Electronic Supplementary Material (ESI) for RSC Advances.
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Electronic Supporting Information (SI)

Metal-free Heterogeneous and Mesoporous Biogenic Graphene-oxide Nanostructure Catalyzed Synthesis of Bioactive Benzylpyrazolyl Coumarin Derivatives

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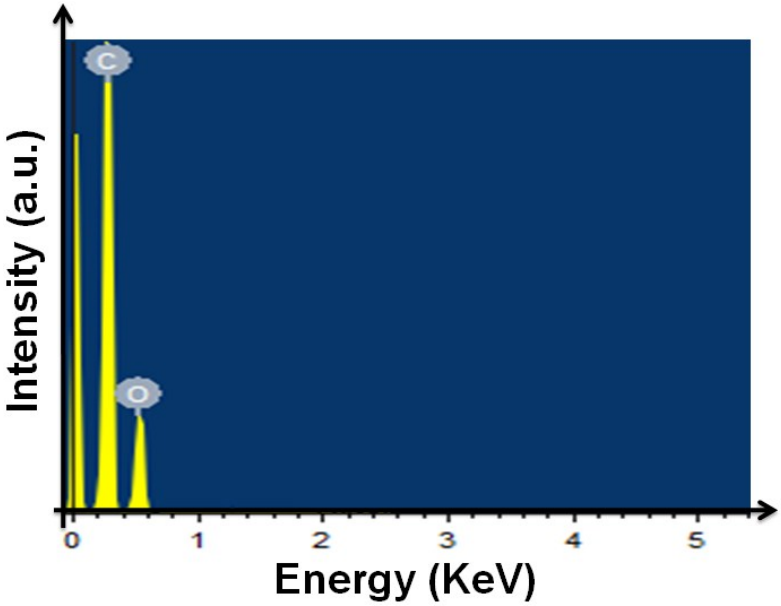


Element	Weight%	Atomic%
C K	65.67	71.82
O K	34.33	28.18
Totals	100.00	

Figure S1: Elemental mapping and analysis of GONPs.

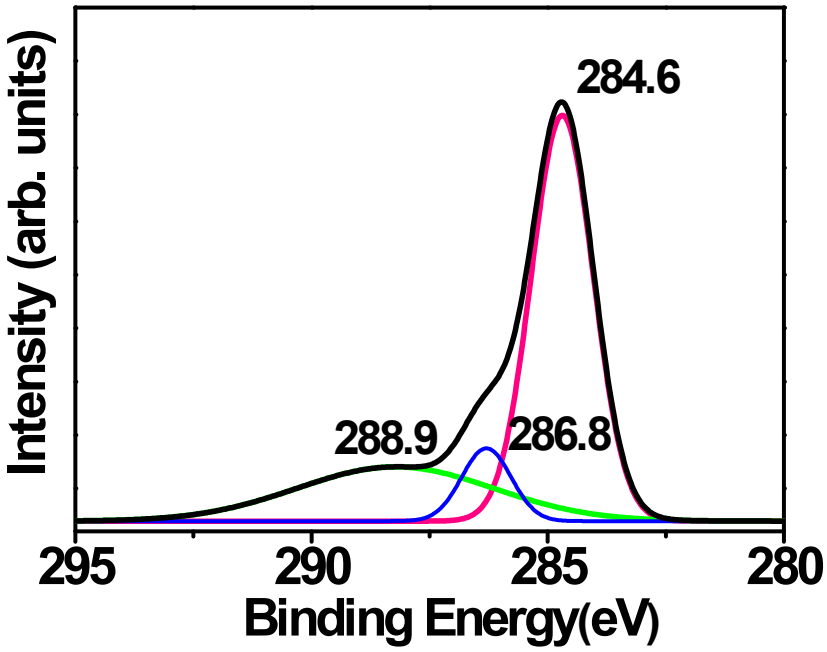
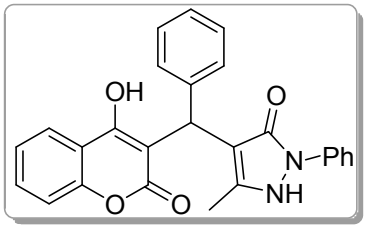


Figure S2: Deconvolution of GONPs XPS C1s peak.

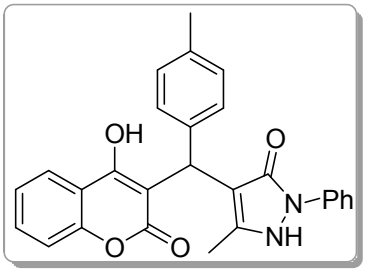
SI-1

4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(phenyl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5a)



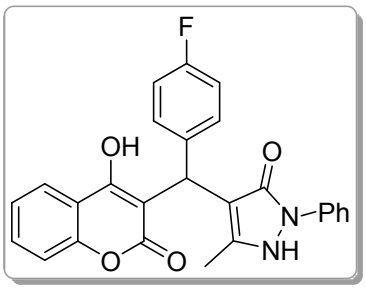
White, solid, MP: 233-234 °C; **IR (Neat):** 3082, 1655, 1615, 1561, 1492, 1185, 1035, 752 cm^{-1};**^{1}H (400 MHz; DMSO-d6):** δ 2.40 (s, 3H), 5.71 (s, 1H), 7.18-7.40 (m, 8H), 7.50 -7.60 (m, 3H), 7.63-7.68 (m, 2H), 7.805 (d, *J*=9 Hz, 2H), 12.84 (bs, 1H), 15.11 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.91, 34.12, 104.05, 105.90. 107.17, 116.37, 118.50, 121.30, 124.29, 124.55, 126.48, 127.17, 127.26 128.69, 129.74, 132.44, 135.59, 139.93, 147.41, 152.45, 162.64, 163.97, 164.72; **EI MS:** m/z (rel.abund.%) 425$[M+1]^{+}$, 90).

4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(p-tolyl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5b)



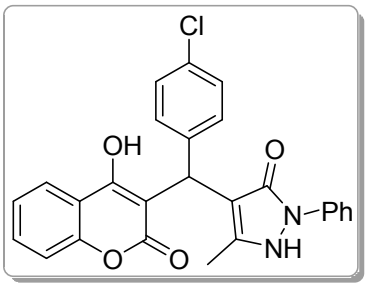
White, solid, MP: 220-222°C; **IR (Neat):** 3083, 1649, 1612, 1494, 1187, 1031, 755 cm^{-1}; **^{1}H (400 MHz; DMSO-d6):** δ 2.245 (s, 3H), 2.390 (s, 3H), 5.664 (s,1H), 7.058 (s, 4H), 7.284-7.377 (m, 3H), 7.516 (t, *J* = 7.6 Hz, 2H), 7.592 (t, 1H, J = 7.6 Hz), 7.694 (d, *J* = 8Hz, 2H), 7.80 (d, *J* = 8Hz, 1H), 12.896 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.93, 20.97, 33.83, 106.08, 107.22, 116.34, 118.59, 121.23, 124.27, 127.08, 127.17, 129.24, 129.71, 132.35, 135.38, 135.75, 136.93, 147.41, 152.45, 162.63, 163.97, 164.71; **EI MS:** m/z (rel. abund.%) 439.09 ($[M+1]^{+}$, 100).

4-((4-fluorophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5c)



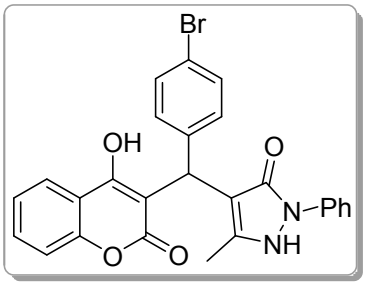
White, solid, MP: 241-243°C;**IR (Neat):** 3081, 1658, 1613, 1566, 1499, 1190, 1040, 761 cm^{-1}**^{1}H (400 MHz; DMSO-d6):** δ 2.393 (s, 3H), 5.677(s, 1H), 7.071 (t, *J* = 8.8 Hz, 2H), 7.195-7.229 (m, 2H), 7.285-7.377(m, 3H), 7.517 (t, *J* = 8 Hz, 2H), 7.578-7.614 (m, 1H), 7.694 (d, *J* = 8Hz, 2H), 7.804 (d, *J* = 7.6 Hz, 1H), 12.816 (bs, 1H), 15.059 (bs 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.91, 33.65, 105.80, 107.18, 115.18, 115.40, 116.35, 118.55, 121.35, 124.30, 127.18, 129.15, 129.22, 129.71, 132.43, 135.63, 135.99, 147.37, 152.49, 162.41, 162.52, 164.06, 164.62; **EI MS:** m/z (rel.abund.%) 443.09 ($[M+1]^+$, 100).

4-((4-chlorophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5d)



White, solid, MP: 227-229 °C;**IR (Neat):** 3078, 1655, 1613, 1570, 1490, 1187, 1099, 756 cm^{-1};**^{1}H (400 MHz; DMSO-d6):** δ 2.373 (s, 3H), 5.658 (s, 1H), 7.203 (d, *J* = 8 Hz, 2H), 7.274 -7.363 (m, 5H), 7.503 (t, *J* = 8.4 Hz, 2H), 7.565 - 7.601 (m, 1H), 7.704 (d, *J* = 8 Hz, 2H), 7.801 (d, *J* = 7.6 Hz, 1H), 12.736 (bs, 1H), 15.179 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.91, 33.75, 105.46, 106.97, 116.40, 118.49, 121.37, 124.31, 124.35, 127.25, 128.58, 129.28, 129.74, 131.13, 132.52, 135.52, 139.05, 147.31, 152.48, 162.36, 164.13, 164.60; **EI MS:**m/z (rel.abund.%) 459.05 $[M+1]^+$, 100), 461.05 ($[M+2]^+$, 40).

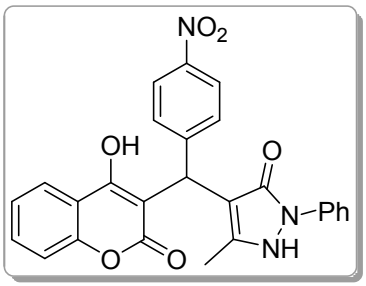
4-((4-bromophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5e)



White, solid, MP: 240-242°C;**IR (Neat):** 3078, 1654, 1613, 1571, 1490, 1187, 1099, 759 cm^{-1}; **^{1}H (400 MHz; DMSO-d6):** δ 2.395 (s, 3H), 5.656 (s, 1H), 7.148 (d, *J* = 8Hz, 2H), 7.287 - 7.379(m, 3H), 7.442 (d, *J* = 8.4 Hz, 2H), 7.518 (t, *J* = 8.4Hz, 2H), 7.576 - 7.618(m, 1H), 7.690 (d, *J* = 8Hz, 2H), 7.803 (dd, *J* = 1.2Hz ,8Hz, 1H), 12.856 (bs, 1H), 15.179 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.93, 33.88, 105.43, 106.86, 116.37, 118.57, 119.57,

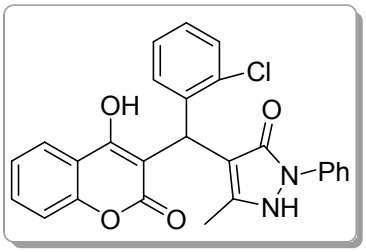
121.36, 124.32, 127.19, 129.71, 131.48, 132.47, 135.64, 139.63, 147.39, 152.51, 162.33, 164.21, 164.60; **EI MS:** m/z (rel.abund.%) 504.94 $[M+2]^+$, 100), 502.99 ($[M+1]^+$, 90).

4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(4-nitrophenyl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5f)



Pale yellow, solid, MP: 248-250 ^{0}C **IR (Neat):** 3073, 1649, 1608, 1560, 1525, 1178, 1039, 748 cm^{-1}; **^{1}H (400 MHz; DMSO-d6):** δ 2.404 (s, 3H), 5.776 (s, 1H), 7.285-7.388 (m, 3H), 7.471-7.536 (m, 4H), 7.604 (t, 1H), 7.703 (d, 2H, J = 7.6 Hz, 2H), 7.790-7.810 (m, 1H), 8.132 (d, J = 8.8 Hz, 2H), 15.219 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.63, 34.27, 104.59, 106.36, 116.10, 118.26, 121.16, 123.49, 124.06, 126.98, 128.46, 129.40, 132.27, 135.25, 146.19, 147.03, 148.31, 152.27, 161.73, 164.24; **EI MS:**m/z (rel.abund.%) 470.05 ($[M+1]^+$ 100).

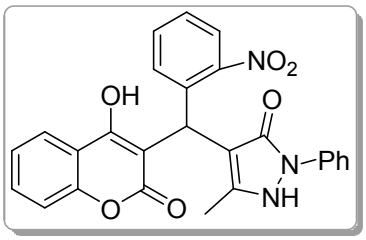
4-((2-chlorophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5g)



White, solid, MP : 238-240°C;**IR (Neat):** 3079, 1655, 1623, 1570, 1493, 1189, 1094, 751 cm^{-1}; **^{1}H (400 MHz; DMSO-d6):** δ 2.386 (s,3H), 5.730 (s,1H), 7.349-7.790(m, 12H), 12.856 (bs, 1H), 14.859 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.69, 33.45, 104.06, 107.01, 115.90, 118.22, 121.04, 123.87, 126.73, 128.27, 129.30, 129.89, 130.18, 131.89, 132.69, 135.29, 137.75, 146.67, 151.98, 162.51, 163.25, 163.61; **EI MS**: m/z (rel.abund.%) 459.05 ($[M+1]^+$ 100), 461.00 ($[M+2]^+$ 40).

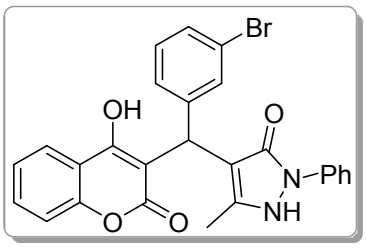
4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(2-nitrophenyl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5h)

Pale yellow, solid, MP: 240-242°C **IR (Neat):** 3073, 1649, 1608, 1560, 1525, 1178, 1039, 748 cm-1; **^{1}H (400 MHz; DMSO-d6):** δ 2.362 (S, 3H), 6.063 (S, 1H), 7.277-7.363(m, 3H), 7.450-7.524(m, 4H), 7.569-7.686(m, 5H), 7.775 (dd, *J* = 1.2Hz, 8Hz, 1H), 14.688, (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.94, 31.76, 79.65 104.19, 106.07, 116.37, 118.20, 121.52, 124.32, 124.69, 127.31, 128.35, 129.71, 130.33,



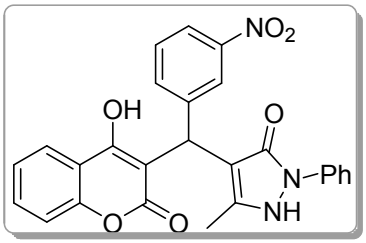
132.39, 132.53, 132.76, 135.50, 147.23, 150.17, 152.48, 162.36, 163.77, 164.23; **EI MS:** m/z (rel.abund.%) 470.12 ([M+1]$^+$ 100).

4-((3-bromophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5i)



White, solid, MP: 224-226°C;**IR (Neat):** 3078, 1655, 1613, 1570, 1490, 1187, 1099, 756 cm-1; **^{1}H (400 MHz; DMSO-d6):** δ 2.397 (s, 3H), 5.707 (s, 1H), 7.203 -7.260 (m, 2H), 7.292 -7.403 (m, 5H), 7.523 (t, *J* = 8 Hz, 2H), 7.581 -7.824 (m, 1 H), 7.694 (d, *J* = 7.6 Hz, *J* = 8 Hz, 2H), 7.816 (dd, *J* = 1.2 Hz, *J* = 8 Hz, 1H), 12.856 (bs, 1H), 15.179 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.93, 34.08, 105.29, 106.83, 116.39, 118.52, 121.39, 122.17, 124.34, 126.62, 127.25, 129.52, 129.74, 129.98, 130.83, 132.49, 135.56, 143.09, 147.45, 152.52, 162.41, 164.18, 164.58; **EI MS:** m/z (rel.abund.%) 504.97 ([M+2]$^+$ 100), 502.94 ([M+1]$^+$ 80).

4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(3-nitrophenyl)methyl)-5-methyl-2-phenyl-1H-pyrazol-3(2H)-one (5j)

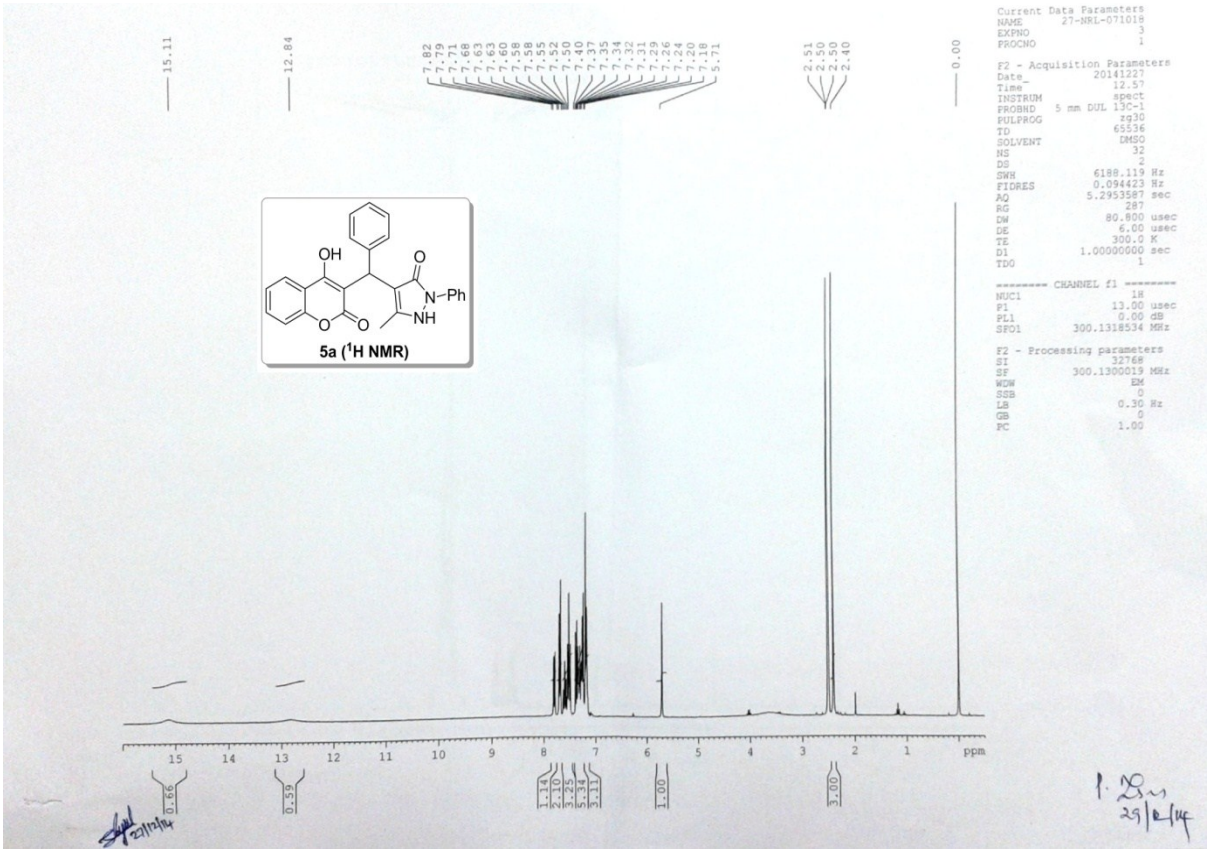


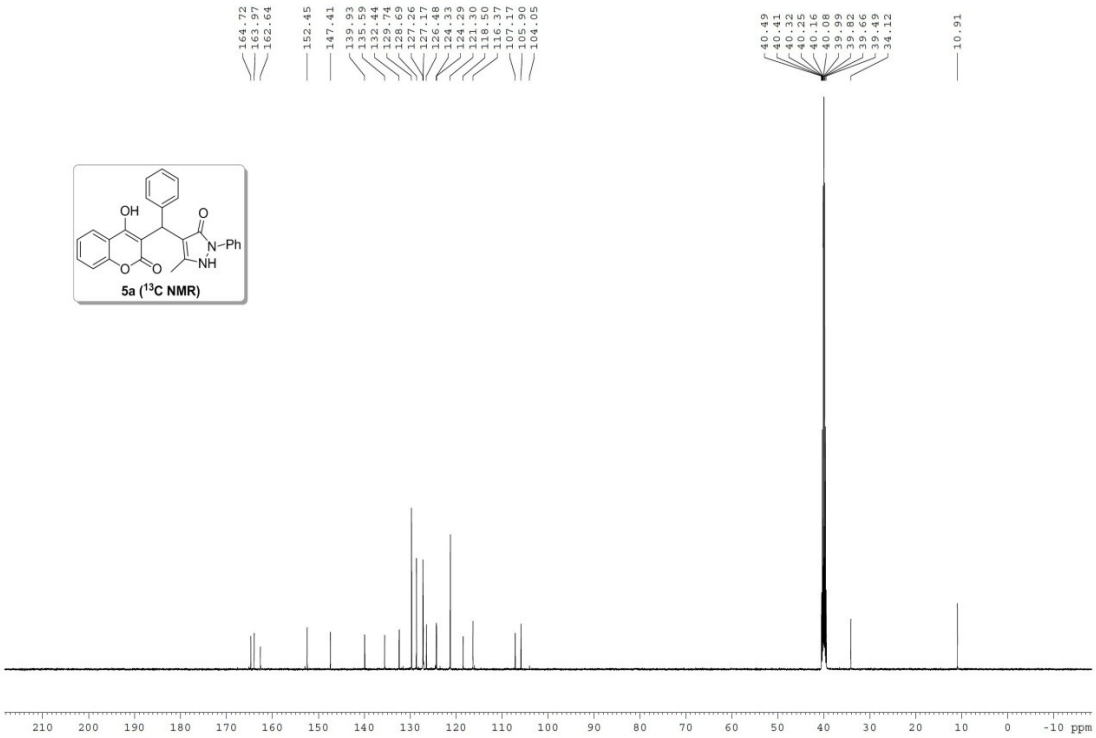
Pale yellow, solid, MP: 241-243 °C;**IR (Neat):** 3074, 1649, 1609, 1560, 1525, 1179, 1103, 749 cm^{-1} **^{1}H (400 MHz; DMSO-d6):** δ 2.501 (H, 3H), 5.797(S, H), 7.309-7.395(m, 3H), 7.525-7.603(m, 4H), 7.693 - 7.714 (m, 3H), 7.808 (d, *J* = 7.6 Hz, 1H), 7.996 (s, 1H), 8.079 (d, *J* = 4.4 Hz, 1H), 15.300 (bs, 1H); **^{13}C (100 MHz, DMSO-d6):** δ 10.63, 33.87, 104.39, 106.24, 116.05, 118.21, 121.14, 121.39, 121.55, 123.99, 126.95, 129.38, 129.86 132.21, 134.11, 135.22, 142.43, 147.11, 148.02, 152.22, 161.64, 164.19; **EI MS:** m/z (rel.abund.%) 470.05 ([M+1]$^+$ 100).

Spectra

.

Representative ^{1}H NMR, ^{13}C NMR and mass spectra of the final product CBDs (5a-5j) were shown in below.

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147.41
139.93
135.59
132.44
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128.69
128.26
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126.48
124.33
124.29
121.30
118.50
116.37
107.17
105.90
104.05
40.49
40.41
40.32
40.25
40.16
40.08
39.99
39.82
39.66
39.49
34.12
10.91
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O
N–Ph
NH
O
O
5a (13C NMR)
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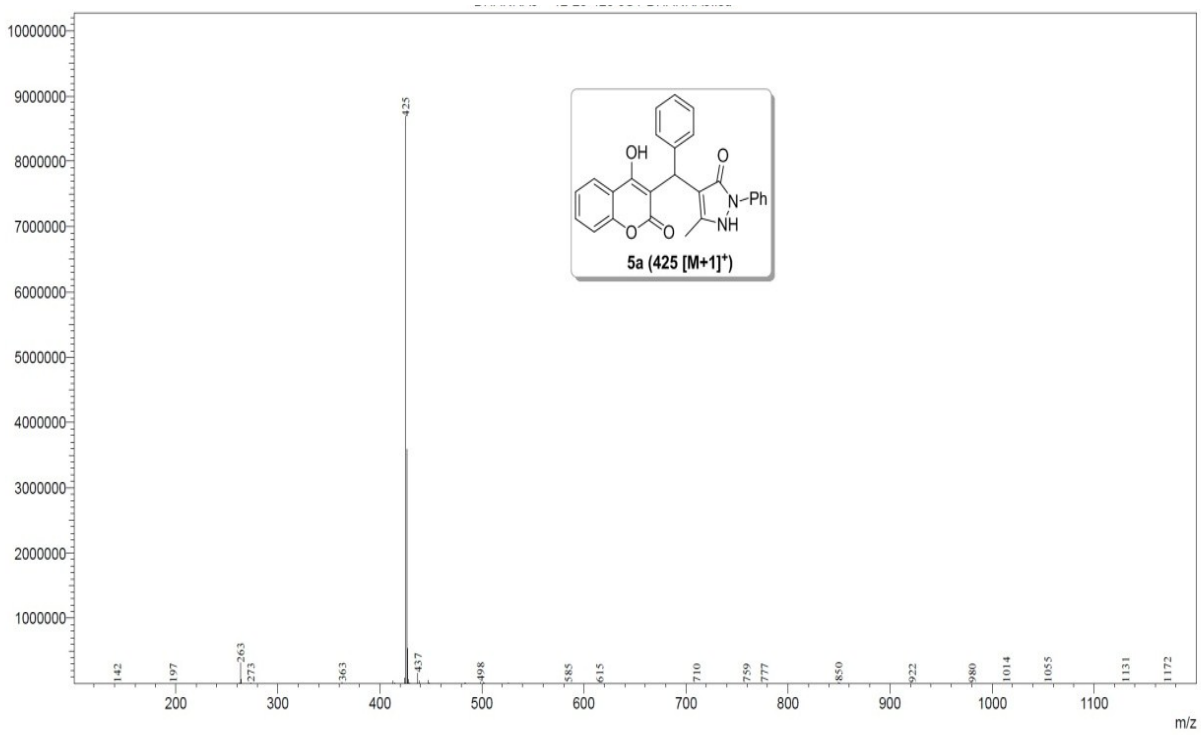
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8000000
7000000
6000000
5000000
4000000
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1000000
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OH
O
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NH
O
O
5a (425 [M+1]+)
142
197
263
273
363
437
498
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922
980
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1131
1172
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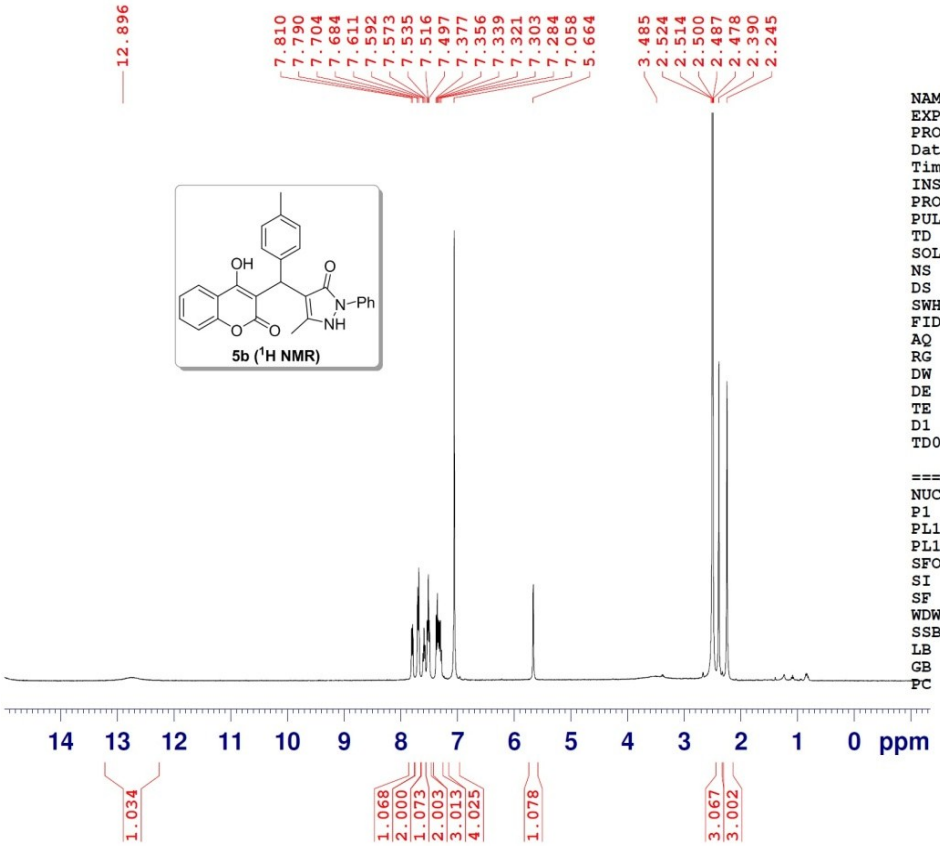
m/z

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TD                 24670
SOLVENT             DMSO
NS                    16
DS                     0
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FIDRES          0.487200 Hz
AQ             1.0263220 sec
RG                   287
DW                41.600 usec
DE                  6.50 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300086 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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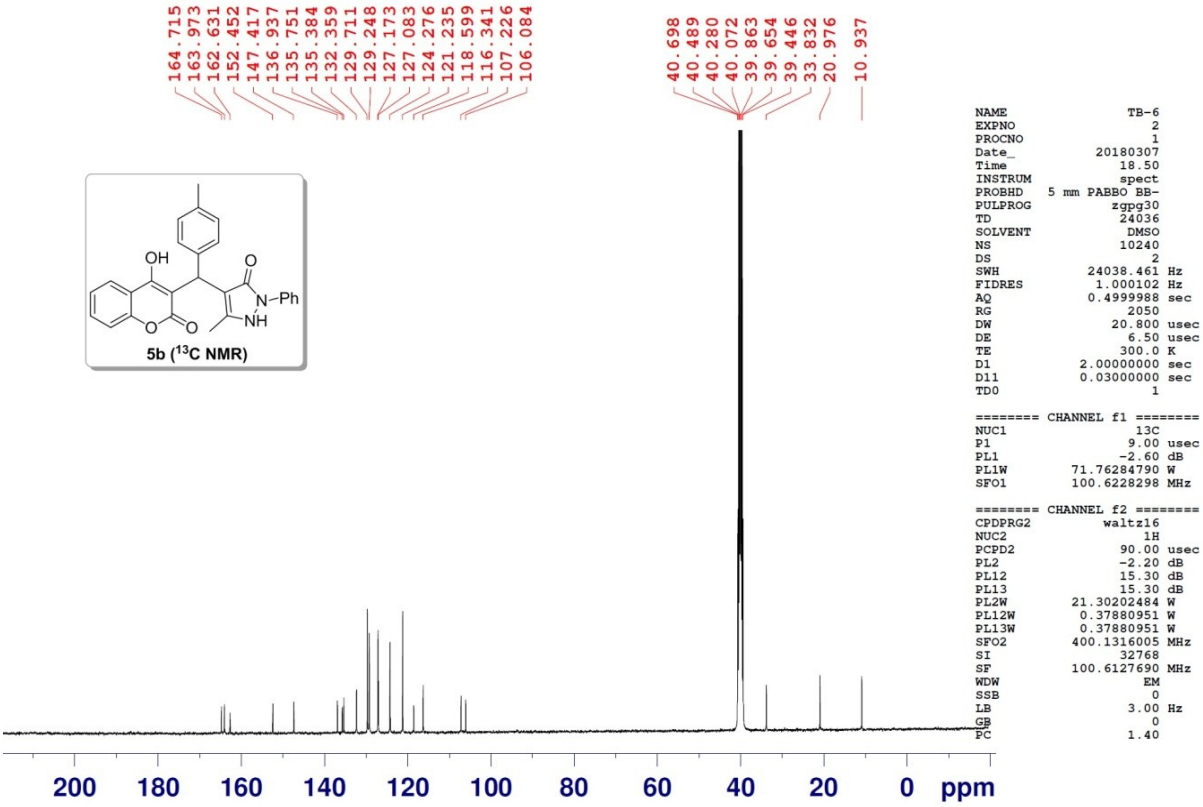
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124.276
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118.599
116.341
107.226
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40.489
40.280
40.072
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39.654
39.446
33.832
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NH
O
O
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PROCNO 1
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Time 18.50
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FIDRES 1.000102 Hz
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RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
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NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
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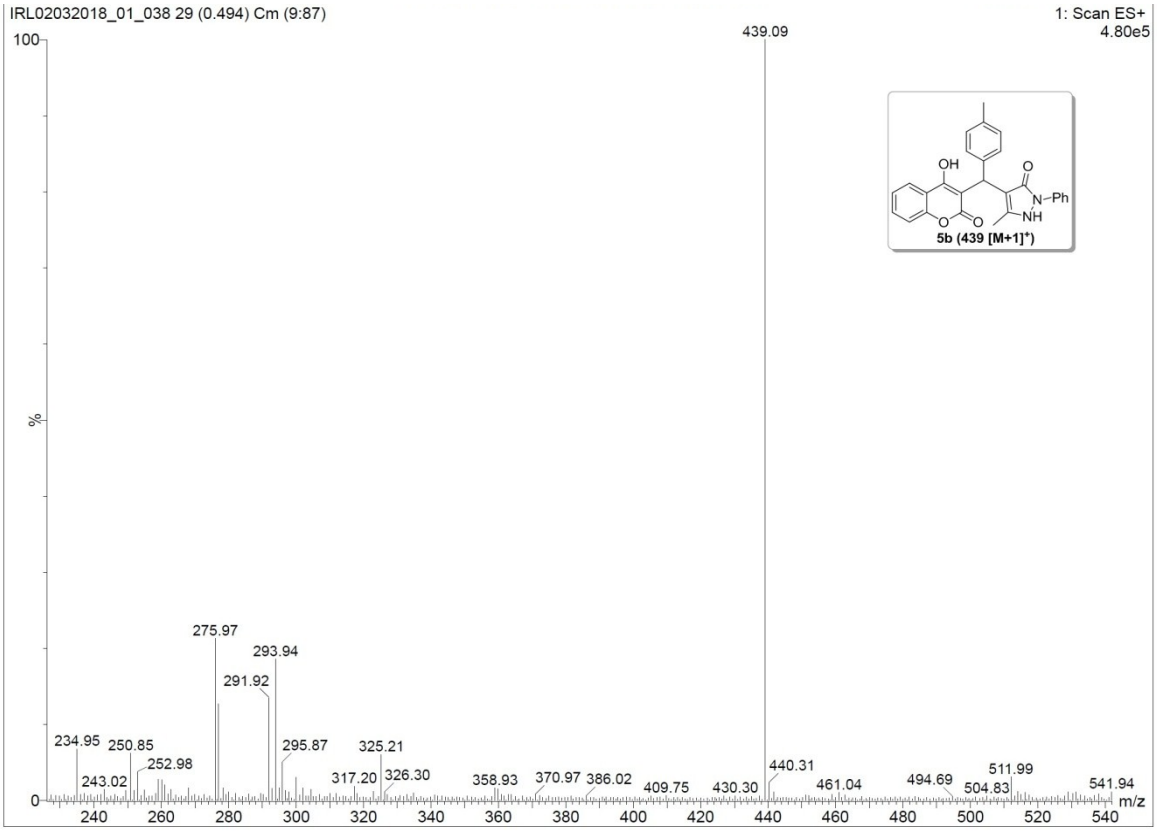
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%
0
439.09
OH
O
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NH
O
O
5b (439 [M+1]$^+$)
275.97
293.94
291.92
234.95
250.85
252.98
243.02
295.87
325.21
317.20
326.30
358.93
370.97
386.02
409.75
430.30
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541.94
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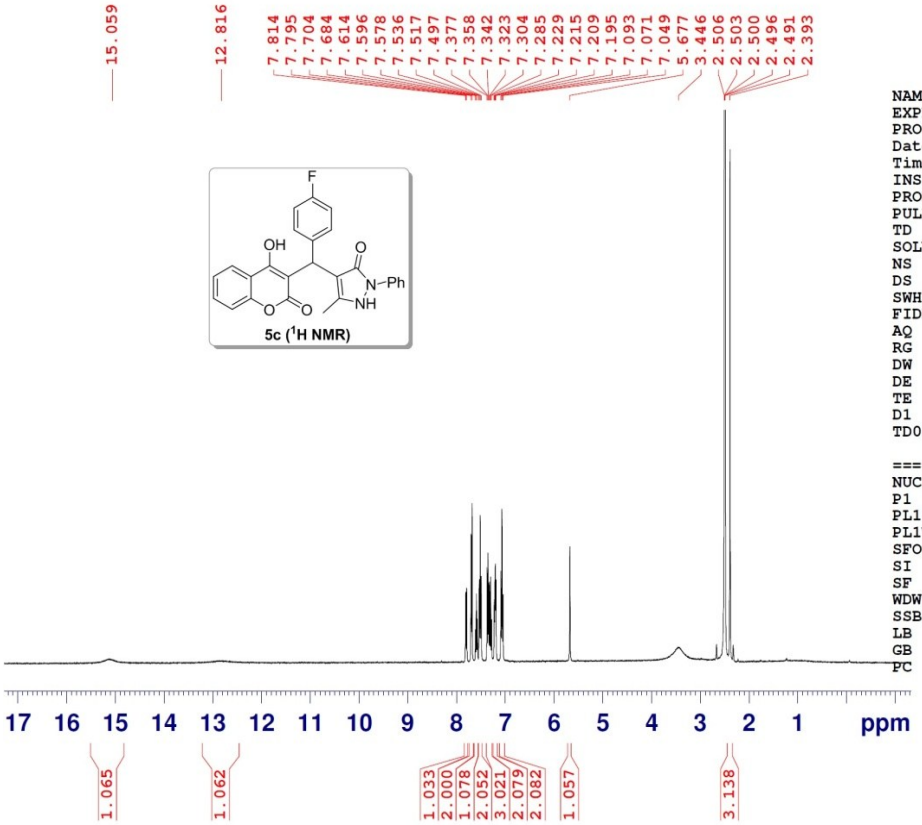
m/z

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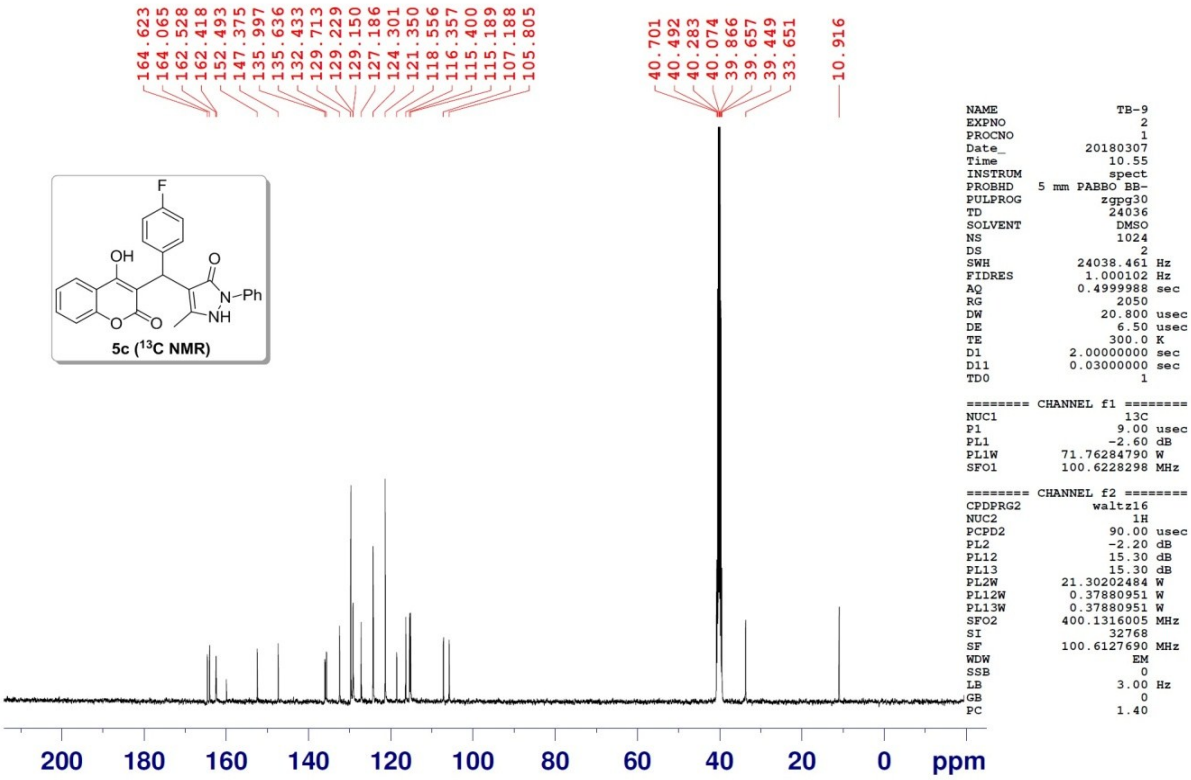
PROCNO                 1
Date_           20180306
Time               17.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES          0.487200 Hz
AQ             1.0263220 sec
RG                   287
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

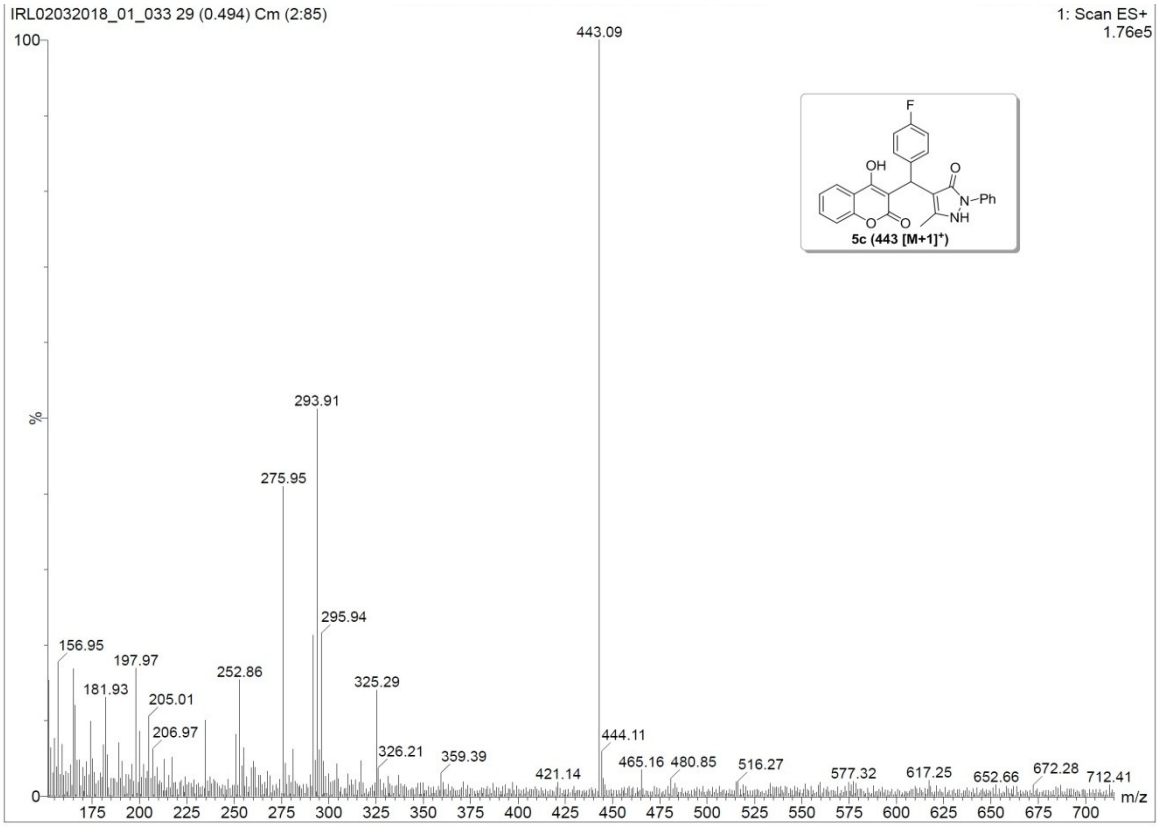
======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300081 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

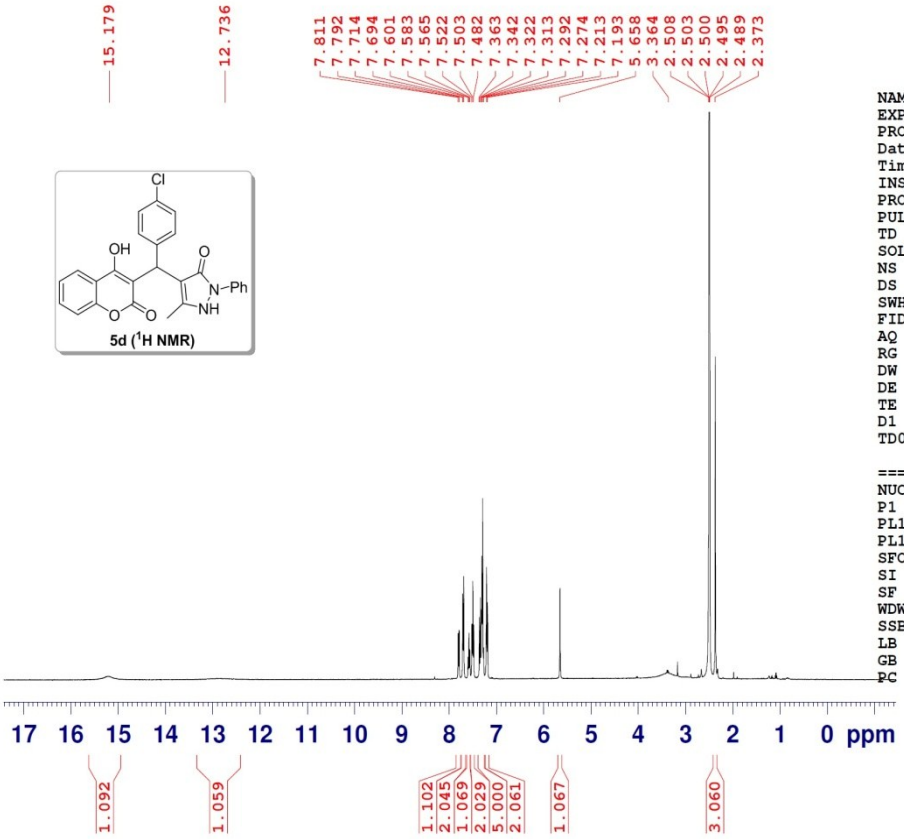



```
NAME                TB-9
EXPNO                  2
PROCNO                 1
Date_           20180307
Time               10.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 24036
SOLVENT             DMSO
NS                  1024
DS                     2
SWH            24038.461 Hz
FIDRES          1.000102 Hz
AQ             0.4999988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -2.60 dB
PL1W         71.76284790 W
SFO1         100.6228298 MHz

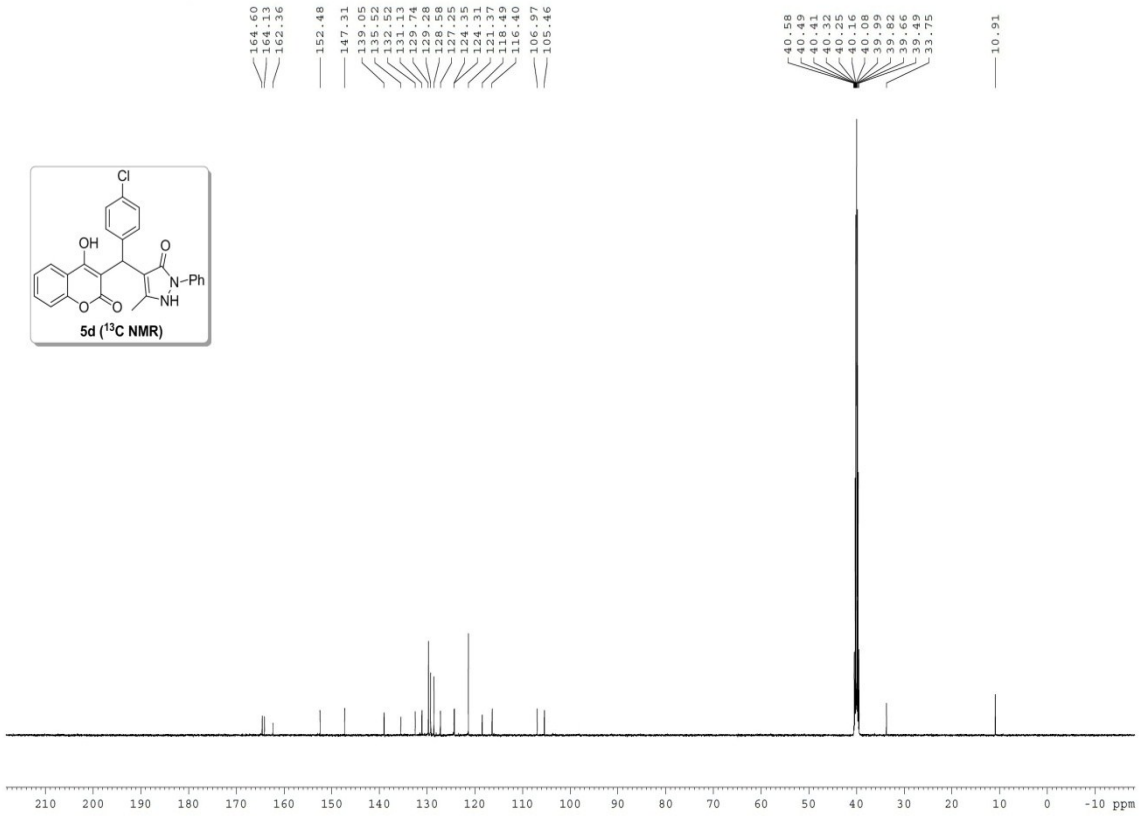
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.20 dB
PL12               15.30 dB
PL13               15.30 dB
PL2W         21.30202484 W
PL12W         0.37880951 W
PL13W         0.37880951 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40
```

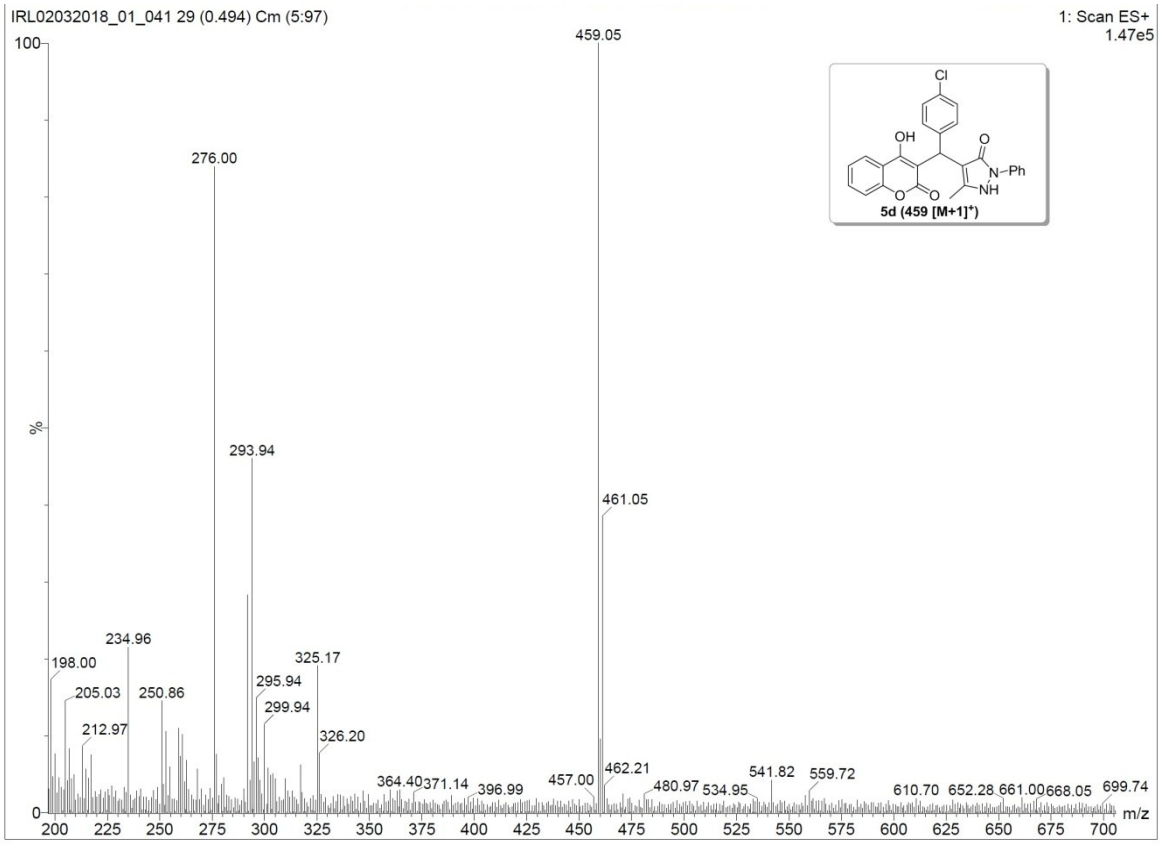


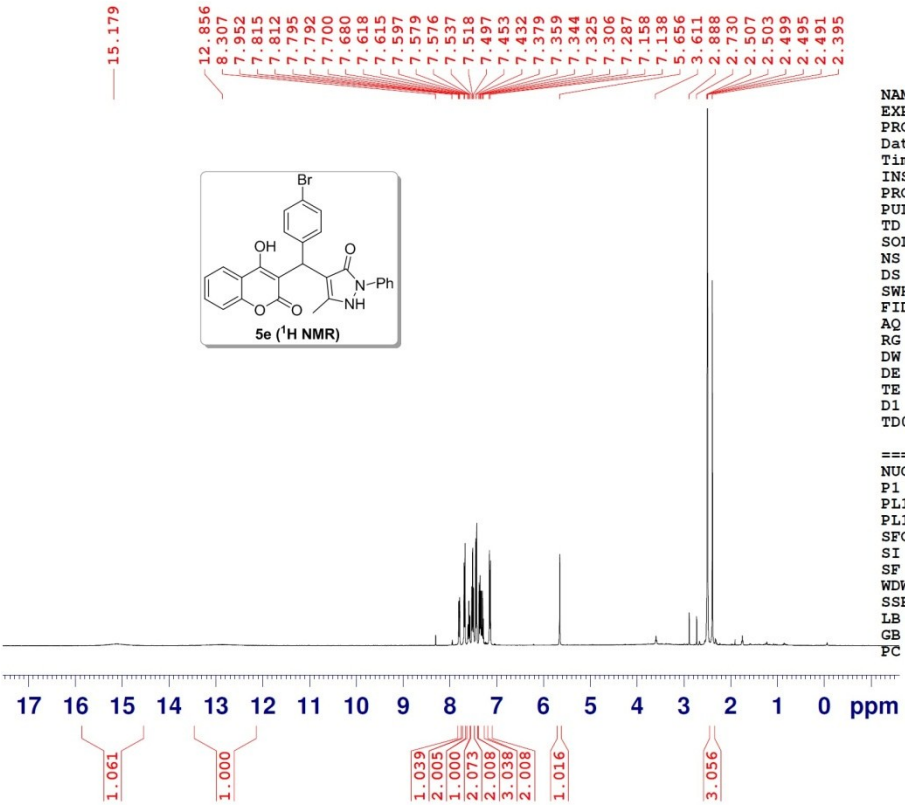



```
NAME                TB-10
EXPNO                   1
PROCNO                  1
Date_            20180306
Time                17.46
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  24670
SOLVENT              DMSO
NS                     32
DS                      0
SWH             12019.230 Hz
FIDRES           0.487200 Hz
AQ              1.0263220 sec
RG                    287
DW                 41.600 usec
DE                   6.50 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -2.20 dB
PL1W         21.30202484 W
SFO1         400.1332711 MHz
SI                  32768
SF           400.1300082 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

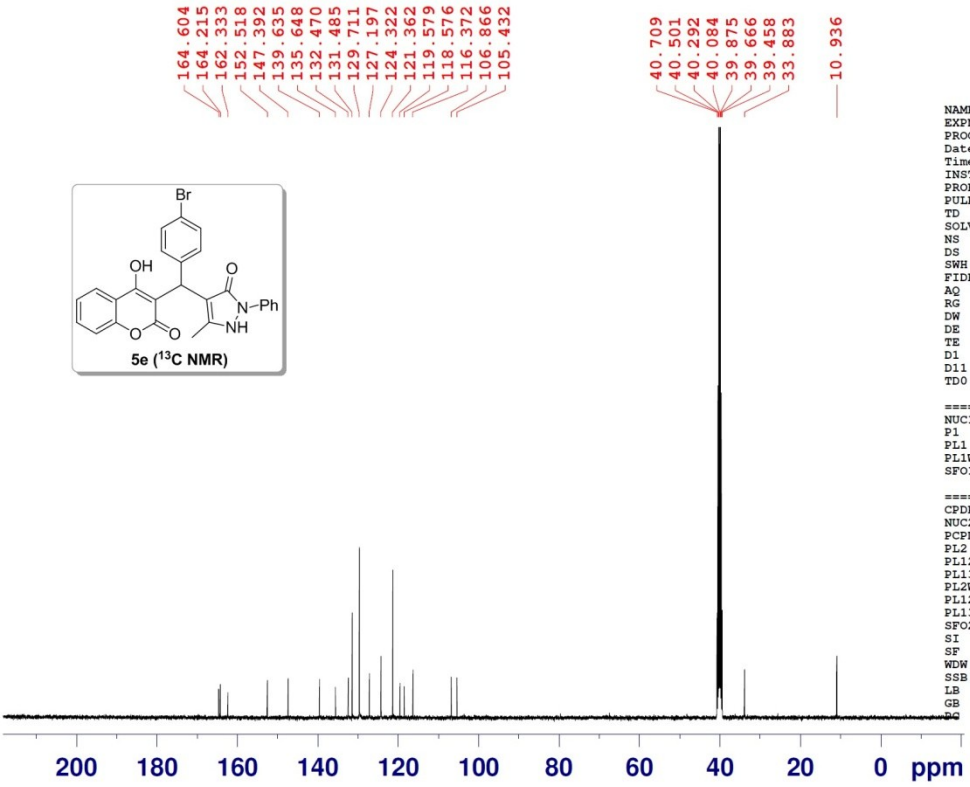

164.60
164.13
162.36
152.48
147.31
139.05
135.52
132.52
131.13
129.74
129.28
128.58
127.25
124.35
124.31
121.37
118.49
116.40
106.97
105.46
40.58
40.49
40.41
40.32
40.25
40.16
40.08
39.99
39.82
39.66
39.49
33.75
10.91
Cl
OH
O
N–Ph
NH
O
O
5d (13C NMR)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm


IRL02032018_01_041 29 (0.494) Cm (5:97)
1: Scan ES+
1.47e5
100
%
0
459.05
276.00
293.94
461.05
234.96
198.00
325.17
205.03
250.86
295.94
299.94
212.97
326.20
364.40
371.14
396.99
457.00
462.21
480.97
534.95
541.82
559.72
610.70
652.28
661.00
668.05
699.74
Cl
OH
O
N–Ph
NH
O
O
5d (459 [M+1]+)
200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700
m/z




```
NAME                TB-7
EXPNO                  1
PROCNO                 1
Date_           20180306
Time               17.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES          0.487200 Hz
AQ             1.0263220 sec
RG                   287
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
TD0                    1

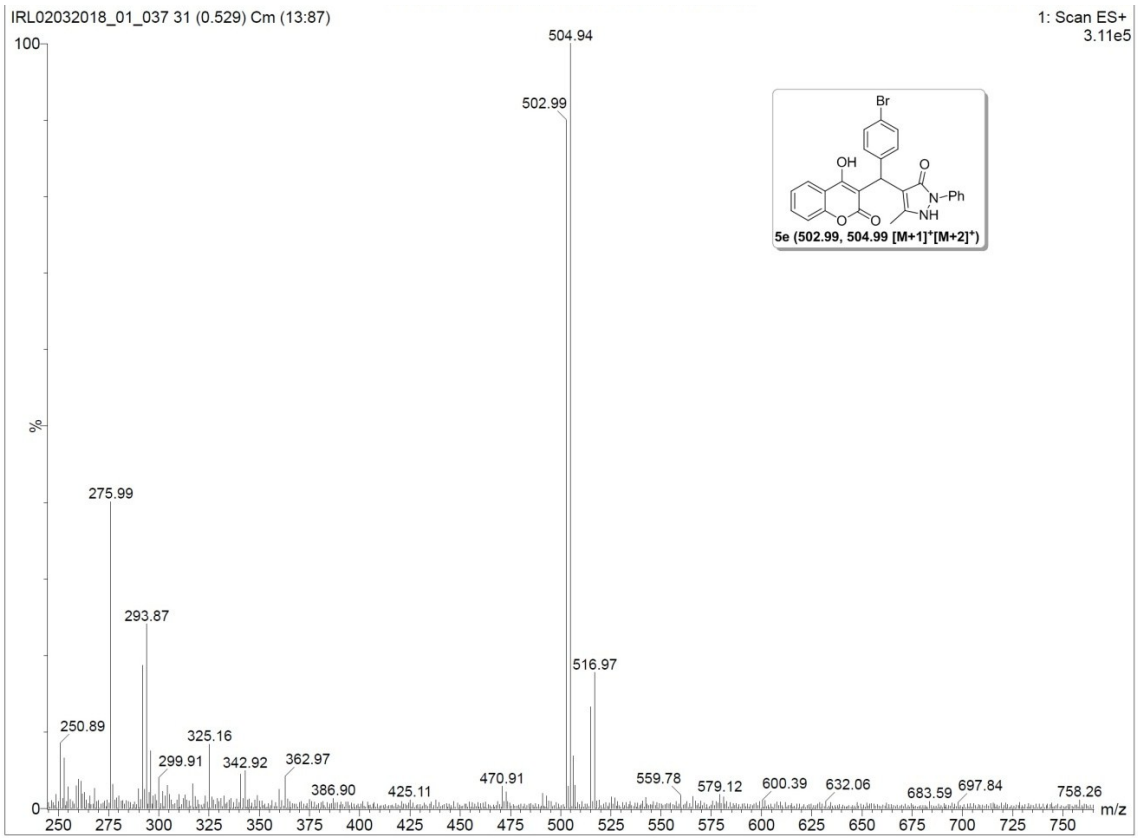
======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300082 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

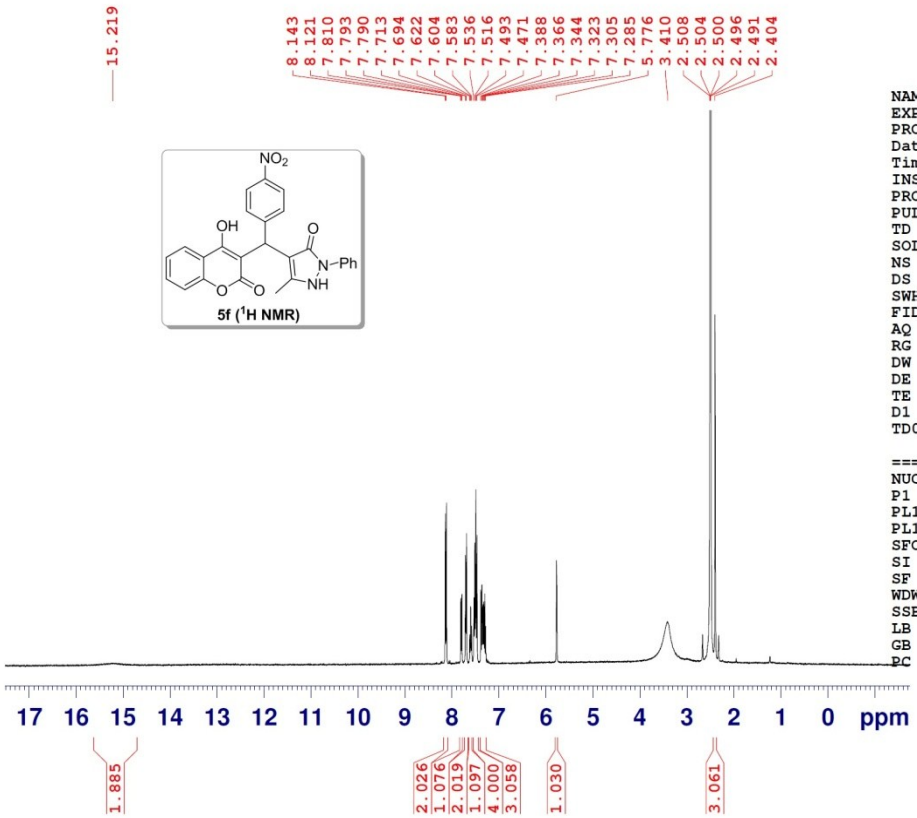



```
NAME                TB-7
EXPNO                  2
PROCNO                 1
Date_           20180307
Time               12.49
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 24036
SOLVENT             DMSO
NS                  1604
DS                     2
SWH            24038.461 Hz
FIDRES          1.000102 Hz
AQ            0.4999988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -2.60 dB
PL1W        71.76284790 W
SFO1       100.6228298 MHz

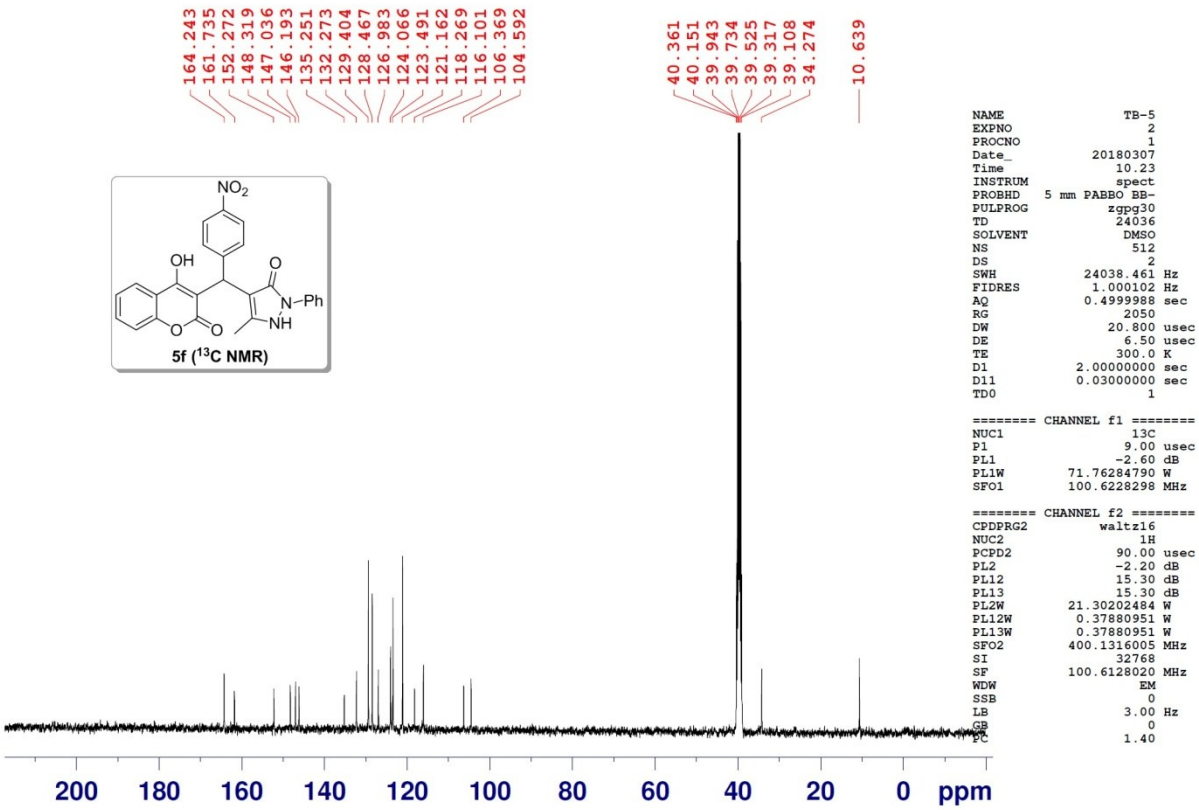
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.20 dB
PL12               15.30 dB
PL13               15.30 dB
PL2W        21.30202484 W
PL12W        0.37880951 W
PL13W        0.37880951 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

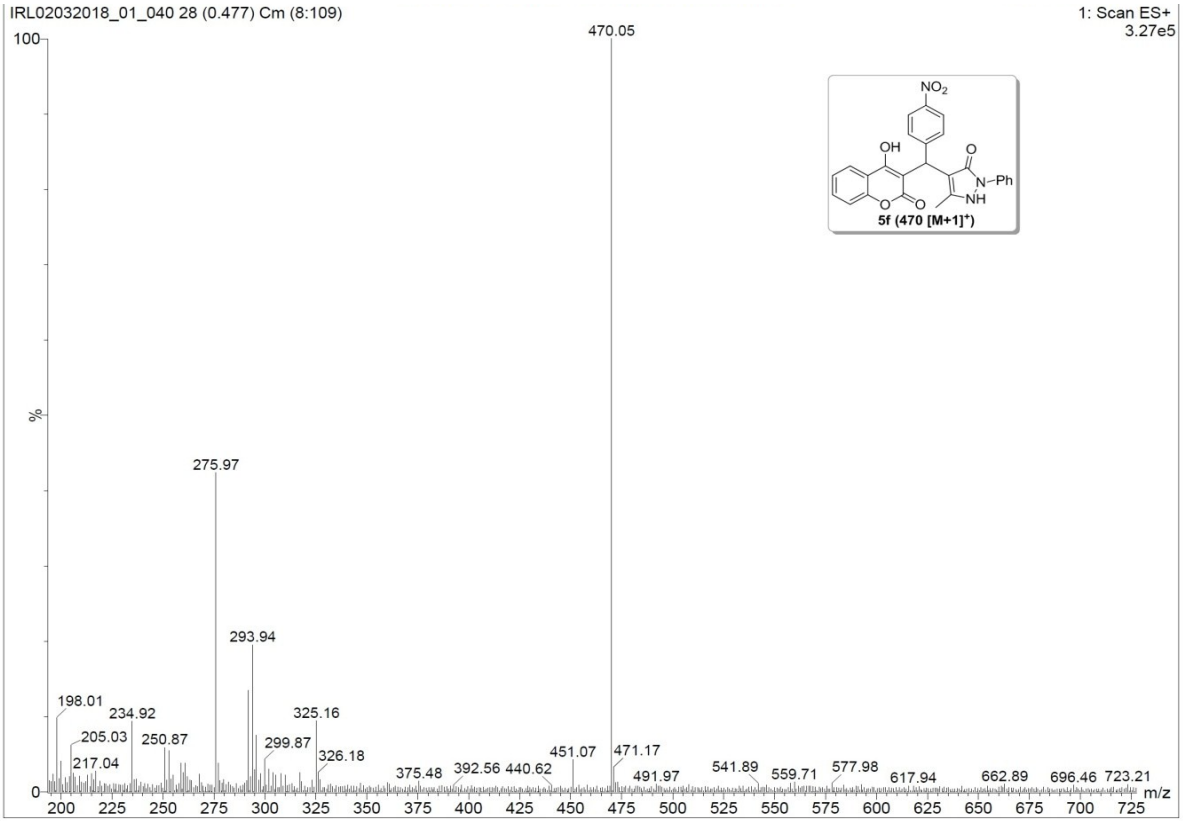


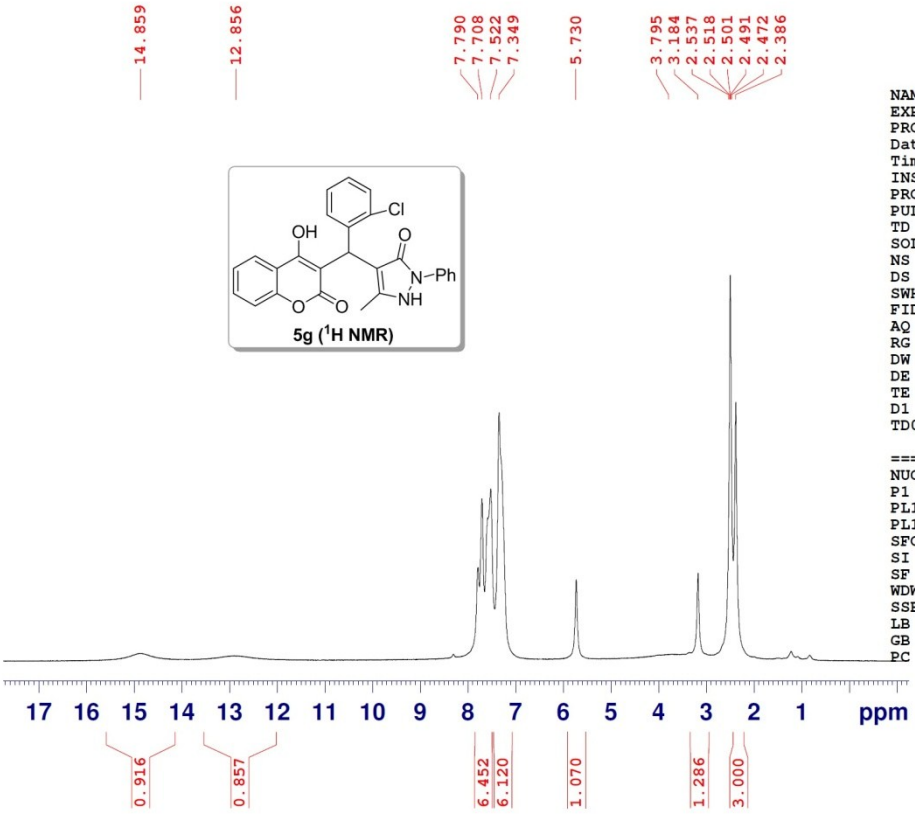



```
NAME                TB-5
EXPNO                  1
PROCNO                 1
Date_           20180306
Time               17.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    32
DS                     0
SWH            12019.230 Hz
FIDRES          0.487200 Hz
AQ             1.0263220 sec
RG                   287
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300081 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

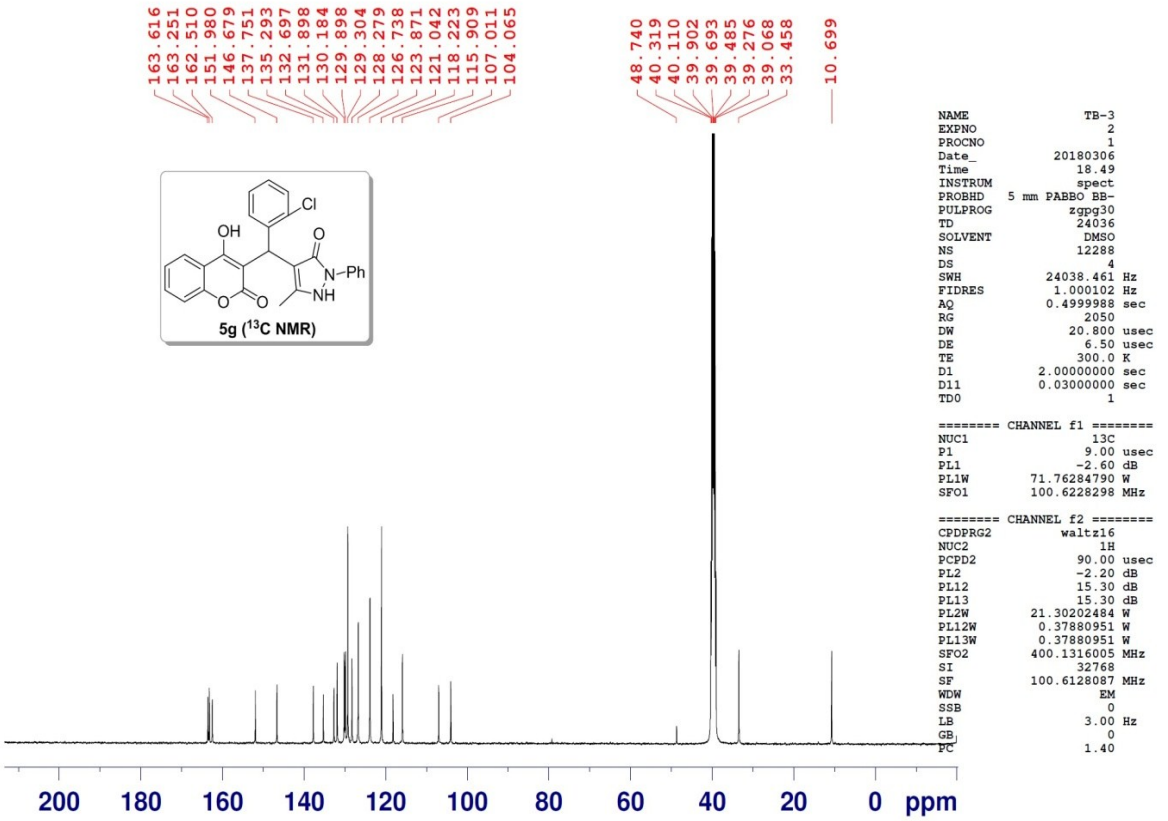

164.243
161.735
152.272
148.319
147.036
146.193
135.251
132.273
129.404
128.467
126.983
124.066
123.491
121.162
118.269
116.101
106.369
104.592
40.361
40.151
39.943
39.734
39.525
39.317
39.108
34.274
10.639
NO2
OH
O
N–Ph
NH
O
O
5f (13C NMR)
NAME TB-5
EXPNO 2
PROCNO 1
Date_ 20180307
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 24036
SOLVENT DMSO
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 1.000102 Hz
AQ 0.4999988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128020 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

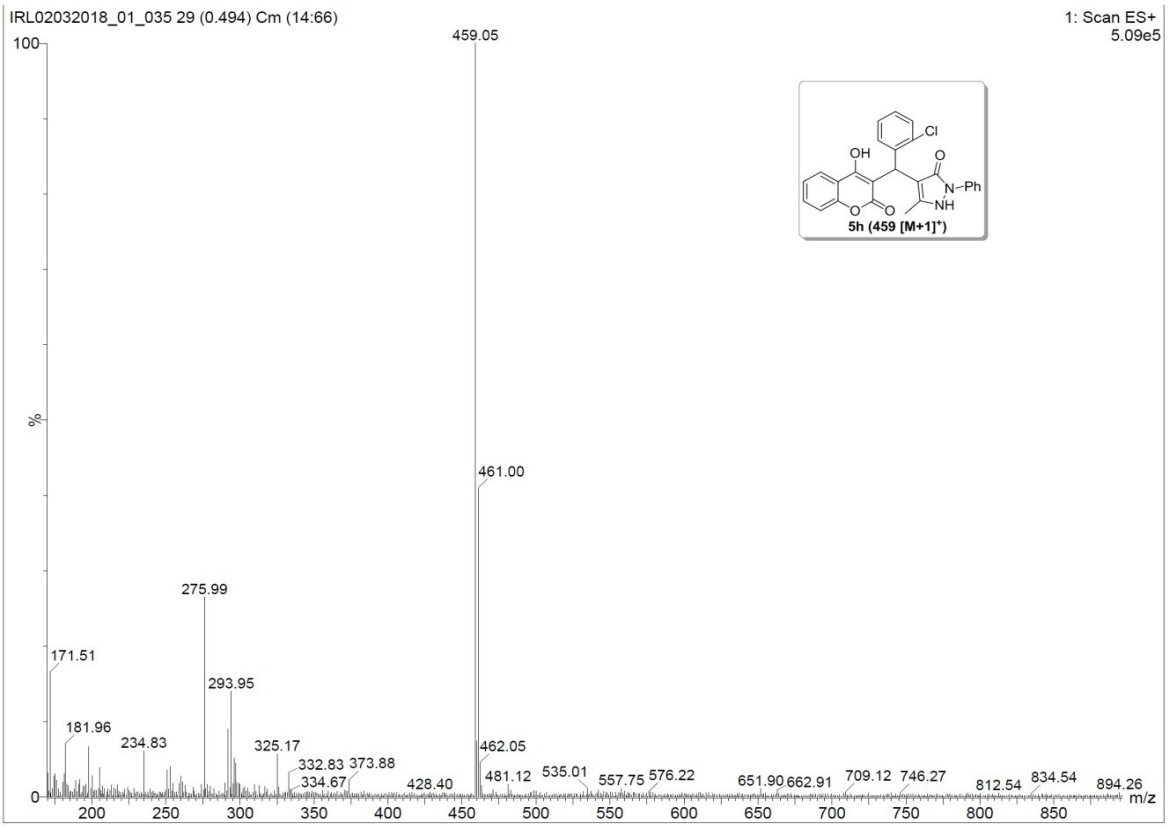

IRL02032018_01_040 28 (0.477) Cm (8:109)
1: Scan ES+
3.27e5
100
%
0
470.05
275.97
293.94
198.01
234.92
205.03
250.87
217.04
325.16
299.87
326.18
375.48
392.56
440.62
451.07
471.17
491.97
541.89
559.71
577.98
617.94
662.89
696.46
723.21
NO2
OH
O
N–Ph
NH
O
O
5f (470 [M+1]+)
200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725
m/z

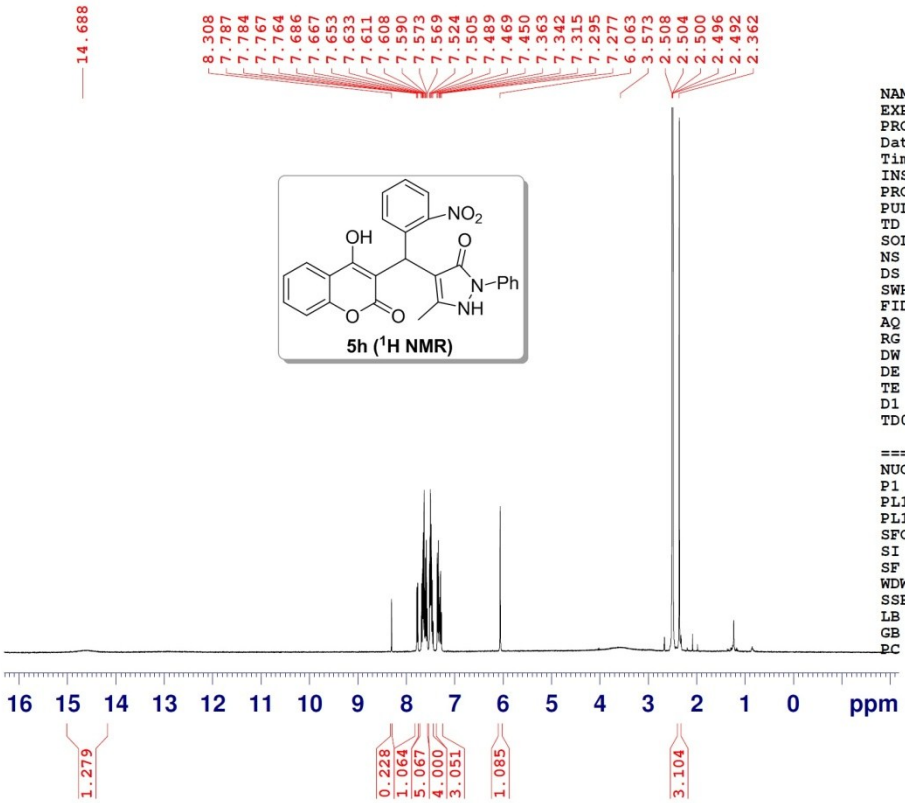



```
NAME                TB-3
EXPNO                  1
PROCNO                 1
Date_           20180306
Time               18.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    32
DS                     0
SWH            12019.230 Hz
FIDRES          0.487200 Hz
AQ             1.0263220 sec
RG                   114
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300121 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

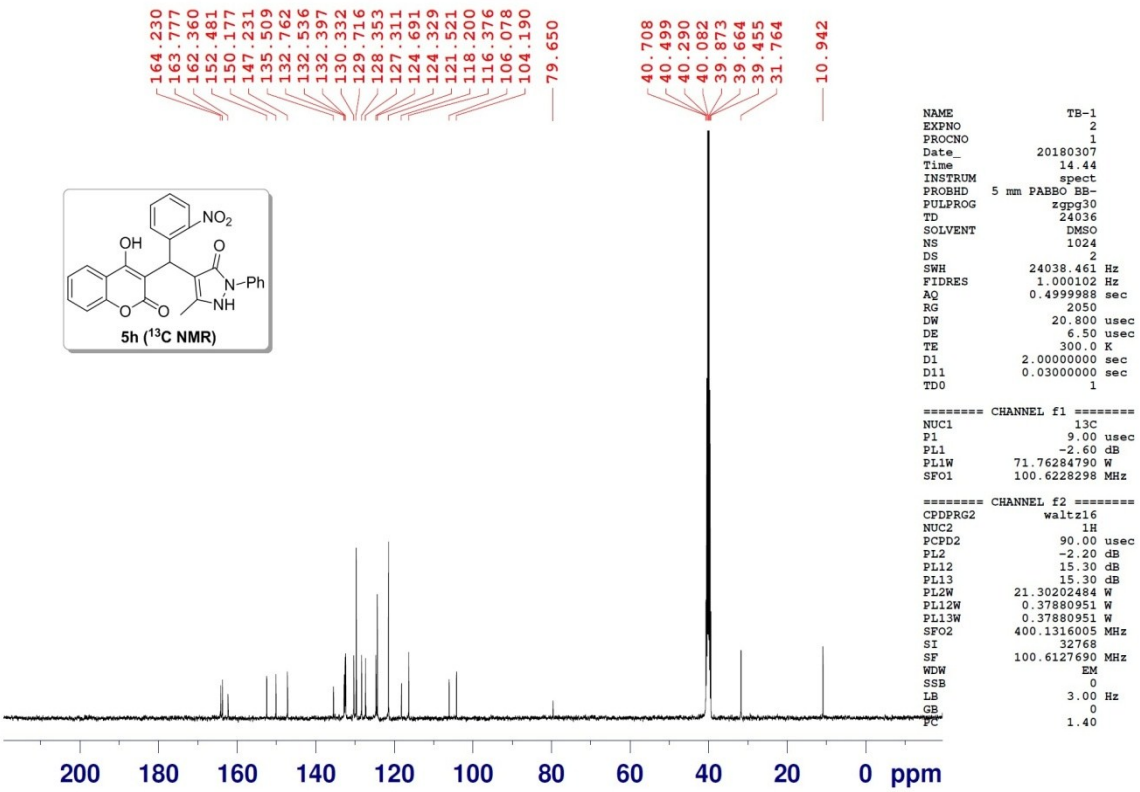

163.616
163.251
162.510
151.980
146.679
137.751
135.293
132.697
131.898
130.184
129.898
129.304
128.279
126.738
123.871
121.042
118.223
115.909
107.011
104.065
48.740
40.319
40.110
39.902
39.693
39.485
39.276
39.068
33.458
10.699
Cl
OH
O
N–Ph
NH
O
O
5g (13C NMR)
NAME TB-3
EXPNO 2
PROCNO 1
Date_ 20180306
Time 18.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 24036
SOLVENT DMSO
NS 12288
DS 4
SWH 24038.461 Hz
FIDRES 1.000102 Hz
AQ 0.4999988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128087 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

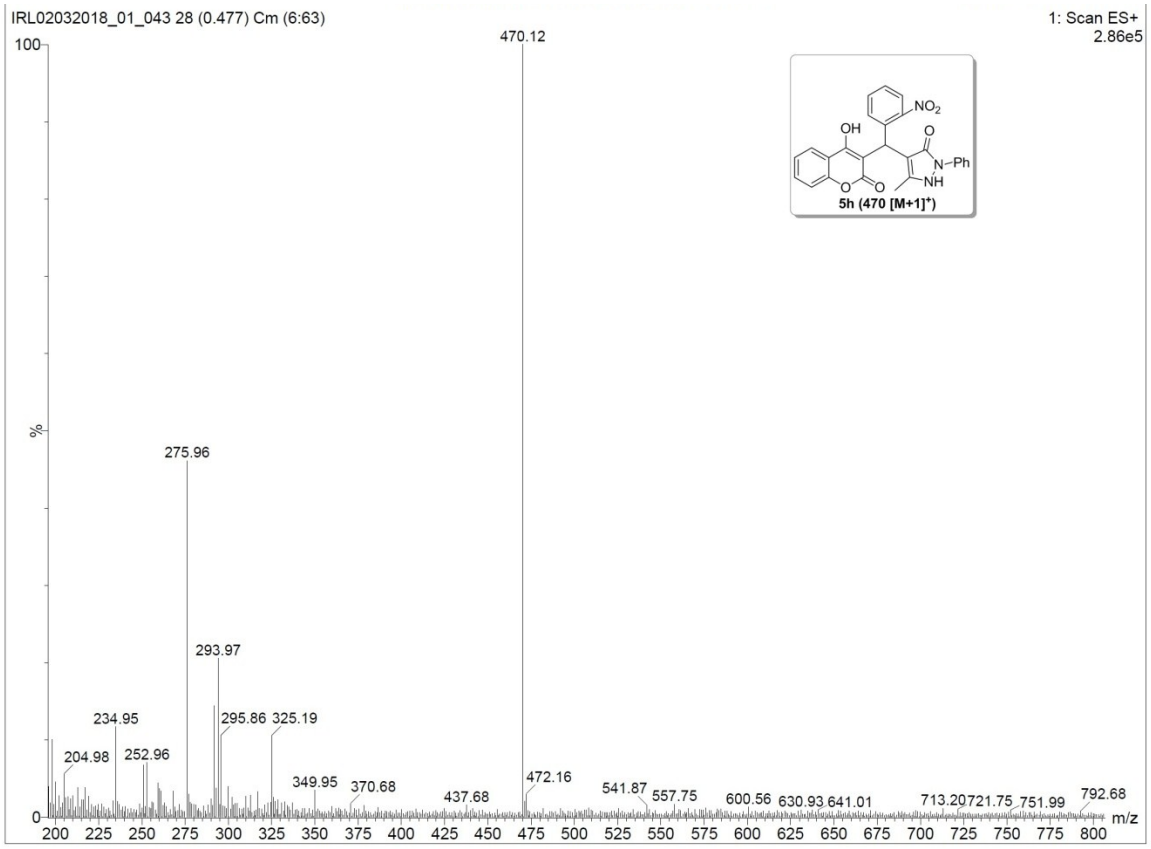

IRL02032018_01_035 29 (0.494) Cm (14:66)
1: Scan ES+
5.09e5
100
%
0
459.05
461.00
462.05
275.99
293.95
171.51
181.96
234.83
325.17
332.83
373.88
334.67
428.40
481.12
535.01
557.75
576.22
651.90
662.91
709.12
746.27
812.54
834.54
894.26
Cl
OH
O
N–Ph
NH
O
O
5h (459 [M+1]+)
200 250 300 350 400 450 500 550 600 650 700 750 800 850
m/z

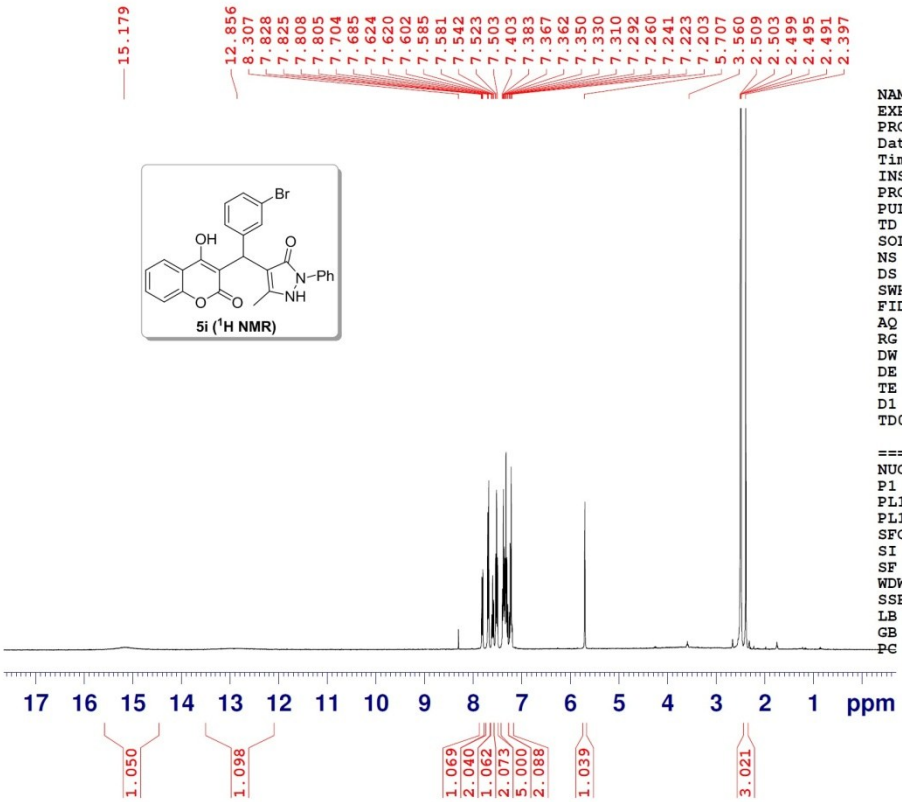



```
NAME                TB-1
EXPNO                  1
PROCNO                 1
Date_           20180306
Time               17.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    16
DS                     0
SWH            8223.685 Hz
FIDRES         0.333348 Hz
AQ            1.4999861 sec
RG                   287
DW                60.800 usec
DE                  6.50 usec
TE                 300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300080 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

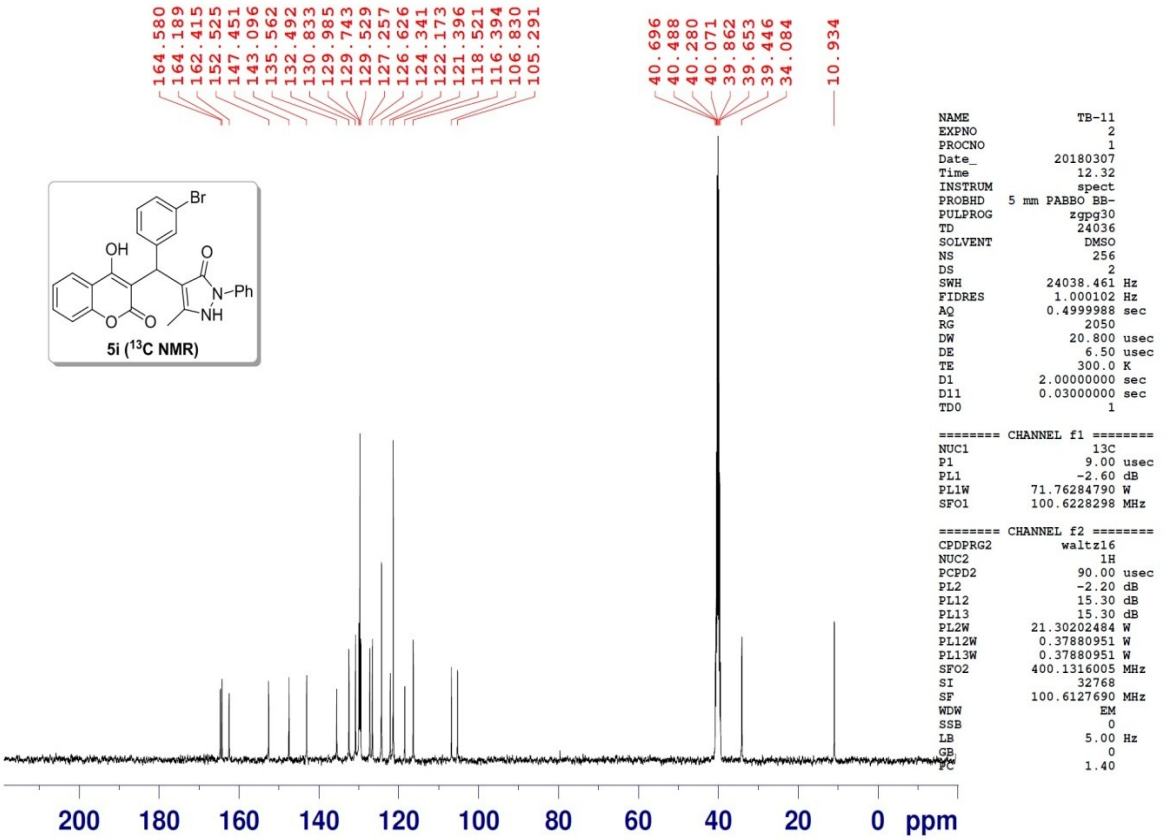

164.230
163.777
162.360
152.481
150.177
147.231
135.509
132.762
132.536
132.397
130.332
129.716
128.353
127.311
124.691
124.329
121.521
118.200
116.376
106.078
104.190
79.650
40.708
40.499
40.290
40.082
39.873
39.664
39.455
31.764
10.942
NO2
OH
O
N–Ph
NH
O
O
5h (13C NMR)
NAME TB-1
EXPNO 2
PROCNO 1
Date_ 20180307
Time 14.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 24036
SOLVENT DMSO
NS 1024
DS 2
SWH 24038.461 Hz
FIDRES 1.000102 Hz
AQ 0.4999988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

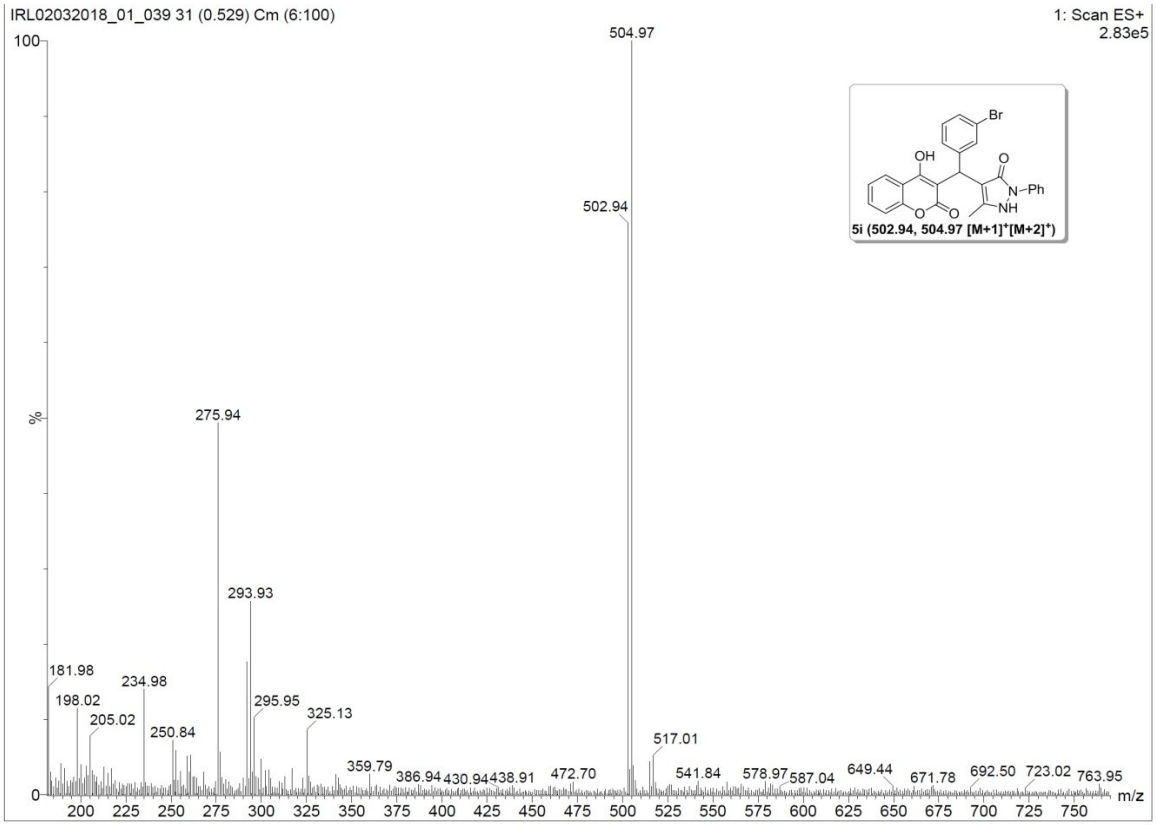

IRL02032018_01_043 28 (0.477) Cm (6:63)
1: Scan ES+
2.86e5
100
%
0
470.12
275.96
293.97
234.95
295.86
325.19
204.98
252.96
349.95
370.68
437.68
472.16
541.87
557.75
600.56
630.93
641.01
713.20
721.75
751.99
792.68
NO2
OH
O
N–Ph
NH
O
O
5h (470 [M+1]+)
200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800
m/z

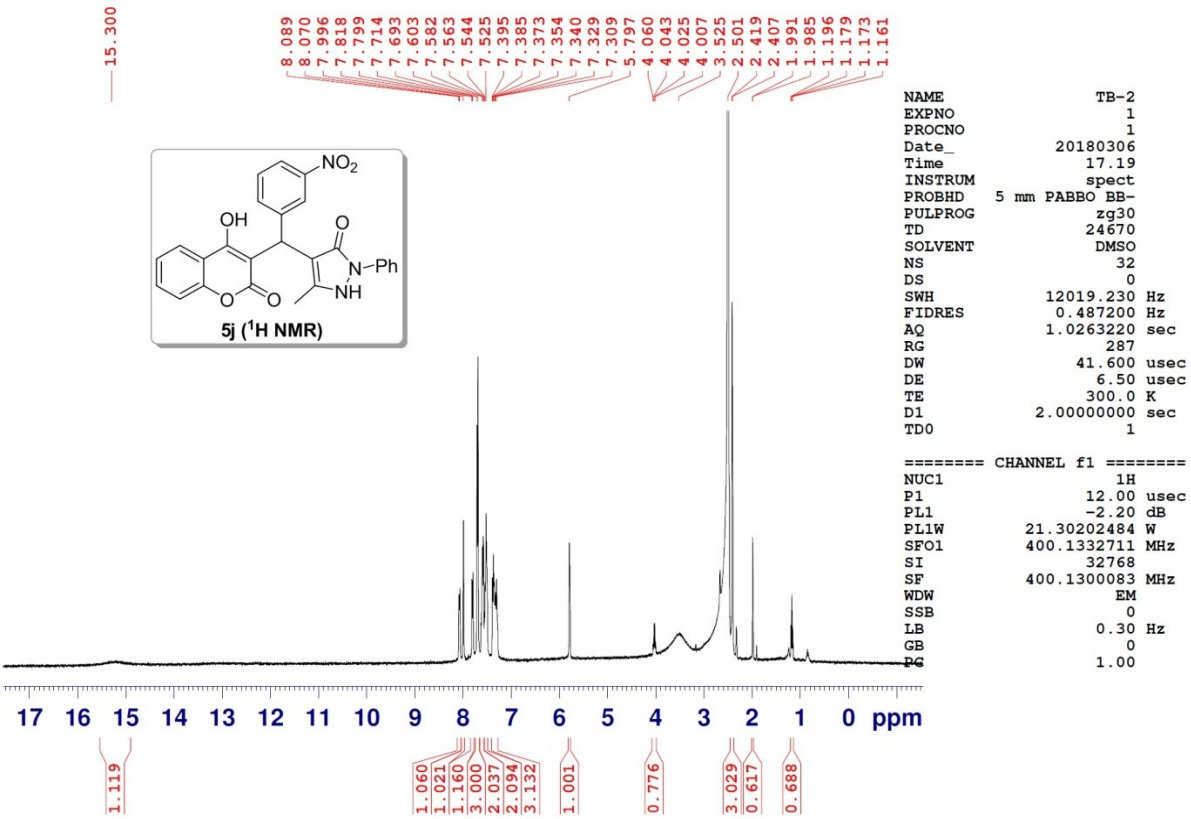



```
NAME                TB-11
EXPNO                   1
PROCNO                  1
Date_            20180306
Time                17.50
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  24670
SOLVENT              DMSO
NS                     16
DS                      0
SWH             12019.230 Hz
FIDRES           0.487200 Hz
AQ              1.0263220 sec
RG                    287
DW                 41.600 usec
DE                  6.50 usec
TE                 300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -2.20 dB
PL1W         21.30202484 W
SFO1         400.1332711 MHz
SI                  32768
SF           400.1300082 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

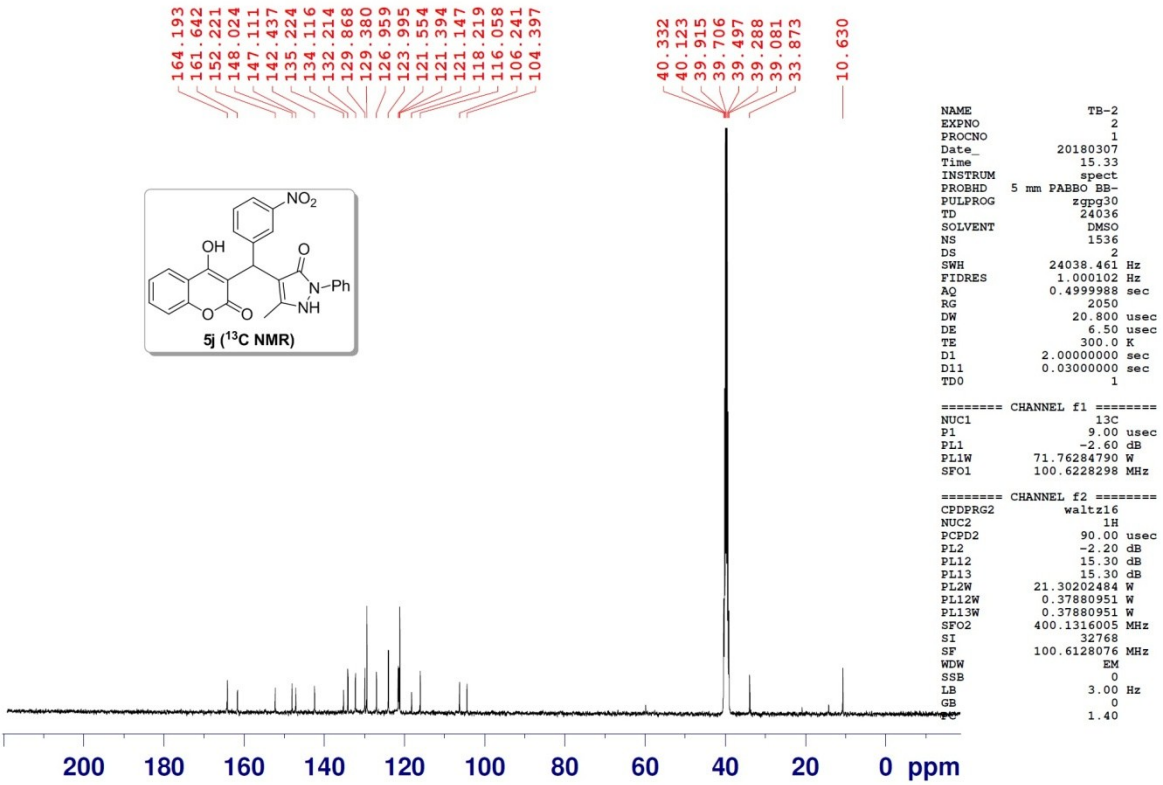

164.580
164.189
162.415
152.525
147.451
143.096
135.562
132.492
130.833
129.985
129.743
129.529
127.257
126.626
124.341
122.173
121.396
118.521
116.394
106.830
105.291
40.696
40.488
40.280
40.071
39.862
39.653
39.446
34.084
10.934
Br
OH
O
N-Ph
NH
O
O
5i (13C NMR)
NAME TB-11
EXPNO 2
PROCNO 1
Date_ 20180307
Time 12.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 24036
SOLVENT DMSO
NS 256
DS 2
SWH 24038.461 Hz
FIDRES 1.000102 Hz
AQ 0.4999988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

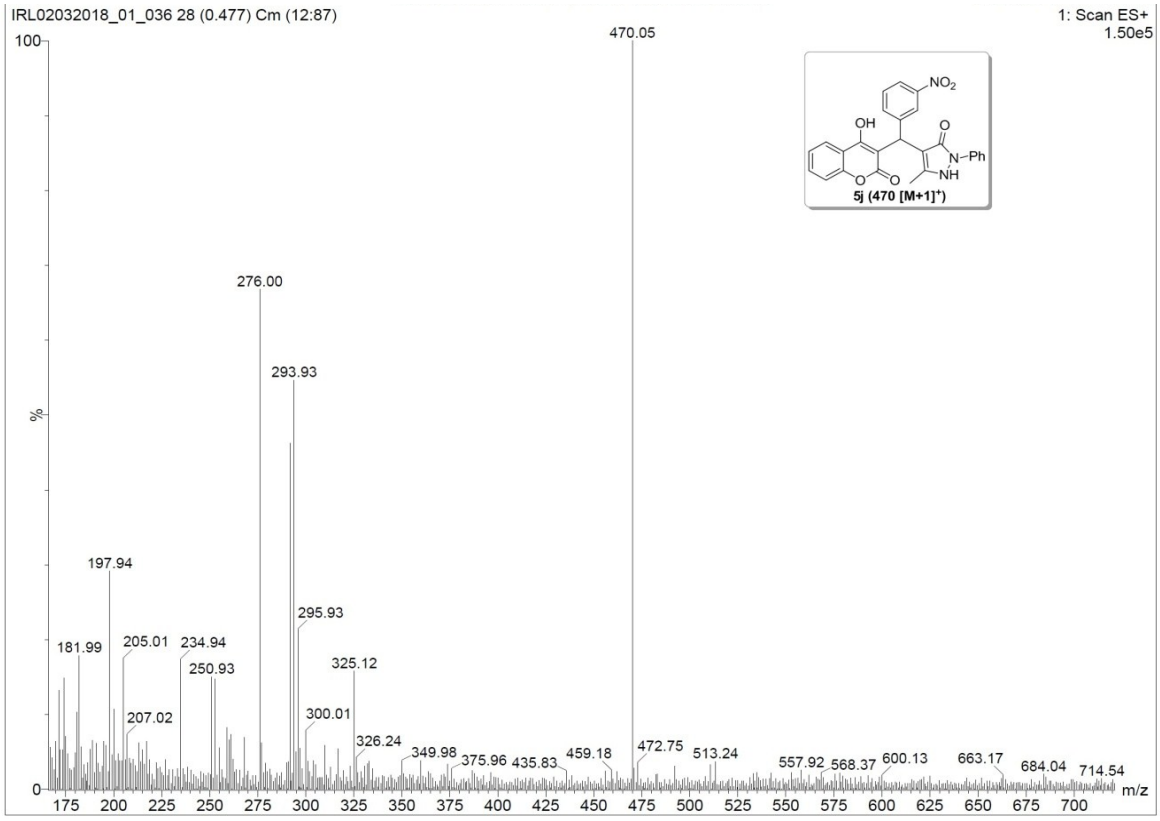

IRL02032018_01_039 31 (0.529) Cm (6:100)
1: Scan ES+
2.83e5
100
%
0
504.97
502.94
275.94
293.93
181.98
198.02
205.02
234.98
250.84
295.95
325.13
359.79
386.94
430.94
438.91
472.70
517.01
541.84
578.97
587.04
649.44
671.78
692.50
723.02
763.95
Br
OH
O
N-Ph
NH
O
O
5i (502.94, 504.97 [M+1]+[M+2]+)
200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750
m/z




```
NAME                TB-2
EXPNO                  1
PROCNO                 1
Date_           20180306
Time               17.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 24670
SOLVENT             DMSO
NS                    32
DS                     0
SWH            12019.230 Hz
FIDRES          0.487200 Hz
AQ            1.0263220 sec
RG                   287
DW                41.600 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.20 dB
PL1W        21.30202484 W
SFO1        400.1332711 MHz
SI                32768
SF          400.1300083 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


164.193
161.642
152.221
148.024
147.111
142.437
135.224
134.116
132.214
129.868
129.380
126.959
123.995
121.554
121.394
121.147
118.219
116.058
106.241
104.397
40.332
40.123
39.915
39.706
39.497
39.288
39.081
33.873
10.630
NO2
OH
O
N–Ph
NH
O
O
5j (13C NMR)
NAME TB-2
EXPNO 2
PROCNO 1
Date_ 20180307
Time 15.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 24036
SOLVENT DMSO
NS 1536
DS 2
SWH 24038.461 Hz
FIDRES 1.000102 Hz
AQ 0.4999988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.60 dB
PL1W 71.76284790 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.20 dB
PL12 15.30 dB
PL13 15.30 dB
PL2W 21.30202484 W
PL12W 0.37880951 W
PL13W 0.37880951 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128076 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


IRL02032018_01_036 28 (0.477) Cm (12:87)
1: Scan ES+
1.50e5
100
%
0
470.05
276.00
293.93
197.94
295.93
181.99
205.01
234.94
250.93
325.12
207.02
300.01
326.24
349.98
375.96
435.83
459.18
472.75
513.24
557.92
568.37
600.13
663.17
684.04
714.54
NO2
OH
O
N–Ph
NH
O
O
5j (470 [M+1]+)
175 200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700
m/z