

Supplemental material for:

**Expeditious Route to Enantioenriched Tetracyclic Core of
Ergot Alkaloids via Organocatalytic Aldol Reaction**

Subhajit Bhunia,[§] Saikat Chauduri,[§] Subhadip De, K. Naresh Babu, and Alakesh Bisai*

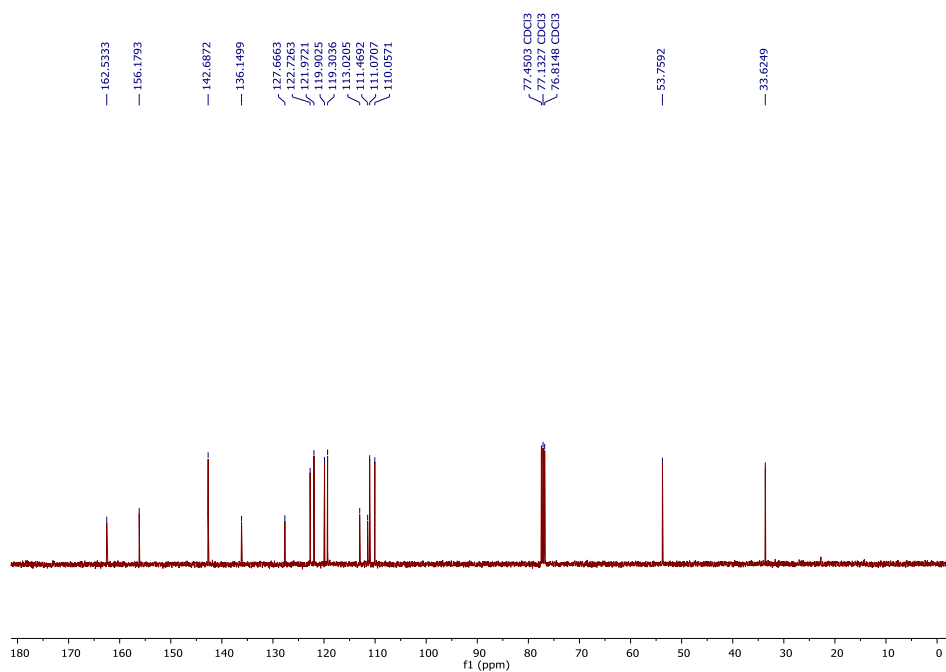
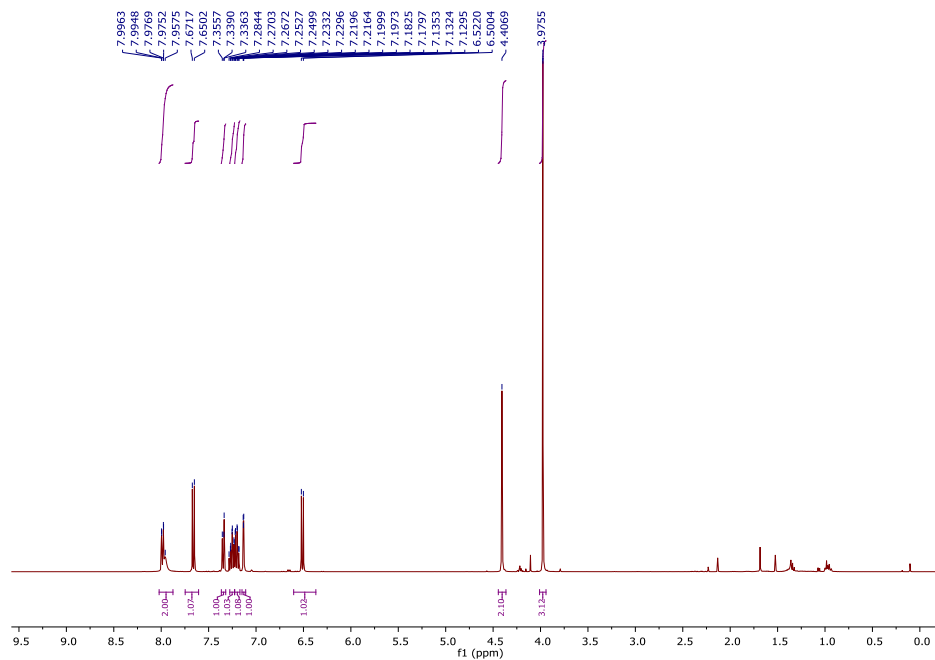
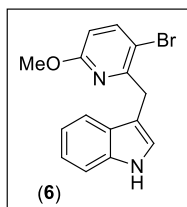
Department of Chemistry, Indian Institute of Science Education and Research Bhopal, Bhopal

Bypass Road, Bhauri, Bhopal - 462 066, Madhya Pradesh, INDIA.

E-Mail: alakesh@iiserb.ac.in

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Spectral Graphics and HPLC Traces

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\NOV-2015\18-NOV-2015\Dr.A.Bisai-SB4-022_1-C,2_01_4284.d
Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-SB4-022
Comment

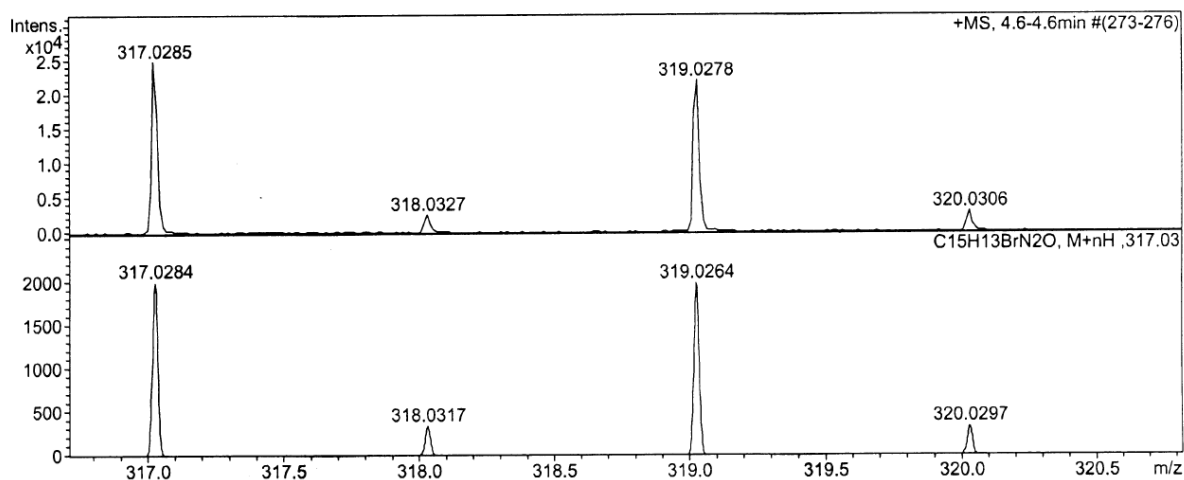
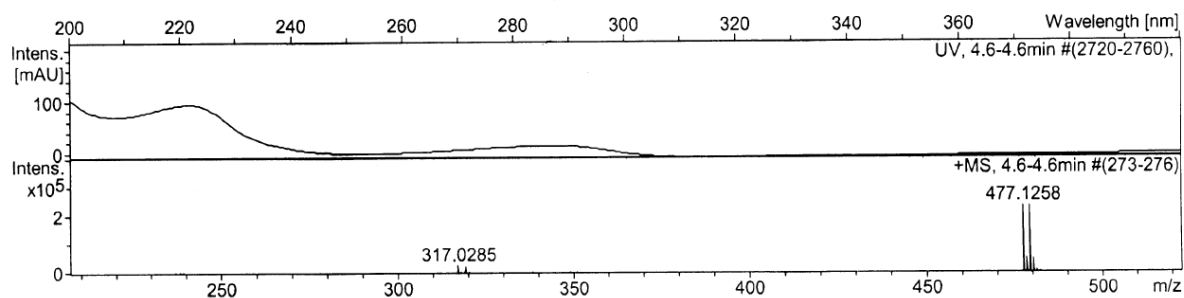
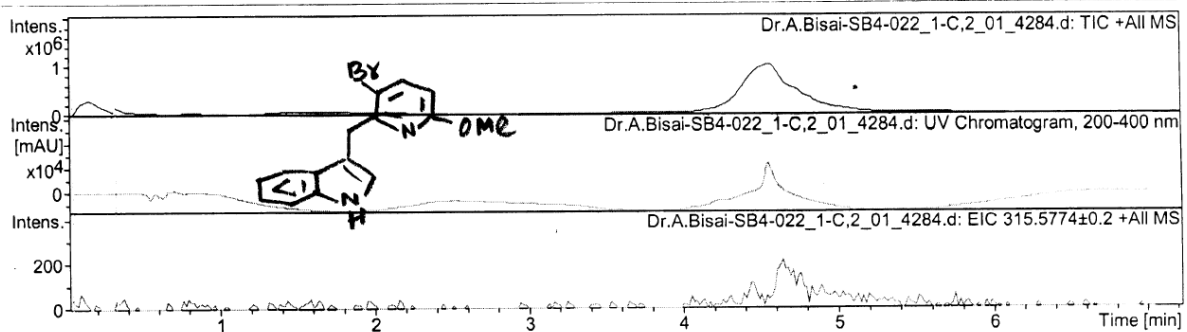
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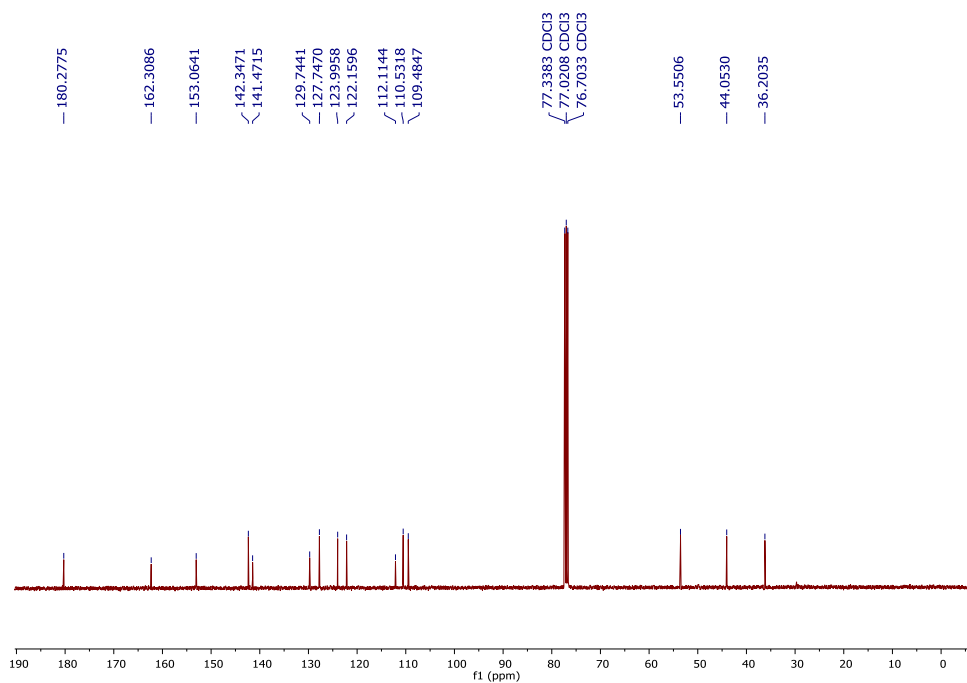
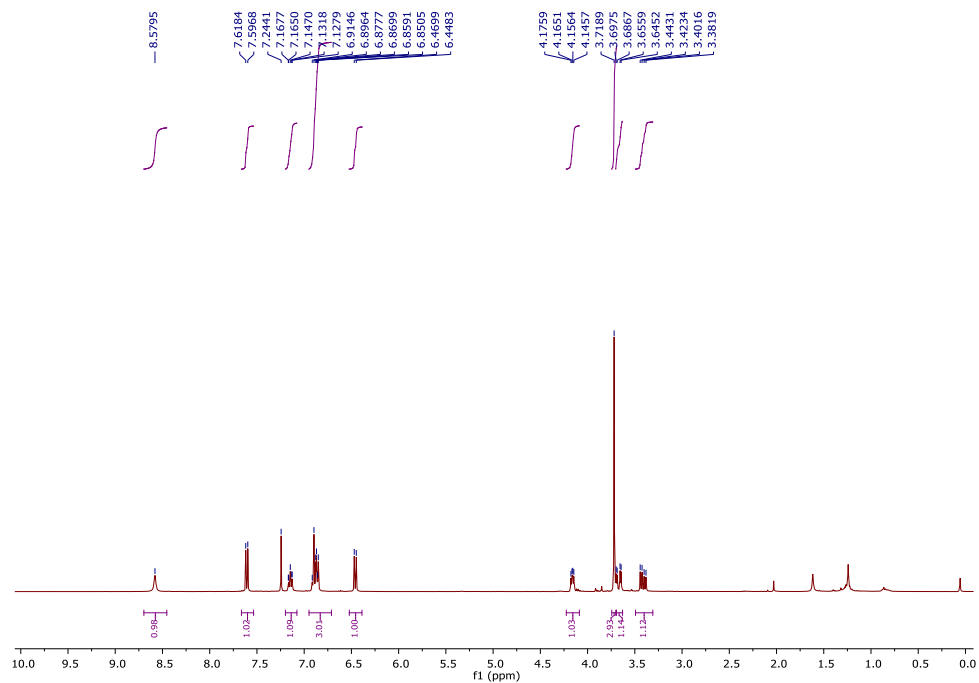
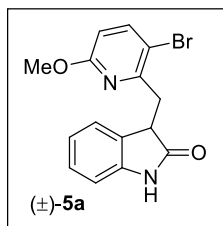
Operator RUCHI

Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Display Report

Analysis Info

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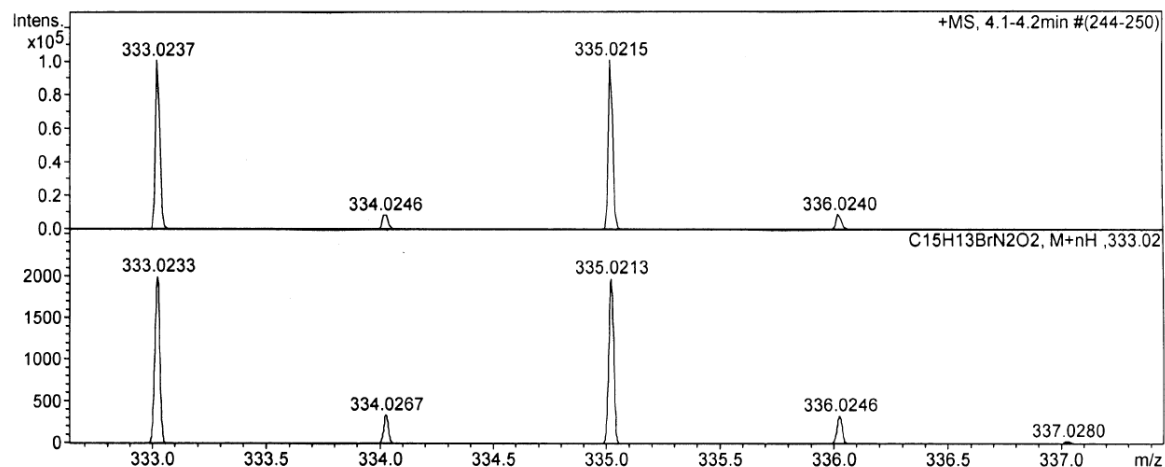
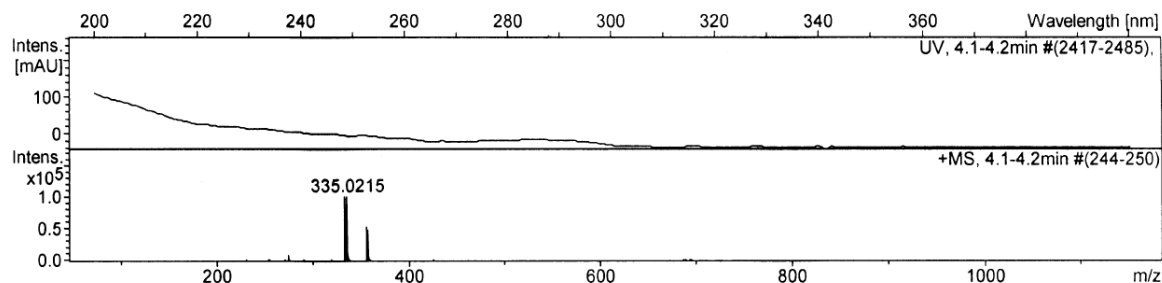
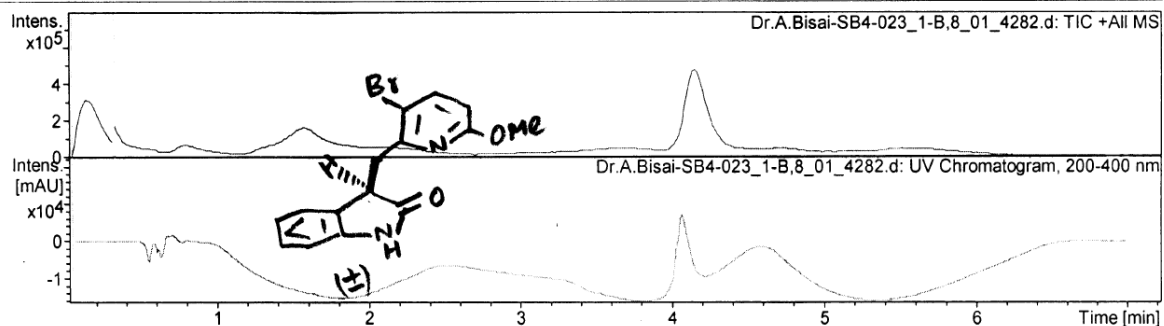
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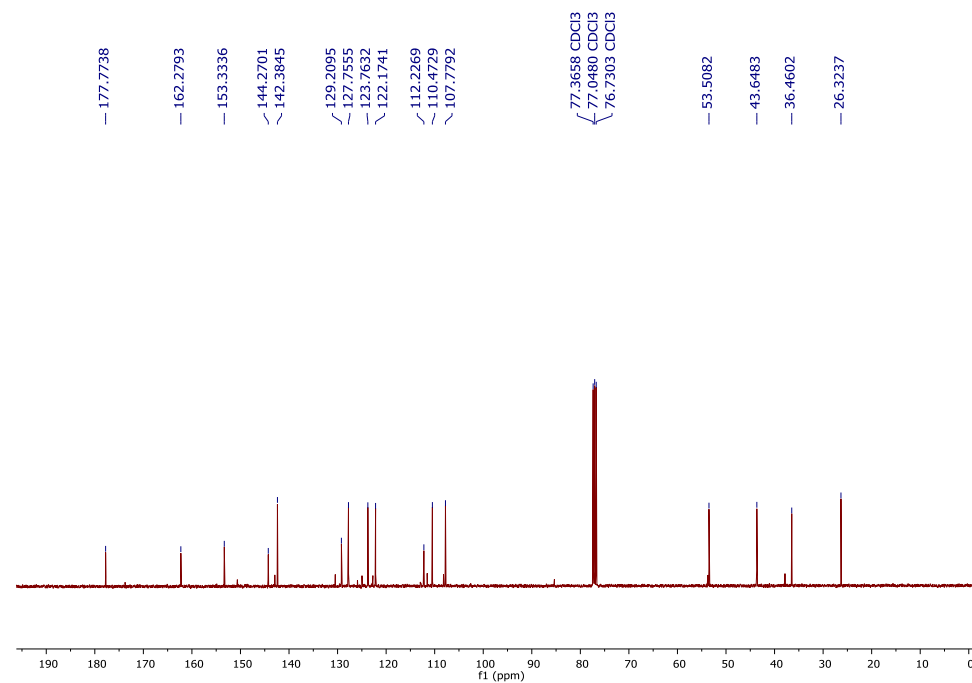
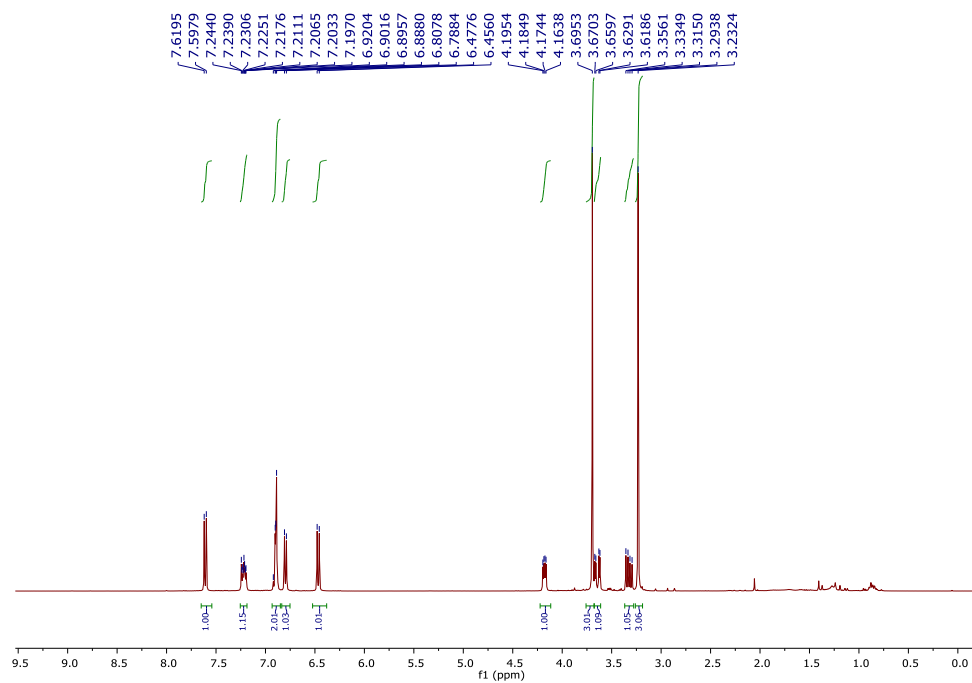
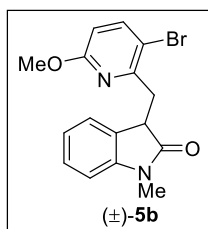
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Instrument micrOTOF-Q II 10330

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Display Report

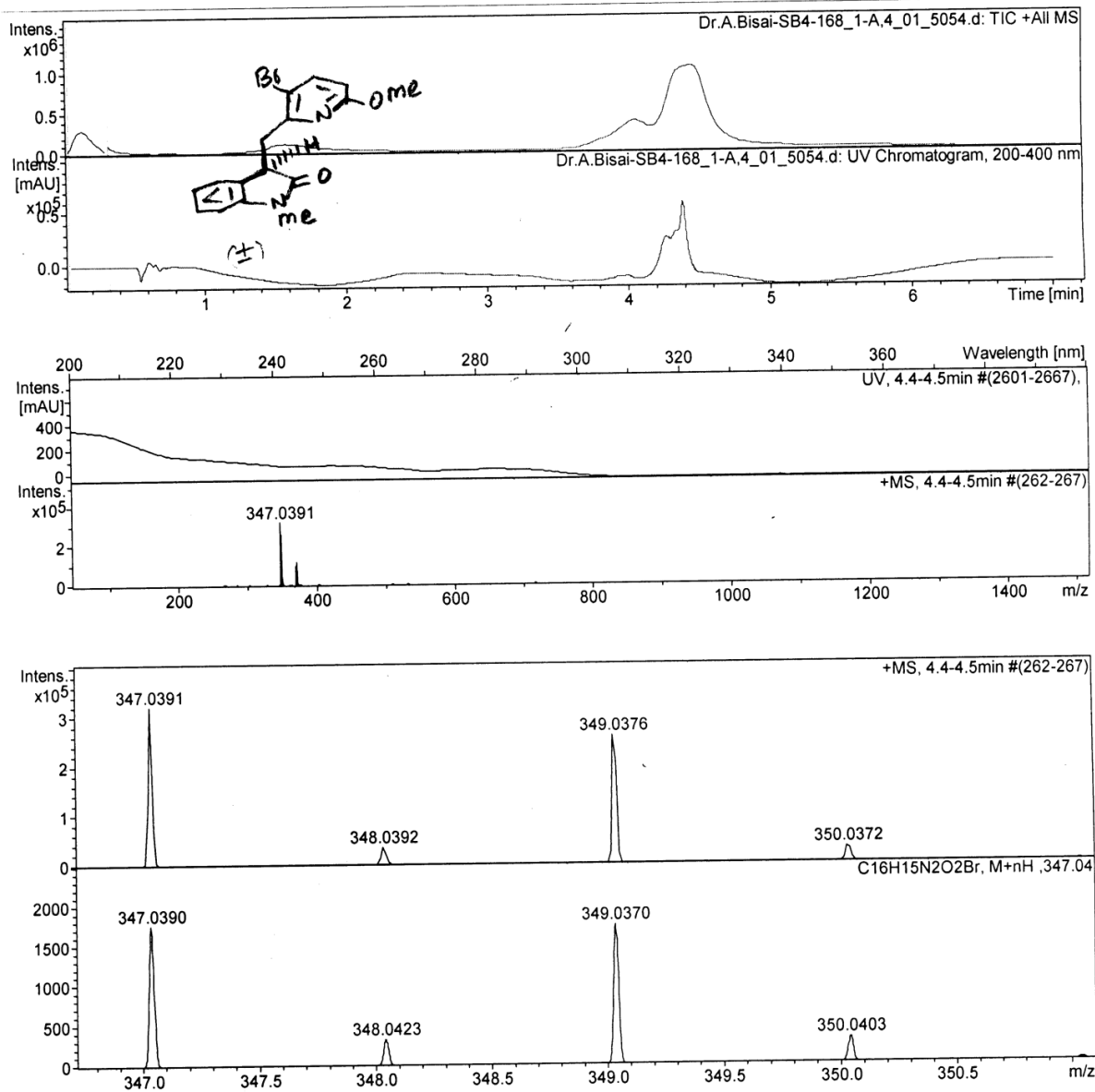
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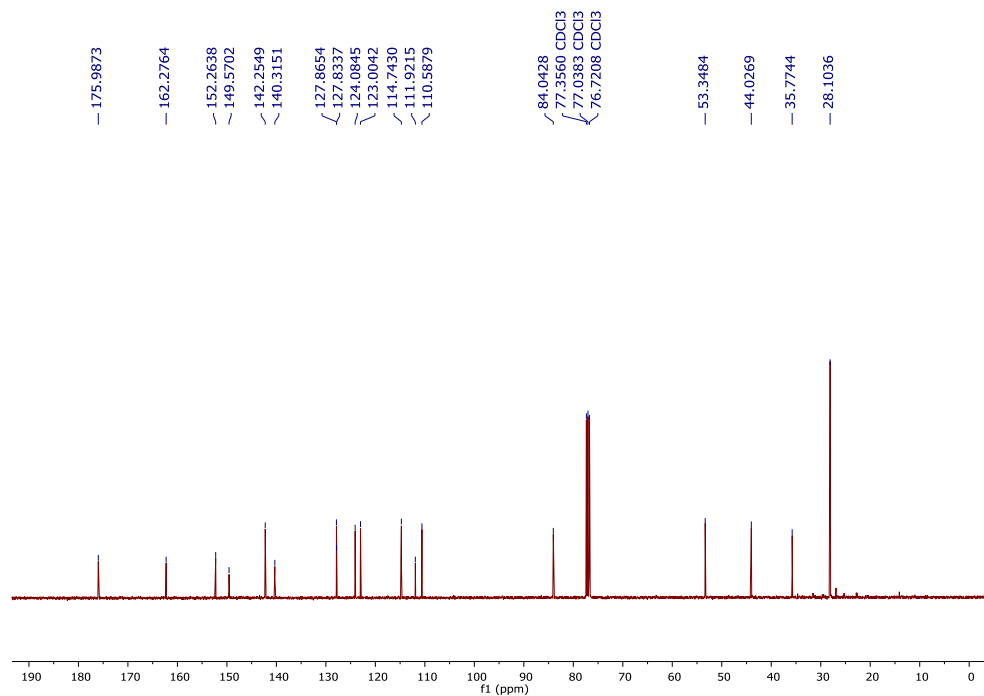
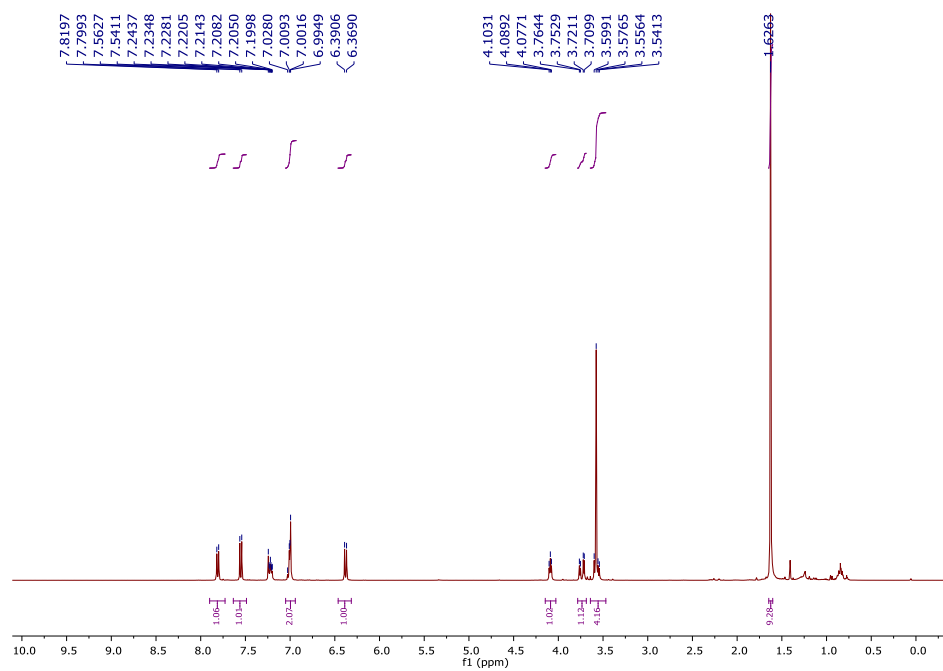
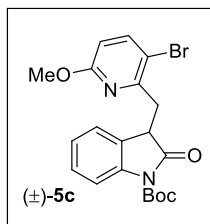
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Scanned copy of mass spectrum of (±)-5b



Display Report

Analysis Info

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Operator RUCHI

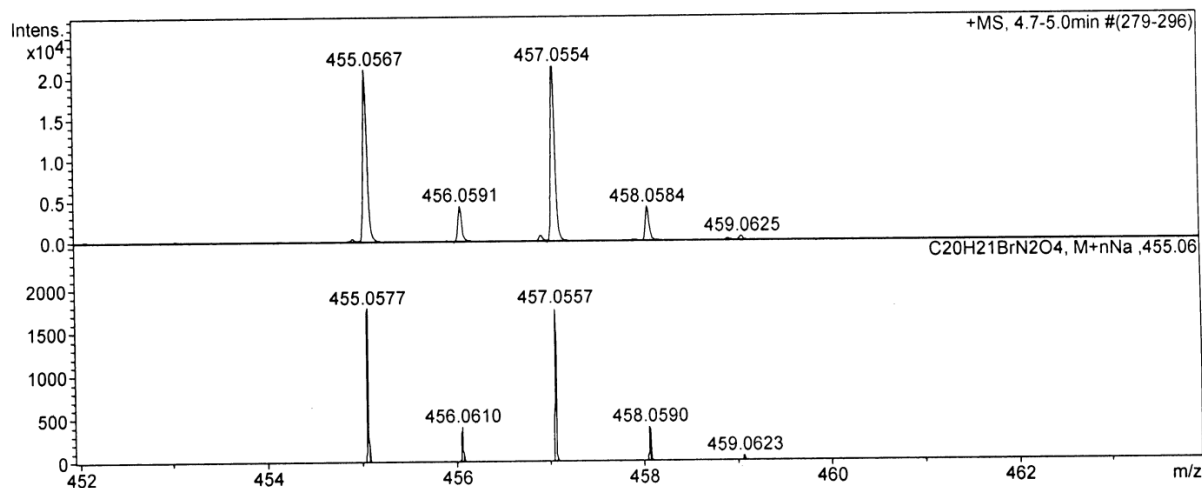
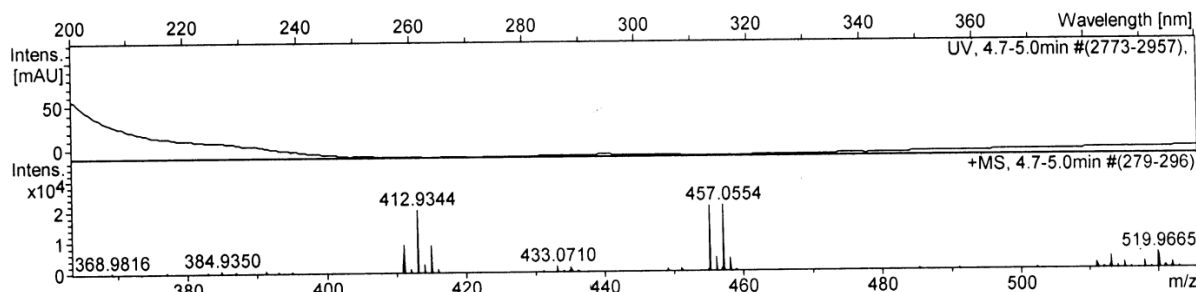
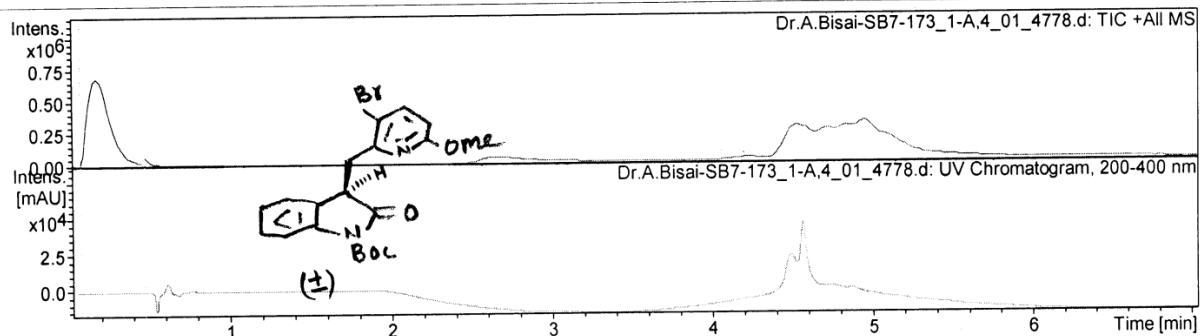
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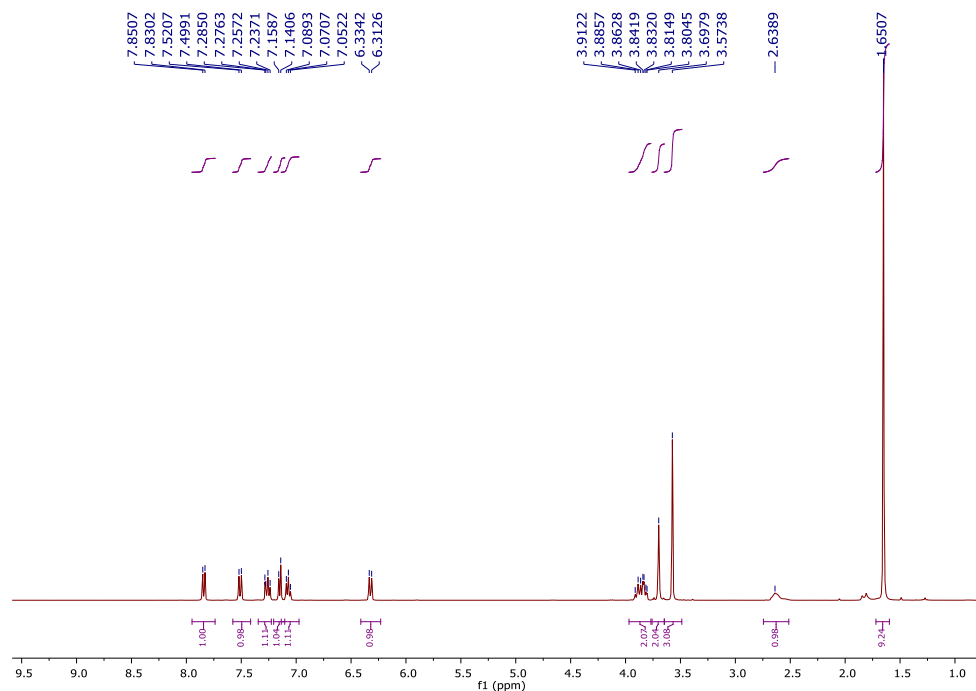
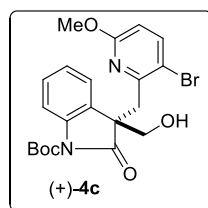
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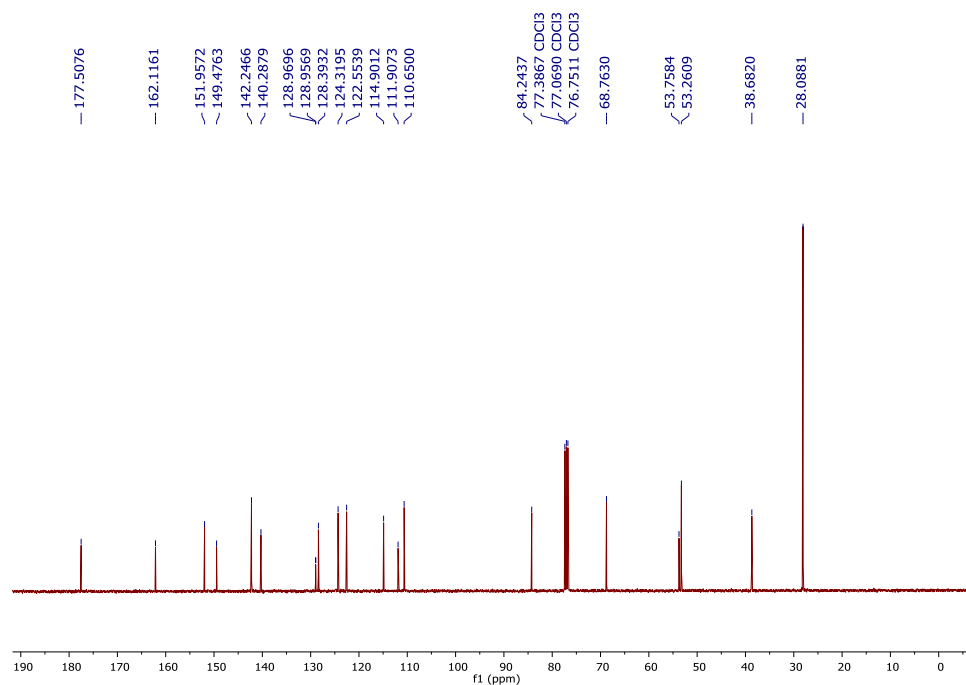
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Scanned copy of mass spectrum of (±)-5c



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^{13}C NMR (100 MHz, CDCl_3) of (+)-**4c**

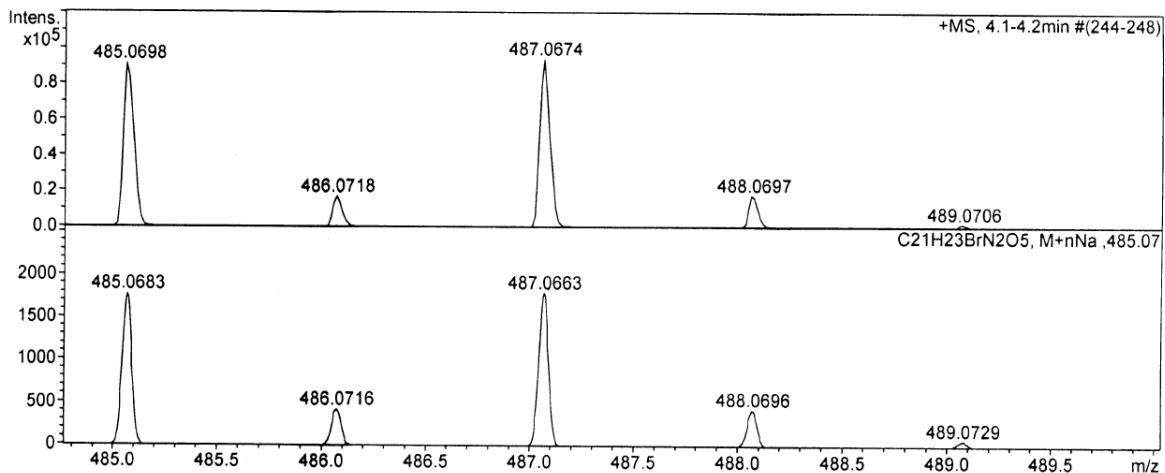
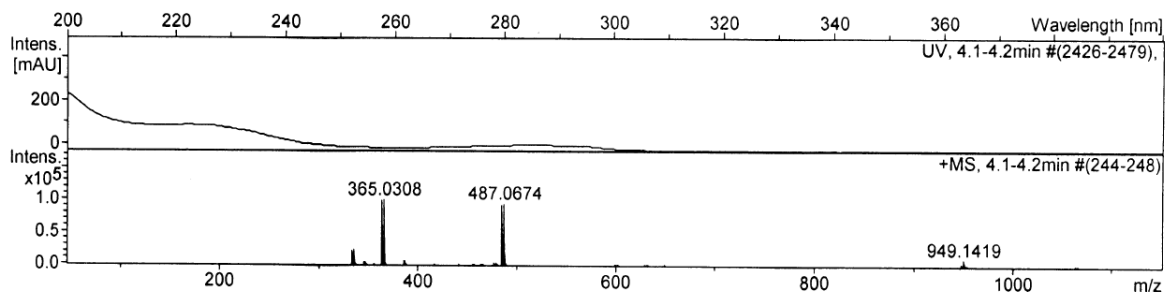
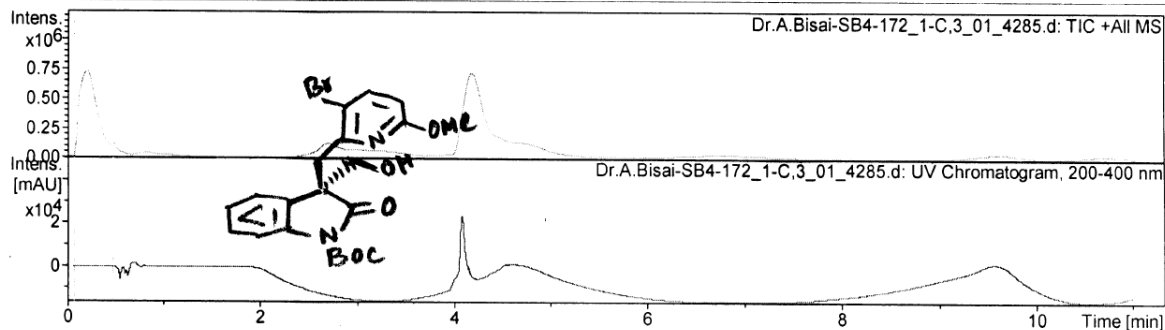
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Sample Name	Dr.A.Bisai-SB4-172	Instrument	microTOF-Q II 10330
Comment			

Acquisition Parameter

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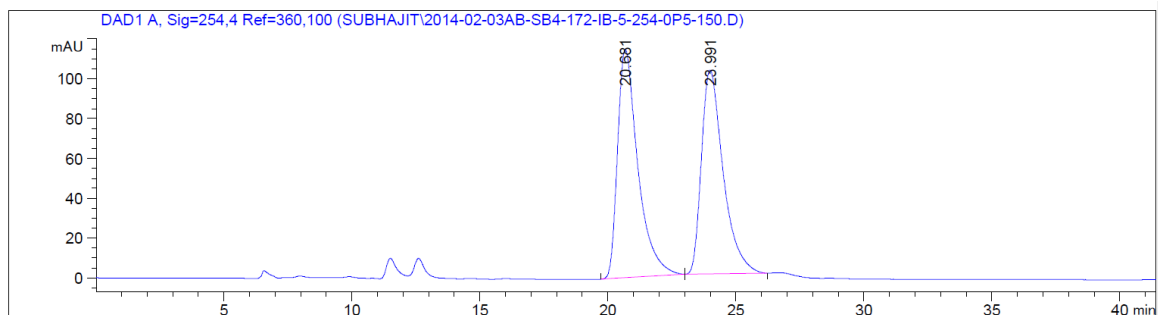


Scanned copy of mass spectrum of (+)-4c

HPLC traces of compound (+)-4c

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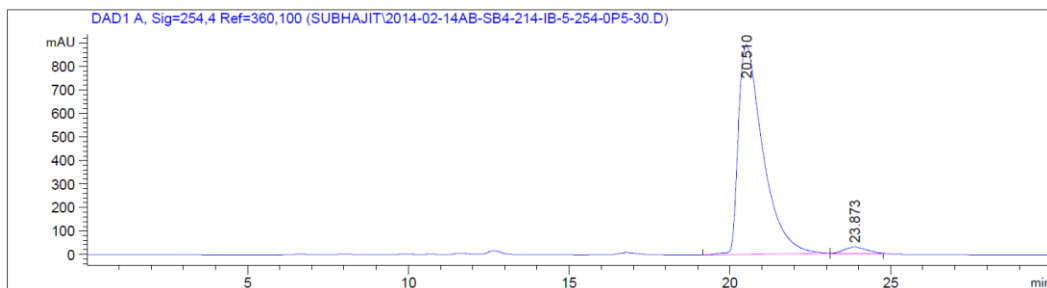


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Racemic

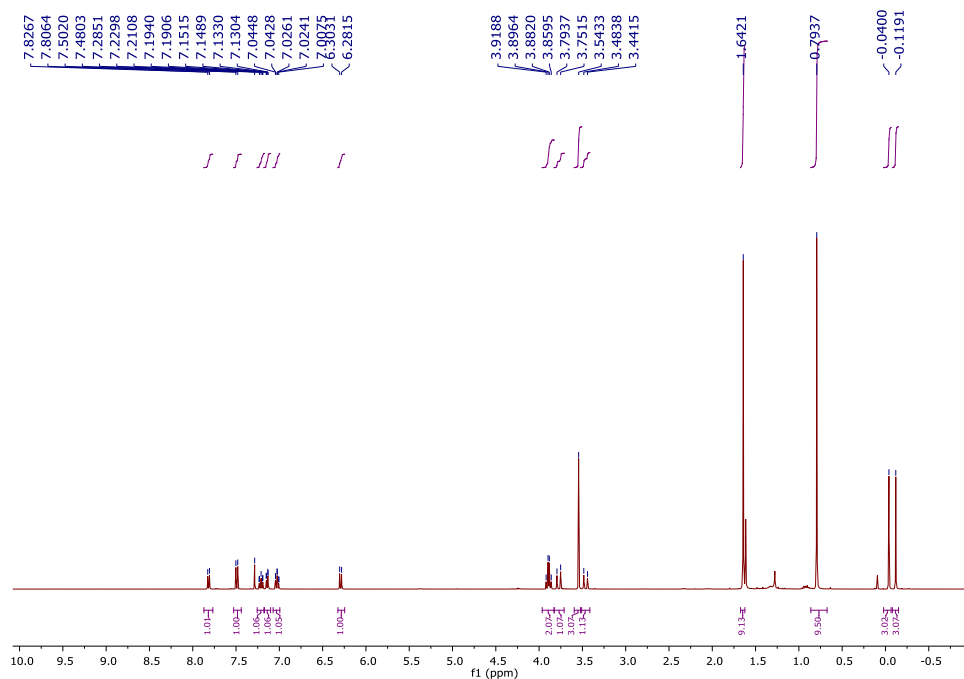
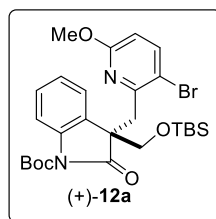
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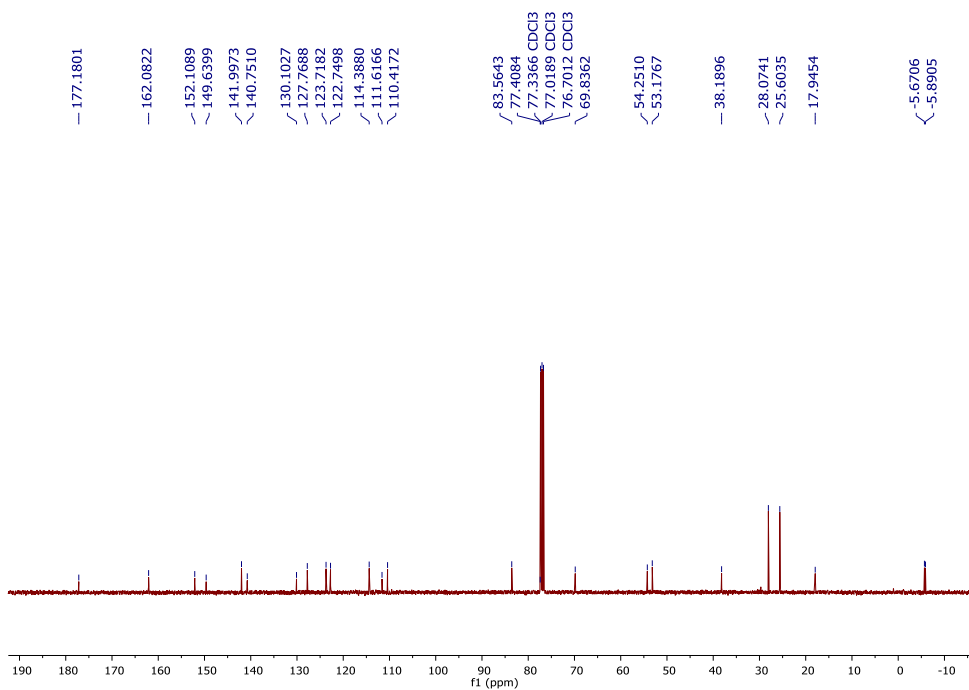


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2	23.873	BBA	0.7653	1222.36719	25.58911	2.5195

Enantiopure



^1H NMR (400 MHz, CDCl_3) of (+)-12a



^{13}C NMR (100 MHz, CDCl_3) of (+)-12a

Display Report

Analysis Info

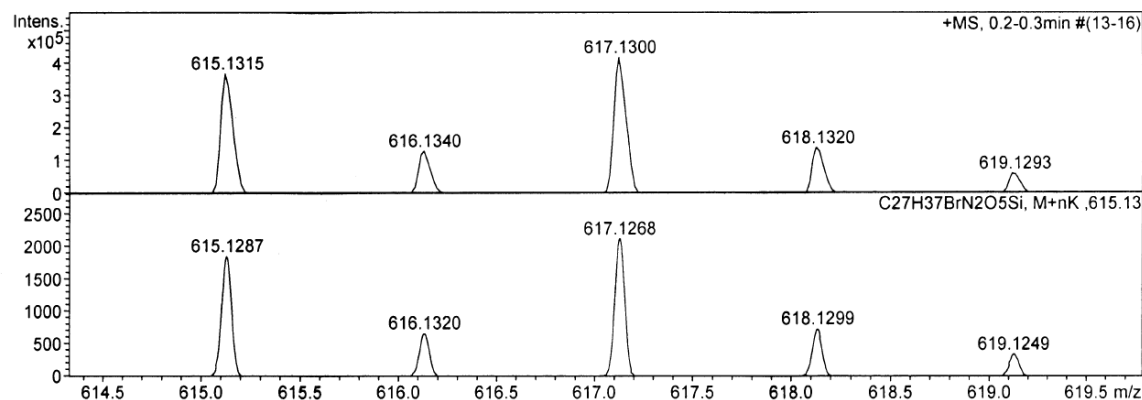
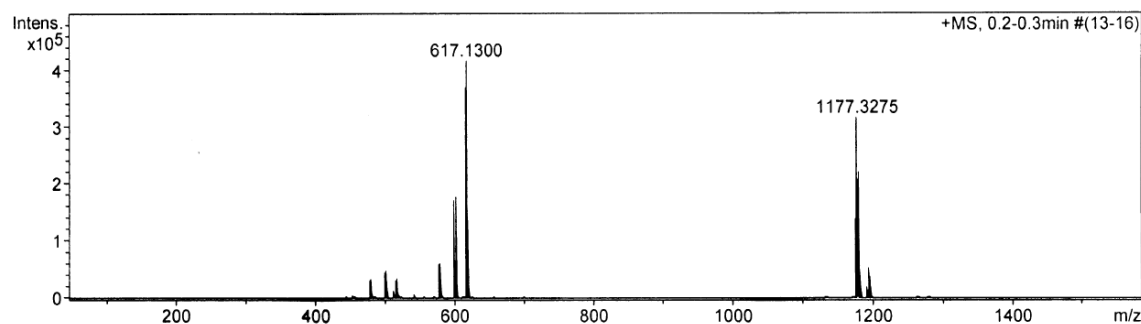
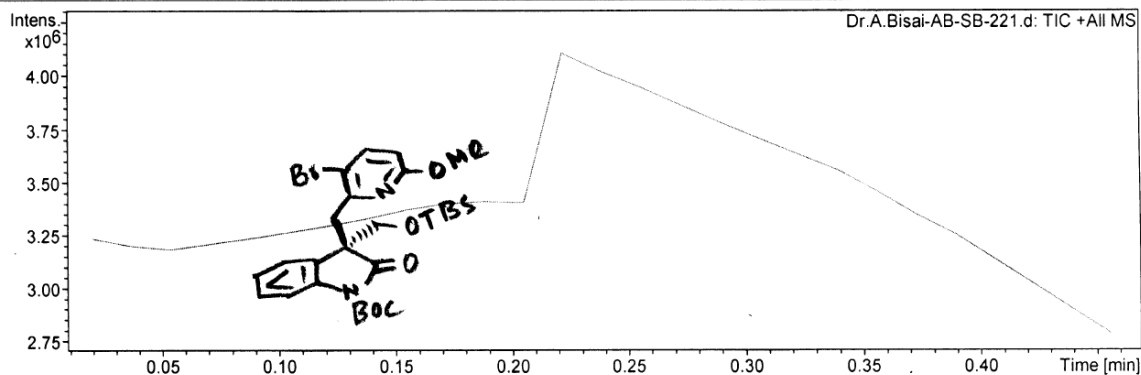
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Sample Name AB-SB-221
Comment

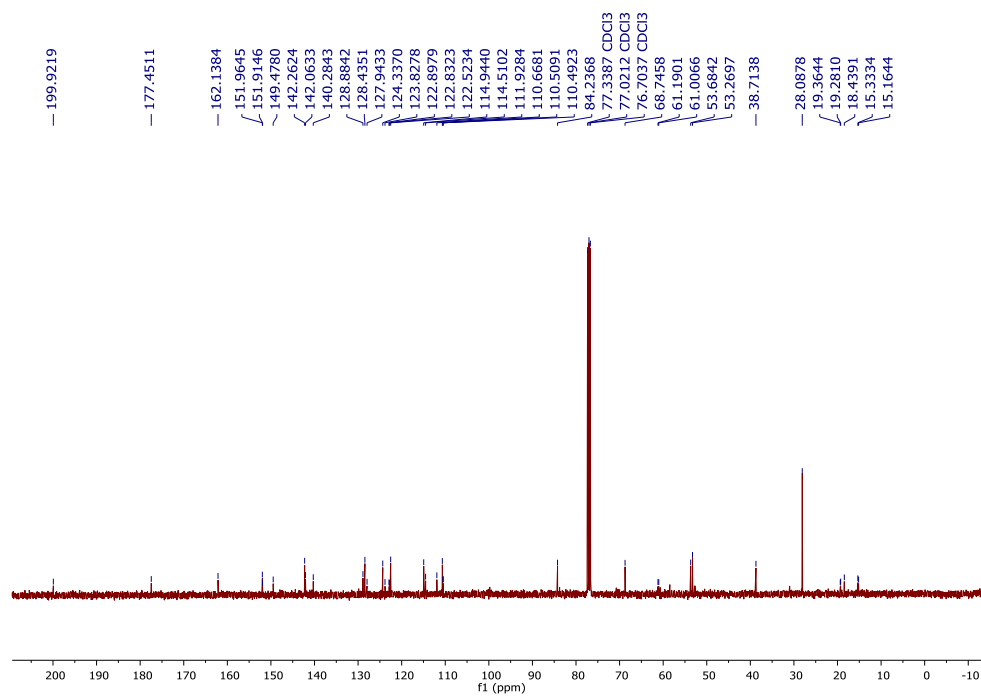
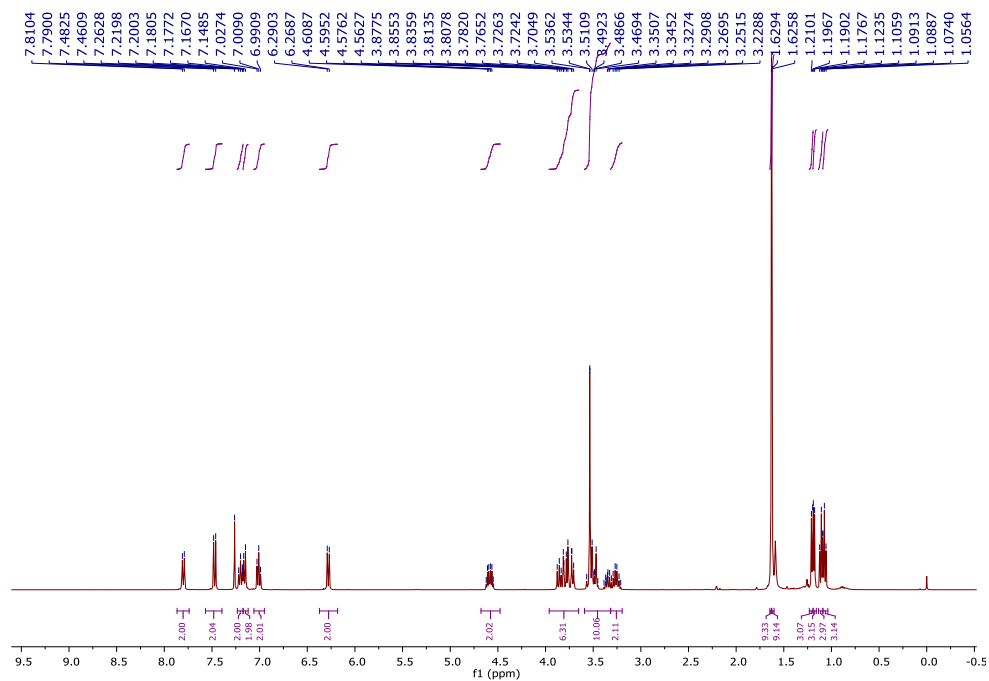
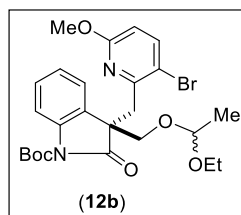
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Operator Ravindra
Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Display Report

Analysis Info

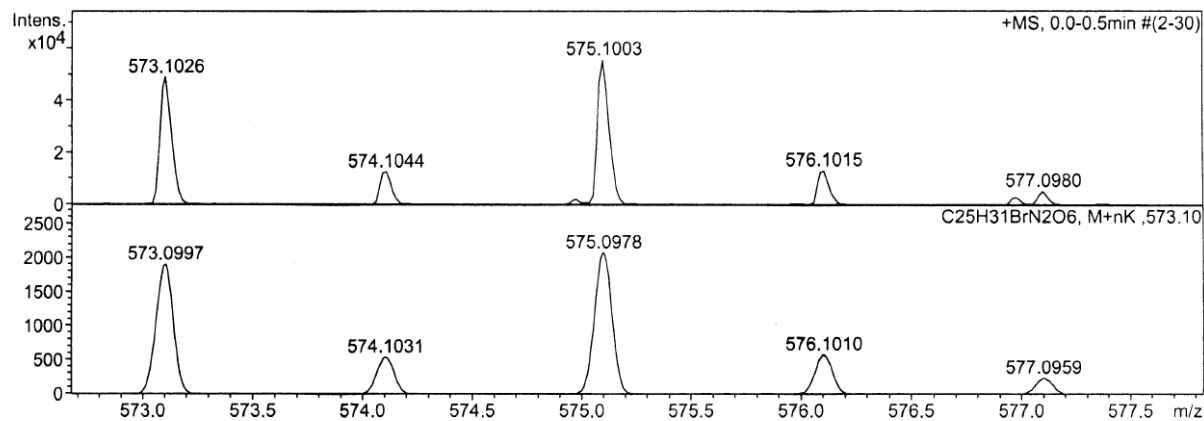
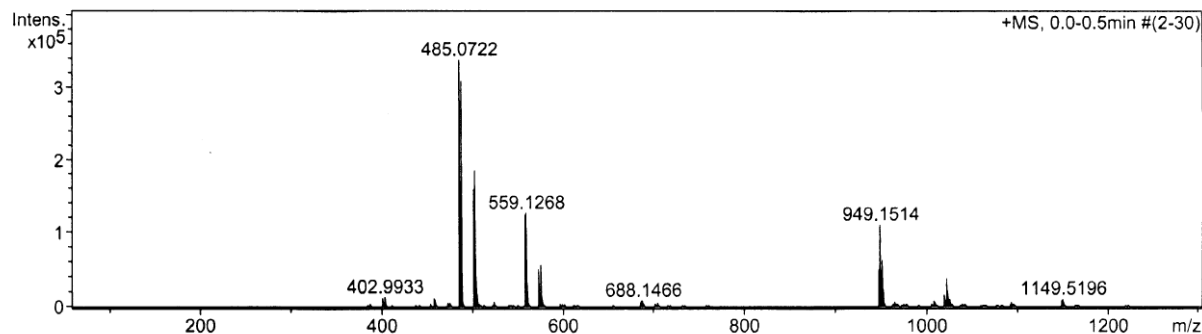
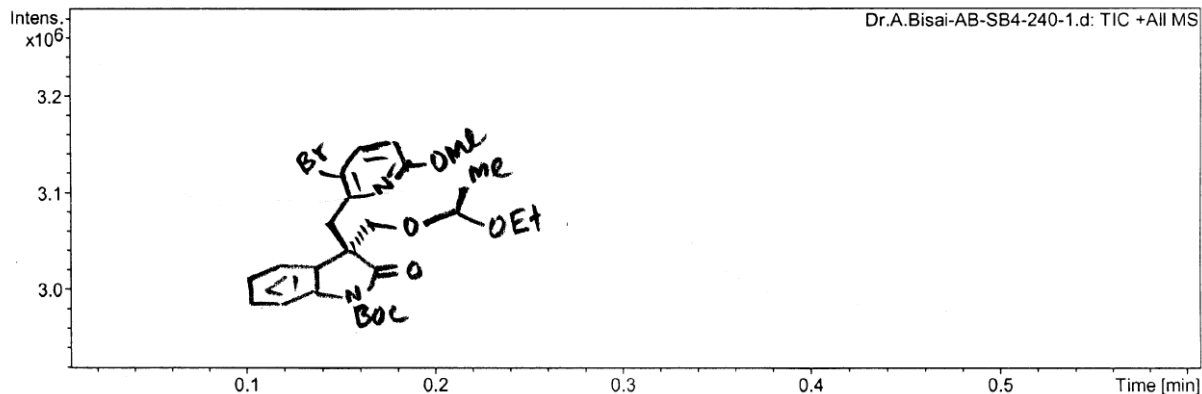
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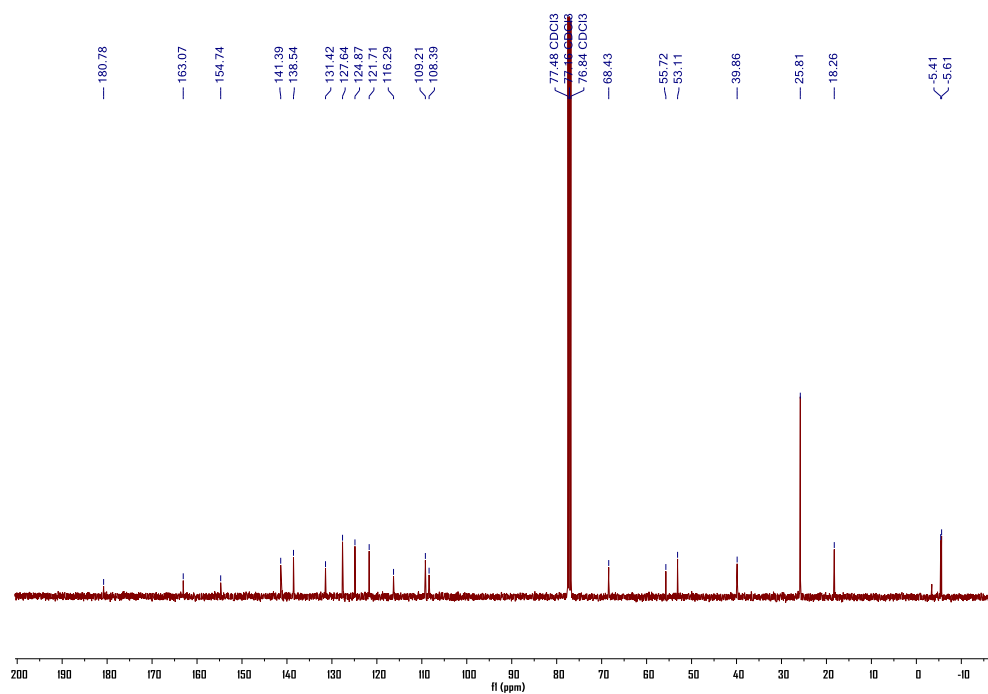
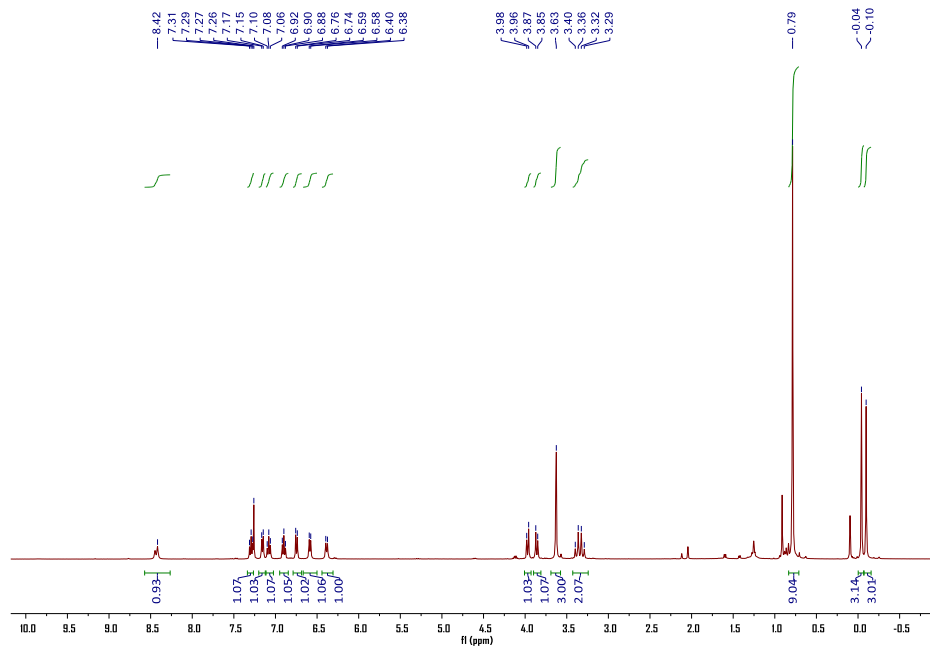
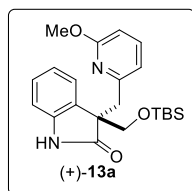
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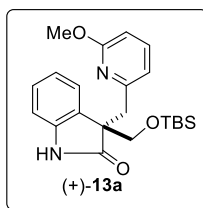
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Instrument micrOTOF-Q II 10330

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Mass Spectrum SmartFormula Report

Analysis Info

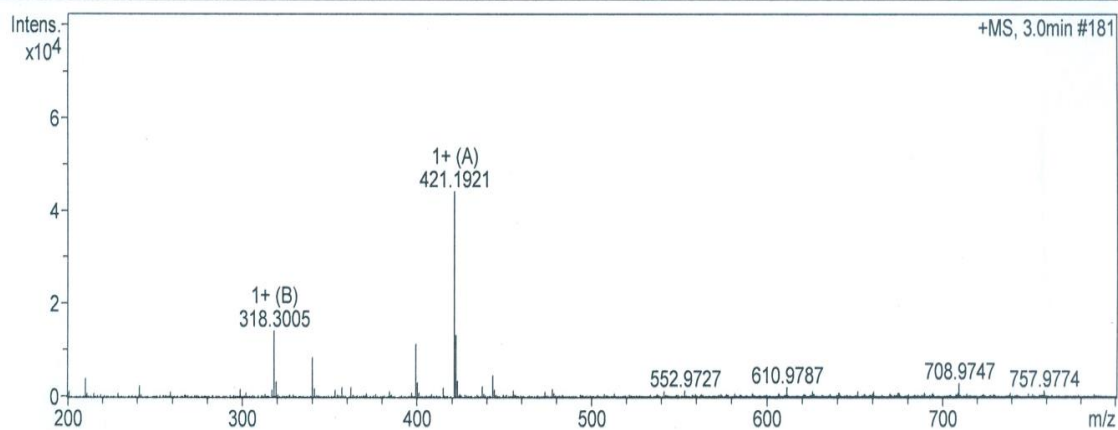
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Comment

Instrument / Ser# maXis 4G 21240

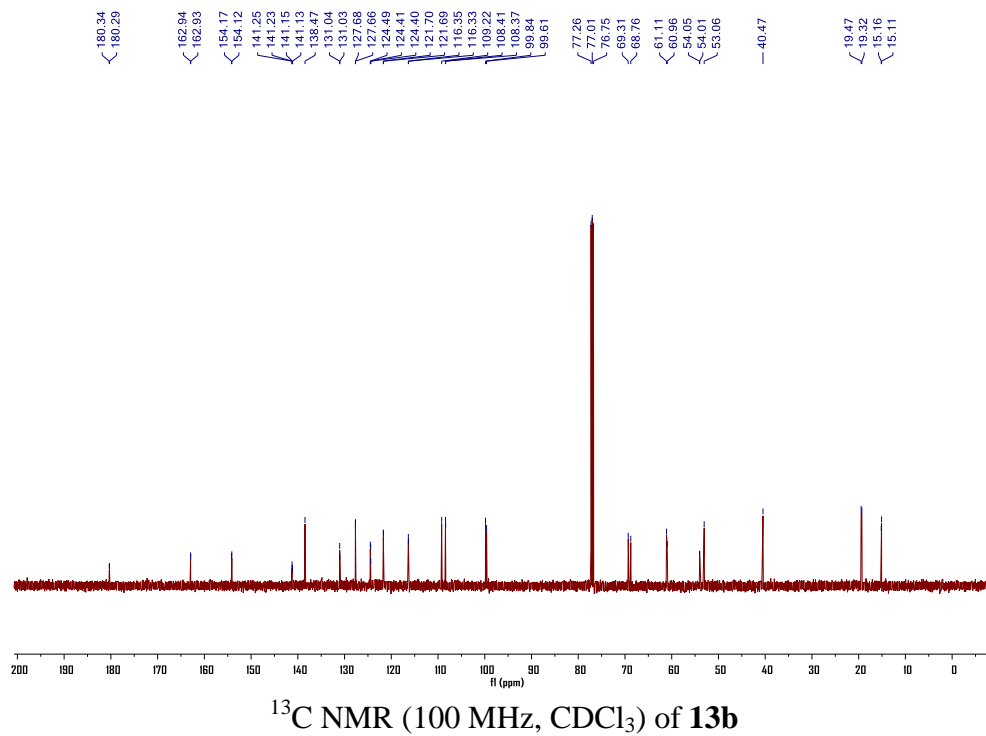
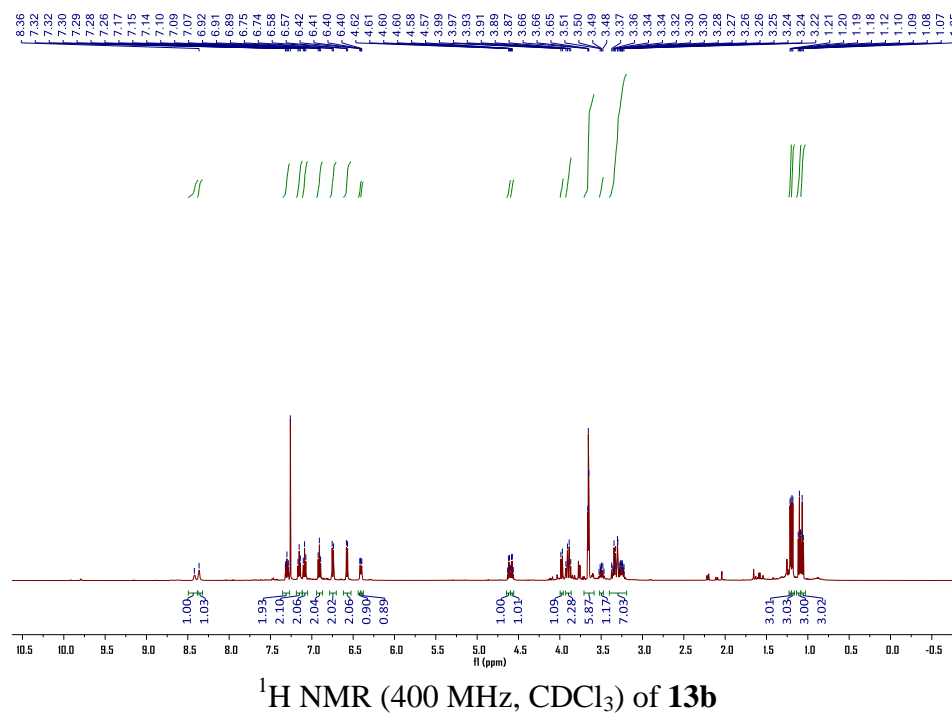
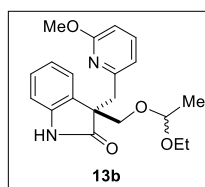
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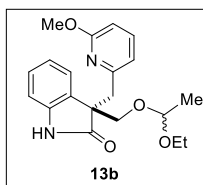
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Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSig ma	rdb	e ⁻ Conf	N-R ule
421.1921	1	C ₂₂ H ₃₀ N ₂ NaO ₃ Si	100.00	421.1918	-0.7	-1.3	2.9	9.5	even	ok

Mass spectrum of compound (+)-13a





Mass Spectrum SmartFormula Report

Analysis Info

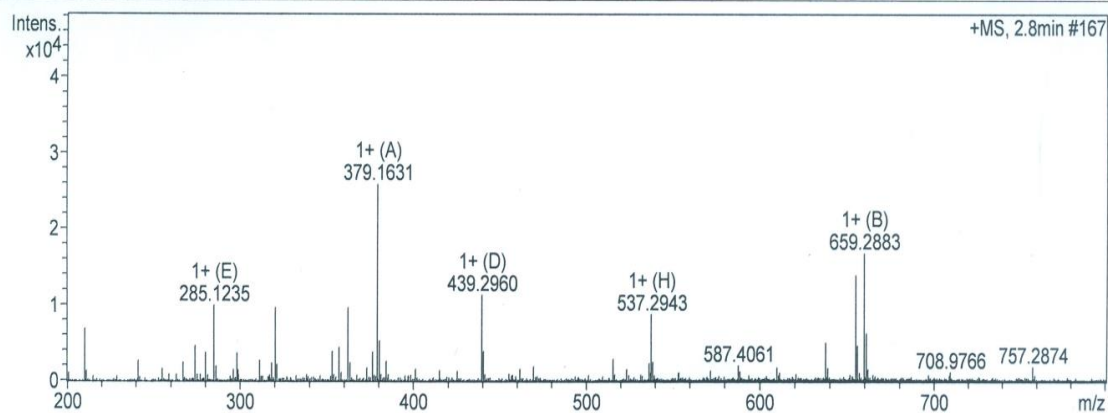
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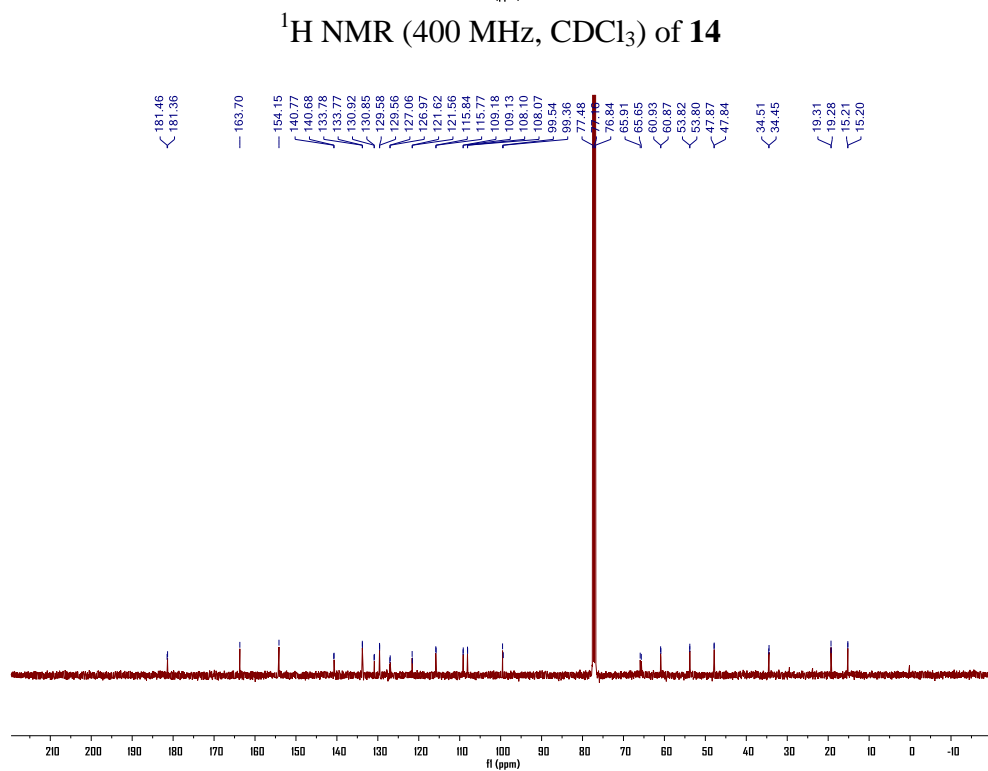
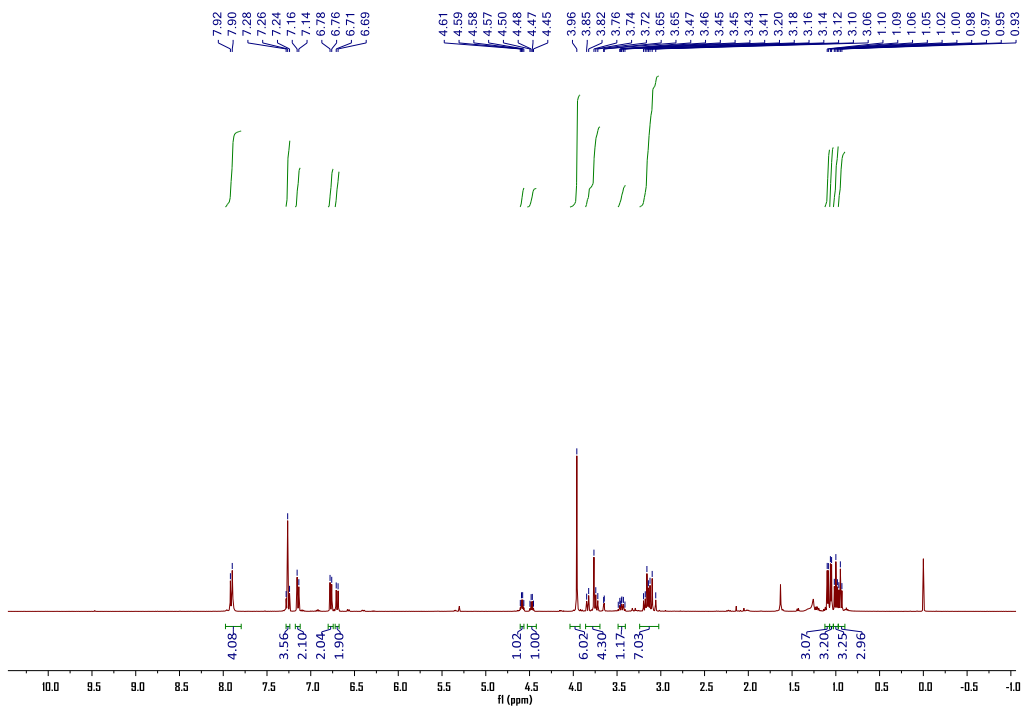
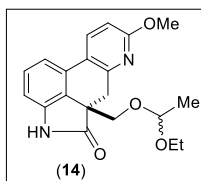
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Mass spectrum of compound **13b**



Display Report

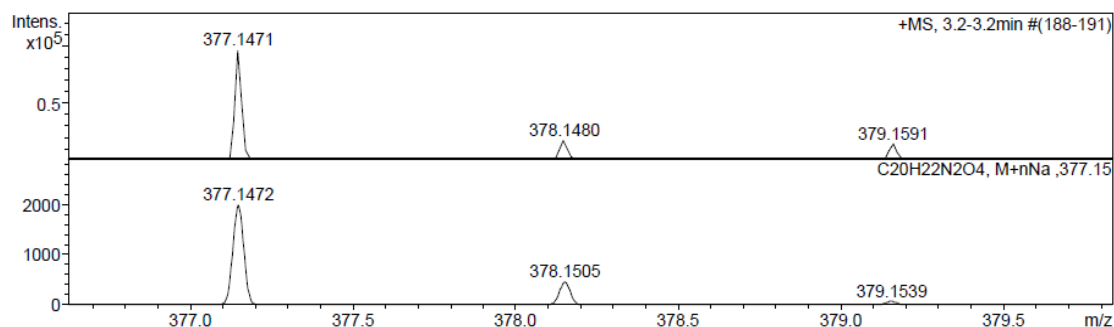
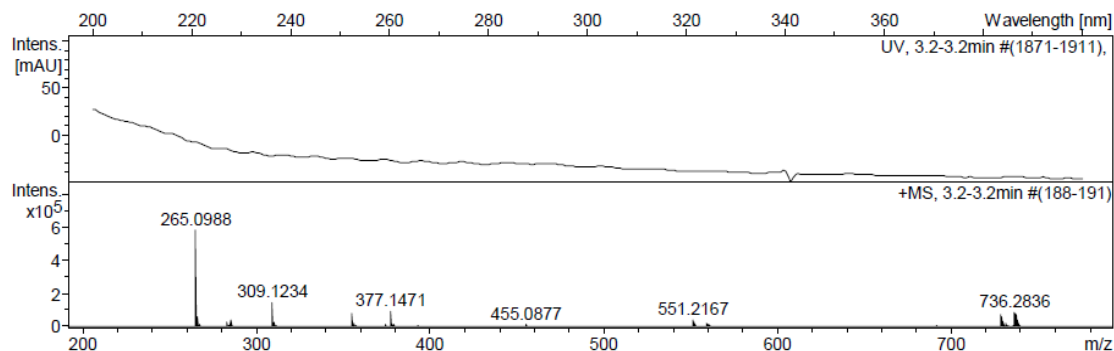
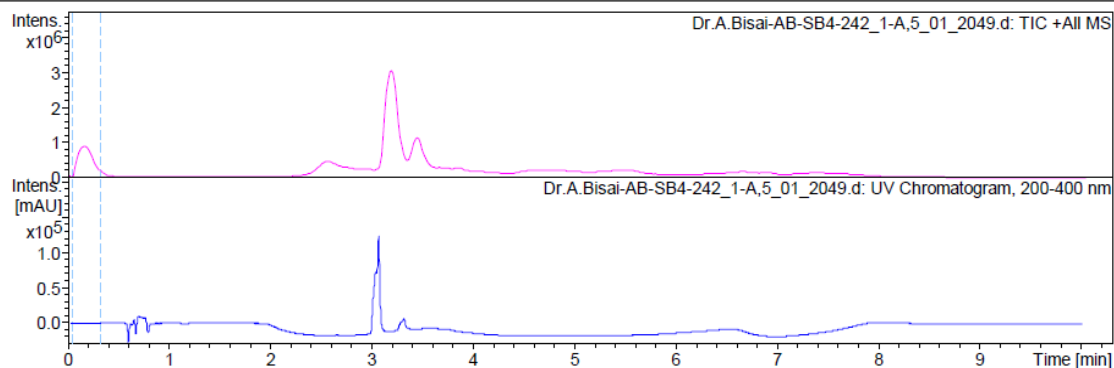
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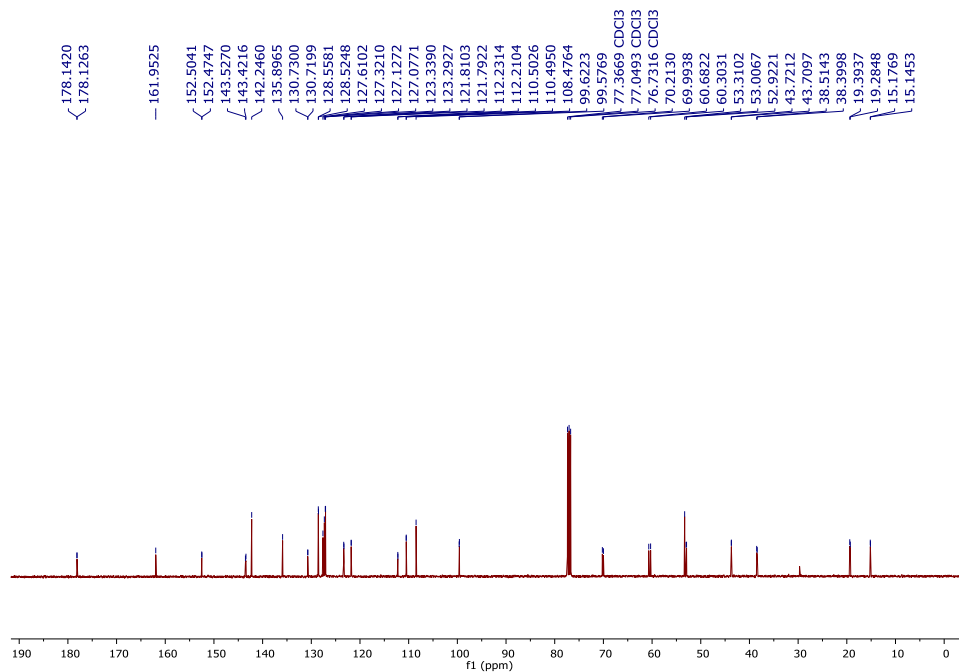
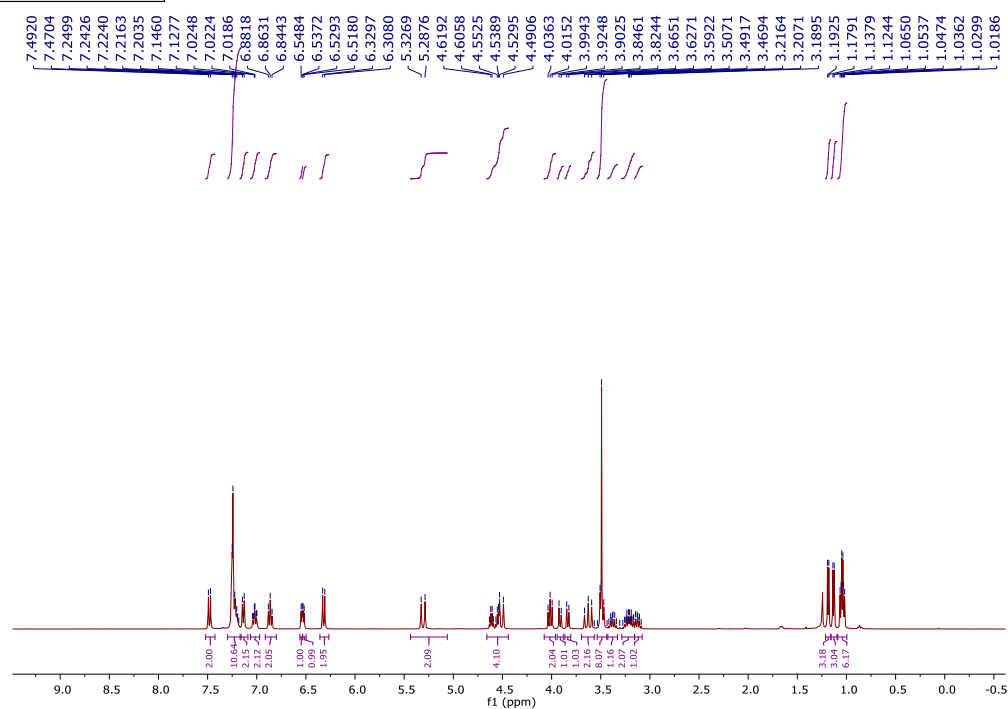
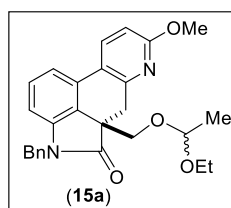
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 Sample Name Dr.A.Bisai-AB-SB4-242
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 Operator Amit
 Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
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Mass spectrum of compound **14**



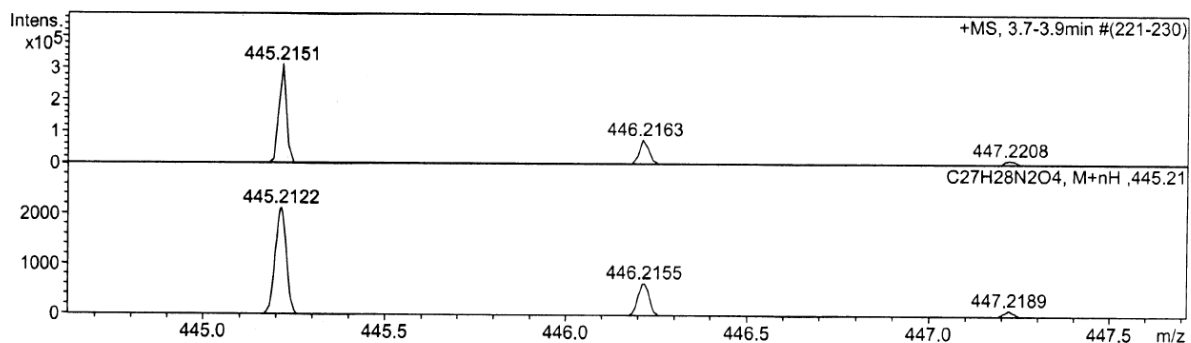
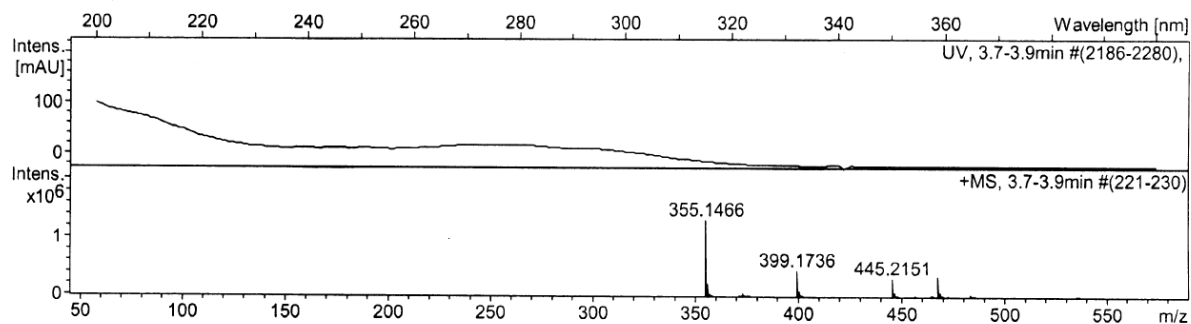
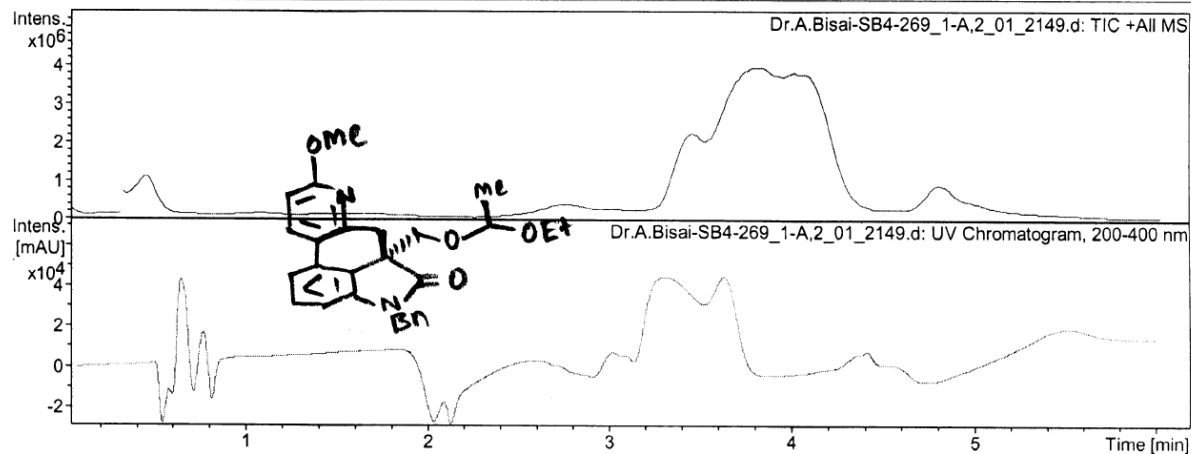
Display Report

Analysis Info

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Method HRLCMS-20 Sept.m Operator Amit
Sample Name Dr.A.Bisai-SB4-269 Instrument micrOTOF-Q II 10330
Comment

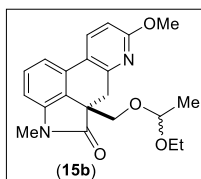
Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (15a)





Mass Spectrum SmartFormula Report

Analysis Info

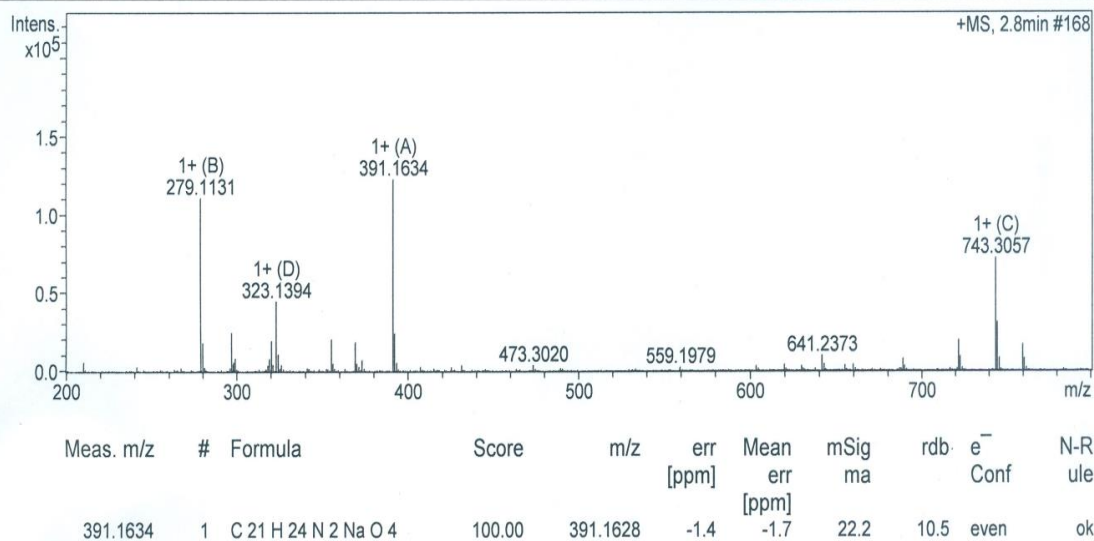
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Sample Name 2016307-SB-07-14-NME
Comment

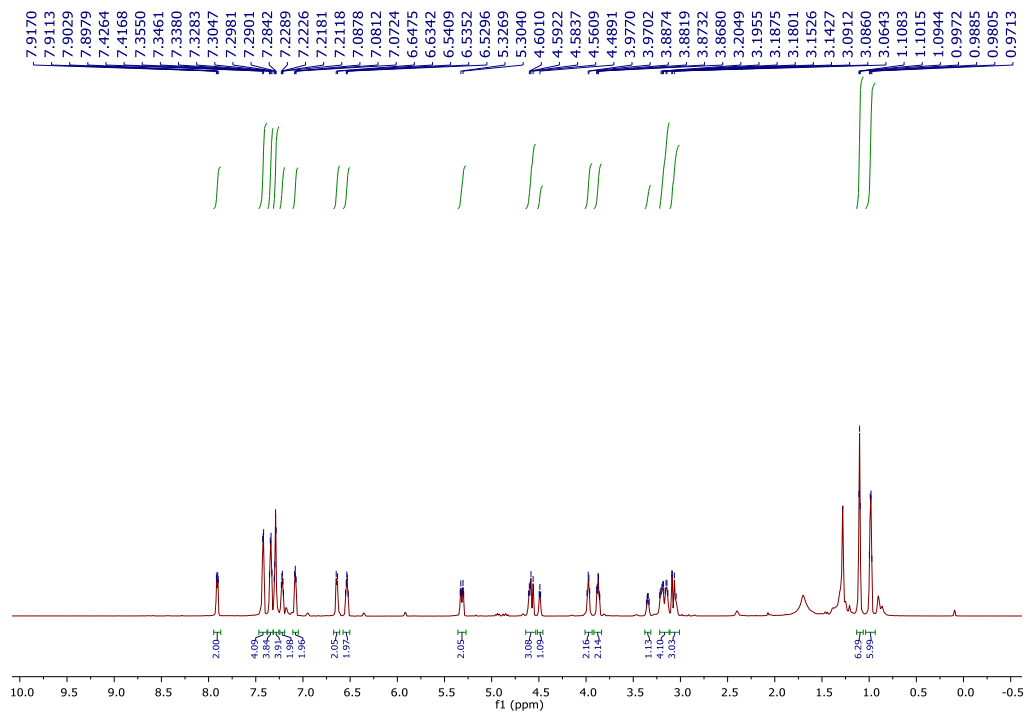
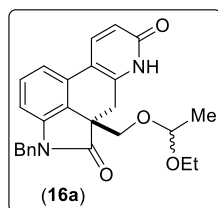
Instrument / Ser# maXis 4G 21240

Acquisition Parameter

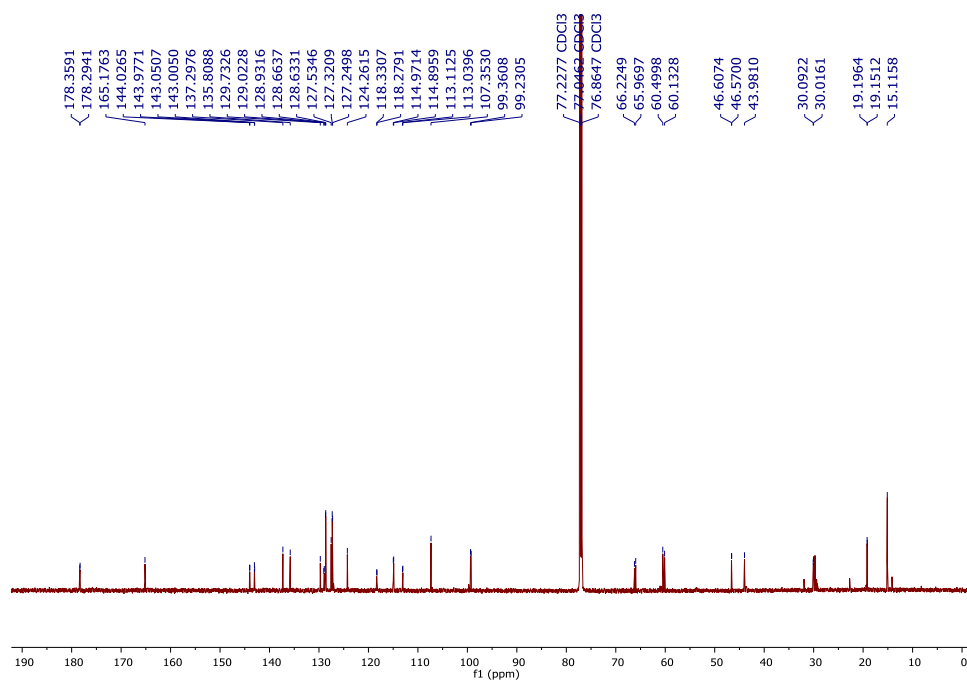
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Focus	Not active	Set Capillary	4000 V	Set Dry Heater	220 °C
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Scan End	1500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Mass spectrum of compound **15b**



^1H NMR (700 MHz, CDCl_3) of **(16a)**



^{13}C NMR (175 MHz, CDCl_3) of **(16a)**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\JAN-2016\06-JAN-2016\Dr.A.Bisai-SB7-177_1-A,7_01_4873.d
Method HRLCMS-20 Sept tune wide.m
Sample Name Dr.A.Bisai-SB7-177
Comment

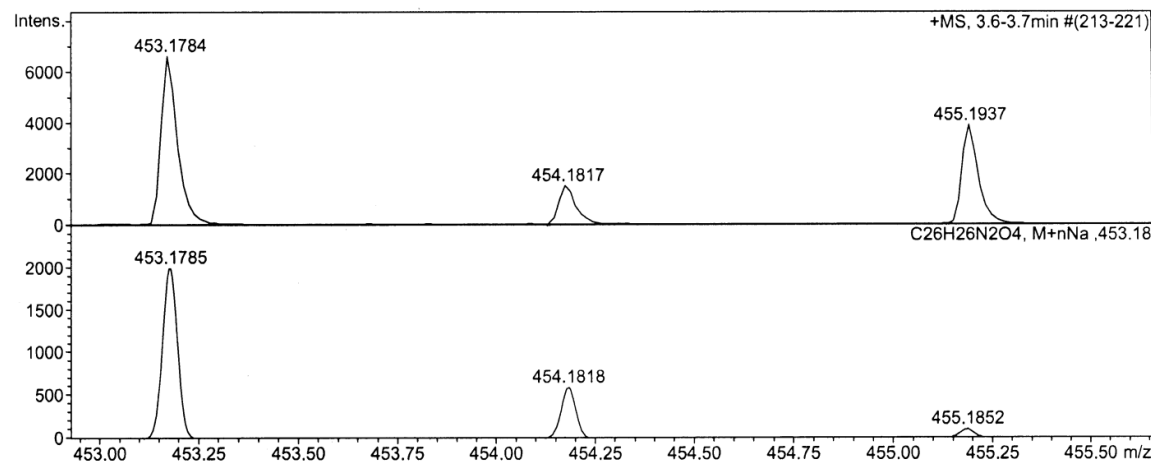
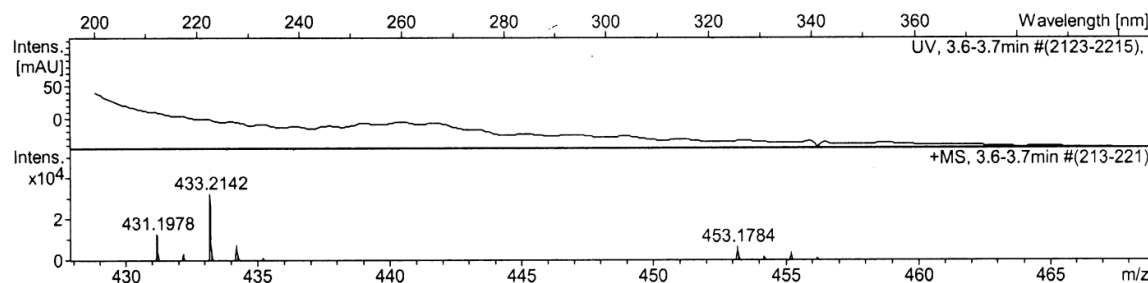
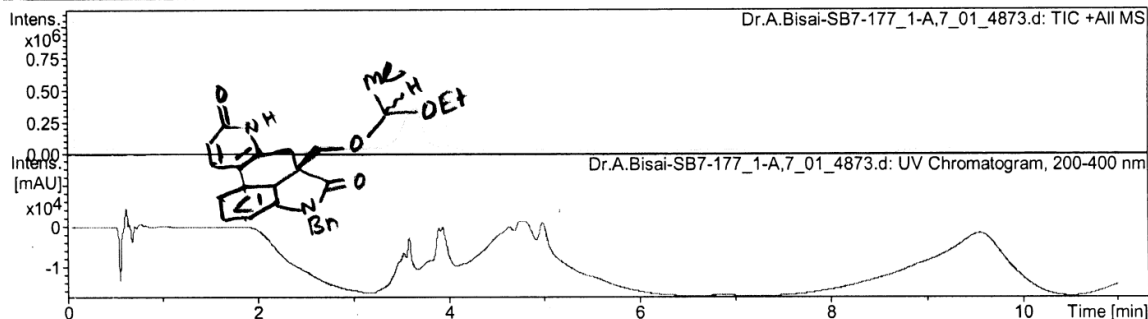
Acquisition Date 1/6/2016 3:29:10 PM

Operator RUCHI

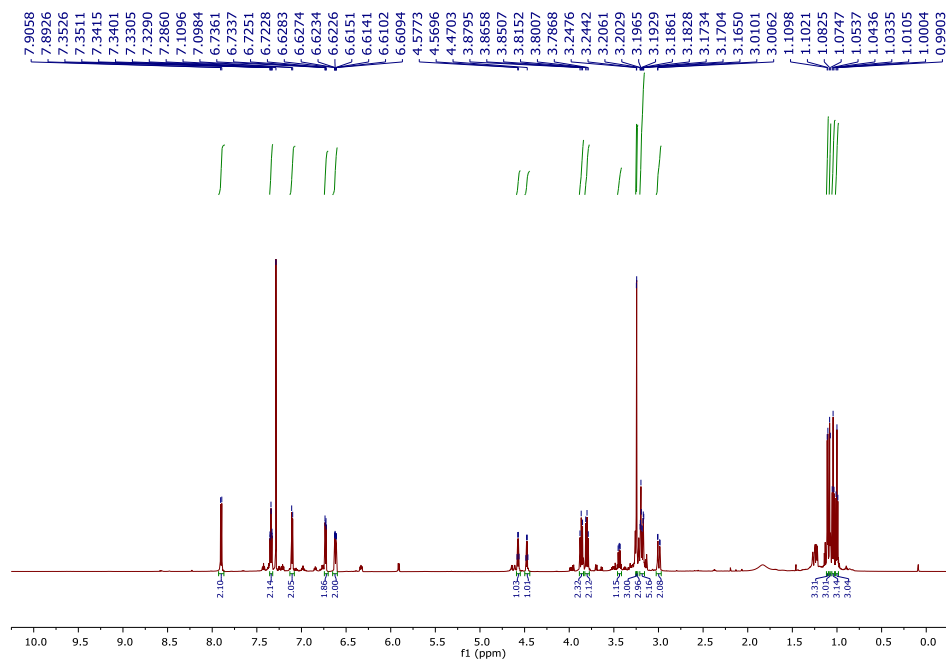
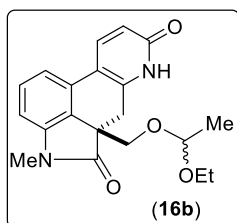
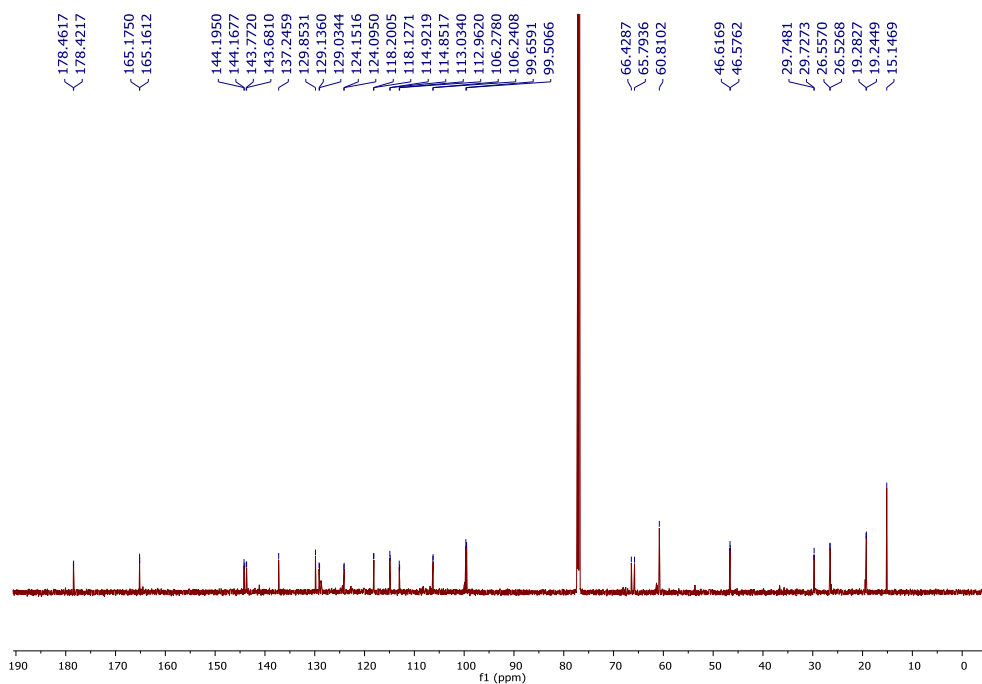
Instrument micrOTOF-Q II 10330

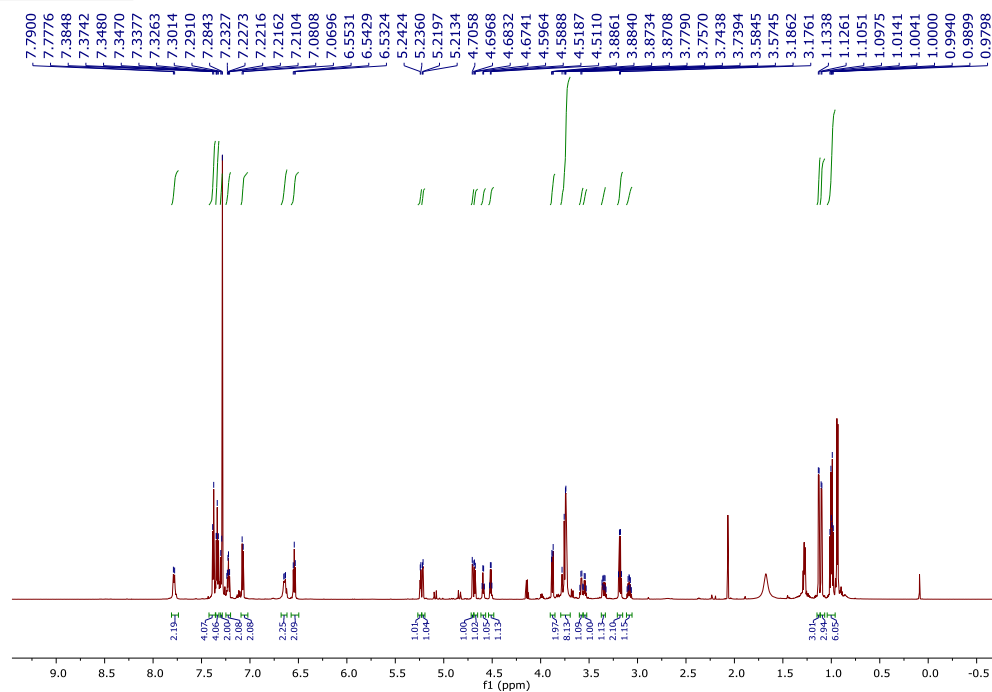
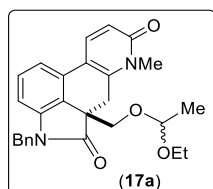
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

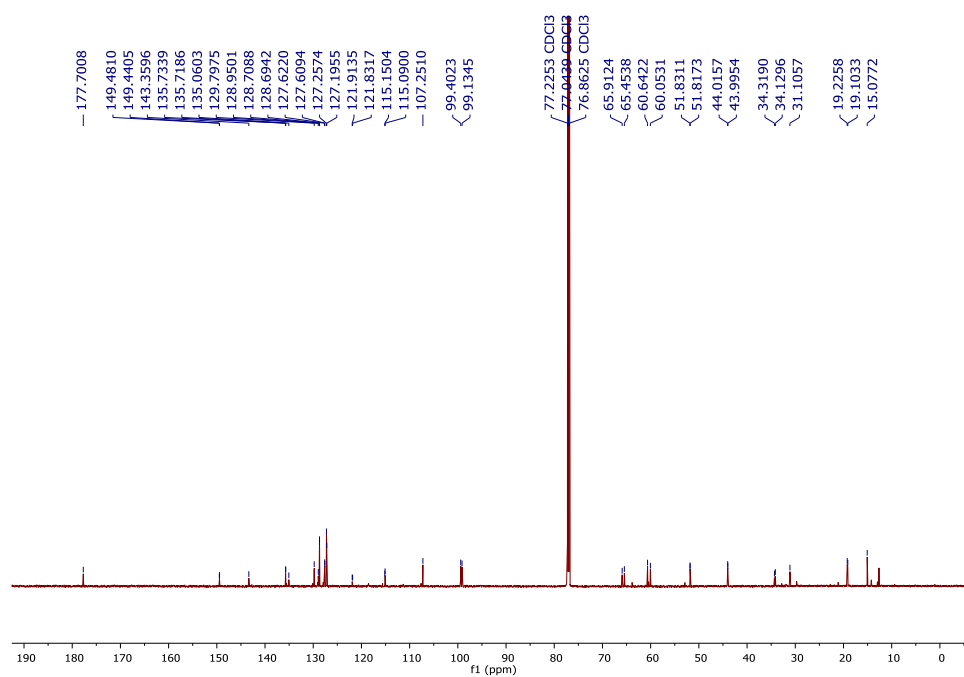


Scanned copy of mass spectrum of (16a)

 ^1H NMR (700 MHz, CDCl_3) of **(16b)** ^{13}C NMR (175 MHz, CDCl_3) of **(16b)**



^1H NMR (700 MHz, CDCl_3) of **(17a)**



^{13}C NMR (175 MHz, CDCl_3) of **(17a)**

Display Report

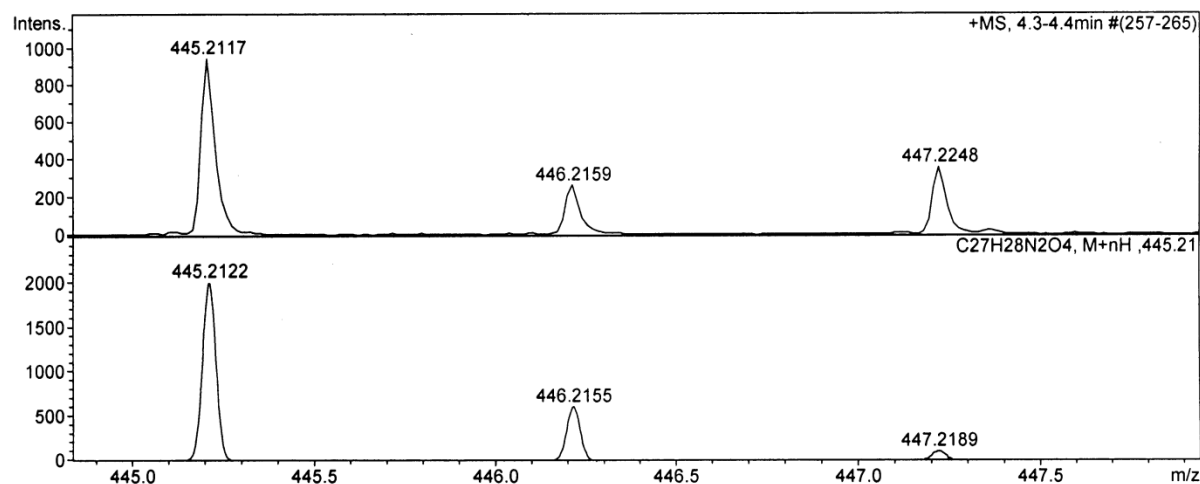
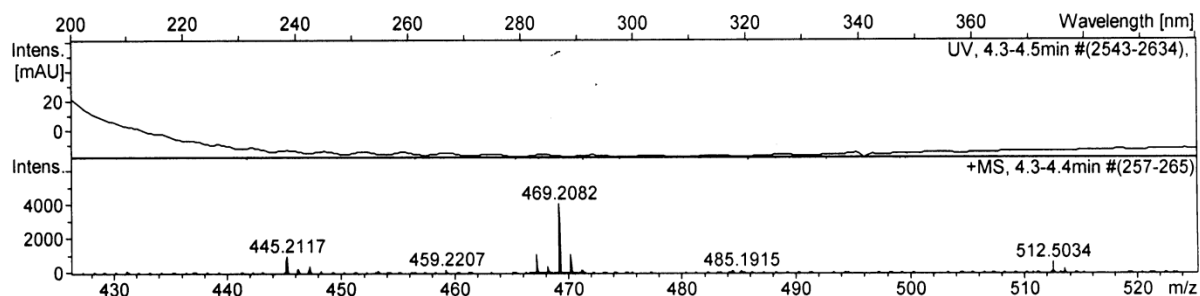
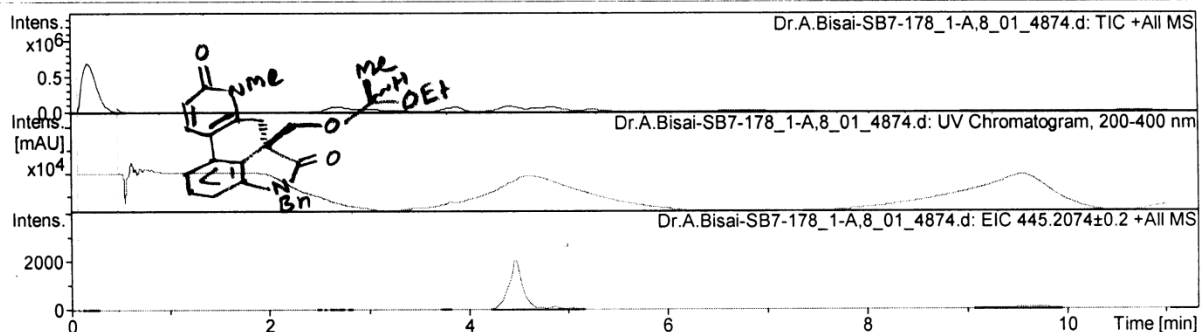
Analysis Info

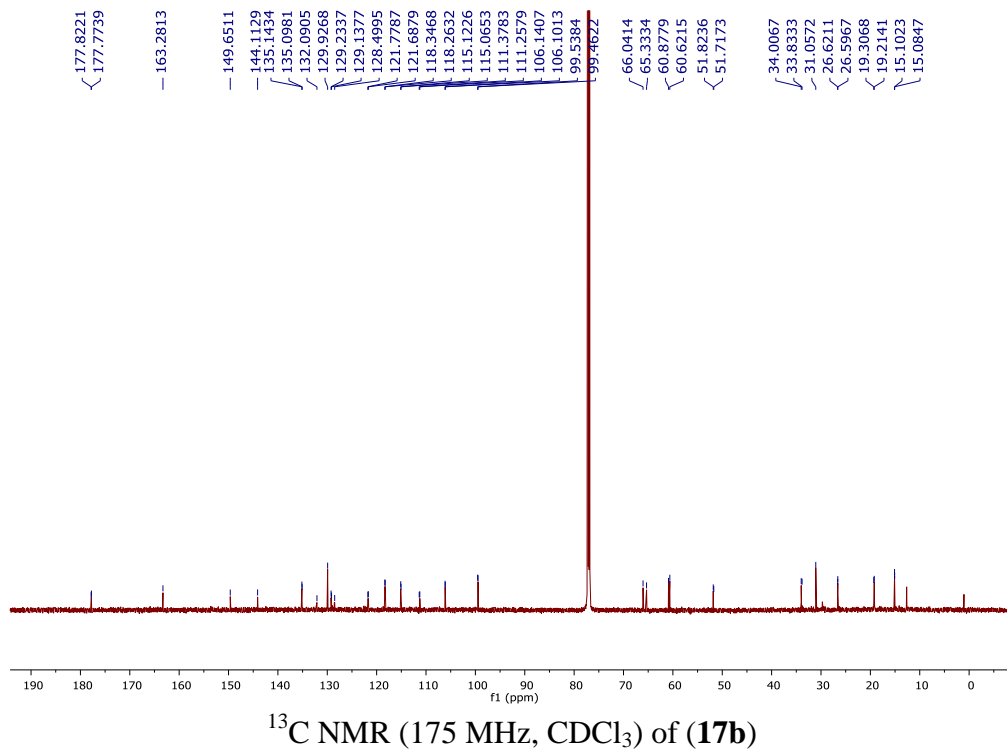
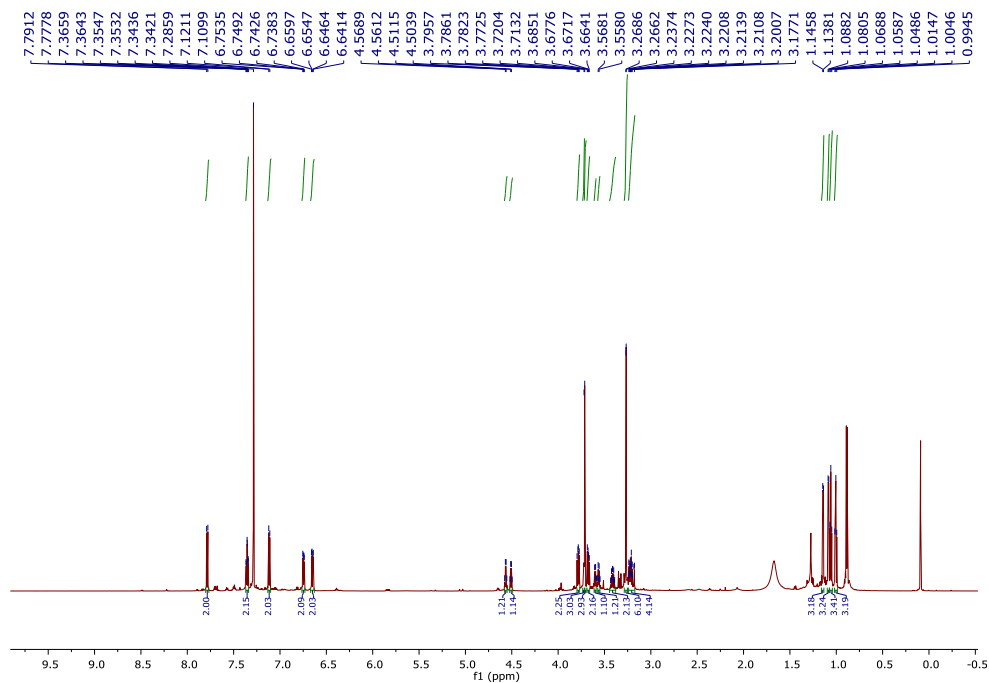
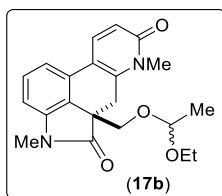
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Method HRLCMS-20 Sept tune wide.m
Sample Name Dr.A.Bisai-SB7-178
Comment

Acquisition Date 1/6/2016 3:41:21 PM
Operator RUCHI
Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste





Display Report

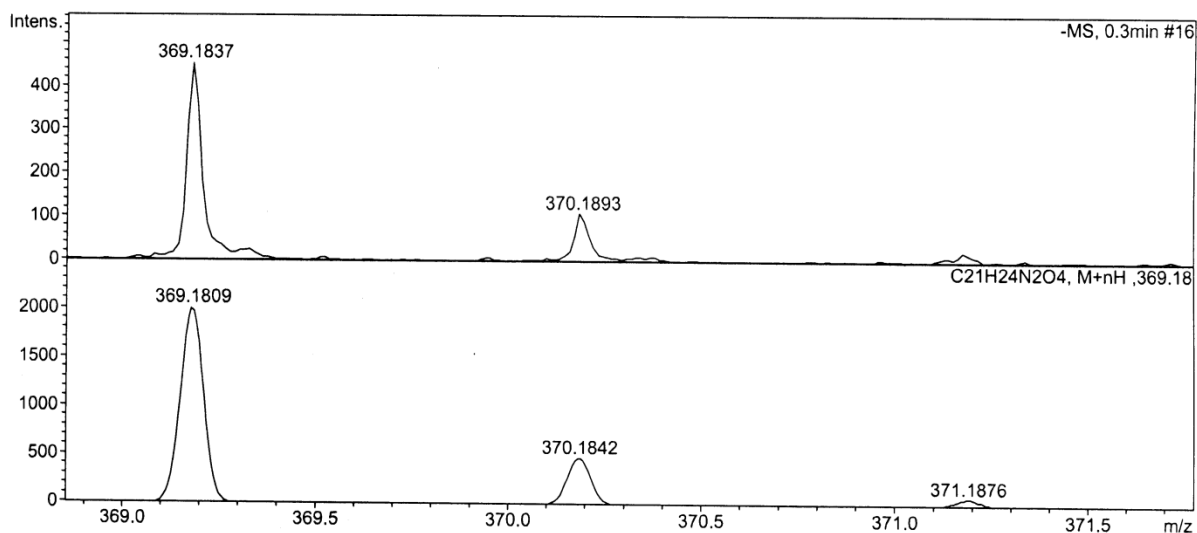
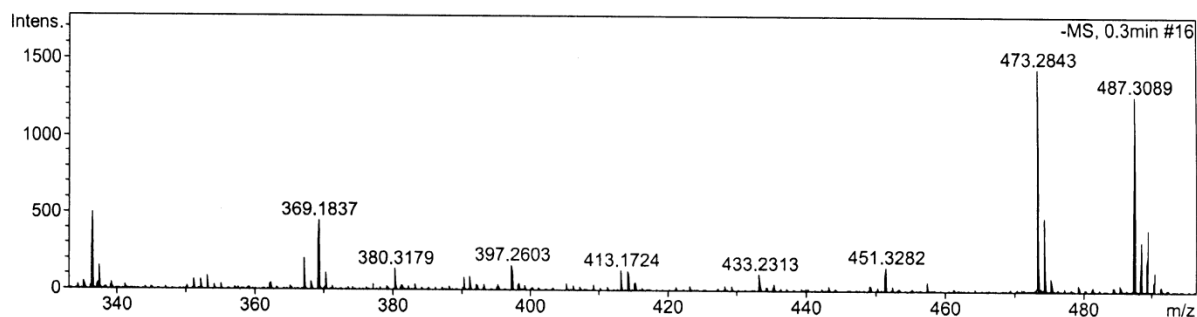
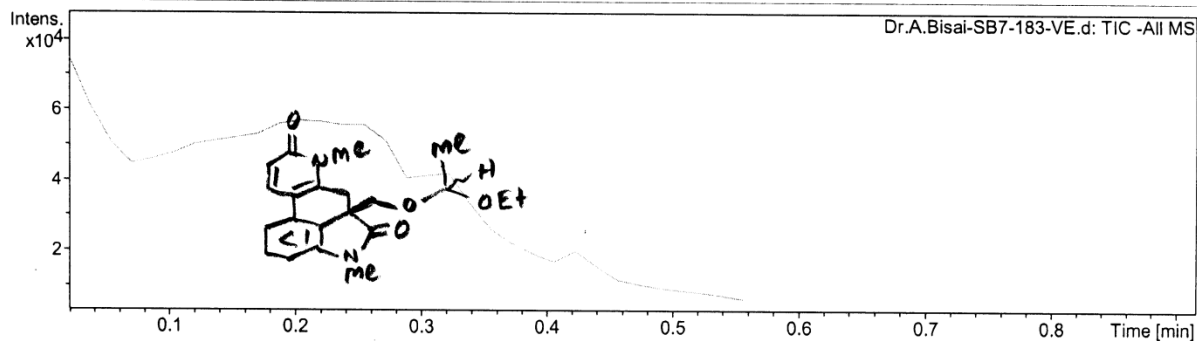
Analysis Info

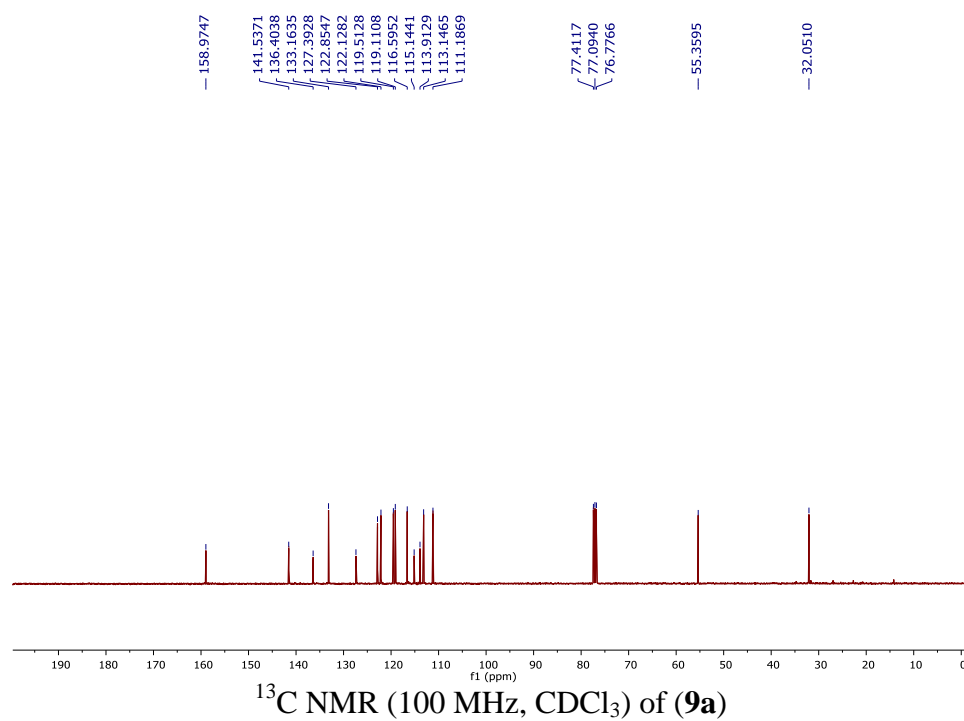
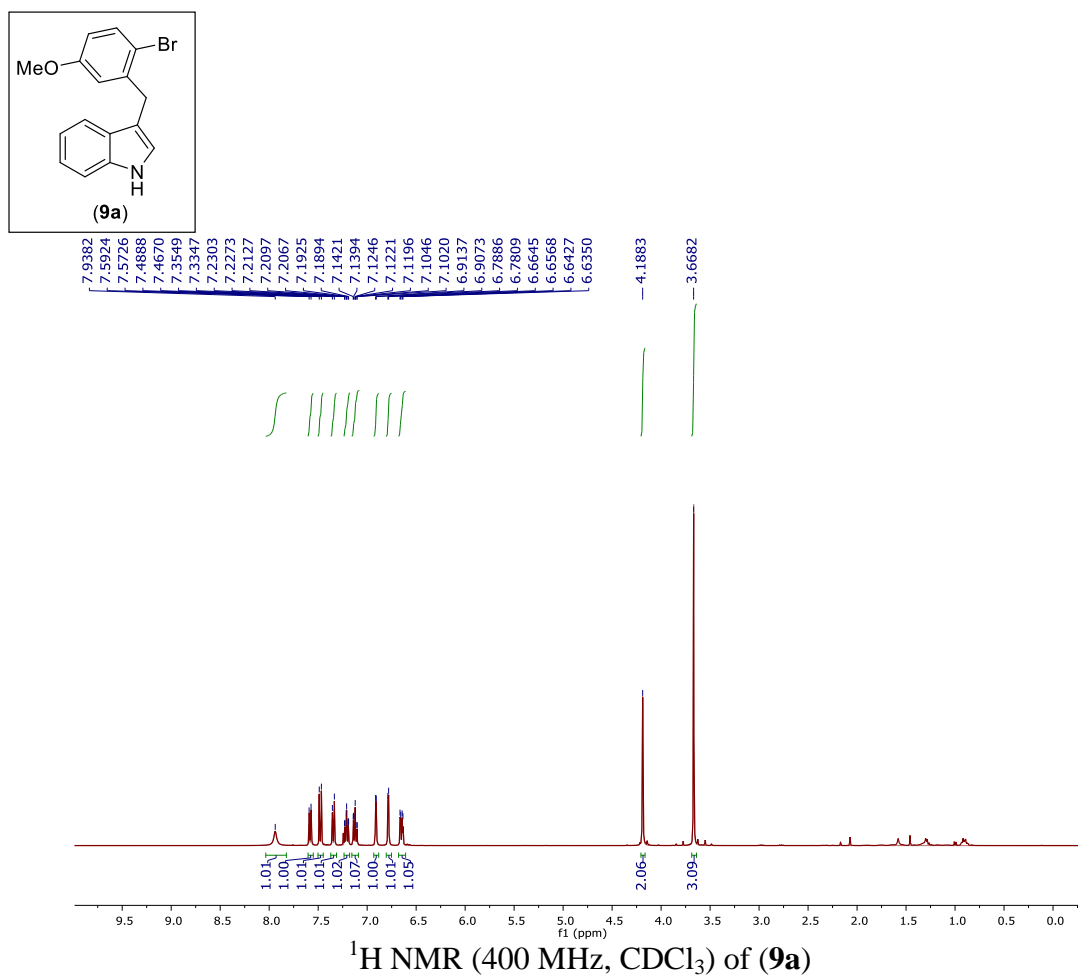
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Method tune_low_APCI.m
Sample Name SB7-183-VE
Comment

Acquisition Date 1/21/2016 3:31:19 PM
Operator RUCHI
Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	APCI	Ion Polarity	Negative	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\12-04-2016\Dr.A.Bisai-SB4-131_1-D,3_01_5856.d
Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-SB4-131
Comment

Acquisition Date 4/12/2016 4:10:25 PM

Operator DIMPLE

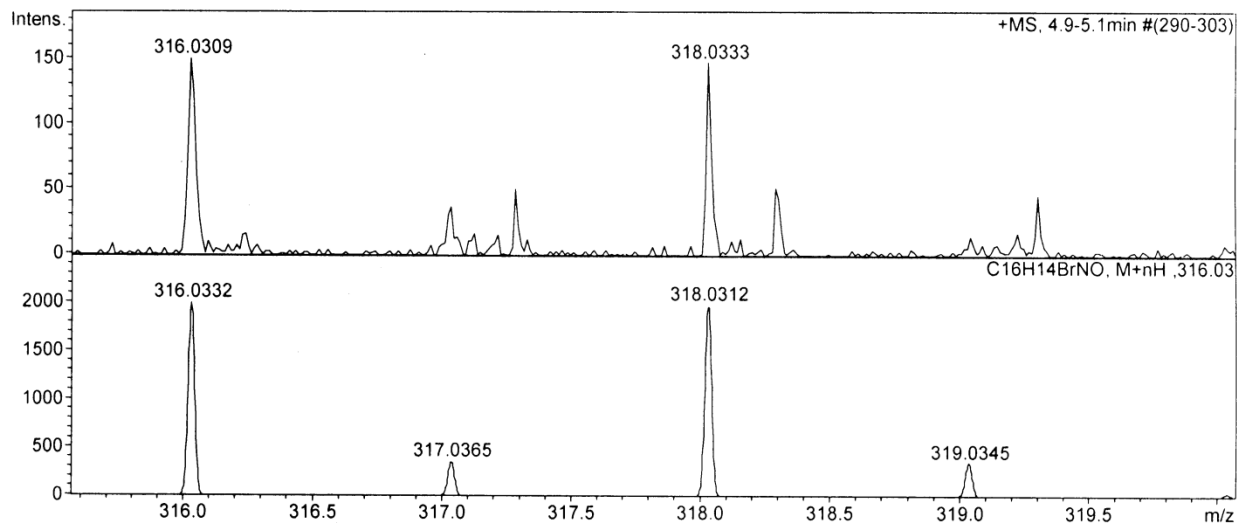
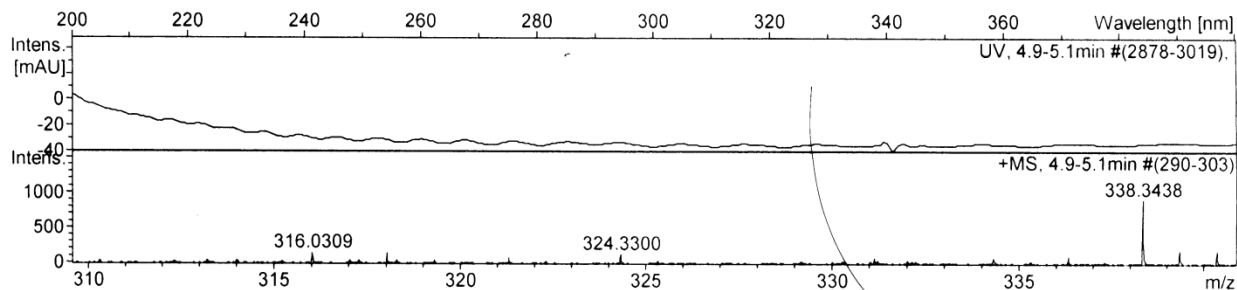
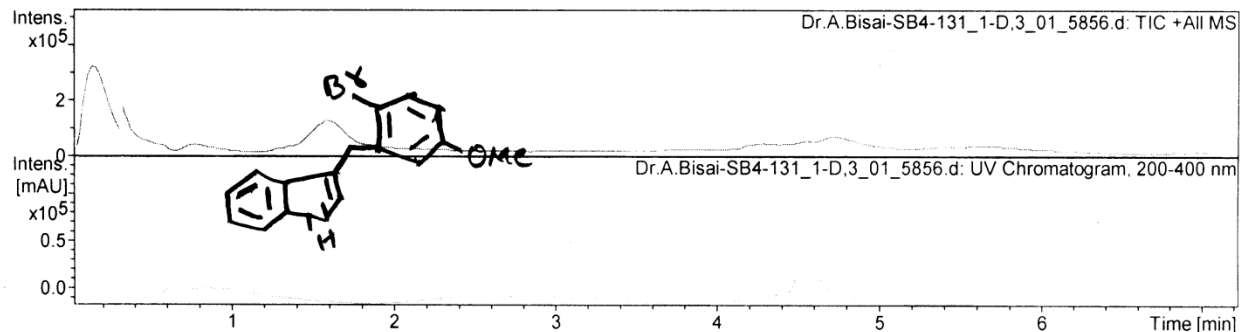
Instrument micrOTOF-Q II 10330

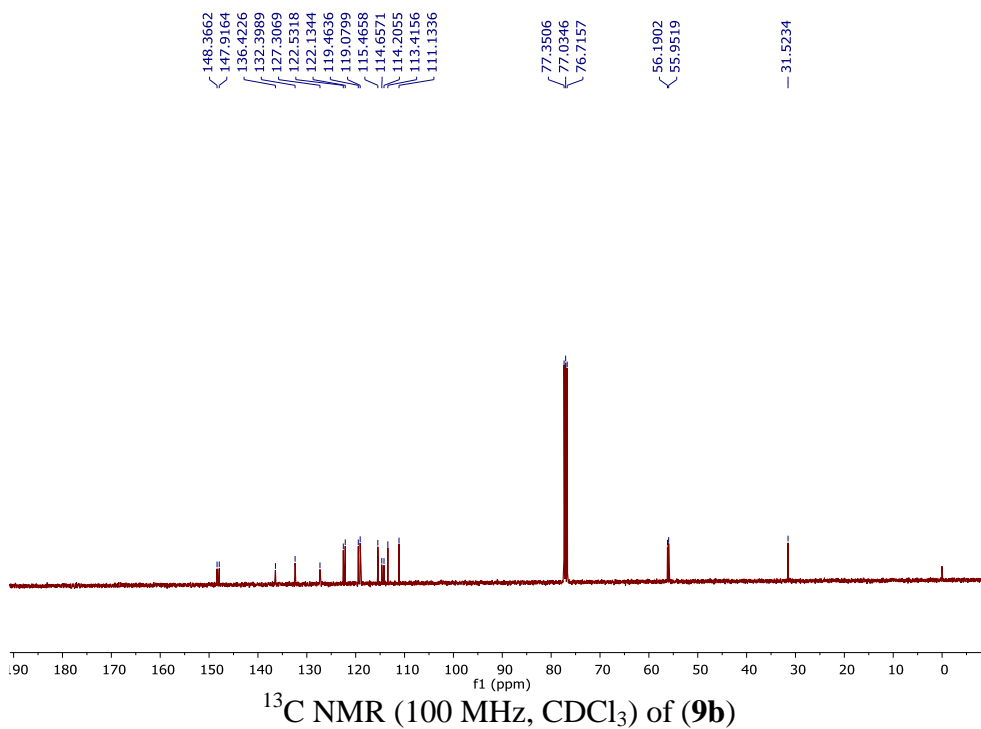
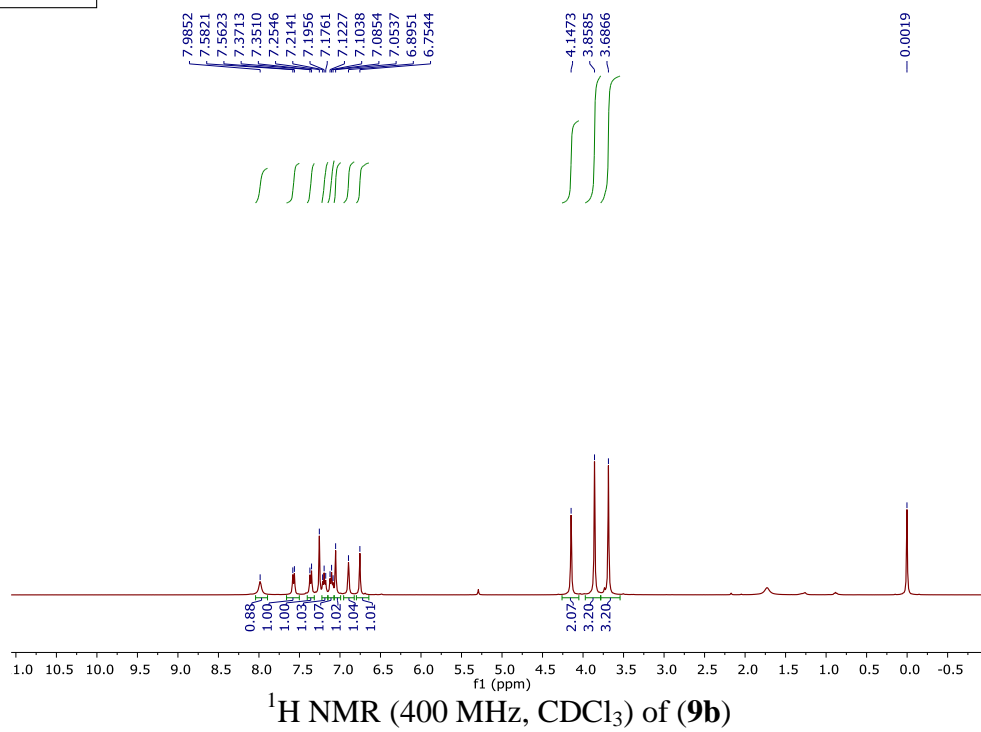
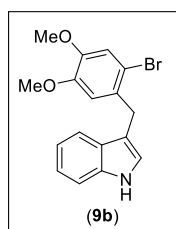
Acquisition Parameter

Source Type ESI
Focus Active
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Scan End 3000 m/z

Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 130.0 Vpp

Set Nebulizer 1.2 Bar
Set Dry Heater 200 °C
Set Dry Gas 7.0 l/min
Set Divert Valve Waste





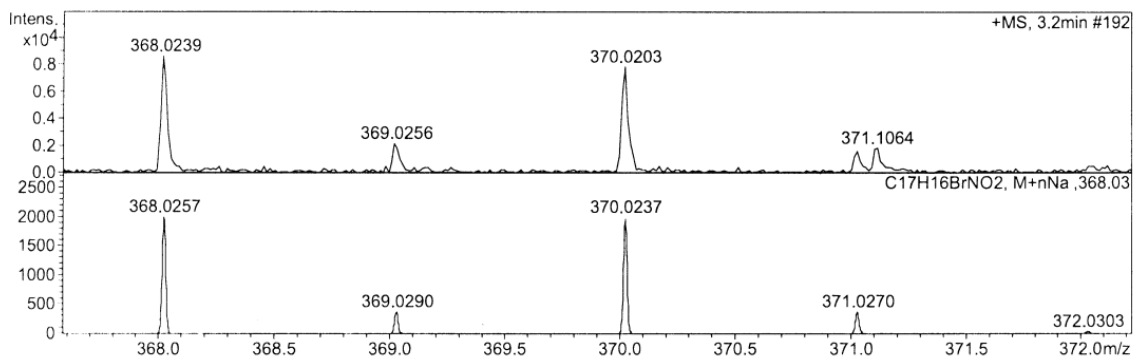
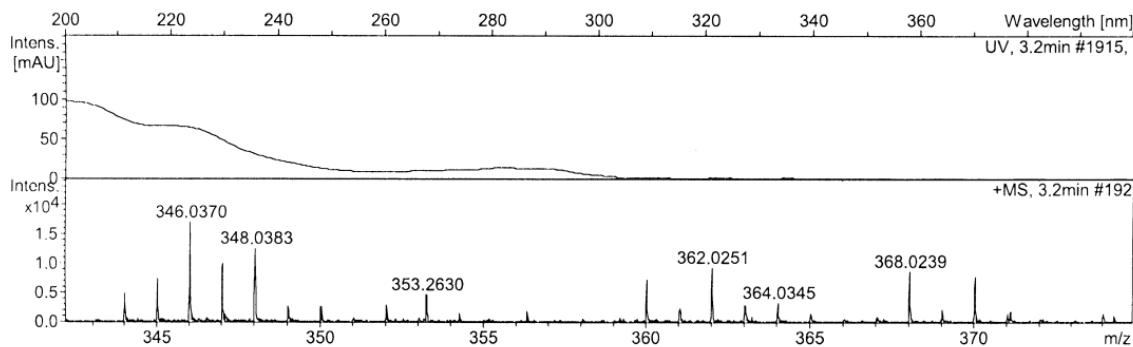
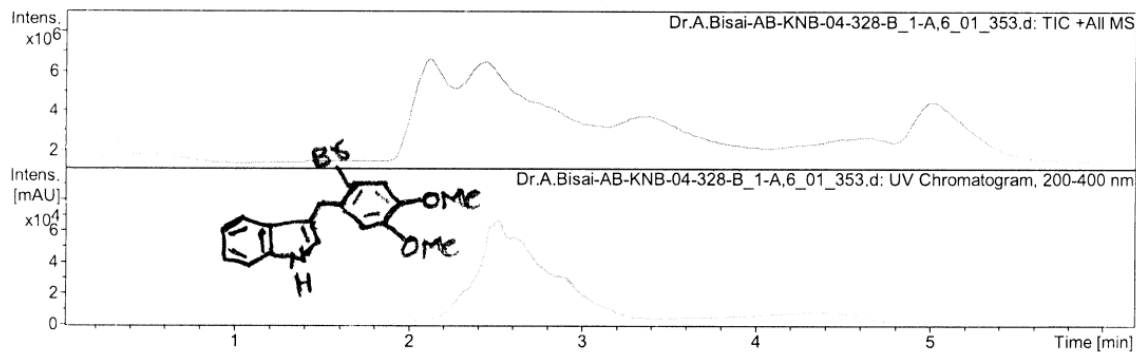
Display Report

Analysis Info

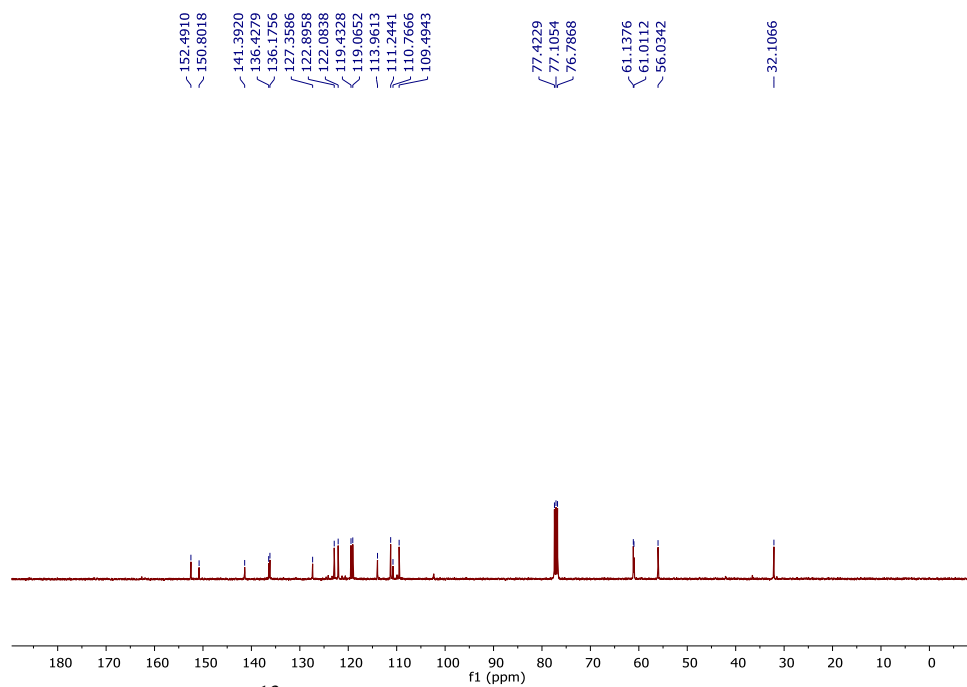
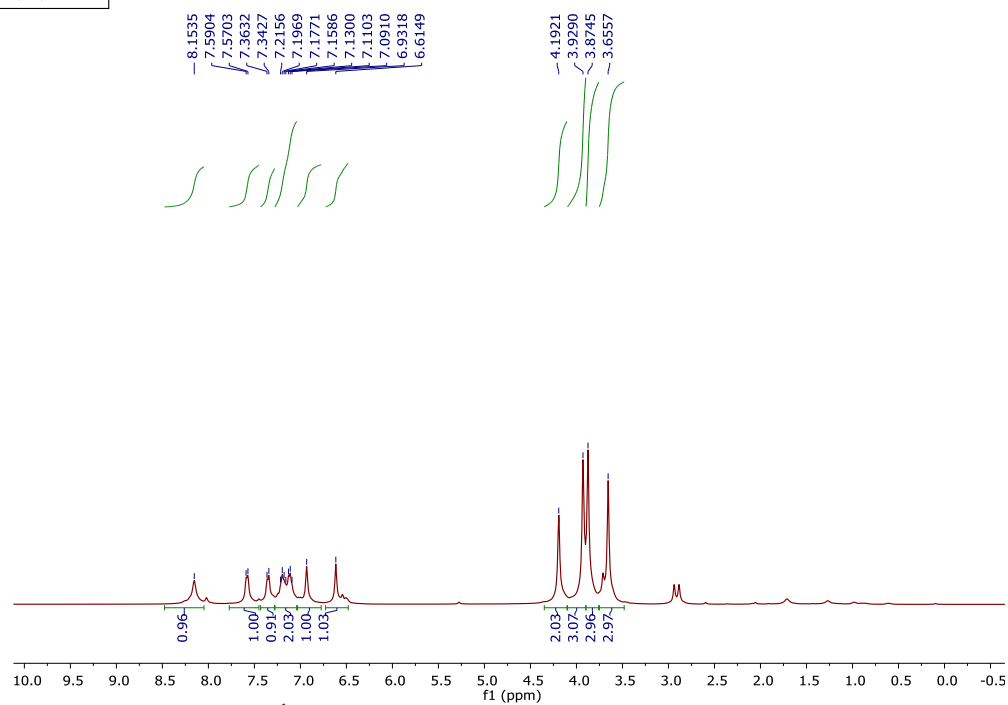
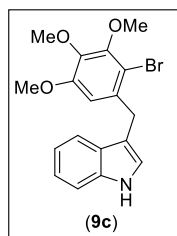
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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr.A.Bisai-AB-KNB-04-328-B	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (9b)



Display Report

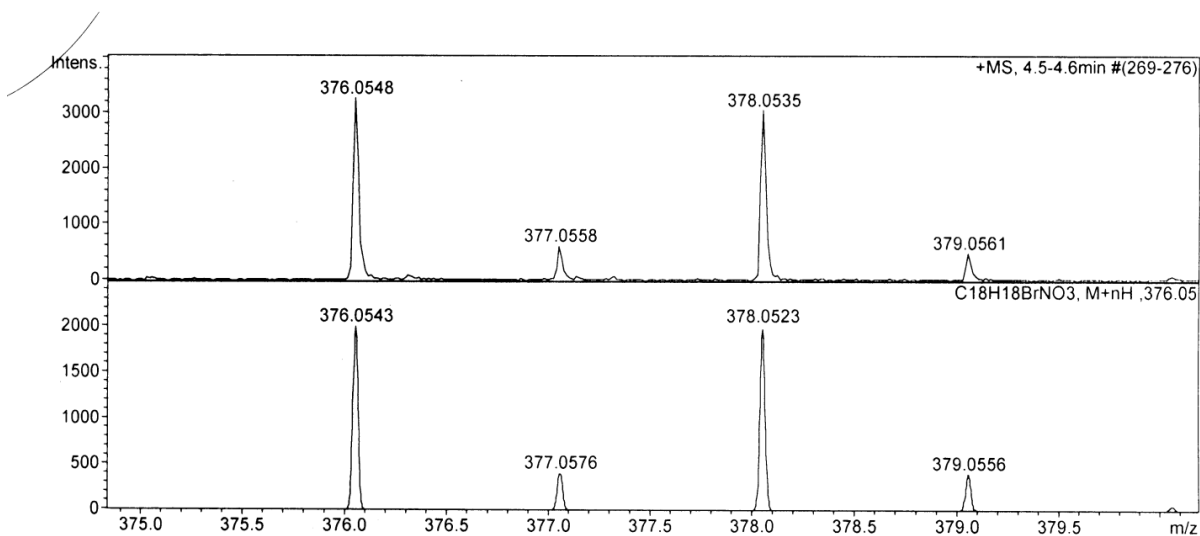
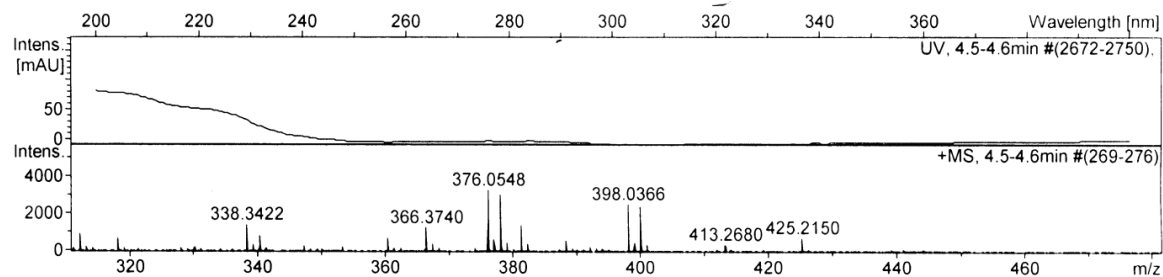
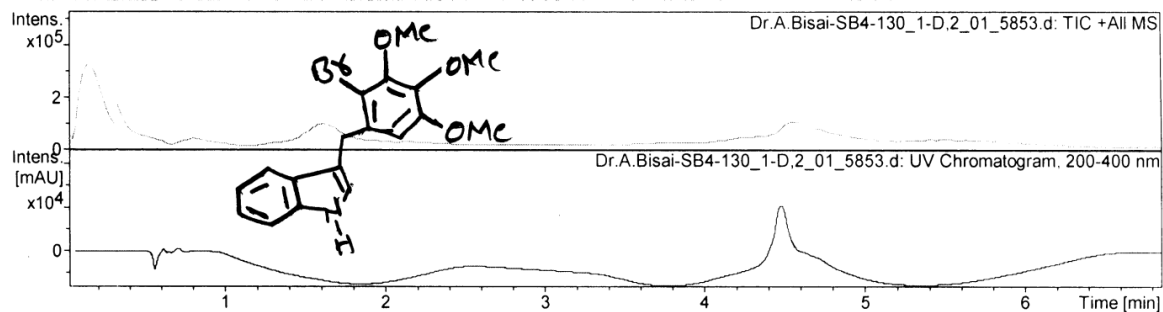
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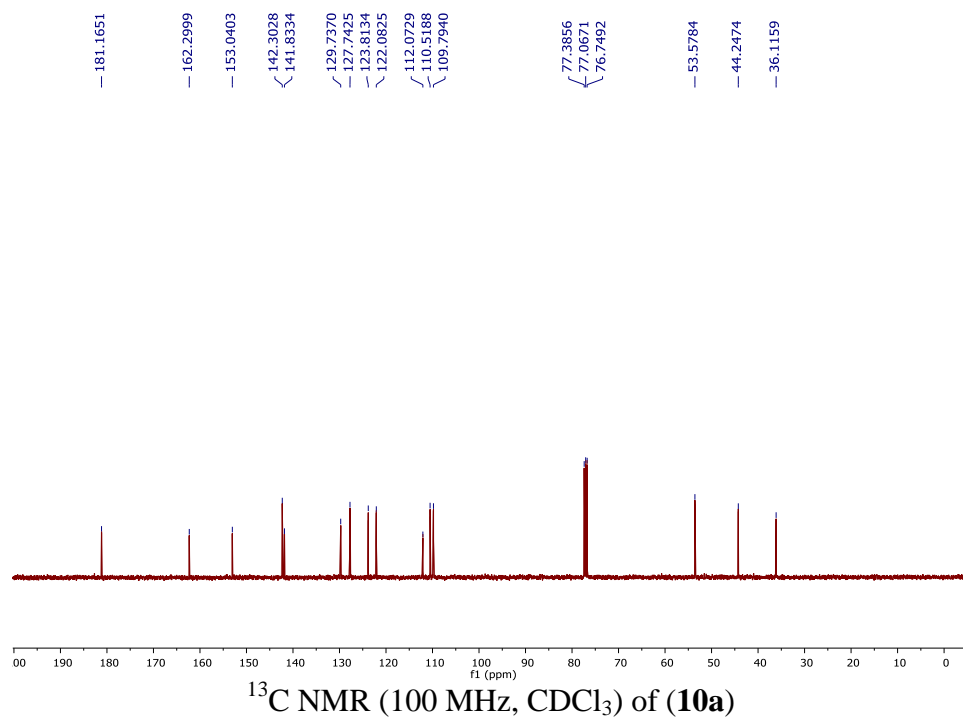
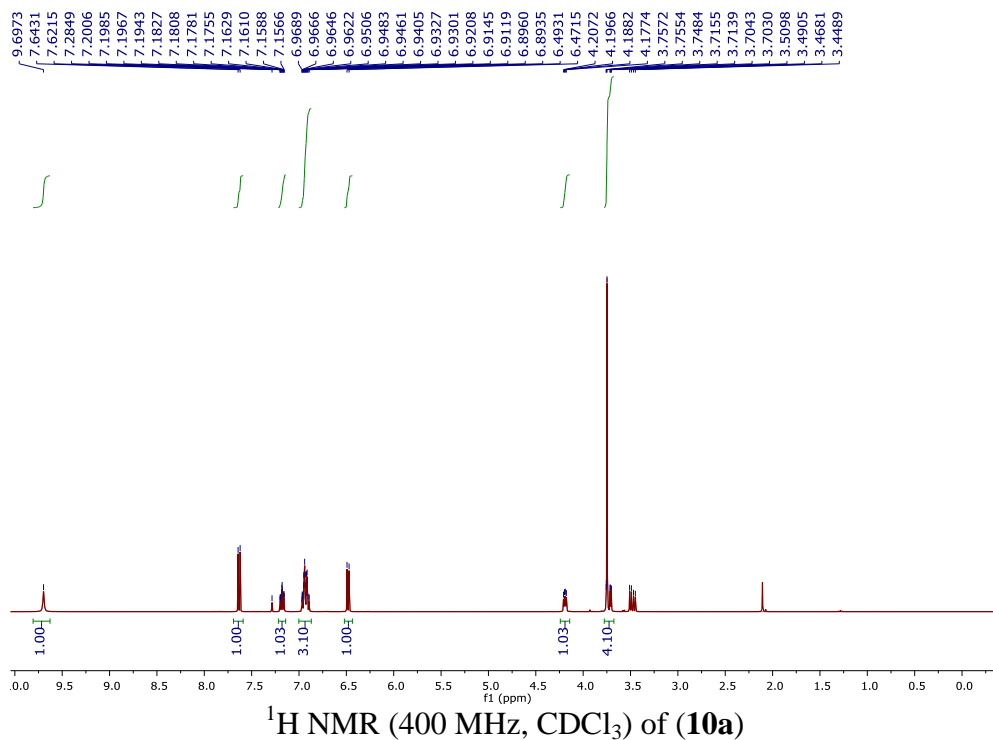
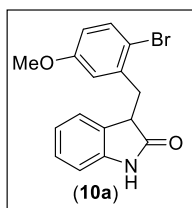
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Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-SB4-130
Comment

Acquisition Date 4/12/2016 3:45:47 PM
Operator DIMPLE
Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





Display Report

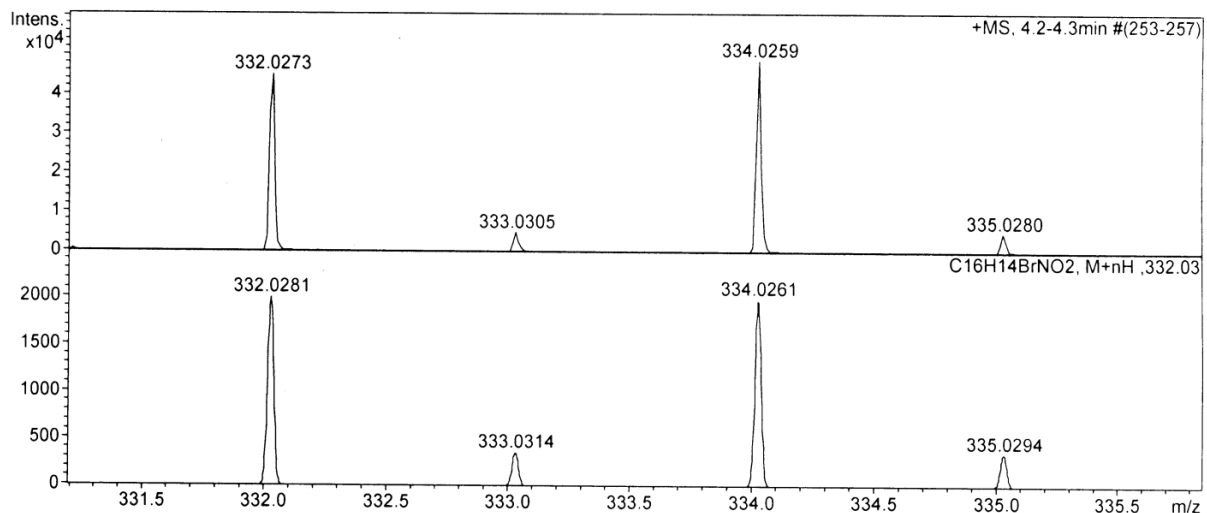
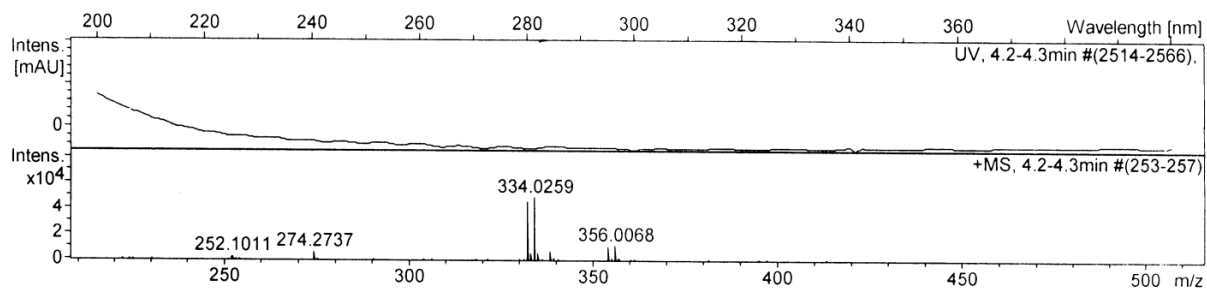
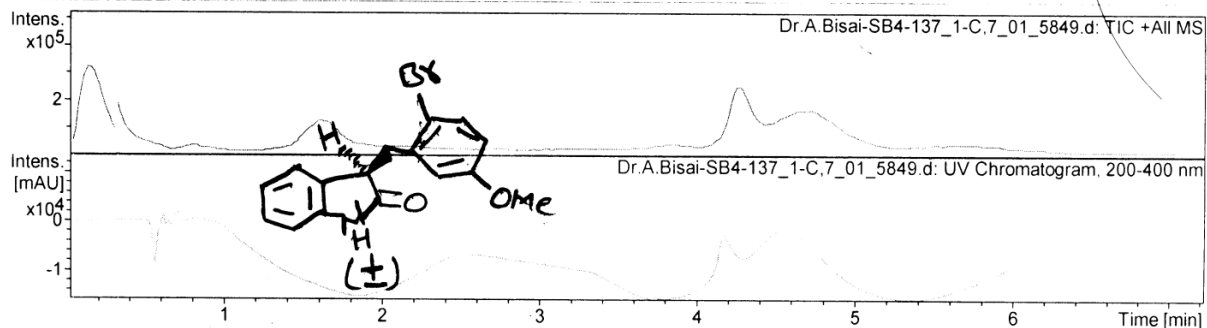
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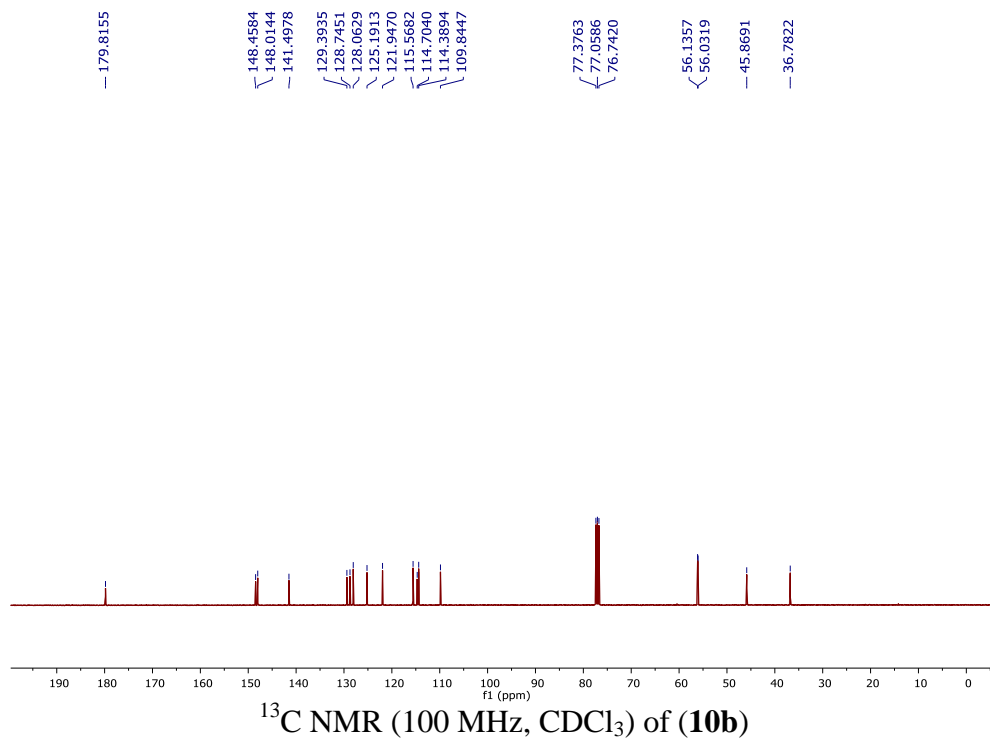
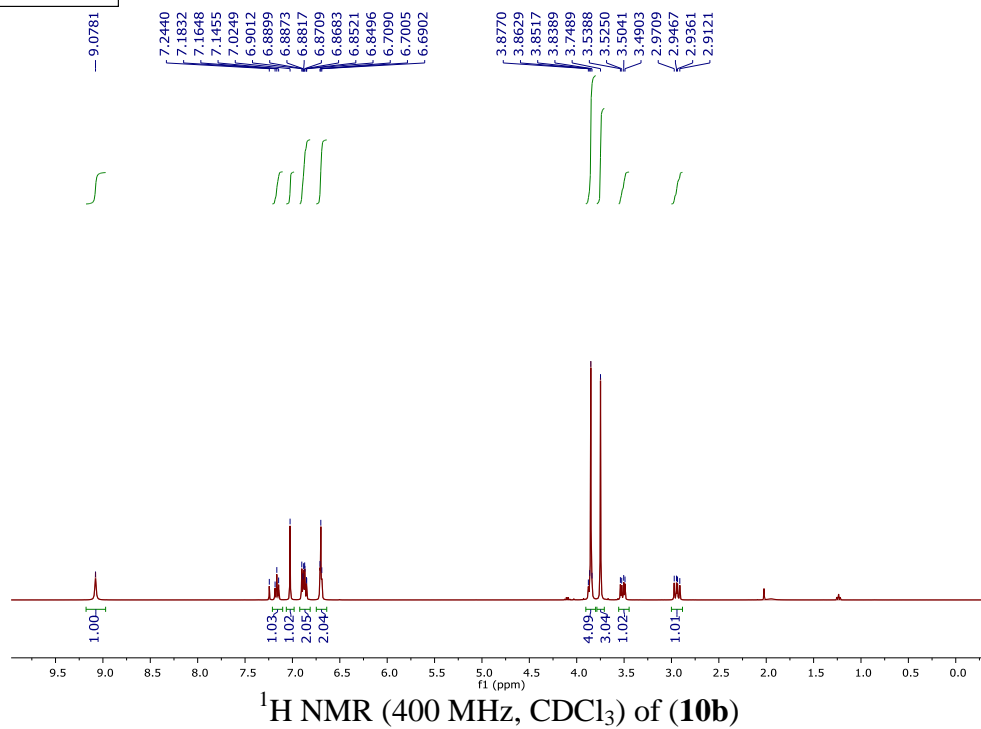
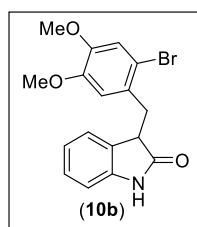
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 Sample Name Dr.A.Bisai-SB4-137
 Comment

Acquisition Date 4/12/2016 3:13:07 PM
 Operator DIMPLE
 Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





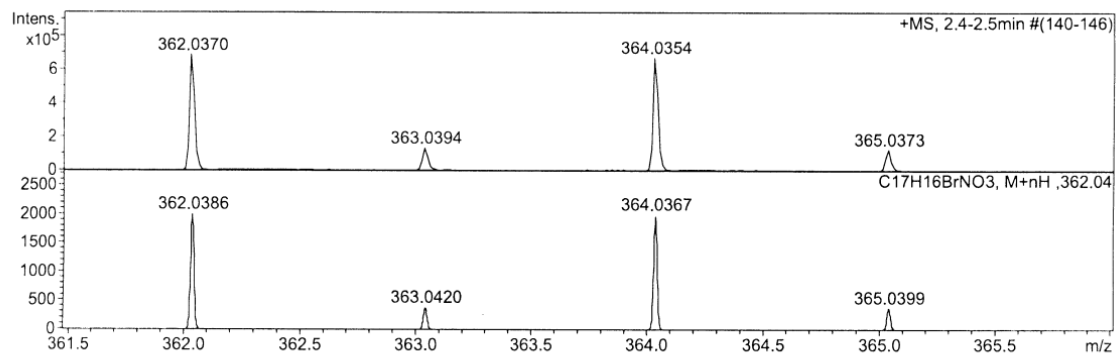
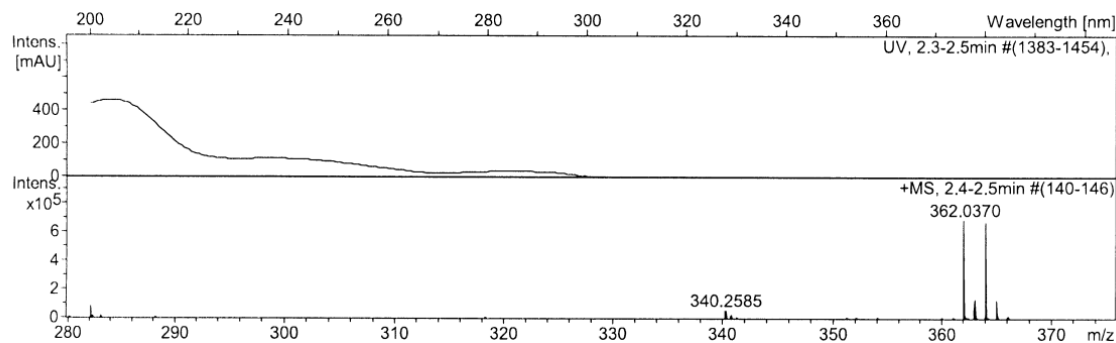
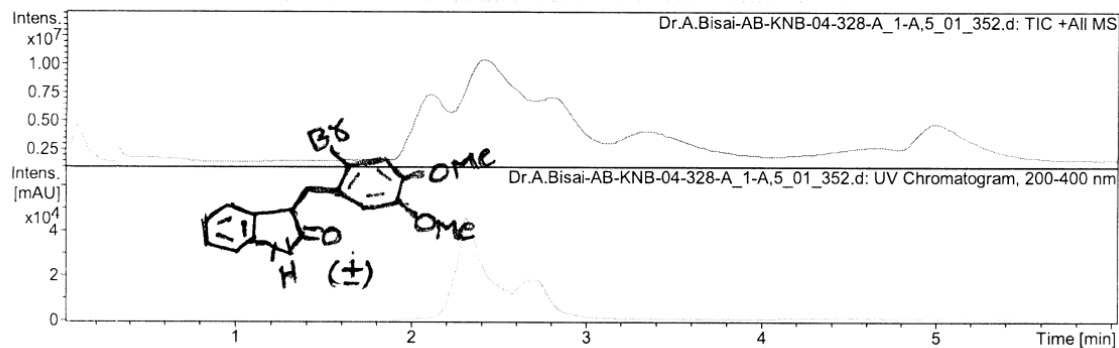
Display Report

Analysis Info

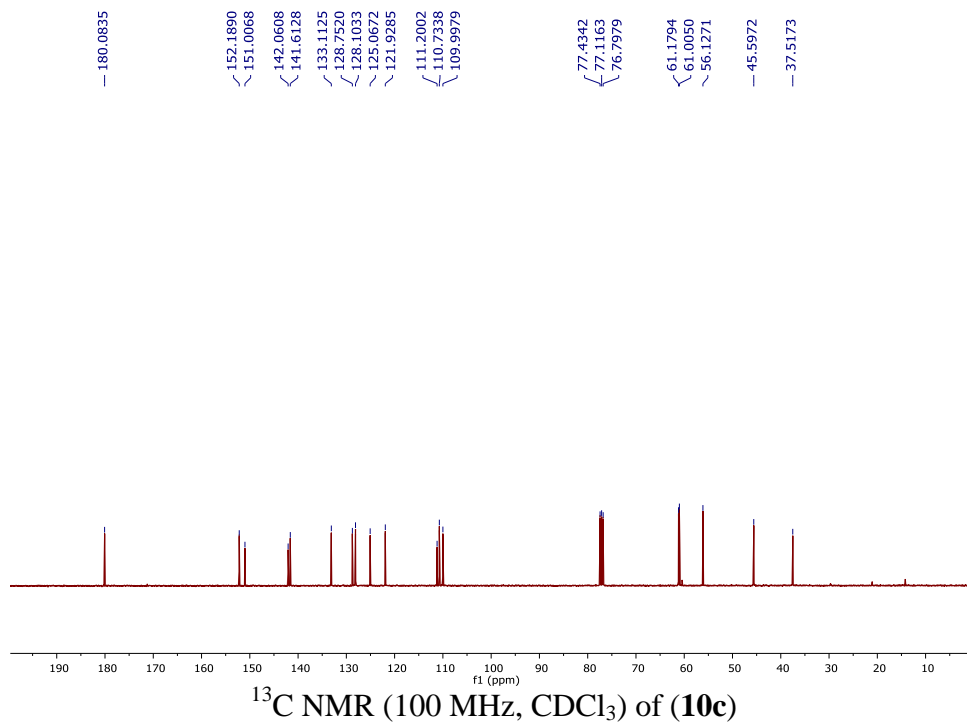
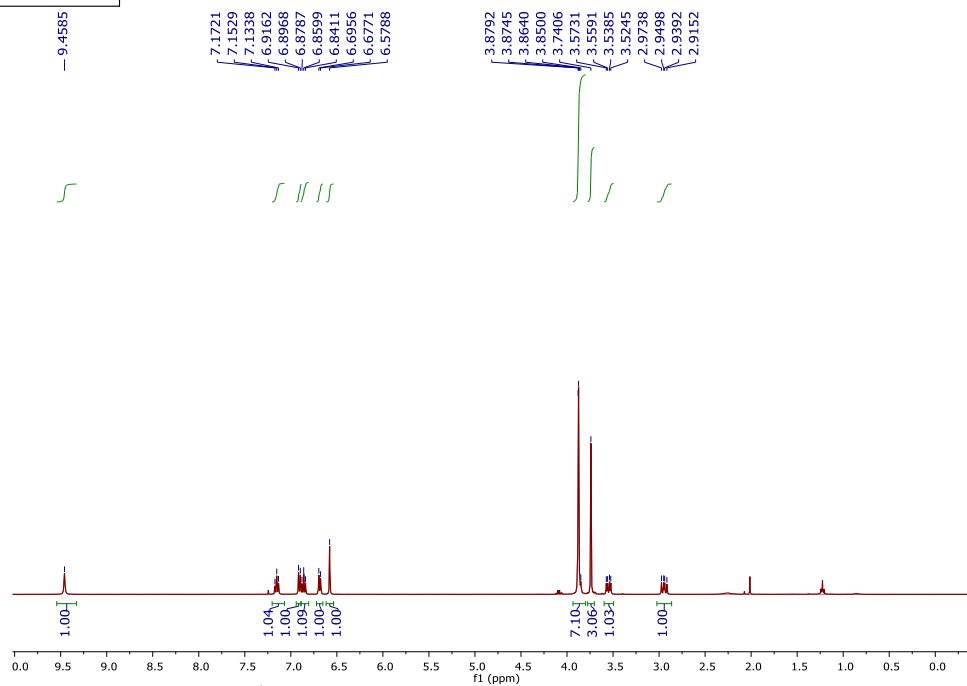
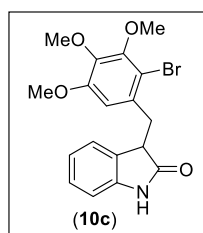
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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr.A.Bisai-AB-KNB-04-328-A	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (10b)



Display Report

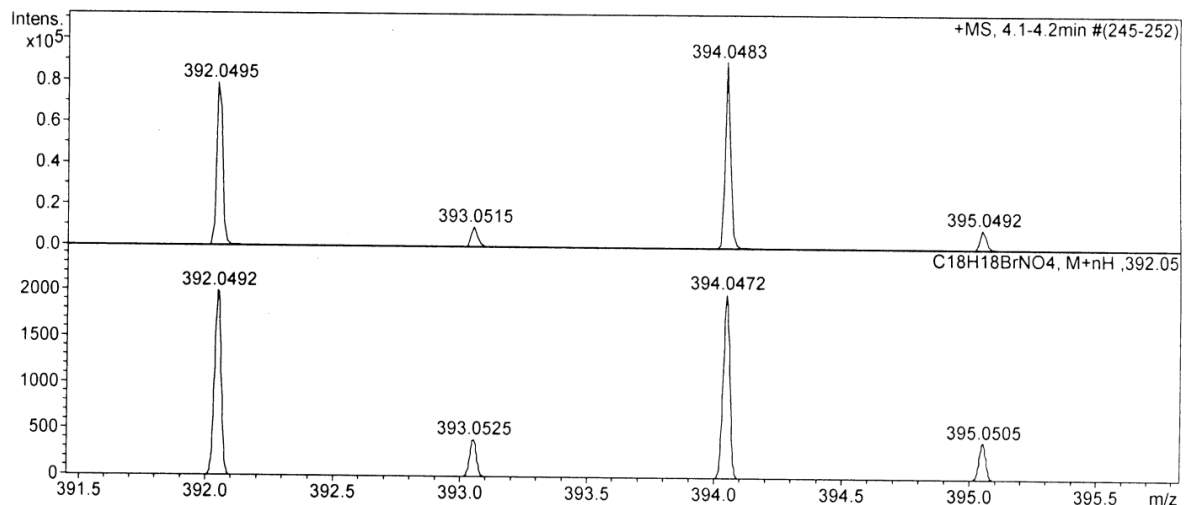
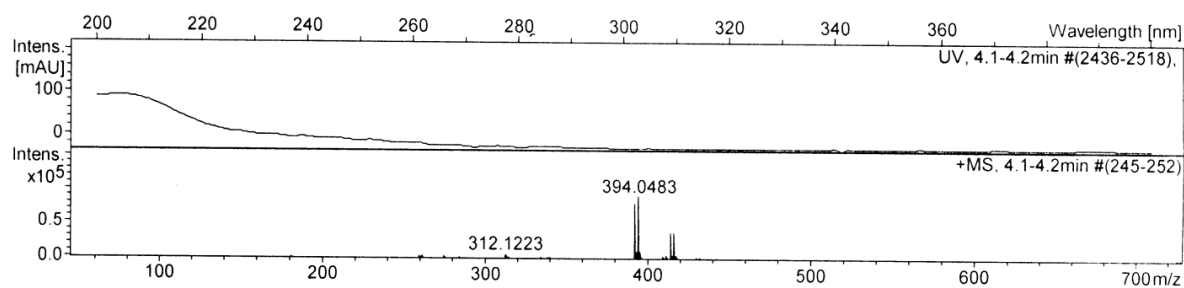
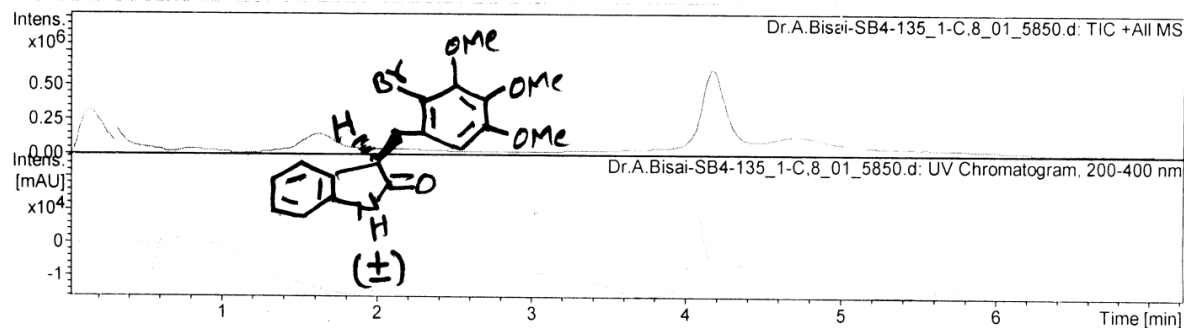
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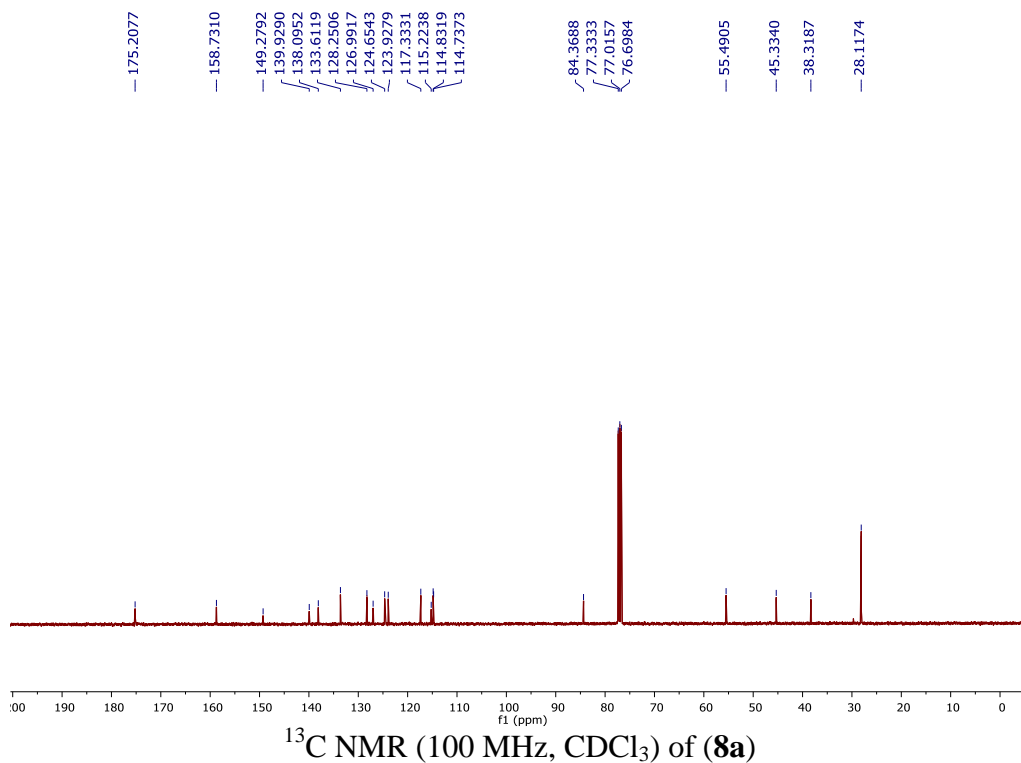
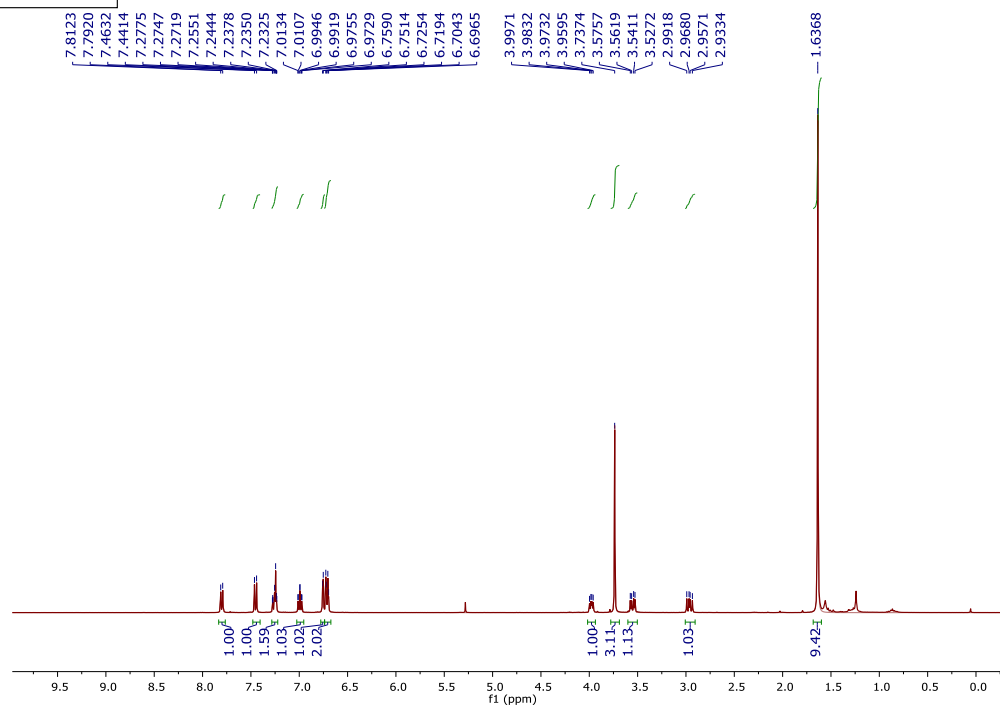
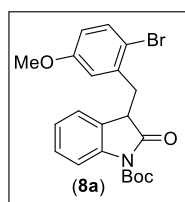
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 Method HRLCMS-20 Sept.m
 Sample Name Dr.A.Bisai-SB4-135
 Comment

Acquisition Date 4/12/2016 3:21:16 PM
 Operator DIMPLe
 Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





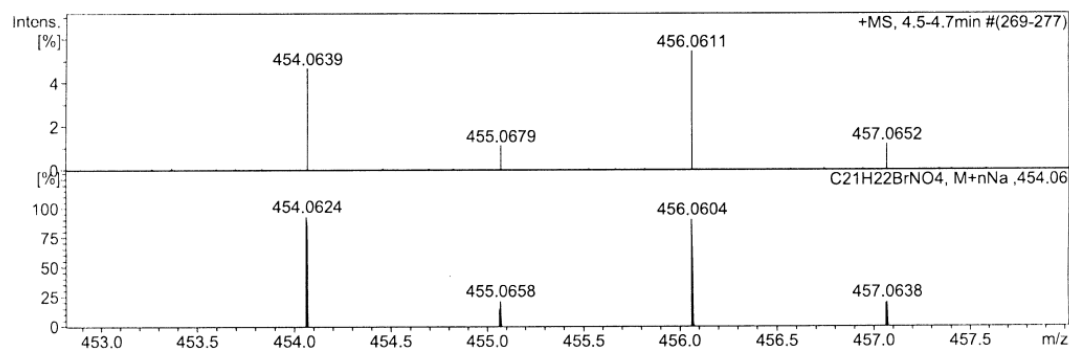
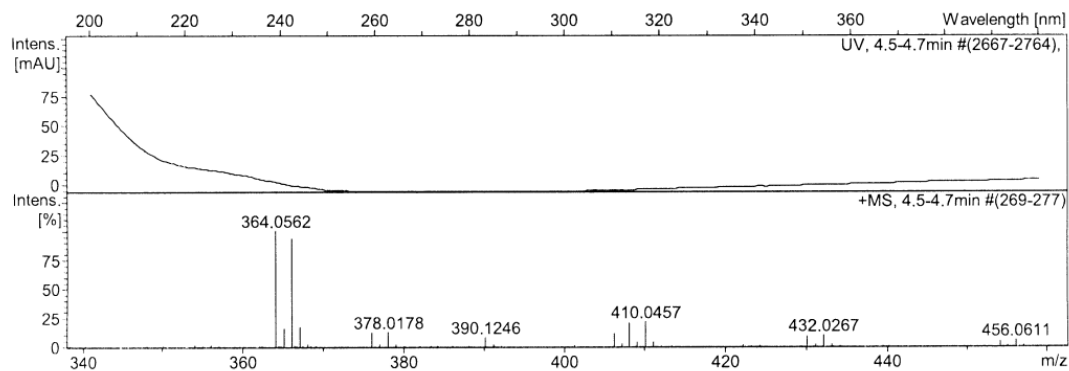
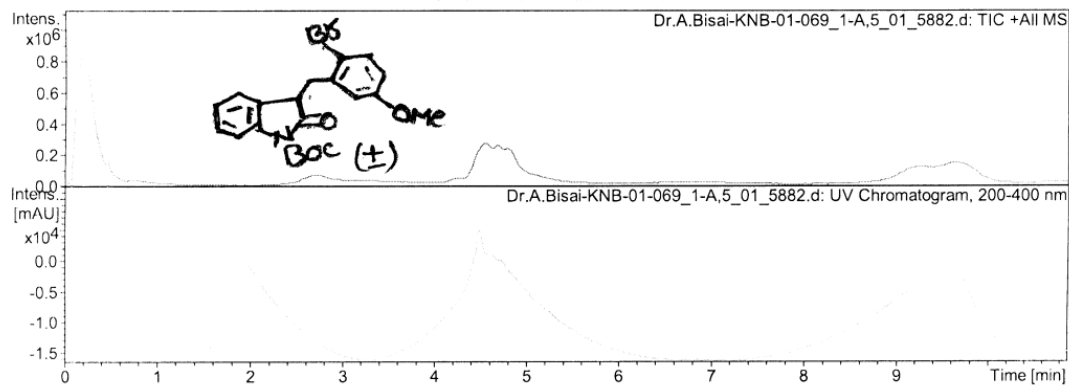
Display Report

Analysis Info

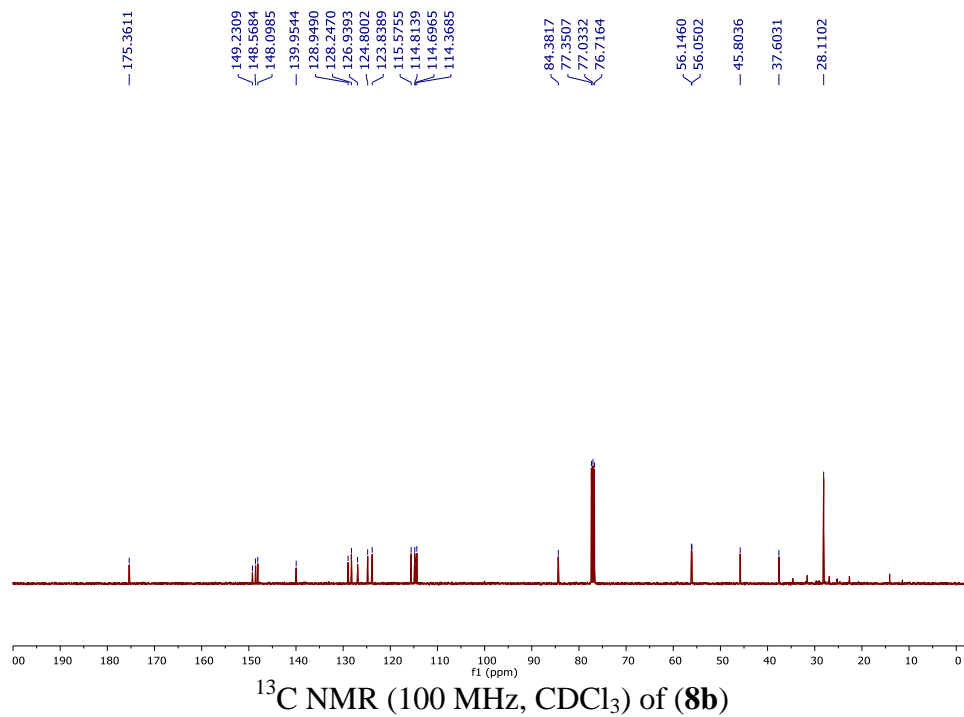
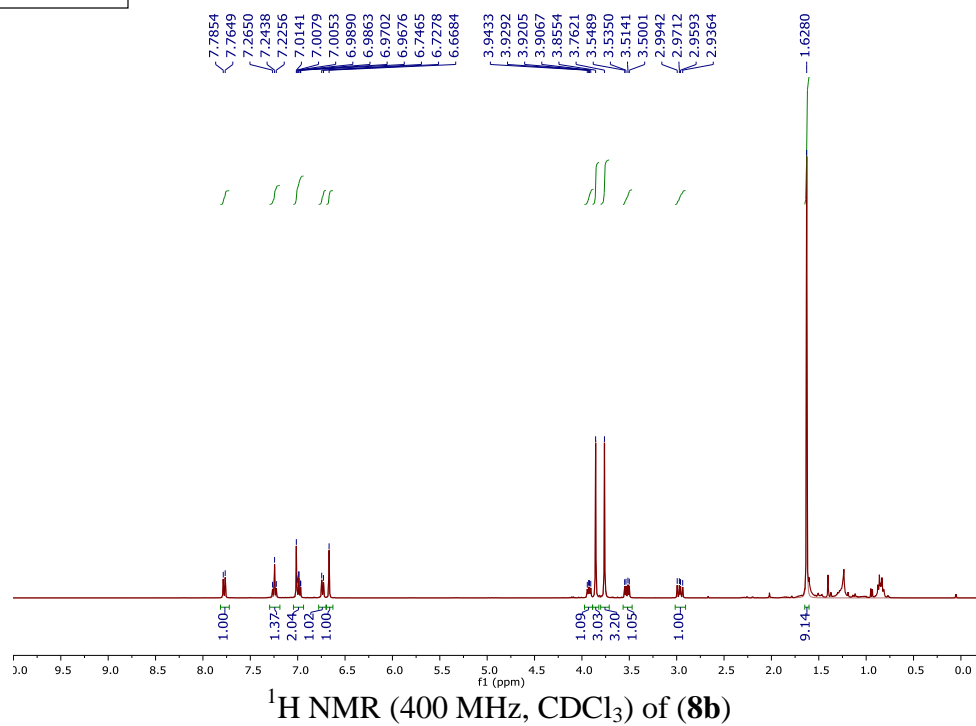
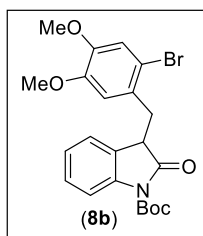
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Method HRLCMS-20 Sept tune wide.m Operator DIMPLE
Sample Name Dr.A.Bisai-KNB-01-069 Instrument microTOF-Q II 10330
Comment

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (8a)



Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\12-04-2016\Dr.A.Bisai-SB4-136_1-C,6_01_5848.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-SB4-136
 Comment

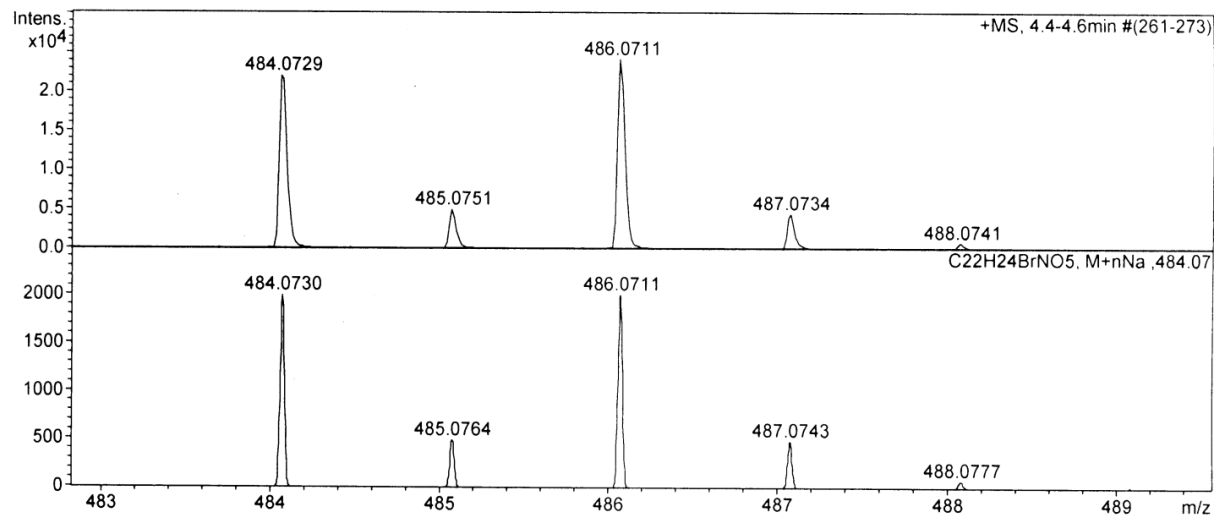
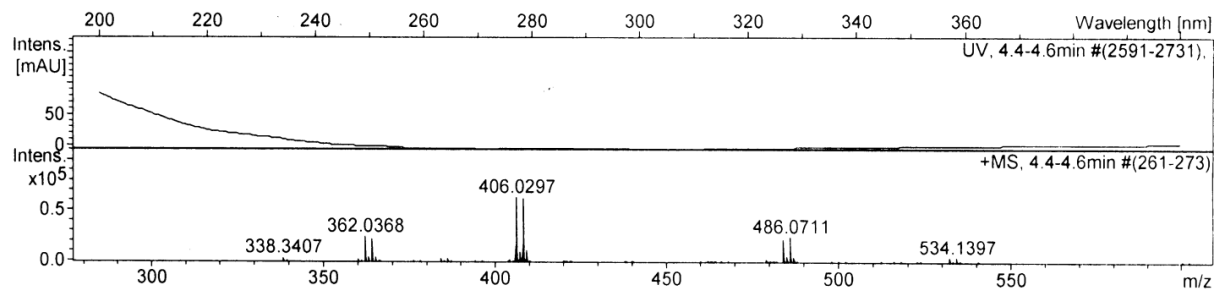
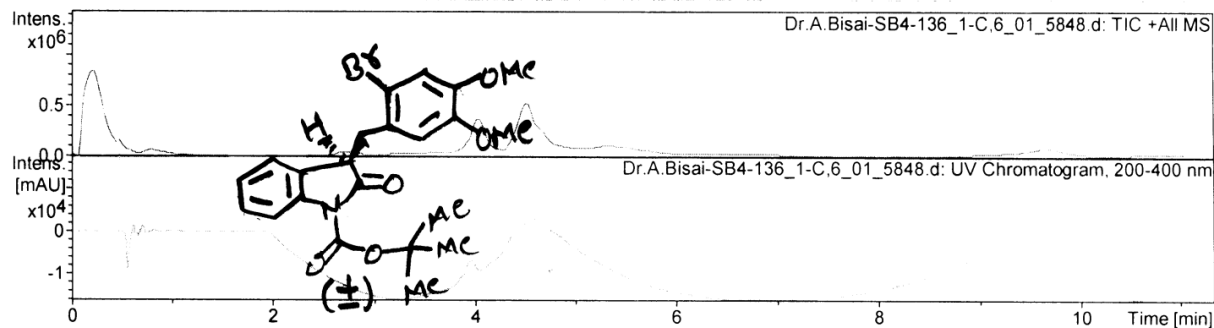
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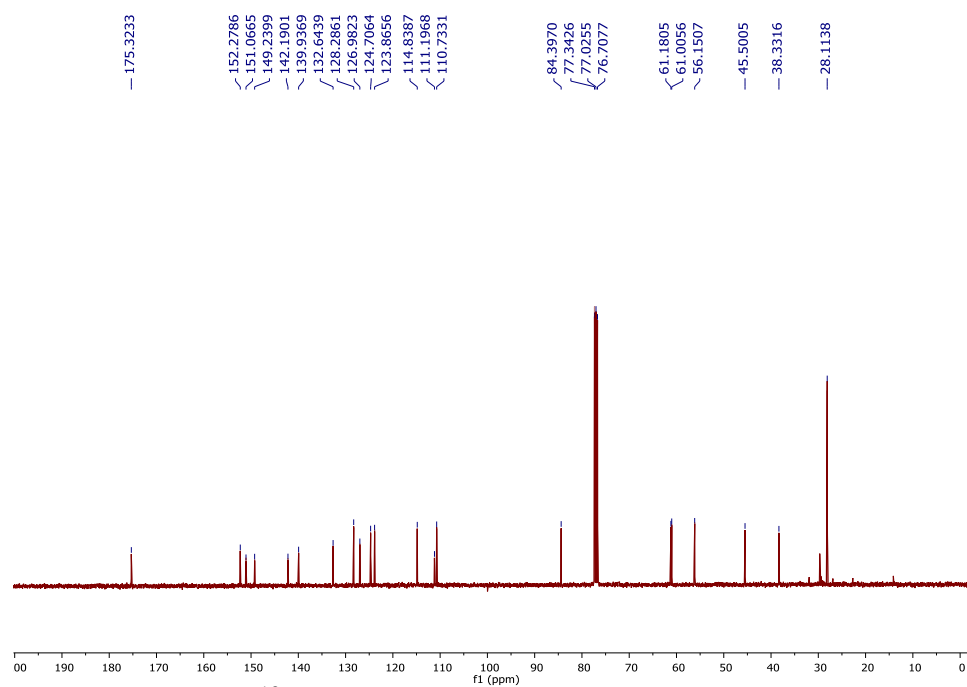
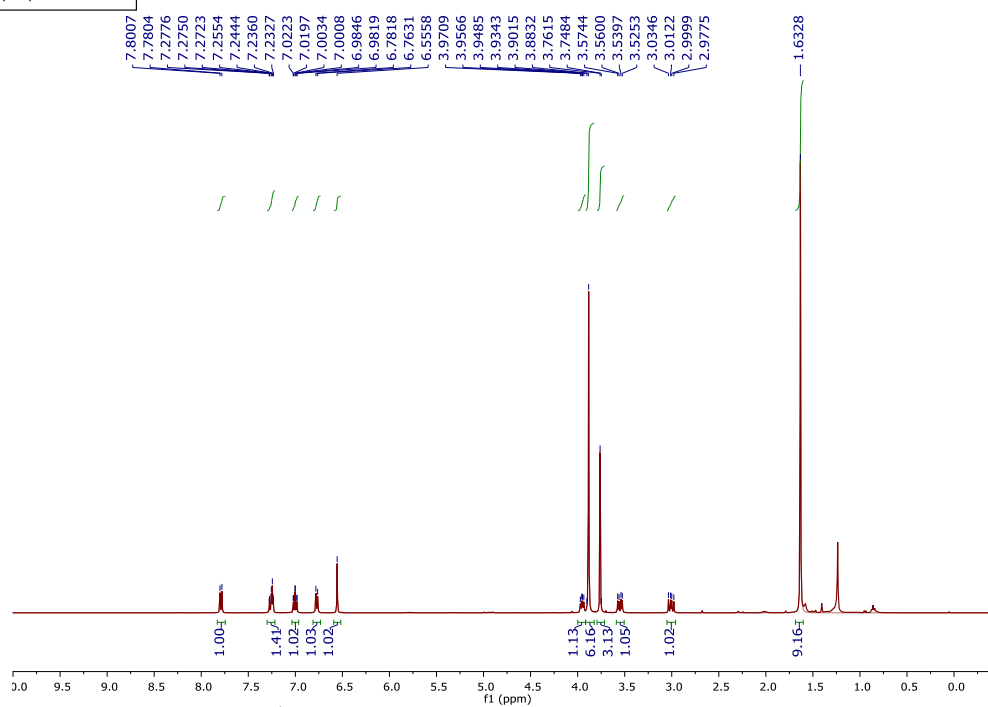
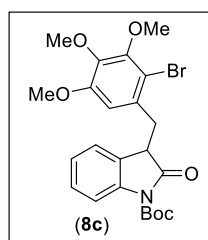
Operator DIMPLE

Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste





Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\13-04-2016\Dr.A.Bisai-KNB-01-067_1-A,7_01_5875.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-KNB-01-067
 Comment

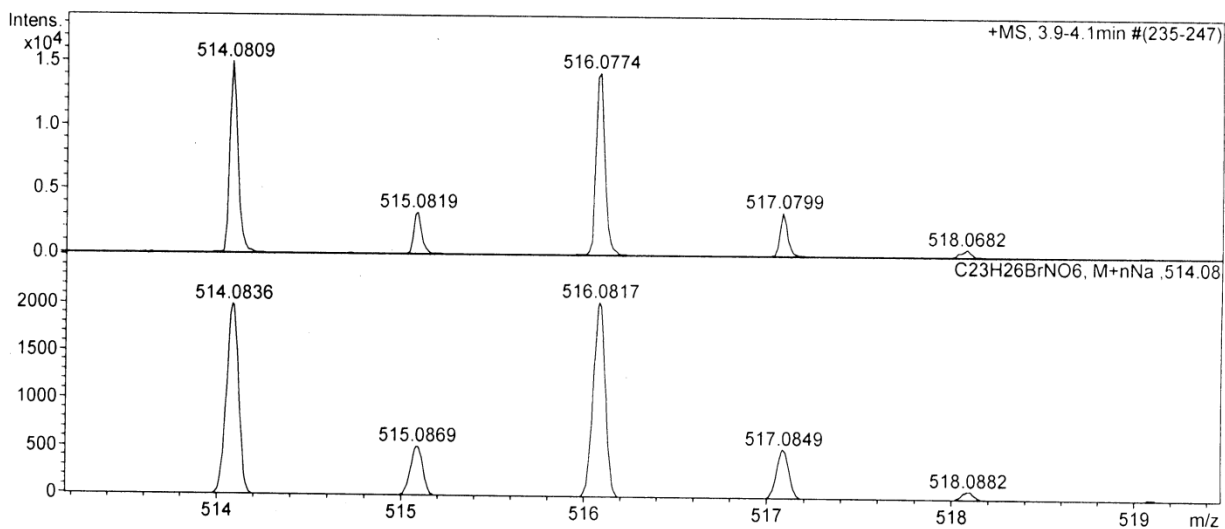
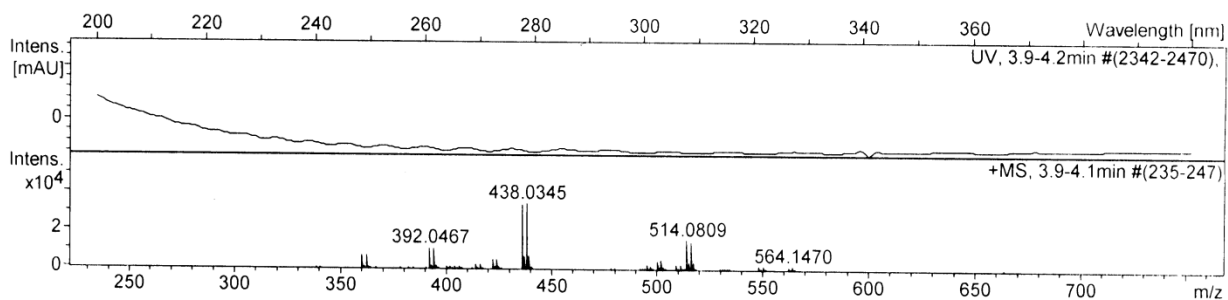
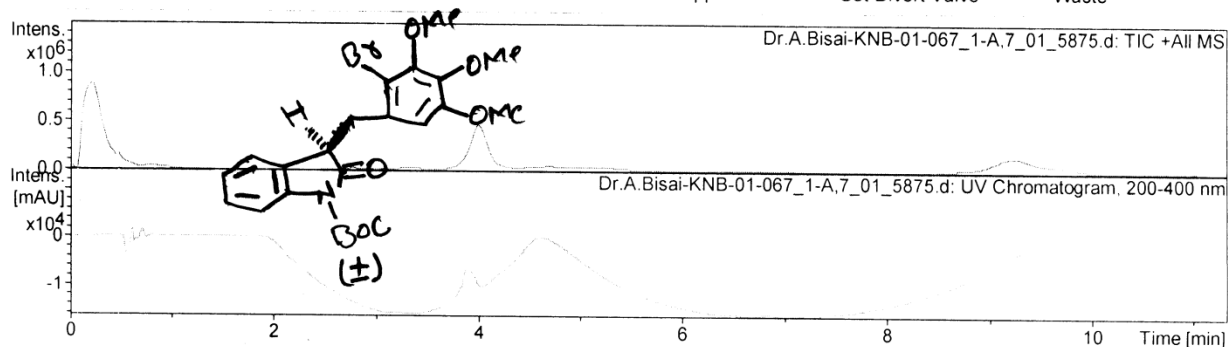
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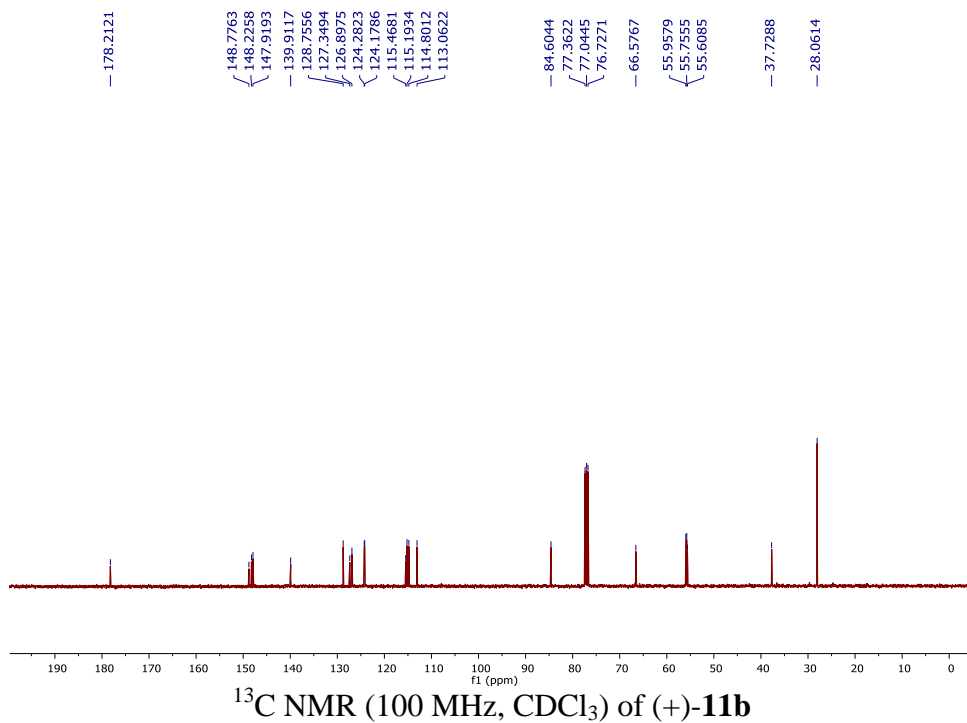
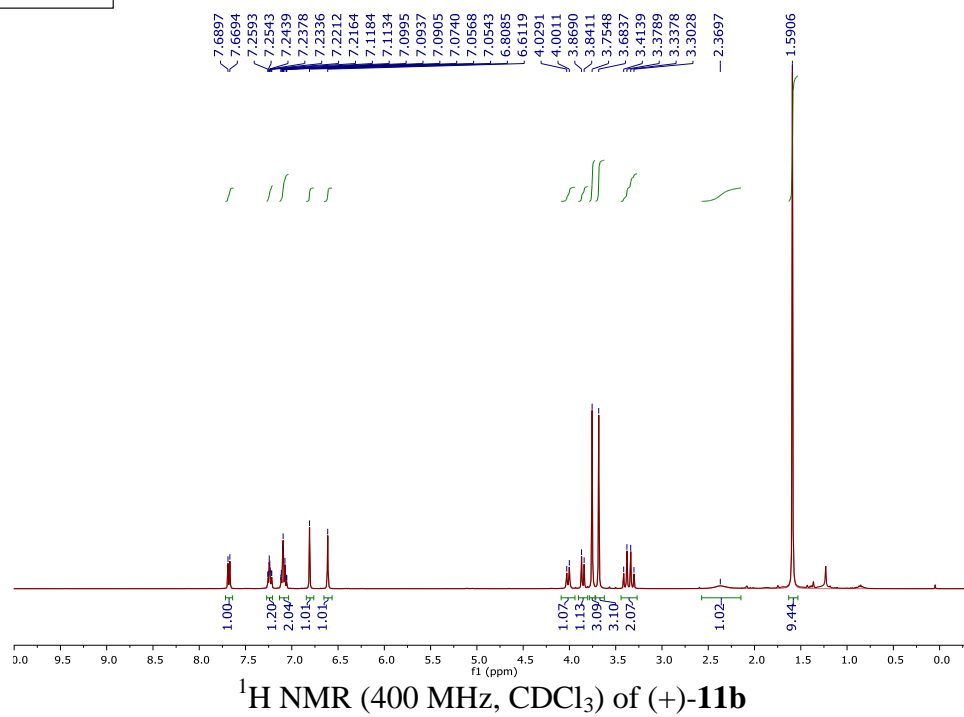
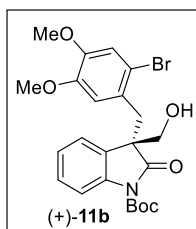
Operator DIMPLE

Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste





Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\13-04-2016\Dr.A.Bisai-KNB-01-066_1-A,6_01_5874.d
Method HRLCMS-20 Sept tune wide.m
Sample Name Dr.A.Bisai-KNB-01-066
Comment

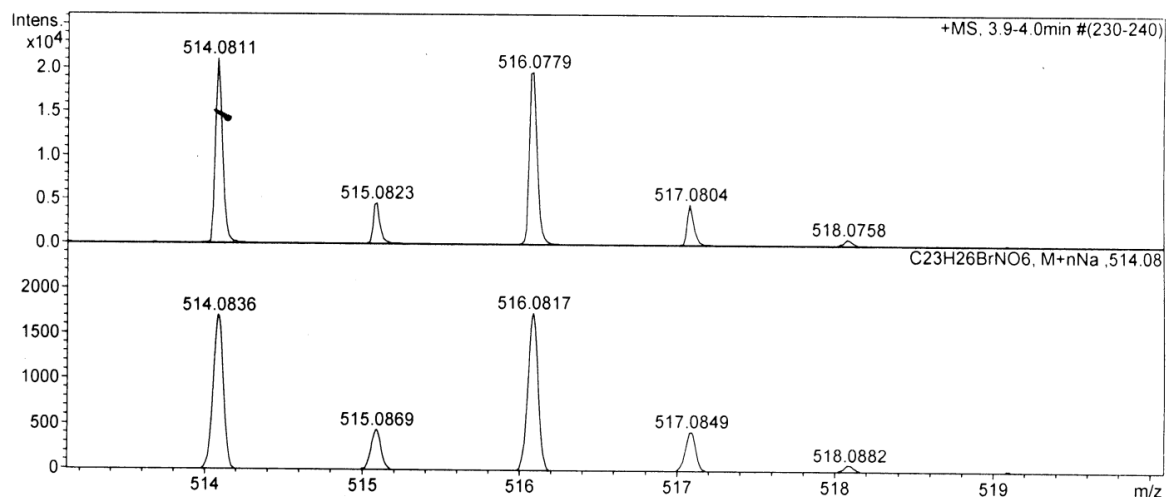
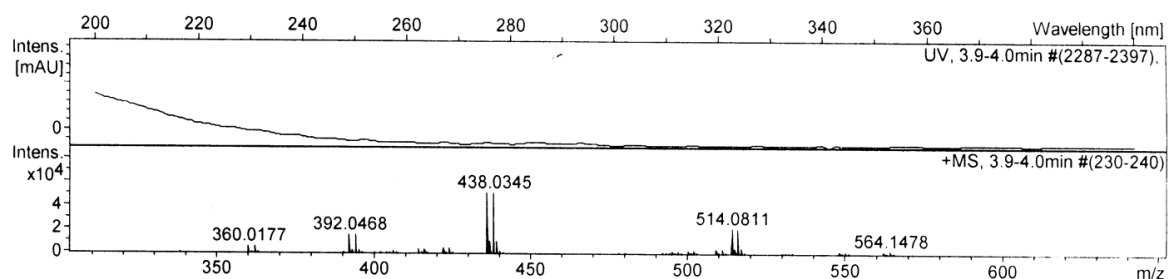
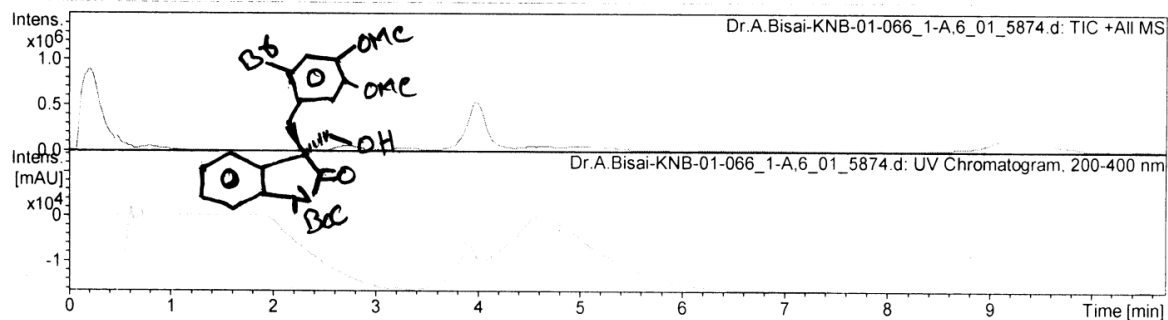
Acquisition Date 4/13/2016 2:05:53 PM

Operator DIMPLe

Instrument micrOTOF-Q II 10330

Acquisition Parameter

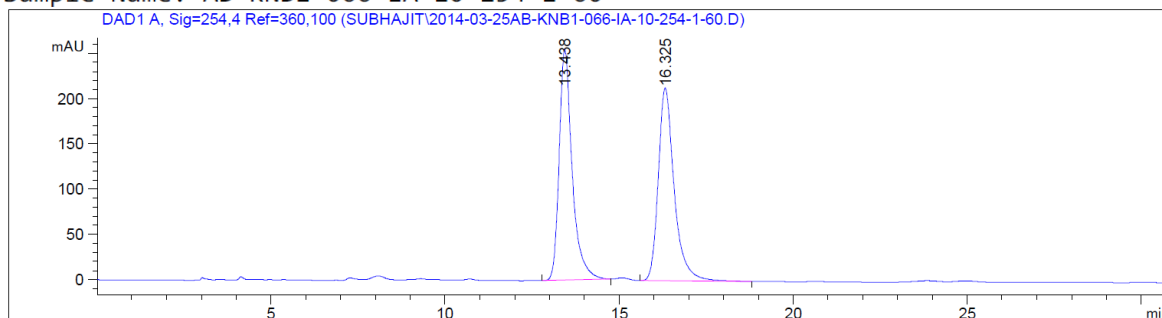
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



HPLC traces of compound (+)-11b:

Data File C:\CHEM32\1\DATA\SUBHAJIT\2014-03-25AB-KNB1-066-IA-10-254-1-60.D

Sample Name: AB-KNB1-066-IA-10-254-1-60



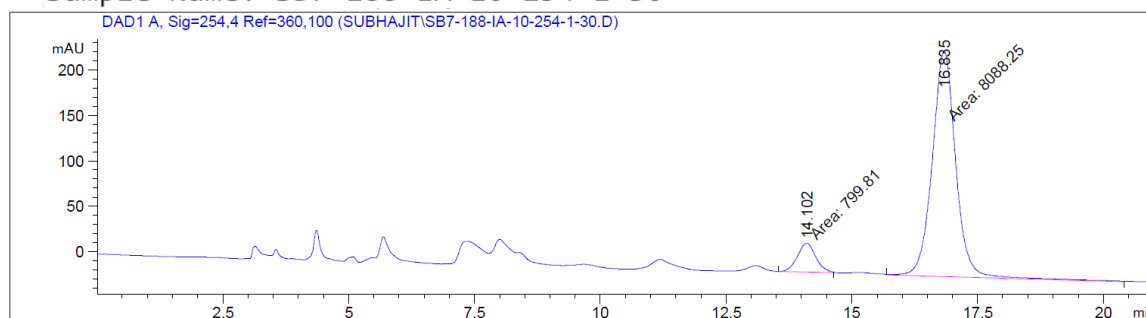
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.438	BB	0.3870	6626.07080	255.15839	49.4599
2	16.325	BB	0.4747	6770.77588	213.09705	50.5401

Totals : 1.33968e4 468.25543

Racemic

Data File C:\CHEM32\1\DATA\SUBHAJIT\SB7-188-IA-10-254-1-30.D

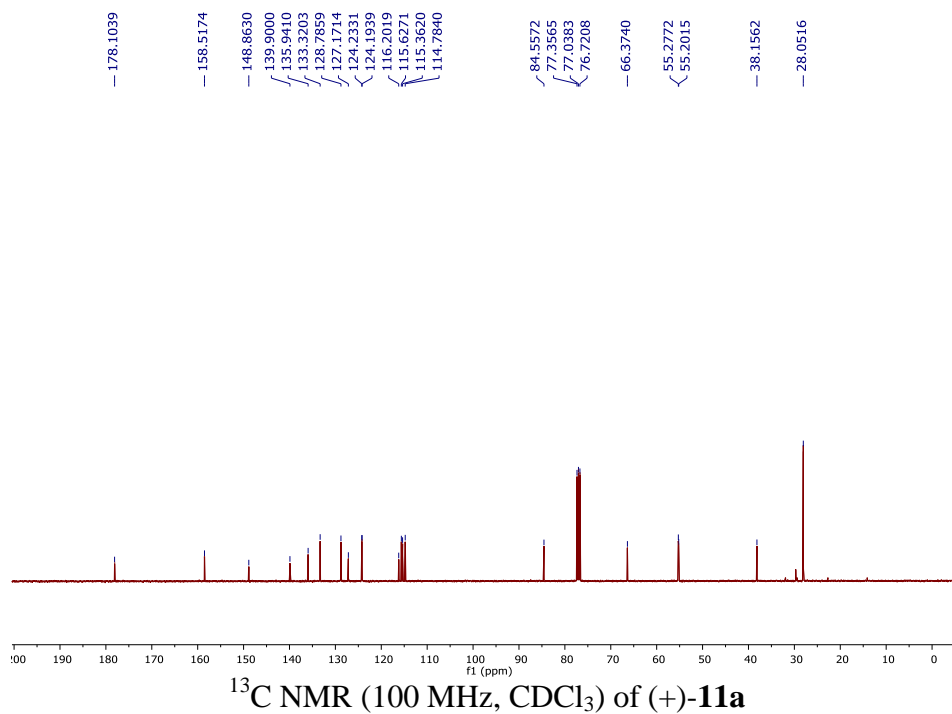
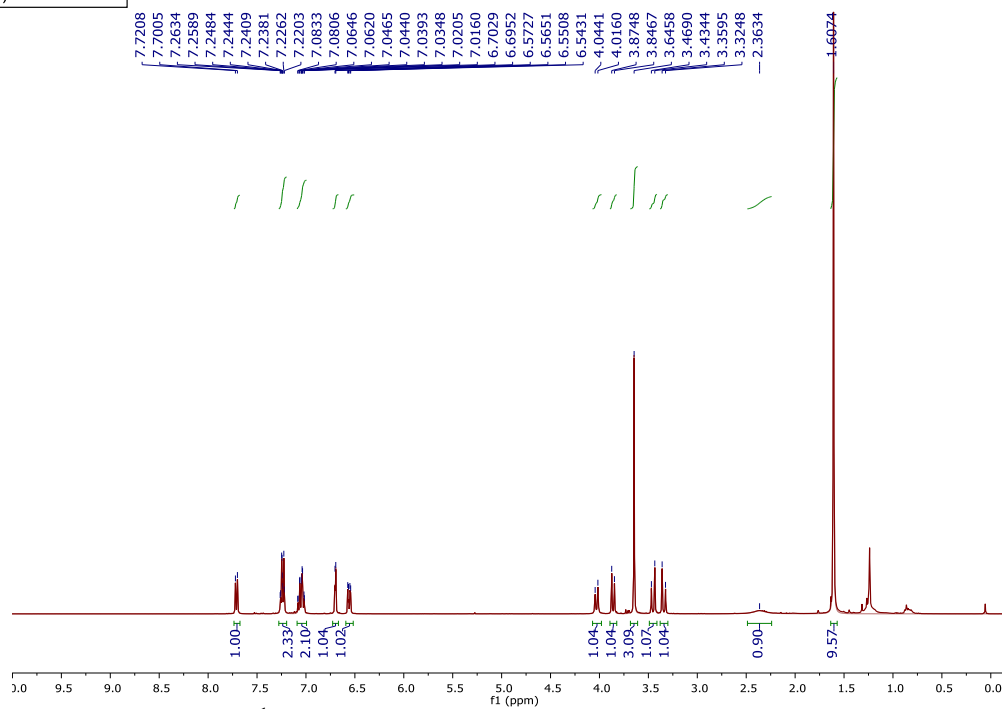
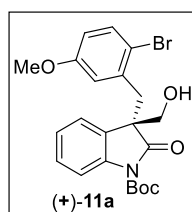
Sample Name: SB7-188-IA-10-254-1-30



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.102	MM	0.4203	799.80957	31.71591	8.9987
2	16.835	MM	0.5432	8088.24951	248.14471	91.0013

Totals : 8888.05908 279.86062

Enantiopure



Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\18-04-2016\Dr.A.Bisai-KNB-01-71_1-A,6_01_5883.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-KNB-01-71
 Comment

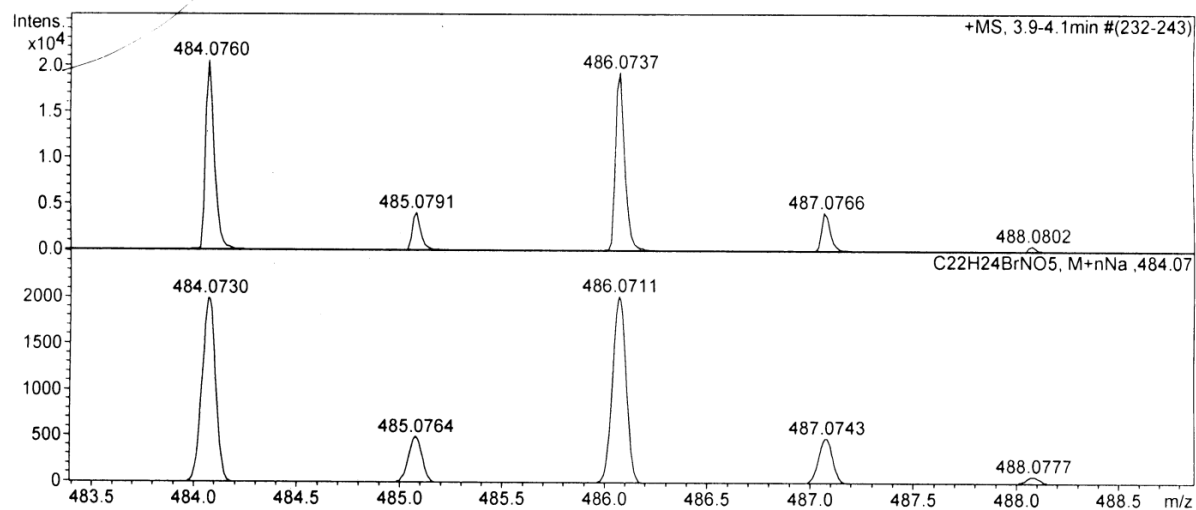
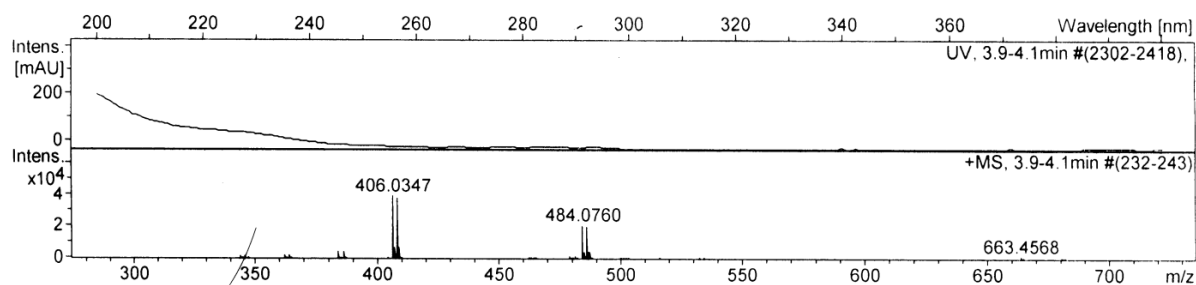
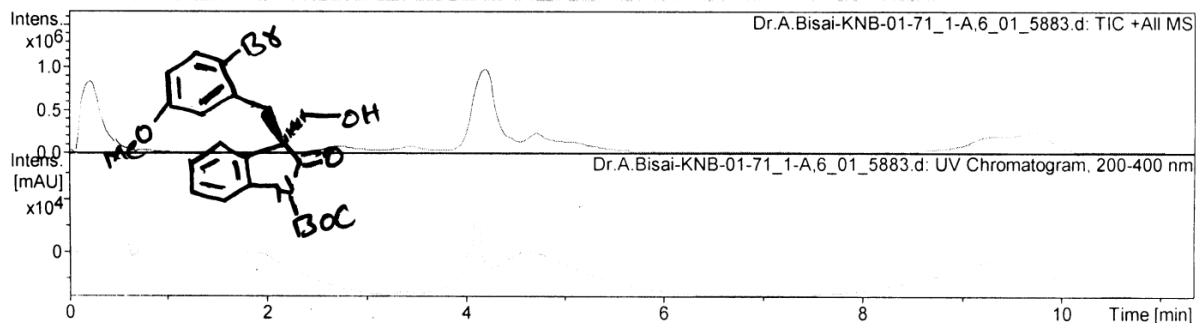
Acquisition Date 4/18/2016 1:36:58 PM

Operator DIMPLE

Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0

printed: 4/18/2016 3:41:22 PM

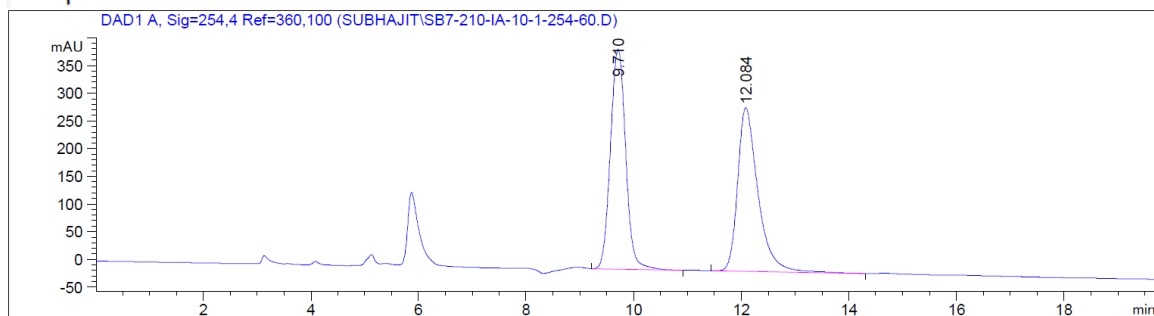
Page 1 of 1

Scanned copy of mass spectrum of (+)-11a

HPLC traces of compound (+)-11a:

Data File C:\CHEM32\1\DATA\SUBHAJIT\SB7-210-IA-10-1-254-60.D

Sample Name: SB7-210-IA-10-1-254-60



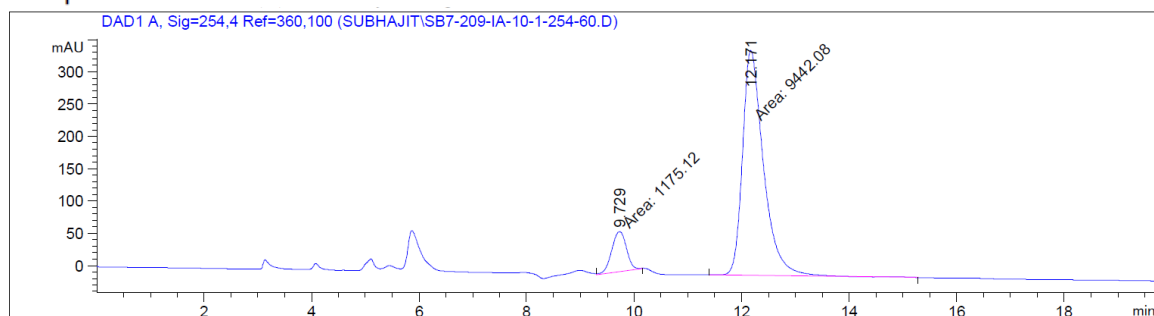
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.710	BB	0.3107	7849.70459	396.89224	50.2048
2	12.084	BB	0.3917	7785.66992	295.22726	49.7952

Totals : 1.56354e4 692.11951

Racemic

Data File C:\CHEM32\1\DATA\SUBHAJIT\SB7-209-IA-10-1-254-60.D

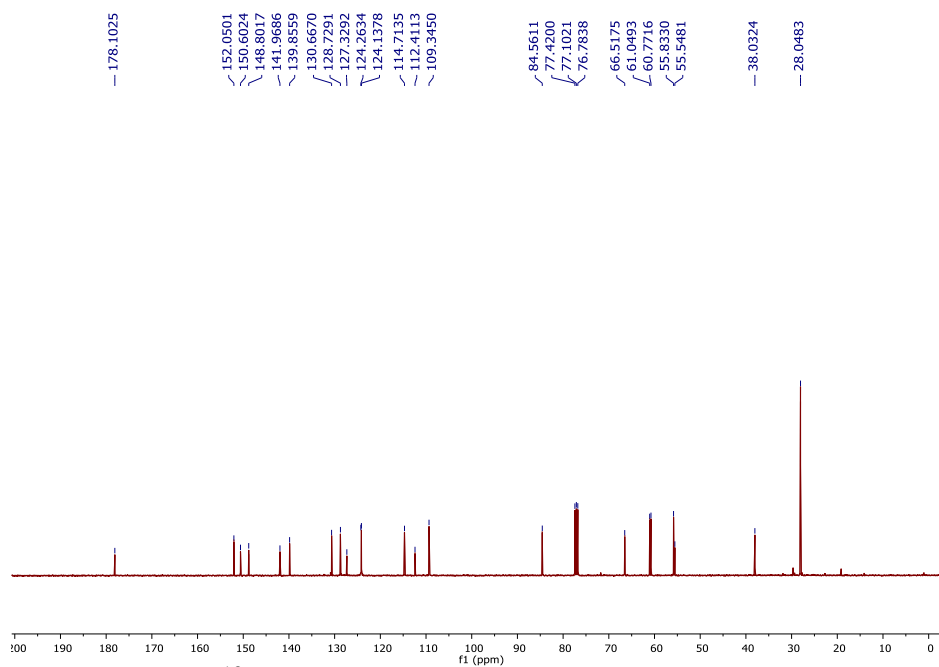
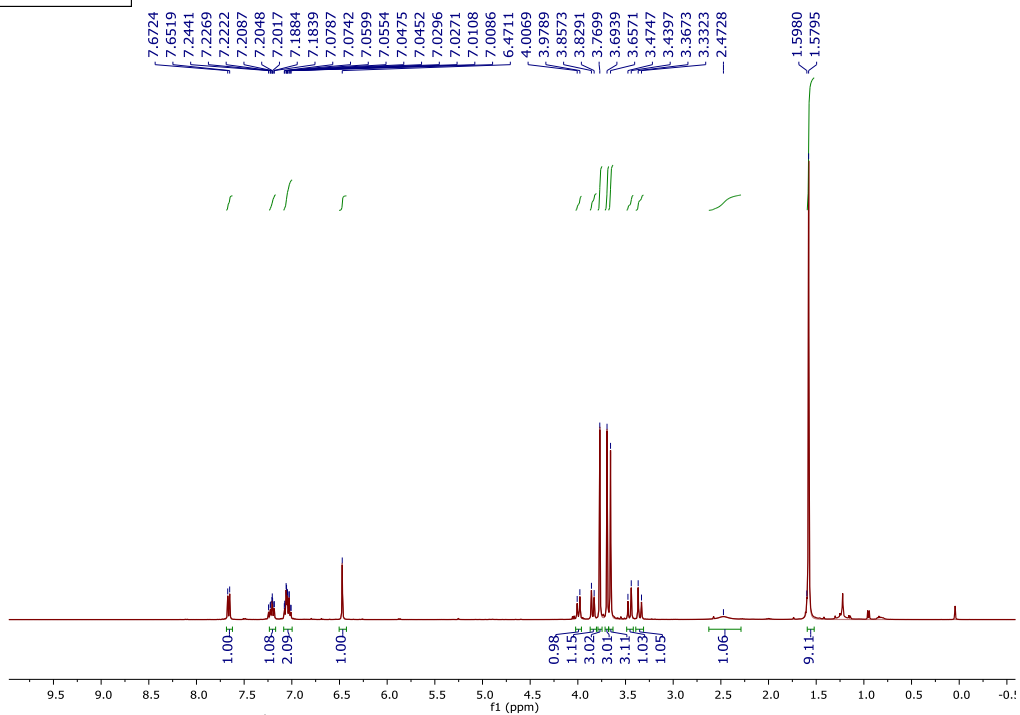
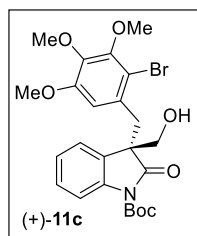
Sample Name: SB7-209-IA-10-1-254-60



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.729	MM	0.3159	1175.11914	62.00706	11.0681
2	12.171	MM	0.4521	9442.07715	348.08295	88.9319

Totals : 1.06172e4 410.09001

Enantiopure



Display Report

Analysis Info

Analysis Name D:\Data\user data\2016\April 2016\18-04-2016\Dr.A.Bisai-KNB-01-068_1-A,4_01_5880.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-KNB-01-068
 Comment

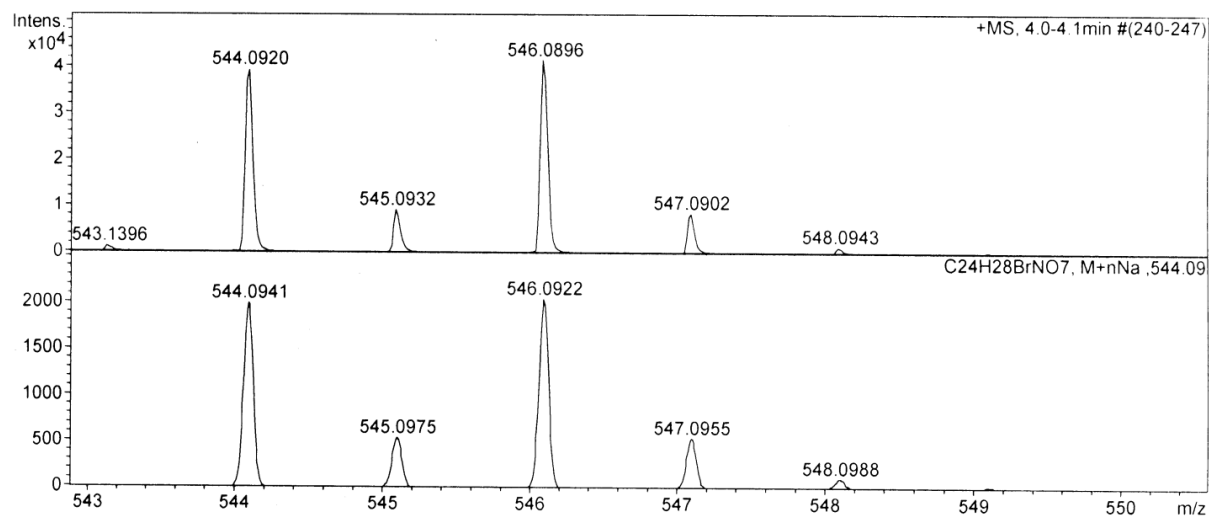
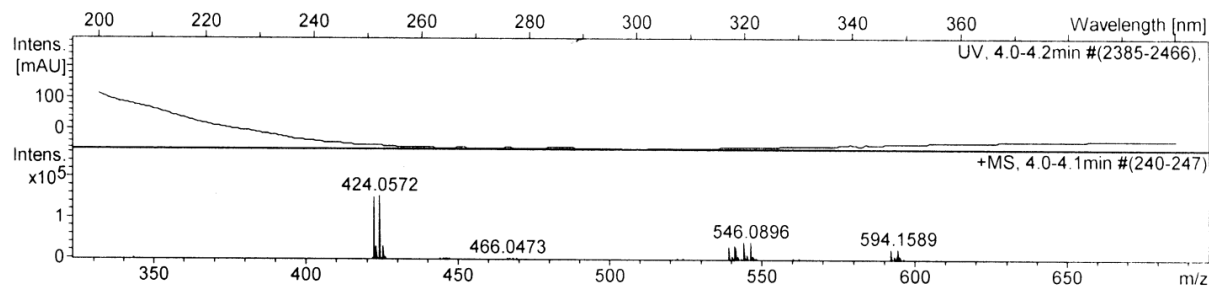
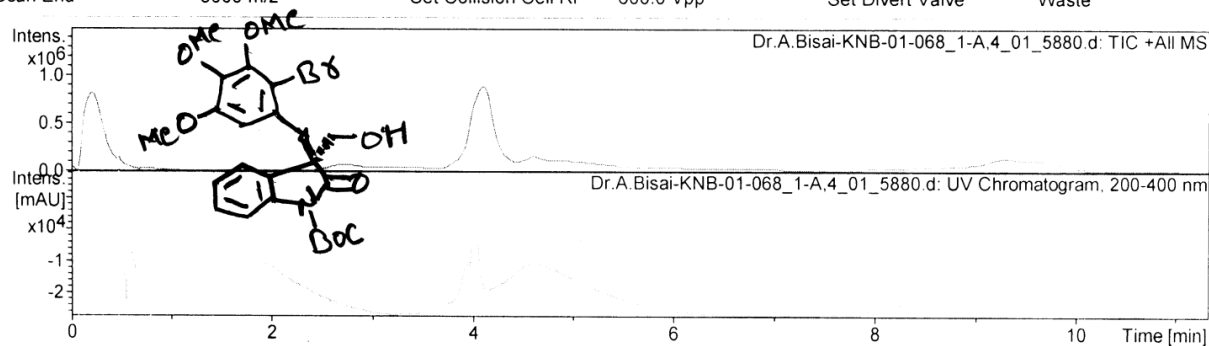
Acquisition Date 4/18/2016 1:00:28 PM

Operator DIMPLE

Instrument micrOTOF-Q II 10330

Acquisition Parameter

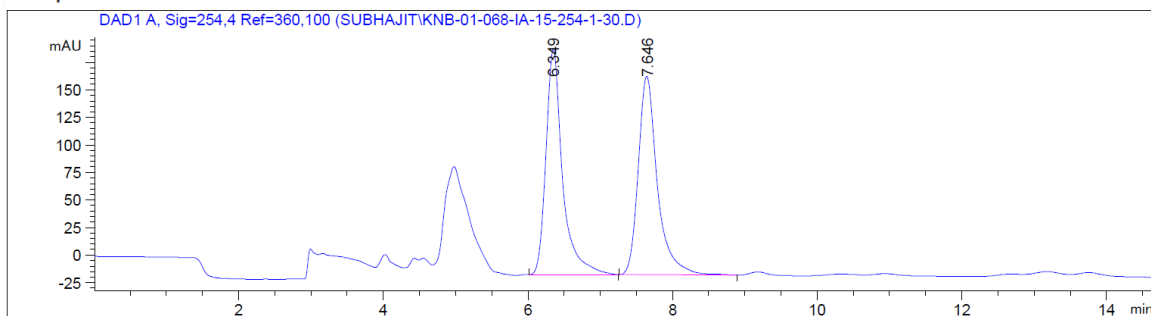
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



HPLC traces of compound (+)-11c:

Data File C:\CHEM32\1\DATA\SUBHAJIT\KNB-01-068-IA-15-254-1-30.D

Sample Name: KNB-01-068-IA-15-254-1-30



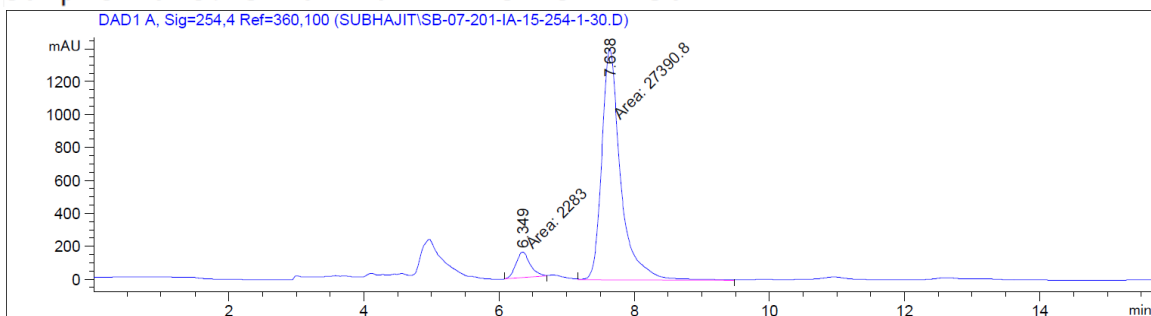
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.349	BB	0.2381	3264.98999	204.68718	49.6223
2	7.646	BB	0.2748	3314.69849	180.06126	50.3777

Totals : 6579.68848 384.74844

Racemic

Data File C:\CHEM32\1\DATA\SUBHAJIT\SB-07-201-IA-15-254-1-30.D

Sample Name: SB-07-201-IA-15-254-1-30



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.349	MM	0.2436	2282.99658	156.16652	7.6936
2	7.638	MM	0.3253	2.73908e4	1403.34412	92.3064

Totals : 2.96738e4 1559.51064

Enantiopure