

Supplementary Material

An efficient total synthesis of vilanterol: an inhaled drug

Kamalaker Reddy Kamireddy,^a Sailam Sri Gogula,^a Venkateswarlu Banoji,^a Sayaji Rao,^a and Balakishan Gorre^{b*}

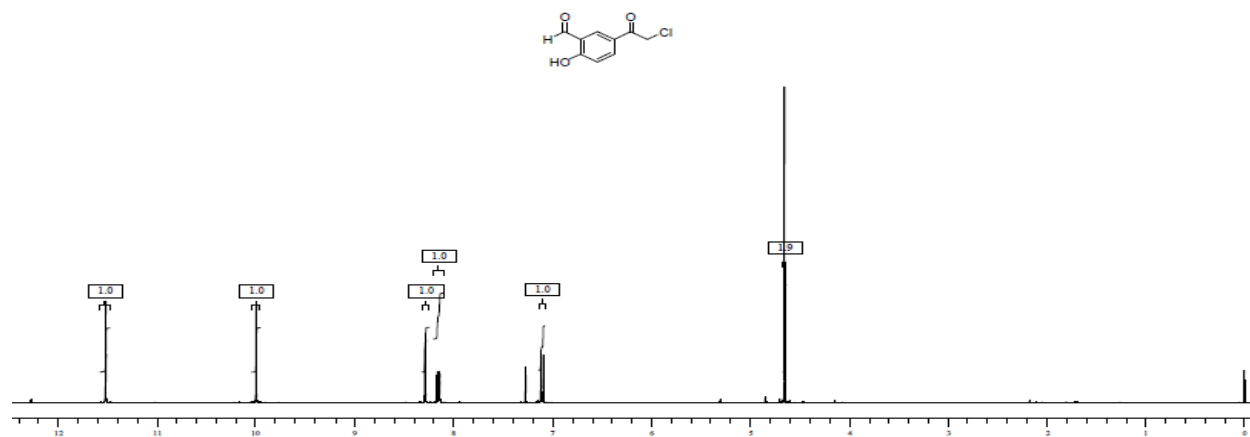
^aUniversity College of Science, Osmania University, Hyderabad, India-500007

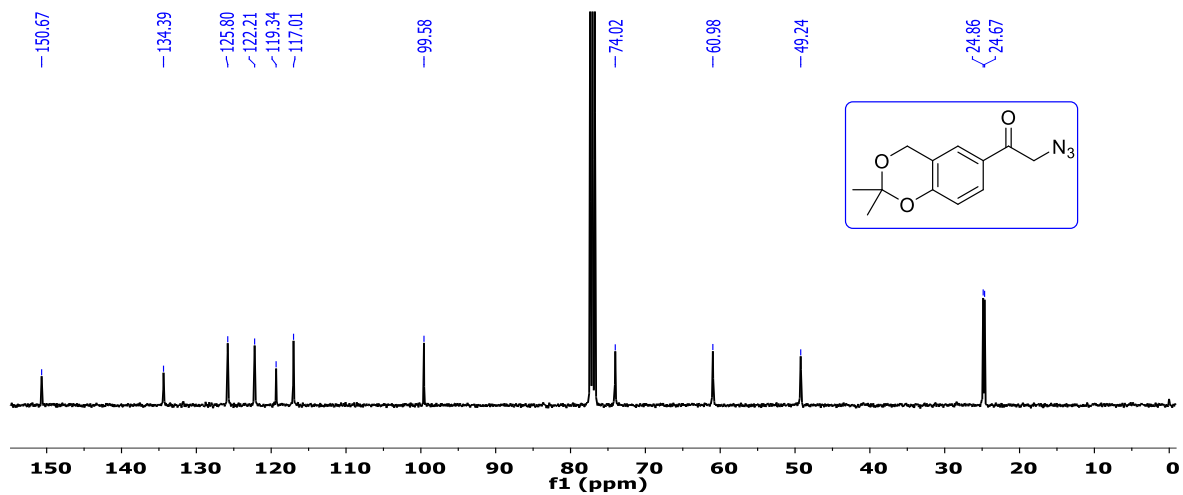
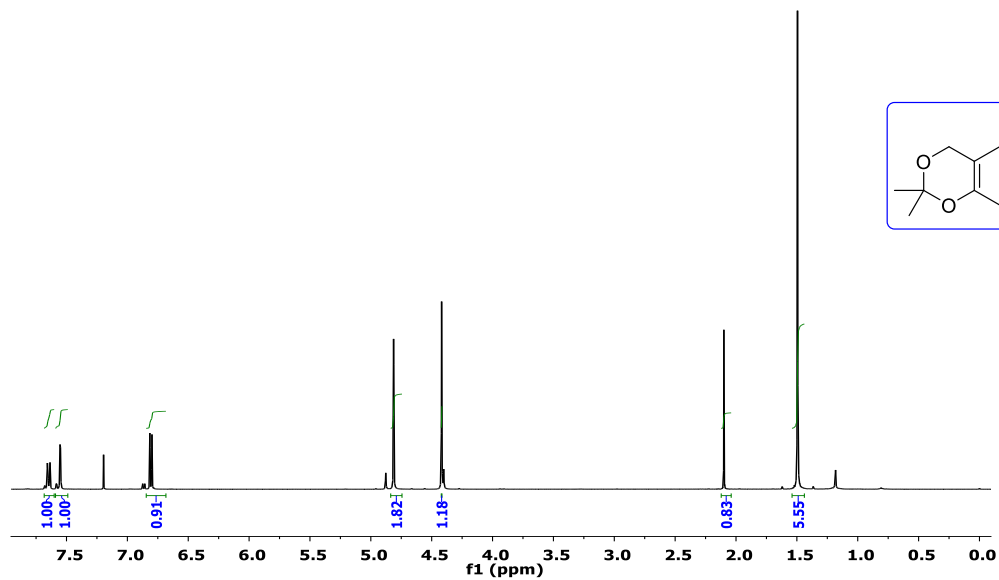
^bDepartment of Chemistry, University College of Science, Telangana University, Telangana, India-503322

Email: gbk@telanganauniversity.ac.in

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^1H (300 MHz, CDCl_3) and ^{13}C (75 MHz, CDCl_3) NMR spectra of compound 5

^1H (500 MHz, CDCl_3) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 6

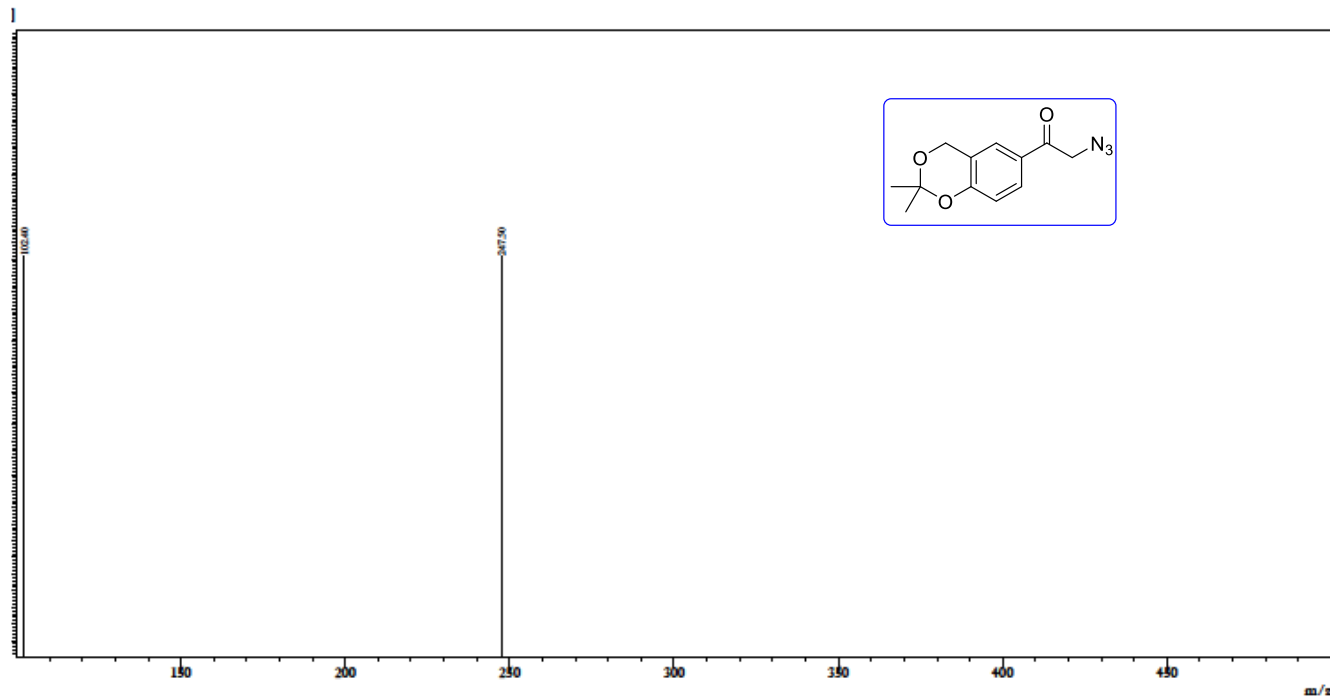
Mass (ESI) of compound 6

OSPC, Dr RATHOD

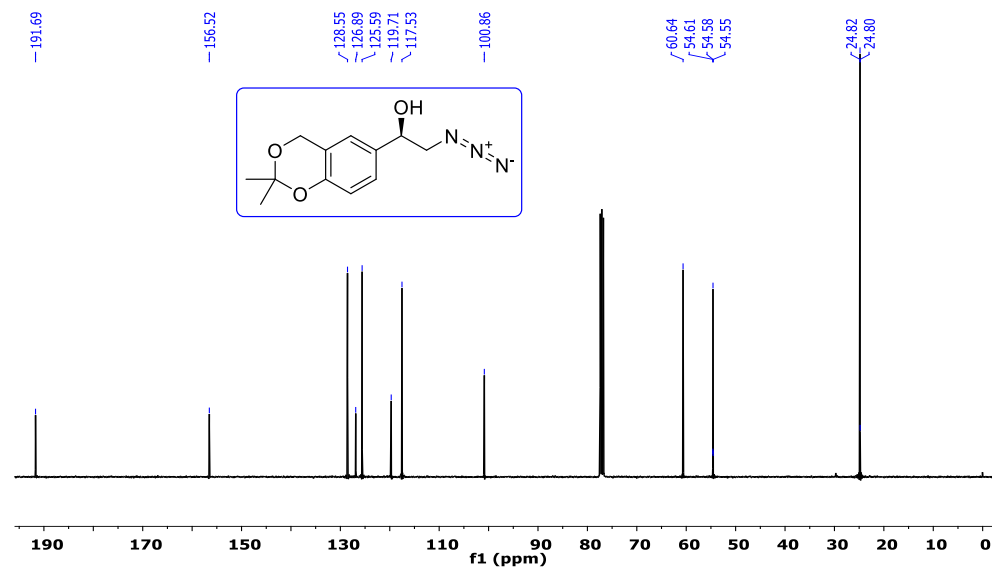
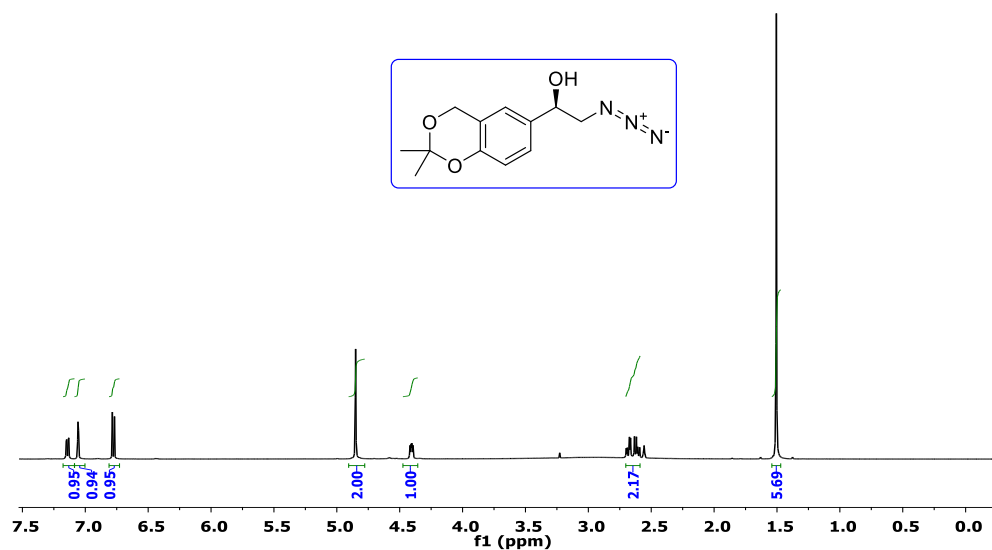
SHIMADZU
LabSolutions

MASS REPORT

Sample Name : PSH-247-6
File : 24052023.28.lcd
Acquired : 5/24/2023 4:21:13 PM
In File : 24052023_532023_1055 AM.lcb

MS Spectrum
D:\DATA\MAY2023\24052023.28.lcdESI Positive
Scan Mode: Single 0.220(S1)

1

^1H (500 MHz, DMSO) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 7

Mass (ESI) of compound 7

OSPC, Dr. RATHOD

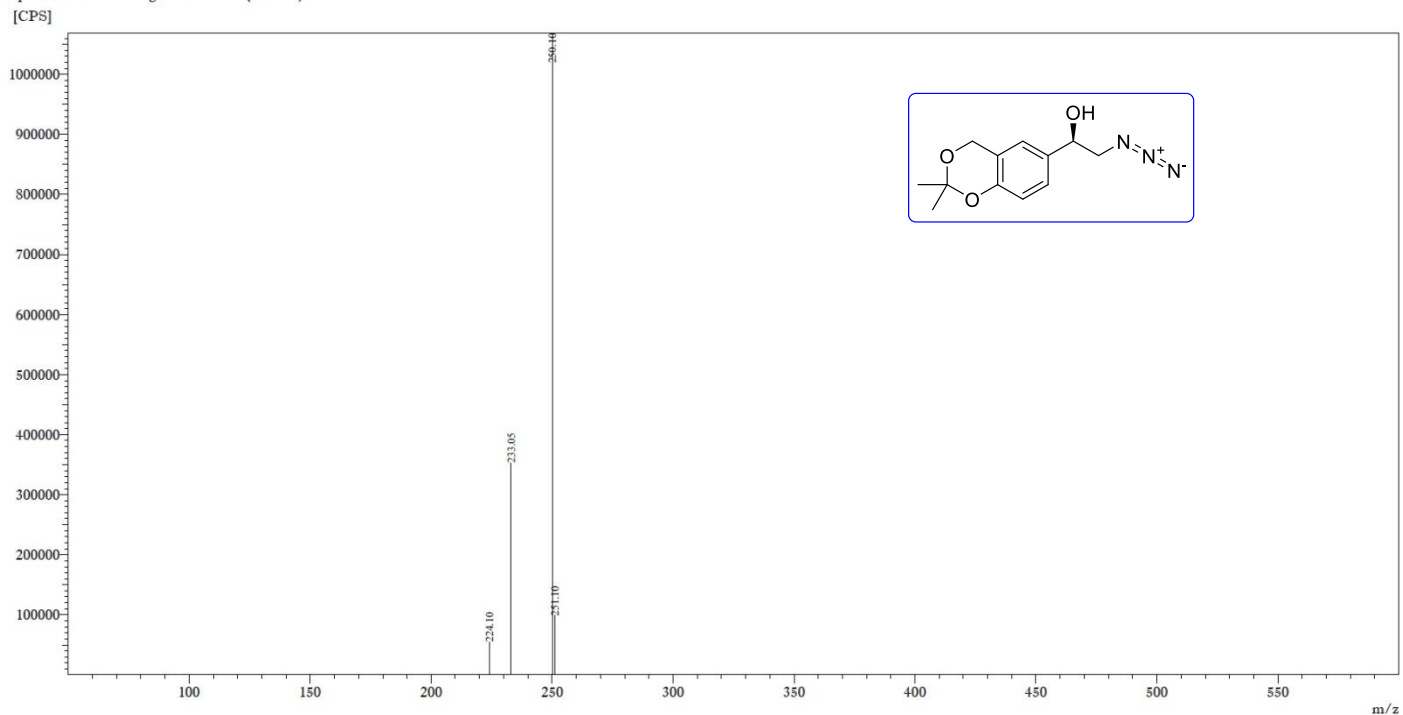


MASS REPORT

Sample Name : HUS-9
Data File : 13032023.32.lcd
Date Acquired : 5/23/2023 11:44:21 AM
Batch File : 12032021-1.lcb

Averaged ESI Positive+
Spectrum Mode: Averaged 0.443-0.859(191-369)

MS Spectrum
D:\DATA\March 2021\12032021.32.lcd

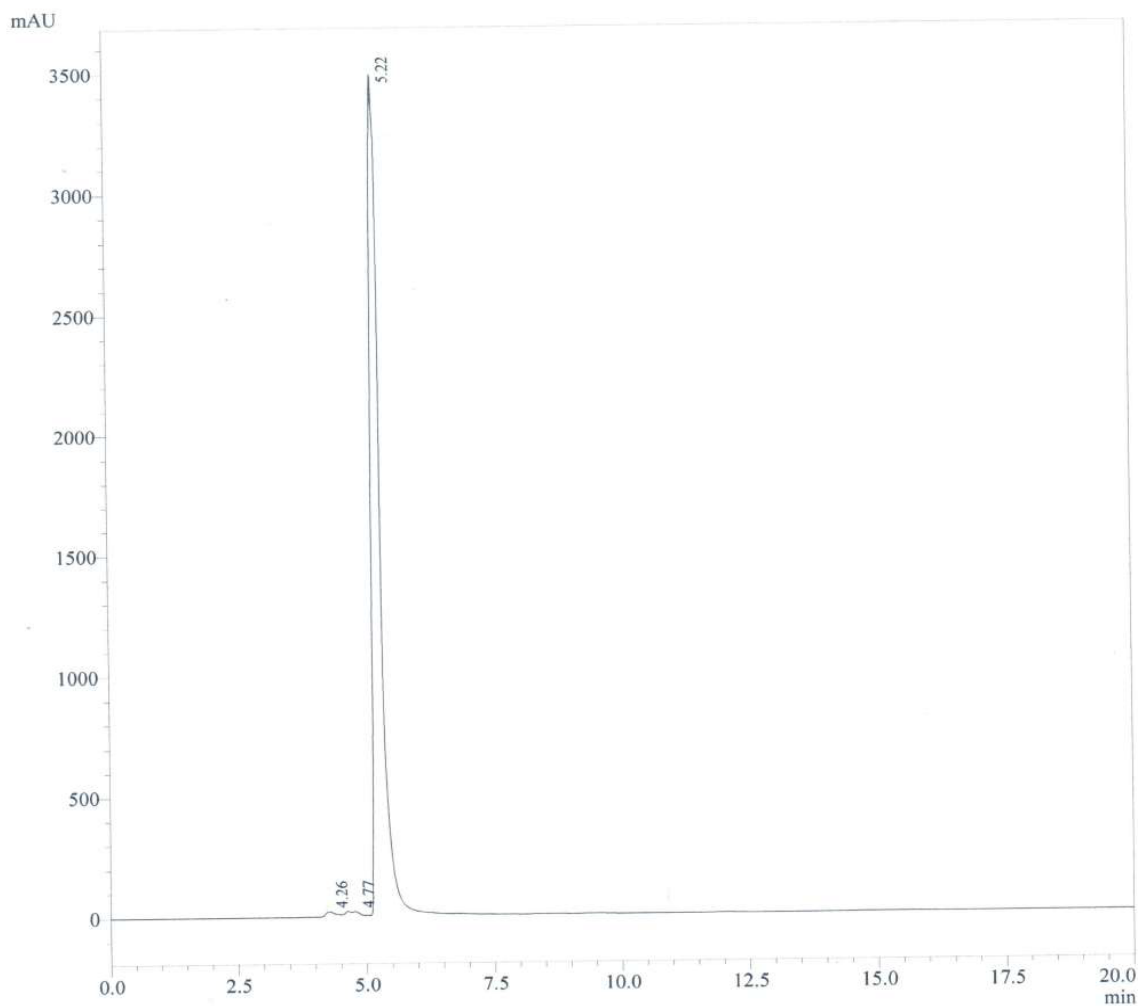


HPLC of compound 7 on IG5 CHIRALPAK column with a Hex/ IPA ratio of 50/50

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
CROP PROTECTION CHEMICALS DIVISION

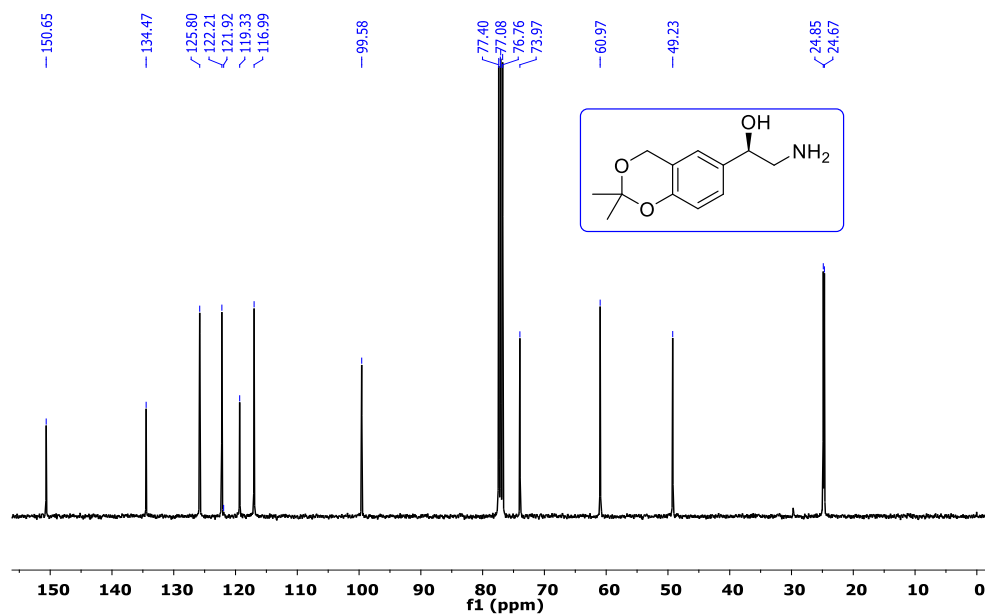
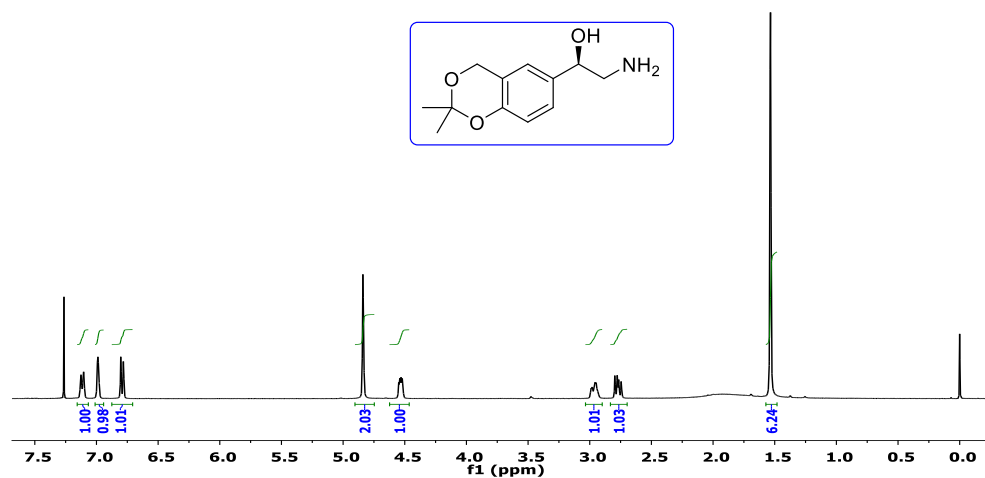
Acquired by : T RAMESH BABU
Sample Name : BVSR
Sample ID : AZIDE
Data Filename : KRED-290.lcd
Method Filename : Deepthi.lcm
Date Acquired : 16/06/2023

D:\BVSR\KRED-290.lcd



PDA Ch1 200nm - 500nm 2nm

Peak#	Ret. Time	Area	Area %
1	4.26	272951	0.67
2	4.77	196199	0.48
3	5.22	40556978	98.86
Total		41026128	100.00

^1H (400 MHz, CDCl_3) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 8

Mass (ESI) of compound 8

OSPC, Dr RATHOD

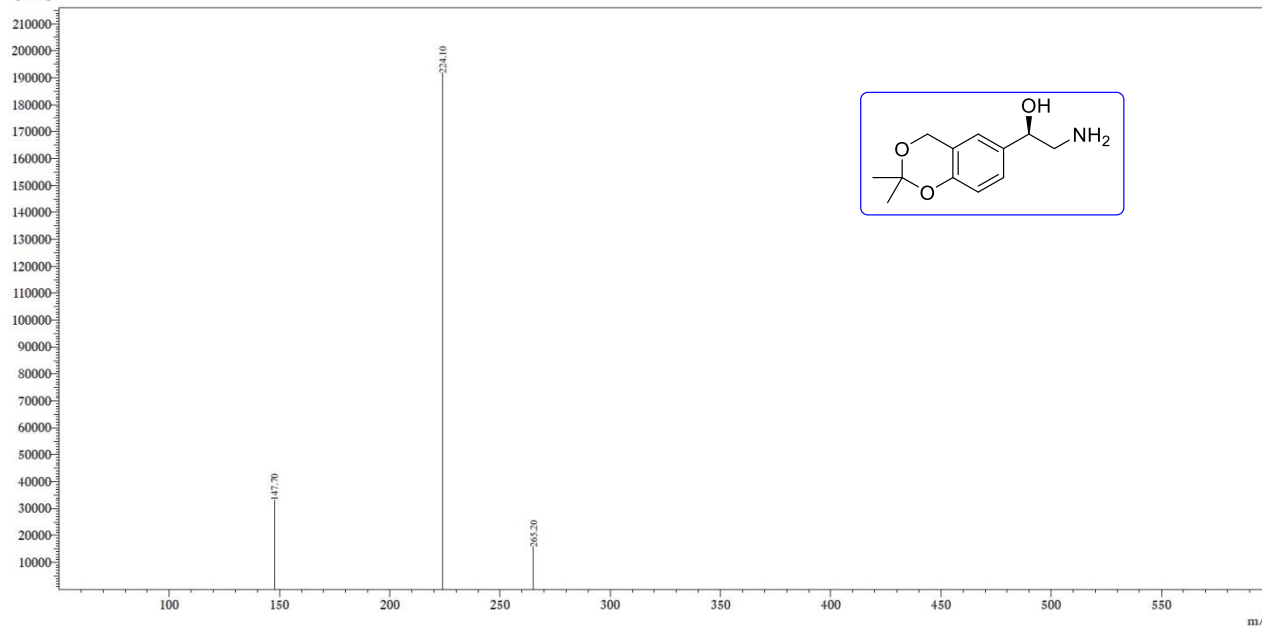
SHIMADZU
LabSolutions

MASS REPORT

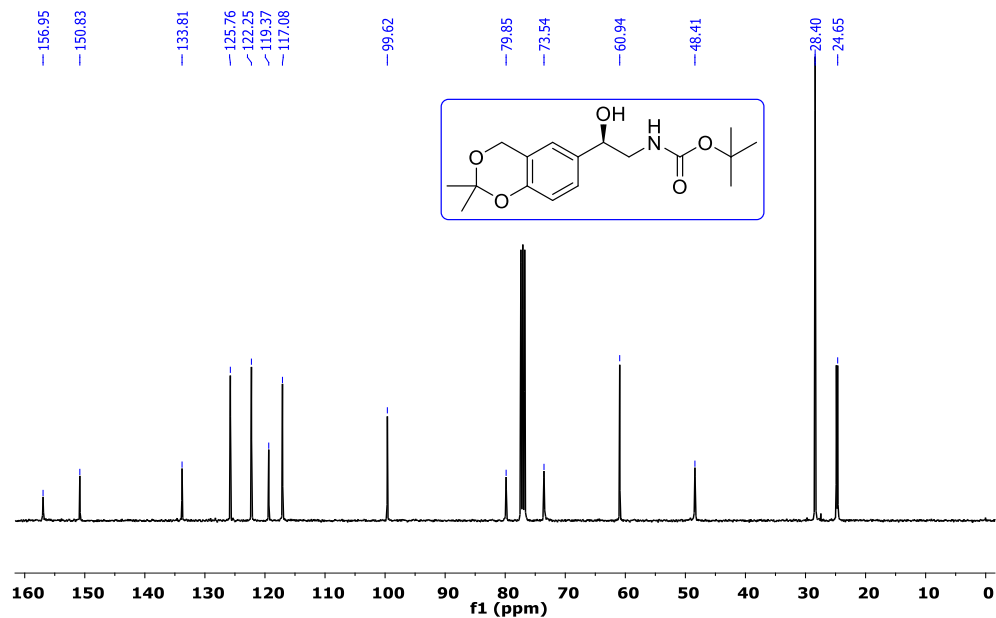
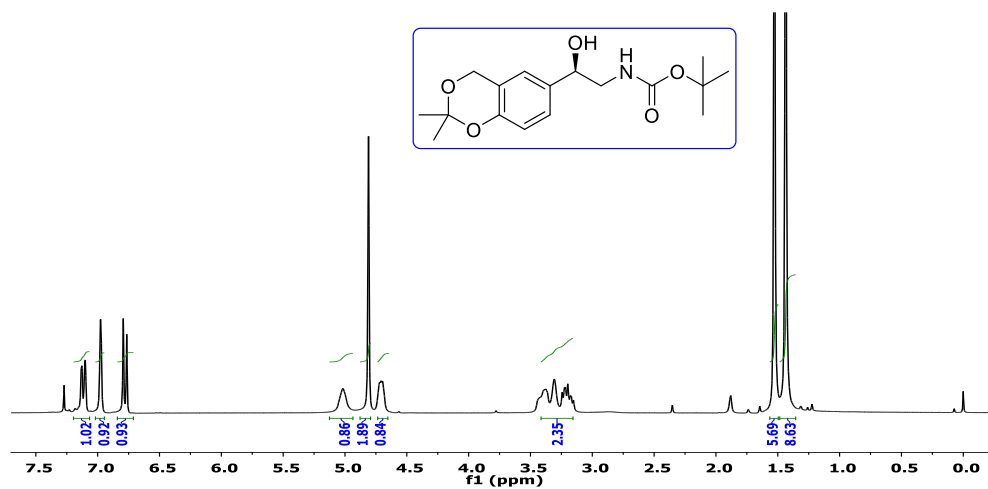
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Date Acquired : 5/24/2023 4:26:32 PM
Batch File : 24052023 532023 1055 AM.lcb

MS Spectrum
D:\DATA\MAY2023\24052023.30.lcd

SingleESI Positive+
Spectrum Mode:Single 0.330(121)
[CPS]



1

^1H (300 MHz, CDCl_3) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 9

Mass (ESI) of compound 9

OSPC, Dr RATHOD

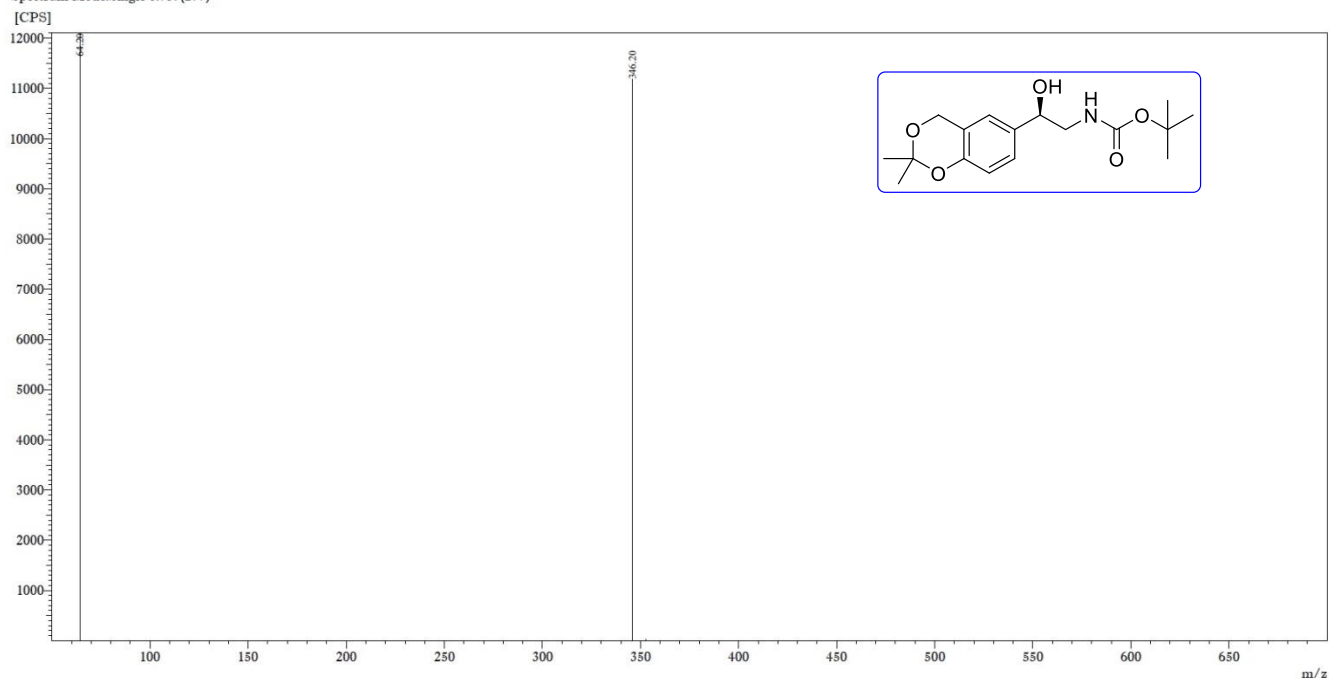
SHIMADZU
LabSolutions

MASS REPORT

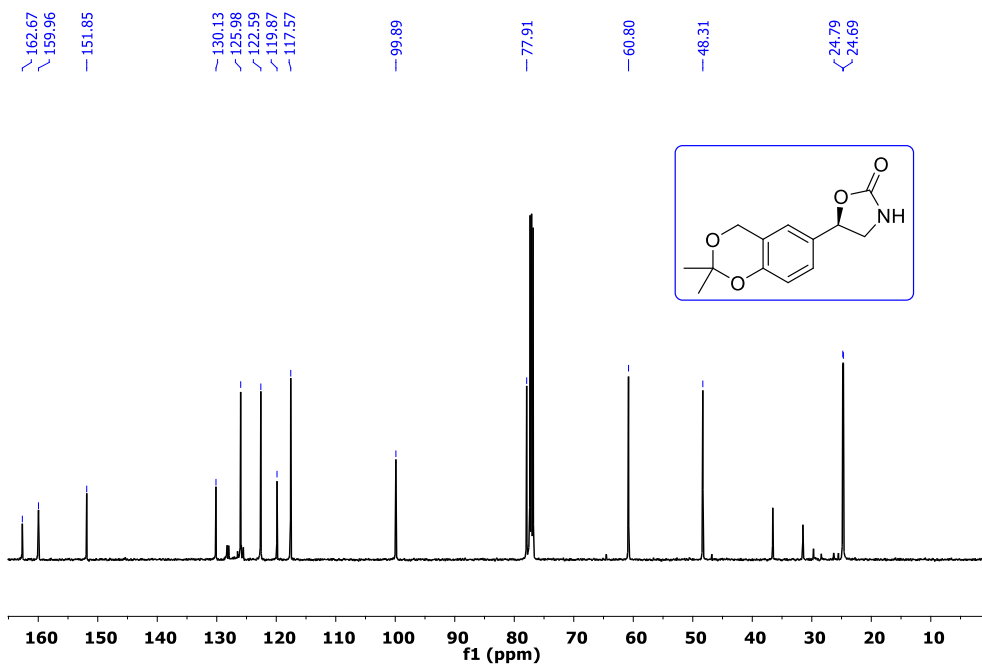
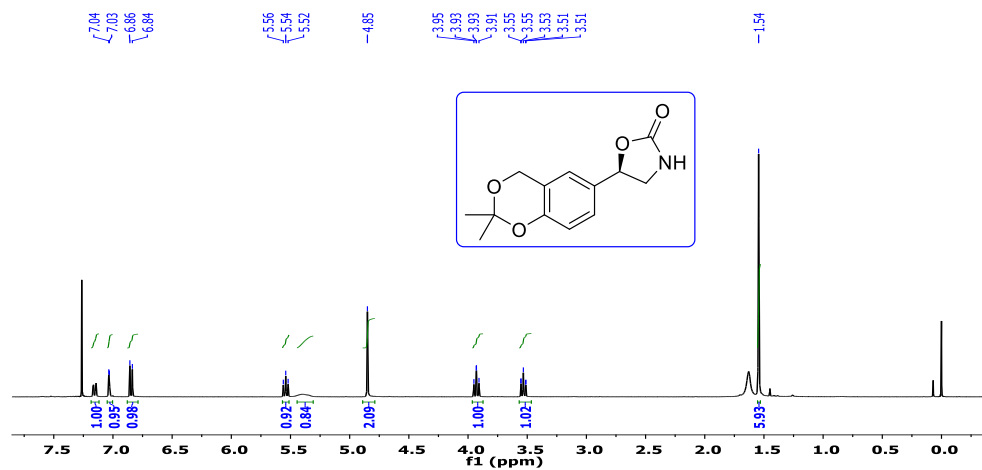
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Date Acquired : 5/24/2023 4:23:52 PM
Batch File : 24052023_532023_1055 AM.lcb

MS Spectrum
D:\DATA\MAY2023\24052023_29.lcd

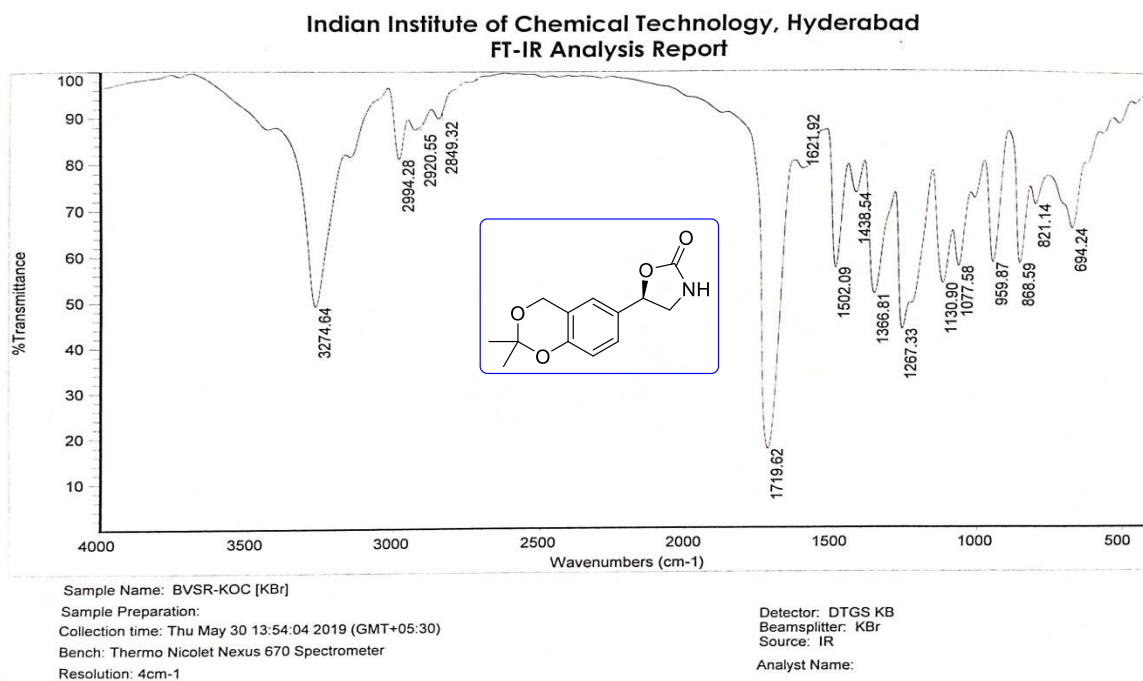
SingleESI Positive+
Spectrum Mode:Single 0.759(277)



1

^1H (400 MHz, CDCl_3) and ^{13}C (126 MHz, CDCl_3) NMR spectra of compound 10

IR spectrum of compound 10



Mass and HRMS (ESI) spectra of compound 10

SHIMADZU
LabSolutions

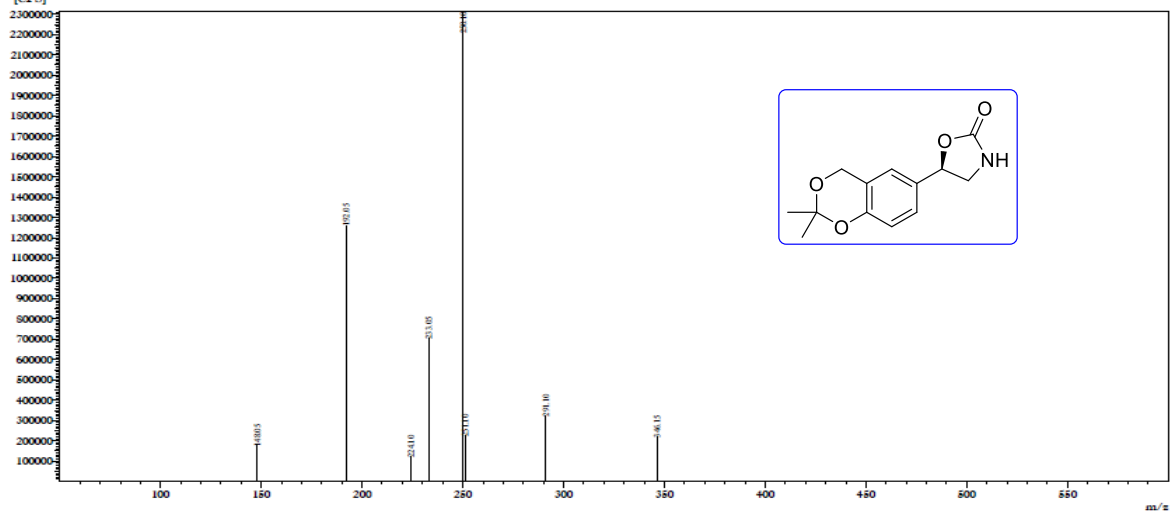
MASS REPORT

OSPC, Dr. RATHOD

Sample Name : HUS-10
Data File : 12032021.28.1.cd
Date Acquired : 3/15/2021 11:33:58 AM
Batch File : 12032021-1.1.cb

Averaged ESI Positive+
Spectrum Mode: Averaged 0.499-0.907(215-347)
[CPS]

MS Spectrum
D:\DATA\March 2021\12032021.28.1.cd



12/03/21 13:30:02

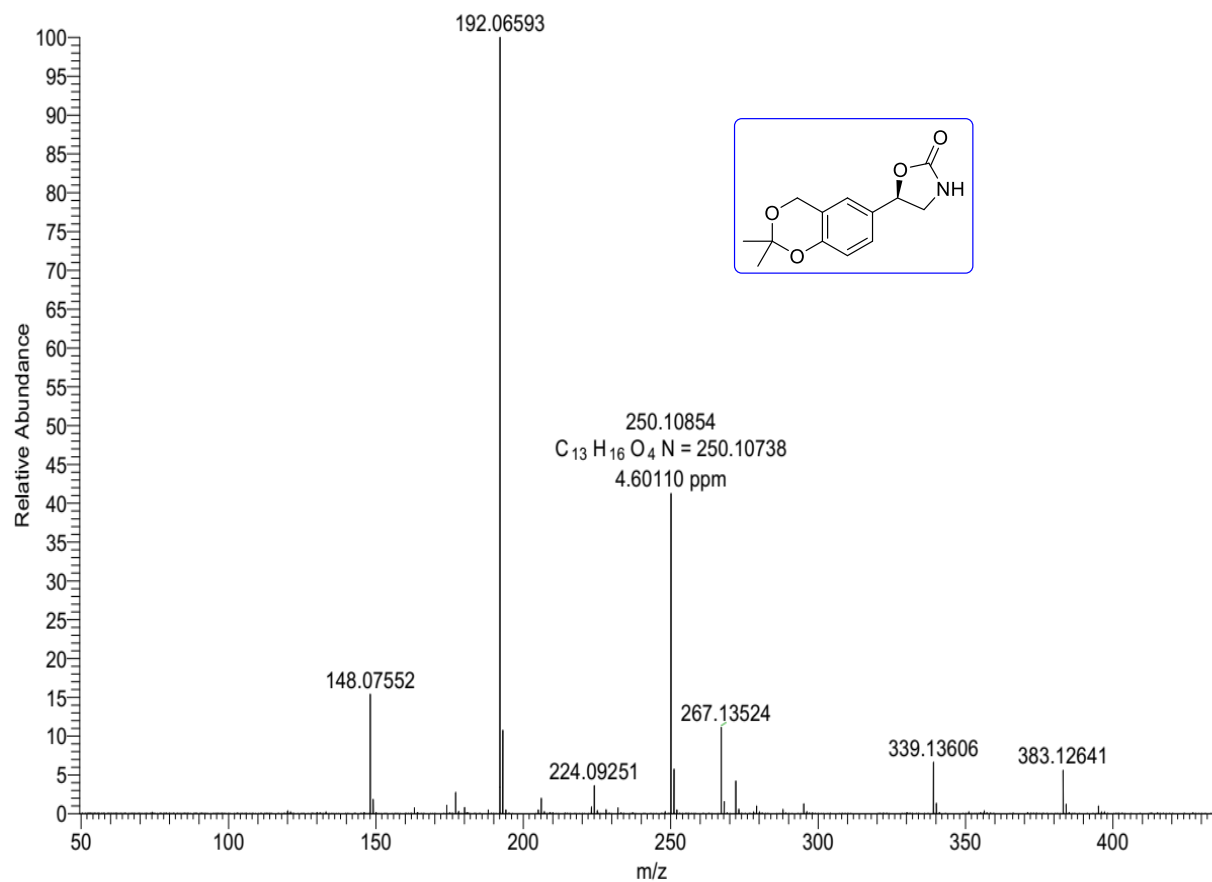
249

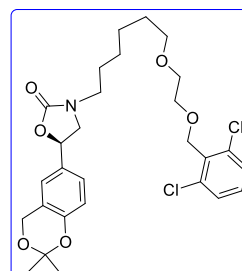
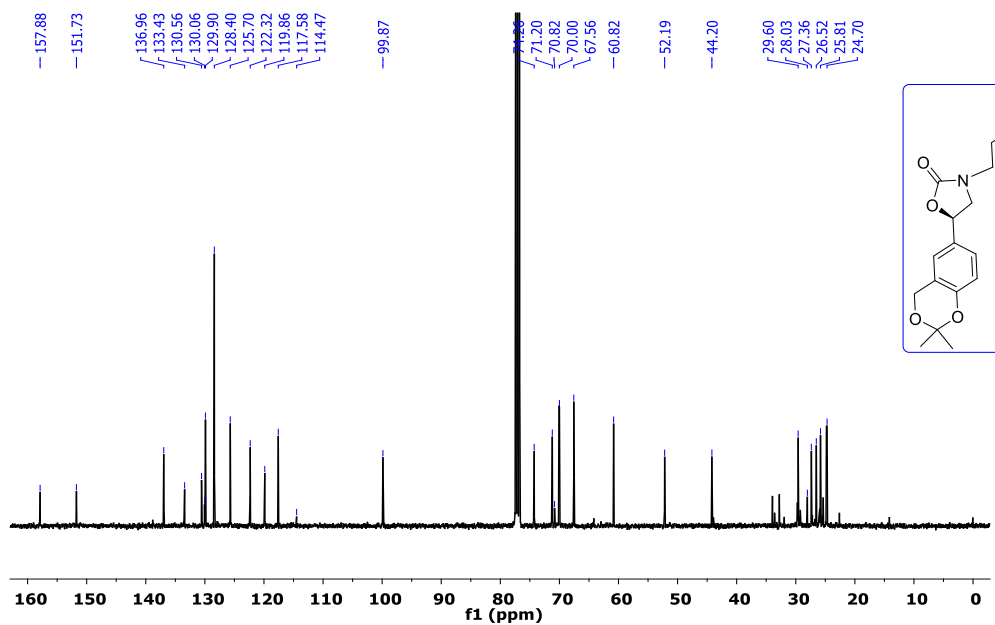
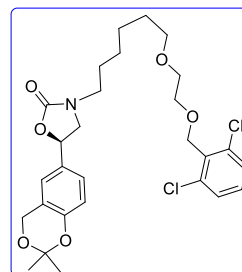
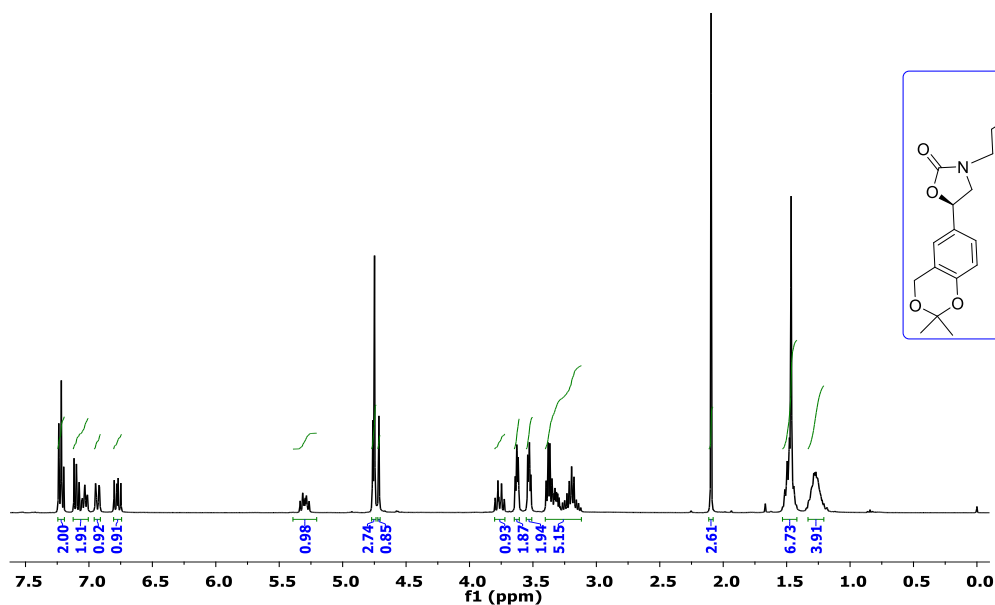
Thermo Scientific Orbitrap Exploris 120

Analysed by G SAIKRISHNA

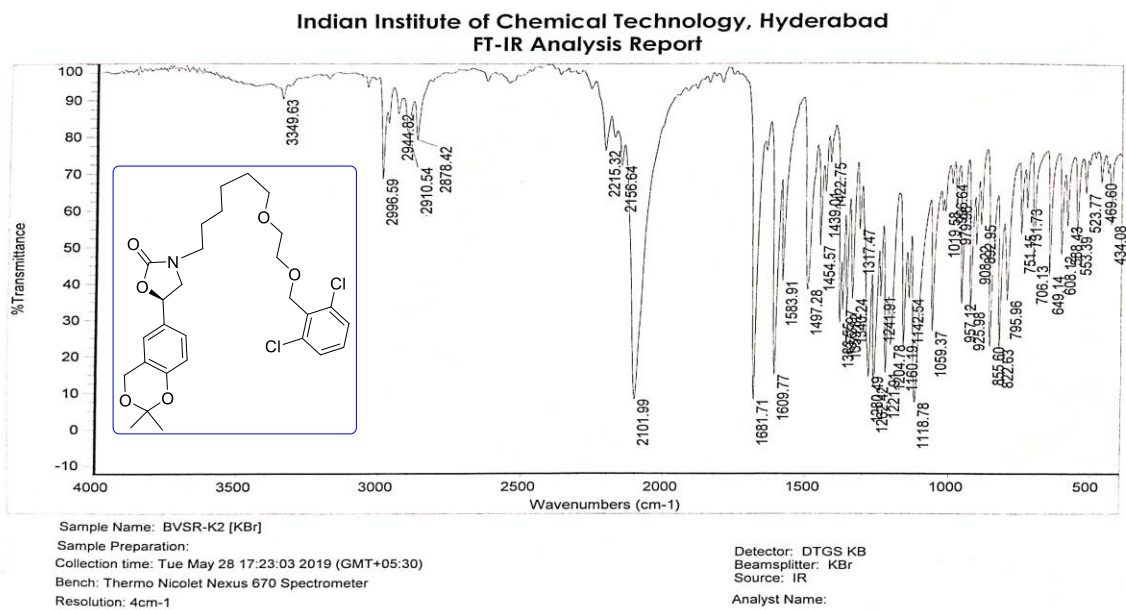
BVSR-02 #11-34 RT: 0.03-0.08 AV: 24 SB: 382 0.32-1.20 NL: 6.14E8

T: FTMS + p ESI Full ms [50.0000-3000.0000]



^1H (400 MHz, CDCl_3) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 12

IR spectrum of compound 12



Mass and HRMS (ESI) spectra of compound 12

SHIMADZU
LabSolutions

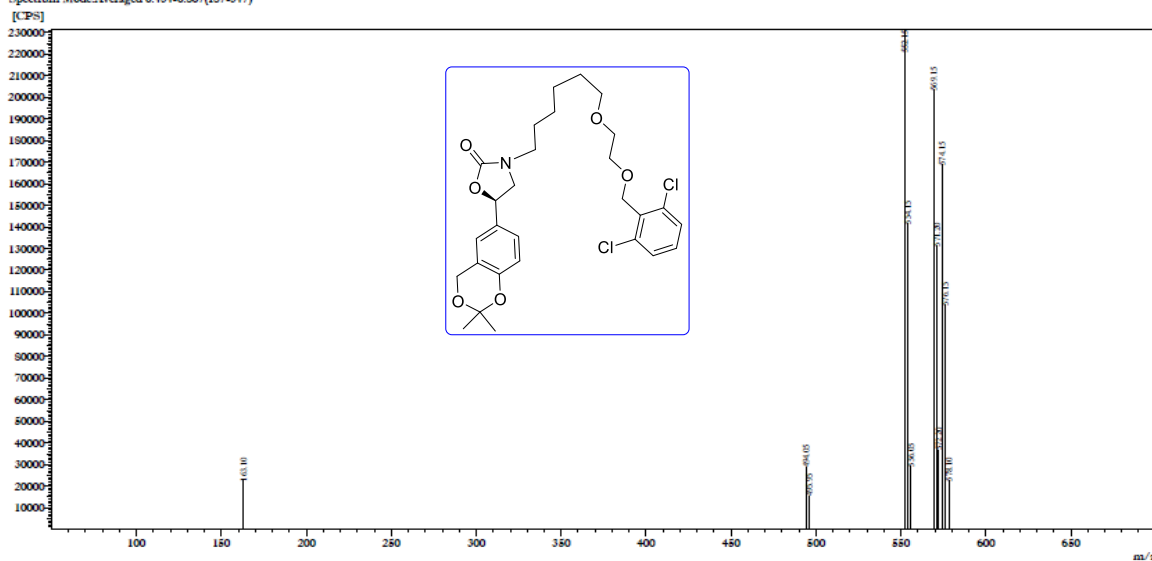
MASS REPORT

OSPC, Dr RATHOD

Sample Name : HUS-12
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 Date Acquired : 3/15/2021 11:41:45 AM
 Batch File : 12032021-1.lcb

MS Spectrum
 D:\DATA\March 2021\12032021.31.lcd

Averaged ESI Positive+
 Spectrum Mode: Averaged 0.434-0.807(187-347)

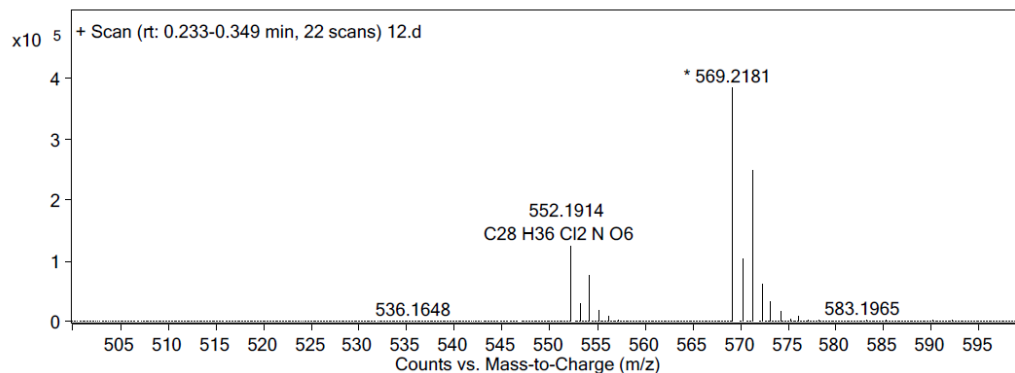


Qualitative Analysis Report

Data File	12.d	Sample Name	
Sample Type	Sample	Position	P1-A1
Instrument Name	Instrument 1	User Name	CSIR-IICT\Analyst
Acq Method	hrms-pos-method.m	Acquired Time	13-09-2021 12:39:47
IRM Calibration Status	Success	DA Method	0.m
Comment		Info.	
Sample Group		Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1)
Stream Name	LC 1		

User Spectra

Fragmentor Voltage Collision Energy Ionization Mode
80 0 ESI

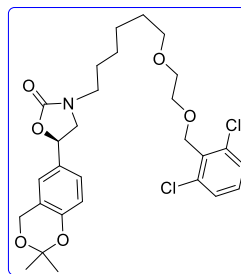


Peak List

m/z	z	Abund
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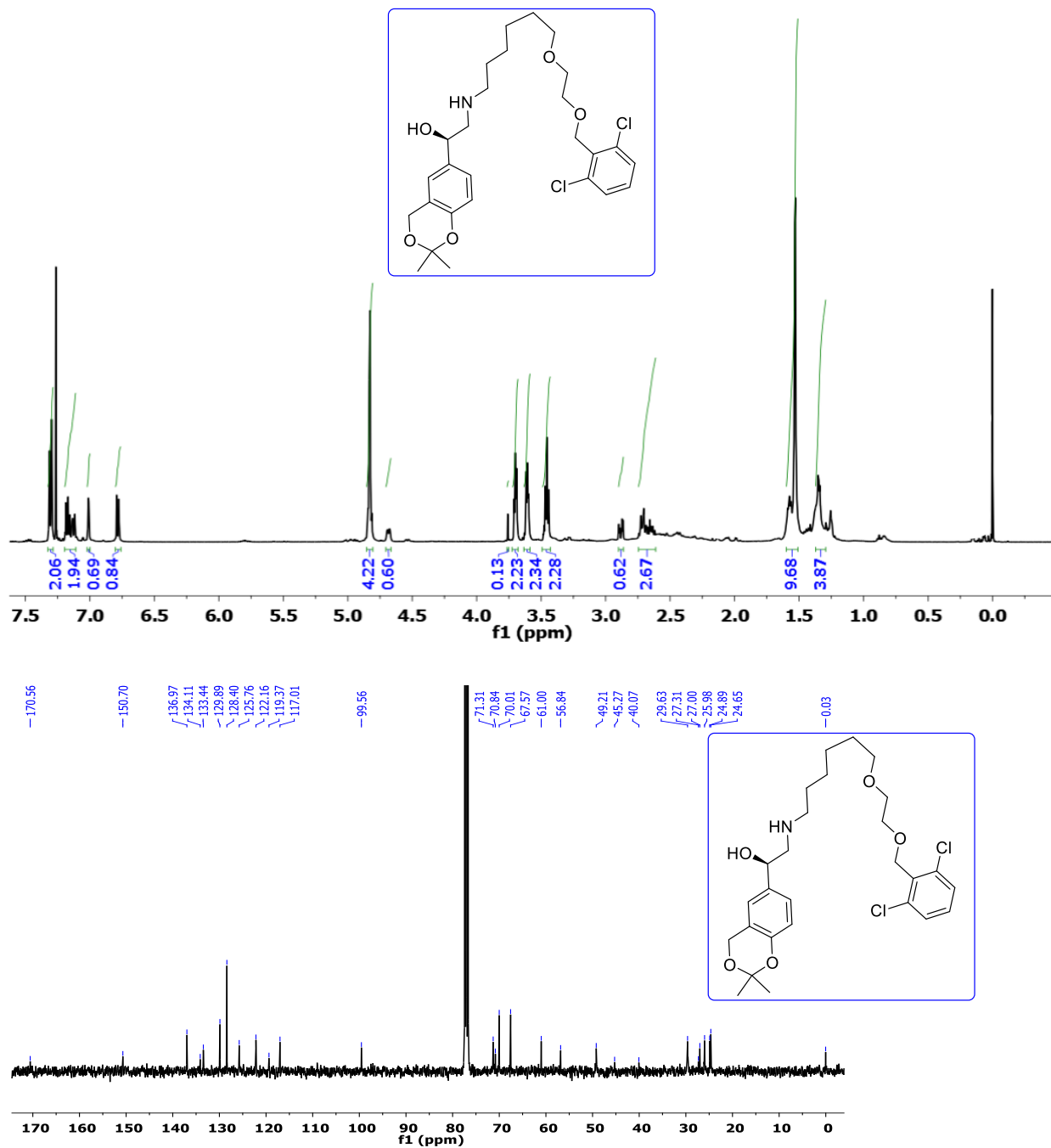
Formula Calculator Element Limits

Element	Min	Max
C	0	30
H	0	60
O	0	6
N	0	1
Cl	0	2

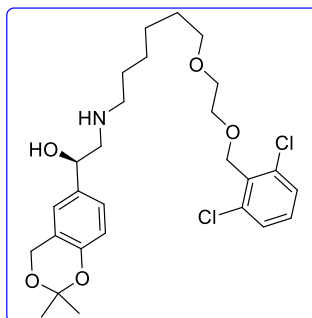
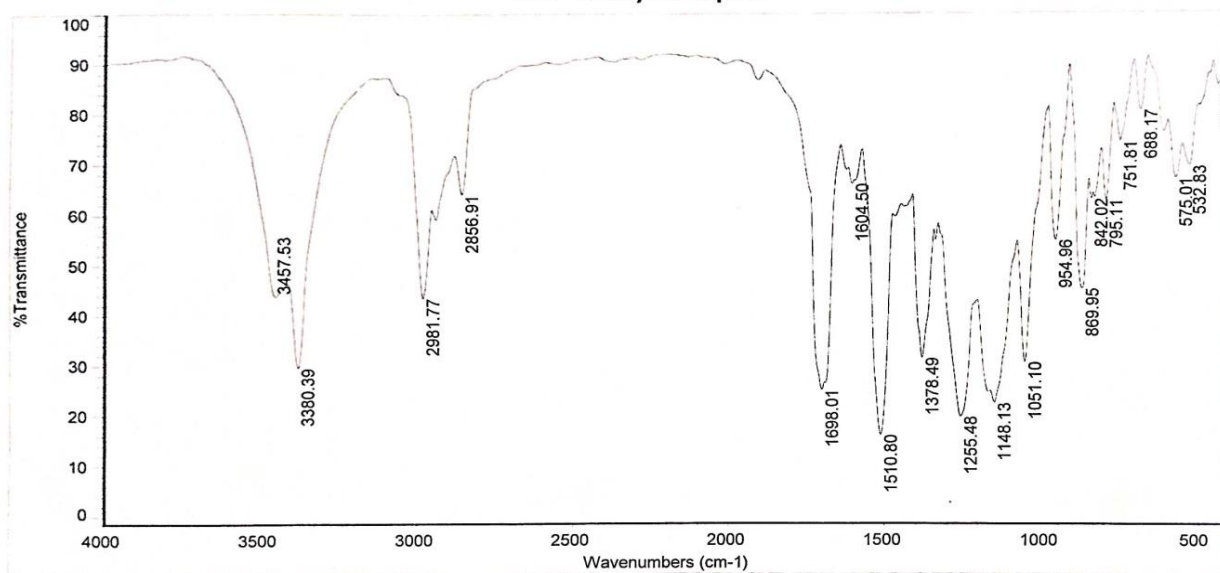


Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C28 H36 Cl2 N O6	True	552.1918	552.19197	0.3	C28 H36 Cl2 N O6	95.69
C28 H35 Cl2 N O6	True	551.18398	551.18414	0.3	C28 H36 Cl2 N O6	95.69

^1H (500 MHz, CDCl_3) and ^{13}C (101 MHz, CDCl_3) NMR spectra of compound 13

IR spectrum of compound 13

Indian Institute of Chemical Technology, Hyderabad
FT-IR Analysis Report

Sample Name: BVSR-KNB [KBr]

Sample Preparation:

Collection time: Thu May 30 13:50:08 2019 (GMT+05:30)

Bench: Thermo Nicolet Nexus 670 Spectrometer

Resolution: 4cm⁻¹

Detector: DTGS KB

Beamsplitter: KBr

Source: IR

Analyst Name:

Mass and HRMS (ESI) spectra of compound 13

OSPC, Dr. RATHOD

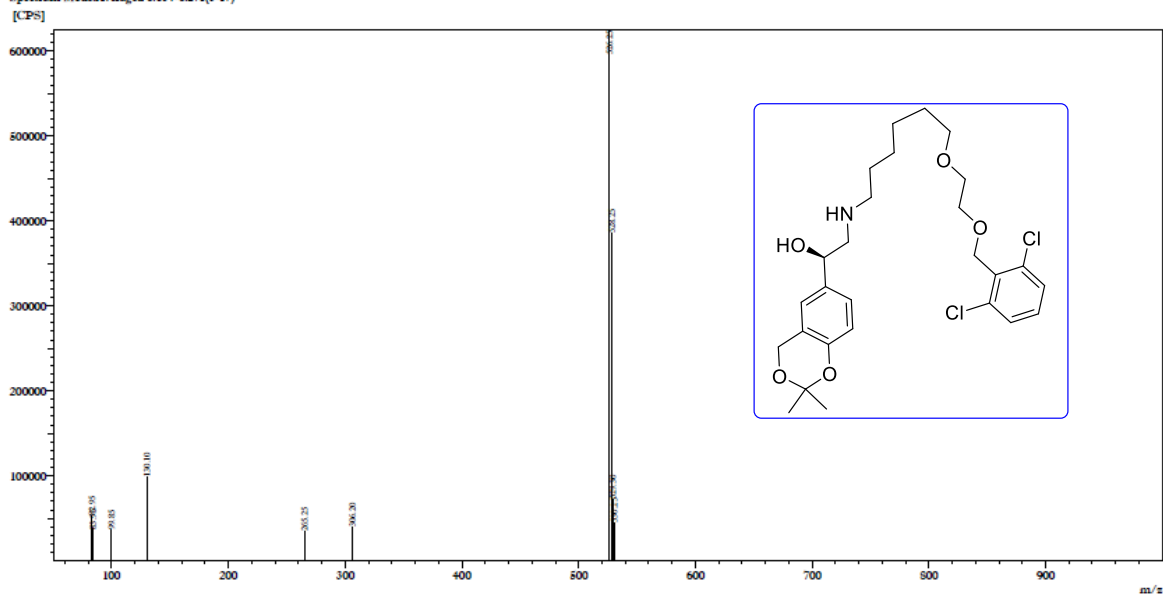
SHIMADZU
LabSolutions

MASS REPORT

Sample Name : HUS-N
Data File : 23082021.15.lcd
Date Acquired : 8/23/2021 11:35:31 AM
Batch File : 23082021-1.lcb

MS Spectrum
D:\DATA\AUG-2021\23082021.15.lcd

Averaged ESI Positive+
Spectrum Mode: Averaged 0.034-0.271 (3-17)



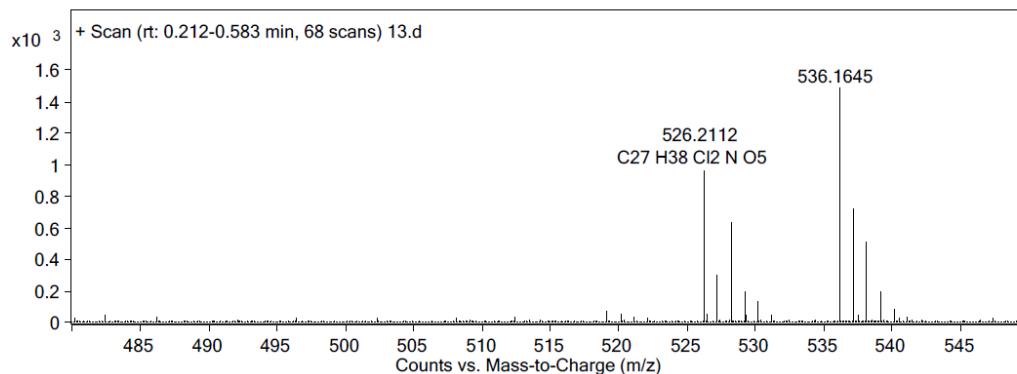
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Qualitative Analysis Report

Data File	13.d	Sample Name	
Sample Type	Sample	Position	P1-A1
Instrument Name	Instrument 1	User Name	CSIR-IICT\Analyst
Acq Method	hrms-pos-method.m	Acquired Time	13-09-2021 12:41:12
IRM Calibration Status	Success	DA Method	0.m
Comment		Info.	
Sample Group		Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1)
Stream Name	LC 1		

User Spectra

Fragmentor Voltage Collision Energy Ionization Mode
80 0 ESI

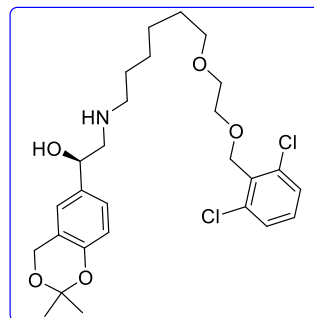


Peak List

m/z	z	Abund
130.1587	1	16312.73

Formula Calculator Element Limits

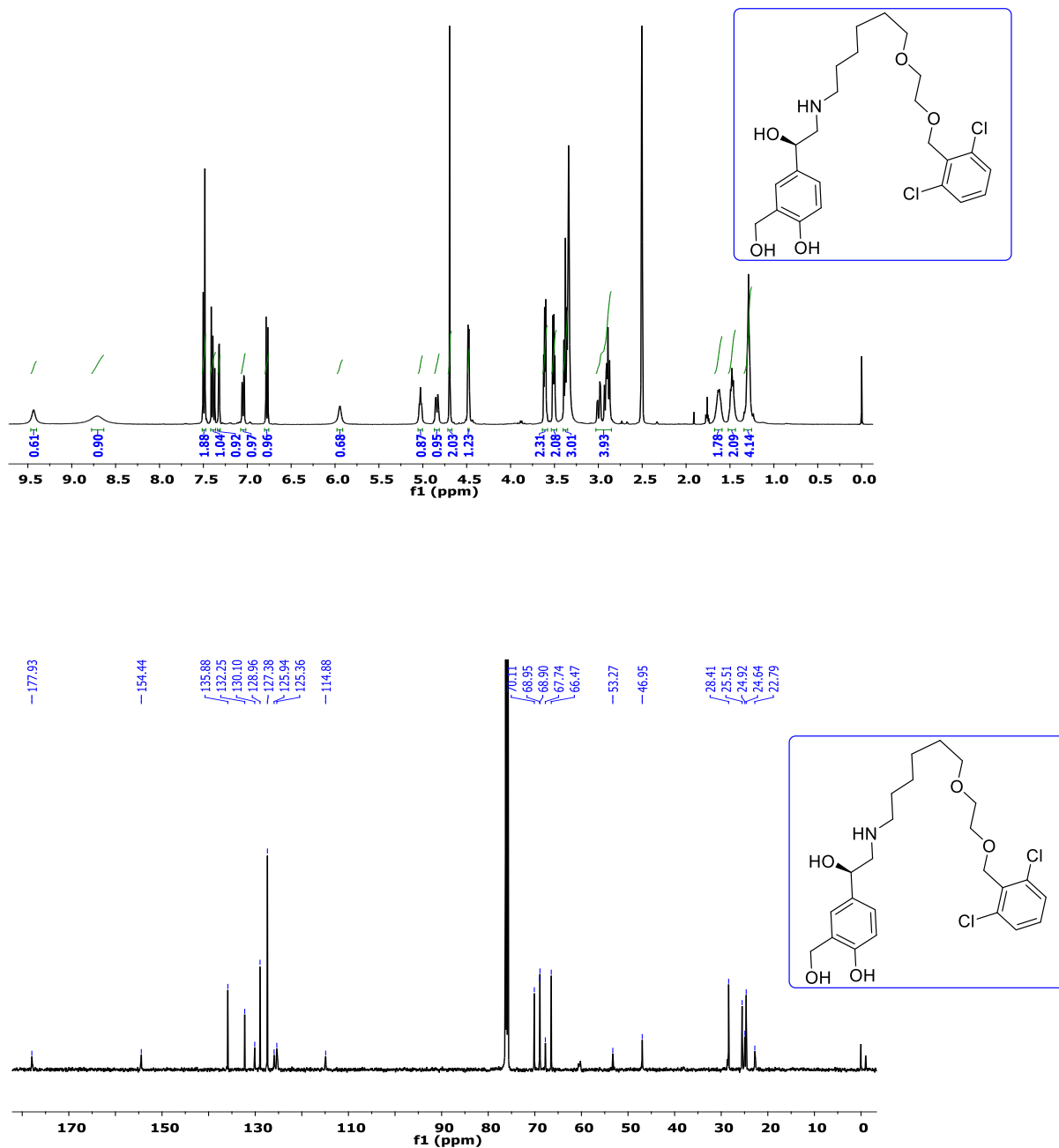
Element	Min	Max
C	0	30
H	0	60
O	0	6
N	0	1
Cl	0	2



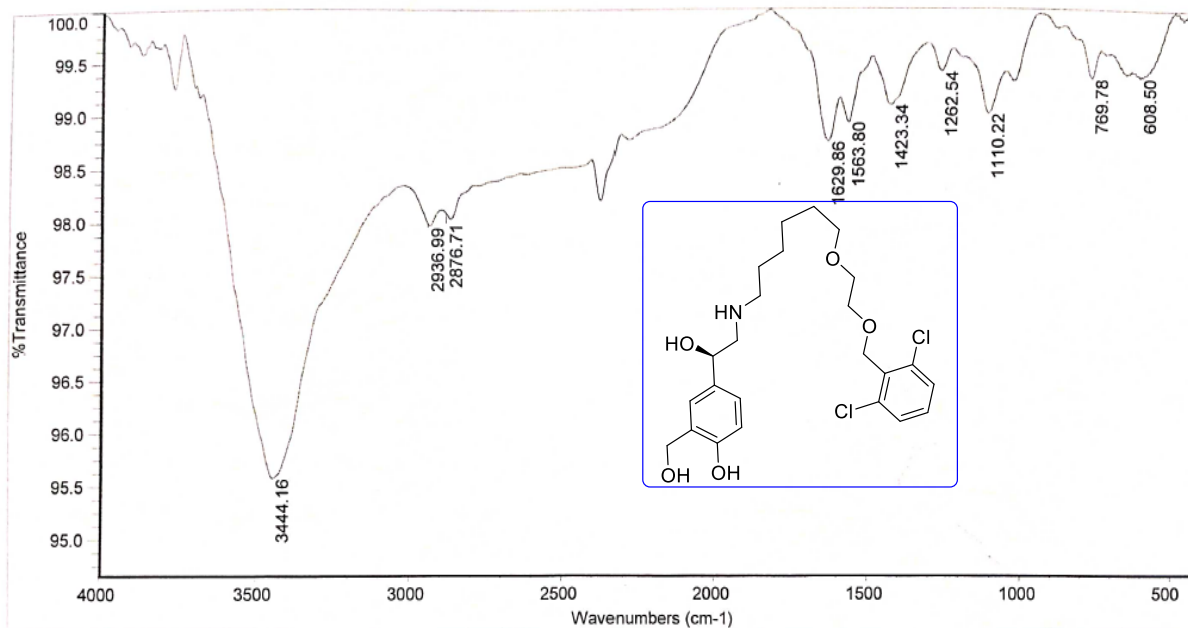
Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C27 H38 Cl2 N O5	True	526.21165	526.2127	2.01	C27 H38 Cl2 N O5	96.37
C27 H37 Cl2 N O5	True	525.20382	525.20488	2.01	C27 H38 Cl2 N O5	96.37
C25 H39 Cl2 N O5	True	503.22192	503.22053	-2.76	C25 H39 Cl2 N Na O5	93.48



^1H (400 MHz, DMSO) and ^{13}C (126 MHz, CDCl_3) NMR spectra of compound 1

IR spectrum of compound 1

Indian Institute of Chemical Technology, Hyderabad
FT-IR Analysis Report

Sample Name: BVSr-KVD [NEAT]

Sample Preparation:

Collection time: Thu May 30 17:09:24 2019 (GMT+05:30)

Bench: Thermo Nicolet Nexus 670 Spectrometer

Resolution: 4cm⁻¹

Detector: DTGS KB

Beamsplitter: KBr

Source: IR

Analyst Name:

Mass and HRMS (ESI) spectra of compound 1

OSPC, Dr. RATHOD

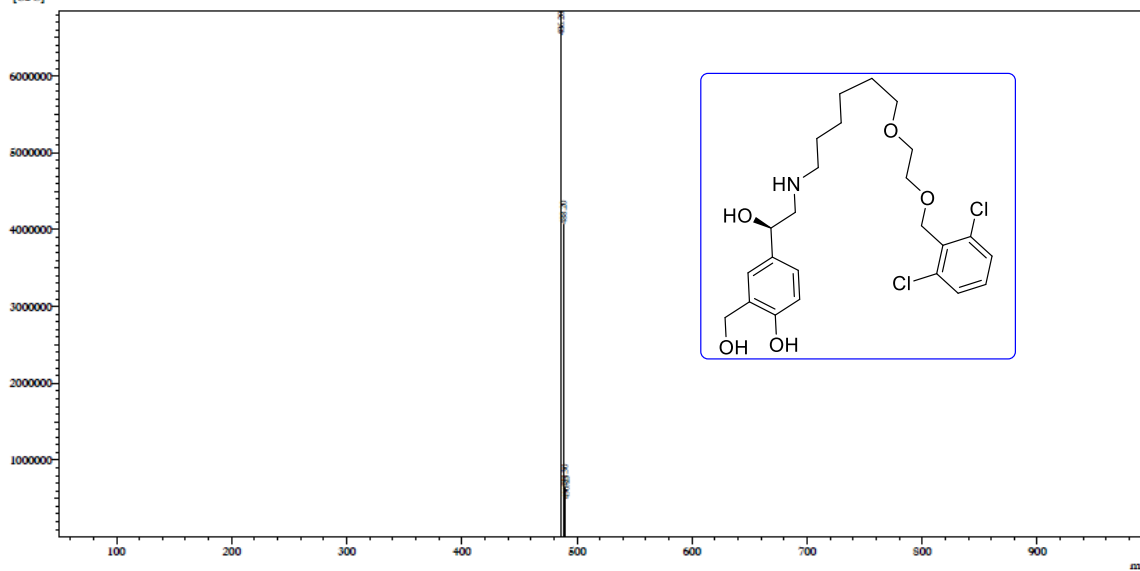
SHIMADZU
LabSolutions

MASS REPORT

Sample Name : HUS-14
Data File : 18082021.40.1cd
Date Acquired : 8/18/2021 5:08:41 PM
Batch File : 18072021-4.1cb

MS Spectrum
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Single:ESI Positive+
Spectrum Mode:Single 0.135(9)
[CPS]



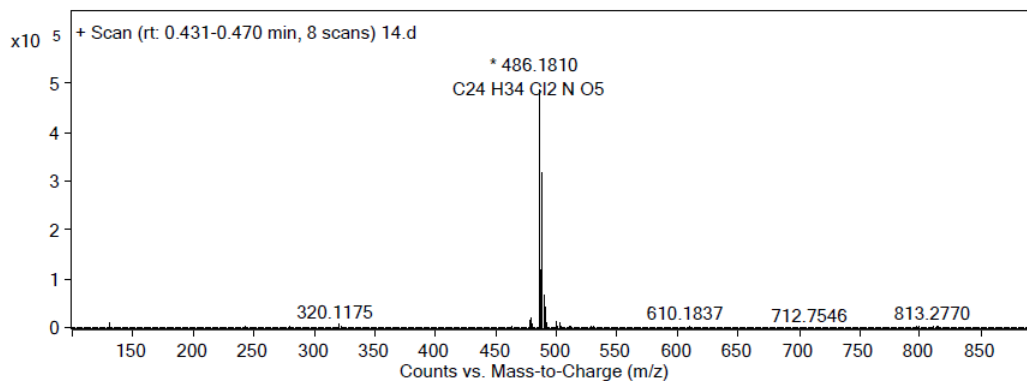
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Qualitative Analysis Report

Data File	14.d	Sample Name	
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IRM Calibration Status	Success	DA Method	0.m
Comment		Info.	
Sample Group		Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1)
Stream Name	LC 1		

User Spectra

Fragmentor Voltage Collision Energy Ionization Mode
80 0 ESI

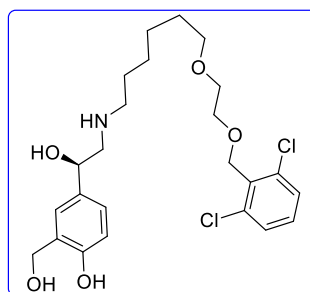


Peak List

m/z	z	Abund	Formula	Ion
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Formula Calculator Element Limits

Element	Min	Max
C	0	25
H	0	60
O	0	6
N	0	1
Cl	0	2



Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C ₂₄ H ₃₄ Cl ₂ N ₂ O ₅	True	486.18137	486.1814	0.07	C ₂₄ H ₃₄ Cl ₂ N ₂ O ₅	97.16
C ₂₄ H ₃₃ Cl ₂ N ₂ O ₅	True	485.17355	485.17358	0.07	C ₂₄ H ₃₄ Cl ₂ N ₂ O ₅	97.16



Agilent Technologies

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Printed at 12:45 PM on 13-Sep-2021