

Green Synthesis of 1, 4-Benzodiazepines over La_2O_3 and $\text{La}(\text{OH})_3$ Catalysts:

Possibility of Langmuir-Hinshelwood adsorption

Archana Singh[#], Veerabhadraiah Palakollu[#], Aman Pandey, Sriram Kanvah* and Sudhanshu

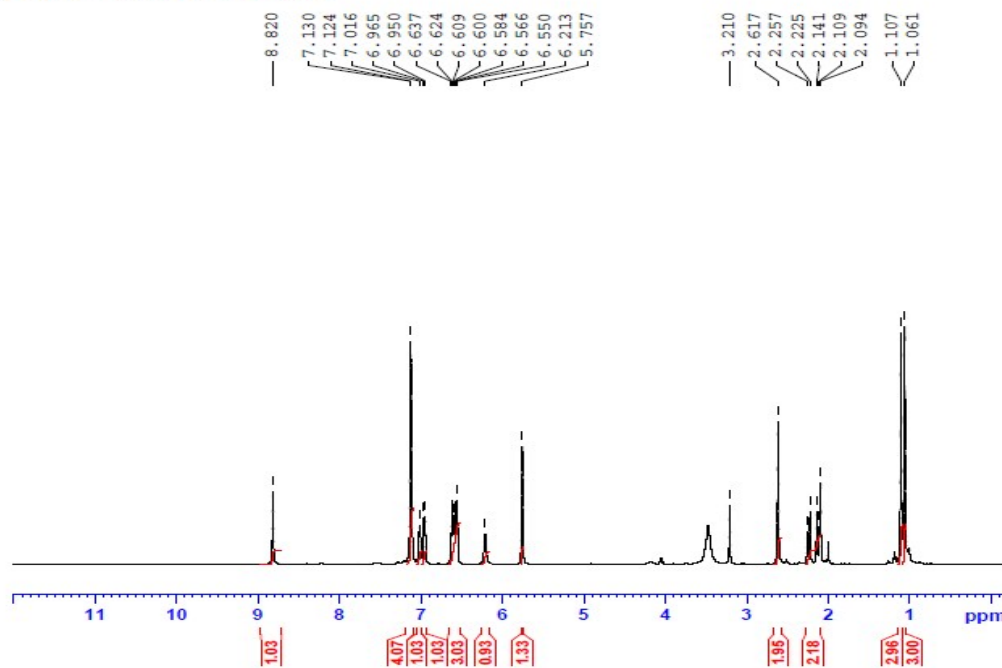
Sharma*

Department of Chemistry, Indian Institute of Technology Gandhinagar

Palaj, Gandhinagar 382355

^1H NMR Spectrum of 1,1-dimethyl-11-phenyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.

opda, benzaldehyde, dimidone



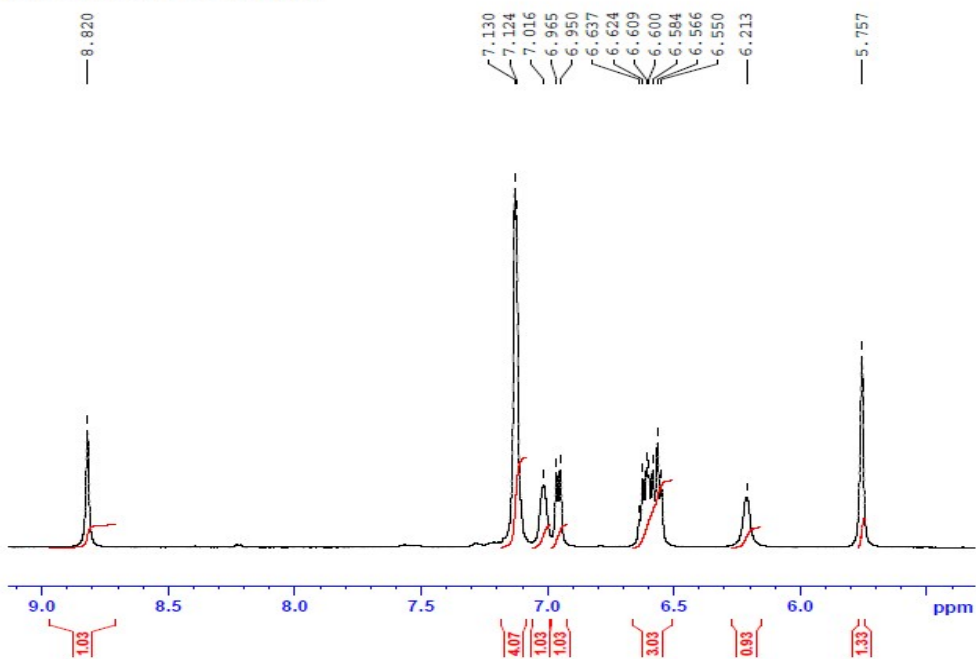
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EXPNO    1021
PROCNO   1

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PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       32
DS       2
SWH      10000.000 Hz
FIDRES   0.152588 Hz
AQ       3.2767999 sec
RG       32.16
DW       50.000 usec
DE       6.50 usec
TE       296.7 K
D1       1.00000000 sec
TDS      1

----- CHANNEL f1 -----
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NUC1    1H
P1      12.15 usec
PLW1    17.00000000 W

F2 - Processing parameters
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SF      500.0900000 MHz
WDW     EM
SSB     0
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PC      1.00
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opda, benzaldehyde, dimidone



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Current Data Parameters
NAME      Bhadra
EXPNO     1021
PROCNO    1

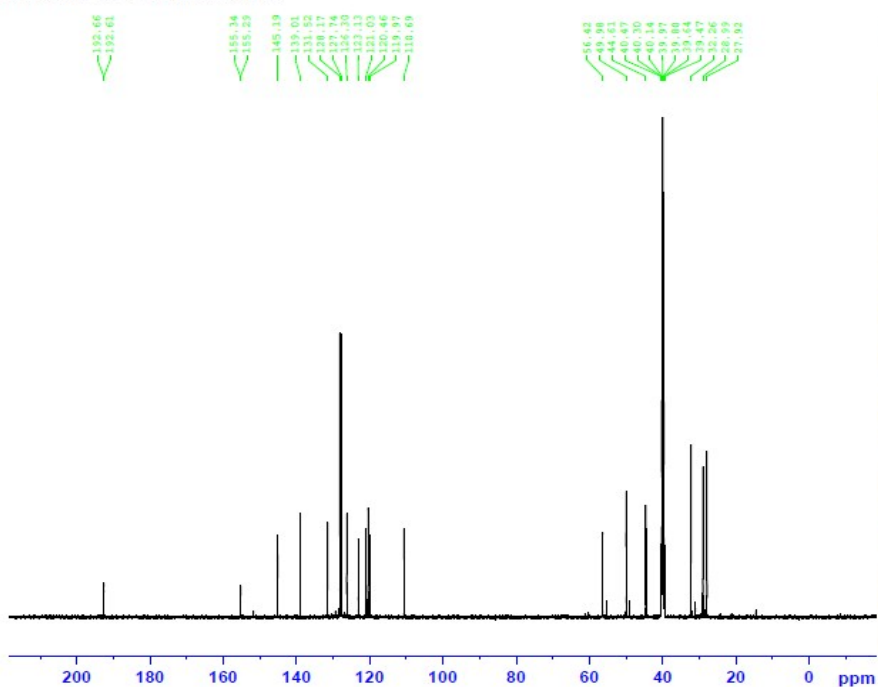
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PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         32
DS         2
SWH        10000.000 Hz
FIDRES     0.152588 Hz
AQ         3.2767999 sec
RG         32.16
DM         50.000 usec
DE         6.50 usec
TE         296.7 K
D1         1.00000000 sec
TD0        1

----- CHANNEL f1 -----
SFO1      500.0930883 MHz
NUC1       1H
P1         12.15 usec
PLW1       17.00000000 W

F2 - Processing parameters
SI         65536
SF         500.0900000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
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¹³C NMR Spectrum of 1,1-dimethyl-11-phenyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

benzaldehyde, dimidone, opda



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Current Data Parameters
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EXPNO     1064
PROCNO    1

F2 - Acquisition Parameters
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PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         2118
DS         4
SWH        29761.904 Hz
FIDRES     0.454121 Hz
AQ         1.1010048 sec
RG         112.97
DM         16.800 usec
DE         6.50 usec
TE         302.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

----- CHANNEL f1 -----
SFO1      125.7603047 MHz
NUC1       13C
P1         8.90 usec
PLW1       29.00000000 W

----- CHANNEL f2 -----
SFO2      500.0920004 MHz
NUC2       1H
CPDPRG12  waltz16
PCPD2     80.00 usec
PLW2       17.00000000 W
PLW12      0.52061999 W
PLW13      0.33320001 W

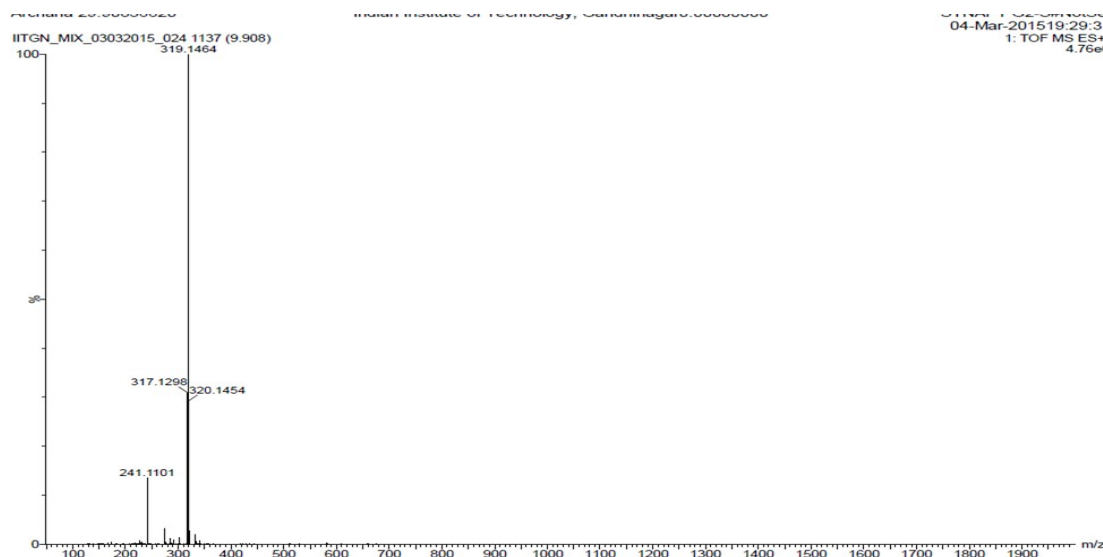
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WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
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Mass spectrum of 1,1-dimethyl-11-phenyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

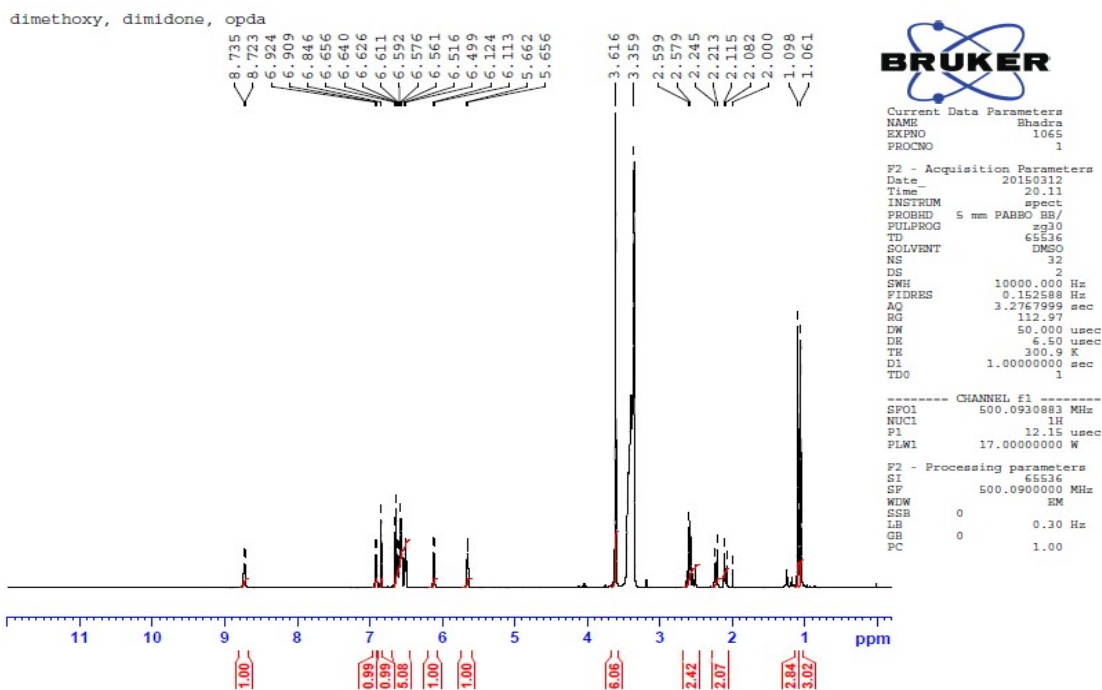
Exact mass: 318.4122

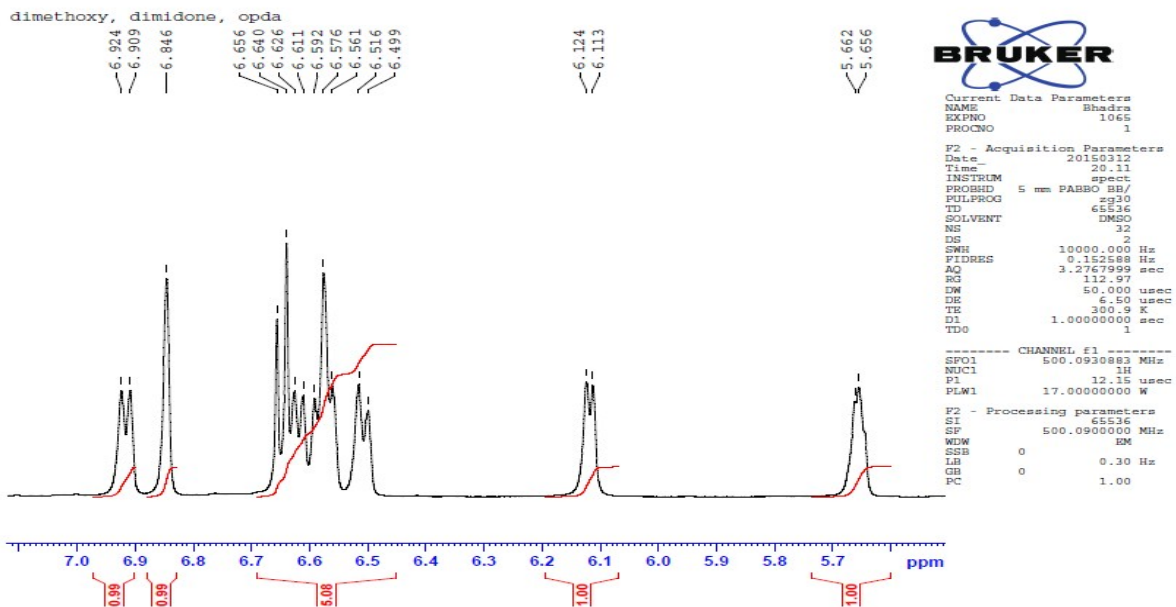
Mass obtained in the positive mode: 319.1464

Elemental composition: C₂₁H₂₂N₂O

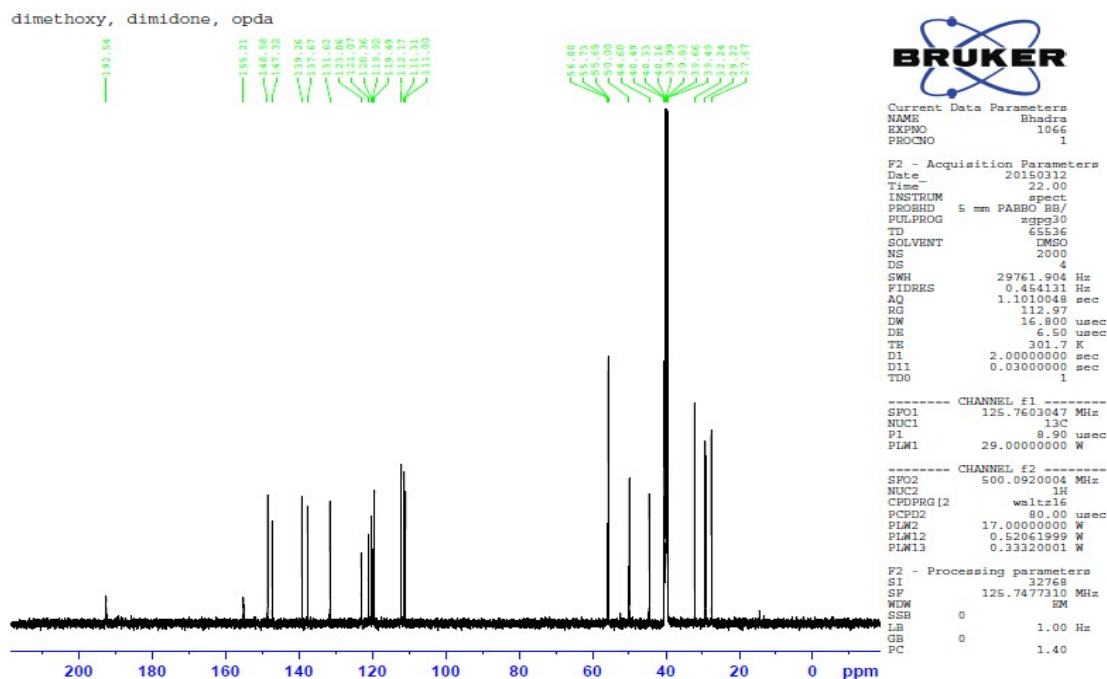


¹H NMR Spectrum of 11-(4-(dimethylamino)phenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one





¹³C NMR Spectrum of 11-(4-(dimethylamino)phenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

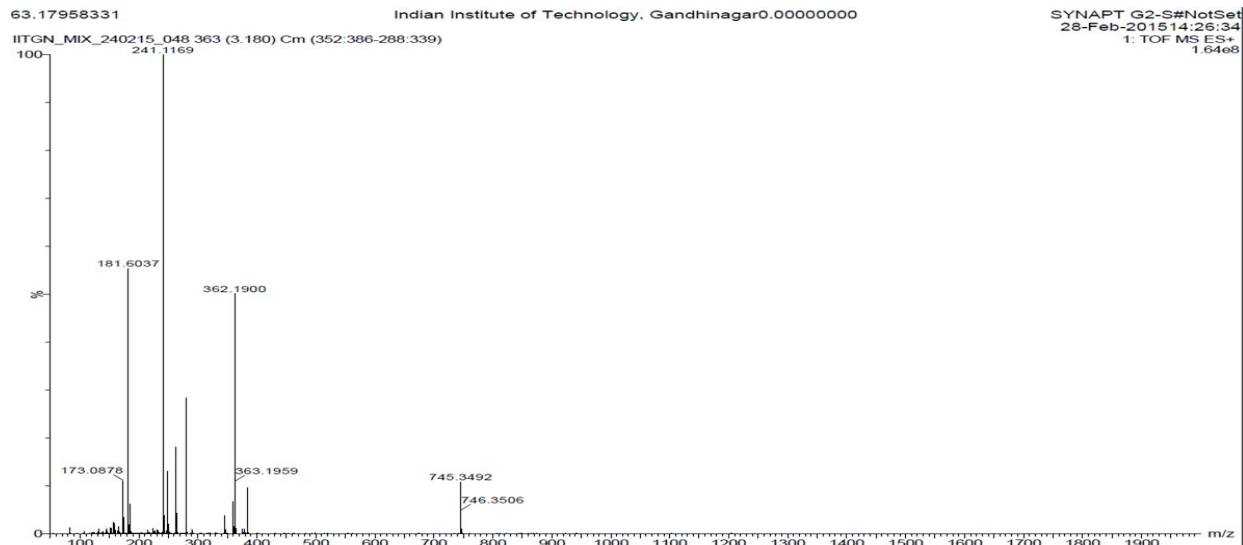


Mass spectrum of 11-(4-(dimethylamino)phenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

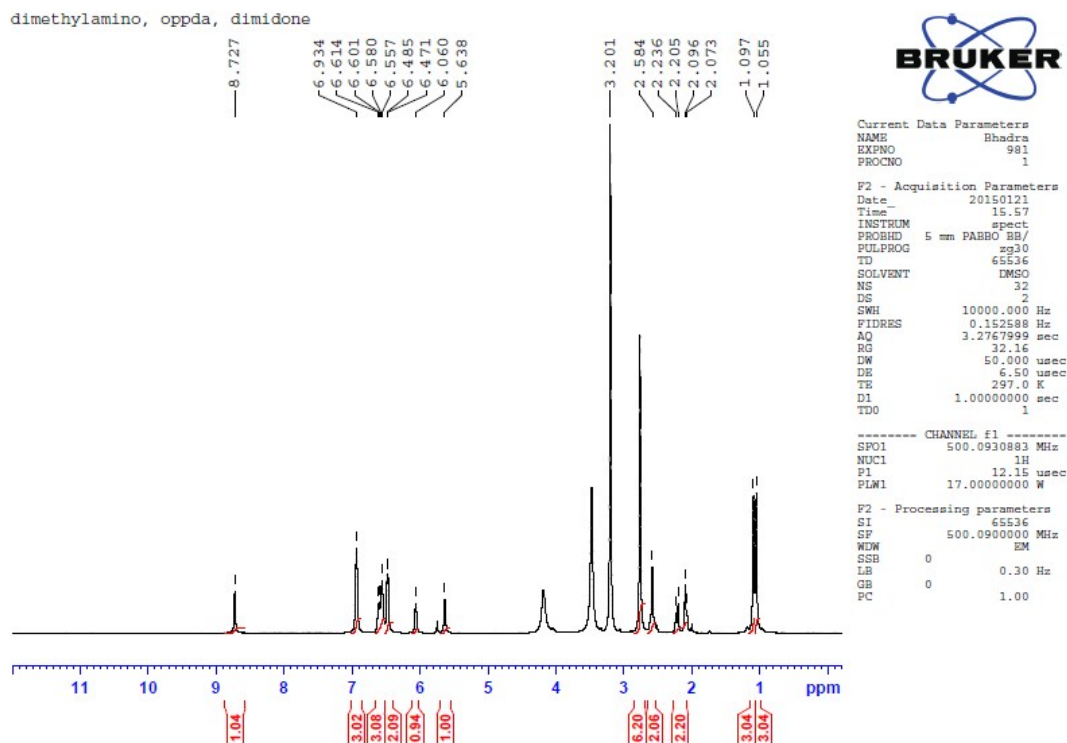
Exact mass: 361.2154

Mass obtained in the positive mode: 362.19

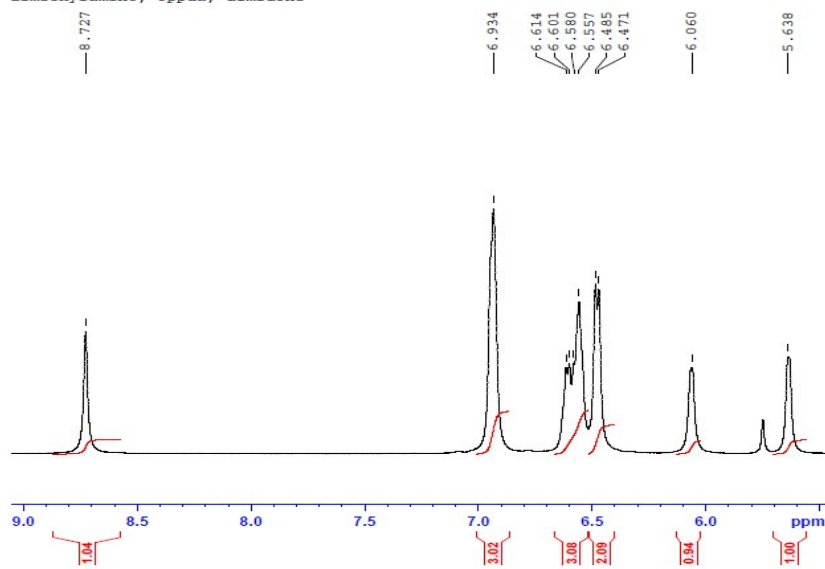
Elemental composition: C₂₃H₂₇N₃O



¹H NMR Spectrum of 11-(2,3-dimethoxyphenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



dimethylamino, oppda, dimidone



```
Current Data Parameters
NAME          Bhadra
EXPNO         981
PROCNO        1

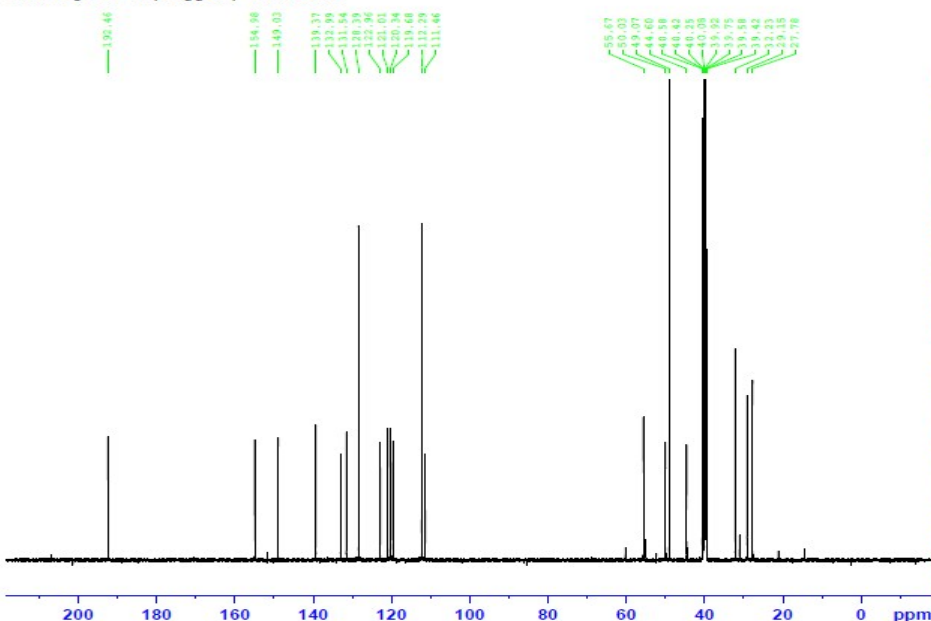
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PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            32
DS            4
SWH           10000.000 Hz
FIDRES        0.15288 Hz
AQ            3.2767999 sec
RG            32.16
DM            50.000 usec
DE            6.50 usec
TE            297.0 K
D1            1.00000000 sec
TD0           1

----- CHANNEL f1 -----
SFO1          500.093083 MHz
NUC1           1H
P1            12.15 usec
PLW1          17.00000000 W

F2 - Processing parameters
SI            65536
SF            500.0900000 MHz
WDW           RM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

¹³C NMR Spectrum of 11-(2,3-dimethoxyphenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

dimethylamino, oppda, dimidone



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Current Data Parameters
NAME          Bhadra
EXPNO         982
PROCNO        1

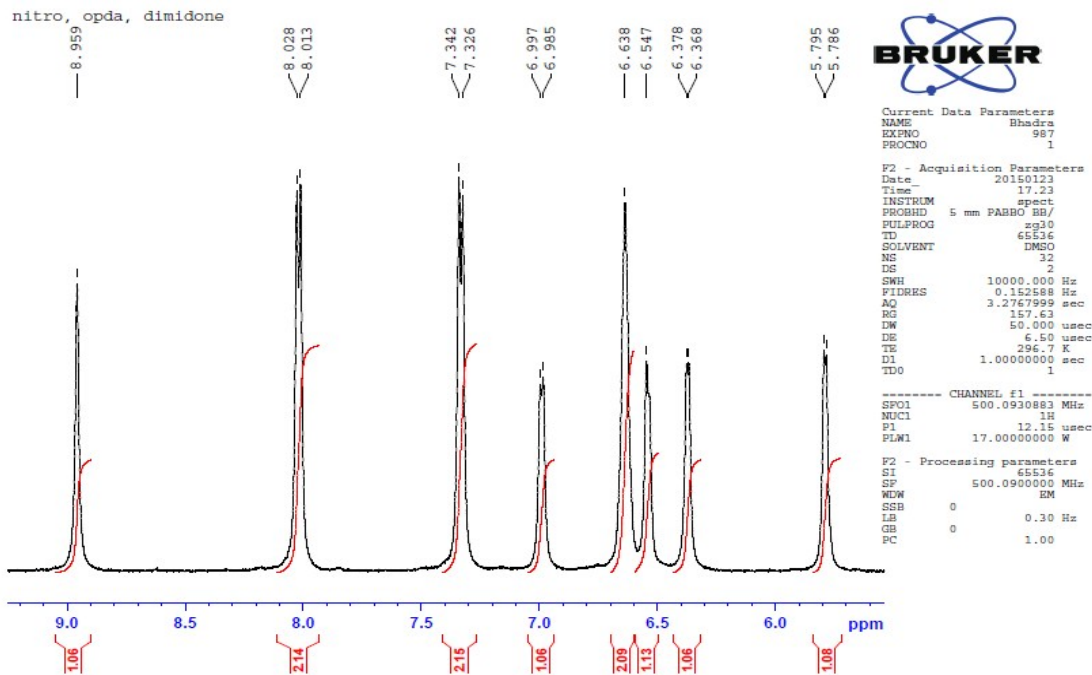
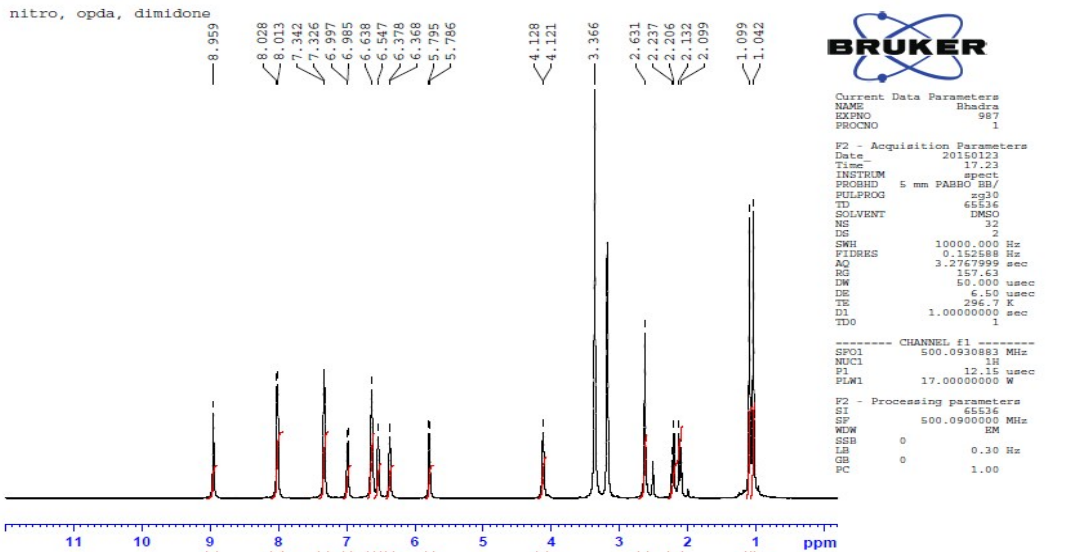
F2 - Acquisition Parameters
Date_         20150121
Time          16.55
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG            112.97
DM            16.800 usec
DE            6.50 usec
TE            298.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

----- CHANNEL f1 -----
SFO1          125.7603047 MHz
NUC1           13C
P1            8.90 usec
PLW1          29.00000000 W

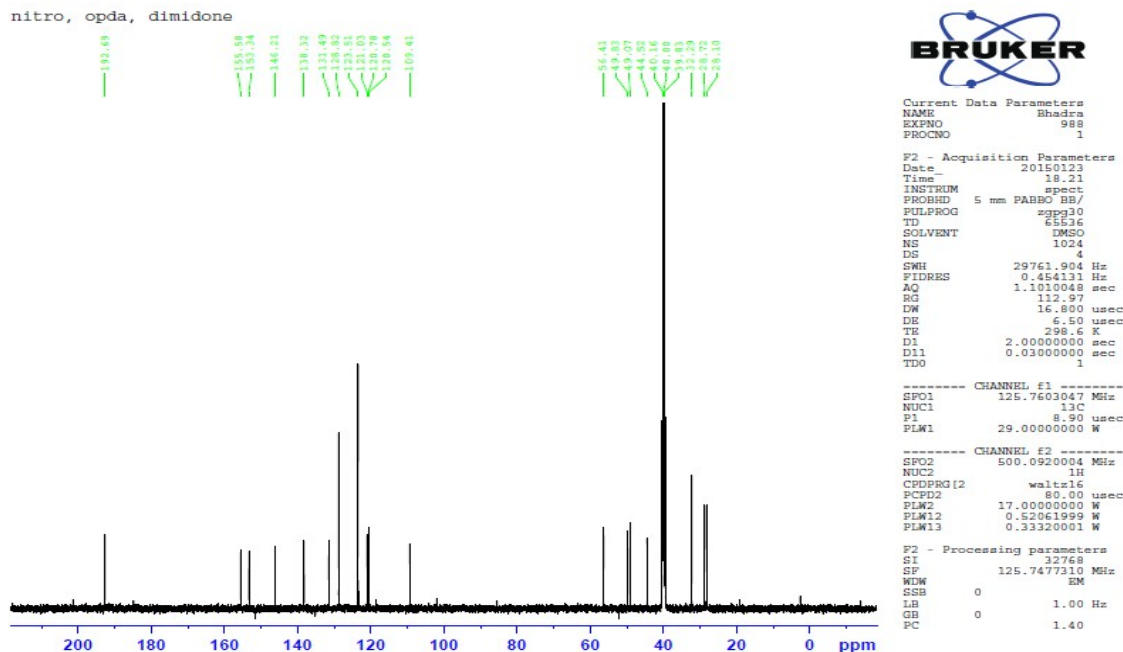
----- CHANNEL f2 -----
SFO2          500.0920004 MHz
NUC2           1H
CPCPD2        waltz16
PCPD2         80.00 usec
PLW2          17.00000000 W
PLW12         0.52061999 W
PLW13         0.33320001 W

F2 - Processing parameters
SI            32768
SF            125.7477310 MHz
WDW           RM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

¹H NMR Spectrum of 1,1-dimethyl-11-(4-nitrophenyl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



¹³C NMR Spectrum of 1,1-dimethyl-11-(4-nitrophenyl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



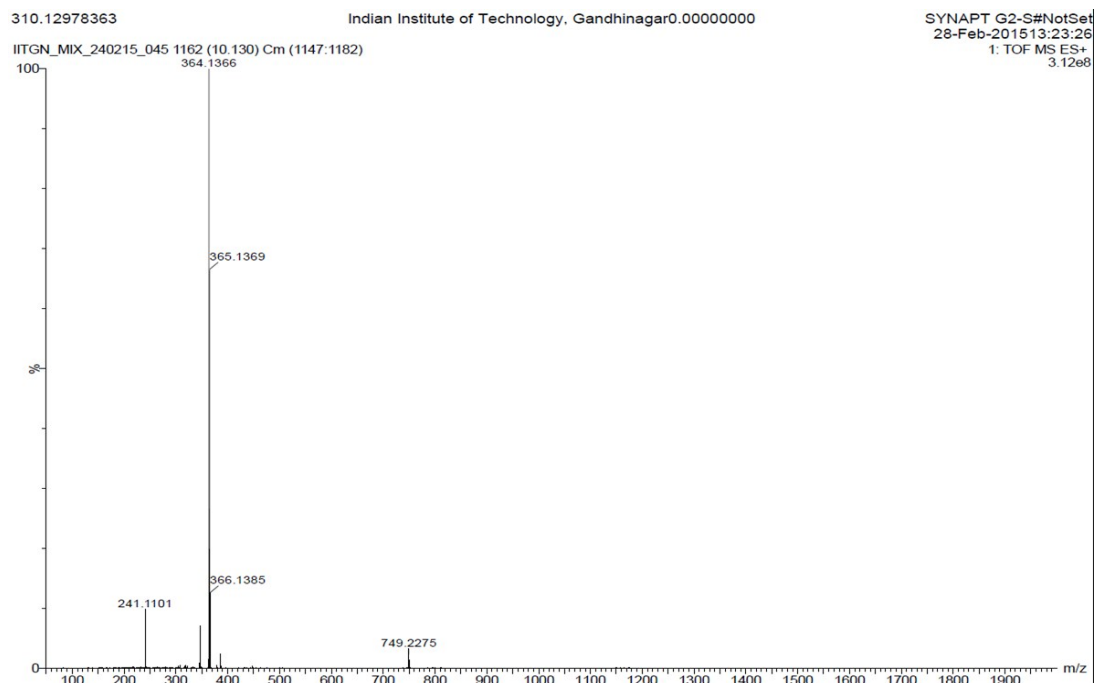
Ma

Mass spectrum of 1,1-dimethyl-11-(4-nitrophenyl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

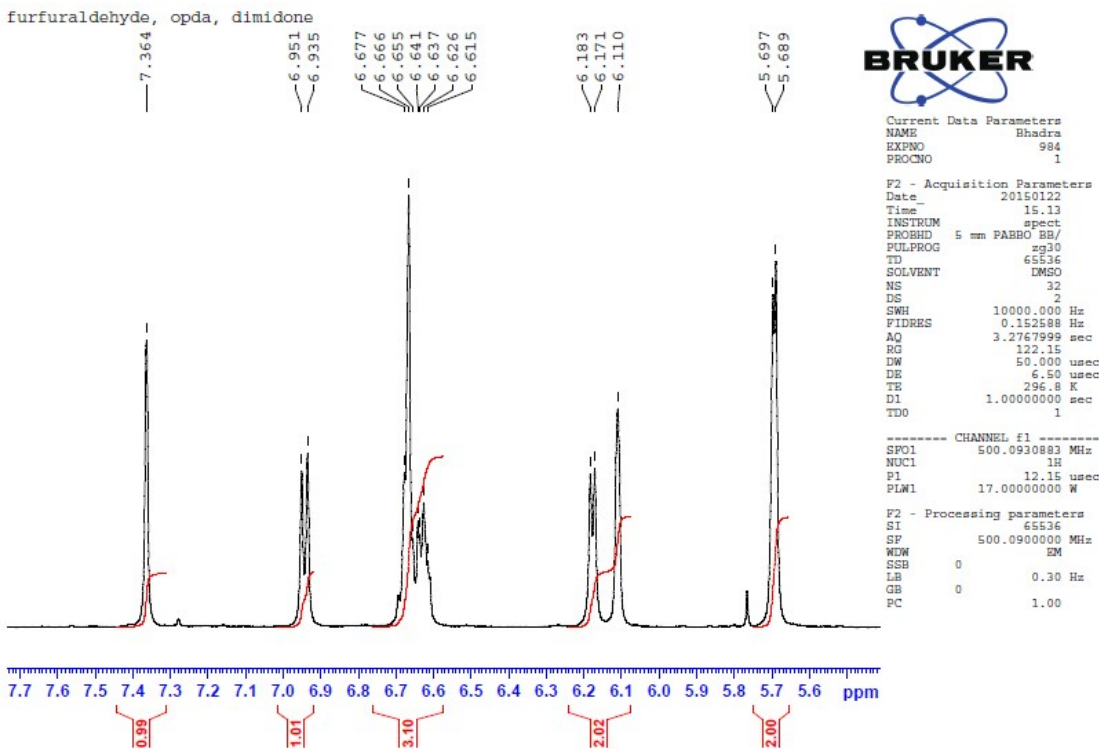
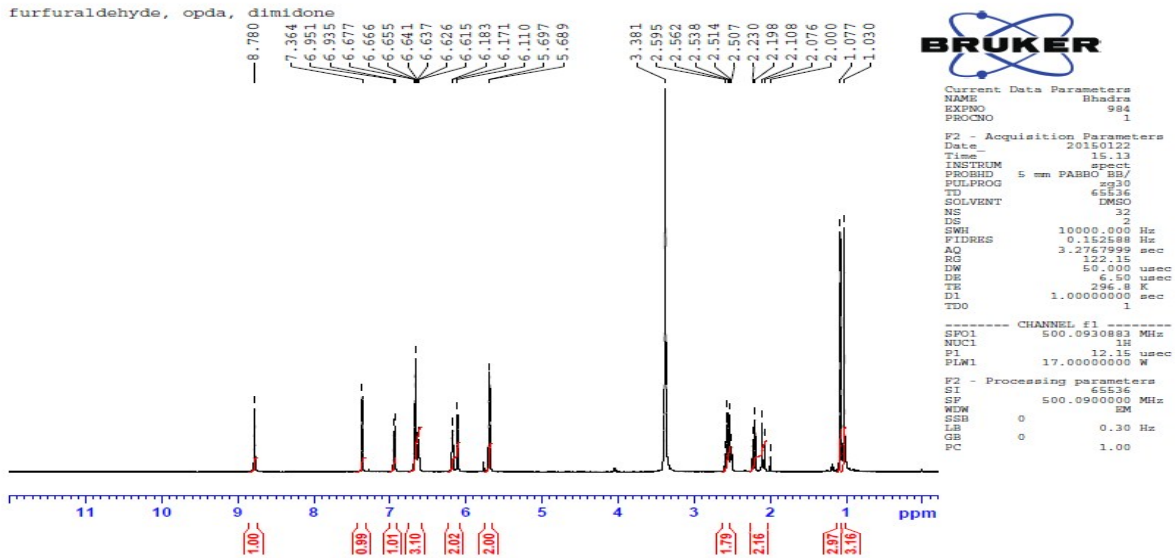
Exact mass: 363.1583

Mass obtained in the positive mode: 364.1366

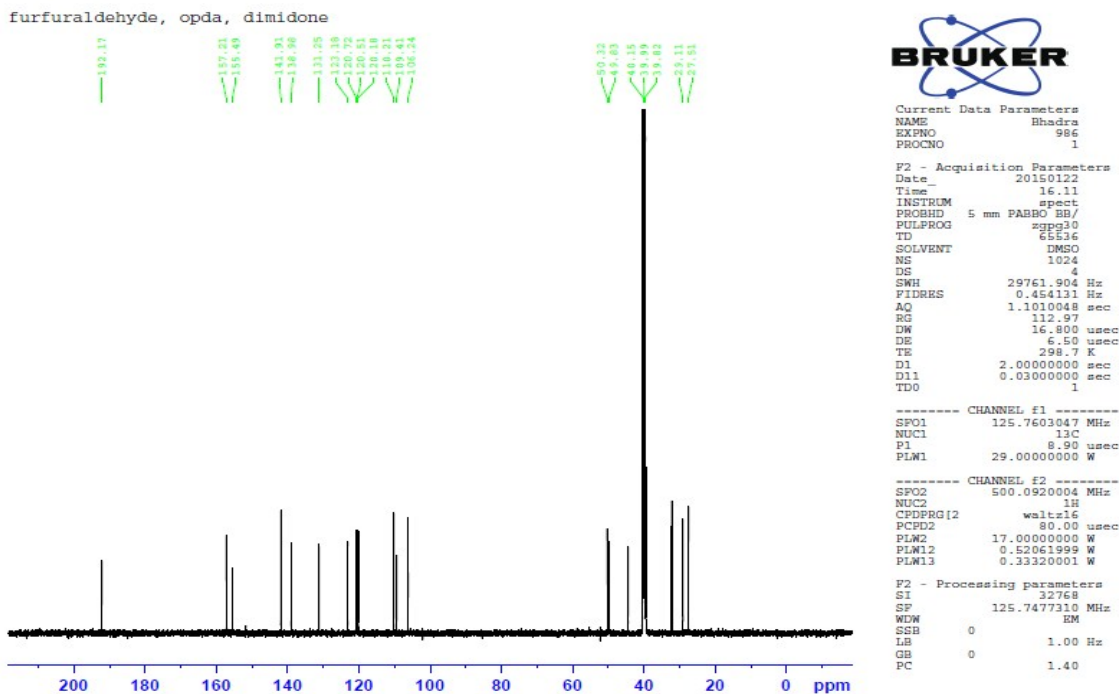
Elemental composition: C₂₁H₂₁N₃O₃



¹H NMR Spectrum of 11-(furan-2-yl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



¹³C NMR Spectrum of 11-(furan-2-yl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



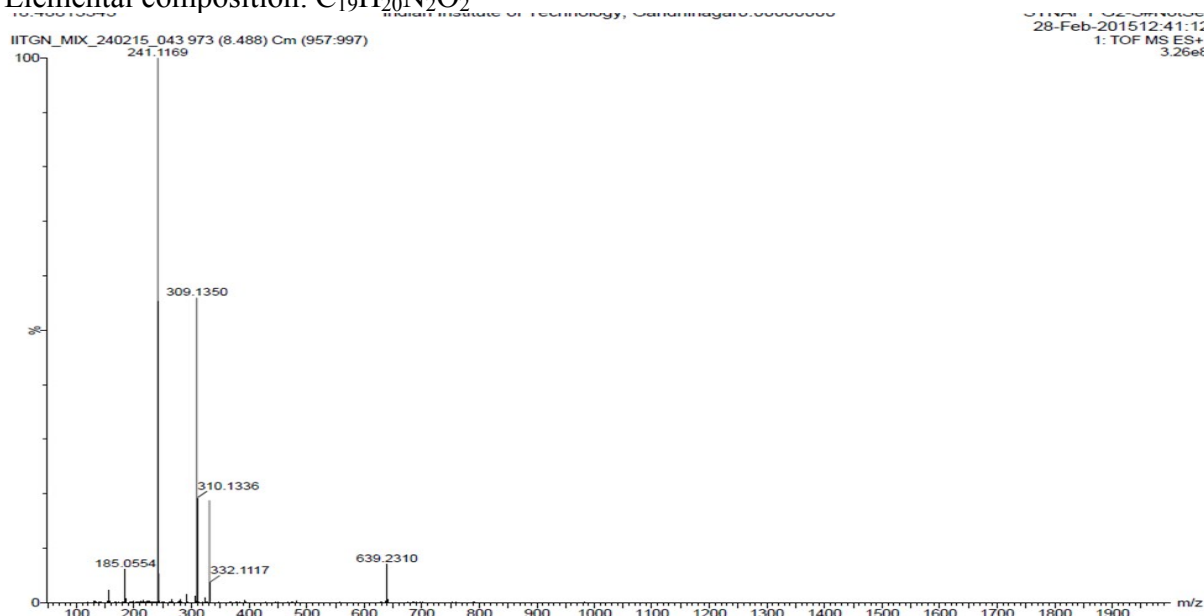
M

Mass spectrum of 11-(furan-2-yl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.

Exact mass: 308.1525

Mass obtained in the positive mode: 309.1350

Elemental composition: C₁₉H₂₀N₂O₂

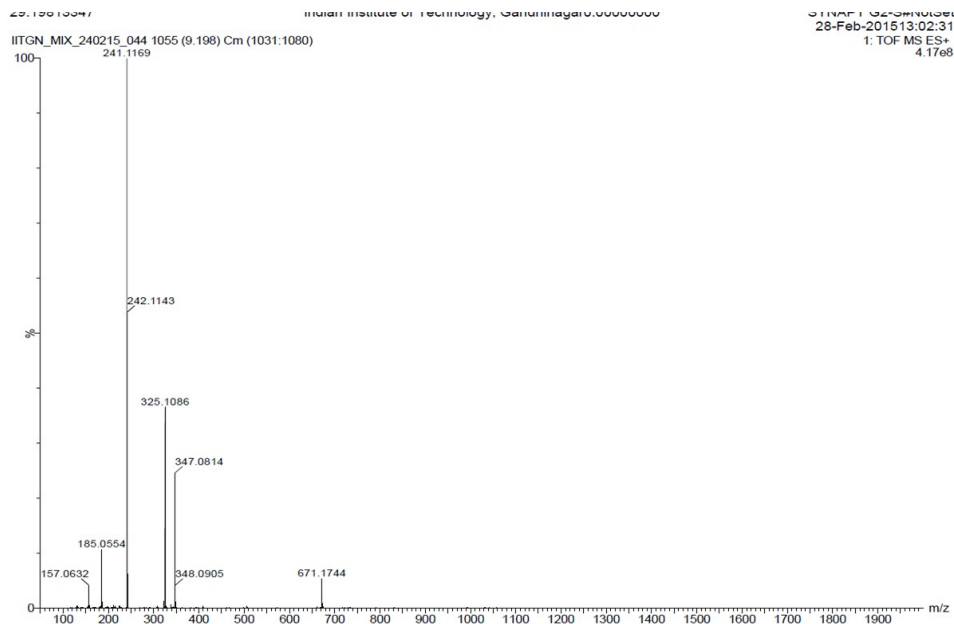


Mass spectrum of 1,1-dimethyl-11-(thiophen-2-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

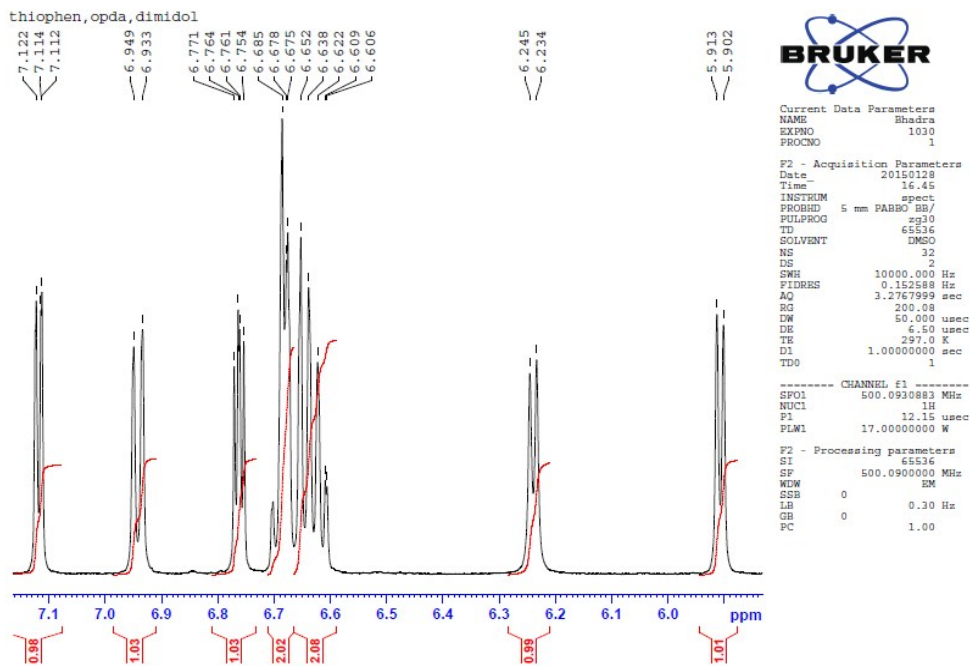
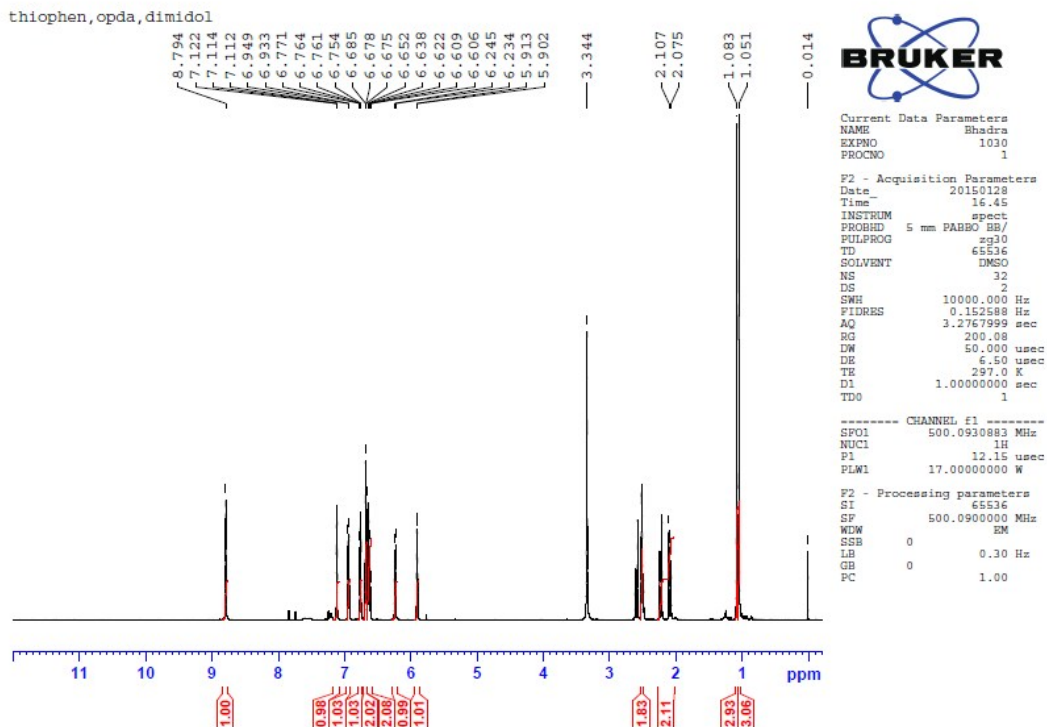
Exact mass: 325.1086

Mass obtained in the positive mode: 325.1086

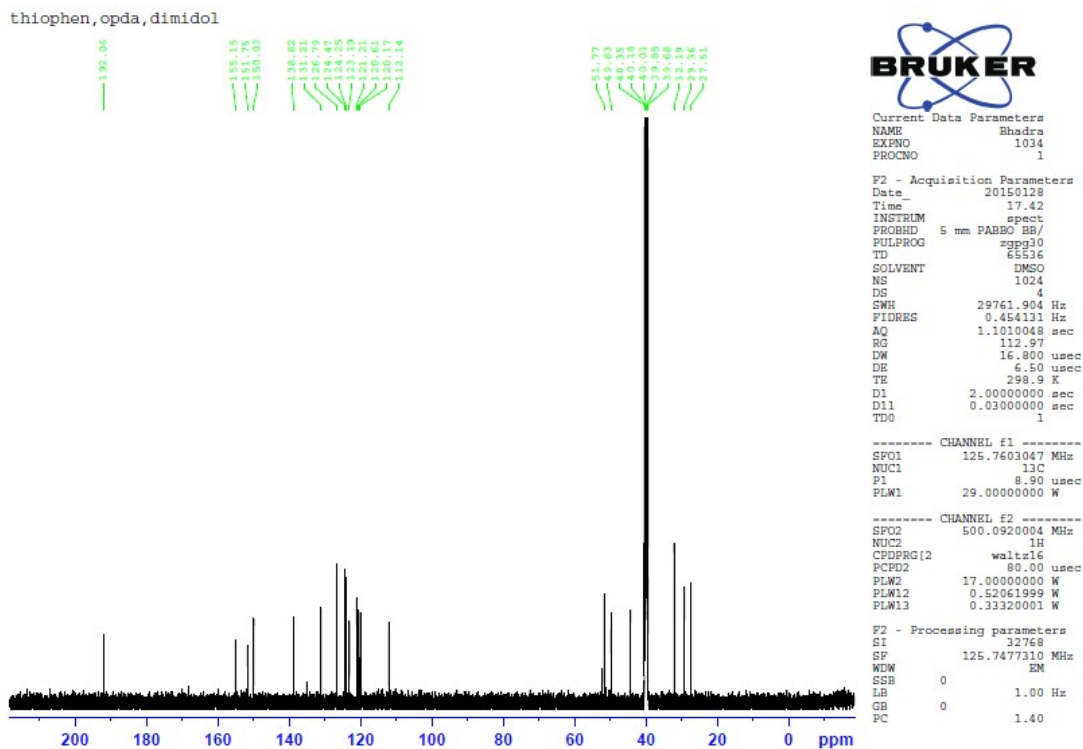
Elemental composition: C₁₉H₂₀N₂O_S



¹H NMR Spectrum of 1,1-dimethyl-11-(thiophen-2-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



¹³C NMR Spectrum of 1,1-dimethyl-11-(thiophen-2-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



Mass spectrum of 1,1-dimethyl-11-(thiophen-2-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

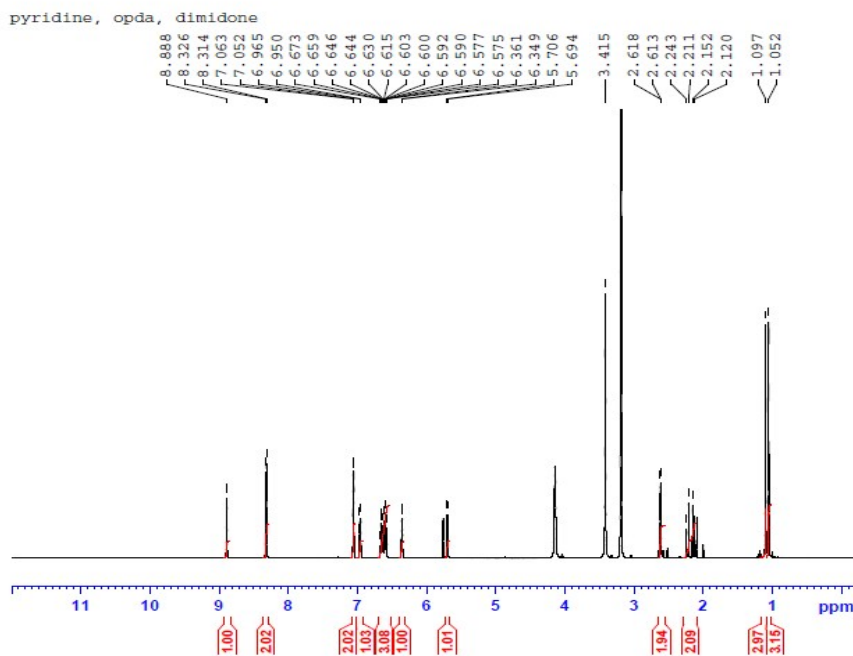
Exact mass: 325.1086

Mass obtained in the positive mode: 325.1086

Elemental composition: C₁₉H₂₀N₂O_S



¹H NMR Spectrum of 1,1-dimethyl-11-(pyridin-4-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



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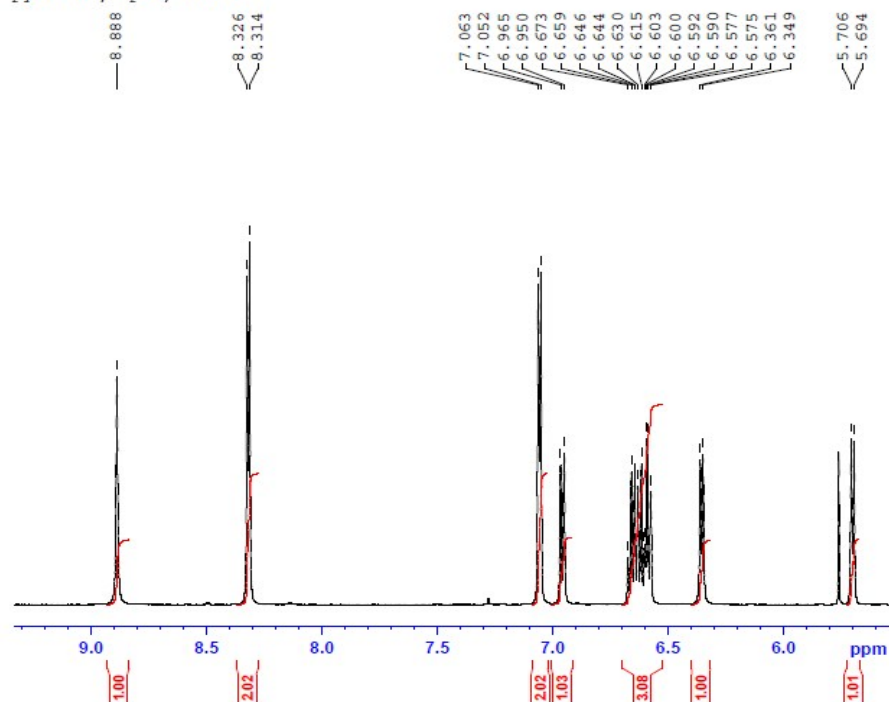
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PROCNO        1

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PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            12
DS            2
SWH           10000.000 Hz
FIDRES        0.152568 Hz
AQ            3.2767999 sec
RG            71.17
DW            50.000 usec
DE            6.50 usec
TE            296.7 K
D1            1.00000000 sec
TDO           1

----- CHANNEL f1 -----
SFO1          500.0930983 MHz
NUC1           1H
P1            12.15 usec
PLW1          17.00000000 W

F2 - Processing parameters
SI            65536
SP            500.0900000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
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pyridine, opda, dimidone



Current Data Parameters
NAME Bhadra
EXPNO 994
PROCNO 1

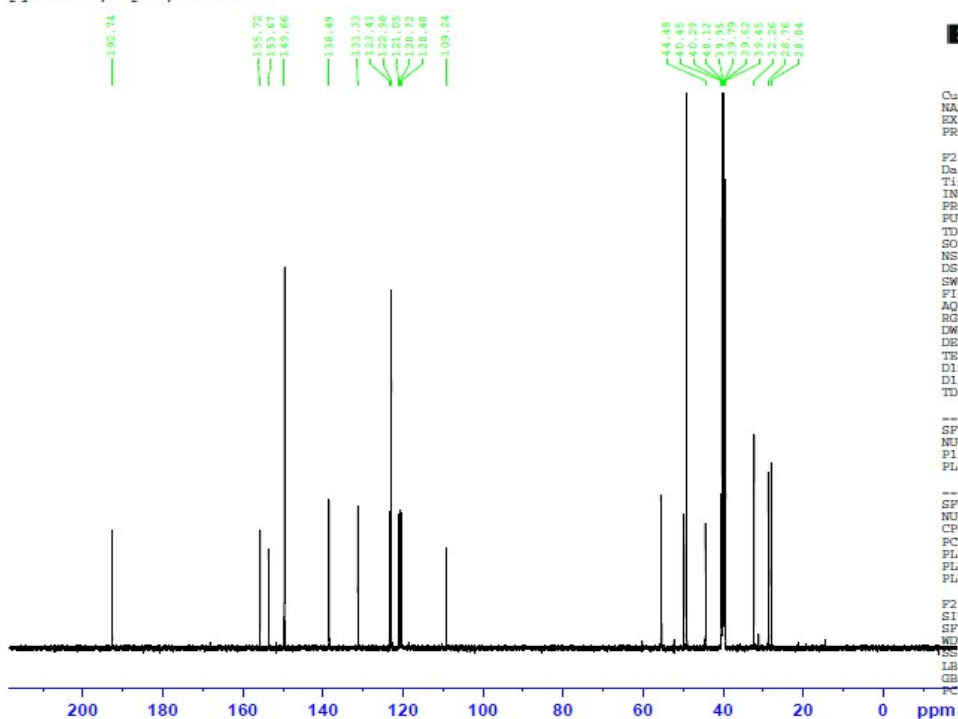
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Time 14.47
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PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 71.17
DW 50.000 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 500.0930883 MHz
NUC1 1H
P1 12.15 usec
PLW1 17.00000000 W

F2 - Processing parameters
SI 65536
SF 500.0900000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR Spectrum of 1,1-dimethyl-11-(pyridin-4-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.

pyridine, opda, dimidone



Current Data Parameters
NAME Bhadra
EXPNO 99
PROCNO 1

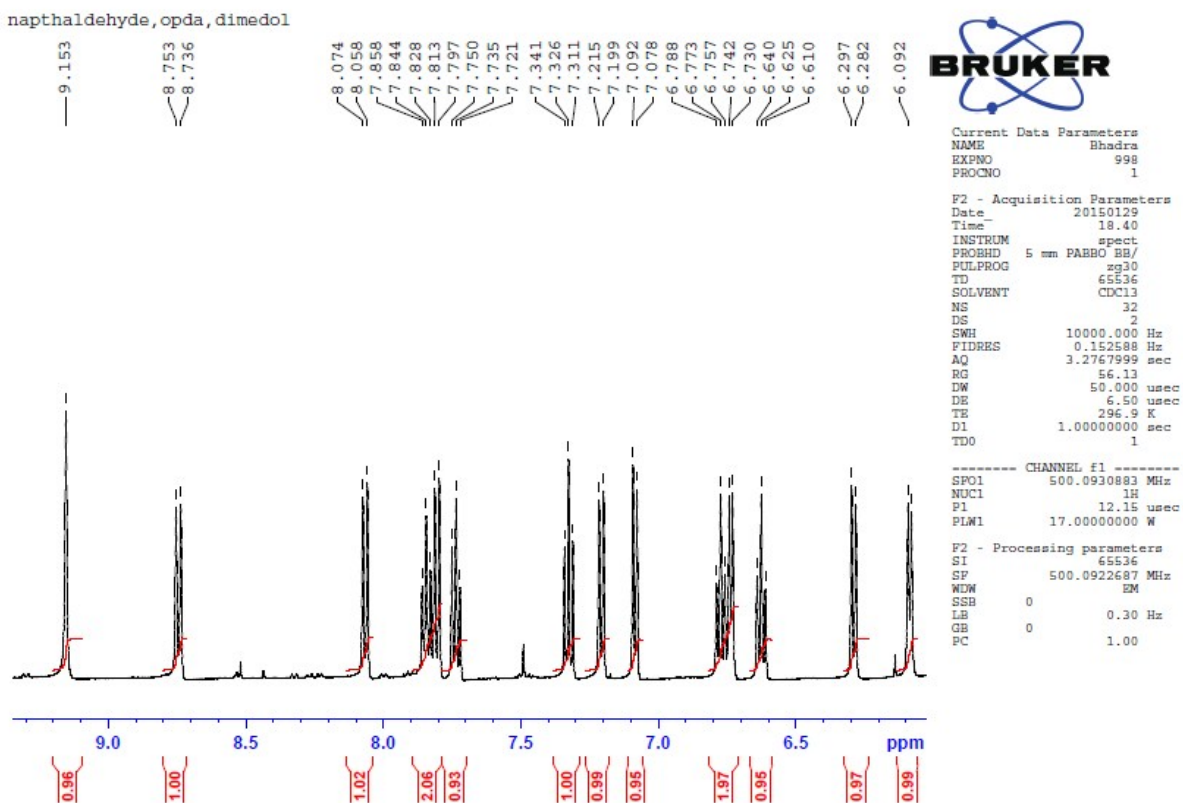
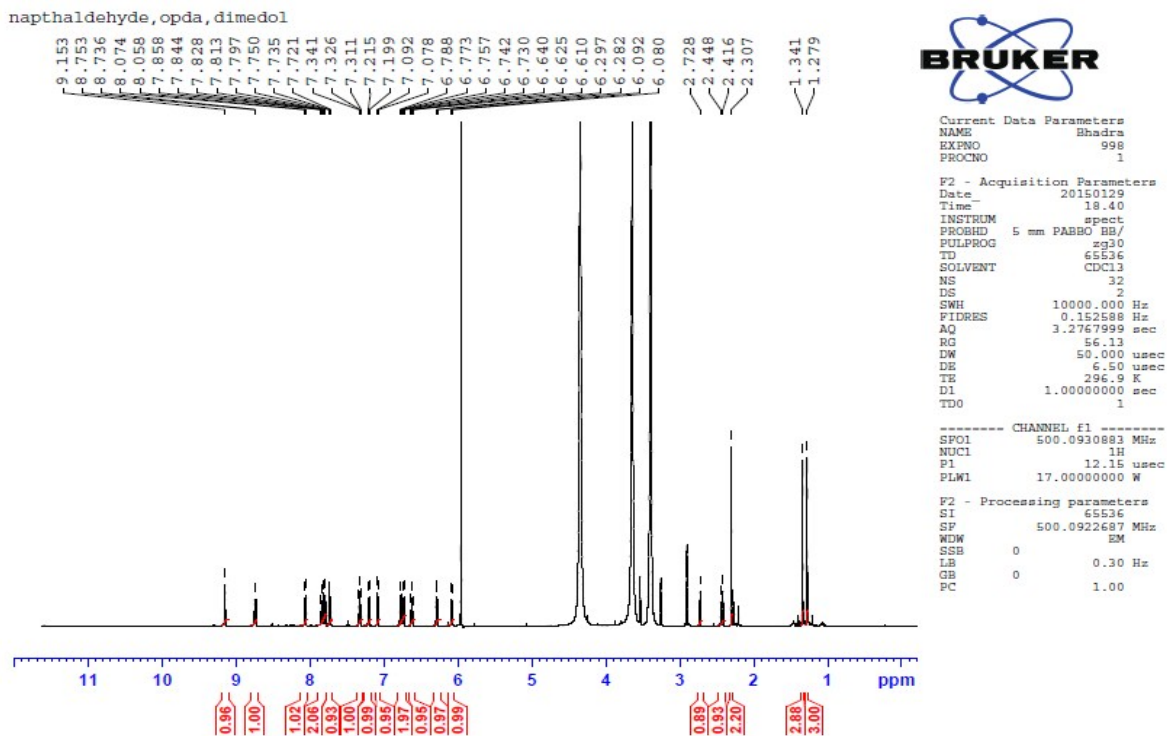
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Time 15.44
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PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 112.97
DW 16.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 125.7603047 MHz
NUC1 13C
P1 8.90 usec
PLW1 29.00000000 W

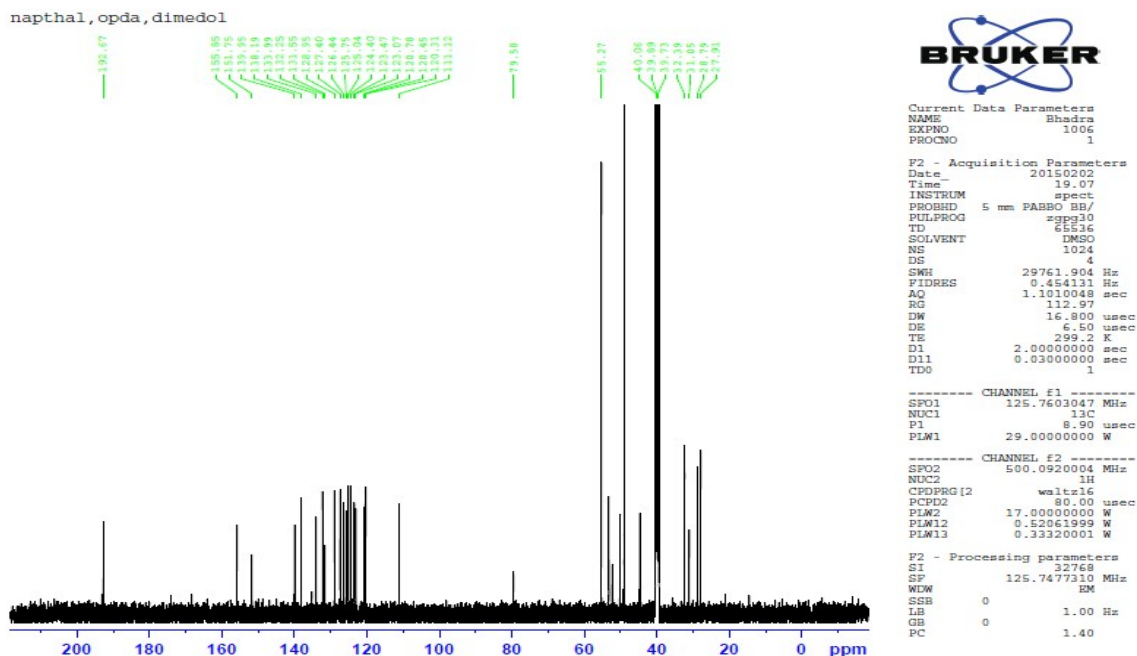
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NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLM2 17.00000000 W
PLW12 0.52061999 W
PLW13 0.33320001 W

F2 - Processing parameters
SI 32768
SF 125.7477310 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Spectrum of 1,1-dimethyl-11-(naphthalen-1-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



¹³C NMR Spectrum of 1,1-dimethyl-11-(naphthalen-1-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.

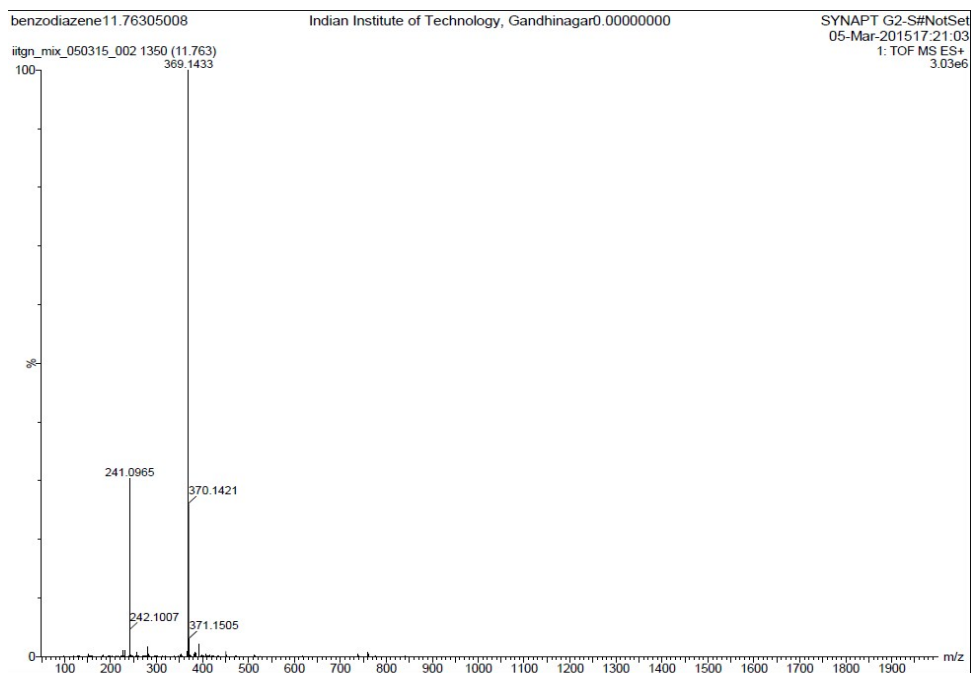


Mass spectrum of 1,1-dimethyl-11-(naphthalen-1-yl)-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

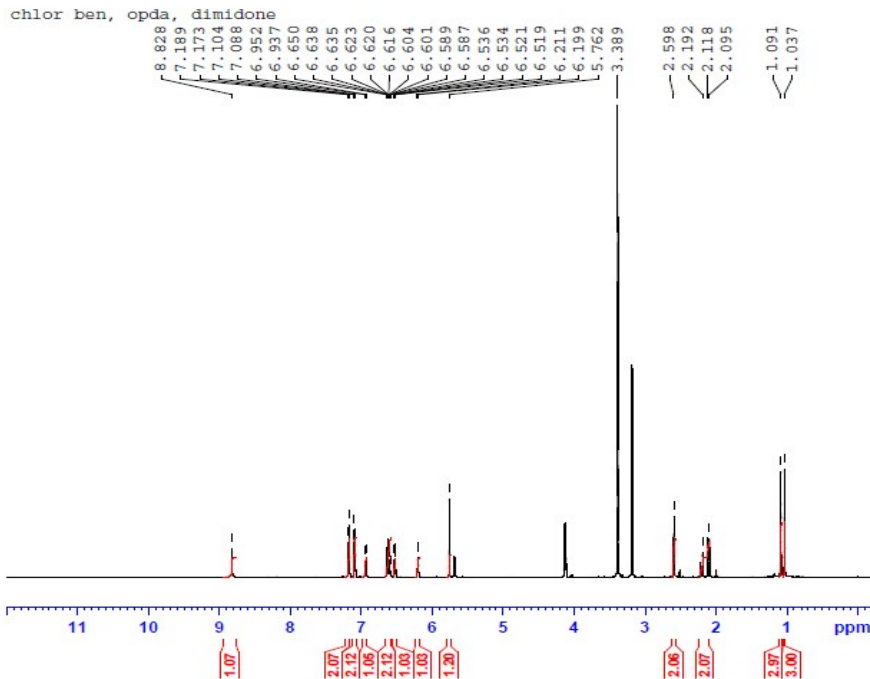
Exact mass: 368.1889

Mass obtained in the positive mode: 369.1433

Elemental composition: C₂₅H₂₄N₂O



¹H NMR Spectrum of 11-(4-chlorophenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.

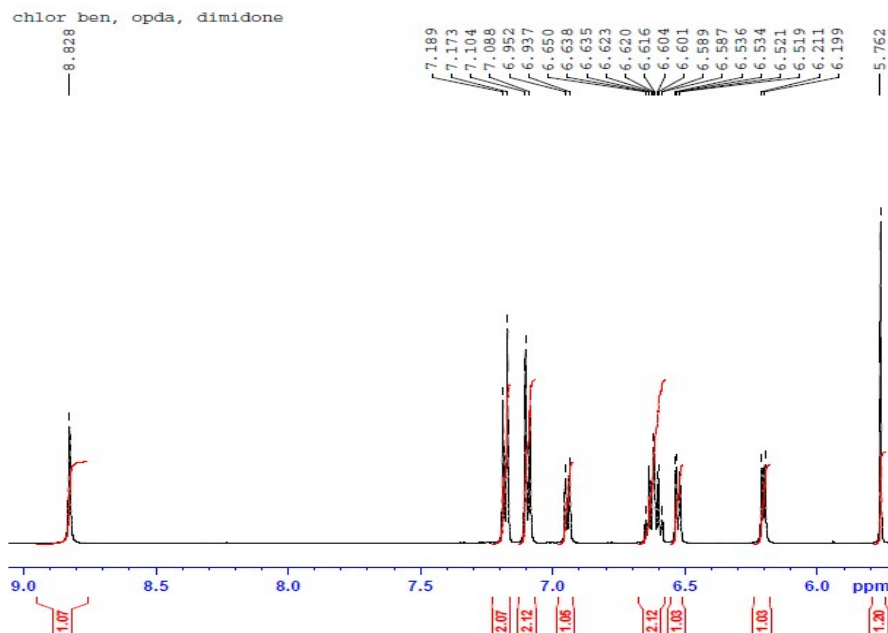


Current Data Parameters
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 EXFNO 1007
 PROCNO 1

F2 - Acquisition Parameters
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 Time 17.00
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 99.08
 DW 50.000 usec
 DE 6.50 usec
 TE 297.0 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 SFO1 500.0930883 MHz
 NUC1 1H
 P1 12.15 usec
 PLW1 17.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.0900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



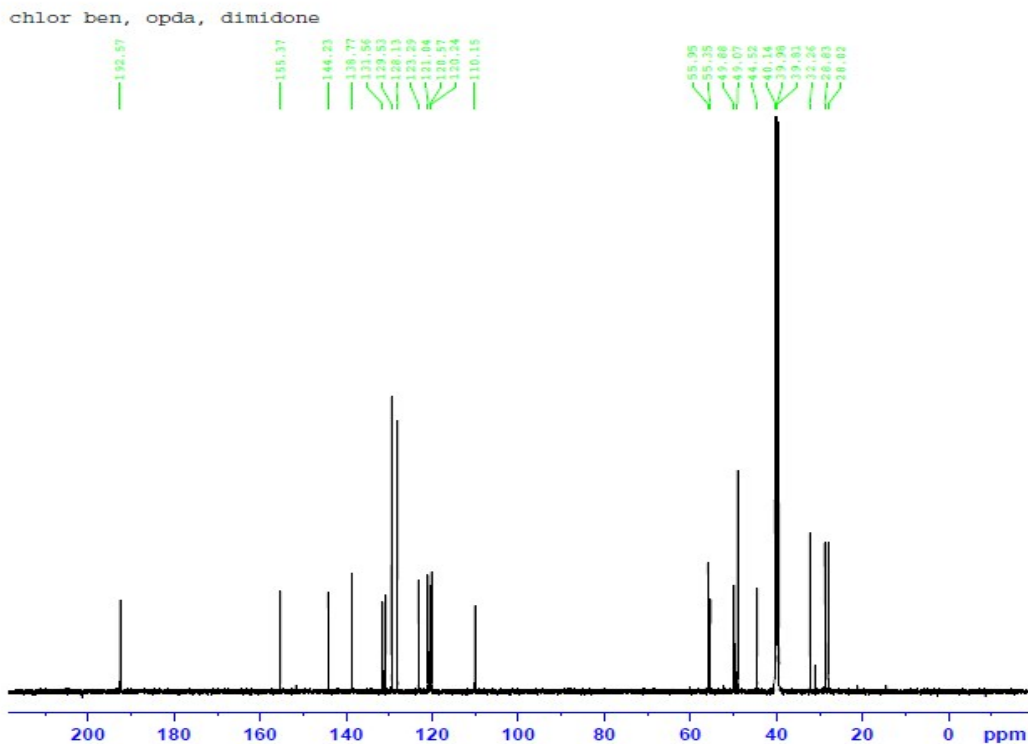
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 PROCNO 1

F2 - Acquisition Parameters
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 99.08
 DW 50.000 usec
 DE 6.50 usec
 TE 297.0 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 SFO1 500.0930883 MHz
 NUC1 1H
 P1 12.15 usec
 PLW1 17.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.0900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR Spectrum of 11-(4-chlorophenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one.



Current Data Parameters
 NAME Bhadra
 EXPNO 1009
 PROCNO 1

F2 - Acquisition Parameters
 Date 20150203
 Time 18.50
 INSTRUM spect
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 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 2000
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 112.97
 DW 16.800 usec
 DE 6.50 usec
 TE 299.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

----- CHANNEL f1 -----
 SPO1 125.7603047 MHz
 NUC1 13C
 P1 8.90 usec
 PLW1 29.0000000 W

----- CHANNEL f2 -----
 SPO2 500.0920004 MHz
 NUC2 1H
 CPDPRG [2] waltz16
 PCPD2 80.00 usec
 PLW2 17.0000000 W
 PLW12 0.52061999 W
 PLW13 0.33320001 W

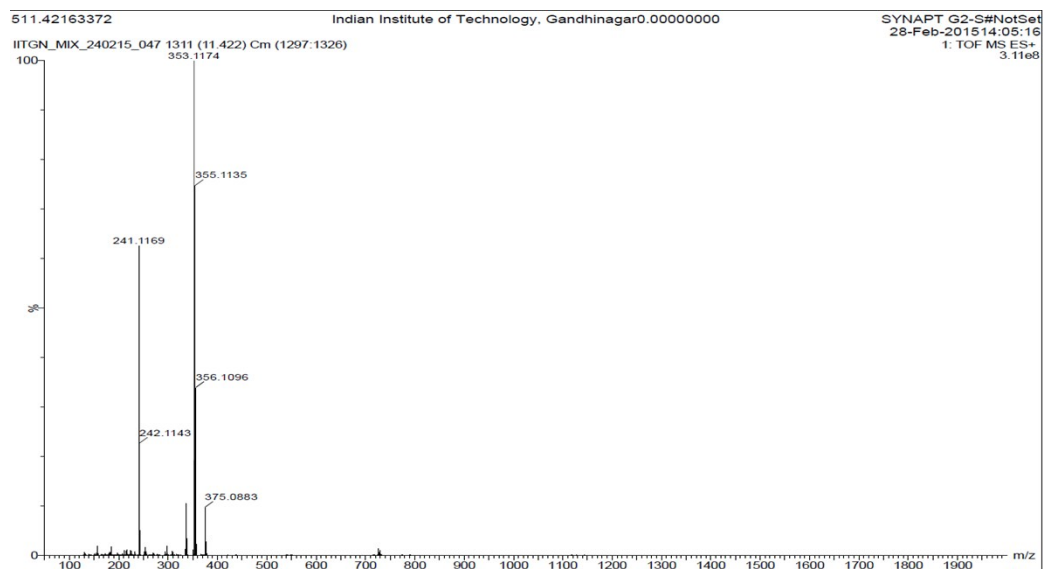
F2 - Processing parameters
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 SF 125.7477310 MHz
 WDW RM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Mass spectrum of 11-(4-chlorophenyl)-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

Exact mass: 352.1342

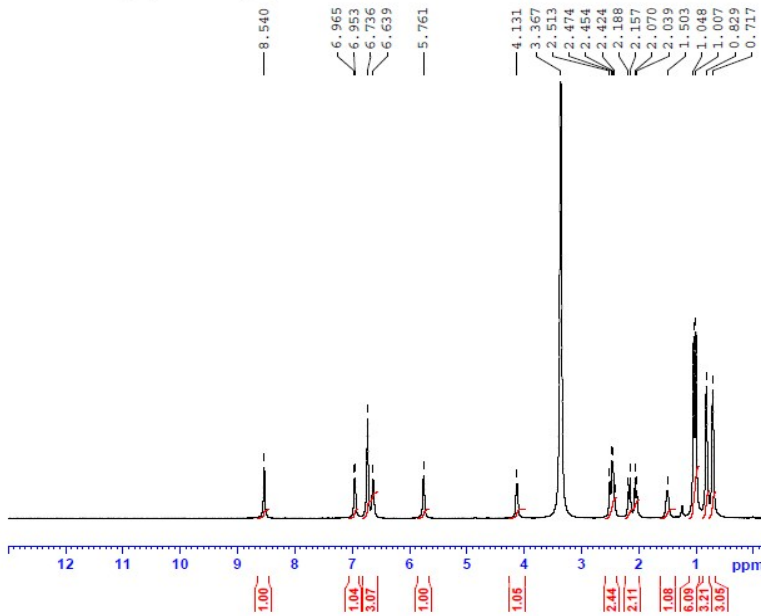
Mass obtained in the positive mode: 353.1174

Elemental composition: C₂₁H₂₁ClN₂O



¹H NMR Spectrum of 11-isobutyl-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

vallan aldehyde, dimidone, opda



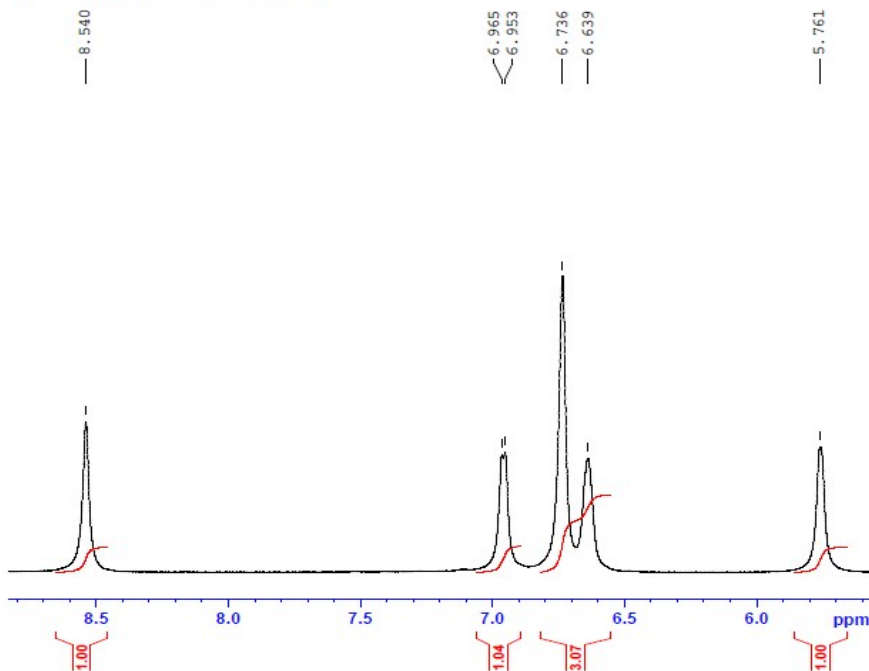
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Current Data Parameters
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EXPNO    1067
PROCNO   1

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Time     22.55
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PROBHD   5 mm PABBO BB/
PULPROG zg30
TD       65536
SOLVENT  DMSO
NS       32
DS       2
SWH      10000.000 Hz
FIDRES   0.152588 Hz
AQ       3.2767999 sec
RG       112.97
DE       50.000 usec
TE       299.7 K
DI       1.0000000 sec
TD0      1

----- CHANNEL f1 -----
SF01    500.0930883 MHz
NUC1     1H
P1      12.15 usec
PLW1    17.00000000 W

F2 - Processing parameters
SI      65536
SF      500.0900000 MHz
WDW     EM
SSB     0
LB      0.30 Hz
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PC      1.00
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vallan aldehyde, dimidone, opda



```
Current Data Parameters
NAME      Bhadra
EXPNO    1067
PROCNO   1

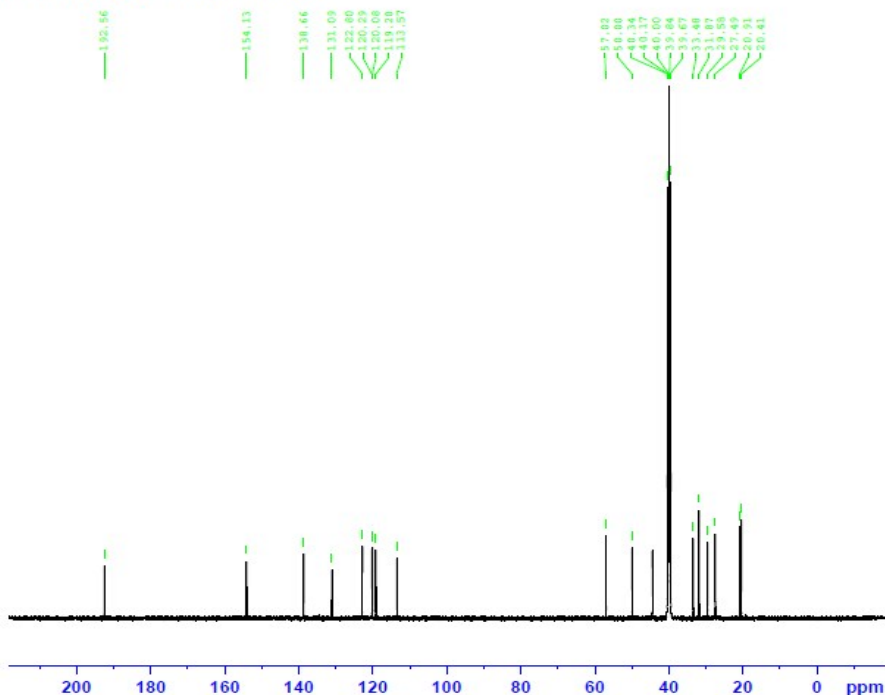
F2 - Acquisition Parameters
Date_    20150312
Time     22.55
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG zg30
TD       65536
SOLVENT  DMSO
NS       32
DS       2
SWH      10000.000 Hz
FIDRES   0.152588 Hz
AQ       3.2767999 sec
RG       112.97
DE       50.000 usec
TE       299.7 K
DI       1.0000000 sec
TD0      1

----- CHANNEL f1 -----
SF01    500.0930883 MHz
NUC1     1H
P1      12.15 usec
PLW1    17.00000000 W

F2 - Processing parameters
SI      65536
SF      500.0900000 MHz
WDW     EM
SSB     0
LB      0.30 Hz
GB      0
PC      1.00
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¹³C NMR Spectrum of 11-isobutyl-1,1-dimethyl-4,5,10,11-tetrahydro-1H-dibenzo[b,e][1,4]diazepin-3(2H)-one

vallan aldehyde, dimidone, opda



```

Current Data Parameters
NAME          Bhadra
EXPNO        1068
PROCNO       1

F2 - Acquisition Parameters
Date_        20150313
Time_        2.30
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg30
TD           65536
SOLVENT      DMSO
NS           4000
DS           4
SWH          29761.904 Hz
FIDRES       0.454131 Hz
AQ           1.1010048 sec
RG           112.97
DW           16.800 usec
DE           6.50 usec
TE           301.0 K
D1           2.0000000 sec
D11          0.03000000 sec
TDO          1

----- CHANNEL f1 -----
SFO1         125.7603047 MHz
NUC1          13C
P1           8.90 usec
PLW1         29.00000000 W

----- CHANNEL f2 -----
SFO2         500.0920004 MHz
NUC2          1H
CPDPRG2      waltz16
PCPD2        80.00 usec
PLW2         17.00000000 W
PLW12        0.52061999 W
PLW13        0.33320001 W

F2 - Processing parameters
SI           32768
SF           125.7477310 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
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