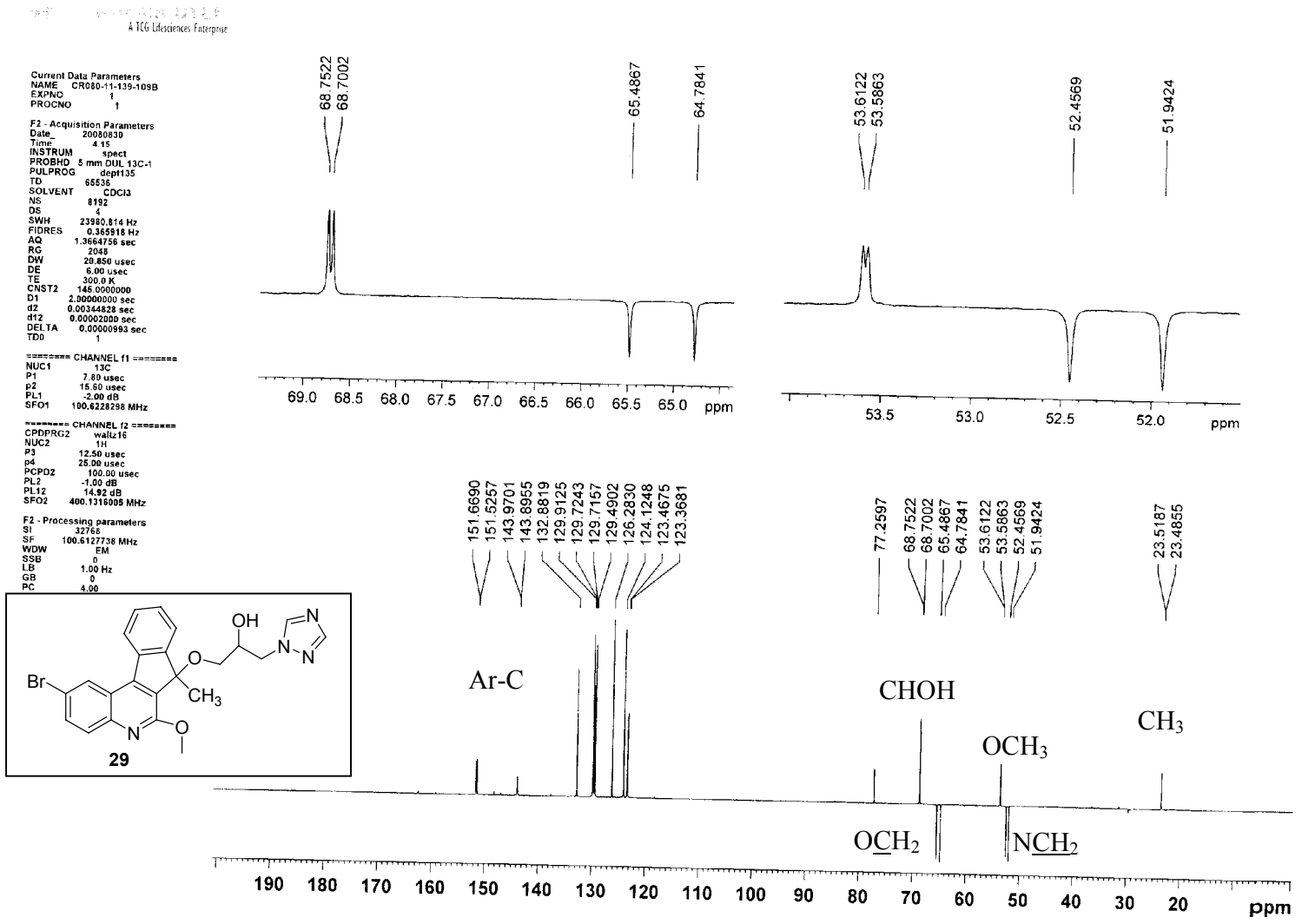
17th October

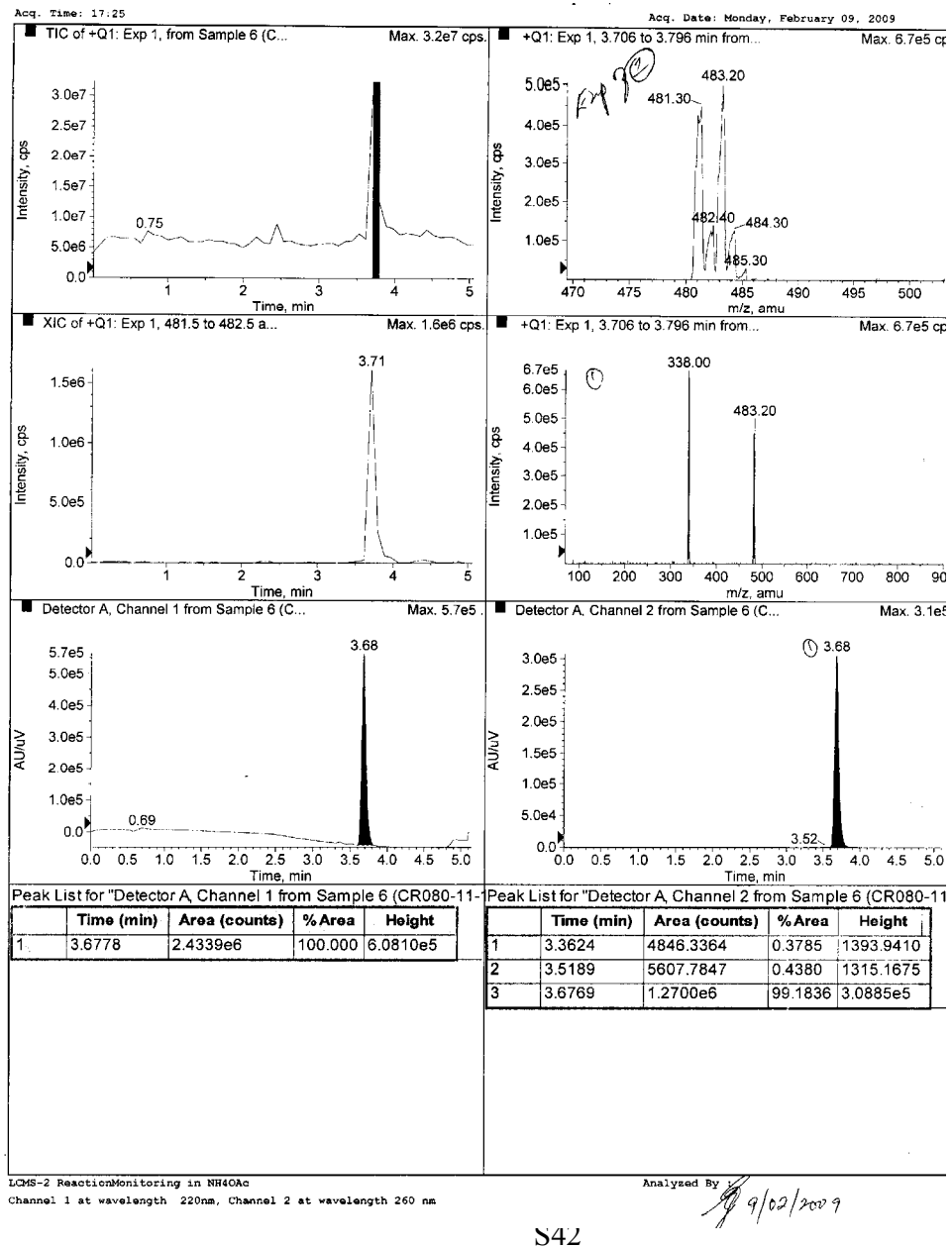

100.0
90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500 400.0
cm-1
3789.14
3436.28
3052.21
2926.57
2854.02
2683.88
2011.36
1724.90
1572.25
1513.69
1498.31
1466.26
1399.04
1365.73
1315.19
1264.84
1201.93
1152.35
1113.26
1071.12
1000.11
895.79
828.15
739.24
706.17
621.66
525.96
Br
O
OH
N
N
CH3
O
28

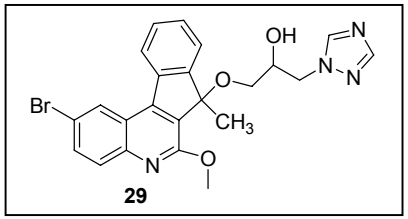

Current Data Parameters
NAME CR080-11-139-109B(A)
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081113
Time 11.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
CHOH
OCH2
29
Ar-H
OCH3, NCH2
NCH2CHOH
CH3
ppm


Current Data Parameters
NAME CR080-11-139-109B(A)
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081114
Time 9.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
OCH3, NCH2
NCH2CHOH
NCH2
OCH2
Ar-H
CH3
ppm
29

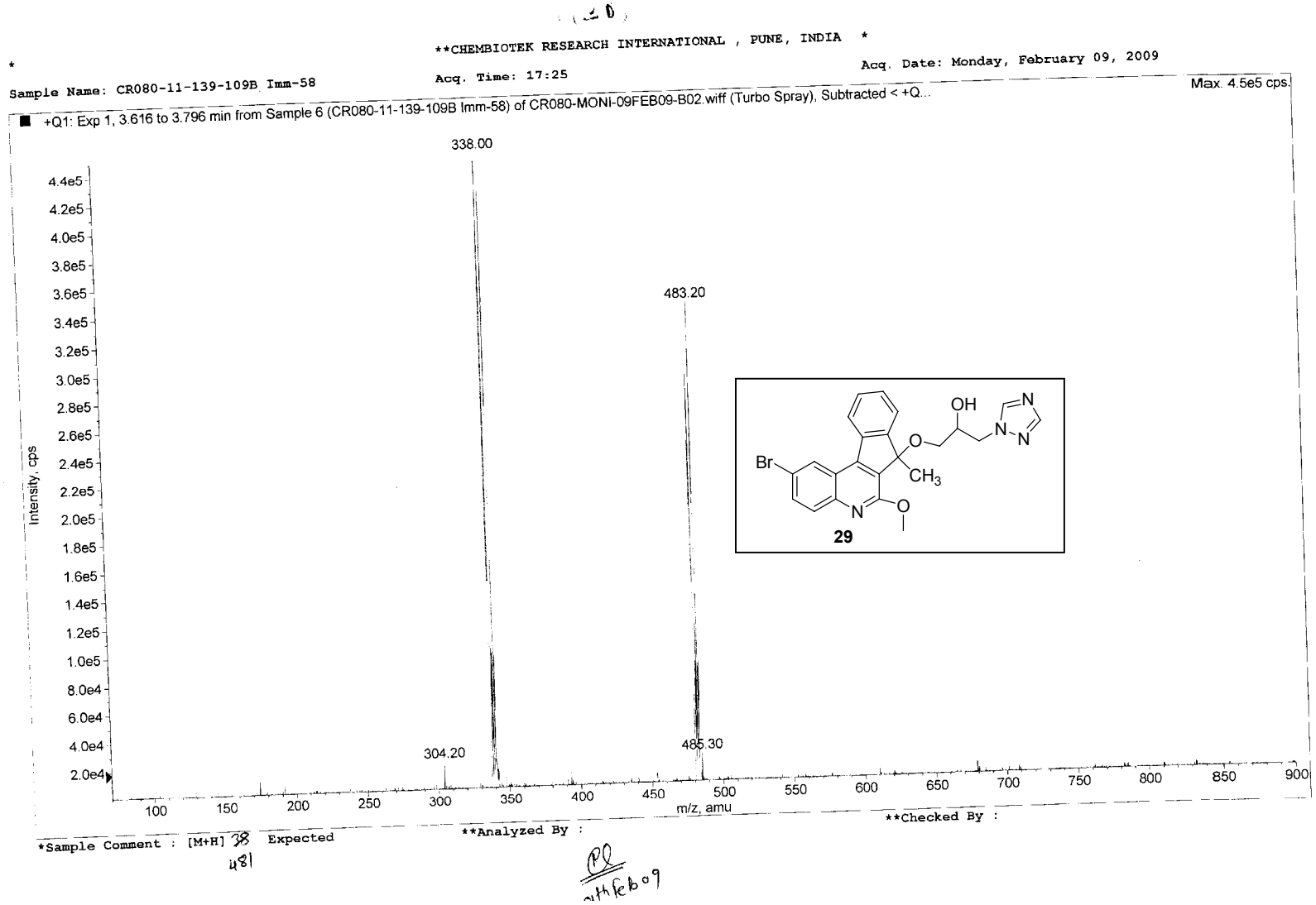

Current Data Parameters
NAME CR080-11-139-109B
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080830
Time 8.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 899
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127799 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00
118.1572
118.1404
84.5814
84.5381
77.3143
77.2036
76.9983
76.6776
68.6834
68.6318
65.4268
64.7255
53.5567
53.5307
52.4040
51.8887
23.4593
23.4251
CH3CO-
CHOH
OCH3
NCH2
120 110 100 90 ppm
78.0 77.5 77.0 76.5 76.0 ppm
70 65 60 55 50 45 40 35 30 25 ppm
159.1366
151.5998
151.4573
148.2033
146.9818
145.8201
145.8001
143.9021
143.8360
137.6016
132.8227
129.8525
129.6625
129.4377
128.0698
126.2243
124.0648
123.4093
123.3082
123.0601
123.0390
118.1572
118.1404
Br
OH
N
N
N
O
O
CH3
29
Ar-C
OCH2
CH3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


A TCG Lifesciences Enterprise
Current Data Parameters
NAME CR080-11-139-109B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080830
Time 4.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 8192
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127738 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00
68.7522
68.7002
65.4867
64.7841
53.6122
53.5863
52.4569
51.9424
69.0 68.5 68.0 67.5 67.0 66.5 66.0 65.5 65.0 ppm
53.5 53.0 52.5 52.0 ppm
151.6690
151.5257
143.9701
143.8955
132.8819
129.9125
129.7243
129.7157
129.4902
126.2830
124.1248
123.4675
123.3681
77.2597
68.7522
68.7002
65.4867
64.7841
53.6122
53.5863
52.4569
51.9424
23.5187
23.4855
OH
N
N
N
O
Br
CH3
N
O
29
Ar-C
CHOH
OCH3
CH3
OCH2
NCH2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


Acq. Time: 17:25
Acq. Date: Monday, February 09, 2009
TIC of +Q1: Exp 1, from Sample 6 (C...
Max. 3.2e7 cps.
Intensity, cps
Time, min
0.75
+Q1: Exp 1, 3.706 to 3.796 min from...
Max. 6.7e5 cp
483.20
481.30
482.40
484.30
485.30
m/z, amu
XIC of +Q1: Exp 1, 481.5 to 482.5 a...
Max. 1.6e6 cps.
3.71
+Q1: Exp 1, 3.706 to 3.796 min from...
Max. 6.7e5 cp
338.00
483.20
Detector A, Channel 1 from Sample 6 (C...
Max. 5.7e5
AU/uV
3.68
0.69
Detector A, Channel 2 from Sample 6 (C...
Max. 3.1e5
3.68
3.52
Peak List for "Detector A, Channel 1 from Sample 6 (CR080-11-
Time (min) | Area (counts) | % Area | Height
1 | 3.6778 | 2.4339e6 | 100.000 | 6.0810e5
Peak List for "Detector A, Channel 2 from Sample 6 (CR080-11
Time (min) | Area (counts) | % Area | Height
1 | 3.3624 | 4846.3364 | 0.3785 | 1393.9410
2 | 3.5189 | 5607.7847 | 0.4380 | 1315.1675
3 | 3.6769 | 1.2700e6 | 99.1836 | 3.0885e5
LCMS-2 ReactionMonitoring in NH4OAc
Channel 1 at wavelength 220nm, Channel 2 at wavelength 260 nm
Analyzed By
9/02/2009


OH
N
N
N
O
Br
CH3
N
O

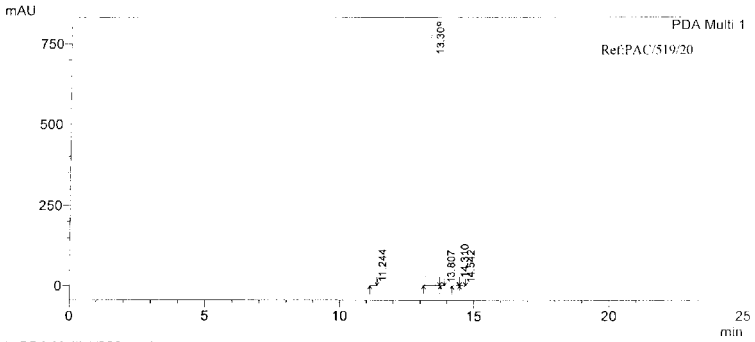
29


**CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *
Sample Name: CR080-11-139-109B Imm-58
Acq. Time: 17:25
Acq. Date: Monday, February 09, 2009
Max. 4.5e5 cps.
+Q1: Exp 1, 3.616 to 3.796 min from Sample 6 (CR080-11-139-109B Imm-58) of CR080-MONI-09FEB09-B02.wiff (Turbo Spray), Subtracted < +Q...
338.00
483.20
304.20
485.30
Intensity, cps
m/z, amu
29
*Sample Comment : [M+H] 481 Expected
**Analyzed By :
**Checked By :

Sample Name : CR080-11-109-109A
Sample ID : CR080-11-109-109A
Column : Zorbax SB C-18 (250 x 4.6 mm)
Vial # : 47
Inj. Volume : 1 uL
Tray # : 1
Acquired by : Pankaj

Data File Name : 21_06_07_CR080-11-109-109A_01.lcd
Method File Name : CR637_05.lcm
Batch File Name : 21_06_07.lcb
Data Acquired : 6/22/2007 12:32:28 AM
Data Processed : 6/22/2007 1:03:32 AM

<Chromatogram>



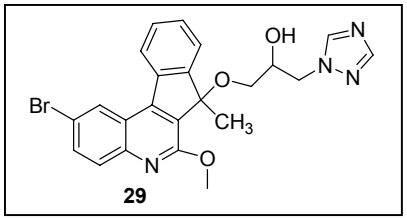
1 PDA Multi 1/228nm 4nm

PeakTable

PDA Ch1 228nm 4nm

Peak#	Ret. Time	Area	Area %	Height
1	11.244	21408	0.351	3201
2	13.308	5948917	97.600	785107
3	13.807	2811	0.046	570
4	14.310	118765	1.948	18781
5	14.542	3309	0.054	466
Total		6095209	100.000	808125

22nd June 07



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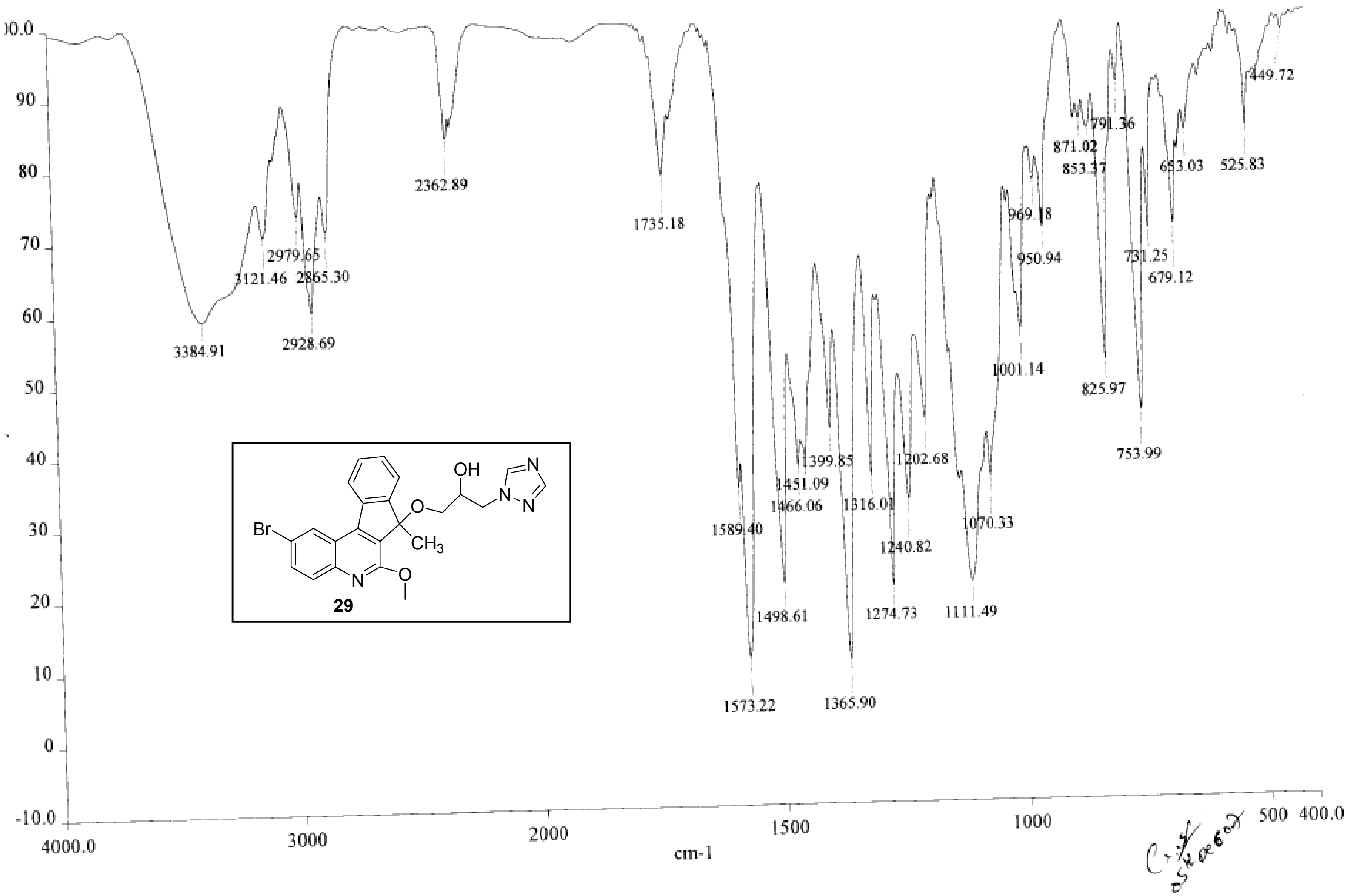
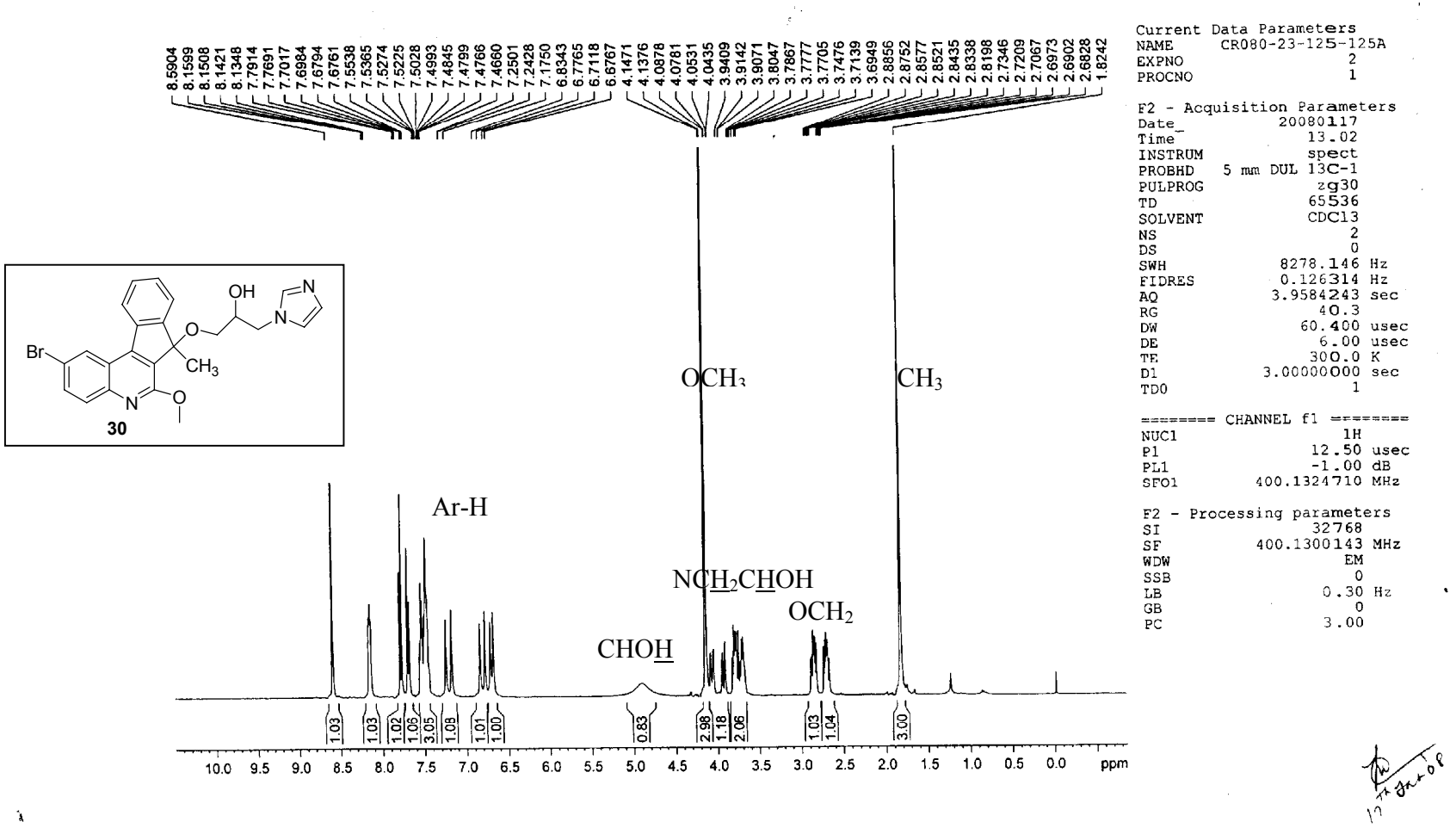
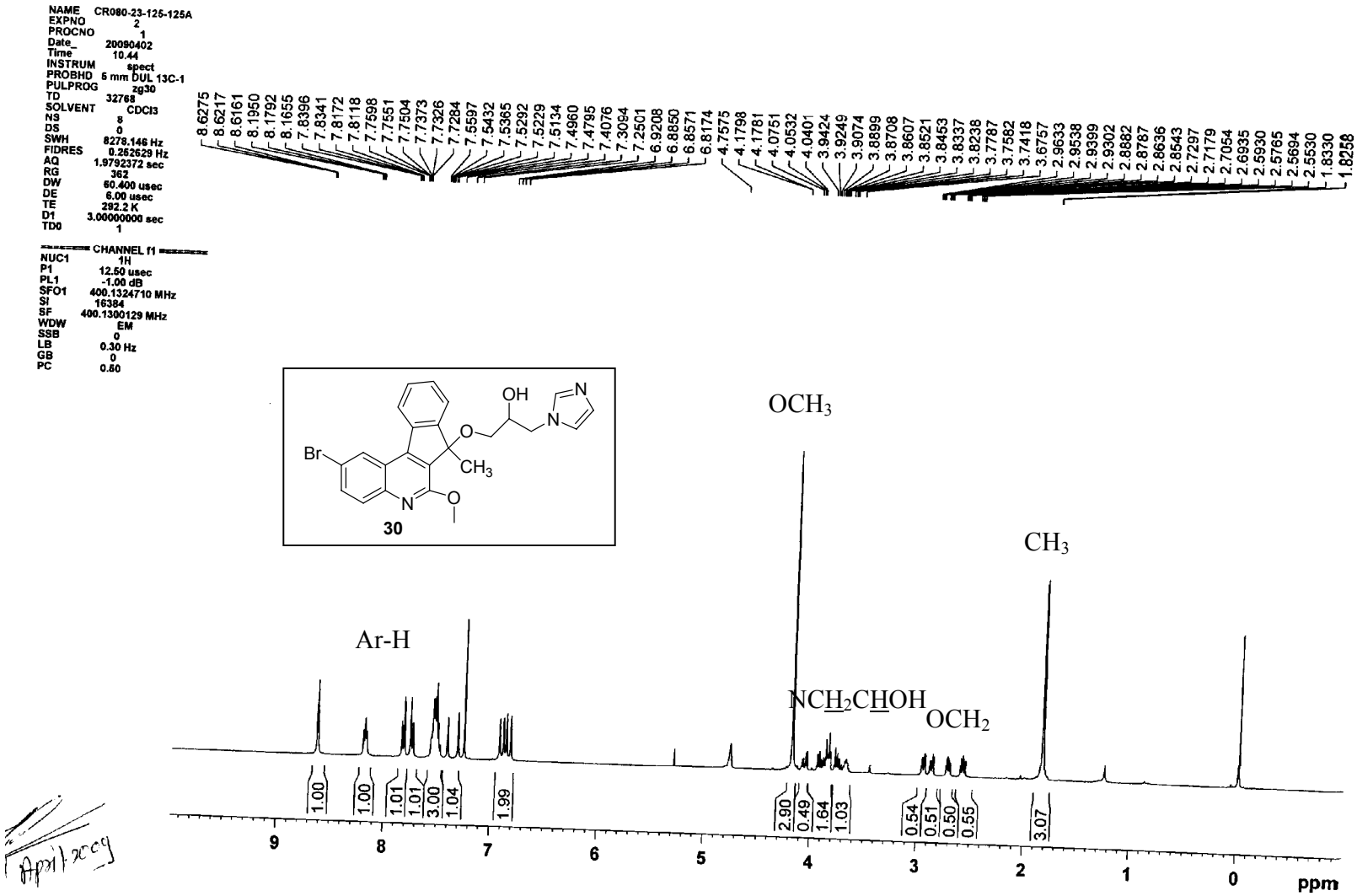
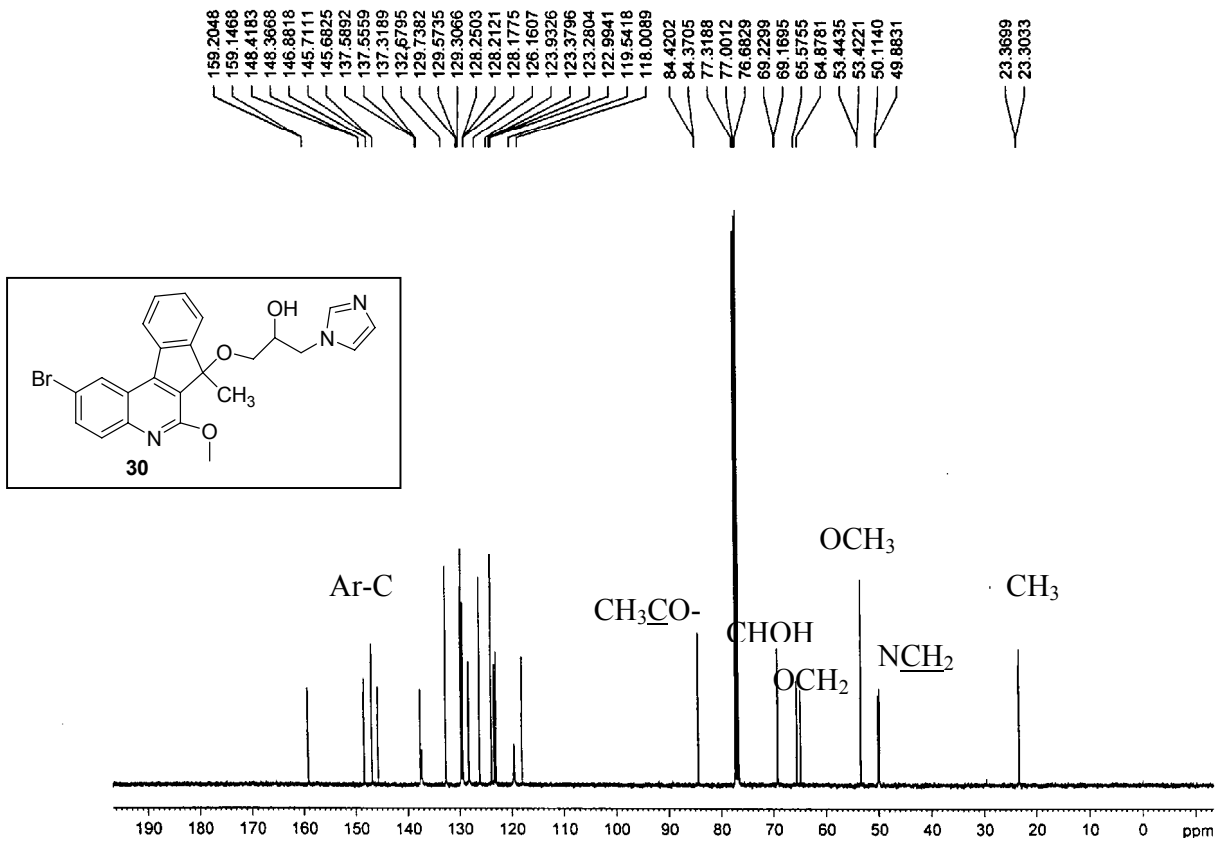

3384.91
3121.46
2979.65
2928.69
2865.30
2362.89
1735.18
1589.40
1573.22
1498.61
1466.06
1451.09
1399.85
1365.90
1316.01
1274.73
1240.82
1202.68
1111.49
1070.33
1001.14
969.18
950.94
871.02
853.37
825.97
791.36
753.99
731.25
679.12
653.03
525.83
449.72
Br
OH
O
N
CH3
29
cm-1
4000.0
3000
2000
1500
1000
500
400.0

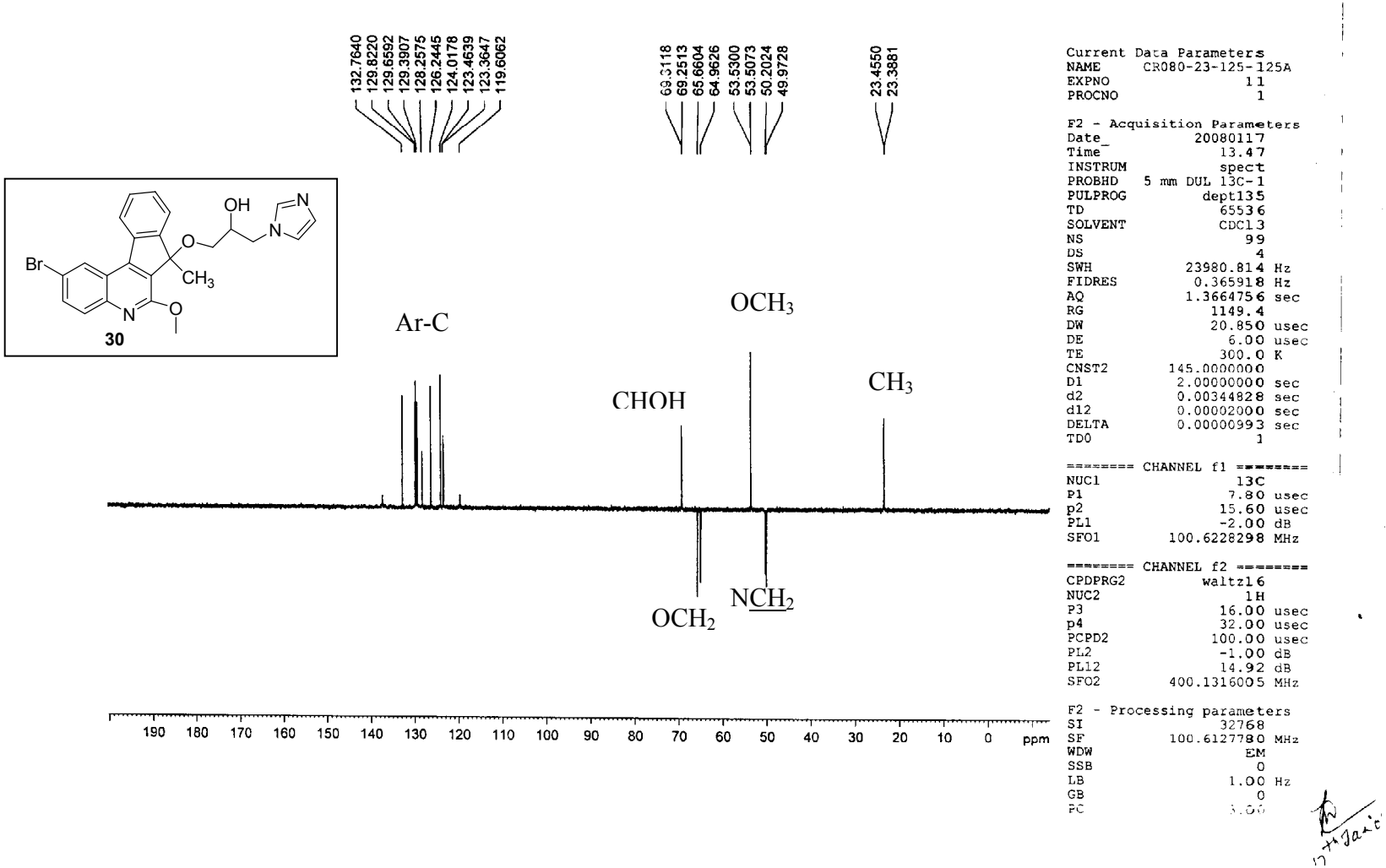


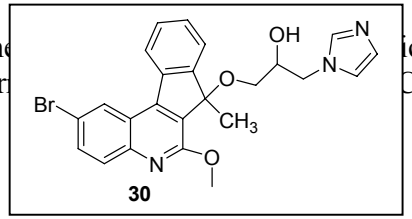

```
Current Data Parameters
NAME      CR080-23-125-125A
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20080117
Time                13.02
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      2
DS                      0
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                   40.3
DW                 60.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            3.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.50 usec
PL1                -1.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300143 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   3.00
```


NAME CR080-23-125-125A
EXPNO 2
PROCNO 1
Date_ 20090402
Time 10.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 292.2 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80
8.6275
8.6217
8.6161
8.1950
8.1792
8.1655
7.8396
7.8341
7.8172
7.8118
7.7598
7.7551
7.7504
7.7373
7.7326
7.7284
7.5597
7.5432
7.5365
7.5292
7.5229
7.5134
7.4960
7.4795
7.4076
7.3064
7.2501
6.9208
6.8850
6.8571
6.8174
4.7575
4.1798
4.1781
4.0751
4.0532
4.0401
3.9424
3.9249
3.9074
3.8899
3.8708
3.8607
3.8521
3.8453
3.8337
3.8238
3.7787
3.7582
3.7418
3.6757
2.9633
2.9538
2.9399
2.9302
2.8882
2.8787
2.8636
2.8543
2.7297
2.7179
2.7054
2.6935
2.5930
2.5765
2.5694
2.5530
1.8330
1.8258
Br
OH
N
O
CH3
N
O
30
OCH3
CH3
Ar-H
NCH2CHOH
OCH2
1.00
1.00
1.01
1.01
3.00
1.04
1.99
2.90
0.49
1.64
1.03
0.54
0.51
0.50
0.55
3.07
9
8
7
6
5
4
3
2
1
0
ppm




```
Current Data Parameters
NAME      CR080-23-125-125A
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20080117
Time                13.10
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  722
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                1149.4
DW               20.850 usec
DE                6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.80 usec
PL1                -2.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -1.00 dB
PL12               14.92 dB
PL13               18.92 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF          100.6127865 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```


132.7640
129.8220
129.6592
129.3907
128.2575
126.2445
124.0178
123.4639
123.3647
119.6062
69.3118
69.2513
65.6604
64.9626
53.5300
53.5073
50.2024
49.9728
23.4550
23.3881
Br
OH
N
N
O
CH3
N
O
30
Ar-C
OCH3
CHOH
CH3
OCH2
NCH2
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 CR080-23-125-125A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080117
Time 13.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 99
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 16.00 usec
p4 32.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00

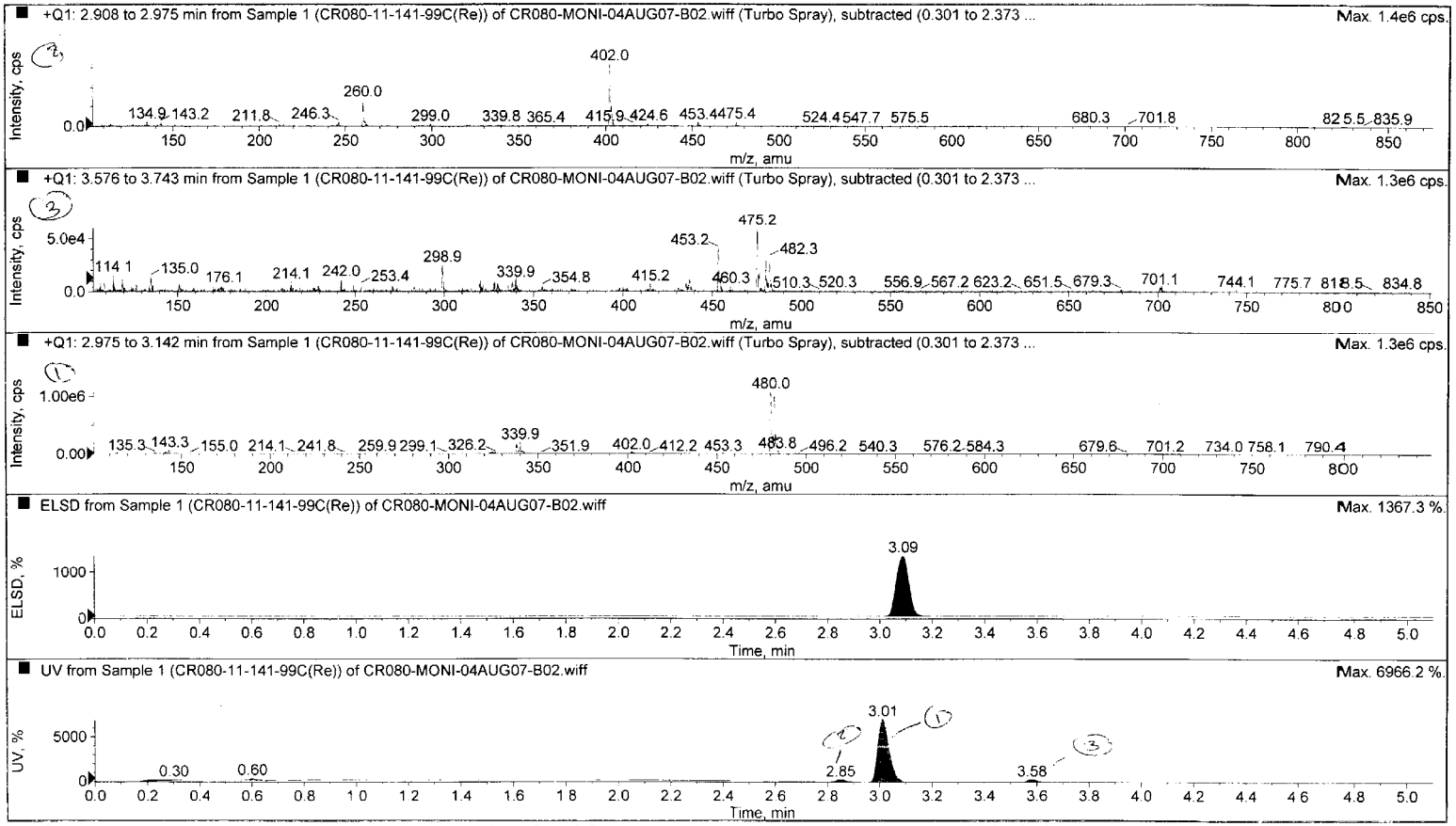


30

(Comp-31)

* **CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA ***

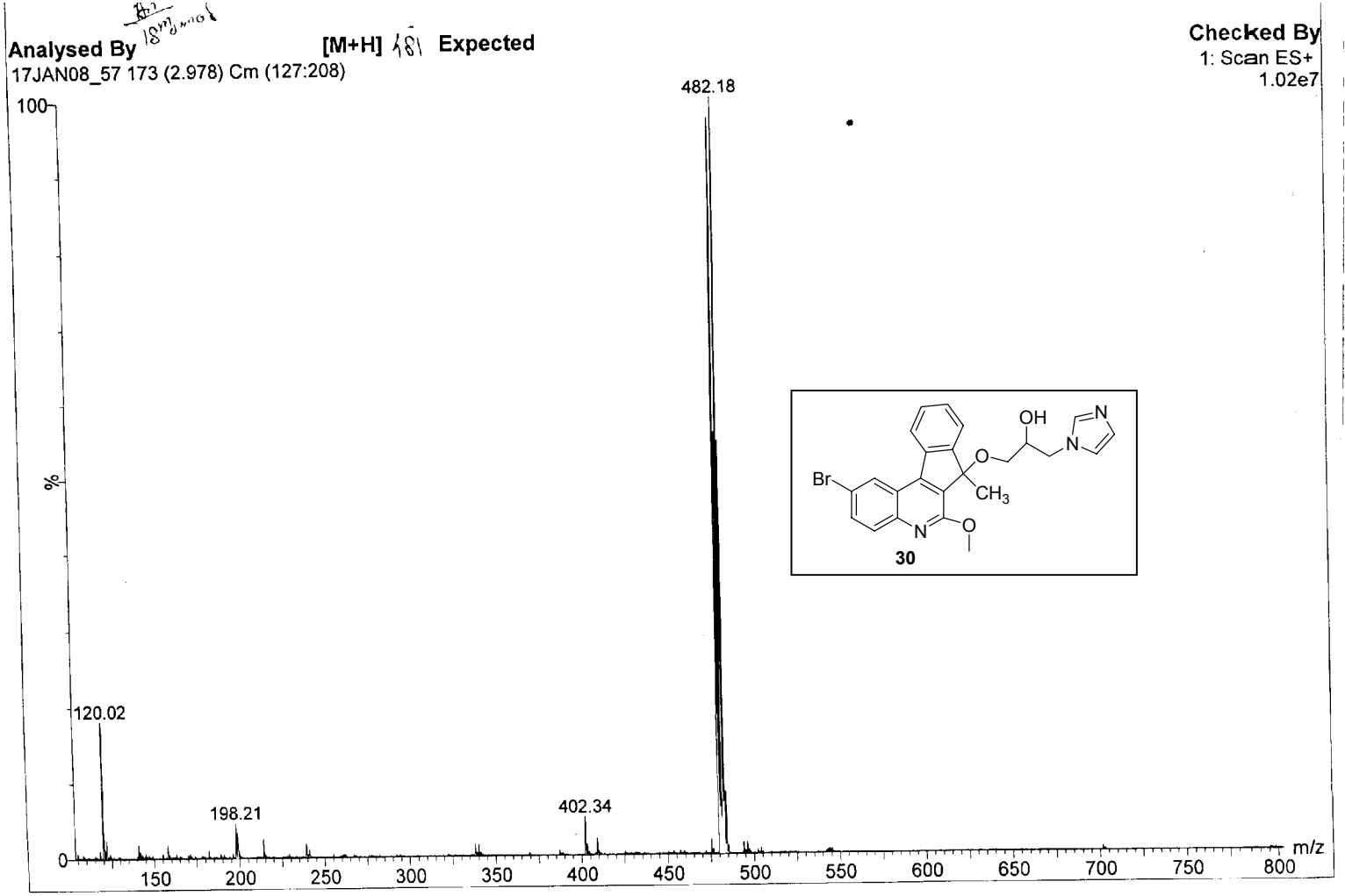
Sample Name: CR080-11-141-99C(Re) (IMM-60) Acq. Time: 18:54 Acq. Date: Saturday, August 04, 2007



Sample Comment: Moisture

**Analyzed By : Ar Ethoxyl (i)

**Checked By :


Analysed By
[M+H] 481 Expected
Checked By
17JAN08_57 173 (2.978) Cm (127:208)
1: Scan ES+
1.02e7
482.18
100
%
120.02
198.21
402.34
0
150
200
250
300
350
400
450
500
550
600
650
700
750
800
m/z
Br
OH
N
N
O
CH3
N
O
30

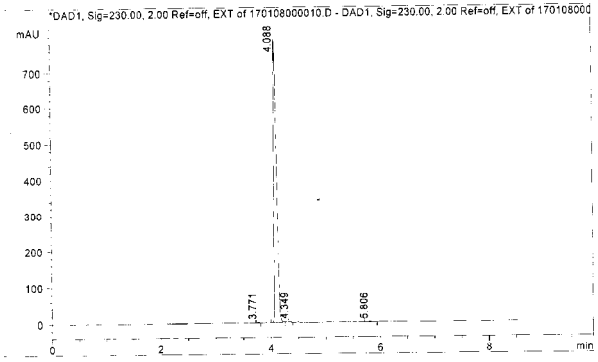
CHEMBIOTEK RESEARCH INTERNATIONAL, PUNE

comp 31

Sample:CR080-23-125-125A
Column :Zorbax SB - C 18 (50x4.6mm) ,1.8u ->

Injection Date	:Thu, 17. Jan. 2008	Location :	Vial 13
Sample Name	:CR080-23-125-125A	Inj. No. :	1
Acq Operator	:PRAKASH	Inj. Vol. :	2 µl
Analysis Method	:C:\CHEM32\2\METHODS\UPLC_GENARAL_GRAD _1.M		
Last Changed	:Thu, 17. Jan. 2008, 02:09:00 pm		

Acq. Method :C:\Chem32\2\DATA\JAN-08\170108B 2008-01-17 12-16-43\UPLC_GENARAL_GRAD _1.M
Method Ref : NP/757/32



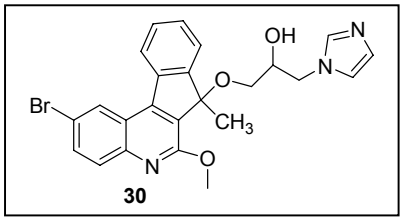
Signal: 1 DAD1, Sig=230.00, 2.00 Ref=off, EXT

Peak #	RT [min]	Width [min]	Area	Area %
1	3.77	0.03	4.21	0.15
2	4.09	0.06	2746.15	99.55
3	4.35	0.03	4.14	0.15
4	5.81	0.08	3.94	0.14

Product Impurity

*** End of Report ***

HPLC Thu, 17. Jan. 2008 03:17:50 pm Page 1 of 1



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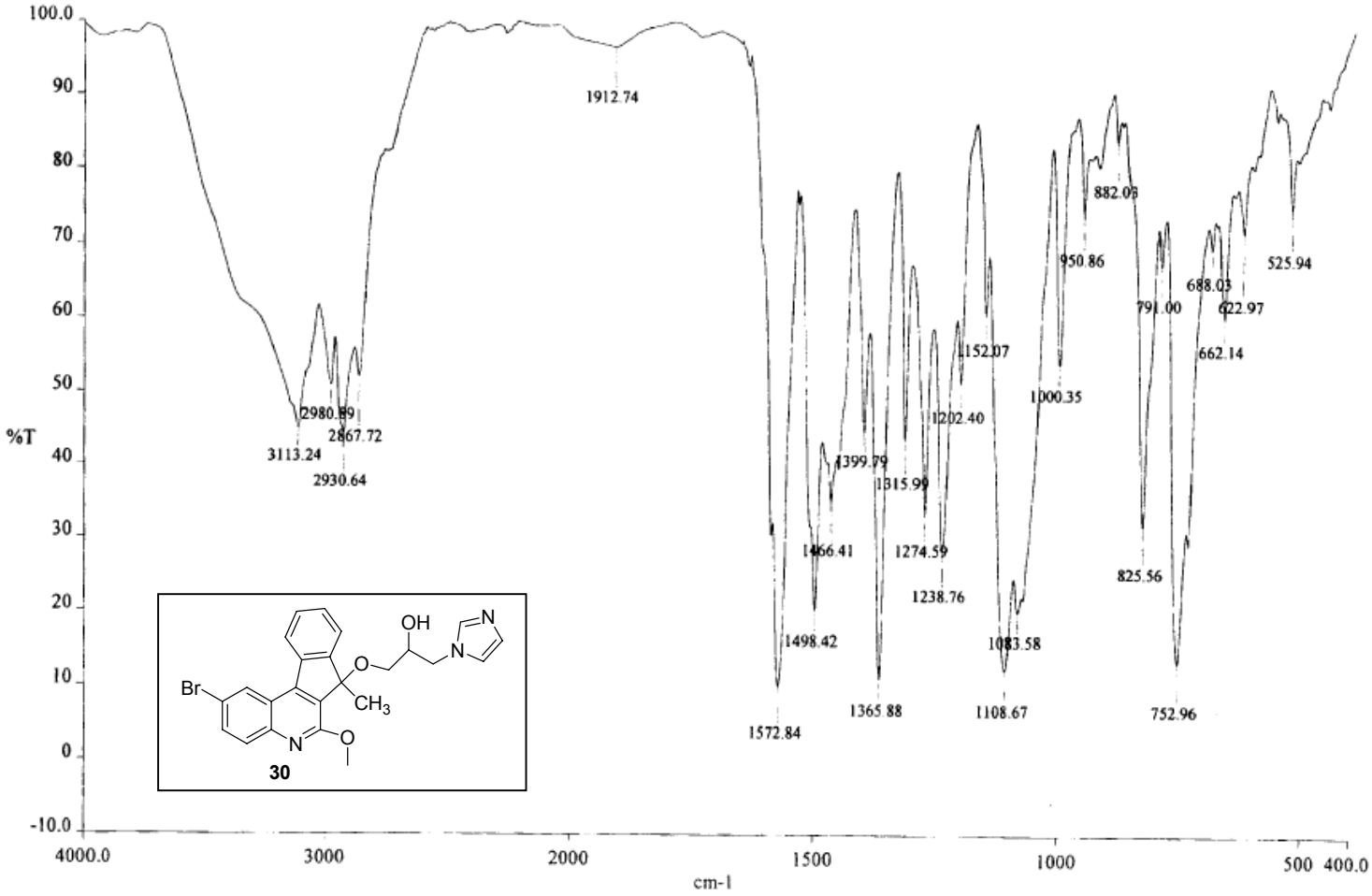
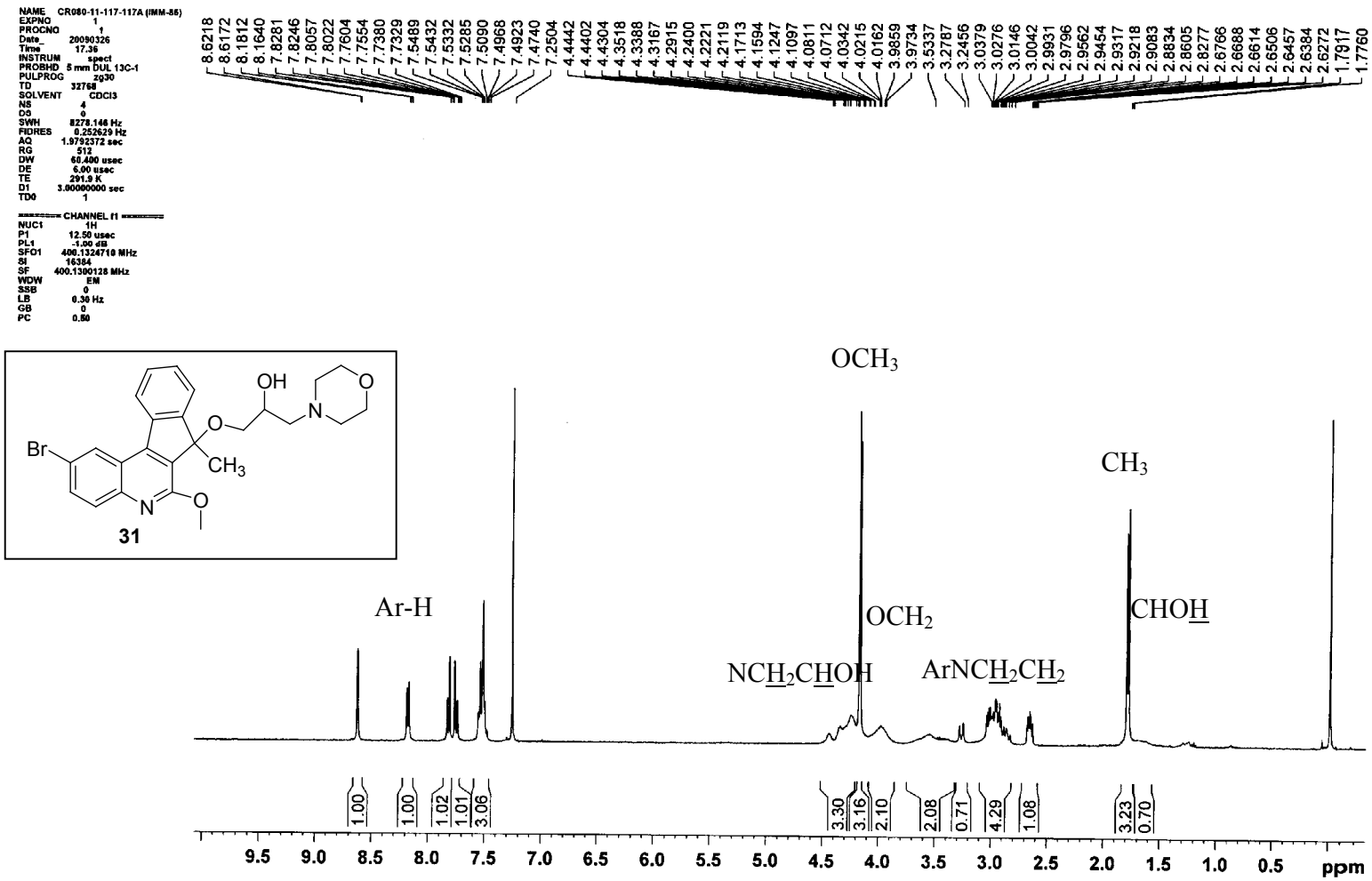
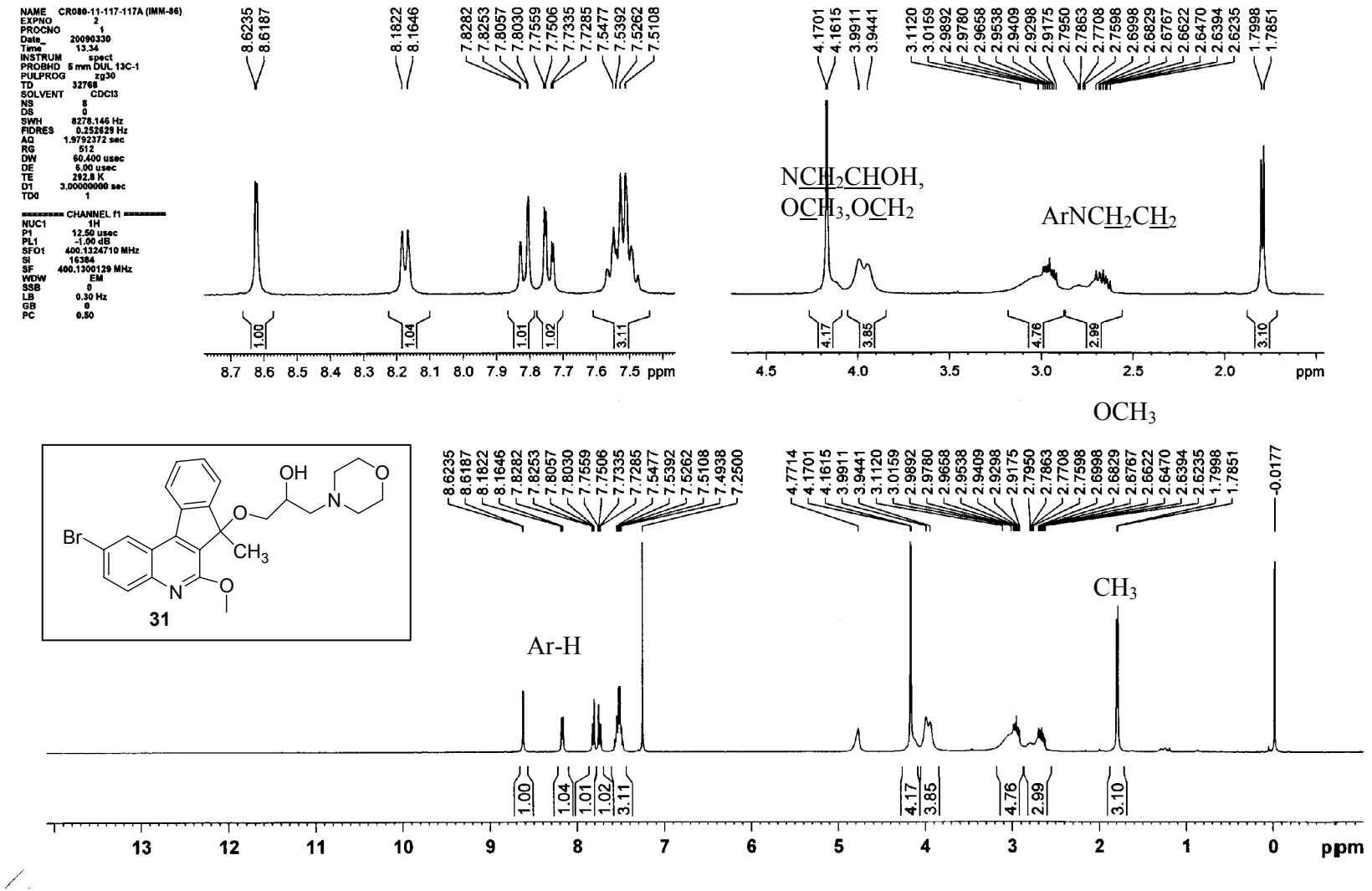
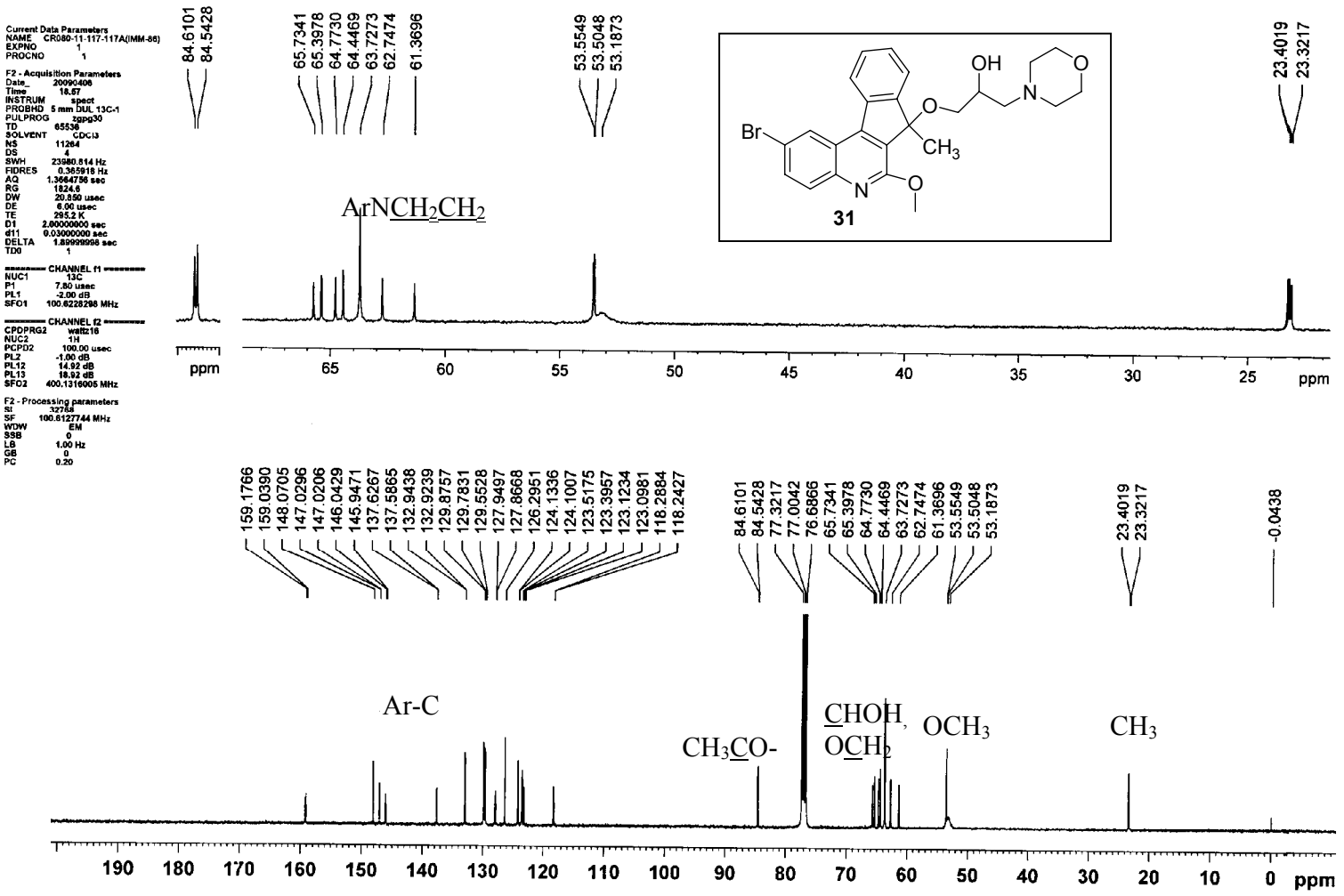
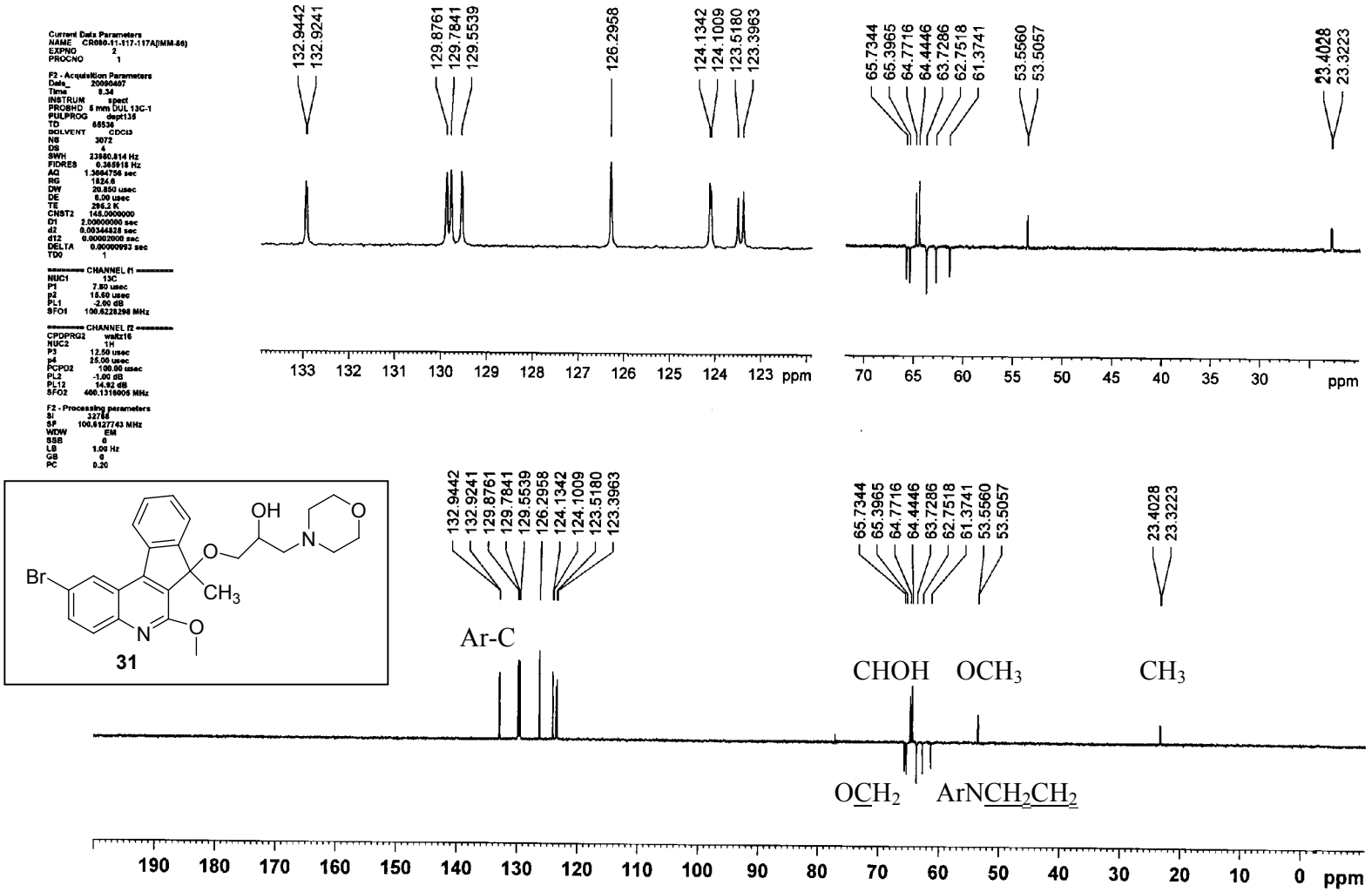
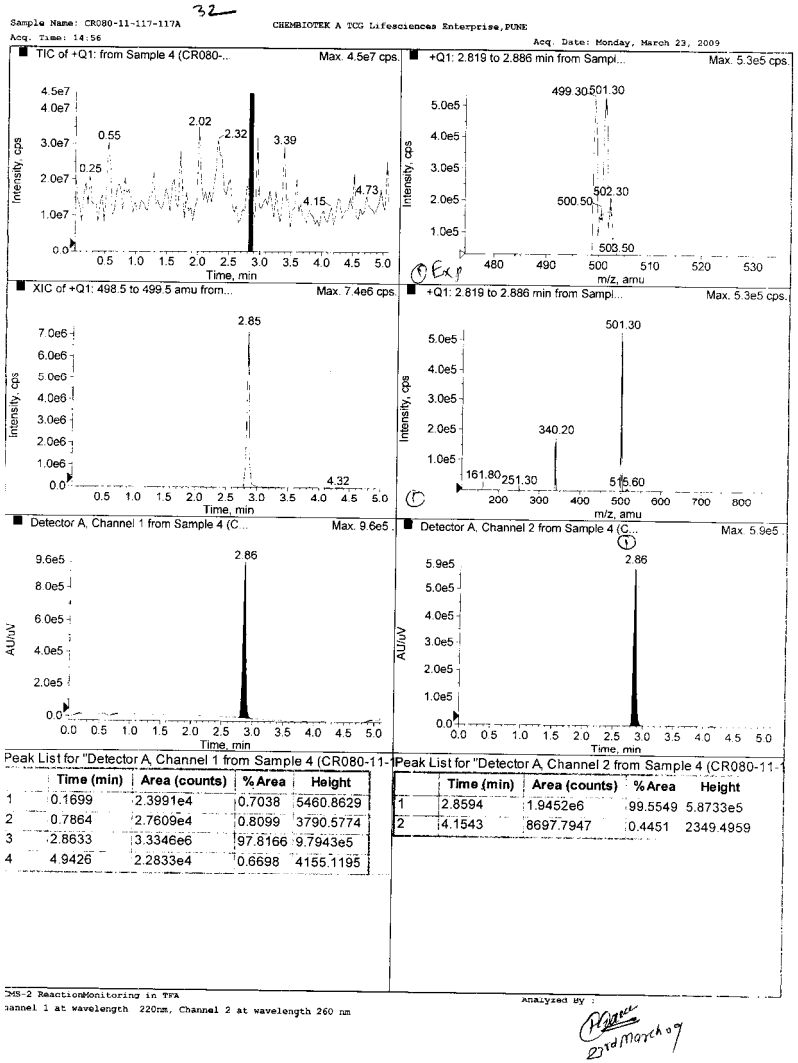

100.0
90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3113.24
2980.89
2930.64
2867.72
1912.74
1572.84
1498.42
1466.41
1399.79
1365.88
1315.99
1274.59
1238.76
1202.40
1152.07
1108.67
1083.58
1000.35
950.86
882.03
825.56
791.00
752.96
688.03
662.14
622.97
525.94
Br
OH
N
O
CH3
30

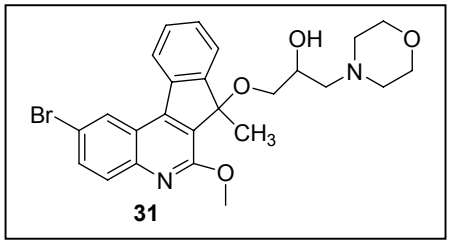

NAME CR080-11-117-117A (IMM-86)
EXPNO 1
PROCNO 1
Date_ 20090326
Time 17.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 512
DW 60.400 usec
DE 6.00 usec
TE 291.9 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80
8.6218 8.6172 8.1812 8.1640 7.8281 7.8246 7.8057 7.8022 7.7604 7.7554 7.7380 7.7329 7.5489 7.5432 7.5332 7.5285 7.5090 7.4968 7.4923 7.4740 7.2504 4.4442 4.4402 4.4304 4.3518 4.3388 4.3167 4.2915 4.2400 4.2221 4.2119 4.1713 4.1594 4.1247 4.1097 4.0811 4.0712 4.0342 4.0215 4.0162 3.9859 3.9734 3.5337 3.2787 3.2456 3.0379 3.0276 3.0146 3.0042 2.9931 2.9796 2.9562 2.9454 2.9317 2.9218 2.9083 2.8834 2.8605 2.8277 2.6766 2.6688 2.6614 2.6506 2.6457 2.6384 2.6272 1.7917 1.7760
Br
OH
O
N
O
CH3
N
O
31
OCH3
CH3
Ar-H
OCH2
CHOH
NCH2CHOH
ArNCH2CH2
1.00 1.00 1.02 1.01 3.06 3.30 3.16 2.10 2.08 0.71 4.29 1.08 3.23 0.70
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

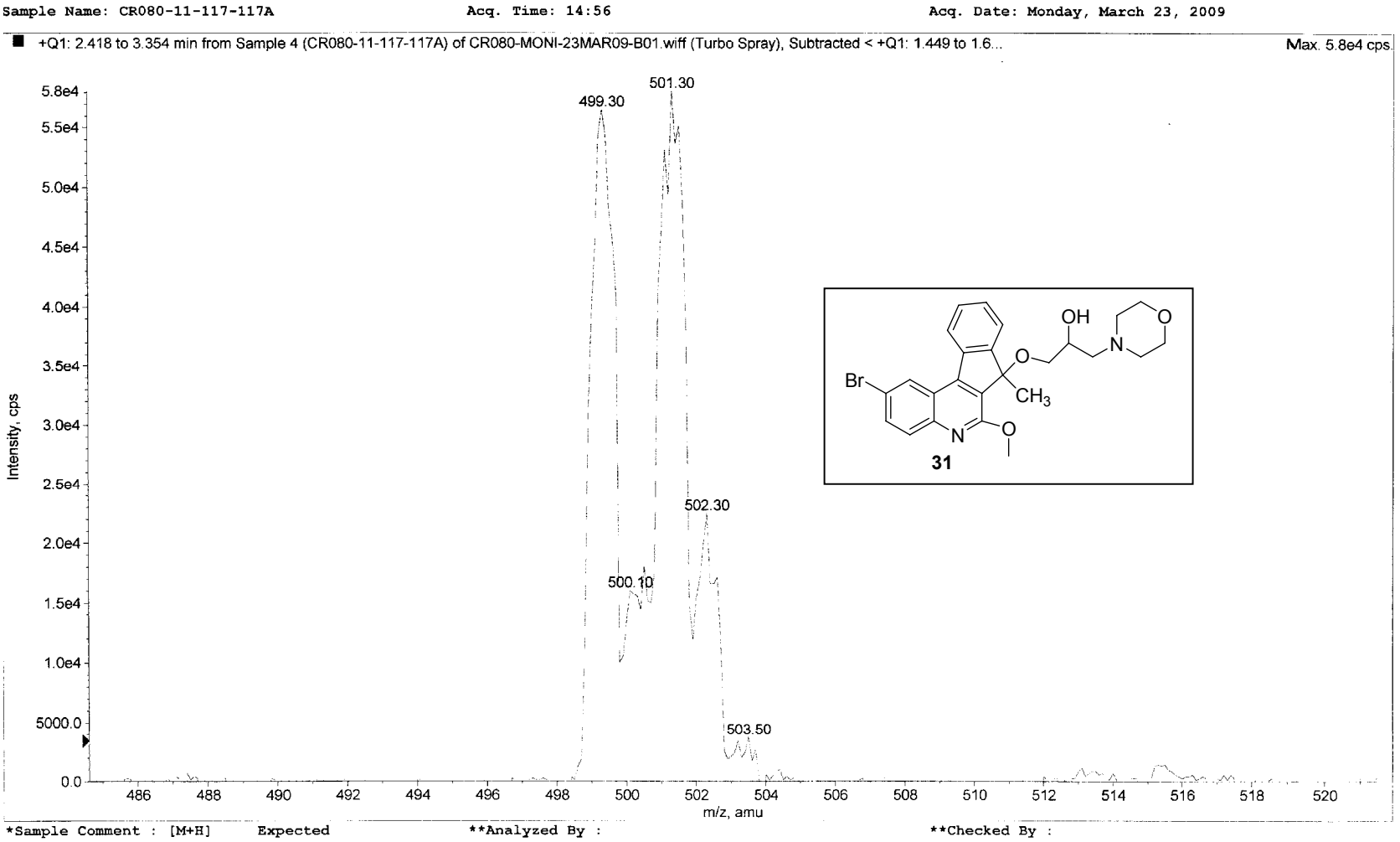

NAME CR080-11-117-117A (IMM-86)
EXPNO 2
PROCNO 1
Date_ 20090330
Time 13.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 512
DW 60.400 usec
DE 6.00 usec
TE 292.8 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50
NCH2CHOH,
OCH3,OCH2
ArNCH2CH2
OCH3
Br
OH
O
N
CH3
31
Ar-H
CH3
ppm


Current Data Parameters
NAME CR080-11-117-117A(IMM-86)
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090406
Time 16.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11264
DS 4
SWH 23980.814 Hz
FIDRES 0.365919 Hz
AQ 1.3664756 sec
RG 1824.6
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
84.6101
84.5428
65.7341
65.3978
64.7730
64.4469
63.7273
62.7474
61.3696
53.5549
53.5048
53.1873
23.4019
23.3217
ArNCH2CH2
Br
OH
O
N
CH3
31
ppm
159.1766
159.0390
148.0705
147.0296
147.0206
146.0429
145.9471
137.6267
137.5865
132.9438
132.9239
129.8757
129.7831
129.5528
127.9497
127.8668
126.2951
124.1336
124.1007
123.5175
123.3957
123.1234
123.0981
118.2884
118.2427
77.3217
77.0042
76.6866
-0.0438
Ar-C
CH3CO-
CHOH,
OCH2
OCH3
CH3


Current Data Parameters
NAME CR080-11-117-117A(IMM-86)
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090407
Time 8.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1824.6
DW 20.850 usec
DE 6.00 usec
TE 294.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
132.9442
132.9241
129.8761
129.7841
129.5539
126.2958
124.1342
124.1009
123.5180
123.3963
65.7344
65.3965
64.7716
64.4446
63.7286
62.7518
61.3741
53.5560
53.5057
23.4028
23.3223
133 132 131 130 129 128 127 126 125 124 123 ppm
70 65 60 55 50 45 40 35 30 ppm
Br
OH
O
N
O
CH3
N
O
31
Ar-C
CHOH
OCH3
CH3
OCH2
ArNCH2CH2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

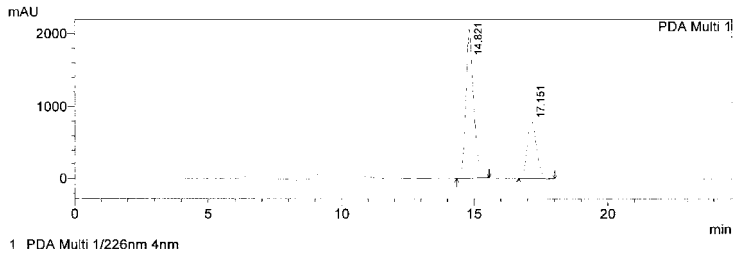

32
Sample Name: CR080-11-117-117A
CHEMBIOTEK A TCG Lifesciences Enterprise,PUNE
Acq. Time: 14:56
Acq. Date: Monday, March 23, 2009
TIC of +Q1: from Sample 4 (CR080-...
Max. 4.5e7 cps.
+Q1: 2.819 to 2.886 min from Sampl...
Max. 5.3e5 cps.
XIC of +Q1: 498.5 to 499.5 amu from...
Max. 7.4e6 cps.
Detector A, Channel 1 from Sample 4 (C...
Max. 9.6e5
Detector A, Channel 2 from Sample 4 (C...
Max. 5.9e5
Intensity, cps
AU/uV
Time, min
m/z, amu
Peak List for "Detector A, Channel 1 from Sample 4 (CR080-11-1
Time (min) | Area (counts) | % Area | Height
1 0.1699 | 2.3991e4 | 0.7038 | 5460.8629
2 0.7864 | 2.7609e4 | 0.8099 | 3790.5774
3 2.8633 | 3.3346e6 | 97.8166 | 9.7943e5
4 4.9426 | 2.2833e4 | 0.6698 | 4155.1195
Peak List for "Detector A, Channel 2 from Sample 4 (CR080-11-1
Time (min) | Area (counts) | % Area | Height
1 2.8594 | 1.9452e6 | 99.5549 | 5.8733e5
2 4.1543 | 8697.7947 | 0.4451 | 2349.4959
MS-2 ReactionMonitoring in TFA
Channel 1 at wavelength 220nm, Channel 2 at wavelength 260 nm
Analyzed by :
23rd March 09


Br
OH
O
N
O
CH3
N
O
31

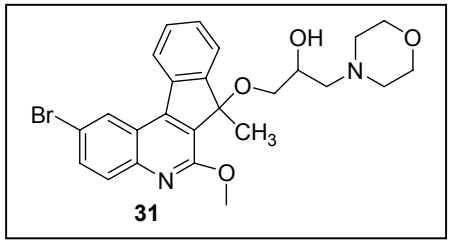

Sample Name: CR080-11-117-117A
Acq. Time: 14:56
Acq. Date: Monday, March 23, 2009
+Q1: 2.418 to 3.354 min from Sample 4 (CR080-11-117-117A) of CR080-MONI-23MAR09-B01.wiff (Turbo Spray), Subtracted < +Q1: 1.449 to 1.6...
Max. 5.8e4 cps.
Intensity, cps
m/z, amu
499.30
501.30
502.30
500.10
503.50
31
*Sample Comment : [M+H] Expected
**Analyzed By :
**Checked By :

Sample Name : CR080-11-117-117A
Sample ID : CR080-11-117-117A
Column : CHIRALPAK AD-H (250 x 4.6 mm)
Vial # : 11
Inj. Volume : 10 uL
Tray # : 1
Acquired by : PANKAJ

Data File Name : 04_10_08_CR080-11-117-117A_05.lcd
Method File Name : CR156_01.lcm
Batch File Name :
Data Acquired : 10/4/2008 12:35:19 PM
Data Processed : 10/4/2008 1:00:02 PM



1 PDA Multi 1/226nm 4nm



PeakTable

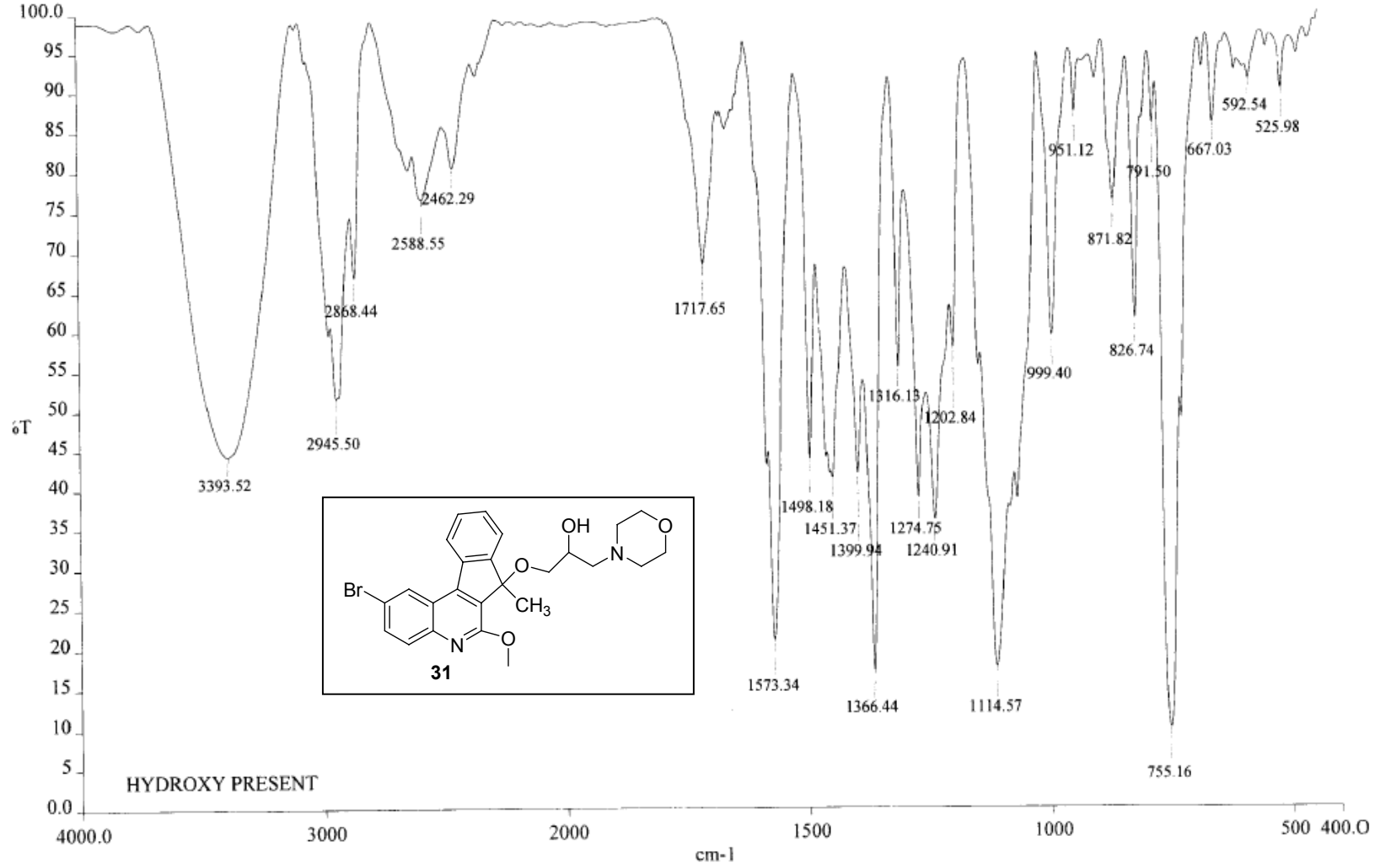
PDA Ch1 226nm 4nm

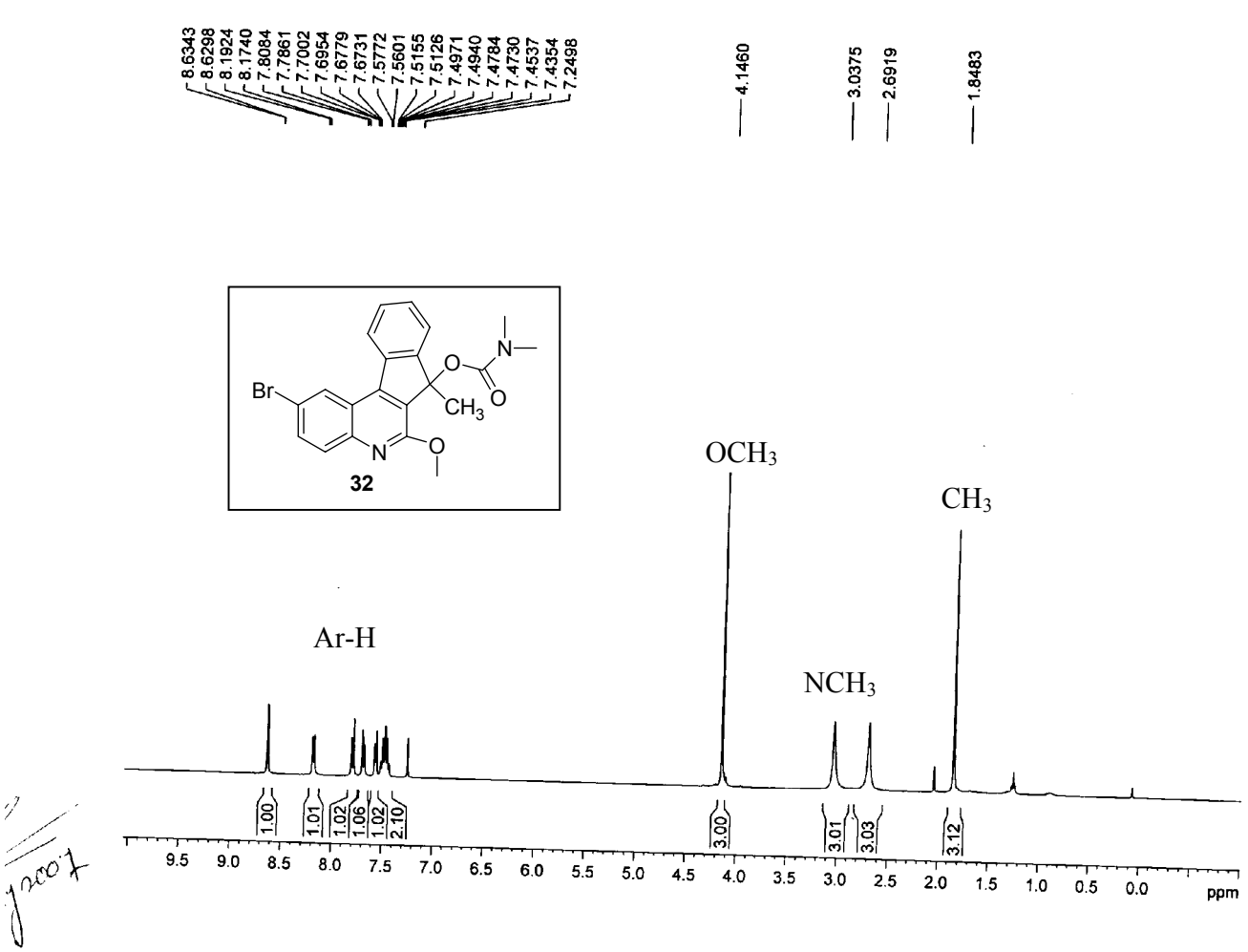
Peak#	Ret. Time	Area	Area %	Height
1	14.821	41759966	68.158	2069998
2	17.151	19509390	31.842	798966
Total		61269357	100.000	2868964

10th oct '08

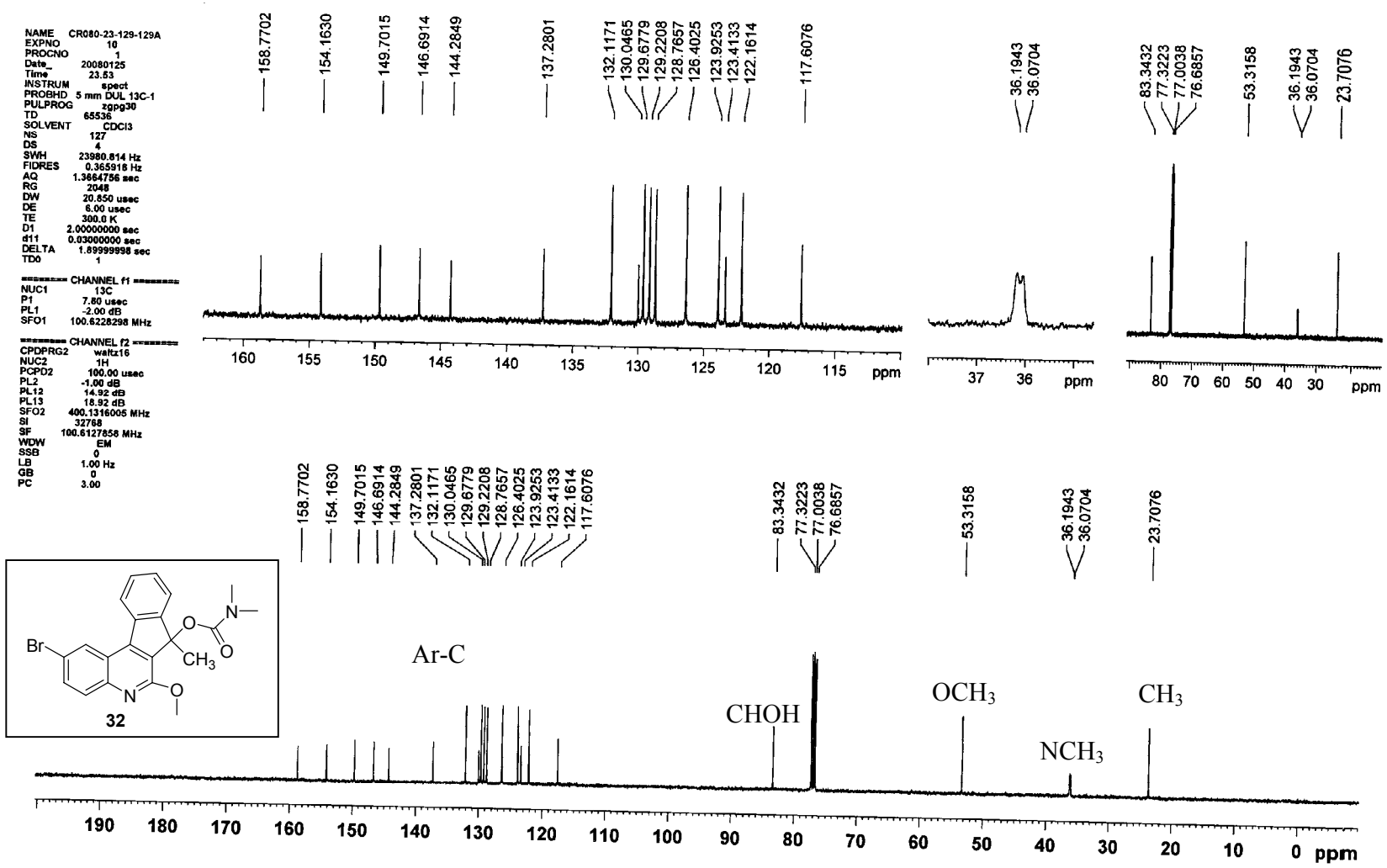
Normal phase.

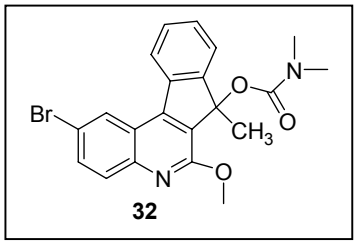
C:\LabSolutions\Data\Project1\HPLC-01\OCT-08\04_10_08_CR080-11-117-117A_05.lcd


100.0
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0.0
%T
3393.52
2945.50
2868.44
2588.55
2462.29
1717.65
1573.34
1498.18
1451.37
1399.94
1366.44
1316.13
1274.75
1240.91
1202.84
1114.57
999.40
951.12
871.82
826.74
791.50
755.16
667.03
592.54
525.98
HYDROXY PRESENT
4000.0
3000
2000
1500
1000
500
400.0
cm-1
Br
O
CH3
N
O
OH
N
O
31

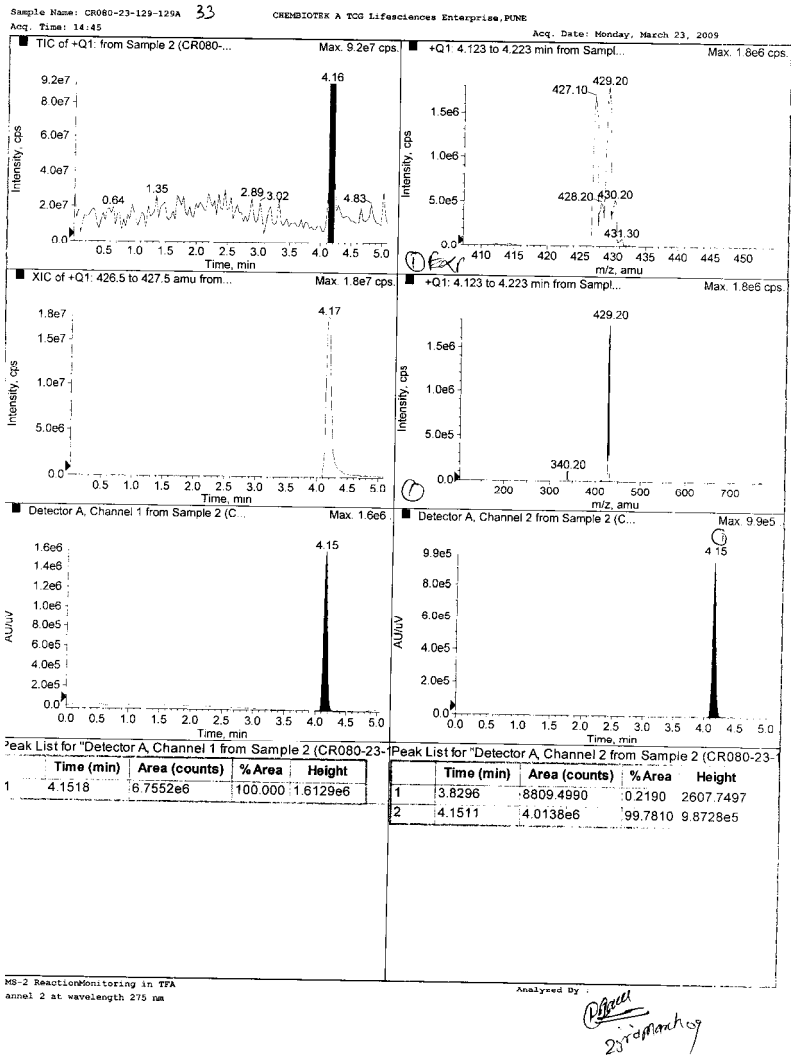


NAME	CR080-11-131-131A
EXPNO	1
PROCNO	1
Date_	20070723
Time	15.32
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	456.1
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.50 usec
PL1	-1.00 dB
SFO1	400.1324710 MHz
SI	32768
SF	400.1300140 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


NAME CR080-23-129-129A
EXPNO 10
PROCNO 1
Date_ 20080125
Time 23.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 127
DS 4
SWH 23980.814 Hz
FIDRES 0.365916 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127858 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00
158.7702
154.1630
149.7015
146.6914
144.2849
137.2801
132.1171
130.0465
129.6779
129.2208
128.7657
126.4025
123.9253
123.4133
122.1614
117.6076
36.1943
36.0704
83.3432
77.3223
77.0038
76.6857
53.3158
23.7076
ppm
Br
CH3
O
N
32
Ar-C
CHOH
OCH3
NCH3
CH3


Br
O
N
CH3
O
N
O

32

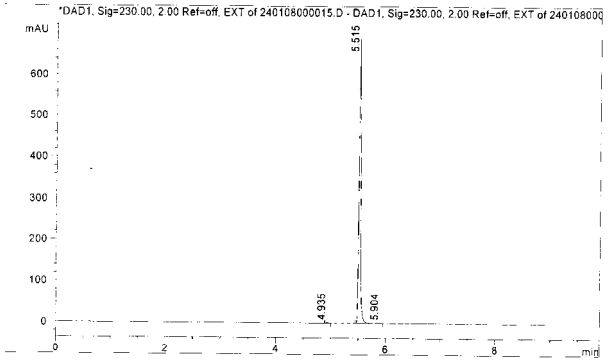

Sample Name: CR080-23-129-129A 33
CHEMBIOTEK A TCG Lifesciences Enterprise,PUNE
Acq. Time: 14:45
Acq. Date: Monday, March 23, 2009
TIC of +Q1: from Sample 2 (CR080-...
Max. 9.2e7 cps.
Intensity, cps
Time, min
+Q1: 4.123 to 4.223 min from Sampl...
Max. 1.8e6 cps.
m/z, amu
XIC of +Q1: 426.5 to 427.5 amu from...
Max. 1.8e7 cps.
+Q1: 4.123 to 4.223 min from Sampl...
Max. 1.8e6 cps.
Detector A, Channel 1 from Sample 2 (C...
Max. 1.6e6
AU/uV
Detector A, Channel 2 from Sample 2 (C...
Max. 9.9e5
Peak List for "Detector A, Channel 1 from Sample 2 (CR080-23-
Time (min) | Area (counts) | %Area | Height
1 | 4.1518 | 6.7552e6 | 100.000 | 1.6129e6
Peak List for "Detector A, Channel 2 from Sample 2 (CR080-23-
Time (min) | Area (counts) | %Area | Height
1 | 3.8296 | 8809.4990 | 0.2190 | 2607.7497
2 | 4.1511 | 4.0138e6 | 99.7810 | 9.8728e5
MS-2 ReactionMonitoring in TFA
annel 2 at wavelength 275 nm
Analyzed By :
23rd March 09

33 comp 33

CHEMBIOTEK RESEARCH INTERNATIONAL, PUNE

Sample:CR080-23-129-129A
Column :Zorbax SB - C 18 (50x4.6mm) ,1.8u ->

Injection Date	:Thu, 24. Jan. 2008	Location :	Vial 11
Sample Name	:CR080-23-129-129A	Inj. No. :	1
Acq Operator	:PRAKASH	Inj. Vol. :	2 µl
Analysis Method	:C:\CHEM32\2\METHODS\UPLC_GENARAL_GRAD _1.M		
Last Changed	:Thu, 24. Jan. 2008, 04:15:08 pm (modified after loading)		
Acq. Method	:C:\Chem32\2\DATA\JAN-08\240108A 2008-01-24 10-11-45\ UPLC GENARAL_GRAD _1.M		
Method Ref	: NP/757/34		



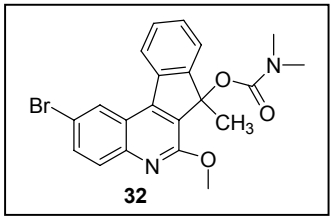
Signal: 1 DAD1, Sig=230.00, 2.00 Ref=off, EXT

Peak #	RT [min]	Width [min]	Area	Area %
1	4.94	0.03	3.33	0.22
2	5.52	0.04	1527.50	99.53
3	5.90	0.04	3.88	0.25

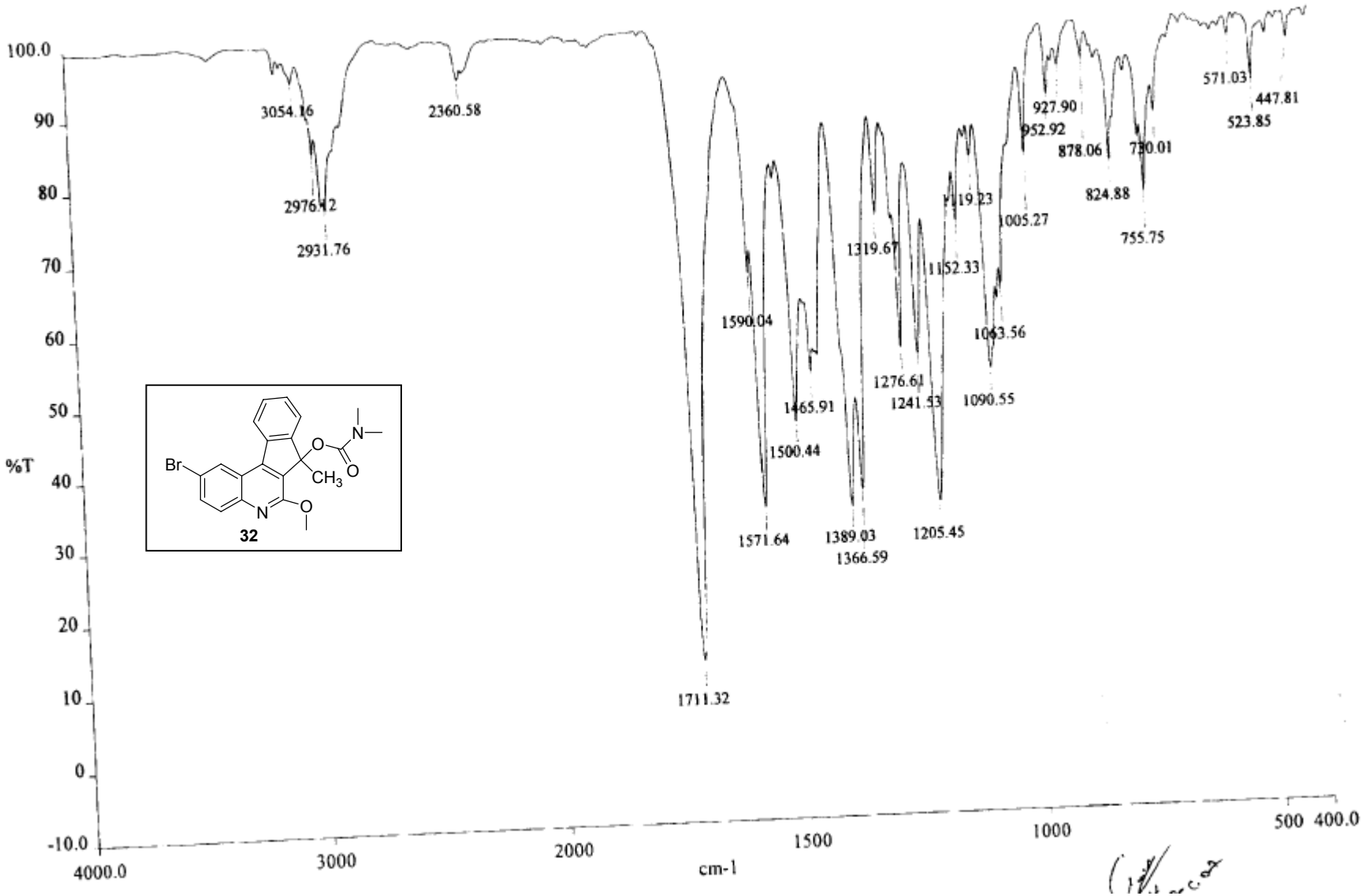
Prakash 12/02/2008

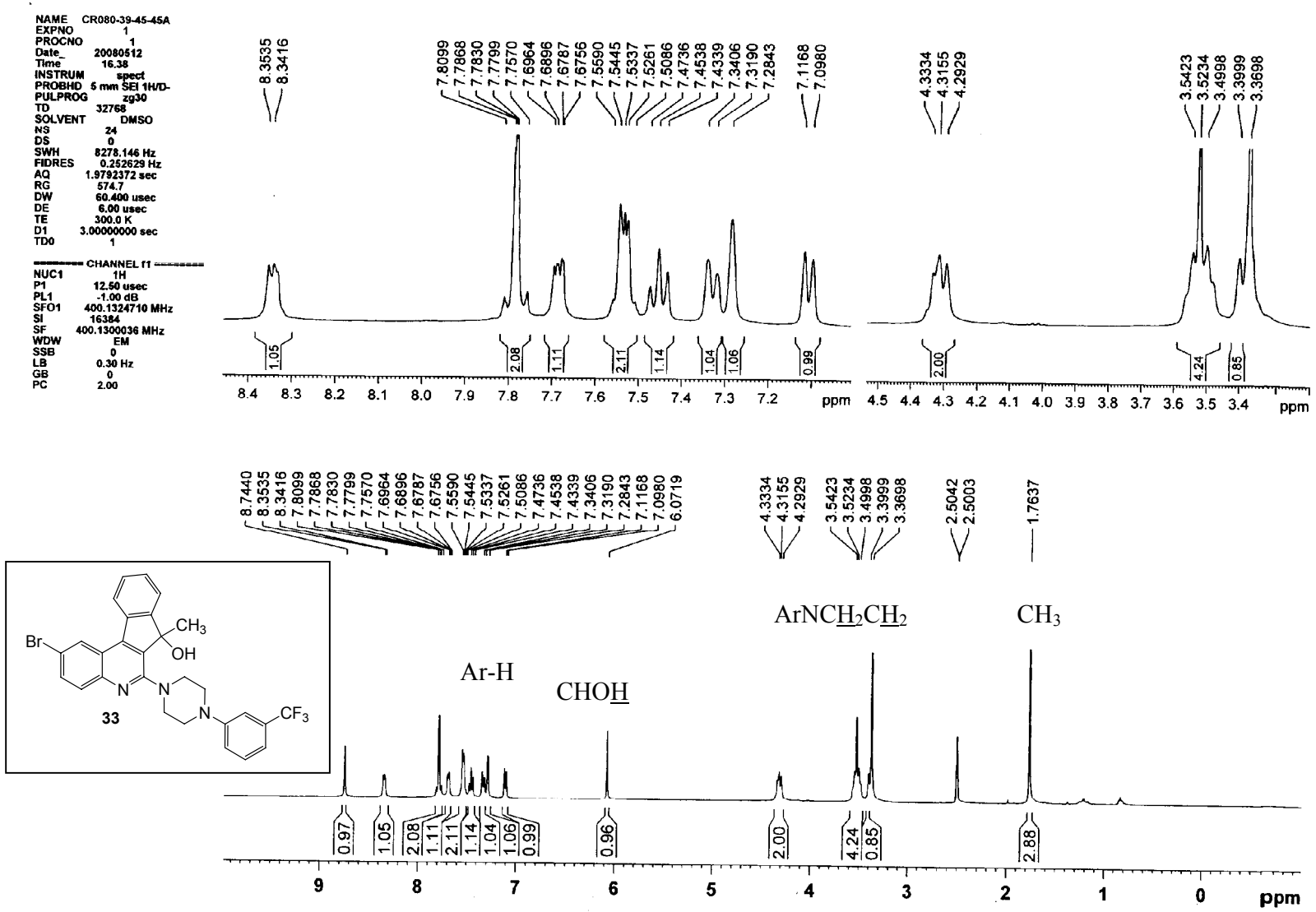
*** End of Report ***

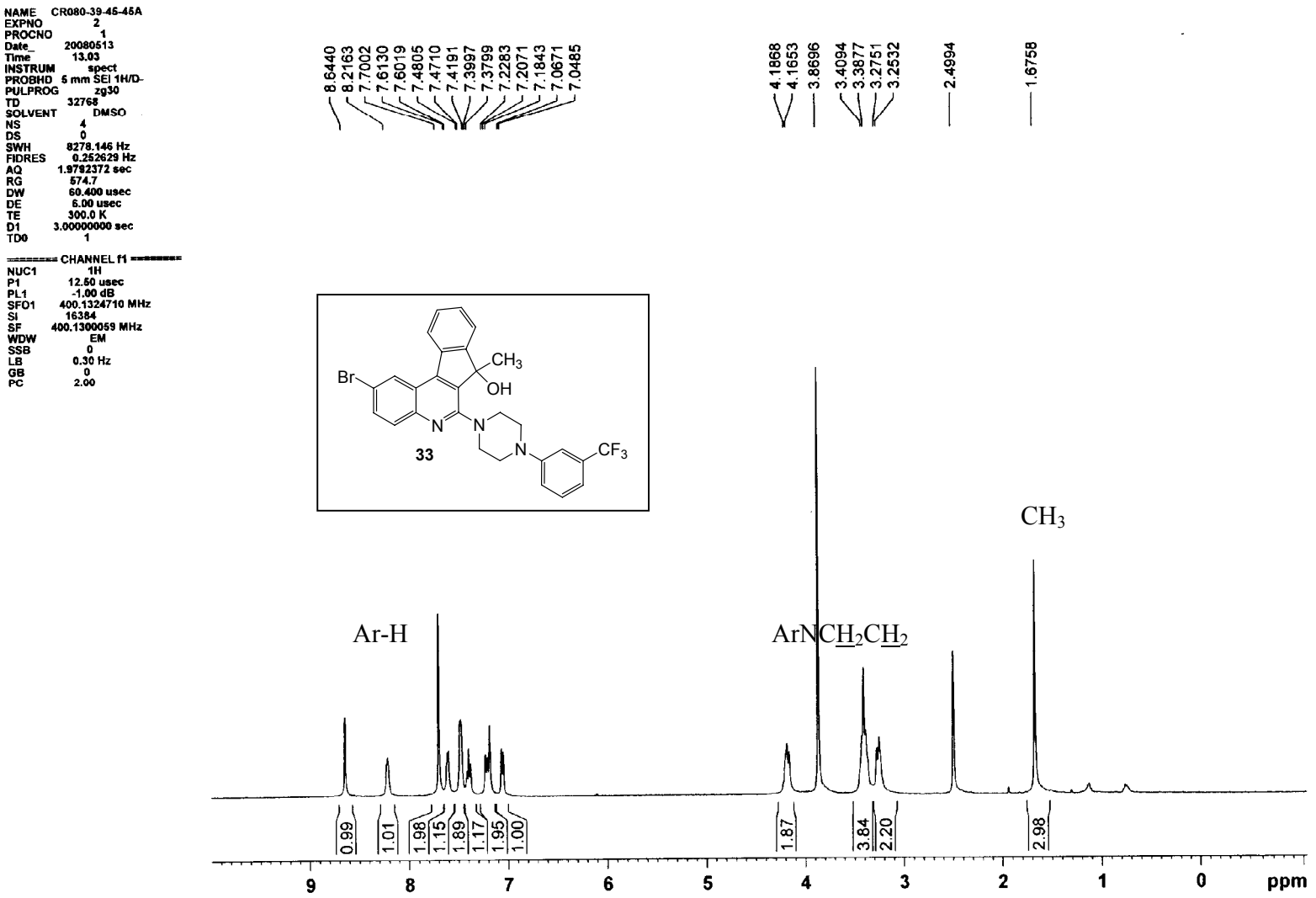
PLC Thu, 24. Jan. 2008 04:21:25 pm Page 1 of 1

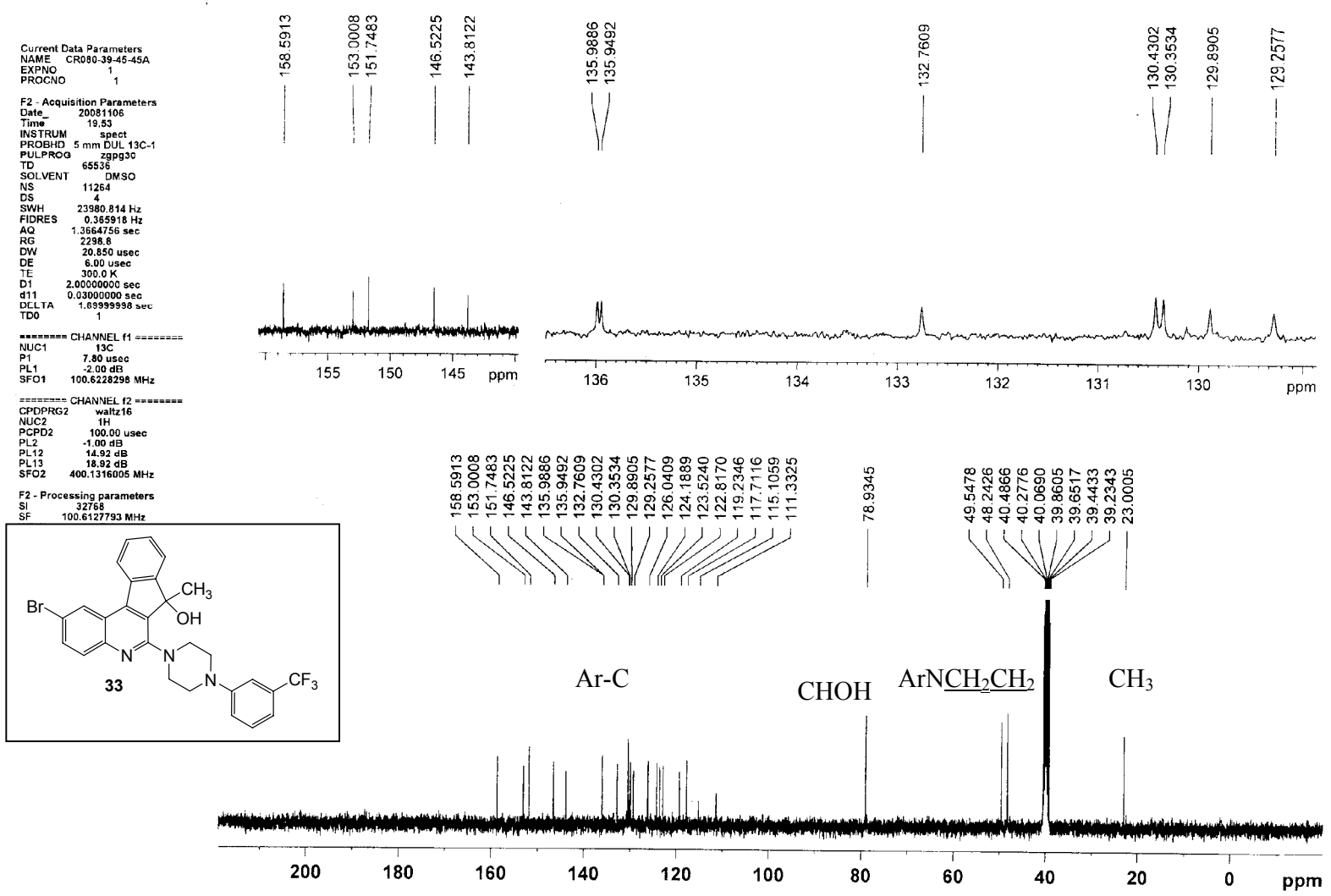


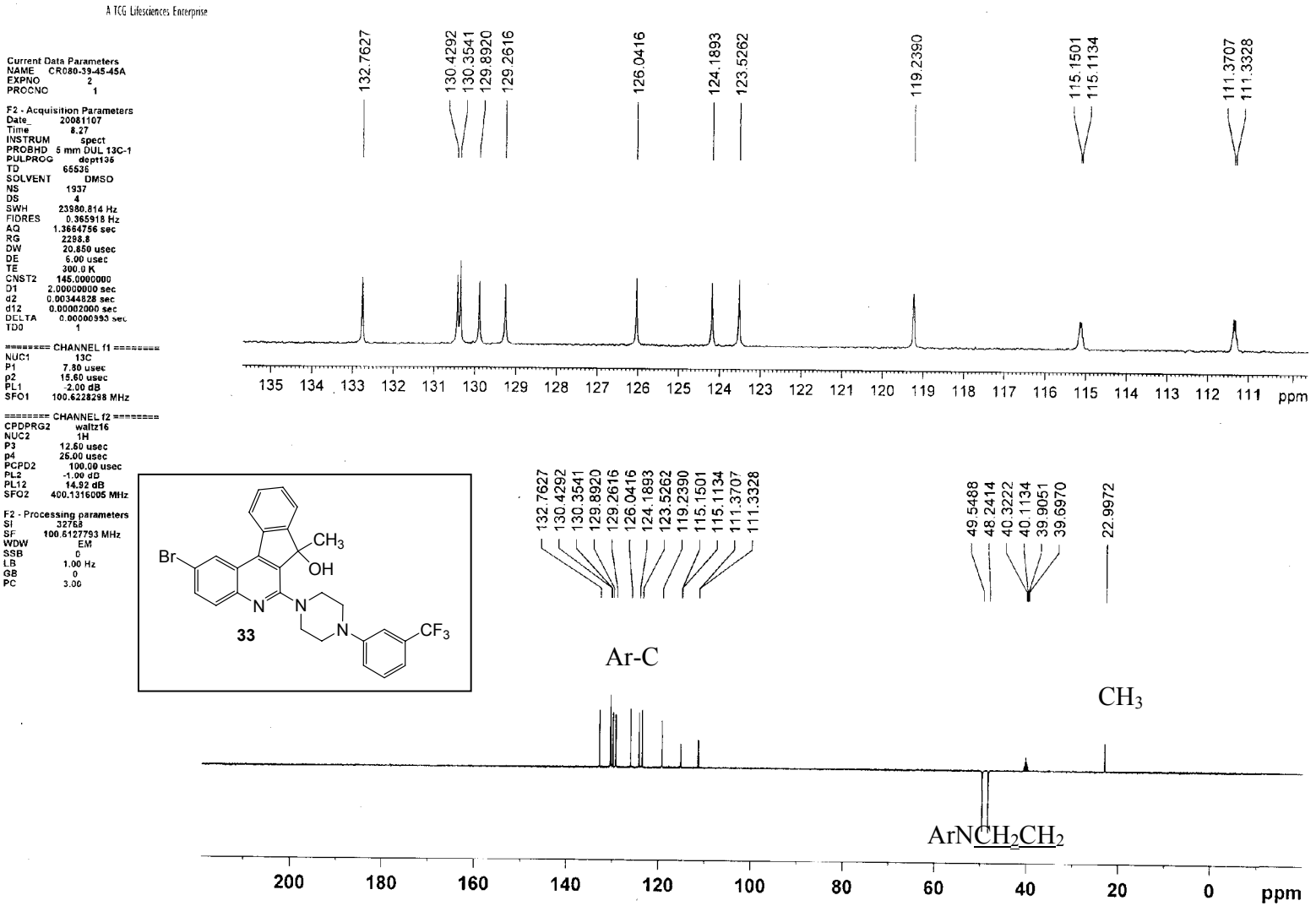
32

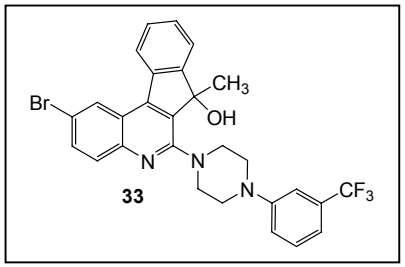

100.0
90
80
70
60
50
40
30
20
10
0
-10.0
%T
4000.0
3000
2000
1500
1000
500 400.0
cm-1
3054.16
2976.42
2931.76
2360.58
1711.32
1590.04
1571.64
1500.44
1465.91
1389.03
1366.59
1319.67
1276.61
1241.53
1205.45
1152.33
1119.23
1090.55
1063.56
1005.27
952.92
927.90
878.06
824.88
755.75
730.01
571.03
523.85
447.81
Br
O
N
CH3
O
O
N
32


NAME CR080-39-45-45A
EXPNO 1
PROCNO 1
Date_ 20080512
Time 16.38
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 24
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
8.3535
8.3416
7.8099
7.7868
7.7830
7.7799
7.7570
7.6964
7.6896
7.6787
7.6756
7.5590
7.5445
7.5337
7.5261
7.5086
7.4736
7.4538
7.4339
7.3406
7.3190
7.2843
7.1168
7.0980
4.3334
4.3155
4.2929
3.5423
3.5234
3.4998
3.3999
3.3698
1.05
2.08
1.11
2.11
1.14
1.04
1.06
0.99
2.00
4.24
0.85
ppm
8.7440
6.0719
2.5042
2.5003
1.7637
Br
CH3
OH
N
N
N
CF3
33
Ar-H
CHOH
ArNCH2CH2
CH3
0.97
0.96
2.88

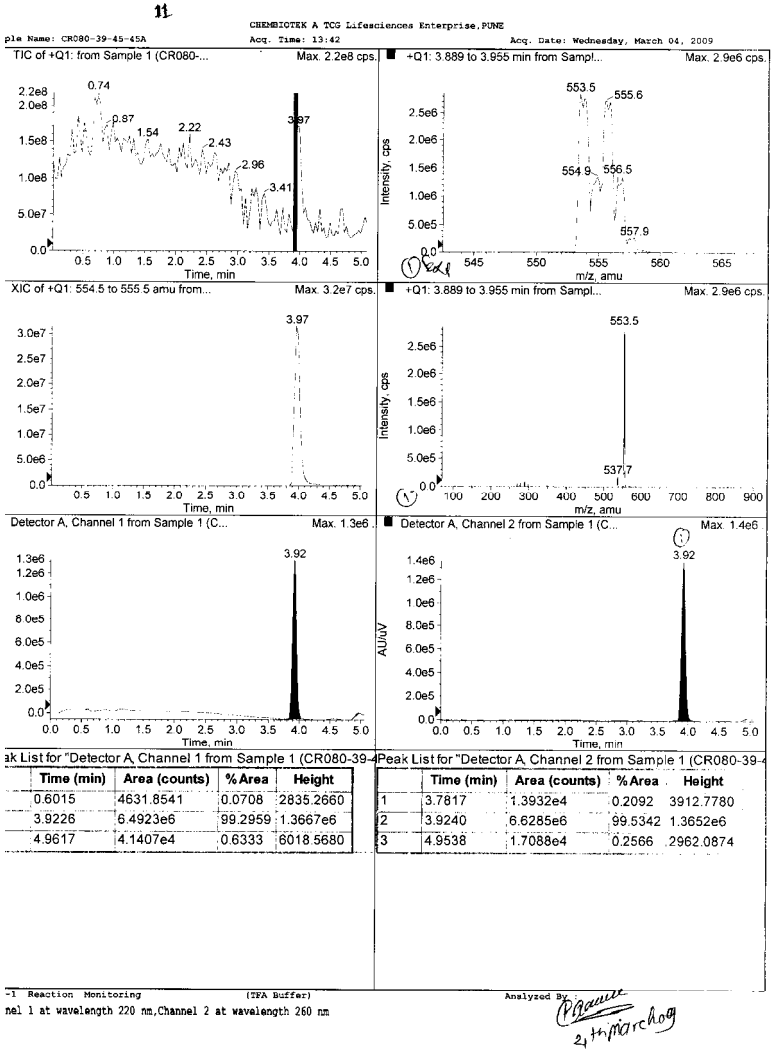

NAME CR080-39-45-45A
EXPNO 2
PROCNO 1
Date_ 20080513
Time 13.03
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 4
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9782372 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
8.6440
8.2163
7.7002
7.6130
7.6019
7.4805
7.4710
7.4191
7.3997
7.3799
7.2283
7.2071
7.1843
7.0671
7.0485
4.1868
4.1653
3.8696
3.4094
3.3877
3.2751
3.2532
2.4994
1.6758
Br
CH3
OH
N
N
N
CF3
33
Ar-H
ArNCH2CH2
CH3
0.99
1.01
1.98
1.15
1.89
1.17
1.95
1.00
1.87
3.84
2.20
2.98
9
8
7
6
5
4
3
2
1
0
ppm


Current Data Parameters
NAME CR080-39-45-45A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081106
Time 19.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 11264
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2298.8
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127793 MHz
158.5913
153.0008
151.7483
146.5225
143.8122
135.9886
135.9492
132.7609
130.4302
130.3534
129.8905
129.2577
155
150
145
ppm
136
135
134
133
132
131
130
ppm
158.5913
153.0008
151.7483
146.5225
143.8122
135.9886
135.9492
132.7609
130.4302
130.3534
129.8905
129.2577
126.0409
124.1889
123.5240
122.8170
119.2346
117.7116
115.1059
111.3325
78.9345
49.5478
48.2426
40.4866
40.2776
40.0690
39.8605
39.6517
39.4433
39.2343
23.0005
Br
CH3
OH
N
N
N
CF3
33
Ar-C
CHOH
ArNCH2CH2
CH3
200
180
160
140
120
100
80
60
40
20
0
ppm


A TCG Lifesciences Enterprise
Current Data Parameters
NAME CR080-39-45-45A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081107
Time 8.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 65536
SOLVENT DMSO
NS 1937
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2298.8
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00000993 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.50 usec
p4 25.00 usec
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127793 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00
132.7627
130.4292
130.3541
129.8920
129.2616
126.0416
124.1893
123.5262
119.2390
115.1501
115.1134
111.3707
111.3328
135 134 133 132 131 130 129 128 127 126 125 124 123 122 121 120 119 118 117 116 115 114 113 112 111 ppm
Br
CH3
OH
N
N
N
CF3
33
132.7627
130.4292
130.3541
129.8920
129.2616
126.0416
124.1893
123.5262
119.2390
115.1501
115.1134
111.3707
111.3328
49.5488
48.2414
40.3222
40.1134
39.9051
39.6970
22.9972
Ar-C
CH3
ArNCH2CH2
200 180 160 140 120 100 80 60 40 20 0 ppm


Br
CH3
OH
N
N
N
CF3

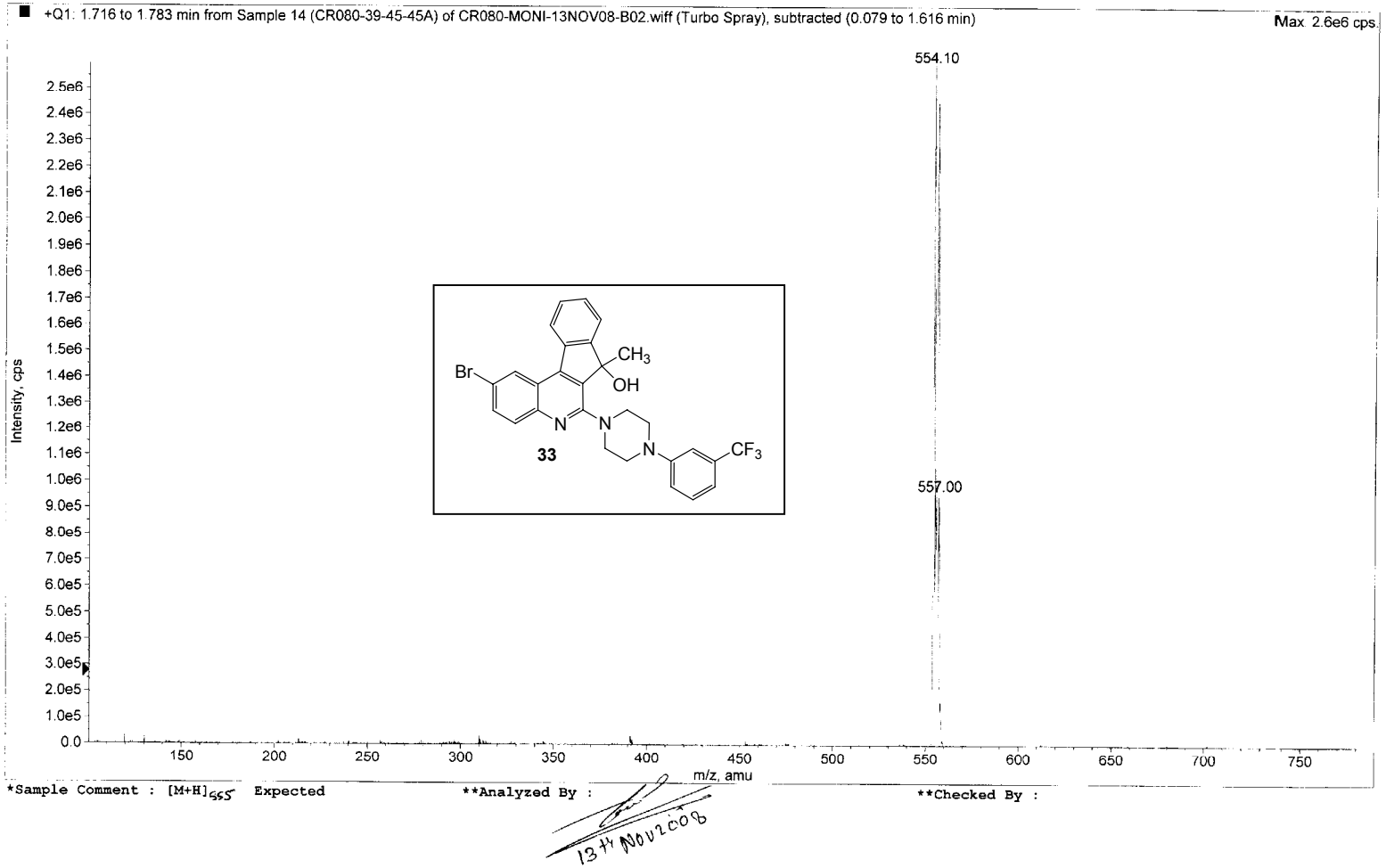
33


11
CHEMBIOTEK A TCG Lifesciences Enterprise,PUNE
Acq. Time: 13:42
Acq. Date: Wednesday, March 04, 2009
TIC of +Q1: from Sample 1 (CR080-...
Max. 2.2e8 cps.
+Q1: 3.889 to 3.955 min from Sampl...
Max. 2.9e6 cps.
Time, min
Intensity, cps
m/z, amu
XIC of +Q1: 554.5 to 555.5 amu from...
Max. 3.2e7 cps.
Detector A, Channel 1 from Sample 1 (C...
Max. 1.3e6
Detector A, Channel 2 from Sample 1 (C...
Max. 1.4e6
AU/uV
Peak List for "Detector A, Channel 1 from Sample 1 (CR080-39-4
Peak List for "Detector A, Channel 2 from Sample 1 (CR080-39-4
Time (min) | Area (counts) | %Area | Height
0.6015 | 4631.8541 | 0.0708 | 2835.2660
3.9226 | 6.4923e6 | 99.2959 | 1.3667e6
4.9617 | 4.1407e4 | 0.6333 | 6018.5680
3.7817 | 1.3932e4 | 0.2092 | 3912.7780
3.9240 | 6.6285e6 | 99.5342 | 1.3652e6
4.9538 | 1.7088e4 | 0.2566 | 2962.0874
Reaction Monitoring
(TFA Buffer)
Analyzed By
nel 1 at wavelength 220 nm,Channel 2 at wavelength 260 nm
24th March09

(35)

**CHEMBIOTEK RESEARCH INTERNATIONAL , PUNE, INDIA *

Sample Name: CR080-39-45-45A Acq. Time: 18:08 Acq. Date: Thursday, November 13, 2008



*Sample Comment : $[M+H]_{555}$ Expected **Analyzed By : **Checked By :

13th Nov 2008

Sample Name : CR080-39-45-45A CI mm 178
Sample ID : CR080-39-45-45A
Column : Zorbax SB C-18 (250 x 4.6 mm)
Vial # : 62
Inj. Volume : 4 uL
Tray # : 1
Acquired by : PRAKASH

Data File Name : 12_05_08_CR080-39-45-45A_02.lcd
Method File Name : GENERAL.lcm
Batch File Name : 12_05_08.lcb
Data Acquired : 5/13/2008 1:07:02 AM
Data Processed : 5/13/2008 10:52:50 AM
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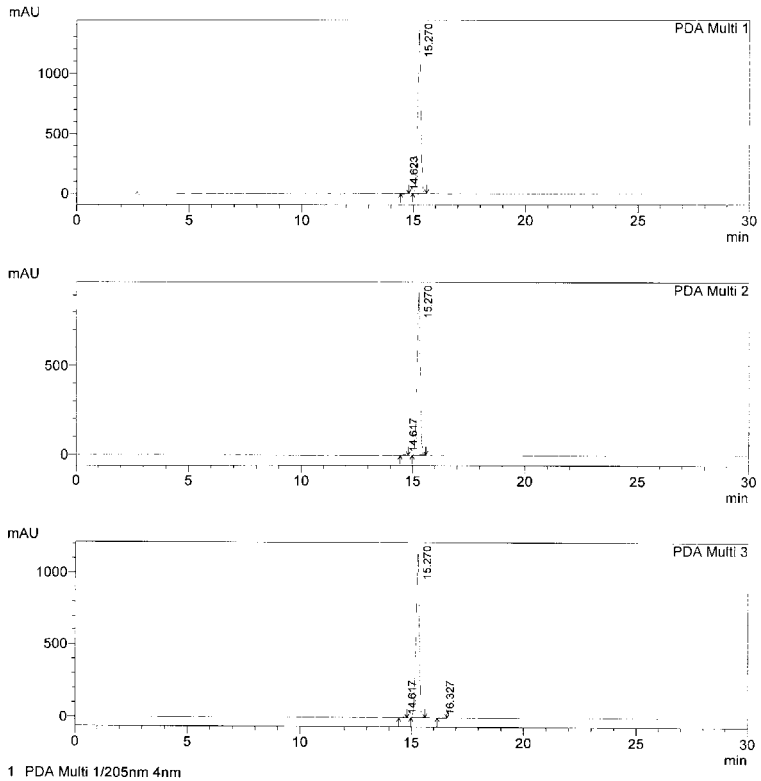


1 PDA Multi 1/205nm 4nm
2 PDA Multi 2/220nm 4nm
3 PDA Multi 3/260nm 4nm

PeakTable

C:\LabSolutions\Project\HPLC-01\MAY-08\12_05_08_CR080-39-45-45A_02.lcc

HPLC-01 PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Area %	Height
1	14.623	22387	0.170	2882
2	15.270	13134395	99.830	1361208
Total		13156782	100.000	1364090

PeakTable

HPLC-01 PDA Ch2 220nm 4nm

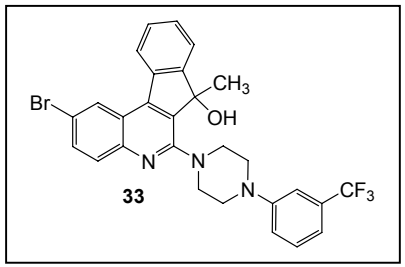
Peak#	Ret. Time	Area	Area %	Height
1	14.617	17830	0.194	2289
2	15.270	9151775	99.806	922278
Total		9169605	100.000	924567

PeakTable

HPLC-01 PDA Ch3 260nm 4nm

Peak#	Ret. Time	Area	Area %	Height
1	14.617	21224	0.186	2721
2	15.270	11373125	99.707	1146475
3	16.327	12188	0.107	1056
Total		11406537	100.000	1150252

Purity with impurity



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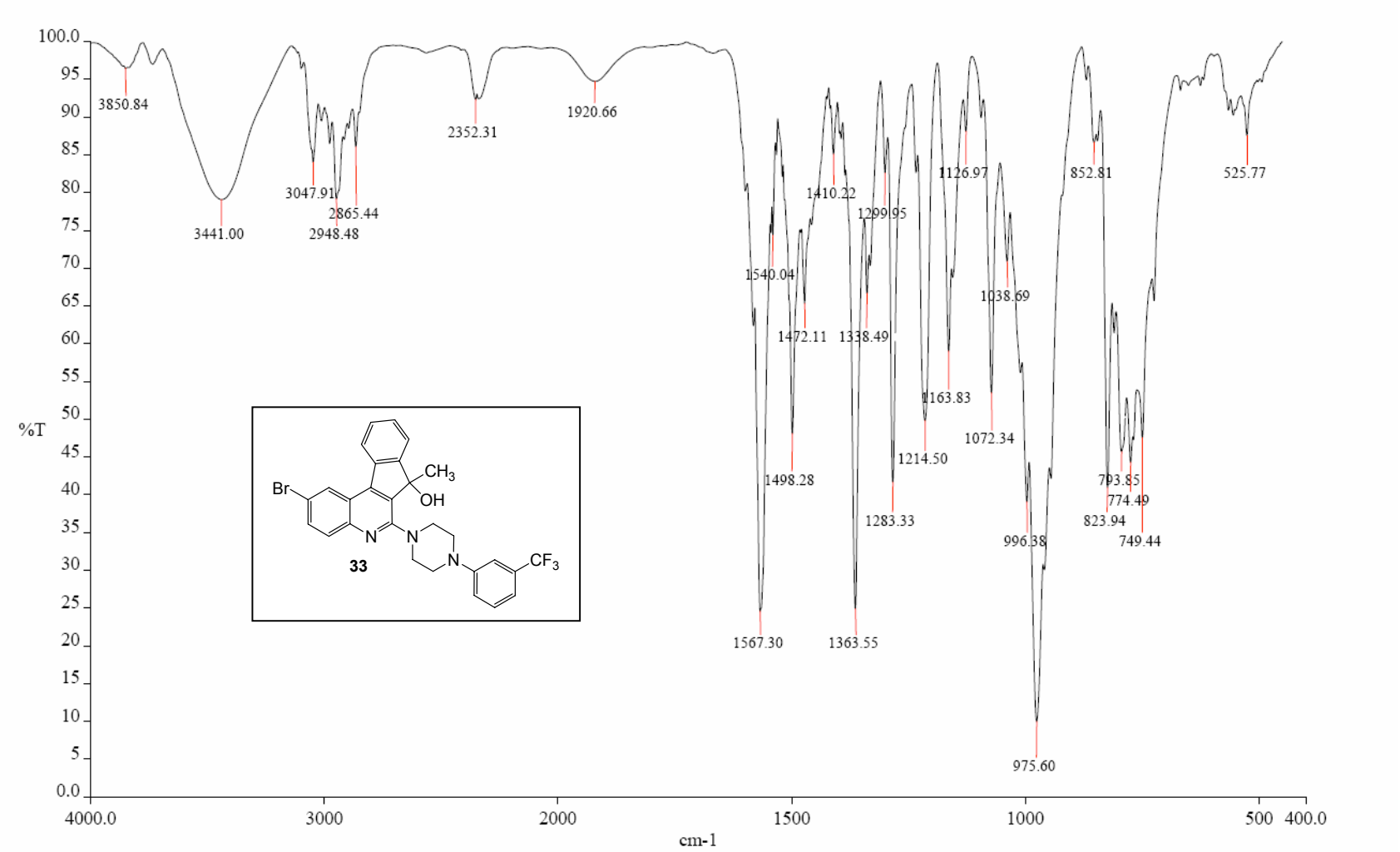
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95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
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%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3850.84
3441.00
3047.91
2948.48
2865.44
2352.31
1920.66
1567.30
1540.04
1498.28
1472.11
1410.22
1363.55
1338.49
1299.95
1283.33
1214.50
1163.83
1126.97
1072.34
1038.69
996.38
975.60
852.81
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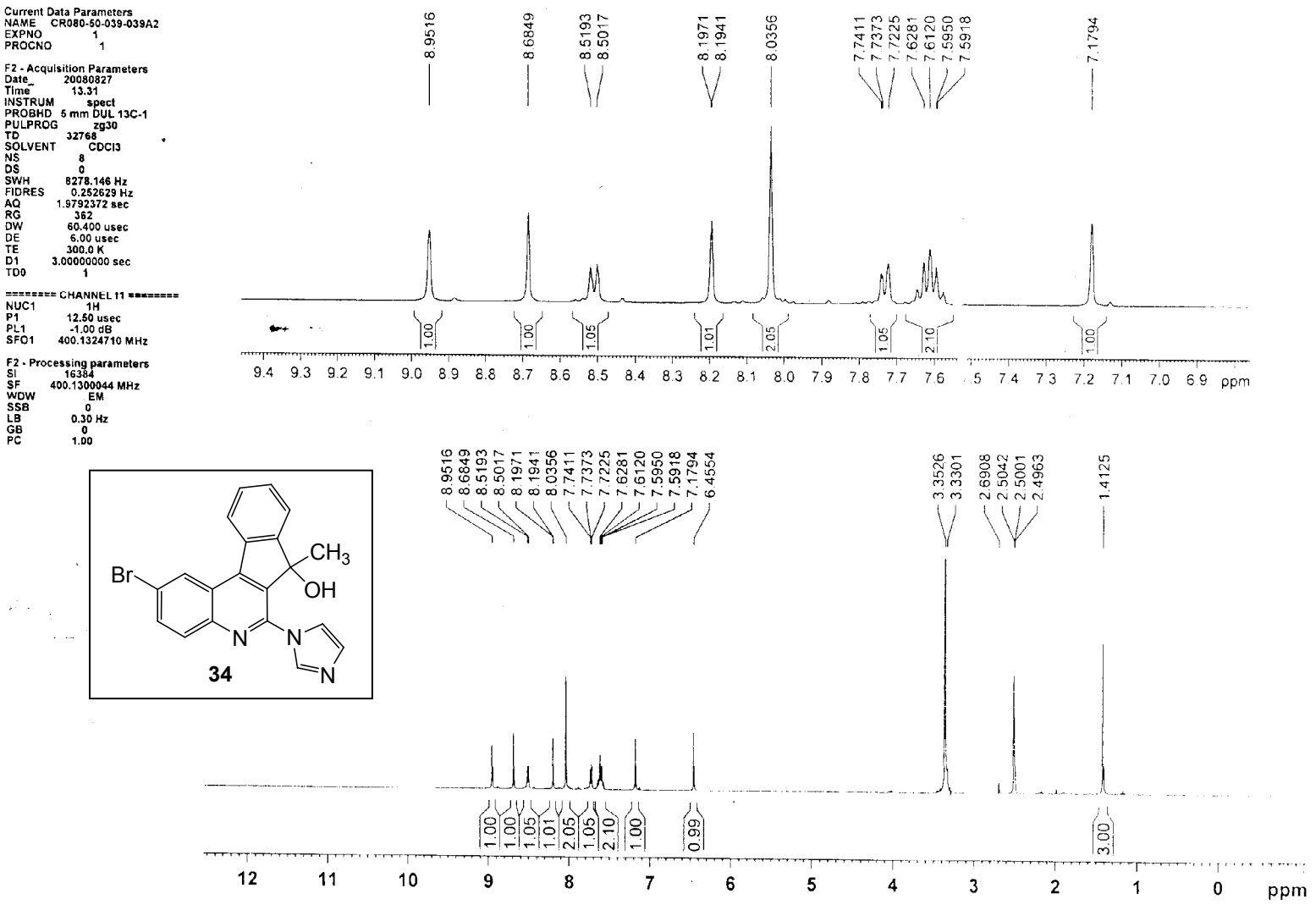
793.85
774.49
749.44
525.77
Br
CH3
OH
N
N
N
CF3
33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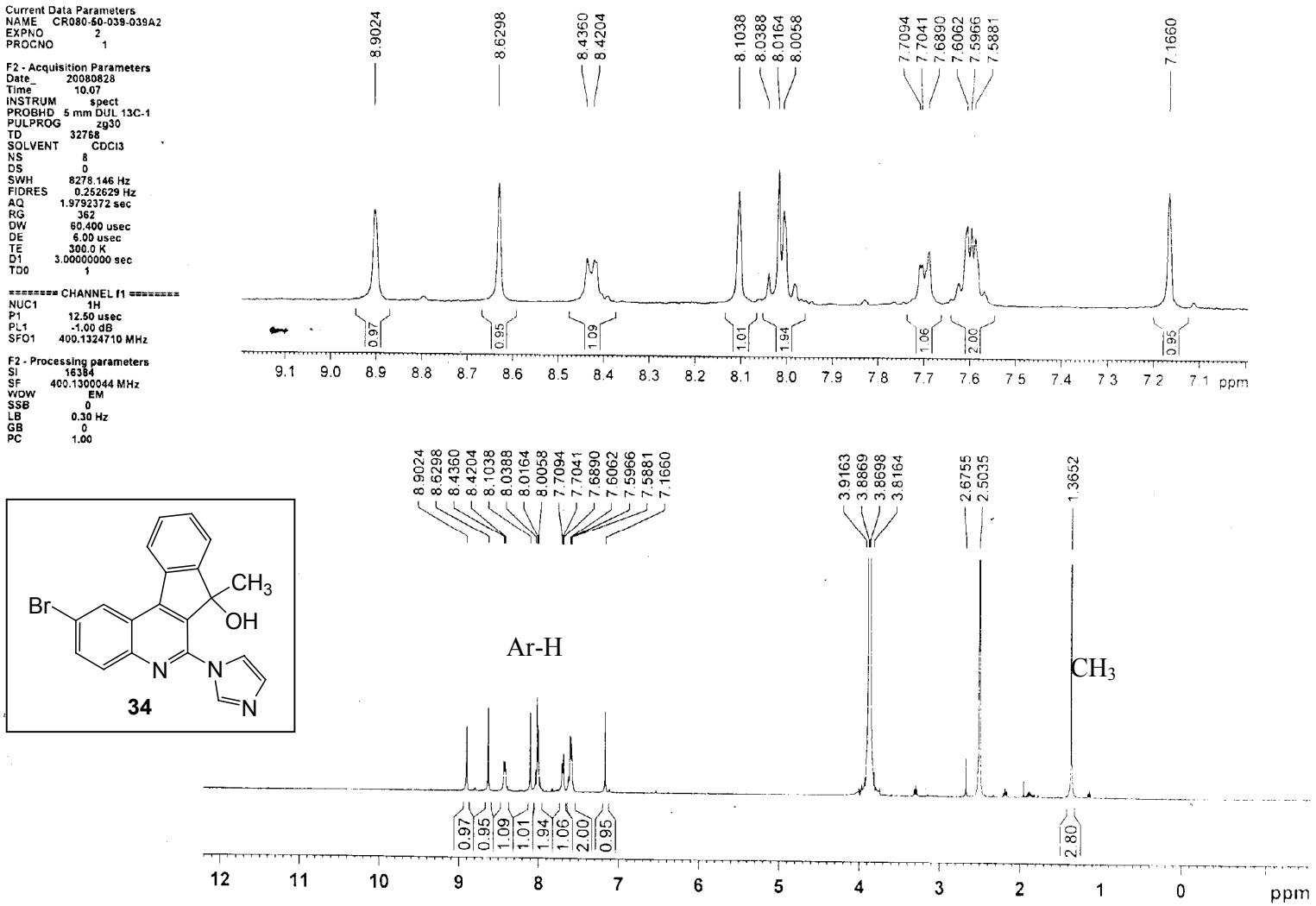
NAME CR080-50-039-039A2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080827
Time 13.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.9516
8.6849
8.5193
8.5017
8.1971
8.1941
8.0356
7.7411
7.7373
7.7225
7.6281
7.6120
7.5950
7.5918
7.1794
1.00
1.00
1.05
1.01
2.05
1.05
2.10
1.00
9.4 9.3 9.2 9.1 9.0 8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm
Br
CH3
OH
N
N
N
34
8.9516
8.6849
8.5193
8.5017
8.1971
8.1941
8.0356
7.7411
7.7373
7.7225
7.6281
7.6120
7.5950
7.5918
7.1794
6.4554
3.3526
3.3301
2.6908
2.5042
2.5001
2.4963
1.4125
1.00
1.00
1.05
1.01
2.05
1.05
2.10
1.00
0.99
3.00
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm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EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
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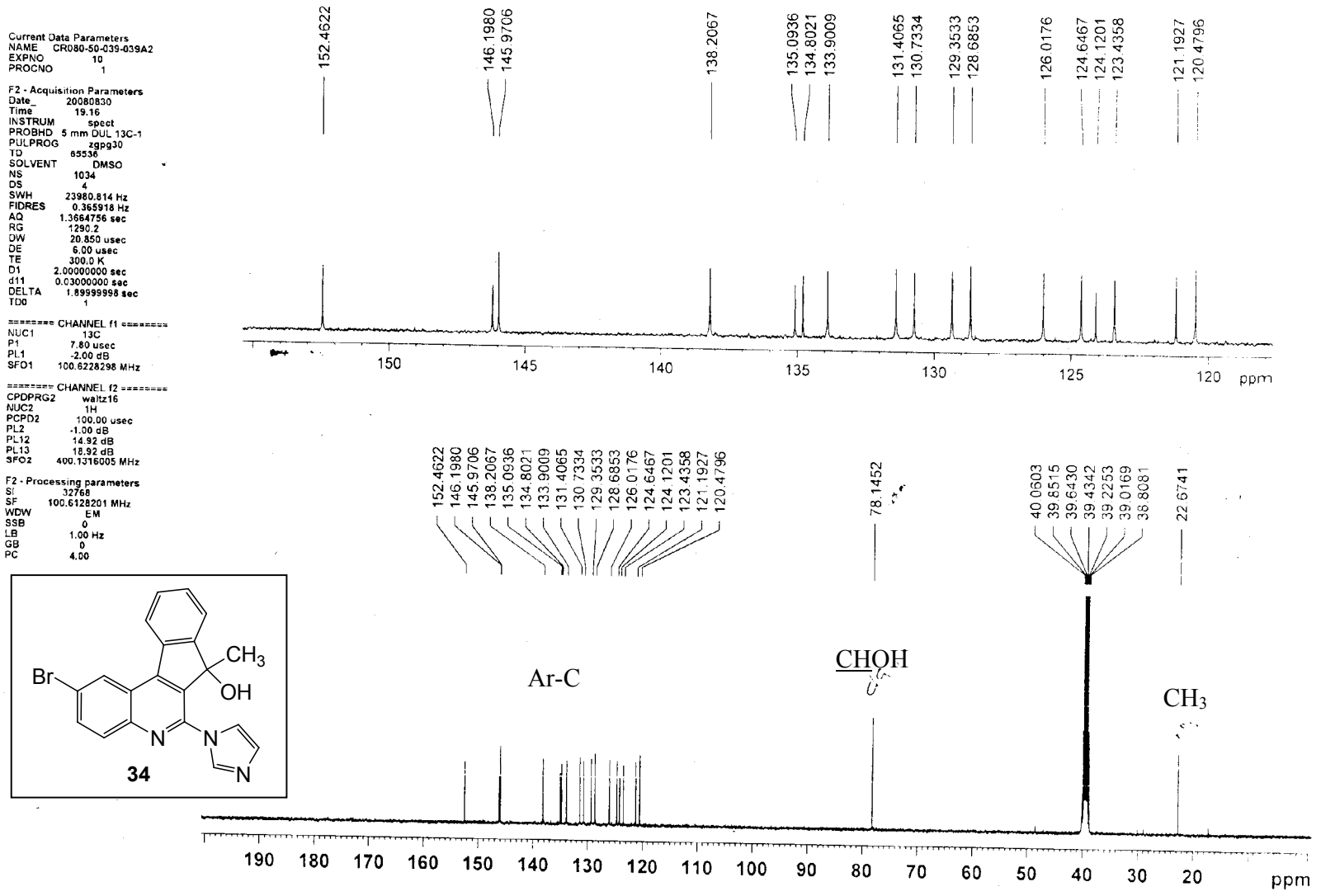
Time 10.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.9024
8.6298
8.4360
8.4204
8.1038
8.0388
8.0164
8.0058
7.7094
7.7041
7.6890
7.6062
7.5966
7.5881
7.1660
0.97
0.95
1.09
1.01
1.94
1.06
2.00
0.95
ppm
Br
CH3
OH
N
N
N
34
Ar-H
CH3
3.9163
3.8869
3.8698
3.8164
2.6755
2.5035
1.3652
2.80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EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
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Time 19.16
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1034
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 14.92 dB
PL13 18.92 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128201 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00
152.4622
146.1980
145.9706
138.2067
135.0936
134.8021
133.9009
131.4065
130.7334
129.3533
128.6853
126.0176
124.6467
124.1201
123.4358
121.1927
120.4796
150
145
140
135
130
125
120
ppm
Br
CH3
OH
N
N
N
34
78.1452
40.0603
39.8515
39.6430
39.4342
39.2253
39.0169
38.8081
22.6741
Ar-C
CHOH
CH3
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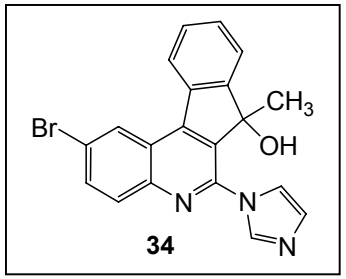
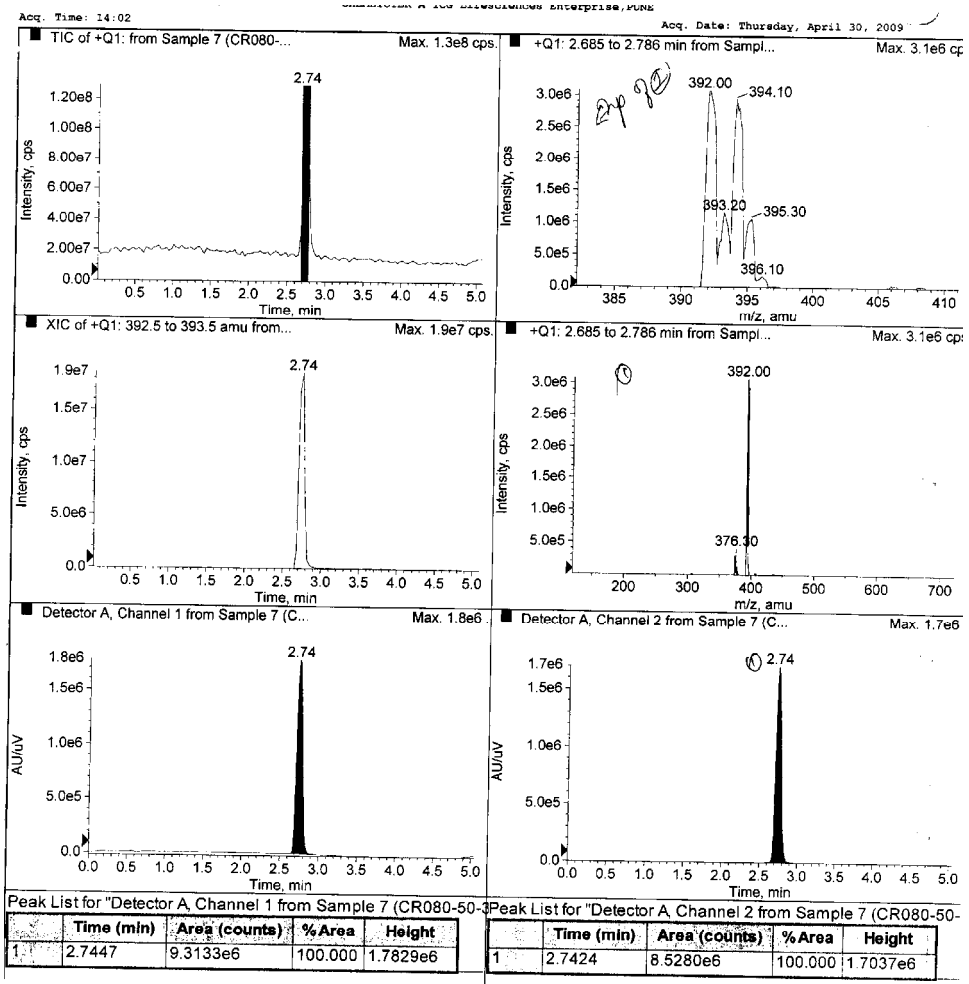
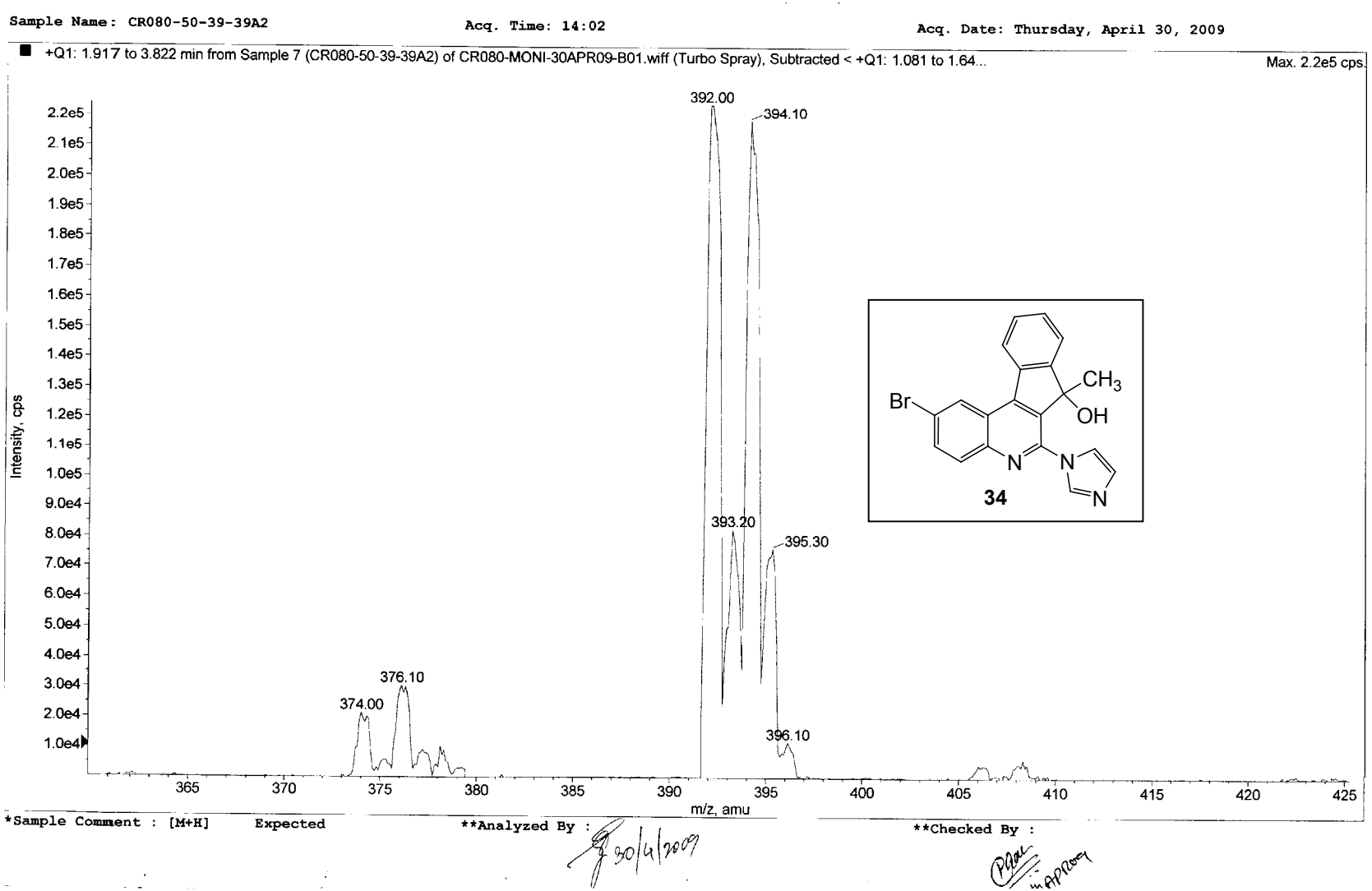
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Br
CH3
OH
N
N
N

34


Acq. Time: 14:02
Acq. Date: Thursday, April 30, 2009
TIC of +Q1: from Sample 7 (CR080-...
Max. 1.3e8 cps.
2.74
Intensity, cps
Time, min
+Q1: 2.685 to 2.786 min from Sampl...
Max. 3.1e6 cp
392.00
394.10
393.20
395.30
396.10
m/z, amu
XIC of +Q1: 392.5 to 393.5 amu from...
Max. 1.9e7 cps.
+Q1: 2.685 to 2.786 min from Sampl...
Max. 3.1e6 cps
376.30
Detector A, Channel 1 from Sample 7 (C...
Max. 1.8e6
AU/uV
Detector A, Channel 2 from Sample 7 (C...
Max. 1.7e6
Peak List for "Detector A, Channel 1 from Sample 7 (CR080-50-3
Time (min) | Area (counts) | %Area | Height
1 | 2.7447 | 9.3133e6 | 100.000 | 1.7829e6
Peak List for "Detector A, Channel 2 from Sample 7 (CR080-50-
Time (min) | Area (counts) | %Area | Height
1 | 2.7424 | 8.5280e6 | 100.000 | 1.7037e6
CMS-2 ReactionMonitoring in TFA
Channel 1 at wavelength 220nm, Channel 2 at wavelength 260 nm
Analyzed By :
30/4/2009

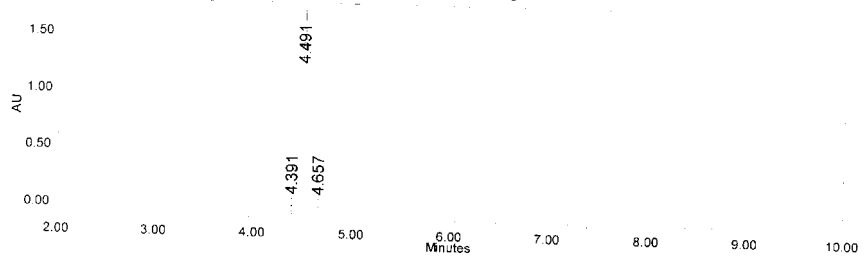

Sample Name: CR080-50-39-39A2
Acq. Time: 14:02
Acq. Date: Thursday, April 30, 2009
+Q1: 1.917 to 3.822 min from Sample 7 (CR080-50-39-39A2) of CR080-MONI-30APR09-B01.wiff (Turbo Spray), Subtracted < +Q1: 1.081 to 1.64...
Max. 2.2e5 cps.
Intensity, cps
m/z, amu
392.00
394.10
393.20
395.30
376.10
374.00
396.10
Br
CH3
OH
N
N
N
34
*Sample Comment : [M+H] Expected
**Analyzed By :
**Checked By :

GENERAL_ASSAY_REPORT

SAMPLE INFORMATION

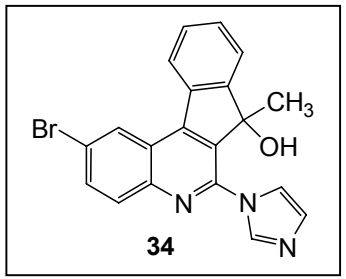
Sample Name	CR080-50-039-039A2	Acquired By	Satheesh
Sample Type	Unknown	Sample Set Name	P19NON019
Vial:	2	Acq. Method Set	P19_CR80_39A2_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_PRO
Injection Volume	2.00 ul	Label	P19090063
Run Time:	19.0 Minutes	Proc. Chanel	[2998 Ch1 205nm@1.2nm]
Date Acquired	1/30/2009 10:37:01 AM IST	Date Processed	1/30/2009 12:16:21 PM IST

Auto-Scaled Chromatogram



Peak Results

	RT	Area	% Area
1	4.391	28183	0.60
2	4.491	4630698	99.08
3	4.657	14630	0.31



34

Reported by User : satheesh Geda (Satheesh)
Report Method : GENERAL_ASSAY_REPORT
Analysed by :

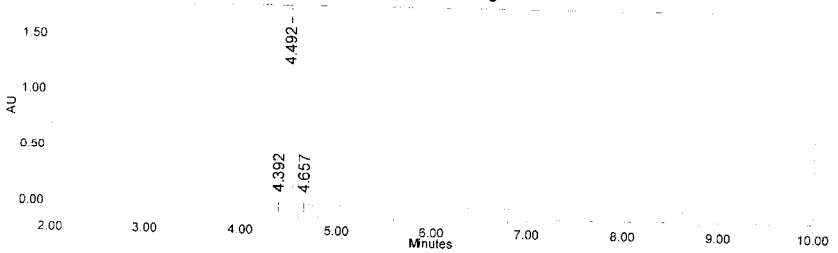
Project Name: HPLC_P19_NON GLP
Date Printed : 2/2/2009
Checked by :

Page: 1 of 1

SAMPLE INFORMATION

Sample Name	CR080-50-039-039A2	Acquired By	Satheesh
Sample Type	Unknown	Sample Set Name	P19NON019
Vial:	2	Acq. Method Set	P19_CR80_39A2_PURITY
Injection #	1	Processing Method	NON_GLP_CR080_PRO
Injection Volume	2.00 ul	Label	P19090064
Run Time:	19.0 Minutes	Proc. Chanel	[2998 Ch1 205nm@1.2nm]
Date Acquired	1/30/2009 10:57:00 AM IST	Date Processed	1/30/2009 12:16:30 PM IST

Auto-Scaled Chromatogram



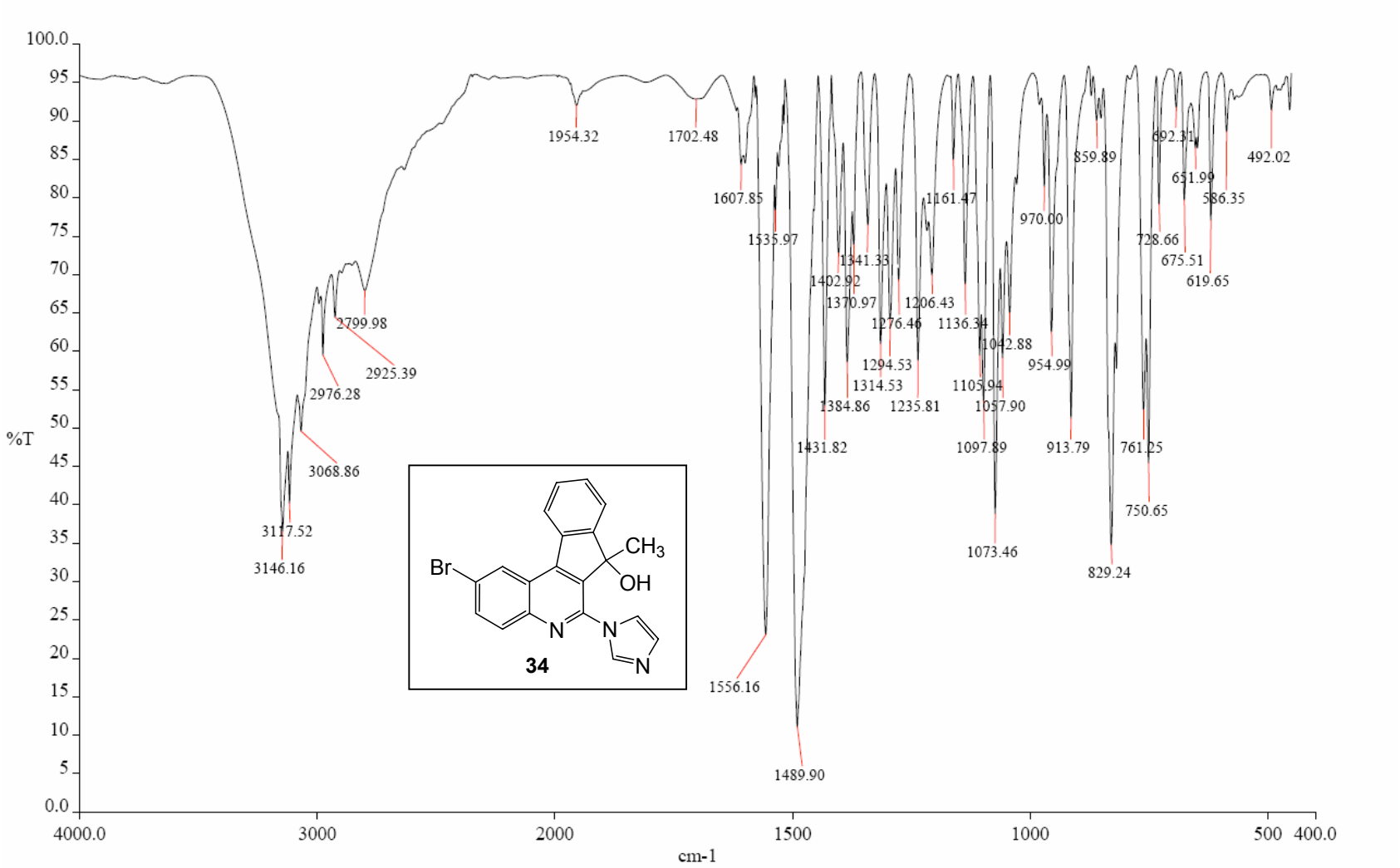
Peak Results

	RT	Area	% Area
1	4.392	27799	0.59
2	4.492	4677169	99.09
3	4.657	15015	0.32

Reported by User : satheesh Geda (Satheesh)
Report Method : GENERAL_ASSAY_REPORT
Analysed by :

Project Name: HPLC_P19_NON GLP
Date Printed : 2/2/2009
Checked by :

Page: 1 of 1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95
90
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55
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45
40
35
30
25
20
15
10
5
0.0
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3146.16
3117.52
3068.86
2976.28
2925.39
2799.98
1954.32
1702.48
1607.85
1556.16
1535.97
1489.90
1431.82
1402.92
1384.86
1370.97
1341.33
1314.53
1294.53
1276.46
1235.81
1206.43
1161.47
1136.34
1105.94
1097.89
1073.46
1057.90
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913.79
859.89
829.24
761.25
750.65
728.66
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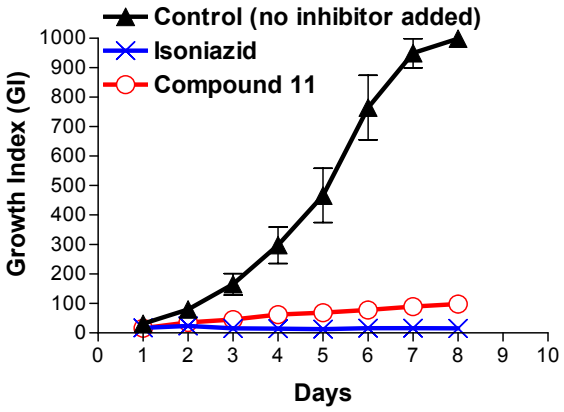
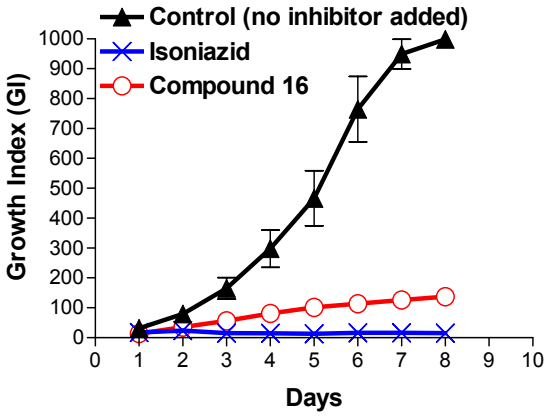
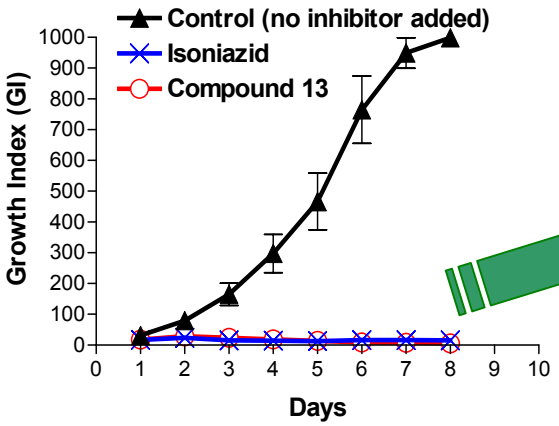
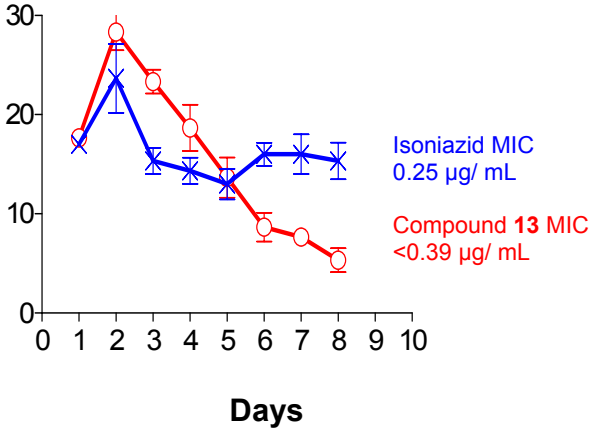
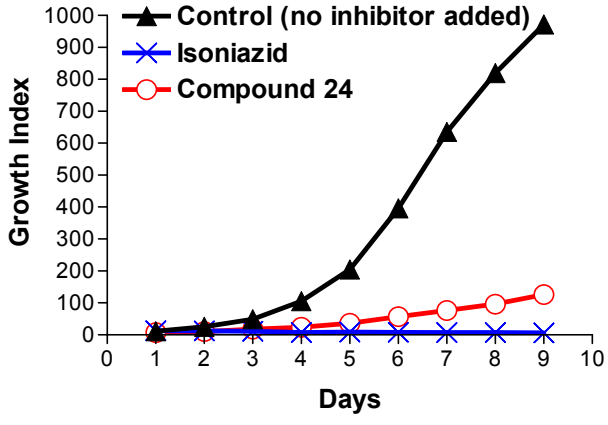
675.51
651.99
619.65
586.35
492.02
Br
CH3
OH
N
N
N
34

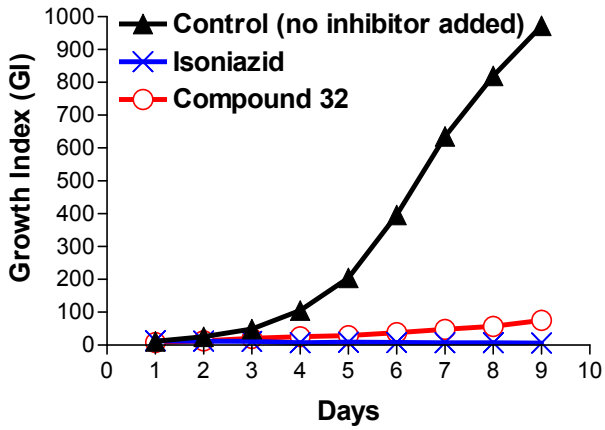

Control (no inhibitor added)
Isoniazid
Compound 11
Growth Index (GI)
Days


Control (no inhibitor added)
Isoniazid
Compound 16
Growth Index (GI)
Days


Control (no inhibitor added)
Isoniazid
Compound 13
Growth Index (GI)
Days


Isoniazid MIC
0.25 µg/ mL
Compound 13 MIC
<0.39 µg/ mL
Days


Control (no inhibitor added)
Isoniazid
Compound 24
Growth Index
Days


Control (no inhibitor added)
Isoniazid
Compound 32
Growth Index (GI)
Days

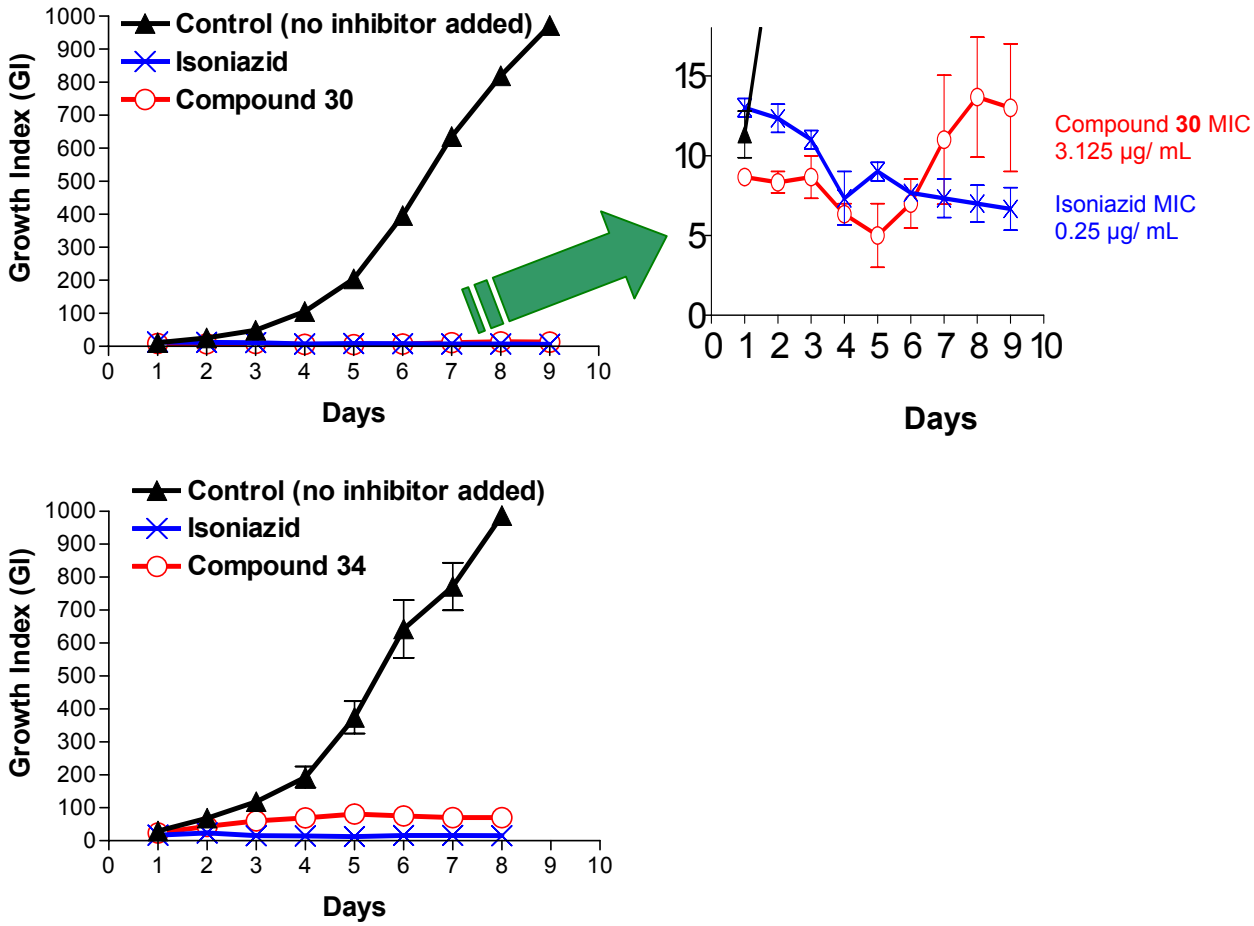


Figure S1: Plots showing Growth Index (GI) of TB H37Rv strain with standard deviation for 8 consecutive days for compounds **11**, **13**, **16**, **24**, **30**, **32** and **34** in comparison with isoniazid at a fixed concentration of 6.25 µg/mL upon single-administration of each of the substances on day-one. Standard deviation are shown in Table S1 in supplementary data.

Antimycobacterial activity:

All the compounds were screened for antimycobacterial activity against mycobacterium tuberculosis by BACTEC 460 radiometric methods. The broth based BACTEC 460 TB system was used for the growth of Mycobacteria. In this growth system, *Mycobacterium Tuberculosis* $H37R_V$ was grown in the C^{14} labelled substrate in 7H12 medium, substrate is utilized by growing *mycobacteria* and $^{14}CO_2$ is produced, which is detected in the form of '**Growth Index'** (GI), which reflects the rate and amount of growth in the medium vial. If an antituberculosis drug will be added to the medium vial, it will suppress the growth of the bacteria which is detected by the decrease of the GI values as compared to the control vial. The positive controls taken in the experiment were the standard therapeutic drugs presently used for the treatment of the tuberculosis, Isoniazid and Rifampin.

The rate of increase in GI values or the change in GI over the previous day GI is called delta (Δ) GI, is compared with that of control values. If ΔGI of the drug-containing vial is equal to or greater than (≥) than that in control vial the organisms are considered resistant to the drug. On the contrary if ΔGI of the drug-containing vial is less (<) than that in control vial the organisms are considered susceptible.

On the basis of the values calculated for ΔG, out of all screened compounds, seven compounds i.e. **11**, **13**, **16, 24, 30, 32** and **34** were found to act as active antimycobacterial agents. Below given table depicts ΔG of the compounds tested.

Table S1: **Biological activity data table**

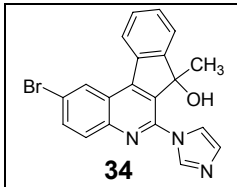
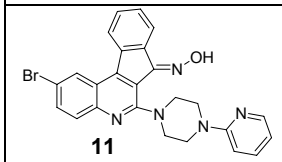
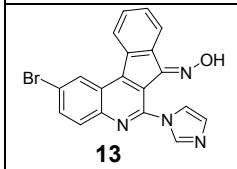
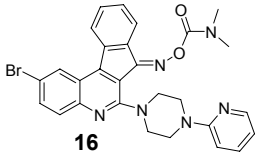
	Days	**1**	**2**	**3**	**4**	**5**	**6**	**Δ GI**	**7**	**Δ GI**	**8**	**Δ GI**	**% Inhibition**
 **34**	**GI**	24	44	59	69	83	81	-2	83	2	90	7	91
		24	44	61	67	74	63	-11	52	-11	46	6	95.4
		22	44	62	73	88	83	-5	76	-7	74	-2	92.6
	SD												**3.15**
 **11**	**GI**	16	35	47	65	79	90	11	107	17	121	14	88.0
		17	39	48	70	75	84	9	97	13	112	15	89.0
		15	32	42	51	53	60	7	64	04	60	4	94.0
	SD												**4.54**
 **13**	**GI**	18	26	24	18	14	9	-5	8	-1	6	-2	99.4
		17	27	21	15	10	6	-4	8	-2	3	-5	99.7
		18	32	25	23	17	11	-6	7	-4	7	0	99.3
	SD												**0.29**
 **16**	**GI**	13	31	54	76	101	108	7	129	21	138	8	86.2
		16	44	71	97	123	142	19	156	14	161	5	84.0
		12	31	43	70	80	92	12	95	03	111	17	89.0
	SD												**3.54**
Isoniazid	**GI**	17	30	14	13	11	14	3	14	0	13	-1	99.7
		18	18	14	13	12	16	4	14	-2	14	0	99.6
		16	23	18	17	16	18	2	20	2	19	-1	98.0
	SD												**1.35**
Control	**GI**	31	76	157	278	461	834	373	999	165	999	0	
		40	110	231	414	629	910	281	999	89	999	0	
		22	52	107	201	310	550	240	850	300	999	149	
	SD												

Table S2: Biological activity data table

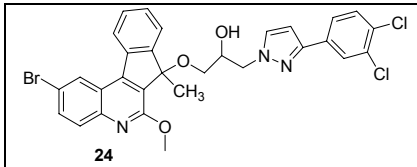
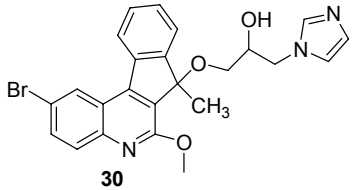
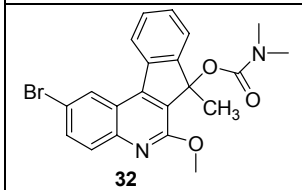
	Days	1	2	3	4	5	6	7	Δ GI	8	Δ GI	9	Δ GI	% Inhibition
 24	**GI**	7	10	17	24	35	53	77	14	98	21	121	23	88.0
		6	10	16	19	31	50	63	13	80	17	107	27	89.3
		8	12	21	28	42	68	88	20	112	24	150	38	85.0
	SD													**3.11**
 30	**GI**	9	9	10	7	7	9	11	2	14	3	17	3	98.3
		9	9	10	7	7	8	18	10	20	2	17	-3	98.3
		8	7	6	5	1	4	4	0	7	3	5	2	99.5
	SD													**0.98**
 32	**GI**	11	16	25	30	30	39	46	7	55	9	69	14	93.0
		7	11	18	23	28	39	49	10	59	10	73	14	92.7
		7	11	17	24	28	36	48	12	58	10	83	25	91.7
	SD													**0.96**
Isoniazid	**GI**	17	30	14	13	11	14	14	0	13	-1			99.7
		18	18	14	13	12	16	14	-2	14	0			99.6
		16	23	18	17	16	18	20	2	19	-1			98.0
	SD													**1.35**
Control	**GI**	14	30	57	122	216	431	643	212	788	145	999	211	
		11	24	43	99	192	384	622	238	801	179	918	117	
		9	21	46	95	206	374	641	267	869	228	999	130	
	SD													

Table S3: Docking, scoring and interaction energy data of compounds (data set I and II).

Comp. Number*	AD4			AD4.1			Interaction Energy			PMF[j]	Xscore[k]
	vdw[a]	elec[b]	IE[c]	vdw[d]	elec[e]	IE[f]	vdw[g]	elec[h]	Tot[i]		
Data Set I											
10	-7.87	-0.06	-7.92	-8.7710	-0.0534	-8.8244	-44.97	-6.70	-51.68	-243.79	6.60
11	-2.25	-0.13	-2.38	-5.6198	-0.1250	-5.7448	-56.09	-26.55	-82.65	-258.73	7.40
12	1.51	-0.15	1.36	-1.9850	-0.1456	-2.1306	-59.43	-15.18	-74.62	-228.93	7.08
13	-5.54	-0.05	-5.59	-7.2919	-0.0425	-7.3344	-50.09	-9.22	-59.32	-244.70	6.59
14	-5.50	-0.04	-5.53	-7.2565	-0.0334	-7.2899	-49.47	-9.47	-58.95	-238.64	6.68
15	-9.21	-0.07	-9.28	-10.4583	-0.0602	-10.5185	-54.72	-10.10	-64.82	-275.21	6.81
16	-1.32	0.03	-1.29	-5.3645	0.0056	-5.3589	-62.32	-24.30	-86.63	-307.29	7.49
17	0.20	-0.06	0.14	-4.3858	-0.0716	-4.4574	-67.11	-18.48	-85.59	-269.78	7.42
18	-12.55	-0.05	-12.60	-14.0854	-0.0453	-14.1307	-64.09	-13.71	-77.86	-244.50	8.48
19	7.18	-0.17	7.02	0.5409	-0.1677	0.3732	-78.22	-3.77	-81.99	-238.18	8.29
20	1.46	-0.03	1.43	-4.4473	-0.0394	-4.4867	-73.09	-18.54	-91.64	-247.44	8.74
Data Set II											
23	-9.82	-0.21	-10.03	-13.0369	-0.1881	-13.2250	-77.06	-25.71	-102.78	-316.12	8.22
24	-7.64	-0.02	-7.67	-11.1361	-0.0325	-11.1686	-68.35	-37.46	-105.81	-325.07	8.22
25	-8.55	0.05	-8.50	-11.1767	0.0467	-11.1300	-68.53	-17.83	-86.36	-287.88	8.68
26	-6.20	-0.07	-6.27	-9.4045	-0.0581	-9.4626	-69.95	-19.98	-89.93	-340.26	8.02
27	-9.44	-0.04	-9.48	-12.4930	-0.0371	-12.5301	-70.12	-8.04	-78.17	-298.04	8.17
28	-8.91	-0.02	-8.93	-10.7990	-0.0290	-10.8280	-56.00	-9.16	-65.17	-291.67	7.14
29	-8.88	-0.02	-8.90	-10.8636	-0.0194	-10.8830	-60.95	-6.10	-67.05	-290.93	6.98

30	-7.46	-0.14	-7.60	-9.7438	-0.1171	-9.8609	-57.26	-5.96	-63.22	150.14	6.63
31	-9.20	-0.37	-9.57	-10.9241	-0.3550	-11.2791	-59.45	-19.56	-79.01	-298.87	6.99
32	-7.37	-0.07	-7.44	-9.0900	-0.0619	-9.1519	-51.78	-6.90	-58.68	-298.59	6.71
33	2.93	-0.10	2.83	-1.5763	-0.1042	-1.6805	-59.85	-15.12	-74.98	-237.39	7.48
34	-5.14	-0.03	-5.17	-7.0758	-0.0248	-7.1006	-50.52	-10.24	-60.77	-248.23	6.70
37	-9.11	0.06	-9.05	-12.1413	0.0435	-12.0978	-69.99	-33.58	-103.5	155.01	8.24

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vdw[a] Autodock 4 (AD4) van der Waals, hydrogen bond and desolvation energy
elec[b] Autodock 4 (AD4) electrostatic energy
IE[c] Autodock 4 (AD4) intermolecular energy
vdw[d] Autodock 4.1 (AD4.1) van der Waals, hydrogen bond and desolvation energy
elec[e] Autodock 4.1 (AD4.1) electrostatic energy
IE[f] Autodock 4.1 (AD4.1) intermolecular energy
vdw[g] van der Waals energy from energy minimised protein-ligand complex
elec[h] electrostatic energy from energy minimised protein-ligand complex
Tot[i] total interaction energy from energy minimised protein-ligand complex
PMF[j] Potential of mean force score (in kcal/mol)
Xscore[k] Average of HPScore, HMScore and HSScore

Theoretical Methods

Homology model comprising of 1 subunit A and 2 subunit C of ATP-synthase of *Mycobacterium tuberculosis,* was generated as described before.[1] The protonation state of three critical amino acid residues was kept as: $Glu61^{C}$ (deprotonated) – $Arg186^{A}$ (protonated) – $Glu61^{C}$ (protonated).

Ligand molecules were sketched in Ghemical[2] irrespective of their stereochemistry and refined with 2500 steps of energy minimisation. Further, a random conformational analysis was performed on all the ligand molecules, with 2000 search steps and each step followed by 300 steps of energy minimisation. Finally, conformation with lowest energy was selected for docking. Ligand molecules were docked into the previously discussed binding site of ATP-synthase[1,3] using AutoDock Tools[4] and AutoDock.[5] Grid size of 64Å×42Å×44Å was used. Docking parameters; population size, number of energy evaluations and number of Lamarckian Genetic Algorithm runs were kept at 250, 25000000 and 100, respectively. The initial position of ligand was defined same as the grid center position (*tran0* option in AutoDock).

AutoDock clustering (with 2Å root mean square) was used to select the ligand conformation/pose. In most of the cases, the best cluster of ligand conformations was the lowest energy cluster too, suggesting statistical significance of results. In this case, the conformation with lowest binding energy was selected. In case of scattered clustering or that best cluster didn't coincide with lowest energy cluster, the conformation with lowest binding energy was selected for further analysis. In most of the cases our results have high statistical significance because of good root mean square clustering. The **.pdbqt* files of selected protein-ligand complexes were saved for further use.

Protein-ligand complexes of all the compounds were subjected to energy minimisation in AMBER.[6] Amino acid residues of protein were treated with *ff03* forcefield,[7] while General Amber Force Field (GAFF)[8] was used for the ligand molecules. Partial charges of ligand molecules were calculated based on AM1-BCC[9] methodology implemented in *antechamber.*[10] The complete protein-ligand system was minimised for 2500 steps of conjugate gradients and 2500 steps of steepest descent minimisation in

AMBER using *sander*. Protein-ligand interaction energy (sum of van der Waals and electrostatic energy) was calculated for the minimised complex of each compound using NAMDEnergy, implemented in NAMD/Visual Molecular Dynamics (VMD).[11,12]

Additional scoring tools such as PMF scoring available in Molden[15] and Xscore[16] were used. The protein-ligand complex was loaded in Molden and PMF score was calculated as shown by the plots of PMF score (y-axis) *vs* conformation (x-axis). During the analysis, the hydrogen bond distance and angle criteria were: Donor-H.....Acceptor distance < 3.5Å and Donor-H-Acceptor angle > 150º. All visualisation of molecules was done using Visual Molecular dynamics (VMD). Docking and forcefield based calculations were performed on 2.2GHz AMD Dual core computing machine running with Red Hat Enterprise Linux 5.

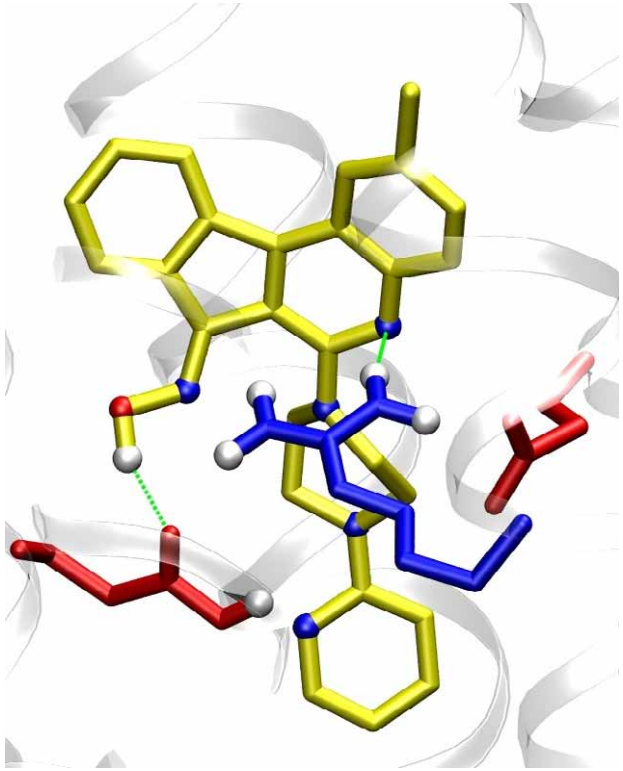

Figure S2: Protein-ligand interactions shown for compound **11**. $Glu61^{C}$, $Arg186^{A}$ and ligand molecule are shown in red, blue and yellow licorice respectively. Nitrogen, Oxygen and Hydrogen are shown as blue, red and white spheres, respectively. Transmembrane helices are shown with light-grey ribbons. Interactions are shown with green dotted line.

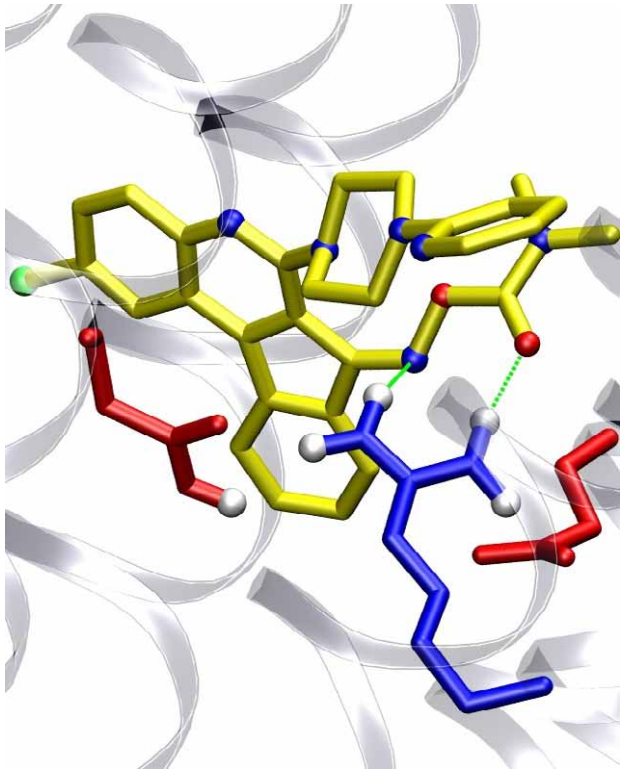

Figure S3: Protein-ligand interactions shown for compound **16**. Glu61^{C}, Arg186^{A} and ligand molecule are shown in red, blue and yellow licorice respectively. Nitrogen, Oxygen and Hydrogen are shown as blue, red and white spheres, respectively. Transmembrane helices are shown with light-grey ribbons. Interactions are shown with green dotted line.

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