# Supplementary Material

# An efficient total synthesis of vilanterol: an inhaled drug

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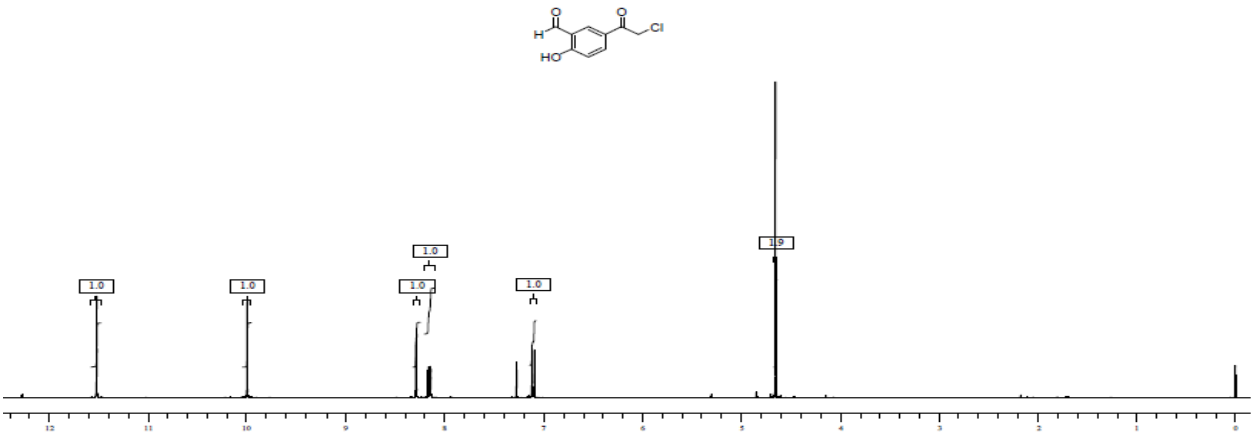
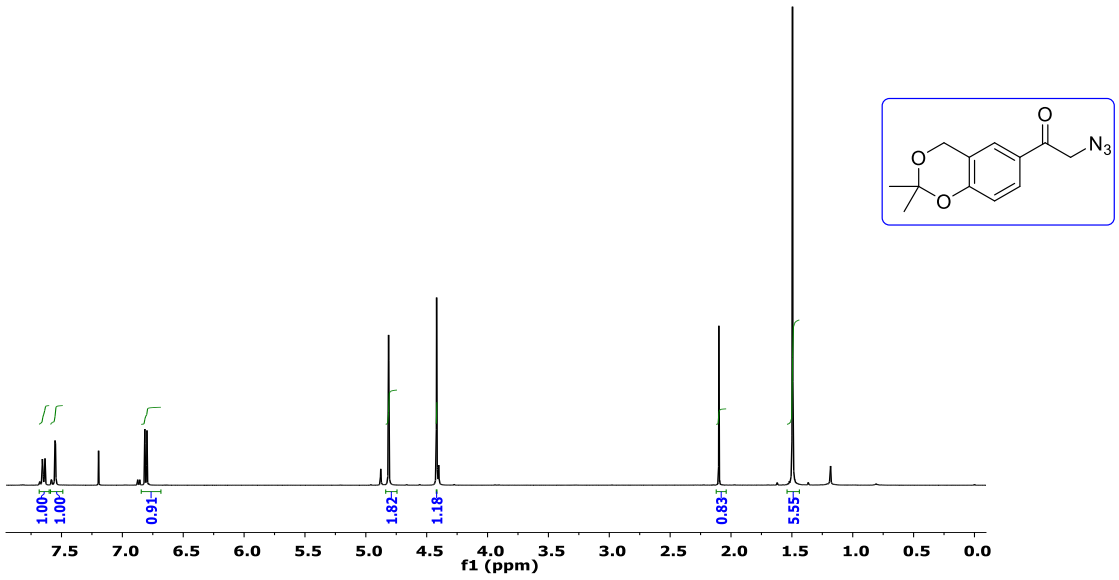
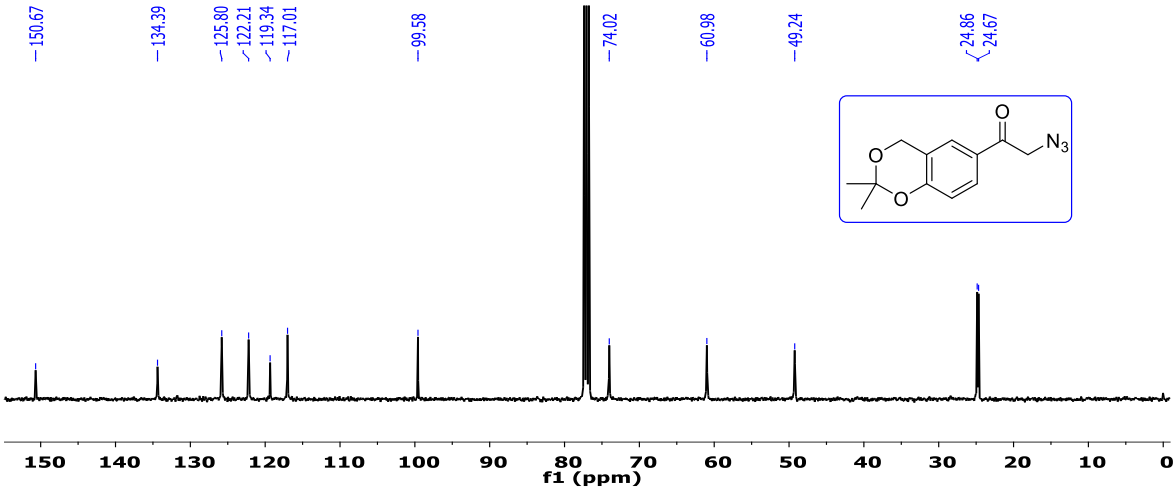
**$^{1}$H (300 MHz, $CDCl_3$) and $^{13}$C (75 MHz, $CDCl_3$) NMR spectra of compound 5**



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



## Mass (ESI) of compound 6

OSPC, Dr RATHOD

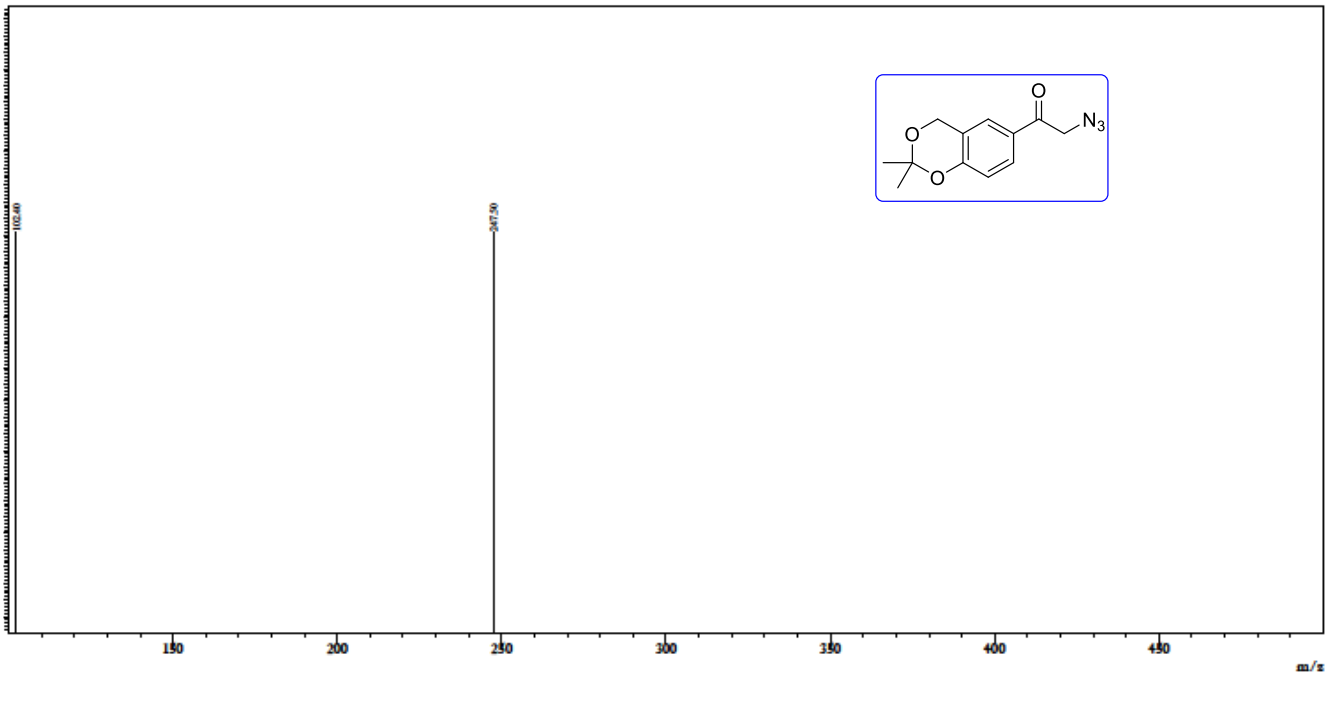
SHIMADZU
LabSolutions

# MASS REPORT

ple Name : PSH-247-6
File : 24052023.28.lcd
Acquired : 5/24/2023 4:21:13 PM
h File : 24052023 532023 1055 AM.lcb

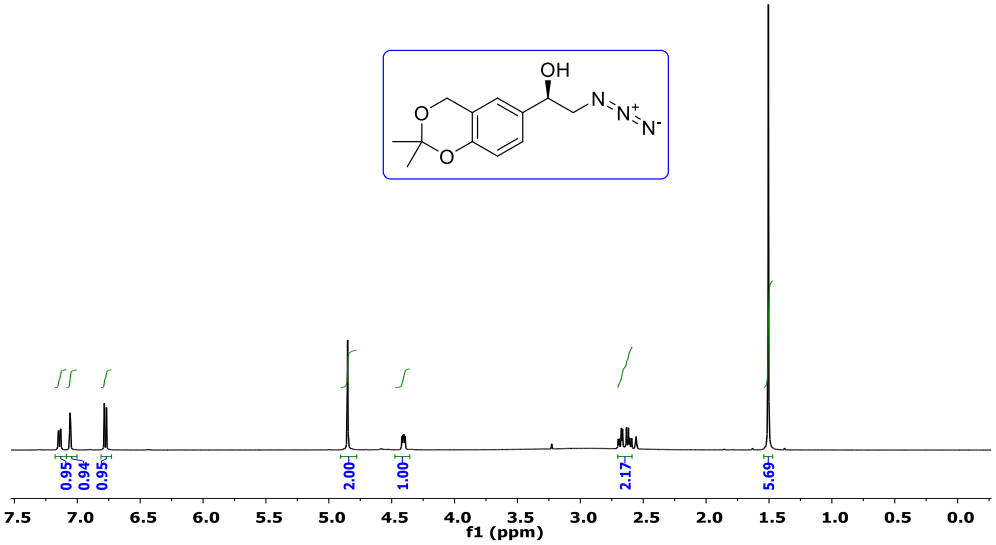
MS Spectrum
D:\DATA\MAY2023\24052023.28.lcd

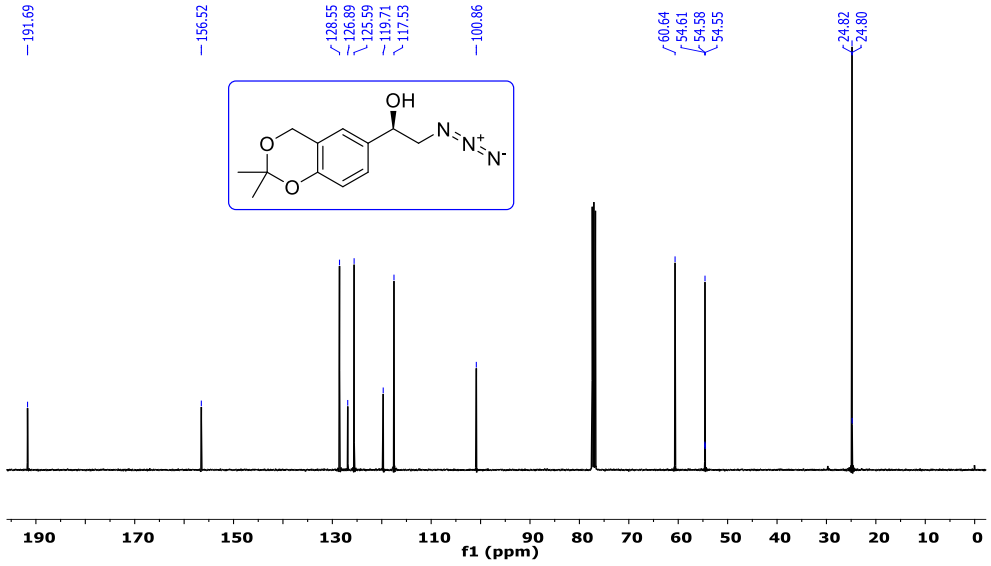
ESI Positive+
um Mode:Single 0.220(S1)




1

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



## Mass (ESI) of compound 7

OSPC, Dr RATHOD

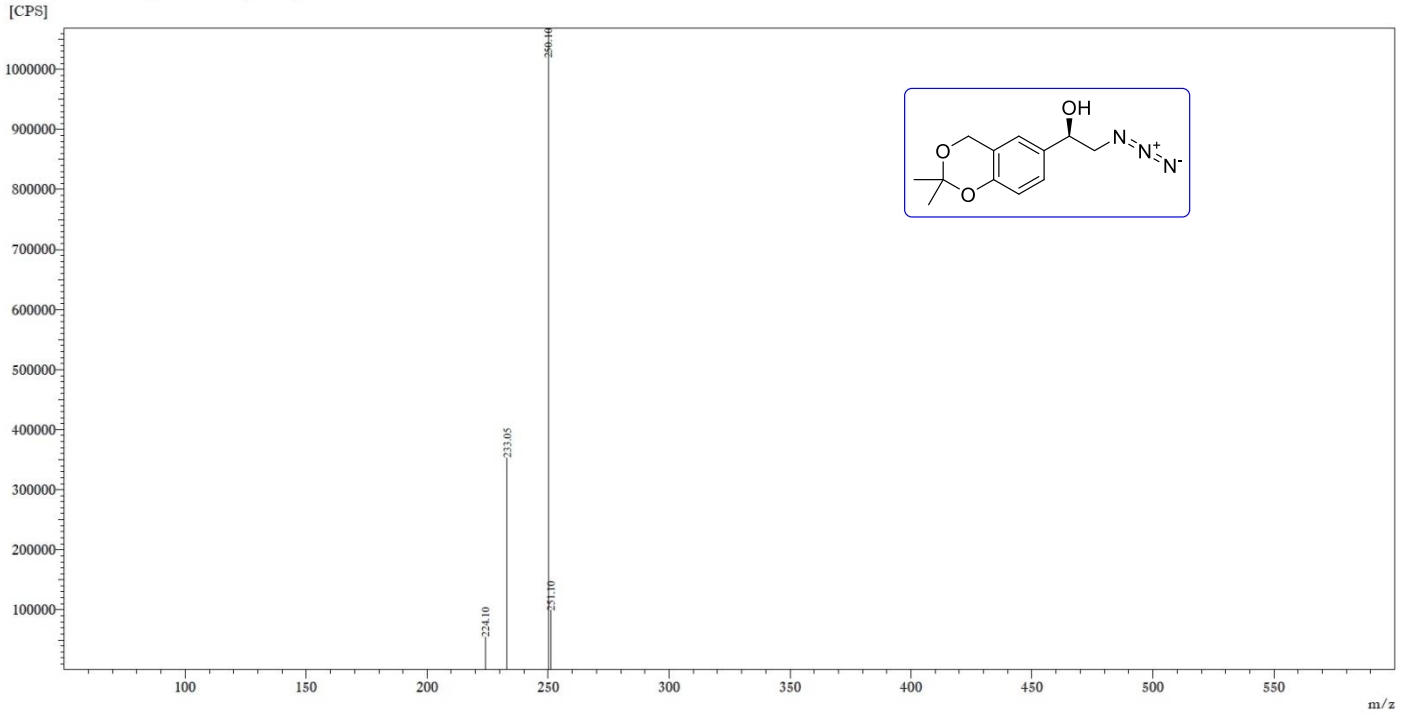
SHIMADZU
LabSolutions

# 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

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

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




1

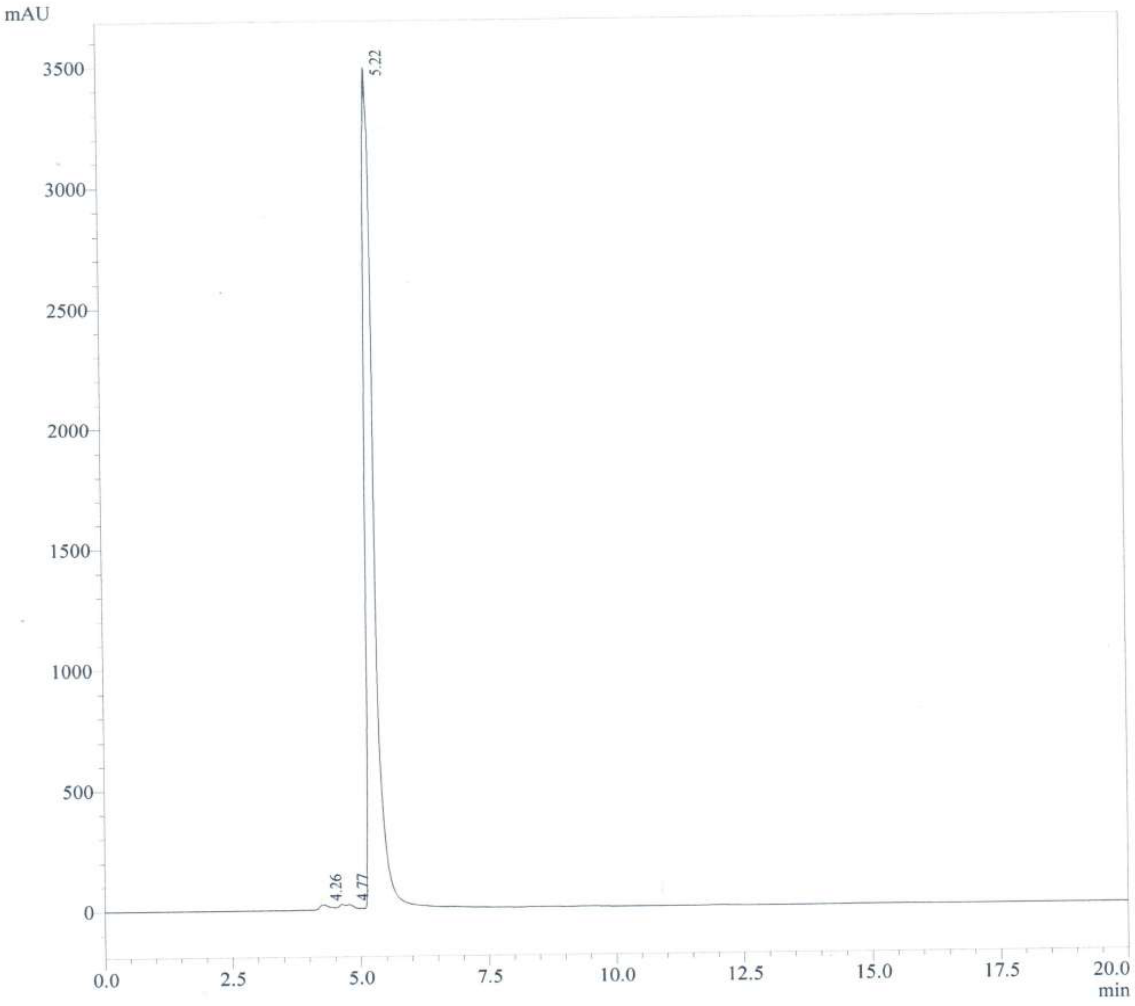
 

**HPLC of compound 7 on IG5 CHIRALPAK column with a Hex/ IPA ratio of 50/50**

**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

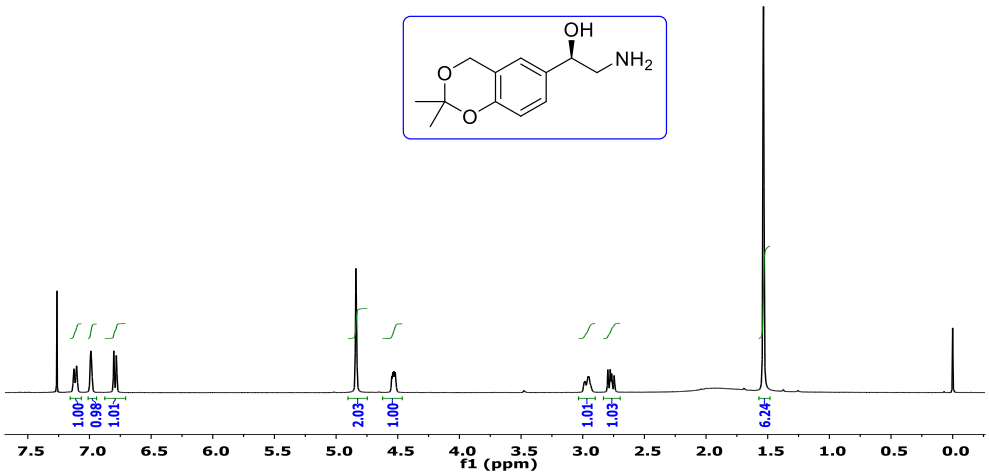
D:\BVSR\KRED-290.lcd

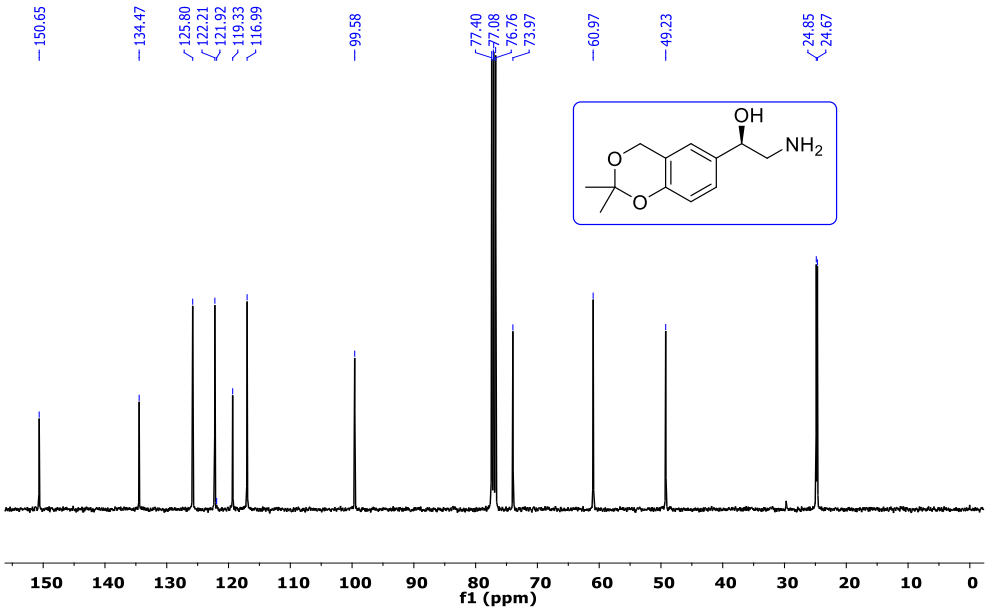



PDA Ch1 200nm - 500nm 2nm

| 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 |

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



## Mass (ESI) of compound 8

OSPC, Dr RATHOD

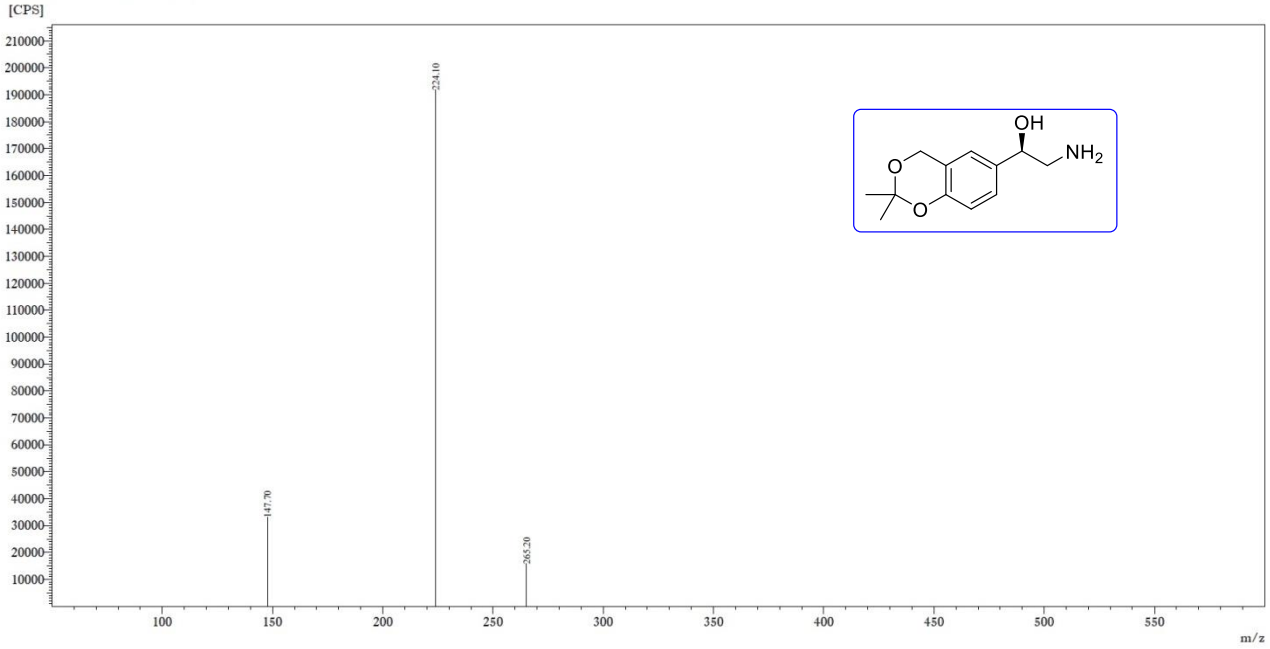
SHIMADZU LabSolutions

MASS REPORT

Sample Name : PSH-223-8
Data File : 24052023.30.lcd
Date Acquired : 5/24/2023 4:26:32 PM
Batch File : 24052023 532023 1055 AM.lcb

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

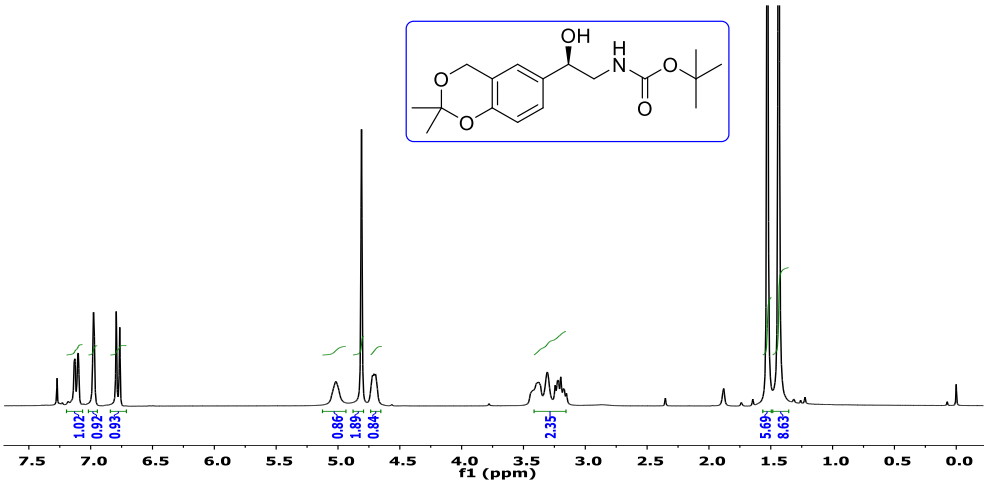
SingleESI Positive+
Spectrum Mode:Single 0.330(121)

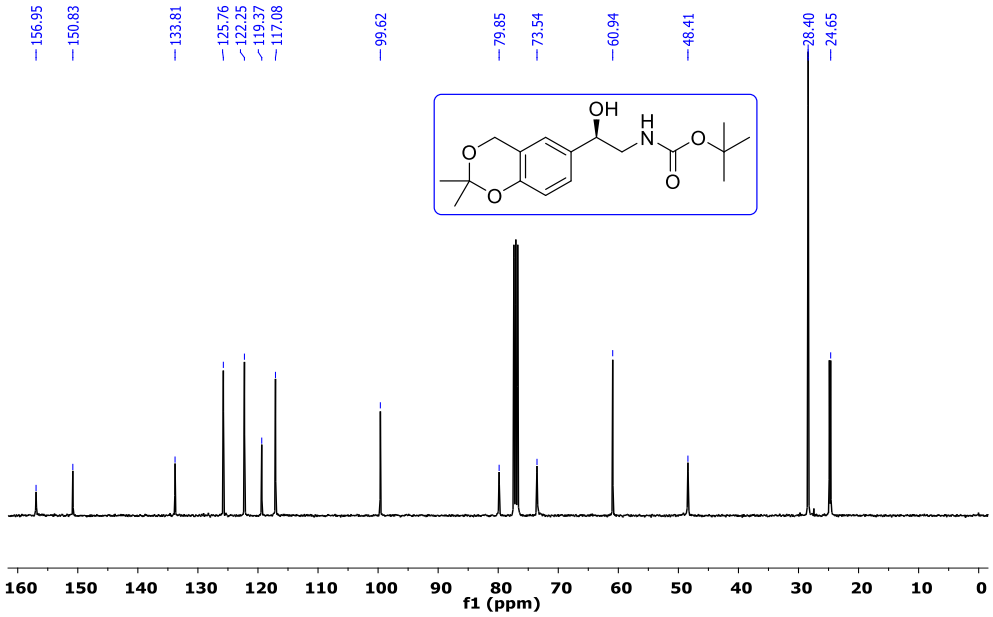



1

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



## Mass (ESI) of compound 9

OSPC, Dr RATHOD

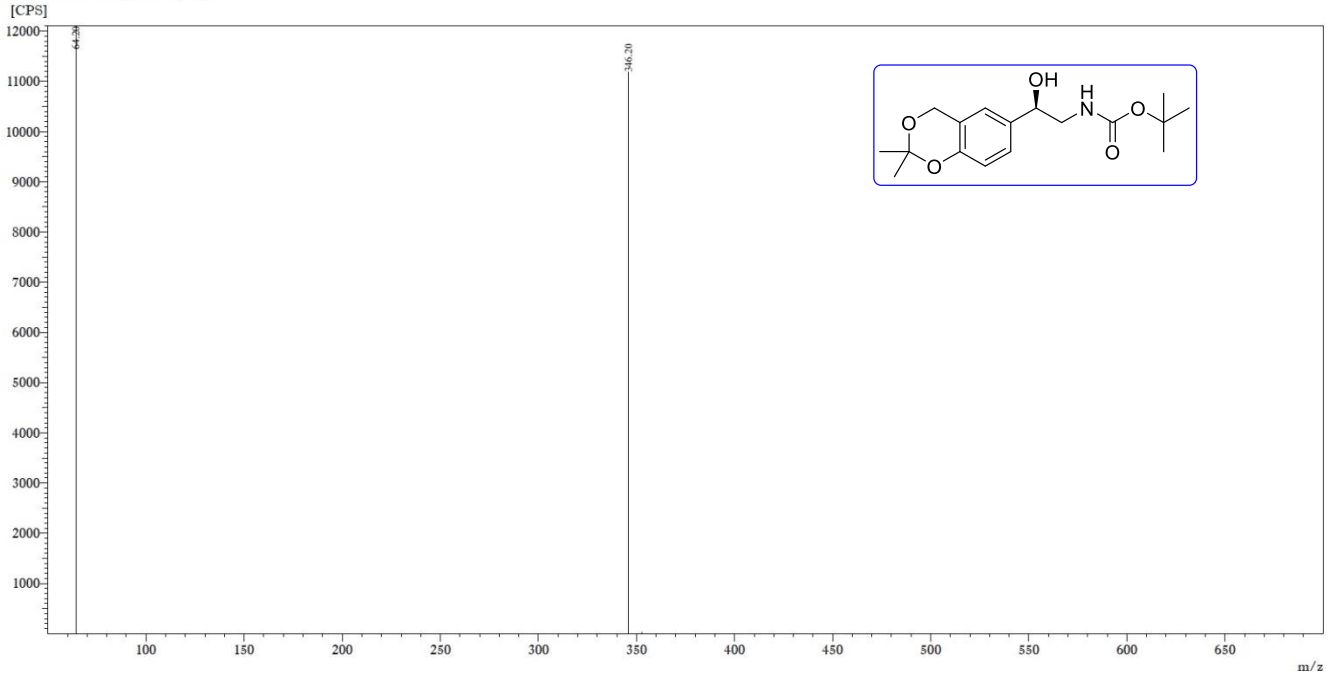
SHIMADZU
LabSolutions

MASS REPORT

Sample Name : PSH-323-9
Data File : 24052023.29.lcd
Date Acquired : 5/24/2023 4:23:52 PM
Batch File : 24052023 532023 1055 AM.lcb

MS Spectrum
D:\DATA\MAY2023\24052023.29.lcd

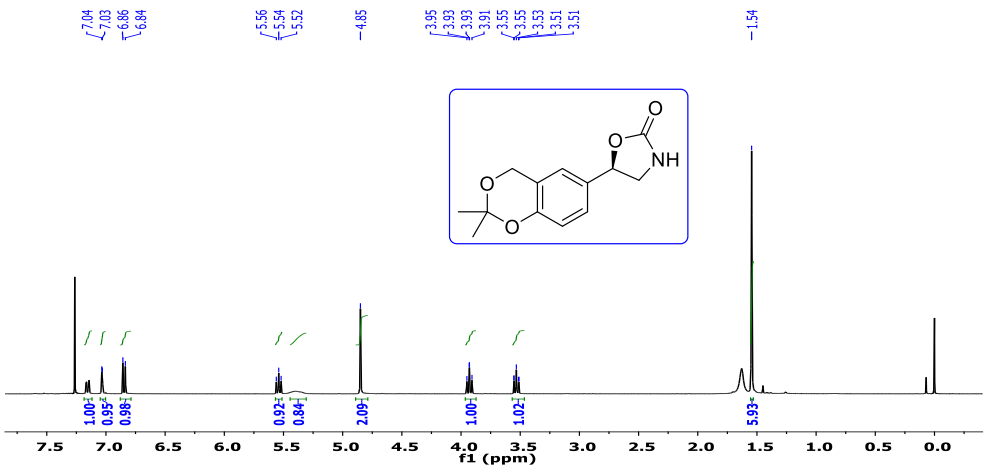
SingleESI Positive+
Spectrum Mode:Single 0.759(277)

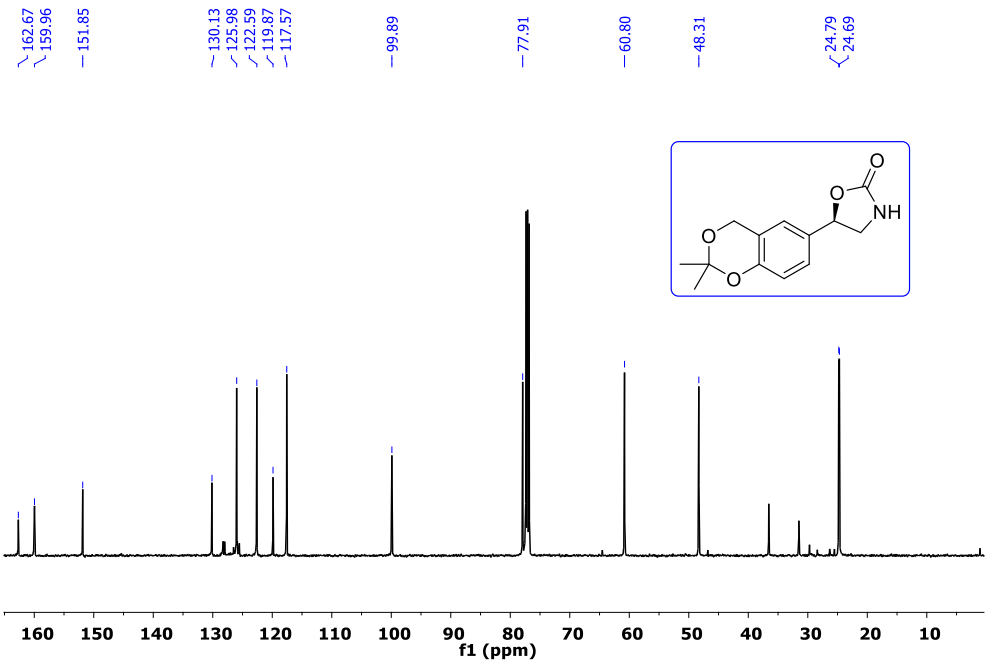



1

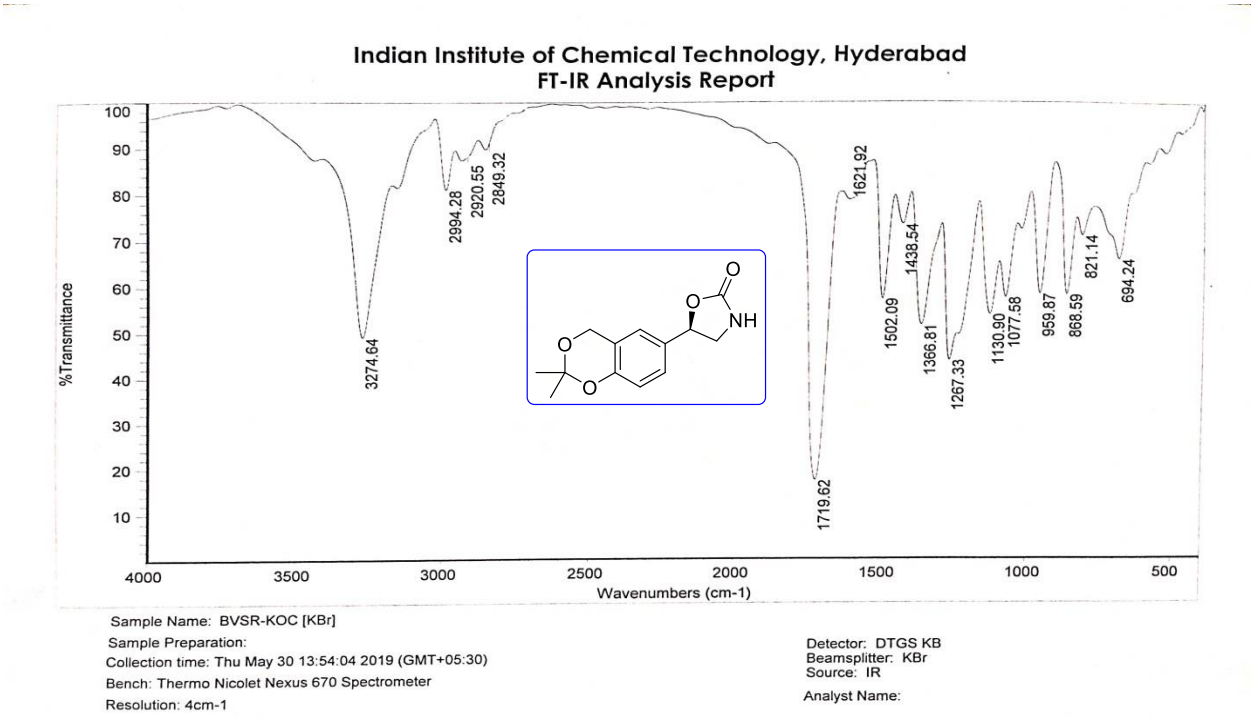
 

## $^{1}H$ (400 MHz, $CDCl_3$) and $^{13}C$ (126 MHz, $CDCl_3$) NMR spectra of compound 10

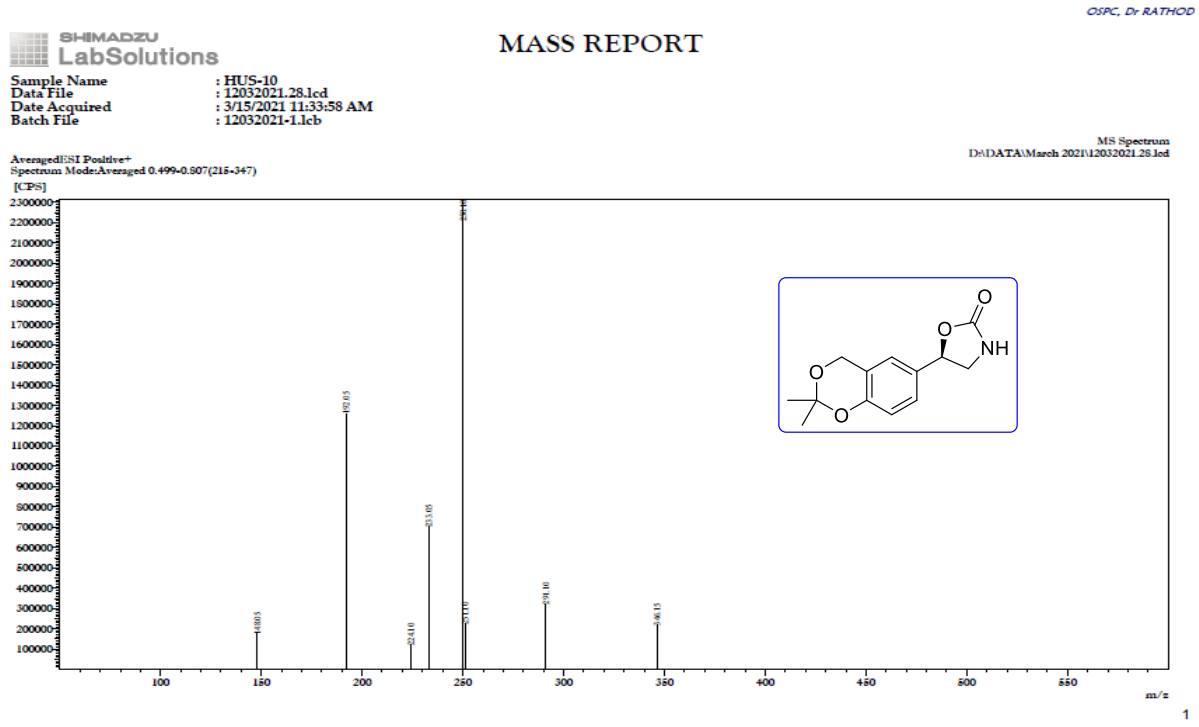


## IR spectrum of compound 10



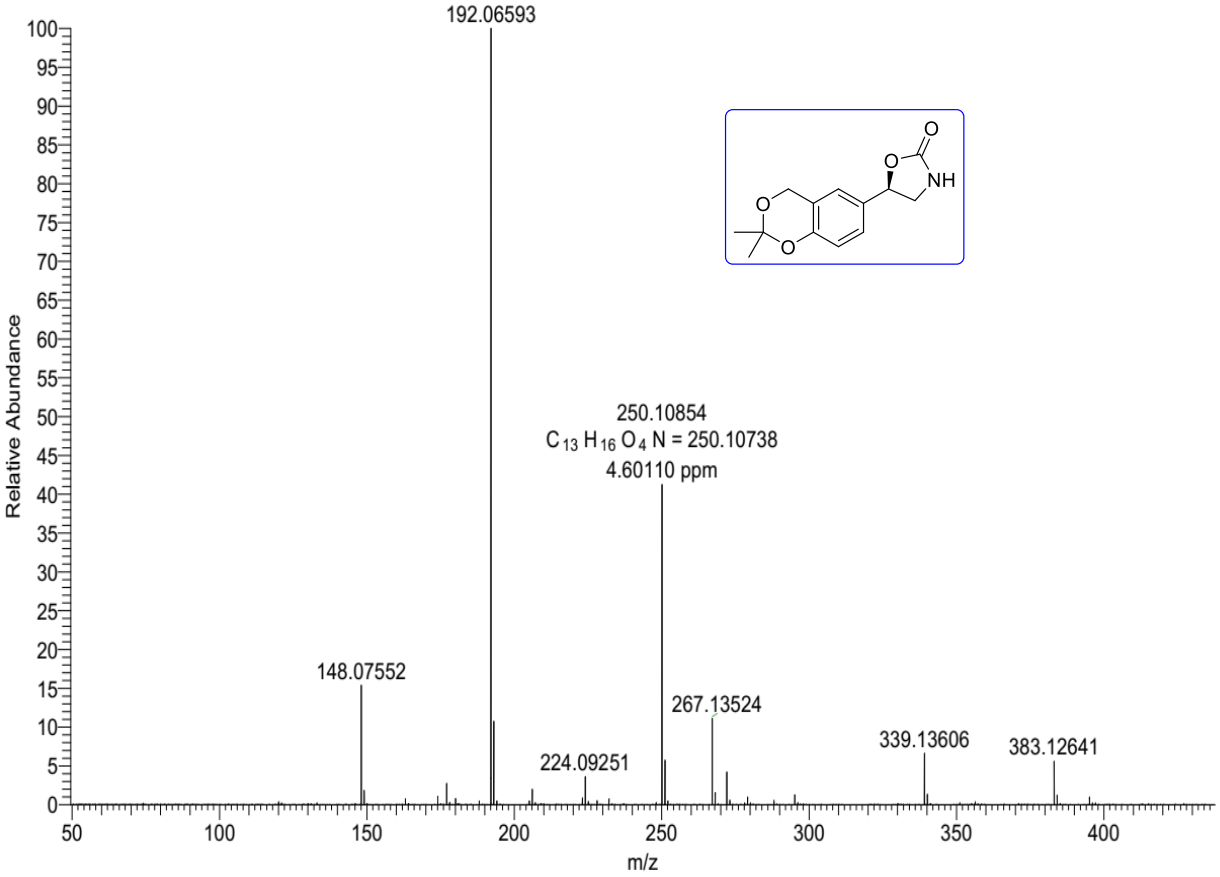

## Mass and HRMS (ESI) spectra of compound 10



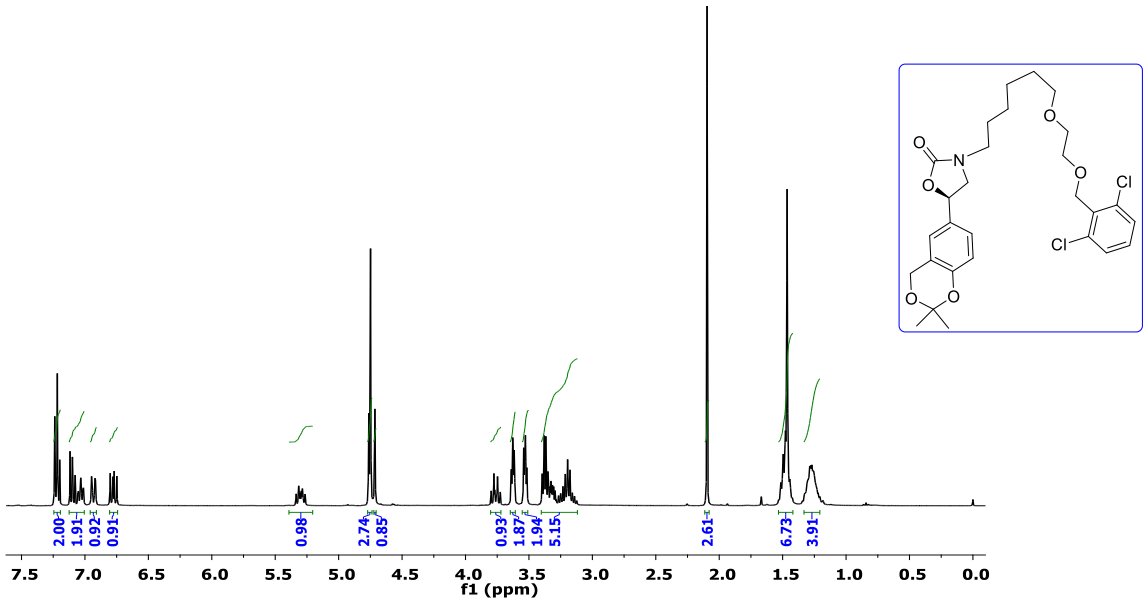

 

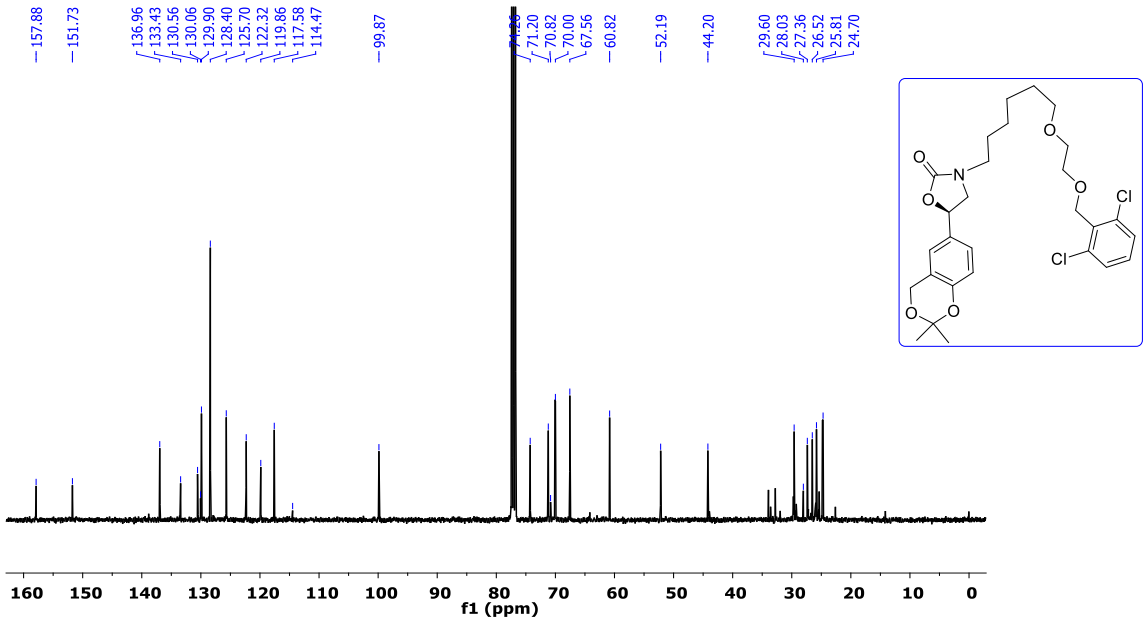
12/03/21 13:30:02
249
Thermo Scientific Orbitrap Exploris 120
Analysed by G SAIKRISHNA

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]

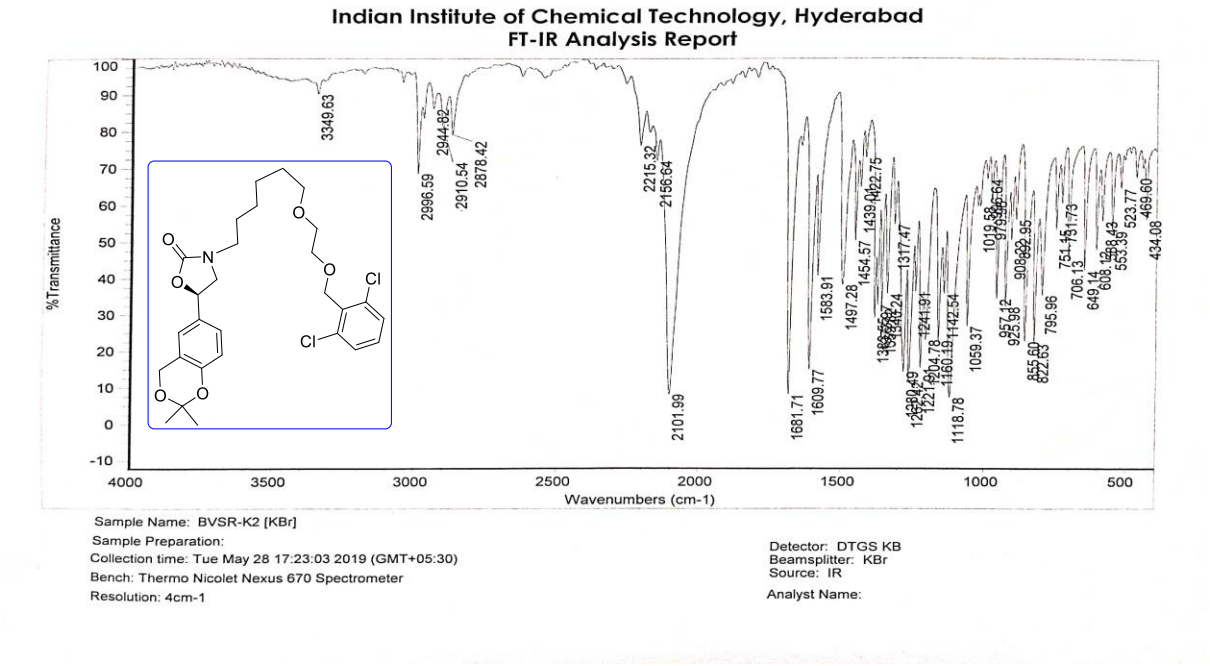


## $^{1}H$ (400 MHz, $CDCl_3$) and $^{13}C$ (101 MHz, $CDCl_3$) NMR spectra of compound 12

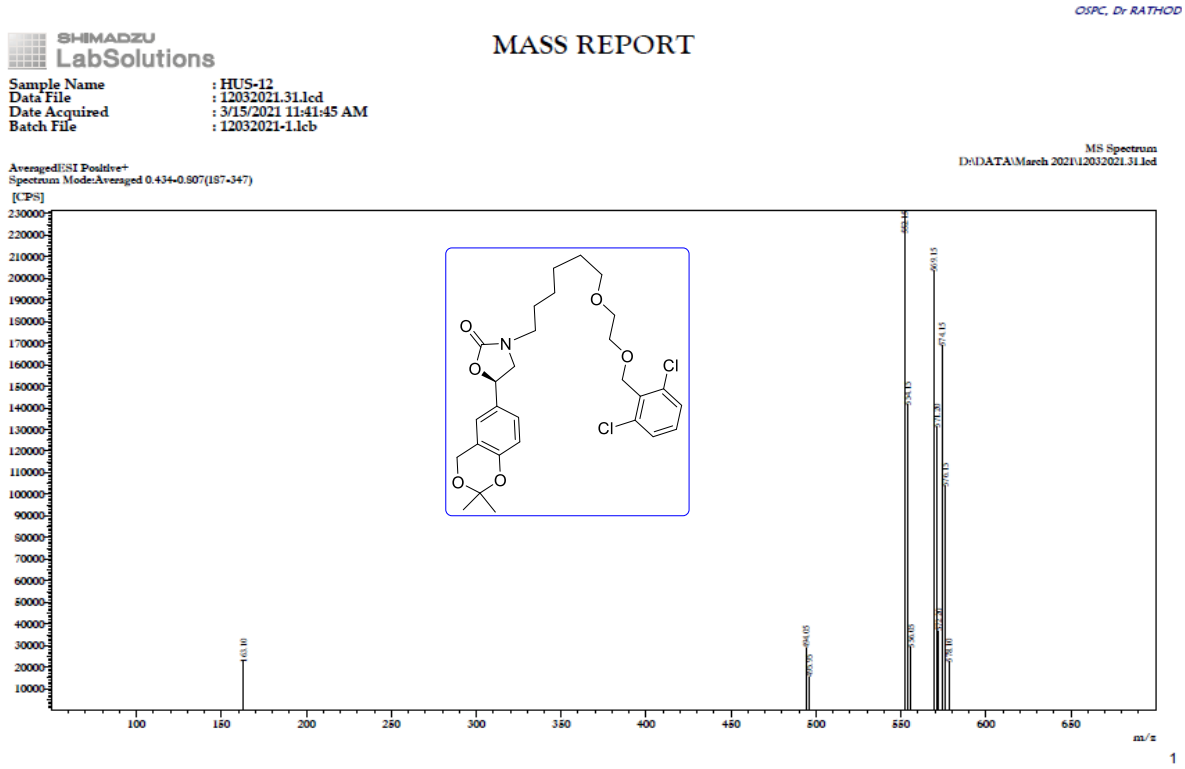


## IR spectrum of compound 12




## Mass and HRMS (ESI) spectra of compound 12



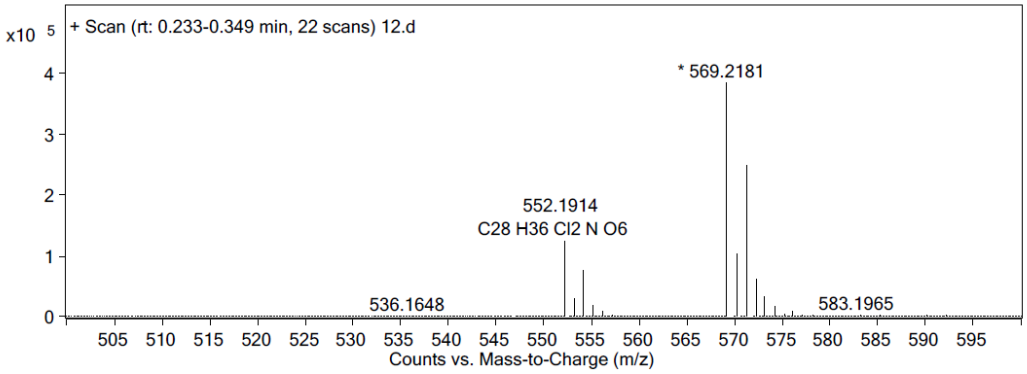

 

## Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **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** | | | |
| **Sample Group** | | **Info.** | |
| **Stream Name** | LC 1 | **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1) |

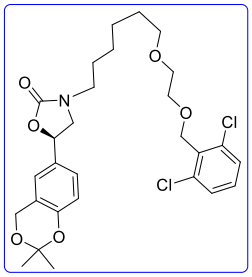
### User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 80 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund |
|---|---|---|
| 569.2181 | 1 | 384313.03 |



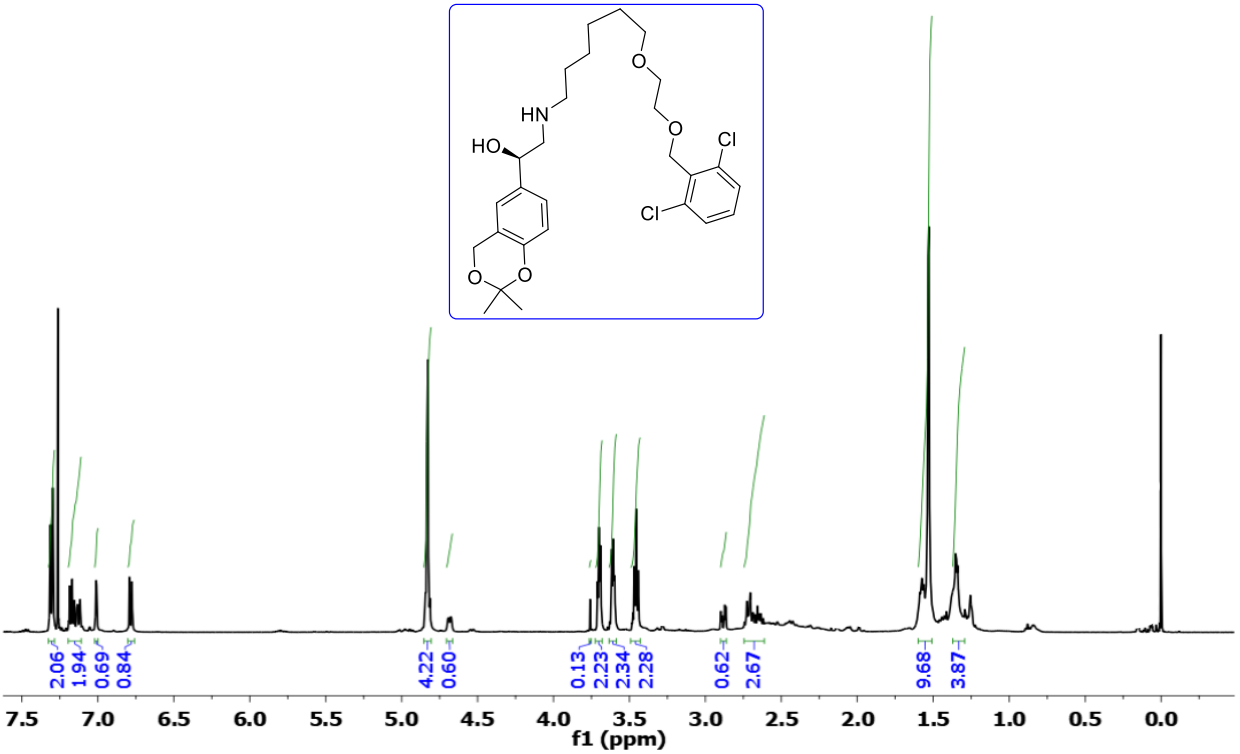
**Formula Calculator Element Limits**

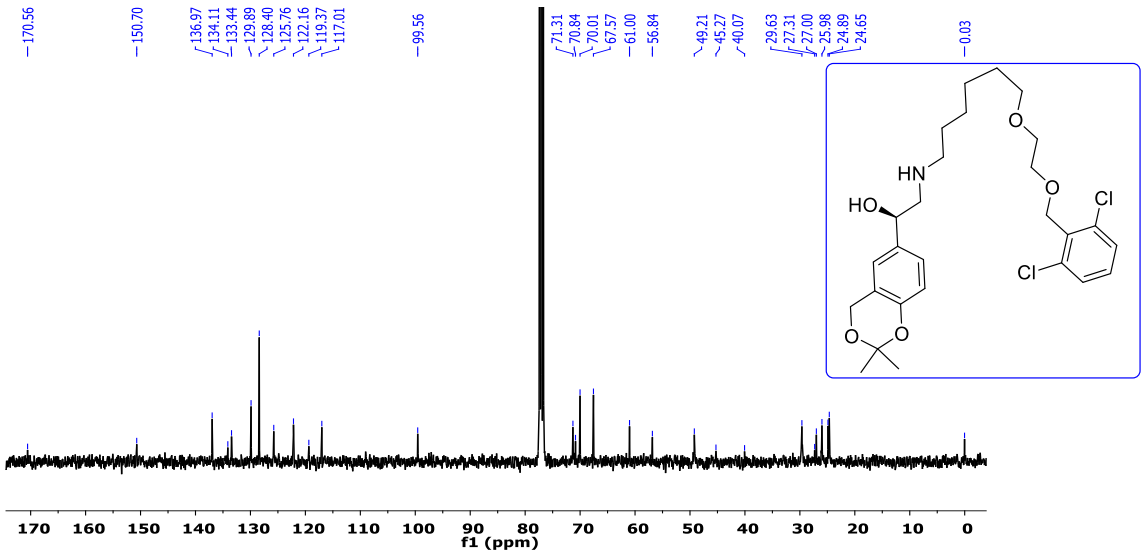
| Element | Min | Max |
|---|---|---|
| C | 0 | 30 |
| H | 0 | 60 |
| O | 0 | 6 |
| N | 0 | 1 |
| Cl | 0 | 2 |

**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 |

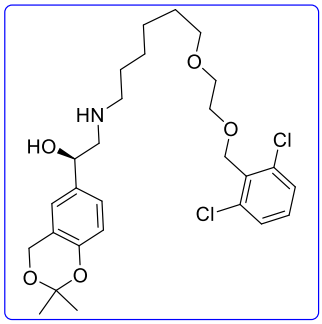
## $^{1}$H (500 MHz, $CDCl_3$) and $^{13}$C (101 MHz, $CDCl_3$) NMR spectra of compound 13

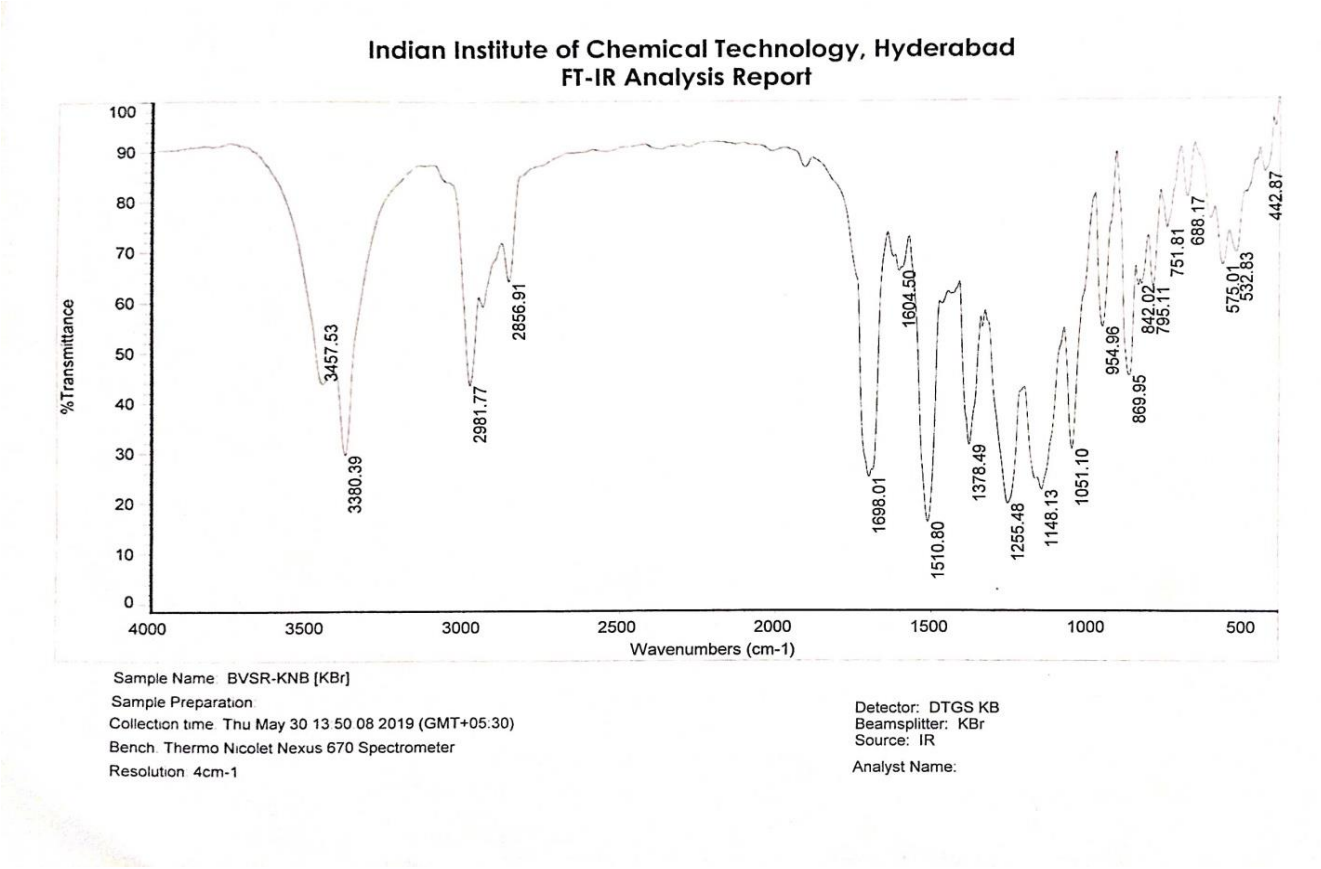


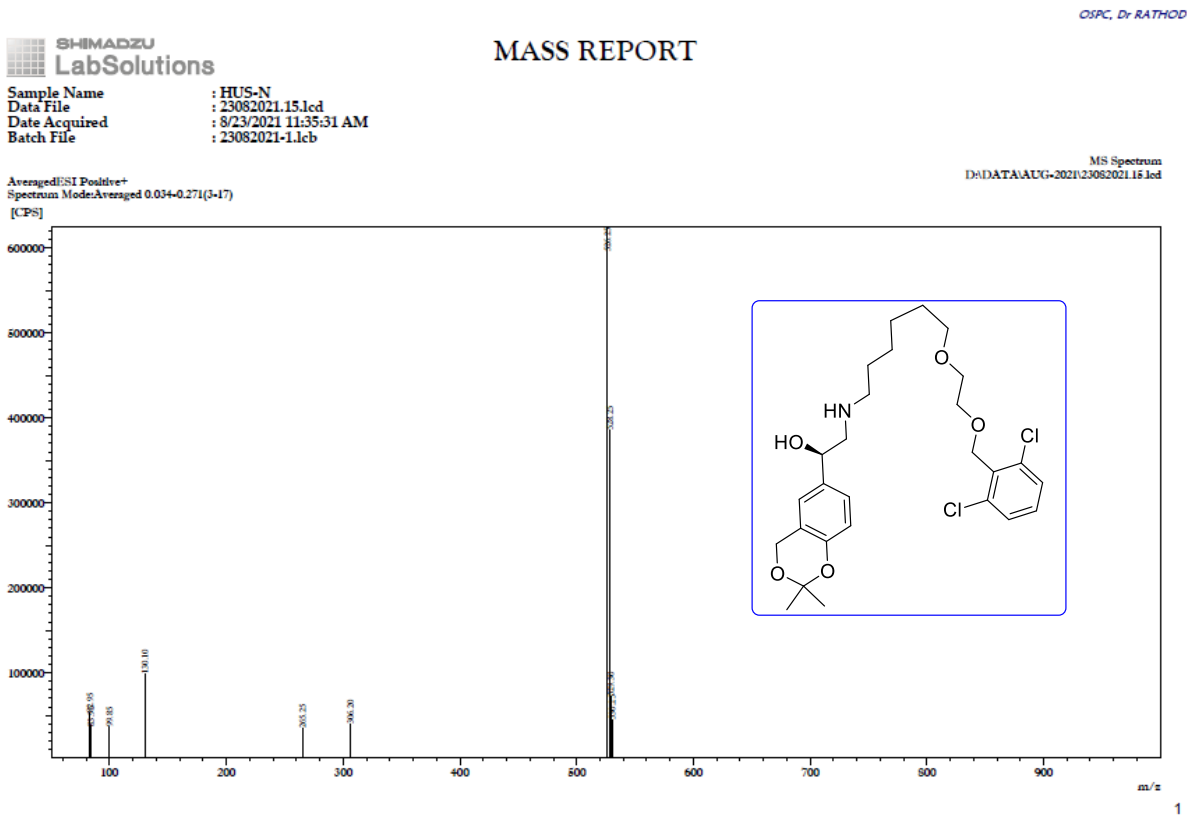
## IR spectrum of compound 13



## Mass and HRMS (ESI) spectra of compound 13

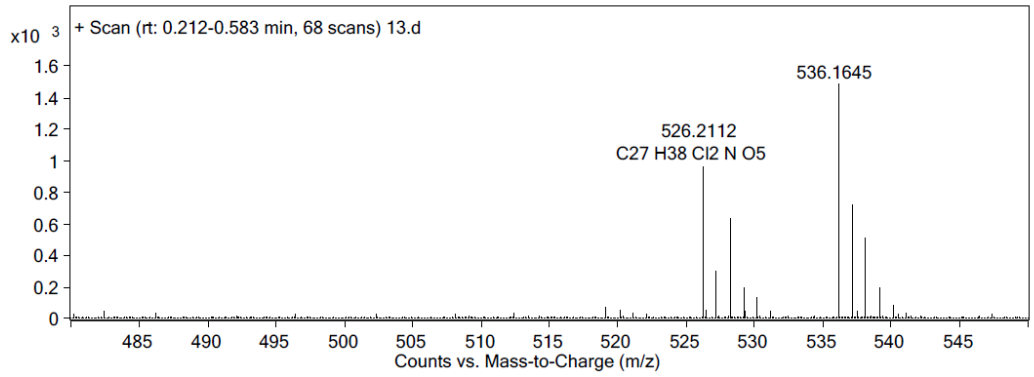


## Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **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** | | | |
| **Sample Group** | | **Info.** | |
| **Stream Name** | LC 1 | **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1) |

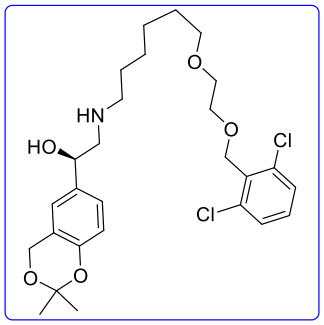
### User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 80 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund |
|---|---|---|
| 130.1587 | 1 | 16312.73 |



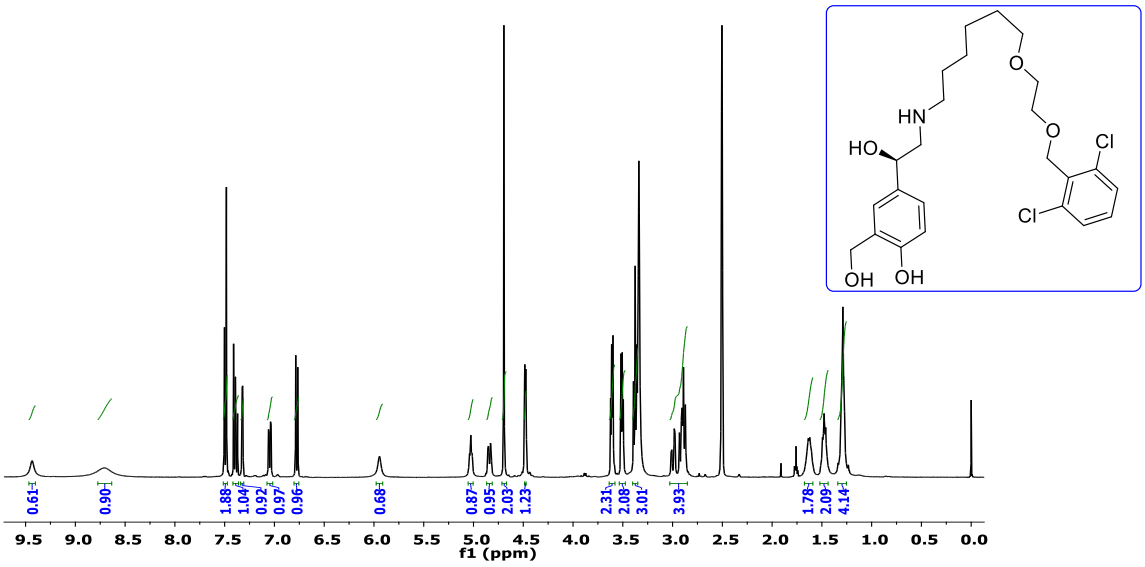

**Formula Calculator Element Limits**

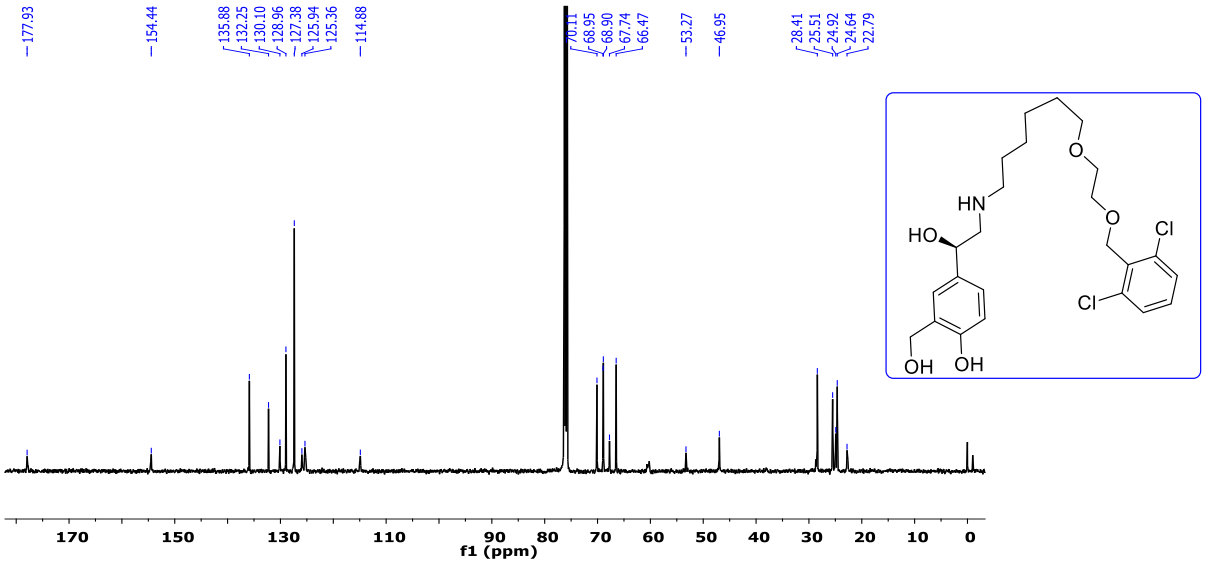
| Element | Min | Max |
|---|---|---|
| C | 0 | 30 |
| H | 0 | 60 |
| O | 0 | 6 |
| N | 0 | 1 |
| Cl | 0 | 2 |

**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 |

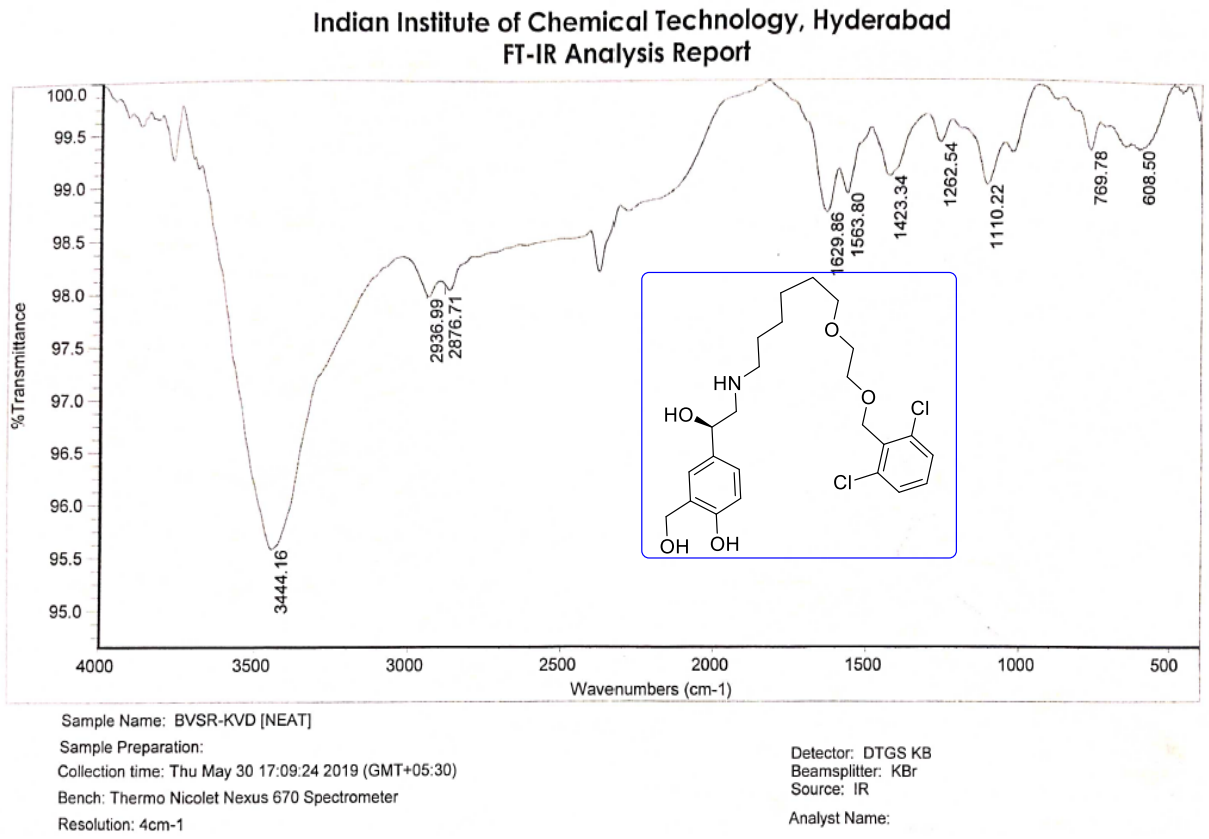
## $^{1}$H (400 MHz, DMSO) and $^{13}$C (126 MHz, $CDCl_3$) NMR spectra of compound 1



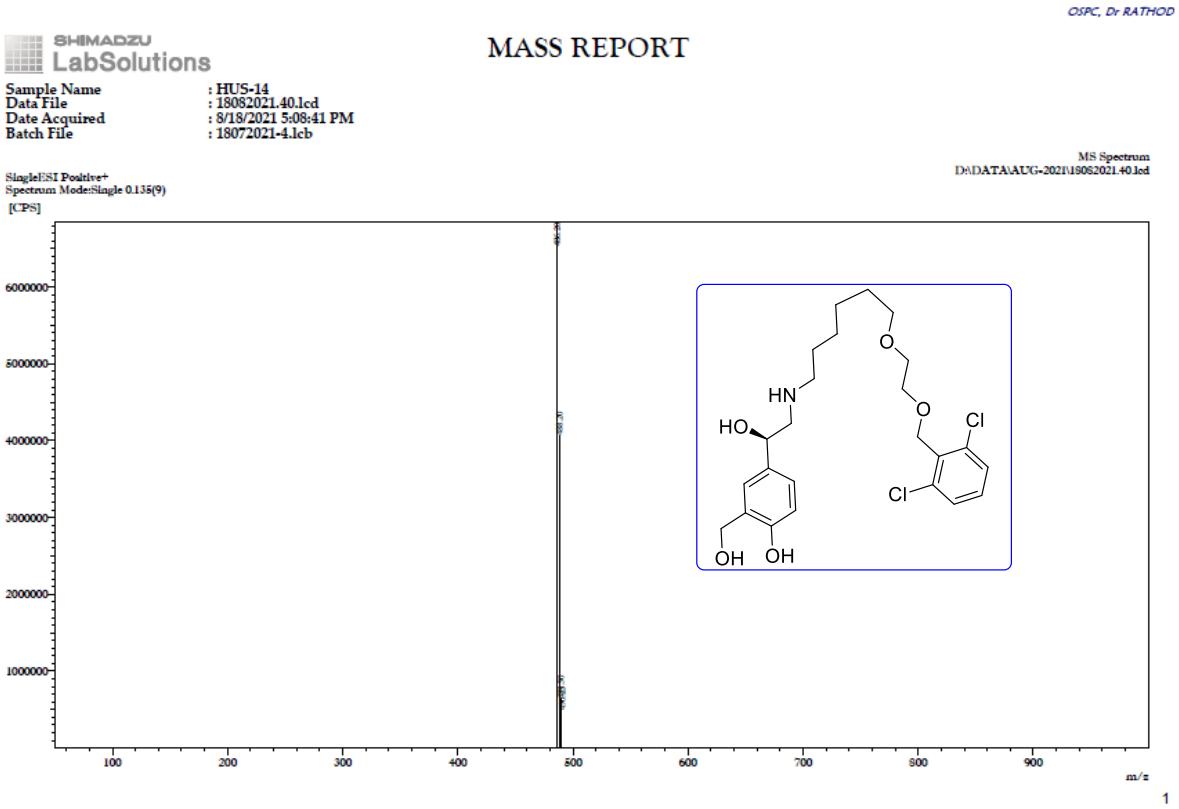

 

## IR spectrum of compound 1



## Mass and HRMS (ESI) spectra of compound 1



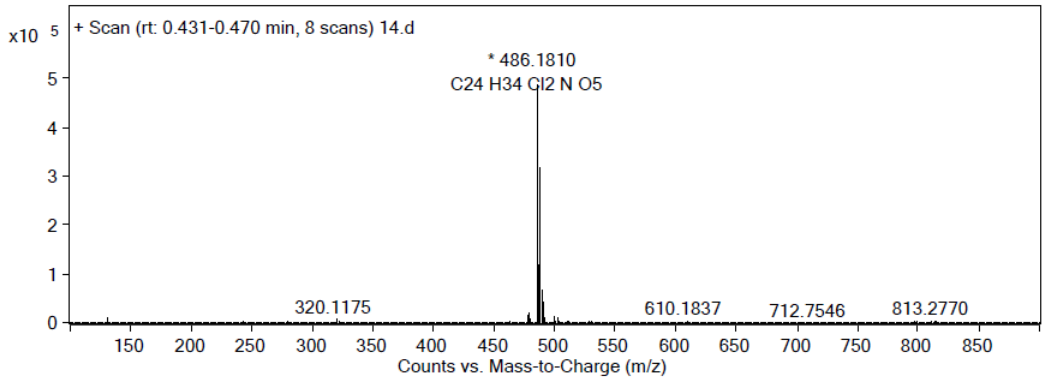

 

## Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data File** | 14.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 12:42:58 |
| **IRM Calibration Status** | Success | **DA Method** | 0.m |
| **Comment** | | | |
| **Sample Group** | | **Info.** | |
| **Stream Name** | LC 1 | **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.06.01 (B6172 SP1) |

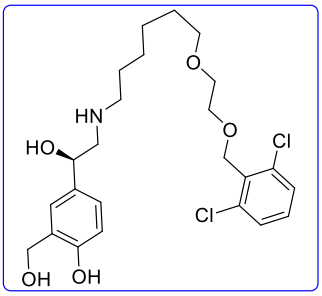
### User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 80 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 486.181 | 1 | 486429.88 | C24 H34 Cl2 N O5 | M+ |




**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 |
|---|---|---|---|---|---|---|
| C24 H34 Cl2 N O5 | True | 486.18137 | 486.1814 | 0.07 | C24 H34 Cl2 N O5 | 97.16 |
| C24 H33 Cl2 N O5 | True | 485.17355 | 485.17358 | 0.07 | C24 H34 Cl2 N O5 | 97.16 |

