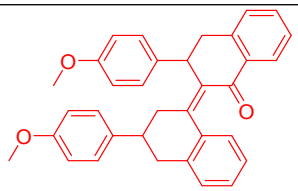
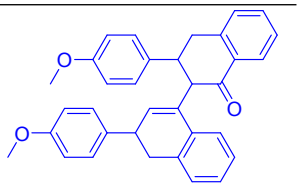
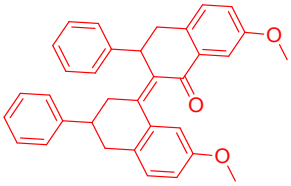
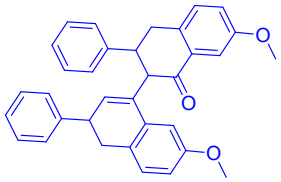
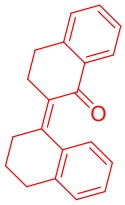
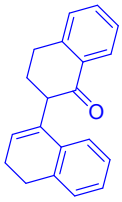
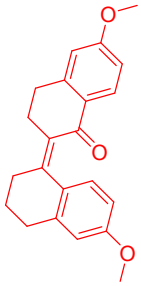
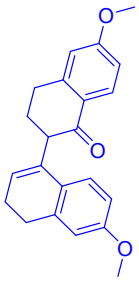
SUPPORTING DATA

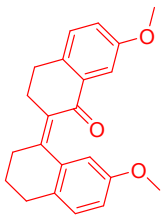
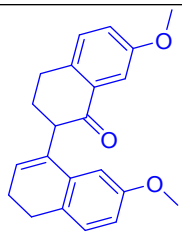
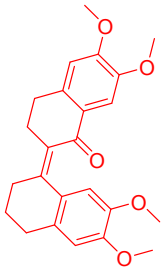
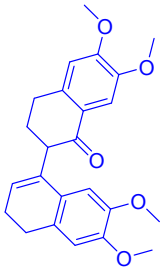
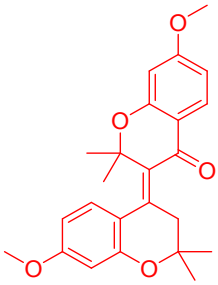
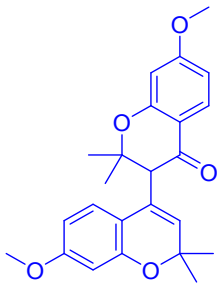
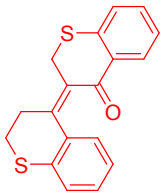
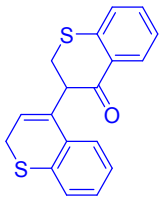
Boron tribromide mediated C-C bond formation in cyclic ketones: a transition metal free approach

Imran Ahmad,[a] Vinay Pathak,[a] Prema G. Vasudev,[b] Hardesh K. Maurya[a] and Atul Gupta[a,*]

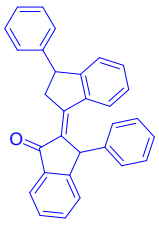
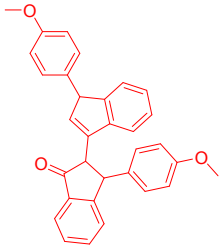
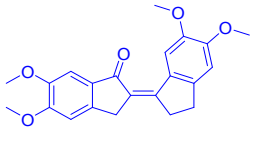
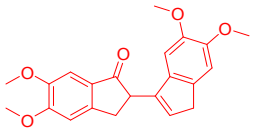
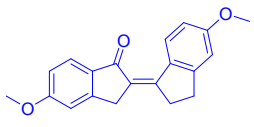
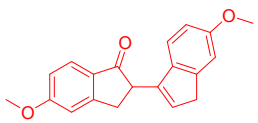
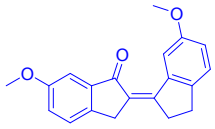
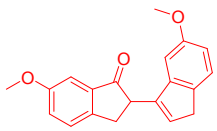
[a] Medicinal Chemistry Department, Central Institute of Medicinal and aromatic Plants, PO. CIMAP, Kukrail Road, Lucknow-226015, India ,Tel.: 0915222718556, E-mail: atul_gupta04@yahoo.co.in
[b] Metabolic & Structural Biology Department, Central Institute of Medicinal and Aromatic Plants, P.O. CIMAP, Kukrail Road, Lucknow-226015, India

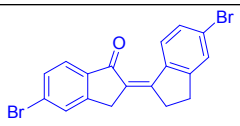
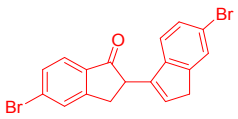
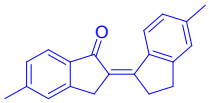
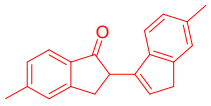
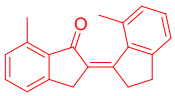
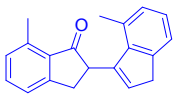
Table S1: Comparison of MM2 steric energy for a given frame for exo and endo product as calculated chemBio3Dultra 11.0 (Blue Color showing formed products and red color other possible product).

S.N.	Structure of exo product	MM2 steric energy for exo product (in kcal/mol)		MM2 steric energy for endo product (in kcal/mol)	Structure of endo product
1		11458.593	>	192.163	 **10a**
2		1372.279	>	254.792	 **10b** continued…..
3		496.389	>	189.254	 **10c**
4		344.529	>	207.737	 **10d**

4		391.343	>	207.609	 **10e**
5		400.689	>	233.975	 **10f**
6		375.424	>	196.832	 **10g**
7		2282.903	>	781.173	 **10h**

continued…..

8	 **12a**	161.822	>	163.457	
9	 **12b**	129.917	<	186.478	
10	 **12c**	88.998	<	134.157	
11	 **12d**	88.434	<	134.308	

12	 **12e**	71.390	<	116.218	
13	 **12f**	71.052	<	116.102	
14		623.649	>	341.255	 **17**

Procedure for calculating MM2 steric energy: i) Draw the structures as shown in supplementary Table S1. ii) Copy the structure and past it in ChemBio3D Ultra 11.0. iii) Open calculations form menu. iv) Select MM2. v) Select compute properties. vii) Select properties. viii) Select Steric energy detail. ix) click run. x) get the total energy from outbox as shown in Table S1.

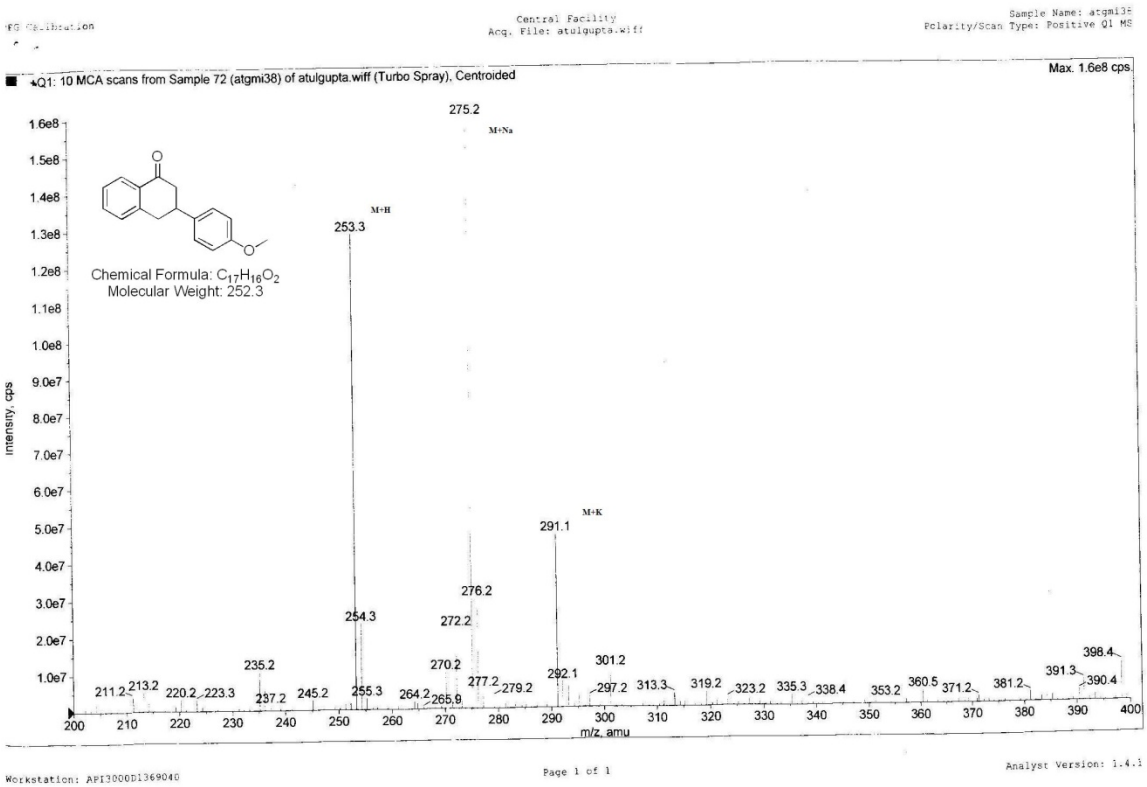
Table S2: C-H...O, C-H...π and π-π interaction in the crystal structure of **21**.[a]

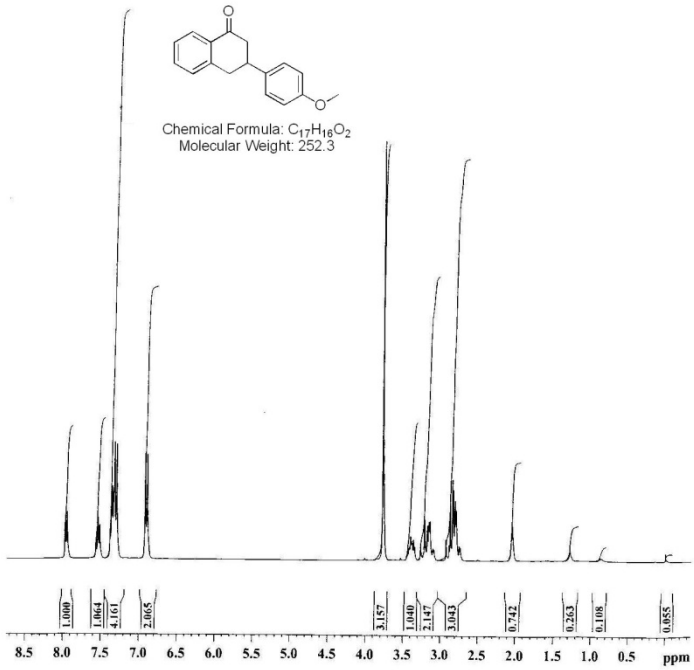
Type				**Symmetry relation**
C-H...O	**D… A (Å)**	**H…A (Å)**	**C-H…A (deg)**	
C8-H...O2’	3.40	2.49	158.0	(x, y, z+1)
C6’-H...O1	3.41	2.54	150.7	-x+1,-y+2,-z+1
C-H...π				
C-H…Cg (ring I)	3.734	2.840	147.0	-x+2,-y+1,-z+1
π…π	**Cg…Cg**	**C…C (R_{clo})**	**Interplanar angle**	
Intramolecular				
III-IV	5.53	4.19 (C15-C16’)	67.2	
II-III	5.77	3.78 (C11-C10’)	47.9	
Intermolecular				
I-II (A)	5.31	3.93 (C5-C5’)	70.0	-x+1, -y+1, -z+1
I-III (B)	4.84	3.66 (C7-C13)	85.8	-x+1, -y+1, -z+1
II-III (C)	5.22	3.72 (C8’-C16)	48.2	-x+1, -y+1, -z+1
I-I (D)	5.37	3.83 (C9-C9)	0.2	-x+2, -y+1, -z+1
I-III (E)	5.86	3.82 (C8-C12)	85.8	-x+2, -y+1, -z+1
III-IV (F)	5.53	3.66 (C15-C14’)	67.2	-x+2, -y+1, -z
II-II (G)	4.03	3.32 (C7’-C7’)	1.3	-x+1, -y+2, -z+1
IV-IV (H)	5.23	3.43(C16’-C16’)	0.0	-x+2, -y+2, -z
IV-II (I)	5.64	3.71 (C14’-C5’)	73.6	-x+1, -y+2, -z
IV-I (J)	5.79	3.58 (C6-C13’)	26.4	x, y+1, z-1

Aromatic rings labelled I-IV as shown in Figure 1b. Cg = centre of gravity. Intermolecular π...π interactions are labelled A – J for clarity in discussion.

Copy of Spectra:

3-(4-methoxyphenyl)-3,4-dihydronaphthalen-1(2*H*)-one (8a):





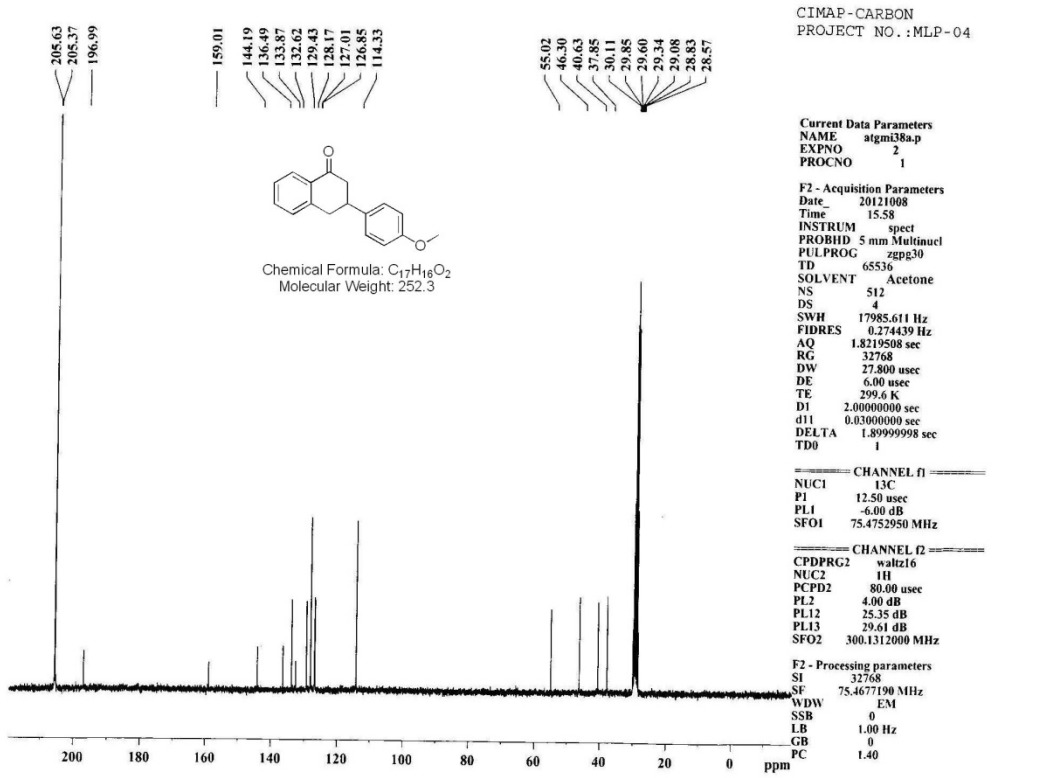
CIMAP PROTON
PROJECT NO.: mlp-02

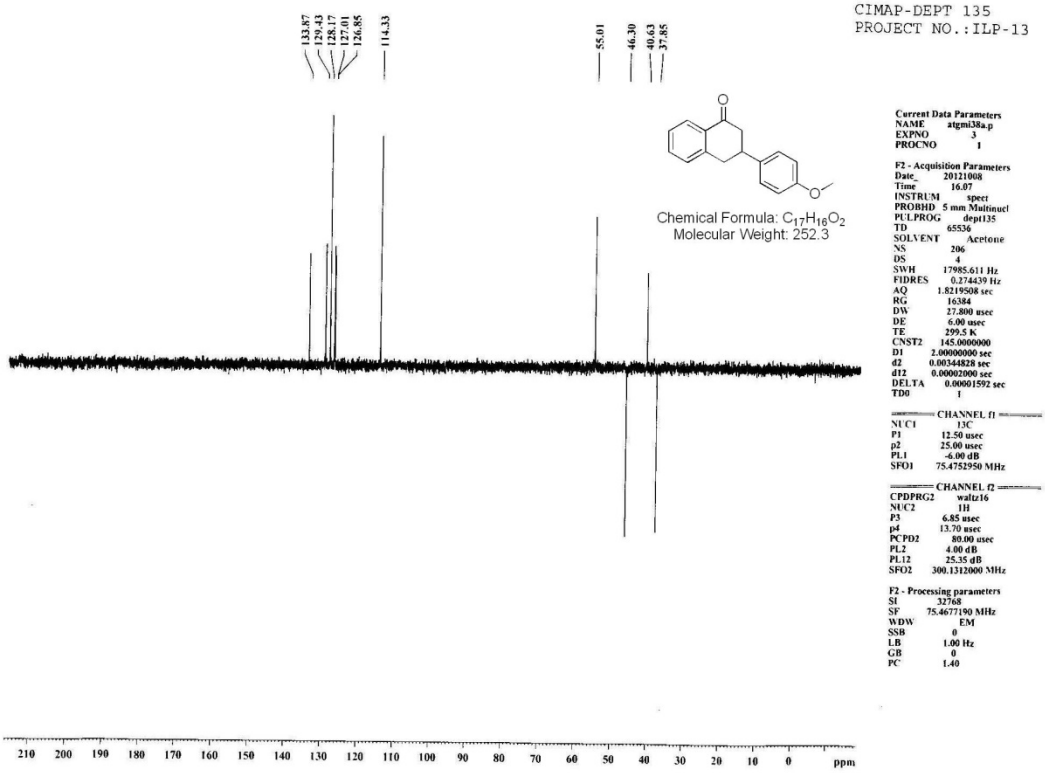
Current Data Parameters
NAME atgmi38a.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121008
Time 15.24
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

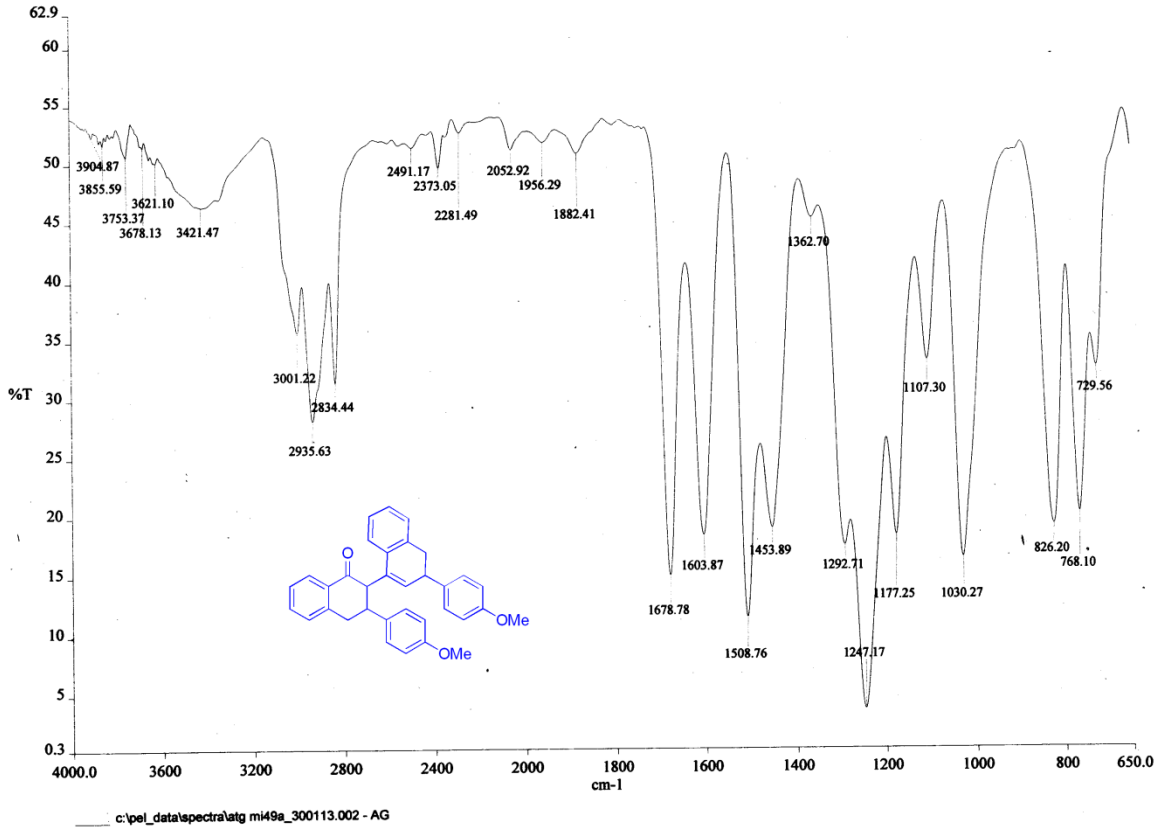
CHANNEL f1
NUC1 1H
P1 6.85 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

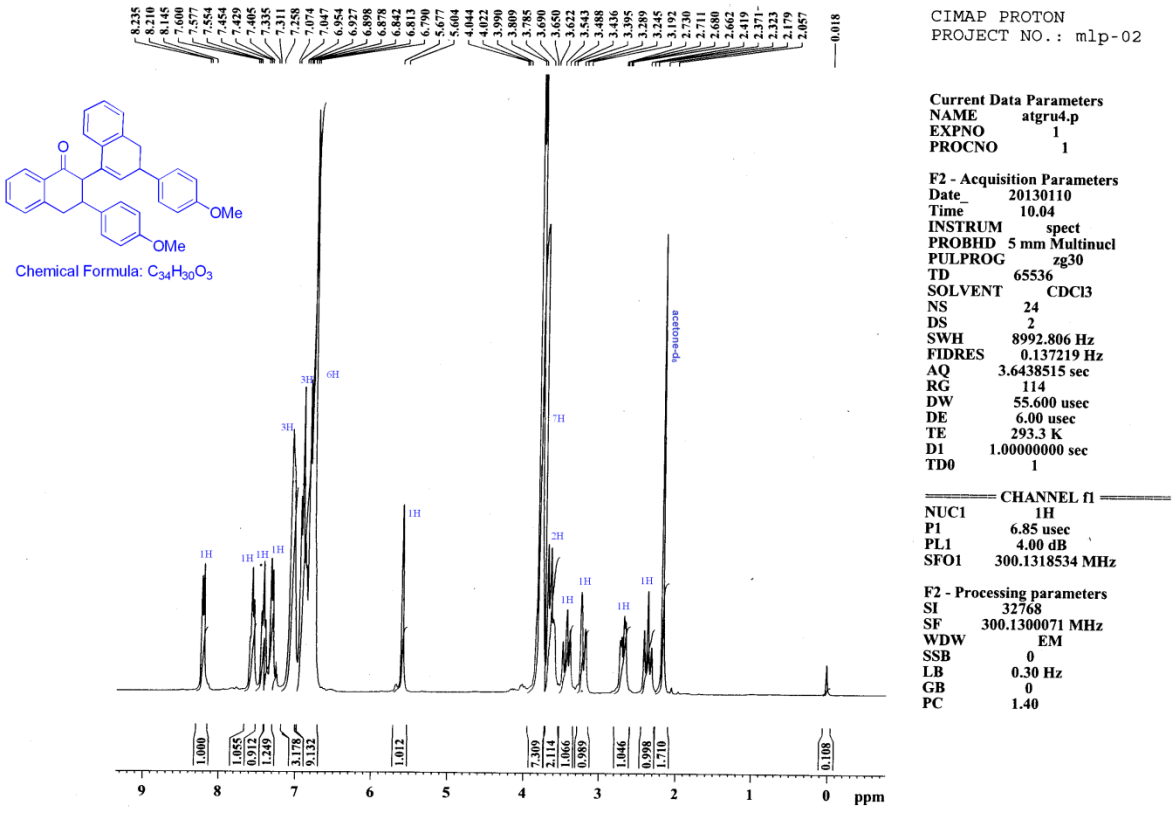
F2 - Processing parameters
SI 32768
SF 300.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40





3,3'-bis(4-methoxyphenyl)-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'*H*)-one (10a):





CIMAP PROTON
PROJECT NO.: mlp-02

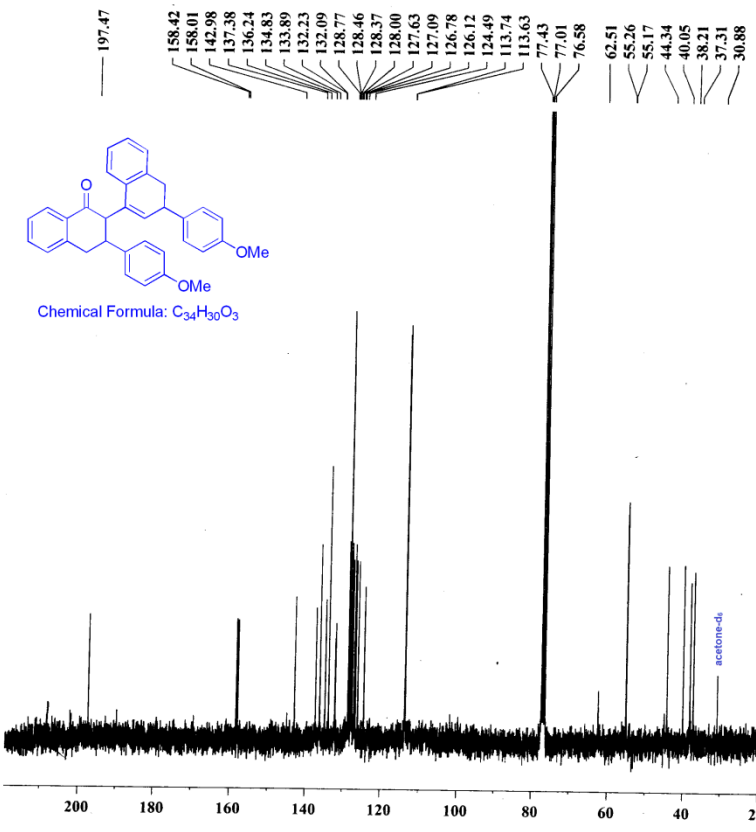
Current Data Parameters
NAME atgru4.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130110
Time 10.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 24
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 114
DW 55.600 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 6.85 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300071 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

CIMAP-CARBON
PROJECT NO.:MLP-04



Current Data Parameters
NAME atgru4.p
EXPNO 2
PROCNO 1

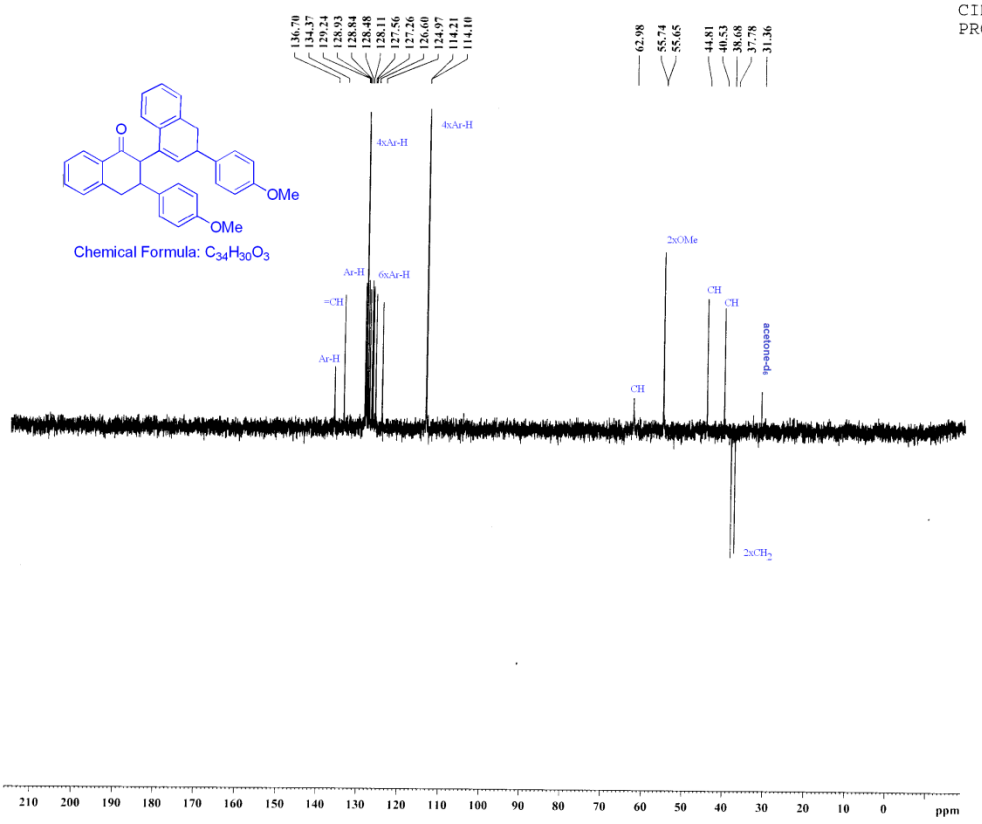
F2 - Acquisition Parameters
Date_ 20130110
Time 10.26
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 514
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.35 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CIMAP-DEPT 135
PROJECT NO.:ILP-13



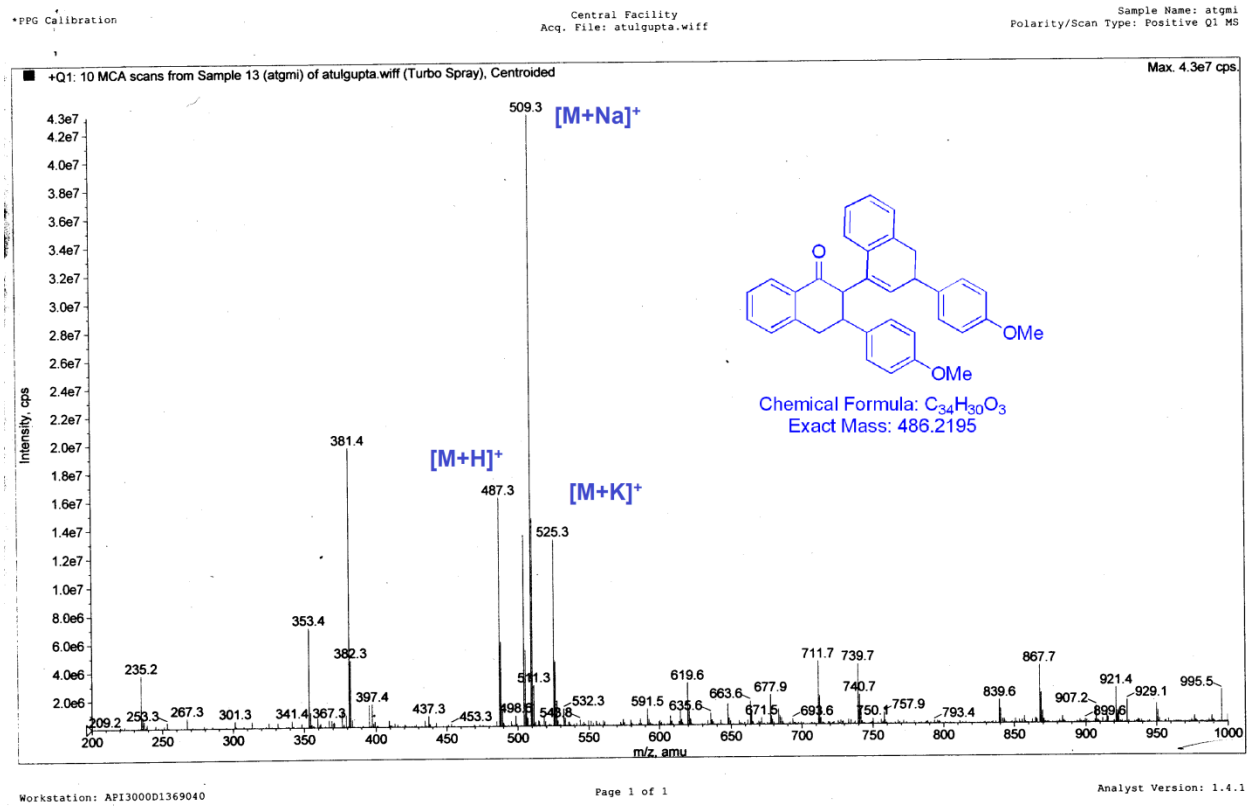
Current Data Parameters
NAME atgru4.p
EXPNO 3
PROCNO 1

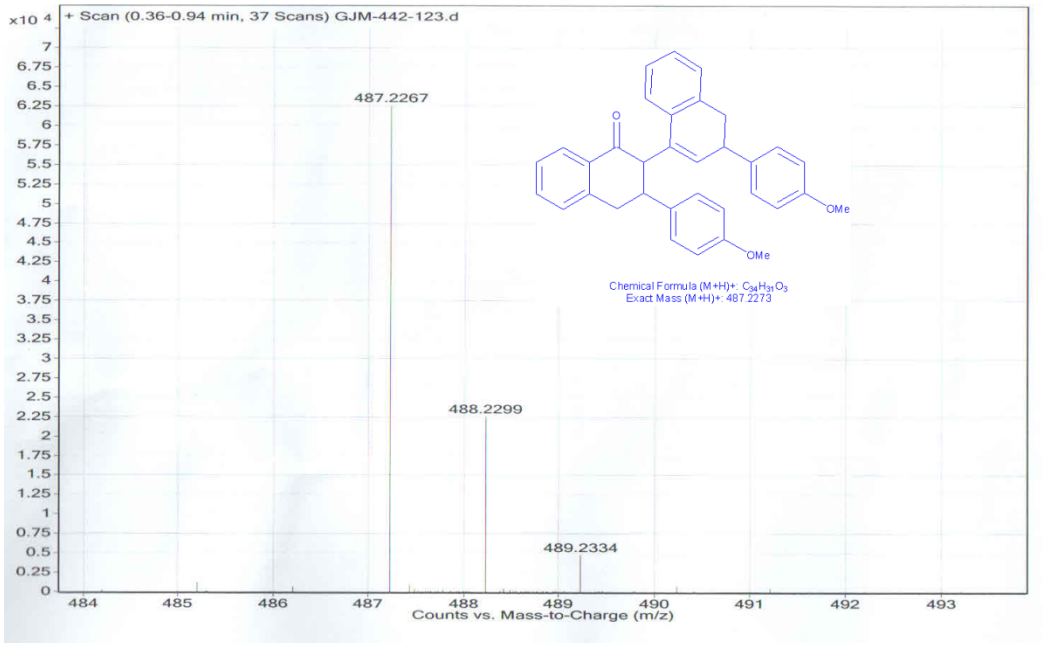
F2 - Acquisition Parameters
Date_ 20130110
Time 10.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 294.4 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001592 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
p2 25.00 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

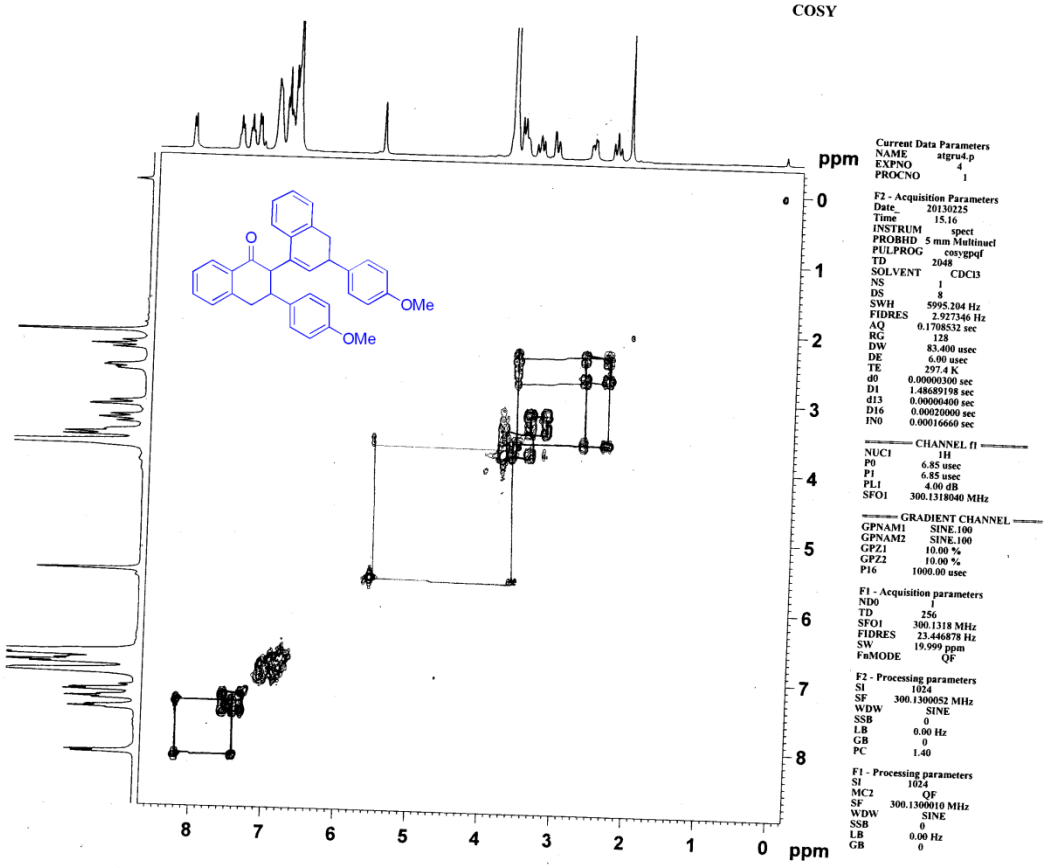
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.85 usec
p4 13.70 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.35 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


*PPG Calibration
Central Facility
Acq. File: atulgupta.wiff
Sample Name: atgmi
Polarity/Scan Type: Positive Q1 MS
+Q1: 10 MCA scans from Sample 13 (atgmi) of atulgupta.wiff (Turbo Spray), Centroided
Max. 4.3e7 cps.
509.3
[M+Na]+
[M+H]+
[M+K]+
381.4
487.3
525.3
Chemical Formula: C34H30O3
Exact Mass: 486.2195
Intensity, cps
m/z, amu
Workstation: API3000D1369040
Page 1 of 1
Analyst Version: 1.4.1


+ Scan (0.36-0.94 min, 37 Scans) GJM-442-123.d
487.2267
488.2299
489.2334
Chemical Formula (M+H)+: C34H31O3
Exact Mass (M+H)+: 487.2273
Counts vs. Mass-to-Charge (m/z)

COSY



Current Data Parameters
NAME atgru4.p
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130225
Time 15.16
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 1
DS 8
SWH 5995.204 Hz
FIDRES 2.927346 Hz
AQ 0.1708532 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 297.4 K
d0 0.00000300 sec
D1 1.48689198 sec
d13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00016660 sec

CHANNEL f1
NUC1 1H
P0 6.85 usec
P1 6.85 usec
PL1 4.00 dB
SFO1 300.1318040 MHz

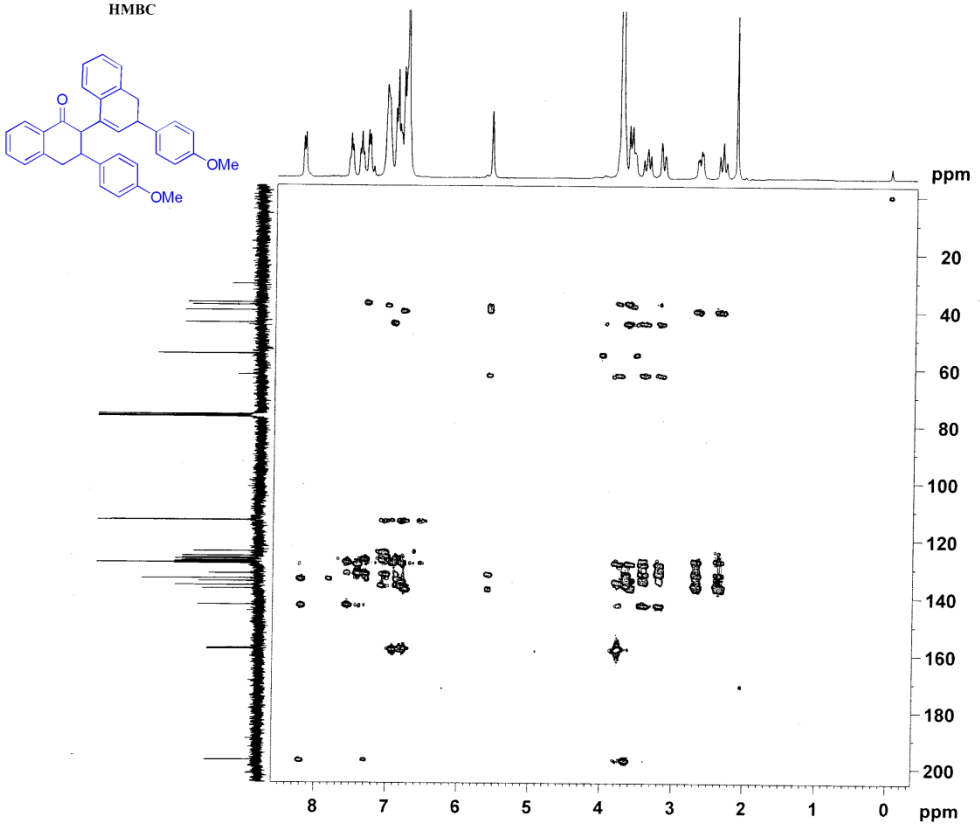
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 1
TD 256
SFO1 300.1318 MHz
FIDRES 23.446878 Hz
SW 19.999 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 300.1300052 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1300010 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

HMBC



Current Data Parameters
NAME atgru4.p
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130225
Time 15.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG hmbcgpl2ndqf
TD 4096
SOLVENT CDCl3
NS 8
DS 16
SWH 5995.204 Hz
FIDRES 1.463673 Hz
AQ 0.3416564 sec
RG 16384
DW 83.400 usec
DE 6.00 usec
TE 298.9 K
CNST6 135.0000000
CNST7 165.0000000
CNST13 7.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d6 0.07142857 sec
D16 0.00020000 sec
DELTA1 0.00250370 sec
DELTA2 0.00183030 sec
DELTA3 0.07022458 sec
IN0 0.00002980 sec

CHANNEL f1
NUC1 1H
P1 6.85 usec
p2 13.70 usec
PL1 4.00 dB
SFO1 300.1318040 MHz

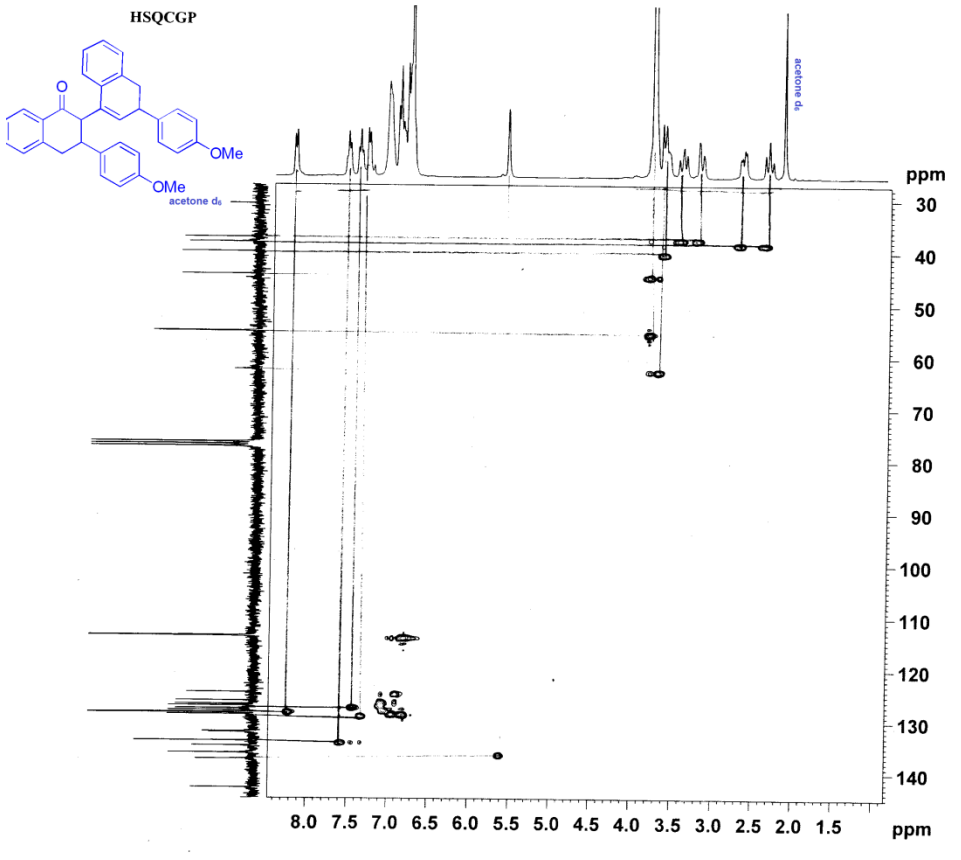
CHANNEL f2
NUC2 13C
P3 12.50 usec
PL2 -6.00 dB
SFO2 75.4752830 MHz

GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPNAM5 SINE.100
GPNAM6 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %
GPZ5 -10.00 %
GPZ6 -5.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 245
SFO1 75.47528 MHz
FIDRES 68.483772 Hz
SW 222.305 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 300.1300095 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 75.4677476 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0



Current Data Parameters
NAME atgru4.p
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130225
Time 15.24
INSTRUM spect
PROBHD 5 mm Multinuci
PULPROG hsqcetgpsi2
TD 1024
SOLVENT DMSO
NS 2
DS 16
SWH 5995.204 Hz
FIDRES 5.854691 Hz
AQ 0.0854516 sec
RG 18390.4
DW 83.400 usec
DE 6.00 usec
TE 297.6 K
CNST2 145.0000000
d0 0.00003300 sec
D1 1.50000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000400 sec
D16 0.00020000 sec
D24 0.00086207 sec
DELTA 0.00126970 sec
DELTA1 0.00120800 sec
DELTA2 0.00006207 sec
DELTA3 0.00052414 sec
IN0 0.00004000 sec
STICNT 128
ZGOPTNS

CHANNEL f1
NUC1 1H
P1 6.85 usec
p2 13.70 usec
P28 1000.00 usec
PL1 4.00 dB
SFO1 300.1318040 MHz

CHANNEL f2
CPDPRG2 garp
NUC2 13C
P3 12.50 usec
p4 25.00 usec
PCPD2 70.00 usec
PL2 -6.00 dB
PL12 8.00 dB
SFO2 75.4734070 MHz

GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.00 %
GPZ4 -5.00 %
P16 1000.00 usec
P19 600.00 usec

F1 - Acquisition parameters
ND0 2
TD 256
SFO1 75.4734 MHz
FIDRES 48.828125 Hz
SW 165.621 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 75.4677190 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

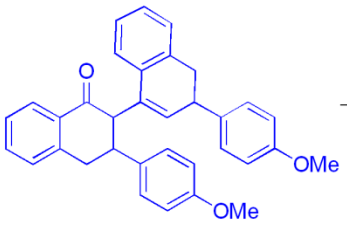
Mobile Phase: ACN;Water (60:40)

Column- Phenomenex 4.6 X 250mm 5 µm

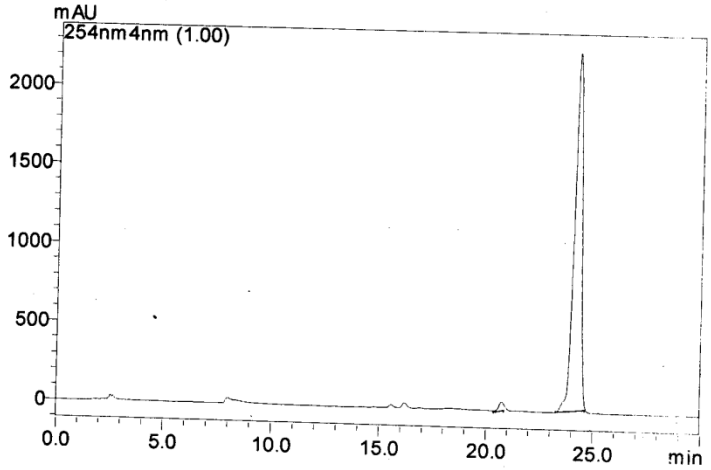
Flow Rate-1 ml-min.

Run Time-30 min.

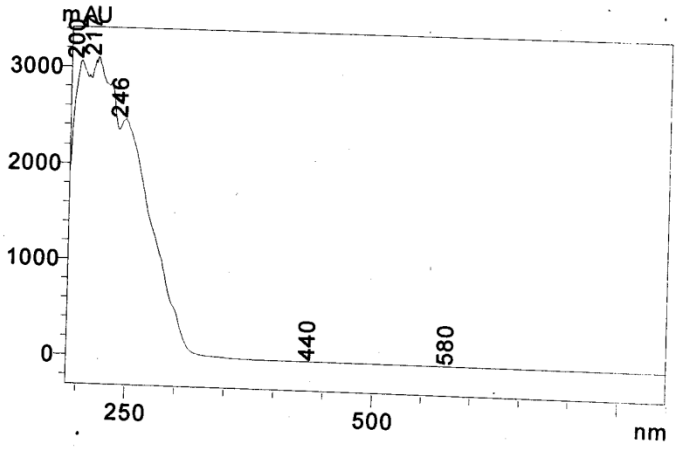
Wavelength-254 nm



HPLC Profile AYG-RI-04



UV Spectra

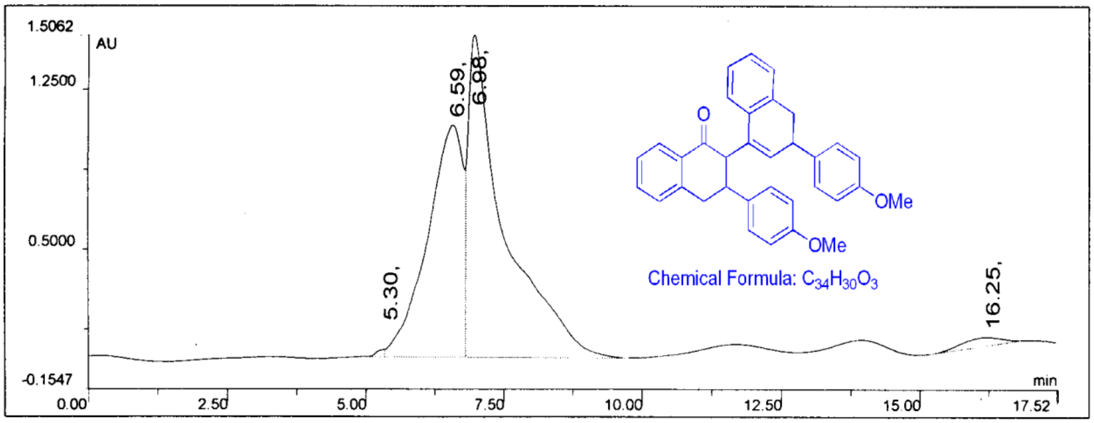


Sample Name : Sample1 **Detector :** UV-VIS – uv – 220

Time : 11:42:47 AM **System :** HPLC

Analysis Type : Percent Method **Run Date :** 8/30/2013

Peak Width	Peak Threshold	Area Reject	Height Reject
16	60	500	60



Area % Height %

Sr. No.	Component Name	Ret. Time.	Area μ volt sec	Height μ volt	Area %	Height %	ID	
1		5.30	139622.69	15708	0.23	1.18	U	S
2		6.59	[illegible]	544320	39.68	40.77	U	BV
3		6.98	[illegible]	756548	58.45	56.67	U	VB
4		16.25	994949.94	18497	1.64	1.39	U	BB
				1335074	**100.00**	**100.00**		

Summary

Total Peaks : 4 **Sample Amount** 100.0000

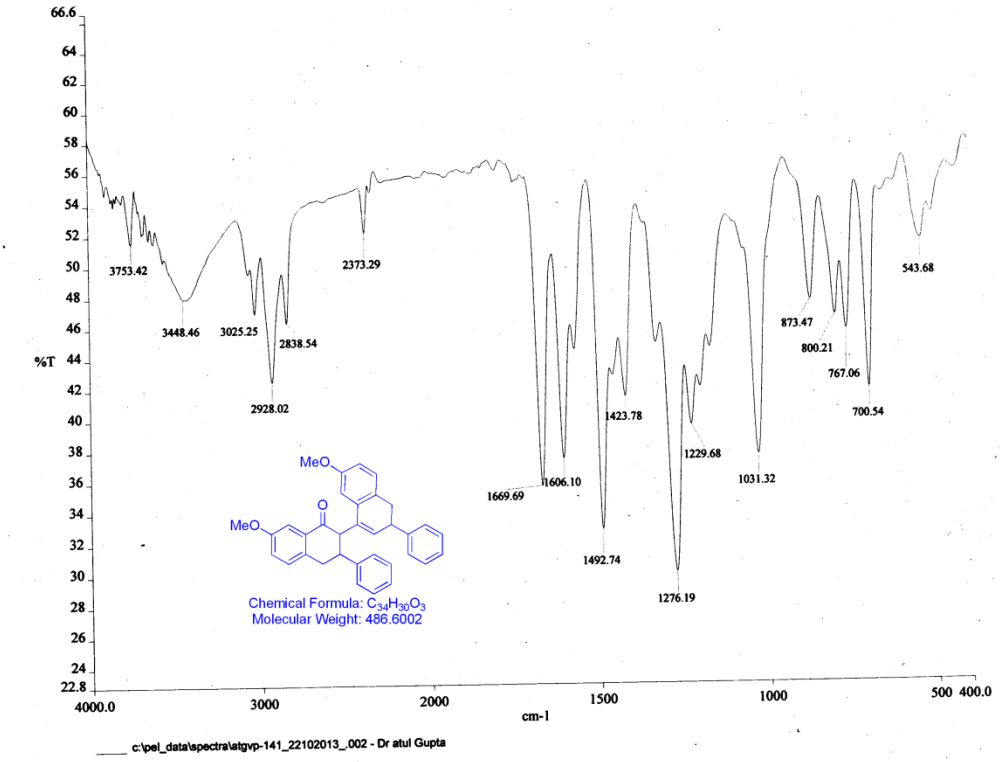
Multiplication 1.0000 **Syssuit** IP

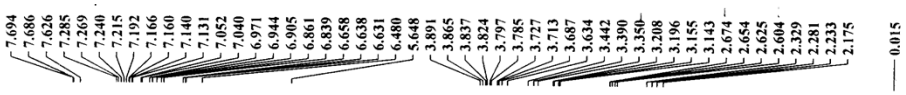
Dilution Factor : 1.0000 **ISTD Amount :** 0.0

Solvent system
MeOH/water in ~~90:10~~, flow rate is .5ml/m

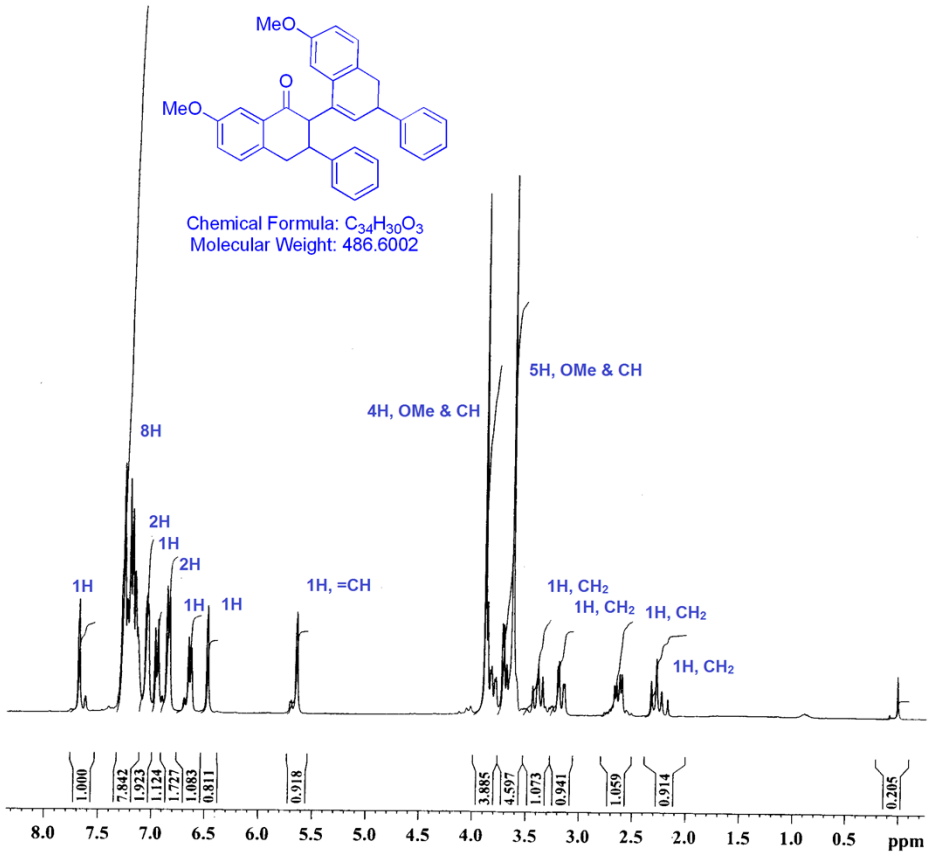
50 : 50

7,7'-dimethoxy-3,3'-diphenyl-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'H)-one (10b):





CIMAP PROTON
PROJECT NO.: mlp-02



Current Data Parameters

NAME	atgvp141.p
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20131007
Time	15.53
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	181
DW	81.000 usec
DE	6.00 usec
TE	300.4 K
D1	1.00000000 sec
TD0	1

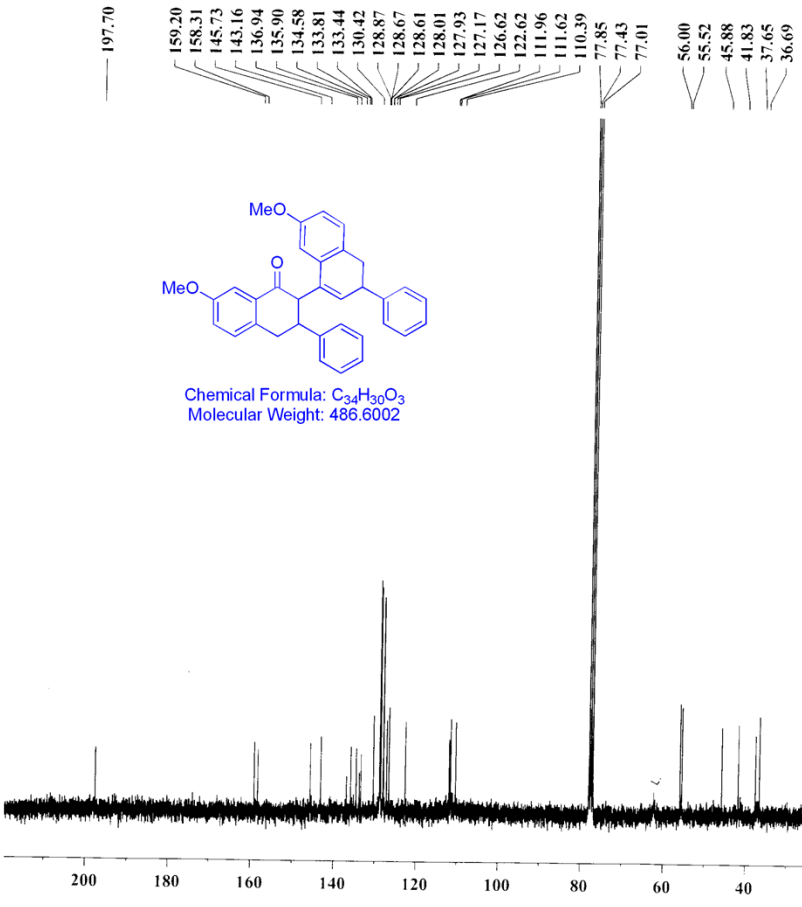
======== CHANNEL f1 ========

NUC1	1H
P1	6.78 usec
PL1	4.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300068 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

CIMAP-CARBON
PROJECT NO.:MLP-04



Current Data Parameters
NAME atgvp141.p
EXPNO 2
PROCNO 1

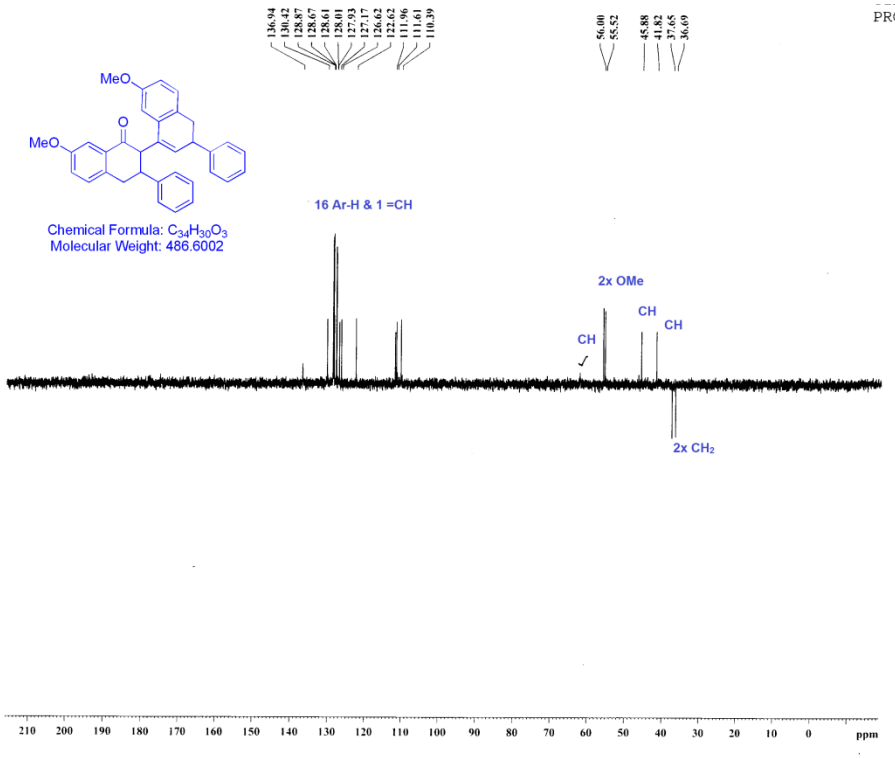
F2 - Acquisition Parameters
Date_ 20131007
Time 16.05
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 604
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 301.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

PROJECT NO.:ILP-13



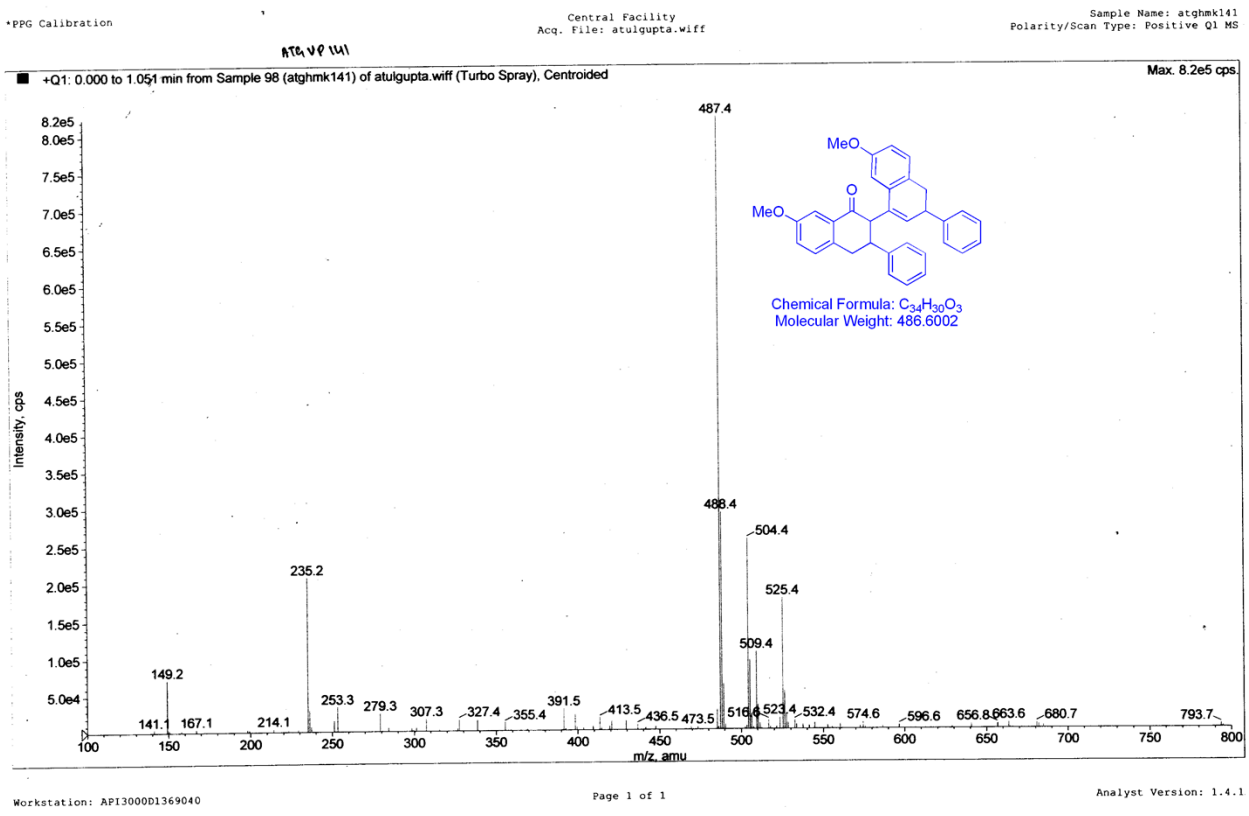
Current Data Parameters
NAME atgvp141.p
EXPNO 3
PROCNO 1

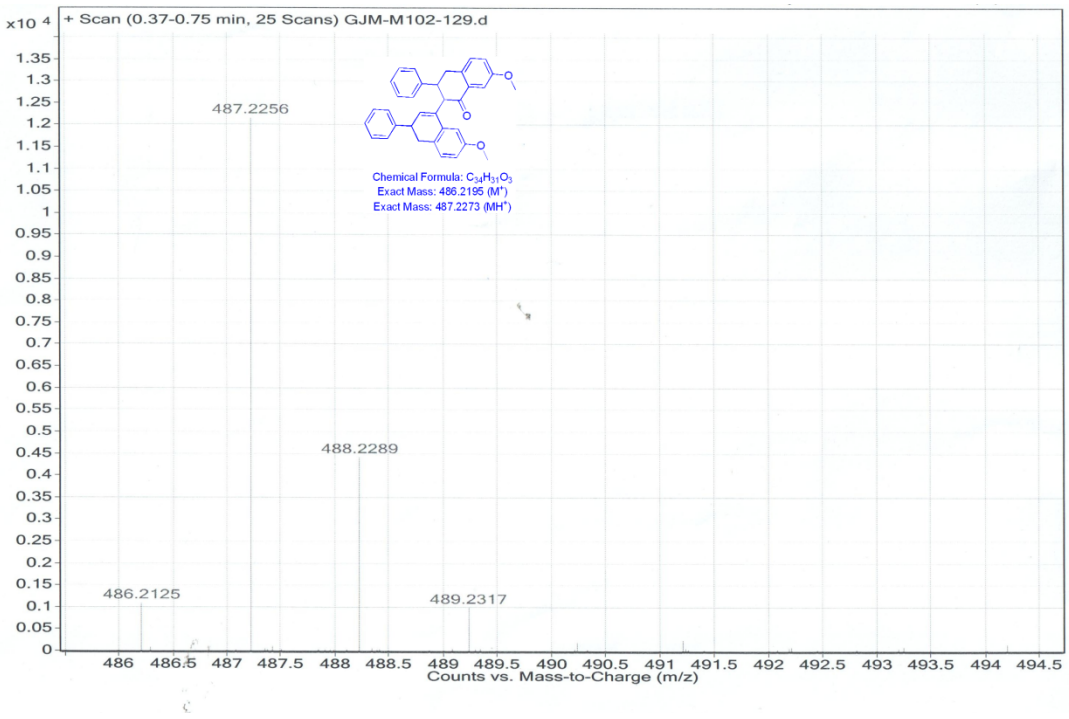
F2 - Acquisition Parameters
Date_ 20131007
Time 16.34
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 331
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 301.1 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

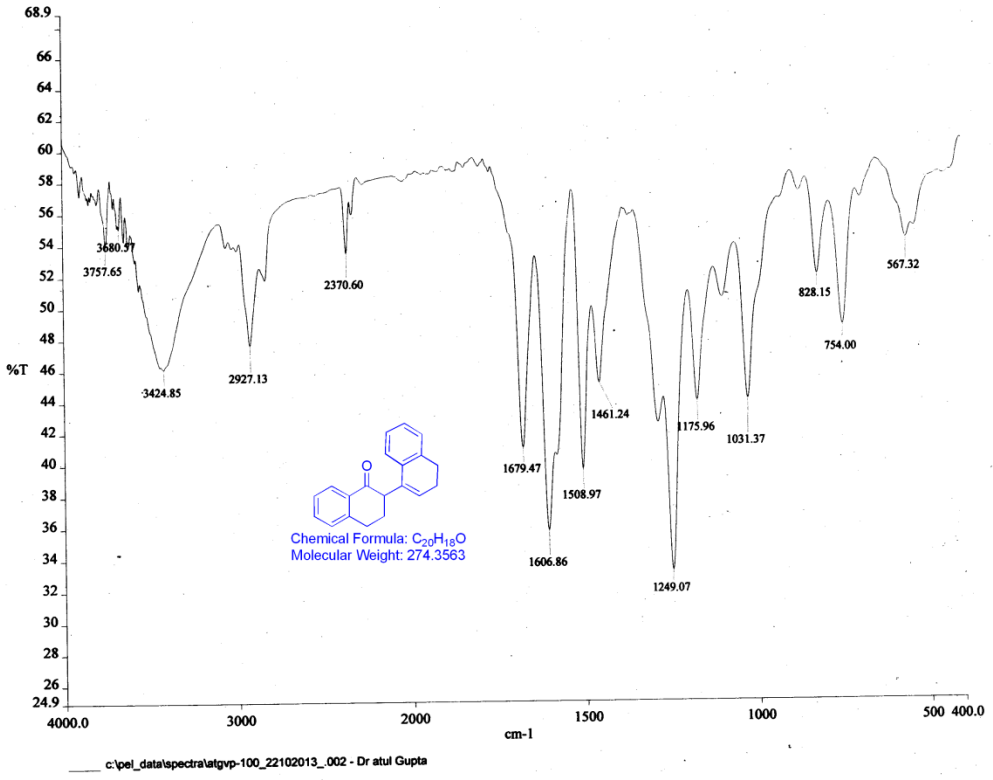
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

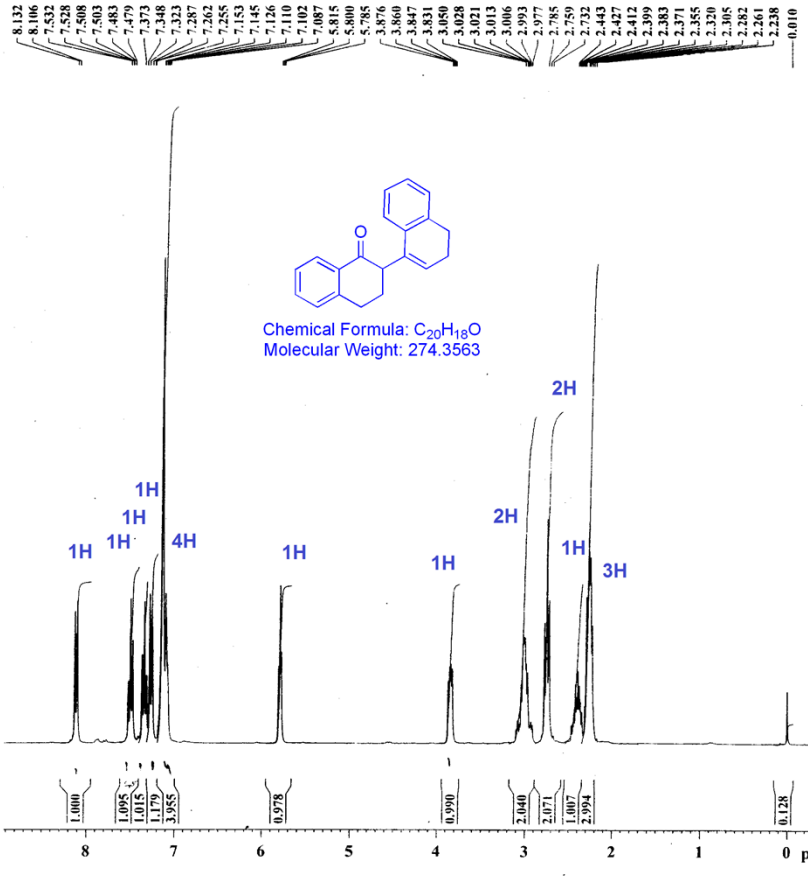
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'*H*)-one (10c):





CIMAP PROTON
PROJECT NO.: mlp-02

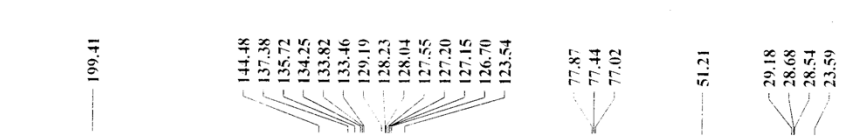
Current Data Parameters
NAME atgmi100.p
EXPNO 1
PROCNO 1

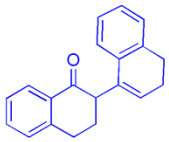
F2 - Acquisition Parameters
Date_ 20130917
Time 14.59
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

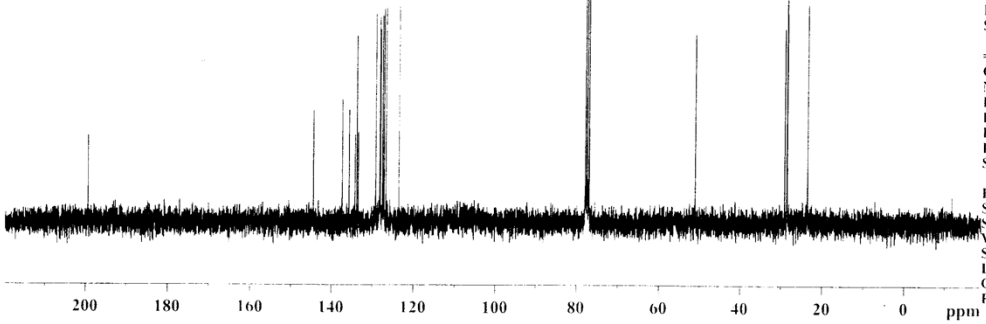
F2 - Processing parameters
SI 32768
SF 300.1300084 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

CIMAP-CARBON
PROJECT NO.:MLP-0





Chemical Formula: $C_{20}H_{18}O$
Molecular Weight: 274.3563



Current Data Parameters
NAME atgmi100.p
EXPNO 2
PROCNO 1

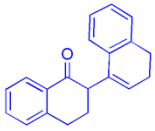
F2 - Acquisition Parameters
Date_ 20130917
Time 15.16
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ======
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

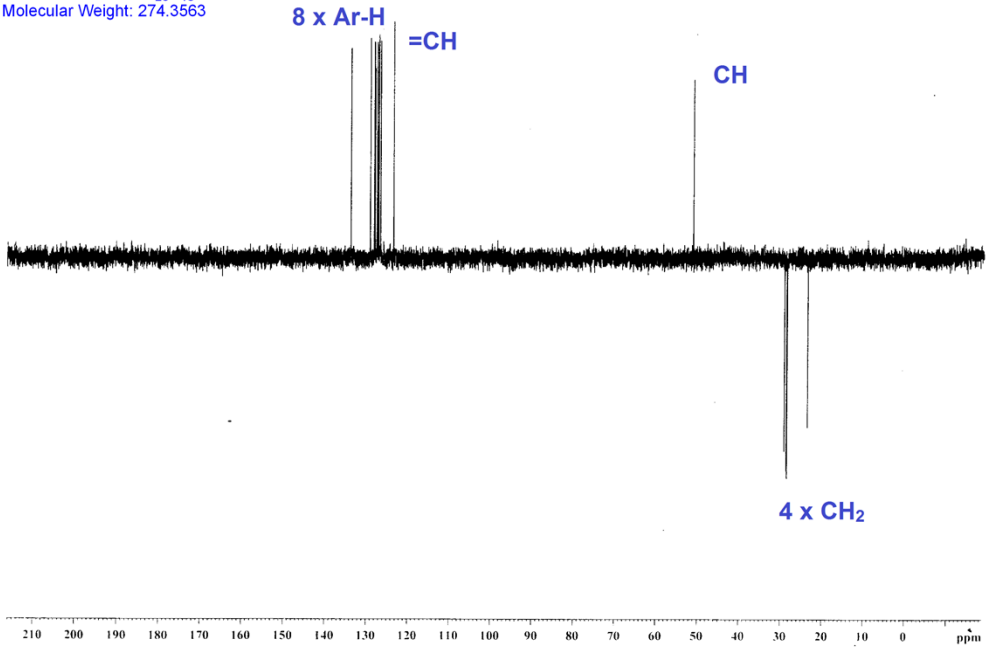
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CIMAP-DEPT 135
PROJECT NO.:ILP-13



Chemical Formula: $C_{20}H_{18}O$
Molecular Weight: 274.3563



Current Data Parameters
NAME atgmi100.p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130917
Time 15.20
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.6 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

======== CHANNEL f1 ======
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Central Institute of Medicinal and Aromatic Plants
(Analytical Chemistry Division)
Lucknow-226015

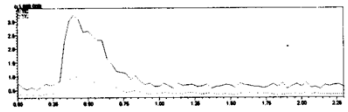
ANALYTICAL TEST REPORT

Sample Information for Direct Mass Analysis of Isolates/synthetic molecule

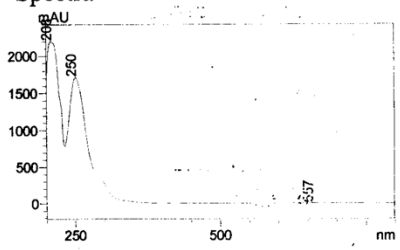
Sample Code : ATG-MI-100
Solubility : MeOH
Name of the Scientist : Dr ATUL GUPTA

Project Code: MLP-C
Mass Range: 200-40

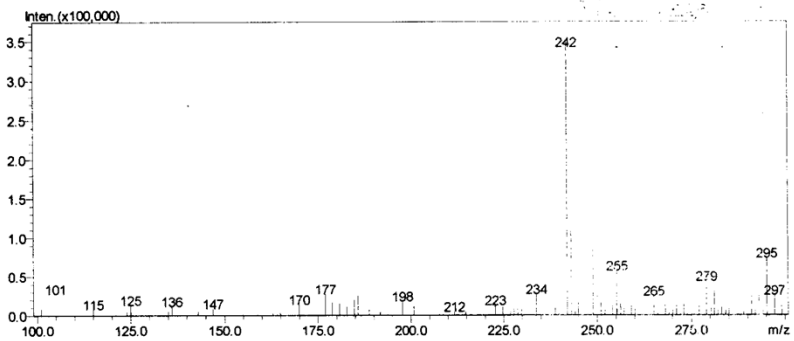
Mass chromatogram



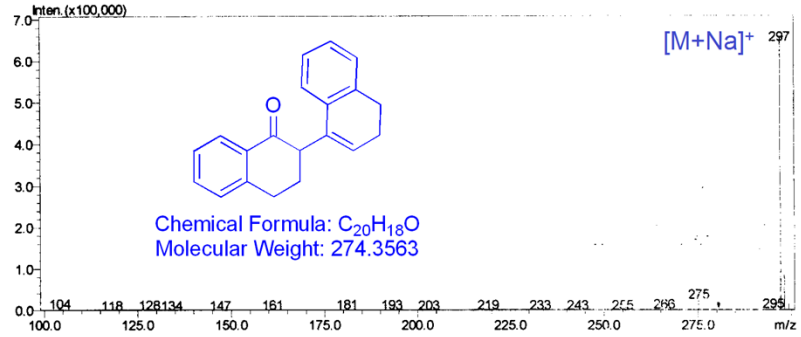
UV-Spectra



ESI-



ESI+

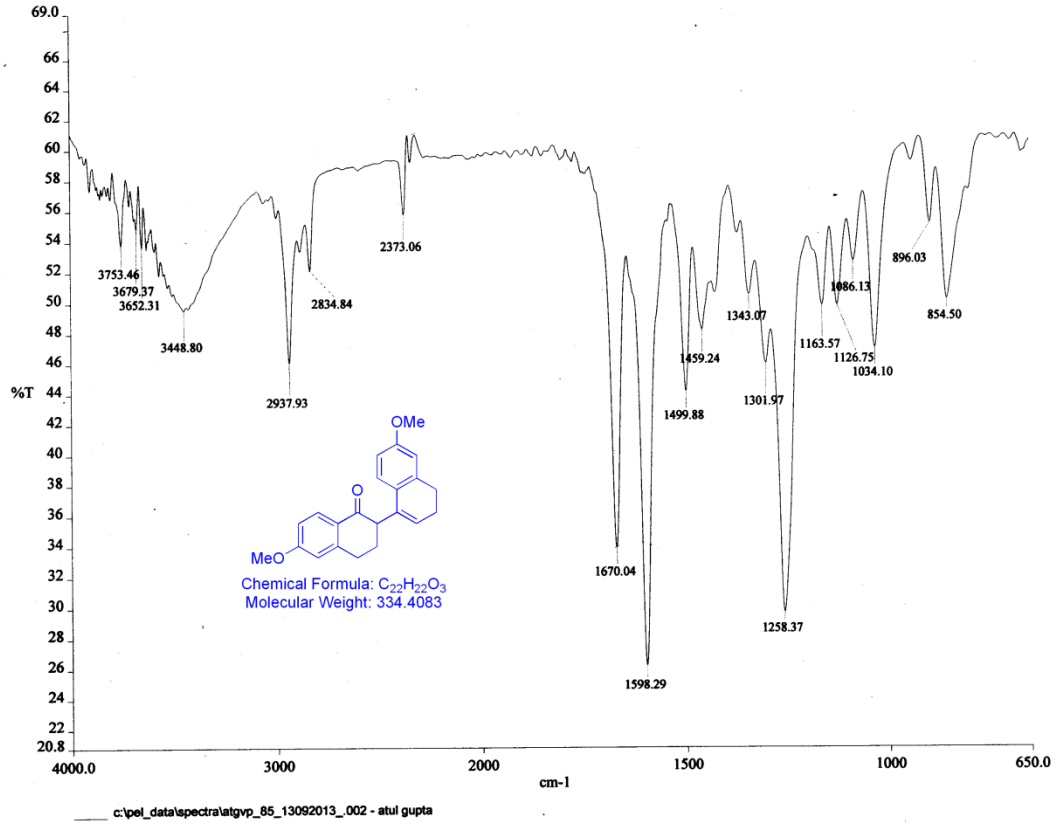


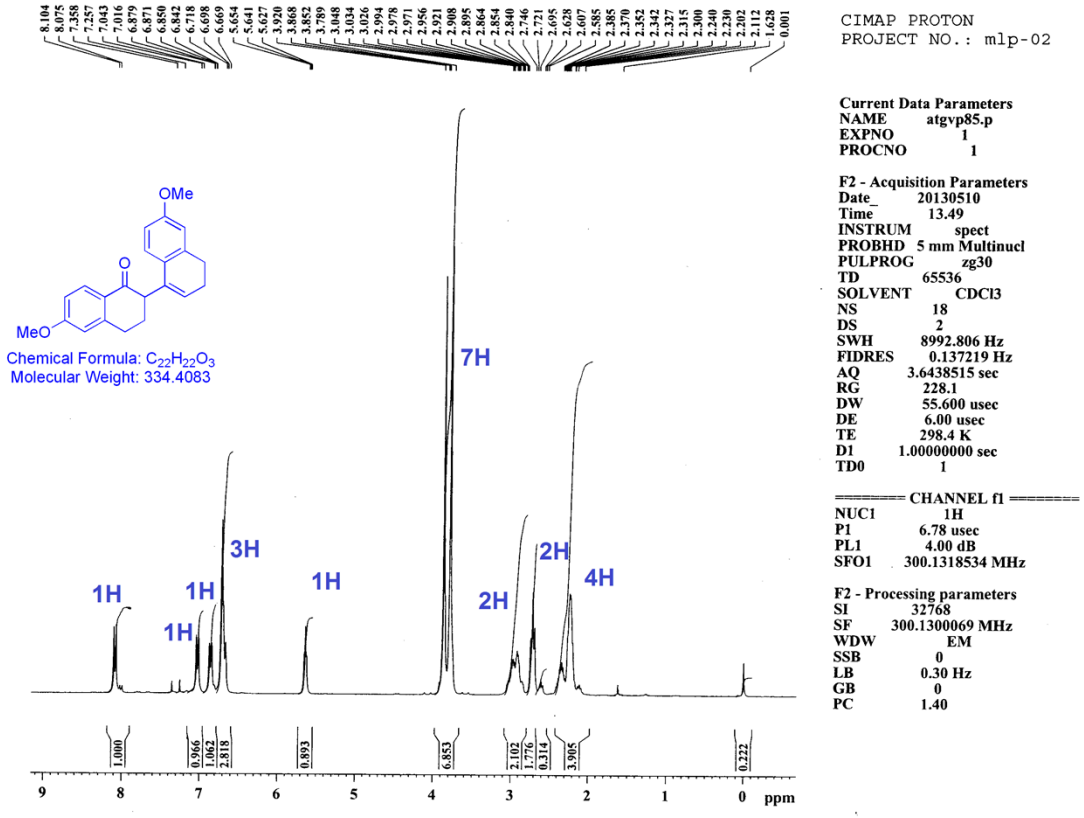
File Path-D:\Direct Mass report- Aug 2010\ATG-MI-100.dcc

Aug 13, 2013

[Dr. Karuna Sh

6,6'-dimethoxy-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'*H*)-one (10d):





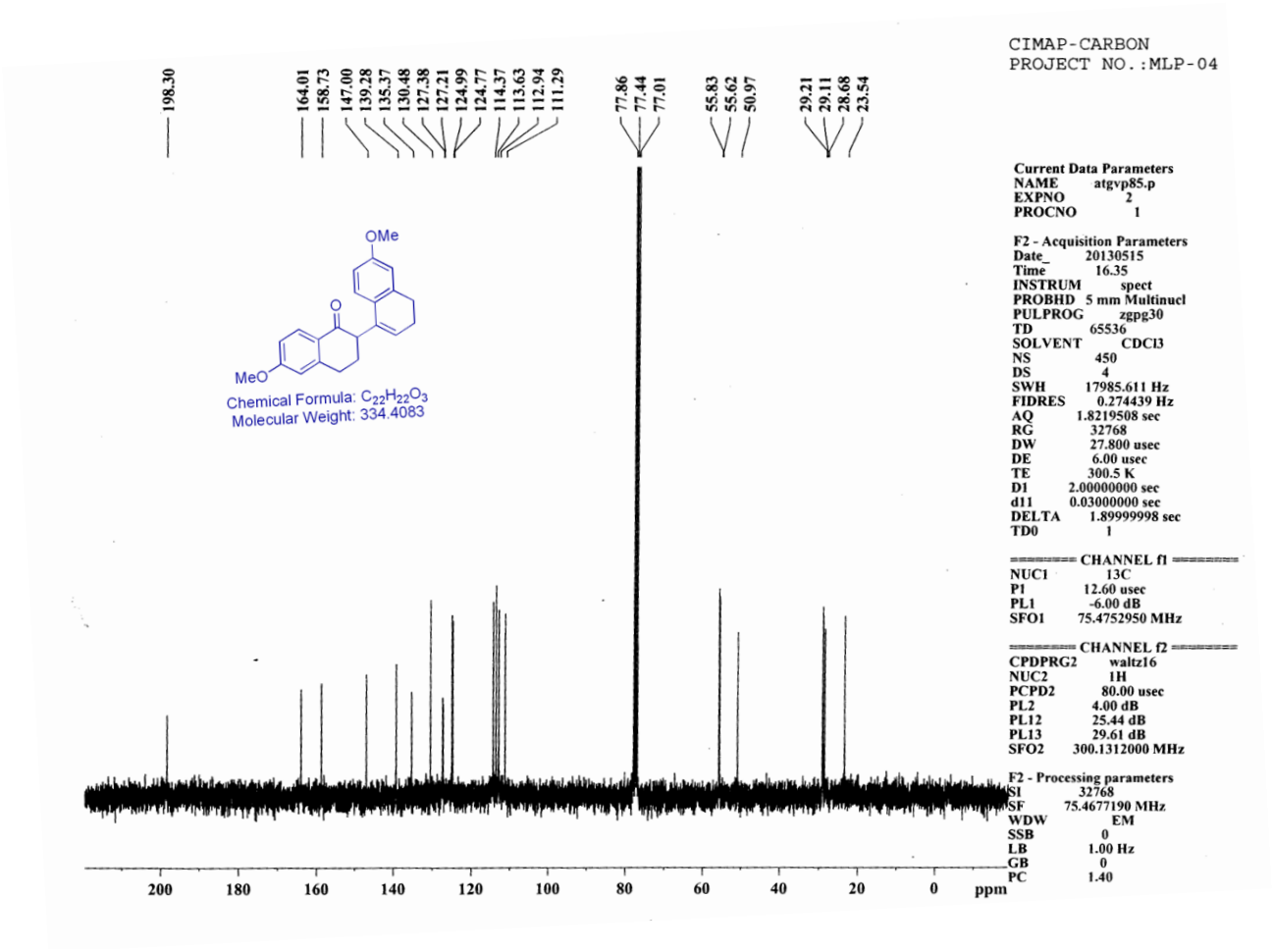
CIMAP PROTON
PROJECT NO.: mlp-02

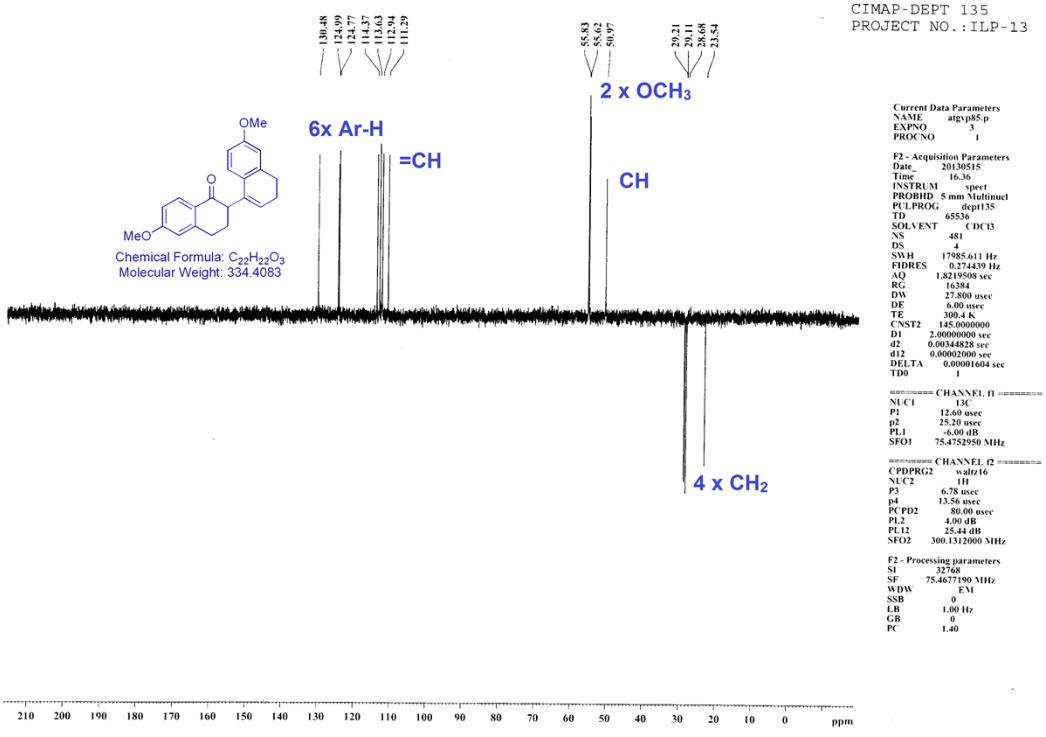
Current Data Parameters
NAME atgvp85.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130510
Time 13.49
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 228.1
DW 55.600 usec
DE 6.00 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1

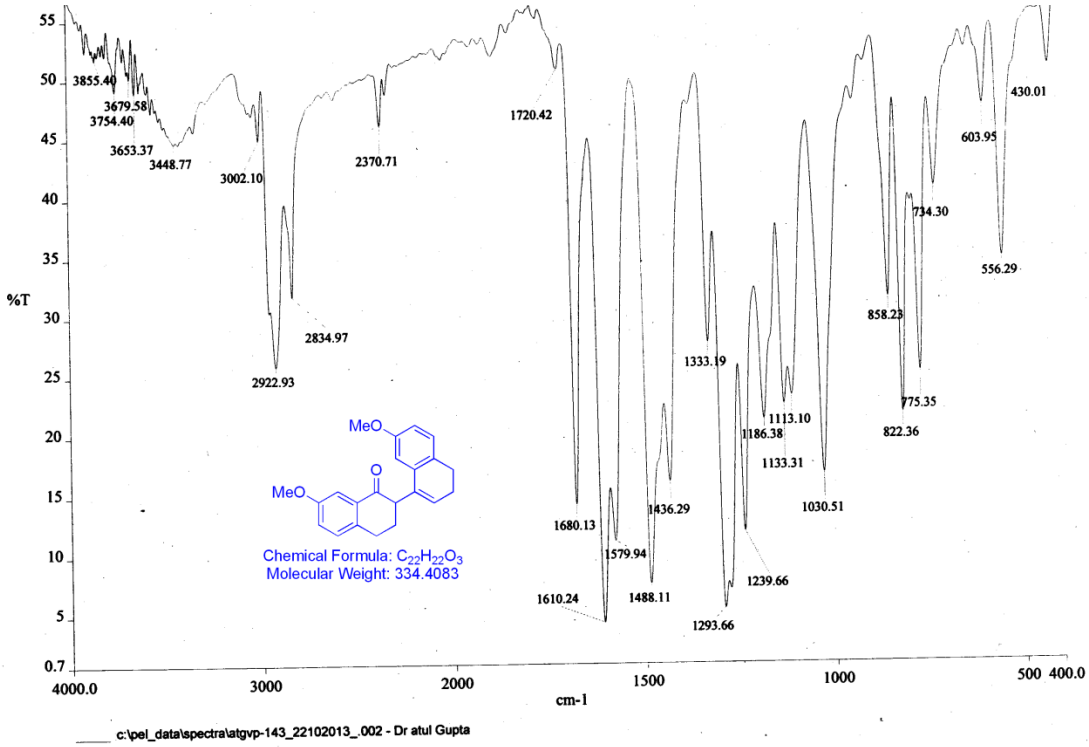
======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

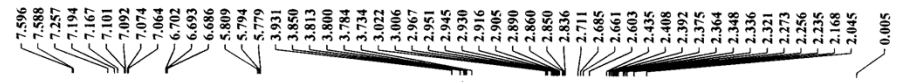
F2 - Processing parameters
SI 32768
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


CIMAP-CARBON
PROJECT NO.:MLP-04
198.30
164.01
158.73
147.00
139.28
135.37
130.48
127.38
127.21
124.99
124.77
114.37
113.63
112.94
111.29
77.86
77.44
77.01
55.83
55.62
50.97
29.21
29.11
28.68
23.54
OMe
O
MeO
Chemical Formula: C22H22O3
Molecular Weight: 334.4083
Current Data Parameters
NAME atgvp85.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130515
Time 16.35
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 450
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

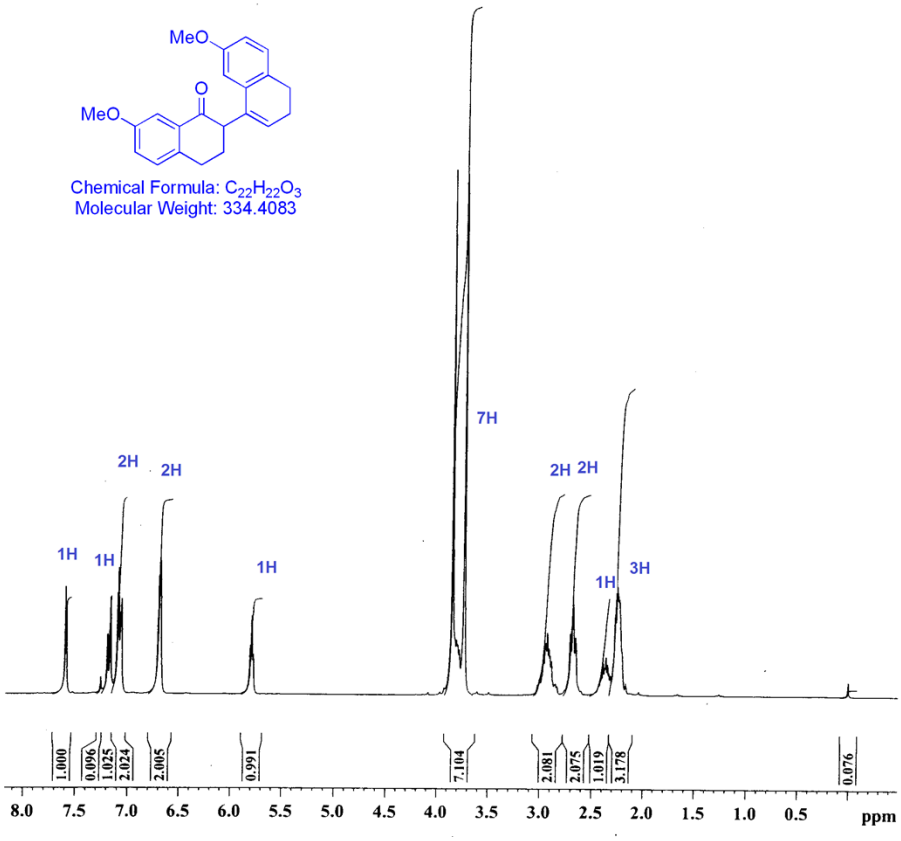

CIMAP-DEPT 135
PROJECT NO.:ILP-13
130.48
124.99
124.77
114.37
113.63
112.94
111.29
55.83
55.62
50.97
29.21
29.11
28.68
23.54
2 x OCH3
6x Ar-H
=CH
CH
4 x CH2
OMe
MeO
Chemical Formula: C22H22O3
Molecular Weight: 334.4083
Current Data Parameters
NAME atgvp85.p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130515
Time 16.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 481
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.4 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

7,7'-dimethoxy-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'H)-one (10e):





CIMAP PROTON
PROJECT NO.: mlp-02

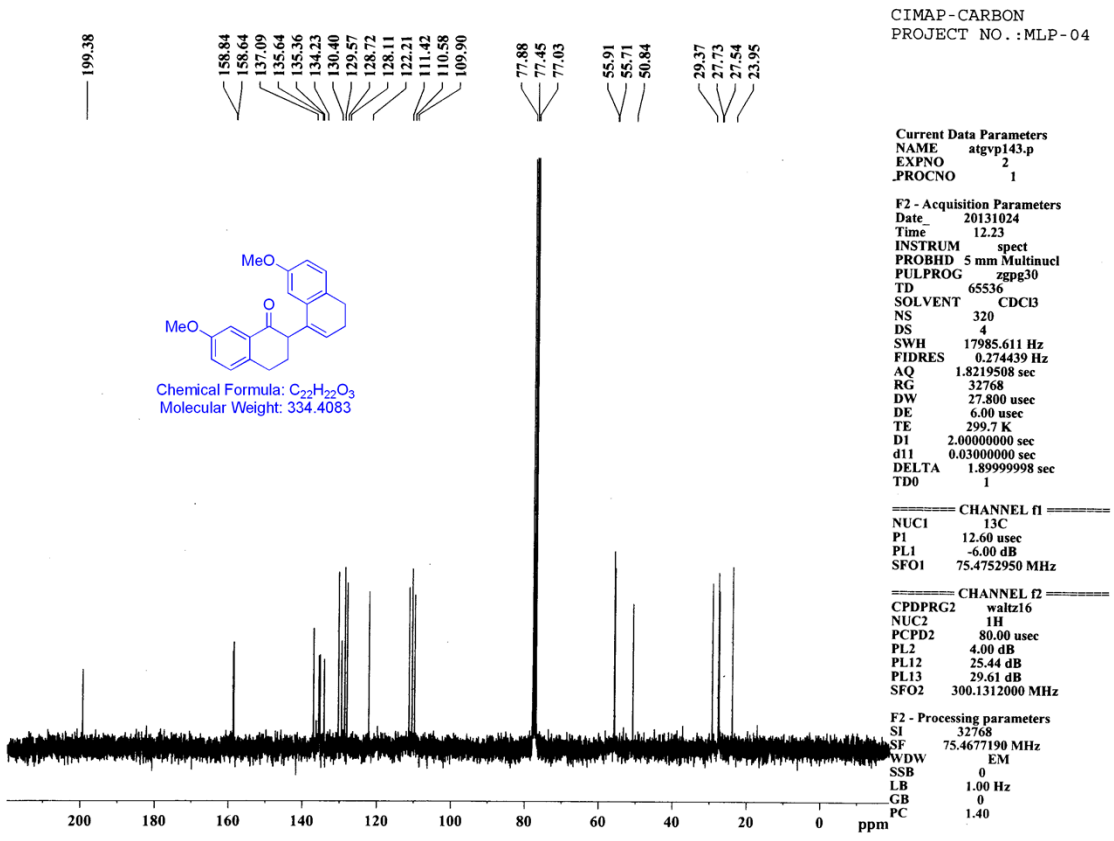


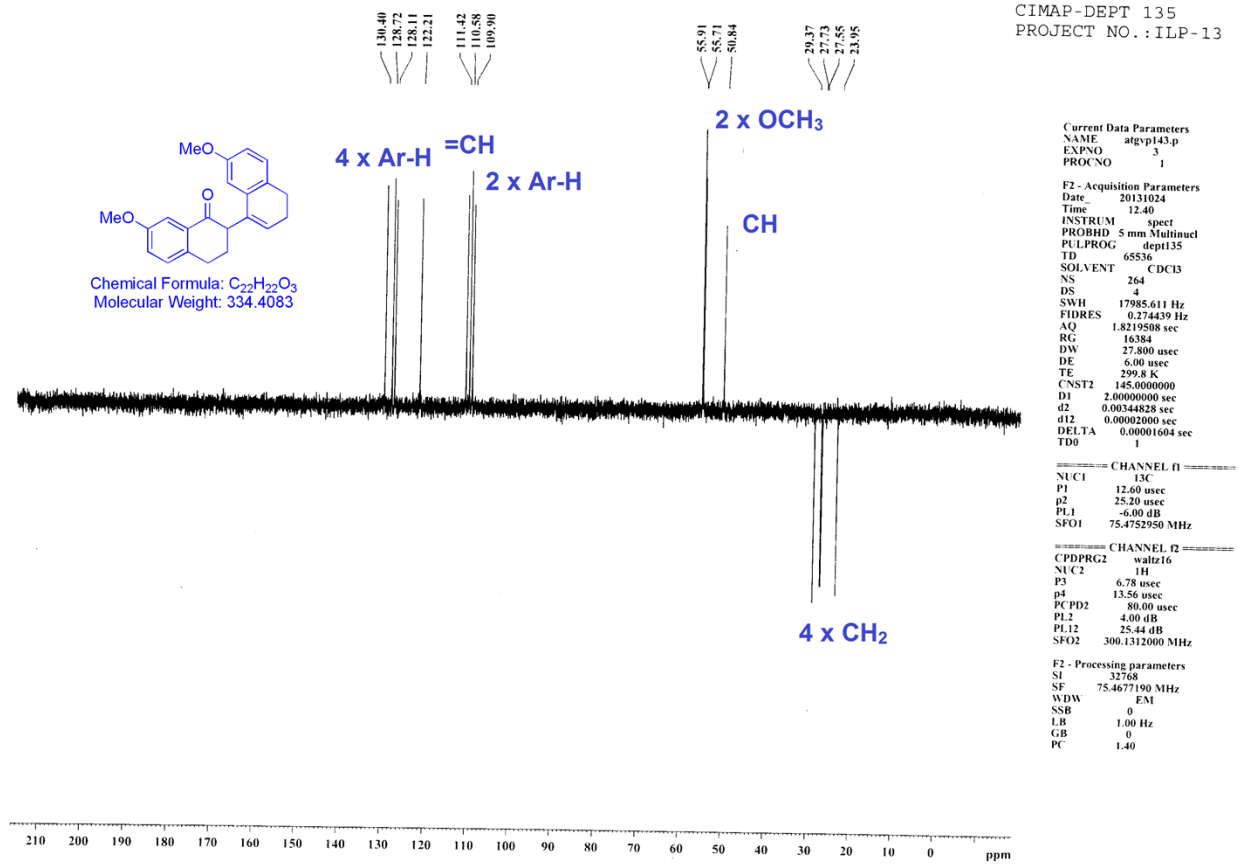
Current Data Parameters
NAME atgvp143.p
EXPNO 1
PROCNO 1

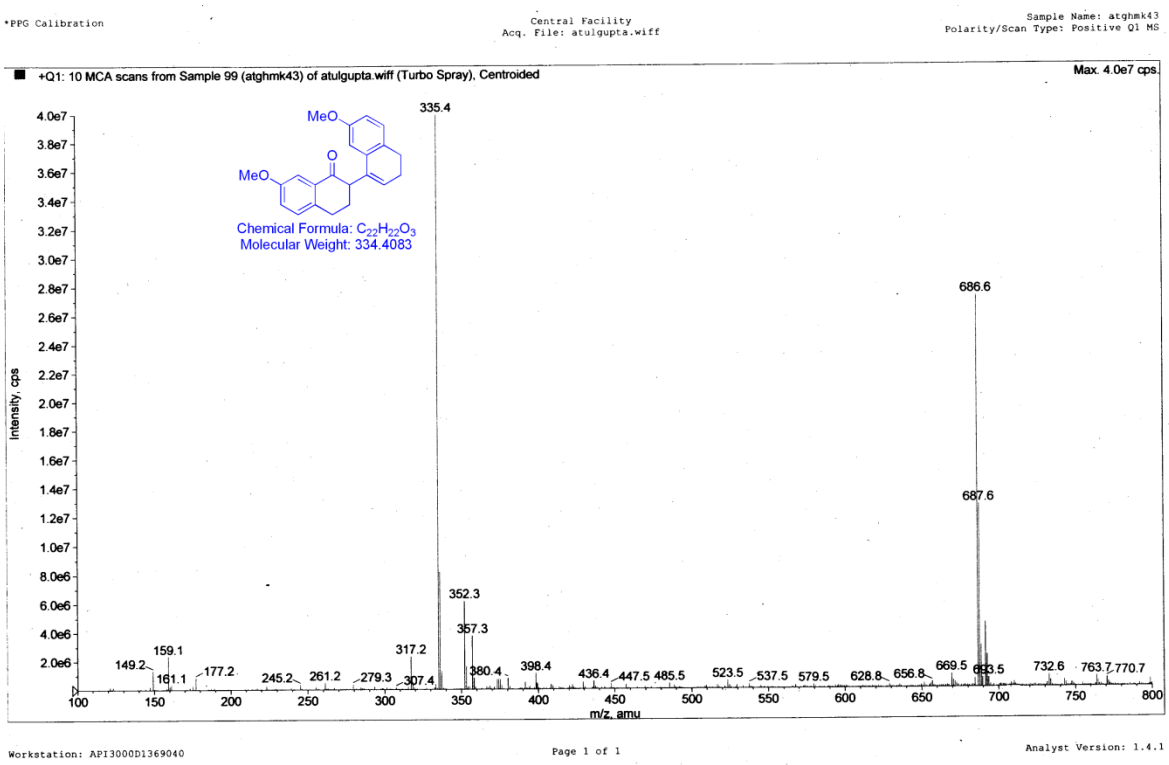
F2 - Acquisition Parameters
Date_ 20131024
Time 12.02
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

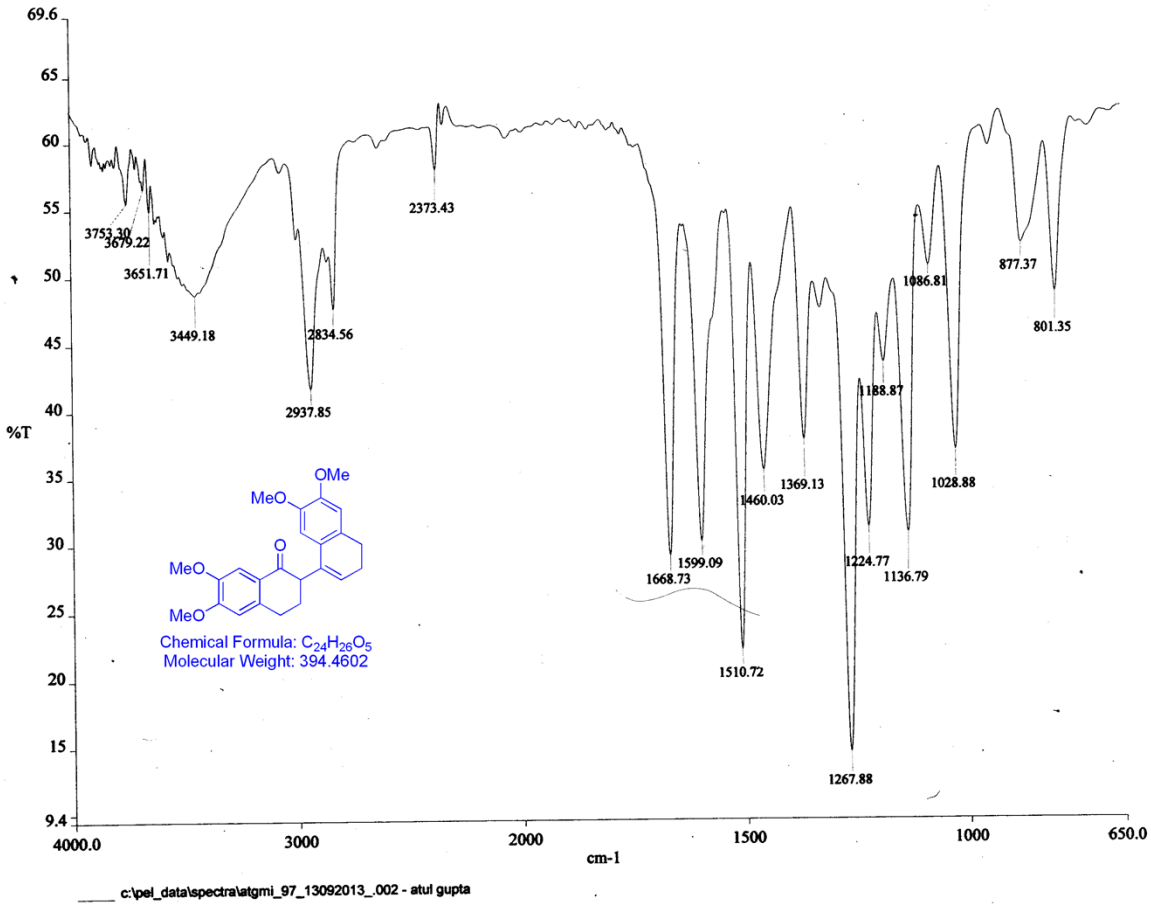
F2 - Processing parameters
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

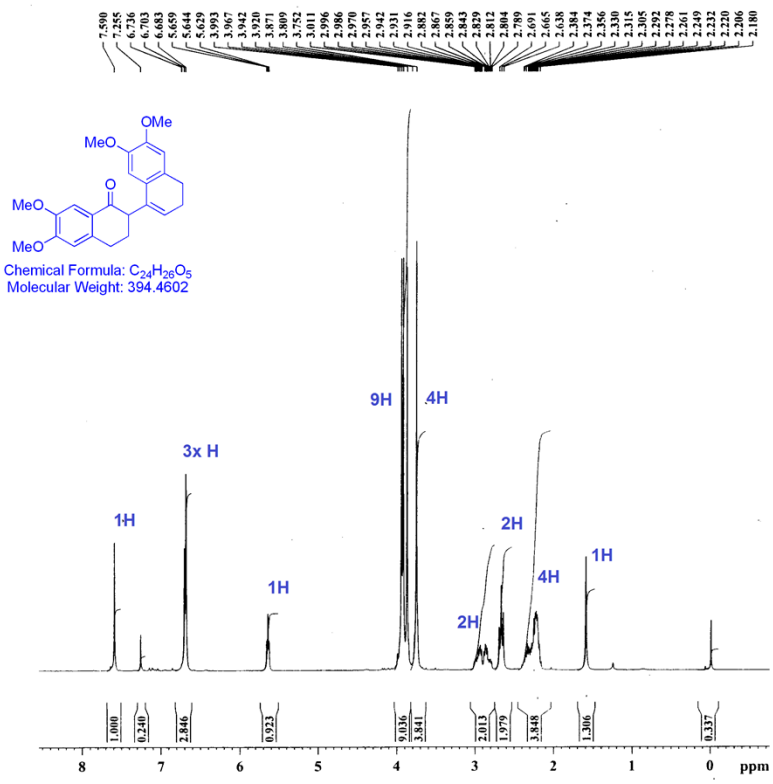

CIMAP-CARBON
PROJECT NO.:MLP-04
199.38
158.84
158.64
137.09
135.64
135.36
134.23
130.40
129.57
128.72
128.11
122.21
111.42
110.58
109.90
77.88
77.45
77.03
55.91
55.71
50.84
29.37
27.73
27.54
23.95
MeO
MeO
O
Chemical Formula: C22H22O3
Molecular Weight: 334.4083
Current Data Parameters
NAME atgvp143.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 12.23
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 320
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 299.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


CIMAP-DEPT 135
PROJECT NO.:ILP-13
130.40
128.72
128.11
122.21
111.42
110.58
109.90
55.91
55.71
50.84
29.37
27.73
27.55
23.95
2 x OCH3
4 x Ar-H
=CH
2 x Ar-H
CH
4 x CH2
MeO
MeO
O
Chemical Formula: C22H22O3
Molecular Weight: 334.4083
Current Data Parameters
NAME atgvp143.p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 12.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 264
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 299.8 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



6,6',7,7'-tetramethoxy-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'H)-one (10f):





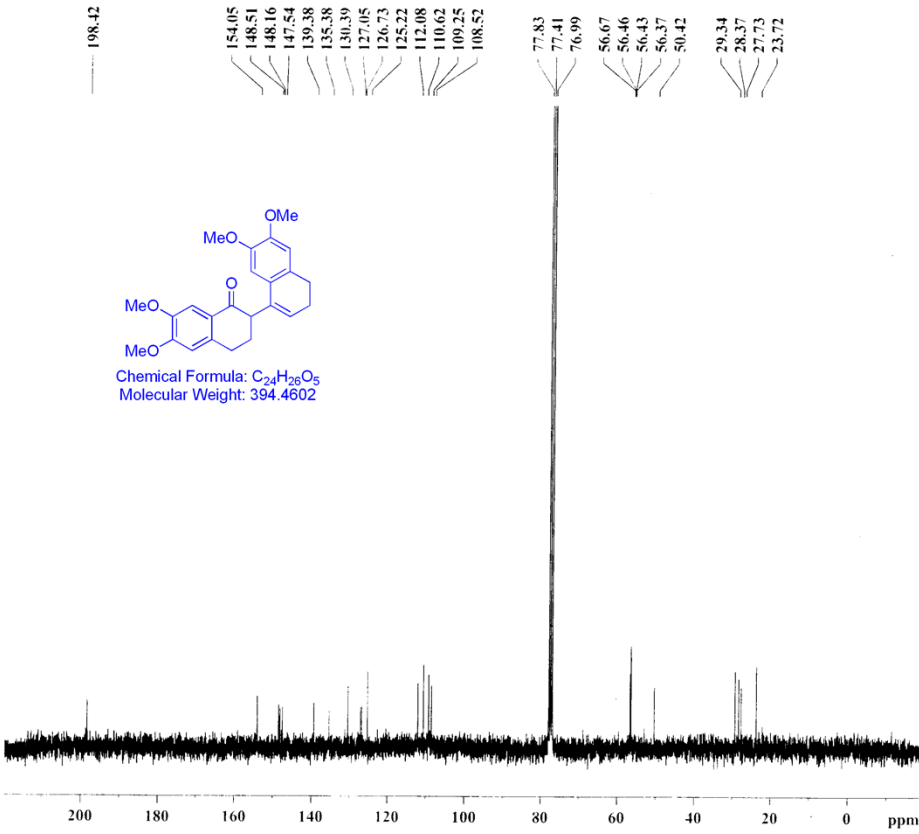
CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters
NAME atgmi97.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130905
Time 16.10
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 301.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



CIMAP-CARBON
PROJECT NO.:MLP-04

Current Data Parameters
NAME atgmi97.p
EXPNO 2
PROCNO 1

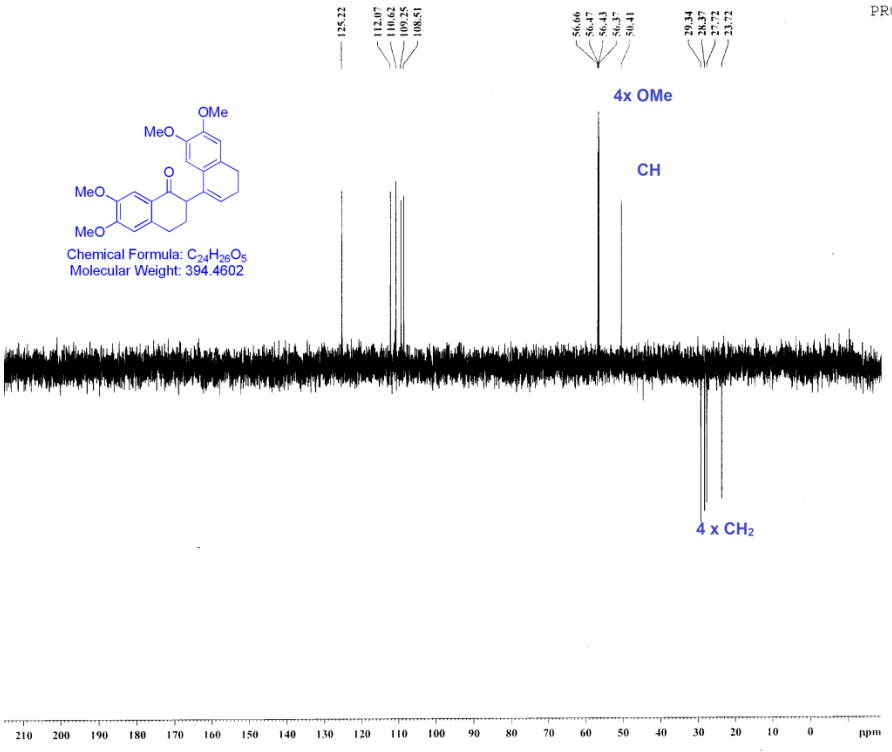
F2 - Acquisition Parameters
Date_ 20130905
Time 16.11
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 301.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CIMAP-DEPT 135
PROJECT NO.:ILP-13



Current Data Parameters
NAME atgmi97.p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date 20130905
Time 16.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 360
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 302.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Central Institute of Medicinal and Aromatic Plants
(Analytical Chemistry Division)
Lucknow-226015

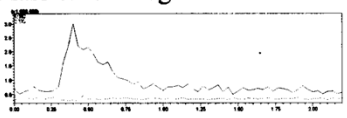
ANALYTICAL TEST REPORT

Sample Information for Direct Mass Analysis of Isolates/synthetic molecule

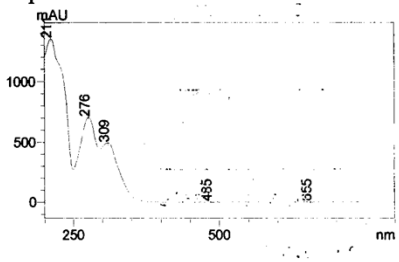
Sample Code : ATG-MI-97
Solubility : MeOH
Name of the Scientist : Dr ATUL GUPTA

Project Code: MLP-0
Mass Range: 300-500

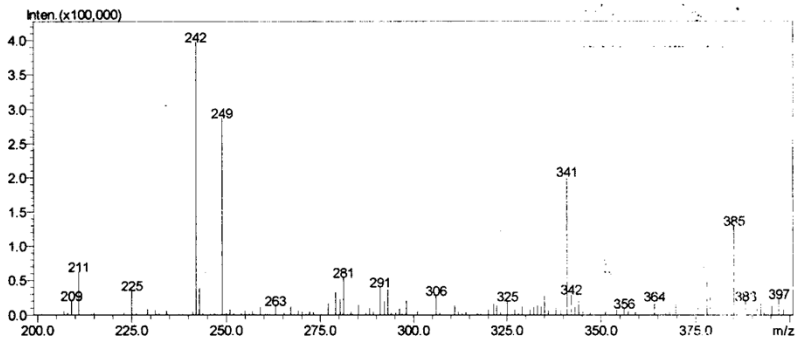
Mass chromatogram



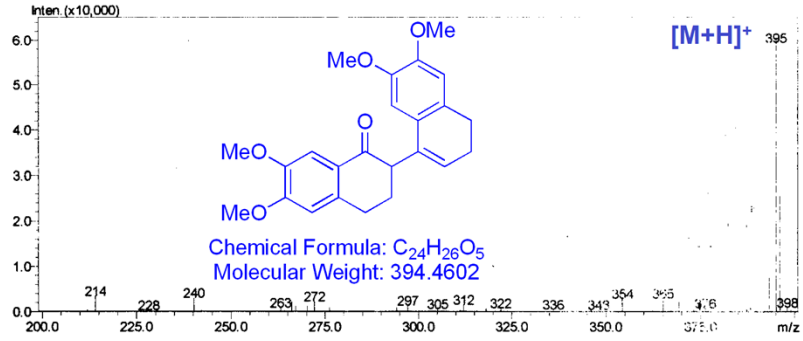
UV-Spectra



ESI+



ESI-

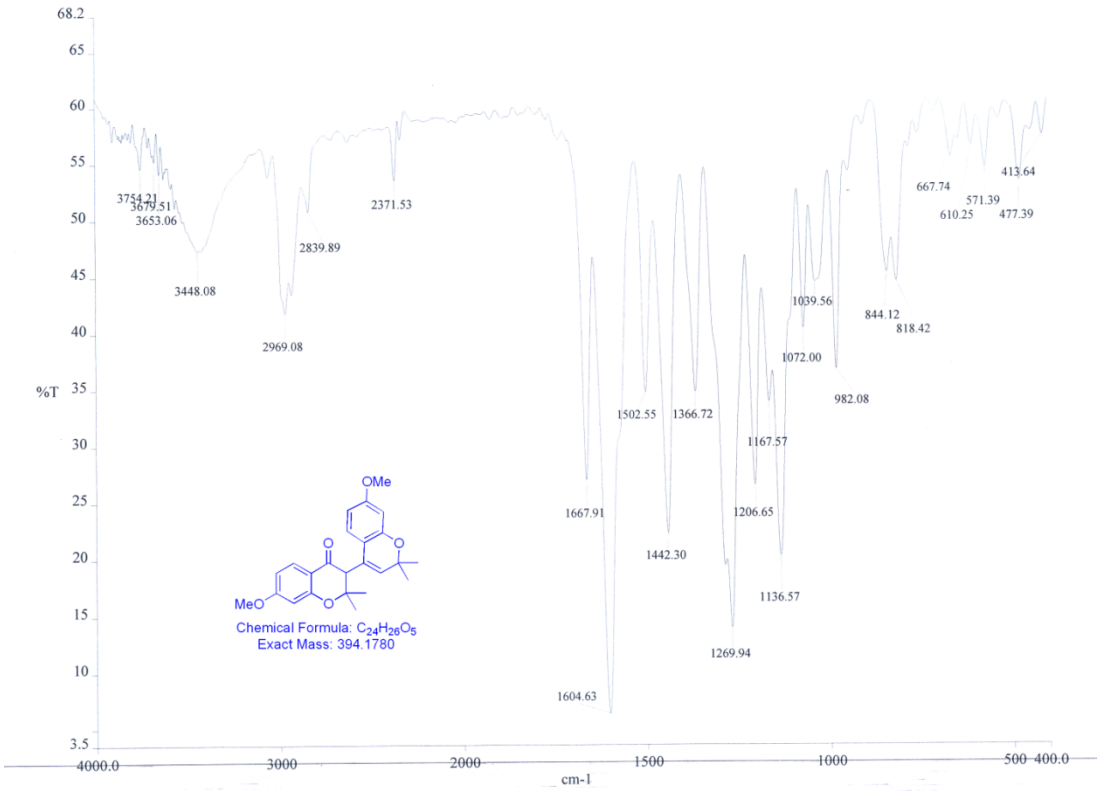


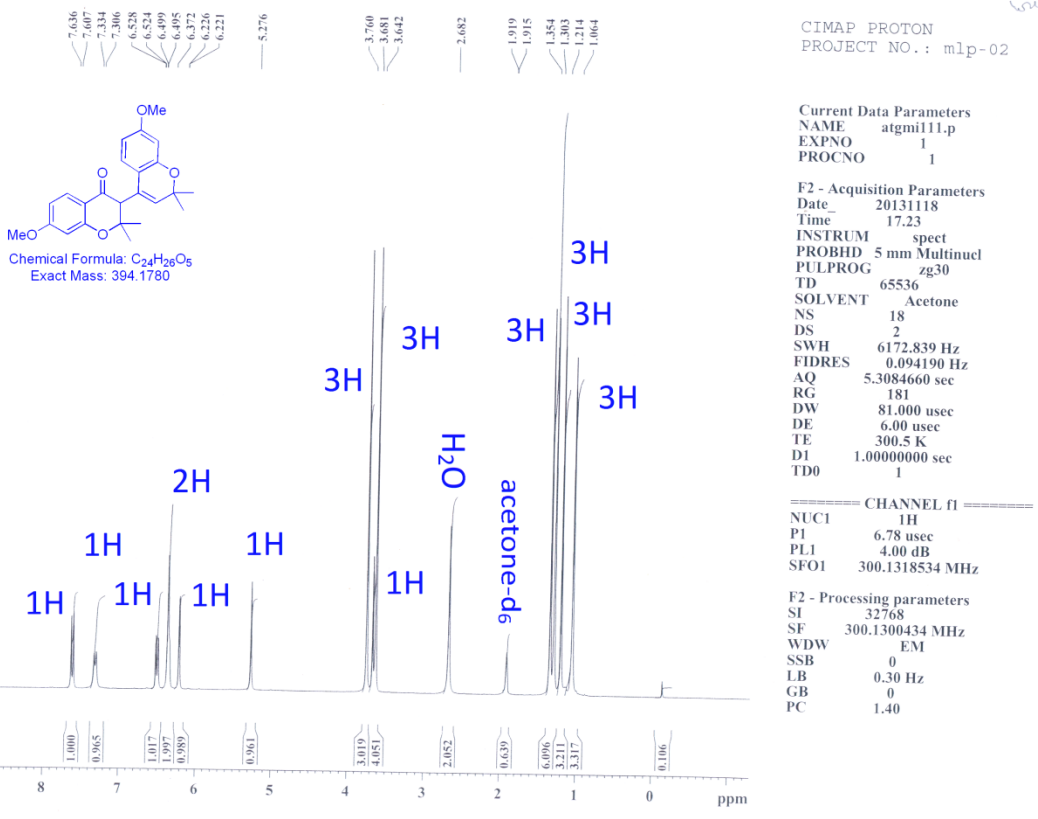
File Path-D:\Direct Mass report- Aug 2010\ATG-MI-97.doc

Aug 13, 2013

[Dr. Karuna Shanker]

6-methoxy-3-(6-methoxy-2,2-dimethyl-2H-chromen-4-yl)-2,2-dimethylchroman-4-one (10g):





CIMAP PROTON
PROJECT NO.: mlp-02

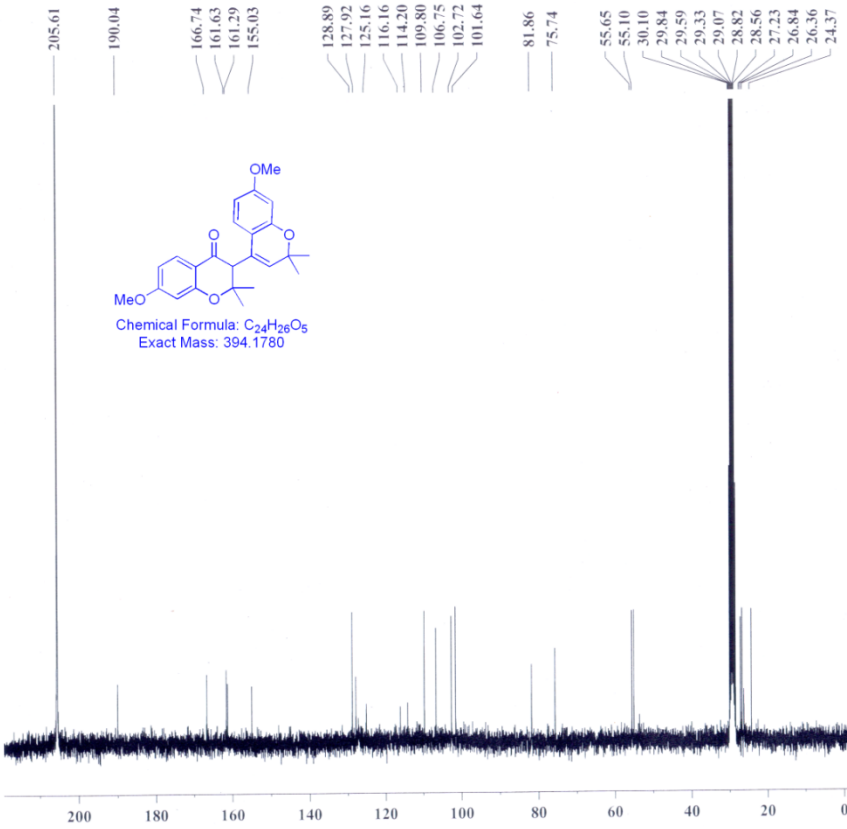
Current Data Parameters
NAME atgmi111.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131118
Time 17.23
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 18
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 300.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300434 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

CIMAP-CARBON
PROJECT NO.:MLP-04



Chemical Formula: $C_{24}H_{26}O_5$
Exact Mass: 394.1780

Current Data Parameters
NAME atgmi111.p
EXPNO 4
PROCNO 1

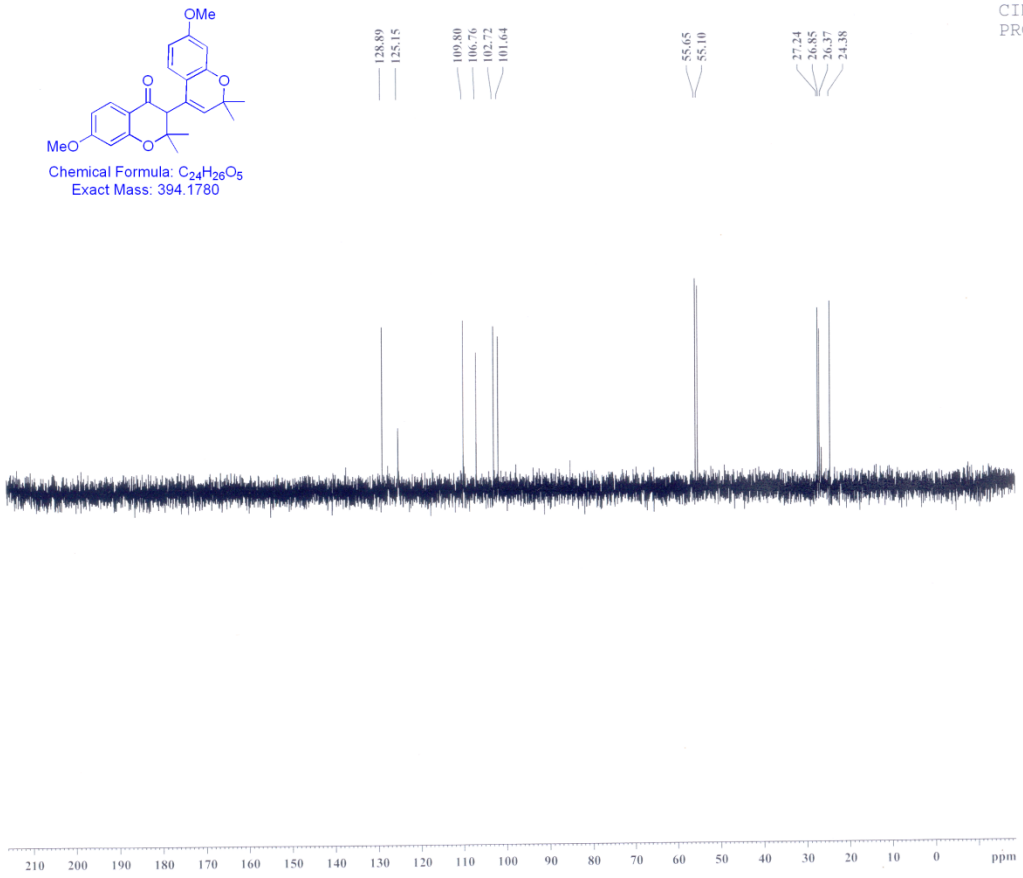
F2 - Acquisition Parameters
Date_ 20131119
Time 17.21
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 515
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ======
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CIMAP-DEPT 135
PROJECT NO.:ILP-13



Chemical Formula: $C_{24}H_{26}O_5$
Exact Mass: 394.1780

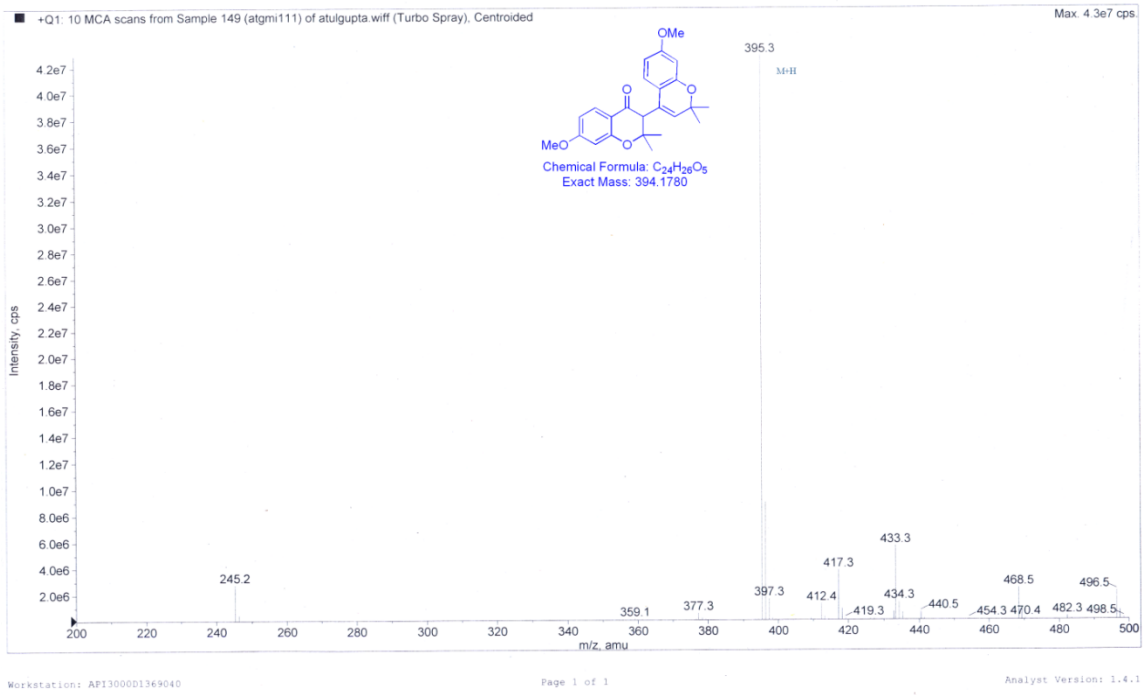
Current Data Parameters
NAME atgmi111-p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131119
Time 17.24
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.6 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

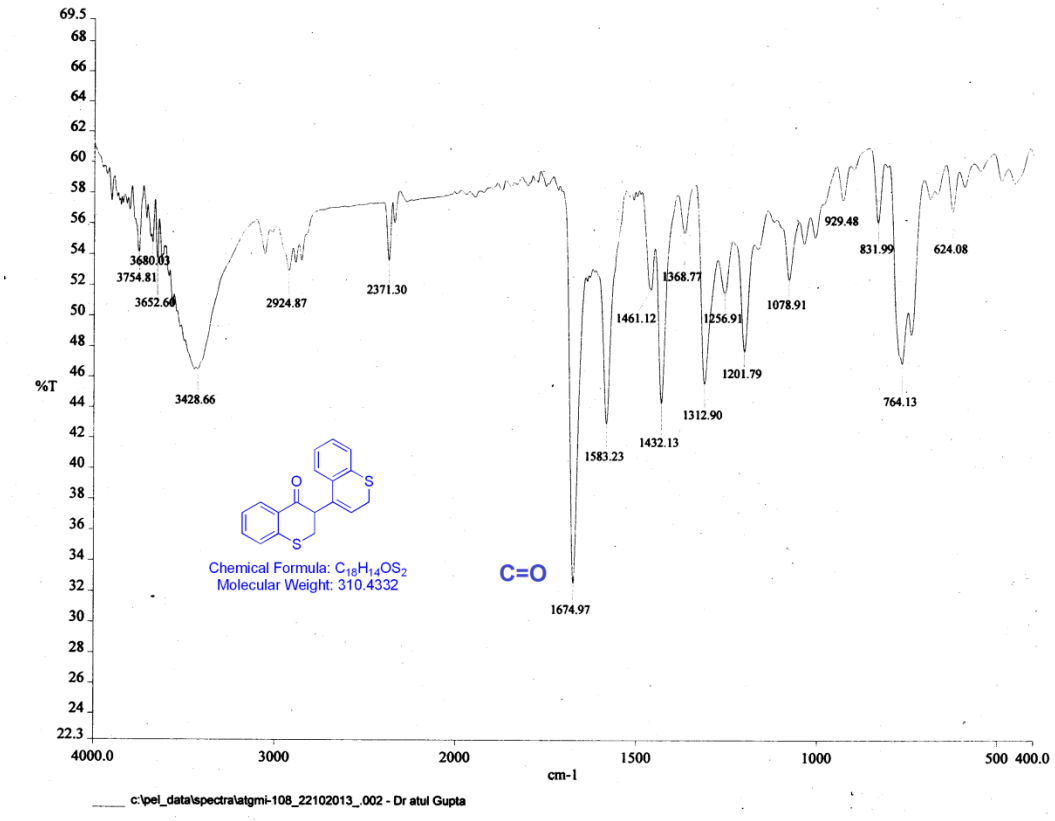
======== CHANNEL f1 ======
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

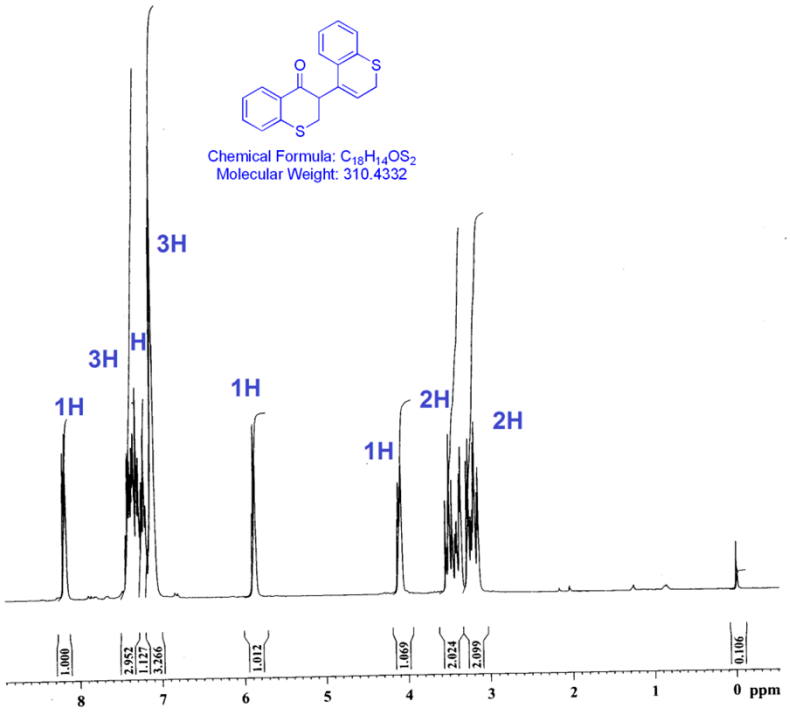
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



3,4'-bithiochroman-4-one (10h):





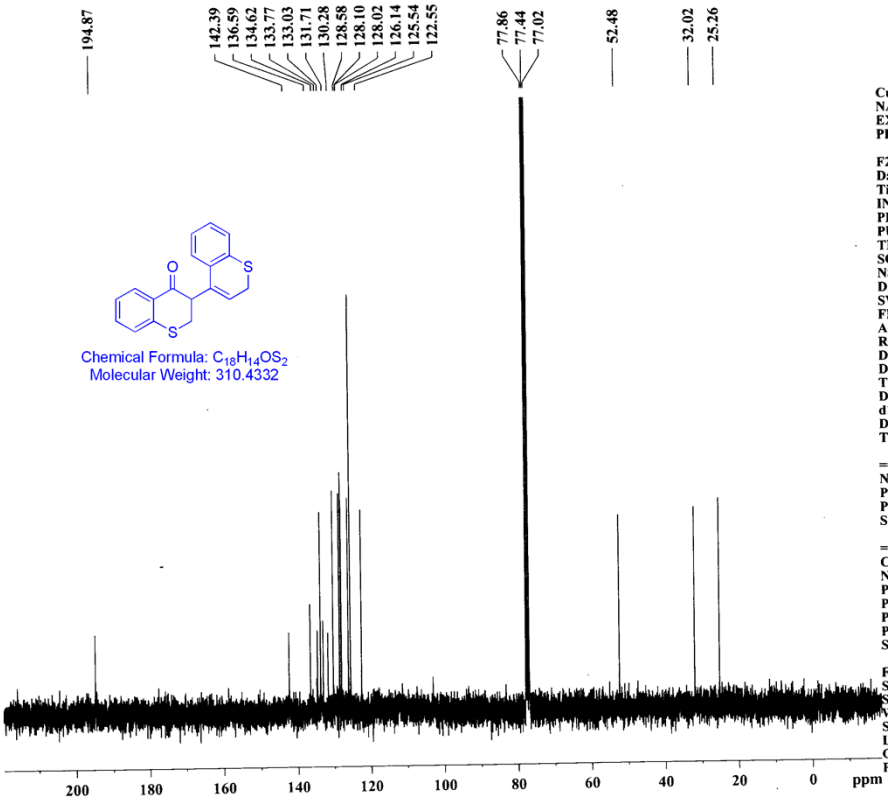
CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters
NAME atgmi108.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131024
Time 13.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



CIMAP-CARBON
PROJECT NO.:MLP-04

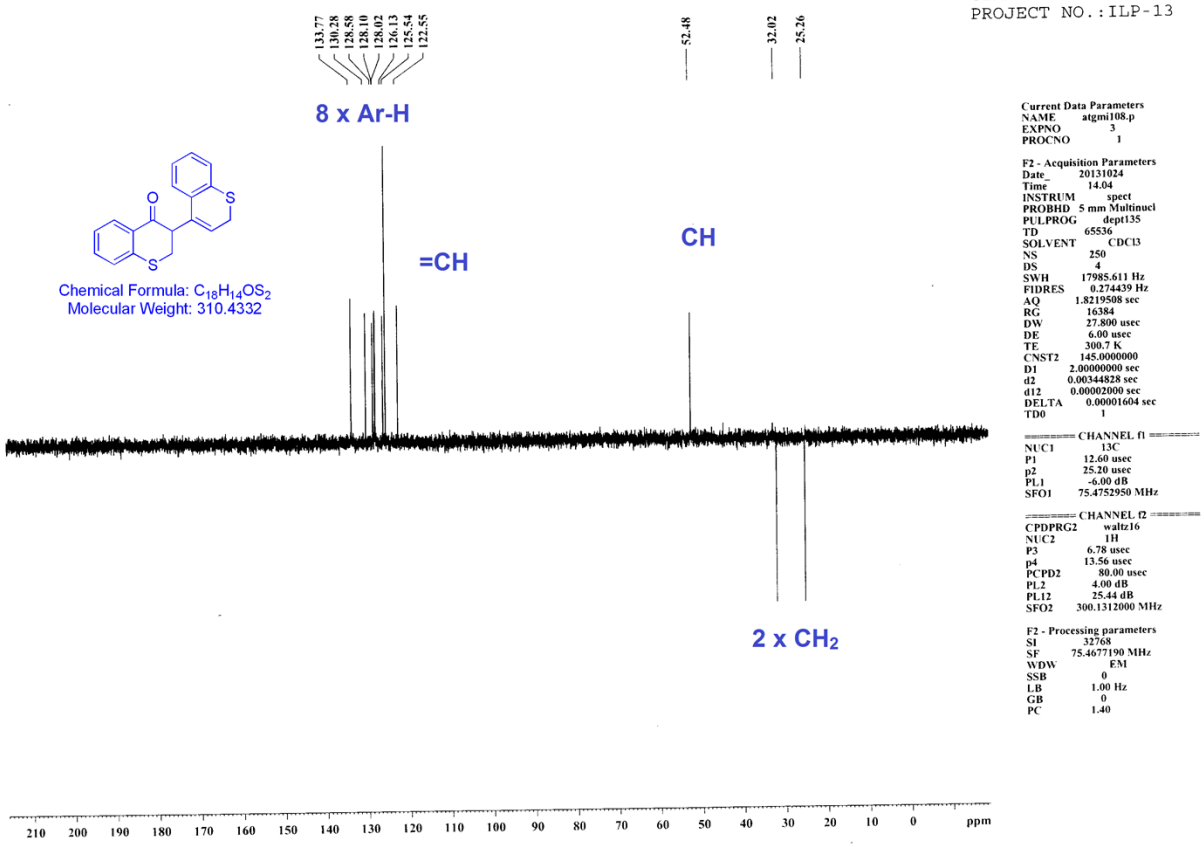
Current Data Parameters
NAME atgmi108.p
EXPNO 2
PROCNO 1

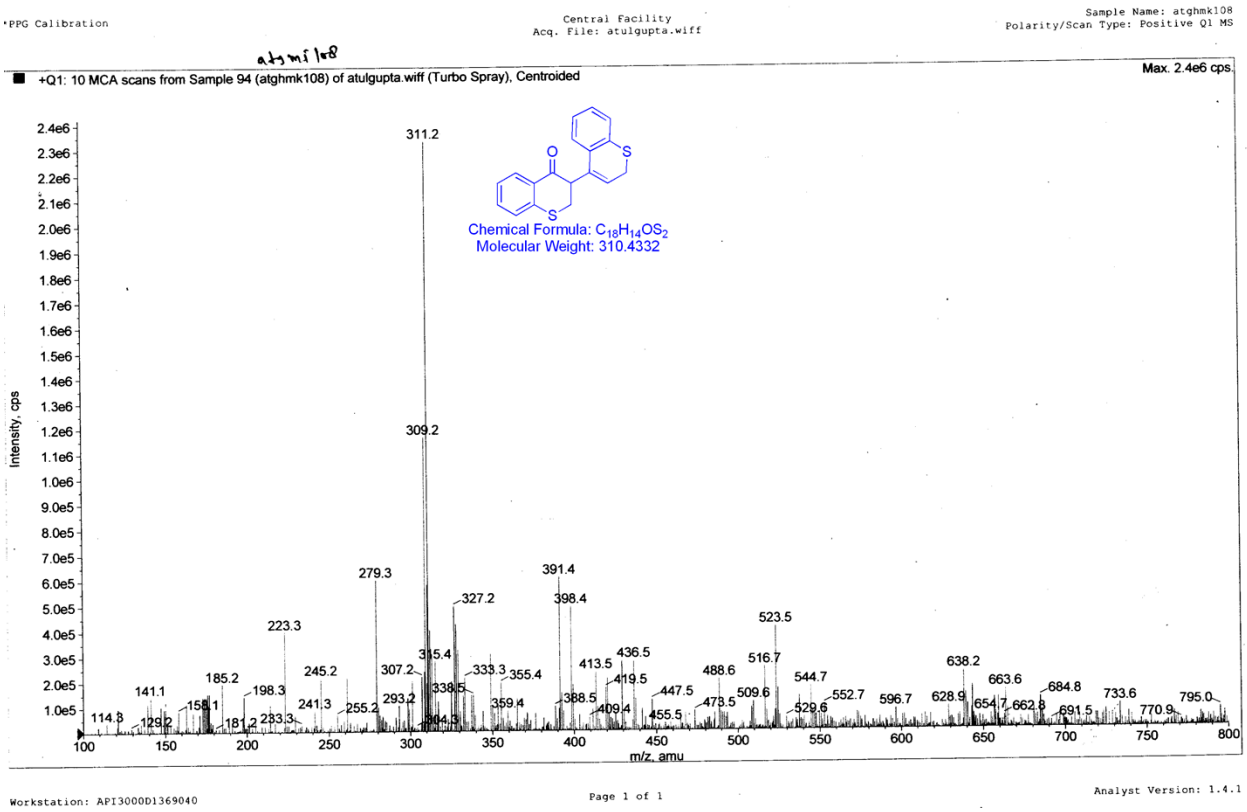
F2 - Acquisition Parameters
Date_ 20131024
Time 14.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

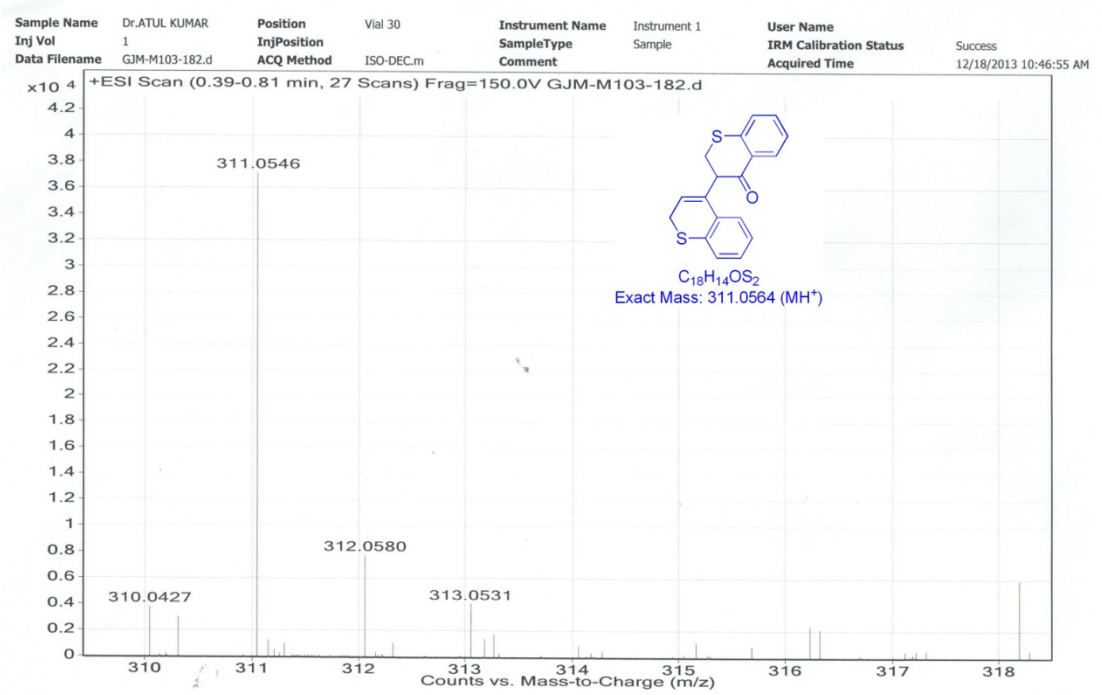
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

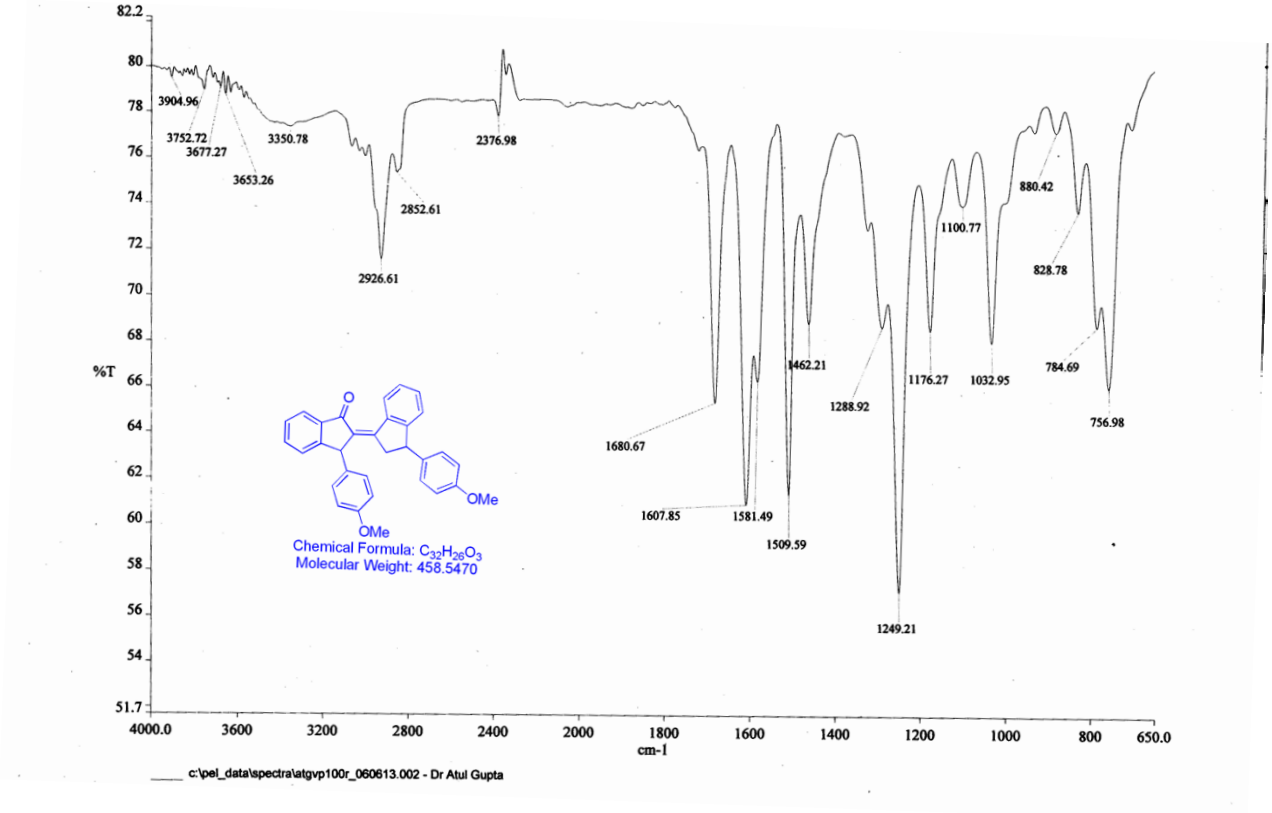
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

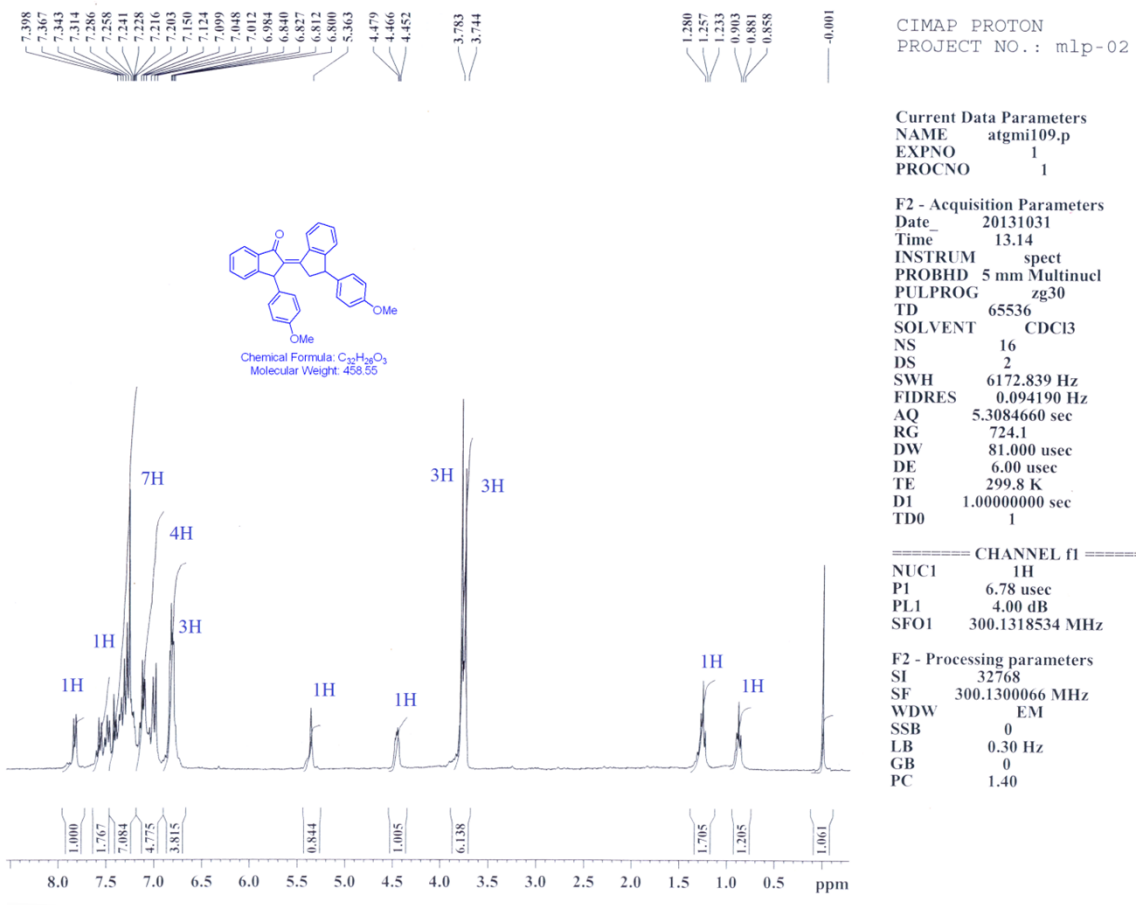

CIMAP-DEPT 135
PROJECT NO.:ILP-13
133.77
130.28
128.58
128.10
128.02
126.13
125.54
122.55
52.48
32.02
25.26
8 x Ar-H
=CH
CH
2 x CH2
Chemical Formula: C18H14OS2
Molecular Weight: 310.4332
Current Data Parameters
NAME atgmi108.p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 14.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 250
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.7 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

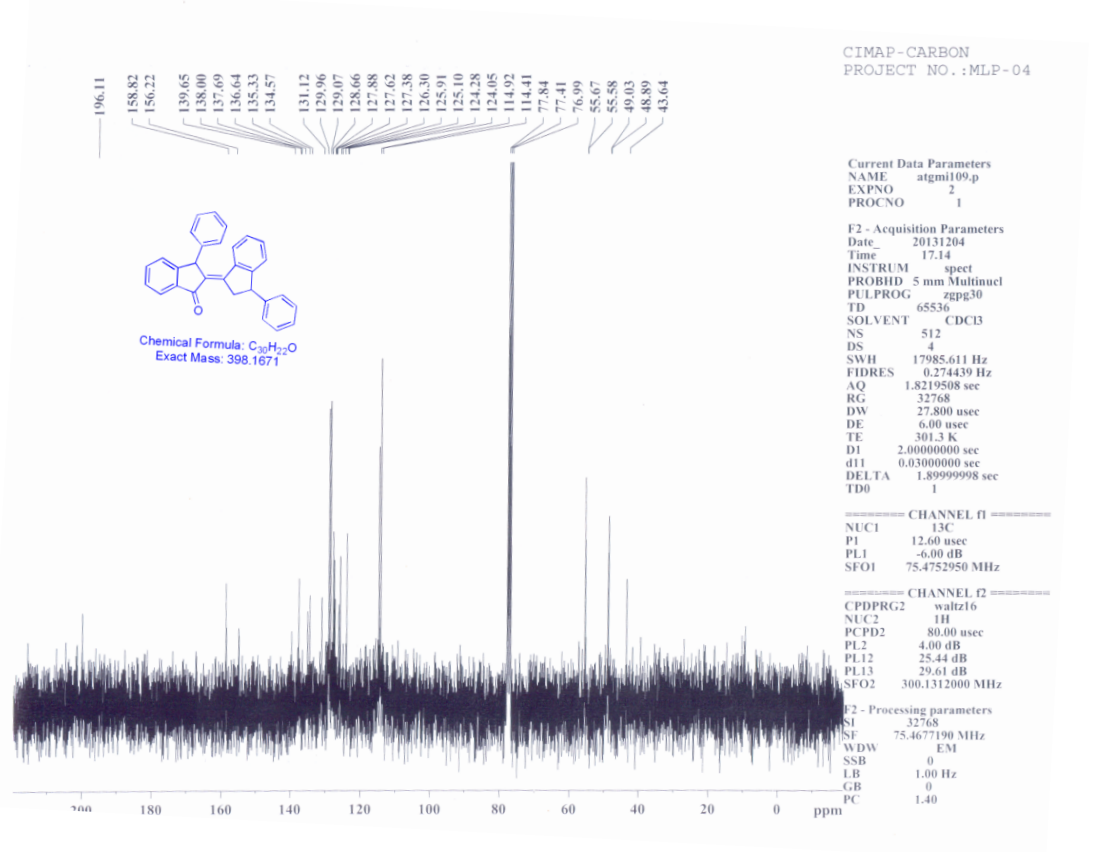

PPG Calibration
Central Facility
Acq. File: atulgupta.wiff
Sample Name: atghmk108
Polarity/Scan Type: Positive Q1 MS
atgmi108
+Q1: 10 MCA scans from Sample 94 (atghmk108) of atulgupta.wiff (Turbo Spray), Centroided
Max. 2.4e6 cps.
Intensity, cps
m/z, amu
311.2
309.2
279.3
391.4
398.4
327.2
523.5
223.3
315.4
436.5
413.5
419.5
638.2
663.6
684.8
733.6
795.0
544.7
516.7
488.6
509.6
552.7
596.7
628.9
654.7
662.8
691.5
770.9
Chemical Formula: C18H14OS2
Molecular Weight: 310.4332
Workstation: API3000D1369040
Page 1 of 1
Analyst Version: 1.4.1

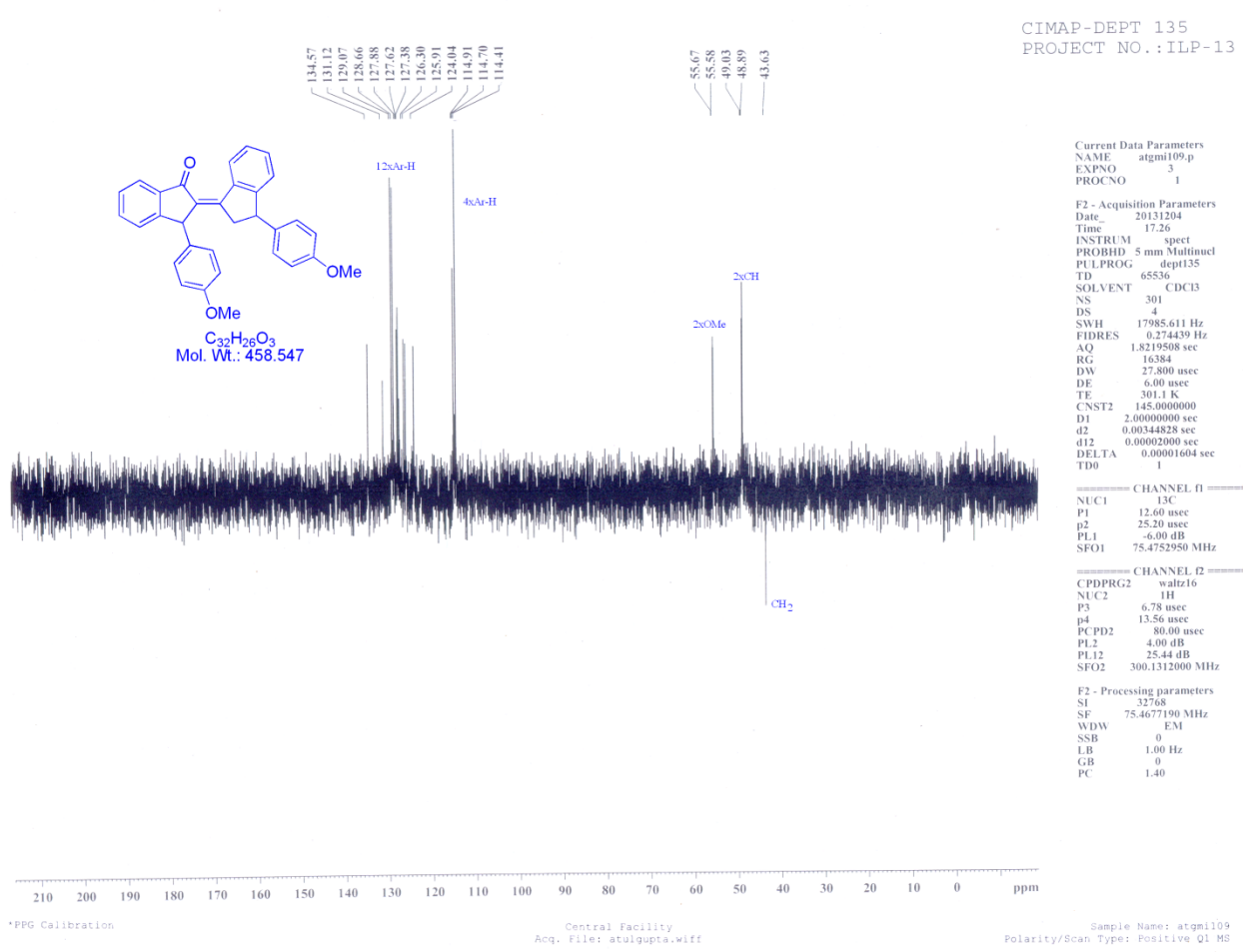


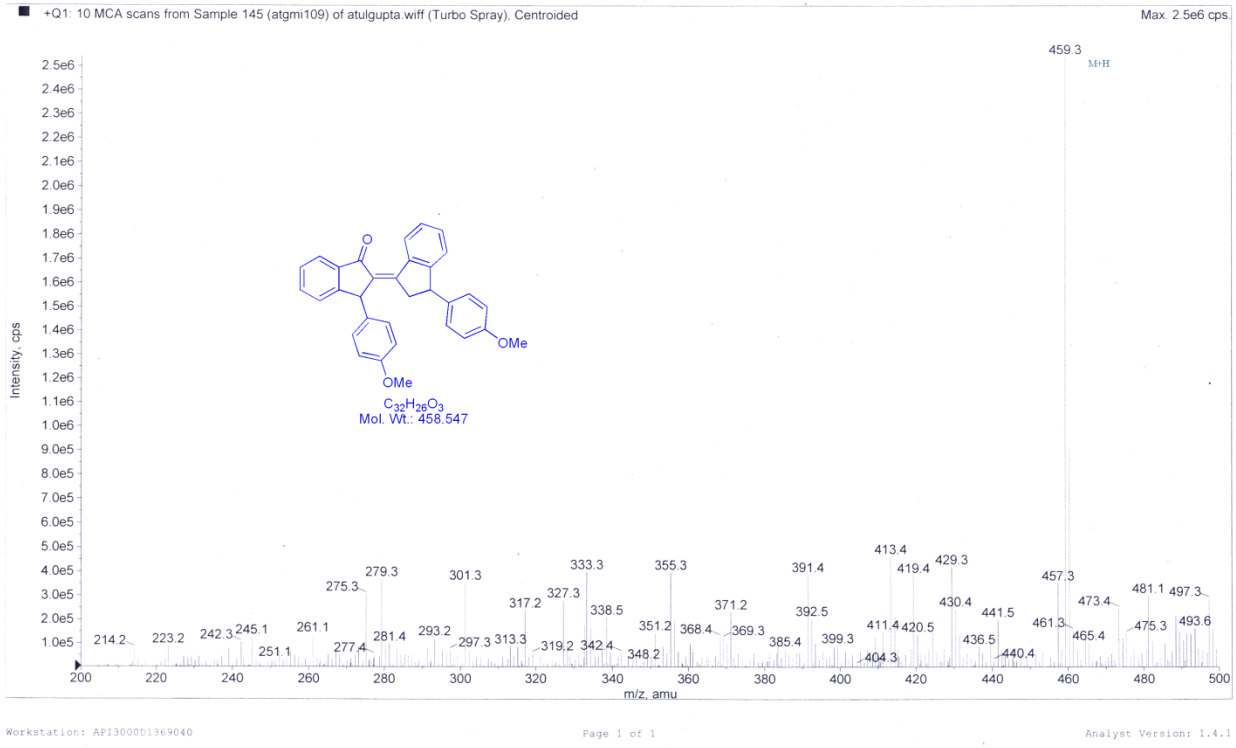
2-[3-(4-methoxyphenyl)-2,3-dihydro-1*H*-inden-1-ylidene]-3-(4-methoxyphenyl)-2,3-dihydro-1*H*-inden-1-one (12a):



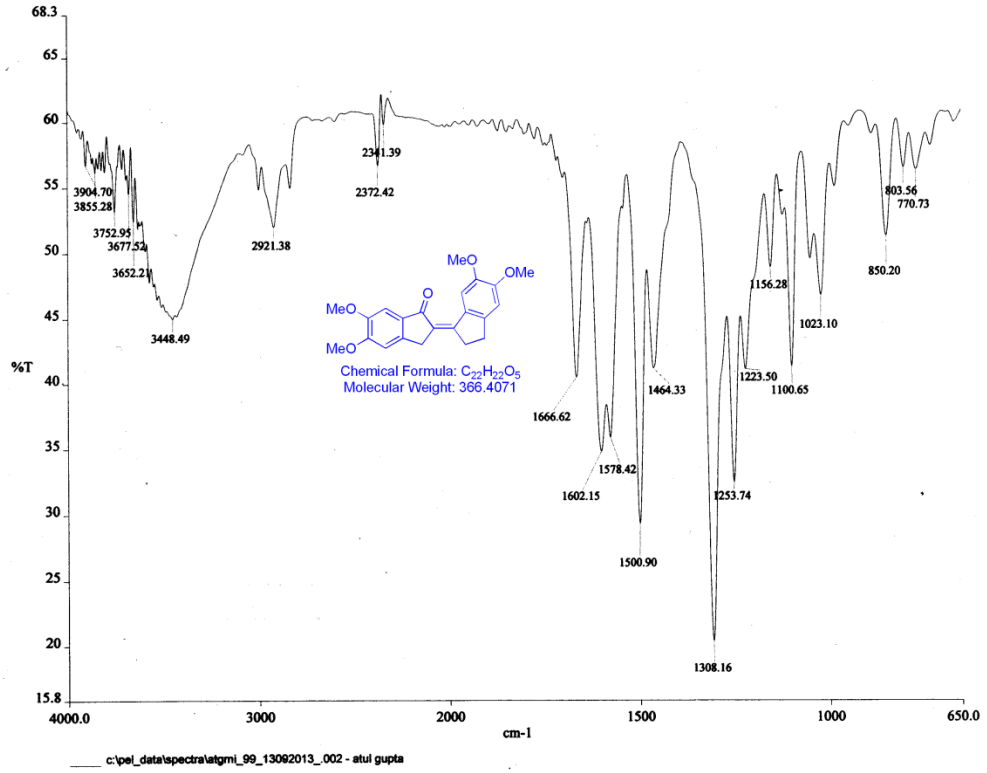

7.398
7.367
7.343
7.314
7.286
7.258
7.241
7.228
7.216
7.203
7.150
7.124
7.099
7.048
7.012
6.984
6.840
6.827
6.812
6.800
5.363
4.479
4.466
4.452
3.783
3.744
1.280
1.257
1.233
0.903
0.881
0.858
-0.001
CIMAP PROTON
PROJECT NO.: mlp-02
Current Data Parameters
NAME atgmi109.p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131031
Time 13.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 724.1
DW 81.000 usec
DE 6.00 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
OMe
OMe
Chemical Formula: C32H28O3
Molecular Weight: 458.55
7H
4H
3H
3H
3H
1H
1H
1H
1H
1H
1H
1.000
1.767
7.084
4.775
3.815
0.844
1.005
6.138
1.705
1.205
1.061
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

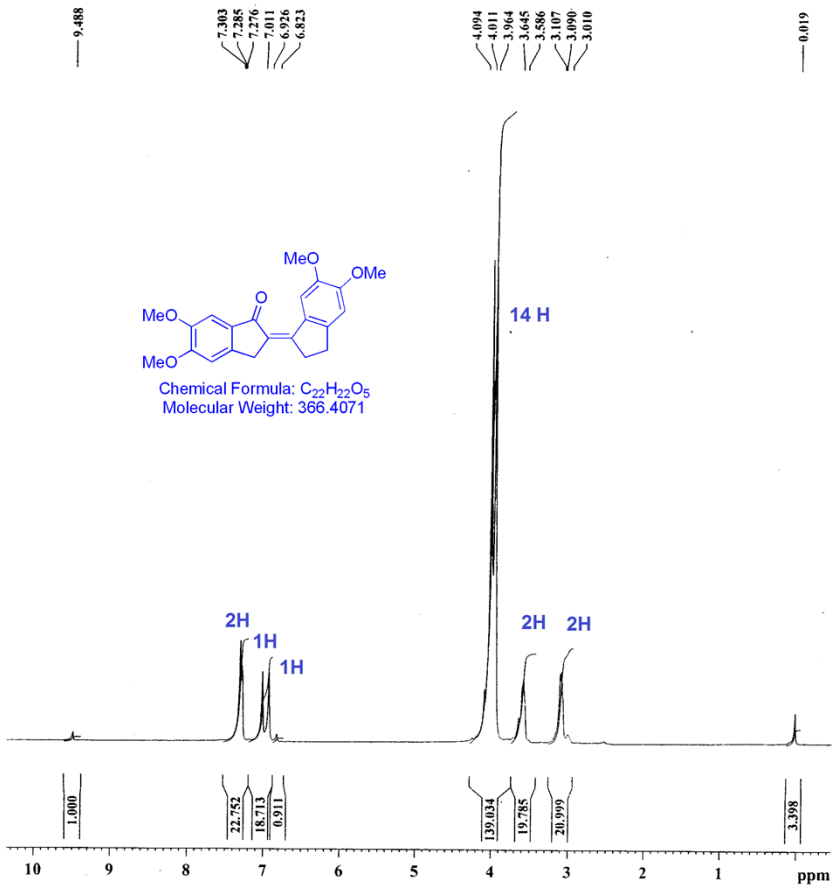

196.11
158.82
156.22
139.65
138.00
137.69
136.64
135.33
134.57
131.12
129.96
129.07
128.66
127.88
127.62
127.38
126.30
125.91
125.10
124.28
124.05
114.92
114.41
77.84
77.41
76.99
55.67
55.58
49.03
48.89
43.64
CIMAP-CARBON
PROJECT NO.:MLP-04
Current Data Parameters
NAME atgmi109.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131204
Time 17.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 301.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Chemical Formula: C30H22O
Exact Mass: 398.1671
200
180
160
140
120
100
80
60
40
20
0
ppm





2-(5,6-dimethoxy-2,3-dihydro-1*H*-inden-1-ylidene)-5,6-dimethoxy-2,3-dihydro-1*H*-inden-1-one (12b):





CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters

NAME	atgmi99.p
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20130903
Time	11.24
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	406.4
DW	81.000 usec
DE	6.00 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1

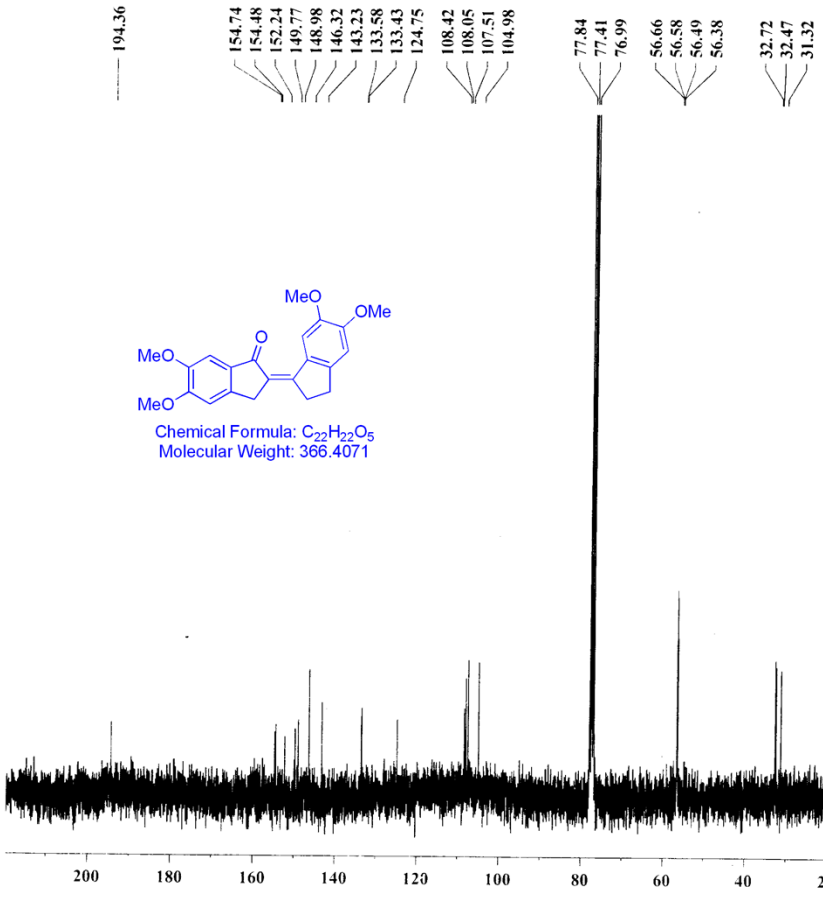
======== CHANNEL f1 ========

NUC1	1H
P1	6.78 usec
PL1	4.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1299974 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

CIMAP-CARBON
PROJECT NO.:MLP-04



Current Data Parameters
NAME atgmi99.p
EXPNO 2
PROCNO 1

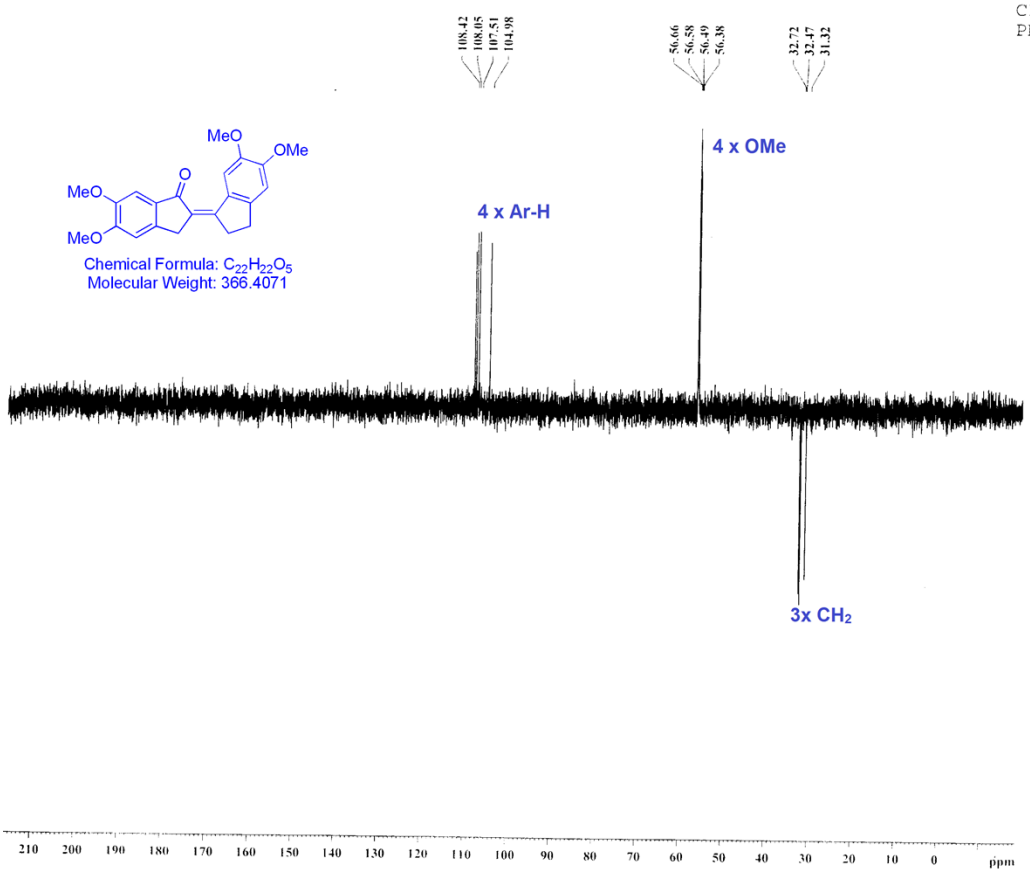
F2 - Acquisition Parameters
Date_ 20130906
Time 12.59
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 946
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 299.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CIMAP-DEPT 135
PROJECT NO.:ILP-13



Current Data Parameters
NAME atgmi99.p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130906
Time 14.28
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 955
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Central Institute of Medicinal and Aromatic Plants
(Analytical Chemistry Division)
Lucknow-226015

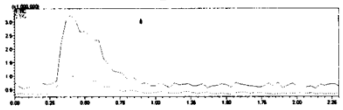
ANALYTICAL TEST REPORT

Sample Information for Direct Mass Analysis of Isolates/synthetic molecule

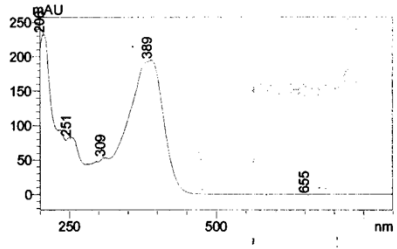
Sample Code : ATG-MI-99
Solubility : MeOH
Name of the Scientist : Dr ATUL GUPTA

Project Code: MLP-0
Mass Range: 200-40

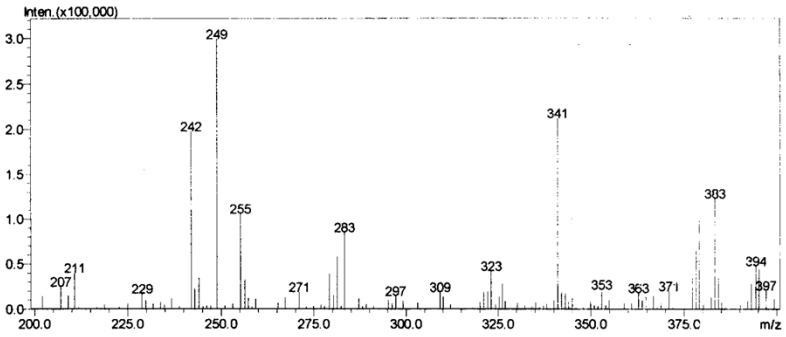
Mass chromatogram



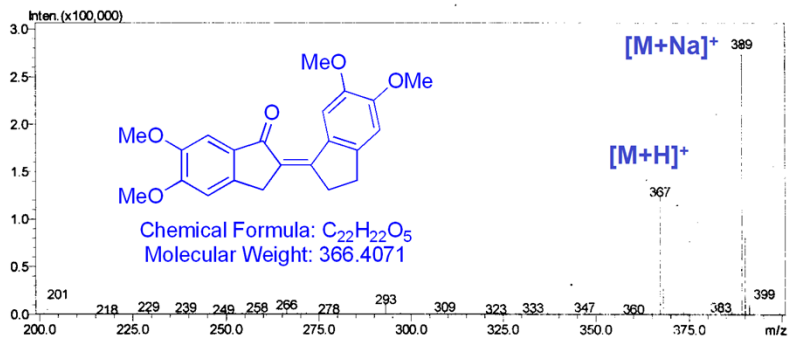
UV-Spectra

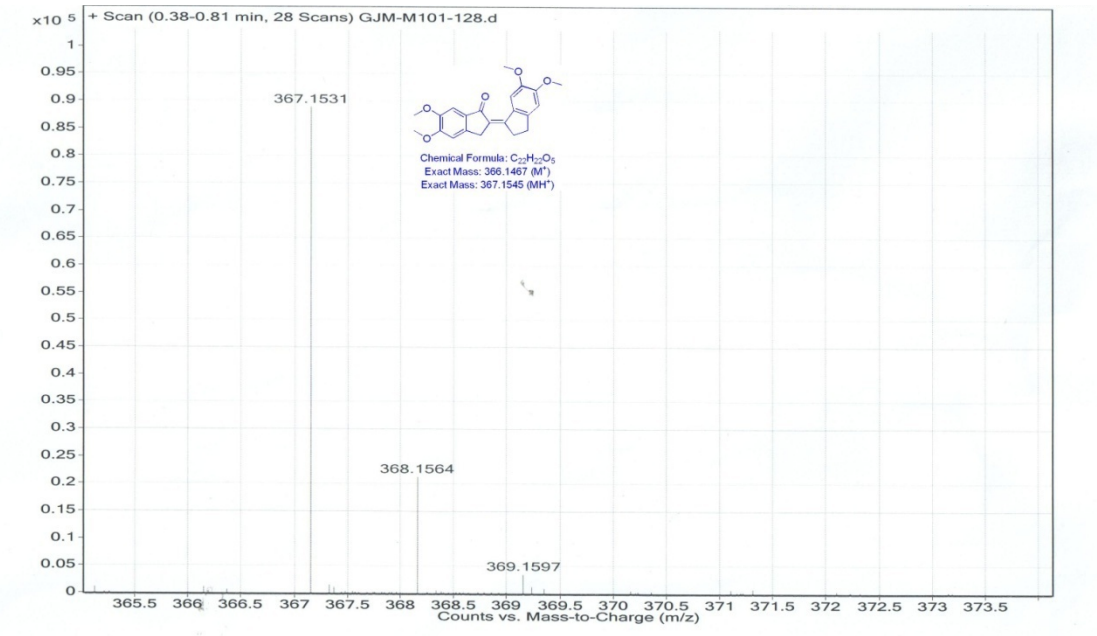


ESI-

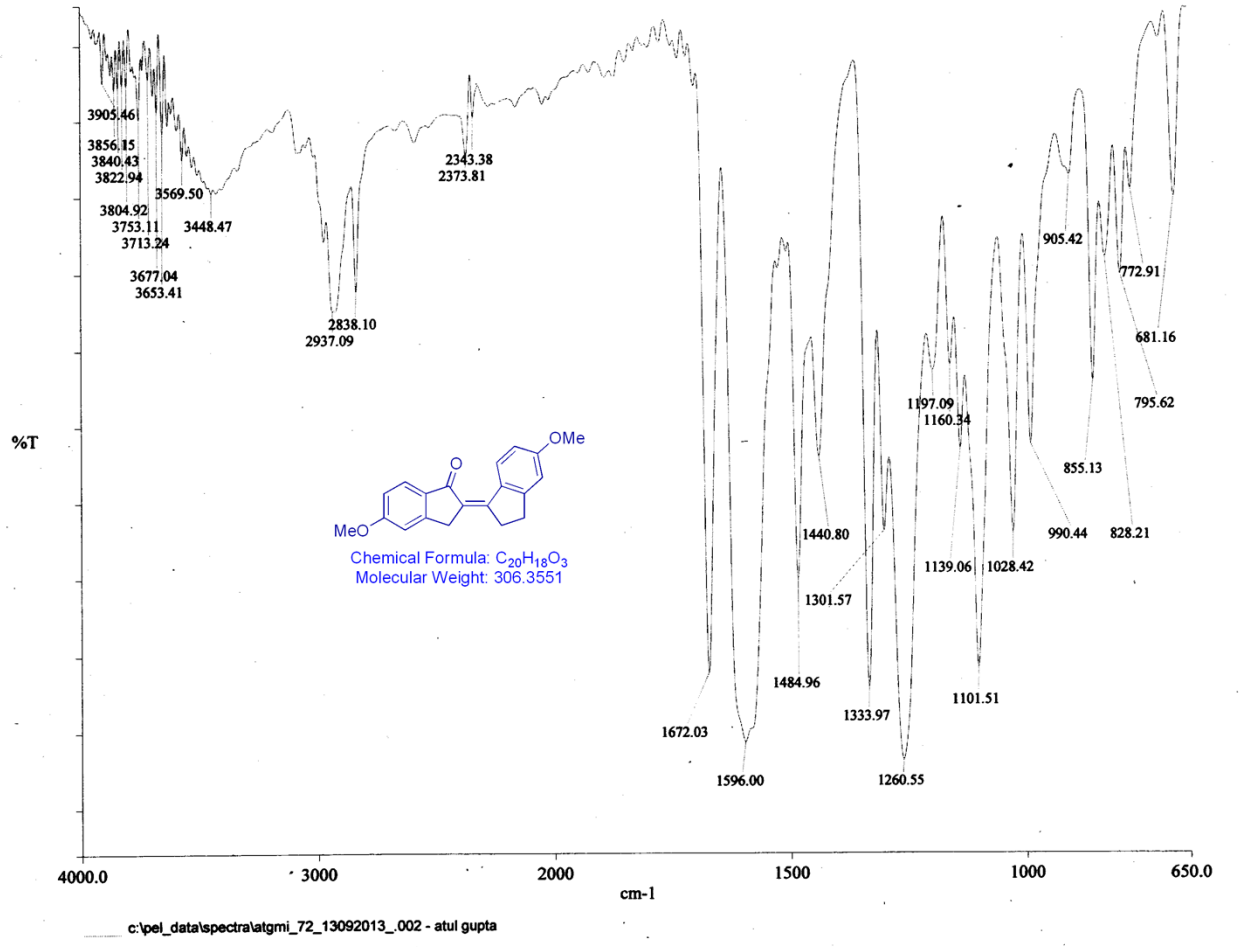


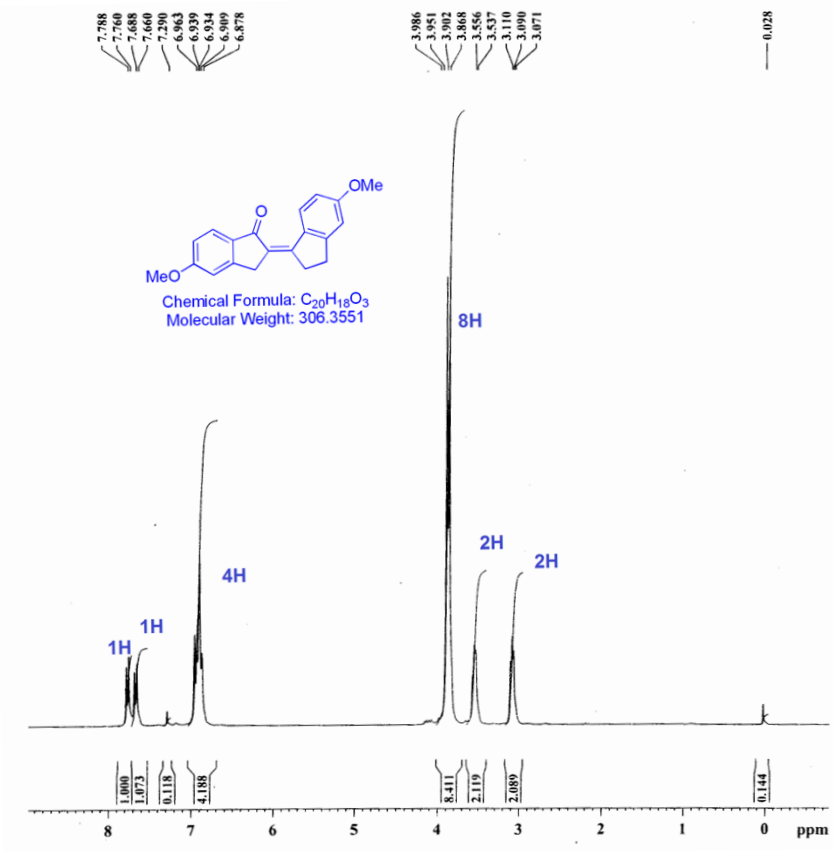
ESI+





5-methoxy-2-(5-methoxy-2,3-dihydro-1*H*-inden-1-ylidene)-2,3-dihydro-1*H*-inden-1-one (12c):





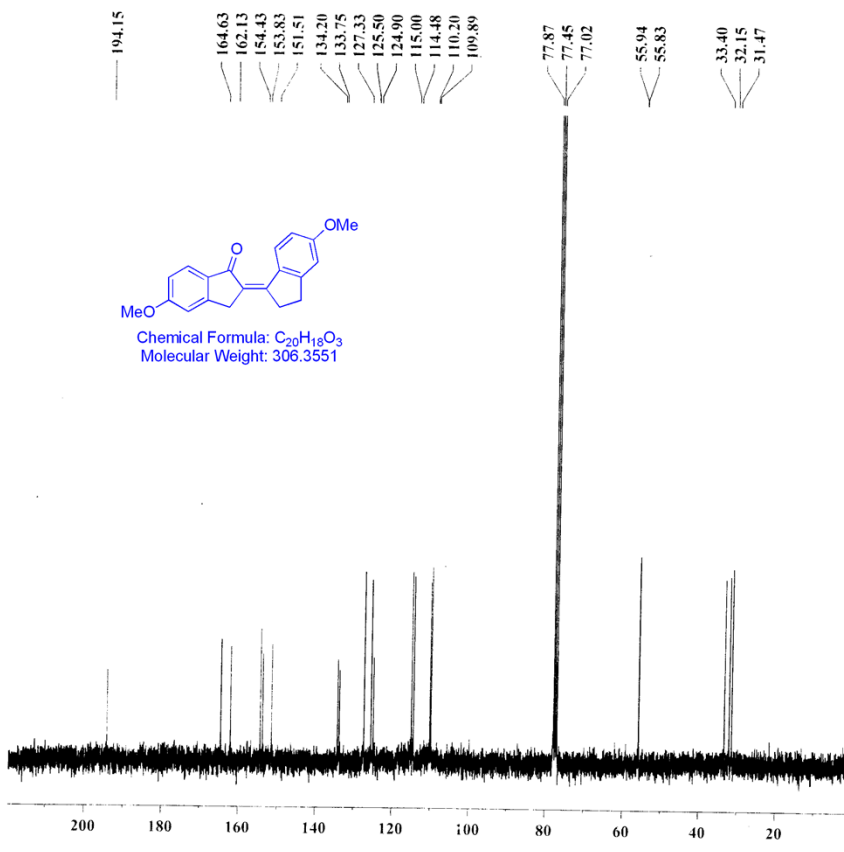
CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters
NAME atgmi72.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130903
Time 11.30
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1299974 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



CIMAP-CARBON
PROJECT NO.:MLP-04

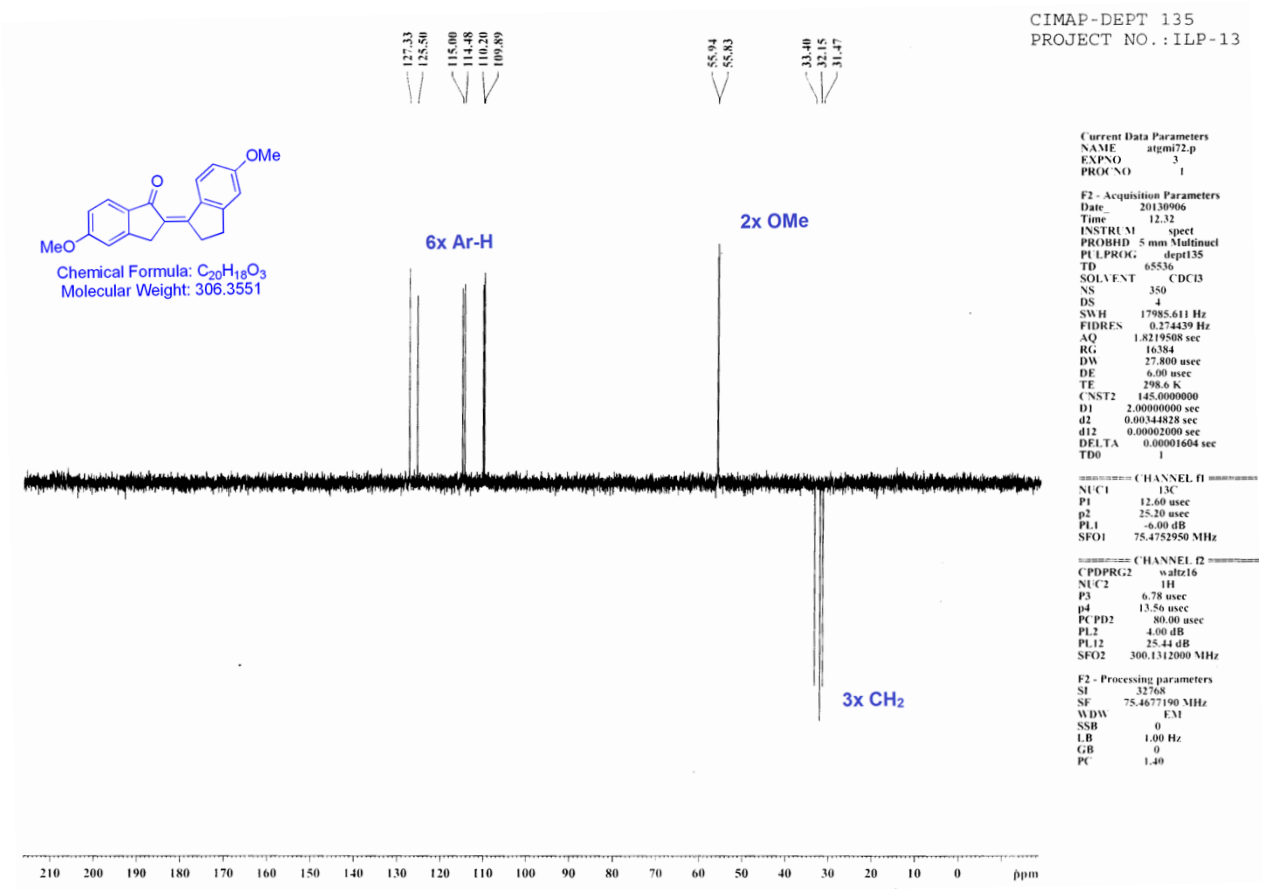
Current Data Parameters
NAME atgmi72.p
EXPNO 2
PROCNO 1

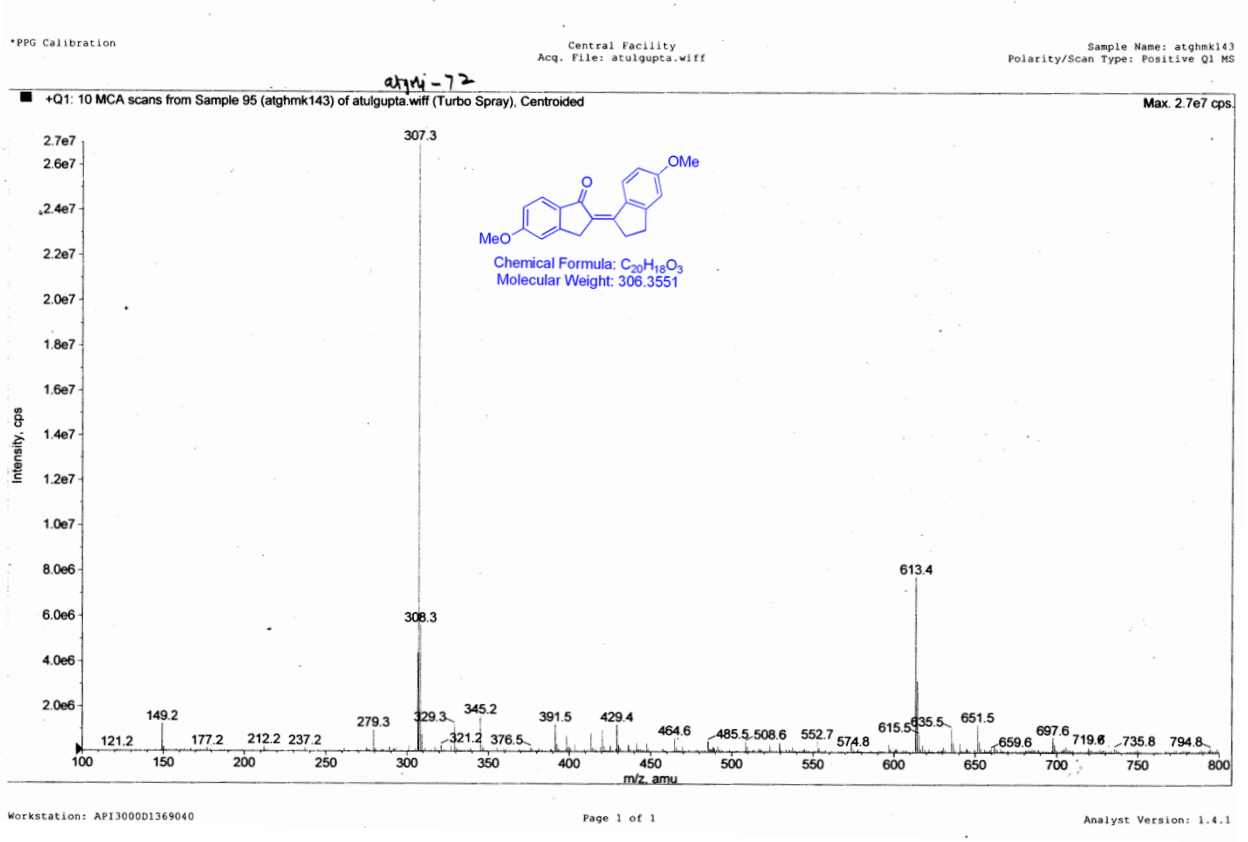
F2 - Acquisition Parameters
Date_ 20130906
Time 12.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 356
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

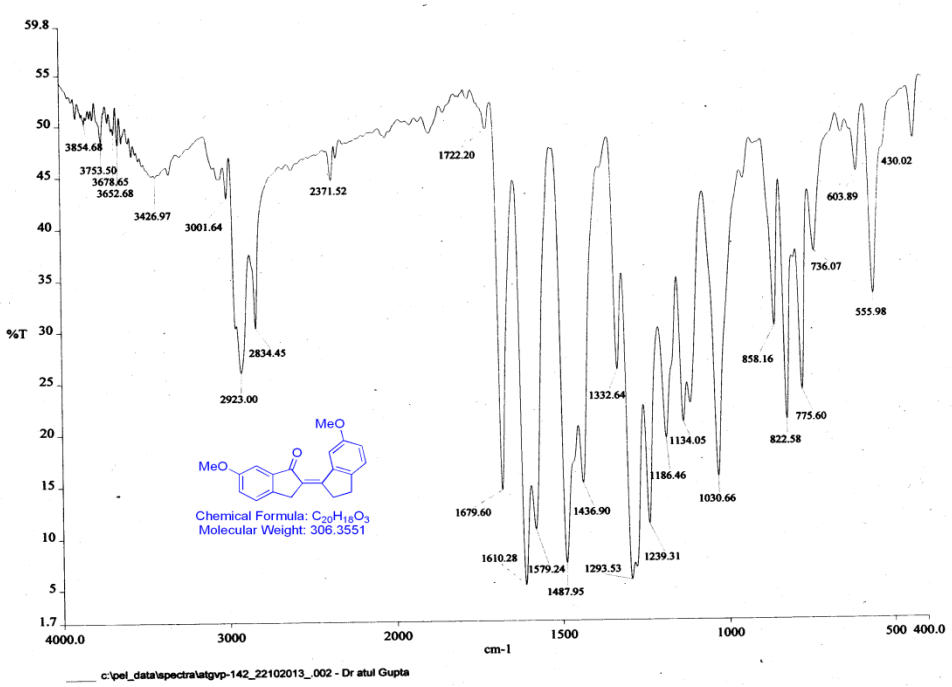
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

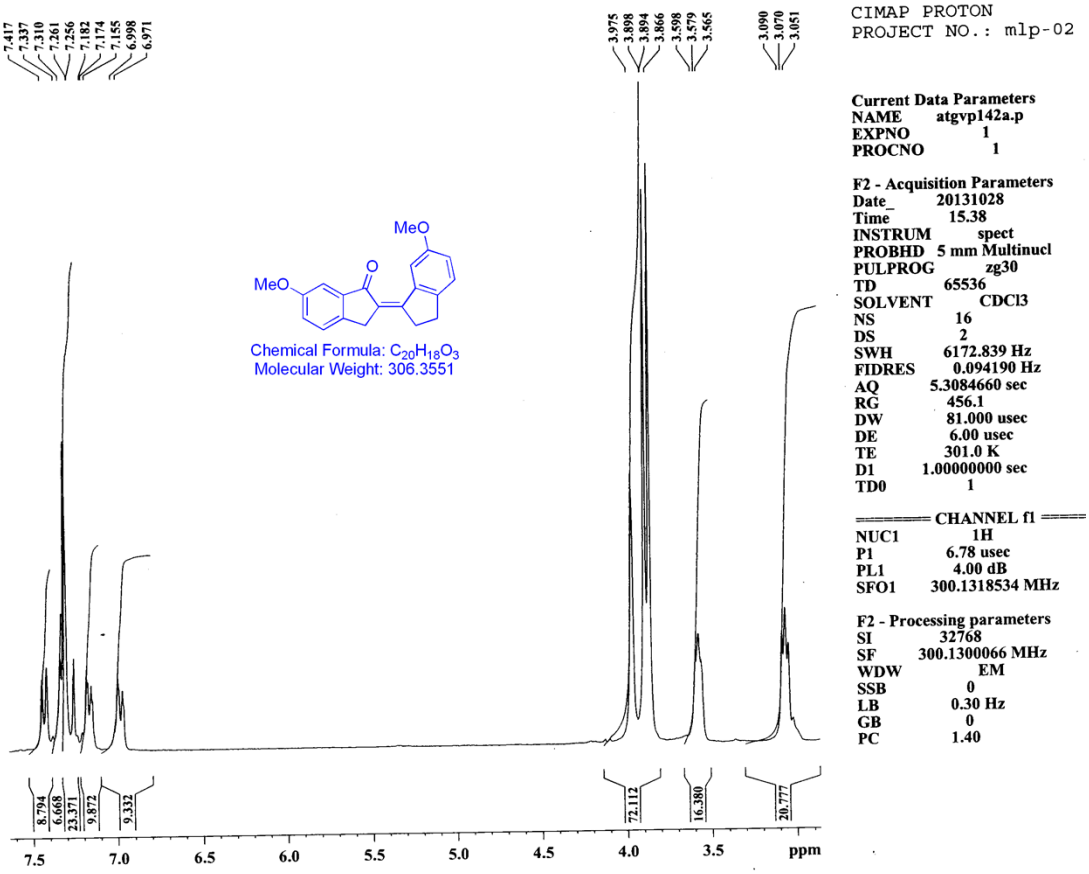
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

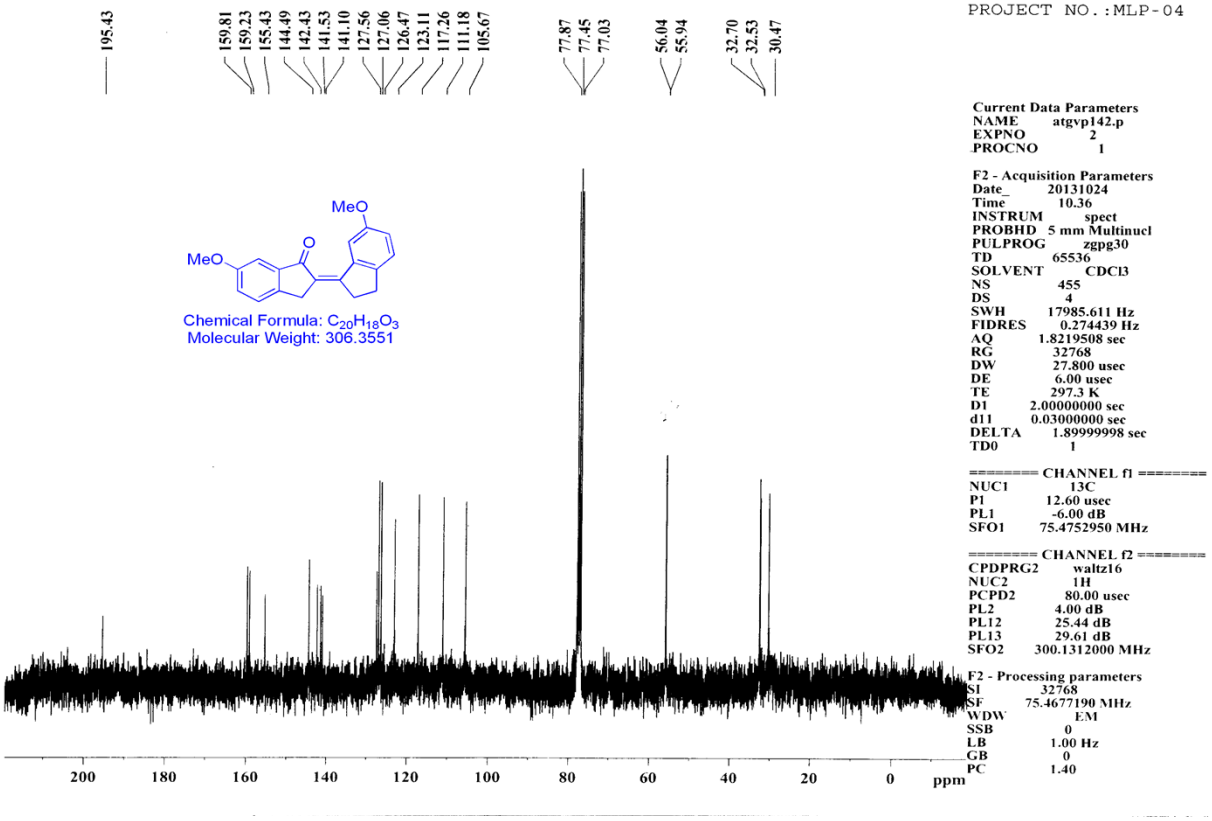

CIMAP-DEPT 135
PROJECT NO.:ILP-13
127.33
125.50
115.00
114.48
110.20
109.89
55.94
55.83
33.40
32.15
31.47
OMe
O
MeO
Chemical Formula: C20H18O3
Molecular Weight: 306.3551
6x Ar-H
2x OMe
3x CH2
Current Data Parameters
NAME atgmi72.p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130906
Time 12.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 350
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 298.6 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

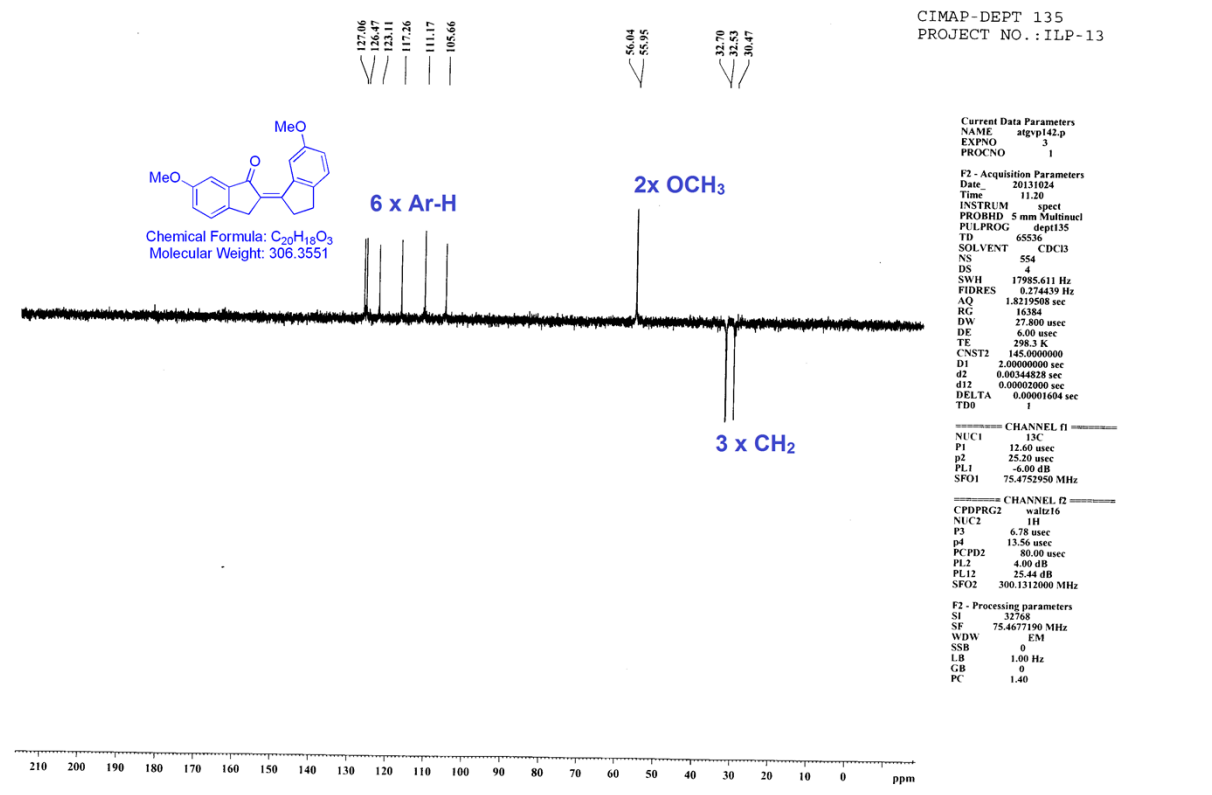

*PPG Calibration
Central Facility
Acq. File: atulgupta.wiff
Sample Name: atghmk143
Polarity/Scan Type: Positive Q1 MS
atghmj-72
+Q1: 10 MCA scans from Sample 95 (atghmk143) of atulgupta.wiff (Turbo Spray), Centroided
Max. 2.7e7 cps.
307.3
308.3
613.4
OMe
O
MeO
Chemical Formula: C20H18O3
Molecular Weight: 306.3551
Intensity, cps
m/z, amu
Workstation: API3000D1369040
Page 1 of 1
Analyst Version: 1.4.1

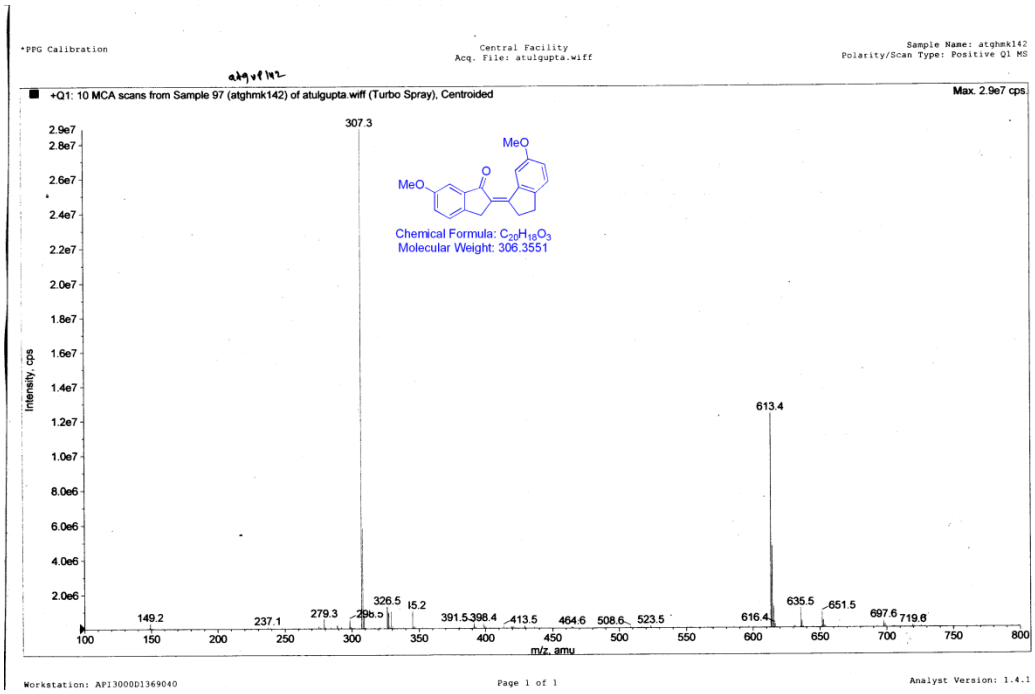
6-methoxy-2-(6-methoxy-2,3-dihydro-1*H*-inden-1-ylidene)-2,3-dihydro-1*H*-inden-1-one (12d):



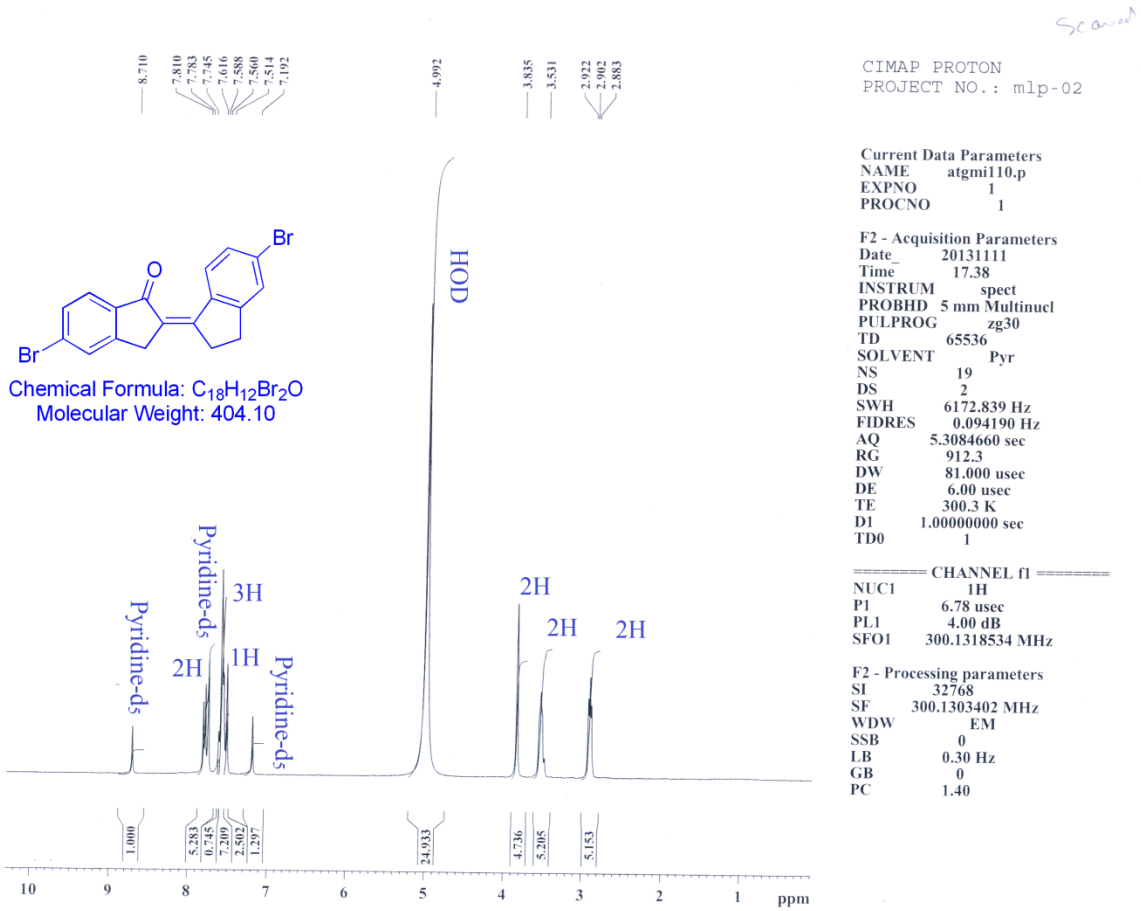



CIMAP-CARBON
PROJECT NO.:MLP-04
195.43
159.81
159.23
155.43
144.49
142.43
141.53
141.10
127.56
127.06
126.47
123.11
117.26
111.18
105.67
77.87
77.45
77.03
56.04
55.94
32.70
32.53
30.47
MeO
MeO
O
Chemical Formula: C20H18O3
Molecular Weight: 306.3551
Current Data Parameters
NAME atgvp142.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 10.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 455
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

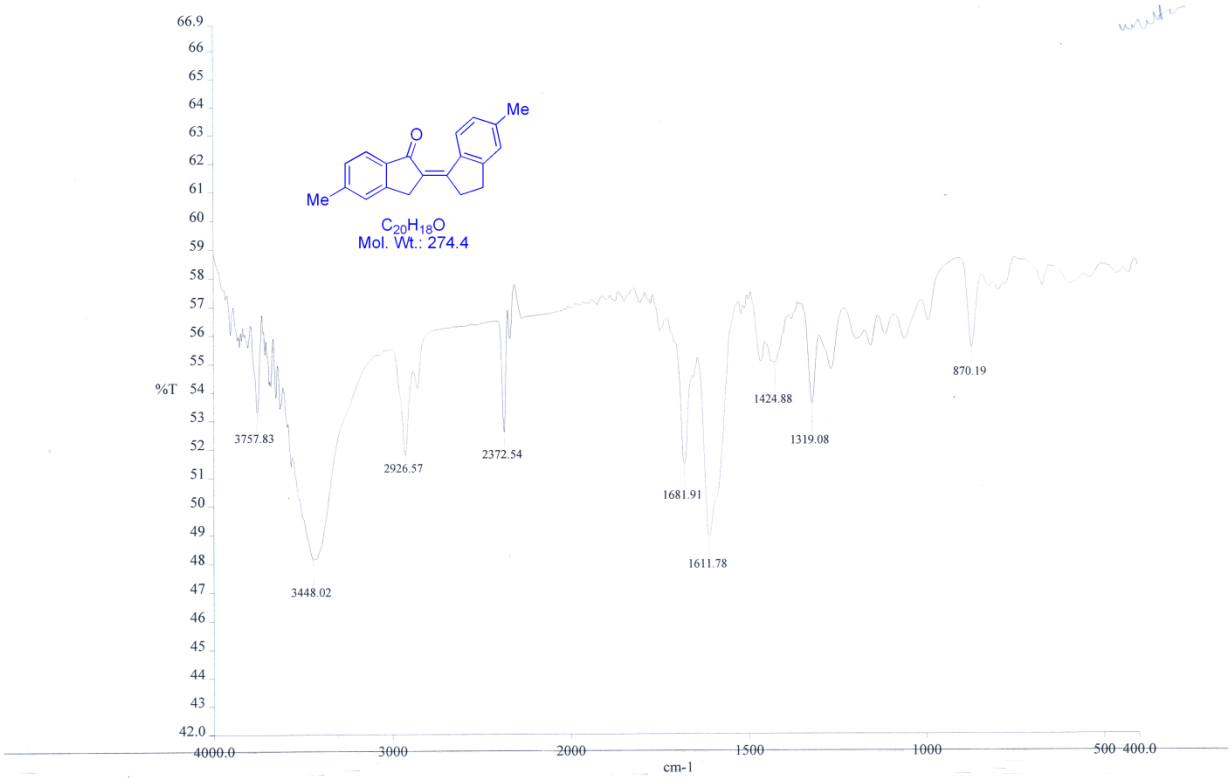

CIMAP-DEPT 135
PROJECT NO.:ILP-13
127.06
126.47
123.11
117.26
111.17
105.66
56.04
55.95
32.70
32.53
30.47
MeO
MeO
O
Chemical Formula: C20H18O3
Molecular Weight: 306.3551
6 x Ar-H
2x OCH3
3 x CH2
Current Data Parameters
NAME atgvp142.p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 11.20
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 554
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 298.3 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



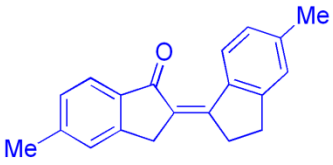
5-bromo-2-(5-bromo-2,3-dihydro-1*H*-inden-1-ylidene)-2,3-dihydro-1*H*-inden-1-one (12e):



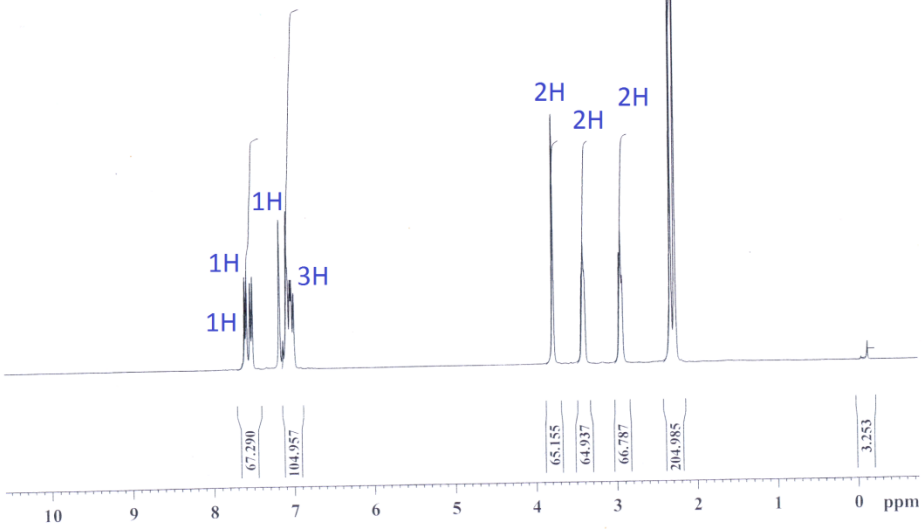
5-methyl-2-(5-methyl-2,3-dihydro-1*H*-inden-1-ylidene)-2,3-dihydro-1*H*-inden-1-one (12f):



CIMAP PROTON
PROJECT NO.: mlp-02



Chemical Formula: $C_{20}H_{18}O$
Molecular Weight: 274.36



Current Data Parameters

NAME	atgmi112.p
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

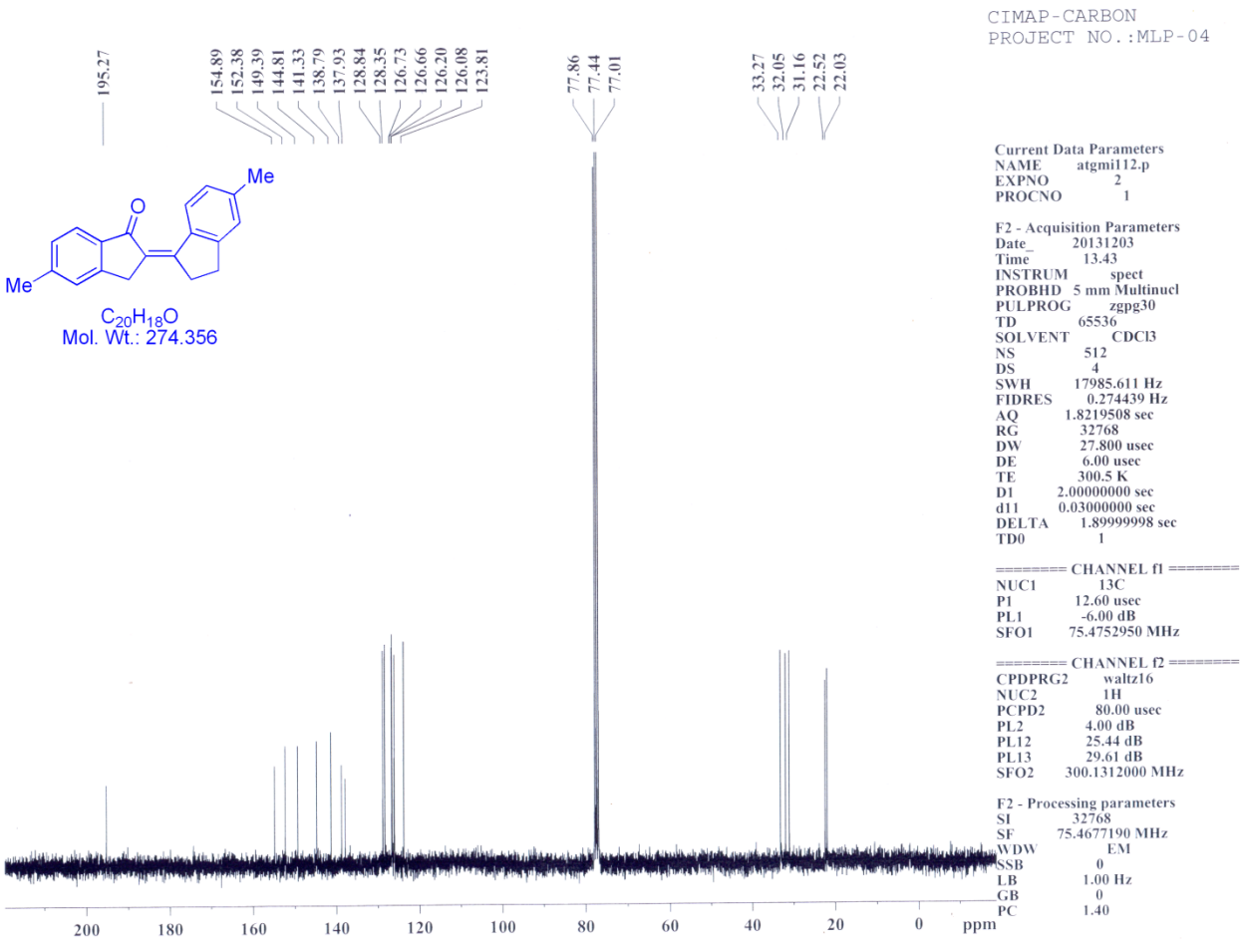
Date_	20131114
Time	10.10
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	17
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	143.7
DW	81.000 usec
DE	6.00 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1

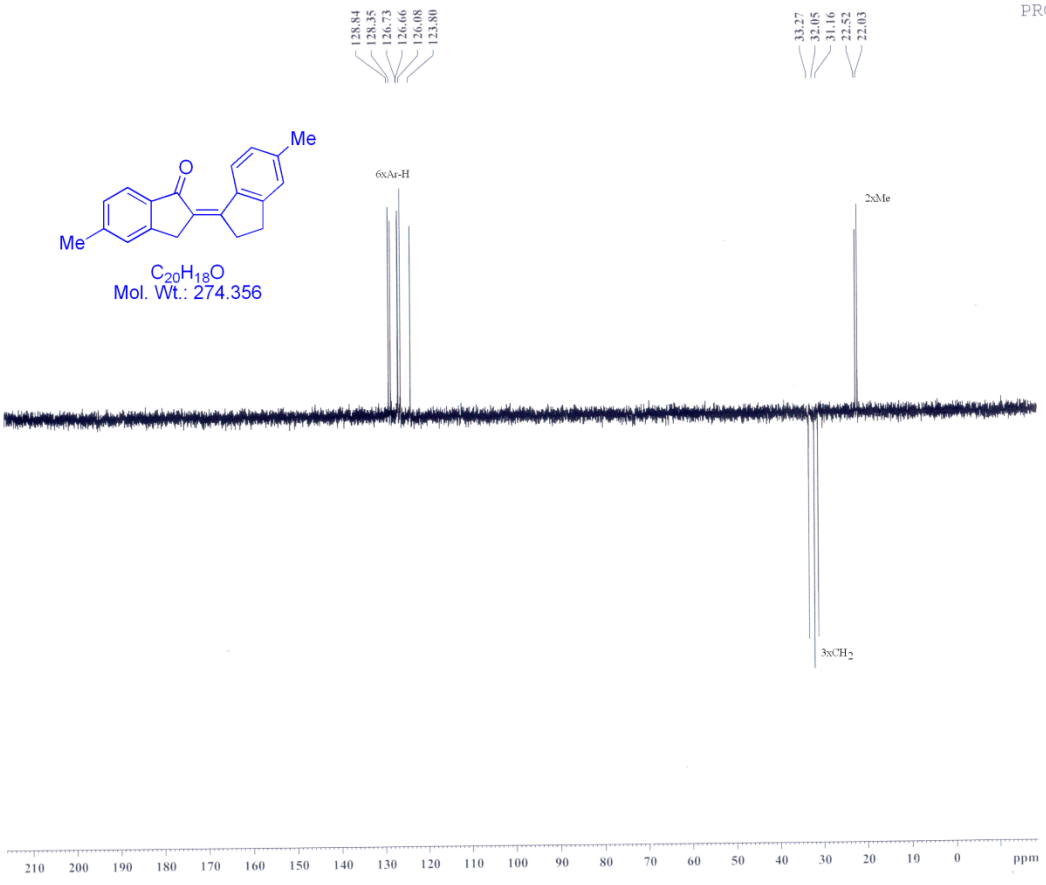
======== CHANNEL f1 ======

NUC1	1H
P1	6.78 usec
PL1	4.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300434 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40


CIMAP-CARBON
PROJECT NO.:MLP-04
195.27
154.89
152.38
149.39
144.81
141.33
138.79
137.93
128.84
128.35
126.73
126.66
126.20
126.08
123.81
77.86
77.44
77.01
33.27
32.05
31.16
22.52
22.03
Me
O
Me
C20H18O
Mol. Wt.: 274.356
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME atgmi112.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 13.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME atgmi112.p
EXPNO 3
PROCNO 1

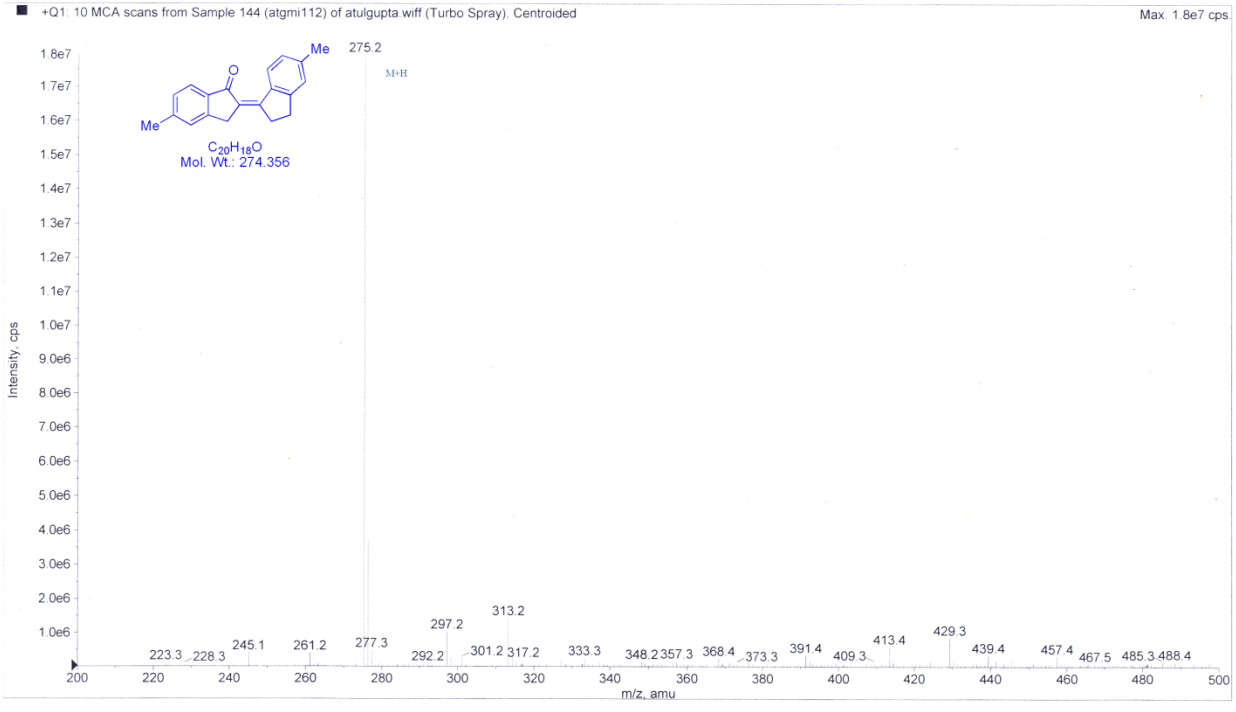
F2 - Acquisition Parameters
Date_ 20131203
Time 14.08
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 393
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.5 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 6.78 usec
p4 13.56 usec
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
SFO2 300.1312000 MHz

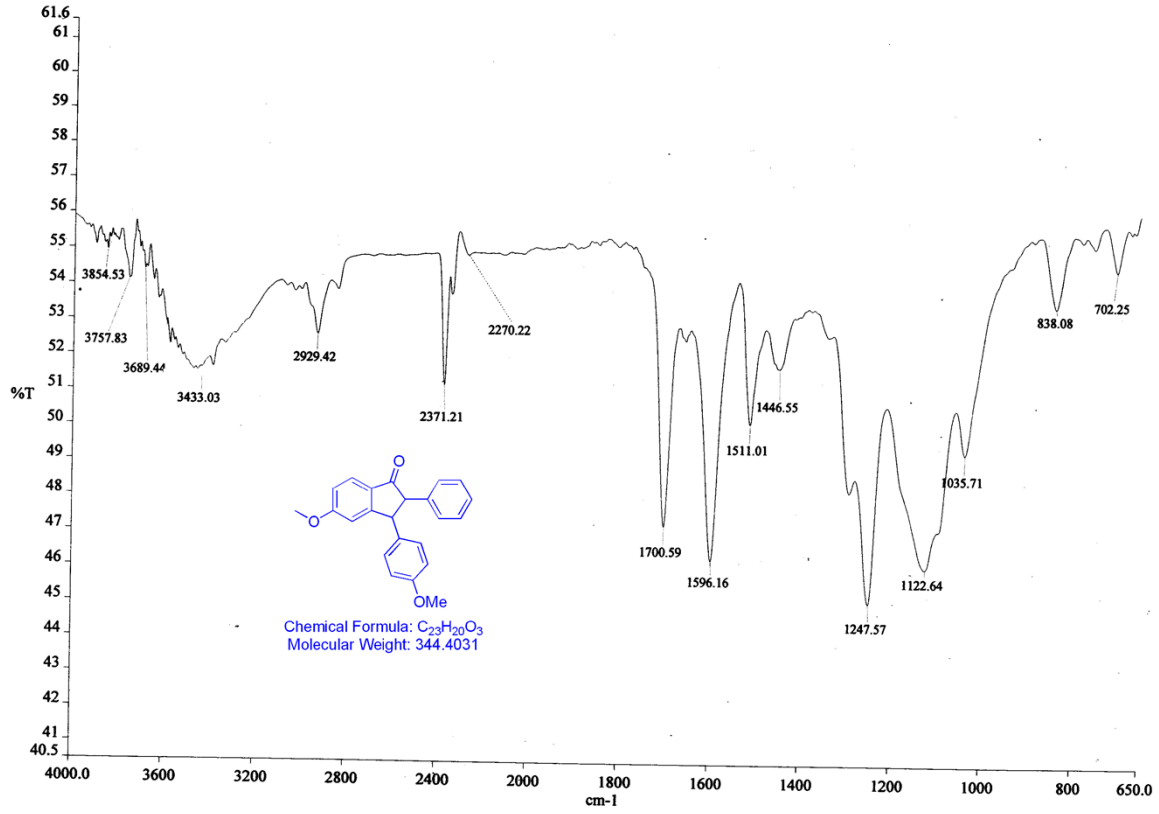
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

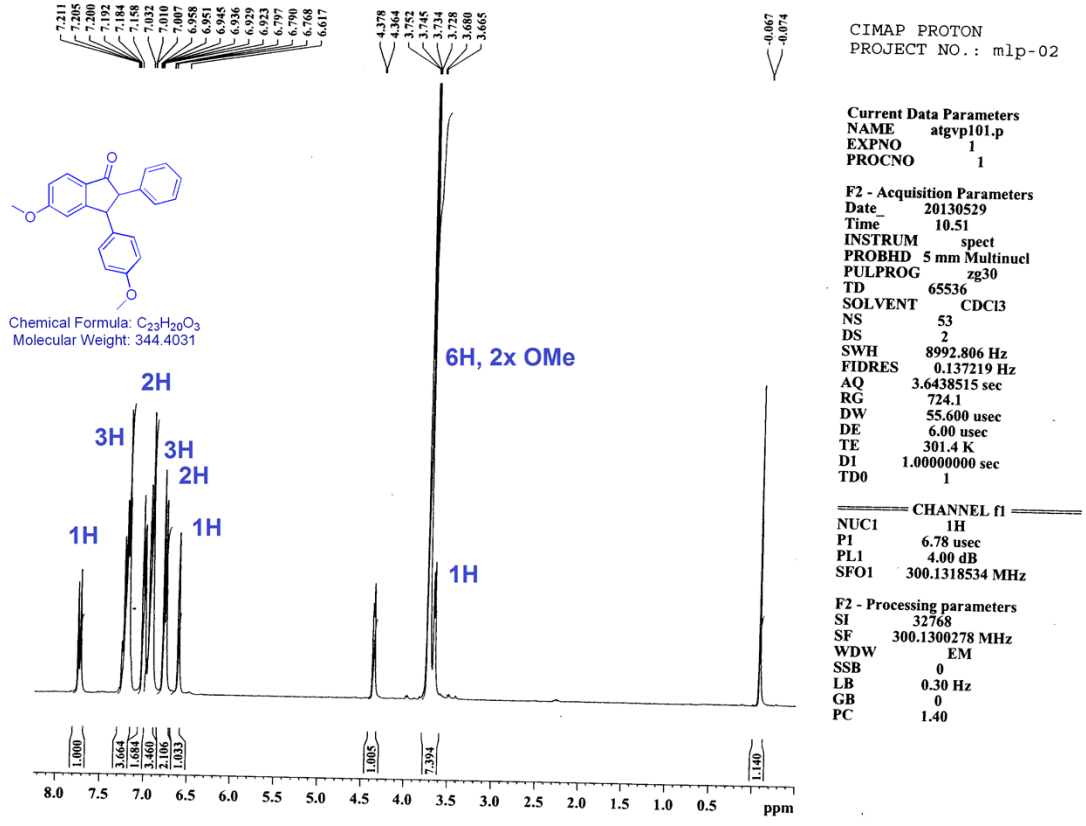
*FPG Calibration
Central Facility
Acq. File: atulgupta.wiff
Sample Name: atgmi112
Polarity/Scan Type: Positive Q1 MS



Workstation: API3000D1369040
Page 1 of 1
Analyst Version: 1.4.1

2,3-dihydro-5-methoxy-3-(4-methoxyphenyl)-2-phenylinden-1-one (15):





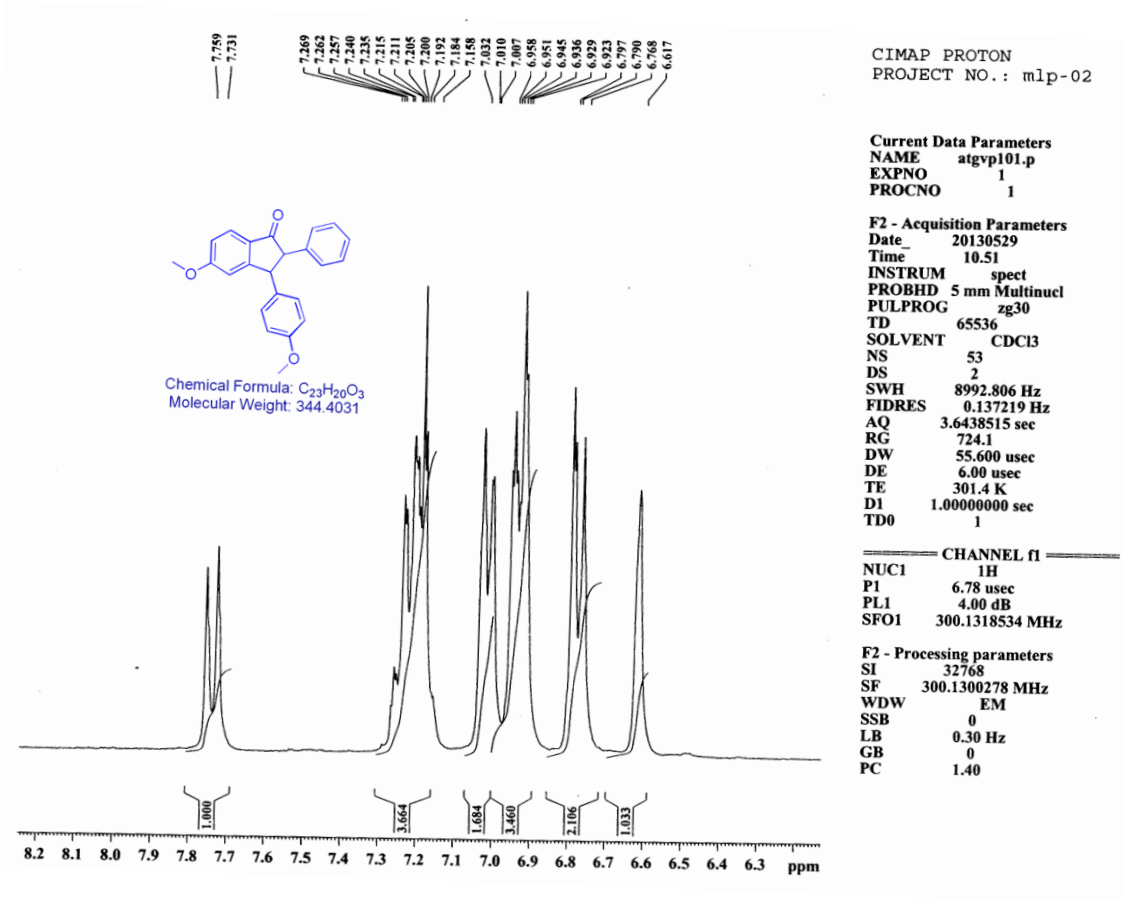
CIMAP PROTON
PROJECT NO.: mlp-02

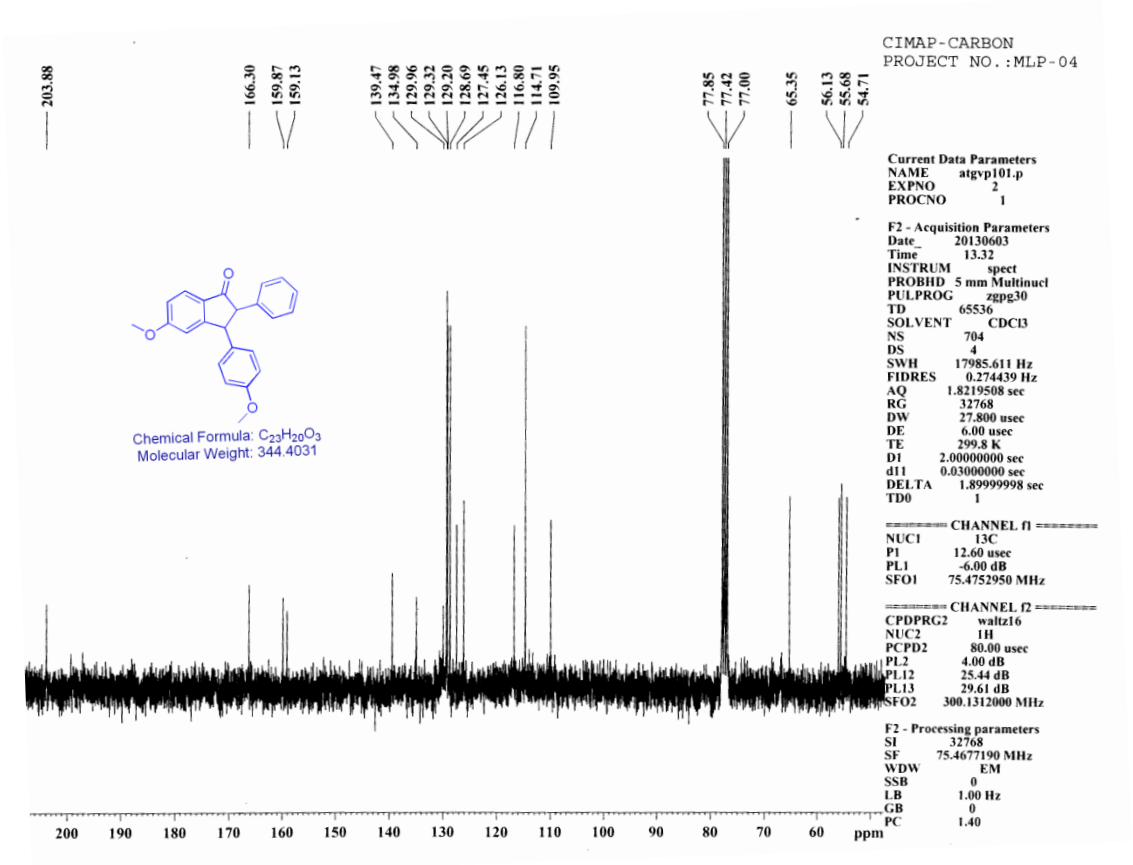
Current Data Parameters
NAME atgvp101.p
EXPNO 1
PROCNO 1

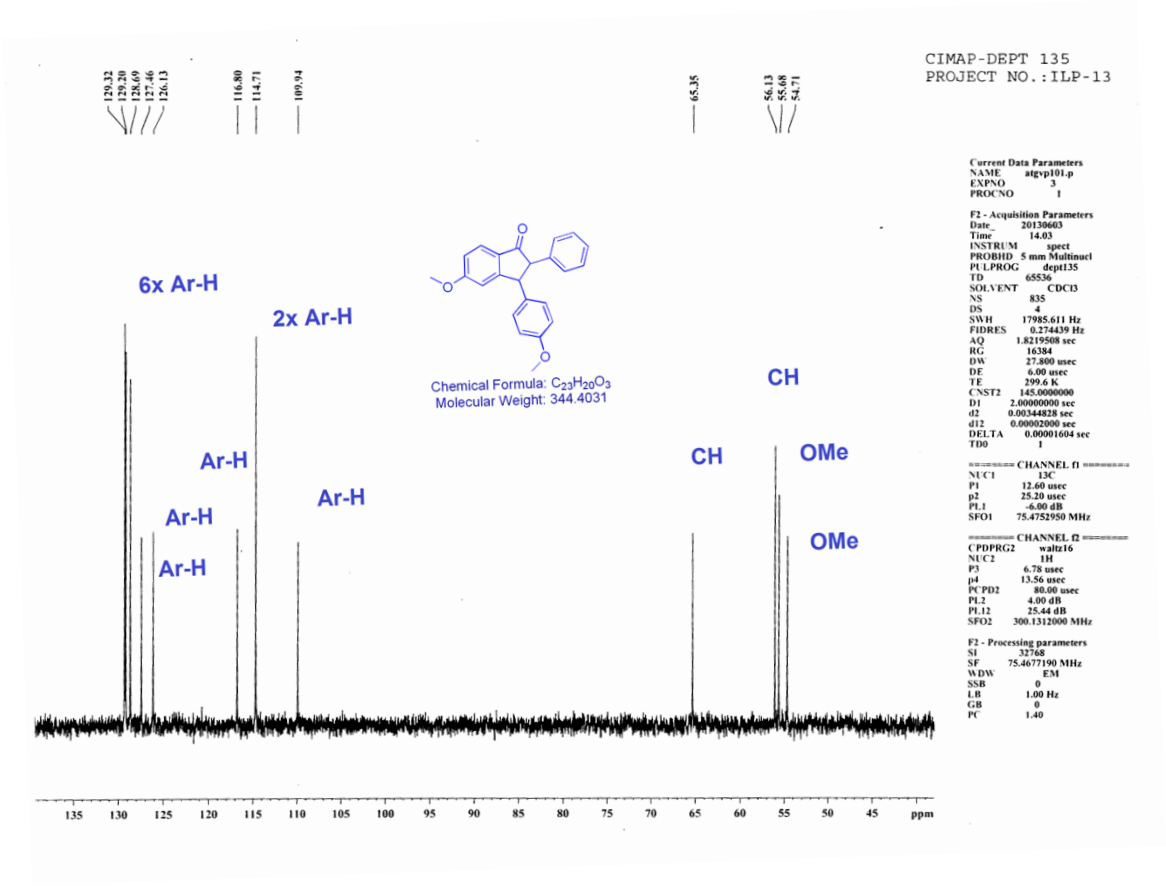
F2 - Acquisition Parameters
Date_ 20130529
Time 10.51
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 53
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 724.1
DW 55.600 usec
DE 6.00 usec
TE 301.4 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

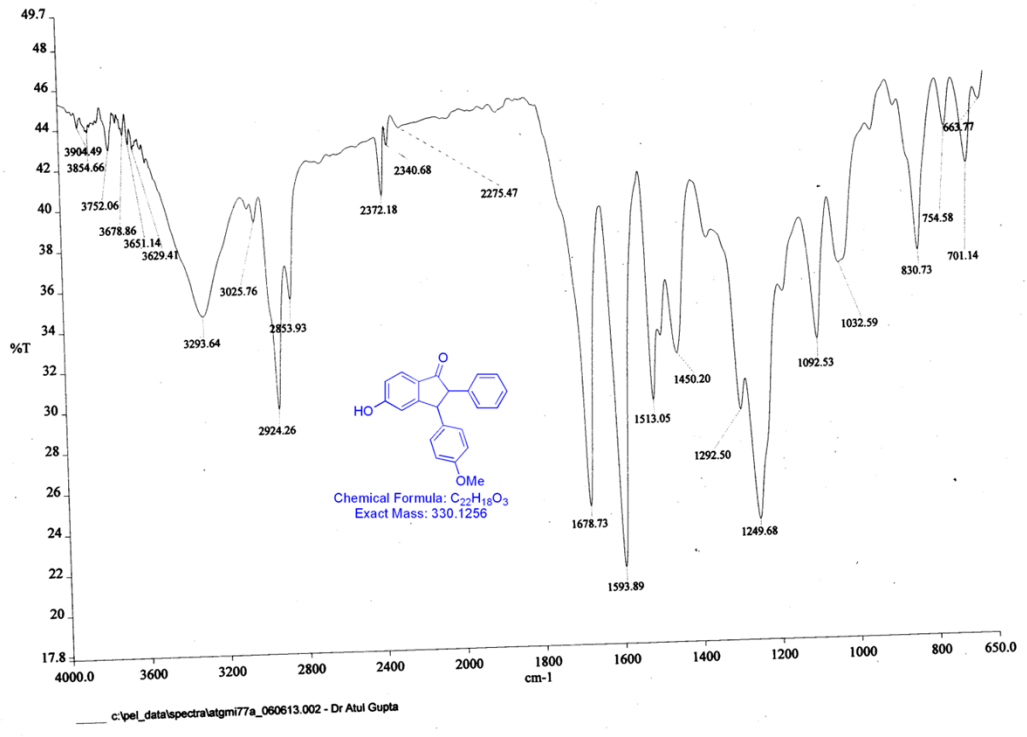
F2 - Processing parameters
SI 32768
SF 300.1300278 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

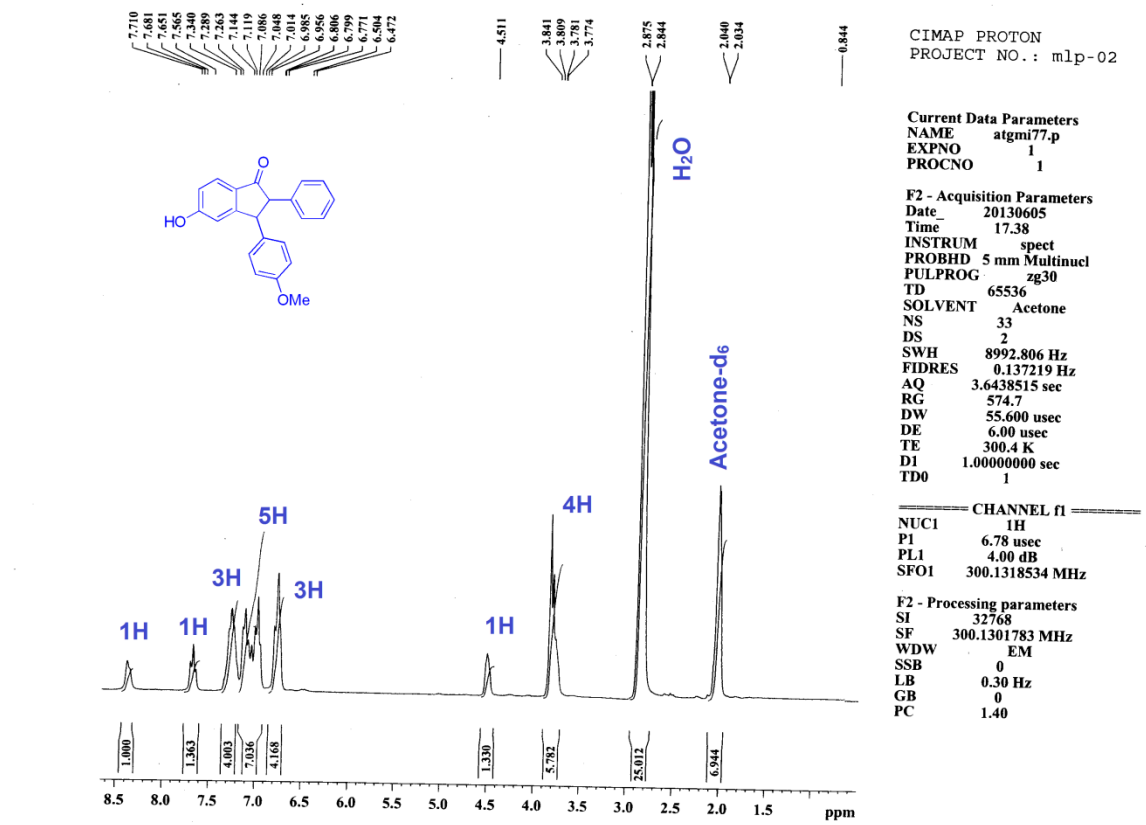

7.759
7.731
7.269
7.262
7.257
7.240
7.235
7.215
7.211
7.205
7.200
7.192
7.184
7.158
7.032
7.010
7.007
6.958
6.951
6.945
6.936
6.929
6.923
6.797
6.790
6.768
6.617
CIMAP PROTON
PROJECT NO.: mlp-02
Chemical Formula: C23H20O3
Molecular Weight: 344.4031
Current Data Parameters
NAME atgvp101.p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130529
Time 10.51
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 53
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 724.1
DW 55.600 usec
DE 6.00 usec
TE 301.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300278 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
1.000
3.664
1.684
3.460
2.106
1.033
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 ppm

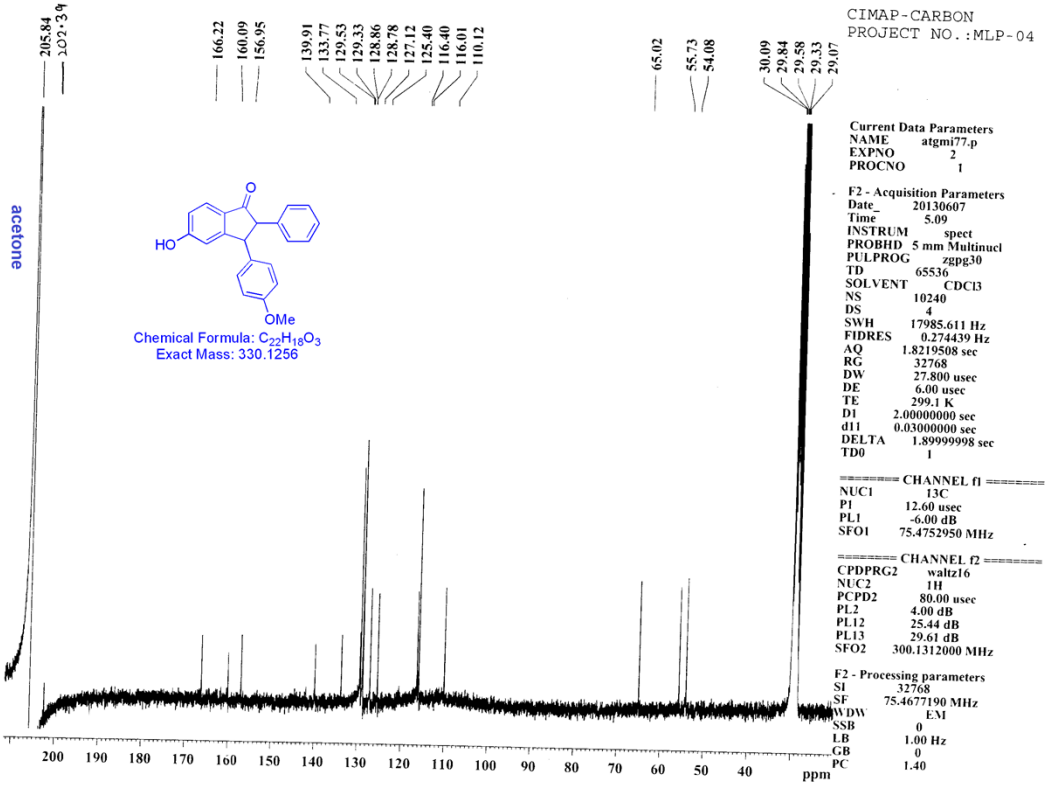

203.88
166.30
159.87
159.13
139.47
134.98
129.96
129.32
129.20
128.69
127.45
126.13
116.80
114.71
109.95
77.85
77.42
77.00
65.35
56.13
55.68
54.71
CIMAP-CARBON
PROJECT NO.:MLP-04
Chemical Formula: C23H20O3
Molecular Weight: 344.4031
Current Data Parameters
NAME atgvp101.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130603
Time 13.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 704
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 299.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 ppm

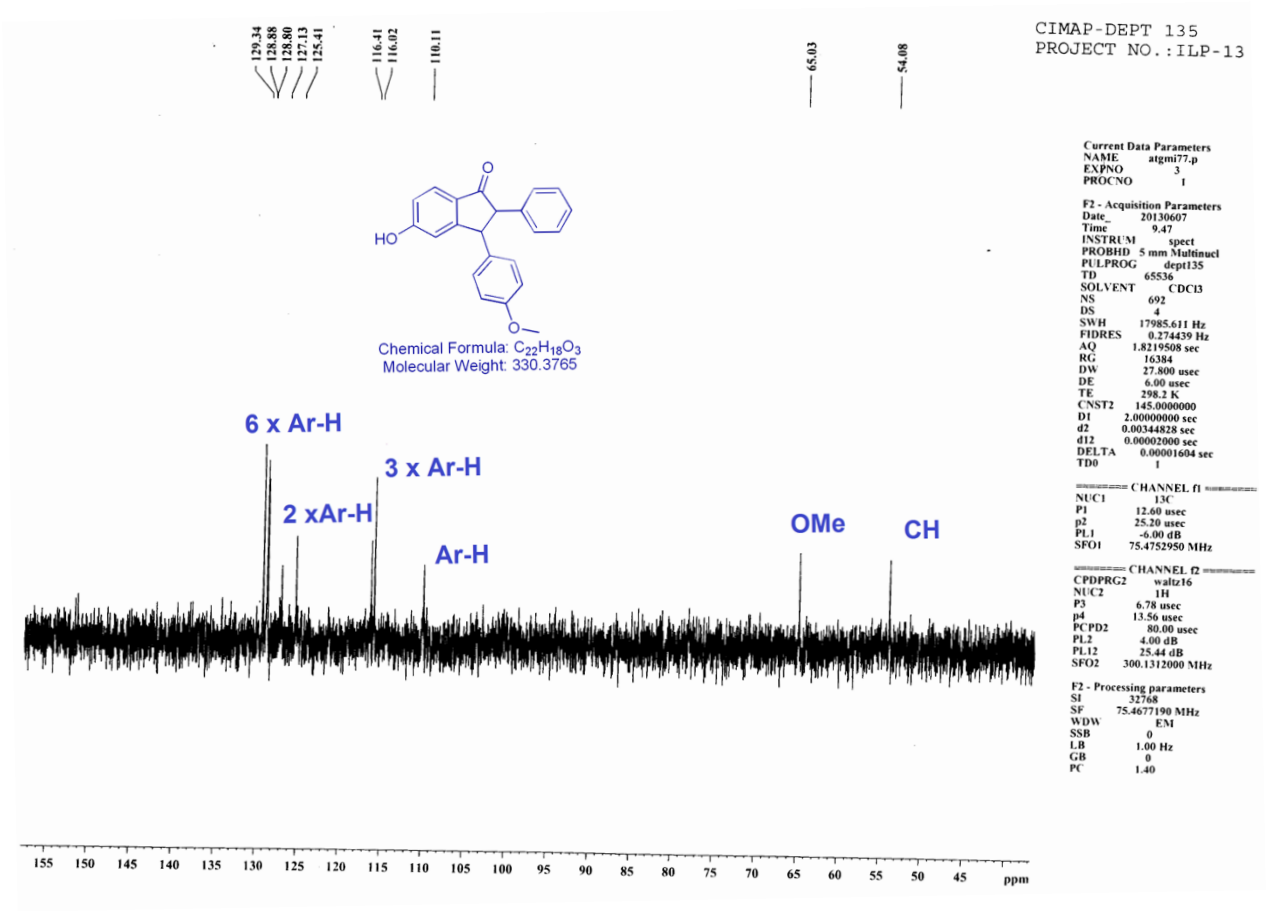


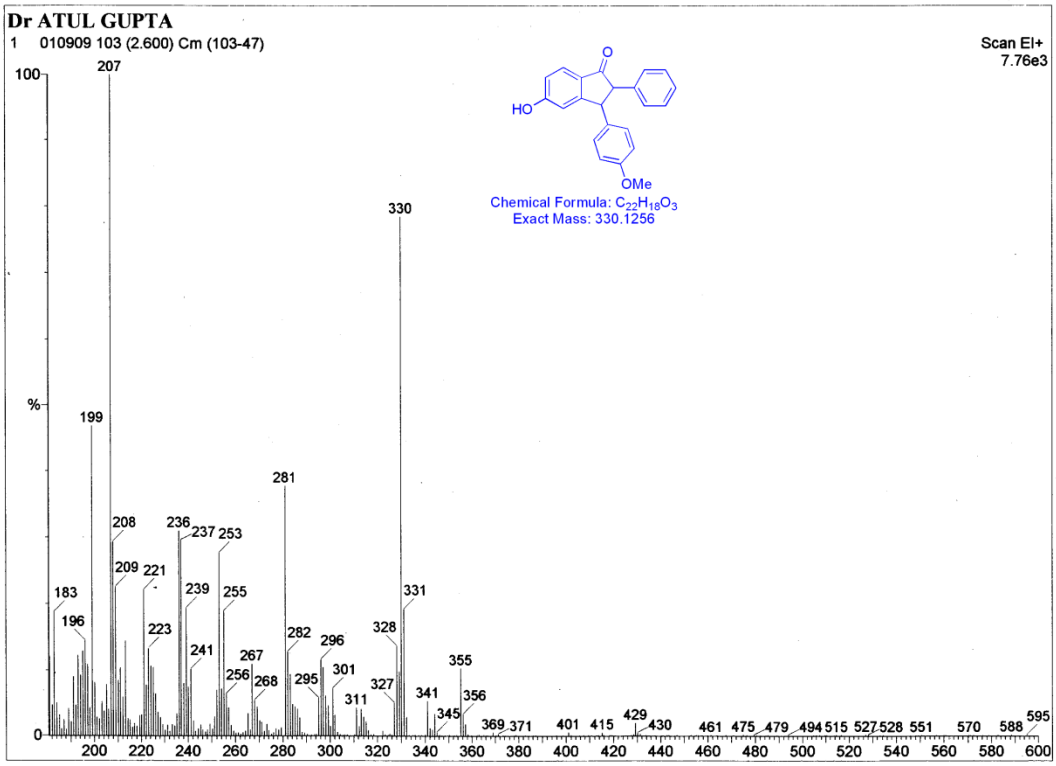
2,3-dihydro-5-hydroxy-3-(4-methoxyphenyl)-2-phenylinden-1-one (16).



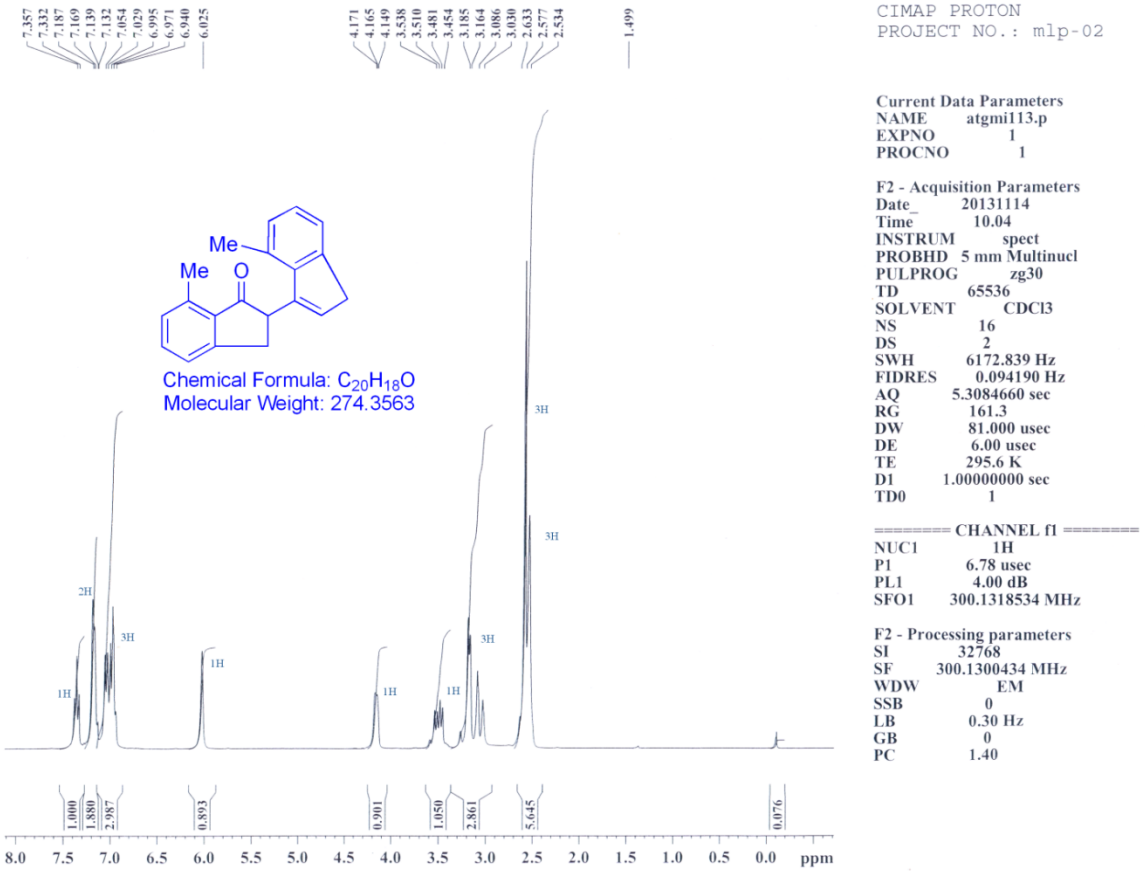

7.710
7.681
7.651
7.565
7.340
7.289
7.263
7.144
7.119
7.086
7.048
7.014
6.985
6.956
6.806
6.799
6.771
6.504
6.472
4.511
3.841
3.809
3.781
3.774
2.875
2.844
2.040
2.034
0.844
H2O
Acetone-d6
HO
OMe
5H
4H
3H
3H
1H
1H
1H
1.000
1.363
4.003
7.036
4.168
1.330
5.782
25.012
6.944
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
ppm
CIMAP PROTON
PROJECT NO.: mlp-02
Current Data Parameters
NAME atgmi77.p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130605
Time 17.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 33
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 574.7
DW 55.600 usec
DE 6.00 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1301783 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


205.84
202.39
166.22
160.09
156.95
139.91
133.77
129.53
129.33
128.86
128.78
127.12
125.40
116.40
116.01
110.12
65.02
55.73
54.08
30.09
29.84
29.58
29.33
29.07
acetone
HO
OMe
Chemical Formula: C22H18O3
Exact Mass: 330.1256
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40
ppm
CIMAP-CARBON
PROJECT NO.:MLP-04
Current Data Parameters
NAME atgmi77.p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130607
Time 5.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10240
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 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 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





2-(2,3-dihydro-4-methyl-1H-inden-3-yl)-7-methyl-1H-inden-1-one (17):



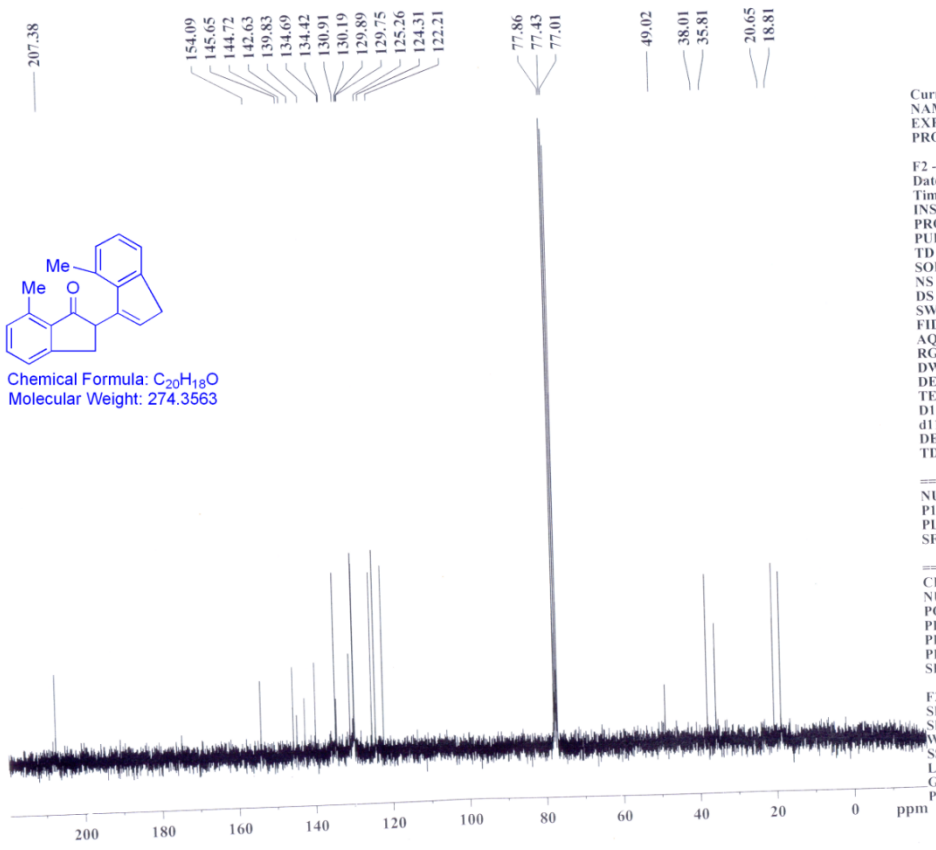
CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters
NAME atgmi113.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131114
Time 10.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 6.78 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300434 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



CIMAP-CARBON
PROJECT NO.:MLP-04

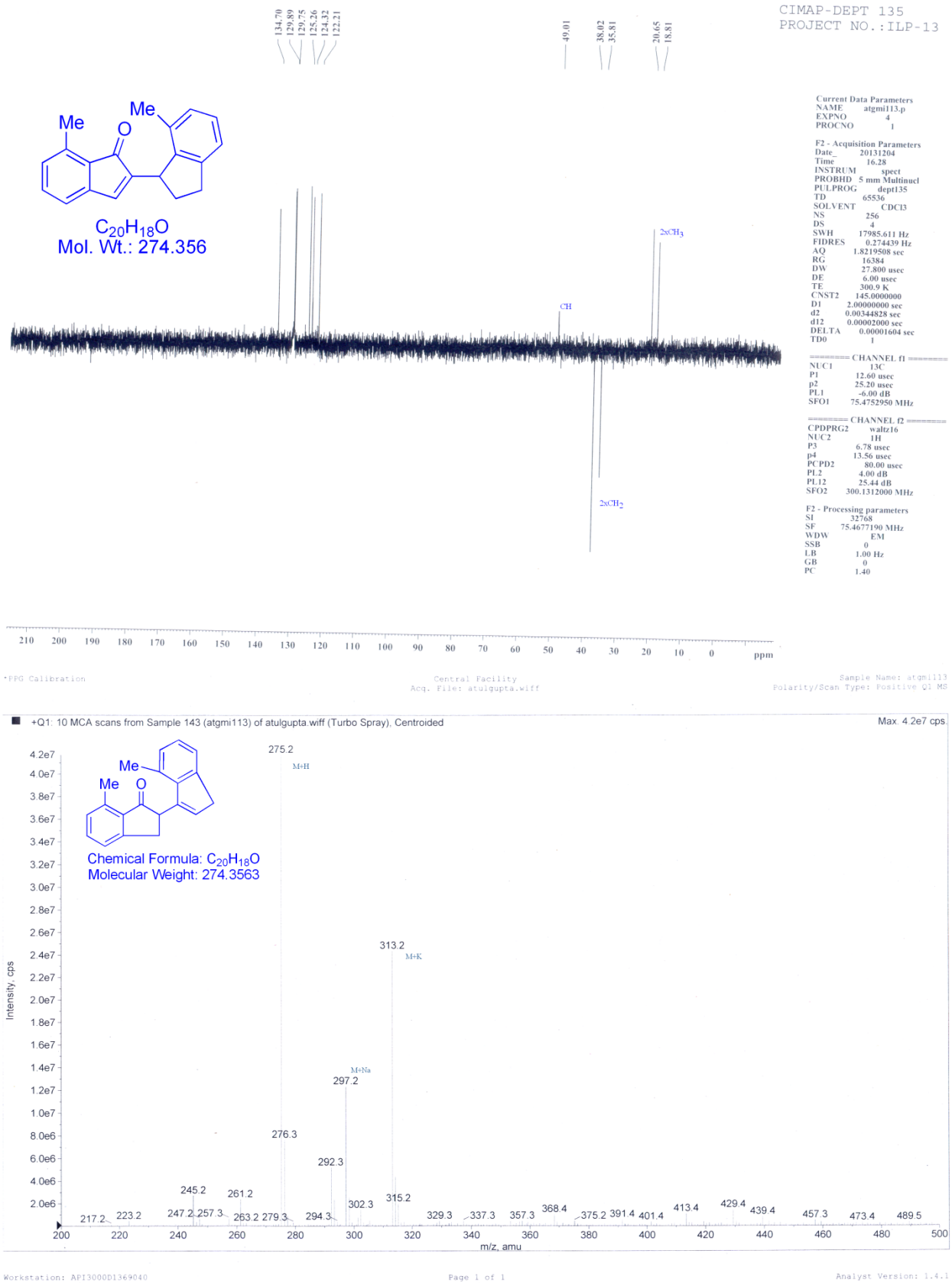
Current Data Parameters
NAME atgmi113.p
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131129
Time 17.21
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 502
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 301.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

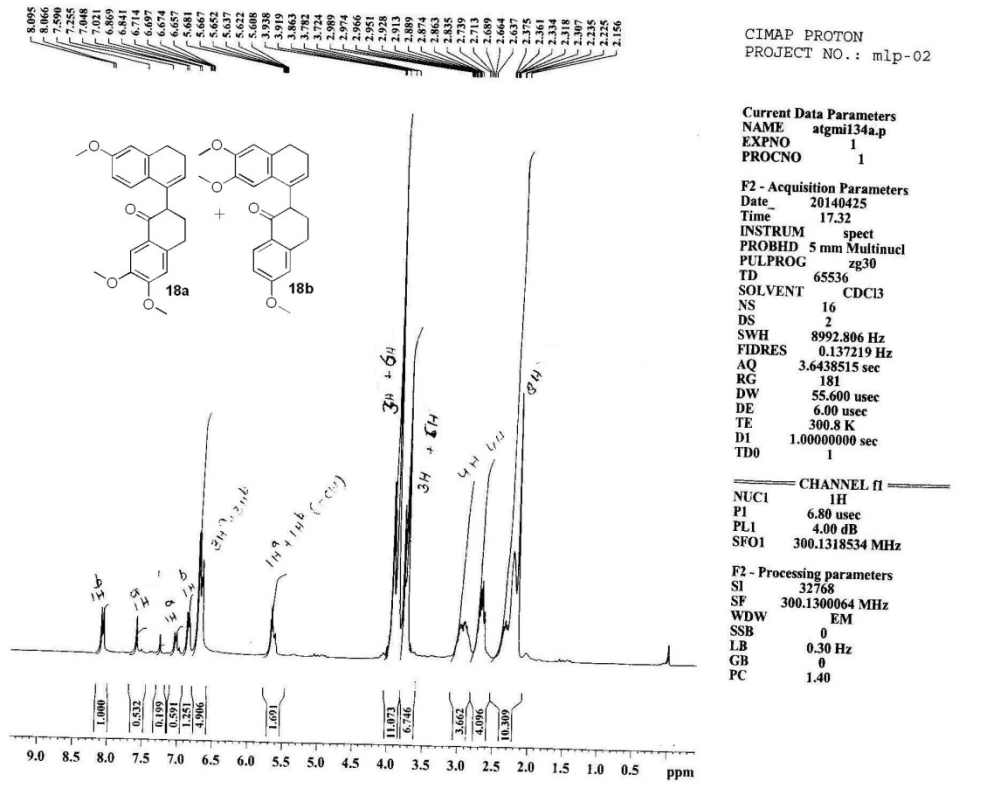
======== CHANNEL f1 ========
NUC1 13C
P1 12.60 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

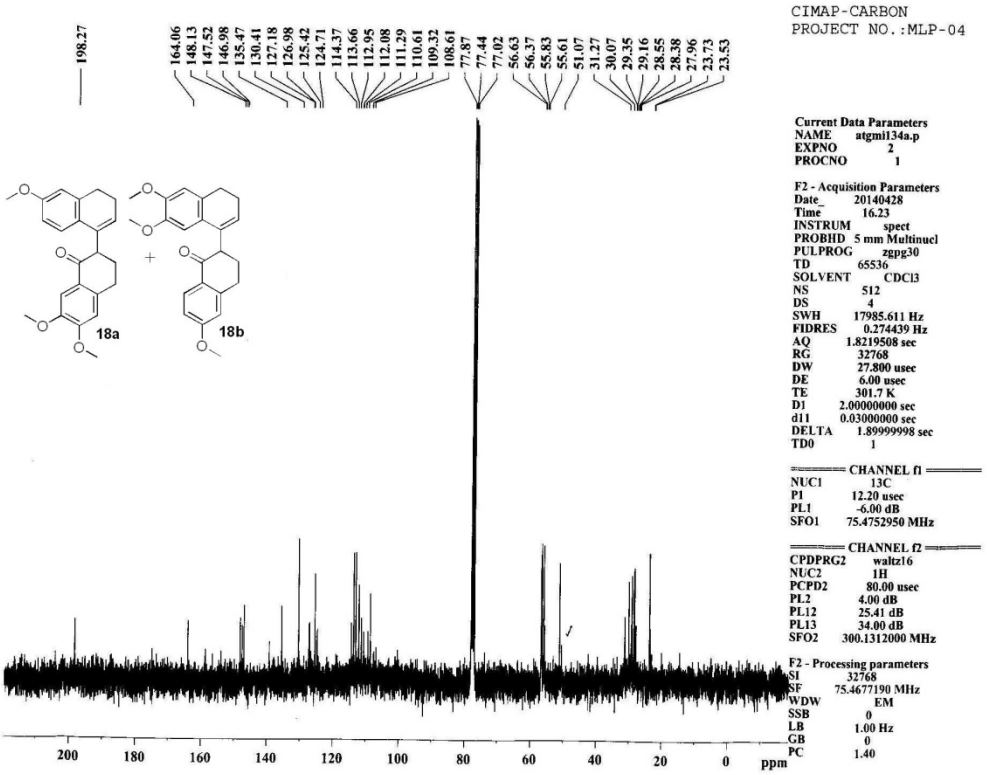
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.44 dB
PL13 29.61 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

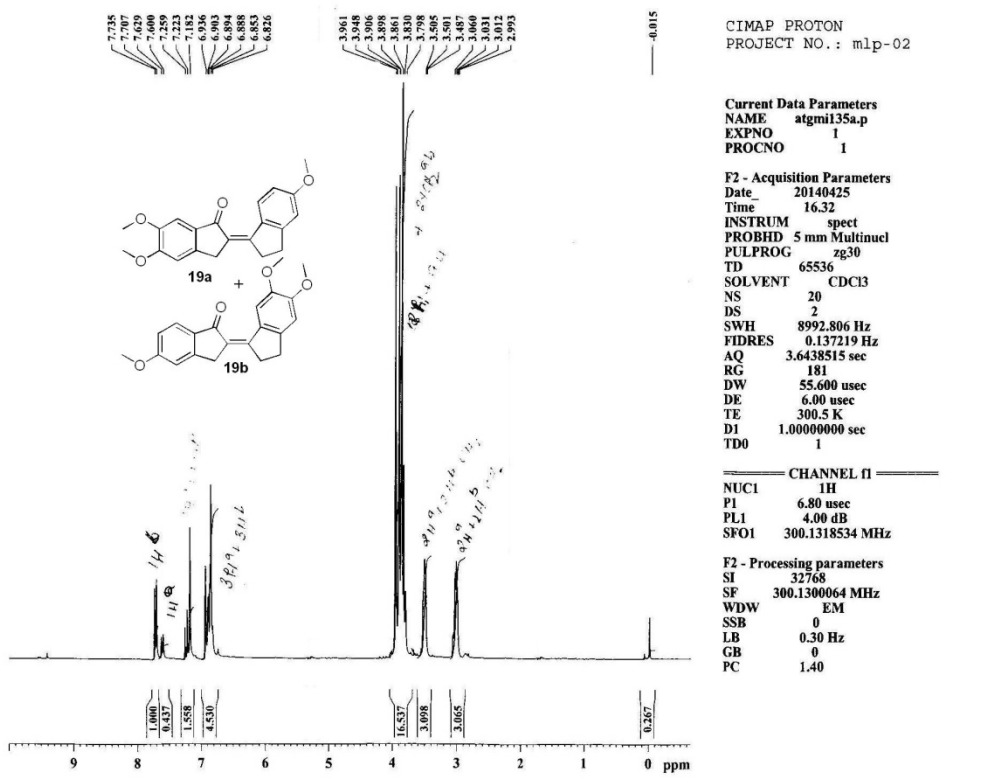


6,6',7'-trimethoxy-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'*H*)-one (18a) and 6,6',7-trimethoxy-3,3',4,4'-tetrahydro-1,2'-binaphthyl-1'(2'*H*)-one (18b)





5,6-dimethoxy-2-(5-methoxy-2,3-dihydro-1*H*-inden-1-ylidene)-2,3-dihydro-1*H*-inden-1-one (19a) and (2*Z*)-2-(5,6-dimethoxy-2,3-dihydro-1*H*-inden-1-ylidene)-5-methoxy -2,3-dihydro-1*H*-inden-1-one (19b):



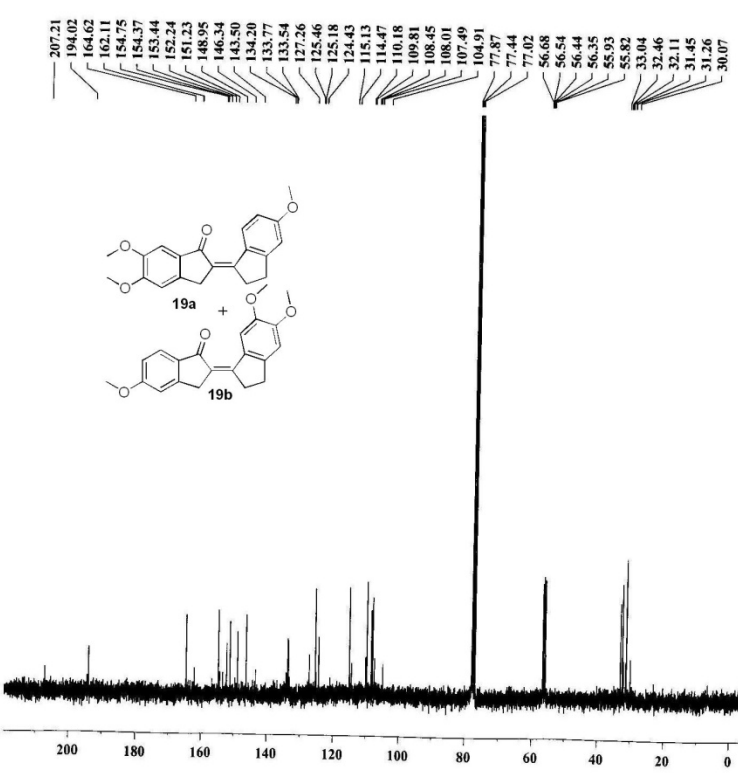
CIMAP PROTON
PROJECT NO.: mlp-02

Current Data Parameters
NAME atgmi135a.p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140425
Time 16.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 2
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 181
DW 55.600 usec
DE 6.00 usec
TE 300.5 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 6.80 usec
PL1 4.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



CIMAP-CARBON
PROJECT NO.:MLP-04

Current Data Parameters
NAME atgmi135a.p
EXPNO 2
PROCNO 1

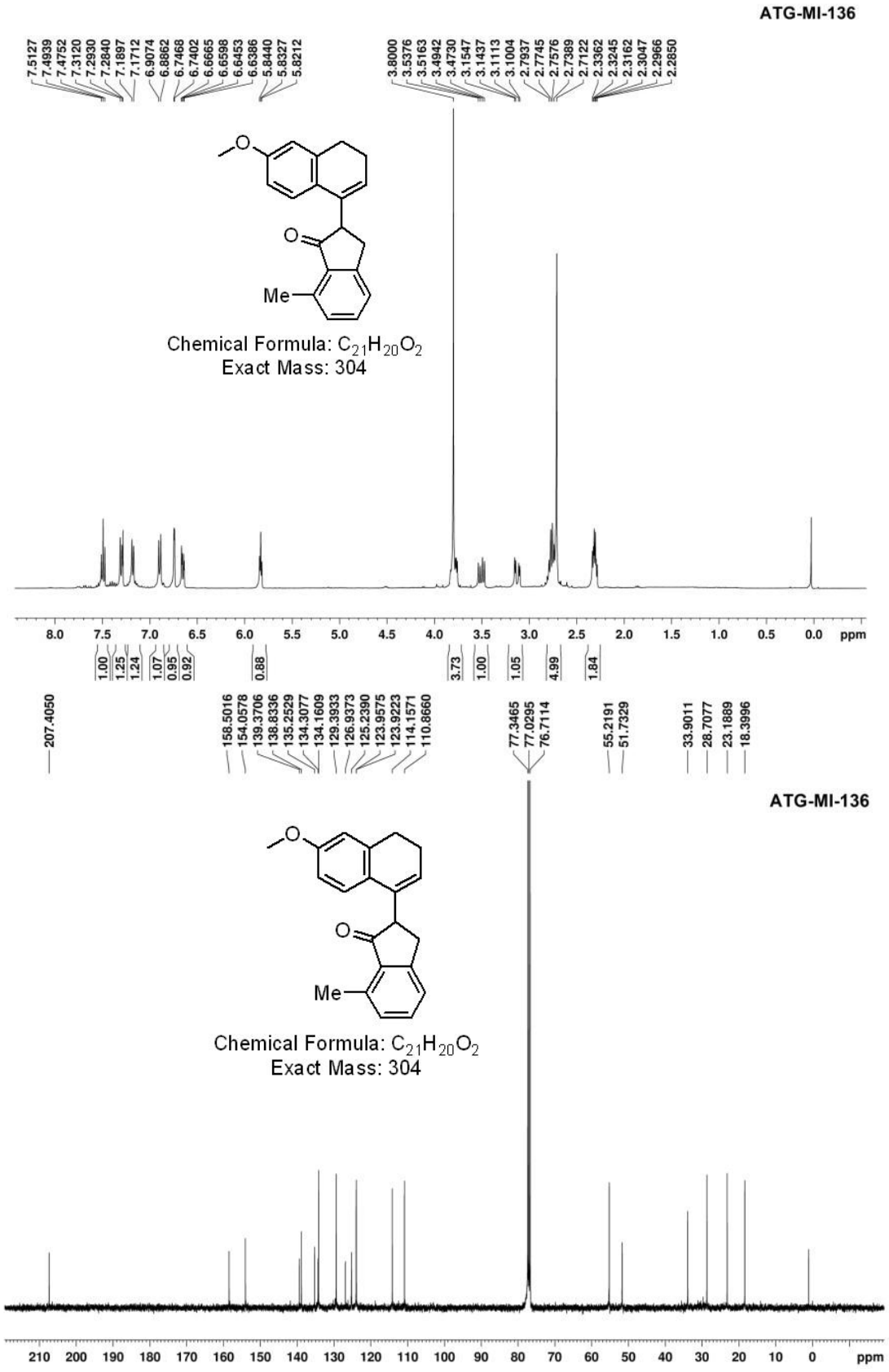
F2 - Acquisition Parameters
Date_ 20140425
Time 16.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 301.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 12.20 usec
PL1 -6.00 dB
SFO1 75.4752950 MHz

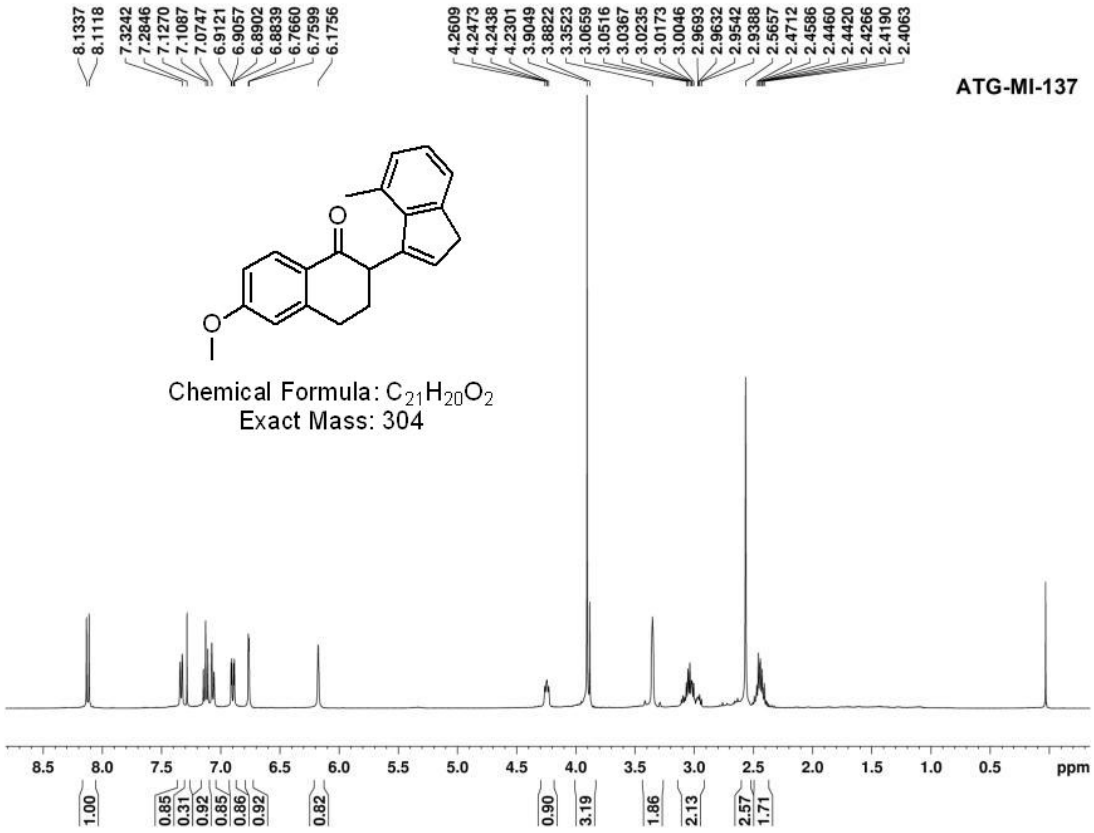
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.41 dB
PL13 34.00 dB
SFO2 300.1312000 MHz

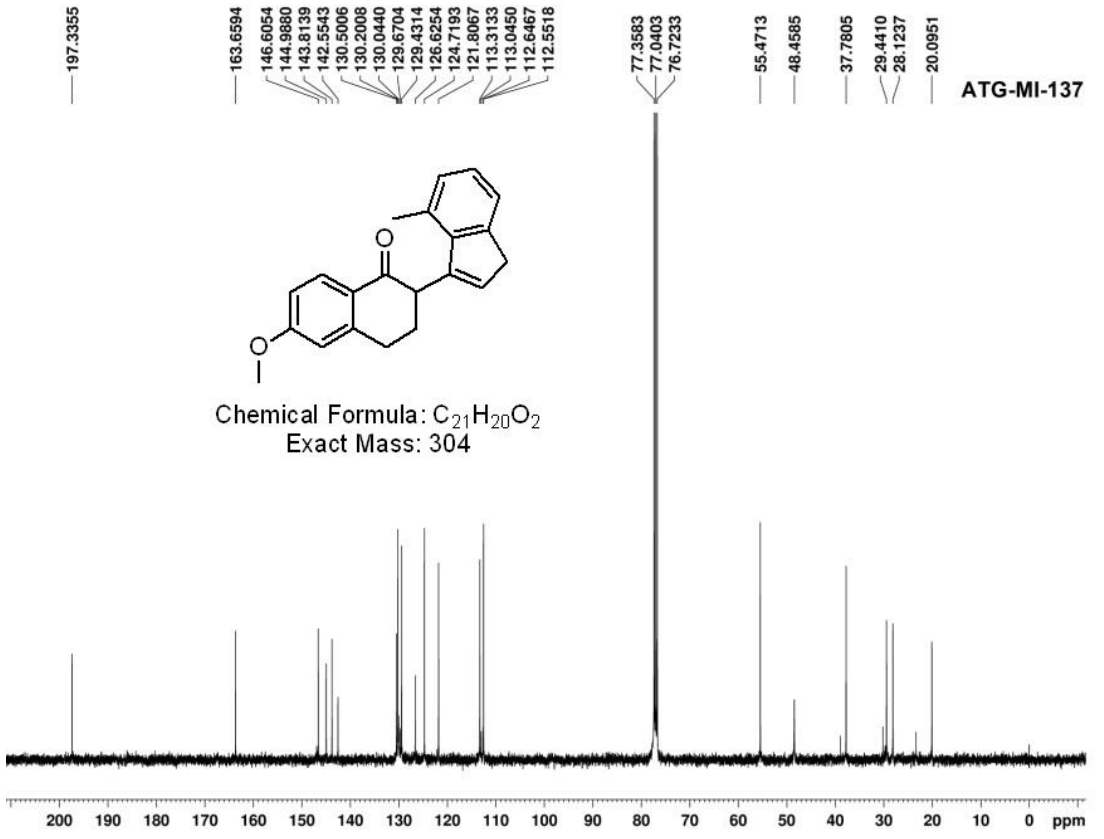
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

2-(6-methoxy-3,4-dihydronaphthalen-1-yl)-7-methyl-2,3-dihydro-1H-inden-1-one (20)



6-methoxy-2-(4-methyl-1H-inden-3-yl)-3,4-dihydronaphthalen-1(2H)-one (21)


8.1337
8.1118
7.3242
7.2846
7.1270
7.1087
7.0747
6.9121
6.9057
6.8902
6.8839
6.7660
6.7599
6.1756
4.2609
4.2473
4.2438
4.2301
3.9049
3.8822
3.3523
3.0659
3.0516
3.0367
3.0235
3.0173
3.0046
2.9693
2.9632
2.9542
2.9388
2.5657
2.4712
2.4586
2.4460
2.4420
2.4266
2.4190
2.4063
ATG-MI-137
O
O
Chemical Formula: C21H20O2
Exact Mass: 304
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.85
0.31
0.92
0.85
0.86
0.92
0.82
0.90
3.19
1.86
2.13
2.57
1.71


197.3355
163.6594
146.6054
144.9880
143.8139
142.5543
130.5006
130.2008
130.0440
129.6704
129.4314
126.6254
124.7193
121.8067
113.3133
113.0450
112.6467
112.5518
77.3583
77.0403
76.7233
55.4713
48.4585
37.7805
29.4410
28.1237
20.0951
ATG-MI-137
O
O
Chemical Formula: C21H20O2
Exact Mass: 304
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm