

Supporting Information

for

Synthesis of compounds related to the anti-migraine drug eletriptan hydrobromide

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* Corresponding author

Characterization data of compounds 2 to 8

ELETRIPTAN DIMER CHP (1641) 160 A.R.NO: S-01011
DMSO-d6

APL-RESEARCH CENTRE

Current Data Parameters
NAME chp-1641-160
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20101101
Time 9.35
INSTRUM dpx300
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 32768
RG 203.7
SOLVENT DMSO
NS 16
AQ 1.8219508 sec
DW 55.600 usec
DE 6.00 usec
D1 2.00000000 sec
P1 6.75 usec
PL1 -6.00 dB
SF01 300.1315006 MHz

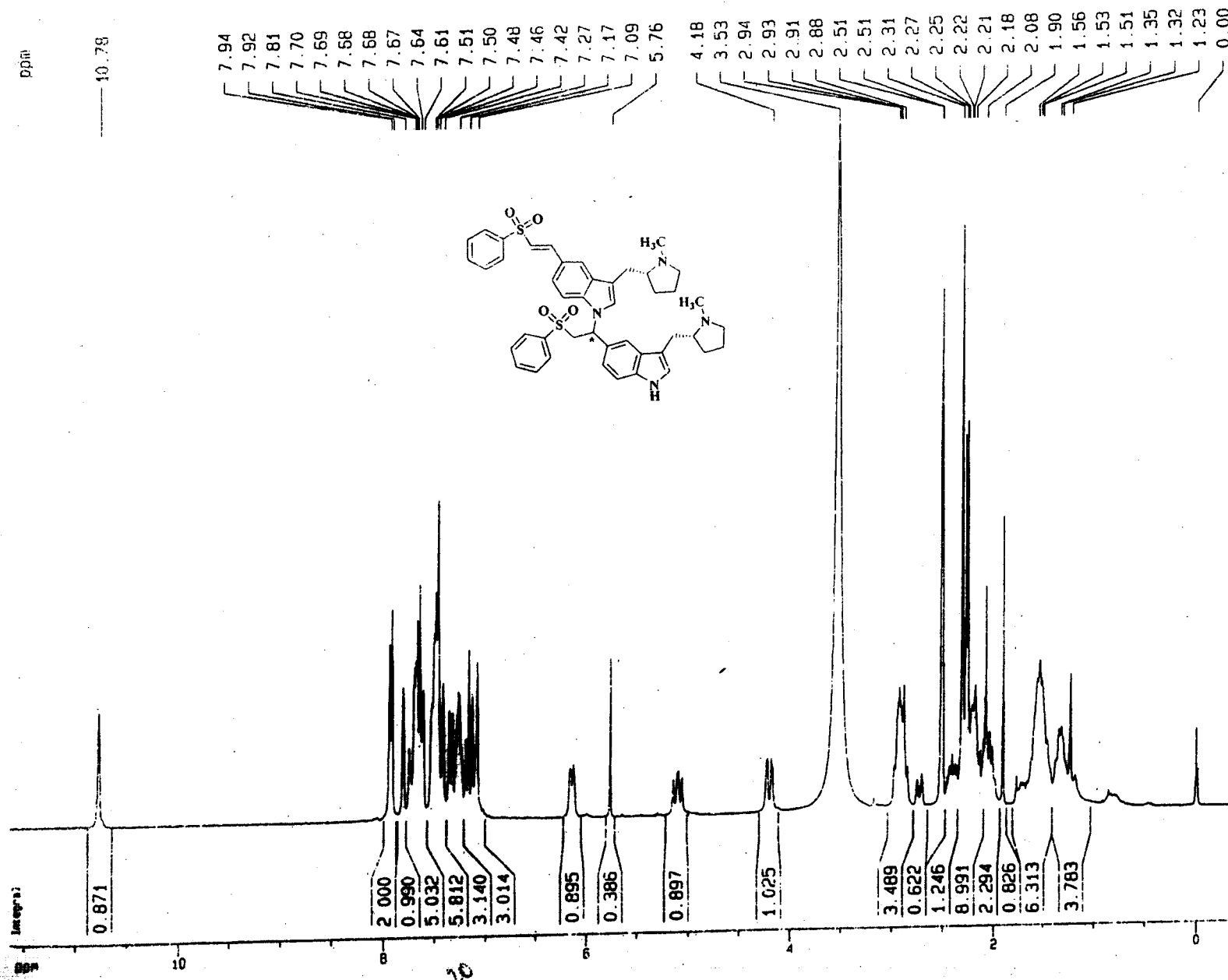
----- CHANNEL f1 -----
NUC1 1H

F2 - Processing parameters

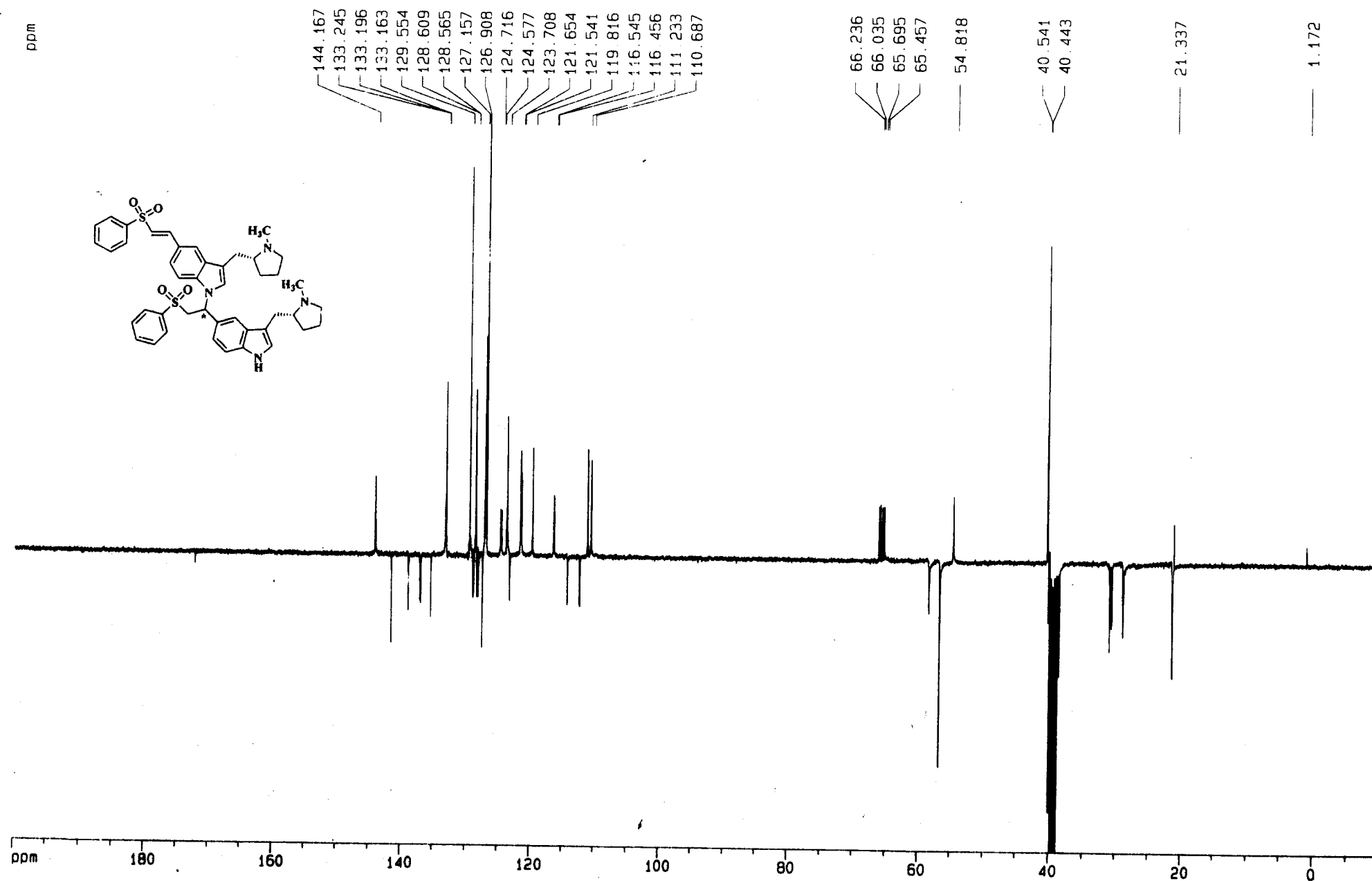
SI 32768
SF 300.1299993 MHz
WDW EM
LB 0.30 Hz
GB 0

1D NMR plot parameters

CX 20.50 cm
F1P 11.634 ppm
F2P -0.351 ppm



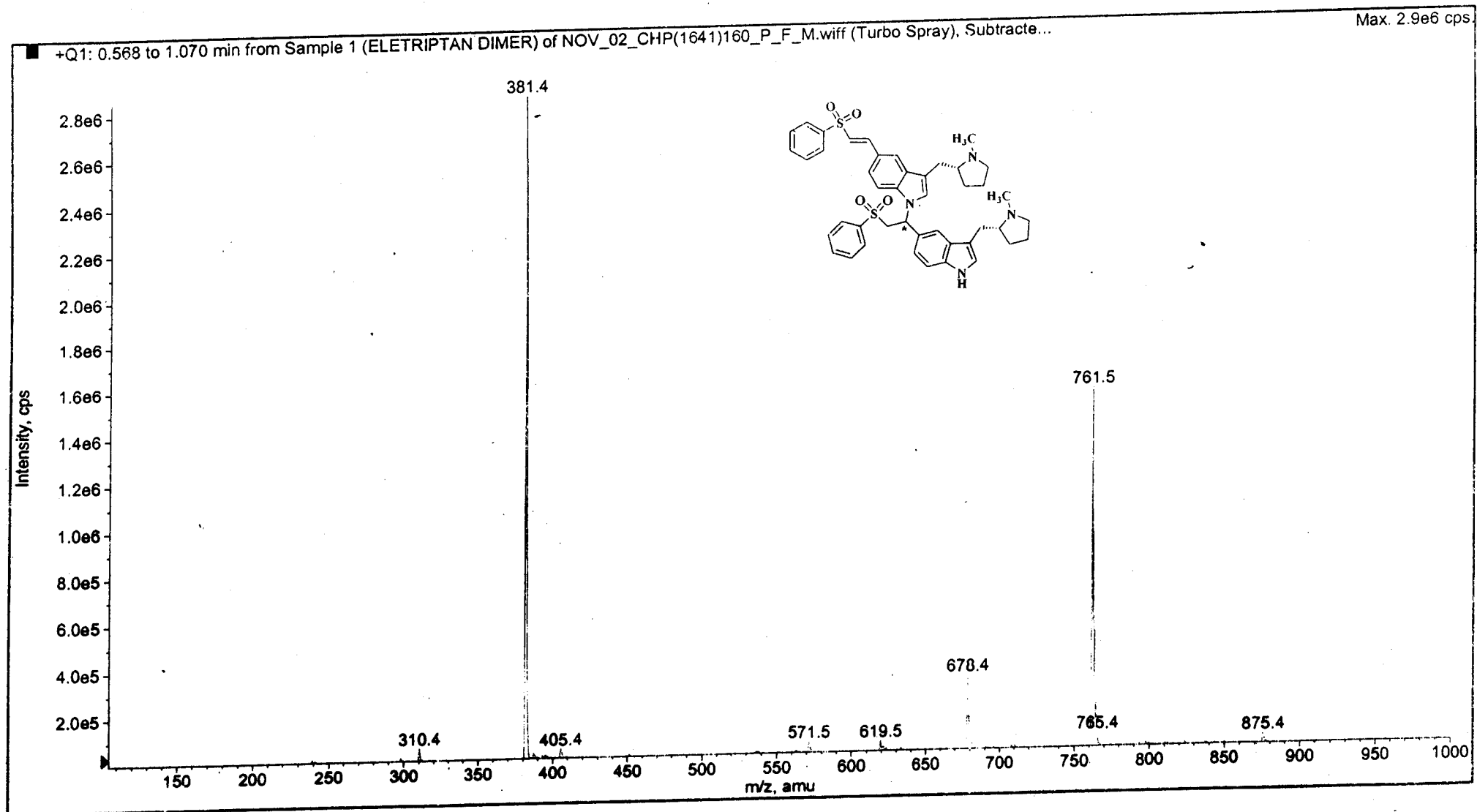
PENDANT-NMR of ELETRIPTAN DIMER CHP (1641) 160 A.R.NO: S-01011
DMSO-d6



Analyst Version: 1.4.2
Acq. Time: 11:33
Acq. Date: Tuesday, November 02, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: B.NO:CHP(1641)160
Sample Name: ELETRIPTAN DIMER
Acq. File: NOV_02_CHP(1641)160_P_F_M.wiff



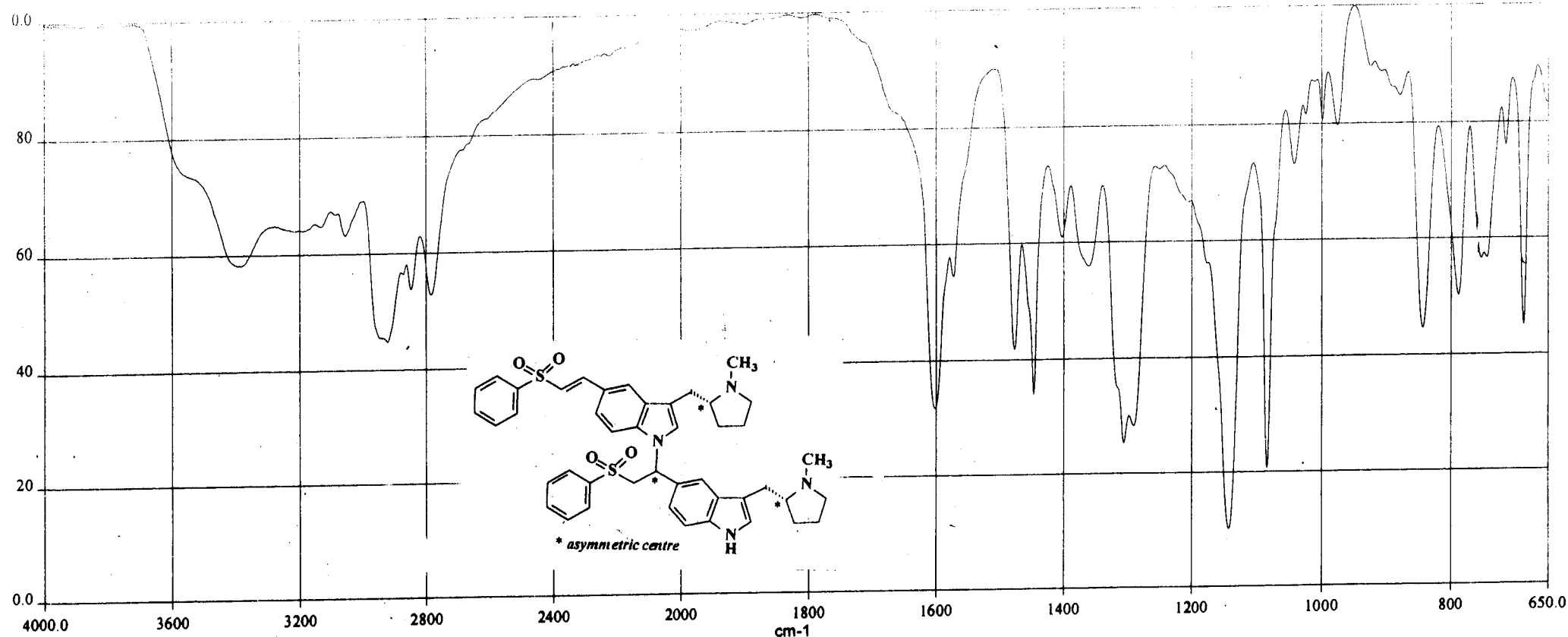
Period/Expt.: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

K.P. Ram
Analyst

Date: Monday, May 23, 2011

APL RESEARCH CENTRE

ARE_018



Status

(ELETRIPTAN DIMER)

Filename: ELETRIPTAN DIMER-1&2-CHP(1641)160.002
Date Created: Monday, May 23, 2011 11:03 AM India Standard Time
Analyst: Administrator
Description: B.NO.: CHP(1641)160
Comments:
SAMPLE: ELETRIPTAN HBr DIMER-1&2
PROJECT: ELETRIPTAN HBr
2.04 mg OF SAMPLE IN 427 mg OF KBr

Abcissa: (cm-1)

Start: 4000.00

End: 450.00

Interval: -1.000000

Ordinate: (%T)

Maximum: 100.00

Minimum: 10.00

Points: 3551

Instrument Model :

Spectrum One

Serial Number: 43000

ELETRIPTAN DIMER-1&2-CHP(1641)160.pk

ELETRIPTAN DIMER-1&2-CHP(1641)160.002 3351 4000.00 650.00 10.00 99.86 4.00 %T 8 1.00
B.NO.: CHP(1641)160
REF 4000 99.69 2000 97.55 600
3391.40 58.16 3056.39 63.10 2923.62 44.86 2848.85 53.84 2784.98 52.83
1601.36 31.72 1572.63 54.55 1476.32 41.80 1446.57 34.00 1402.80 60.94
1360.53 55.99 1305.42 25.23 1290.33 28.24 1143.06 9.99 1084.20 20.64
1042.23 73.10 1024.40 81.67 998.88 80.51 975.69 79.73 878.36 84.78
843.40 44.68 787.94 50.23 752.60 56.43 714.54 76.06 687.29 45.03

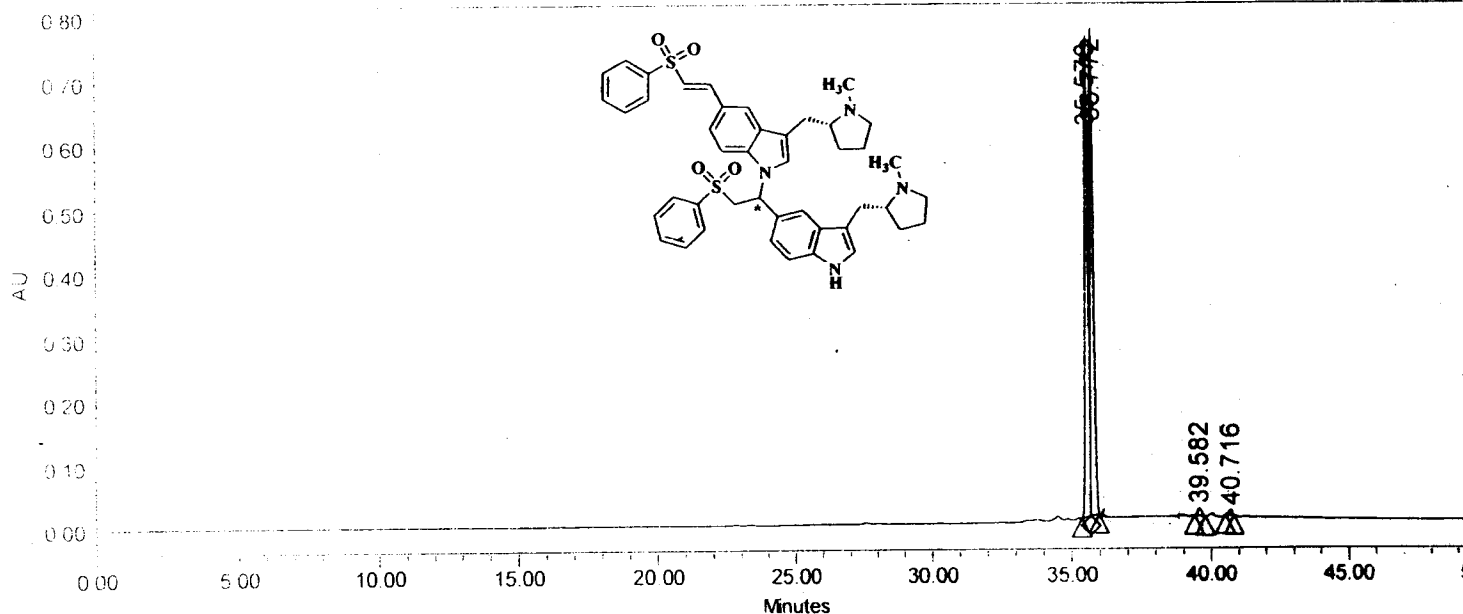
23/05/2011

APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012RCII_AE088

Sample ID	CHP(1641)160/Eletriptan Dimer1&2 (ELETRIPTAN DIMER)	Proc. Chnl. Descr	W2996 PDA 225.0 nm
Run Time	60.0 Minutes	Date Acquired	3/29/2012 10:09:10 PM IST
Vial	33	Acq. Method Set	Eletriptan_RS_MET
Injection	1	Date Processed	3/30/2012 5:17:25 PM IST
Injection Volume	20.00 ul	Processing Method	Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu V \cdot sec$)	% Area	RT Ratio	Name
1	35.58	5928850	47.96	1.00	Eletriptan Dimer-1
2	35.77	6243522	50.50	1.01	Eletriptan Dimer-2
3	39.58	118176	0.96	1.11	Peak9
4	40.72	72638	0.59	1.14	Peak10

ELETRIPTAN N-OXIDES-1 CHP (1708) 21 A.R.NO: S-01012671
DMSO-d6

APL-RESEARCH CENTRE

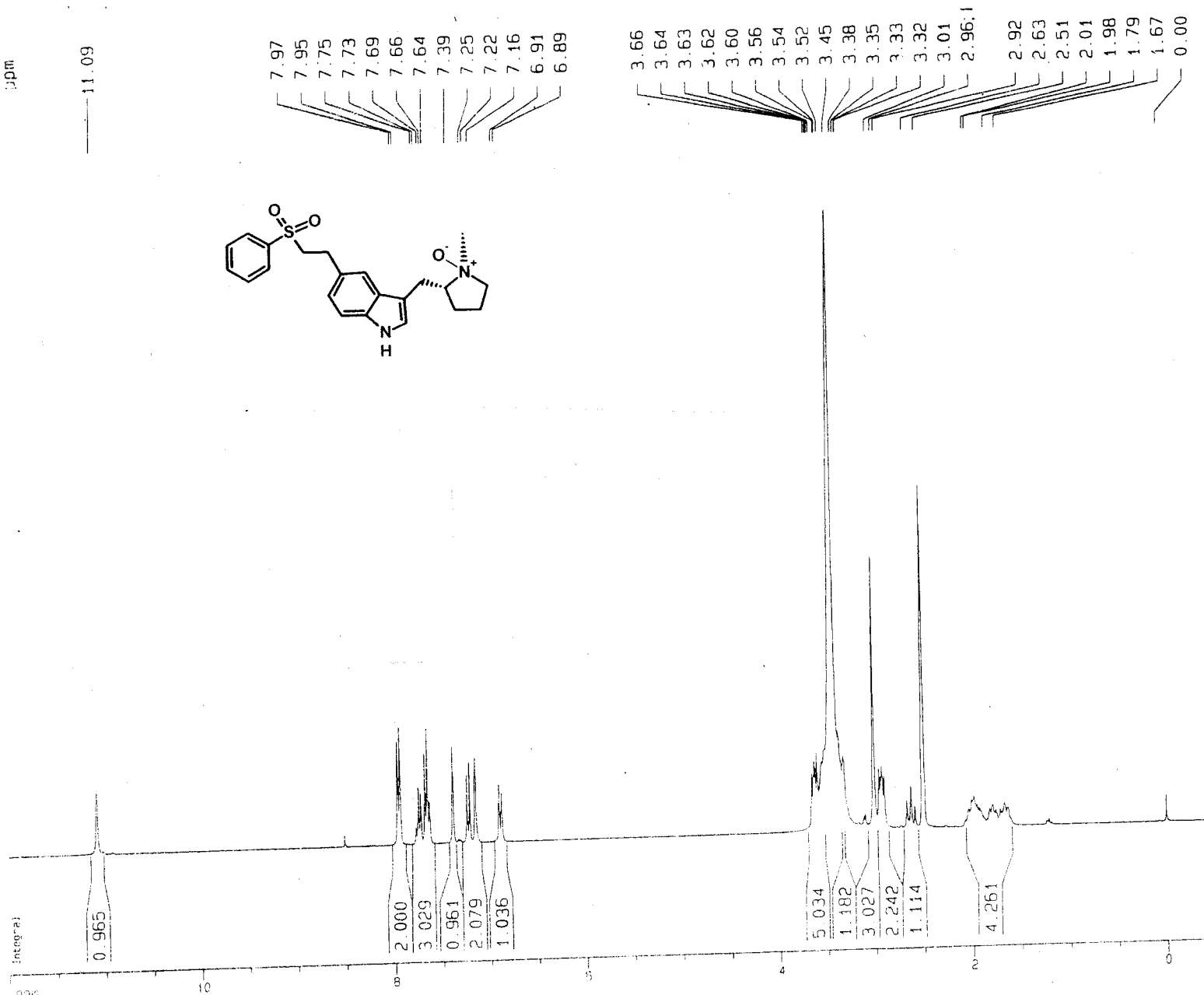
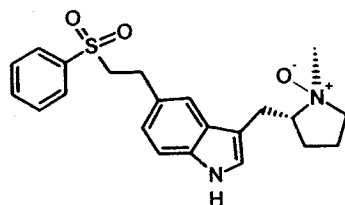
Current Data Parameters
NAME chp-1708-21
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20101215
Time 9.56
INSTRUM dpx300
PROBHD 5 mm BBO BB-
PULPROG zg30
TO 32768
RG 256
SOLVENT DMSO
NS 113
AQ 1.8219508 sec
OW 55.600 usec
OE 6.00 usec
OI 2.00000000 sec
P1 6.75 usec
PL1 -6.00 dB
SF01 300.1315006 MHz
===== CHANNEL f1 =====
NUC1 1H

F2 - Processing parameters
SI 32768
SF 300.1300003 MHz
WOW EM
LB 0.30 Hz
GB 0.

10 NMR plot parameters
CX 20.50 cm
F1P 12.000 ppm
F2P -0.500 ppm



Gmpt

SAMPLE: chp-1761-14

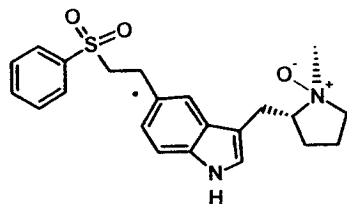
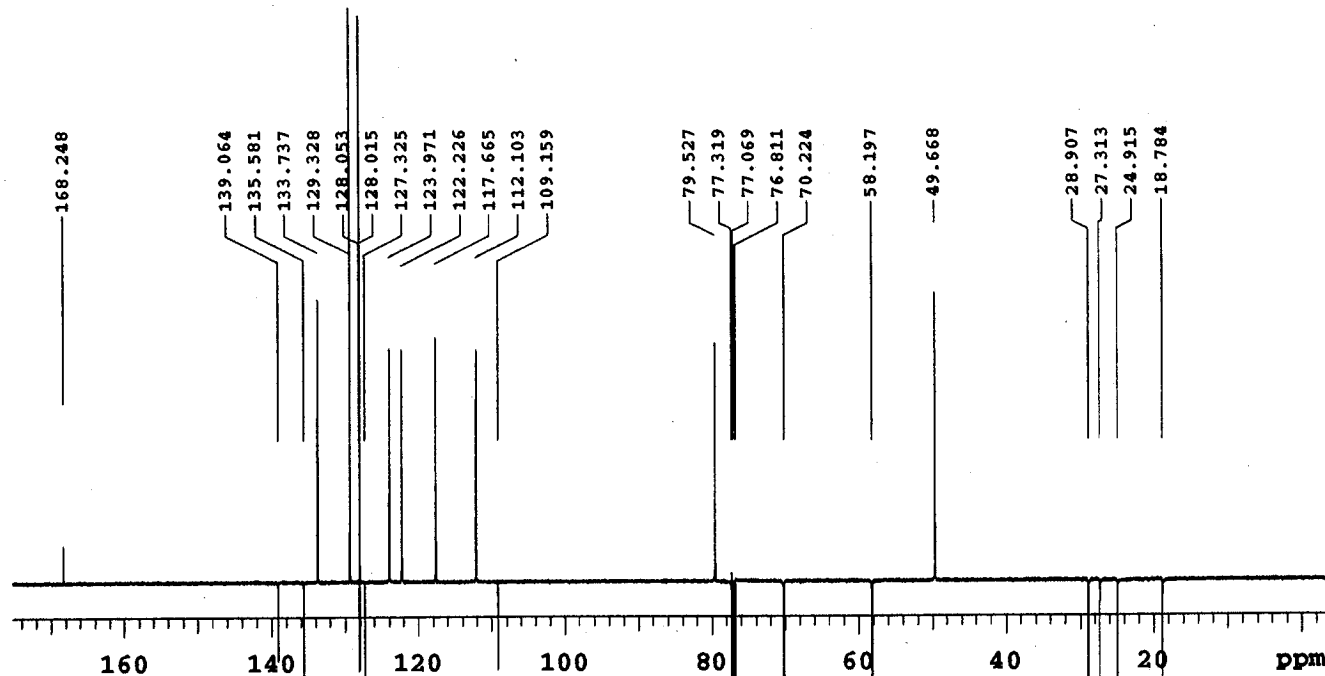
Eletriptan N-oxide-I
chp-1761-14

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: apl
File: DEPT_01
VNMR5-500 "APLV500"

PULSE SEQUENCE: DEPT
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.022 sec
Width 31250.0 Hz
8000 repetitions

OBSERVE C13, 125.6793948
DECOUPLE H1, 499.8207327
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated

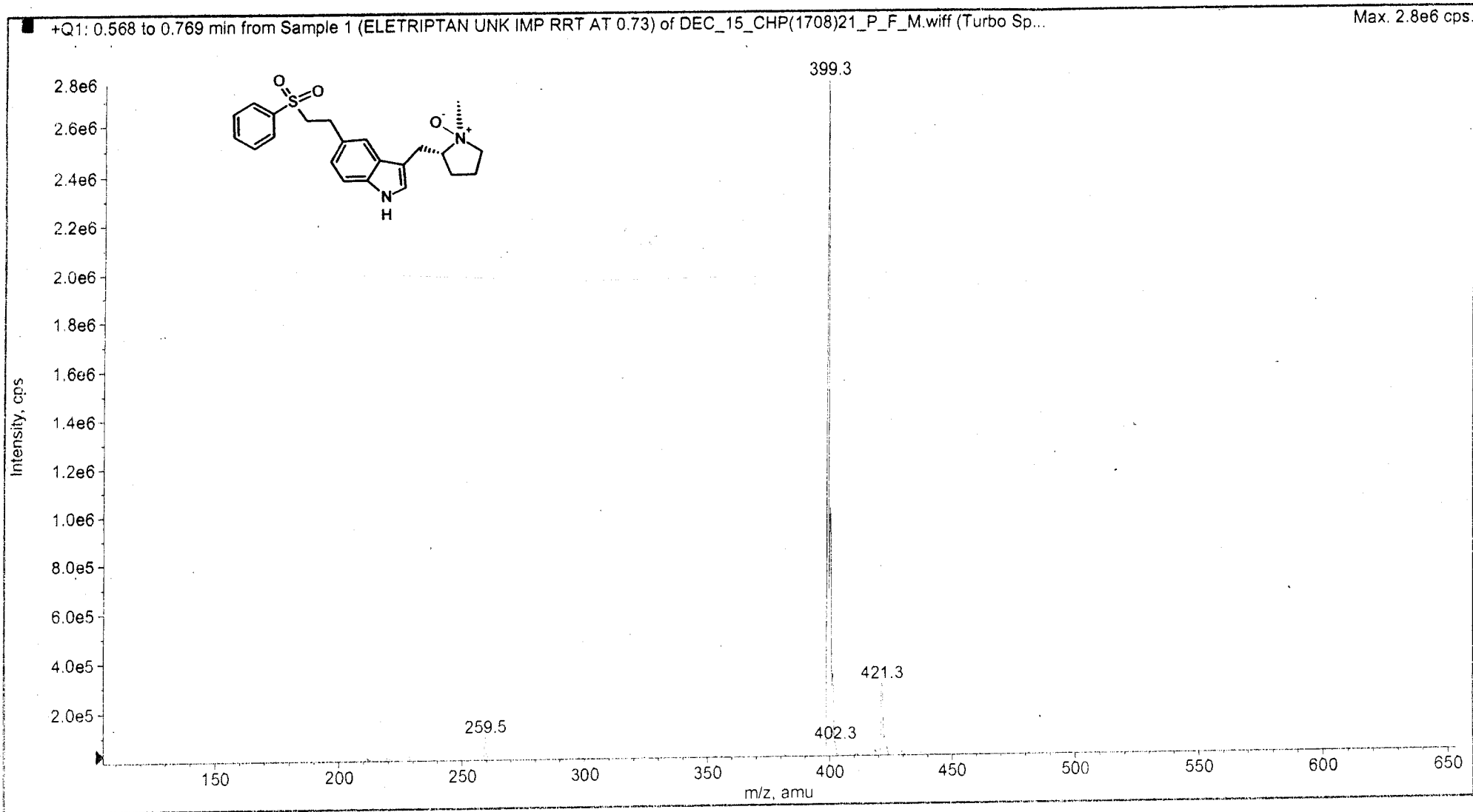
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 4.5 hours



Analyst Version: 1.4.2
Acq. Time: 11:23
Acq. Date: Wednesday, December 15, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: B NO:CHP(1708)21
Sample Name: ELETRIPTAN UNK IMP RRT AT
Acq. File: DEC_15_CHP(1708)21_P_F_M.wiff



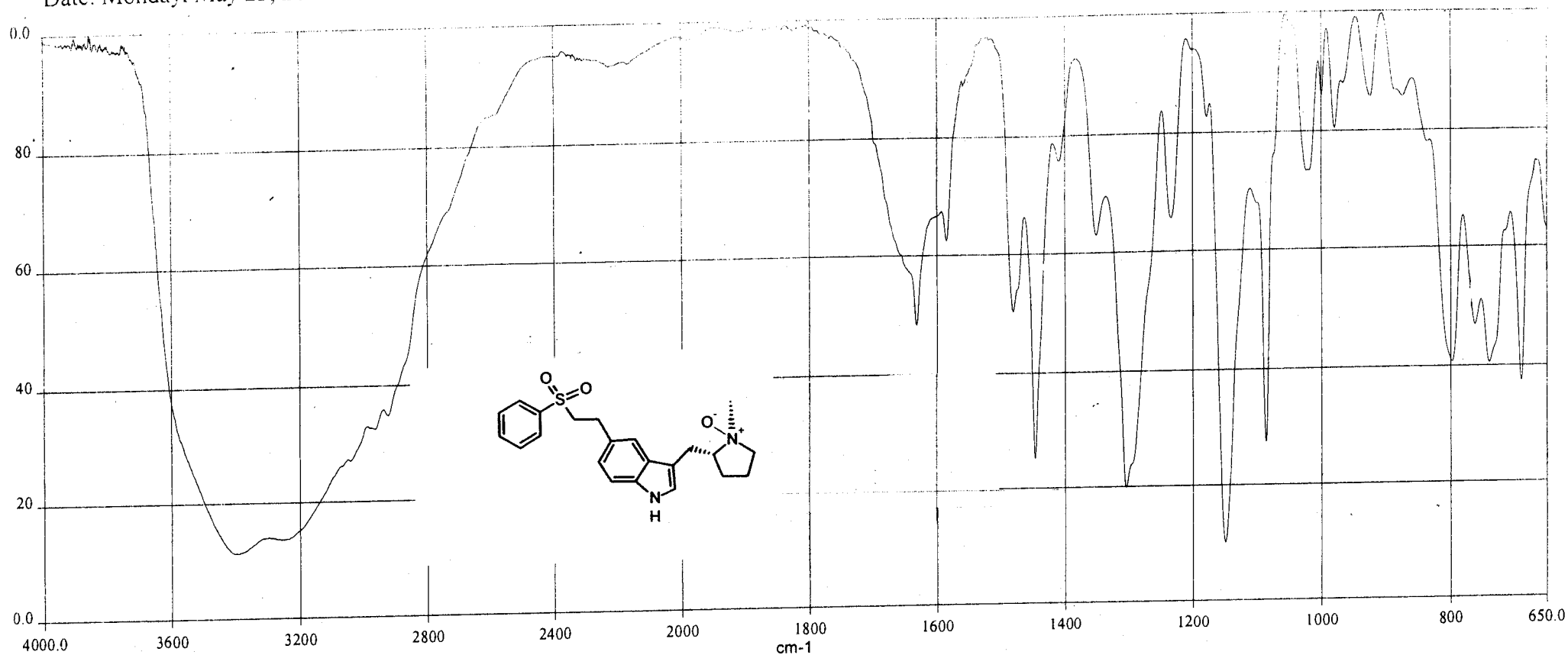
Period/Expt: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

[Signature]
Analyst

Date: Monday, May 23, 2011

APL RESEARCH CENTRE

ARE_018



Status

Filename: ELETRIPTAN HBr N-OXIDE-1-CHP(1708)21.002
Date Created: Monday, May 23, 2011 10:48 AM India Standard Time
Analyst: Administrator
Description: B.NO.: CHP(1708)21
Comments:
SAMPLE: ELETRIPTAN HBr N-OXIDE-1
PROJECT: ELETRIPTAN HBr
1.91 mg OF SAMPLE IN 403 mg OF KBr

Abscissa: (cm-1)
Start: 4000.00
End: 450.00
Interval: -1.000000

Ordinate: (%T)
Maximum: 100.00
Minimum: 10.00
Points: 3551

Instrument Model: Spectrum One

ELETRIPTAN HBr N-OXIDE-1-CHP(1708)21.pk

ELETRIPTAN HBr N-OXIDE-1-CHP(1708)21.002 3351 4000.00 650.00 10.00 100.00 4.00 %T 8 1.00
B.NO.: CHP(1708)21
REF 4000 99.08 2000 97.49 600
3910.67 97.65 3874.71 97.59 3864.39 97.84 3846.49 97.36 3826.59 97.31
3810.96 97.20 3758.36 96.62 3391.62 11.18 2923.69 34.76 2229.39 92.87
1632.09 48.36 1584.60 62.45 1480.47 50.35 1446.29 25.08 1409.28 75.78
1350.72 63.08 1303.71 19.79 1233.41 65.71 1177.79 82.71 1148.88 9.99
1085.84 27.21 1022.81 73.43 999.03 85.91 979.31 80.47 924.64 85.70
873.89 85.73 797.32 40.76 761.80 46.85 739.45 40.53 689.55 37.28

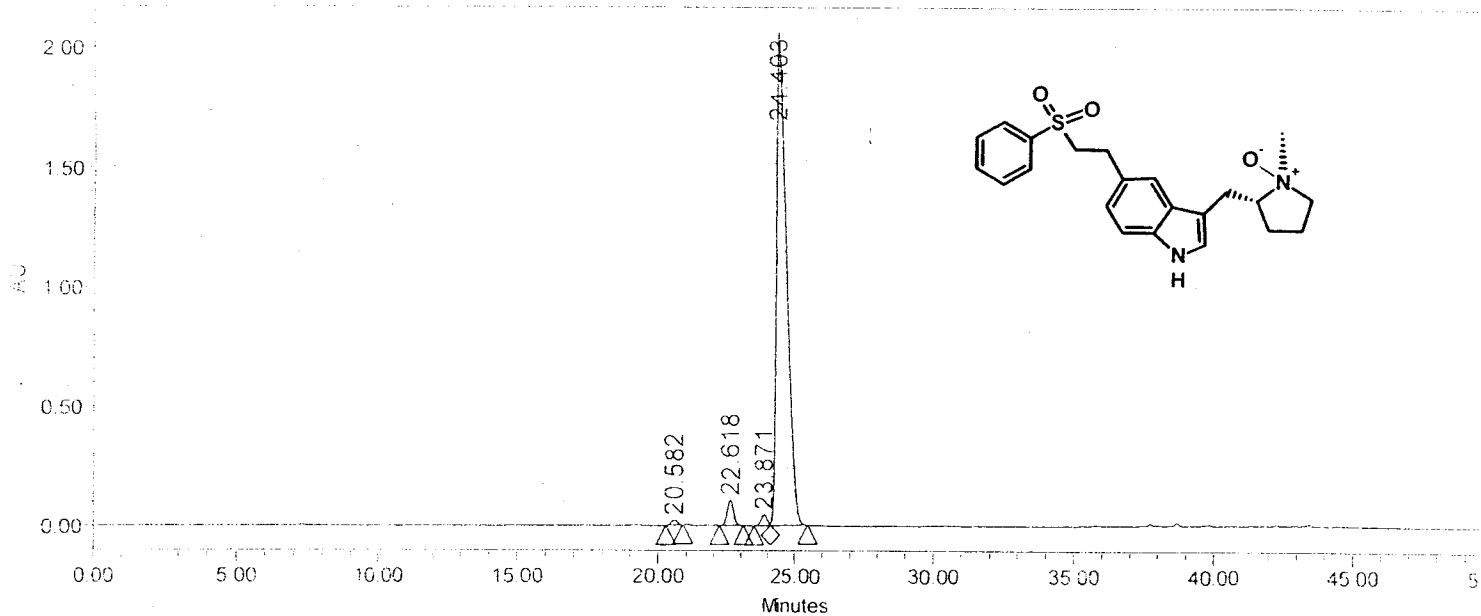
23/05/2011

APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012\RCII_AE088

Sample ID	CHP(1708)21/Eletriptan -N-Oxide-1	Proc. Chnl. Descr :	W2996 PDA 225.0 nm
Run Time	60.0 Minutes	Date Acquired :	3/29/2012 8:07:02 PM IST
Vial	31	Acq. Method Set :	Eletriptan_RS_MET
Injection	1	Date Processed :	3/30/2012 5:16:09 PM IST
Injection Volume :	20.00 ul	Processing Method :	Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu\text{V}\cdot\text{sec}$)	% Area	RT Ratio	Name
1	20.58	329734	0.58	0.84	Peak6
2	22.62	1653197	2.93	0.93	Peak7
3	23.87	741458	1.32	0.98	Peak8
4	24.40	53651457	95.17	1.00	Eletriptan-N-Oxide-1

SAMPLE: chp-1708-19

Eletriptan N-Oxide-II
chp-1708-19

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: apl
File: PROTON_01
VNMRS-500 "APLV500"

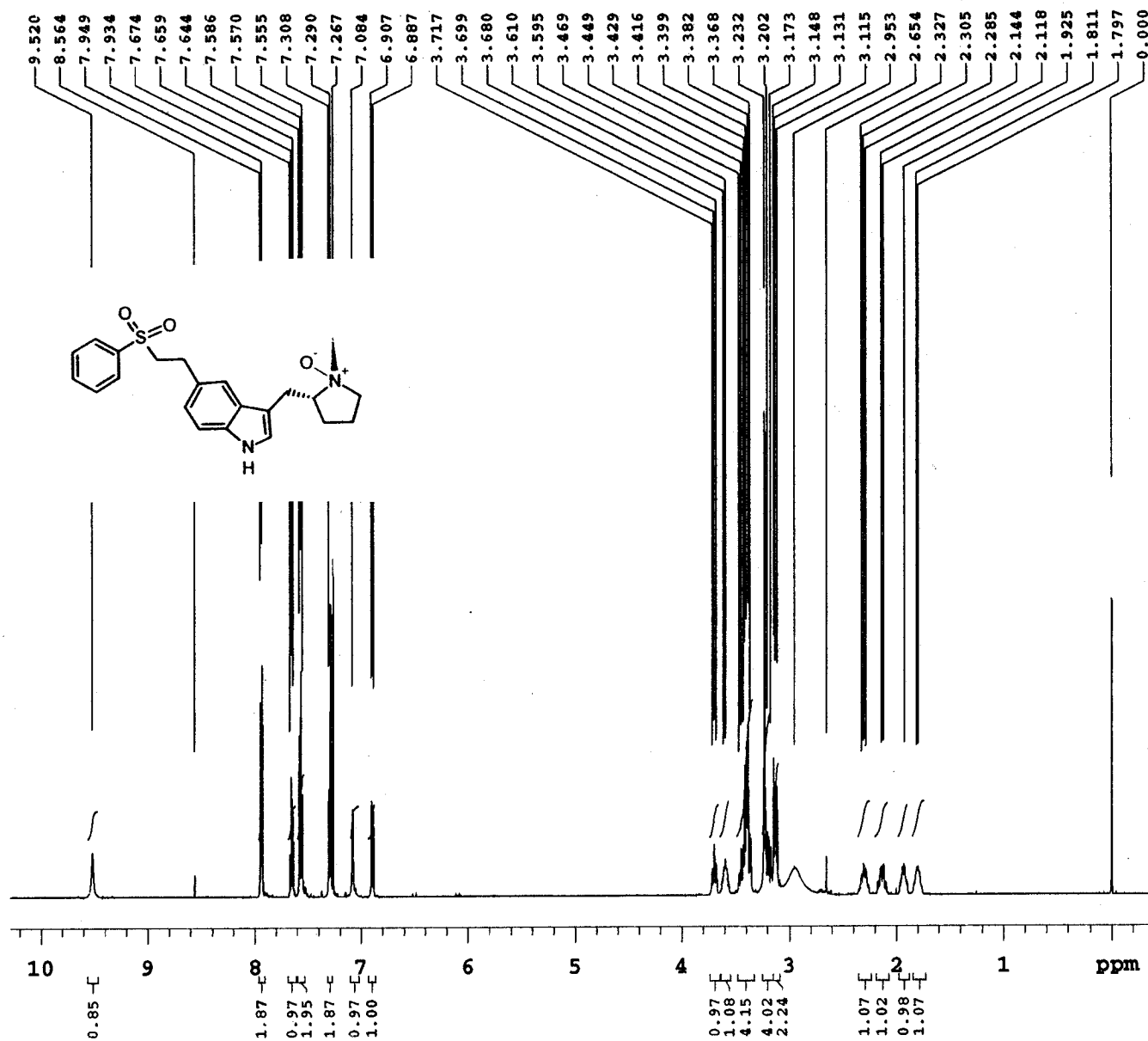
PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 9058.0 Hz
64 repetitions

OBSERVE H1, 499.8182339

DATA PROCESSING

FT size 32768
Total time 3 minutes



SAMPLE: chp-1761-25

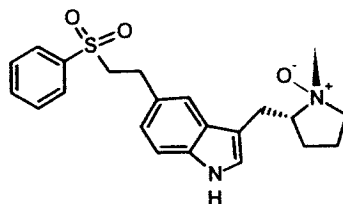
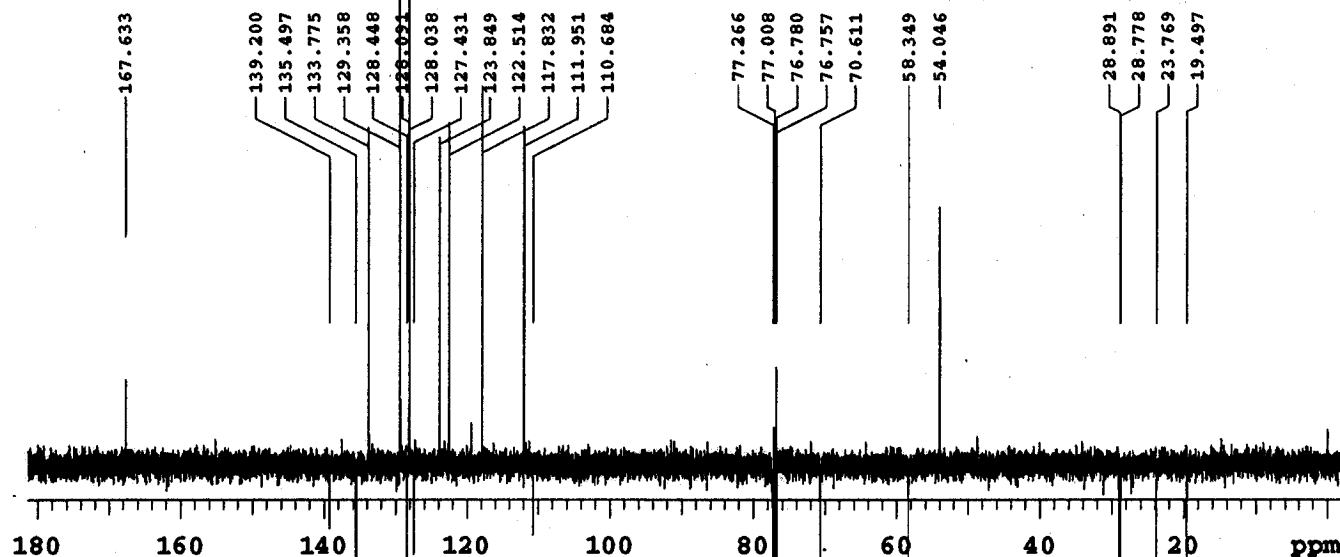
Eletriptan N-oxide-II
chp-1761-25

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: apl
File: DEPT_01
VNMR5-500 "APLV500"

PULSE SEQUENCE: DEPT
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.022 sec
Width 31250.0 Hz
3264 repetitions

OBSERVE C13, 125.6793948
DECOUPLE H1, 499.8207327
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated

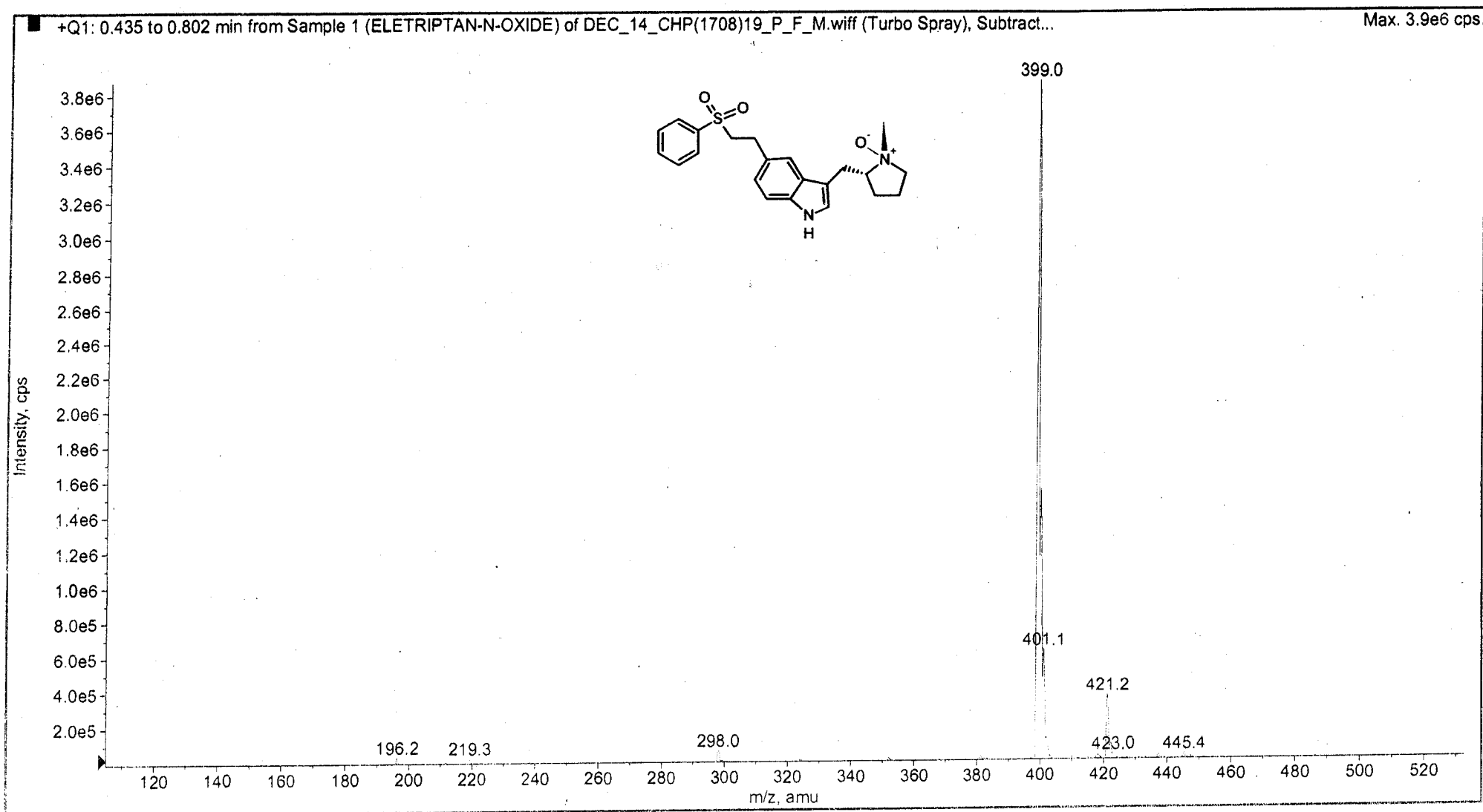
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 110 minutes



Analyst Version: 1.4.2
Acq. Time: 10:26
Acq. Date: Tuesday, December 14, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: B.NO:CHP(1708)19
Sample Name: ELETRIPTAN-N-OXIDE
Acq. File: DEC_14_CHP(1708)19_P_F_M.wiff



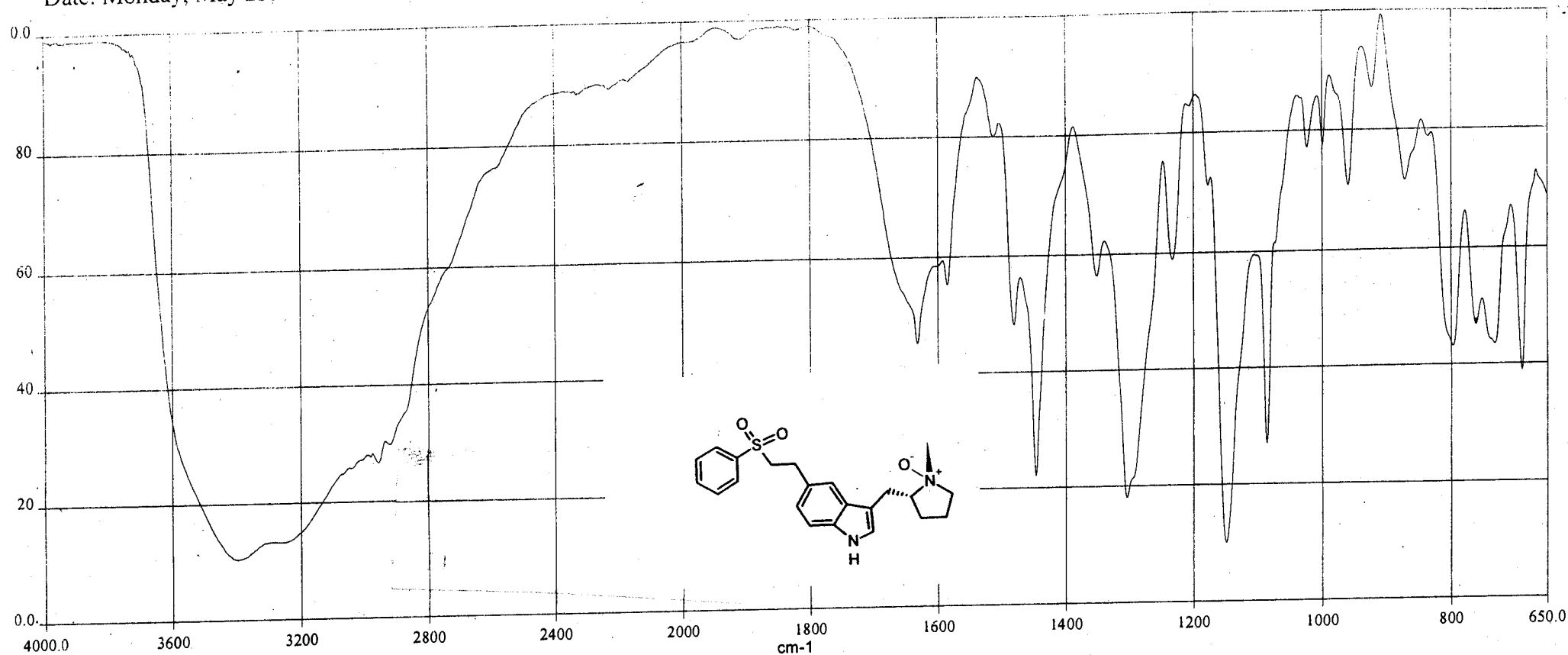
Period/Expt.: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

K.P. Pan
Analyst

Date: Monday, May 23, 2011

APL RESEARCH CENTRE

ARE_018



Status

Filename: ELETRIPTAN N-OXIDE-2-CHP(1708)19.002
Date Created: Monday, May 23, 2011 11:15 AM India Standard Time
Analyst: Administrator
Description: B.NO.: CHP(1708)19
Comments:
SAMPLE: ELETRIPTAN N-OXIDE-2
PROJECT: ELETRIPTAN HBr
1.73 mg OF SAMPLE IN 388 mg OF KBr

Abscissa: (cm-1)
Start: 4000.00
End: 450.00
Interval: -1.000000

Ordinate: (%T)
Maximum: 100.00
Minimum: 10.00
Points: 3551

Instrument Model: Spectrum One

ELETRIPTAN N-OXIDE-2-CHP(1708)19.pk

ELETRIPTAN N-OXIDE-2-CHP(1708)19.002 3351 4000.00 650.00 10.00 99.23 4.00 %T 8 1.00
B.NO.: CHP(1708)19
REF 4000 99.12 2000 96.68 600
3400.65 10.42 2957.91 26.76 1910.97 97.05 1631.91 45.10 1584.52 55.18
1512.64 80.05 1480.71 48.01 1446.58 22.05 1351.07 56.32 1303.73 17.87
1232.22 58.94 1176.97 71.23 1148.74 9.98 1085.73 27.12 1022.98 77.17
998.80 77.55 959.58 70.76 922.45 87.02 871.23 71.49 796.65 43.13
760.98 46.69 731.55 43.44 689.35 38.85

23/05/2011

APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012\RCII_AE088

Sample ID : CHP(1708)19/Eletriptan -N-Oxide-2

Proc. Chnl. Descr : W2996 PDA 225.0 nm

Run Time : 60.0 Minutes

Date Acquired : 3/29/2012 9:08:05 PM IST

Vial : 32

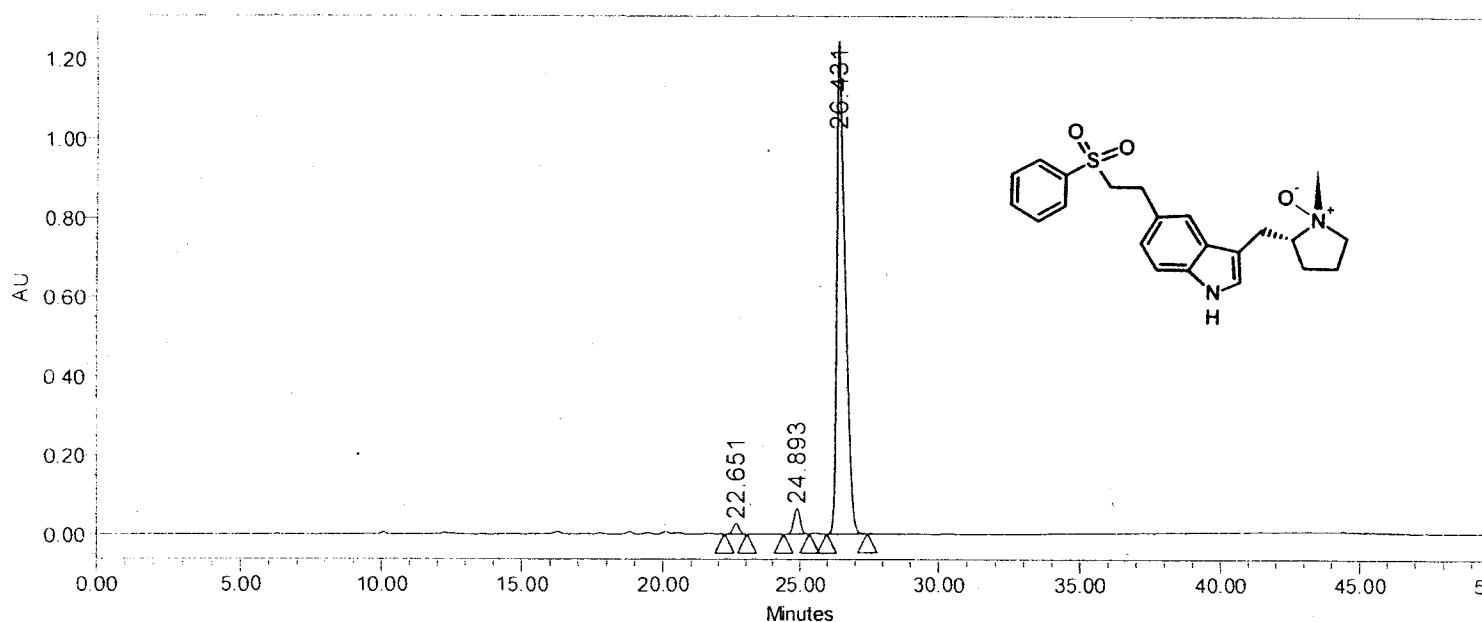
Acq. Method Set : Eletriptan_RS_MET

Injection : 1

Date Processed : 3/30/2012 5:17:07 PM IST

Injection Volume : 20.00 ul

Processing Method : Eletriptan_RS_Pro



Peak Results

	RT (min)	Area (μV*sec)	% Area	RT Ratio	Name
1	22.65	417966	1.52	0.86	Peak7
2	24.89	1066365	3.87	0.94	Peak8
3	26.43	26087242	94.62	1.00	Eletriptan-N-Oxide-2

ELETRIPTAN UNK IMP ART-0.87 CHP (1580) 137 a.r.no: s-010041
 (ETHYL INDOLE PYRROLIDINE IMPURITY) CDC13

APL-RESEARCH CENTRE

Current Data Parameters

NAME chp-1580-137

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20100514

Time 10.44

INSTRUM dpx300

PROBHD 5 mm BBO BB-

PULPROG zg30

TD 32768

RG 362

SOLVENT CDC13

NS 16

AQ 1.8219508 sec

DW 55.600 usec

DE 6.00 usec

DI 2.00000000 sec

P1 6.75 usec

PL1 -6.00 dB

SF01 300.1315006 MHz

----- CHANNEL f1 -----

NUC1 1H

F2 - Processing parameters

SI 32768

SF 300.1300070 MHz

WDW EM

LB 0.30 Hz

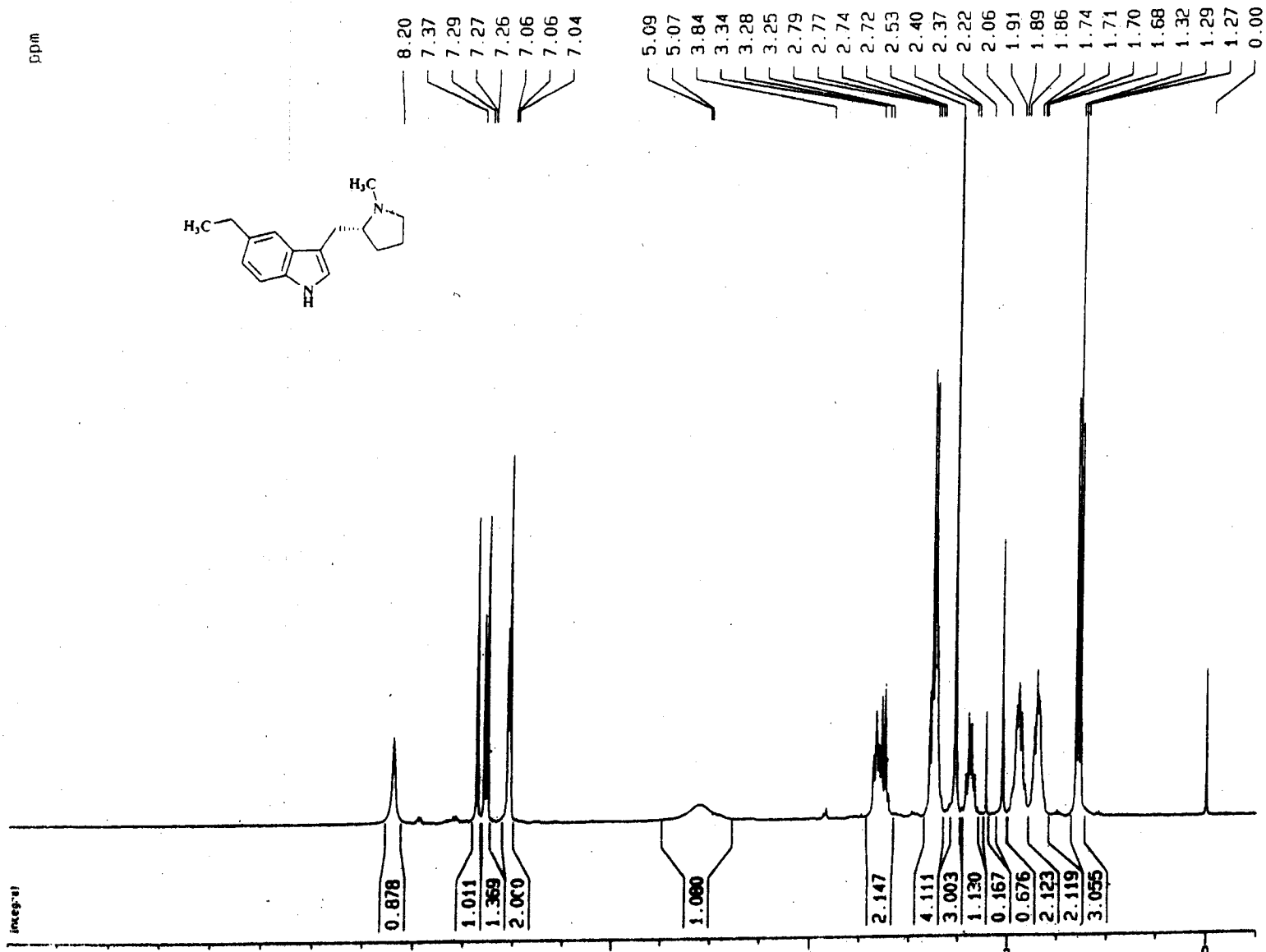
GB 0

1D NMR plot parameters

CX 20.50 cm

F1P 12.000 ppm

F2P -0.500 ppm



SAMPLE: msk-1679-140

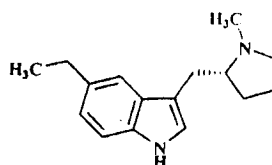
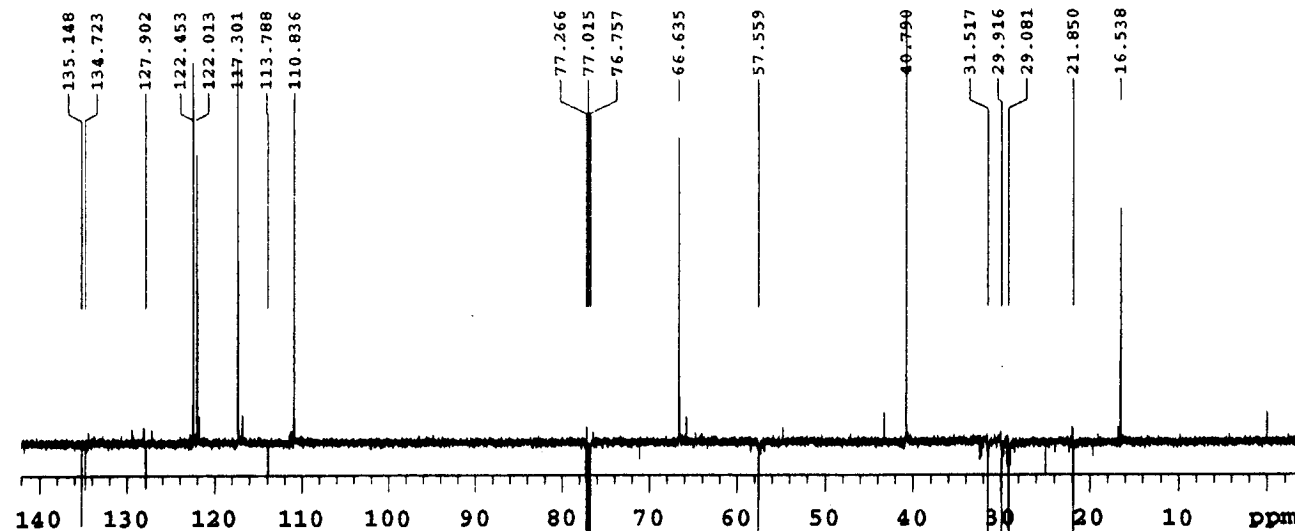
Ethyl Indole pyrrolidine imp
msk-1679-140

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: apl
File: DEPT_01
VNMRS-500 "APLV500"

PULSE SEQUENCE: DEPT
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.022 sec
Width 31250.0 Hz
9000 repetitions

OBSERVE C13, 125.6793948
DECOUPLE H1, 499.8207327
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 5.1 hours

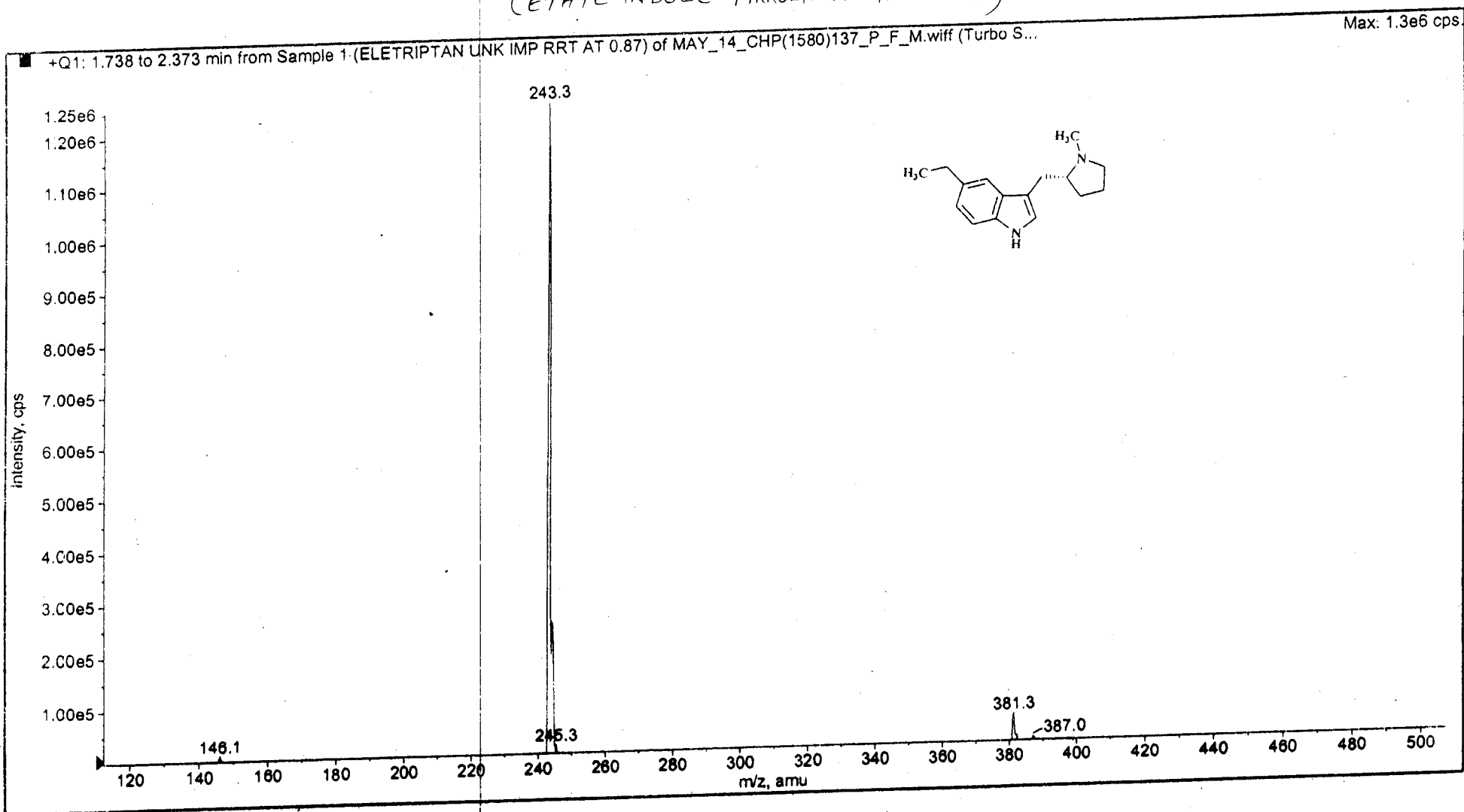


Analyst Version: 1.4.2
Acq. Time: 09:24
Acq. Date: Friday, May 14, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: BNO.:CHP(1580)137
Sample Name: ELETRIPTAN UNK IMP RRT AT
Acq. File: MAY_14_CHP(1580)137_P_F_M.wiff

(ETHYL INDOLE PYRROLIDINE IMPURITY)



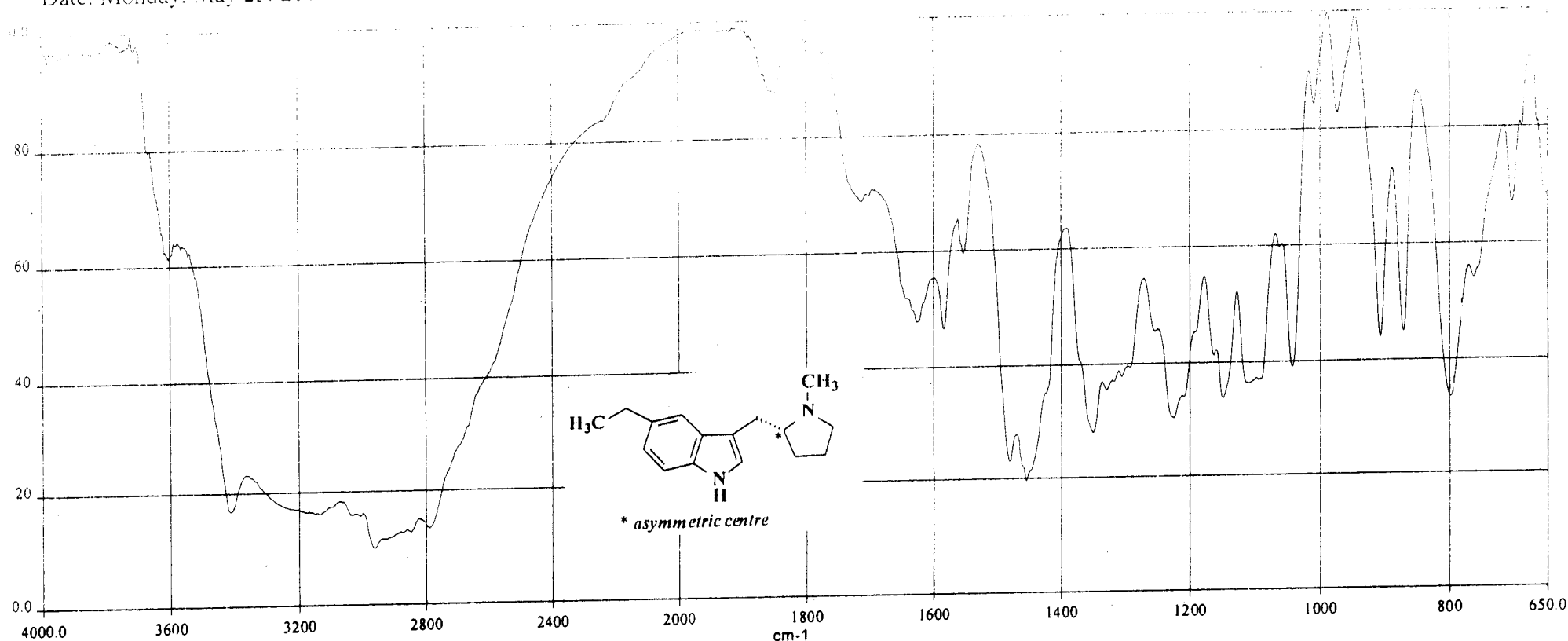
Period/Expt.: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

QC

Date: Monday, May 23, 2011

APL RESEARCH CENTRE

ARE_018



Status (ETHYL INDOLE PYRROLIDINE IMPURITY)

Filename: ETHYL INDOLE PROLINE-MSK(1679)140.002
Date Created: Monday, May 23, 2011 10:07 AM India Standard Time
Analyst: Administrator
Description: B.NO.: MSK(1679)140
Comments:
SAMPLE: ETHYL INDOLE PROLINE
PROJECT: ELETRIPTAN HBr
NEAT BG

Abcissa: (cm-1)
Start: 4000.00
End: 490.00
Interval: -1.000000

Ordinate: (%T)
Maximum: 100.00
Minimum: 10.00
Points: 3511

Instrument Model: Spectrum One
Instrument Serial Number: 53009

ETHYL INDOLE PROLINE-MSK(1679)140.pk

ETHYL INDOLE PROLINE-MSK(1679)140.002 3351 4000.00 650.00 10.00 99.70 4.00 %T 8 1.00
B.NO.: MSK(1679)140
REF 4000 99.31 2000 97.99 600
3981.16 95.31 3948.26 95.95 3751.20 97.03 3710.32 96.78 3604.33 61.33
3411.30 16.76 3134.92 16.05 2963.40 9.99 2790.47 13.41 1850.00 87.61
1712.77 68.76 1625.98 47.88 1583.58 46.59 1552.04 59.42 1480.82 22.94
1454.98 19.52 1350.20 28.06 1329.68 35.64 1224.45 30.45 1148.67 33.84
1110.97 36.37 1042.31 38.97 1009.36 84.14 973.21 82.70 906.43 43.94
870.78 44.90 798.24 33.59 762.14 54.00 703.99 66.91

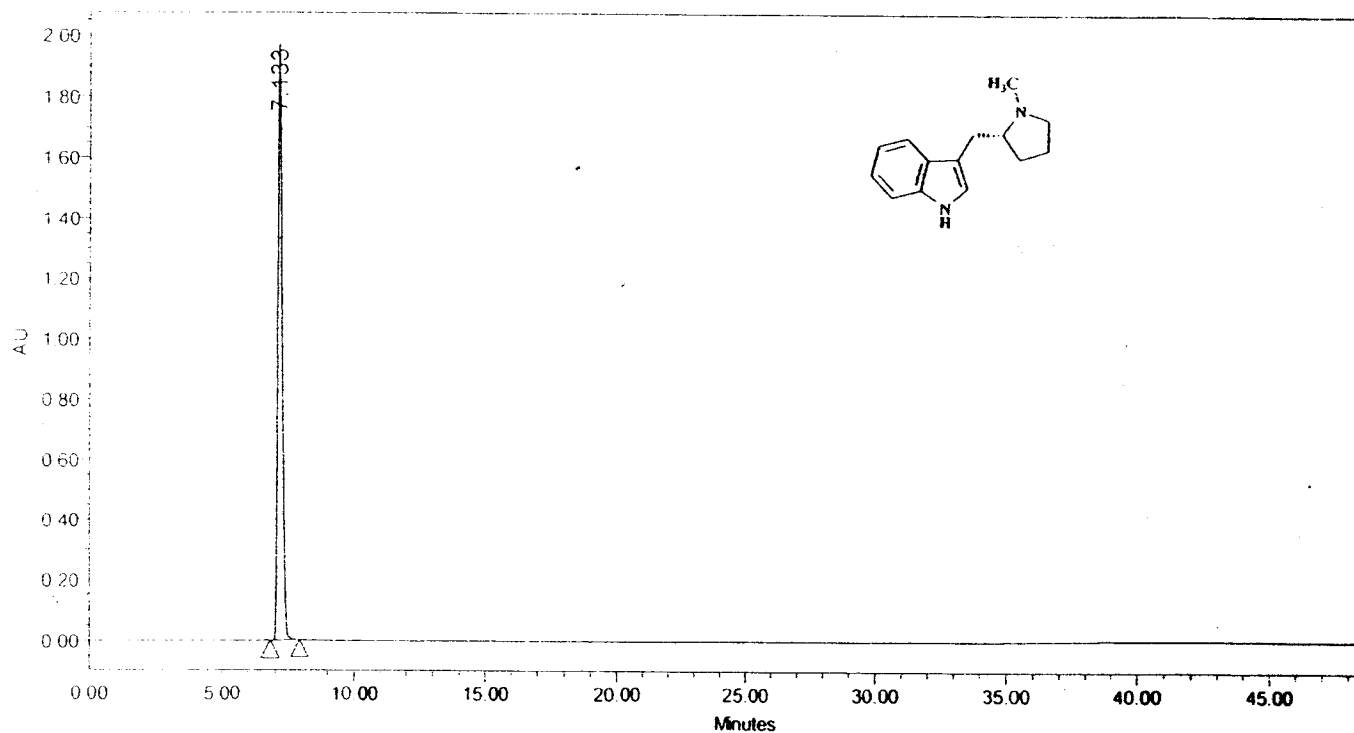
23/05/2011

APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012\RCII_AE088

Sample ID	ELE(1130)42//Desbromo indole pyrrolidine imp	Proc. Chnl. Descr :	W2996 PDA 225.0 nm
Run Time	60.0 Minutes	Date Acquired :	3/29/2012 6:04:57 PM IST
Vial	29	Acq. Method Set :	Eletriptan_RS_MET
Injection	1	Date Processed :	3/30/2012 9:33:03 AM IST
Injection Volume :	20.00 ul	Processing Method :	Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu V \cdot sec$)	% Area	Name
1	7.13	23663578	100.00	Desbromo indole pyrrolidine imp

DESBROMO BIP ELE (1130) 42 A.R.NO: S-010067

(DESBROMO INDOLE PYRROLIDINE IMPURITY) CDCl₃

APL-RESEARCH CENTRE

Current Data Parameters
NAME ele-1130-42

PROCNO 1

F2 - Acquisition Parameters

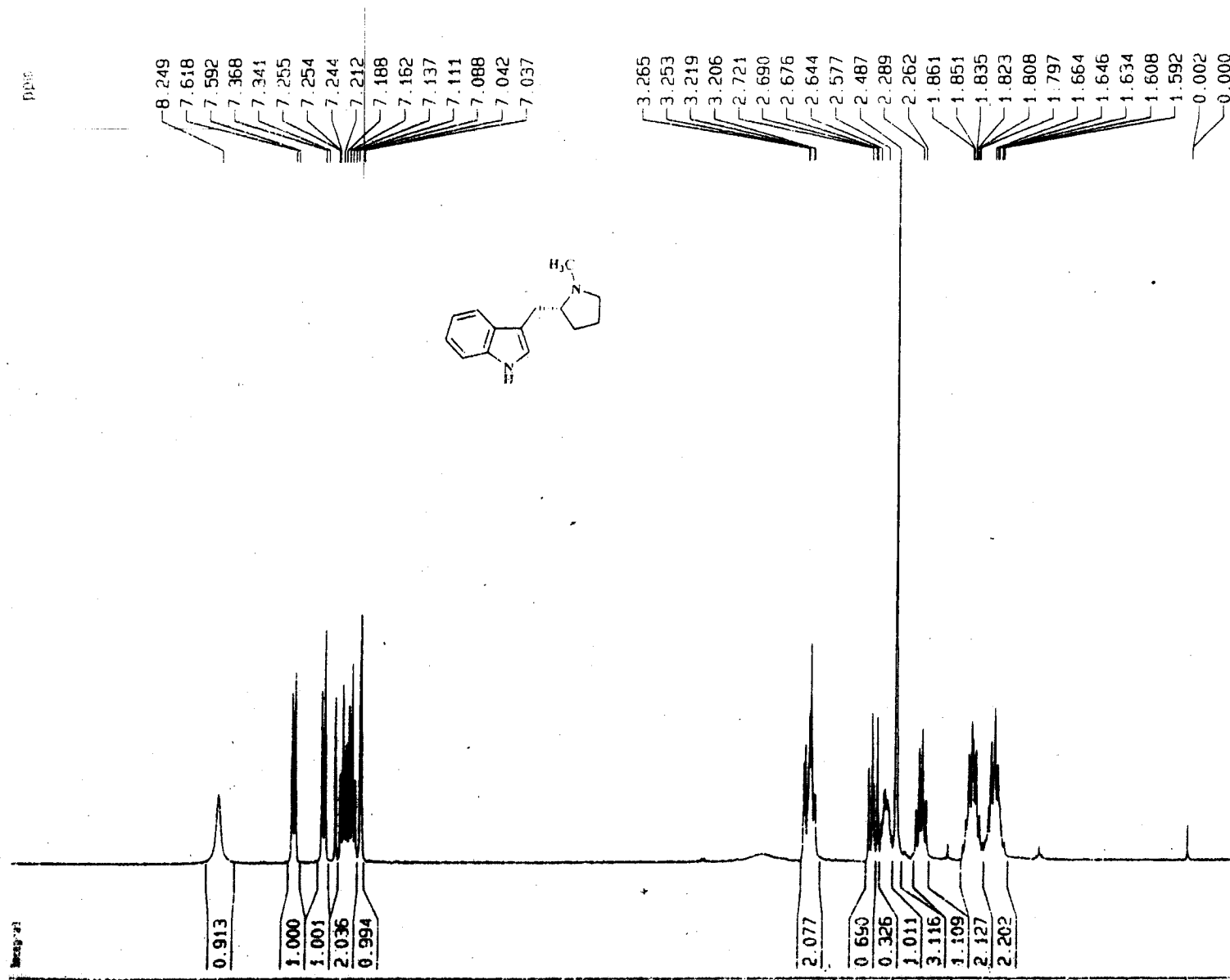
Date_ 20100621
Time 16.49
INSTRUM dpx300
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 32768
RG 362
SOLVENT CDCl₃
NS 16
AQ 1.8219508 sec
DQ 55.600 usec
DE 6.00 usec
D1 2.00000000 sec
P1 6.75 usec
PL1 -6.00 dB
SFO1 300.1315006 MHz
----- CHANNEL f1 -----
NUC1 1H

F2 - Processing parameters

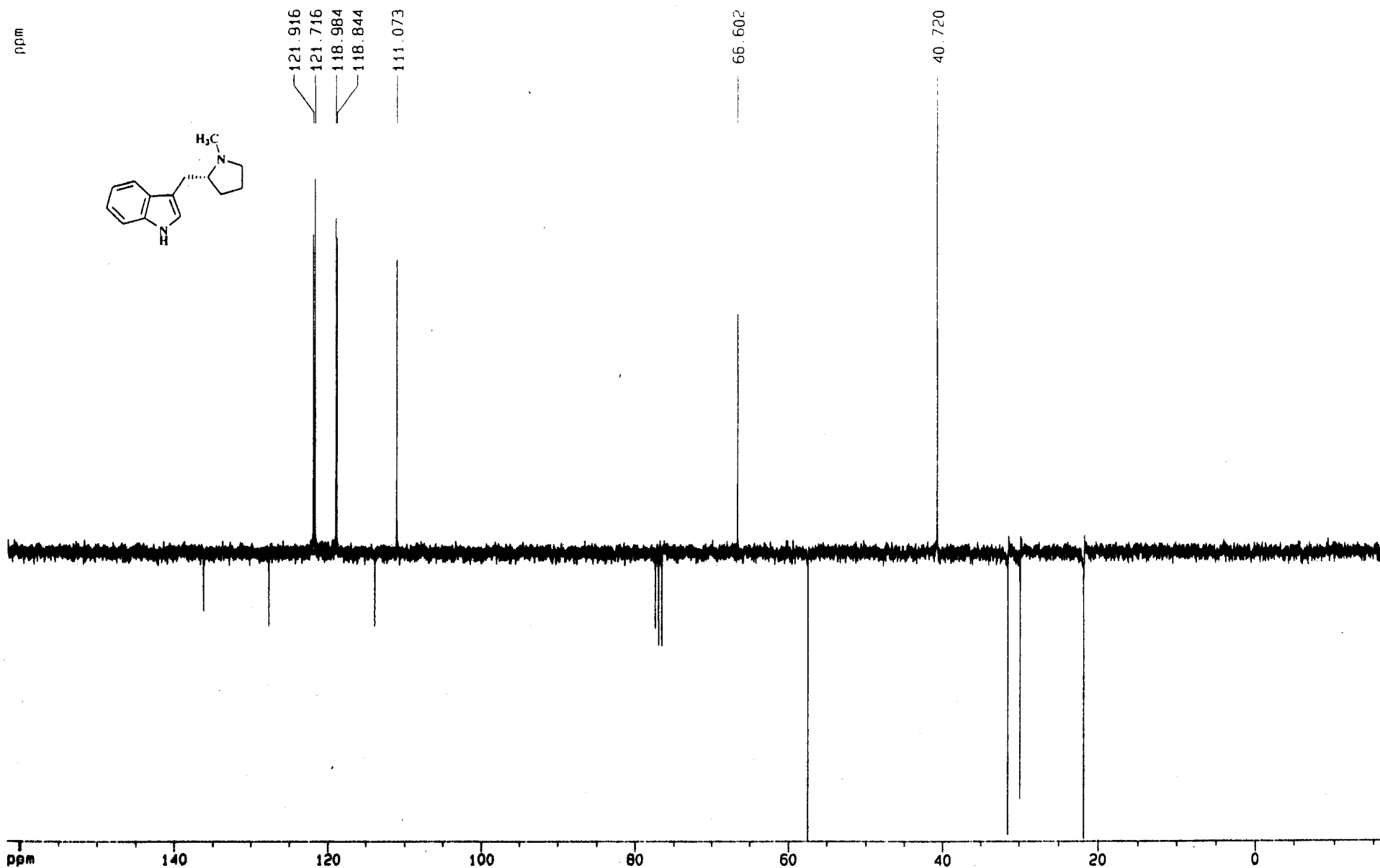
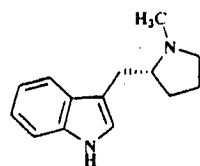
SI 32768
SF 300.1300083 MHz
WDW EM
LB 0.30 Hz
GB 0

1D NMR plot parameters

CX 20.50 cm
F1P 10.000 ppm
F2P -0.500 ppm



(DES BROMO INDOLE PYRROLIDINE IMPURITY)
PENDANT-NMR of DESBROMO INDOLE PROLINE ELE (1130) 42
CDC13

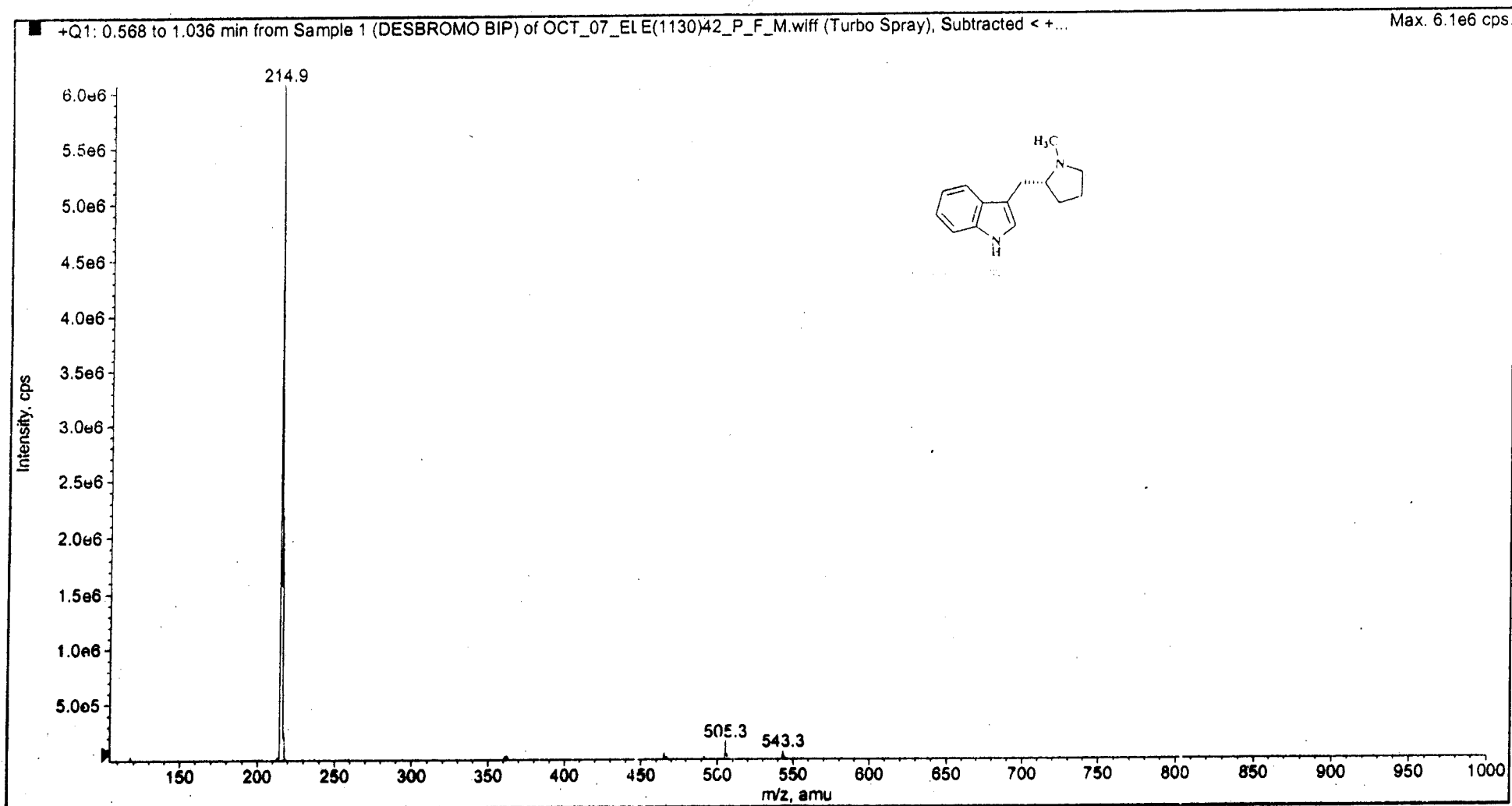


Analyst Version: 1.4.2
Acq. Time: 10:29
Acq. Date: Thursday, October 07, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: B.NO:ELE(1130)42
Sample Name: DESBROMO BIP
Acq. File: OCT_07_ELE(1130)42_P_F_M.wiff

(DES BROMO INDOLE PYRROLIDINE IMPURITY)



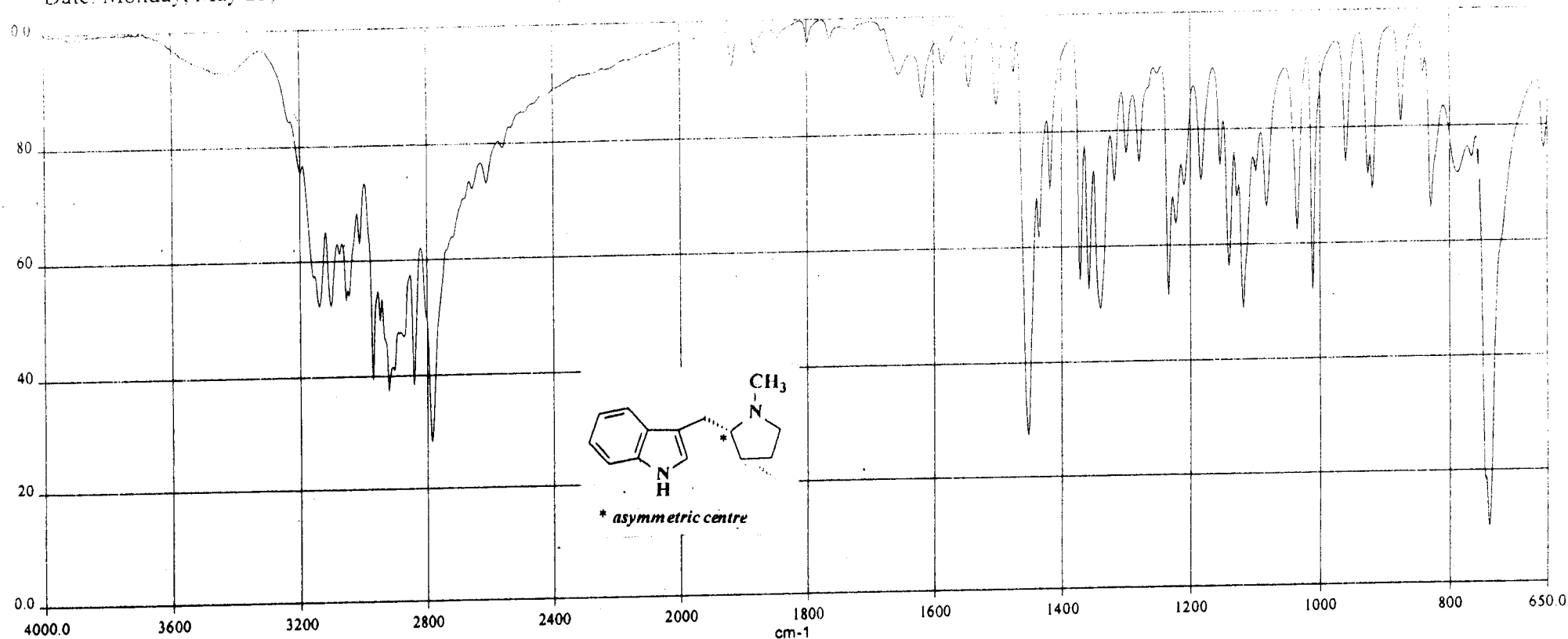
Period/Expt.: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

K.A. Brown
Analyst

Date: Monday, May 23, 2011

APL RESEARCH CENTRE

ARE_018



Status

(DES BROMO INDOLE PYRROLIDINE IMPURITY)

Filename: DESBROMO ANALOG OF ELETRIPTAN-ELE(1130)42.002
Date Created: Monday, May 23, 2011 11:27 AM India Standard Time
Analyst: Administrator
Description: B.NO.: ELE(1130)42
Comments:
SAMPLE: DESBROMO ANALOG OF ELETRIPTAN
PROJECT: ELETRIPTAN HBr
1.86 mg OF SAMPLE IN 397 mg OF KBr

Abcissa: (cm-1)

Start: 4000.00

End: 450.00

Interval: -1.000000

Ordinate: (%T)

Maximum: 100.00

Minimum: 10.00

Points: 3551

DESBROMO ANALOG OF ELETRIPTAN-ELE(1130)42.pk

DESBROMO ANALOG OF ELETRIPTAN-ELE(1130)42.002 3351 4000.00 650.00 10.00 99.90 4.00 %
B.NO.: ELE(1130)42

REF 4000 99.77 2000 96.53 600

3428.53	92.47	3199.11	75.57	3139.70	52.25	3102.35	52.28	3074.80	61.25
3054.38	53.20	3045.28	53.99	3012.40	63.04	2972.03	39.40	2949.87	49.62
2922.95	37.49	2842.99	38.49	2785.71	28.46	2658.41	72.39	2612.72	73.31
2562.21	79.26	1917.71	92.40	1880.99	94.60	1847.11	96.93	1798.60	96.14
1762.74	96.75	1655.70	90.30	1618.30	86.32	1586.64	92.07	1545.50	88.02
1502.27	84.90	1474.64	90.31	1451.99	27.56	1434.30	61.90	1417.20	70.32
1371.23	54.24	1357.86	52.68	1340.02	49.14	1317.86	71.40	1299.31	76.19
1278.73	74.62	1250.68	89.75	1233.05	51.25	1221.62	63.97	1209.47	70.52
1183.05	71.36	1153.03	73.83	1138.89	56.16	1126.62	68.37	1116.89	48.99
1097.67	72.62	1081.02	66.71	1034.69	62.53	1010.61	52.11	959.44	74.18
925.98	71.90	919.03	69.34	875.71	80.89	841.40	89.33	828.78	66.02
787.66	71.97	765.86	74.59	739.23	9.95	655.30	76.04		

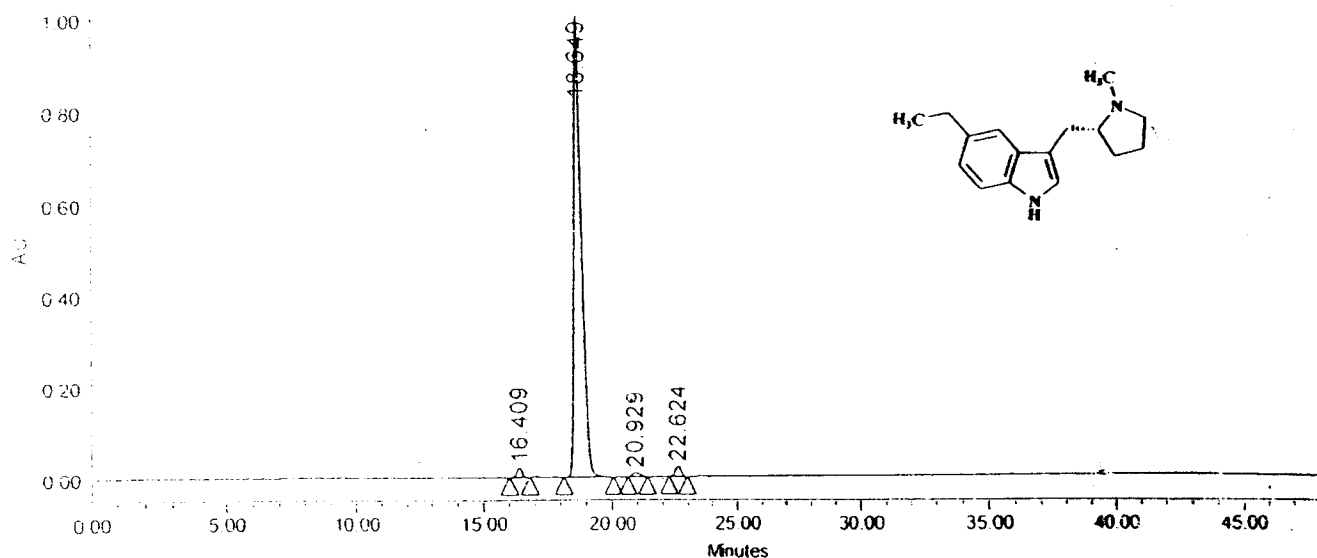
23/05/2011

APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID RCII_AE088

PROJECT NAME MAR_2012RCII_AE088

Sample ID	CHP(1580)137/ Ethyl indole pyrrolidine imp	Proc. Chnl. Descr	W2996 PDA 225.0 nm
Run Time	60.0 Minutes	Date Acquired	3/29/2012 7:05:59 PM IS
Vial	30	Acq Method Set	Eletriptan_RS_MET
Injection	1	Date Processed	3/30/2012 5:15:44 PM IS
Injection Volume	20.00 ul	Processing Method	Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu V \cdot sec$)	% Area	RT Ratio	Name
1	16.41	301273	1.44	0.88	Peak5
2	18.65	20062648	95.88	1.00	Ethyl indole pyrrolidine imp
3	20.93	200745	0.96	1.12	Peak6
4	22.62	360622	1.72	1.21	Peak7

ELETRIPTAN STG-II UNK IMP RT-21.20 KSA (1637) 65 A.R.NO: S-010077
 (ELETRIPTAN METHOXY IMPURITY) DMSO-d6

APL-RESEARCH CENTRE

Current Data Parameters
 NAME ksa-1637-ele
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20100717
 Time 10.33
 INSTRUM dpx300
 PROBHD 5 mm BBO BB-
 PULPROG zg30
 TD 32768
 RG 287.4
 SOLVENT DMSO
 NS 16
 AQ 1.8219508 sec
 DW 55.600 usec
 DE 6.00 usec
 D1 2.0000000 sec
 P1 6.75 usec
 PL1 -6.00 dB
 SF01 300.1315006 MHz

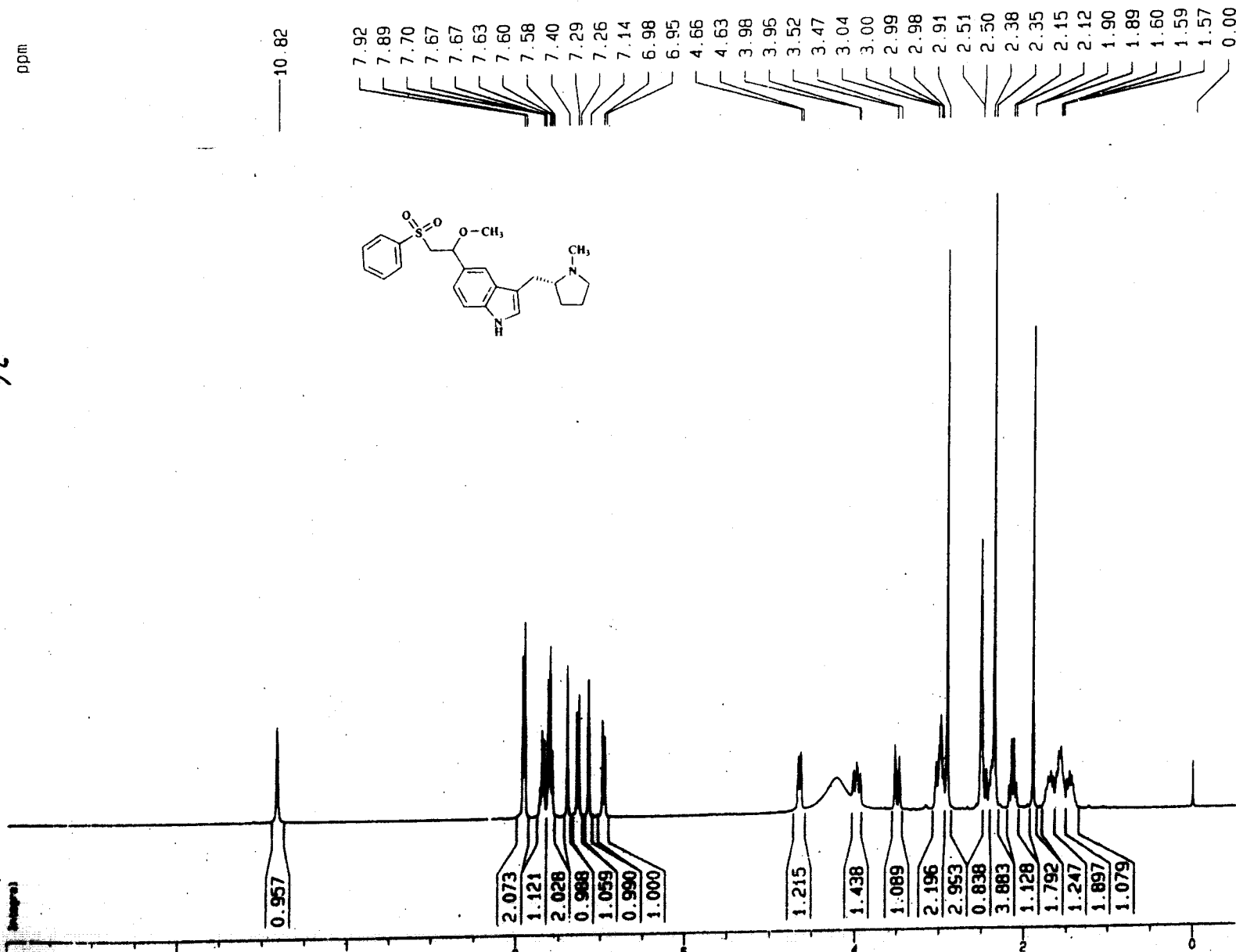
----- CHANNEL f1 -----
 NUC1 1H

F2 - Processing parameters

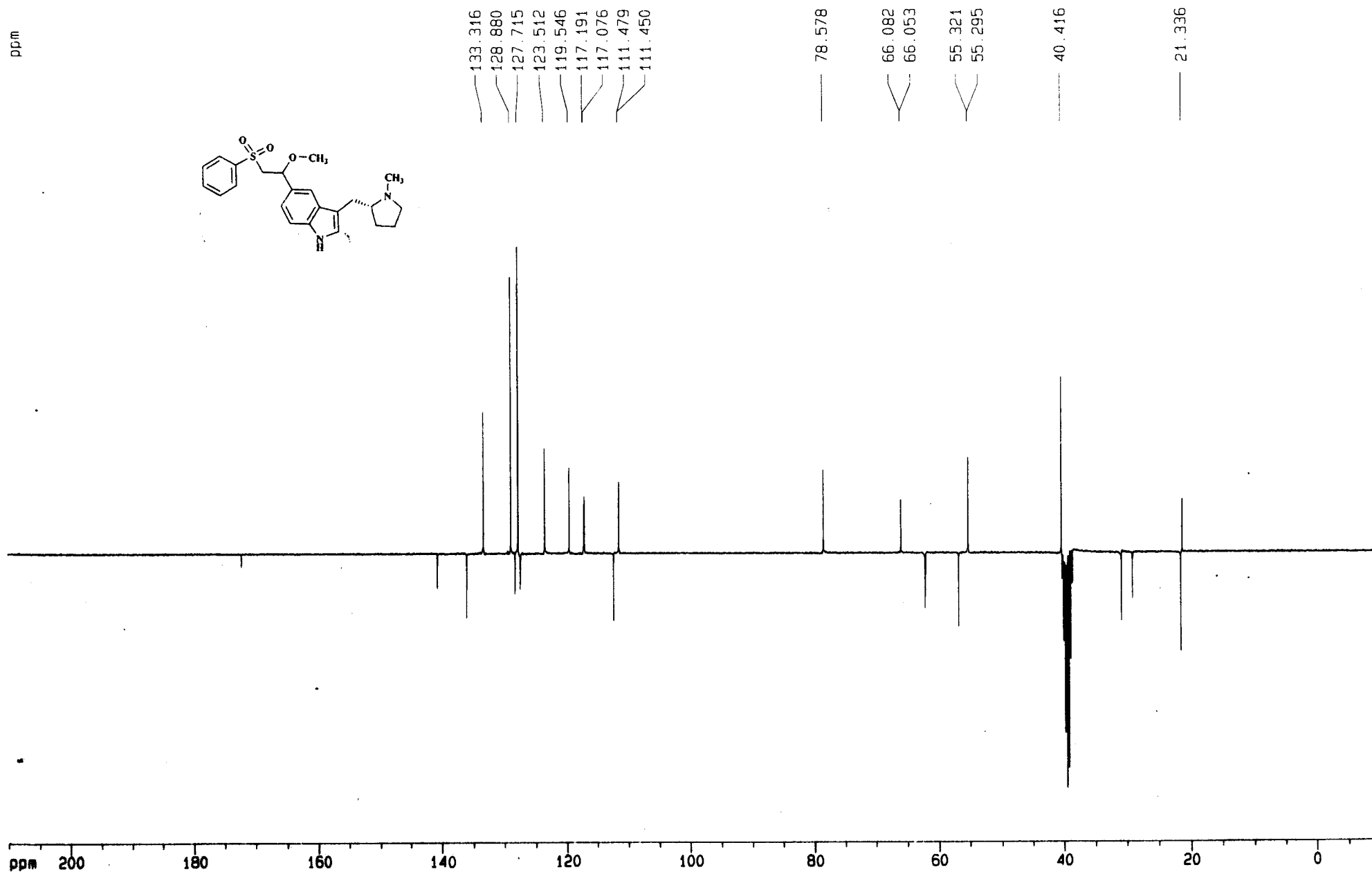
SI 32768
 SF 300.1300004 MHz
 WDW EM
 LB 0.30 Hz
 GB 0

1D NMR plot parameters

CX 20.50 cm
 F1P 14.000 ppm
 F2P -0.500 ppm



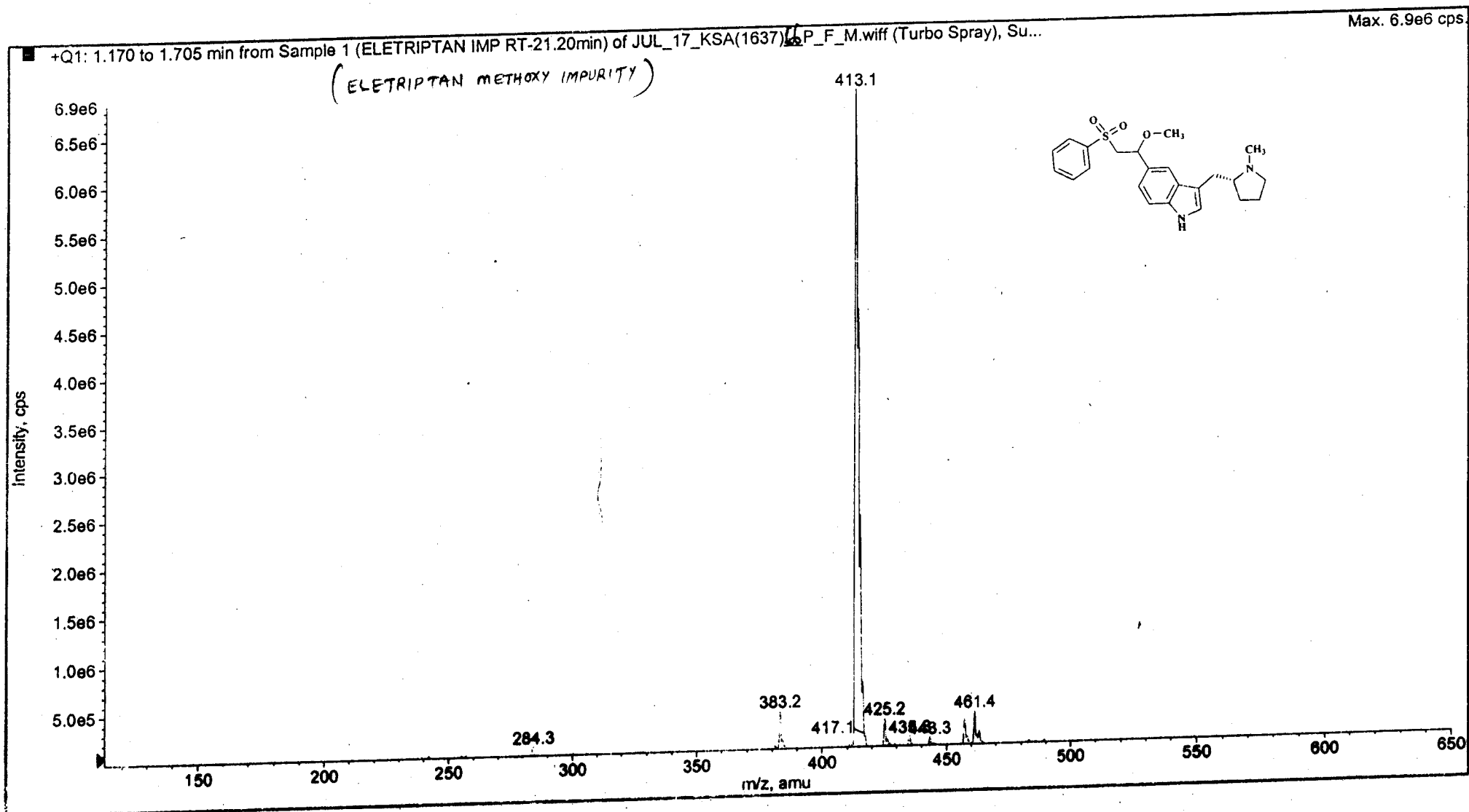
PENDANT-NMR of METHOXY ELETRIPTAN KSA (1637) 66 A.R.NO: S-010077
DMSO-d6



Analyst Version: 1.4.2
Acq. Time: 10:52
Acq. Date: Saturday, July 17, 2010

APL RESEARCH CENTRE
ANALYTICAL RESEARCH DEPARTMENT

Sample Comment: BNO.:KSA(1637)
Sample Name: ELETRIPTAN IMP RT-21.20min
Acq. File: JUL_17_KSA(1637)_P_F_M.wiff



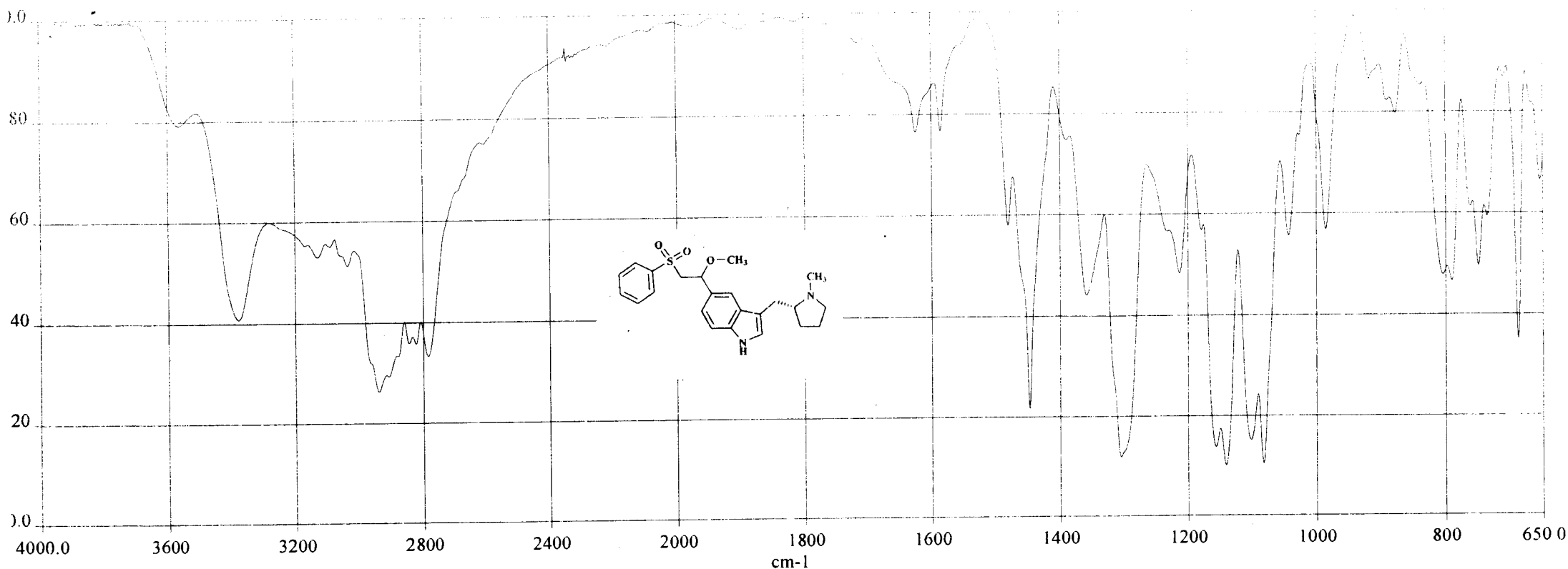
Analyst

Period/Expt.: Period 1, Experiment 1
Scan Mode: Profile
Polarity/Scan Type: Positive Q1 MS

Date: Saturday, March 17, 2012

APL RESEARCH CENTRE

RCII_AE009



Status

Filename: ELETRIPTAN METHOXY IMPURITY-KSA(1637)66.002
Date Created: Saturday, March 17, 2012 3:14 PM India Standard Time
Analyst: Analyst
Description: B.NO:KSA(1637)66
Comments:
SAMPLE:ELETRIPTAN METHOXY IMPURITY
PROJECT:ELETRIPTAN HBR
2.25 mg of sample in 455 of KBr

Abscissa: (cm-1)
Start: 4000.00
End: 450.00
Interval: -1.000000

Ordinate: (%T)
Maximum: 100.00
Minimum: 10.00
Points: 3551

Instrument Model : Spectrum One
Instrument Serial Number : 43000

ELETRIPTAN METHOXY IMPURITY-KSA(1637)66.pk

ELETRIPTAN METHOXY IMPURITY-KSA(1637)66.002 3351 4000.00 650.00 10.00 99.60 4.00 %T 8 1.00
B.NO:KSA(1637)66
REF 4000 99.51 2000 98.38 600
3563.14 79.14 3379.40 40.82 3130.25 53.04 3037.14 51.34 2938.62 26.29
2844.18 35.85 2822.47 35.71 2785.17 33.29 2343.57 91.20 1900.10 97.12
1624.30 76.53 1585.29 76.70 1480.43 57.93 1446.98 21.70 1358.37 44.17
1303.88 11.79 1213.28 48.41 1178.45 56.78 1156.16 13.76 1140.80 9.98
1102.30 15.12 1082.57 10.35 1042.55 55.65 985.34 56.81 918.23 86.53
877.02 79.70 804.07 47.93 789.90 46.72 748.06 49.65 734.05 59.10
709.69 86.87 687.95 35.12 654.16 66.42

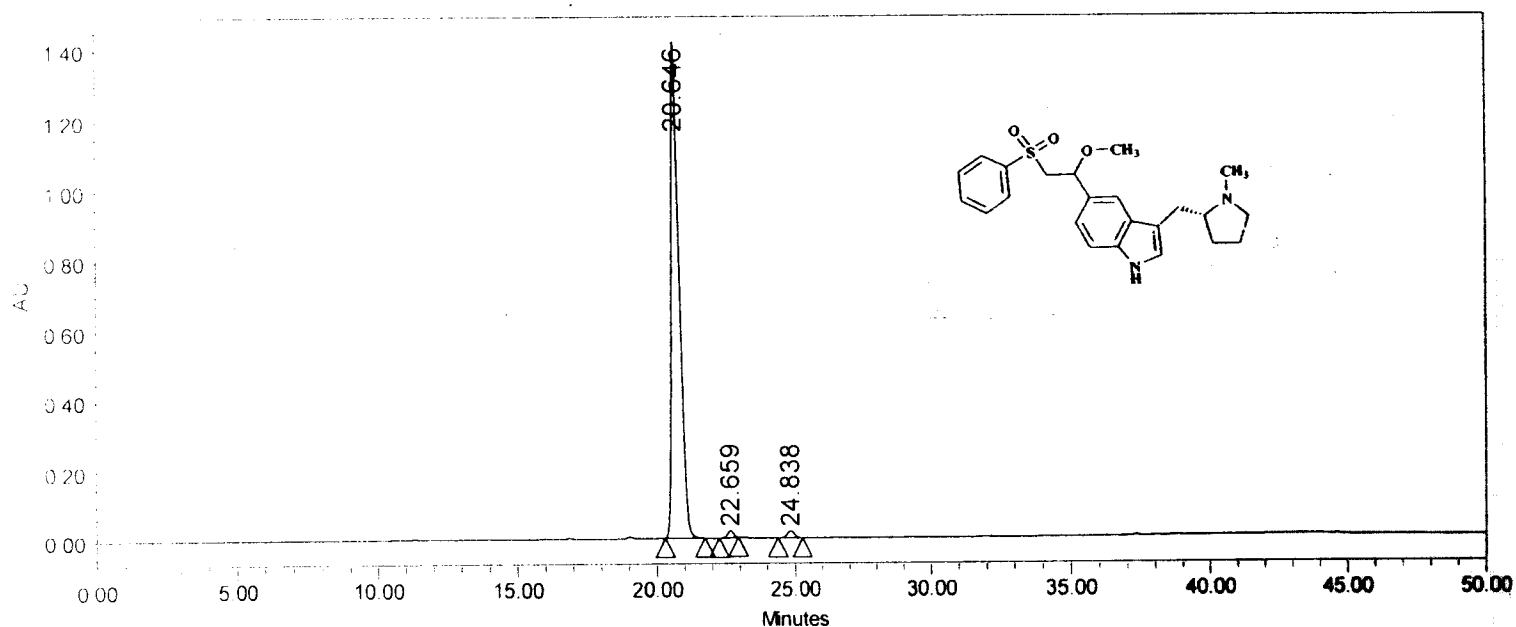
APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012\RCII_AE088

Sample ID : #KSA(1637)66/Eletriptan Methoxy imp
Run Time : 60.0 Minutes
Vial : 27
Injection : 1
Injection Volume : 20.00 ul

Proc. Chnl. Descr : W2996 PDA 225.0 nm
Date Acquired : 3/29/2012 4:00:13 PM IST
Acq. Method Set : Eletriptan_RS_MET
Date Processed : 3/30/2012 5:18:55 PM IST
Processing Method : Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu V \cdot sec$)	% Area	RT Ratio	Name
1	20.65	28054773	97.66		Eletriptan Methoxy Imp
2	22.66	298205	1.04	1.10	Peak2
3	24.84	374071	1.30	1.20	Peak3

(ELETRIPTAN TETRACYCLIC IMPURITY)

SAMPLE: msk-1739-141

Eletriptan Unk Imp
msk-1739-141

Solvent: dmsc
Temp. 25.0 C / 298.1 K
Operator: apl
File: PROTON_01
VNMRS-500 "APLV500"

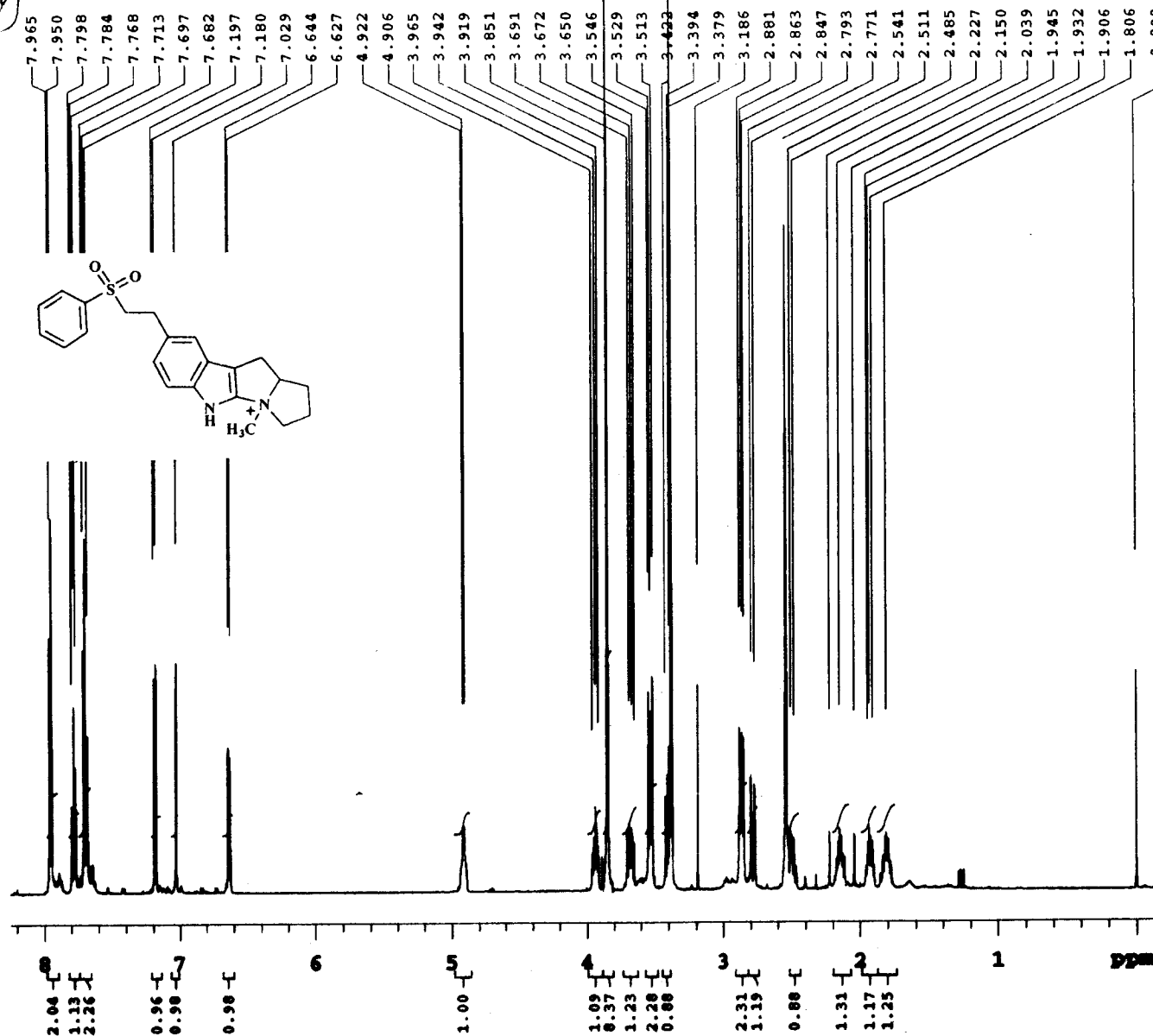
PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
32 repetitions

OBSERVE H1, 499.8205833

DATA PROCESSING

FT size 32768
Total time 1 minutes



(ELETRIPTAN TETRACYCLIC IMPURITY)

SAMPLE: msk-1739-141

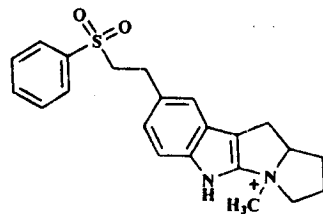
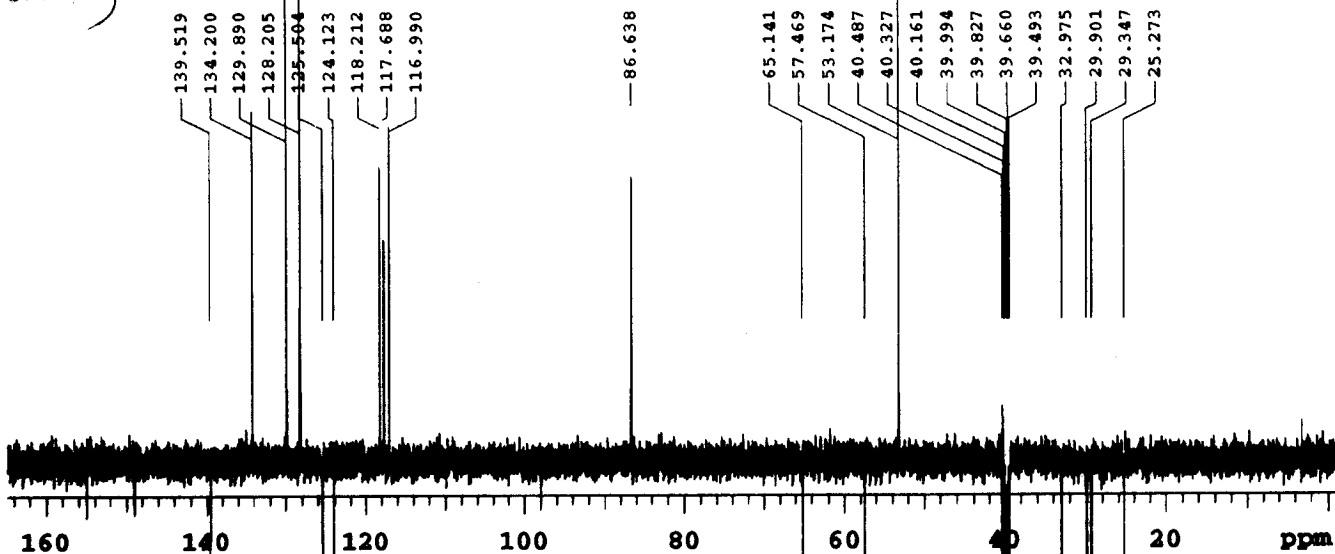
Eletriptan unk Imp
msk-1739-141

Solvent: dmsc
Temp. 25.0 C / 298.1 K
Operator: apl
File: dept_01
VMRS-500 "APLV500"

PULSE SEQUENCE: DEPT
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.022 sec
Width 31250.0 Hz
1920 repetitions

OBSERVE C13, 125.6799918
DECOUPLE M1, 499.8231069
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 64 minutes

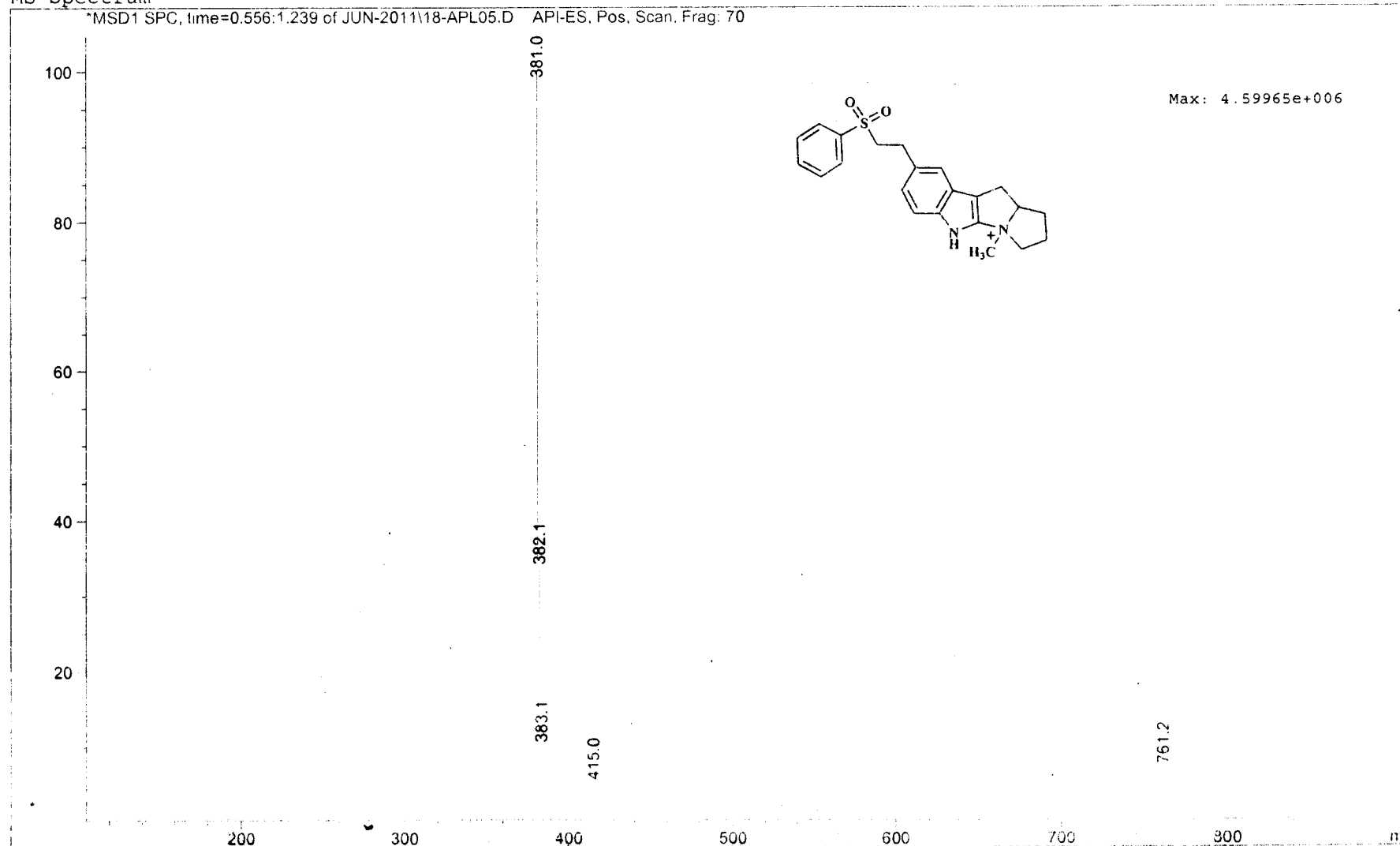


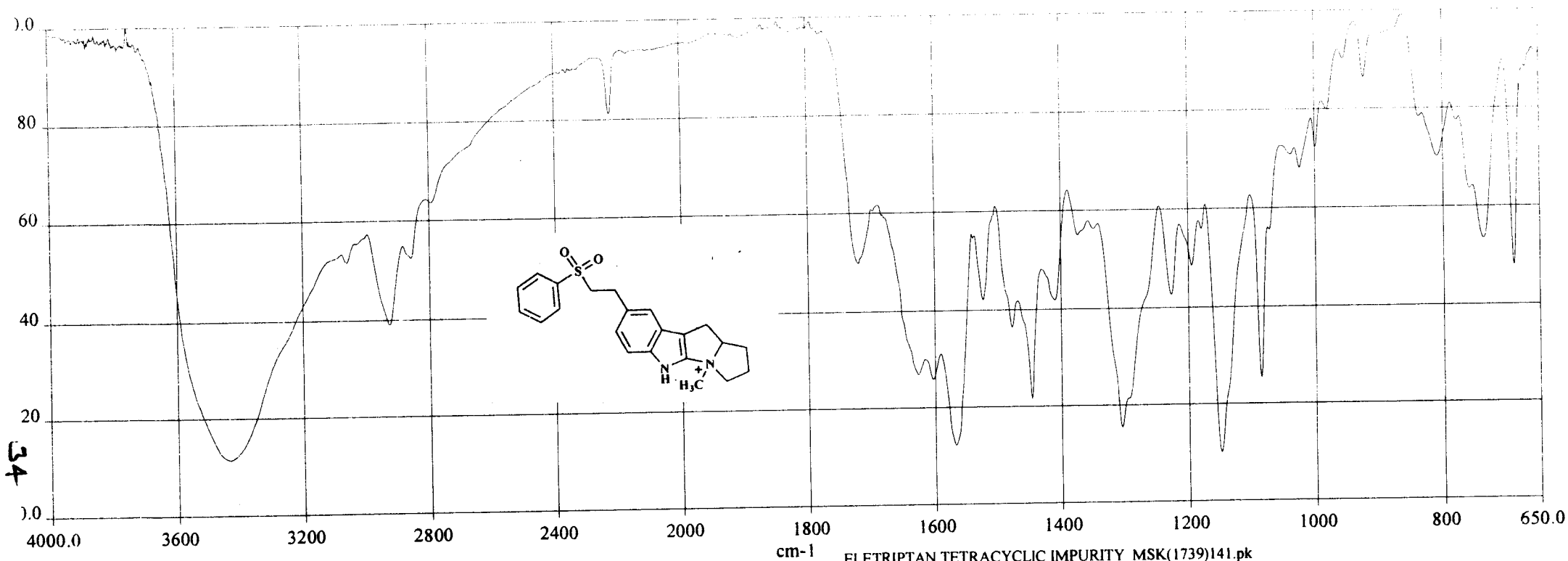
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Injection Date	: 6/18/2011 6:03:48 PM	Seq. Line	: 2
Sample Name	: MSK(1739)141	Location	: Vial 6
Acq. Operator	: MSR/KPR	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 10 μ l
Different Inj Volume from Sequence !		Actual Inj Volume	: 5 μ l
Acq. Method	: C:\HPCHEM\1\METHODS\POS_NEG.M		
Last changed	: 6/18/2011 5:42:15 PM by MSR/KPR		
Analysis Method	: C:\HPCHEM\1\METHODS\POS_NEG.M		
Last changed	: 7/8/2011 4:23:52 PM by MSR/KPR		
POS-NEG METHOD.			

(ELETRIPTAN TETRACYCLIC IMPURITY)

MS Spectrum





Status

Filename: ELETRIPTAN TETRACYCLIC IMPURITY_MSK(1739)141.001

Date Created: Wednesday, March 14, 2012 6:15 PM India Standard Time

Analyst: Analyst

Description: B.NO:MSK(1739)141 A.R.NO:S-1202058

Comments:

SAMPLE: ELETRIPTAN TETRACYCLIC IMPURITY

PROJECT: ELETRIPTAN HBR

2.01 mg OF SAMPLE IN 410.00 mg OF KBr

Abscissa: (cm-1)

Start: 4000.00

End: 450.00

Interval: -1.000000

Ordinate: (%T)

Maximum: 100.00

Minimum: 10.00

Points: 3551

Instrument Model:

Spectrum One

Instrument Serial Number:

42000

ELETRIPTAN TETRACYCLIC IMPURITY_MSK(1739)141.pk

ELETRIPTAN TETRACYCLIC IMPURITY_MSK(1739)141.001 3351 4000.0 650.0 10.0 100.0 4.0 %T 8 1.0

B.NO:MSK(1739)141

A.R.NO:S-1202058

REF 4000 98.6 2000 95.2 600

3925.0 96.8 3880.0 95.7 3835.5 96.7 3815.5 96.3 3779.3 95.3

3436.5 11.3 3062.6 51.5 2926.4 39.1 2857.3 52.4 2225.9 81.1

1907.0 96.1 1826.9 97.1 1719.3 49.8 1626.7 27.0 1603.0 25.9

1567.4 12.1 1523.1 42.0 1478.0 36.4 1446.8 21.6 1409.4 41.8

1373.0 54.9 1348.3 56.2 1305.0 15.3 1226.7 42.6 1194.6 48.5

1178.7 55.9 1148.9 9.9 1085.6 25.4 1038.0 70.8 1023.8 68.1

998.6 72.3 980.6 79.9 954.5 90.1 923.0 86.3 808.8 70.2

737.1 53.4 689.4 48.0

14/3/12

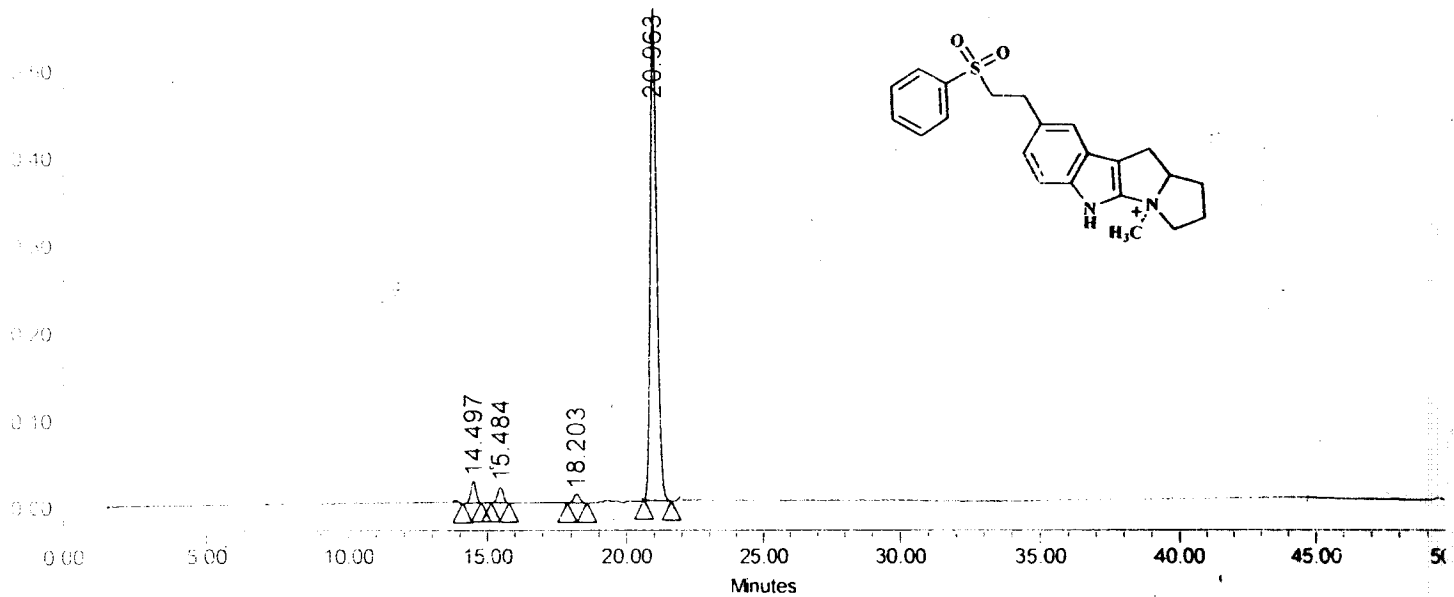
APL RESEARCH CENTRE ANALYTICAL RESEARCH DEPARTMENT

INSTRUMENT ID : RCII_AE088

PROJECT NAME : MAR_2012\RCII_AE088

Sample ID : MSK(1739)141/Eletriptan Tetra Cyclic
imp
Run Time : 60.0 Minutes
Vial : 28
Injection : 1
Injection Volume : 20.00 ul

Proc. Chnl. Descr : W2996 PDA 225.0 nm
Date Acquired : 3/29/2012 5:03:43 PM IST
Acq. Method Set : Eletriptan_RS_MET
Date Processed : 3/30/2012 5:20:17 PM IST
Processing Method : Eletriptan_RS_Pro



Peak Results

	RT (min)	Area ($\mu V \cdot sec$)	% Area	RT Ratio	Name
1	14.50	342690	3.43	0.69	Peak3
2	15.48	252697	2.53	0.74	Peak4
3	18.20	163072	1.63	0.87	Peak5
4	20.96	9225094	92.40	1.00	Eletriptan Tetra Cyclic imp

**APL RESEARCH CENTRE-II
ANALYTICAL RESEARCH DEPARTMENT**

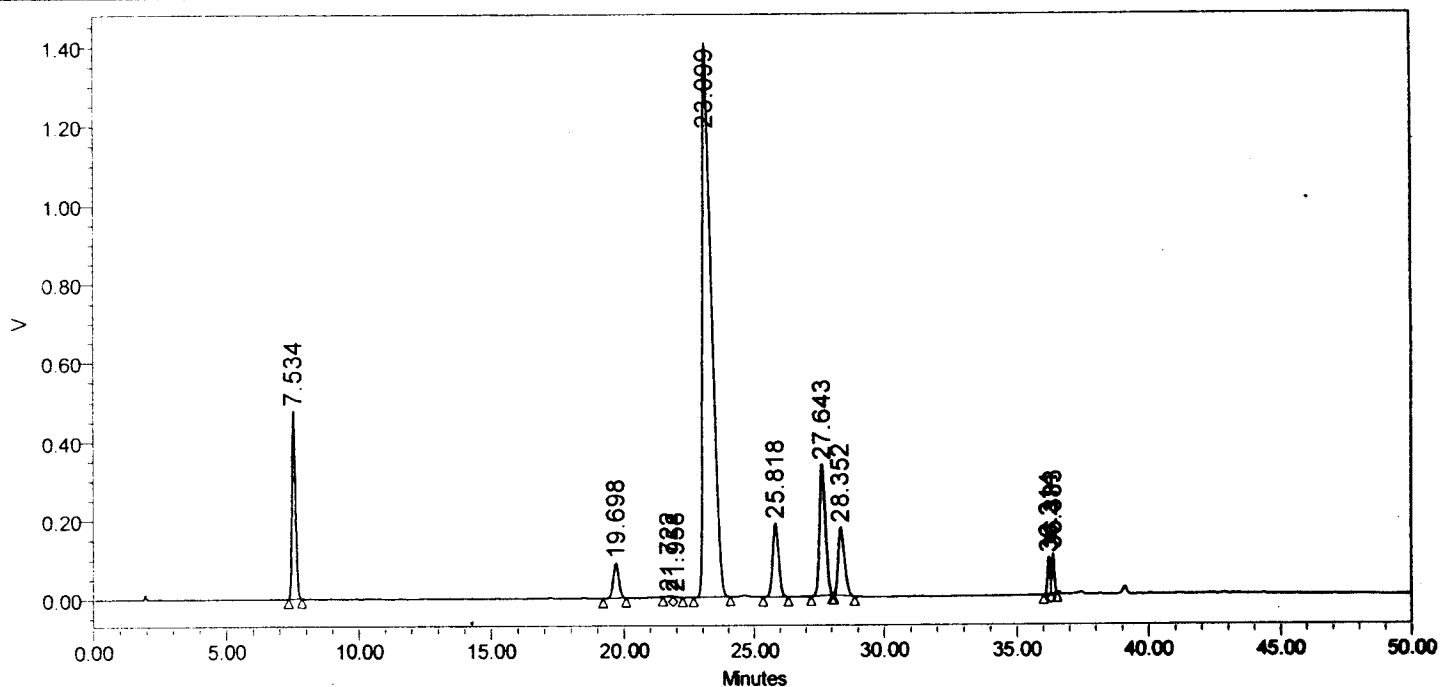
INSTRUMENT ID RCII_AE155

PROJECT NAME APR_2012\RCII_AE155

Sample ID: Imp Mix

Vial: 11
Injection: 1
Injection Volume: 20.00 ul
Run Time: 60.0 Minutes

Proc. Chnl. Descr.: Detector A 225nm
Date Acquired: 4/12/2012 11:53:49 AM IST
Acq. Method Set: Eletriptan_RS_MET
Date Processed: 4/13/2012 9:35:28 AM IST
Processing Method: Eletriptan_RS_Proc1



PEAK RESULTS

	RT	Area ($\mu\text{V}\cdot\text{sec}$)	% Area	RT Ratio	Compound Name	Compound Label
1	7.53	4543538	8.76	0.33	Desbromo Indole Pyrrolidine imp	6
2	19.70	1290120	2.49	0.85	Ethyl Indole Pyrrolidine imp	5
3	21.72	39054	0.08	0.94	Eletriptan Methoxy imp	7
4	21.96	30510	0.06	0.95	Eletriptan Tetracyclic imp	8
5	23.10	33414027	64.39	1.00	Eletriptan	1
6	25.82	2981761	5.75	1.12	Eletriptan N-Oxide-1	3
7	27.64	5416067	10.44	1.20	Eletriptan N-Oxide-2	4
8	28.35	2867398	5.53	1.23	Des acetyl ensulphone derivative	13
9	36.21	642276	1.24	1.57	Eletriptan Dimer	2
10	36.38	665175	1.28	1.58	Eletriptan Dimer	2