Chiral Bicyclic Guanidine−Catalyzed Enantioselective Sulfenylation of Oxindoles and Benzofuran-2(3*H*)-ones

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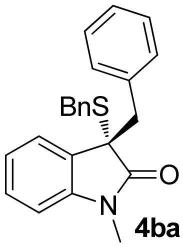
1. Copies of HPLC spectra



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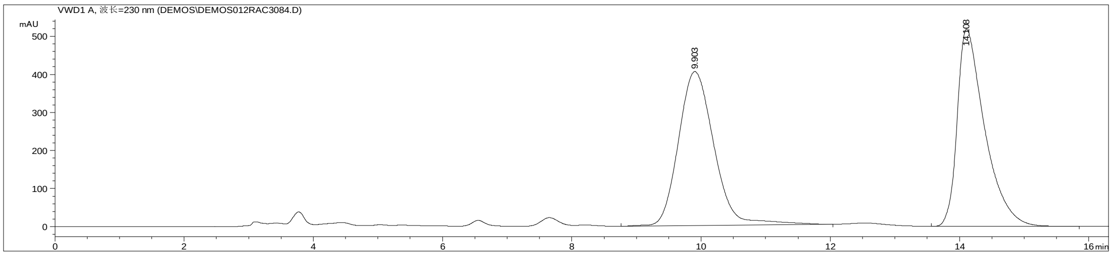

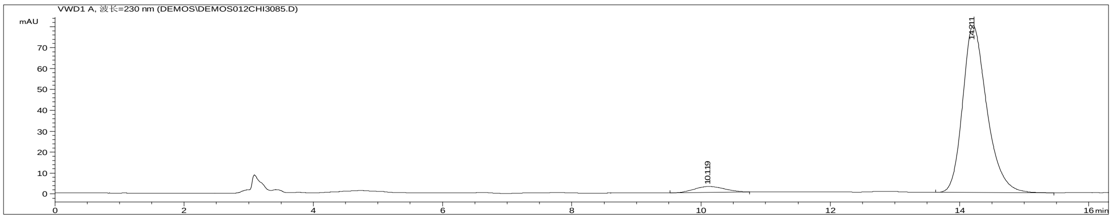

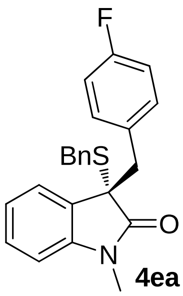

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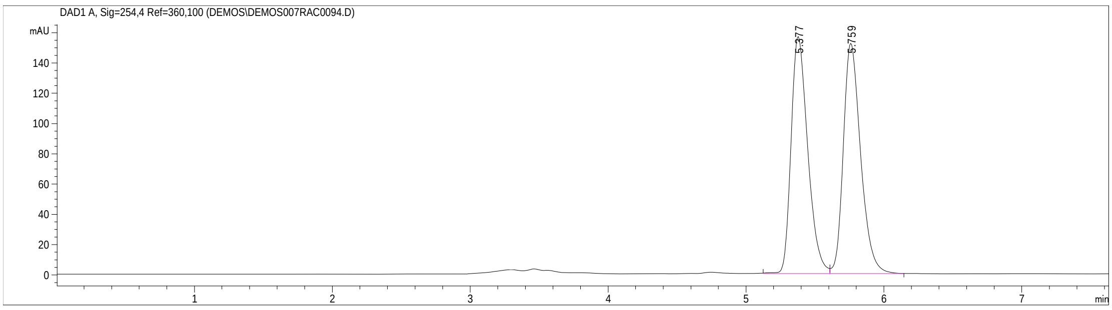

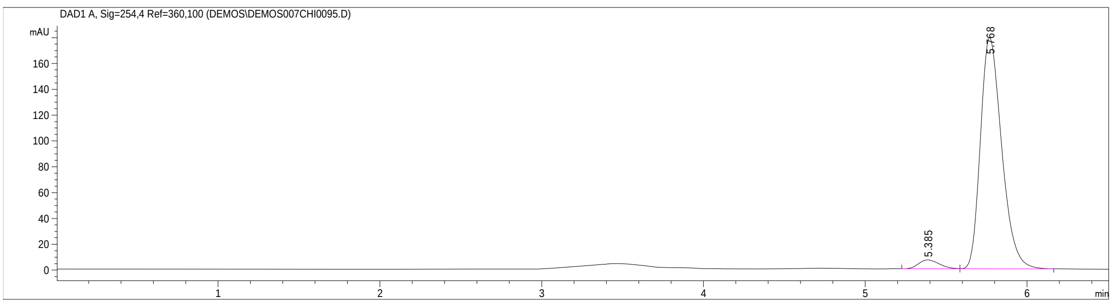

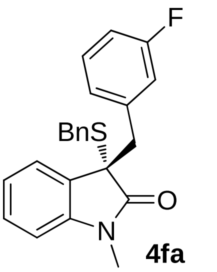
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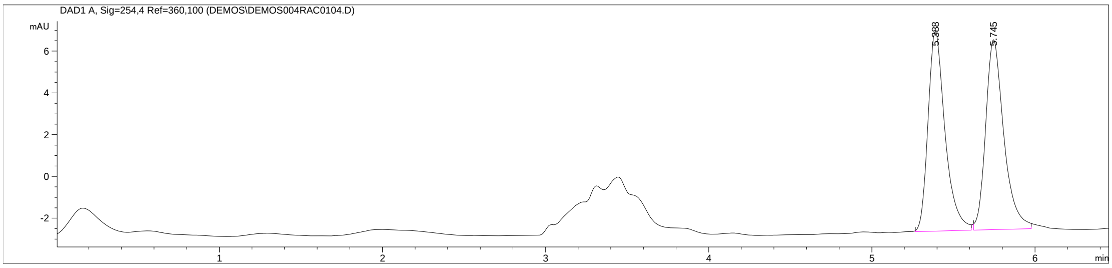
O
N

4fa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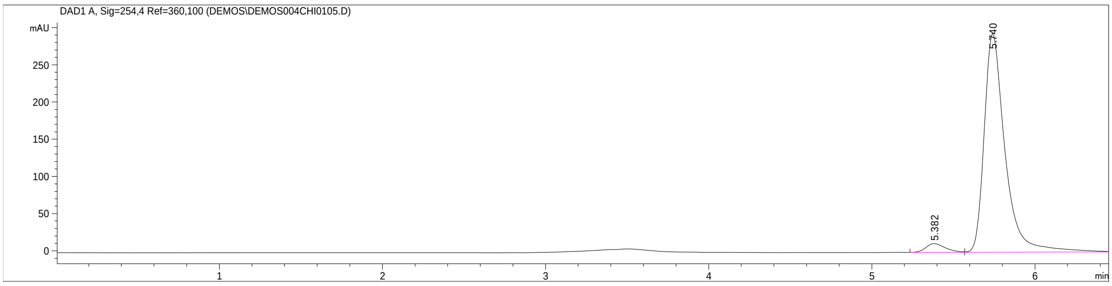
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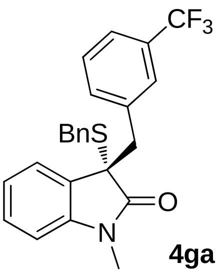
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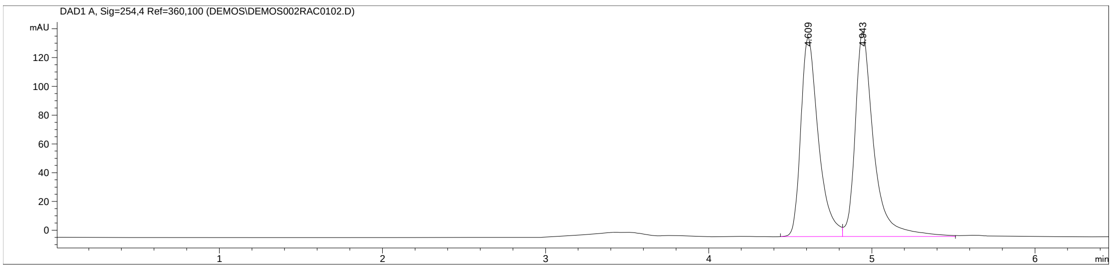
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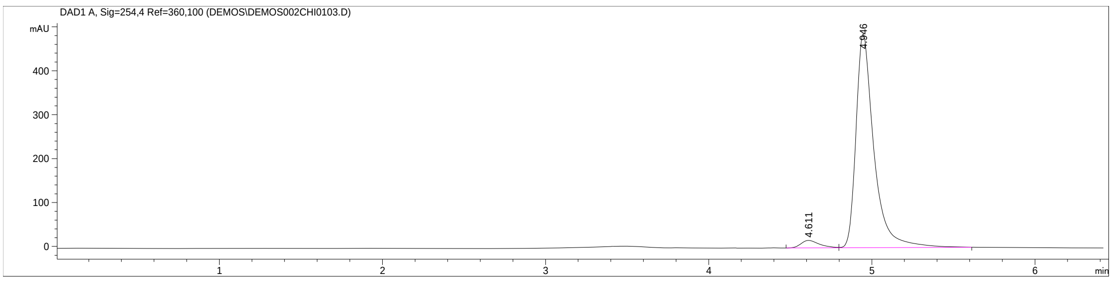
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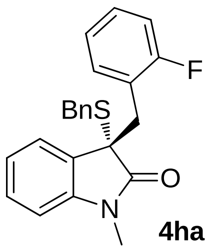

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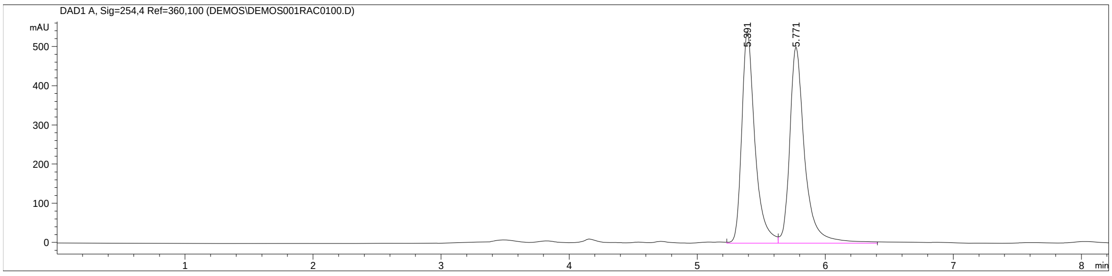
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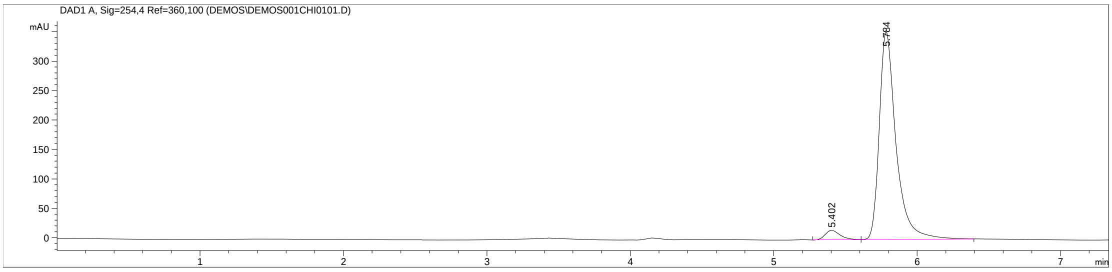
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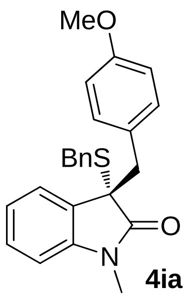

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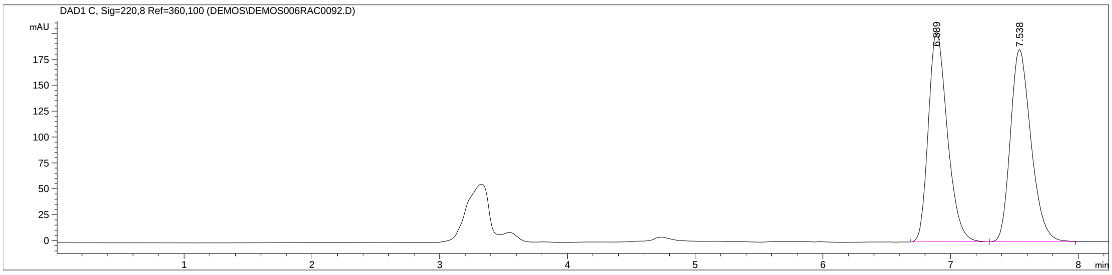
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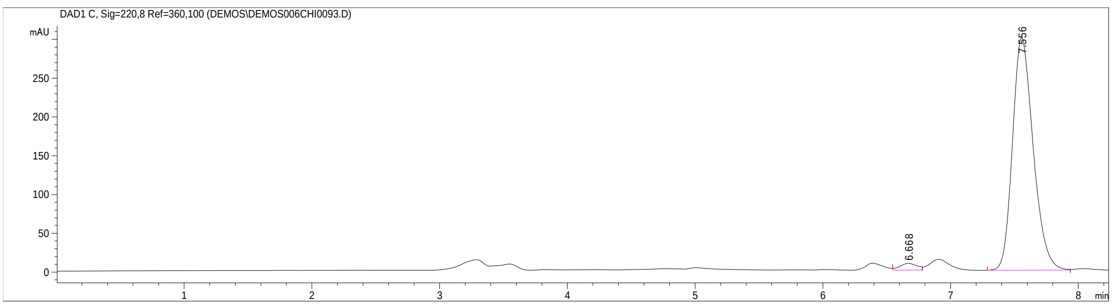
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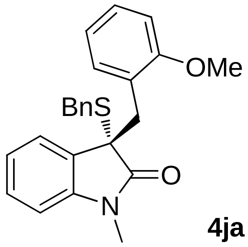
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BnS
O
N

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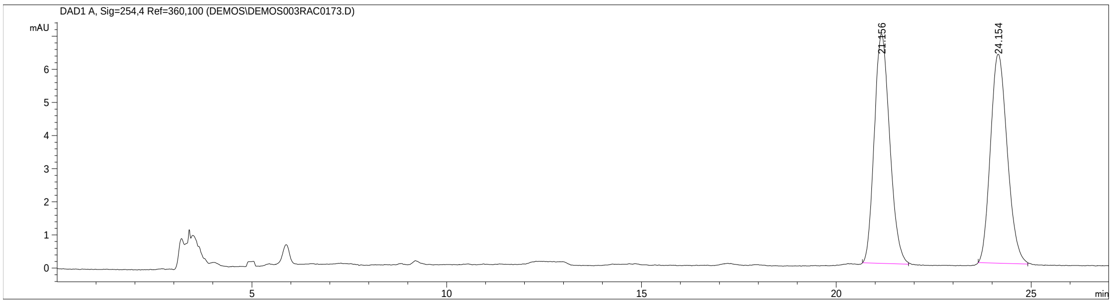
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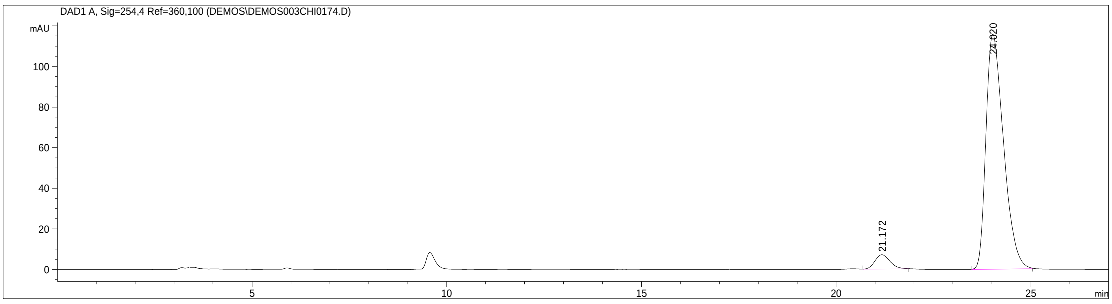
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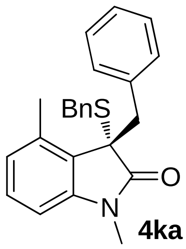
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N

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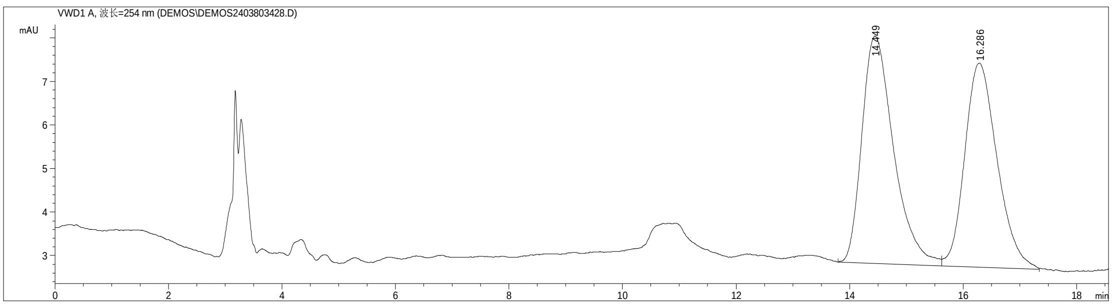
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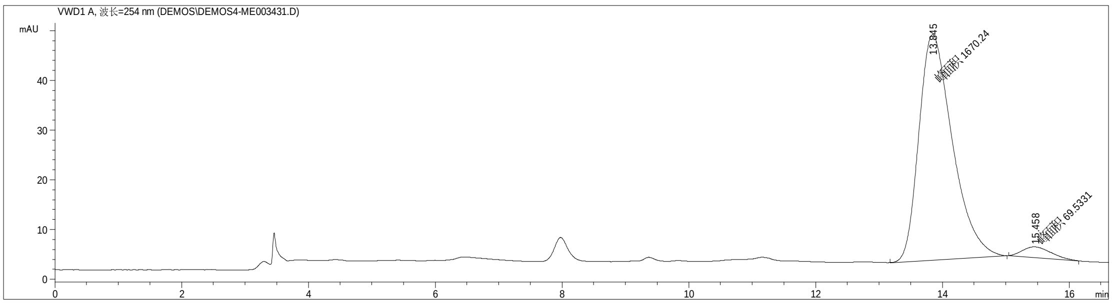
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峰面积: 69.5331
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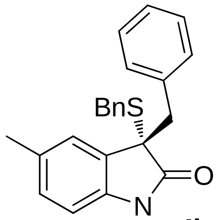
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O
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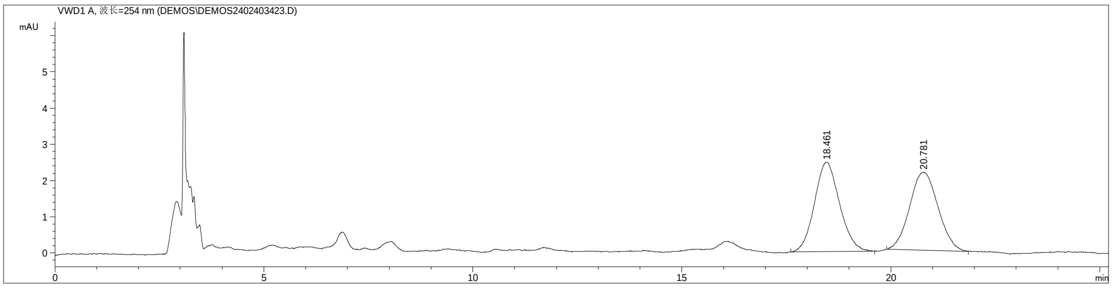
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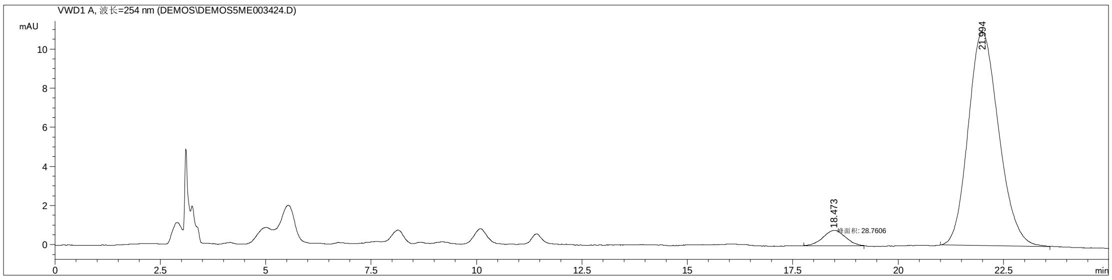
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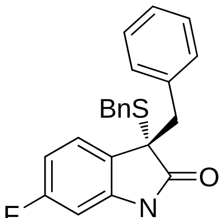
BnS
O
F
N

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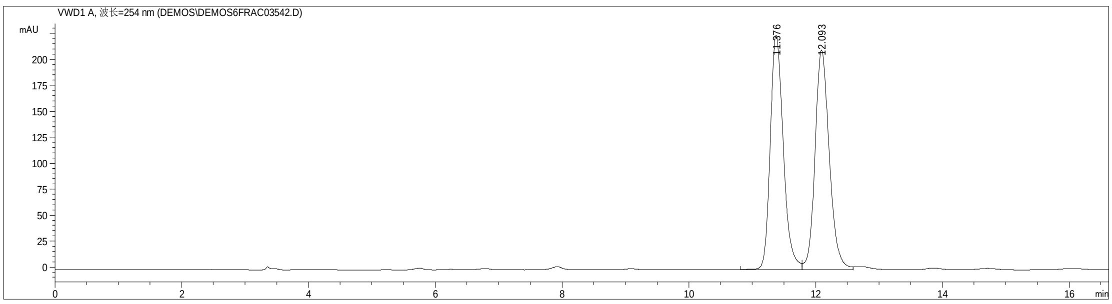
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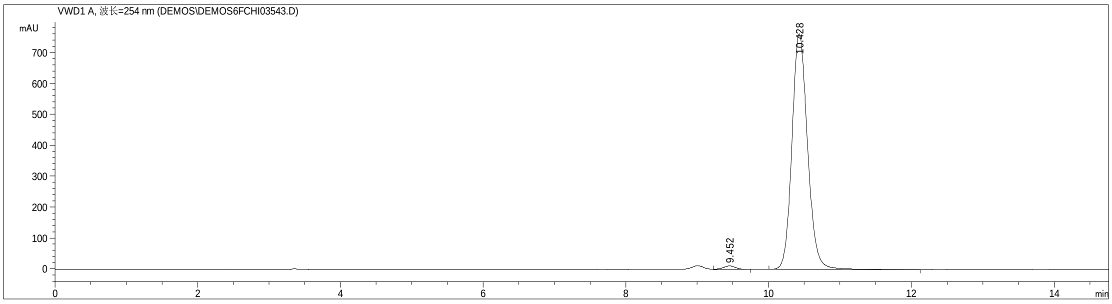
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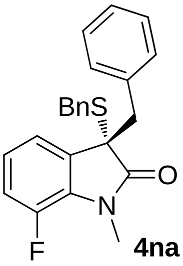
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O
N
F

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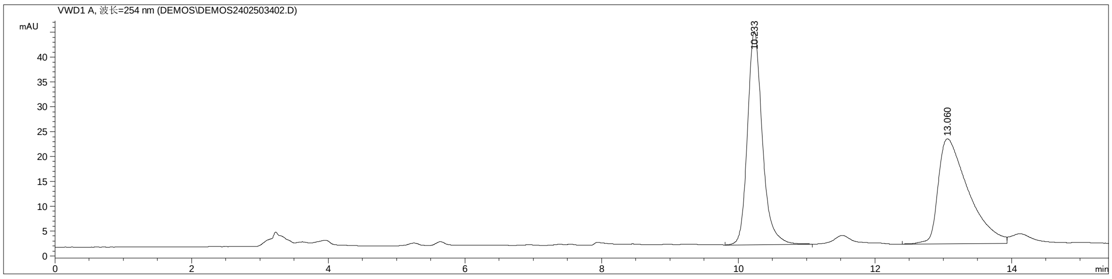
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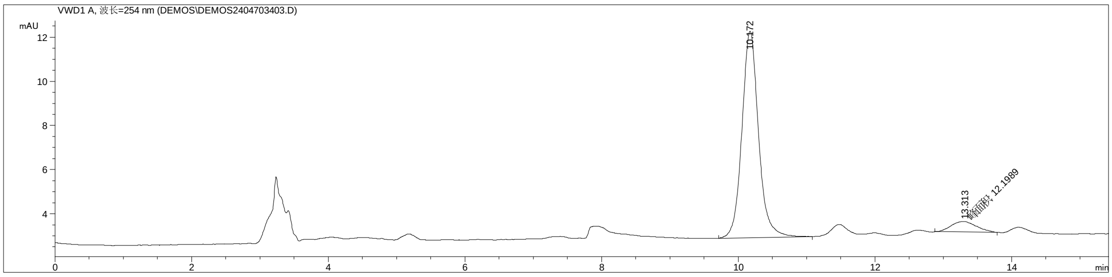
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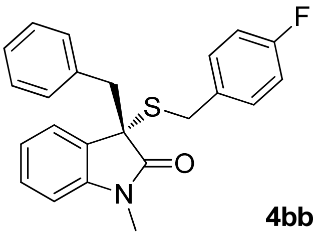
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O
N

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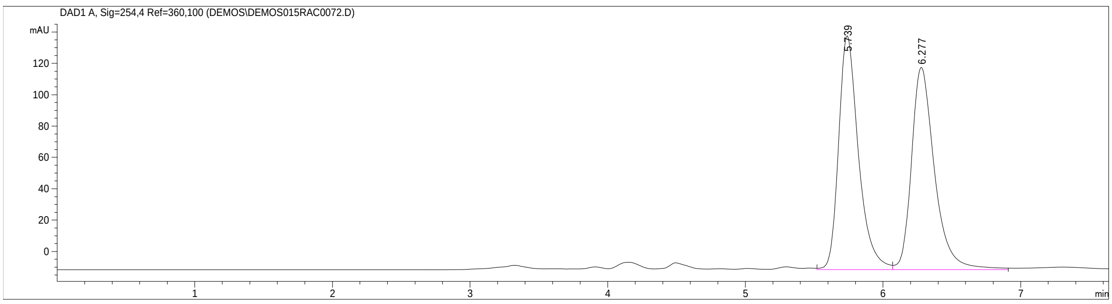
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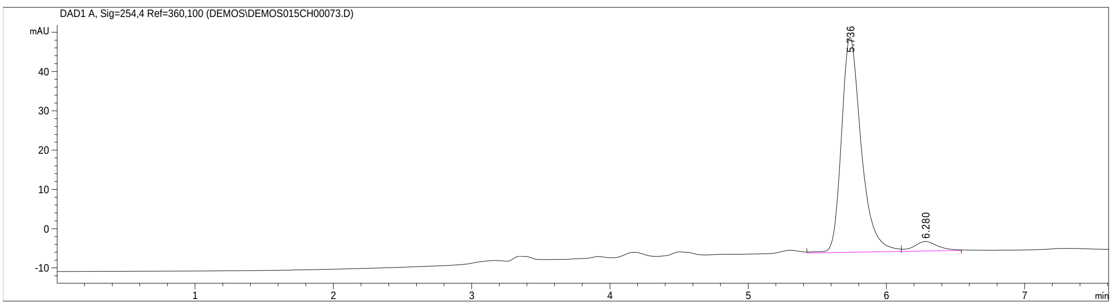
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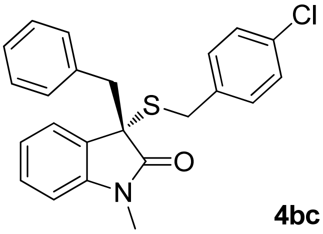
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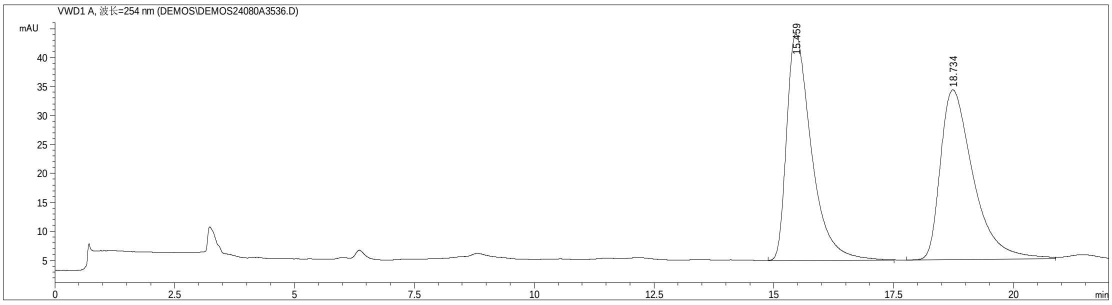
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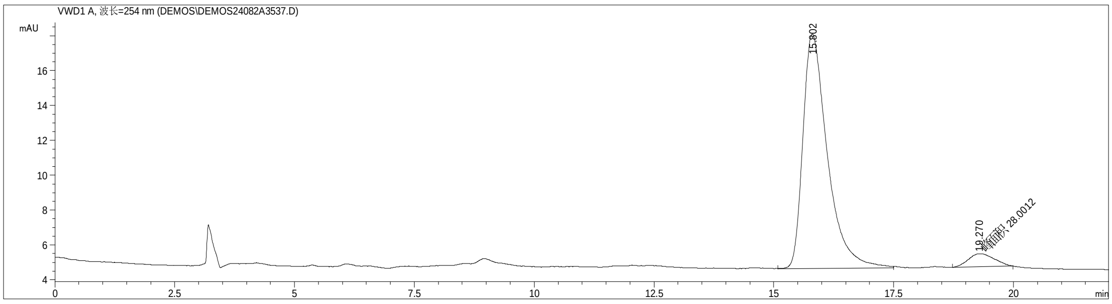
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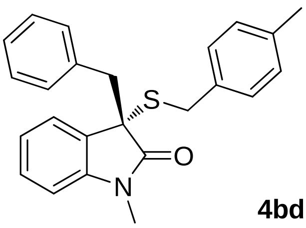
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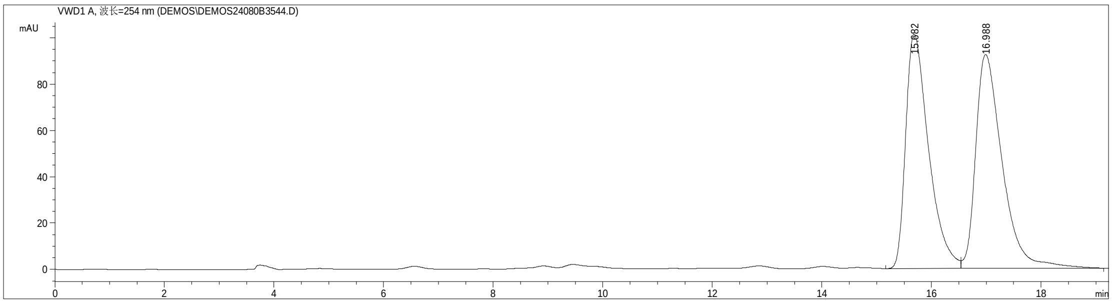
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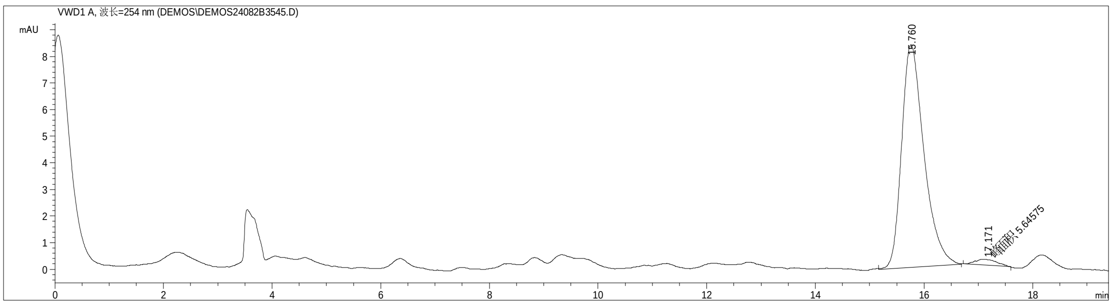
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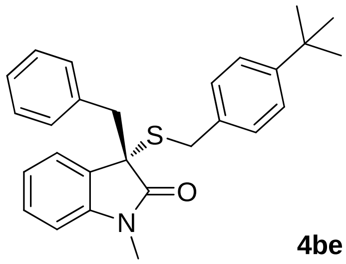

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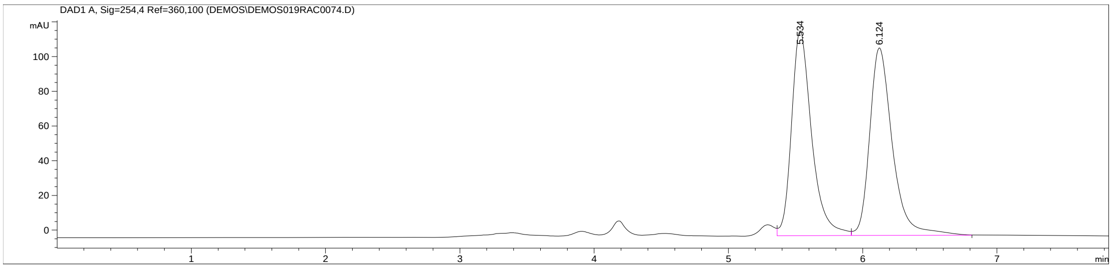
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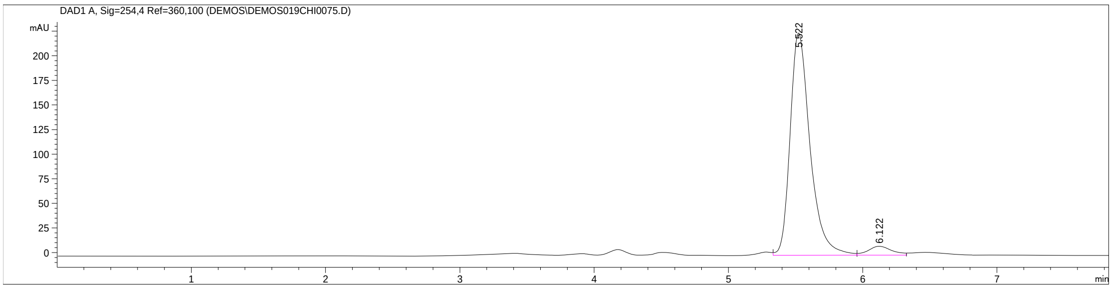
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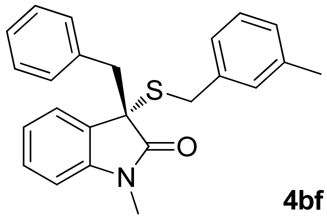

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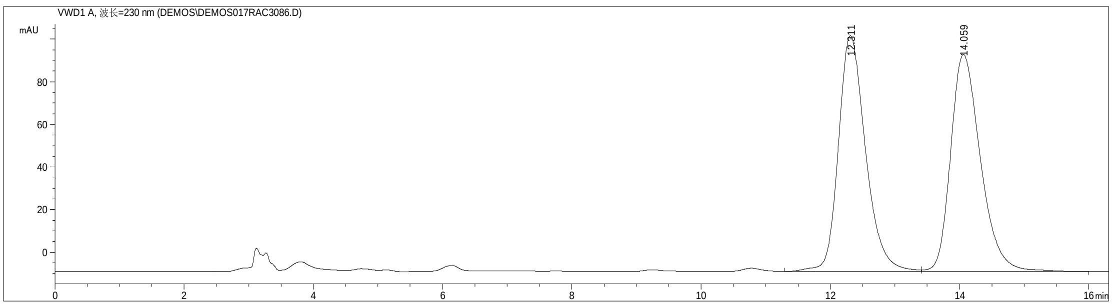
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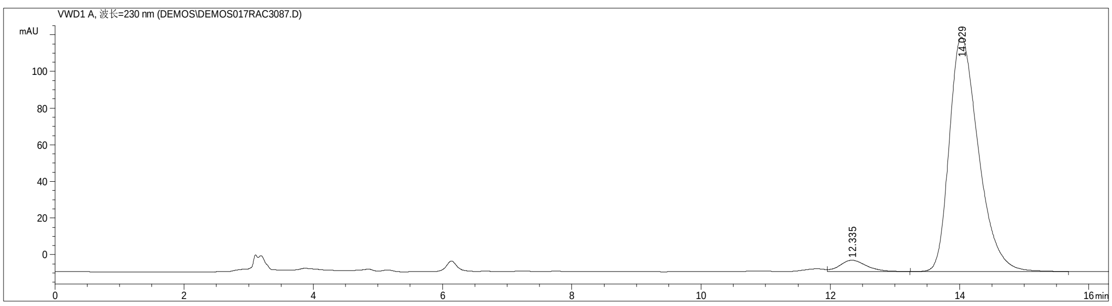
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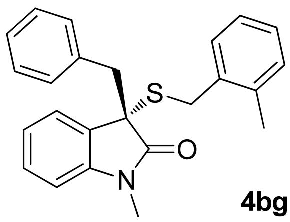

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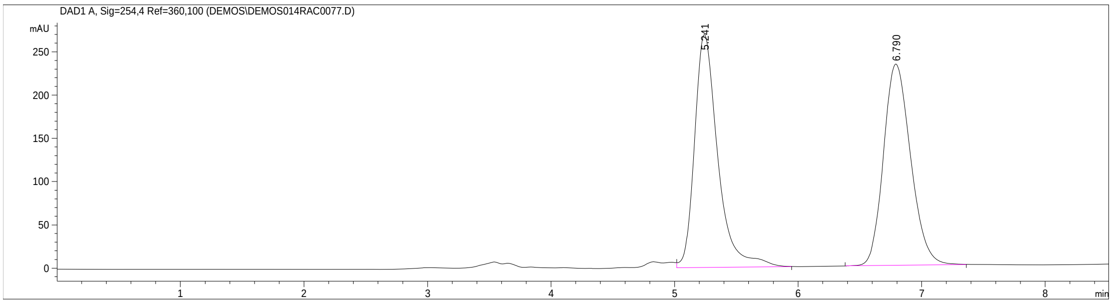
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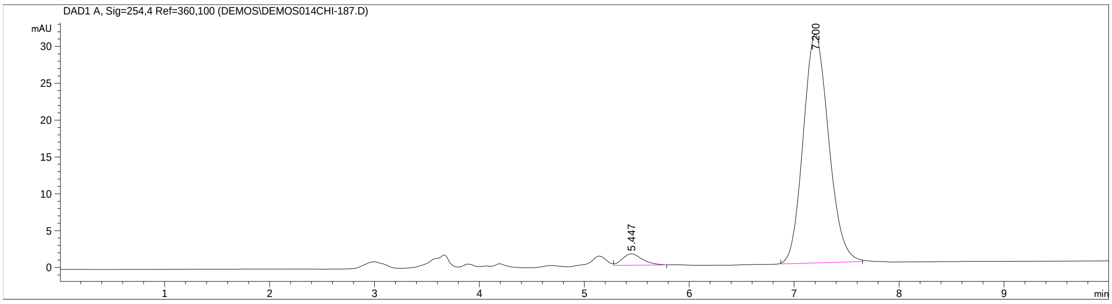
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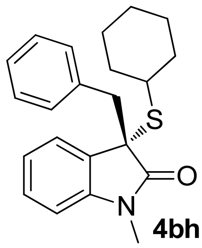

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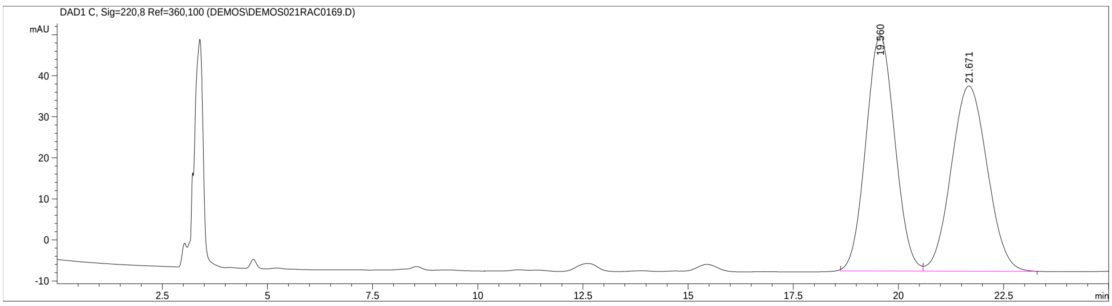
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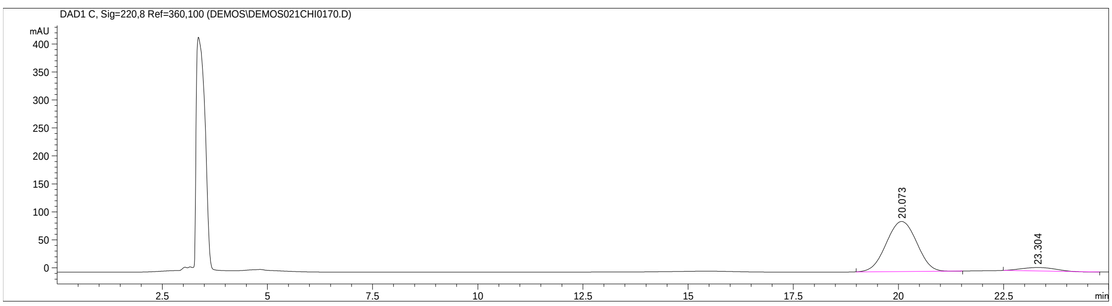
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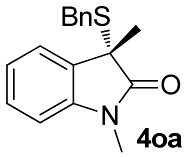

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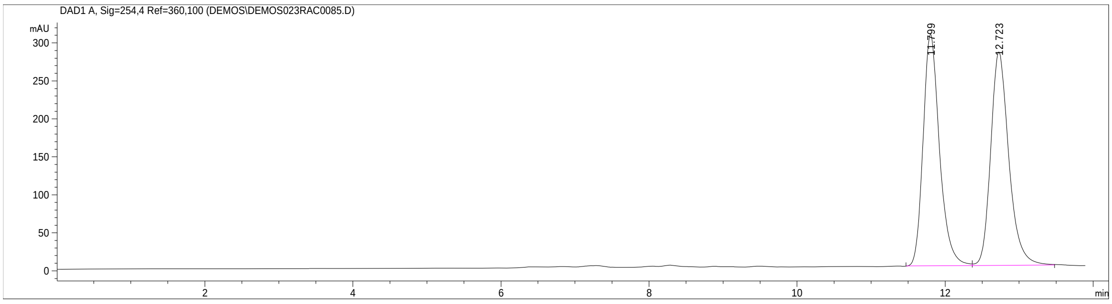
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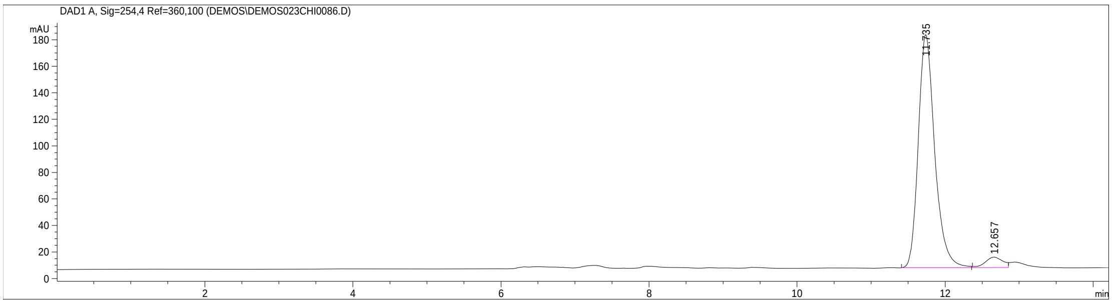
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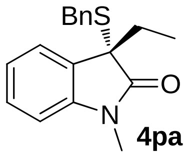
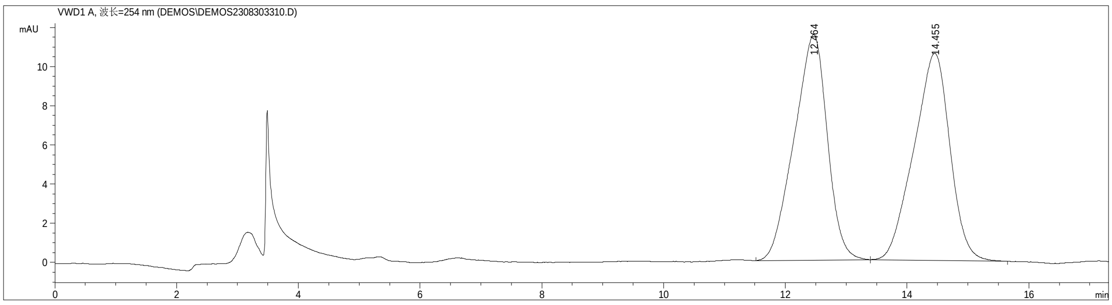
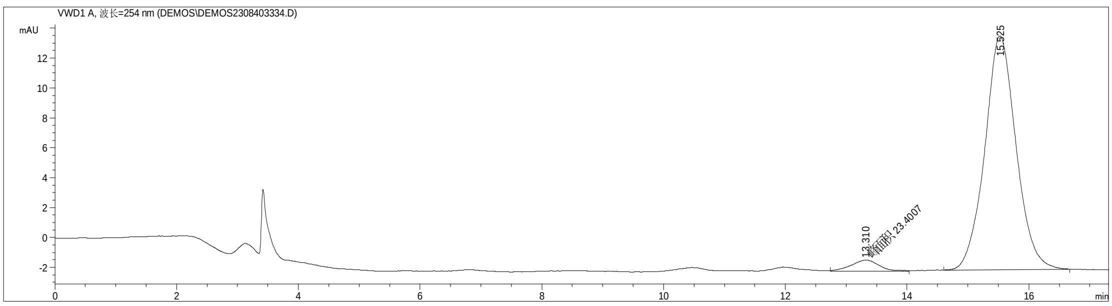
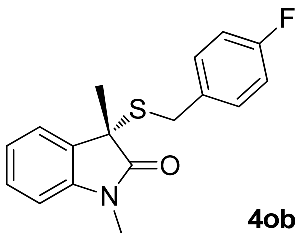
12.657
min



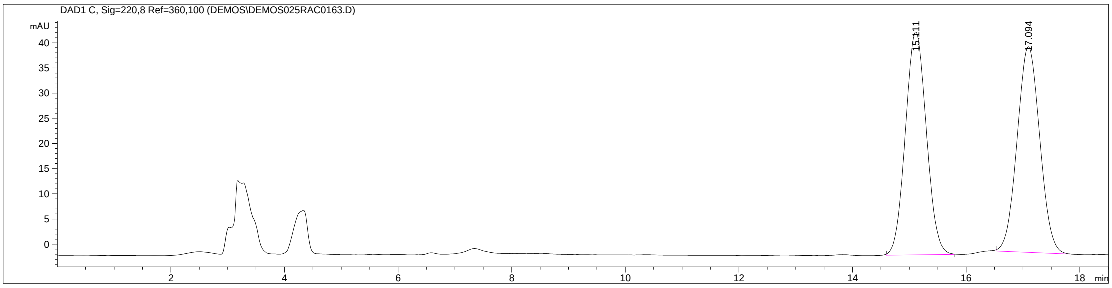
4pa


VWD1 A, 波长=254 nm (DEMOS\DEMOS2308303310.D)
mAU
12.464
14.455
min

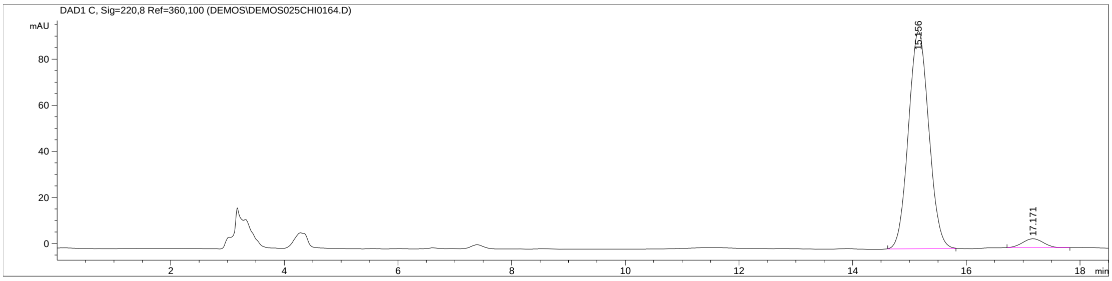

VWD1 A, 波长=254 nm (DEMOS\DEMOS2308403334.D)
mAU
13.310
15.525
min



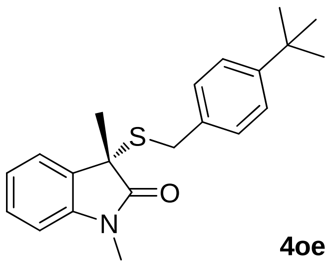
4ob



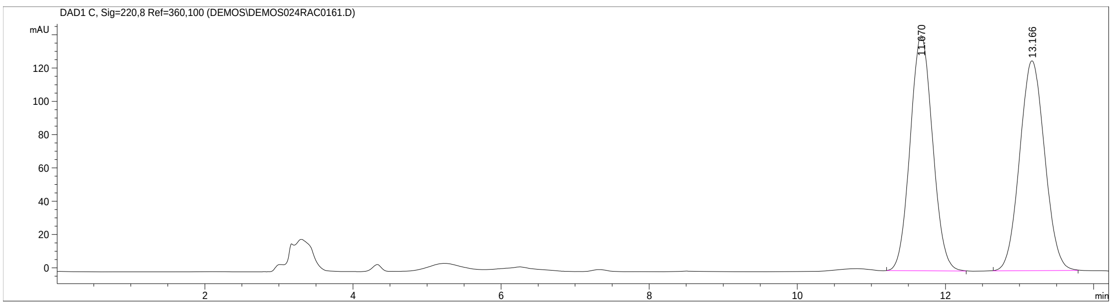
DAD1 C, Sig=220,8 Ref=360,100 (DEMOS\DEMOS025RAC0163.D)
mAU
15.111
17.094
min



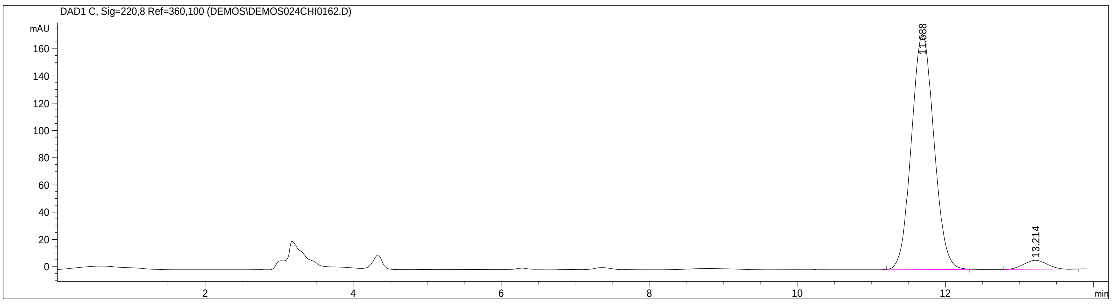
DAD1 C, Sig=220,8 Ref=360,100 (DEMOS\DEMOS025CHI0164.D)
mAU
15.156
17.171
min



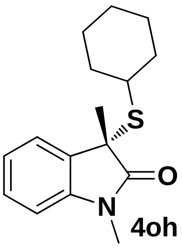
4oe



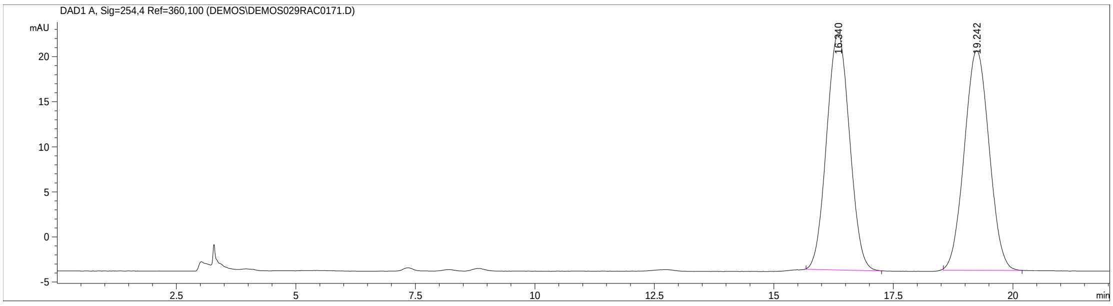
DAD1 C, Sig=220,8 Ref=360,100 (DEMOS\DEMOS024RAC0161.D)
mAU
11.670
13.166
min

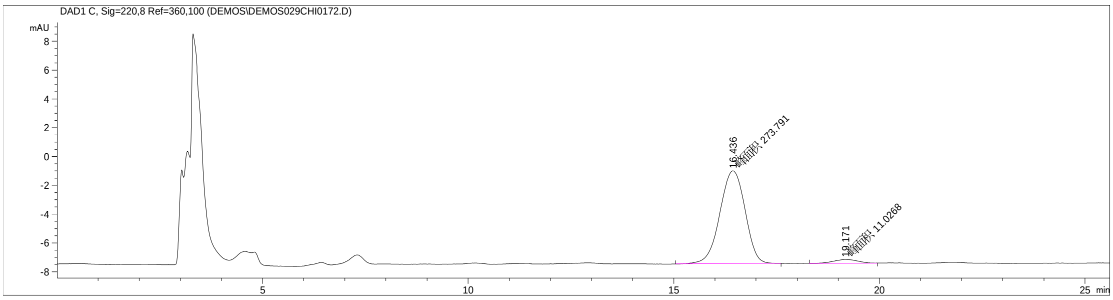


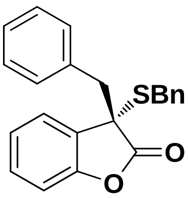
DAD1 C, Sig=220,8 Ref=360,100 (DEMOS\DEMOS024CHI0162.D)
mAU
11.688
13.214
min



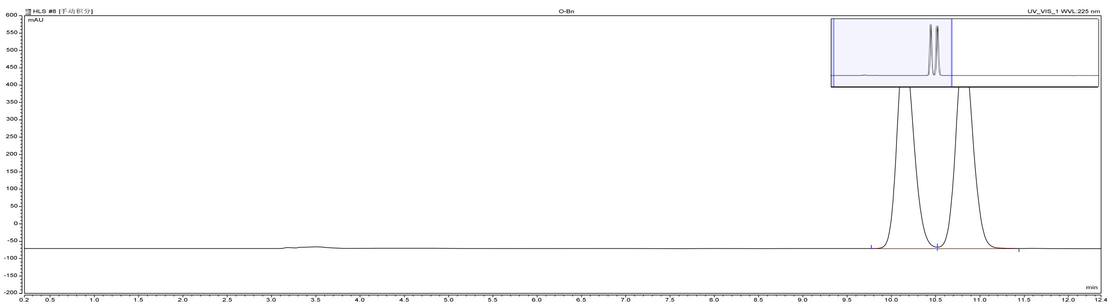
4oh

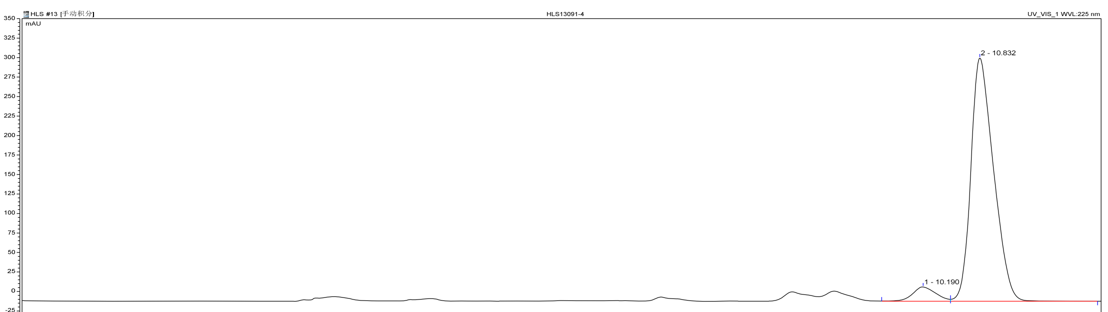

DAD1 A, Sig=254,4 Ref=360,100 (DEMOS\DEMOS029RAC0171.D)
mAU
16.340
19.242
min

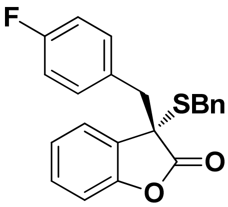

DAD1 C, Sig=220,8 Ref=360,100 (DEMOS\DEMOS029CHI0172.D)
mAU
16.436
273.791
19.171
11.0268
min



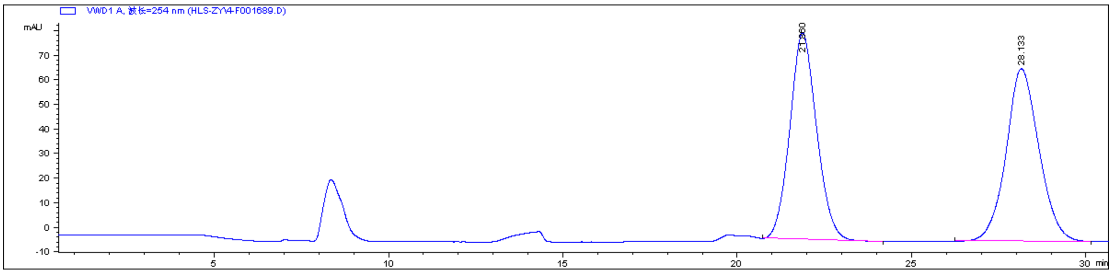
5aa

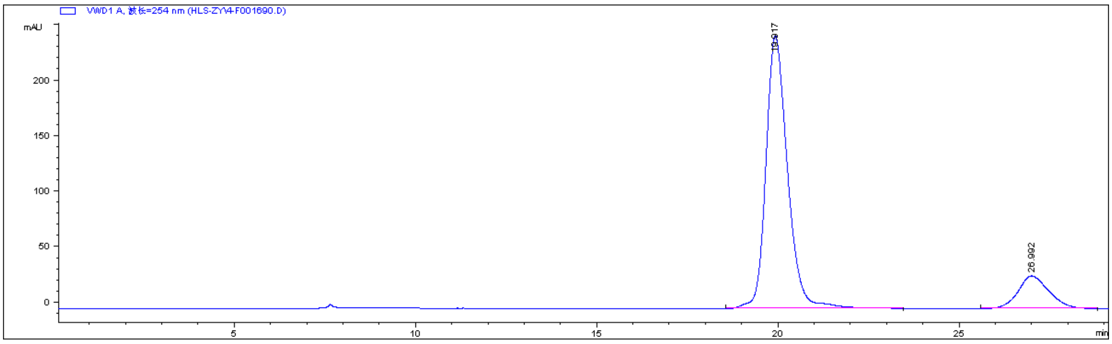

HLS #8 [手动积分]
O-Bn
UV_VIS_1 WVL:225 nm
mAU
min

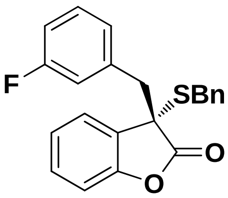

HLS #13 [手动积分]
HLS13091-4
UV_VIS_1 WVL:225 nm
mAU
1 - 10.190
2 - 10.832
min


F
SBn
O
O

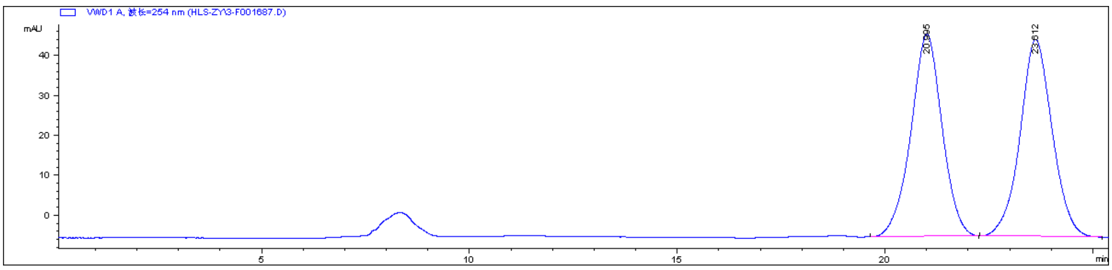
5ba

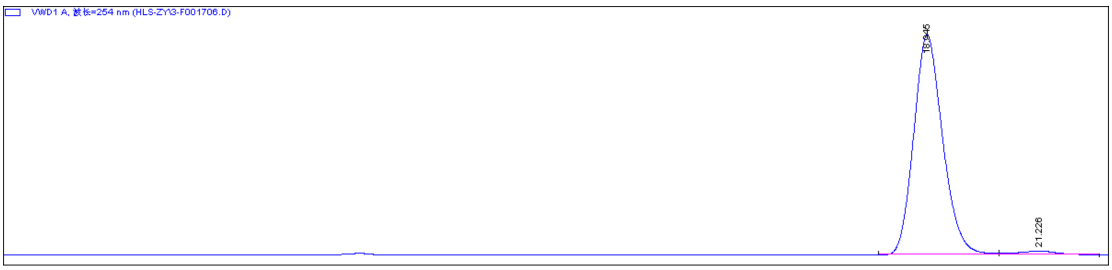

VWD1 A, 波长=254 nm (HLS-ZY\4-F001689.D)
mAU
21.960
28.133
min

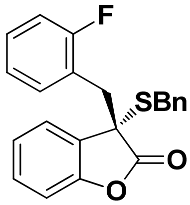

VWD1 A, 波长=254 nm (HLS-ZY\4-F001690.D)
mAU
19.917
26.992
min


F
SBn
O
O

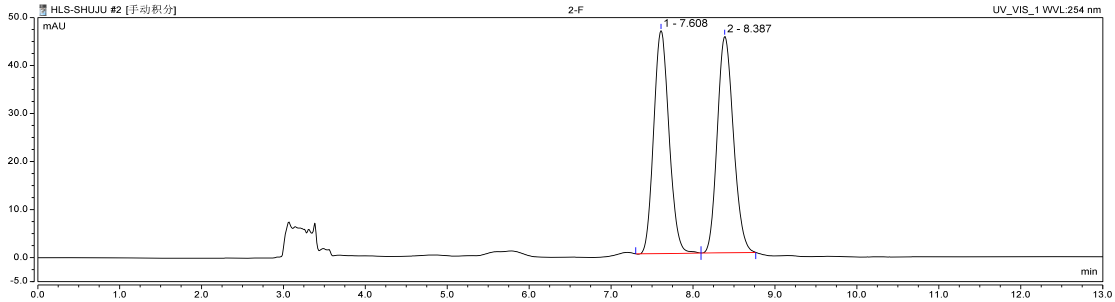
5ca

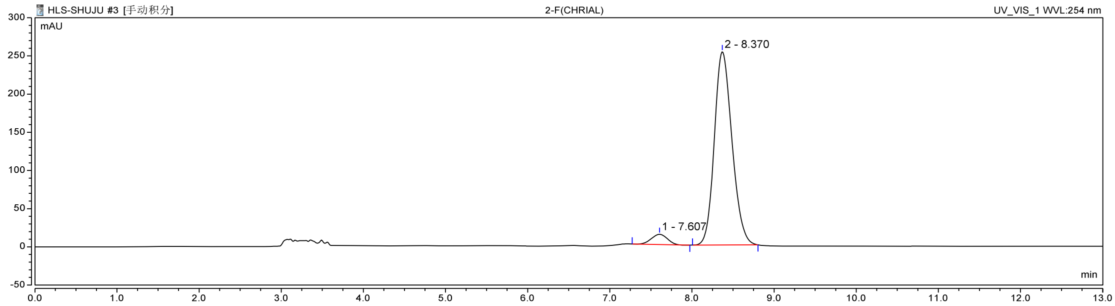

VWD1 A, 波长=254 nm (HLS-ZY\3-F001687.D)
mAU
20.905
23.612
min

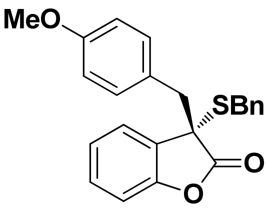

VWD1 A, 波长=254 nm (HLS-ZY\3-F001706.D)
18.945
21.226


F
SBn
O
O

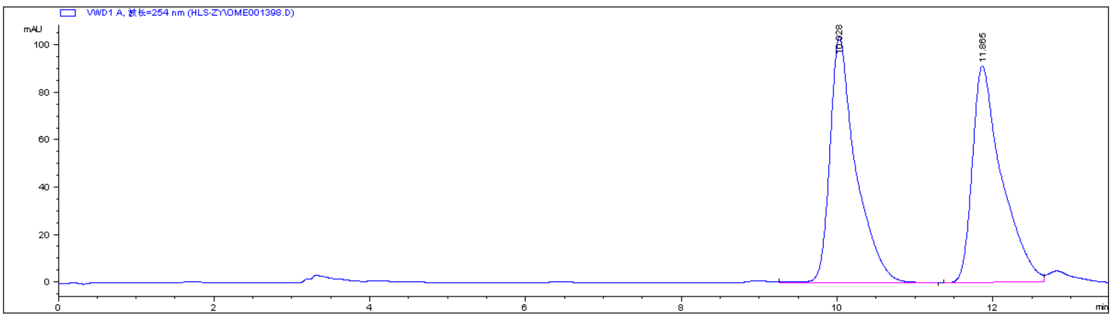
5da

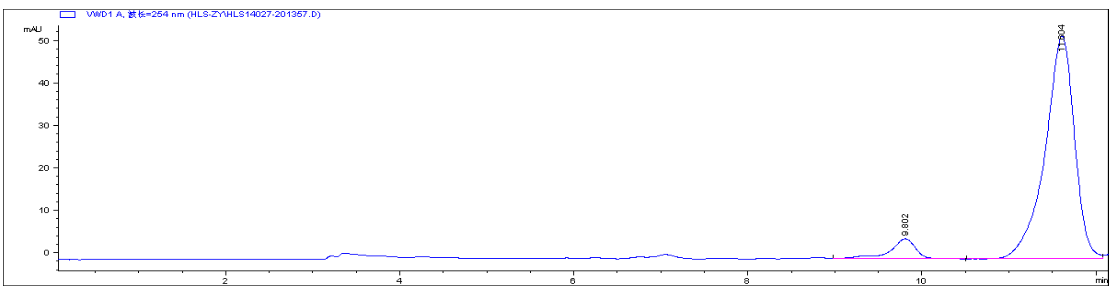

HLS-SHUJU #2 [手动积分]
2-F
UV_VIS_1 WVL:254 nm
mAU
1 - 7.608
2 - 8.387
min

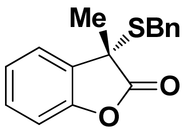

HLS-SHUJU #3 [手动积分]
2-F(CHRIAL)
UV_VIS_1 WVL:254 nm
mAU
2 - 8.370
1 - 7.607
min


MeO
SBn
O
O

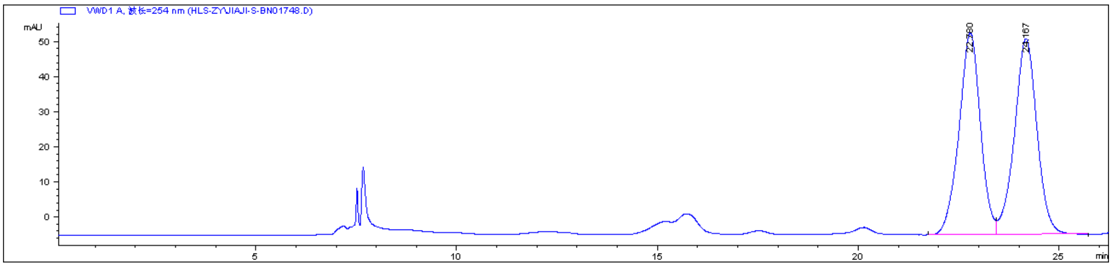
5ea

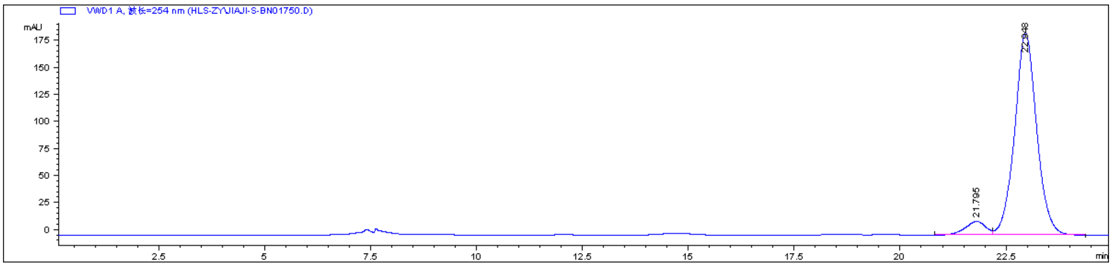

VWD1 A, 波长=254 nm (HLS-ZY\DME001398.D)
mAU
10.028
11.865
min

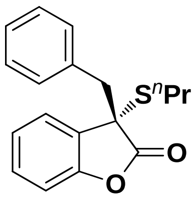

VWD1 A, 波长=254 nm (HLS-ZY\HLS14027-201357.D)
mAU
9.802
11.504
min


Me
SBn
O
O

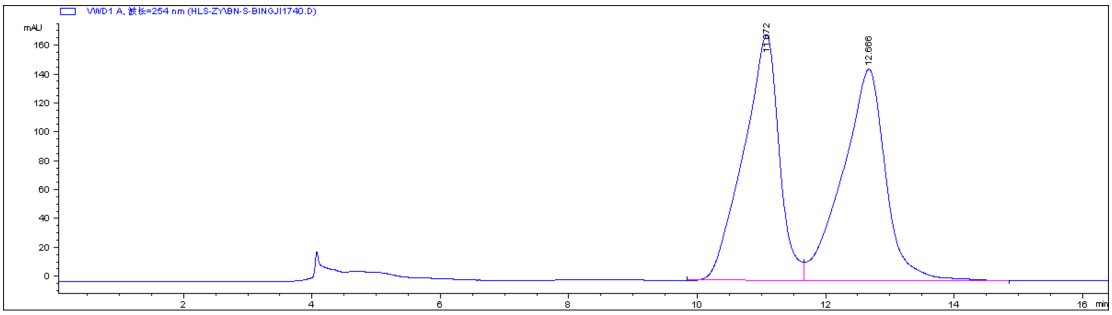
5fa

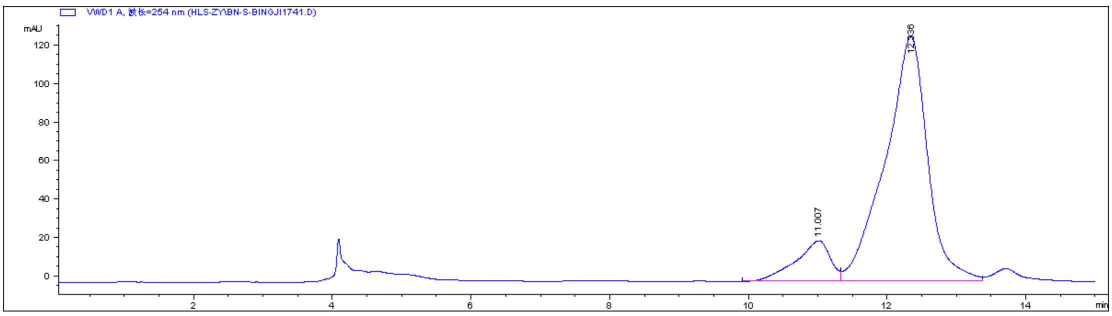

VWD1 A, 波长=254 nm (HLS-ZY\JIAJI-S-BN01748.D)
mAU
min

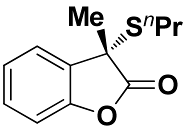

VWD1 A, 波长=254 nm (HLS-ZY\JIAJI-S-BN01750.D)
mAU
21.795
min


S^{n}Pr
O
O

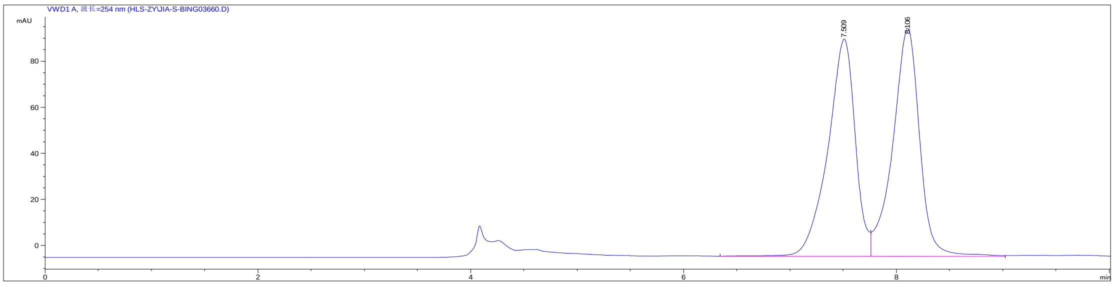
5ai

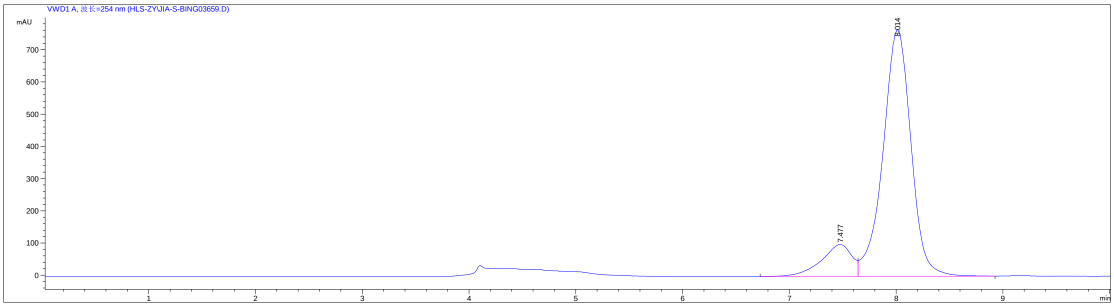

VWD1 A, 波长=254 nm (HLS-ZY\BN-S-BINGJI1740.D)
mAU
12.666
min

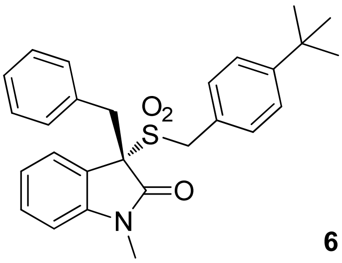

VWD1 A, 波长=254 nm (HLS-ZY\BN-S-BINGJI1741.D)
mAU
11.007
min


Me
S^{n}Pr
O
O

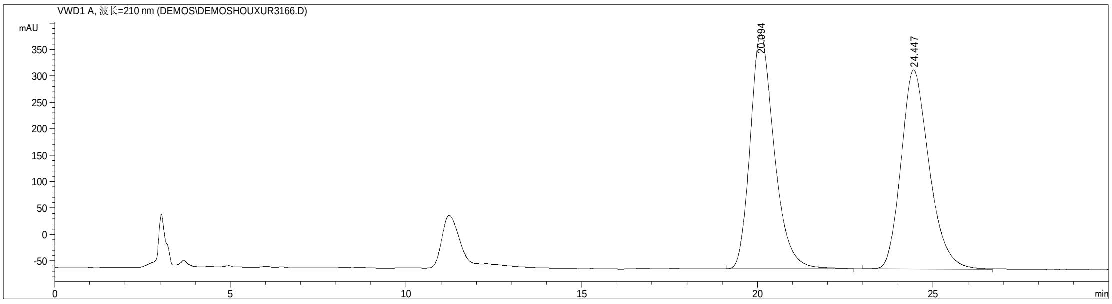
5fi

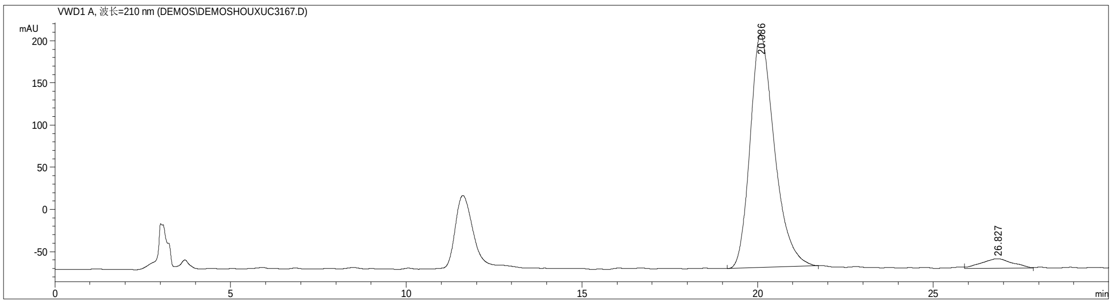

VWD1 A, 波长=254 nm (HLS-ZY\JIA-S-BING03660.D)
mAU
7.509
8.106
min


VWD1 A, 波长=254 nm (HLS-ZY\JIA-S-BING03659.D)
mAU
7.477
8.014
min

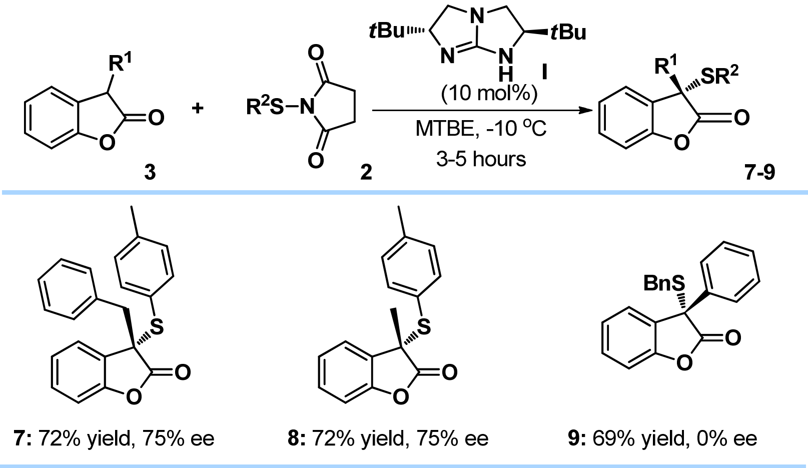

O2
S
O
N

6


VWD1 A, 波长=210 nm (DEMOS\DEMOSHOUXUR3166.D)
mAU
20.094
24.447
min

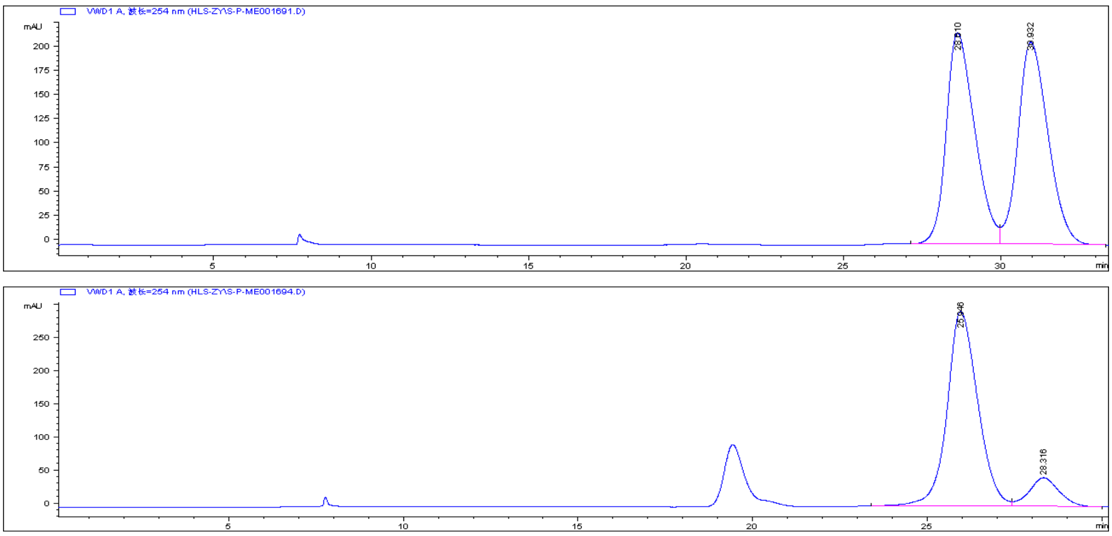

VWD1 A, 波长=210 nm (DEMOS\DEMOSHOUXUC3167.D)
mAU
20.086
26.827
min

2. Other sulfenylation reactions between benzofuran-2(3*H*)-ones 3 and *N*-(sulfanyl)succinimides 2[a]

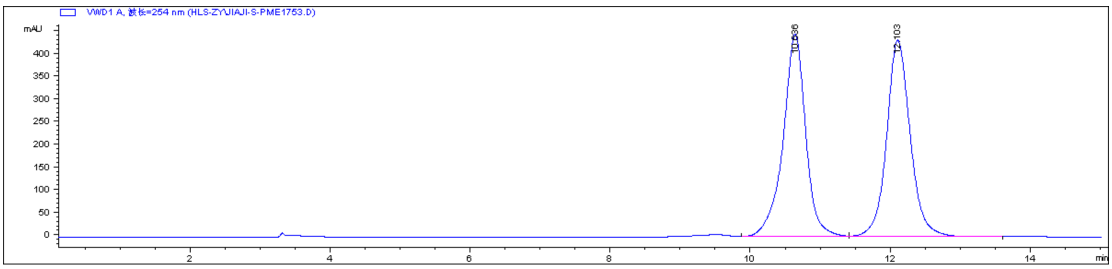


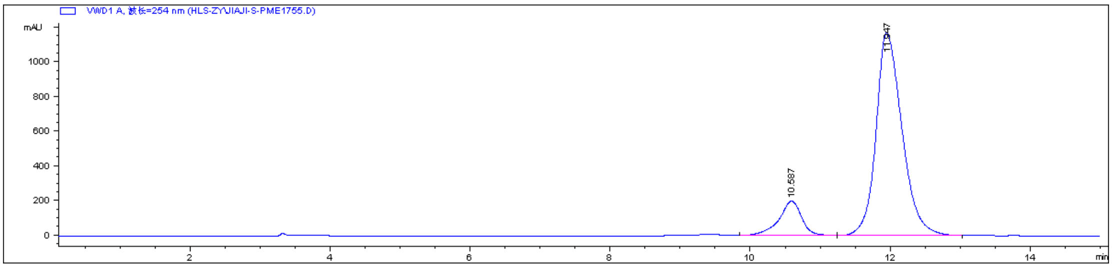
[a] The reaction was carried out with 0.1 mmol of **3**, 0.15 mmol of **2** and 0.01 mmol of catalyst in 3.0 mL MTBE at −10 °C (MTBE = methyl *tert*-butyl methyl ether). Yield of isolated product. Ees were determined by HPLC analysis on a chiral stationary phase.

7, (*S*)-(+)-3-benzyl-3-(*p*-tolylthio)benzofuran-2(3*H*)-one

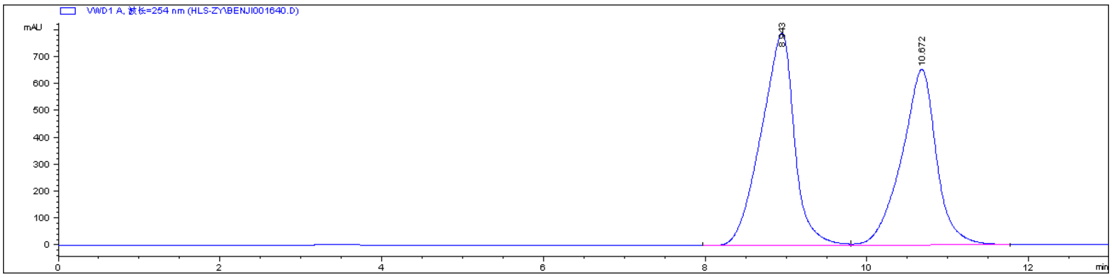


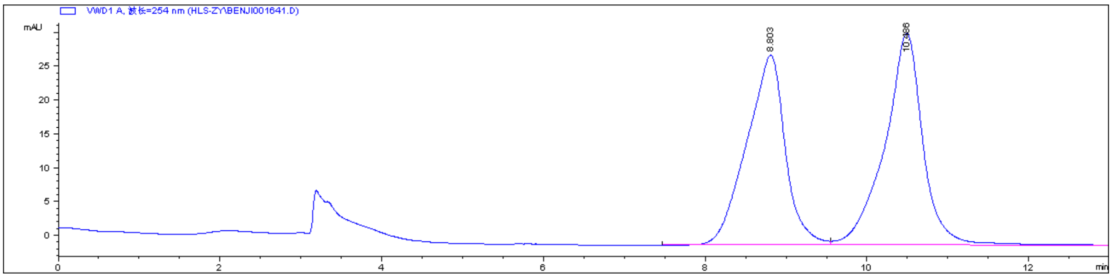
8, (*S*)-(+)-3-methyl-3-(*p*-tolylthio)benzofuran-2(3*H*)-one





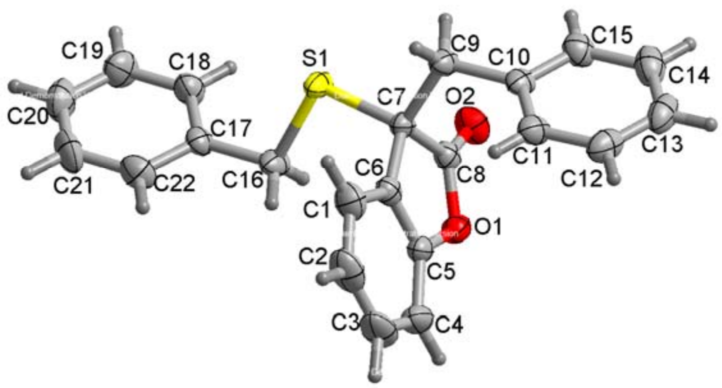
9, (*S*)-3-(benzylthio)-3-phenylbenzofuran-2(3*H*)-one





3. Determination of the absolute configuration by *X*-ray crystallography

The absolute configurations of sulfenylated products **4** and **5** were assigned based on X-ray crystallographic analysis of a single crystal of **5aa** (CCDC 1407355).



Displacement ellipsoids are drawn at the 30% probability level.

Table 1 Crystal data and structure refinement for his.

Identification code	his
Empirical formula	$C_{22}H_{18}O_2S$
Formula weight	346.42
Temperature/K	291.15
Crystal system	monoclinic
Space group	$P2_1$
a/Å	8.2262(3)
b/Å	13.4475(5)
c/Å	8.5887(5)
α/°	90
β/°	104.637(5)
γ/°	90
Volume/$Å^3$	919.27(7)
Z	2

ρ_{calc}g/cm^3	1.252
μ/mm^{-1}	1.646
F(000)	364.0
Crystal size/mm^3	0.2 × 0.13 × 0.12
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	10.646 to 134.152
Index ranges	-9 ≤ h ≤ 7, -16 ≤ k ≤ 16, -10 ≤ l ≤ 9
Reflections collected	6917
Independent reflections	3285 [R_{int} = 0.0334, R_{sigma} = 0.0422]
Data/restraints/parameters	3285/1/227
Goodness-of-fit on F^2	1.061
Final R indexes [I>=2σ (I)]	R_1 = 0.0357, wR_2 = 0.0892
Final R indexes [all data]	R_1 = 0.0401, wR_2 = 0.0940
Largest diff. peak/hole / e Å^{-3}	0.16/-0.14
Flack parameter	0.008(12)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for his. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	8768(5)	6786(4)	8977(5)	71.3(11)
C2	9207(7)	7746(4)	9450(7)	92.3(17)
C3	9587(7)	8434(4)	8404(8)	96.5(19)
C4	9510(6)	8185(3)	6803(7)	78.1(13)
C5	9044(4)	7232(2)	6368(4)	51.9(8)
C6	8674(4)	6538(2)	7396(4)	47.3(7)
C7	8260(4)	5585(2)	6499(4)	43.3(6)
C8	8440(4)	5868(3)	4829(4)	48.5(7)
C9	6493(4)	5155(3)	6421(5)	55.5(8)
C10	5064(4)	5804(3)	5520(5)	52.9(8)
C11	4687(4)	6697(3)	6156(5)	62.8(10)
C12	3348(5)	7268(3)	5302(7)	76.6(12)
C13	2377(5)	6957(4)	3847(6)	81.6(14)
C14	2733(6)	6080(4)	3225(6)	82.4(14)

C15	4076(5)	5497(3)	4045(5)	67.4(10)
C16	11765(4)	5187(3)	7396(6)	63.9(10)
C17	13147(3)	4511(3)	8258(4)	50.0(7)
C18	13397(5)	3600(3)	7620(5)	64.1(10)
C19	14662(6)	2978(4)	8396(7)	80.9(13)
C20	15718(6)	3245(4)	9800(7)	90.7(16)
C21	15517(6)	4137(5)	10465(5)	90.4(16)
C22	14229(5)	4775(3)	9702(5)	70.5(11)
O1	8890(3)	6845.7(19)	4827(3)	57.7(6)
O2	8238(4)	5373(2)	3658(3)	73.1(8)
S1	9752.8(9)	4594.4(6)	7337.8(12)	56.7(3)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for his. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	65(2)	87(3)	60(2)	-9(2)	11.0(18)	3(2)
C2	86(3)	96(4)	82(3)	-43(3)	-2(3)	9(3)
C3	84(3)	55(3)	129(5)	-40(3)	-12(3)	4(2)
C4	67(2)	44(2)	111(4)	5(2)	1(2)	-3.3(18)
C5	41.3(16)	43.5(16)	67(2)	5.9(15)	6.3(14)	2.0(13)
C6	38.3(15)	49.5(17)	53.1(17)	-4.2(13)	9.6(13)	4.5(13)
C7	35.3(14)	38.3(14)	56.0(17)	4.0(13)	11.2(12)	3.9(11)
C8	37.6(15)	52.6(17)	55.1(18)	-2.3(15)	11.3(13)	2.7(13)
C9	40.5(16)	46.3(18)	81(2)	7.4(17)	18.5(16)	0.0(14)
C10	35.5(16)	52.5(19)	74(2)	8.0(16)	20.9(15)	-2.0(13)
C11	44.8(18)	56(2)	90(3)	3.0(19)	21.5(18)	1.3(15)
C12	59(2)	61(2)	118(4)	16(2)	38(2)	13.7(19)
C13	53(2)	95(3)	102(3)	40(3)	27(2)	21(2)
C14	51(2)	117(4)	77(3)	18(3)	12(2)	3(2)
C15	48.4(19)	76(3)	79(3)	-2(2)	18.6(18)	-0.1(18)
C16	37.0(16)	54(2)	100(3)	15(2)	16.7(18)	-3.5(14)
C17	35.0(13)	55.0(17)	60.9(18)	0.8(17)	13.9(12)	-1.6(15)
C18	52(2)	65(2)	70(2)	-9.6(18)	6.5(17)	1.3(17)
C19	66(3)	66(3)	109(4)	-5(2)	20(2)	19(2)
C20	59(2)	105(4)	102(4)	24(3)	8(2)	26(3)
C21	68(3)	129(5)	59(3)	0(3)	-13(2)	-4(3)
C22	68(2)	76(3)	68(2)	-20(2)	20.0(19)	-4(2)
O1	59.6(14)	53.8(14)	59.7(14)	13.2(11)	15.5(11)	0.8(11)
O2	72.1(17)	85(2)	62.1(16)	-20.6(15)	16.3(13)	0.4(15)

S1	39.1(4)	43.4(4)	87.5(6)	18.3(4)	15.6(3)	4.5(4)

Table 4 Bond Lengths for his.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.374(7)	C10	C11	1.387(5)
C1	C6	1.381(5)	C10	C15	1.384(6)
C2	C3	1.379(9)	C11	C12	1.390(6)
C3	C4	1.401(8)	C12	C13	1.367(7)
C4	C5	1.362(5)	C13	C14	1.357(7)
C5	C6	1.371(5)	C14	C15	1.392(6)
C5	O1	1.398(4)	C16	C17	1.497(5)
C6	C7	1.490(4)	C16	S1	1.827(3)
C7	C8	1.527(4)	C17	C18	1.378(6)
C7	C9	1.550(4)	C17	C22	1.378(5)
C7	S1	1.832(3)	C18	C19	1.370(6)
C8	O1	1.365(4)	C19	C20	1.344(7)
C8	O2	1.182(4)	C20	C21	1.357(7)
C9	C10	1.511(5)	C21	C22	1.391(7)

Table 5 Bond Angles for his.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	117.6(5)	C11	C10	C9	121.4(4)
C1	C2	C3	121.5(5)	C15	C10	C9	119.9(3)
C2	C3	C4	121.2(4)	C15	C10	C11	118.7(4)
C5	C4	C3	115.7(5)	C10	C11	C12	119.8(4)
C4	C5	C6	123.8(4)	C13	C12	C11	121.0(4)
C4	C5	O1	124.2(4)	C14	C13	C12	119.4(4)
C6	C5	O1	112.0(3)	C13	C14	C15	120.9(5)
C1	C6	C7	131.6(4)	C10	C15	C14	120.2(4)
C5	C6	C1	120.2(4)	C17	C16	S1	108.6(2)
C5	C6	C7	108.1(3)	C18	C17	C16	120.9(3)
C6	C7	C8	102.0(3)	C22	C17	C16	121.7(4)
C6	C7	C9	115.6(3)	C22	C17	C18	117.3(4)
C6	C7	S1	112.3(2)	C19	C18	C17	121.4(4)
C8	C7	C9	111.4(3)	C20	C19	C18	120.8(5)
C8	C7	S1	109.6(2)	C19	C20	C21	119.6(4)
C9	C7	S1	105.9(2)	C20	C21	C22	120.4(4)

O1	C8	C7	109.4(3)	C17	C22	C21	120.5(4)
O2	C8	C7	129.2(3)	C8	O1	C5	108.5(3)
O2	C8	O1	121.4(3)	C16	S1	C7	101.89(15)
C10	C9	C7	114.0(3)				

Table 6 Torsion Angles for his.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C2	C3	C4	1.2(8)	C9	C7	C8	O2	-55.7(4)
C1	C6	C7	C8	-178.9(4)	C9	C7	S1	C16	-179.3(3)
C1	C6	C7	C9	60.0(5)	C9	C10	C11	C12	-179.1(3)
C1	C6	C7	S1	-61.6(4)	C9	C10	C15	C14	178.5(4)
C2	C1	C6	C5	1.3(6)	C10	C11	C12	C13	0.9(6)
C2	C1	C6	C7	179.4(4)	C11	C10	C15	C14	0.1(6)
C2	C3	C4	C5	-0.1(7)	C11	C12	C13	C14	-0.4(7)
C3	C4	C5	C6	-0.4(6)	C12	C13	C14	C15	-0.2(7)
C3	C4	C5	O1	179.8(3)	C13	C14	C15	C10	0.4(7)
C4	C5	C6	C1	-0.3(5)	C15	C10	C11	C12	-0.7(5)
C4	C5	C6	C7	-178.7(3)	C16	C17	C18	C19	179.7(4)
C4	C5	O1	C8	178.7(3)	C16	C17	C22	C21	-179.1(4)
C5	C6	C7	C8	-0.6(3)	C17	C16	S1	C7	172.8(3)
C5	C6	C7	C9	-121.7(3)	C17	C18	C19	C20	-0.9(8)
C5	C6	C7	S1	116.7(3)	C18	C17	C22	C21	0.2(6)
C6	C1	C2	C3	-1.8(8)	C18	C19	C20	C21	0.7(9)
C6	C5	O1	C8	-1.2(4)	C19	C20	C21	C22	0.0(9)
C6	C7	C8	O1	-0.1(3)	C20	C21	C22	C17	-0.4(8)
C6	C7	C8	O2	-179.6(4)	C22	C17	C18	C19	0.5(6)
C6	C7	C9	C10	64.0(4)	O1	C5	C6	C1	179.6(3)
C6	C7	S1	C16	-52.2(3)	O1	C5	C6	C7	1.2(4)
C7	C8	O1	C5	0.8(3)	O2	C8	O1	C5	-179.7(3)
C7	C9	C10	C11	-71.2(4)	S1	C7	C8	O1	-119.3(2)
C7	C9	C10	C15	110.4(4)	S1	C7	C8	O2	61.2(4)
C8	C7	C9	C10	-51.8(4)	S1	C7	C9	C10	-170.9(3)
C8	C7	S1	C16	60.4(3)	S1	C16	C17	C18	65.5(4)
C9	C7	C8	O1	123.8(3)	S1	C16	C17	C22	-115.3(4)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for his.

Atom	x	y	z	U(eq)
H1	8543	6320	9694	86
H2	9250	7936	10501	111
H3	9900	9074	8767	116
H4	9760	8641	6084	94
H9A	6397	4508	5907	67
H9B	6385	5059	7510	67
H11	5329	6914	7150	75
H12	3110	7871	5726	92
H13	1480	7343	3289	98
H14	2072	5866	2237	99
H15	4309	4899	3600	81
H16A	11862	5310	6310	77
H16B	11841	5820	7953	77
H18	12692	3404	6643	77
H19	14792	2363	7946	97
H20	16580	2820	10312	109
H21	16246	4324	11436	108
H22	14097	5383	10171	85

Experimental

The crystal was kept at 291.15 K during data collection. Using Olex2[1], the structure was solved with the ShelXS[2] structure solution program using Direct Methods and refined with the ShelXL[3] refinement package using Least Squares minimisation.

Reference:

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. *Appl. Cryst*. 42, 339−341.
2. Sheldrick, G.M. (2008). *Acta Cryst.* A64, 112−122.
3. Sheldrick, G.M. (2008). *Acta Cryst.* A64, 112−122.

Crystal structure determination of [his]

Crystal Data for $C_{22}H_{18}O_2S$ (M =346.42 g/mol): monoclinic, space group $P2_1$ (no. 4), a = 8.2262(3) Å, b = 13.4475(5) Å, c = 8.5887(5) Å, β = 104.637(5)°, V = 919.27(7) Å^3, Z = 2, T = 291.15 K, μ(CuKα) = 1.646 mm^{-1}, $Dcalc$ = 1.252 g/cm^3, 6917 reflections measured (10.646° ≤ 2Θ

≤ 134.152°), 3285 unique (R_{int} = 0.0334, R_{sigma} = 0.0422) which were used in all calculations. The final R_1 was 0.0357 (I > 2σ(I)) and wR_2 was 0.0940 (all data).

Refinement model description

Number of restraints - 1, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

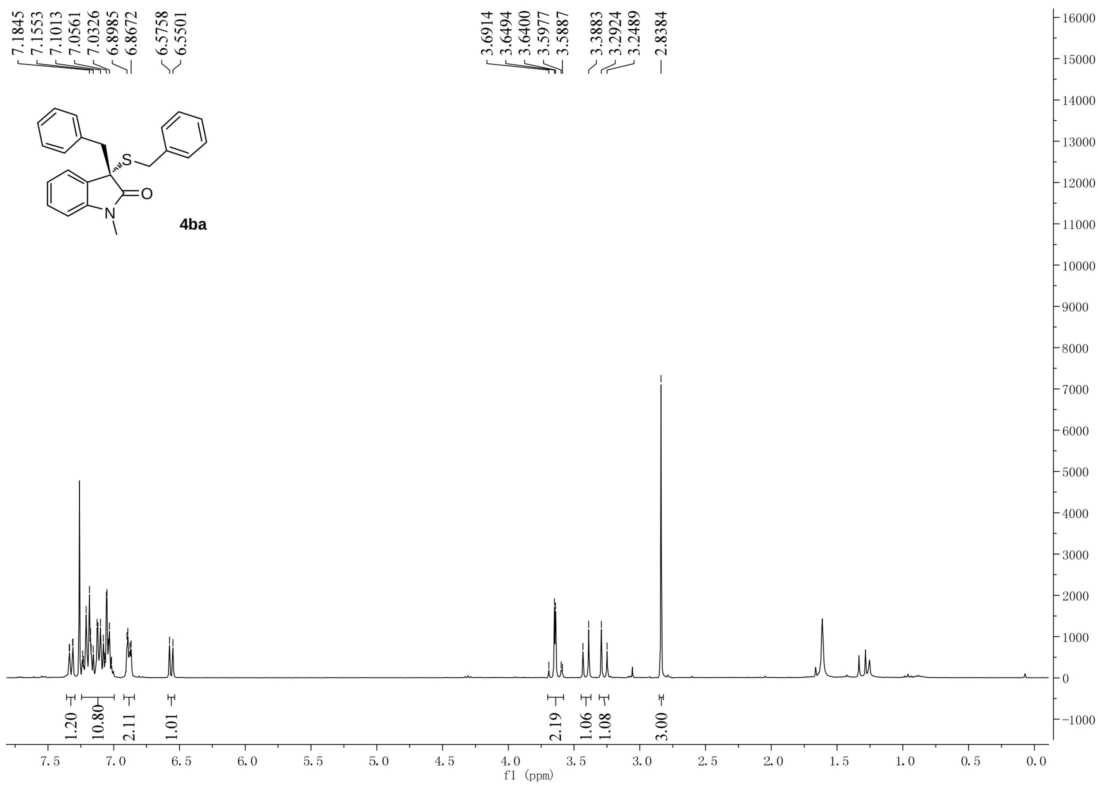
2.a Secondary CH2 refined with riding coordinates:

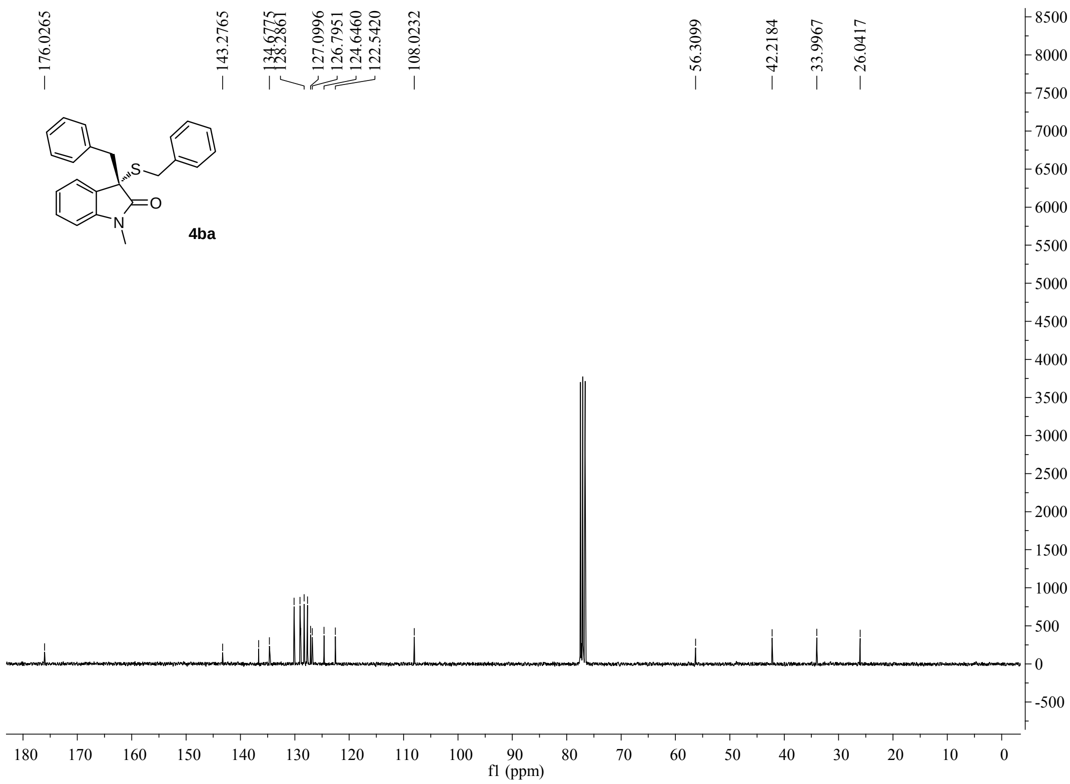
C9(H9A,H9B), C16(H16A,H16B)

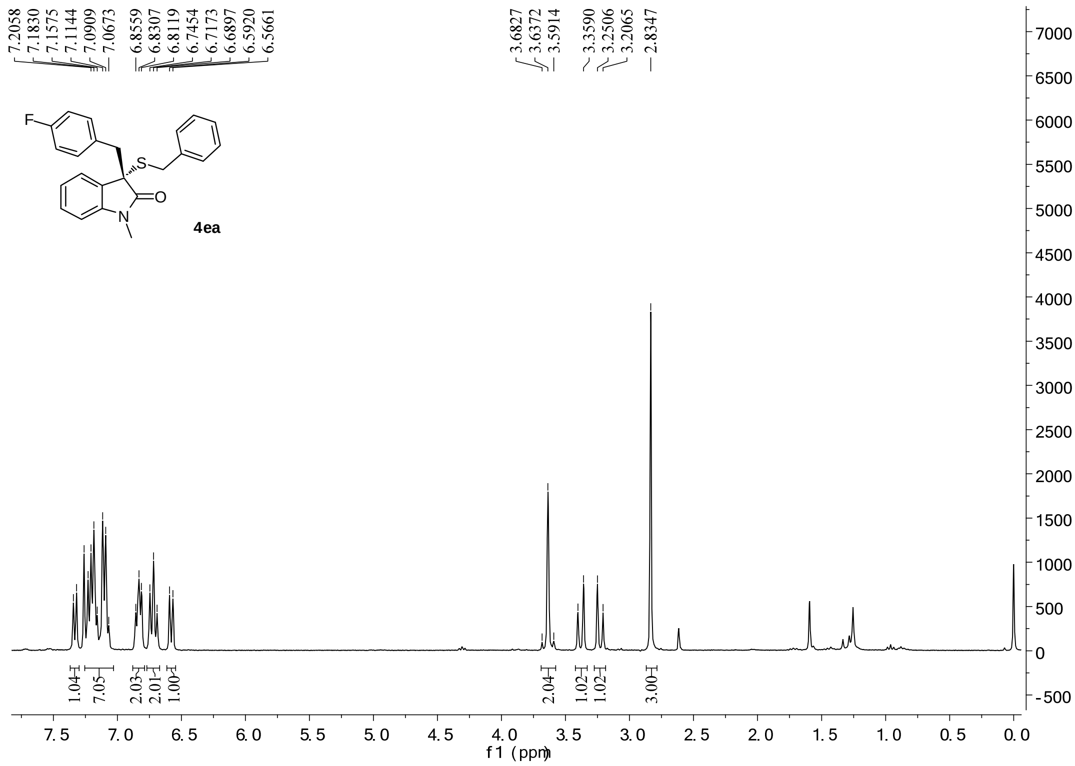
2.b Aromatic/amide H refined with riding coordinates:

C1(H1), C2(H2), C3(H3), C4(H4), C11(H11), C12(H12), C13(H13), C14(H14), C15(H15), C18(H18), C19(H19), C20(H20), C21(H21), C22(H22)

4. Copies of NMR spectra



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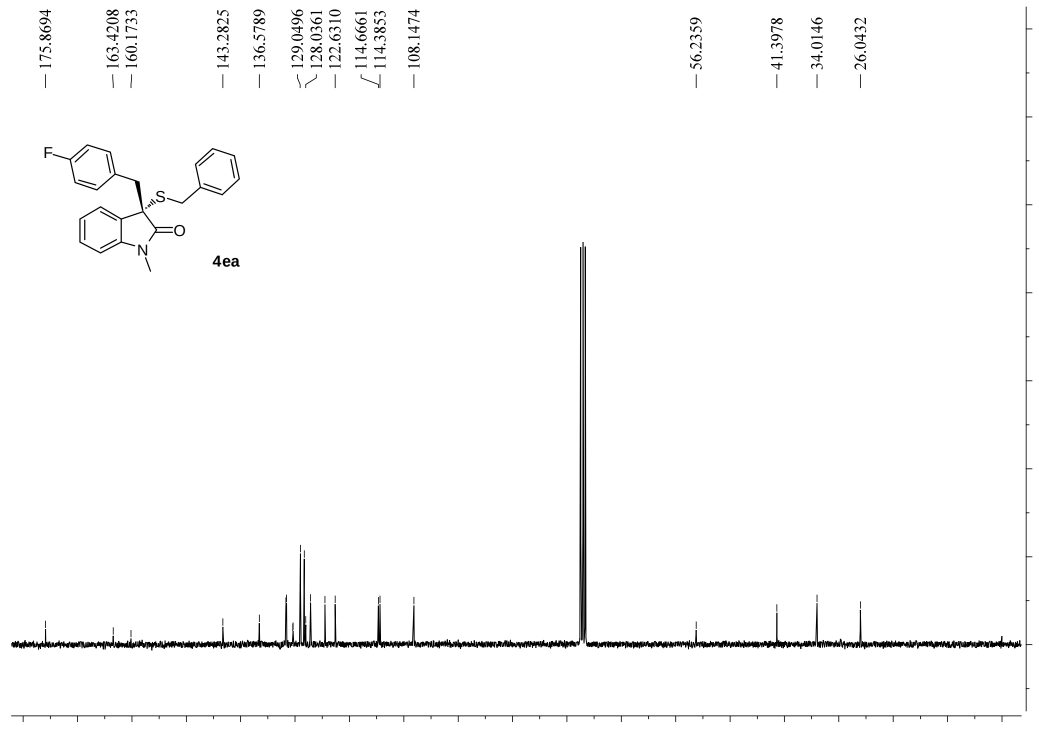
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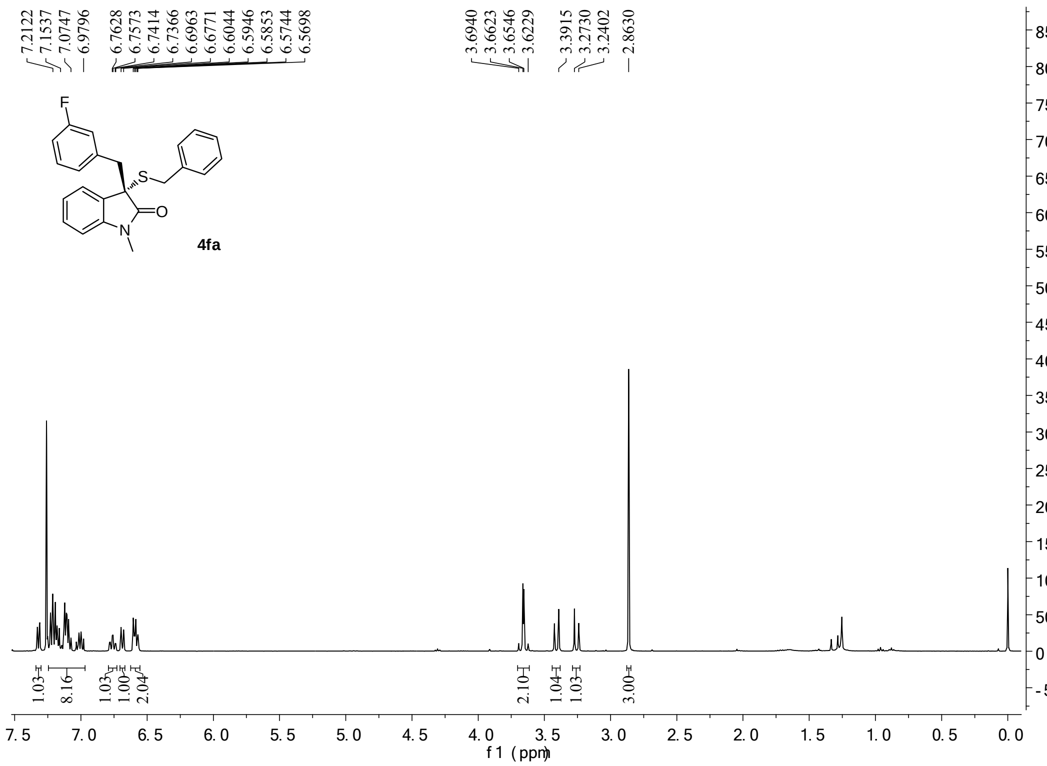
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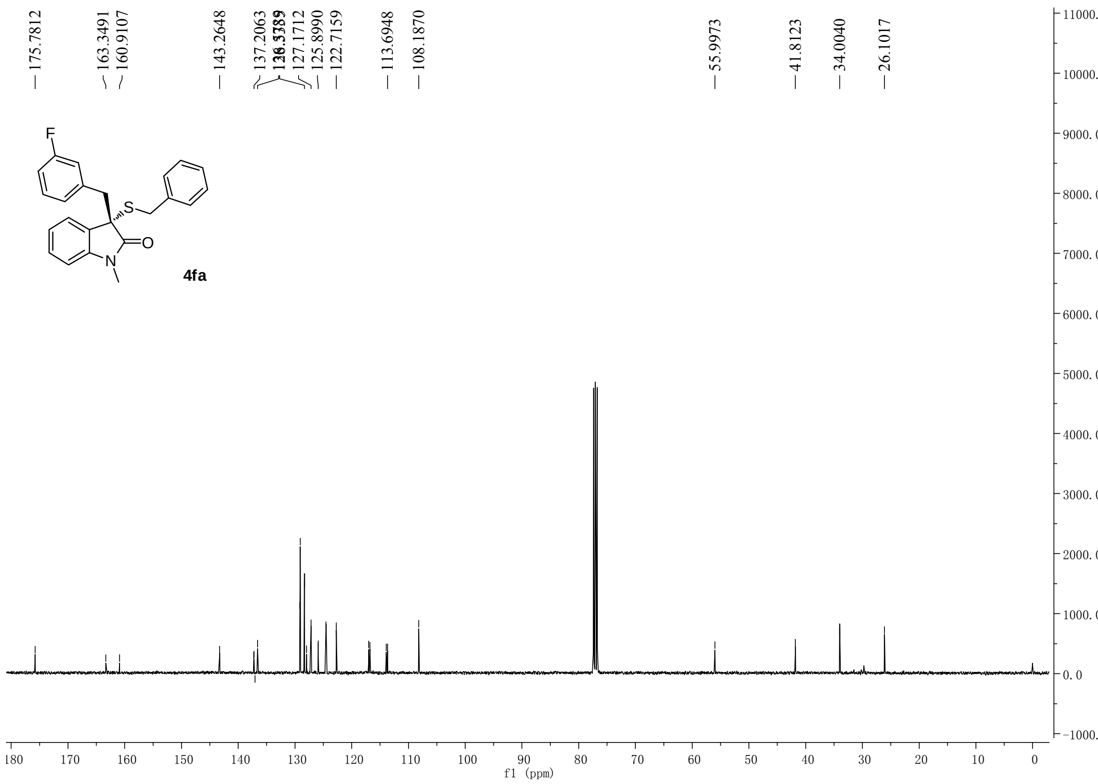
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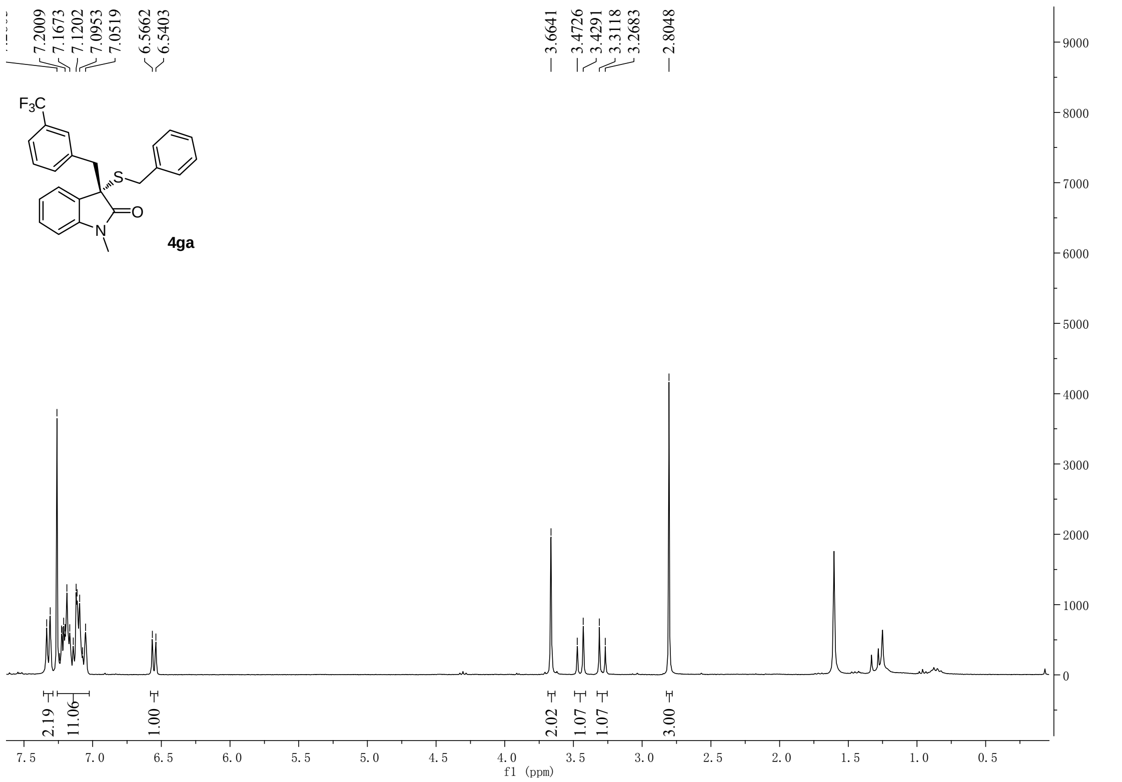

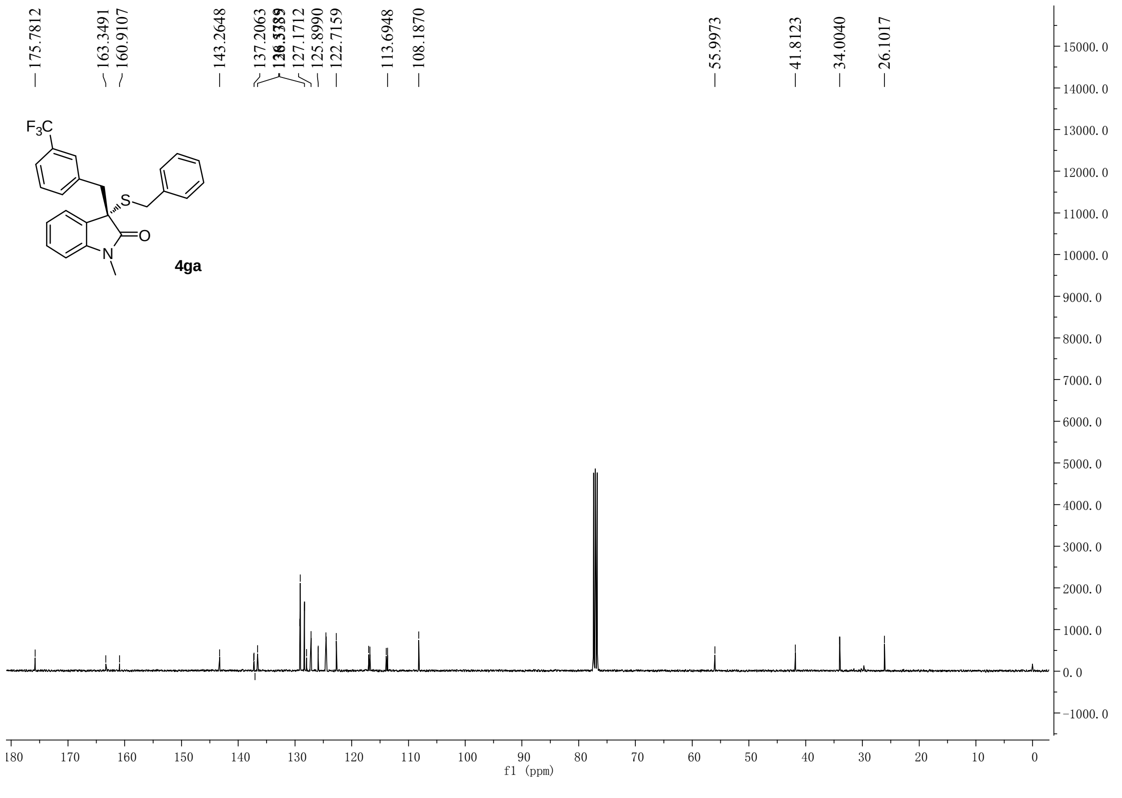

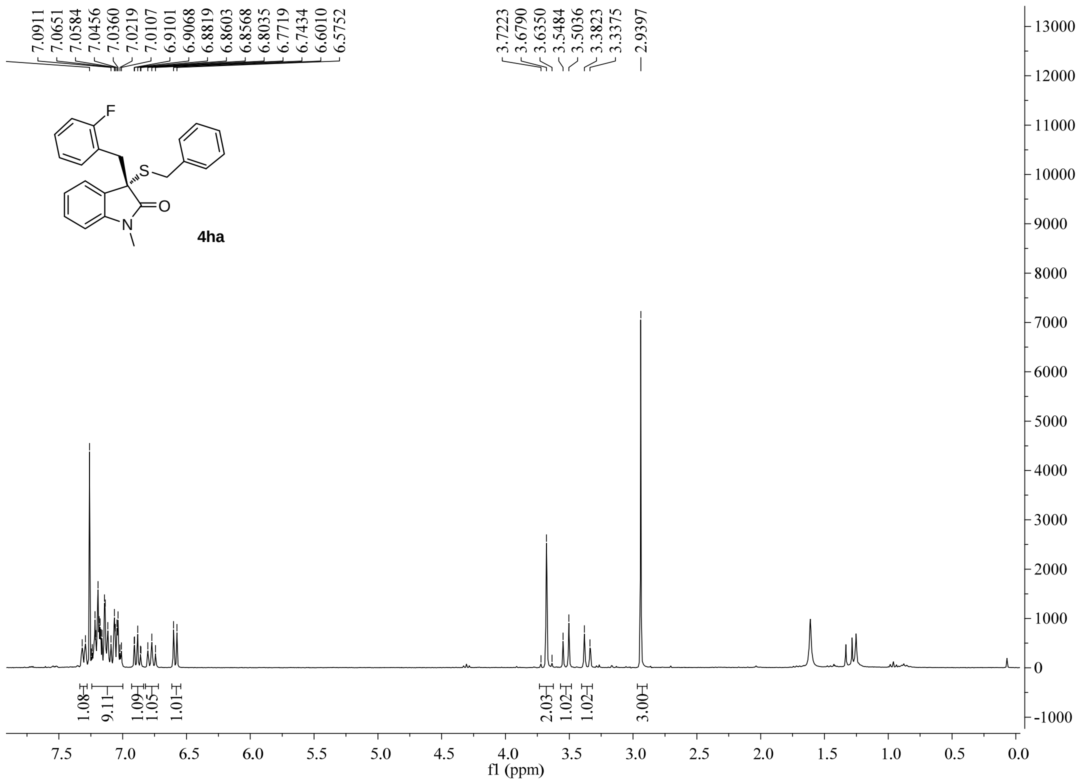
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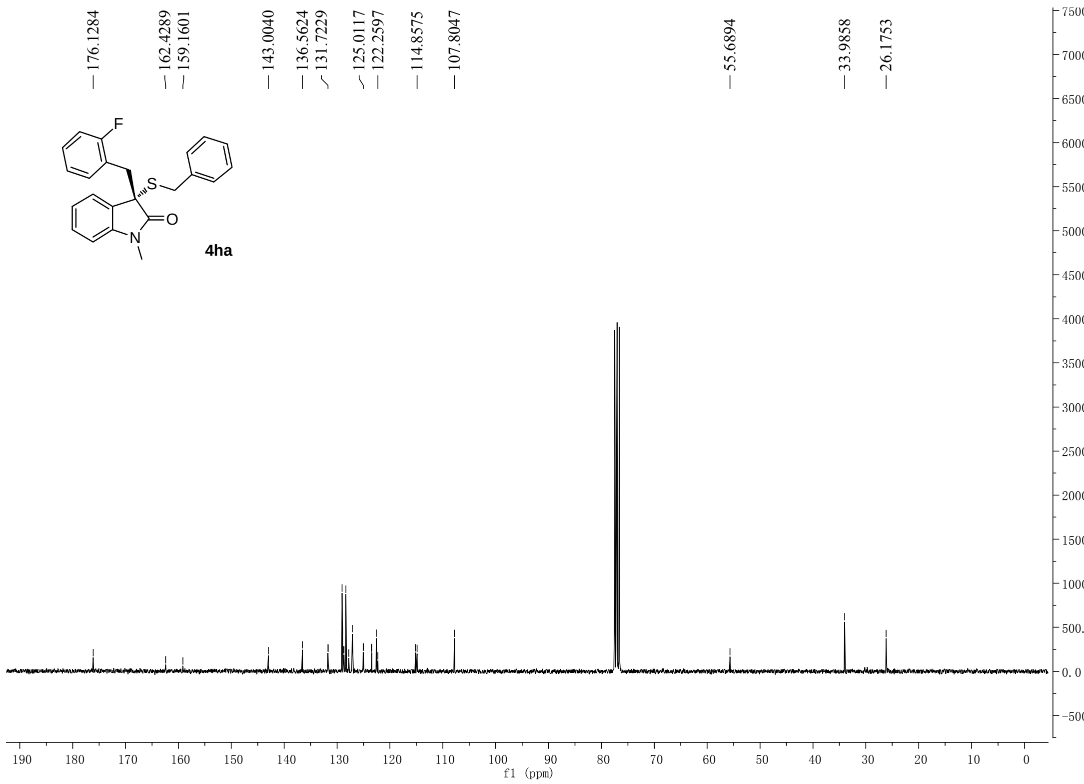
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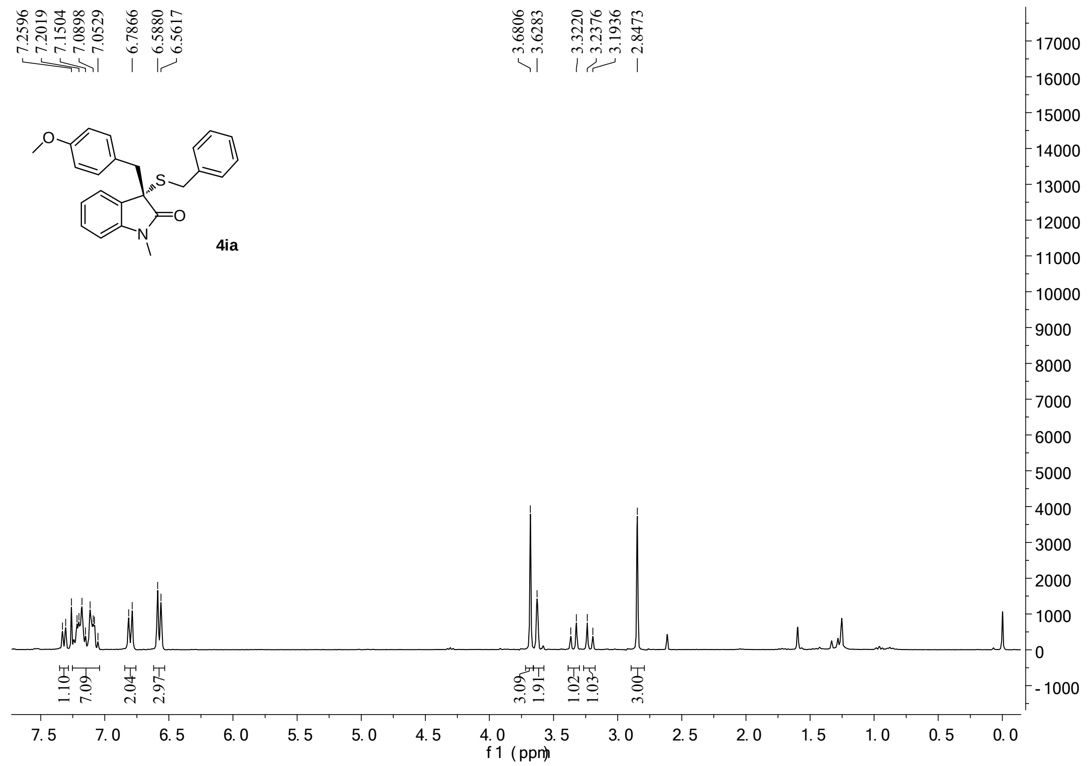
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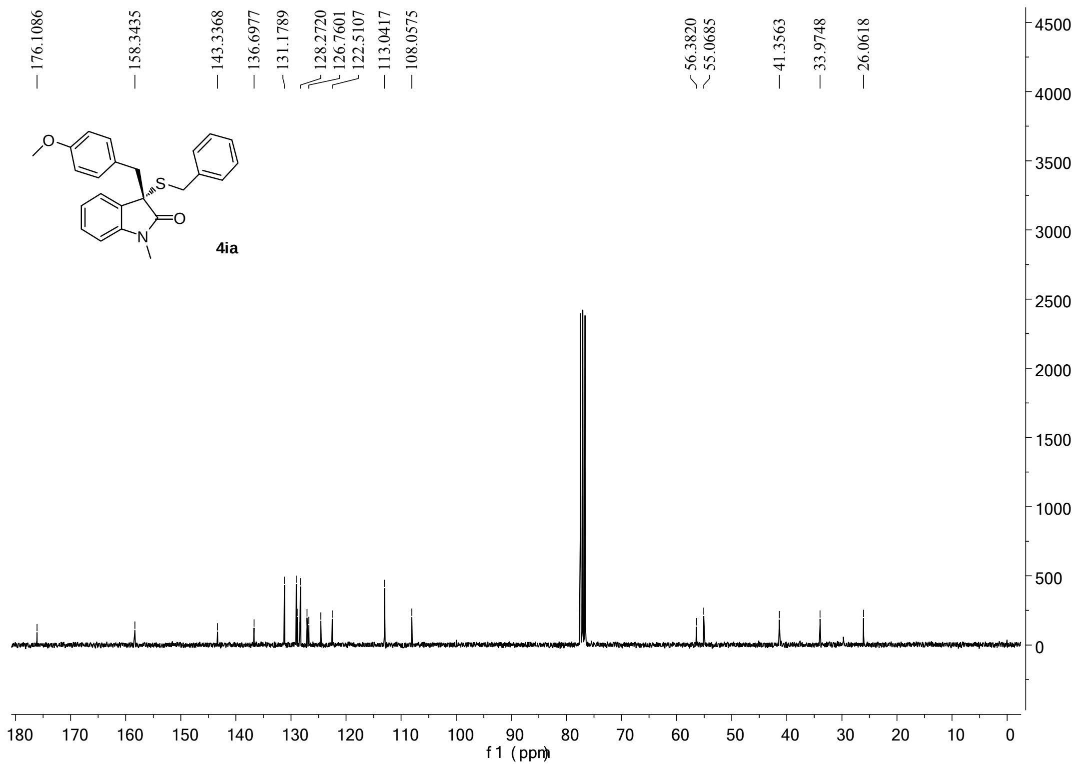
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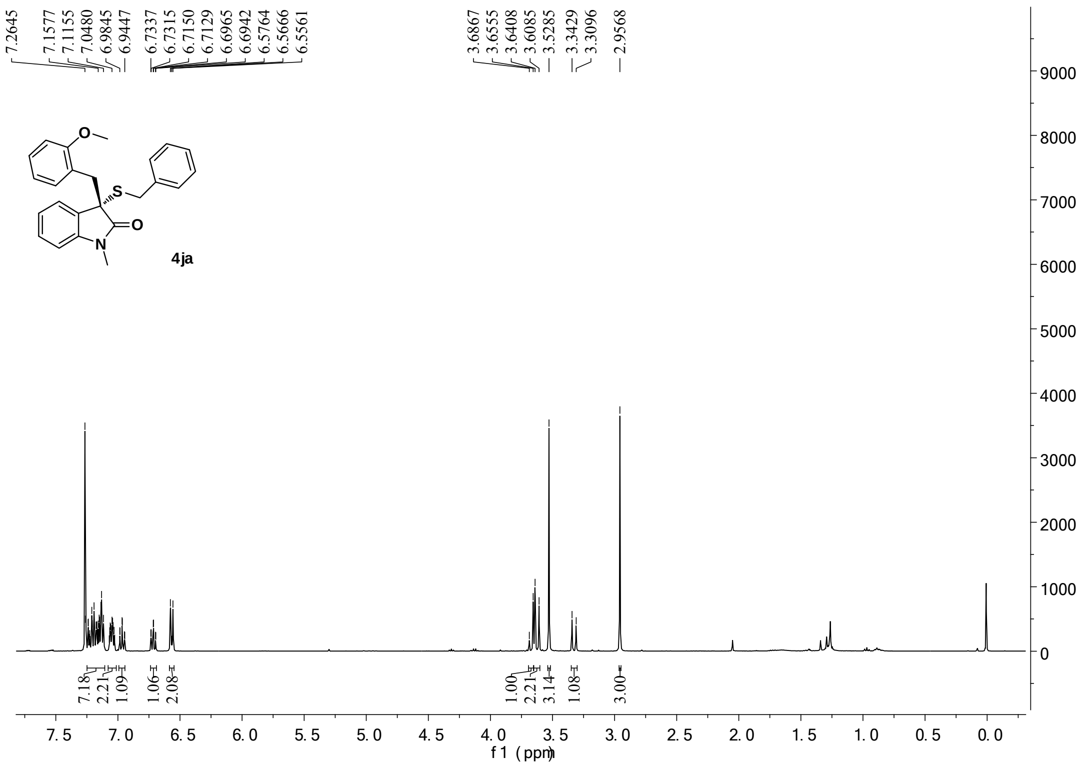
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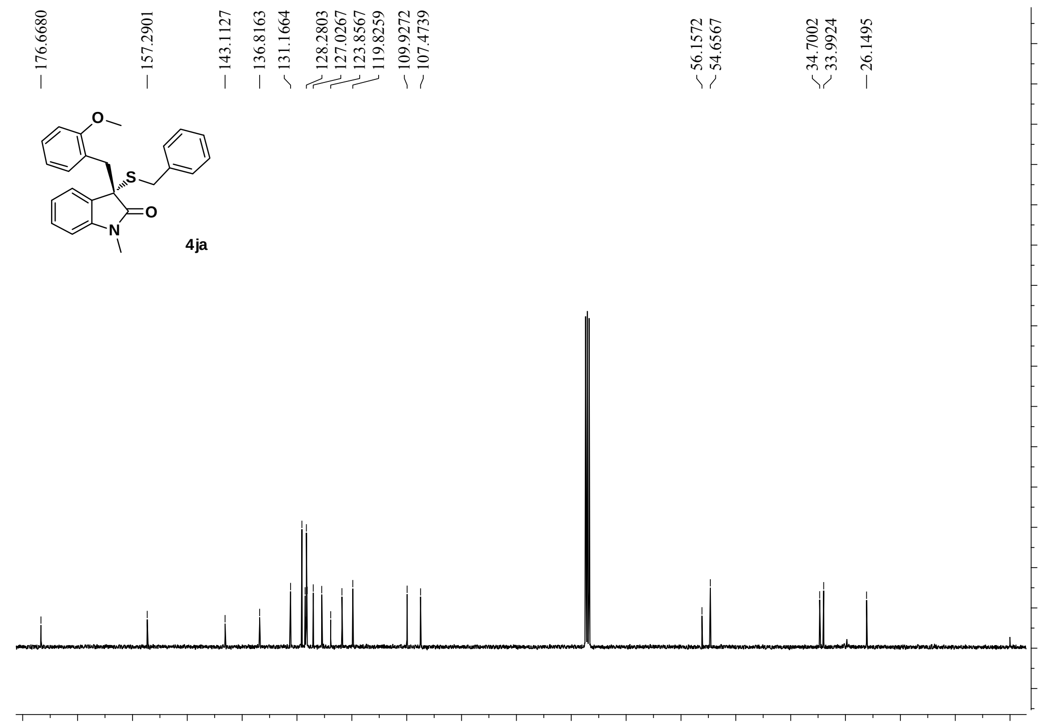
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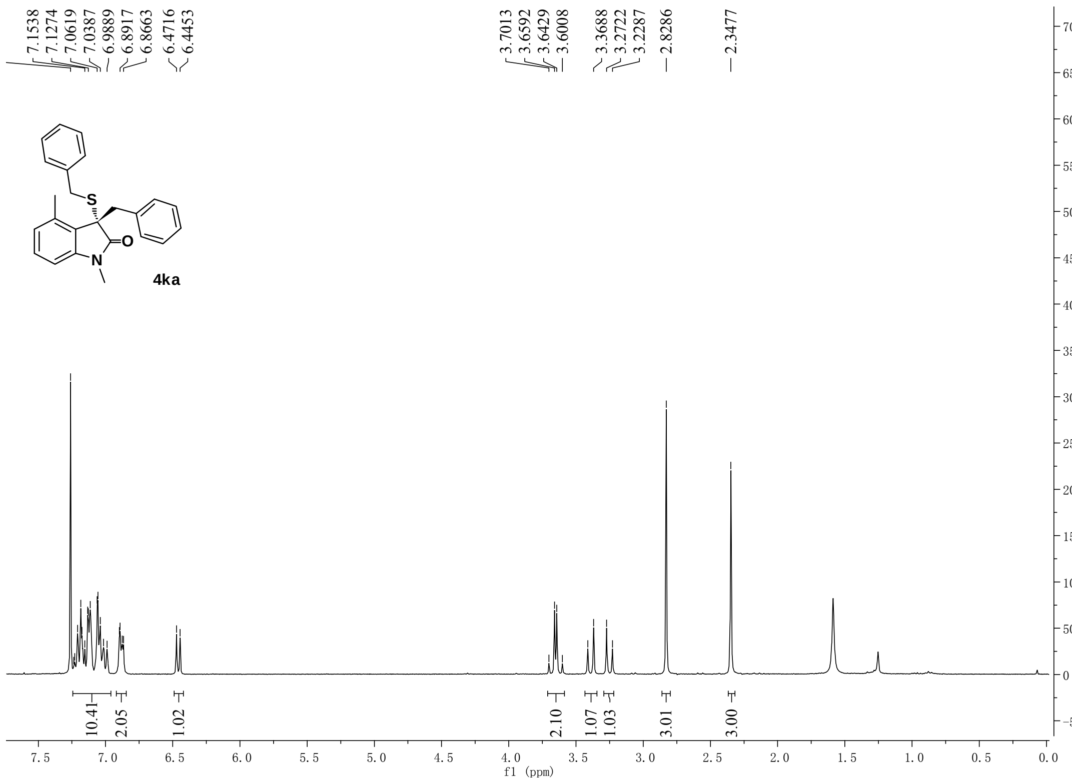
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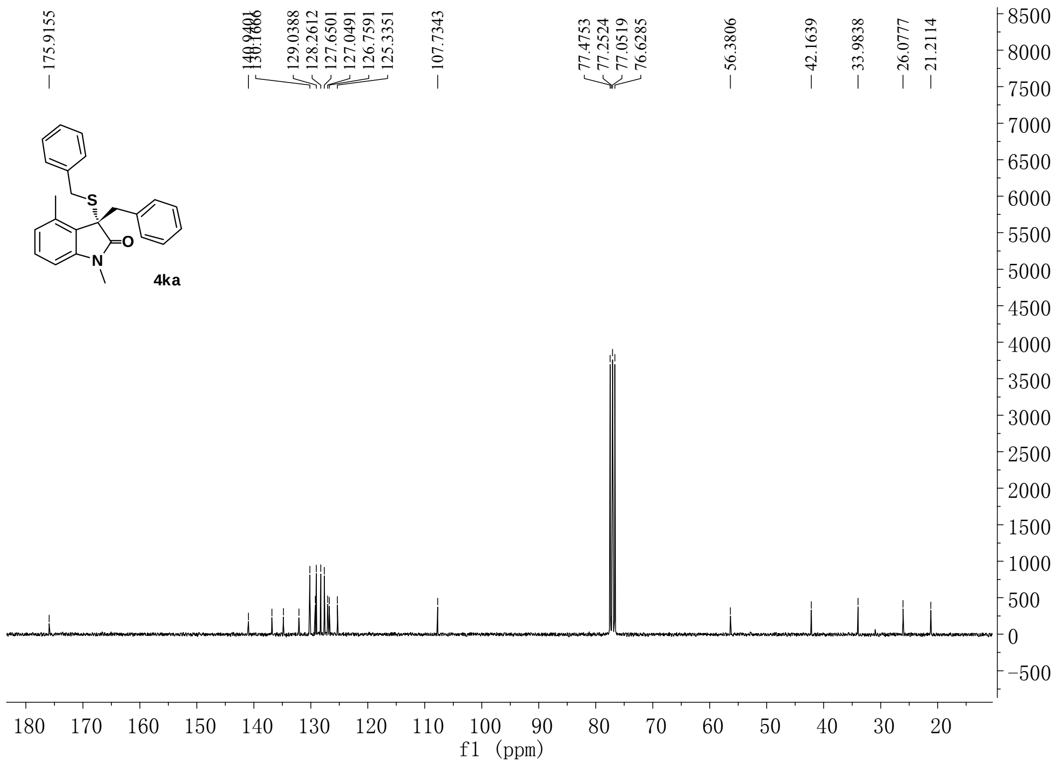
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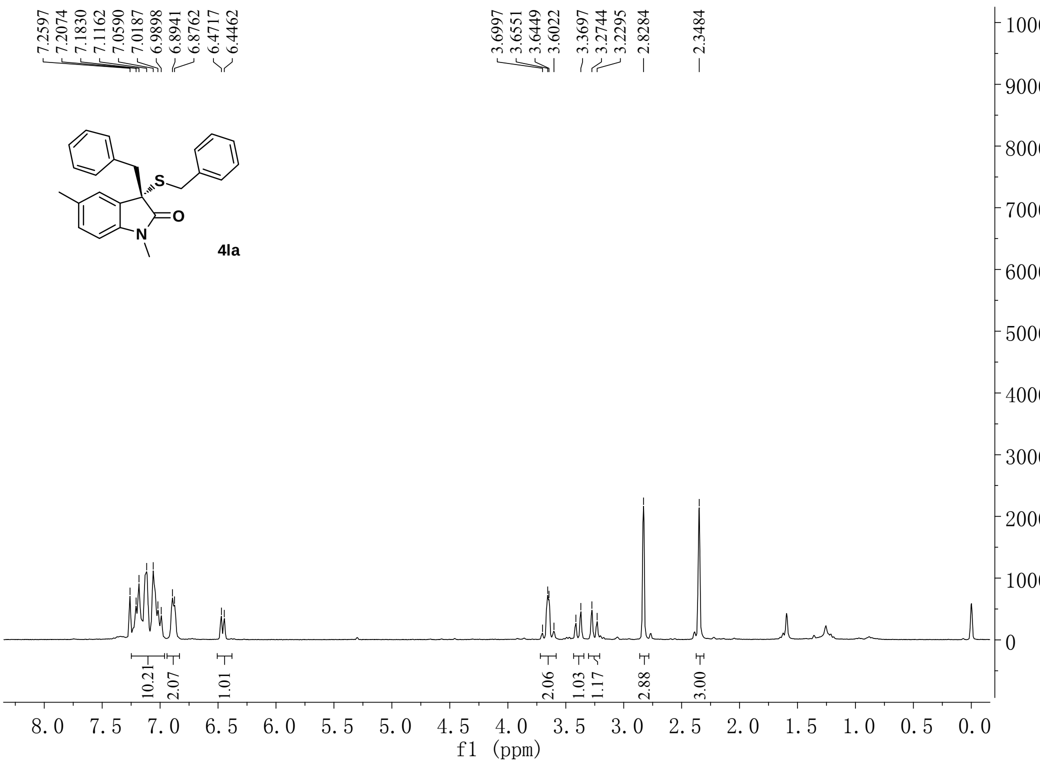
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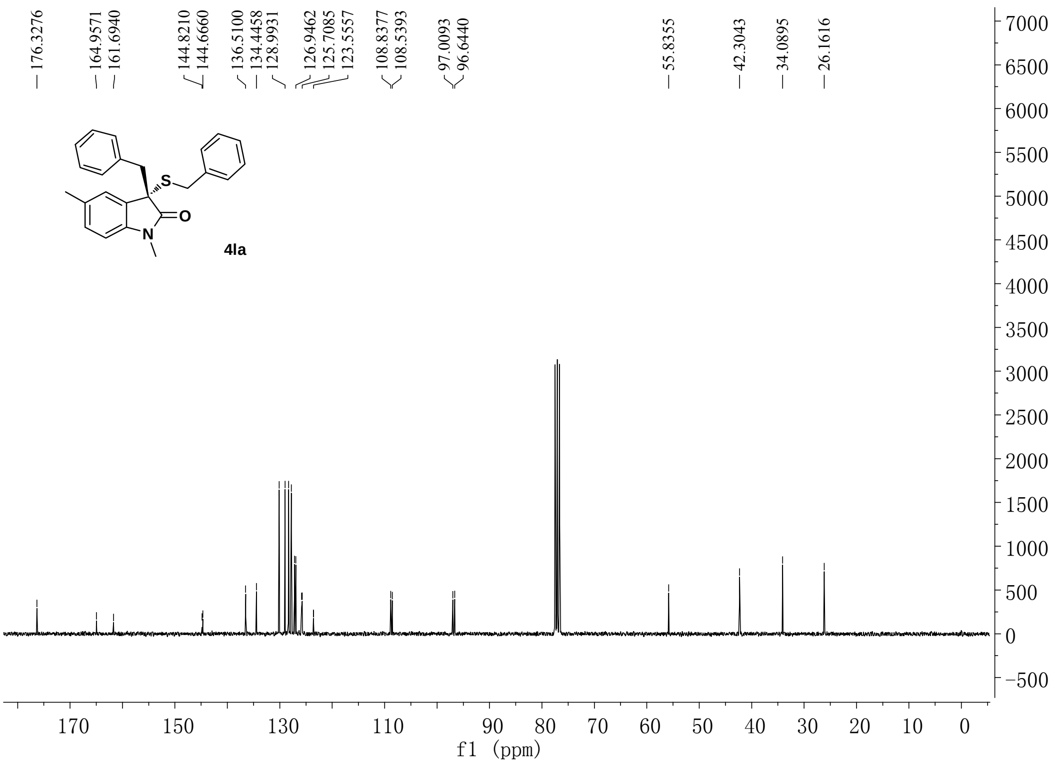
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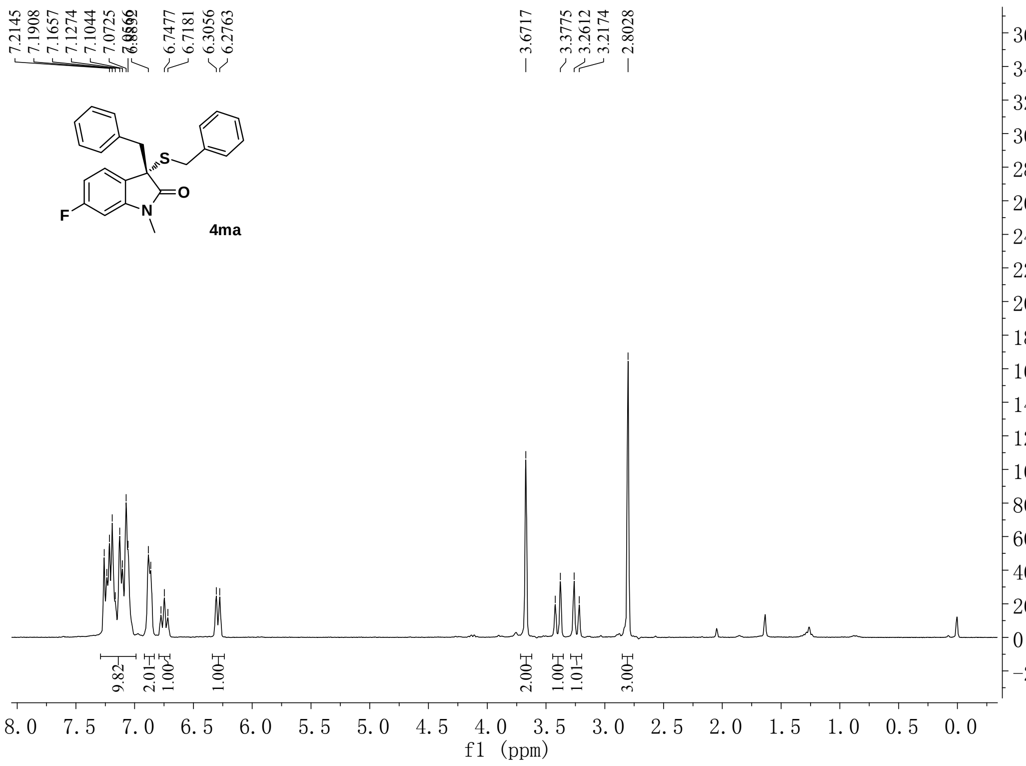
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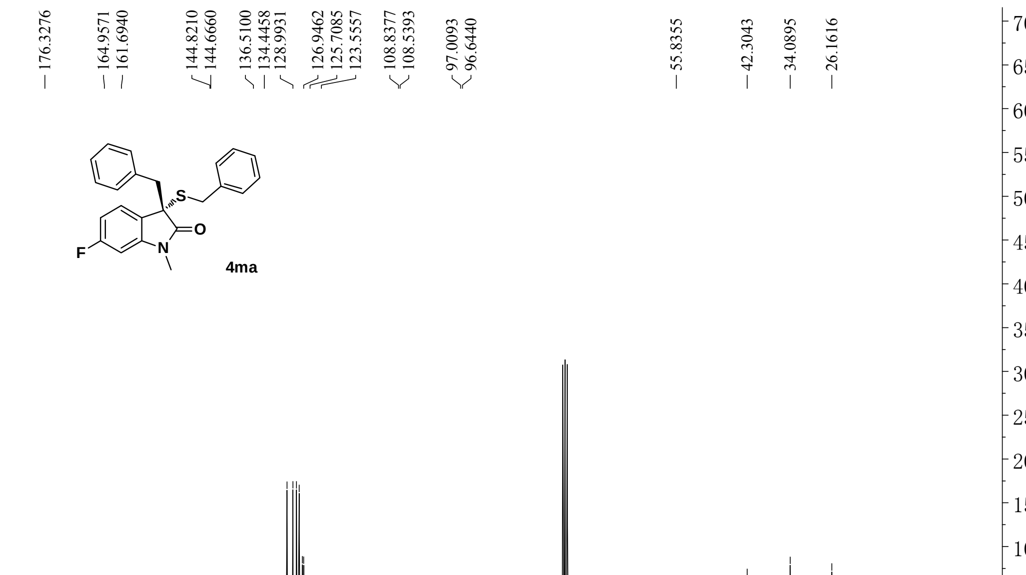
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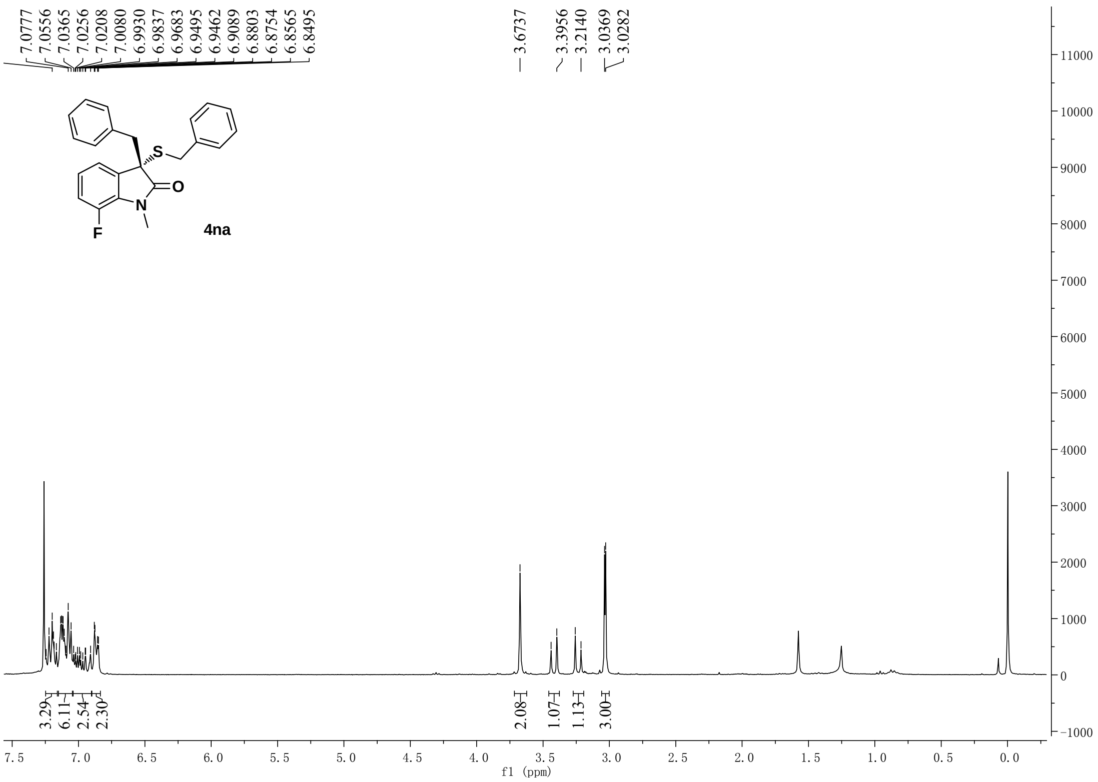
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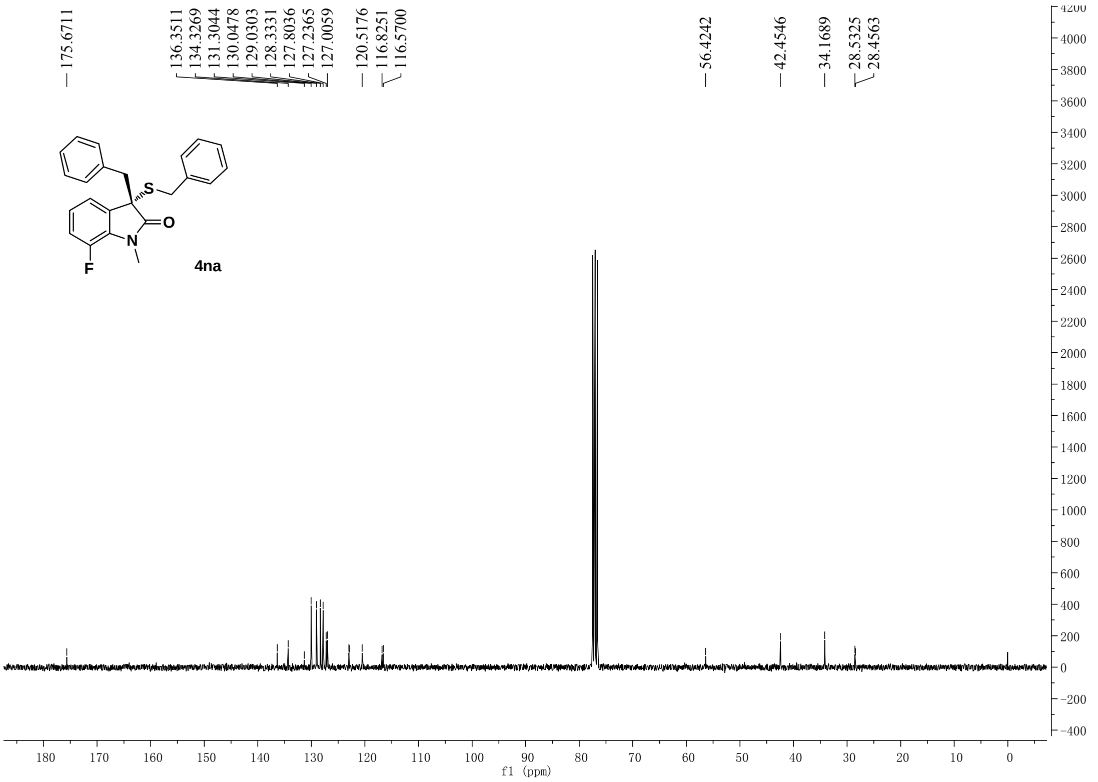
f1 (ppm)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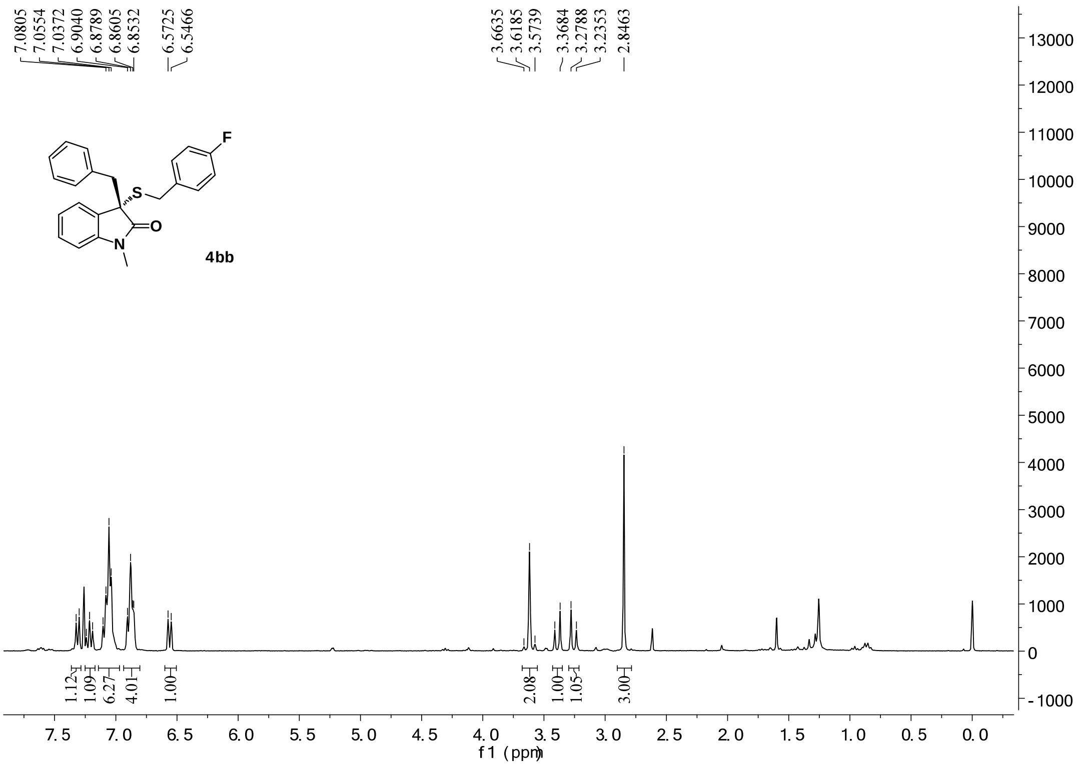
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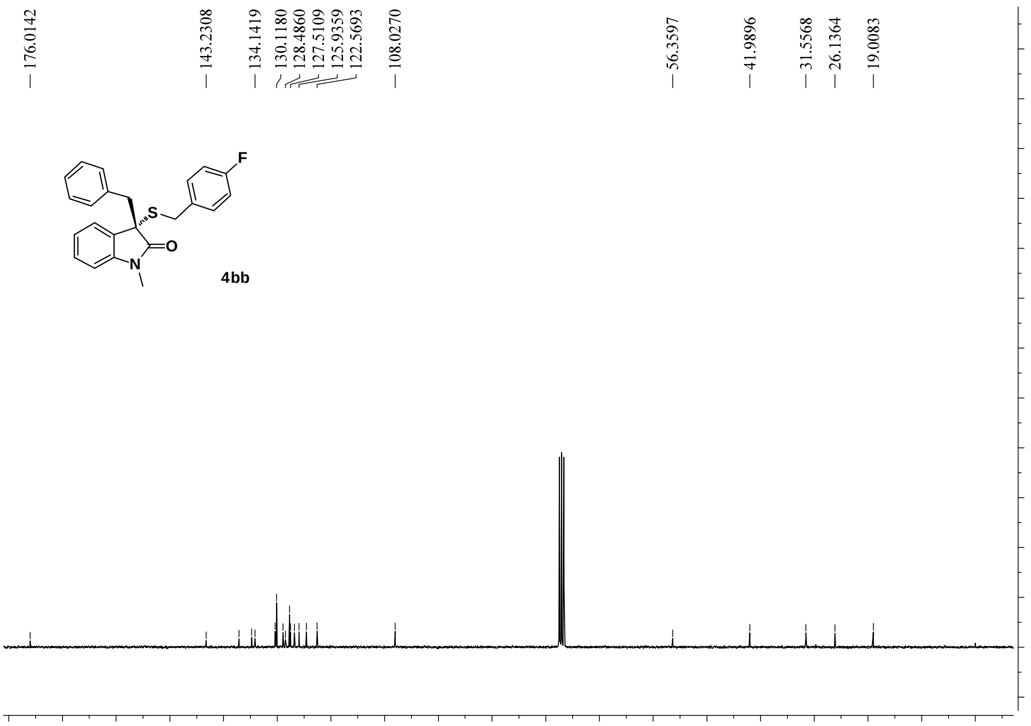
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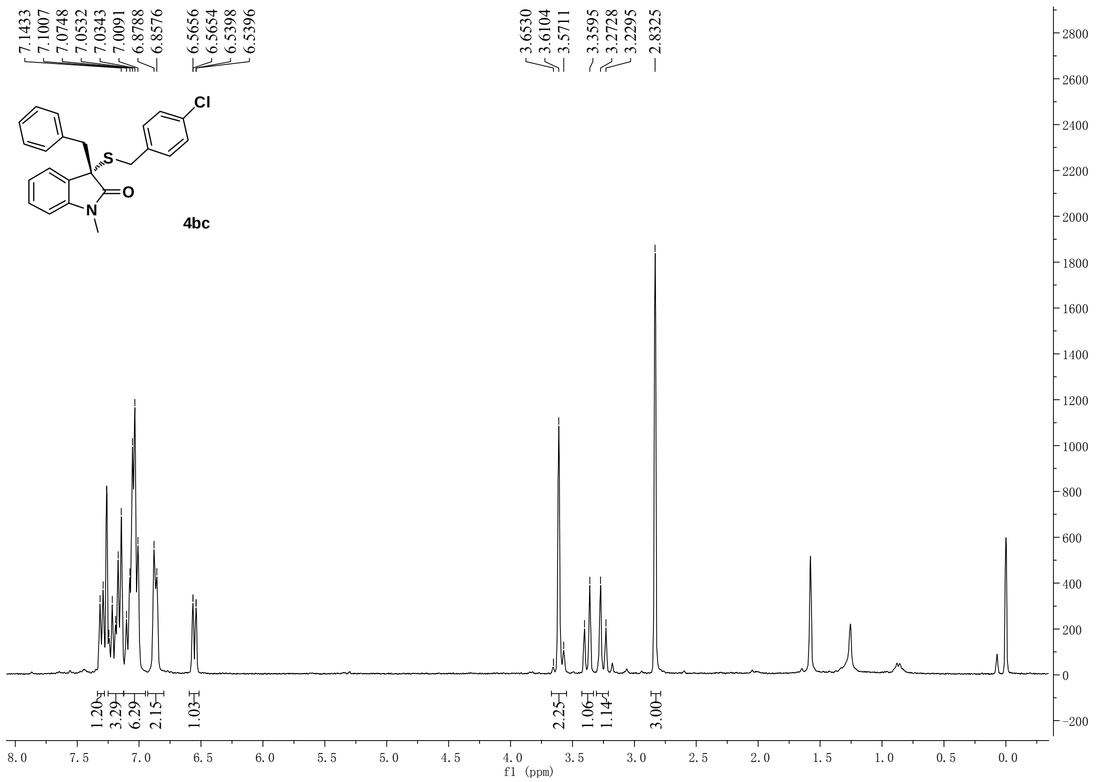
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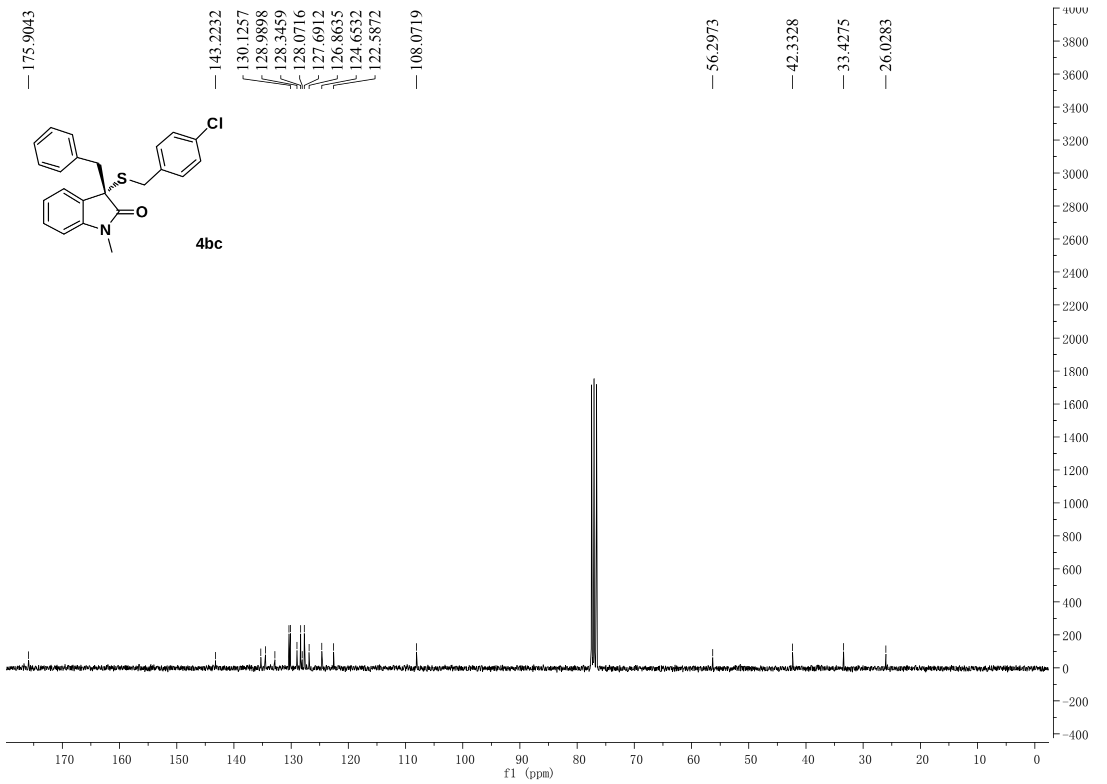
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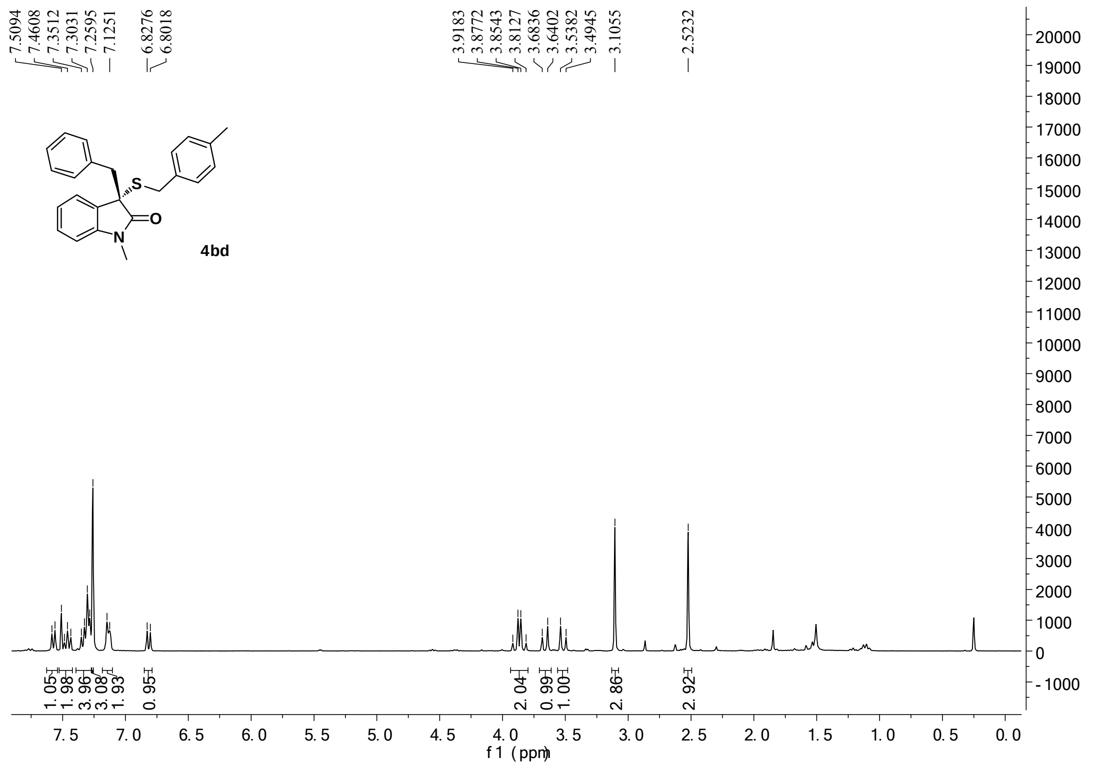
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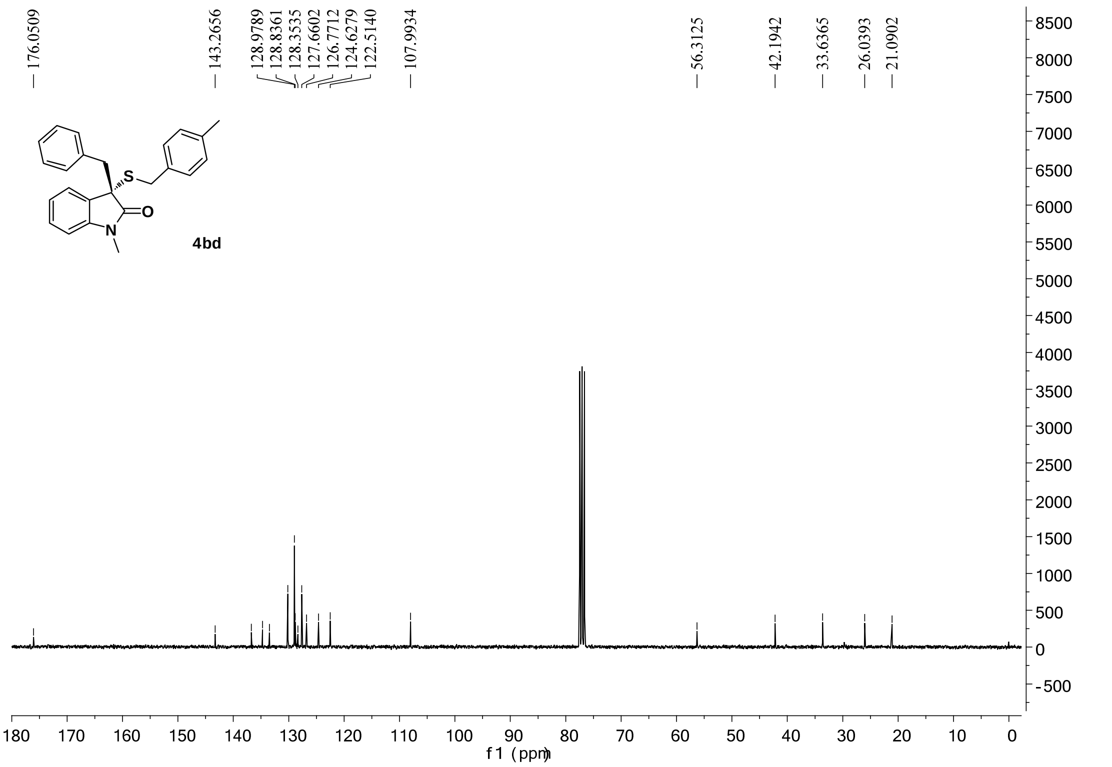
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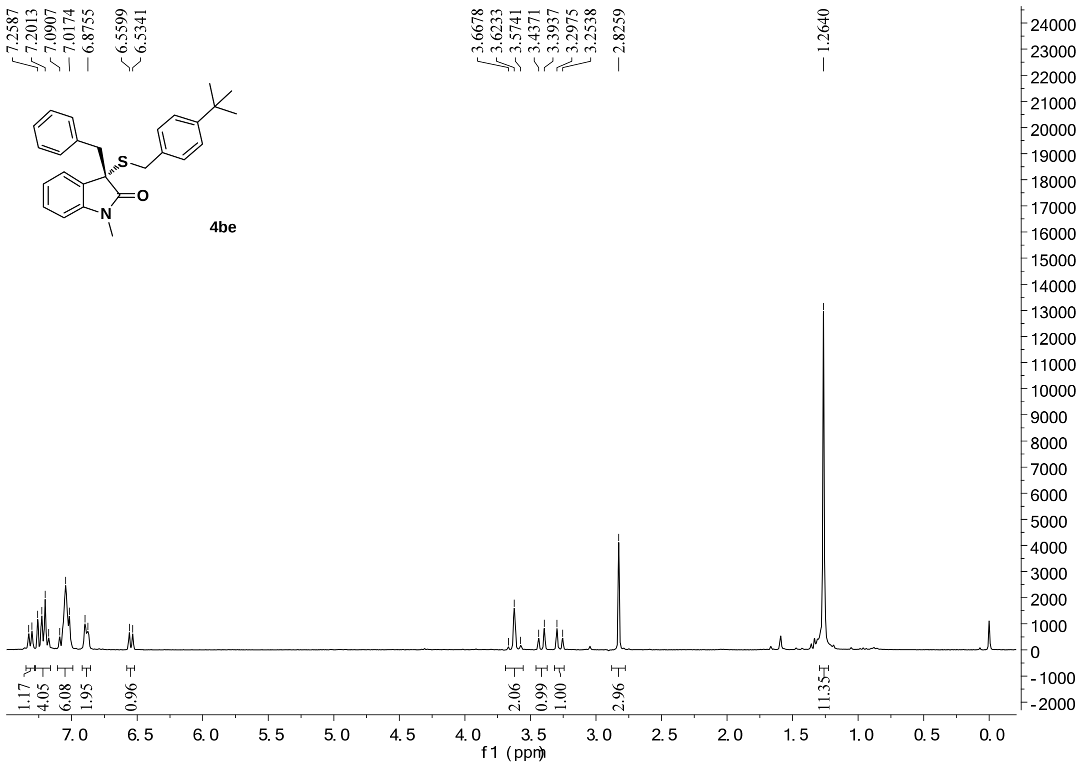
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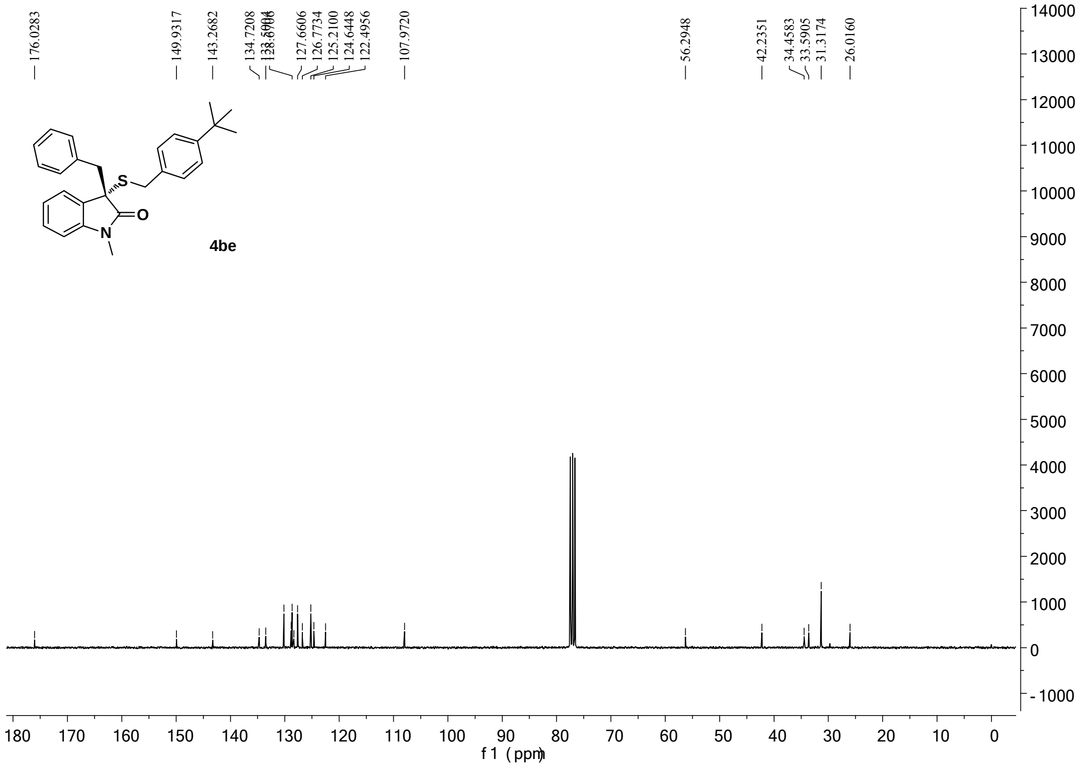
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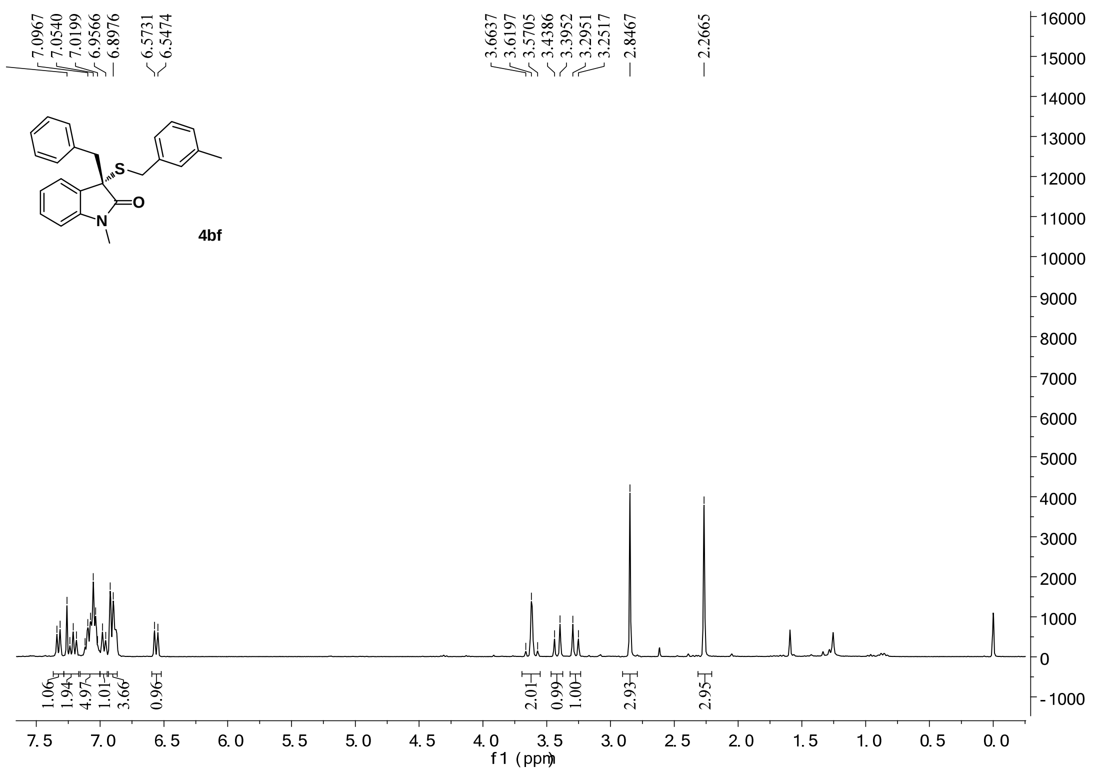
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4.97
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3.66
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2.01
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1.00
2.93
2.95
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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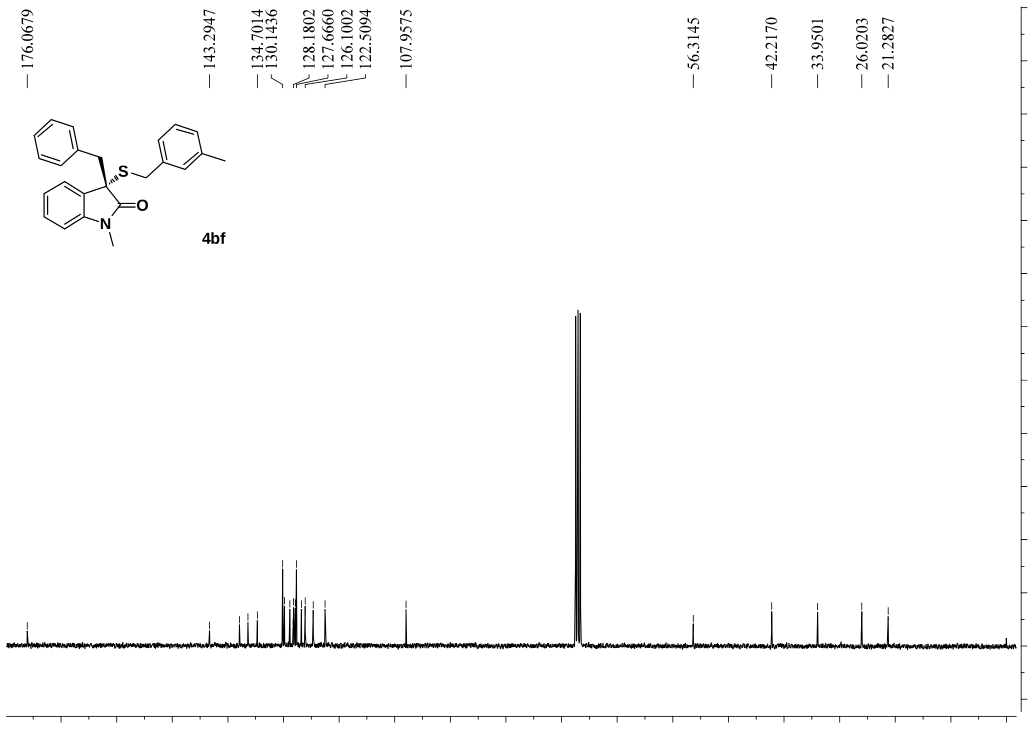
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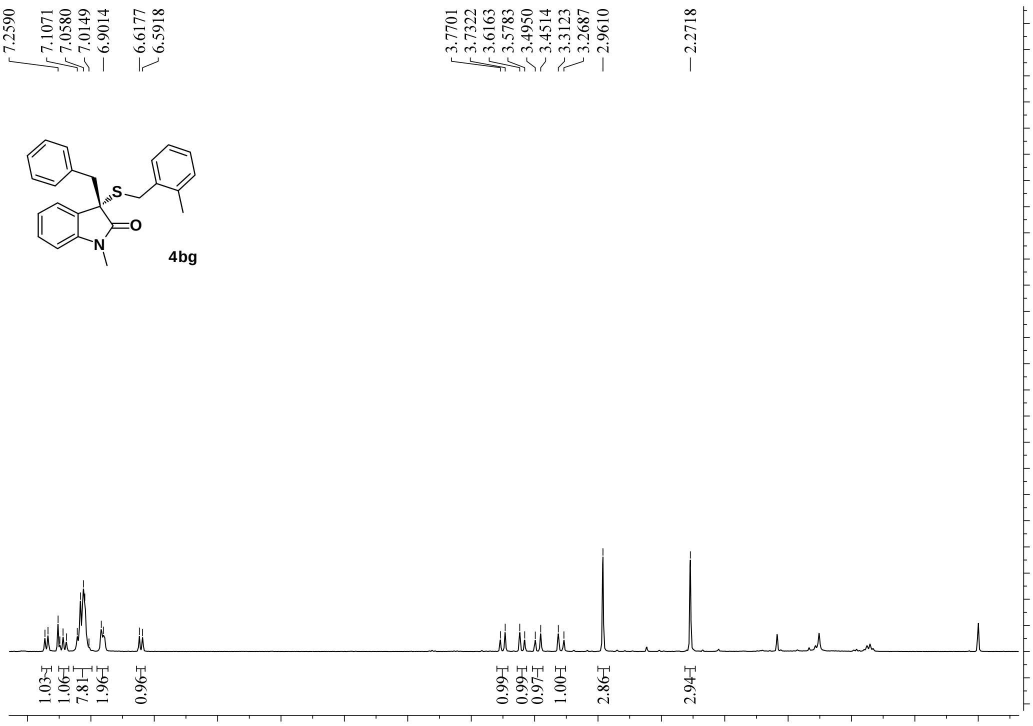
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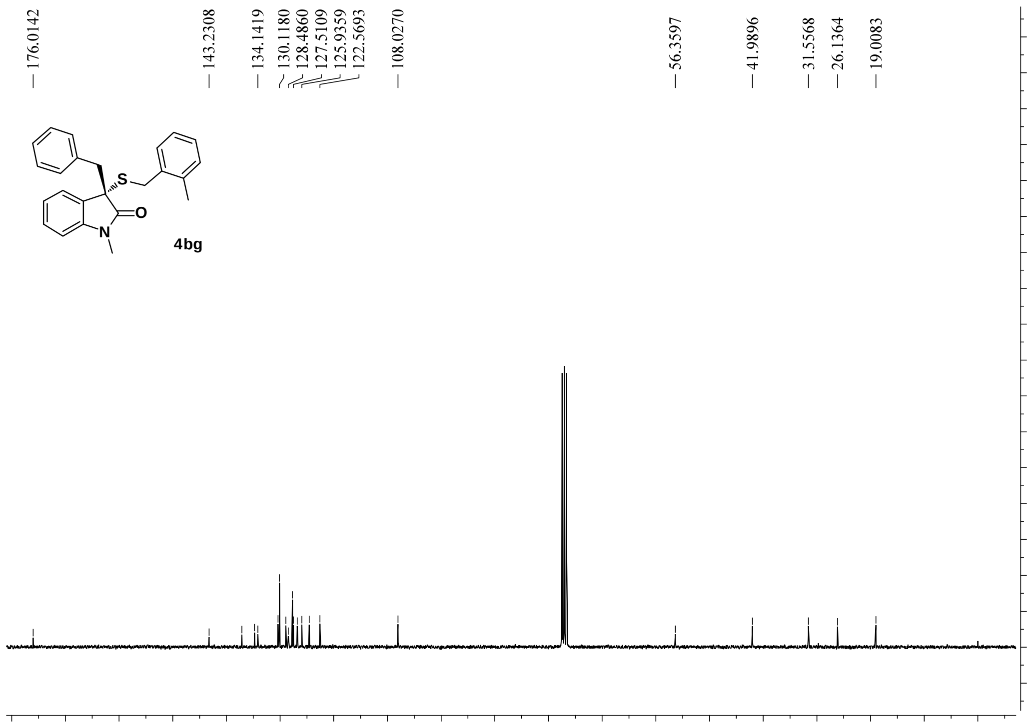
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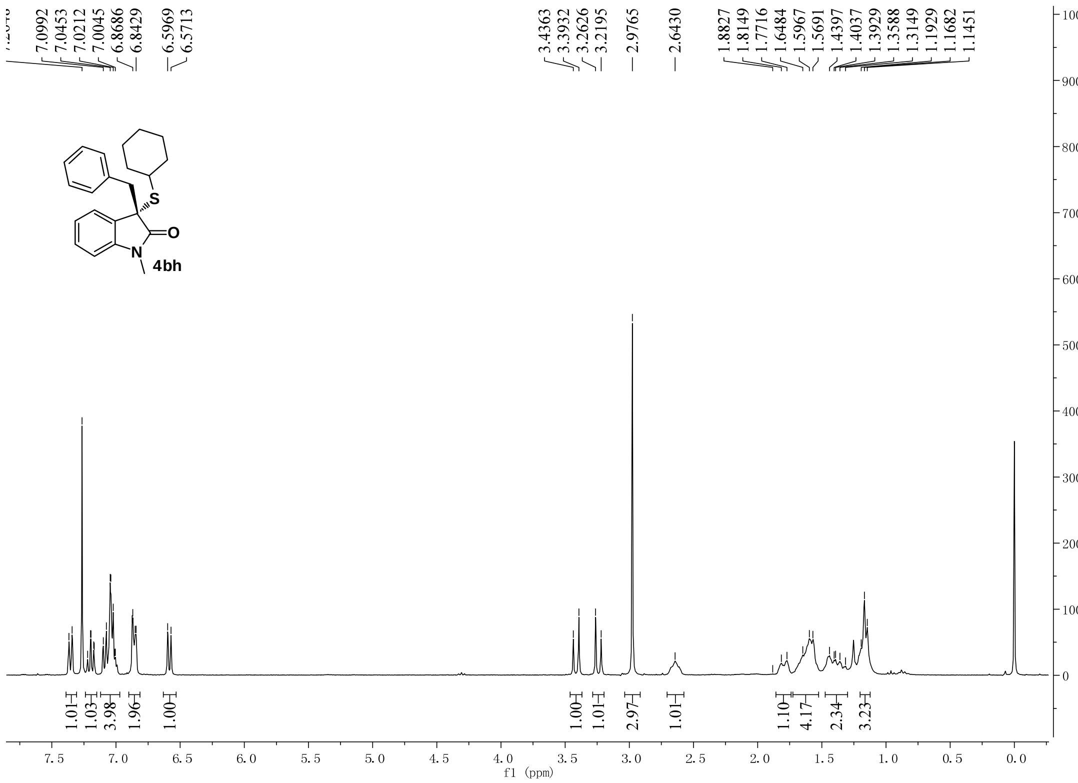
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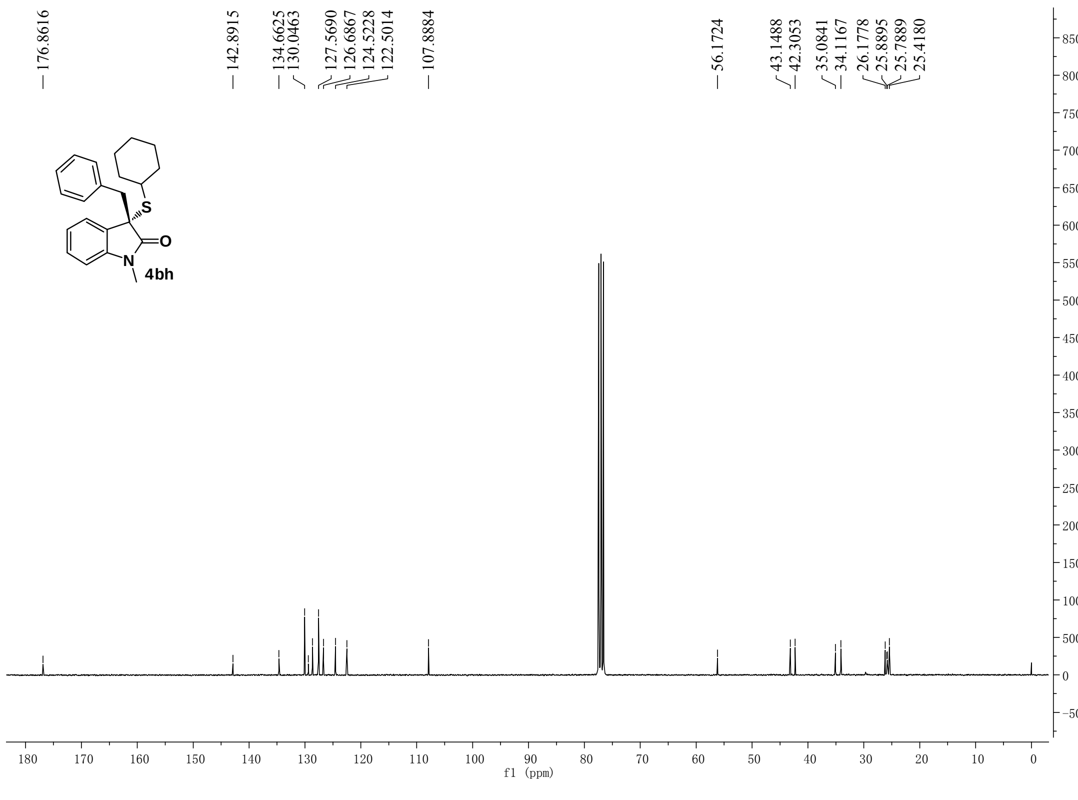
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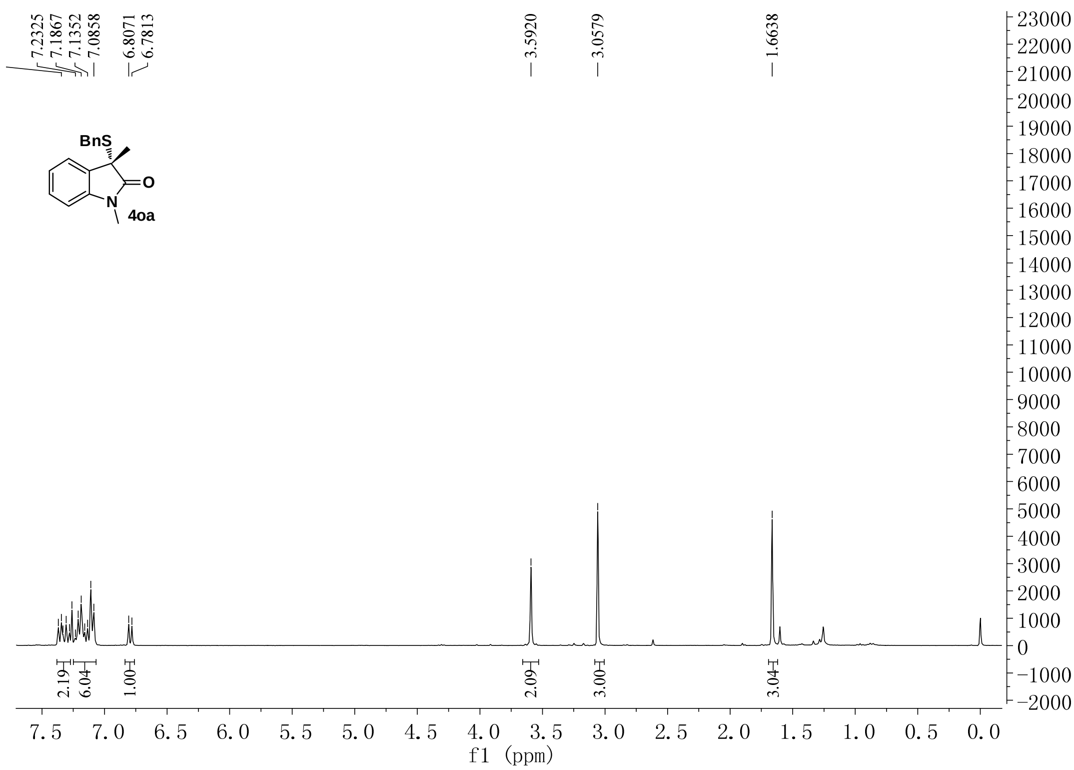

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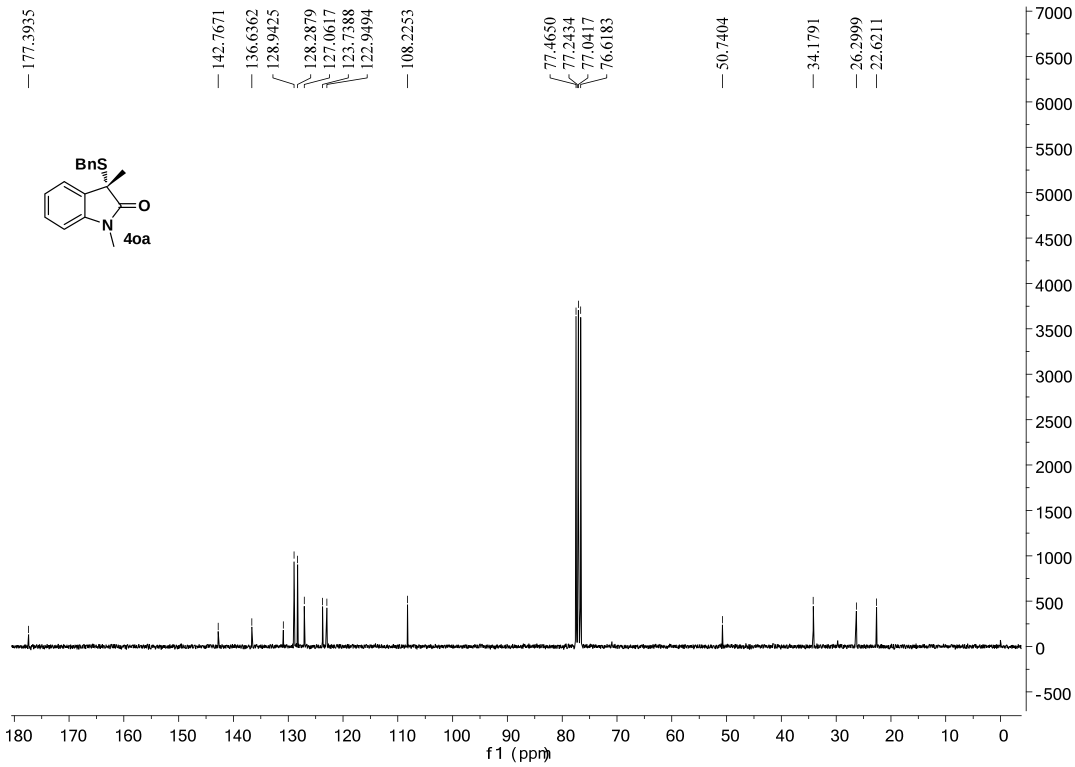

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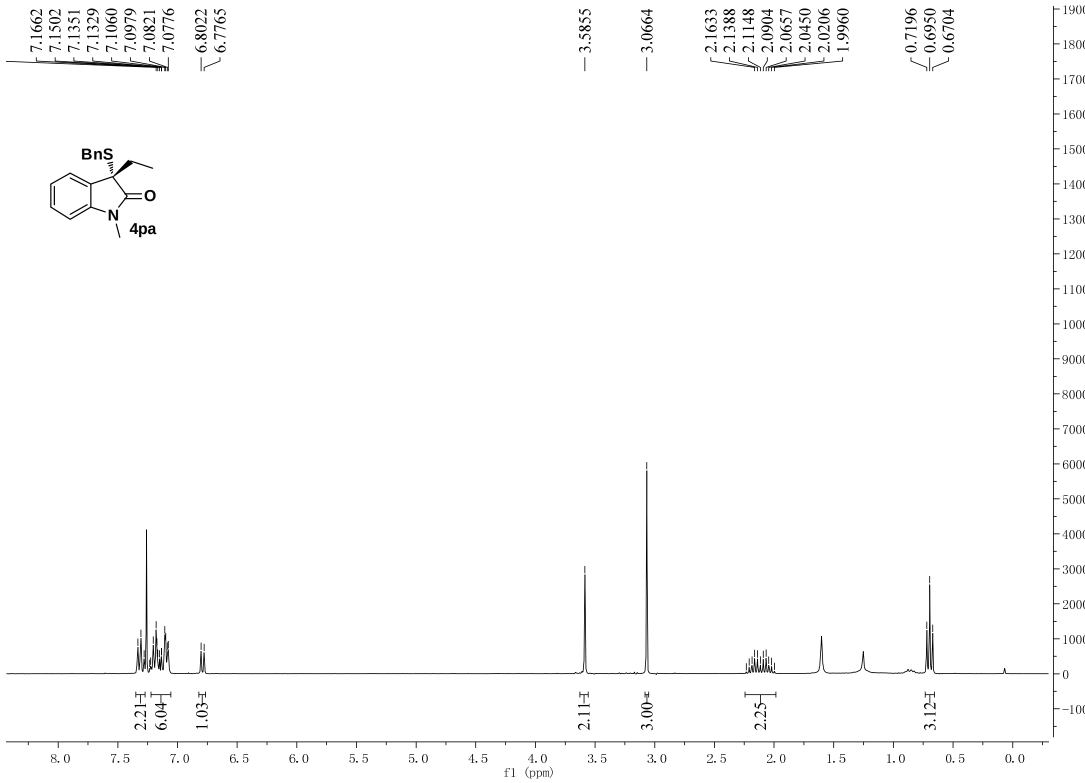
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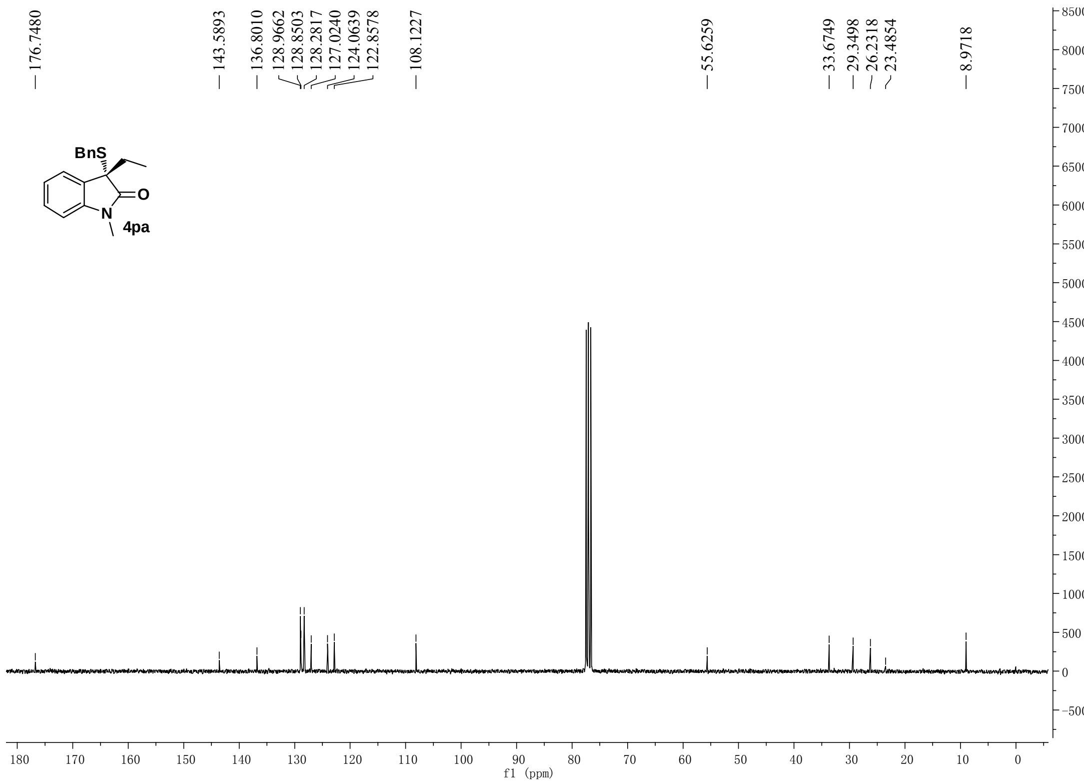
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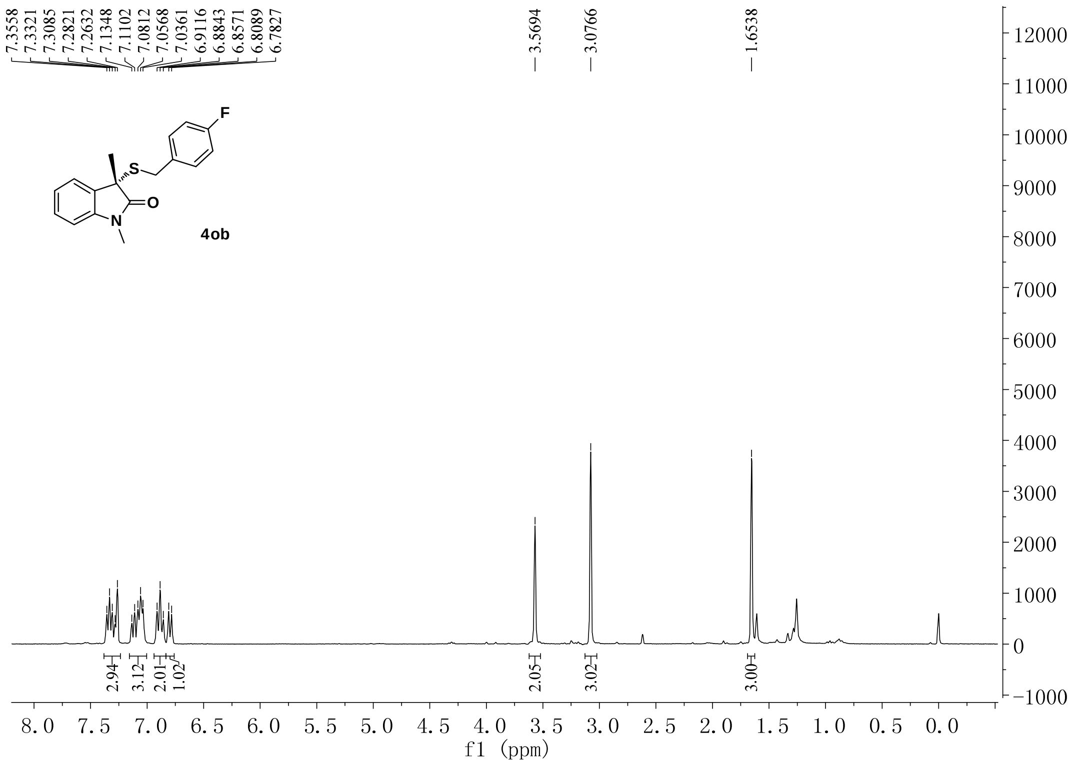
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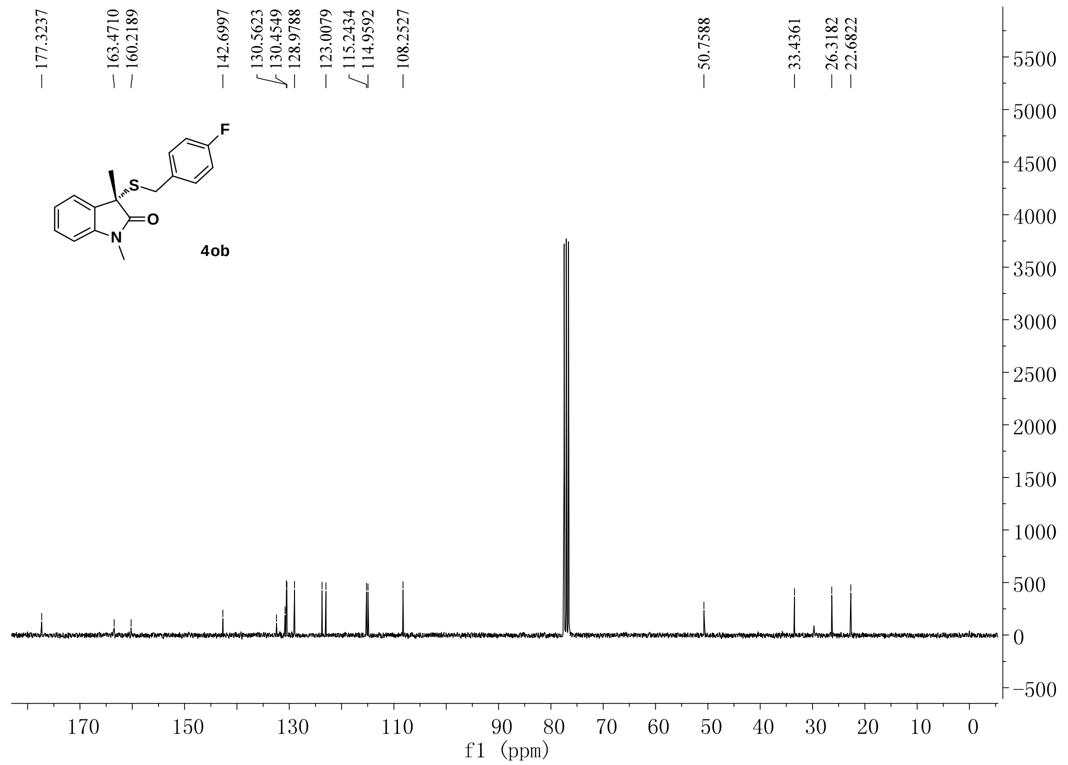
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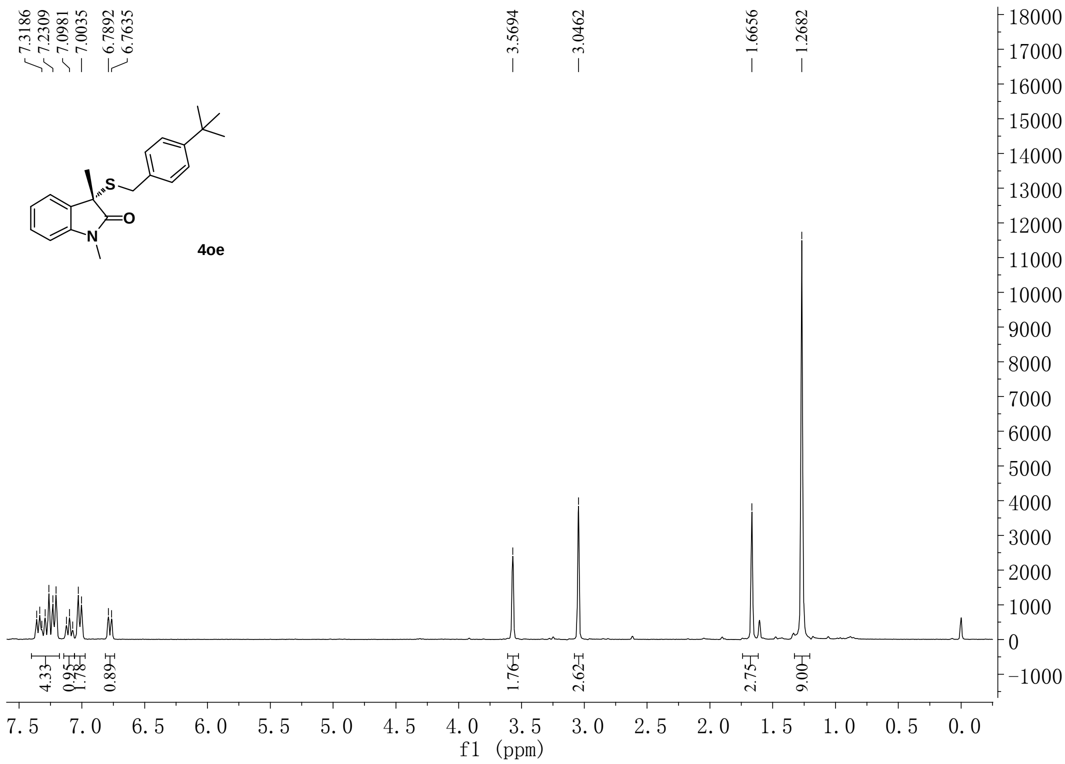
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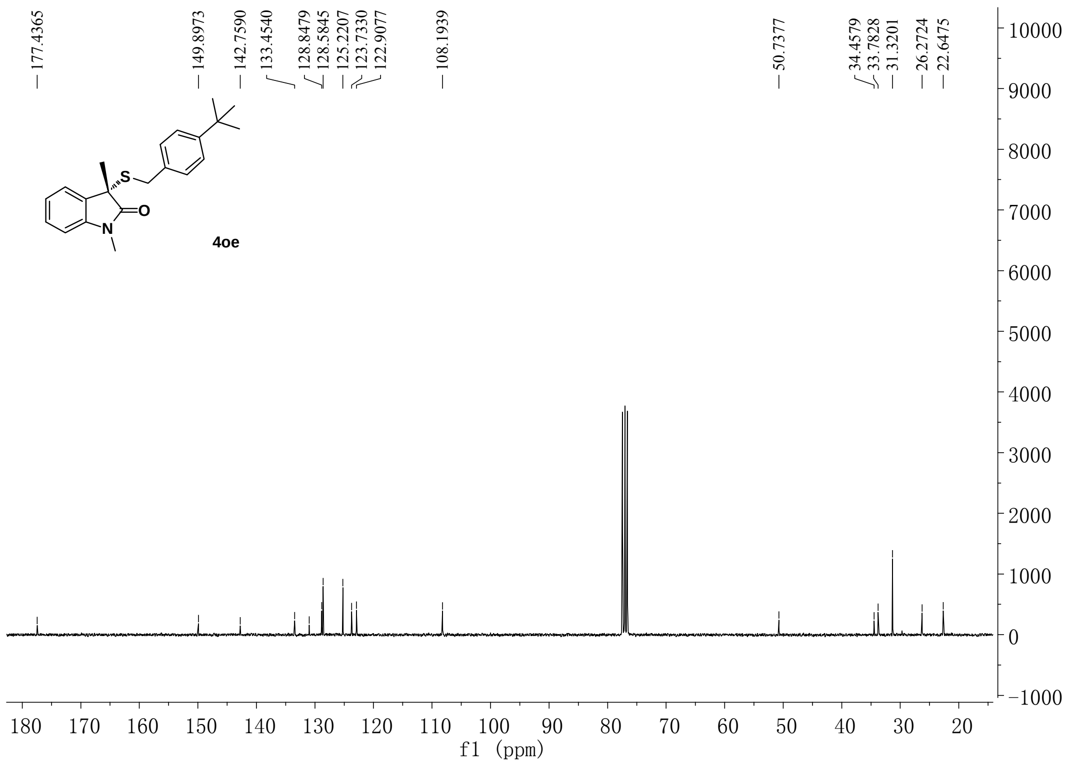
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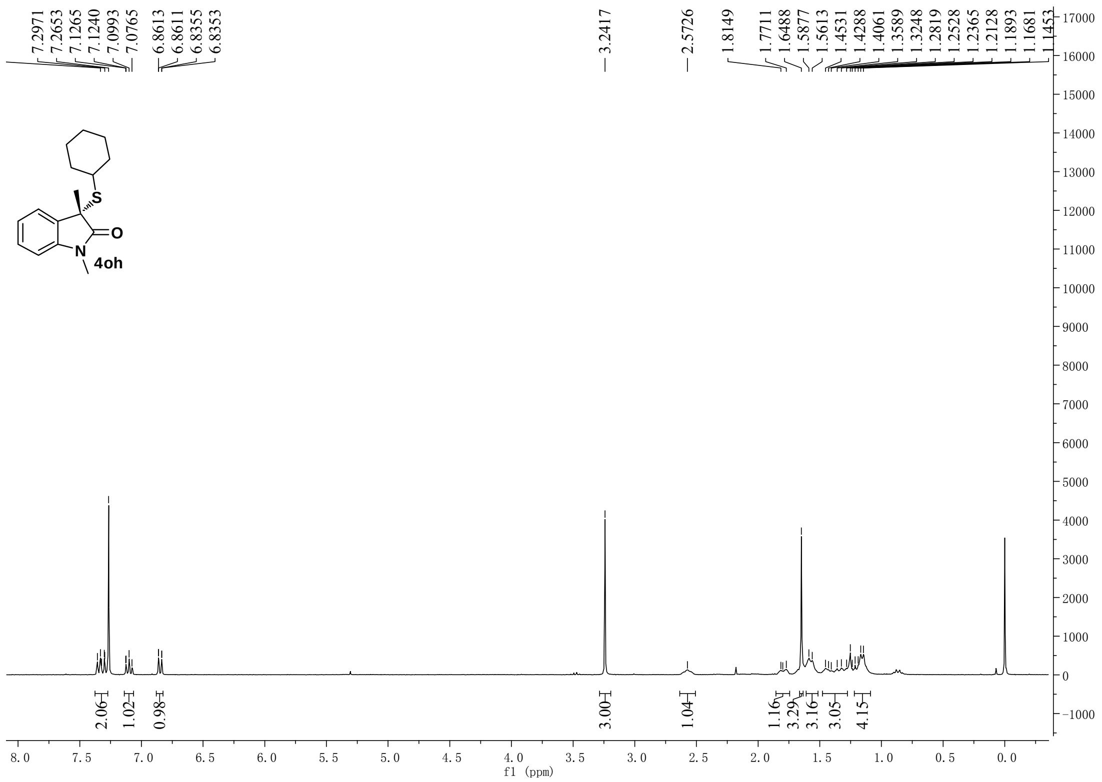
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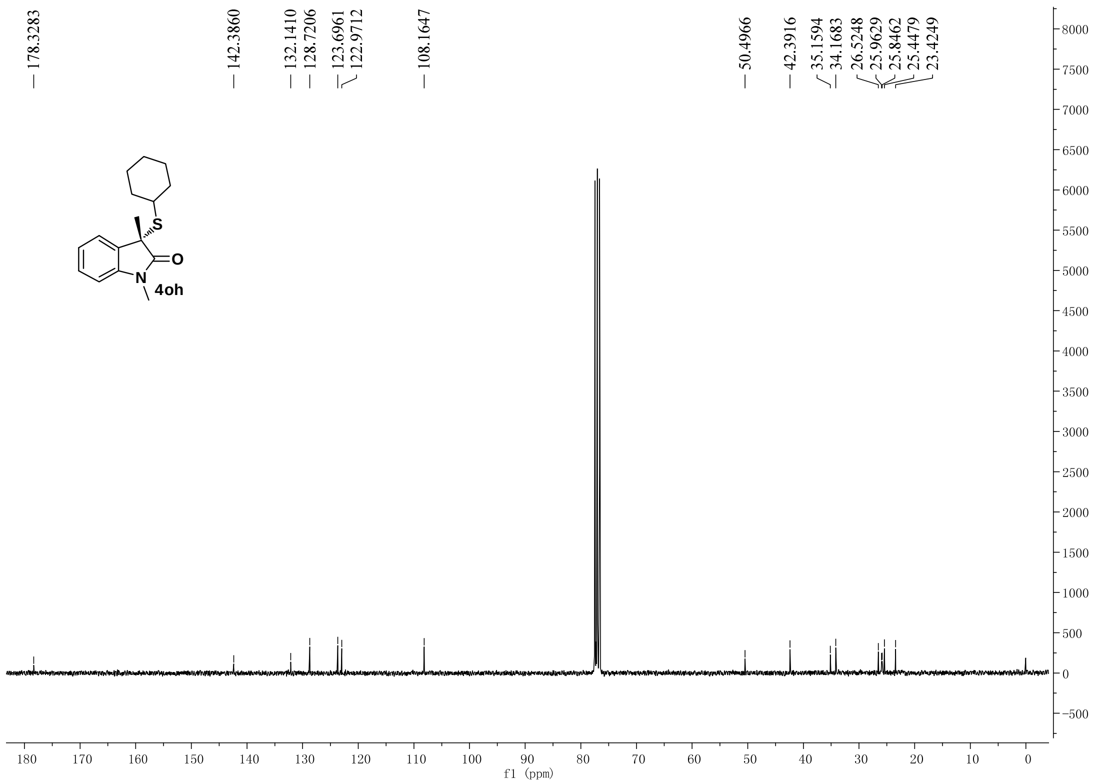
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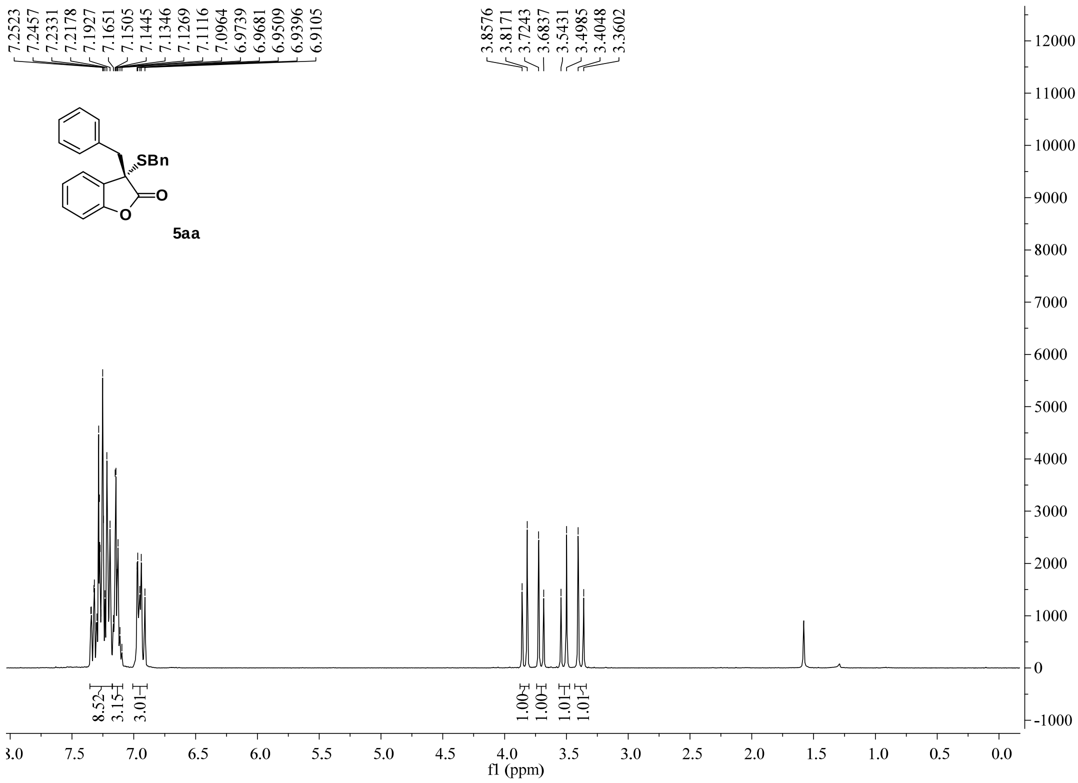

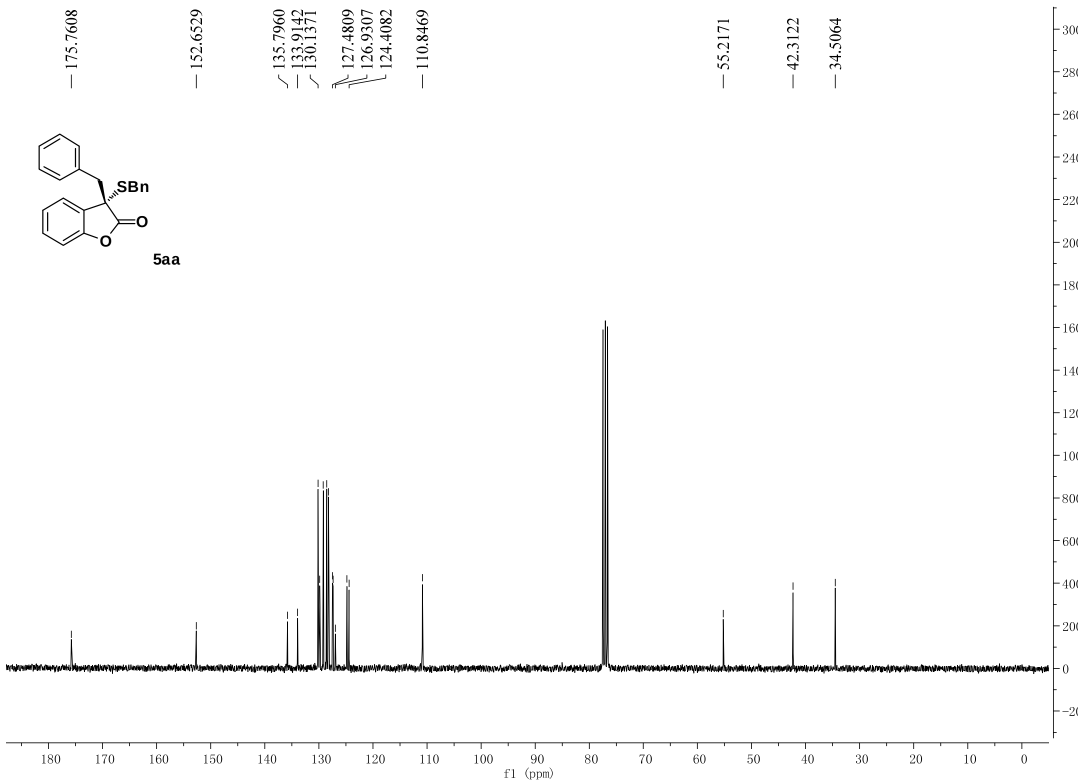

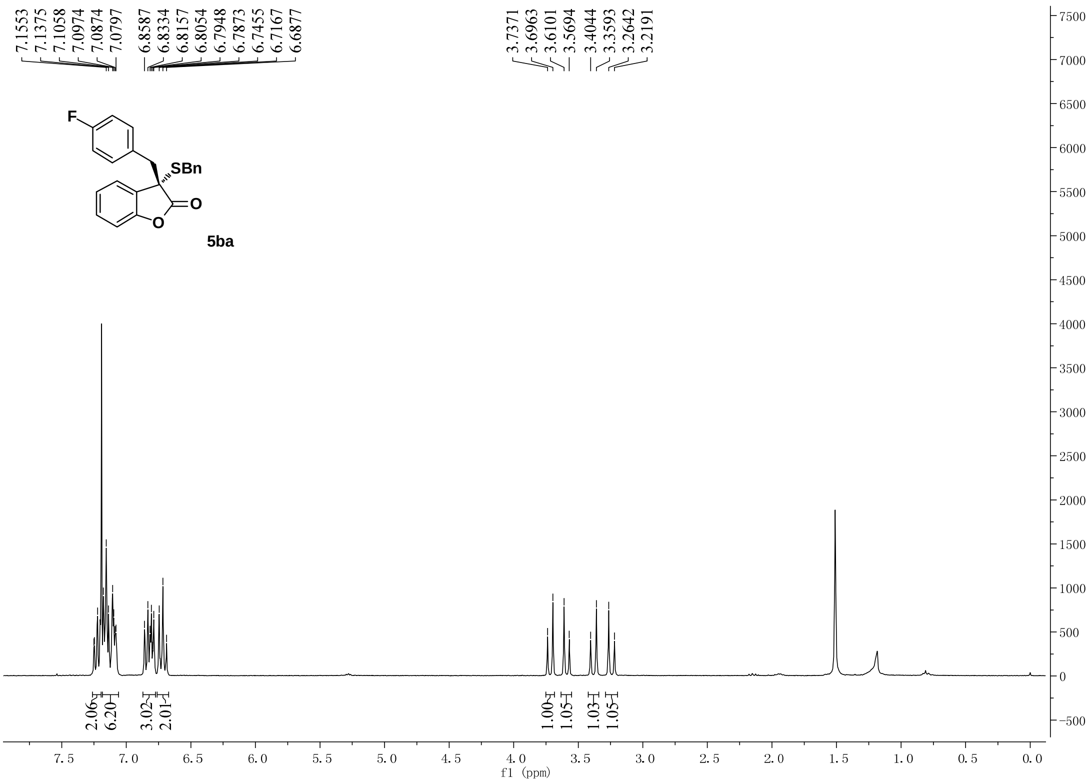
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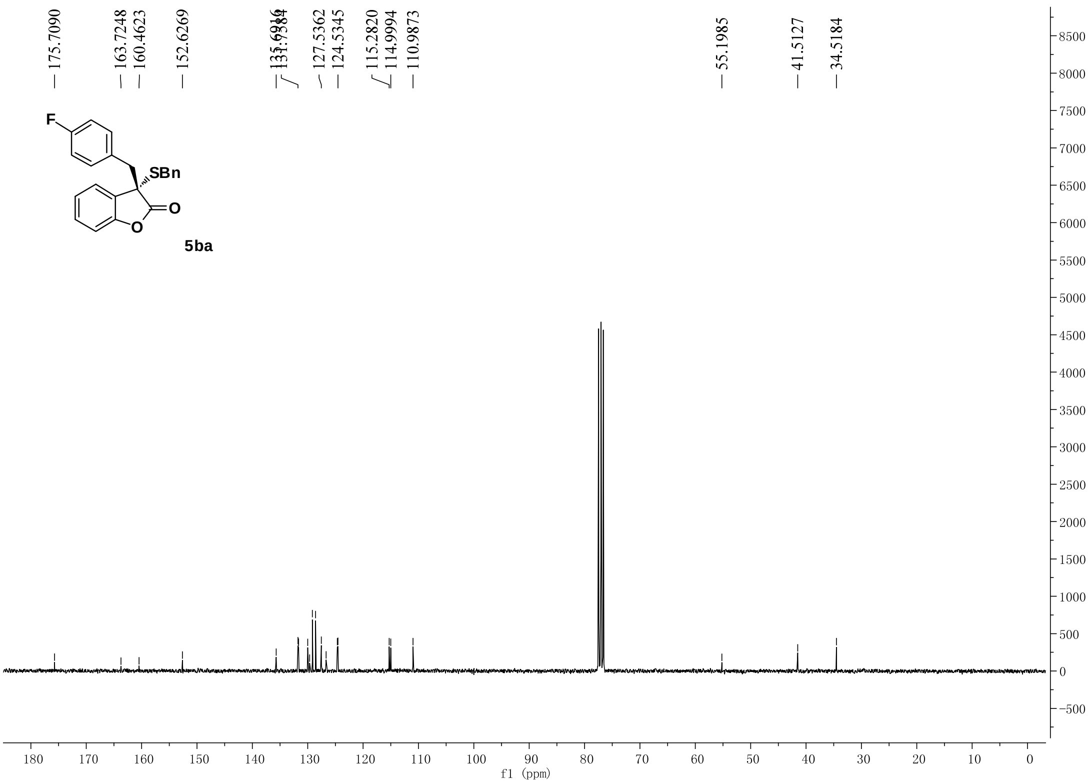
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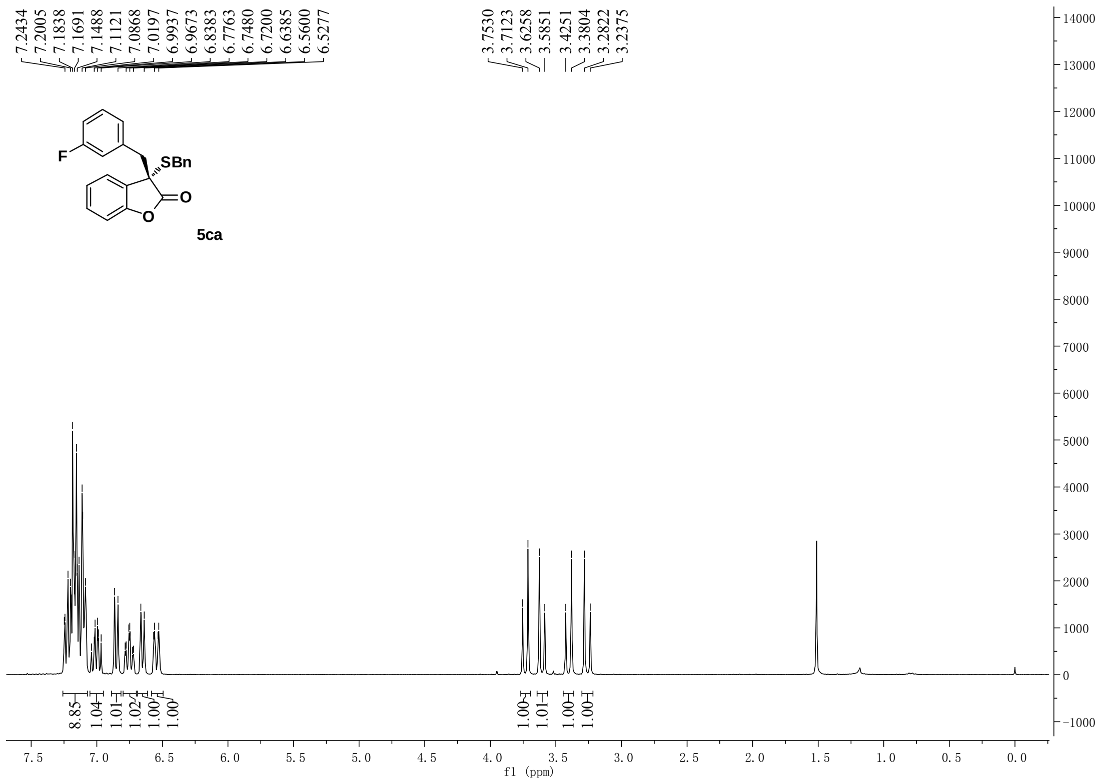
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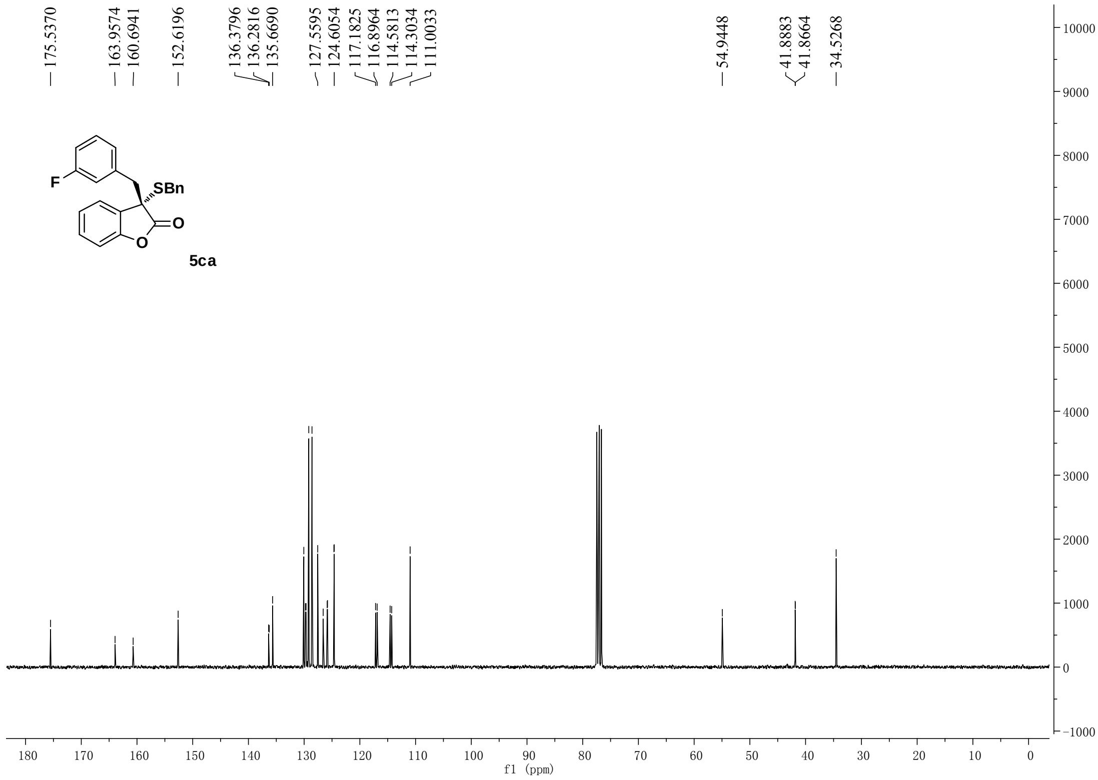
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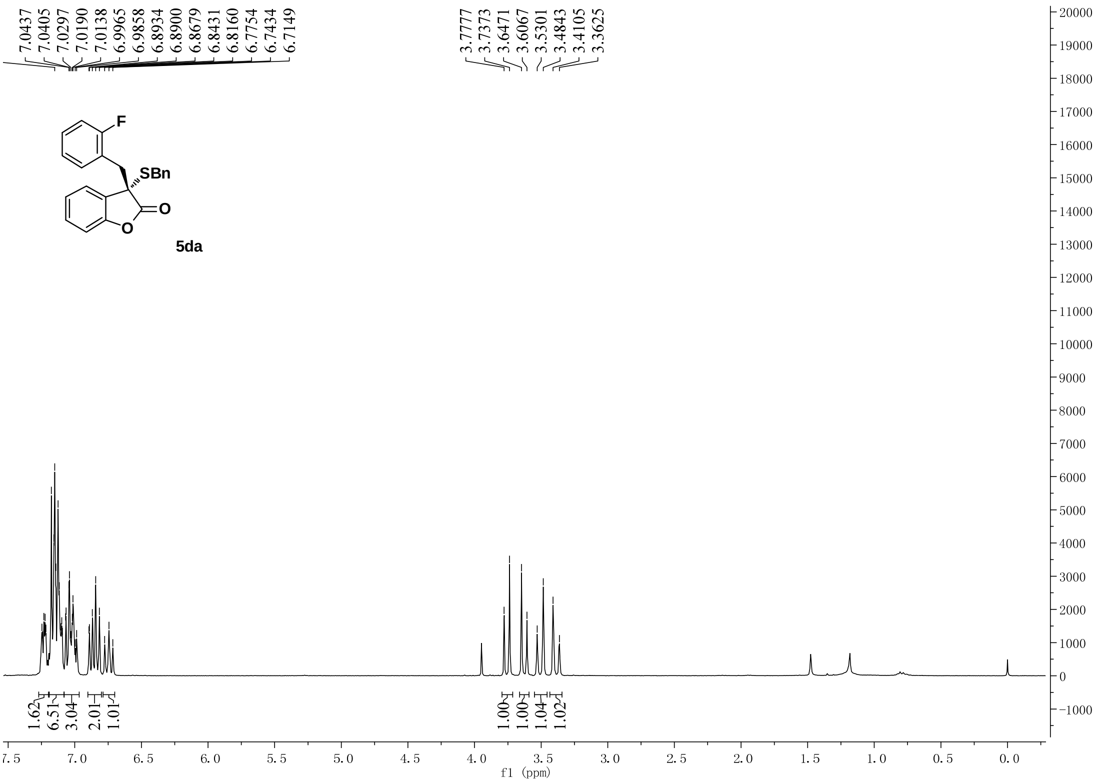
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6.8160
6.7754
6.7434
6.7149
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3.7373
3.6471
3.6067
3.5301
3.4843
3.4105
3.3625
F
SBn
O
O
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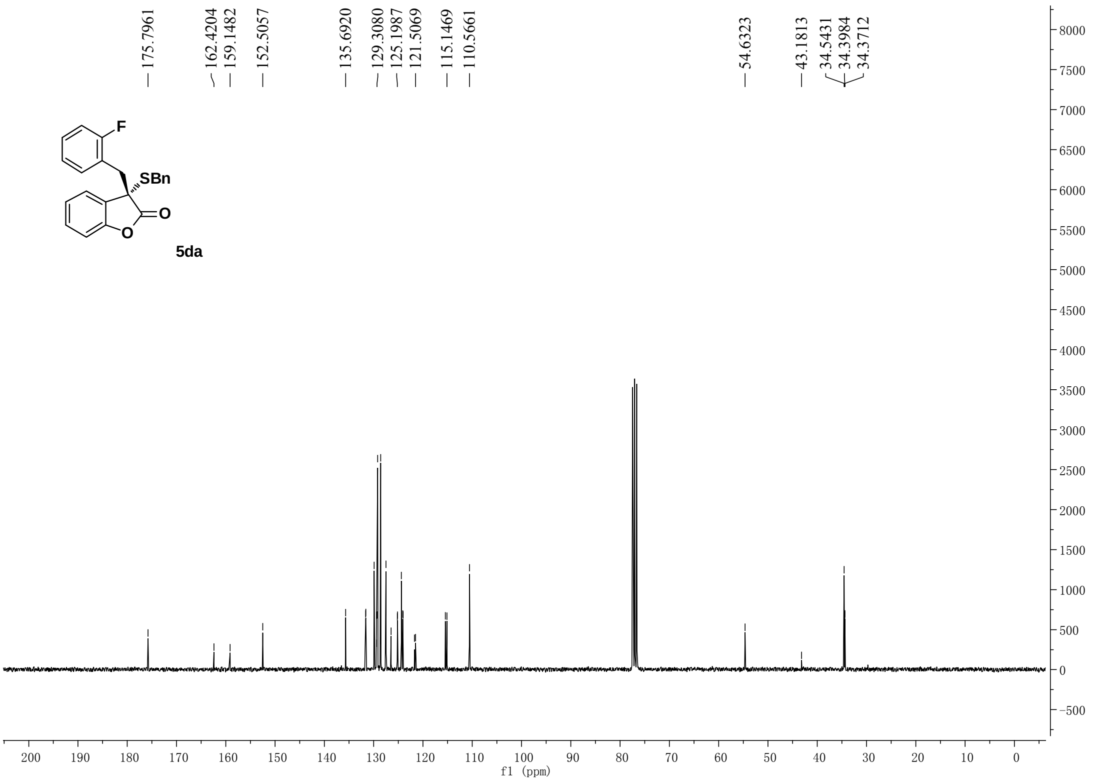
2.01
1.01
1.00
1.00
1.04
1.02
f1 (ppm)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F
SBn
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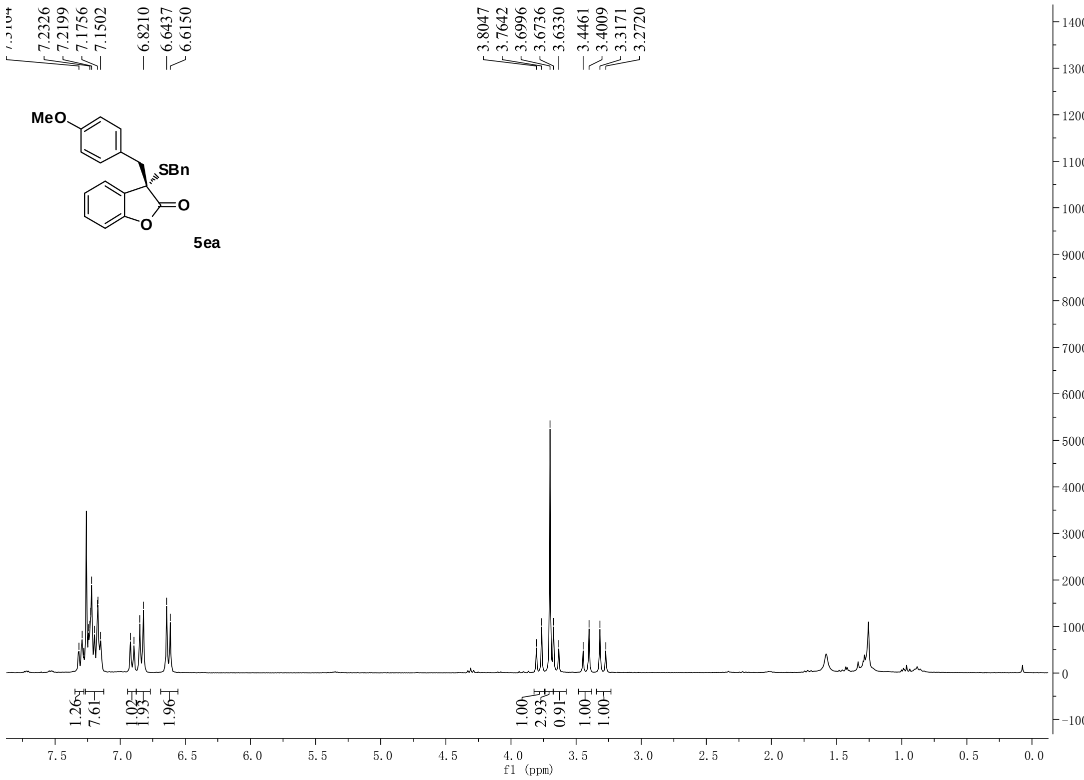
f1 (ppm)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3.6330
3.4461
3.4009
3.3171
3.2720
MeO
SBn
O
O
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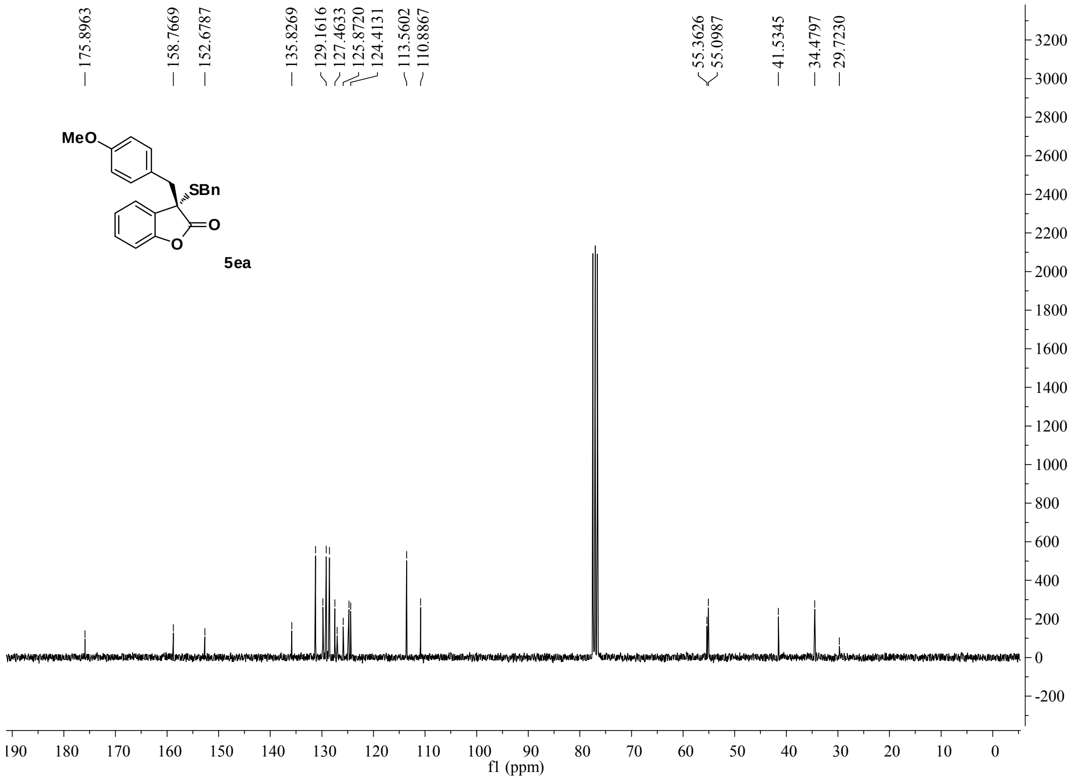
1.00
f1 (ppm)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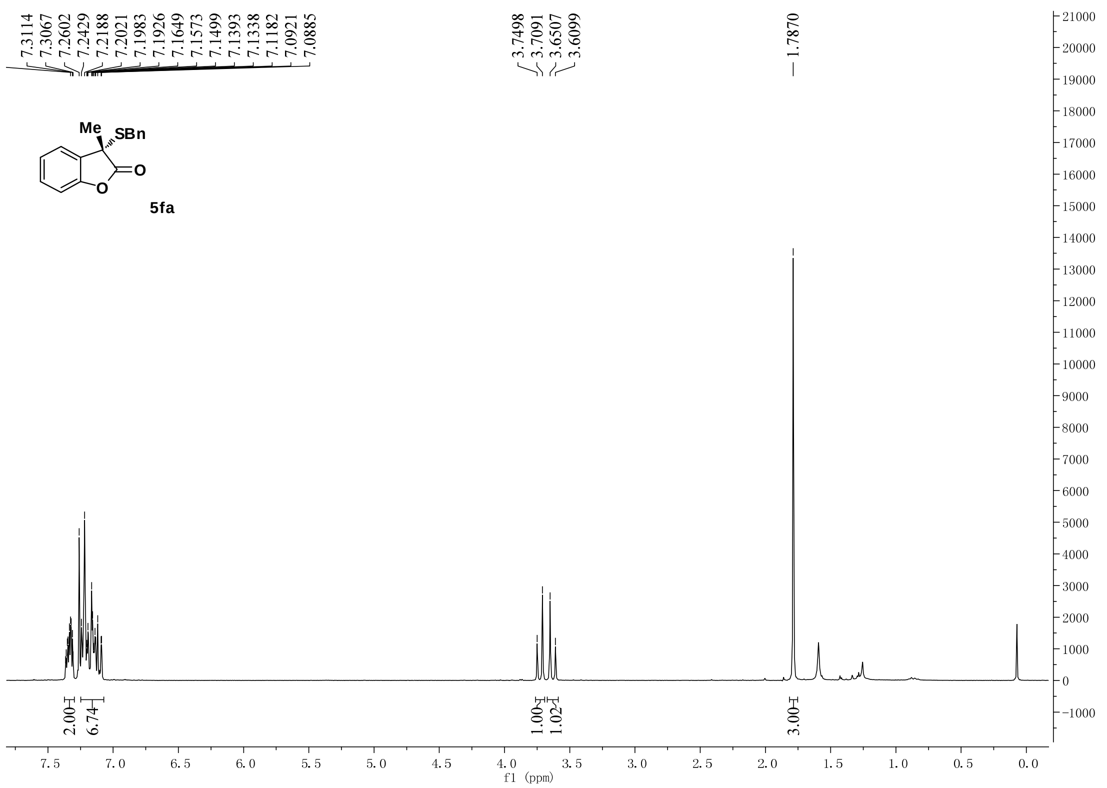
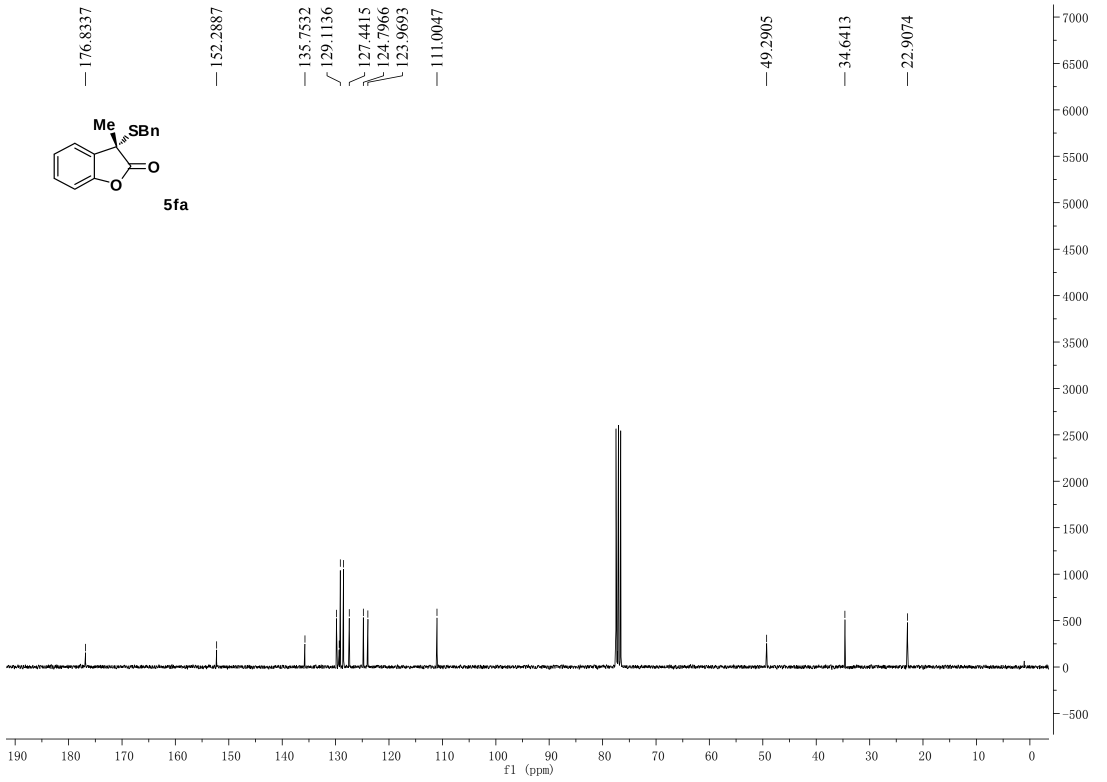
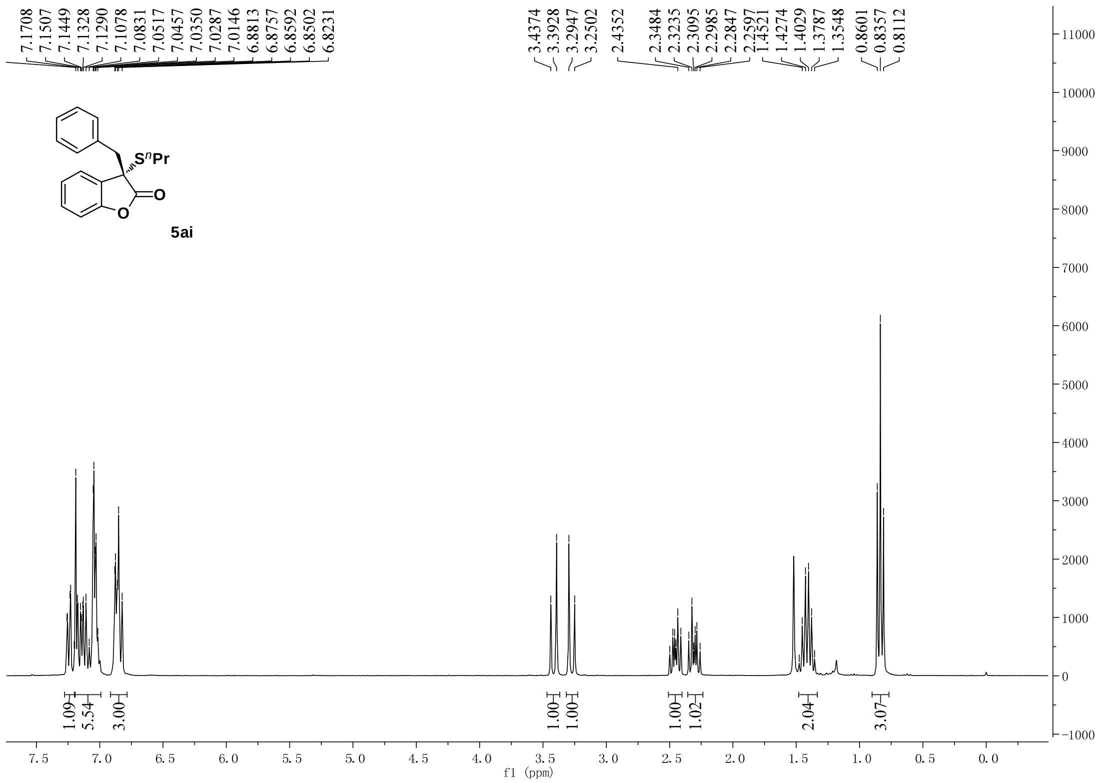
55.0987
41.5345
34.4797
29.7230
MeO
SBn
O
O
5ea
f1 (ppm)



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7.0146
6.8813
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6.8592
6.8502
6.8231
3.4374
3.3928
3.2947
3.2502
2.4352
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2.3095
2.2985
2.2847
2.2597
1.4521
1.4274
1.4029
1.3787
1.3548
0.8601
0.8357
0.8112
S^{n}Pr
O
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5ai
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5.54
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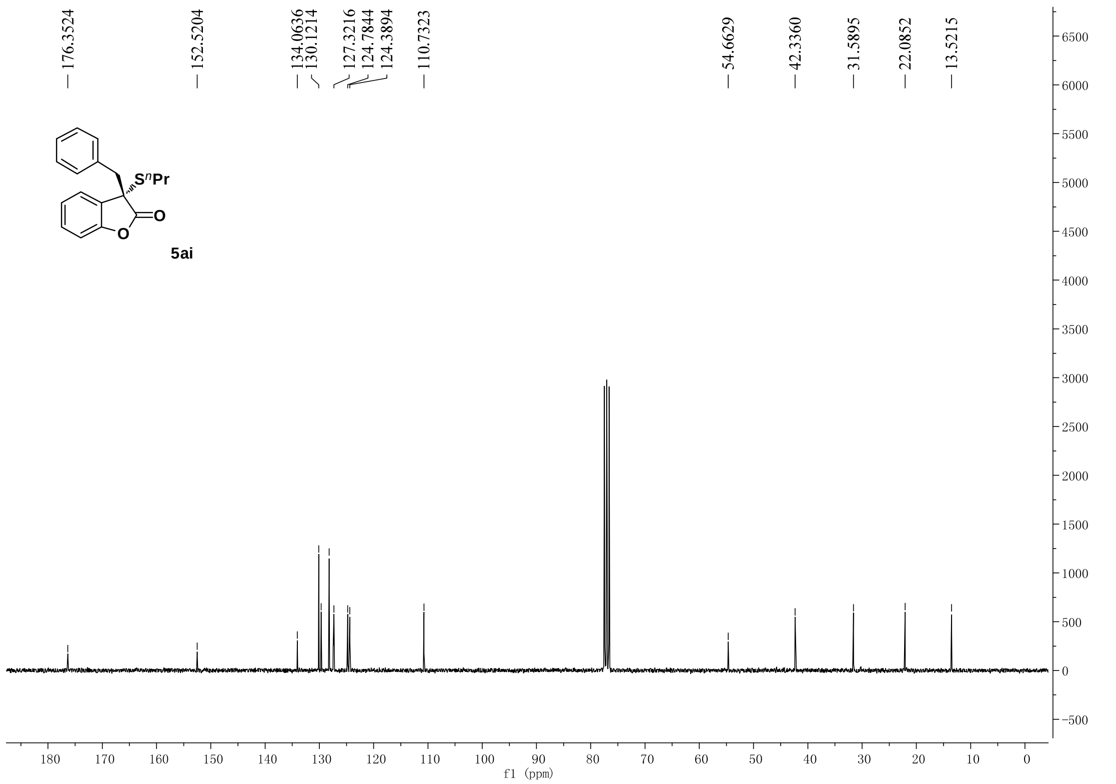
1.00
1.00
1.00
1.02
2.04
3.07
f1 (ppm)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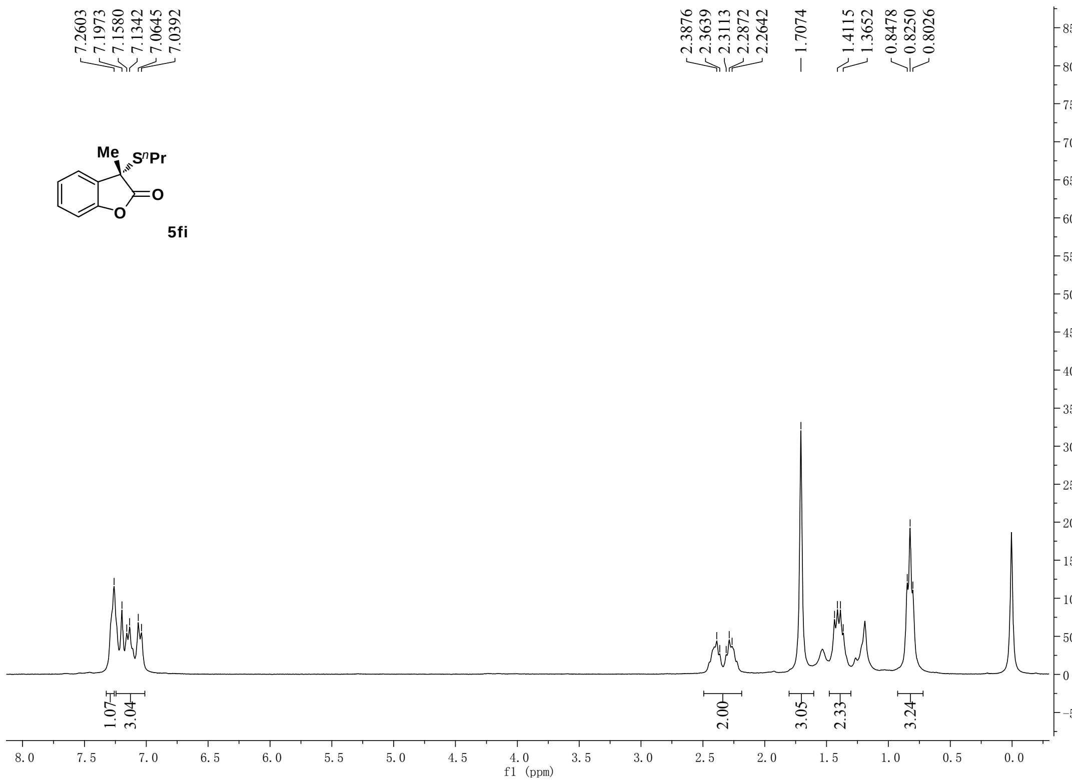
S^{n}Pr
O
O
5ai
f1 (ppm)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2.2642
1.7074
1.4115
1.3652
0.8478
0.8250
0.8026
Me
S^{n}Pr
O
O
5fi
1.07
3.04
2.00
3.05
2.33
3.24
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
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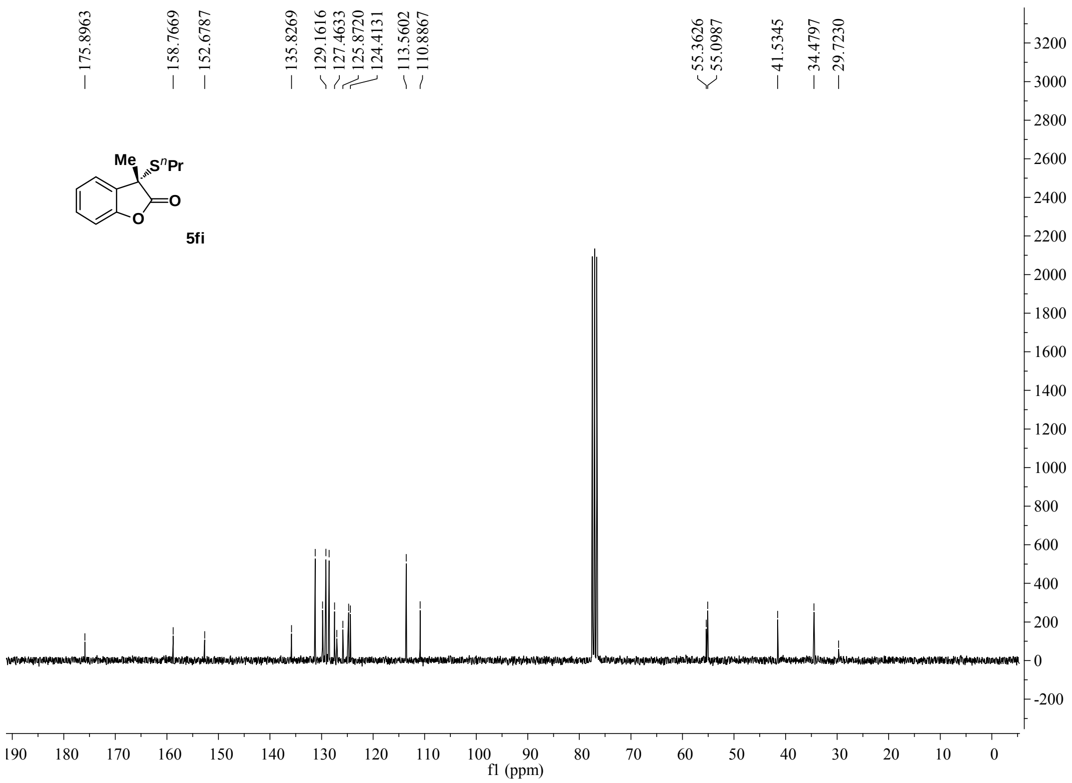
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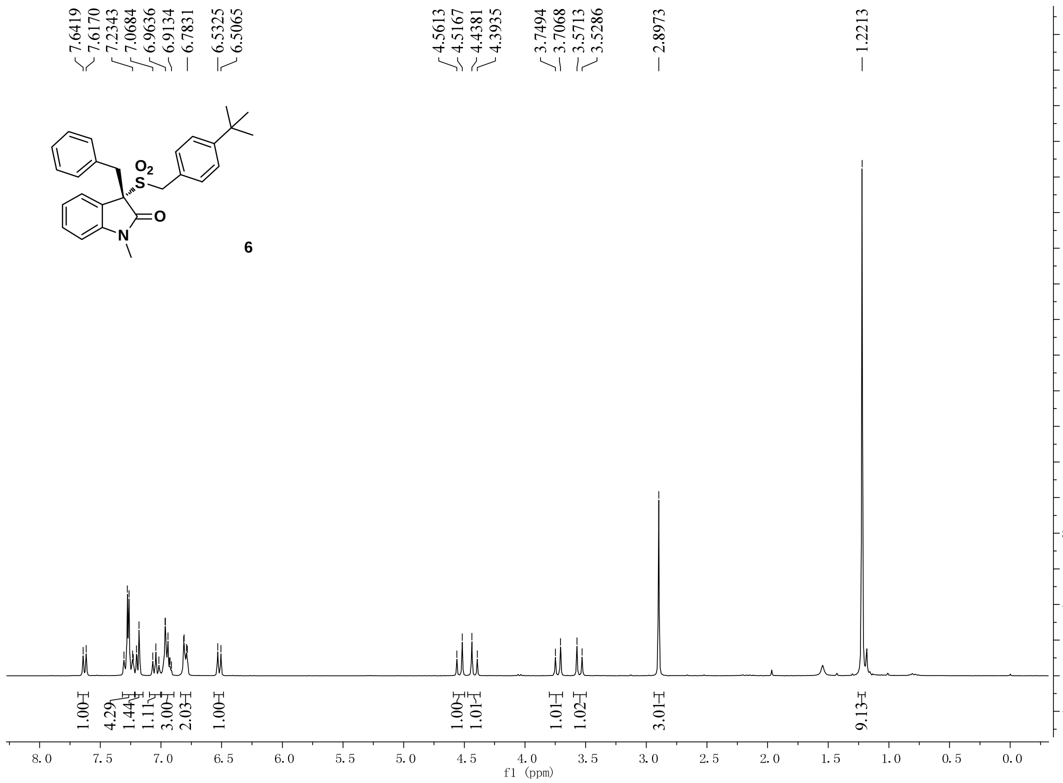
55.3626
55.0987
41.5345
34.4797
29.7230
Me
S^{n}Pr
O
O
5fi
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200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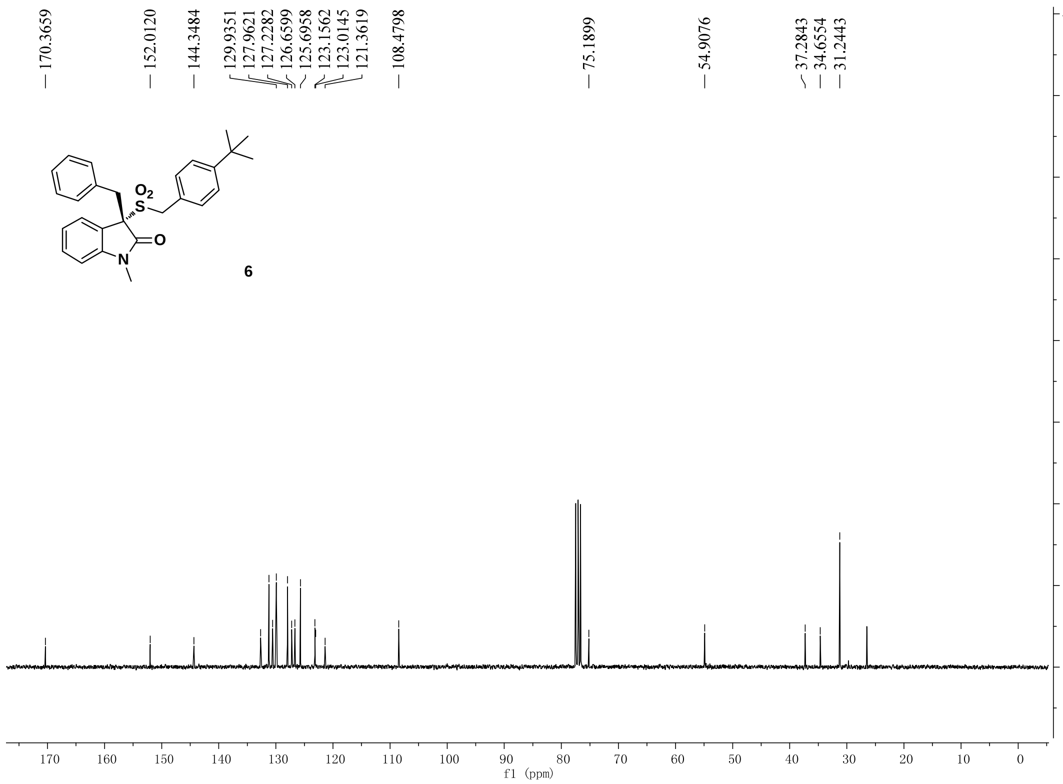
4.4381
4.3935
3.7494
3.7068
3.5713
3.5286
2.8973
1.2213
O_2
S
O
N
6
1.00
4.29
1.44
1.11
3.00
2.03
1.00
1.00
1.01
1.01
1.02
3.01
9.13
f1 (ppm)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54.9076
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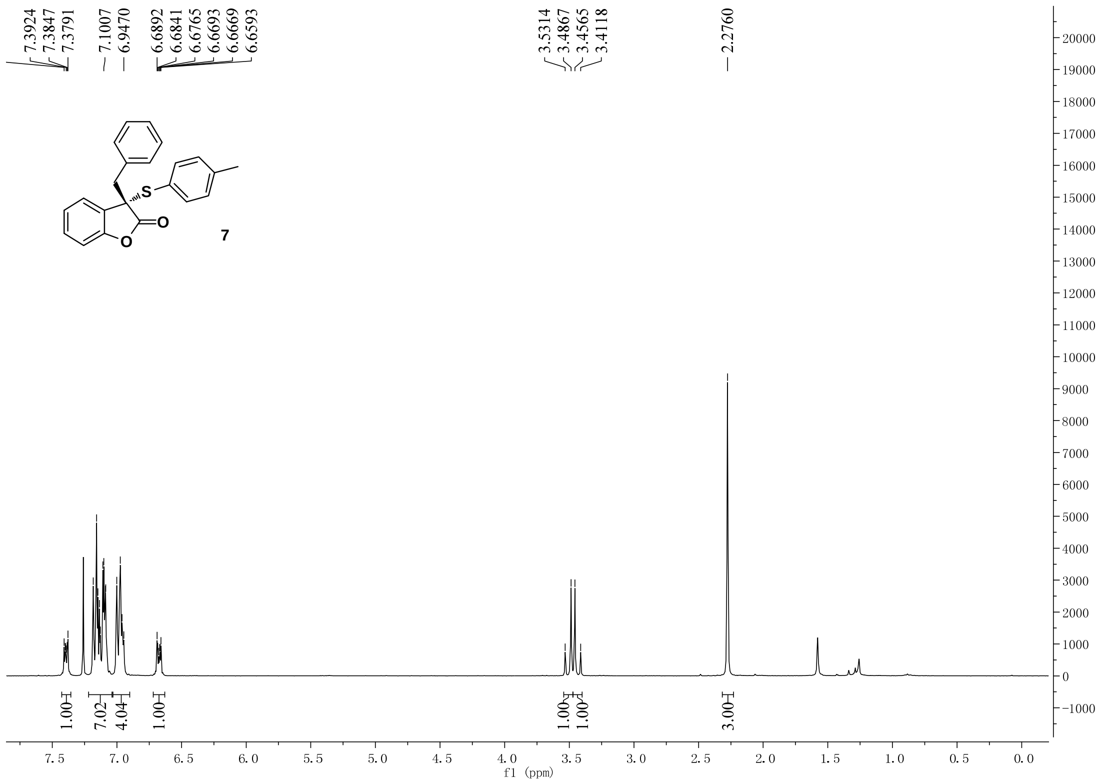
34.6554
31.2443
O_2
S
O
N
6
f1 (ppm)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3.5314
3.4867
3.4565
3.4118
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O
O
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7.02
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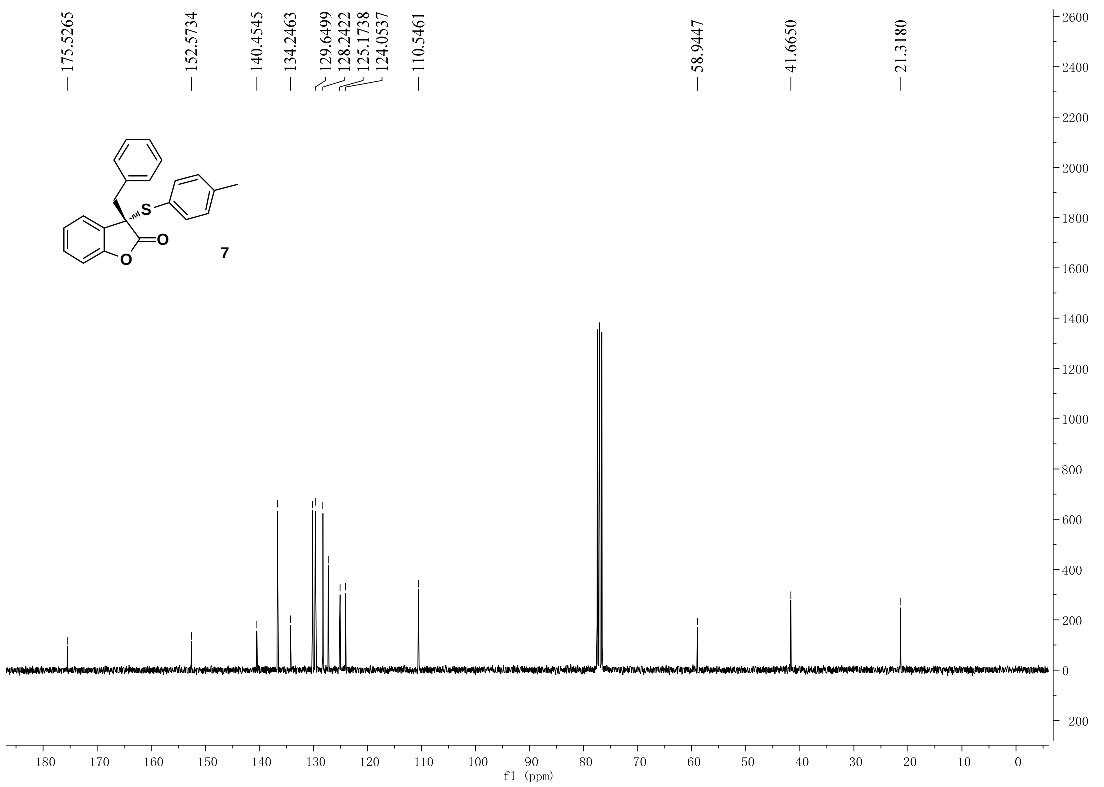
1.00
1.00
3.00
f1 (ppm)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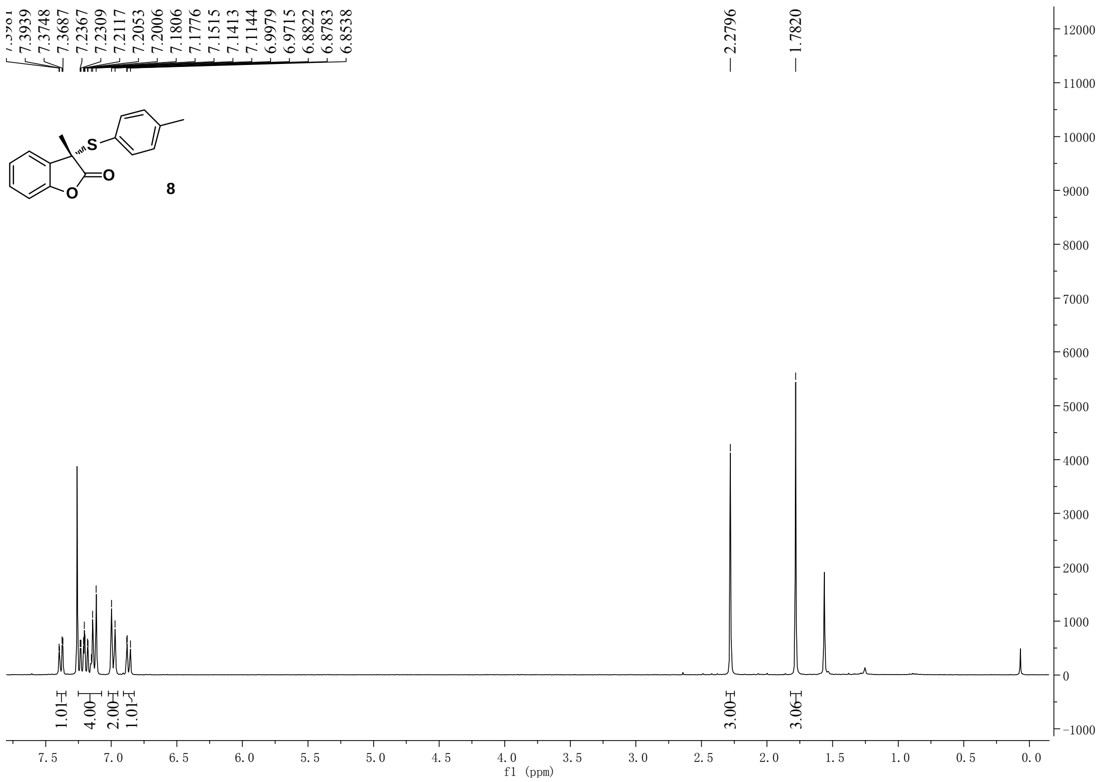
21.3180
S
O
O
7
f1 (ppm)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7.1413
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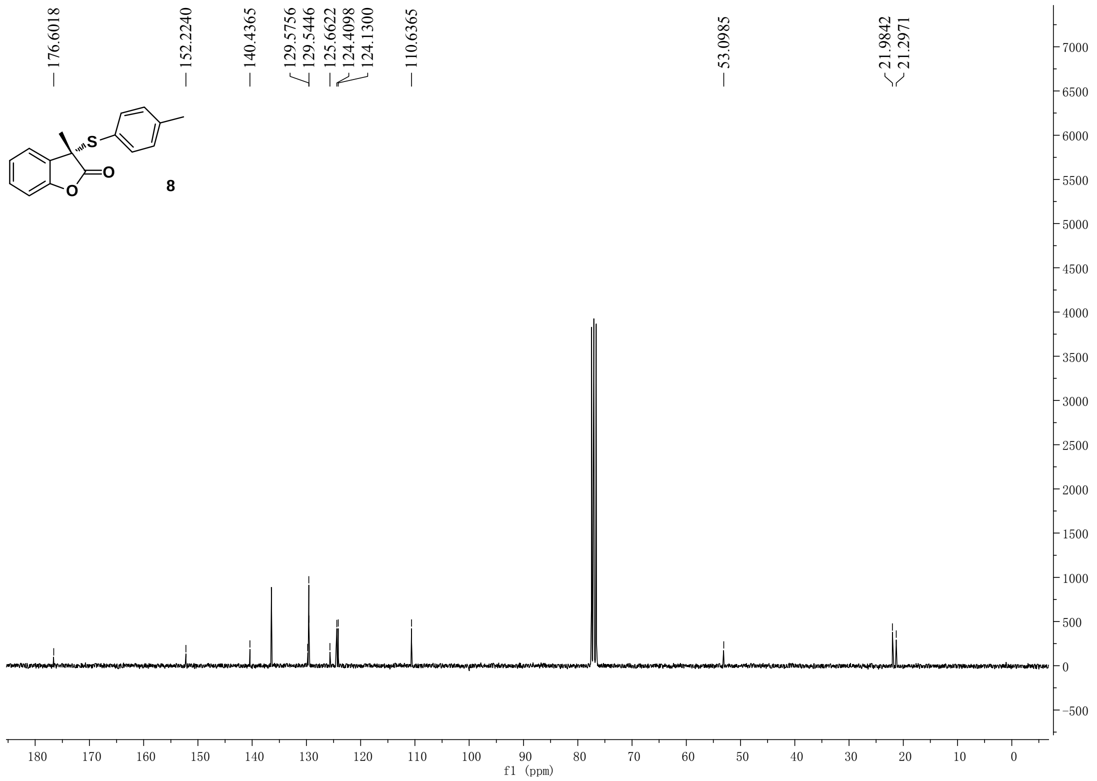
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8
f1 (ppm)