Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Palladium Mediated Intramolecular Multiple C-X/C-H Cross Coupling & C-H Activation: Synthesis of Carbazole Alkaloids Calothrixin B & Murrayaquinone A

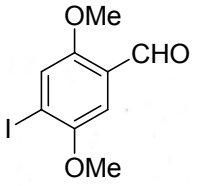
Srinivasan. A Kaliyaperumal, Shyamapada Banerjee, Syam Kumar. U. K.*

Technology Development Centre, Custom Pharmaceutical Services, Dr. Reddy's Laboratories Ltd, Miyapur, Hyderabad, Pin 500 049, India.

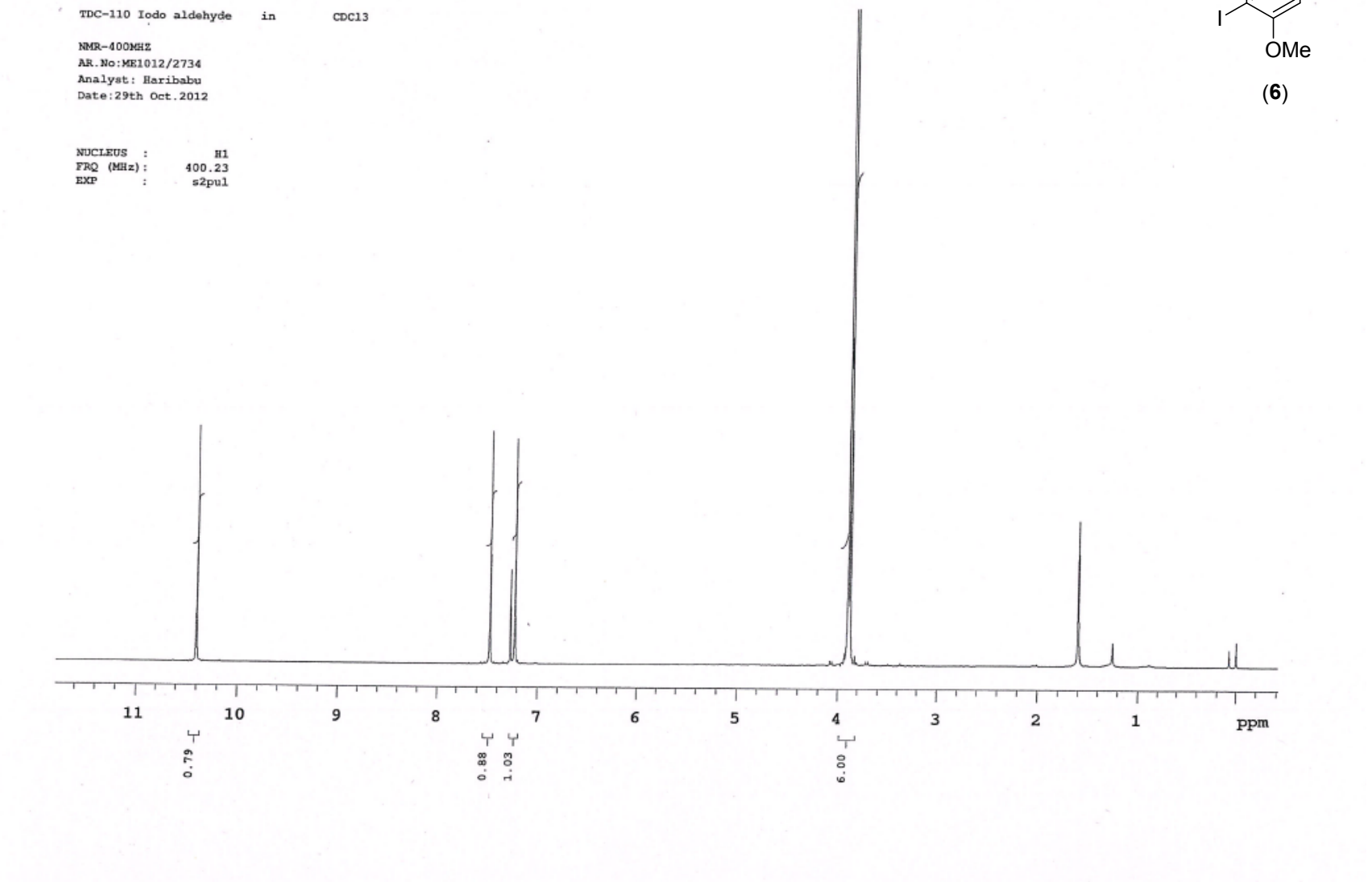
syam_kmr@yahoo.com.

Supporting Information

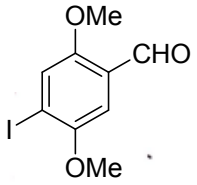
^{1}H NMR of 4-iodo-2,5-dimethoxybenzaldehyde (6)



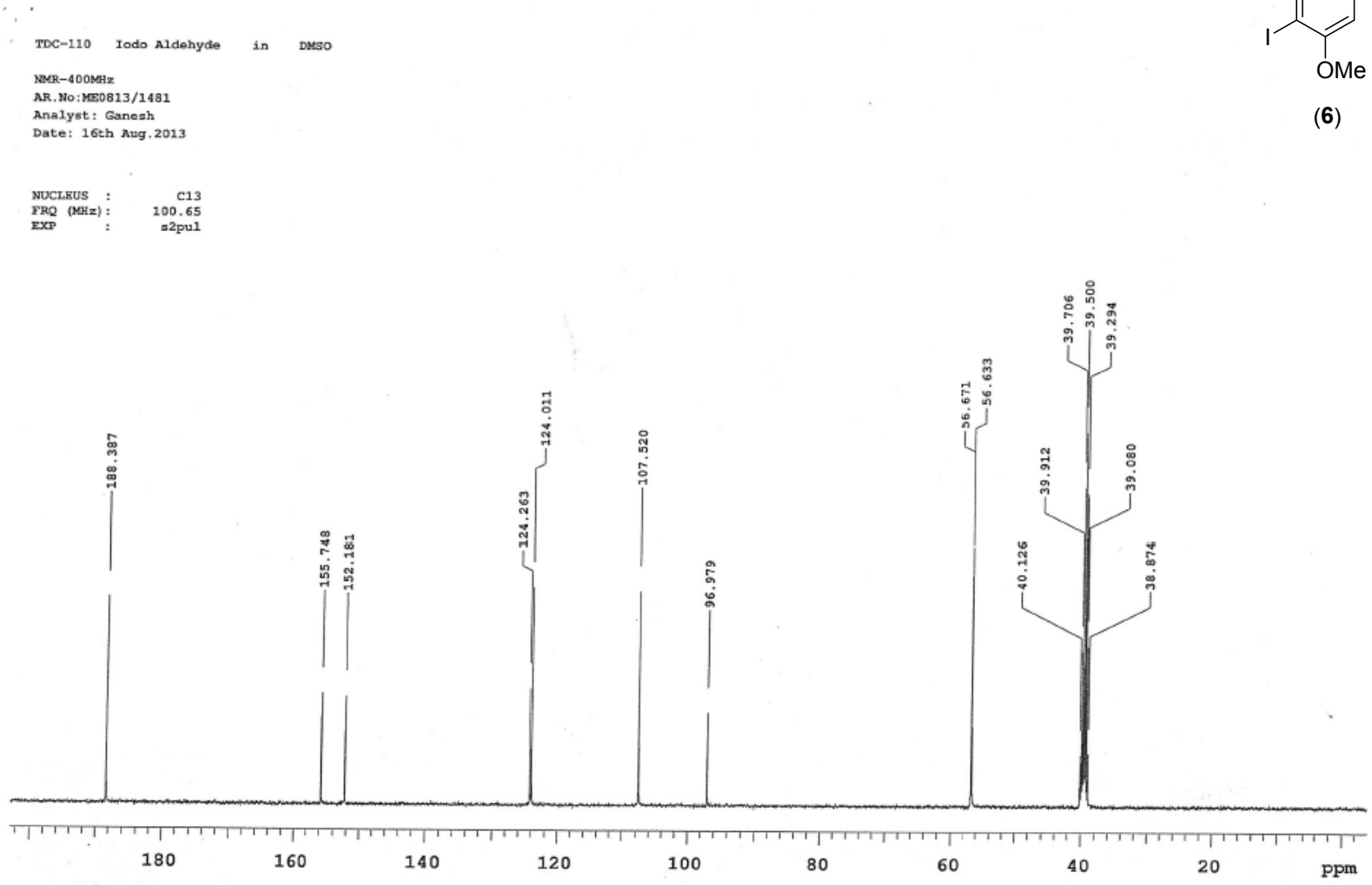
(**6**)



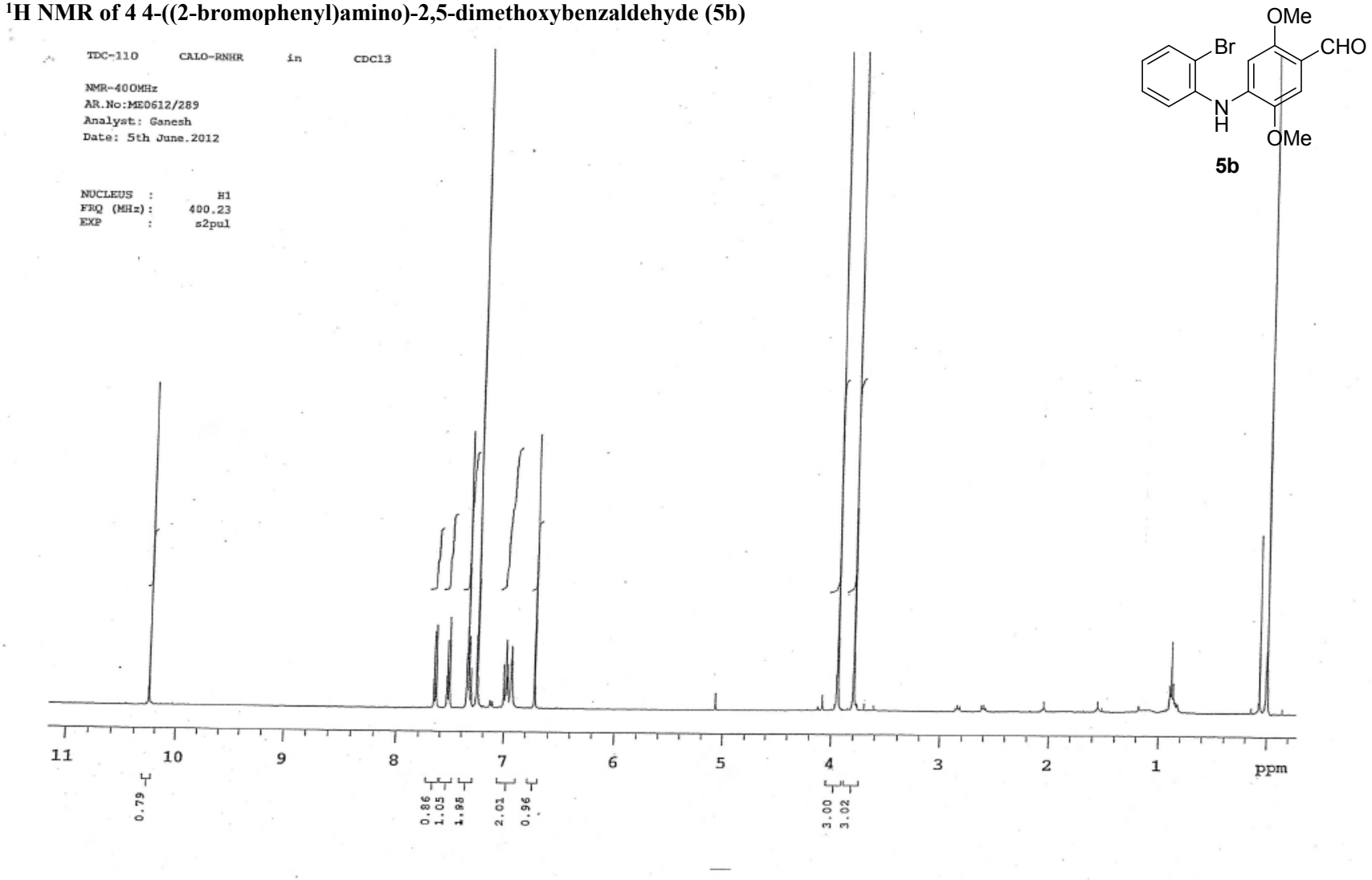
^{13}C NMR of 4-iodo-2,5-dimethoxybenzaldehyde (6)



(**6**)

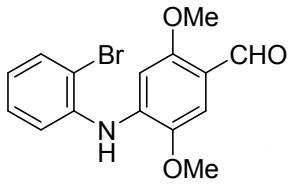


[1]H NMR of 4 4-((2-bromophenyl)amino)-2,5-dimethoxybenzaldehyde (5b)

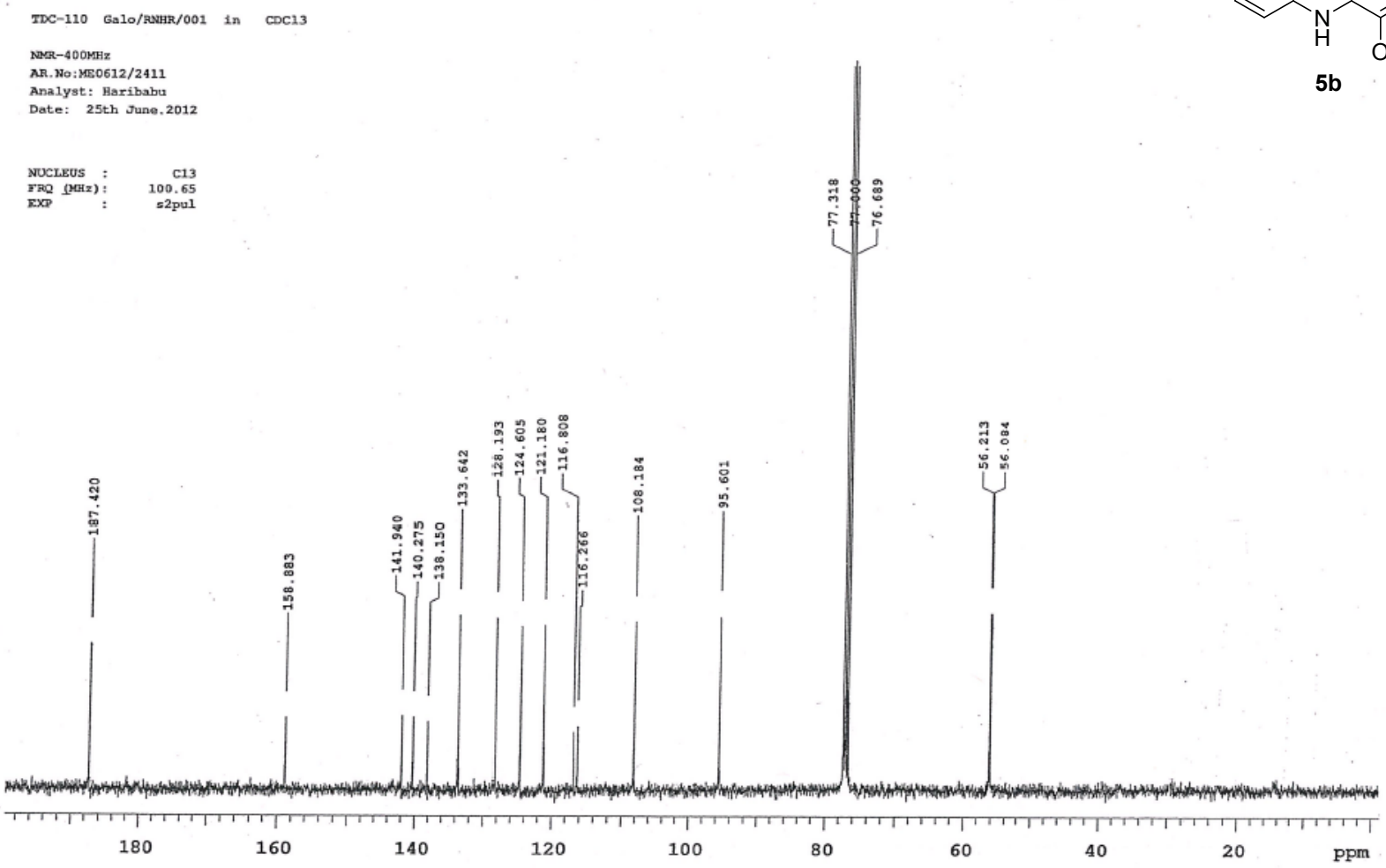


5b

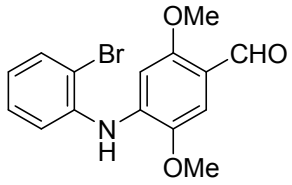
^{13}C NMR of 4-((2-bromophenyl)amino)-2,5-dimethoxybenzaldehyde (5b)



5b



Mass Spectrum of 4-((2-bromophenyl)amino)-2,5-dimethoxybenzaldehyde (5b)



5b

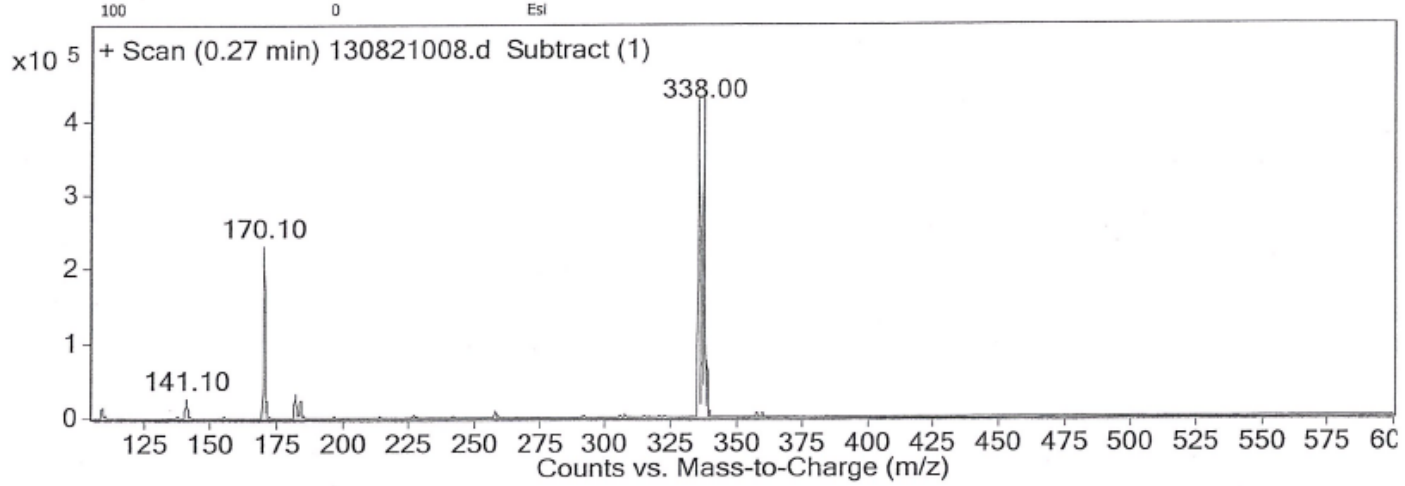
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130821008.d	**Sample Name**	RNHR
Sample Type	Sample	**Position**	Vial 28
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

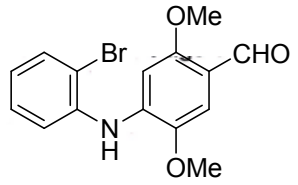
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of 4-((2-bromophenyl)amino)-2,5-dimethoxybenzaldehyde (5b)



5b

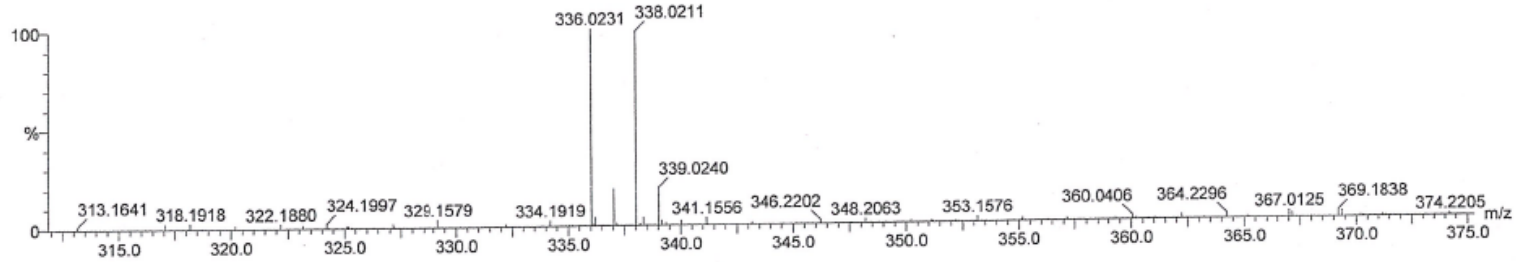
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
36 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-20 H: 0-20 N: 0-2 O: 0-4 Br: 0-1

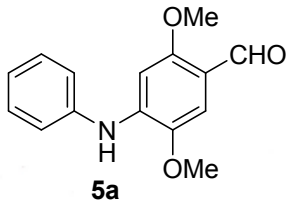
R-NHR
130926012 13 (0.478) Cm (13:15-1:2)

1: TOF MS ES+
1.04e+004

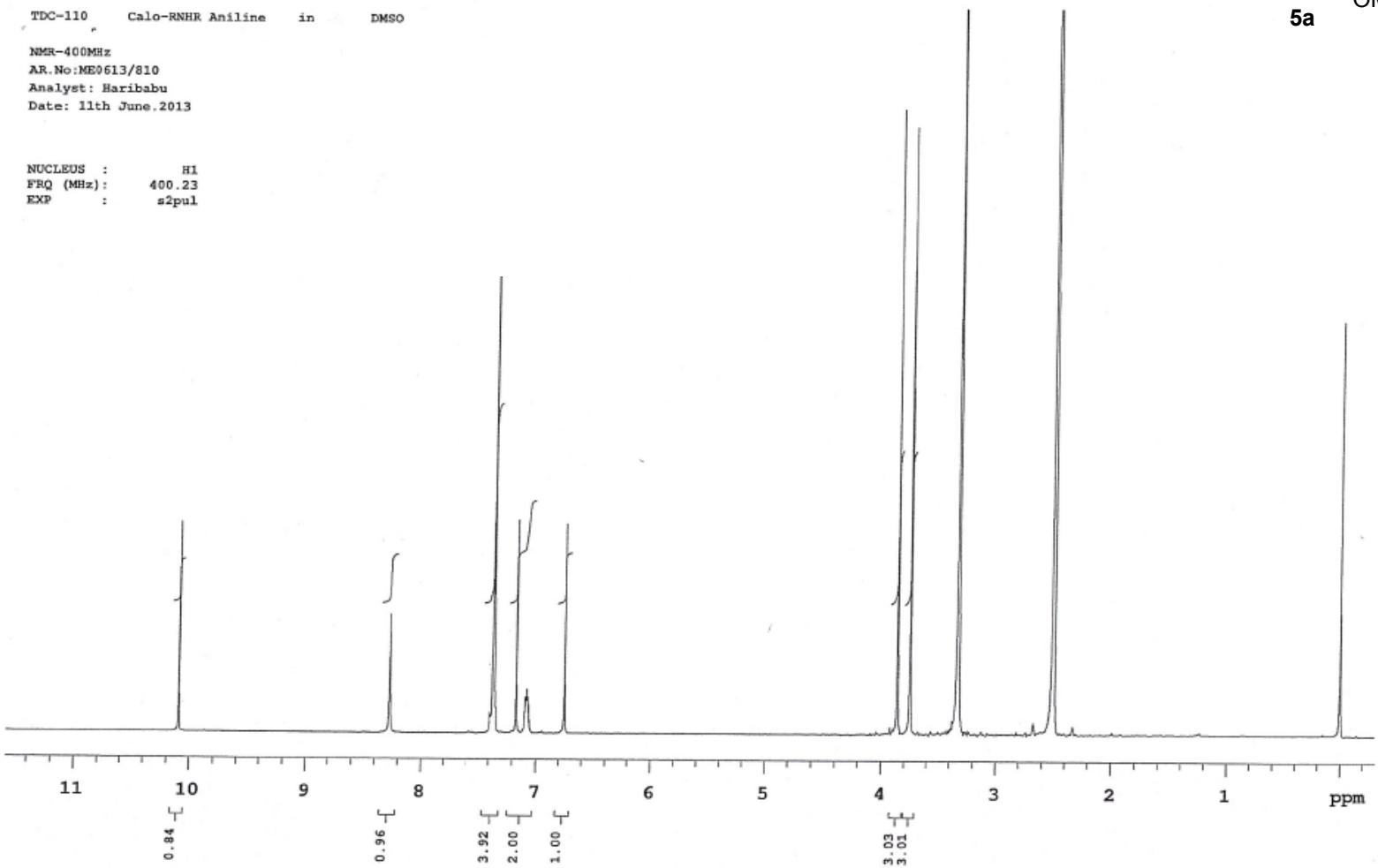


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
336.0231	336.0235	-0.4	-1.2	8.5	12.2	C15 H15 N O3 Br

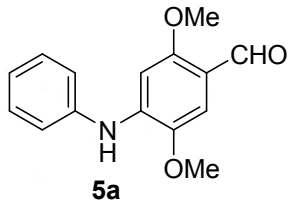
^{1}H NMR of 2,5-dimethoxy-4-(phenylamino)benzaldehyde (5a)



5a

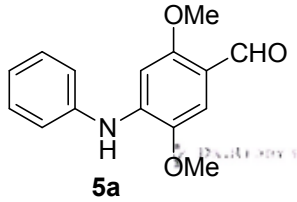


^{13}C NMR of 2,5-dimethoxy-4-(phenylamino)benzaldehyde (5a)



5a

Mass Spectrum of 2,5-dimethoxy-4-(phenylamino)benzaldehyde (5a)



5a

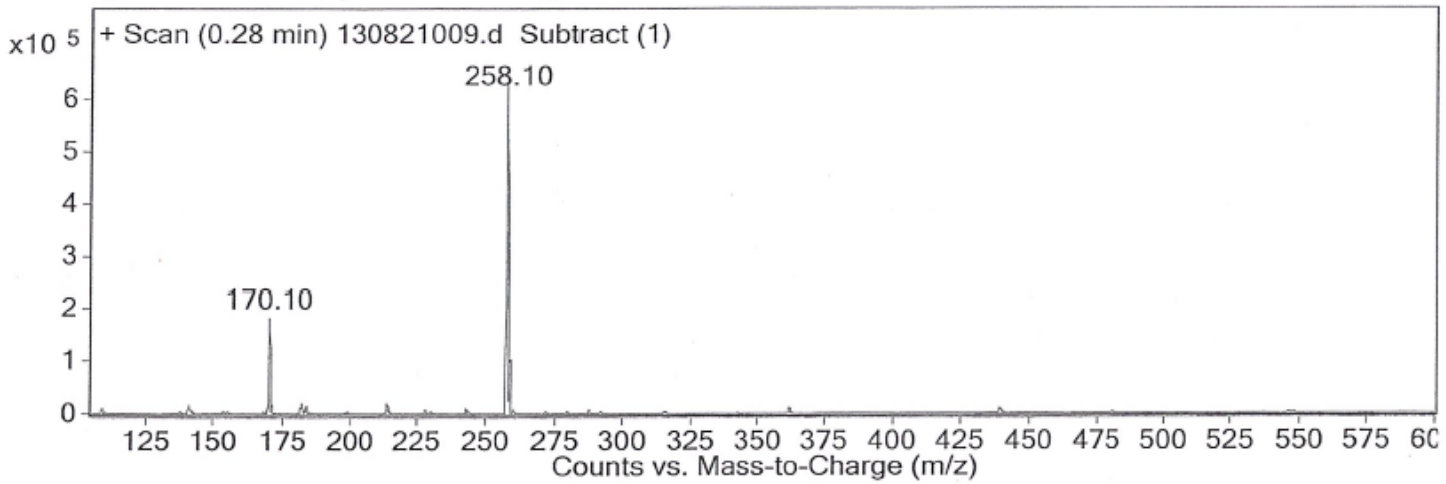
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130821009.d	**Sample Name**	RNHR Aniline
Sample Type	Sample	**Position**	Vial 29
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

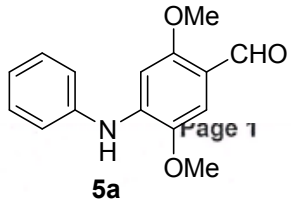
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of 2,5-dimethoxy-4-(phenylamino)benzaldehyde (5a)



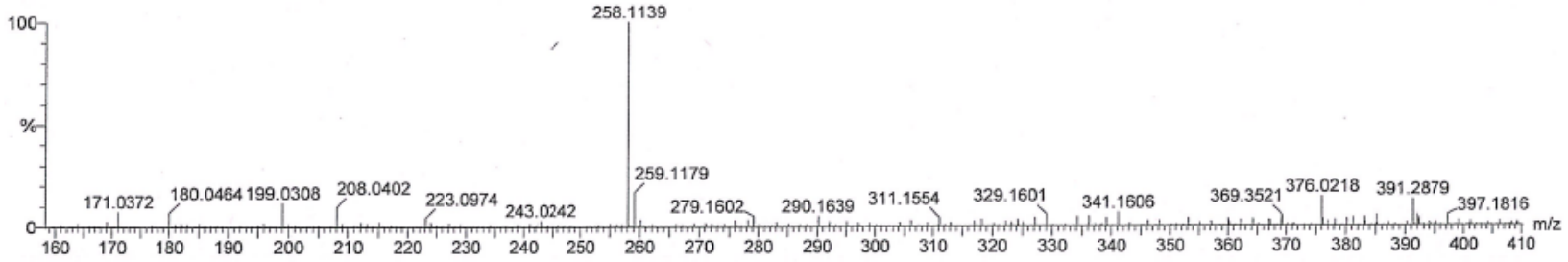
5a

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

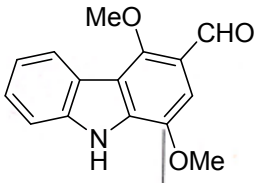
Monoisotopic Mass, Even Electron Ions
53 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-30 H: 0-40 N: 0-2 O: 0-4
R-NHR Aniline
130926010-1 13 (0.479) Cm (13:15-1:2)

1: TOF MS ES+
5.03e+003

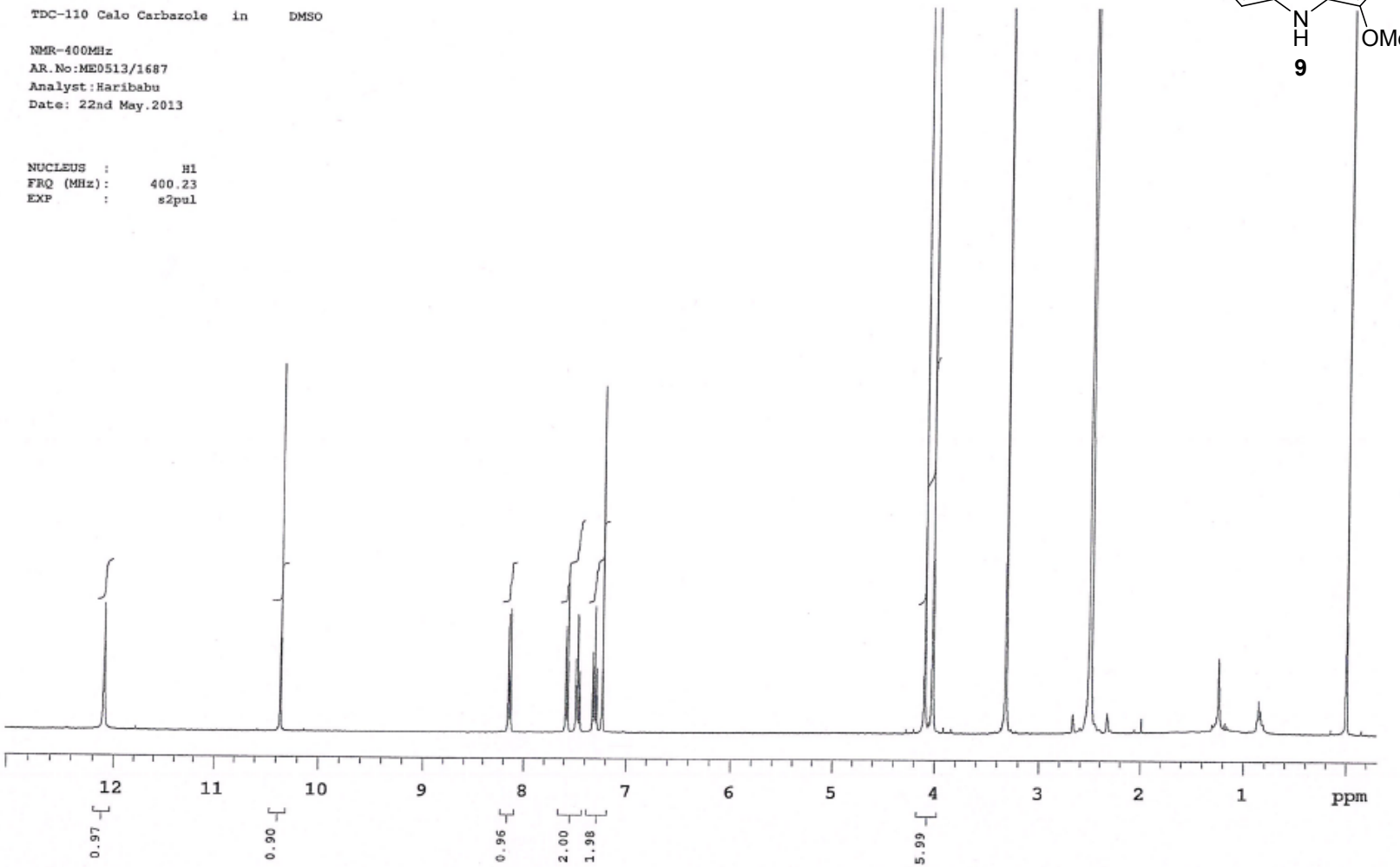


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
258.1139	258.1130	0.9	3.5	8.5	n/a	C15 H16 N O3

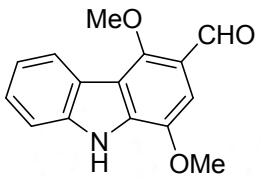
^{1}H NMR of 1,4-dimethoxy-9*H*-carbazole-3-carbaldehyde (9)



9

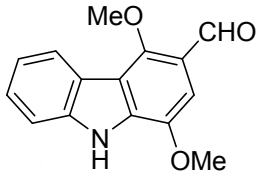


Expansion of ^{1}H NMR of 1,4-dimethoxy-9*H*-carbazole-3-carbaldehyde (9)

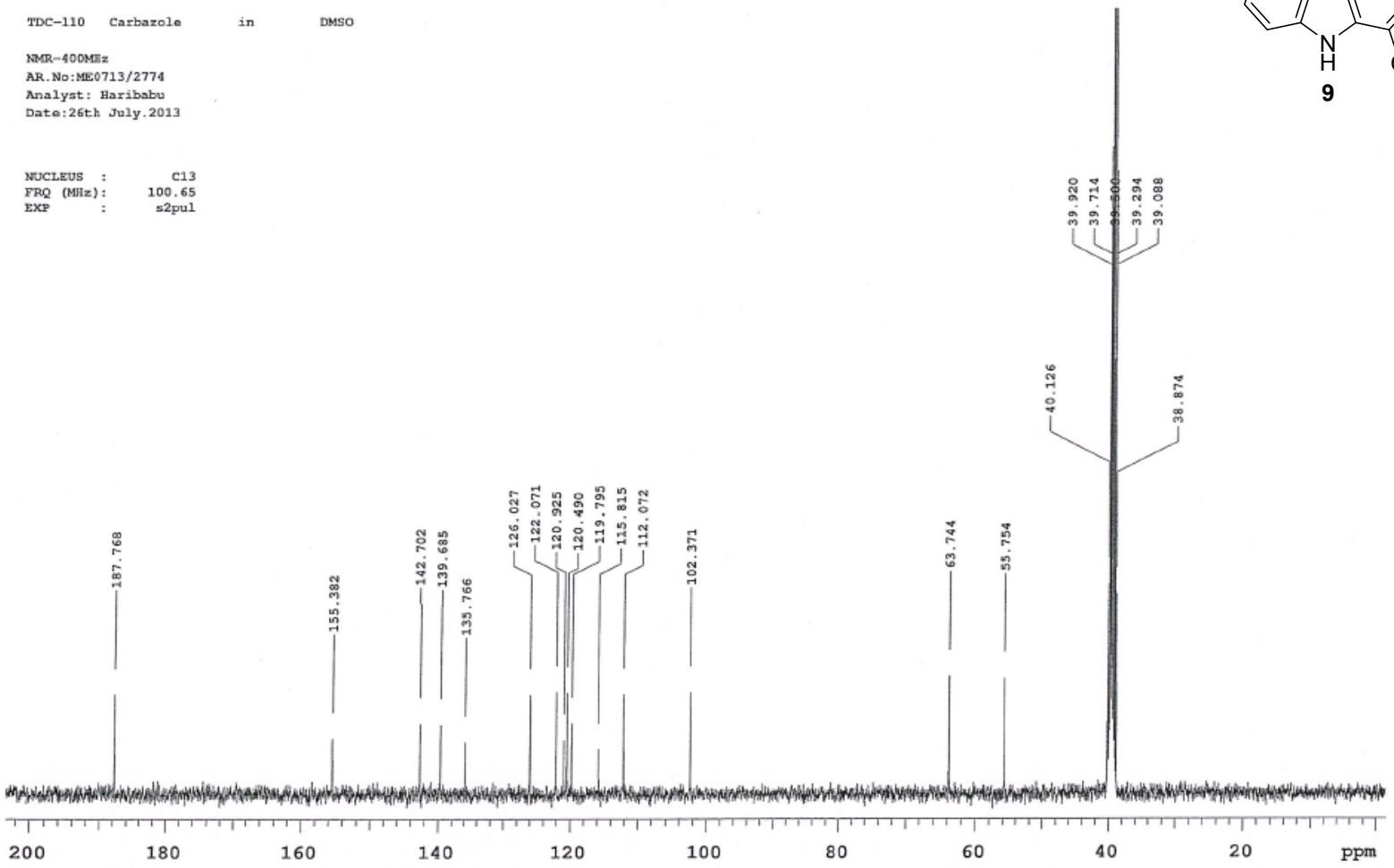


9

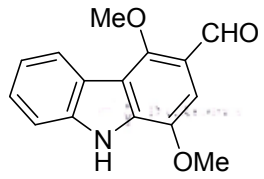
^{13}C NMR of 1,4-dimethoxy-9*H*-carbazole-3-carbaldehyde (9)



9



Mass Spectrum of 1,4-dimethoxy-9*H*-carbazole-3-carbaldehyde (9)



9

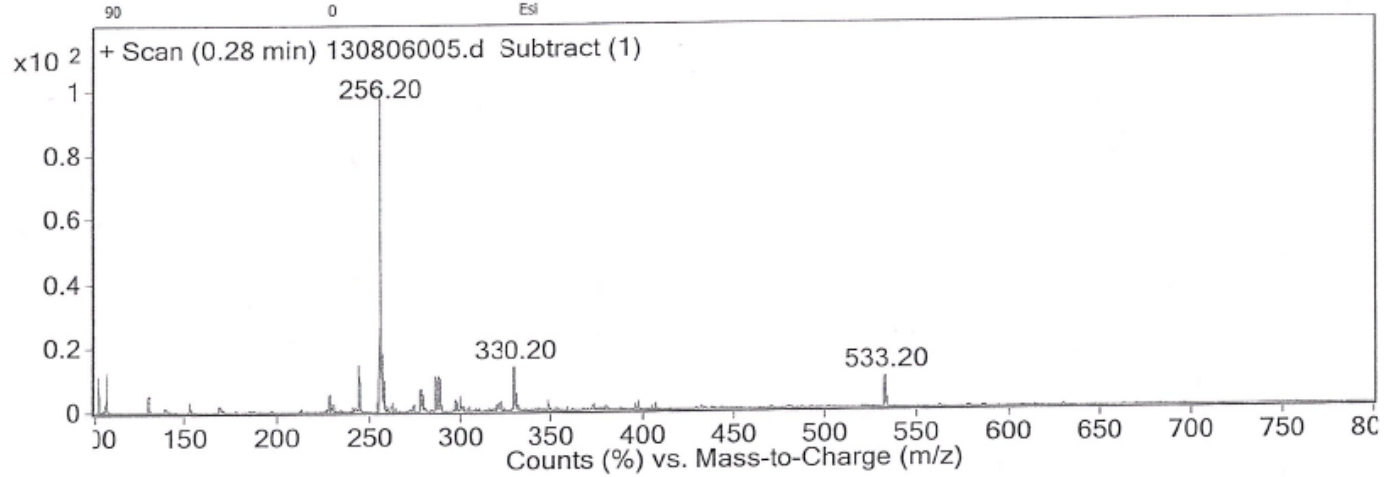
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130806005.d	**Sample Name**	Carbazole
Sample Type	Sample	**Position**	Vial 96
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

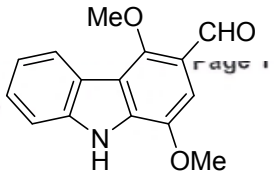
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
90	0	Esi



--- End Of Report ---

HRMS of 1,4-dimethoxy-9*H*-carbazole-3-carbaldehyde (9)



9

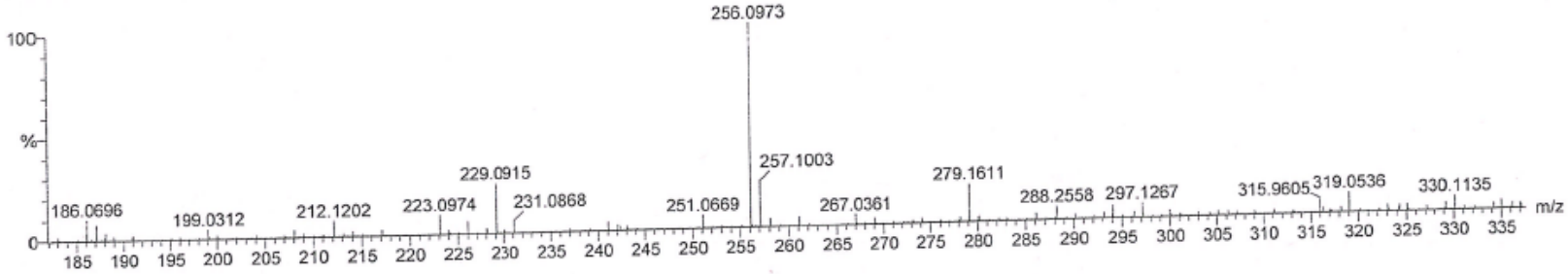
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
23 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-25 H: 0-20 N: 0-2 O: 0-3

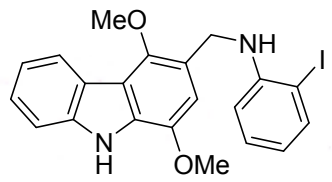
Carbazole
130926016 13 (0.478) Cm (13:17)

1: TOF MS ES+
1.13e+004

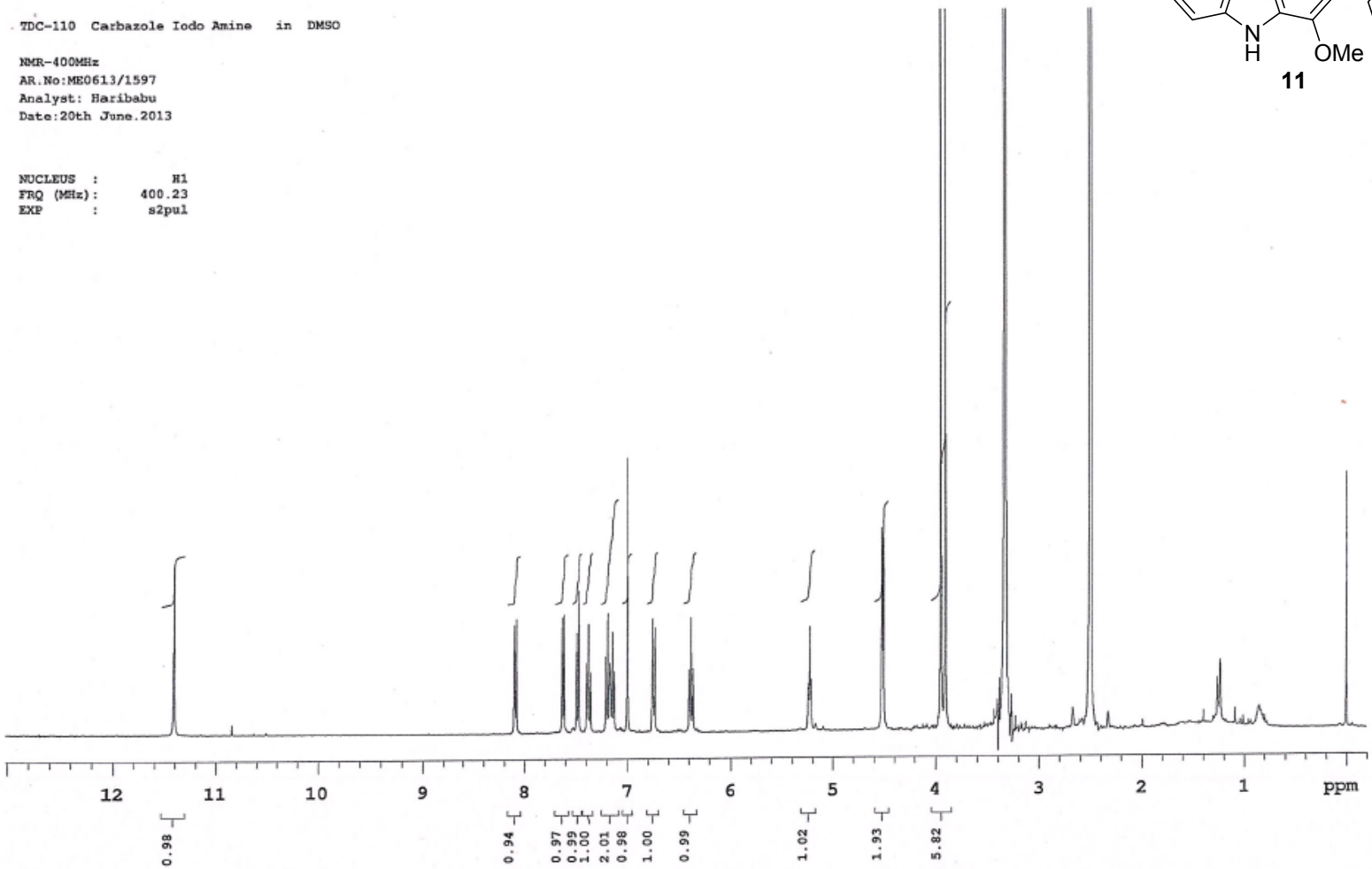


Minimum:		5.0	5.0	-5.0		
Maximum:				80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
256.0973	256.0974	-0.1	-0.4	9.5	145.4	C15 H14 N O3

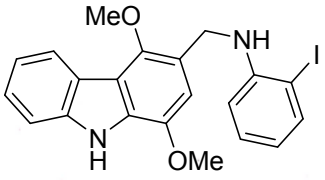
^{1}H NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-2-iodoaniline (11).



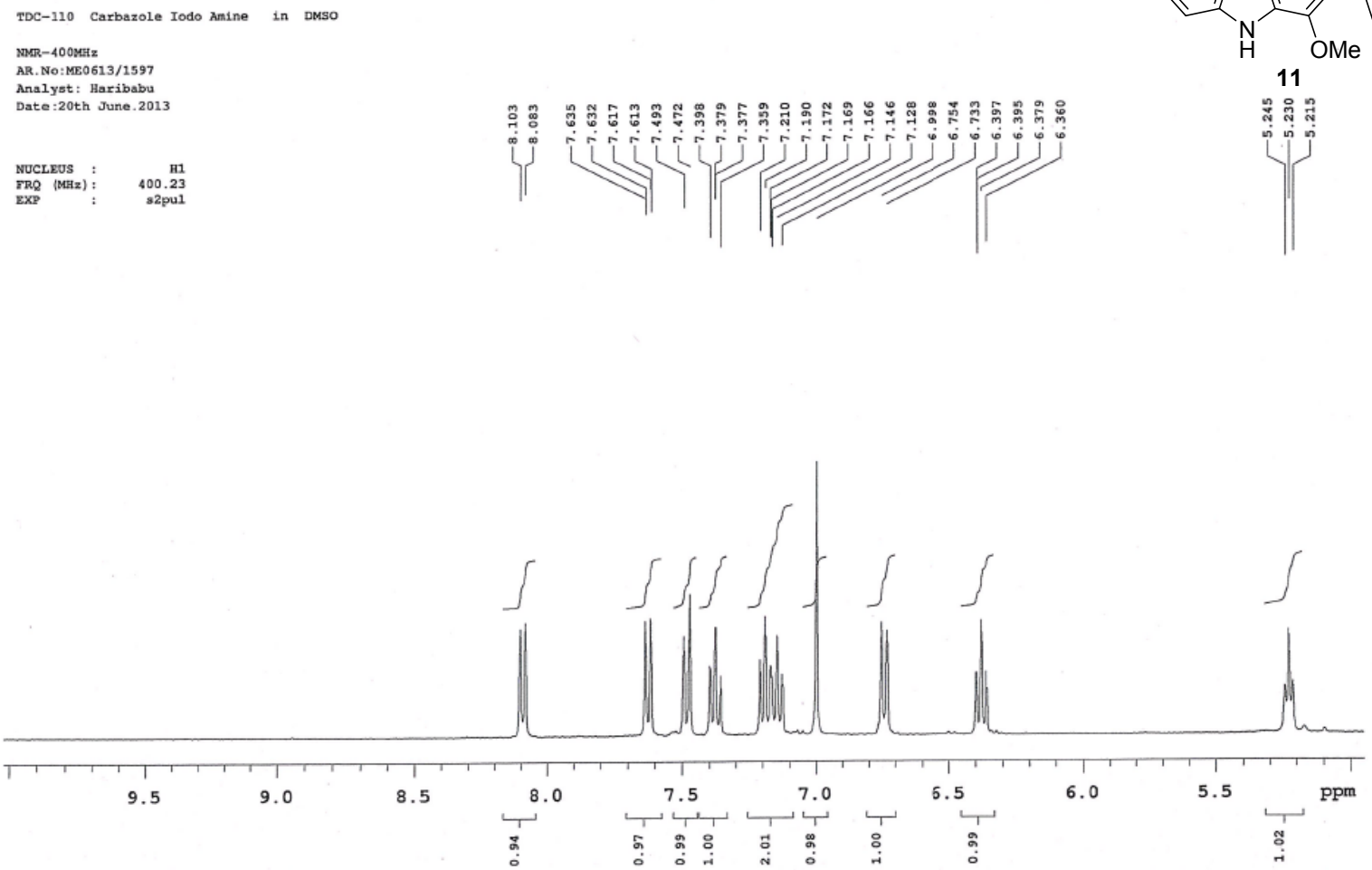
11



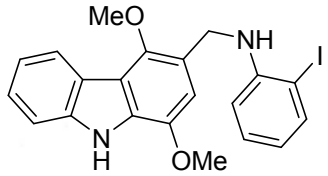
Expansion of ^{1}H NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-2-iodoaniline (11).



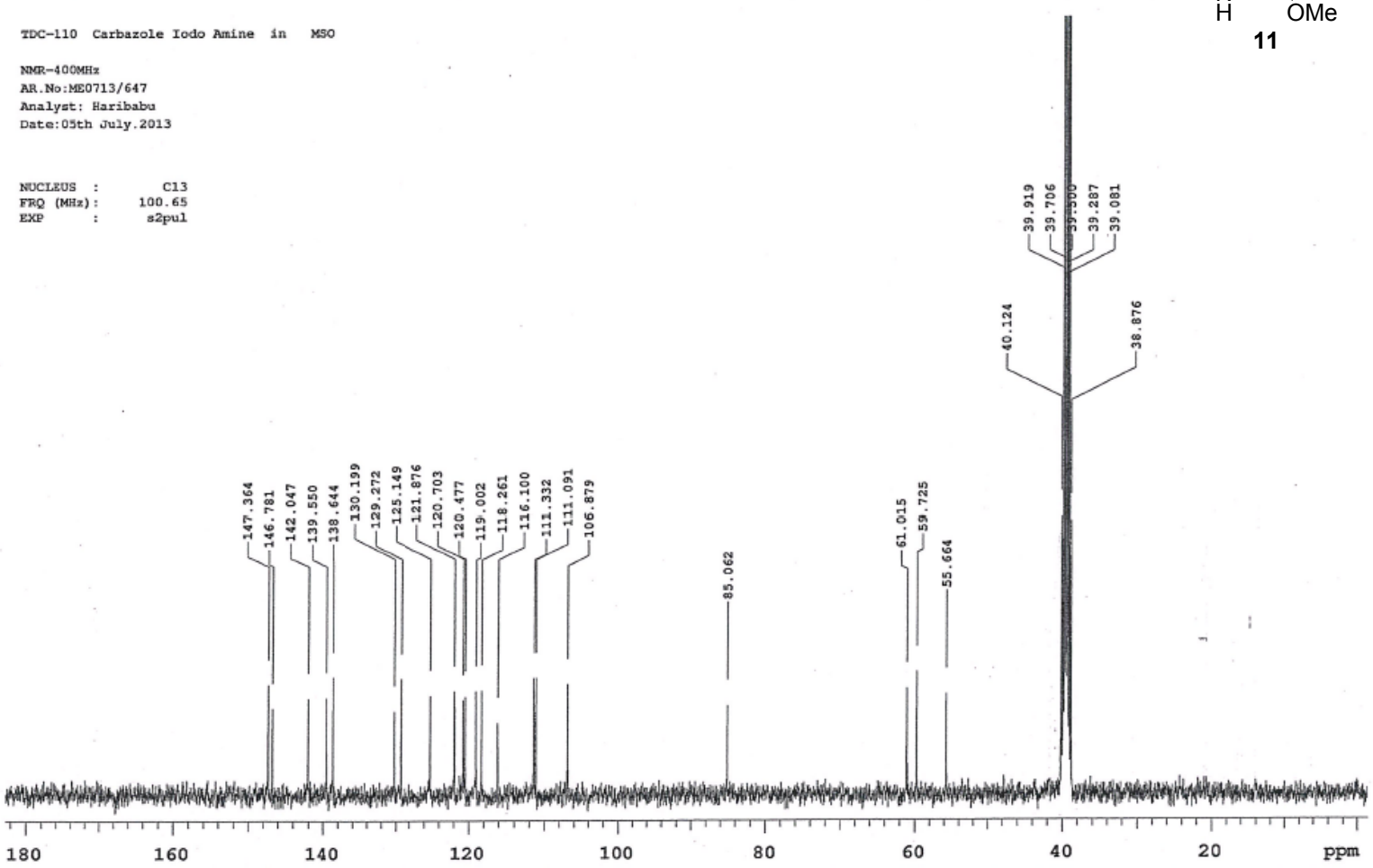
11



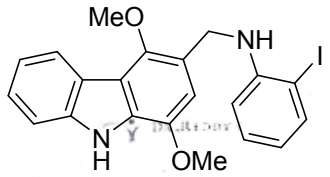
^{13}C NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-2-iodoaniline (11).



11



Mass Spectrum of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-2-iodoaniline (11).



11

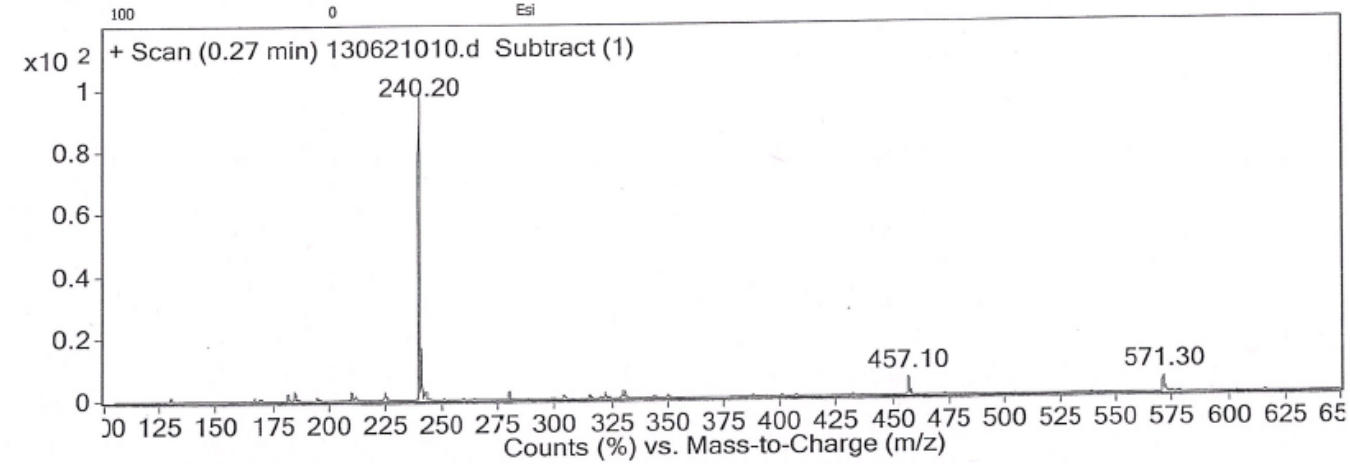
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130621010.d	**Sample Name**	Carbazole iodo Amine
Sample Type	Sample	**Position**	Vial 89
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

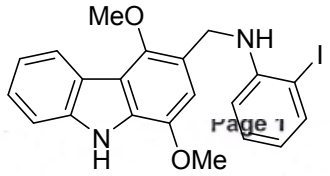
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-2-iodoaniline (11).



11

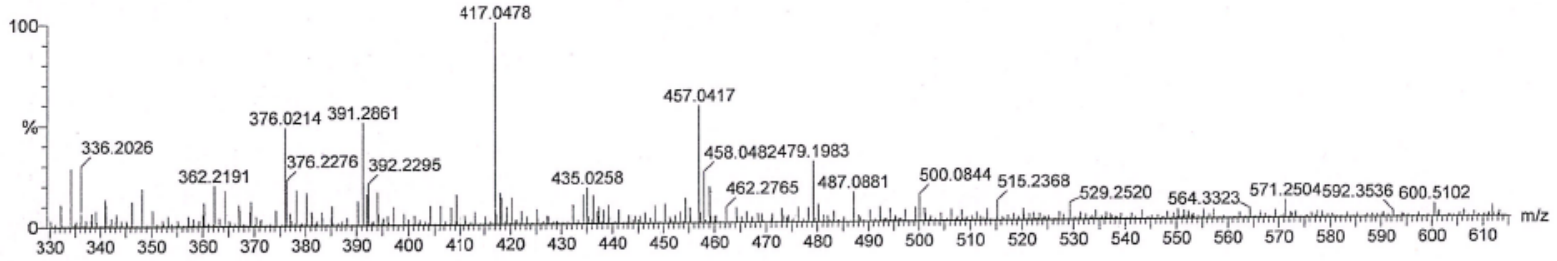
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-25 H: 0-20 N: 0-2 O: 0-2 I: 0-1

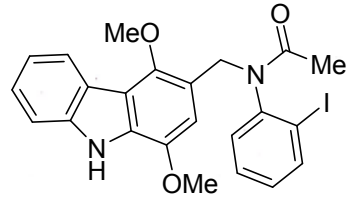
Carbazole iodoamine
130926011 9 (0.338) Cm (4:14)

1: TOF MS ES+
1.60e+004

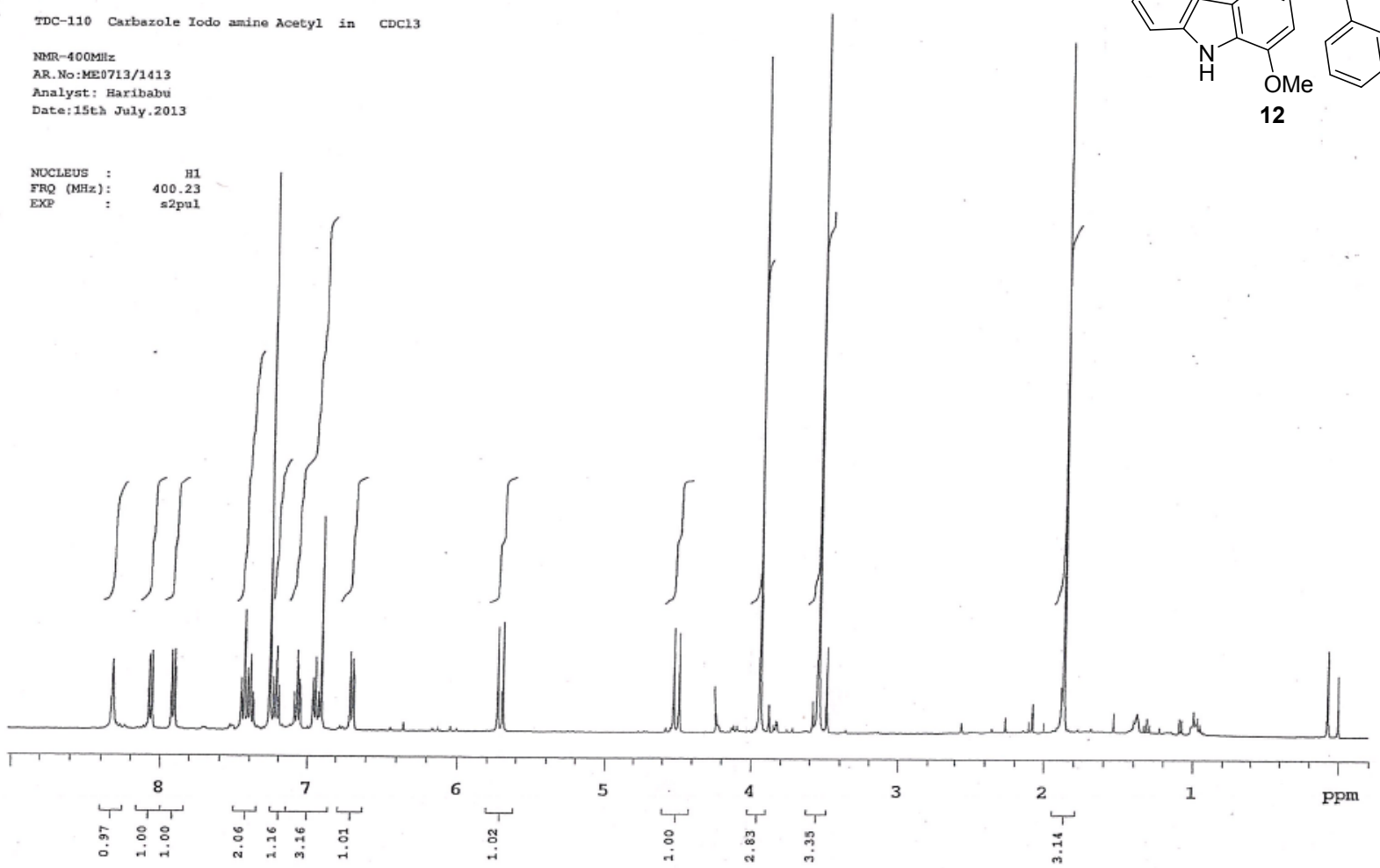


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
457.0417	457.0413	0.4	0.9	13.5	1686.9	C21 H18 N2 O2 I

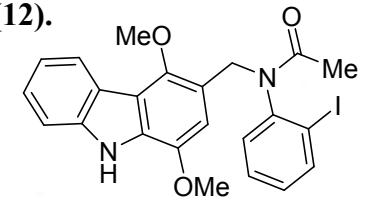
^{1}H NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-*N*-(2-iodophenyl)acetamide (12).



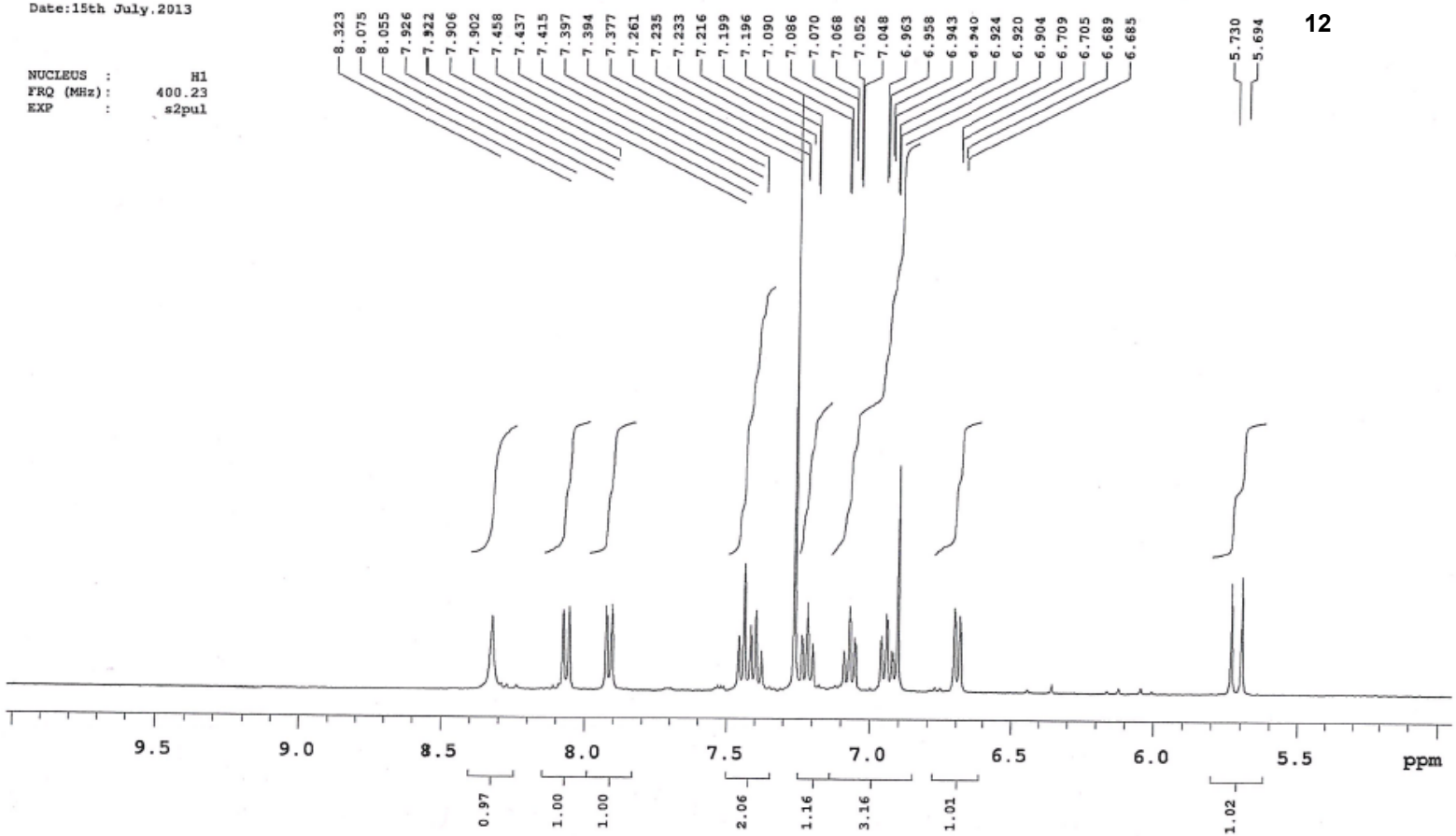
12



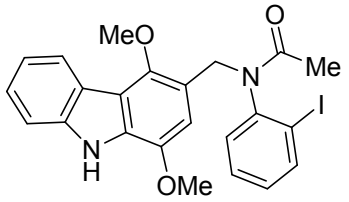
Expansion of ^{1}H NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-*N*-(2-iodophenyl)acetamide (12).



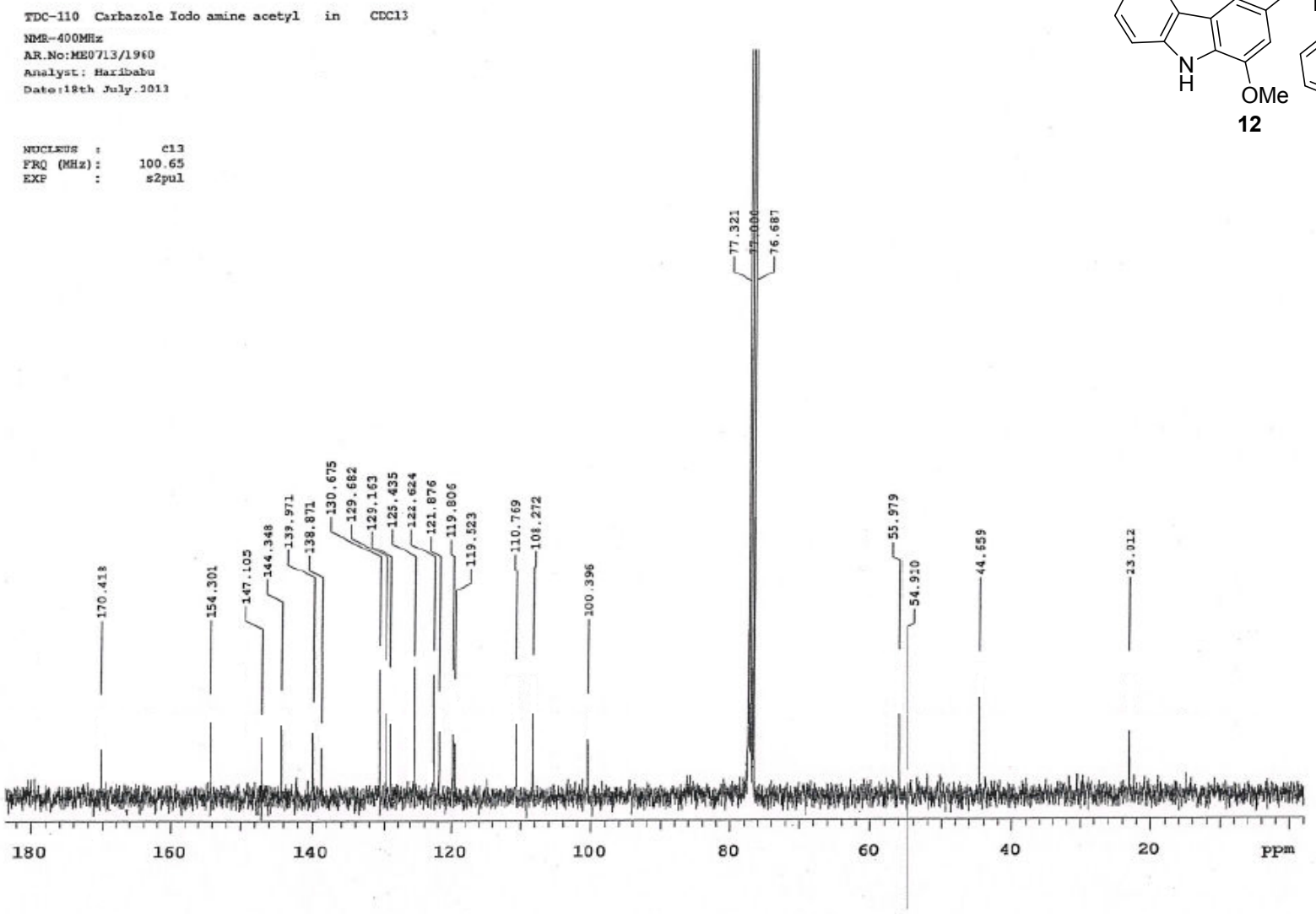
12



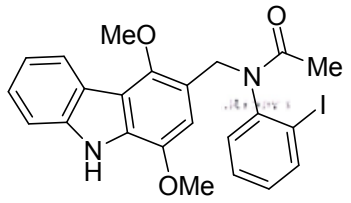
^{13}C NMR of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-*N*-(2-iodophenyl)acetamide (12).



12



Mass Spectrum of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-*N*-(2-iodophenyl)acetamide (12).



12

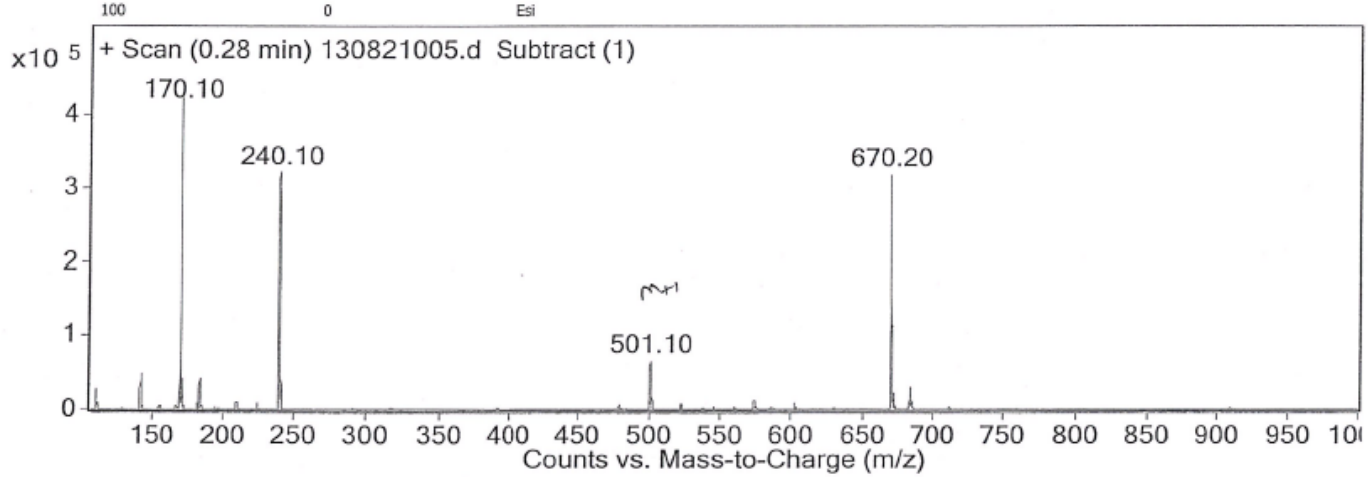
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130821005.d	**Sample Name**	Carbazole Iodo amine Acetyl
Sample Type	Sample	**Position**	Vial 25
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

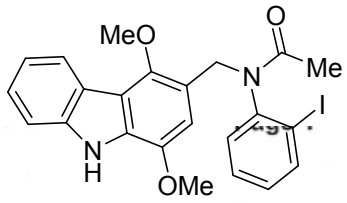
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of *N*-((1,4-dimethoxy-9*H*-carbazol-3-yl)methyl)-*N*-(2-iodophenyl)acetamide (12).



12

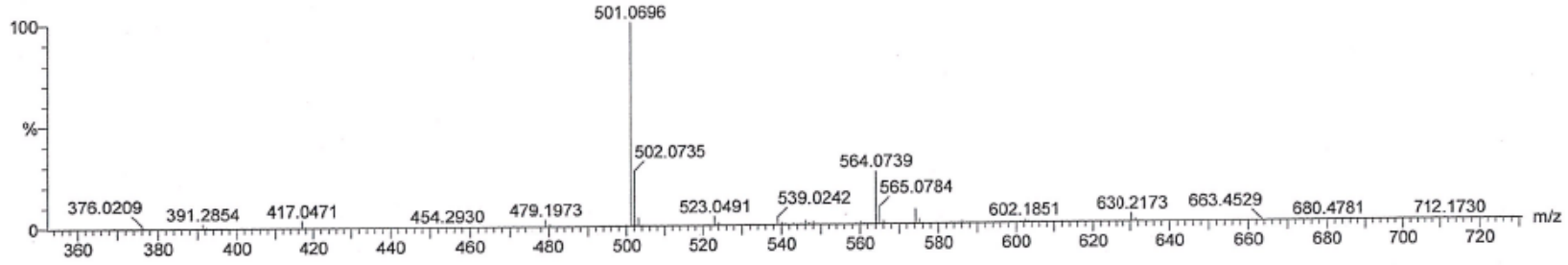
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
27 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-25 H: 0-25 N: 0-2 O: 0-4 I: 0-1

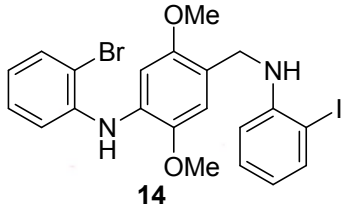
Carbazole Iodo amine Acetyl
130926020 13 (0.479) Cm (13:16-1:2)

1: TOF MS ES+
1.24e+005

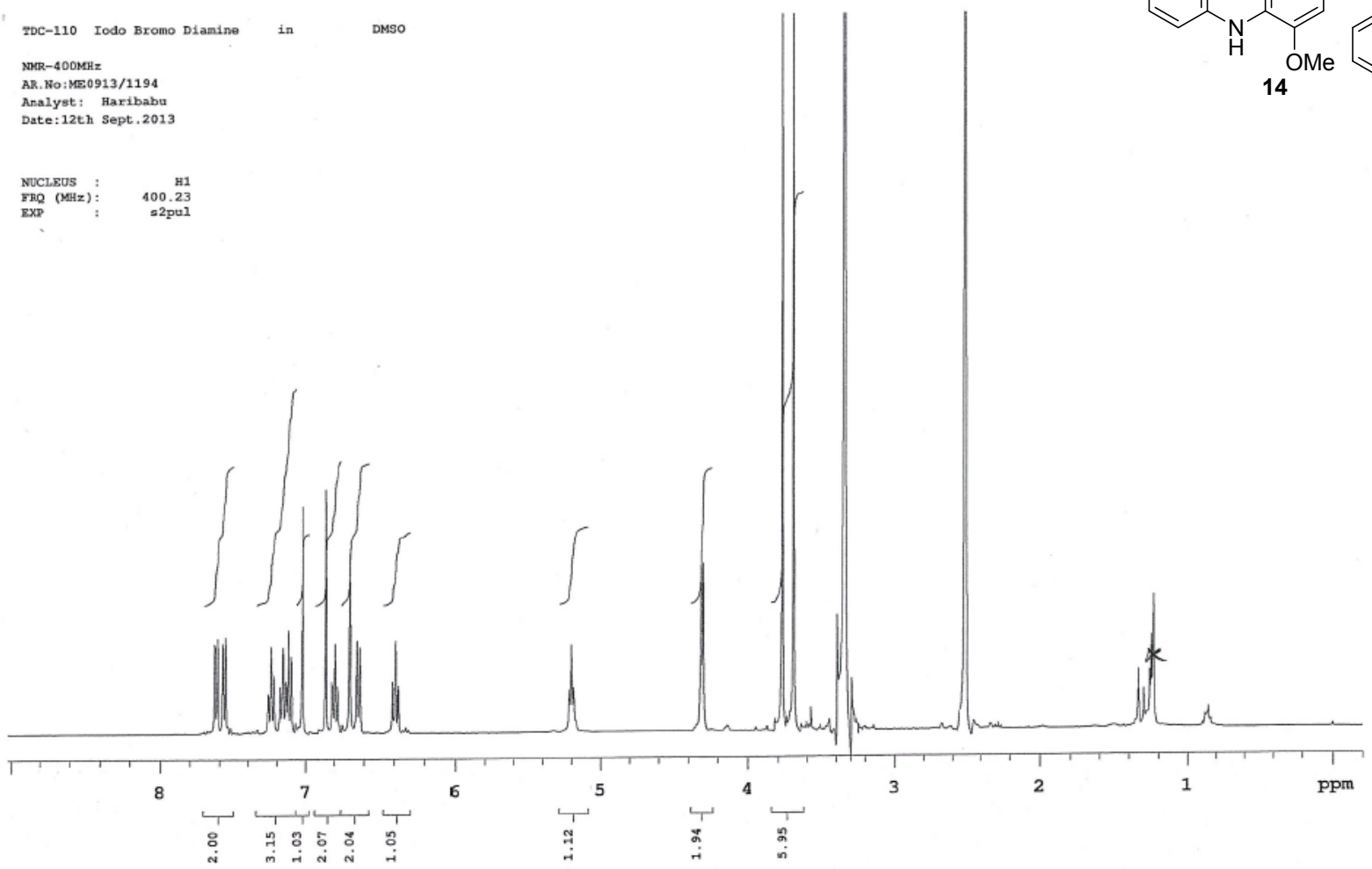


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	LBE	i-FIT	Formula
501.0696	501.0675	2.1	4.2	13.5	41.2	C23 H22 N2 O3 I

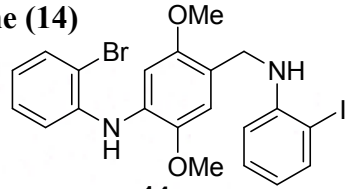
^{1}H NMR of *N*-(2-bromophenyl)-4-(((2-iodophenyl) amino)methyl)-2,5-dimethoxyaniline (14)



14

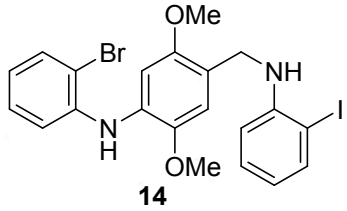


Expansion of ^{1}H NMR of *N*-(2-bromophenyl)-4-(((2-iodophenyl) amino)methyl)-2,5-dimethoxyaniline (14)

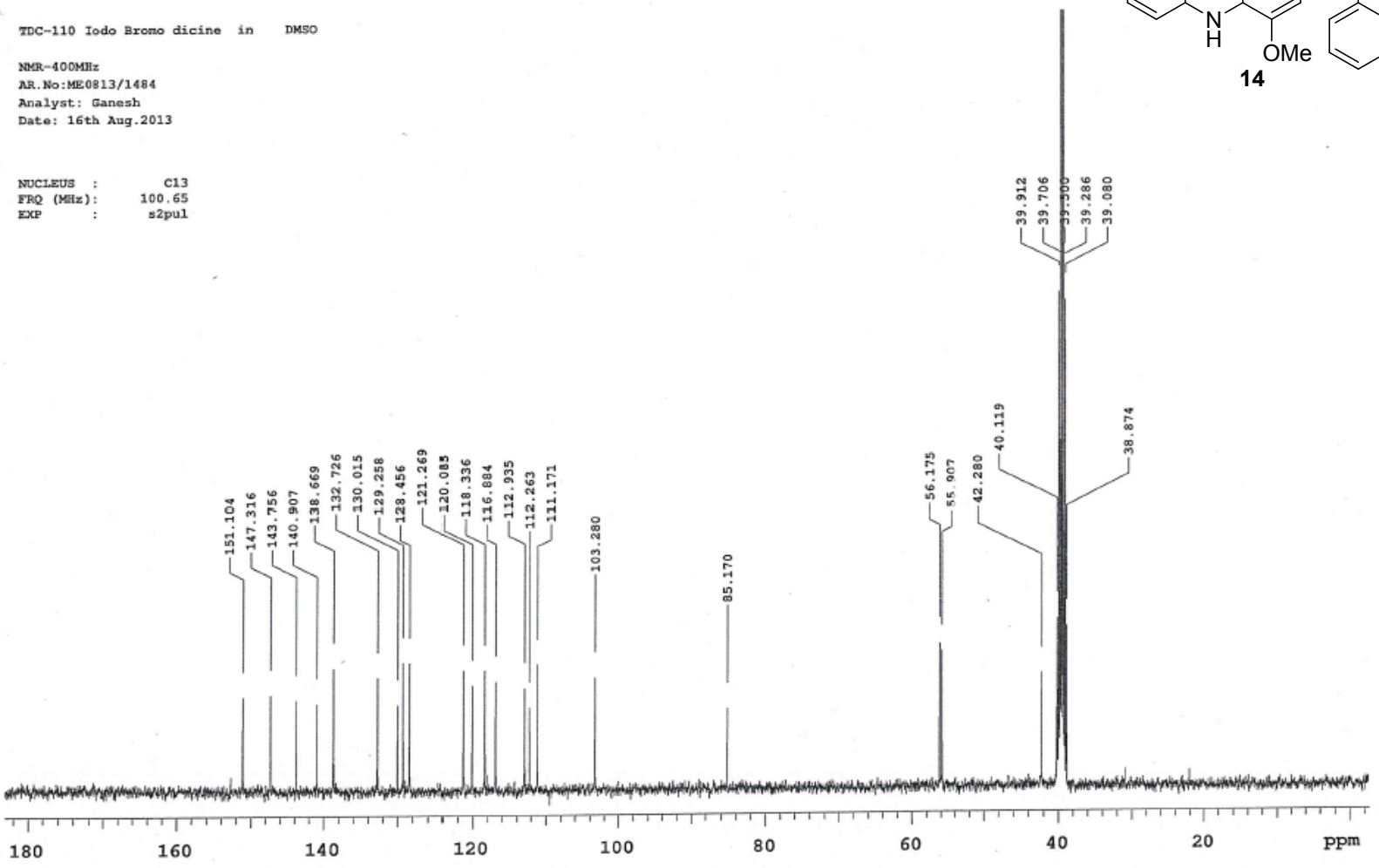


14

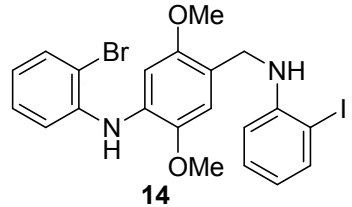
^{13}C NMR of *N*-(2-bromophenyl)-4-(((2-iodophenyl) amino)methyl)-2,5-dimethoxyaniline (14)



14



Mass Spectrum of *N*-(2-bromophenyl)-4-(((2-iodophenyl) amino)methyl)-2,5-dimethoxyaniline (14)



14

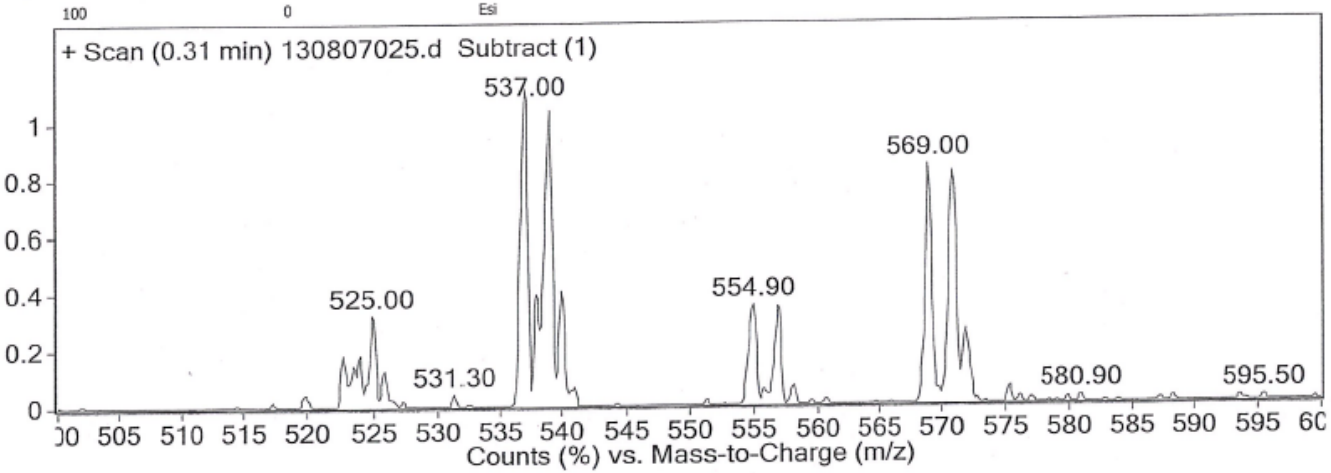
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130807025.d	**Sample Name**	IODO BROMO DIAMINE
Sample Type	Sample	**Position**	Vial 45
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

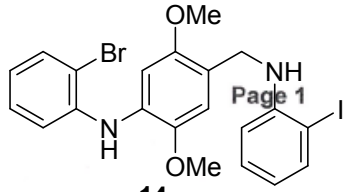
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of *N*-(2-bromophenyl)-4-(((2-iodophenyl) amino)methyl)-2,5-dimethoxyaniline (14)



14

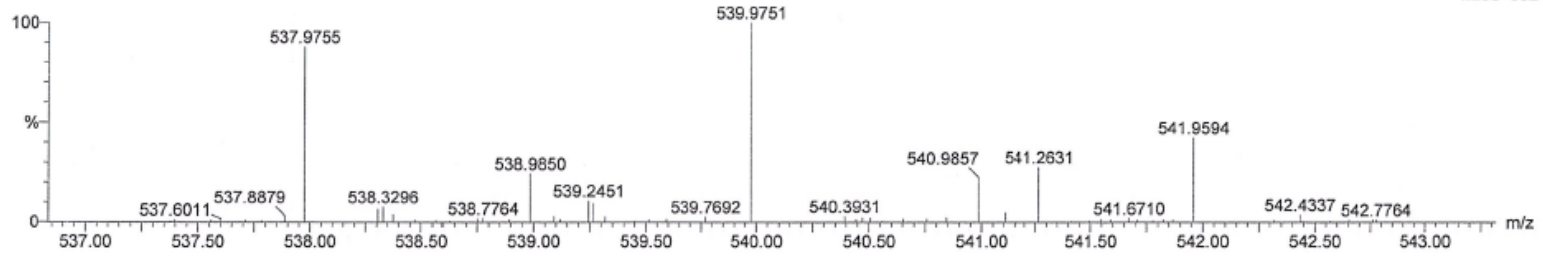
Elemental Composition Report

Single Mass Analysis
Tolerance = 12.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
42 formula(e) evaluated with 1 results within limits (up to 10 best isotopic matches for each mass)
Elements Used:
C: 0-21 H: 0-21 N: 0-3 O: 0-2 Br: 0-1 I: 0-1

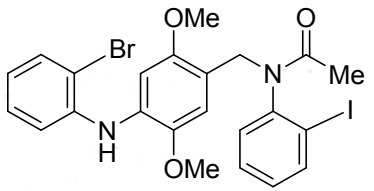
IODO BROMO DIAMINE
131008003 7 (0.251) Cm (6:7-1)

1: TOF MS ES+
4.23e+002



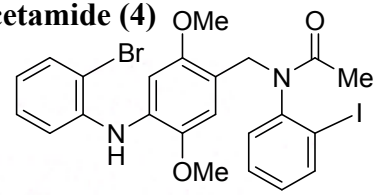
Minimum:				-5.0		
Maximum:		5.0	12.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
538.9850	538.9831	1.9	3.5	11.5	187.5	C21 H21 N2 O2 Br I

^{1}H NMR of *N*-(4-((2-bromophenyl)amino)-2,5-dimethoxybenzyl)-*N*-(2-iodophenyl)acetamide (4)

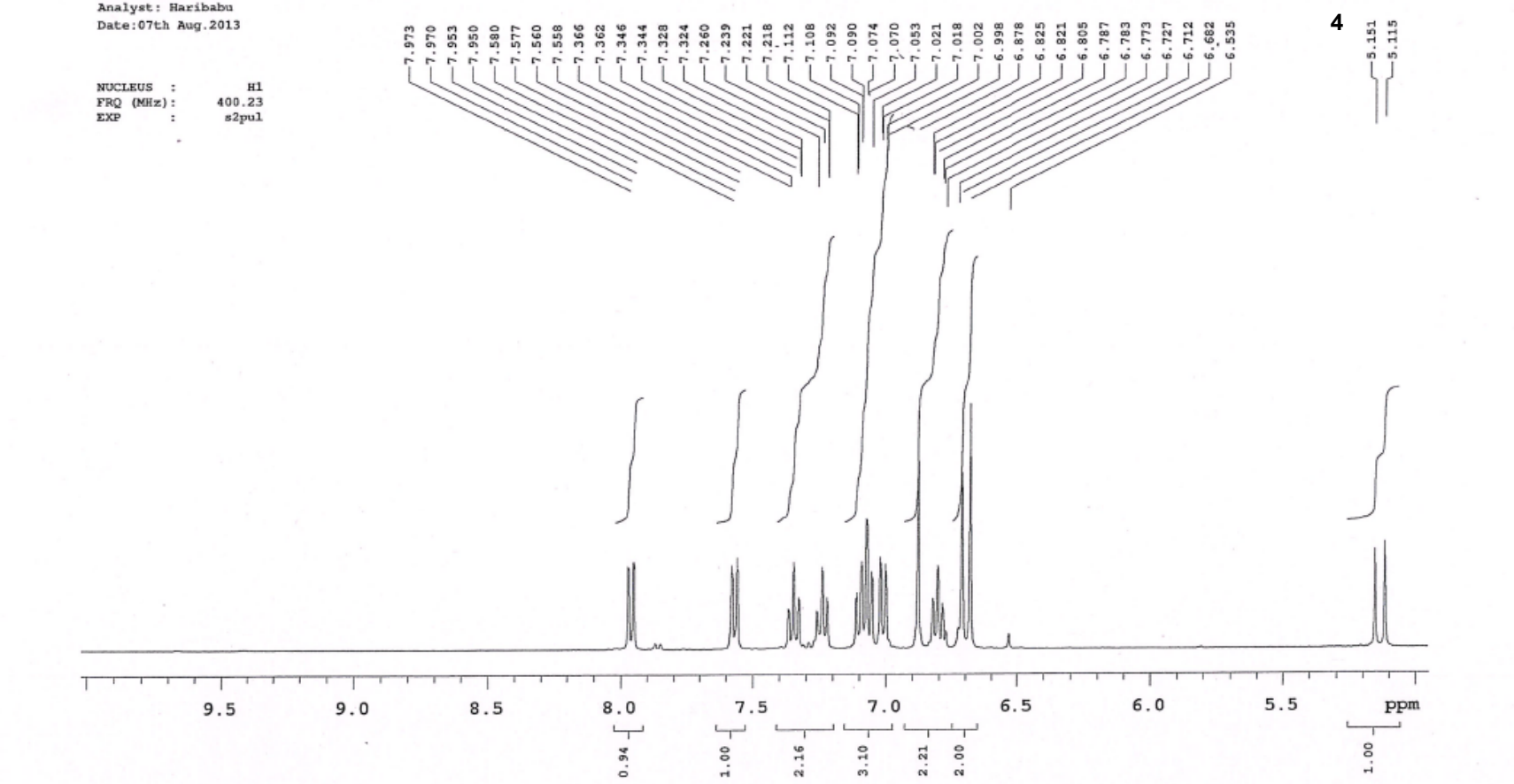


4

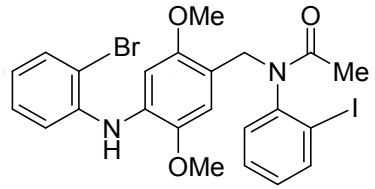
Expansion of ^{1}H NMR of *N*-(4-((2-bromophenyl)amino)-2,5-dimethoxybenzyl)-*N*-(2-iodophenyl)acetamide (4)



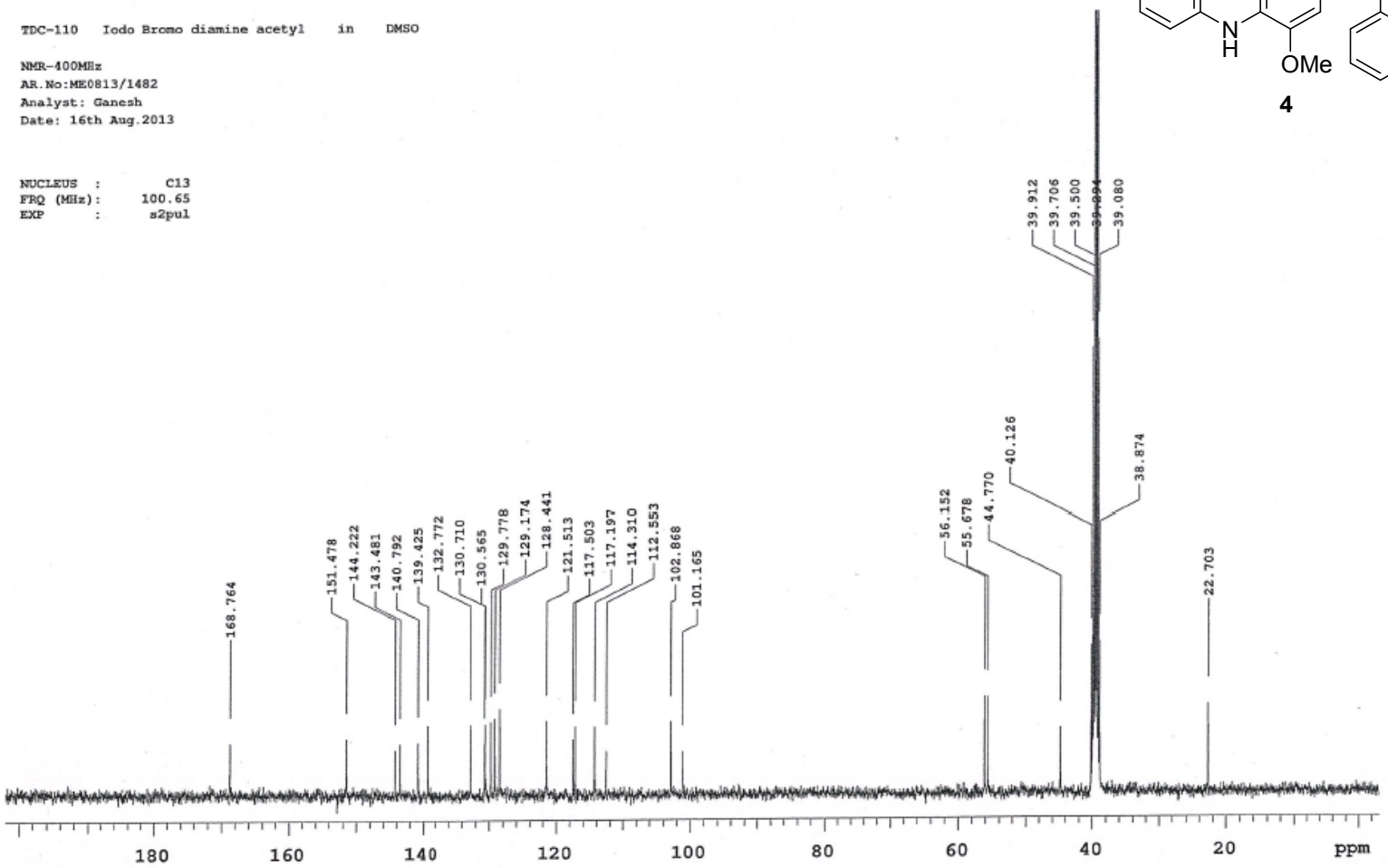
4



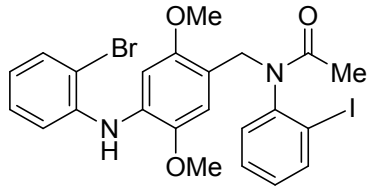
^{13}C NMR of *N*-(4-((2-bromophenyl)amino)-2,5-dimethoxybenzyl)-*N*-(2-iodophenyl)acetamide (4)



4



Mass Spectrum of *N*-(4-((2-bromophenyl)amino)-2,5-dimethoxybenzyl)-*N*-(2-iodophenyl)acetamide (4)



4

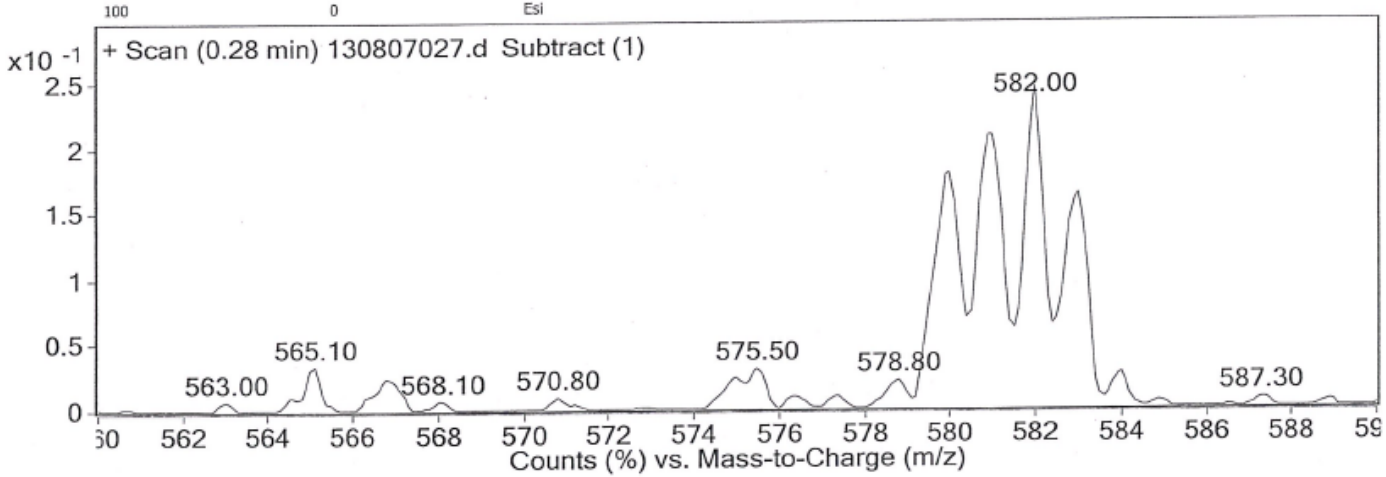
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130807027.d	**Sample Name**	IODO BROMO ACETYL F-II
Sample Type	Sample	**Position**	Vial 47
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

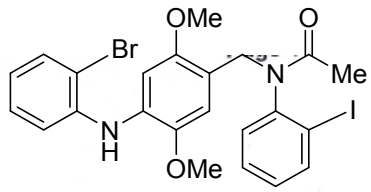
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of *N*-(4-((2-bromophenyl)amino)-2,5-dimethoxybenzyl)-*N*-(2-iodophenyl)acetamide (4)



4

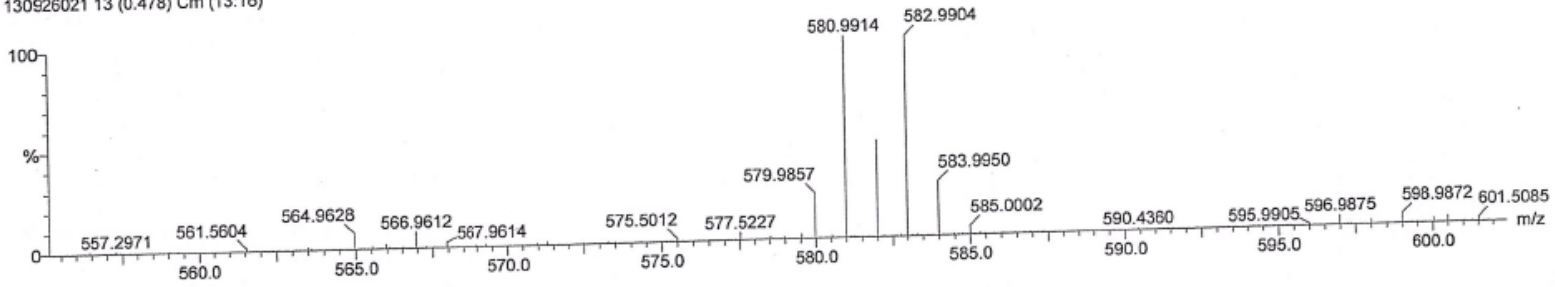
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
57 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-25 H: 0-25 N: 0-2 O: 0-4 Br: 0-1 I: 0-1

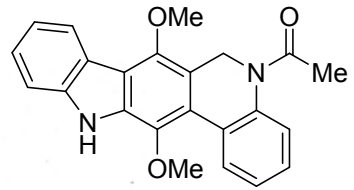
Iodobromo diamine acetyl
130926021 13 (0.478) Cm (13:16)

1: TOF MS ES+
1.39e+004

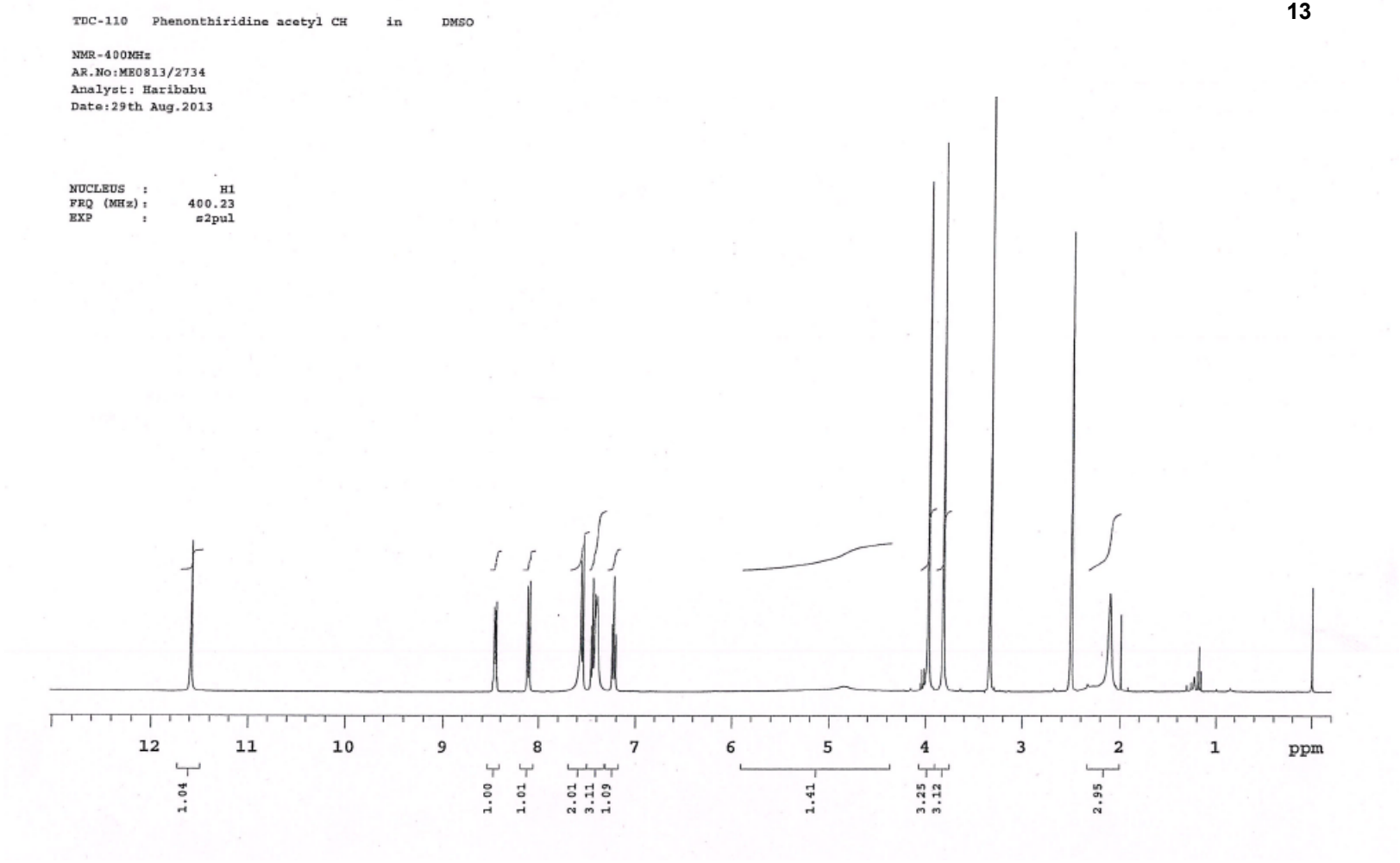


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
580.9914	580.9937	-2.3	-4.0	12.5	631.5	C23 H23 N2 O3 Br I

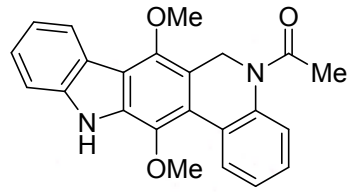
^{1}H NMR of 1-(7,13-dimethoxy-6,12-dihydro-5*H*-indolo[3,2-*j*]phenanthridin-5-yl)ethanone (13)



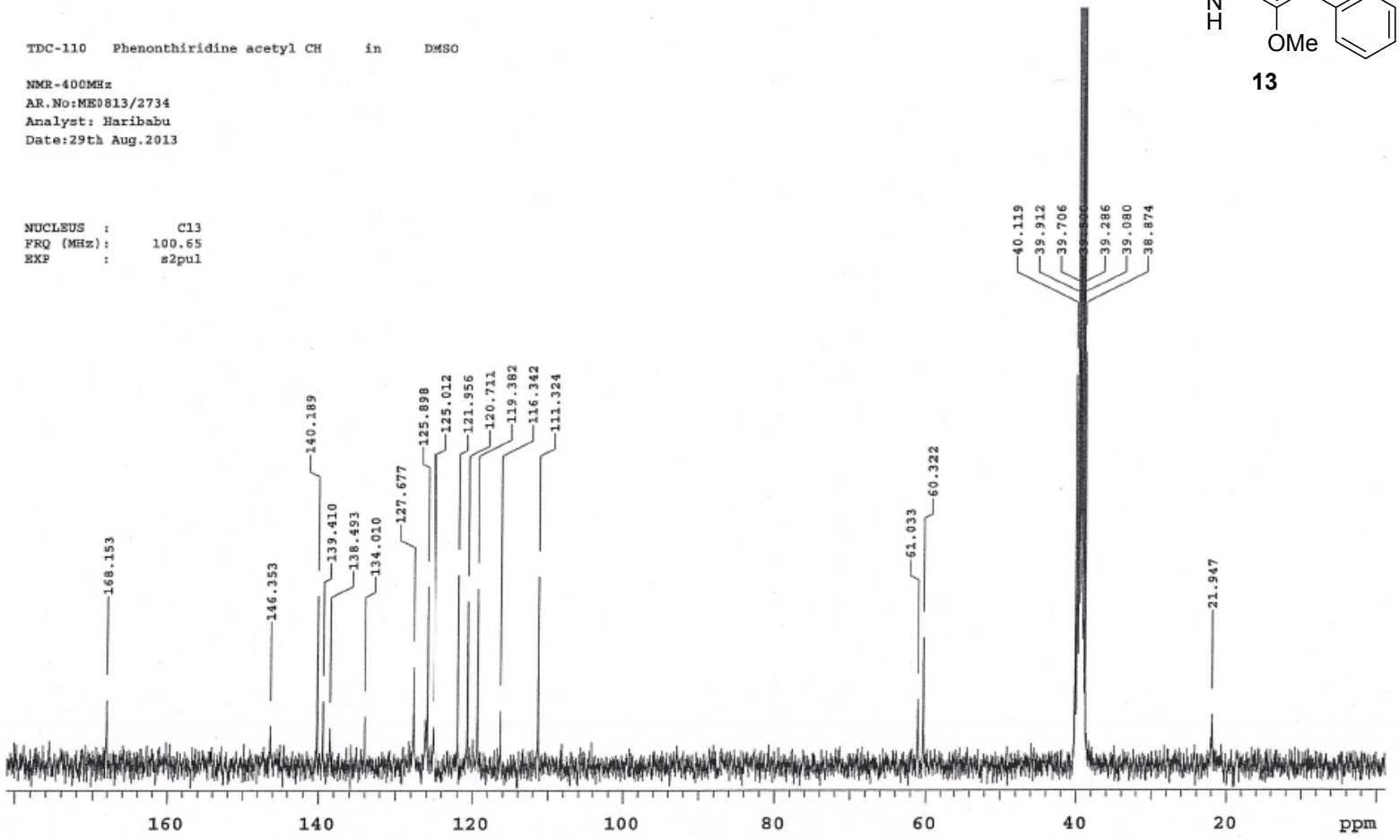
13



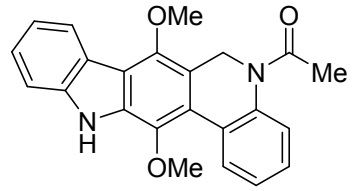
^{13}C NMR of 1-(7,13-dimethoxy-6,12-dihydro-5*H*-indolo[3,2-*j*]phenanthridin-5-yl)ethanone (13)



13



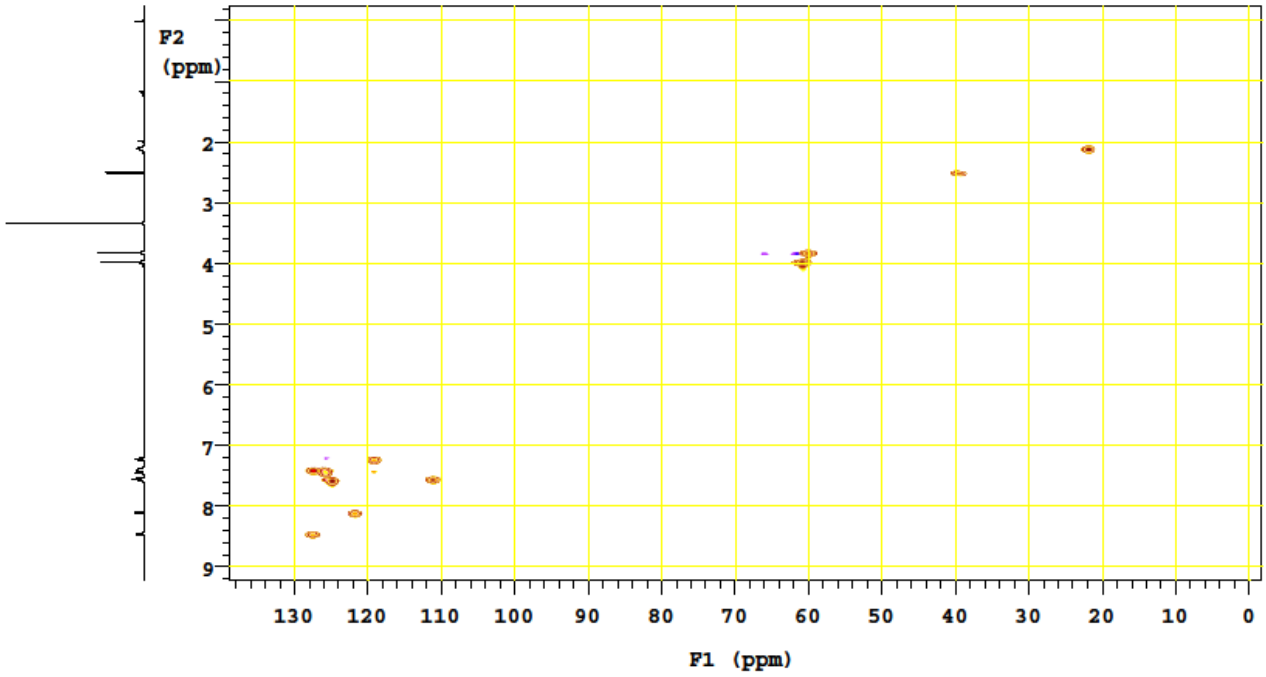
HSQC of 1-(7,13-dimethoxy-6,12-dihydro-5*H*-indolo[3,2-*j*]phenanthridin-5-yl)ethanone (13)



13

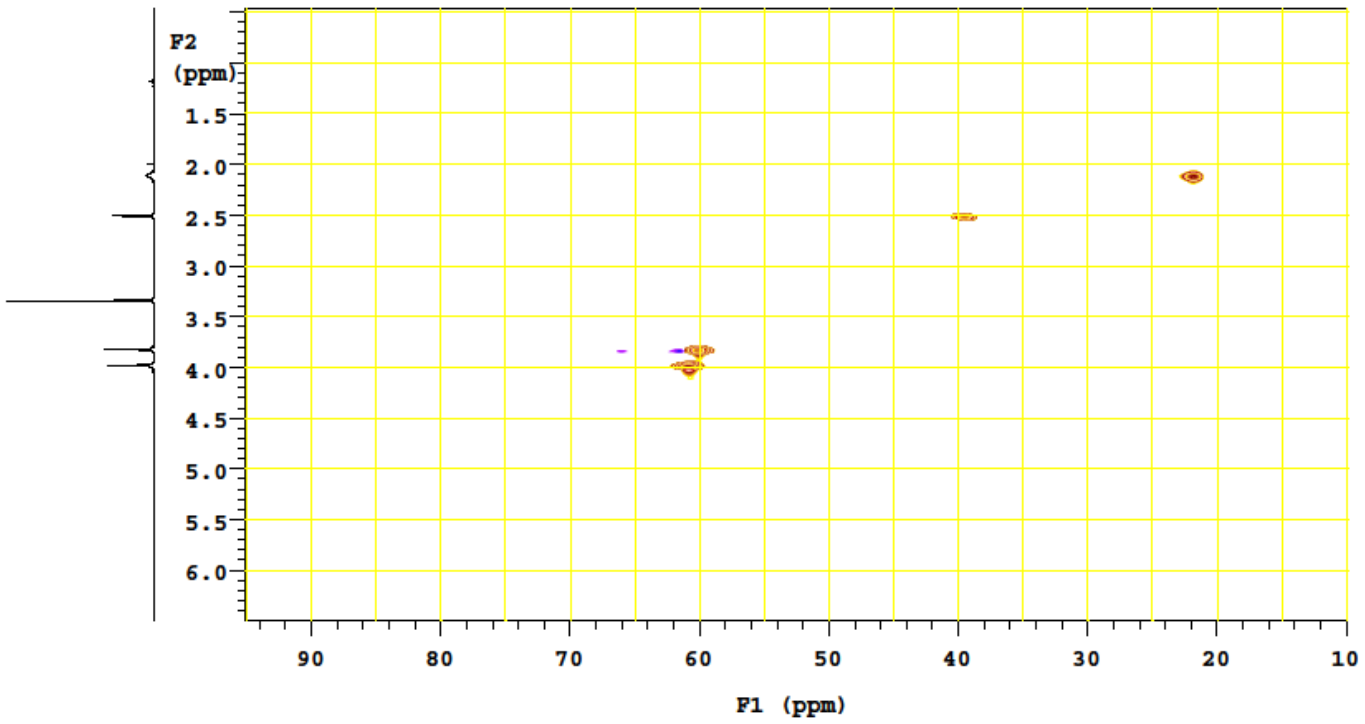
TDC-110 Phenonthiridine acetyl CH in DMSO

NMR-400MHz
AR.No:ME0813/2734
Analyst: Haribabu
Date:29th Aug.2013



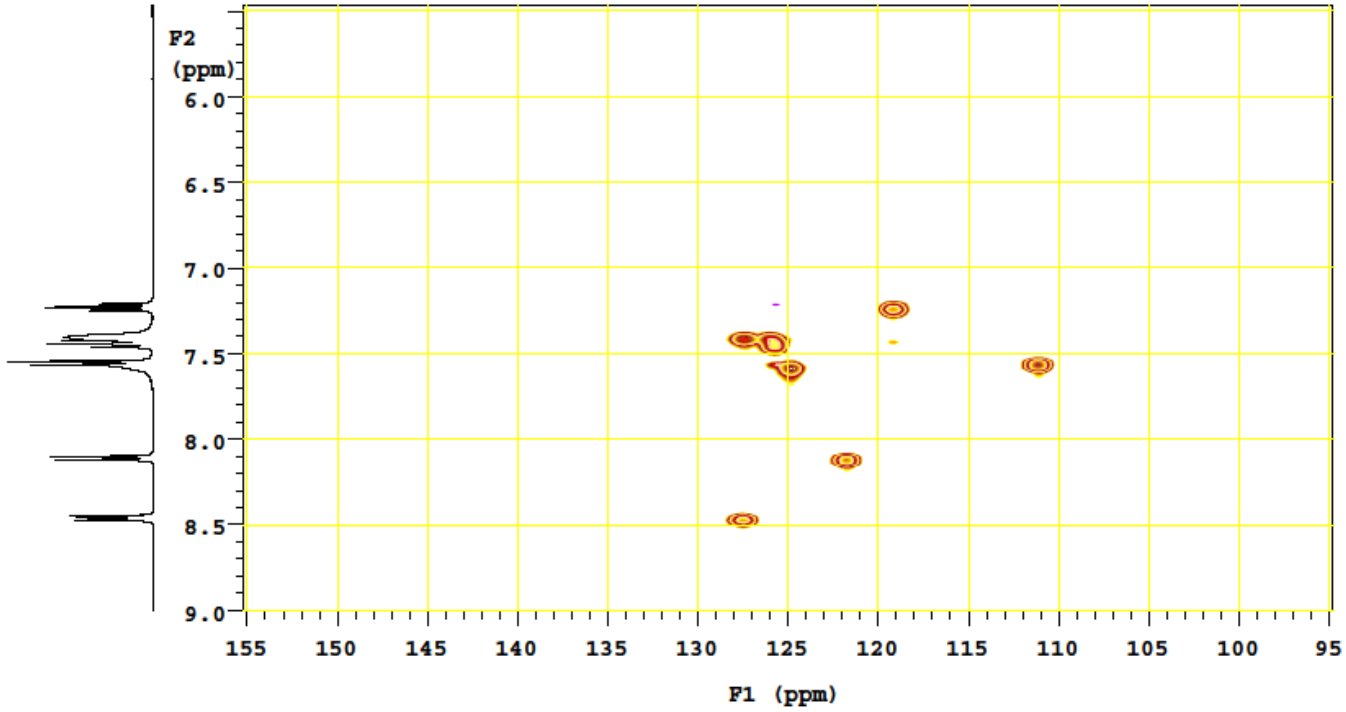
TDC-110 Phenonthiridine acetyl CH in DMSO

NMR-400MHz
AR.No:ME0813/2734
Analyst: Haribabu
Date:29th Aug.2013

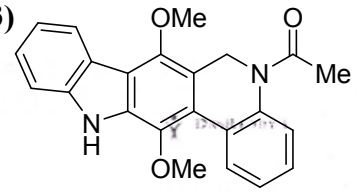


TDC-110 Phenonthiridine acetyl CH in DMSO

NMR-400MHz
AR.No:ME0813/2734
Analyst: Haribabu
Date:29th Aug.2013



Mass Spectrum of 1-(7,13-dimethoxy-6,12-dihydro-5*H*-indolo[3,2-*j*]phenanthridin-5-yl)ethanone (13)



13

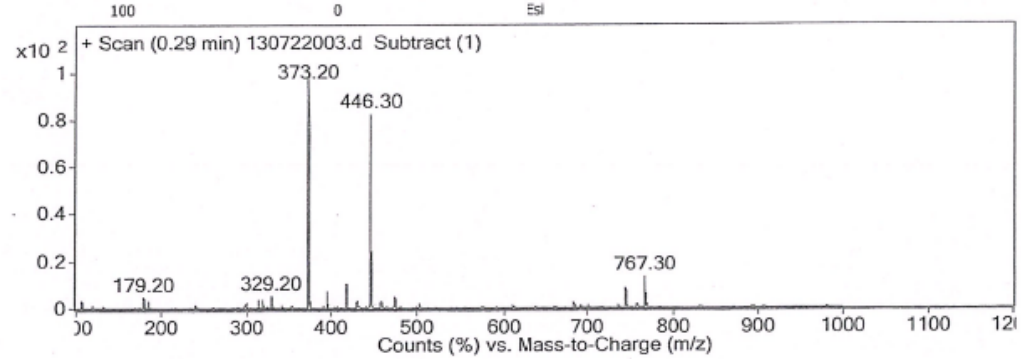
CPS,MIYAPUR

LCMS Analysis Report

Data Filename	130722003.d	**Sample Name**	Phenanthracin acid
Sample Type	Sample	**Position**	Vial 87
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

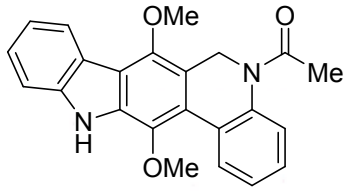
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---

HRMS of 1-(7,13-dimethoxy-6,12-dihydro-5*H*-indolo[3,2-*j*]phenanthridin-5-yl)ethanone (13)



13

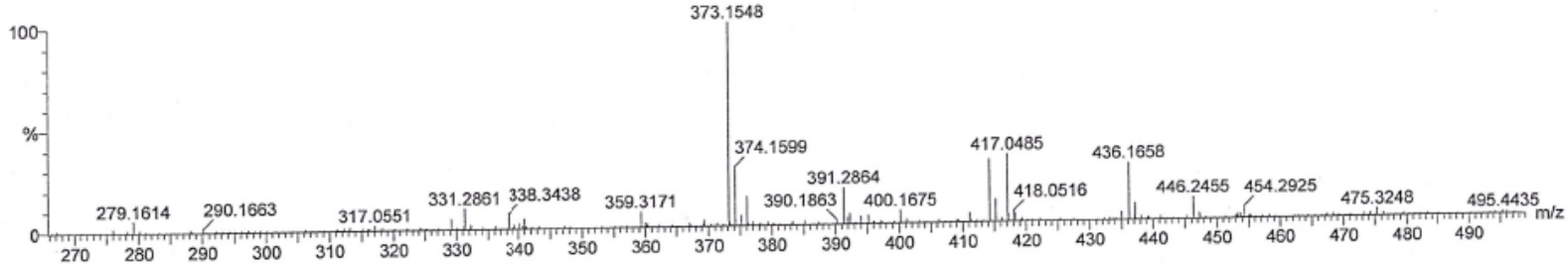
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
46 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-25 H: 0-25 N: 0-2 O: 0-4 Br: 0-1

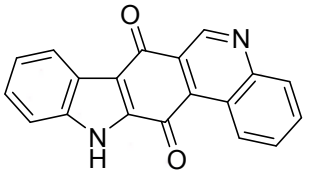
Phenanthindine acetyl
130926013 12 (0.419) Cm (12:17-1:2)

1: TOF MS ES+
3.48e+004

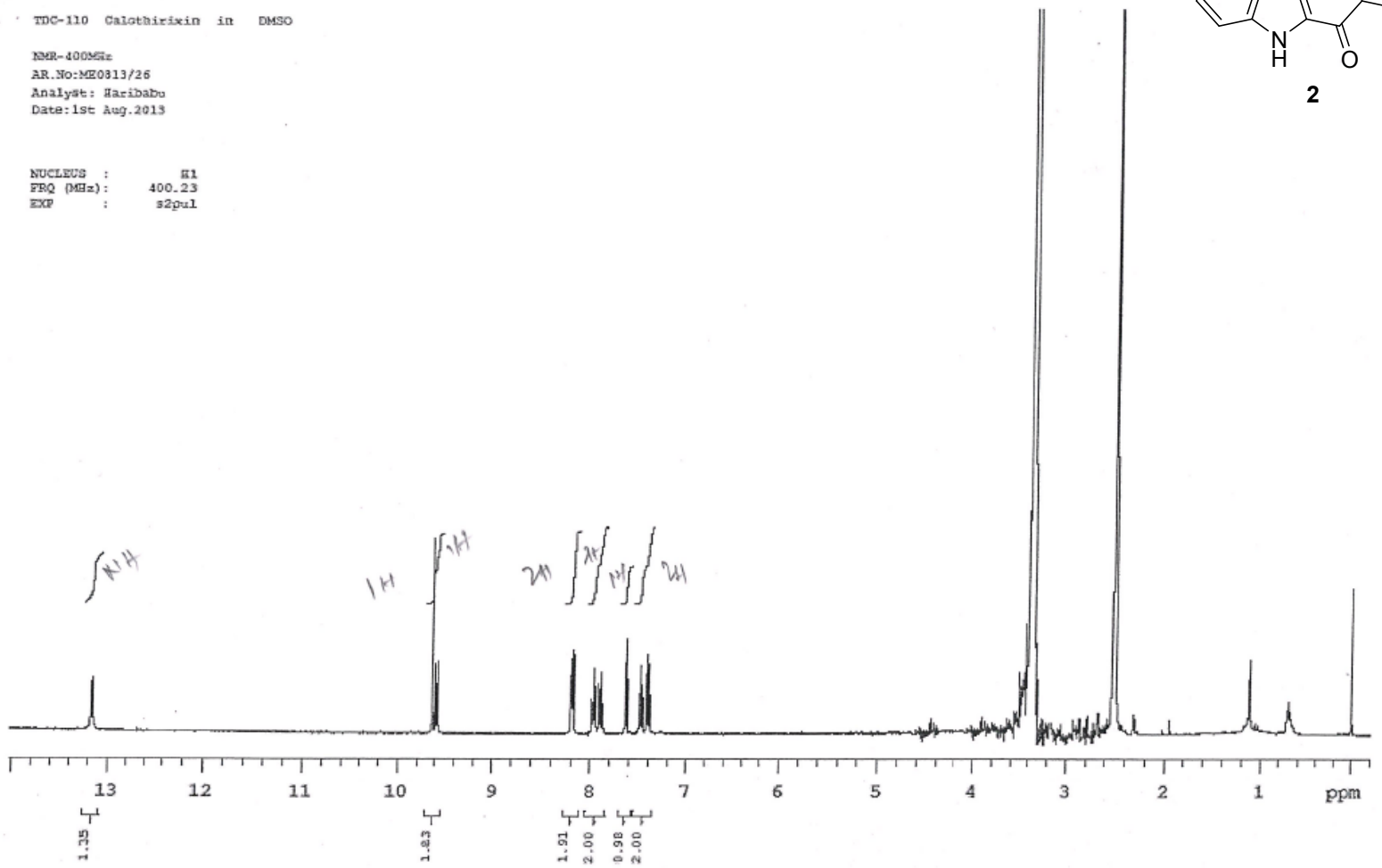


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
373.1548	373.1552	-0.4	-1.1	14.5	55.4	C23 H21 N2 O3

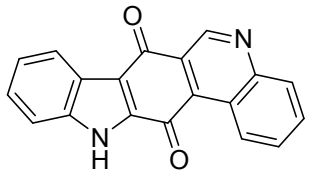
^{1}H NMR of Calothrixin B (2)



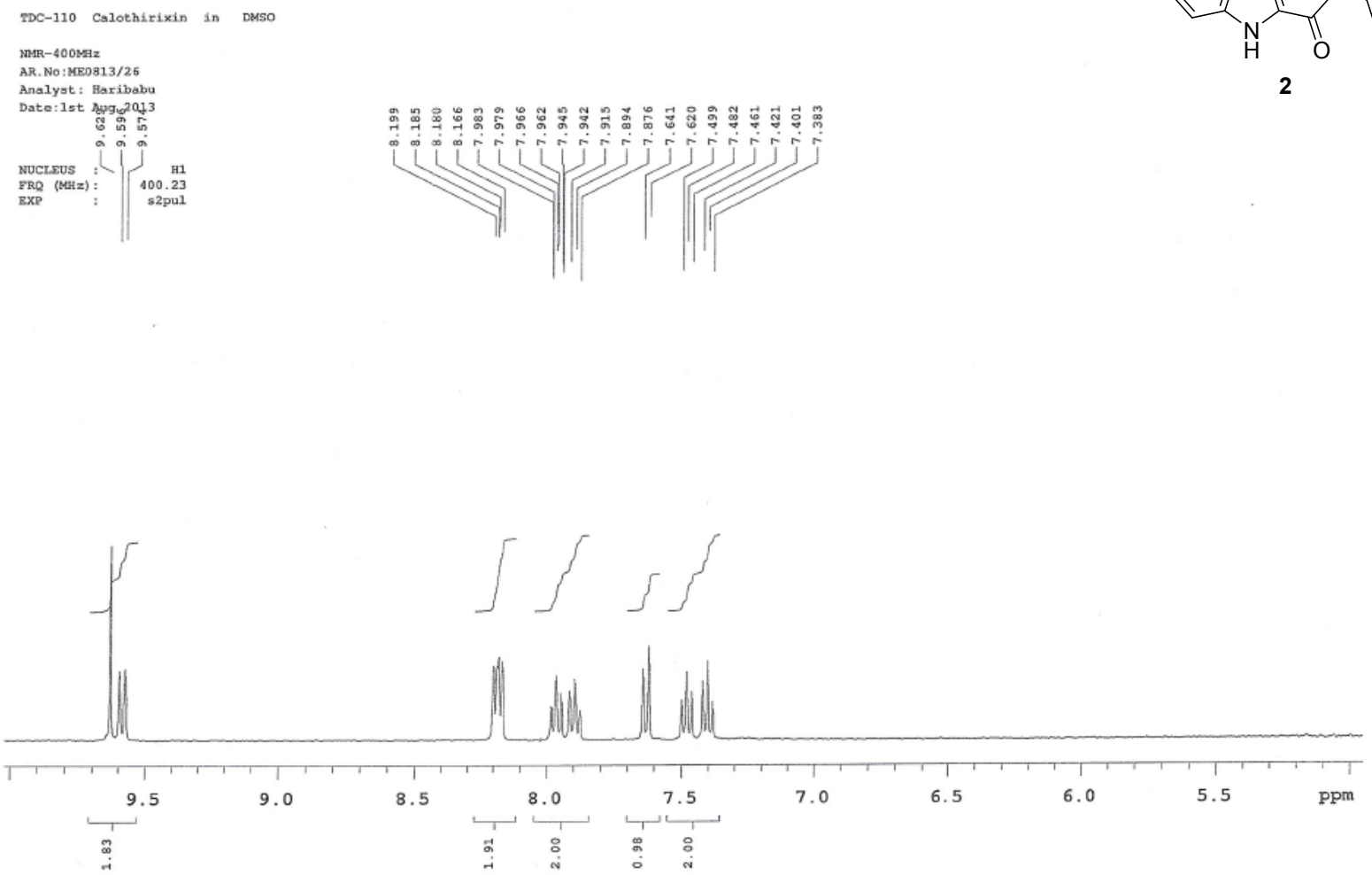
2



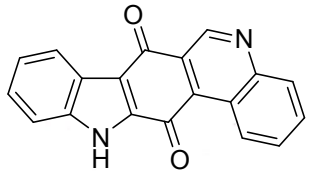
Expansion of ^{1}H NMR of Calothrixin B (2)



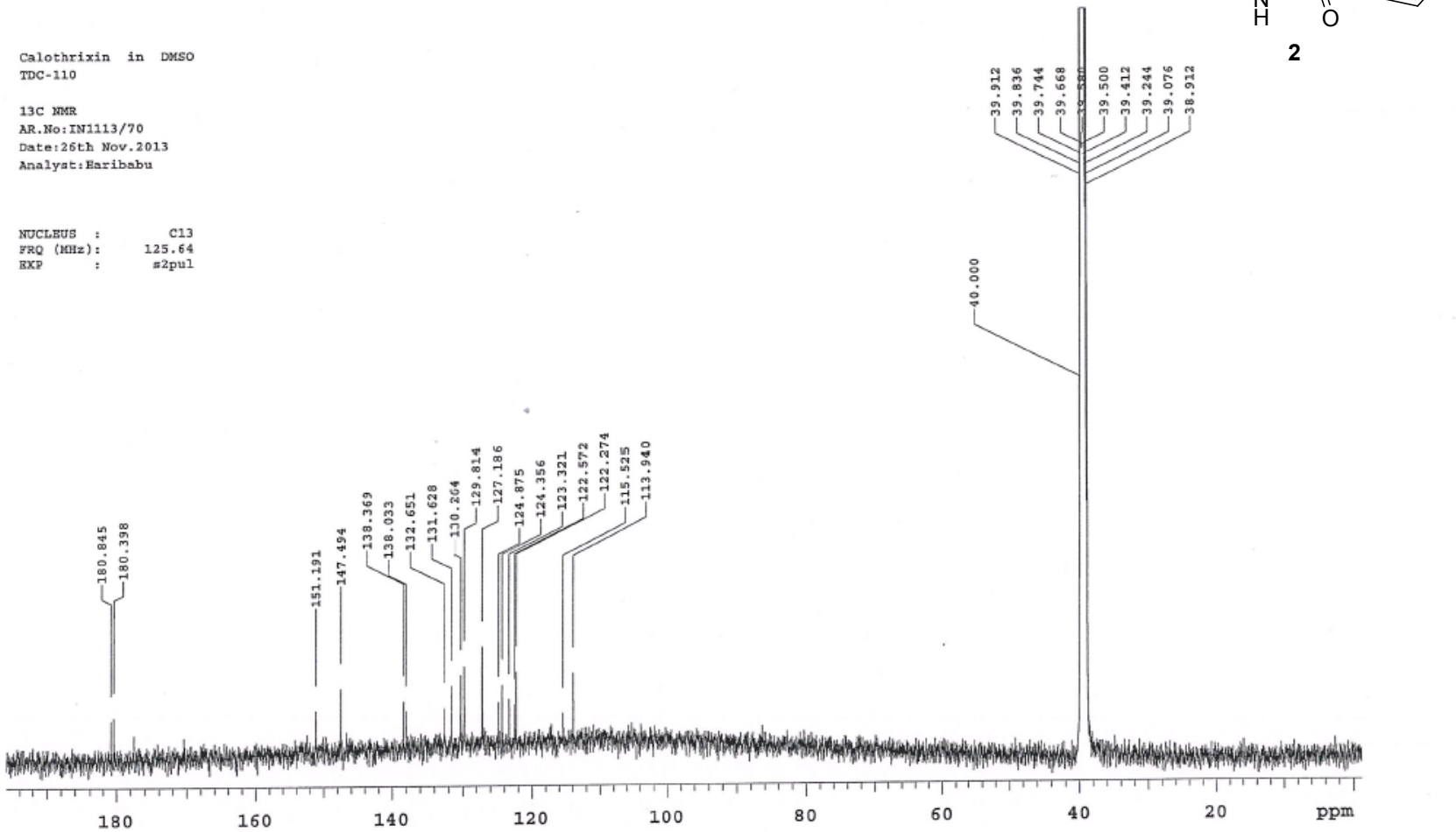
2



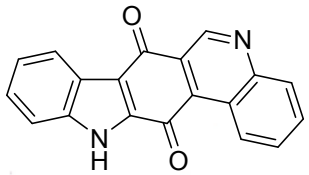
^{13}C NMR of Calothrixin B (2)



2



Mass Spectrum of Calothrixin B (2)



2

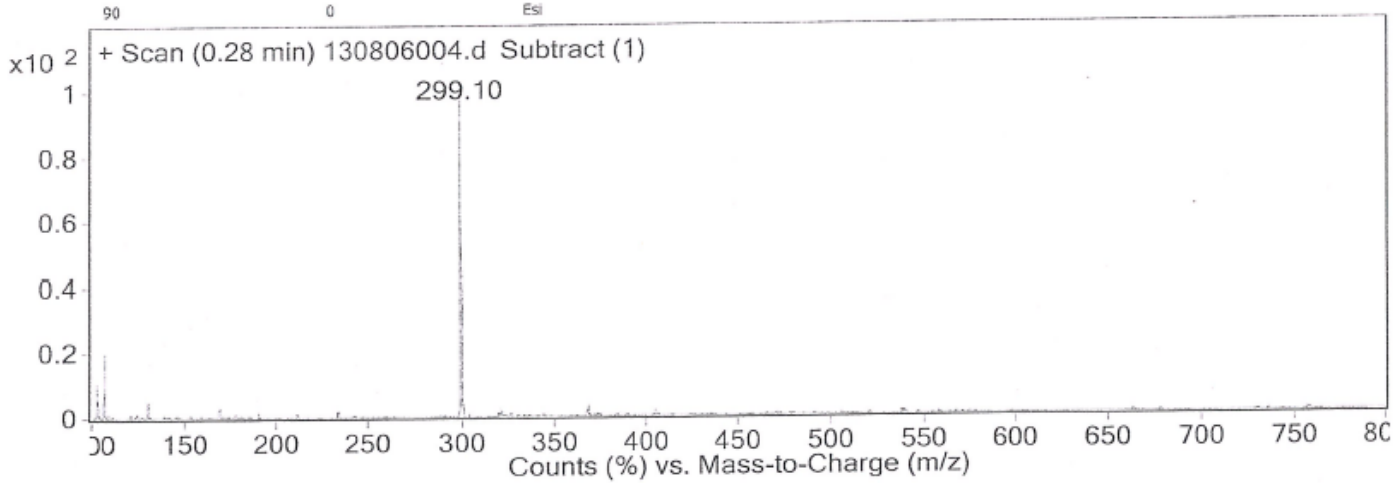
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130806004.d	**Sample Name**	Calothrixin
Sample Type	Sample	**Position**	Vial 95
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

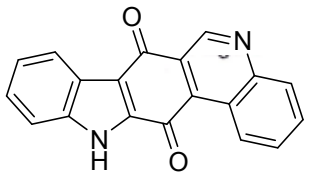
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
90	0	Esi



--- End Of Report ---

HRMS of Calothrixin B (2)



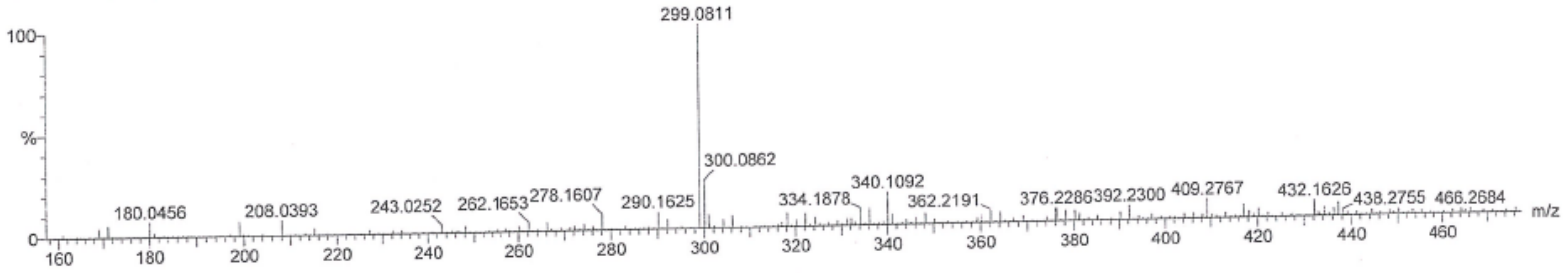
2

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

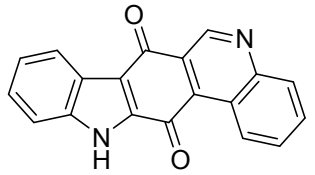
Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-20 H: 0-20 N: 0-2 O: 0-2
Calothrixin
130926017 9 (0.338) Cm (9:12-1:4)

1: TOF MS ES+
1.55e+004



Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
299.0811	299.0821	-1.0	-3.3	15.5	226.3	C19 H11 N2 O2

HMBC of Calothrixin B (2)

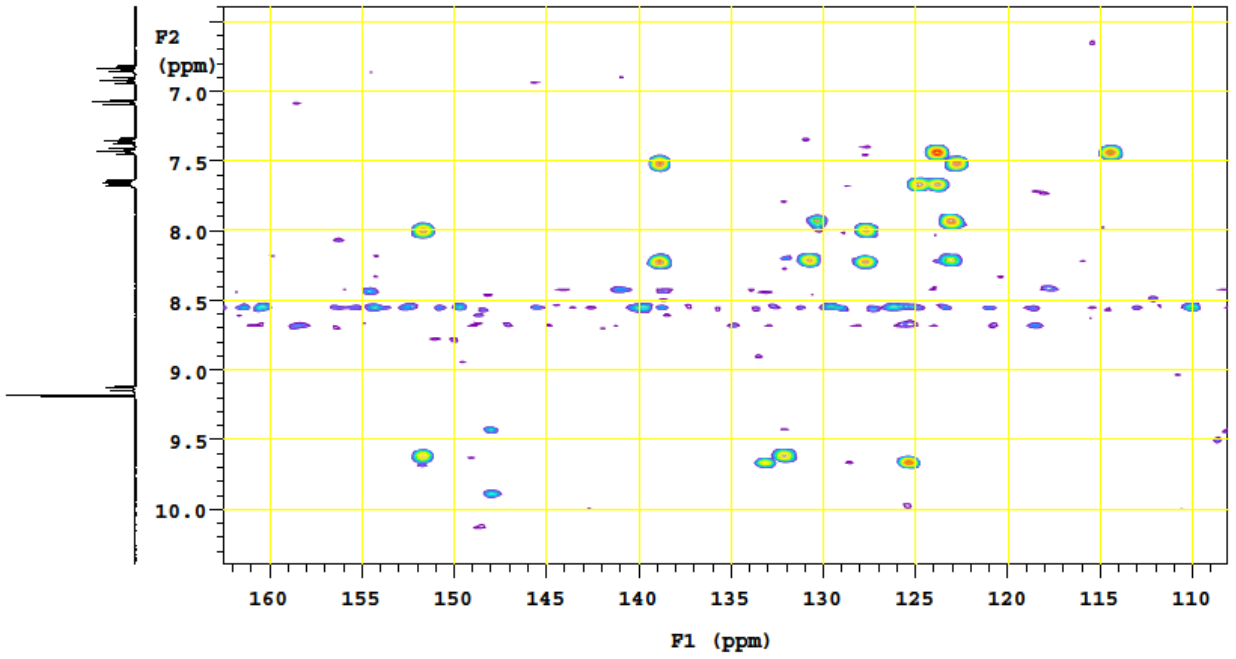


2

TDC-110 Calothrixin B in DMSO

NMR-400MHz
AR.No:ME1113/2106
Analyst: Haribabu
Date: 25th Nov.2013

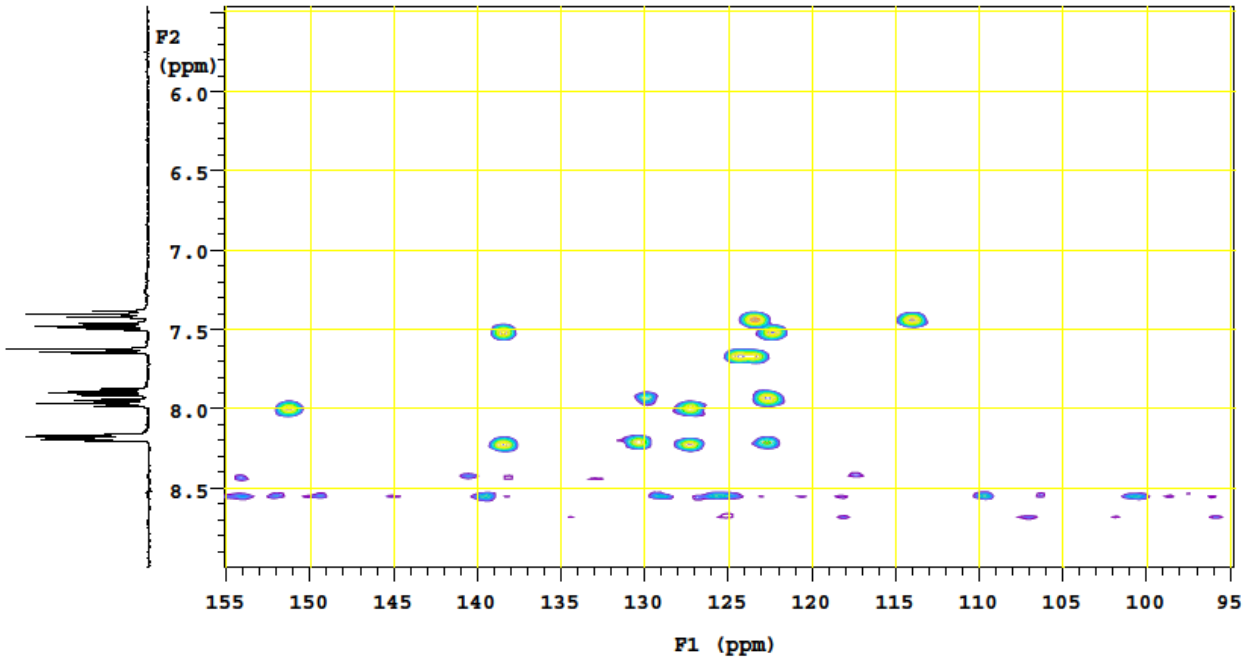
NUCLEUS : H1
FRQ (MHz): 400.22
EXP : gHMBC



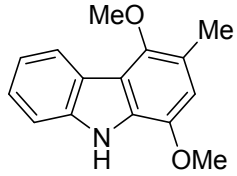
TDC-110 Calothrixin B in DMSO

NMR-400MHz
AR.No:ME1113/2106
Analyst: Haribabu
Date: 25th Nov.2013

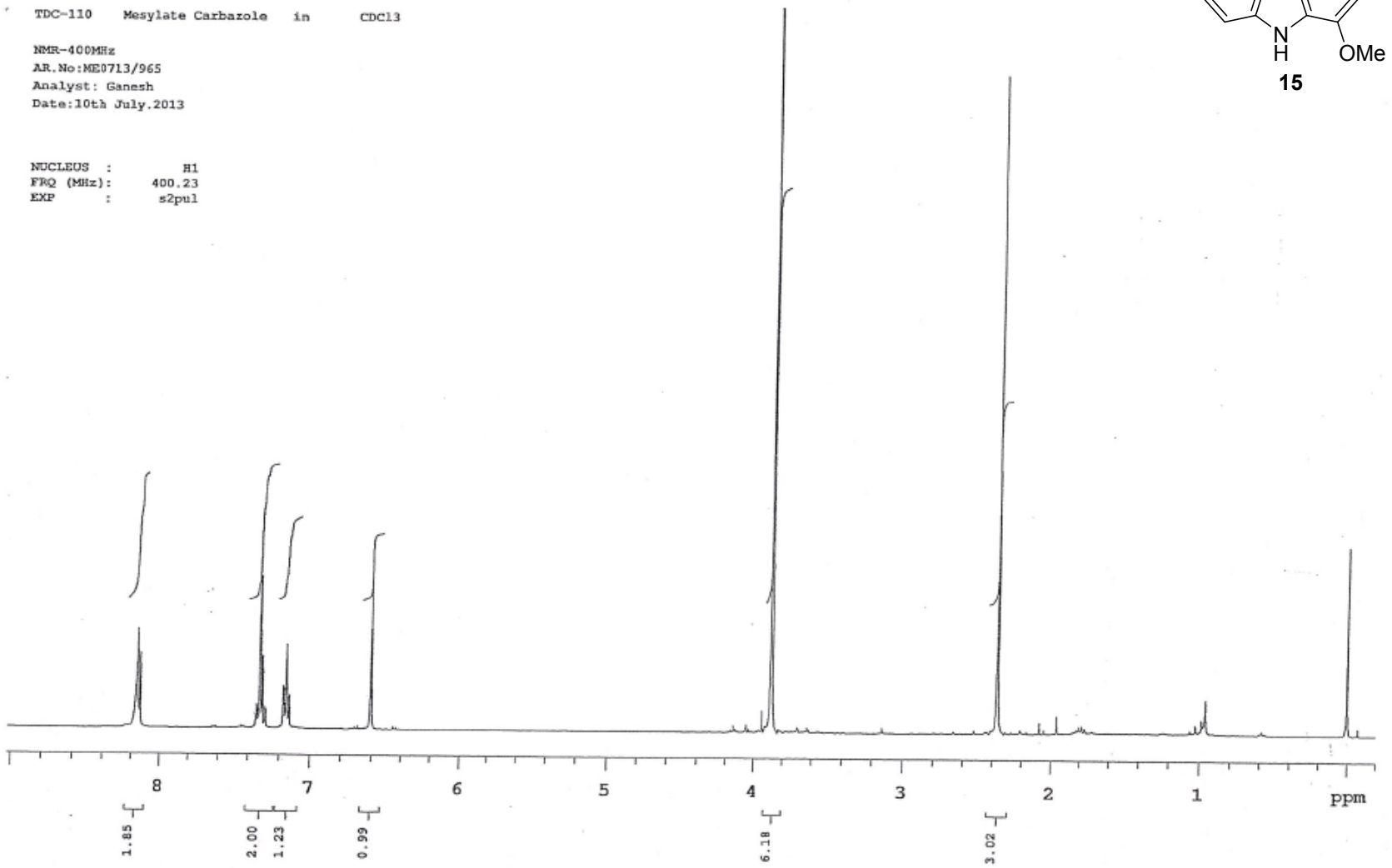
NUCLEUS : H1
FRQ (MHz): 400.22
EXP : gHMBC



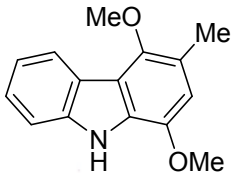
^{1}H NMR of 1,4-dimethoxy-3-methyl-9*H*-carbazole (15)



15

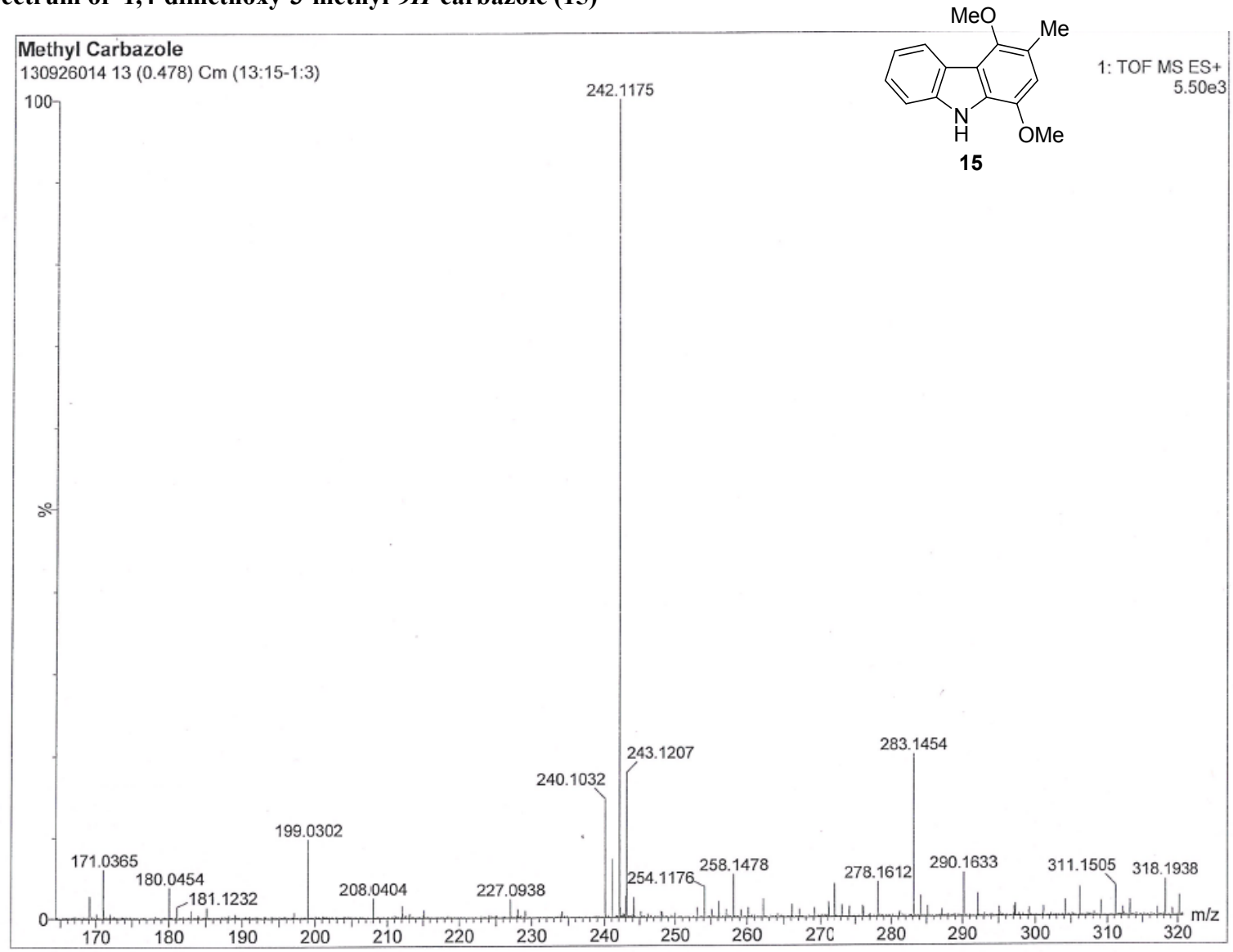


^{13}C NMR of 1,4-dimethoxy-3-methyl-9*H*-carbazole (15)

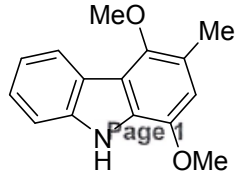


15

Mass Spectrum of 1,4-dimethoxy-3-methyl-9*H*-carbazole (15)



HRMS of 1,4-dimethoxy-3-methyl-9*H*-carbazole (15)



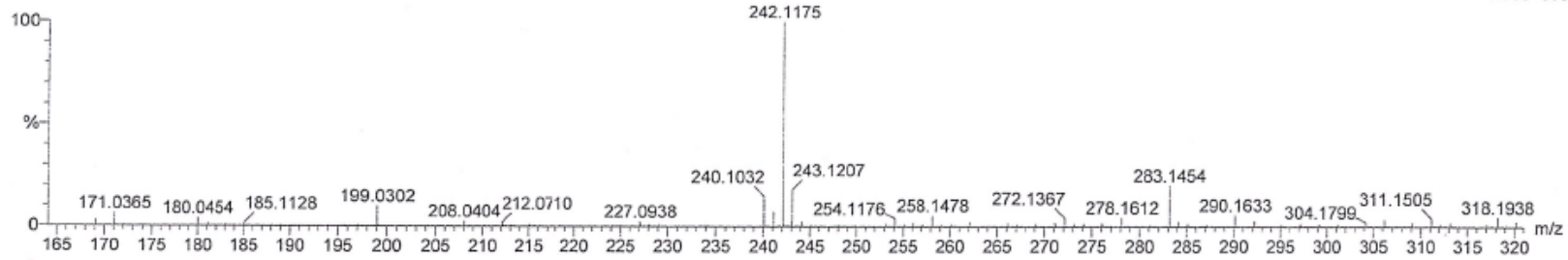
15

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

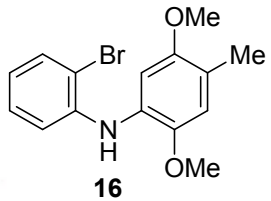
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-20 H: 0-20 N: 0-2 O: 0-2
Methyl Carbazole
130926014 13 (0.478) Cm (13:15-1:3)

1: TOF MS ES+
5.50e+003

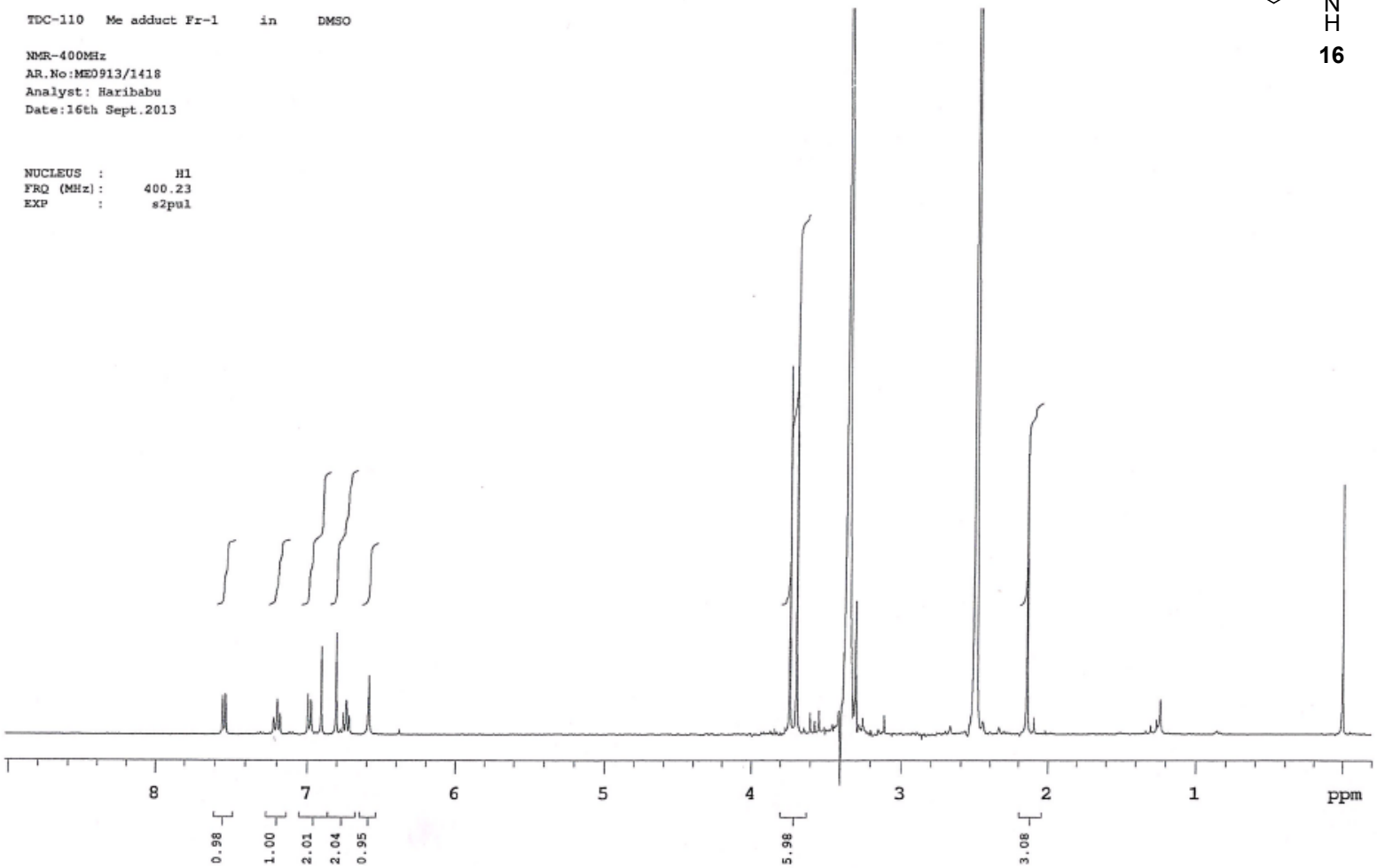


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
242.1175	242.1181	-0.6	-2.5	8.5	17.2	C15 H16 N O2

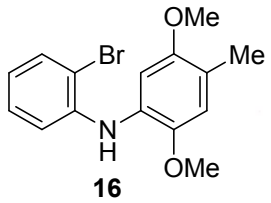
^{1}H NMR of *N*-(2-bromophenyl)-2, 5-dimethoxy-4-methylaniline (16)



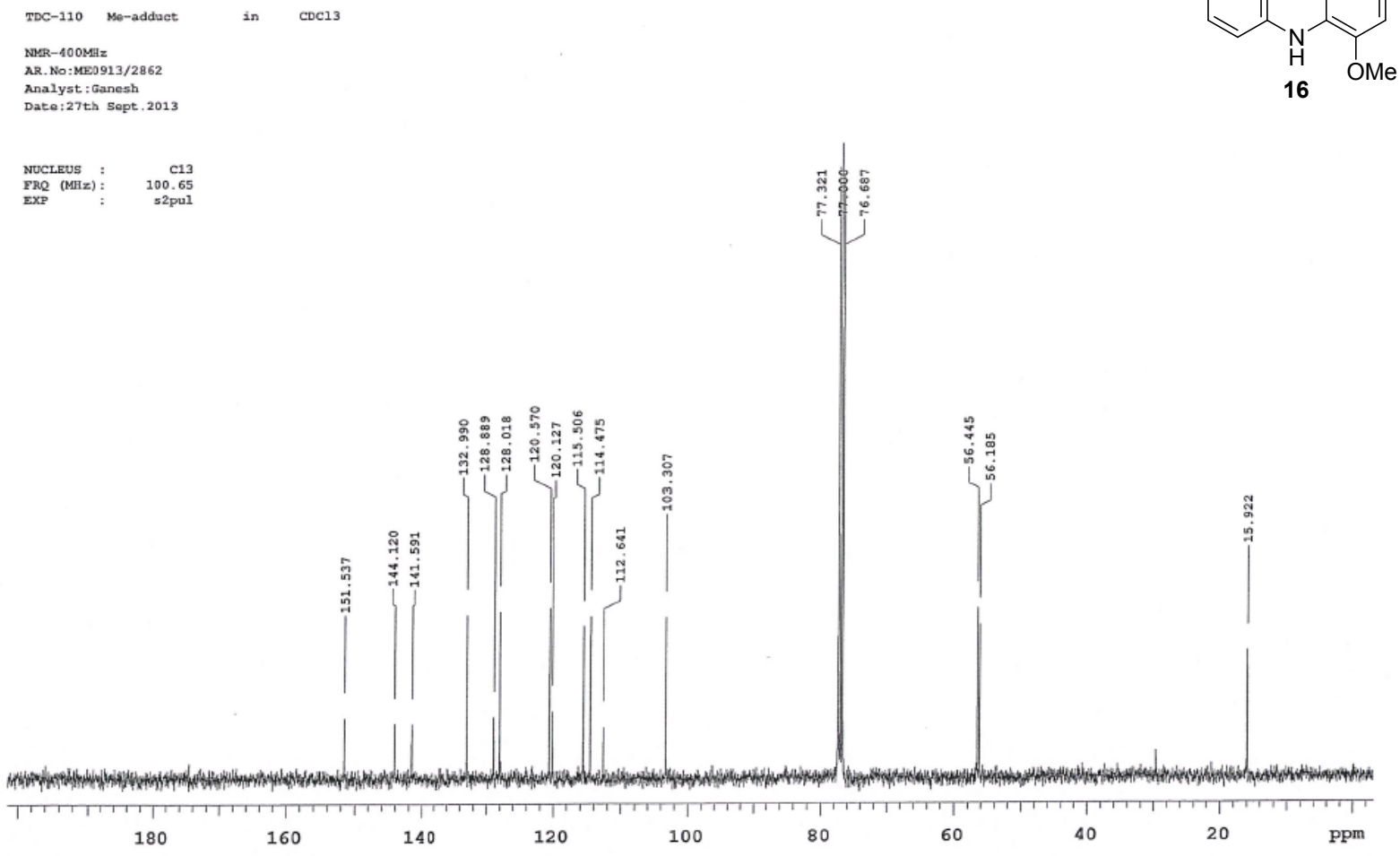
16



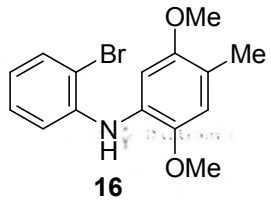
^{13}C NMR of *N*-(2-bromophenyl)-2, 5-dimethoxy-4-methylaniline (16)



16



Mass Spectrum of of *N*-(2-bromophenyl)-2, 5-dimethoxy-4-methylaniline (16)



16

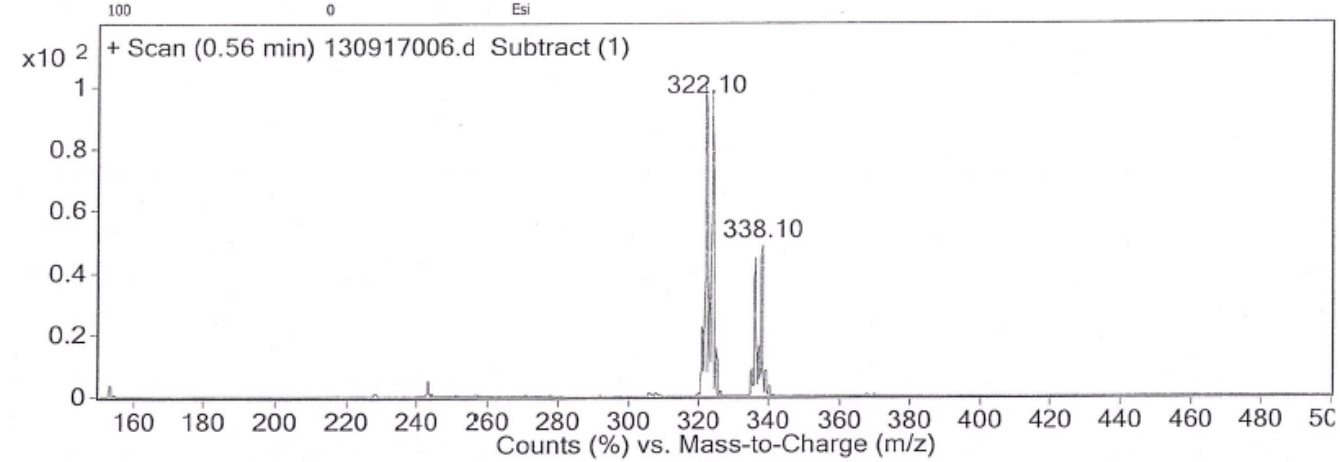
CPS,MIYAPUR

Mass Analysis Report

Data Filename	130917006.d	**Sample Name**	Me-adduct
Sample Type	Sample	**Position**	Vial 11
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	CUBI3.m	**Comment**	

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



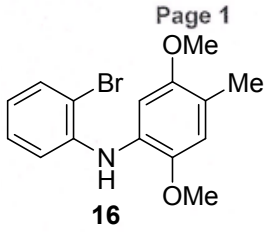
--- End Of Report ---

HRMS of *N*-(2-bromophenyl)-2, 5-dimethoxy-4-methylaniline (16)

Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

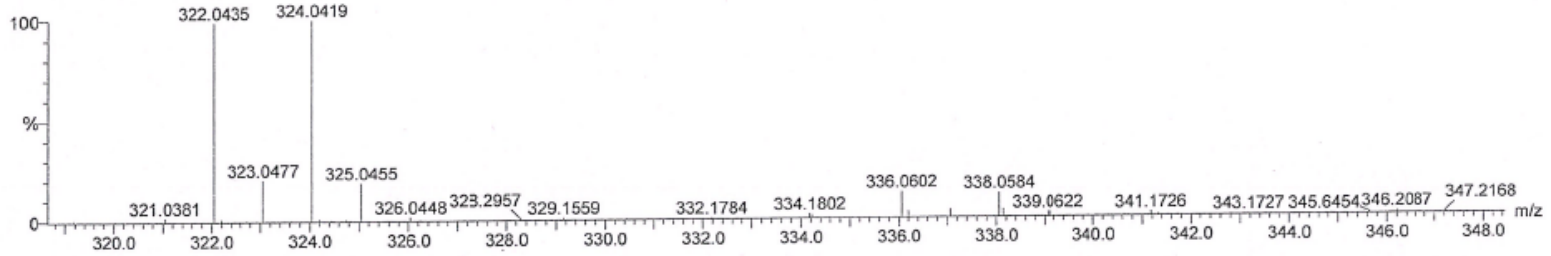
Monoisotopic Mass, Even Electron Ions
64 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-16 H: 0-18 N: 0-2 O: 0-3 Br: 0-3



16

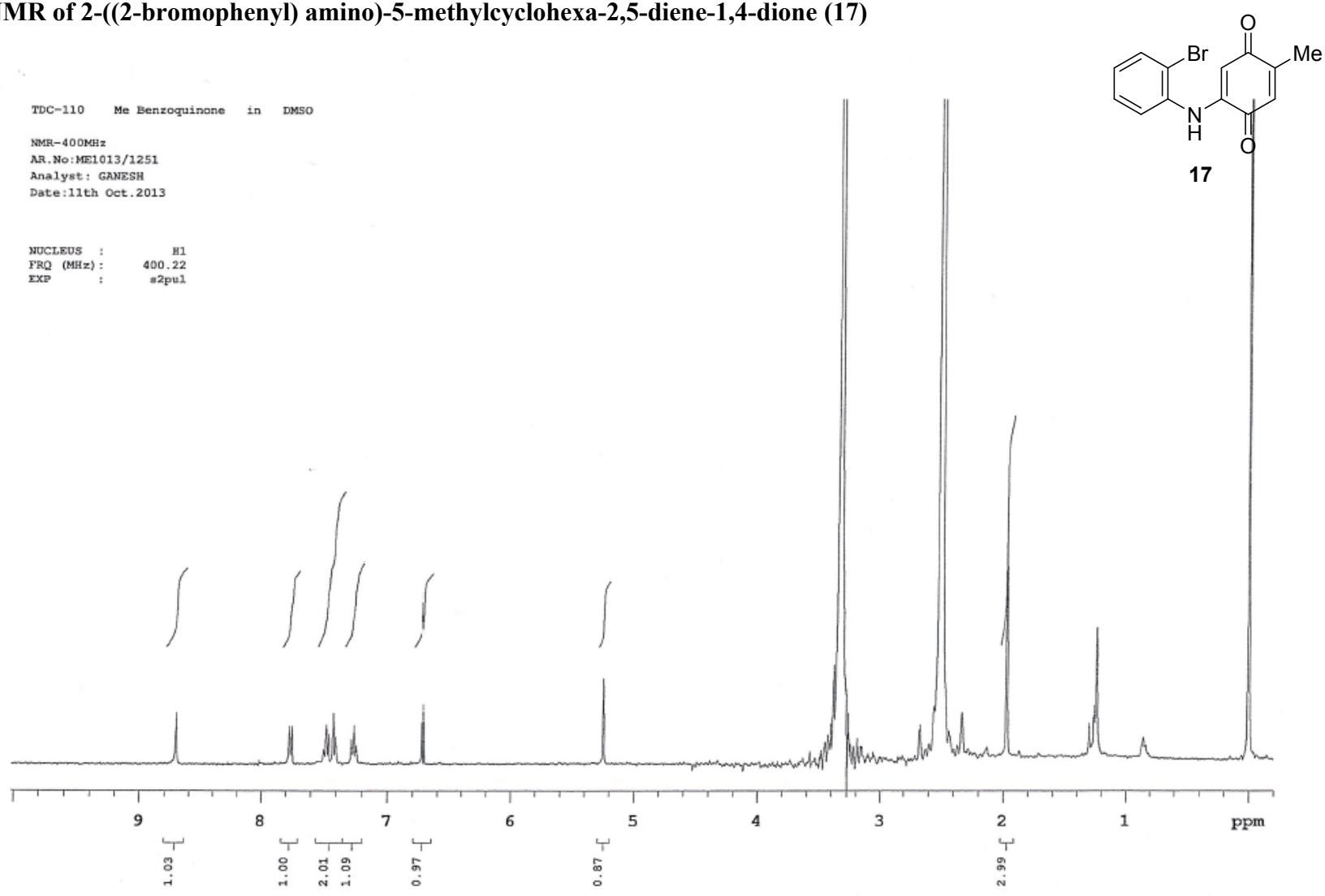
ME ADDUCT
130920016 16 (0.560) Cm (16:19-26:33)

1: TOF MS ES+
3.97e+003

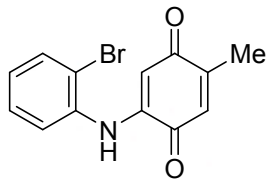


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
322.0435	322.0443	-0.8	-2.5	7.5	11.8	C15 H17 N O2 Br

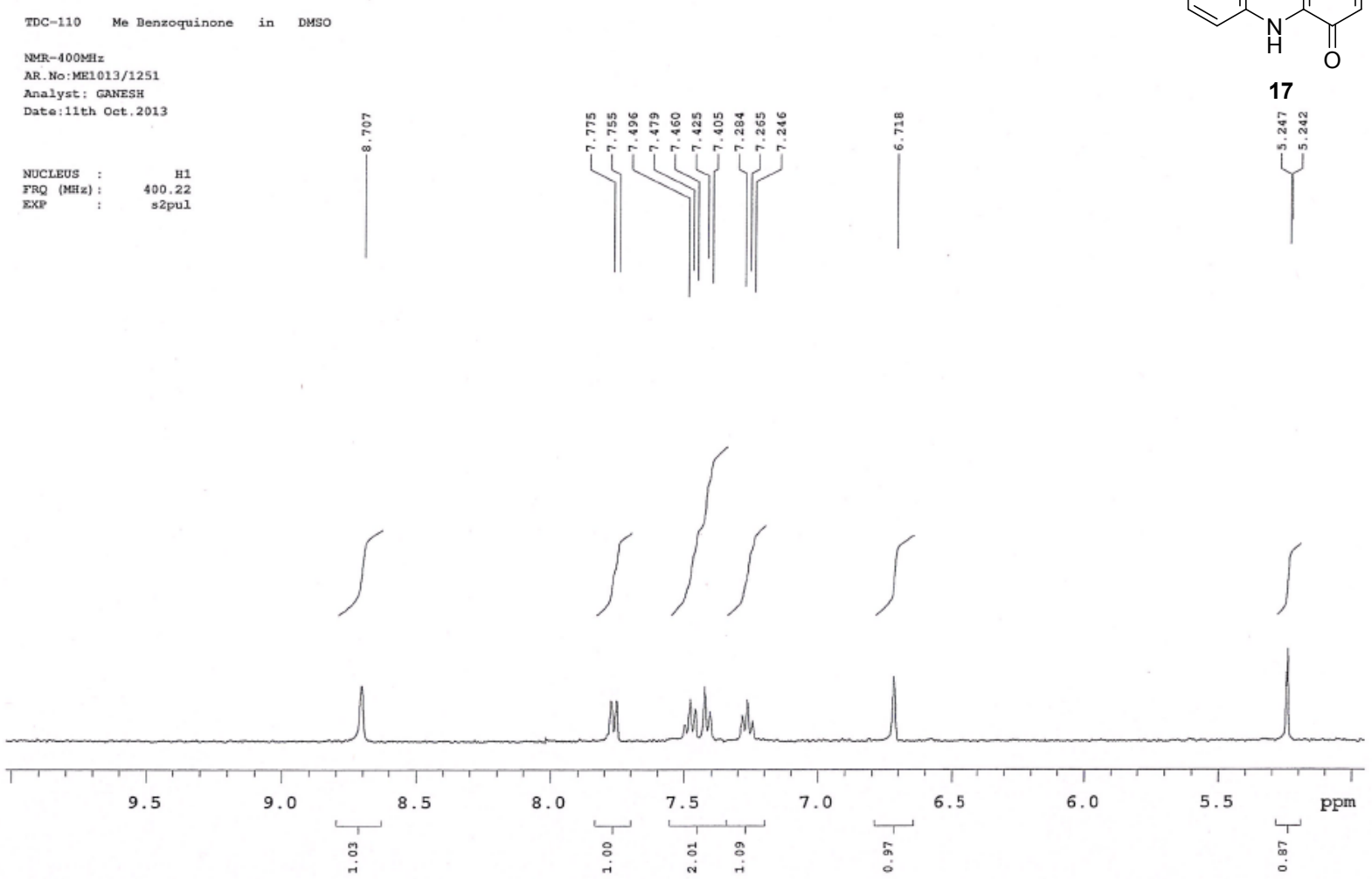
^{1}H NMR of 2-((2-bromophenyl) amino)-5-methylcyclohexa-2,5-diene-1,4-dione (17)



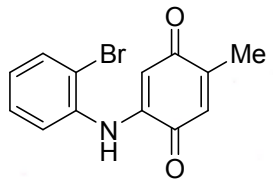
Expansion ^{1}H NMR of 2-((2-bromophenyl) amino)-5-methylcyclohexa-2,5-diene-1,4-dione (17)



17

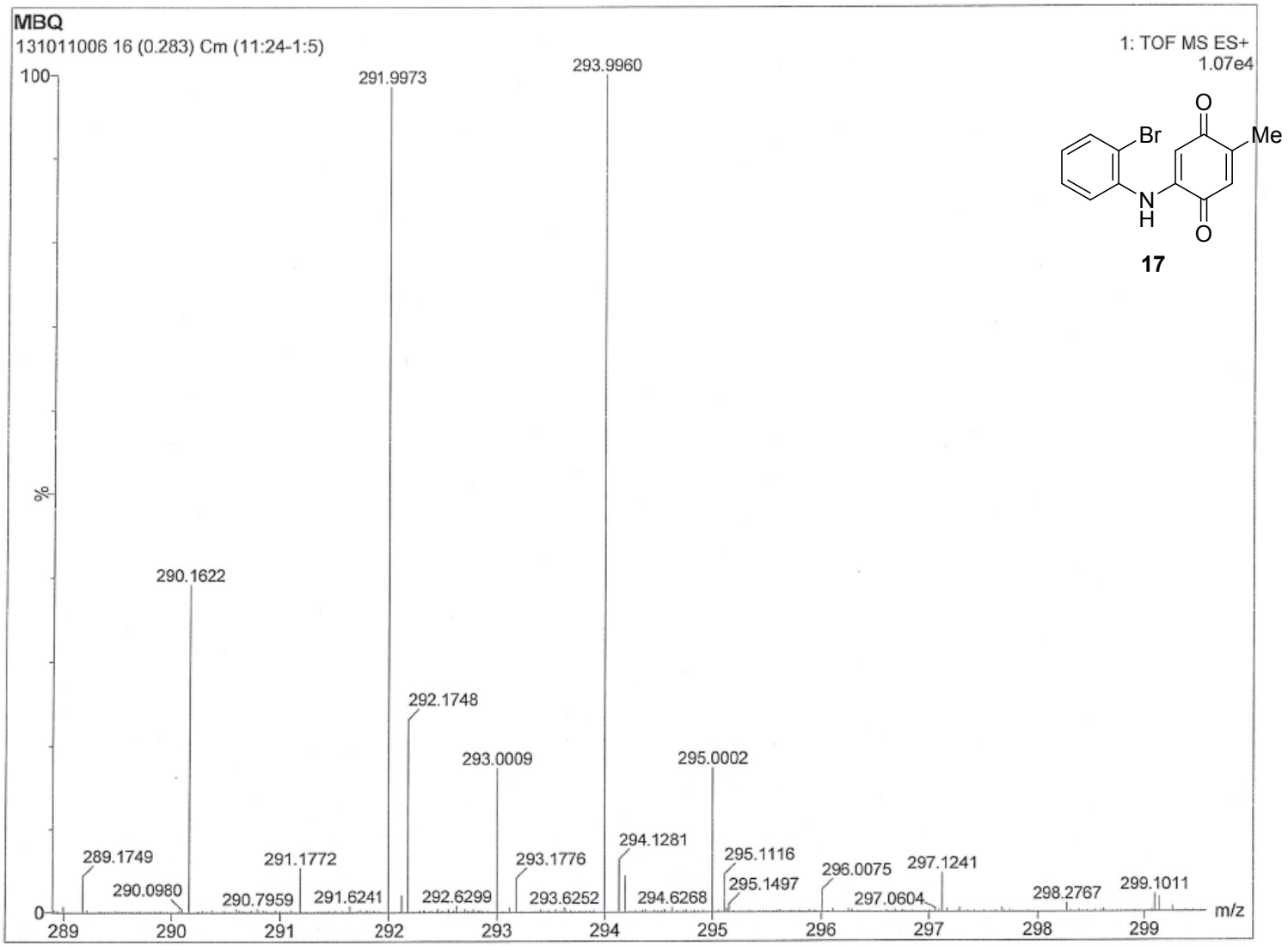


^{13}C NMR of 2-((2-bromophenyl) amino)-5-methylcyclohexa-2,5-diene-1,4-dione (17)

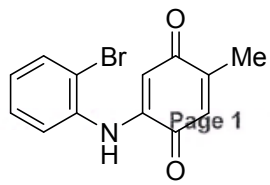


17

Mass Spectrum of 2-((2-bromophenyl) amino)-5-methylcyclohexa-2,5-diene-1,4-dione (17)



HRMS of 2-((2-bromophenyl) amino)-5-methylcyclohexa-2,5-diene-1,4-dione (17)



17

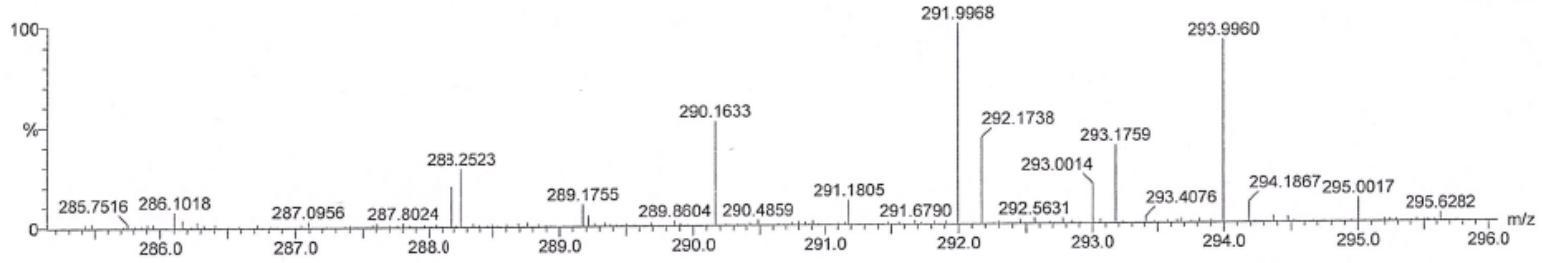
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -5.0, max = 80.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 4

Monoisotopic Mass, Even Electron Ions
22 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-15 H: 0-13 N: 0-2 O: 0-3 Br: 0-1

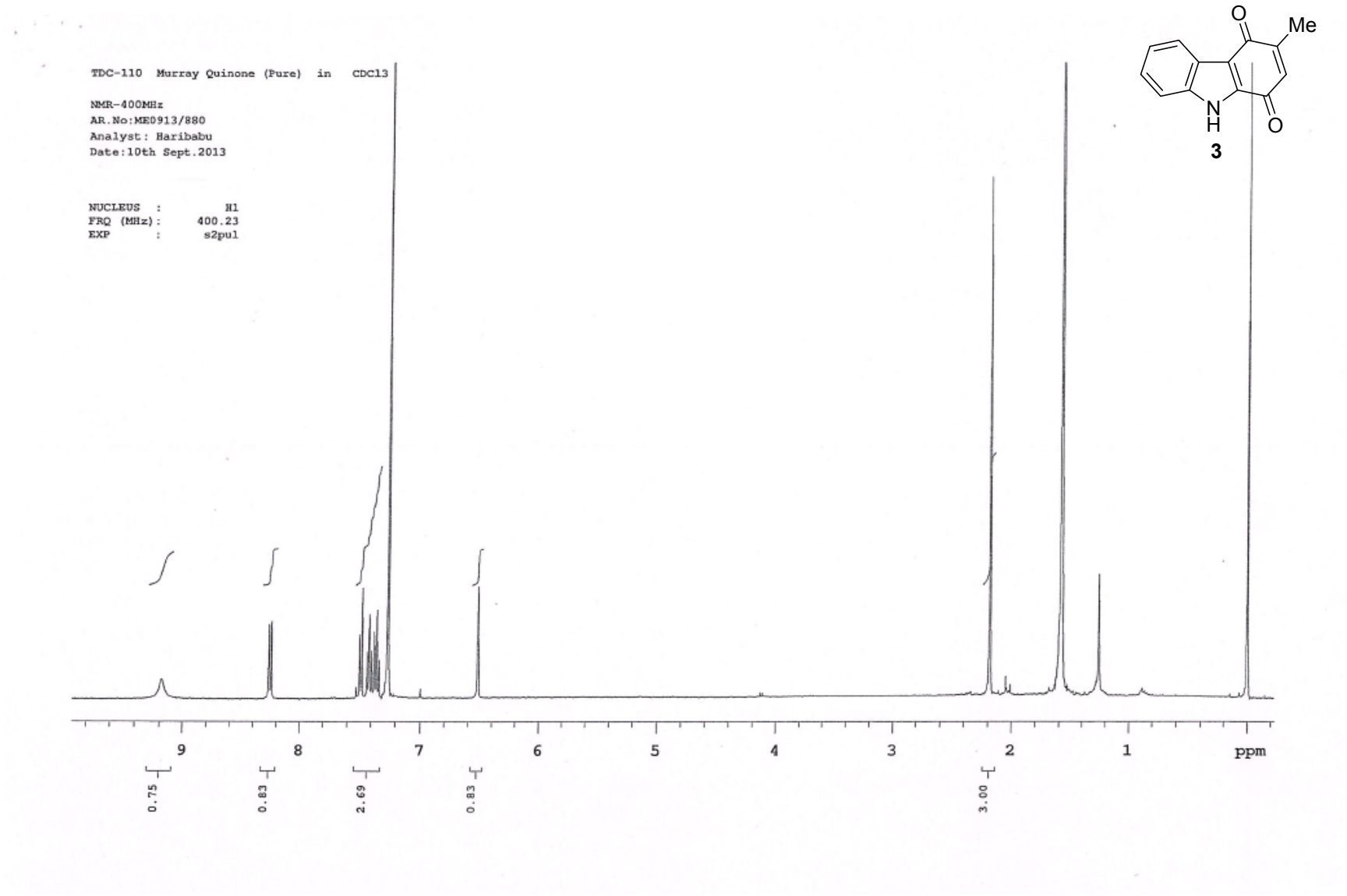
MBQ
131011006 16 (0.283) Cm (15:24-5:11)

1: TOF MS ES+
1.01e+003

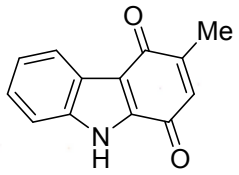


Minimum:				-5.0		
Maximum:		5.0	5.0	80.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
291.9968	291.9973	-0.5	-1.7	8.5	10.6	C13 H11 N O2 Br

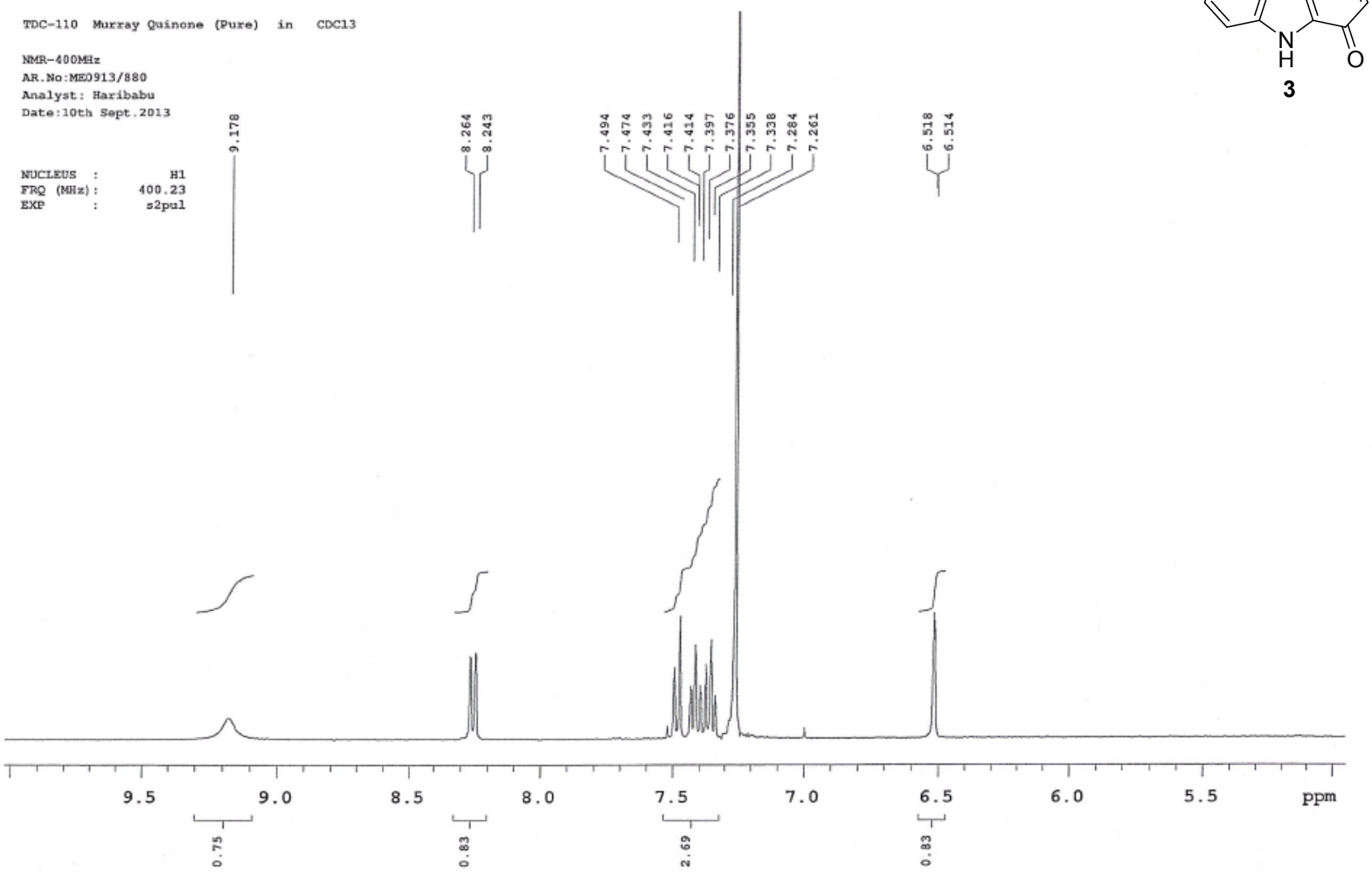
^{1}H NMR of Murrayaquinone A (3)



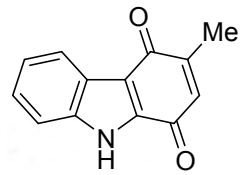
Expansion ^{1}H NMR of Murrayaquinone A (3)



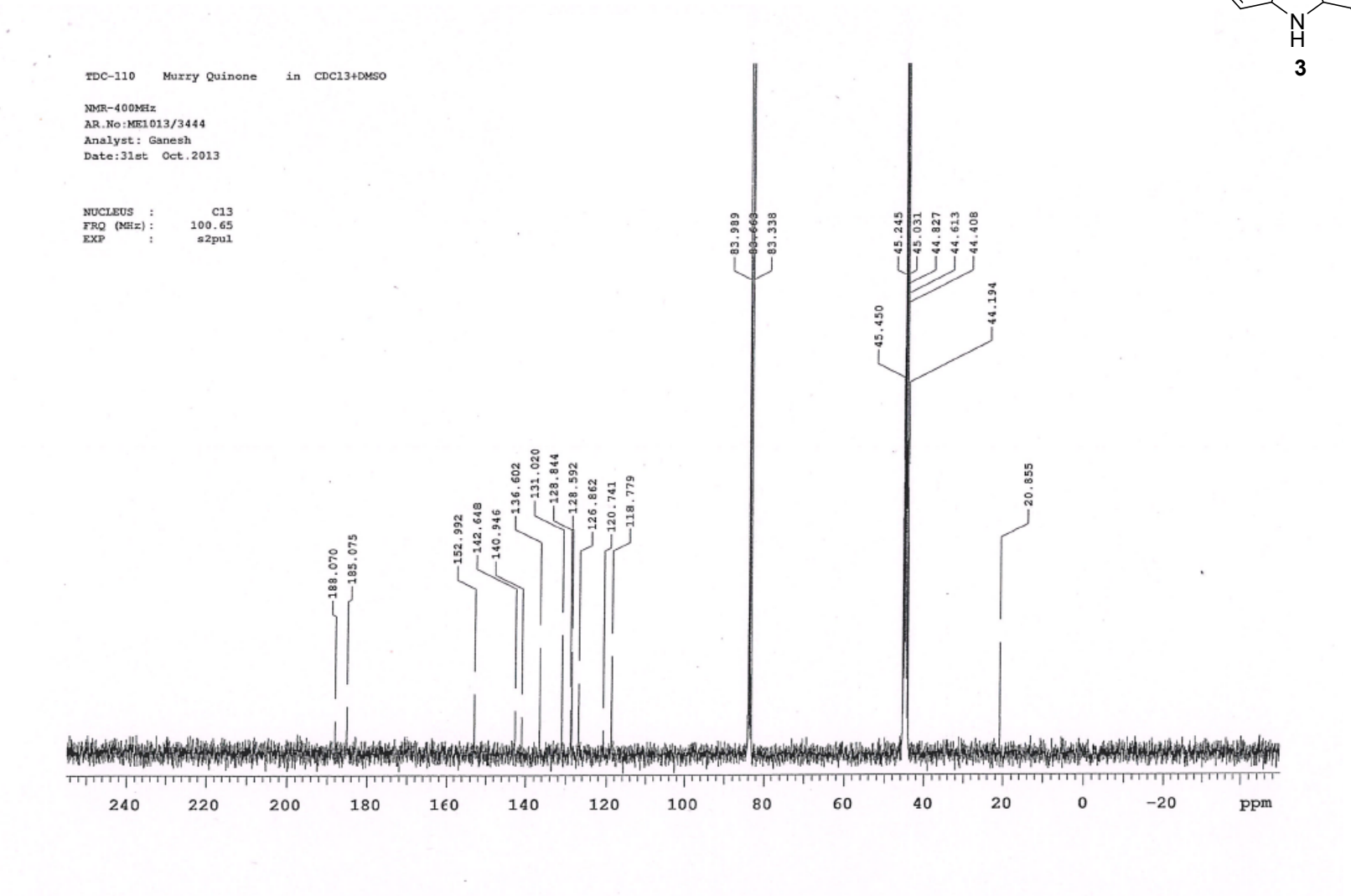
3



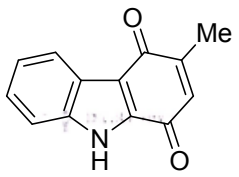
^{13}C NMR of Murrayaquinone A (3)



3



Mass Spectrum of Murrayaquinone A (3)



3

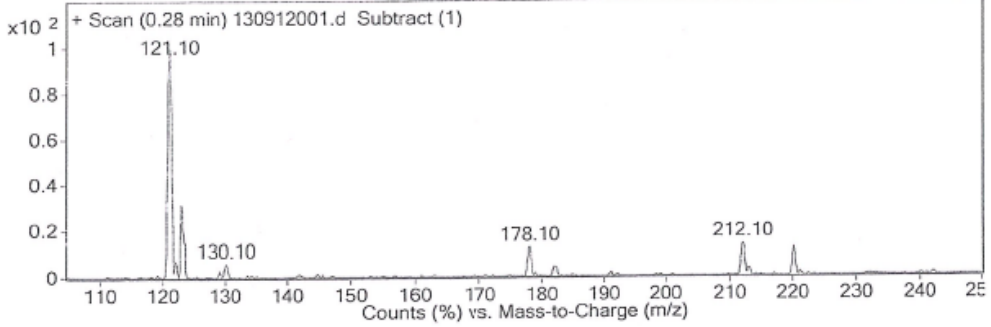
CPS,MIYAPUR

LCMS Analysis Report

Data Filename	130912001.d	**Sample Name**	Murrayquinone
Sample Type	Sample	**Position**	Vial 74
Instrument Name	Instrument 1	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	Esi



--- End Of Report ---