SUPPORTING INFORMATION (SI)

Synthesis, and Biological Evaluation of Orally Active Prodrugs of Indomethacin

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Contents of SI:

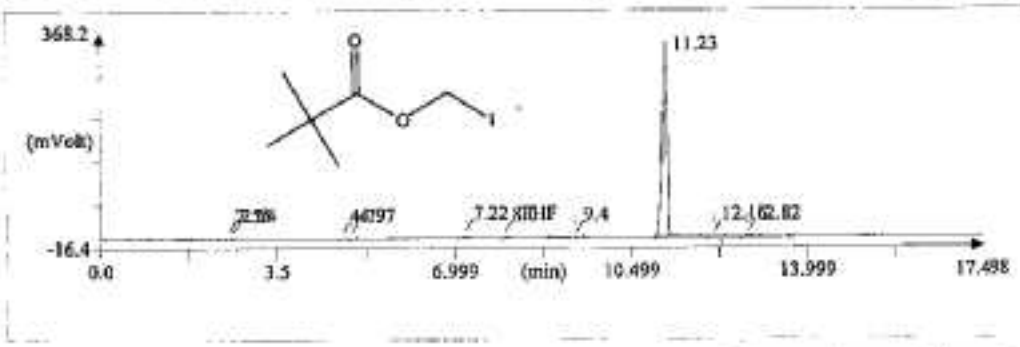
S. No.	SI Description	Page Number
1.	Chromatographic purity of pro-moiety **2** (GC)	S3
2.	^{1}H NMR of pro-moiety **2**	S4
3.	IR of pro-moiety **2**	S5
4.	Chromatographic purity of pro-moiety **3** (GC)	S6
5.	^{1}H NMR of pro-moiety **3**	S7
6.	IR of pro-moiety **3**	S8
7.	Chromatographic purity of pro-moiety **4** (GC)	S9
8.	^{1}H NMR of pro-moiety **4**	S10
9.	IR of pro-moiety **4**	S11
10.	Chromatographic purity of prodrug **1a** (HPLC)	S12
11.	UV Spectrum of prodrug **1a**	S13
12.	^{1}H NMR of prodrug **1a**	S14
13.	^{13}C NMR of prodrug **1a**	S15
14.	IR of prodrug **1a**	S16
15.	Mass spectrum of prodrug **1a**	S17
16.	XRD diffractogram for prodrug **1a**	S17-18
17.	Chromatographic purity of prodrug **1b** (HPLC)	S19
18.	UV Spectrum of prodrug **1b**	S20
19.	^{1}H NMR of prodrug **1b**	S21
20.	^{13}C NMR of prodrug **1b**	S22
21.	IR of prodrug **1b**	S23
22.	Mass spectrum of prodrug **1b**	S24
23.	XRD diffractogram for prodrug **1b**	S24-25
24.	Chromatographic purity of prodrug **1c** (HPLC)	S26
25.	UV Spectrum of prodrug **1c**	S27
26.	^{1}H NMR of prodrug **1c**	S28
27.	Mass spectrum of prodrug **1c**	S28
28.	^{13}C NMR of prodrug **1c**	S29

29.	IR of prodrug **1c**	S30
30.	XRD diffractogram for prodrug **1c**	S31-32
31.	Chromatographic purity of prodrug **1d** (HPLC)	S33
32.	UV Spectrum of prodrug **1d**	S34
33.	^{1}H NMR of prodrug **1d**	S35
34.	Mass spectrum of prodrug **1d**	S35
35.	^{13}C NMR of prodrug **1d**	S36
36.	IR of prodrug **1d**	S37
37.	XRD diffractogram for prodrug **1d**	S38-39

1. Chromatographic purity (GC) of pro-moiety **2** (Iodomethyl pivalate)

CALIBRATION REPORT(PDL.03.08)

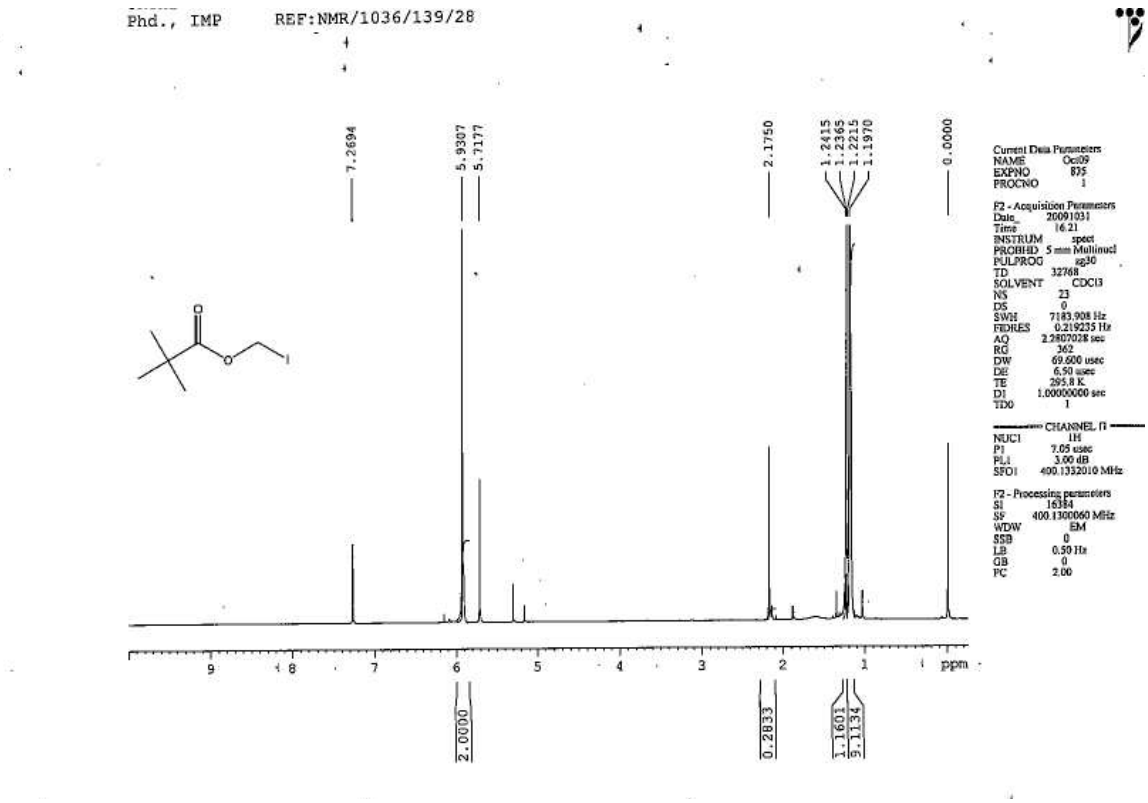
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Analysed:	05/18/08 16:04
Printed:	5/18/08 16:26
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Channel:	Channel A
Chromatogram filename:	C:\Chrom-Card for TRACE\data\Jun'08\180608IMP009.dat



Peak Number (#)	Retention Time (min)	Component Name	Area (.1*uV*sec)	Area (%)
1	2.550		1672	0.007
2	2.643		5443	0.022
3	4.790		1132	0.005
4	4.970		1009	0.004
5	7.222		164522	0.676
6	7.995		29665	0.122
7	9.403		16003	0.066
8	11.227		24114200	99.01
9	12.158		8132	0.033
10	12.822		12805	0.053

ANALYSED BY: REVIEWED BY:

2. ^{1}H NMR of pro-moiety **2**



3. IR of pro-moiety **2**

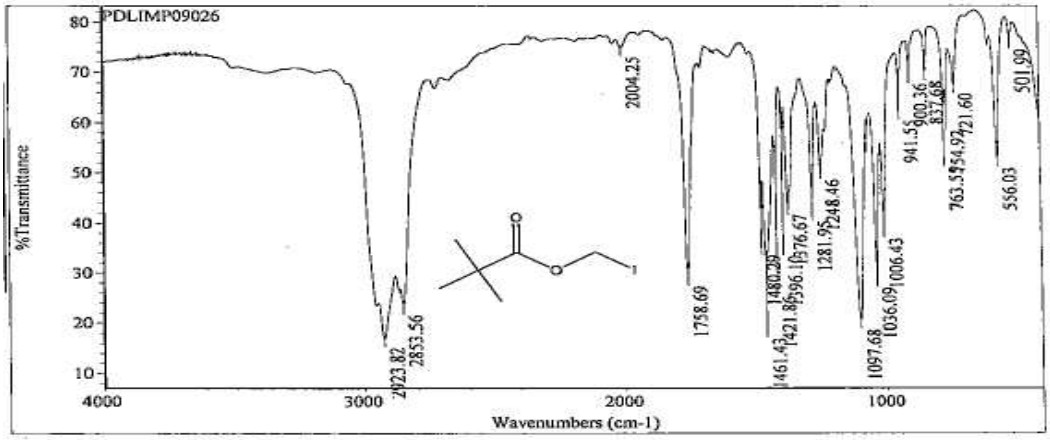
SAMPLE INFORMATION

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Description :

Collection time: Sun Oct 25 14:07:53 2009 (GMT+05:30)

Printed On : Sun Oct 25 14:08:28 2009 (GMT+05:30) User name: PDLAB



Number of sample scans: 32
Number of background scans: 32
Resolution: 4.000
Sample gain: 1.0
Mirror velocity: 0.6329
Aperture: 100.00

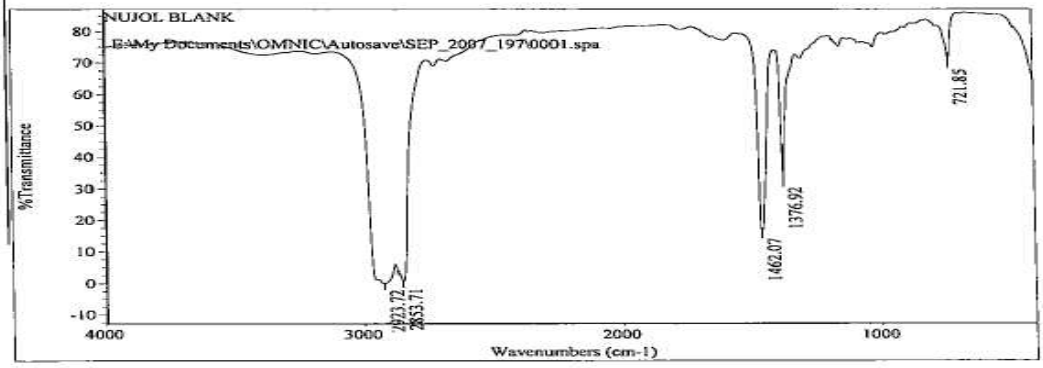
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Printed On : Sun Oct 25 14:01:34 2009 (GMT+05:30) User name: PDLAB



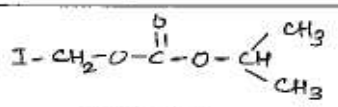
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Number of background scans: 32
Resolution: 4.000
Sample gain: 1.0
Mirror velocity: 0.6329
Aperture: 100.00

4. Chromatographic purity of pro-moiety **3** (GC)

Software Version	: 6.3.1.0504	Date	: 12/14/2008 10:07:08 AM
Sample Name	: IMIPC A/DISTILLATION	Data Acquisition Time	: 12/14/2008 9:49:12 AM
Instrument Name	: PDL/GC- PE/44	Channel	: A
Rack/Vial	: 0/0	Operator	: pdlab
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 1		

Result File :
Sequence File : C:\GC DATA\Sequence\131208IEIPC.seq

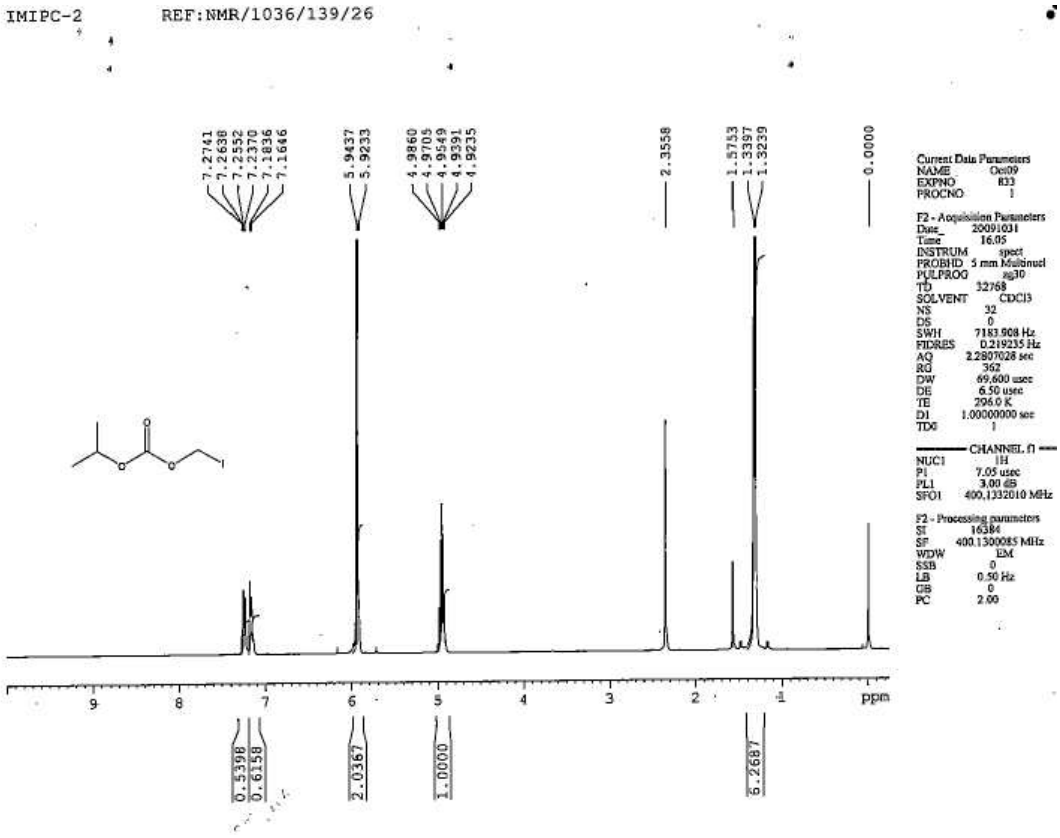
Final product (side chain)



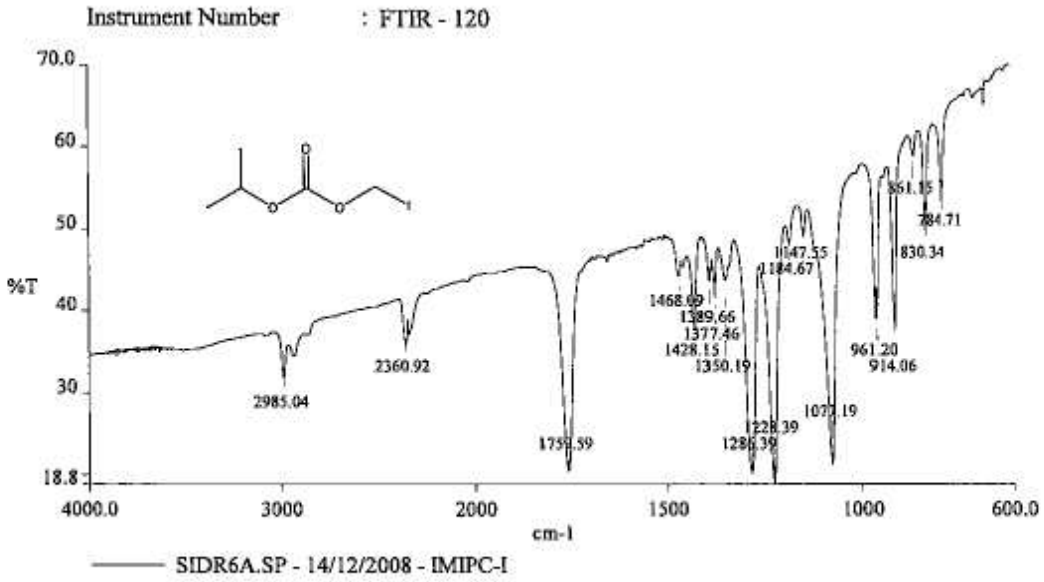
IMIPC
(Idomethyl isopropyl carbonate)

Peak #	Component Name	Time [min]	Area [uV*sec]	Area [%]	Rel. RT	
1		1.511	5999.67	0.60	1.00	→ ACN
2		4.207	3851.95	0.39	2.78	→ CMIPC (Starting)
3	IEIPC	6.664	983402.61	99.01	4.41	→ IMIPC (Product)
			993254.23	100.00		

5. ^{1}H NMR of pro-moiety **3**



6. IR of pro-moiety **3**



SAMPLE INFORMATION

Status - c:\pel_data\spectra\rm\sidr6a.sp

Filename: sidr6a.sp
Date Created: Sun Dec 14 18:31:24 2008
Analyst: PDL
Description: IMIPC-I
Comments: IMIPC-I

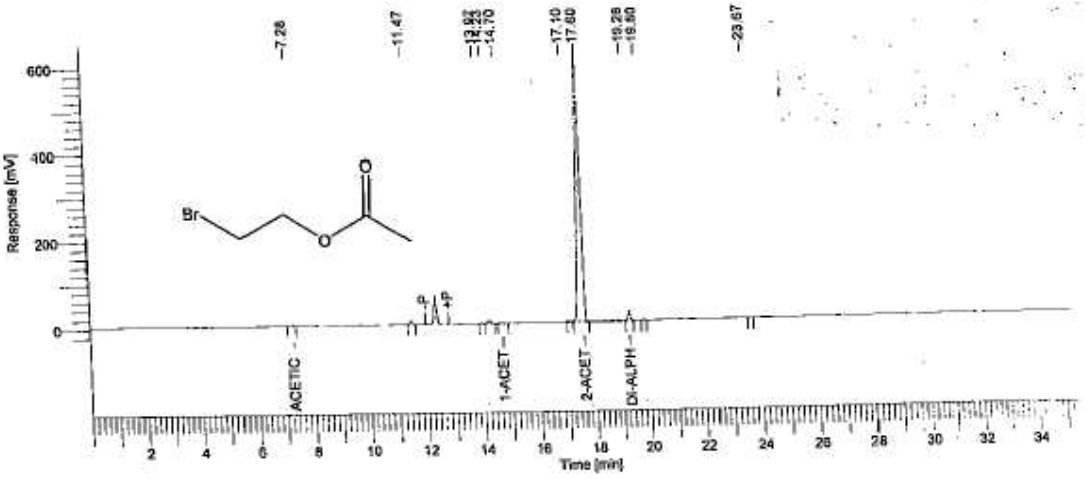
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Points: 1701
Printed on : Date: 14/12/2008 Time: 18:36:13

7. Chromatographic purity of pro-moiety **4** (GC)

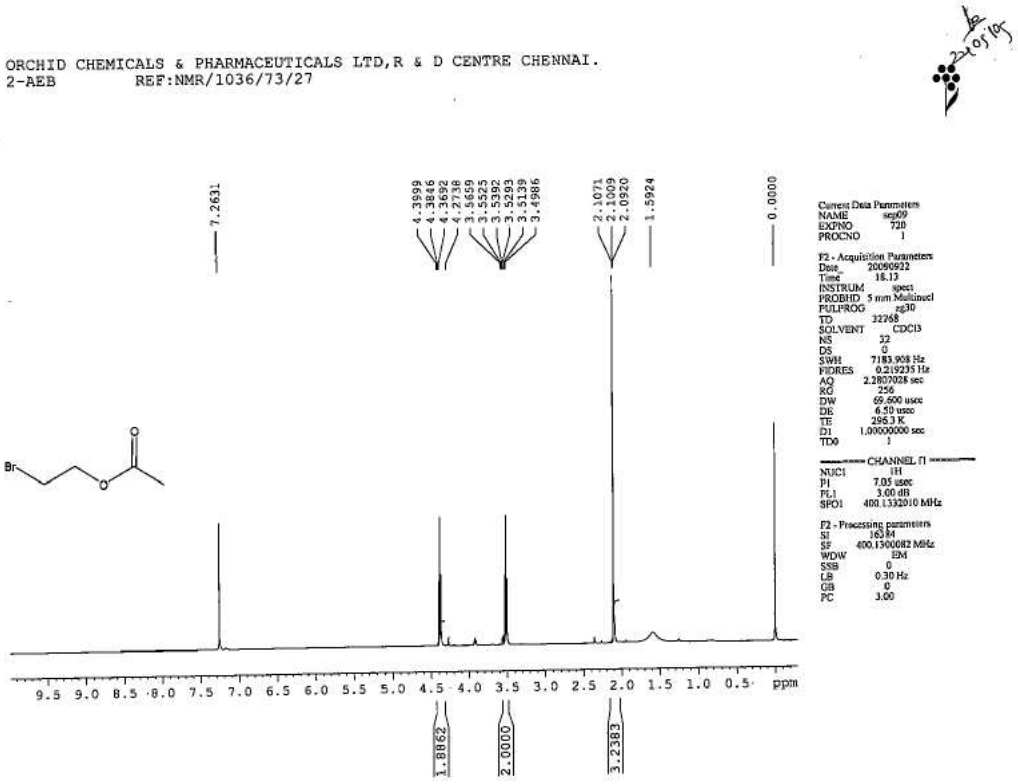
Software Version	: 6.3.1.0504	Date	: 5/24/2009 12:55:11 PM
Sample Name	: 2AEB A/BRINE WASH	Data Acquisition Time	: 5/24/2009 11:48:43 AM
Instrument Name	: PDL/GC- PE/44	Channel	: A
Rack/Vial	: 0/0	Operator	: pdlab
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 2		

Result File : C:\GC DATA\Data\may_09\240509_AEB002.rst
Sequence File : C:\GC DATA\Sequence\2405_AEB.seq
Sample Notes:
TC_047D_94

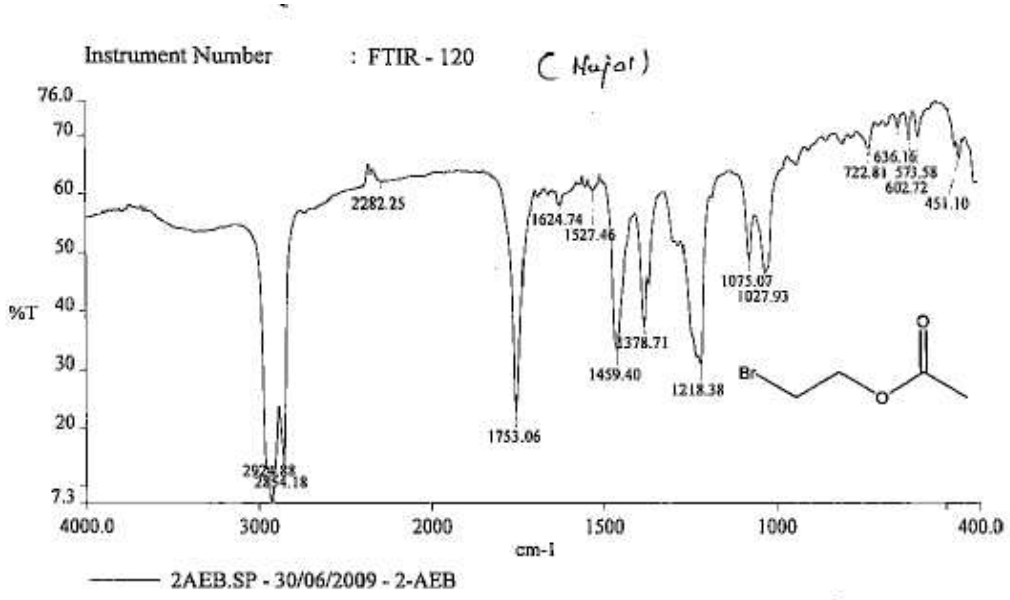


Peak #	Component Name	Time [min]	Area [uV*sec]	Area [%]	Rel. RT
1	Acetic acid	7.276	8324.28	0.14	1.00
2		11.466	39472.66	0.65	1.58
3		13.969	1101.00	0.02	1.92
4		14.231	31310.33	0.51	1.96
5	1-Acetoxy ethyl brom	14.696	6672.13	0.11	2.02
6		17.099	1268.58	0.02	2.35
7	2-Acetoxy ethyl brom	17.598	5891052.27	96.72	2.42 → 2-AEB
8	Di-alpha-bromoethyl	19.279	104990.74	1.72	2.65
9		19.803	5091.52	0.08	2.72
10		23.670	1841.18	0.03	3.25
			6091124.70	100.00	

8. ^{1}H NMR of pro-moiety **4**



9. IR of pro-moiety **4**



SAMPLE INFORMATION

Status - c:\pel_data\spectra\jun09120\2aeb.sp

Filename: 2aeb.sp
Date Created: Tue Jun 30 12:28:20 2009
Analyst: PDL
Description: 2-AEB
Comments:

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End: 400.00
Interval: -2.000000

Ordinate: (%T)
Maximum: 75.97
Minimum: 7.26
Points: 1801
Printed on : Date: 30/06/2009 Time: 12:28:47

10. Chromatographic purity of prodrug **1a** (HPLC)

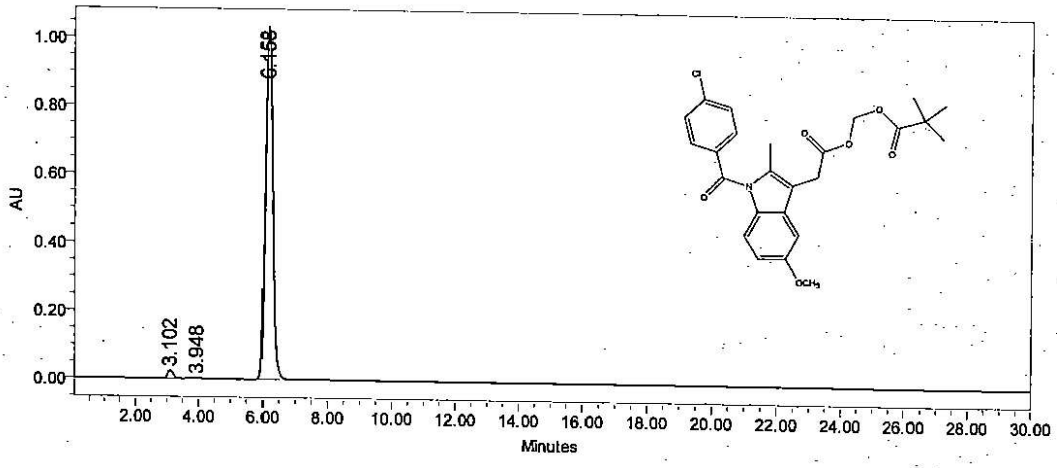
SID REACTION MONITORING

HPLC ID:PDL/HPLCW/26
COLUMN:149

STP CODE:TC014B49

SAMPLE INFORMATION

Sample Name:	SID-4002 (FINAL)	Acquired By:	orchid
Sample Type:	Unknown	Date Acquired:	17/08/2008 13:54:45
Vial:	6	Acq. Method Set:	FURPROFEN_MODI
Injection #:	1	Date Processed:	17/08/2008 15:03:24
Injection Volume:	20.00 ul	Processing Method:	FURPROFEN RS TC014B49
Run Time:	30.0 Minutes	Channel Name:	WvIn Ch7
Sample Set Name:	170808 FURPROFEN RS	Proc. Chnl. Descr.:	PDA 254.0 nm



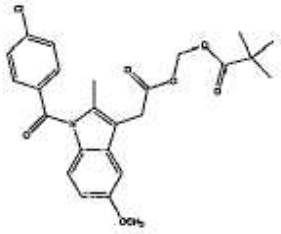
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1	3.102	275135	1.68	201.8
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3	6.158	16063501	98.13	208.8

11. UV Spectrum of prodrug **1a**

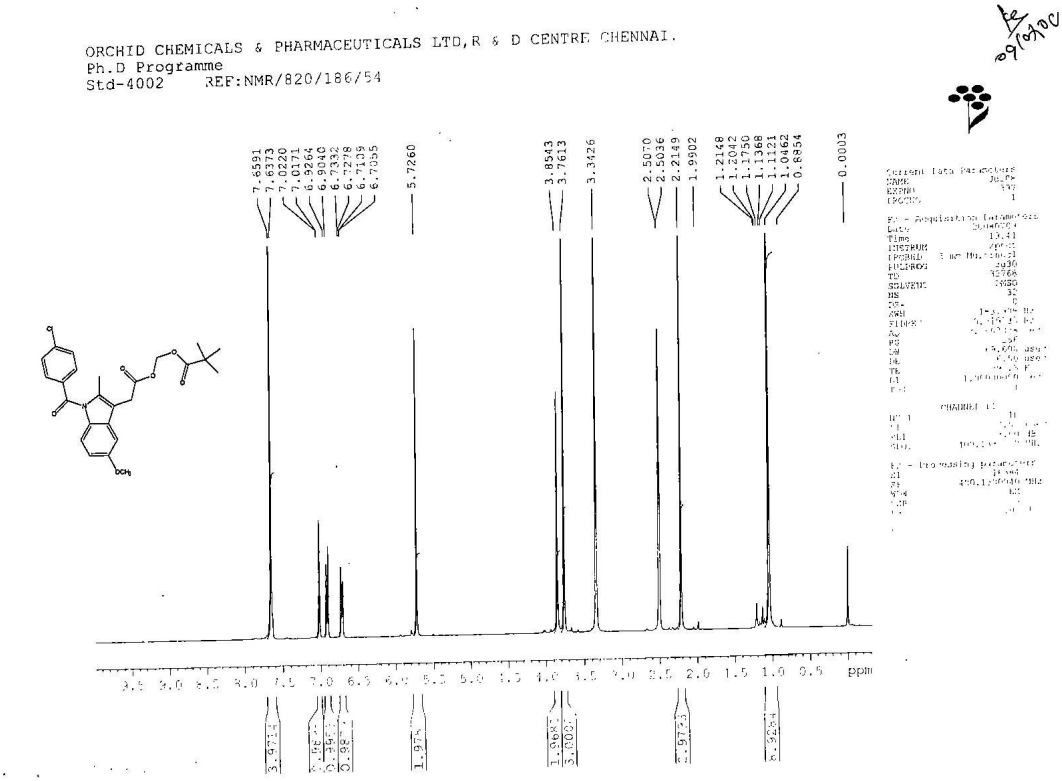
Date : 2/26/2010 Time : 13:18:45

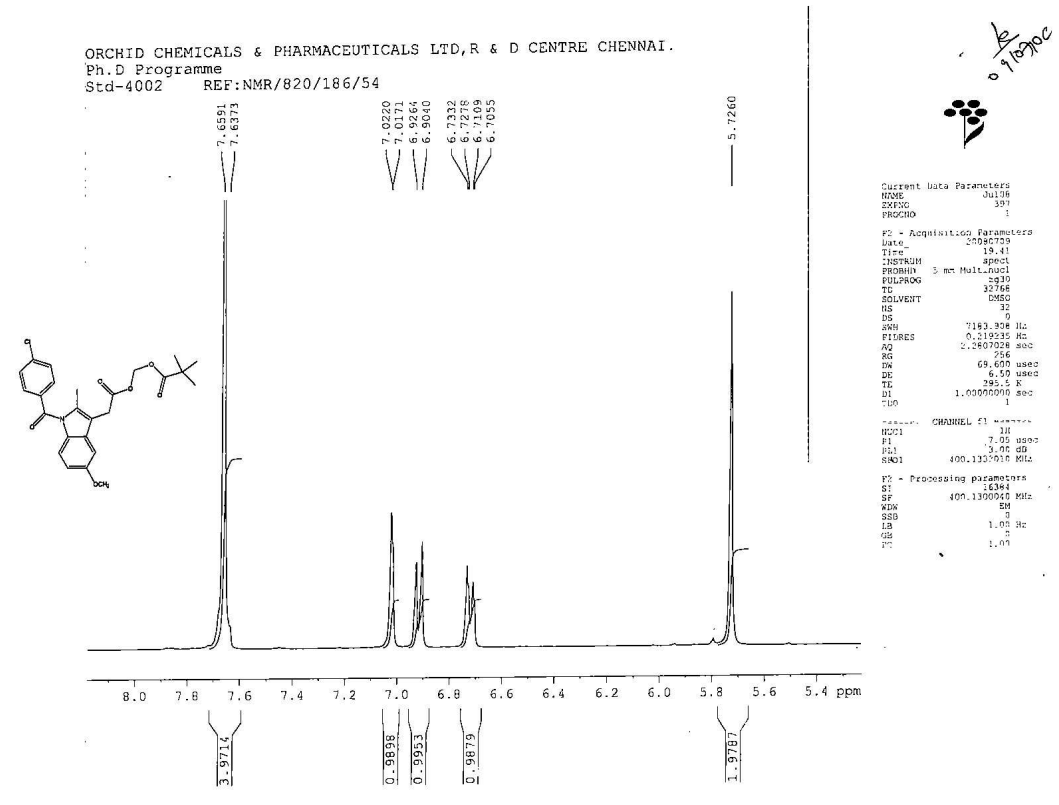
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nent: SID-4002
shold: 0.0200
:issa units: nm
inate units: A

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2	272.49	0.3176	Peak
1	301.20	0.2155	Base

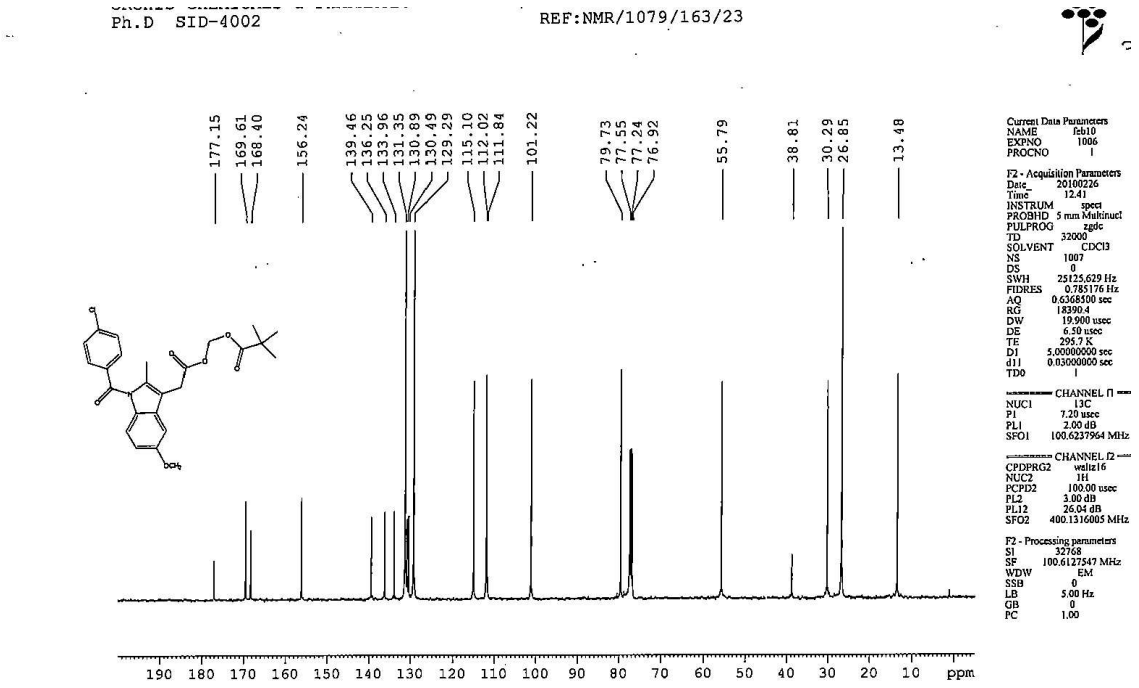


12. ^{1}H NMR of prodrug **1a**

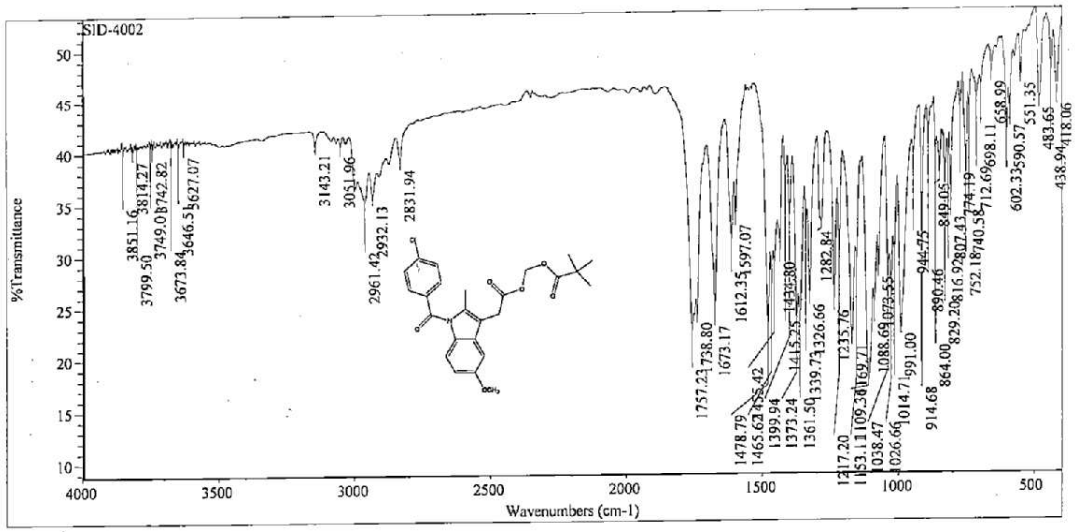




13. ^{13}C NMR of prodrug **1a**



14. IR of prodrug **1a**

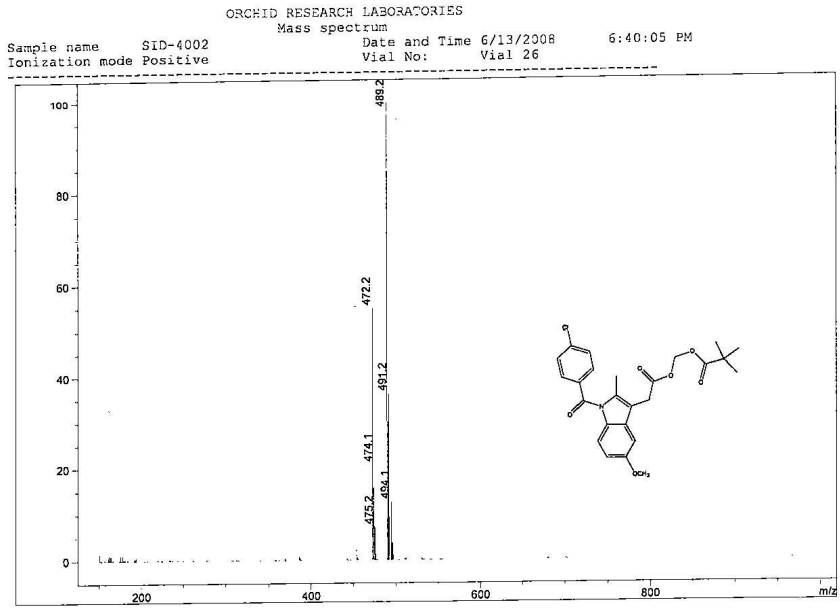


Mon Feb 15 18:00:10 2010 (GMT+05:30)
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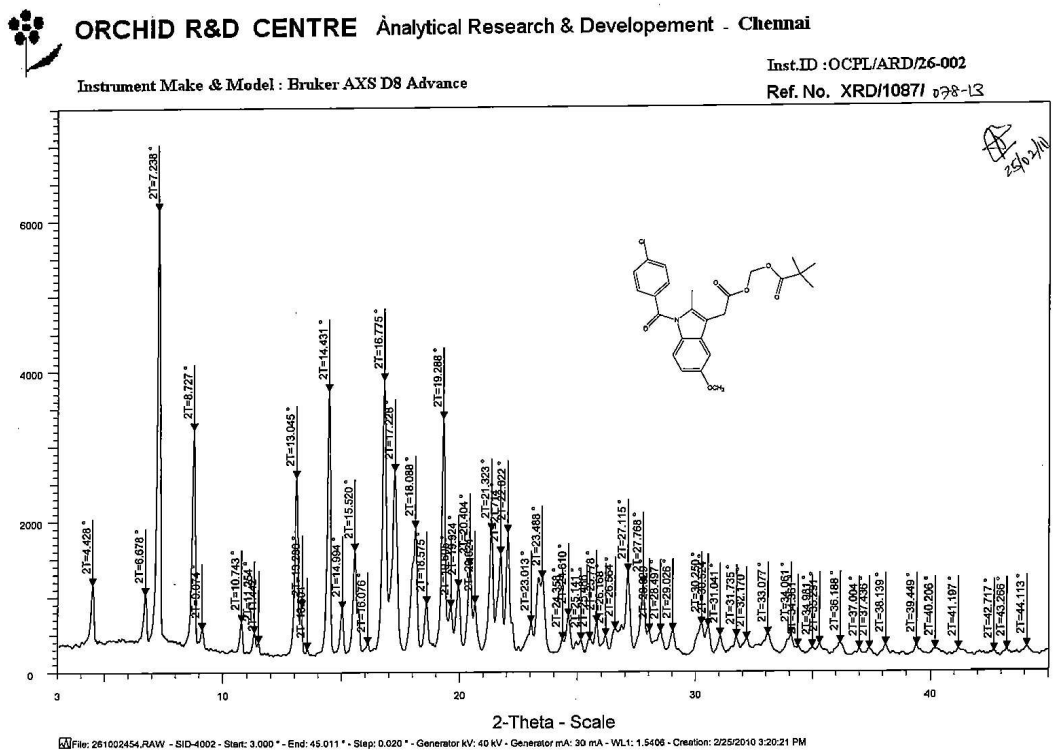
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Sensitivity:	60	
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Position:	438.94	Intensity:	47.213
Position:	483.65	Intensity:	44.555
Position:	551.35	Intensity:	46.007
Position:	590.57	Intensity:	43.843
Position:	602.33	Intensity:	41.441
Position:	658.99	Intensity:	47.054
Position:	698.11	Intensity:	45.909

15. Mass spectrum of prodrug **1a**

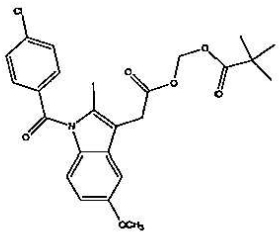


16. XRD diffractogram for prodrug **1a**



16. XRD diffractogram for prodrug **1a**

261002454.RAW BID-4902			
Angle	d value	Intensity	Intensity
2-Theta °	Angstrom	Count	%
4.43	19.94	1155	18.6
6.68	13.23	1025	16.7
7.24	12.20	6160	100
8.73	10.12	3220	52.3
9.07	9.74	556	9.1
10.74	8.23	663	10.8
11.25	7.86	517	8.4
11.44	7.73	382	6.2
13.05	6.78	2577	41.8
13.28	6.66	855	13.9
13.50	6.55	290	4.7
14.43	6.13	3736	60.6
14.99	5.90	839	13.6
15.52	5.70	1604	26
16.08	5.51	361	5.9
16.78	5.28	3878	62.9
17.23	5.14	2663	43.2
18.09	4.90	1902	30.9
18.58	4.77	898	14.6
19.29	4.60	3357	54.5
19.61	4.52	850	13.8
19.92	4.45	1120	18.2
20.40	4.35	1403	22.8
20.62	4.30	902	14.6
21.32	4.16	1868	30.3
21.71	4.09	1553	25.2
22.02	4.03	1839	29.8
23.01	3.86	637	10.3
23.49	3.78	1230	20
24.36	3.65	415	6.7
24.61	3.61	728	11.8
25.14	3.54	411	6.7
25.49	3.49	416	6.8
25.78	3.45	638	10.4
26.17	3.40	463	7.5
26.58	3.35	554	9
27.12	3.29	1317	21.4
27.77	3.21	1145	18.6
28.03	3.18	525	8.5
28.50	3.13	517	8.4
29.03	3.07	525	8.5
30.25	2.95	610	9.9
30.52	2.93	586	9.5
31.04	2.88	461	7.5
31.74	2.82	441	7.2
32.17	2.78	409	6.6
33.08	2.71	469	7.6
34.06	2.63	469	7.6
34.36	2.61	307	5
34.99	2.56	296	4.8
35.29	2.54	343	5.6
36.19	2.48	352	5.7
37.00	2.43	279	4.5
37.44	2.40	281	4.6
38.14	2.36	328	5.3
39.45	2.28	324	5.3
40.21	2.24	277	4.5
41.20	2.19	269	4.4
42.72	2.12	238	3.9
43.27	2.09	261	4.2
44.11	2.05	295	4.8



17. Chromatographic purity of prodrug **1b** (HPLC)

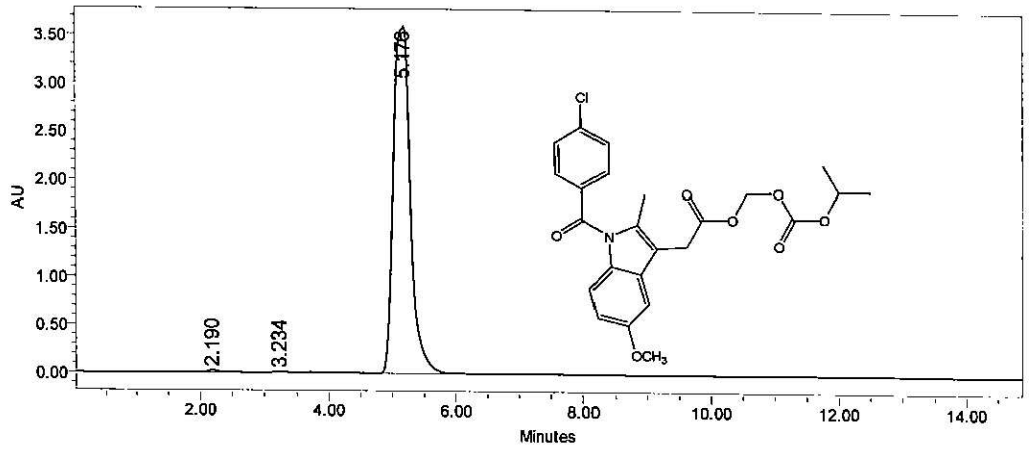
SID REACTION MONITORING

HPLC ID:PDL/HPLCW/26
COLUMN:149

STP CODE:TC014B49

SAMPLE INFORMATION

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Sample Type:	Unknown	Date Acquired:	12/04/2009 16:39:58
Vial:	10	Acq. Method Set:	FURPROFEN_MODI
Injection #:	1	Date Processed:	12/04/2009 16:56:18
Injection Volume:	20.00 ul	Processing Method:	FURPROFEN RS TC014B49
Run Time:	30.0 Minutes	Channel Name:	Wvln Ch47
Sample Set Name:	120409 FURPROFEN RS	Proc. Chnl. Descr.:	PDA 254.0 nm



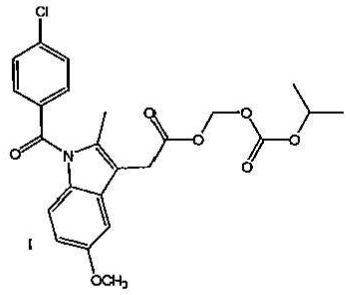
	RT	Area	% Area	Peak Lambda Max.
1	2.190	265255	0.40	328.4
2	3.234	144243	0.21	200.6
3	5.176	66701649	99.39	233.5

18. UV spectrum of prodrug **1b**

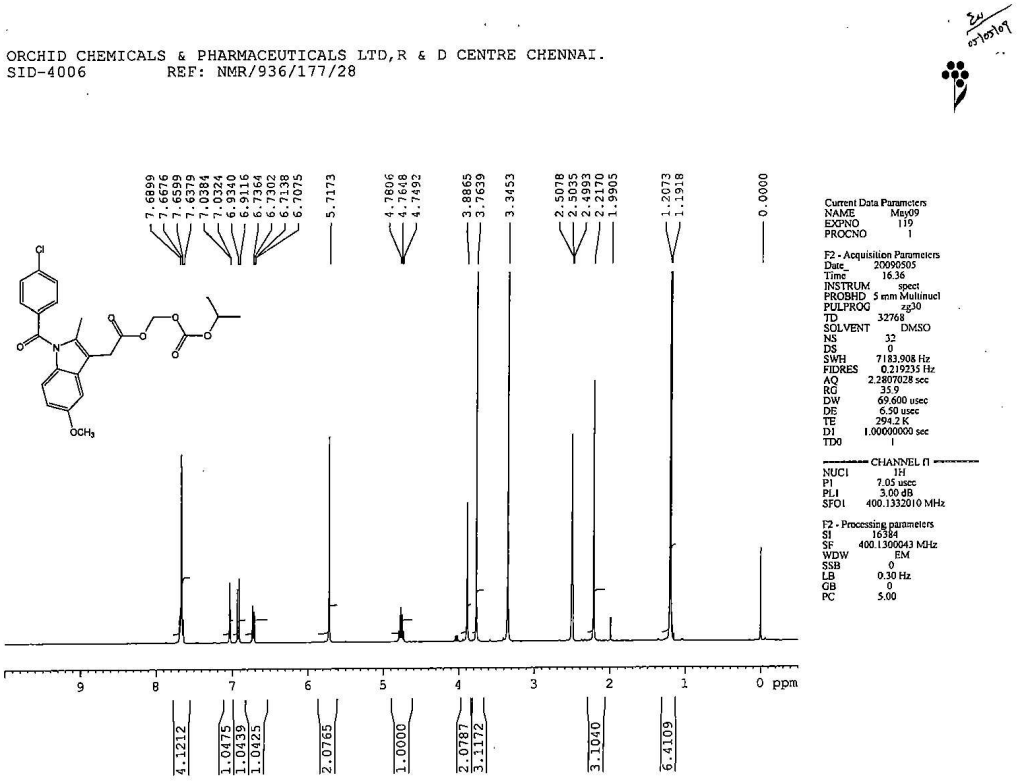
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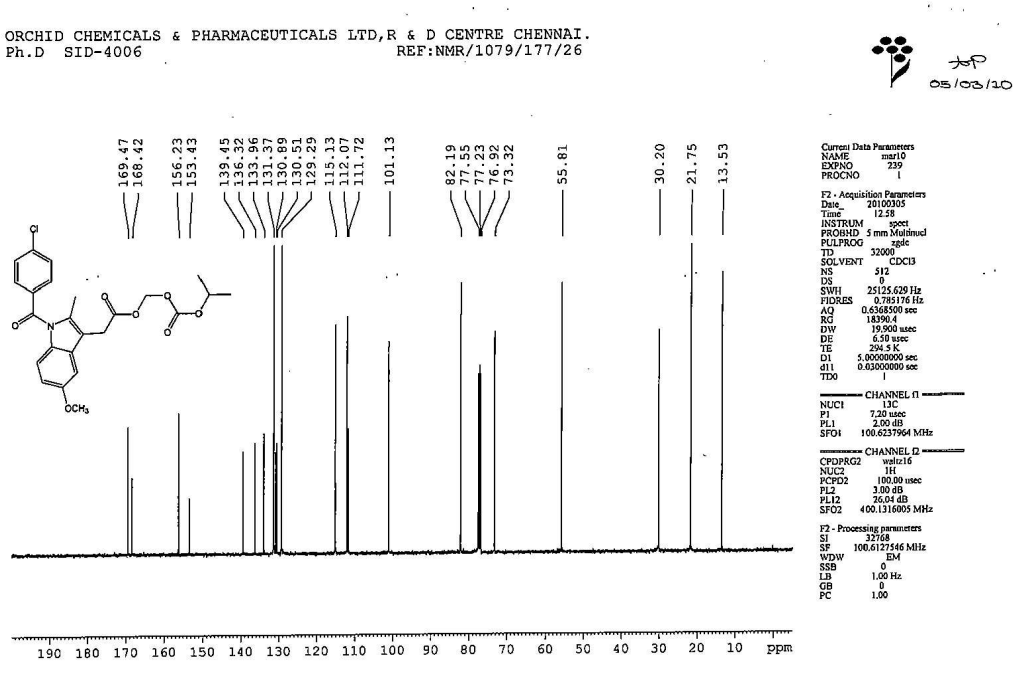
o.	Abscissa	Ordinate	Type
1	319.24	0.2280	Peak
2	272.49	0.2969	Peak
1	302.07	0.1944	Base



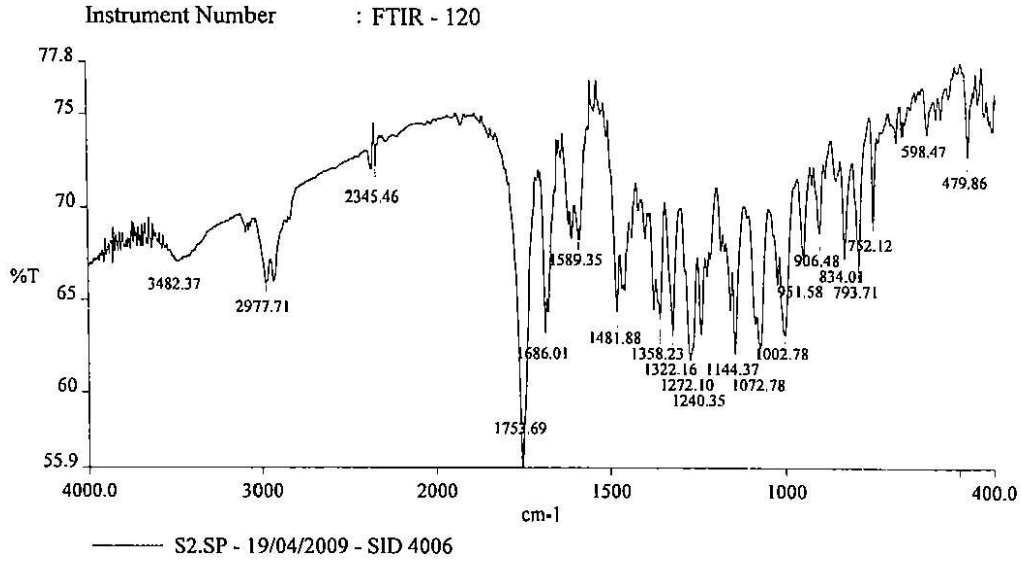
19. ^{1}H NMR of prodrug **1b**



20. ^{13}C NMR of prodrug **1b**



21. IR of prodrug **1b**



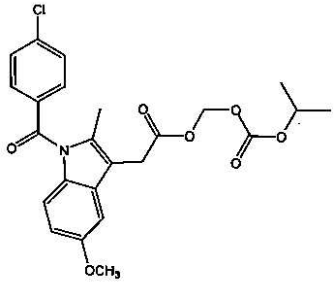
SAMPLE INFORMATION

Status - c:\pel_data\spectra\mar09120\s2.sp

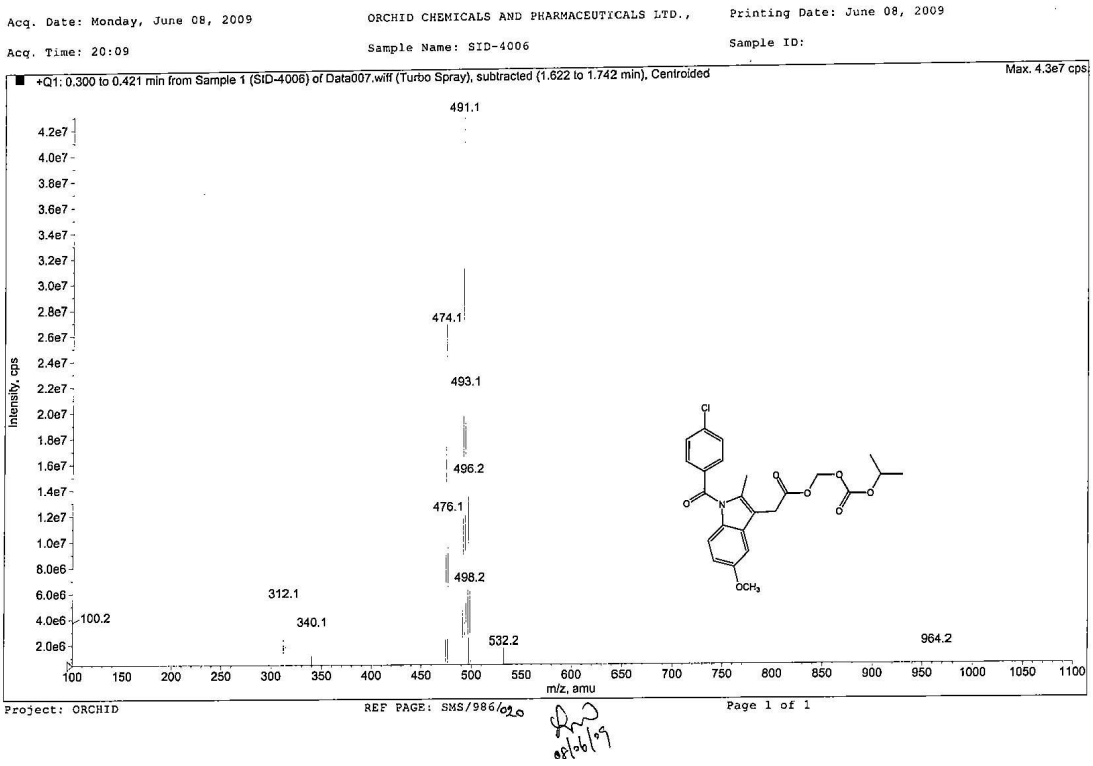
Filename: s2.sp
Date Created: Sun Apr 19 11:35:21 2009
Analyst: PDL
Description: SID 4006
Comments: SID 4006

Abscissa: (cm-1)
Start: 4000.00
End: 400.00
Interval: -2.000000

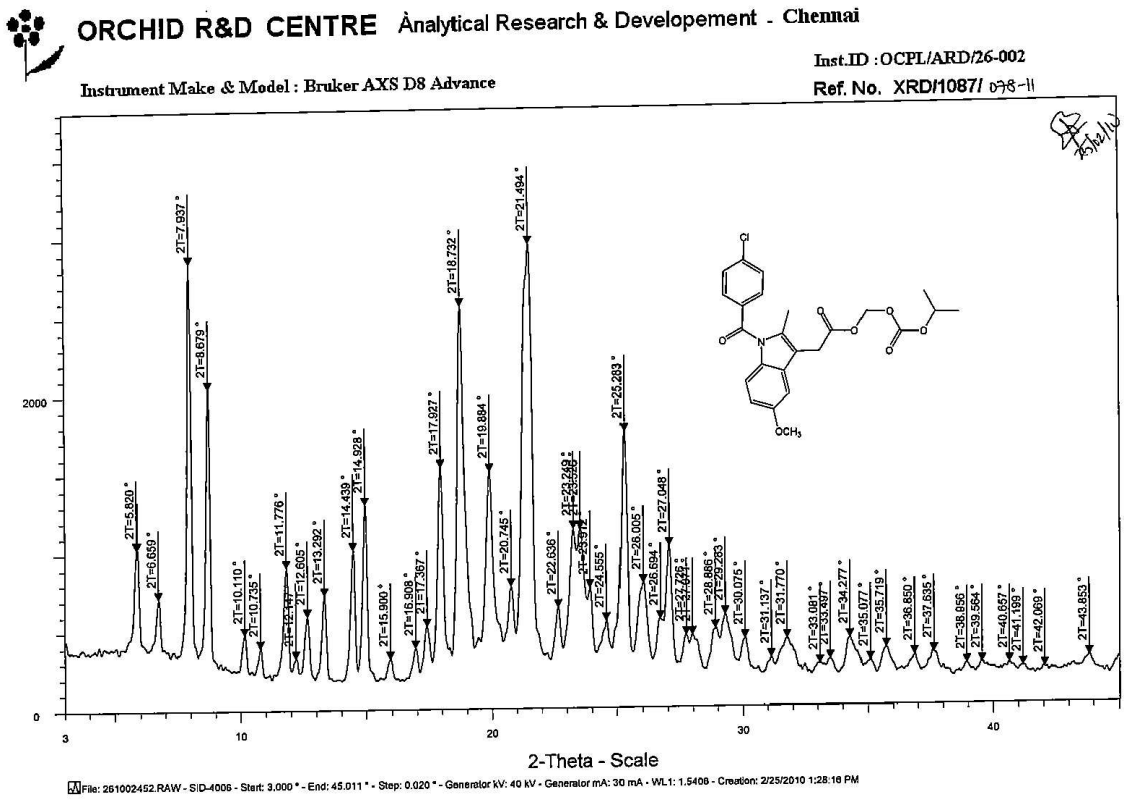
Ordinate: (%T)
Maximum: 77.82
Minimum: 55.92
Points: 1801
Printed on : Date: 19/04/2009 Time: 11:35:27



22. Mass spectrum of **1b**

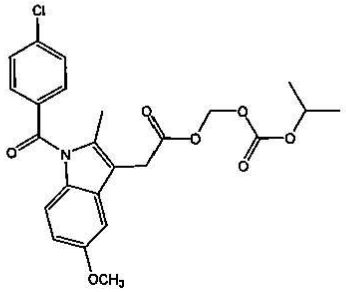


23. XRD diffractogram for prodrug **1b**



23. XRD diffractogram for prodrug **1b**

261002452.RAW SID-4006			
Angle	d value	Intensity	Intensity
2-Theta °	Angstrom	Count	%
5.82	15.17	1028	34.9
6.68	13.26	711	24.1
7.94	11.13	2849	96.8
8.68	10.18	2047	69.4
10.11	8.74	475	16.1
10.74	8.23	391	13.3
11.78	7.51	904	30.8
12.15	7.28	324	11
12.61	7.02	590	20
13.28	6.68	724	24.8
14.44	6.13	1013	34.3
14.93	5.93	1298	44
15.90	5.57	315	10.7
16.91	5.24	383	13
17.37	5.10	521	17.7
17.93	4.94	1531	51.9
18.73	4.73	2584	88.9
19.88	4.46	1502	50.9
20.75	4.28	771	26.1
21.49	4.13	2950	100
22.84	3.92	633	21.5
23.25	3.82	1132	38.4
23.53	3.78	1130	38.3
23.91	3.72	757	25.7
24.58	3.62	545	18.5
25.28	3.52	1743	59.1
26.01	3.42	784	26.6
26.69	3.34	552	18.7
27.05	3.29	1018	34.5
27.73	3.21	449	15.2
27.97	3.19	449	15.2
28.89	3.09	488	16.5
29.28	3.05	578	19.6
30.08	2.97	423	14.3
31.14	2.87	301	10.2
31.77	2.81	421	14.3
33.08	2.71	257	8.7
33.50	2.67	286	9.7
34.28	2.61	420	14.3
35.08	2.56	275	9.3
35.72	2.51	357	12.1
36.85	2.44	308	10.5
37.64	2.39	322	10.9
38.96	2.31	248	8.4
39.56	2.28	252	8.6
40.68	2.22	249	8.4
41.20	2.19	227	7.7
42.07	2.15	219	7.4
43.85	2.06	287	9.7



24. Chromatographic purity of prodrug **1c** (HPLC)

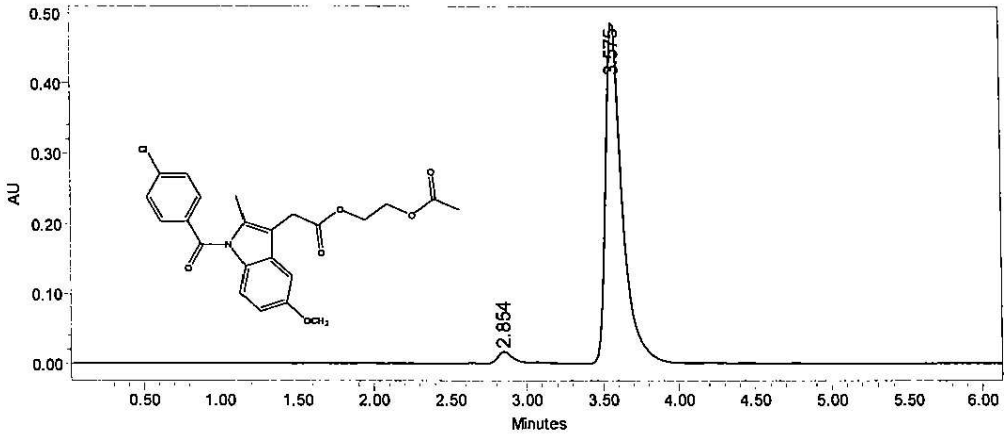
SID REACTION MONITORING

HPLC ID:PDL/HPLCW/26
COLUMN:149

STP CODE:TC014B49

SAMPLE INFORMATION

Sample Name:	SID 4007 FINAL	Acquired By:	orchid
Sample Type:	Unknown	Date Acquired:	02/08/2009 21:51:23
Vial:	22	Acq. Method Set:	FURPROFEN_MODI
Injection #:	1	Date Processed:	03/08/2009 15:27:59
Injection Volume:	0.50 ul	Processing Method:	FURPROFEN RS TC014B49
Run Time:	20.0 Minutes	Channel Name:	Wvln Ch65
Sample Set Name:	020709 FURPROFEN RM	Proc. Chnl. Descr.:	PDA 254.0 nm



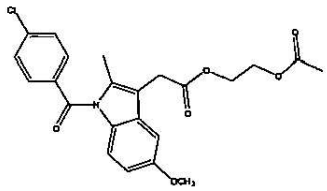
	RT	Area	% Area	Peak Lambda Max.
1	2.854	26167	0.69	193.6
2	3.575	3743219	99.31	207.6

25. UV spectrum of prodrug **1c**

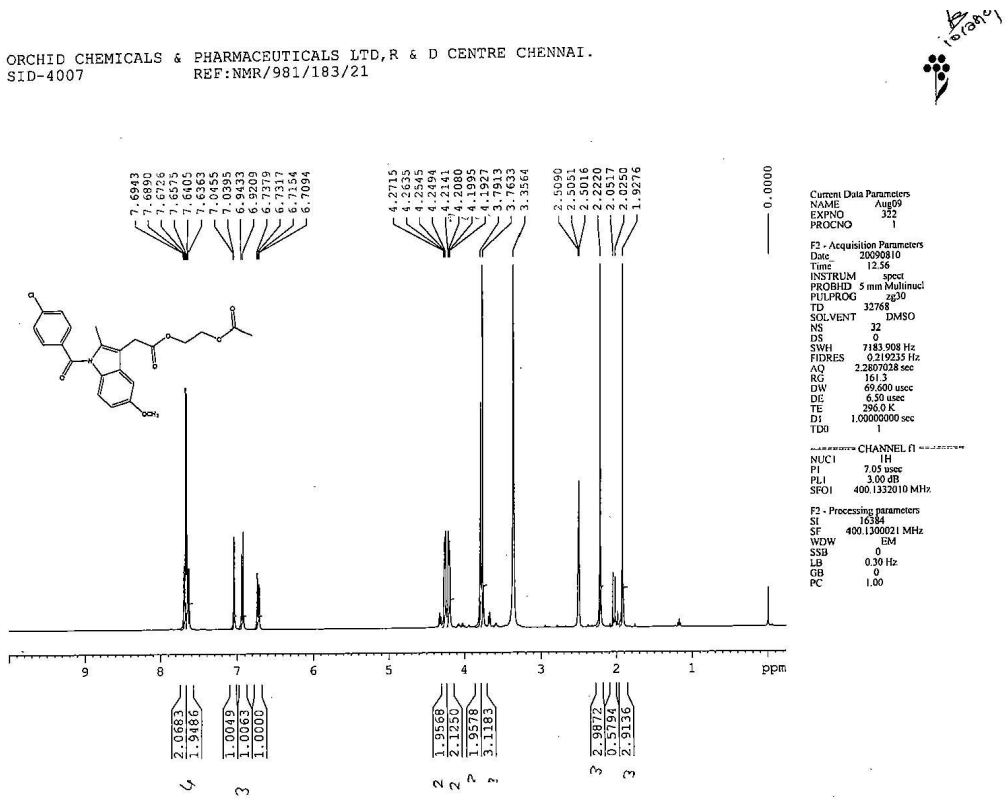
Date : 2/28/2010 Time : 14:08:15

Spectrum: SID-4007
Comment: SID-4007
Threshold: 0.0200
Abscissa units: nm
Ordinate units: A

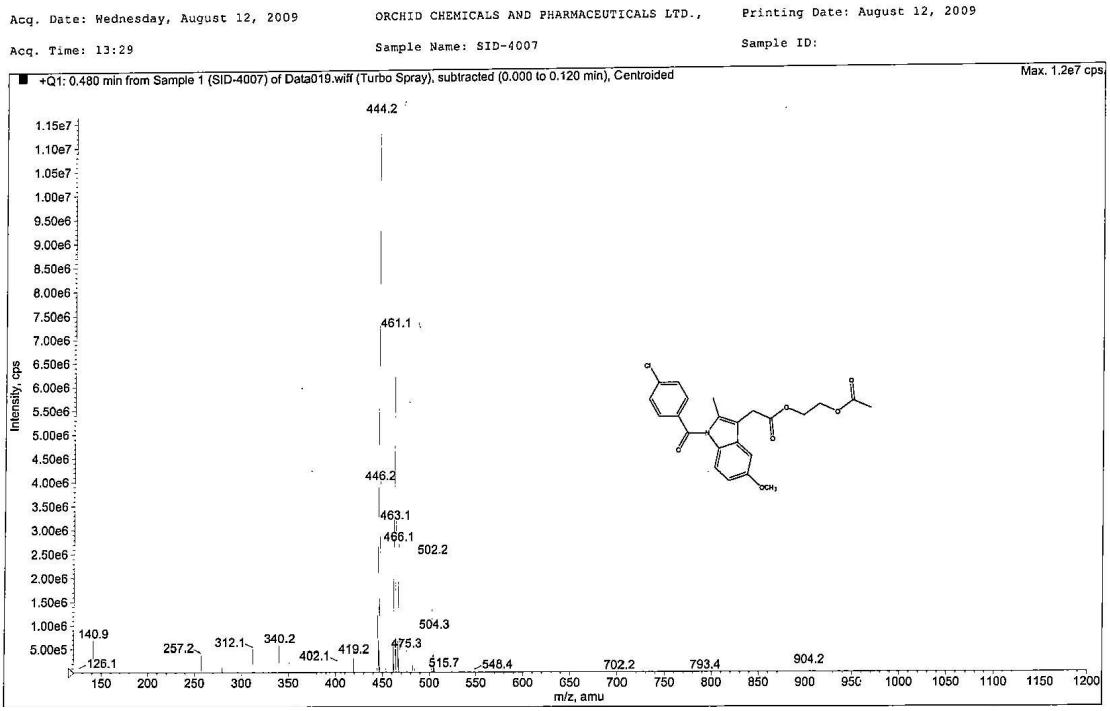
No.	Abscissa	Ordinate	Type
1	319.85	0.2589	Peak
2	271.11	0.3531	Peak
1	301.94	0.2232	Base



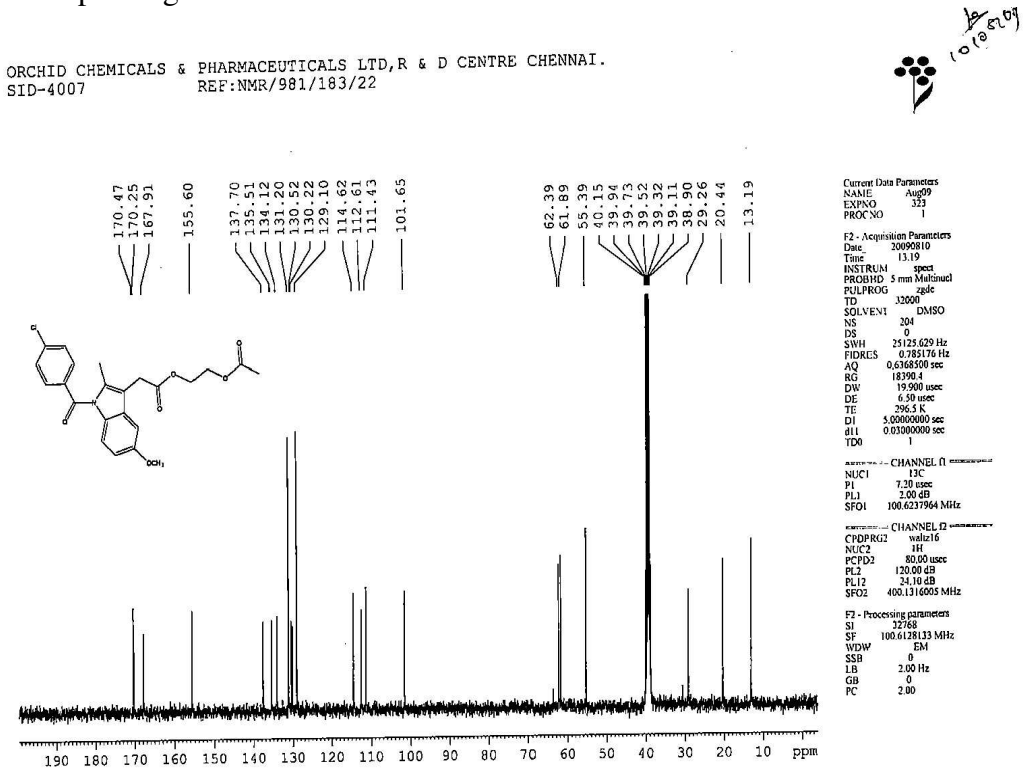
26. ^{1}H NMR of prodrug **1c**



27. Mass spectrum of **1c**

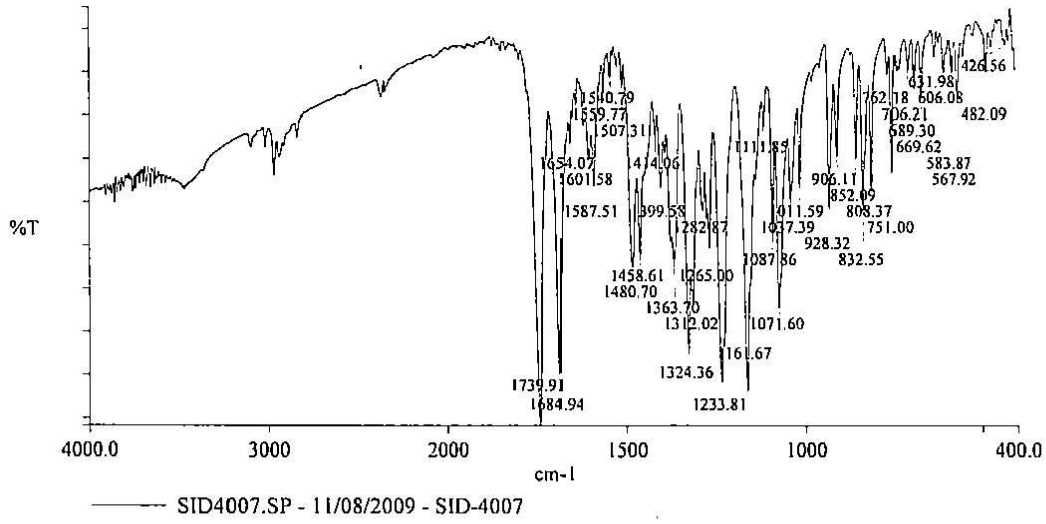


28. ^{13}C NMR of prodrug **1c**



29. IR of prodrug **1c**

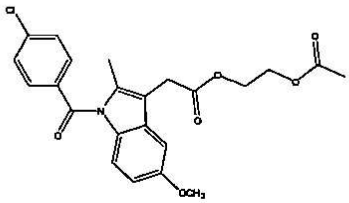
Instrument Number : FTIR - 120



SAMPLE INFORMATION

Status - c:\pel_data\spectra\aug09120\sid4007.sp

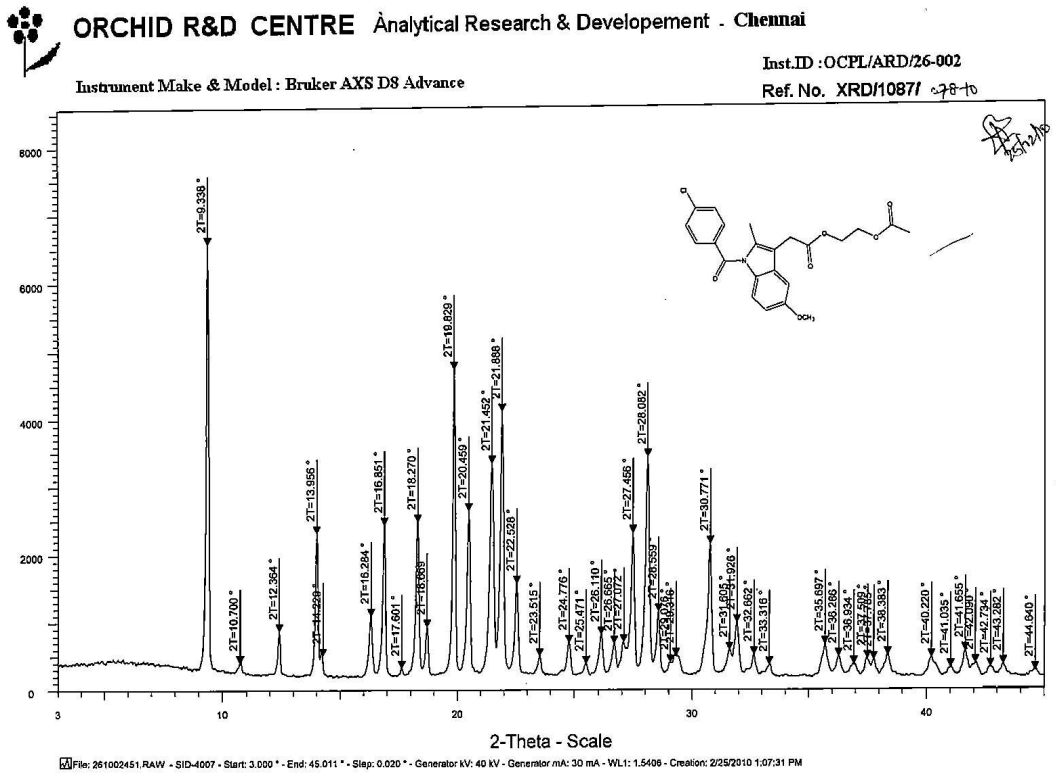
Filename: sid4007.sp
Date Created: Tue Aug 11 16:50:24 2009
Analyst: PDL
Description: SID-4007
Comments:



Abscissa: (cm-1)
Start: 4000.00
End: 400.00
Interval: -2.000000

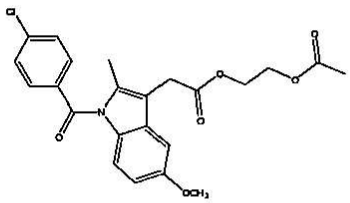
Ordinate: (%T)
Maximum: 77.37
Minimum: 29.14
Points: 1801
Printed on : Date: 11/08/2009 Time: 16:51:51

30. XRD diffractogram for prodrug **1c**



30. XRD diffractogram for prodrug **1c**

261002451.RAW SID-4007			
Angle	d value	Intensity	Intensity
2-Theta °	Angstrom	Count	%
9.34	9.46	6581	100
10.70	8.26	389	5.9
12.36	7.15	853	13
13.96	6.34	2299	34.9
14.23	6.22	482	7.3
16.28	5.44	1078	16.4
16.85	5.26	2418	36.7
17.60	5.03	297	4.5
18.27	4.85	2460	37.4
18.67	4.75	911	13.8
19.83	4.47	4723	71.8
20.46	4.34	2622	39.8
21.45	4.14	3320	50.4
21.89	4.06	4093	62.2
22.53	3.94	1553	23.6
23.52	3.78	472	7.2
24.79	3.59	668	10.1
25.47	3.49	358	5.4
26.11	3.41	790	12
26.67	3.34	658	10
27.07	3.29	675	10.3
27.46	3.25	2285	34.7
28.08	3.17	3403	51.7
28.56	3.12	1125	17.1
29.08	3.07	371	5.6
29.32	3.04	467	7.1
30.77	2.90	2119	32.2
31.61	2.83	552	8.4
31.93	2.80	960	14.6
32.66	2.74	488	7.4
33.32	2.69	335	5.1
35.70	2.51	634	9.6
36.29	2.47	454	6.9
36.93	2.43	338	5.1
37.51	2.40	416	6.3
37.79	2.38	402	6.1
38.38	2.34	470	7.1
40.22	2.24	433	6.6
41.04	2.20	283	4.3
41.66	2.17	530	8
42.09	2.15	344	5.2
42.73	2.11	290	4.4
43.28	2.09	330	5
44.64	2.03	234	3.6



31. Chromatographic purity of prodrug **1d** (HPLC)

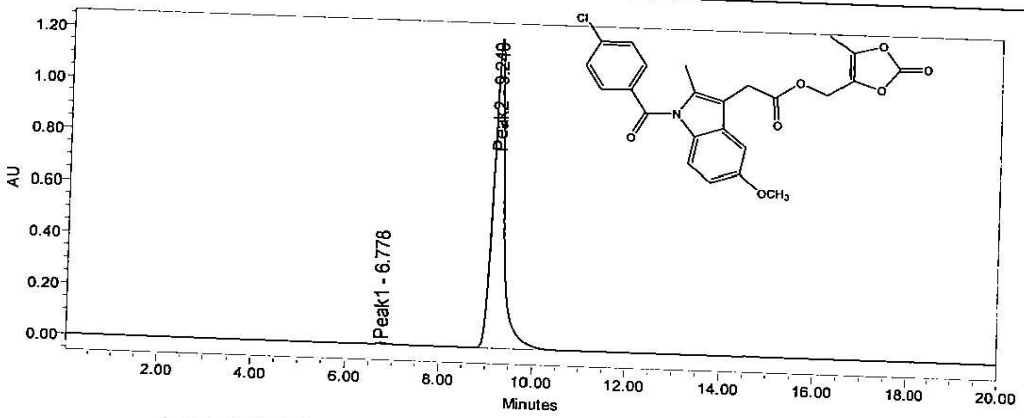
SID REACTION MONITORING

HPLC ID:PDL/HPLCW/26
COLUMN:149

STP CODE:TC014B49

SAMPLE INFORMATION

Sample Name:	SID-4009 FINAL	Acquired By:	orchid
Sample Type:	Unknown	Date Acquired:	22/08/2009 17:34:56
Vial:	21	Acq. Method Set:	FURPROFEN_MODI
Injection #:	1	Date Processed:	22/08/2009 17:57:49
Injection Volume:	10.00 ul	Processing Method:	FURPROFEN RS TC014B49
Run Time:	20.0 Minutes	Channel Name:	Wvln Ch85
Sample Set Name:	220809 FURPROFEN RS	Proc. Chnl. Descr.:	PDA 254.0 nm



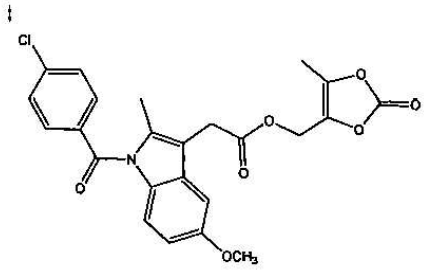
	RT	Area	% Area	Peak Lambda Max.	Name
1	6.778	92829	0.46	199.4	Peak1
2	9.240	20263239	99.54	211.2	Peak2

32. UV spectrum of prodrug **1d**

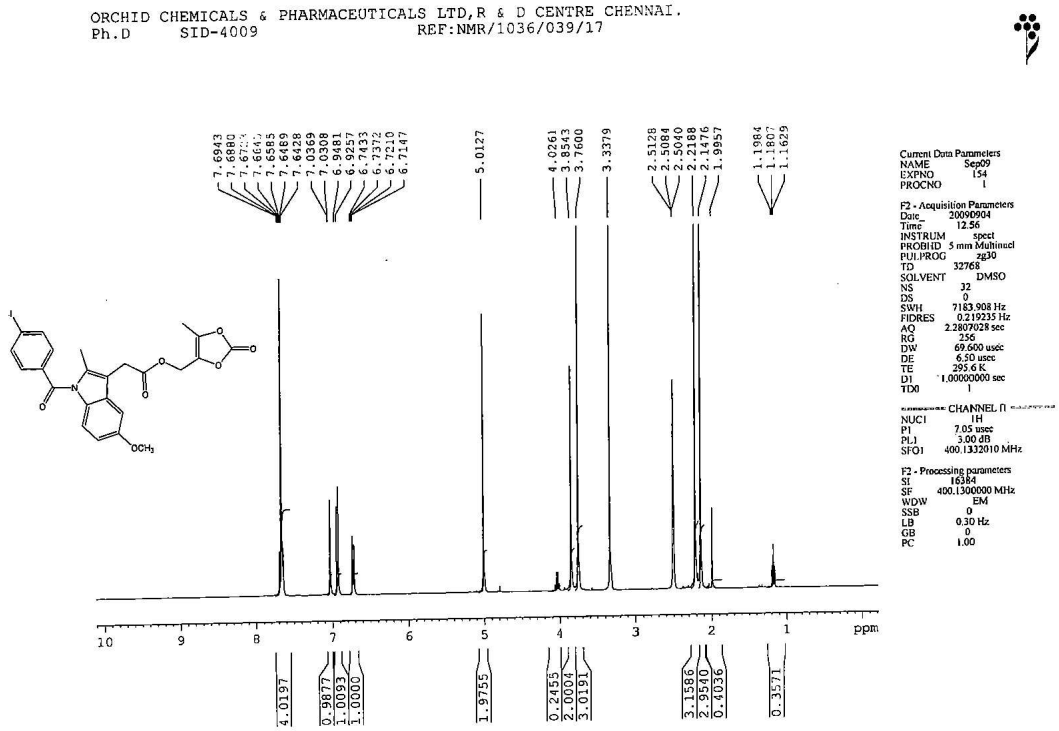
Date : 2/28/2010 Time : 14:56:45

Spectrum: SID-4009
Comment: SID-4009
Threshold: 0.0200
Abscissa units: nm
Ordinate units: A

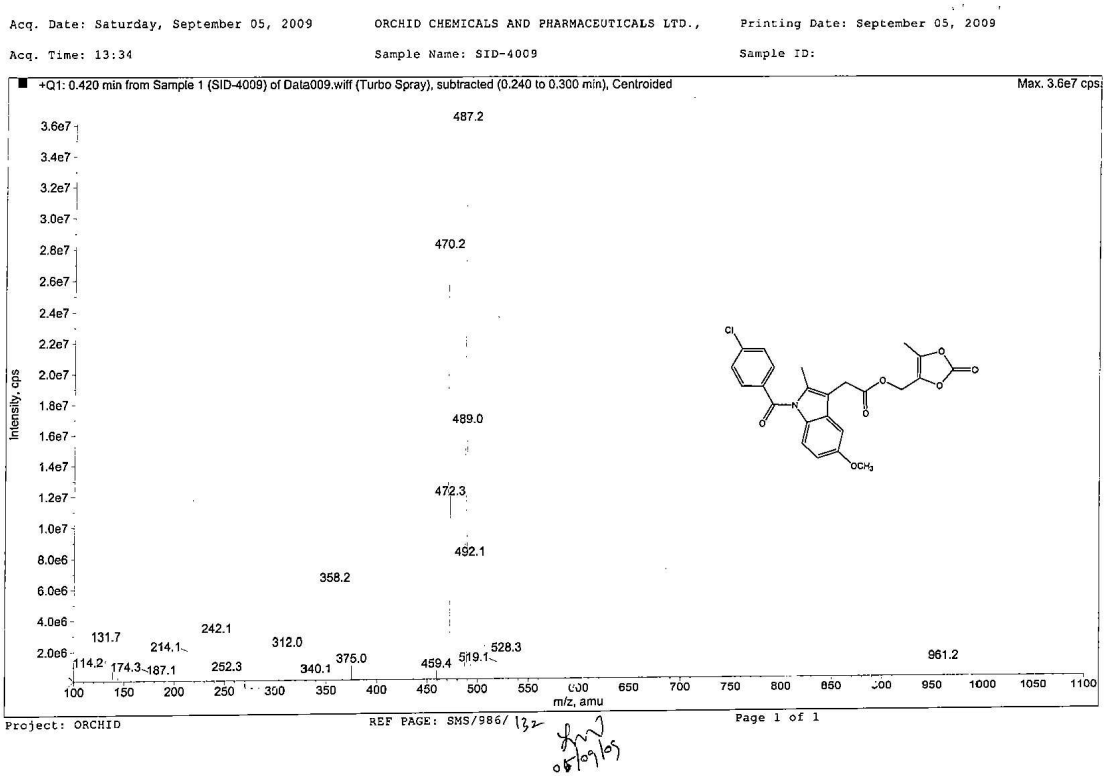
No.	Abscissa	Ordinate	Type
1	319.61	0.1986	Peak
2	268.90	0.3377	Peak
1	303.82	0.1770	Base



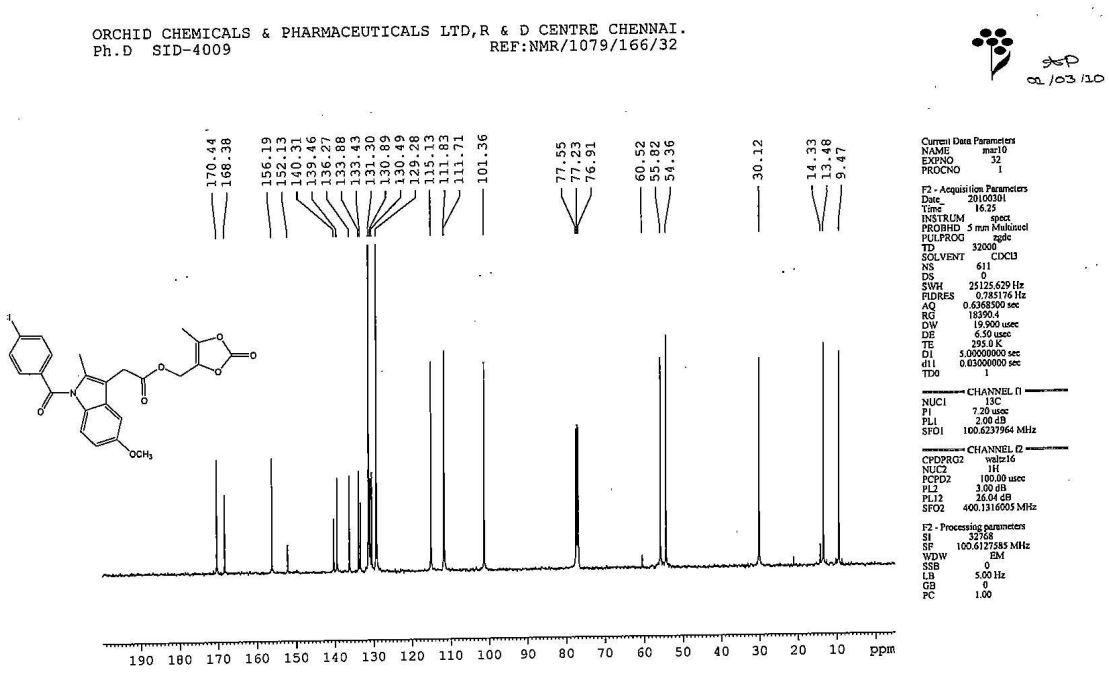
33. ^{1}H NMR of prodrug **1d**



34. Mass spectrum of **1d**



35. ^{13}C NMR of prodrug **1d**



36. IR of prodrug **1d**

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SAMPLE INFORMATION

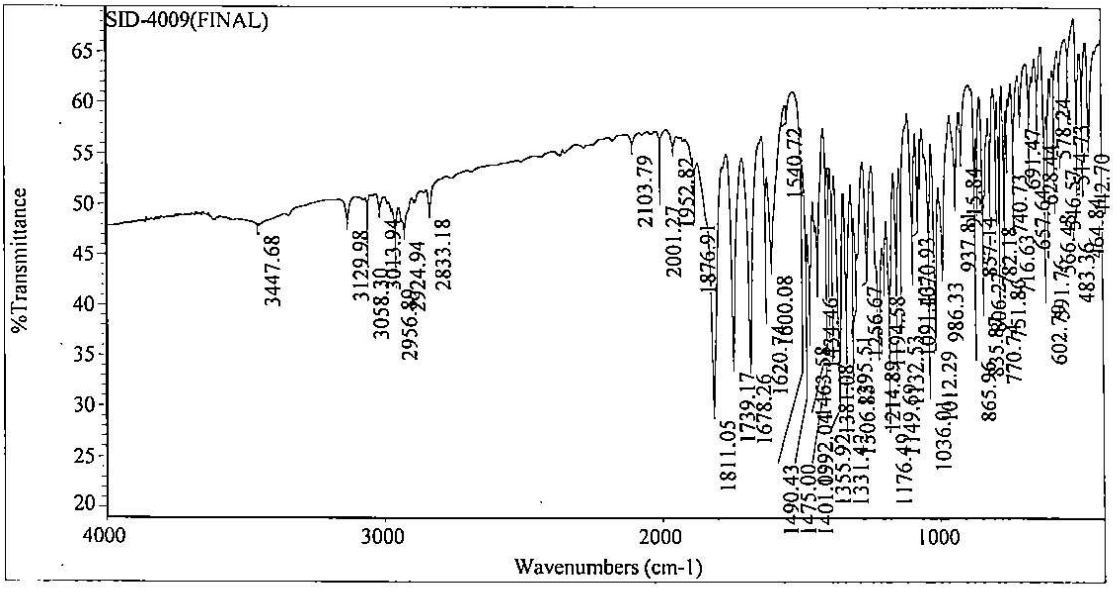
Sample Name : **SID-4009(FINAL)** Instrument No.: FTIR - 197

Description : SID-4009(FINAL)

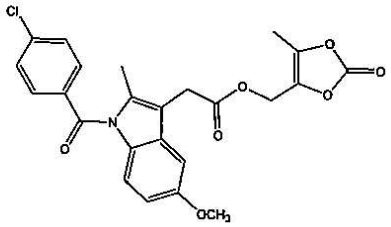
Collection time: Mon Feb 15 17:27:42 2010 (GMT+05:30)

Printed On : Mon Feb 15 17:30:51 2010 (GMT+05:30) User name: ARULMURUGAN P.

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SID-4009(FINAL)



Number of sample scans: 32
Number of background scans: 32
Resolution: 4.000
Sample gain: 1.0
Mirror velocity: 0.6329
Aperture: 100.00

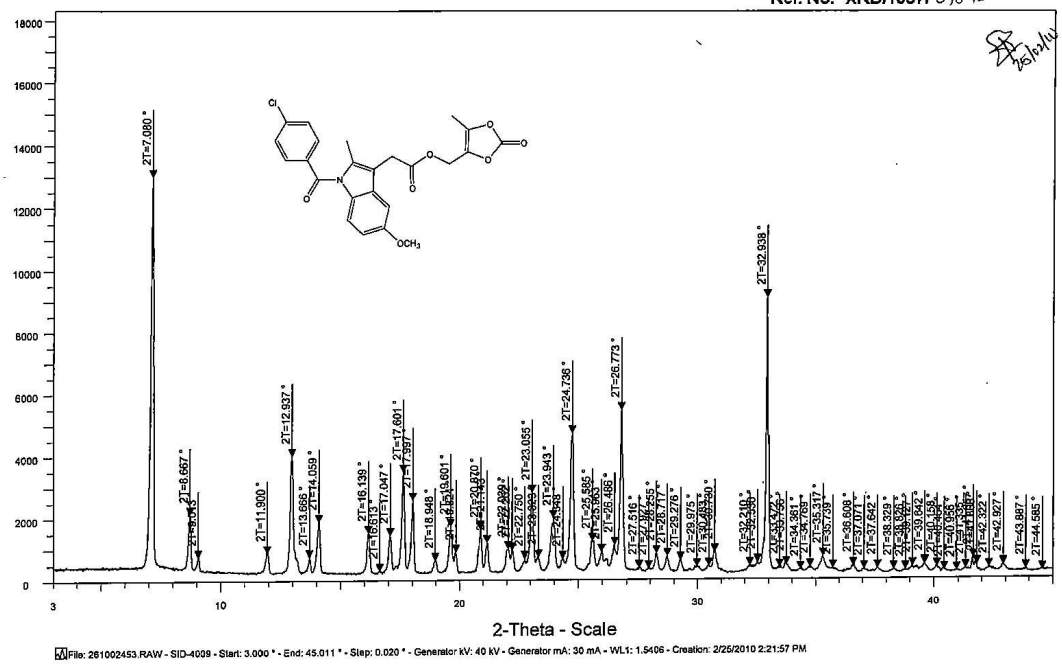
37. XRD diffractogram for prodrug **1d**

ORCHID R&D CENTRE Analytical Research & Developement - **Chennai**

Instrument Make & Model : Bruker AXS D8 Advance

Inst.ID : OCPL/ARD/26-002

Ref. No. XRD/1087/ 096-12-



File: 261002453.RAW - SID-4009 - Start: 3.000 ° - End: 45.011 ° - Step: 0.020 ° - Generator kV: 40 kV - Generator mA: 30 mA - WL1: 1.5406 - Creation: 2/25/2010 2:21:57 PM

37. XRD diffractogram for prodrug **1d**

261002453.RAW 6ID-4009			
Angle	d value	Intensity	Intensity
2-Theta °	Angstrom	Count	%
7.08	12.47	13023	100
8.87	10.19	2132	16.4
9.01	9.80	734	5.6
11.90	7.43	865	6.6
12.94	6.84	4007	30.8
13.67	6.47	721	5.5
14.06	6.29	1870	14.4
16.14	5.49	1498	11.5
16.61	5.33	282	2.2
17.05	5.20	1426	10.9
17.60	5.03	3477	26.7
18.00	4.92	2568	19.7
18.95	4.68	606	4.7
19.50	4.53	1716	13.2
19.82	4.47	871	6.7
20.87	4.25	1595	12.2
21.14	4.20	1171	9
22.04	4.03	973	7.5
22.20	4.00	919	7.1
22.75	3.91	659	5.1
23.06	3.85	2802	21.5
23.32	3.81	690	5.3
23.94	3.71	1975	15.2
24.35	3.65	643	4.9
24.74	3.60	4707	36.1
25.59	3.48	1218	9.4
25.90	3.43	859	6.7
26.49	3.36	1084	8.3
26.77	3.33	5455	41.8
27.52	3.24	351	2.7
27.93	3.19	338	2.6
28.28	3.16	808	6.2
28.72	3.11	712	5.5
29.28	3.05	589	4.5
29.98	2.98	386	3
30.48	2.93	401	3.1
30.73	2.91	847	6.5
32.21	2.78	411	3.2
32.54	2.75	511	3.9
32.84	2.72	9091	69.8
33.47	2.67	356	2.7
33.78	2.65	443	3.4
34.38	2.61	291	2.2
34.77	2.58	359	2.8
35.32	2.54	688	5.3
35.74	2.51	319	2.4
36.81	2.45	395	3
37.07	2.42	289	2.2
37.64	2.39	311	2.4
38.33	2.35	270	2.1
38.83	2.32	259	2
39.13	2.30	365	2.8
39.64	2.27	433	3.3
40.16	2.24	351	2.7
40.46	2.23	242	1.9
40.96	2.20	218	1.7
41.34	2.18	288	2.2
41.66	2.17	608	4.7
41.81	2.16	402	3.1
42.32	2.13	316	2.4
42.93	2.11	386	3
43.88	2.06	262	2
44.59	2.03	215	1.7

