Electronic Supplementary Material (ESI) for New Journal of Chemistry.

"Sonochemistry-An innovative opportunity towards a one-pot three-component synthesis of novel pyridylpiperazine derivatives catalysed by Meglumine in water"

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Sample Green Metrics calculation for the synthesis of 4a

Raw Material	(in mg)	Product and waste	(in mg)
Salicylaldehyde	122	Product	422
Pyridyl piperazine	162	Waste	61.4
4-bromo phenyl boronic acid	199	Recovered Meglumine (98%)	19.1
Meglumine	19.5		
Total	**502.5**	**Total**	**502.5**

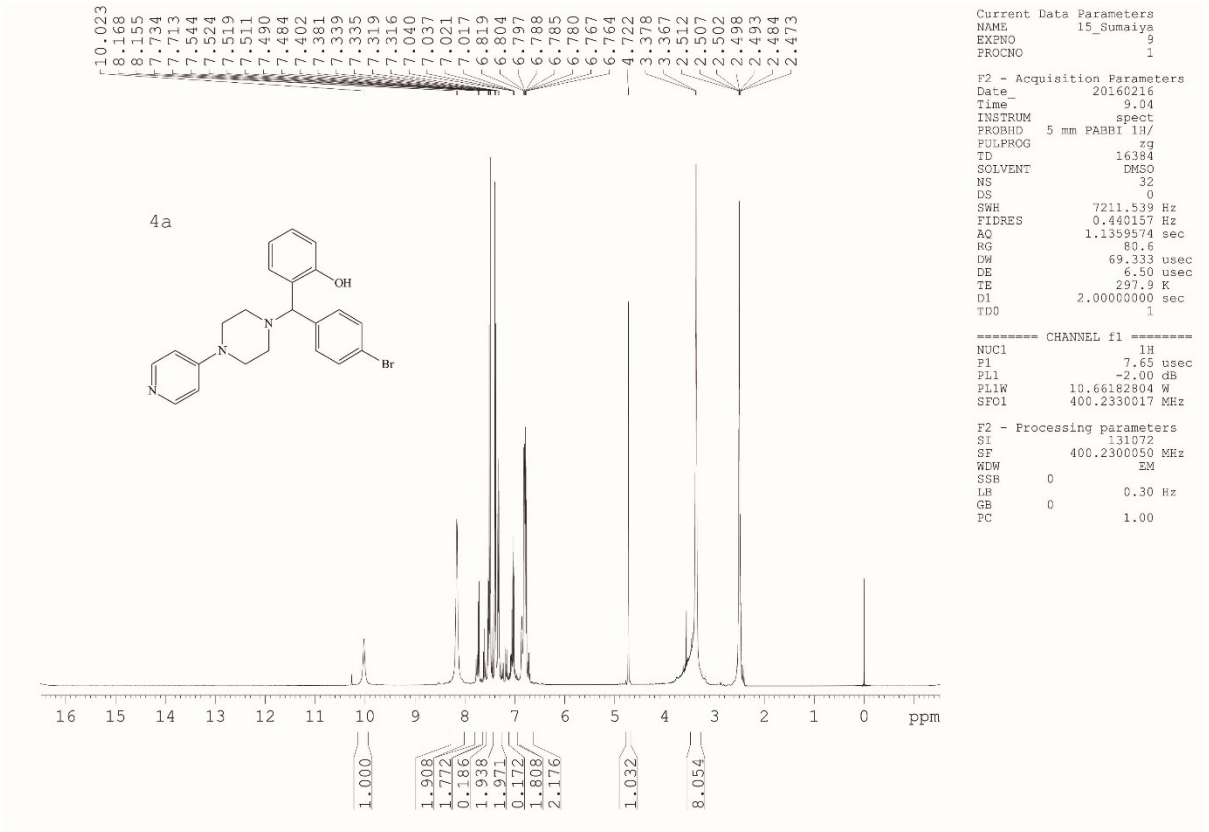
Consideration:

- Water used for the workup was not considered for the calculation.
- The solvent used for recrystallization was not considered.

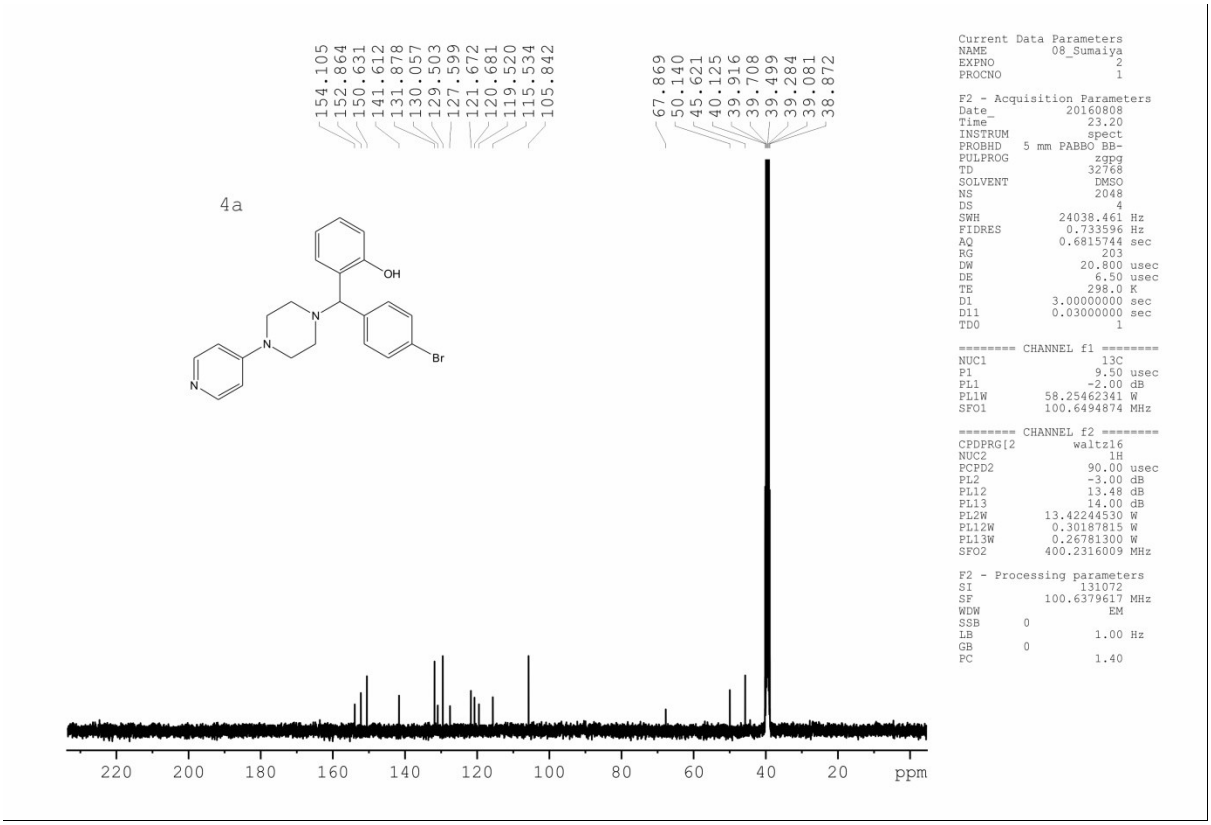
E-factor (E)	$= \frac{61.4 \text{ mg of waste}}{422 \text{ mg of product}}$ $= 0.1454$
Mass Intensity	$= \frac{502.5 \text{ mg of raw material used}}{422 \text{ mg of product}}$ $= 1.1907$
Process Mass Intensity (PMI)	= E factor + 1 = 0.1454 + 1 = 1.1454
Atom economy	$= \frac{\text{Molecular mass of the desired product}}{\text{Molecular mass of all the reactant}}$ $= \frac{422}{122 + 162 + 199}$ $= \frac{422}{483}$ = 0.8737
Generalised Reaction Mass efficiency (RME)	$= \frac{1}{1 + E}$ $= \frac{1}{1 + 0.1454}$ = 0.8730
Kernal RME	= Atom economy × Yield = 0.8737 × 0.98 = 0.8563
Curzon's RME	$= \frac{\text{Mass of the product}}{\text{Mass of the reactants + catalyst}}$ $= \frac{0.422}{0.483 + 19.5}$ = 0.0211
Environmental impact factor based on molecular weight (E_{mw})	$= \frac{1}{\text{Atom economy}} - 1$ $= \frac{1}{0.8737} - 1$

	= 1.1445 - 1 = 0.1445
Yield (%)	$= \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$ $= \frac{413.6}{422} \times 100$ = 0.98 × 100 = 98%

^{1}H NMR spectrum of 4a

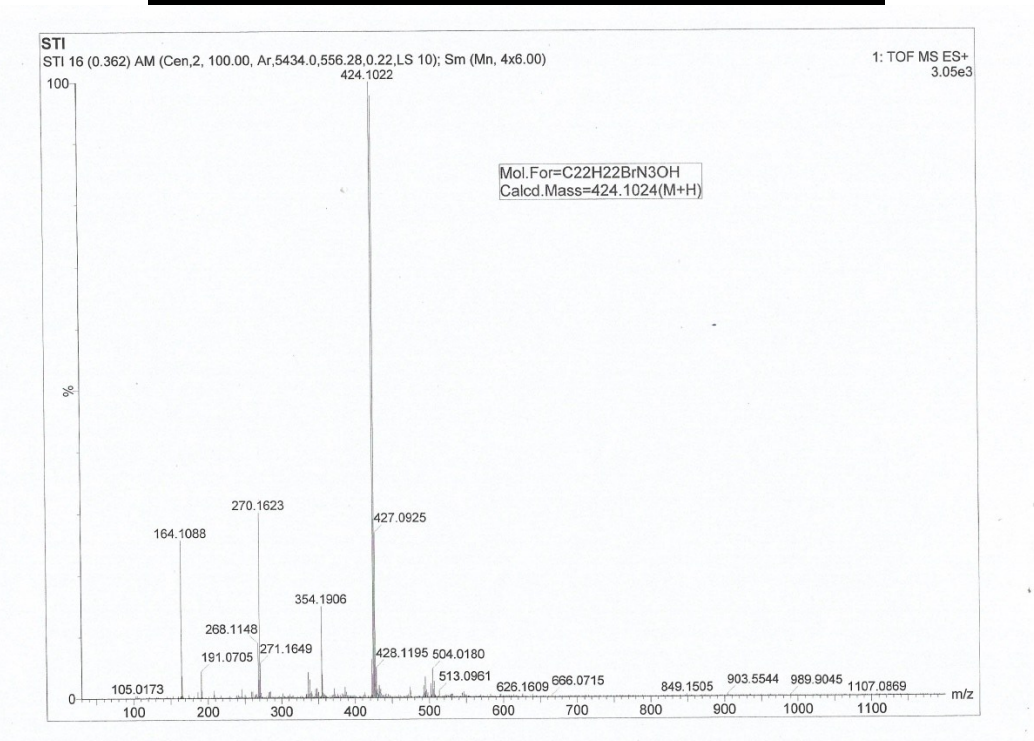


^{13}C NMR spectrum of 4a



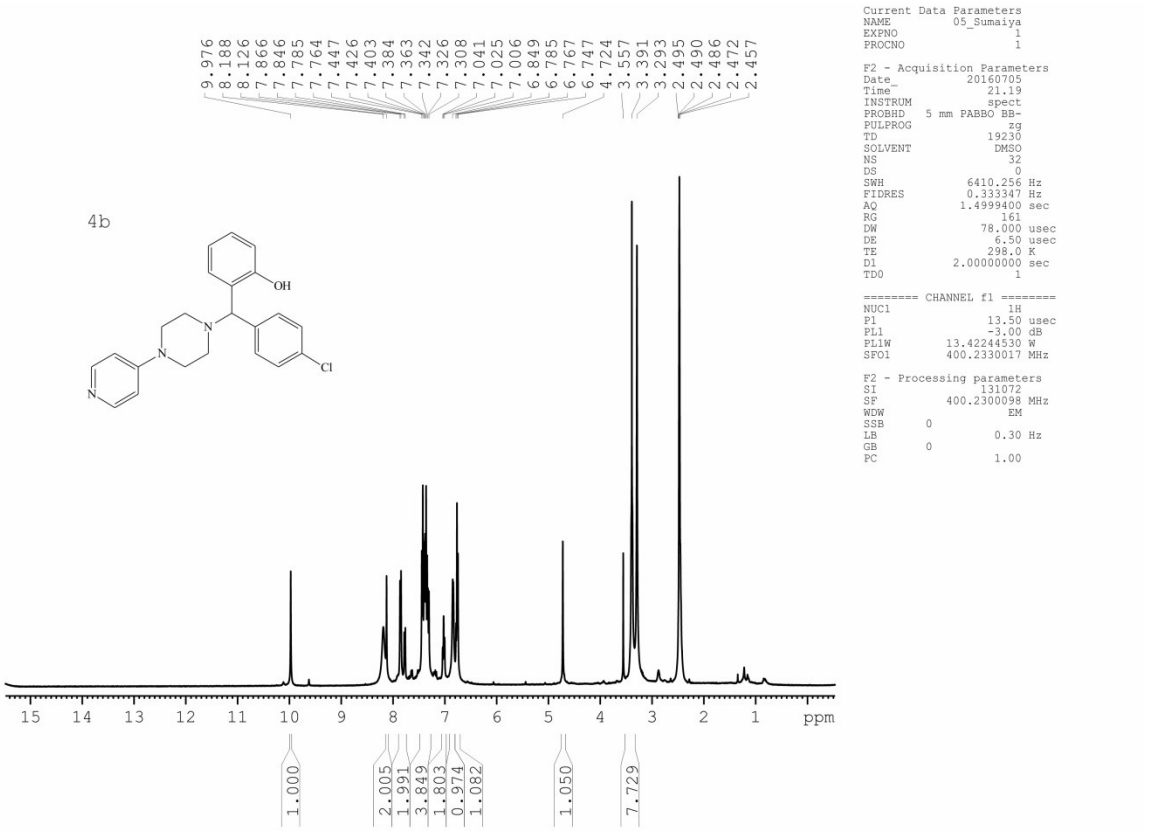
HRMS of 4a

[redacted]



[redacted]

^{1}H NMR spectrum of 4b

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EXPNO                  1
PROCNO                 1

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PULPROG              zg
TD                19230
SOLVENT            DMSO
NS                   32
DS                    0
SWH            6410.256 Hz
FIDRES         0.333347 Hz
AQ             1.4999400 sec
RG                 161
DW              78.000 usec
DE               6.50 usec
TE              298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1               -3.00 dB
PL1W        13.42244530 W
SFO1        400.2330017 MHz

F2 - Processing parameters
SI                131072
SF          400.2300098 MHz
WDW                   EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
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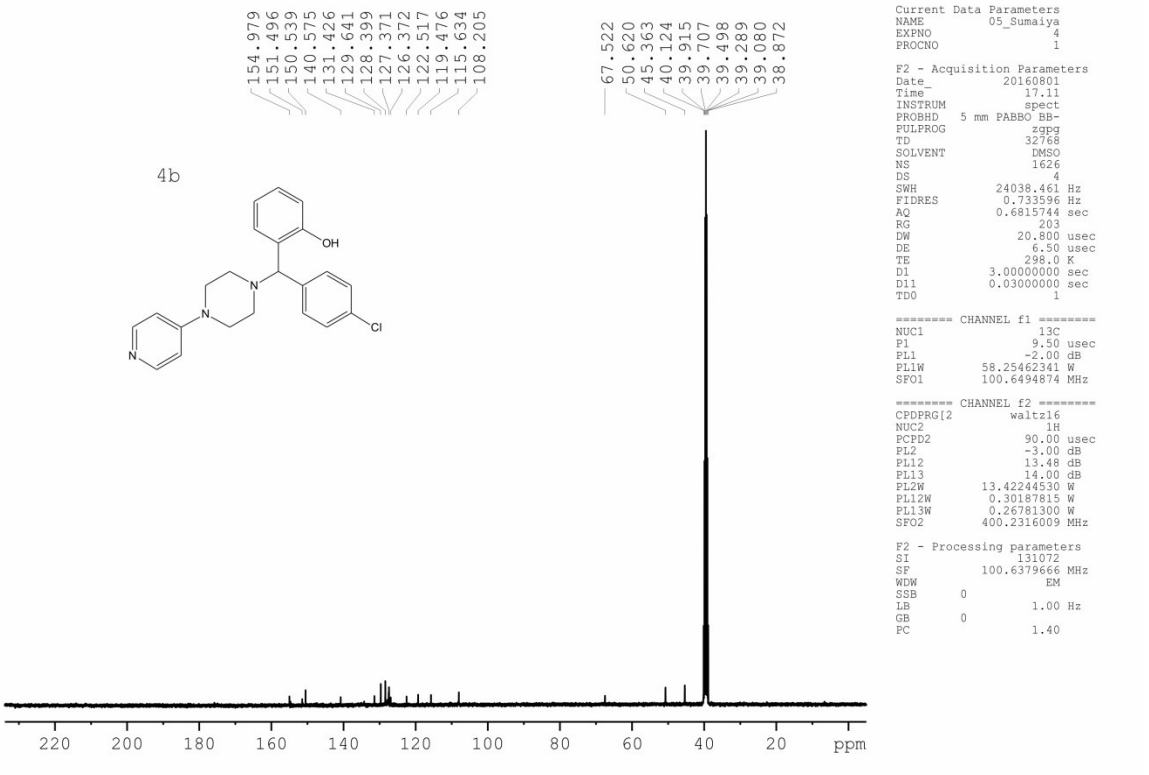
^{13}C NMR spectrum of 4b

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EXPNO                  4
PROCNO                 1

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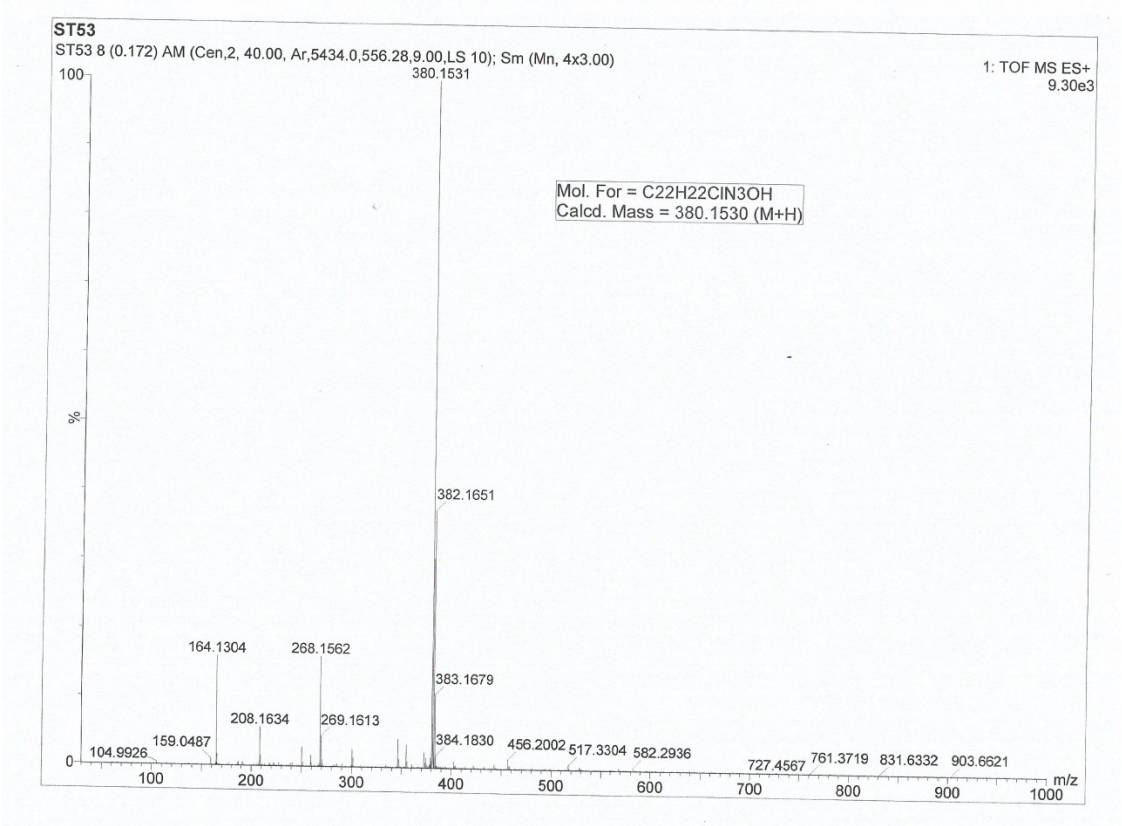
PULPROG           zgpg
TD                32768
SOLVENT            DMSO
NS                  1626
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                 203
DW              20.800 usec
DE               6.50 usec
TE              298.0 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1               -2.00 dB
PL1W        58.25462341 W
SFO1        100.6494874 MHz

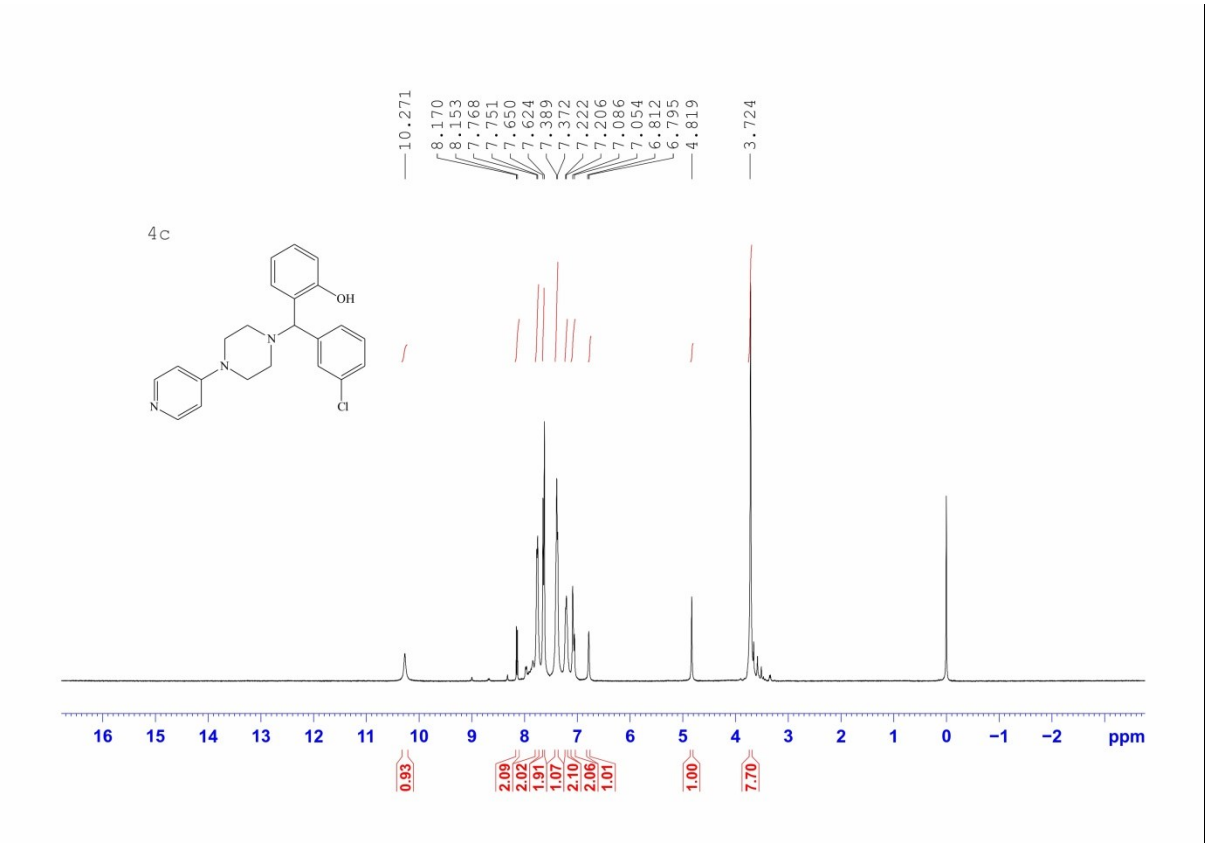
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              13.48 dB
PL13              14.00 dB
PL2W        13.42244530 W
PL12W        0.30187815 W
PL13W        0.26781300 W
SFO2        400.2316009 MHz

F2 - Processing parameters
SI                131072
SF          100.6379666 MHz
WDW                   EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

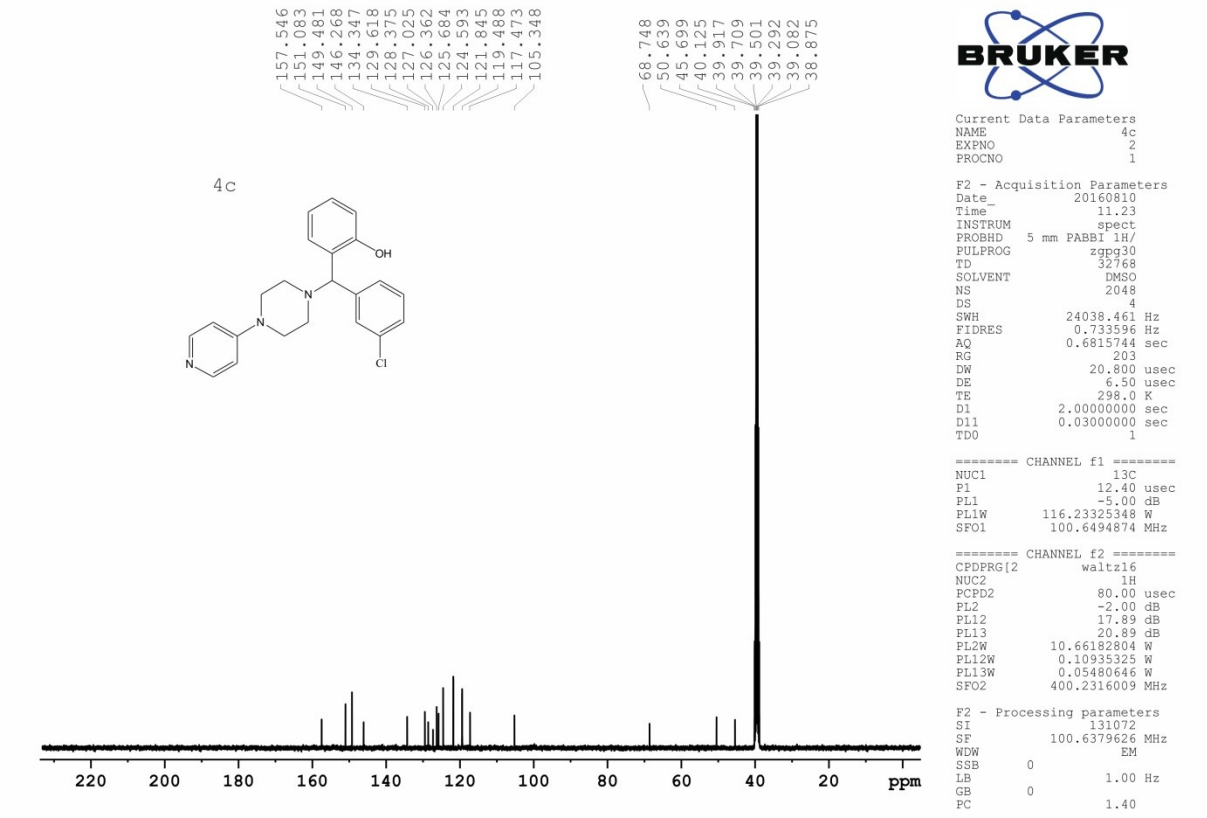
HRMS of 4b



^{1}H NMR spectrum of 4c



^{13}C NMR spectrum of 4c

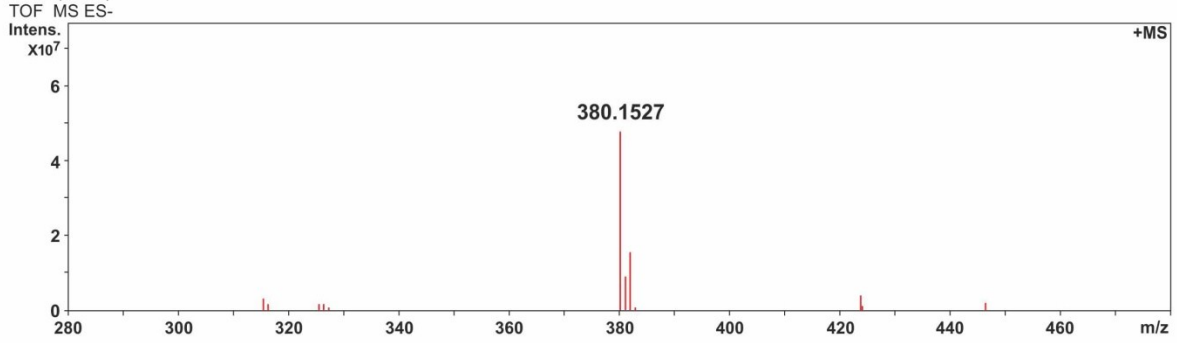


HRMS of 4c

Elemental Composition Report **Page 1**

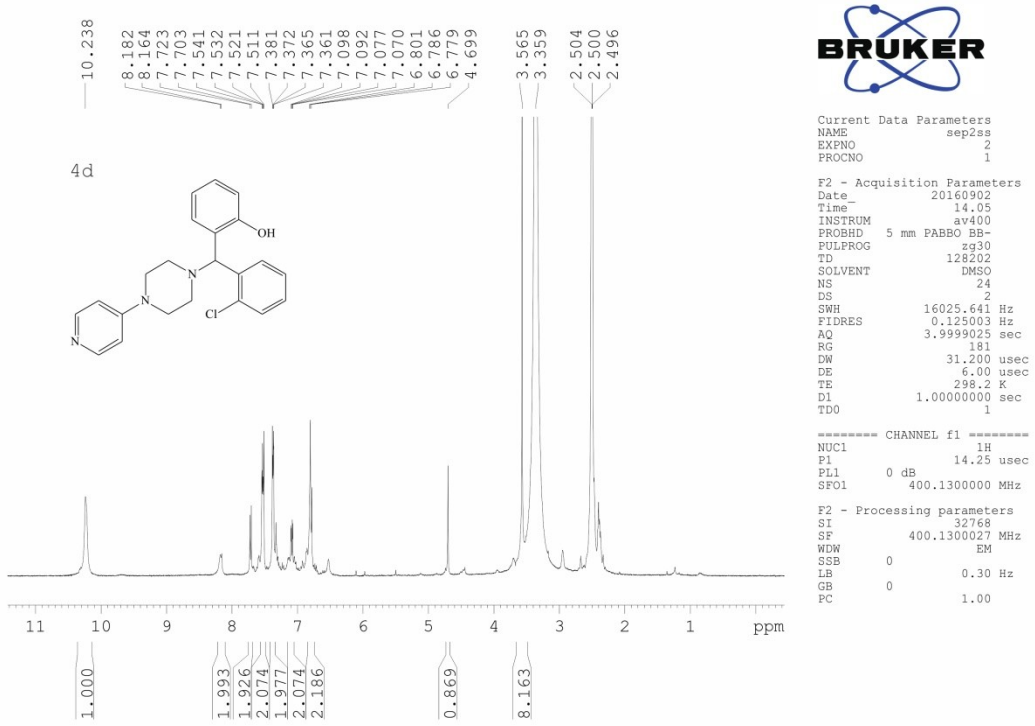
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
15 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-1
4c 32 (0.751)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
380.1527	1	C 22 H 23 Cl N 3 O 4	100.00	380.1530	0.5	1.7	8.8	13.1	even	ok

^{1}H NMR spectrum of 4d



BRUKER

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EXPNO                      2
PROCNO                     1

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Time                  14.05
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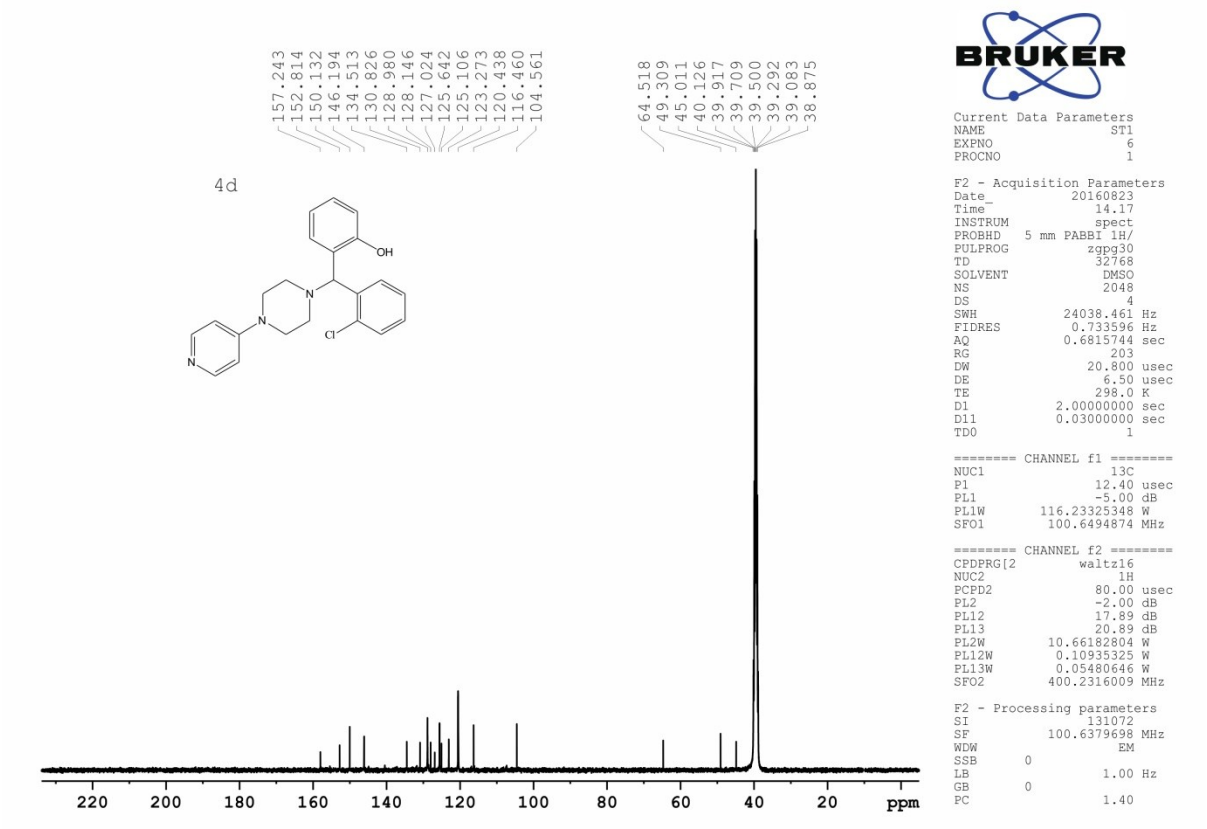
PULPROG               zg30
TD                  128202
SOLVENT               DMSO
NS                       24
DS                        2
SWH               16025.641 Hz
FIDRES             0.125003 Hz
AQ                3.9999025 sec
RG                      181
DW                   31.200 usec
DE                    6.00 usec
TE                   298.2 K
D1               1.00000000 sec
TD0                        1

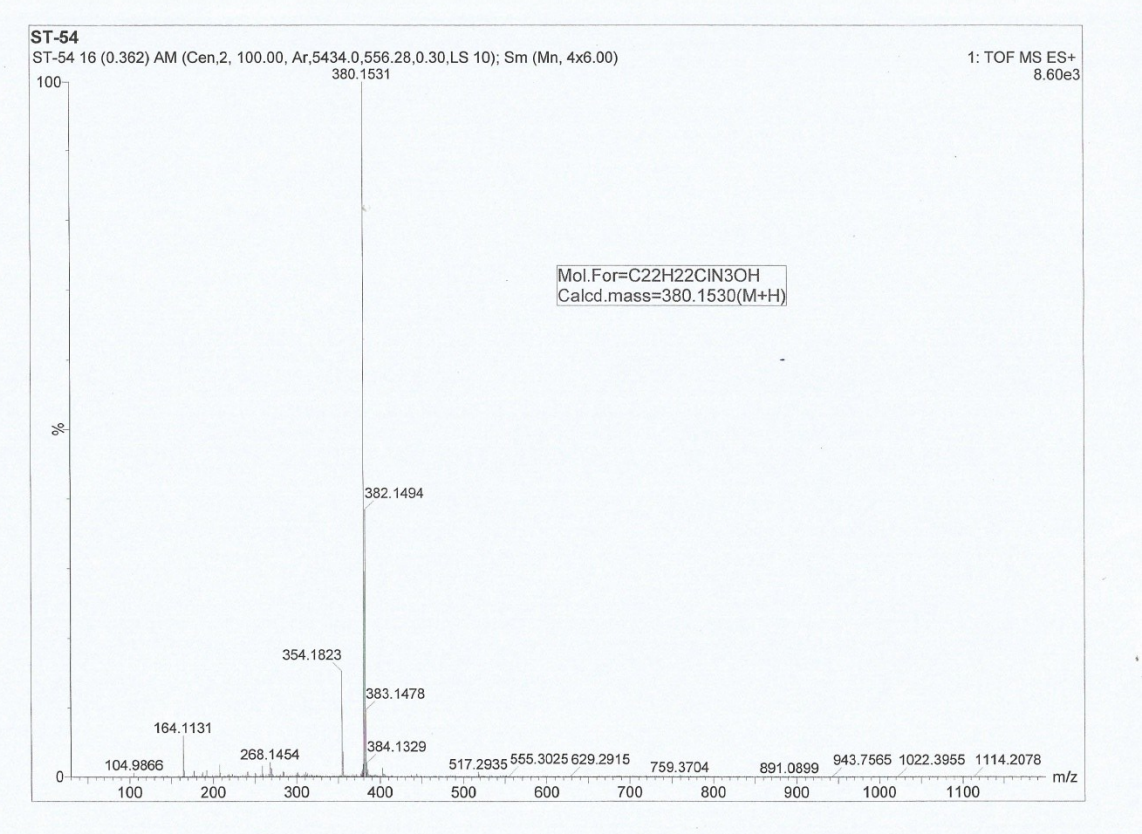
======== CHANNEL f1 ========
NUC1                      1H
P1                    14.25 usec
PL1          0 dB
SFO1          400.1300000 MHz

F2 - Processing parameters
SI                     32768
SF               400.1300027 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

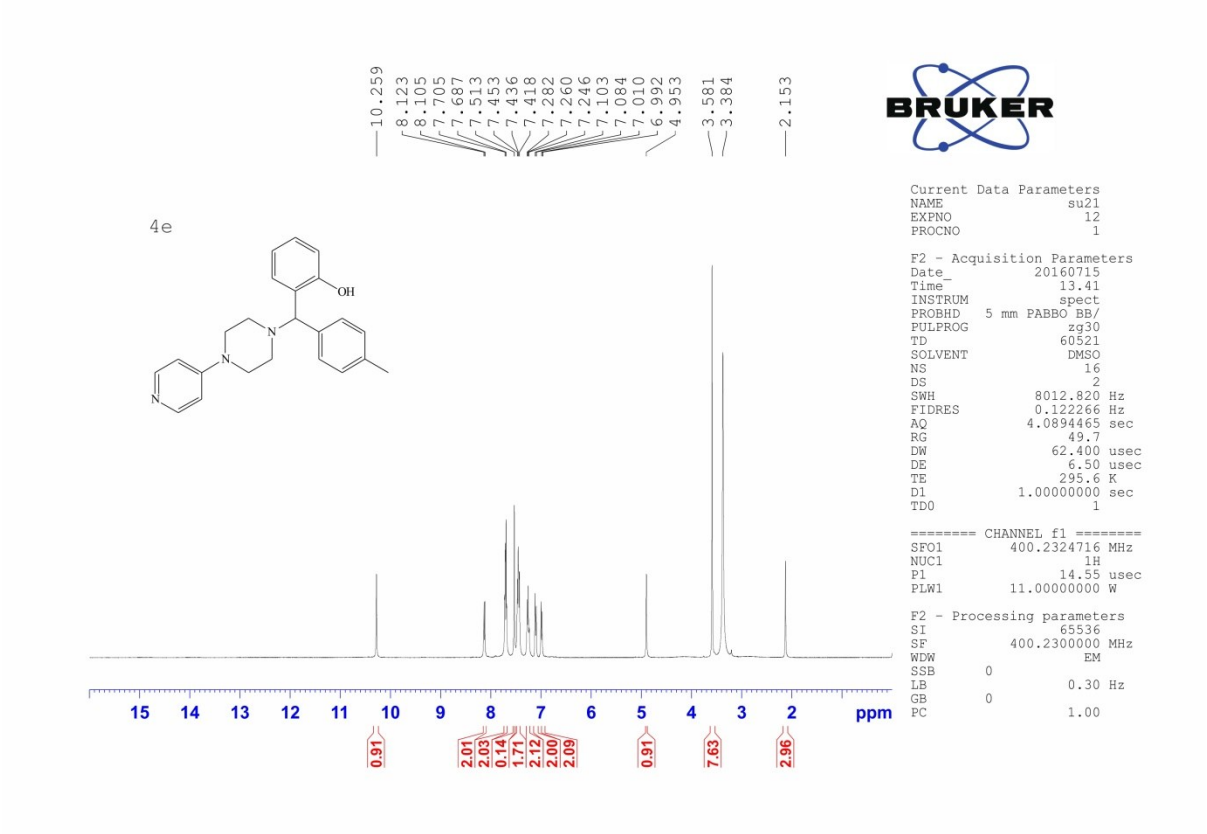
^{13}C NMR spectrum of 4d


157.243
152.814
150.132
146.194
134.513
130.826
128.980
128.146
127.024
125.642
125.106
123.273
120.438
116.460
104.561
64.518
49.309
45.011
40.126
39.917
39.709
39.500
39.292
39.083
38.875
4d
OH
N
N
N
Cl
220
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME ST1
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160823
Time 14.17
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.40 usec
PL1 -5.00 dB
PL1W 116.23325348 W
SFO1 100.6494874 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.89 dB
PL13 20.89 dB
PL2W 10.66182804 W
PL12W 0.10935325 W
PL13W 0.05480646 W
SFO2 400.2316009 MHz
F2 - Processing parameters
SI 131072
SF 100.6379698 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

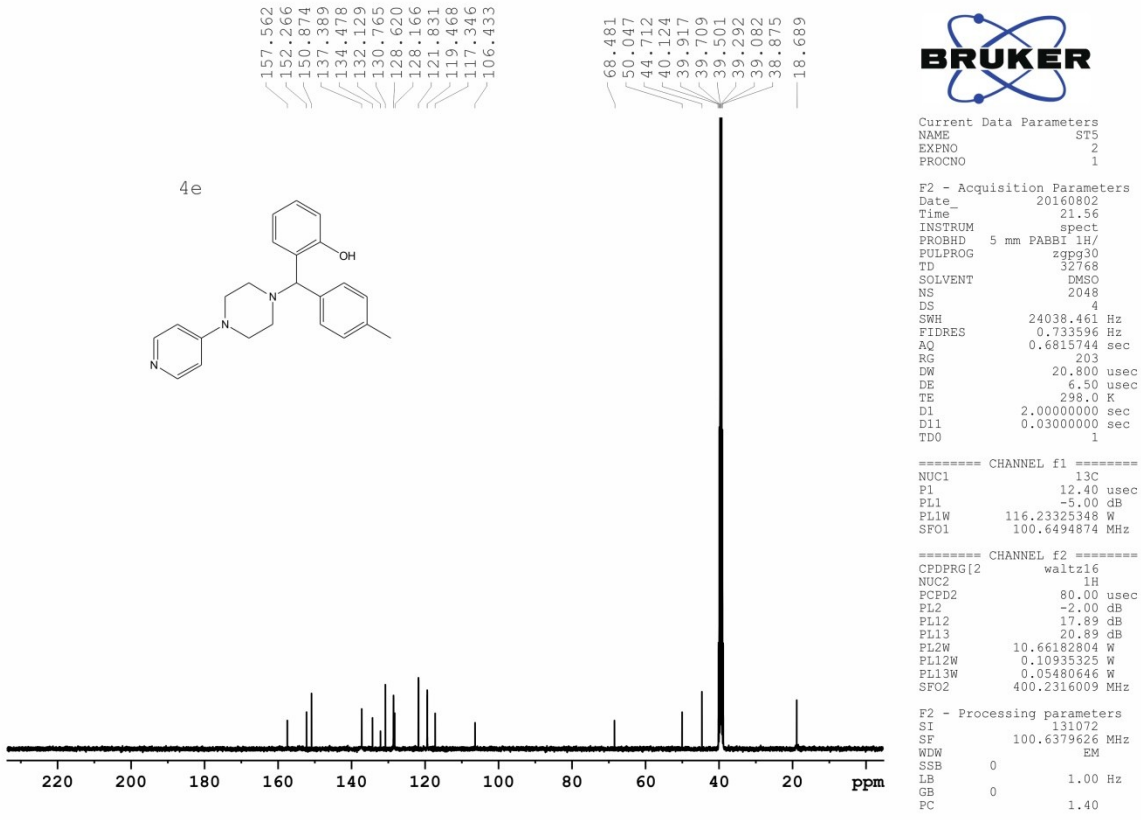
HRMS of 4d



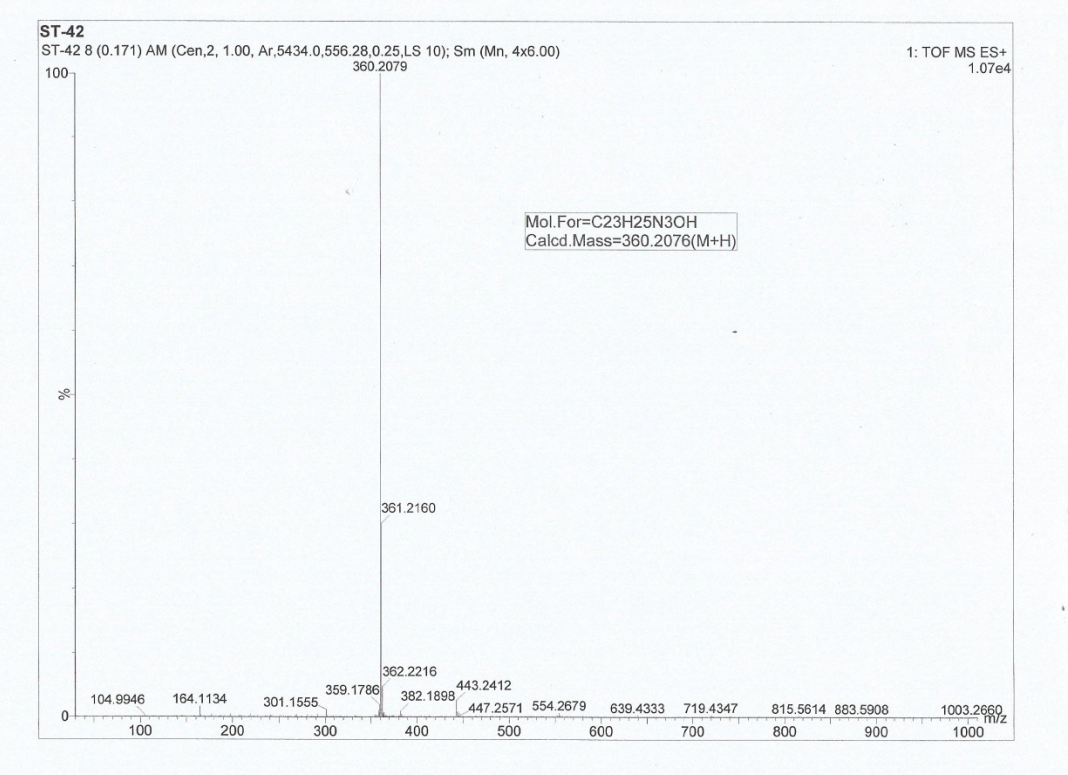
^{1}H NMR spectrum of 4e



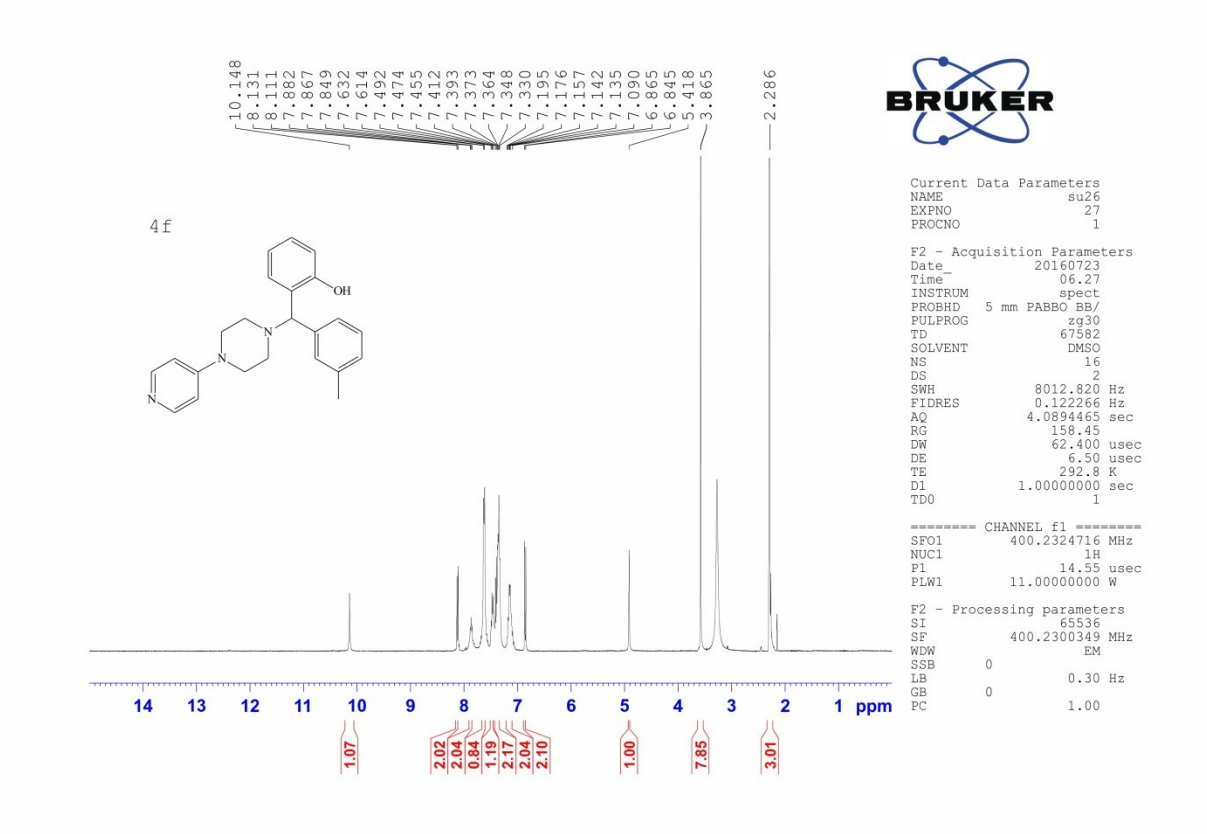
^{13}C NMR spectrum of 4e



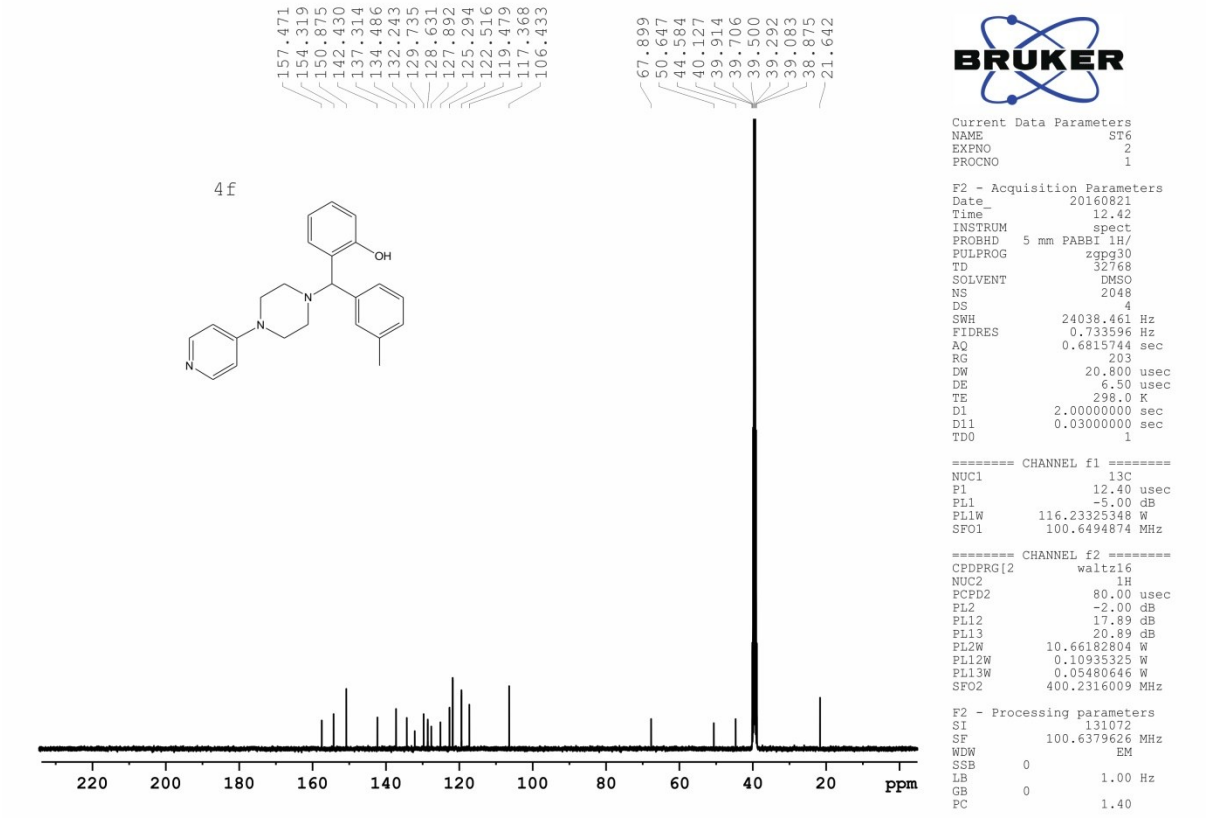
HRMS of 4e



^{1}H NMR spectrum of 4f



^{13}C NMR spectrum of 4f



HRMS of 4f

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

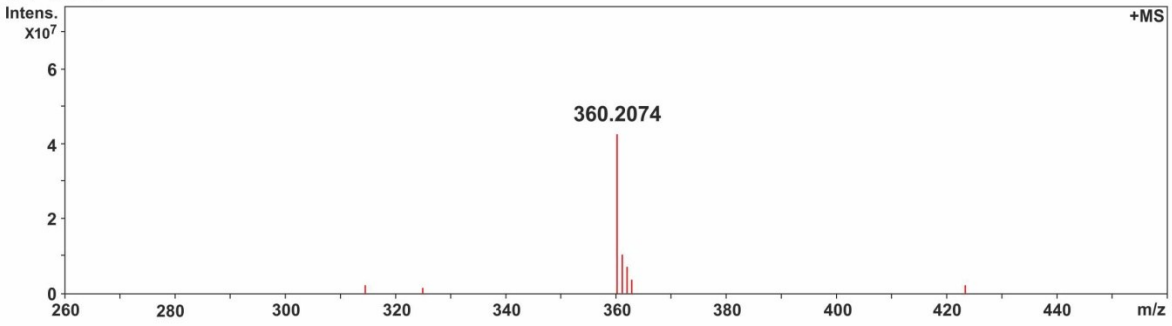
18 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5

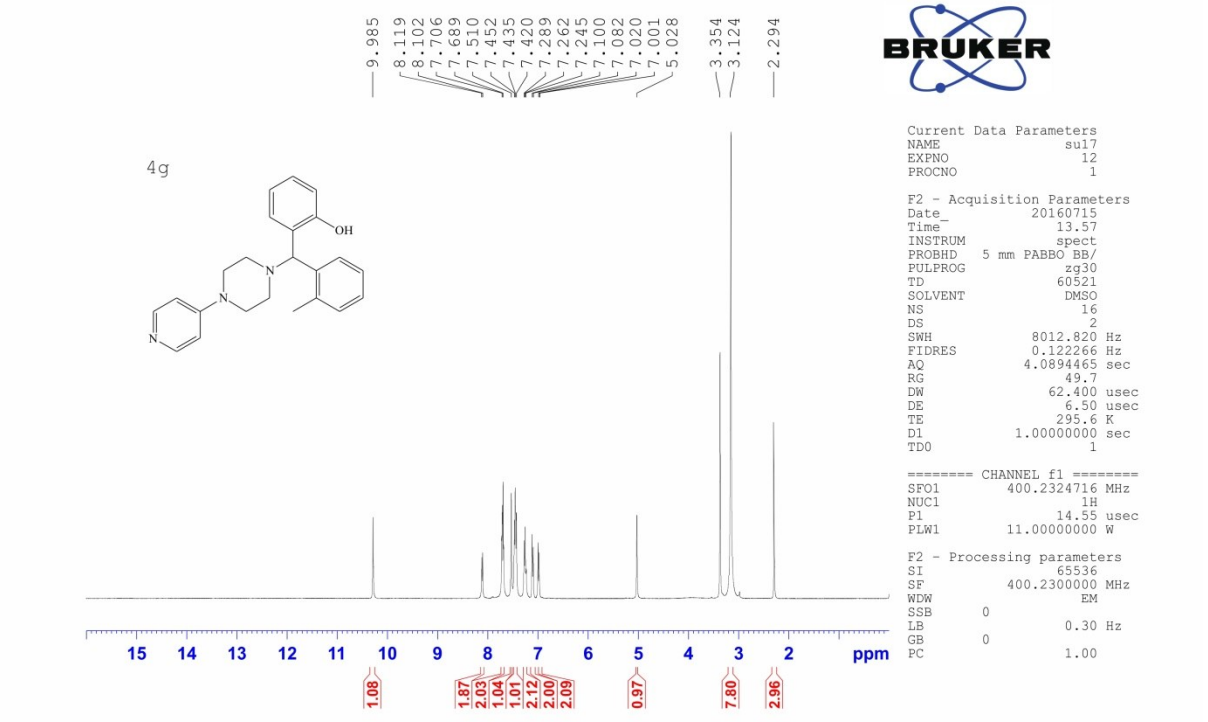
4f 27 (0.528)

TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
360.2074	1	C 23 H 26 N 3 O	100.00	360.2076	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4g



BRUKER

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EXPNO                   12
PROCNO                   1

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Time                13.57
INSTRUM             spect
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PULPROG             zg30
TD                 60521
SOLVENT             DMSO
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                   49.7
DW                 62.400 usec
DE                  6.50 usec
TE                 295.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.2324716 MHz
NUC1                   1H
P1                  14.55 usec
PLW1         11.00000000 W

F2 - Processing parameters
SI                  65536
SF           400.2300000 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
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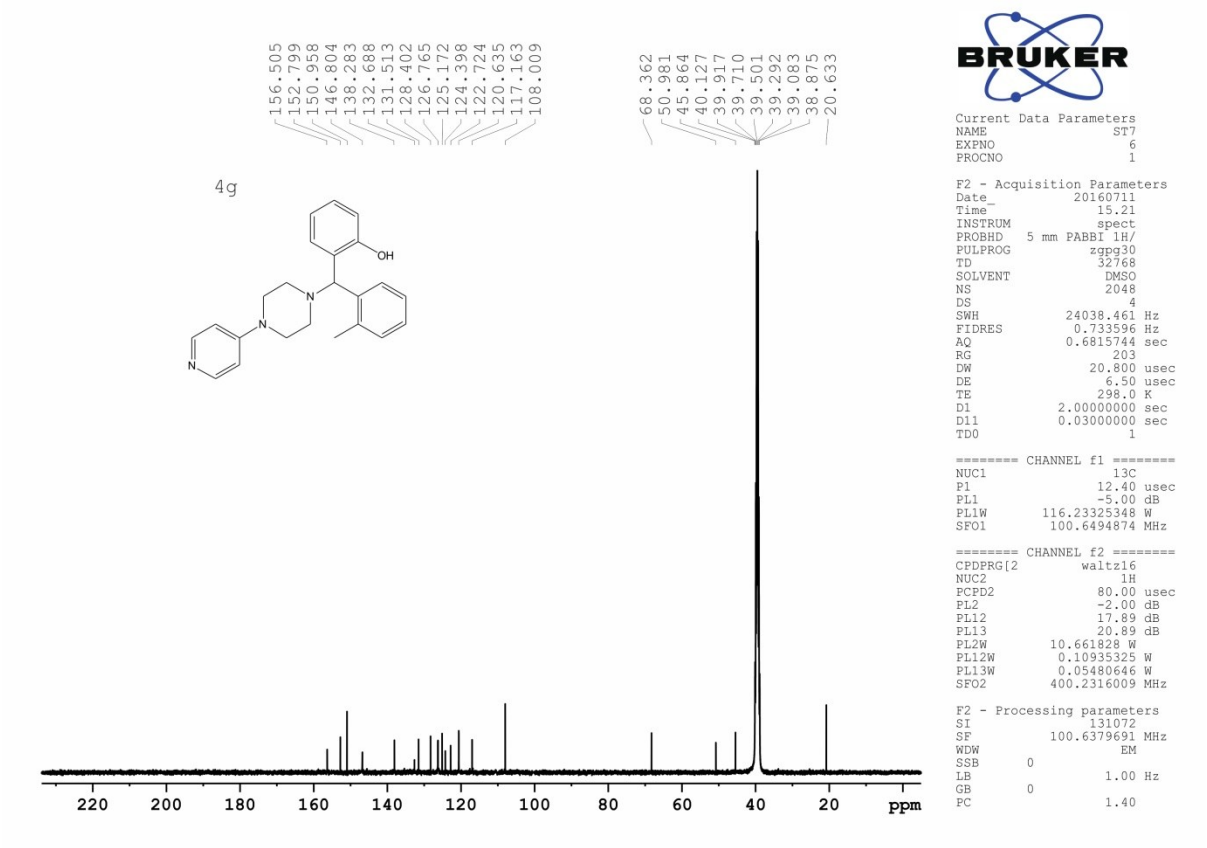
^{13}C NMR spectrum of 4g



HRMS of 4g

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

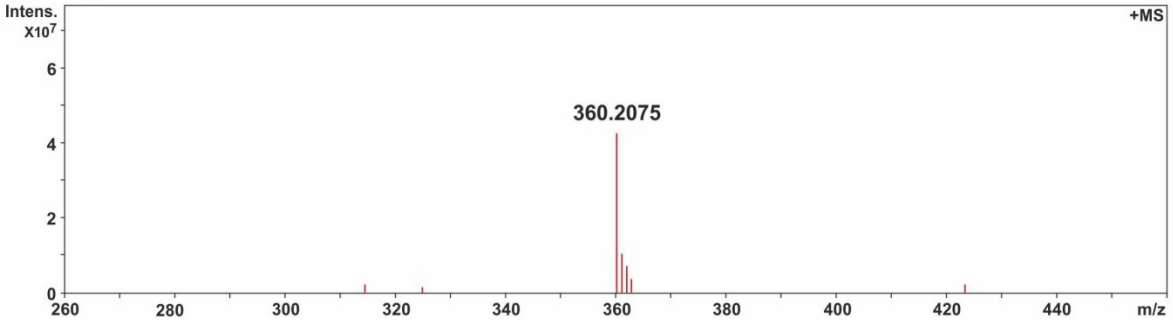
18 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5

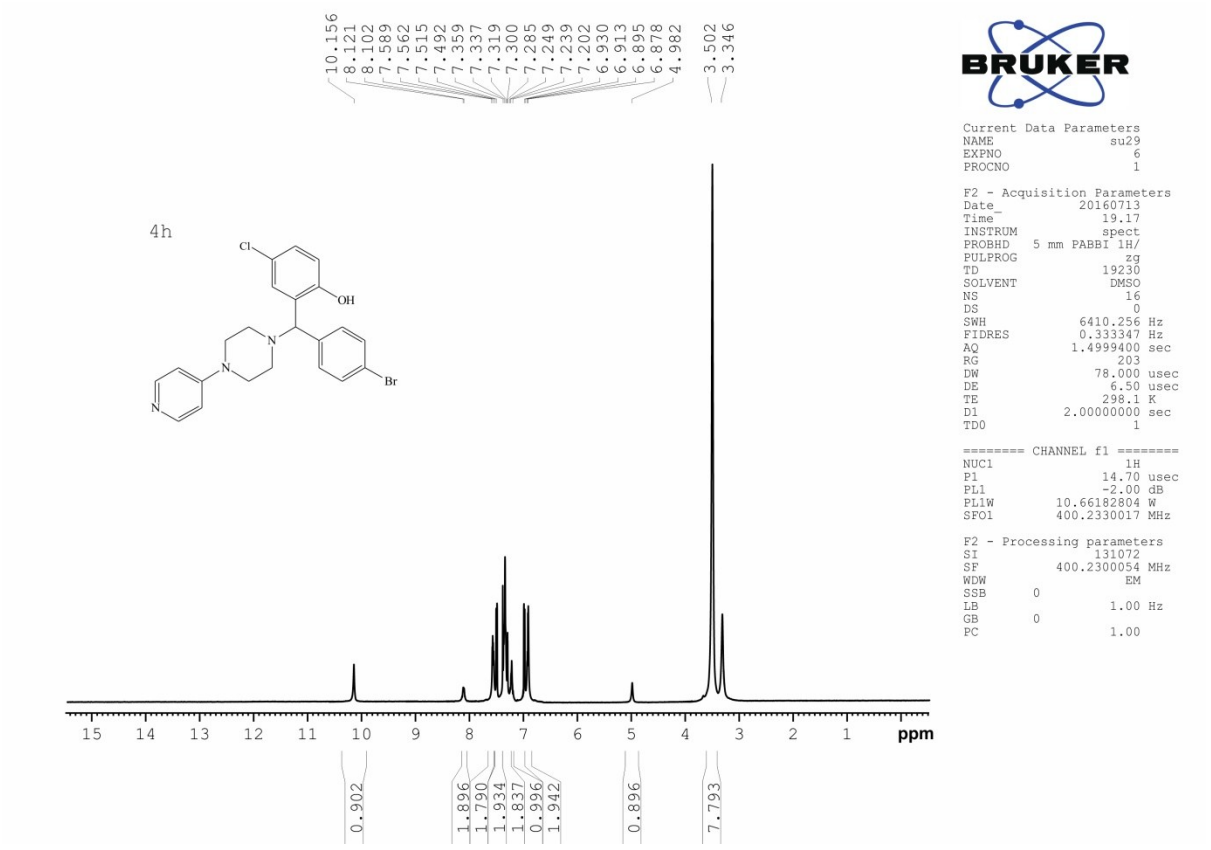
4g 27 (0.528)

TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
360.2075	1	C 23 H 26 N 3 O	100.00	360.2076	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4h

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NAME                  su29
EXPNO                    6
PROCNO                   1

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Time              19.17
INSTRUM           spect
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PULPROG             zg
TD                19230
SOLVENT            DMSO
NS                   16
DS                    0
SWH            6410.256 Hz
FIDRES         0.333347 Hz
AQ             1.4999400 sec
RG                  203
DW               78.000 usec
DE                6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.70 usec
PL1               -2.00 dB
PL1W        10.66182804 W
SFO1        400.2330017 MHz

F2 - Processing parameters
SI                 131072
SF           400.2300054 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
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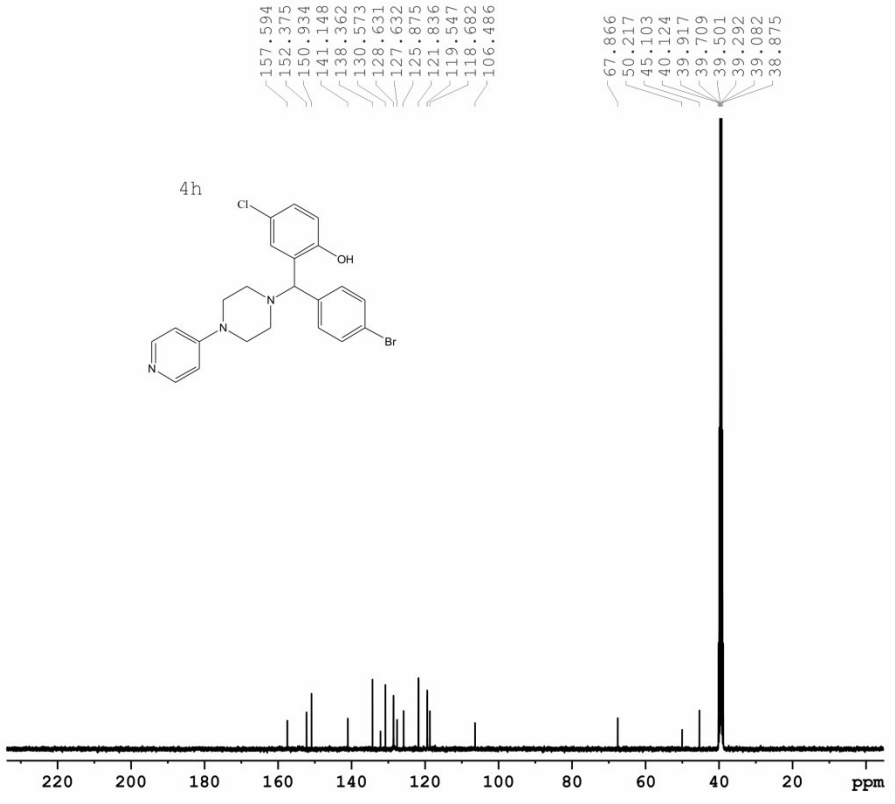
^{13}C NMR spectrum of 4h

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EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160810
Time              20.02
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                32768
SOLVENT            DMSO
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.40 usec
PL1               -5.00 dB
PL1W       116.23325348 W
SFO1        100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL2W        10.66182804 W
PL12W        0.10935325 W
PL13W        0.05480646 W
SFO2        400.2316009 MHz

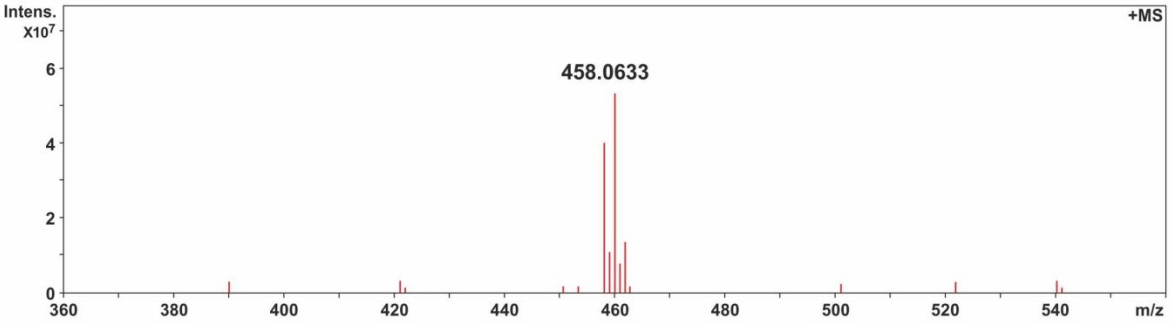
F2 - Processing parameters
SI                 131072
SF           100.6379626 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

HRMS of 4h

Elemental Composition Report **Page 1**

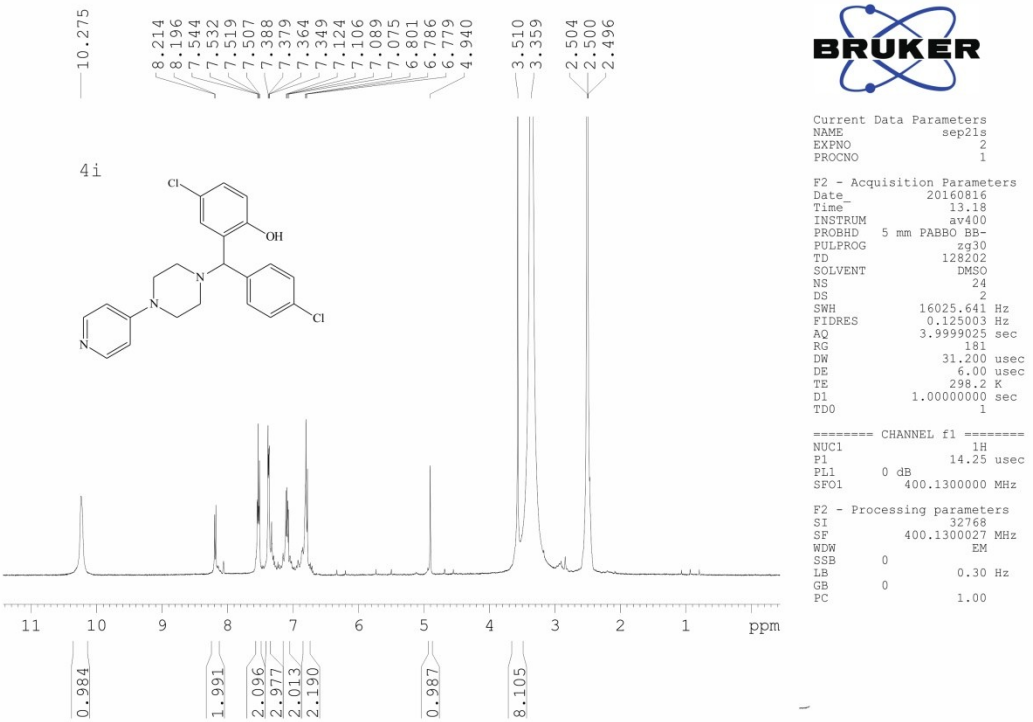
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5 Br: 0-1 Cl: 0-1
4h 40 (2.328)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
458.0633	1	C 22 H 22 Br Cl N 3 O	100.00	458.0635	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4i



BRUKER

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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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Time                  13.18
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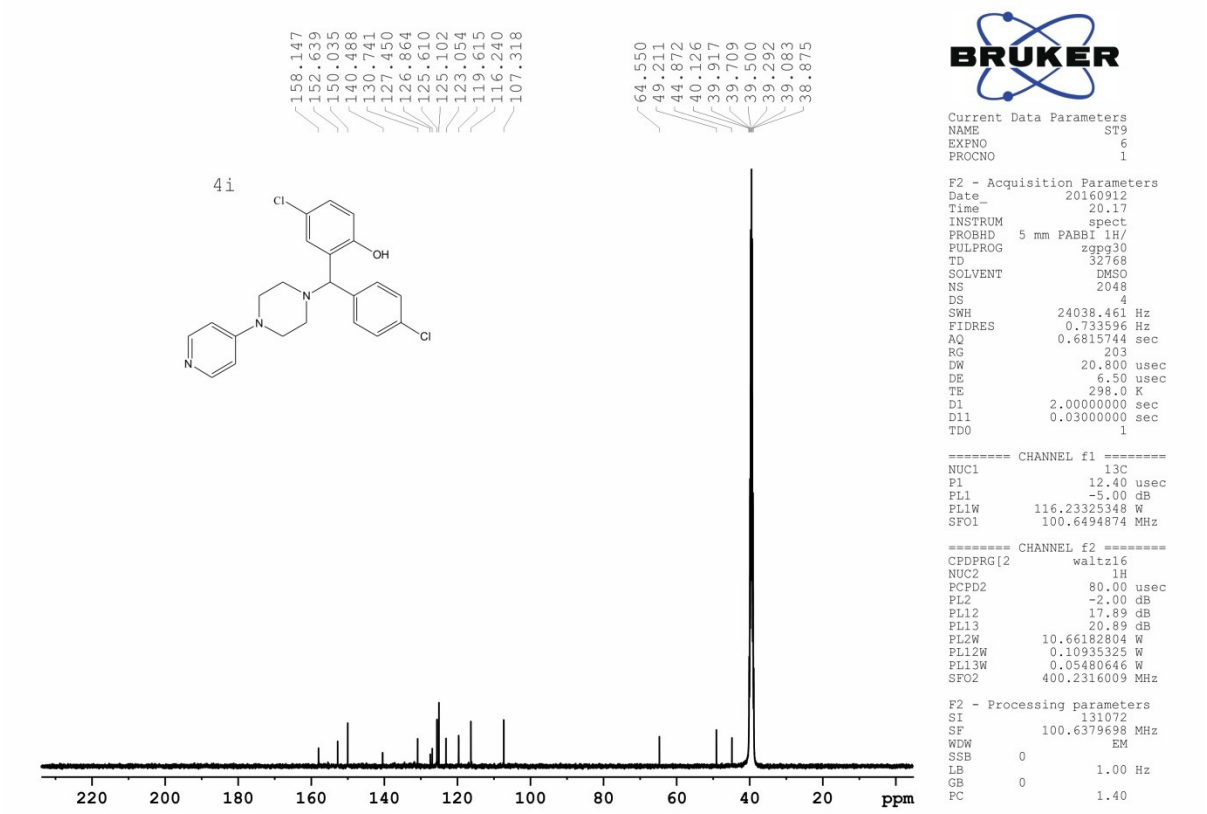
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                   128202
SOLVENT                DMSO
NS                       24
DS                        2
SWH              16025.641 Hz
FIDRES            0.125003 Hz
AQ               3.9999025 sec
RG                      181
DW                   31.200 usec
DE                    6.00 usec
TE                   298.2 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.25 usec
PL1         0 dB
SFO1          400.1300000 MHz

F2 - Processing parameters
SI                       32768
SF            400.1300027 MHz
WDW                        EM
SSB         0
LB                    0.30 Hz
GB          0
PC                    1.00
```

^{13}C NMR spectrum of 4i



HRMS of 4i

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

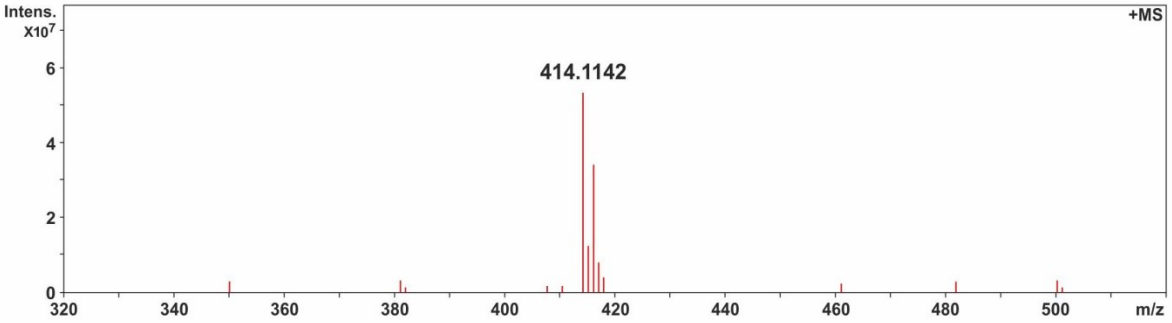
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-2

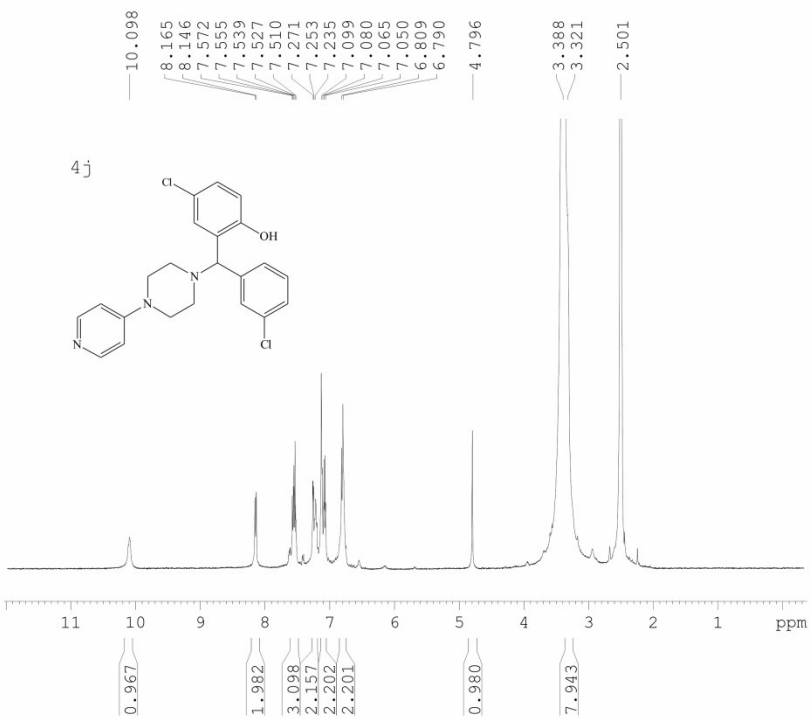
4i 40 (1.108)

TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
414.1142	1	C 22 H 22 Cl 2 N 3 O	100.00	414.1140	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4j



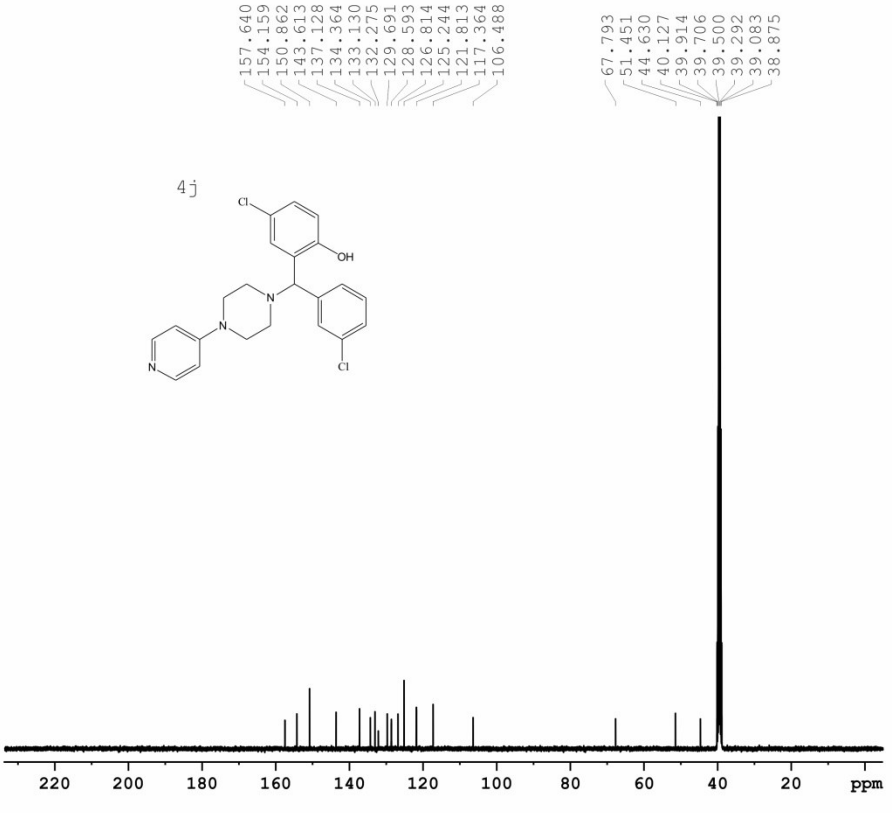

```
Current Data Parameters
NAME                  sep9ss
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160908
Time                  13.51
INSTRUM               av400
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    96152
SOLVENT                DMSO
NS                       24
DS                        2
SWH              16025.641 Hz
FIDRES            0.166670 Hz
AQ                2.9999423 sec
RG                    161.3
DW                   31.200 usec
DE                    6.00 usec
TE                   296.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.25 usec
PL1          0 dB
SFO1           400.1300000 MHz

F2 - Processing parameters
SI                     32768
SF               400.1300025 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{13}C NMR spectrum of 4j




```
Current Data Parameters
NAME                    ST10
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160816
Time                  21.37
INSTRUM               spect
PROBHD    5 mm PABBI 1H/
PULPROG             zgpg30
TD                    32768
SOLVENT                DMSO
NS                     2048
DS                        4
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.40 usec
PL1                    -5.00 dB
PL1W         116.23325348 W
SFO1          100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                    -2.00 dB
PL12                   17.89 dB
PL13                   20.89 dB
PL2W          10.66182804 W
PL12W          0.10935325 W
PL13W          0.05480646 W
SFO2          400.2316009 MHz

F2 - Processing parameters
SI                    131072
SF           100.6379626 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

HRMS of 4j

Elemental Composition Report Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

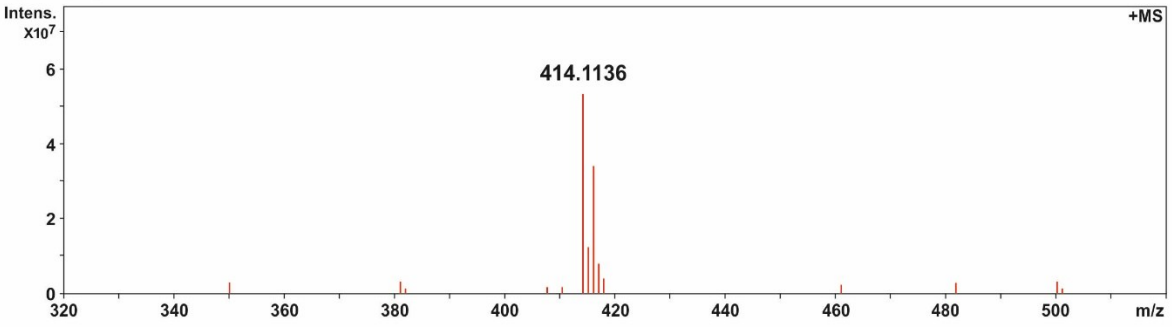
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-2

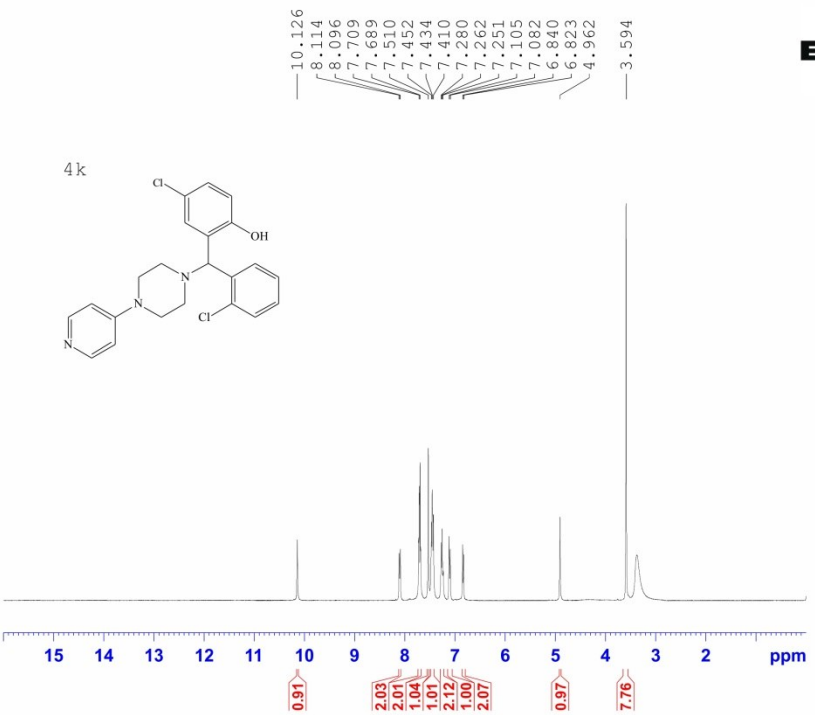
4j 49 (1.108)

TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e$_i$ Conf	N-Rule
414.1136	1	C 22 H 22 Cl 2 N 3 O	100.00	414.1140	0.5	1.8	8.7	12.0	even	ok

^{1}H NMR spectrum of 4k



BRUKER

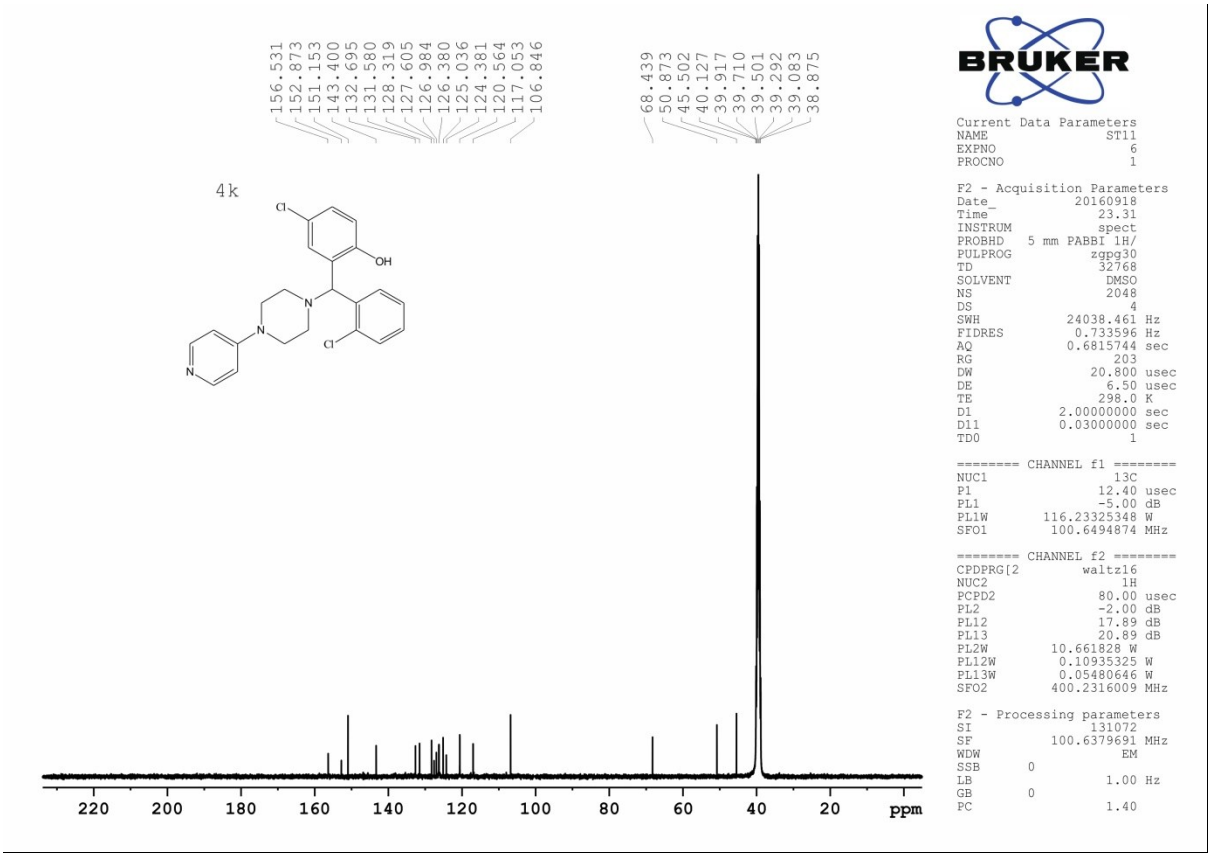
```
Current Data Parameters
NAME                  su26ss
EXPNO                     12
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160811
Time                  11.43
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    60521
SOLVENT                DMSO
NS                        16
DS                         2
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                      49.7
DW                   62.400 usec
DE                    6.50 usec
TE                   295.6 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.2324716 MHz
NUC1                       1H
P1                    14.55 usec
PLW1            11.00000000 W

F2 - Processing parameters
SI                        65536
SF               400.2300000 MHz
WDW                         EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{13}C NMR spectrum of 4k

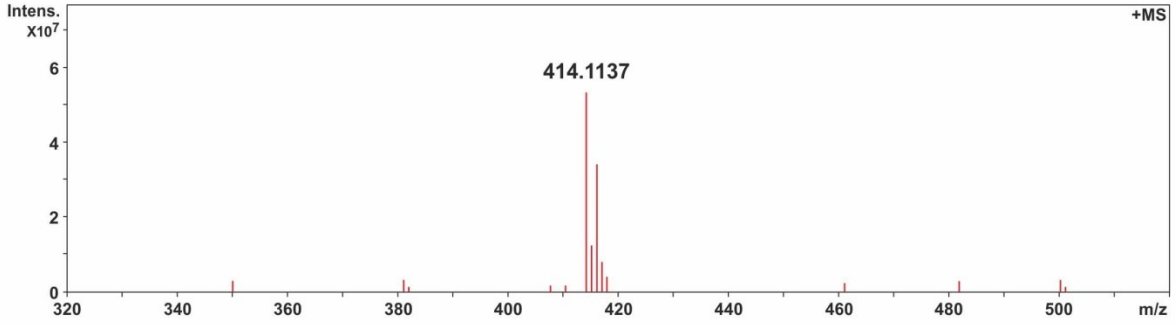


HRMS of 4k

Elemental Composition Report **Page 1**

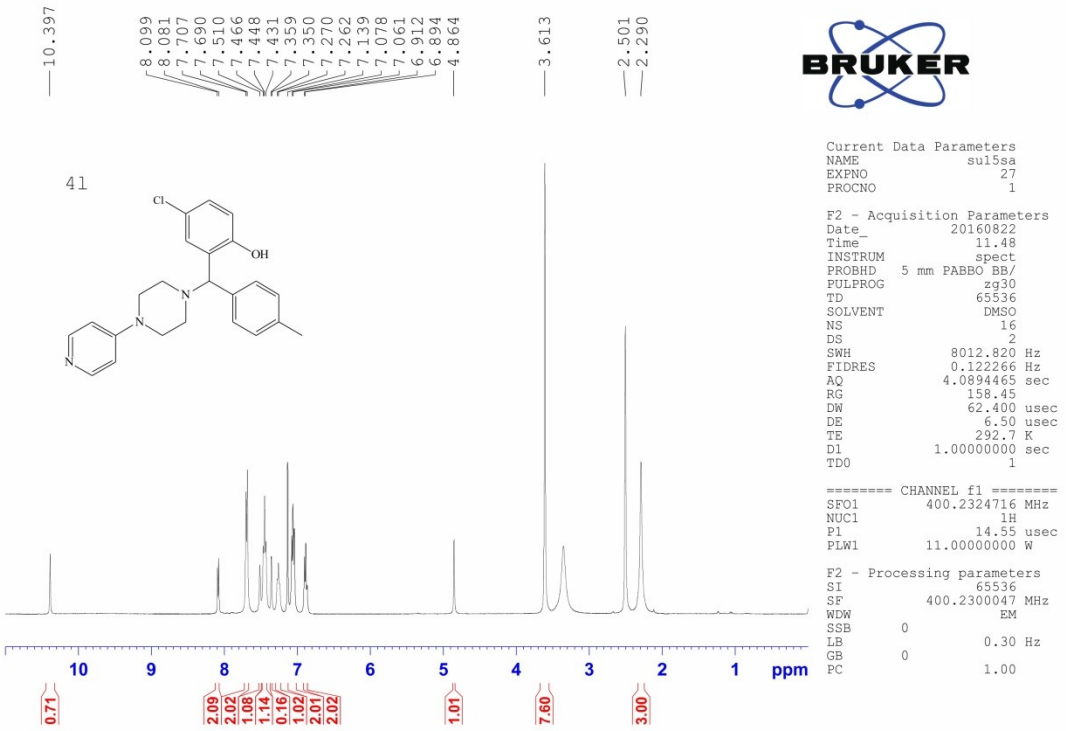
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-2
4k 43 (1.108)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
414.1137	1	C 22 H 22 Cl 2 N 3 O	100.00	414.1140	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4l



BRUKER

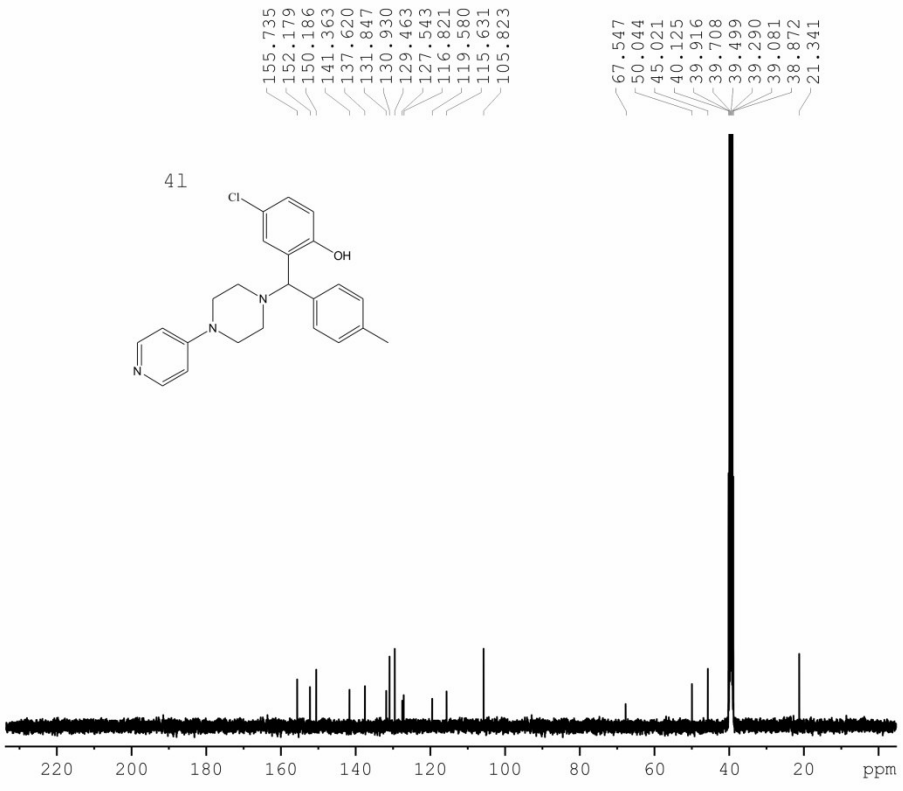
```
Current Data Parameters
NAME                  su15sa
EXPNO                     27
PROCNO                     1

F2 - Acquisition Parameters
Date_               20160822
Time                   11.48
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                        16
DS                         2
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894465 sec
RG                    158.45
DW                    62.400 usec
DE                     6.50 usec
TE                    292.7 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.2324716 MHz
NUC1                       1H
P1                      14.55 usec
PLW1             11.00000000 W

F2 - Processing parameters
SI                        65536
SF               400.2300047 MHz
WDW                        EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

^{13}C NMR spectrum of 4l




```
Current Data Parameters
NAME                  08_SUM
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20160918
Time                   22.10
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg
TD                    32768
SOLVENT                DMSO
NS                      2048
DS                         4
SWH               24038.461 Hz
FIDRES                0.733596 Hz
AQ                 0.6815744 sec
RG                       203
DW                    20.800 usec
DE                     6.50 usec
TE                    298.0 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.50 usec
PL1                    -2.00 dB
PL1W             58.25462341 W
SFO1            100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   13.48 dB
PL13                   14.00 dB
PL2W             13.42244530 W
PL12W             0.30187815 W
PL13W             0.26781300 W
SFO2            400.2316009 MHz

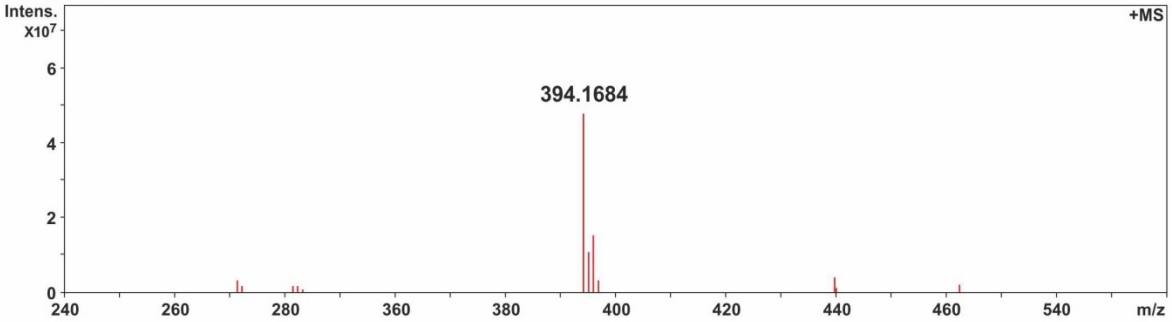
F2 - Processing parameters
SI                       131072
SF               100.6379617 MHz
WDW                        EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

HRMS of 4l

Elemental Composition Report **Page 1**

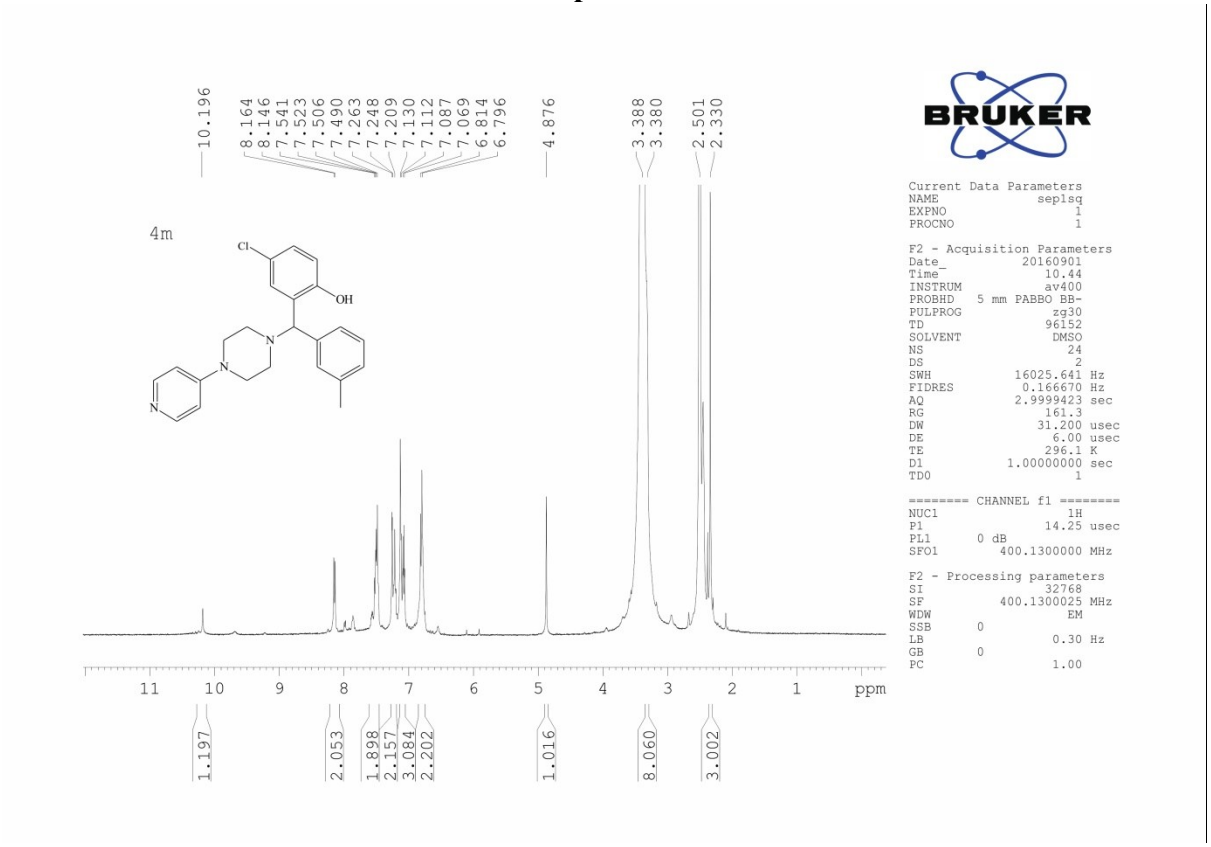
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-1
4l 27 (0.315)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡ Conf	N-Rule
394.1684	1	C 23 H 25 Cl N 3 O	100.00	394.1686	0.5	1.7	8.8	12.1	even	ok

^{1}H NMR spectrum of 4m



BRUKER

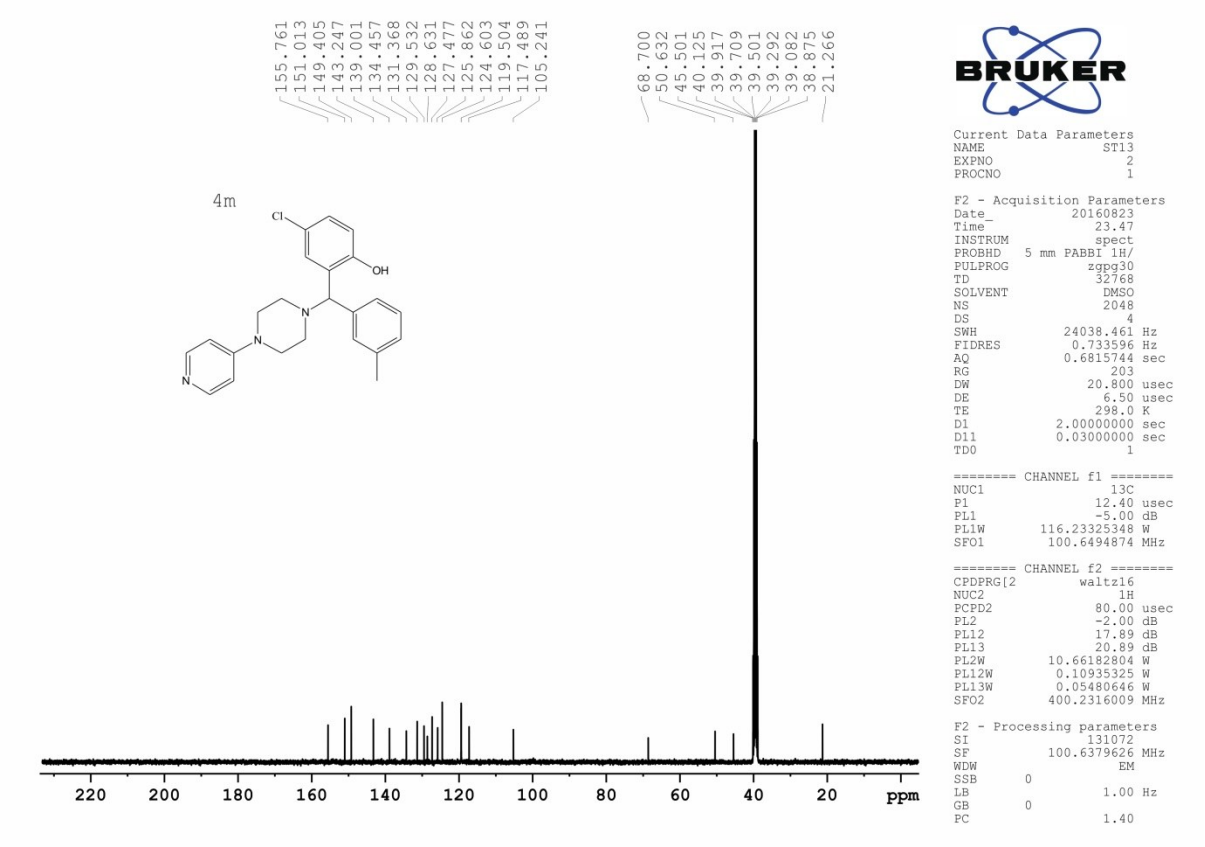
```
Current Data Parameters
NAME                  seplsq
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160901
Time                 10.44
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  96152
SOLVENT               DMSO
NS                      24
DS                       2
SWH              16025.641 Hz
FIDRES            0.166670 Hz
AQ                2.9999423 sec
RG                   161.3
DW                  31.200 usec
DE                   6.00 usec
TE                  296.1 K
D1               1.00000000 sec
TD0                        1

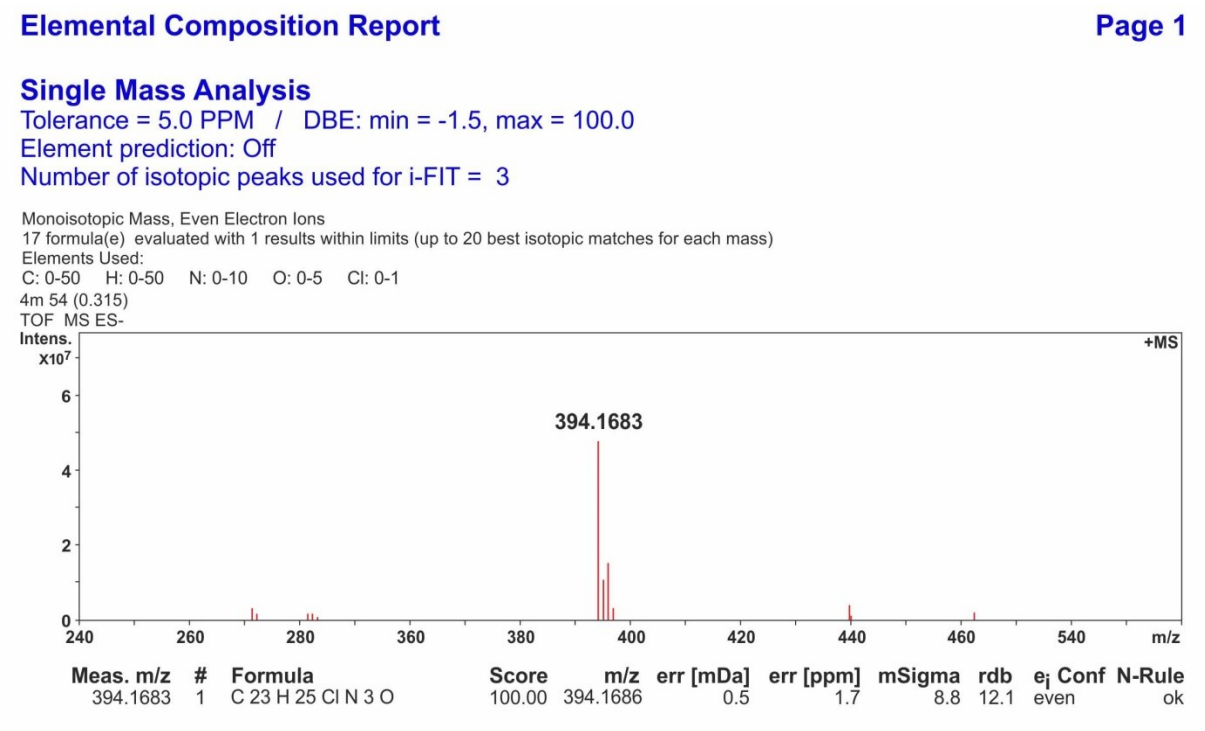
======== CHANNEL f1 ========
NUC1                      1H
P1                    14.25 usec
PL1          0 dB
SFO1          400.1300000 MHz

F2 - Processing parameters
SI                     32768
SF              400.1300025 MHz
WDW                       EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

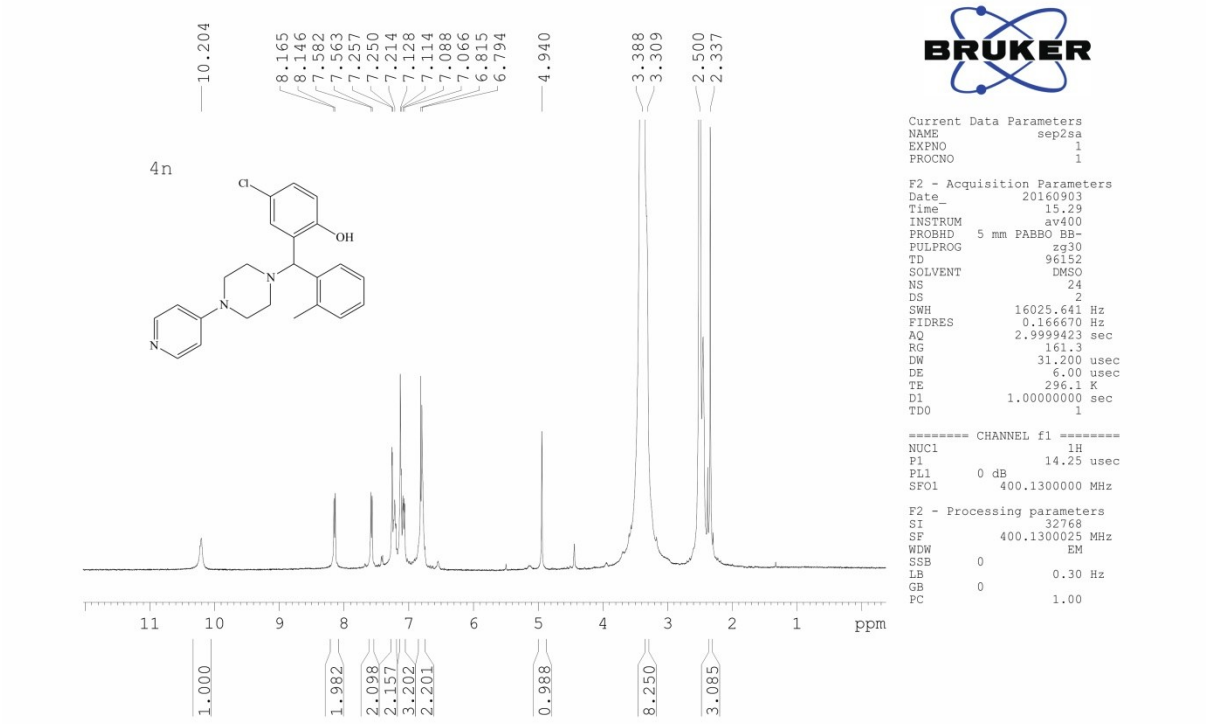
^{13}C NMR spectrum of 4m



HRMS of 4m



^{1}H NMR spectrum of 4n



BRUKER

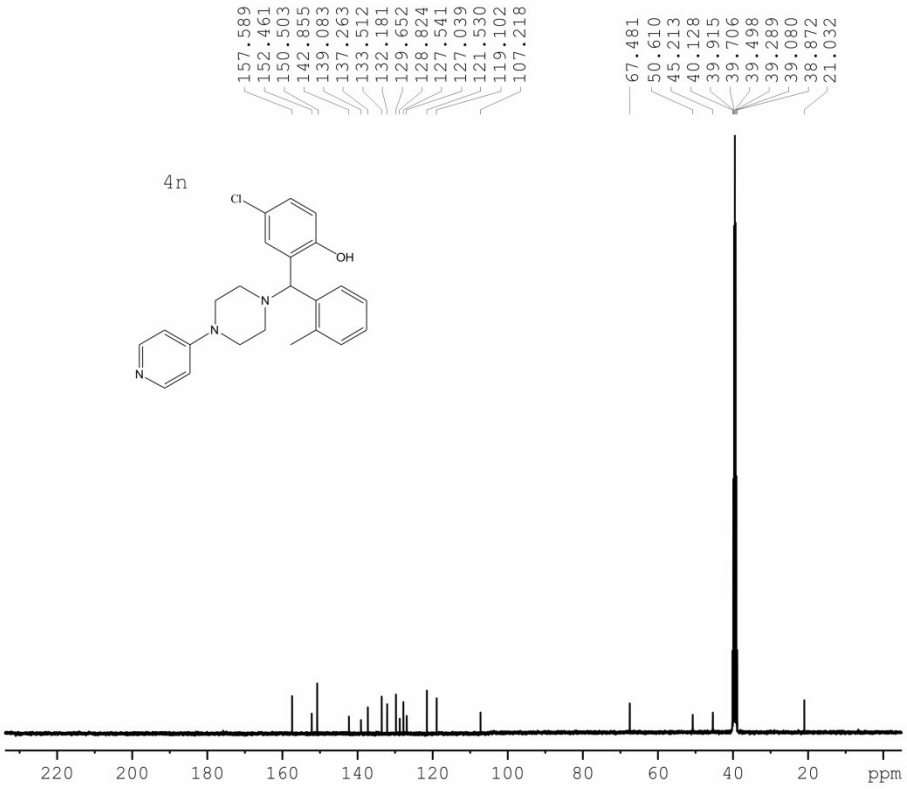
```
Current Data Parameters
NAME                  sep2sa
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160903
Time                  15.29
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    96152
SOLVENT                DMSO
NS                       24
DS                        2
SWH                16025.641 Hz
FIDRES              0.166670 Hz
AQ                  2.9999423 sec
RG                      161.3
DW                     31.200 usec
DE                      6.00 usec
TE                     296.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.25 usec
PL1          0 dB
SFO1            400.1300000 MHz

F2 - Processing parameters
SI                     32768
SF                400.1300025 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{13}C NMR spectrum of 4n




```
Current Data Parameters
NAME                  07_Sum
EXPNO                      4
PROCNO                     1

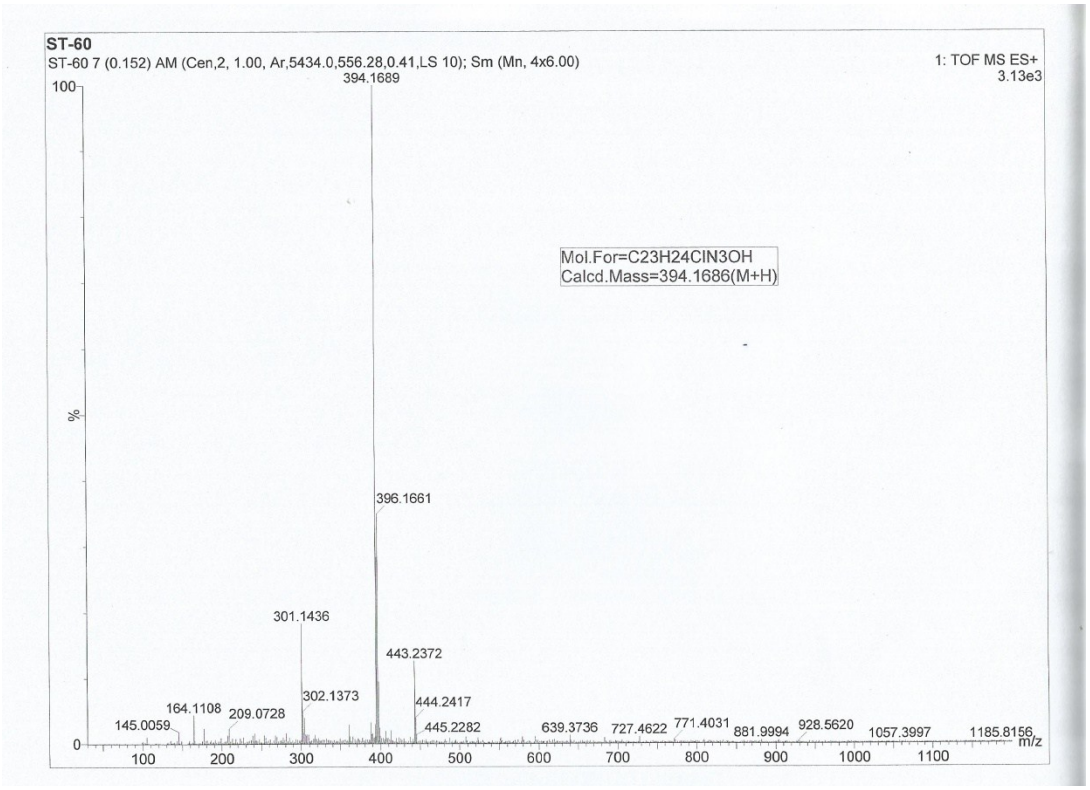
F2 - Acquisition Parameters
Date_              20160801
Time                  16.45
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zgpg
TD                    32768
SOLVENT                DMSO
NS                     1626
DS                        4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  0.6815744 sec
RG                      203
DW                     20.800 usec
DE                      6.50 usec
TE                     298.0 K
D1                3.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                      9.50 usec
PL1                    -2.00 dB
PL1W              58.25462341 W
SFO1              100.6494874 MHz

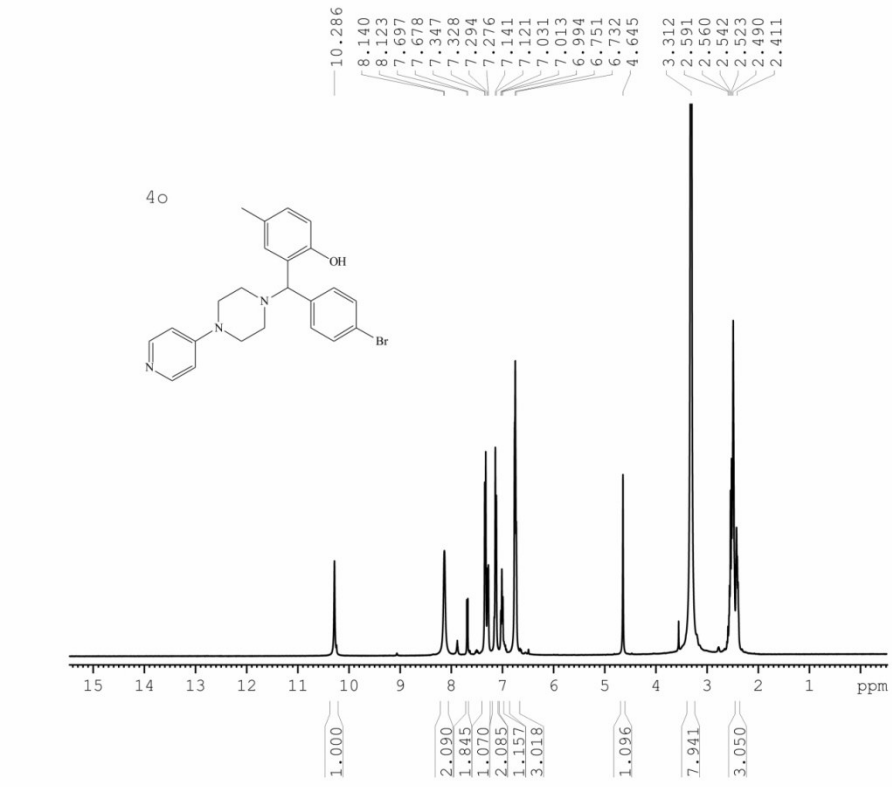
======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   13.48 dB
PL13                   14.00 dB
PL2W              13.42244530 W
PL12W              0.30187815 W
PL13W              0.26781300 W
SFO2              400.2316009 MHz

F2 - Processing parameters
SI                    131072
SF                100.6379666 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

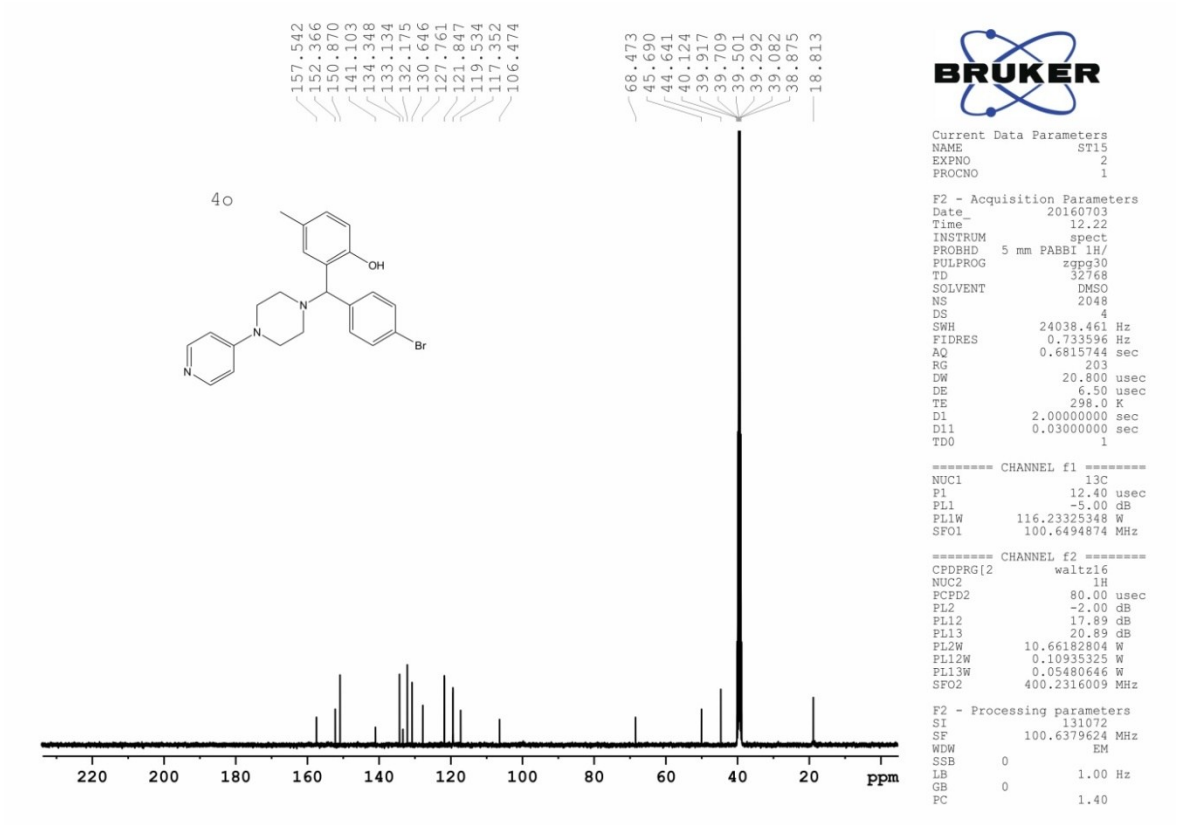
HRMS of 4n



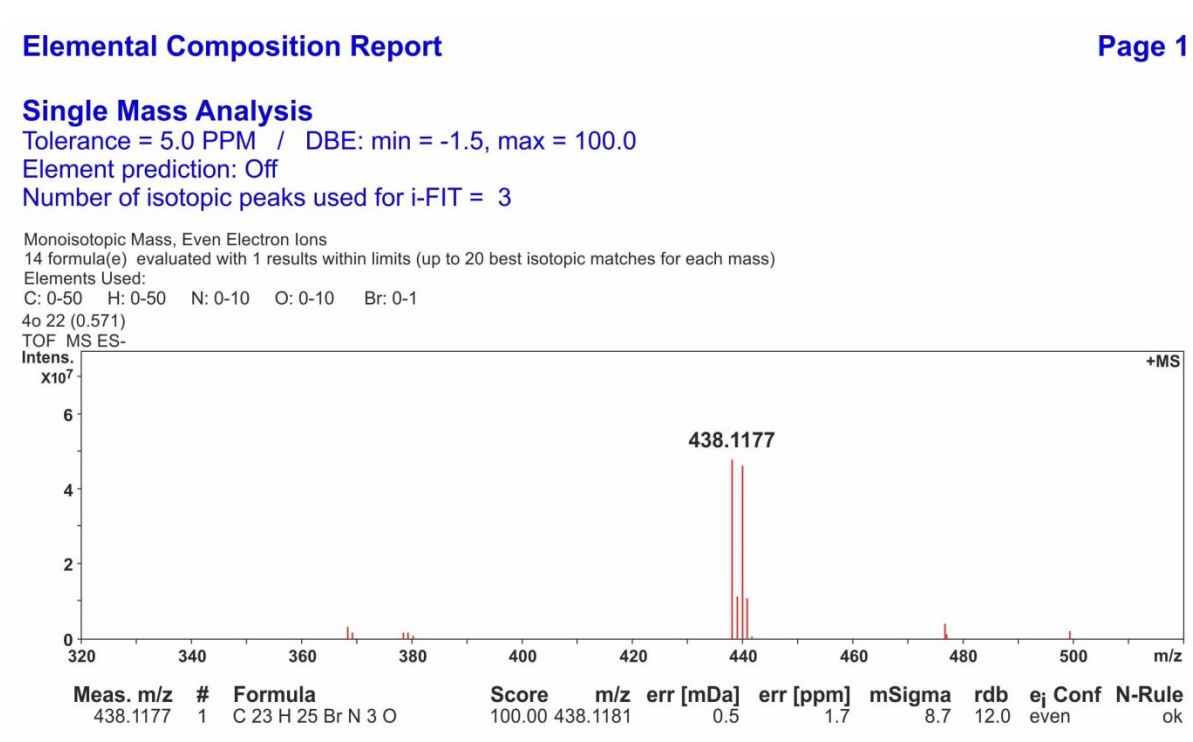
^{1}H NMR spectrum of 4o



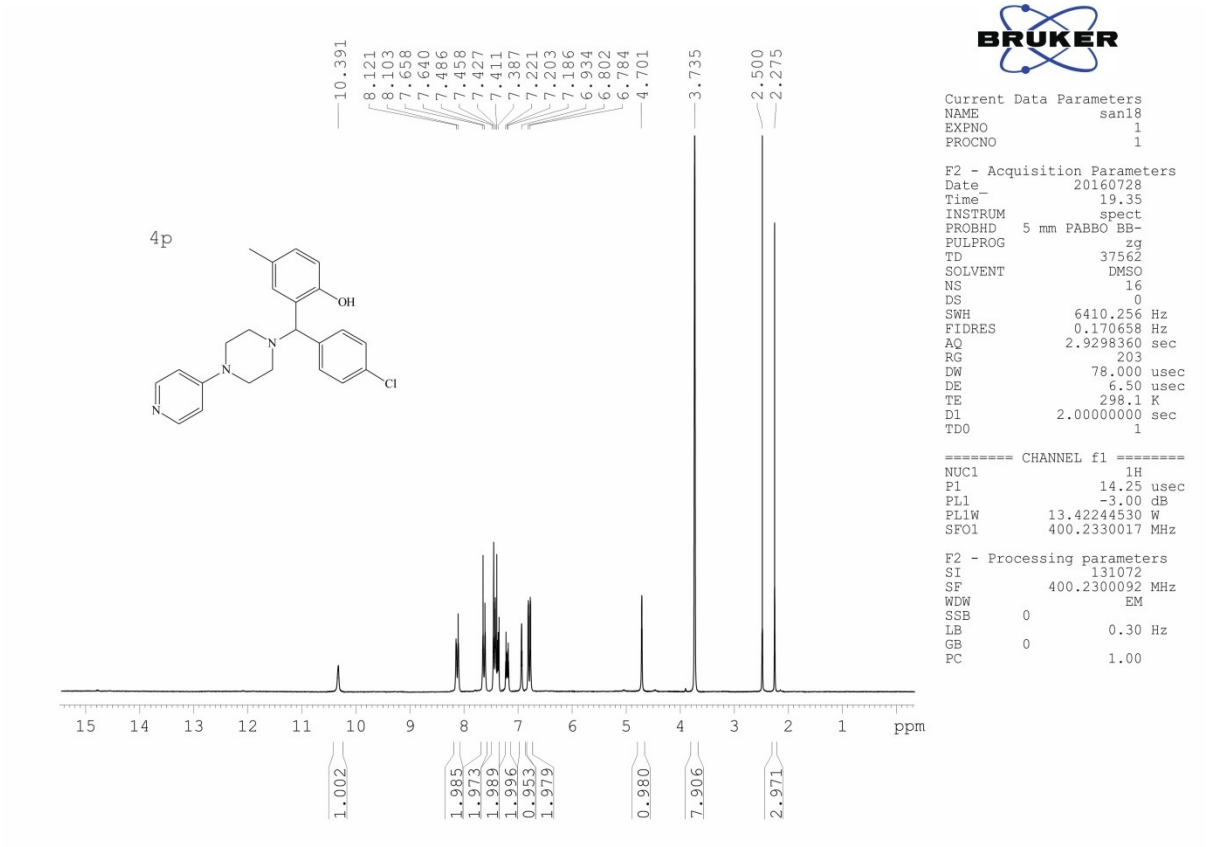
^{13}C NMR spectrum of 4o



HRMS of 4o



^{1}H NMR spectrum of 4p



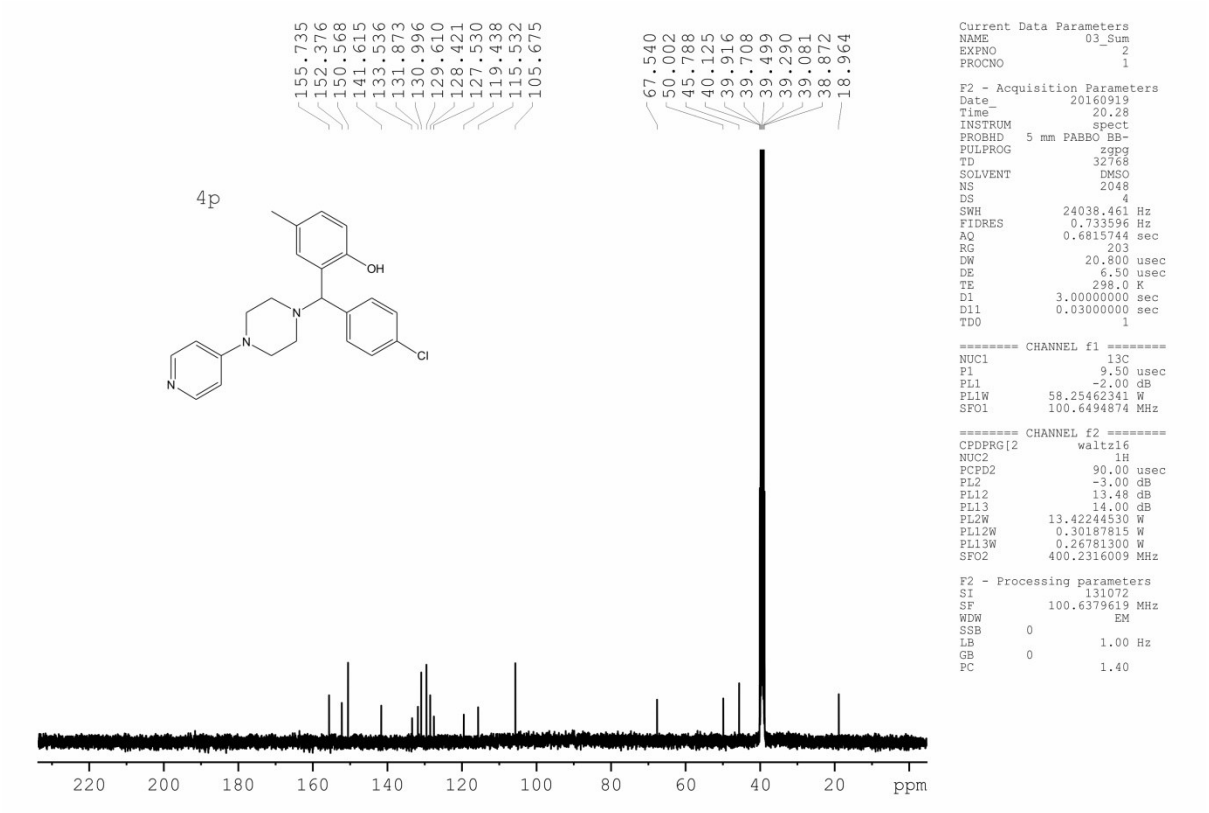

```
Current Data Parameters
NAME                  san18
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20160728
Time                  19.35
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                   37562
SOLVENT               DMSO
NS                      16
DS                       0
SWH                6410.256 Hz
FIDRES            0.170658 Hz
AQ                2.9298360 sec
RG                     203
DW                  78.000 usec
DE                   6.50 usec
TE                  298.1 K
D1              2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.25 usec
PL1                  -3.00 dB
PL1W          13.42244530 W
SFO1          400.2330017 MHz

F2 - Processing parameters
SI                   131072
SF            400.2300092 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of 4p




```
Current Data Parameters
NAME                  03_Sum
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160919
Time                  20.28
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                   32768
SOLVENT               DMSO
NS                    2048
DS                       4
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                     203
DW                  20.800 usec
DE                   6.50 usec
TE                  298.0 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.50 usec
PL1                  -2.00 dB
PL1W          58.25462341 W
SFO1          100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 13.48 dB
PL13                 14.00 dB
PL2W          13.42244530 W
PL12W          0.30187815 W
PL13W          0.26781300 W
SFO2          400.2316009 MHz

F2 - Processing parameters
SI                   131072
SF            100.6379619 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

HRMS of 4p

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

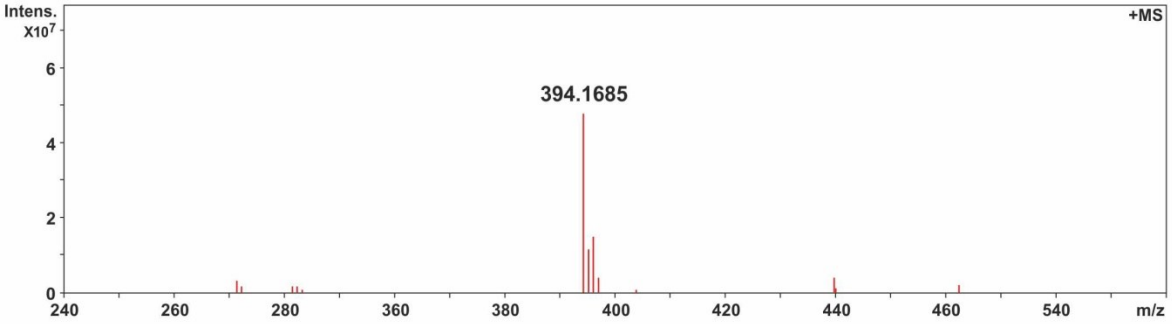
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-1

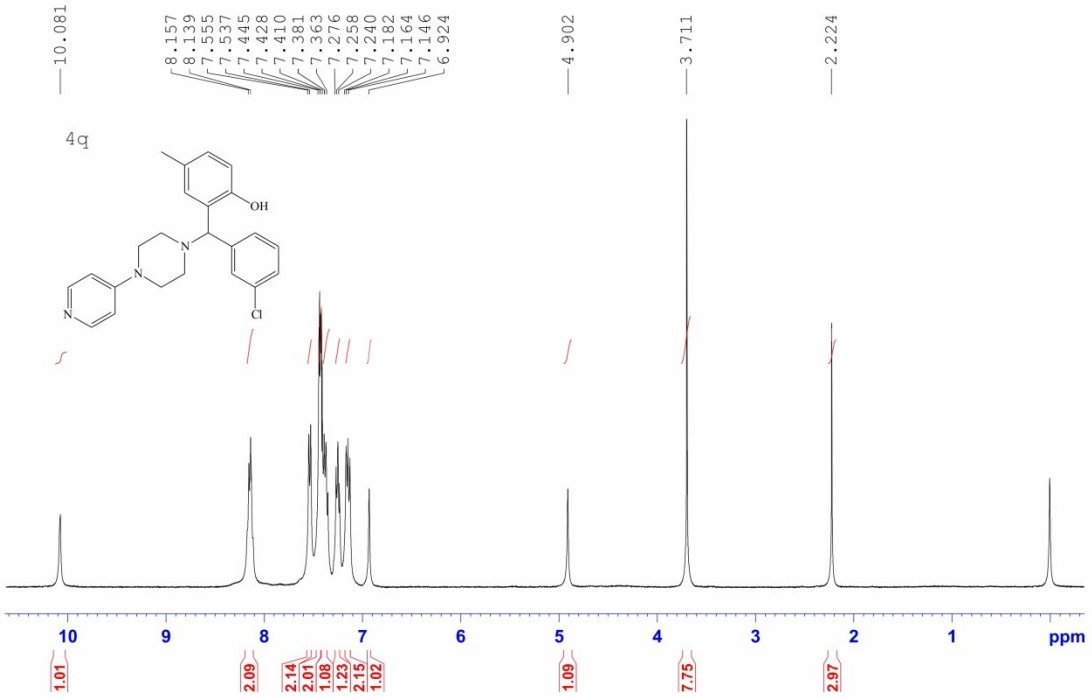
4p 50 (0.621)

TOF MS ES-

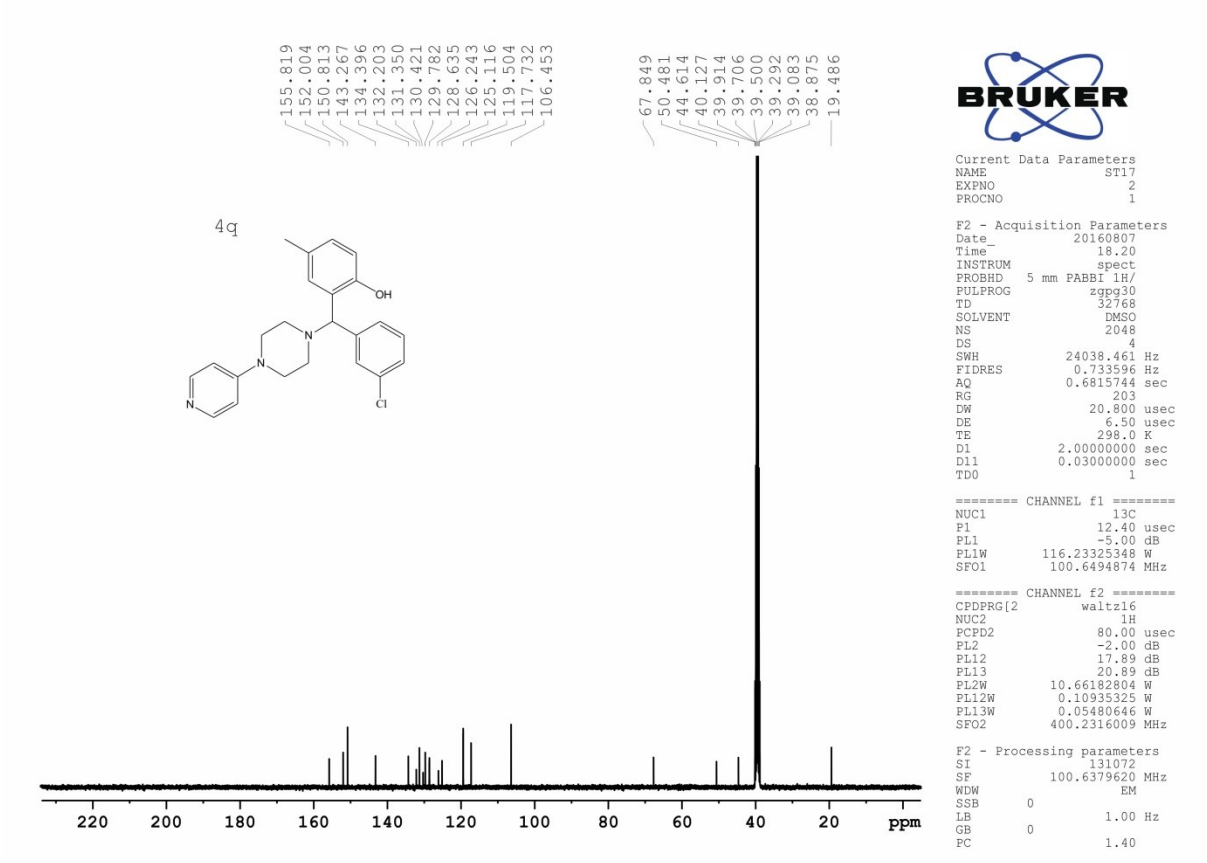


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
394.1685	1	C 23 H 25 Cl N 3 O	100.00	394.1686	0.5	1.7	8.8	12.1	even	ok

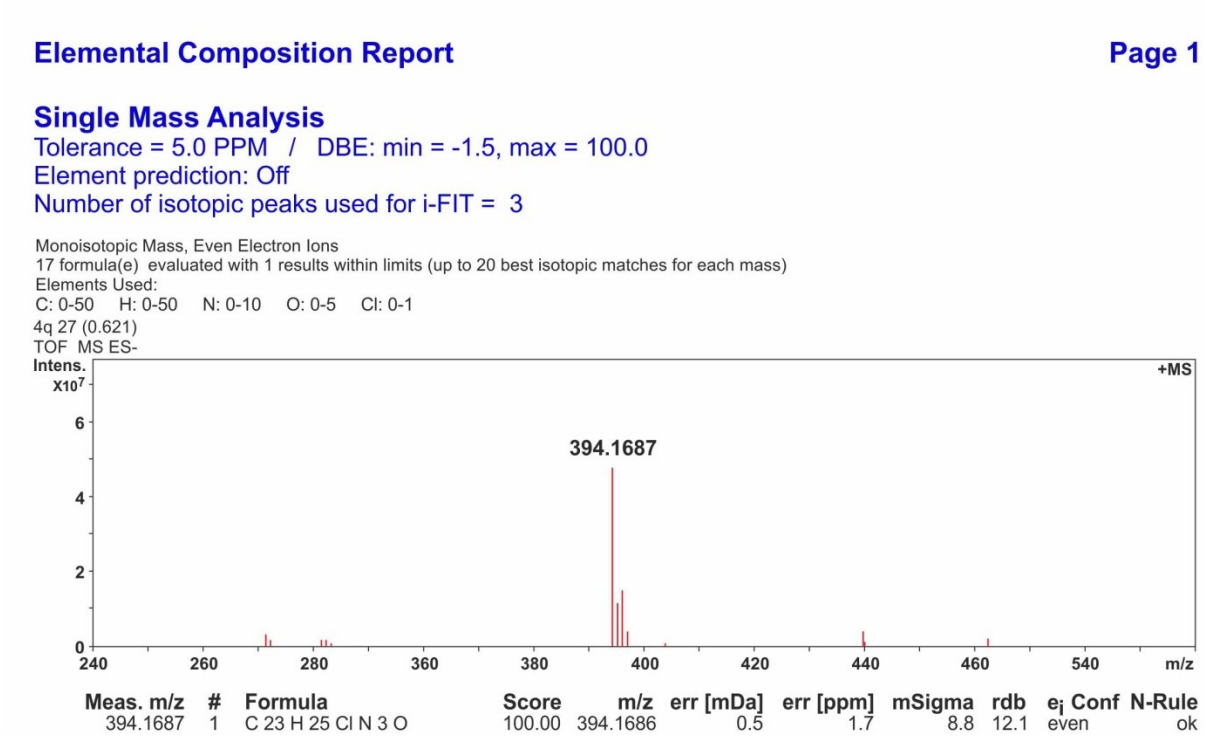
^{1}H NMR spectrum of 4q



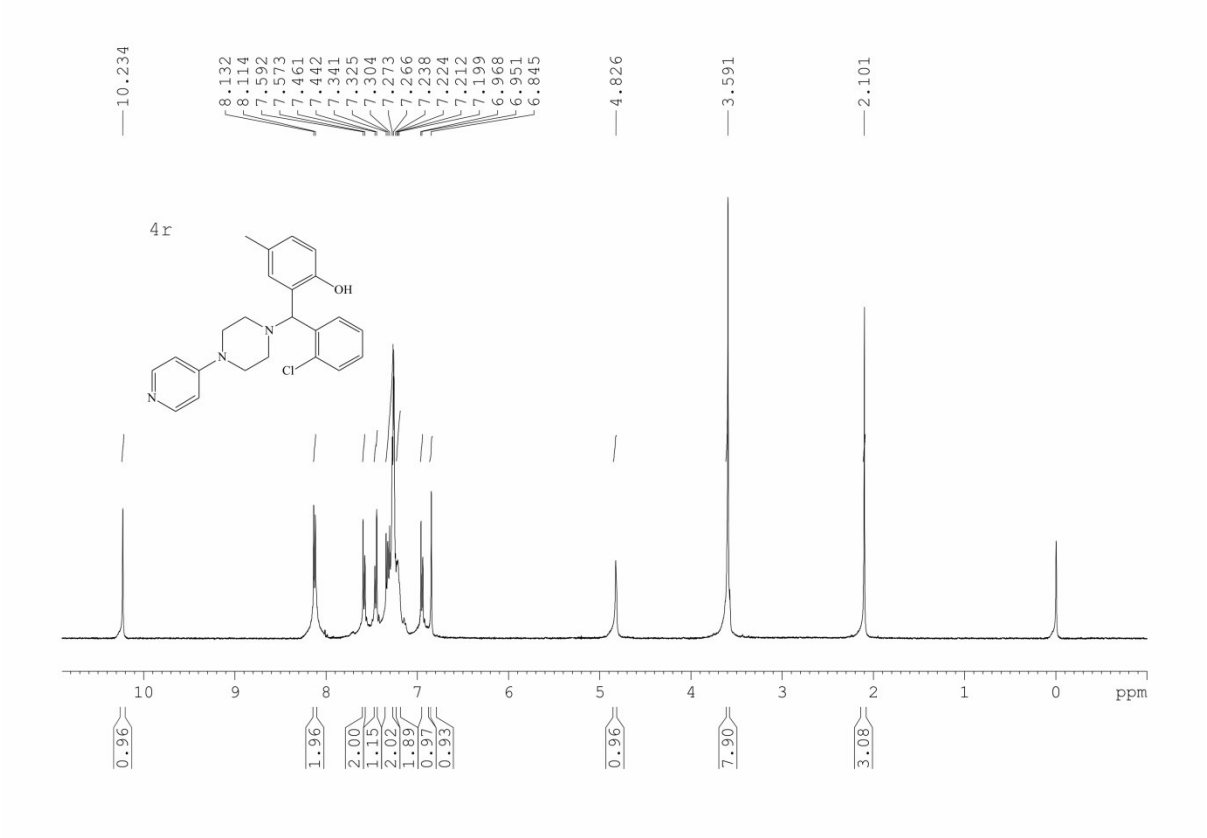
^{13}C NMR spectrum of 4q



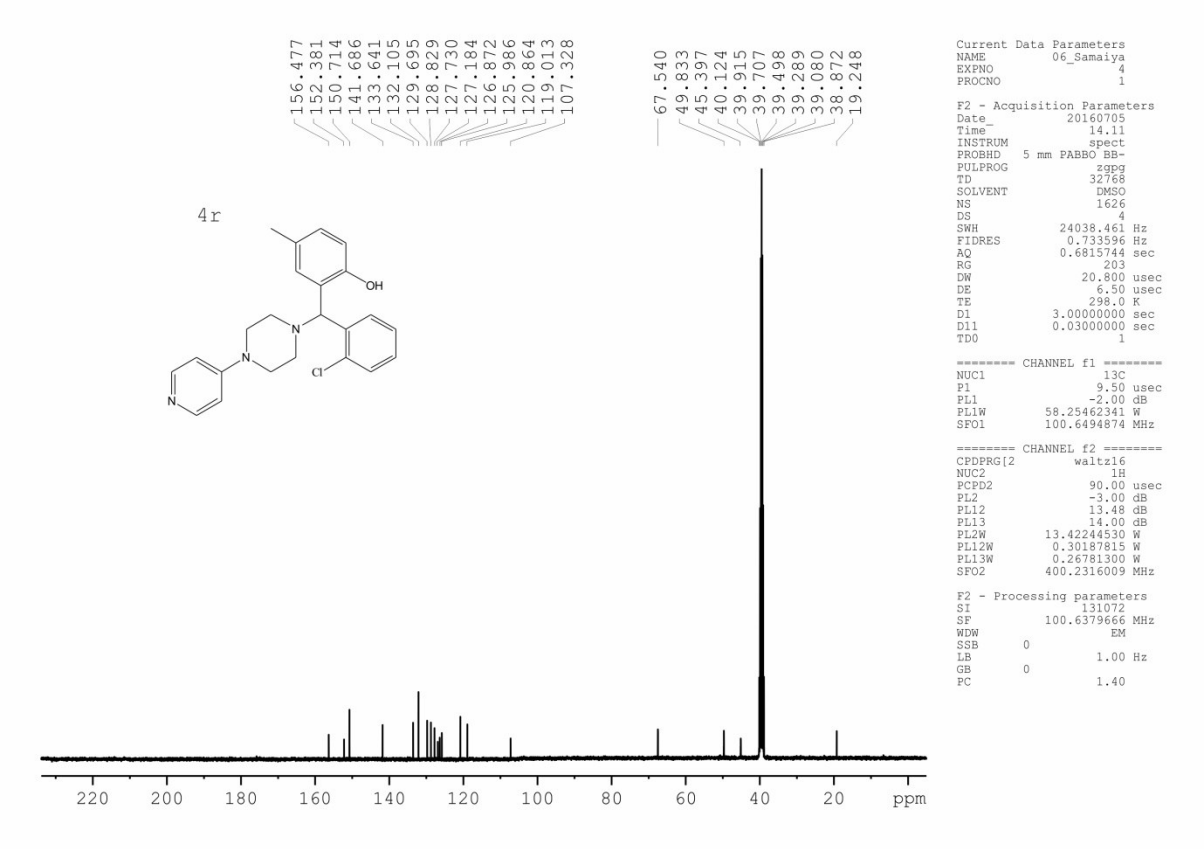
HRMS of 4q



^{1}H NMR spectrum of 4r



^{13}C NMR spectrum of 4r



HRMS of 4r

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

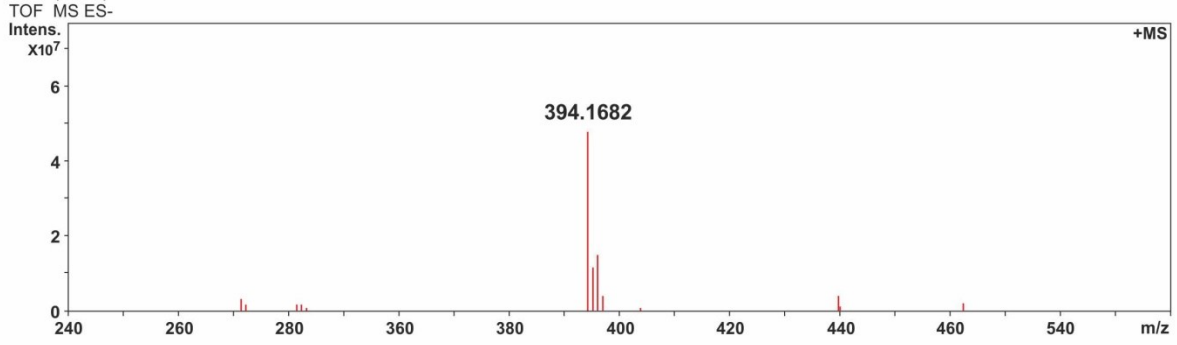
17 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-10 O: 0-5 Cl: 0-1

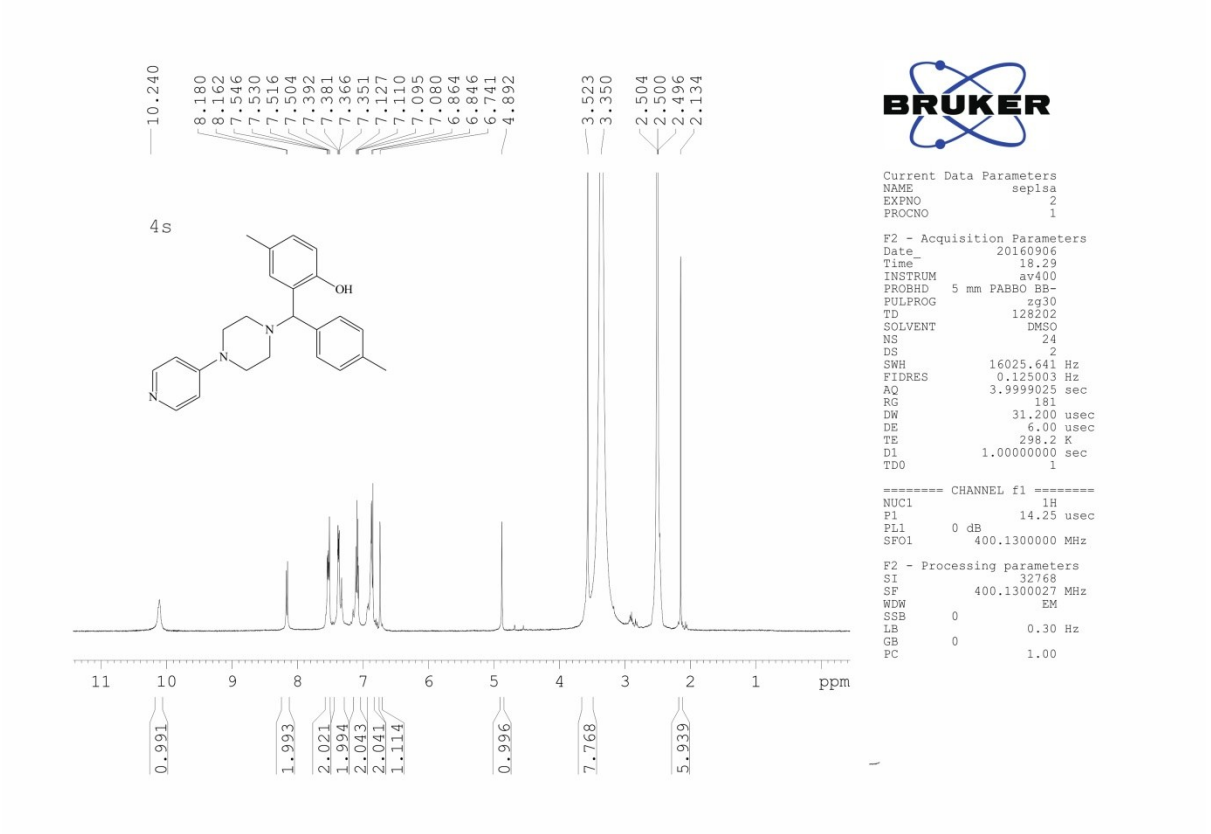
4r 22 (0.621)

TOF MS ES-

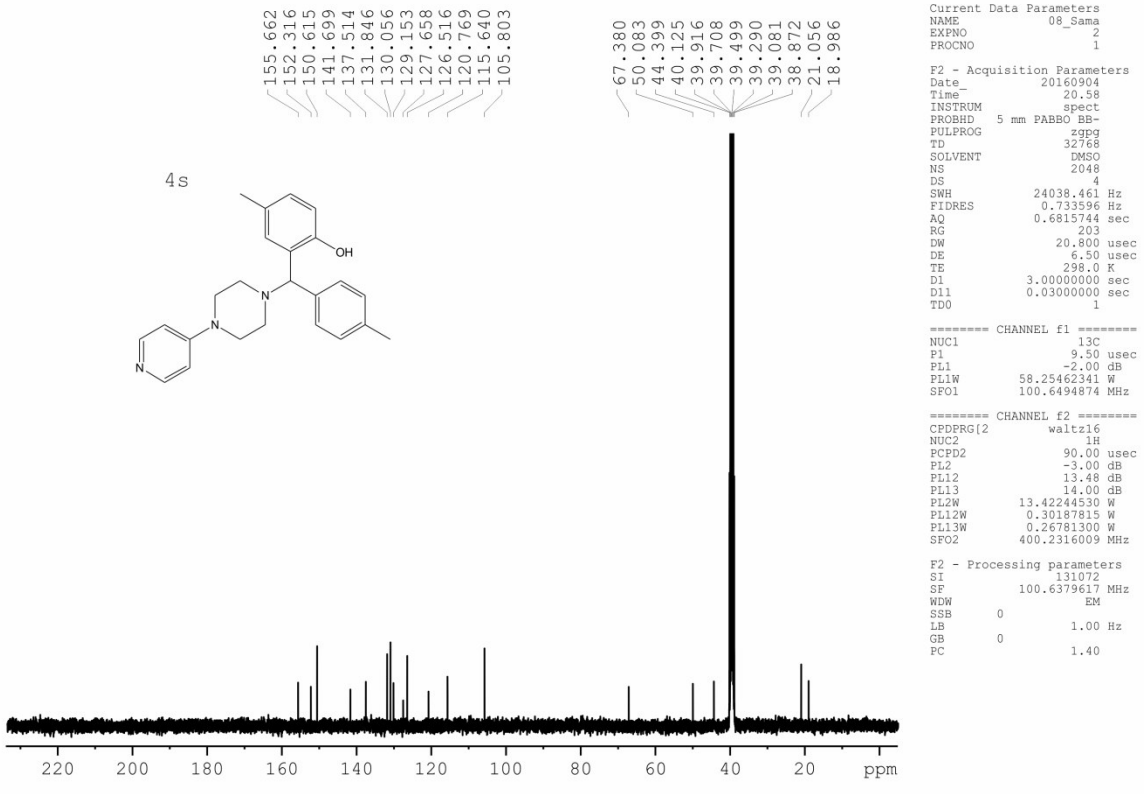


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
394.1682	1	C 23 H 25 Cl N 3 O	100.00	394.1686	0.5	1.7	8.8	12.1	even	ok

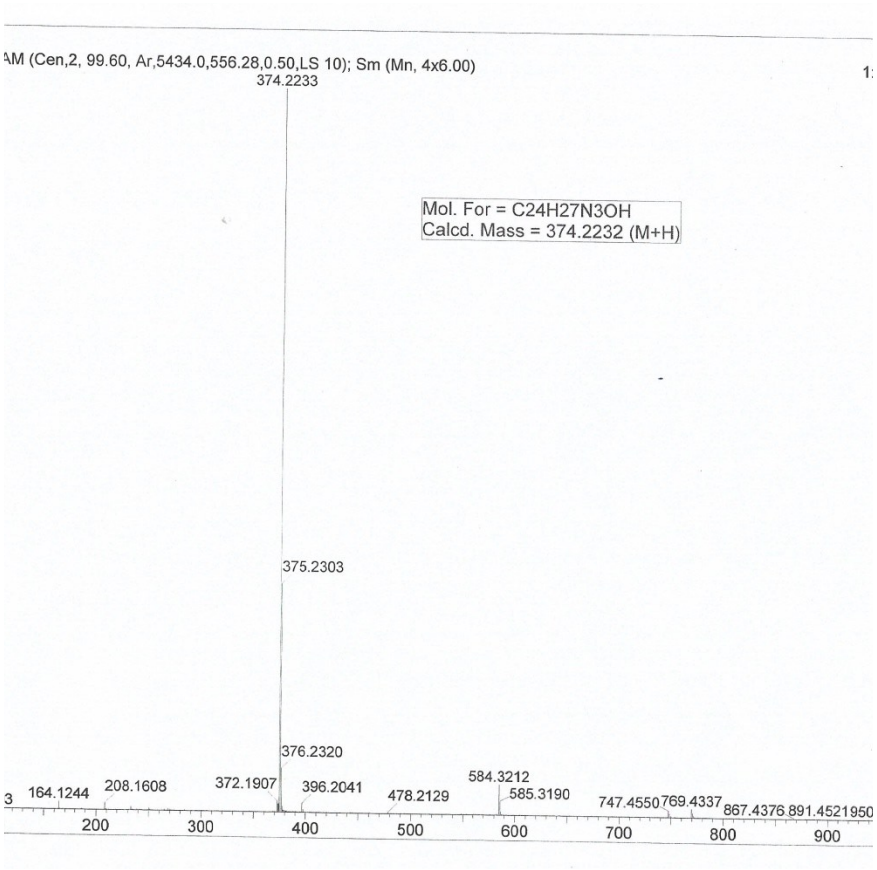
^{1}H NMR spectrum of 4s



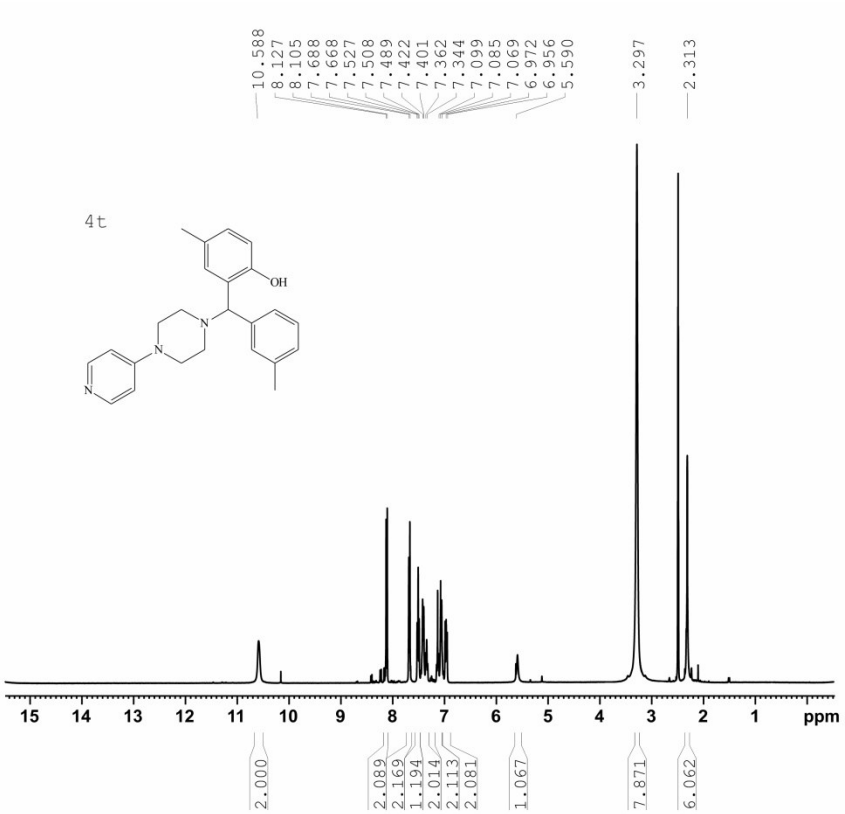
^{13}C NMR spectrum of 4s



HRMS of 4s



^{1}H NMR spectrum of 4t



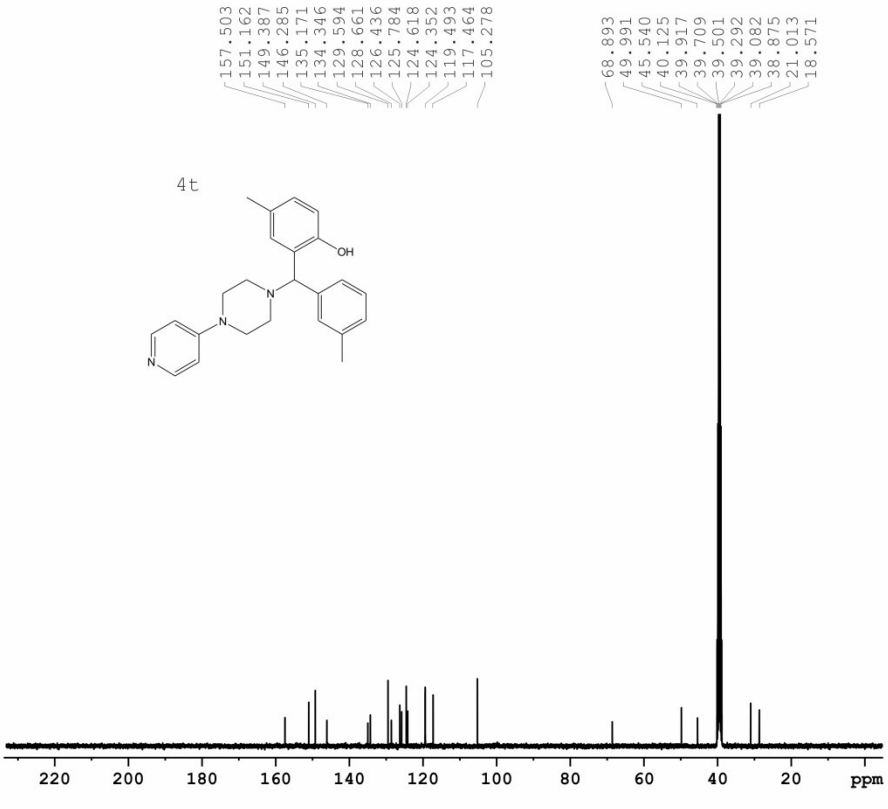

```
Current Data Parameters
NAME          27_Sumaiya
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160728
Time              13.30
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                    32
DS                     0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559039 sec
RG                   203
DW              78.000 usec
DE               6.50 usec
TE              298.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.17 usec
PL1               -3.00 dB
PL1W        13.42244530 W
SFO1        400.2330017 MHz

F2 - Processing parameters
SI                131072
SF          400.2300110 MHz
WDW                   EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

^{13}C NMR spectrum of 4t

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Current Data Parameters
NAME                 ST20
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160918
Time              23.18
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                32768
SOLVENT            DMSO
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                  203
DW              20.800 usec
DE               6.50 usec
TE              298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.40 usec
PL1               -5.00 dB
PL1W       116.23325348 W
SFO1        100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              17.89 dB
PL13              20.89 dB
PL2W        10.66182804 W
PL12W        0.10935325 W
PL13W        0.05480646 W
SFO2        400.2316009 MHz

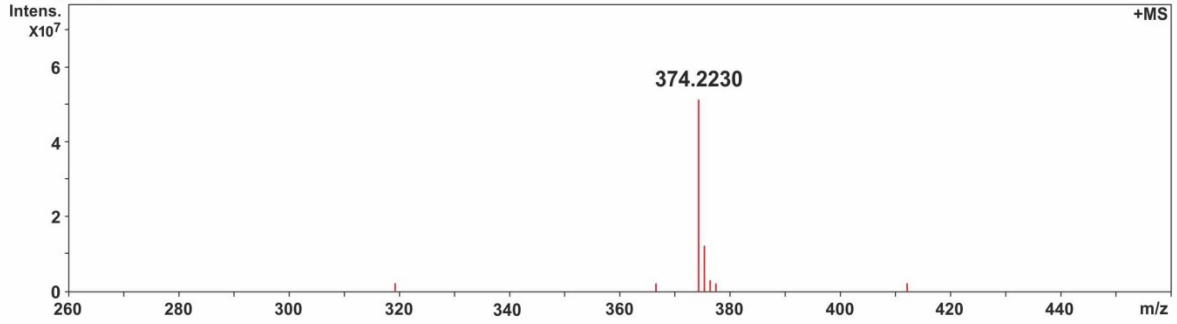
F2 - Processing parameters
SI                131072
SF          100.6379629 MHz
WDW                   EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

HRMS of 4t

Elemental Composition Report Page 1

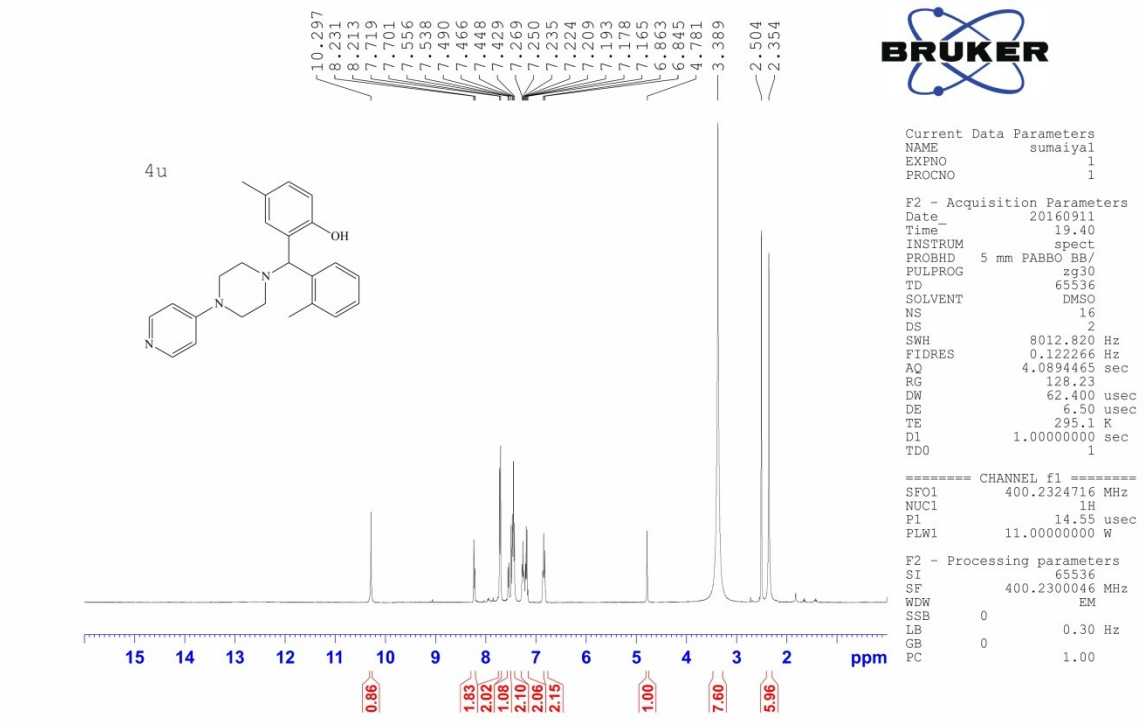
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5
4t 31 (1.037)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
374.2230	1	C 24 H 28 N 3 O	100.00	374.2232	0.5	1.7	8.7	12.0	even	ok

^{1}H NMR spectrum of 4u



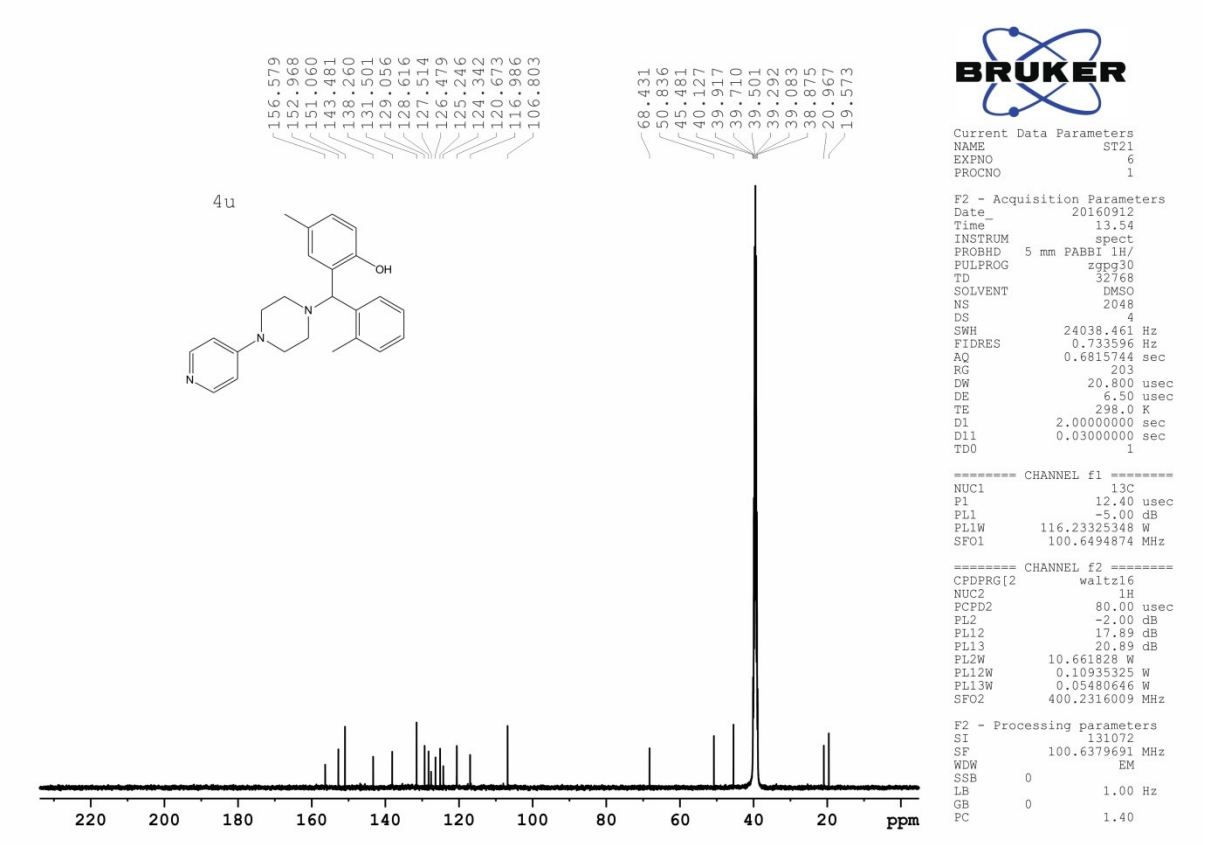

```
Current Data Parameters
NAME                  sumaiyal
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20160911
Time                     19.40
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                      128.23
DW                      62.400 usec
DE                       6.50 usec
TE                      295.1 K
D1                   1.00000000 sec
TD0                          1

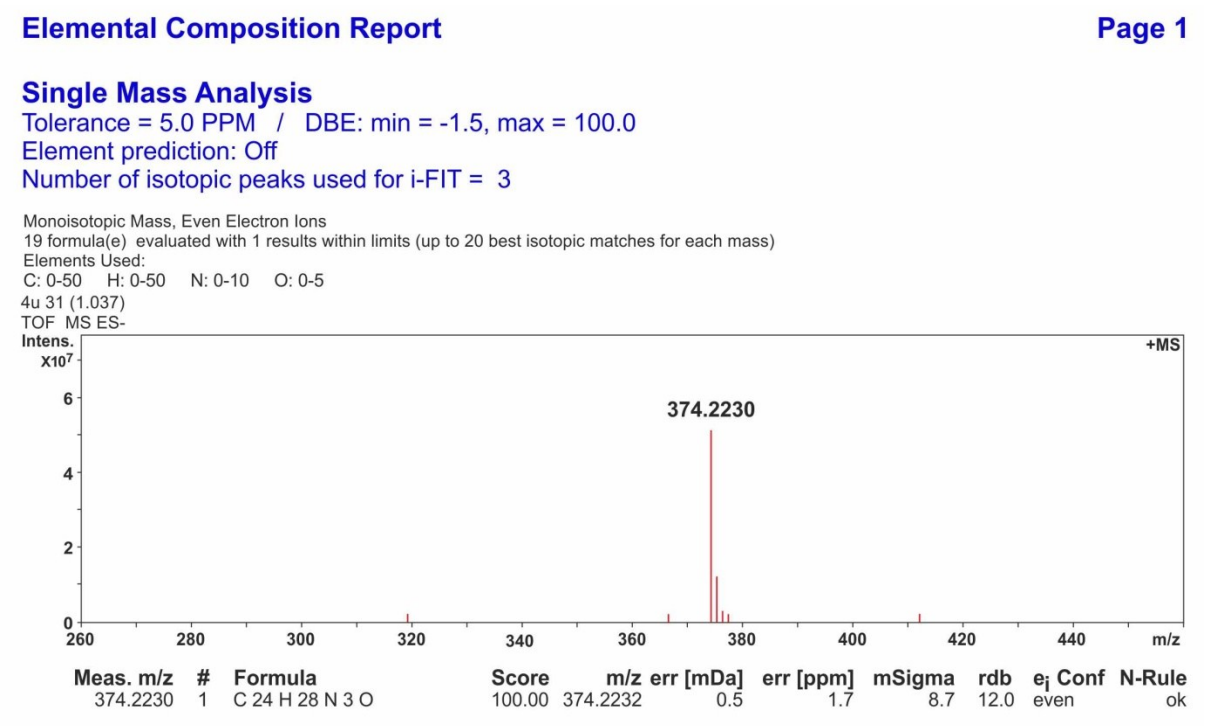
======== CHANNEL f1 ========
SFO1                400.2324716 MHz
NUC1                        1H
P1                       14.55 usec
PLW1                11.00000000 W

F2 - Processing parameters
SI                          65536
SF                  400.2300046 MHz
WDW                            EM
SSB                 0
LB                           0.30 Hz
GB                  0
PC                           1.00
```

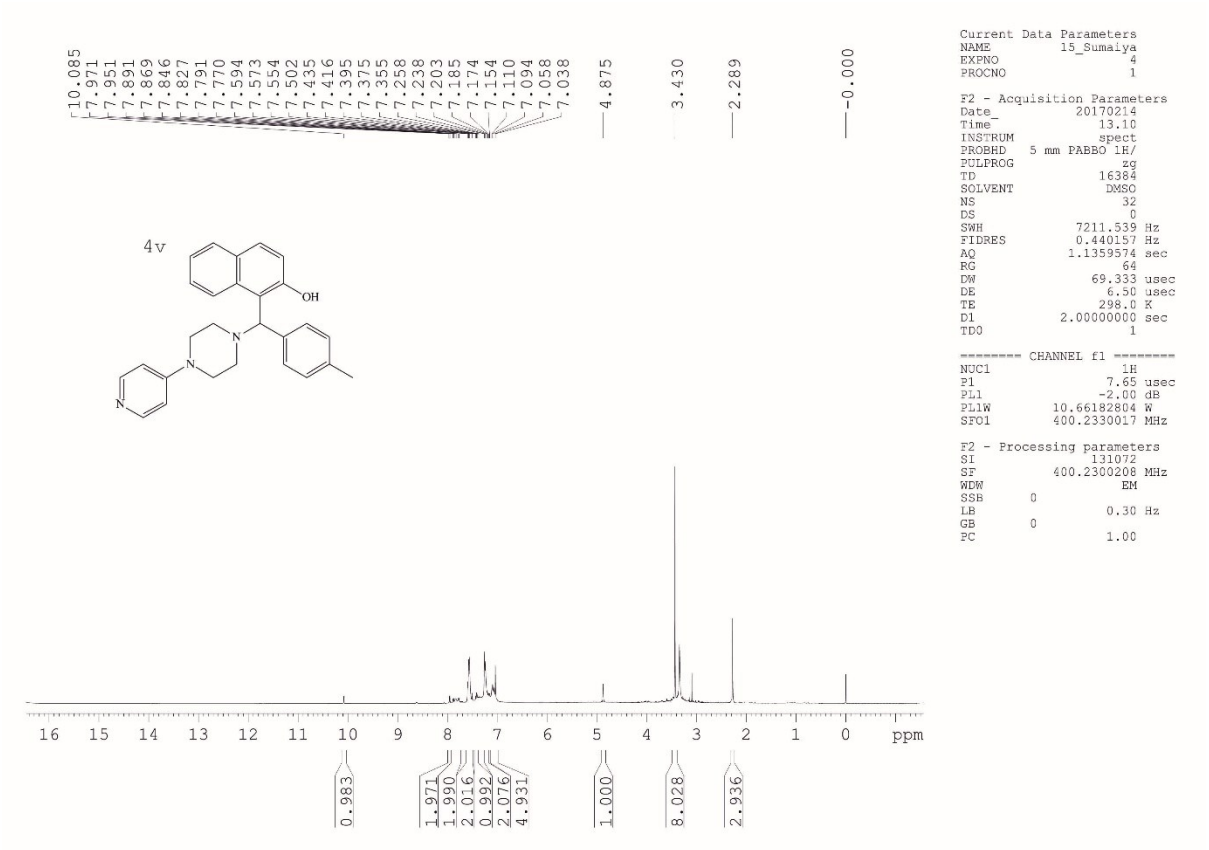
^{13}C NMR spectrum of 4u



HRMS of 4u



^{1}H NMR spectrum of 4v



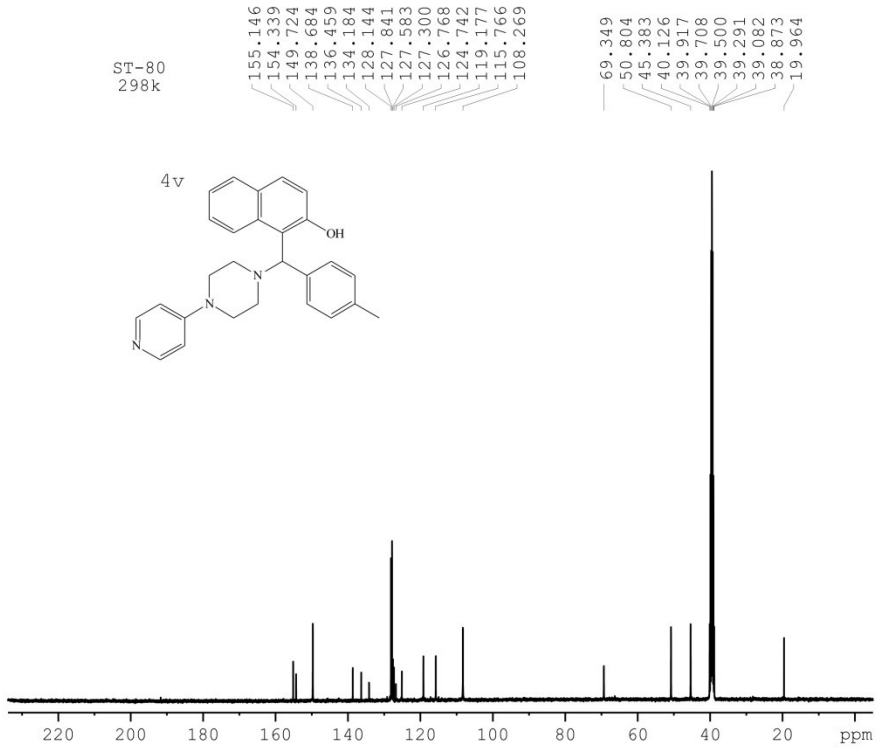

```
Current Data Parameters
NAME                  15_Sumaiya
EXPNO                          4
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20170214
Time                      13.10
INSTRUM                   spect
PROBHD    5 mm PABBO 1H/
PULPROG                      zg
TD                        16384
SOLVENT                    DMSO
NS                           32
DS                            0
SWH                    7211.539 Hz
FIDRES                 0.440157 Hz
AQ                     1.1359574 sec
RG                           64
DW                       69.333 usec
DE                        6.50 usec
TE                       298.0 K
D1                    2.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         7.65 usec
PL1                       -2.00 dB
PL1W                10.66182804 W
SFO1                400.2330017 MHz

F2 - Processing parameters
SI                        131072
SF                  400.2300208 MHz
WDW                           EM
SSB                0
LB                         0.30 Hz
GB                 0
PC                         1.00
```

^{13}C NMR spectrum of 4v




```
Current Data Parameters
NAME                  05_Sumiya
EXPNO                          6
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20171402
Time                      21.19
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                   zgpg
TD                        32768
SOLVENT                    DMSO
NS                           4096
DS                              4
SWH                    24038.461 Hz
FIDRES                 0.733596 Hz
AQ                     0.6815744 sec
RG                           203
DW                       20.800 usec
DE                        6.50 usec
TE                       298.0 K
D1                    3.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         9.50 usec
PL1                       -2.00 dB
PL1W                58.25462341 W
SFO1                100.6494874 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                          1H
PCPD2                     90.00 usec
PL2                       -3.00 dB
PL12                      13.48 dB
PL13                      14.00 dB
PL2W                13.42244530 W
PL12W                0.30187815 W
PL13W                0.26781300 W
SFO2                400.2316009 MHz

F2 - Processing parameters
SI                        131072
SF                  100.6379674 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```

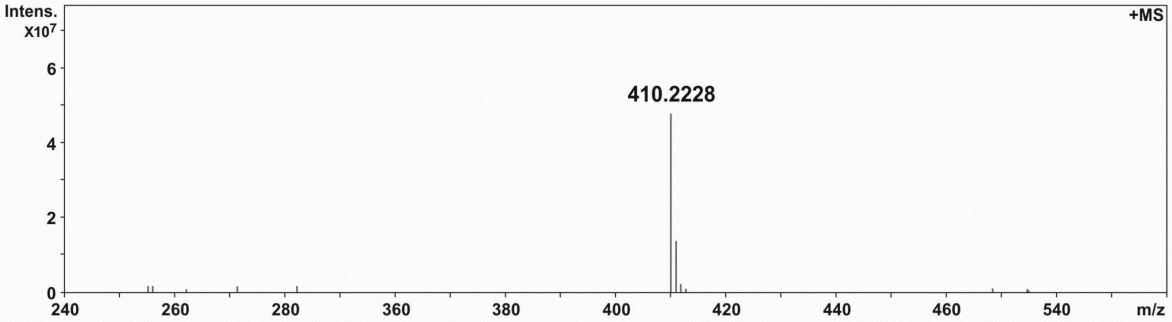
HRMS of 4v

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
15 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-50 N: 0-10 O: 0-5
4v 80 (0.453)
TOF MS ES-



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e_i Conf	N-Rule
410.2228	1	C 27 H 28 N 3 O	100.00	410.2232	0.5	1.7	8.8	15.1	even	ok