

Supplementary Information

Synthesis of Aryl anilinoimide based derivatives as glycogen synthase kinase-3 β inhibitors with potential role as antidepressant agents

Mushtaq A Tantray^a, Imran Khan^a, Hinna Hamid^{a*}, Mohammad Sarwar Alam^a, Abhijeet Dhulap^b, Abul Kalam^c

^a*Department of Chemistry, Faculty of Science, Jamia Hamdard (Hamdard University), New Delhi 110062, India.*

^b*CSIR- Unit for Research and Development of Information Products (URDIP), Pune 411038, India*

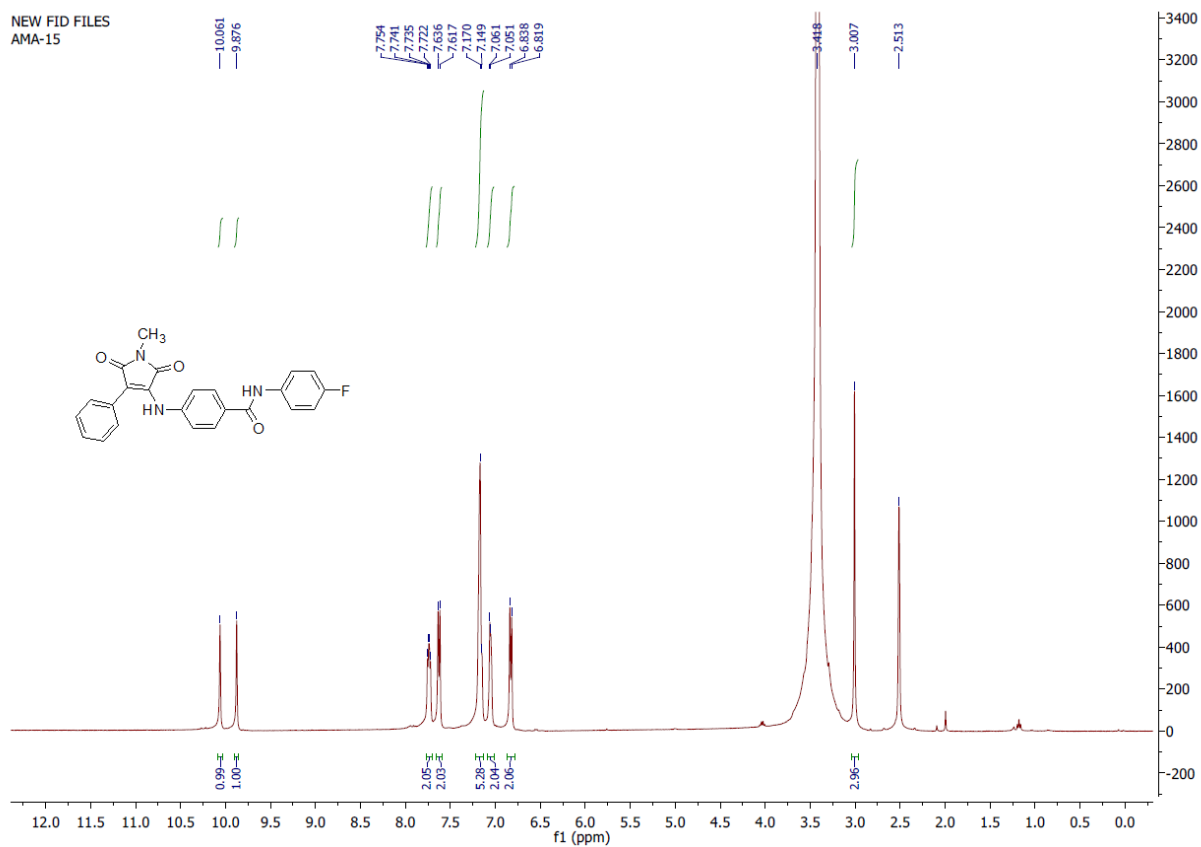
^c*Department of Pharmacology, Faculty of Pharmacy, Jamia Hamdard (Hamdard University), New Delhi 110062, India.*

Correspondence to Dr. Hinna Hamid, Jamia Hamdard (Hamdard University) Hamdard Nagar, New Delhi 110062, Email ID. hhamid@jamiyahamdard.ac.in. Phone: +91 11 26059688 (5559).

Submitted to: New Journal of Chemistry

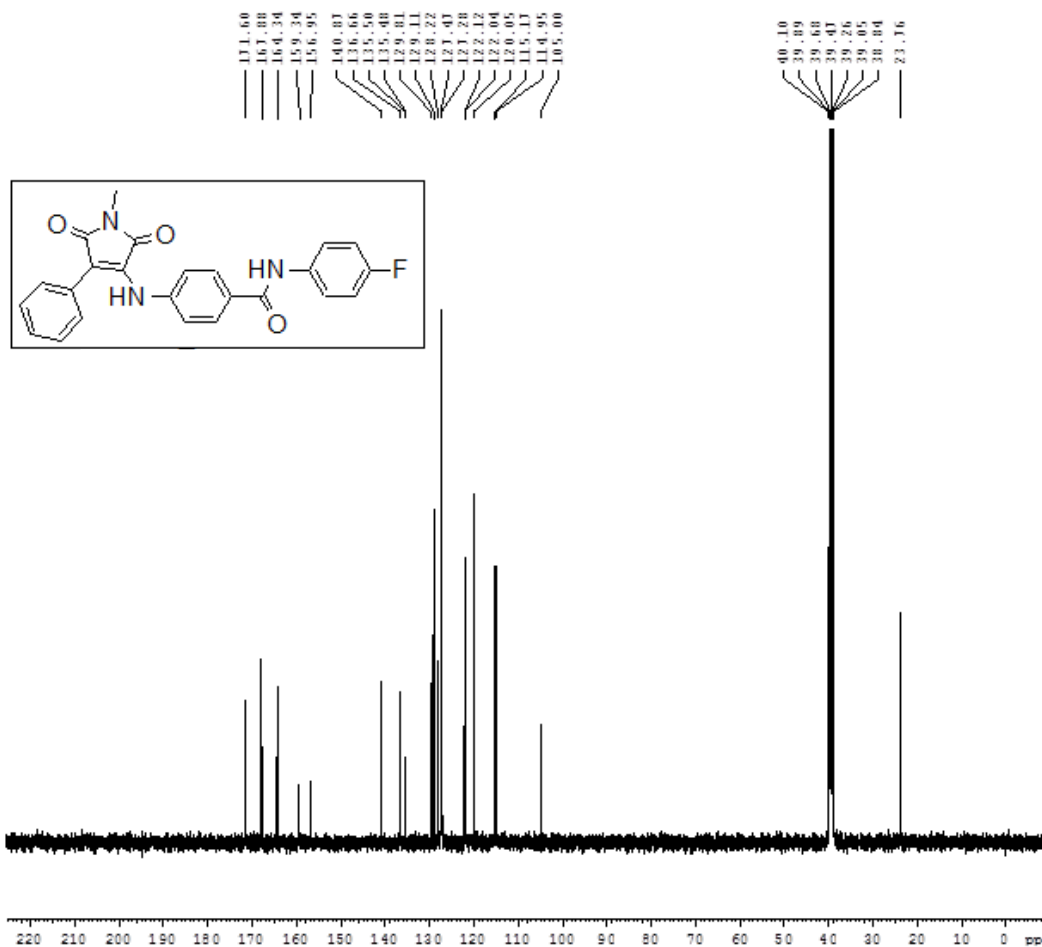
Spectral characterization of representative compounds:

4-(2,5-dihydro-1-methyl-2,5-dioxo-4-phenyl-1H-pyrrol-3-ylamino)-N-(4-fluorophenyl)benzamide (7c)



Proton NMR (7c)

AMA-15



BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME Nov13-2014
EXPNO 560
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141114
Time 14.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

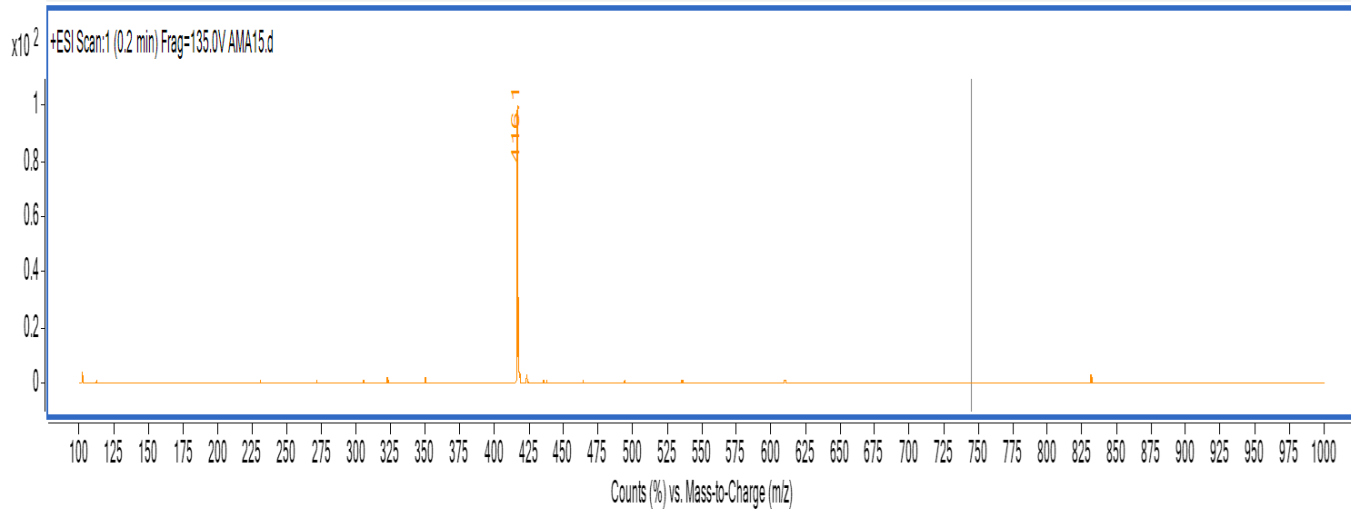
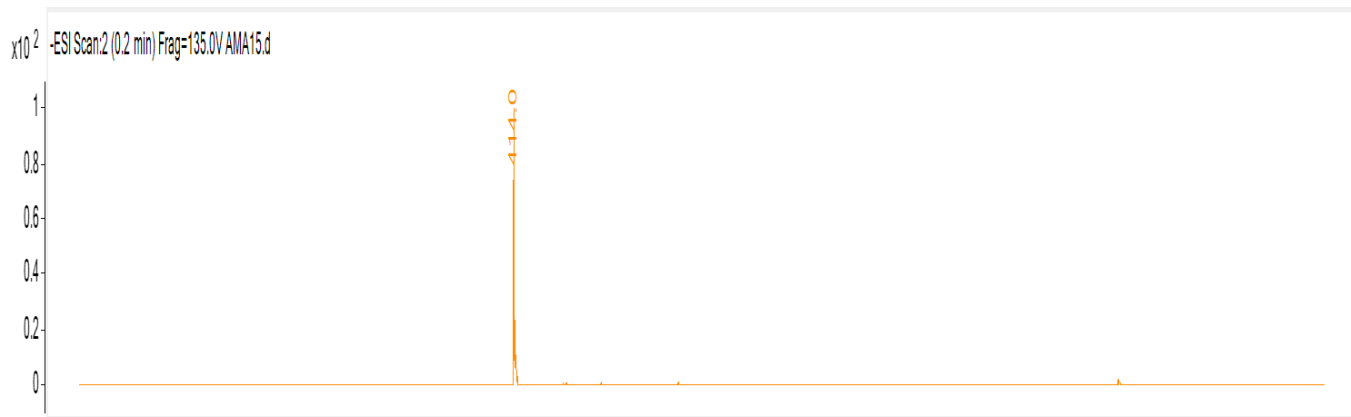
==== CHANNEL f1 =====
NUC1 13C
P1 9.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.31 dB
PL13 18.00 dB
SFO2 400.1216005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

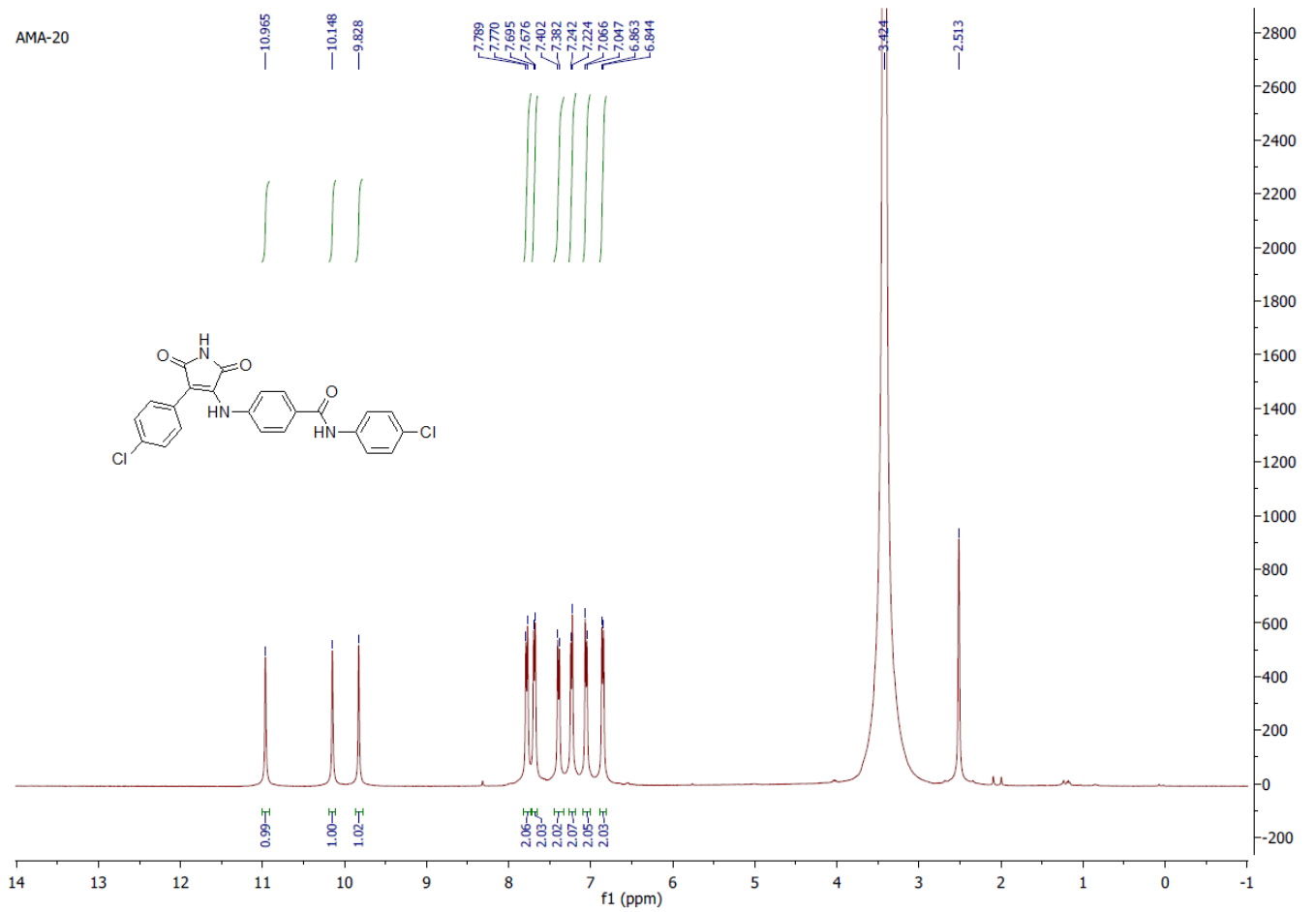
avtar saifpu@yahoo.co.in

C-13 NMR (7c)



ESI-MS (7c)

4-(4-(4-chlorophenyl)-2,5-dihydro-2,5-dioxo-1H-pyrrol-3-ylamino)-N-(4-chlorophenyl)benzamide
(8h)



Proton NMR (8h)

AMA-20

BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME Nov13-2014
EXRNO 600
PROCNO 1

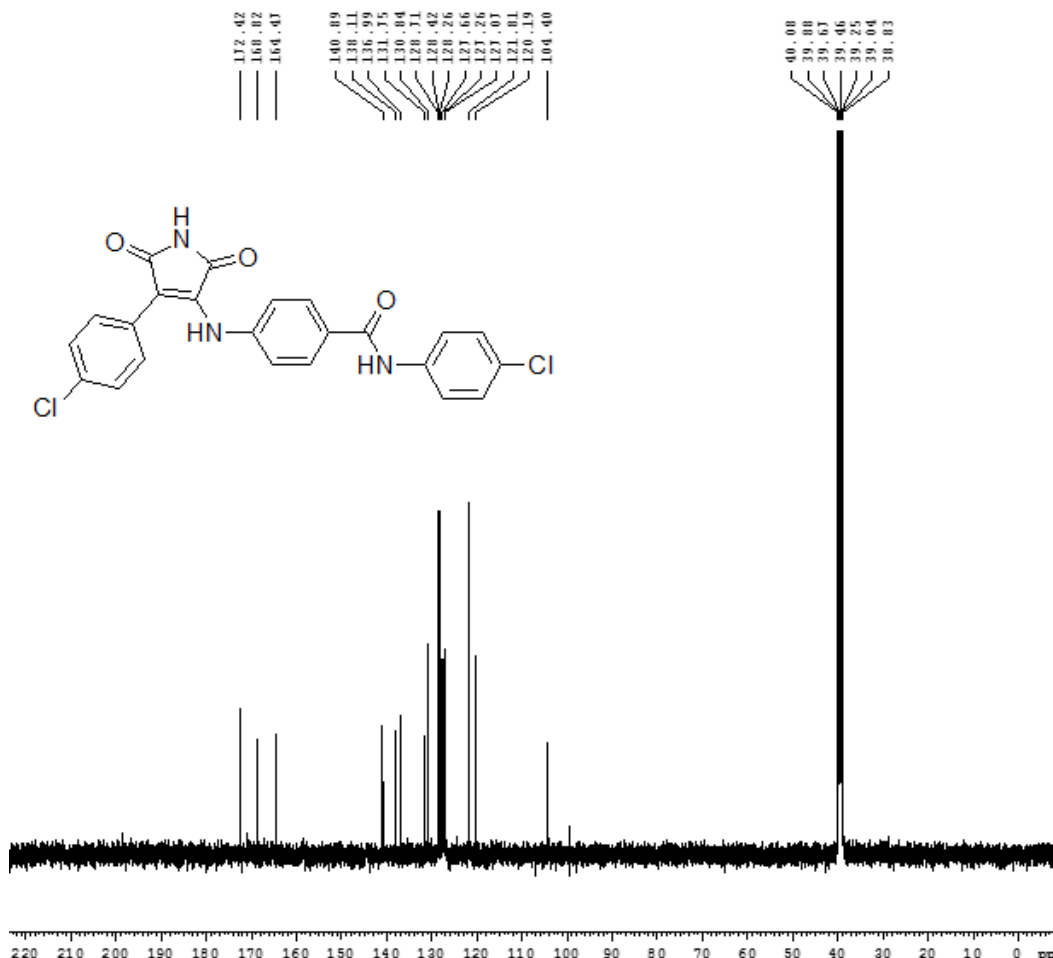
F2 - Acquisition Parameters
Date_ 20141114
Time_ 16.33
INSTRUM spect
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 36
DW 16.800 usec
DE 6.00 usec
TE 294.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.60 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

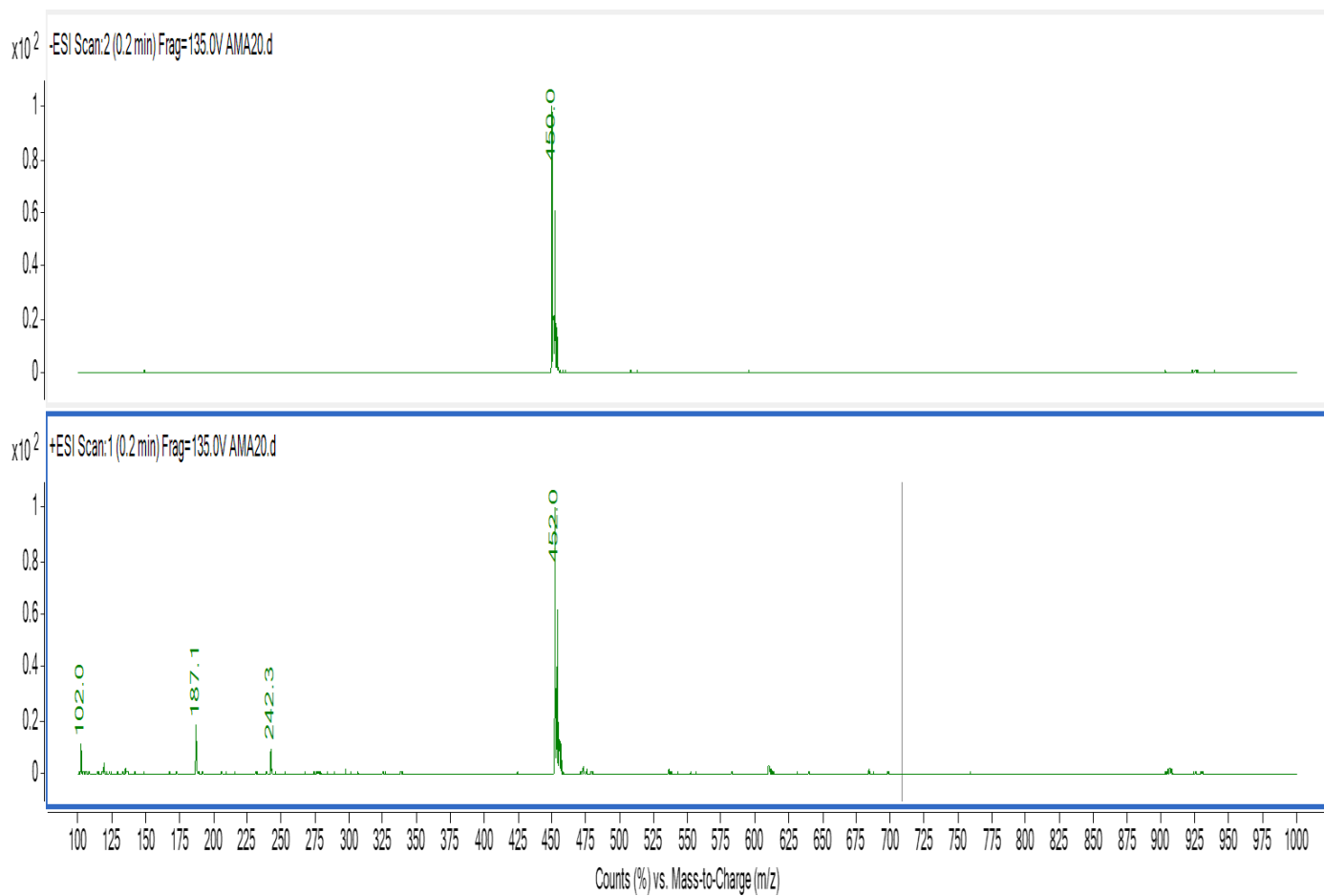
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.31 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

avtar saifpu@yahoo.co.in



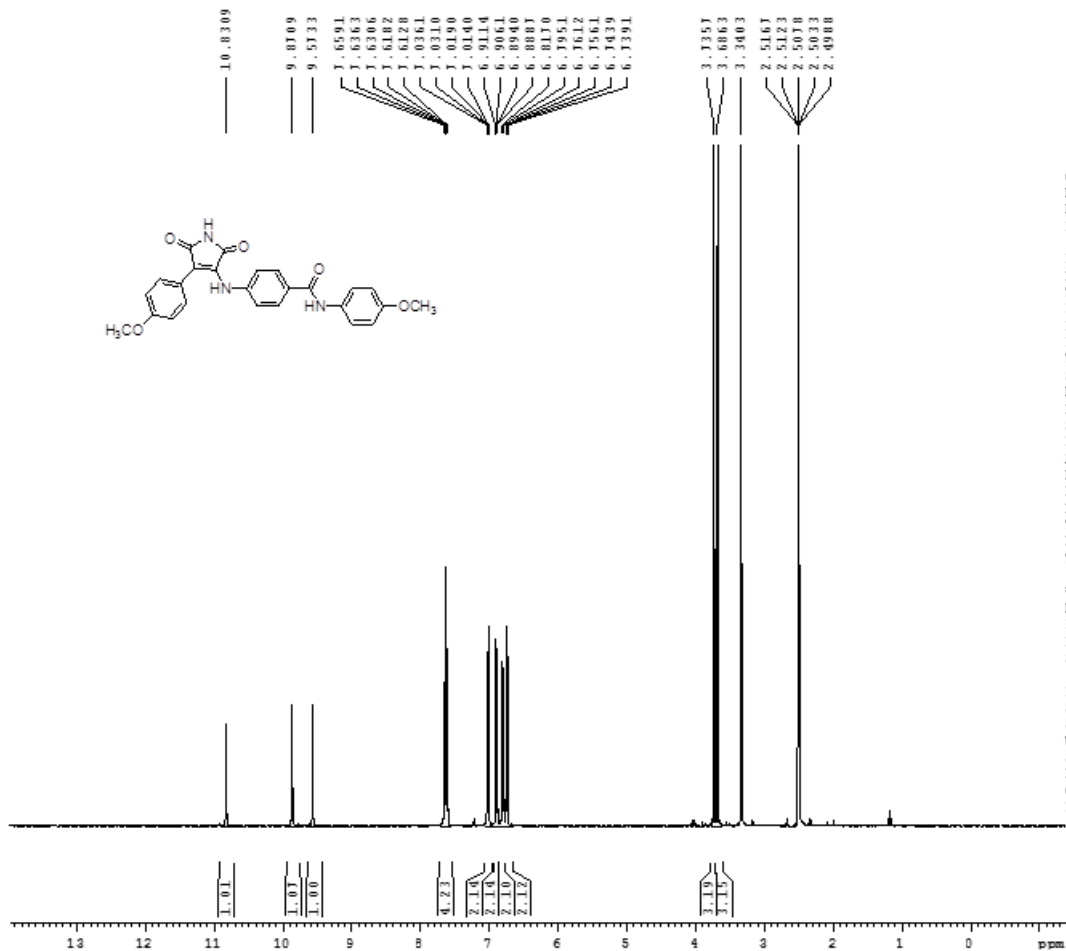
C-13 NMR (8h)



ESI-MS (8h)

4-(2,5-dihydro-4-(4-methoxyphenyl)-2,5-dioxo-1H-pyrrol-3-ylamino)-N-(4-methoxyphenyl)benzamide
(8j)

AMA-22



BRUKER
 AVANCE II 400 NMR
 Spectrometer
 SAIF
 Panjab University
 Chandigarh

Current Data Parameters
 NAME May15-2015
 EXPNO 430
 PROCNO 1

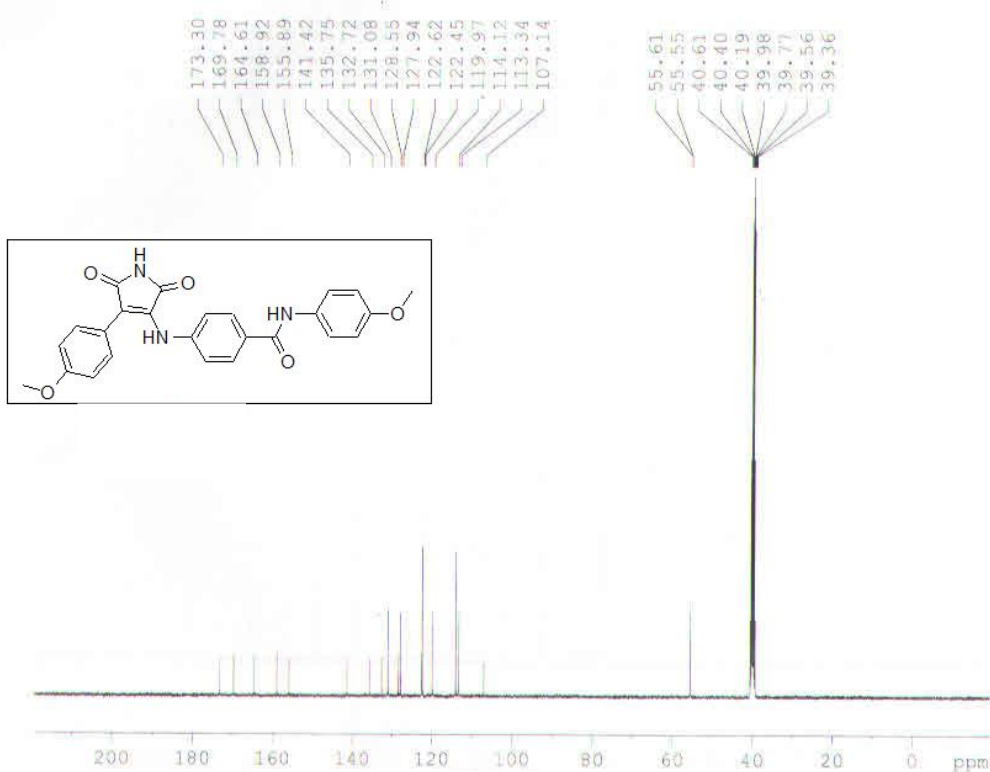
F2 - Acquisition Parameters
 Date_ 20150515
 Time 17.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 812
 DW 41.600 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.90 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

avtar saifpu@yahoo.co.in

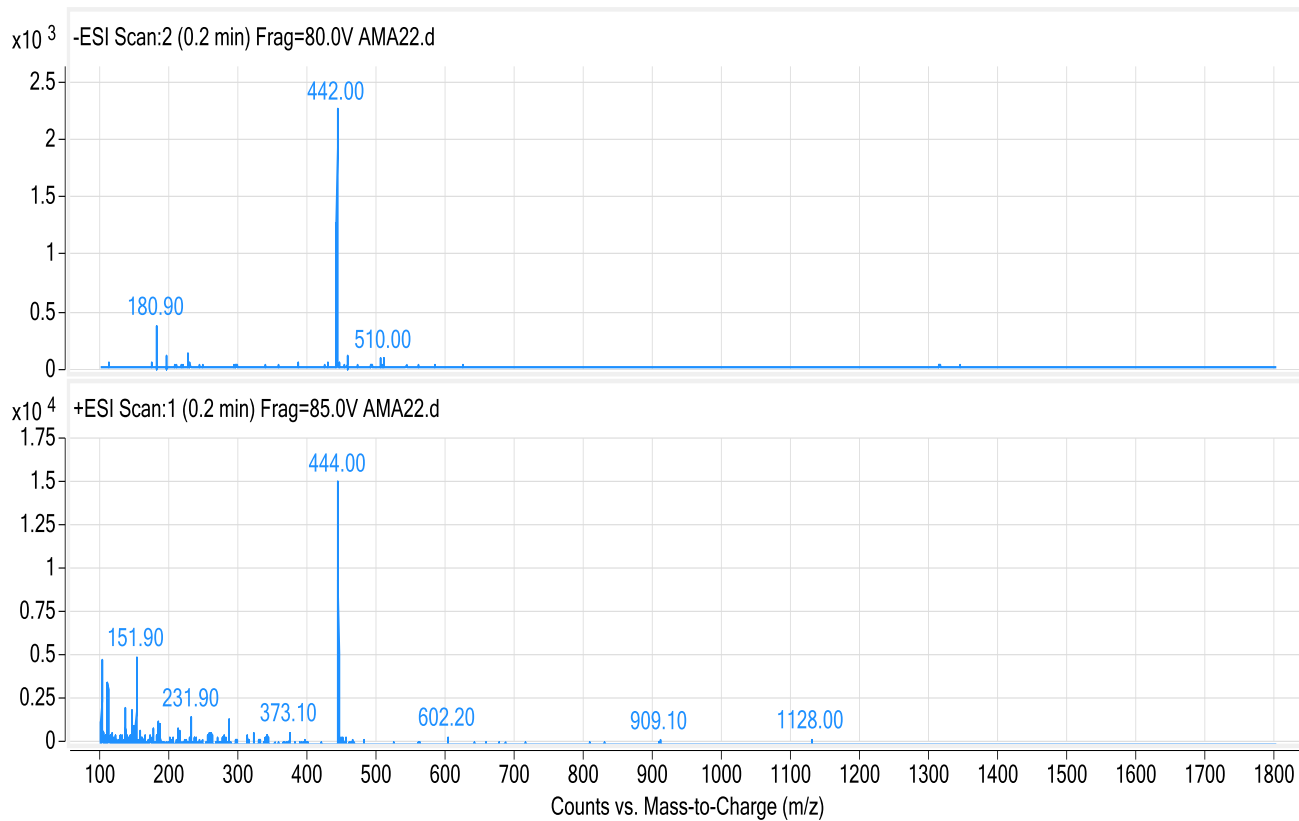
Proton NMR (8j)



```

===== CHANNEL f1 =====
NAME: 13C NMR MUSHTAQ AT
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20120506
Time: 11:55
PROBHD: 5 mm BBO 51-1H
PULPROG: zgpg30
TE: 300.2
SFO: 125.76
AQ: 0.04
RG: 655
WDW: EM
SSB: 0
GB: 0
PC: 1.00
===== CHANNEL f2 =====
NAME:
PROCNO:
F2 - Processing parameters
SI: 32768
SF: 125.7612500 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00
    
```

C-13 NMR (8j)

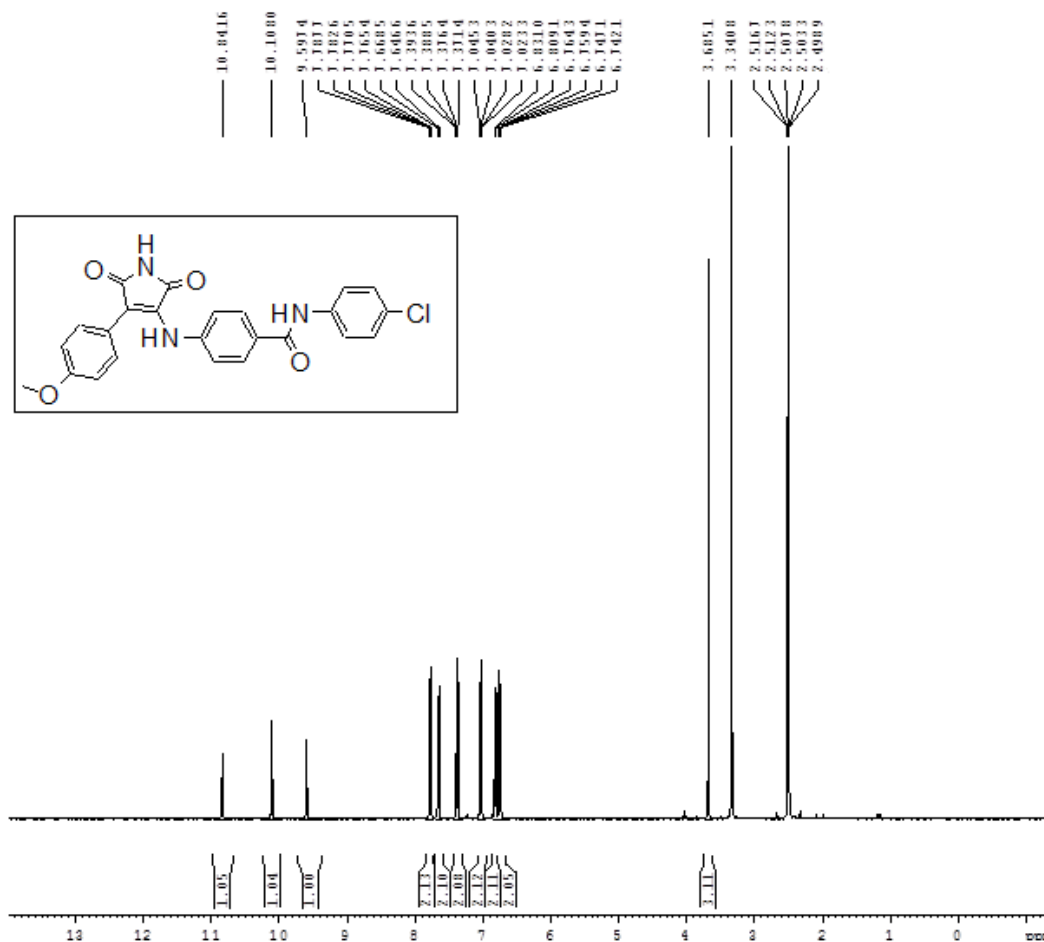


ESI-MS (8j)

4-(2,5-dihydro-4-(4-methoxyphenyl)-2,5-dioxo-1H-pyrrol-3-ylamino)-N-(4-chlorophenyl)benzamide

(81)

AMA-24



BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME May15-2015
EXPMO 450
PROCNO 1

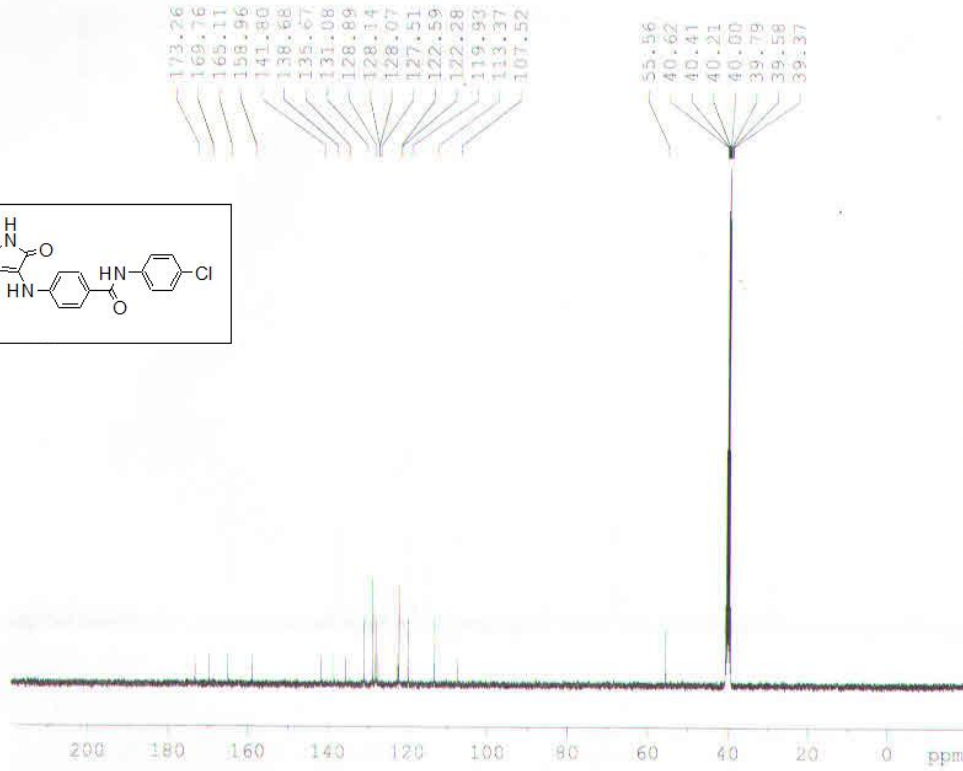
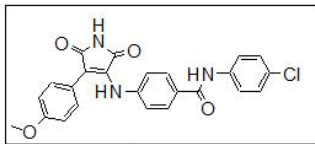
F2 - Acquisition Parameters
Date_ 20150515
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 812
DW 41.600 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

Proton NMR (81)



Source: Data: 20120502
NAME: JH_SNS_MUSHTAQ_AT_AYA_24
EXPNO: 1

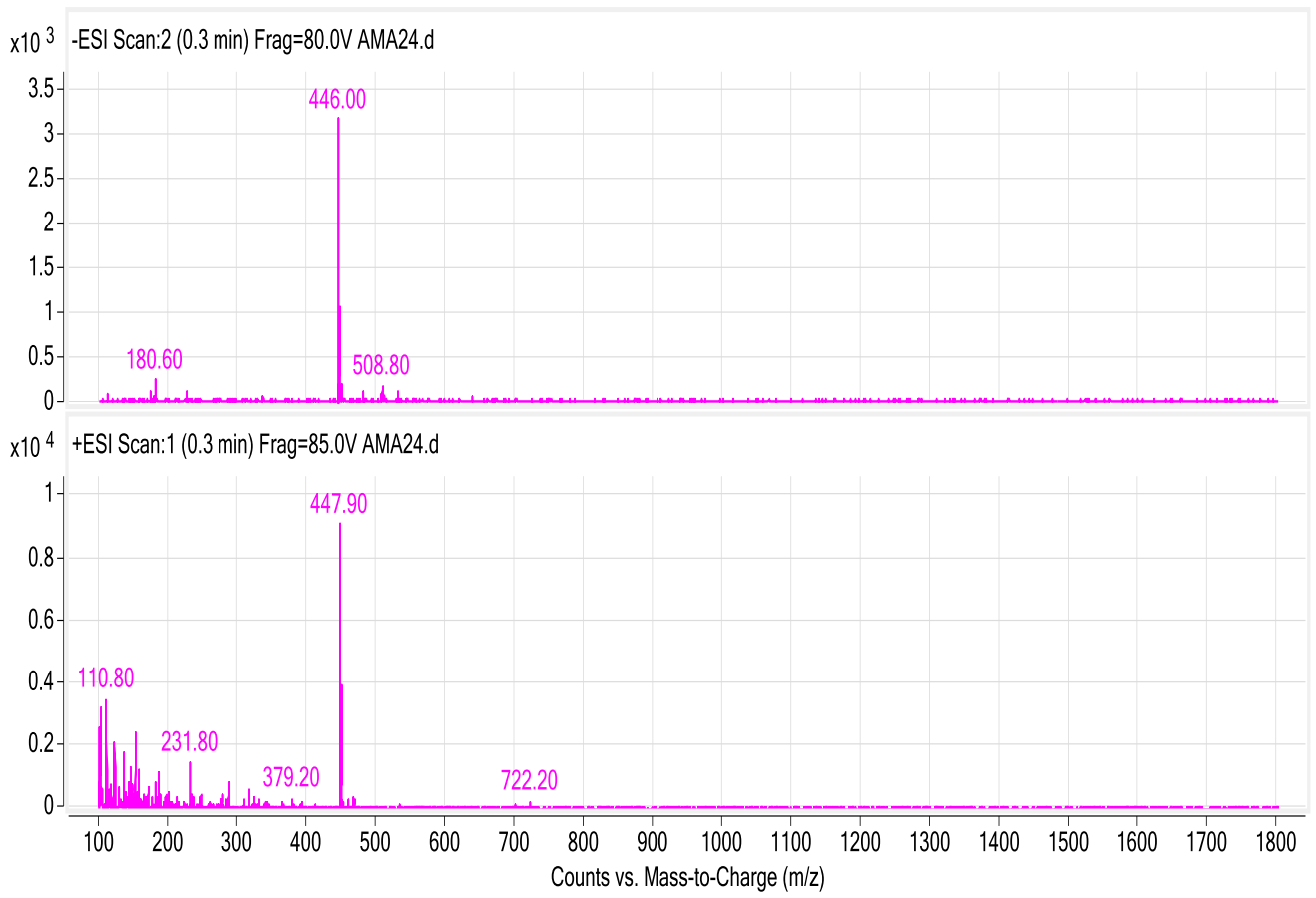
===== Acquisition Parameters =====
Date_ 20120502
Time 15:44
INSTRUM spect
PROBHD 5 mm BBO BP-1H
PULPROG zgpg30
PC 39936
SFO 400.146
AQ 0.350
RG 384
SD 4
SI 2400.461 Hz
FIDRES 2.766728 Hz
AQ 0.3631900 sec
RG 384
TM 20.400 sec
TE 300.2 K
DT 250.1 Hz
QT 2.0000000 sec
SFO 400.1460000 MHz
NUC1 13C
NUC2 13C
P1 1.00 sec
PC 1.00 sec

===== CHANNEL f1 =====
NUC1 13C
P1 1.00 sec
PC 1.00 sec
SFO 100.628380 MHz

===== CHANNEL f2 =====
NAME waftz2
P1 1.00
PC 1.00
SFO 400.146000 MHz

===== PROCESSING PARAMETERS =====
SI 2400.461
SF 400.146000 MHz
WDW EM
SSB 0
GB 0
PC 1.00 sec
CD 0
SD 4

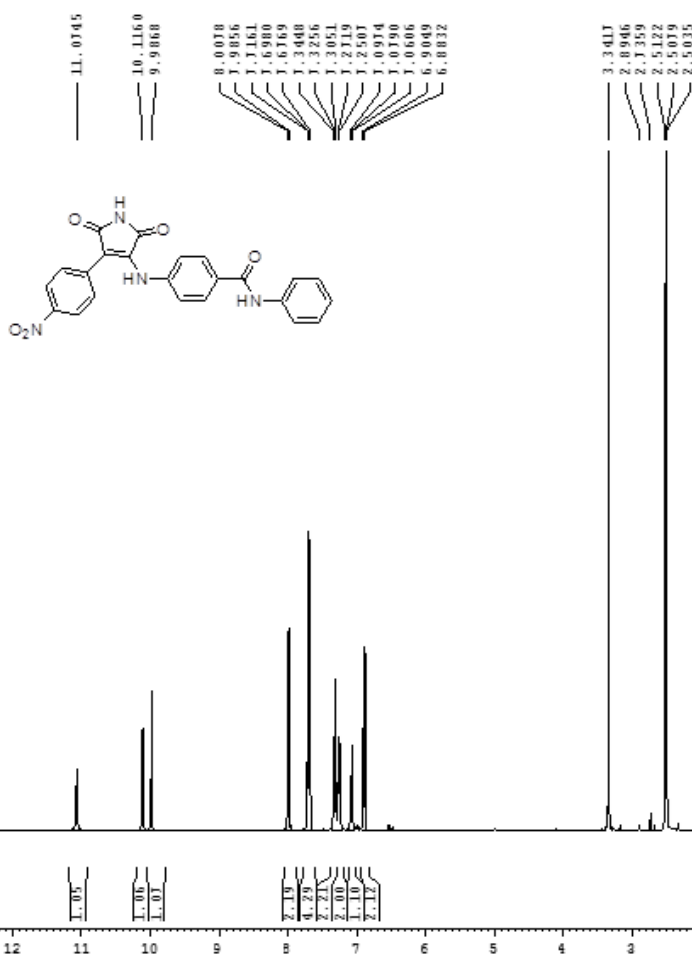
C-13 (81)



ESI-MS (81)

4-(2,5-dihydro-4-(4-nitrophenyl)-2,5-dioxo-1H-pyrrol-3-ylamino)-N-phenylbenzamide (8m)

AMA-25



BRUKER
 AVANCE II 400 NMR
 Spectrometer
 SAIF
 Panjab University
 Chandigarh

Current Data Parameters
 NAME May15-2015
 EXPNO 460
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150515
 Time 18.06
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 362
 DW 41.600 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.00000000 sec
 TDO 1

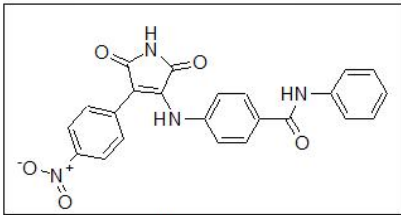
===== CHANNEL f1 =====
 NUC1 1H
 P1 10.90 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

avtar saifpu@yahoo.co.in

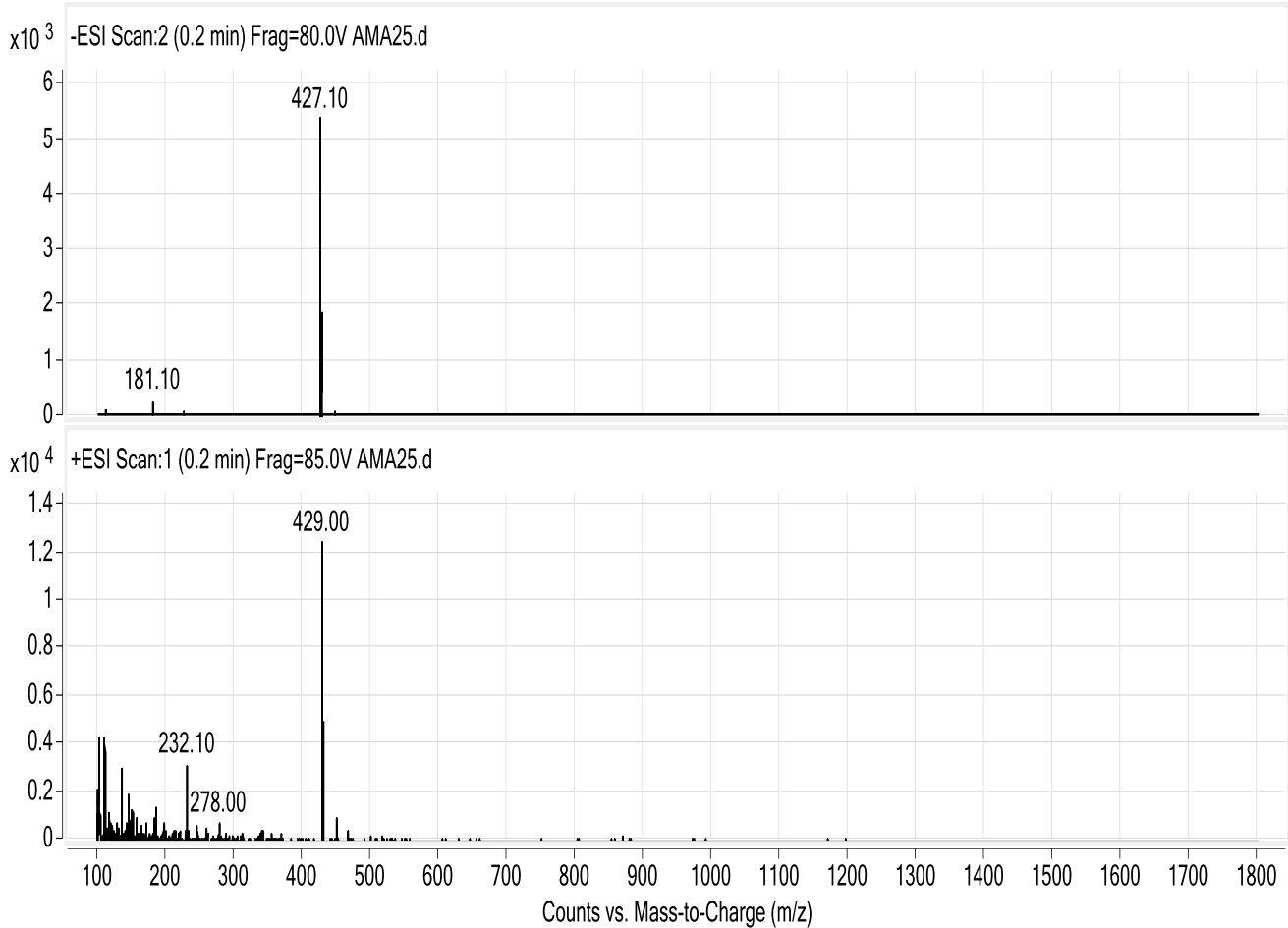
Proton NMR (8m)

JH SNS MUSHTAQ AT AMA 25



```
=====
Experiment: 13C NMR
Date_: 01/05/2015
Time_: 11:37
INSTRUM: spect
PROBHD: 5 mm BBO-EBB-1H
PULPROG: zgpg30
TD: 65536
SFO: 125.761
AQ: 0.500
RG: 1024
AQ2: 0.010
SFO2: 400.141
PC: 1.00
=====
F2 - Acquisition Parameters
Date_: 01/05/2015
Time_: 11:37
INSTRUM: spect
PROBHD: 5 mm BBO-EBB-1H
PULPROG: zgpg30
TD: 65536
SFO: 125.761
AQ: 0.500
RG: 1024
AQ2: 0.010
SFO2: 400.141
PC: 1.00
=====
F2 - Processing parameters
SI: 32768
SF: 125.761
WDW: EM
SSB: 0
GB: 0
PC: 1.00
SC: 32768
SF2: 400.141
WDW2: EM
SSB2: 0
GB2: 0
PC2: 1.00
=====
F2 - Reference parameters
SI: 32768
SF: 125.761
WDW: EM
SSB: 0
GB: 0
PC: 1.00
SC: 32768
SF2: 400.141
WDW2: EM
SSB2: 0
GB2: 0
PC2: 1.00
=====
```

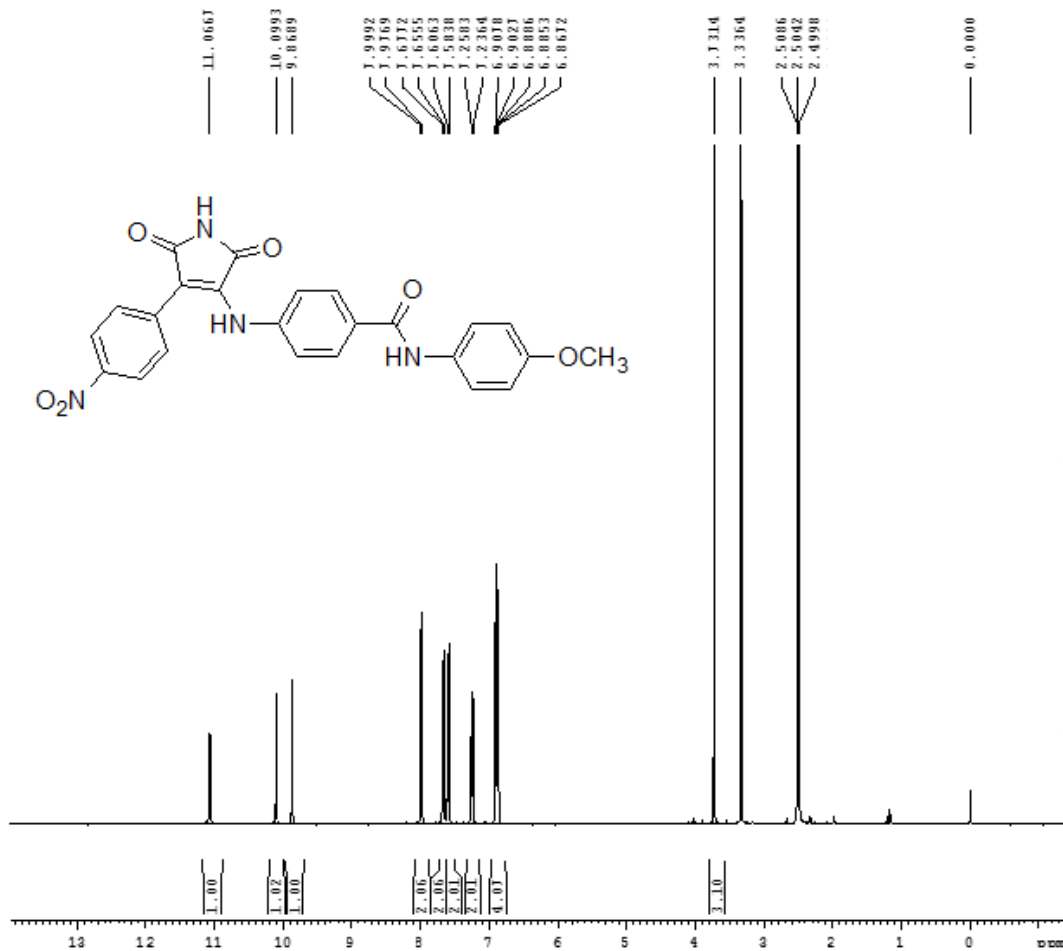
C-13 (8m)



ESI-MS (8m)

4-(2,5-dihydro-4-(4-nitrophenyl)-2,5-dioxo-1H-pyrrol-3-ylamino)-N-(4-methoxyphenyl)benzamide
(8n)

AMA-26



BRUKER
 AVANCE II 400 NMR
 Spectrometer
 SAIF
 Panjab University
 Chandigarh

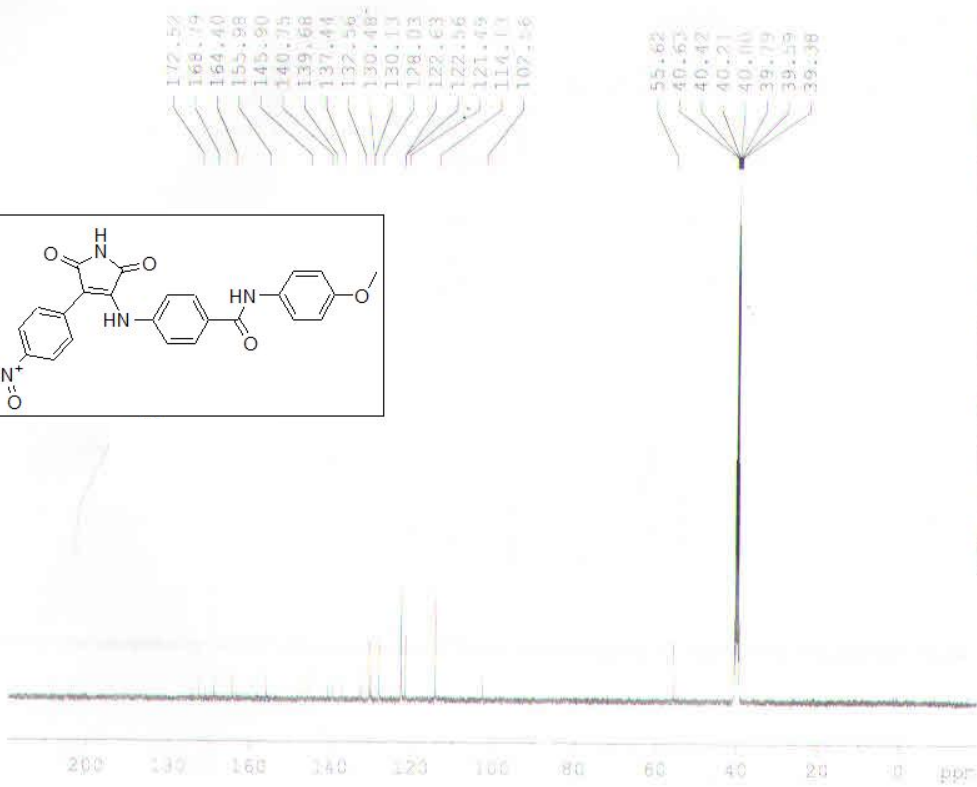
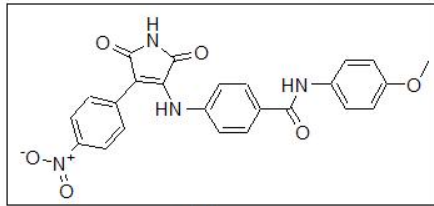
Current Data Parameters
 NAME May15-2015
 EXPNO 470
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150515
 Time 18.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 812
 DW 41.600 usec
 DE 6.00 usec
 TE 295.6 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.90 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130015 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Proton NMR (8n)



C-13 (8n)



ESI-MS (8n)

4-(2,5-dihydro-2,5-dioxo-4-phenyl-1H-pyrrol-3-ylamino)-N-(4-methoxyphenyl)benzamide (8b)

AMA-30

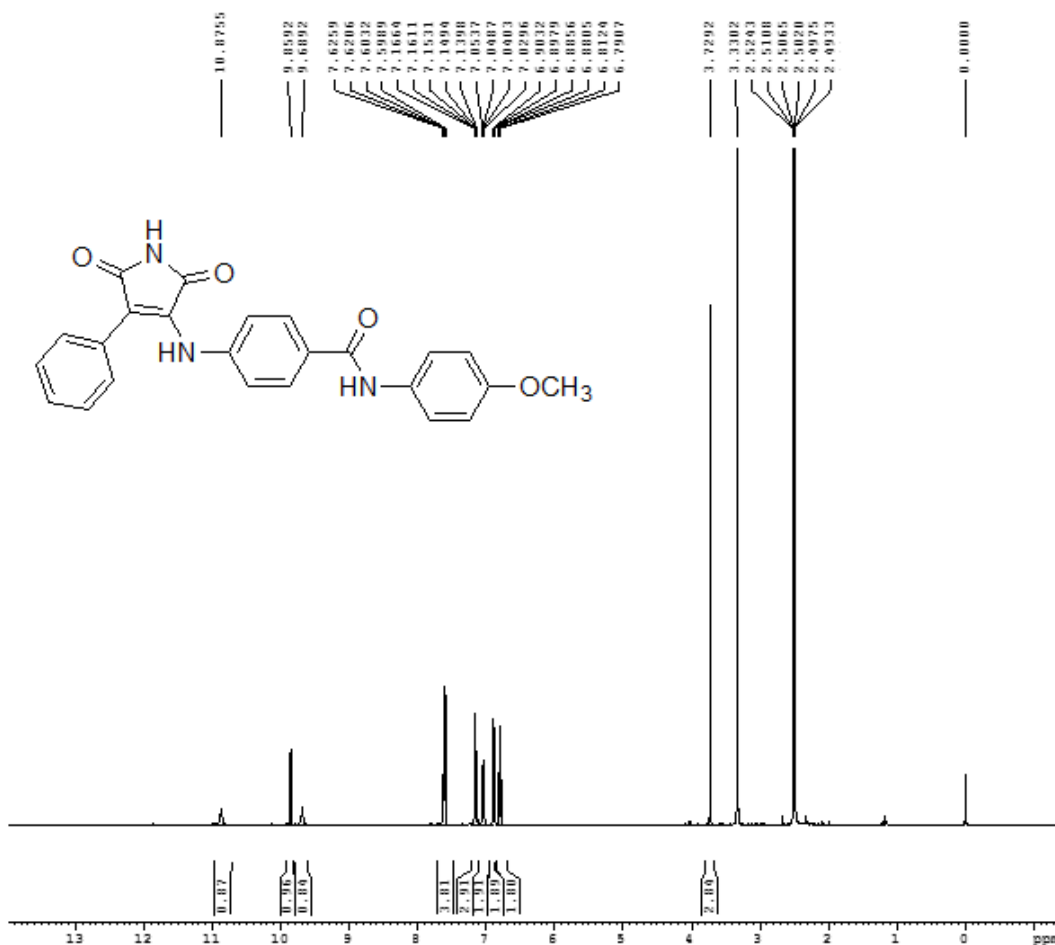
BRUKER
 AVANCE II 400 NMR
 Spectrometer
 SAIF
 Panjab University
 Chandigarh

Current Data Parameters
 NAME May15-2015
 EXPNO 510
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150515
 Time 18.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 456
 DW 41.600 usec
 DE 6.00 usec
 TE 295.7 K
 D1 1.00000000 sec
 TD0 1

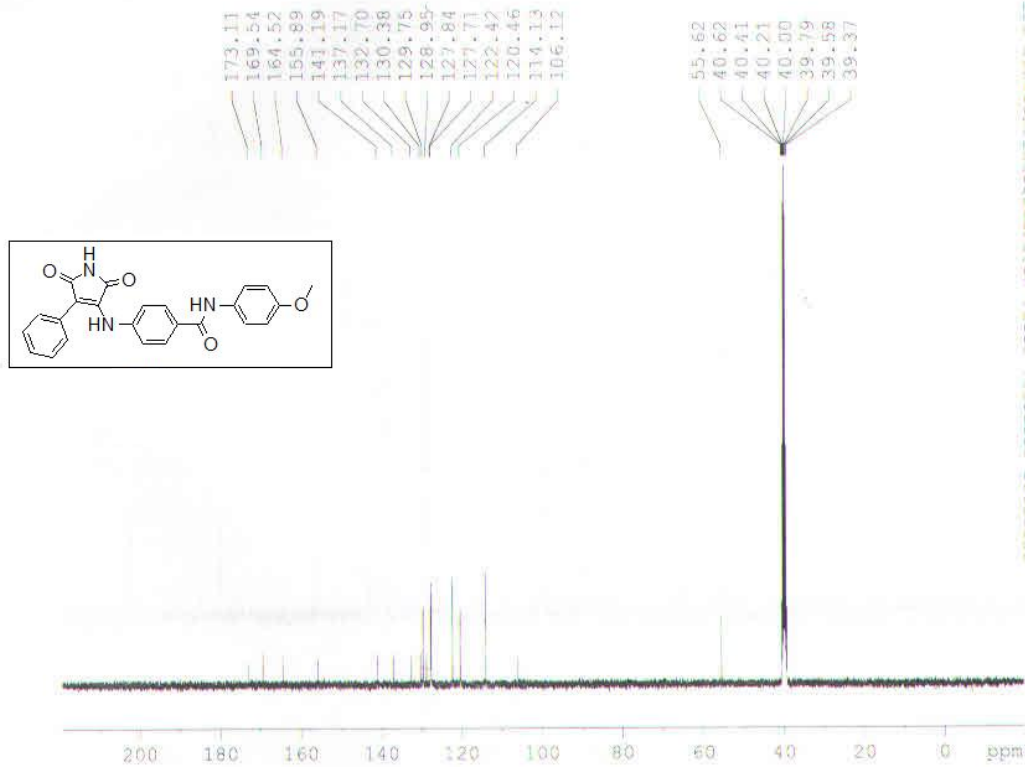
==== CHANNEL f1 =====
 NUC1 1H
 P1 10.90 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300023 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



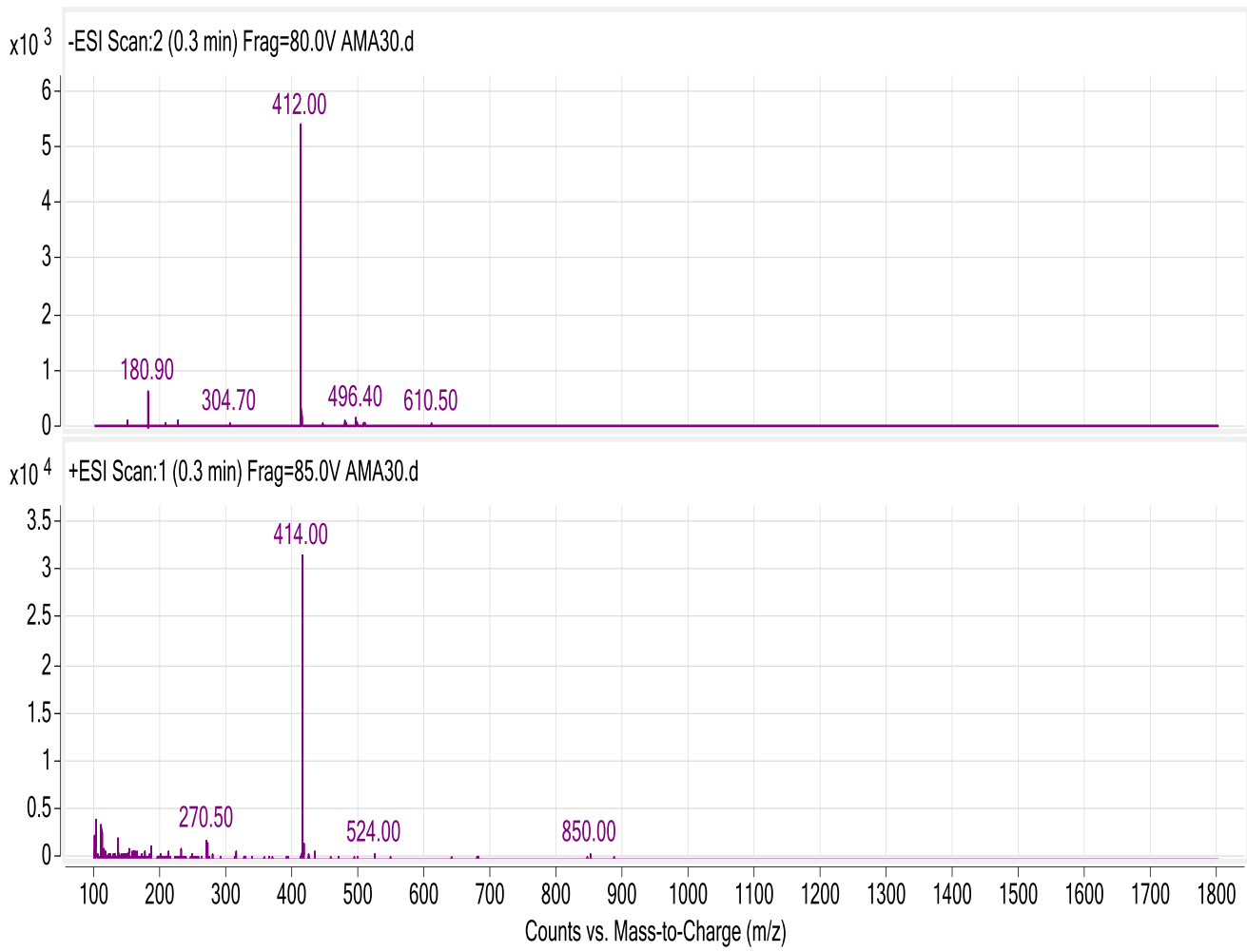
avtar saifpu@yahoo.co.in

Proton NMR (8b)



===== DATA PARAMETERS =====
NAME: JH SNS MUSHTAQ AT AMA 30
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_ : 20100601
Time : 11:22
INSTRUM : spect
PROBHD : 5 mm BBO 1H/13
PULPROG : zgpg30
TD : 65536
SOLVENT : DMSO
NS : 256
DS : 4
SWH : 24031.141 Hz
FIDRES : 0.36638 Hz
AQ : 1.61198 sec
RG : 328
WV : 20.800 MHz
DE : 6.00 MHz
TE : 299.2 K
SI : 0.0000000000000000
F1 : 101.6253600 MHz
DELTA : 1.59879584 sec
FID
===== CHANNEL f1 =====
NUC1 : 13C
PL1 : 0.00 MHz
PL2 : 12.00 MHz
SFO1 : 100.6261899 MHz
===== CHANNEL f2 =====
CPDPRG2 : zgpg30
NUC2 : 1H
PL2 : 0.00 MHz
PL3 : 0.00 MHz
PL4 : 18.48 MHz
PL5 : 20.00 MHz
SFO2 : 400.1418005 MHz
F2 - Processing parameters
SI : 1744
SF : 100.6261899 MHz
WDW : EM
SSB : 0
GB : 0
PC : 1.00 Hz
SC : 1.44

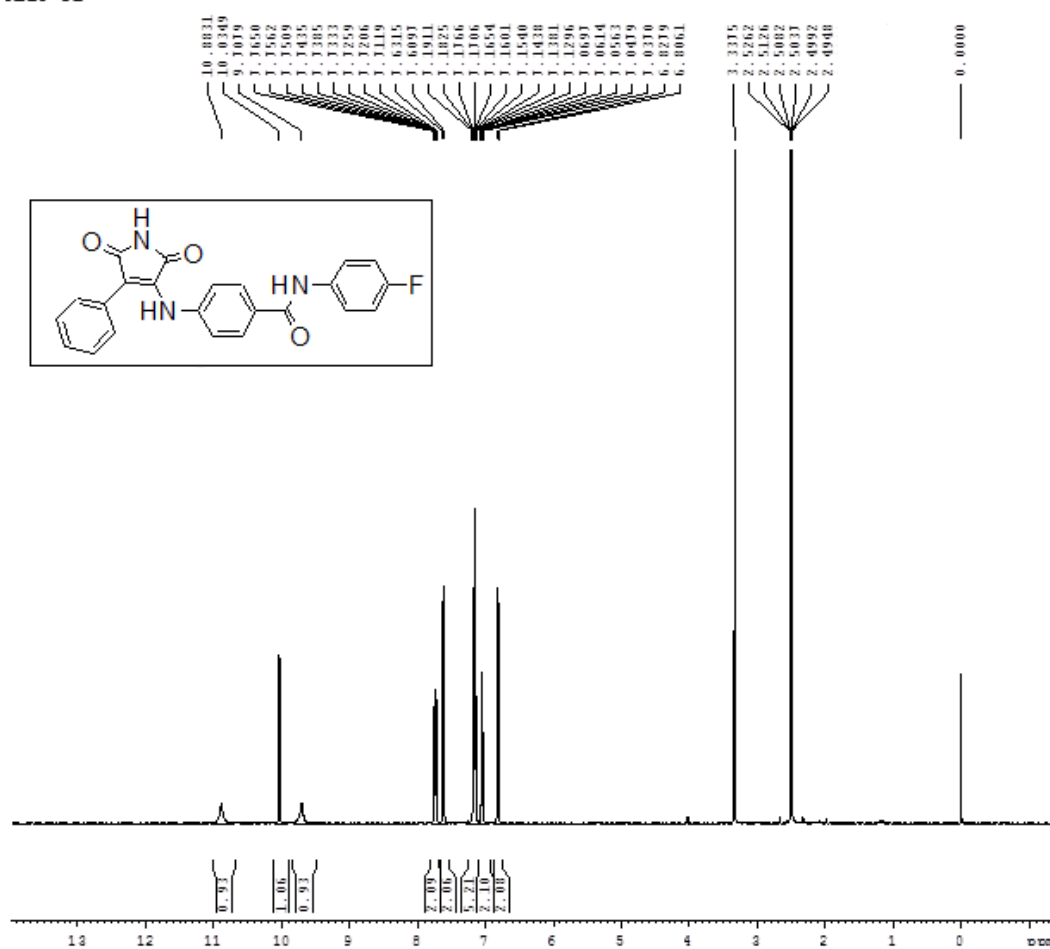
C-13 (8b)



ESI-MS (8b)

4-(2,5-dihydro-2,5-dioxo-4-phenyl-1H-pyrrol-3-ylamino)-N-(4-fluorophenyl)benzamide (8c)

AMA-31



BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME May15-2015
EXPNO 520
PROCNO 1

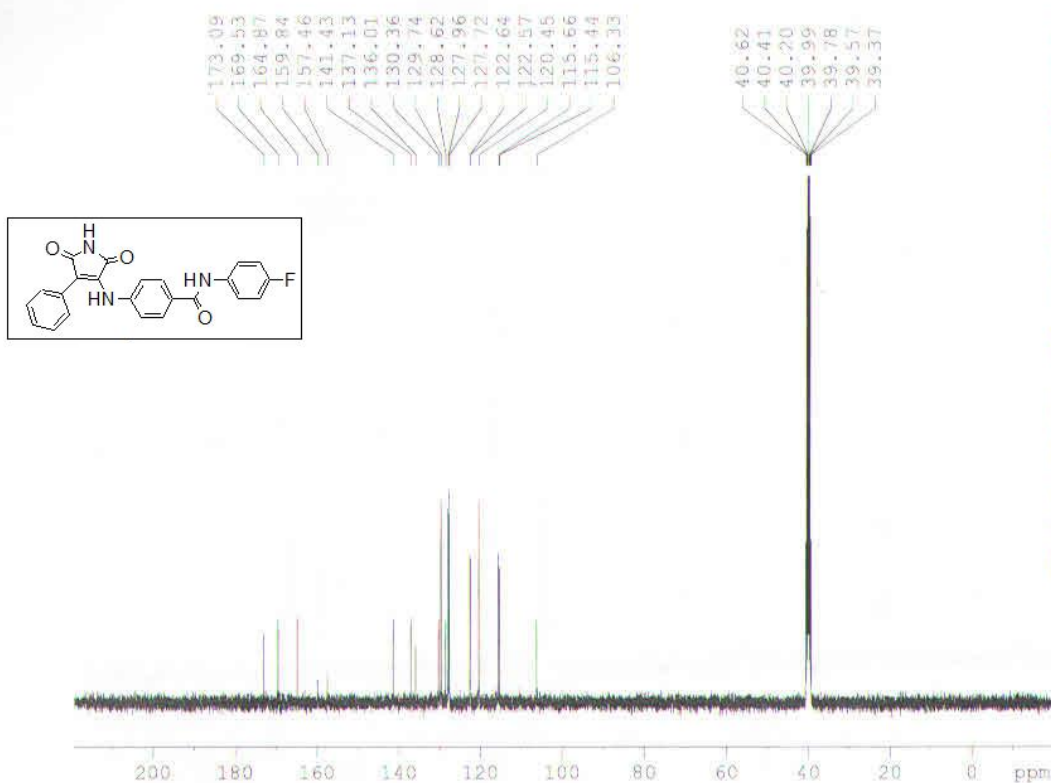
F2 - Acquisition Parameters
Date_ 20150515
Time 18.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.182399 Hz
AQ 2.7262477 sec
RG 812
DW 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

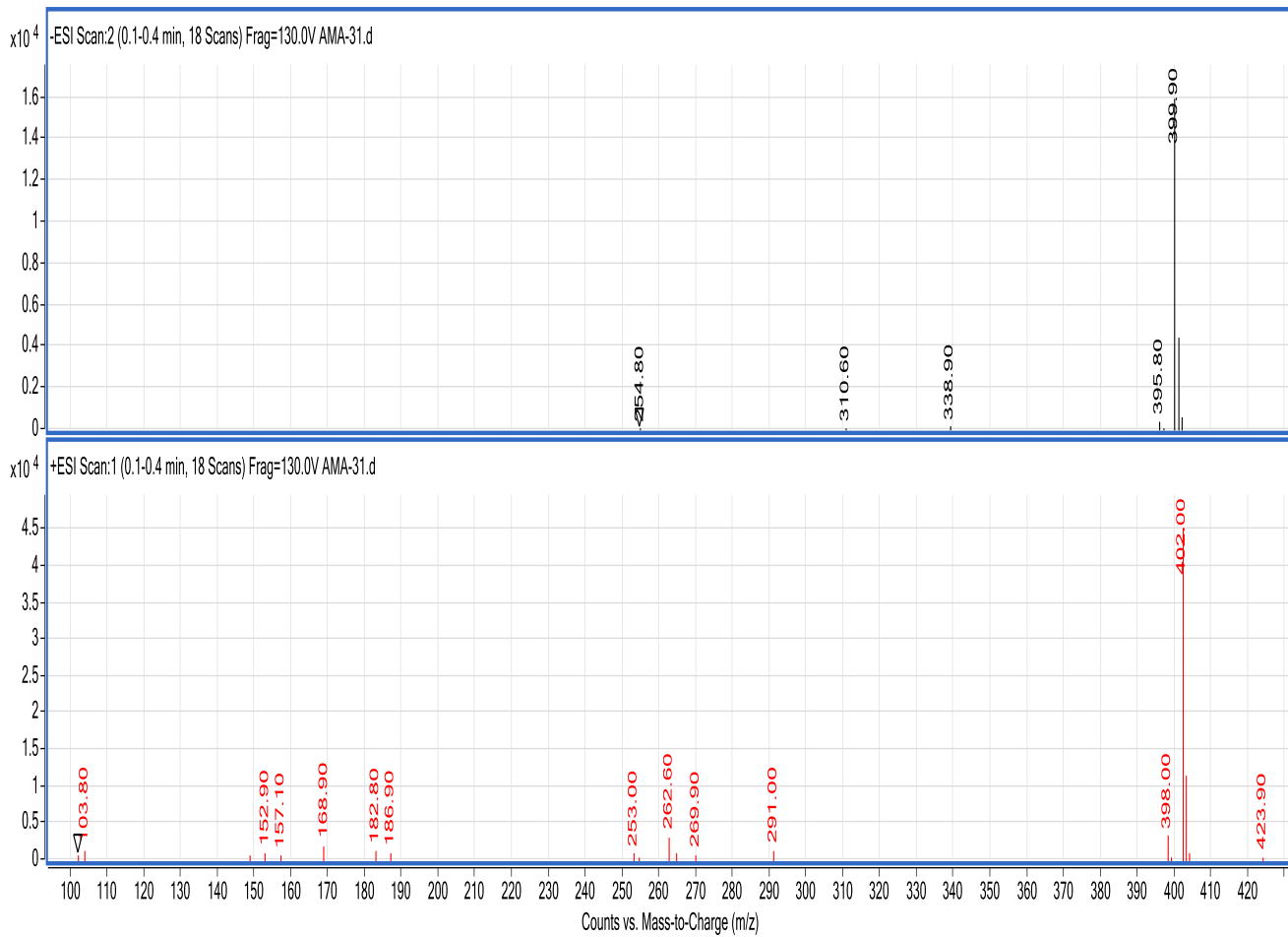
F2 - Processing parameters
SI 32768
SF 400.1300017 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

Proton NMR (8c)



C-13 (8c)



ESI-MS (8c)

4-(2,5-dihydro-2,5-dioxo-4-phenyl-1H-pyrrol-3-ylamino)-N-(4-chlorophenyl)benzamide (8d)

AMA-32



BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME May15-2015
EXPNO 530
PROCNO 1

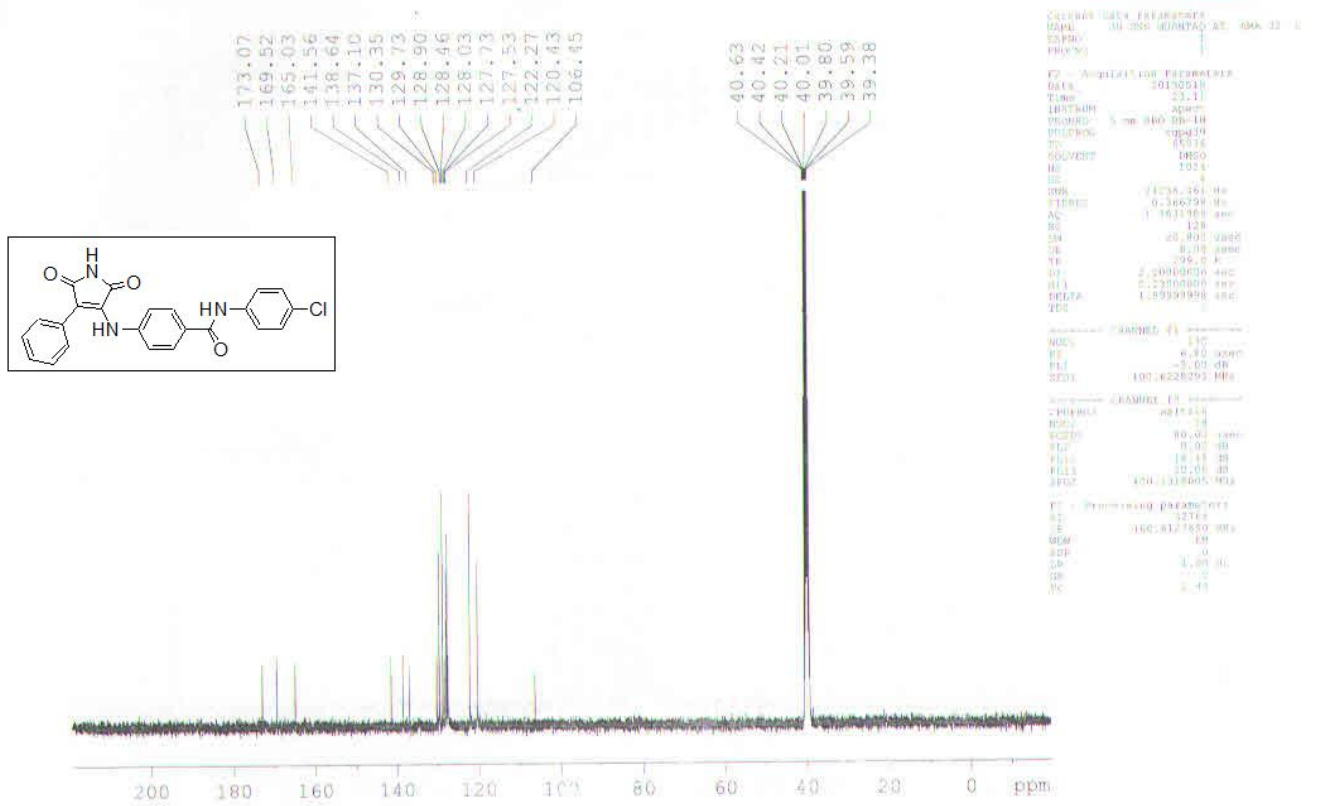
F2 - Acquisition Parameters
Date_ 20150515
Time 18.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 912
DN 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.0000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

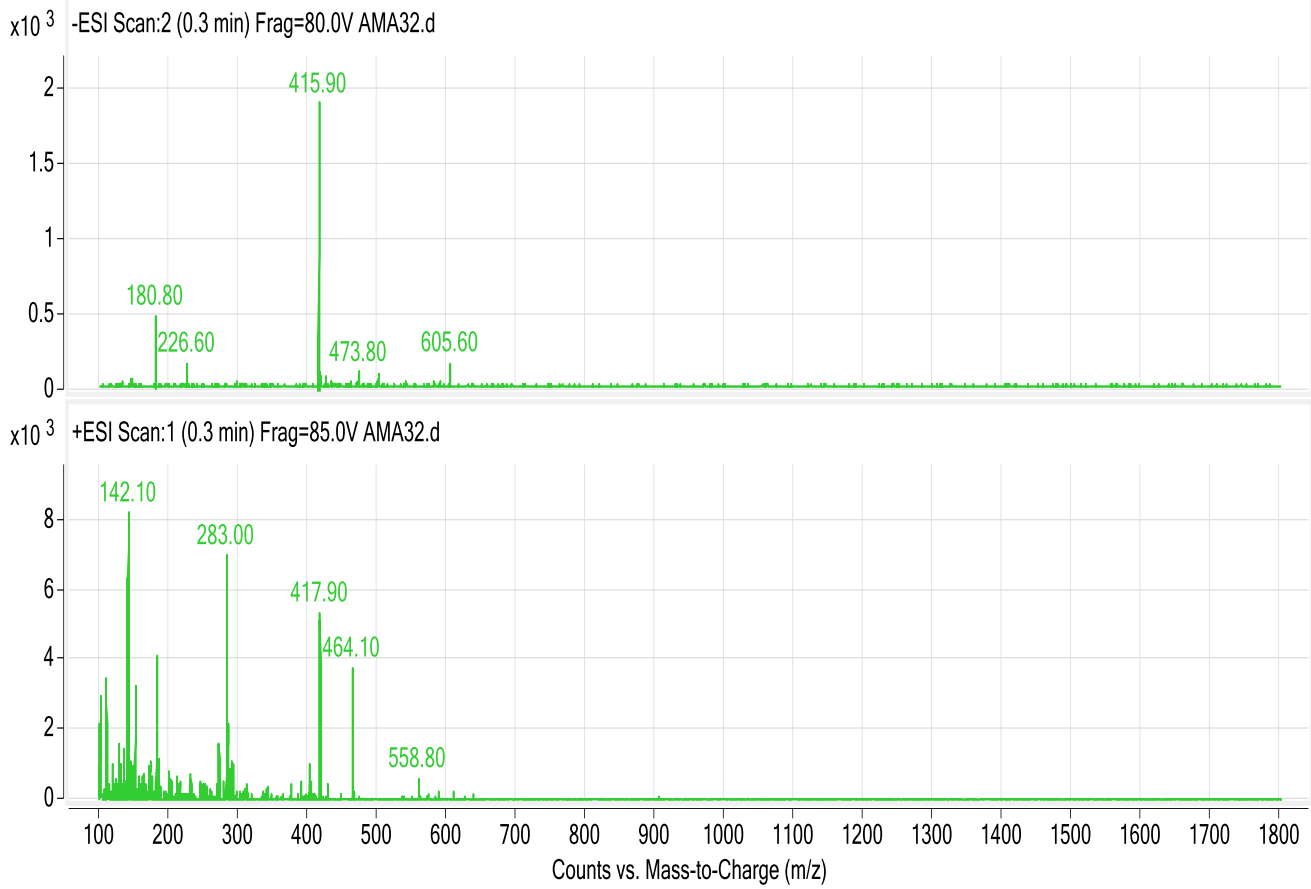
F2 - Processing parameters
SI 32768
SF 400.1300024 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

Proton NMR (8d)



C-13 (8d)



ESI-MS (8d)