Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

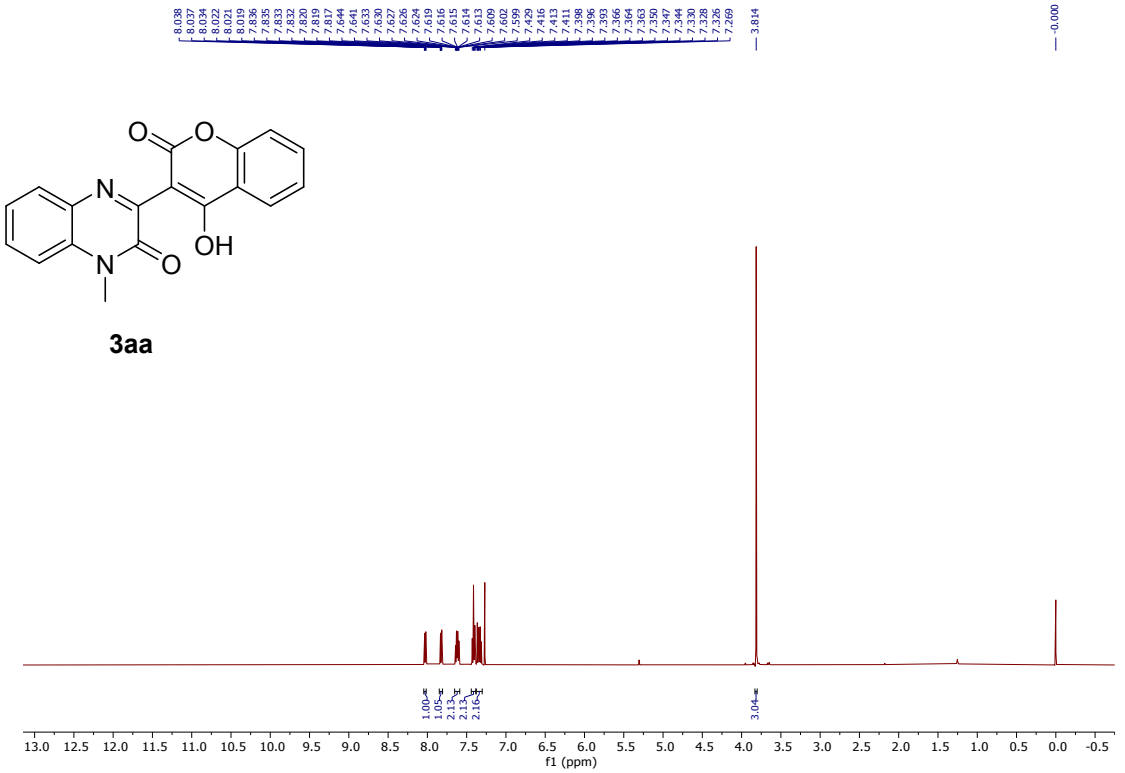
Supporting Information

tert-Butylhydroperoxide (TBHP) mediated oxidative cross-dehydrogenative coupling of quinoxaline-2(1H)-ones with 4-hydroxycoumarins, 4-hydroxy-6-methyl-2-pyrone and 2-hydroxy-1,4-naphthoquinone under metal-free conditions

Suraj Sharma,[a,b] Nibedita Baruah Dutta,[a,b,c] Mayurakhi Bhuyan,[a,b] Babulal Das[d] and Gakul Baishya[a,b]*

CONTENTS:

^{1}H-NMR (500 MHz, $CDCl_3$)



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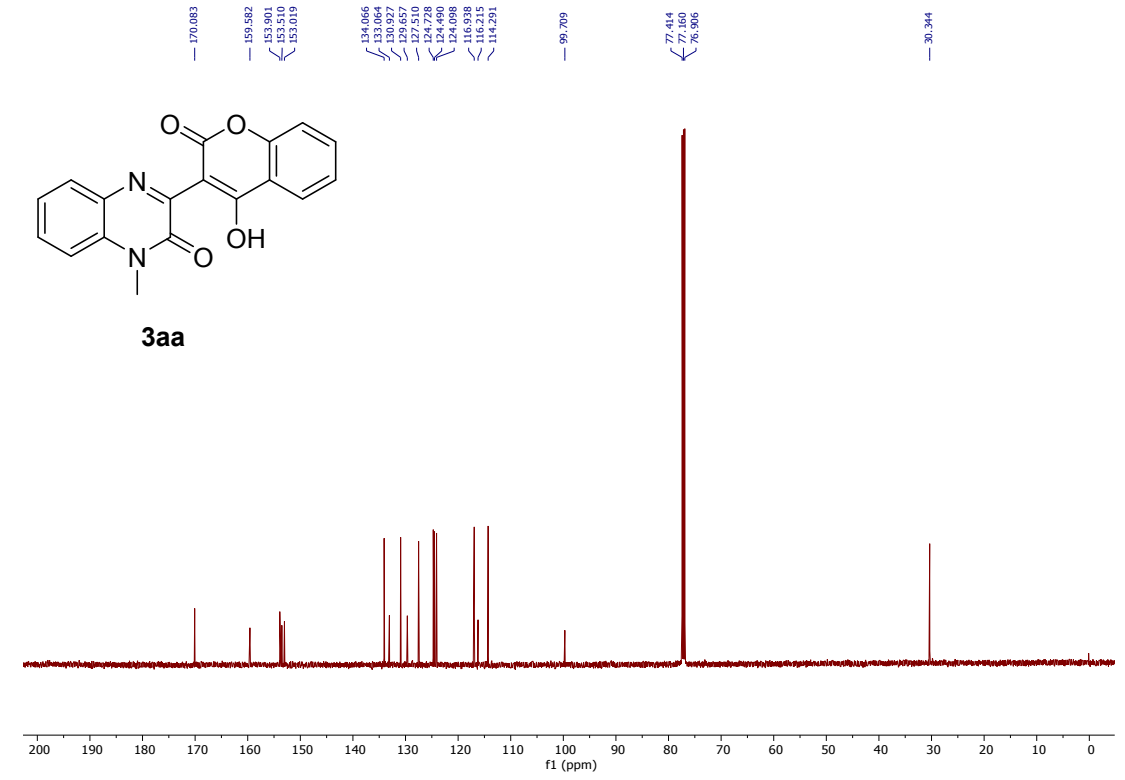
^{13}C-NMR (125 MHz, $CDCl_3$)



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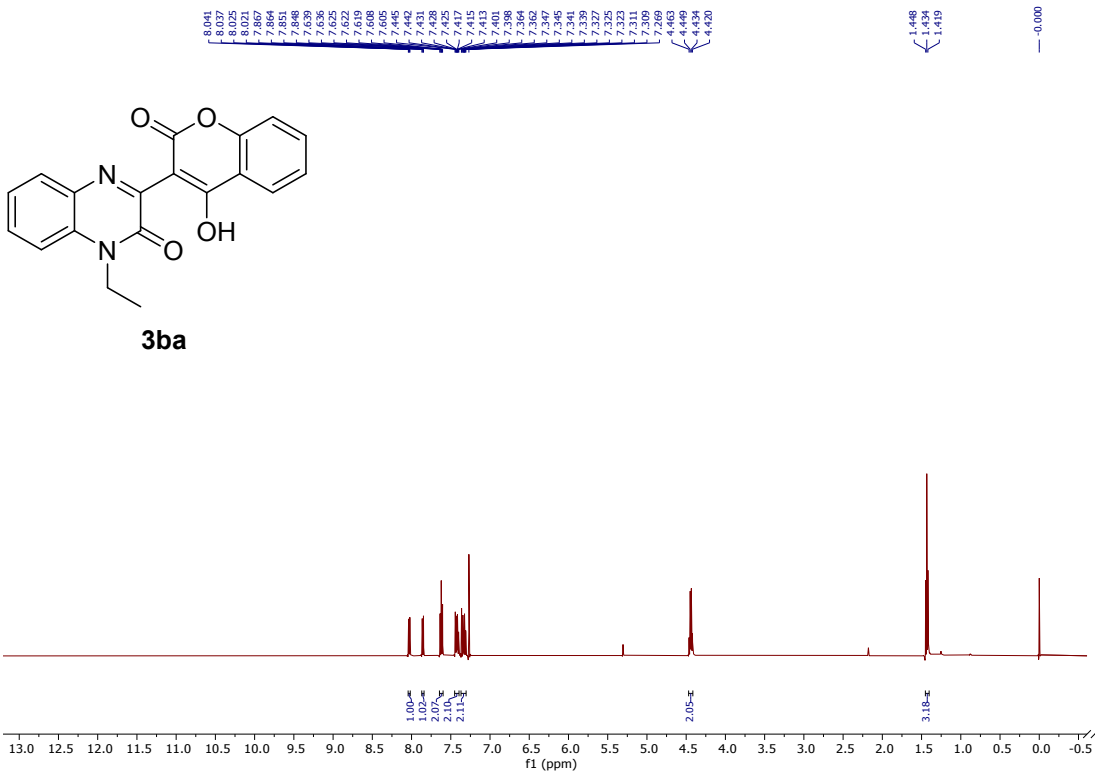

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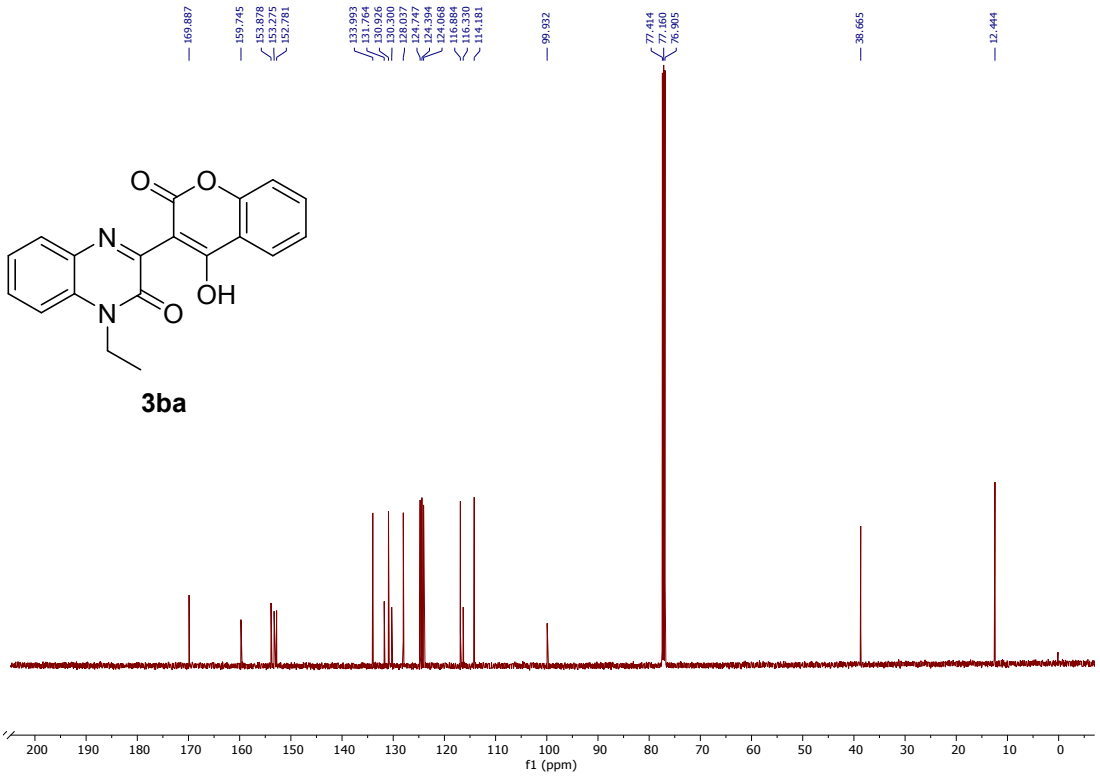

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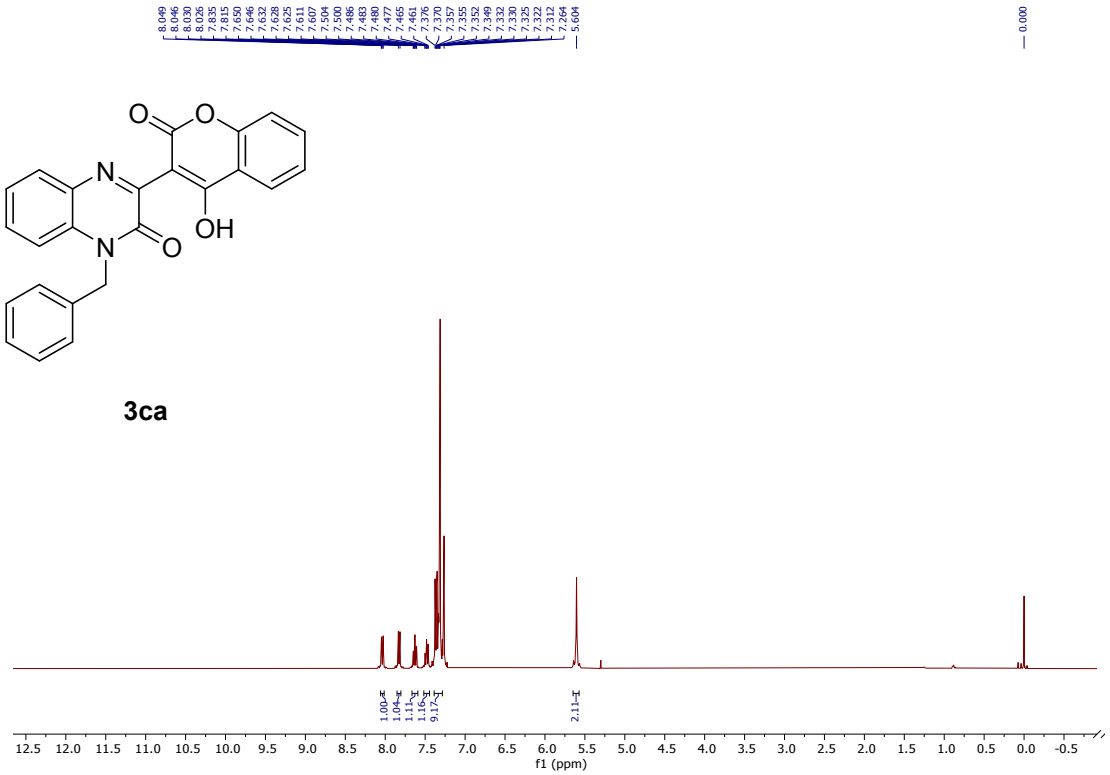

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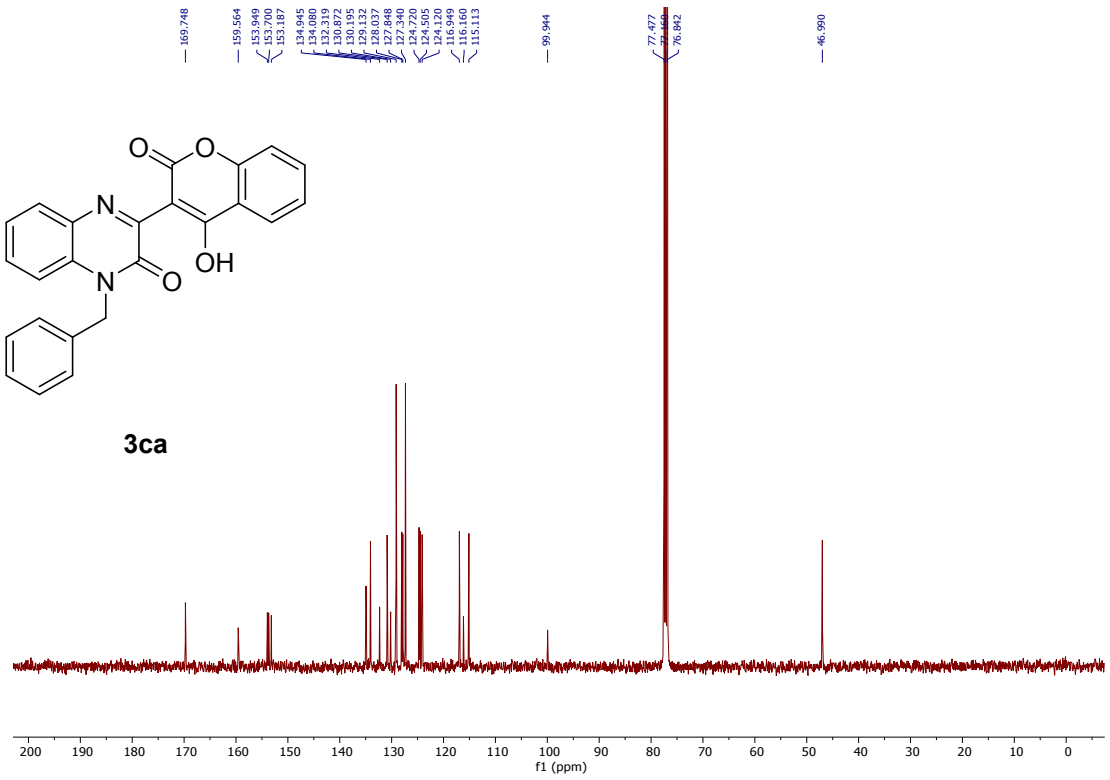

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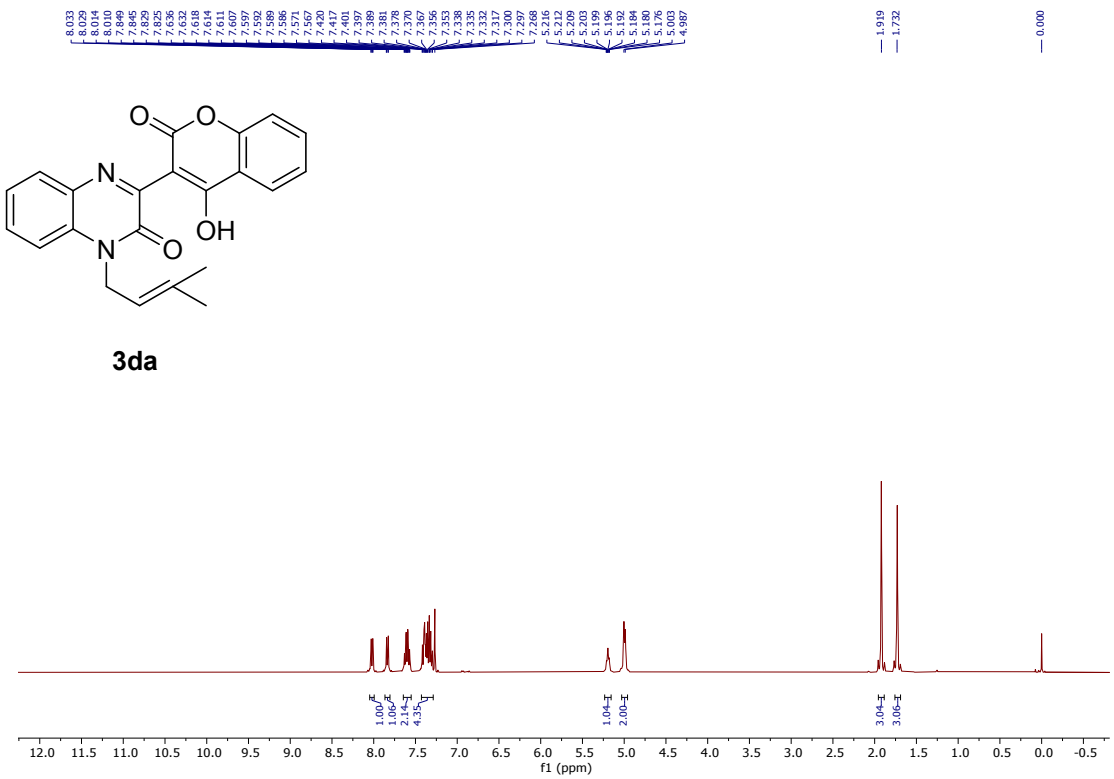

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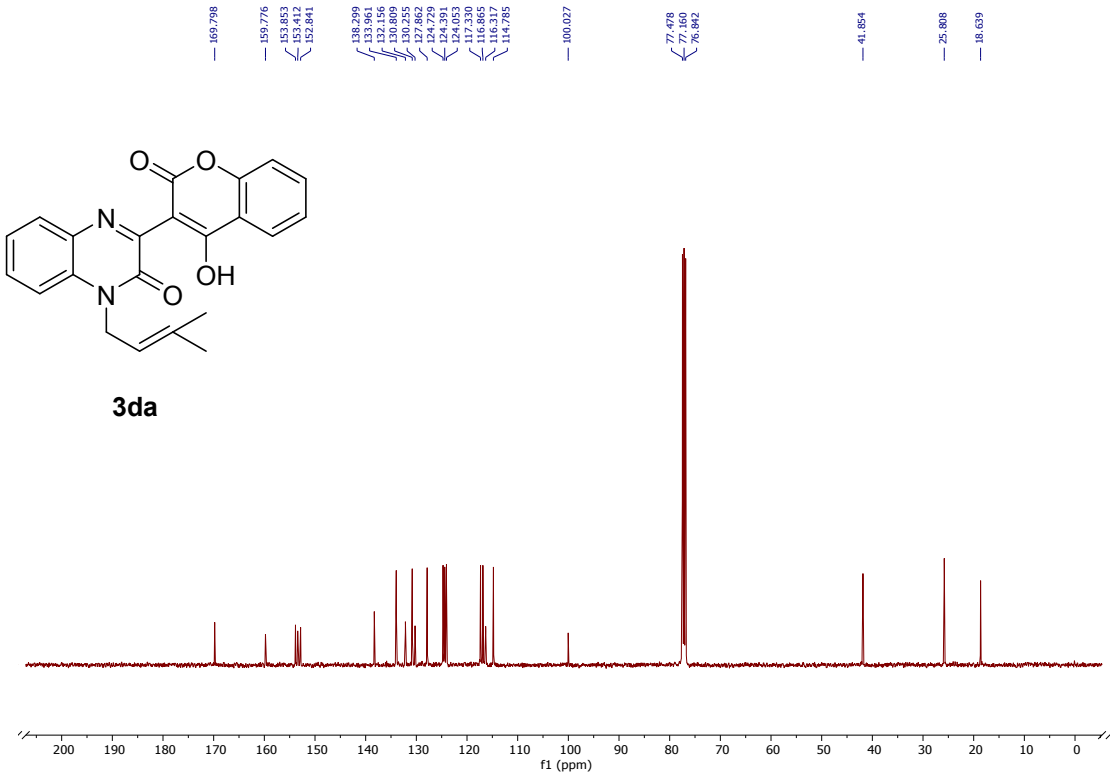

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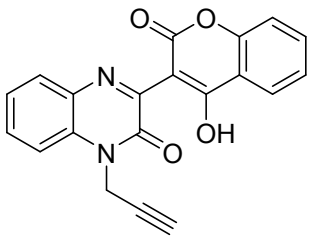

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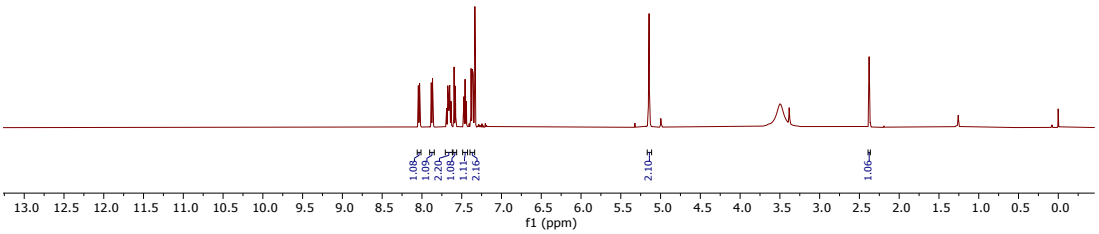

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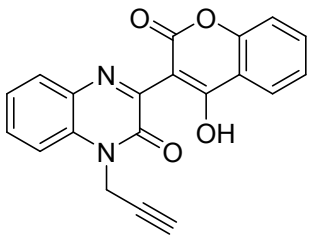

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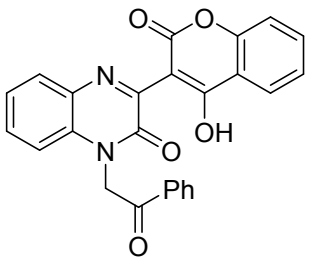

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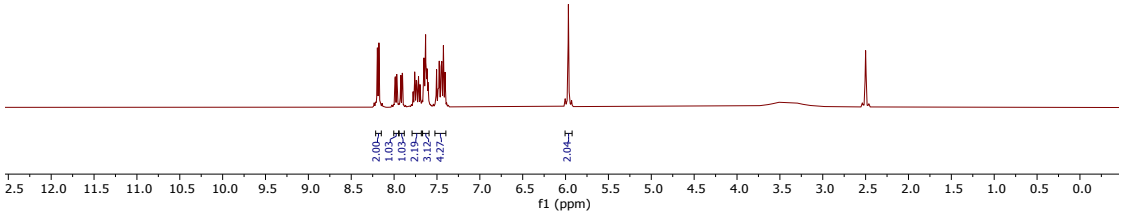

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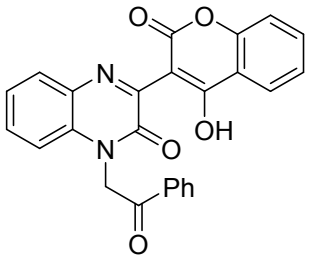

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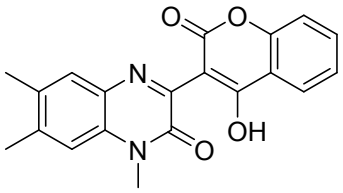

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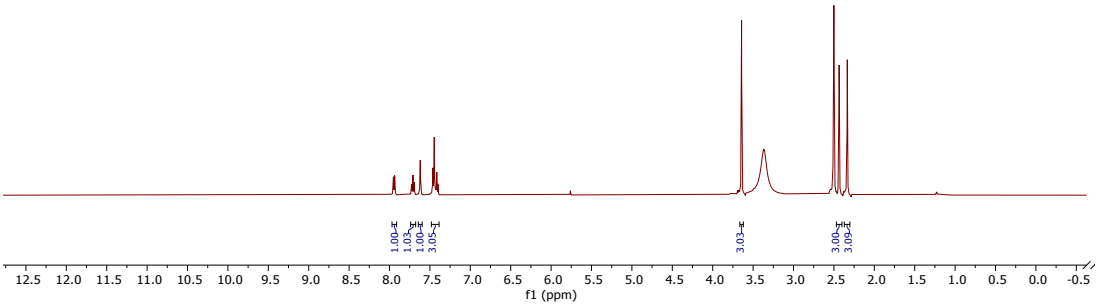

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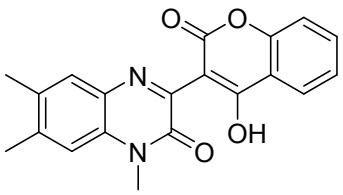

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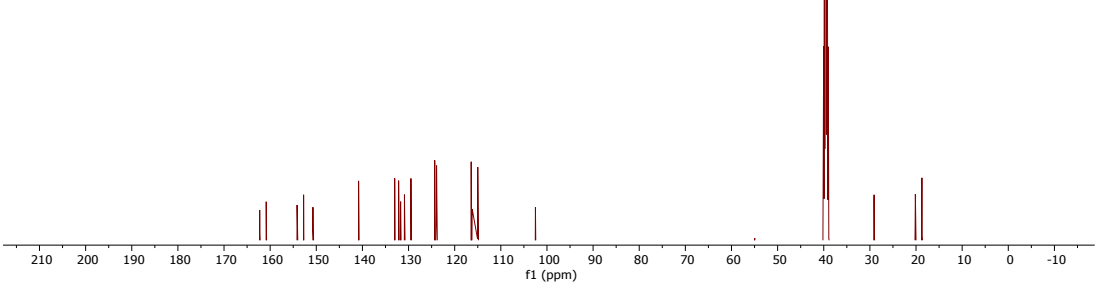

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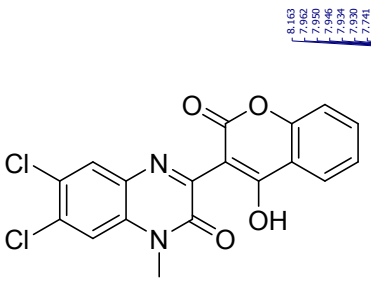

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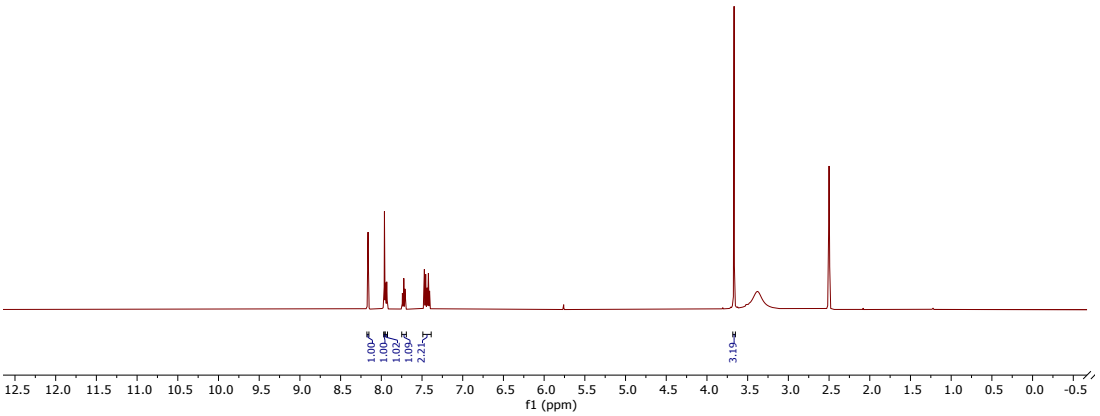

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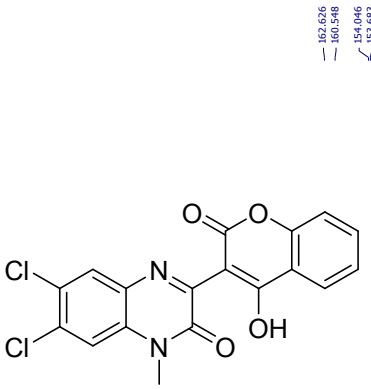

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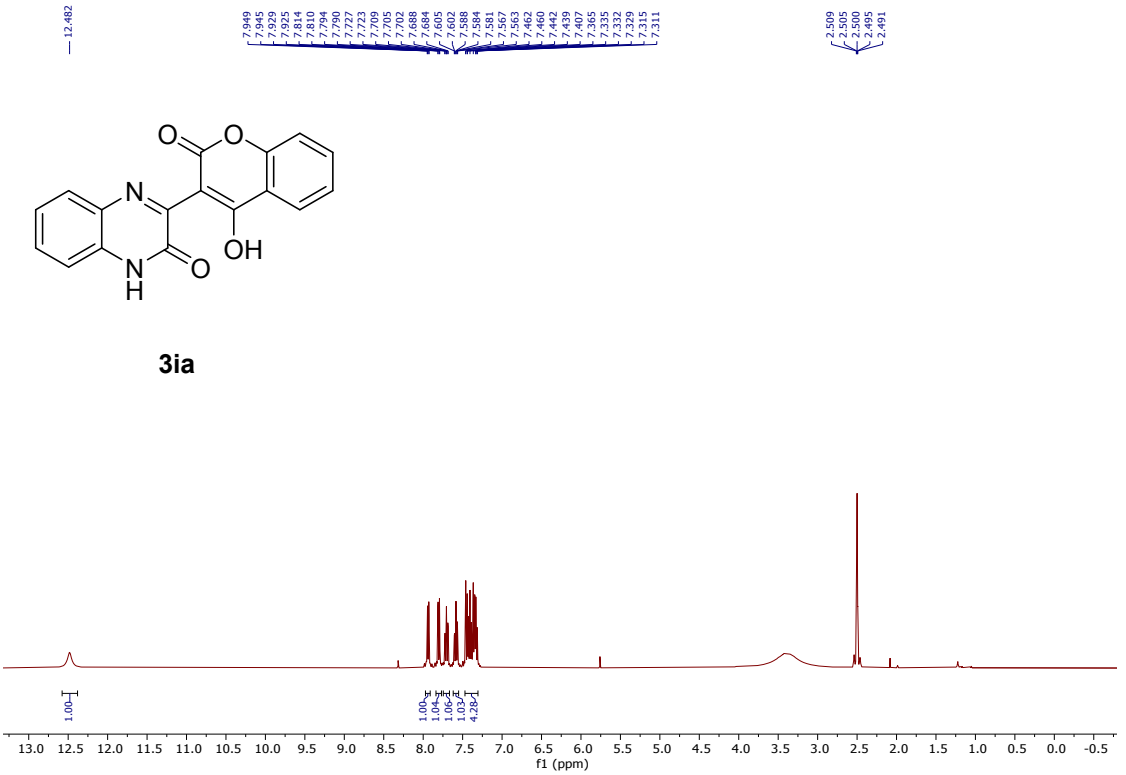

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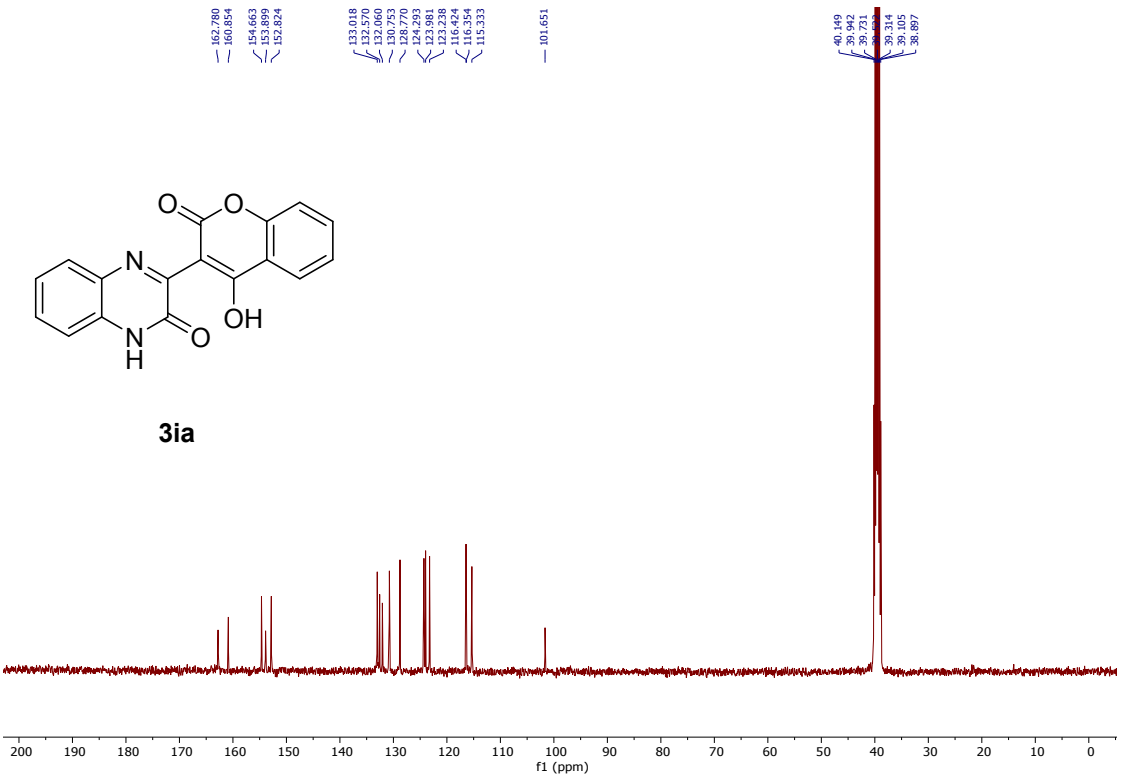

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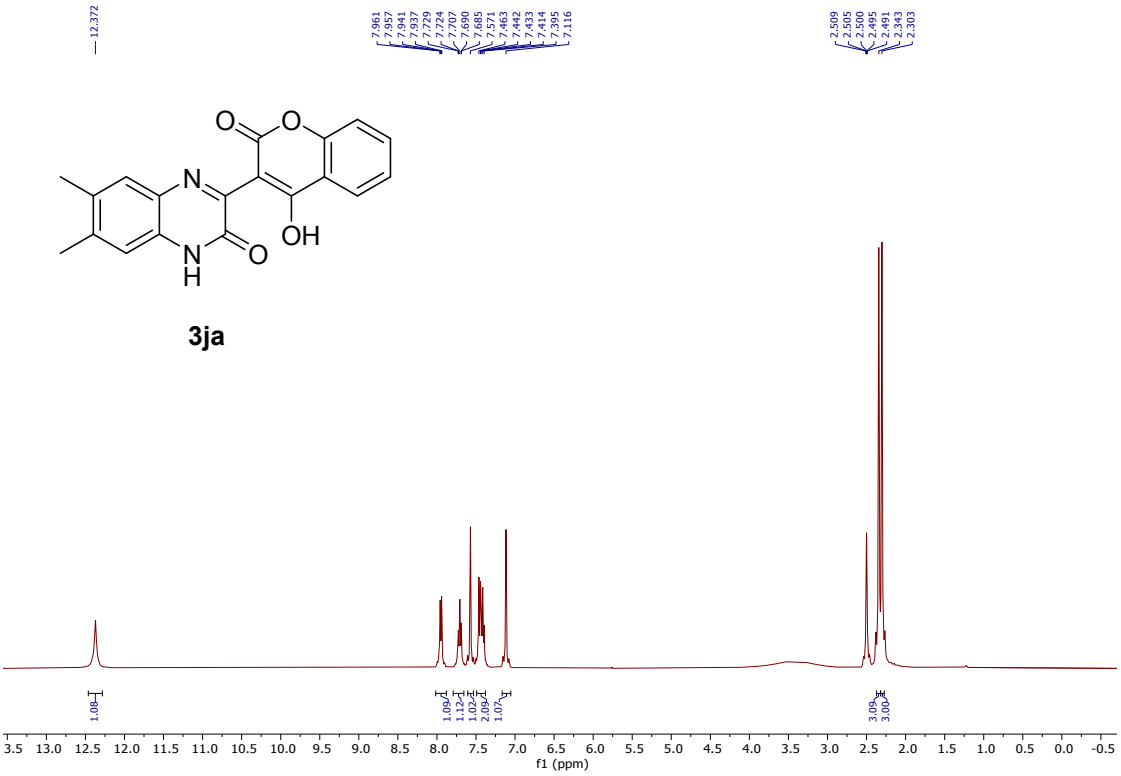

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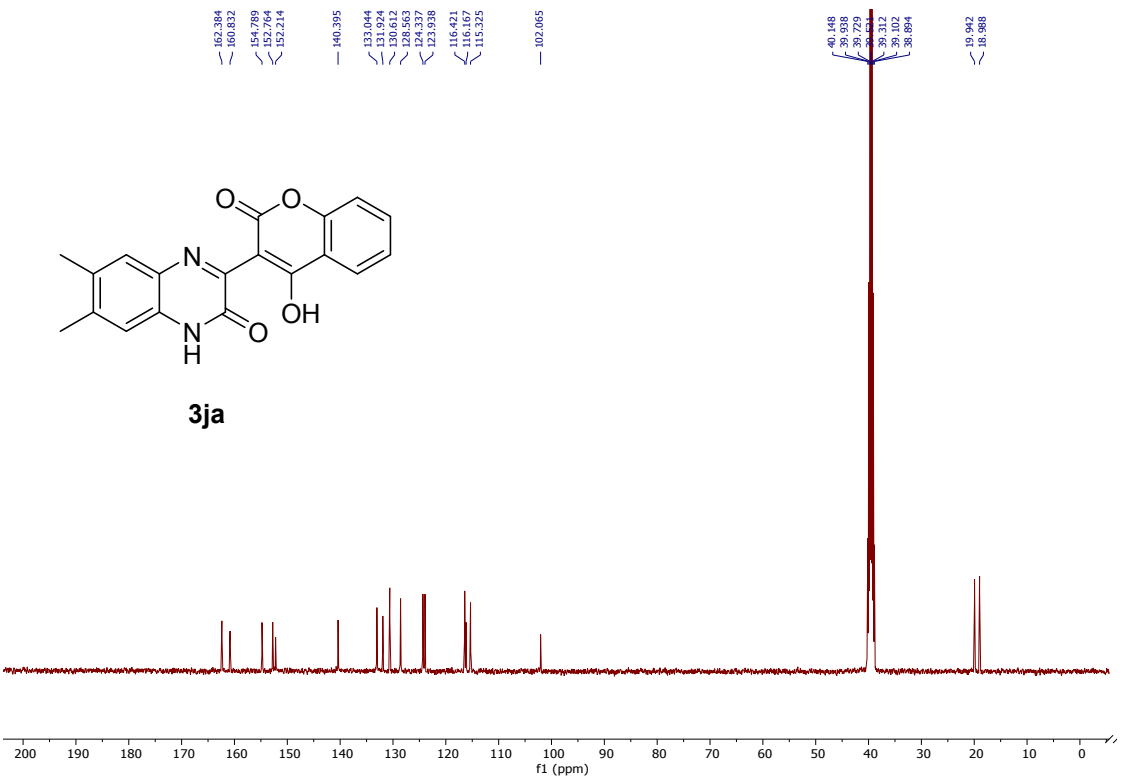

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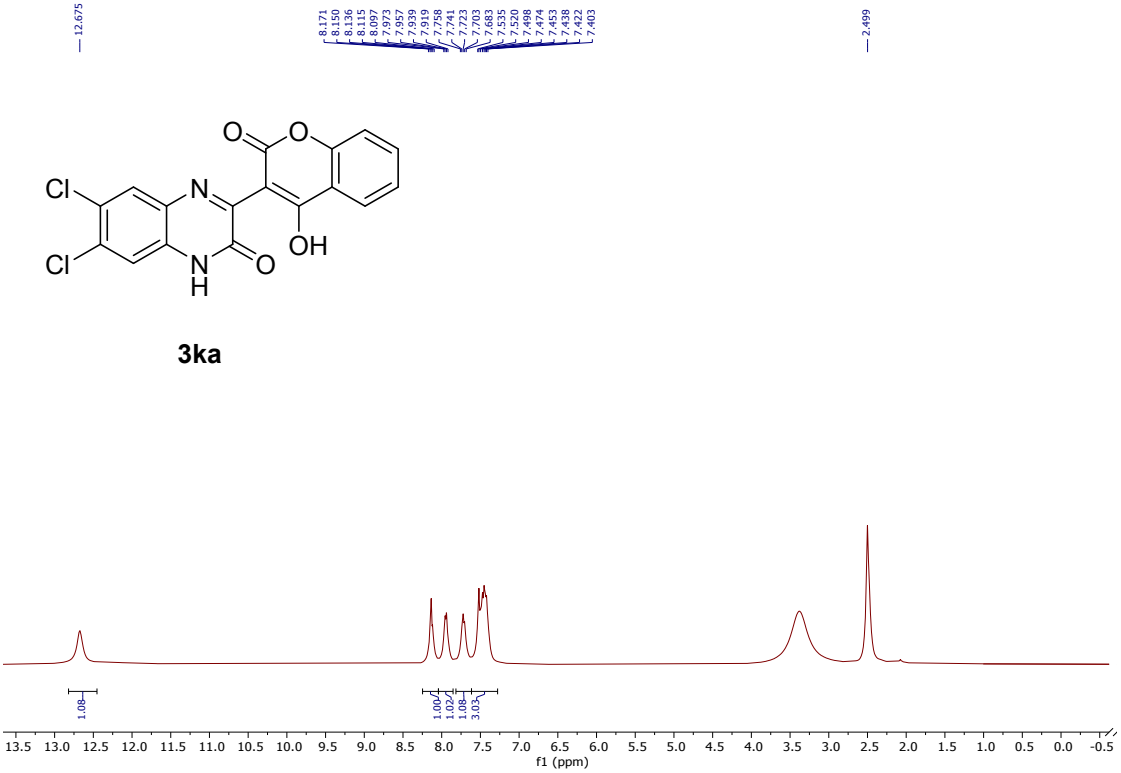

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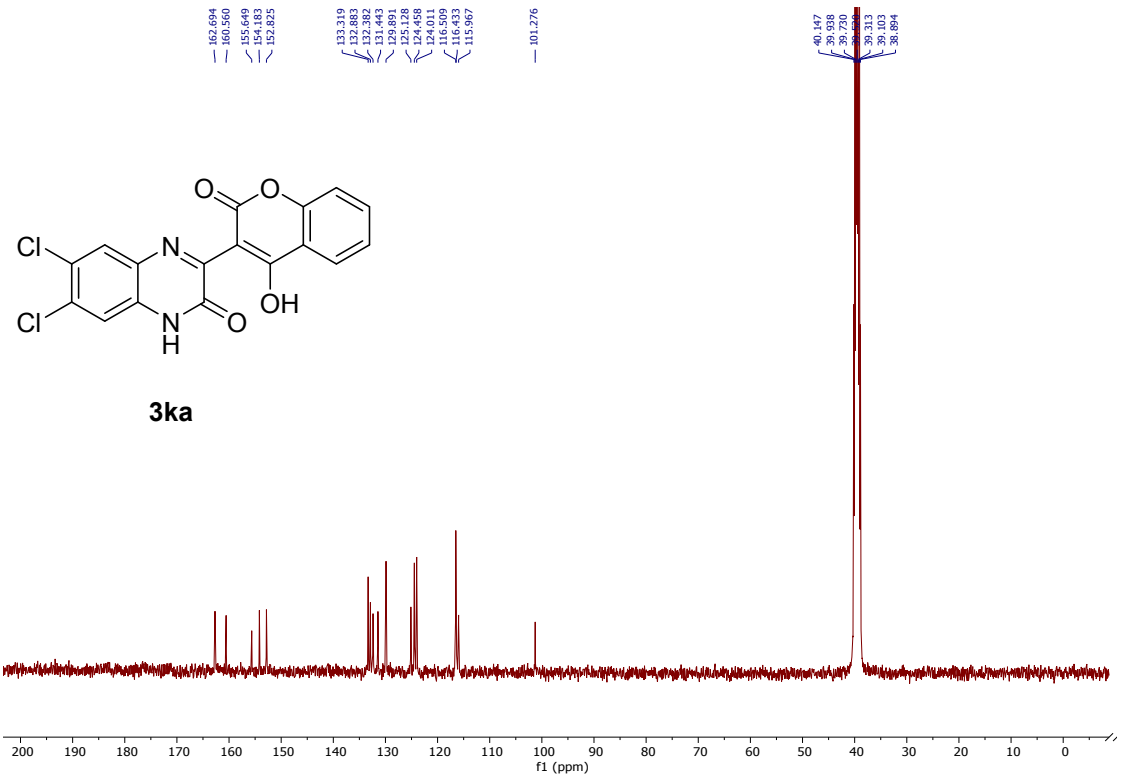

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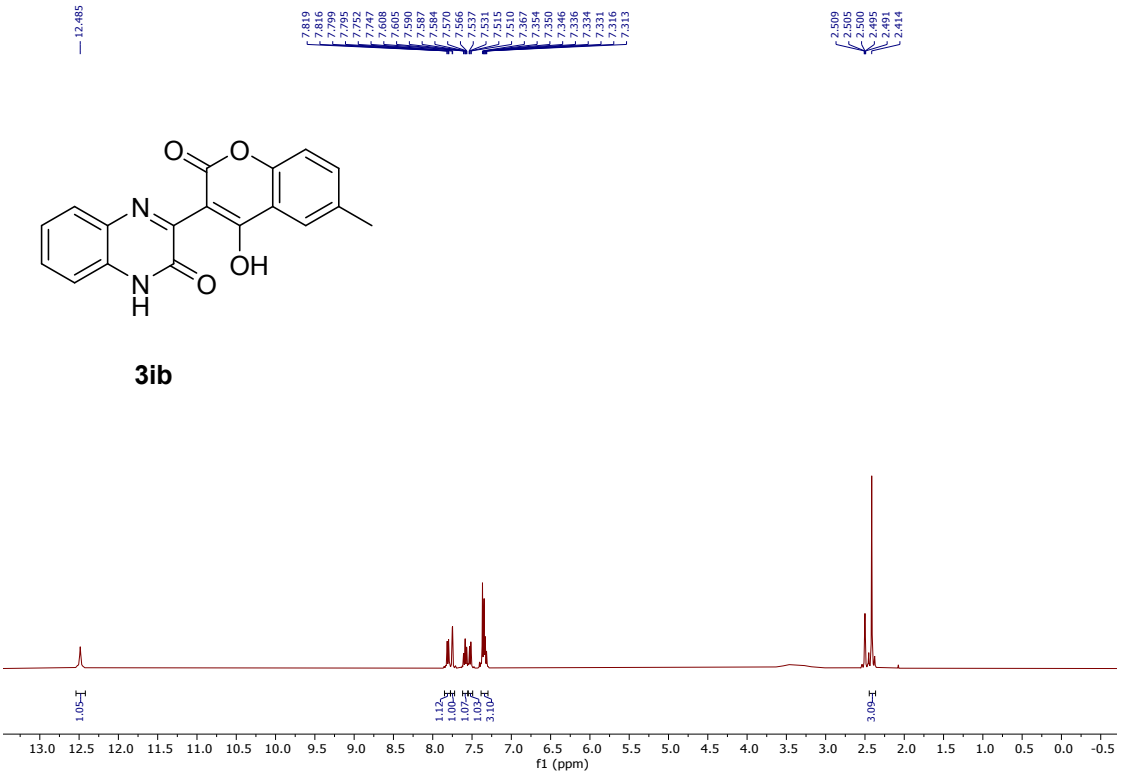

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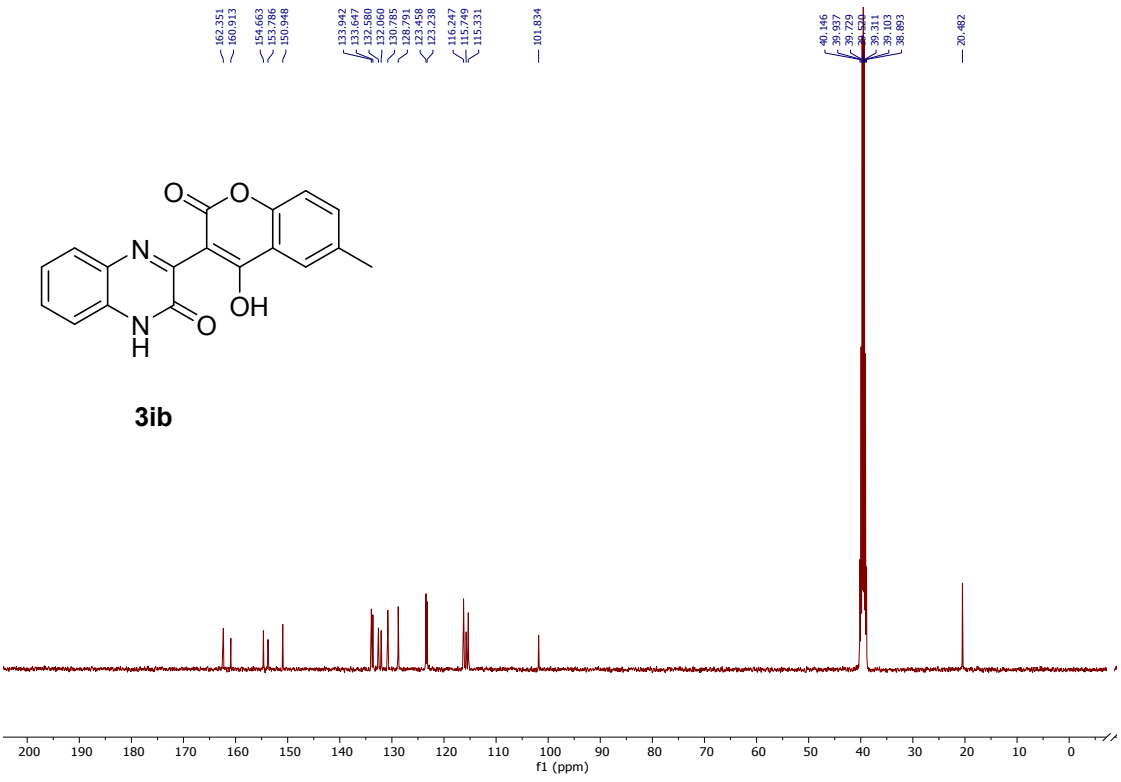

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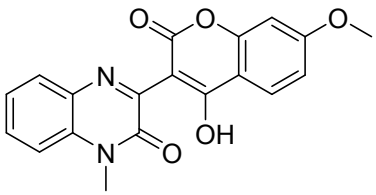
^{1}H-NMR (500 MHz, $CDCl_3$)



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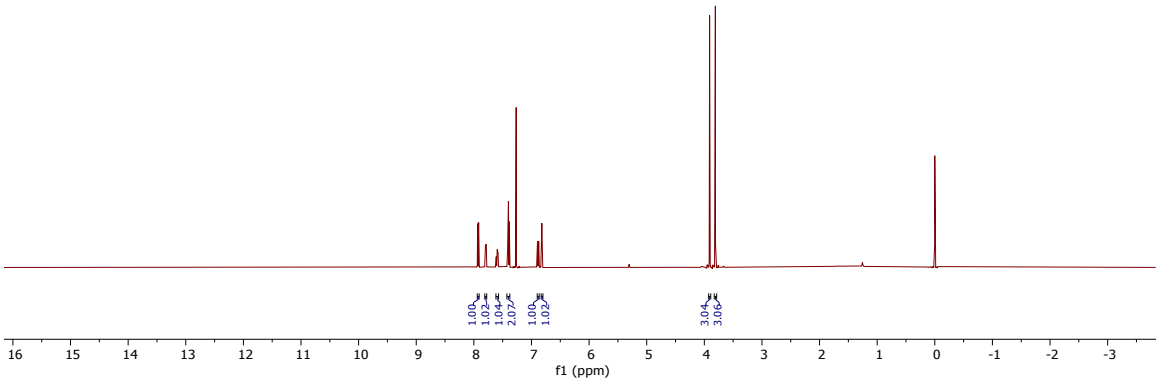
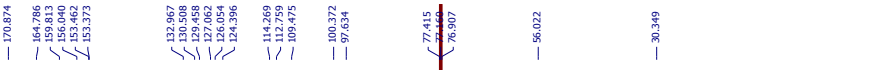
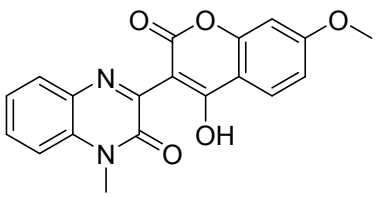


^{13}C-NMR (125 MHz, $CDCl_3$)





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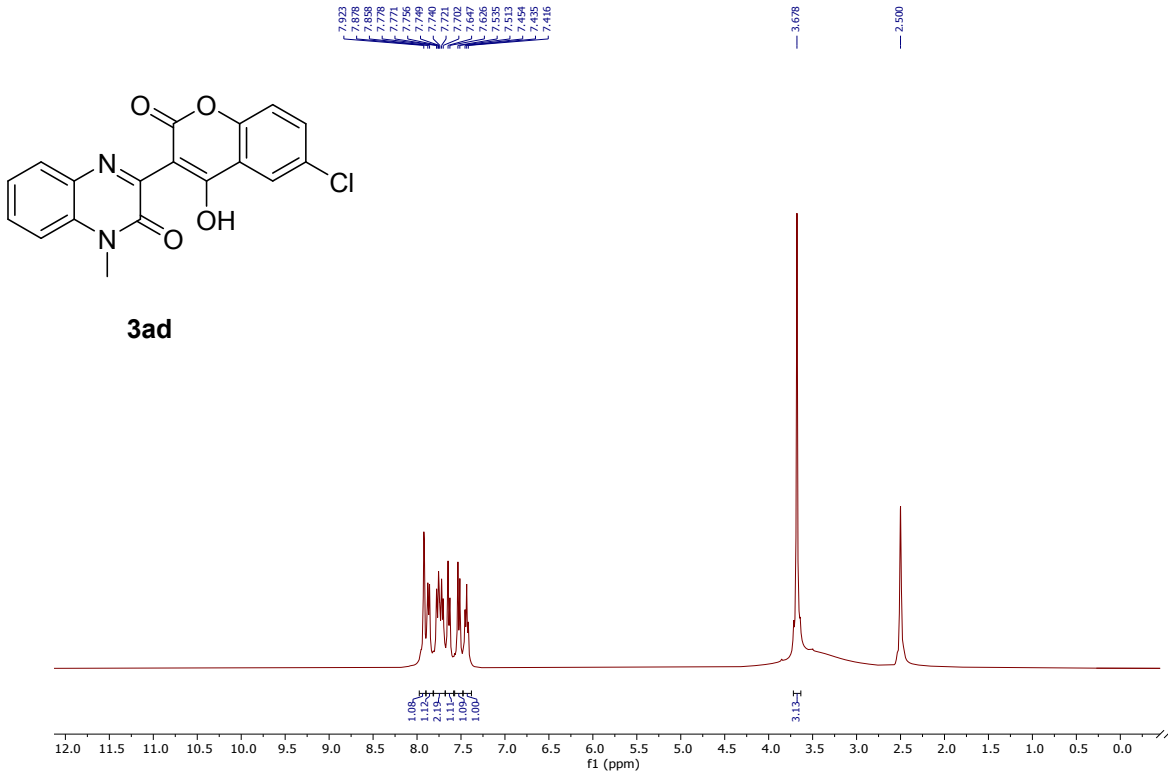

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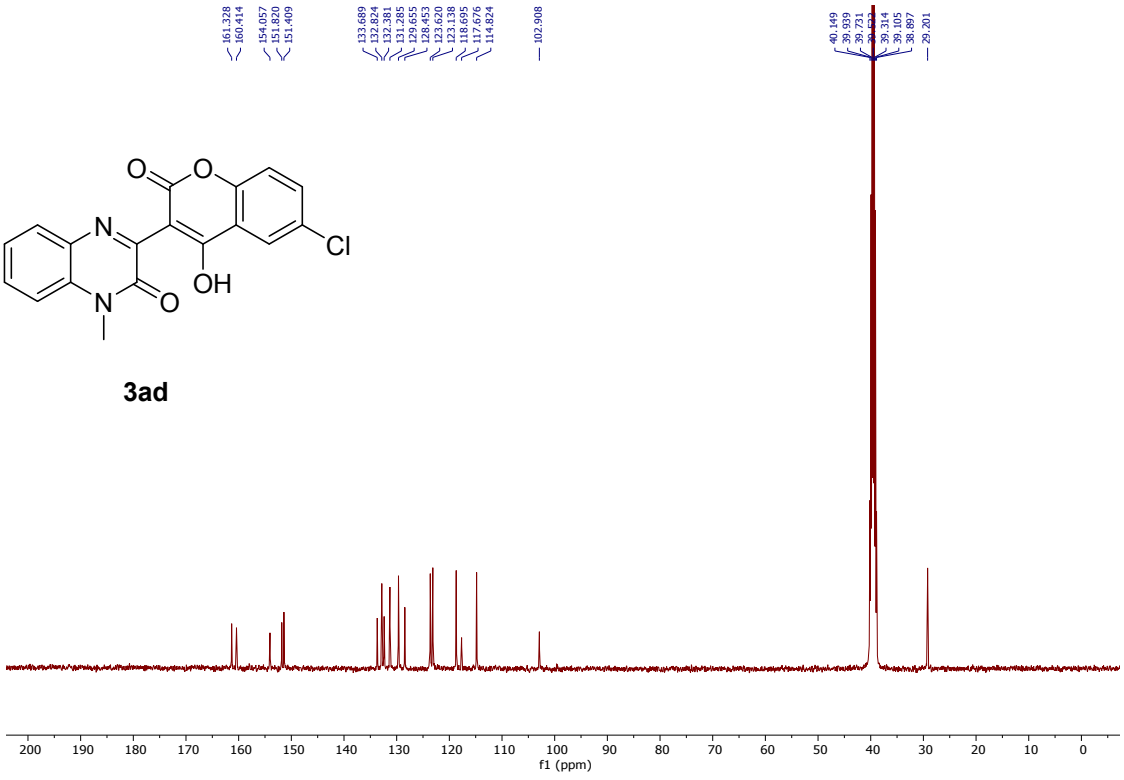

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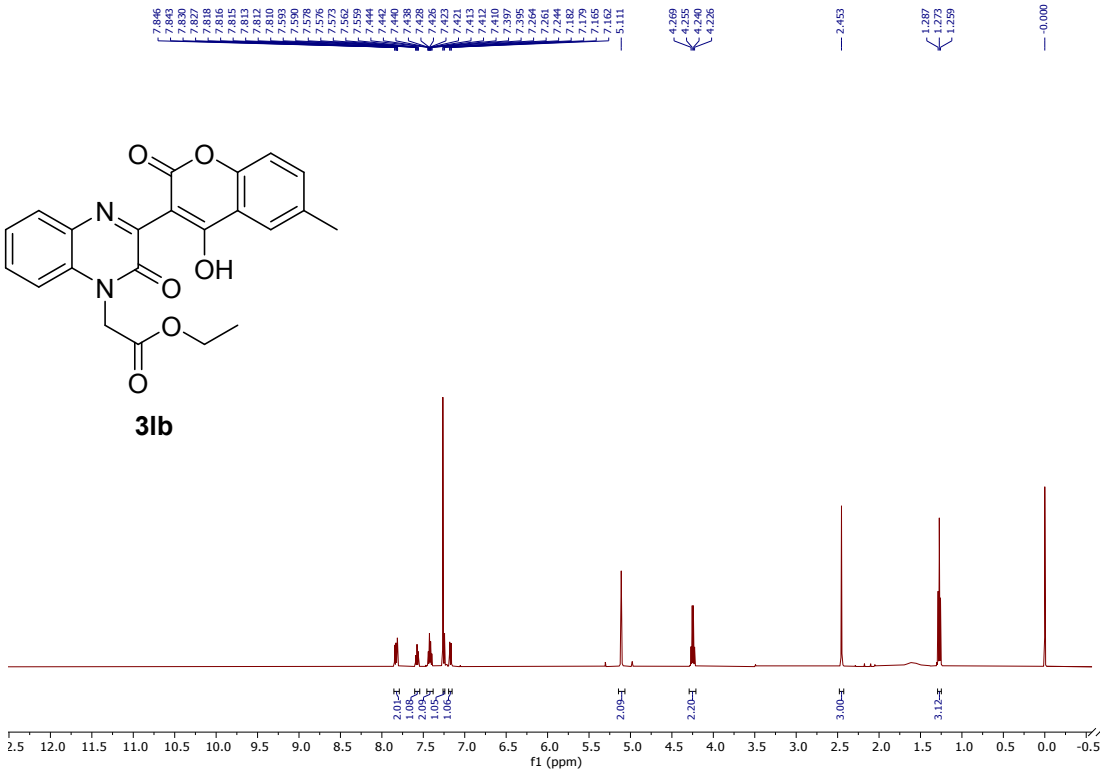

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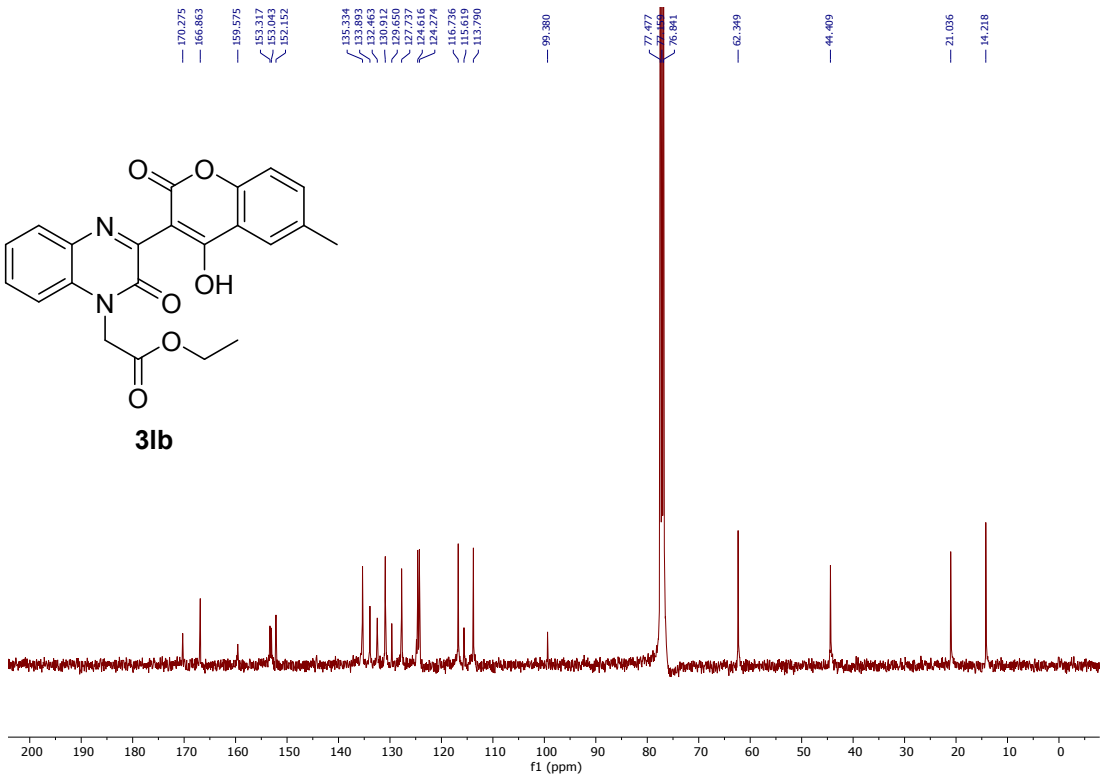

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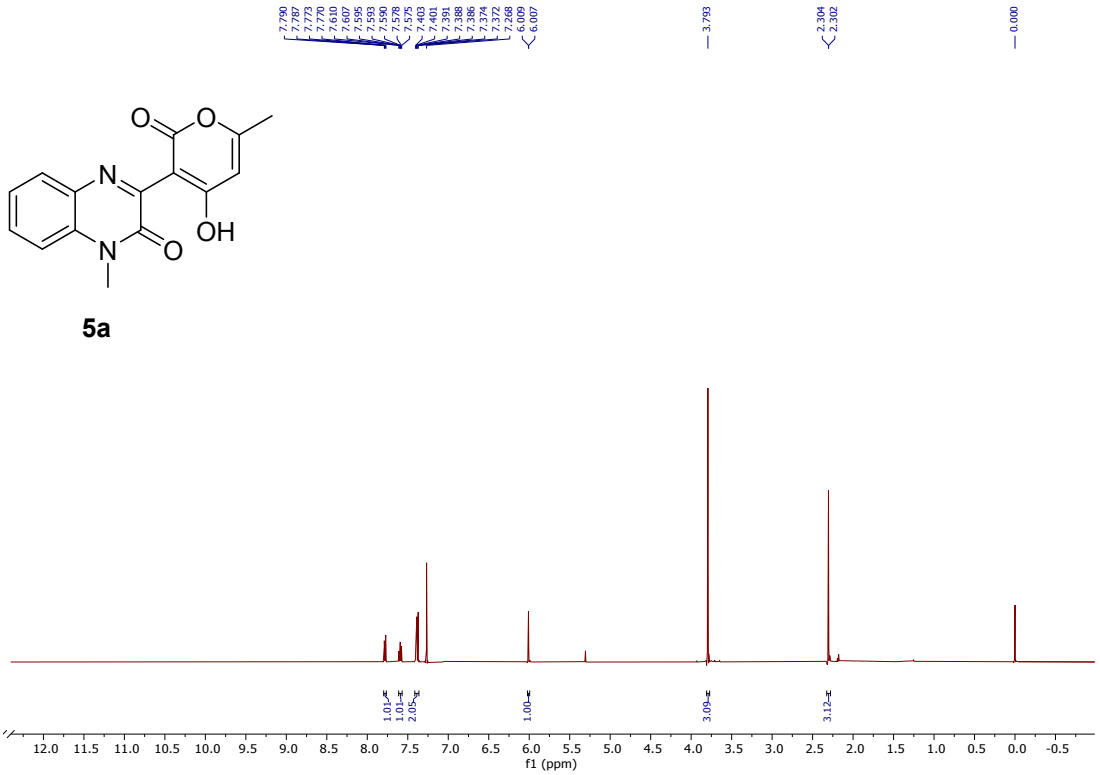

5a

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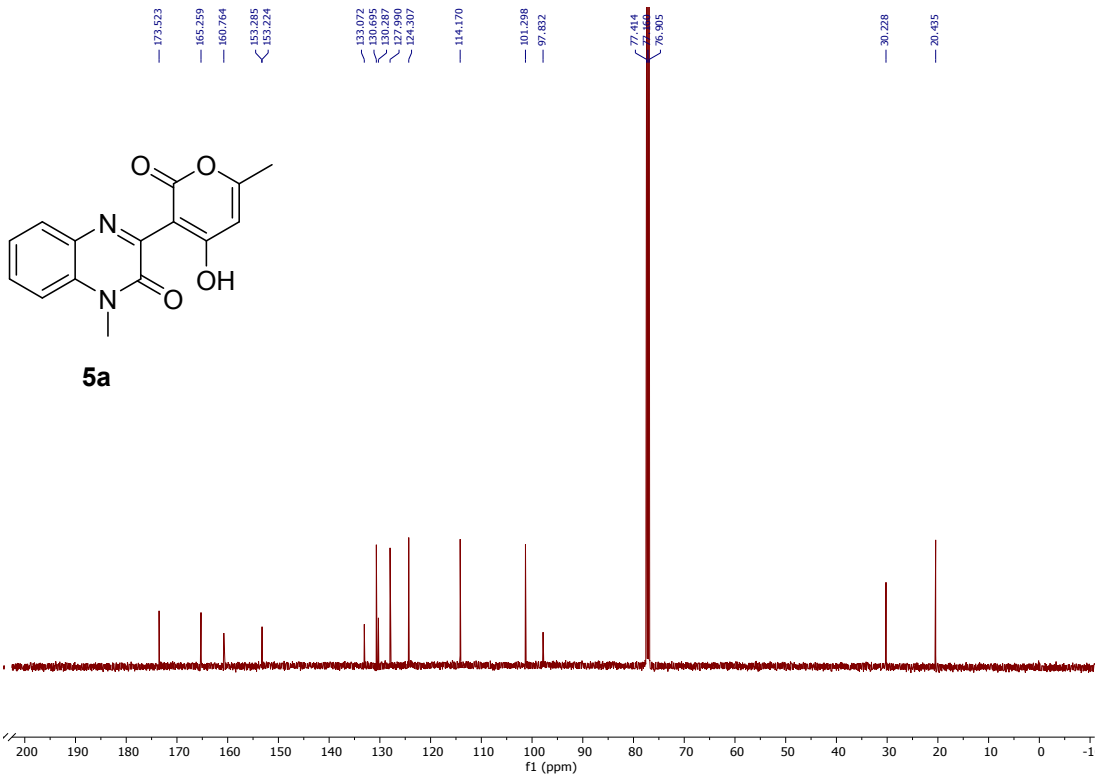

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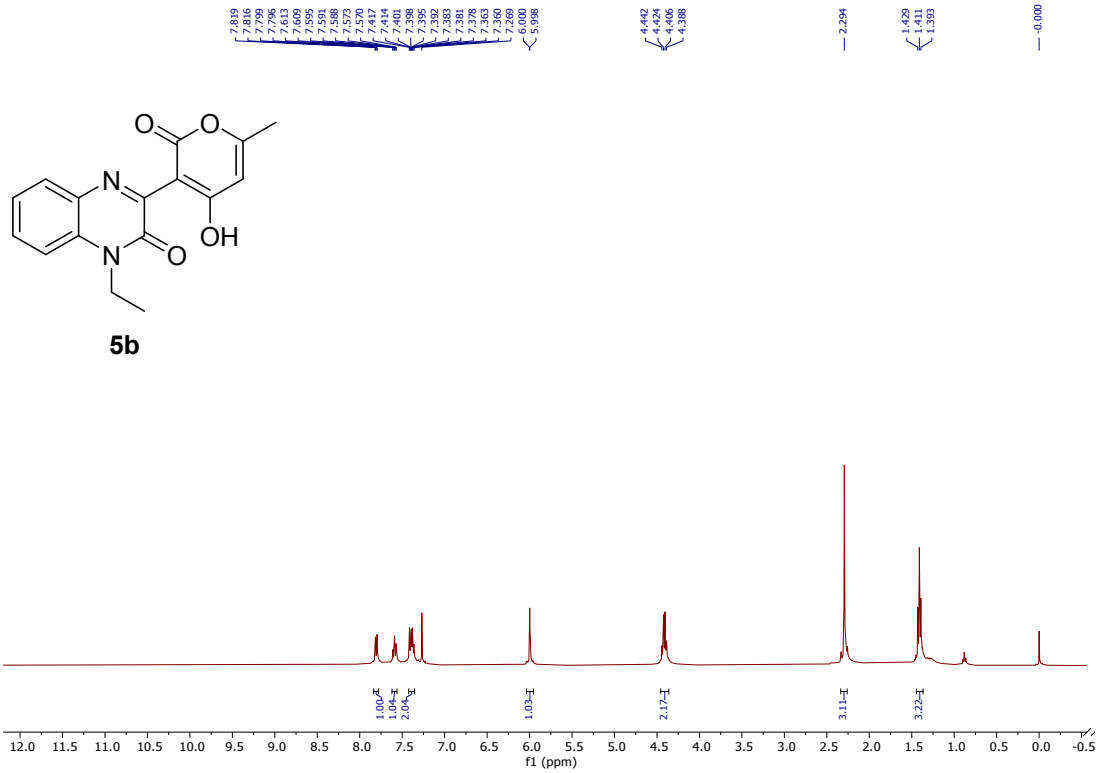

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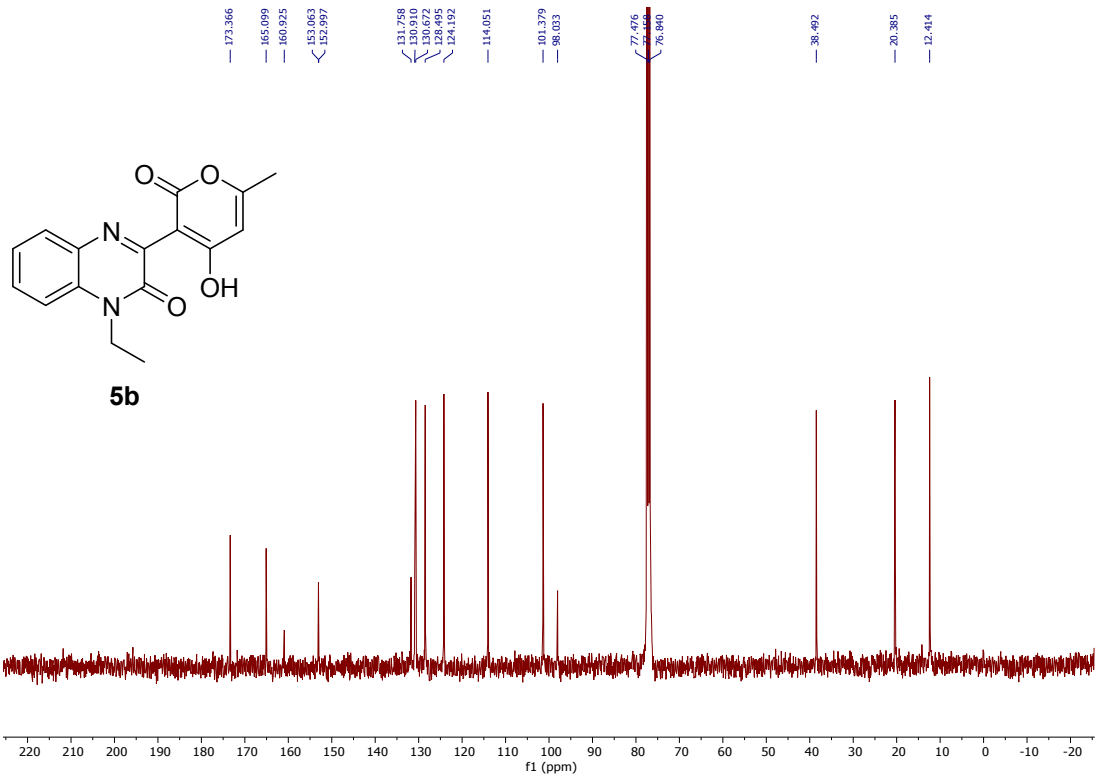

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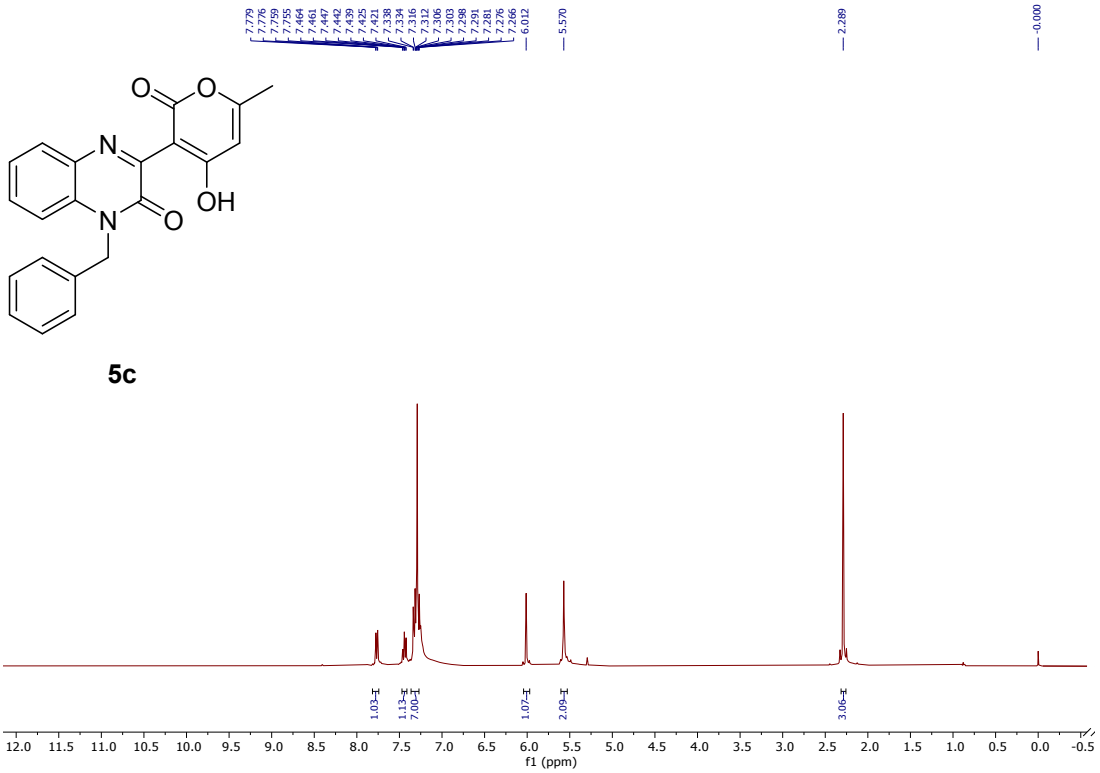

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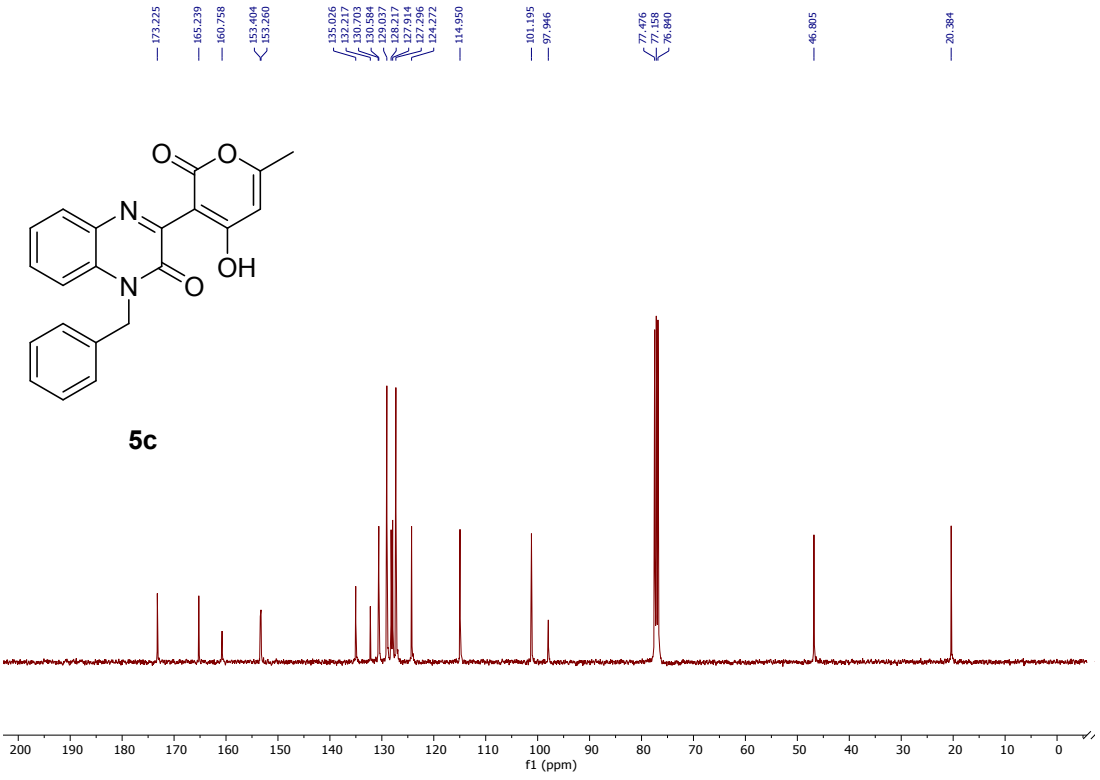

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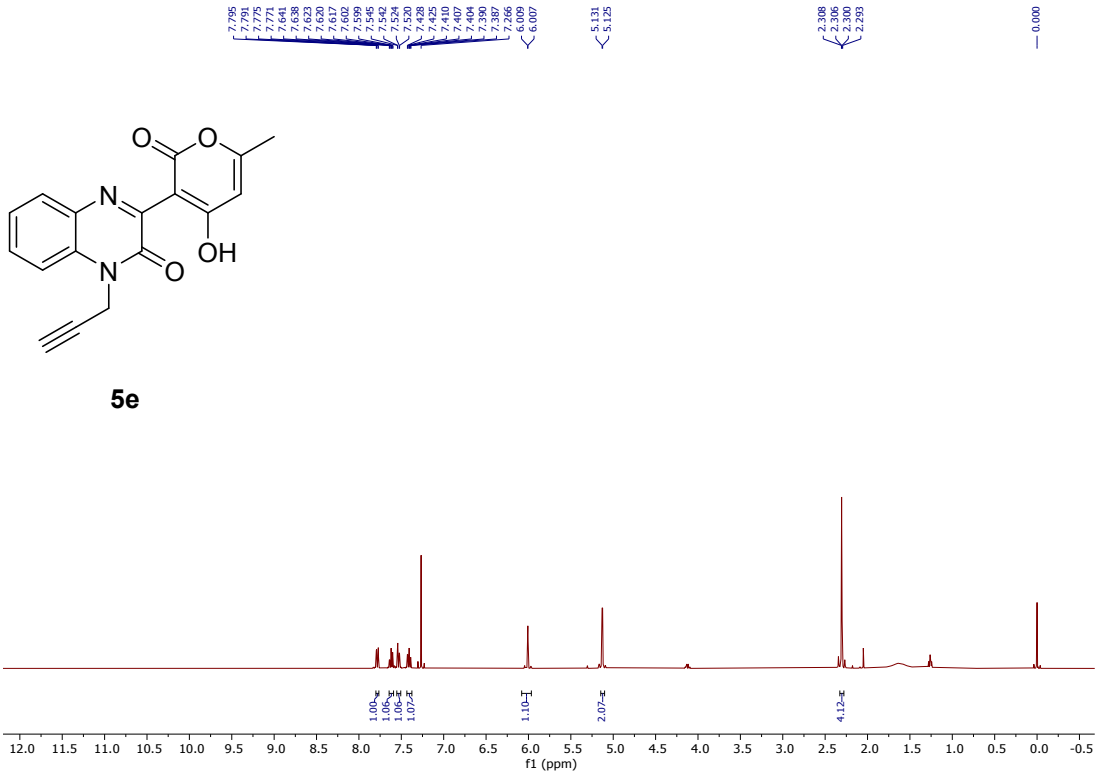

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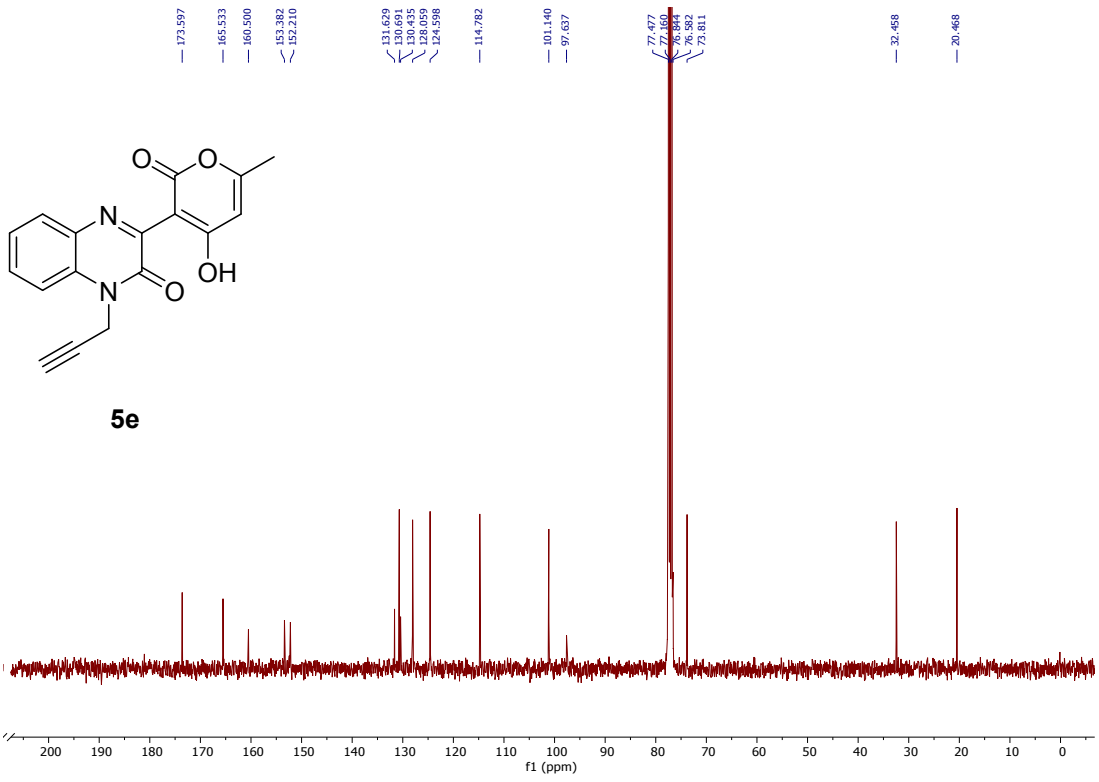

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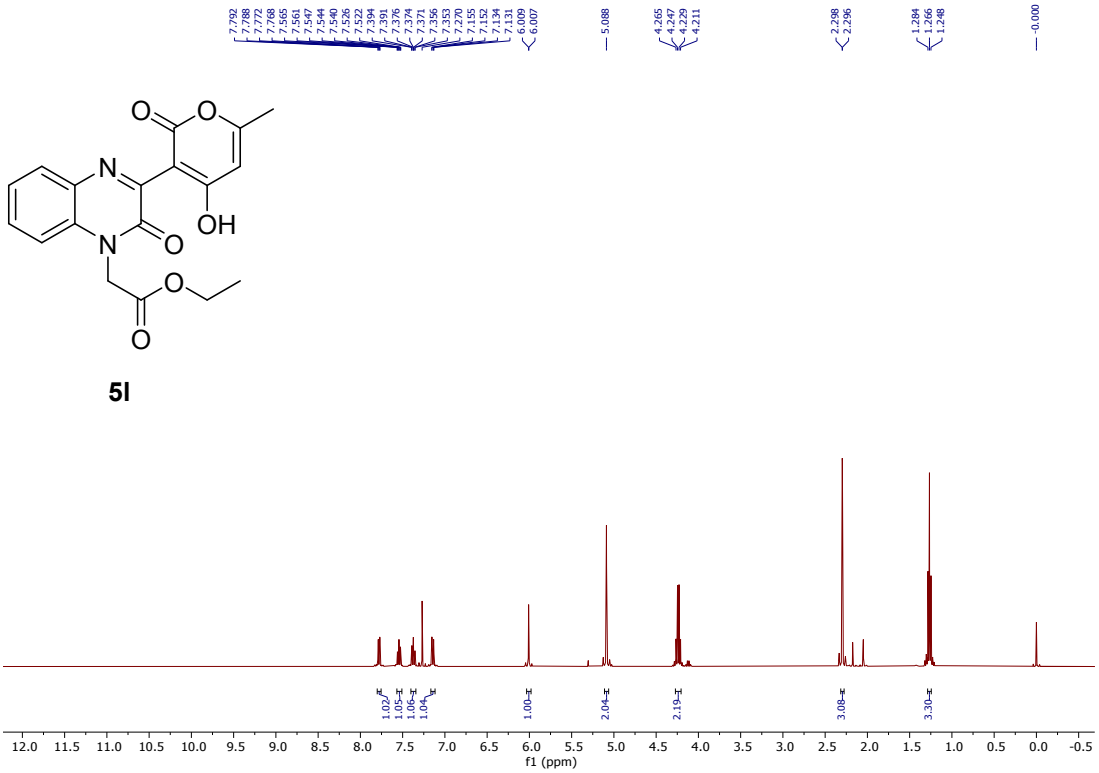

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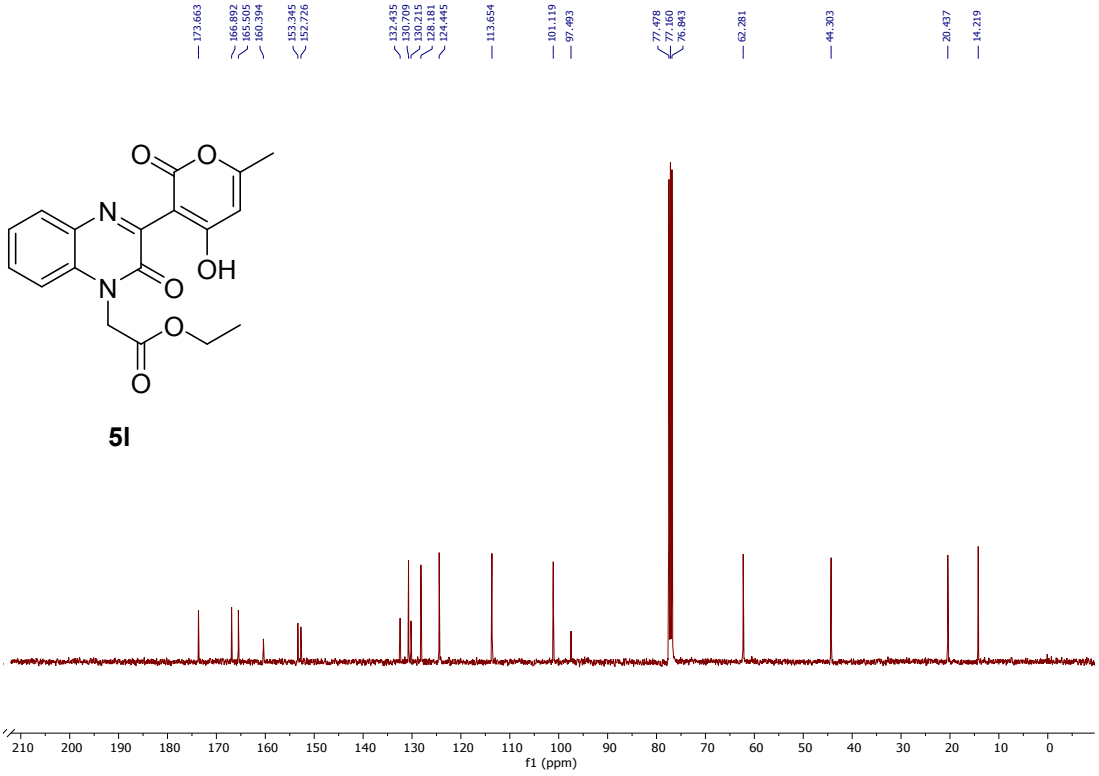

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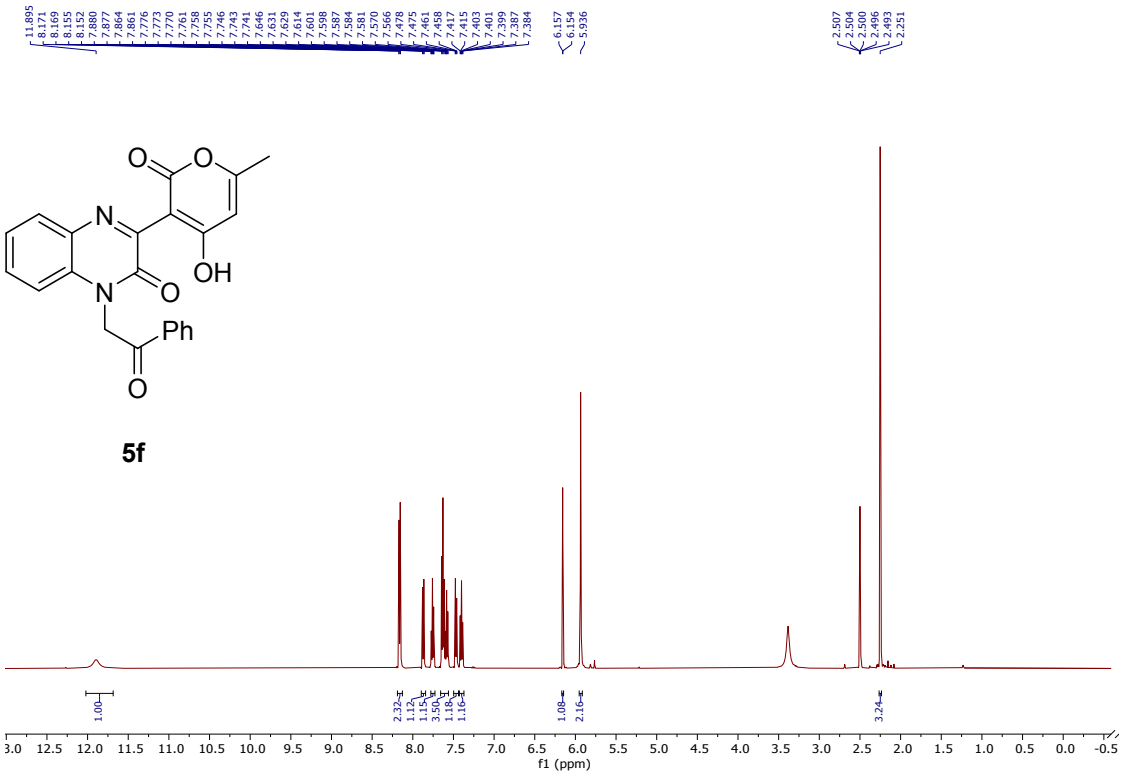

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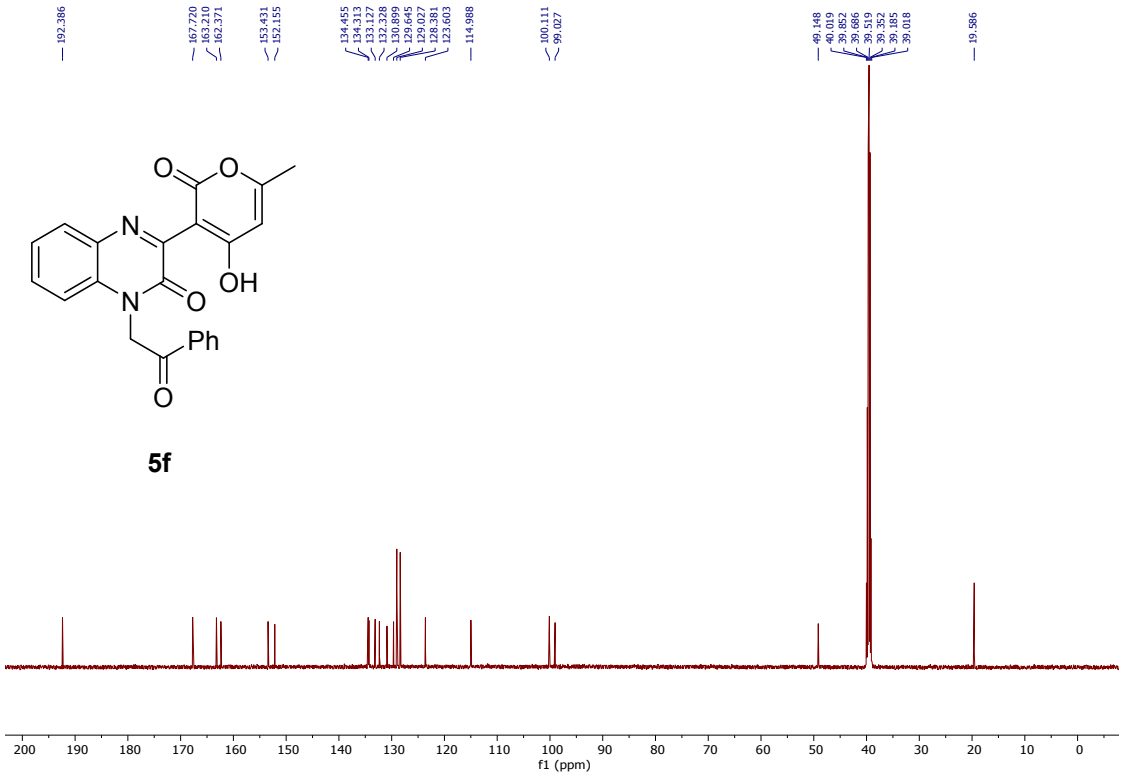

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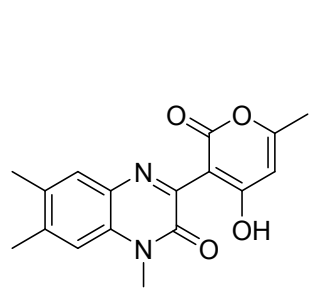

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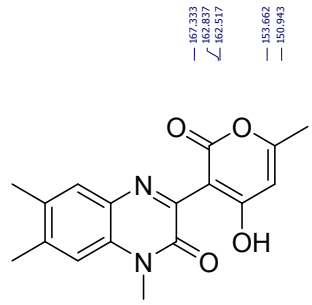

5g

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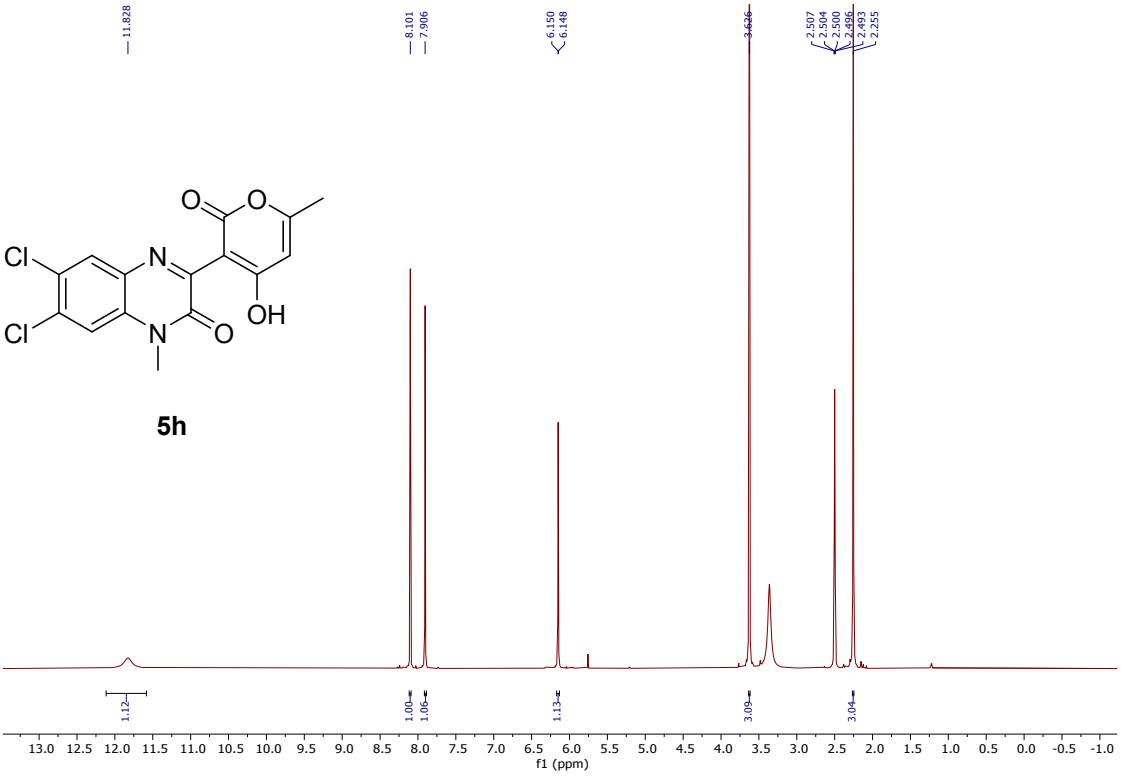

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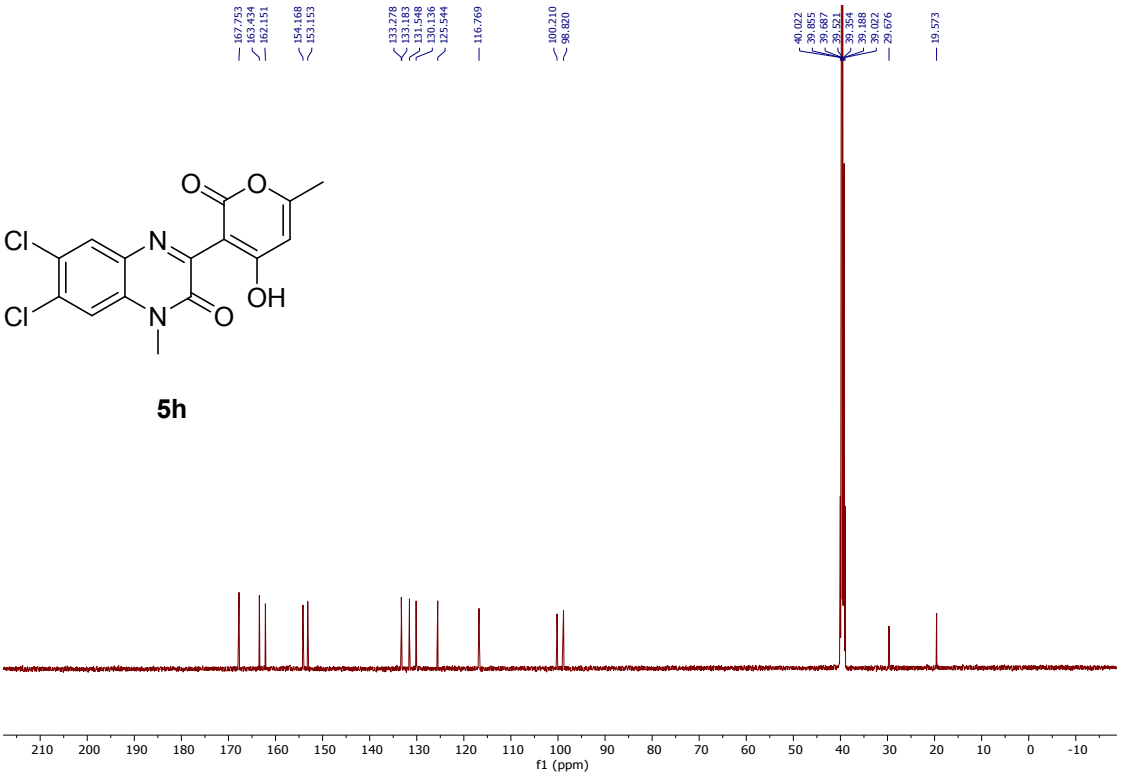
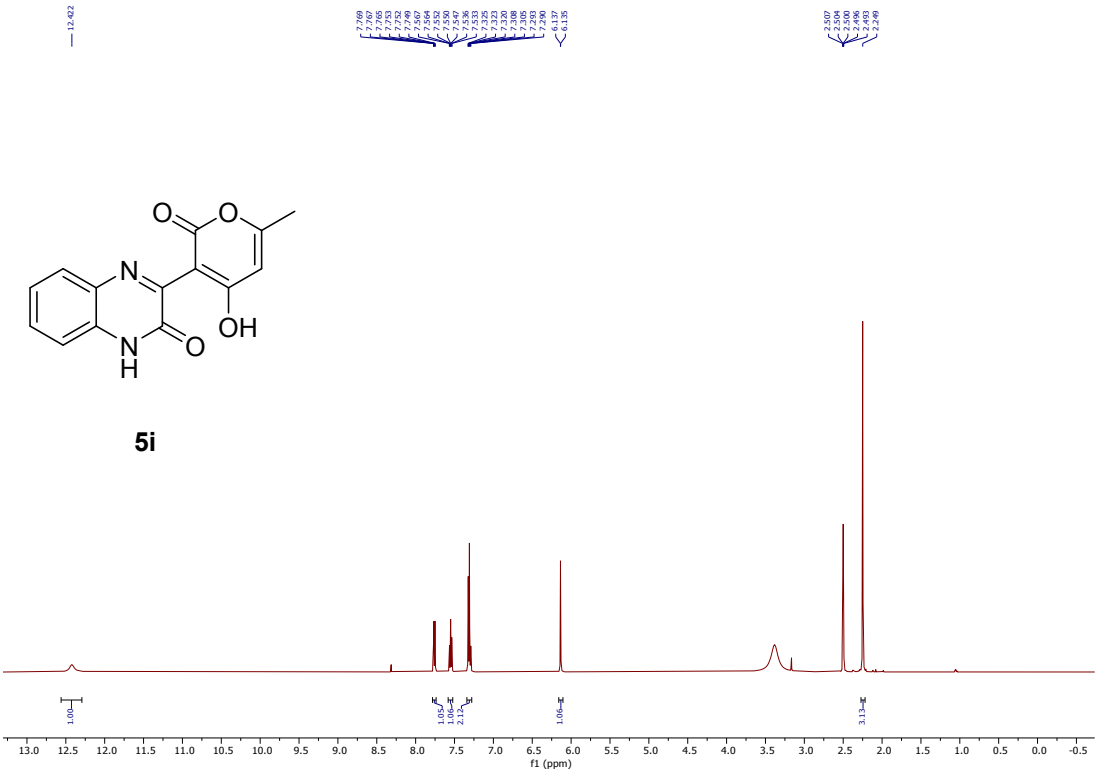


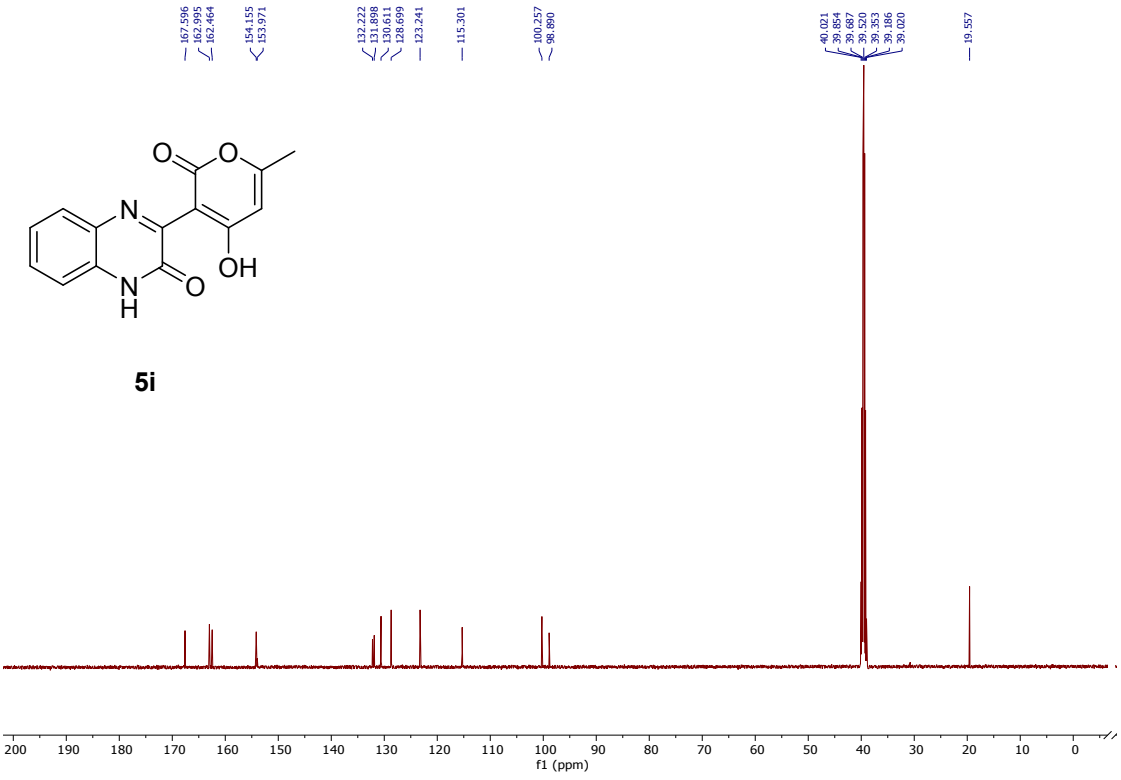
5h

^{1}H-NMR (500 MHz, DMSO-d_6)



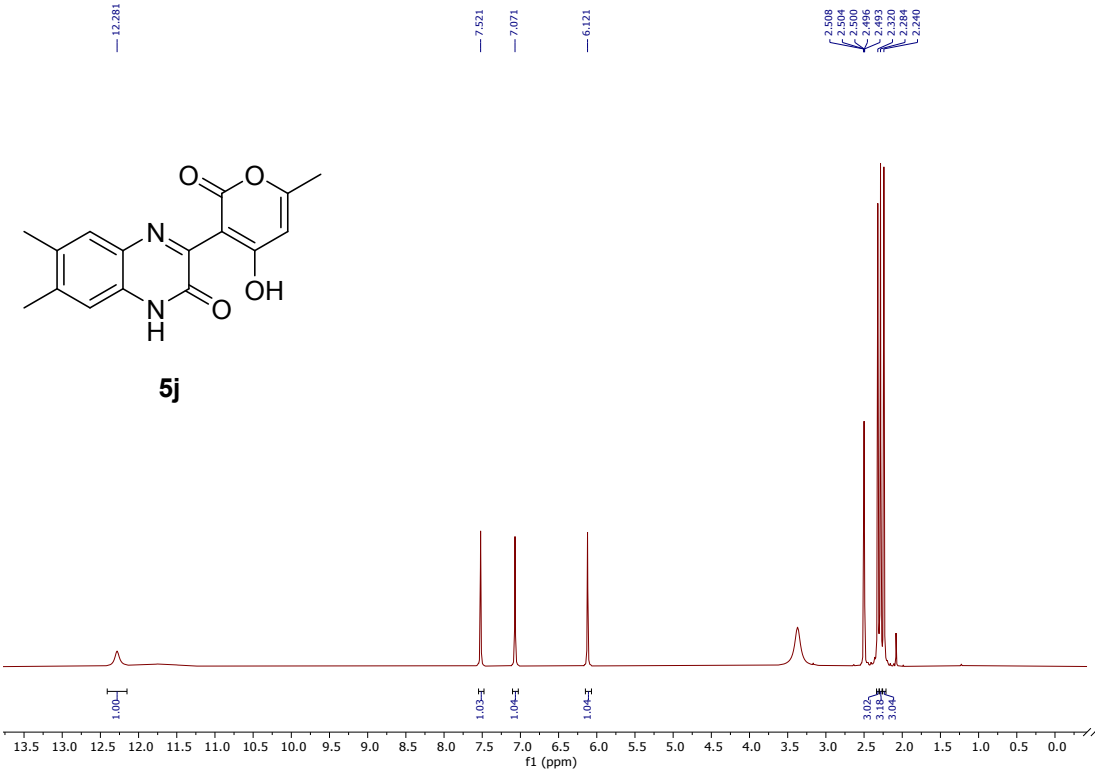
5i

^{13}C-NMR (125 MHz, DMSO-d_6)



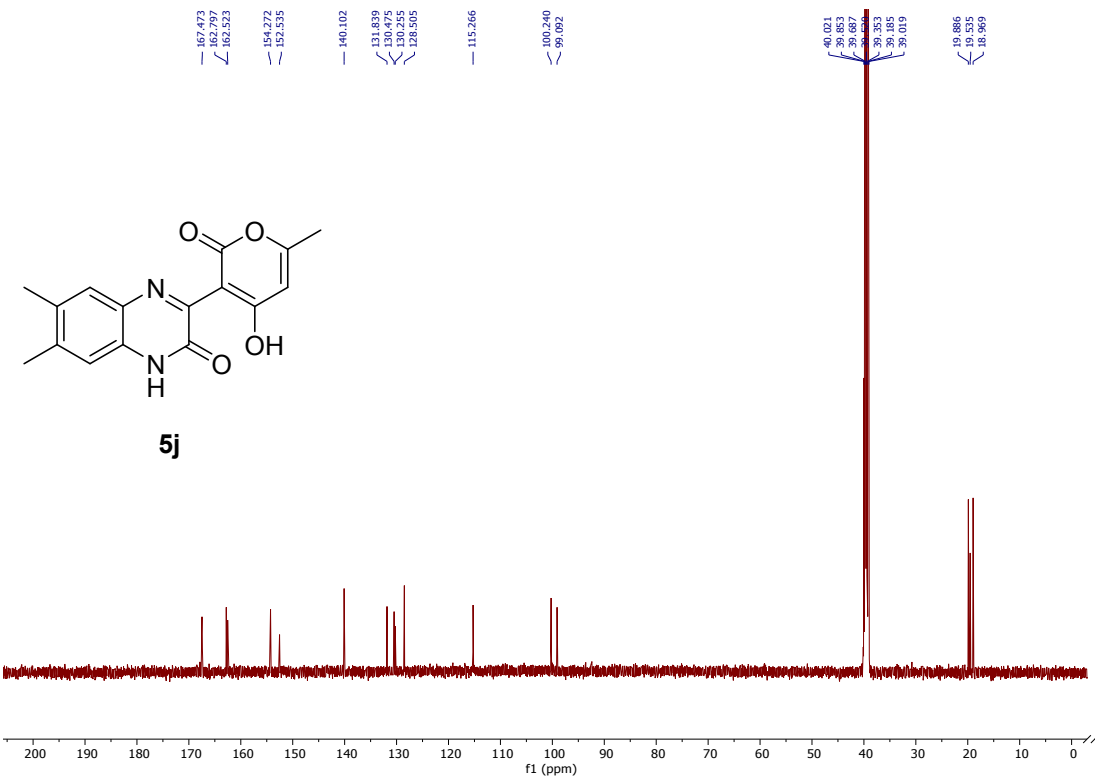
5i

^{1}H-NMR (500 MHz, DMSO-d_6)



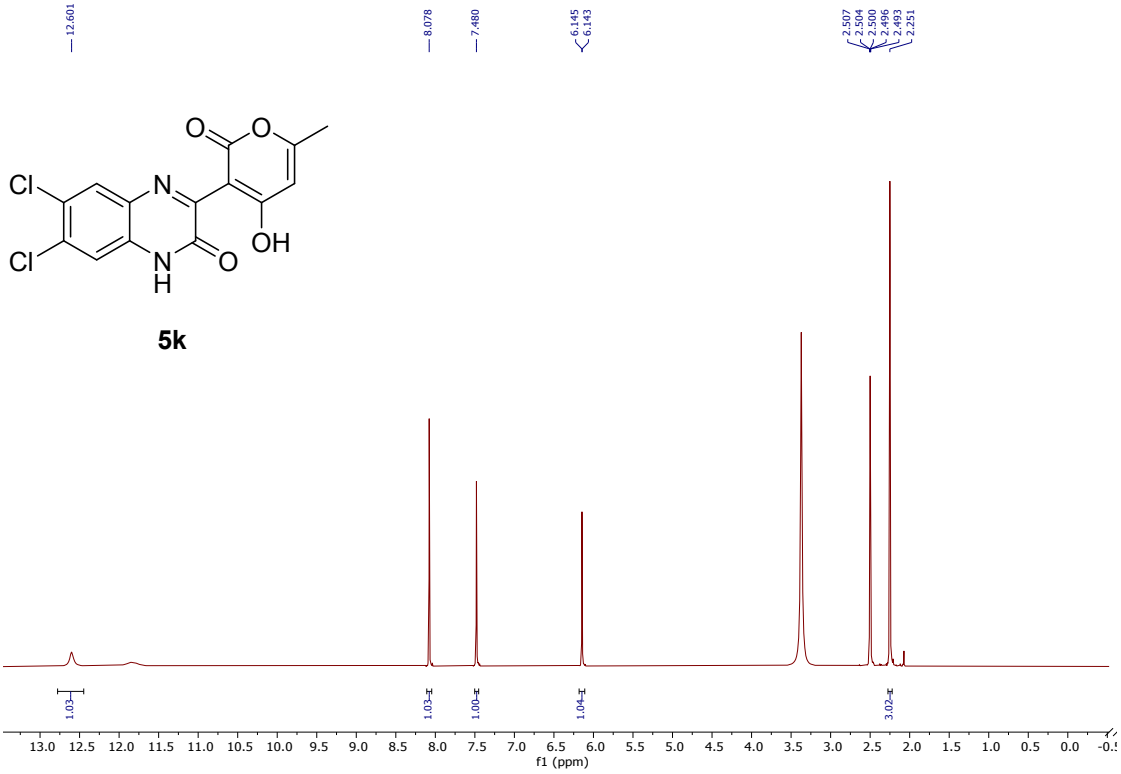
5j

^{13}C-NMR (125 MHz, DMSO-d_6)

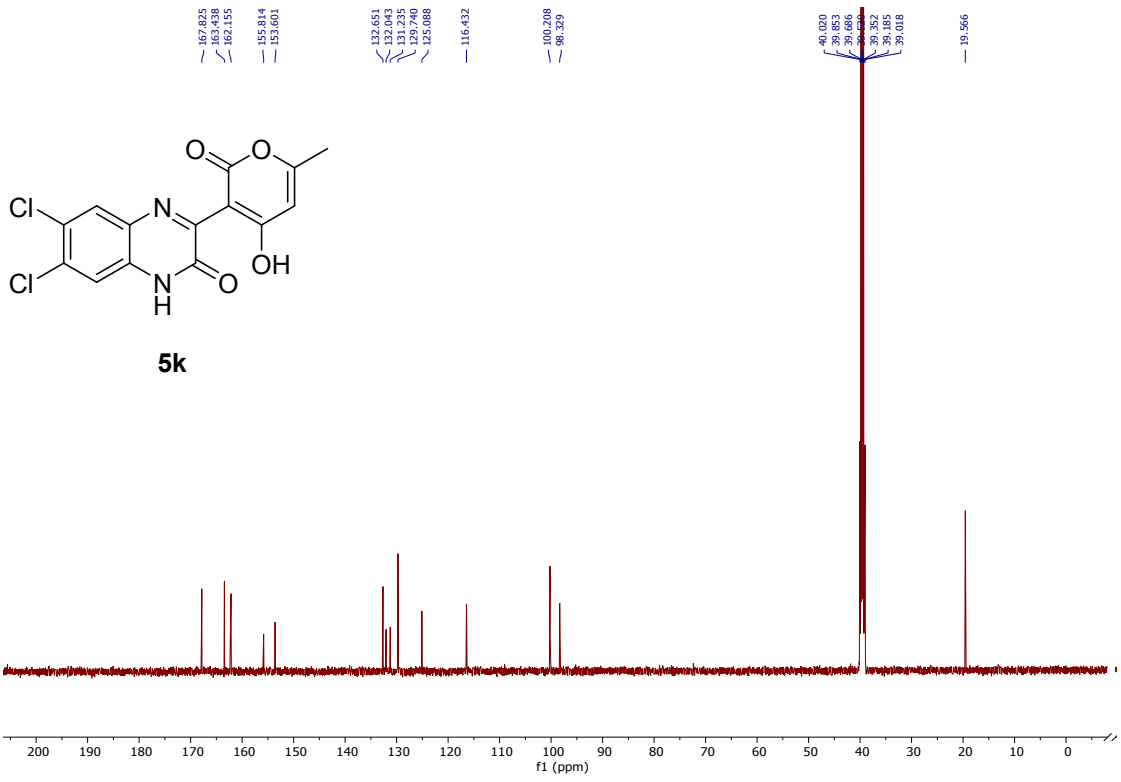


5j

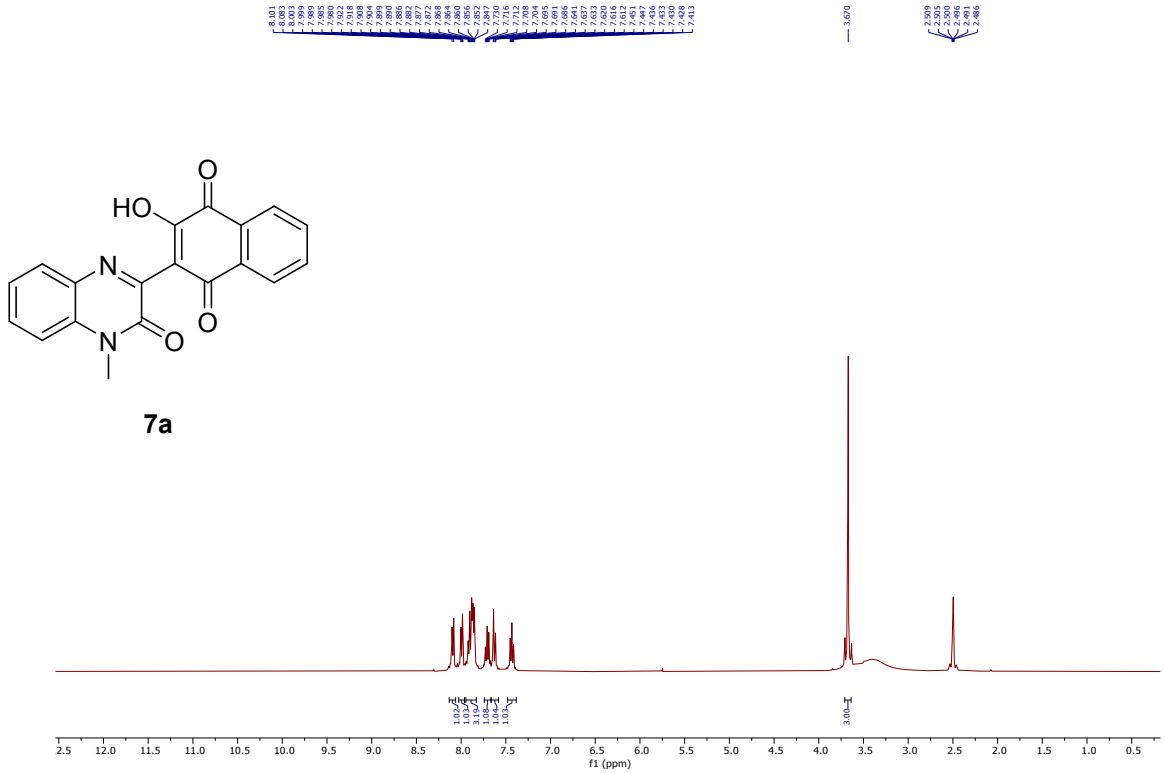
^{1}H-NMR (500 MHz, DMSO-d_6)



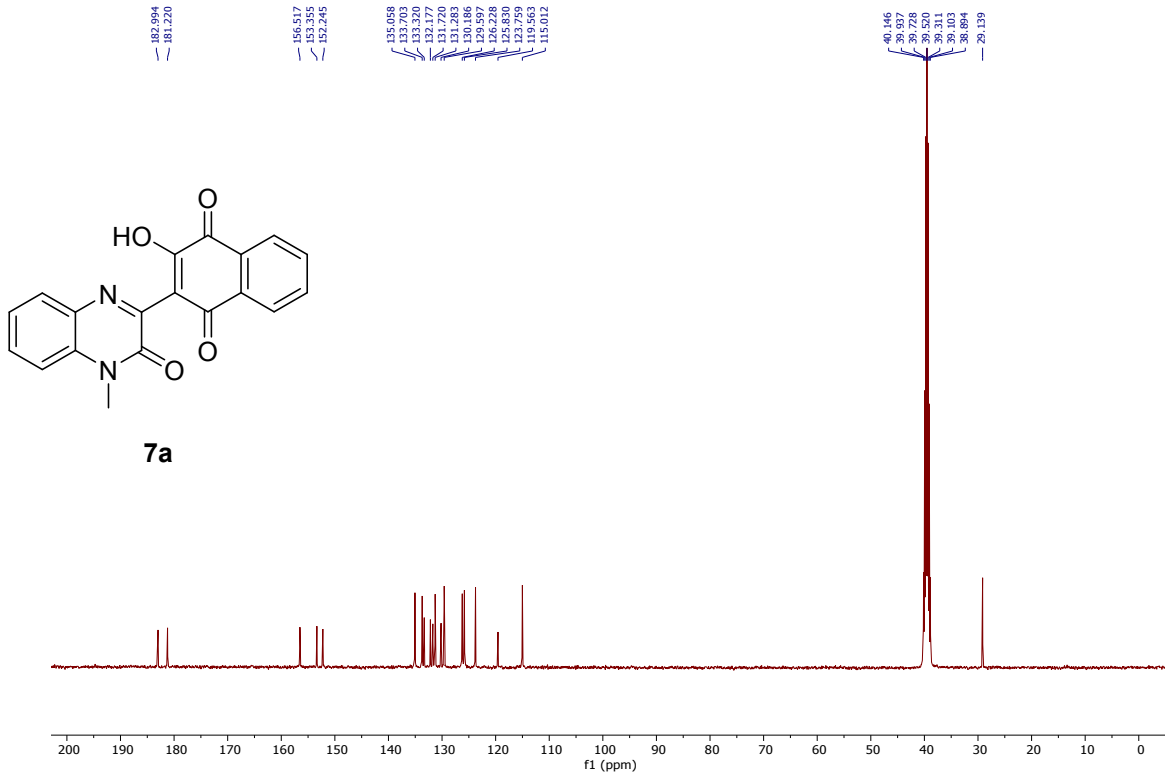
^{13}C-NMR (125 MHz, DMSO-d_6)



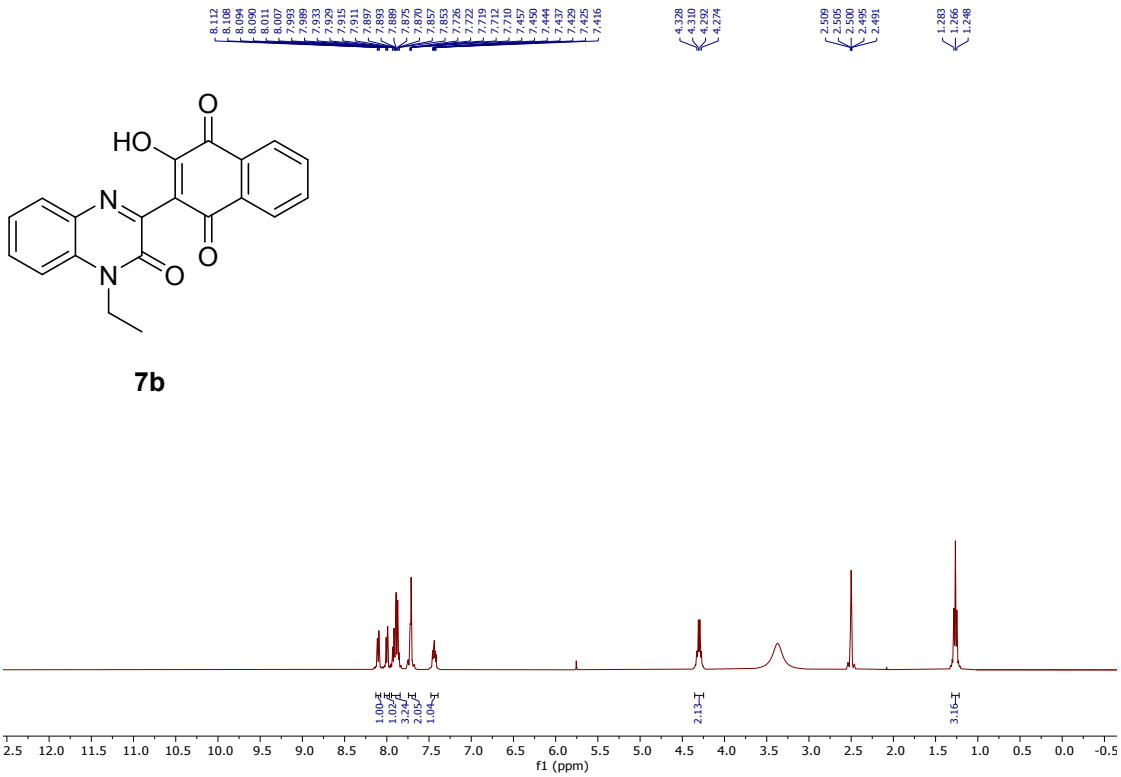
^{1}H-NMR (400 MHz, DMSO-d_6)



^{13}C-NMR (100 MHz, DMSO-d_6)

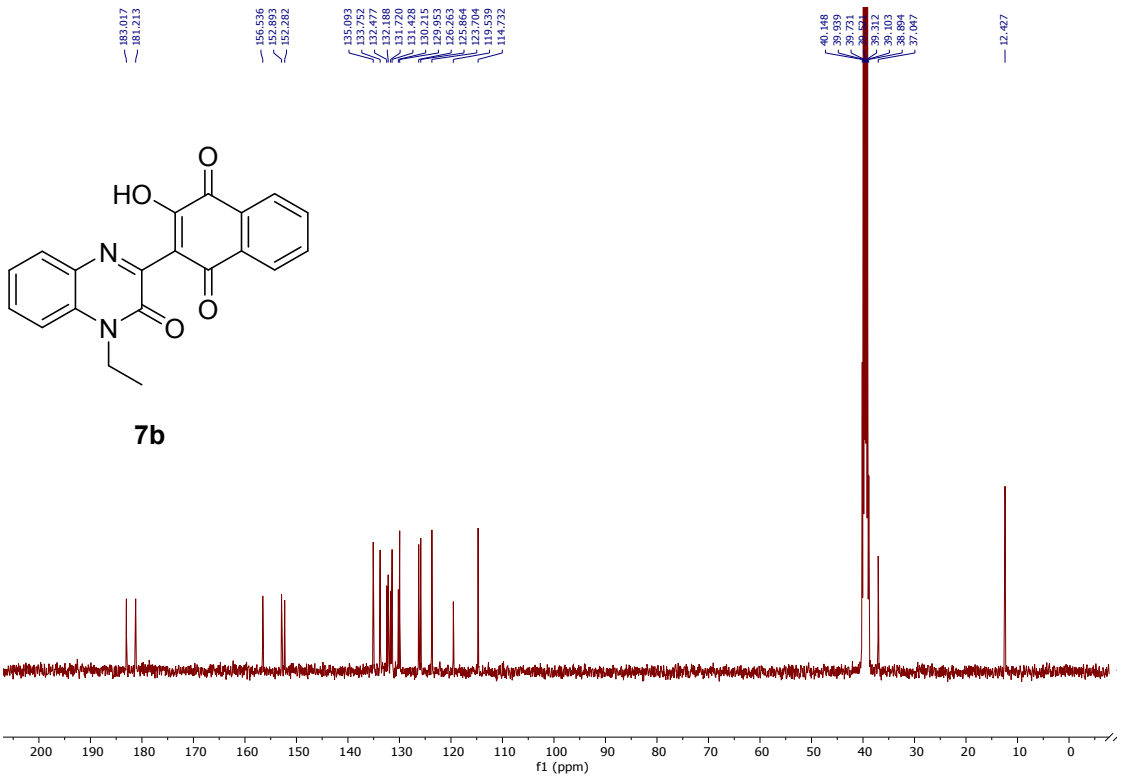


^{1}H-NMR (400 MHz, DMSO-d_6)



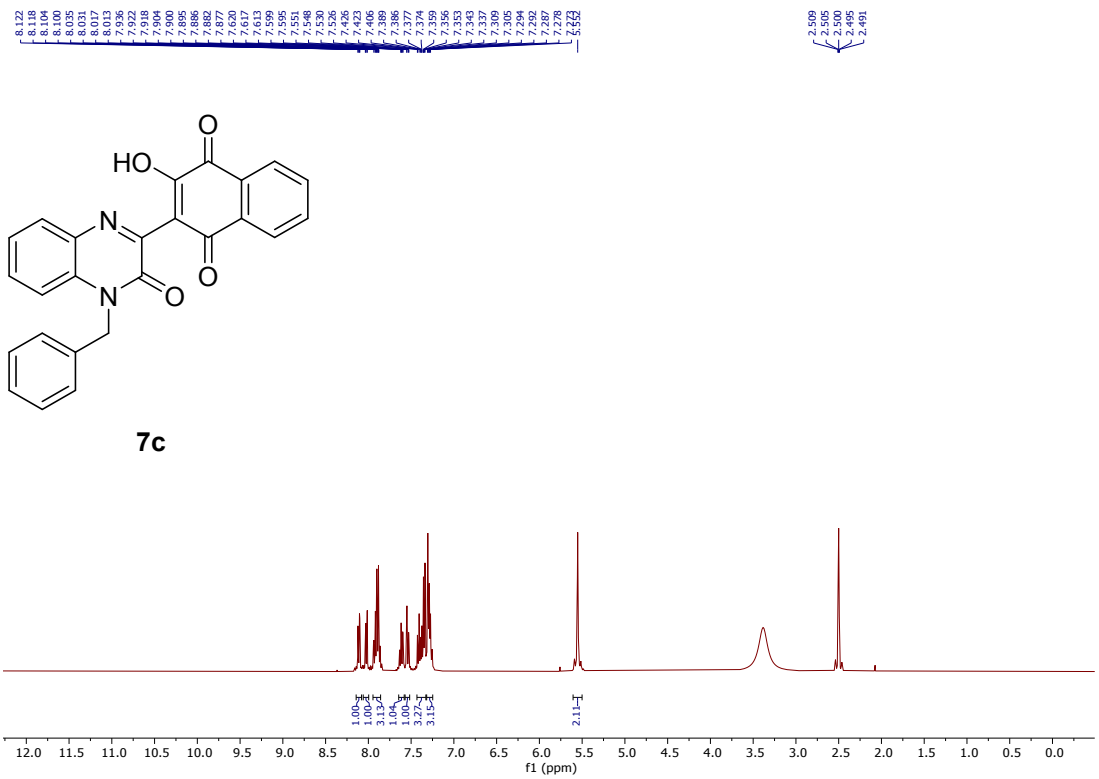
7b

^{13}C-NMR (100 MHz, DMSO-d_6)

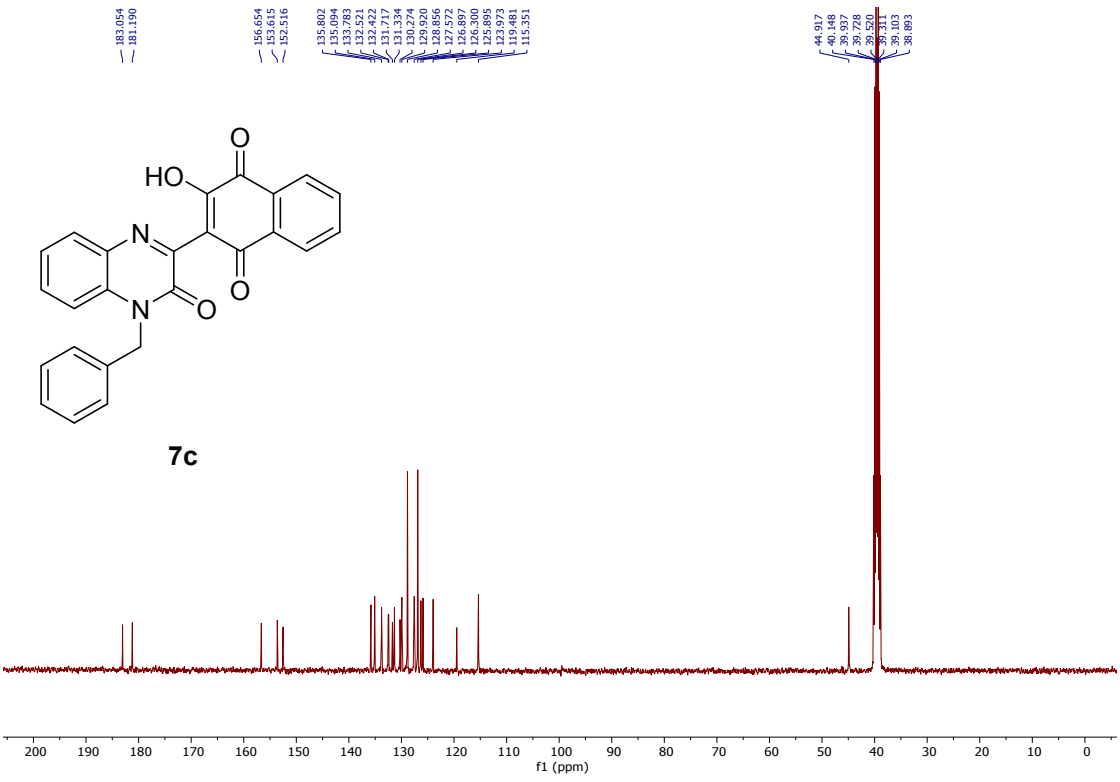


7b

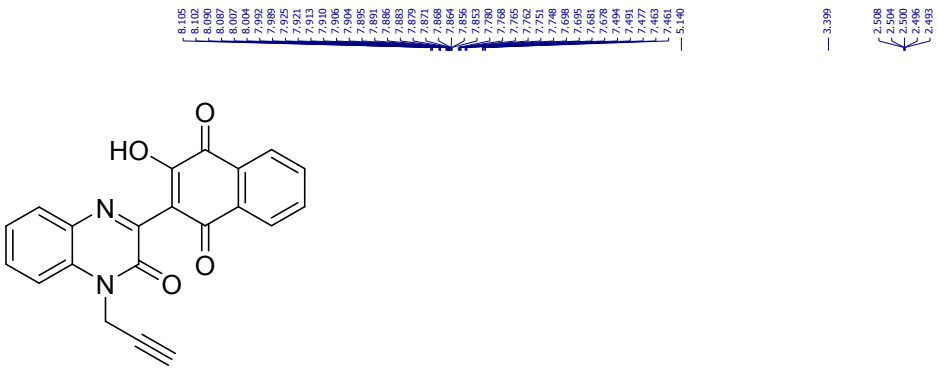
^{1}H-NMR (400 MHz, DMSO-d_6)



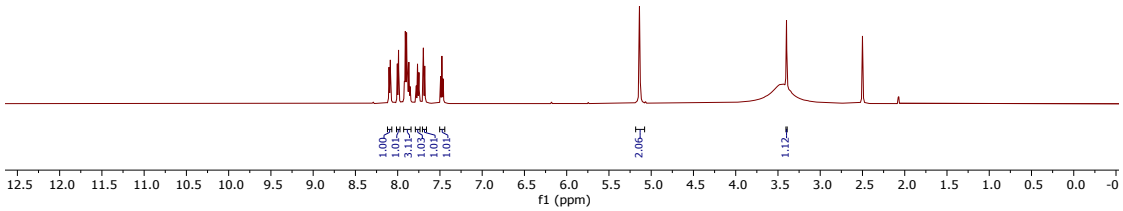
^{13}C-NMR (100 MHz, DMSO-d_6)



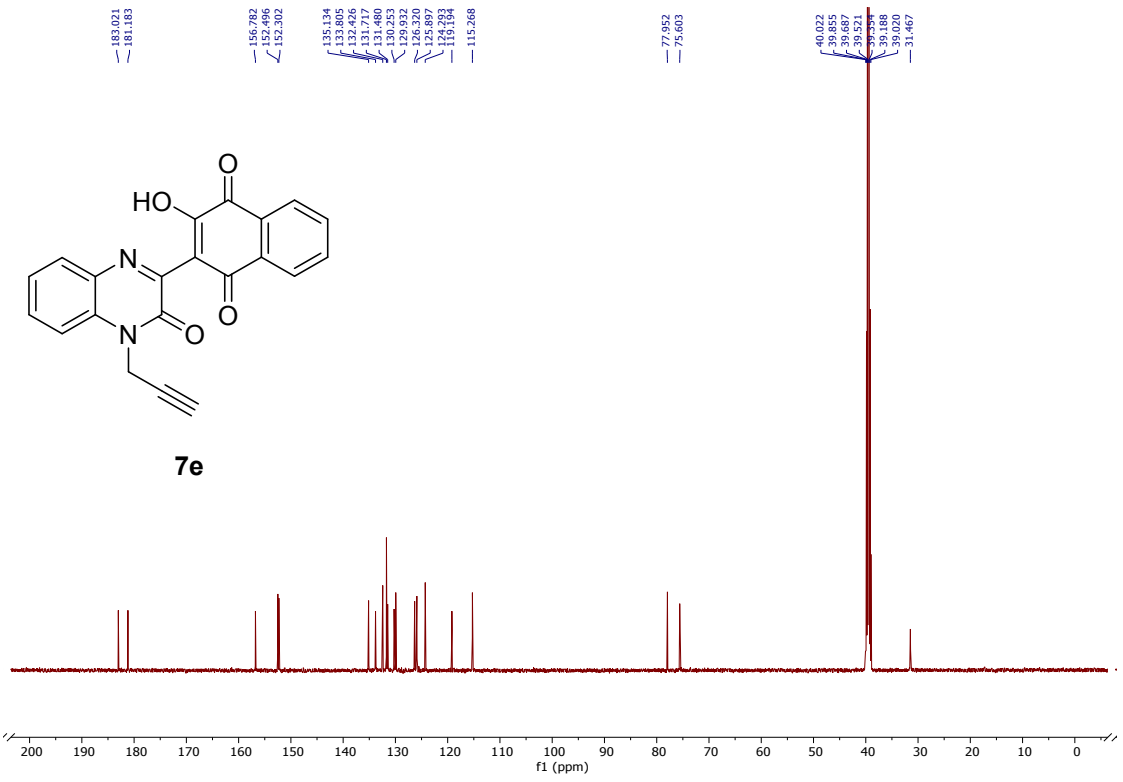
^{1}H-NMR (500 MHz, DMSO-d_6)



7e

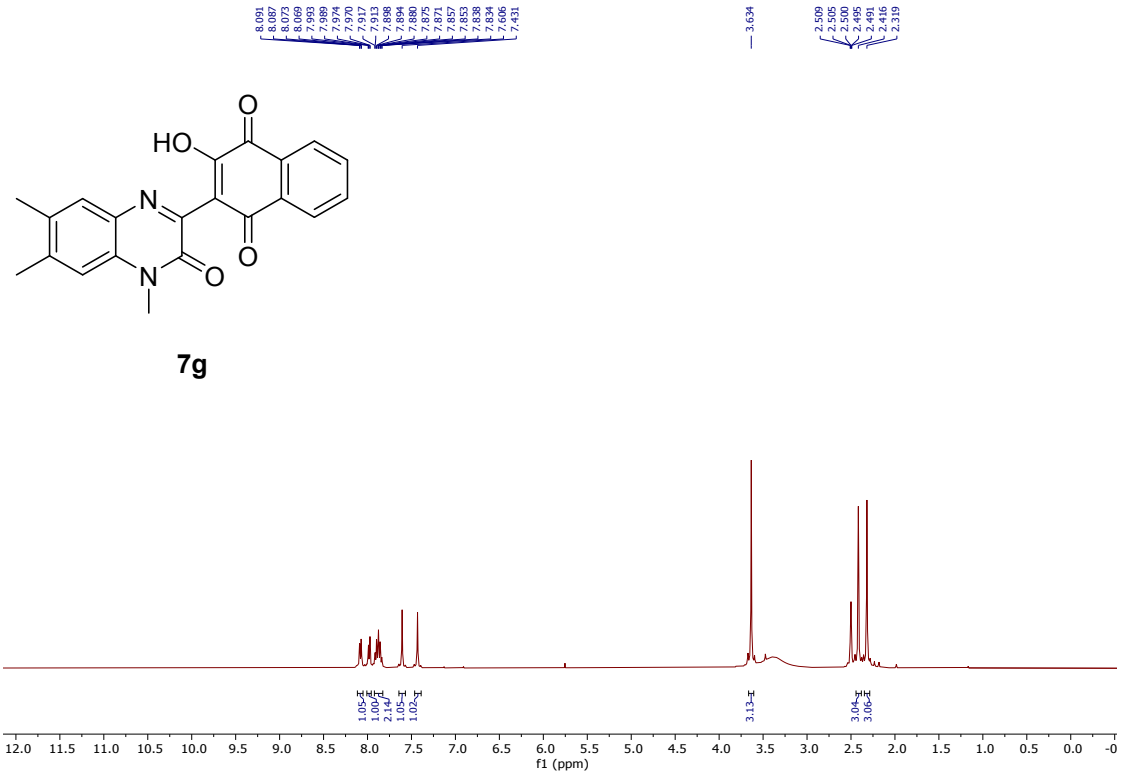


^{13}C-NMR (125 MHz, DMSO-d_6)



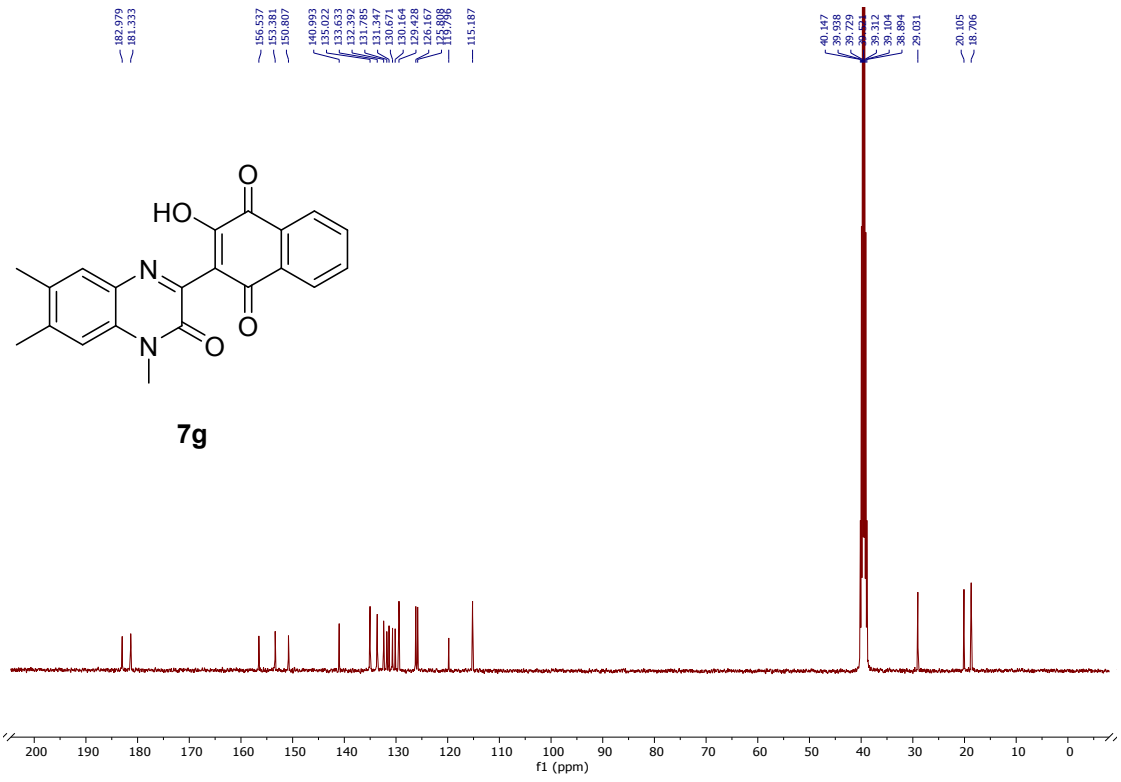
7e

^{1}H-NMR (400 MHz, DMSO-d_6)



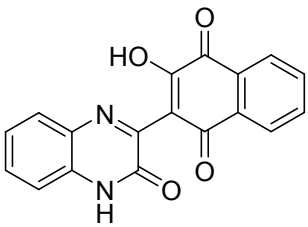
7g

^{13}C-NMR (100 MHz, DMSO-d_6)

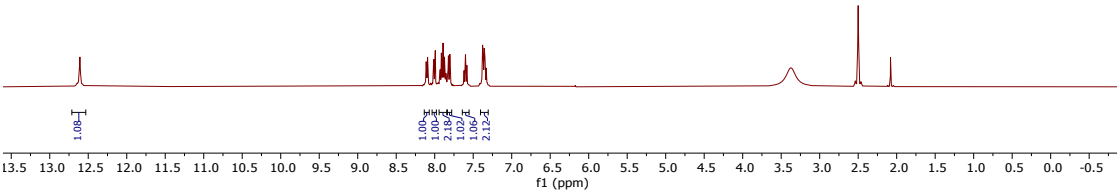


7g

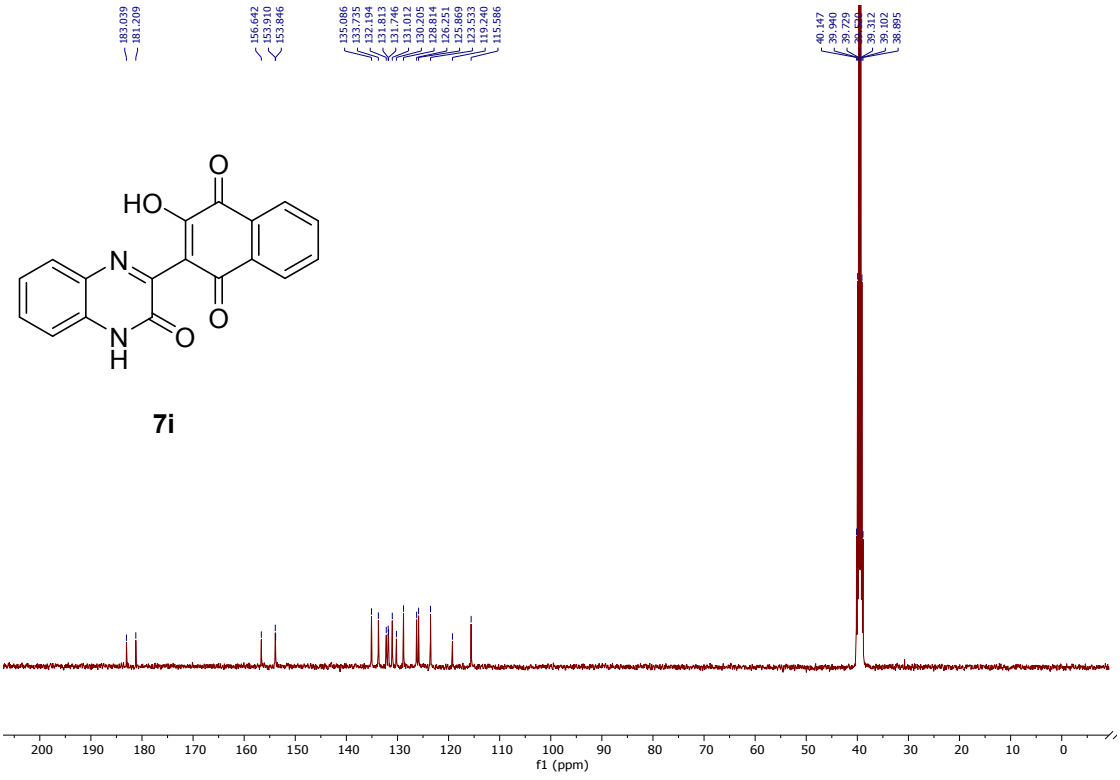
^{1}H-NMR (400 MHz, DMSO-d_6)



7i

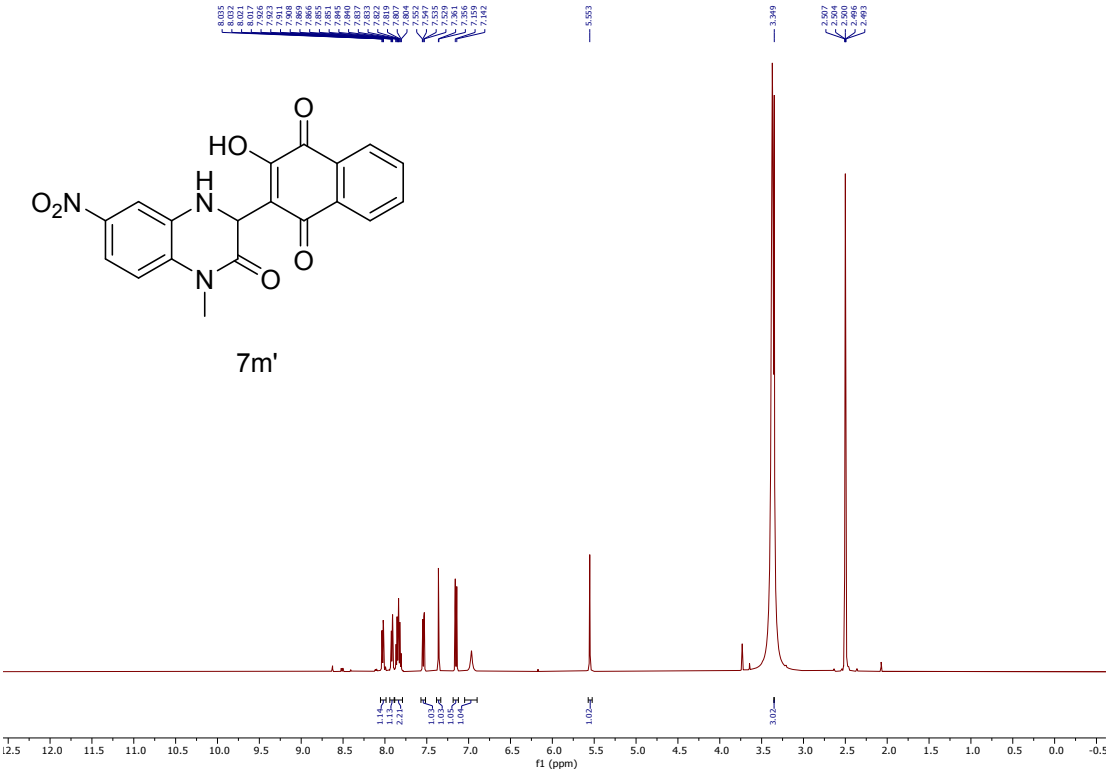


^{13}C-NMR (100 MHz, DMSO-d_6)

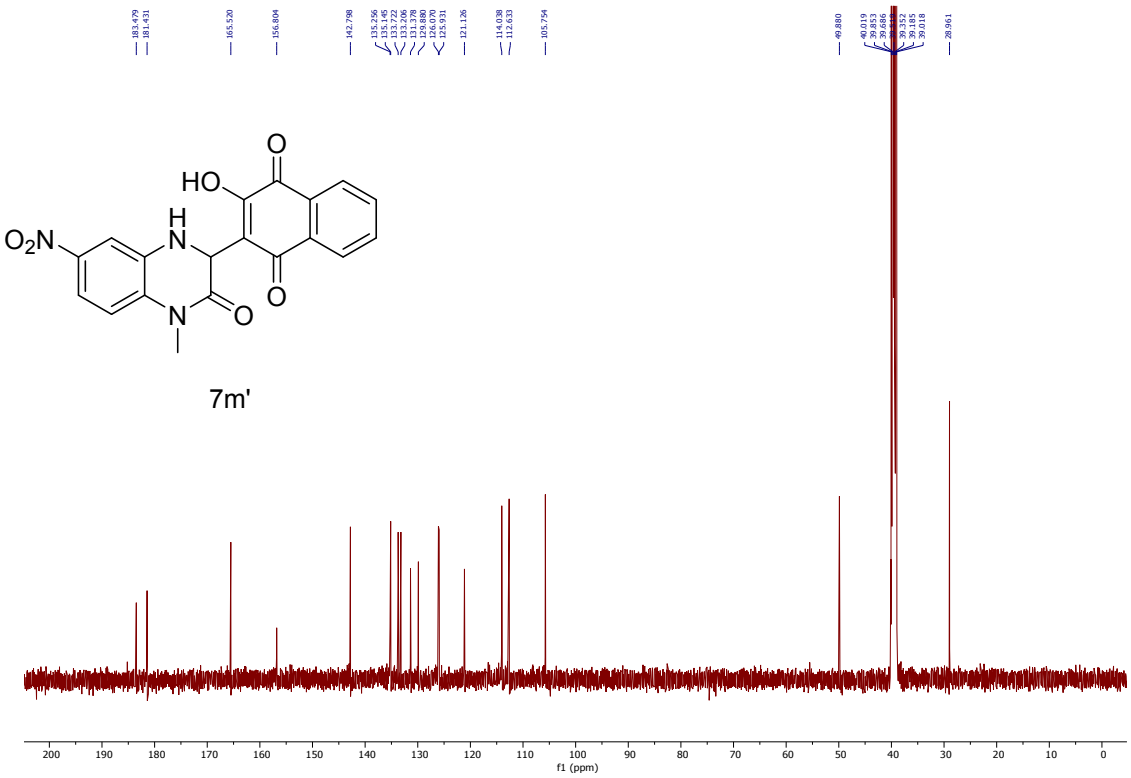


7i

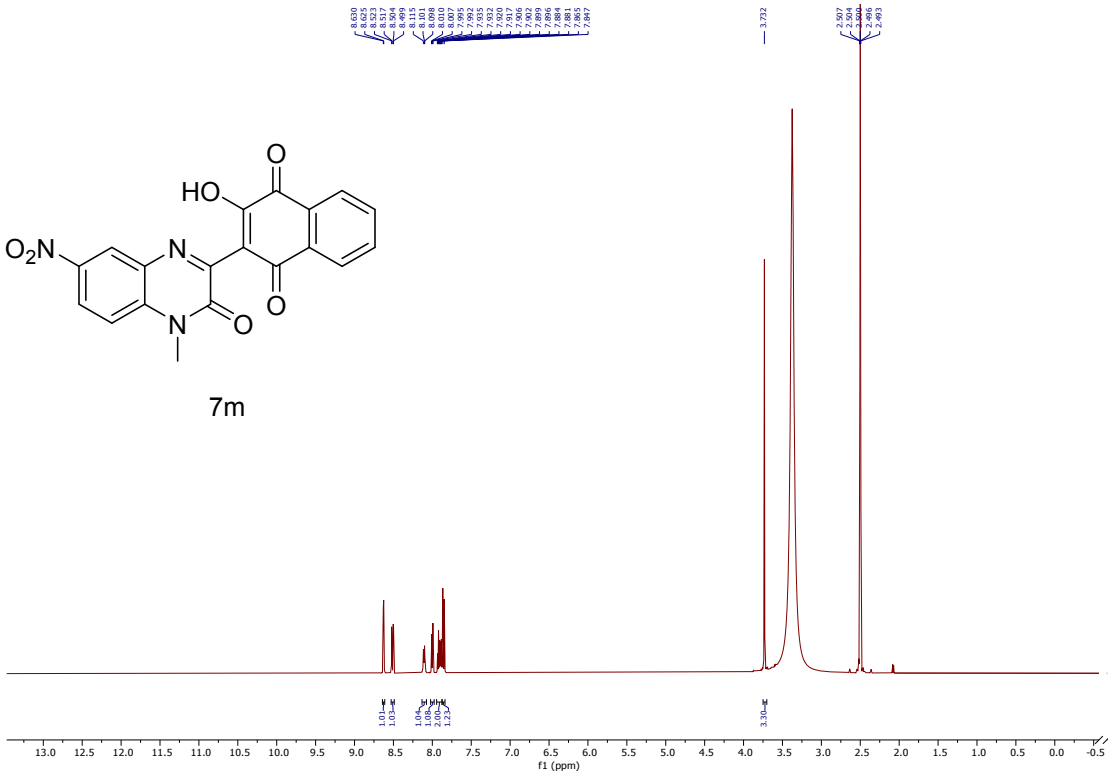
^{1}H-NMR (500 MHz, DMSO-d_6)



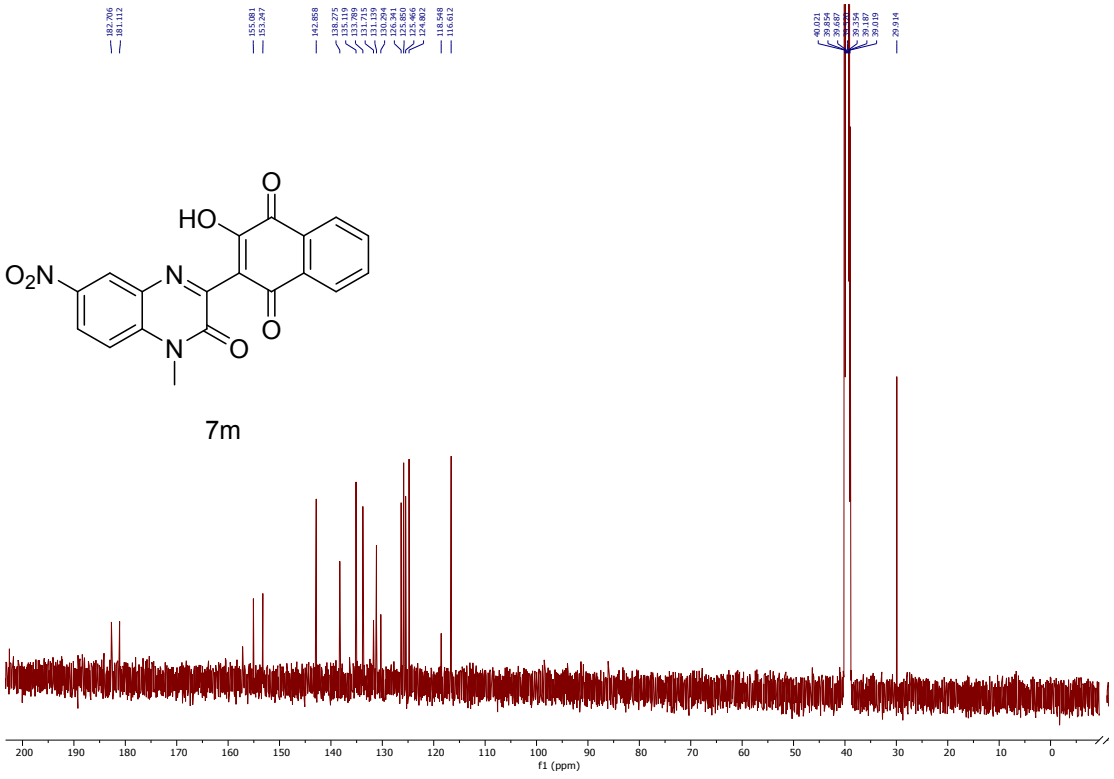
^{13}C-NMR (125 MHz, DMSO-d_6)



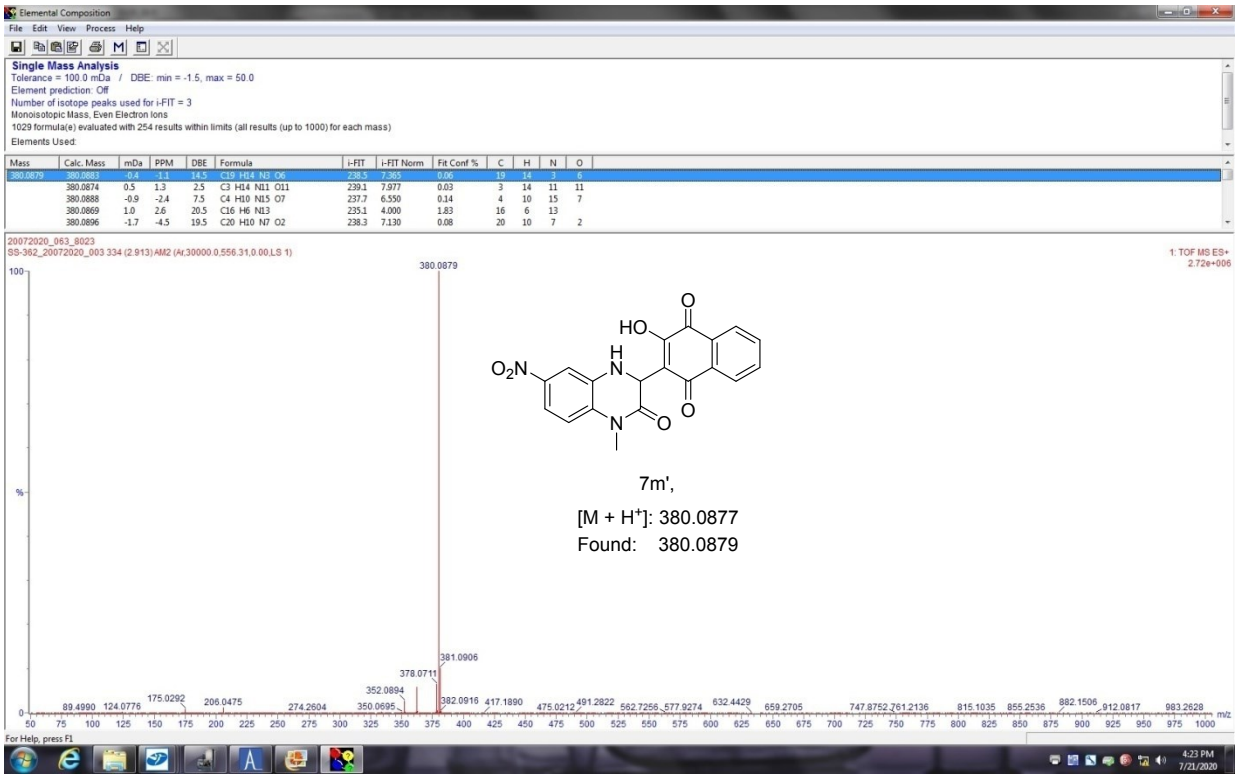
^{1}H-NMR (500 MHz, DMSO-d_6)

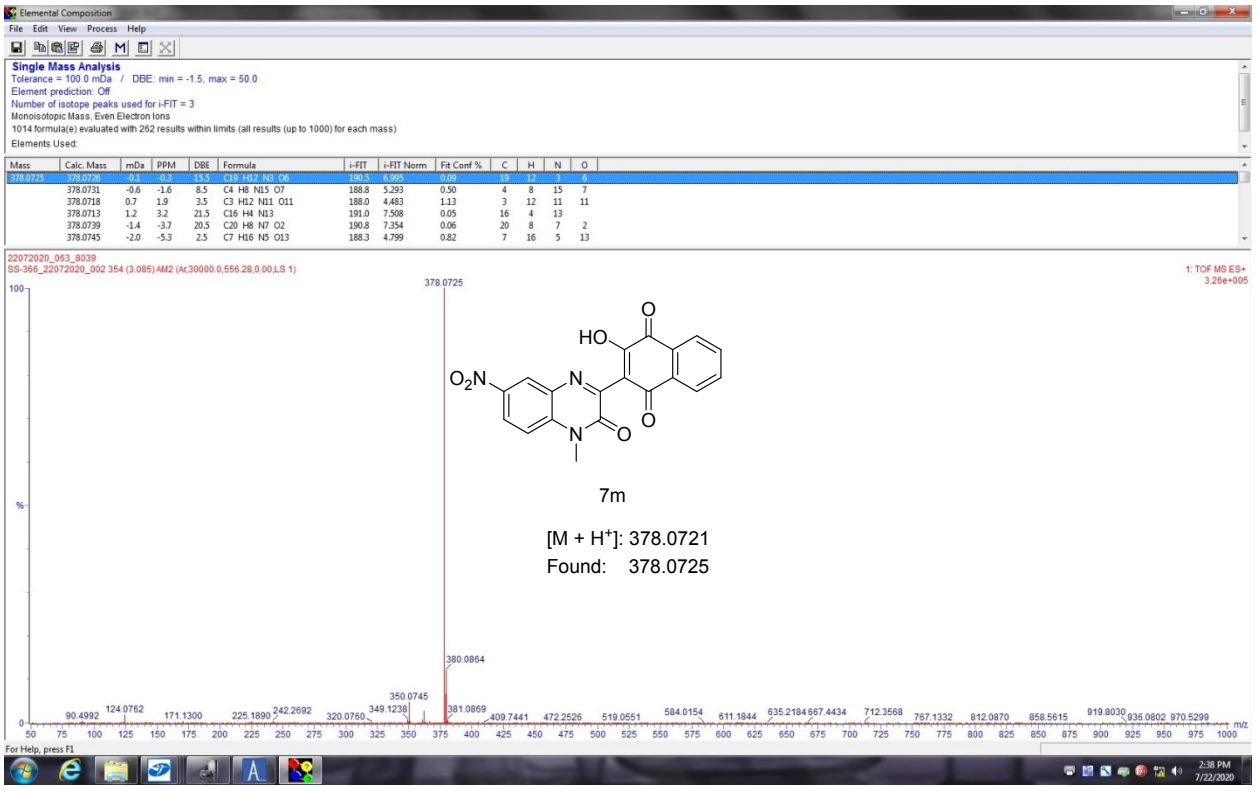


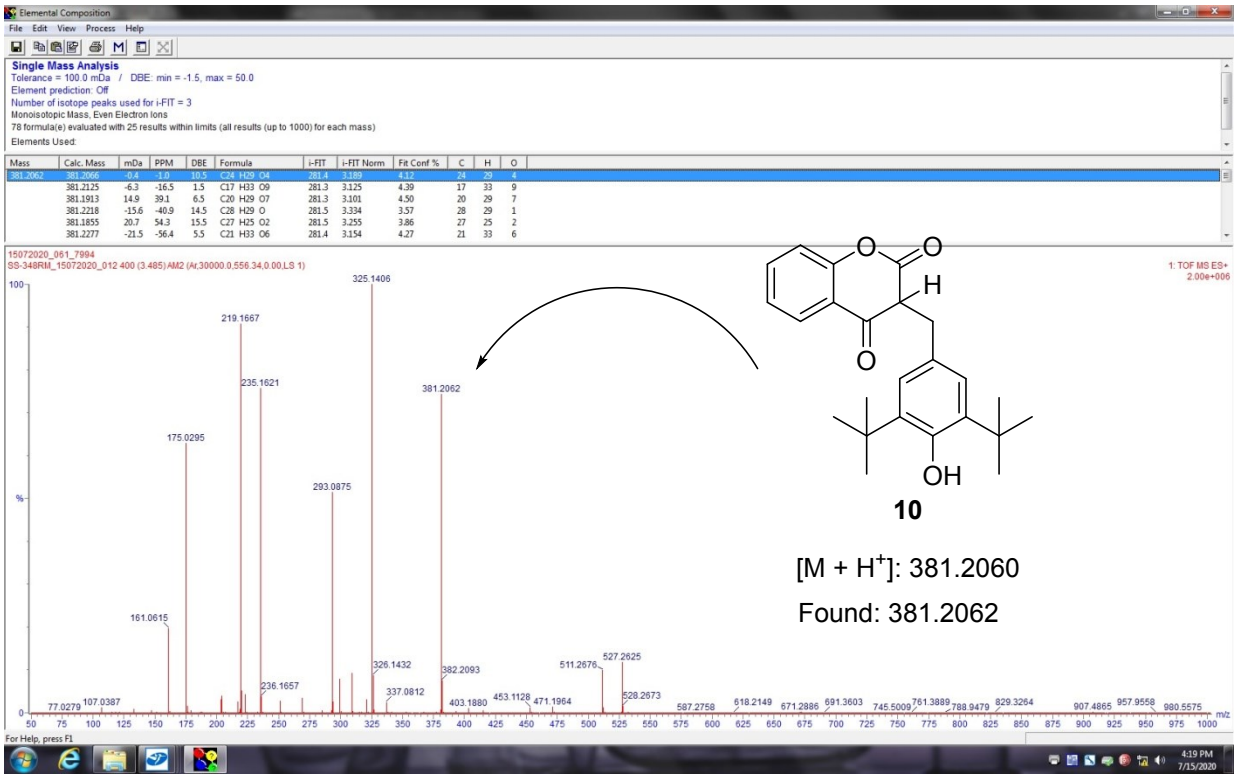
^{13}C-NMR (125 MHz, DMSO-d_6)



HRMS Data


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1029 formula(e) evaluated with 254 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass | Calc. Mass | mDa | PPM | DBE | Formula | i-FIT | i-FIT Norm | Fit Conf % | C | H | N | O
380.0879 | 380.0883 | -0.4 | -1.1 | 14.5 | C19 H14 N3 O6 | 238.5 | 7.365 | 0.06 | 19 | 14 | 3 | 6
380.0874 | 0.5 | 1.3 | 2.5 | C3 H14 N11 O11 | 239.1 | 7.977 | 0.03 | 3 | 14 | 11 | 11
380.0888 | -0.9 | -2.4 | 7.5 | C4 H10 N15 O7 | 237.7 | 6.550 | 0.14 | 4 | 10 | 15 | 7
380.0869 | 1.0 | 2.6 | 20.5 | C16 H6 N13 | 235.1 | 4.000 | 1.83 | 16 | 6 | 13
380.0896 | -1.7 | -4.5 | 19.5 | C20 H10 N7 O2 | 238.3 | 7.130 | 0.08 | 20 | 10 | 7 | 2
20072020_063_8023
SS-362_20072020_003 334 (2.913) AM2 (Ar,30000.0,556.31,0.00,LS 1)
1: TOF MS ES+
2.72e+006
380.0879
381.0906
378.0711
352.0894
382.0916
HO
O
H
N
O2N
N
O
O
7m',
[M + H+]: 380.0877
Found: 380.0879
For Help, press F1
4:23 PM
7/21/2020


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1014 formula(e) evaluated with 262 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass | Calc. Mass | mDa | PPM | DBE | Formula | i-FIT | i-FIT Norm | Fit Conf % | C | H | N | O
378.0725 | 378.0729 | -0.4 | -1.0 | 15.5 | C19 H10 N3 O6 | 190.3 | 6.995 | 0.09 | 19 | 12 | 3 | 6
378.0731 | -0.6 | -1.6 | 8.5 | C4 H8 N15 O7 | 188.8 | 5.293 | 0.50 | 4 | 8 | 15 | 7
378.0718 | 0.7 | 1.9 | 3.5 | C3 H12 N11 O11 | 188.0 | 4.483 | 1.13 | 3 | 12 | 11 | 11
378.0713 | 1.2 | 3.2 | 21.5 | C16 H4 N13 | 191.0 | 7.508 | 0.05 | 16 | 4 | 13
378.0739 | -1.4 | -3.7 | 20.5 | C20 H8 N7 O2 | 190.8 | 7.354 | 0.06 | 20 | 8 | 7 | 2
378.0745 | -2.0 | -5.3 | 2.5 | C7 H16 N5 O13 | 188.3 | 4.799 | 0.82 | 7 | 16 | 5 | 13
22072020_063_8039
SS-366_22072020_002 354 (3.085) AM2 (Ar,30000.0,556.28,0.00,LS 1)
1: TOF MS ES+
3.26e+005
378.0725
380.0864
350.0745
HO
O
O2N
N
N
O
7m
[M + H+]: 378.0721
Found: 378.0725
For Help, press F1
2:38 PM
7/22/2020


Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
78 formula(e) evaluated with 25 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H O
381.2062 381.2066 -0.4 -1.0 10.5 C24 H29 O4 281.4 3.189 4.12 24 29 4
381.2125 -6.3 -16.5 1.5 C17 H33 O9 281.3 3.125 4.39 17 33 9
381.1913 14.9 39.1 6.5 C20 H29 O7 281.3 3.101 4.50 20 29 7
381.2218 -15.6 -40.9 14.5 C28 H29 O 281.5 3.334 3.57 28 29 1
381.1855 20.7 54.3 15.5 C27 H25 O2 281.5 3.255 3.86 27 25 2
381.2277 -21.5 -56.4 5.5 C21 H33 O6 281.4 3.154 4.27 21 33 6
15072020_061_7994
SS-348RM_15072020_012 400 (3.485) AM2 (Ar,30000.0,556.34,0.00,LS 1)
1: TOF MS ES+
2.00e+006
325.1406
219.1667
235.1621
381.2062
175.0295
293.0875
161.0615
326.1432
382.2093
236.1657
337.0812
527.2625
511.2676
77.0279
107.0387
403.1880
453.1128
471.1964
528.2673
O
O
H
O
OH
10
[M + H+]: 381.2060
Found: 381.2062

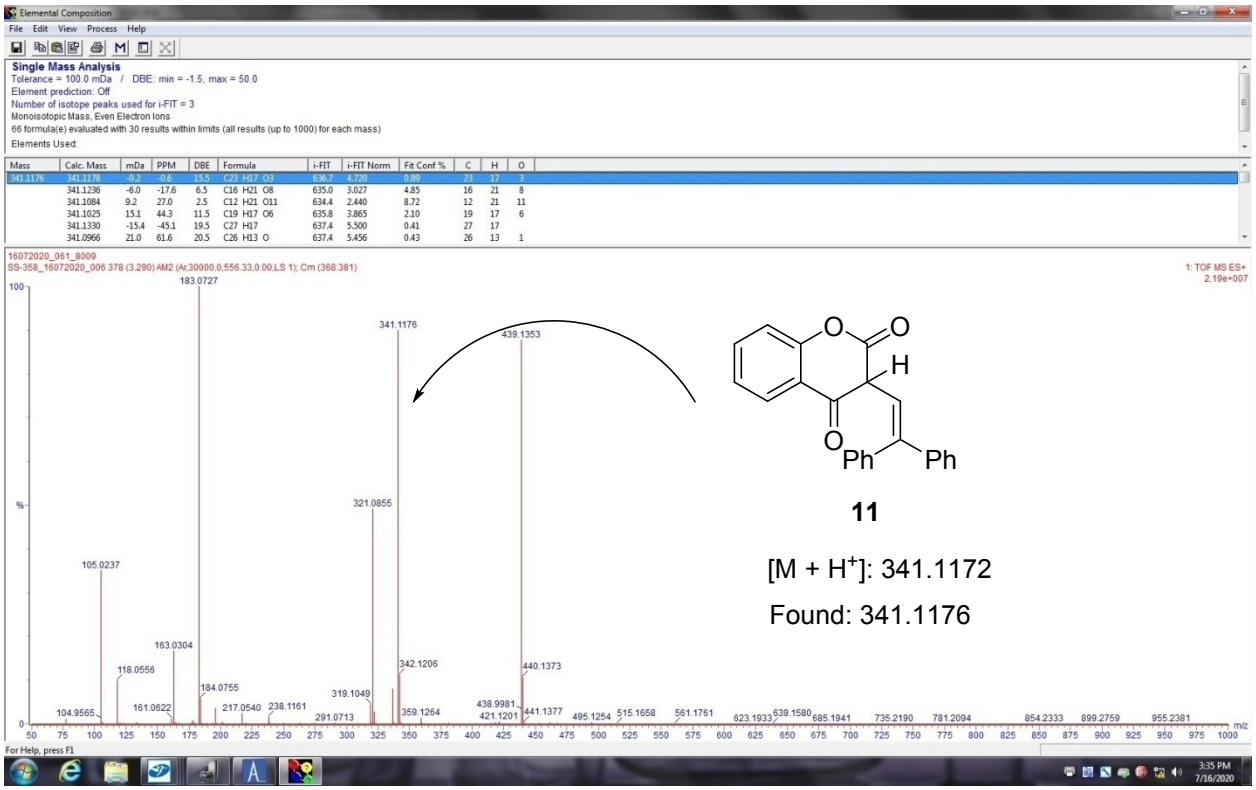

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 30 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H O
341.1176 341.1178 -0.2 -0.6 15.5 C23 H17 O3 636.7 4.720 0.89 23 17 3
341.1236 -6.0 -17.6 6.5 C16 H21 O8 635.0 3.027 4.85 16 21 8
341.1084 9.2 27.0 2.5 C12 H21 O11 634.4 2.440 8.72 12 21 11
341.1025 15.1 44.3 11.5 C19 H17 O6 635.8 3.865 2.10 19 17 6
341.1330 -15.4 -45.1 19.5 C27 H17 637.4 5.500 0.41 27 17
341.0966 21.0 61.6 20.5 C26 H13 O 637.4 5.456 0.43 26 13 1
16072020_061_8009
SS-358_16072020_006 378 (3.290) AM2 (Ar,30000.0,556.33,0.00,LS 1); Cm (368:381)
1: TOF MS ES+
2.19e+007
183.0727
341.1176
439.1353
321.0855
105.0237
163.0304
118.0556
342.1206
440.1373
184.0755
319.1049
104.9565
161.0622
217.0540
238.1161
359.1264
O
O
H
O
Ph
Ph
11
[M + H+]: 341.1172
Found: 341.1176

Table: Substrate Scope in Lipinski's rule of five

Compound	HRMS[a]	H-bond acceptor	H-bond donor	Log P[b]
3aa	321.0880	6	1	2.63
3ba	335.1039	6	1	3.01
3ca	397.1184	6	1	4.23
3da	375.1341	6	1	4.31
3ea	345.0873	6	1	2.79
3fa	425.1132	7	1	3.78
3ga	349.1184	6	1	3.44
3ha	389.0095	6	1	3.90
3ia	307.0717	6	2	2.12
3ja	335.1027	6	2	2.92
3ka	374.9937	6	2	3.38
3ib	321.0872	6	2	2.54
3ac	351.0983	7	1	2.67
3ad	355.0478	6	1	3.29
3lb	407.1244	8	1	3.30
5a	285.0871	6	1	1.55
5b	299.1028	6	1	1.93
5c	361.1186	6	1	3.14
5e	309.0873	6	1	1.71
5l	357.1082	8	1	1.79
5f	389.1135	7	1	2.70
5g	313.1182	6	1	2.35
5h	353.0091	6	1	2.81
5i	271.0714	6	2	1.03
5j	299.1021	6	2	1.84
5k	338.9918	6	2	2.29
7a	333.0870	6	1	2.29
7b	347.1029	6	1	2.67
7c	409.1205	6	1	3.88
7e	357.0867	6	1	2.45
7g	361.1183	6	1	3.09
7i	319.0710	6	2	1.77

[a] HRMS was recorded by electron spray ionization technique with a Q-TOF mass analyzer.

[b] Log P estimated by using Molinspiration software.