Electronic Supplementary Material (ESI) for RSC Advances.

Construction of Cyclopentane-fused Coumarins via DBU-Catalyzed [3 + 2] Cycloaddition of 3-homoacyl Coumarins with Cyclic 1-azadienes

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General Information

All reactions were performed under N_2 atmospheres in oven-dried glassware with magnetic stirring. Unless otherwise stated, all reagents were purchased from commercial suppliers. Organic solutions were concentrated under reduced pressure on a rotary evaporator. Reactions were monitored through thin layer chromatography (TLC) on silica gel–precoated glass plates. Chromatograms were visualized by fluorescence quenching with UV light at 254 nm. Flash column chromatography was performed using Qingdao Haiyang flash silica gel (200–300 mesh). ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ using a 400 MHz NMR instrument (referenced internally to Me_4Si). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet; d = doublet; q = quartet; m = multiplet; br = broad), coupling constant (Hz), and integral. Data for ^{13}C NMR spectra are reported in terms of chemical shift. Accurate mass measurements were performed using an Agilent instrument with the ESI-MS technique. Melting points were determined on a Stuard SMP3 melting apparatus. X-ray crystallographic data were collected using a Bruker APEX-II CCD.

General procedure for preparation of cyclic 1-azadienes 1[1] and 4[1a]

Cyclic 1-azadienes **1** and **4** were prepared by the reported procedure.

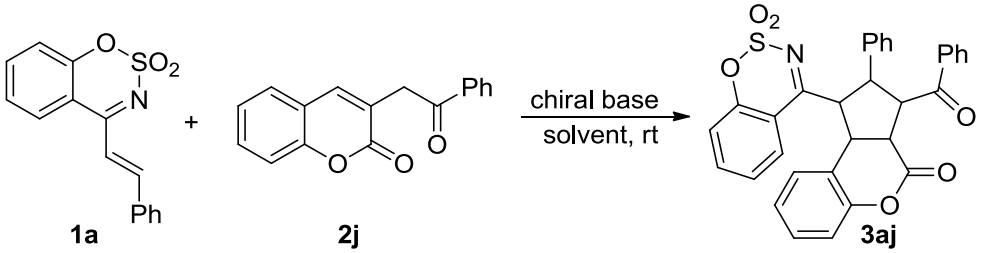
General procedure for preparation of 3-homoacyl coumarins 2[2]

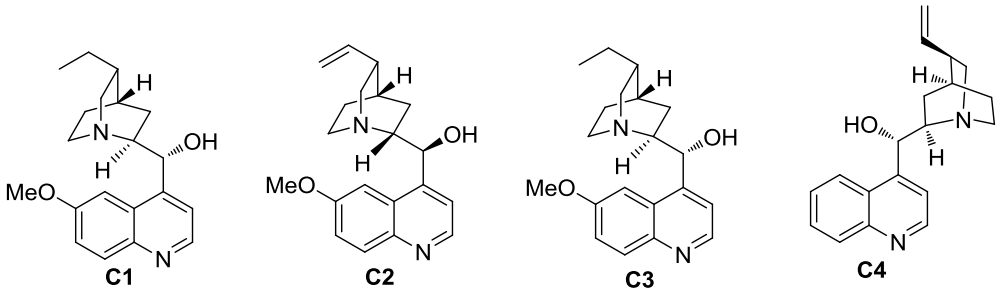
3-homoacyl coumarins **2** were prepared by the reported procedure.

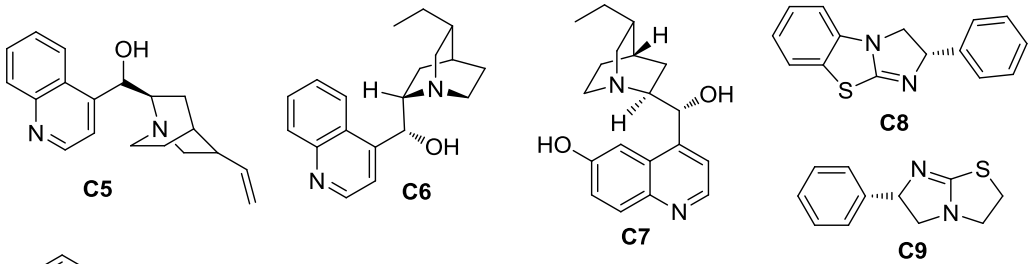
General Procedure for the [3+2] Cycloaddition Reaction of Cyclic 1-azadienes 1 or 4 with 3-homoacyl Coumarins 2

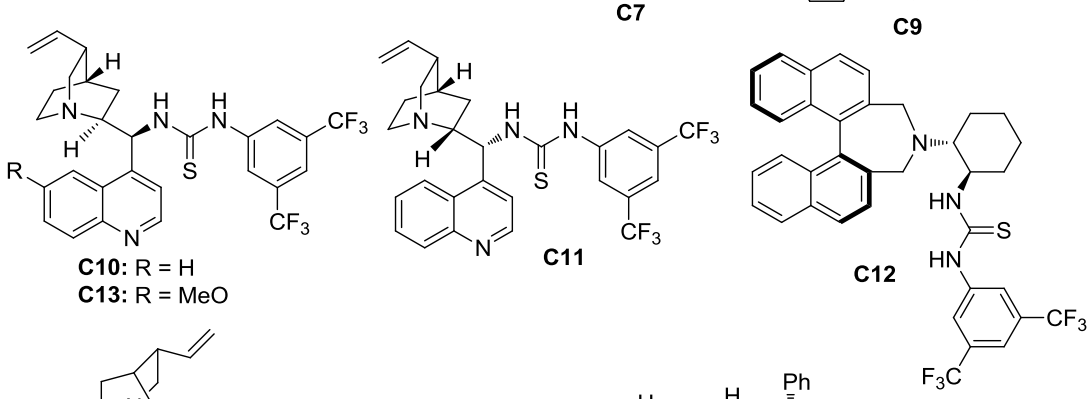
Under a nitrogen atmosphere, cyclic 1-azadiene **1** or **4** (0.1 mmol), 3-homoacyl coumarins **2** (0.12 mmol) and 20 mol% DBU were mixed in THF (2 mL). Then the reaction mixture was vigorously stirred at room temperature and monitored by TLC. After completion, the reaction mixture was centrated under reduced pressure and the mixture was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:2) to give the corresponding product **3** or **5**.

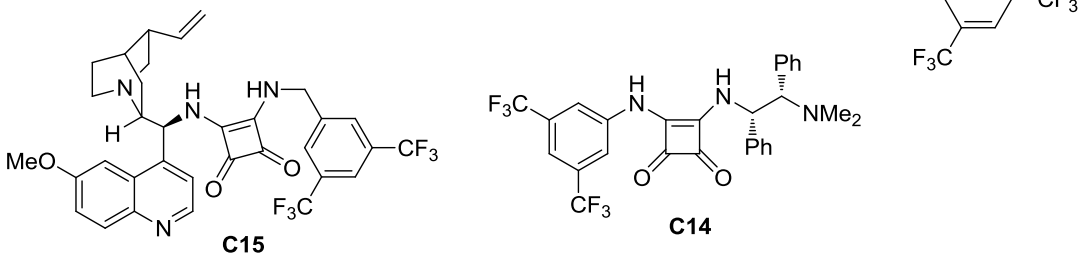
Table S1. Screening of enantioselective reaction conditions[a]

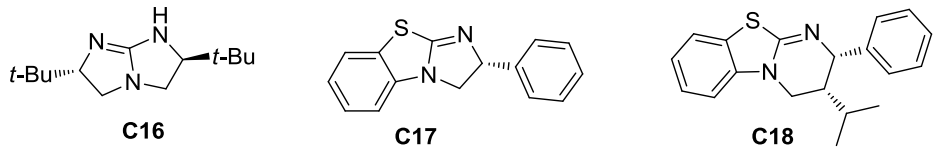












Entry	Catalyst	Solvent	Time(h)	Yield[b] (%)	dr[c]	ee[c] (%)
1	**C1**	CH_2Cl_2	120	0	-	-
2	**C2**	CH_2Cl_2	120	0	-	-
3	**C3**	CH_2Cl_2	120	0	-	-

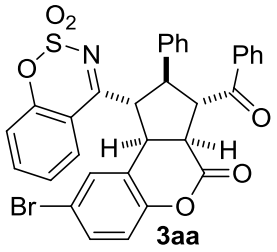
4	**C4**	CH_2Cl_2	120	0	-	-
5	**C5**	CH_2Cl_2	120	0	-	-
6	**C6**	CH_2Cl_2	120	0	-	-
7	**C7**	CH_2Cl_2	120	0	-	-
8	**C8**	CH_2Cl_2	120	0	-	-
9	**C9**	CH_2Cl_2	120	0	-	-
10	**C10**	CH_2Cl_2	120	0	-	-
11	**C11**	CH_2Cl_2	120	0	-	-
12	**C12**	CH_2Cl_2	120	0	-	-
13	**C13**	CH_2Cl_2	120	0	-	-
14	**C14**	CH_2Cl_2	120	0	-	-
15	**C15**	CH_2Cl_2	120	0	-	-
16	**C16**	CH_2Cl_2	120	0	-	-
17	**C17**	CH_2Cl_2	120	0	-	-
18	**C18**	CH_2Cl_2	120	0	-	-
19	**C1**	THF	120	0	-	-
20	**C2**	THF	120	0	-	-
21	**C3**	THF	120	0	-	-
22	**C4**	THF	120	0	-	-
23	**C5**	THF	120	0	-	-
24	**C6**	THF	120	0	-	-
25	**C7**	THF	120	0	-	-
26	**C8**	THF	120	0	-	-
27	**C9**	THF	120	0	-	-
28	**C10**	THF	120	0	-	-
29	**C11**	THF	120	0	-	-
30	**C12**	THF	120	0	-	-
31	**C13**	THF	120	0	-	-
32	**C14**	THF	120	0	-	-
33	**C15**	THF	120	0	-	-
34	**C16**	THF	120	0	-	-
35	**C17**	THF	120	0	-	-
36	**C18**	THF	120	0	-	-
37	**C1**	CH_3CN	120	46	>20:1	27.3
38	**C2**	CH_3CN	120	52.4	>20:1	9.5
39	**C3**	CH_3CN	120	60	>20:1	8
40	**C4**	CH_3CN	120	31	>20:1	0
41	**C5**	CH_3CN	120	46	>20:1	4
42	**C6**	CH_3CN	120	0	-	-
43	**C7**	CH_3CN	120	0	-	-
44	**C8**	CH_3CN	120	0	-	-
45	**C9**	CH_3CN	120	31	>20:1	4.5
46	**C10**	CH_3CN	120	0	-	-
47	**C11**	CH_3CN	120	41	>20:1	2

48	**C12**	CH_3CN	120	0	-	-
49	**C13**	CH_3CN	120	0	-	-
50	**C14**	CH_3CN	120	0	-	-
51	**C15**	CH_3CN	120	0	-	-
52	**C16**	CH_3CN	120	73	>20:1	11
53	**C17**	CH_3CN	120	0	-	-
54	**C18**	CH_3CN	120	36	>20:1	11

[a] Reactions were carried out with **1a** (0.1 mmol), **2j** (0.12mmol), and 20 mol% catalyst in 2 mL of CH_3CN at rt. [b] Isolated yields. [c] Determined by ^1H NMR. [d] Determined by HPLC analysis.

Characterization Data for the [3+2] Cycloaddition Reaction Products 3 and 5.

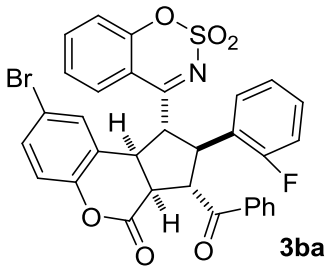
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3aa)



3aa

Orange solid (86%, 54.0mg), mp = 228 – 230 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.9 Hz, 2H), 7.49 – 7.34 (m, 2H), 7.31 – 7.19 (m, 4H), 7.17 – 7.01 (m, 6H), 6.91 (d, *J* = 8.7 Hz, 1H), 6.71 (t, *J* = 7.7 Hz, 1H), 6.54 (d, *J* = 8.1 Hz, 1H), 5.02 (d, *J* = 6.9 Hz, 1H), 4.11 (dd, *J* = 11.8, 7.7 Hz, 1H), 3.81 (dd, *J* = 10.9, 7.1 Hz, 2H), 3.62 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.2, 167.9, 152.4, 148.7, 138.4, 136.3, 133.5, 132.9, 131.6, 130.8, 128.4, 128.3, 127.6, 127.0, 126.9, 126.7, 124.2, 121.3, 117.8, 117.6, 116.7, 115.4, 57.9, 54.2, 54.0, 44.2, 44.1. HRMS (ESI) calcd for $C_{32}H_{22}BrNO_6SNa^+$ $[M+Na]^+$ 650.0243, found 650.0242.

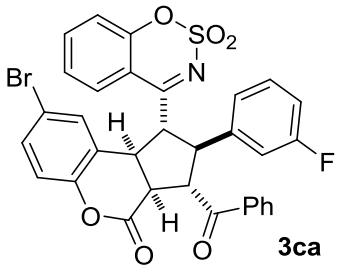
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(2-fluorophenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ba)



3ba

Yellow solid (85%, 54.9mg), mp = 193 – 195 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.9 Hz, 2H), 7.48 – 7.37 (m, 2H), 7.33 – 7.19 (m, 4H), 7.15 – 7.03 (m, 3H), 6.96 – 6.87 (m, 2H), 6.83 – 6.78 (m, 3H), 5.06 (d, *J* = 7.5 Hz, 1H), 4.15 – 4.01 (m, 2H), 3.94 (t, *J* = 11.2 Hz, 1H), 3.76 (d, *J* = 8.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.0, 167.5, 159.8 (d, *J* = 246.4 Hz), 152.5, 148.7, 136.4, 133.7, 132.9, 131.6, 130.9, 129.4 (d, *J* = 4.0 Hz), 128.9 (d, *J* = 8.5 Hz), 128.1, 127.7, 126.3, 124.8 (d, *J* = 12.3 Hz), 124.2, 124.2 (d, *J* = 3.2 Hz), 121.0, 117.9, 117.7, 116.6, 115.4, 115.1, 114.9, 54.7, 54.7, 52.9, 48.4, 44.0, 43.9. HRMS (ESI) calcd for $C_{32}H_{21}BrFNO_6SNa^+$ $[M+Na]^+$ 668.0149, found 668.0148.

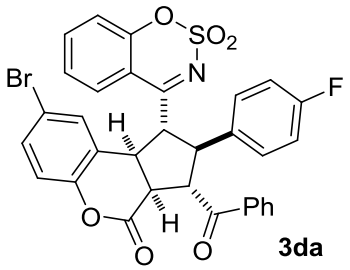
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(3-fluorophenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ca)



3ca

White solid (90%, 58.2mg), mp = 232 – 234 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.50 – 7.41 (m, 2H), 7.28 (t, *J* = 7.3 Hz, 3H), 7.17 – 7.06 (m, 3H), 6.96 – 6.73 (m, 5H), 6.63 (d, *J* = 8.1 Hz, 1H), 4.99 (d, *J* = 7.0 Hz, 1H), 4.05 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.91 (dd, *J* = 10.4, 7.1 Hz, 1H), 3.77 (d, *J* = 7.7 Hz, 1H), 3.62 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 176.9, 167.7, 162.1 (d, *J* = 248.0 Hz), 152.4, 148.6, 141.0 (d, *J* = 7.1 Hz), 136.6, 133.4, 133.0, 131.7, 130.8, 130.0 (d, *J* = 8.5 Hz), 128.2, 127.7, 126.5, 124.3, 122.8 (d, *J* = 2.8 Hz), 120.9, 118.0, 117.7, 116.7, 115.4, 114.1, 113.9 (d, *J* = 6.2 Hz), 113.6, 57.4, 53.7, 53.1, 44.4, 44.3. HRMS (ESI) calcd for $C_{32}H_{21}BrFNO_6SNa^+$ $[M+Na]^+$ 668.0149, found 668.0148.

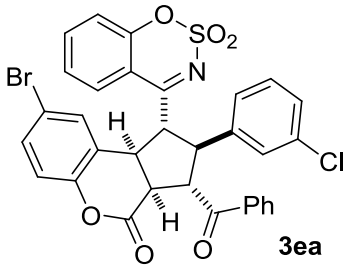
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(4-fluorophenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3da)



3da

White solid (85%, 55.0mg), mp = 245 – 247 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.32 – 7.23 (m, 3H), 7.18 – 7.01 (m, 4H), 6.95 – 6.71 (m, 4H), 6.59 (d, *J* = 8.0 Hz, 1H), 4.97 (d, *J* = 6.9 Hz, 1H), 4.05 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.86 (dd, *J* = 10.4, 7.1 Hz, 1H), 3.78 (d, *J* = 7.7 Hz, 1H), 3.59 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.3, 177.0, 167.9, 161.1 (d, *J* = 247.8 Hz), 152.4, 148.6, 136.5, 134.2 (d, *J* = 3.1 Hz), 133.4, 133.0, 131.7, 130.8, 128.6 (d, *J* = 8.1 Hz), 128.2, 127.7, 126.5, 124.3, 121.0, 118.0, 117.6, 116.7, 115.4, 115.3, 115.2, 57.7, 53.9, 53.0, 44.3, 44.2. HRMS (ESI) calcd for $C_{32}H_{21}BrFNO_6SNa^+$ $[M+Na]^+$ 668.0149, found 668.0148.

3-benzoyl-8-bromo-2-(3-chlorophenyl)-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ea)

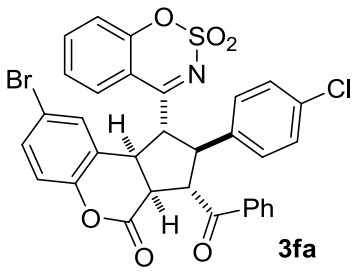


3ea

Orange solid (86%, 57.0mg), mp = 239 – 241 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.8 Hz, 2H), 7.52 – 7.42 (m, 2H), 7.33 – 7.24 (m, 3H), 7.17 – 7.04 (m, 5H), 7.01 – 6.88 (m, 2H), 6.79 (t, *J* = 7.7 Hz, 1H), 6.64 (d, *J* = 8.1 Hz, 1H), 4.97 (d, *J* = 6.9 Hz, 1H), 4.03 (dd, *J* = 11.6, 7.9 Hz, 1H), 3.91 (dd, *J* = 10.2, 7.2 Hz, 2H), 3.76 (d, *J* = 7.7 Hz, 1H), 3.61 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 176.8, 167.7, 152.4, 148.6, 140.5, 136.6, 134.2, 133.4, 133.1, 131.7, 130.8, 129.6, 128.2, 127.7, 127.2, 126.9, 126.5, 125.2, 124.2, 120.9, 118.0, 117.7, 116.7, 115.3, 57.3, 53.7, 52.8, 44.3. HRMS (ESI) calcd for $C_{32}H_{21}BrClNO_6SNa^+$ $[M+Na]^+$ 683.9854, found 683.9851.

3-benzoyl-8-bromo-2-(4-chlorophenyl)-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-1,3,3a,9b-t

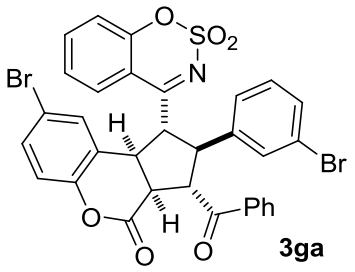
etrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3fa)



3fa

Yellow solid (80%, 53.0mg), mp = 72 – 74 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.7 Hz, 2H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.29 (t, *J* = 7.8 Hz, 3H), 7.17 – 7.09 (m, 4H), 7.04 (d, *J* = 7.9 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 1H), 6.79 (t, *J* = 7.7 Hz, 1H), 6.59 (d, *J* = 8.0 Hz, 1H), 4.96 (d, *J* = 6.9 Hz, 1H), 4.03 (dd, *J* = 11.9, 7.7 Hz, 1H), 3.89 (dd, *J* = 10.6, 7.0 Hz, 1H), 3.78 (d, *J* = 7.7 Hz, 1H), 3.58 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.3, 176.8, 167.9, 152.4, 148.6, 137.0, 136.6, 133.4, 133.0, 132.8, 131.7, 130.8, 128.5, 128.3, 128.2, 127.7, 126.5, 124.3, 120.9, 118.0, 117.7, 116.8, 115.3, 57.6, 53.7, 52.8, 44.5, 44.3. HRMS (ESI) calcd for $C_{32}H_{21}BrClNO_6SNa^+$ $[M+Na]^+$ 683.9854, found 683.9853.

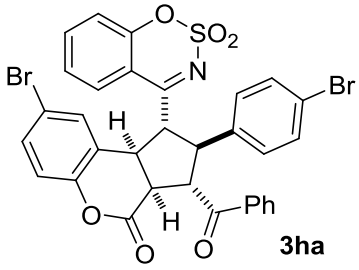
3-benzoyl-8-bromo-2-(3-bromophenyl)-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-1,3,3a,9b-t etrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ga)



3ga

Orange solid (79%, 55.9mg), mp = 233 – 235 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.54 – 7.42 (m, 2H), 7.34 – 7.22 (m, 5H), 7.19 – 7.08 (m, 2H), 7.06 – 7.00 (m, 2H), 6.92 (d, *J* = 8.7 Hz, 1H), 6.79 (t, *J* = 7.7 Hz, 1H), 6.64 (d, *J* = 8.1 Hz, 1H), 4.97 (d, *J* = 7.0 Hz, 1H), 4.03 (dd, *J* = 11.8, 7.7 Hz, 1H), 3.90 (dd, *J* = 10.6, 7.0 Hz, 1H), 3.76 (d, *J* = 7.8 Hz, 1H), 3.61 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 176.8, 167.7, 152.4, 148.6, 140.7, 136.6, 133.4, 133.1, 131.7, 130.9, 130.1, 129.9, 129.9, 128.2, 127.8, 126.5, 125.7, 124.2, 122.3, 120.9, 118.0, 117.7, 116.8, 115.3, 57.3, 53.6, 52.8, 44.3, 44.2. HRMS (ESI) calcd for $C_{32}H_{21}Br_2NO_6SNa^+$ $[M+Na]^+$ 727.9349, found 727.9345.

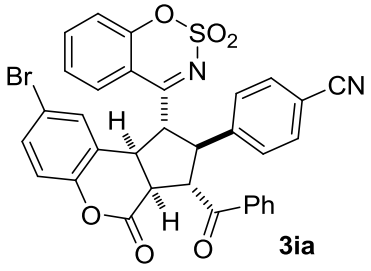
3-benzoyl-8-bromo-2-(4-bromophenyl)-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ha)



3ha

Orange solid (80%, 56.6mg), mp = 231 – 233 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.9 Hz, 2H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.33 – 7.24 (m, 5H), 7.16 – 7.09 (m, 2H), 6.98 (d, *J* = 7.9 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 1H), 6.80 (t, *J* = 7.7 Hz, 1H), 6.59 (d, *J* = 8.1 Hz, 1H), 4.96 (d, *J* = 6.9 Hz, 1H), 4.02 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.89 (dd, *J* = 10.3, 7.1 Hz, 1H), 3.78 (d, *J* = 7.7 Hz, 1H), 3.58 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 176.8, 167.8, 152.4, 148.6, 137.5, 136.6, 133.4, 133.1, 131.7, 131.5, 130.8, 128.6, 128.2, 127.7, 126.5, 124.3, 120.9, 120.9, 118.0, 117.7, 116.7, 115.3, 57.5, 53.7, 52.8, 44.6, 44.3. HRMS (ESI) calcd for $C_{32}H_{21}Br_2NO_6SNa^+$ $[M+Na]^+$ 727.9349, found 727.9345.

4-(3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-4-oxo-1,2,3,3a,4,9b-hexahydrocyclopenta[*c*]chromen-2-yl)benzonitrile (3ia)

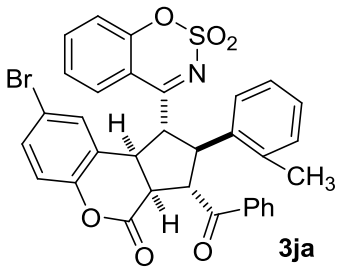


3ia

Yellow solid (75%, 49.0mg), mp = 62 – 64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.9 Hz, 2H), 7.54 – 7.40 (m, 4H), 7.35 – 7.23 (m, 5H), 7.15 (d, *J* = 8.3 Hz, 1H), 7.06 (s, 1H), 6.93 (d, *J* = 8.7 Hz, 1H), 6.80 (t, *J* = 7.7 Hz, 1H), 6.60 (d, *J* = 8.1 Hz, 1H), 4.99 (d, *J* = 7.0 Hz, 1H), 4.13 (dd, *J* = 10.5, 7.1 Hz, 1H), 3.99 (dd, *J* = 11.7, 7.7 Hz, 1H), 3.76 (d, *J* = 7.7 Hz, 1H), 3.62 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.9, 176.3, 167.6, 152.5, 148.6, 144.0, 136.9, 133.4, 133.2, 132.1, 131.9, 130.8, 128.1, 127.9, 126.2, 124.4, 120.5, 118.2, 117.7, 117.1, 116.8, 115.3, 110.9, 57.0, 53.4,

52.4, 45.0, 44.6. HRMS (ESI) calcd for $C_{33}H_{21}BrN_2O_6SNa^+$ $[M+Na]^+$ 675.0196 found 675.0193.

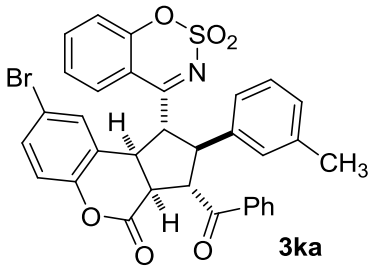
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(o-tolyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ja)



3ja

White solid (79%, 50.8mg), mp = 190 – 192 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.54 – 7.36 (m, 3H), 7.34 – 7.18 (m, 5H), 7.07 (d, *J* = 8.3 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 8.2 Hz, 1H), 6.74 – 6.68 (m, 2H), 6.52 (d, *J* = 8.1 Hz, 1H), 4.96 (d, *J* = 7.1 Hz, 1H), 4.17 (dd, *J* = 11.8, 7.9 Hz, 1H), 4.07 (dd, *J* = 10.4, 7.2 Hz, 1H), 3.87 (d, *J* = 7.9 Hz, 1H), 3.63 (t, *J* = 11.2 Hz, 1H), 1.57 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.3, 168.1, 152.4, 148.7, 137.0, 136.4, 136.2, 133.6, 132.8, 131.6, 130.9, 129.6, 128.1, 127.6, 126.8, 126.7, 126.7, 125.6, 124.2, 121.3, 117.8, 117.6, 116.7, 115.2, 58.4, 55.4, 49.4, 43.8, 43.7, 18.1. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_6SNa^+$ $[M+Na]^+$ 664.0400 found 664.0399.

3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(m-tolyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ka)

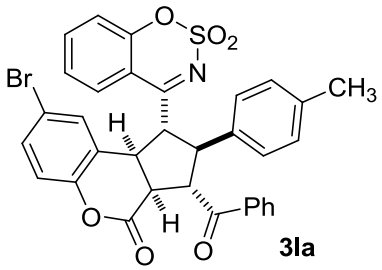


3ka

Orange solid (76%, 48.8mg), mp = 226 – 228 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.0 Hz, 2H), 7.49 – 7.36 (m, 2H), 7.31 – 7.19 (m, 4H), 7.12 – 7.00 (m, 2H), 6.95 – 6.86 (m, 3H), 6.80 (s, 1H), 6.72 (t, *J* = 7.7 Hz, 1H), 6.56 (d, *J* = 8.1 Hz, 1H), 5.00 (d, *J* = 6.9 Hz, 1H), 4.09 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.91 – 3.70 (m, 2H), 3.61 (t, *J* = 11.1 Hz, 1H), 2.11 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 177.3, 167.9, 152.4, 148.7, 138.3, 138.1, 136.2, 133.5, 132.8, 131.5, 130.9, 128.3, 128.3,

127.7, 127.6, 127.6, 126.8, 124.1, 123.7, 121.3, 117.8, 117.6, 116.7, 115.5, 57.8, 54.1, 54.0, 44.2, 44.1, 20.3. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_6SNa^+$ $[M+Na]^+$ 664.0400 found 664.0399.

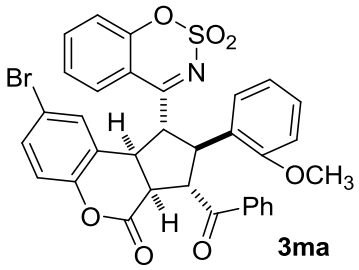
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(p-tolyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3la)



3la

Yellow solid (73%, 46.9mg), mp = 201 – 203 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.0 Hz, 2H), 7.43 (q, *J* = 8.0 Hz, 2H), 7.30 – 7.22 (m, 3H), 7.08 (d, *J* = 8.3 Hz, 1H), 6.98 – 6.87 (m, 5H), 6.72 (t, *J* = 7.7 Hz, 1H), 6.55 (d, *J* = 8.1 Hz, 1H), 4.99 (d, *J* = 6.9 Hz, 1H), 4.08 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.86 – 3.72 (m, 2H), 3.60 (t, *J* = 11.1 Hz, 1H), 2.19 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 177.3, 168.0, 152.4, 148.7, 136.7, 136.3, 135.3, 133.5, 132.8, 131.5, 130.8, 129.0, 128.3, 127.6, 126.8, 126.7, 124.1, 121.3, 117.8, 117.6, 116.7, 115.4, 58.0, 54.0, 53.8, 44.2, 44.2, 20.0. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_6SNa^+$ $[M+Na]^+$ 664.0400 found 664.0399.

3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(2-methoxyphenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ma)

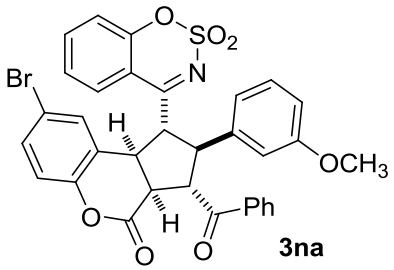


3ma

Orange solid (67%, 44.1mg), mp = 56 – 58 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.40 (q, *J* = 8.1 Hz, 2H), 7.35 – 7.18 (m, 4H), 7.12 – 7.02 (m, 2H), 6.93 – 6.86 (m, 2H), 6.76 – 6.58 (m, 4H), 5.09 (d, *J* = 7.2 Hz, 1H), 4.22 – 4.03 (m, 2H), 3.92 (dd, *J* = 9.8, 7.5 Hz, 1H), 3.78 (d, *J* = 7.7 Hz, 1H), 3.61 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.2, 177.9, 167.9, 155.9, 152.3, 148.8, 134.0, 133.9, 132.6, 131.3, 131.0, 129.1, 128.4, 128.3, 127.5, 126.5, 125.3, 124.0, 121.9, 120.3, 117.,

117.56, 116.6, 115.6, 109.7, 54.1, 53.9, 52.7, 50.8, 43.7, 43.1. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_7SNa^+$ $[M+Na]^+$ 680.0349 found 680.0348.

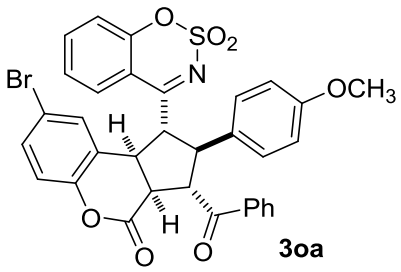
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(3-methoxyphenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3na)



3na

Orange solid (75%, 49.4mg), mp = 93 – 94 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.44 (q, *J* = 7.5 Hz, 2H), 7.31 – 7.21 (m, 3H), 7.13 – 7.04 (m, 2H), 6.91 (d, *J* = 8.7 Hz, 1H), 6.80 – 6.68 (m, 2H), 6.65 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.62 – 6.51 (m, 2H), 5.03 (d, *J* = 6.8 Hz, 1H), 4.08 (dd, *J* = 11.8, 7.8 Hz, 1H), 3.85 – 3.76 (m, 2H), 3.67 – 3.56 (m, 1H), 3.57 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.3, 167.9, 159.2, 152.4, 148.7, 140.0, 136.4, 133.5, 132.9, 131.6, 130.8, 129.5, 128.3, 127.6, 126.7, 124.2, 121.2, 118.7, 117.8, 117.6, 116.7, 115.5, 112.9, 112.1, 57.8, 54.2, 54.1, 53.7, 44.2, 44.2. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_7SNa^+$ $[M+Na]^+$ 680.0349 found 680.0347.

3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(4-methoxyphenyl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3oa)

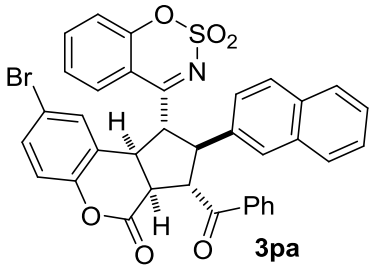


3oa

Yellow solid (77%, 50.7mg), mp = 205 – 207 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.9 Hz, 2H), 7.43 (q, *J* = 7.6, 7.2 Hz, 2H), 7.31 – 7.21 (m, 4H), 7.09 (d, *J* = 8.3 Hz, 1H), 6.99 (d, *J* = 8.2 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 1H), 6.76 (t, *J* = 7.7 Hz, 1H), 6.67 (d, *J* = 8.1 Hz, 2H), 6.58 (d, *J* = 8.1 Hz, 1H), 4.97 (d, *J* = 6.9 Hz, 1H), 4.08 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.79 (d, *J* = 7.8 Hz, 1H), 3.75 (dd, *J* =

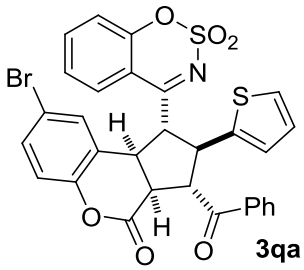
10.3, 7.1 Hz, 1H), 3.66 (s, 3H), 3.59 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 177.4, 168.0, 158.1, 152.4, 148.7, 136.3, 133.5, 132.9, 131.5, 130.8, 130.2, 128.3, 128.0, 127.6, 126.8, 124.2, 121.3, 117.8, 117.6, 116.7, 115.4, 113.6, 58.0, 54.3, 54.1, 53.6, 44.1, 44.1. HRMS (ESI) calcd for $C_{33}H_{24}BrNO_7SNa^+$ $[M+Na]^+$ 680.0349 found 680.0348.

3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(naphthalen-2-yl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3pa)



Orange solid (72%, 48.8mg), mp = 122 – 124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.2 Hz, 3H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.46 – 7.21 (m, 7H), 7.17 (d, *J* = 8.5 Hz, 3H), 7.02 (d, *J* = 8.3 Hz, 1H), 6.93 (d, *J* = 8.7 Hz, 1H), 6.55 – 6.44 (m, 2H), 5.13 (d, *J* = 6.9 Hz, 1H), 4.13 (dd, *J* = 11.8, 7.7 Hz, 1H), 4.02 (dd, *J* = 10.4, 6.9 Hz, 1H), 3.86 (d, *J* = 7.8 Hz, 1H), 3.74 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.2, 168.0, 152.3, 148.7, 136.3, 135.4, 133.5, 132.9, 132.2, 131.6, 130.8, 128.7, 128.3, 127.6, 126.8, 126.6, 126.5, 125.6, 125.4, 124.0, 123.6, 121.2, 117.7, 117.7, 116.7, 115.4, 57.6, 54.1, 53.8, 44.4, 44.3. HRMS (ESI) calcd for $C_{36}H_{24}BrNO_6SNa^+$ $[M+Na]^+$ 700.0400, found 700.0398.

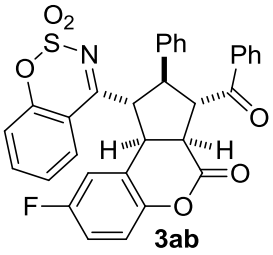
3-benzoyl-8-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-(thiophen-2-yl)-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3qa)



Orange solid (74%, 46.9mg), mp = 228 – 230 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.0 Hz, 2H), 7.55 – 7.42 (m, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.27 (dd, *J* = 8.8, 2.3 Hz, 1H), 7.14 (d, *J* = 8.3 Hz,

2H), 7.08 – 7.02 (m, 1H), 6.94 – 6.83 (m, 2H), 6.80 – 6.67 (m, 3H), 5.05 (d, *J* = 7.1 Hz, 1H), 4.27 (dd, *J* = 10.3, 7.1 Hz, 1H), 4.06 (dd, *J* = 11.8, 7.9 Hz, 1H), 3.80 – 3.61 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 177.0, 167.5, 152.5, 148.7, 141.7, 136.5, 133.6, 133.0, 131.7, 130.7, 128.3, 127.8, 126.5, 125.6, 124.4, 124.3, 120.9, 118.0, 117.7, 116.7, 115.7, 58.1, 55.1, 48.2, 44.5, 44.2. HRMS (ESI) calcd for $C_{30}H_{20}BrNO_6S_2Na^+$ $[M+Na]^+$ 655.9808, found 655.9806.

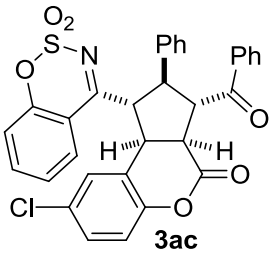
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-8-fluoro-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3ab)



3ab

Orange solid (88%, 49.9mg), mp = 237 – 239 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.9 Hz, 2H), 7.48 – 7.34 (m, 2H), 7.27 – 7.21 (m, 2H), 7.17 – 6.95 (m, 7H), 6.92 – 6.79 (m, 2H), 6.72 (t, *J* = 7.7 Hz, 1H), 6.54 (d, *J* = 8.1 Hz, 1H), 5.04 (d, *J* = 6.9 Hz, 1H), 4.14 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.92 – 3.74 (m, 2H), 3.64 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 177.4, 168.2, 158.1 (d, *J* = 246.1 Hz), 152.4, 145.7 (d, *J* = 2.4 Hz), 138.4, 136.3, 133.6, 132.9, 128.4, 128.3, 127.6, 127.0, 126.9, 126.8, 124.2, 120.8 (d, *J* = 8.0 Hz), 117.8, 117.4 (d, *J* = 8.6 Hz), 115.6, 115.4, 114.6, 114.4, 57.9, 54.6, 54.0, 44.4, 44.2. HRMS (ESI) calcd for $C_{32}H_{22}FNO_6SNa^+$ $[M+Na]^+$ 590.1044 found 590.1047.

3-benzoyl-8-chloro-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3ac)

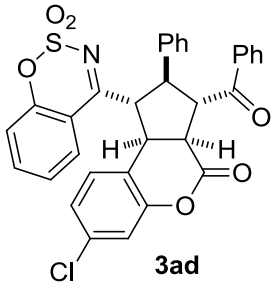


3ac

Orange solid (78%, 45.6mg), mp = 228 – 230 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.0 Hz,

2H), 7.56 – 7.35 (m, 2H), 7.29 – 7.20 (m, 2H), 7.17 – 7.03 (m, 8H), 6.97 (d, *J* = 8.7 Hz, 1H), 6.71 (t, *J* = 7.7 Hz, 1H), 6.54 (d, *J* = 8.1 Hz, 1H), 5.03 (d, *J* = 6.9 Hz, 1H), 4.12 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.90 – 3.75 (m, 2H), 3.62 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.2, 168.0, 152.5, 148.2, 138.4, 136.3, 133.5, 132.9, 129.3, 128.6, 128.4, 128.3, 127.9, 127.6, 127.0, 126.9, 126.7, 124.2, 120.9, 117.8, 117.3, 115.4, 57.9, 54.4, 54.0, 44.2, 44.2. HRMS (ESI) calcd for $C_{32}H_{22}ClNO_6SNa^+$ $[M+Na]^+$ 606.0749 found 606.0749.

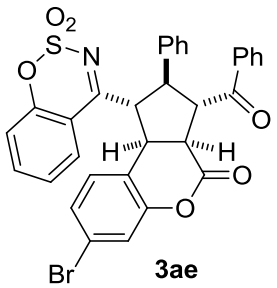
3-benzoyl-7-chloro-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3ad)



3ad

Orange solid (84%, 49.1mg), mp = 190 – 192 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.9 Hz, 2H), 7.50 – 7.35 (m, 2H), 7.29 – 7.20 (m, 2H), 7.17 – 7.00 (m, 8H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.74 (t, *J* = 7.7 Hz, 1H), 6.53 (d, *J* = 8.1 Hz, 1H), 5.03 (d, *J* = 7.0 Hz, 1H), 4.18 (dd, *J* = 11.7, 7.9 Hz, 1H), 3.89 – 3.72 (m, 2H), 3.61 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 178.5, 169.0, 153.5, 151.1, 139.4, 137.3, 135.0, 134.6, 133.9, 130.0, 129.5, 129.4, 128.7, 128.0, 127.9, 125.5, 125.4, 118.9, 118.8, 117.4, 116.5, 59.1, 55.7, 55.1, 45.4, 45.1. HRMS (ESI) calcd for $C_{32}H_{22}ClNO_6SNa^+$ $[M+Na]^+$ 606.0749 found 606.0750.

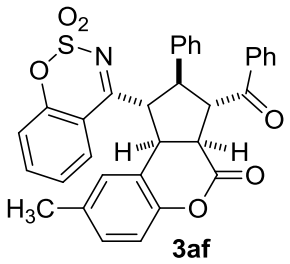
3-benzoyl-7-bromo-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3ae)



3ae

Orange solid (86%, 54.0mg), mp = 197 – 199 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.48 – 7.34 (m, 2H), 7.29 – 7.20 (m, 2H), 7.17 – 6.96 (m, 8H), 6.74 (t, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 8.1 Hz, 1H), 5.03 (d, *J* = 7.0 Hz, 1H), 4.17 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.95 – 3.74 (m, 2H), 3.61 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 177.4, 167.9, 152.4, 150.1, 138.3, 136.3, 133.5, 132.9, 129.2, 128.4, 128.3, 127.6, 127.3, 127.0, 126.8, 124.3, 121.5, 119.2, 118.3, 117.8, 115.4, 58.0, 54.7, 54.0, 44.3, 44.1. HRMS (ESI) calcd for $C_{32}H_{22}BrNO_6SNa^+$ $[M+Na]^+$ 650.0243 found 650.0243.

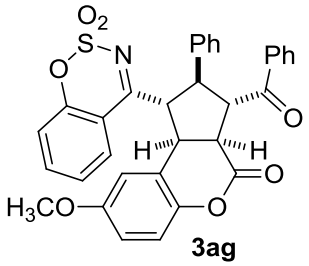
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-8-methyl-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3af)



3af

White solid (87%, 49.0mg), mp = 220 – 222 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.9 Hz, 2H), 7.52 – 7.31 (m, 2H), 7.29 – 7.20 (m, 2H), 7.16 – 7.01 (m, 6H), 6.98 – 6.87 (m, 2H), 6.84 (s, 1H), 6.70 (t, *J* = 7.7 Hz, 1H), 6.55 (d, *J* = 8.1 Hz, 1H), 5.03 (d, *J* = 7.0 Hz, 1H), 4.07 (dd, *J* = 11.8, 7.7 Hz, 1H), 3.90 (dd, *J* = 10.5, 6.9 Hz, 1H), 3.76 (d, *J* = 7.8 Hz, 1H), 3.63 (t, *J* = 11.2 Hz, 1H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 177.7, 168.7, 152.4, 147.5, 138.7, 136.0, 133.9, 133.7, 132.8, 129.0, 128.4, 128.3, 128.3, 127.6, 126.9, 126.8, 124.1, 118.7, 117.6, 115.6, 115.6, 58.1, 54.1, 54.0, 44.9, 44.8, 19.5. HRMS (ESI) calcd for $C_{33}H_{25}NO_6SNa^+$ $[M+Na]^+$ 586.1295 found 586.1297.

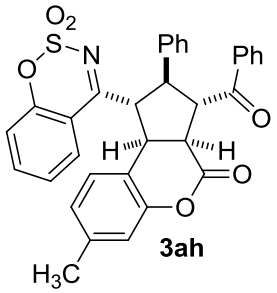
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-8-methoxy-2-phenyl-1,3,3a,9b-tetrahyd rocyclopenta[*c*]chromen-4(2*H*)-one (3ag)



3ag

White solid (94%, 54.5mg), mp = 203 – 205 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.47 – 7.34 (m, 2H), 7.30 – 7.20 (m, 2H), 7.17 – 7.01 (m, 6H), 6.94 (d, *J* = 8.9 Hz, 1H), 6.77 – 6.65 (m, 2H), 6.64 – 6.58 (m, 1H), 6.55 (d, *J* = 8.1 Hz, 1H), 5.04 (d, *J* = 7.0 Hz, 1H), 4.14 (dd, *J* = 11.7, 7.9 Hz, 1H), 3.87 (dd, *J* = 10.4, 7.1 Hz, 1H), 3.75 (d, *J* = 7.9 Hz, 1H), 3.64 (t, *J* = 11.2 Hz, 1H), 3.53 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 177.8, 168.6, 155.5, 152.4, 143.4, 138.6, 136.2, 133.7, 132.8, 128.3, 128.3, 127.6, 126.9, 126.9, 124.3, 119.6, 117.7, 117.0, 115.8, 115.5, 110.7, 58.0, 54.6, 54.3, 54.2, 45.1, 44.5. HRMS (ESI) calcd for $C_{33}H_{25}NO_7SNa^+$ $[M+Na]^+$ 602.1244 found 602.1246.

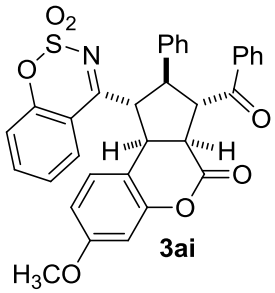
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-7-methyl-2-phenyl-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ah)



3ah

Orange solid (86%, 48.5mg), mp = 186 – 188 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.49 – 7.32 (m, 2H), 7.29 – 7.19 (m, 2H), 7.17 – 7.05 (m, 5H), 7.03 (d, *J* = 8.3 Hz, 1H), 6.95 (d, *J* = 7.7 Hz, 1H), 6.83 (s, 1H), 6.70 (t, *J* = 8.3 Hz, 2H), 6.53 (d, *J* = 8.1 Hz, 1H), 5.04 (d, *J* = 7.0 Hz, 1H), 4.12 (dd, *J* = 11.7, 7.8 Hz, 1H), 3.89 (dd, *J* = 10.5, 7.1 Hz, 1H), 3.76 (d, *J* = 7.8 Hz, 1H), 3.62 (t, *J* = 11.2 Hz, 1H), 2.20 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 177.9, 168.7, 152.4, 149.5, 139.0, 138.7, 136.0, 133.7, 132.8, 128.3, 128.3, 127.6, 127.6, 126.9, 126.9, 126.8, 125.0, 124.2, 117.7, 116.2, 116.0, 115.6, 58.2, 54.3, 54.0, 44.8, 44.7, 20.1. HRMS (ESI) calcd for $C_{33}H_{25}NO_6SNa^+$ $[M+Na]^+$ 586.1295 found 586.1298.

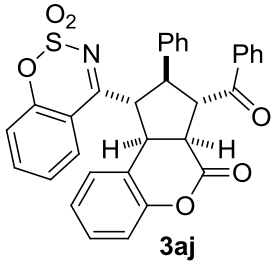
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-7-methoxy-2-phenyl-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3ai)



3ai

Yellow solid (83%, 48.1mg), mp = 231 – 233 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.53 – 7.33 (m, 2H), 7.29 – 7.20 (m, 2H), 7.17 – 7.05 (m, 5H), 7.04 (d, *J* = 8.3 Hz, 1H), 6.96 (d, *J* = 8.5 Hz, 1H), 6.73 (t, *J* = 7.7 Hz, 1H), 6.57 (d, *J* = 5.5 Hz, 1H), 6.55 (s, 1H), 6.43 (dd, *J* = 8.9, 2.5 Hz, 1H), 5.02 (d, *J* = 7.1 Hz, 1H), 4.10 (dd, *J* = 11.8, 7.8 Hz, 1H), 3.89 (dd, *J* = 10.6, 7.1 Hz, 1H), 3.76 (d, *J* = 7.9 Hz, 1H), 3.65 (s, 3H), 3.61 (t, *J* = 11.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 178.0, 168.6, 159.5, 152.4, 150.5, 138.7, 136.1, 133.7, 132.8, 128.6, 128.3, 128.3, 127.6, 127.0, 126.9, 126.8, 124.2, 117.7, 115.6, 110.9, 109.9, 101.6, 58.3, 54.5, 54.2, 53.9, 44.8, 44.6. HRMS (ESI) calcd for $C_{33}H_{25}NO_7SNa^+$ $[M+Na]^+$ 602.1244 found 602.1246.

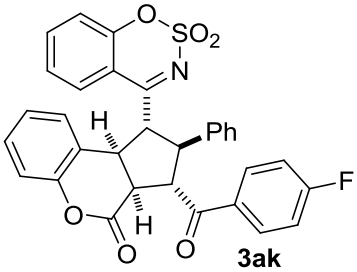
3-benzoyl-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3aj)



3aj

Orange solid (83%, 46.7mg), mp = 187 – 189 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.40 (dt, *J* = 24.6, 7.5 Hz, 2H), 7.25 (t, *J* = 7.7 Hz, 2H), 7.18 – 6.99 (m, 9H), 6.88 (t, *J* = 7.5 Hz, 1H), 6.70 (t, *J* = 7.7 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 1H), 5.04 (d, *J* = 7.0 Hz, 1H), 4.14 (dd, *J* = 11.8, 7.8 Hz, 1H), 3.91 (dd, *J* = 10.5, 7.1 Hz, 1H), 3.80 (d, *J* = 7.8 Hz, 1H), 3.65 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 177.7, 168.5, 152.4, 149.7, 138.6, 136.1, 133.7, 132.8, 128.6, 128.3, 128.3, 128.0, 127.6, 126.9, 126.8, 124.2, 119.1, 117.7, 115.9, 115.6, 58.1, 54.2, 54.1, 45.0, 44.8. HRMS (ESI) calcd for $C_{32}H_{23}NO_6SNa^+$ $[M+Na]^+$ 572.1138, found 572.1141.

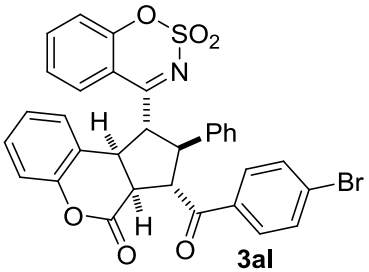
1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-3-(4-fluorobenzoyl)-2-phenyl-1,3,3a,9b-tetrahydro cyclopenta[*c*]chromen-4(2*H*)-one (3ak)



3ak

Orange solid (90%, 51.1mg), mp = 173 – 175 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.72 (m, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.19 – 7.00 (m, 9H), 6.97 – 6.80 (m, 3H), 6.71 (t, *J* = 7.7 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 1H), 4.99 (d, *J* = 7.1 Hz, 1H), 4.13 (dd, *J* = 11.8, 7.8 Hz, 1H), 3.87 (dd, *J* = 10.5, 7.2 Hz, 1H), 3.80 (dd, *J* = 7.8, 1.6 Hz, 1H), 3.66 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.2, 177.6, 168.5, 165.1 (d, *J* = 256.4 Hz), 152.4, 149.6, 138.4, 136.1, 131.1 (d, *J* = 9.5 Hz), 130.1 (d, *J* = 2.9 Hz), 128.6, 128.4, 128.0, 127.0, 126.8, 126.8, 124.2 (d, *J* = 2.4 Hz), 119.0, 117.8, 116.0, 115.5, 114.9, 114.7, 58.0, 54.3, 54.1 45.1, 44.6. HRMS (ESI) calcd for $C_{32}H_{22}FNO_6SNa^+$ $[M+Na]^+$ 590.1044 found 590.1049.

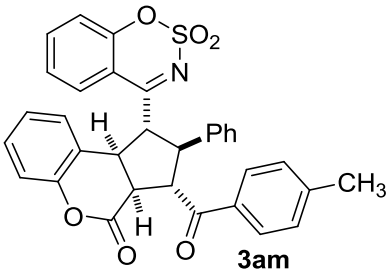
3-(4-bromobenzoyl)-1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-2-phenyl-1,3,3a,9b-tetrahydr ocyclopenta[*c*]chromen-4(2*H*)-one (3al)



3al

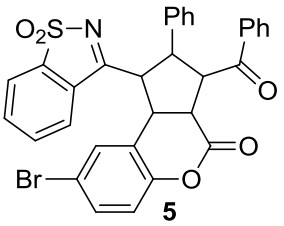
Orange solid (92%, 57.8mg), mp = 72 – 74 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.60 (s, 1H), 7.42 – 7.34 (m, 3H), 7.19 – 6.99 (m, 9H), 6.88 (t, *J* = 7.5 Hz, 1H), 6.71 (t, *J* = 7.7 Hz, 1H), 6.53 (d, *J* = 8.1 Hz, 1H), 4.97 (d, *J* = 7.1 Hz, 1H), 4.12 (dd, *J* = 11.6, 7.9 Hz, 1H), 3.86 (dd, *J* = 10.1, 7.4 Hz, 1H), 3.80 (d, *J* = 7.8 Hz, 1H), 3.65 (t, *J* = 11.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 177.6, 168.5, 152.4, 149.6, 138.4, 136.2, 132.4, 130.9, 129.8, 128.6, 128.5, 128.2, 127.9, 127.0, 126.8, 124.3, 124.2, 119.0, 117.8, 116.0, 115.5, 58.0, 54.2, 54.1, 45.1, 44.6. HRMS (ESI) calcd for $C_{32}H_{22}BrNO_6SNa^+$ $[M+Na]^+$ 650.0243 found 650.0243.

1-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-3-(4-methylbenzoyl)-2-phenyl-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (3am)



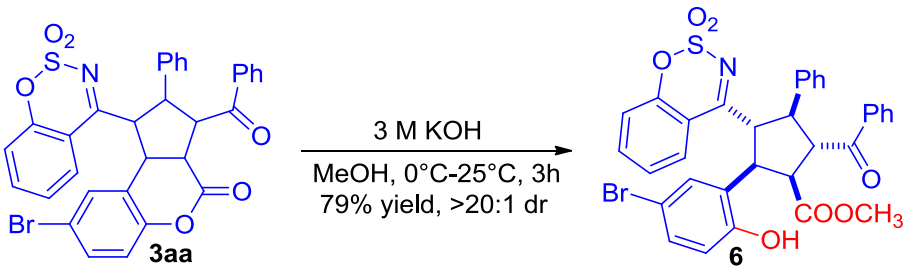
Orange solid (80%, 45.1mg), mp = 185 – 187 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.0 Hz, 2H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.16 – 6.99 (m, 11H), 6.87 (t, *J* = 7.5 Hz, 1H), 6.69 (t, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 8.0 Hz, 1H), 5.01 (d, *J* = 6.9 Hz, 1H), 4.14 (dd, *J* = 11.8, 7.8 Hz, 1H), 3.90 (dd, *J* = 10.4, 7.0 Hz, 1H), 3.78 (d, *J* = 7.8 Hz, 1H), 3.64 (t, *J* = 11.2 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 177.8, 168.6, 152.4, 149.7, 143.8, 138.7, 136.1, 131.2, 128.5, 128.4, 128.3, 128.3, 128.0, 126.9, 126.9, 126.8, 124.2, 124.2, 119.2, 117.7, 115.9, 115.6, 58.1, 54.4, 53.9, 45.1, 44.9, 20.6. HRMS (ESI) calcd for $C_{33}H_{25}NO_6SNa^+$ $[M+Na]^+$ 586.1295 found 586.1296.

3-benzoyl-8-bromo-1-(1,1-dioxidobenzo[*d*]isothiazol-3-yl)-2-phenyl-1,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(2*H*)-one (5)



White solid (85%, 52.1mg), mp = 212 – 214 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.8 Hz, 2H), 7.69 (d, *J* = 7.5 Hz, 1H), 7.49 – 7.36 (m, 3H), 7.32 – 7.20 (m, 3H), 7.20 – 7.13 (m, 3H), 7.13 – 7.04 (m, 3H), 6.90 (d, *J* = 8.7 Hz, 1H), 6.20 (d, *J* = 7.8 Hz, 1H), 5.04 (d, *J* = 6.6 Hz, 1H), 4.19 (dd, *J* = 11.9, 7.9 Hz, 1H), 3.90 – 3.68 (m, 2H), 3.44 (t, *J* = 11.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 173.7, 167.9, 148.8, 138.9, 138.7, 133.5, 132.9, 132.7, 132.2, 131.6, 131.5, 129.3, 128.5, 128.3, 127.6, 127.1, 127.1, 123.2, 121.5, 121.4, 117.6, 116.7, 55.5, 54.5, 54.1, 44.5, 43.4. HRMS (ESI) calcd for $C_{32}H_{22}BrNO_5SNa^+$ $[M+Na]^+$ 634.0294, found 634.0893.

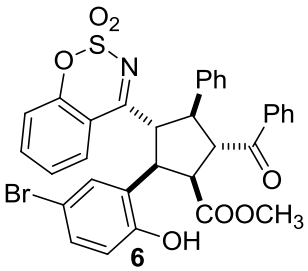
Further Transformation of 3aa



The compound **3aa** (62.8 mg, 0.1 mmol) was dissolved in 1 mL MeOH, and 5 μL 3M KOH until pH was 9, then the reaction mixture was stirred for 3 h at room temperature. The solvent was removed and the crude product was purified through silica gel (petroleum ether/EtOAc 2:1) to afford the desired product **6**.

methyl

2-benzoyl-5-(5-bromo-2-hydroxyphenyl)-4-(2,2-dioxidobenzo[*e*][1,2,3]oxathiazin-4-yl)-3-phenyl cyclopentanecarboxylate (6)

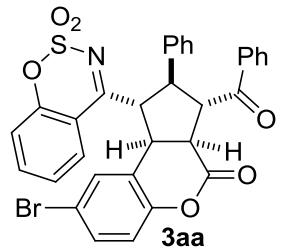


White solid (79%, 52.2mg), mp = 98 – 100 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.8 Hz, 2H), 7.49 – 7.33 (m, 4H), 7.22 (t, *J* = 7.8 Hz, 3H), 7.14 – 6.97 (m, 5H), 6.99 – 6.87 (m, 2H), 6.56 (d, *J* = 8.5 Hz, 1H), 6.38 (s, 1H), 4.85 (dd, *J* = 9.3, 4.9 Hz, 1H), 4.74 (t, *J* = 11.0 Hz, 1H), 4.52 (t, *J* = 10.4 Hz, 1H), 3.99 – 3.81 (m, 2H), 3.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 179.9, 174.1, 152.4, 152.2, 138.2, 136.0, 134.9, 132.5, 130.5, 129.5, 128.1, 127.7, 127.5, 127.1, 126.8, 126.8, 124.9, 124.5, 117.7, 116.6, 115.7, 111.2, 55.4, 53.9, 52.9, 51.1, 49.7, 47.1. HRMS (ESI) calcd for $C_{33}H_{26}BrNO_7SNa^+$ $[M+Na]^+$ 682.0506, found 682.1152.

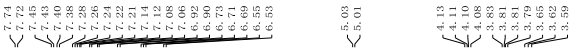
References

1. (a) X. Feng, Z. Zhou, C. Ma, X. Yin, R. Li, Y.-C. Chen. *Angew. Chem. Int. Ed.* **2013**, *52*, 14173. (b) C. Ma, J. Gu, B. Teng, Q. Zhou, R. Li, Y.-C. Chen. *Org. Lett.* **2013**, *15*, 6206. (c) Y. Wu, Y. Liu, W. Yang, H. Liu, L. Zhou, Z. Sun, H. Guo. *Adv. Synth. Catal*. **2016**, *358*, 3517. (d) Q. An, J. Shen, N. Butt, D. Liu, Y. Liu, W. Zhang. *Adv. Synth. Catal.* **2015**, *357*, 3627.

2. Y.-R. Chen, M. R. Ganapuram, K.-H. Hsieh, K.-H. Chen, P. Karanam, S. S. Vagh, Y.-C. Liou and W. Lin, *Chem. Commun.*, 2018, **54**, 12702.


O2
S
N
O
Ph
Ph
O
H
H
O
Br
O

3aa


7.74
7.72
7.45
7.43
7.40
7.38
7.28
7.26
7.24
7.21
7.14
7.12
7.08
7.06
6.92
6.90
6.73
6.71
6.69
6.55
6.53
5.03
5.01
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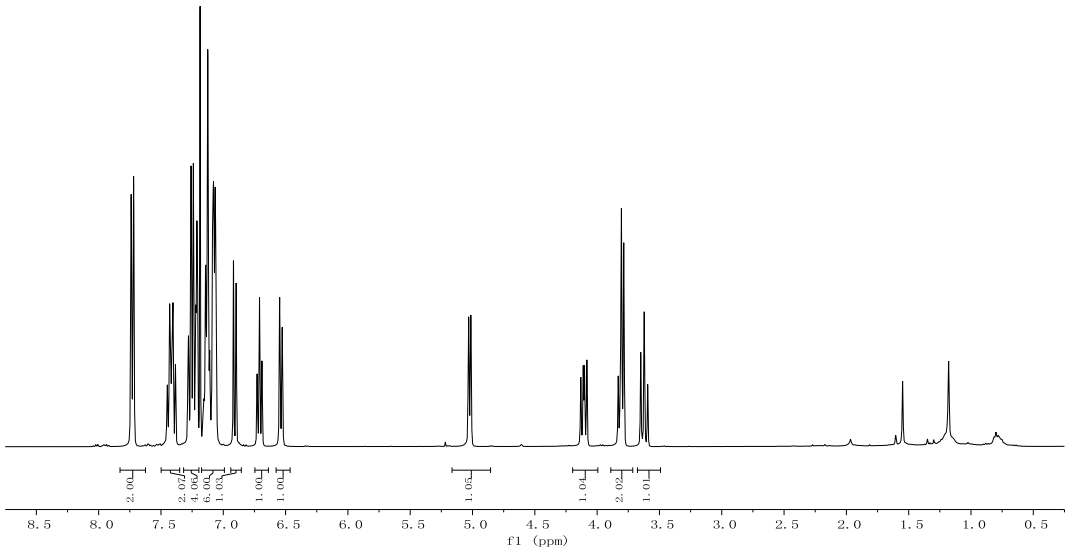
4.11
4.10
4.08
3.83
3.81
3.81
3.79
3.65
3.62
3.59


2.00
2.07
4.06
6.00
1.03
1.00
1.00
1.05
1.04
2.02
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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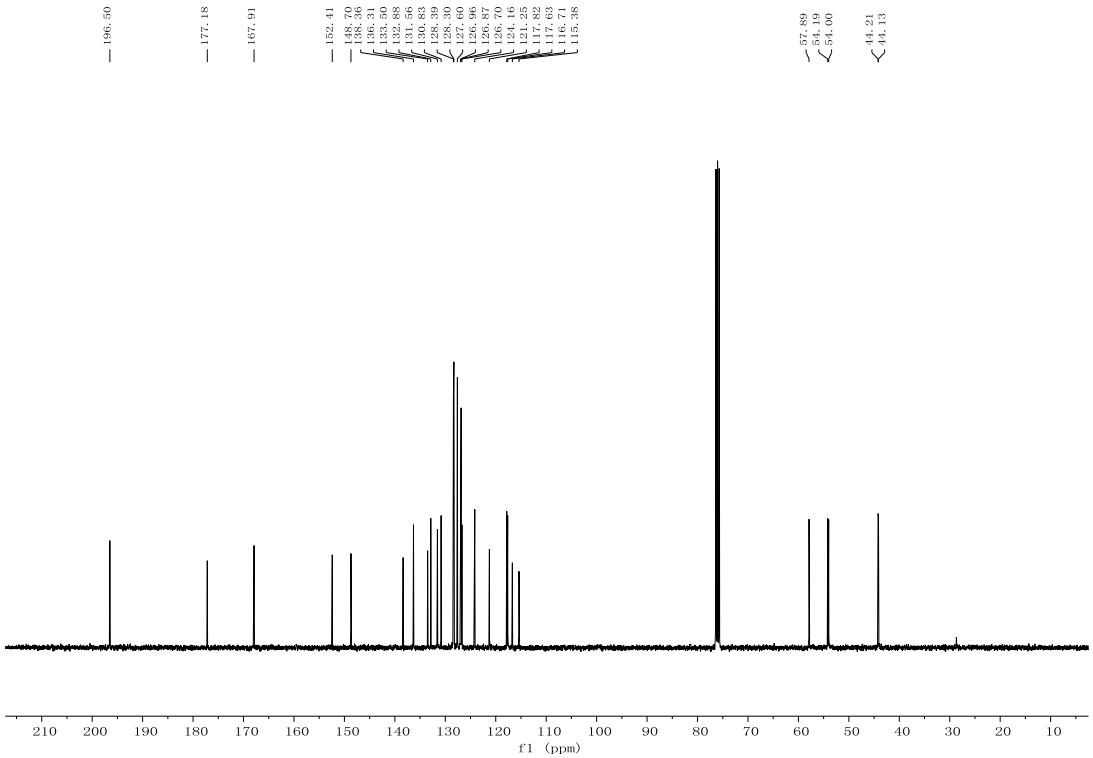
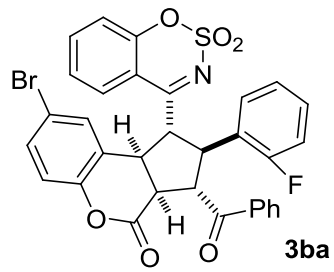
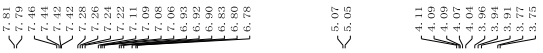
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


196.50
177.18
167.91
152.41
148.70
138.36
136.31
133.50
132.88
131.56
130.83
129.79
128.30
127.60
126.96
126.87
126.70
124.16
121.25
117.82
117.63
116.71
115.38
57.89
54.19
54.00
44.21
44.13
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
Br
H
F
Ph
O
H
O
O

3ba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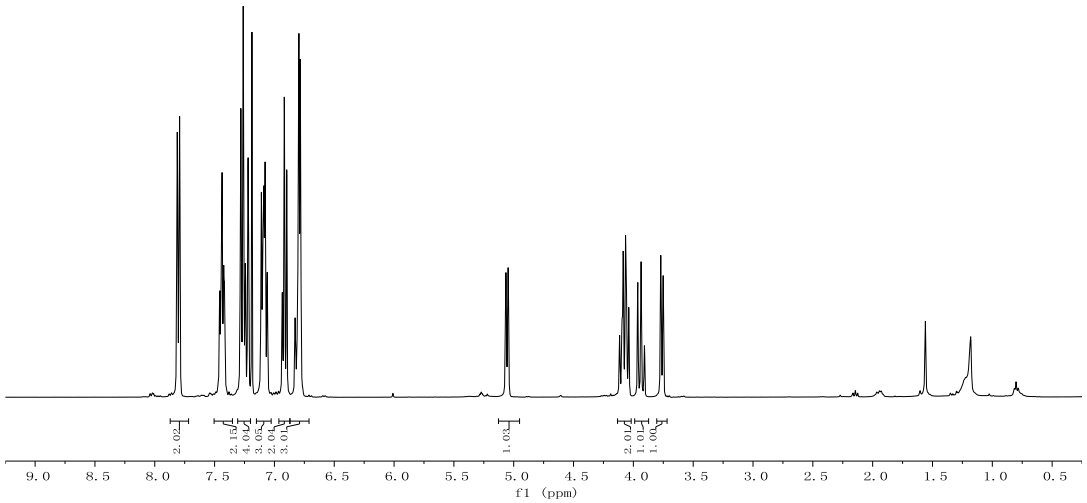
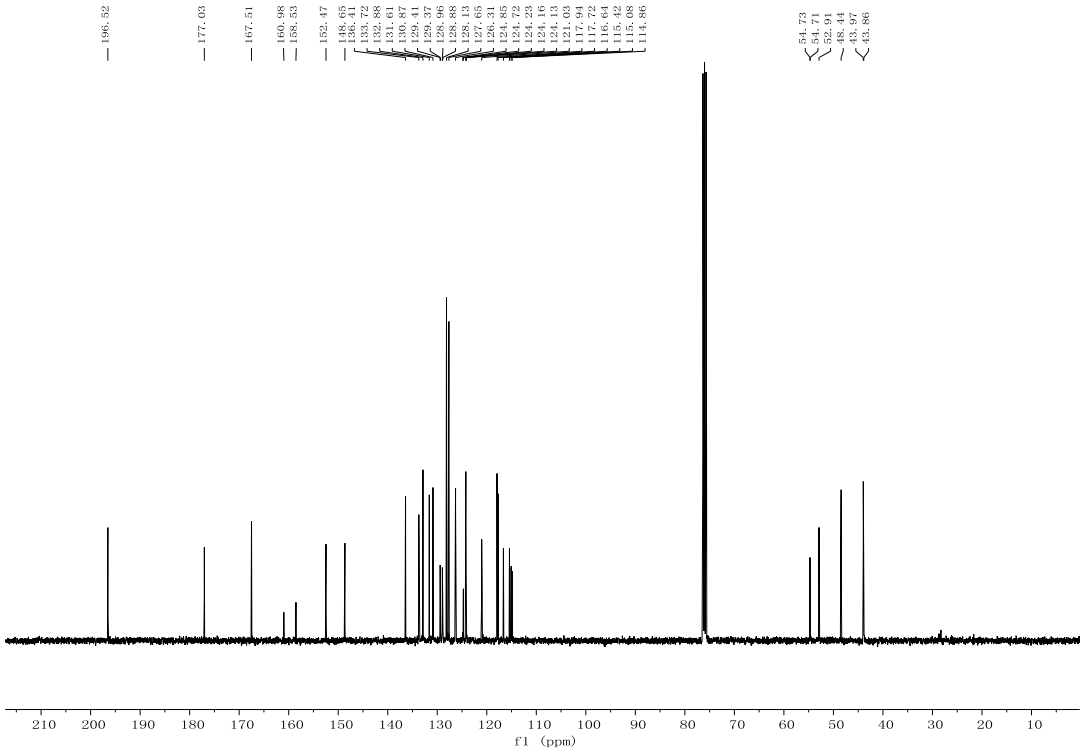
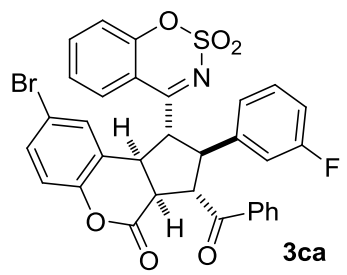
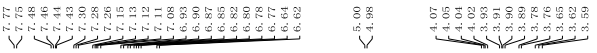
7.79
7.46
7.44
7.42
7.42
7.28
7.26
7.24
7.22
7.11
7.09
7.08
7.06
6.93
6.92
6.90
6.83
6.80
6.78
5.07
5.05
4.11
4.09
4.09
4.07
4.04
3.96
3.94
3.91
3.77
3.75

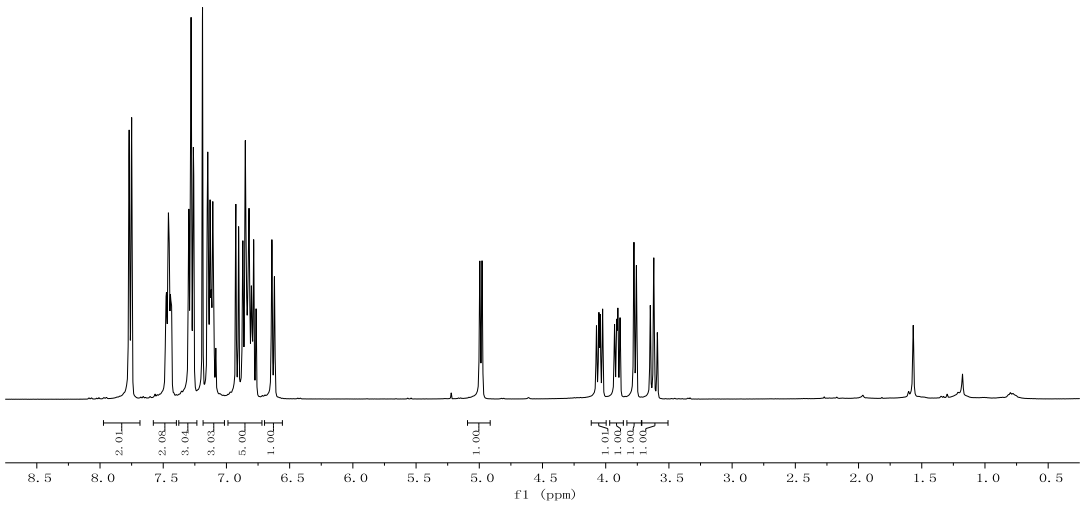

2.02
2.15
4.04
3.05
2.04
3.01
1.03
2.01
1.01
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

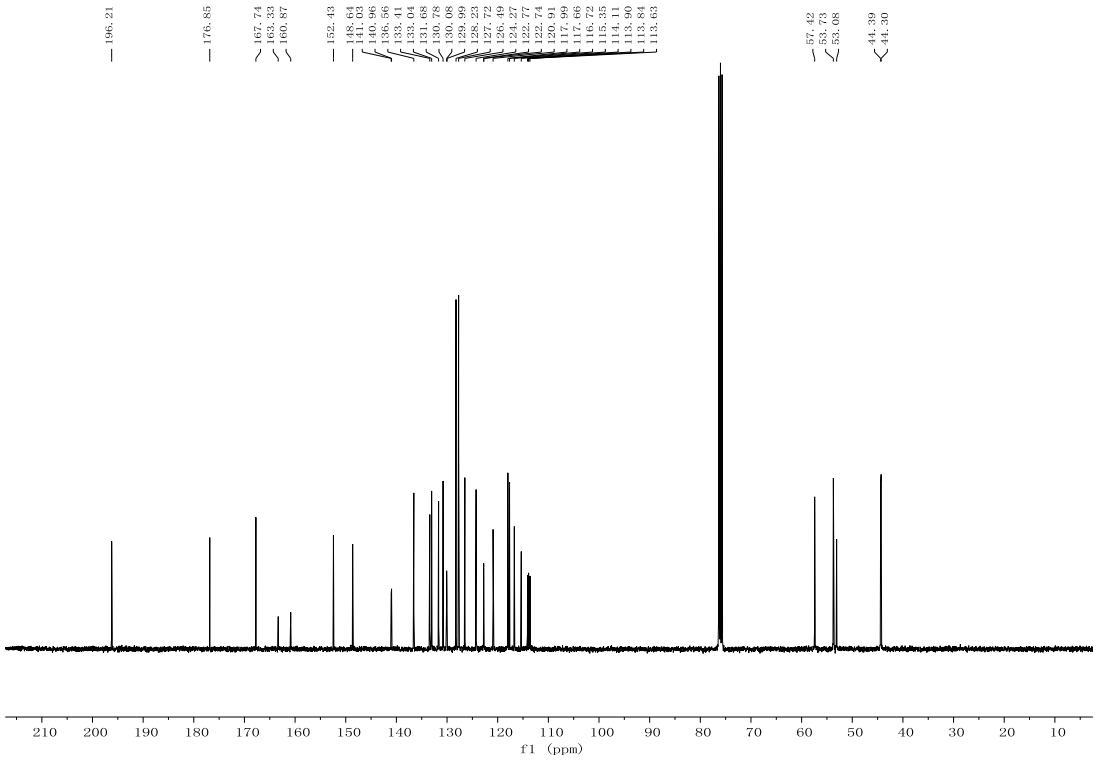

196.52
177.03
167.51
160.98
158.53
152.47
148.65
136.41
135.72
132.88
131.61
130.87
129.41
129.37
128.96
128.88
128.13
127.65
126.31
124.85
124.72
124.23
124.16
124.13
121.03
117.94
117.72
116.64
115.42
115.08
114.86
54.73
54.71
52.91
44.44
43.97
43.86
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


O
SO2
N
Br
H
F
Ph
O
H
O
O

3ca


7.77
7.75
7.48
7.46
7.44
7.43
7.30
7.28
7.26
7.15
7.13
7.12
7.11
7.08
6.93
6.90
6.87
6.85
6.82
6.80
6.78
6.77
6.64
6.62
5.00
4.98
4.07
4.05
4.04
4.02
3.93
3.91
3.90
3.89
3.78
3.76
3.65
3.62
3.59


2.01
2.08
3.04
3.02
5.00
1.00
1.00
1.02
1.00
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


196.21
176.85
167.74
163.33
160.87
152.43
148.64
141.03
140.96
136.56
133.41
133.04
131.68
130.78
130.06
129.99
128.23
127.72
126.49
124.27
122.77
122.74
120.91
117.99
117.66
116.72
115.35
114.11
113.90
113.84
113.63
57.42
53.73
53.08
44.39
44.30
210
200
190
180
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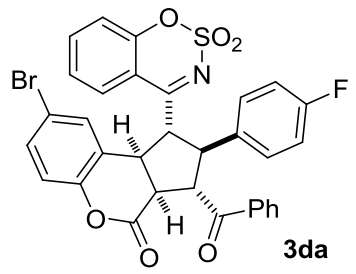
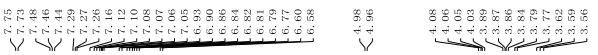
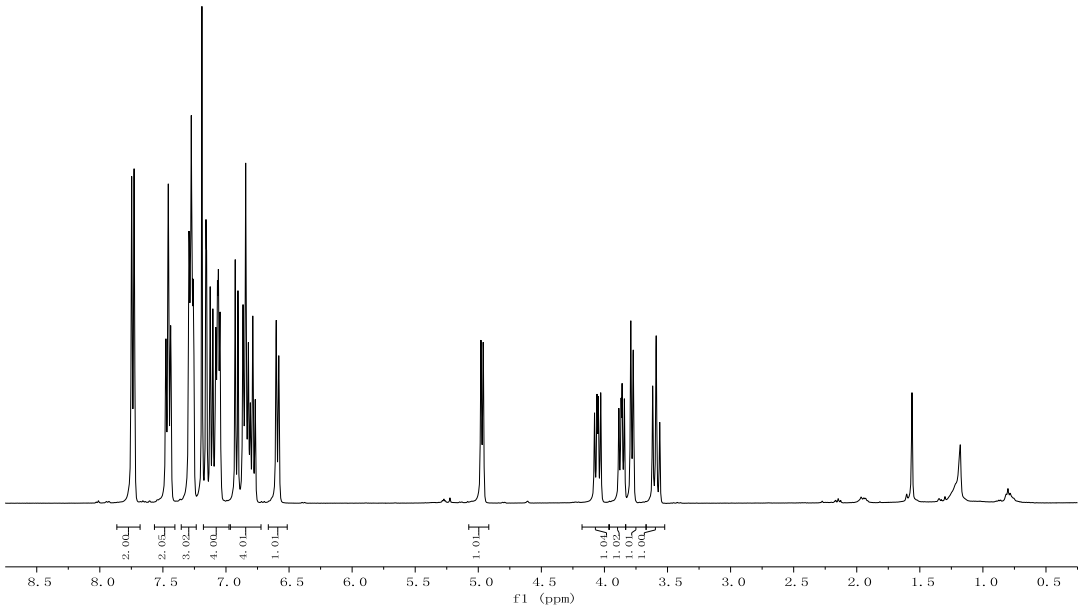
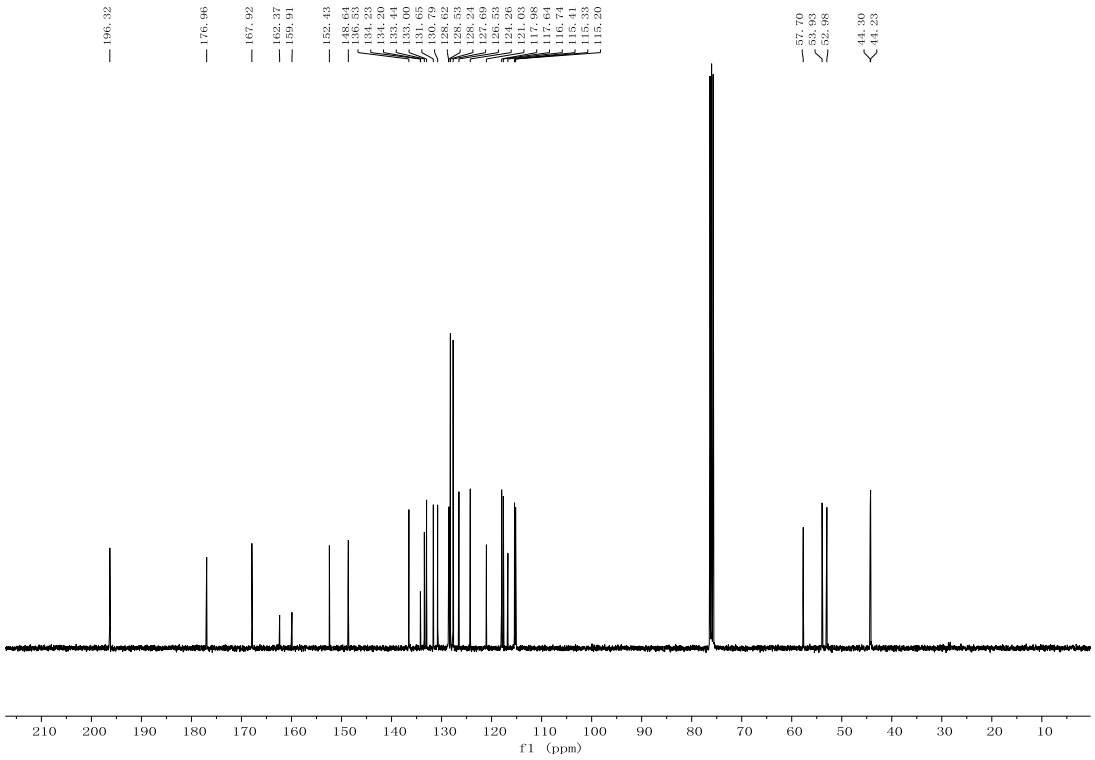
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

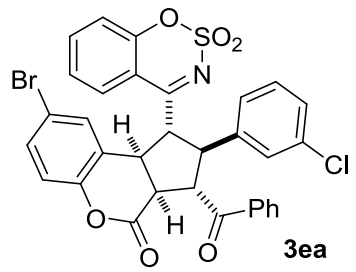


3da

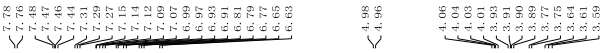







O
SO2
N
Br
H
Cl
Ph
O
H
O
O

3ea


7.78
7.76
7.48
7.47
7.46
7.44
7.31
7.29
7.27
7.15
7.14
7.12
7.09
7.07
6.99
6.97
6.93
6.91
6.81
6.79
6.77
6.65
6.63
4.98
4.96
4.06
4.04
4.03
4.01
3.93
3.91
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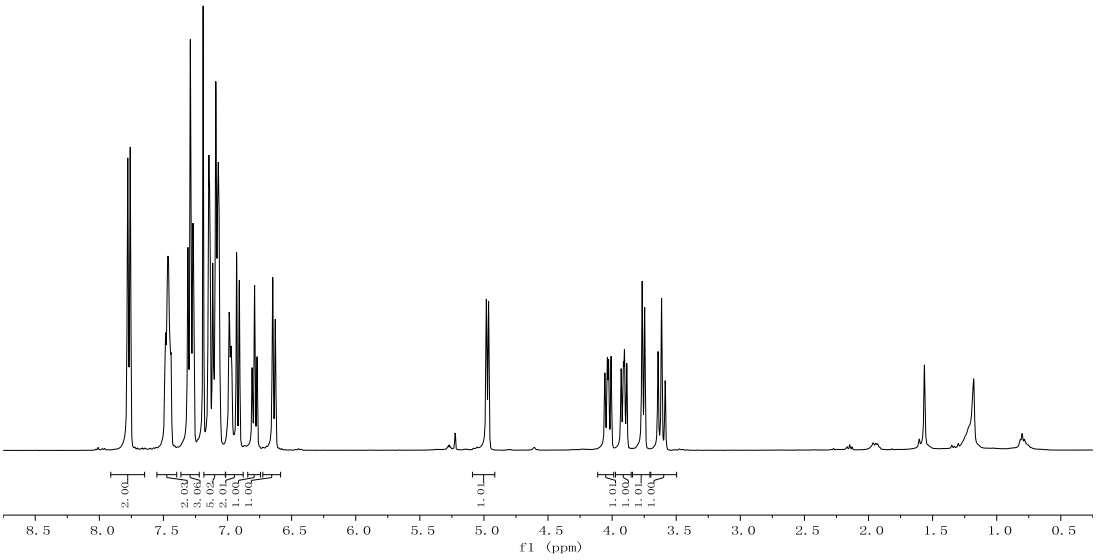
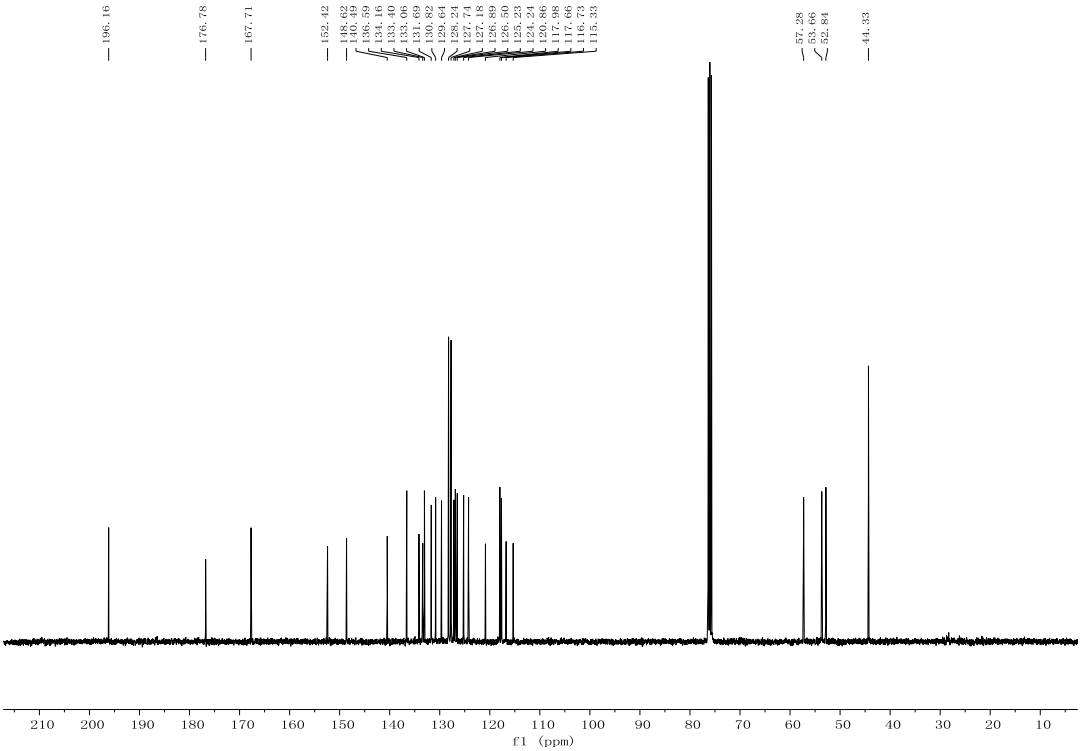
3.89
3.77
3.75
3.64
3.61
3.59


2.00
2.03
3.00
5.02
2.00
1.00
1.01
1.01
1.00
1.01
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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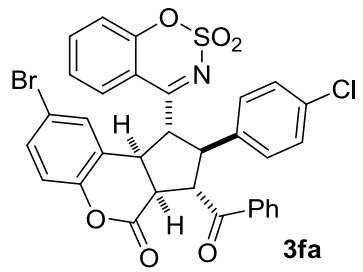
152.42
148.62
140.49
136.59
134.16
133.40
133.06
131.69
130.82
129.64
128.24
127.74
127.18
126.89
126.50
125.23
124.24
120.86
117.98
117.66
116.73
115.33
57.28
53.66
52.84
44.33
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
Br
Cl
H
Ph
O
H
O
O

3fa


7.76
7.74
7.49
7.47
7.45
7.31
7.29
7.27
7.14
7.12
7.05
7.03
6.93
6.91
6.81
6.79
6.78
6.60
6.58
4.97
4.95
4.06
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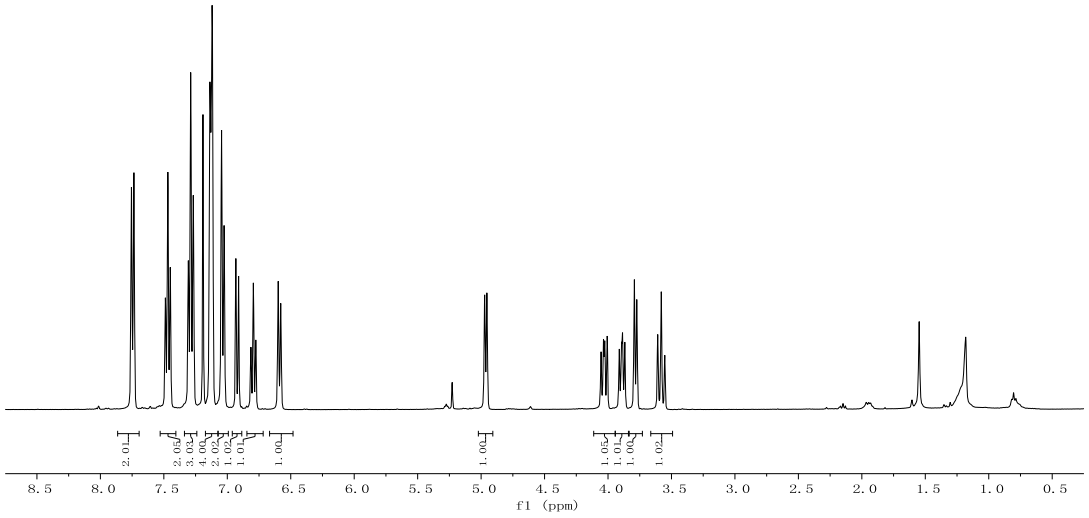
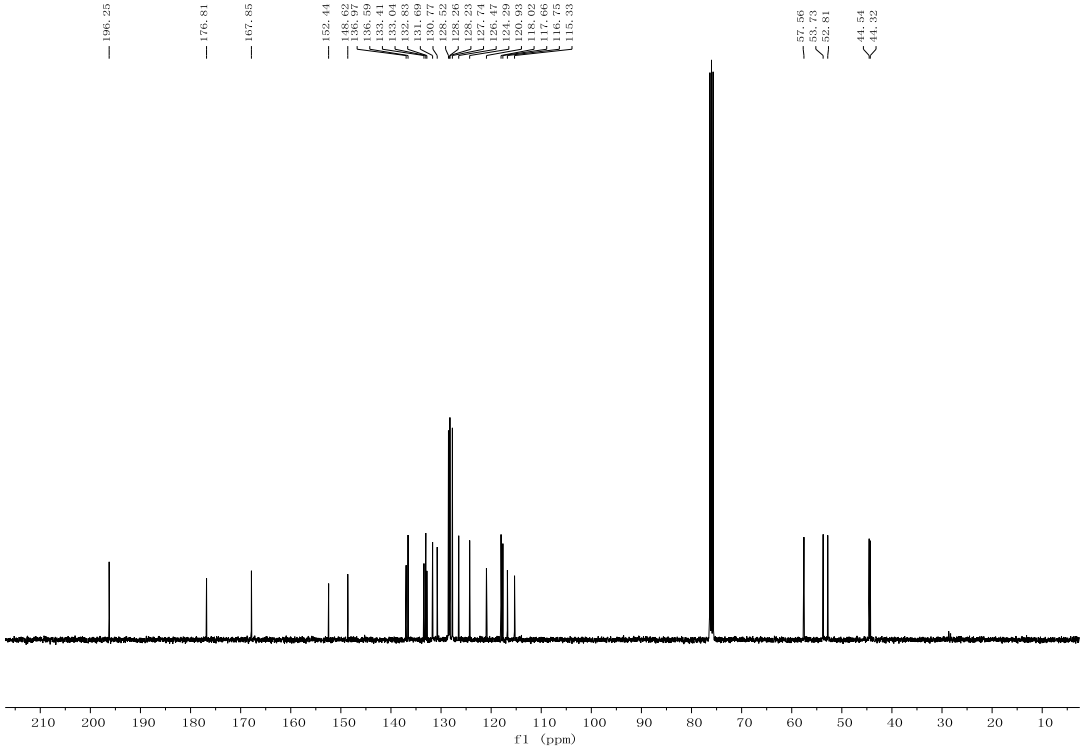
3.87
3.79
3.77
3.61
3.58
3.55


2.01
2.05
3.03
4.00
2.02
1.02
1.01
1.00
1.00
1.05
1.01
1.00
1.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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152.44
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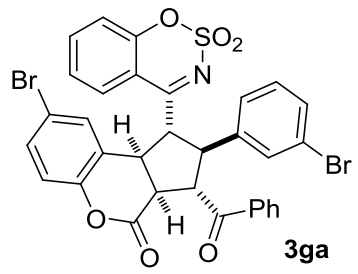
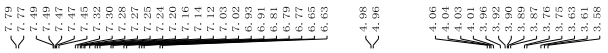
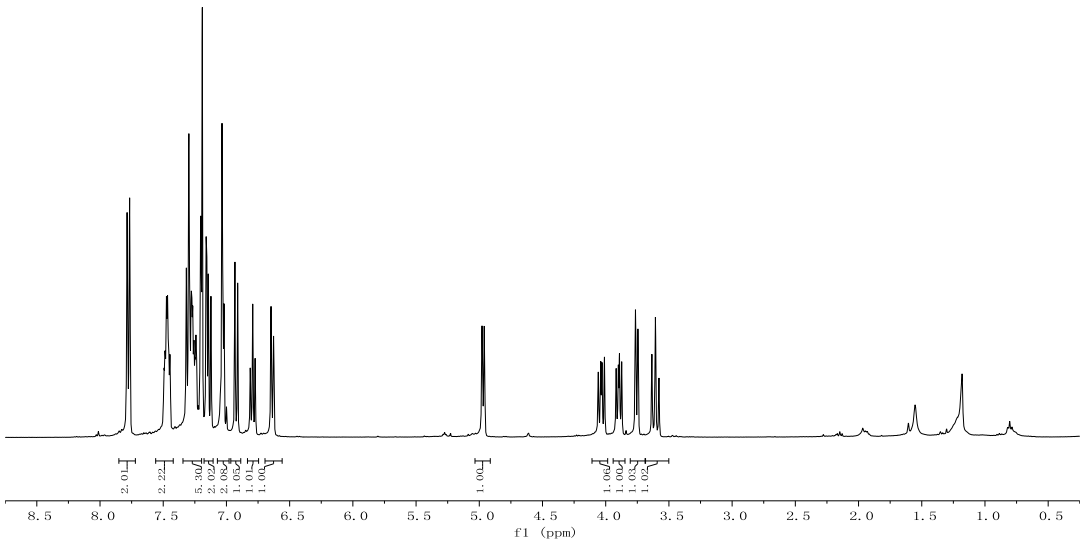
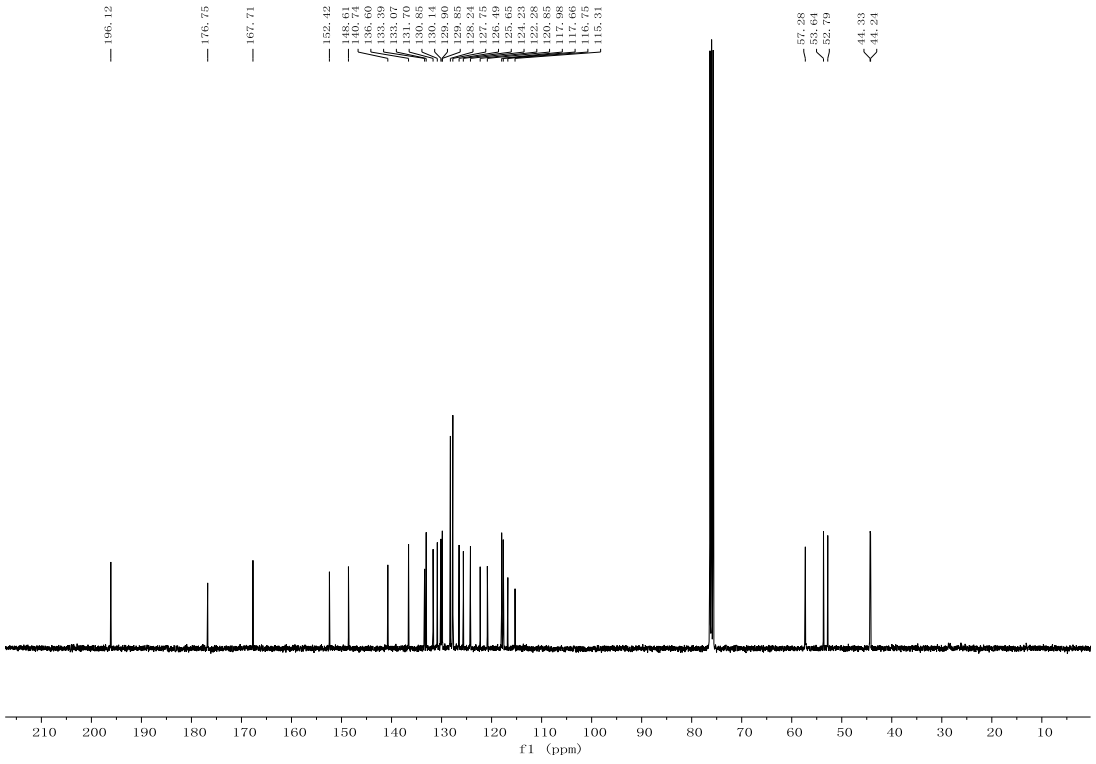
136.97
136.59
133.41
133.04
132.83
131.69
130.77
128.52
128.26
128.23
127.74
126.47
124.29
120.93
118.02
117.66
116.75
115.33
57.56
53.73
52.81
44.54
44.32
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

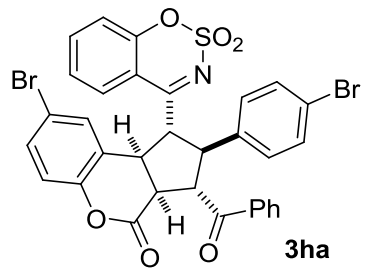


3ga



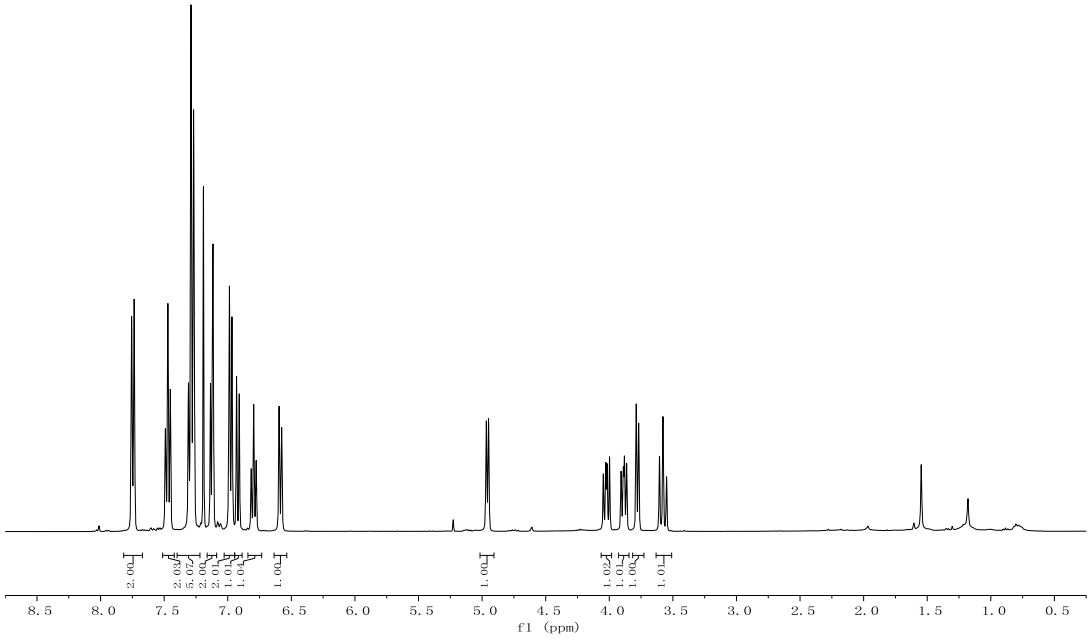


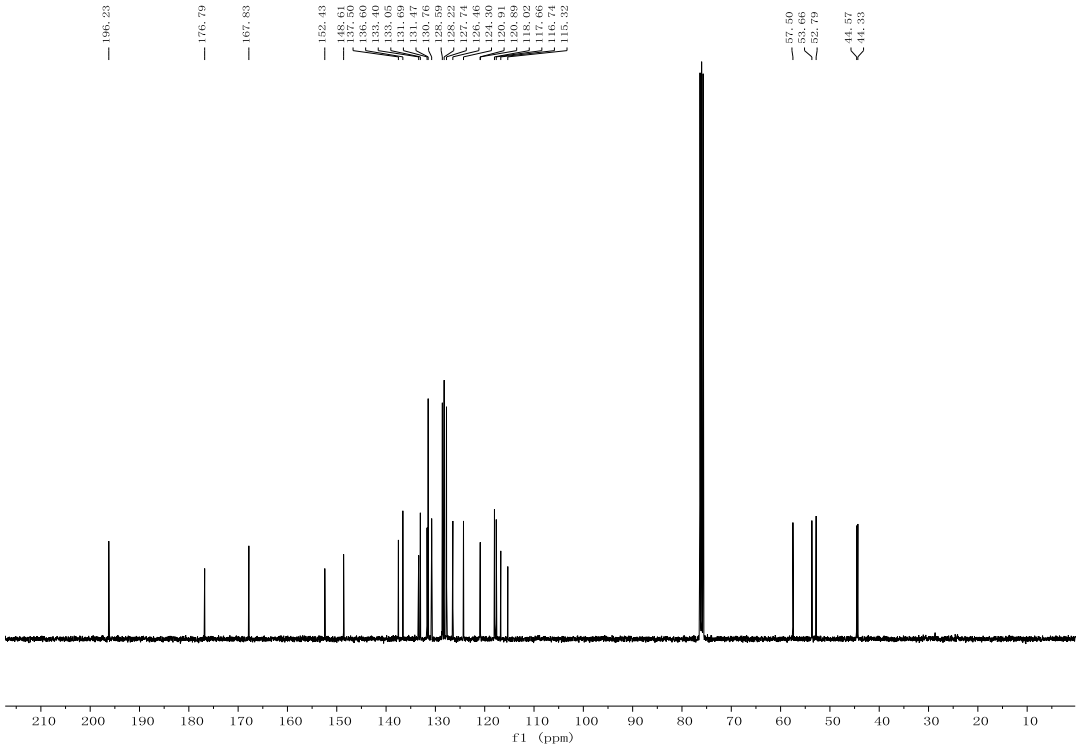


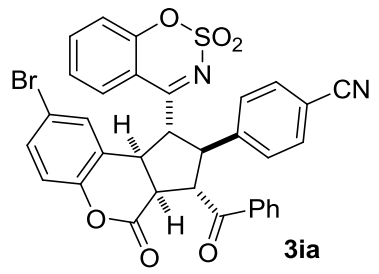

O
SO2
N
Br
Br
H
Ph
O
H
O
O

3ha


7.76
7.74
7.49
7.47
7.45
7.31
7.29
7.27
7.14
7.13
6.99
6.97
6.93
6.91
6.82
6.80
6.78
6.60
6.58
4.97
4.95
4.05
4.03
4.02
4.00
3.91
3.89
3.88
3.86
3.79
3.77
3.61
3.58
3.55

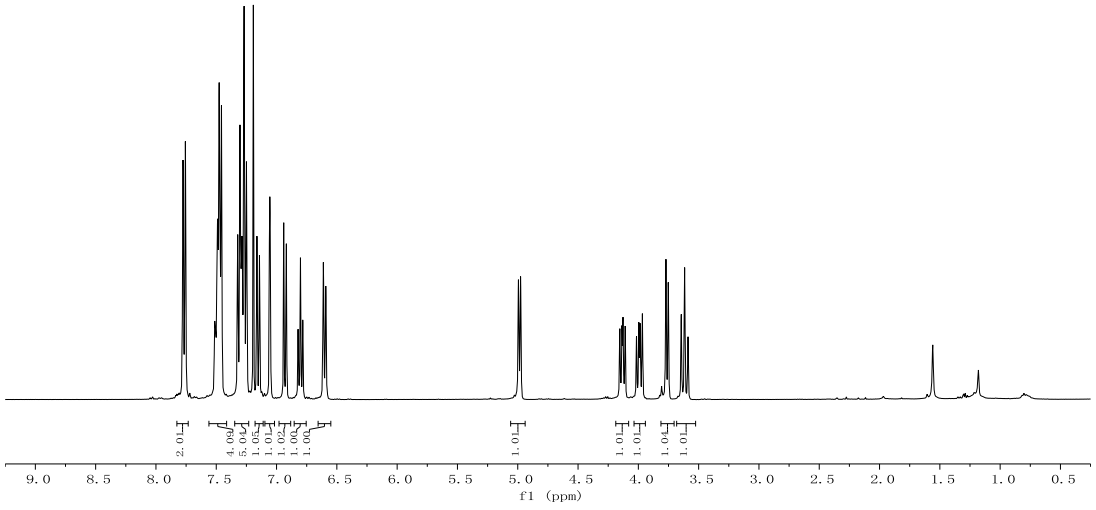

2.00
2.03
5.07
2.00
2.00
1.04
1.04
1.00
1.00
1.02
1.01
1.00
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

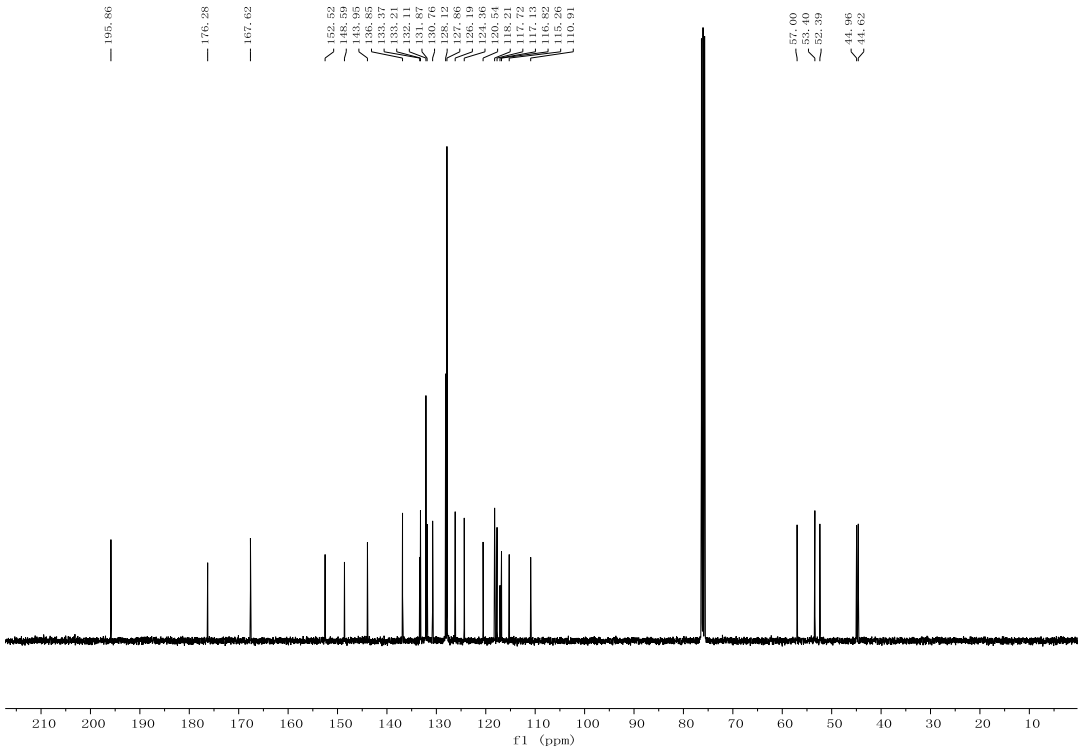

196.23
176.79
167.83
152.43
148.61
137.60
136.60
133.40
133.05
131.69
131.47
130.76
128.59
128.22
127.74
126.46
124.30
120.91
120.89
118.02
117.66
116.74
115.32
57.50
53.66
52.79
44.57
44.33
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
Br
CN
H
Ph
O
H
O
O

3ia


7.78
7.76
7.51
7.49
7.48
7.46
7.32
7.30
7.29
7.27
7.25
7.16
7.14
7.06
6.94
6.92
6.82
6.80
6.78
6.61
6.59
4.99
4.98
4.15
4.14
4.13
4.11
4.02
4.00
3.99
3.97
3.77
3.75
3.64
3.62
3.59


2.01
4.09
5.04
1.05
1.01
1.02
1.00
1.00
1.01
1.01
1.04
1.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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176.28
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152.52
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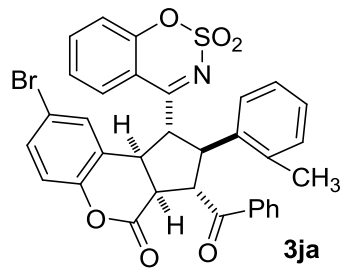
143.95
136.85
133.37
133.21
132.11
131.87
130.76
128.12
127.86
126.19
124.36
120.54
118.21
117.72
117.13
116.82
115.26
110.91
57.00
53.40
52.39
44.96
44.62
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
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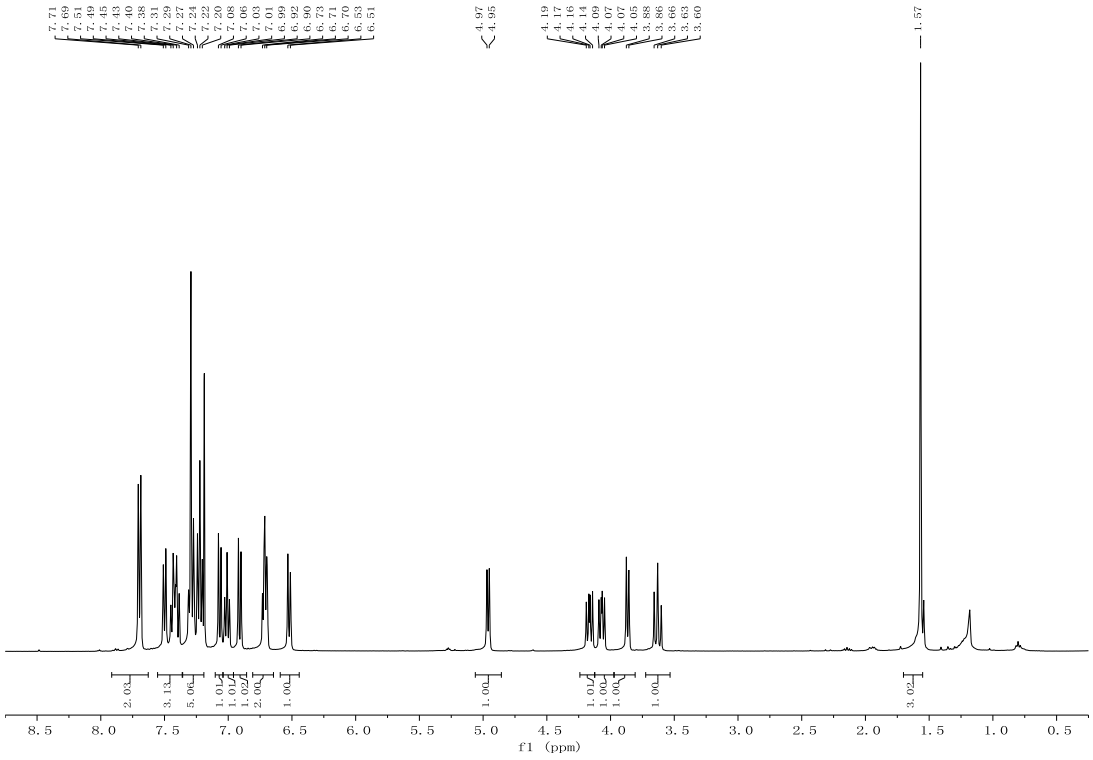
Br
H
CH3
Ph
O
H
O
O

3ja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7.08
7.06
7.03
7.01
6.99
6.92
6.90
6.73
6.71
6.70
6.53
6.51
4.97
4.95
4.19
4.17
4.16
4.14
4.09
4.07
4.07
4.05
3.88
3.86
3.66
3.63
3.60
1.57
2.03
3.13
5.06
1.01
1.01
1.02
2.00
1.00
1.00
1.01
1.00
1.00
1.00
3.02
8.5
8.0
7.5
7.0
6.5
6.0
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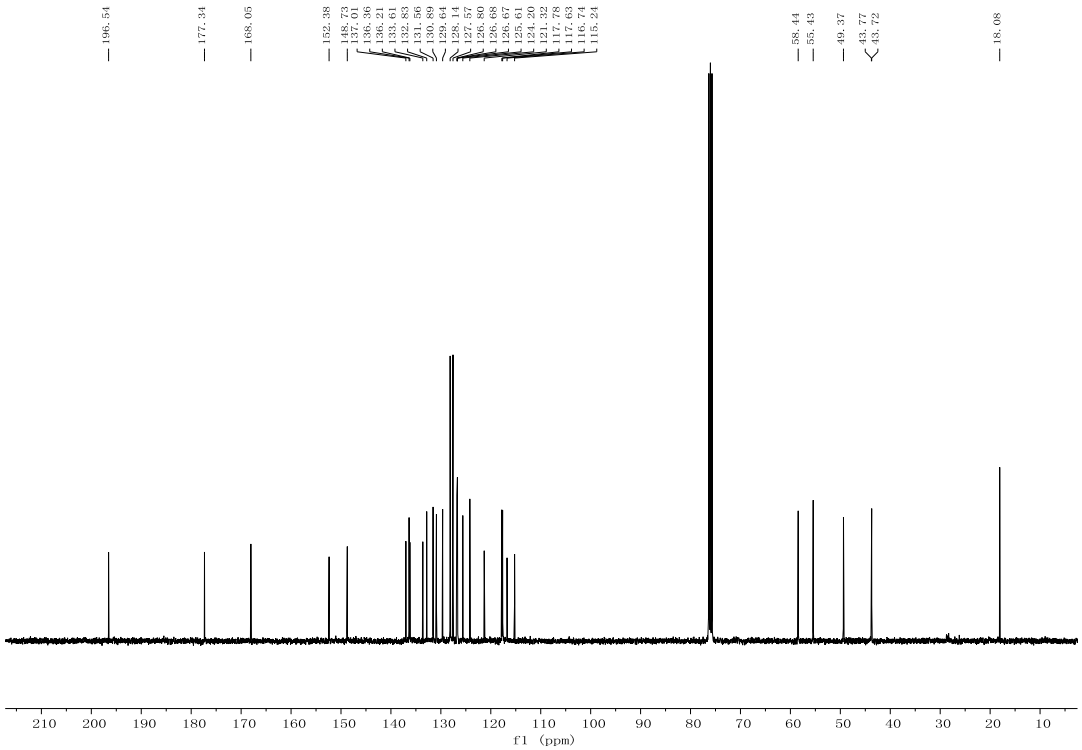
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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132.83
131.56
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129.64
128.14
127.57
126.80
126.68
126.67
125.64
124.90
121.32
117.78
117.63
116.74
115.24
58.44
55.43
49.37
43.77
43.72
18.08
210
200
190
180
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120
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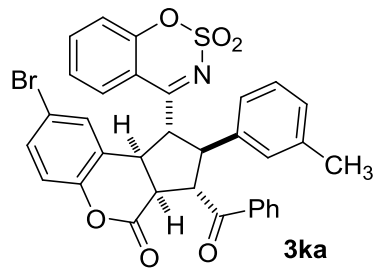
70
60
50
40
30
20
10
f1 (ppm)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SO2
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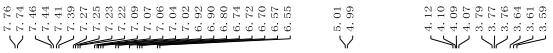
Br
H
CH3
Ph
O
H
O
O

3ka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7.25
7.23
7.22
7.09
7.07
7.06
7.04
7.02
6.92
6.90
6.80
6.74
6.72
6.70
6.57
6.55
5.01
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4.10
4.09
4.07
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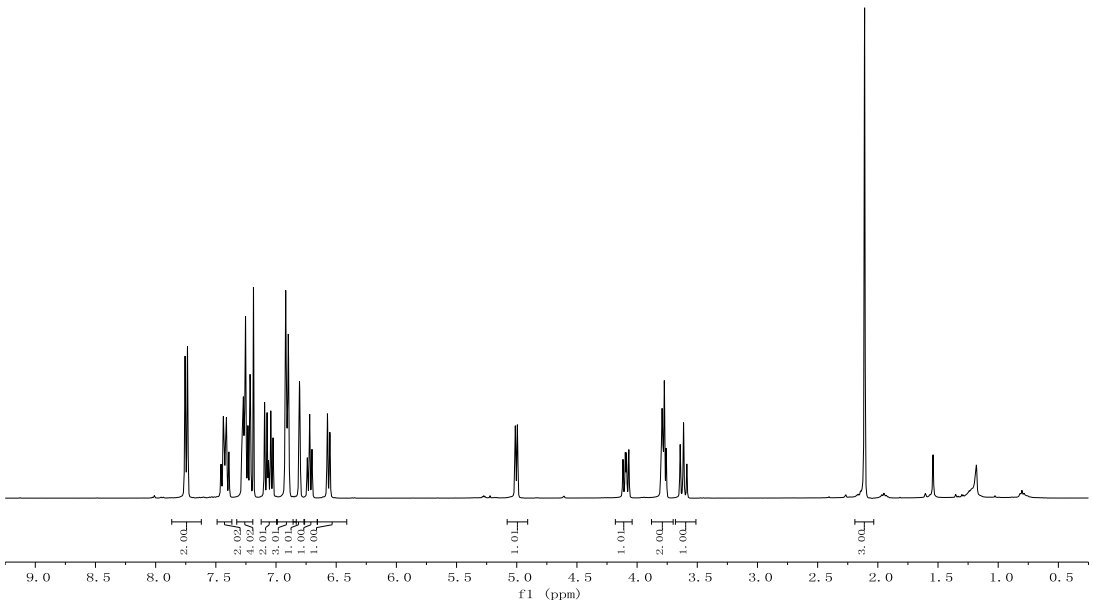
3.76
3.64
3.61
3.59
2.11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2.02
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3.01
1.01
1.00
1.00
1.01
1.01
2.00
1.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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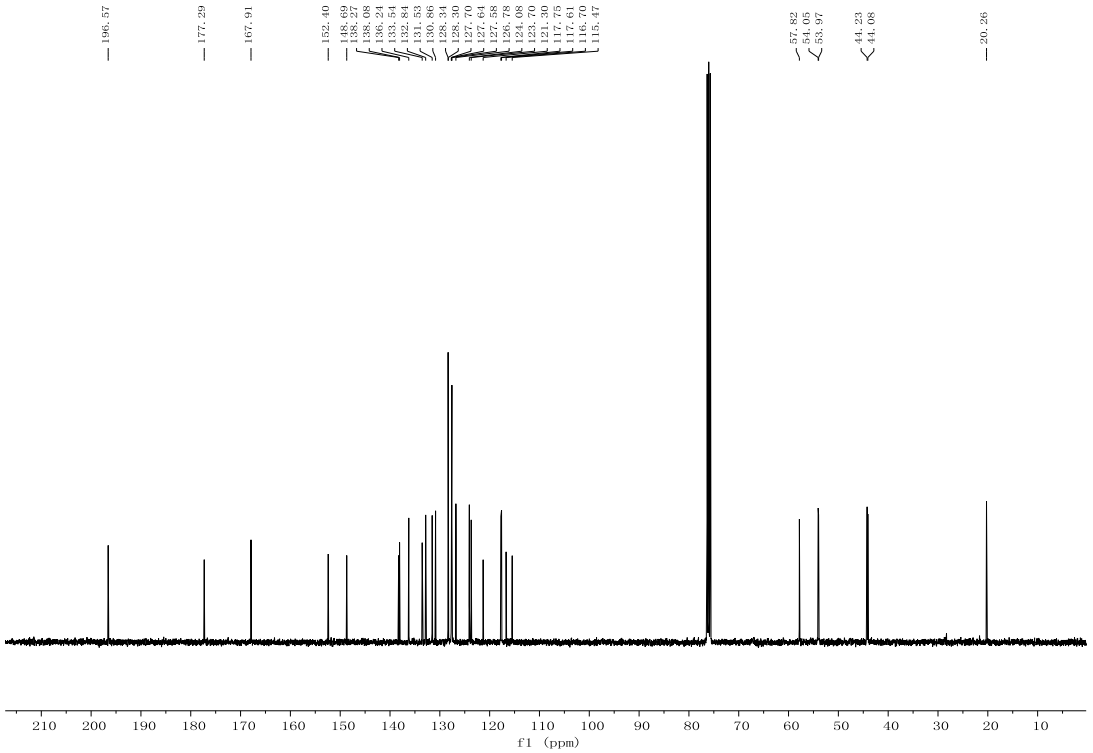
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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132.84
131.53
130.86
128.34
128.30
127.70
127.64
127.58
126.78
124.06
123.70
121.30
117.75
117.61
116.70
115.47
57.82
54.05
53.97
44.23
44.08
20.26
210
200
190
180
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150
140
130
120
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100
90
80
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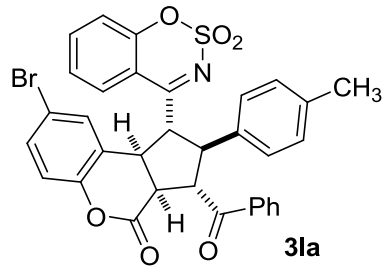
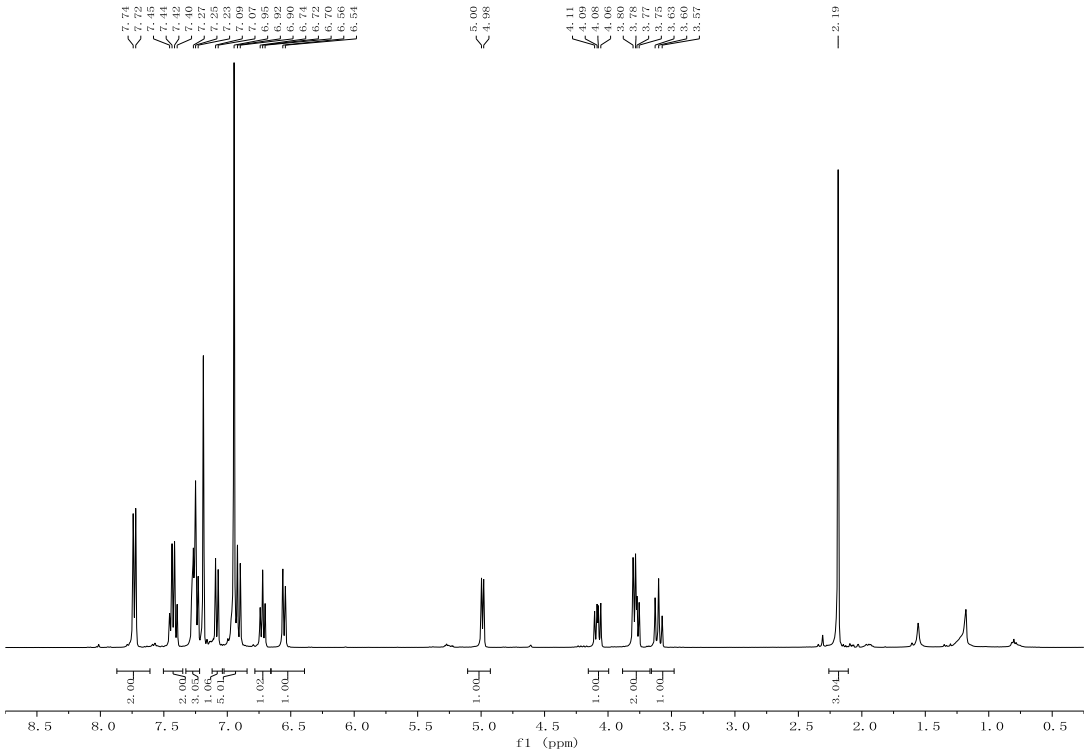
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
Br
CH3
H
Ph
O
H
O
O

3la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7.42
7.40
7.27
7.25
7.23
7.09
7.07
6.95
6.92
6.90
6.74
6.72
6.70
6.56
6.54
5.00
4.98
4.11
4.09
4.08
4.06
3.80
3.78
3.77
3.75
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3.60
3.57
2.19
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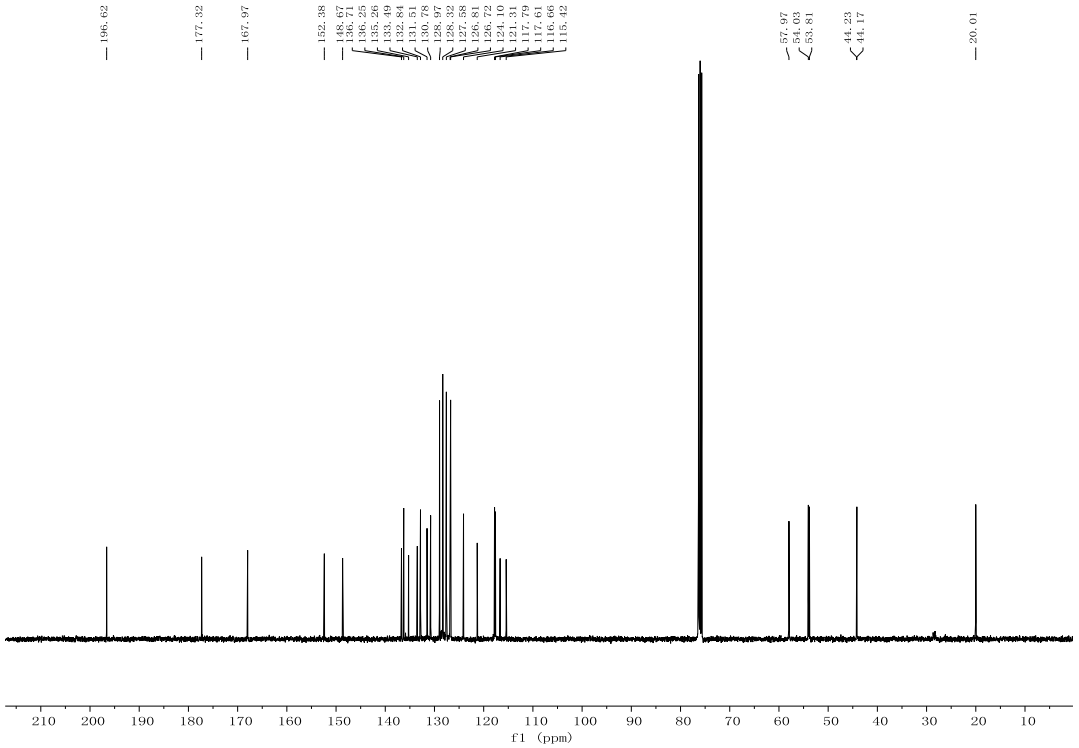
1.06
5.01
1.02
1.00
1.00
1.00
2.00
1.00
3.04
f1 (ppm)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148.67
136.71
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132.84
131.51
130.78
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128.32
127.88
126.81
126.72
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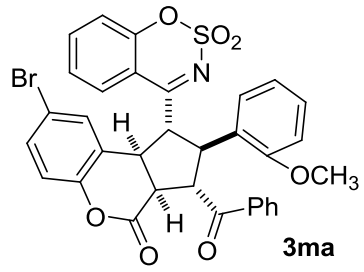
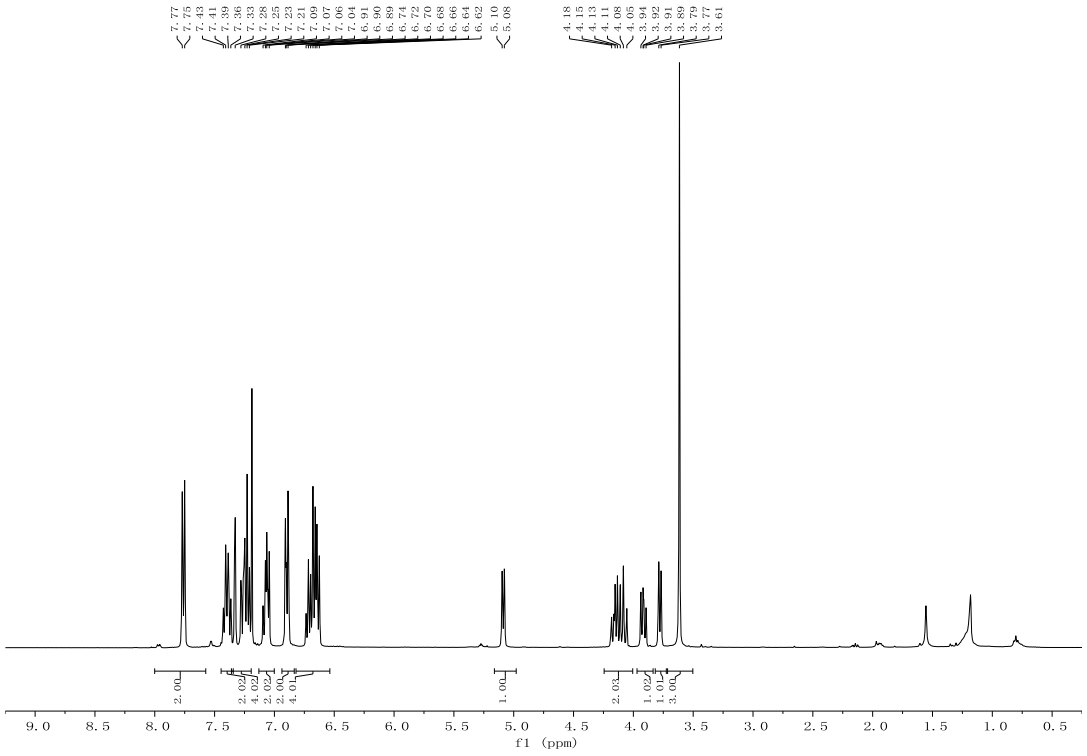
53.81
44.23
44.17
20.01
f1 (ppm)


O
SO2
N
Br
H
OCH3
Ph
H
O
O
O

3ma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7.28
7.25
7.23
7.21
7.09
7.07
7.06
7.04
6.91
6.90
6.89
6.74
6.72
6.70
6.68
6.66
6.64
6.62
5.10
5.08
4.18
4.15
4.13
4.11
4.08
4.05
3.94
3.92
3.91
3.89
3.79
3.77
3.61
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2.02
4.02
2.02
2.00
4.01
1.00
2.03
1.02
1.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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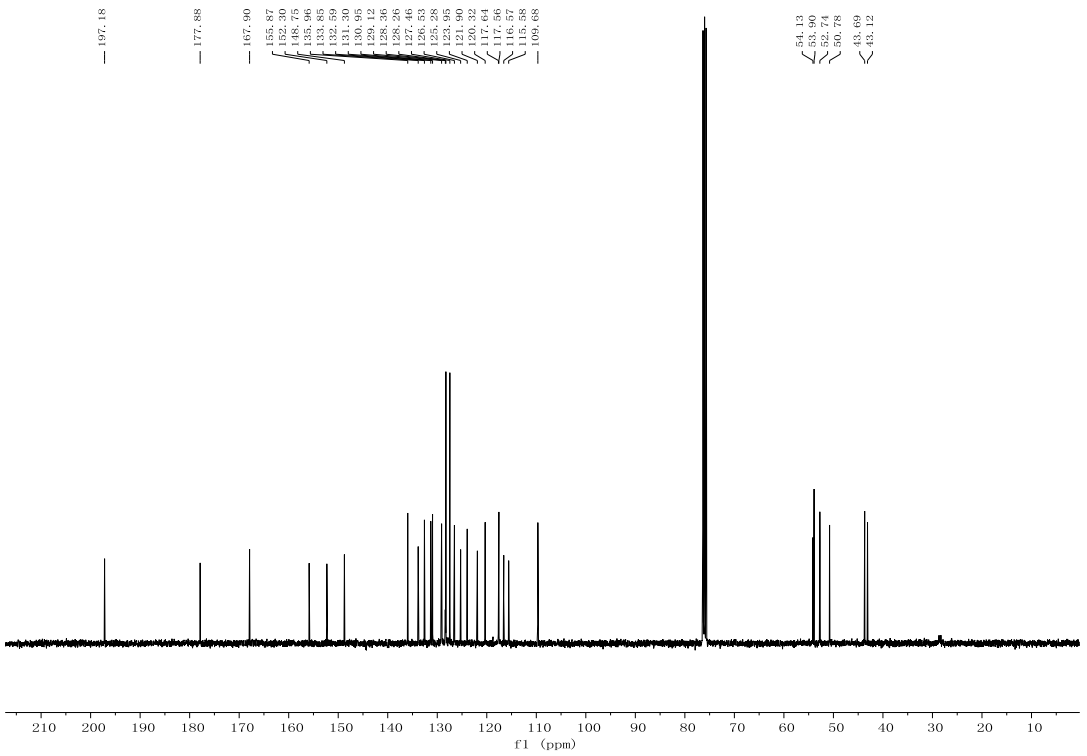
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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130.95
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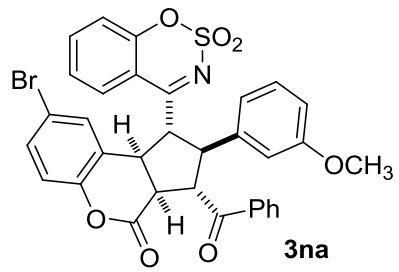
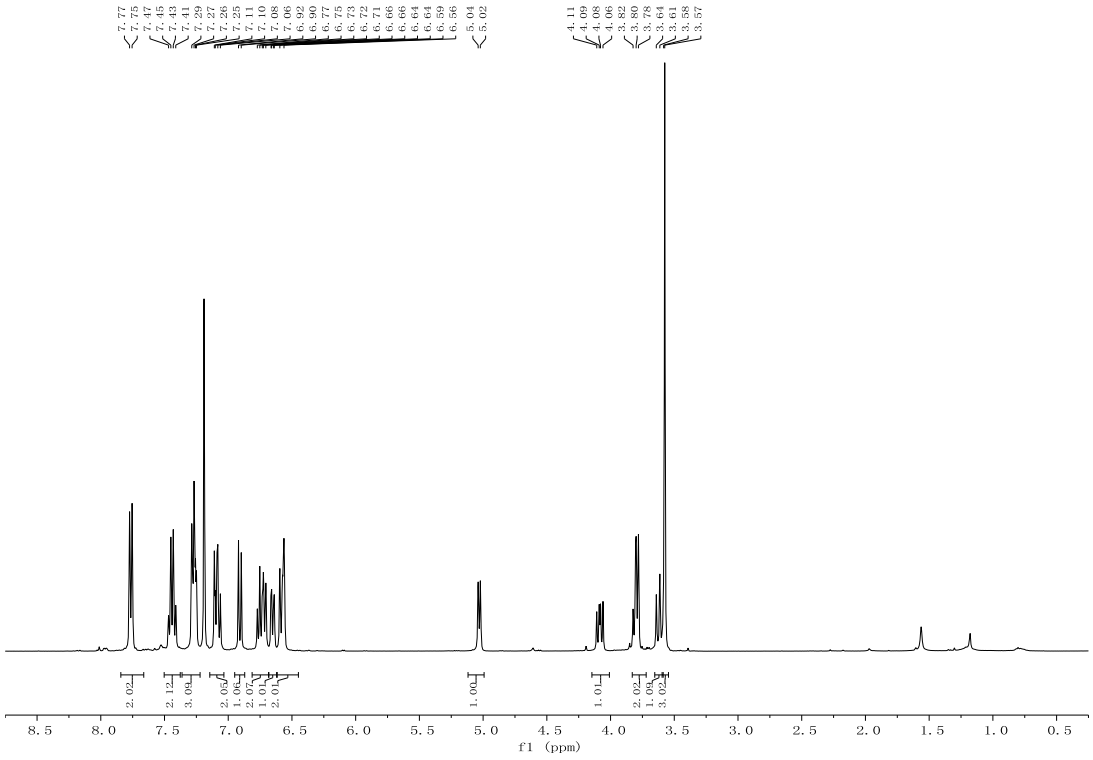
128.36
128.26
127.46
126.53
125.38
123.95
121.90
120.32
117.64
117.56
116.57
115.58
109.68
54.13
53.90
52.74
50.78
43.69
43.12
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
Br
H
OCH3
Ph
O
H
O
O

3na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7.41
7.29
7.27
7.26
7.25
7.11
7.10
7.08
7.06
6.92
6.90
6.77
6.75
6.73
6.72
6.71
6.66
6.66
6.64
6.64
6.59
6.56
5.04
5.02
4.11
4.09
4.08
4.06
3.82
3.80
3.78
3.64
3.64
3.58
3.57
2.02
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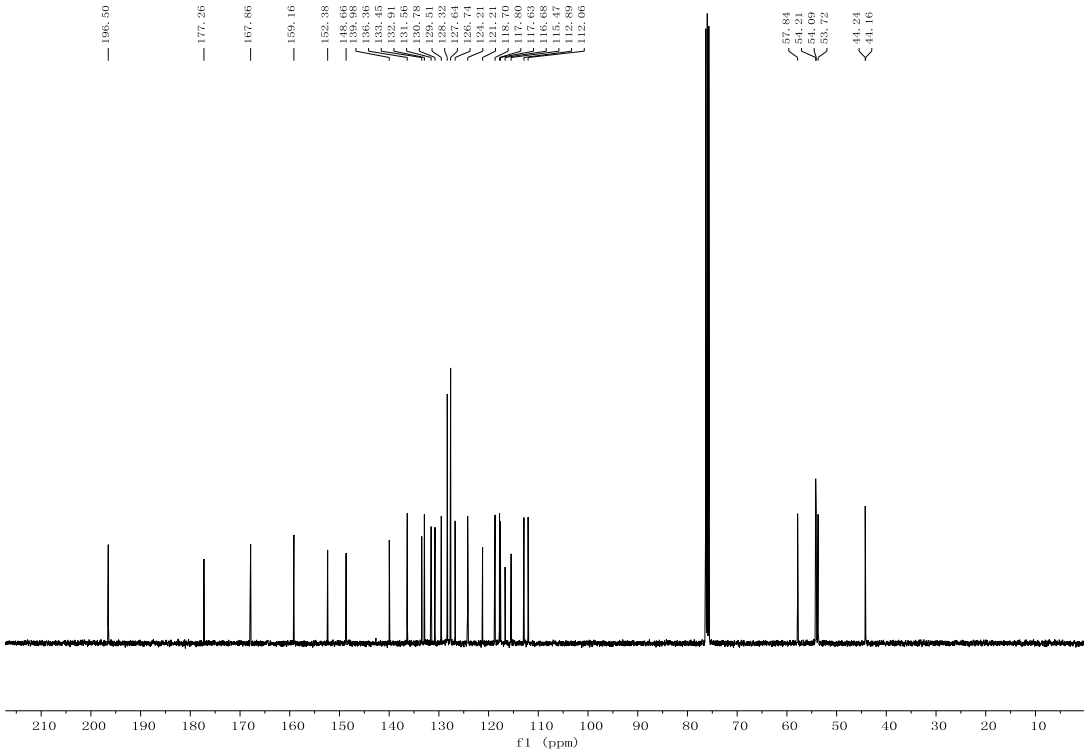
1.01
2.02
1.09
3.02
f1 (ppm)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132.91
131.56
130.78
129.51
128.32
127.64
126.74
124.21
121.21
118.70
117.80
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116.68
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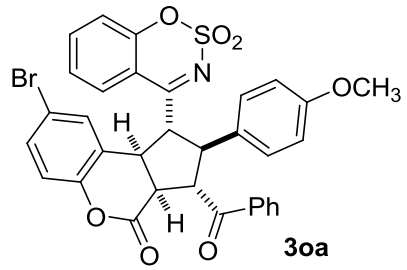
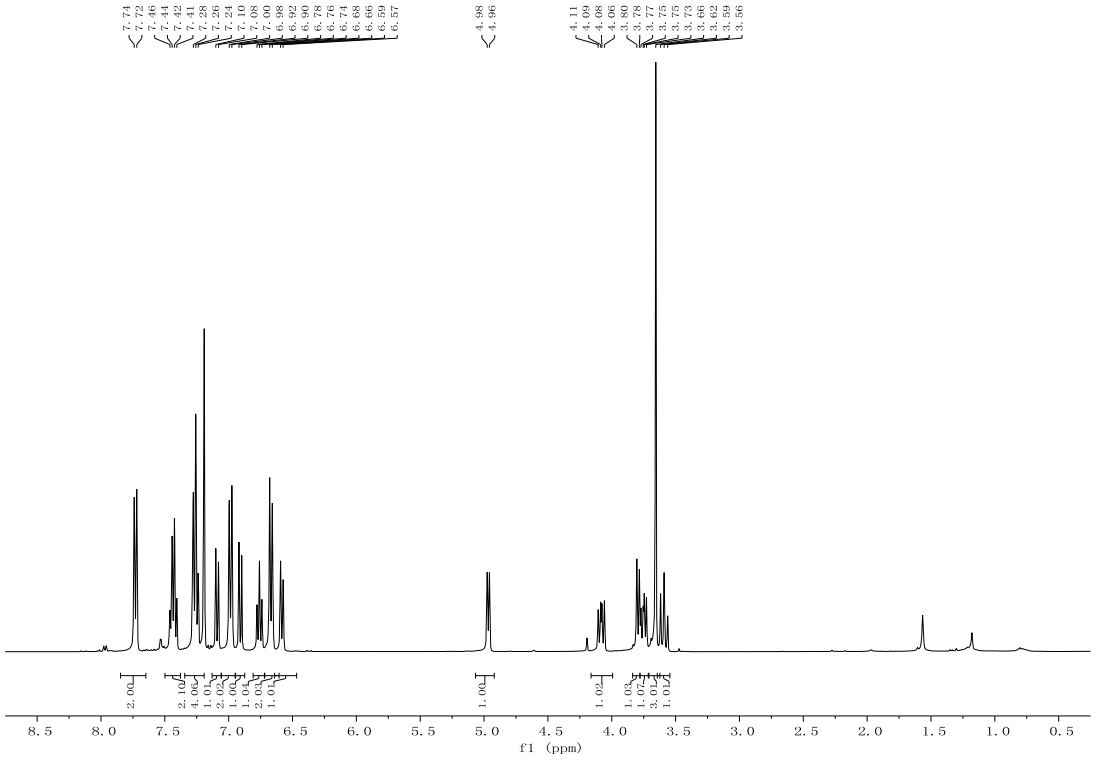
112.89
112.06
57.84
54.23
54.09
53.72
44.24
44.16
f1 (ppm)


O
SO2
N
Br
OCH3
H
Ph
O
H
O
O

3oa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7.24
7.10
7.08
7.00
6.98
6.92
6.90
6.78
6.76
6.74
6.68
6.66
6.59
6.57
4.98
4.96
4.11
4.09
4.08
4.06
3.80
3.78
3.77
3.75
3.75
3.73
3.66
3.62
3.59
3.56
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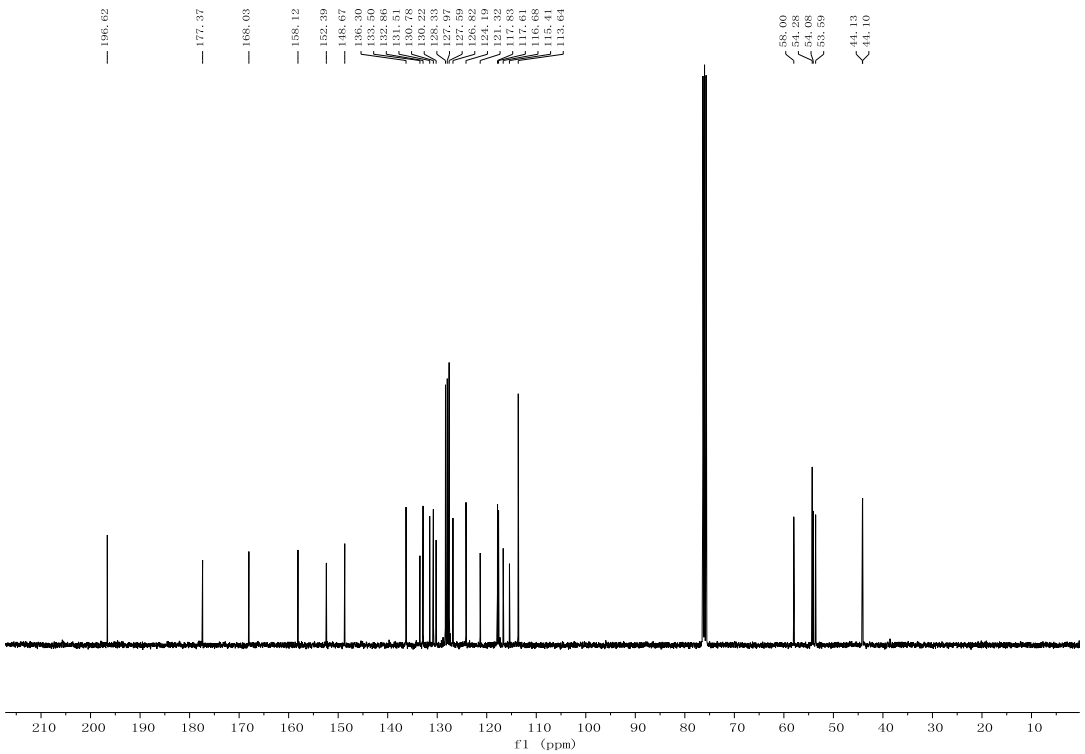
2.10
4.06
1.01
2.02
1.00
1.04
2.03
1.01
1.00
1.02
1.03
1.07
3.01
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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133.50
132.86
131.51
130.78
130.22
128.33
127.97
127.59
126.82
124.19
121.32
117.83
117.61
116.68
115.41
113.64
58.00
54.28
54.08
53.59
44.13
44.10
210
200
190
180
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160
150
140
130
120
110
100
90
80
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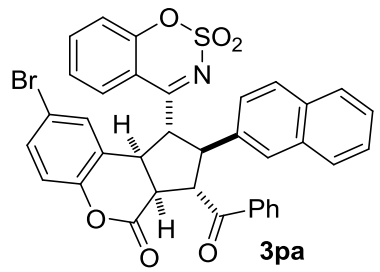
60
50
40
30
20
10
f1 (ppm)



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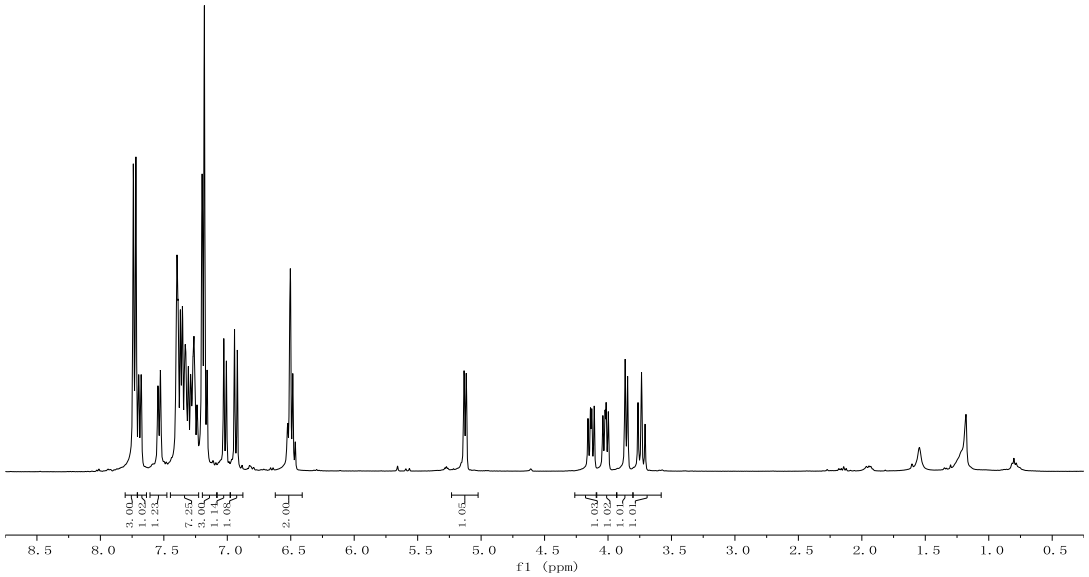
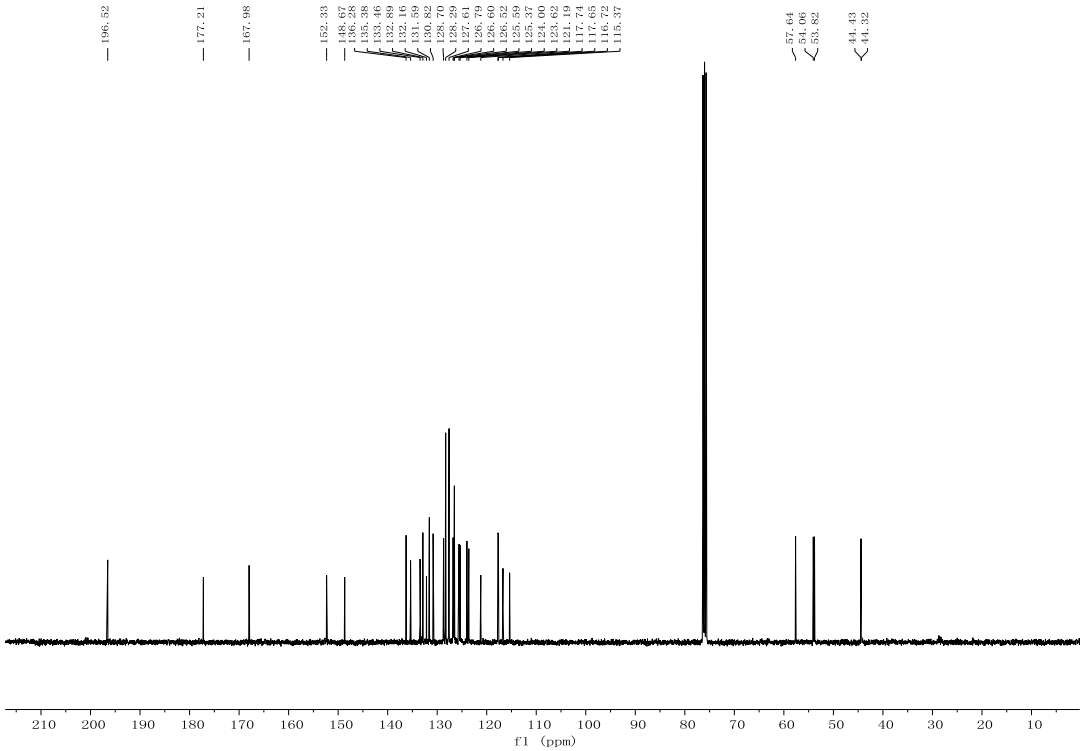
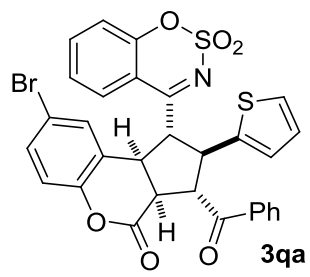
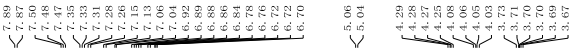





O
SO2
N
Br
S
H
Ph
O
H
O
O

3qa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7.15
7.13
7.06
7.04
6.92
6.89
6.88
6.86
6.84
6.78
6.76
6.72
6.72
6.70
5.06
5.04
4.29
4.28
4.27
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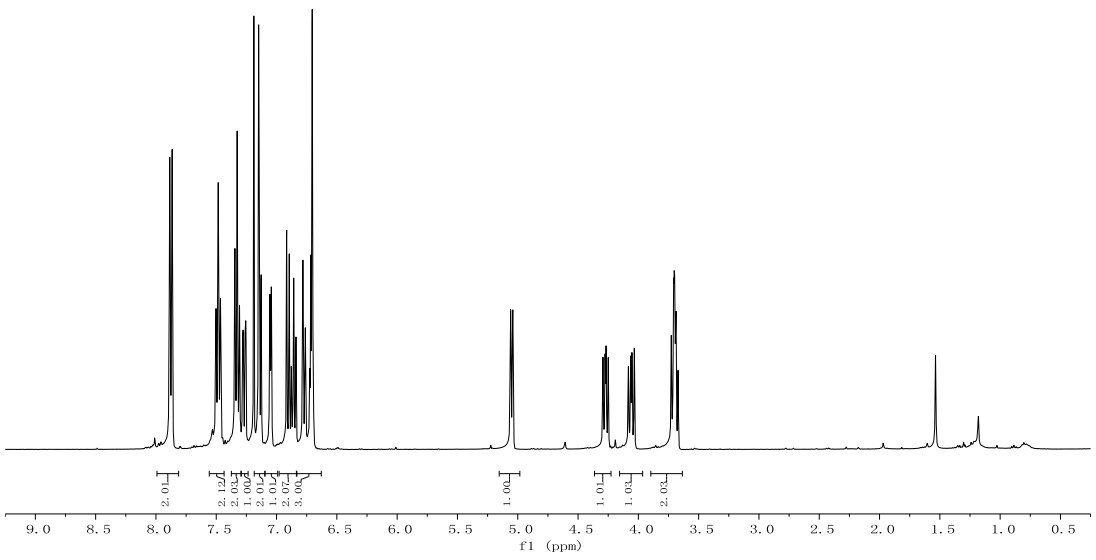
3.67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2.01
1.01
2.07
3.00
1.00
1.01
1.03
2.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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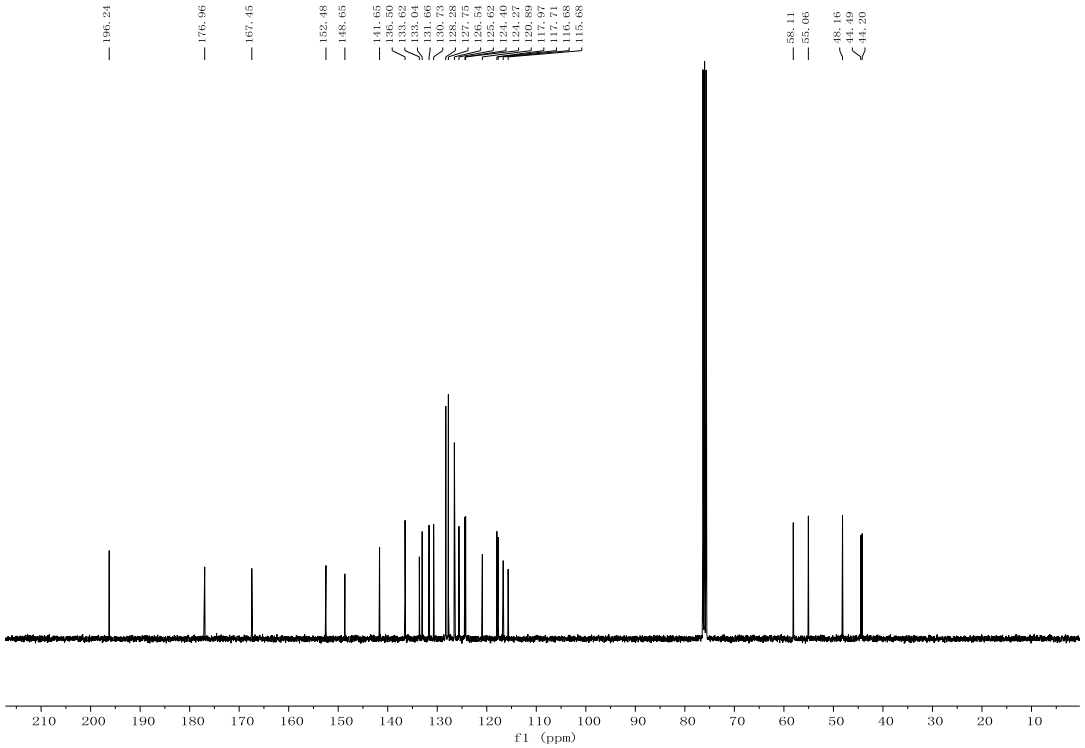
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


196.24
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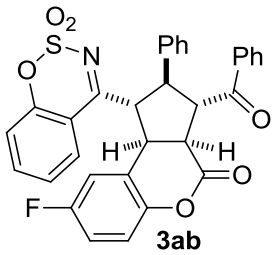
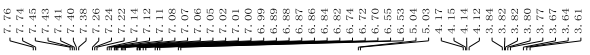
167.45
152.48
148.65
141.65
136.50
133.62
133.04
131.66
130.73
128.28
127.75
126.54
125.62
124.40
124.27
120.89
117.97
117.71
116.68
115.68
58.11
55.06
48.16
44.49
44.20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O2
S
N
O
Ph
Ph
O
H
H
O
F
O

3ab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7.43
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7.26
7.24
7.22
7.14
7.12
7.11
7.08
7.07
7.06
7.05
7.02
7.01
7.00
6.99
6.89
6.88
6.87
6.86
6.84
6.82
6.74
6.72
6.70
6.55
6.53
5.04
5.03
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4.15
4.14
4.12
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3.82
3.82
3.80
3.77
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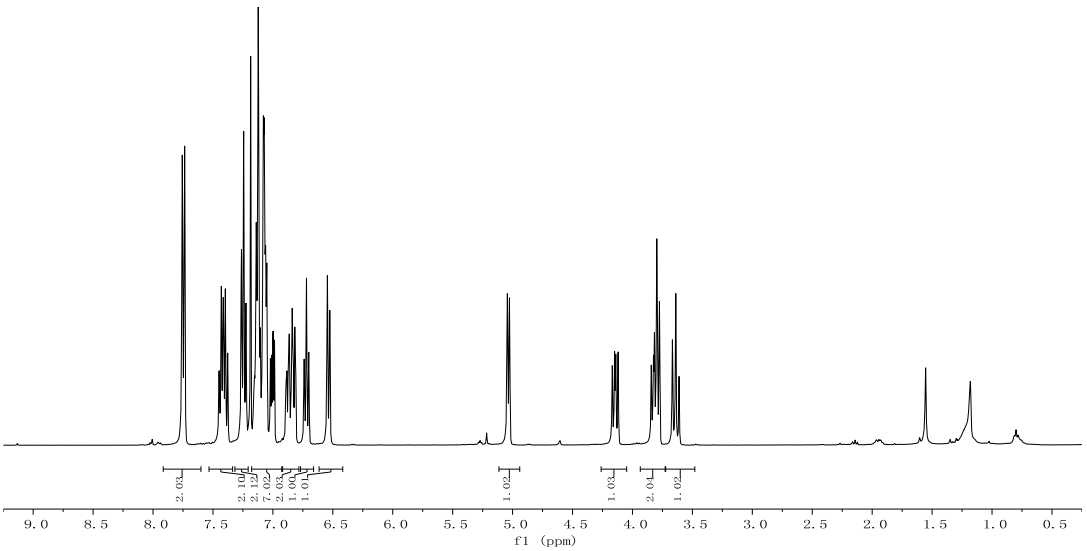
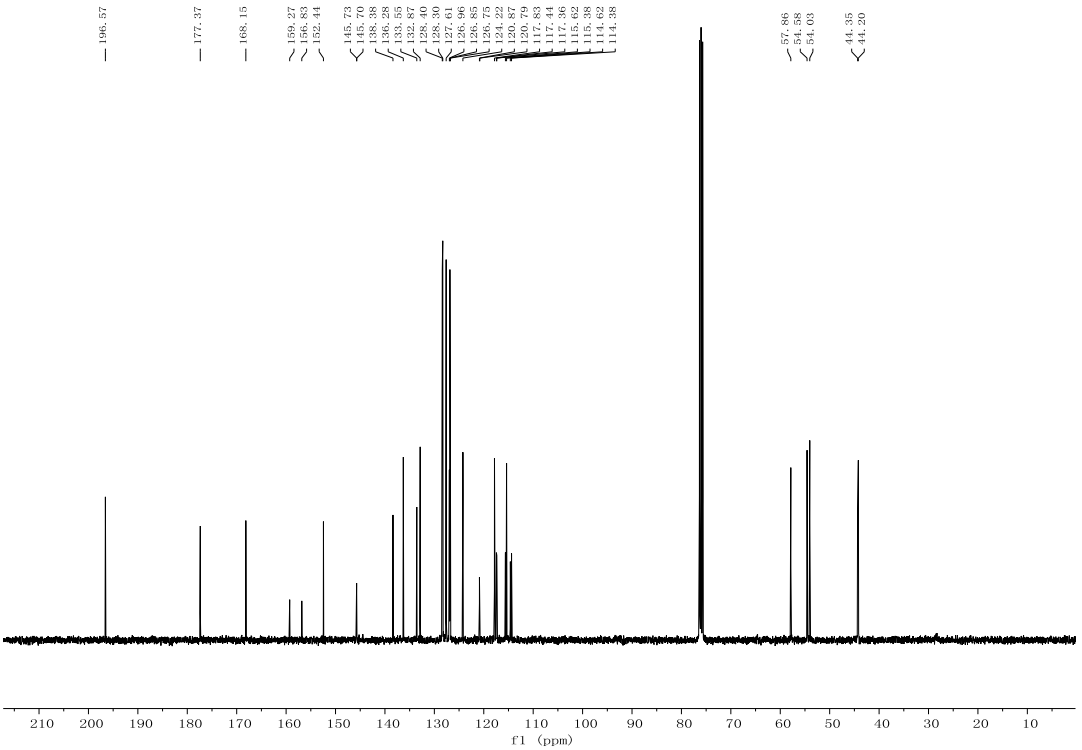
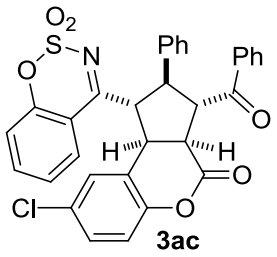
3.64
3.61

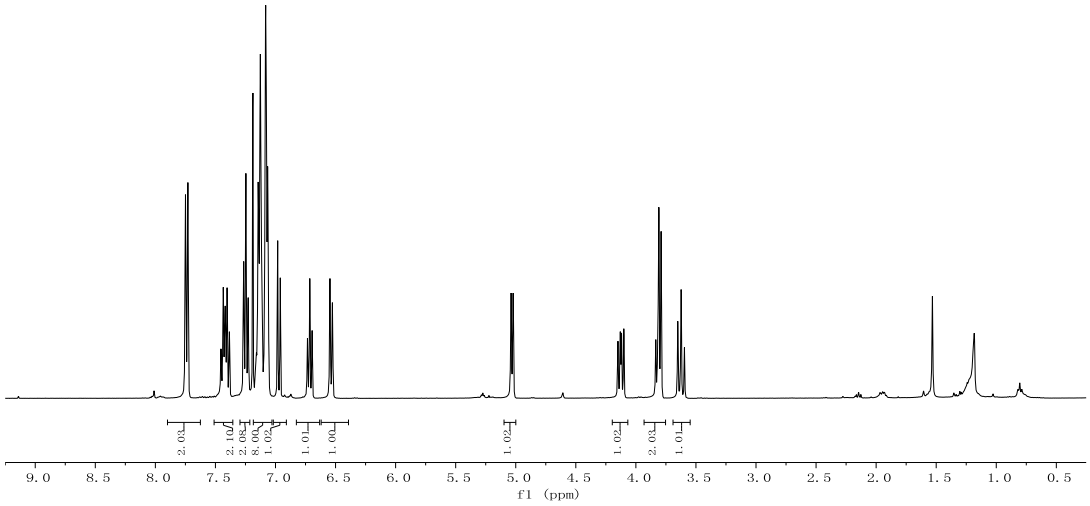

2.03
2.10
2.12
7.02
2.03
2.00
1.01
1.02
1.03
2.04
1.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

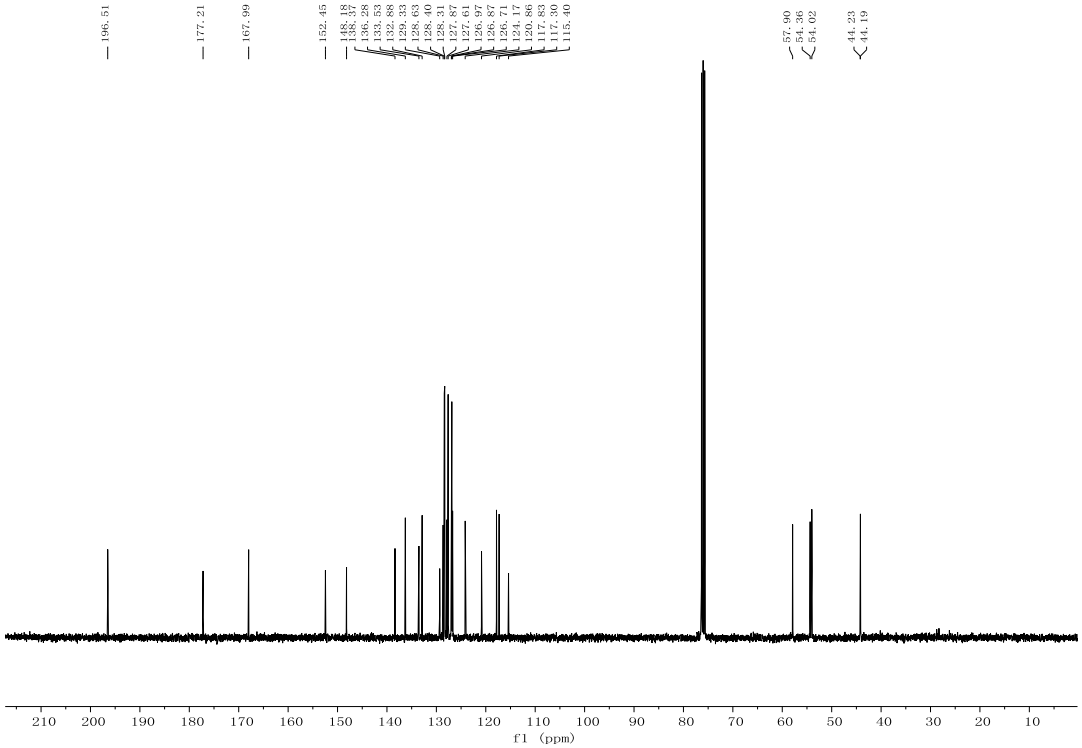

196.57
177.37
168.15
159.27
156.83
152.44
145.73
145.70
138.38
136.28
133.55
132.87
128.40
128.30
127.61
126.96
126.85
126.75
124.22
120.87
120.79
117.83
117.44
117.36
115.62
115.38
114.62
114.38
57.86
54.58
54.03
44.35
44.20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O2
S
N
O
Ph
Ph
O
H
H
O
Cl
O

3ac


7.75
7.73
7.45
7.43
7.42
7.40
7.38
7.36
7.25
7.23
7.14
7.13
7.08
7.07
6.98
6.96
6.73
6.71
6.70
6.55
6.53
5.04
5.02
4.15
4.13
4.12
4.10
3.83
3.81
3.79
3.65
3.62
3.60


2.03
2.10
2.08
8.00
1.02
1.01
1.00
1.02
1.02
2.03
1.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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177.21
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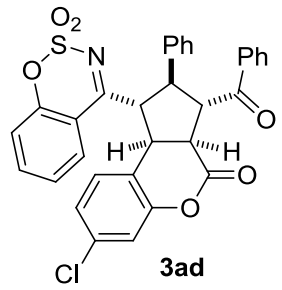
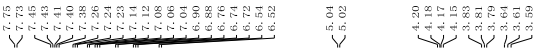
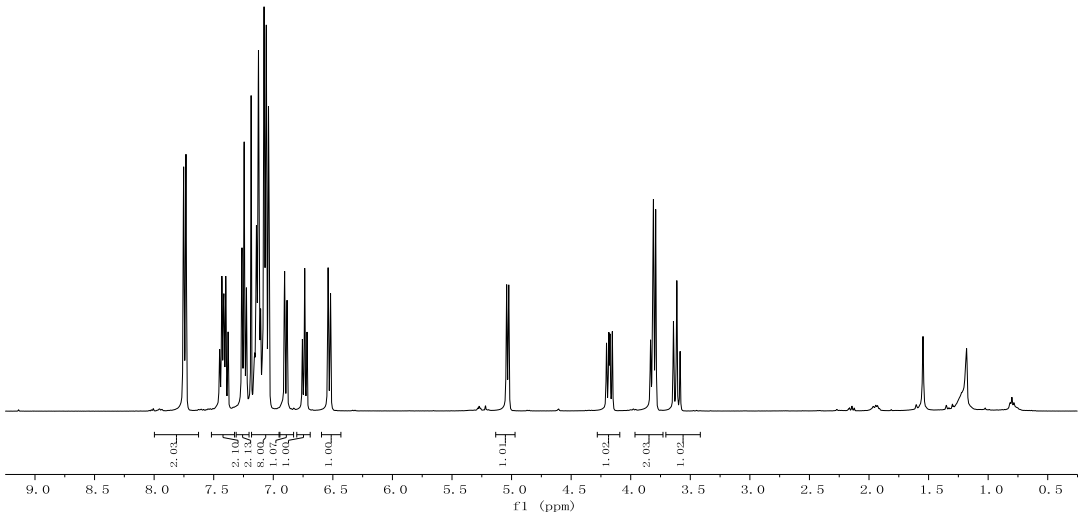
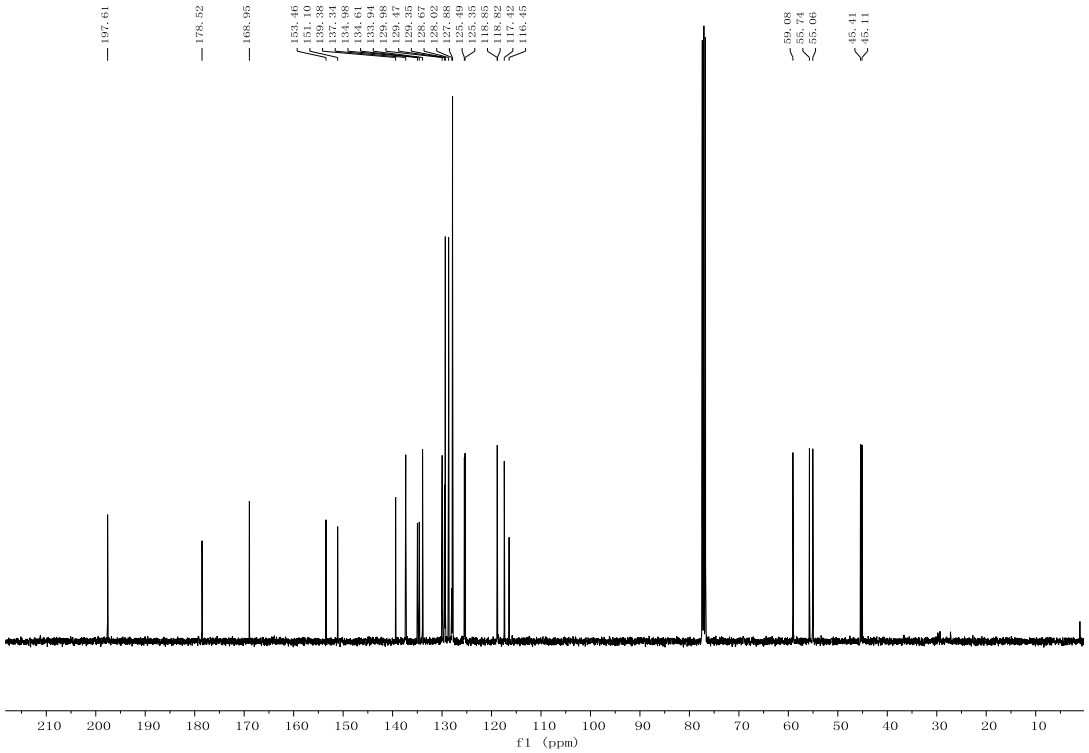
148.18
138.37
136.28
133.53
132.88
129.33
128.63
128.40
128.31
127.87
127.61
126.97
126.57
126.71
124.17
120.86
117.83
117.30
115.40
57.90
54.36
54.02
44.23
44.19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

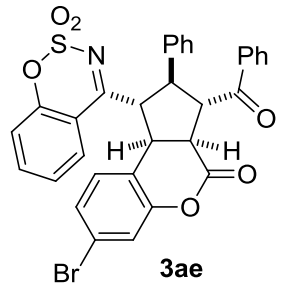

O2
S
N
O
Ph
Ph
O
H
H
O
O
Cl

3ad


7.75
7.73
7.45
7.43
7.41
7.40
7.38
7.26
7.24
7.23
7.14
7.12
7.08
7.06
7.04
6.90
6.88
6.76
6.74
6.72
6.54
6.52
5.04
5.02
4.20
4.18
4.17
4.15
3.83
3.81
3.79
3.64
3.61
3.59

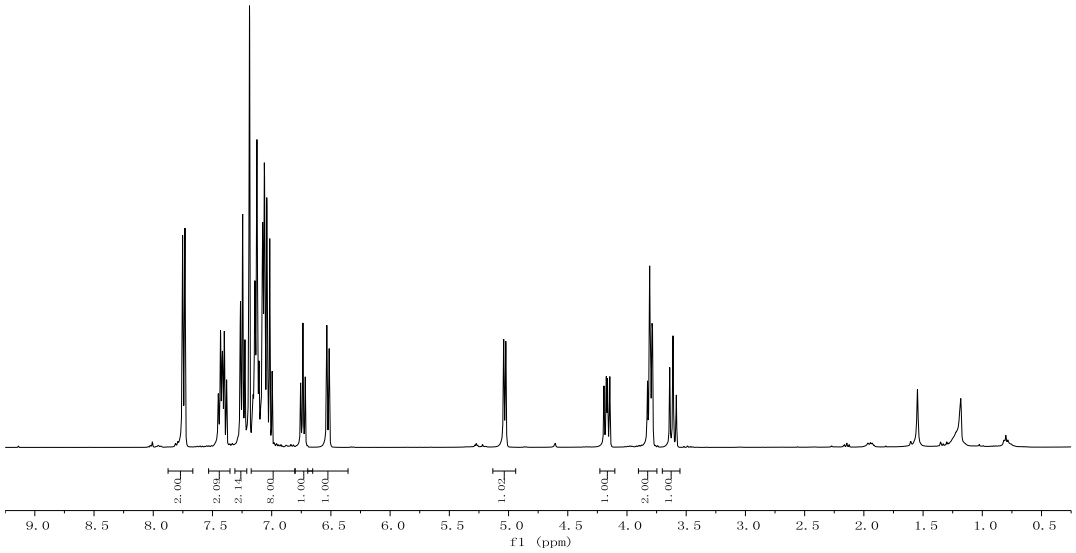

2.03
2.10
2.13
8.06
1.07
1.06
1.06
1.01
1.02
2.03
1.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

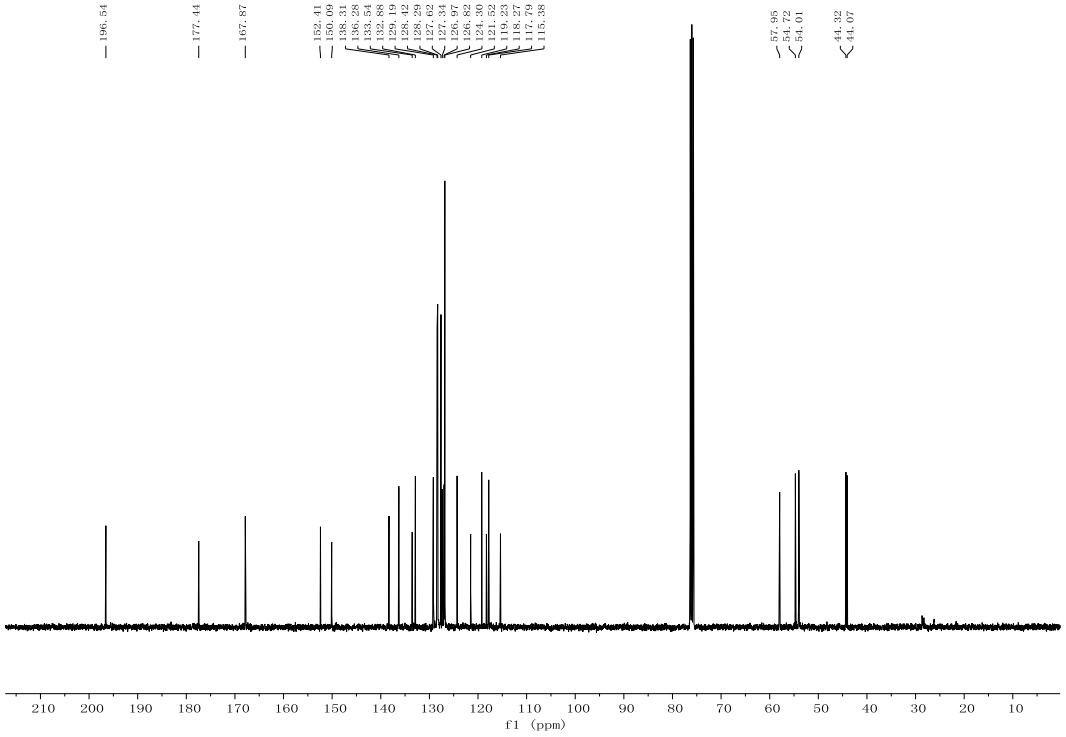

197.61
178.52
168.95
153.46
151.10
139.38
137.34
134.98
134.61
133.94
129.98
129.47
129.35
128.67
128.02
127.88
125.49
125.35
118.85
118.82
117.42
116.45
59.08
55.74
55.06
45.41
45.11
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O2
S
N
O
Ph
Ph
O
H
H
O
O
Br

3ae


7.75
7.73
7.45
7.43
7.42
7.40
7.38
7.26
7.24
7.23
7.14
7.12
7.11
7.08
7.06
7.04
7.02
7.00
6.76
6.74
6.72
6.53
6.51
5.04
5.02
4.19
4.17
4.16
4.15
3.83
3.81
3.79
3.79
3.64
3.61
3.58


2.00
2.09
2.14
8.00
1.00
1.00
1.02
1.00
2.00
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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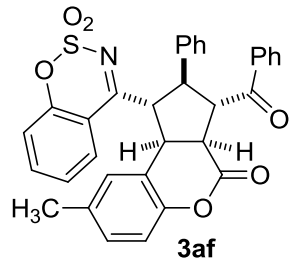
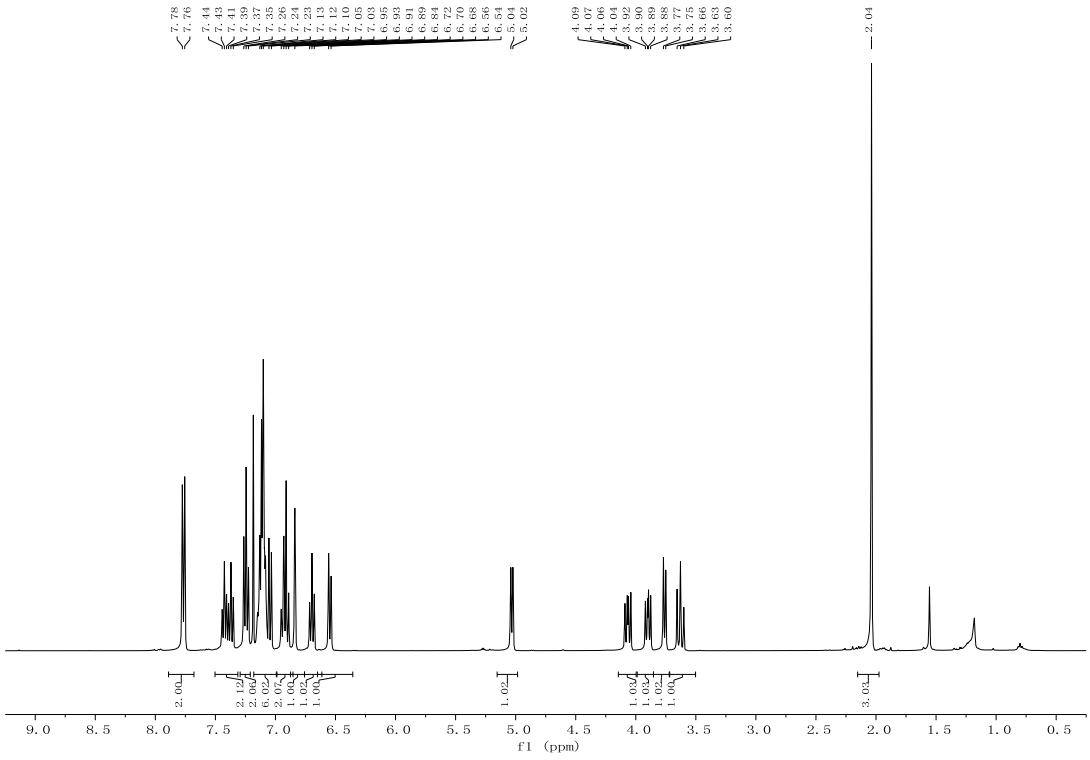
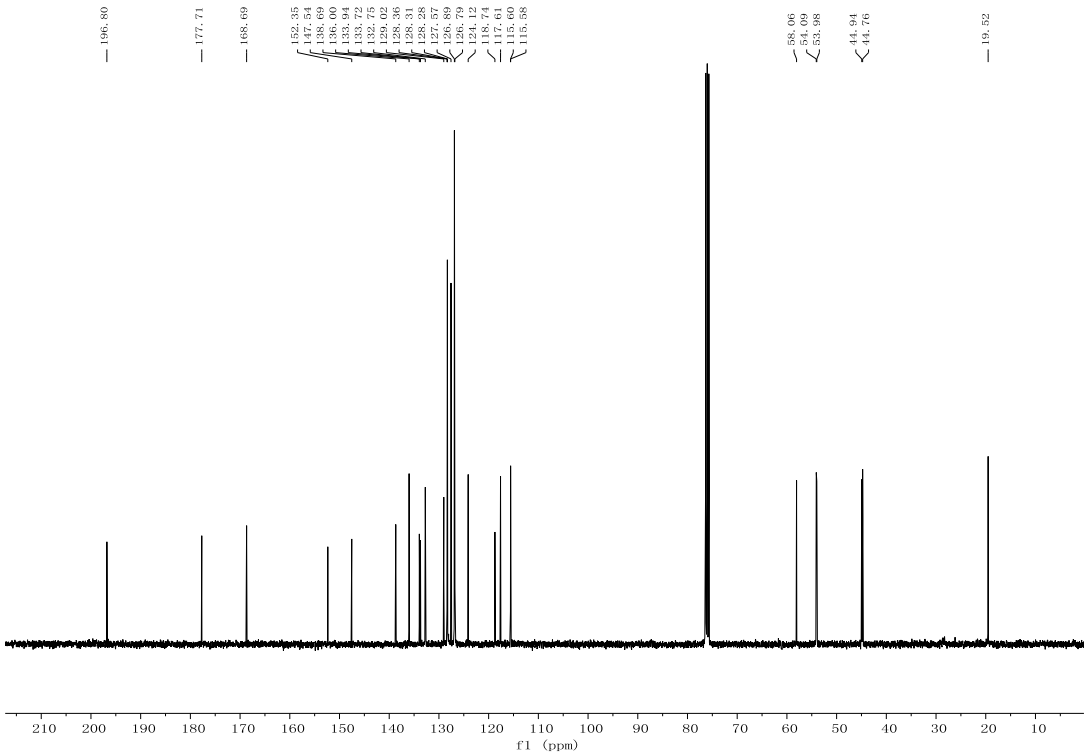
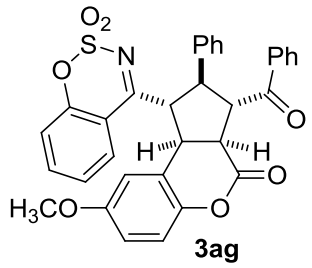
177.44
167.87
152.41
150.09
138.31
136.28
133.54
132.88
129.19
128.42
128.29
127.62
127.34
126.97
126.82
124.30
124.52
119.23
118.27
117.79
115.38
57.95
54.72
54.01
44.32
44.07
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O2
S
N
O
Ph
Ph
O
H
H
O
H3C
O

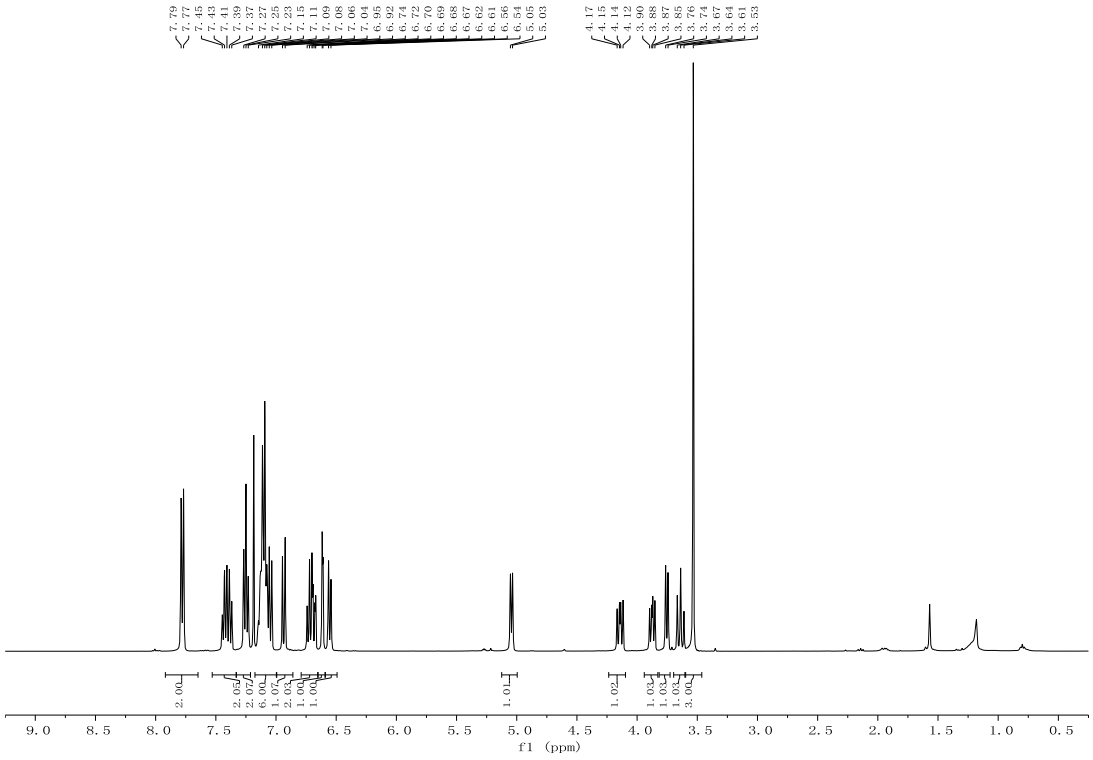
3af

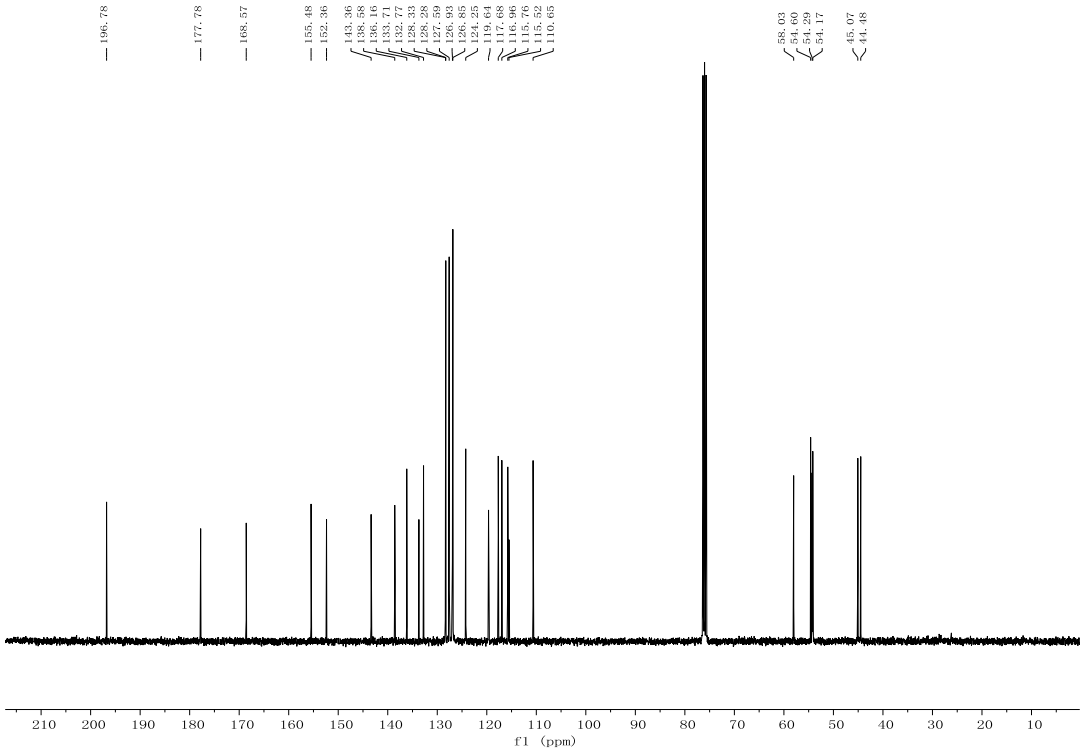

7.78
7.76
7.44
7.43
7.41
7.39
7.37
7.35
7.26
7.24
7.23
7.13
7.12
7.10
7.05
7.03
6.95
6.93
6.91
6.89
6.84
6.72
6.70
6.68
6.56
6.54
5.04
5.02
4.09
4.07
4.06
4.04
3.92
3.90
3.89
3.88
3.77
3.75
3.66
3.63
3.60
2.04
2.00
2.19
2.06
6.02
2.07
1.00
1.02
1.00
1.02
1.03
1.02
1.02
1.00
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

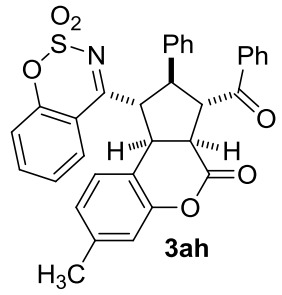

196.80
177.71
168.69
152.35
147.54
138.69
136.00
133.94
133.72
132.75
129.02
128.36
128.31
128.28
127.57
126.89
126.79
124.12
118.74
117.61
115.60
115.58
58.06
54.09
53.98
44.94
44.76
19.52
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



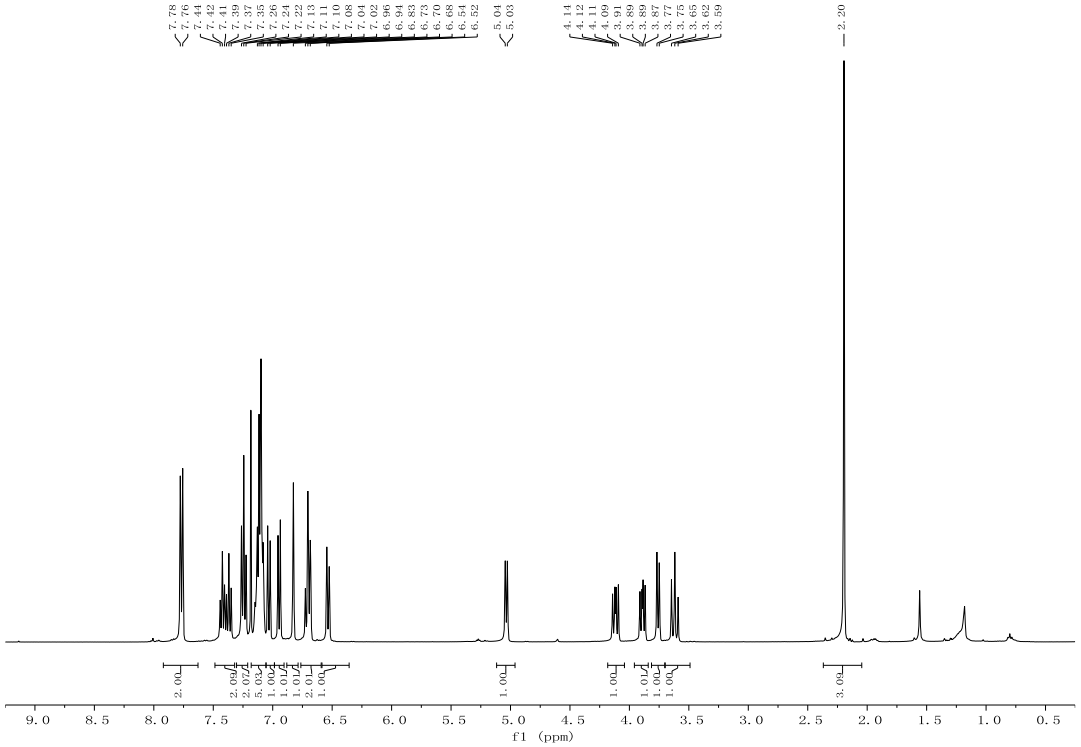
3ag






O2
S
N
O
Ph
Ph
O
H
H
O
O
H3C

3ah


7.78
7.76
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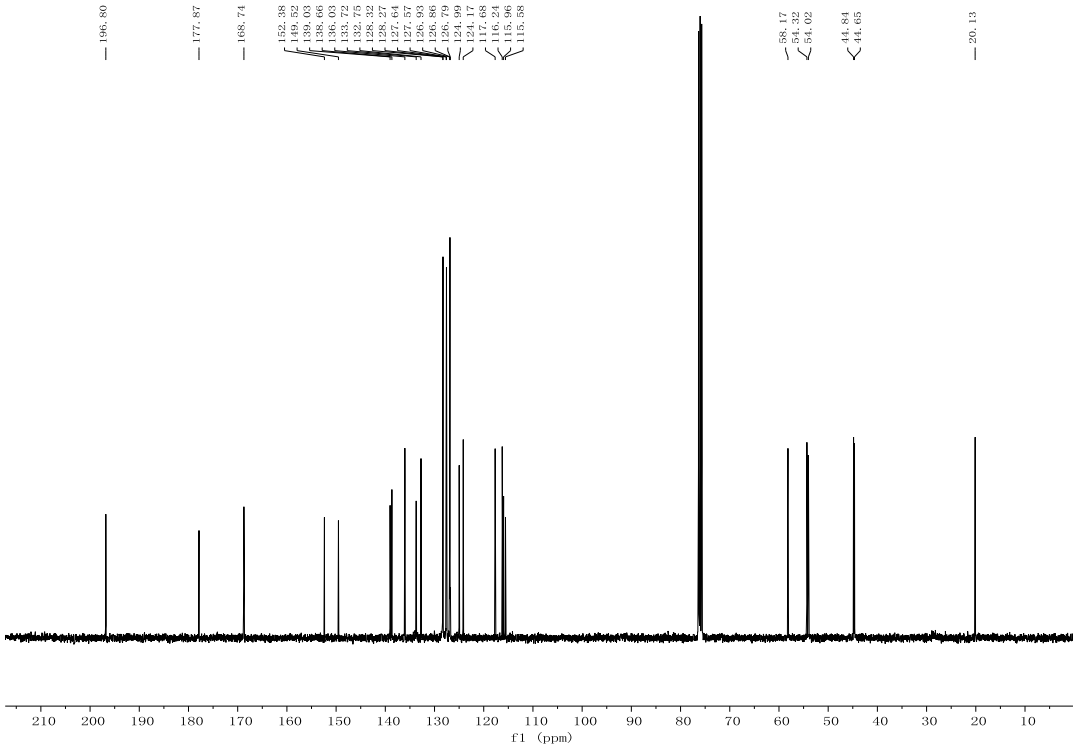
7.42
7.41
7.39
7.37
7.35
7.26
7.24
7.22
7.13
7.11
7.10
7.08
7.04
7.02
6.96
6.94
6.83
6.73
6.70
6.68
6.54
6.52
5.04
5.03
4.14
4.12
4.11
4.09
3.91
3.89
3.89
3.87
3.77
3.75
3.65
3.62
3.59
2.20
2.00
2.00
2.07
5.03
1.00
1.01
2.01
1.04
1.00
1.00
1.01
1.00
1.00
3.09
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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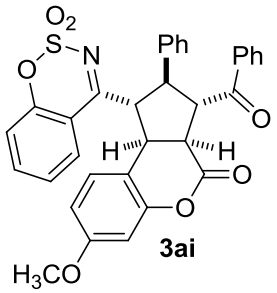
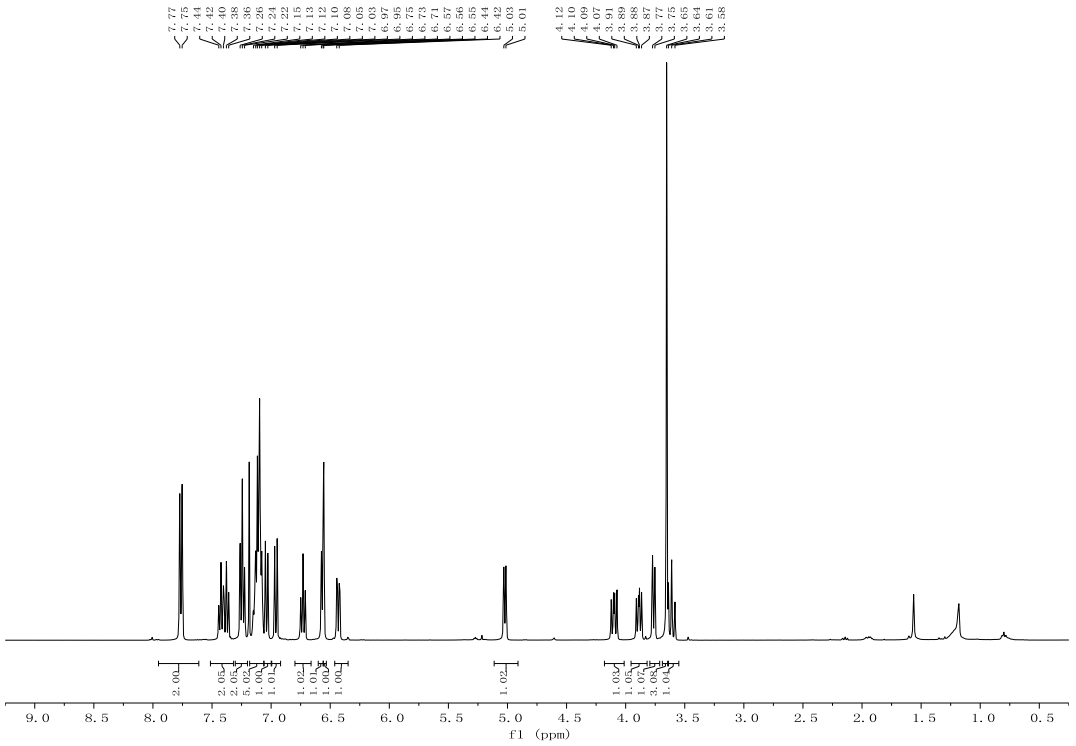
177.87
168.74
152.38
149.32
139.03
138.66
136.03
133.72
132.75
128.32
128.27
127.64
127.57
126.88
126.86
126.79
124.99
124.17
117.68
116.24
115.96
115.58
58.17
54.32
54.02
44.84
44.65
20.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


O2
S
N
O
Ph
Ph
O
H
H
O
O
H3CO

3ai


7.77
7.75
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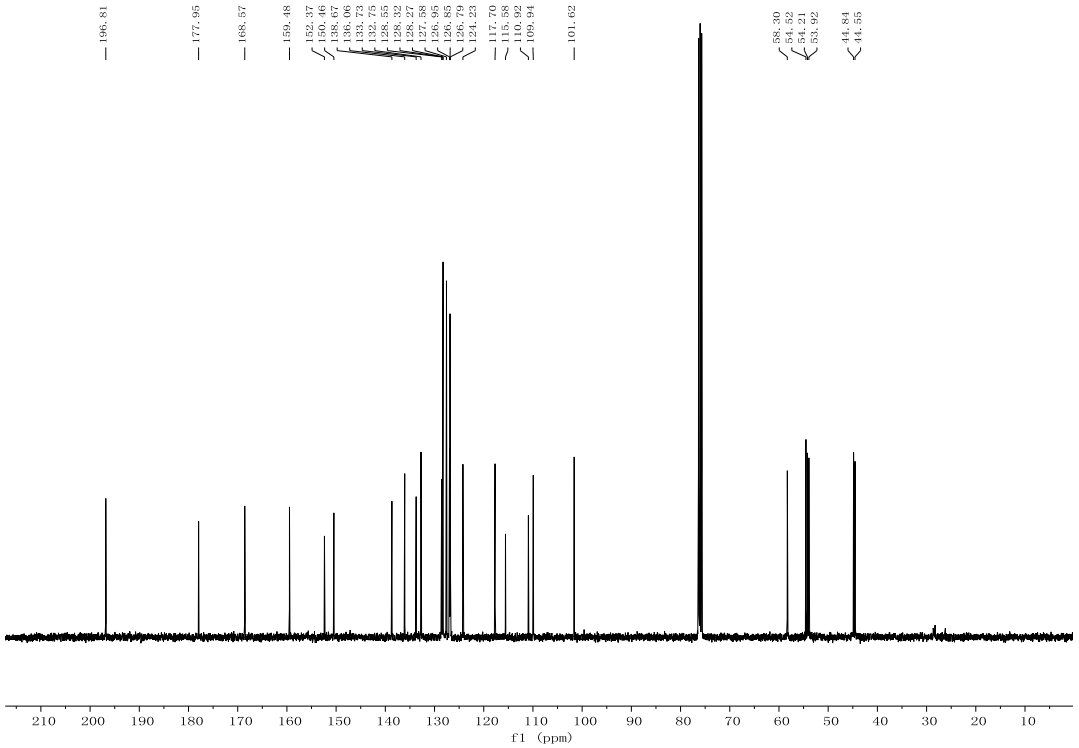
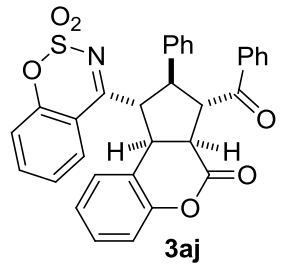
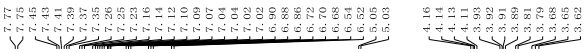
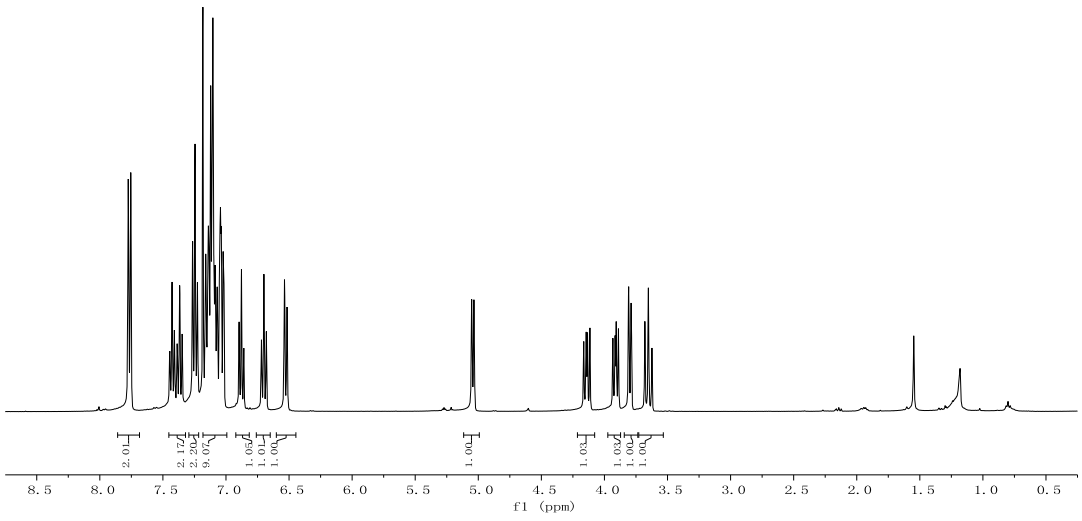
7.42
7.40
7.38
7.36
7.26
7.24
7.22
7.15
7.13
7.12
7.10
7.08
7.05
7.03
6.97
6.95
6.75
6.73
6.71
6.57
6.56
6.55
6.44
6.42
5.03
5.01
4.12
4.10
4.09
4.07
3.91
3.89
3.88
3.87
3.77
3.75
3.65
3.64
3.61
3.58
2.00
2.05
2.05
5.02
1.00
1.01
1.02
1.01
1.00
1.00
1.02
1.03
1.05
1.07
3.08
1.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

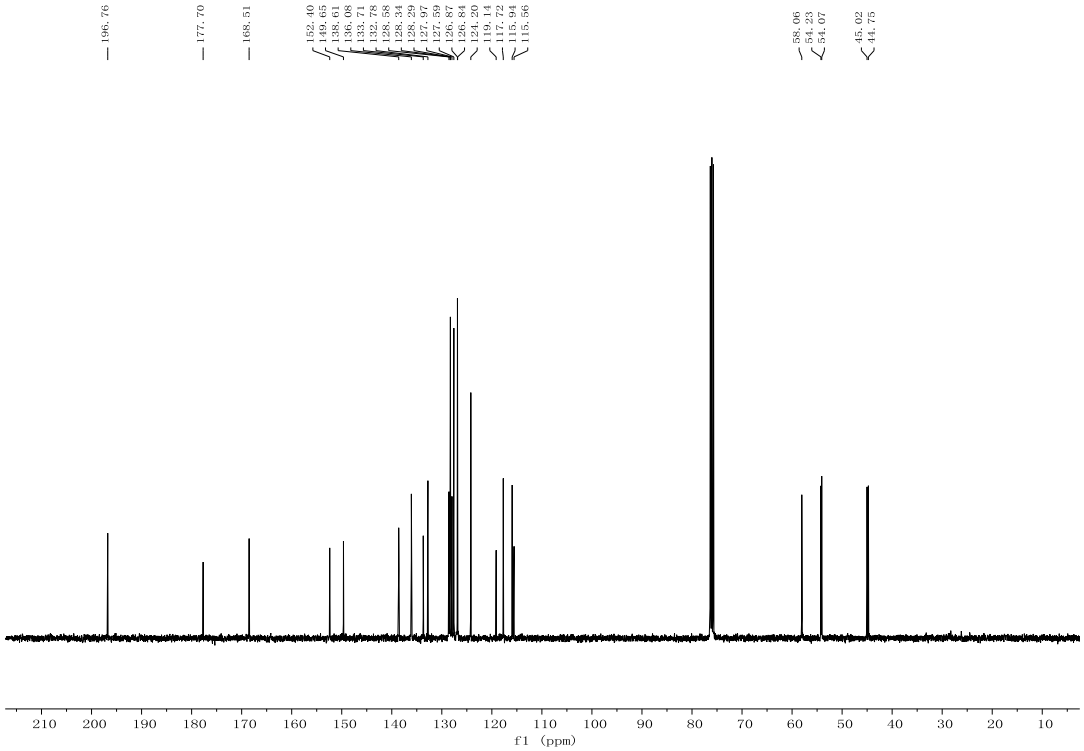

196.81
177.95
168.57
159.48
152.37
150.46
138.67
136.06
133.73
132.75
128.55
128.32
128.27
127.58
126.85
126.65
126.79
124.23
117.70
115.98
110.92
109.94
101.62
58.30
54.52
54.21
53.92
44.84
44.55
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

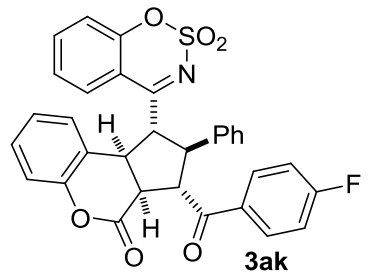


3aj

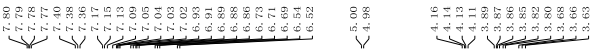


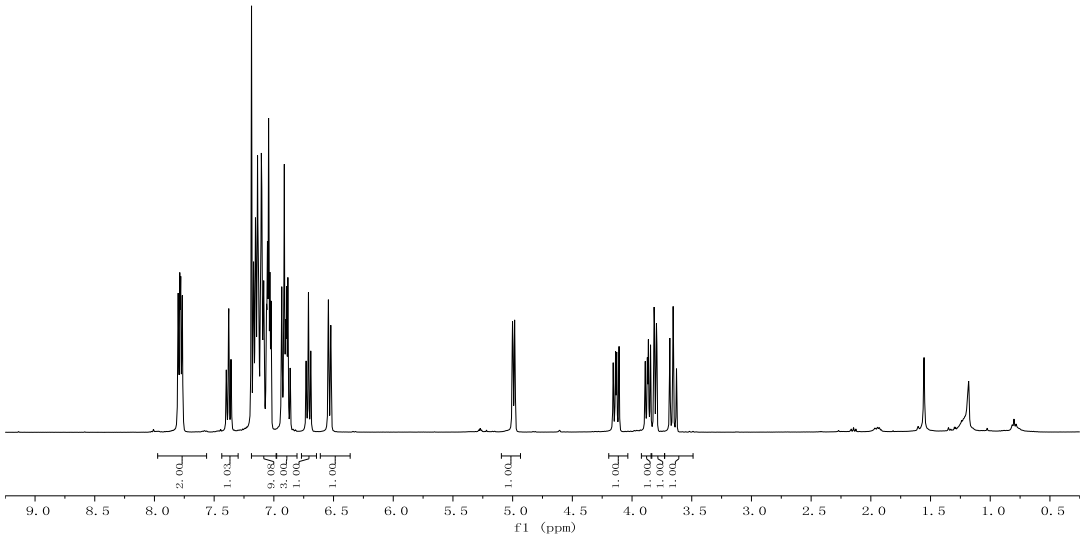


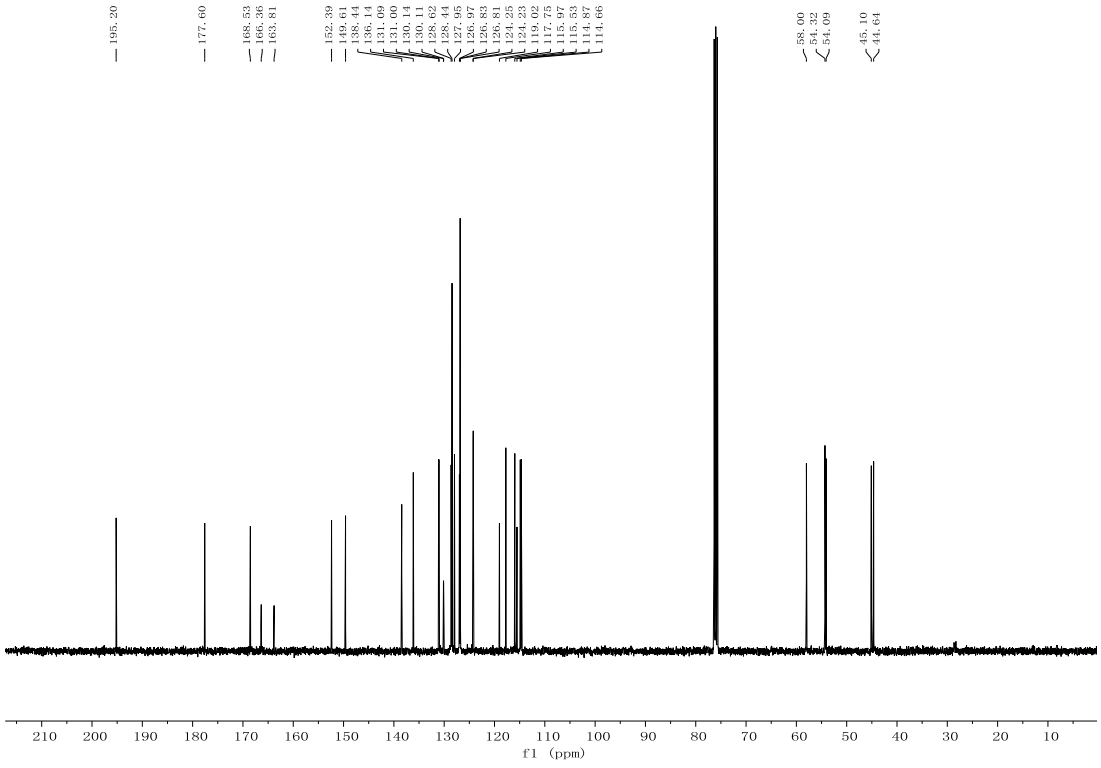



O
SO2
N
H
Ph
F
O
H
O
O

3ak


7.80
7.79
7.78
7.77
7.40
7.38
7.36
7.17
7.15
7.13
7.09
7.05
7.04
7.03
7.02
6.93
6.91
6.89
6.88
6.86
6.73
6.71
6.69
6.54
6.52
5.00
4.98
4.16
4.14
4.13
4.11
3.89
3.87
3.86
3.85
3.82
3.80
3.68
3.66
3.63


2.00
1.04
9.09
3.00
1.00
1.00
1.00
1.00
1.00
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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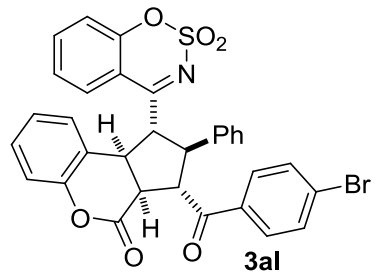
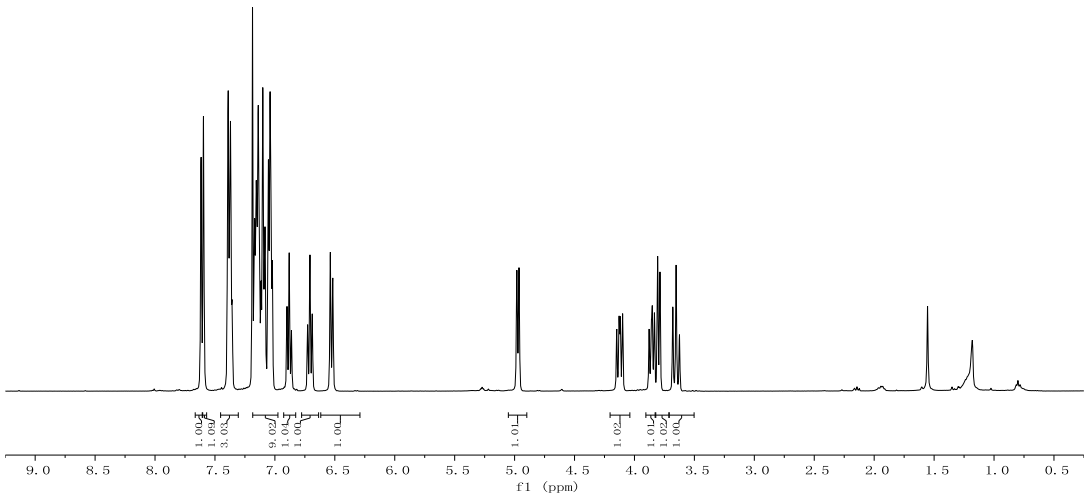
177.60
168.53
166.36
163.81
152.39
149.61
138.44
136.14
131.09
131.00
130.14
130.11
128.62
128.44
127.95
126.97
126.83
126.81
124.25
124.23
119.02
117.75
117.57
115.53
114.87
114.66
58.00
54.32
54.09
45.10
44.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


O
SO2
N
H
Ph
Br
O
H
O
O

3al


7.62
7.60
7.39
7.37
7.36
7.17
7.16
7.14
7.11
7.10
7.08
7.05
7.04
7.02
6.99
6.88
6.86
6.73
6.71
6.69
6.54
6.52
4.98
4.96
4.15
4.13
4.12
4.10
3.88
3.86
3.85
3.83
3.81
3.79
3.68
3.65
3.62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1.09
3.03
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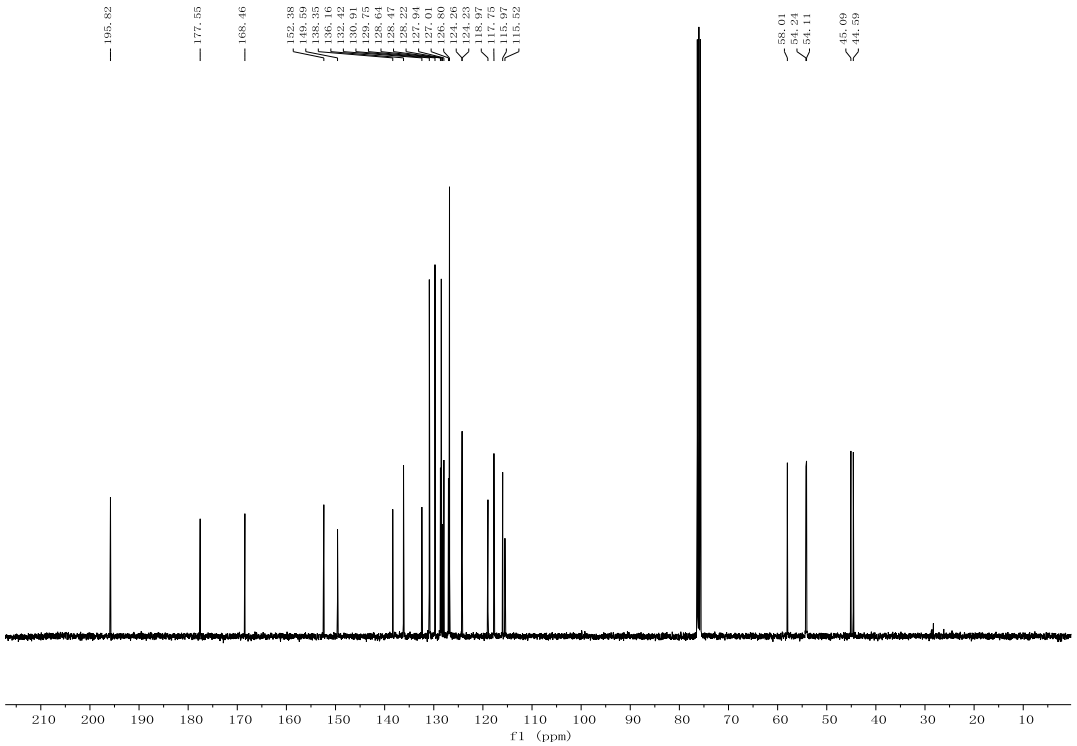
1.01
1.00
1.00
1.01
1.02
1.01
1.02
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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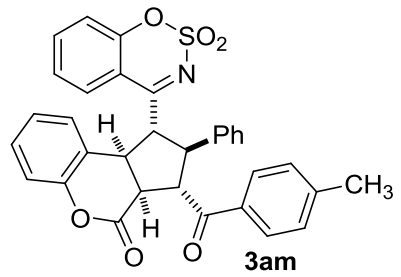
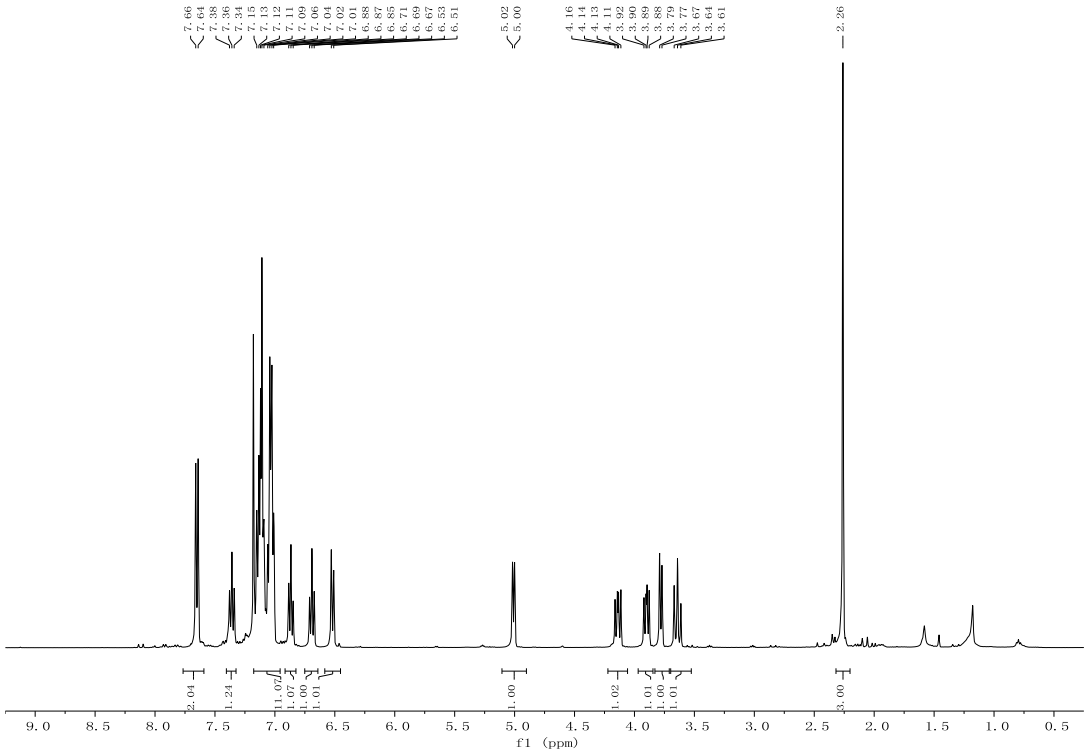
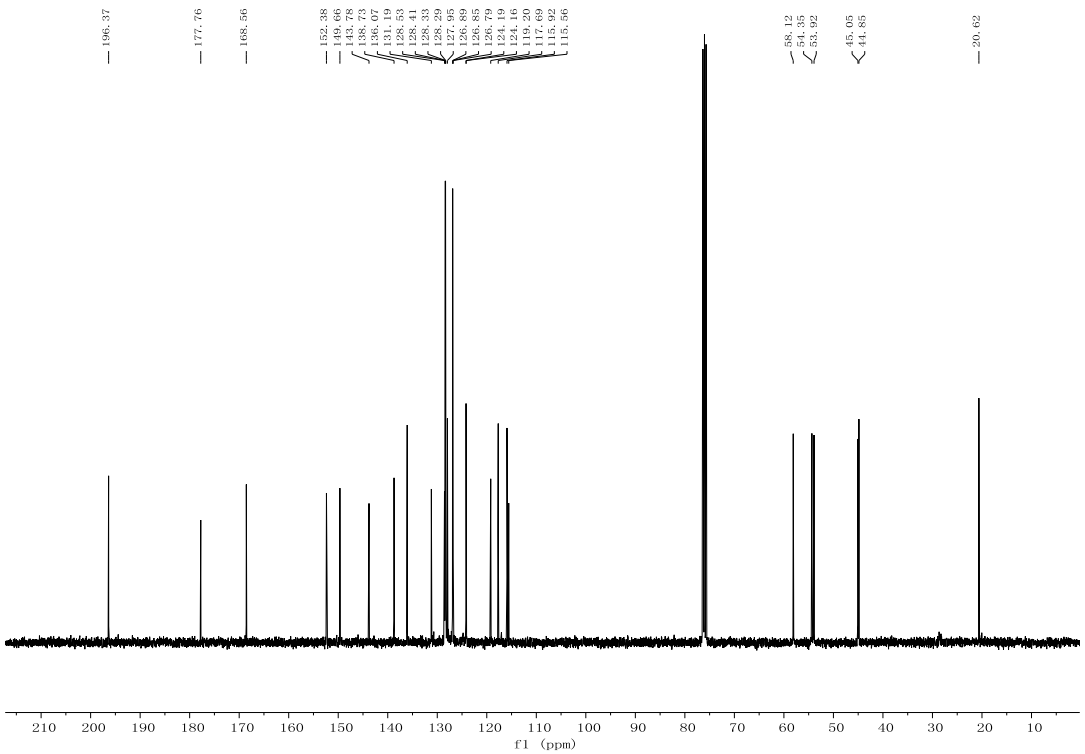
138.35
136.16
132.42
130.91
129.75
128.64
128.47
128.22
127.94
127.01
126.80
124.26
124.23
118.97
117.75
117.57
115.52
58.01
54.24
54.11
45.09
44.59
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O
SO2
N
H
Ph
CH3
O
H
O
O

3am


7.66
7.64
7.38
7.36
7.34
7.15
7.13
7.12
7.11
7.09
7.06
7.04
7.02
7.01
6.88
6.87
6.85
6.71
6.69
6.67
6.53
6.51
5.02
5.00
4.16
4.14
4.13
4.11
3.92
3.90
3.89
3.88
3.79
3.77
3.67
3.64
3.61
2.26
2.04
1.24
11.07
1.07
1.00
1.01
1.00
1.02
1.01
1.00
1.01
3.00
f1 (ppm)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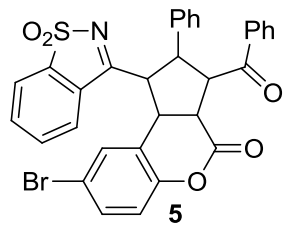
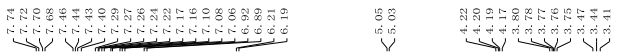
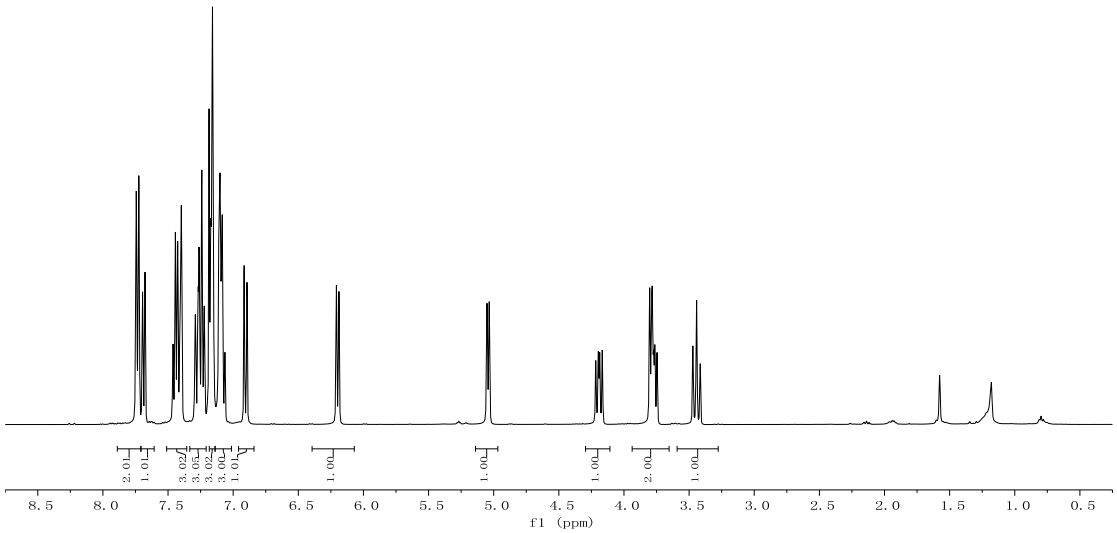
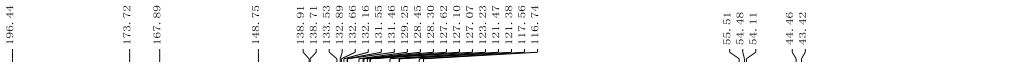
136.07
131.19
128.53
128.41
128.33
128.29
127.95
126.89
126.85
126.79
124.19
124.16
119.20
117.69
115.92
115.56
58.12
54.35
53.92
45.05
44.85
20.62
f1 (ppm)

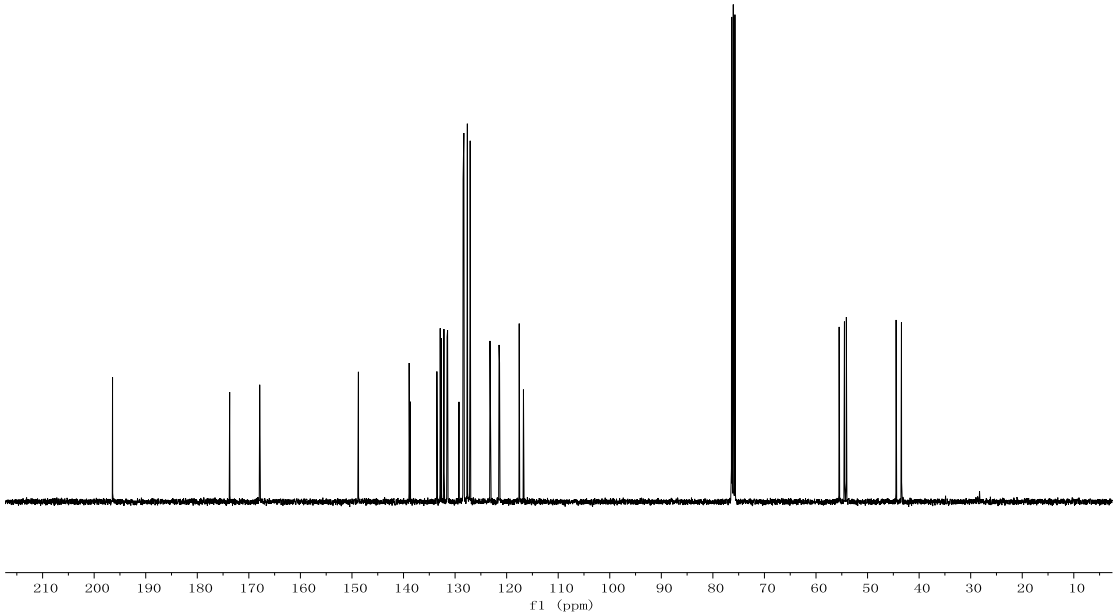

O2S–N
Ph
Ph
O
O
Br
O

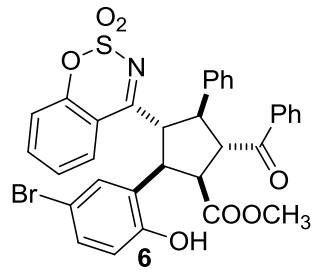
5


7.74
7.72
7.70
7.68
7.46
7.44
7.43
7.40
7.29
7.27
7.26
7.24
7.22
7.17
7.16
7.10
7.08
7.06
6.92
6.89
6.21
6.19
5.05
5.03
4.22
4.20
4.19
4.17
3.80
3.78
3.77
3.76
3.75
3.47
3.44
3.41

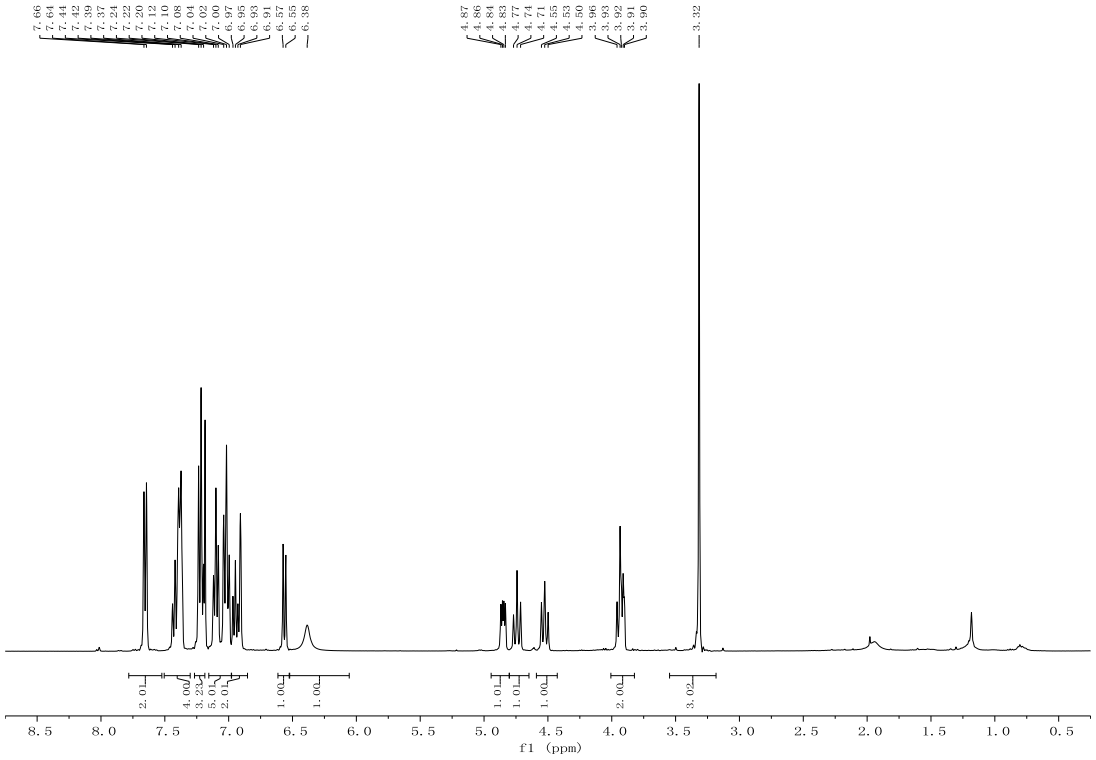

2.01
1.01
3.02
3.05
3.02
3.00
1.01
1.00
1.00
1.00
2.00
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

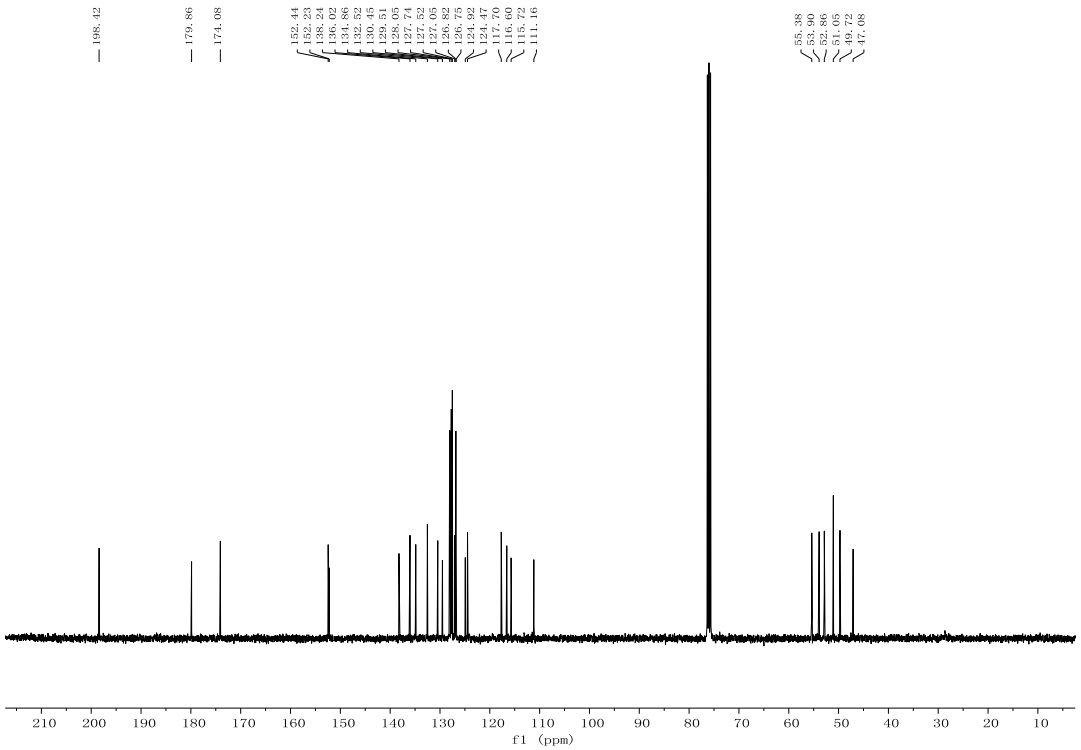

196.44
173.72
167.89
148.75
138.91
138.71
133.53
132.89
132.66
132.16
131.55
131.46
129.25
128.45
128.30
127.62
127.10
127.07
123.23
121.47
121.38
117.56
116.74
55.51
54.48
54.11
44.46
43.42


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


O2
S
O
N
Ph
Ph
O
Br
COOCH3
OH

6


7.66
7.64
7.44
7.42
7.39
7.37
7.24
7.22
7.20
7.12
7.10
7.08
7.04
7.02
7.00
6.97
6.95
6.93
6.84
6.57
6.55
6.38
4.87
4.86
4.84
4.83
4.77
4.74
4.71
4.55
4.53
4.50
3.96
3.93
3.92
3.91
3.90
3.32
2.01
4.00
3.22
5.01
2.01
1.00
1.00
1.01
1.01
1.00
2.00
3.02
f1 (ppm)


198.42
179.86
174.08
152.44
152.23
138.24
136.02
134.86
132.52
130.45
129.51
128.05
127.74
127.53
127.05
126.82
126.75
124.92
124.47
117.70
116.60
115.72
111.16
55.38
53.90
52.86
51.05
49.72
47.08
f1 (ppm)

X-Ray Crystallography Data

Crystallographic data for **3aj** have been deposited with the Cambridge Crystallographic Data Centre as deposition number CCDC 2073841. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

X-Ray Crystallography Data of **3aj**

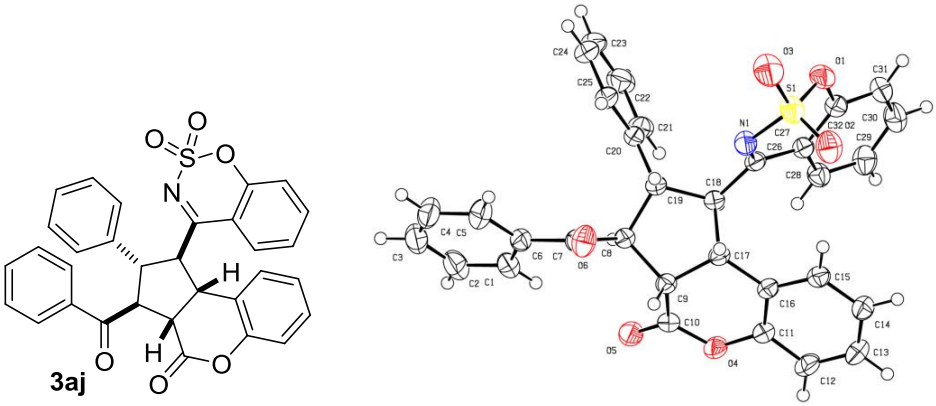


Table 1. Crystal data and structure refinement for **3aj**.

Identification code	**3aj**	
Empirical formula	C32 H23 N O6 S	
Formula weight	549.57	
Temperature	193(2) K	
Wavelength	1.34139 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.5537(10) Å	α= 114.381(3)°.
	b = 11.0474(11) Å	β= 99.000(3)°.
	c = 13.2402(13) Å	γ = 101.915(3)°.
Volume	1324.2(2) $Å^3$	
Z	2	
Density (calculated)	1.378 Mg/m^3	
Absorption coefficient	0.967 mm^{-1}	
F(000)	572	
Crystal size	0.160 x 0.120 x 0.110 mm^3	
Theta range for data collection	3.312 to 52.989°.	
Index ranges	-12<=h<=12, -13<=k<=13, -15<=l<=15	

Reflections collected	13998
Independent reflections	4655 [R(int) = 0.0673]
Completeness to theta = 52.989°	99.2 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4655 / 0 / 361
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0758, wR2 = 0.2104
R indices (all data)	R1 = 0.0793, wR2 = 0.2158
Extinction coefficient	n/a
Largest diff. peak and hole	0.382 and -0.502 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3aj**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	-1134(2)	1396(2)	6040(2)	43(1)
C(2)	-2256(3)	578(3)	6134(2)	54(1)
C(3)	-2099(3)	-105(3)	6792(2)	54(1)
C(4)	-825(3)	14(3)	7351(2)	56(1)
C(5)	291(3)	799(3)	7243(2)	49(1)
C(6)	139(2)	1508(2)	6590(2)	36(1)
C(7)	1372(2)	2343(2)	6486(2)	33(1)
C(8)	1387(2)	3694(2)	6416(2)	29(1)
C(9)	1865(2)	3736(2)	5371(2)	29(1)
C(10)	728(2)	3712(2)	4508(2)	32(1)
C(11)	2303(2)	5260(2)	4111(2)	33(1)
C(12)	2459(2)	5776(3)	3328(2)	43(1)
C(13)	3683(3)	6700(3)	3517(2)	45(1)
C(14)	4731(2)	7101(2)	4471(2)	38(1)
C(15)	4558(2)	6558(2)	5234(2)	33(1)
C(16)	3327(2)	5631(2)	5068(2)	29(1)
C(17)	3113(2)	5019(2)	5877(2)	28(1)
C(18)	2926(2)	5986(2)	7048(2)	27(1)
C(19)	2455(2)	4929(2)	7489(2)	29(1)
C(20)	1977(2)	5481(2)	8562(2)	33(1)
C(21)	1100(2)	6293(2)	8696(2)	40(1)
C(22)	710(3)	6798(3)	9707(2)	52(1)
C(23)	1185(3)	6506(3)	10591(2)	58(1)

C(24)	2030(3)	5695(3)	10458(2)	55(1)
C(25)	2433(2)	5183(2)	9454(2)	42(1)
C(26)	4239(2)	7102(2)	7812(2)	29(1)
C(27)	4464(2)	8527(2)	7987(2)	31(1)
C(28)	3596(2)	8920(2)	7344(2)	36(1)
C(29)	3863(2)	10291(3)	7547(2)	44(1)
C(30)	4980(3)	11296(2)	8414(2)	47(1)
C(31)	5852(2)	10952(2)	9060(2)	43(1)
C(32)	5593(2)	9574(2)	8826(2)	35(1)
N(1)	5139(2)	6686(2)	8253(2)	35(1)
O(1)	6479(2)	9251(2)	9507(1)	42(1)
O(2)	7350(2)	7697(2)	8048(2)	52(1)
O(3)	7239(2)	7502(2)	9809(2)	61(1)
O(4)	1025(2)	4367(2)	3869(1)	39(1)
O(5)	-431(2)	3100(2)	4317(1)	44(1)
O(6)	2389(2)	1992(2)	6493(2)	46(1)
S(1)	6667(1)	7736(1)	8897(1)	40(1)

Table 3. Bond lengths [Å] and angles [°] for **3aj**.

C(1)-C(6)	1.379(3)
C(1)-C(2)	1.389(3)
C(1)-H(1)	0.9500
C(2)-C(3)	1.380(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.381(4)
C(3)-H(3)	0.9500
C(4)-C(5)	1.377(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.398(3)
C(5)-H(5)	0.9500
C(6)-C(7)	1.492(3)
C(7)-O(6)	1.213(3)
C(7)-C(8)	1.531(3)
C(8)-C(19)	1.554(3)
C(8)-C(9)	1.560(2)
C(8)-H(8)	1.0000
C(9)-C(10)	1.509(3)

C(9)-C(17)	1.536(3)
C(9)-H(9)	1.0000
C(10)-O(5)	1.200(3)
C(10)-O(4)	1.354(2)
C(11)-C(16)	1.379(3)
C(11)-C(12)	1.388(3)
C(11)-O(4)	1.397(2)
C(12)-C(13)	1.384(3)
C(12)-H(12)	0.9500
C(13)-C(14)	1.384(3)
C(13)-H(13)	0.9500
C(14)-C(15)	1.388(3)
C(14)-H(14)	0.9500
C(15)-C(16)	1.399(3)
C(15)-H(15)	0.9500
C(16)-C(17)	1.506(2)
C(17)-C(18)	1.549(2)
C(17)-H(17)	1.0000
C(18)-C(26)	1.513(3)
C(18)-C(19)	1.537(2)
C(18)-H(18)	1.0000
C(19)-C(20)	1.511(3)
C(19)-H(19)	1.0000
C(20)-C(25)	1.394(3)
C(20)-C(21)	1.399(3)
C(21)-C(22)	1.386(3)
C(21)-H(21)	0.9500
C(22)-C(23)	1.386(4)
C(22)-H(22)	0.9500
C(23)-C(24)	1.370(5)
C(23)-H(23)	0.9500
C(24)-C(25)	1.387(3)
C(24)-H(24)	0.9500
C(25)-H(25)	0.9500
C(26)-N(1)	1.297(3)
C(26)-C(27)	1.455(3)
C(27)-C(32)	1.395(3)
C(27)-C(28)	1.406(3)

C(28)-C(29)	1.382(3)
C(28)-H(28)	0.9500
C(29)-C(30)	1.387(4)
C(29)-H(29)	0.9500
C(30)-C(31)	1.374(3)
C(30)-H(30)	0.9500
C(31)-C(32)	1.379(3)
C(31)-H(31)	0.9500
C(32)-O(1)	1.398(3)
N(1)-S(1)	1.6291(17)
O(1)-S(1)	1.6013(18)
O(2)-S(1)	1.4185(18)
O(3)-S(1)	1.4089(18)
C(6)-C(1)-C(2)	120.0(2)
C(6)-C(1)-H(1)	120.0
C(2)-C(1)-H(1)	120.0
C(3)-C(2)-C(1)	120.1(3)
C(3)-C(2)-H(2)	119.9
C(1)-C(2)-H(2)	119.9
C(4)-C(3)-C(2)	120.0(2)
C(4)-C(3)-H(3)	120.0
C(2)-C(3)-H(3)	120.0
C(5)-C(4)-C(3)	120.2(3)
C(5)-C(4)-H(4)	119.9
C(3)-C(4)-H(4)	119.9
C(4)-C(5)-C(6)	120.1(3)
C(4)-C(5)-H(5)	120.0
C(6)-C(5)-H(5)	120.0
C(1)-C(6)-C(5)	119.6(2)
C(1)-C(6)-C(7)	121.87(19)
C(5)-C(6)-C(7)	118.5(2)
O(6)-C(7)-C(6)	121.08(19)
O(6)-C(7)-C(8)	118.85(18)
C(6)-C(7)-C(8)	120.03(18)
C(7)-C(8)-C(19)	108.64(15)
C(7)-C(8)-C(9)	112.58(16)
C(19)-C(8)-C(9)	104.40(15)
C(7)-C(8)-H(8)	110.4

C(19)-C(8)-H(8)	110.4
C(9)-C(8)-H(8)	110.4
C(10)-C(9)-C(17)	115.25(16)
C(10)-C(9)-C(8)	110.55(16)
C(17)-C(9)-C(8)	106.26(15)
C(10)-C(9)-H(9)	108.2
C(17)-C(9)-H(9)	108.2
C(8)-C(9)-H(9)	108.2
O(5)-C(10)-O(4)	117.17(18)
O(5)-C(10)-C(9)	124.03(18)
O(4)-C(10)-C(9)	118.74(17)
C(16)-C(11)-C(12)	122.3(2)
C(16)-C(11)-O(4)	122.73(17)
C(12)-C(11)-O(4)	114.94(19)
C(13)-C(12)-C(11)	118.9(2)
C(13)-C(12)-H(12)	120.6
C(11)-C(12)-H(12)	120.6
C(14)-C(13)-C(12)	120.4(2)
C(14)-C(13)-H(13)	119.8
C(12)-C(13)-H(13)	119.8
C(13)-C(14)-C(15)	119.7(2)
C(13)-C(14)-H(14)	120.1
C(15)-C(14)-H(14)	120.1
C(14)-C(15)-C(16)	121.0(2)
C(14)-C(15)-H(15)	119.5
C(16)-C(15)-H(15)	119.5
C(11)-C(16)-C(15)	117.66(18)
C(11)-C(16)-C(17)	120.13(18)
C(15)-C(16)-C(17)	122.20(18)
C(16)-C(17)-C(9)	113.56(15)
C(16)-C(17)-C(18)	116.83(16)
C(9)-C(17)-C(18)	103.61(14)
C(16)-C(17)-H(17)	107.5
C(9)-C(17)-H(17)	107.5
C(18)-C(17)-H(17)	107.5
C(26)-C(18)-C(19)	113.70(15)
C(26)-C(18)-C(17)	110.40(15)
C(19)-C(18)-C(17)	100.76(14)

C(26)-C(18)-H(18)	110.5
C(19)-C(18)-H(18)	110.5
C(17)-C(18)-H(18)	110.5
C(20)-C(19)-C(18)	115.61(16)
C(20)-C(19)-C(8)	114.84(15)
C(18)-C(19)-C(8)	103.32(14)
C(20)-C(19)-H(19)	107.5
C(18)-C(19)-H(19)	107.5
C(8)-C(19)-H(19)	107.5
C(25)-C(20)-C(21)	118.9(2)
C(25)-C(20)-C(19)	119.0(2)
C(21)-C(20)-C(19)	122.14(18)
C(22)-C(21)-C(20)	120.0(2)
C(22)-C(21)-H(21)	120.0
C(20)-C(21)-H(21)	120.0
C(21)-C(22)-C(23)	120.5(3)
C(21)-C(22)-H(22)	119.7
C(23)-C(22)-H(22)	119.7
C(24)-C(23)-C(22)	119.7(2)
C(24)-C(23)-H(23)	120.1
C(22)-C(23)-H(23)	120.1
C(23)-C(24)-C(25)	120.6(2)
C(23)-C(24)-H(24)	119.7
C(25)-C(24)-H(24)	119.7
C(24)-C(25)-C(20)	120.3(2)
C(24)-C(25)-H(25)	119.9
C(20)-C(25)-H(25)	119.9
N(1)-C(26)-C(27)	123.46(18)
N(1)-C(26)-C(18)	115.61(18)
C(27)-C(26)-C(18)	120.88(17)
C(32)-C(27)-C(28)	117.23(19)
C(32)-C(27)-C(26)	119.51(18)
C(28)-C(27)-C(26)	123.26(18)
C(29)-C(28)-C(27)	120.7(2)
C(29)-C(28)-H(28)	119.7
C(27)-C(28)-H(28)	119.7
C(28)-C(29)-C(30)	119.7(2)
C(28)-C(29)-H(29)	120.2

C(30)-C(29)-H(29)	120.2
C(31)-C(30)-C(29)	121.3(2)
C(31)-C(30)-H(30)	119.4
C(29)-C(30)-H(30)	119.4
C(30)-C(31)-C(32)	118.4(2)
C(30)-C(31)-H(31)	120.8
C(32)-C(31)-H(31)	120.8
C(31)-C(32)-C(27)	122.7(2)
C(31)-C(32)-O(1)	117.38(19)
C(27)-C(32)-O(1)	119.84(19)
C(26)-N(1)-S(1)	119.18(15)
C(32)-O(1)-S(1)	116.12(13)
C(10)-O(4)-C(11)	122.36(16)
O(3)-S(1)-O(2)	119.90(12)
O(3)-S(1)-O(1)	105.02(11)
O(2)-S(1)-O(1)	108.18(10)
O(3)-S(1)-N(1)	109.64(10)
O(2)-S(1)-N(1)	108.61(10)
O(1)-S(1)-N(1)	104.36(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3aj**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	45(1)	32(1)	42(1)	16(1)	5(1)	1(1)
C(2)	43(1)	42(1)	60(2)	17(1)	8(1)	-1(1)
C(3)	58(2)	36(1)	60(2)	17(1)	28(1)	3(1)
C(4)	72(2)	49(1)	60(2)	35(1)	29(1)	13(1)
C(5)	55(2)	48(1)	58(1)	35(1)	23(1)	16(1)
C(6)	43(1)	28(1)	34(1)	13(1)	15(1)	8(1)
C(7)	37(1)	32(1)	32(1)	16(1)	9(1)	10(1)
C(8)	32(1)	28(1)	26(1)	13(1)	7(1)	8(1)
C(9)	33(1)	28(1)	26(1)	13(1)	6(1)	9(1)
C(10)	35(1)	32(1)	24(1)	11(1)	5(1)	6(1)
C(11)	37(1)	33(1)	27(1)	14(1)	9(1)	8(1)
C(12)	48(1)	54(1)	31(1)	26(1)	8(1)	10(1)

C(13)	56(1)	51(1)	38(1)	29(1)	18(1)	14(1)
C(14)	42(1)	38(1)	37(1)	19(1)	16(1)	9(1)
C(15)	36(1)	35(1)	29(1)	13(1)	10(1)	11(1)
C(16)	32(1)	31(1)	26(1)	14(1)	10(1)	11(1)
C(17)	28(1)	31(1)	25(1)	14(1)	6(1)	9(1)
C(18)	28(1)	28(1)	24(1)	12(1)	6(1)	7(1)
C(19)	29(1)	31(1)	25(1)	14(1)	4(1)	7(1)
C(20)	31(1)	34(1)	25(1)	13(1)	4(1)	-1(1)
C(21)	35(1)	43(1)	31(1)	10(1)	8(1)	4(1)
C(22)	46(1)	48(1)	42(1)	5(1)	16(1)	3(1)
C(23)	62(2)	55(2)	29(1)	6(1)	17(1)	-12(1)
C(24)	63(2)	58(2)	29(1)	20(1)	9(1)	-7(1)
C(25)	46(1)	45(1)	31(1)	21(1)	5(1)	2(1)
C(26)	29(1)	34(1)	24(1)	14(1)	6(1)	7(1)
C(27)	31(1)	32(1)	28(1)	14(1)	8(1)	8(1)
C(28)	33(1)	34(1)	38(1)	18(1)	4(1)	8(1)
C(29)	42(1)	43(1)	57(1)	31(1)	13(1)	16(1)
C(30)	50(1)	32(1)	59(1)	22(1)	17(1)	10(1)
C(31)	41(1)	34(1)	43(1)	15(1)	9(1)	1(1)
C(32)	33(1)	37(1)	31(1)	15(1)	7(1)	6(1)
N(1)	31(1)	37(1)	37(1)	21(1)	2(1)	7(1)
O(1)	35(1)	43(1)	36(1)	17(1)	-4(1)	3(1)
O(2)	38(1)	55(1)	68(1)	30(1)	20(1)	13(1)
O(3)	44(1)	68(1)	66(1)	41(1)	-14(1)	7(1)
O(4)	36(1)	48(1)	30(1)	22(1)	1(1)	4(1)
O(5)	33(1)	54(1)	37(1)	24(1)	-1(1)	0(1)
O(6)	47(1)	47(1)	65(1)	37(1)	24(1)	23(1)
S(1)	28(1)	44(1)	47(1)	25(1)	0(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3aj**.

	x	y	z	U(eq)
H(1)	-1245	1877	5597	51
H(2)	-3132	490	5745	64
H(3)	-2868	-657	6861	65
H(4)	-719	-448	7811	67

H(5)	1166	860	7612	59
H(8)	478	3834	6385	35
H(9)	2147	2886	4985	35
H(12)	1738	5499	2673	52
H(13)	3805	7060	2988	54
H(14)	5566	7744	4604	46
H(15)	5285	6820	5880	40
H(17)	3915	4715	6044	34
H(18)	2208	6418	6929	33
H(19)	3232	4595	7665	34
H(21)	771	6499	8094	48
H(22)	114	7348	9795	63
H(23)	926	6866	11286	69
H(24)	2343	5482	11059	66
H(25)	3024	4626	9373	51
H(28)	2818	8235	6763	43
H(29)	3284	10544	7096	53
H(30)	5146	12241	8564	56
H(31)	6614	11646	9653	51

Table 6. Torsion angles [°] for **3aj**.