SUPPORTING INFORMATION

One-pot synthesis of new fused 4,5-bridged 1,2,5-triazepine-3,6-diones, 1,2,5-triazepine-3,7-diones heterocycles by Petasis reaction:

Subhasish Neogi, Amrita Roy and Dinabandhu Naskar*

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XV. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Benzyl-5-(3-fluoro-4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione][Table 1, 2m(A)]

XVI. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Benzyl-5-(3-fluoro-4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione][Table 1, 2m(B)]

XVII. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Benzyl-4-naphthalen-1-yl-hexahydro-3a,6,7-triaza-azulene-5,8-dione][Table 2, 4a(A)]

XVIII. ^{1}H NMR, ^{13}C NMR, and LCMS of [6-Benzyl-4-naphthalen-1-yl-hexahydro-3a,6,7-triaza-azulene-5,8-dione][Table 2, 4a(B)]

XIX. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Benzyl-4-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-5,8-dione][Table 2, 4b]

XX. ^{1}H NMR, ^{13}C NMR and LCMS of [6-Cyclohexylmethyl-4-phenyl-hexahydro-3a,6,7-triaza-azulene-5,8-dione][Table 2, 4c]

XXI. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Cyclohexylmethyl-4-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-5,8-dione][Table 2, 4d]

XXII. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [7-Benzyl-6-(4-methoxy-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]][Table 3, 6a]

XXIII. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [7-Benzyl-6-phenyl-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione][Table 3, 6b]

XXIV. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [6-Benzo[1,3]dioxol-5-yl-7-benzyl-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione][Table 3, 6c]

XXV. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [7-Benzyl-6-(3-fluoro-4-methoxy-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]][Table 3, 6d]

XXVI. ^{1}H NMR, ^{13}C NMR, LCMS and HPLC of [7-Benzyl-6-(4-methoxy-2-methyl-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione][Table 3, 6e]

I. Experimental Section:

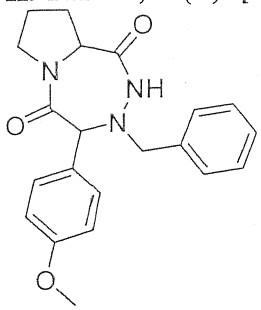
All starting material were purchased and used without further purification. All compounds are commercially available unless otherwise specified by a reference. ^{1}H NMR spectra were obtained using 400-MHz spectrometer. Analytical thin layer chromatography (TLC) was conducted on aluminium backed silica gel plates (0.2 mm). Developed plates were visualized with UV light or a ninhydrin solution. Silica gel column chromatography was performed using silica gel (60-120 mesh).

General procedure for the synthesis of 4,5-bridged 1,2,5-triazepine-3,6-diones :

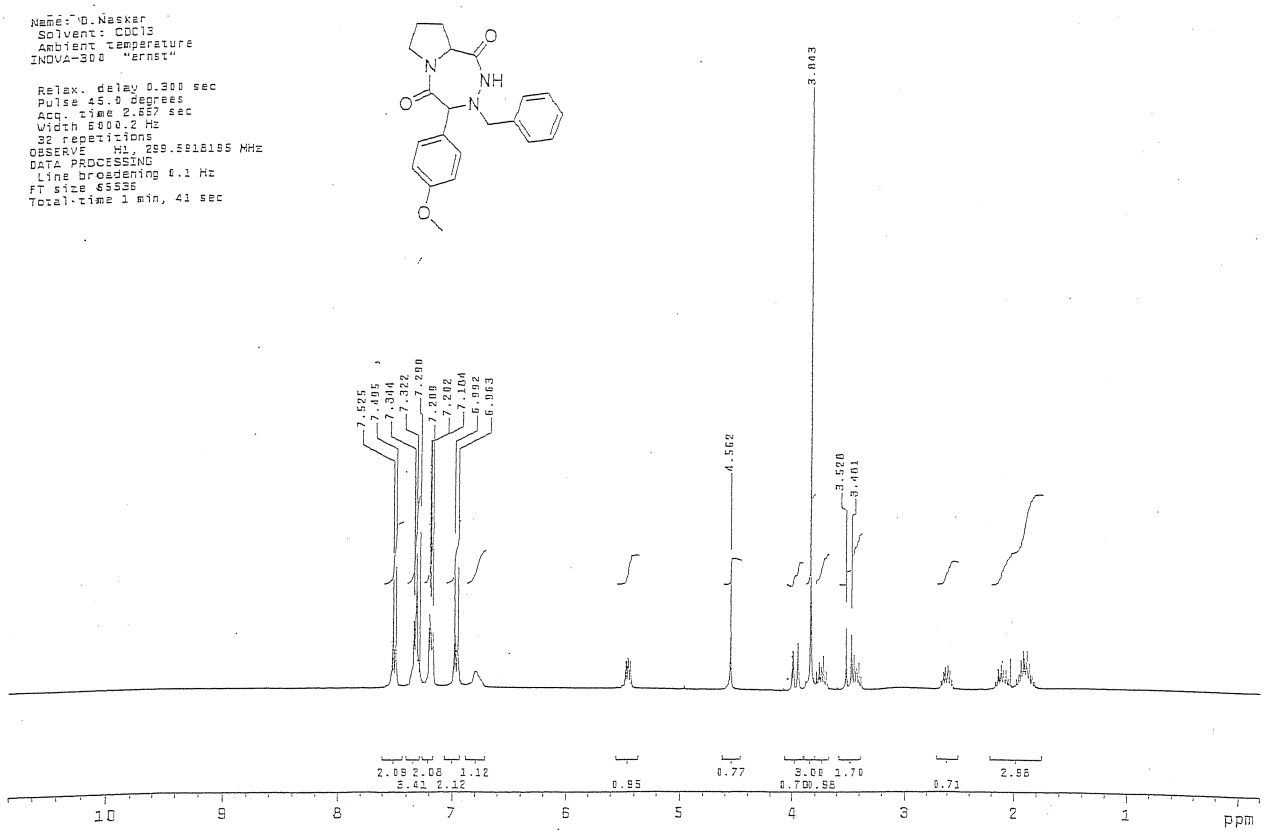
To a stirred mixture of glyoxylic acid monohydrate (184 mg, 2 mmol) in DCM (5 mL) was added *N*-1-Boc-*N*-2-(benzyl)-hydrazine (446 mg, 2 mmol) followed by *p*-methoxyphenyl boronic acid (304 mg, 2 mmol). The resulting mixture was stirred at ambient temperature for 24 h and to this solution, were added *L*-proline methyl ester hydrochloride (331 mg, 2 mmol), HBTU (759 mg, 2 mmol), DIEA (774 mg, 6

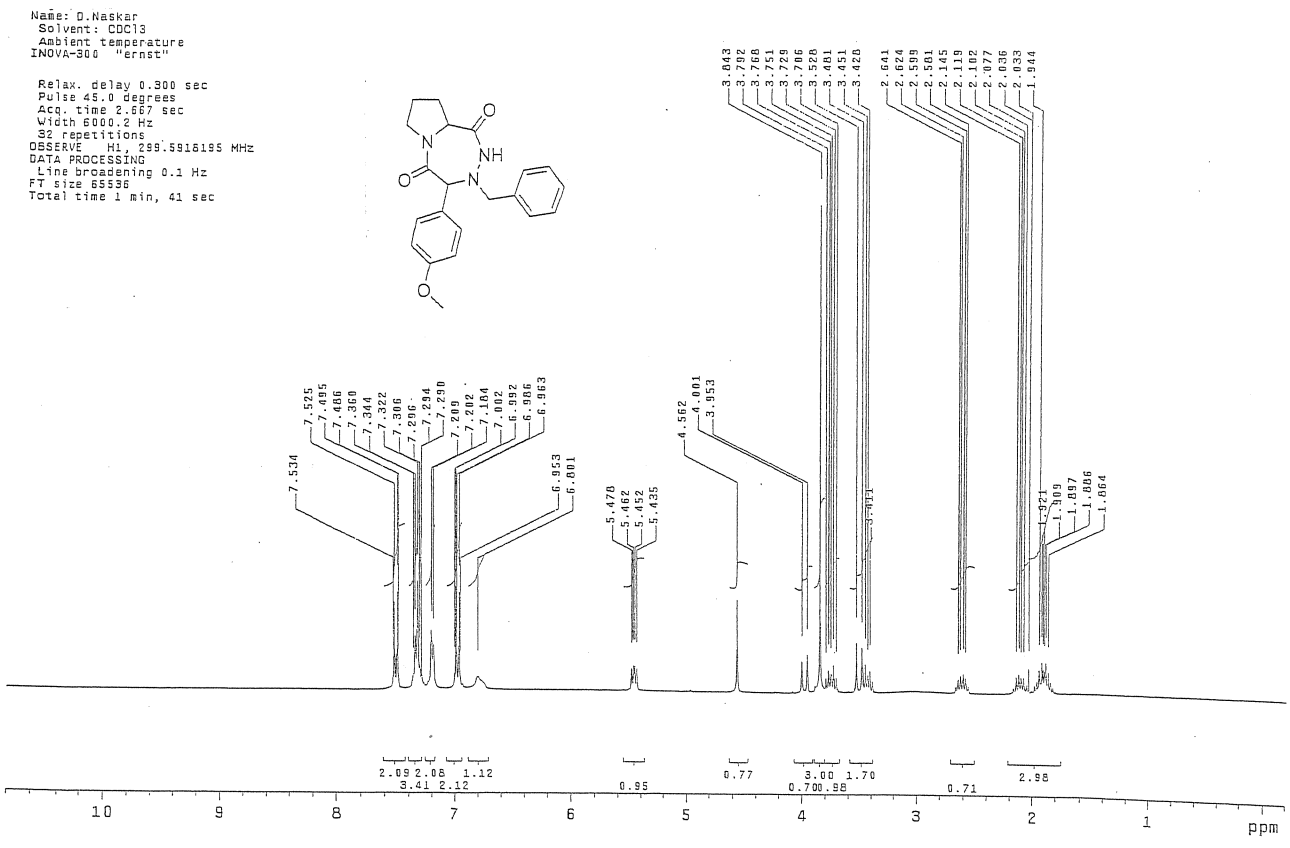
mmol), the reaction mixture was stirred at ambient temperature for 4 h and after this time, the solvent was removed and dried under reduced pressure. To this reaction mixture was added 4.0M HCl (4 mL) in dioxane. The resulting mixture was stirred at ambient temperature for 3h. The solvent was evaporated and dried under reduced pressure. To a solution of this reaction mixture in toluene (20 mL) was added HOAc (0.20 mL) and refluxed for overnight. The solvent was removed and the crude product was purified by HPLC to afford 310 mg (42%) of **2a** as a white solid. The diastereomers (**A** and **B**) have been separated by preparative HPLC; [Polaris C18 column (250 x 500 mm, 10 micron particle size), mobile phase 0.1% aqueous TFA/CH_3CN linear gradient over 55 min, 60 mL/min]; analytical HPLC: Polaris C18 column (4.6 x 250 mm, 3 micron particle size), mobile phase 0.1% aqueous phosphoric acid/CH_3CN linear gradient over 30 min, 1 mL/min, one peak detected by ELS and UV at 220 nm, t_R =5.64 (**A**). another peak detected by ELS and UV at 220 nm, t_R =7.66 (**B**).

II. Table 1, 2a(A): [6-Benzyl-5-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid, m.p (Met-TempII): 108-109°C (uncorrected); $[\alpha]_D$ -30.90 (c=0.488, $CHCl_3$, at 20°C); ^1H NMR ($CDCl_3$, 300 MHz): δ = 1.86-2.14 (m, 3H), 2.63 (m, 1H), 3.42 (m, 1H), 3.53 (d, J= 14.1 Hz, 1H), 3.75 (m, 1H), 3.84 (s, 3H), 3.98 (d, J= 14.4 Hz, 1H), 4.56 (s, 1H), 5.46 (m, 1H), 6.80 (br. s, 1H), 6.99 (d, J= 8.7 Hz, 2H), 7.21 (m, 2H), 7.34 (m, 3H), 7.53 (d, J= 9 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): 22.88, 29.23, 48.55, 55.73, 57.32, 75.96, 115.11, 128.45, 129.17, 129.83, 130.19, 131.39, 135.36, 160.38, 169.79, 172.57; LCMS (ELSD): 366.1 ($M+H^+$); HRMS: 366.180917 [Calcd for $C_{21}H_{24}N_3O_3$ 366.181767 $(M+H)^+$].

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F2 - Processing parameters

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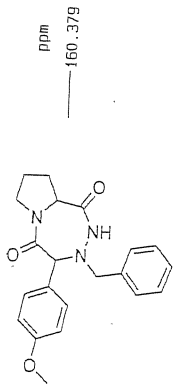

Current Data Parameters

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PROCNO	1

F2 - Acquisition Parameters

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PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	24999
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
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RG	5792.6
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DE	4.50 usec
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D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777800 MHz

========== CHANNEL f2 ==========

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1D NMR plot parameters

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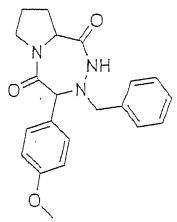

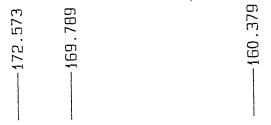

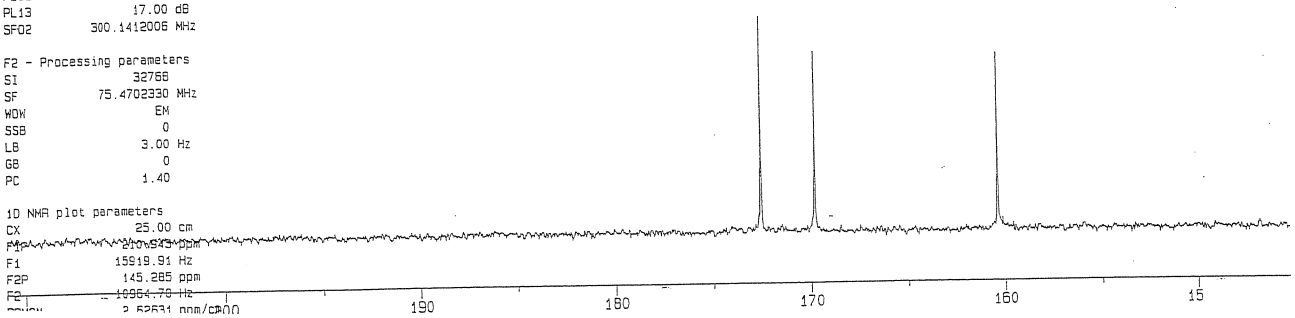

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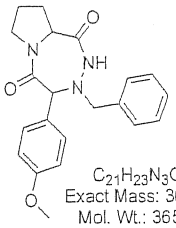

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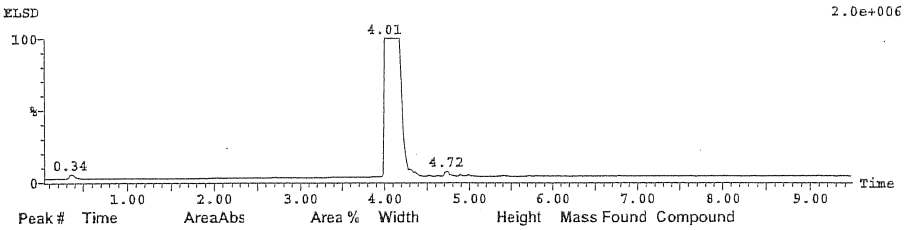
Mol. Wt.: 365.43



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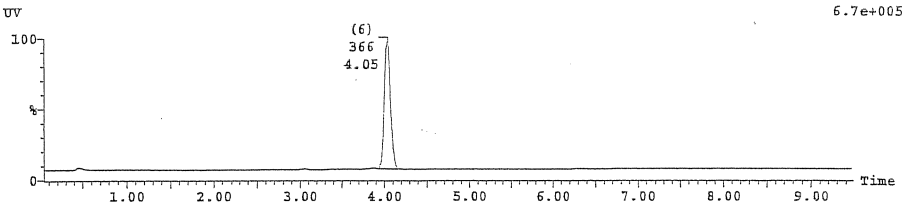

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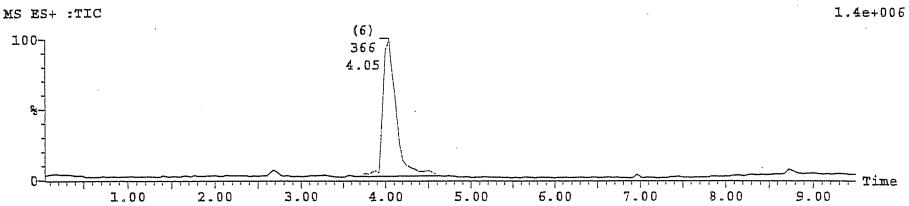

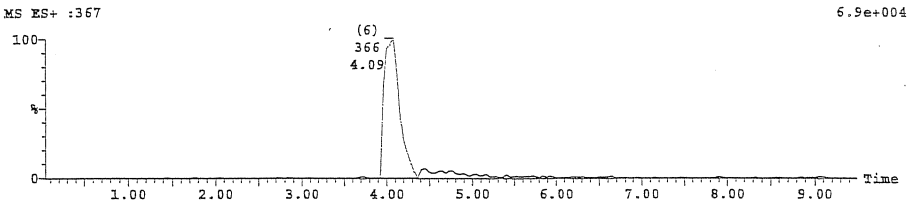

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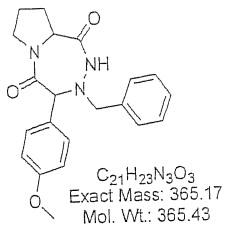

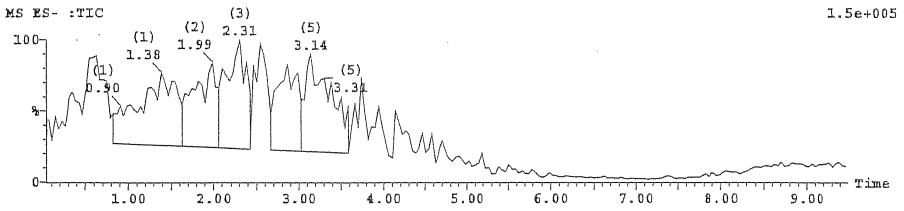

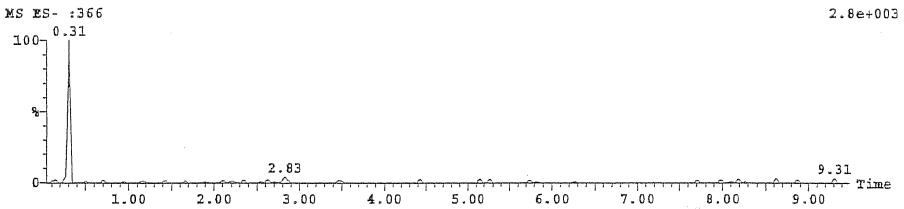

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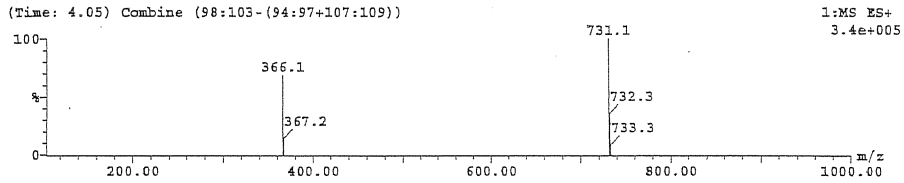

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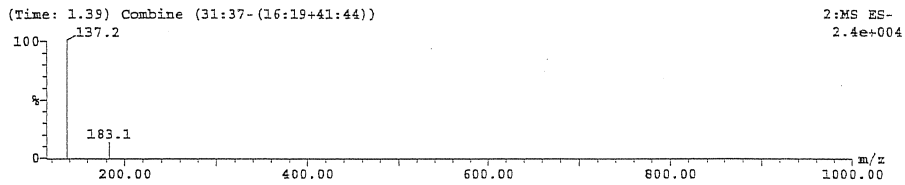

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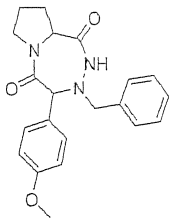

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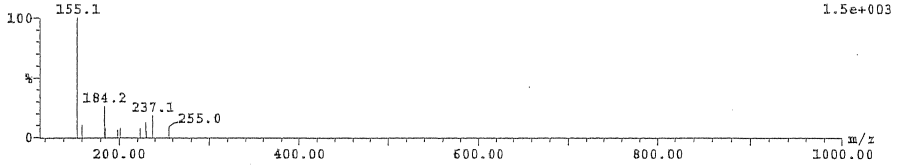

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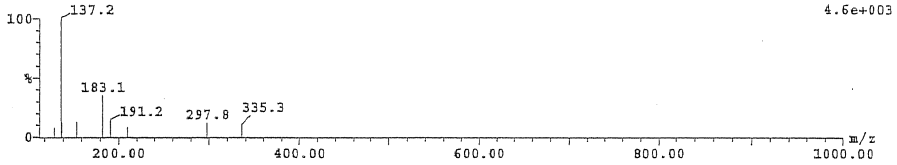

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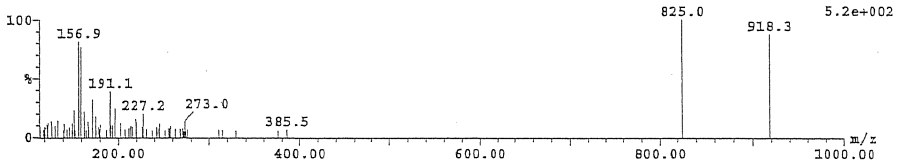

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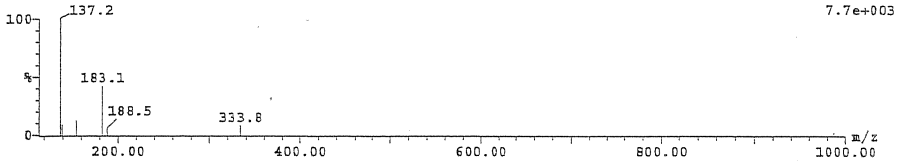

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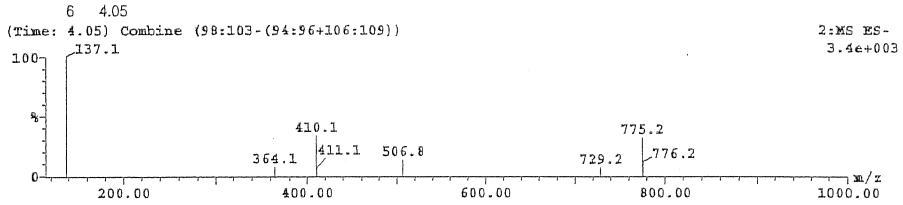

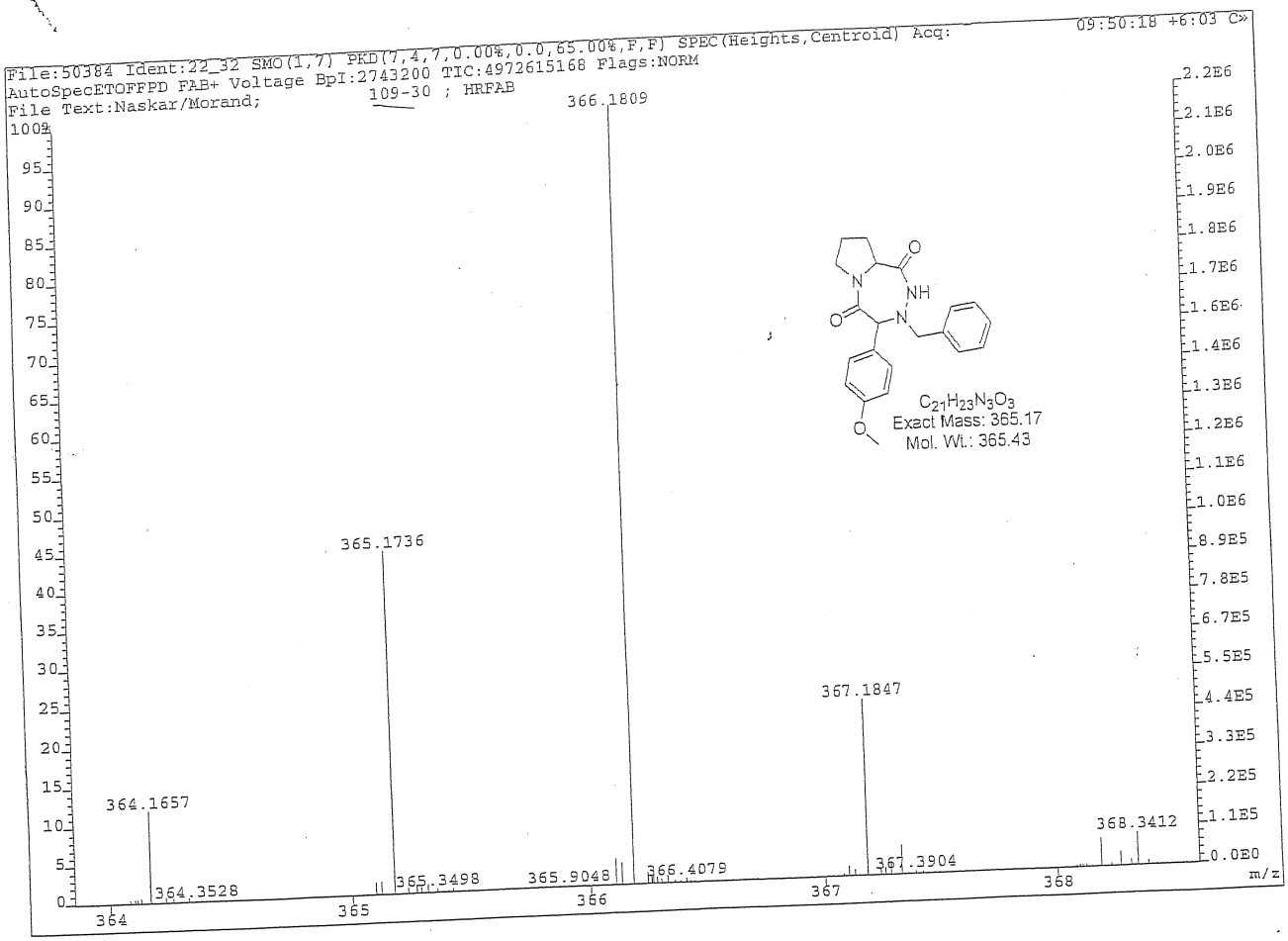
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Exact Mass: 365.17
Mol. Wt.: 365.43
m/z

Elemental Composition

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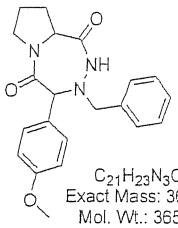

$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

29(A)

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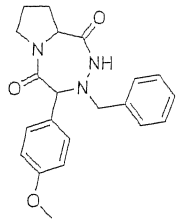

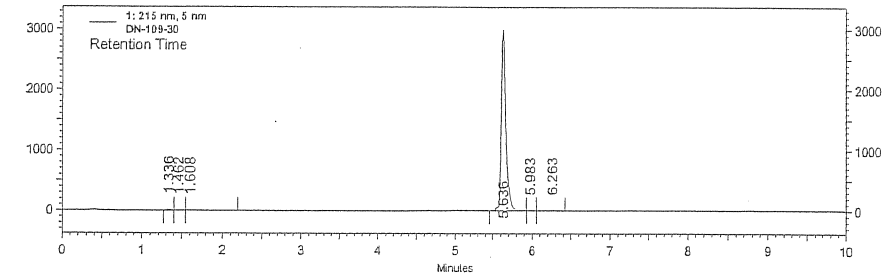

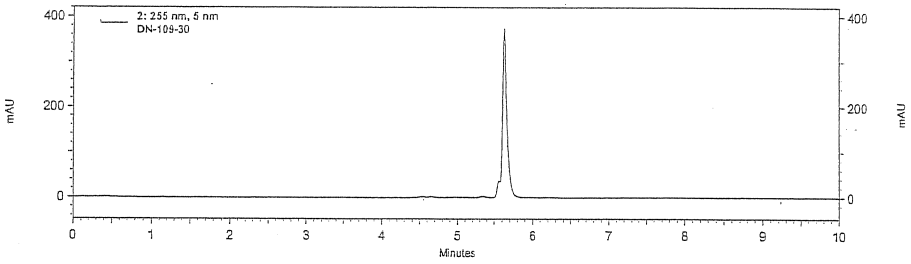

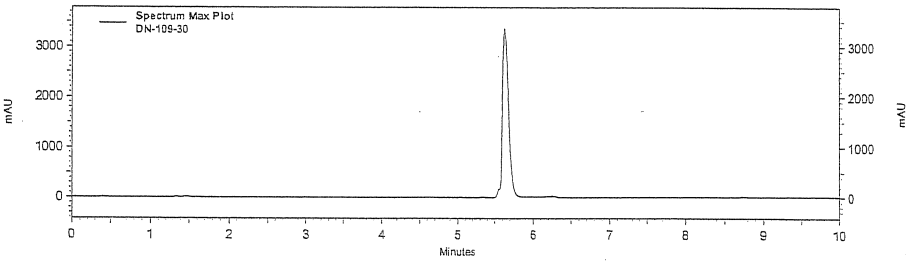

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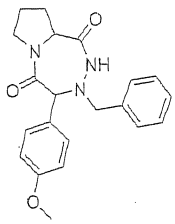
VV	1.462	65206	0.51
VV	1.608	68817	0.54
VB	5.636	12351533	96.97
SV	5.983	50137	0.39
VV	6.263	129708	1.02
VV			
Totals		12737463	100.00



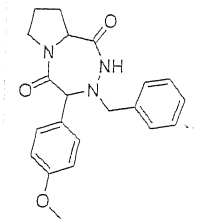
Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\c18short10min.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

Area Percent Report

Data File: D:\Public\SYSTEM2\DN-109-30.dat

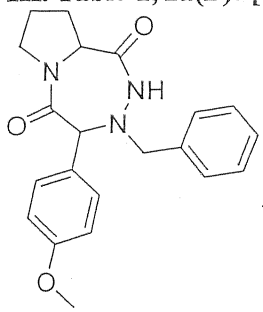
2: 255 nm, 5 nm
Results (Original)

Name	Retention Time	Area	Area Percent	Integration Codes
	5.563	89013	5.57	VS
	5.633	1510406	94.43	SV
Totals		1599419	100.00	

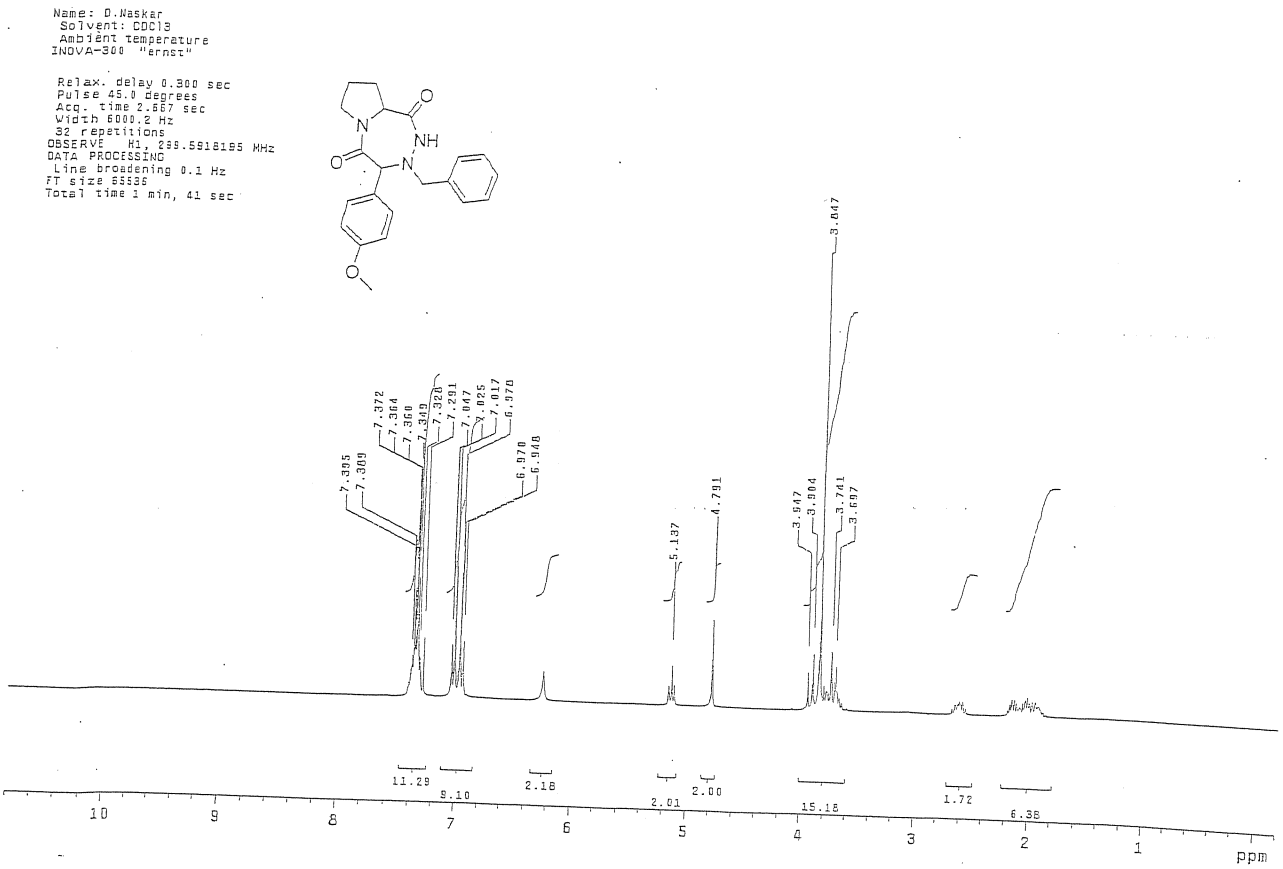


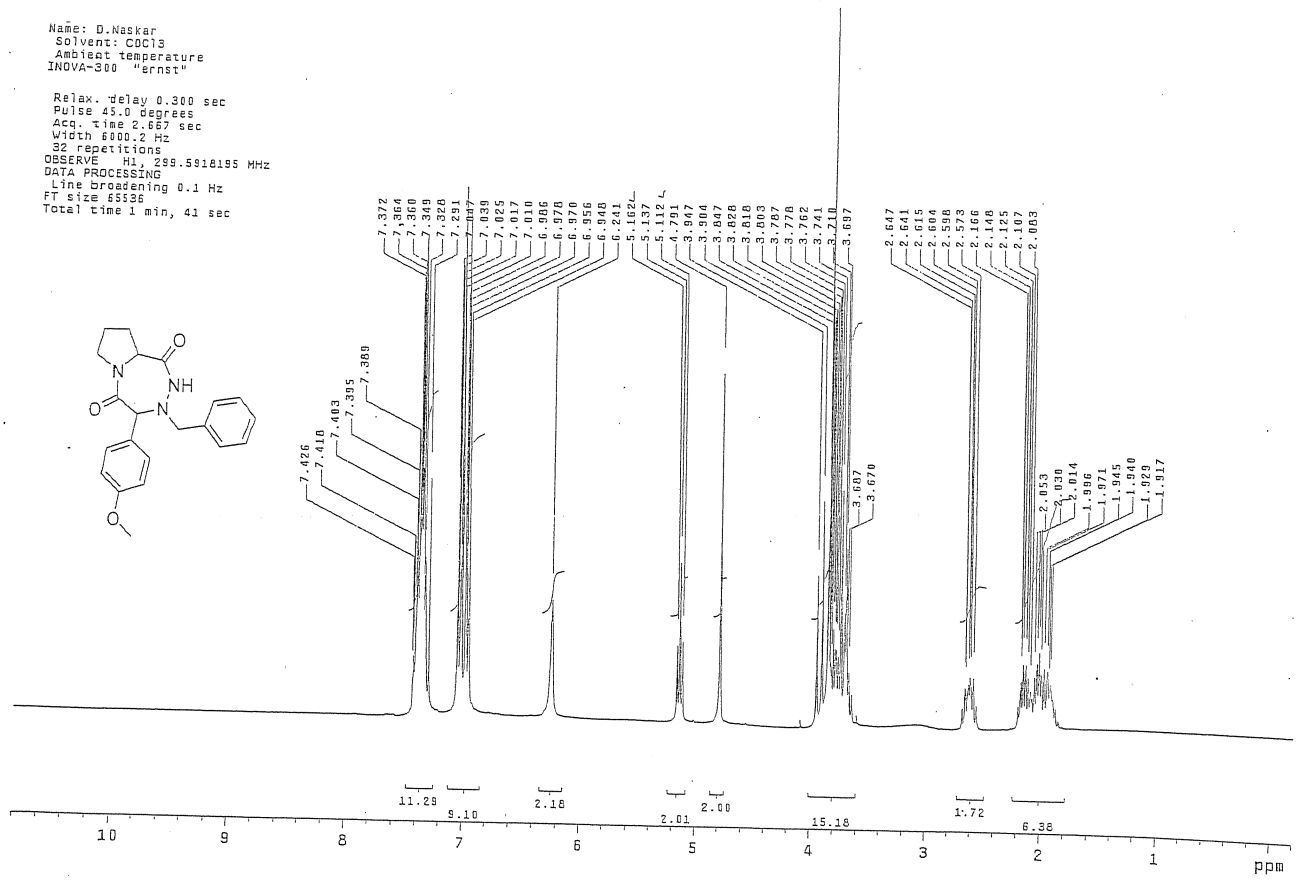
Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\c18short10min.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

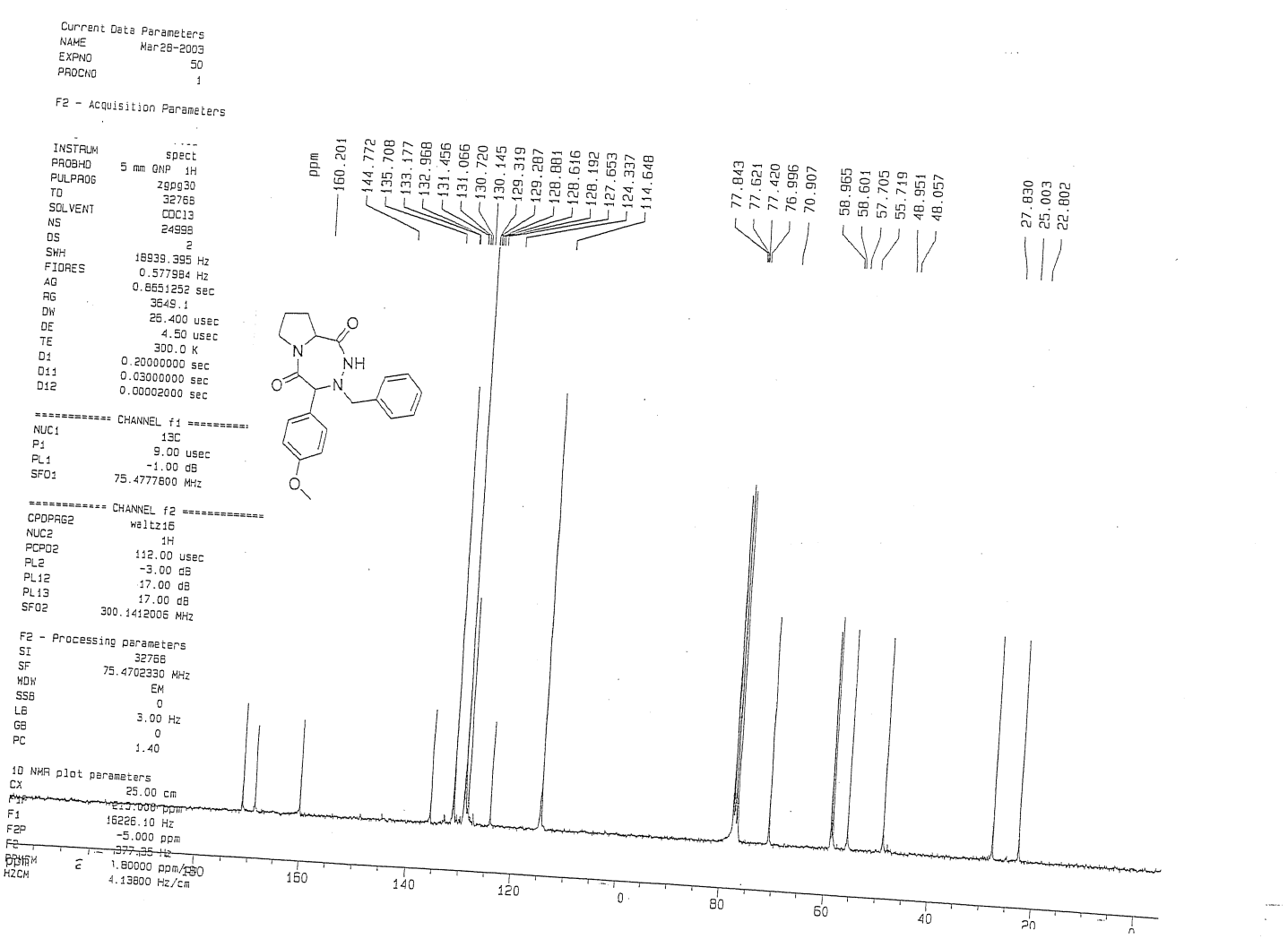
III. Table 1, 2a(B): [6-Benzyl-5-(4-methoxy-phenyl)-hexahydro-3a,6,7- triaza-azulene e-4,8-dione]



White solid, m.p (Met-TempII): 179-180°C (uncorrected); $[\alpha]_D$ -29.959 (c=0.484, $CHCl_3$, at 20°C); ^1H NMR ($CDCl_3$, 300MHz): δ = 1.92-2.2 (m, 3H), 2.64 (m, 1H), 3.67 (m, 1H), 3.72 (d, J = 13.2 Hz, 1H), 3.77 (m, 1H), 3.85 (s, 3H), 3.93 (d, J= 12.9 Hz, 1H), 4.79 (s, 1H), 5.14 (m, 1H), 6.24 (br. s, 1H), 6.96 (d, J= 9 Hz, 2H), 7.04 (d, J= 9 Hz, 2H), 7.29-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$, 75MHz): 22.80, 27.83, 48.95, 55.72, 58.60, 58.97, 70.91, 114.65, 124.34, 128.62, 129.29, 129.32, 131.07, 132.97, 160.20, 169.09, 171.49; LCMS (ELSD): 366.1 ($M+H^+$); HRMS: 366.180545 [Calcd for $C_{21}H_{24}N_3O_3$ 366.181767 $(M+H)^+$].




Name: D.Naskar
Solvent: CDCl3
Ambient temperature
INOVA-300 "ernst"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 2.667 sec
Width 6000.2 Hz
32 repetitions
OBSERVE H1, 299.5918195 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 41 sec
O
N
NH
O
N
O
7.426
7.410
7.403
7.395
7.369
7.372
7.364
7.360
7.349
7.328
7.291
7.047
7.039
7.025
7.017
7.010
6.986
6.978
6.970
6.956
6.948
6.241
5.162
5.137
5.112
4.791
3.947
3.904
3.847
3.828
3.818
3.803
3.787
3.778
3.762
3.741
3.710
3.697
3.687
3.670
2.647
2.641
2.615
2.604
2.598
2.573
2.166
2.148
2.125
2.107
2.083
2.053
2.030
2.014
1.996
1.971
1.945
1.940
1.929
1.917
11.29
9.10
2.18
2.01
2.00
15.18
1.72
6.38
10
9
8
7
6
5
4
3
2
1
ppm


Current Data Parameters
NAME Mar28-2009
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 24998
DS 2
SWH 18939.395 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 3649.1
DW 26.400 usec
DE 4.50 usec
TE 300.0 K
D1 0.20000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -1.00 dB
SFO1 75.4777800 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 112.00 usec
PL2 -3.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 300.1412006 MHz
F2 - Processing parameters
SI 32768
SF 75.4702330 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1 16226.10 Hz
F2P -5.000 ppm
F2 -377.35 Hz
PPMCM 1.80000 ppm/cm
HZCM 4.13600 Hz/cm
ppm
160.201
144.772
135.708
133.177
132.968
131.456
131.066
130.720
130.145
129.319
129.207
128.881
128.616
128.192
127.653
124.337
114.648
77.843
77.621
77.420
76.996
70.907
58.965
58.601
57.705
55.719
48.951
48.057
27.830
25.003
22.802
NH
O
O
N
N
O
200
150
140
120
0
80
60
40
20

Current Data Parameters
NAME Mar28-2003
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	24998
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	3649.1
DW	26.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777800 MHz

========== CHANNEL f2 ==========

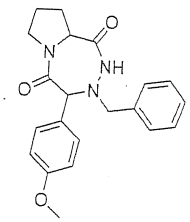
CPDPRG2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412006 MHz

F2 - Processing parameters

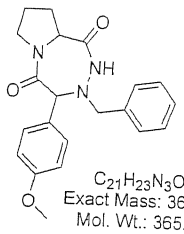
SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

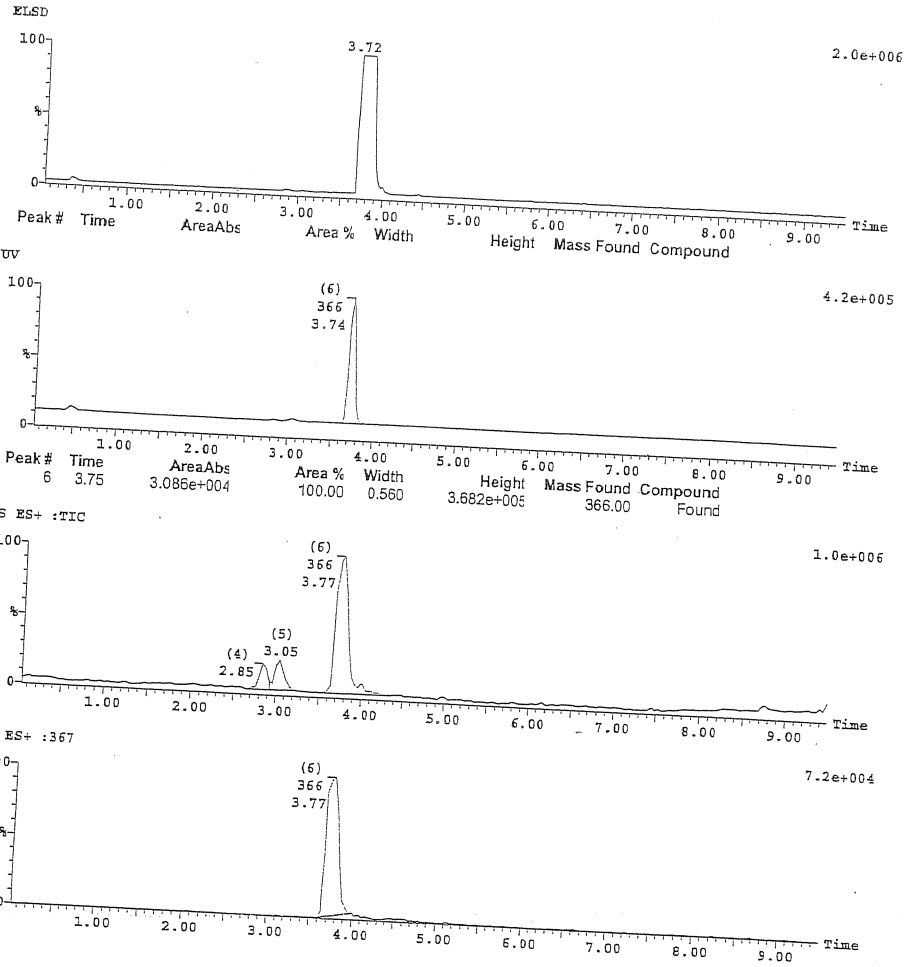
CX	25.00 cm
F1P	213.678 ppm
F1	16277.25 Hz
F2P	147.810 ppm
F2	11155.23 Hz
PPMCM	8.71470 ppm/cm
HZCM	657.87897 Hz/cm



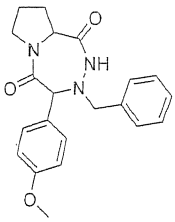
Sample Report (continued):

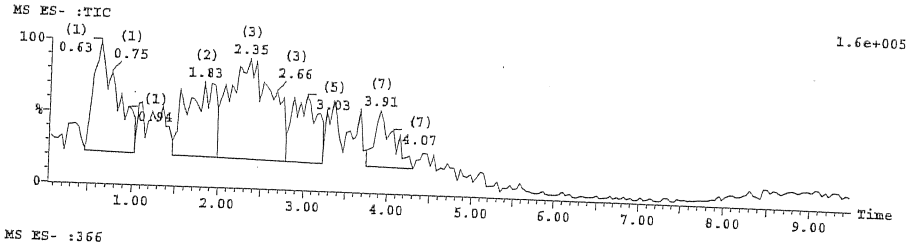


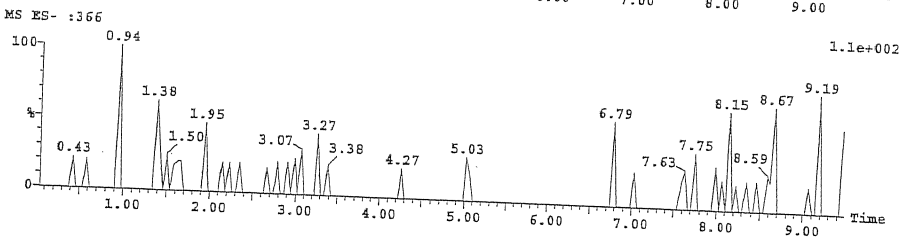
$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43



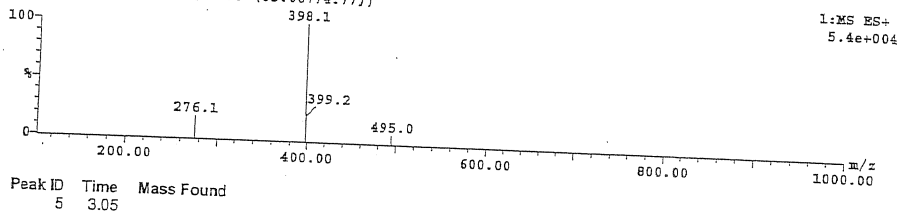
Sample Report (continued):



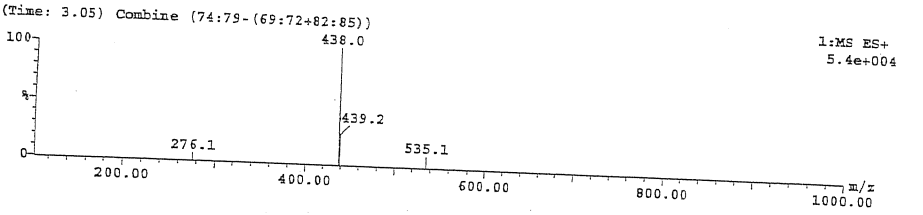




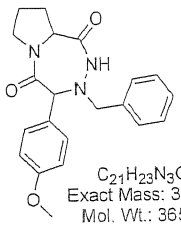
Peak ID Time Mass Found
4 2.85
(Time: 2.85) Combine (69:73-(63:66+74:77))



Peak ID Time Mass Found
5 3.05
(Time: 3.05) Combine (74:79-(69:72+82:85))

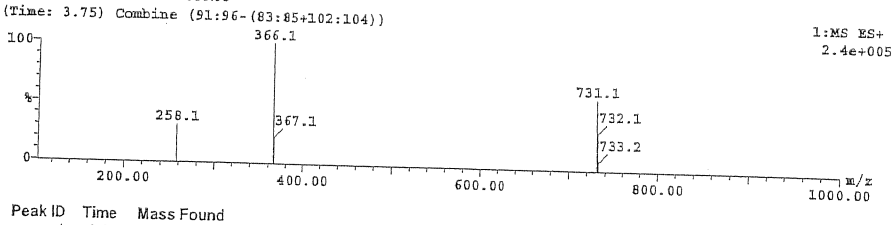


Sample Report (continued):

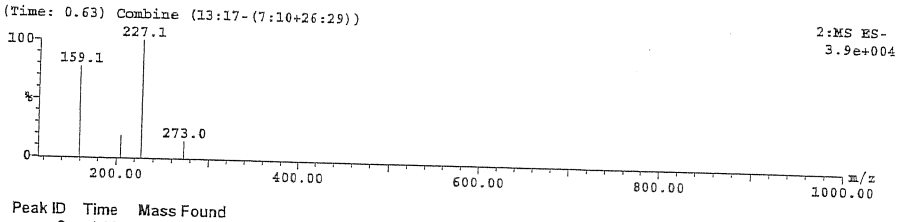


$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

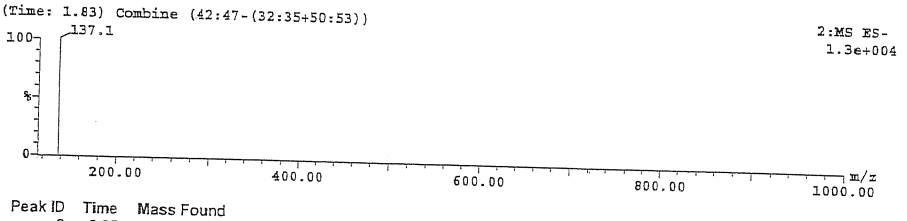
Peak ID Time Mass Found
6 3.77 366.00
(Time: 3.75) Combine (91:96-(83:85+102:104))



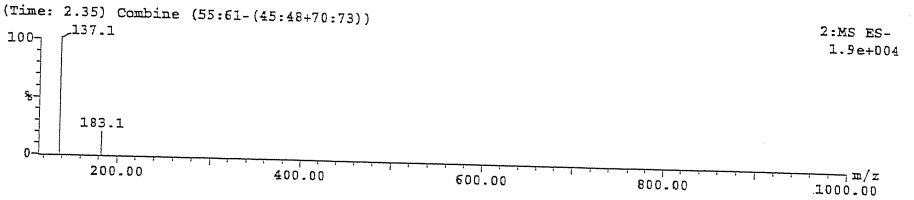
Peak ID Time Mass Found
1 0.63
(Time: 0.63) Combine (13:17-(7:10+26:29))



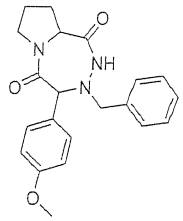
Peak ID Time Mass Found
2 1.83
(Time: 1.83) Combine (42:47-(32:35+50:53))



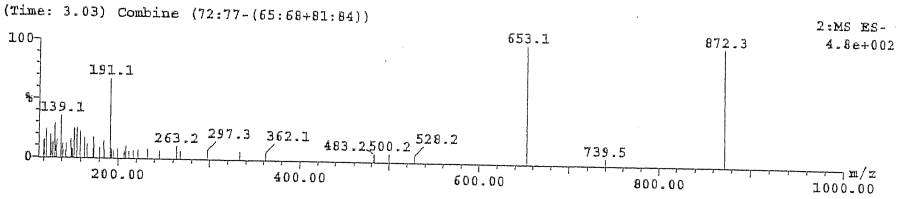
Peak ID Time Mass Found
3 2.35
(Time: 2.35) Combine (55:61-(45:48+70:73))



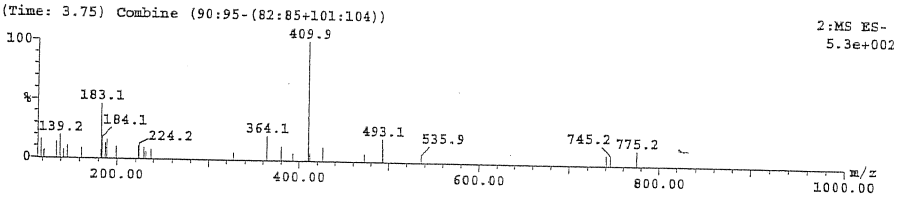
Sample Report (continued):



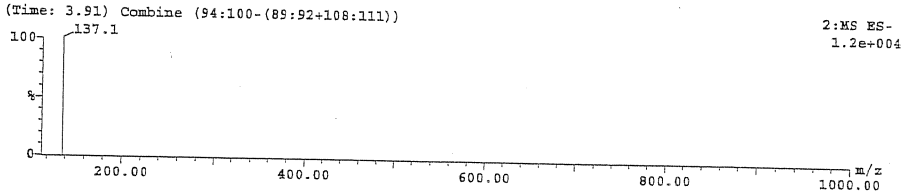
Peak ID Time Mass Found
5 3.05
(Time: 3.03) Combine (72:77-(65:68+81:84))

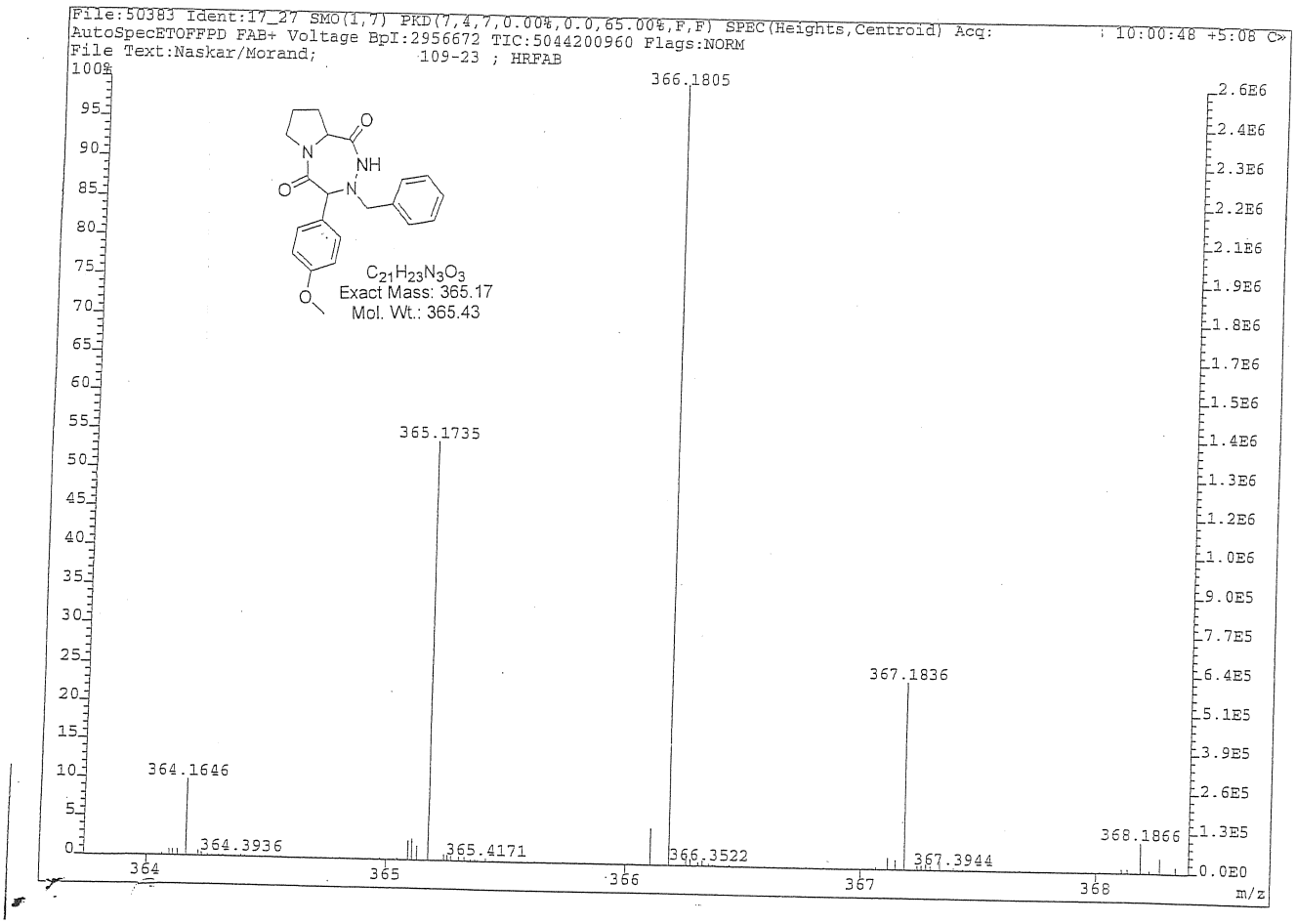


Peak ID Time Mass Found
6 3.77
(Time: 3.75) Combine (90:95-(82:85+101:104))



Peak ID Time Mass Found
7 3.91
(Time: 3.91) Combine (94:100-(89:92+108:111))

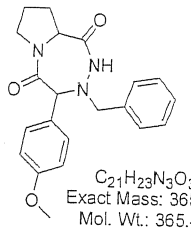



File:50383 Ident:17_27 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F) SPEC(Heights,Centroid) Acq:
AutoSpecETOFFPD FAB+ Voltage BpI:2956672 TIC:5044200960 Flags:NORM
File Text:Naskar/Morand; 109-23 ; HRFAB
10:00:48 +5:08
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
2.6E6
2.4E6
2.3E6
2.2E6
2.1E6
1.9E6
1.8E6
1.7E6
1.5E6
1.4E6
1.3E6
1.2E6
1.0E6
9.0E5
7.7E5
6.4E5
5.1E5
3.9E5
2.6E5
1.3E5
0.0E0
O
NH
N
O
N
O
$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43
366.1805
365.1735
367.1836
364.1646
364.3936
365.4171
366.3522
367.3944
368.1866
364
365
366
367
368
m/z

Elemental Composition

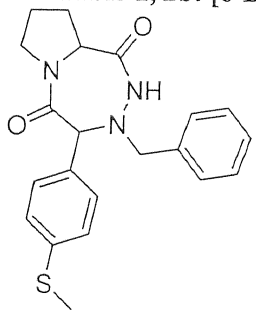
File:50383 Ident:17_27 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F)
~~AutoSpecETOFFID FAB+ Voltage EnT:2956672~~ TIC:5044200960 Flags:NORM
File Text:Naskar/Morand; 109-23 HRFAB
~~Heteroatom Max:~~ 20 Ion: Both Even and Odd
Limits:

363.748	5.0						-0.5	0	0	0	0	3
368.392	100.0		10.0		Theo:		100.0	25	1	90	3	3
Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O
367.183577	23.9			4.2	1.5	367.185122	11.5	20	1	24	3	3
366.180545	100.0	(M+H)+		3.3	1.2	366.181767	11.5	21		24	3	3
				-8.9	-3.2	366.177297	12.0	20	1	23	3	3
365.173483	53.8			1.3	0.5	365.173942	12.0	21		23	3	3
364.164626	9.7			4.1	1.5	364.166117	12.5	21		22	3	3
				-8.2	-3.0	364.161647	13.0	20	1	21	3	3

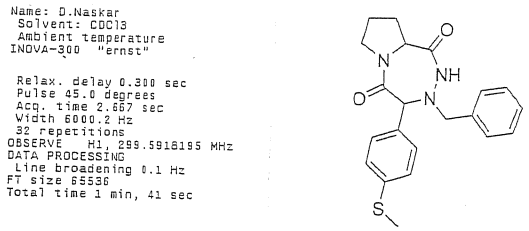


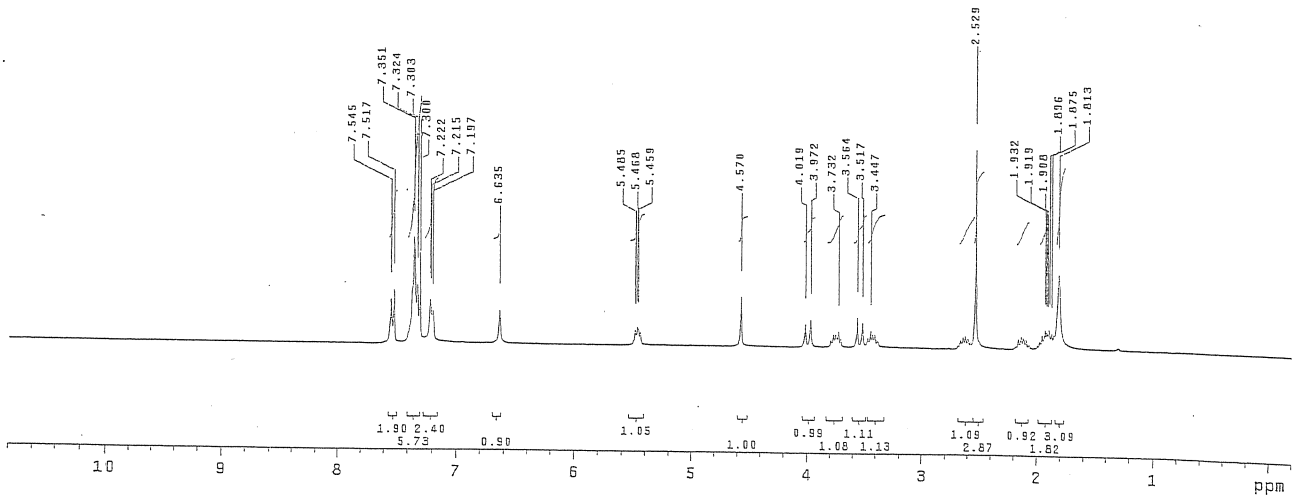
$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

IV. Table 1, 2b: [6-Benzyl-5-(4-methylsulfanyl-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White Solid; m.p (Met-Temp): 178°-179°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 1.87-1.93 (m, 3H), 2.52 (s, 3H), 2.55 (m, 1H), 3.44 (m, 1H), 3.54 (d, J= 14.1 Hz, 1H), 3.73 (m, 1H), 3.99 (d, J= 14.1 Hz, 1H), 4.57 (s, 1H), 5.45-5.48 (m, 1H), 6.63 (br. s, 1H), 7.19-7.22 (m, 2H), 7.3-7.35 (m, 5H); 7.53 (d, J= 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): 15.98, 22.88, 26.9, 48.53, 57.3, 57.46, 76.13, 127.47, 128.52, 129.04, 129.19, 134.76, 135.09, 140.05, 169.2, 172.5; LCMS (ELSD): 381.9 ($M+H^+$); HRMS: 382.1581 [Calculated for $C_{21}H_{24}N_3O_2S$ 382.1589 $(M+H)^+$].





Current Data Parameters

EXPNO 30
PROCNO 1

F2 - Acquisition Parameters

INSTRUM	spect
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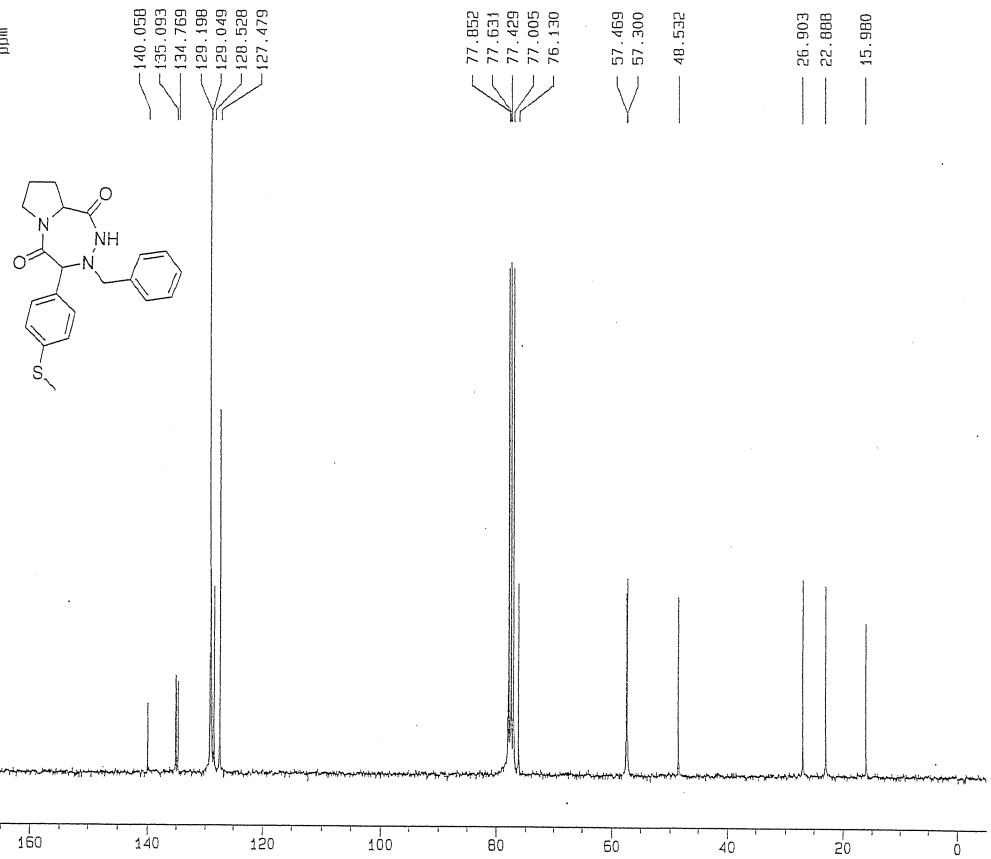
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	9387
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	4096
DW	26.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
============ CHANNEL f1 ============	
NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777800 MHz
============ CHANNEL f2 ============	
CPDPRG2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412006 MHz

F2 - Processing parameters

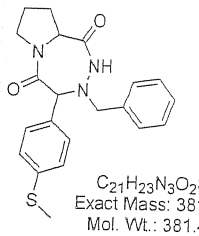
SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	25.00 cm
F1P	215.800 ppm
F1	16226.10 Hz
F2P	-5.000 ppm
F2	-377.35 Hz
PPMCM	8.80000 ppm/cm
HZCM	664.13800 Hz/cm

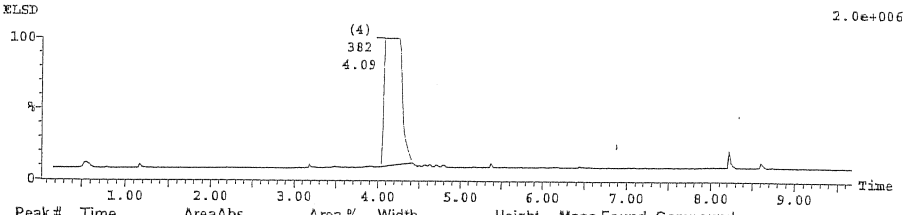


Sample Report:



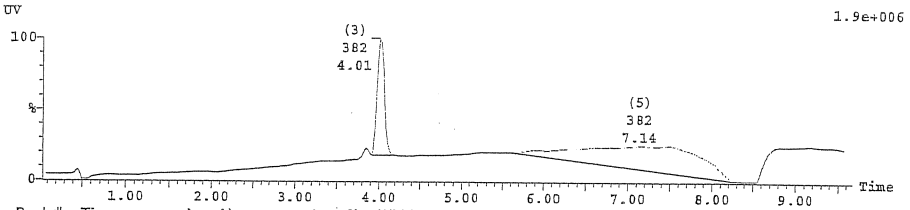
$C_{21}H_{23}N_3O_2S$
Exact Mass: 381.15
Mol. Wt.: 381.49

ELSD



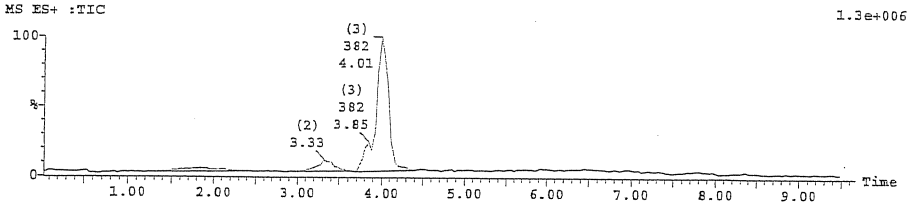
Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
4	4.09	4.526e+005	100.00	0.380	1.847e+006	382.00	Found

UV

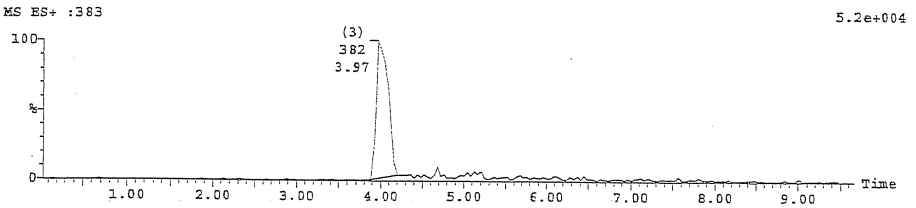


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
3	4.02	1.547e+005	17.41	0.280	1.513e+006	382.00	Found
5	7.14	5.346e+005	60.17	2.640	3.051e+005	382.00	Found
6	9.14	1.992e+005	22.42	1.040	2.117e+005		

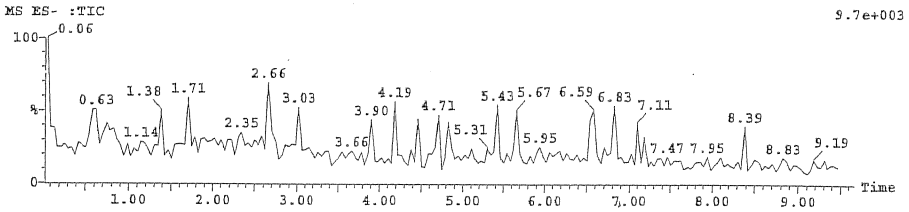
MS ES+ :TIC

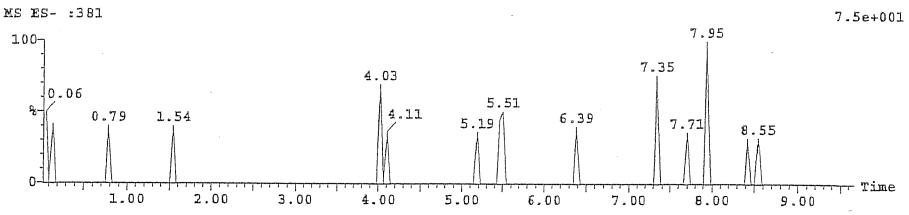


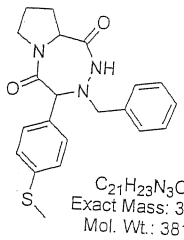
MS ES+ :383



Sample Report (continued):



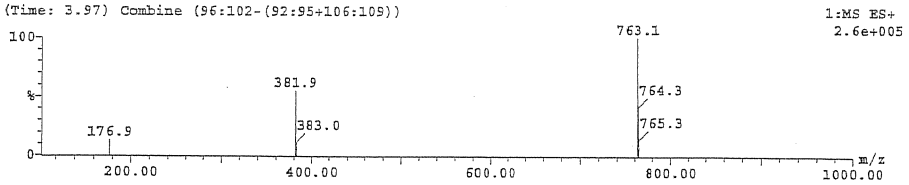




$C_{21}H_{23}N_3O_2S$
Exact Mass: 381.15
Mol. Wt.: 381.49

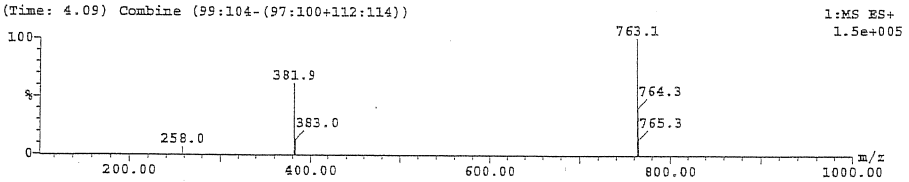
Peak ID	Time	Mass Found
3	4.01	382.00

(Time: 3.97) Combine (96:102-(92:95+106:109))

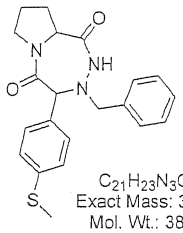


Peak ID	Time	Mass Found
4	4.09	382.00

(Time: 4.09) Combine (99:104-(97:100+112:114))

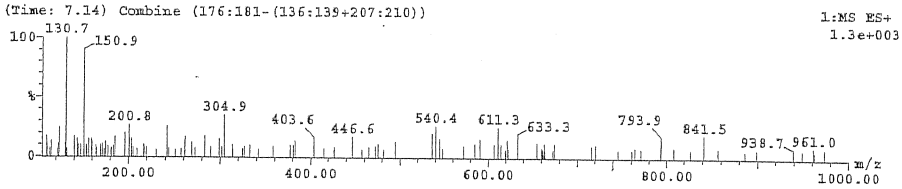


Sample Report (continued):

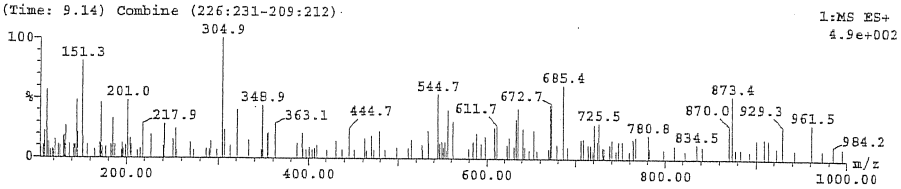


$C_{21}H_{23}N_3O_2S$
Exact Mass: 381.15
Mol. Wt.: 381.49

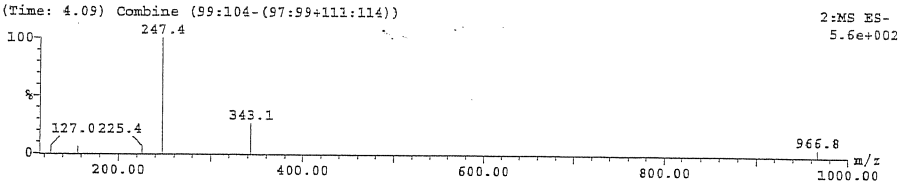
Peak ID Time Mass Found
5 7.14 382.00
(Time: 7.14) Combine (176:181-(136:139+207:210))



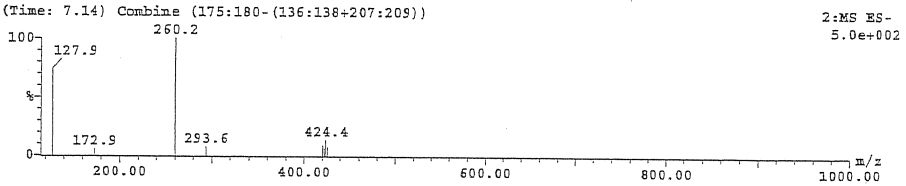
Peak ID Time Mass Found
6 9.14
(Time: 9.14) Combine (226:231-209:212)



Peak ID Time Mass Found
4 4.09
(Time: 4.09) Combine (99:104-(97:99+111:114))



Peak ID Time Mass Found
5 7.14
(Time: 7.14) Combine (175:180-(136:138+207:209))

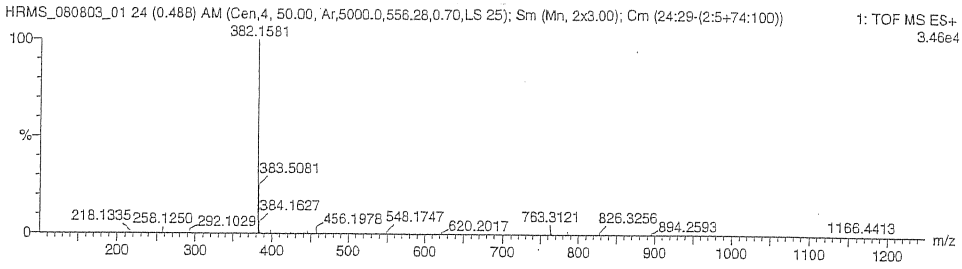


Elemental Composition Report

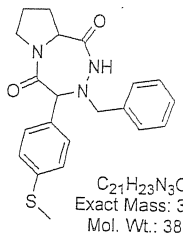
Single Mass Analysis

Tolerance = 30.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

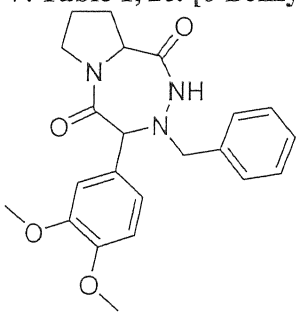


Minimum:				-1.5		
Maximum:		200.0	30.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
382.1581	382.1589	-0.8	-2.1	11.5	1	C21 H24 N3 O2 S

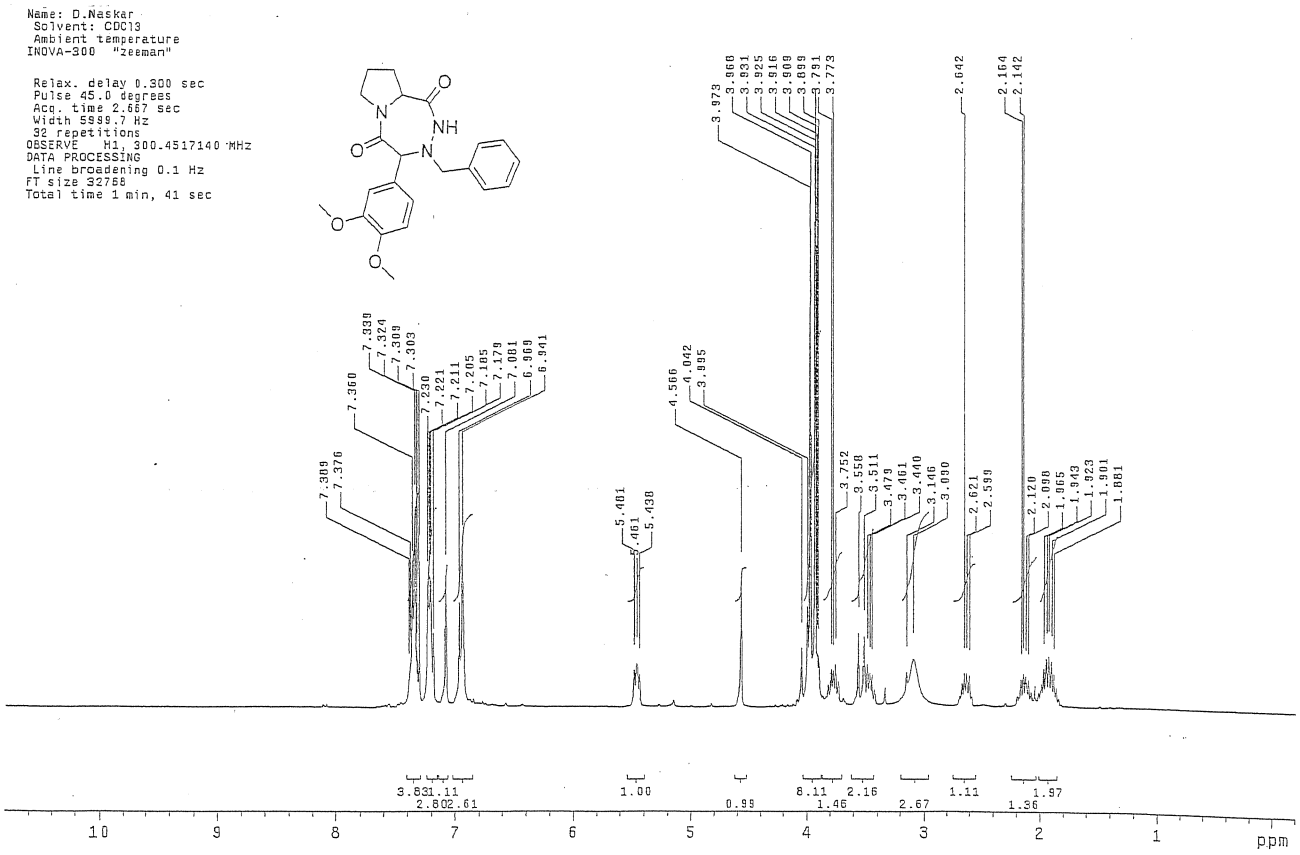


$C_{21}H_{23}N_3O_2S$
Exact Mass: 381.15
Mol. Wt.: 381.49

V. Table 1, 2c: [6-Benzyl-5-(3,4-dimethoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White Solid; m.p (Met-Temp): 85°- 86°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 1.88-1.96 (m, 2H) , 2.09-2.16 (m, 1H), 2.64 (m, 1H), 3.46 (m, 1H), 3.53 (d, J= 14.1 Hz, 1H), 3.77 (m, 1H), 3.93 (s, 3H), 3.96 (s, 3H), 4.02 (d, J= 14.1 Hz, 1H), 4.56 (s, 1H), 5.43-5.48 (m, 1H), 6.94-6.96 (m, 1H), 7.08 (s, 1H), 7.17-7.23 (m, 3H); 7.30-7.38 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz): 22.89, 27.78, 49.02, 56.34, 56.46, 57.32, 57.44, 112.18, 117.61, 120.91, 121.22, 125.13, 128.43, 128.94, 129.09, 135.50, 149.92, 169.73, 172.85; LCMS (ESI): 396.1 ($M+H^+$); HRMS: 396.1921 [Calculated for $C_{22}H_{26}N_3O_4$ 396.1923 $(M+H)^+$].



Current Data Parameters

EXPNO 230
PROCNO 1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	24998
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	6502
DW	26.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777600 MHz

========== CHANNEL f2 ==========

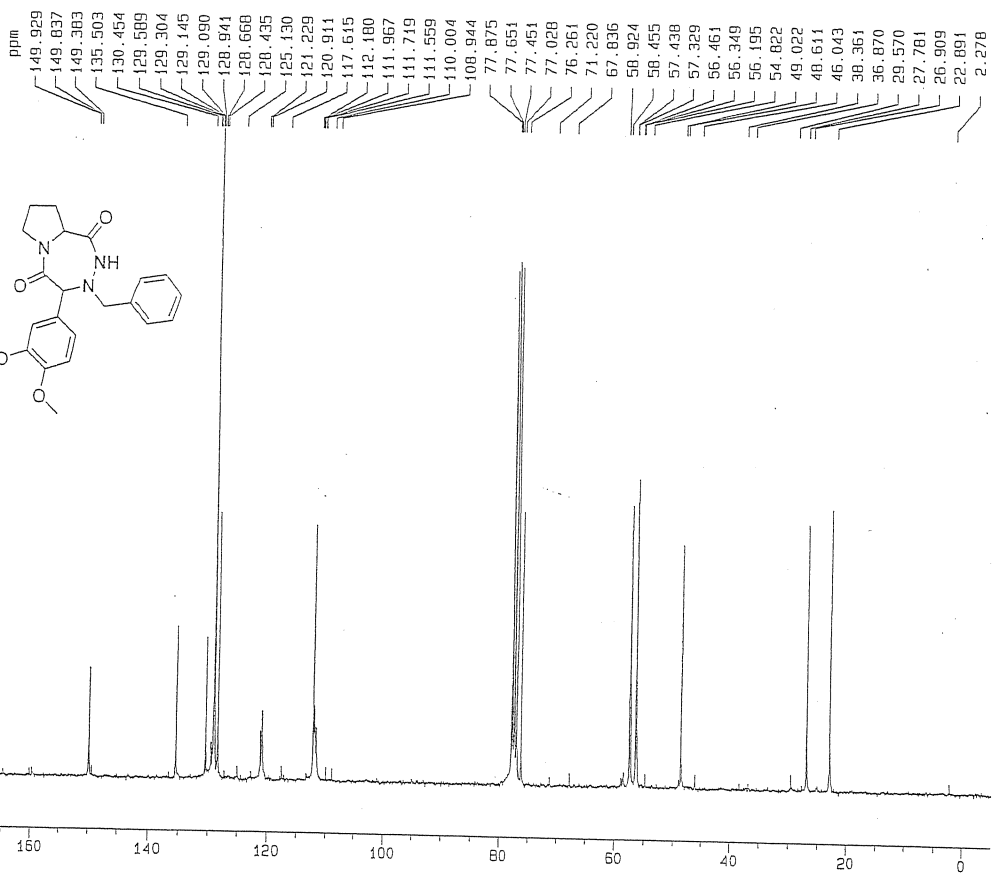
CPDPRG2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412005 MHz

F2 - Processing parameters

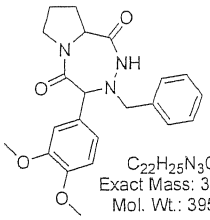
SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

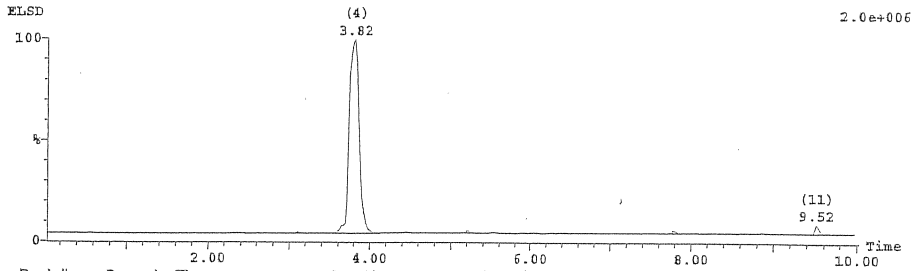
CX	25.00 cm
F1P	215.000 ppm
F1	16226.10 Hz
F2P	-5.000 ppm
F2	-377.35 Hz
PPMCM	8.60000 ppm/cm
HZCM	664.13800 Hz/cm



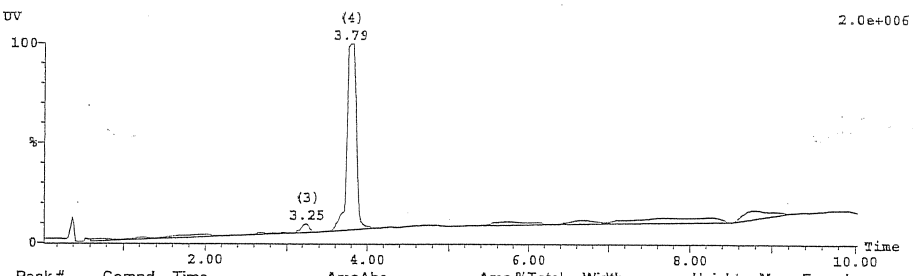
Sample Report (continued):



$C_{22}H_{25}N_3O_4$
Exact Mass: 395.18
Mol. Wt.: 395.45



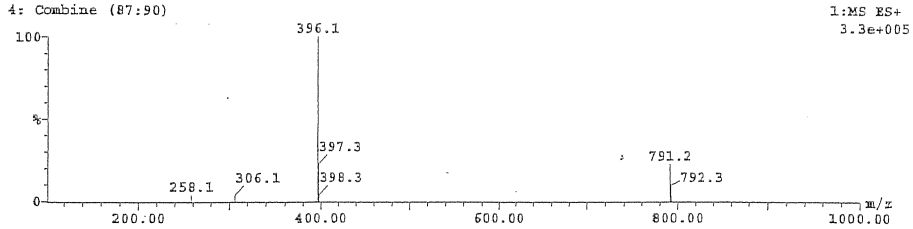
Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		0.39	6.171e+002	0.20	0.190	7.751e+003	
2		3.10	9.551e+002	0.32	0.170	1.450e+004	
4		3.82	2.877e+005	95.46	0.670	1.950e+006	
5		5.20	1.303e+003	0.43	0.170	2.076e+004	
8		7.78	1.946e+003	0.65	0.280	2.897e+004	
11		9.52	5.704e+003	1.89	0.220	9.622e+004	



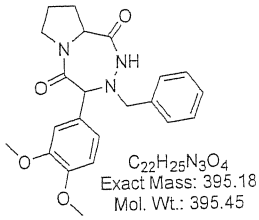
Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
3		3.25	4.913e+004	10.33	2.750	9.020e+004	
4		3.79	2.637e+005	55.41	0.760	1.896e+006	
6		5.74	1.984e+004	4.17	1.240	3.629e+004	
7		6.65	1.388e+004	2.92	0.590	3.928e+004	
9		8.26	6.365e+004	13.38	1.530	5.451e+004	
10		8.77	3.393e+004	7.13	0.730	9.229e+004	

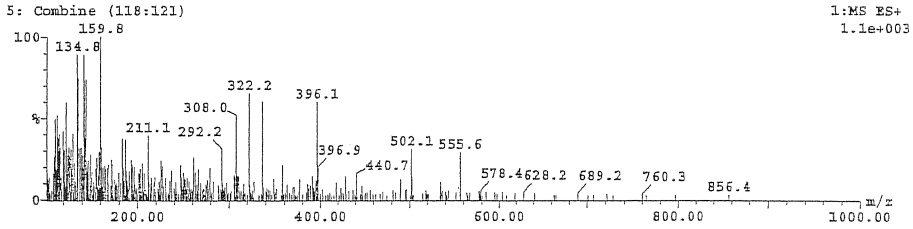
Sample Report (continued):

Mass Found Compound

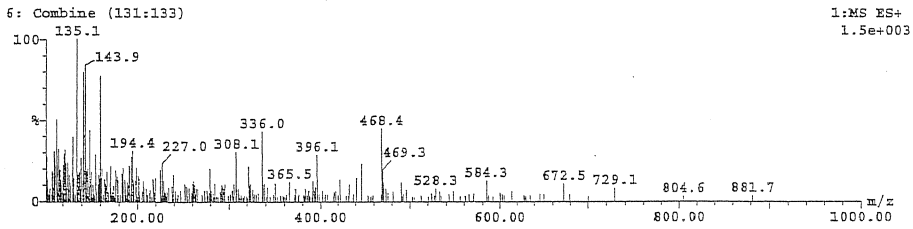


Mass Found Compound



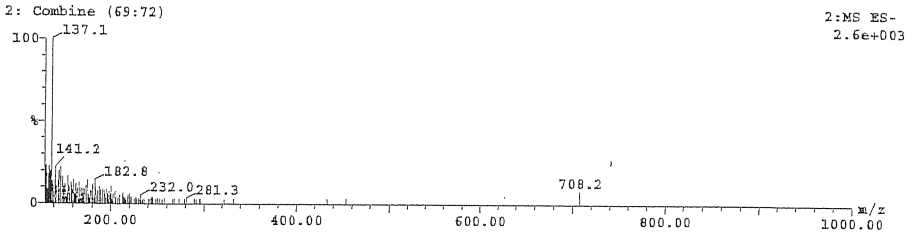


Mass Found Compound

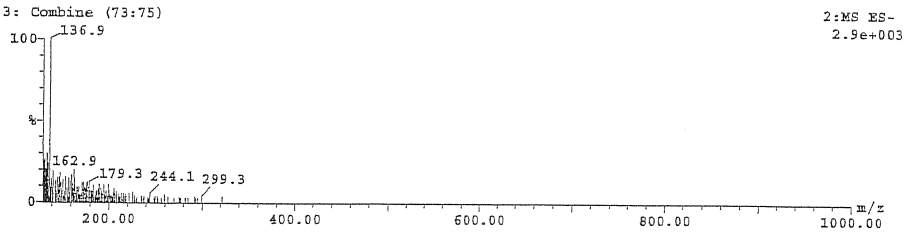


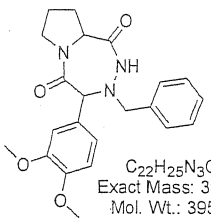
Sample Report (continued):

Mass Found Compound



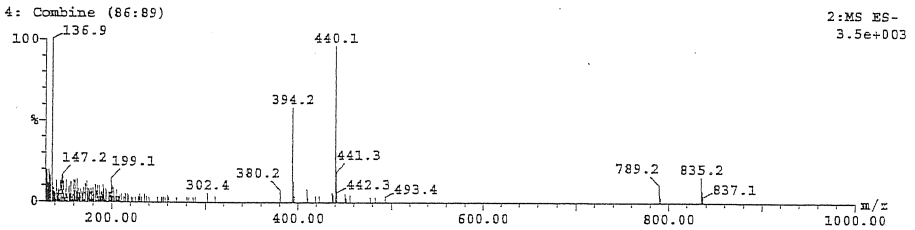
Mass Found Compound





$C_{22}H_{25}N_3O_4$
Exact Mass: 395.18
Mol. Wt.: 395.45

Mass Found Compound

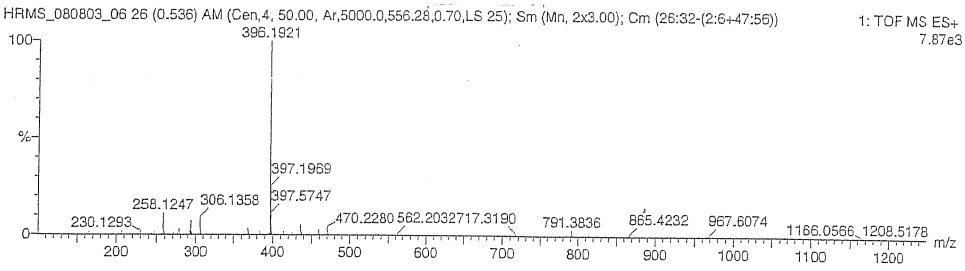


Single Mass Analysis
Tolerance = 30.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

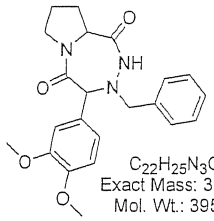
Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

HRMS_080803_06 26 (0.536) AM (Cen,4, 50.00, Ar,5000.0,556.28,0.70,LS 25); Sm (Mn, 2x3.00); Cm (26:32-(2:6+47:56))

1: TOF MS ES+
7.87e3

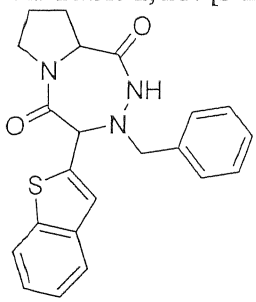


Minimum:				-1.5		
Maximum:		200.0	30.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
396.1921	396.1923	-0.3	-0.6	11.5	1	C22 H26 N3 O4

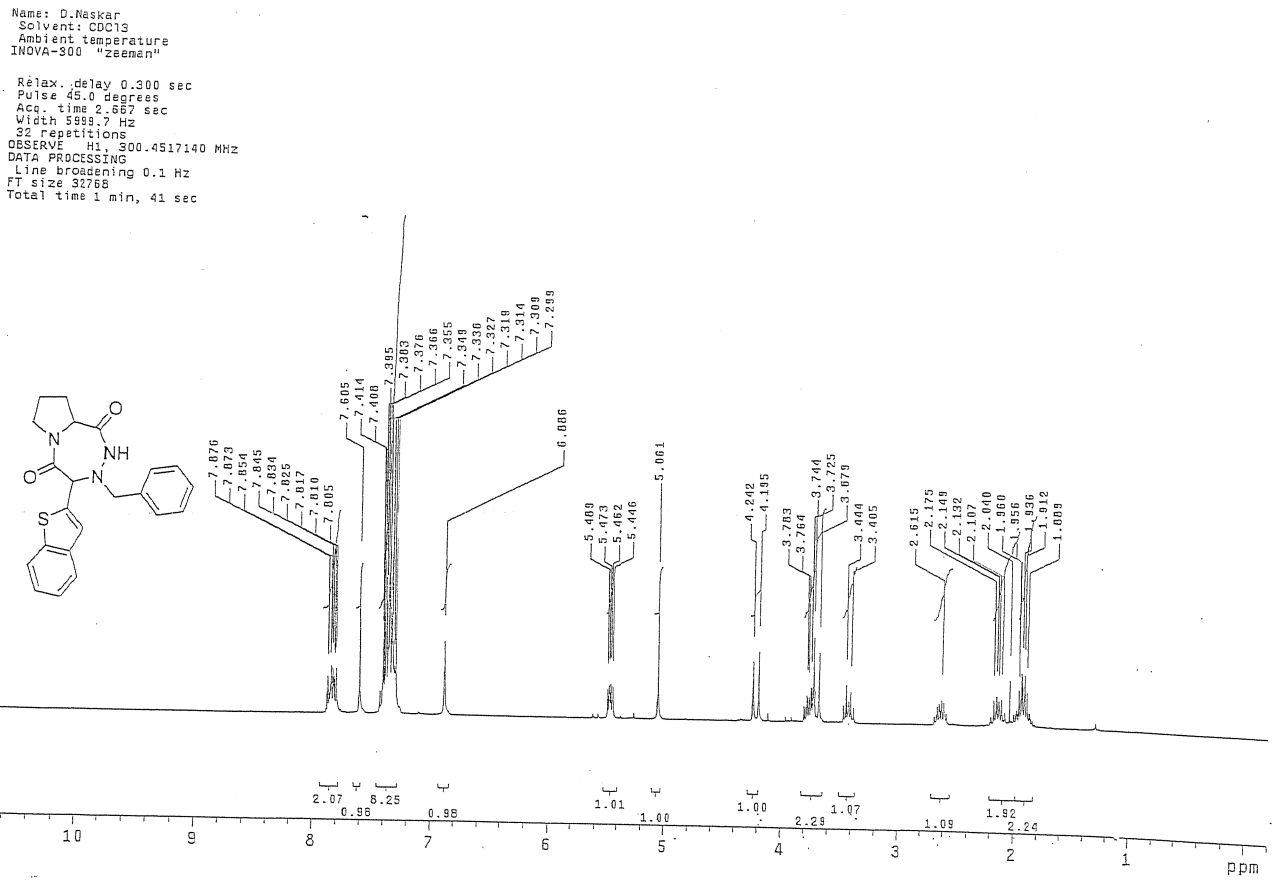


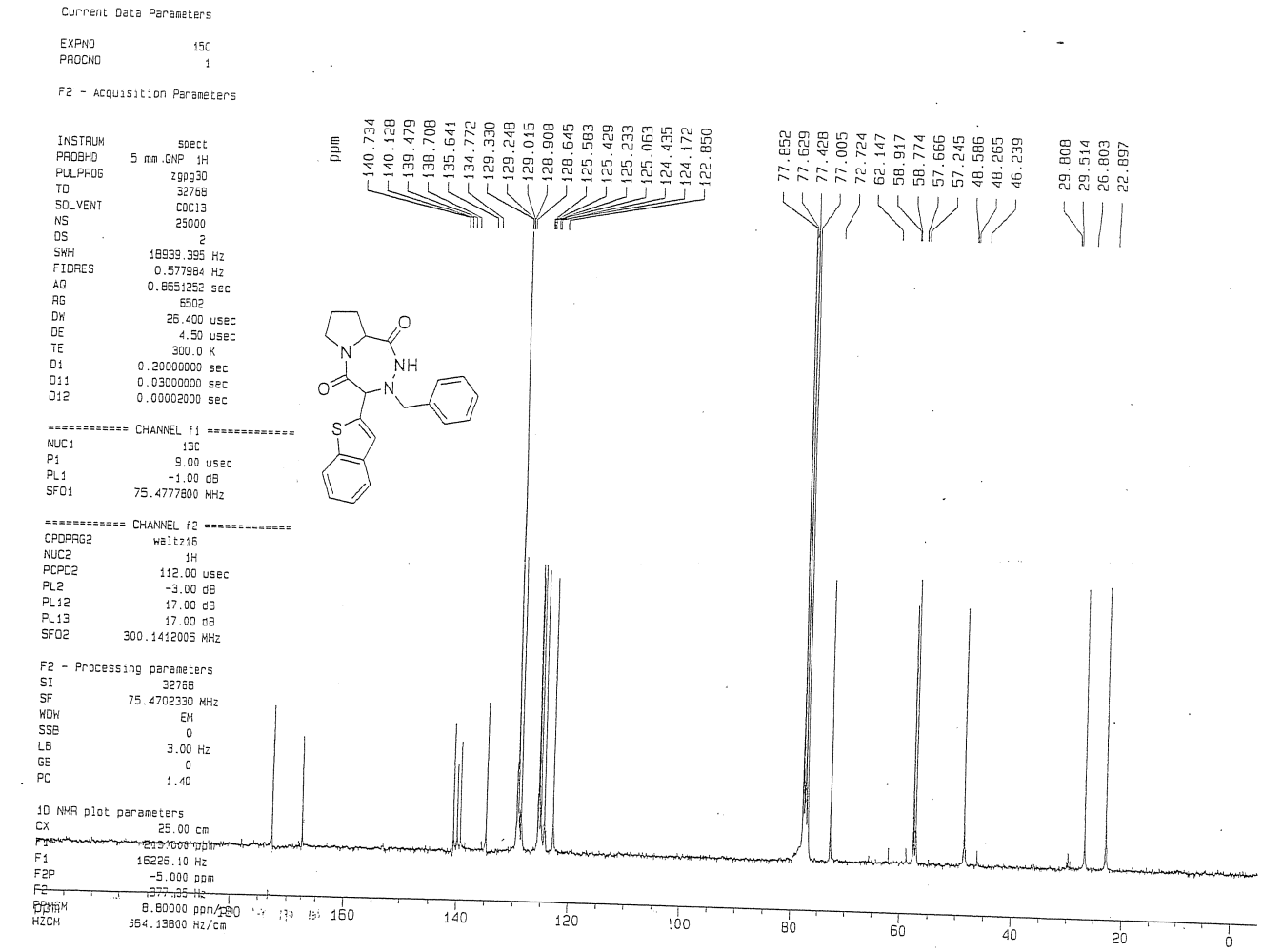
$C_{22}H_{25}N_3O_4$
Exact Mass: 395.18
Mol. Wt.: 395.45

VI. Table 1, 2d: [5-Benzo[*b*]thiophen-2-yl-6-benzyl--hexahydro-3a,6,7-triaza-azulene-4,8-dione]

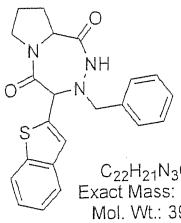


White Solid; m.p (Met-Temp): 223°-224°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 1.88-1.96 (m, 2H), 2.1-2.17 (m, 1H), 2.61 (m, 1H), 3.40-3.44 (m, 1H), 3.67-3.78 (m, 2H), 4.22 (d, J= 14.1 Hz, 1H), 5.06 (s, 1H), 5.44-5.48 (m, 1H), 6.88 (br. s, 1H), 7.29-7.41 (m, 7H), 7.60 (s, 1H), 7.80-7.87 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): 22.89, 26.80, 48.58, 57.24, 57.66, 72.72, 122.85, 125.06, 125.23, 125.42, 125.58, 128.90, 129.01, 129.33, 134.77, 139.47, 140.13, 140.73, 168.5, 172.8; LCMS (ELSD): 392.1 ($M+H^+$); HRMS: 392.148332 [Calculated for $C_{22}H_{22}N_3O_2S$ 392.148874 $(M+H)^+$].

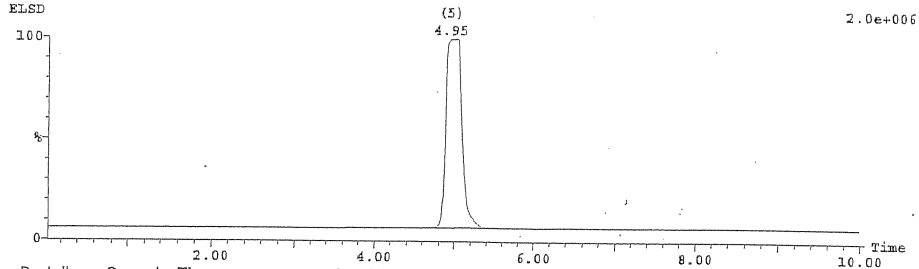



Current Data Parameters
EXPNO 150
PROCNO 1
F2 - Acquisition Parameters
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 25000
DS 2
SWH 18939.395 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 6502
DW 26.400 usec
DE 4.50 usec
TE 300.0 K
D1 0.20000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 9.00 usec
PL1 -1.00 dB
SFO1 75.4777800 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 112.00 usec
PL2 -3.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 300.1412006 MHz
F2 - Processing parameters
SI 32768
SF 75.4702330 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 213.608 ppm
F1 16226.10 Hz
F2P -5.000 ppm
F2 -377.35 Hz
PPMCM 8.80000 ppm/cm
HZCM 364.13800 Hz/cm
ppm
140.734
140.128
139.479
138.708
135.641
134.772
129.330
129.248
129.015
128.908
128.645
125.583
125.429
125.233
125.063
124.435
124.172
122.850
77.852
77.629
77.428
77.005
72.724
62.147
58.917
58.774
57.666
57.245
48.586
48.265
46.239
29.808
29.514
26.803
22.897
O
N
NH
O
N
S
160
140
120
100
80
60
40
20
0

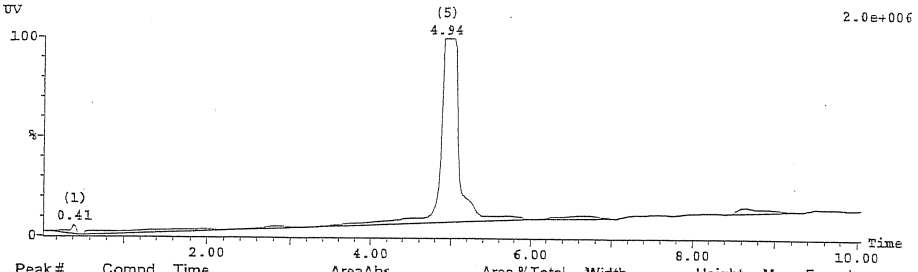
Sample Report (continued):



$C_{22}H_{21}N_3O_2S$
Exact Mass: 391.14
Mol. Wt.: 391.49

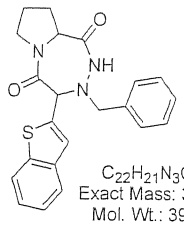


Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		0.39	7.424e+002	0.17	0.300	7.457e+003	
4		4.51	2.119e+002	0.05	0.240	2.131e+003	
5		4.95	4.418e+005	99.47	0.780	1.915e+006	
6		5.44	8.006e+002	0.18	0.260	8.165e+003	
7		5.75	2.615e+002	0.06	0.220	4.531e+003	
8		6.44	1.521e+002	0.03	0.230	1.602e+003	



Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		0.41	9.671e+003	1.54	0.370	9.960e+004	
2		0.58	1.237e+004	1.97	0.410	3.717e+004	
3		1.32	1.065e+004	1.70	0.460	2.602e+004	
5		4.94	5.502e+005	87.61	4.660	1.877e+006	
9		6.67	1.791e+004	2.85	0.930	3.485e+004	
10		8.65	2.377e+004	3.79	0.870	6.010e+004	

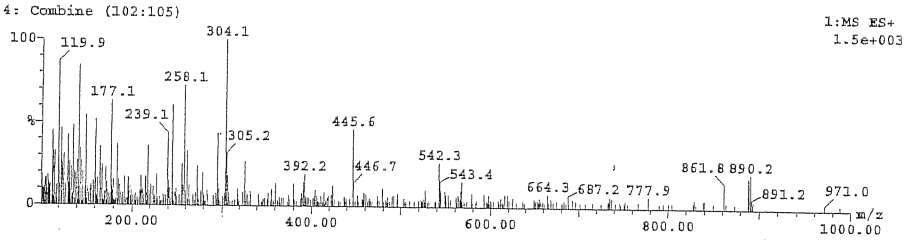
Sample Report (continued):



$C_{22}H_{21}N_3O_2S$
Exact Mass: 391.14
Mol. Wt.: 391.49

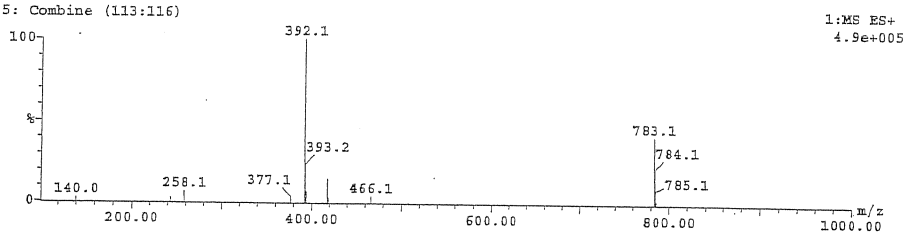
Mass Found Compound

4: Combine (102:105)



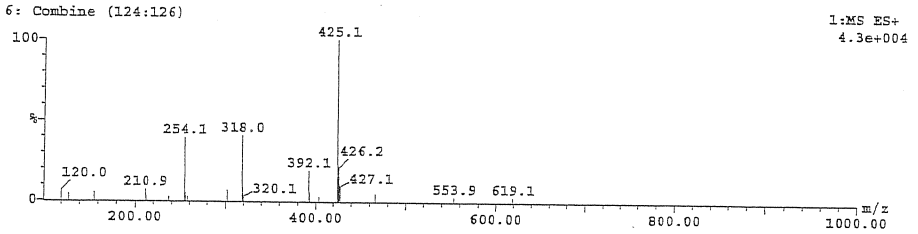
Mass Found Compound

5: Combine (113:116)

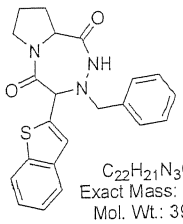


Mass Found Compound

6: Combine (124:126)

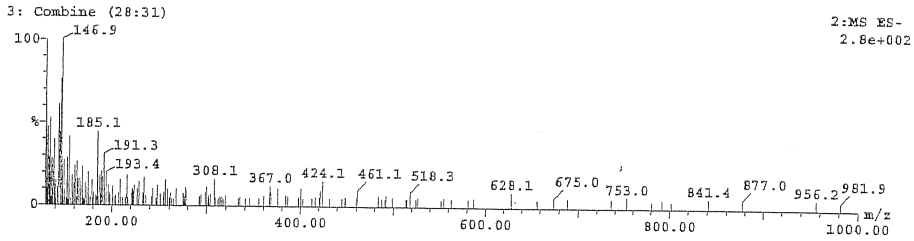


Sample Report (continued):

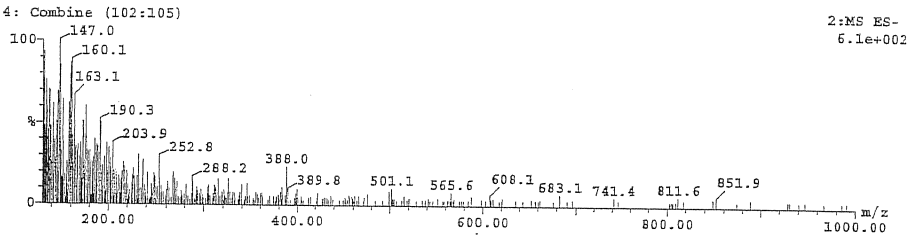


$C_{22}H_{21}N_3O_2S$
Exact Mass: 391.14
Mol. Wt.: 391.49

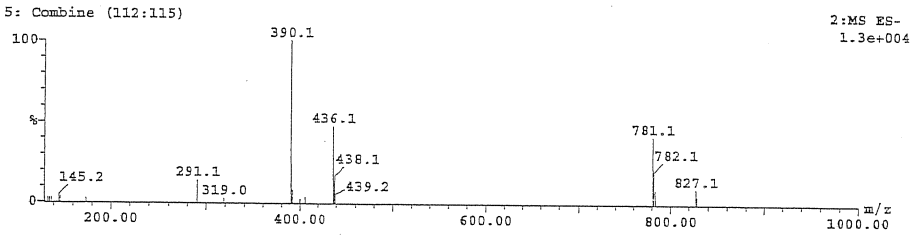
Mass Found Compound



Mass Found Compound



Mass Found Compound



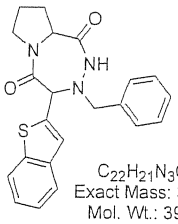
Elemental Composition

File:50793 Ident:17_27 SM0(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F)
AutoSpecETOFFPD FAB+ Voltage Bpi:2630976 TIC:5980897280 Flags:NORM
File Text:Naskar7Morand; 96-75 HRFAB
Heteroatom Max: 50 Ion: Both Even and Odd
Limits:

300.657	1.0					-0.5	0	0	0	3	2	1
396.278	100.0		10.0			50.0	90	1	90	3	2	1

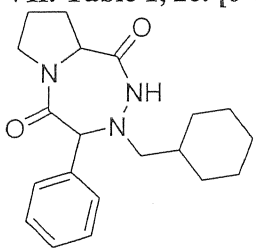
Theor.

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O	S
393.145234	27.4			3.5	1.4	393.146629	13.5	21	1	22	3	2	1
392.142332	100.0		$(M+H)^+$	2.4	0.9	392.143274	13.5	22		22	3	2	1
				-9.0	-3.5	392.138004	14.0	21	1	21	3	2	1
391.134406	40.0			2.7	1.0	391.135449	14.0	22		21	3	2	1
				-8.8	-3.4	391.130979	14.5	21	1	20	3	2	1
390.127664	9.6			-0.1	0.0	390.127624	14.5	22		20	3	2	1

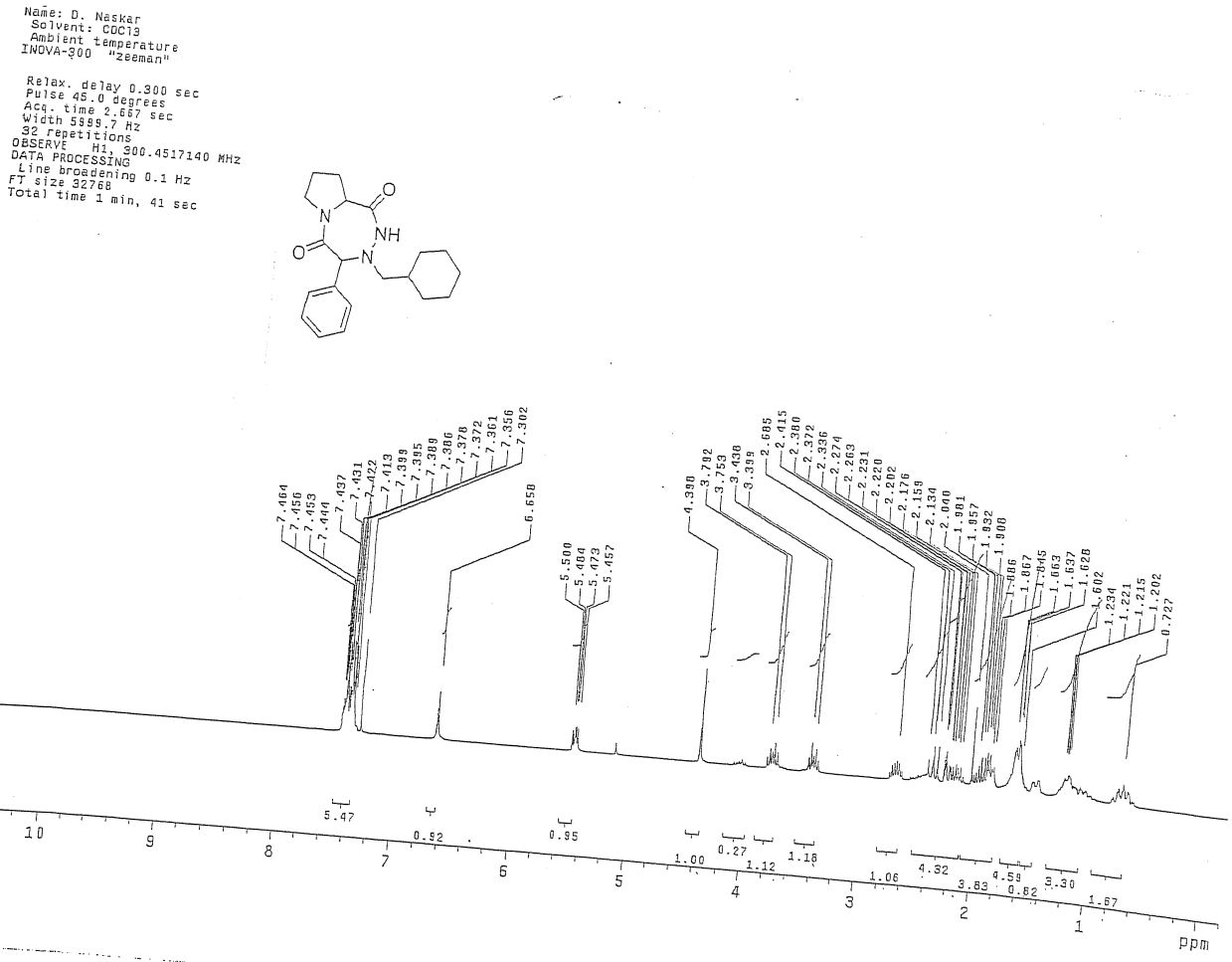


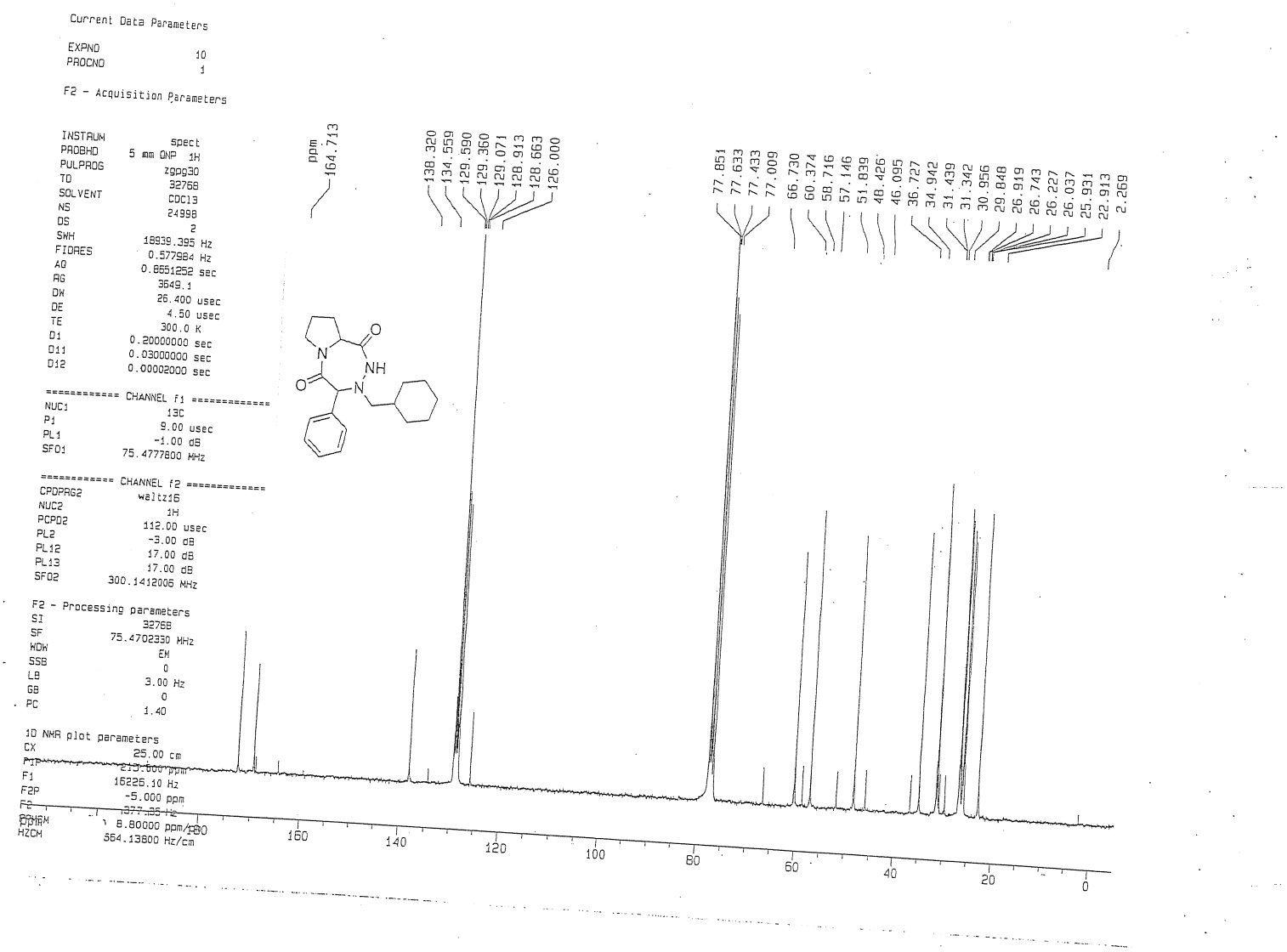
$C_{22}H_{21}N_3O_2S$
Exact Mass: 391.14
Mol. Wt.: 391.49

VII. Table 1, 2e: [6-Cyclohexylmethyl-5-phenyl-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White Solid; m.p (Met-Temp): 185°- 186°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 0.72 (m, 2H), 1.20-1.23 (m, 3H), 1.62-1.66 (m, 4H), 1.84-2.04 (m, 3H), 2.13-2.41 (m, 4H), 2.68 (m, 1H), 3.39-3.43 (m, 1H), 3.75-3.79 (m, 1H), 4.39 (s, 1H), 5.45-5.5 (m, 1H), 6.65 (br. s, 1H), 7.3-7.46 (m, 5H), ^{13}C NMR ($CDCl_3$, 75 MHz): 22.91, 25.93, 26.03, 26.74, 26.91, 31.34, 31.43, 34.94, 46.09, 48.42, 57.14, 60.37, 126, 128.66, 129.07, 129.36, 129.59, 138.32, 169.68, 172.73; LCMS (ESI): 342.2 ($M+H^+$); HRMS: 342.2172 [Calculated for $C_{20}H_{28}N_3O_2$ 342.2182 $(M+H)^+$].




Current Data Parameters
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 24998
DS 2
SWH 18939.395 Hz
FIDRES 0.577984 Hz
AQ 0.8651252 sec
RG 3649.1
DW 26.400 usec
DE 4.50 usec
TE 300.0 K
D1 0.20000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 -1.00 dB
SFO1 75.4777800 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 112.00 usec
PL2 -3.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 300.1412006 MHz
F2 - Processing parameters
SI 32768
SF 75.4702330 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1 15225.10 Hz
F2P -5.000 ppm
PPMCM 8.80000 ppm/cm
HZCM 664.13800 Hz/cm
ppm
164.713
138.320
134.559
129.590
129.360
129.071
128.913
128.663
126.000
77.851
77.633
77.433
77.009
66.730
60.374
58.716
57.146
51.839
48.426
46.095
36.727
34.942
31.439
31.342
30.956
29.848
26.919
26.743
26.227
26.037
25.931
22.913
2.269
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NH
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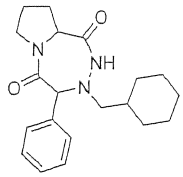
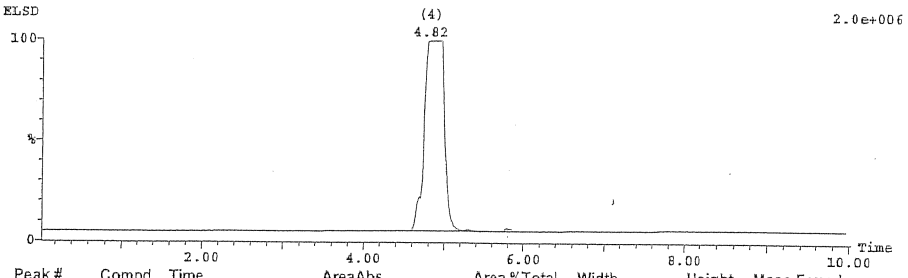
140
120
100
80
60
40
20
0

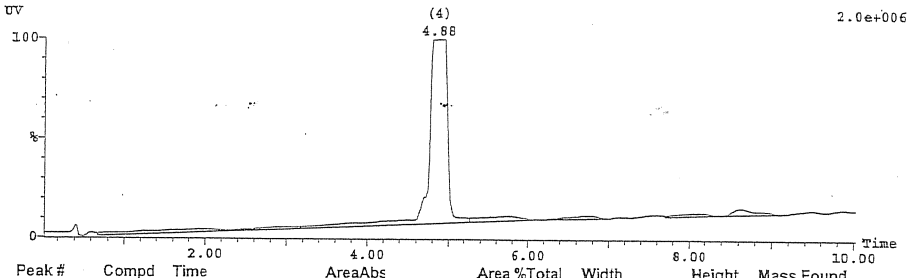
Sample Report (continued):



$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

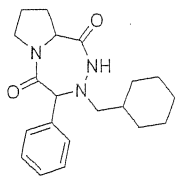


Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		2.34	1.123e+003	0.20	1.130	1.703e+003	
3		4.21	1.288e+003	0.23	0.620	3.963e+003	
4		4.82	5.510e+005	98.36	0.830	1.934e+006	
5		5.31	1.773e+003	0.32	0.240	1.531e+004	
6		5.79	2.586e+003	0.46	0.280	2.905e+004	
8		8.13	1.181e+003	0.21	0.960	2.045e+003	



Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
2		3.86	8.129e+004	12.01	3.280	3.852e+004	
4		4.88	4.907e+005	72.49	1.300	1.877e+006	
6		5.74	3.038e+004	4.49	0.860	5.077e+004	
7		6.74	1.390e+004	2.05	0.760	3.332e+004	
8		8.10	1.330e+004	1.96	0.680	2.907e+004	
9		8.62	2.057e+004	3.04	0.710	6.656e+004	

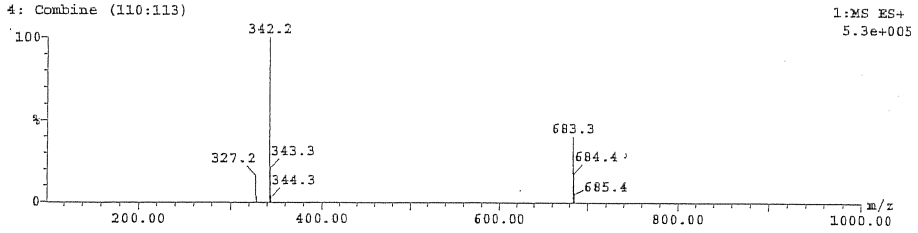
Sample Report (continued):



$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

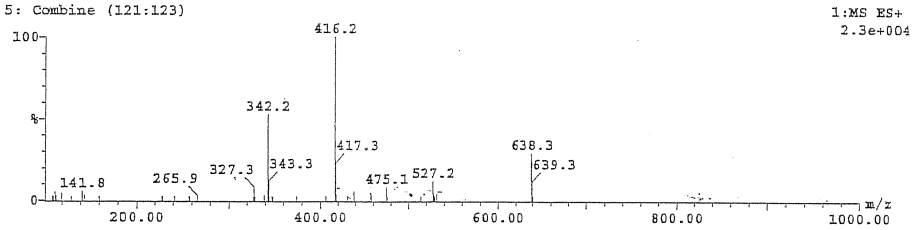
Mass Found Compound

4: Combine (110:113)



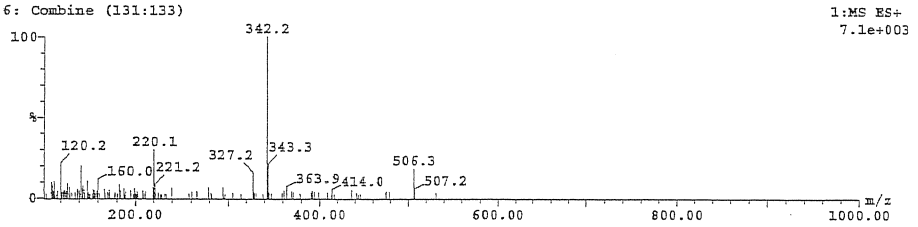
Mass Found Compound

5: Combine (121:123)



Mass Found Compound

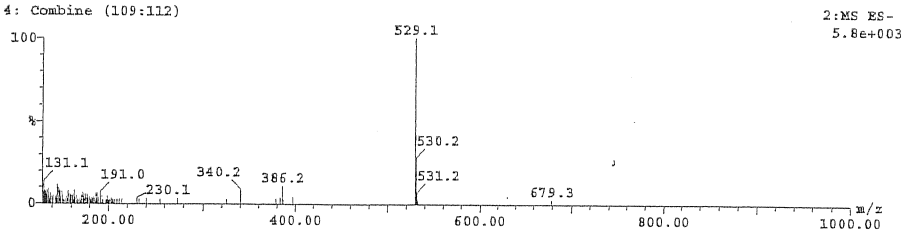
6: Combine (131:133)



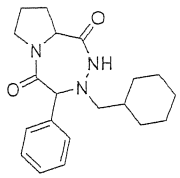
Sample Report (continued):

Mass Found Compound

4: Combine (109:112)

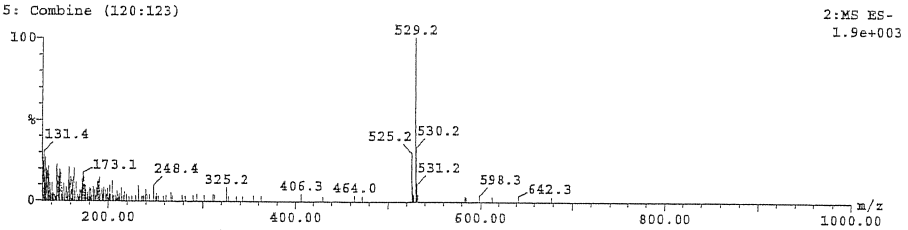


Mass Found Compound



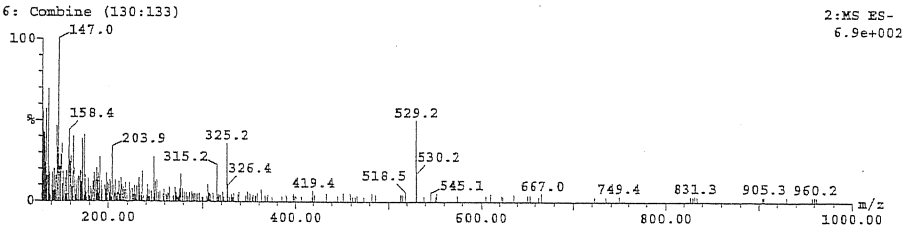
$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

5: Combine (120:123)



Mass Found Compound

6: Combine (130:133)



Elemental Composition Report

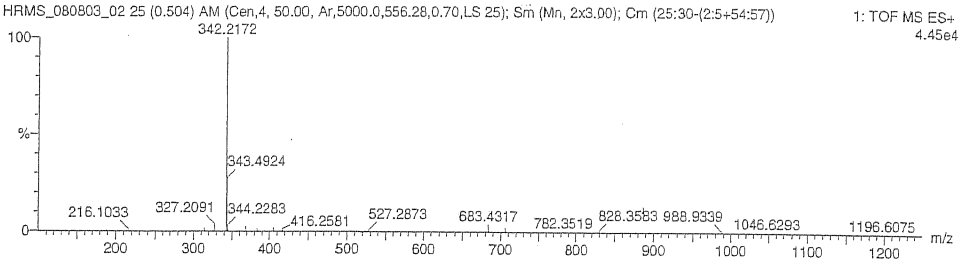
Single Mass Analysis

Tolerance = 30.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

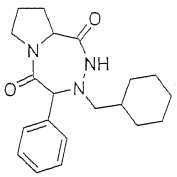
Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

HRMS_080803_02 25 (0.504) AM (Cen,4, 50.00, Ar,5000.0,556.28,0.70,LS 25); Sm (Mn, 2x3.00); Cm (25:30-(2:5+54:57))

1: TOF MS ES+
4.45e4

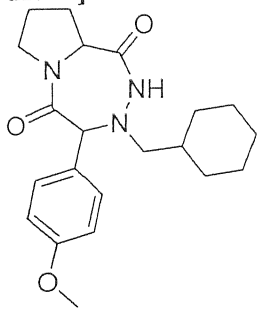


Minimum:				-1.5		
Maximum:		200.0	30.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
342.2172	342.2182	-1.0	-2.8	8.5	1	C20 H28 N3 O2

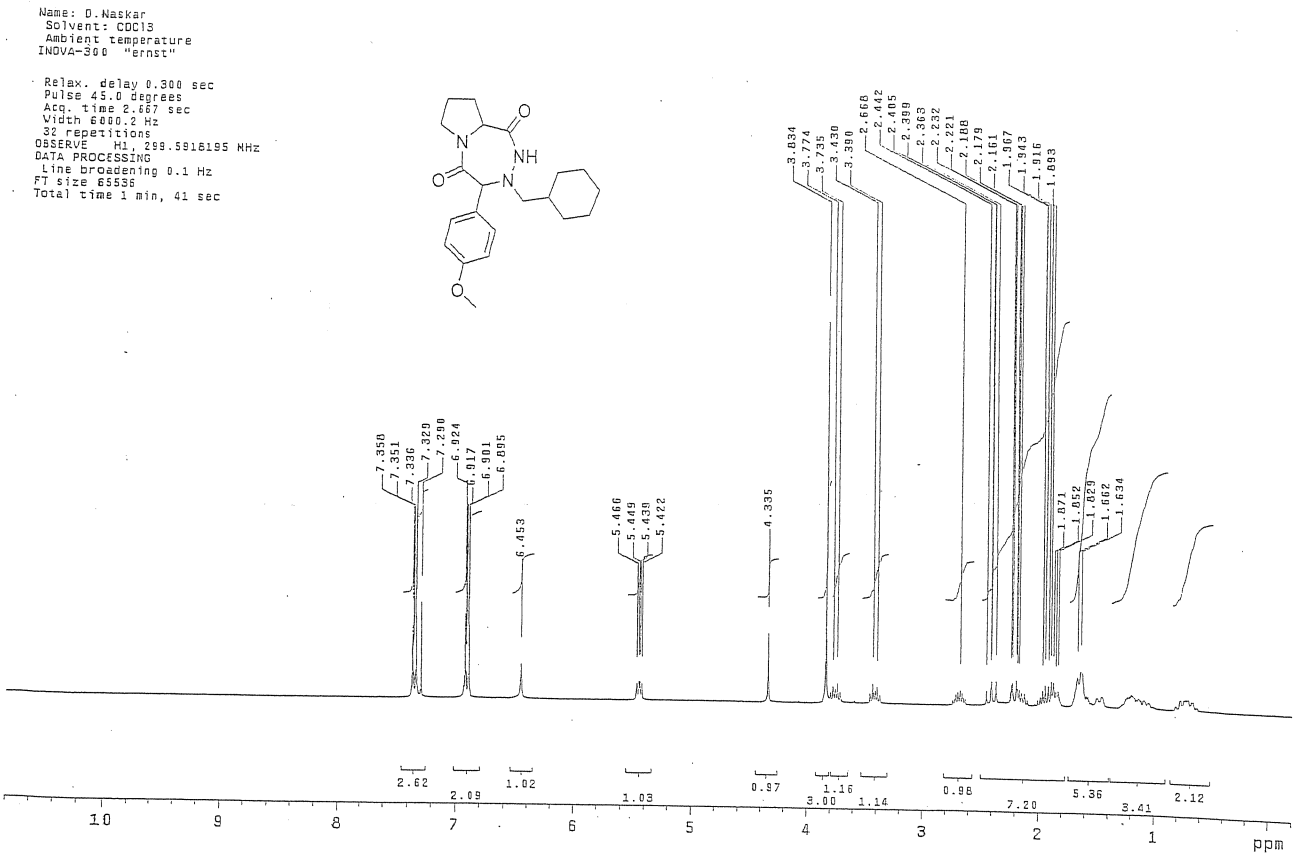


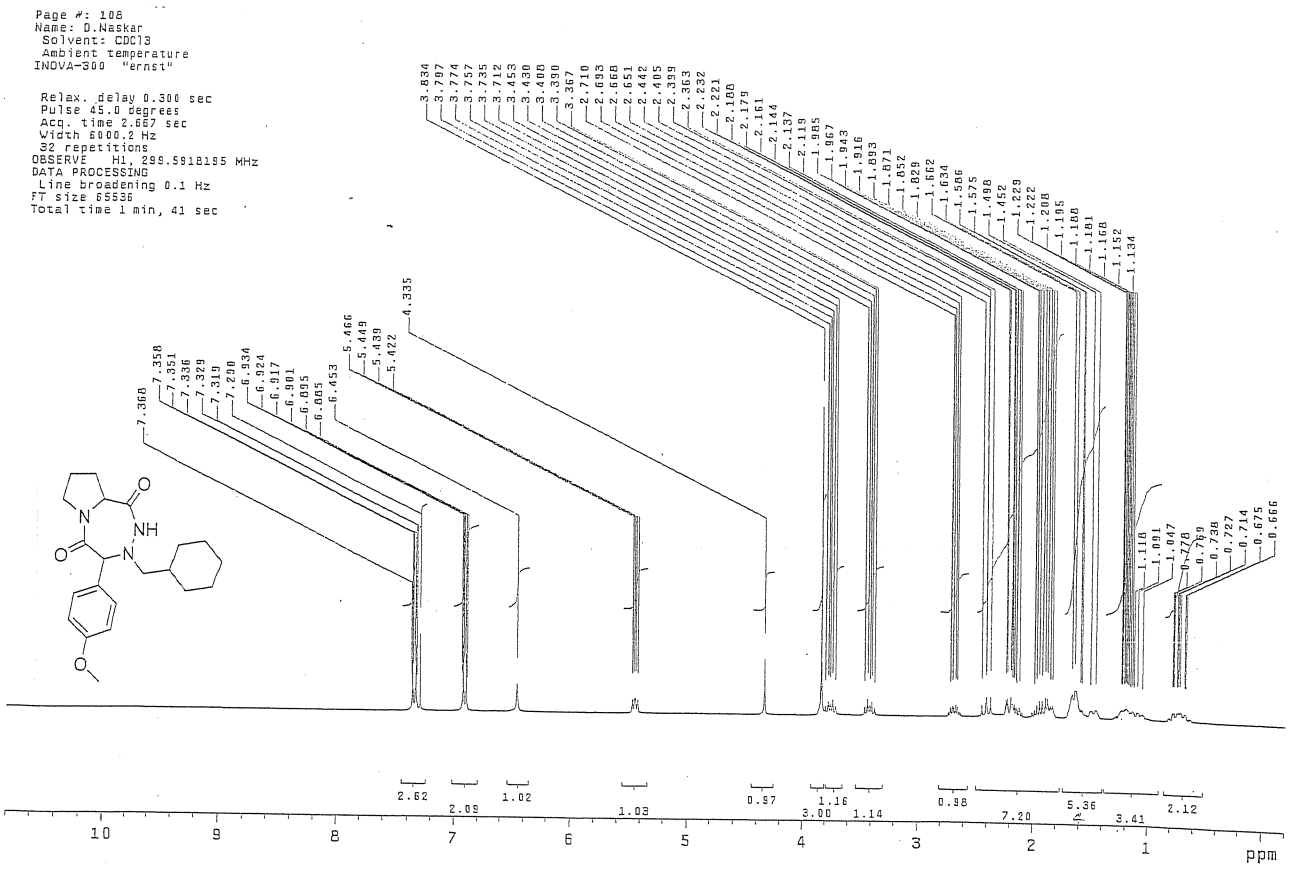
$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

VIII. Table 1, 2f: [6-Cyclohexylmethyl-5-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White Solid; m.p (Met-Temp): 90°-91°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 0.66-0.78 (m, 2H), 1.05-1.23 (m, 3H),1.45-1.50 (m, 2H), 1.57-1.66 (m, 2H), 1.83-1.99 (m, 3H), 2.12-2.23 (m, 2H), 2.36-2.44 (m, 2H), 2.65-2.71 (m, 1H), 3.37-3.45 (m, 1H), 3.71-3.79 (m, 1H), 3.83 (s, 3H), 4.34 (s, 1H), 5.42-5.47 (m, 1H), 6.45 (br. s, 1H), 6.91 (d, J= 8.7 Hz, 2H), 7.35 (d, J = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$,75MHz): 22.91, 25.71, 25.95, 26.75, 26.92, 31.37, 31.48, 34.95, 48.42, 55.67, 57.15, 55.98, 60.35, 114.77, 129.74, 130.40, 133.21, 160.15, 169.96, 172.73: LCMS (ELSD): 372.1 ($M+H^+$); HRMS: 372.228179 [Calculated for $C_{21}H_{30}N_3O_3$ 372.228717 $(M+H)^+$].




Page #: 108
Name: D.Naskar
Solvent: CDCl3
Ambient temperature
INOVA-300 "ernst"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 2.667 sec
Width 6000.2 Hz
32 repetitions
OBSERVE H1, 299.5918195 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 41 sec
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3.797
3.774
3.757
3.735
3.712
3.453
3.430
3.400
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3.367
2.710
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0.675
0.666
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N
NH
O
N
O
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2.09
1.02
1.03
0.97
1.16
3.00
1.14
0.98
7.20
5.36
3.41
2.12
10
9
8
7
6
5
4
3
2
1
ppm

Current Data Parameters

EXPNO 70
PROCNO 1

F2 - Acquisition Parameters

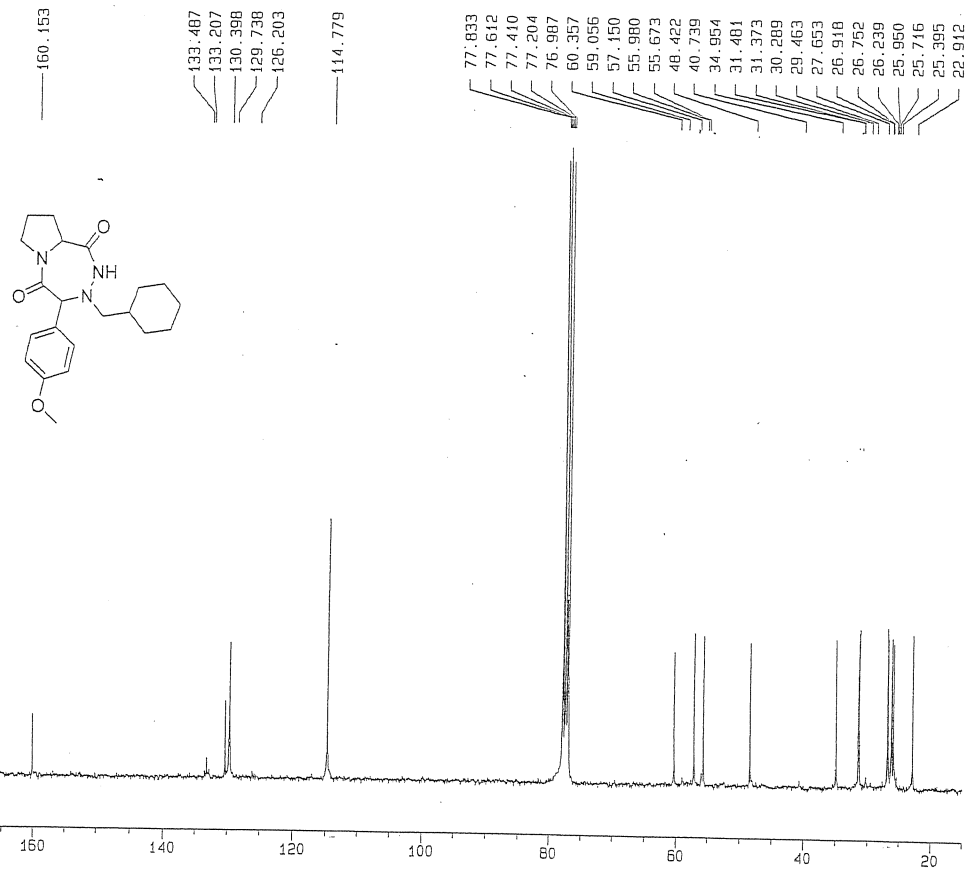
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	25000
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	4096
DW	25.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
========== CHANNEL f1 ==========	
NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777800 MHz
========== CHANNEL f2 ==========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412005 MHz

F2 - Processing parameters

SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

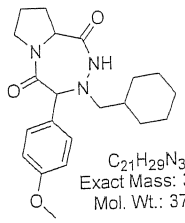
1D NMR plot parameters

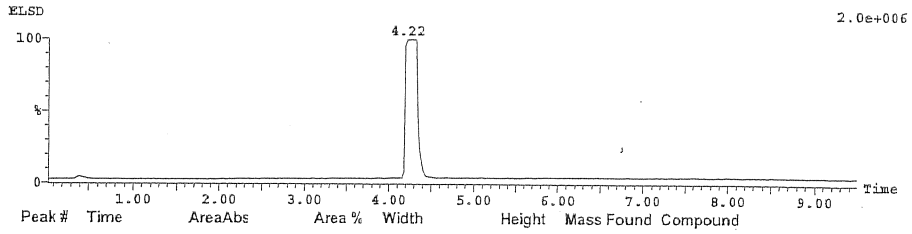
CX	25.00 cm
F1P	215.080 ppm
F1	16226.10 Hz
F2P	15.000 ppm
F2	1132.05 Hz
PPMCM	18.00000 ppm/cm
HZCM	03.76184 Hz/cm



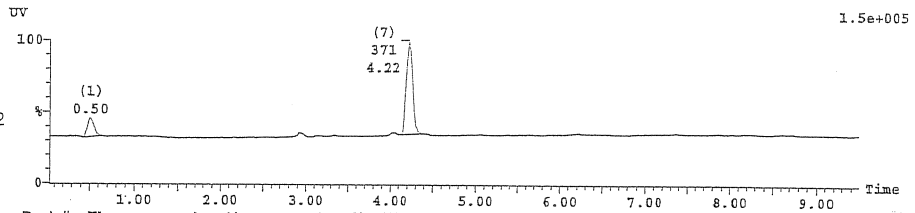
Comment / MW:COS/371

Sample Report:

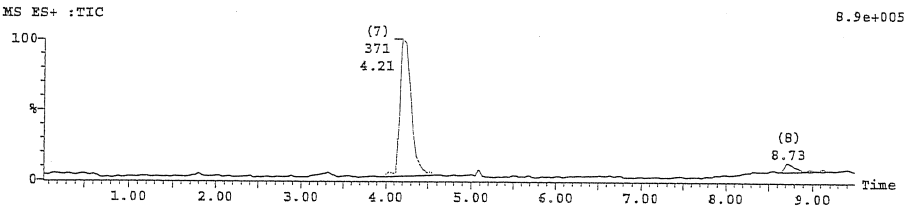


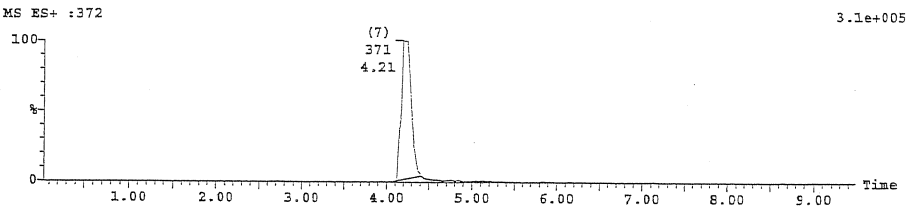


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound

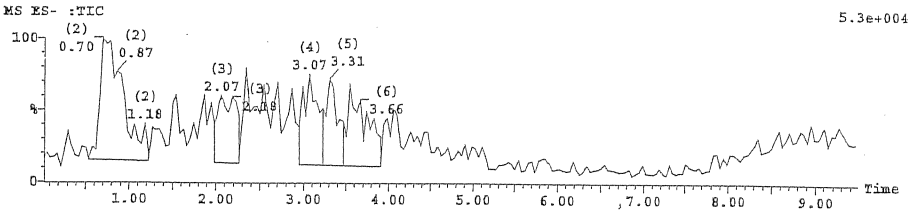


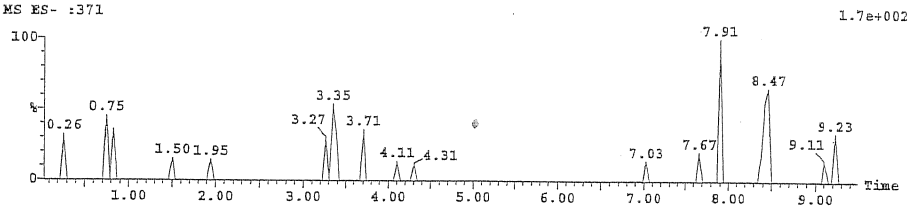
Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
1	0.51	1.695e+003	17.07	0.220	1.974e+004		
7	4.23	8.236e+003	82.93	0.300	9.616e+004	371.00	Found

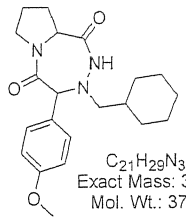




Sample Report (continued):



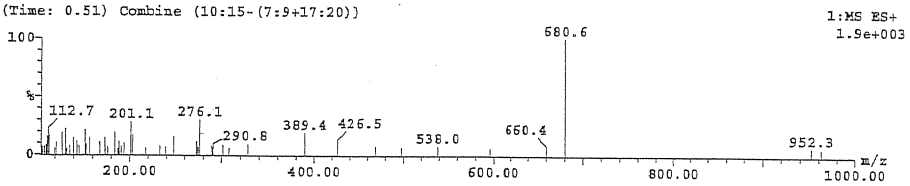




$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

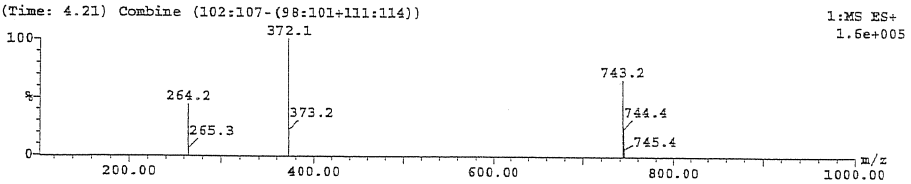
Peak ID Time Mass Found
1 0.51

(Time: 0.51) Combine (10:15-(7:9+17:20))

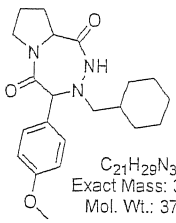


Peak ID Time Mass Found
7 4.21 371.00

(Time: 4.21) Combine (102:107-(98:101+111:114))

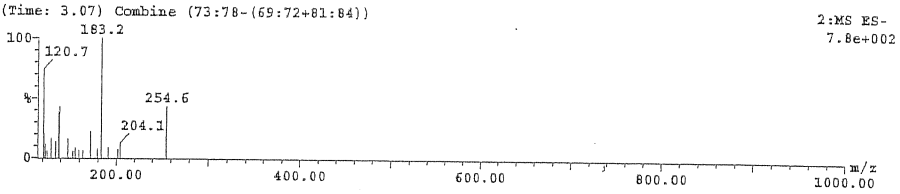


Sample Report (continued):

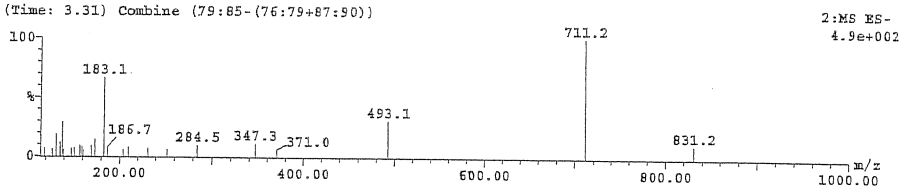


$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

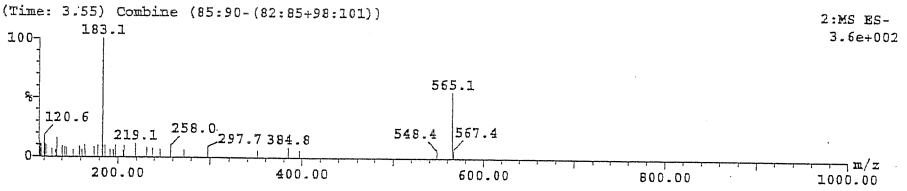
Peak ID Time Mass Found
4 3.07
(Time: 3.07) Combine (73:78-(69:72+81:84))



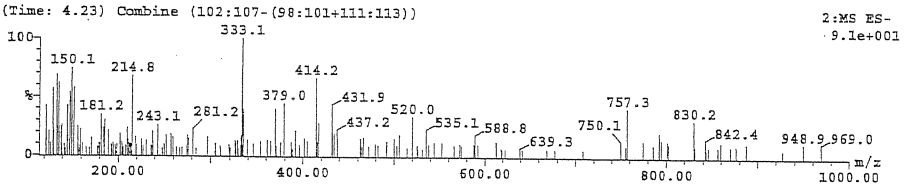
Peak ID Time Mass Found
5 3.31
(Time: 3.31) Combine (79:85-(76:79+87:90))

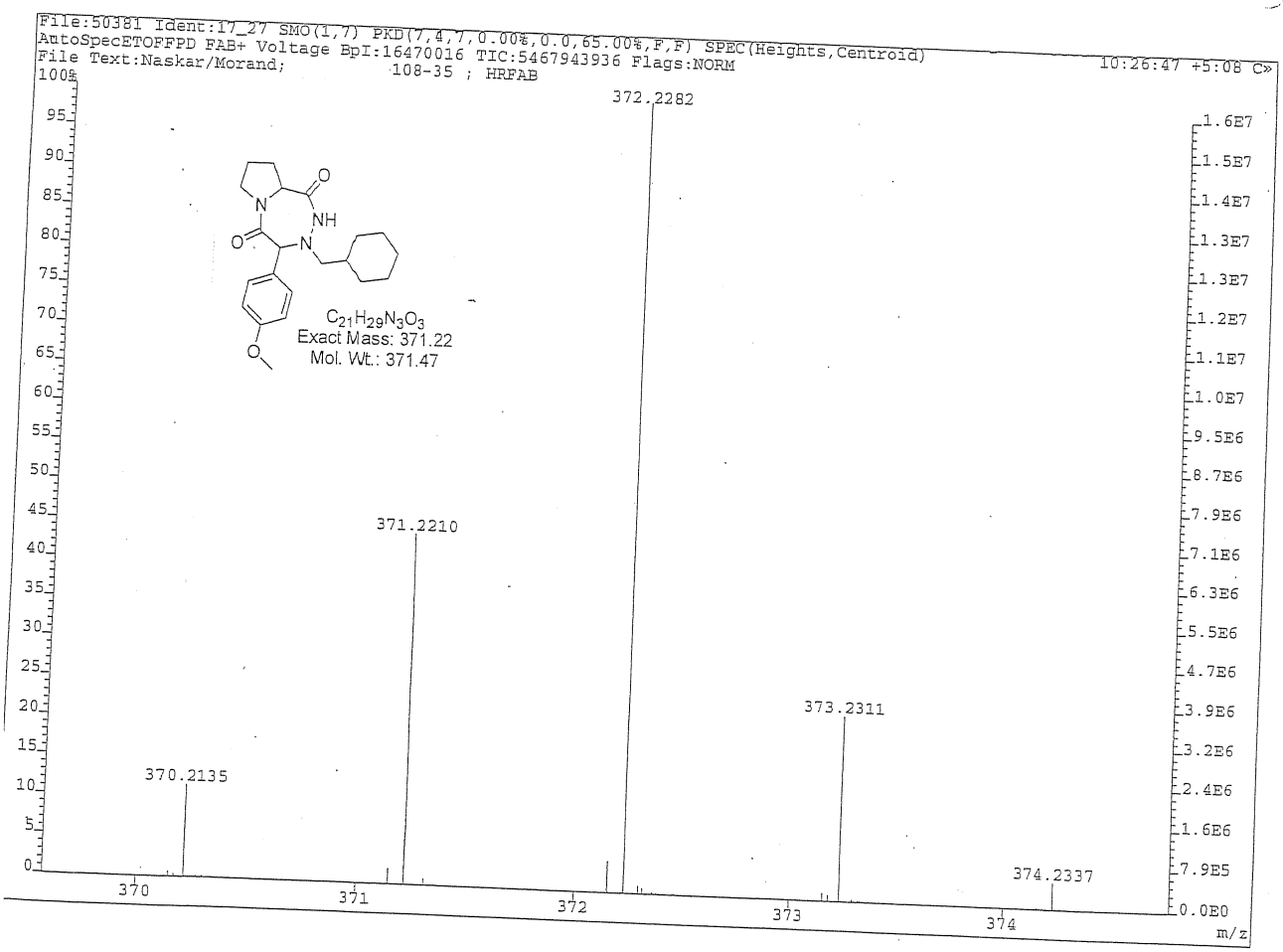


Peak ID Time Mass Found
6 3.55
(Time: 3.55) Combine (85:90-(82:85+98:101))



Peak ID Time Mass Found
7 4.21
(Time: 4.23) Combine (102:107-(98:101+111:113))



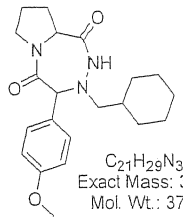

File:50381 Ident:17_27 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F) SPEC(Heights,Centroid)
AutoSpecETOFFPD FAB+ Voltage BpI:16470016 TIC:5467943936 Flags:NORM
File Text:Naskar/Morand; 108-35 ; HRFAB
10:26:47 +5:08 C»
100%
372.2282
371.2210
370.2135
373.2311
374.2337
C21H29N3O3
Exact Mass: 371.22
Mol. Wt.: 371.47
m/z

Elemental Composition

File:50381 Ident:17_27 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F)
AutoSpecETOFFPD FAB+ Voltage BpI:16470016 TIC:5467943936 Flags:NORM
File Text:Naskar/Morand; 108-35 ; HRFAB
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

369.578	5.0					-0.5	0	0	0	0	3
374.773	100.0		10.0		Theor.	100.0	25	1	90	3	3

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O
373.231130	23.2			2.5	0.9	373.232072	8.5	20	1	30	3	3
372.228179	100.0	(M+H)+		1.4	0.5	372.228717	8.5	21		30	3	3
371.221042	44.2			-0.4	-0.2	371.220892	9.0	21		29	3	3
370.213479	11.4			-1.1	-0.4	370.213067	9.5	21		28	3	3

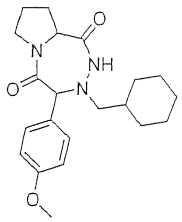


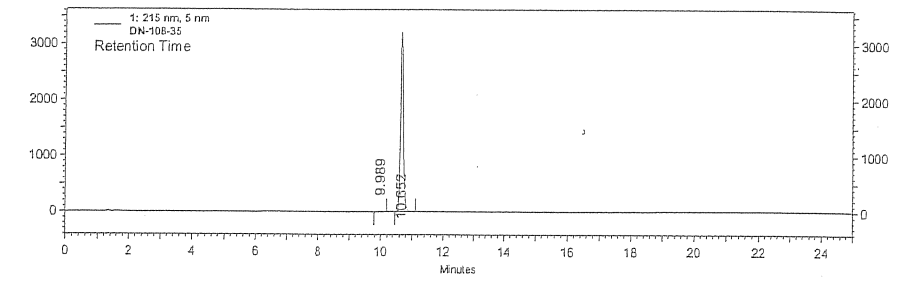
$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

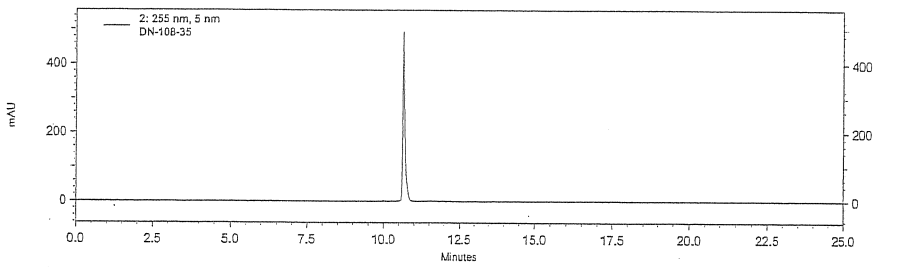
Area Percent Report

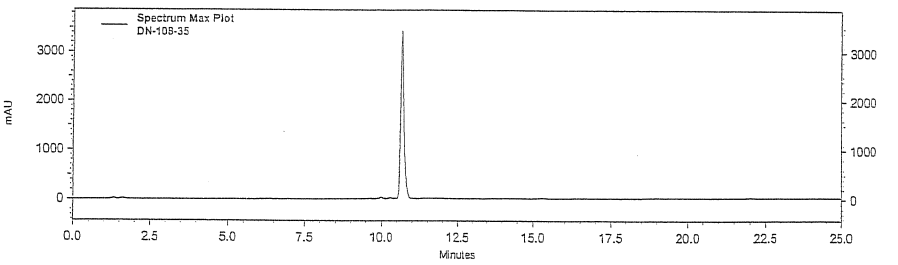
Data File: D:\Public\SYSTEM2\DN-108-35.dat

Analyst: System
Sample ID: DN-108-35 Vial: A06 Injection Volume: 10





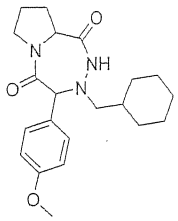




Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\C18 Standard.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

Data File: D:\Public\SYSTEM2\DN-108-35.dat

		9.989	79450	0.39
VV		10.652	20084325	99.61
VV				
	Totals		20163775	100.00



Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\C18 Standard.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

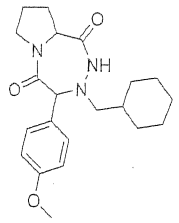
Area Percent Report

Data File: D:\Public\SYSTEM2\DN-108-35.dat

2: 255 nm, 5 nm
Results (Original)

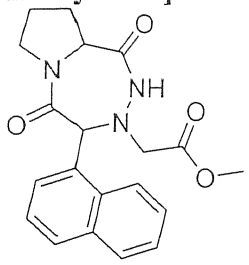
Name	Retention Time	Area	Area Percent	Integration Codes
	10.650	2929504	100.00	VV

Totals		2929504	100.00	

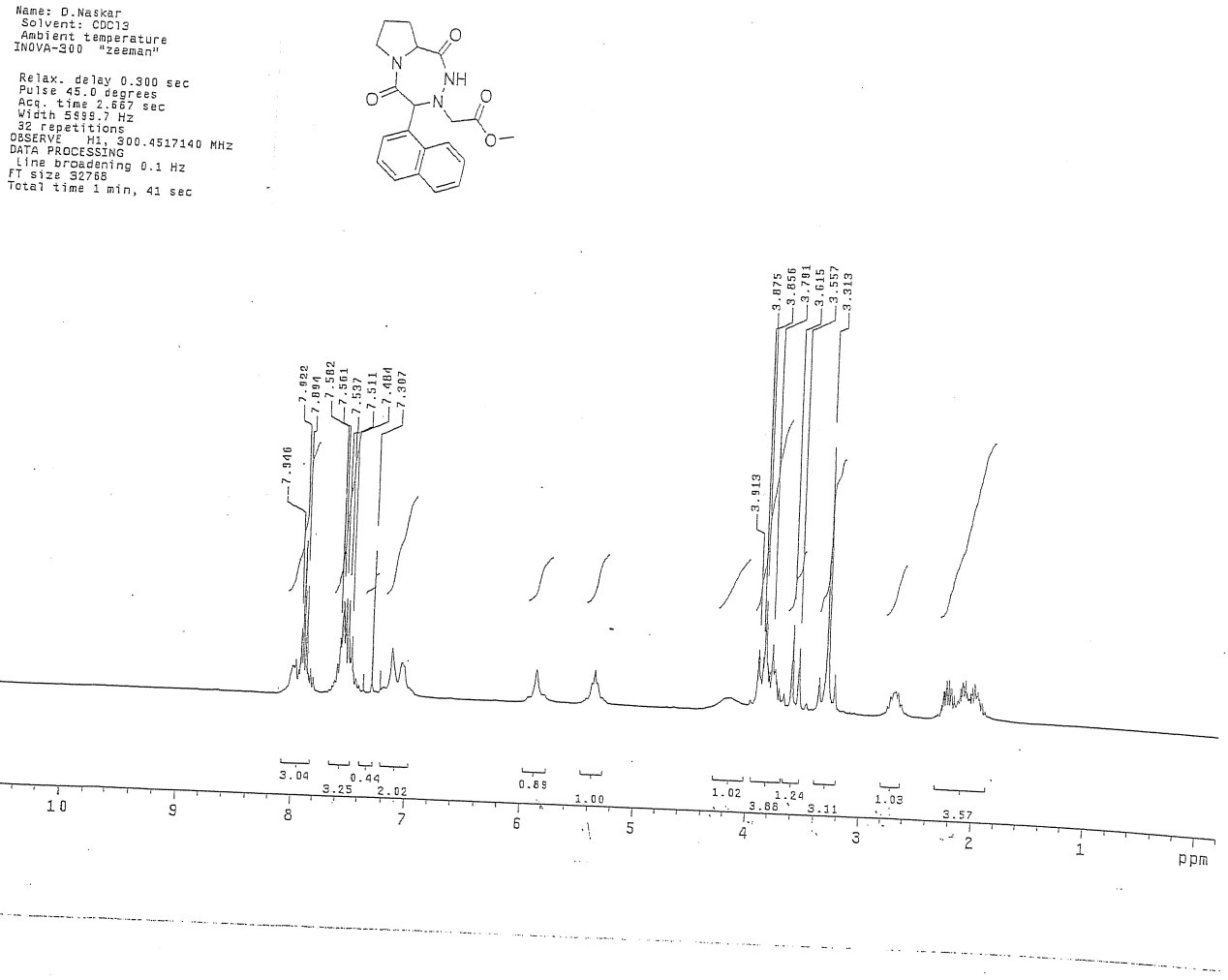


Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\C18 Standard.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

IX. Table 1, 2g: [(5-Naphthalen-1-yl-4,8-dioxo-octahydro-3a,6,7-triaza-azulen-6-yl)-acetic acid methyl ester]



White Solid; m.p (Met-Temp): 181°-182°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300 MHz): δ= 1.85-2.37 (m, 3H), 2.6-2.8 (m, 1H), 3.31(s, 3H), 3.55-3.61 (m, 1H), 3.79-3.91 (m, 3H), 5.25-5.45 (m, 1H), 5.75-5.98 (m, 1H), 7.01 (br s., 1H), 7.15 (m, 1H), 7.48-7.58 (m, 3H), 7.89-7.94 (m, 3H); ^{13}C NMR ($CDCl_3$, 75MHz): 22.85, 27.87, 49.33, 52.25, 56.71, 58.68, 67.03, 122.61, 125.7, 126.25, 127.27, 127.81, 129.64, 129.85, 129.99, 132.79, 134.47, 169.80, 172.41; LCMS (ELSD): 368.1 ($M+H^+$); HRMS: 368.1604 [Calculated for $C_{20}H_{22}N_3O_4$ 368.1610 $(M+H)^+$].



Current Data Parameters

EXPNO	50
PROCNO	1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	24999
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	4096
DW	26.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777600 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412006 MHz

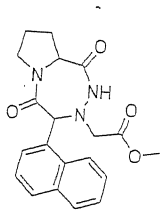
F2 - Processing parameters

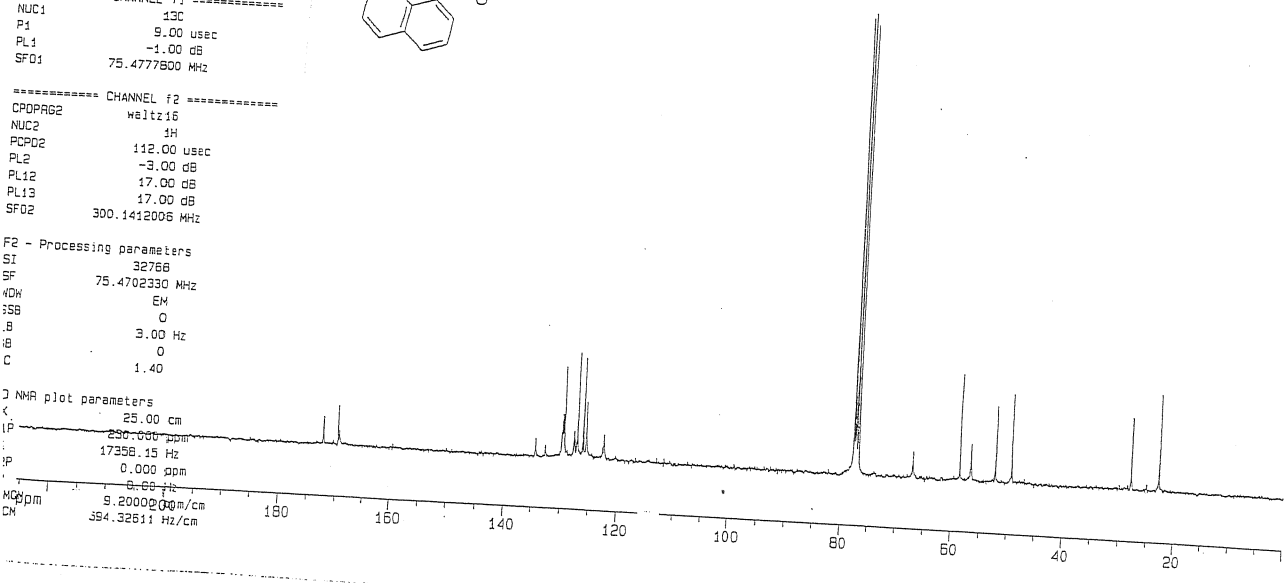
SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

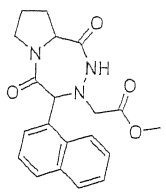
X	25.00 cm
XP	230.686 ppm
	17358.15 Hz
YP	0.000 ppm
	0.00 Hz
MCY	9.20000 ppm
CM	394.32611 Hz/cm

ppm
172.415
169.802
169.520
134.475
132.793
129.990
129.850
129.649
127.813
127.278
126.258
125.703
122.611
77.840
77.619
77.416
76.993
67.036
58.686
56.711
52.256
49.334
27.879
25.093
22.855

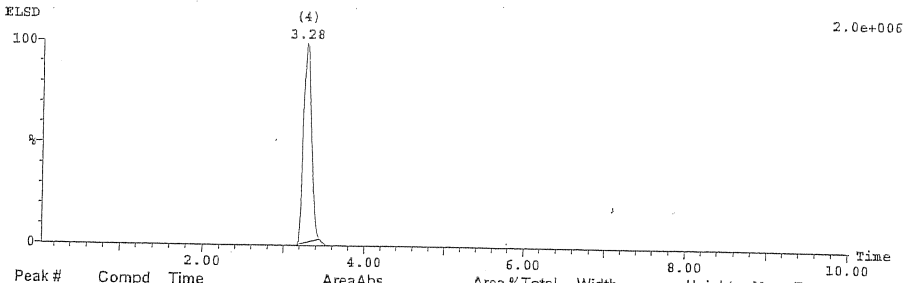




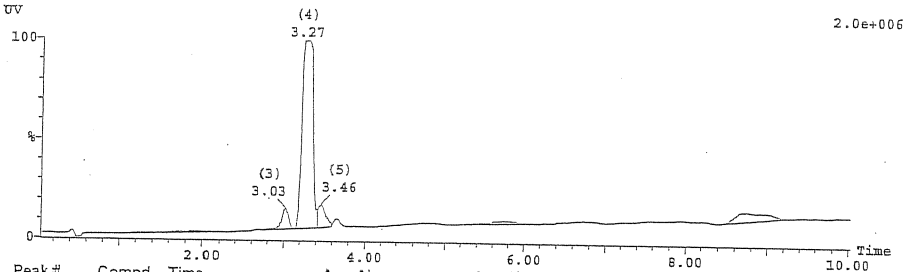
Sample Report (continued):



$C_{20}H_{21}N_3O_4$
Exact Mass: 367.15
Mol. Wt.: 367.40

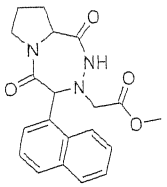


Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		1.07	1.783e+002	0.08	0.090	5.760e+003	
4		3.28	2.292e+005	99.68	0.280	1.988e+006	
6		5.85	5.461e+002	0.24	0.130	9.060e+003	



Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
2		1.99	1.173e+004	2.61	1.240	1.389e+004	
3		3.03	2.134e+004	4.75	0.390	2.036e+005	
4		3.27	2.923e+005	65.06	0.280	1.911e+006	
5		3.47	2.534e+004	5.64	0.180	2.336e+005	
6		5.76	1.071e+004	2.38	0.780	2.914e+004	
8		8.71	4.234e+004	9.42	0.820	9.554e+004	

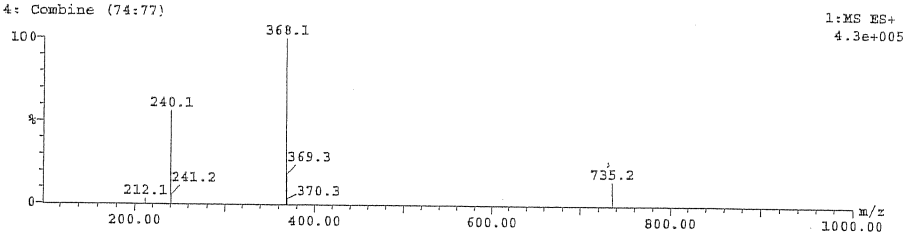
Sample Report (continued):



$C_{20}H_{21}N_3O_4$
Exact Mass: 367.15
Mol. Wt.: 367.40

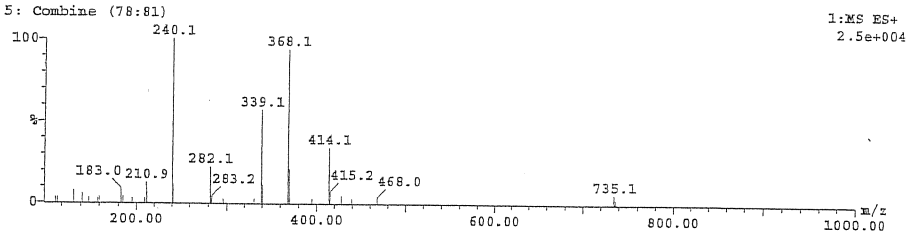
Mass Found Compound

4: Combine (74:77)



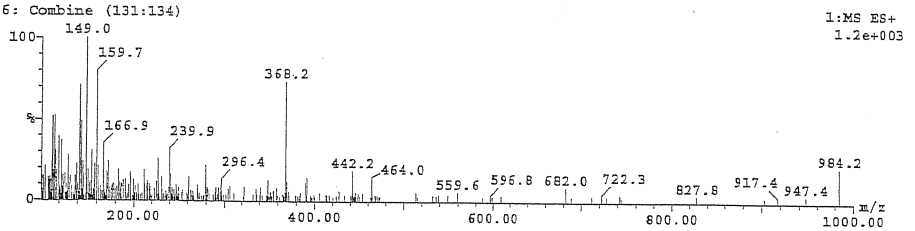
Mass Found Compound

5: Combine (78:81)

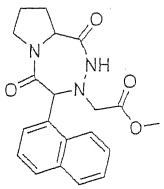


Mass Found Compound

6: Combine (131:134)

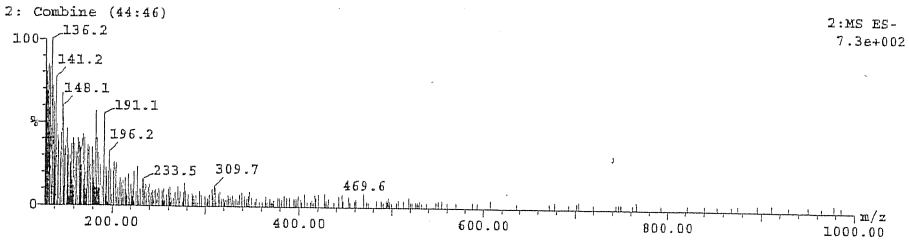


Sample Report (continued):

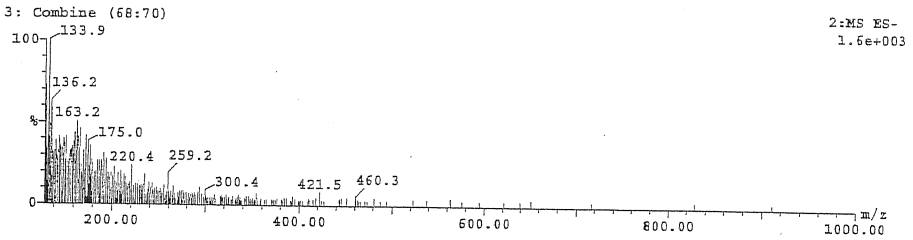


$C_{20}H_{21}N_3O_4$
Exact Mass: 367.15
Mol. Wt.: 367.40

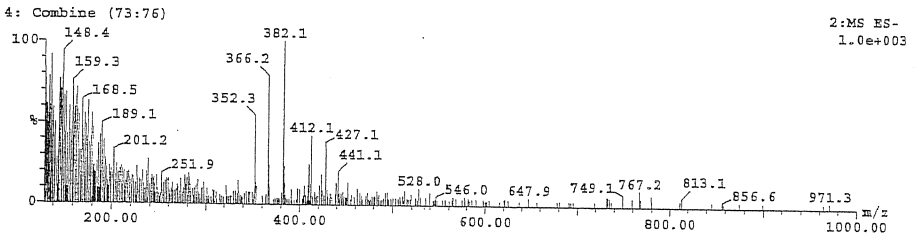
Mass Found Compound



Mass Found Compound



Mass Found Compound

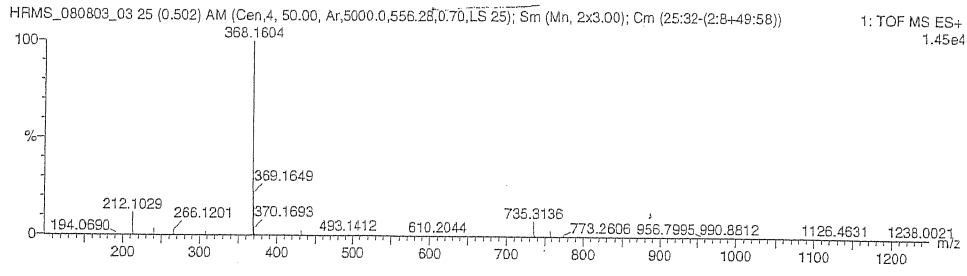


Elemental Composition Report

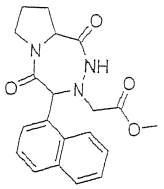
Single Mass Analysis

Tolerance = 30.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

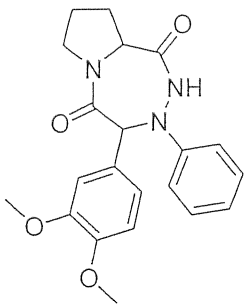


Minimum:				-1.5		
Maximum:		200.0	30.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
368.1604	368.1610	-0.7	-1.8	11.5	1	C20 H22 N3 O4

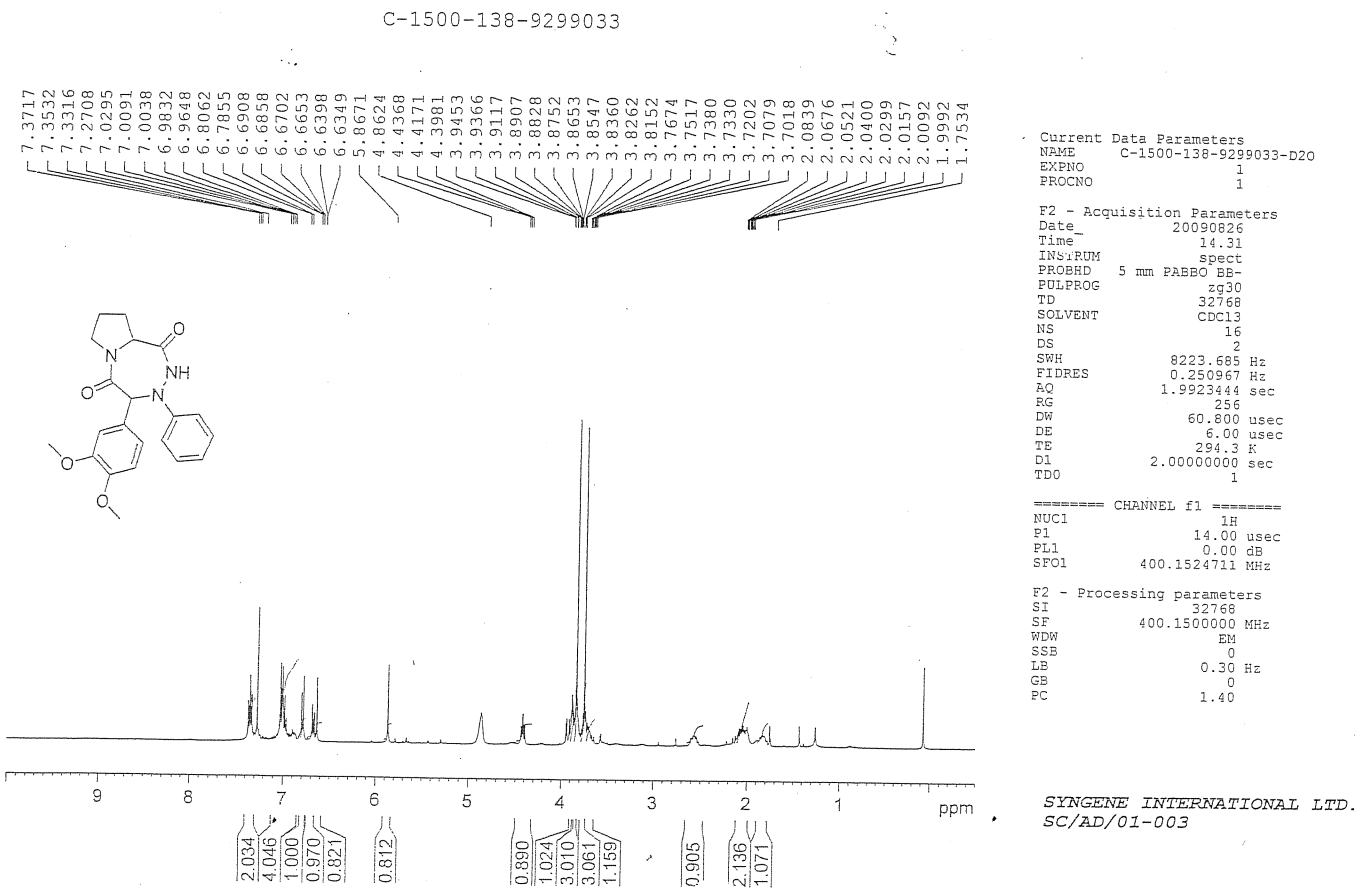


$C_{20}H_{21}N_3O_4$
Exact Mass: 367.15
Mol. Wt.: 367.40

X. Table 1, 2h: [5-(3,4-Dimethoxy-phenyl)-6-phenyl-hexahydro-3a,6,7-triaza-azulene-4,8-dione]

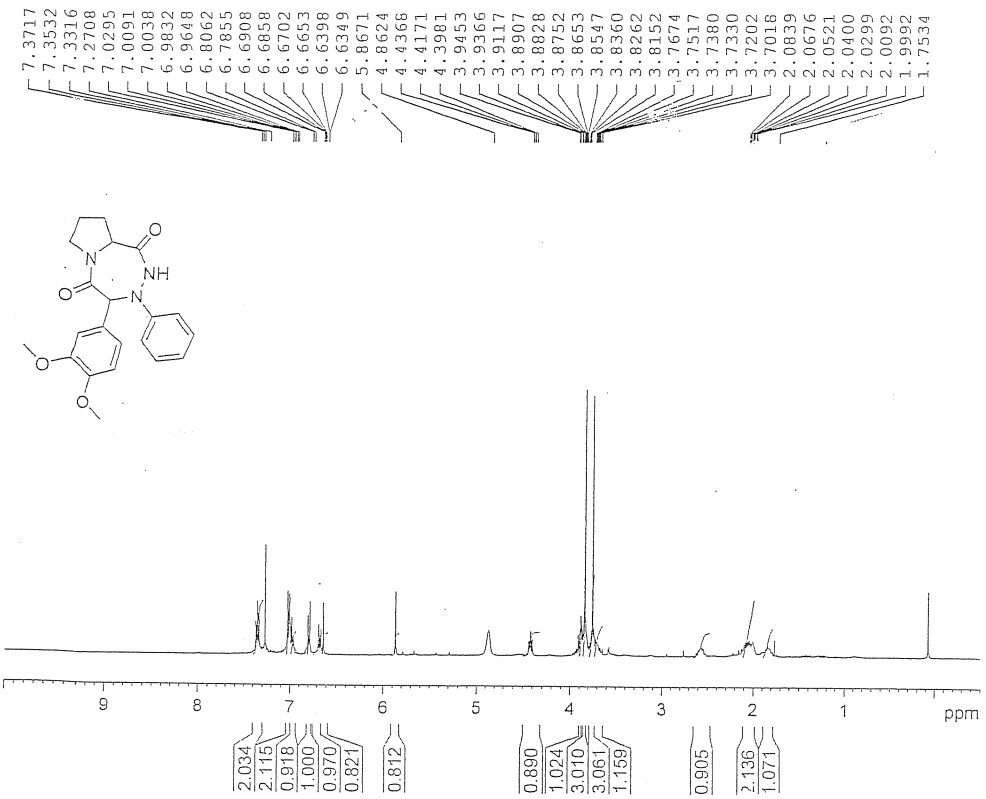


White solid; m.p (Met-Temp): 73°-74°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.75 (m, 1H), 1.99-2.08 (m, 2H), 2.55-2.60 (m, 1H), 3.70-3.74 (m, 1H), 3.75 (s, 3H), 3.82 (s, 3H), 3.87-3.94 (m, 1H), 4.39-4.44 (m, 1H), 5.87 (s, 1H), 6.63 (s, 1H), 6.67-6.69 (d, J=8.2, 1H), 6.79-6.81 (d, J=8.28, 1H), 6.96-7.03 (m, 4H), 7.33-7.37 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.07, 27.87, 48.71, 55.77, 57.83, 66.42, 111.19, 111.54, 112.76, 120.92, 121.17, 126.79, 129.92, 145.88, 149.12, 149.27, 168.89, 172.55; LCMS (UV): 382.1 ($M+H^+$). Anal. Calcd. for $C_{21}H_{23}N_3O_4$: C, 66.13; H, 6.08; N, 11.02. Found: C, 66.22; H, 6.12; N, 11.11.



SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

C-1500-138-9299033-D2O




```
Current Data Parameters
NAME      C-1500-138-9299033-D2O
EXPNO                 1
PROCNO                1

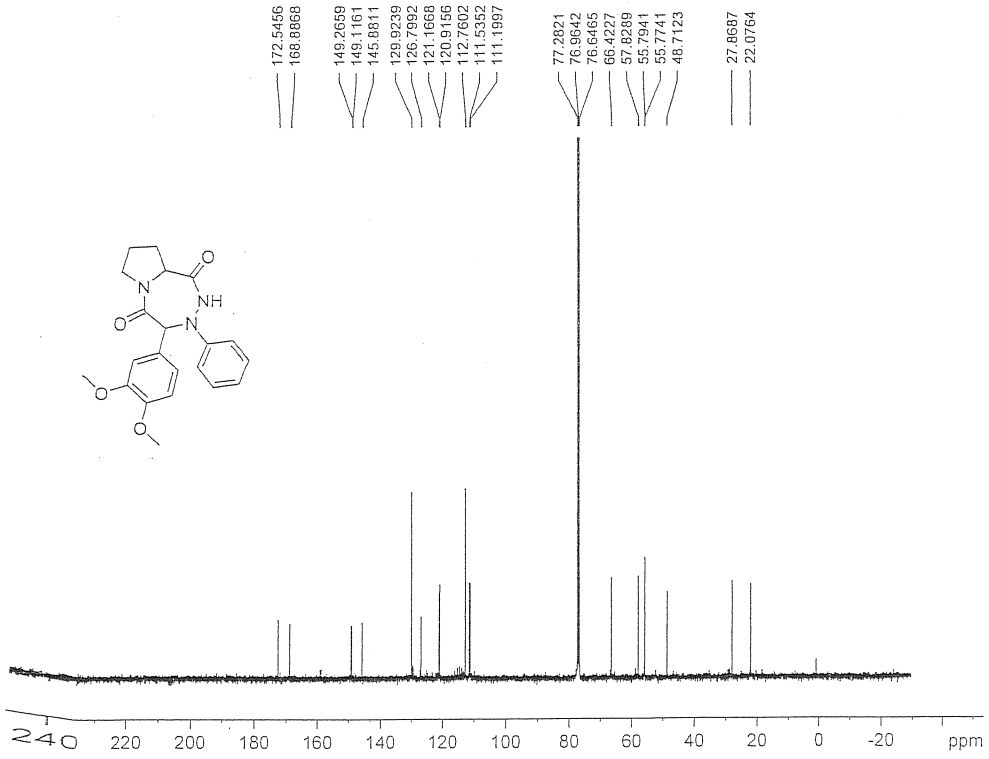
F2 - Acquisition Parameters
Date            20090826
                  14.31
   RUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             1.9923444 sec
RG                  256
DW                60.800 usec
DE                 6.00 usec
TE                294.3 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                32768
SF          400.1500000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

C -1500-138-9199033




```
Current Data Parameters
NAME      C -1500-138-9199033
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20090825
Time                  21.48
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                    1547
DS                       2
SWH             30120.482 Hz
FIDRES           0.919204 Hz
AQ               0.5439988 sec
RG                   912.3
DW                  16.600 usec
DE                   6.00 usec
TE                  297.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -1.00 dB
SFO1         100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2         400.2316009 MHz

F2 - Processing parameters
SI                   32768
SF           100.6379211 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

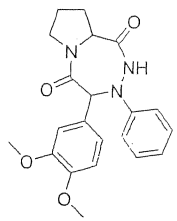
SYNGENE INTERNATIONAL LTD.
SC/AD/01-002

Syngene International Pvt Ltd
A Biocon Company

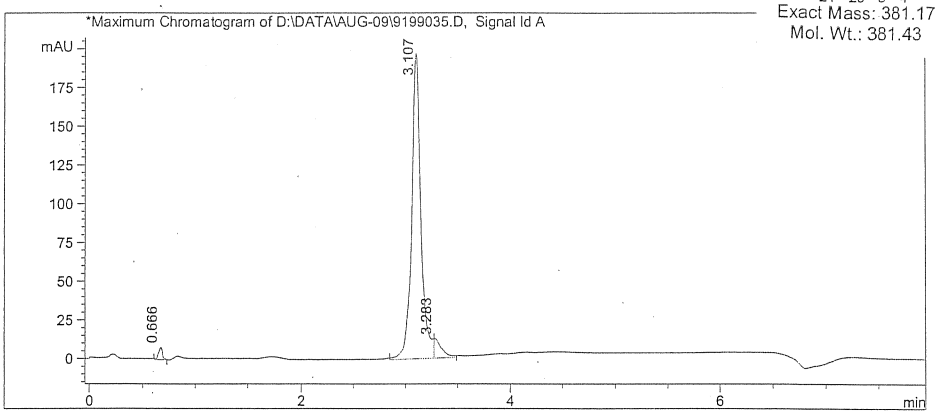
Data file	: D:\DATA\AUG-09\9199035.D	Vial No.	: Vial 6
Injection Date	: 27/08/2009	Injection vol	: 2 uL
Sample Name	:C-1500-138	Acq Method	:AT3070FM1.m

Method info :Method : A : 0.1 % HCOOH, B : MEOH , Flow : 1.0 mL/min
column :Atlantis dC18 (50*4.6) mm, 5.0u

Time	% B
0	30
3	95
5	95
5.5	30
8	30



$C_{21}H_{23}N_3O_4$
Exact Mass: 381.17
Mol. Wt.: 381.43



Peak No	RT min	Area	Area %
1	0.666	1.944e+001	1.473
2	3.107	1.239e+003	93.933
3	3.283	6.061e+001	4.594

Analysed By :

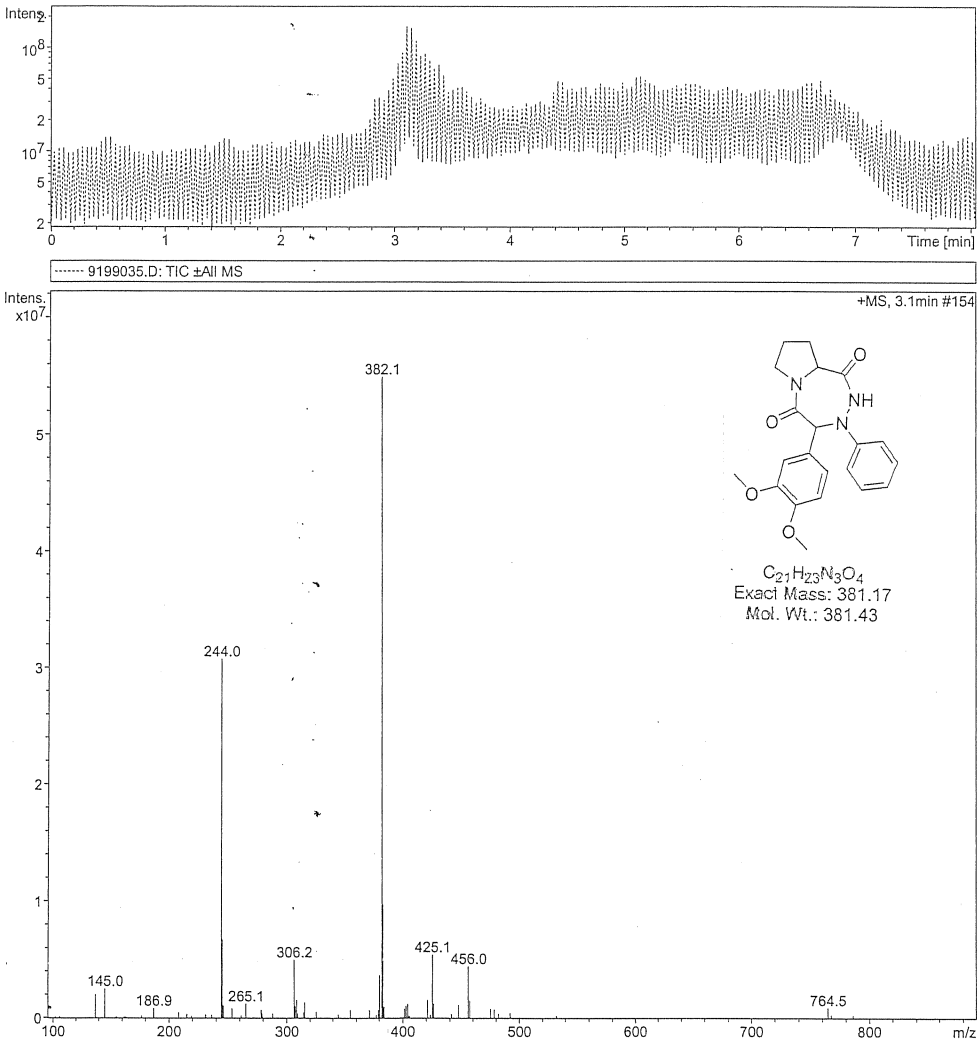
Instrument Code : SC/AD/10-002

MASS REPORT

SYNGENE INTL PVT LTD

Data File: D:\DATA\AUG-09\9199035.D **Instrument:** LC-MSD-Trap-XCT

Method: AT3070FM1.M **Sample Name:** C-1500-138



Analysed By :

Instrument code : SC/AD/10-002

Syngene International Ltd
A Biocon Company

HPLC REPORT

Data file :C:\CHEM32\1\DATA\AUG09\9199033.D Vial No. : Vial 3

Injection Date :26/08/09 01:37:50 PM Injection vol : 1 µl

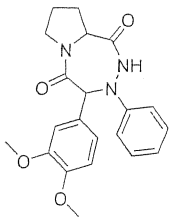
Sample Name :C-1500-138 Operator : ANAND

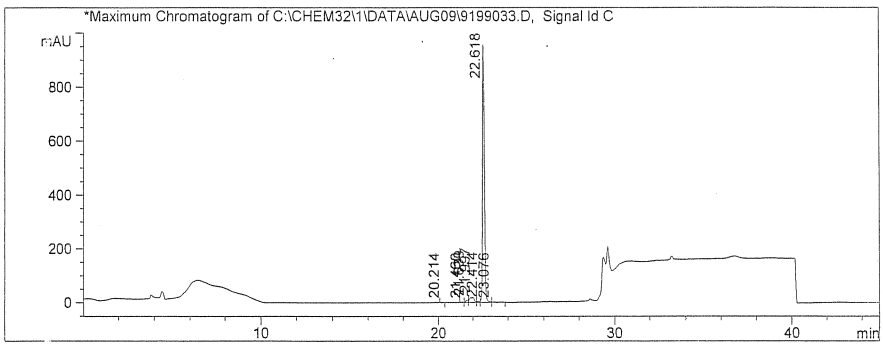
Acq Method ->C:\CHEM32\1\METHODS\SY_TM9010.M

Sample info :

Method info : A:0.1%TFA B:MeOH
Symmetry C18(4.6X250)mm,5um
Flow:0.7mL/min

Time	% B
0	10
20	70
25	100
35	100
38	10
45	10





Peak No	RT min	Area	Area %
1	20.214	7.677	0.100
2	21.460	6.722	0.087
3	21.639	86.589	1.125
4	21.997	337.231	4.380
5	22.414	46.021	0.598
6	22.618	7187.858	93.363
7	23.076	26.702	0.347

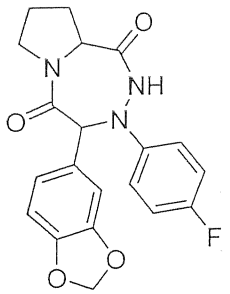
End of report

Analysed By :

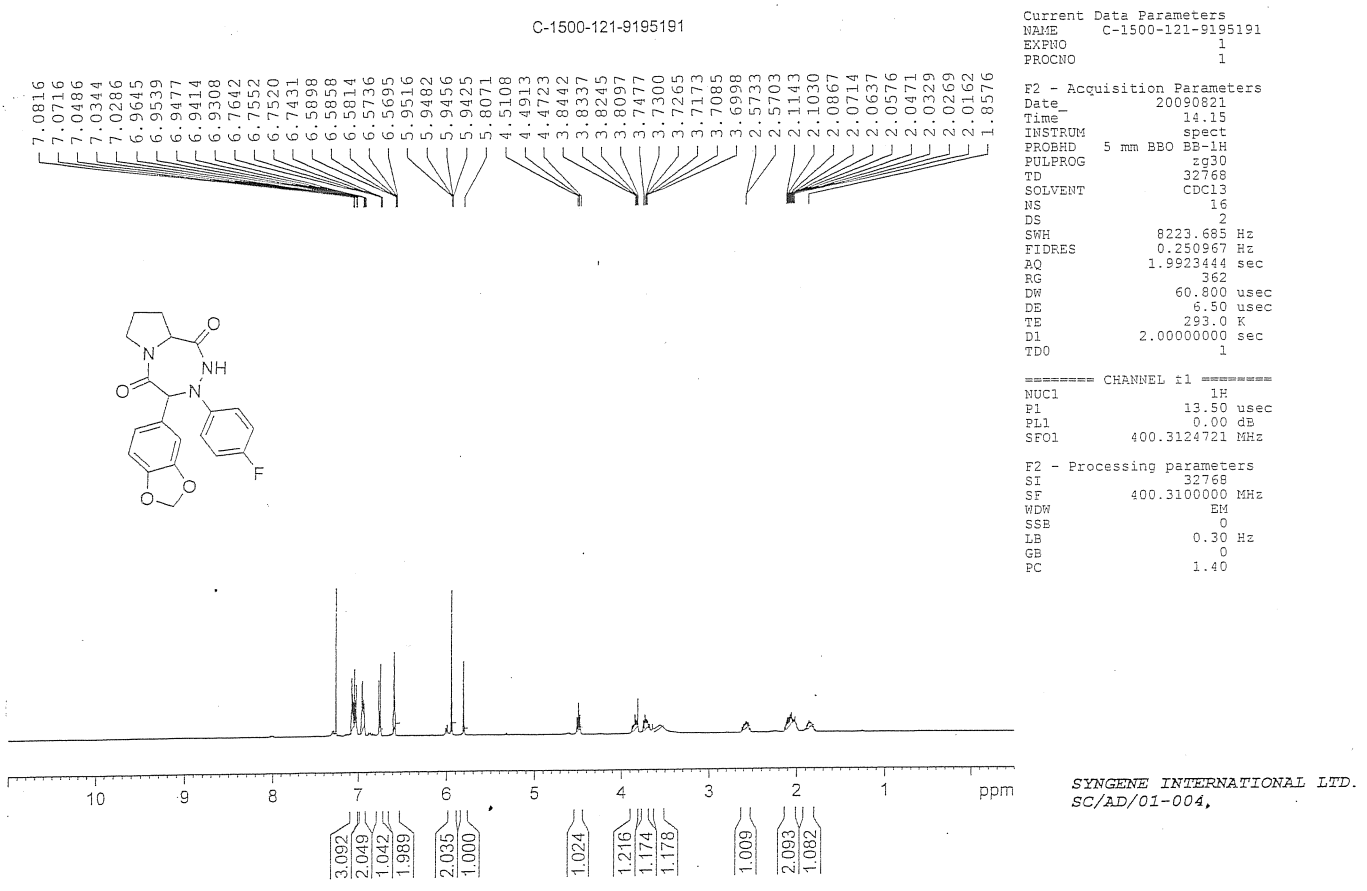
Instrument Code : SC/AD/04-063

Checked By :

XI. Table 1, 2i: [5-Benzo[1,3]dioxol-5-yl-6-(4-fluoro-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]

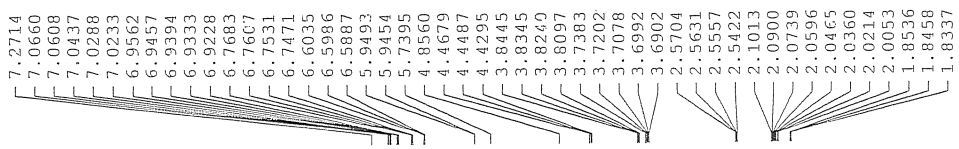


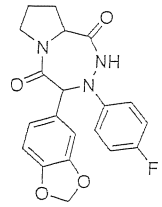
White solid; m.p (Met-Temp): 95°-96°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.86 (m, 1H), 2.02-2.11 (m, 2H), 2.57 (m, 1H), 3.70-3.75 (m, 1H), 3.81-3.84 (m, 1H), 4.47-4.51 (m, 1H), 5.81 (s, 1H), 5.95 (s, 2H), 6.57-6.59 (m, 2H), 6.74-6.76 (d, J=8.44, 1H), 6.93-6.96 (m, 2H), 7.03-7.08 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.09, 27.82, 48.69, 57.80, 67.20, 101.33, 108.62, 108.74, 114.31, 116.35, 116.58, 122.16, 127.90, 142.11, 147.91, 148.10, 156.44, 158.84, 168.28, 172.50; LCMS (UV): 384.2 ($M+H^+$). Anal. Calcd. for $C_{20}H_{18}FN_3O_4$: C, 62.66; H, 4.73; N, 10.96. Found: C, 62.73; H, 4.81; N, 10.89.

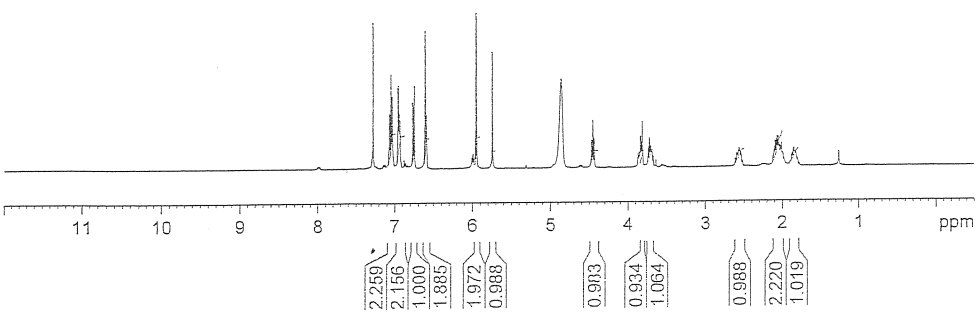


SYNGENE INTERNATIONAL LTD.
SC/AD/01-004.

C-1500-121-9195191-D2O







Current Data Parameters
NAME C-1500-121-9195191-D2O
EXPNO 1
PROCNO 1

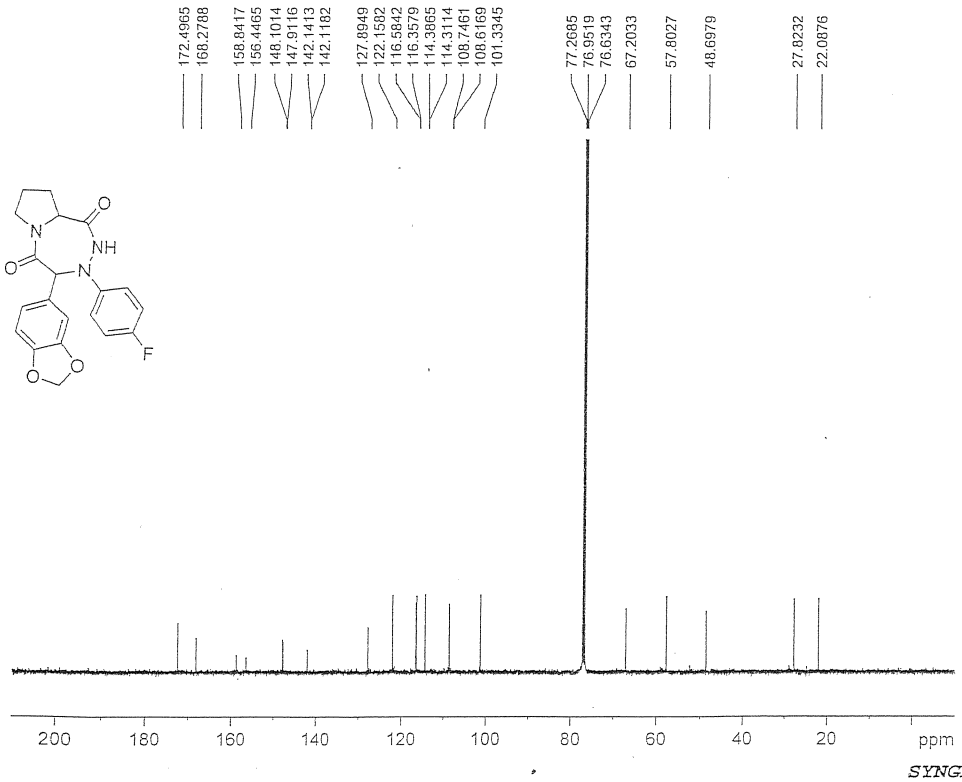
F2 - Acquisition Parameters
Date_ 20000422
Time 16
INSTRUM ject
PROBHD 5 mm PADUO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 294.5 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 32768
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

C-1500-121-9195191



Current Data Parameters

NAME	C-1500-121-9195191
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090821
Time	19.25
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1775
DS	2
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	0.5439988 sec
RG	1024
DW	16.600 usec
DE	6.00 usec
TE	296.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.30 usec
PL1	-1.00 dB
SFO1	100.6499905 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	15.00 dB
PL13	15.00 dB
SFO2	400.2316009 MHz

F2 - Processing parameters

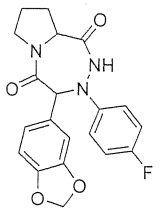
SI	32768
SF	100.6379211 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SYNGENE INTERNATIONAL LTD.
SC/AD/01-002

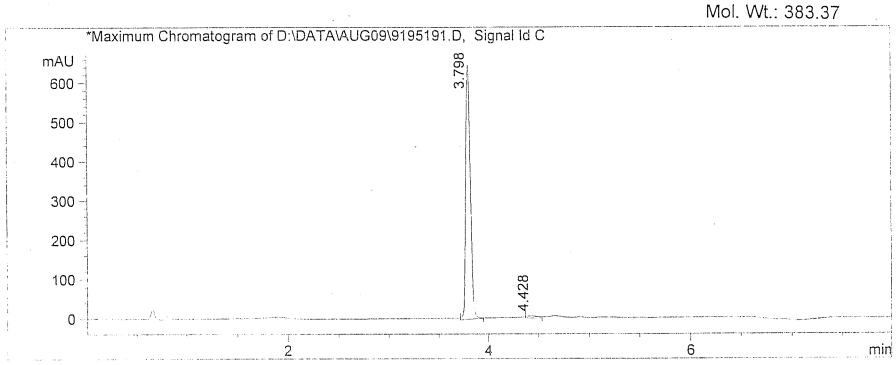
Syngene International Ltd LC/MS REPORT
A Biocon Company

Data file : D:\DATA\AUG09\9195191.D
Vial No. : P1-D-06
Injection Date : 8/21/2009 6:08:56 PM
Injection vol : 2ul
Sample Name : C-1500-121
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

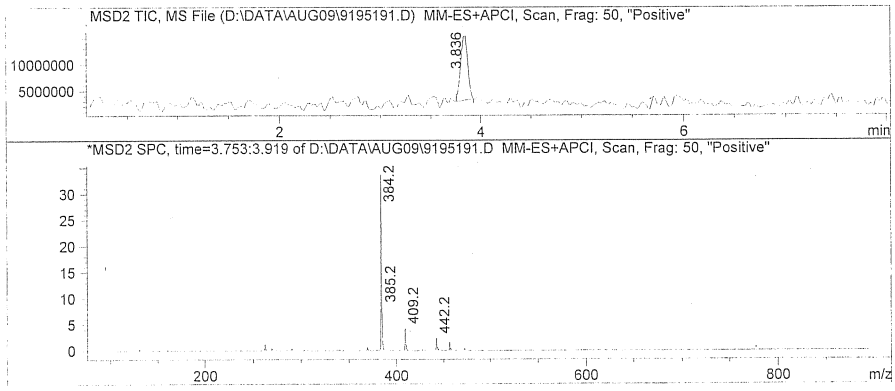
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{20}H_{18}FN_3O_4$
Exact Mass: 383.13
Mol. Wt.: 383.37



Peak No	RT min	Area	Area %
1	3.798	2.183e+003	97.890
2	4.428	4.707e+001	2.110



Analysed by :

Instrument Code : SC/AD/10-014

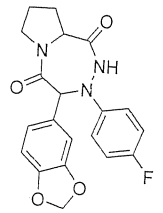
Syngene International Ltd
A Biocon Company

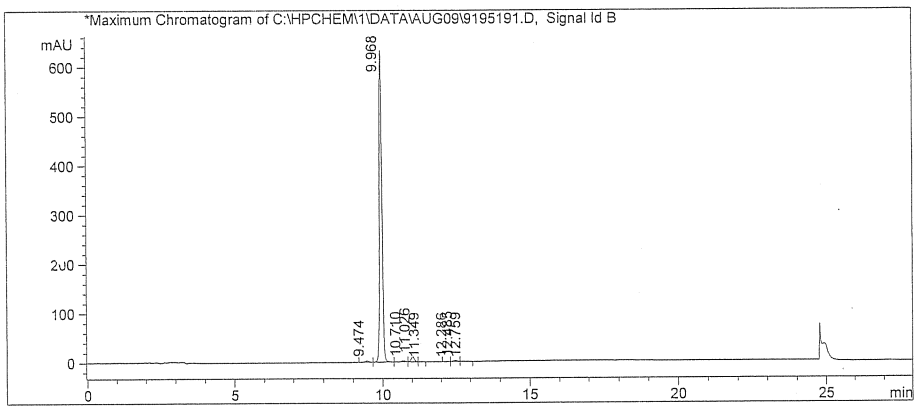
HPLC REPORT

Data file	:C:\HPCHEM\1\DATA\AUG09\9195191.D	Vial location:	Vial 11
Injection Date	:21/Aug/2009 12:42:06 PM	Injection vol :	3 µl
Sample Name	:C-1500-121	Operator :	VINAYAGAM
Sample info	:	Acq Method :	SY_A7030.M

Method info : Mobile Phase: A:10mM NH4OAc B: ACN
Column:Symmetry C18(4.6X250)mm,5µ SC/LC/1062
Flow: 0.8mL/min

TIME	%B
0	30
15	100
20	100
23	30
28	30





Peak No	RT min	Area	Area %	Name
1	9.474	22.754	0.57	
2	9.968	3850.311	96.04	
3	10.710	20.647	0.51	
4	11.026	84.702	2.11	
5	11.349	4.084	0.10	
6	12.286	2.092	0.05	
7	12.485	20.720	0.52	
8	12.759	3.845	0.10	

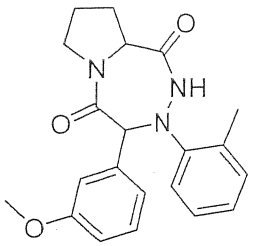
End of report

Analysed By : A.

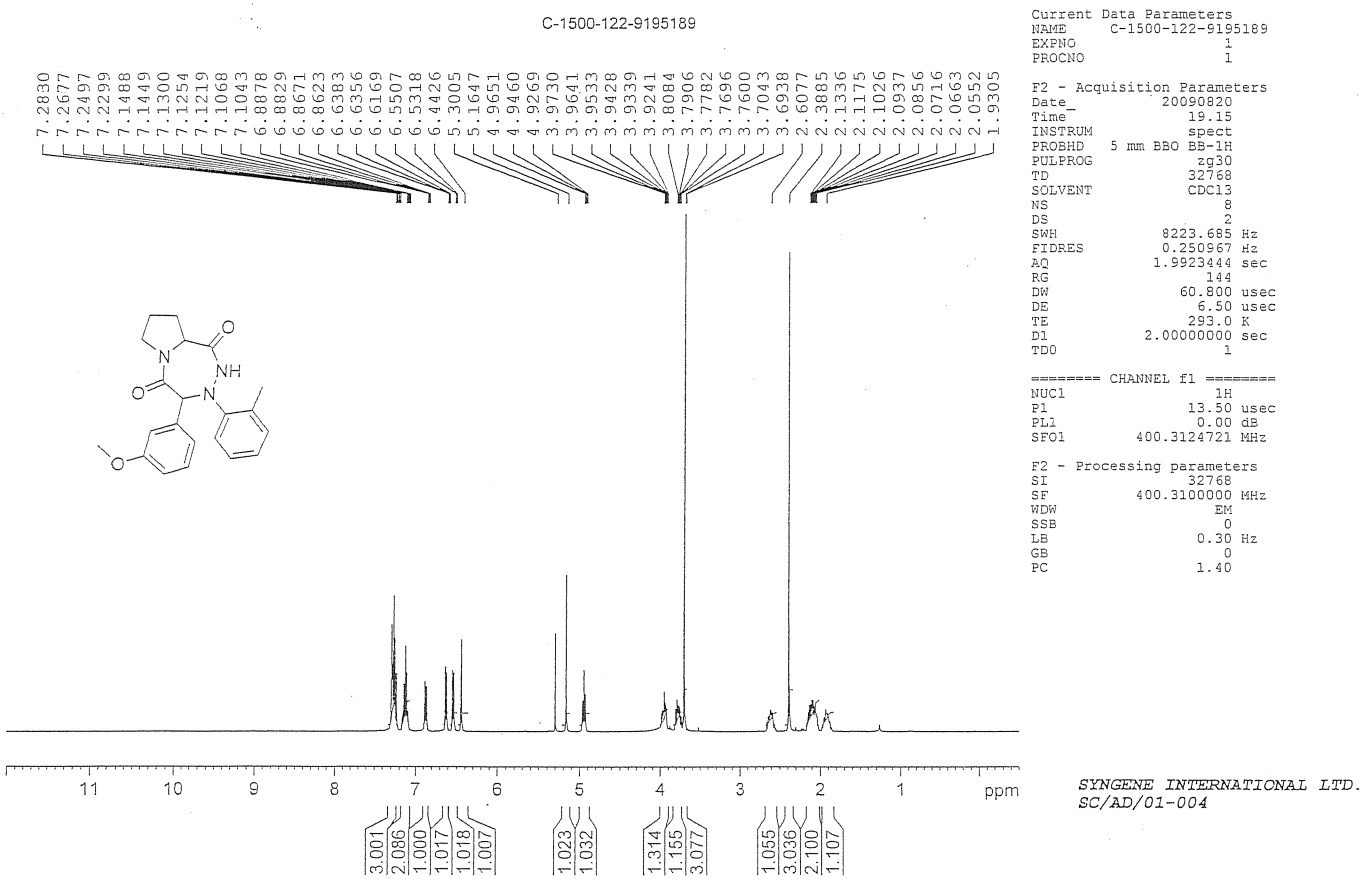
Checked By : RM

Instrument Code : SC/AD/04-15

XII. Table 1, 2j: [5-(3-Methoxy-phenyl)-6-o-tolyl-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid; m.p (Met-Temp): 78°-79°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.93 (m, 1H), 2.06-2.13 (m, 2H), 2.39 (s, 3H), 2.61 (m, 1H), 3.69 (s, 3H), 3.76-3.81 (m, 1H), 3.92-3.97 (m, 1H), 4.93-4.97 (m, 1H), 5.16 (s, 1H), 6.44 (s, 1H), 6.53-6.55 (d, J=7.56, 1H), 6.61-6.63 (d, J=7.6, 1H), 6.86-6.88 (d, J=8.24, 1H), 7.10-7.15 (m, 2H), 7.22-7.28 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 19.16, 22.38, 27.81, 48.69, 55.20, 58.34, 71.87, 114.22, 114.88, 119.75, 121.33, 125.22, 126.72, 129.85, 130.09, 132.10, 134.76, 145.43, 159.69, 168.60, 172.36; LCMS (UV): 366.2 ($M+H^+$). Anal. Calcd. for $C_{21}H_{23}N_3O_3$: C, 69.02; H, 6.34; N, 11.50. Found: C, 69.14; H, 6.40; N, 11.55.

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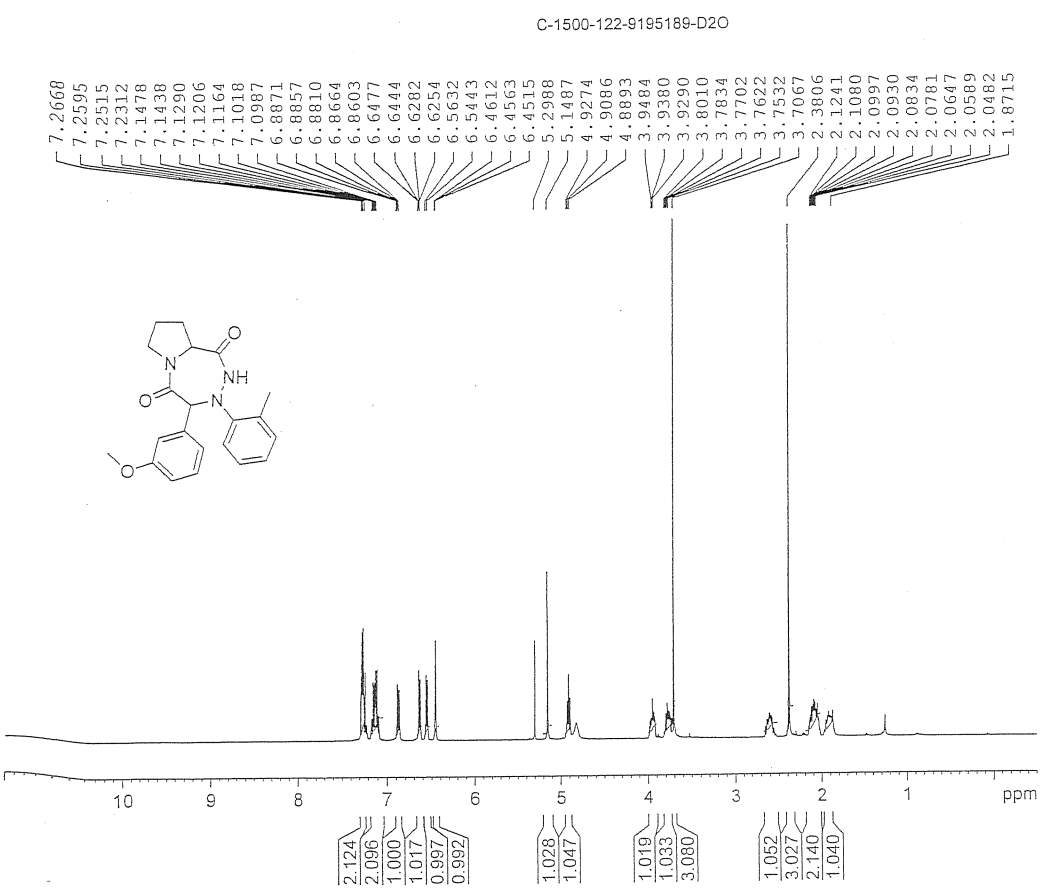
Current Data Parameters
NAME      C-1500-122-9195189
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20090820
Time                  19.15
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8223.685 Hz
FIDRES            0.250967 Hz
AQ                1.9923444 sec
RG                      144
DW                 60.800 usec
DE                  6.50 usec
TE                  293.0 K
D1              2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.50 usec
PL1                    0.00 dB
SFO1          400.3124721 MHz

F2 - Processing parameters
SI                    32768
SF            400.3100000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004




```
Current Data Parameters
NAME      C-1500-122-9195189-D2O
EXPNO                         1
PROCNO                        1

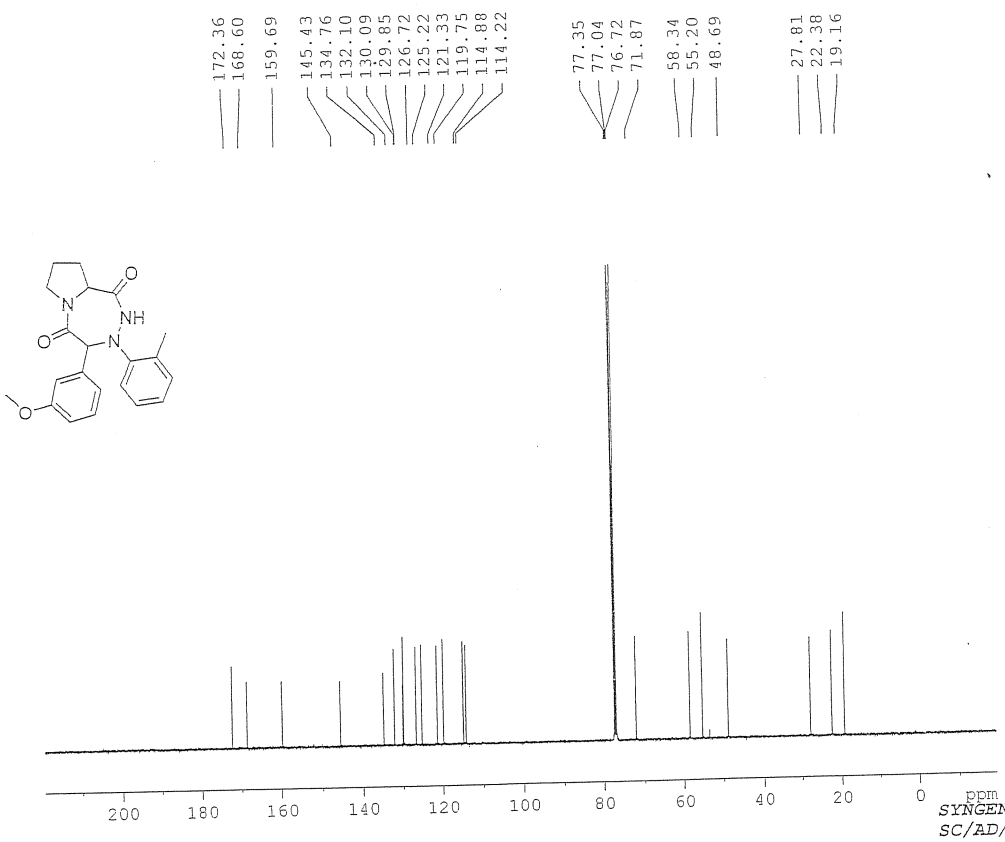
F2 - Acquisition Parameters
Date_               20090821
Time                   16.01
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.250967 Hz
AQ                1.9923444 sec
RG                       161
DW                  60.800 usec
DE                   6.50 usec
TE                   293.0 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.50 usec
PL1                    0.00 dB
SFO1           400.3124721 MHz

F2 - Processing parameters
SI                     32768
SF             400.3100000 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004

C-1500-122-9195189



Current Data Parameters

NAME	C-1500-122-9195189
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090821
Time	0.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	2000
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	161
DW	20.800 usec
DE	6.00 usec
TE	295.3 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	7.13 usec
PL1	-3.00 dB
SFO1	100.6278593 MHz

======== CHANNEL f2 ========

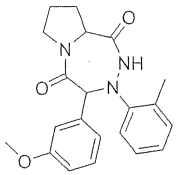
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.14 dB
PL13	15.00 dB
PL2	0.00 dB
SFO2	400.1516006 MHz

F2 - Processing parameters

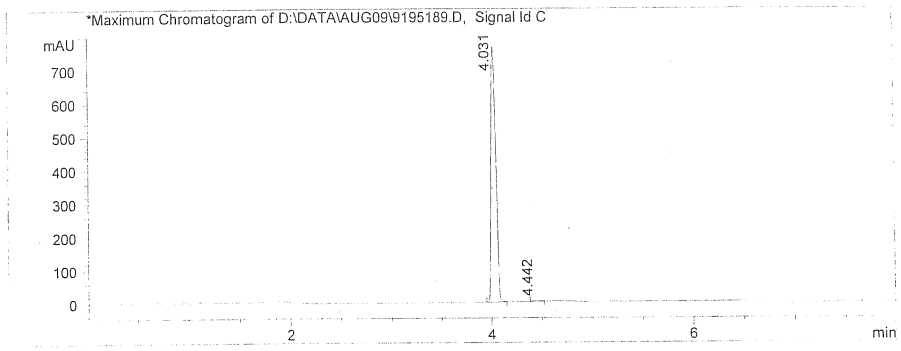
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Data file : D:\DATA\AUG09\9195189.D
Vial No. : P2-F-05
Injection Date : 8/20/2009 7:47:15 PM
Injection vol : 2ul
Sample Name : C-1500-122
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

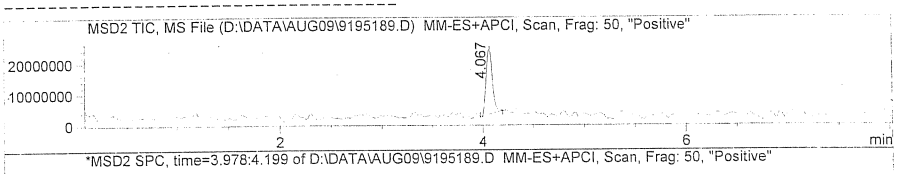
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HCOOH B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY

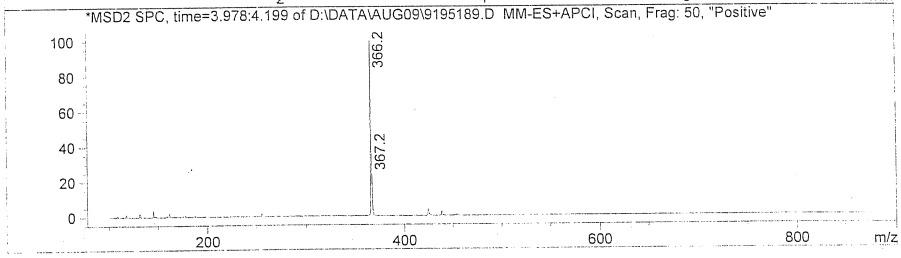


$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43



Peak No	RT min	Area	Area %
1	4.031	2.479e+003	99.180
2	4.442	2.049e+001	0.820





Analysed by :

Instrument Code : SC/AD/10-014

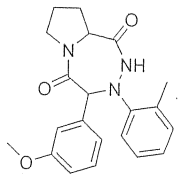
Syngene International Ltd
A Biocon Company

HPLC REPORT

Data file	:C:\HPCHEM\1\DATA\AUG09\9195189.D	Vial location:	Vial 4
Injection Date	:21/Aug/2009 9:41:00 AM	Injection vol :	1 µl
Sample Name	:C-1500-122	Operator :	VINAYAGAM
Sample info	:	Acq Method :	SY_A7030.M

Method info : Mobile Phase: A:10mM NH4OAc B: ACN
Column:Symmetry C18(4.6X250)mm,5µ SC/LC/1062
Flow: 0.8mL/min

TIME	%B
0	30
15	100
20	100
23	30
28	30



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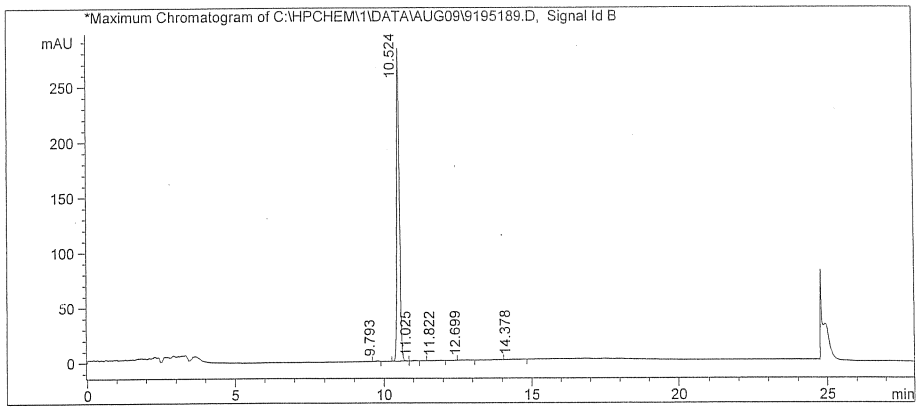


Peak No	RT min	Area	Area %	Name
1	9.793	4.988	0.28	
2	10.524	1772.189	98.43	
3	11.025	4.101	0.23	
4	11.822	4.106	0.23	
5	12.699	7.240	0.40	
6	14.378	7.914	0.44	

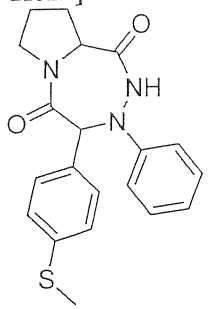
End of report

nalysed By :

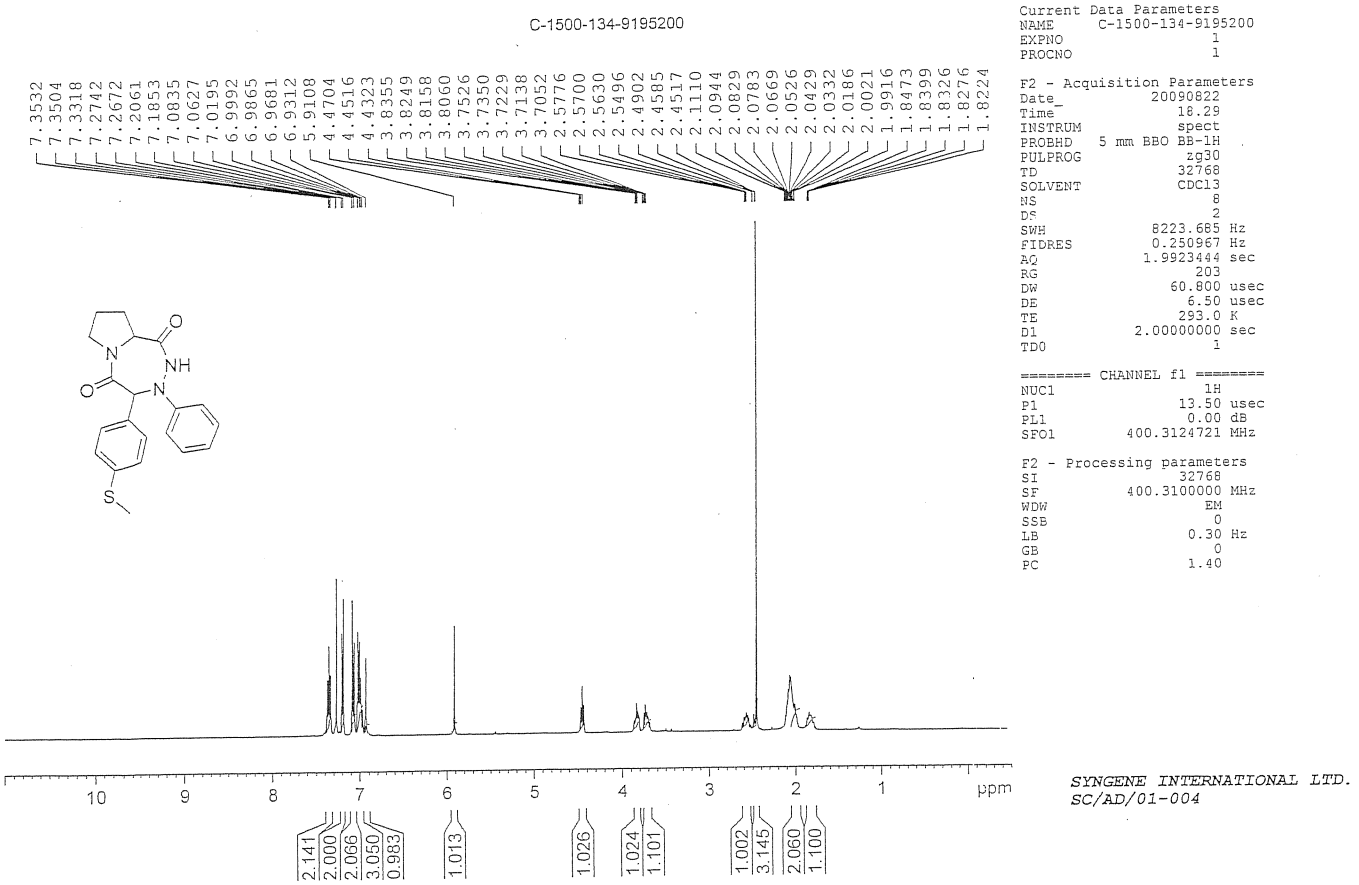
Checked By :

Instrument Code : SC/AD/04-15

XIII. Table 1, 2k: [5-(4-Methylsulfanyl-phenyl)-6-phenyl-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid; m.p (Met-Temp): 189°-190°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.82-1.85 (m, 1H), 1.99-2.11 (m, 2H), 2.45 (s, 3H), 2.54-2.58 (m, 1H), 3.71-3.75 (m, 1H), 3.81-3.84 (m, 1H), 4.43-4.47 (m, 1H), 5.91 (s, 1H), 6.93 (s, 1H), 6.97-7.02 (m, 3H), 7.06-7.08 (d, J=8.32, 2H), 7.19-7.21 (d, J=8.32, 2H), 7.33-7.35 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 17.94, 24.59, 30.36, 50.99, 60.02, 68.68, 115.11, 123.54, 129.16, 131.34, 132.38, 134.05, 141.79, 148.42, 170.31, 175.06; LCMS (UV): 368.2 ($M+H^+$). Anal. Calcd. for $C_{20}H_{21}N_3O_2S$: C, 65.37; H, 5.76; N, 11.44. Found: C, 65.32; H, 5.81; N, 11.38.



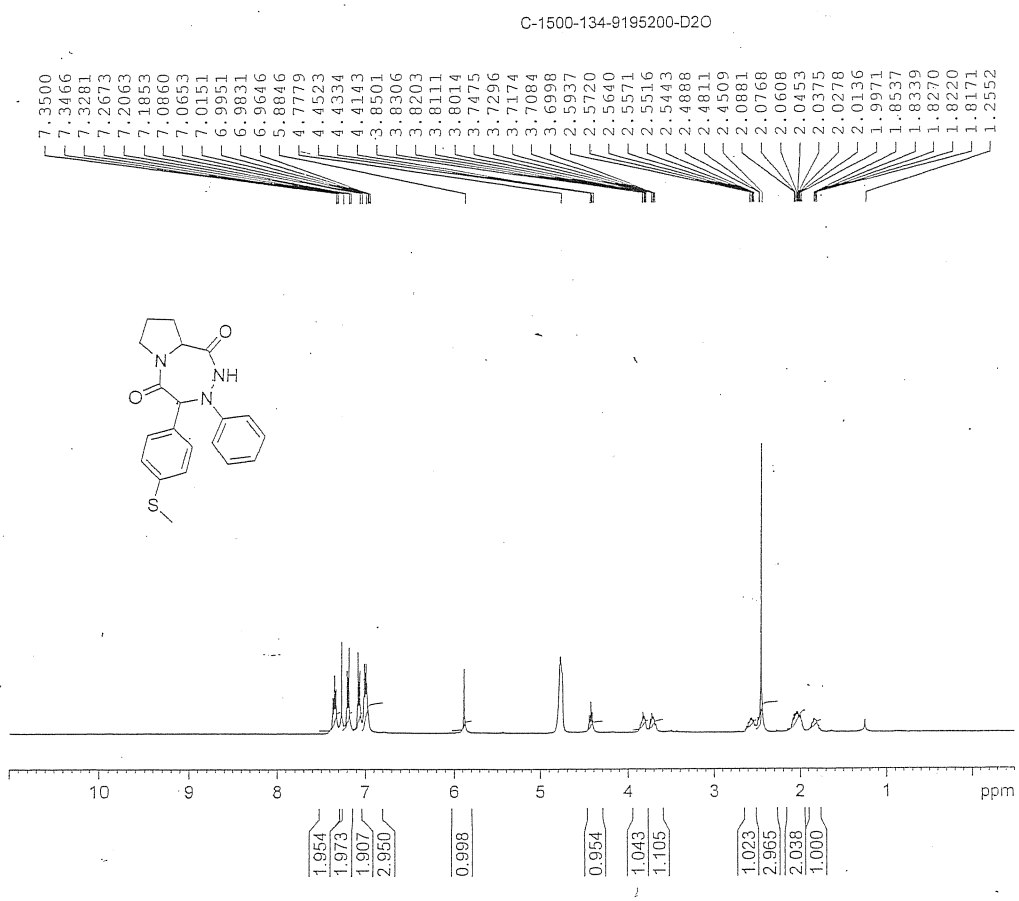

```
Current Data Parameters
NAME      C-1500-134-9195200
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20090822
Time                  18.29
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8223.685 Hz
FIDRES             0.250967 Hz
AQ                 1.9923444 sec
RG                      203
DW                   60.800 usec
DE                    6.50 usec
TE                   293.0 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.50 usec
PL1                    0.00 dB
SFO1          400.3124721 MHz

F2 - Processing parameters
SI                        32768
SF             400.3100000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004




```
Current Data Parameters
NAME      C-1500-134-9195200-D2O
EXPNO                 1
PROCNO                1

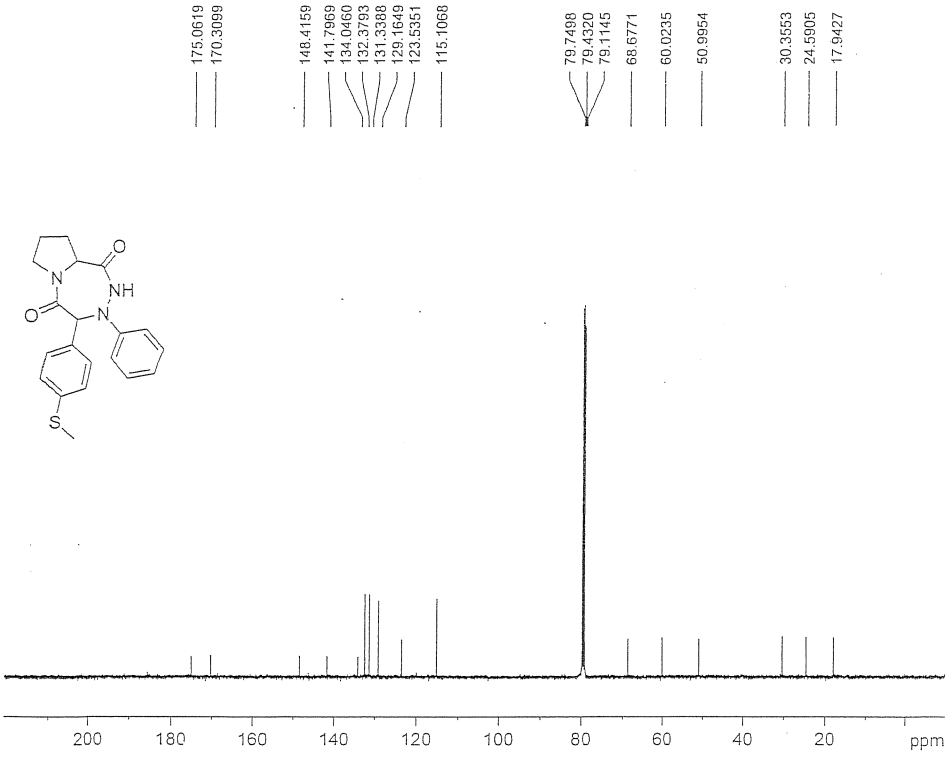
F2 - Acquisition Parameters
Date_          20090824
Time               8.28
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                 203
DW              60.800 usec
DE               6.50 usec
TE              293.0 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              13.50 usec
PL1              0.00 dB
SFO1       400.3124721 MHz

F2 - Processing parameters
SI               32768
SF        400.3100000 MHz
WDW                 EM
SSB                  0
LB               0.30 Hz
GB                   0
PC               1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004

C-1500-134-9195200



Current Data Parameters

NAME	C-1500-134-9195200
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090824
Time	11.51
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	2048
DS	2
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	0.5439988 sec
RG	645.1
DW	16.600 usec
DE	6.00 usec
TE	296.4 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.30 usec
PL1	-1.00 dB
SFO1	100.6499905 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	15.00 dB
PL13	15.00 dB
SFO2	400.2316009 MHz

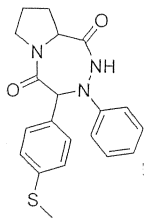
F2 - Processing parameters

SI	32768
SF	100.6379211 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

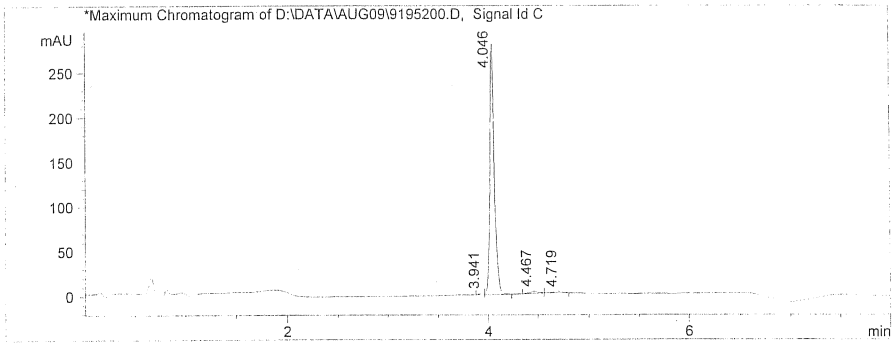
SYNGENE INTERNATIONAL LTD.
SC/AD/01-002

Data file : D:\DATA\AUG09\9195200.D
Vial No. : P1-B-09
Injection Date : 8/24/2009 11:59:28 AM
Injection vol : 2ul
Sample Name : C-1500-134
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

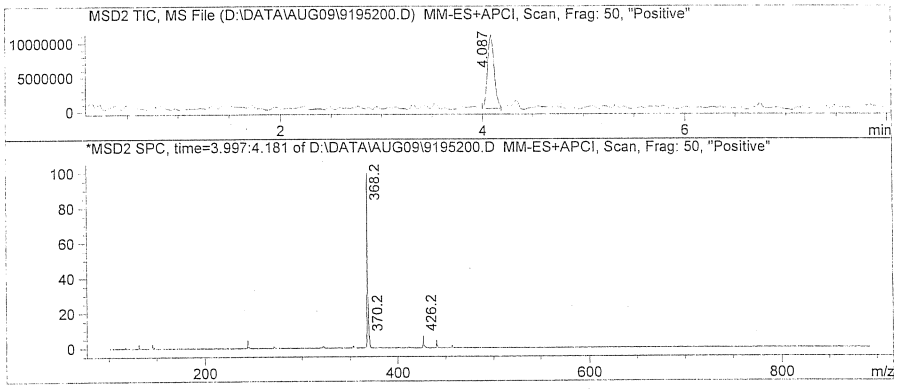
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{20}H_{21}N_3O_2S$
Exact Mass: 367.14
Mol. Wt.: 367.46



Peak No	RT min	Area	Area %
1	3.941	6.166e+000	0.664
2	4.046	8.981e+002	96.653
3	4.467	1.274e+001	1.371
4	4.719	1.219e+001	1.312



Analysed by : A

Instrument Code : SC/AD/10-014

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HPLC REPORT

Data file :C:\CHEM32\1\DATA\AUG09\9195200.D Vial No. -> P1-A-01

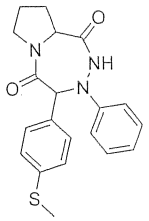
Injection Date :8/24/2009 9:07:48 AM Injection vol : 1 µl

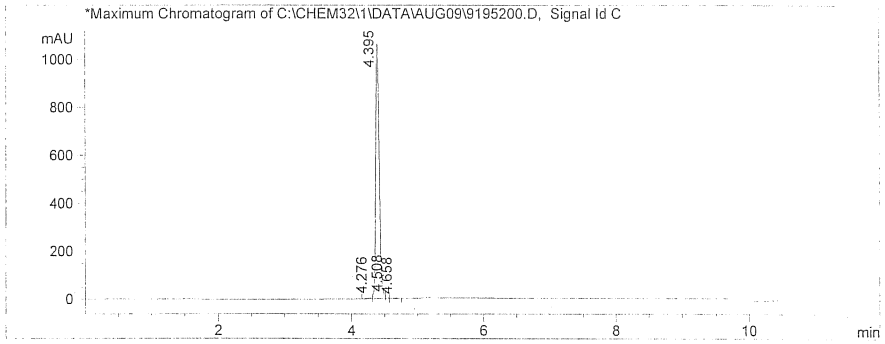
Sample Name :C-1500-134 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\S_AM73.M

Method info : A:10mM NH4OAC B:MeOH
Hypersil BDS C18(4.6X50)mm,5u
Flow:0.8mL/min

Time	%B
0	30
4	90
8	90
9	30
12	30





Peak No	RT min	Area	Area %
1	4.276	24.898	0.634
2	4.395	3877.893	98.748
3	4.508	15.911	0.405
4	4.658	8.371	0.213

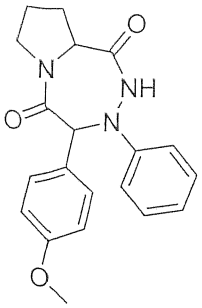
End of report

Analysed By :

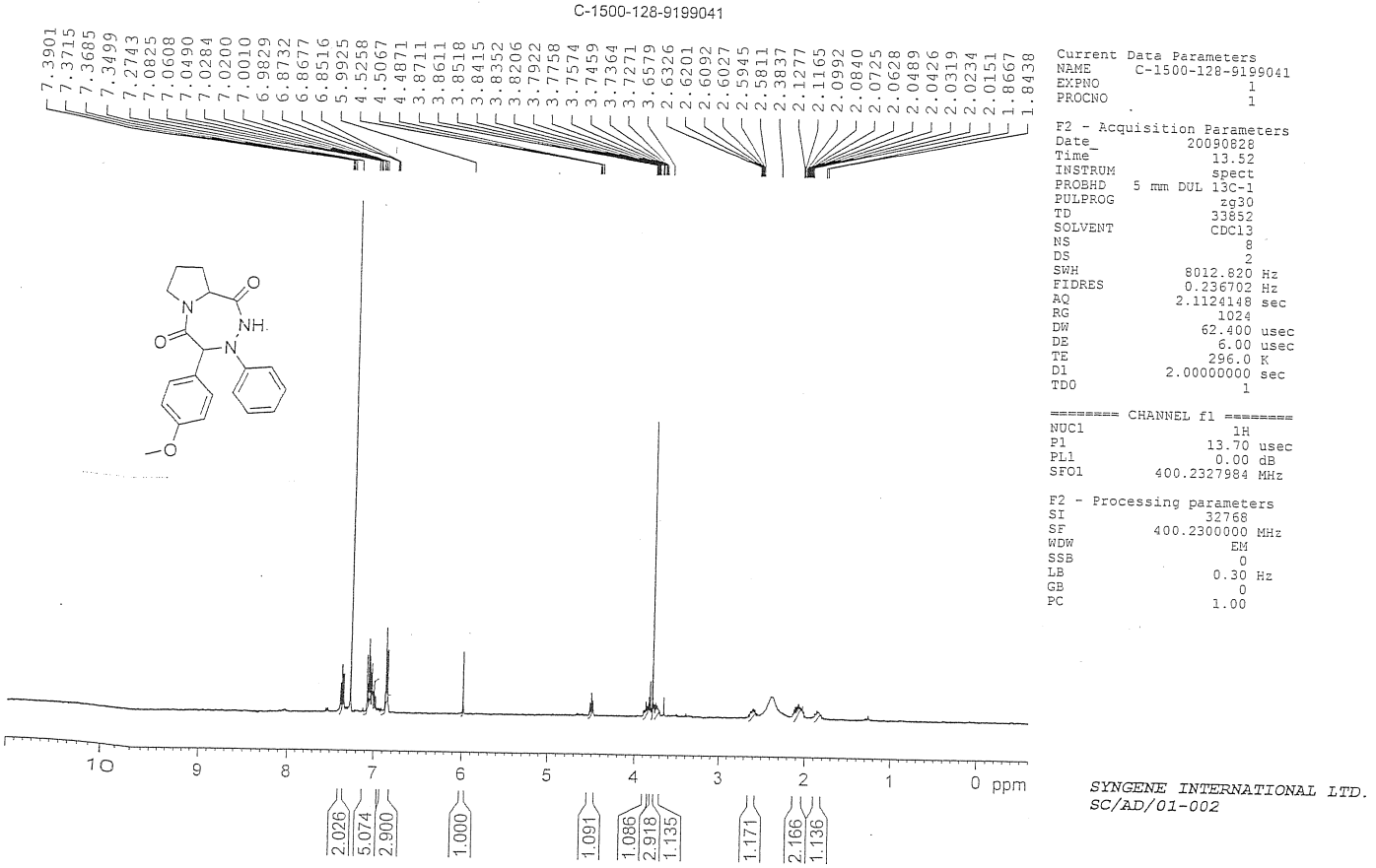
Instrument Code : SC/AD/04-062

Checked By :

XIV. Table 1, 2l: [5-(4-Methoxy-phenyl)-6-phenyl-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid; m.p (Met-Temp): 69°-70°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.84-1.87 (m, 1H), 2.01-2.12 (m, 2H), 2.58-2.63 (m, 1H), 3.73-3.75 (m, 1H), 3.80 (s, 3H), 3.81-3.87 (m, 1H), 4.49-4.53 (m, 1H), 5.99 (s, 1H), 6.84 (s, 1H), 6.85-6.87 (m, 3H), 6.98-7.08 (m, 5H), 7.35-7.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.16, 27.96, 48.57, 55.31, 57.67, 66.22, 112.71, 114.43, 121.03, 127.03, 129.74, 129.94, 146.08, 159.69, 168.31, 172.70; LCMS (UV): 352.2 ($M+H^+$). Anal. Calcd. for $C_{20}H_{21}N_3O_3$: C, 68.36; H, 6.02; N, 11.96. Found: C, 68.23; H, 6.09; N, 11.88.




```
Current Data Parameters
NAME      C-1500-128-9199041
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20090828
Time                   13.52
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    33852
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8012.820 Hz
FIDRES            0.236702 Hz
AQ                2.1124148 sec
RG                     1024
DW                  62.400 usec
DE                   6.00 usec
TE                   296.0 K
D1             2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.70 usec
PL1                    0.00 dB
SFO1          400.2327984 MHz

F2 - Processing parameters
SI                    32768
SF             400.2300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

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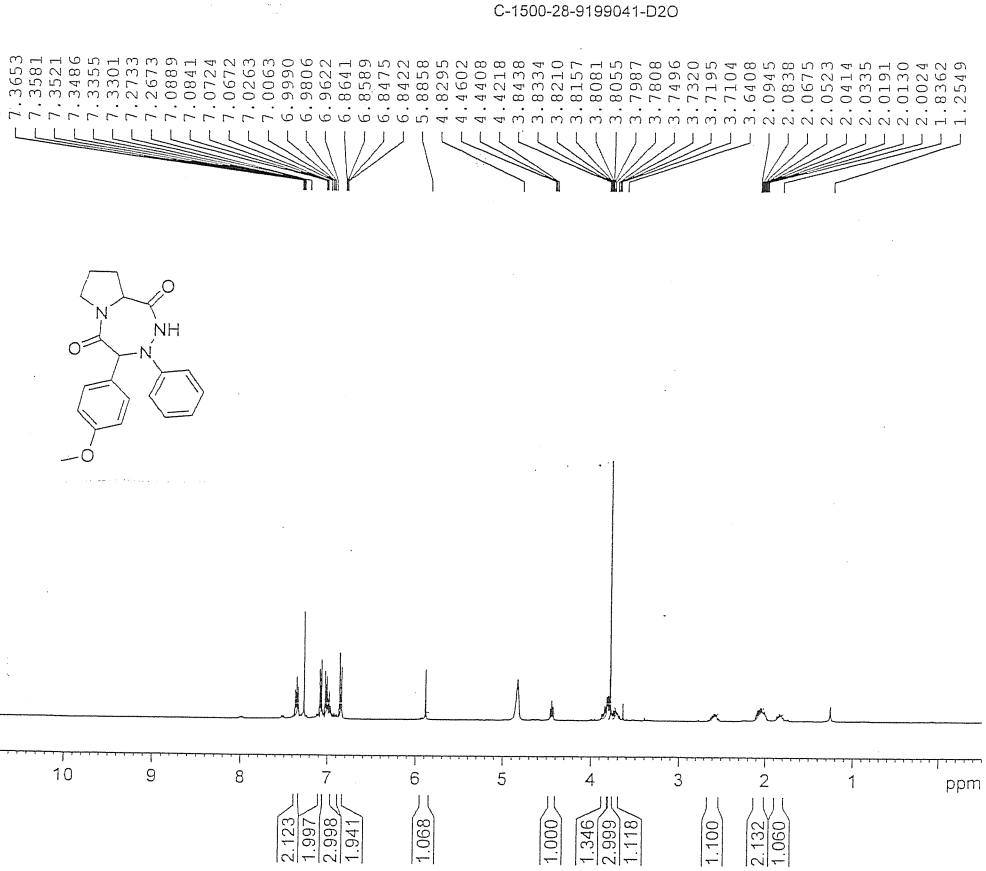
SC/AD/01-002

C-1500-28-9199041-D2O



Current Data Parameters

NAME	C-1500-28-9199041-D2O
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090829
Time	9.30
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	406
DW	60.800 usec
DE	6.50 usec
TE	293.0 K
D1	2.00000000 sec
TD0	1

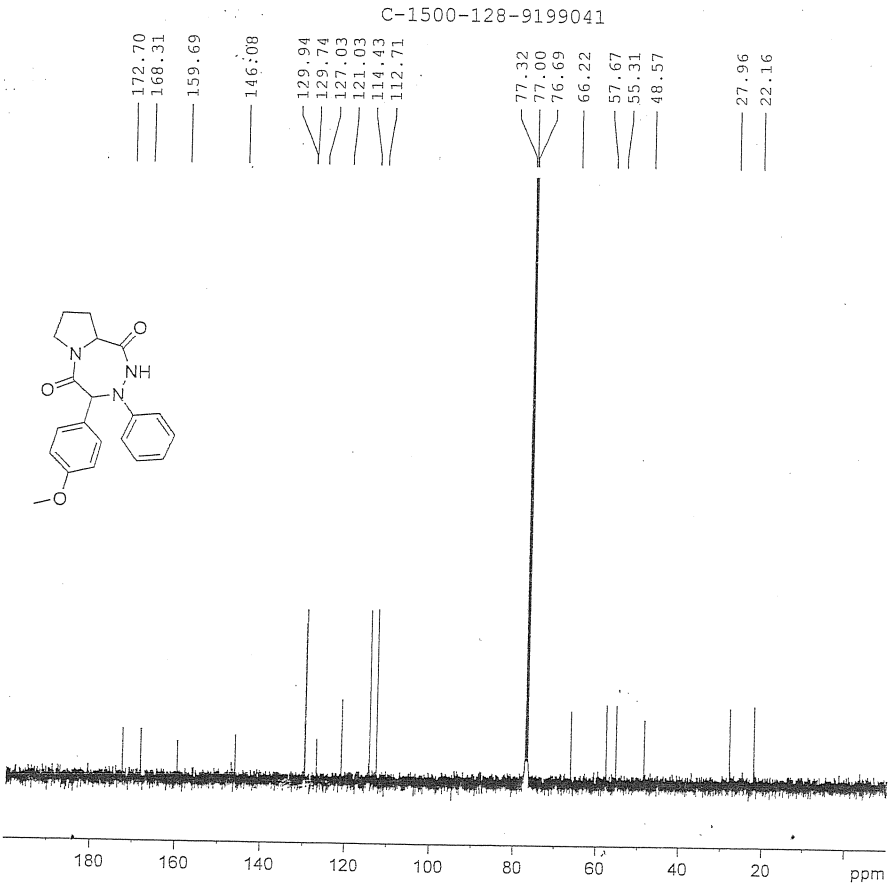
======== CHANNEL f1 ========

NUC1	1H
P1	13.50 usec
PL1	0.00 dB
SFO1	400.3124721 MHz

F2 - Processing parameters

SI	32768
SF	400.3100000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004



Current Data Parameters

NAME	C-1500-128-9199041
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090829
Time	11.02
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1608
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	293.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.25 usec
PL1	0.00 dB
SFO1	100.6680954 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.46 dB
PL13	16.00 dB
PL2	0.00 dB
SFO2	400.3116012 MHz

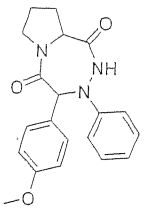
F2 - Processing parameters

SI	32768
SF	100.6580300 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

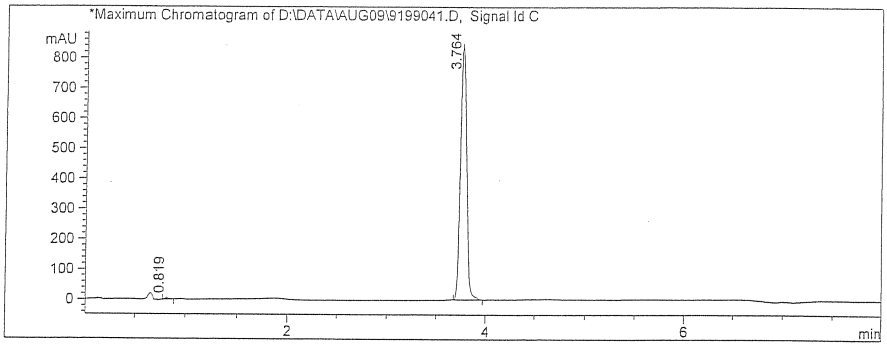
SYNGENE INTERNATIONAL LTD
SC/AD/01-004

Data file : D:\DATA\AUG09\9199041.D
Vial No. : P2-F-02
Injection Date : 8/28/2009 3:25:18 PM
Injection vol : 2ul
Sample Name : C-1500-128
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

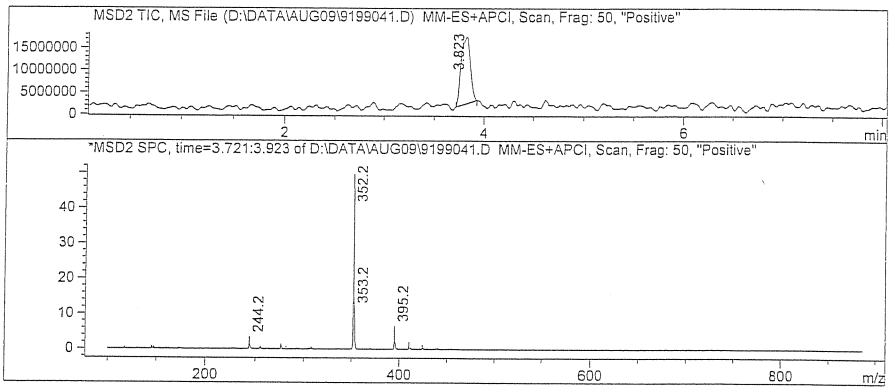
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{20}H_{21}N_3O_3$
Exact Mass: 351.1
Mol. Wt.: 351.40



Peak No	RT min	Area	Area %
1	0.819	1.666e+001	0.438
2	3.764	3.788e+003	99.562



Analysed by :

Instrument Code : SC/AD/10-014

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HPLC REPORT

Data file :C:\CHEM32\1\DATA\AUG09\9199041.D Vial No. : Vial 51

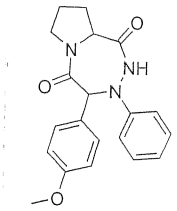
Injection Date :8/28/2009 2:39:23 PM Injection vol : 1 µl

Sample Name :C-1500-128 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\SY_T7030.M

Method info :A:0.1% TFA B:ACN
SYMMETRY C18(4.6X250)mm,5um,
Flow:0.8mL/min

Time	%B
0	30
15	100
20	100
23	30
28	30



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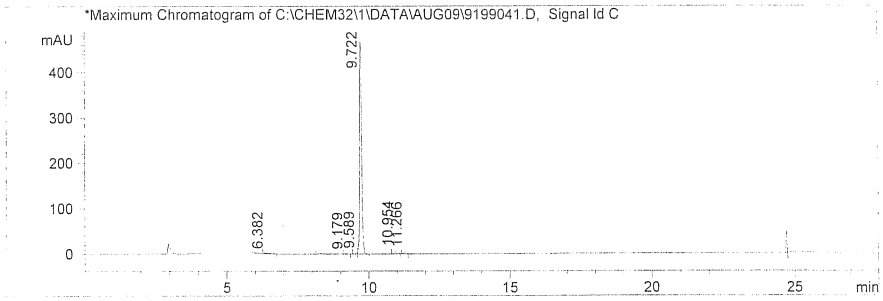


Peak No	RT min	Area	Area %
1	6.382	14.102	0.590
2	9.179	5.309	0.222
3	9.589	11.036	0.462
4	9.722	2272.342	95.047
5	10.954	52.101	2.179
6	11.266	35.863	1.500

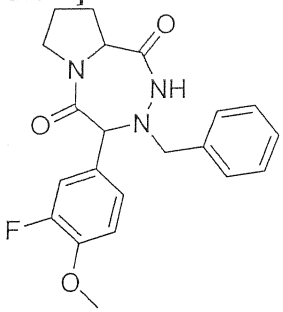
End of report

Analysed By :

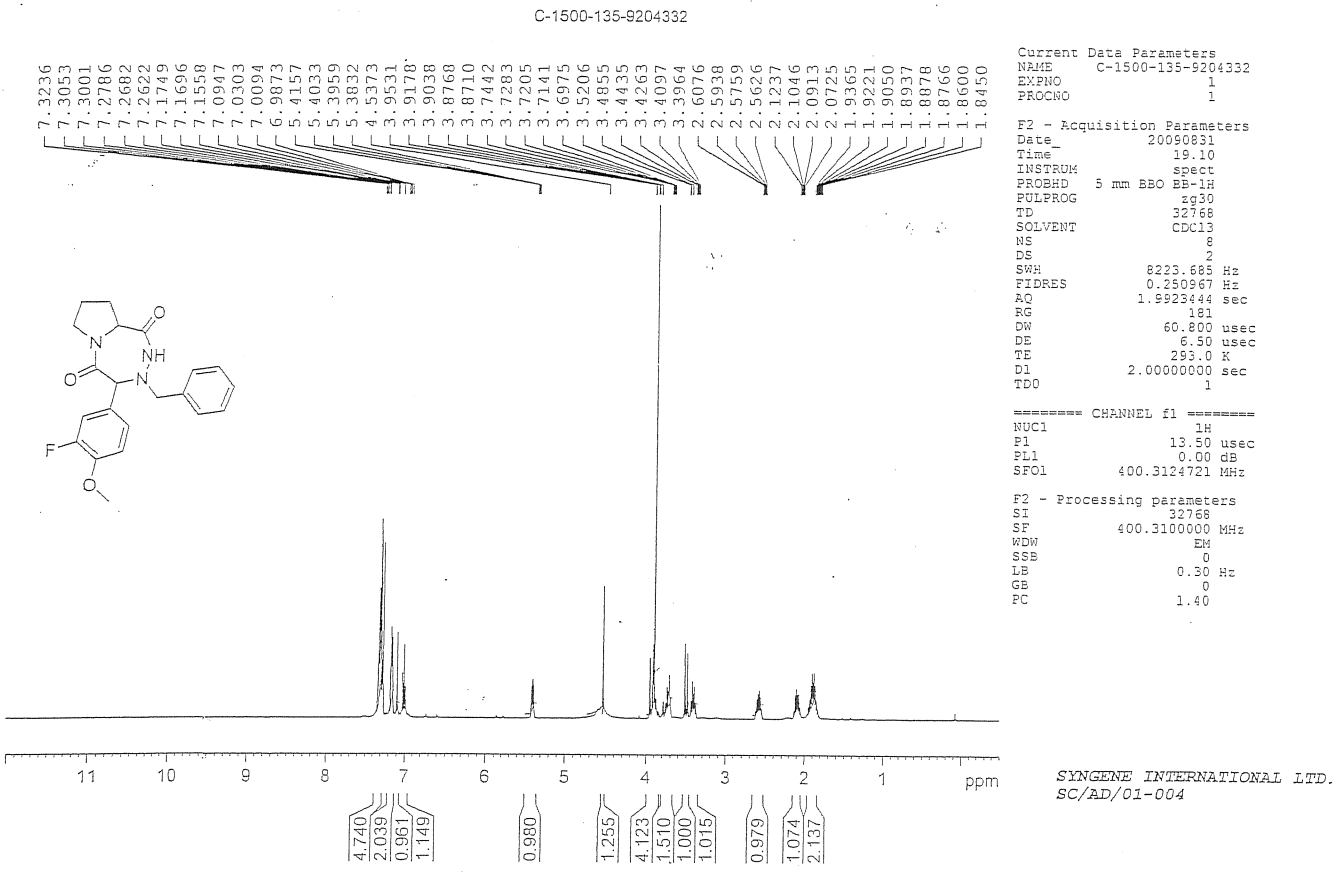
Instrument Code : SC/AD/04-064

Checked By :

XV. Table 1, 2m(A): [6-Benzyl-5-(3-fluoro-4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid; m.p (Met-Temp): 85°-87°C (uncorrected); $[\alpha]_D$ -18.52 (c=0.866, CH_2Cl_2, at 20°C); ^1H NMR ($CDCl_3$, 400 MHz): δ= 1.85-1.94 (m, 2H), 2.07-2.12 (m, 1H), 2.56-2.61 (m, 1H), 3.39-3.44 (m, 1H), 3.49-3.52 (m, 1H), 3.70-3.74 (m, 1H), 3.87 (s, 3H), 3.91-3.95 (m, 1H), 4.54 (s, 1H), 5.38-5.42 (m, 1H), 6.98-7.03 (m, 1H), 7.09 (s, 1H), 7.15-7.17 (d, J=7.64, 2H), 7.26-7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.44, 26.46, 48.26, 56.30, 56.92, 57.08, 75.17, 113.90, 124.83, 128.23, 128.77, 128.87, 130.18, 134.49, 148.19, 148.30, 151.42, 153.88, 168.86, 172.14; LCMS (UV): 384.2 ($M+H^+$). Anal. Calcd. for $C_{21}H_{22}FN_3O_3$: C, 65.78; H, 5.78; N, 10.96. Found: C, 65.69; H, 5.75; N, 10.83.



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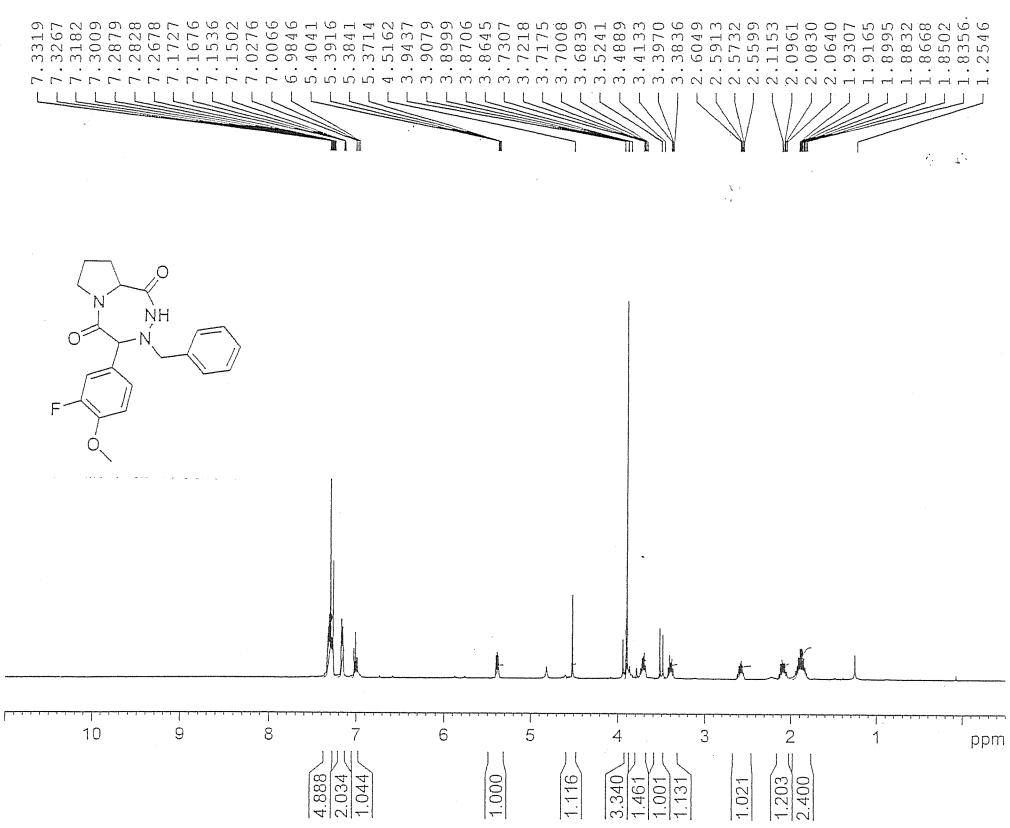
SC/AD/01-004

C-1500-135-9204332D2O



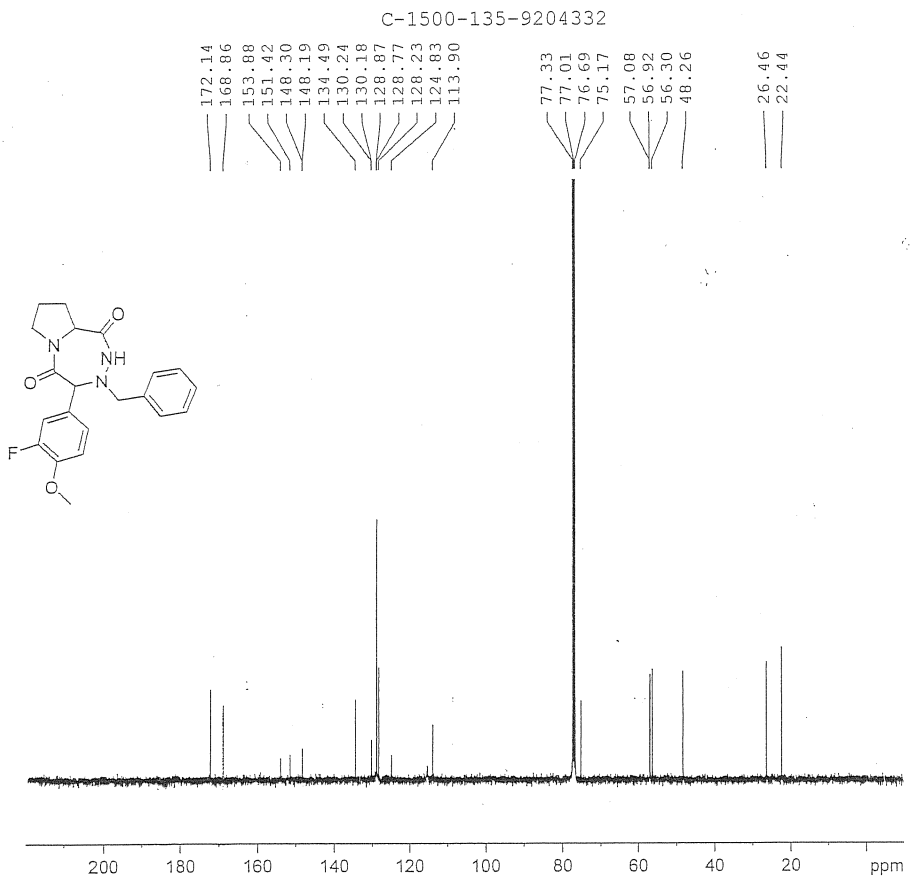

```
Current Data Parameters
NAME      C-1500-135-9204332D
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20090901
Time               10.49
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ             1.9923444 sec
RG                   161
DW                60.800 usec
DE                 6.50 usec
TE                293.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.50 usec
PL1                0.00 dB
SFO1         400.3124721 MHz

F2 - Processing parameters
SI                 32768
SF          400.3100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004



Current Data Parameters

NAME	C-1500-135-9204332
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090831
Time	20.50
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	2048
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	293.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.25 usec
PL1	0.00 dB
SFO1	100.6680954 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.46 dB
PL13	16.00 dB
PL2	0.00 dB
SFO2	400.3116012 MHz

F2 - Processing parameters

SI	32768
SF	100.6580300 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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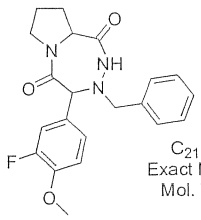
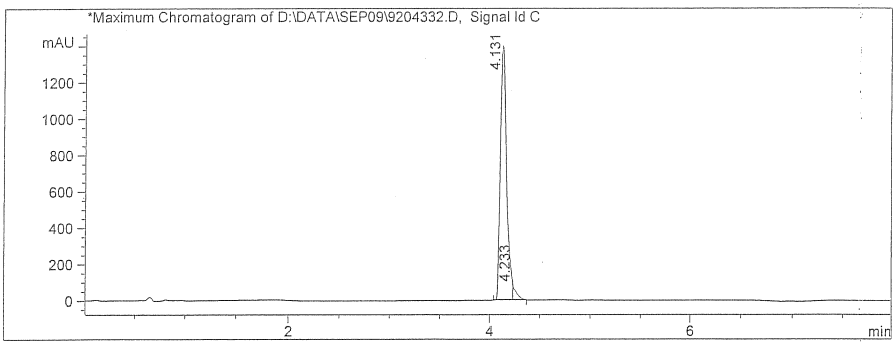
SC/AD/01-004

Data file : D:\DATA\SEP09\9204332.D
Vial No. : P1-A-08
Injection Date : 9/1/2009 11:30:55 AM
Injection vol : 2ul
Sample Name : C-1500-135
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

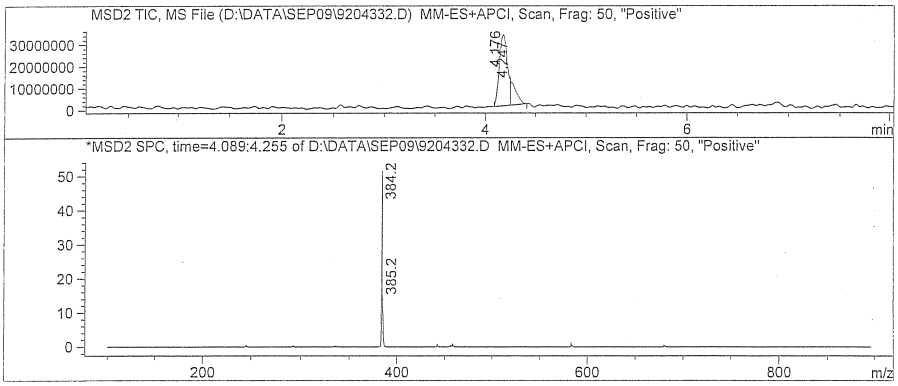
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{21}H_{22}FN_3O_3$
Exact Mass: 383.16
Mol. Wt.: 383.42



Peak No	RT min	Area	Area %
1	4.131	6.012e+003	97.201
2	4.233	1.731e+002	2.799



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HPLC REPORT

Data file :C:\CHEM32\1\DATA\SEP09\9204332.D Vial No. -> P1-E-01

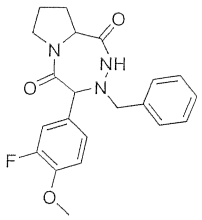
Injection Date :9/1/2009 9:06:49 AM Injection vol : 1 µl

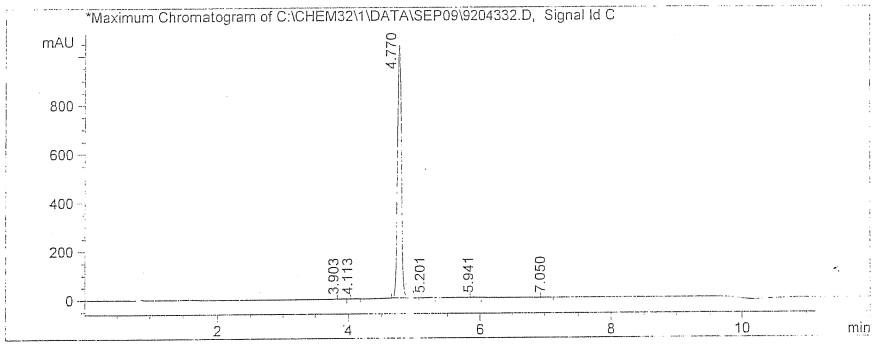
Sample Name :C-1500-135 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\S_AM73.M

Method info : A:10mM NH4OAC B:MeOH
Hypersil BDS C18(4.6X50)mm,5u
Flow:0.8mL/min

Time	%B
0	30
4	90
8	90
9	30
12	30





Peak No	RT min	Area	Area %
1	3.903	3.294	0.076
2	4.113	3.110	0.072
3	4.770	4319.696	99.512
4	5.201	7.744	0.178
5	5.941	4.106	0.095
6	7.050	2.933	0.068

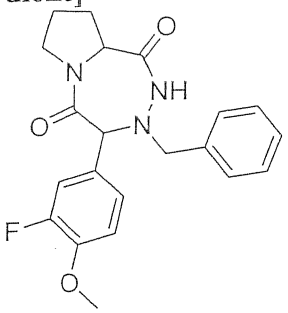
End of report

Analysed By :

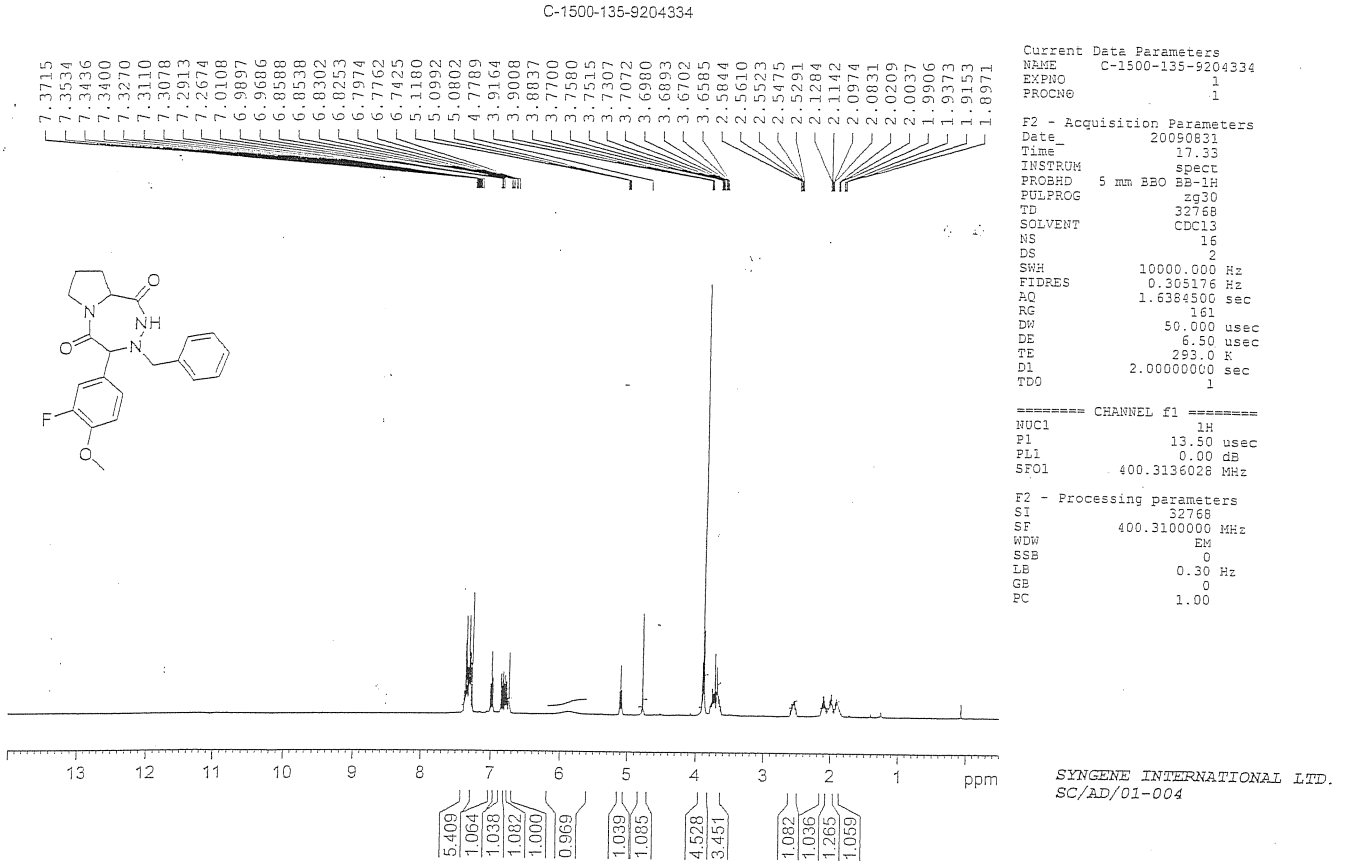
Checked By :

Instrument Code : SC/AD/04-062

XVI. Table 1, 2m(B) [6-Benzyl-5-(3-fluoro-4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-4,8-dione]



White solid; m.p (Met-Temp): 80°-82°C (uncorrected); $[\alpha]_D$ -49.25 (c=0.944, CH_2Cl_2, at 20°C); ^1H NMR ($CDCl_3$, 400 MHz): δ= 1.89-1.93 (m, 1H), 1.99-2.02 (m, 1H), 2.08-2.13 (m, 1H), 2.53-2.58 (m, 1H), 3.66-3.77 (m, 3H), 3.88 (s, 3H), 3.90-3.91 (m, 1H), 4.78 (s, 1H), 5.08-5.12 (m, 1H), 6.74 (s, 1H), 6.78-6.86 (m, 2H), 6.97-7.01 (m, 1H), 7.27-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.38, 27.37, 48.59, 56.30, 58.14, 58.48, 70.10, 113.90, 117.54, 124.89, 125.88, 128.32, 128.91, 128.95, 135.07, 147.90, 148.01, 150.91, 153.38, 168.07, 171.03; LCMS (UV): 384.2 ($M+H^+$). Anal. Calcd. for $C_{21}H_{22}FN_3O_3$: C, 65.78; H, 5.78; N, 10.96. Found: C, 65.74; H, 5.81; N, 10.99.




```
Current Data Parameters
NAME      C-1500-135-9204334
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_           20090831
Time_              17.33
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ             1.6384500 sec
RG                   161
DW                50.000 usec
DE                 6.50 usec
TE                293.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1                0.00 dB
SFO1        400.3136028 MHz

F2 - Processing parameters
SI                 32768
SF          400.3100000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

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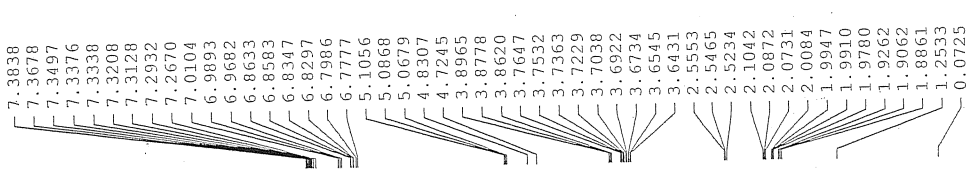
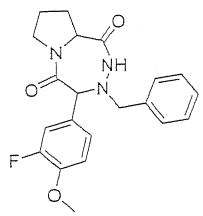
SC/AD/01-004

C-1500-135-9204334D2O





Current Data Parameters

NAME	C-1500-135-9204334D
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090901
Time	10.47
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	161
DW	60.800 usec
DE	6.50 usec
TE	293.0 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	13.50 usec
PL1	0.00 dB
SFO1	400.3124721 MHz

F2 - Processing parameters

SI	32768
SF	400.3100000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

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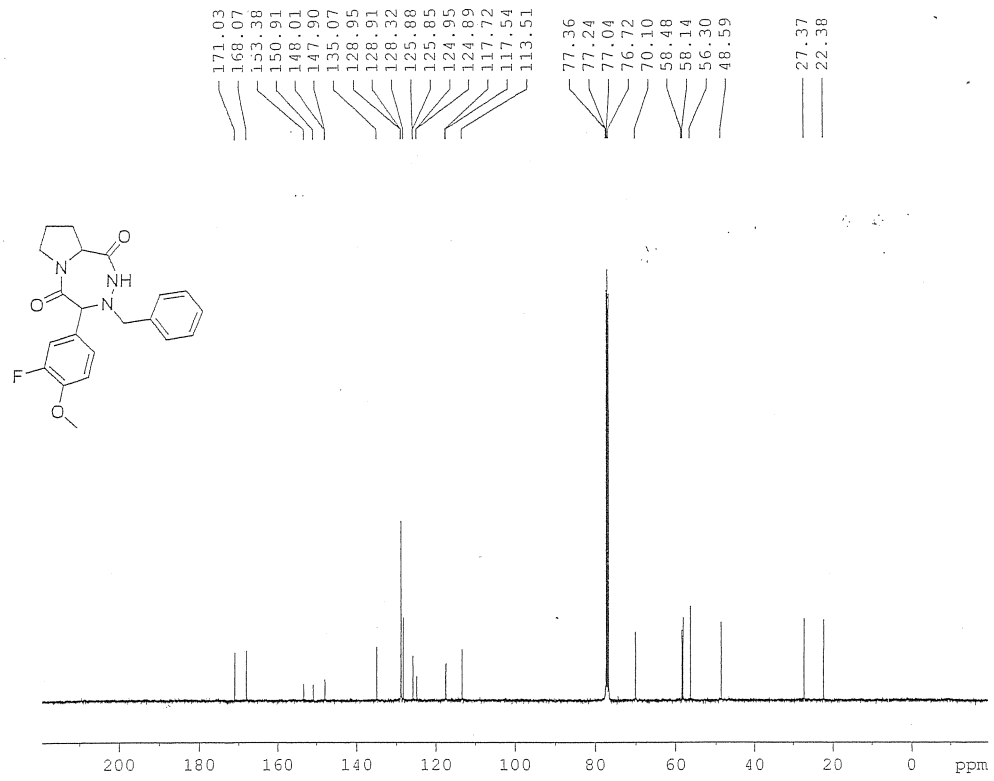
SC/AD/01-004

C-1500-135-9204334



Current Data Parameters

NAME	C-1500-135-9204334
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090901
Time	5.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	500
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	114
DW	20.800 usec
DE	6.00 usec
TE	295.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	7.13 usec
PL1	-3.00 dB
SFO1	100.6278593 MHz

======== CHANNEL f2 ========

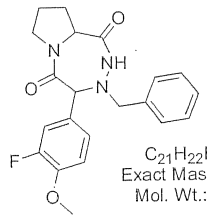
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.14 dB
PL13	15.00 dB
PL2	0.00 dB
SFO2	400.1516006 MHz

F2 - Processing parameters

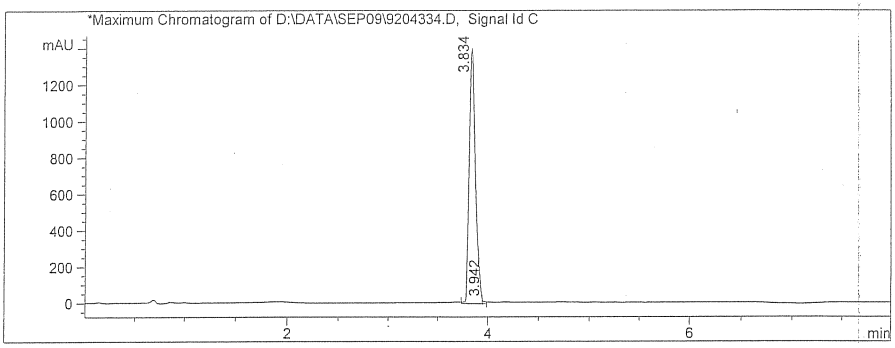
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Data file : D:\DATA\SEP09\9204334.D
Vial No. : P1-A-09
Injection Date : 9/1/2009 11:40:32 AM
Injection vol : 2ul
Sample Name : C-1500-135
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

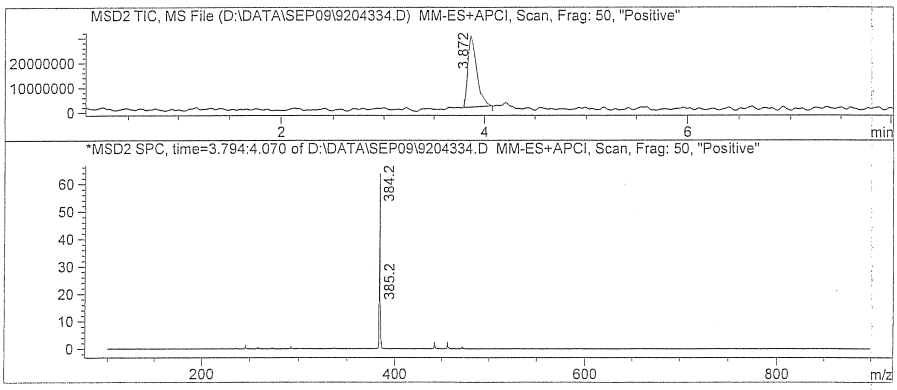
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{21}H_{22}FN_3O_3$
Exact Mass: 383.16
Mol. Wt.: 383.42



Peak No	RT min	Area	Area %
1	3.834	6.133e+003	99.464
2	3.942	3.307e+001	0.536



Analysed by :

Instrument Code : SC/AD/10-014

Syngene International Ltd
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HPLC REPORT

Data file :C:\CHEM32\1\DATA\AUG09\9204334.D Vial No. -> P1-C-04

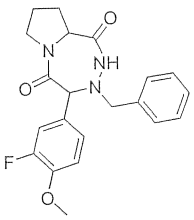
Injection Date :8/31/2009 5:34:06 PM Injection vol : 1 µl

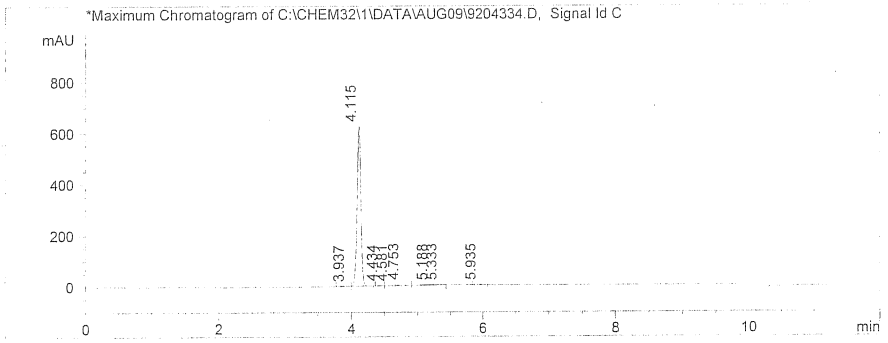
Sample Name :C-1500-135 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\S_AM73.M

Method info : A:10mM NH4OAC B:MeOH
Hypersil BDS C18(4.6X50)mm,5u
Flow:0.8mL/min

Time	%B
0	30
4	90
8	90
9	30
12	30





Peak No	RT min	Area	Area %
1	3.937	5.936	0.230
2	4.115	2482.573	96.151
3	4.434	4.832	0.187
4	4.581	4.466	0.173
5	4.753	33.310	1.290
6	5.188	25.938	1.005
7	5.333	18.617	0.721
8	5.935	6.273	0.243

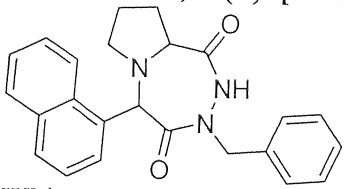
End of report

Analysed By :

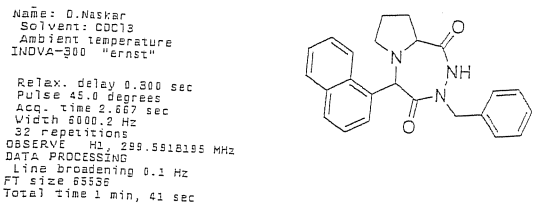
Instrument Code : SC/AD/04-062

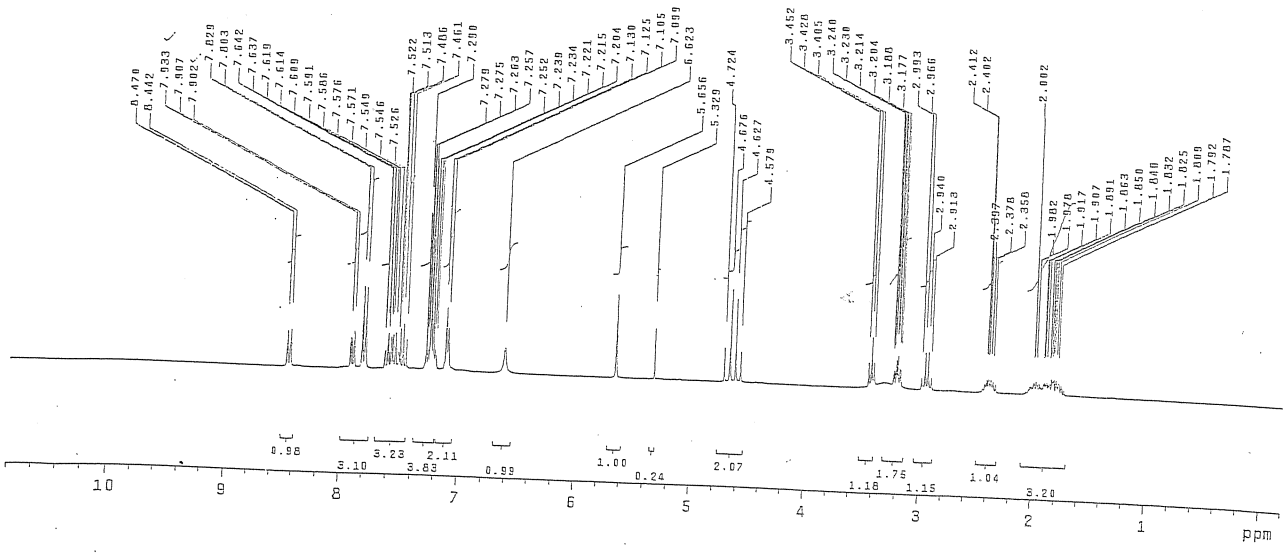
Checked By :

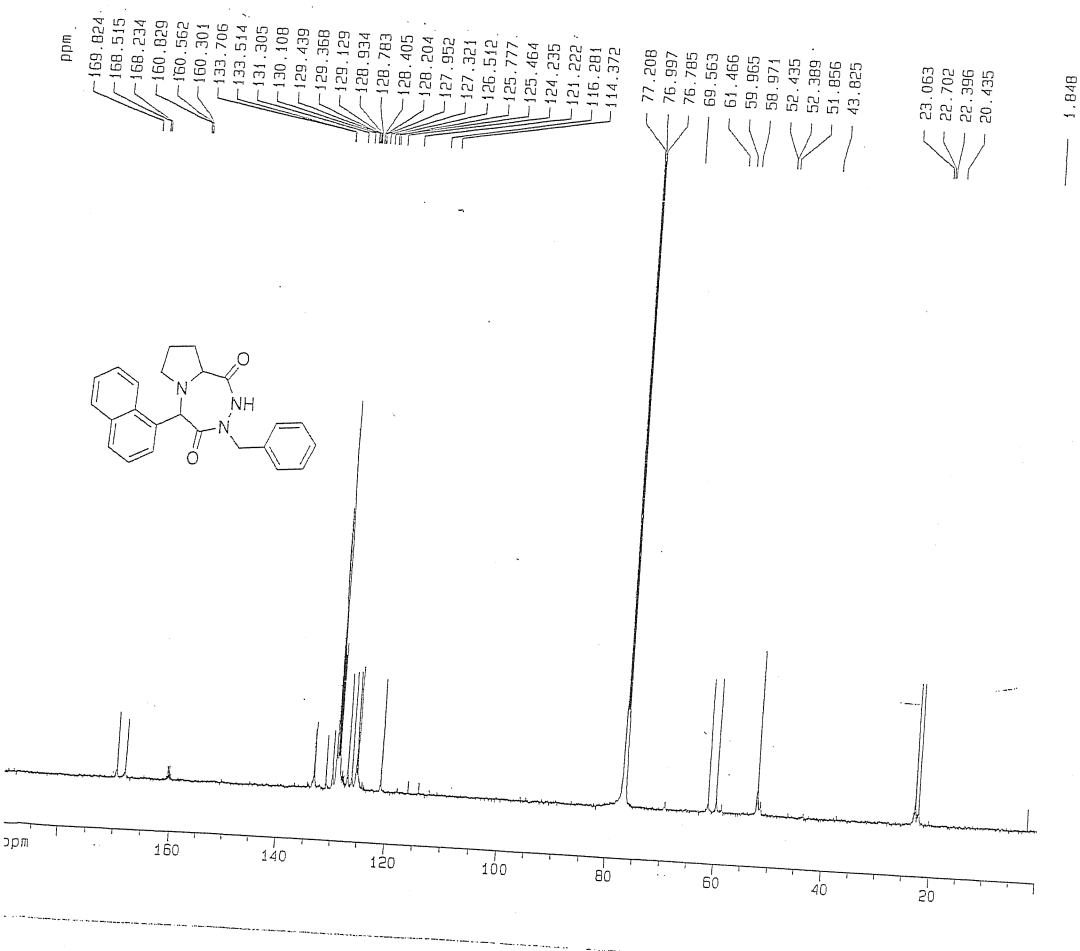
XVII. Table 2, 4a(A): [6-Benzyl-4-naphthalen-1-yl-hexahydro-3a,6,7-triaza-azulene-5,8-dione]



White solid; m.p (Met-Temp): 210°-211°C (uncorrected); $[\alpha]_D$ +84.607 (c=0.890, $CHCl_3$, at 20°C); ^1H NMR ($CDCl_3$, 300MHz): δ= 1.78-2.14 (m, 3H), 2.36-2.41 (m, 1H), 2.91-2.99 (m, 1H), 3.18-3.24 (m, 1H), 3.41-3.45 (m, 1H), 4.58-4.72 (m, 2H), 5.65 (s, 1H), 6.62 (br s, 1H), 7.10-7.13 (m, 2H), 7.20-7.29 (m, 3H), 7.46-7.64 (m, 3H), 7.82 (d, J = 7.82 Hz, 2H), 7.91 (d, J=9.3 Hz, 1H), 8.46 (d, J= 8.46 Hz, 1H); ^{13}C NMR($CDCl_3$, 75MHz): 22.40, 23.06, 52.39, 52.44, 59.97, 61.47, 121.22, 125.46, 125.78, 126.51, 127.32, 128.78, 128.93, 129.13, 129.37, 129.44, 130.11, 131.31, 133.51; 133.71, 168.23, 169.82; LCMS (ELSD): 386 ($M+H^+$); HRMS: 386.185503 [Calculated for $C_{24}H_{24}N_3O_2$ 386.186852 $(M+H)^+$].







Current Data Parameters

EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	40890
DS	4
SWH	37593.984 Hz
FIDRES	1.147277 Hz
AQ	0.4358644 sec
RG	1024
DW	13.300 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	150.9193478 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	-6.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	600.1324005 MHz

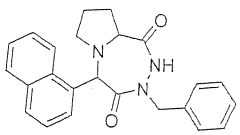
F2 - Processing parameters

SI	32768
SF	150.9028157 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.40

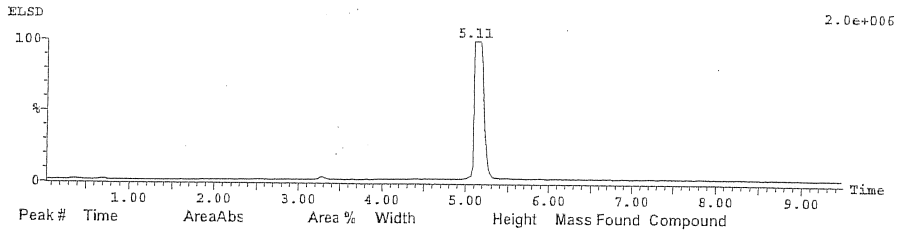
1D NMR plot parameters

CX	20.00 cm
F1P	190.000 ppm
F1	28671.54 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	9.50000 ppm/cm
HZCM	1433. Hz/cm

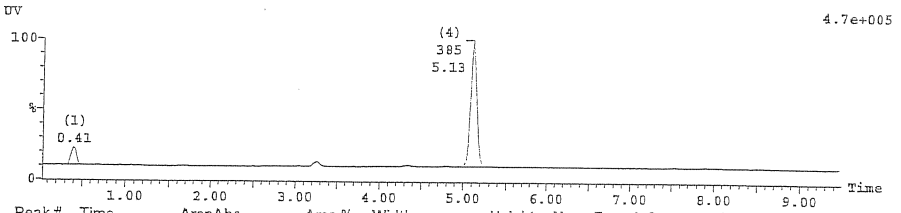
Sample Report (continued):



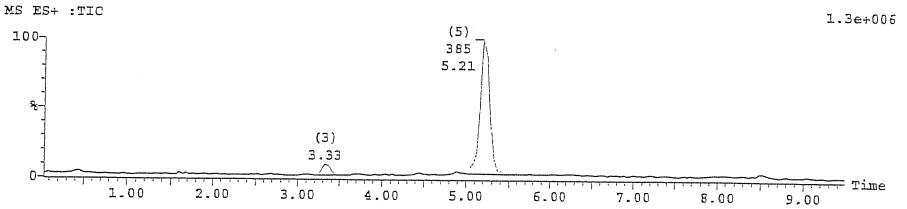
$C_{24}H_{23}N_3O_2$
Exact Mass: 385.18
Mol. Wt.: 385.46

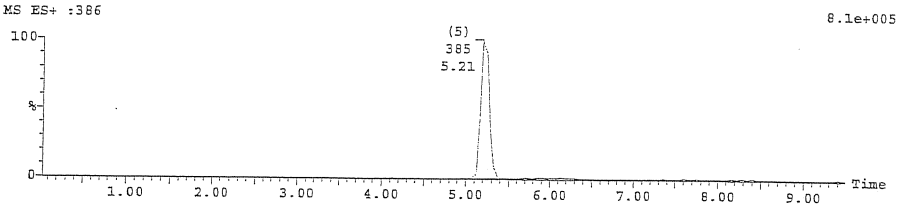


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound

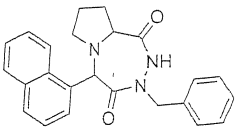


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
1	0.41	4.373e+003	10.70	0.200	5.881e+004		
4	5.13	3.648e+004	89.30	0.600	4.230e+005	385.00	Found



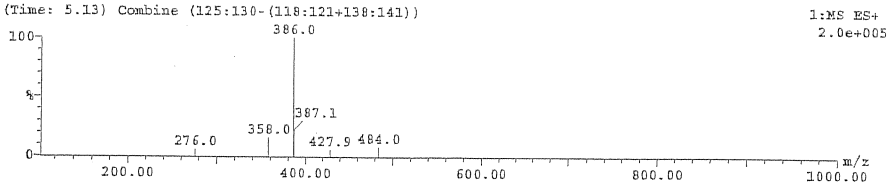


Sample Report (continued):

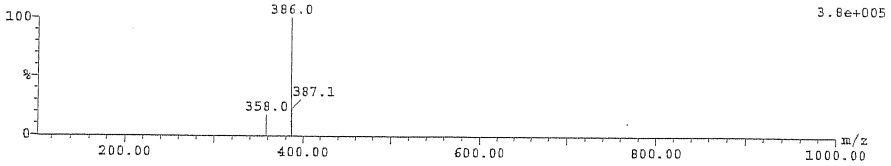


$C_{24}H_{23}N_3O_2$
Exact Mass: 385.18
Mol. Wt.: 385.46

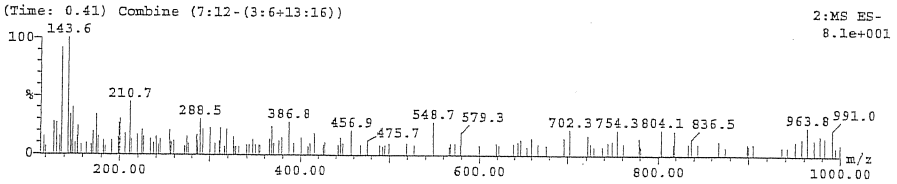
Peak ID Time Mass Found
4 5.13 385.00
(Time: 5.13) Combine (125:130-(118:121+138:141))



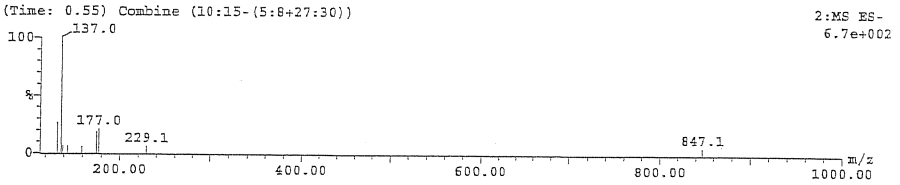
Peak ID Time Mass Found
5 5.21 385.00
(Time: 5.21) Combine (128:133-(118:121+140:143))



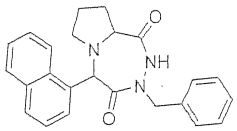
Peak ID Time Mass Found
1 0.41
(Time: 0.41) Combine (7:12-(3:6+13:16))



Peak ID Time Mass Found
2 0.55
(Time: 0.55) Combine (10:15-(5:8+27:30))

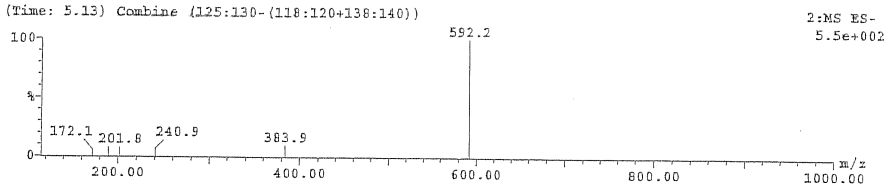


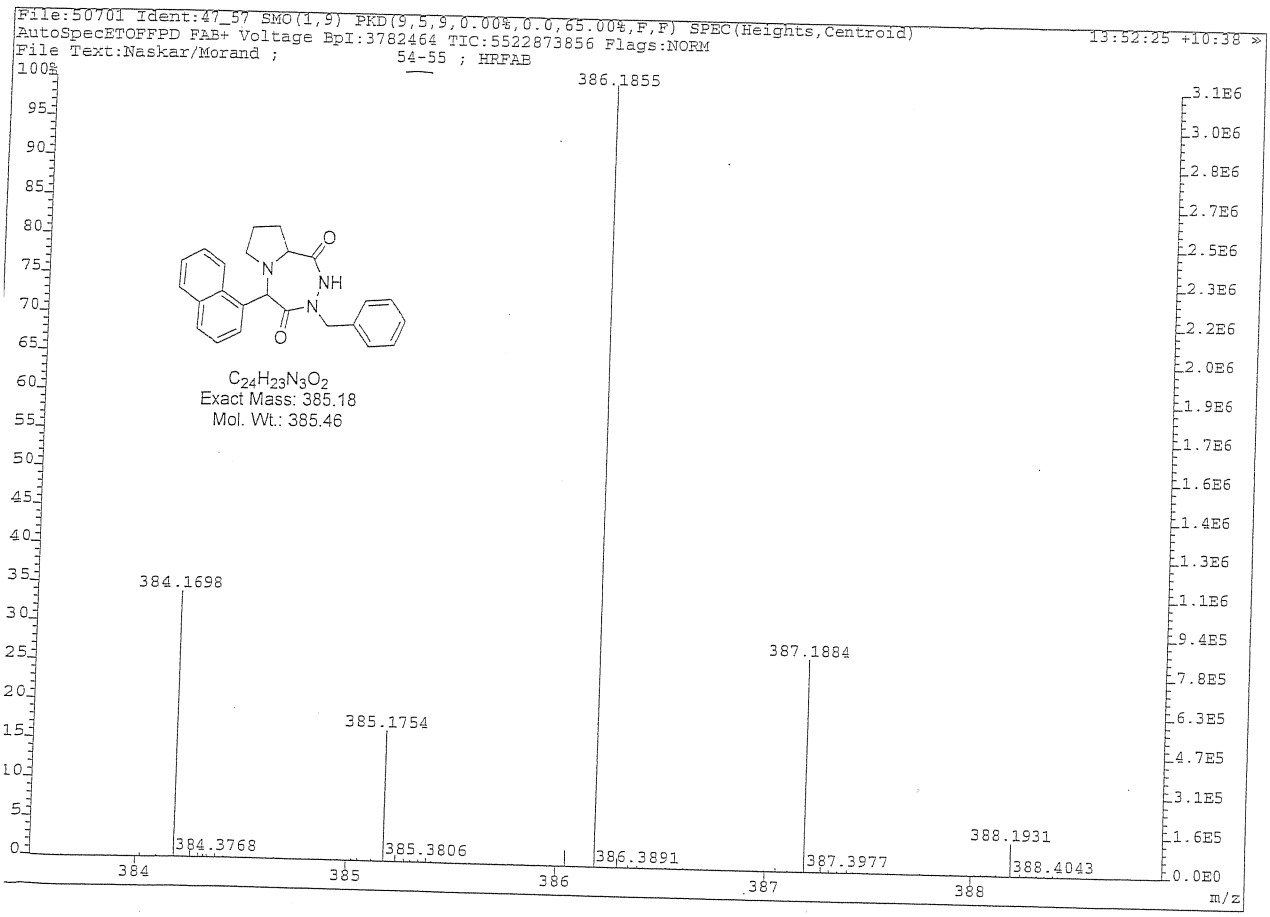
Sample Report (continued):



$C_{24}H_{23}N_3O_2$
Exact Mass: 385.18
Mol. Wt.: 385.46

Peak ID Time Mass Found
4 5.13

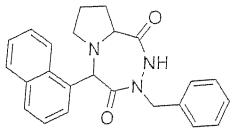



File:50701 Ident:47_57 SMO(1,9) PKD(9,5,9,0.00%,0.0,65.00%,F,F) SPEC(Heights,Centroid)
AutoSpecETOFFPD FAB+ Voltage BpI:3782464 TIC:5522873856 Flags:NORM
File Text:Naskar/Morand ; 54-55 ; HRFAB
13:52:25 +10:38
C24H23N3O2
Exact Mass: 385.18
Mol. Wt.: 385.46
386.1855
384.1698
387.1884
385.1754
384.3768
385.3806
386.3891
387.3977
388.1931
388.4043
m/z

Elemental Composition

File:50701 Ident:47_57 SMO(1,9) PKD(9,5,9,0.00%,0.0,65.00%,F,F)
AutoSpecETOFFPD_FAB+ Voltage_EpI:3782464 TIC:5522873856 Flags:NORM
File Text:Naskar/Morand ; 54-55 ; HRFAB
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

383.509	1.0					-0.5	0	0	0	3	2	
388.933	100.0		10.0			20.0	30	1	40	3	2	
Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O
387.188448	26.9			4.5	1.8	387.190207	14.5	23	1	24	3	2
386.185503	100.0			3.5	1.3	386.186852	14.5	24		24	3	2
				-8.1	-3.1	386.182382	15.0	23	1	23	3	2
385.175445	16.6			-2.3	-0.9	385.174557	15.5	23	1	22	3	2
				9.3	3.6	385.179027	15.0	24		23	3	2
384.169810	33.9			3.6	1.4	384.171202	15.5	24		22	3	2
				-8.0	-3.1	384.166732	16.0	23	1	21	3	2

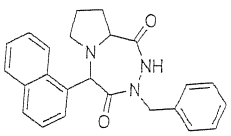


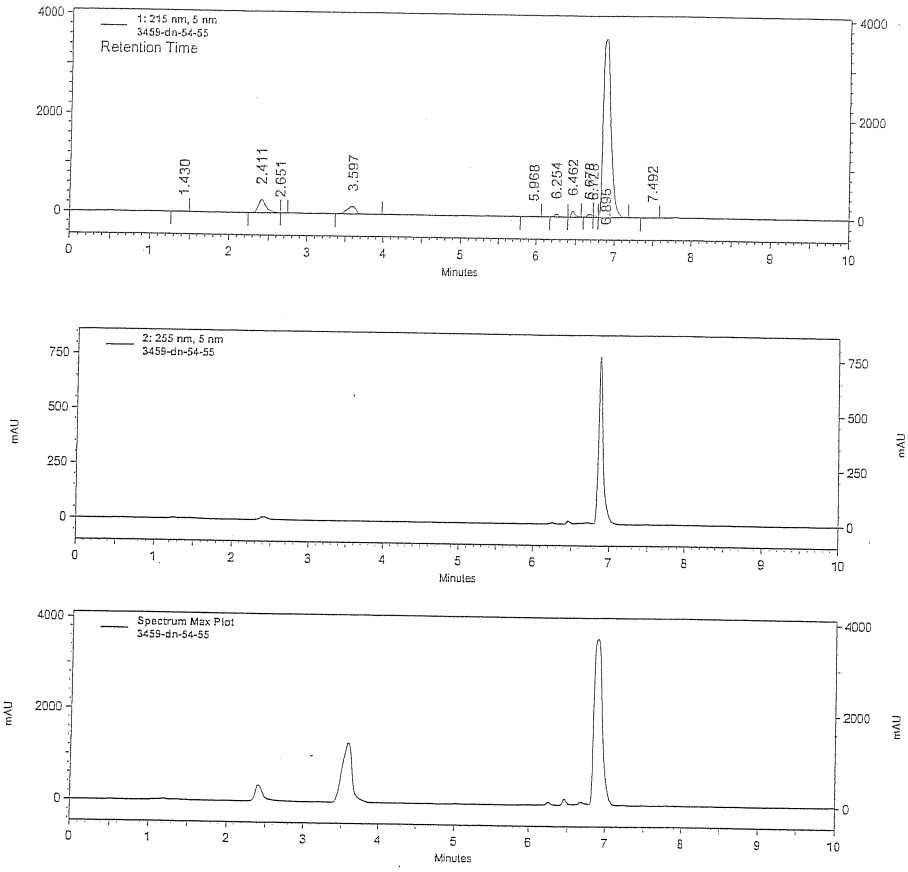
$C_{24}H_{23}N_3O_2$
Exact Mass: 385.18
Mol. Wt.: 385.46

Area Percent Report

Data File: C:\ChromQuest\3459-dn-54-55.dat

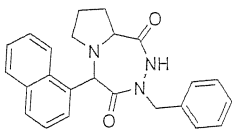
Analyst: System
Sample ID: 54-55 Vial: A08 Injection Volume: 10





Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\30acnsjs.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

		1.430	100465	0.34
SS		2.411	1906194	6.39
SS		2.651	65647	0.22
SS		3.597	1362145	4.57
VV		5.968	73635	0.25
VV		6.254	279718	0.94
SV		6.462	522876	1.75
VV		6.678	310279	1.04
SS		6.728	124864	0.42
SV		6.895	25009432	83.89
VS		7.492	57123	0.19
VS				
	Totals		29812378	100.00



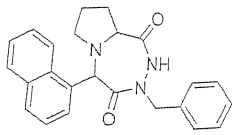
Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\30acnsjs.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

Area Percent Report

Data File: C:\ChromQuest\ 54-55.dat

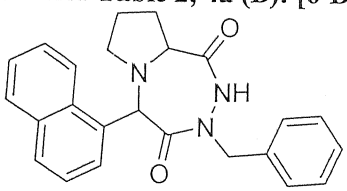
2: 255 nm, 5 nm
Results (Original)

Name	Retention Time	Area	Area Percent	Integration Codes
	6.462	60564	1.79	VV
	6.726	53290	1.58	SV
	6.882	3263006	96.63	VV
Totals		3376860	100.00	

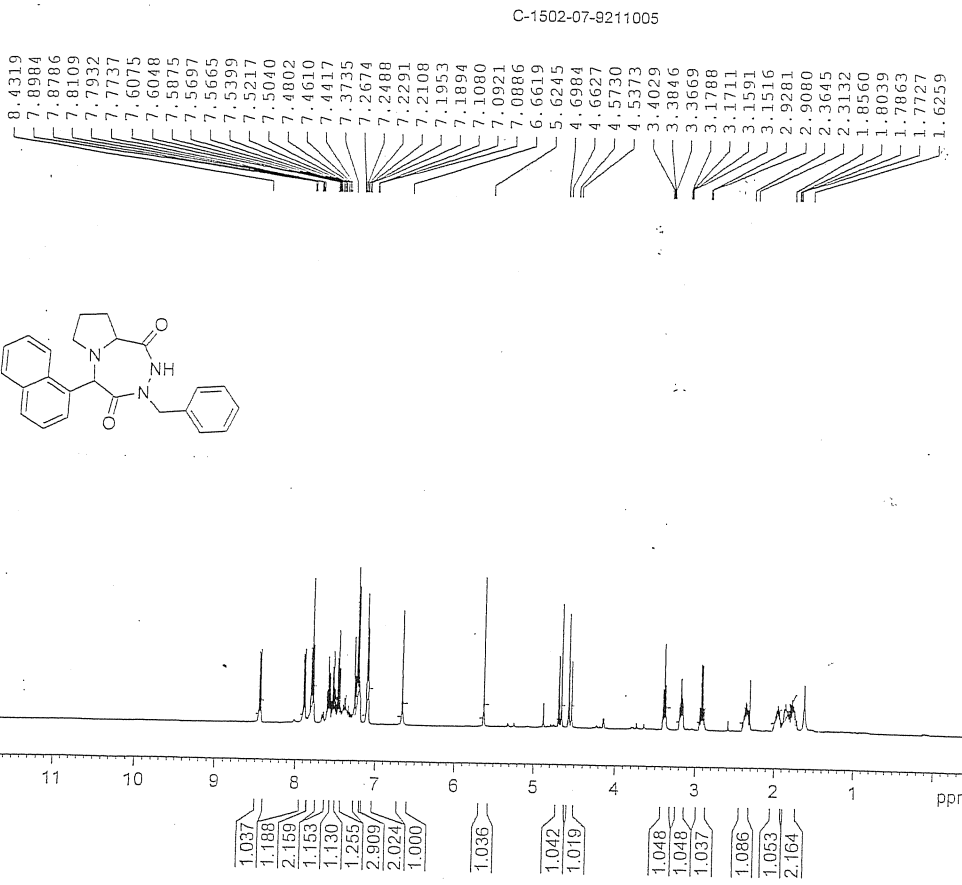


Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\30acnsjs.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

XVIII. Table 2, 4a (B): [6-Benzyl-4-naphthalen-1-yl-hexahydro-3a,6,7-triaza-azulene-5,8-dione]



White solid; m.p (Met-Temp): 215°-216°C (uncorrected) $[\alpha]_D$ +93.171 (c=0.946, $CHCl_3$, at 20°C); ^1H NMR ($CDCl_3$, 400MHz): δ= 1.77-2.14 (m, 3H), 2.31-2.36 (m, 1H), 2.91-2.93 (m, 1H), 3.16-3.18 (m, 1H), 3.37-3.40 (m, 1H), 4.54-4.57 (m, 1H), 4.66-4.70 (m, 1H), 5.62 (s, 1H), 6.66 (br s, 1H), 7.10-7.11 (m, 2H), 7.19-7.27 (m, 3H), 7.44-7.61 (m, 3H), 7.77-7.83 (m, 2H), 7.88-7.90 (d, J=7.92 Hz, 1H), 8.43-8.45 (d, J=8.48 Hz, 1H); ^{13}C NMR($CDCl_3$, 100MHz): 22.94, 24.42, 52.97, 54.03, 58.55, 68.23, 123.55, 124.51, 125.44, 125.66, 126.54, 128.41, 128.43, 128.48, 128.77, 128.87, 129.18, 130.85, 132.61, 133.75; 133.80, 169.84, 171.62; LCMS (ELSD): 386.2 ($M+H^+$); Anal. Calcd. for $C_{24}H_{23}N_3O_2$: C, 74.78; H, 6.01; N, 10.90. Found: C, 74.69; H, 6.05; N, 10.92

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Current Data Parameters
NAME      C-1502-07-9211005
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
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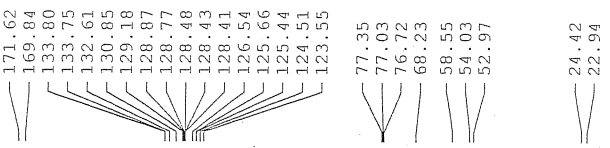
Time                18.43
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH              8223.685 Hz
FIDRES           0.250967 Hz
AQ               1.9923444 sec
RG                   228
DW                60.800 usec
DE                 6.50 usec
TE                293.0 K
D1             2.00000000 sec
TD0                    1

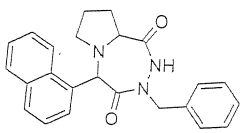
======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1                0.00 dB
SFO1         400.3124721 MHz

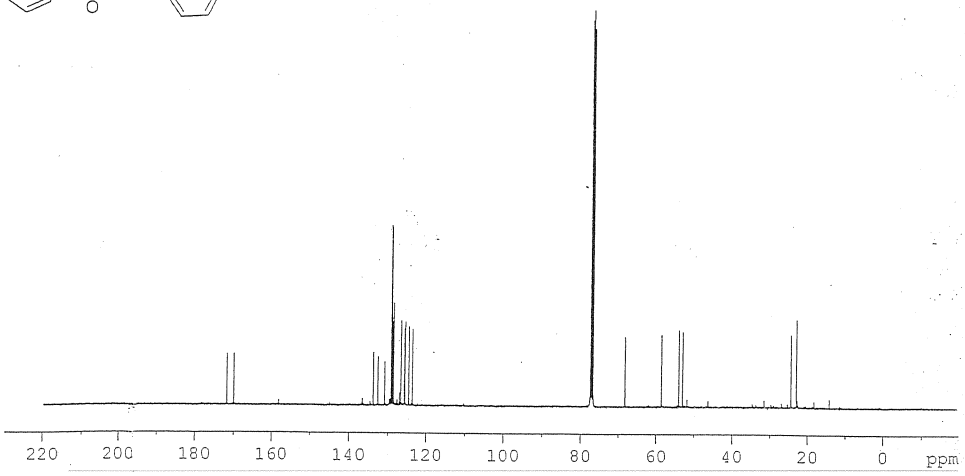
F2 - Processing parameters
SI                 32768
SF           400.3100000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.40
```

SYNGENE INTERNATIONAL LTD.
SC/AD/01-004

C-1502-07-9211005







Current Data Parameters

NAME	C-1502-07-9211005
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

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PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	5000
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	114
DW	20.800 usec
DE	6.00 usec
TE	295.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	7.13 usec
PL1	-3.00 dB
SFO1	100.6278593 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.14 dB
PL13	15.00 dB
PL2	0.00 dB
SFO2	400.1516006 MHz

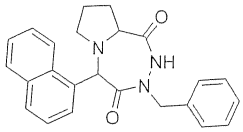
F2 - Processing parameters

SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

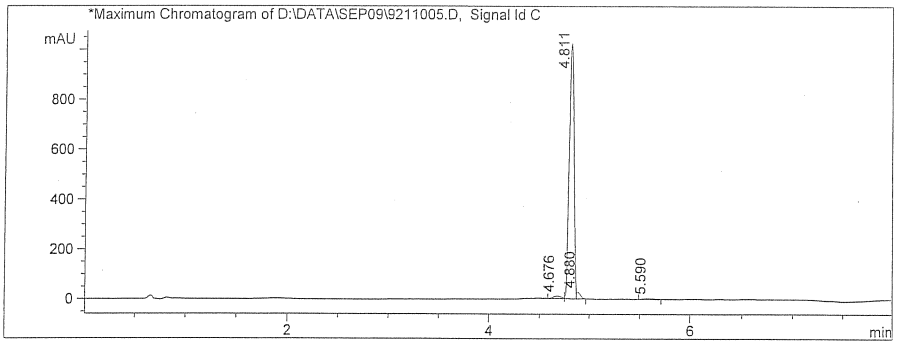
SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

Data file : D:\DATA\SEP09\9211005.D
Vial No. : P1-A-01
Injection Date : 9/22/2009 8:55:23 AM
Injection vol : 2ul
Sample Name : C-1502-07
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

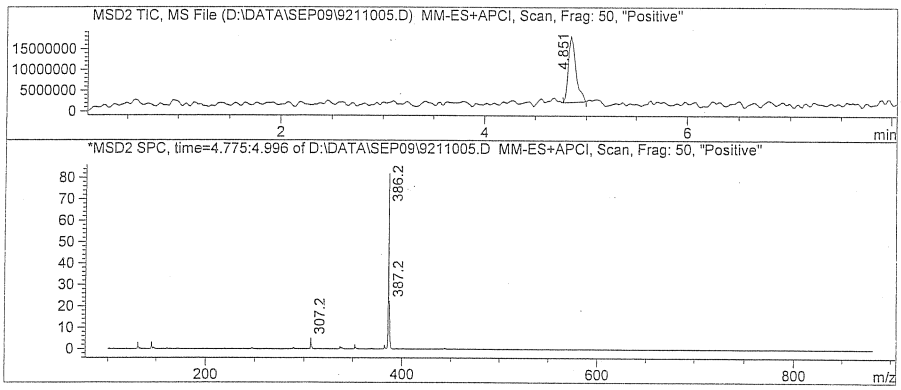
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.5 6.0 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{24}H_{23}N_3O_2$
Exact Mass: 385.18
Mol. Wt.: 385.46



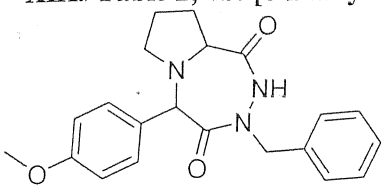
Peak No	RT min	Area	Area %
1	4.676	5.917e+001	1.630
2	4.811	3.484e+003	95.946
3	4.880	6.678e+001	1.839
4	5.590	2.122e+001	0.585



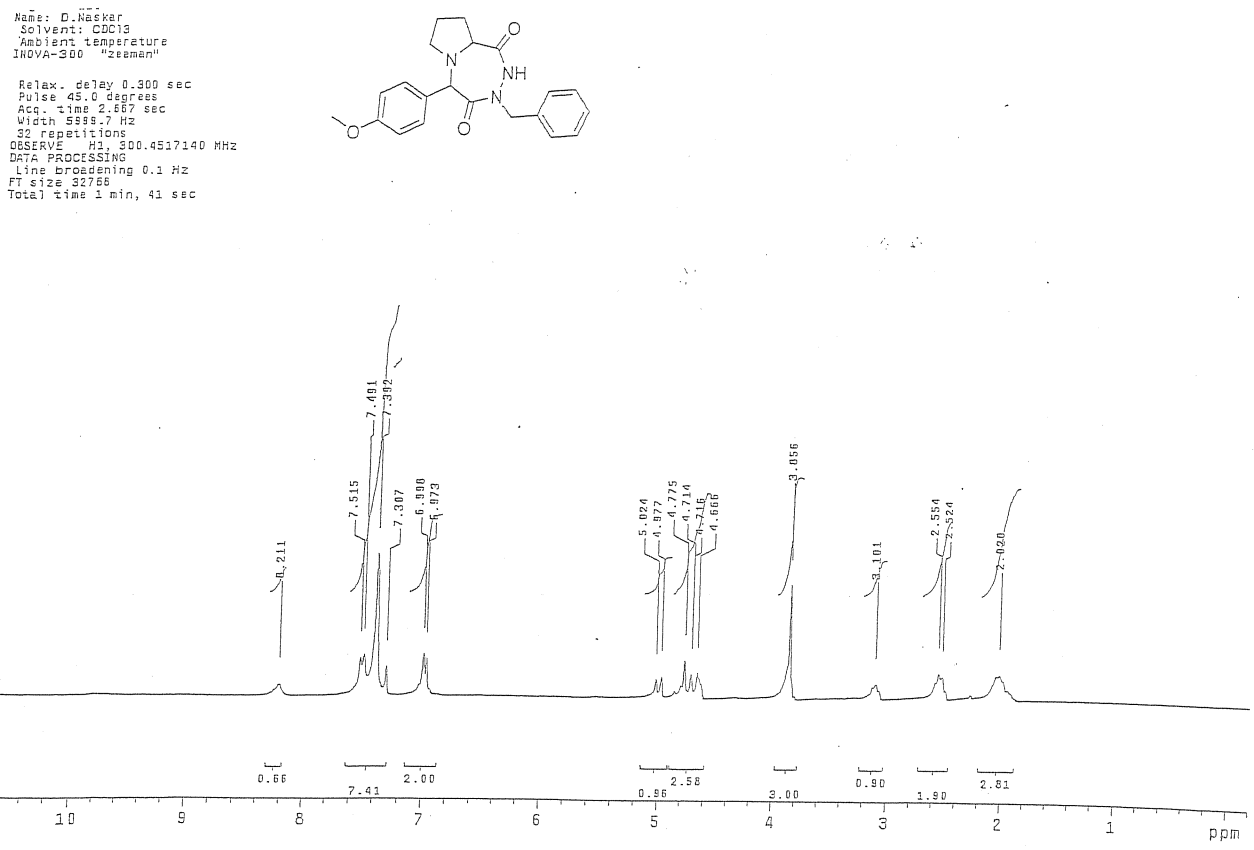
Analysed by :

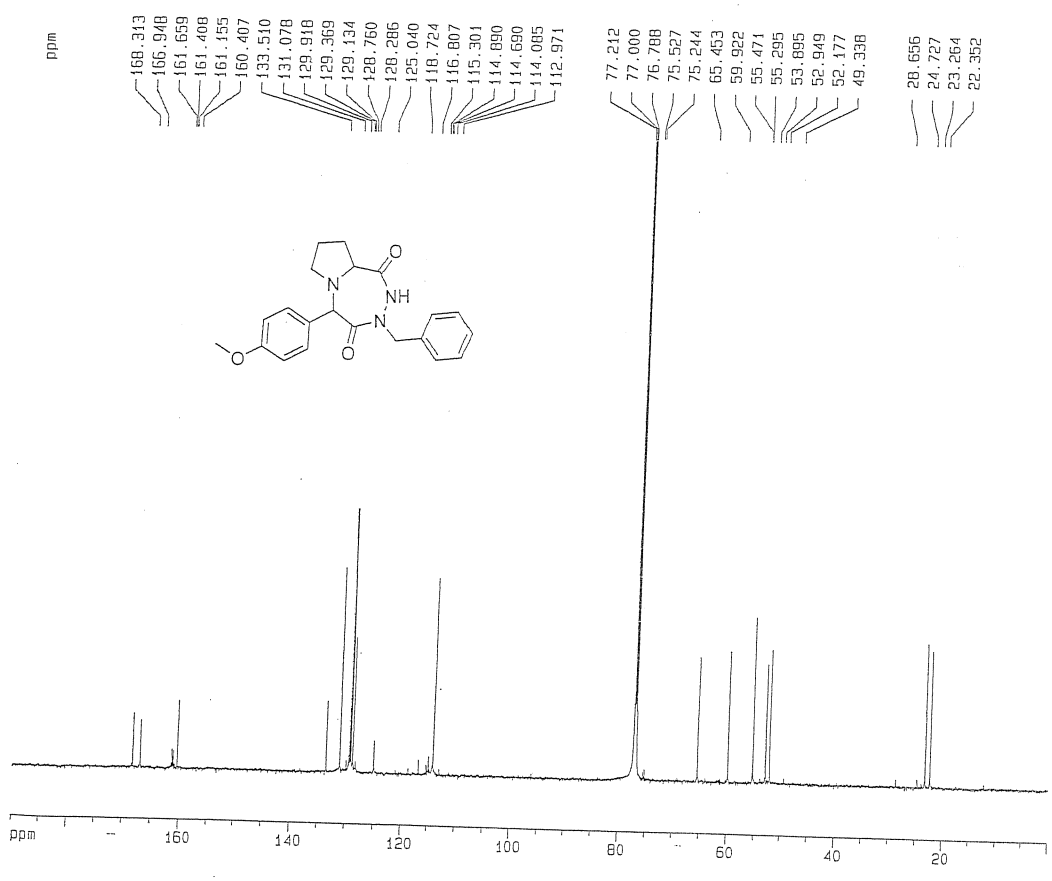
Instrument Code : SC/AD/10-014

XIX.. Table 2, 4b: [6-Benzyl-4-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-5,8-dione]



White solid, m.p (Met-TempII): 64-65°C (uncorrected); ^{1}H NMR ($CDCl_3$, 300MHz): δ = 2.02 (m, 3H), 2.55 (m, 2H), 3.10 (m, 1H), 3.86 (s, 3H), 4.66 (m, 2H), 4.78 (m, 1H), 5.02 (d, J= 14.1 Hz, 1H), 6.99 (d, J= 7.5 Hz, 2H), 7.49 (m, 5H), 7.52 (d, J= 7.2 Hz, 2H), 8.21 (br. s, 1H); ^{13}C NMR ($CDCl_3$, 75MHz): 22.35, 23.26, 52.18, 52.95, 55.29, 59.92, 65.45, 114.08, 125.04, 128.76, 129.13, 129.37; 131.08, 133.51, 160.41, 166.95, 168.31; LCMS (ELSD): 366 ($M+H^+$); HRMS: 366.181249 [Calcd for $C_{21}H_{24}N_3O_3$ 366.181767 $(M+H)^+$].





Current Data Parameters

EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

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PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	42821
DS	4
SWH	37593.984 Hz
FIDRES	1.147277 Hz
AQ	0.4358544 sec
RG	4096
DW	13.300 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	150.9193478 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	-6.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	600.1324005 MHz

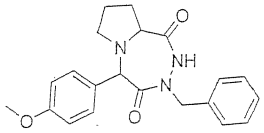
F2 - Processing parameters

SI	32768
SF	150.9028157 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

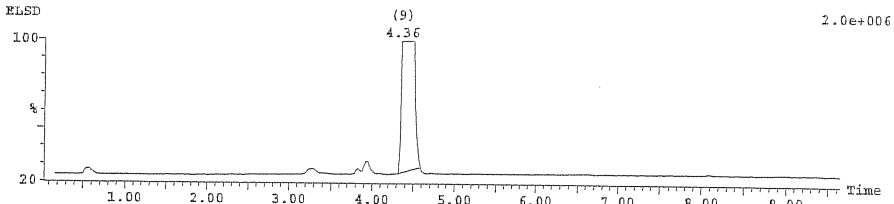
1D NMR plot parameters

CX	20.00 cm
F1P	190.000 ppm
F1	28671.54 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	9.50000 ppm/cm
HZCM	1433.5 Hz/cm

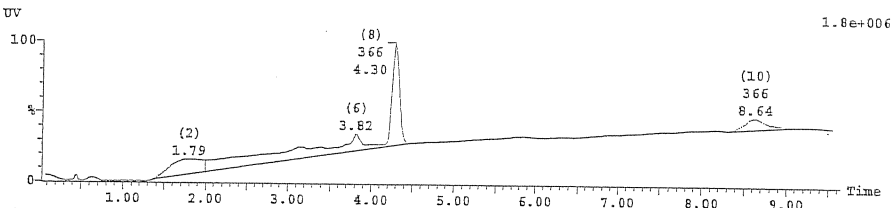
Sample Report:



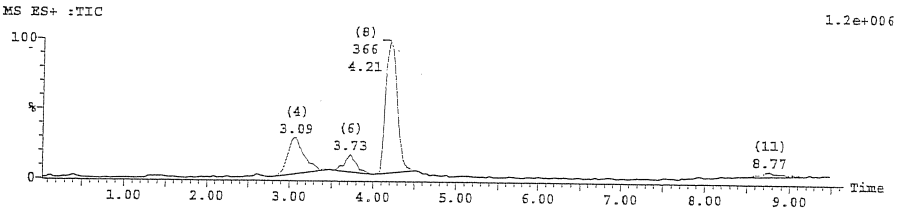
$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

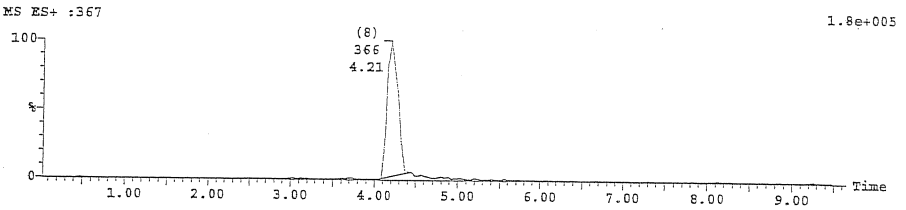


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
9	4.37	2.722e+005	100.00	0.280	1.520e+006		

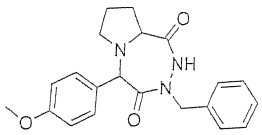


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
2	1.80	8.535e+004	17.02	0.620	1.932e+005		
6	3.82	2.314e+005	46.14	1.960	2.135e+005		
8	4.30	1.411e+005	28.14	0.540	1.315e+006	366.00	Found
10	8.64	4.366e+004	8.71	0.760	1.456e+005	366.00	Found



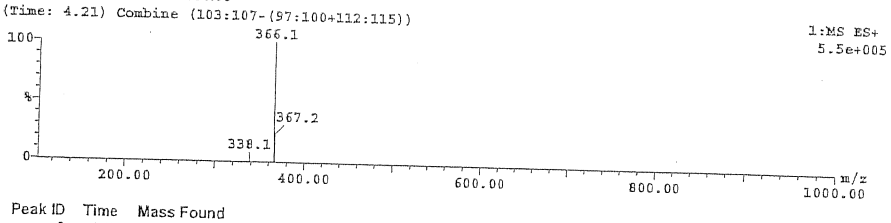


Sample Report (continued):

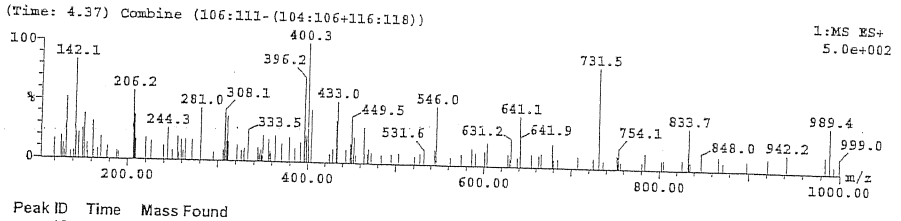


$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

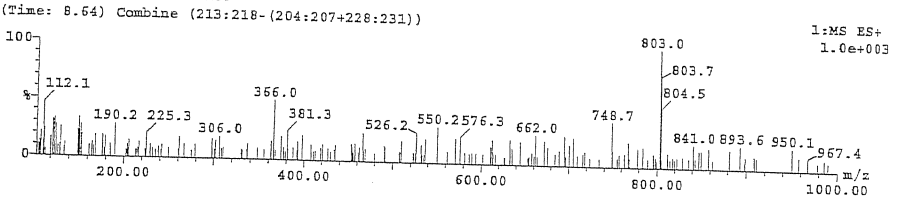
Peak ID Time Mass Found
8 4.21 366.00
(Time: 4.21) Combine (103:107-(97:100+112:115))



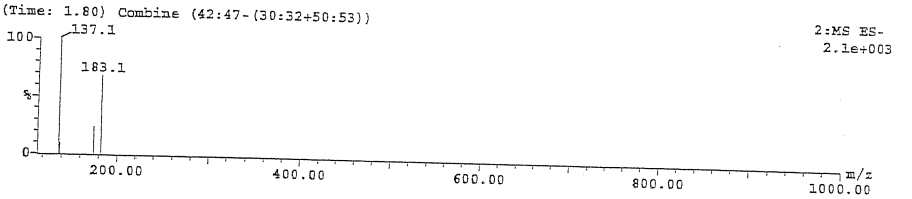
Peak ID Time Mass Found
9 4.37
(Time: 4.37) Combine (106:111-(104:106+116:118))



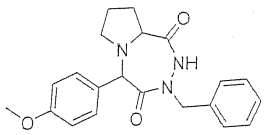
Peak ID Time Mass Found
10 8.64 366.00
(Time: 8.64) Combine (213:218-(204:207+228:231))



Peak ID Time Mass Found
2 1.80
(Time: 1.80) Combine (42:47-(30:32+50:53))

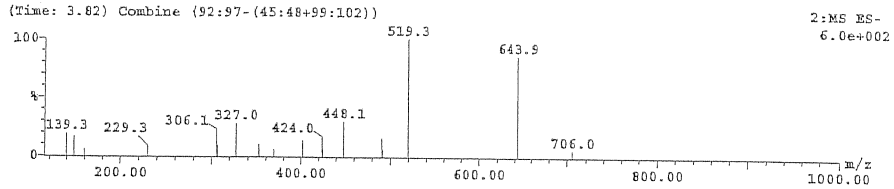


Sample Report (continued):

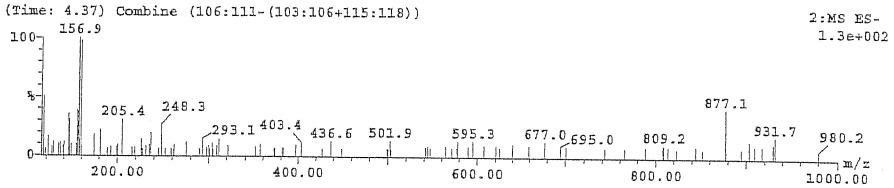


$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

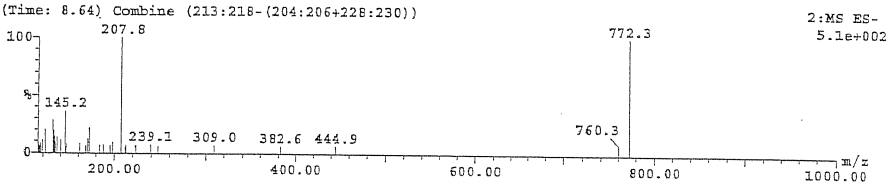
Peak ID Time Mass Found
6 3.73

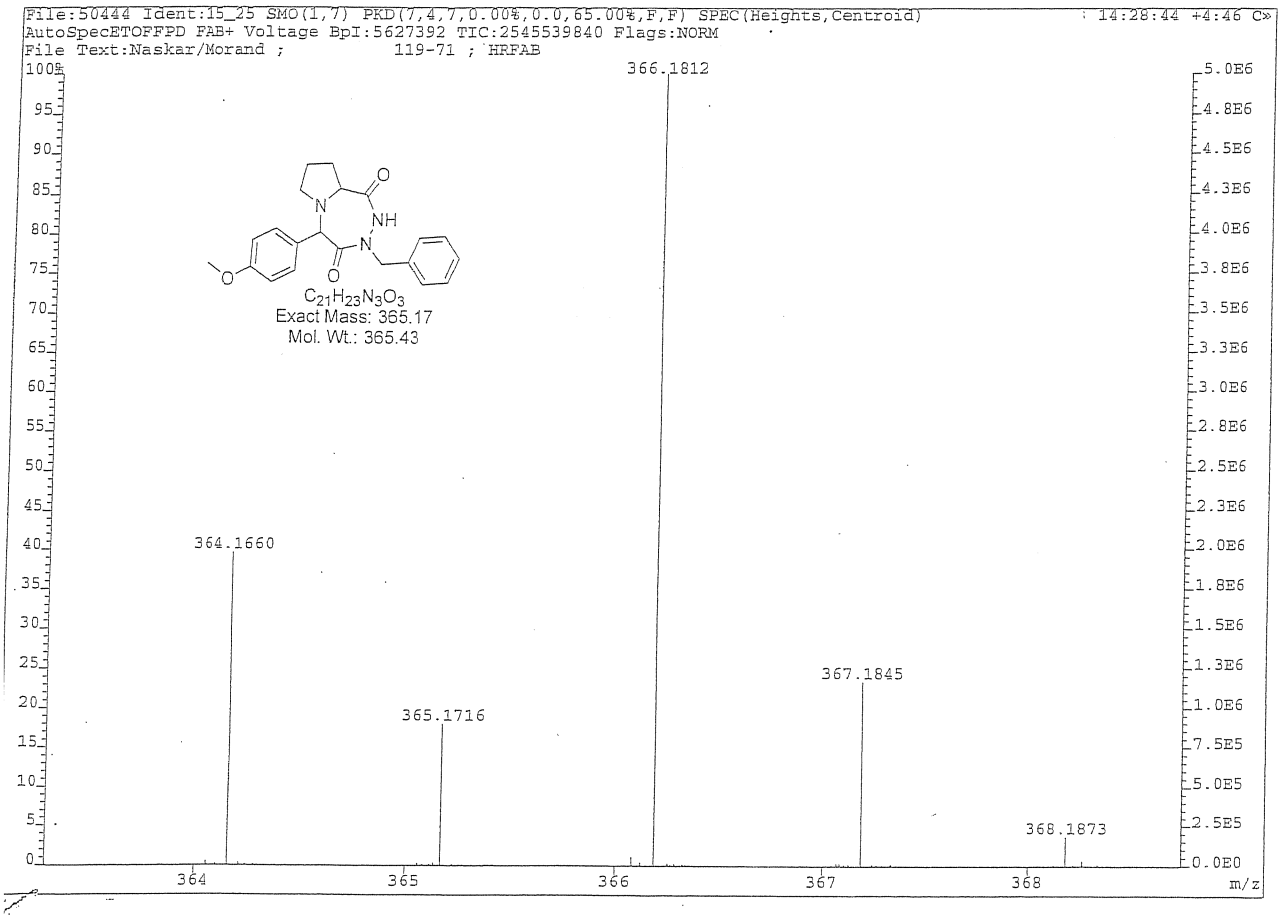


Peak ID Time Mass Found
9 4.37



Peak ID Time Mass Found
10 8.64



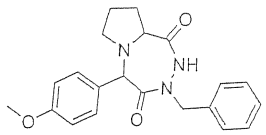

File:50444 Ident:15_25 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F) SPEC(Heights,Centroid)
AutoSpecETOFFPD FAB+ Voltage BpI:5627392 TIC:2545539840 Flags:NORM
File Text:Naskar/Morand ; 119-71 ; HRFAB
366.1812
364.1660
365.1716
367.1845
368.1873
C21H23N3O3
Exact Mass: 365.17
Mol. Wt.: 365.43
m/z

Elemental Composition

File:50444 Ident:15_25 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F)
~~AutoSpecETOFFPB-FAB+ Voltage~~ BpI:5627392 TIC:2545539840 Flags:NORM
File Text:Naskar/Morand ; 119-71 ; HRFAB
Heteroatom Max: 50 Ion: Both Even and Odd
Limits:

363.309	3.0					-0.5	0	0	0	3	3
368.745	100.0		10.0		Theor.	150.0	80	1	110	3	3

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O
367.184502	23.3			1.7	0.6	367.185122	11.5	20	1	24	3	3
366.181249	100.0	$(M+H)^+$		1.4	0.5	366.181767	11.5	21		24	3	3
365.171583	17.9			-5.8	-2.1	365.169472	12.5	20	1	22	3	3
				6.5	2.4	365.173942	12.0	21		23	3	3
364.166000	39.5			0.3	0.1	364.166117	12.5	21		22	3	3

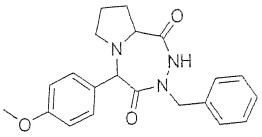


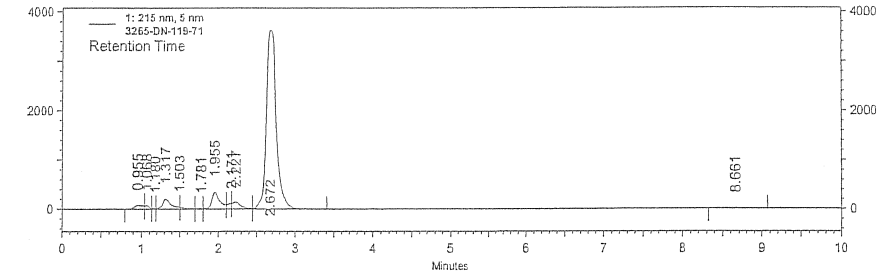
$C_{21}H_{23}N_3O_3$
Exact Mass: 365.17
Mol. Wt.: 365.43

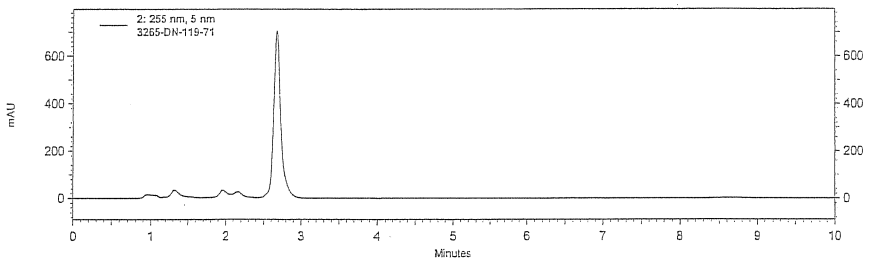
Area Percent Report

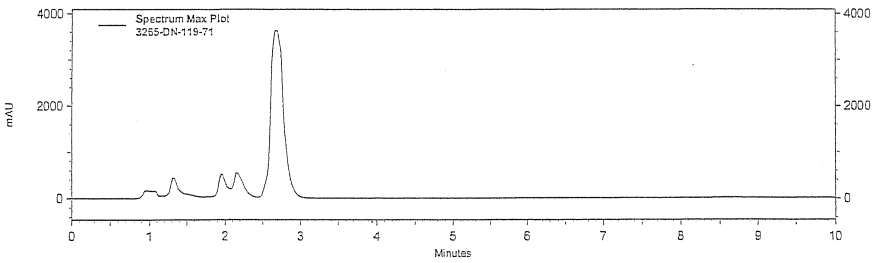
Data File: C:\ChromQuest\ -119-71.dat

Analyst: System
Sample ID: 3265-DN-119-71 Vial: A01 Injection Volume: 10



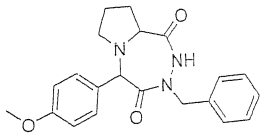






Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\50acnsjs.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

		0.955	593786	1.47
SV		1.068	292539	0.72
VV		1.180	60474	0.15
VS		1.317	1688232	4.17
SS		1.503	253051	0.63
SV		1.781	72871	0.18
VS		1.955	2491941	6.16
SV		2.171	410787	1.02
VS		2.227	1074351	2.66
SV		2.672	33404453	82.59
VB		8.661	103536	0.26
BB				
	Totals		40446021	100.00



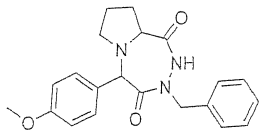
Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\50acnsjs.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

Area Percent Report

Data File: C:\ChromQuest\ -119-71.dat

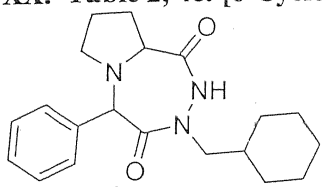
2: 255 nm, 5 nm
Results (Original)

Name	Retention Time	Area	Area Percent	Integration Codes

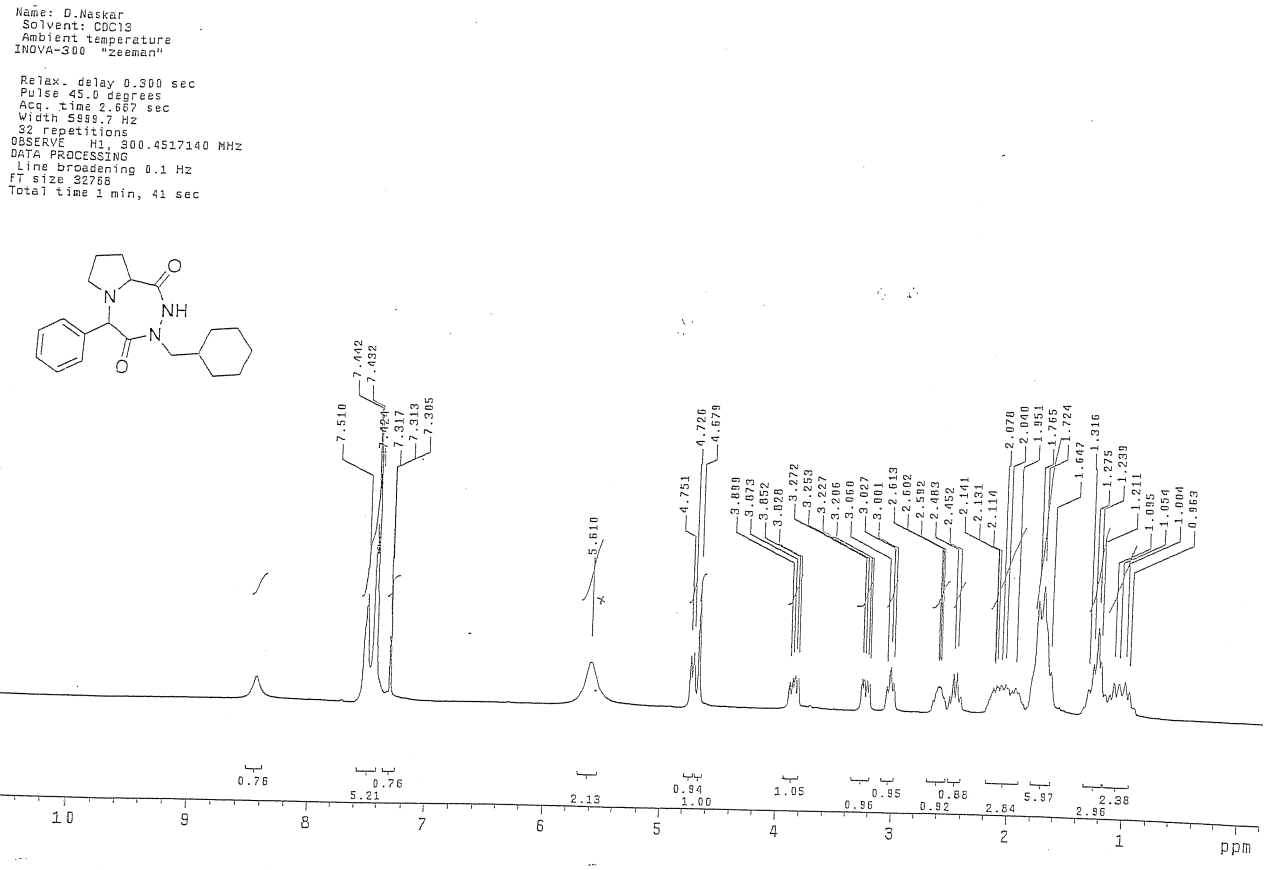


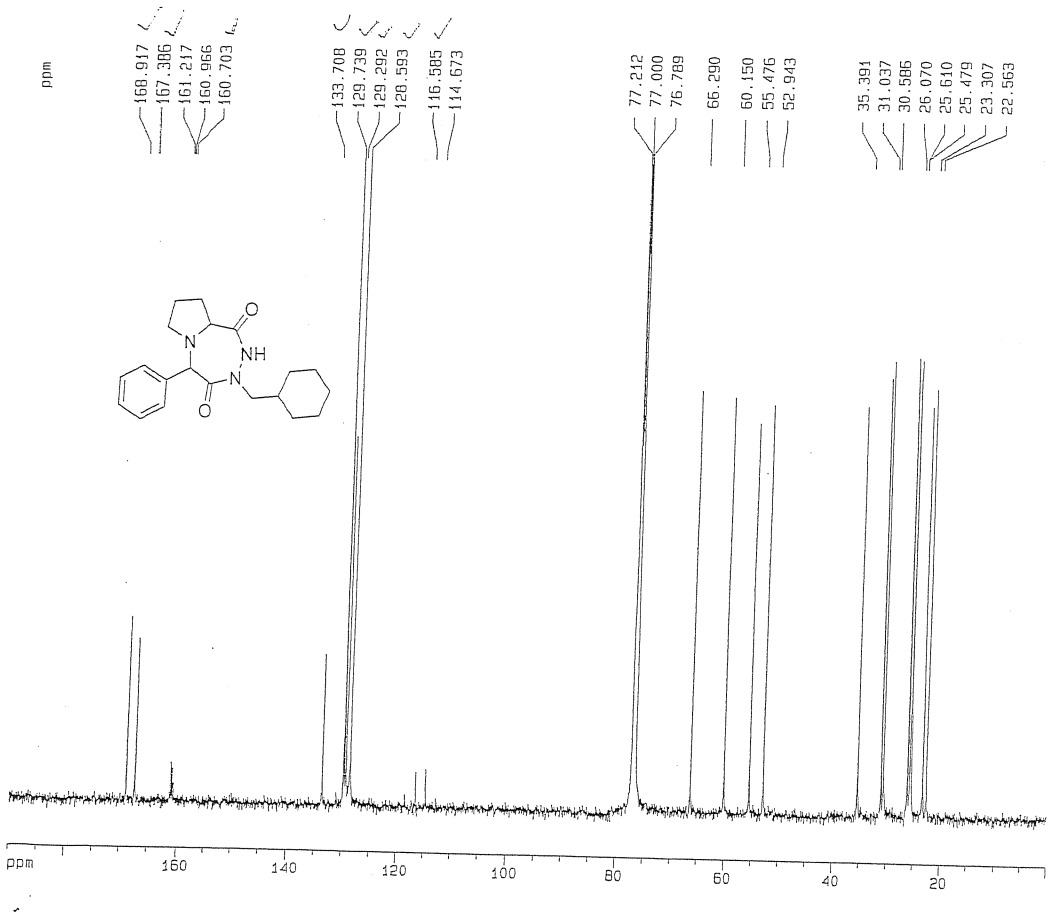
Instrument Name:	System 2	Software Version:	2.51
Acquisition Method:	C:\ChromQuest\METHODS\50acnsjs.met		
Sequence:	C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq		

XX. Table 2, 4c: [6-Cyclohexylmethyl-4-phenyl-hexahydro-3a,6,7-triaza-azulene-5,8-dione]



Liquid; ^{1}H NMR ($CDCl_3$, 300MHz):δ= 0.96-1.28 (m, 5H), 1.65-1.77 (m, 6H), 1.95-2.14 (m, 3H), 2.45 (m, 1H), 2.61 (m, 1H), 3.06 (m, 1H), 3.27 (m, 1H), 3.87 (m, 1H), 4.68 (s, 1H), 4.75 (m, 1H), 7.43-7.51 (m, 5H), 8.45 (br. s, 1H); ^{13}C NMR($CDCl_3$, 75MHz): 22.56, 23.31, 25.48, 25.61, 26.07, 30.59, 31.04, 35.39, 52.94, 55.48, 60.15, 66.29, 116.59, 128.59, 129.29, 129.74, 133.71, 160.70; 167.39, 168.92; LCMS (ELSD): 342.2 ($M+H^+$); HRMS: 342.2167 [Calculated for $C_{20}H_{28}N_3O_2$ 342.2182 $(M+H)^+$].





Current Data Parameters

EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	zgpg30
TD	32000
SOLVENT	CDCl3
NS	44175
DS	4
SWH	37593.984 Hz
FIDRES	1.174812 Hz
AQ	0.4256500 sec
RG	1149.4
DW	13.300 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	150.9193478 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	-6.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	600.1324005 MHz

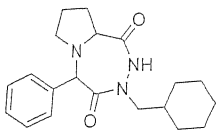
F2 - Processing parameters

SI	32768
SF	150.9028134 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.40

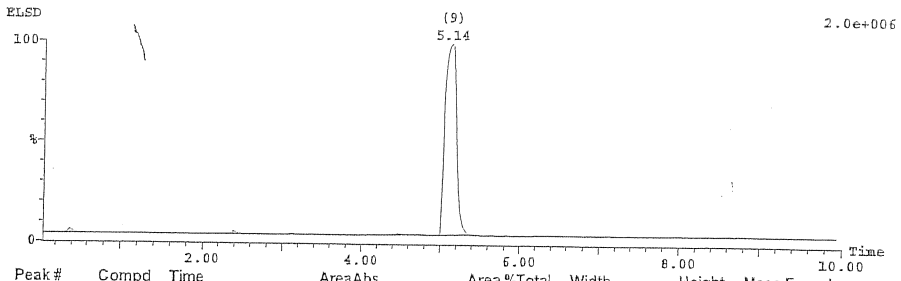
1D NMR plot parameters

CX	20.00 cm
F1P	190.000 ppm
F1	28671.54 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	9.50000 ppm/cm
HZCM	1433.57678 Hz/cm

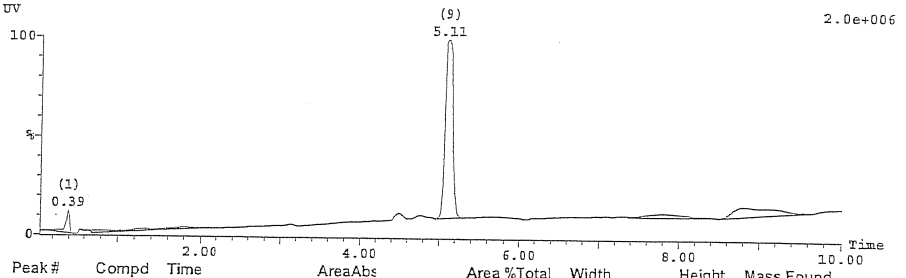
Sample Report (continued):



$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

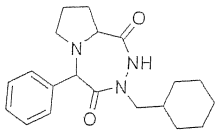


Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		0.37	3.058e+003	0.92	0.220	4.177e+004	
4		2.39	1.855e+003	0.56	0.340	2.396e+004	
5		3.49	4.469e+002	0.13	0.280	4.625e+003	
7		4.49	1.745e+003	0.53	0.260	1.619e+004	
8		4.73	1.054e+003	0.32	0.240	1.020e+004	
9		5.14	3.222e+005	96.97	0.430	1.953e+006	



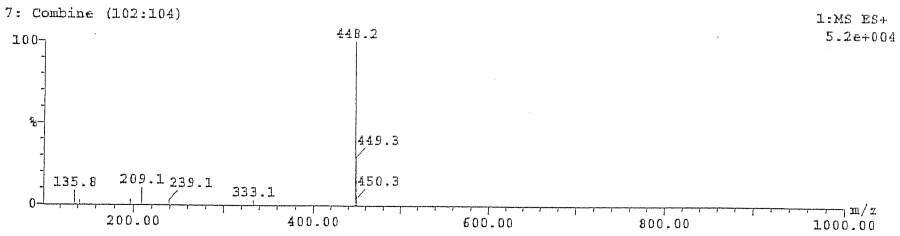
Peak #	Compd	Time	AreaAbs	Area %Total	Width	Height	Mass Found
1		0.39	1.577e+004	4.44	0.350	2.316e+005	
2		1.37	1.398e+004	3.94	0.740	2.125e+004	
3		1.89	1.008e+004	2.84	0.720	1.598e+004	
9		5.11	2.058e+005	57.98	0.430	1.828e+006	
13		7.76	2.512e+004	7.08	1.080	4.082e+004	
14		8.82	5.974e+004	16.83	1.150	1.026e+005	

Sample Report (continued):

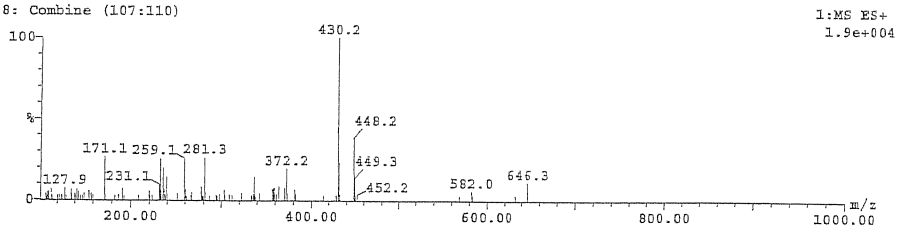


$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

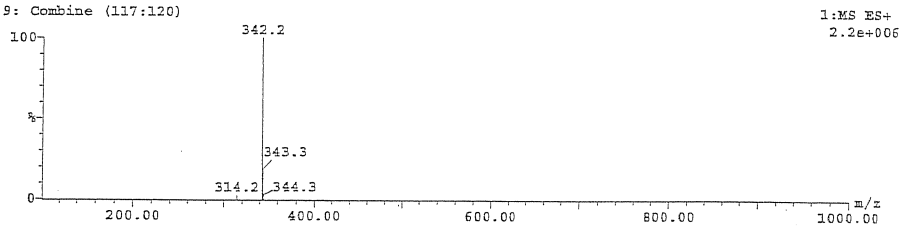
Mass Found Compound



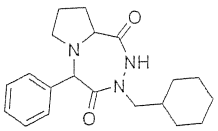
Mass Found Compound



Mass Found Compound

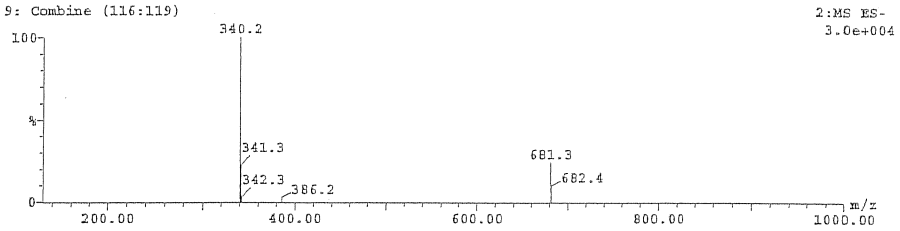


Sample Report (continued):

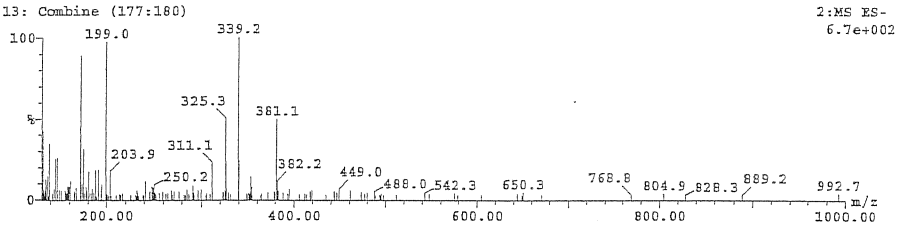


$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

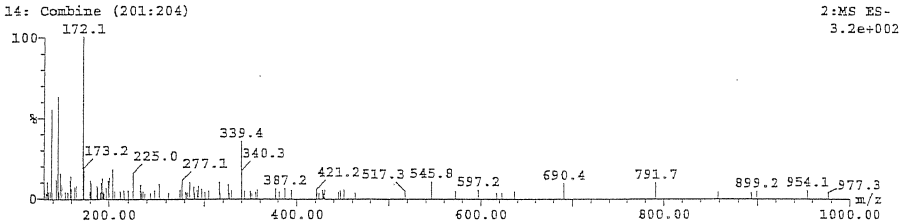
Mass Found Compound



Mass Found Compound



Mass Found Compound

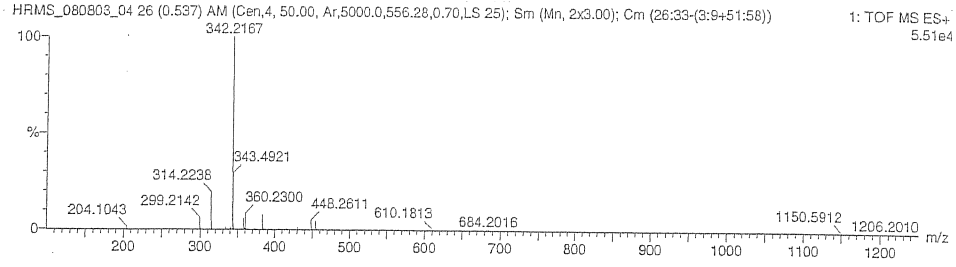


Elemental Composition Report

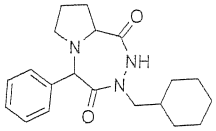
Single Mass Analysis

Tolerance = 30.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

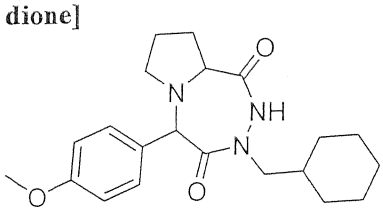


Minimum:				-1.5		
Maximum:		200.0	30.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
342.2167	342.2182	-1.4	-4.2	8.5	1	C20 H28 N3 O2

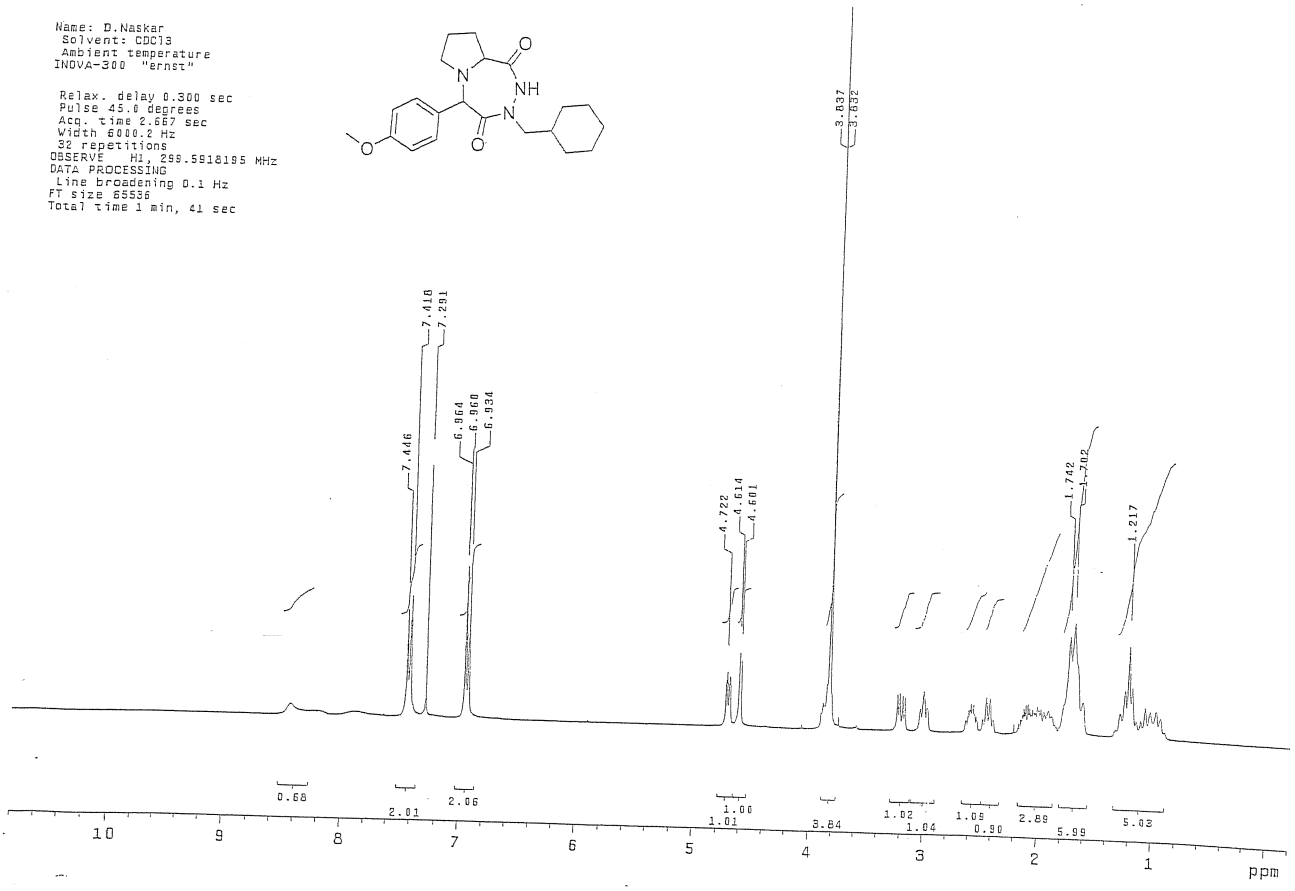


$C_{20}H_{27}N_3O_2$
Exact Mass: 341.21
Mol. Wt.: 341.45

XXI. Table 2, 4d: [6-Cyclohexylmethyl-4-(4-methoxy-phenyl)-hexahydro-3a,6,7-triaza-azulene-5,8-dione]



White Solid; m.p (Met-Temp): 89°-90°C (uncorrected); ^{1}H NMR($CDCl_3$, 300MHz):δ= 0.93-1.22 (m, 5H), 1.7-1.74 (m, 6H), 1.85-2.21(m, 3H), 2.45(m, 1H), 2.60 (m, 1H), 3.1(m, 1H), 3.21(m, 1H), 3.83(s, 3H), 3.84(m,1H), 4.61(m, 1H), 4.72(m, 1H), 6.95(d, J=9 Hz, 2H), 7.43(d, J= 8.4Hz, 2H), 8.4 (br. s, 1H); ^{13}C NMR($CDCl_3$, 75MHz): 22.91, 23.76, 25.89, 26.03, 26.48, 30.98, 31.44, 35.81, 53.32, 55.7, 55.9, 60.42, 66.04, 114.43, 125.84, 131.44, 160.74, 167.84, 169.16; LCMS (ELSD): 372.1 ($M+H^+$); HRMS: 372.229450 [Calculated for $C_{21}H_{30}N_3O_3$ 372.228717 $(M+H)^+$].



Current Data Parameters

EXPNO 140
PROCNO 1

F2 - Acquisition Parameters

INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	25000
DS	2
SWH	18939.395 Hz
FIDRES	0.577984 Hz
AQ	0.8651252 sec
RG	4096
DW	26.400 usec
DE	4.50 usec
TE	300.0 K
D1	0.20000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	9.00 usec
PL1	-1.00 dB
SFO1	75.4777800 MHz

========== CHANNEL f2 ==========

PCPD2	waltz16
NUC2	1H
PCPD2	112.00 usec
PL2	-3.00 dB
PL12	17.00 dB
PL13	17.00 dB
SFO2	300.1412006 MHz

- Processing parameters

SI	32768
SF	75.4702330 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

WA plot parameters

WFR	25.00 cm
WFP	225.483 ppm
	17015.77 Hz
WFP	-25.488 ppm
	-1923.62 Hz
WSP	0.200807 ppm/cm
	57.57574 Hz/cm

169.160
167.843
162.013
161.509
160.744
131.444
125.839
118.152
114.434
77.836
77.613
77.413
76.990
66.043
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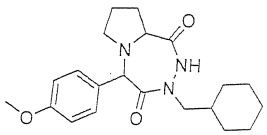
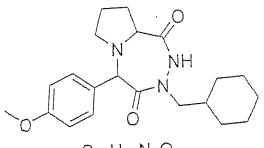
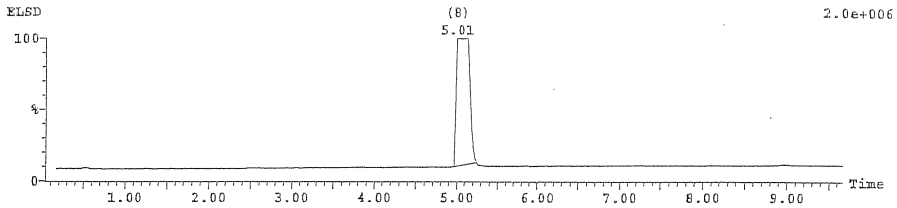
26.478
26.028
25.896
23.765
22.914



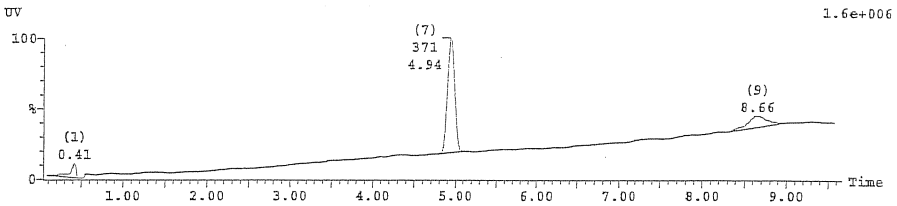
Sample Report:



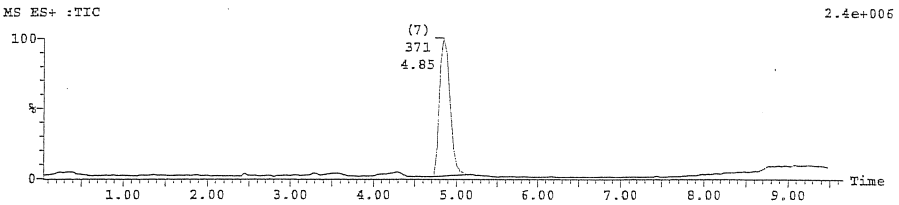
$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

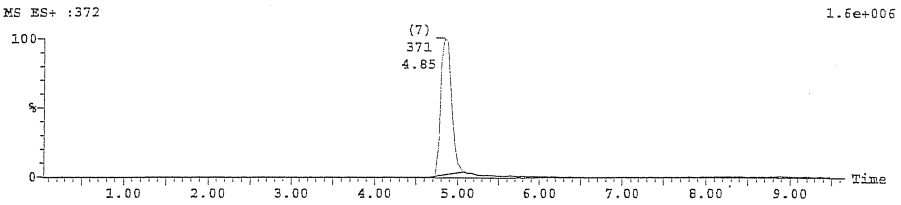


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
8	5.01	3.423e+005	100.00	0.300	1.833e+006		

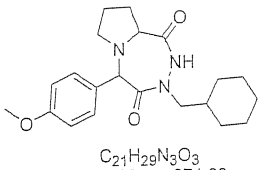


Peak #	Time	AreaAbs	Area %	Width	Height	Mass Found	Compound
1	0.42	1.427e+004	7.91	0.340	1.690e+005		
7	4.94	1.324e+005	73.44	0.300	1.314e+006	371.00	Found
9	8.66	3.361e+004	18.64	0.640	1.268e+005		

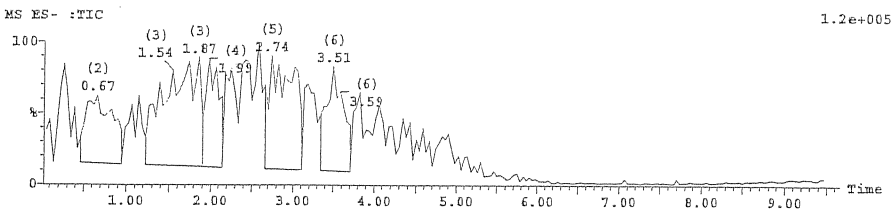


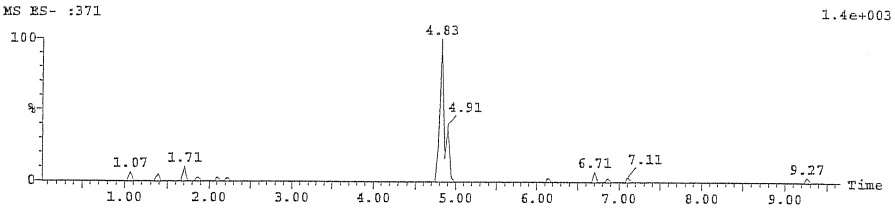


Sample Report (continued):



$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47





Peak ID Time Mass Found
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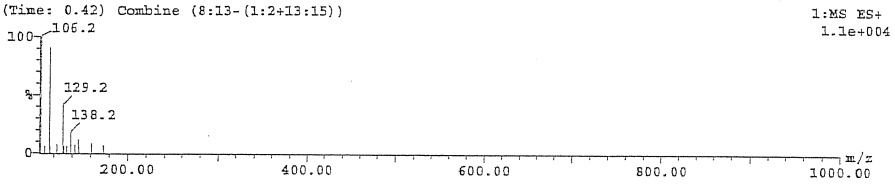
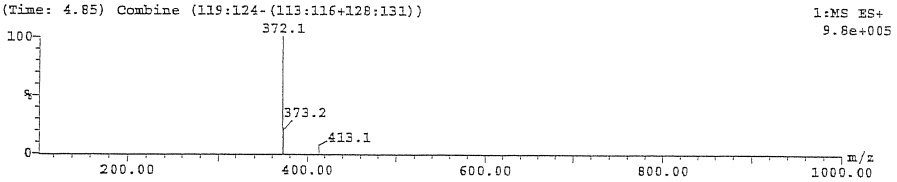
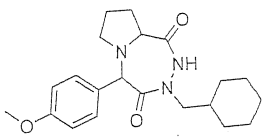


Peak ID Time Mass Found
7 4.85 371.00

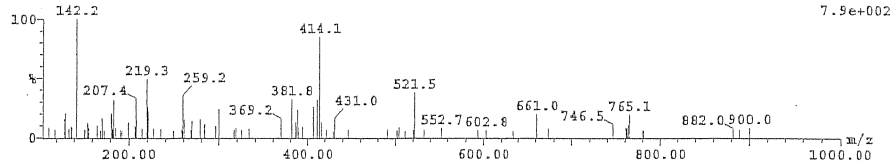


Sample Report (continued):

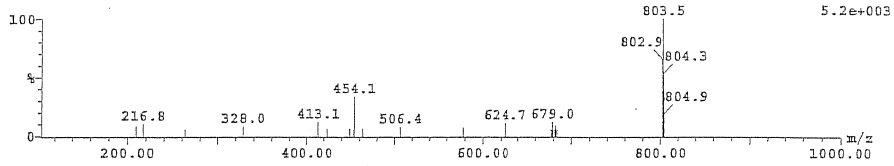


$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

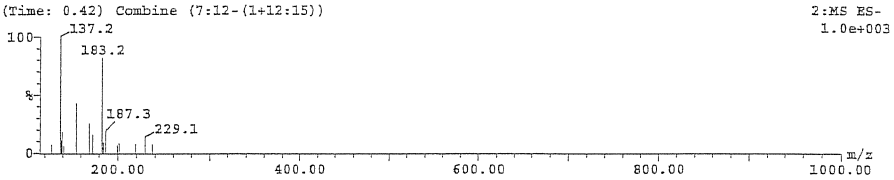
Peak ID Time Mass Found
8 5.01
(Time: 5.01) Combine (122:127-(120:122+132:135))
1:MS ES+
7.9e+002



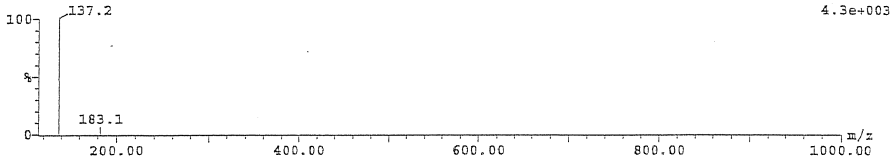
Peak ID Time Mass Found
9 8.66
(Time: 8.66) Combine (214:219-(204:206+225:227))
1:MS ES+
5.2e+003

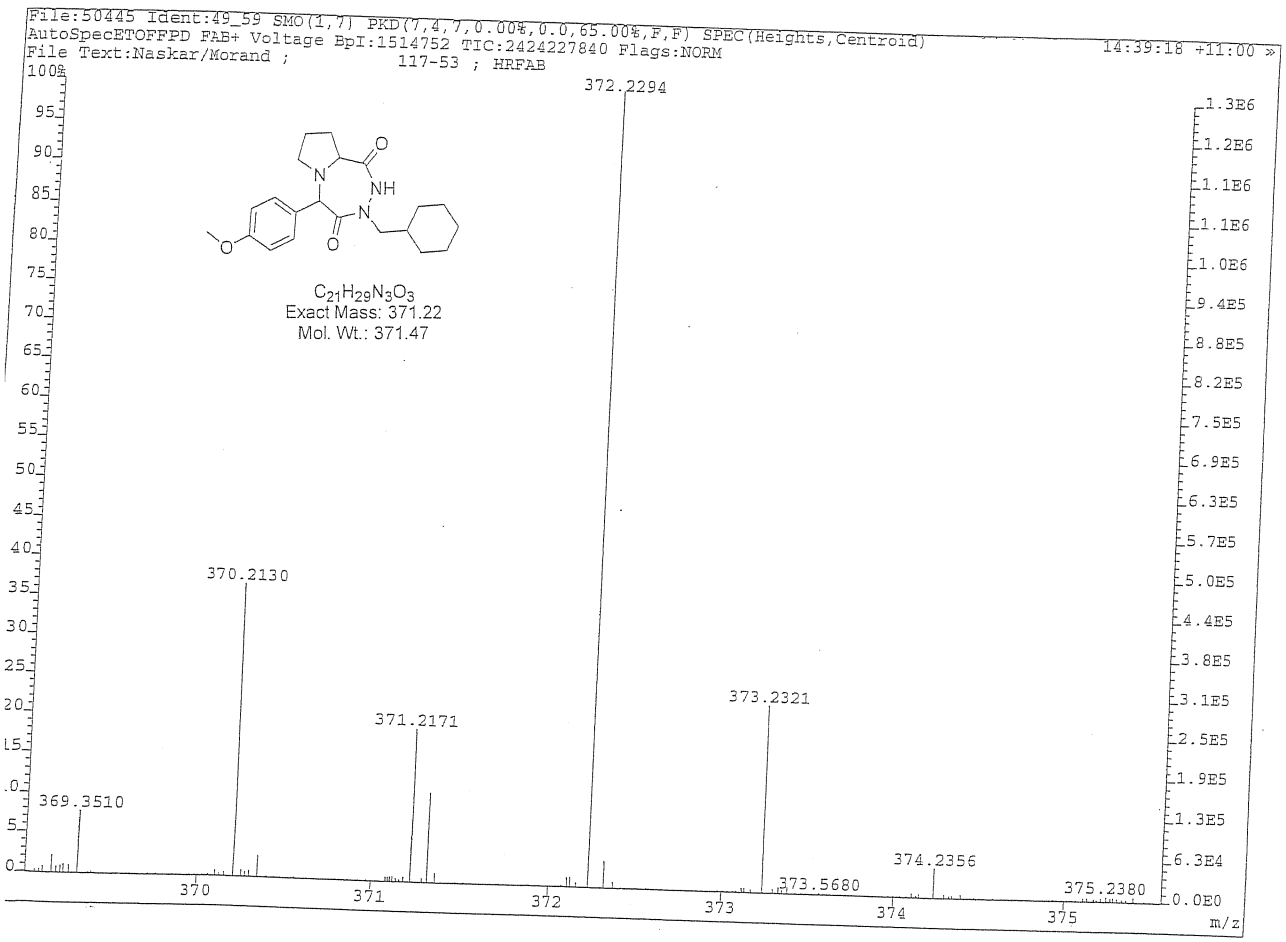


Peak ID Time Mass Found
1 0.42
(Time: 0.42) Combine (7:12-(1+12:15))
2:MS ES-
1.0e+003



Peak ID Time Mass Found
8 5.01
(Time: 5.01) Combine (122:127-(119:122+132:134))
2:MS ES-
4.3e+003




File:50445 Ident:49_59 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F) SPEC(Heights,Centroid)
AutoSpecETOFFPD FAB+ Voltage BpI:1514752 TIC:2424227840 Flags:NORM
File Text:Naskar/Morand ; 117-53 ; HRFAB
14:39:18 +11:00
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
O
N
NH
N
O
O
C21H29N3O3
Exact Mass: 371.22
Mol. Wt.: 371.47
372.2294
370.2130
371.2171
373.2321
369.3510
374.2356
373.5680
375.2380
370
371
372
373
374
375
m/z
1.3E6
1.2E6
1.1E6
1.1E6
1.0E6
9.4E5
8.8E5
8.2E5
7.5E5
6.9E5
6.3E5
5.7E5
5.0E5
4.4E5
3.8E5
3.1E5
2.5E5
1.9E5
1.3E5
6.3E4
0.0E0

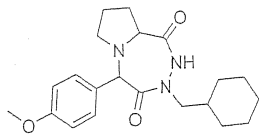
Elemental Composition

File:50445 Ident:49_59 SMO(1,7) PKD(7,4,7,0.00%,0.0,65.00%,F,F)
AutoSpecETOFFPD FAB+ Voltage BpI:1514752 TIC:2424227840 Flags:NORM
File Text:Naskar/Morand ; 117-53 ; HRFAB
Heteroatom Max: 50 Ion: Both Even and Odd
Limits:

369.053	3.0						-0.5	0	0	0	3	3
375.563	100.0			10.0			150.0	80	1	110	3	3
Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O
373.232144	23.3			-0.2	-0.1	373.232072	8.5	20	1	30	3	3
372.320857	3.3			4.7	1.8	372.322618	1.5	20		42	3	3
				-7.3	-2.7	372.318147	2.0	19	1	41	3	3
372.229450	100.0			-2.0	-0.7	372.228717	8.5	21		30	3	3
371.315564	11.1			-2.1	-0.8	371.314793	2.0	20		41	3	3
371.217111	18.9			-1.9	-0.7	371.216422	9.5	20	1	28	3	3
370.212989	36.7			0.2	0.1	370.213067	9.5	21		28	3	3

Theo

(M+H)+

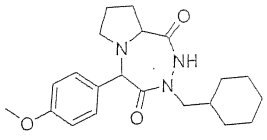


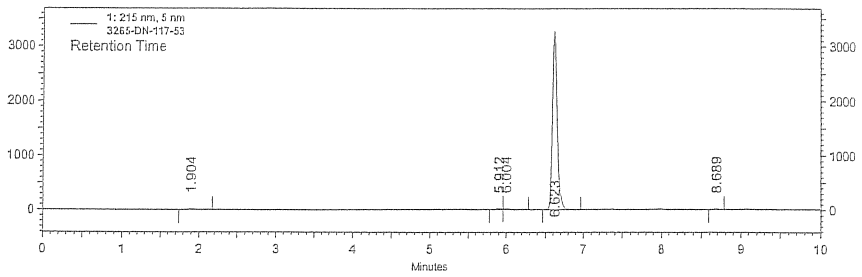
$C_{21}H_{29}N_3O_3$
Exact Mass: 371.22
Mol. Wt.: 371.47

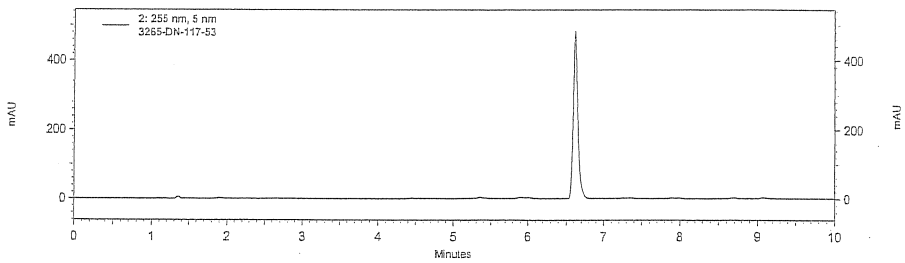
Area Percent Report

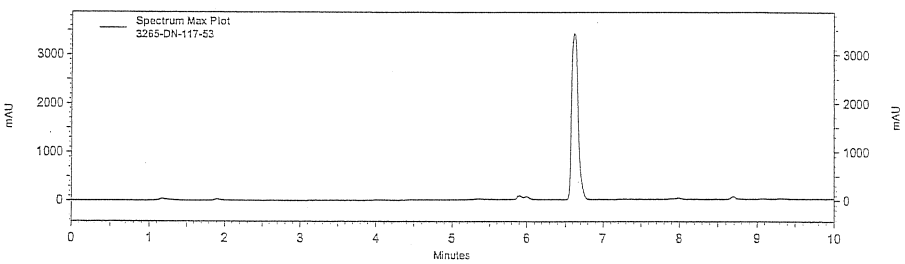
Data File: D:\Public\SYSTEM2\ 117-53.dat

Analyst: System
Sample ID: 3265-DN-117-53 Vial: A01 Injection Volume: 10









Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\c18short10min.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

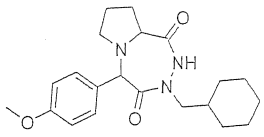
Area Percent Report

Data File: D:\Public\SYSTEM2\ 117-53.dat

1: 215 nm, 5 nm
Results
(Original)
Sample Data

	1.904	138524	0.94	SS
	5.912	82131	0.56	BV
	6.004	116430	0.79	VV
	6.623	14375828	97.27	VV
	8.689	67048	0.45	VS

Totals		14779961	100.00	



Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\c18short10min.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

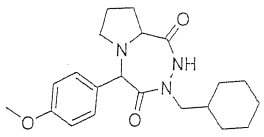
Area Percent Report

Data File: D:\Public\SYSTEM2\ 117-53.dat

2: 255 nm, 5 nm
Results (Original)

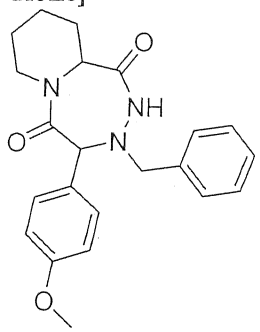
Name	Retention Time	Area	Area Percent	Integration Codes
	6.623	2053047	100.00	BV

Totals		2053047	100.00	

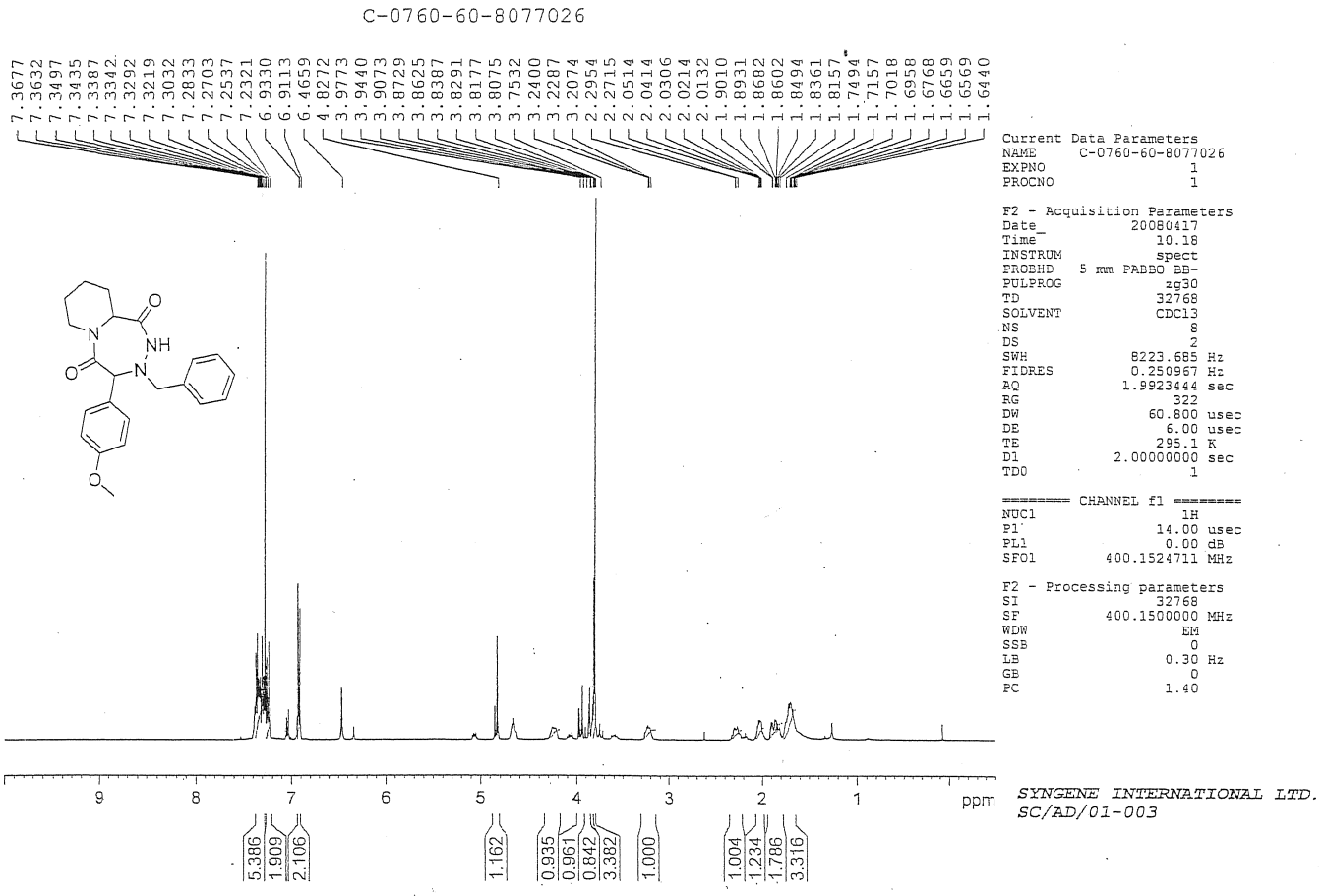


Instrument Name: System 2 Software Version: 2.51
Acquisition Method: C:\ChromQuest\METHODS\c18short10min.met
Sequence: C:\ChromQuest\SEQUENCE\hcs2790jzc-129-3-2-9.seq

XXII . Table 3, 6a: [7-Benzyl-6-(4-methoxy-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]

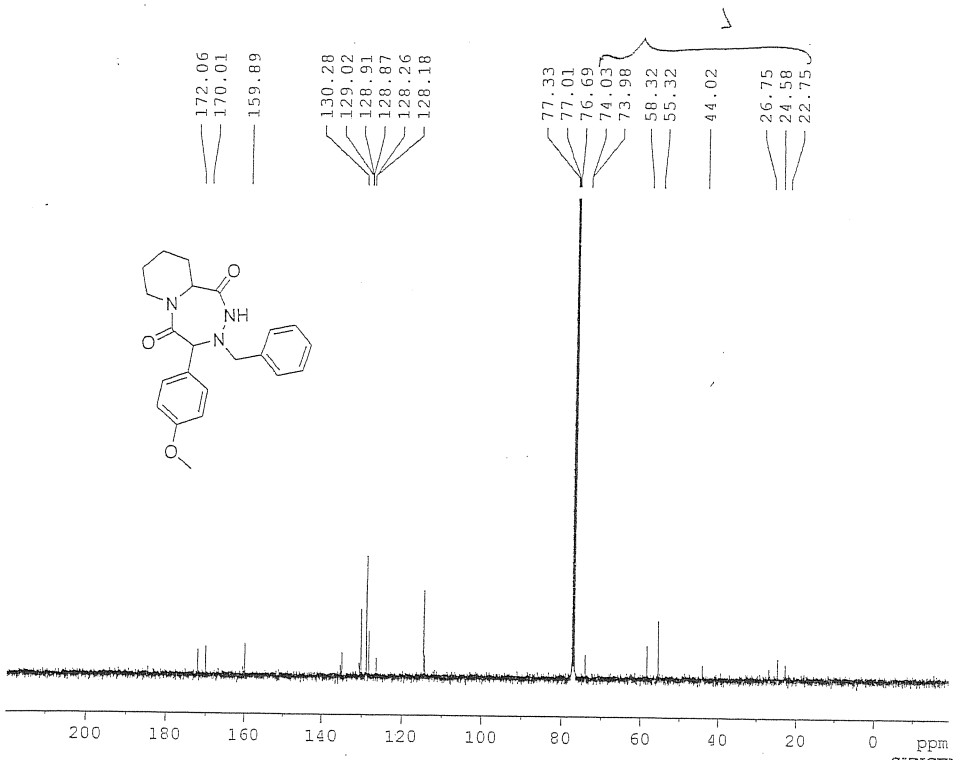


White solid; m.p (Met-Temp): 220°-221°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.64-1.75 (m, 3H), 1.82-1.90 (m, 2H), 2.01-2.05 (m, 1H), 2.26-2.29 (m, 1H), 3.20-3.24 (m, 1H), 3.81 (s, 3H), 3.86-3.87 (m, 1H), 3.94 (m, 1H), 4.23 (m, 1H), 4.83 (m, 1H), 6.47 (br. s, 1H), 6.92 (d, J= 8.68 Hz, 2H), 7.23-7.27 (m, 2H), 7.30-7.36 (m, 5H); ^{13}C NMR($CDCl_3$, 100 MHz): 22.75, 24.58, 26.75, 44.02, 55.32, 58.32, 73.98, 74.03, 114.51, 128.18, 128.26, 128.87, 128.91, 129.02, 130.28, 159.89, 170.01, 172.06; LCMS (UV): 380.5 ($M+H^+$).Anal. Calcd. for $C_{22}H_{25}N_3O_3$: C, 69.64; H, 6.64; N, 11.07. Found: C, 69.73; H, 6.69, N, 11.19.



SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

C-0760-60-8077026

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Current Data Parameters
NAME      C-0760-60-8077026
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080417
Time                 10.44
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     815
DS                       4
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6816244 sec
RG                    80.6
DW                20.800 usec
DE                 6.00 usec
TE                 296.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  7.13 usec
PL1                -3.00 dB
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL12              15.14 dB
PL13              15.00 dB
PL2                0.00 dB
SFO2         400.1516006 MHz

F2 - Processing parameters
SI                   32768
SF          100.6177980 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.00
```

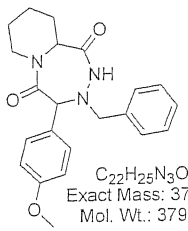
SYNGENE INTERNATIONAL LTD.
SC/AD/01-003

Acq. File: 8077026.wiff
Acq. Date: Thursday, April 17, 2008

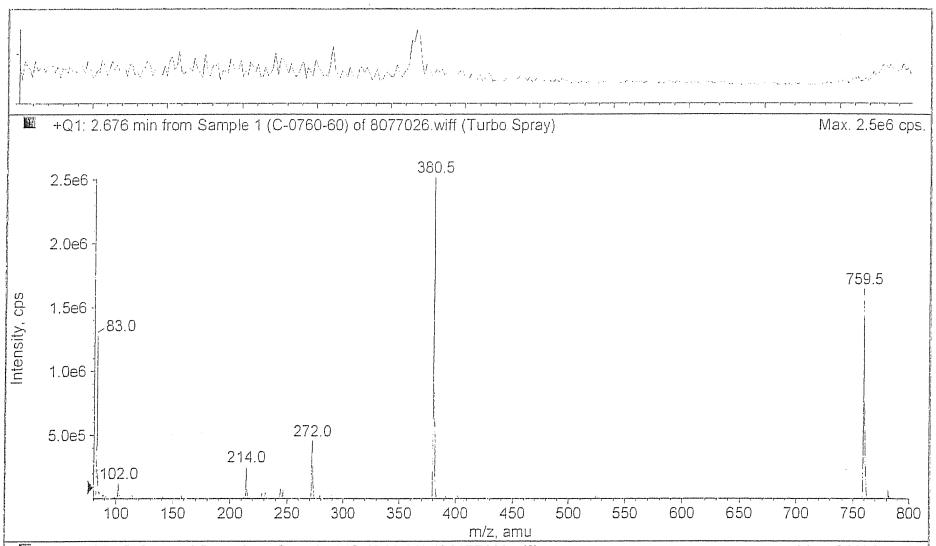
Syngene Intl.Ltd.
LC/MS REPORT

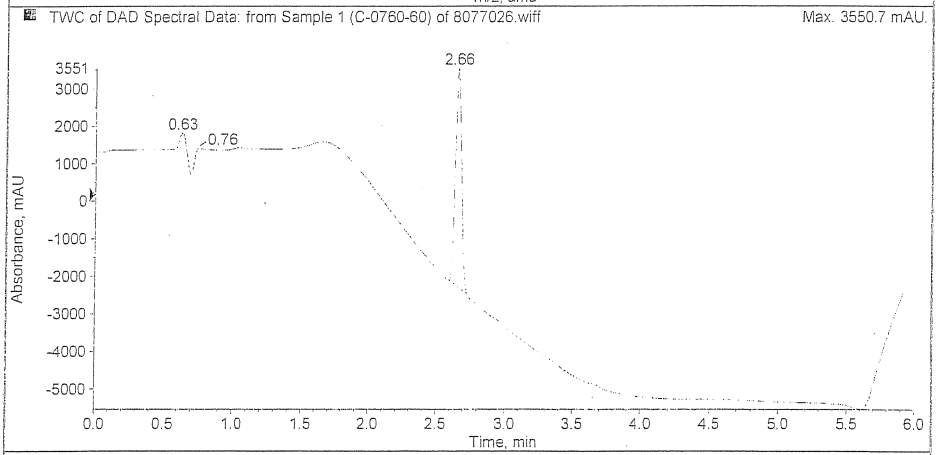
A: 0.1 % HCOOH, B: ACN, FR:1.0mL/min
COLUMN: SunfireC18(4.6X50) mm,5µ
B:0min-20%-1.0 min-65%, 2.0-4.0min-95%,4.5-6.0min-20%

Method Path: D:\Analyst Dat
Sample Name: C-0760-60
Sample ID: 14



$C_{22}H_{25}N_3O_3$
Exact Mass: 379.19
Mol. Wt.: 379.45





Peak List for "TWC of DAD Spectral Data: from Sample 1 (C-0760-60) of 8077026.wiff"

	Time (min)	Area (mAU x min)	% Area	Height (mAU)	% Height	Width (min)
1	2.6623	1.7917e4	100.000	5871.3456	100.0000	0.1333

Analysed By

SC/AD/10-003

Syngene International Ltd
A Biocon Company

HPLC REPORT

Data file : C:\CHEM32\1\DATA\APR08\8076969.D Vial No. : Vial 31

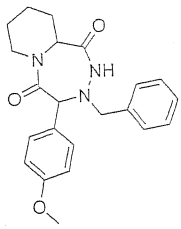
Injection Date :17/04/08 09:08:47 AM Injection vol : 6 µl

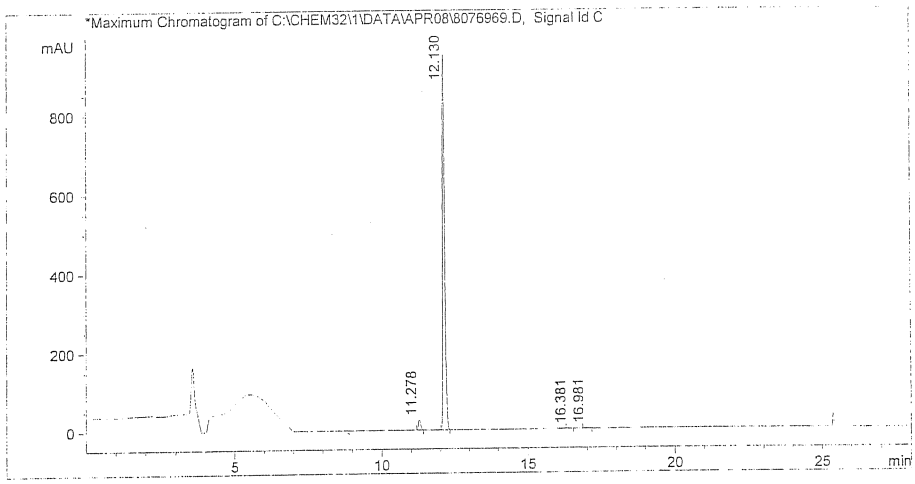
Sample Name : C-0760-60 Operator : Aravind

Sample info :
Acq Method :C:\CHEM32\1\METHODS\YA_TF7030.M

Method info : A:0.1% TFA in water B:ACN
YMC-AQ-(4.6X250)mm,5um
Flow:0.8mL/min

Time	% of B
0	30
15	100
20	100
23	30
28	30





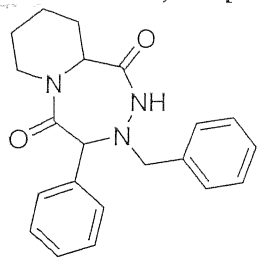
Peak No	RT min	Area	Area %
1	11.278	136.365	2.654
2	12.130	4979.673	96.930
3	16.381	6.629	0.129
4	16.981	14.734	0.287

End of report

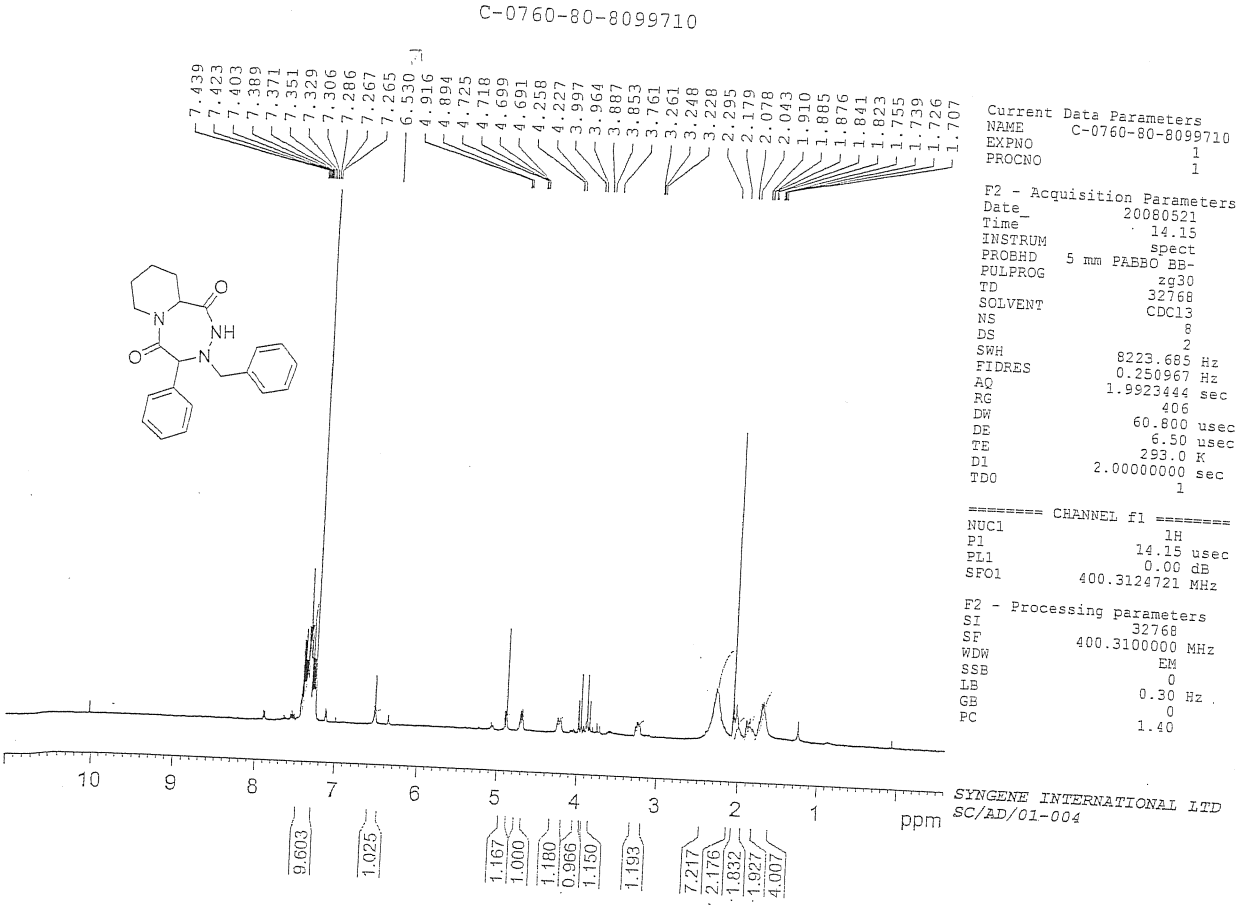
Analysed By :

Instrument Code : SC/AD/04-064

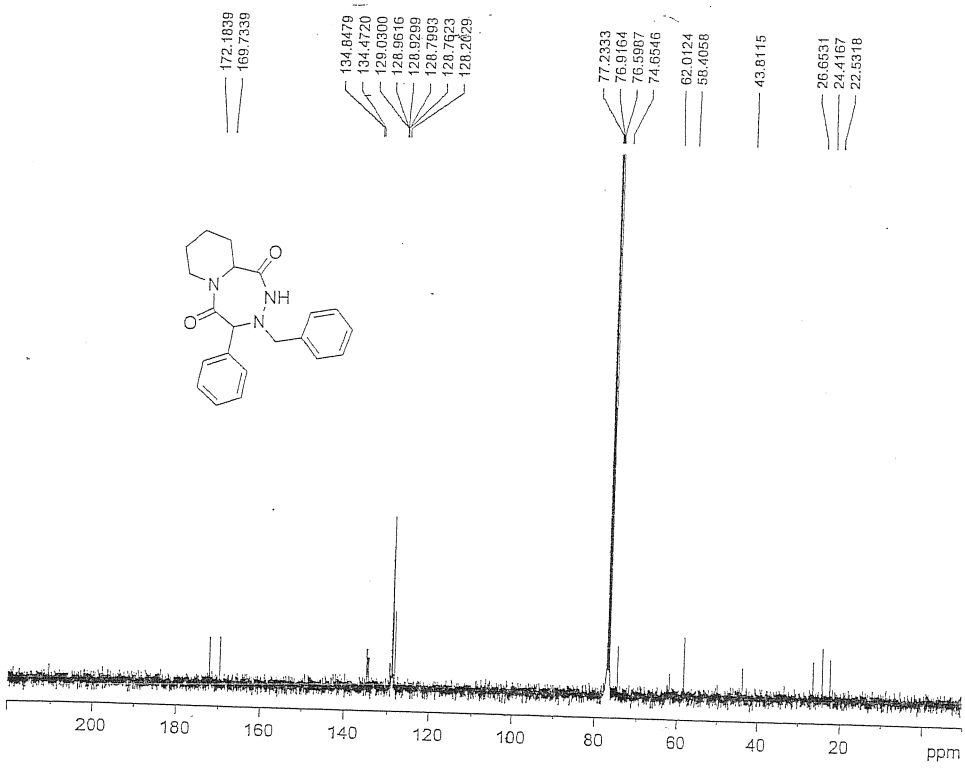
XXIII. Table 3, 6b: [7-Benzyl-6-phenyl-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]



White solid; m.p (Met-Temp): 167°-168°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.70-1.76 (m, 3H), 1.82-1.84 (m, 2H), 1.87-1.91 (m, 1H), 2.29 (m, 1H), 3.22 (m, 1H), 3.85-3.88 (m, 1H), 3.96-3.99 (m, 1H), 4.23-4.25 (m, 1H), 4.89-4.92 (m, 1H), 6.53 (br. s, 1H), 7.3-7.43 (m, 10H); ^{13}C NMR($CDCl_3$, 100 MHz): 22.53, 24.42, 26.65, 43.81, 58.41, 62.01, 74.65, 128.20, 128.76, 128.79, 128.93, 128.96, 129.03, 134.47, 134.85, 169.73, 172.18; LCMS (UV): 350.2 ($M+H^+$).Anal. Calcd. for $C_{21}H_{23}N_3O_2$: C, 72.18; H, 6.63; N, 12.03. Found: C, 72.15; H, 6.7, N, 12.11.



C-0760-80-8099710



Current Data Parameters

NAME	C-0760-80-8099710
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080522
Time	11.36
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1300
DS	2
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	0.5439988 sec
RG	1024
DW	16.600 usec
DE	6.00 usec
TE	299.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.30 usec
PL1	-1.00 dB
SFO1	100.6499905 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	15.00 dB
PL13	15.00 dB
SFO2	400.2316009 MHz

F2 - Processing parameters

SI	32768
SF	100.6379211 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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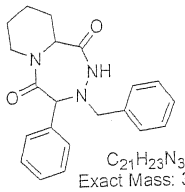
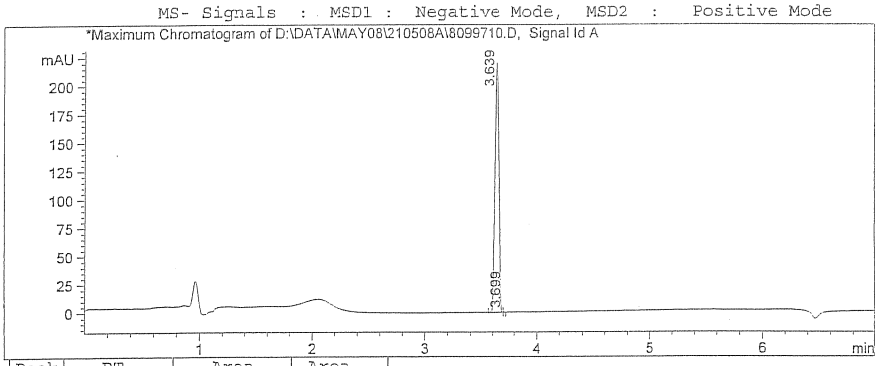
SC/AD/01-002

Data file : D:\DATA\MAY08\210508A\8099710.D
Vial No. : P1-E-05
Injection Date : 5/21/2008
Injection vol : 2 µL
Sample Name : C-0760-80
Acq Method : D:\DATA\MAY08\210508A\AT30-70 HCOOH.M

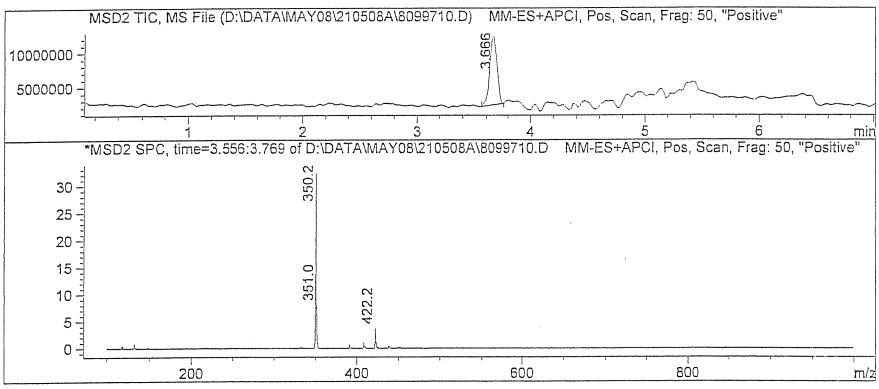
Method info :A : HCOOH ; B- ACN, Flow = 1.0 mL/min,
Column- Atlantis dC18 (75X4.6mm-5µm)
Time (min.): 0--3.5 3.5--4.5 4.5--5.0 5.0--7.0
% B : 30--95 95 95--30 30
MS-SCAN, ES\APCI: DAUL POLARITY, SINGLE QUAD.
MS- Signals : MSD1 : Negative Mode, MSD2 : Positive Mode



$C_{21}H_{23}N_3O_2$
Exact Mass: 349.18
Mol. Wt.: 349.43



Peak No	RT min	Area	Area %
1	3.639	6.017e+002	99.988
2	3.699	7.065e-002	0.012



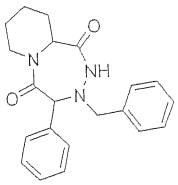
Analysed by :

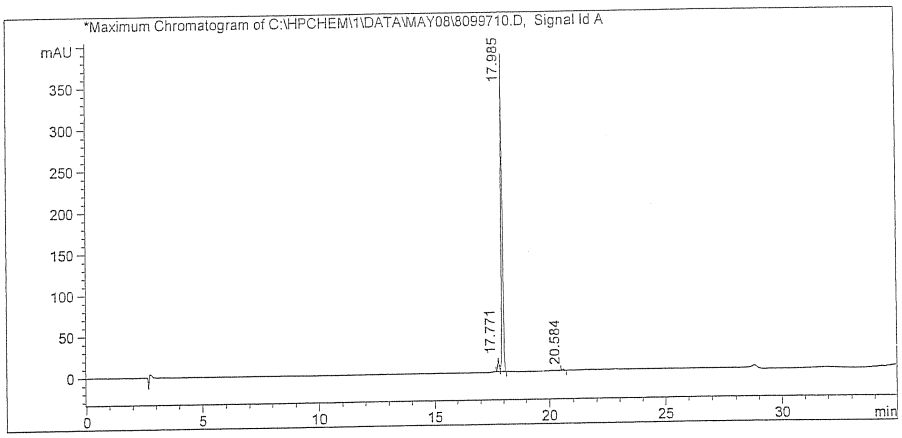
HPLC REPORT

Data file	:C:\HPCHEM\1\DATA\MAY08\8099710.D	Vial No.	:Vial 25
Injection Date	:5/22/2008 9:30:41 AM	Injection vol	:8µL
Sample Name	:C-0760-80	Injection No.	:1
Sample info	:	Acq Method	:XB_A9010.M

Method info : Column:XBRIDGE C18 (4.6*150)mm 3.5u
Mobile phase:A=10 mM NH4OAC; B=ACN
Flow 0.7ml/min

Time(min)	%B
0	10
5	10
15	60
20	95
25	95
28	10
35	10





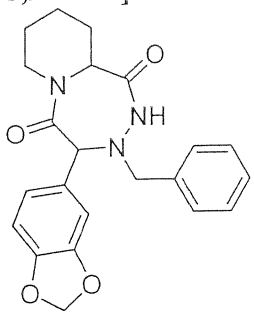
Peak No	RT min	Area	Area %
1	17.771	70.6368	4.480
2	17.864	7.1260	0.452
3	17.985	1489.2017	94.441
4	20.584	9.9012	0.628

End of report

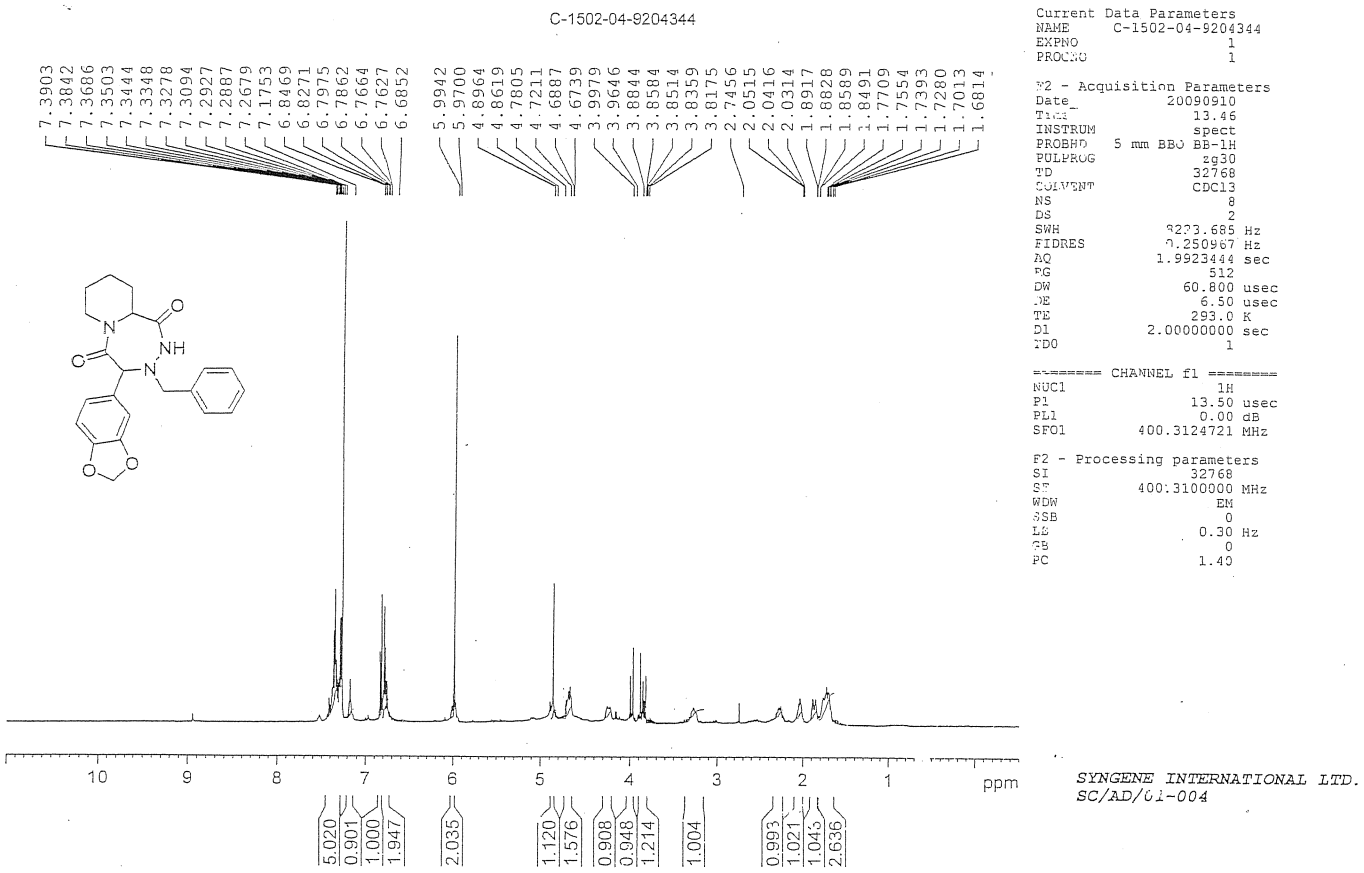
Analysed By :

Instrument Code : SC/AD/04-007

XXIV. Table 3, 6c: [6-Benzo[1,3]dioxol-5-yl-7-benzyl-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]



White solid; m.p: (Met-Temp): 61°-62°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.68-1.77 (m, 3H), 1.85-1.89 (m, 1H), 2.03-2.05 (m, 1H), 2.24 (m, 1H), 3.26-3.28 (m, 1H), 3.81-3.88 (m, 1H), 3.96-3.99 (m, 1H), 4.17 (m, 1H), 4.67-4.78 (m, 1H), 4.86 (s, 1H), 5.98 (s, 2H), 6.68-6.85 (m, 3H), 7.17 (m, 1H), 7.27-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.35, 24.31, 26.47, 43.86, 58.19, 61.88, 74.27, 101.35, 108.42, 108.69, 122.78, 128.20, 128.26, 128.37, 128.67, 134.74, 148.09, 148.25, 169.96,172.30; LCMS (UV): 394.2 ($M+H^+$). Anal. Calcd. for $C_{22}H_{23}N_3O_4$: C, 67.16; H, 5.89; N, 10.68. Found: C, 67.22; H, 5.85; N, 10.59.

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Current Data Parameters
NAME      C-1502-04-9204344
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20090910
Time                  13.46
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                9223.685 Hz
FIDRES             0.250967 Hz
AQ                 1.9923444 sec
RG                      512
DW                   60.800 usec
DE                    6.50 usec
TE                   293.0 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                   0.00 dB
SFO1          400.3124721 MHz

F2 - Processing parameters
SI                    32768
SF             400.3100000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.40
```

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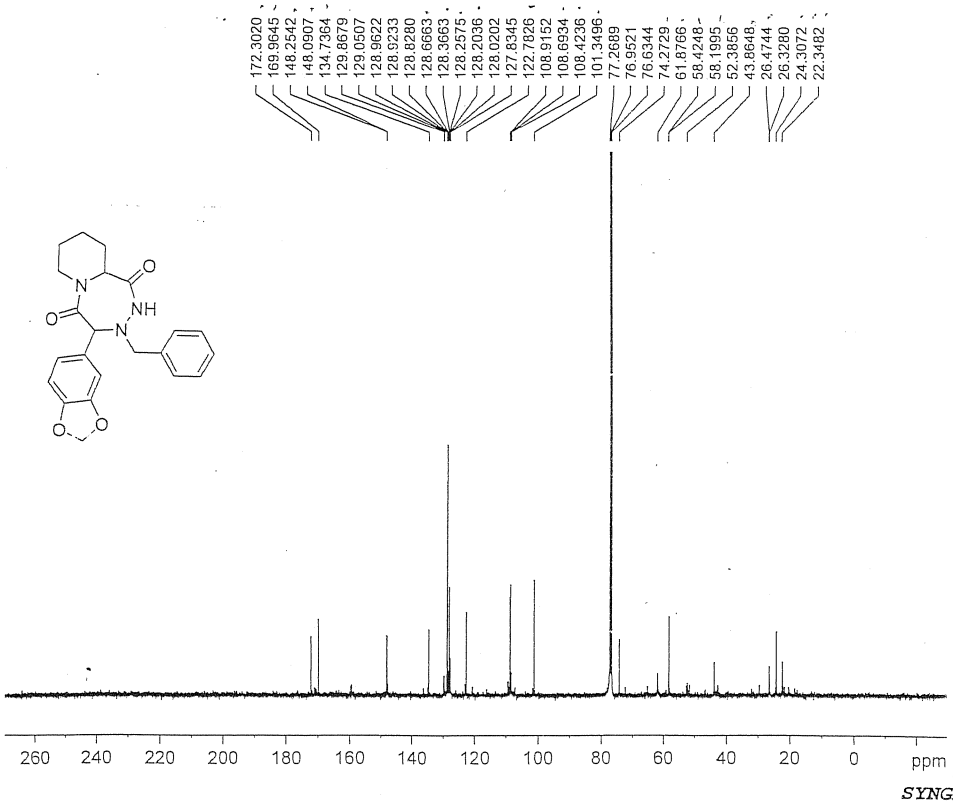
SC/AD/01-004

C-1502-04-9204344A



Current Data Parameters

NAME	C-1502-04-9204344A
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090912
Time	3.25
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	10000
DS	2
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	0.5439988 sec
RG	812.7
DW	16.600 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.30 usec
PL1	-1.00 dB
SFO1	100.6499905 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	15.00 dB
PL13	15.00 dB
SFO2	400.2316009 MHz

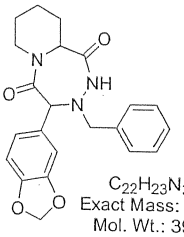
F2 - Processing parameters

SI	32768
SF	100.6379211 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

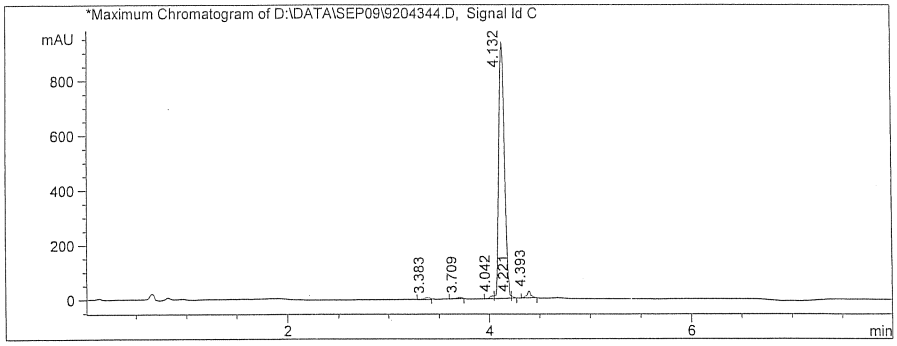
SYNGENE INTERNATIONAL LTD.
SC/AD/01-002

Data file : D:\DATA\SEP09\9204344.D
Vial No. : P1-D-08
Injection Date : 9/10/2009 1:43:41 PM
Injection vol : 2ul
Sample Name : C-1502-04
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

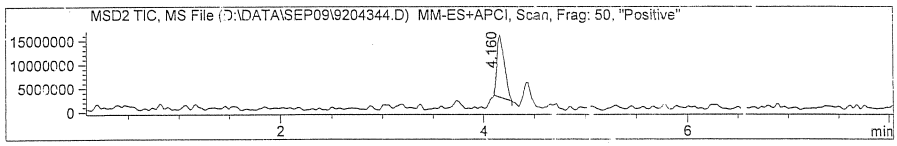
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{22}H_{23}N_3O_4$
Exact Mass: 393.17
Mol. Wt.: 393.44

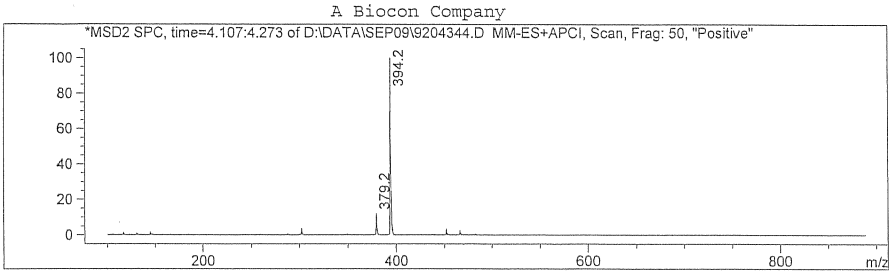


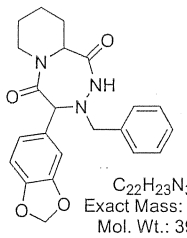
Peak No	RT min	Area	Area %
1	3.383	3.216e+001	0.818
2	3.709	1.759e+001	0.447
3	4.042	2.856e+001	0.727
4	4.132	3.780e+003	96.158
5	4.221	8.189e+000	0.208
6	4.393	6.450e+001	1.641



Analysed by :

Instrument Code : SC/AD/10-014





$C_{22}H_{23}N_3O_4$
Exact Mass: 393.17
Mol. Wt.: 393.44

Analysed by :

Instrument Code : SC/AD/10-014

Syngene International Ltd
A Biocon Company

HPLC REPORT

Data file :C:\CHEM32\1\DATA\SEP09\9204344B.D Vial No. : Vial 2

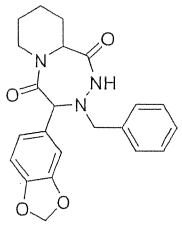
Injection Date :9/10/2009 3:04:19 PM Injection vol : 4 µl

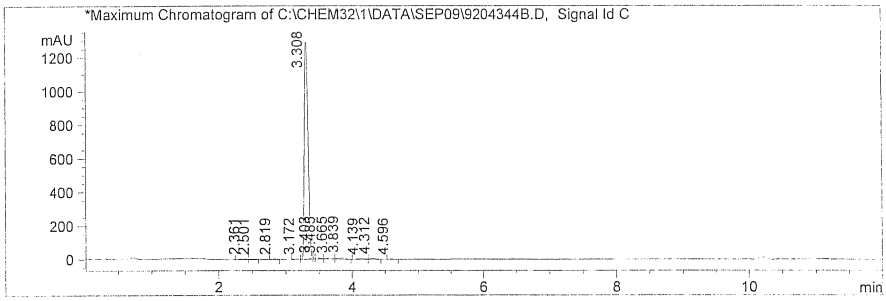
Sample Name :C-1502-04 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\S_AC73.M

Method info :A:10mM NH4OAc B:ACN
Hypersil BDS C18(4.6X50)mm,5um
Flow:0.8mL/min

Time	%B
0	30
4	100
8	100
9	30
12	30





Peak No	RT min	Area	Area %
1	2.361	16.821	0.318
2	2.501	8.448	0.160
3	2.819	10.167	0.192
4	3.172	5.380	0.102
5	3.308	5014.516	94.870
6	3.403	13.775	0.261
7	3.485	39.471	0.747
8	3.665	50.899	0.963
9	3.839	83.386	1.578
10	4.139	10.555	0.200
11	4.312	26.238	0.496
12	4.596	6.031	0.114

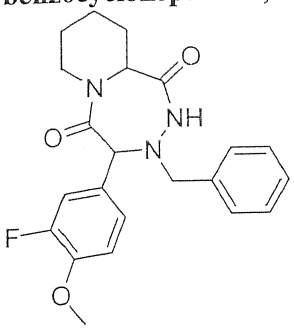
End of report

Analysed By :

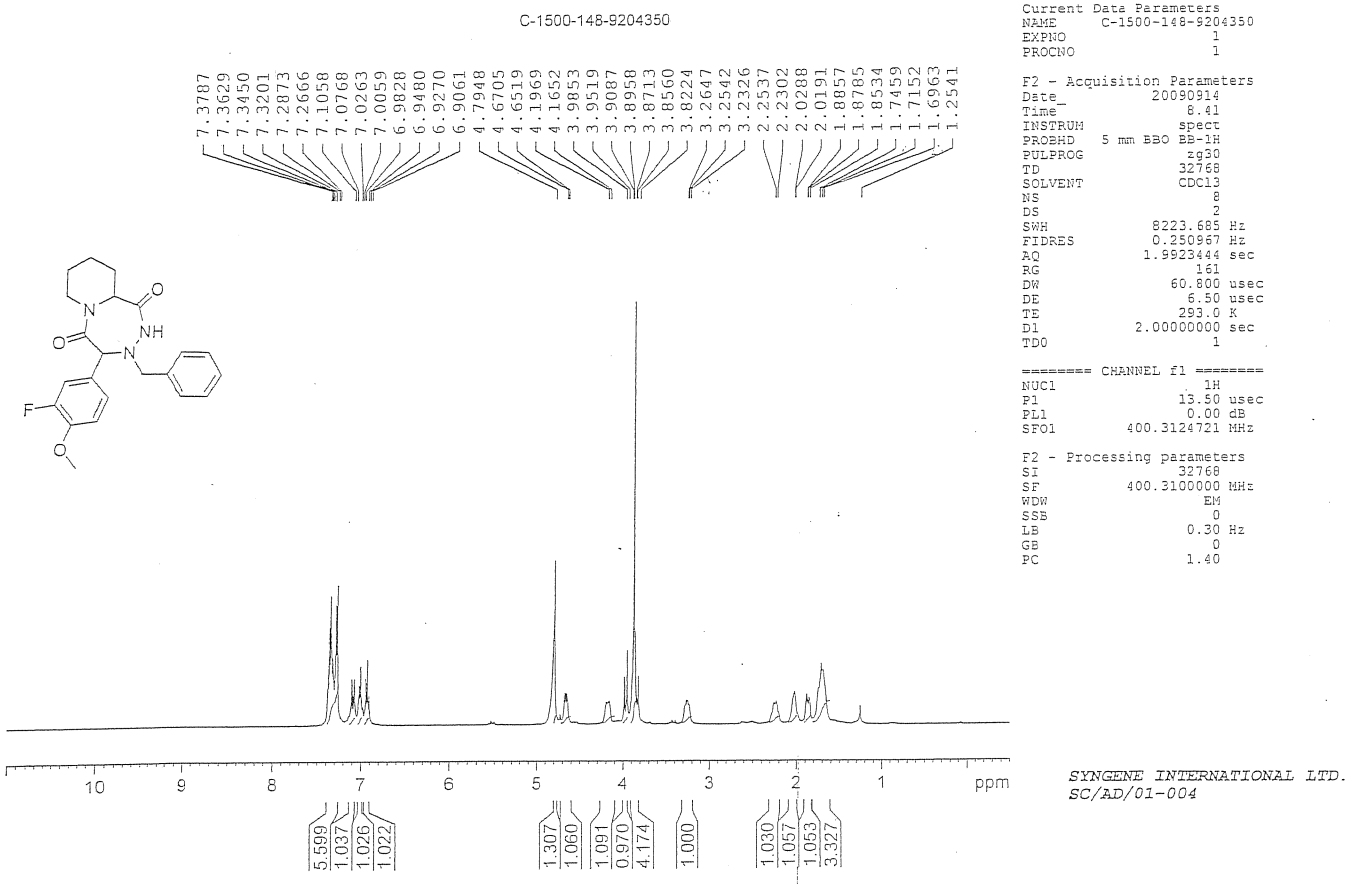
Checked By :

Instrument Code : SC/AD/04-064

XXV. Table 3, 6d [7-Benzyl-6-(3-fluoro-4-methoxy-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]



White solid; m.p: (Met-Temp): 80°-81°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.69-1.75 (m, 3H), 1.85-1.89 (m, 1H), 2.02-2.03 (m, 1H), 2.23-2.25 (m, 1H), 3.23-3.26 (m, 1H), 3.82-3.91 (m, 4H), 3.95-3.99 (m, 1H), 4.17-4.19 (m, 1H), 4.65-4.67 (m, 1H), 4.80 (s, 1H), 6.91-6.95 (m, 1H), 6.98-7.02 (m, 1H), 7.08-7.11 (m, 1H), 7.27-7.39 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): 22.35, 24.32, 26.41, 43.97, 56.18, 58.23, 61.84, 73.73, 113.62, 116.42, 116.61, 125.07, 126.95, 128.32, 128.85, 128.95, 134.63, 148.04, 148.15, 151.10, 153.57, 169.65, 172.27; LCMS (UV): 398.2 ($M+H^+$). Anal. Calcd. for $C_{22}H_{24}FN_3O_3$: C, 66.48; H, 6.09; N, 10.57. Found: C, 66.59; H, 6.15; N, 10.55.



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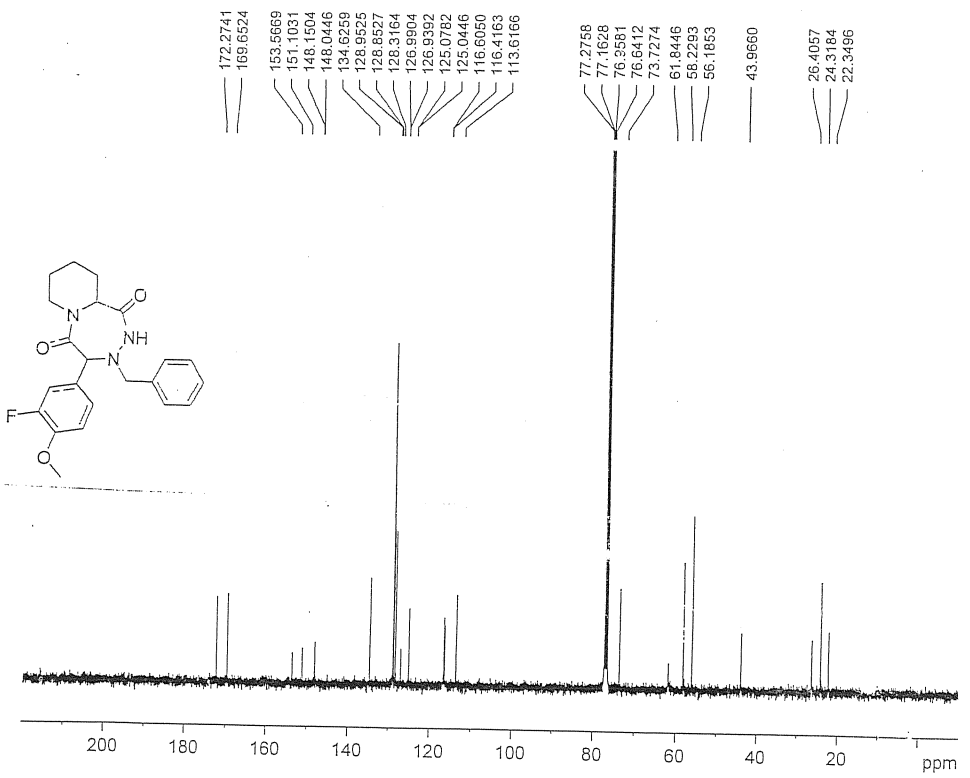
SC/AD/01-004

C-1500-148-9204350



Current Data Parameters

NAME	C-1500-148-9204350
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090912
Time	18.29
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1300
DS	2
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	0.5439988 sec
RG	645.1
DW	16.600 usec
DE	6.00 usec
TE	296.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.30 usec
PL1	-1.00 dB
SFO1	100.6499905 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	15.00 dB
PL13	15.00 dB
SFO2	400.2316009 MHz

F2 - Processing parameters

SI	32768
SF	100.6379211 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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SC/AD/01-002

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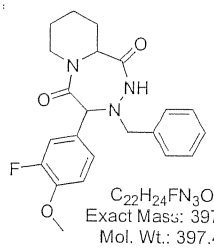
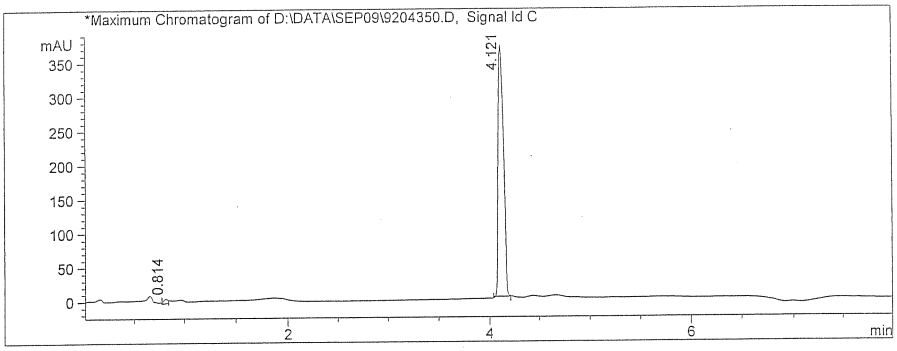
Data file : D:\DATA\SEP09\9204350.D
Vial No. : P1-E-03
Injection Date : 9/12/2009 5:15:37 PM
Injection vol : 2ul
Sample Name : C-1500-148
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

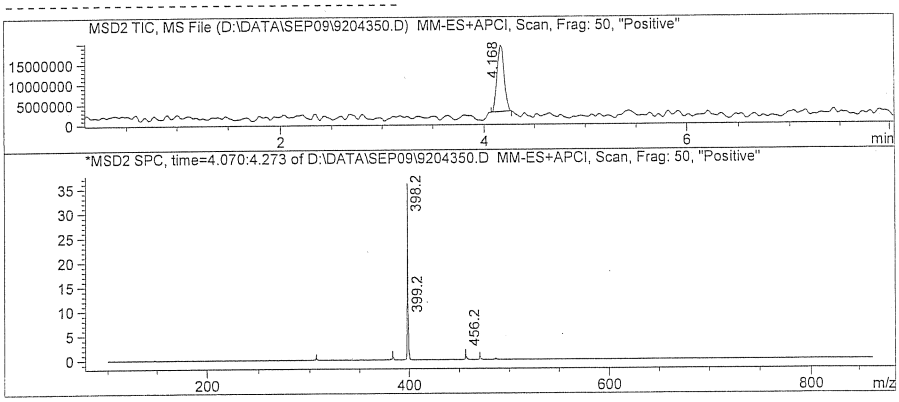
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.0 5.5 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{22}H_{24}FN_3O_3$
Exact Mass: 397.18
Mol. Wt.: 397.44



Peak No	RT min	Area	Area %
1	0.814	2.169e+001	1.560
2	4.121	1.369e+003	98.440



Analysed by :

Instrument Code : SC/AD/10-014

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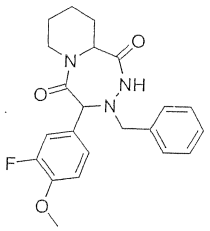
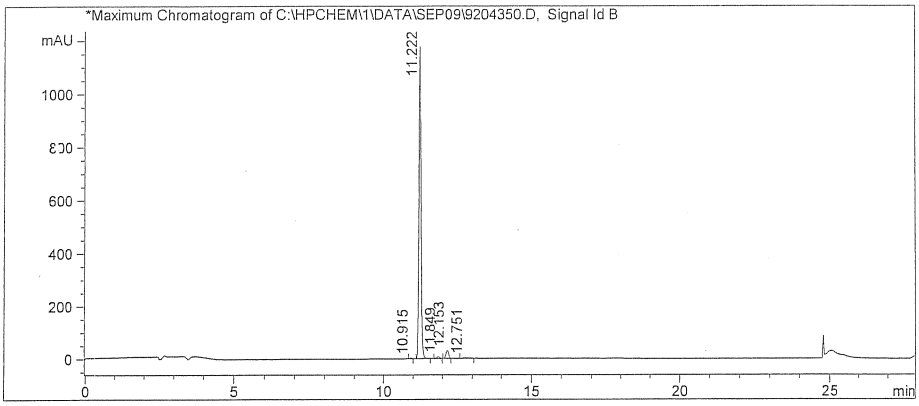
HPLC REPORT

Data file	:C:\HPCHEM\1\DATA\SEP09\9204350.D	Vial location:	Vial 15
Injection Date	:14/Sep/2009 9:33:05 AM	Injection vol :	2 µl
Sample Name	: C-1500-148	Operator :	VINA
Sample info	:	Acq Method :	SY_A7030.M

Method info : Mobile Phase: A:10mM NH4OAc B: ACN
Column:Symmetry C18(4.6X250)mm,5µ SC/LC/1062
Flow: 0.8mL/min

TIME	%B
0	30
15	100
20	100
23	30
28	30





Peak No	RT min	Area	Area %
1	10.915	8.944	0.15
2	11.222	5728.248	95.74
3	11.849	40.757	0.68
4	12.153	167.482	2.80
5	12.751	37.506	0.63

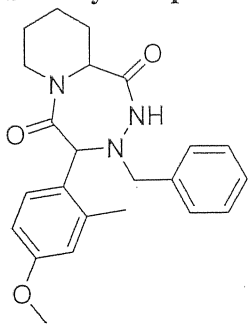
End of report

Analysed By :

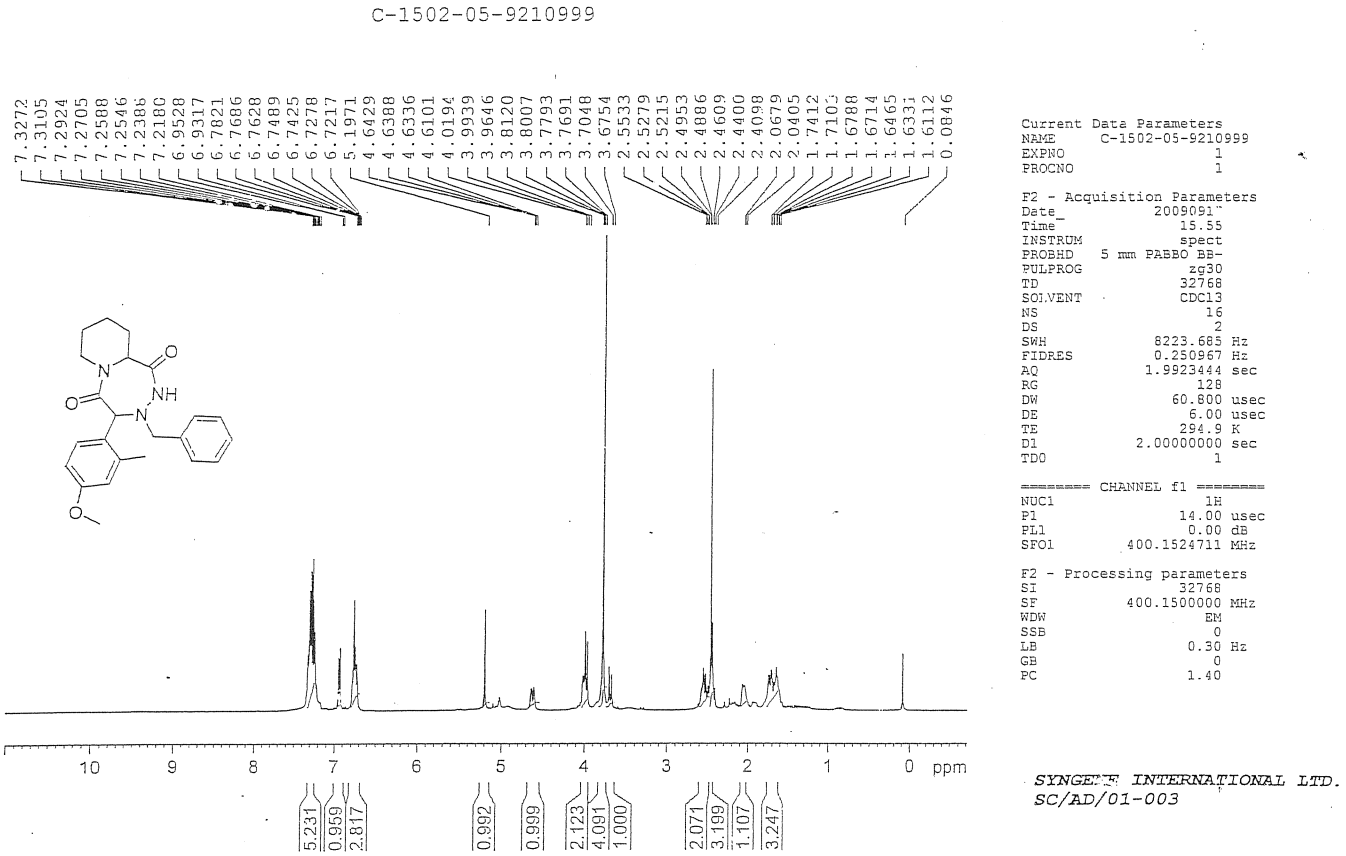
Checked By :

Instrument Code : SC/AD/04-15

XXVI. Table 3, 6e [7-Benzyl-6-(4-methoxy-2-methyl-phenyl)-hexahydro-4a,7,8-triaza-benzocycloheptene-5,9-dione]



White solid; m.p (Met-Temp): 60°-61°C (uncorrected); ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 1.61-1.74 (m, 3H), 2.04-2.06 (m, 1H), 2.46 (s, 3H), 2.49-2.55 (m, 2H), 3.68-3.3.70 (s, 1H), 3.77-3.80 (m, 4H), 3.96-4.02 (m, 2H), 4.61-4.62 (m, 1H), 5.19 (s, 1H), 6.72-6.78 (m, 3H), 6.93-6.95 (m, 1H), 7.22-7.33 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz): 20.05, 24.39, 24.54, 31.73, 42.42, 53.99, 55.19, 59.07, 62.46, 111.86, 116.55, 127.85, 128.45, 128.54, 128.70, 128.89, 136.85, 139.30, 159.24, 163.39, 165.86, 170.85, 173.18; LCMS (UV): 394.2 ($M+H^+$). Anal. Calcd. for $C_{23}H_{27}N_3O_3$: C, 70.21; H, 6.92; N, 10.68. Found: C, 70.30; H, 6.89; N, 10.66.

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Current Data Parameters
NAME      C-1502-05-9210999
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              2009091
Time                 15.55
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.250967 Hz
AQ               1.9923444 sec
RG                     128
DW                60.800 usec
DE                 6.00 usec
TE                 294.9 K
D1            2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                   32768
SF           400.1500000 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                  1.40
```

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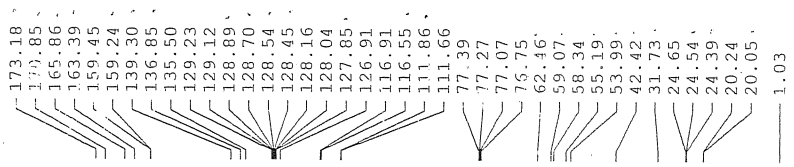
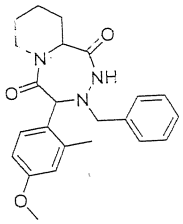
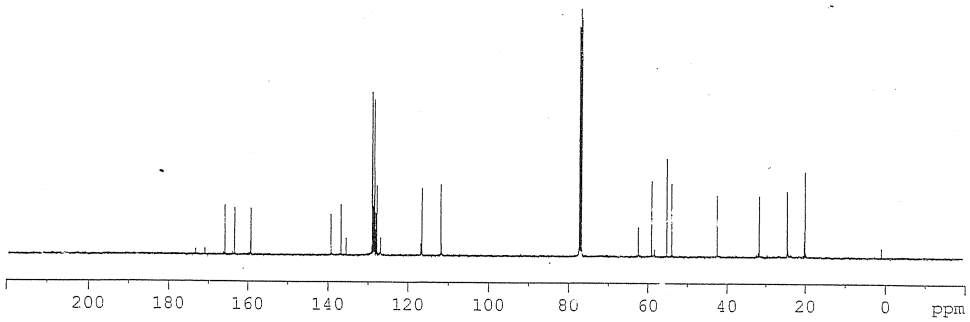
SC/AD/01-003

C-1502-05-9210999







Current Data Parameters

NAME	C-1502-05-9210999
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20090917
Time	21.15
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPRG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1000
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	0.6816244 sec
RG	80.6
DW	20.800 usec
DE	6.00 usec
TE	295.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	7.13 usec
PL1	-3.00 dB
SFO1	100.6278593 MHz

======== CHANNEL f2 ========

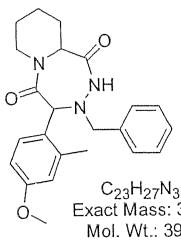
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.14 dB
PL13	15.00 dB
PL2	0.00 dB
SFO2	400.1516006 MHz

F2 - Processing parameters

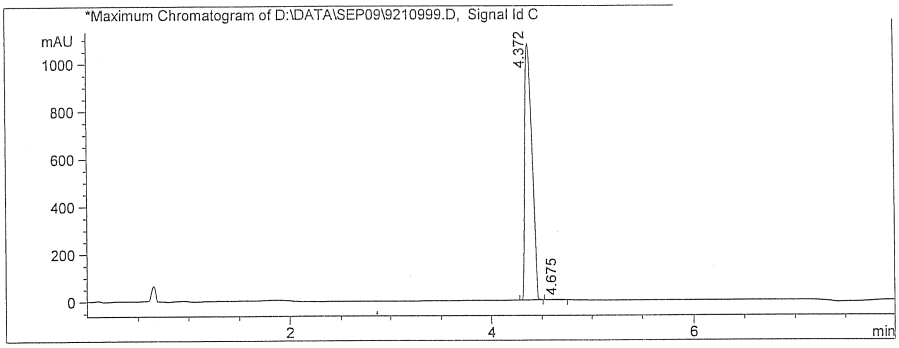
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Data file : D:\DATA\SEP09\9210999.D
Vial No. : P2-A-06
Injection Date : 9/16/2009 7:48:27 PM
Injection vol : 2ul
Sample Name : C-1502-05
Acq Method : C:\CHEM32\1\METHODS\EP7030FM.M

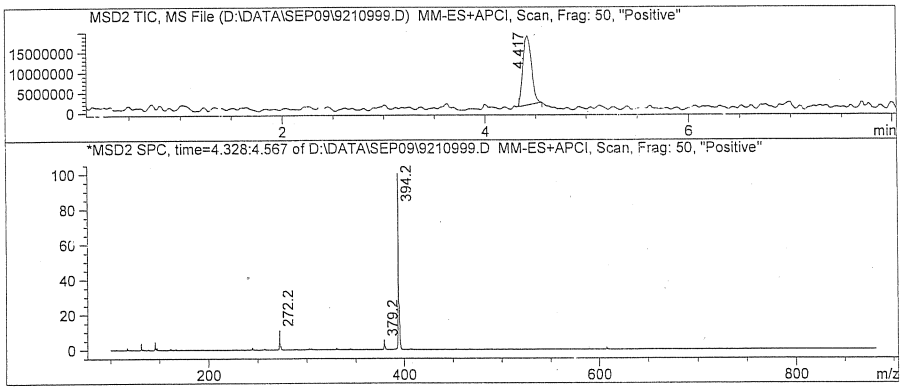
Method info :Column-:Eclipse Plus C18(50X4.6)mm,5µm
MOBILE PHASE::A : 0.1%HC00H B: MeOH
Flow = 0.8 mL/min
Time (min.): 0 3.0 5.5 6.0 8
% B : 30 95 95 30 30
MS-SCAN, ESI\APCI: DUAL POLARITY



$C_{23}H_{27}N_3O_3$
Exact Mass: 393.21
Mol. Wt.: 393.48



Peak No	RT min	Area	Area %
1	4.372	5.358e+003	99.662
2	4.675	1.815e+001	0.338



Analysed by :

Instrument Code : SC/AD/10-014

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HPLC REPORT

Data file :C:\CHEM32\1\DATA\SEP09\9210994A.D Vial No. : Vial 32

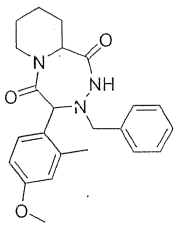
Injection Date :9/15/2009 3:45:10 PM Injection vol : 1 µl

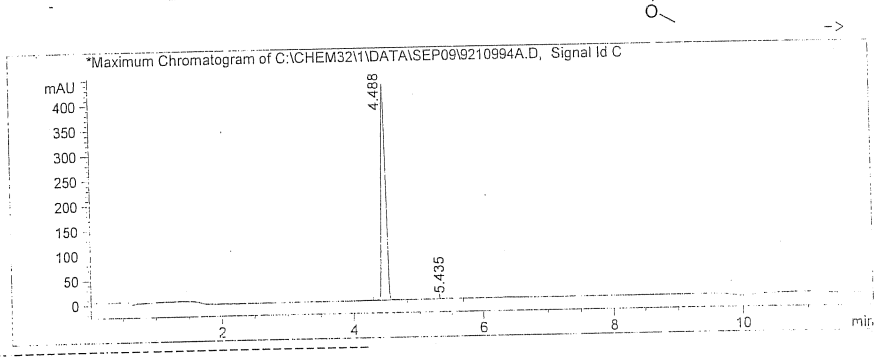
Sample Name :C-1502-05 Operator : HEMA

Sample info : Acq Method : C:\CHEM32\1\METHODS\ZE_AC73.M

Method info :A:10mM NH4OAc B:ACN
Zorbax Extend C18(4.6X50)mm,5um
Flow:0.8mL/min

Time	%B
0	30
4	90
8	90
9	30
12	30





Peak No	RT min	Area	Area %
1	4.488	1995.281	99.508
2	5.435	9.871	0.492

End of report

Analysed By :

Instrument Code : SC/AD/04-064

Checked By :