Supporting Information

Chiral iodine-catalyzed asymmetric oxyamination of unactivated olefins

Gui-Ping Han,[a] Jia-Qi Wang,[a] Jing-Feng Zhao,[a] Dao-Yong Zhu,[b] Lu-Wen Zhang,[a] and Wei He*,[a]

[a]Department of Chemistry, School of Pharmacy, Air Force Medical University, 169 Changle West Road, Xi'an 710032, P. R. China. E-mail: weihechem@fmmu.edu.cn.
[b]School of Pharmacy, Lanzhou University, Lanzhou 730000, China.

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1. General Information

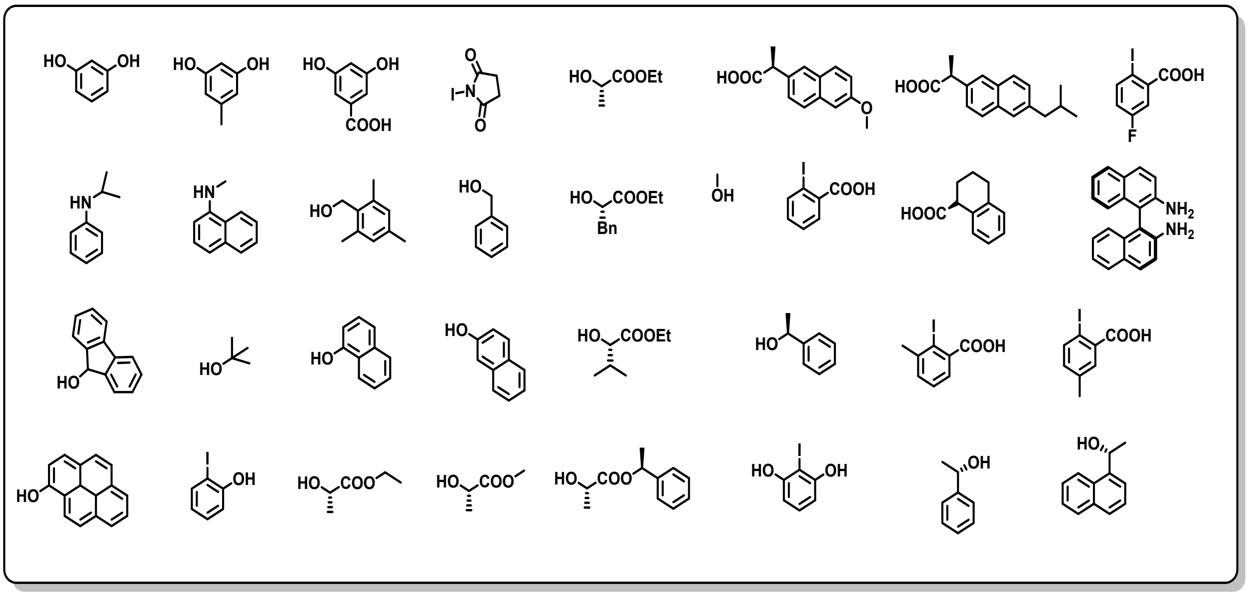
Commercially available reagents were used as received without further purification. All reactions were carried out in 10 mL sealed tubes filled with nitrogen, using 365-370 nm LED purchased from SHANGHAI 3S TECHNOLOGY for light irradiation. Reactions were monitored by thin layer chromatography (TLC) using glass 0.25 mm silica gel plates with visualization under UV light (254 nm and 365 nm), or $KMnO_4$ staining solution as chromogenic agents. Organic solutions were concentrated under reduced pressure on a Heidolph rotary evaporator. Chromatographic purification of products was accomplished by flash chromatography on Merck Silica Gel 60F-254 (200-400 mesh). TBADT (tetrabutylammonium decatungstate) was synthesized according to the reported methods[1]. All of allyl sulfones **2** were synthesized according to the literatures[2], and all of the spectra were in full accordance with the data in the literatures.

^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance III-400 in solvents as indicated. Chemical shift are reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: ^1H-NMR: δ 7.26; ^{13}C-NMR: δ 77.0). Data for ^1H NMR and ^{19}F are reported as follows: chemical shift referred to TMS (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad singlet, dd = doublet of doublets, td = triplet of doublets etc.), coupling constants (*J*) are reported in units of hertz (Hz). Data for ^{13}C NMR are reported in terms of chemical shift. High-resolution mass spectra (HRMS) were performed on an AB Sciex $X500_R$ QTOF spectrometer.

2. Starting Materials

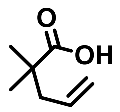
2.1Raw materials for preparing catalysts

Directly available starting materials for the preparation of chiral catalysts：

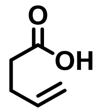


2.2Raw materials for preparing alkenyl carboxylic acid substrates

Alkyl carboxylic acid raw materials can be purchased directly:



3h



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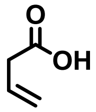

3v

Raw materials for preparing alkenyl carboxylic acid substrates：

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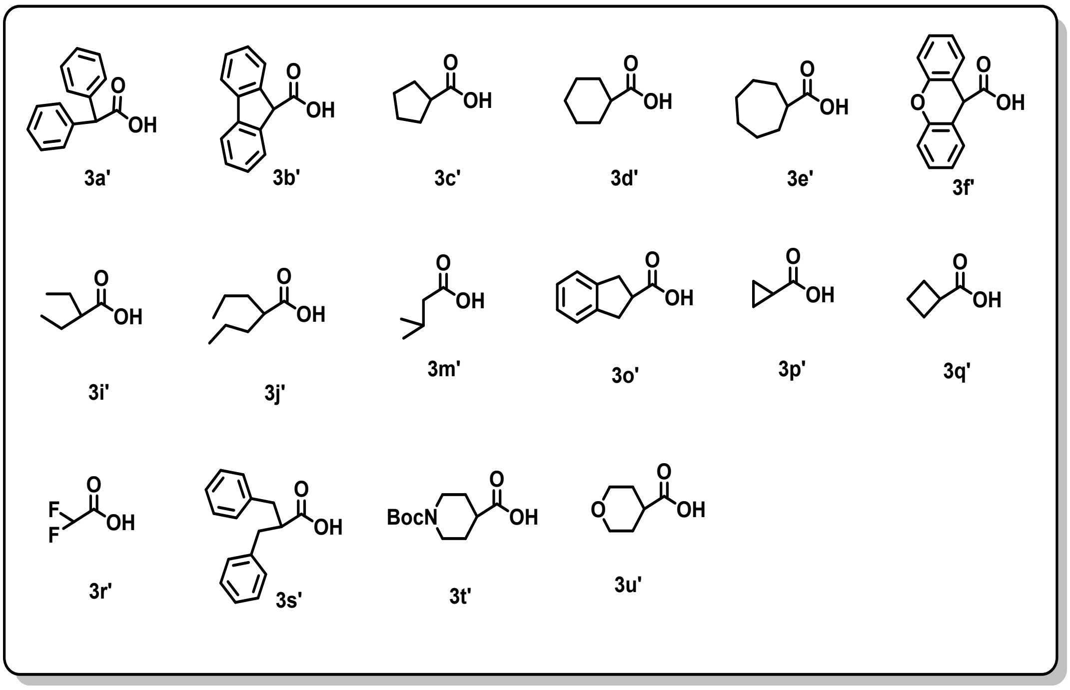
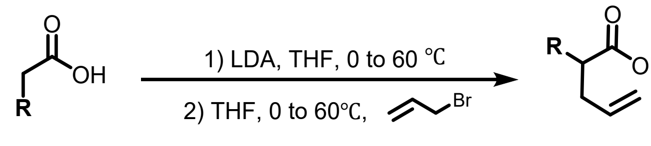
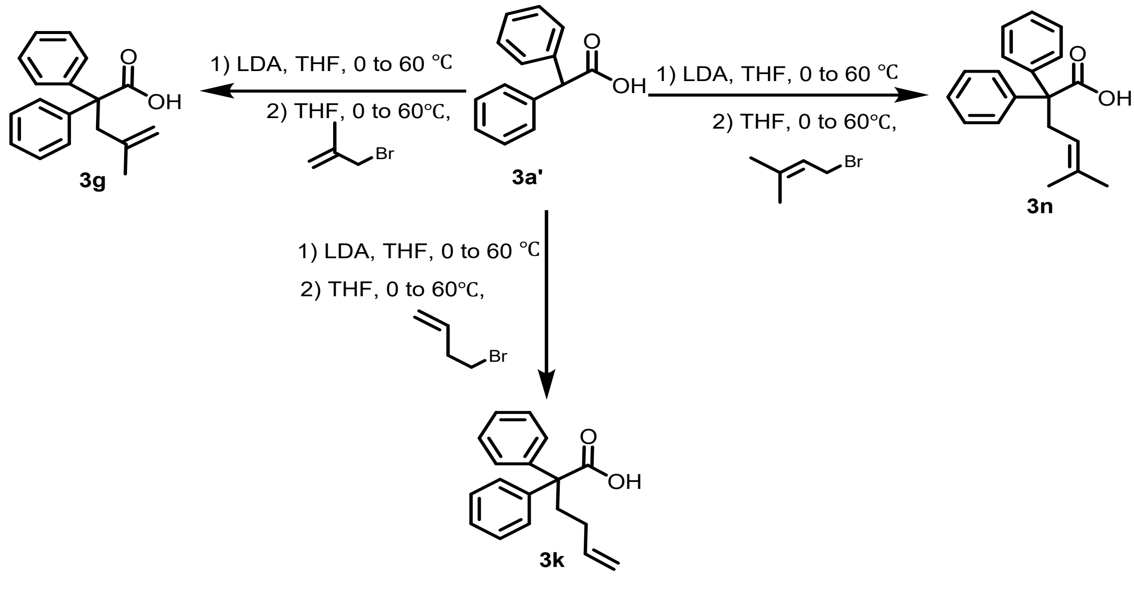




3a'-3f'、3i'-3j'、3m'、3o'-3u'

3a-3f、3i-3j、3m、3o-3u

Method 2：



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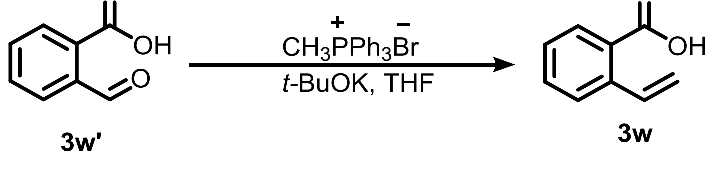
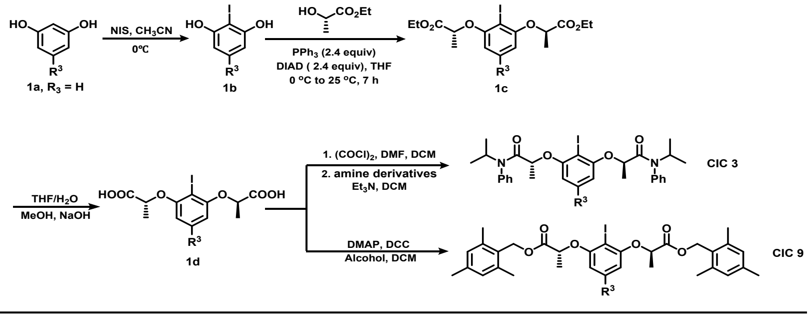


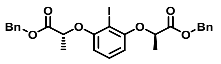
3w'

3w

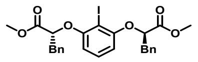
3. Synthesis of Chiral Aryl Iodine Catalysts

3.1 Synthesis of Chiral Aryl Iodine Catalysts Based on Bilateral Lactic Acid Structure





CIC 1



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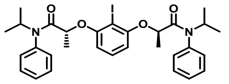
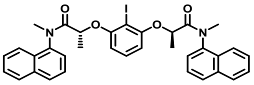


CIC 3



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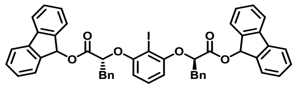

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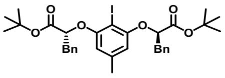

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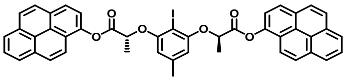

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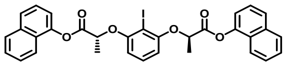
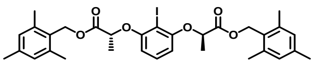


CIC 8



CIC 9

3.1.1 Synthesis of Chiral aryl iodine catalyst（CIC 3）:

Synthesis of 2-iodoresorcinol（1b）:

Resorcinol (1.65 g, 15 mmol, 1.0 equiv.) was added to a 100 mL reaction flask, 30 mL of CHCN was added to fully dissolve, and stirred in an ice water bath at 0 ℃ for 10 minutes. Subsequently, N-iodosuccinimide (3.7g, 1.1 equiv.) was slowly added, and the solution immediately became orange. The reaction was stopped after 15 minutes, and the reaction was quenched with saturated sodium thiosulfate solution, and then extracted with ethyl acetate (3 × 50mL). After the organic phase was combined, it was washed with saturated NaCl water again, and then dried with anhydrous sodium sulfate. After filtration, the solvent was removed under reduced pressure. The residue was separated by rapid column chromatography (mobile phase PE / DCM = 2 : 1) to obtain 1.9 g white solid product 1b, with a yield of 53 %.

Synthesis of diethyl (2R, 2'R) -2,2 ' - (2-iodo-1,3-phenylene) bis (oxy) dipropionate (1c) :

Triphenylphosphine (6.3 g, 2.4 equiv.) and 1b (2.36 g, 10 mmol, 1.0 equiv.) were put into a 100 mL reaction bottle, and then 30 mL tetrahydrofuran was added to fully dissolve and stirred in an ice water bath at 0 ℃ for 15 minutes. Subsequently, ethyl L-lactate (2.7mL, 2.4equiv.) was added under argon protection, and then diisopropyl azodicarboxylate (4.75mL, 2.4equiv.) was slowly added. After all was added, it was moved to room temperature to continue the reaction for 8-12 hours. The reaction was monitored by TLC until the raw material completely disappeared. The solvent tetrahydrofuran was removed under reduced pressure, and the sample was loaded by dry method. After rapid column chromatography separation (mobile phase PE / EA = 10 : 1), 4.1 g colorless oily liquid product 1c was obtained, with a yield of 94 %.

Synthesis of (2R, 2'R) -2,2 ' - (2-iodo-1,3-phenylene) bis (oxy) dipropionic acid (1d) :

1c (4.36 g, 10 mmol) was put into a 250 mL reaction flask, 50 mL tetrahydrofuran, 50 mL anhydrous methanol and 50 mL sodium hydroxide solution with a concentration of 2 mol/L were added, and stirred at room temperature for 15 hours. Then tetrahydrofuran and methanol were removed by rotary evaporator under reduced pressure, and the pH of the remaining solution was adjusted to acidity by concentrated hydrochloric acid. The reaction solution was observed to become white turbid liquid. It was extracted with ethyl acetate (3×50mL). After the organic phase was combined, it was washed with saturated NaCl solution, dried with anhydrous sodium sulfate, filtered, and the ethyl acetate was removed under reduced pressure. A small amount of mixture was retained and recrystallized with petroleum ether / dichloromethane solvent system to obtain 2.7g white solid product for 1d, with a yield of 71 %.

Synthesis of Chiral aryl iodine catalyst（CIC 3）:

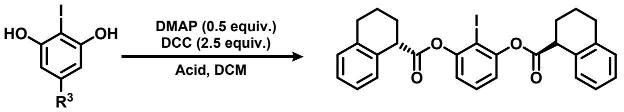
Under the protection of argon, 1d (380 mg, 1 mmol, 1.0 equiv.) was loaded into a 100 mL reaction flask, added with 20 mL of dichloromethane solution to fully dissolve, and stirred in an ice water bath at 0 ℃ for 15 minutes. Subsequently, oxalyl chloride (0.34 mL, 4.0 equiv) was added to the reaction bottle, and 2 ~ 3 drops of N, N-dimethylformamide were added to produce a large number of bubbles. Continue to stir for ten minutes, and then rise to room temperature for 5 hours. After the reaction was completed, the dichloromethane solution and the remaining oxalyl chloride in the reaction bottle were removed by decompression and protected by argon. The crude product of the obtained acyl chloride intermediate was dissolved in 10 mL dichloromethane solution for use. Another 100 mL reaction bottle was added with N-isopropylaniline (0.3 mL, 2.2 equiv) and triethylamine (0.56 mL, 4.0 equiv) under argon protection, dissolved with a small amount of dichloromethane, and stirred in an ice water bath at 0 ℃. After 15 minutes, the previously prepared acyl chloride intermediate solution was slowly added dropwise to the reaction flask with a syringe. After the dropwise addition was completed, the reaction was continued at room temperature for 12 hours. After the completion of the reaction, 50 mL saturated ammonium chloride solution was used to quench the reaction, and the mixture was extracted with dichloromethane (3 × 50 mL). The organic phase was combined, dried with anhydrous sodium sulfate, filtered, and removed under reduced pressure. The dichloromethane solution was loaded by dry method, and the crude product was separated by rapid column chromatography (mobile phase PE / EA = 5 : 1) to obtain white solid product CIC 3 with a yield of 63 %. Chiral aryl iodide catalyst CIC 4 was obtained by the same method.

3.1.2 Synthesis of Chiral aryl iodine catalyst（CIC 9）

1d (380 mg, 1 mmol, 1.0 equiv.) and 2,4,6-trimethylbenzyl alcohol (330 mg, 2.2 equiv.) were put into a 100 mL reaction flask, dissolved in 25 mL dichloromethane solution, and stirred in an ice water bath at 0 ℃ for 15 minutes. Subsequently, 4-dimethylaminopyridine (DMAP, 61 mg, 0.5 equiv.) and dicyclohexylcarbodiimide (DCC, 516 mg, 2.5 equiv.) were added to the bottle. After all was added, the reaction was continued at room temperature for 12 hours. It was observed that the raw material points in the thin layer chromatography disappeared, the reaction was quenched with saturated ammonium chloride solution, and extracted with dichloromethane (3×50mL). After the organic phase was combined, it was dried with anhydrous sodium sulfate, filtered, and the dichloromethane solution was removed under reduced pressure. The dry sample was loaded, and the crude product was separated by rapid column chromatography (mobile phase PE / EA = 10 : 1) to obtain white solid product CIC 9, with a yield of 70 %. Chiral aryl iodide catalysts CIC 1、CIC 2、CIC 5、CIC 6、CIC 7 and CIC 8 were obtained by the same method.

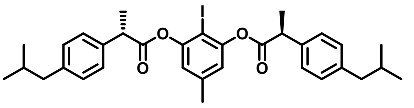
3.2 Synthesis of chiral aryl iodide catalysts based on bilateral ester side chains :

3.2.1 Synthesis of 2-iodo-3-(((R)-1,2,3,4-tetrahydronaphthalene-1-carbonyl)oxy)phenyl(S)-1,2,3,4-tetrahydronaphthalene-1-carboxylate (CIC 10) :

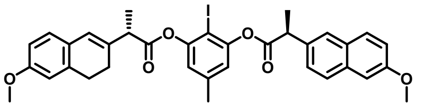


1b, R$_3$ = H

CIC 10



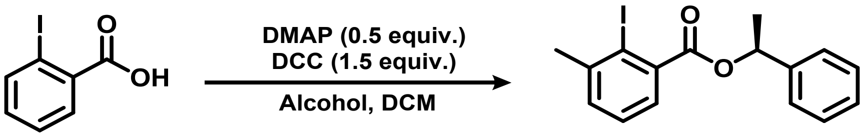
CIC 11



CIC 12

1b (236 mg, 1 mmol, 1.0 equiv.) and (R)-1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (388 mg, 2.2 equiv.) were put into a 100 mL reaction bottle, dissolved in 25 mL dichloromethane solution, and stirred in an ice water bath at 0 ℃ for 15 minutes. Subsequently, 4-dimethylaminopyridine (61 mg, 0.5 equiv.) and dicyclohexylcarbodiimide (516 mg, 2.5 equiv.) were added to the bottle. After all added, move to room temperature to continue the reaction for 12 hours. It was observed that the raw material points in the thin layer chromatography disappeared, the reaction was quenched with saturated ammonium chloride solution, and extracted with dichloromethane (3×50mL). After the organic phase was combined, it was dried with anhydrous sodium sulfate, filtered, and the dichloromethane solution was removed under reduced pressure. The dry sample was loaded, and the crude product was separated by rapid column chromatography (mobile phase PE / DCM = 2 : 1) to obtain colorless yellow oily liquid product Compound CIC 10, the yield was 65 %. Chiral aryl iodide catalysts CIC 11 and CIC 12 were obtained by the same method.

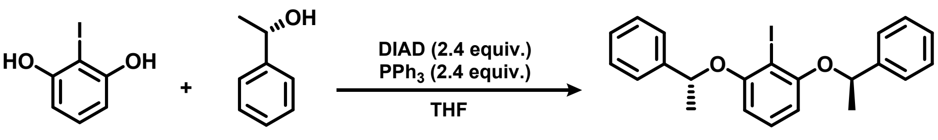
3.2.2 Synthesis of chiral aryl iodide catalysts (S)-1-phenylethyl 2-iodo-3-methylbenzoate based on unilateral ester side chains (CIC 13) :



CIC 13

2-iodobenzoic acid (248 mg, 1 mmol, 1.0 equiv.) and (S)-1-phenylethan-1-ol (122 mg, 1.0 equiv.) were put into a 100 mL reaction flask, dissolved in 25 mL dichloromethane solution, and stirred in an ice water bath at 0 °C for 15 min. Subsequently, 4-dimethylaminopyridine (DMAP, 61 mg, 0.5 equiv.) and dicyclohexylcarbodiimide (DCC, 309 mg, 1.5 equiv.) were added to the bottle. After all added, move to room temperature to continue the reaction for 12 hours. It was observed that the raw material points in the thin layer chromatography disappeared, the reaction was quenched with saturated ammonium chloride solution, and extracted with dichloromethane (3×50mL). After the organic phase was combined, it was dried with anhydrous sodium sulfate, filtered, and the dichloromethane solution was removed under reduced pressure. The dry sample was loaded, and the crude product was separated by rapid column chromatography (mobile phase PE / EA = 10 : 1) to obtain colorless oily product CIC 13, with a yield of 75 %.

3.2.3 Synthesis route of aryl iodide catalyst based on bilateral ether bond structure (CIC 14):

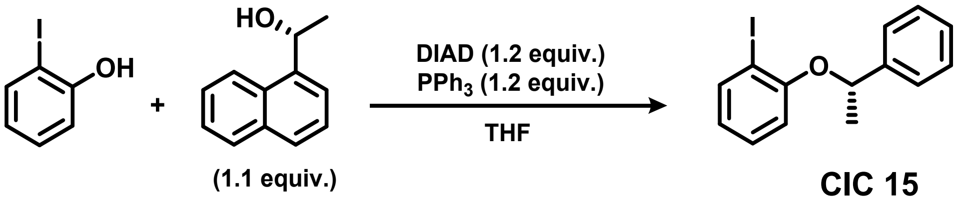


E-1

Under the protection of nitrogen, 2-iodo-1,3-benzenediol (236 mg, 1 mmol, 1.0 equiv.) and triphenylphosphine (629 mg, 2.4 equiv.) were loaded into a 100 mL reaction flask, and 30 mL of tetrahydrofuran solution was added to fully dissolve and stirred in an ice water bath at 0 ℃ for 15 minutes. Subsequently, (S) -1-phenylethanol (268 mg. 2.2 equiv.). Continue stirring for ten minutes, and then slowly add diisopropyl azodicarboxylate (0.47 mL, 2.4 equiv.). After all added, move to room temperature to continue the reaction for 12 hours.

After the raw material points in the thin layer chromatography completely disappeared, the solvent tetrahydrofuran was removed under reduced pressure, and the sample was loaded by dry method. After rapid column chromatography separation (mobile phase PE / EA = 30 : 1), 366 mg colorless oily liquid product CIC 14 was obtained, and the yield was 82 %.

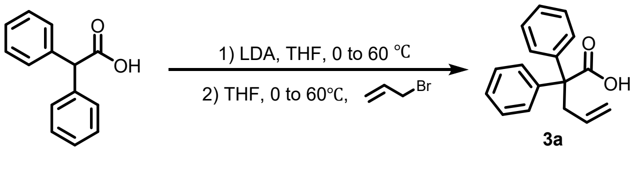
3.2.4 Synthesis of chiral aryl iodide catalysts (S)-1-iodo-2-(1-phenylethoxy)benzene based on unilateral ether bond side chains (CIC 15):

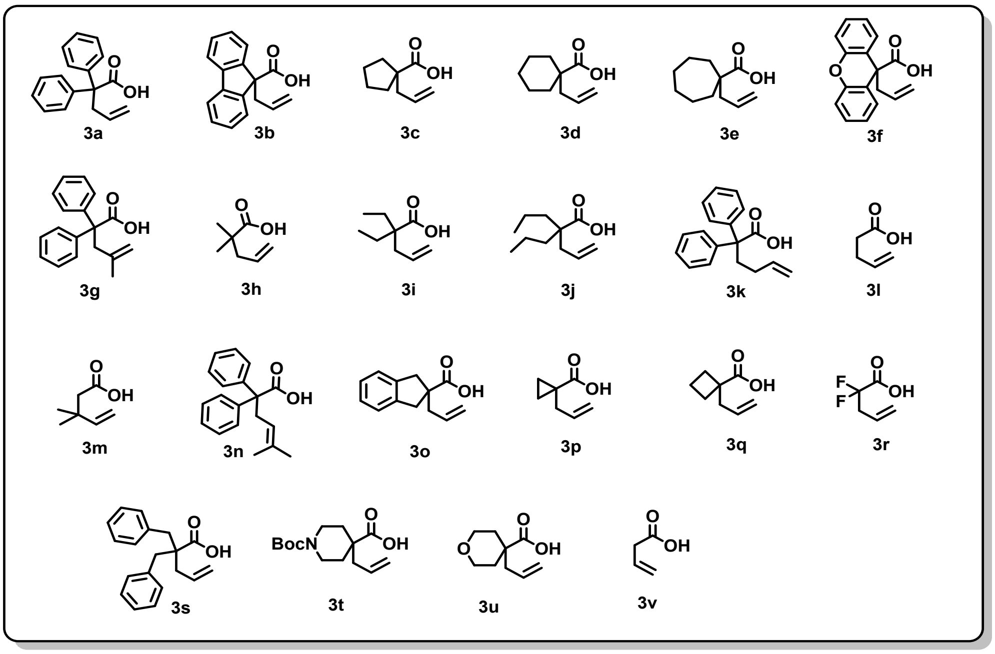


2-Iodophenol (220 mg, 1 mmol, 1.0 equiv.) and triphenylphosphine (314 mg, 1.2 equiv.) were put into a 100 mL reaction bottle, and 20 mL tetrahydrofuran solution was added to fully dissolve and stirred in an ice water bath at 0 °C for 15 minutes. Subsequently, (S)-1-phenylethan-1-ol (134mg, 1.1 equiv.) was added to the flask under argon protection. Continue stirring for ten minutes, and then slowly add diisopropyl azodicarboxylate (DIAD, 0.24 mL, 1.2 equiv.). After all added, move to room temperature to continue the reaction for 12 hours. After the raw material points in the thin layer chromatography completely disappeared, the solvent tetrahydrofuran was removed under reduced pressure, and the sample was loaded by dry method. After rapid column chromatography separation (mobile phase PE / EA = 50 : 1), colorless yellow oily liquid product CIC 15 was obtained with a yield of 58 %.

4. Design and synthesis of alkenyl carboxylic acid substrates

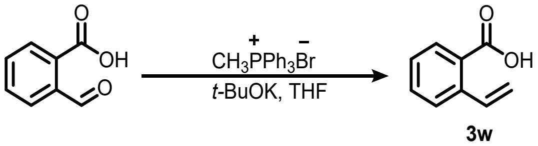
4.1 The synthesis method of alkenyl carboxylic acid substrate :





Under the protection of argon, 2,2-diphenylacetic acid (2.12 g, 10 mmol) was added to a 100 mL reaction flask, and then 30 mL THF was added to fully dissolve. The reaction flask was placed in an ice water bath at 0 ℃ and stirred. After ten minutes, LDA (15 mL, 30 mmol, 2.0 mol / L in THF) was slowly added, and the solution was observed to change from colorless to brown. After the addition, the reaction bottle was stirred in an oil bath at 60 ℃ for 1 h. Subsequently, the reaction was cooled to 0 ℃, and 3-bromopropene (1.8 mL, 15 mmol) was slowly added. At this time, the solution became pale yellow, and the stirring reaction was continued at 60 ℃ after the addition was completed. After 16 h of reaction, the reaction was slowly cooled, then the pH of the reaction solution was adjusted to 1 with 2 M hydrochloric acid solution, and ethyl acetate was used for extraction (50 mL × 3). The organic phase was combined, washed with saturated NaCl water and dried with anhydrous Na_2SO_4.After filtration, the ethyl acetate solution was removed by rotary evaporator under reduced pressure to obtain a yellow liquid crude product. The crude product was separated by rapid column chromatography (PE : EA = 10 : 1) to obtain 2.16 g white solid product 3a with a yield of 86 %. 3b ~ 3g, 3i ~ 3k, 3m ~ 3u were synthesized by the same method.

4.2 The second method for the synthesis of alkenyl carboxylic acid substrates :



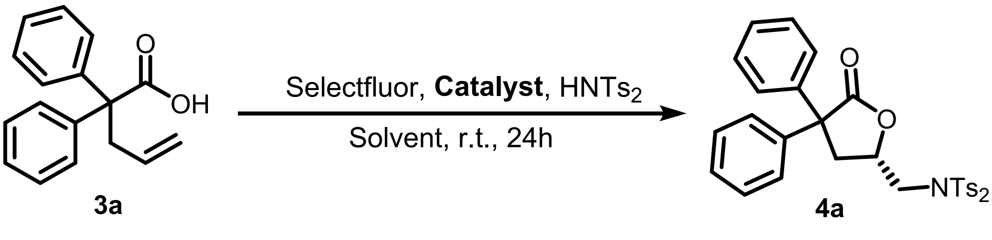
3w

Potassium tert-butoxide (3.4 g, 30.0 mmol) and methyltriphenylphosphonium bromide (7.2 g, 20.0 mmol) were added to a 250 mL round-bottom flask, and then 90 mL THF was added to make it fully dissolved. Stir at room temperature for 1 h under argon protection. Subsequently, the tetrahydrofuran solution of o-carboxybenzaldehyde (1.5 g, 10.0 mmol, in 10 mL THF) was slowly added to the reaction bottle, and then moved to an 80 ° C oil bath for reflux for 12 h. After the reaction was completed, the reaction was quenched with saturated NH4Cl solution, and then extracted with ethyl acetate (50 mL × 3). The ethyl acetate was combined, washed with saturated NaCl solution, dried with anhydrous sodium sulfate, and filtered under reduced pressure to remove ethyl acetate to obtain a pale yellow liquid crude product. The crude product was separated by fast column chromatography (PE : EA = 5 : 1) to obtain 1.18 g white solid product 3w, and the yield was 80 %.

5. Study on asymmetric oxidative amination of alkenyl carboxylic acid substrates catalyzed by chiral aryl iodides

5.1 Optimization of reaction conditions

Table S1 Screening of chiral catalysts



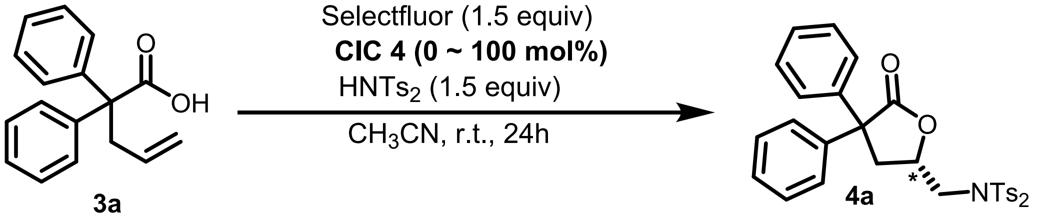
3a **4a**

Entry[a]	Catalyst	Oxidant	Solvent	yield (%) [b]	*ee* (%) [c]
1	**CIC 1**	Selectfluor	CH_3CN	86	35
2	**CIC 2**	Selectfluor	CH_3CN	80	13

3	**CIC 3**	Selectfluor	CH_3CN	75	20
4	**CIC 4**	Selectfluor	CH_3CN	79	43
5	**CIC 5**	Selectfluor	CH_3CN	74	12
6	**CIC 6**	Selectfluor	CH_3CN	81	13
7	**CIC 7**	Selectfluor	CH_3CN	42	48
8	**CIC 8**	Selectfluor	CH_3CN	70	37
9	**CIC 9**	Selectfluor	CH_3CN	71	24
10	**CIC 10**	Selectfluor	CH_3CN	41	21
11	**CIC 11**	Selectfluor	CH_3CN	<5	-
12	**CIC 12**	Selectfluor	CH_3CN	<5	-
13	**CIC 13**	Selectfluor	CH_3CN	<5	-
14	**CIC 14**	Selectfluor	CH_3CN	74	12
15	**CIC 15**	Selectfluor	CH_3CN	69	14

[a]Reaction conditions: 3a (0.2 mmol), Catalyst (15 mol %), HNTs2 (1.5 equiv, 0.3 mmol), and Oxidant (1.75 equiv) in Solvent (0.5M, 4 mL) were stirred in a Glass tube at RT for 24 h. [b]Yields after purification by column chromatography. [c]ee was determined by HPLC.

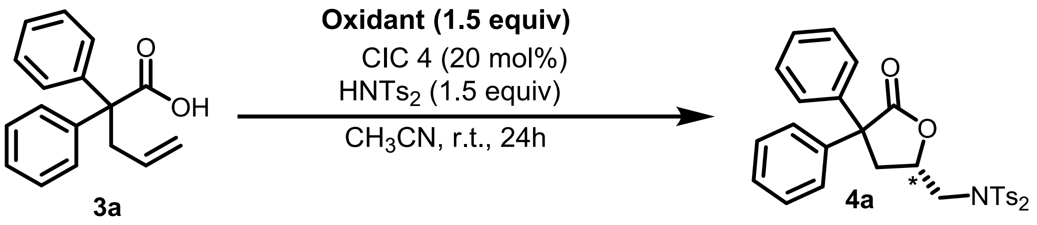
Table S2 The amount of chiral aryl iodide catalyst CIC4



Entry[a]	**CIC4** (mol %)	Yield of **4a**(%)[b]	*ee* of **4a**(%)[c]
1	0	0	0
2	5	67	22
3	10	74	37
4	15	79	43
5	**20**	**83**	**49**
6	30	83	48
7	50	83	49
8	100	82	49

[a] Reaction conditions : 3a (0.2 mmol), Selectfluor (1.5 equiv.), $HNTs_2$ (1.5 equiv.), CH_3CN (4 mL), r.t., for 24 h.[b].Separation yield.[c] Enantioselectivity was obtained by HPLC analysis.

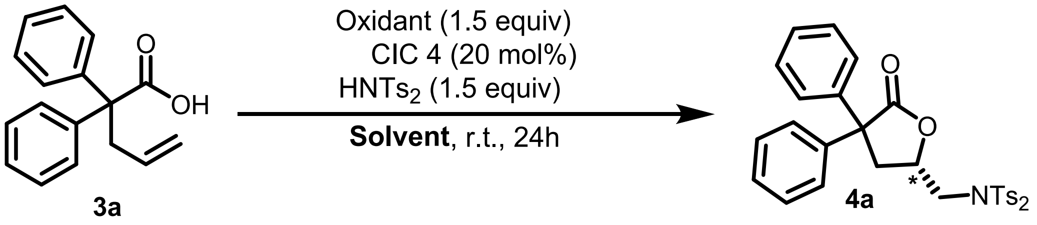
Table S3 Screening of oxidants



Entry[a]	**Oxidant**	Yield of **4a**(%)[b]	*ee* of **4a**(%)[c]
1	Selectfluor	84	48
2	*m*-CPBA	78	47
3	CHP	-	-
4	H_5IO_6	-	-
5	Triazox	73	44
6	NaOCl • $5H_2O$	-	-
7	TBHP	-	-
8	TBPB	-	-

[a] Reaction conditions : 3a (0.2 mmol), Oxidant (1.5 equiv.), $HNTs_2$ (1.5 equiv.), CH_3CN (4 mL), r.t., for 24 h.[b].Separation yield.[c] Enantioselectivity was analyzed by HPLC.

Table S4 Screening of reaction solvents

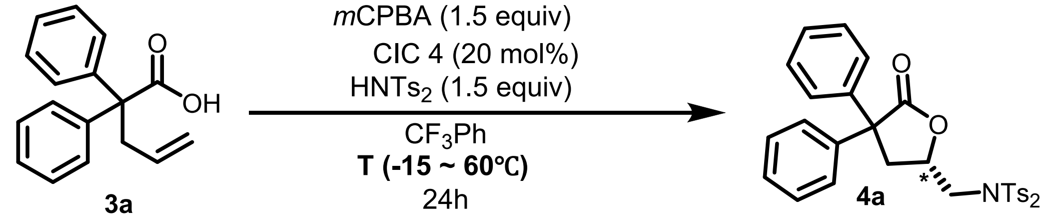


Entry[a]	Solvent	Yield of **4a**(%)[b]	*ee* of **4a**(%)[c]
1	Selectfluor+CH_3CN	84	48
2	Selectfluor+DMF	-	-
3	Selectfluor+DMSO	-	-
4	Selectfluor+HFIP	-	-
5	Selectfluor+MeOH	64	13
6	*m*-CPBA+CH_3CN	80	47
7	***m*-CPBA+CF_3Ph**	**79**	**76**
8	*m*-CPBA+THF	-	-

9	*m*-CPBA+DCM	75	55
10	*m*-CPBA+*t*BuOMe	-	-
11	*m*-CPBA+MeOH	59	12
12	*m*-CPBA+DCE	74	72
13	*m*-CPBA+$CHCl_3$	71	70
14	*m*-CPBA+Toluene	63	36
15	*m*-CPBA+CCl_4	77	47
16	*m*-CPBA+HFIP	-	-
17	*m*-CPBA+TFA	Trace	-
18	*m*-CPBA+Et_2O	48	33

[a] Reaction conditions : 3a (0.2 mmol), CIC4 (20 mol %), Oxidant (1.5 equiv.), $HNTs_2$ (1.5 equiv.), Solvent (4 mL), r.t., for 24 h.[b] Separation yield.[c] Enantioselectivity was analyzed by HPLC.

Table S5 Screening of reaction temperature



Entry[a]	Temperature (°C)	Yield of **4a**(%)[b]	*ee* of **4a**(%)[c]
1	-15°C	61	40
2	0°C	64	63
3	25°C	79	77
4	**40°C**	**82**	**95**
5	50°C	83	89
6	60°C	85	82

[a] Reaction conditions : 3a (0.2 mmol), mCPBA (1.5 equiv.), $HNTs_2$ (1.5 equiv.), CF_3Ph (4 mL), T ℃, for 24 h.[b] Separation yield.[c] Enantioselectivity was obtained by HPLC analysis.

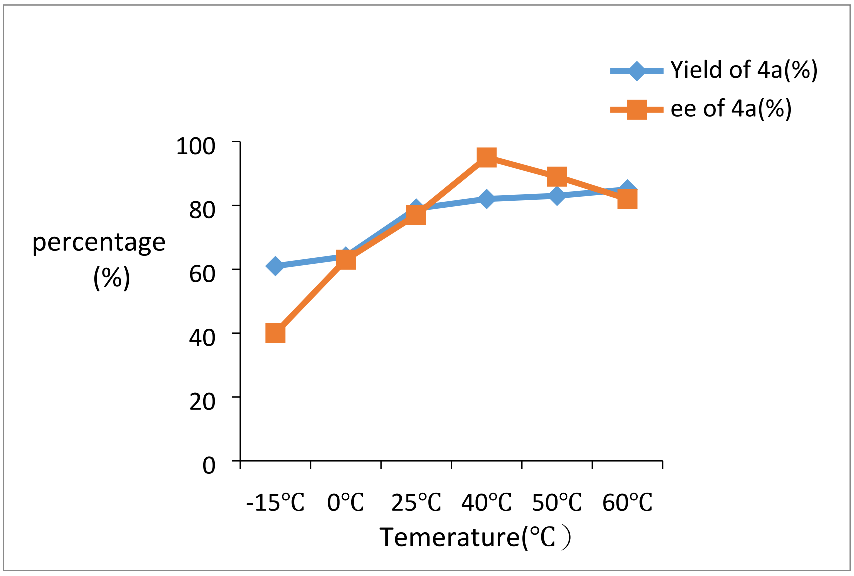
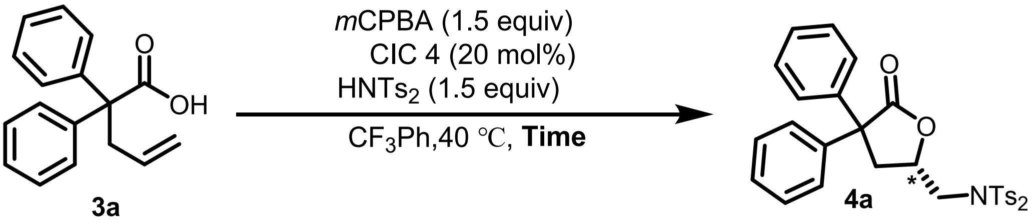


Fig. S1The curve of ee value of the product changing with temperature

Table S6 Screening of reaction time



Entry[a]	Time (h)	*ee* of **4a**(%)[b]
1	5	23
2	10	28
3	15	47
4	20	68
5	25	96
6	30	97
7	**35**	**99**
8	40	98
9	50	98
10	60	91
11	70	94
12	80	93
13	90	89
14	100	82

[a] Reaction conditions : 3a (0.2 mmol), mCPBA (1.5 equiv.), $HNTs_2$ (1.5 equiv.), CF_3Ph (4 mL), 40 ℃, for 5 to 100 h.[b] The enantioselectivity was obtained by HPLC analysis.

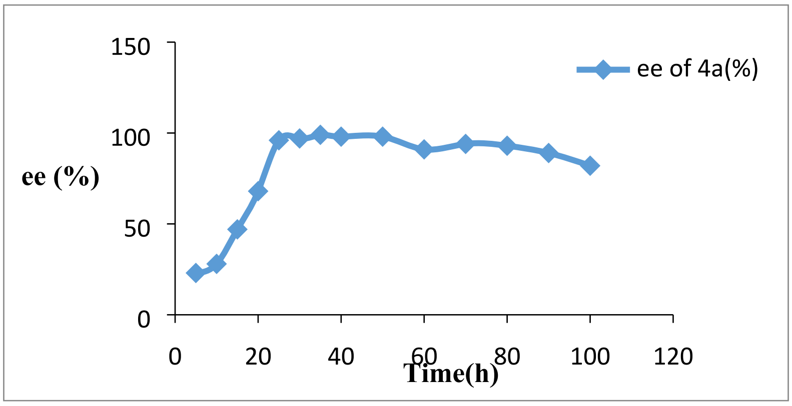
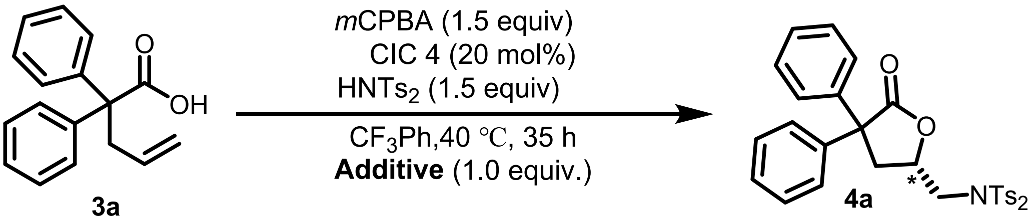


Fig. S2 The curve of the ee value of the product with the reaction time is shown.

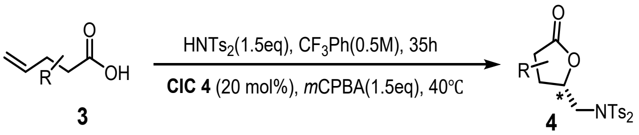
Table S7 Screening of additives

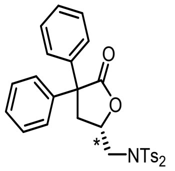


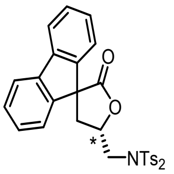
Entry[a]	Additive (1.0 equiv.)	Yield of **4a**(%)[b]	*ee* of **4a**(%)[c]
1	$BF_3 \cdot Et_2O$	43	61
2	Pyridine	Trace	-
3	**No Additive**	**83**	**99**

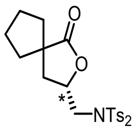
[a] Reaction conditions : 3a (0.2 mmol), mCPBA (1.5 equiv.), $HNTs_2$ (1.5 equiv.), CF_3Ph (4 mL), 40 ℃, for 35 h, Additive (1.0 equiv.).[b] separation yield.[c] enantioselectivity was obtained by HPLC analysis.

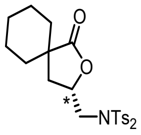
5.2 Study on substrate applicability

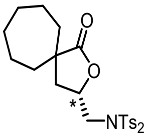


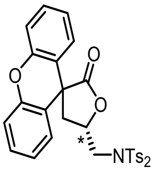

4a
83% yields[b], 99% *ee*[c]

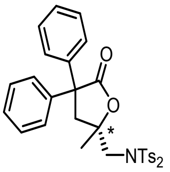

4b
48% yields[b], 90% *ee*[c]

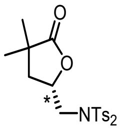

4c
70% yields[b], 64% *ee*[c]

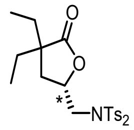

4d
63% yields[b], 66% *ee*[c]

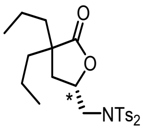

4e
67% yields[b], 73% *ee*[c]

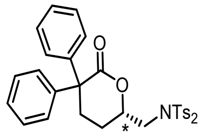

4f
27% yields[b], 81% *ee*[c]

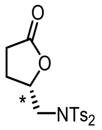

4g
59% yields[b], 97% *ee*[c]

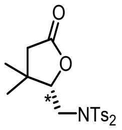

4h
68% yields[b], 62% *ee*[c]

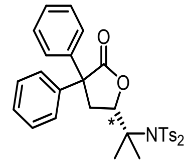

4i
62% yields[b], 33% *ee*[c]

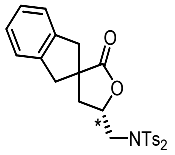

4j
70% yields[b], 55% *ee*[c]

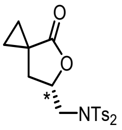

4k
67% yields[b], 92% *ee*[c]

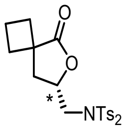

4l[d]
62% yields[b], 55% *ee*[c]

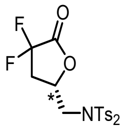

4m[d]
53% yields[b], 64% *ee*[c]

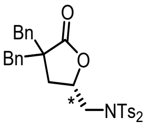

4n
Not detective

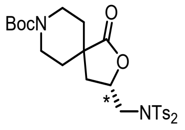

4o
78% yields[b], 94% *ee*[c]

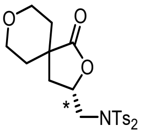

4p
61% yields[b], 29% *ee*[c]

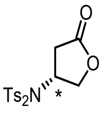

4q
57% yields[b], 31% *ee*[c]

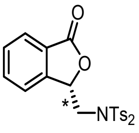

4r
45% yields[b], 29% *ee*[c]

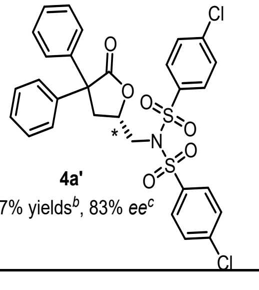

4s
72% yields[b], 89% *ee*[c]


4t
67% yields[b], 79% *ee*[c]


4u
65% yields[b], 89% *ee*[c]


4v
Trace


4w
48% yields[b], 24% *ee*[c]


4a'
77% yields[b], 83% *ee*[c]

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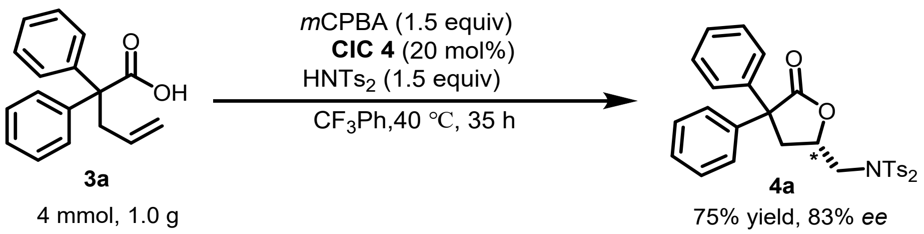
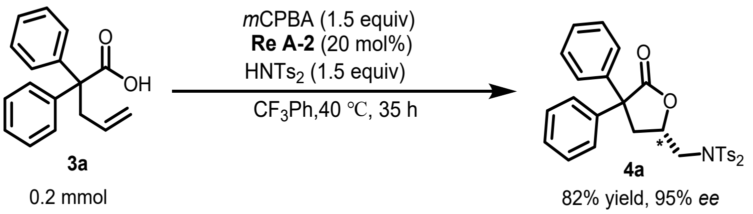
[a] Reaction conditions were as follows : 3 (0.2 mmol), mCPBA (1.5 equiv.), HNTs$_2$ (1.5 equiv.), CF$_3$Ph (4 mL), 40 ℃, for 35 h.[b] Separation yield.[c] The enantioselectivity was analyzed by HPLC.[d] Using catalyst CIC5.

5.3 Oxidative amination gram-scale reaction of alkenyl carboxylic acid



5.4 Recovery and reuse of catalyst

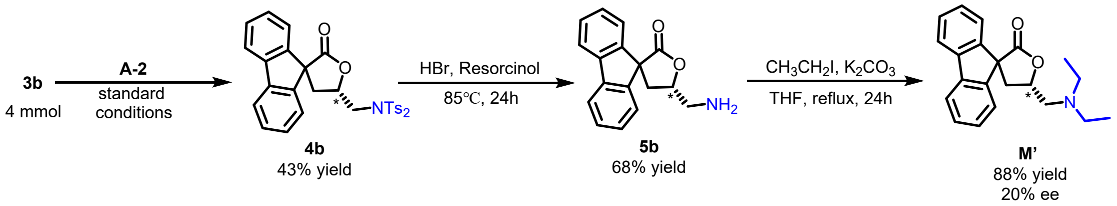


Recycle 1

6. Application of Chiral Aryl Iodine catalyzed Alkene Bifunctionalization Reaction in Drug SynthesisStructural characterization

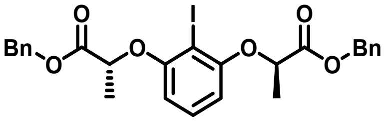
Synthesis of **5b**: A 100 mL high-temperature and pressure-resistant reaction bottle was taken, and product **4b** (1 g, 1.7 mmol), resorcinol (935 mg, 8.5 mmol), HBr solution (16 mL, 33 % wt in acetic acid) were added. After all addition, the reaction was heated overnight at 85℃. After the reaction is completed, the acetic acid in the solution is removed by a rotary evaporator. Distilled water (50 mL) was added to the residual liquid to dissolve, and then the water phase (50 mL × 3) was extracted and washed with Et_2O. The water phase was collected and the water phase was yellow. The pH of the aqueous phase was adjusted to >7 by adding saturated $NaHCO_3$ solution, and then the aqueous phase was extracted with DCM (50 mL × 3). The organic phase was dried, filtered, and decompressed to remove the solvent to obtain the crude product. The crude product was separated by column chromatography (DCM : MeOH = 10: 1) to obtain 313 mg yellow solid compound **5b** with a yield of 68 %.

Synthesis of **M'**: A 100 mL reaction flask was added with **5b** (313 mg, 1.18 mmol), K_2CO_3 (1.4 g, 9.9 mmol), 15 mL of iodoethane and 20 mL of THF solution, and refluxed at 69℃ for 24 h. After the reaction, the solvent was removed by vacuum rotary evaporation. The residue was separated by column chromatography (DCM: MeOH = 15: 1) to obtain 334 mg of pale yellow solid compound **M'**, with a yield of 88 % and an enantioselectivity of 20 % *ee*.

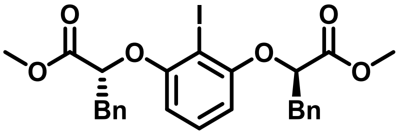


7 Structural characterization

7.1 Characterization of the catalysts

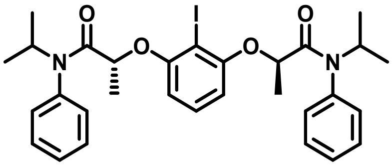


Dibenzyl 2,2'-((2-iodo-1,3-phenylene)bis(oxy))(2R,2'R)-dipropionate (CIC 1) : Colorless oil; (89 %.); (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.23 (m, 10H), 7.00 (t, *J* = 8.2 Hz, 1H), 6.30 (d, *J* = 8.4 Hz, 2H), 5.17 (s, 4H), 4.79 (q, *J* = 6.8 Hz, 2H), 1.70 (d, *J* = 6.4 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.48, 158.23, 135.36, 129.55, 128.58, 128.39, 128.23, 107.05, 80.73, 74.23, 66.92, 18.56. HRMS (ESI) *m/z*: $C_{26}H_{25}IO_6$ [M + H]$^+$ Cal: 561.0769; Found: 561.0770.

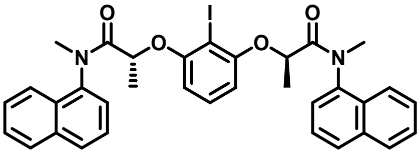


Dimethyl 2,2'-((2-iodo-1,3-phenylene)bis(oxy))(2R,2'R)-bis(3-phenylpropanoate) **(CIC 2).**[1] Colorless oil; (80 %.); (EtOAc/petroleum ether = 1:8); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.33 (m, 4H), 7.29 – 7.18 (m, 6H), 7.00 (td, *J* = 8.4, 2.3 Hz, 1H), 6.17 (d, *J* = 8.3 Hz, 2H), 4.77 (dd, *J* = 7.8, 4.2 Hz, 2H), 3.61 (d, *J* = 2.6 Hz, 6H), 3.28 (qt, *J* = 14.1, 6.2 Hz, 4H). HRMS (ESI) *m/z*: $C_{26}H_{25}IO_6$ [M + H]$^+$ Cal: 561.0769; Found: 561.0771.

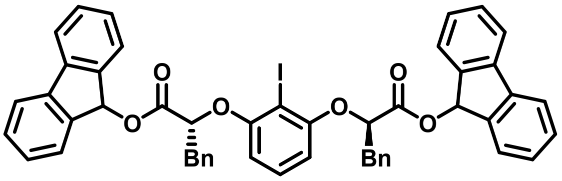
[1]J.C. Sarie, C. Thiehoff, J. Neufeld, C.G. Daniliuc, R. Gilmour, Enantioselective Synthesis of 3-Fluorochromanes via Iodine(I)/Iodine(III) Catalysis, *Angew. Chem. Int. Ed.*, 2020, **59**(35), 15069-15075.



(2R,2'R)-2,2'-((2-iodo-1,3-phenylene)bis(oxy))bis(N-isopropyl-N-phenylpropanamide) **(CIC 3).** White solid (63%.); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.34 (h, J = 9.7, 8.4 Hz, 6H), 7.06 – 6.98 (m, 3H), 6.91 (d, J = 7.2 Hz, 2H), 6.26 (d, J = 8.2 Hz, 2H), 5.01 (p, J = 6.9 Hz, 2H), 4.40 (q, J = 6.4 Hz, 2H), 1.47 (d, J = 6.4 Hz, 6H), 1.03 (t, J = 5.9 Hz, 12H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.74, 158.14, 137.04, 130.37, 130.26, 129.47, 129.13, 128.78, 128.60, 109.09, 83.64, 73.81, 46.64, 20.83, 20.74, 18.13, 17.88. HRMS (ESI) *m*/*z*: $C_{30}H_{35}IN_2O_4$ [M+H]$^+$ Cal: 615.1714; Found: 615.1714.

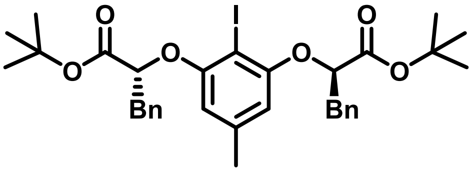


(2R,2'R)-2,2'-((2-iodo-1,3-phenylene)bis(oxy))bis(N-methyl-N-(naphthalen-1-yl)propanamide) **(CIC 4).** Yellow solid (66%.); (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.67 (m, 5H), 7.60 – 7.30 (m, 8H), 7.13 – 6.79 (m, 2H), 6.14 (dt, *J* = 20.7, 8.2 Hz, 2H), 4.58 – 4.35 (m, 2H), 3.37 (dd, *J* = 12.7, 4.8 Hz, 6H), 1.49 (dd, *J* = 24.2, 6.5 Hz, 3H), 1.35 (dd, *J* = 12.1, 6.4 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.51, 171.13, 157.95, 138.67, 138.52, 134.74, 129.96, 129.92, 129.87, 129.23, 129.12, 128.96, 128.82, 128.77, 128.56, 128.47, 127.44, 127.25, 126.99, 126.66, 125.89, 125.55, 122.62, 122.10, 109.56, 109.16, 83.91, 73.47, 37.70, 18.20. HRMS (ESI) *m*/*z*: $C_{34}H_{31}IN_2O_4$ [M+H]$^+$ Cal: 659.1401; Found: 659.1400.

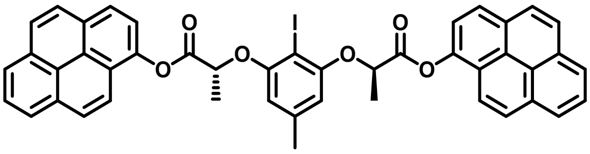


Di(9H-fluoren-9-yl) 2,2'-((2-iodo-1,3-phenylene)bis(oxy))(2R,2'R)-bis(3-phenylpropanoate) **(CIC 5).** White solid (70%.); (EtOAc/petroleum ether = 1:1); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 (t, *J* = 7.1 Hz, 4H), 7.47 – 7.41 (m, 4H), 7.40 – 7.36 (m, 3H), 7.26 (ddd, *J* = 45.7, 14.0, 7.2 Hz, 11H), 7.12 (t, *J* = 7.5 Hz, 2H), 7.04 – 6.95 (m, 3H), 6.71 (s, 2H), 6.33 (d, *J* = 8.3 Hz, 2H), 4.95 (dd, *J* = 7.8, 5.2 Hz, 2H), 3.45 (dd, *J* = 13.9, 7.8 Hz, 2H), 3.36 (dd, *J* = 14.0, 5.1

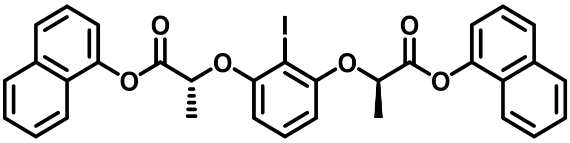
Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.26, 158.15, 141.44, 141.17, 141.07, 140.99, 135.75, 130.09, 129.71, 129.63, 129.39, 128.57, 127.85, 127.83, 127.16, 126.21, 125.71, 120.08, 120.03, 106.44, 78.84, 75.99, 39.05. HRMS (ESI) *m/z*: $C_{50}H_{37}IO_6$ $[M + H]^+$ Cal: 861.1708; Found: 861.1710.



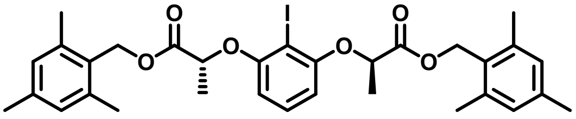
Di-tert-butyl 2,2'-((2-iodo-5-methyl-1,3-phenylene)bis(oxy))(2R,2'R)-bis(3-phenylpropanoate) **(CIC 6).** Colorless oil; (64 %.); (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 (d, *J* = 6.8 Hz, 4H), 7.31 (t, *J* = 7.1 Hz, 4H), 7.27 – 7.23 (m, 2H), 6.11 (d, *J* = 8.0 Hz, 2H), 4.70 (dt, *J* = 8.4, 4.3 Hz, 2H), 3.35 (dd, *J* = 14.0, 7.7 Hz, 2H), 3.25 (dd, *J* = 14.0, 4.9 Hz, 2H), 2.18 (s, 3H), 1.32 (s, 18H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.64, 157.93, 157.86, 139.43, 136.39, 130.08, 128.26, 126.85, 107.16, 107.04, 82.10, 38.99, 27.80, 27.77, 21.70. HRMS (ESI) *m/z*: $C_{33}H_{39}IO_6$ $[M + H]^+$ Cal: 659.1864; Found: 659.1863.



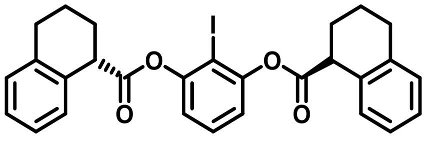
Di(pyren-1-yl) 2,2'-((2-iodo-5-methyl-1,3-phenylene)bis(oxy))(2R,2'R)-dipropionate **(CIC 7).** White solid (75%.); (EtOAc/petroleum ether = 1:1); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 – 8.11 (m, 6H), 8.06 – 7.96 (m, 8H), 7.93 – 7.87 (m, 2H), 7.76 (d, *J* = 8.1 Hz, 2H), 6.70 (s, 2H), 5.30 (q, *J* = 7.0 Hz, 2H), 2.46 (s, 3H), 2.10 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.86, 158.30, 143.71, 140.72, 131.10, 130.87, 129.54, 128.28, 127.33, 127.04, 126.39, 125.63, 125.57, 125.34, 124.94, 124.43, 123.04, 119.96, 119.28, 108.80, 74.59, 22.06, 18.95. HRMS (ESI) *m/z*: $C_{45}H_{31}IO_6$ $[M + H]^+$ Cal: 795.1238; Found: 795.1239.



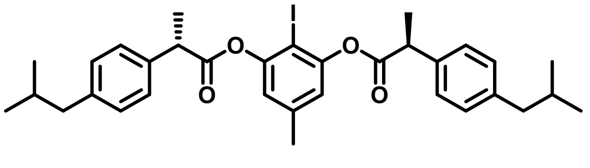
Di(naphthalen-1-yl)2,2'-((2-iodo-1,3-phenylene)bis(oxy))(2R,2'R)-dipropionate **(CIC 8).** White solid (71%.); (EtOAc/petroleum ether = 1:1); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, J = 8.0 Hz, 2H), 7.65 (t, J = 7.3 Hz, 4H), 7.39 (dt, J = 20.3, 7.3 Hz, 6H), 7.24 (t, J = 8.3 Hz, 1H), 7.17 – 7.13 (m, 2H), 6.64 (d, J = 8.2 Hz, 2H), 5.12 (q, J = 6.4 Hz, 2H), 1.93 (d, J = 6.5 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.20, 157.45, 145.06, 133.62, 128.91, 126.98, 125.53, 125.28, 124.24, 119.98, 116.69, 106.40, 73.51, 17.78. HRMS (ESI) *m/z*: $C_{32}H_{25}IO_6$ $[M + H]^+$ Cal: 633.0769; Found: 633.0776.



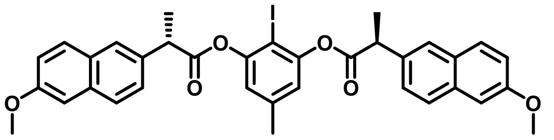
Bis(2,4,6-trimethylbenzyl)2,2'-((2-iodo-1,3-phenylene)bis(oxy))(2R,2'R)-dipropionate **(CIC 9).** White solid (70%.); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.02 (t, J = 8.3 Hz, 1H), 6.86 (s, 4H), 6.32 (dd, J = 8.3, 3.8 Hz, 2H), 5.24 (qd, J = 12.1, 3.4 Hz, 4H), 4.75 (q, J = 6.7 Hz, 2H), 2.29 (d, J = 6.0 Hz, 18H), 1.69 (d, J = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.80, 158.27, 138.66, 138.29, 129.41, 129.09, 128.44, 106.97, 74.26, 61.94, 21.04, 19.51, 18.59. HRMS (ESI) *m/z*: $C_{32}H_{37}IO_6$ $[M + Na]^+$ Cal: 667.1527; Found: 667.1532.



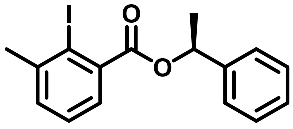
2-iodo-3-(((R)-1,2,3,4-tetrahydronaphthalene-1-carbonyl)oxy)phenyl (S)-1,2,3,4-tetrahydronaphthalene-1-carboxylate **(CIC 10).** Yellow solid (65%.); (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.32 (m, 3H), 7.27 – 7.19 (m, 4H), 7.19 – 7.15 (m, 2H), 6.97 (d, J = 8.1 Hz, 2H), 4.17 (t, J = 5.9 Hz, 2H), 2.88 (dtd, J = 23.4, 16.7, 6.2 Hz, 4H), 2.47 (qd, J = 8.1, 2.7 Hz, 2H), 2.19 (dddd, J = 32.7, 17.3, 8.9, 3.5 Hz, 4H), 1.87 (ddd, J = 13.9, 10.3, 6.2 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.14, 152.67, 137.52, 132.32, 129.89, 129.58, 129.44, 127.24, 125.96, 120.21, 88.06, 44.92, 29.11, 26.59, 20.58. HRMS (ESI) *m/z*: $C_{28}H_{25}IO_4$ $[M + Na]^+$ Cal: 575.0690; Found: 575.0710.



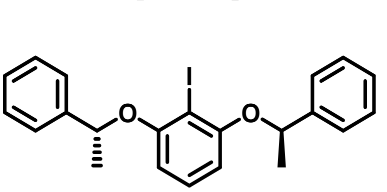
2-iodo-5-methyl-1,3-phenylene (2S,2'S)-bis(2-(4-isobutylphenyl)propanoate) **(CIC 11).** White solid (58%.); (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.30 (m, 4H), 7.15 (d, J = 7.6 Hz, 4H), 6.69 (s, 2H), 3.99 (q, J = 7.3 Hz, 2H), 2.48 (d, J = 7.2 Hz, 4H), 2.27 (s, 3H), 1.86 (dq, J = 14.1, 7.1 Hz, 2H), 1.66 (d, J = 7.0 Hz, 6H), 0.98 – 0.89 (m, 12H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.13, 152.10, 140.96, 140.21, 136.70, 129.47, 127.70, 120.95, 83.49, 45.37, 45.08, 30.25, 22.44, 20.96, 18.43. HRMS (ESI) *m/z*: $C_{33}H_{39}IO_4$ [M + H]$^+$ Cal: 627.1966; Found: 627.1961.



2-iodo-5-methyl-1,3-phenylene (2S,2'S)-bis(2-(6-methoxynaphthalen-2-yl)propanoate) **(CIC 12).** Yellow solid (59%.); (EtOAc/petroleum ether = 1:1); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.72 (m, 6H), 7.53 (dd, J = 8.6, 1.9 Hz, 2H), 7.20 – 7.12 (m, 4H), 6.66 (s, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.93 (s, 6H), 2.23 (s, 3H), 1.74 (d, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.08, 157.78, 152.09, 140.31, 134.57, 133.93, 129.41, 128.97, 127.33, 126.61, 126.53, 120.98, 119.11, 105.63, 83.57, 55.35, 45.68, 20.93, 18.53. HRMS (ESI) *m/z*: $C_{35}H_{31}IO_6$ [M + H]$^+$ Cal: 675.1238; Found: 675.1228.

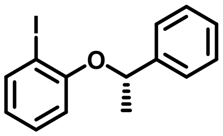


(S)-1-phenylethyl 2-iodo-3-methylbenzoate **(CIC 13).** Colourless oil (75%.); (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, J = 7.5 Hz, 2H), 7.38 – 7.25 (m, 5H), 7.22 (t, J = 7.5 Hz, 1H), 6.12 (q, J = 6.6 Hz, 1H), 2.47 (s, 3H), 1.68 (d, J = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.60, 143.35, 141.19, 138.73, 131.64, 128.55, 128.05, 127.77, 127.01, 126.39, 99.84, 74.05, 29.63, 22.15. HRMS (ESI) *m/z*: $C_{16}H_{15}IO_2$ [M + H]$^+$ Cal: 367.0189; Found: 367.0190.



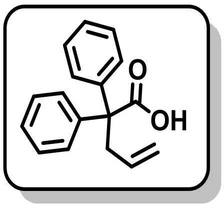
((1R,1'R)-((2-iodo-1,3-phenylene)bis(oxy))bis(ethane-1,1-diyl))dibenzene **(CIC 14).** Colourless oil

(82%.); (EtOAc/petroleum ether = 1:50); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.29 (m, 4H), 7.29 – 7.20 (m, 4H), 7.20 – 7.16 (m, 2H), 6.81 (t, *J* = 8.3 Hz, 1H), 6.17 (d, *J* = 8.3 Hz, 2H), 5.27 (q, *J* = 6.4 Hz, 2H), 1.62 (d, *J* = 6.4 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.99, 142.87, 129.12, 128.62, 127.52, 125.65, 106.99, 81.04, 77.38, 24.57. HRMS (ESI) *m/z*: $C_{22}H_{21}IO_2$ [M + H]$^+$ Cal: 445.0659; Found: 445.0661.

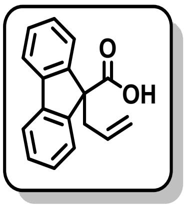


(S)-1-iodo-2-(1-phenylethoxy)benzene **(CIC 15).** Pale yellow oily (58%.); (EtOAc/petroleum ether = 1:50);^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.61 (m, 1H), 7.32 (d, *J* = 7.1 Hz, 2H), 7.29 – 7.16 (m, 3H), 7.04 (t, *J* = 7.7 Hz, 1H), 6.56 (dq, *J* = 10.8, 6.9, 5.2 Hz, 2H), 5.27 (q, *J* = 6.2, 4.9 Hz, 1H), 1.62 (d, *J* = 3.9 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.50, 142.59, 139.48, 129.18, 128.69, 127.64, 125.68, 122.52, 114.19, 87.69, 77.38, 24.50. HRMS (ESI) *m/z*: $C_{14}H_{13}IO$ [M + H]$^+$ Cal: 325.0084; Found: 325.0078.

7.2 Characterization of Vinyl Carboxylic Acid Substrates

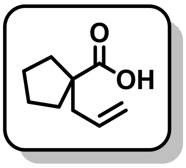


2,2-diphenylpent-4-enoic acid **(3a).** White solid (86%.); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.34 – 7.18 (m, 10H), 5.56 (ddt, *J* = 17.2, 10.3, 6.9 Hz, 1H), 4.96 – 4.85 (m, 2H), 3.11 (d, *J* = 6.9 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 174.81, 142.88, 134.65, 128.66, 127.69, 126.48, 117.92, 59.51, 42.08. HRMS (ESI) *m/z*: $C_{17}H_{16}O_2$ [M−H]$^-$ Cal: 251.1078; Found: 251.1079.

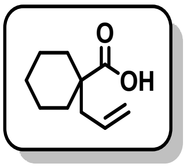


9-allyl-9H-fluorene-9-carboxylic acid **(3b).** Pale yellow solid （64%.）; (EtOAc/petroleum ether = 1:6); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.85 (d, *J* = 7.5 Hz, 2H), 7.59 (d, *J* = 7.3 Hz, 2H), 7.34 (dt, *J* =

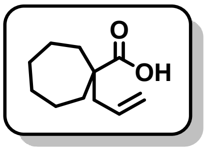
23.8, 7.6 Hz, 4H), 5.58 (dq, $J = 17.1$, 8.1 Hz, 1H), 4.92 (dd, $J = 28.7$, 13.7 Hz, 2H), 2.73 (t, $J = 6.8$ Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 146.48, 140.43, 135.40, 127.00, 126.84, 124.61, 119.87, 117.08, 46.16, 36.68. HRMS (ESI) m/z: $C_{17}H_{14}O_2$ $[M + H]^+$ Cal: 251.1067; Found: 251.1067.



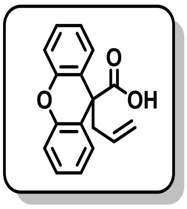
1-allylcyclopentane-1-carboxylic acid **(3c).** Pale yellow oil (67%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.09 (s, 1H), 5.69 (ddt, $J = 17.3$, 10.2, 7.2 Hz, 1H), 5.08 – 4.96 (m, 2H), 2.28 (d, $J = 7.2$ Hz, 2H), 2.01 – 1.89 (m, 2H), 1.56 (qt, $J = 7.0$, 4.9, 3.6 Hz, 4H), 1.44 (tt, $J = 8.5$, 3.9 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 178.14, 135.22, 117.25, 52.68, 42.30, 34.96, 24.56. HRMS (ESI) m/z: $C_9H_{14}O_2$ $[M-H]^-$ Cal: 153.0921; Found: 153.0934.



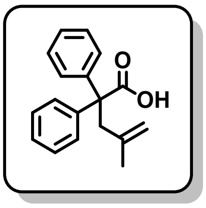
1-allylcyclohexane-1-carboxylic acid **(3d).** Pale yellow oil (59%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.14 (s, 1H), 5.67 (ddt, $J = 15.7$, 10.8, 7.5 Hz, 1H), 5.05 – 4.94 (m, 2H), 2.17 (d, $J = 7.5$ Hz, 2H), 1.89 (dd, $J = 13.1$, 5.1 Hz, 2H), 1.50 (tq, $J = 9.6$, 4.4 Hz, 3H), 1.34 – 1.12 (m, 5H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 177.01, 133.92, 117.40, 45.91, 43.89, 33.19, 25.35, 22.69. HRMS (ESI) m/z: $C_{10}H_{16}O_2$ $[M-H]^-$ Cal: 167.1078; Found: 167.1079.



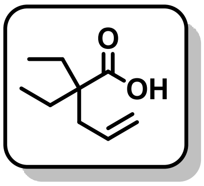
1-allylcycloheptane-1-carboxylic acid **(3e).** Yellow oil (66%); (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.11 (s, 1H), 5.72 – 5.58 (m, 1H), 5.07 – 4.94 (m, 2H), 2.19 (d, $J = 7.4$ Hz, 2H), 1.94 (t, $J = 9.4$ Hz, 2H), 1.44 (dq, $J = 18.7$, 10.3 Hz, 10H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 178.05, 134.60, 117.50, 48.68, 44.47, 35.40, 29.46, 23.05. HRMS (ESI) m/z: $C_{11}H_{18}O_2$ $[M-H]^-$ Cal: 181.1234; Found: 181.1235.



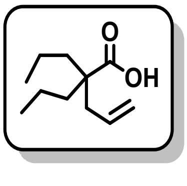
***9-allyl-9H-xanthene-9-carboxylic acid* (3f).** Yellow solid (48%); (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.29 (t, *J* = 7.7 Hz, 4H), 7.18 – 7.05 (m, 4H), 5.13 (ddt, *J* = 17.4, 10.7, 7.1 Hz, 1H), 4.82 – 4.67 (m, 2H), 2.99 (d, *J* = 7.1 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 174.34, 149.94, 132.95, 128.69, 127.51, 123.37, 121.52, 118.90, 116.12, 49.10, 44.42. HRMS (ESI) *m/z*: $C_{17}H_{14}O_3$ [M−H]$^-$ Cal: 265.0870; Found: 265.0878.



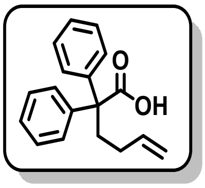
***4-methyl-2,2-diphenylpent-4-enoic acid* (3g).** White solid (70%); (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 7.32 – 7.24 (m, 8H), 7.23 – 7.15 (m, 2H), 4.68 – 4.63 (m, 1H), 4.50 (d, J = 2.4 Hz, 1H), 3.12 (s, 2H), 1.32 (s, 3H). ^{13}C NMR (101 MHz, DMSO-d$_6$) δ 174.67, 143.64, 142.06, 128.62, 127.56, 126.34, 114.71, 59.33, 45.05, 24.15. HRMS (ESI) *m/z*: $C_{18}H_{18}O_2$ [M + H]$^+$ Cal: 267.1380; Found: 263.1378.



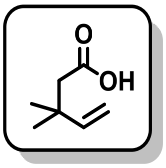
***2,2-diethylpent-4-enoic acid* (3i).** Colorless oil (62%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.09 (s, 1H), 5.66 (ddt, *J* = 17.3, 10.2, 7.3 Hz, 1H), 5.14 – 4.97 (m, 2H), 2.22 (d, *J* = 7.4 Hz, 2H), 1.49 – 1.44 (m, 4H), 0.84 (t, 3H), 0.75 (t, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 177.25, 134.30, 117.52, 48.04, 36.83, 26.61, 24.38, 11.60, 8.23. HRMS (ESI) *m/z*: $C_9H_{16}O_2$ [M−H]$^-$ Cal: 155.1078; Found: 155.1079.



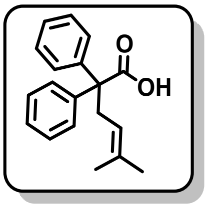
2,2-dipropylpent-4-enoic acid **(3j).** Colorless oil (63%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.12 (s, 1H), 5.67 (ddt, *J* = 17.4, 10.3, 7.4 Hz, 1H), 5.25 – 4.75 (m, 2H), 2.23 (d, *J* = 7.4 Hz, 2H), 1.46 – 1.35 (m, 4H), 1.25 – 1.11 (m, 4H), 0.85 (q, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 177.46, 134.37, 117.54, 47.96, 44.41, 37.86, 36.99, 34.10, 20.08, 16.79, 14.48, 13.84. HRMS (ESI) *m*/*z*: $C_{11}H_{20}O_2$ [M−H]$^-$ Cal: 183.1391; Found: 183.1392.



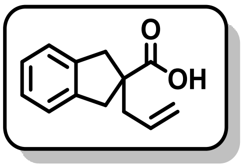
2,2-diphenylhex-5-enoic acid **(3k).** White solid (65%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.79 (s, 1H), 7.28 (dq, *J* = 15.1, 7.3 Hz, 10H), 5.77 (ddt, *J* = 16.6, 9.3, 6.1 Hz, 1H), 4.90 (d, *J* = 11.6 Hz, 2H), 2.45 – 2.29 (m, 2H), 1.75 (q, *J* = 7.1 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 174.92, 143.14, 138.29, 128.57, 127.79, 126.49, 114.60, 59.40, 36.65, 29.42. HRMS (ESI) *m*/*z*: $C_{18}H_{18}O_2$ [M−H]$^-$ Cal: 265.1234; Found: 265.1235.



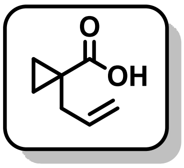
3,3-dimethylpent-4-enoic acid **(3m).** Colorless oil (69%); (EtOAc/petroleum ether = 1:8); ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.93 (s, 1H), 5.90 (dd, *J* = 17.5, 10.7 Hz, 1H), 4.98 – 4.86 (m, 2H), 2.19 (s, 2H), 1.07 (s, 6H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 172.52, 147.18, 110.49, 46.02, 35.40, 26.59. HRMS (ESI) *m*/*z*: $C_7H_{12}O_2$ [M−H]$^-$ Cal: 127.0765; Found: 127.0767.



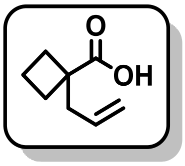
5-methyl-2,2-diphenylhex-4-enoic acid **(3n).** White solid (67%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.74 (s, 1H), 7.31 – 7.20 (m, 10H), 5.03 (td, *J* = 6.8, 3.6 Hz, 1H), 3.03 (d, *J* = 6.9 Hz, 2H), 1.52 (s, 3H), 1.24 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 175.07, 143.09, 133.43, 128.71, 127.55, 126.32, 120.12, 59.79, 36.37, 25.66, 17.36. HRMS (ESI) *m*/*z*: $C_{19}H_{20}O_2$ [M + H]$^+$ Cal: 281.1536; Found: 281.1564.



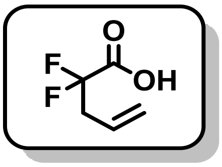
2-allyl-2,3-dihydro-1H-indene-2-carboxylic acid **(3o).** White solid (50%); (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.17 (dt, *J* = 7.4, 3.6 Hz, 2H), 7.14 – 7.04 (m, 2H), 5.72 (ddt, *J* = 17.5, 10.4, 7.2 Hz, 1H), 5.14 – 4.97 (m, 2H), 3.34 (s, 1H), 3.30 (s, 1H), 2.86 (d, *J* = 16.2 Hz, 2H), 2.41 (d, *J* = 7.3 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 177.34, 141.17, 134.47, 126.34, 124.27, 117.92, 52.92, 42.02, 41.19. HRMS (ESI) *m/z*: $C_{13}H_{14}O_2$ [M−H]$^-$ Cal: 201.0921; Found: 201.0922.



1-allylcyclopropane-1-carboxylic acid **(3p).** Colorless oil (47%); (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.10 (s, 1H), 5.83 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.06 – 4.94 (m, 2H), 2.23 (d, *J* = 6.7 Hz, 2H), 1.03 (q, *J* = 3.7 Hz, 2H), 0.70 (q, *J* = 3.8 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 175.89, 136.14, 116.18, 36.62, 22.21, 14.03. HRMS (ESI) *m/z*: $C_7H_{10}O_2$ [M−H]$^-$ Cal: 125.0608; Found: 125.0608.

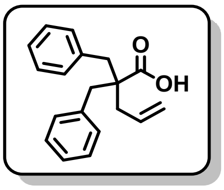


1-allylcyclobutane-1-carboxylic acid **(3q).** Colorless oil (53%); (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, DMSO-d_6) δ 5.68 (ddt, *J* = 17.2, 10.2, 7.0 Hz, 1H), 5.12 – 4.99 (m, 2H), 2.43 (d, *J* = 7.0 Hz, 2H), 2.27 (td, *J* = 9.9, 8.7, 4.0 Hz, 2H), 1.90 – 1.74 (m, 4H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 177.44, 134.25, 117.41, 46.21, 41.17, 28.75, 14.75. HRMS (ESI) *m/z*: $C_8H_{12}O_2$ [M−H]$^-$ Cal: 139.0765; Found: 139.0765.

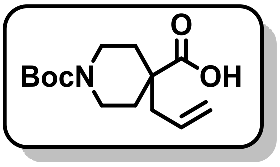


2,2-difluoropent-4-enoic acid **(3r).** Yellow oil (66%); (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400

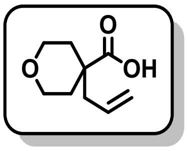
MHz, DMSO-d_6) δ 5.71 (ddt, *J* = 17.2, 10.0, 7.0 Hz, 1H), 5.27 (t, *J* = 13.8 Hz, 2H), 2.85 (td, *J* = 17.0, 7.0 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.04, 164.73, 164.42, 127.72, 127.66, 127.61, 121.58, 118.03, 115.55, 113.07, 38.46, 38.23, 37.99. HRMS (ESI) *m*/*z*: $C_5H_6F_2O_2$ [M−H]$^-$ Cal: 135.0263; Found: 135.0262.



2,2-dibenzylpent-4-enoic acid **(3s).** Yellow oil (89%); (EtOAc/petroleum ether = 1:4); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.64 (s, 1H), 7.30 – 7.16 (m, 10H), 6.05 (ddt, *J* = 17.1, 10.1, 6.8 Hz, 1H), 5.26 – 5.05 (m, 2H), 3.01 (d, *J* = 13.6 Hz, 2H), 2.82 (d, *J* = 13.7 Hz, 2H), 2.08 (d, *J* = 6.9 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 181.36, 142.68, 139.72, 135.15, 133.23, 131.62, 123.88, 56.03, 46.67, 40.35. HRMS (ESI) *m*/*z*: $C_{19}H_{20}O_2$ [M−H]$^-$ Cal: 279.1391; Found: 279.1391.

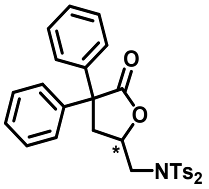


4-allyl-1-(tert-butoxycarbonyl)piperidine-4-carboxylic acid **(3t).** White solid (60%); (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.51 (s, 1H), 5.67 (ddt, *J* = 17.4, 10.1, 7.4 Hz, 1H), 5.09 – 5.01 (m, 2H), 3.69 (dt, *J* = 14.0, 4.1 Hz, 2H), 2.87 (s, 2H), 2.23 (d, *J* = 7.3 Hz, 2H), 1.88 (dt, *J* = 13.4, 3.6 Hz, 2H), 1.38 (s, 9H), 1.28 (ddd, *J* = 13.9, 11.0, 4.2 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 176.12, 153.86, 133.32, 118.14, 78.52, 44.59, 43.03, 32.36, 28.04. HRMS (ESI) *m*/*z*: $C_{14}H_{23}NO_4$ [M−H]$^-$ Cal: 268.1554; Found: 268.1554.

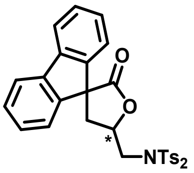


4-allyltetrahydro-2H-pyran-4-carboxylic acid **(3u).** Pale yellow oil (65%); (EtOAc/petroleum ether = 1:4); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.46 (s, 1H), 5.68 (ddt, *J* = 17.5, 10.5, 7.4 Hz, 1H), 5.09 – 4.95 (m, 2H), 3.70 (dt, *J* = 11.9, 3.9 Hz, 2H), 3.32 (td, *J* = 11.3, 2.5 Hz, 2H), 2.23 (d, *J* = 7.4 Hz, 2H), 1.87 (d, *J* = 13.7 Hz, 2H), 1.40 (ddd, *J* = 14.4, 10.8, 4.5 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 176.31, 133.23, 118.03, 64.36, 43.85, 43.67, 33.28. HRMS (ESI) *m*/*z*: $C_9H_{14}O_3$ [M−H]$^-$ Cal: 169.0870; Found: 169.0870.

7.3 Data characterization and chiral results of aminolactone products

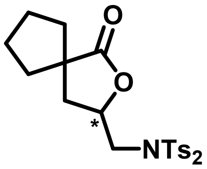


4-methyl-N-((5-oxo-4,4-diphenyltetrahydrofuran-2-yl)methyl)-N-tosylbenzenesulfonamide **(4a)**. TLC (PE:EA = 4:1), $R_f = 0.54$, Pale yellow solid, yield 83 %; (EtOAc/petroleum ether = 1:5);^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, $J = 8.0$ Hz, 4H), 7.24 – 7.11 (m, 14H), 4.51 (dq, $J = 10.7, 5.4$ Hz, 1H), 4.07 (dd, $J = 15.7, 6.3$ Hz, 1H), 3.78 (dd, $J = 15.7, 5.0$ Hz, 1H), 2.86 (dd, $J = 13.1, 5.1$ Hz, 1H), 2.59 (dd, $J = 13.2, 10.3$ Hz, 1H), 2.34 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.96, 145.36, 141.46, 139.46, 136.32, 129.77, 128.96, 128.58, 128.44, 127.81, 127.67, 127.35, 127.23, 57.76, 51.19, 40.97, 21.71. HRMS (ESI) *m/z*: $C_{31}H_{29}NO_6S_2$ $[M+H]^+$ Calcd for: 576.1509; Found: 576.1503. $[\alpha]_D^{25}$ -6.312 (c = 1.0, $CHCl_3$). Enantiomeric excess: 99%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 11.326 min (minor), t_R = 13.337 min (major).

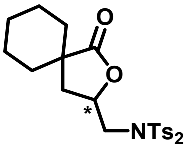


4-methyl-N-((2'-oxo-4',5'-dihydro-2'H-spiro[fluorene-9,3'-furan]-5'-yl)methyl)-N-tosylbenzenesulfonamide **(4b)**. TLC (PE:EA = 3:1), $R_f = 0.41$， White solid, yield of 48 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (d, $J = 8.2$ Hz, 4H), 7.73 (d, $J = 7.6$ Hz, 2H), 7.44 – 7.39 (m, 2H), 7.32 (dt, $J = 19.3, 7.8$ Hz, 8H), 5.29 (dtd, $J = 10.8, 6.7, 4.3$ Hz, 1H), 4.38 (dd, $J = 15.8, 7.0$ Hz, 1H), 3.97 (dd, $J = 15.8, 4.5$ Hz, 1H), 2.71 (dd, $J = 13.5, 10.0$ Hz, 1H), 2.60 (dd, $J = 13.5, 6.5$ Hz, 1H), 2.43 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.68, 145.52, 145.48, 144.70, 141.44, 140.62, 136.23, 129.82, 128.96, 128.93, 128.82, 128.34, 128.05, 123.60, 122.81, 120.69, 120.41, 58.48, 52.10, 38.95, 21.71. HRMS (ESI) *m/z*: $C_{31}H_{27}NO_6S_2$ $[M+H]^+$ Calcd for: 574.1353; Found: 574.1352. $[\alpha]_D^{25}$ -61.964 (c = 1.0, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiracel-

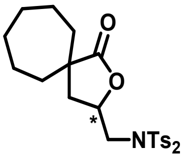
IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.2 mL/min, T = 50℃, 245 nm): t$_R$ = 8.034 min (major), t$_R$ = 11.584 min (minor).



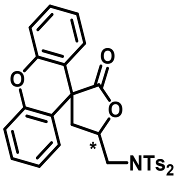
4-methyl-N-((1-oxo-2-oxaspiro[4.4]nonan-3-yl)methyl)-N-tosylbenzenesulfonamid **(4c)**. TLC (PE:EA = 5:1), R$_f$ = 0.45，Pale yellow solid, yield 70 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.5 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.74 – 4.63 (m, 1H), 4.11 (dd, *J* = 15.7, 7.3 Hz, 1H), 3.70 (dd, *J* = 15.8, 4.5 Hz, 1H), 2.46 (s, 6H), 2.17 – 2.07 (m, 2H), 1.87 – 1.75 (m, 4H), 1.72 – 1.57 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 181.20, 145.33, 136.28, 129.69, 128.75, 51.71, 49.79, 40.26, 37.41, 36.87, 25.41, 25.35, 21.69. HRMS (ESI) *m*/*z*: $C_{23}H_{27}NO_6S_2$ [M + H]$^+$ Calcd for: 478.1353; Found: 478.1354. $[\alpha]_D^{25}$ -2.798 (c = 1.0, CHCl$_3$). Enantiomeric excess: 64%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t$_R$ = 8.758 min (minor), t$_R$ = 10.826 min (major).



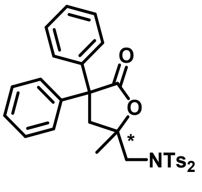
4-methyl-N-((1-oxo-2-oxaspiro[4.5]decan-3-yl)methyl)-N-tosylbenzenesulfonamide **(4d)**. TLC (PE:EA = 5:1), R$_f$ = 0.47, Pale yellow solid, yield 63 %; (EtOAc/petroleum ether = 1:5);^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 6.6 Hz, 4H), 7.36 (d, *J* = 7.9 Hz, 4H), 4.70 (ddt, *J* = 10.3, 8.0, 5.4 Hz, 1H), 4.15 – 4.04 (m, 1H), 3.75 – 3.65 (m, 1H), 2.46 (s, 6H), 2.29 – 2.21 (m, 1H), 1.72 (dq, *J* = 44.4, 12.2, 11.3 Hz, 6H), 1.44 (d, *J* = 13.0 Hz, 1H), 1.41 – 1.12 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 180.27, 145.33, 136.31, 129.69, 128.76, 52.09, 44.60, 36.69, 34.09, 31.71, 25.20, 22.09, 22.01, 21.69. HRMS (ESI) *m*/*z*: $C_{24}H_{29}NO_6S_2$ [M + H]$^+$ Calcd for: 492.1509; Found: 492.1510. $[\alpha]_D^{25}$ +6.555 (c = 1.0, CHCl$_3$). Enantiomeric excess: 66%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t$_R$ = 9.218 min (minor), t$_R$ = 10.443 min (major).



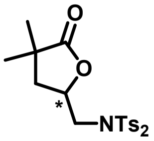
***4-methyl-N-((1-oxo-2-oxaspiro[4.6]undecan-3-yl)methyl)-N-tosylbenzenesulfonamide* (4e)**. TLC (PE:EA = 3:1), $R_f = 0.56$, Pale yellow solid, yield 67 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, $J = 8.0$ Hz, 4H), 7.35 (d, $J = 8.0$ Hz, 4H), 4.68 (dtd, $J = 10.5, 6.7, 4.2$ Hz, 1H), 4.14 – 4.04 (m, 1H), 3.69 (dd, $J = 15.7, 4.5$ Hz, 1H), 2.46 (s, 6H), 2.16 (dd, $J = 12.9, 6.2$ Hz, 1H), 1.95 (ddd, $J = 14.5, 10.5, 2.0$ Hz, 1H), 1.84 – 1.72 (m, 2H), 1.71 – 1.66 (m, 2H), 1.63 (s, 2H), 1.60 – 1.54 (m, 2H), 1.54 – 1.47 (m, 2H), 1.44 – 1.32 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 181.17, 145.32, 136.31, 129.68, 128.75, 52.02, 47.11, 39.28, 36.96, 35.45, 29.27, 29.15, 23.57, 23.50, 21.69. HRMS (ESI) *m*/*z*: $C_{25}H_{31}NO_6S_2$ $[M + H]^+$ Calcd for: 506.1666; Found: 506.1670. $[\alpha]_D^{25}$ +7.682 (c = 1.0, $CHCl_3$). Enantiomeric excess: 73%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 8.995 min (minor), t_R = 11.957 min (major).



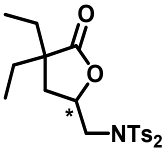
***4-methyl-N-((2-oxo-4,5-dihydro-2H-spiro[furan-3,9'-xanthen]-5-yl)methyl)-N-tosylbenzenesulfonamide* (4f)**. TLC (PE:EA = 3:1), $R_f = 0.49$, Yellow solid, yield of 27 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, $J = 8.1$ Hz, 4H), 7.37 – 7.28 (m, 6H), 7.19 – 7.00 (m, 6H), 5.05 – 4.86 (m, 1H), 4.28 (dd, $J = 15.8, 7.2$ Hz, 1H), 3.78 (dd, $J = 15.7, 4.6$ Hz, 1H), 2.56 (dd, $J = 13.5, 6.4$ Hz, 1H), 2.46 (s, 6H), 2.22 (dd, $J = 13.5, 9.8$ Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.90, 151.50, 150.71, 145.47, 136.22, 129.76, 129.34, 129.13, 128.75, 127.12, 125.57, 124.30, 124.05, 122.88, 122.18, 117.50, 116.76, 51.66, 49.42, 46.68, 21.70. HRMS (ESI) *m*/*z*: $C_{31}H_{27}NO_7S_2$ $[M + H]^+$ Calcd for: 590.1302; Found: 590.1302. $[\alpha]_D^{25}$ +1.015(c = 1.0, $CHCl_3$). Enantiomeric excess: 81%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 245 nm): t_R = 15.583 min (major), t_R = 17.248 min (minor).



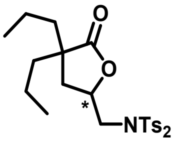
4-methyl-N-((2-methyl-5-oxo-4,4-diphenyltetrahydrofuran-2-yl)methyl)-N-tosylbenzenesulfonamide **(4g)**. TLC (PE:EA = 5:1), R_f = 0.48, White solid, yield 59 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 – 7.91 (m, 4H), 7.36 – 7.30 (m, 4H), 7.26 – 7.19 (m, 8H), 7.12 – 7.05 (m, 2H), 4.23 (d, *J* = 16.4 Hz, 1H), 3.93 (d, *J* = 16.3 Hz, 1H), 3.20 (d, *J* = 13.9 Hz, 1H), 2.92 (d, *J* = 13.8 Hz, 1H), 2.37 (s, 6H), 1.13 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.64, 145.09, 142.58, 142.19, 136.75, 129.63, 128.88, 128.63, 128.38, 127.33, 127.26, 126.98, 82.91, 57.77, 56.20, 44.84, 24.89, 21.67. HRMS (ESI) *m*/*z*: $C_{32}H_{31}NO_6S_2$ [M + H]$^+$ Calcd for: 590.1666; Found: 590.1660. $[\alpha]_D^{25}$ +1.870 (c = 1.0, $CHCl_3$). Enantiomeric excess: 97%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 6.428 min (minor), t_R = 8.737 min (major).



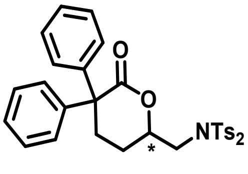
N-((4,4-dimethyl-5-oxotetrahydrofuran-2-yl)methyl)-4-methyl-N-tosylbenzenesulfonamide **(4h)**. TLC (PE:EA = 3:1), R_f = 0.42, White solid, yield 68 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.0 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.73 (tq, *J* = 10.4, 5.7, 4.8 Hz, 1H), 4.10 (dd, *J* = 15.8, 7.3 Hz, 1H), 3.70 (dd, *J* = 15.9, 4.1 Hz, 1H), 2.46 (s, 6H), 2.10 (dd, *J* = 12.9, 6.2 Hz, 1H), 1.77 (dd, *J* = 12.8, 9.8 Hz, 1H), 1.23 (s, 3H), 1.20 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 179.62, 144.32, 135.22, 128.67, 127.74, 74.25, 50.87, 39.62, 39.12, 23.91, 23.38, 20.68. HRMS (ESI) *m*/*z*: $C_{21}H_{25}NO_6S_2$ [M + H]$^+$ Calcd for: 452.1196; Found: 452.1191. $[\alpha]_D^{25}$ -3.792 (c = 1.0, $CHCl_3$). Enantiomeric excess: 62%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 7.323 min (minor), t_R = 9.637 min (major).



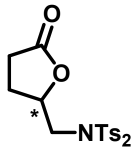
N-((4,4-diethyl-5-oxotetrahydrofuran-2-yl)methyl)-4-methyl-N-tosylbenzenesulfonamide **(4i)**. TLC (PE:EA = 3:1), R_f = 0.45, Pale yellow solid, yield 62 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.1 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.70 (dtd, *J* = 14.5, 7.2, 4.7 Hz, 1H), 4.13 – 4.03 (m, 1H), 3.67 (dd, *J* = 15.7, 4.7 Hz, 1H), 2.46 (s, 6H), 2.05 – 1.99 (m, 1H), 1.84 (dd, *J* = 13.3, 9.2 Hz, 1H), 1.60 – 1.54 (m, 4H), 0.88 (td, *J* = 7.5, 2.0 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.66, 144.31, 135.24, 128.66, 127.74, 51.24, 47.40, 33.81, 28.07, 27.27, 20.67, 7.60. HRMS (ESI) *m/z*: $C_{23}H_{29}NO_6S_2$ $[M + H]^+$ Calcd for: 480.1509; Found: 480.1516. $[\alpha]_D^{25}$ +1.659 (c = 1.0, $CHCl_3$). Enantiomeric excess: 33%, determined by HPLC (Chiralcel OD-H, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 254 nm): t_R = 8.078 min (minor), t_R = 9.248 min (major).



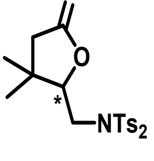
4-methyl-N-((5-oxo-4,4-dipropyltetrahydrofuran-2-yl)methyl)-N-tosylbenzenesulfonamide **(4j)**. TLC (PE:EA = 3:1), R_f = 0.53, Pale yellow oily, yield 70 %; (EtOAc/petroleum ether = 1:5);^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.89 (m, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.67 (dtd, *J* = 14.5, 7.1, 4.6 Hz, 1H), 4.08 (td, *J* = 15.5, 7.2 Hz, 1H), 3.67 (dd, *J* = 15.7, 4.6 Hz, 1H), 2.46 (s, 6H), 2.06 – 2.00 (m, 1H), 1.85 (dd, *J* = 13.2, 9.4 Hz, 1H), 1.55 – 1.42 (m, 4H), 1.39 – 1.20 (m, 4H), 0.90 (dt, *J* = 10.1, 7.2 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.84, 144.30, 135.27, 128.66, 127.74, 51.23, 46.74, 38.03, 37.36, 34.75, 20.67, 16.59, 16.56, 13.36, 13.30. HRMS (ESI) *m/z*: $C_{25}H_{33}NO_6S_2$ $[M + H]^+$ Calcd for: 508.1822; Found: 508.1823. $[\alpha]_D^{25}$ +2.309 (c = 1.0, $CHCl_3$). Enantiomeric excess: 55%, determined by HPLC (Chiralcel OD-H, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 260 nm): t_R = 6.670 min (minor), t_R = 7.547 min (major).



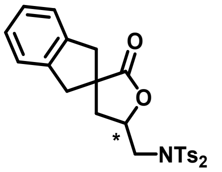
4-methyl-N-((6-oxo-5,5-diphenyltetrahydro-2H-pyran-2-yl)methyl)-N-tosylbenzenesulfonamide **(4k)**. TLC (PE:EA = 3:1), R_f = 0.51, Pale yellow solid, yield 67 %; (EtOAc/petroleum ether = 1:5);^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.1 Hz, 4H), 7.36 – 7.25 (m, 10H), 7.23 – 7.13 (m, 4H), 4.77 – 4.66 (m, 1H), 4.09 (td, *J* = 15.7, 15.1, 7.3 Hz, 1H), 3.50 (dd, *J* = 15.7, 4.3 Hz, 1H), 2.71 (ddd, *J* = 14.4, 9.3, 5.1 Hz, 1H), 2.57 (dt, *J* = 14.3, 5.7 Hz, 1H), 2.44 (s, 6H), 1.90 (dq, *J* = 11.2, 5.4 Hz, 1H), 1.64 (s, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.10, 145.19, 142.51, 141.48, 136.12, 129.62, 128.93, 128.50, 128.19, 128.15, 127.52, 127.30, 56.89, 51.93, 31.54, 22.92, 21.69. HRMS (ESI) *m*/*z*: $C_{32}H_{31}NO_6S_2$ [M + H]$^+$Calcd for: 590.1666; Found: 590.1667. $[\alpha]_D^{25}$ -11.636 (c = 1.0, $CHCl_3$). Enantiomeric excess: 92%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 10.507 min (minor), t_R = 20.464 min (major).



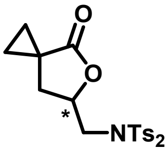
4-methyl-N-((5-oxotetrahydrofuran-2-yl)methyl)-N-tosylbenzenesulfonamide **(4l)**. TLC (PE:EA = 2:1), R_f = 0.37, White solid, yield 62 %; (EtOAc/petroleum ether = 1:4);^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.2 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.83 (dt, *J* = 12.2, 5.8 Hz, 1H), 4.07 (dd, *J* = 15.8, 7.5 Hz, 1H), 3.66 (dd, *J* = 15.8, 4.7 Hz, 1H), 2.52 (q, *J* = 8.5, 7.4 Hz, 2H), 2.46 (s, 6H), 2.28 (q, *J* = 7.1, 6.3 Hz, 1H), 2.03 – 1.92 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.86, 145.39, 136.15, 129.71, 128.75, 51.21, 28.03, 25.22, 21.71. HRMS (ESI) *m*/*z*: $C_{19}H_{21}NO_6S_2$ [M + H]$^+$ Calcd for: 424.0883; Found: 424.0882. $[\alpha]_D^{25}$ +4.893 (c = 1.0, $CHCl_3$). Enantiomeric excess: 55%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 245 nm): t_R = 8.703 min (minor), t_R = 15.250 min (major).



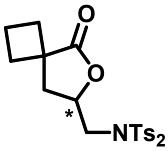
N-((3,3-dimethyl-5-oxotetrahydrofuran-2-yl)methyl)-4-methyl-N-tosylbenzenesulfonamide **(4m)**. TLC (PE:EA = 3:1), R_f = 0.52, White solid, yield of 53 %; (EtOAc/petroleum ether = 1:3);^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 8.2 Hz, 4H), 7.37 (d, J = 8.0 Hz, 4H), 4.51 – 4.43 (m, 1H), 4.15 (dd, J = 16.1, 9.6 Hz, 1H), 3.55 (dd, J = 16.2, 1.7 Hz, 1H), 2.46 (s, 6H), 2.38 (d, J = 16.9 Hz, 1H), 2.33 (s, 1H), 1.24 (s, 3H), 1.06 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.70, 145.25, 136.28, 129.62, 128.91, 86.59, 48.08, 44.25, 38.92, 25.24, 21.70, 21.04. HRMS (ESI) *m*/*z*: $C_{21}H_{25}NO_6S_2$ [M + H]$^+$Calcd for: 452.1196; Found: 452.1197. $[\alpha]_D^{25}$ +5.172 (c = 1.0, $CHCl_3$). Enantiomeric excess: 64%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 260 nm): t_R = 5.136 min (minor), t_R = 7.882 min (major).



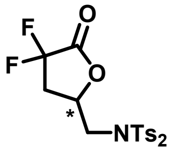
4-methyl-N-((2-oxo-1',3',4,5-tetrahydro-2H-spiro[furan-3,2'-inden]-5-yl)methyl)-N-tosylbenzenesulfonamide **(4o)**. TLC (PE:EA = 3:1), R_f = 0.5, White solid, yield of 78 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.93 (m, 4H), 7.36 (d, J = 8.2 Hz, 4H), 7.19 (dq, J = 8.6, 3.2, 2.2 Hz, 4H), 4.86 – 4.75 (m, 1H), 4.21 – 4.11 (m, 1H), 3.72 (dd, J = 15.8, 4.4 Hz, 1H), 3.59 (d, J = 16.0 Hz, 1H), 3.27 (d, J = 15.6 Hz, 1H), 2.91 (dd, J = 37.5, 15.8 Hz, 2H), 2.46 (s, 6H), 2.30 (dd, J = 12.9, 6.0 Hz, 1H), 1.90 (dd, J = 13.0, 9.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.60, 144.37, 139.66, 138.29, 135.14, 128.69, 127.72, 126.15, 125.99, 123.48, 123.43, 50.56, 49.58, 42.35, 39.58, 20.68. HRMS (ESI) *m*/*z*: $C_{27}H_{27}NO_6S_2$ [M + H]$^+$Calcd for: 526.1353; Found: 526.1353. $[\alpha]_D^{25}$ +9.819 (c = 1.0, $CHCl_3$). Enantiomeric excess: 94%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 15.002 min (minor), t_R = 19.988 min (major).



4-methyl-N-((4-oxo-5-oxaspiro[2.4]heptan-6-yl)methyl)-N-tosylbenzenesulfonamide **(4p)**. TLC (PE:EA = 3:1), R_f = 0.31; Pale yellow solid, yield 61 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, J = 8.0 Hz, 4H), 7.36 (d, J = 8.0 Hz, 4H), 4.94 (p, J = 6.8 Hz, 1H), 4.13 (dd, J = 15.5, 7.4 Hz, 1H), 3.71 (dd, J = 15.6, 5.1 Hz, 1H), 2.46 (s, 6H), 2.30 (dd, J = 12.9, 7.9 Hz, 1H), 2.10 (dd, J = 13.0, 6.2 Hz, 1H), 1.32 – 1.22 (m, 2H), 1.02 – 0.92 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.69, 145.34, 136.20, 129.70, 128.74, 51.54, 32.64, 21.70, 19.79, 15.23, 15.07. HRMS (ESI) *m/z*: $C_{21}H_{23}NO_6S_2$ [M + H]$^+$ Calcd for: 450.1040; Found: 450.1035. $[\alpha]_D^{25}$ +1.021 (c = 1.0, $CHCl_3$). Enantiomeric excess: 29%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 260 nm): t_R = 9.274 min (minor), t_R = 11.457 min (major).

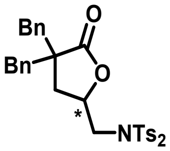


4-methyl-N-((5-oxo-6-oxaspiro[3.4]octan-7-yl)methyl)-N-tosylbenzenesulfonamide **(4q)**. TLC (PE:EA = 3:1), R_f = 0.39; Pale yellow solid, yield 57 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.2 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.66 (qd, *J* = 7.4, 4.7 Hz, 1H), 4.02 (dd, *J* = 15.8, 7.5 Hz, 1H), 3.64 (dd, *J* = 15.7, 4.6 Hz, 1H), 2.58 – 2.52 (m, 1H), 2.46 (s, 6H), 2.39 (dd, *J* = 12.9, 6.4 Hz, 2H), 2.13 (qd, *J* = 8.8, 7.7, 3.5 Hz, 1H), 2.06 – 1.97 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 179.88, 145.33, 136.22, 129.68, 128.76, 51.52, 43.87, 39.06, 31.31, 29.84, 21.69, 16.41. HRMS (ESI) *m/z*: $C_{22}H_{25}NO_6S_2$ [M + H]$^+$ Calcd for: 464.1196; Found: 464.1199. $[\alpha]_D^{25}$ +1.539 (c = 1.0, $CHCl_3$). Enantiomeric excess: 31%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 254 nm): t_R = 8.891 min (minor), t_R = 10.793 min (major).

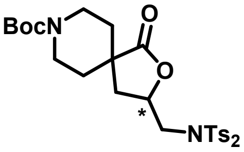


N-((4,4-difluoro-5-oxotetrahydrofuran-2-yl)methyl)-4-methyl-N-tosylbenzenesulfonamide **(4r)**. TLC (PE:EA = 3:1), R_f = 0.55, Colorless oily, yield 45 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.0 Hz, 4H), 7.37 (d, *J* = 8.0 Hz, 4H), 4.95 (qd, *J* = 7.2, 4.6 Hz, 1H), 4.14 (dd, *J* = 16.2, 7.4 Hz, 1H), 3.77 (dd, *J* = 16.0, 4.6 Hz, 1H), 2.78 (tdd, *J* = 15.0, 9.1, 6.7 Hz, 1H), 2.47 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.92, 145.80, 135.74, 129.88, 128.69, 50.76, 35.42, 35.20, 34.98, 21.73. HRMS (ESI) *m/z*: $C_{19}H_{19}F_2NO_6S_2$ [M + H]$^+$ Calcd for: 460.0695; Found: 460.0696. $[\alpha]_D^{25}$ -2.814 (c = 1.0, $CHCl_3$). Enantiomeric excess: 29%, determined by HPLC

(Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 235 nm): t_R = 5.980 min (minor), t_R = 10.743 min (major).

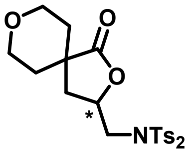


N-((4,4-dibenzyl-5-oxotetrahydrofuran-2-yl)methyl)-4-methyl-N-tosylbenzenesulfonamide **(4s)**. TLC (PE:EA = 4:1), R_f = 0.55, White solid, yield of 72 %; (EtOAc/petroleum ether = 1:8); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 8.2 Hz, 4H), 7.31 (d, *J* = 9.1 Hz, 5H), 7.26 (d, *J* = 13.7 Hz, 5H), 7.18 (dt, *J* = 7.6, 3.6 Hz, 4H), 3.72 – 3.61 (m, 1H), 3.36 (dd, *J* = 15.7, 6.8 Hz, 1H), 3.19 (d, *J* = 13.7 Hz, 1H), 3.16 – 3.07 (m, 2H), 2.73 (dd, *J* = 17.4, 13.5 Hz, 2H), 2.44 (s, 6H), 2.14 (dd, *J* = 13.6, 7.5 Hz, 1H), 1.92 (dd, *J* = 13.6, 9.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 179.75, 145.11, 136.52, 136.19, 135.93, 130.44, 129.90, 129.59, 128.70, 128.57, 127.44, 127.09, 51.77, 51.42, 44.36, 43.46, 32.70, 21.69. HRMS (ESI) *m/z*: $C_{33}H_{33}NO_6S_2$ [M + H]$^+$Calcd for: 604.1822; Found: 604.1823. $[\alpha]_D^{25}$ +4.350 (c = 1.0, CHCl$_3$). Enantiomeric excess: 89%, determined by HPLC (Chiralcel OD-H, *n*-hexane/2-propanol = 80/20, flow rate 1.2 mL/min, T = 40℃, 214 nm): t_R = 11.963 min (major), t_R = 19.588 min (minor).

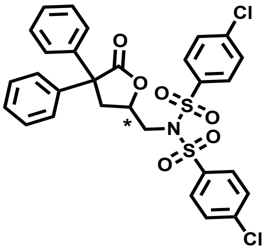


tert-butyl-3-(((4-methyl-N-tosylphenyl)sulfonamido)methyl)-1-oxo-2-oxa-8-azaspiro[4.5]decane-8-carboxylate **(4t)**. TLC (PE:EA = 2:1), R_f = 0.49, White solid, yield of 67 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (dd, *J* = 8.5, 2.2 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.76 (s, 1H), 4.11 (dd, *J* = 15.8, 7.1 Hz, 1H), 3.94 (s, 1H), 3.80 (s, 1H), 3.72 (dd, *J* = 15.8, 4.5 Hz, 1H), 3.06 (dt, *J* = 24.0, 11.5 Hz, 2H), 2.47 (s, 6H), 2.24 (dd, *J* = 13.0, 6.4 Hz, 1H), 1.89 (t, *J* = 10.6 Hz, 1H), 1.75 (d, *J* = 10.9 Hz, 2H), 1.53 (d, *J* = 17.8 Hz, 2H), 1.46 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.67, 153.57, 144.44, 135.14, 128.71, 127.72, 78.90, 50.79, 41.62, 35.68, 32.24, 30.51, 27.39, 20.68. HRMS (ESI) *m/z*: $C_{28}H_{36}N_2O_8S_2$ [M + H]$^+$ Calcd for: 593.1986; Found: 593.1996. $[\alpha]_D^{25}$ +5.967 (c = 0.5, CHCl$_3$). Enantiomeric excess: 79%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-

propanol = 80/20, flow rate 1.2 mL/min, T = 50℃, 235 nm): t_R = 11.128 min (minor), t_R = 13.365 min (major).

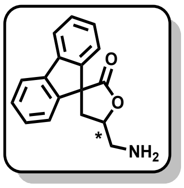


4-methyl-N-((1-oxo-2,8-dioxaspiro[4.5]decan-3-yl)methyl)-N-tosylbenzenesulfonamide **(4u)**. TLC (PE:EA = 1:1), R_f = 0.57, White solid, yield of 65 %; (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.0 Hz, 4H), 7.36 (d, *J* = 8.0 Hz, 4H), 4.76 (dtd, *J* = 10.7, 6.7, 4.3 Hz, 1H), 4.15 – 4.07 (m, 1H), 4.02 (dt, *J* = 11.9, 4.5 Hz, 1H), 3.89 (dt, *J* = 12.1, 4.5 Hz, 1H), 3.72 (dd, *J* = 15.8, 4.5 Hz, 1H), 3.49 (dddd, *J* = 21.8, 12.2, 9.5, 2.9 Hz, 2H), 2.46 (s, 6H), 2.32 (dd, *J* = 13.1, 6.3 Hz, 1H), 2.03 (dt, *J* = 9.9, 7.4 Hz, 1H), 1.90 – 1.75 (m, 2H), 1.45 (tdd, *J* = 17.9, 14.9, 6.9, 3.3 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.60, 145.45, 136.17, 129.72, 128.75, 63.92, 63.60, 51.83, 41.88, 37.20, 33.62, 32.02, 21.70. HRMS (ESI) *m/z*: $C_{23}H_{27}NO_7S_2$ [M + H]$^+$ Calcd for: 494.1302; Found: 494.1297. $[\alpha]_D^{25}$ -7.384 (c = 1.0, $CHCl_3$). Enantiomeric excess: 89%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 245 nm): t_R = 12.640 min (minor), t_R = 17.126 min (major).

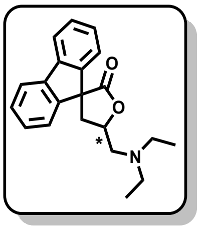


4-chloro-N-((4-chlorophenyl)sulfonyl)-N-((5-oxo-4,4-diphenyltetrahydrofuran-2-yl)methyl)benzenesulfonamide **(4a')**. TLC (PE:EA = 3:1), R_f = 0.69, White solid, yield of 77 %; (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.3 Hz, 4H), 7.40 (d, *J* = 8.3 Hz, 4H), 7.18 (dq, *J* = 32.6, 7.8 Hz, 10H), 4.48 (h, *J* = 4.6 Hz, 1H), 4.05 (td, *J* = 15.1, 14.4, 7.1 Hz, 1H), 3.74 (dd, *J* = 15.8, 3.9 Hz, 1H), 2.86 (dd, *J* = 13.1, 5.2 Hz, 1H), 2.54 (dd, *J* = 13.2, 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 175.73, 141.25, 141.18, 139.30, 137.45, 130.17, 129.54, 129.06, 128.56, 127.96, 127.58, 127.52, 127.16, 57.68, 51.74, 40.82. HRMS (ESI) *m/z*: $C_{29}H_{23}Cl_2NO_6S_2$ [M + H]$^+$Calcd for: 616.0417; Found:

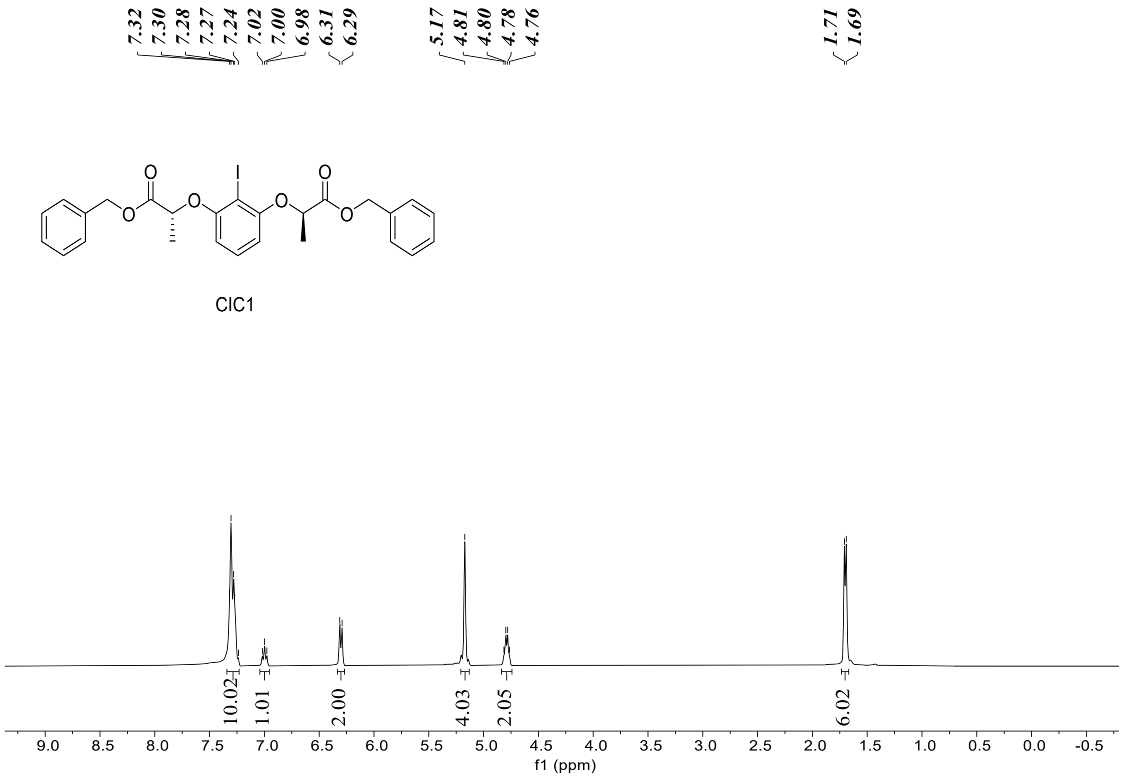
616.0416. $[\alpha]_D^{25}$ -5.913 (c = 1.0, $CHCl_3$). Enantiomeric excess: 83%, determined by HPLC (Chiracel-IA$_3$, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 254 nm): t_R = 11.763 min (minor), t_R = 22.577 min (major).



***5'-(aminomethyl)-4',5'-dihydro-2'H-spiro[fluorene-9,3'-furan]-2'-one* (5b).** TLC (DCM:MeOH = 10:1), R_f = 0.32, （DCM/MeOH = 10:1）, Yellow solid, yield of 68 %. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.82 (dd, *J* = 17.6, 8.3 Hz, 3H), 7.60 (d, *J* = 7.0 Hz, 2H), 7.43 – 7.34 (m, 2H), 7.32 (s, 1H), 5.25 (s, 1H), 4.48 (s, 1H), 3.59 (d, *J* = 11.0 Hz, 1H), 3.34 (s, 2H), 2.34 (t, *J* = 11.7 Hz, 1H), 1.89 (d, *J* = 12.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 170.31, 150.79, 150.47, 140.87, 140.70, 128.05, 127.95, 127.76, 127.67, 124.64, 123.86, 120.52, 120.18, 62.00, 57.31, 48.91, 42.42. HRMS (ESI) *m/z*: $C_{17}H_{15}NO_2$ [M + H]$^+$ Calcd for: 266.1176; Found: 266.1175. $[\alpha]_D^{25}$ -53.294 (c = 0.6, $CHCl_3$).



***5'-((diethylamino)methyl)-4',5'-dihydro-2'H-spiro[fluorene-9,3'-furan]-2'-one* (M').** TLC (DCM:MeOH = 10:1), R_f = 0.54, （DCM/MeOH = 15:1）, light yellow solid, yield of 88 %. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.91 (dd, *J* = 7.5, 3.7 Hz, 2H), 7.68 (dd, *J* = 20.8, 7.5 Hz, 2H), 7.50 – 7.34 (m, 4H), 5.24 (dq, *J* = 10.8, 5.7 Hz, 1H), 2.98 – 2.84 (m, 2H), 2.79 (dd, *J* = 13.2, 9.9 Hz, 1H), 2.70 – 2.57 (m, 5H), 1.03 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 176.48, 146.27, 145.67, 140.93, 139.94, 128.61, 128.53, 128.14, 128.10, 123.96, 123.24, 120.55, 120.32, 77.60, 58.29, 56.77, 47.31, 38.05, 11.90. HRMS (ESI) *m/z*: $C_{21}H_{23}NO_2$ [M + H]$^+$ Calcd for: 322.1802; Found: 322.1799. $[\alpha]_D^{25}$ -37.812 (c = 0.5, $CHCl_3$). Enantiomeric excess: 20%, determined by HPLC (Chiralpak AD-H, *n*-hexane/2-propanol = 80/20, flow rate 1.0 mL/min, T = 30℃, 230 nm): t_R = 9.880 min (major), t_R = 13.645 min (minor).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4.76
1.71
1.69
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I
O
O
O
O
O
10.02
1.01
2.00
4.03
2.05
6.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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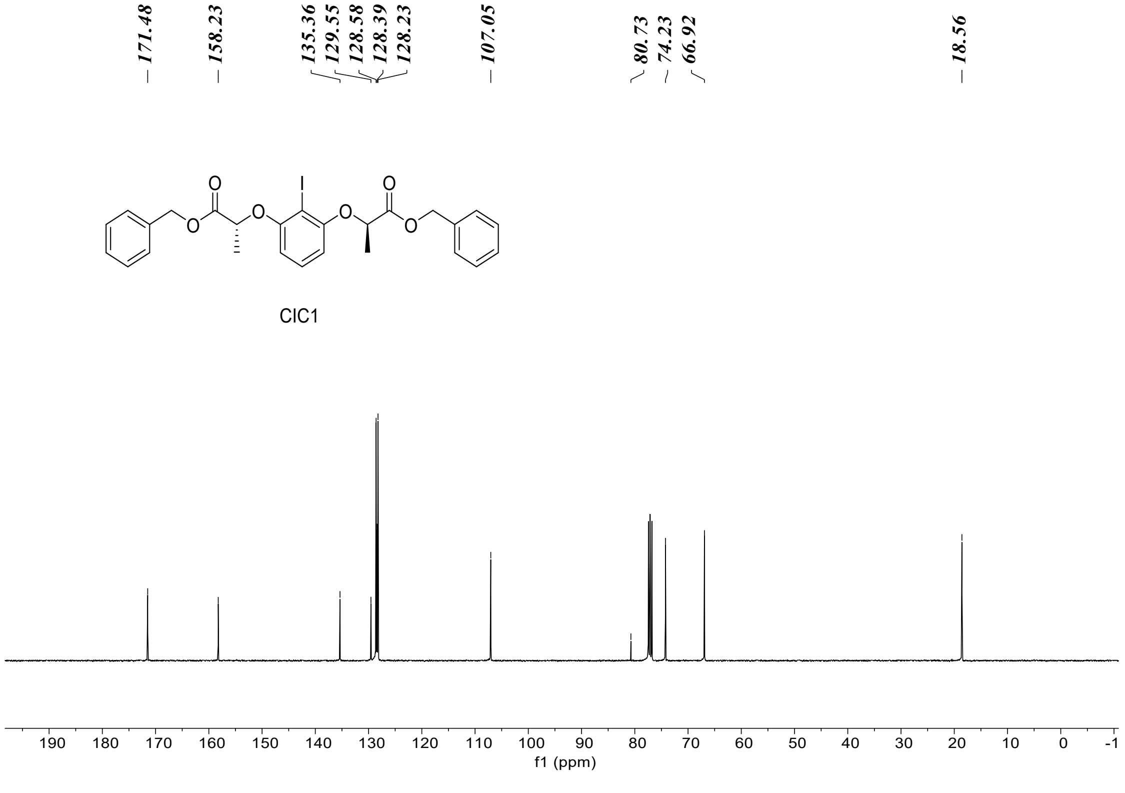
f1 (ppm)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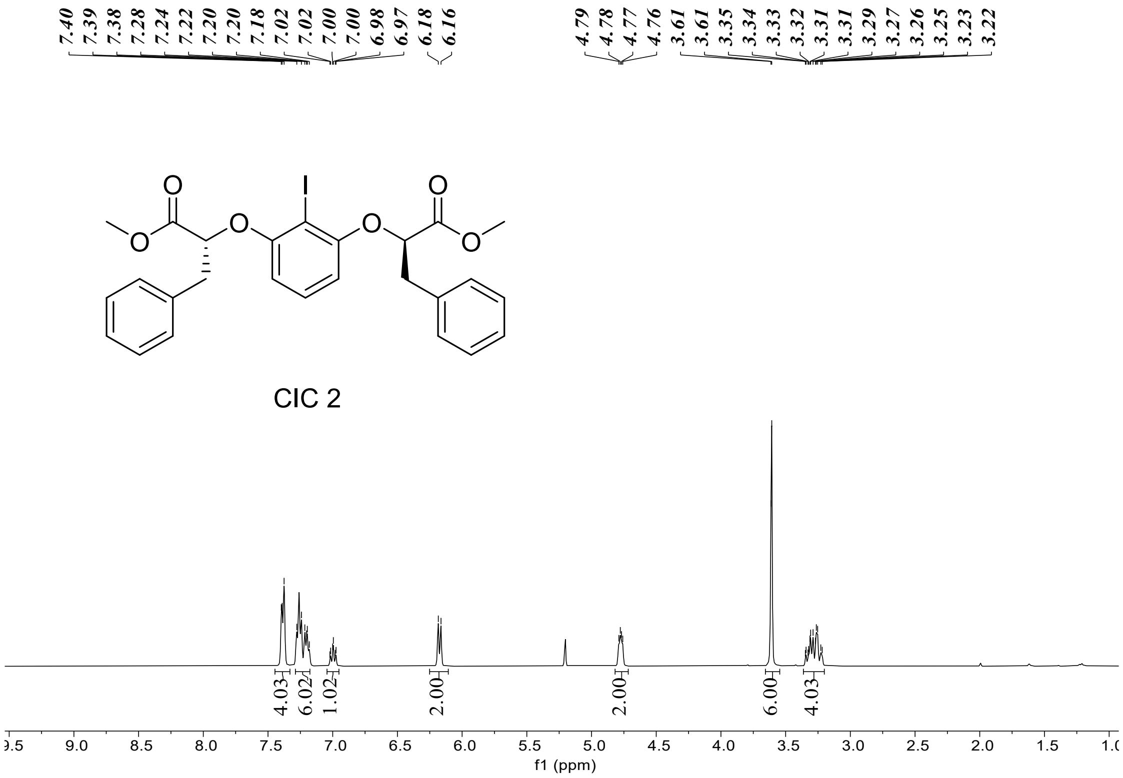
f1 (ppm)



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2.00
6.00
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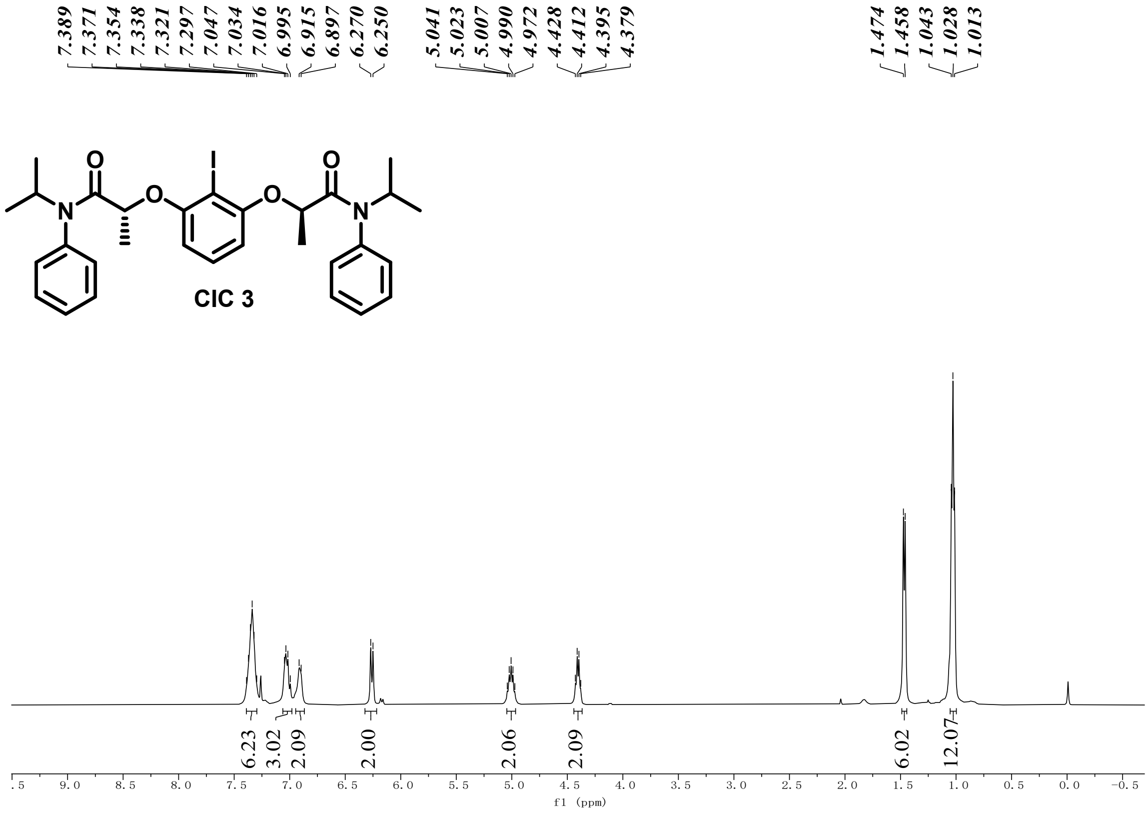
f1 (ppm)

CIC 2



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2.06
2.09
6.02
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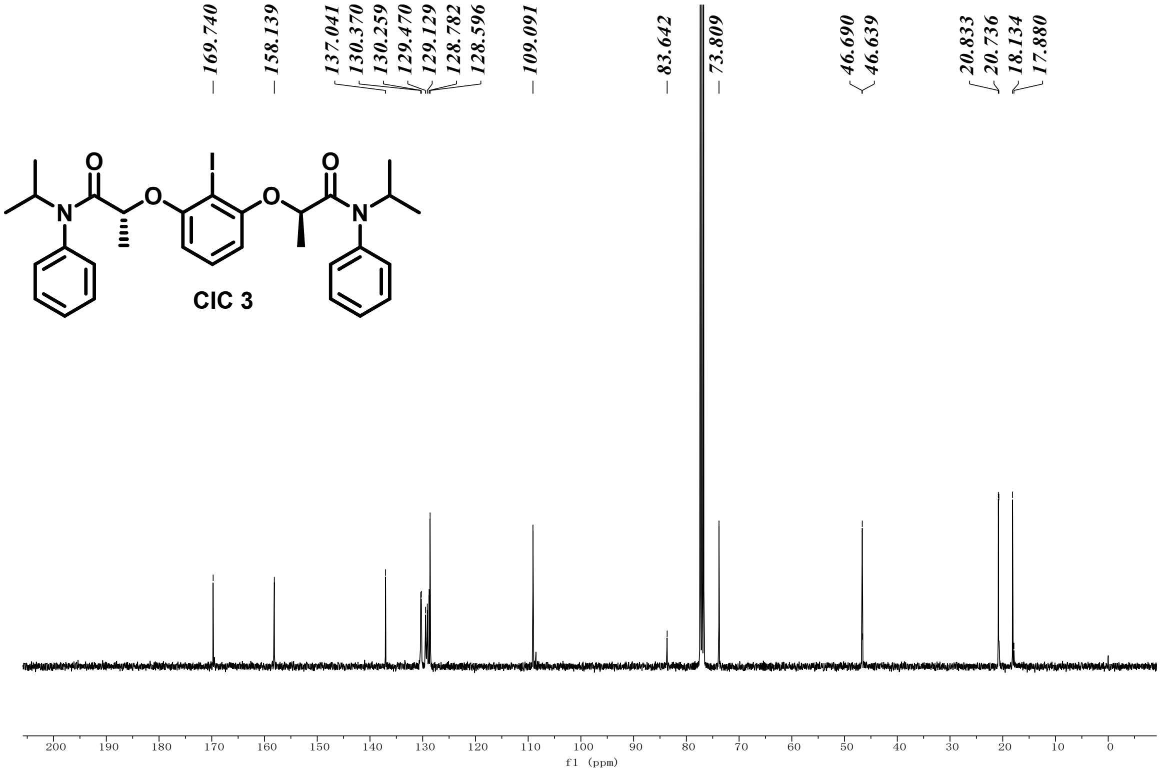
f1 (ppm)

CIC 3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20.736
18.134
17.880
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O
N
O
N
CIC 3
200
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180
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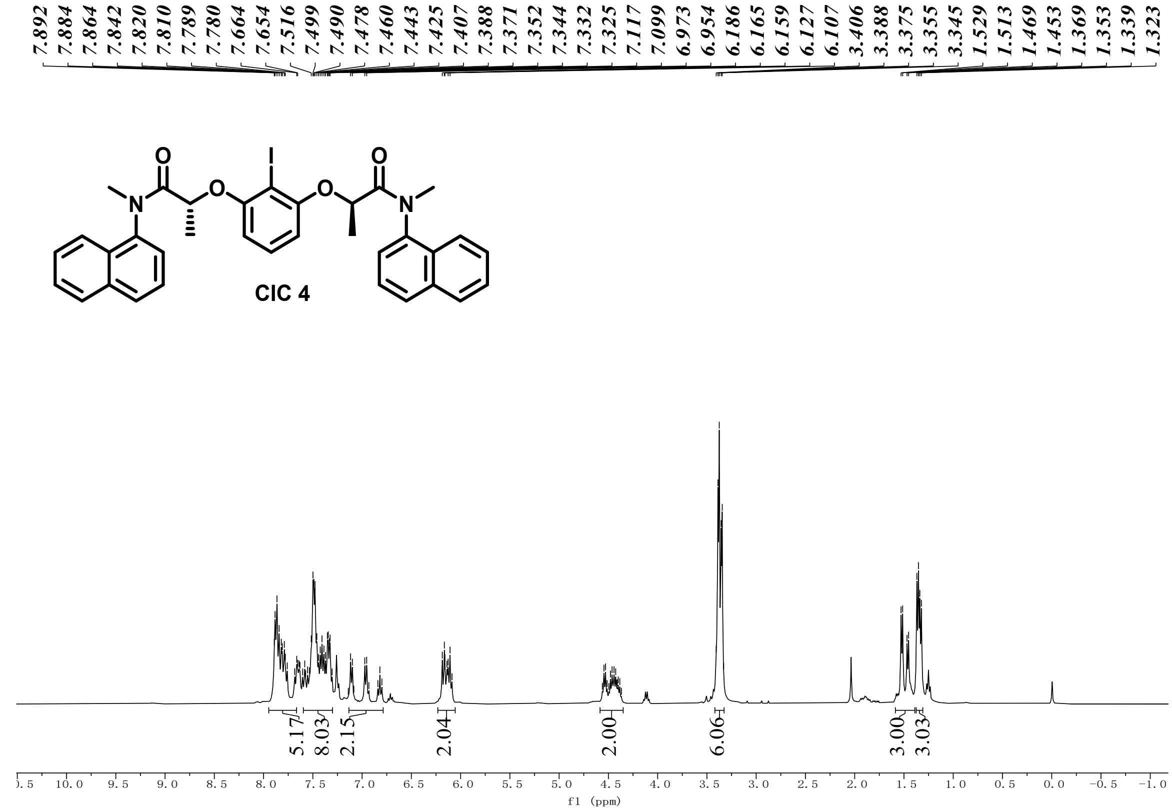
f1 (ppm)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CIC 4
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3.00
3.03
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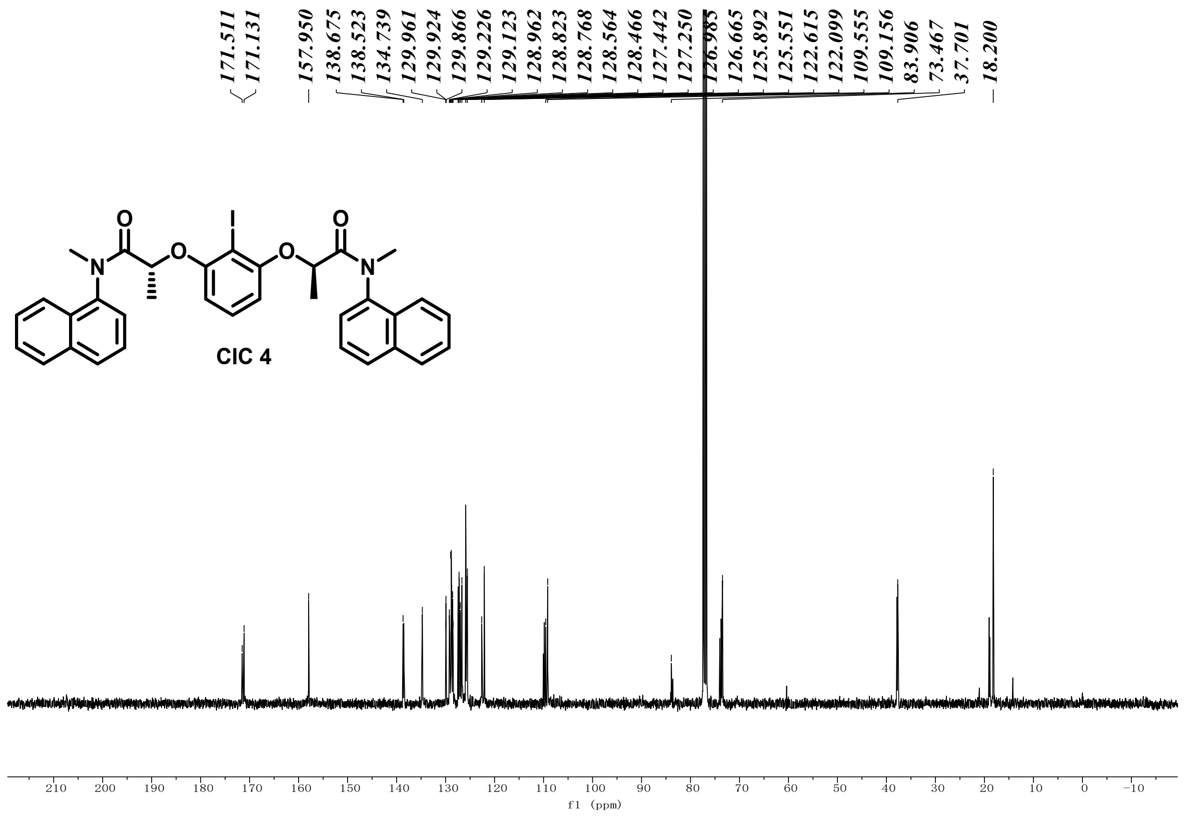
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128.564
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127.442
127.250
126.985
126.665
125.892
125.551
122.615
122.099
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73.467
37.701
18.200
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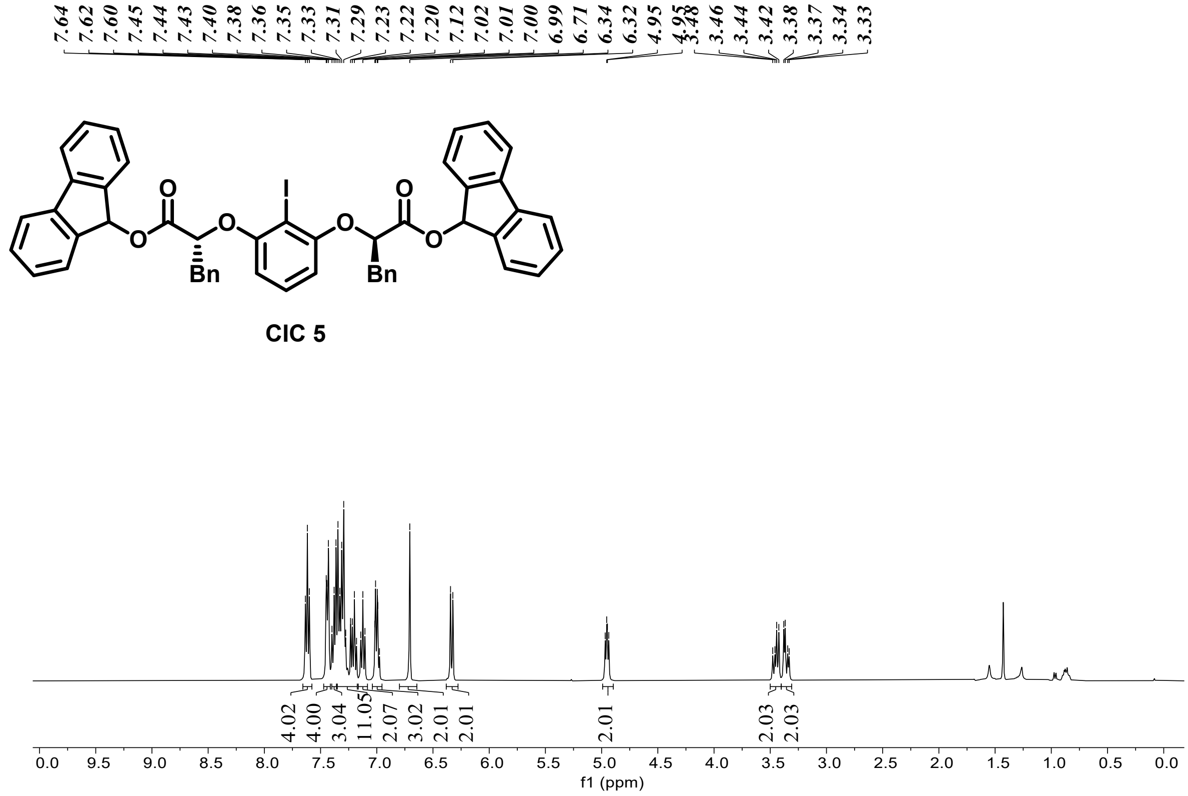
f1 (ppm)

CIC 4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6.34
6.32
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3.44
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3.33
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Bn
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4.00
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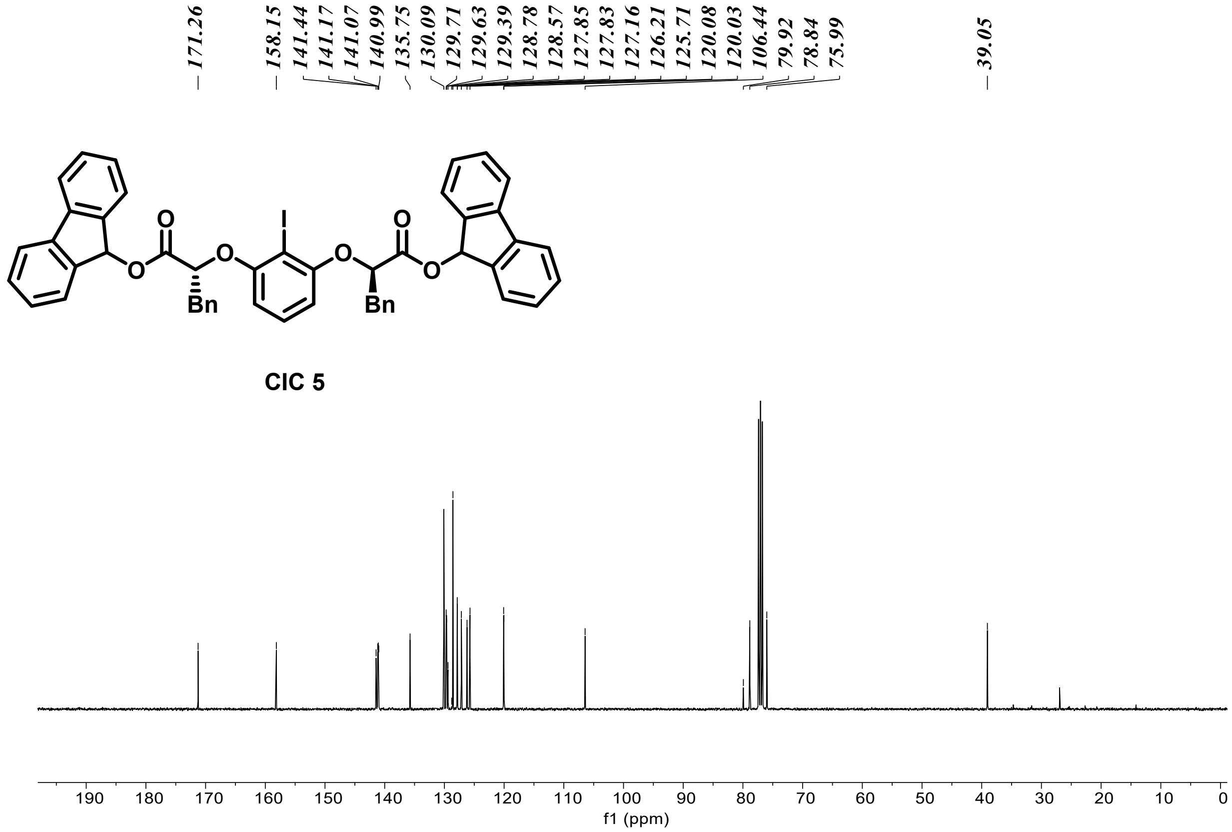
2.07
3.02
2.01
2.01
2.01
2.03
2.03
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

CIC 5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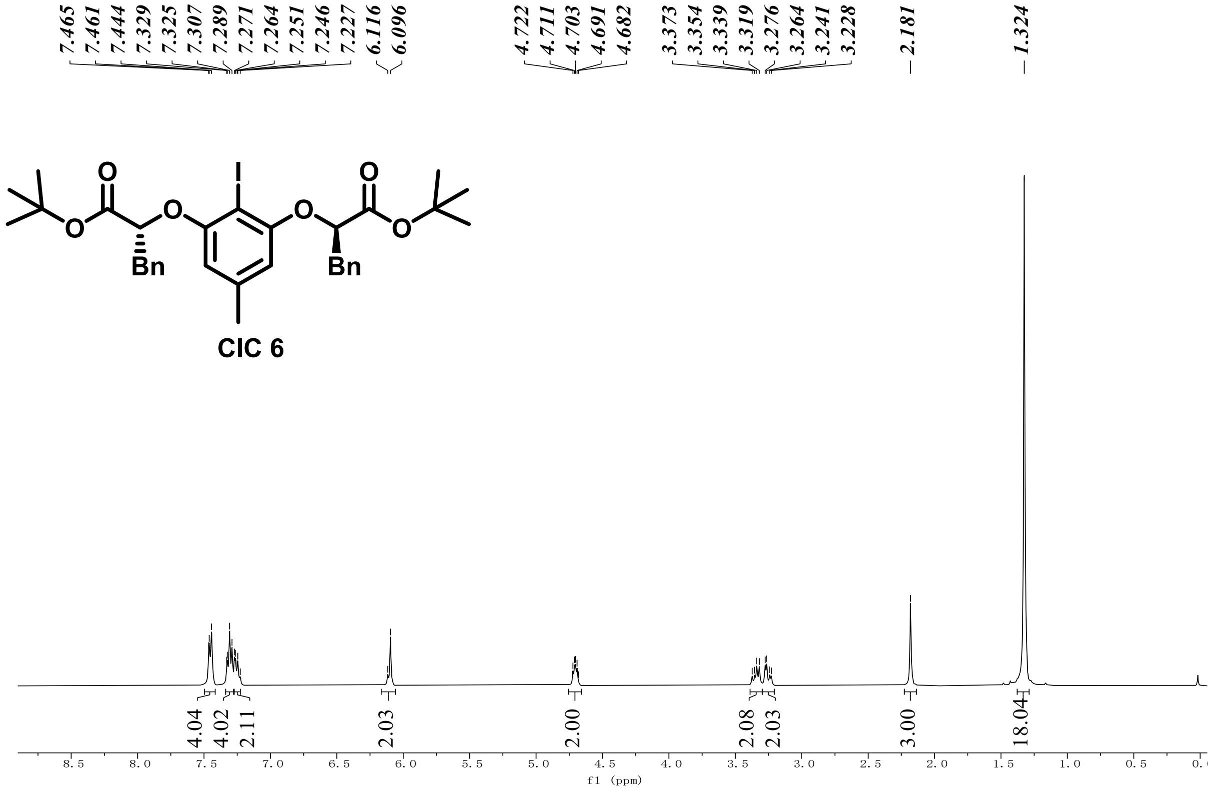
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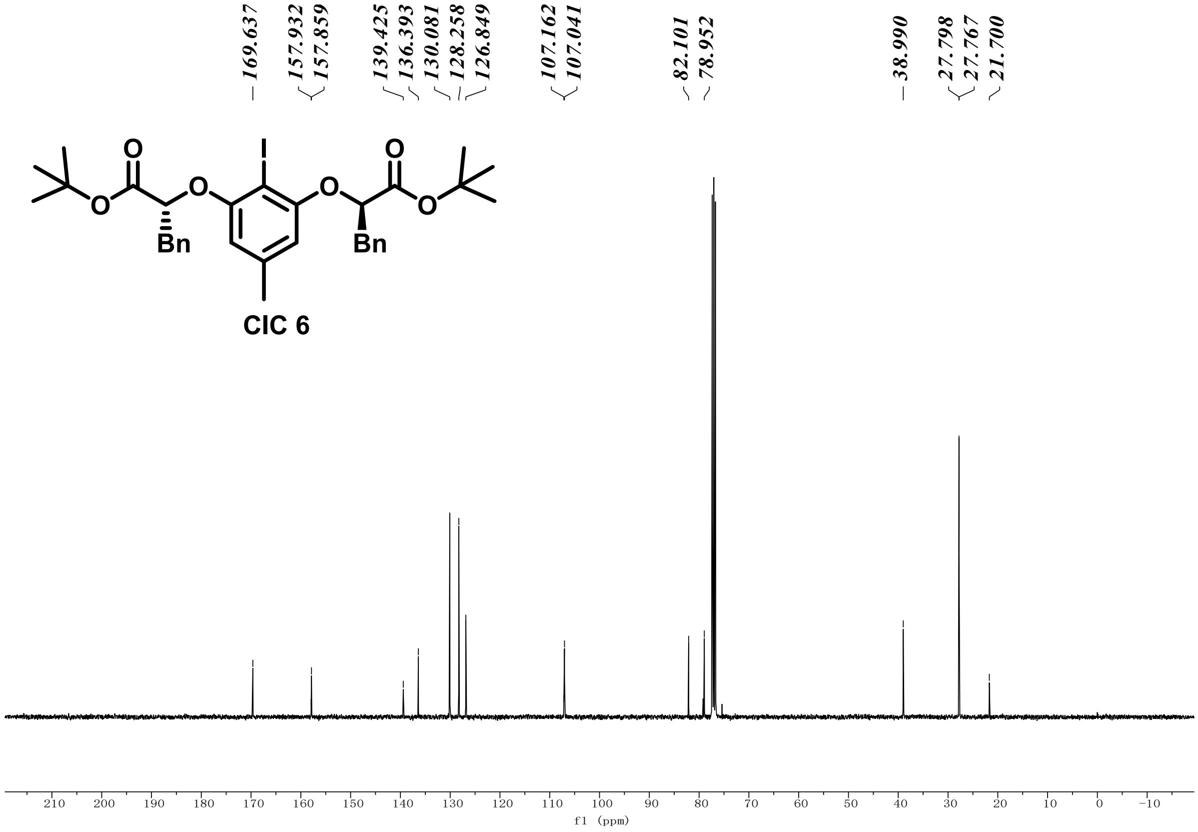
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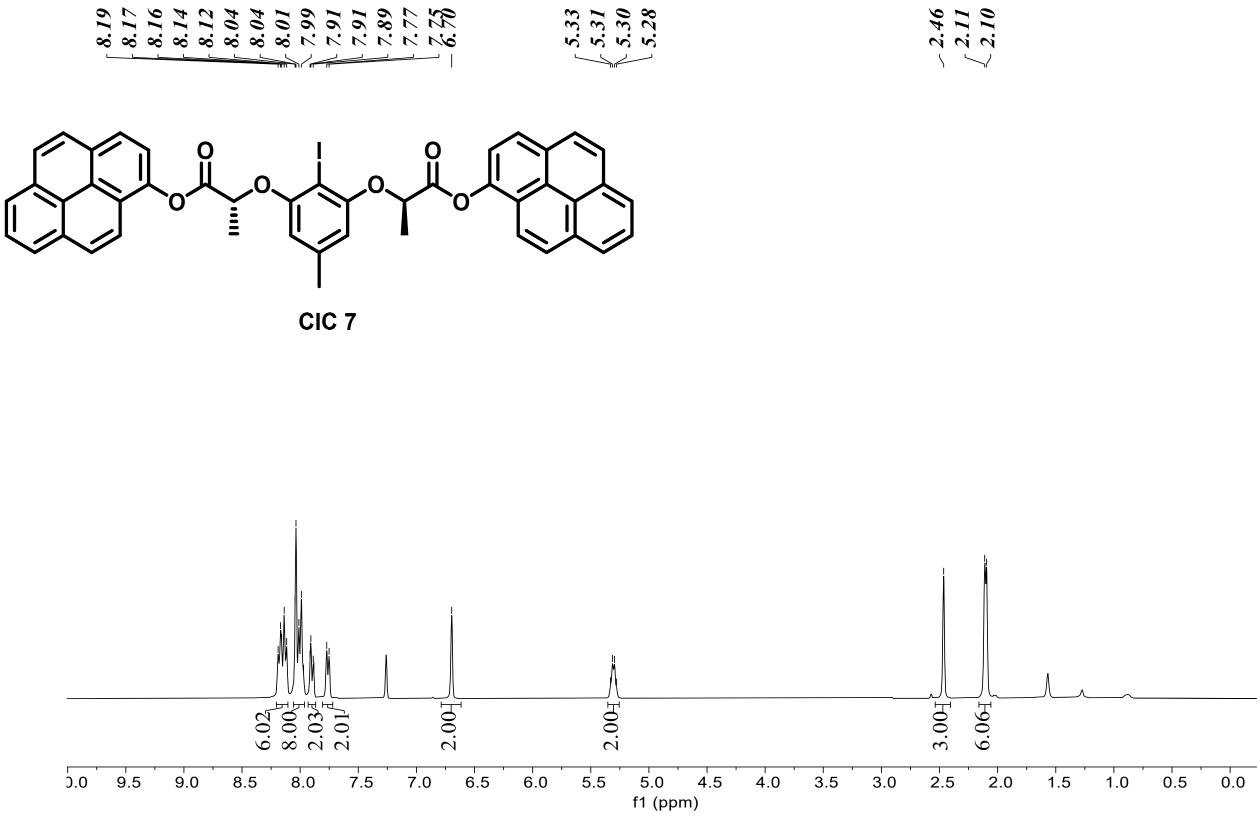
f1 (ppm)

CIC 6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I
O
O
O
O
O
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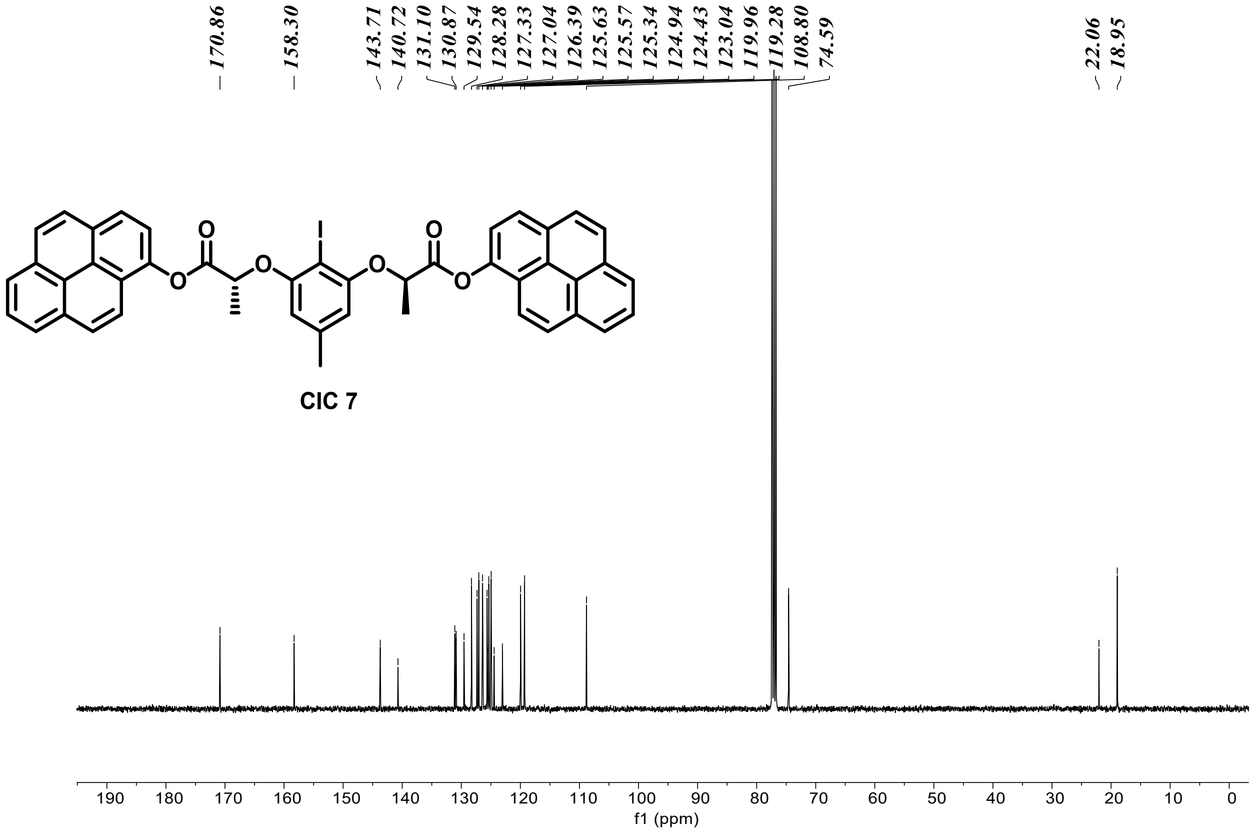
8.00
2.03
2.01
2.00
2.00
3.00
6.06
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

CIC 7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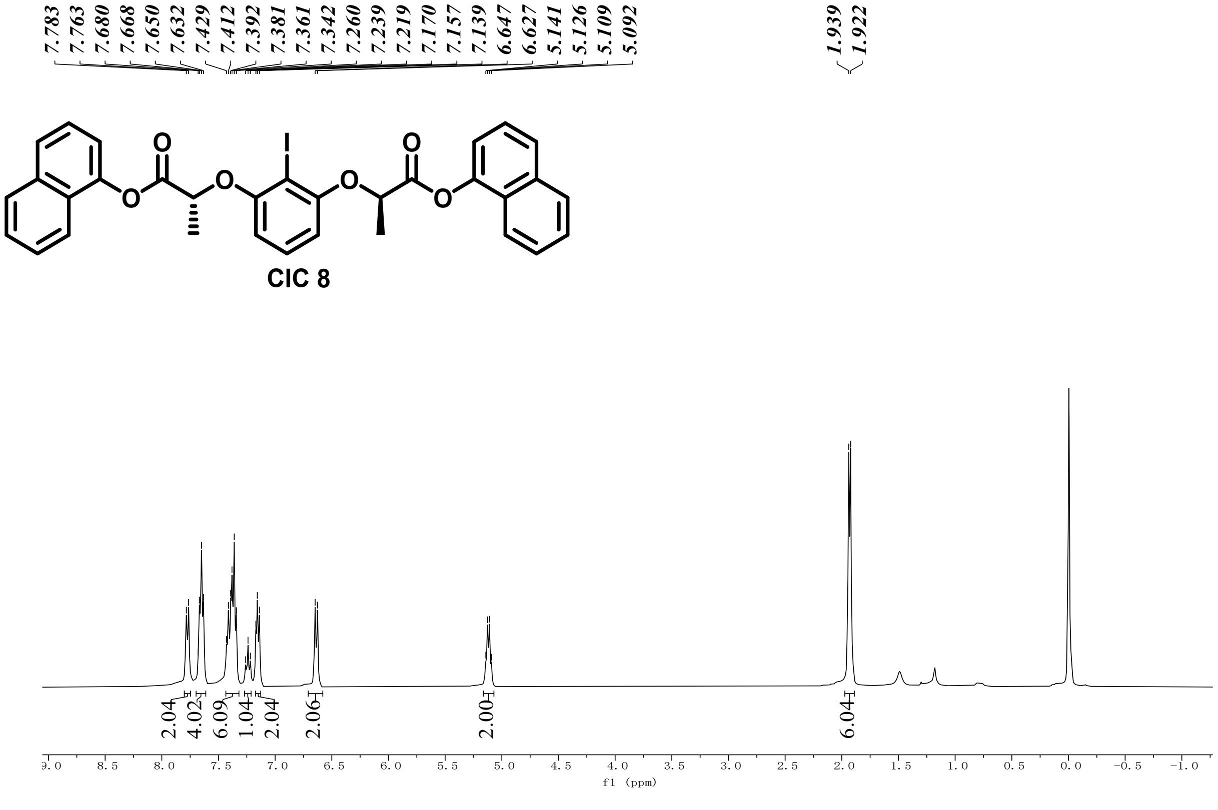
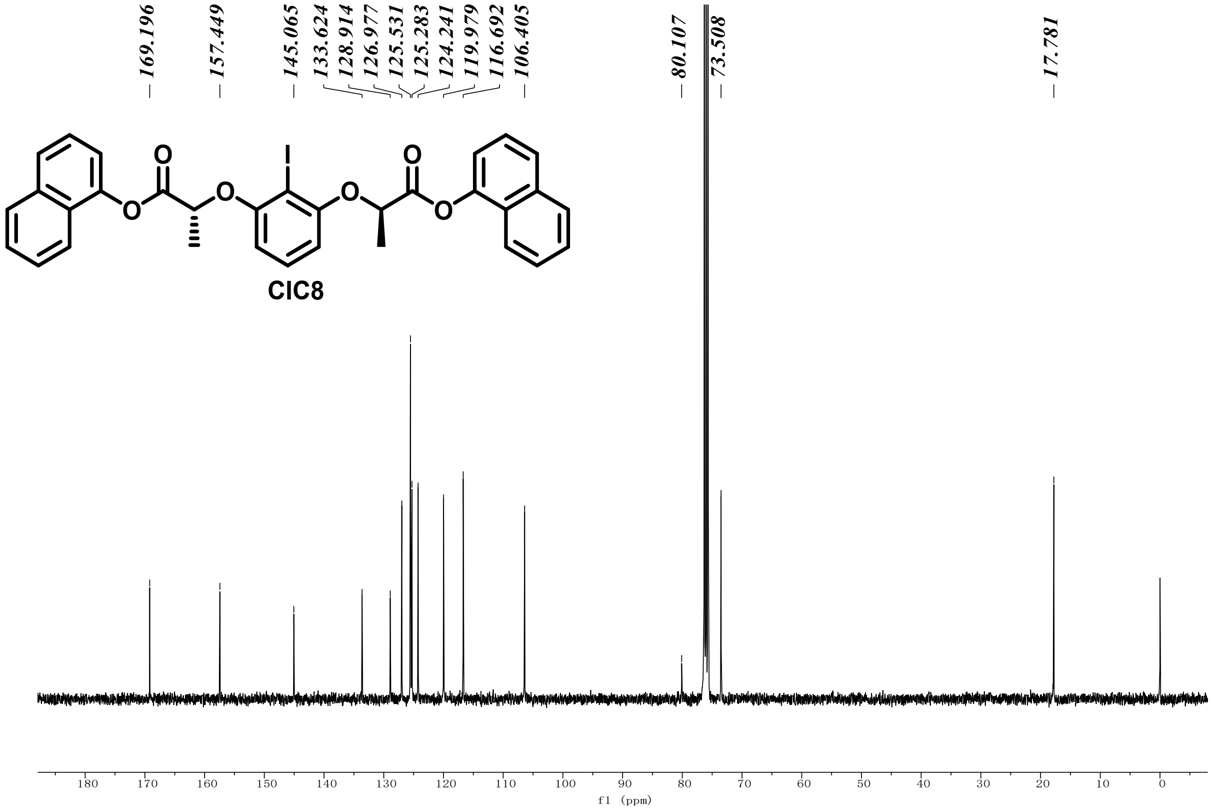
140.72
131.10
130.87
129.54
128.28
127.33
127.04
126.39
125.63
125.57
125.34
124.94
124.43
123.04
119.96
119.28
108.80
74.59
22.06
18.95
O
O
O
O
O
O
I
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

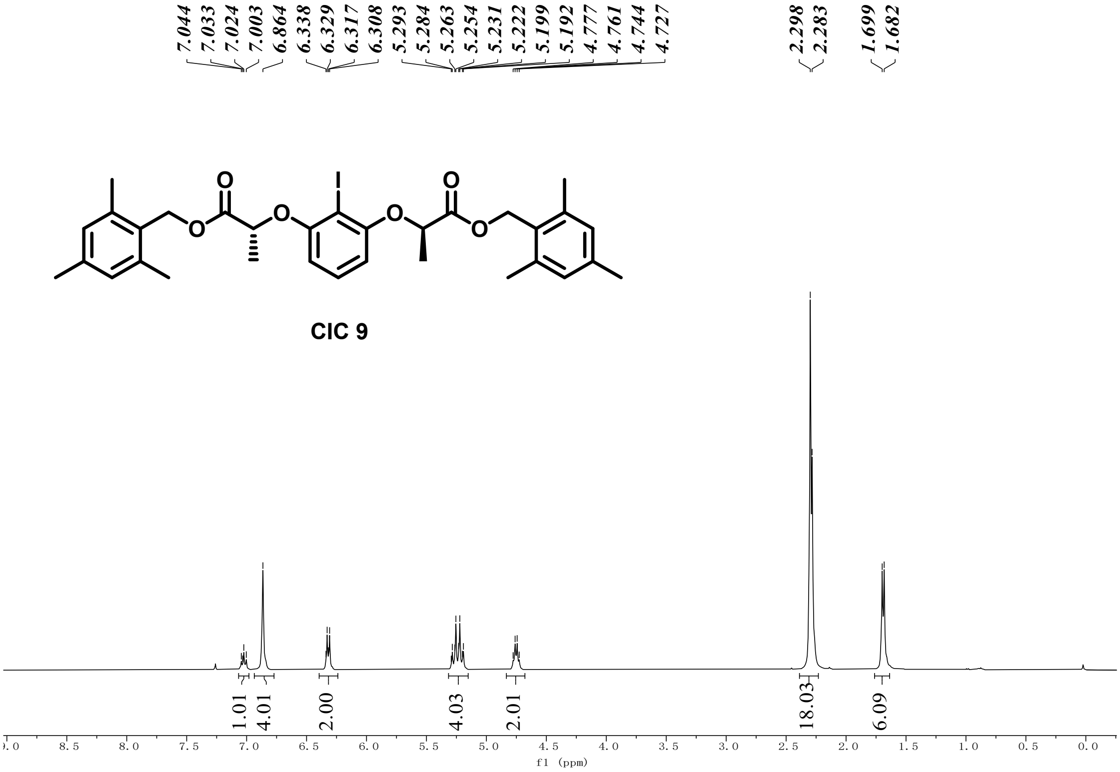
CIC 7


7.783
7.763
7.680
7.668
7.650
7.632
7.429
7.412
7.392
7.381
7.361
7.342
7.260
7.239
7.219
7.170
7.157
7.139
6.647
6.627
5.141
5.126
5.109
5.092
1.939
1.922
O
O
O
O
O
O
I
2.04
4.02
6.09
1.04
2.04
2.06
2.00
6.04
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
0.0
−0.5
−1.0
f1 (ppm)

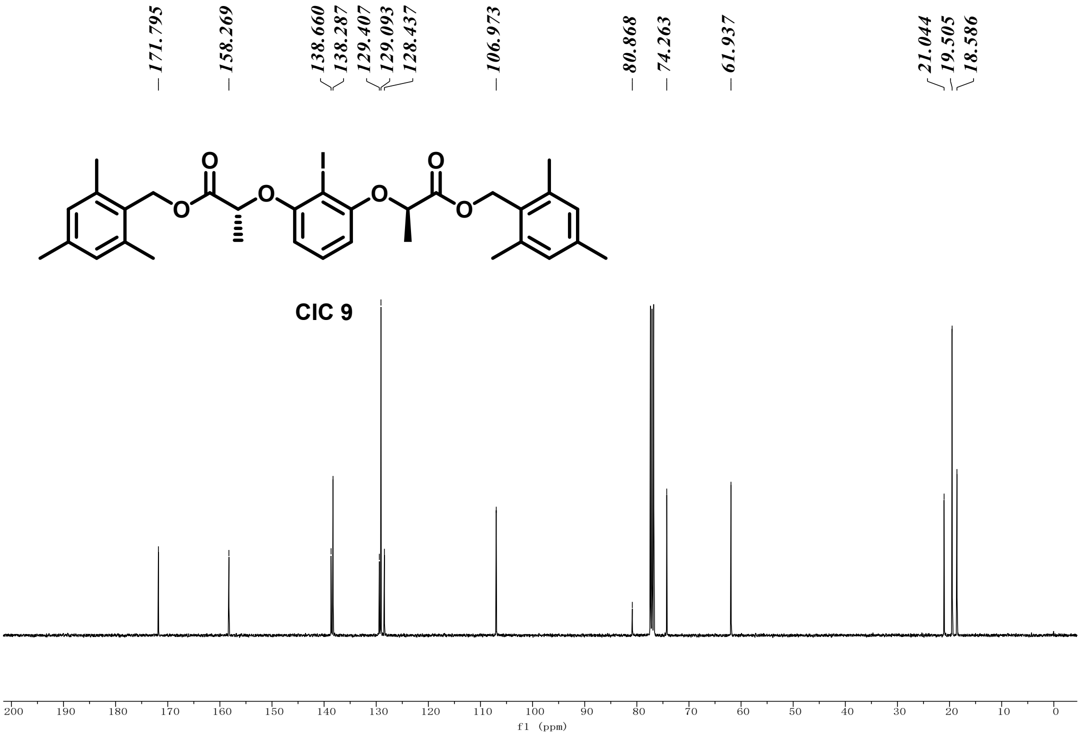
CIC 8


169.196
157.449
145.065
133.624
128.914
126.977
125.531
125.283
124.241
119.979
116.692
106.405
80.107
73.508
17.781
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

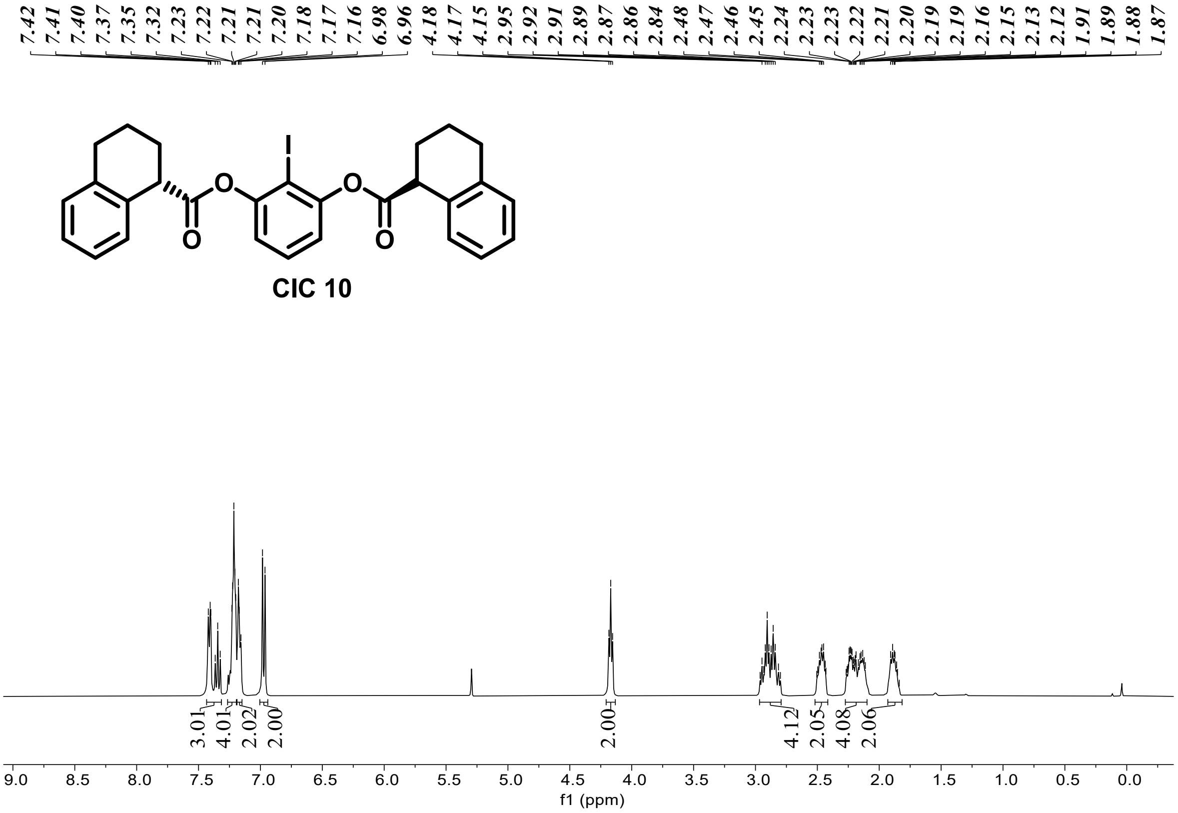
CIC8


7.044
7.033
7.024
7.003
6.864
6.338
6.329
6.317
6.308
5.293
5.284
5.263
5.254
5.231
5.222
5.199
5.192
4.777
4.761
4.744
4.727
2.298
2.283
1.699
1.682
1.01
4.01
2.00
4.03
2.01
18.03
6.09
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
0.0
f1 (ppm)

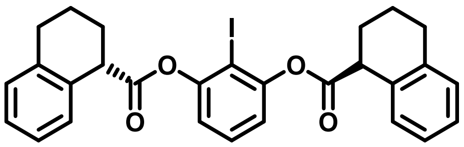
CIC 9


171.795
158.269
138.660
138.287
129.407
129.093
128.437
106.973
80.868
74.263
61.937
21.044
19.505
18.586
I
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

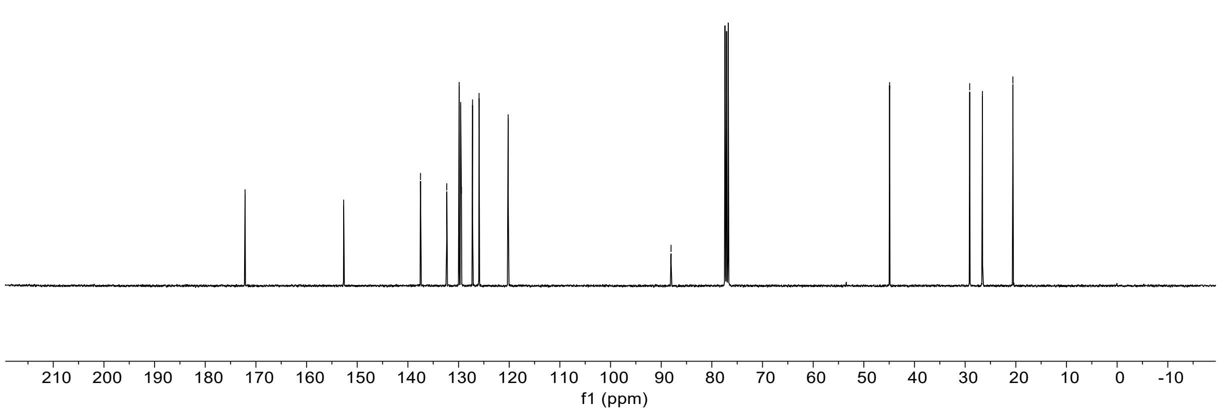
CIC 9

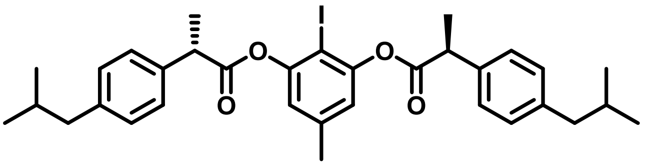

7.42
7.41
7.40
7.37
7.35
7.32
7.23
7.22
7.21
7.21
7.20
7.18
7.17
7.16
6.98
6.96
4.18
4.17
4.15
2.95
2.92
2.91
2.89
2.87
2.86
2.84
2.48
2.47
2.46
2.45
2.24
2.23
2.23
2.22
2.21
2.20
2.19
2.19
2.16
2.15
2.13
2.12
1.91
1.89
1.88
1.87
I
O
3.01
4.01
2.02
2.00
2.00
4.12
2.05
4.08
2.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

CIC 10

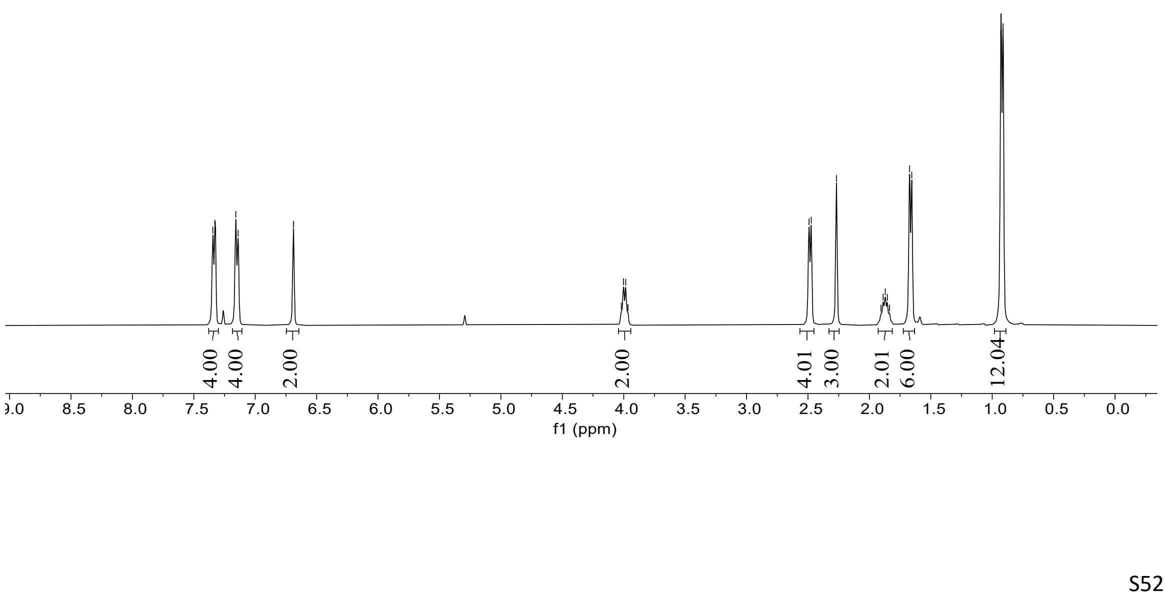


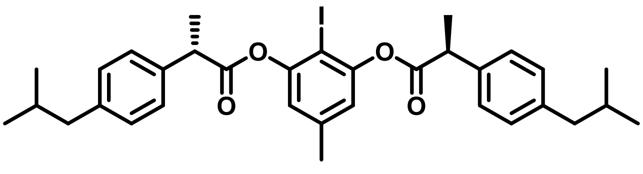
CIC 10


172.14
152.67
137.52
132.32
129.89
129.58
129.44
127.24
125.96
120.21
88.06
44.92
29.11
26.59
20.58
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

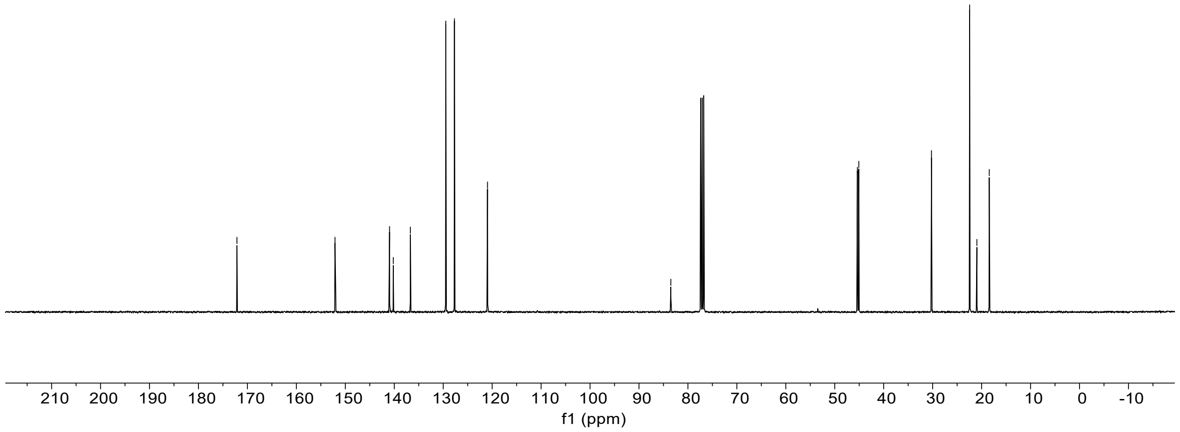


CIC 11

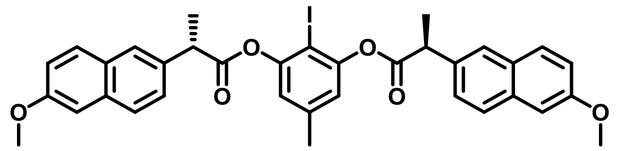

7.35
7.33
7.32
7.16
7.14
6.69
4.02
4.00
3.98
3.97
2.49
2.47
2.27
1.91
1.89
1.87
1.85
1.84
1.67
1.65
0.93
0.92
0.91
4.00
4.00
2.00
2.00
4.01
3.00
2.01
6.00
12.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



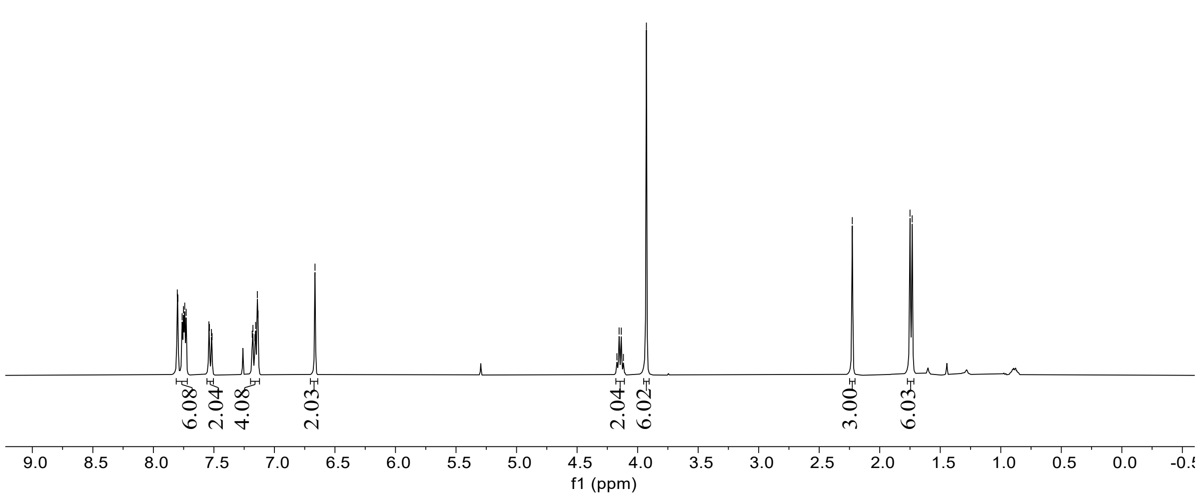
CIC11



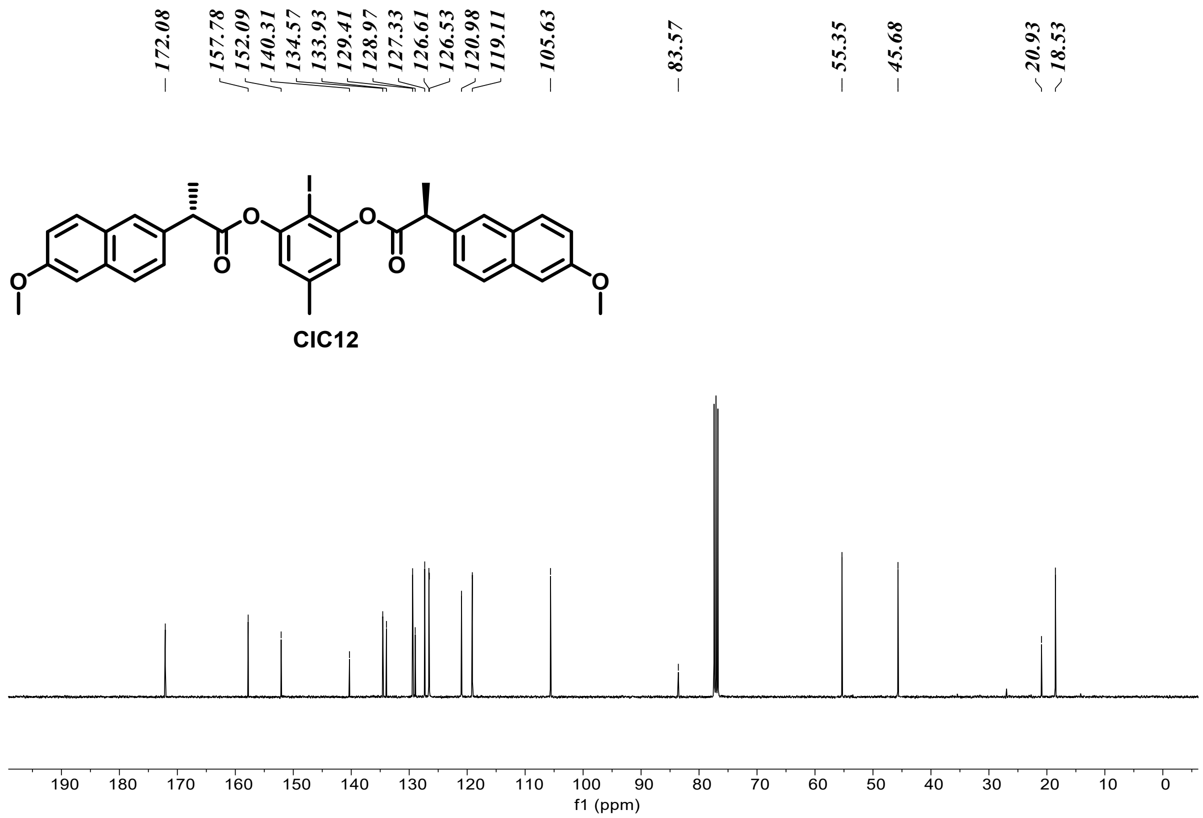
172.13
152.10
140.96
140.21
136.70
129.47
127.70
120.95
83.49
45.37
45.08
30.25
22.44
20.96
18.43
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

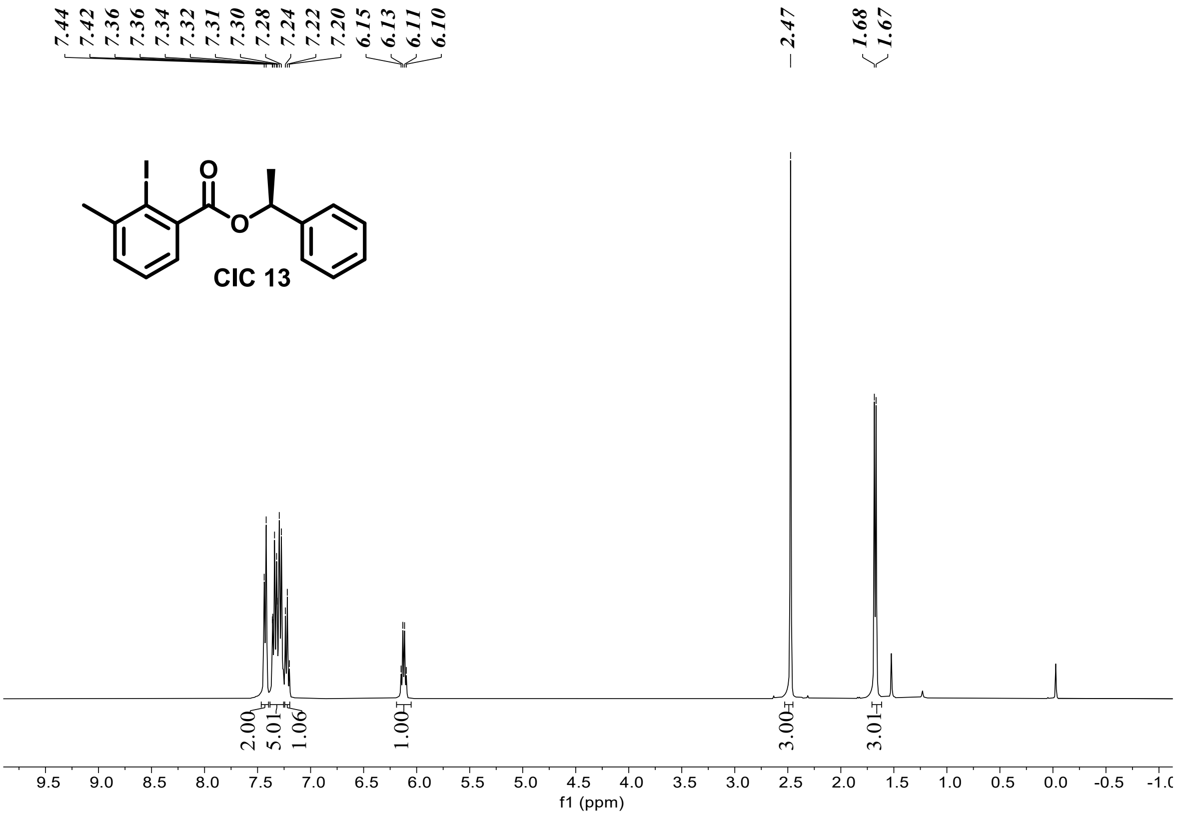


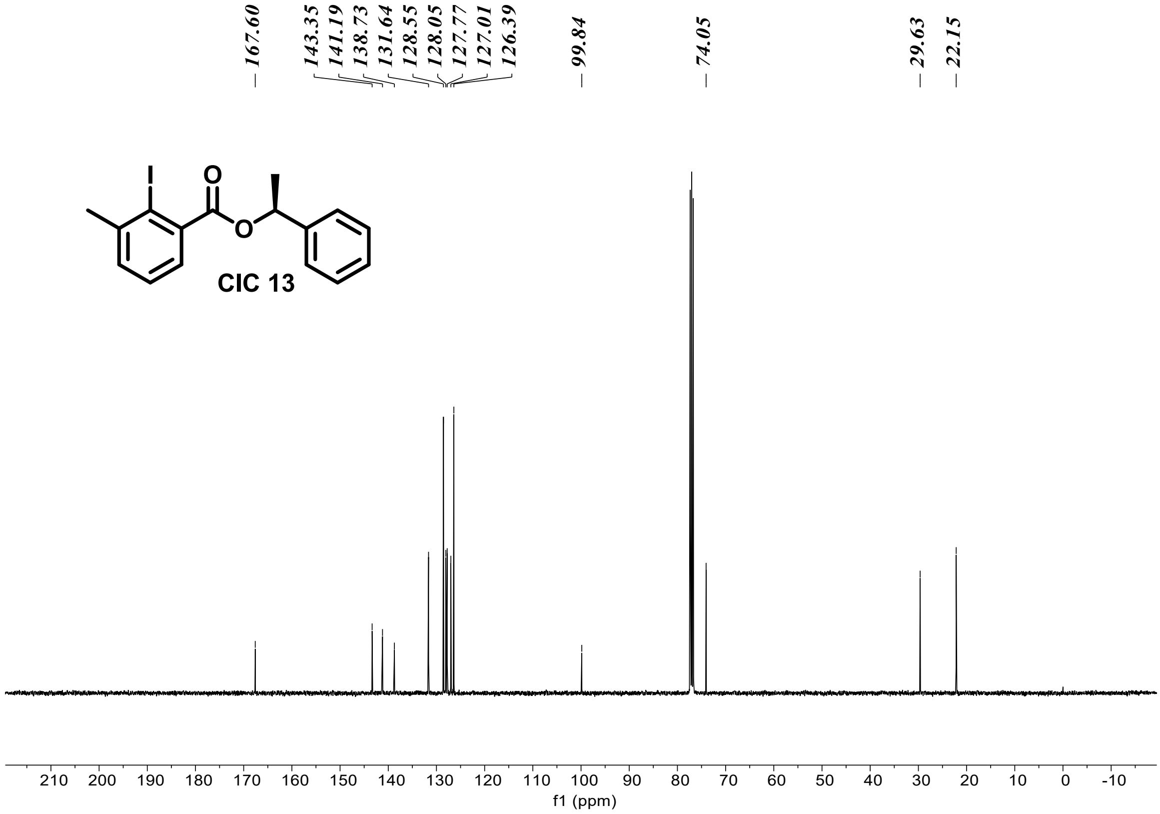
CIC12



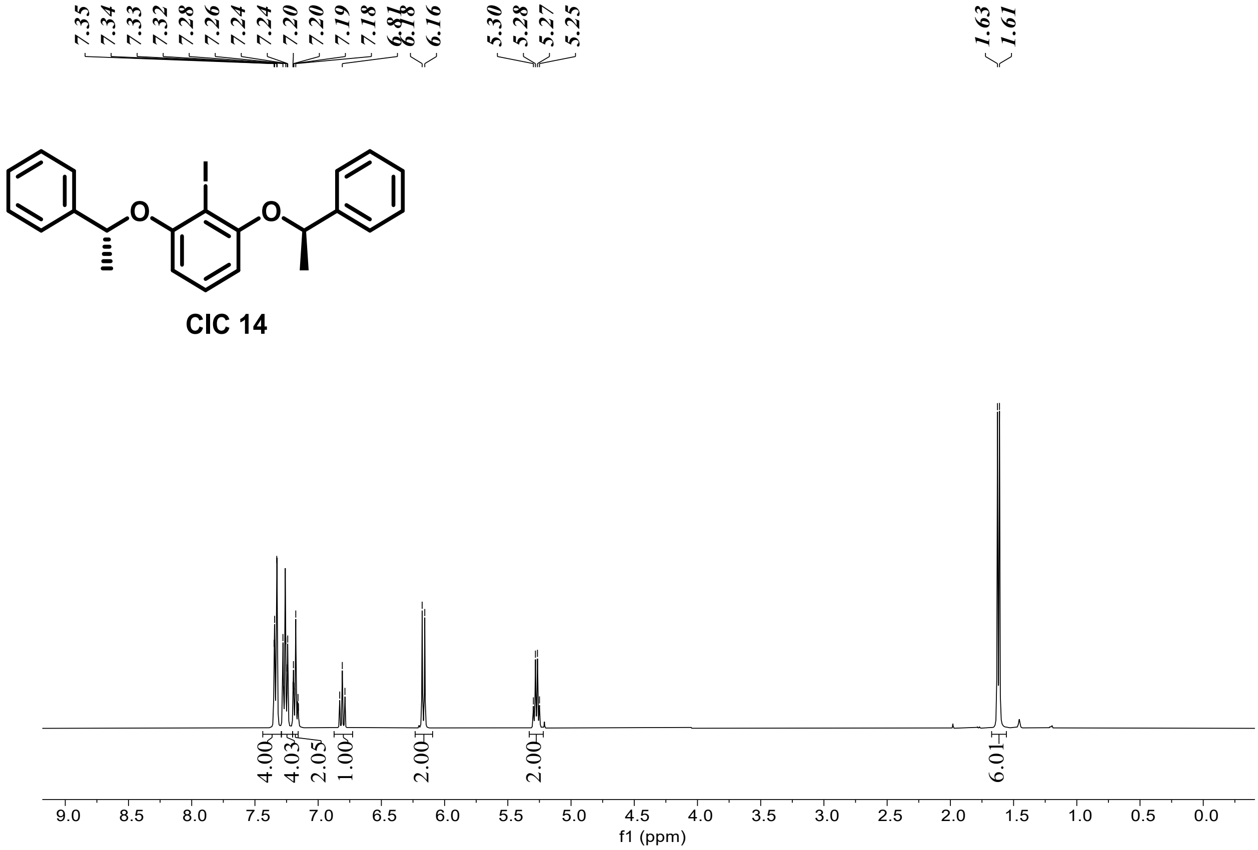
7.80
7.80
7.76
7.75
7.74
7.73
7.54
7.54
7.16
7.14
7.14
6.66
4.17
4.15
4.14
4.12
3.93
2.23
1.75
1.73
6.08
2.04
4.08
2.03
2.04
6.02
3.00
6.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


172.08
157.78
152.09
140.31
134.57
133.93
129.41
128.97
127.33
126.61
126.53
120.98
119.11
105.63
83.57
55.35
45.68
20.93
18.53
CIC12
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

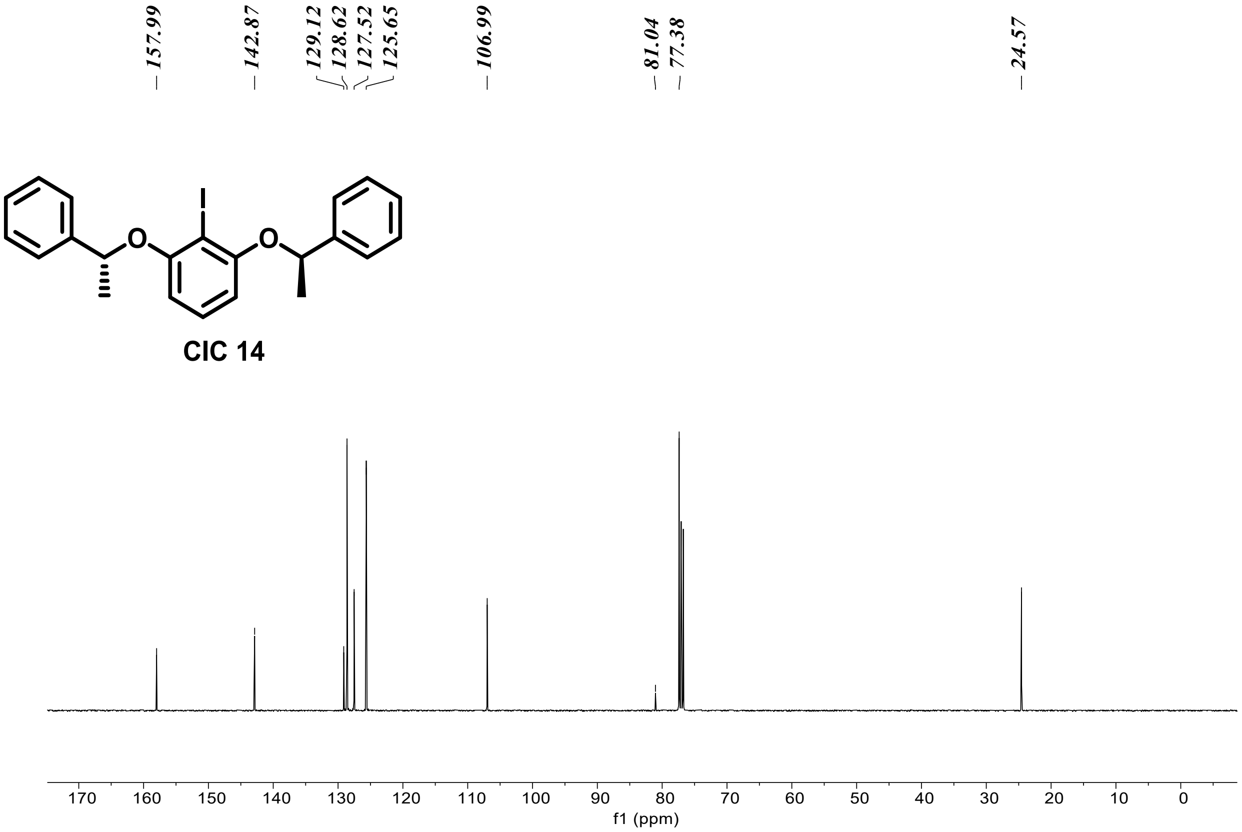

7.44
7.42
7.36
7.36
7.34
7.32
7.31
7.30
7.28
7.24
7.22
7.20
6.15
6.13
6.11
6.10
2.47
1.68
1.67
CIC 13
2.00
5.01
1.06
1.00
3.00
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


167.60
143.35
141.19
138.73
131.64
128.55
128.05
127.77
127.01
126.39
99.84
74.05
29.63
22.15
I
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

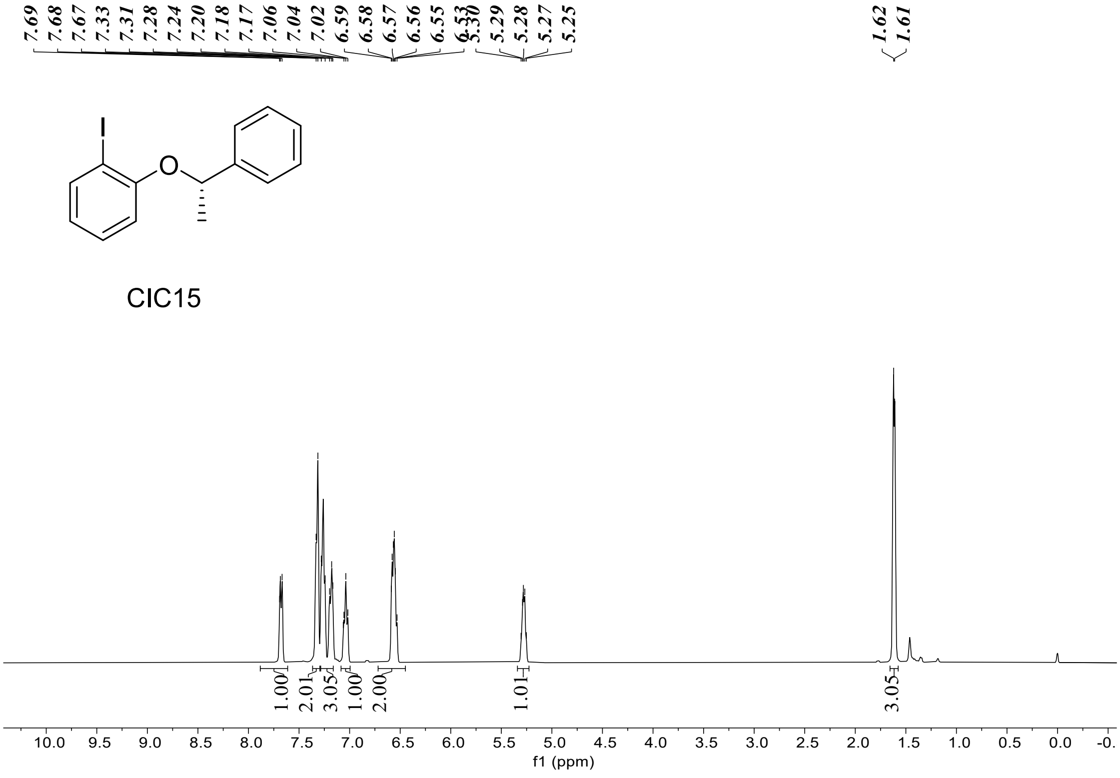
CIC 13


7.35
7.34
7.33
7.32
7.28
7.26
7.24
7.24
7.20
7.20
7.19
7.18
6.81
6.18
6.16
5.30
5.28
5.27
5.25
1.63
1.61
O
I
O
4.00
4.03
2.05
1.00
2.00
2.00
6.01
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
0.0
f1 (ppm)

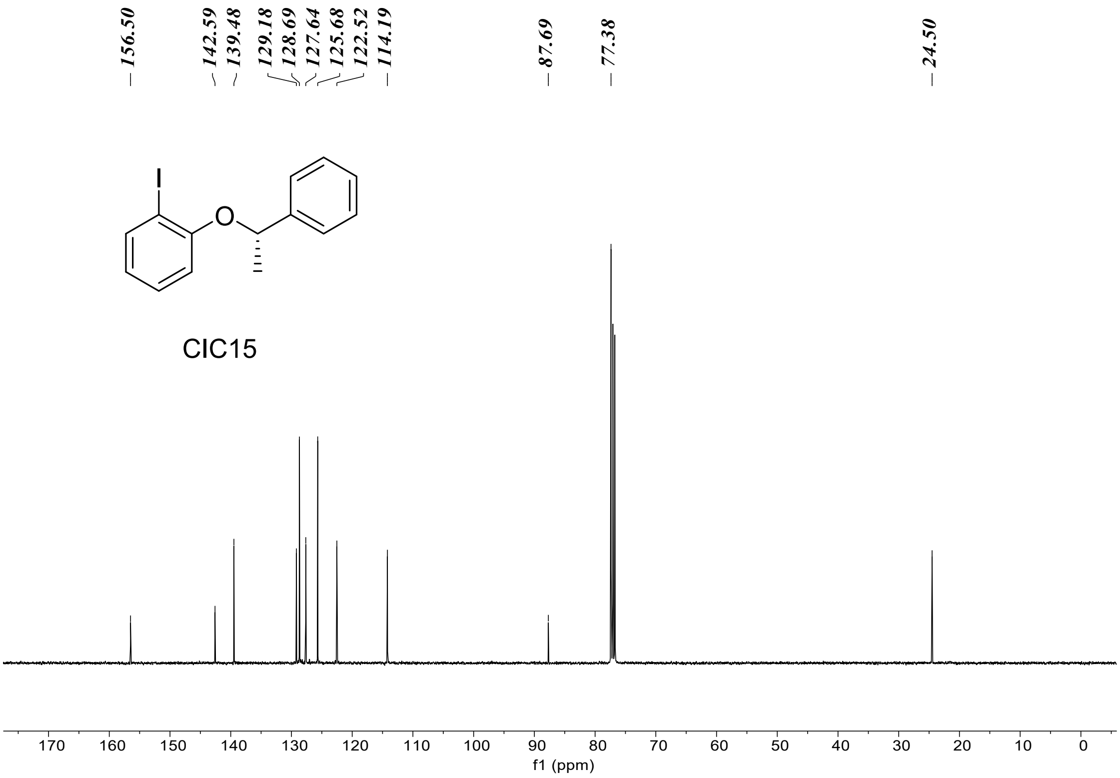
CIC 14

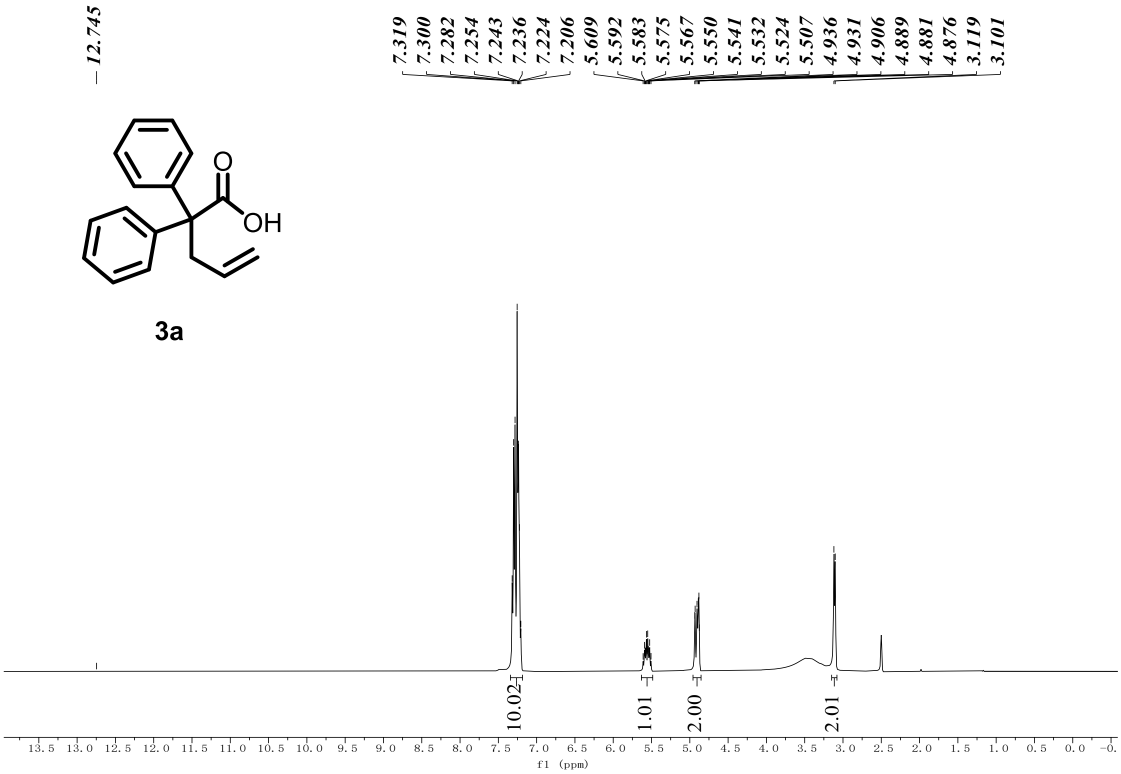

157.99
142.87
129.12
128.62
127.52
125.65
106.99
81.04
77.38
24.57
I
O
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

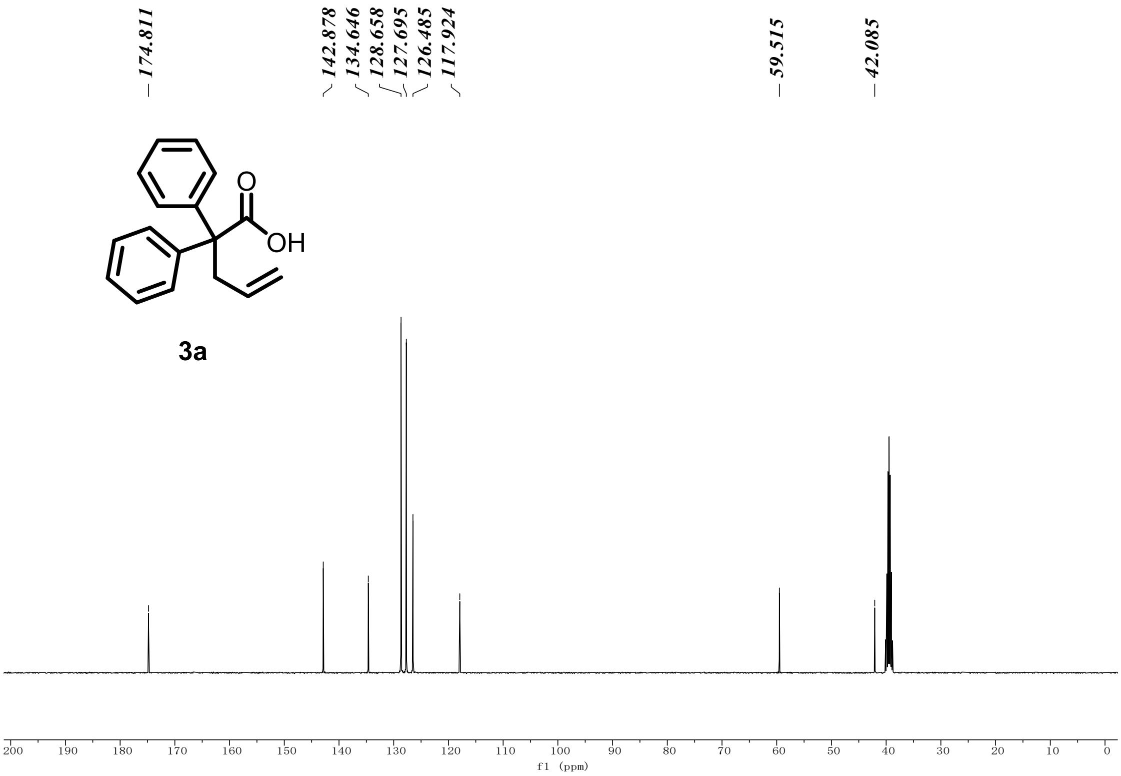
CIC 14


7.69
7.68
7.67
7.33
7.31
7.28
7.24
7.20
7.18
7.17
7.06
7.04
7.02
6.59
6.58
6.57
6.56
6.55
6.53
5.30
5.29
5.28
5.27
5.25
1.62
1.61
I
O
1.00
2.01
3.05
1.00
2.00
1.01
3.05
10.0
9.5
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
0.0
-0.
f1 (ppm)

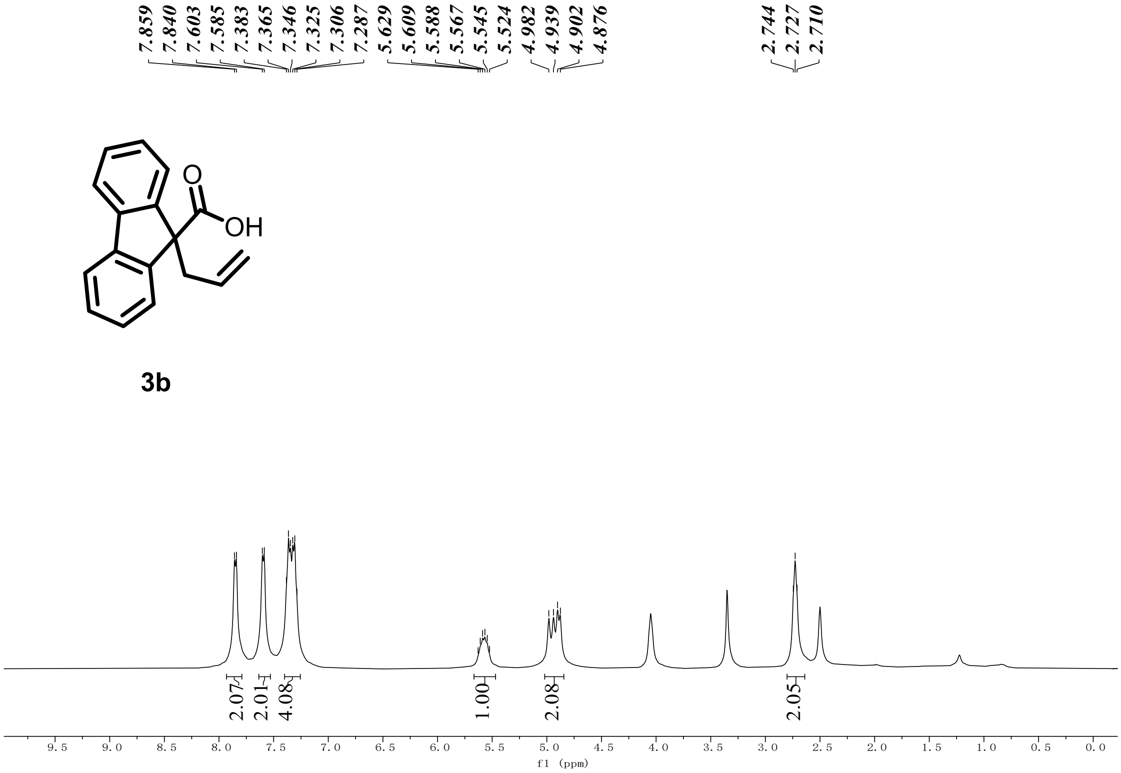
CIC15


156.50
142.59
139.48
129.18
128.69
127.64
125.68
122.52
114.19
87.69
77.38
24.50
I
O
CIC15
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

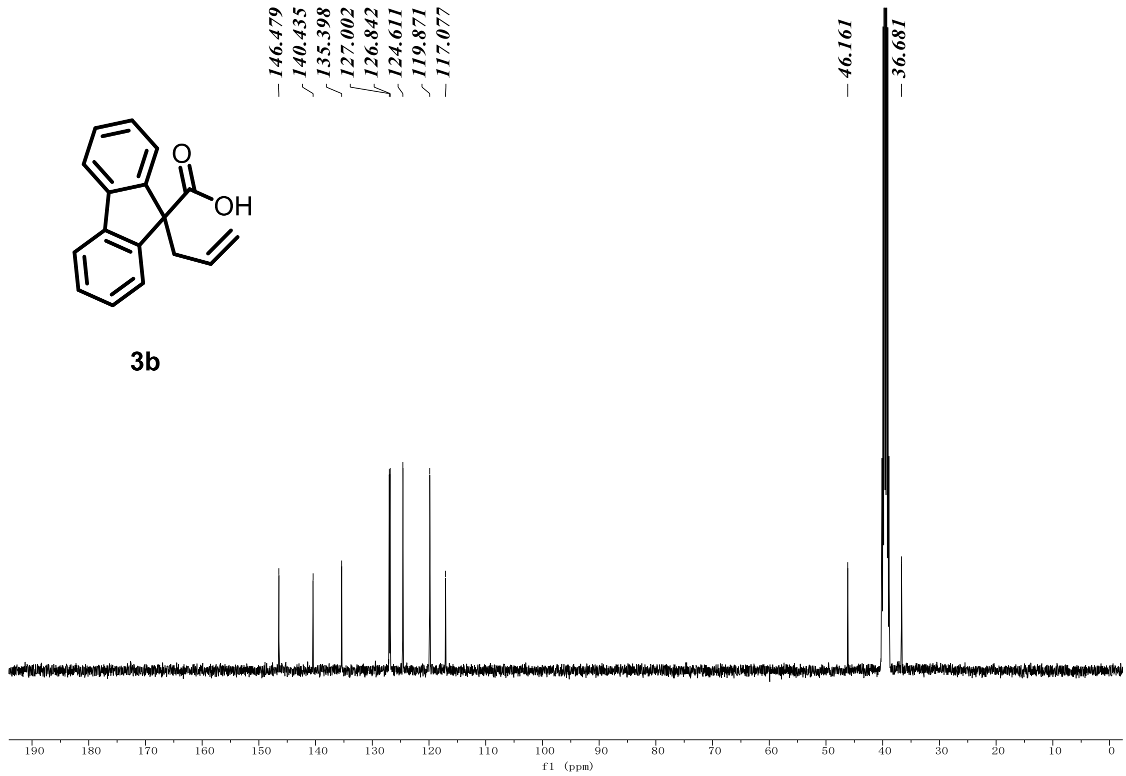

12.745
7.319
7.300
7.282
7.254
7.243
7.236
7.224
7.206
5.609
5.592
5.583
5.575
5.567
5.550
5.541
5.532
5.524
5.507
4.936
4.931
4.906
4.889
4.881
4.876
3.119
3.101
O
OH
3a
10.02
1.01
2.00
2.01
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


174.811
142.878
134.646
128.658
127.695
126.485
117.924
59.515
42.085
O
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

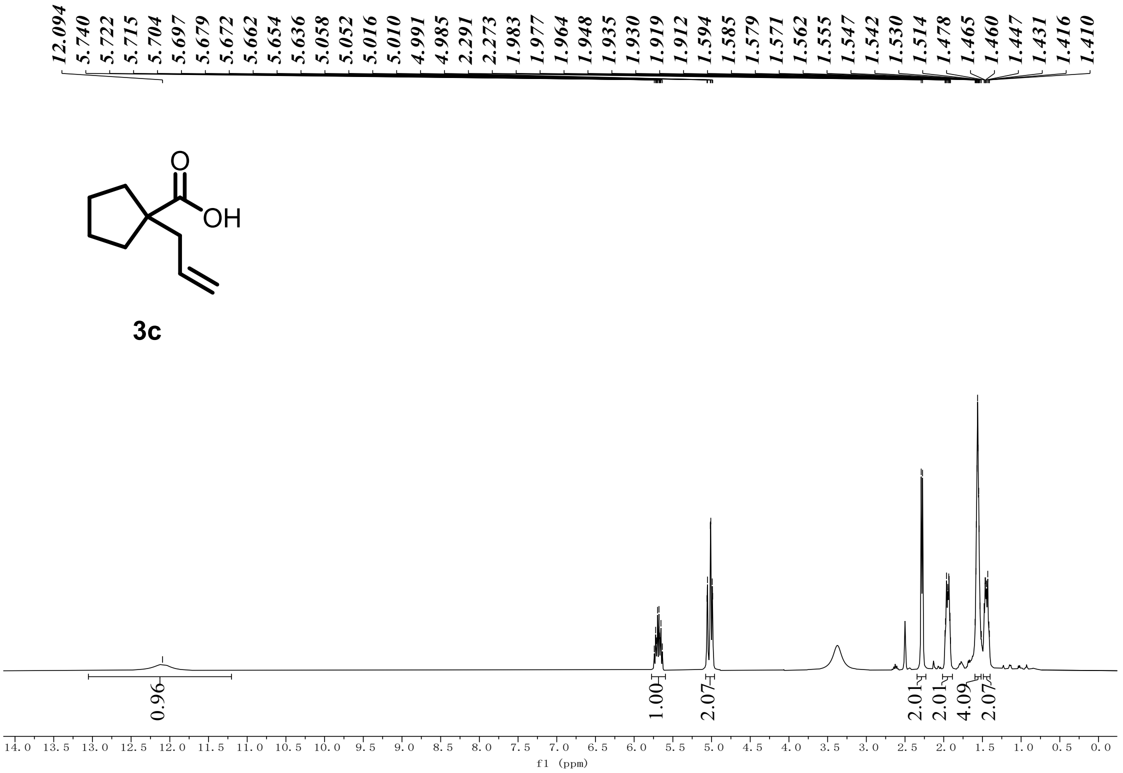
3a


7.859
7.840
7.603
7.585
7.383
7.365
7.346
7.325
7.306
7.287
5.629
5.609
5.588
5.567
5.545
5.524
4.982
4.939
4.902
4.876
2.744
2.727
2.710
O
OH
2.07
2.01
4.08
1.00
2.08
2.05
9.5
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
0.0
f1 (ppm)

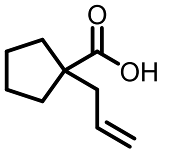
3b


146.479
140.435
135.398
127.002
126.842
124.611
119.871
117.077
46.161
36.681
O
OH
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

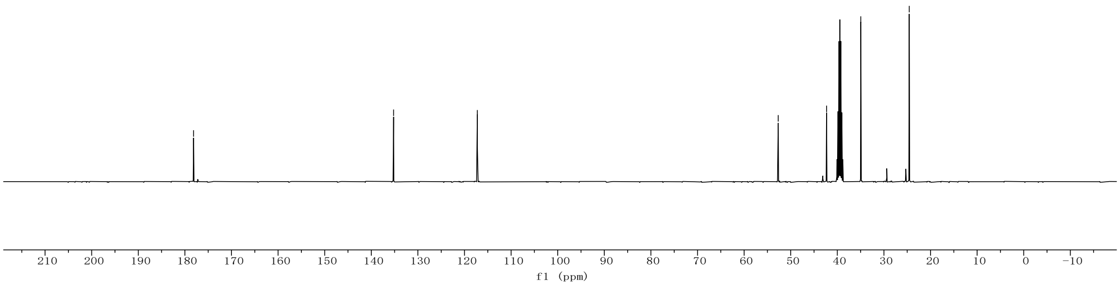
3b


12.094
5.740
5.722
5.715
5.704
5.697
5.679
5.672
5.662
5.654
5.636
5.058
5.052
5.016
5.010
4.991
4.985
2.291
2.273
1.983
1.977
1.964
1.948
1.935
1.930
1.919
1.912
1.594
1.585
1.579
1.571
1.562
1.555
1.547
1.542
1.530
1.514
1.478
1.465
1.460
1.447
1.431
1.416
1.410
O
OH
0.96
1.00
2.07
2.01
2.01
4.09
2.07
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0
f1 (ppm)

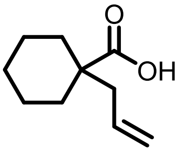
3c



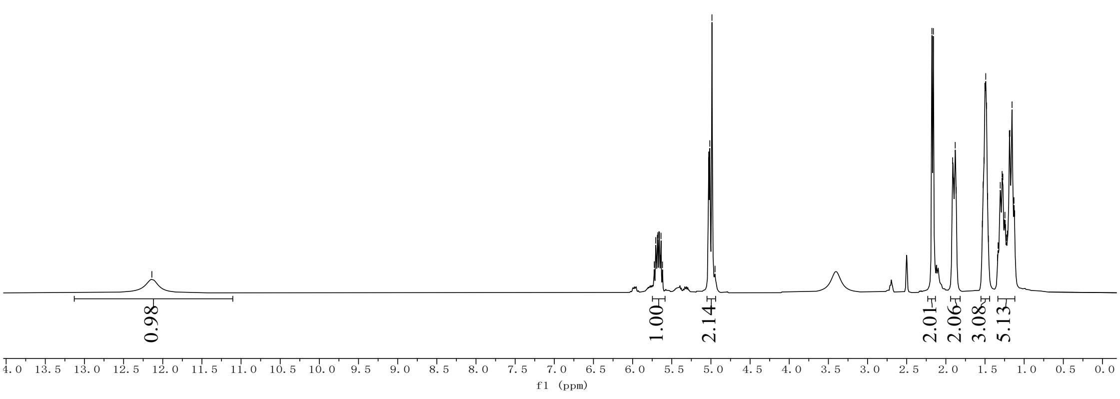
3c



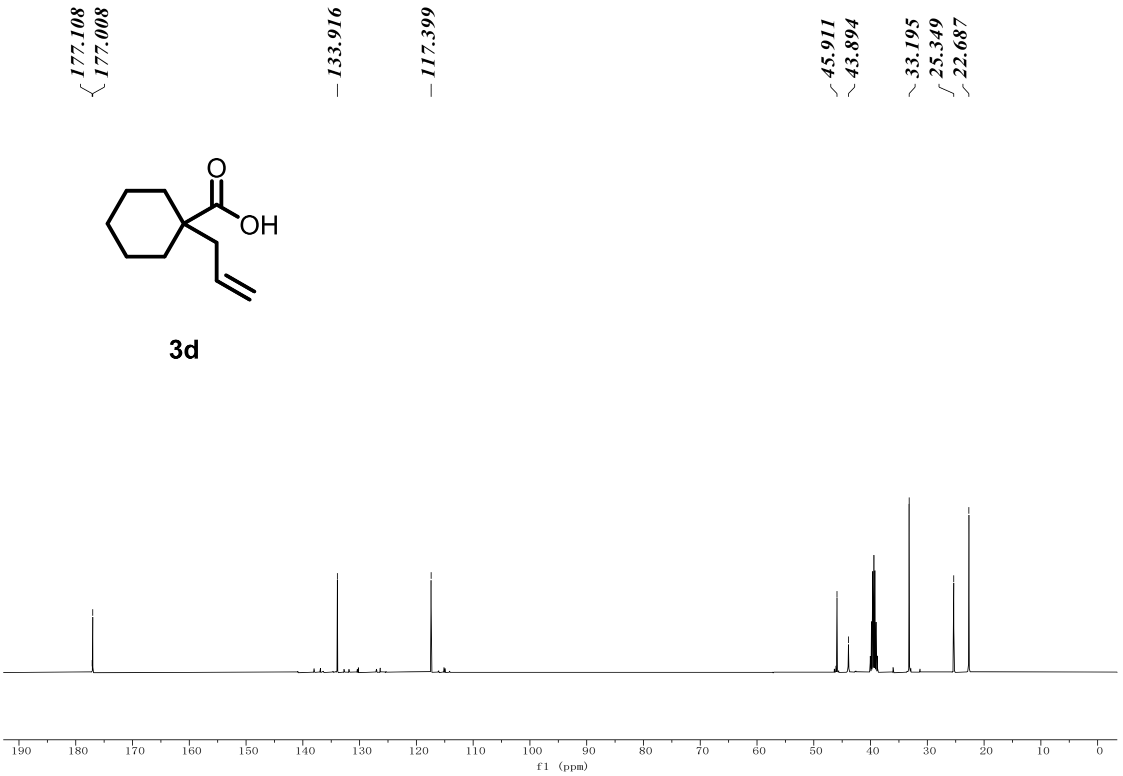
178.136
135.217
117.249
52.681
42.304
34.961
24.562
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)



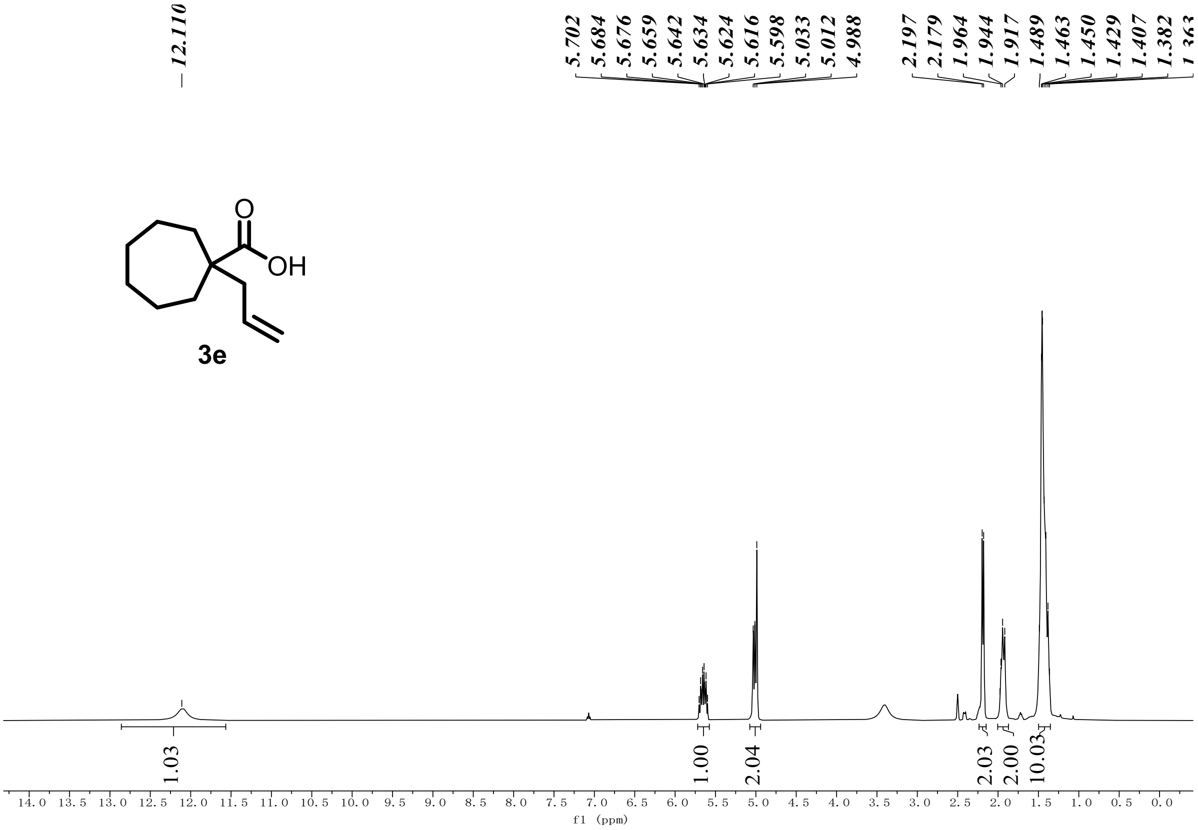
3d



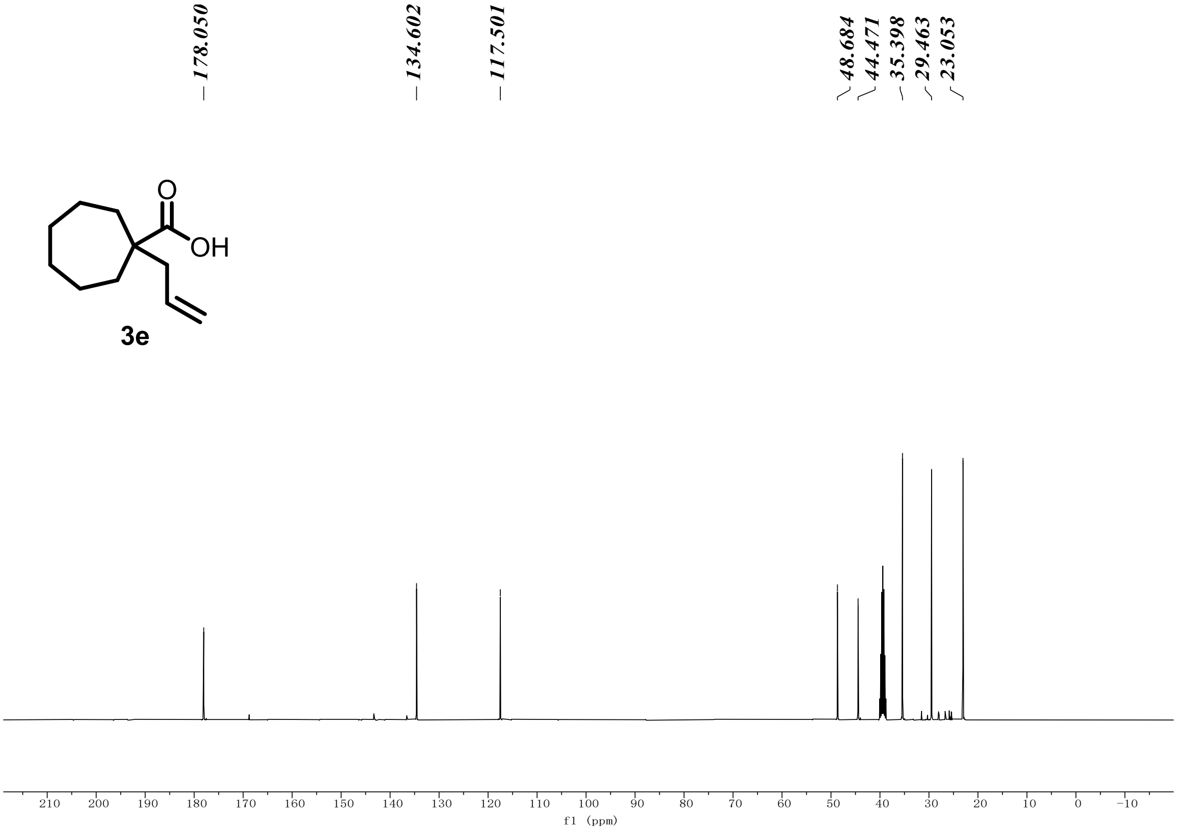
12.139
5.723
5.704
5.696
5.684
5.677
5.664
5.658
5.645
5.637
5.619
5.029
5.014
4.987
4.948
2.178
2.160
1.913
1.900
1.880
1.868
1.535
1.524
1.513
1.501
1.491
1.481
1.468
1.457
1.340
1.333
1.305
1.282
1.272
1.255
1.246
1.238
1.226
1.215
1.189
1.182
1.155
1.130
1.121
0.98
1.00
2.14
2.01
2.06
3.08
5.13
4.0
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
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
0.0
f1 (ppm)

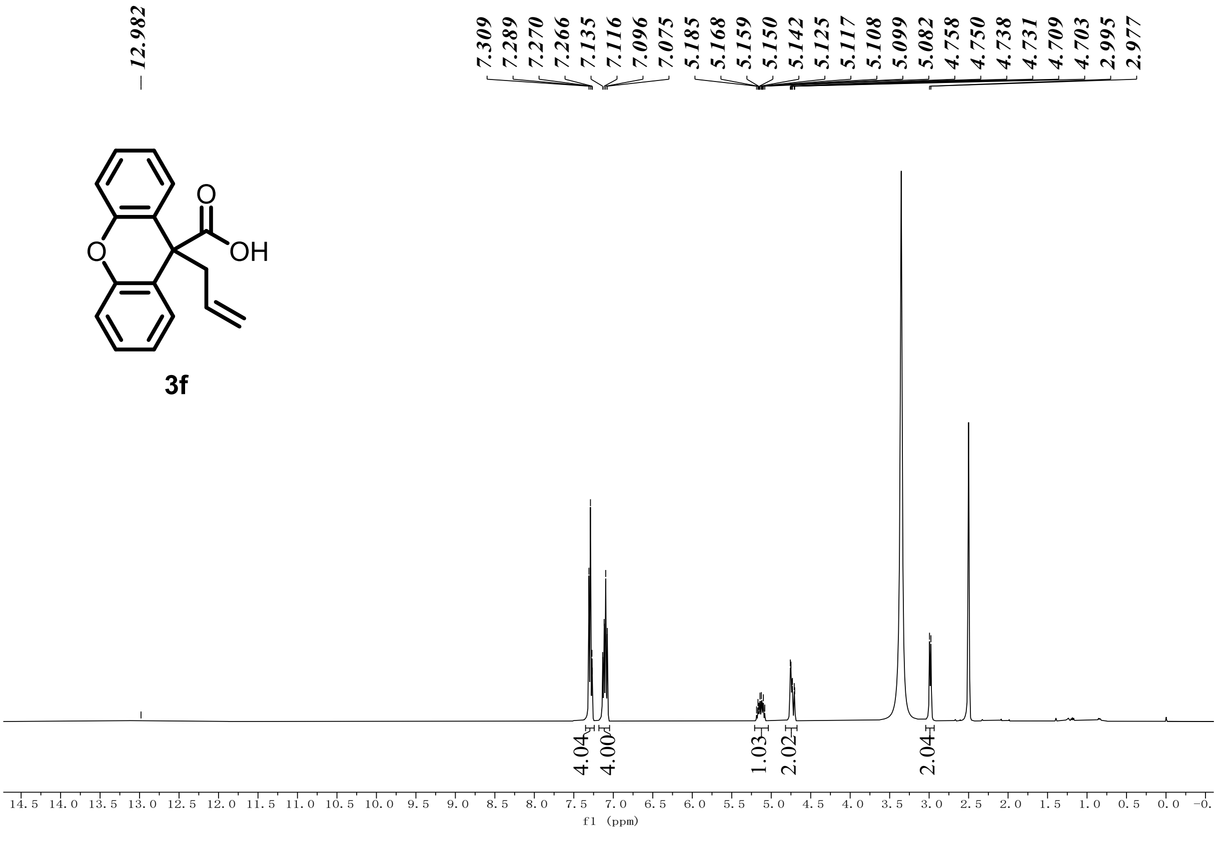

177.108
177.008
133.916
117.399
45.911
43.894
33.195
25.349
22.687
O
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

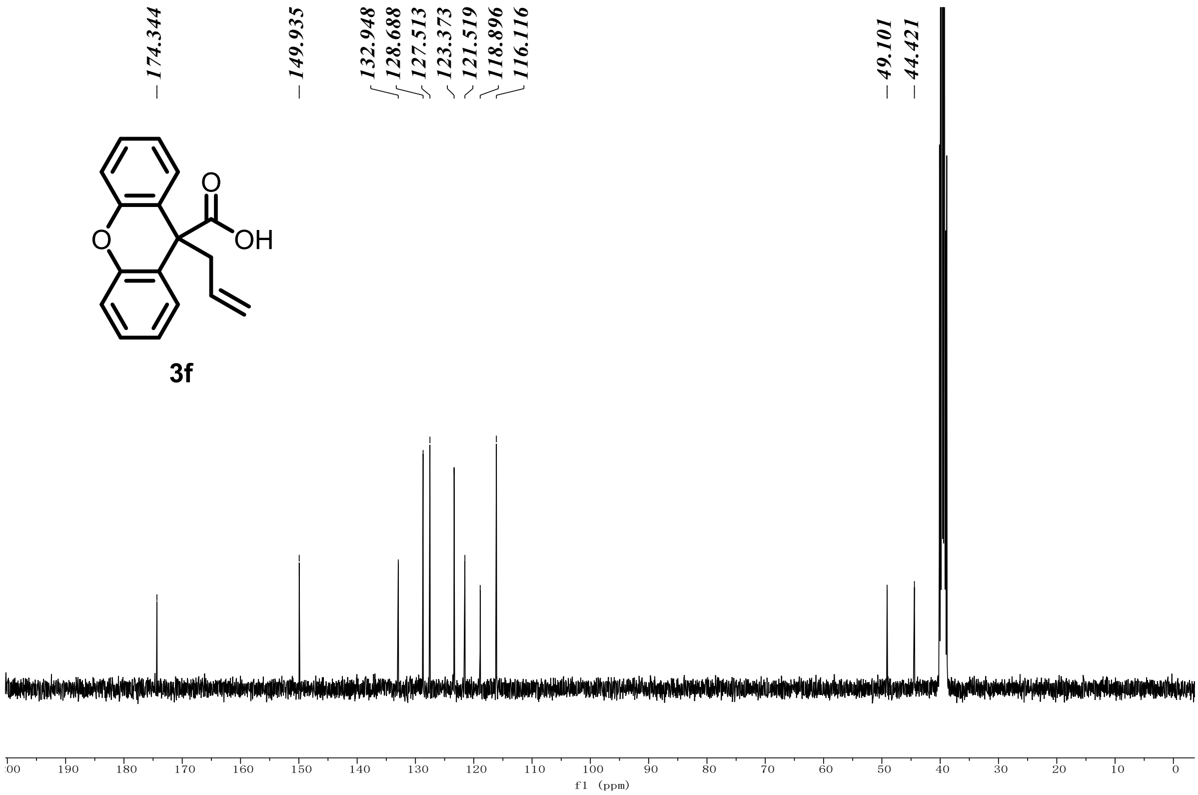
3d


12.110
5.702
5.684
5.676
5.659
5.642
5.634
5.624
5.616
5.598
5.033
5.012
4.988
2.197
2.179
1.964
1.944
1.917
1.489
1.463
1.450
1.429
1.407
1.382
O
OH
1.03
1.00
2.04
2.03
2.00
10.03
14.0
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
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
0.0
f1 (ppm)

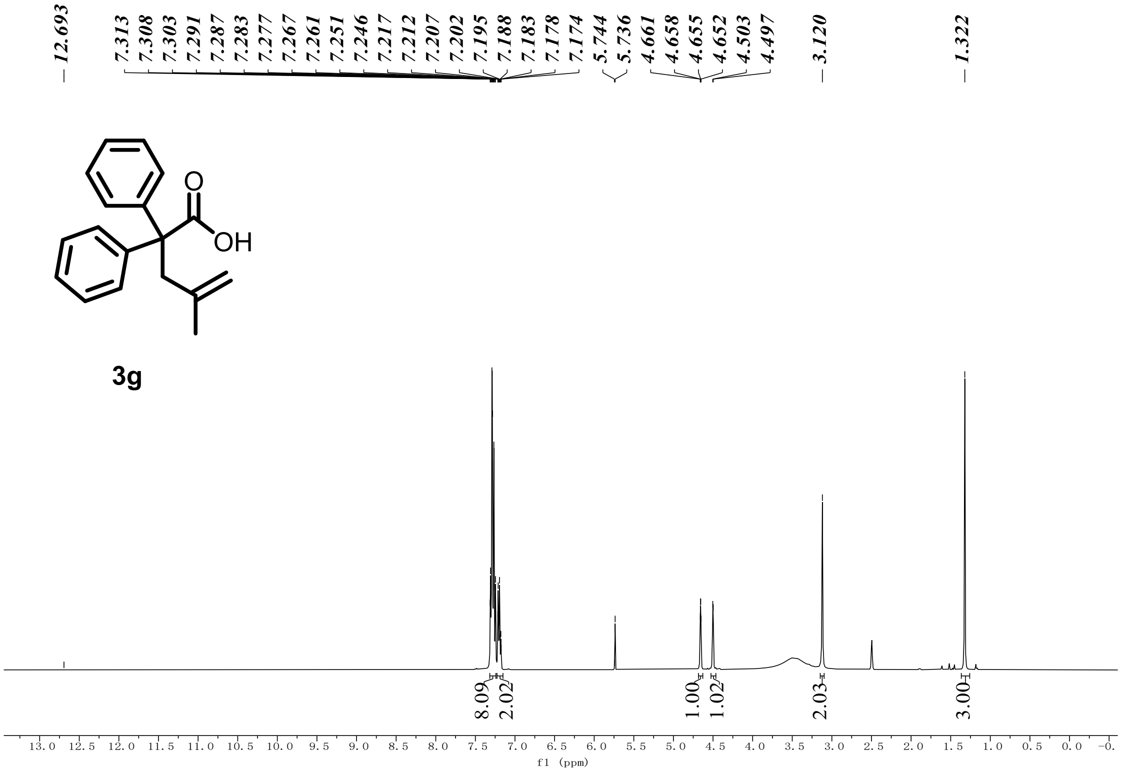
3e


178.050
134.602
117.501
48.684
44.471
35.398
29.463
23.053
O
OH
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

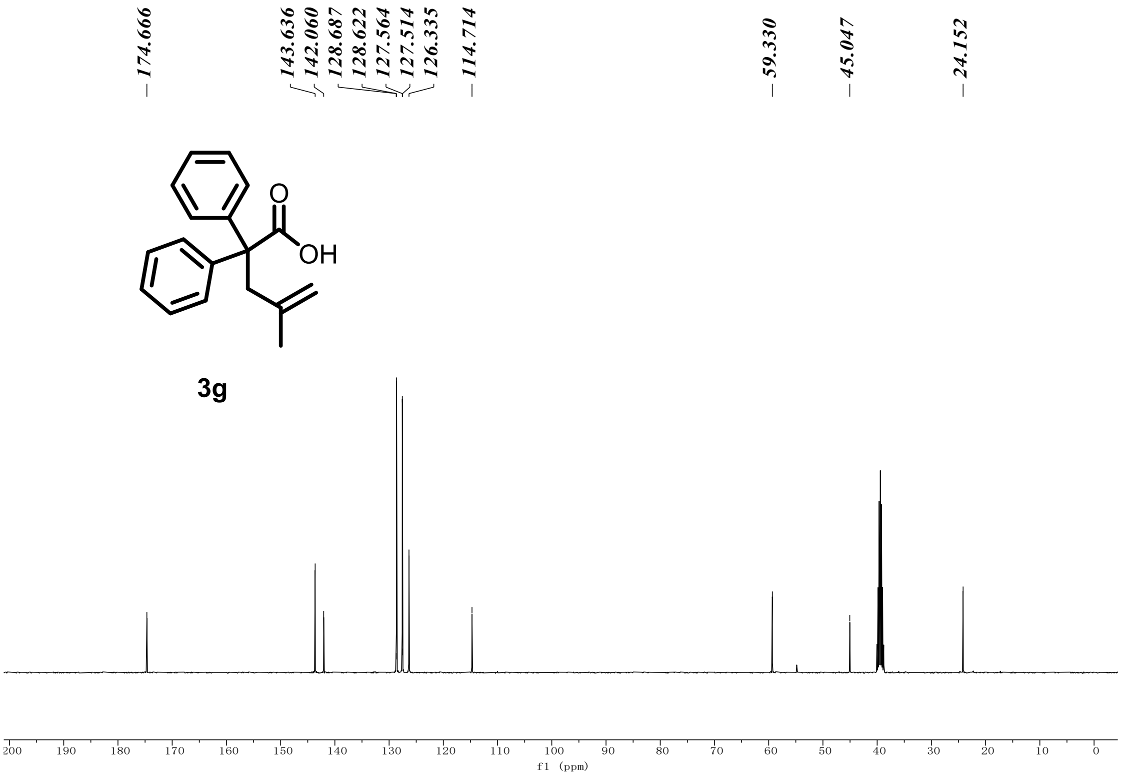

12.982
7.309
7.289
7.270
7.266
7.135
7.116
7.096
7.075
5.185
5.168
5.159
5.150
5.142
5.125
5.117
5.108
5.099
5.082
4.758
4.750
4.738
4.731
4.709
4.703
2.995
2.977
O
O
OH
3f
4.04
4.00
1.03
2.02
2.04
14.5
14.0
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.
f1 (ppm)

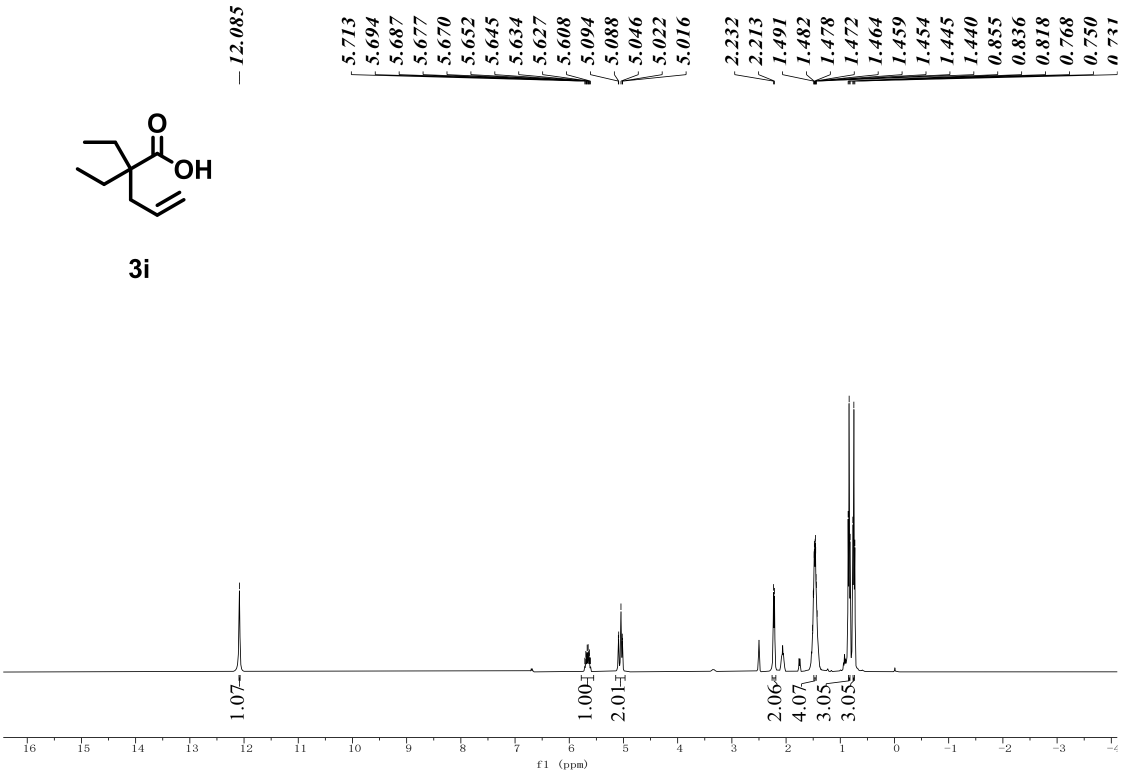

174.344
149.935
132.948
128.688
127.513
123.373
121.519
118.896
116.116
49.101
44.421
O
OH
O
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

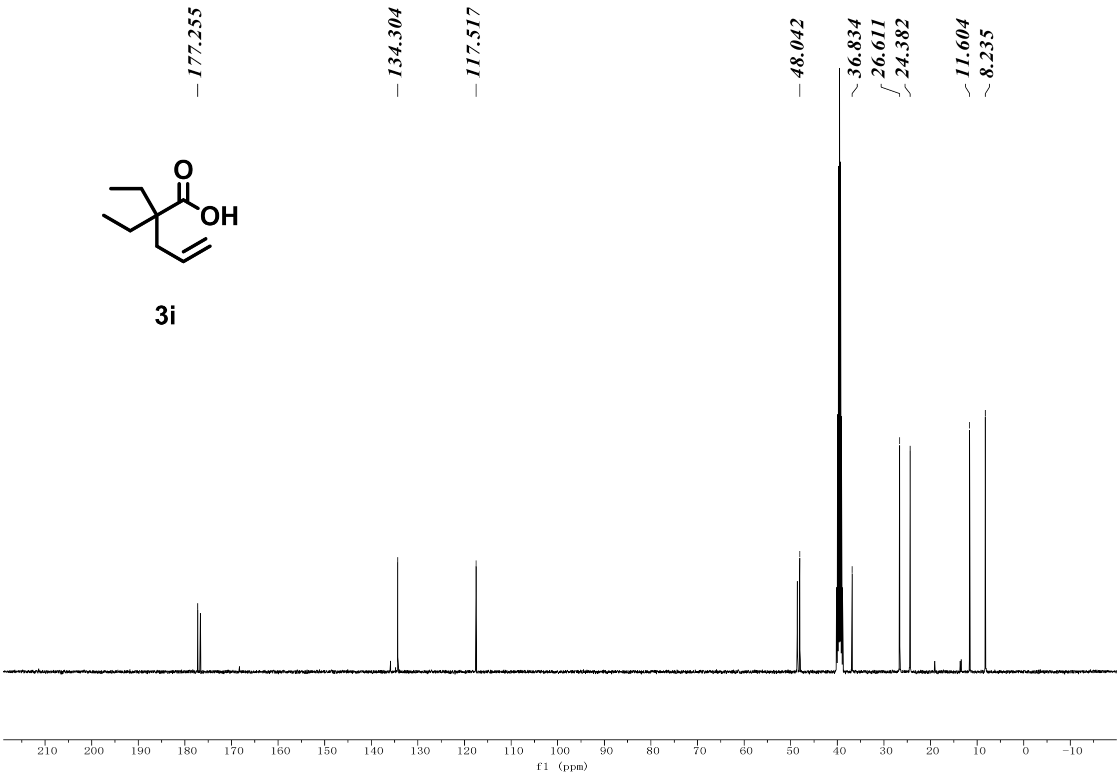
3f


12.693
7.313
7.308
7.303
7.291
7.287
7.283
7.277
7.267
7.261
7.251
7.246
7.217
7.212
7.207
7.202
7.195
7.188
7.183
7.178
7.174
5.744
5.736
4.661
4.658
4.655
4.652
4.503
4.497
3.120
1.322
O
OH
8.09
2.02
1.00
1.02
2.03
3.00
f1 (ppm)
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0 -0.

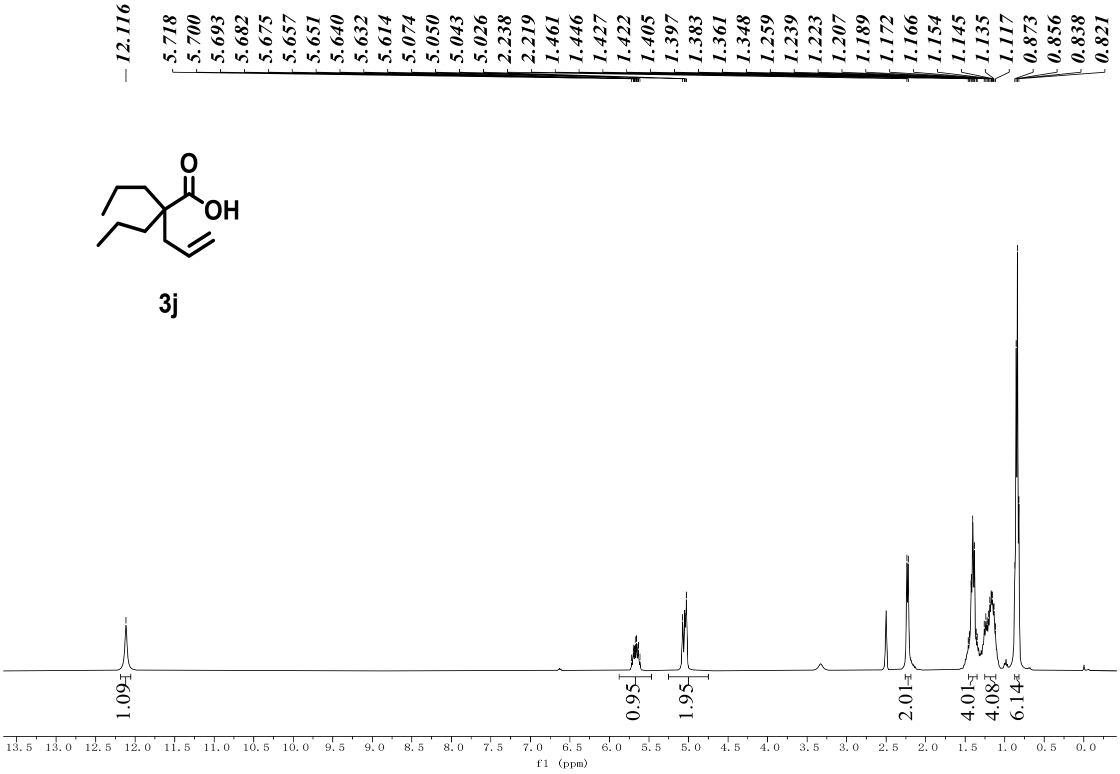
3g


174.666
143.636
142.060
128.687
128.622
127.564
127.514
126.335
114.714
59.330
45.047
24.152
O
OH
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

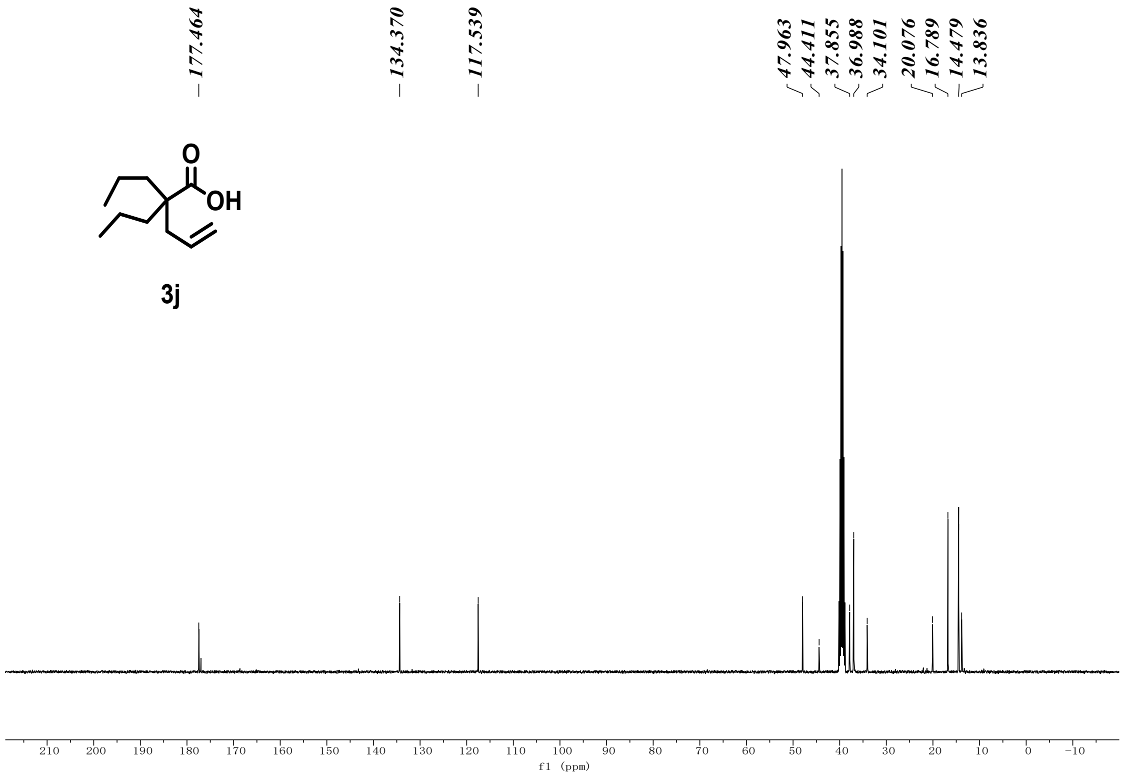

12.085
5.713
5.694
5.687
5.677
5.670
5.652
5.645
5.634
5.627
5.608
5.094
5.088
5.046
5.022
5.016
2.232
2.213
1.491
1.482
1.478
1.472
1.464
1.459
1.454
1.445
1.440
0.855
0.836
0.818
0.768
0.750
0.731
O
OH
3i
1.07
1.00
2.01
2.06
4.07
3.05
3.05
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
−4
f1 (ppm)

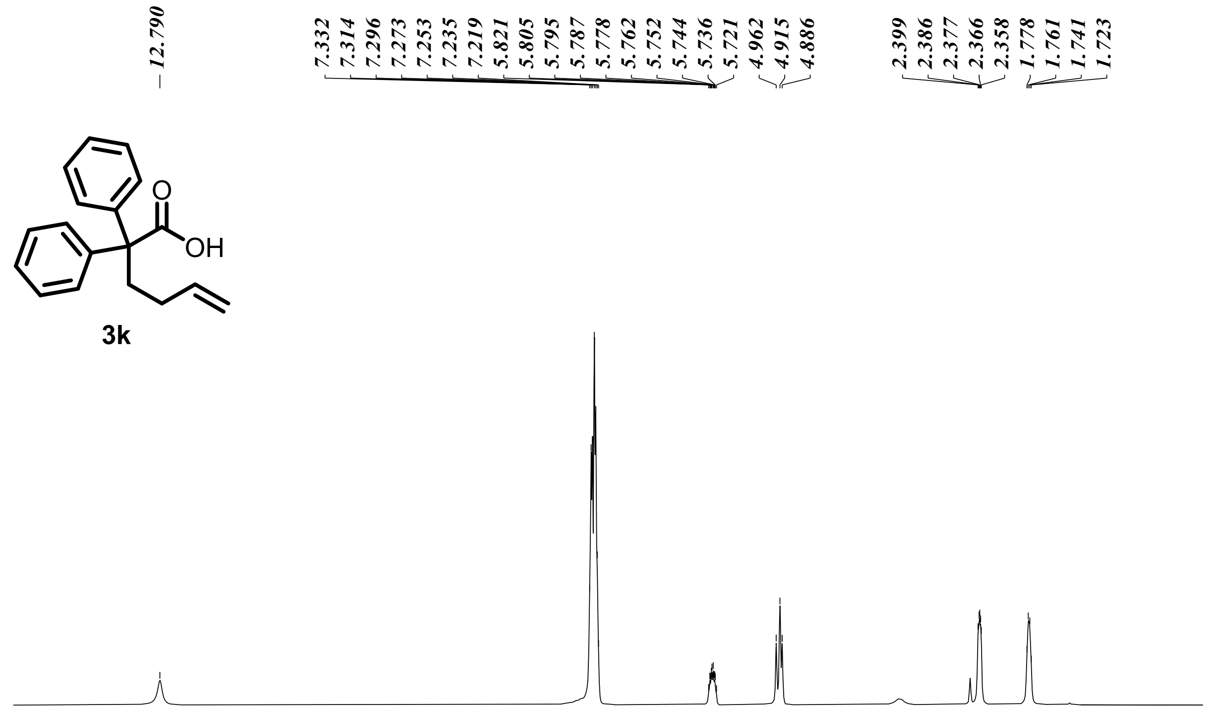

177.255
134.304
117.517
48.042
36.834
26.611
24.382
11.604
8.235
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

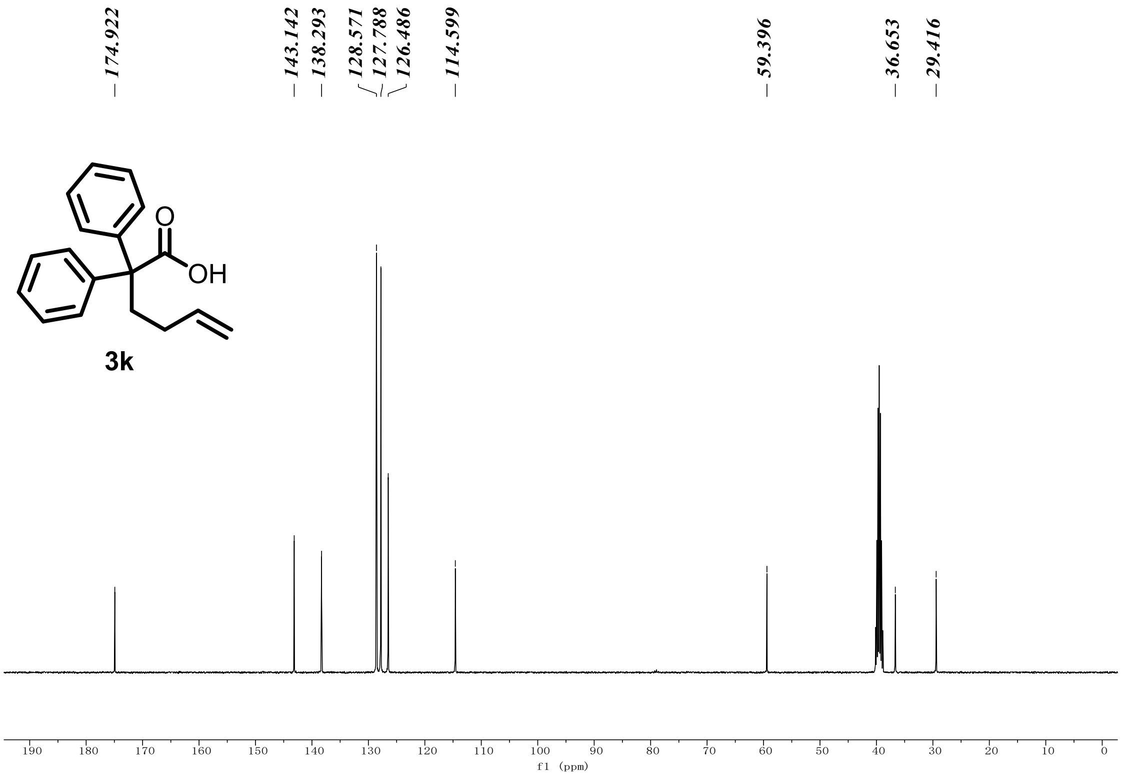
3i

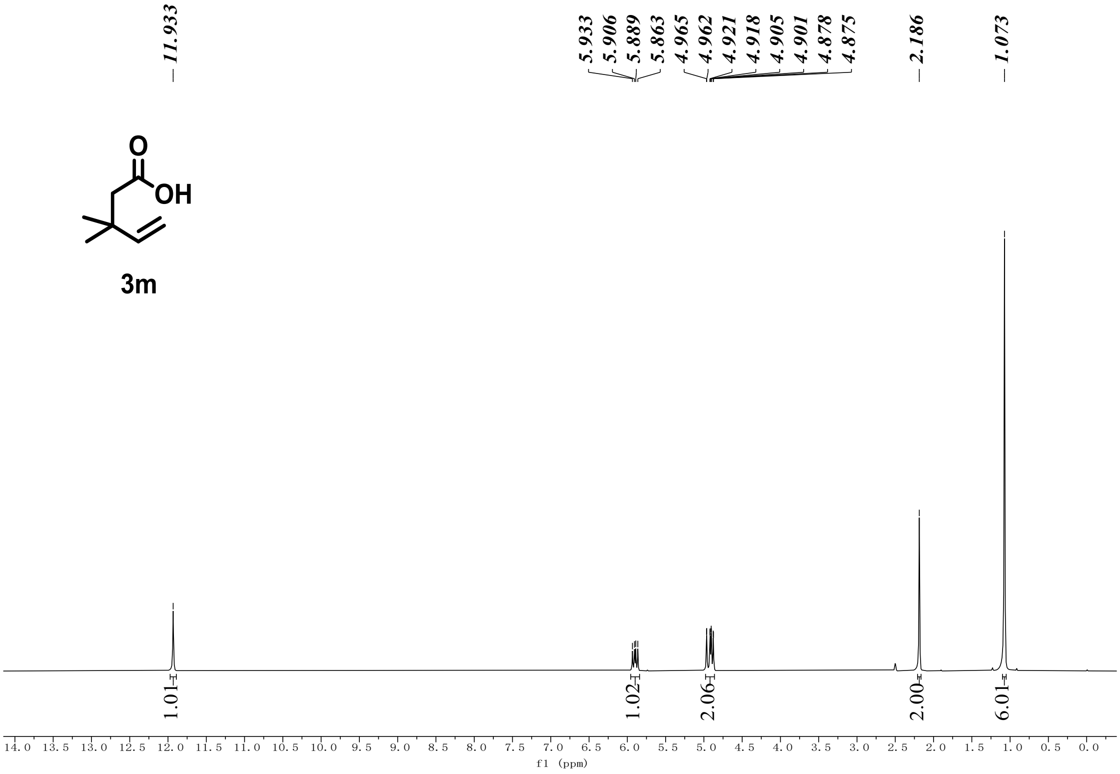

12.116
5.718
5.700
5.693
5.682
5.675
5.657
5.651
5.640
5.632
5.614
5.074
5.050
5.043
5.026
2.238
2.219
1.461
1.446
1.427
1.422
1.405
1.397
1.383
1.361
1.348
1.259
1.239
1.223
1.207
1.189
1.172
1.166
1.154
1.145
1.135
1.117
0.873
0.856
0.838
0.821
O
OH
1.09
0.95
1.95
2.01
4.01
4.08
6.14
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0
f1 (ppm)

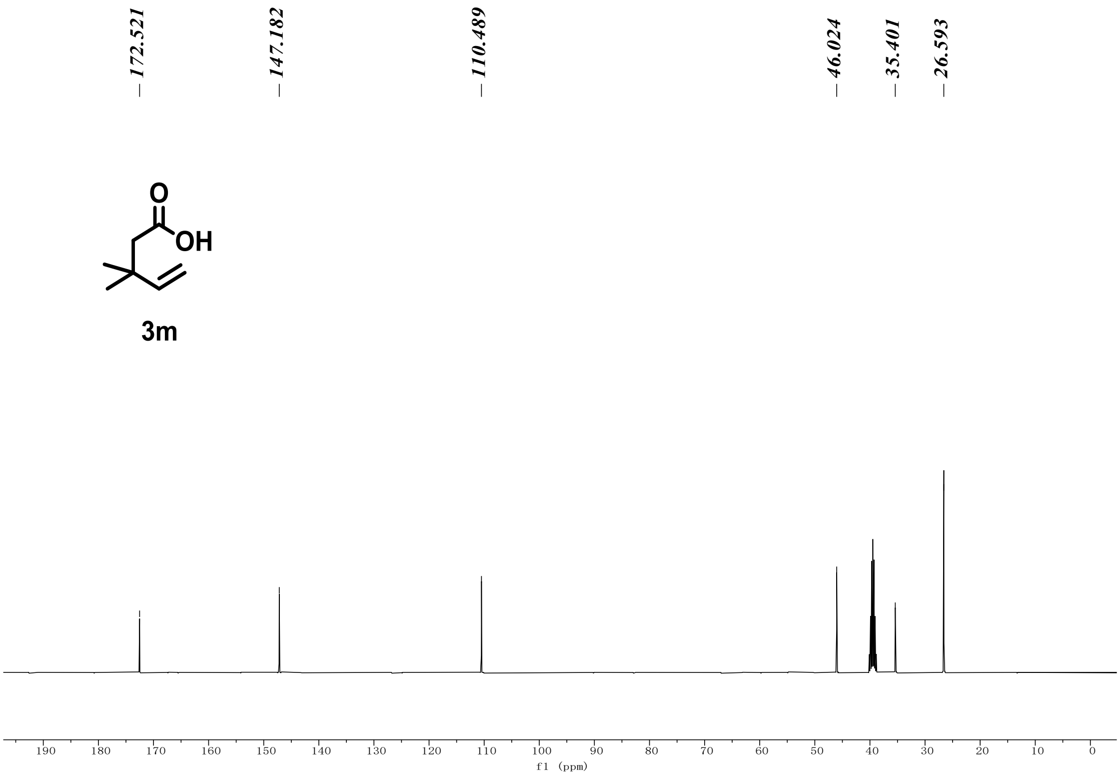
3j


177.464
134.370
117.539
47.963
44.411
37.855
36.988
34.101
20.076
16.789
14.479
13.836
O
OH
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

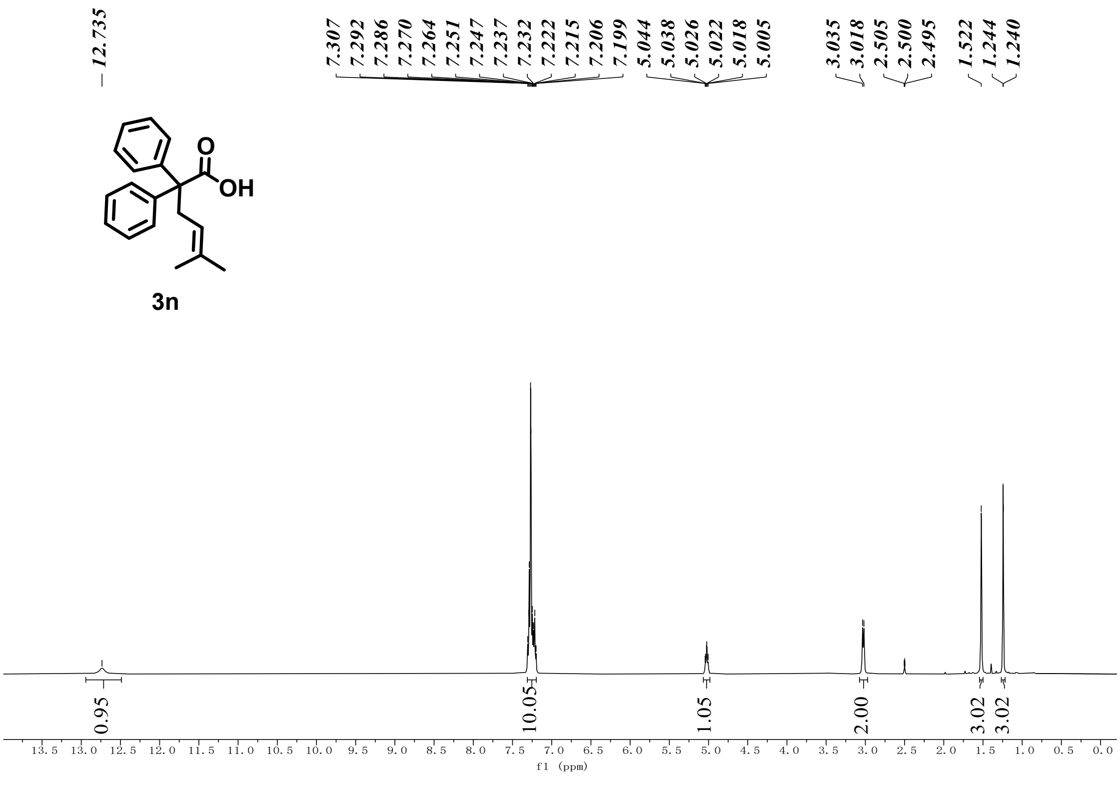

12.790
7.332
7.314
7.296
7.273
7.253
7.235
7.219
5.821
5.805
5.795
5.787
5.778
5.762
5.752
5.744
5.736
5.721
4.962
4.915
4.886
2.399
2.386
2.377
2.366
2.358
1.778
1.761
1.741
1.723
O
OH
3k
1.02
10.06
1.00
2.00
2.01
2.01
14.5 14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


174.922
143.142
138.293
128.571
127.788
126.486
114.599
59.396
36.653
29.416
O
OH
3k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

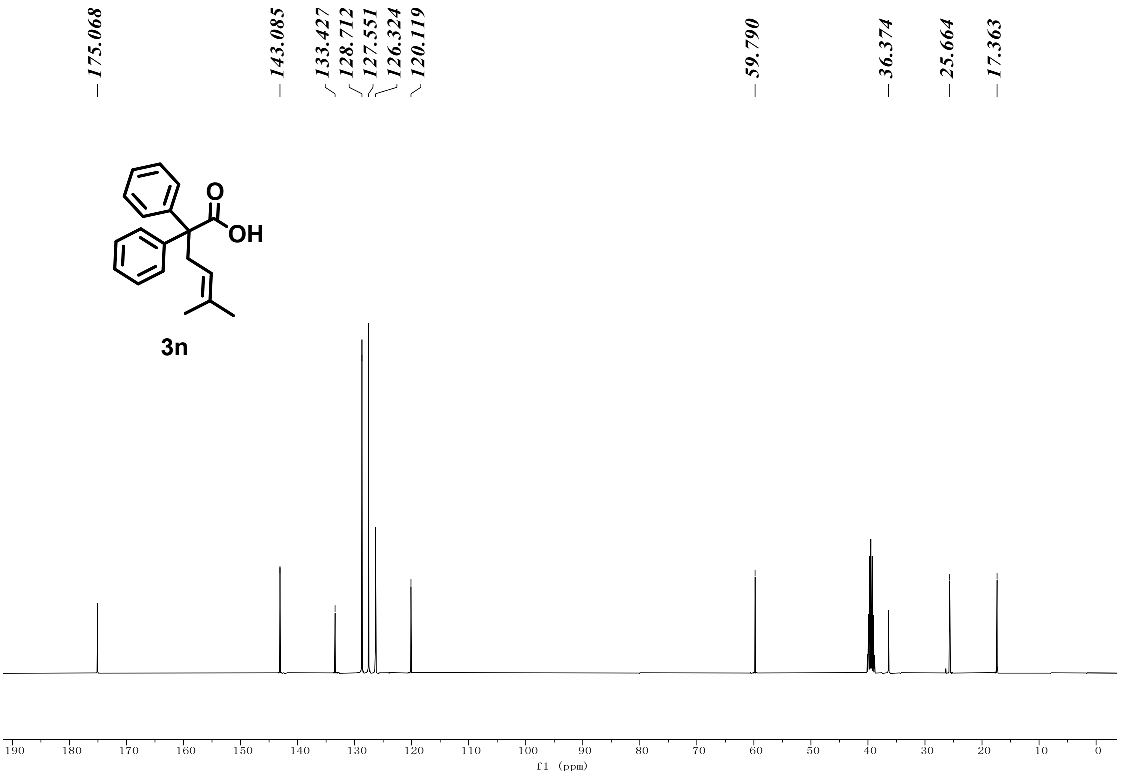

11.933
5.933
5.906
5.889
5.863
4.965
4.962
4.921
4.918
4.905
4.901
4.878
4.875
2.186
1.073
O
OH
3m
1.01
1.02
2.06
2.00
6.01
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

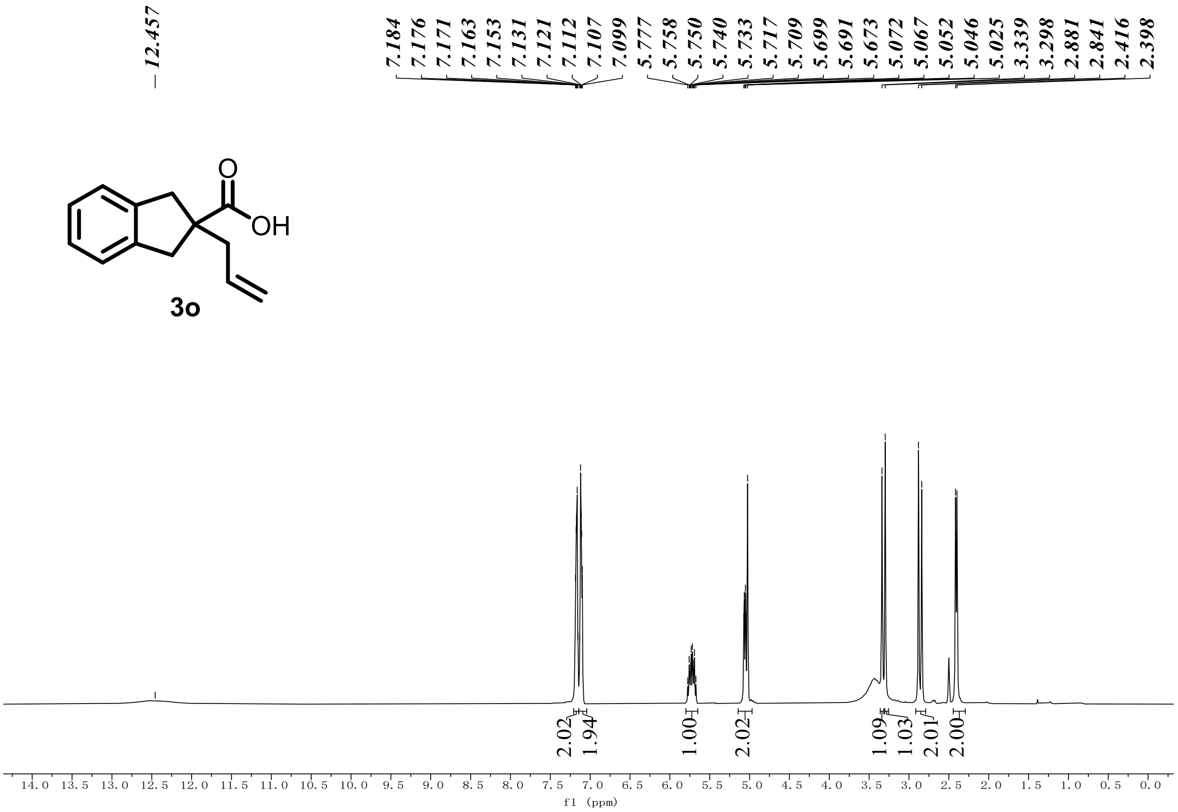

172.521
147.182
110.489
46.024
35.401
26.593
OH
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

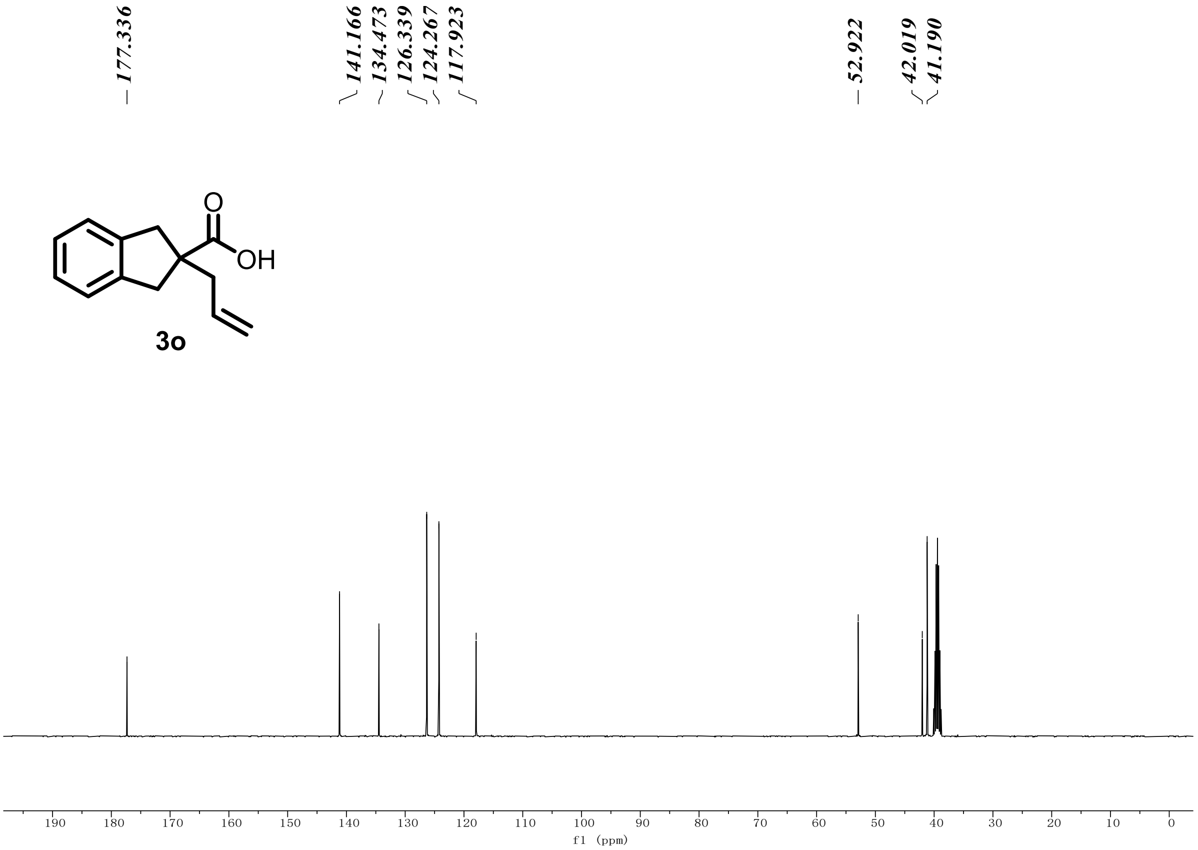
3m


12.735
7.307
7.292
7.286
7.270
7.264
7.251
7.247
7.237
7.232
7.222
7.215
7.206
7.199
5.044
5.038
5.026
5.022
5.018
5.005
3.035
3.018
2.505
2.500
2.495
1.522
1.244
1.240
O
OH
0.95
10.05
1.05
2.00
3.02
3.02
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0
f1 (ppm)

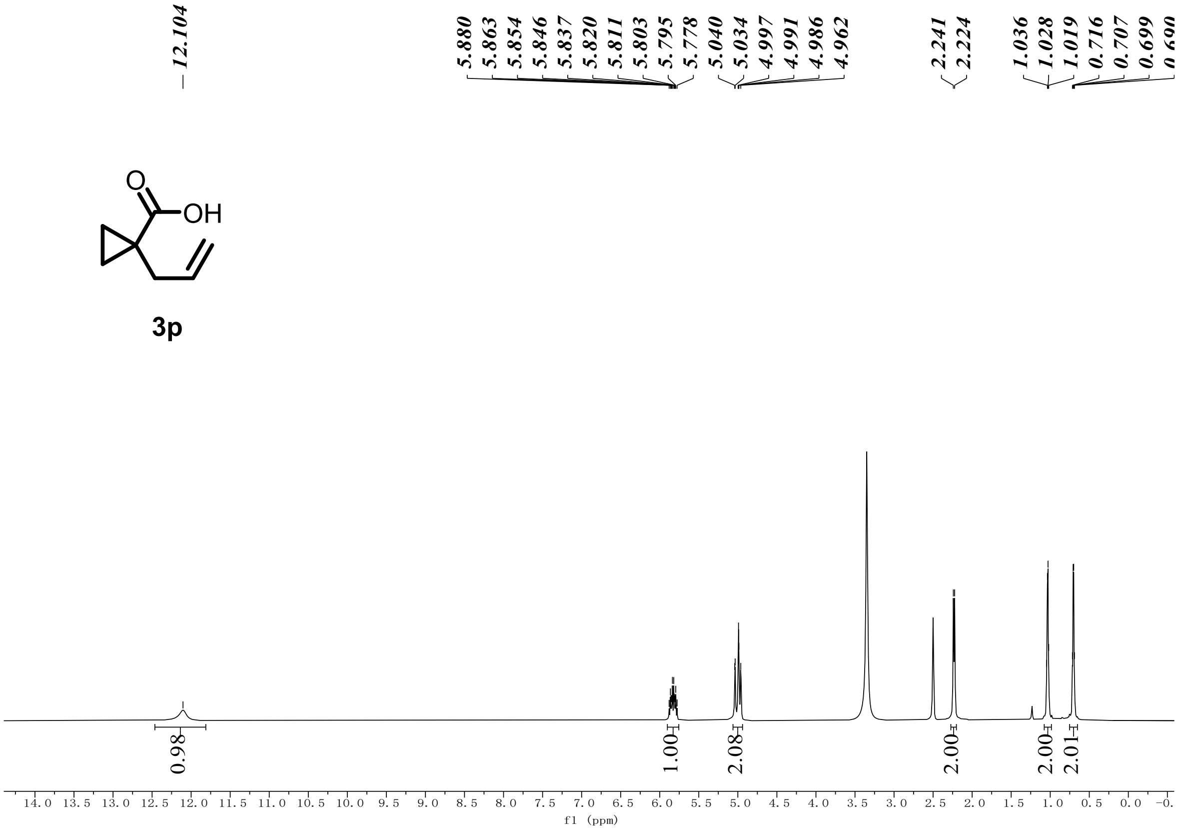
3n


175.068
143.085
133.427
128.712
127.551
126.324
120.119
59.790
36.374
25.664
17.363
OH
O
3n
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

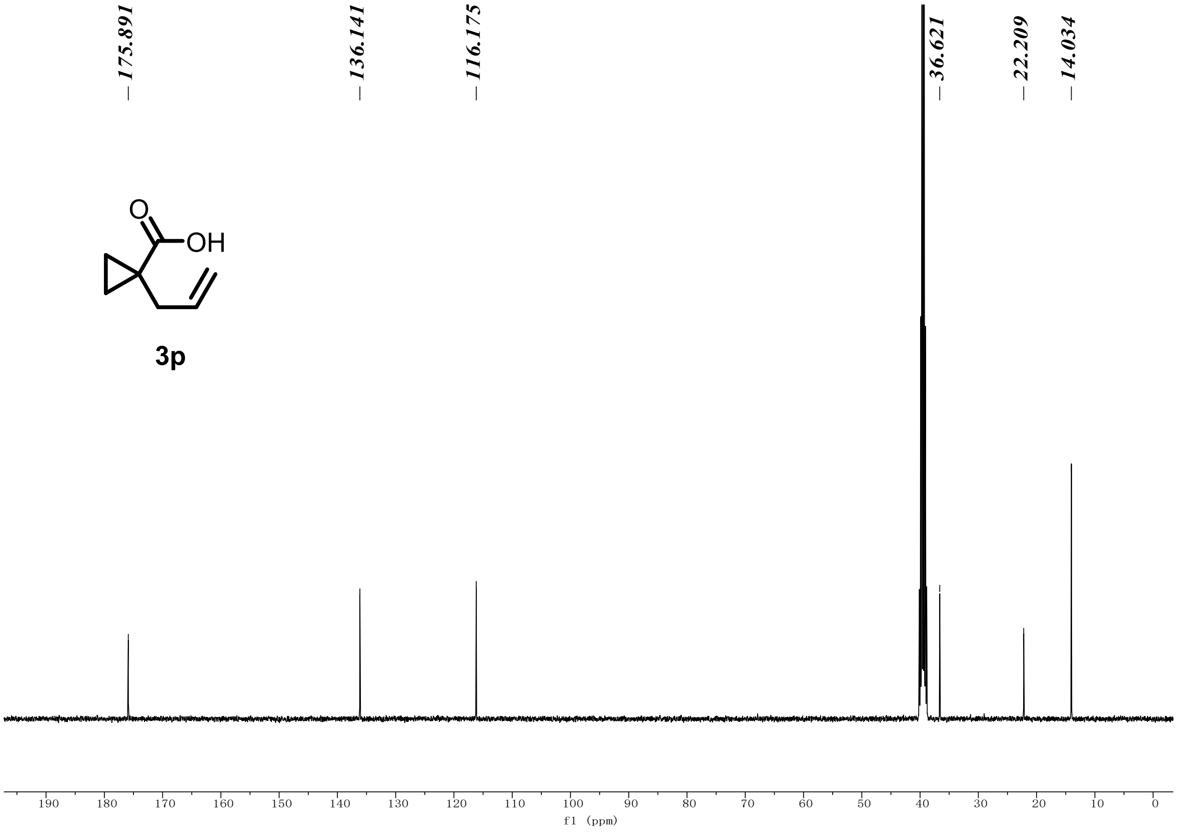

12.457
7.184
7.176
7.171
7.163
7.153
7.131
7.121
7.112
7.107
7.099
5.777
5.758
5.750
5.740
5.733
5.717
5.709
5.699
5.691
5.673
5.072
5.067
5.052
5.046
5.025
3.339
3.298
2.881
2.841
2.416
2.398
O
OH
3o
2.02
1.94
1.00
2.02
1.09
1.03
2.01
2.00
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 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 0.0
f1 (ppm)

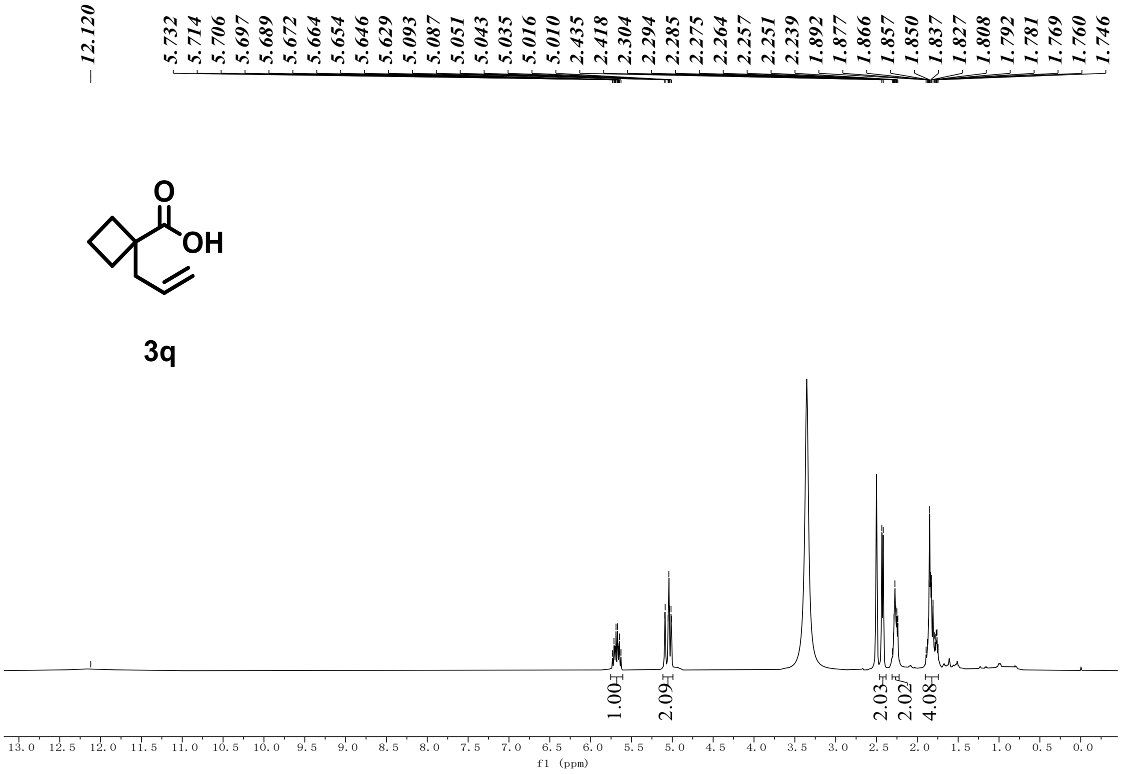

177.336
141.166
134.473
126.339
124.267
117.923
52.922
42.019
41.190
O
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

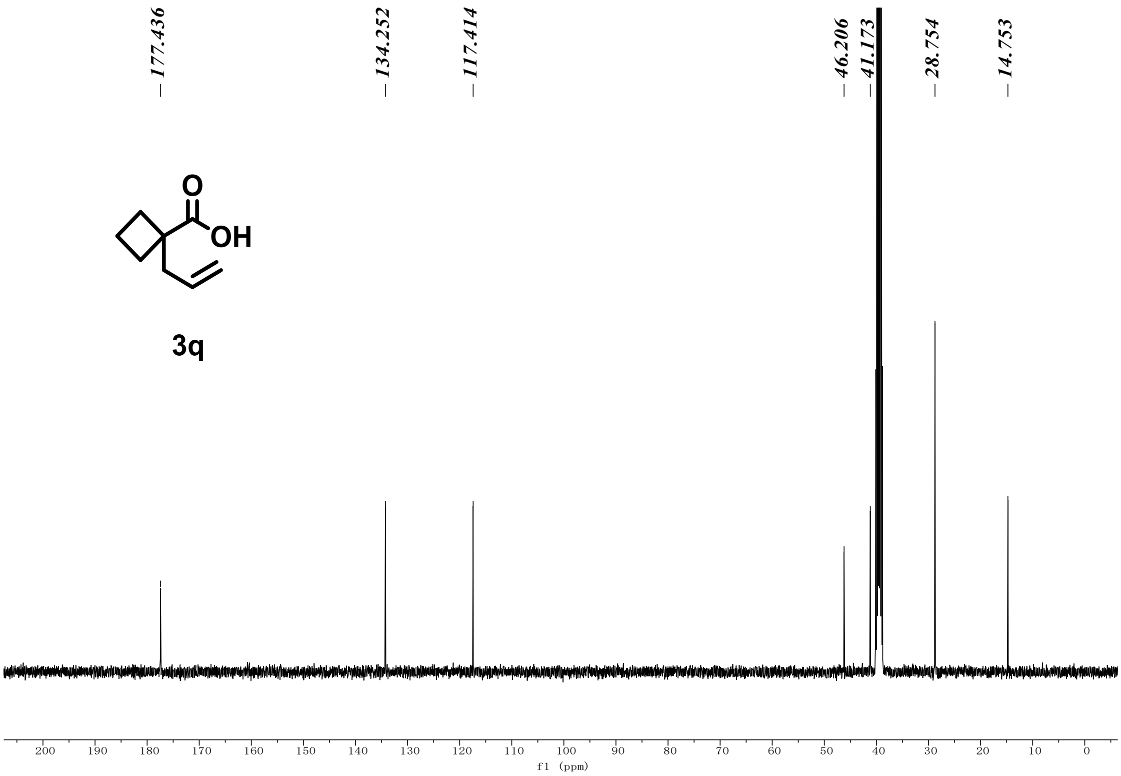
3o


12.104
5.880
5.863
5.854
5.846
5.837
5.820
5.811
5.803
5.795
5.778
5.040
5.034
4.997
4.991
4.986
4.962
2.241
2.224
1.036
1.028
1.019
0.716
0.707
0.699
0.690
O
OH
0.98
1.00
2.08
2.00
2.00
2.01
14.0
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
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
0.0
-0.
f1 (ppm)

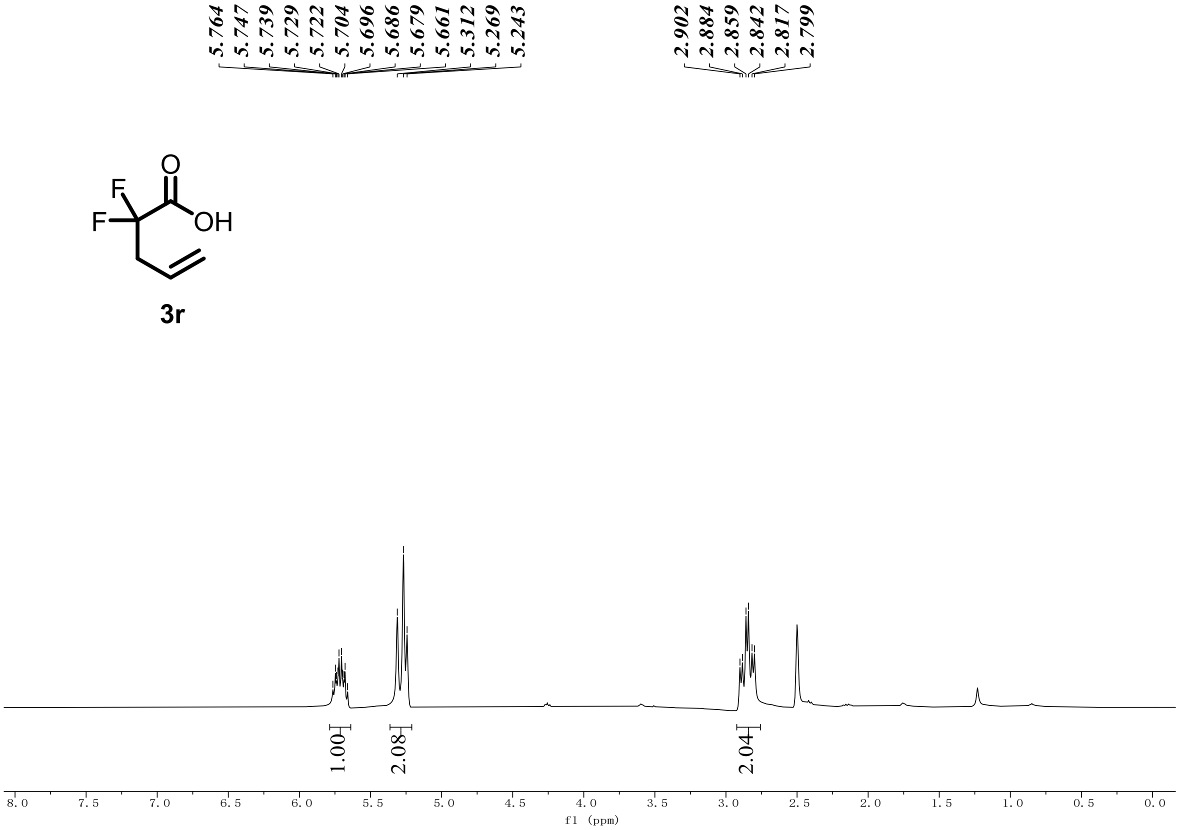
3p


175.891
136.141
116.175
36.621
22.209
14.034
3p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

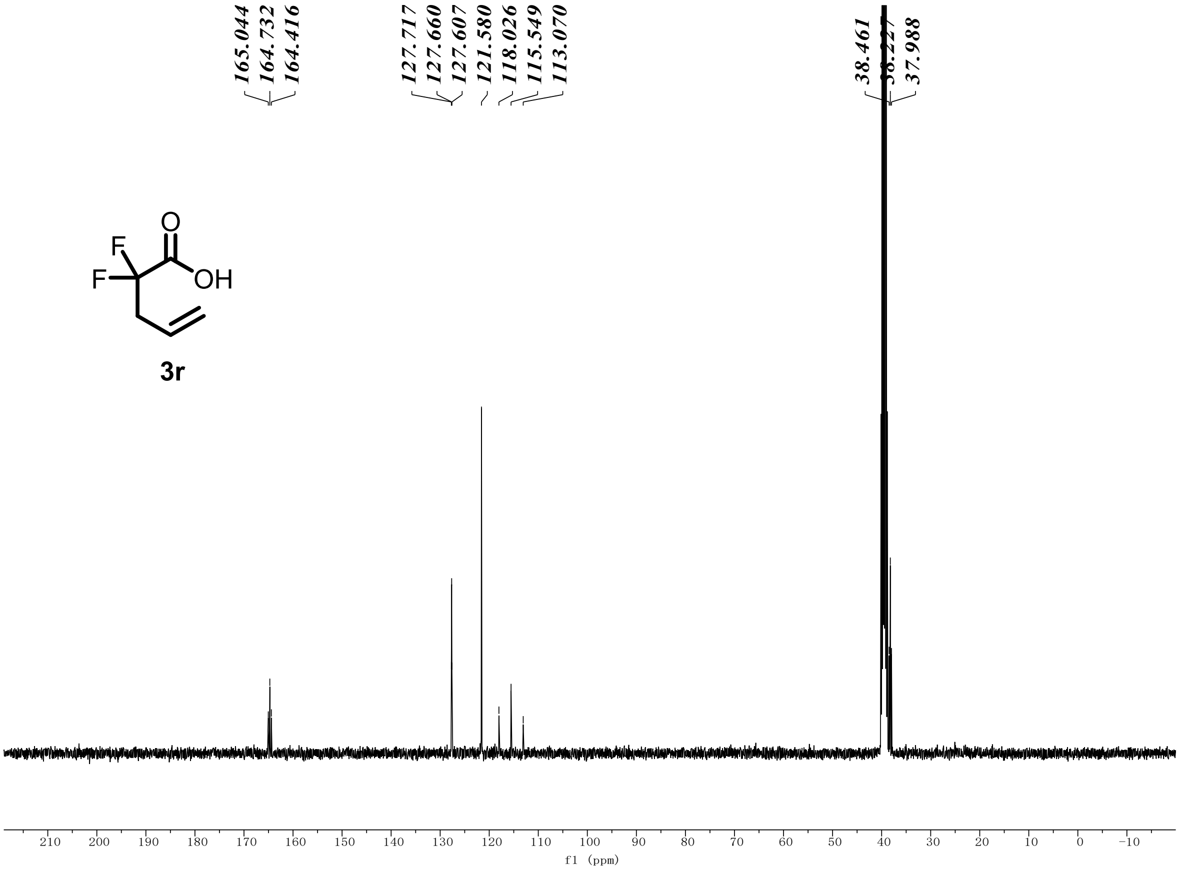

12.120
5.732
5.714
5.706
5.697
5.689
5.672
5.664
5.654
5.646
5.629
5.093
5.087
5.051
5.043
5.035
5.016
5.010
2.435
2.418
2.304
2.294
2.285
2.275
2.264
2.257
2.251
2.239
1.892
1.877
1.866
1.857
1.850
1.837
1.827
1.808
1.792
1.781
1.769
1.760
1.746
3q
1.00
2.09
2.03
2.02
4.08
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 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 0.0
f1 (ppm)

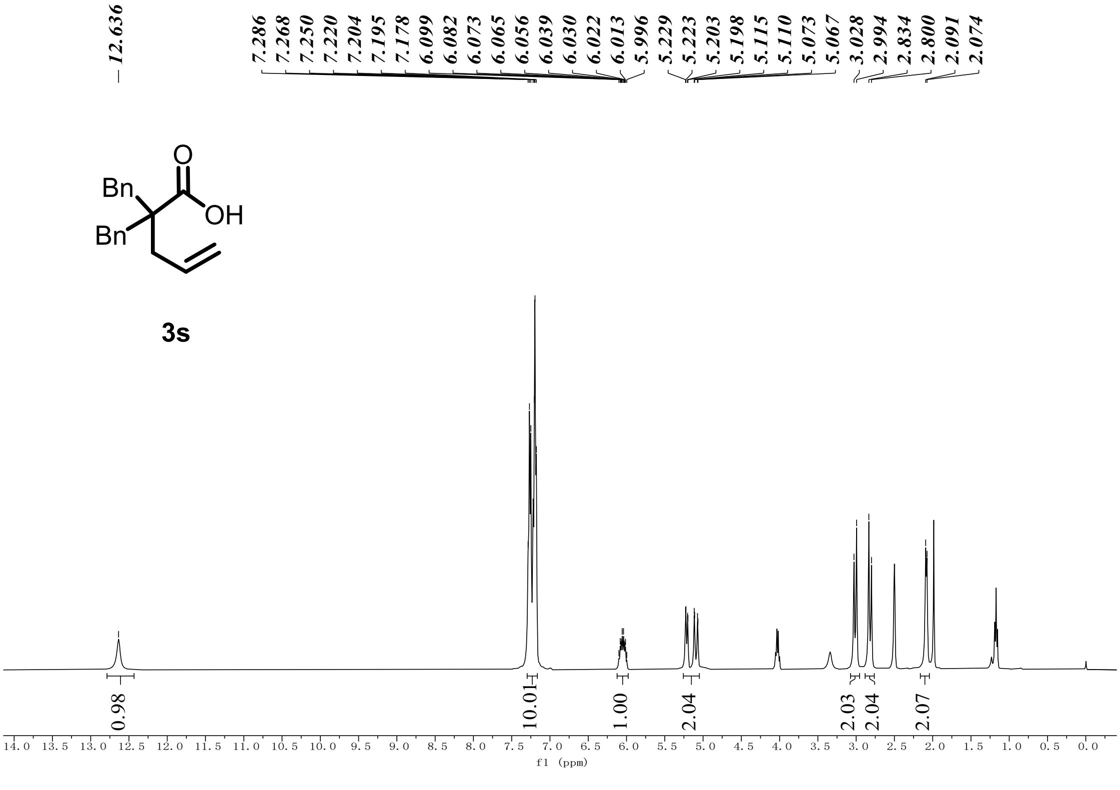

177.436
134.252
117.414
46.206
41.173
28.754
14.753
O
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

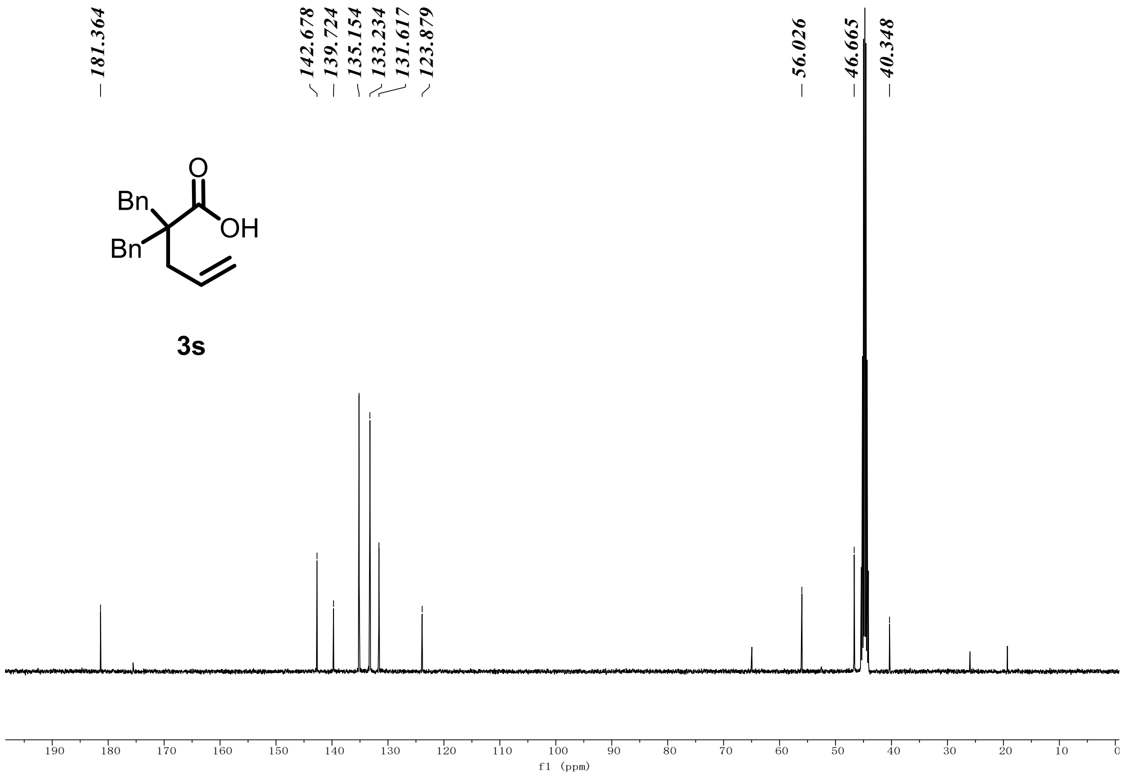
3q


5.764
5.747
5.739
5.729
5.722
5.704
5.696
5.686
5.679
5.661
5.312
5.269
5.243
2.902
2.884
2.859
2.842
2.817
2.799
O
F
F
OH
1.00
2.08
2.04
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)

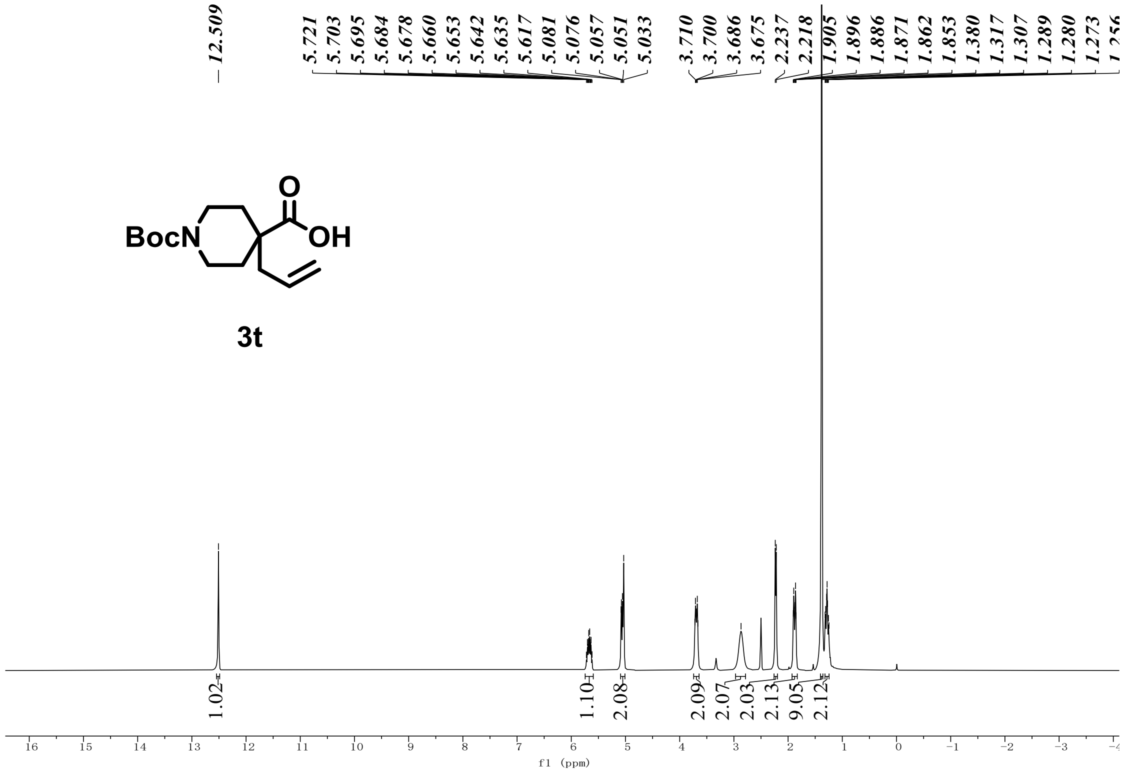
3r


165.044
164.732
164.416
127.717
127.660
127.607
121.580
118.026
115.549
113.070
38.461
38.227
37.988
F
F
O
OH
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

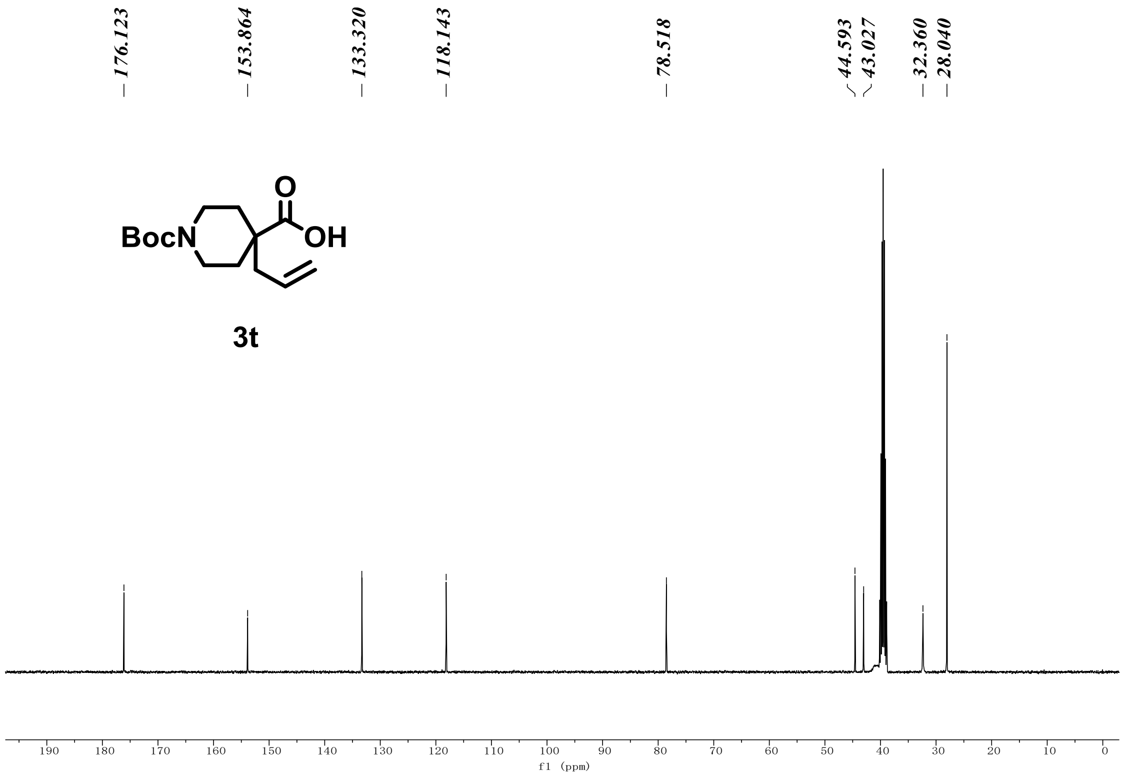

12.636
7.286
7.268
7.250
7.220
7.204
7.195
7.178
6.099
6.082
6.073
6.065
6.056
6.039
6.030
6.022
6.013
5.996
5.229
5.223
5.203
5.198
5.115
5.110
5.073
5.067
3.028
2.994
2.834
2.800
2.091
2.074
Bn
Bn
O
OH
3s
0.98
10.01
1.00
2.04
2.03
2.04
2.07
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 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 0.0
f1 (ppm)


181.364
142.678
139.724
135.154
133.234
131.617
123.879
56.026
46.665
40.348
Bn
Bn
O
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

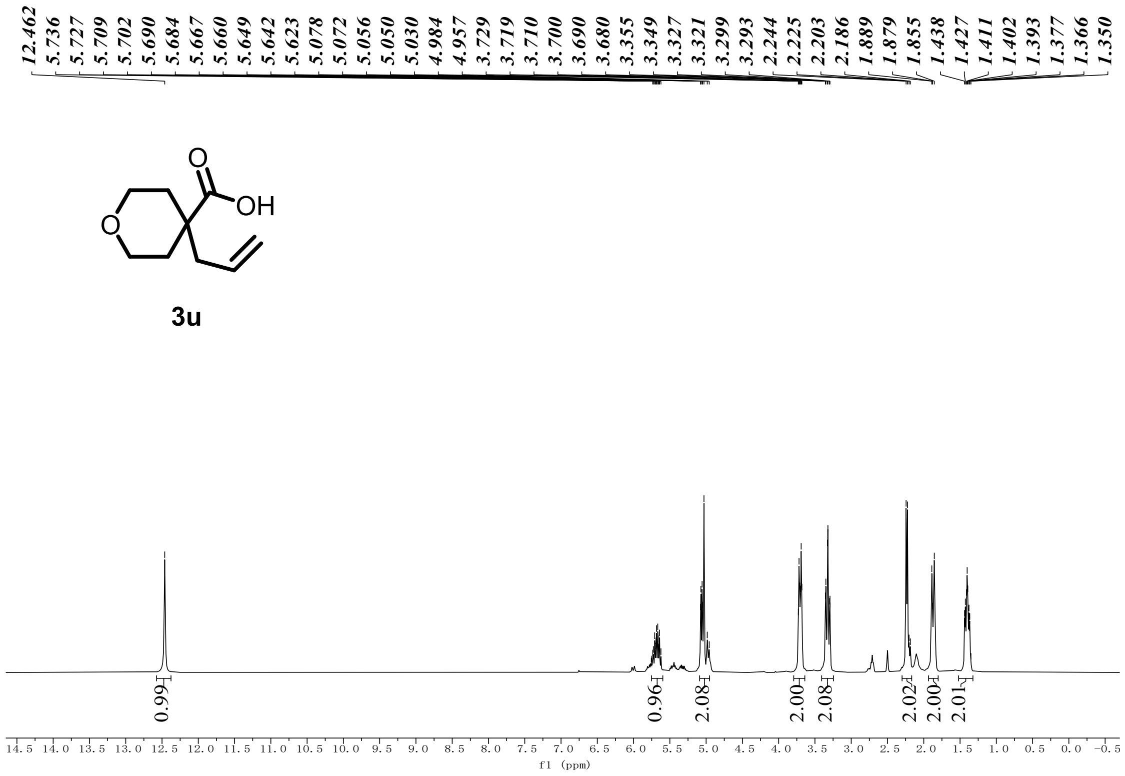
3s


12.509
5.721
5.703
5.695
5.684
5.678
5.660
5.653
5.642
5.635
5.617
5.081
5.076
5.057
5.051
5.033
3.710
3.700
3.686
3.675
2.237
2.218
1.905
1.896
1.886
1.871
1.862
1.853
1.380
1.317
1.307
1.289
1.280
1.273
BocN
O
OH
1.02
1.10
2.08
2.09
2.07
2.03
2.13
9.05
2.12
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

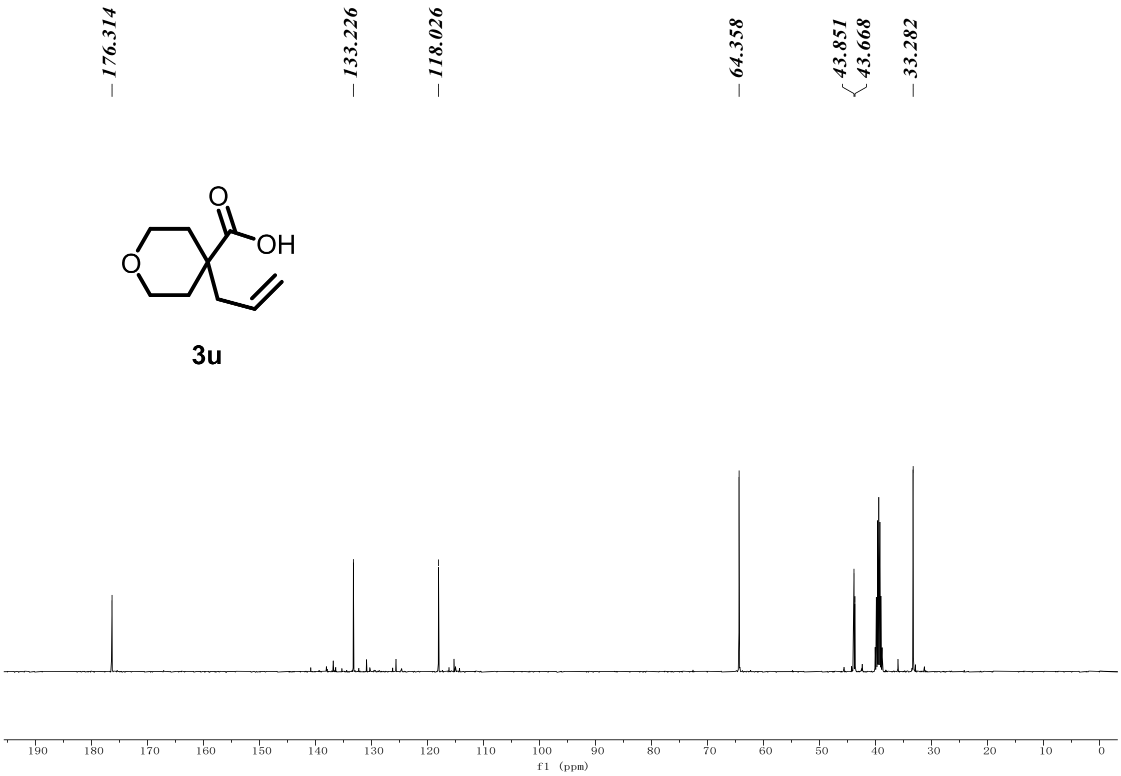
3t


176.123
153.864
133.320
118.143
78.518
44.593
43.027
32.360
28.040
BocN
O
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

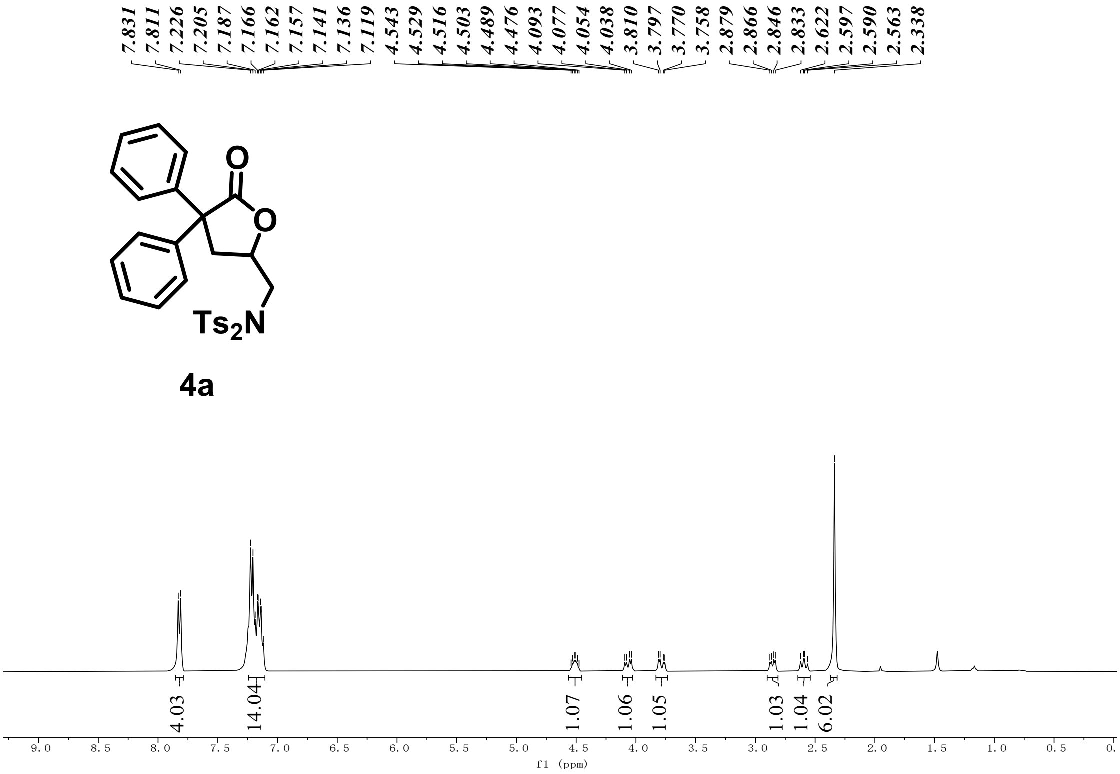
3t


12.462
5.736
5.727
5.709
5.702
5.690
5.684
5.667
5.660
5.649
5.642
5.623
5.078
5.072
5.056
5.050
5.030
4.984
4.957
3.729
3.719
3.710
3.700
3.690
3.680
3.355
3.349
3.327
3.321
3.299
3.293
2.244
2.225
2.203
2.186
1.889
1.879
1.855
1.438
1.427
1.411
1.402
1.393
1.377
1.366
1.350
O
O
OH
0.99
0.96
2.08
2.00
2.08
2.02
2.00
2.01
14.5
14.0
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
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
0.0
-0.5
f1 (ppm)

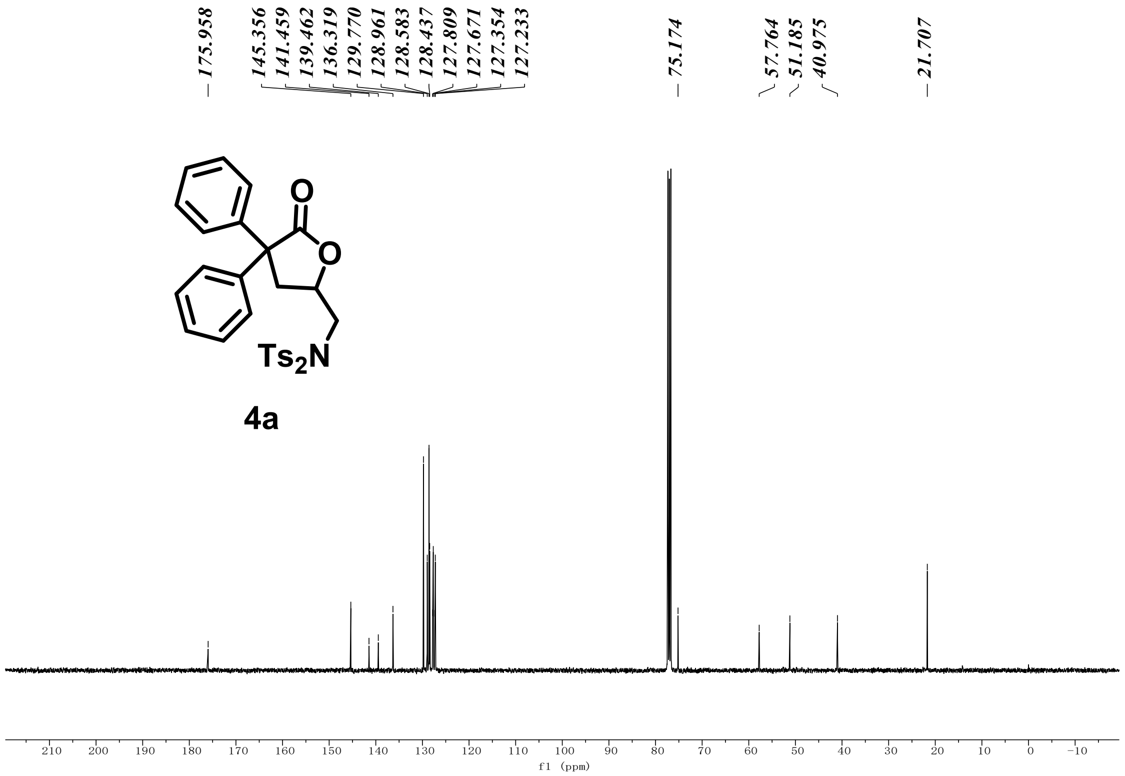
3u


176.314
133.226
118.026
64.358
43.851
43.668
33.282
O
OH
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

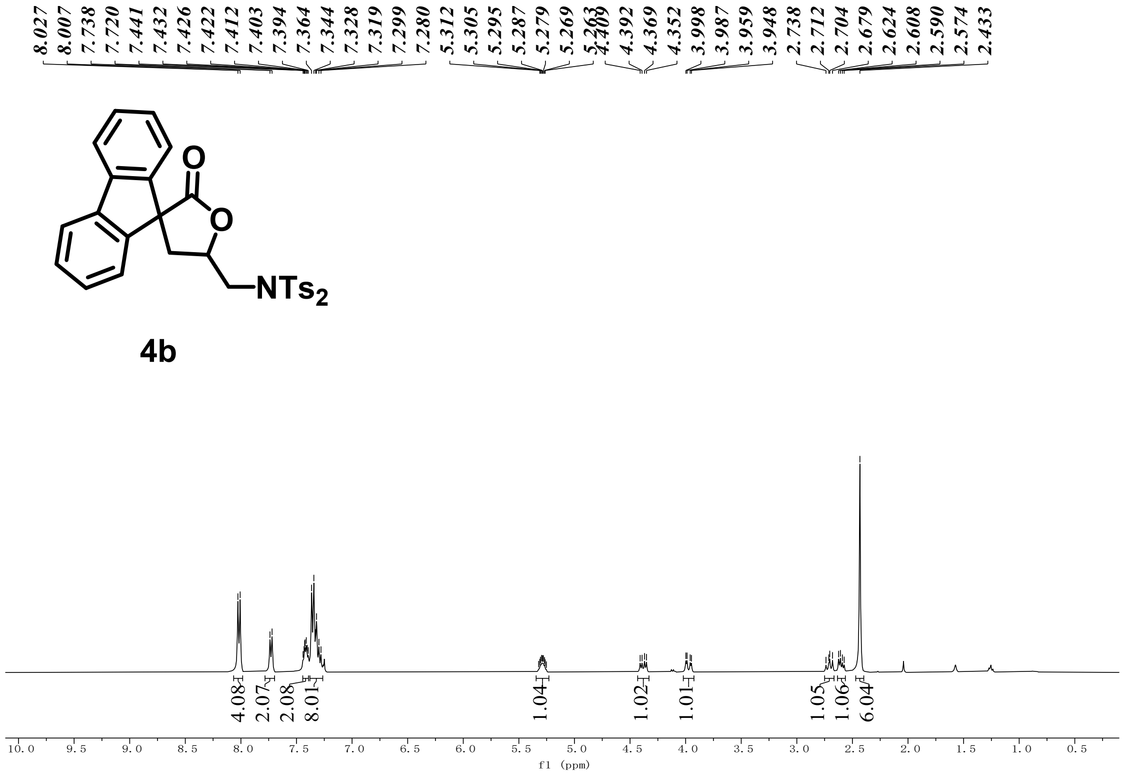
3u


7.831
7.811
7.226
7.205
7.187
7.166
7.162
7.157
7.141
7.136
7.119
4.543
4.529
4.516
4.503
4.489
4.476
4.093
4.077
4.054
4.038
3.810
3.797
3.770
3.758
2.879
2.866
2.846
2.833
2.622
2.597
2.590
2.563
2.338
O
O
Ts2N
4.03
14.04
1.07
1.06
1.05
1.03
1.04
6.02
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
0.
f1 (ppm)

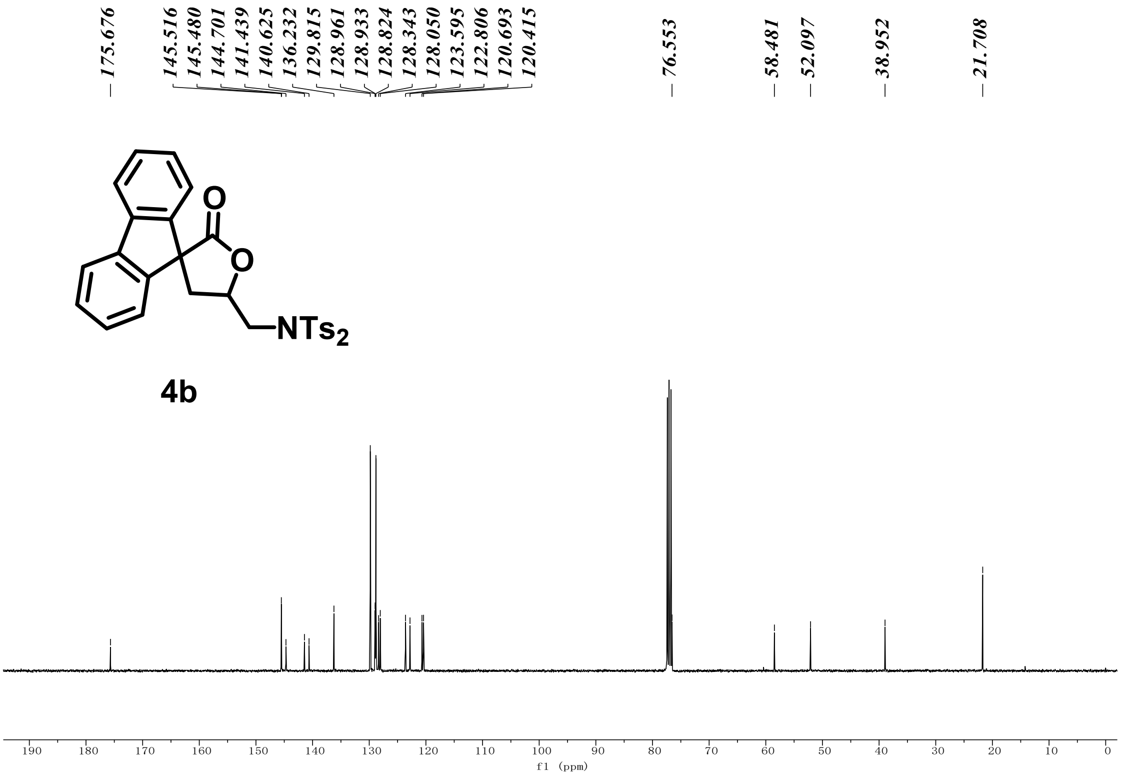
4a


175.958
145.356
141.459
139.462
136.319
129.770
128.961
128.583
128.437
127.809
127.671
127.354
127.233
75.174
57.764
51.185
40.975
21.707
O
O
Ts2N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

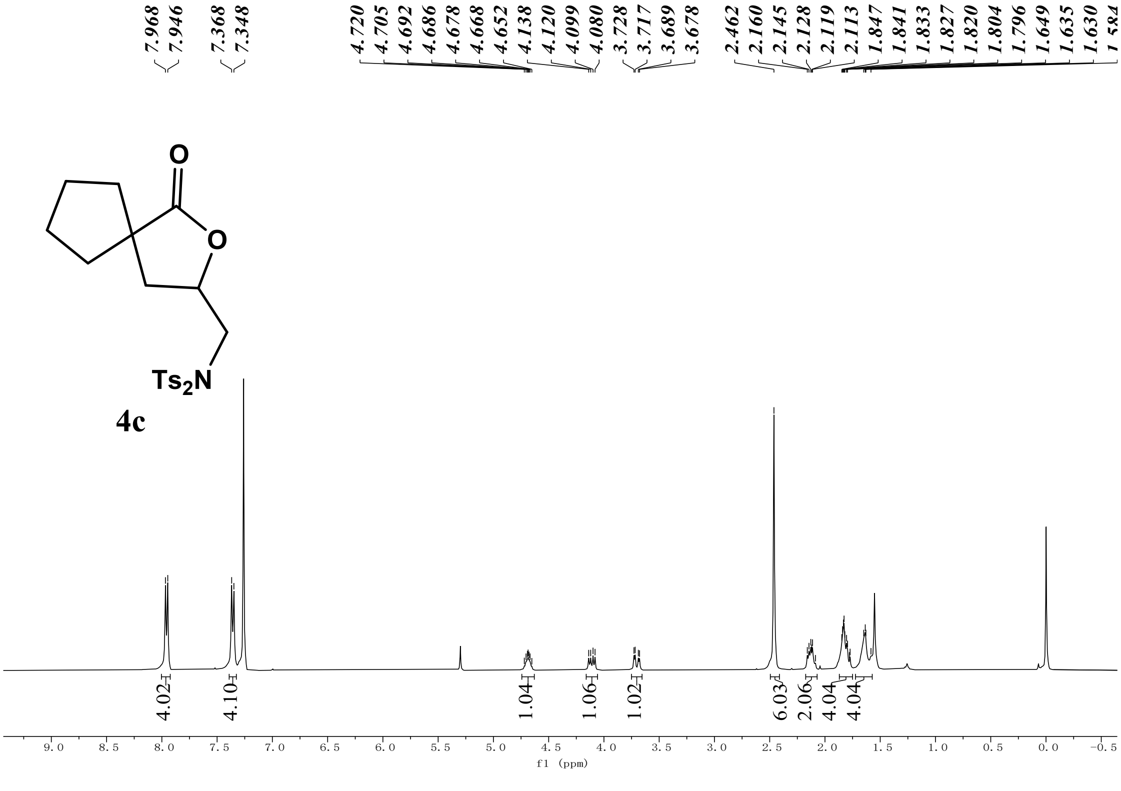
4a


8.027
8.007
7.738
7.720
7.441
7.432
7.426
7.422
7.412
7.403
7.394
7.364
7.344
7.328
7.319
7.299
7.280
5.312
5.305
5.295
5.287
5.279
5.269
5.263
4.409
4.392
4.369
4.352
3.998
3.987
3.959
3.948
2.738
2.712
2.704
2.679
2.624
2.608
2.590
2.574
2.433
O
O
NTs2
4.08
2.07
2.08
8.01
1.04
1.02
1.01
1.05
1.06
6.04
10.0 9.5 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)

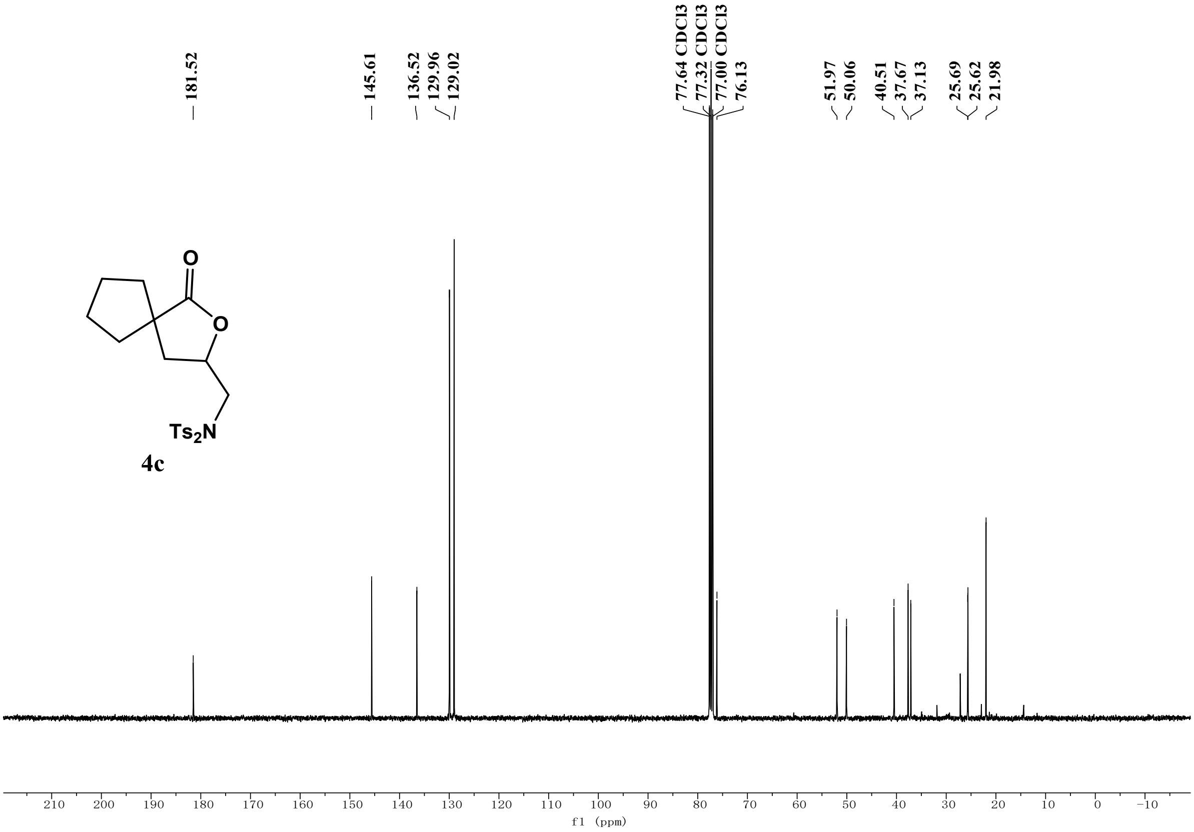
4b


175.676
145.516
145.480
144.701
141.439
140.625
136.232
129.815
128.961
128.933
128.824
128.343
128.050
123.595
122.806
120.693
120.415
76.553
58.481
52.097
38.952
21.708
O
O
NTs$_2$
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

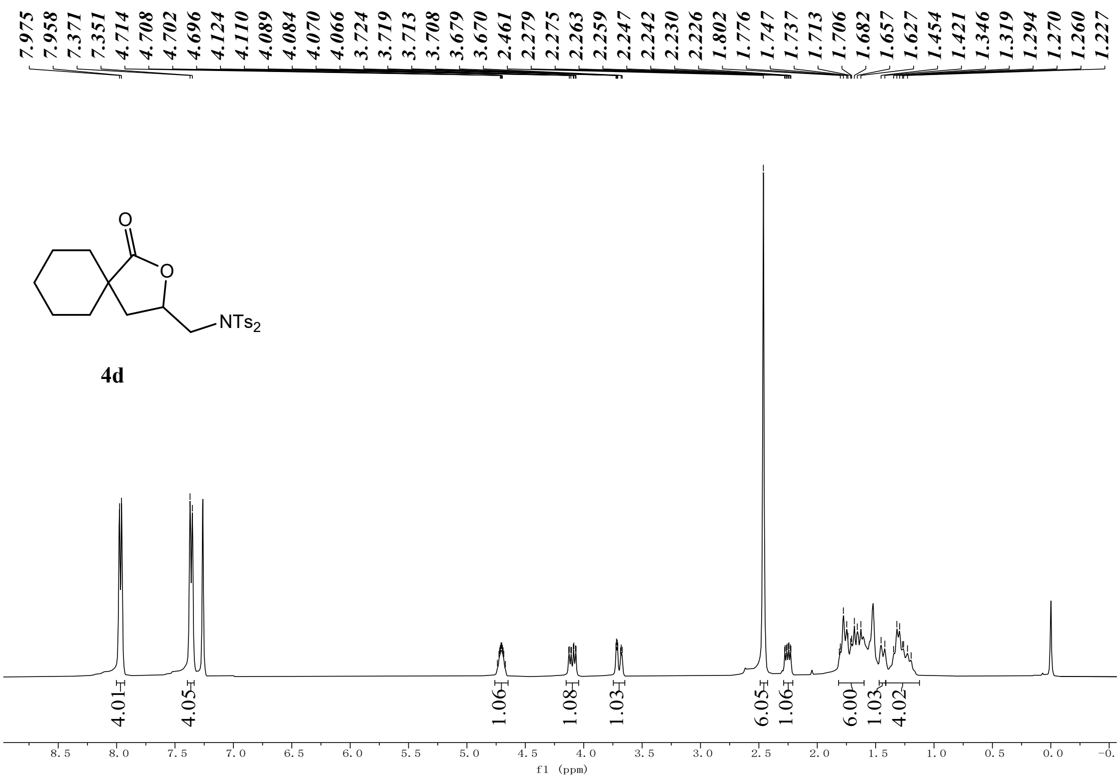
4b


7.968
7.946
7.368
7.348
4.720
4.705
4.692
4.686
4.678
4.668
4.652
4.138
4.120
4.099
4.080
3.728
3.717
3.689
3.678
2.462
2.160
2.145
2.128
2.119
2.113
1.847
1.841
1.833
1.827
1.820
1.804
1.796
1.649
1.635
1.630
1.58
O
O
Ts$_2$N
4.02
4.10
1.04
1.06
1.02
6.03
2.06
4.04
4.04
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
0.0
-0.5
f1 (ppm)

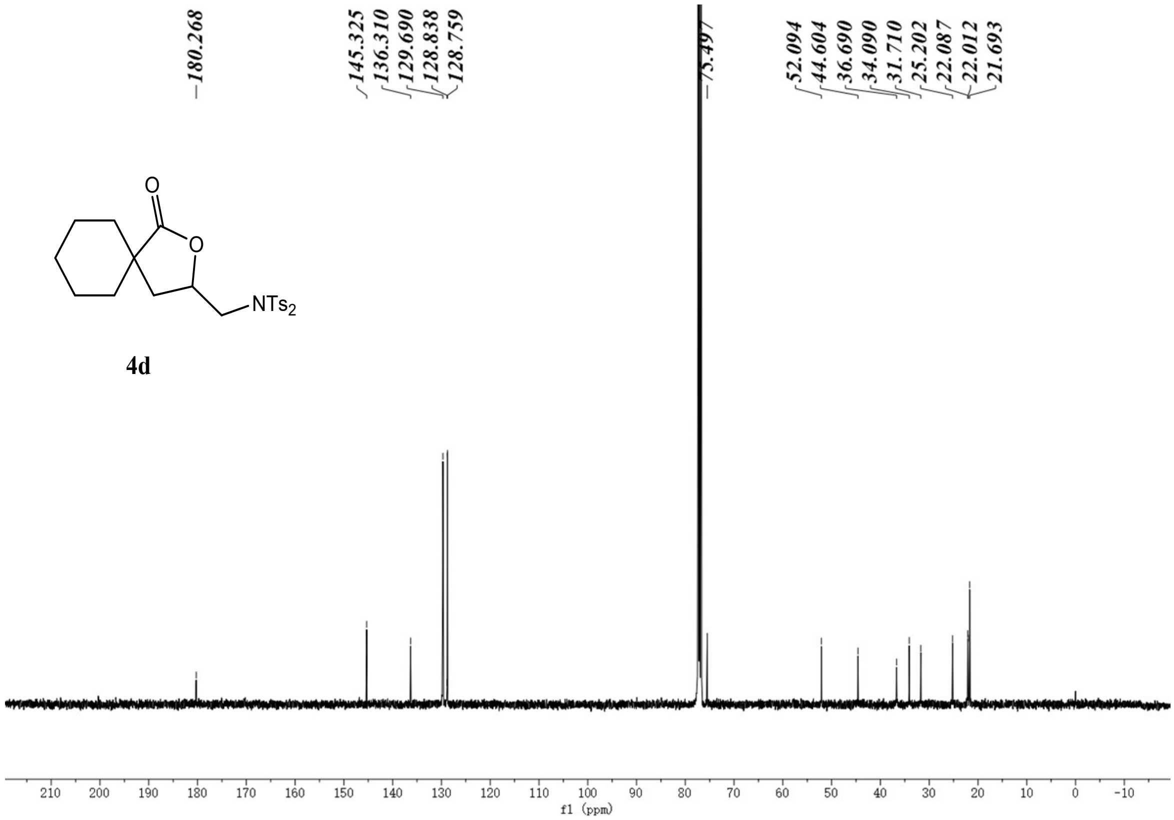
4c


181.52
145.61
136.52
129.96
129.02
77.64 CDCl3
77.32 CDCl3
77.00 CDCl3
76.13
51.97
50.06
40.51
37.67
37.13
25.69
25.62
21.98
O
O
Ts$_2$N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

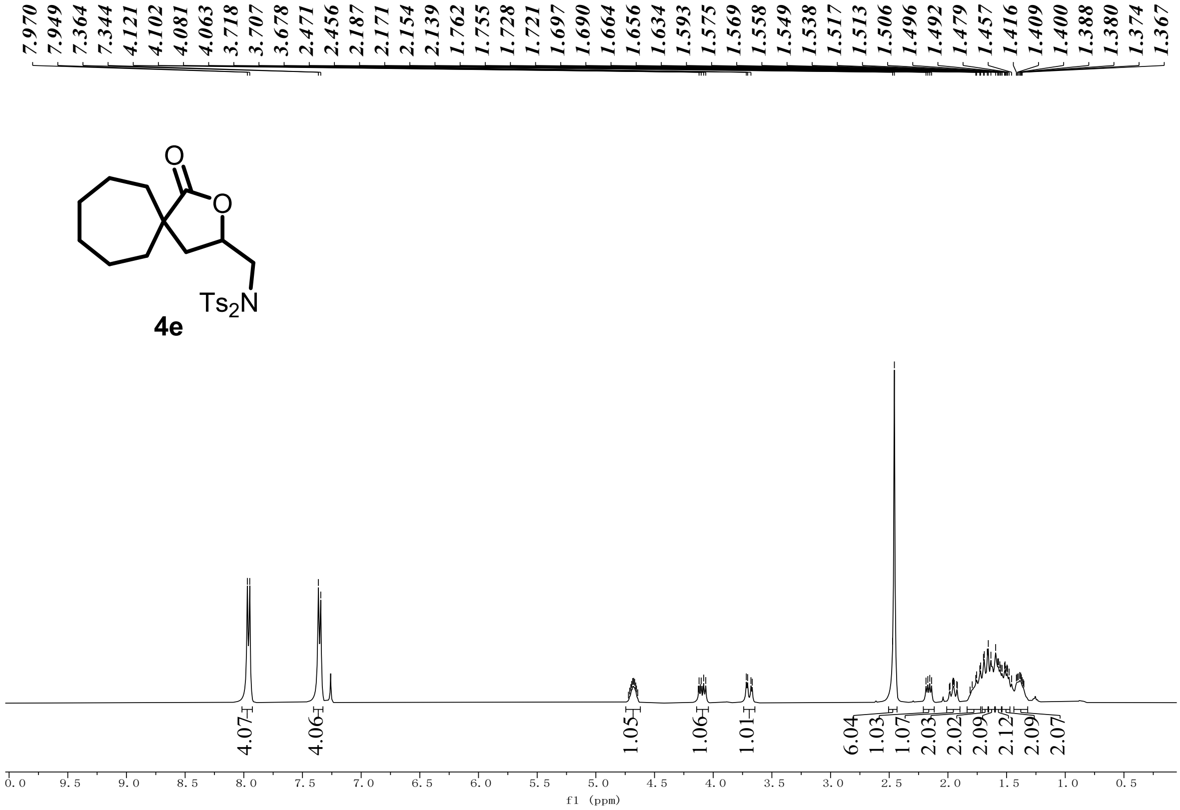
4c


7.975
7.958
7.371
7.351
4.714
4.708
4.702
4.696
4.124
4.110
4.089
4.084
4.070
4.066
3.724
3.719
3.713
3.708
3.679
3.670
2.461
2.279
2.275
2.263
2.259
2.247
2.242
2.230
2.226
1.802
1.776
1.747
1.737
1.713
1.706
1.682
1.657
1.627
1.454
1.421
1.346
1.319
1.294
1.270
1.260
1.227
O
O
NTs$_2$
4.01
4.05
1.06
1.08
1.03
6.05
1.06
6.00
1.03
4.02
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 0.0 −0.
f1 (ppm)

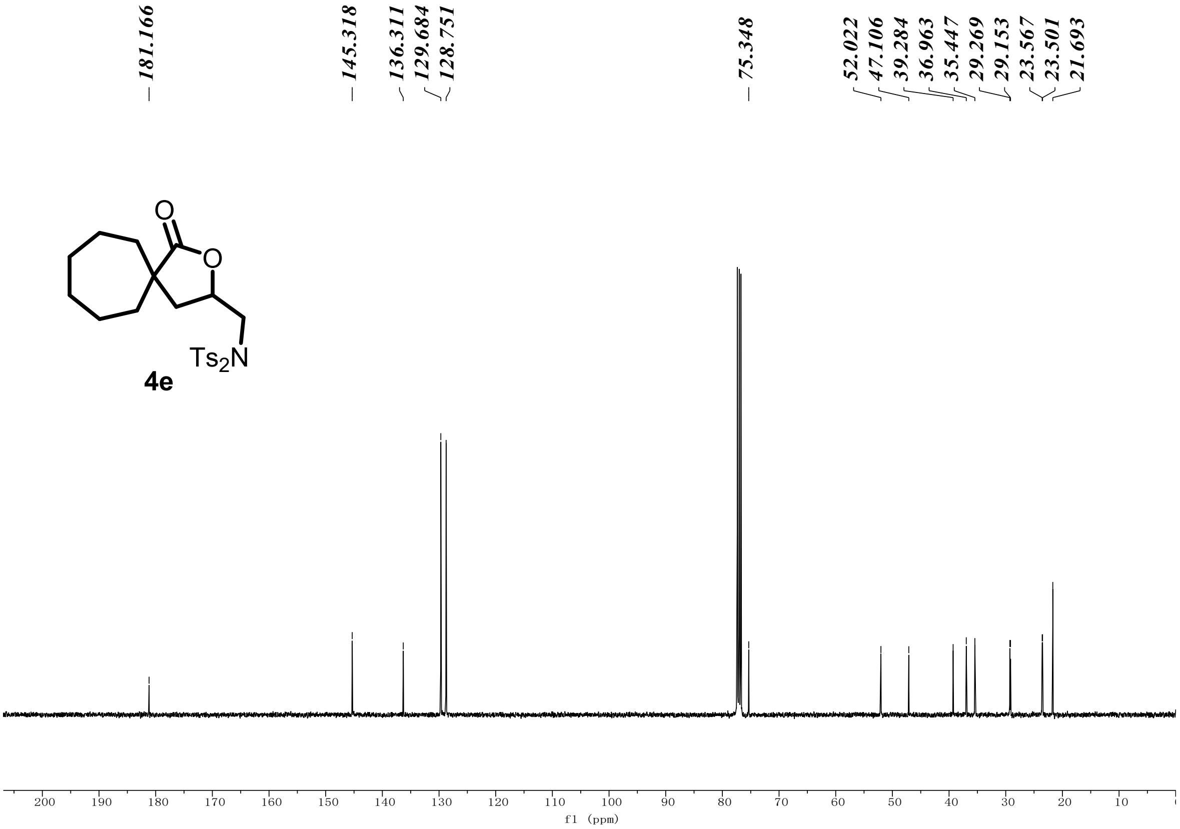
4d


180.268
145.325
136.310
129.690
128.838
128.759
75.497
52.094
44.604
36.690
34.090
31.710
25.202
22.087
22.012
21.693
O
O
NTs$_2$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

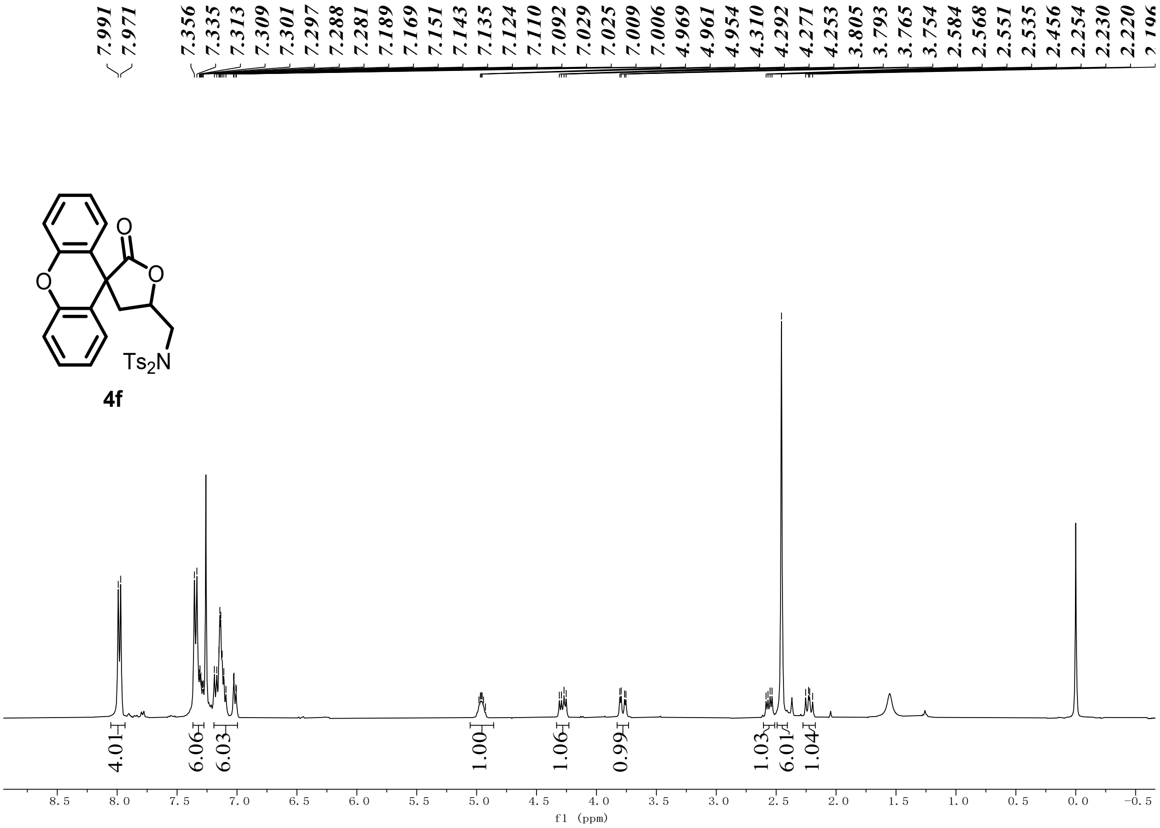
4d


7.970
7.949
7.364
7.344
4.121
4.102
4.081
4.063
3.718
3.707
3.678
2.471
2.456
2.187
2.171
2.154
2.139
1.762
1.755
1.728
1.721
1.697
1.690
1.664
1.656
1.634
1.593
1.575
1.569
1.558
1.549
1.538
1.517
1.513
1.506
1.496
1.492
1.479
1.457
1.416
1.409
1.400
1.388
1.380
1.374
1.367
O
O
Ts$_2$N
4.07
4.06
1.05
1.06
1.01
6.04
1.03
1.07
2.03
2.02
2.09
2.12
2.09
2.07
0.0
9.5
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)

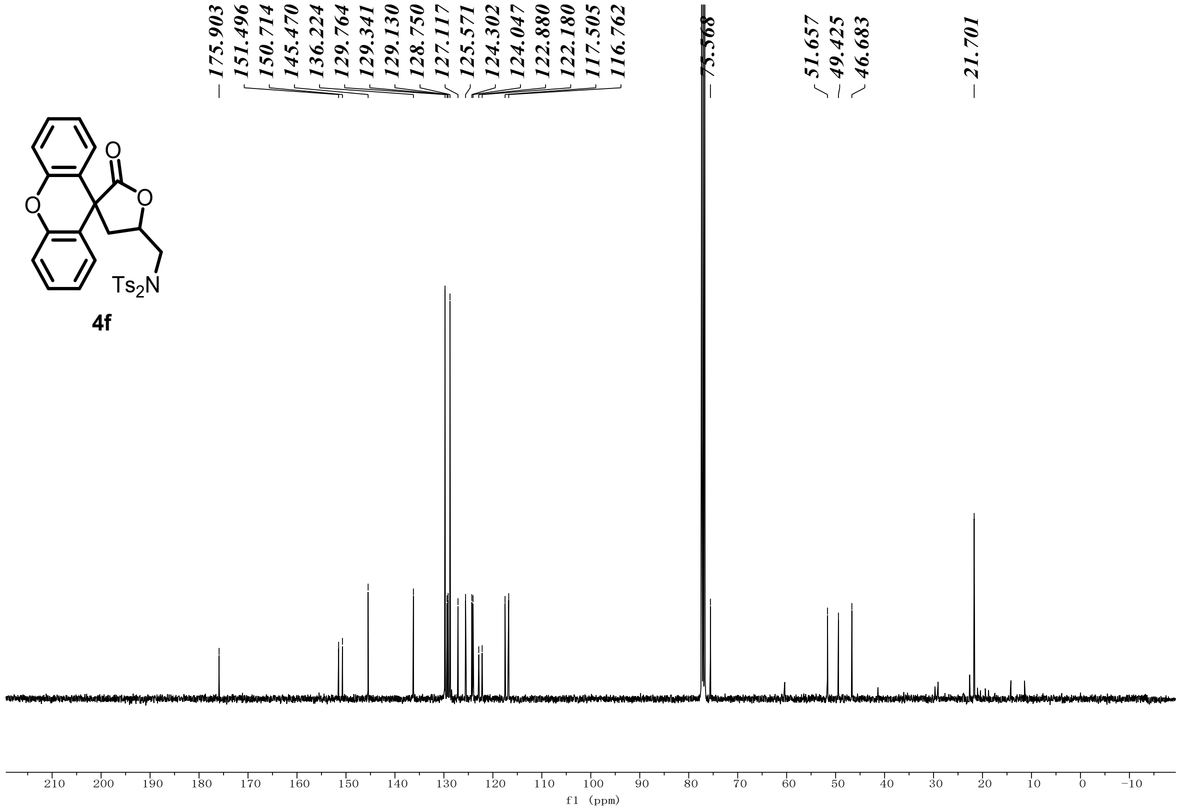
4e

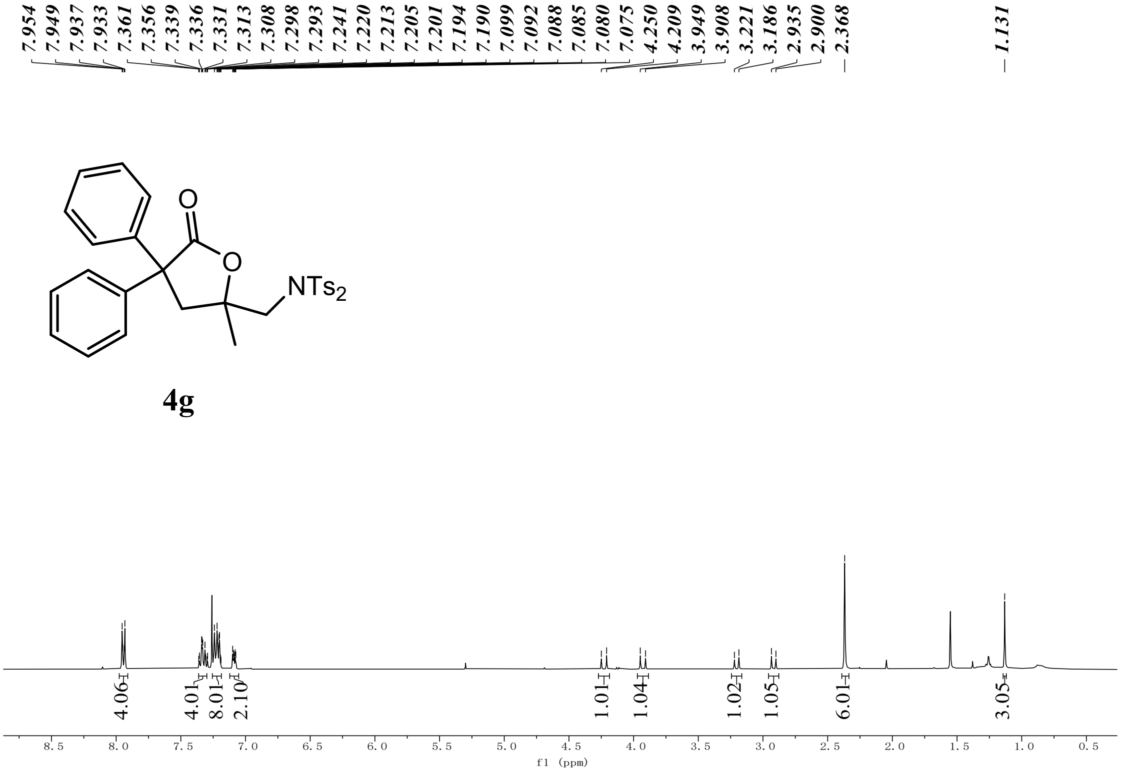

181.166
145.318
136.311
129.684
128.751
75.348
52.022
47.106
39.284
36.963
35.447
29.269
29.153
23.567
23.501
21.693
O
O
Ts2N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

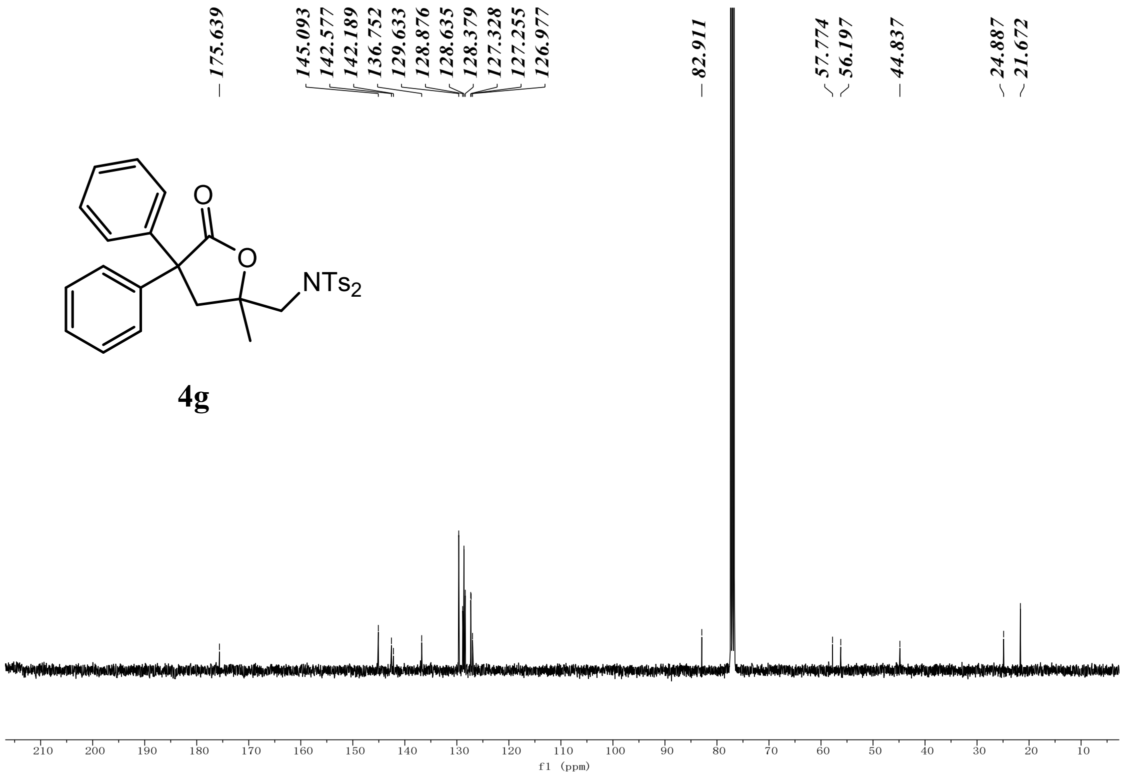
4e


7.991
7.971
7.356
7.335
7.313
7.309
7.301
7.297
7.288
7.281
7.189
7.169
7.151
7.143
7.135
7.124
7.110
7.092
7.029
7.025
7.009
7.006
4.969
4.961
4.954
4.310
4.292
4.271
4.253
3.805
3.793
3.765
3.754
2.584
2.568
2.551
2.535
2.456
2.254
2.230
2.220
O
O
O
Ts2N
4.01
6.06
6.03
1.00
1.06
0.99
1.03
6.01
1.04
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
0.0
-0.5
f1 (ppm)

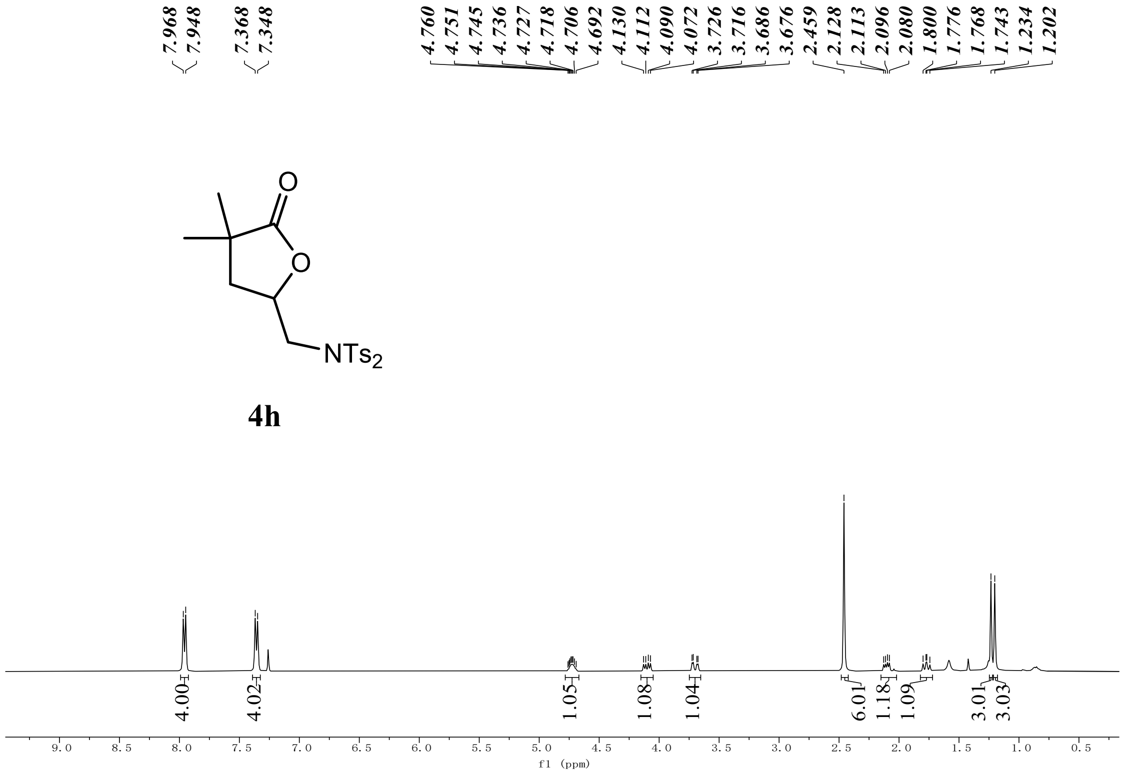
4f


175.903
151.496
150.714
145.470
136.224
129.764
129.341
129.130
128.750
127.117
125.571
124.302
124.047
122.880
122.180
117.505
116.762
75.568
51.657
49.425
46.683
21.701
O
O
O
Ts$_2$N
4f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

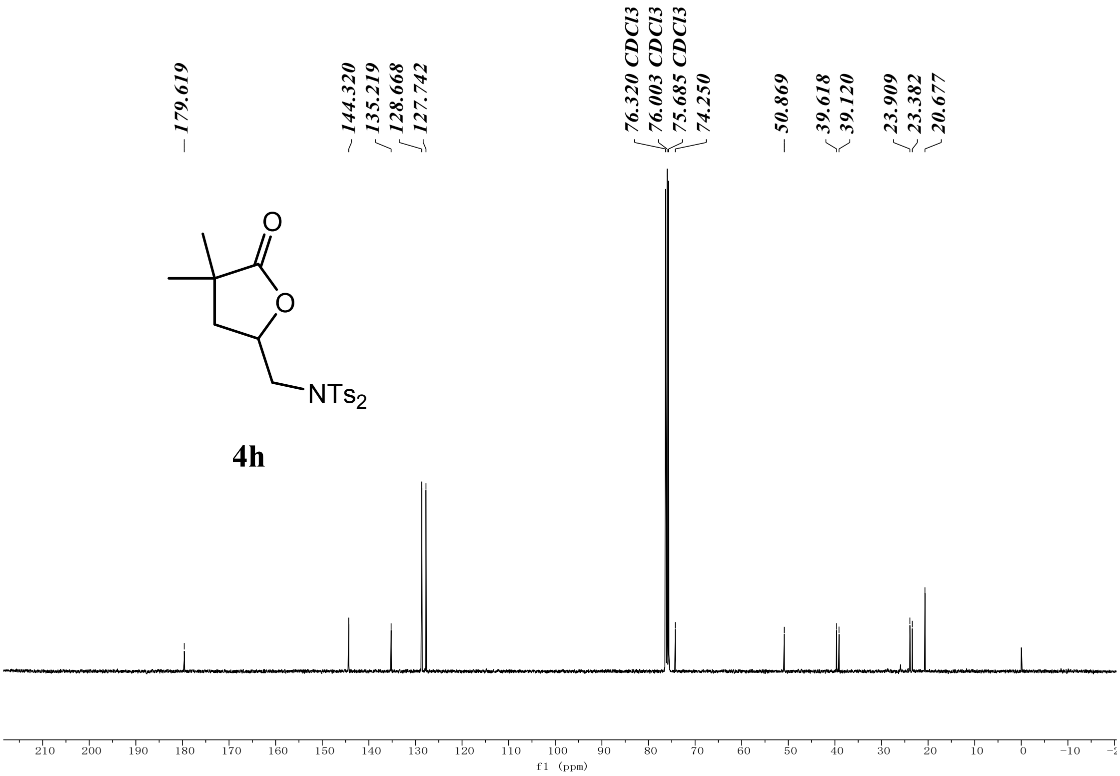

7.954
7.949
7.937
7.933
7.361
7.356
7.339
7.336
7.331
7.313
7.308
7.298
7.293
7.241
7.220
7.213
7.205
7.201
7.194
7.190
7.099
7.092
7.088
7.085
7.080
7.075
4.250
4.209
3.949
3.908
3.221
3.186
2.935
2.900
2.368
1.131
O
O
NTs$_2$
4g
4.06
4.01
8.01
2.10
1.01
1.04
1.02
1.05
6.01
3.05
8.5 8.0 7.5 7.0 6.5 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)


175.639
145.093
142.577
142.189
136.752
129.633
128.876
128.635
128.379
127.328
127.255
126.977
82.911
57.774
56.197
44.837
24.887
21.672
O
O
NTs2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

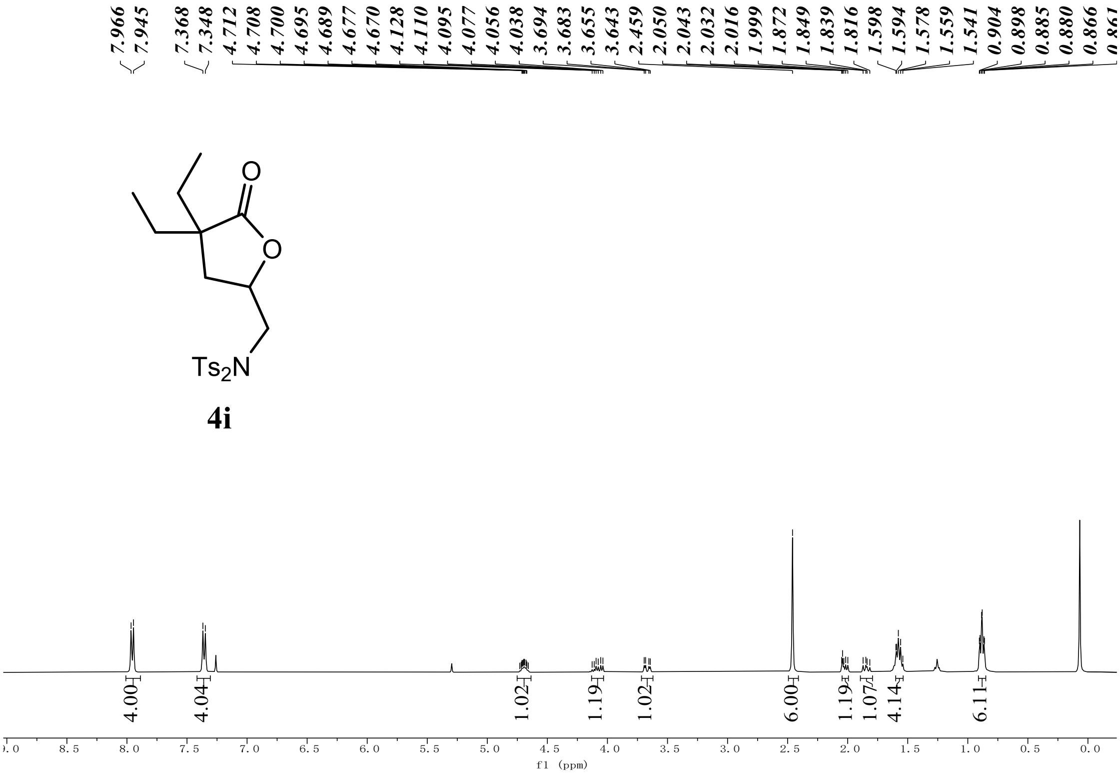
4g


7.968
7.948
7.368
7.348
4.760
4.751
4.745
4.736
4.727
4.718
4.706
4.692
4.130
4.112
4.090
4.072
3.726
3.716
3.686
3.676
2.459
2.128
2.113
2.096
2.080
1.800
1.776
1.768
1.743
1.234
1.202
O
O
NTs2
4.00
4.02
1.05
1.08
1.04
6.01
1.18
1.09
3.01
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)

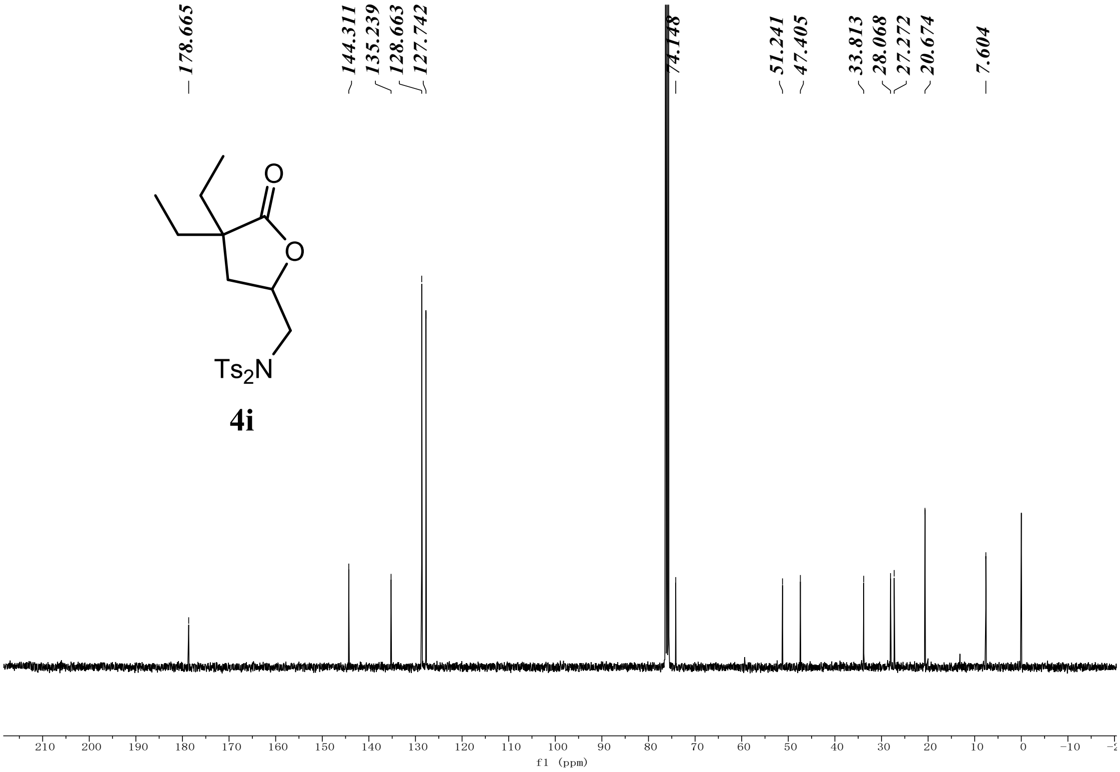
4h


179.619
144.320
135.219
128.668
127.742
76.320 CDCl3
76.003 CDCl3
75.685 CDCl3
74.250
50.869
39.618
39.120
23.909
23.382
20.677
O
O
NTs2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

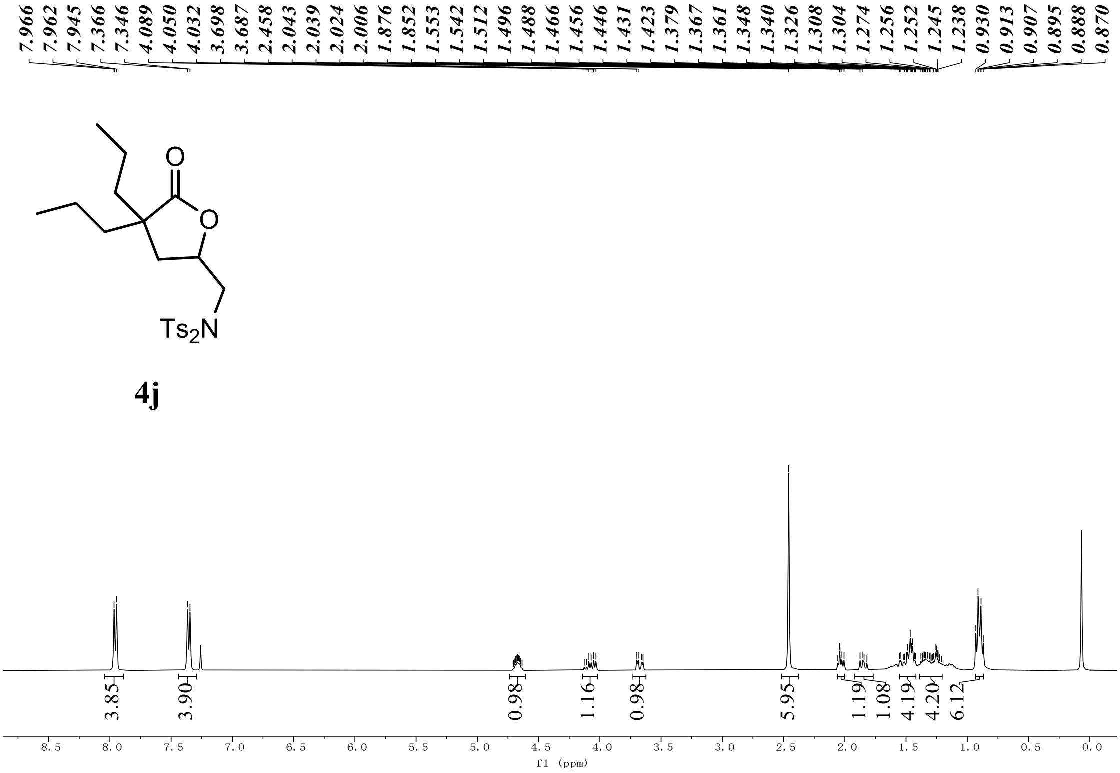
4h


7.966
7.945
7.368
7.348
4.712
4.708
4.700
4.695
4.689
4.677
4.670
4.128
4.110
4.095
4.077
4.056
4.038
3.694
3.683
3.655
3.643
2.459
2.050
2.043
2.032
2.016
1.999
1.872
1.849
1.839
1.816
1.598
1.594
1.578
1.559
1.541
0.904
0.898
0.885
0.880
0.866
0.861
O
O
Ts2N
4.00
4.04
1.02
1.19
1.02
6.00
1.19
1.07
4.14
6.11
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
0.0
f1 (ppm)

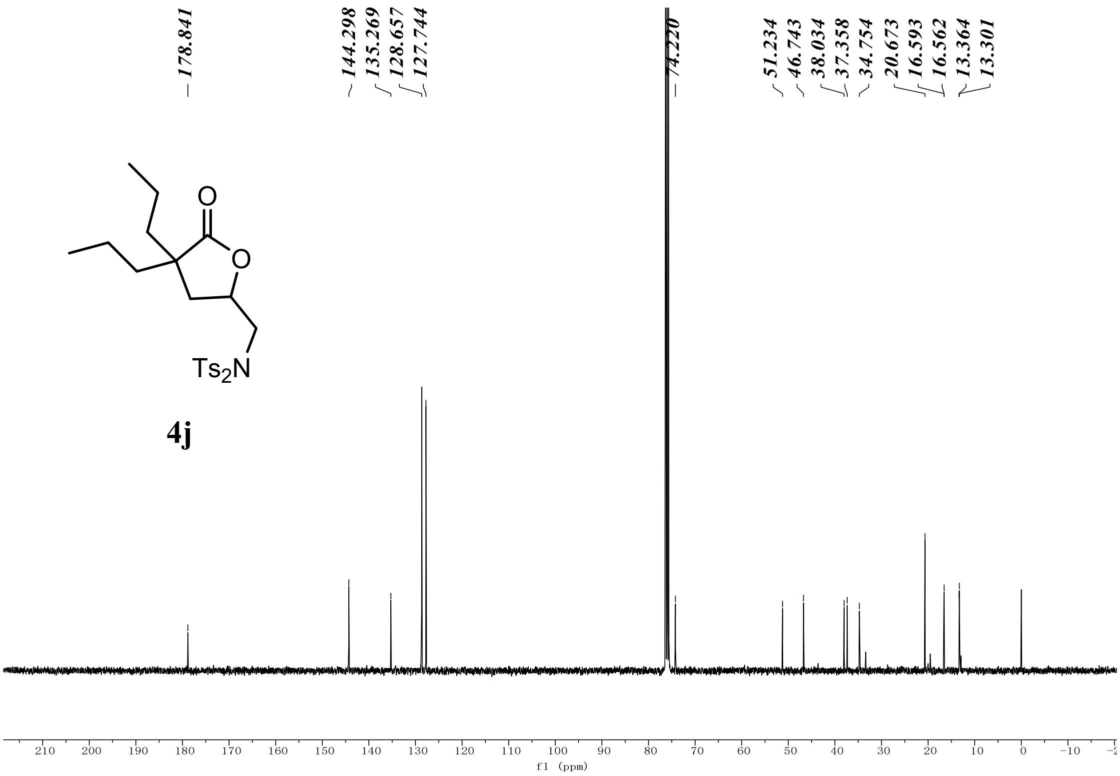
4i


178.665
144.311
135.239
128.663
127.742
74.148
51.241
47.405
33.813
28.068
27.272
20.674
7.604
O
O
Ts$_2$N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

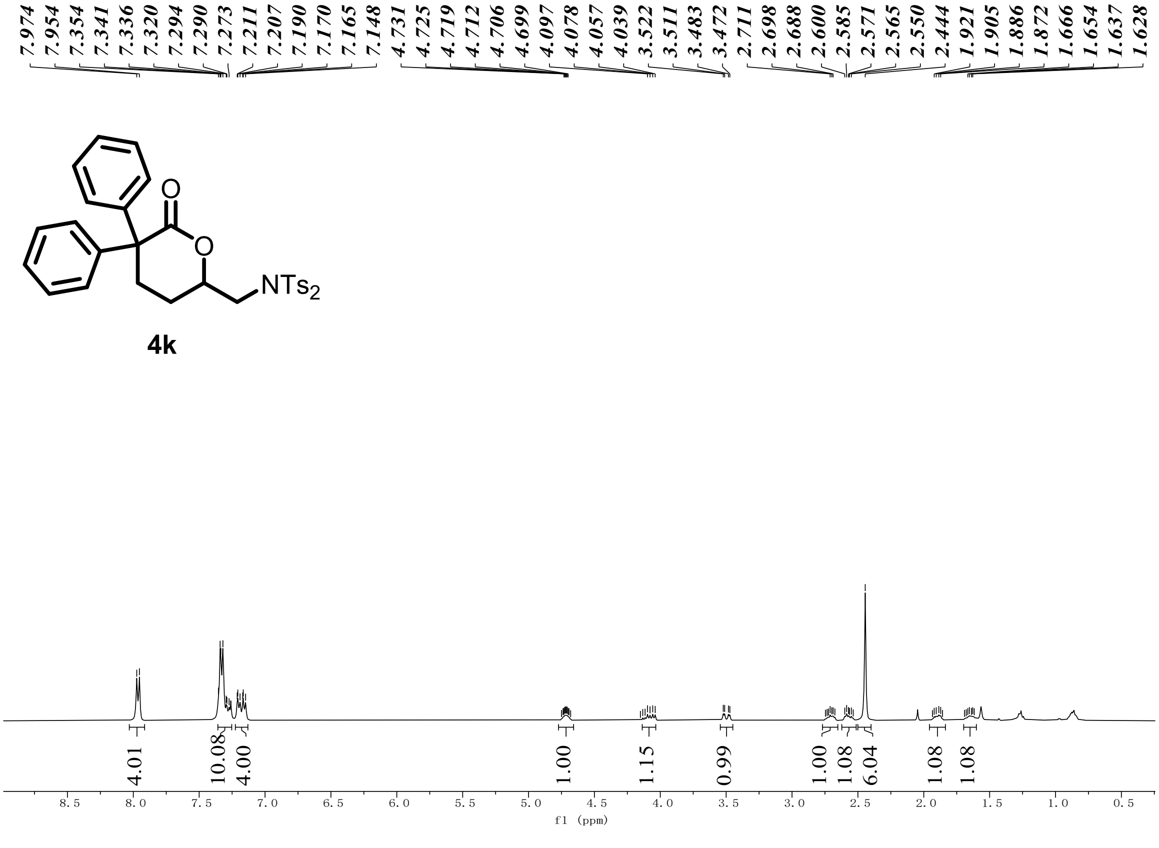
4i


7.966
7.962
7.945
7.366
7.346
4.089
4.050
4.032
3.698
3.687
2.458
2.043
2.039
2.024
2.006
1.876
1.852
1.553
1.542
1.512
1.496
1.488
1.466
1.456
1.446
1.431
1.423
1.379
1.367
1.361
1.348
1.340
1.326
1.308
1.304
1.274
1.256
1.252
1.245
1.238
0.930
0.913
0.907
0.895
0.888
0.870
O
O
Ts$_2$N
3.85
3.90
0.98
1.16
0.98
5.95
1.19
1.08
4.19
4.20
6.12
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
0.0
f1 (ppm)

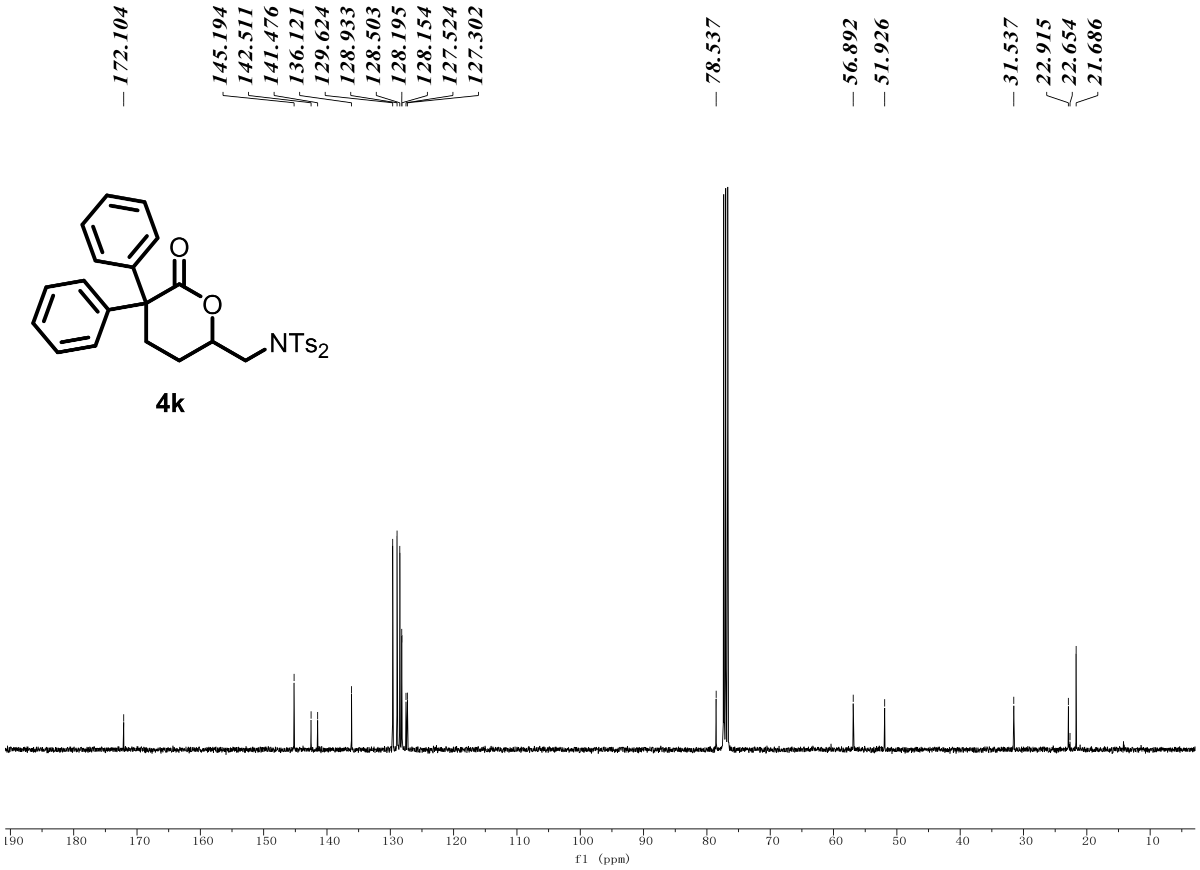
4j


178.841
144.298
135.269
128.657
127.744
74.220
51.234
46.743
38.034
37.358
34.754
20.673
16.593
16.562
13.364
13.301
O
O
Ts2N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

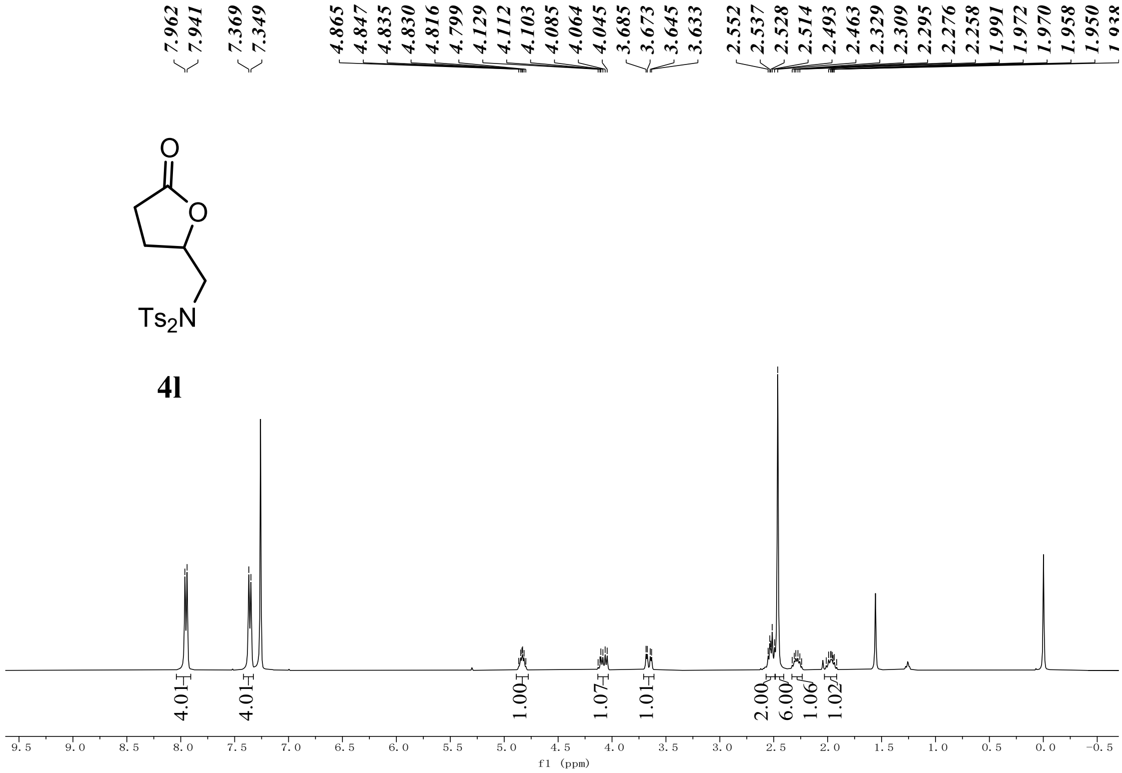
4j


7.974
7.954
7.354
7.341
7.336
7.320
7.294
7.290
7.273
7.211
7.207
7.190
7.170
7.165
7.148
4.731
4.725
4.719
4.712
4.706
4.699
4.097
4.078
4.057
4.039
3.522
3.511
3.483
3.472
2.711
2.698
2.688
2.600
2.585
2.571
2.565
2.550
2.444
1.921
1.905
1.886
1.872
1.666
1.654
1.637
1.628
O
O
NTs2
4.01
10.08
4.00
1.00
1.15
0.99
1.00
1.08
6.04
1.08
1.08
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)

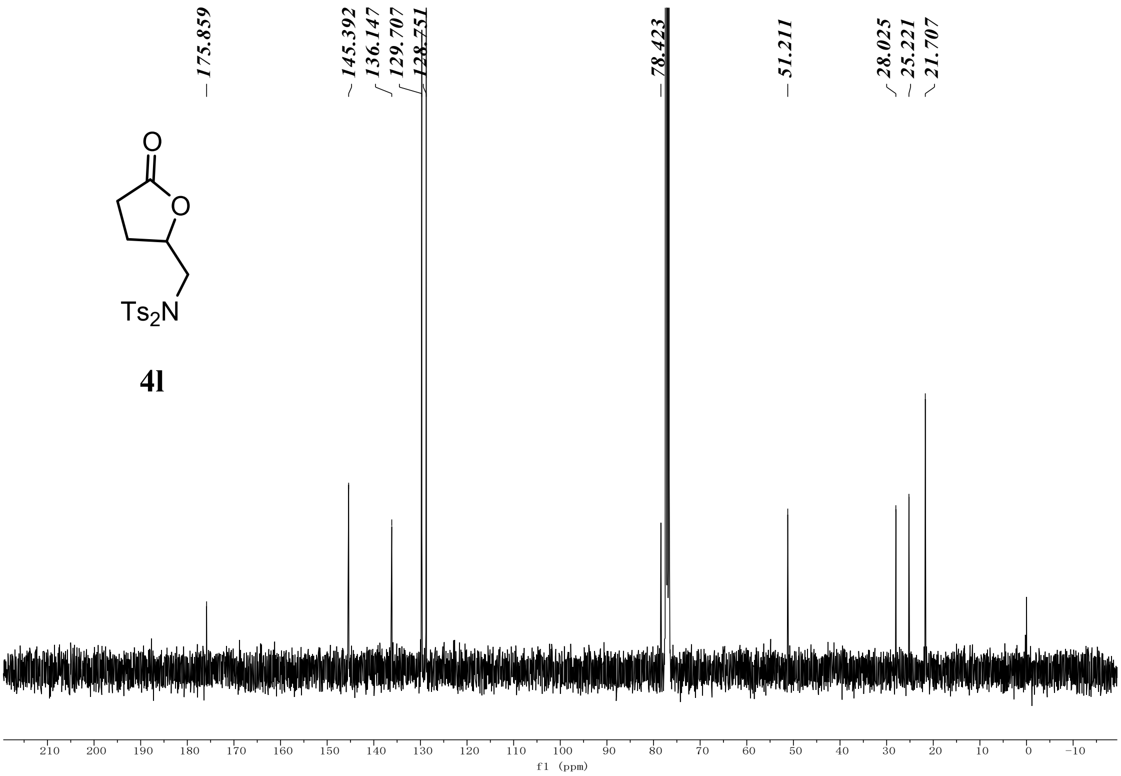
4k

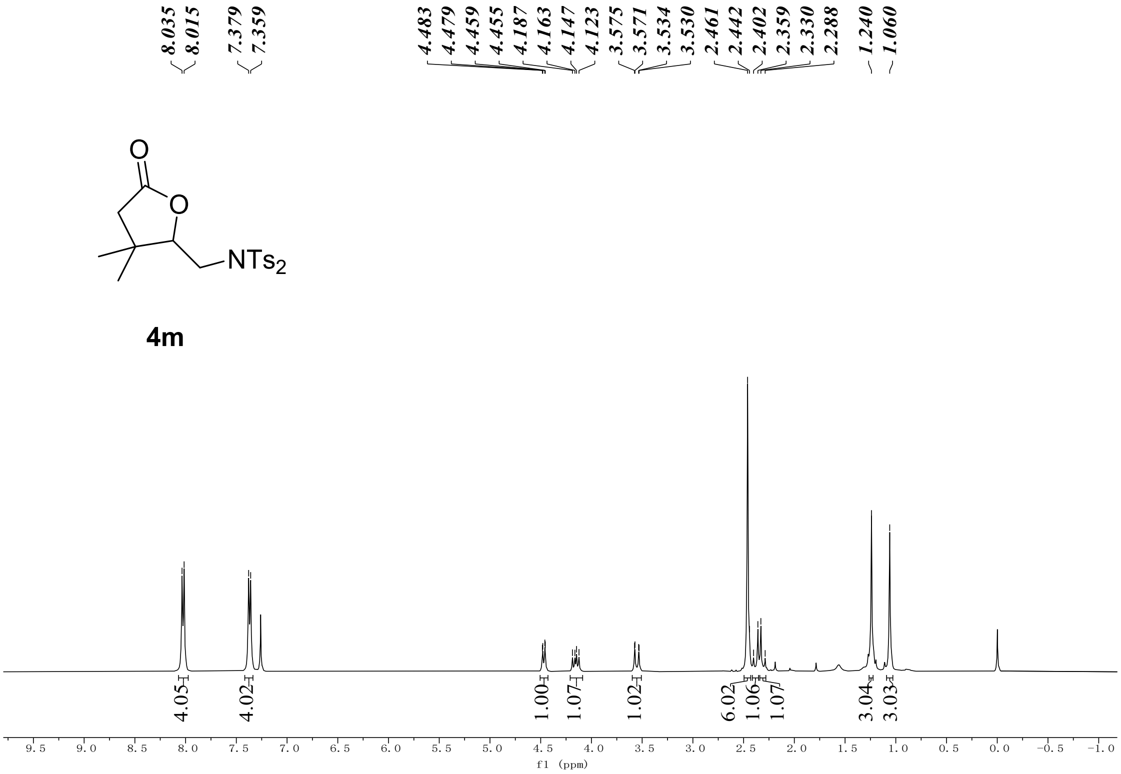

172.104
145.194
142.511
141.476
136.121
129.624
128.933
128.503
128.195
128.154
127.524
127.302
78.537
56.892
51.926
31.537
22.915
22.654
21.686
O
O
NTs2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

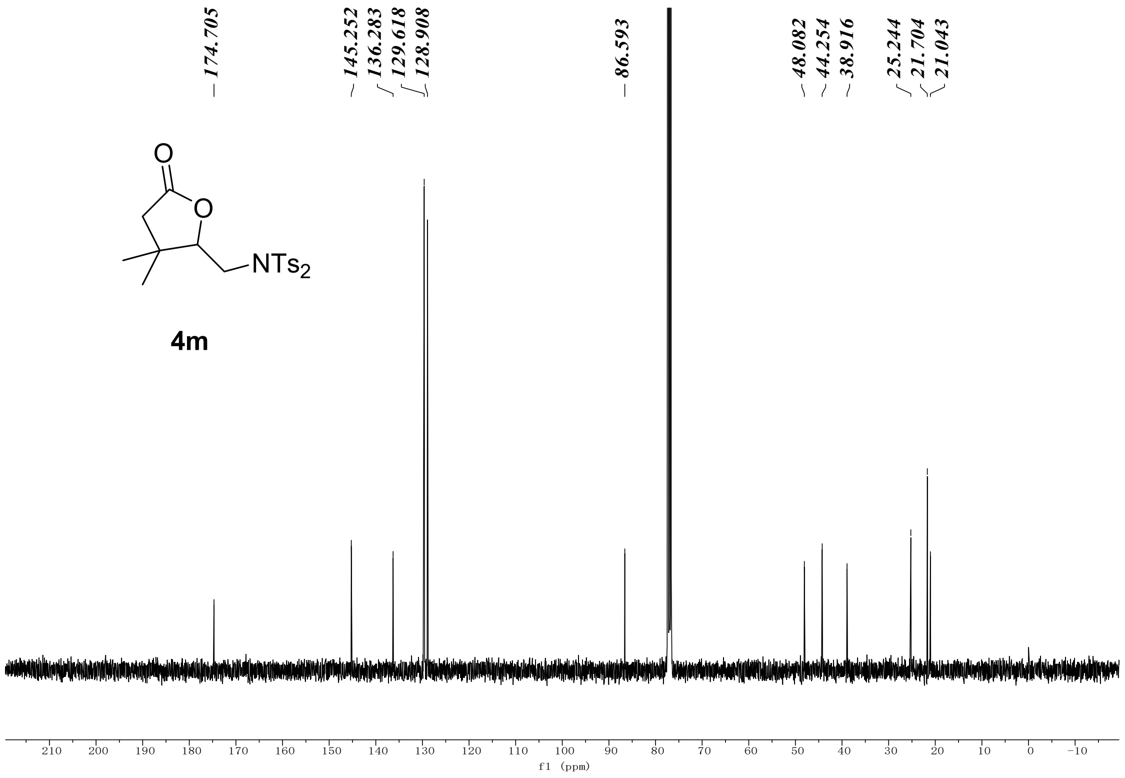
4k

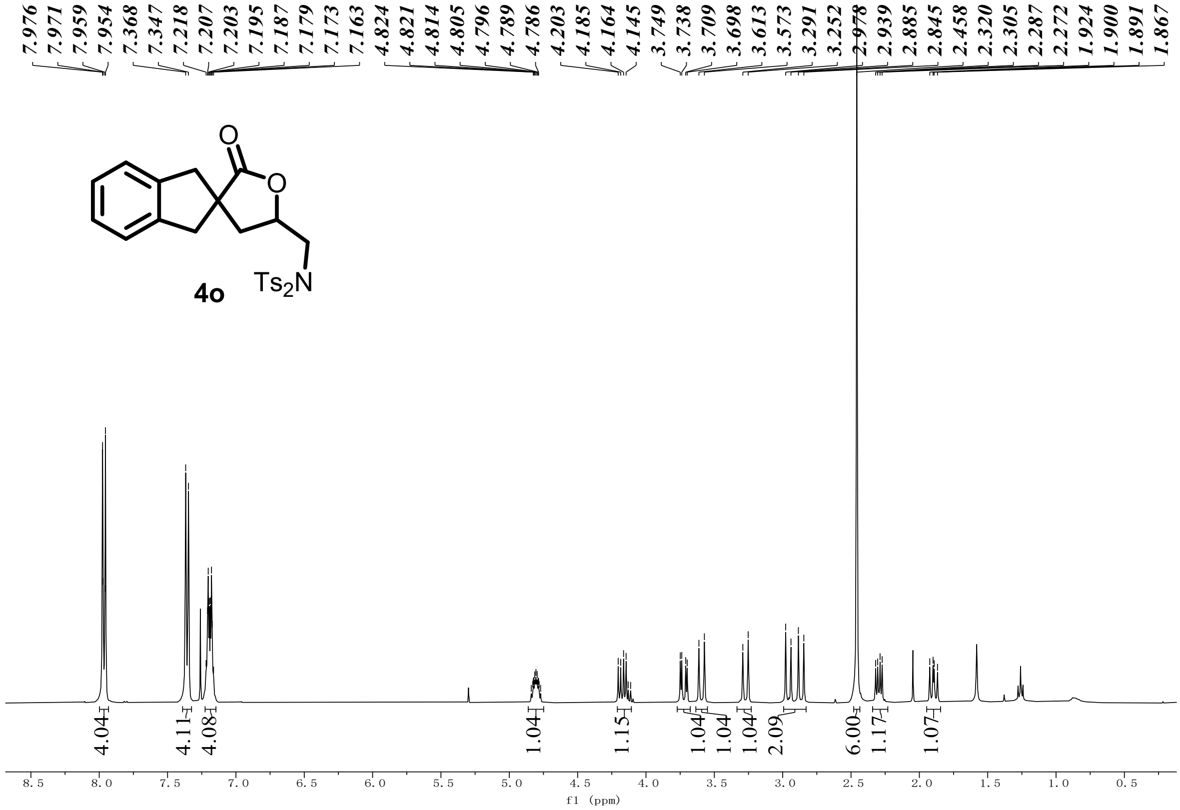

7.962
7.941
7.369
7.349
4.865
4.847
4.835
4.830
4.816
4.799
4.129
4.112
4.103
4.085
4.064
4.045
3.685
3.673
3.645
3.633
2.552
2.537
2.528
2.514
2.493
2.463
2.329
2.309
2.295
2.276
2.258
1.991
1.972
1.970
1.958
1.950
1.938
O
O
Ts2N
4.01
4.01
1.00
1.07
1.01
2.00
6.00
1.06
1.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

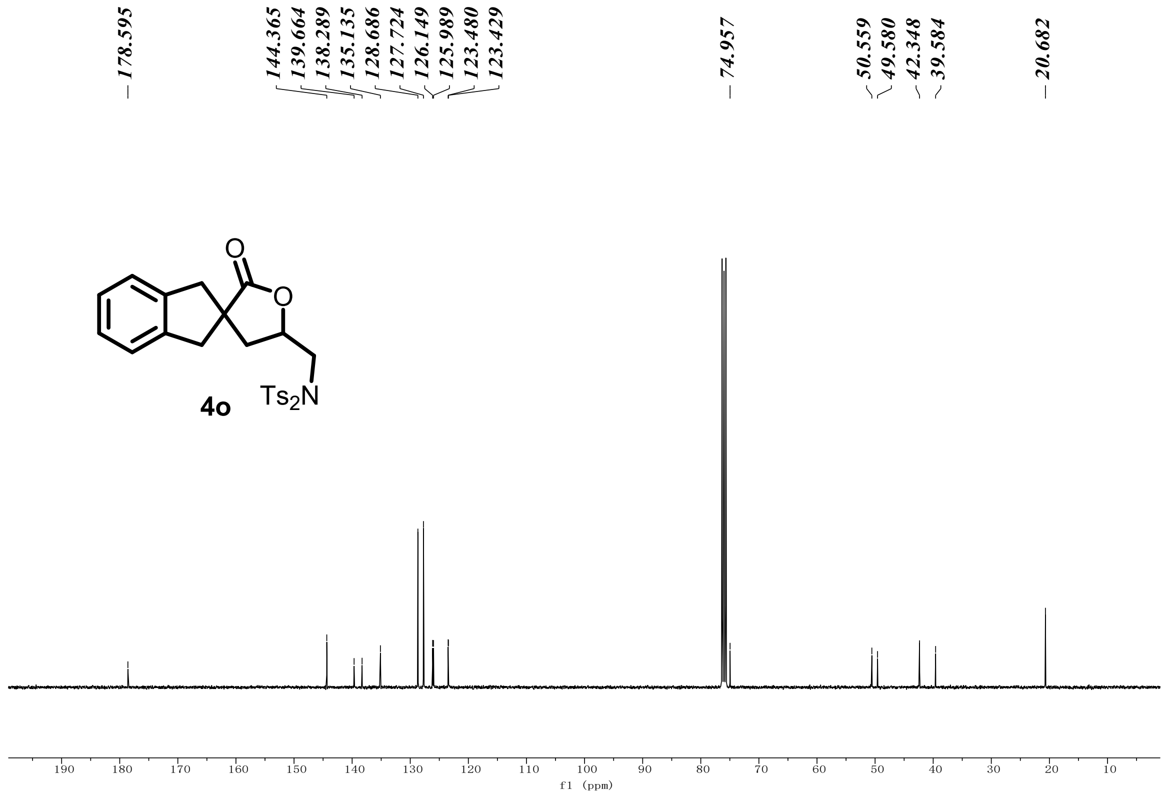
4l

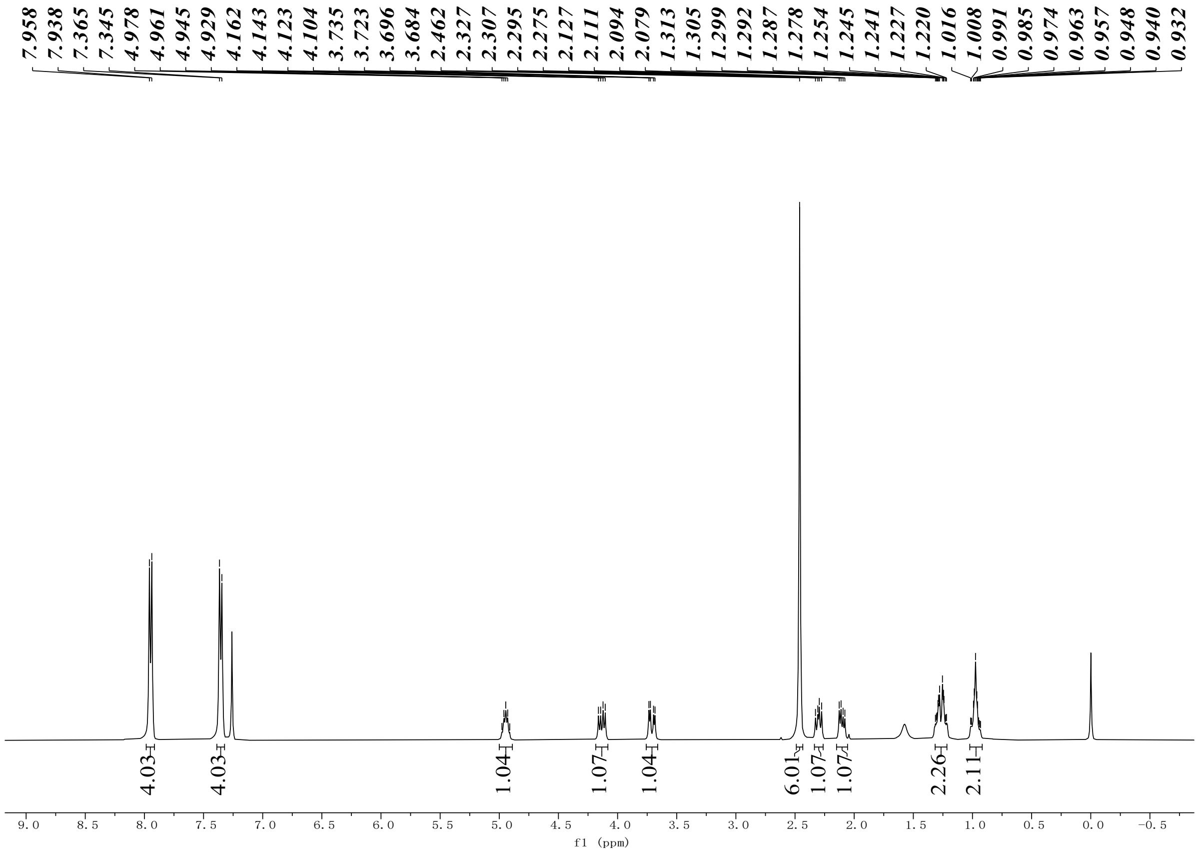

175.859
145.392
136.147
129.707
128.751
78.423
51.211
28.025
25.221
21.707
O
O
Ts2N
4l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

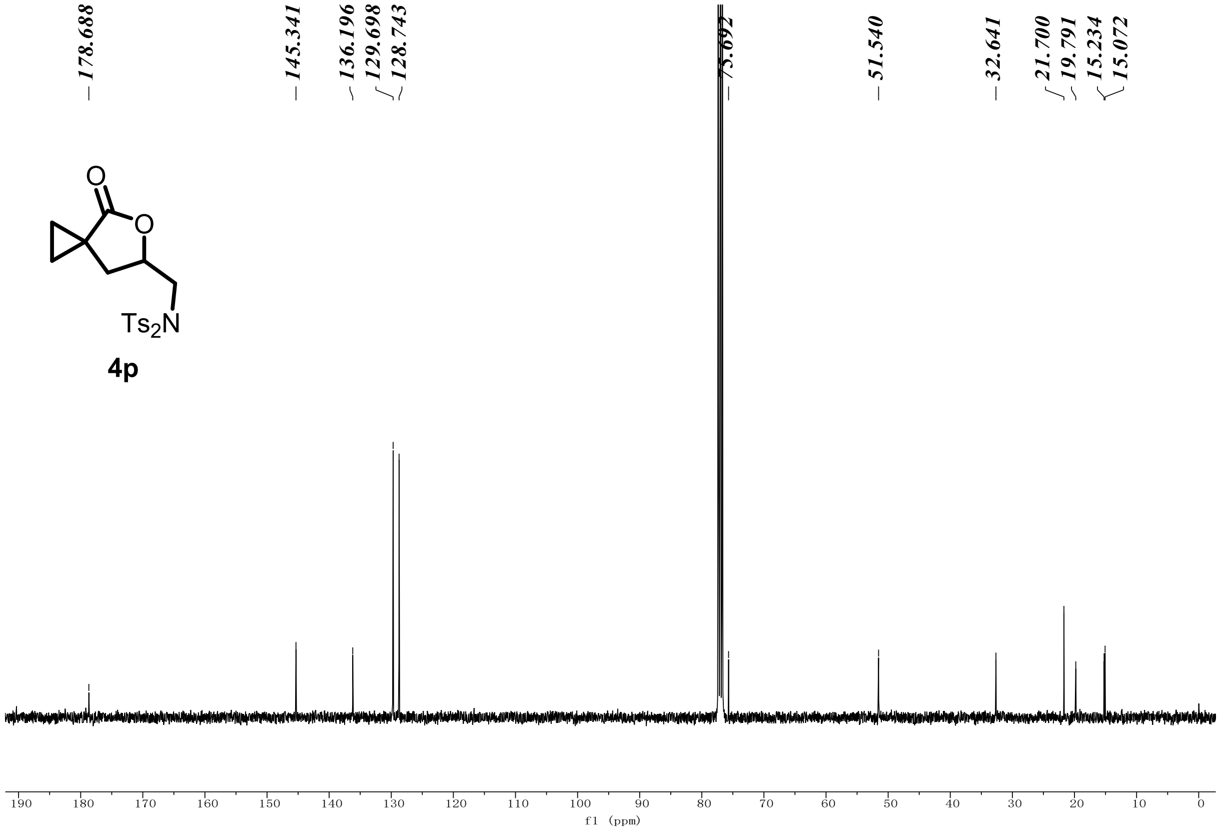

8.035
8.015
7.379
7.359
4.483
4.479
4.459
4.455
4.187
4.163
4.147
4.123
3.575
3.571
3.534
3.530
2.461
2.442
2.402
2.359
2.330
2.288
1.240
1.060
O
O
NTs2
4m
4.05
4.02
1.00
1.07
1.02
6.02
1.06
1.07
3.04
3.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

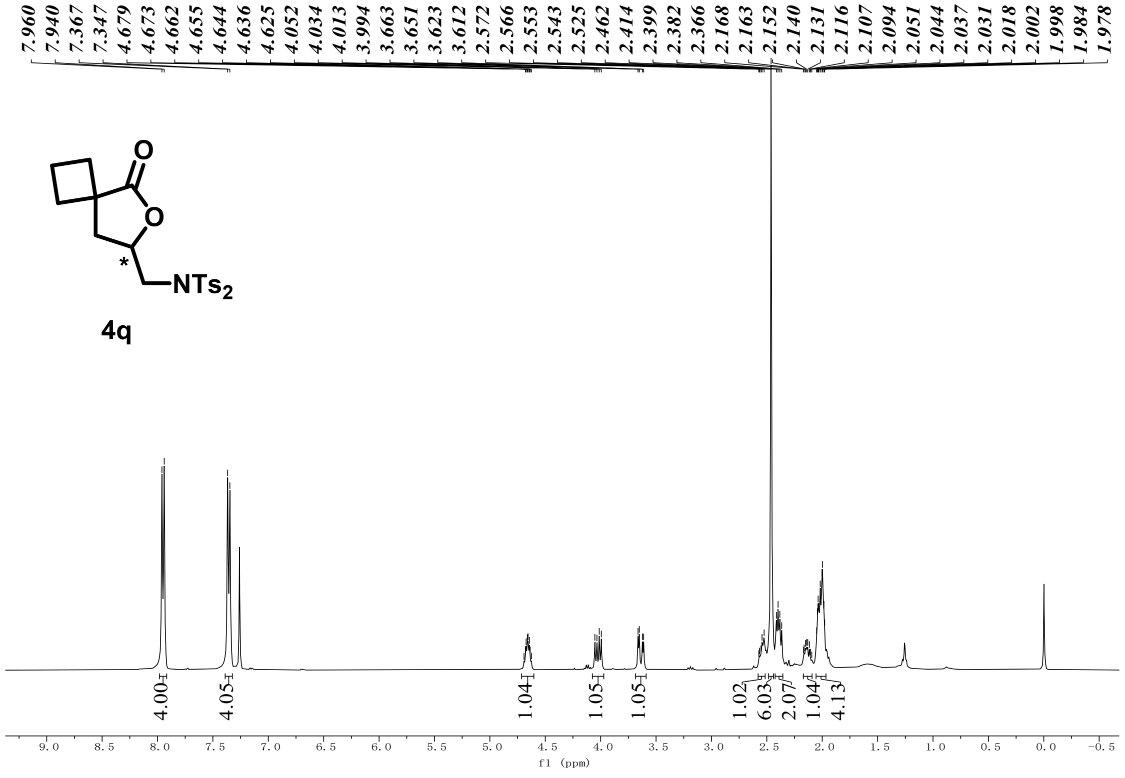

174.705
145.252
136.283
129.618
128.908
86.593
48.082
44.254
38.916
25.244
21.704
21.043
O
O
NTs2
4m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

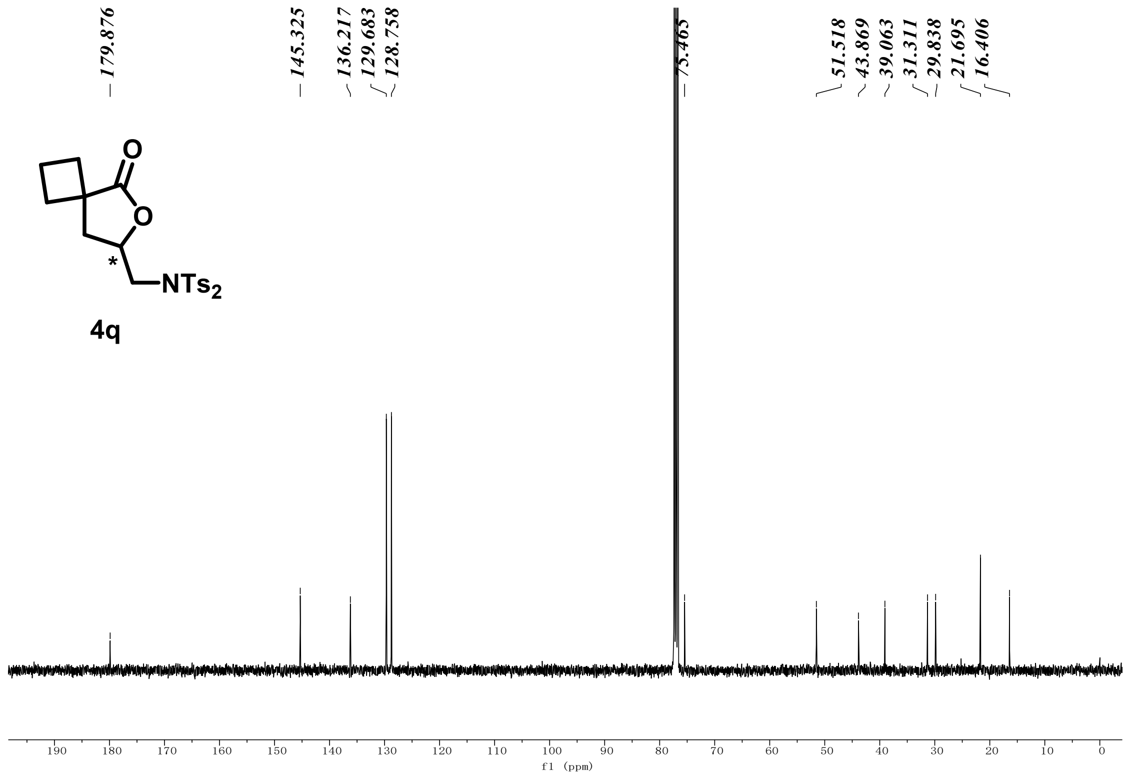

7.976
7.971
7.959
7.954
7.368
7.347
7.218
7.207
7.203
7.195
7.187
7.179
7.173
7.163
4.824
4.821
4.814
4.805
4.796
4.789
4.786
4.203
4.185
4.164
4.145
3.749
3.738
3.709
3.698
3.613
3.573
3.291
3.252
2.978
2.939
2.885
2.845
2.458
2.320
2.305
2.287
2.272
1.924
1.900
1.891
1.867
O
O
4o
Ts2N
4.04
4.11
4.08
1.04
1.15
1.04
1.04
1.04
2.09
6.00
1.17
1.07
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)


178.595
144.365
139.664
138.289
135.135
128.686
127.724
126.149
125.989
123.480
123.429
74.957
50.559
49.580
42.348
39.584
20.682
O
O
Ts_2N
4o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

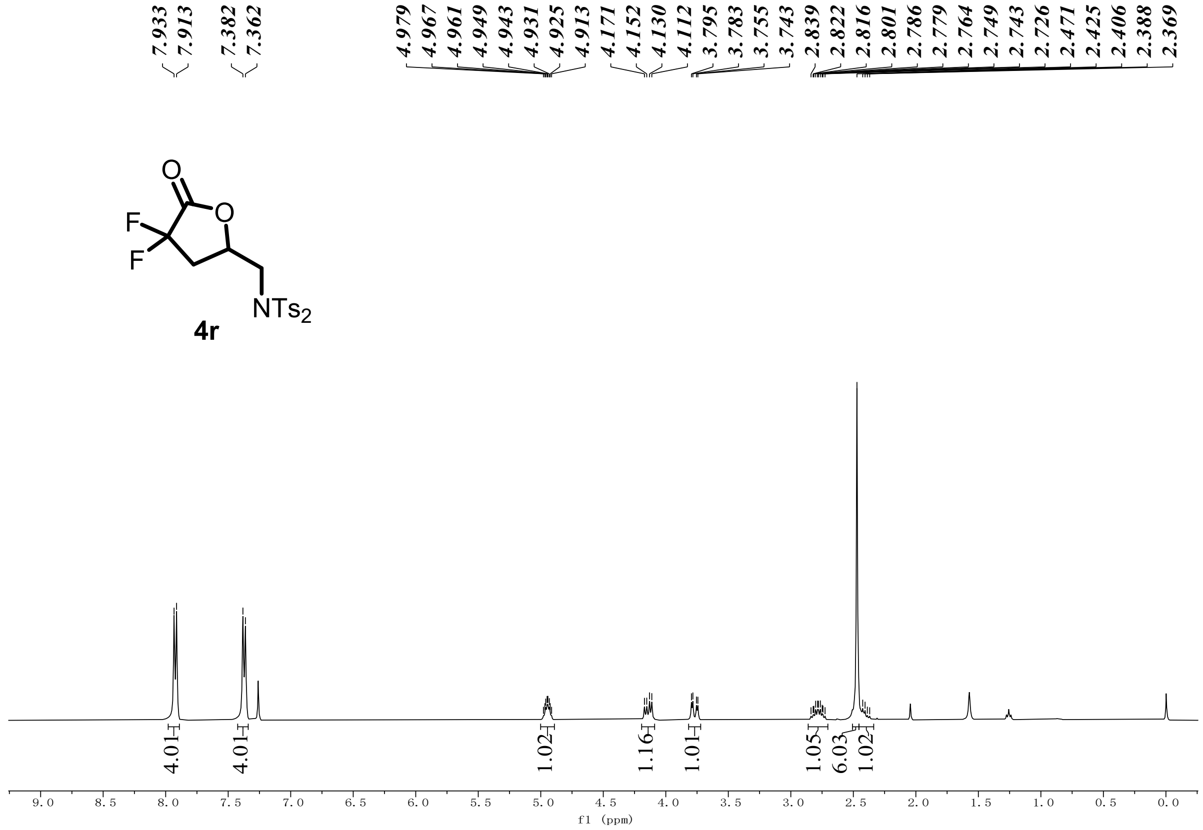

7.958
7.938
7.365
7.345
4.978
4.961
4.945
4.929
4.162
4.143
4.123
4.104
3.735
3.723
3.696
3.684
2.462
2.327
2.307
2.295
2.275
2.127
2.111
2.094
2.079
1.313
1.305
1.299
1.292
1.287
1.278
1.254
1.245
1.241
1.227
1.220
1.016
1.008
0.991
0.985
0.974
0.963
0.957
0.948
0.940
0.932
4.03
4.03
1.04
1.07
1.04
6.01
1.07
1.07
2.26
2.11
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
0.0
−0.5
f1 (ppm)


178.688
145.341
136.196
129.698
128.743
75.692
51.540
32.641
21.700
19.791
15.234
15.072
O
O
Ts2N
4p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

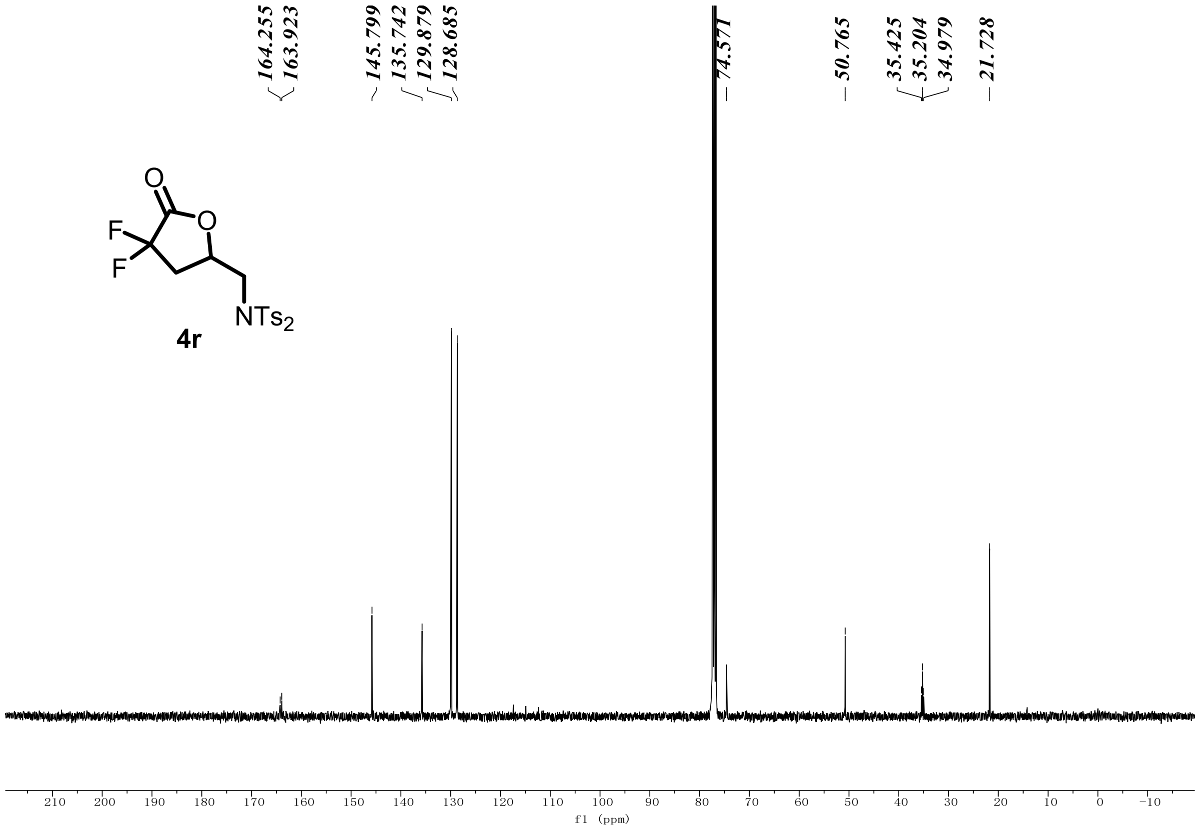

7.960
7.940
7.367
7.347
4.679
4.673
4.662
4.655
4.644
4.636
4.625
4.052
4.034
4.013
3.994
3.663
3.651
3.623
3.612
2.572
2.566
2.553
2.543
2.525
2.462
2.414
2.399
2.382
2.366
2.168
2.163
2.152
2.140
2.131
2.116
2.107
2.094
2.051
2.044
2.037
2.031
2.018
2.002
1.998
1.984
1.978
O
O
*
NTs2
4q
4.00
4.05
1.04
1.05
1.05
1.02
6.03
2.07
1.04
4.13
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 0.0 -0.5
f1 (ppm)

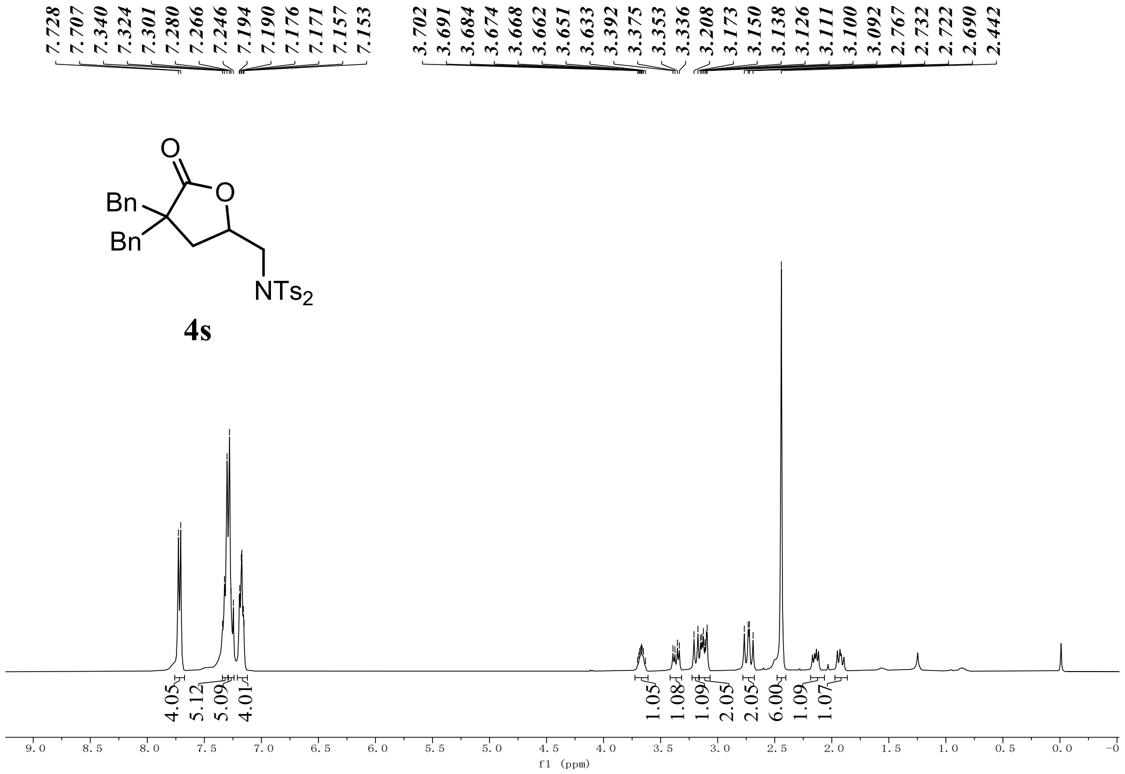

179.876
145.325
136.217
129.683
128.758
75.465
51.518
43.869
39.063
31.311
29.838
21.695
16.406
O
O
*
NTs2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

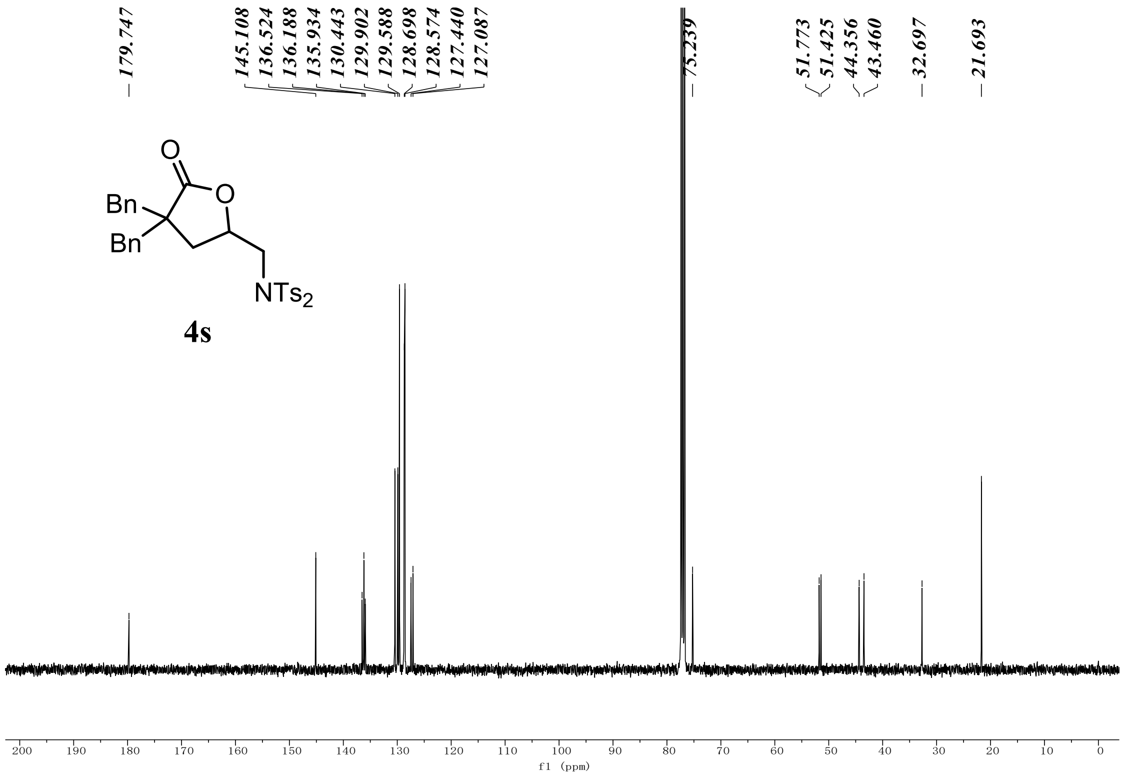
4q


7.933
7.913
7.382
7.362
4.979
4.967
4.961
4.949
4.943
4.931
4.925
4.913
4.171
4.152
4.130
4.112
3.795
3.783
3.755
3.743
2.839
2.822
2.816
2.801
2.786
2.779
2.764
2.749
2.743
2.726
2.471
2.425
2.406
2.388
2.369
O
O
F
F
NTs2
4.01
4.01
1.02
1.16
1.01
1.05
6.03
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
0.0
f1 (ppm)

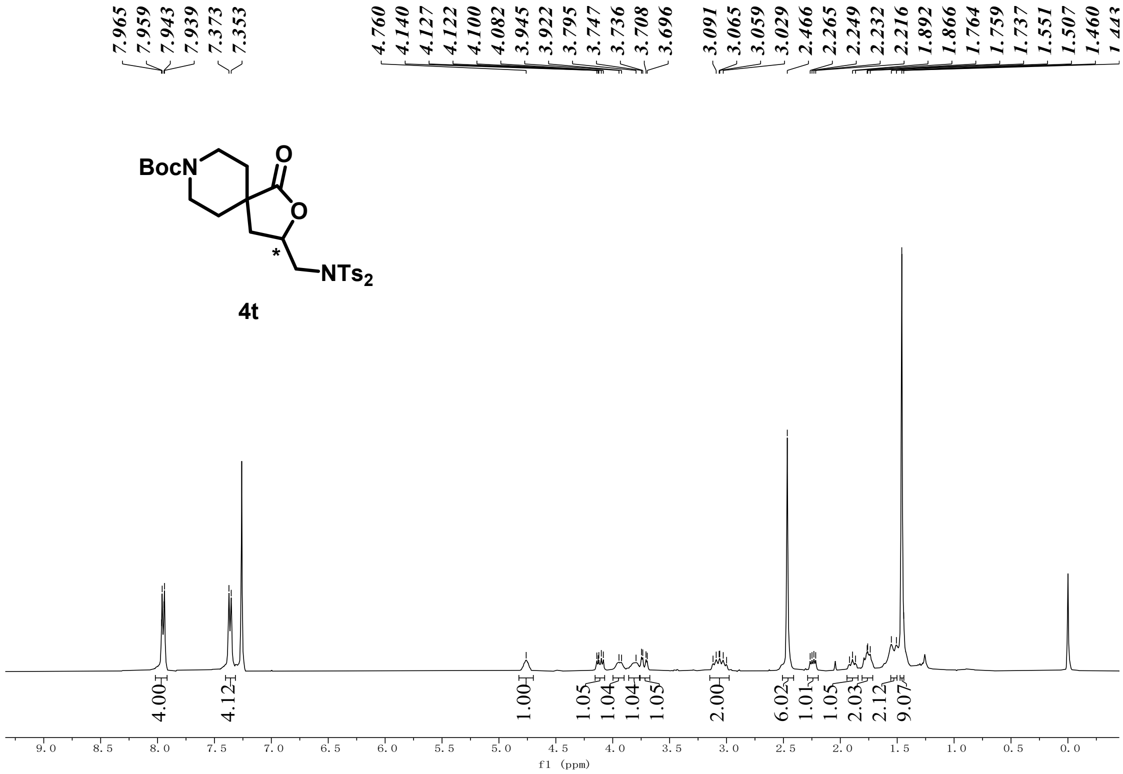
4r


164.255
163.923
145.799
135.742
129.879
128.685
74.571
50.765
35.425
35.204
34.979
21.728
O
O
F
F
NTs2
4r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

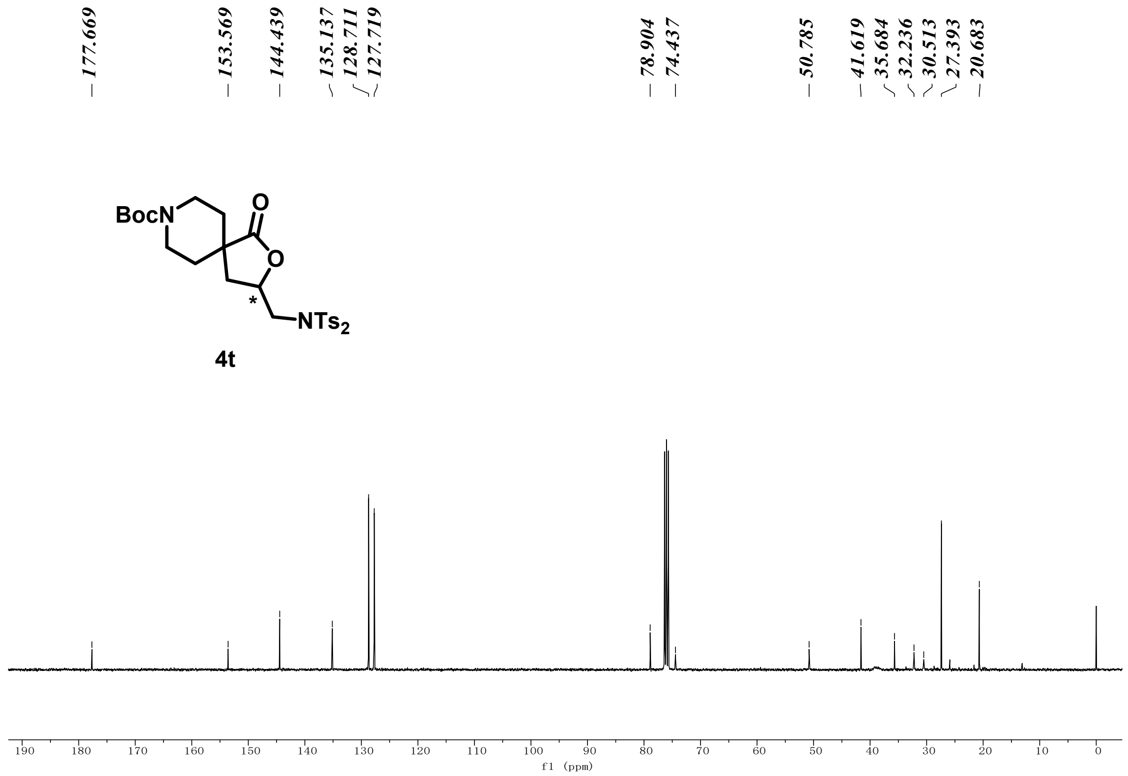

7.728
7.707
7.340
7.324
7.301
7.280
7.266
7.246
7.194
7.190
7.176
7.171
7.157
7.153
3.702
3.691
3.684
3.674
3.668
3.662
3.651
3.633
3.392
3.375
3.353
3.336
3.208
3.173
3.150
3.138
3.126
3.111
3.100
3.092
2.767
2.732
2.722
2.690
2.442
O
O
Bn
Bn
NTs2
4s
4.05
5.12
5.09
4.01
1.05
1.08
1.09
2.05
2.05
6.00
1.09
1.07
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 0.0 −0
f1 (ppm)


179.747
145.108
136.524
136.188
135.934
130.443
129.902
129.588
128.698
128.574
127.440
127.087
75.239
51.773
51.425
44.356
43.460
32.697
21.693
O
O
Bn
Bn
NTs2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4s

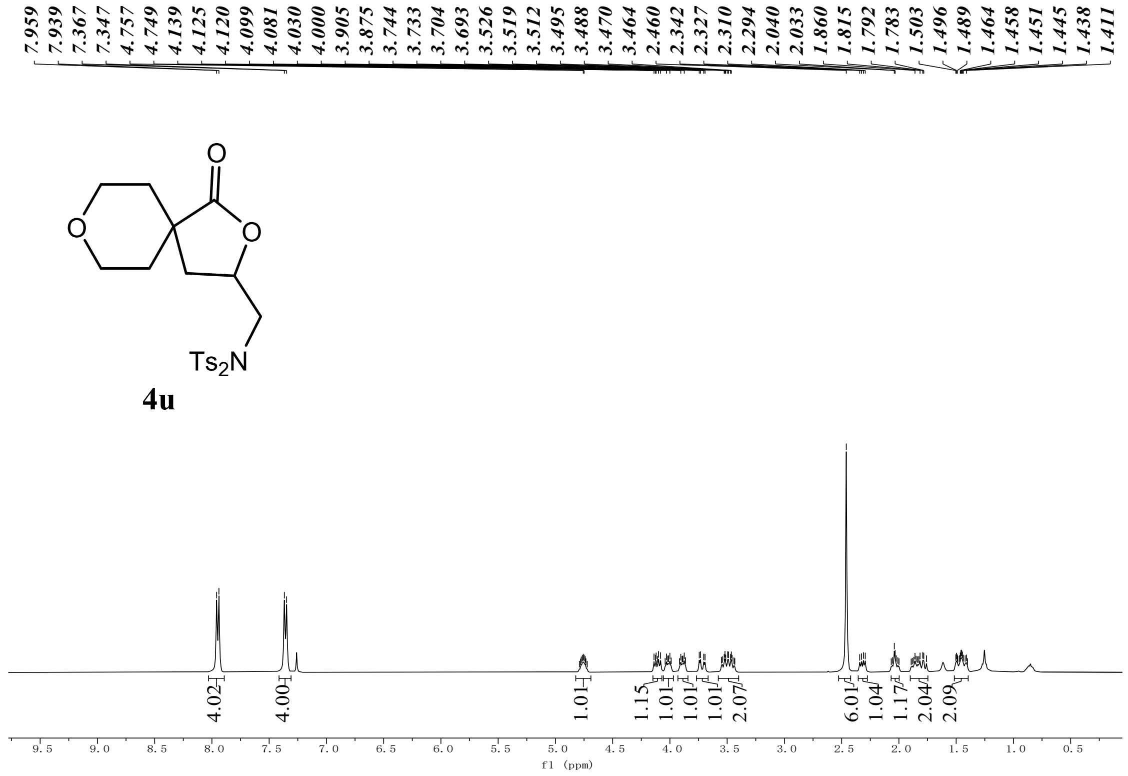

7.965
7.959
7.943
7.939
7.373
7.353
4.760
4.140
4.127
4.122
4.100
4.082
3.945
3.922
3.795
3.747
3.736
3.708
3.696
3.091
3.065
3.059
3.029
2.466
2.265
2.249
2.232
2.216
1.892
1.866
1.764
1.759
1.737
1.551
1.507
1.460
1.443
BocN
O
O
*
NTs2
4.00
4.12
1.00
1.05
1.04
1.04
1.05
2.00
6.02
1.01
1.05
2.03
2.12
9.07
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
0.0
f1 (ppm)

4t



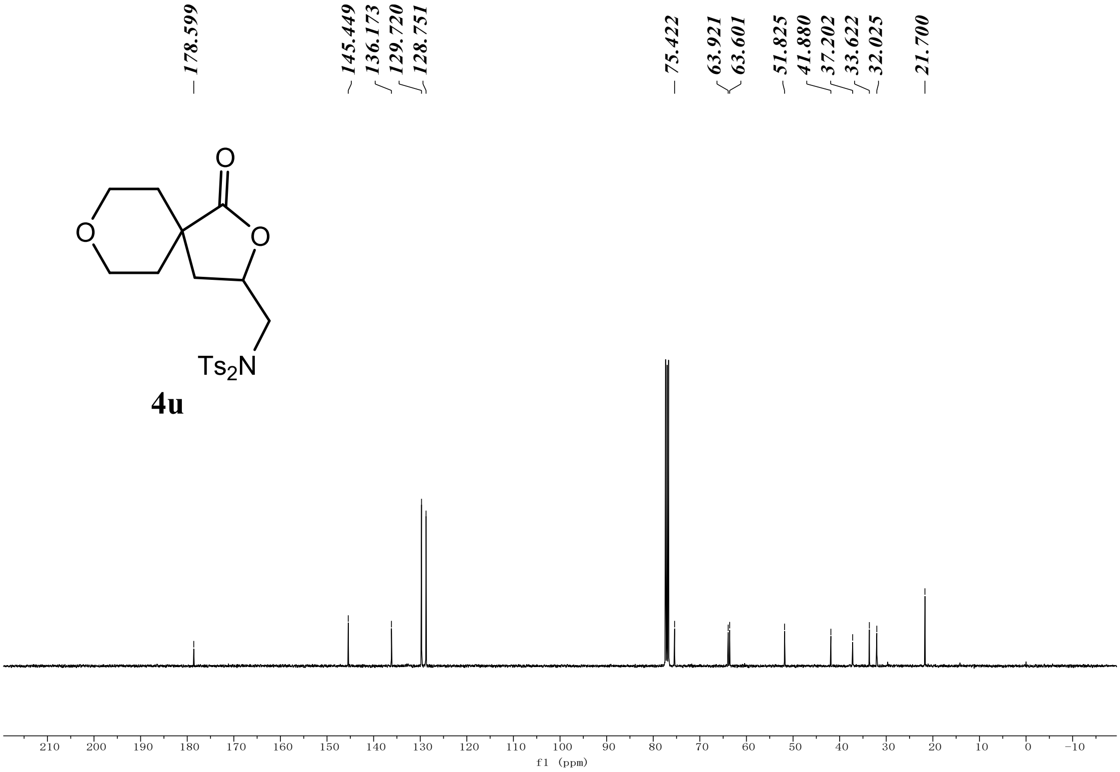
4t

177.669
153.569
144.439
135.137
128.711
127.719
78.904
74.437
50.785
41.619
35.684
32.236
30.513
27.393
20.683
BocN
O
O
*
NTs2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



4u

7.959
7.939
7.367
7.347
4.757
4.749
4.139
4.125
4.120
4.099
4.081
4.030
4.000
3.905
3.875
3.744
3.733
3.704
3.693
3.526
3.519
3.512
3.495
3.488
3.470
3.464
2.460
2.342
2.327
2.310
2.294
2.040
2.033
1.860
1.815
1.792
1.783
1.503
1.496
1.489
1.464
1.458
1.451
1.445
1.438
1.411
O
O
O
Ts2N
4.02
4.00
1.01
1.15
1.01
1.01
1.01
2.07
6.01
1.04
1.17
2.04
2.09
9.5 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)


178.599
145.449
136.173
129.720
128.751
75.422
63.921
63.601
51.825
41.880
37.202
33.622
32.025
21.700
O
O
O
Ts2N
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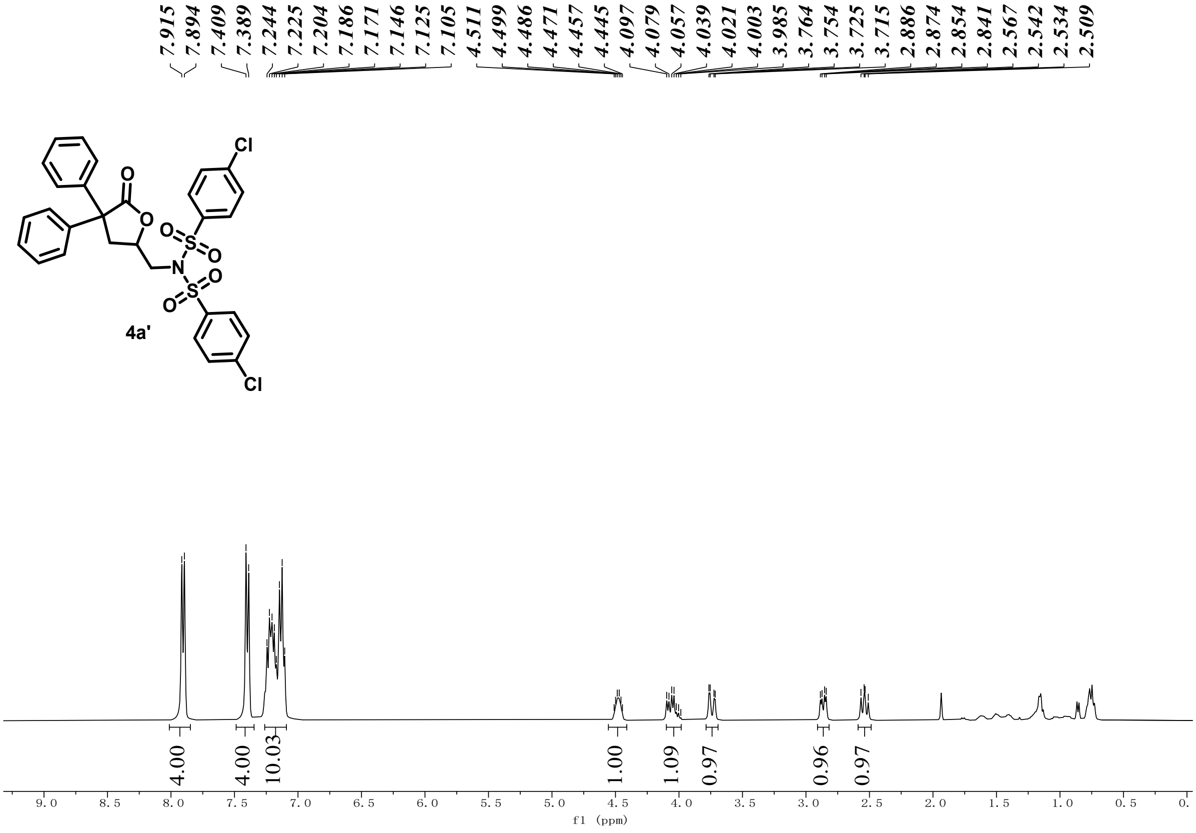
f1 (ppm)

4u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4.499
4.486
4.471
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4.445
4.097
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4.057
4.039
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4.003
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3.754
3.725
3.715
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2.874
2.854
2.841
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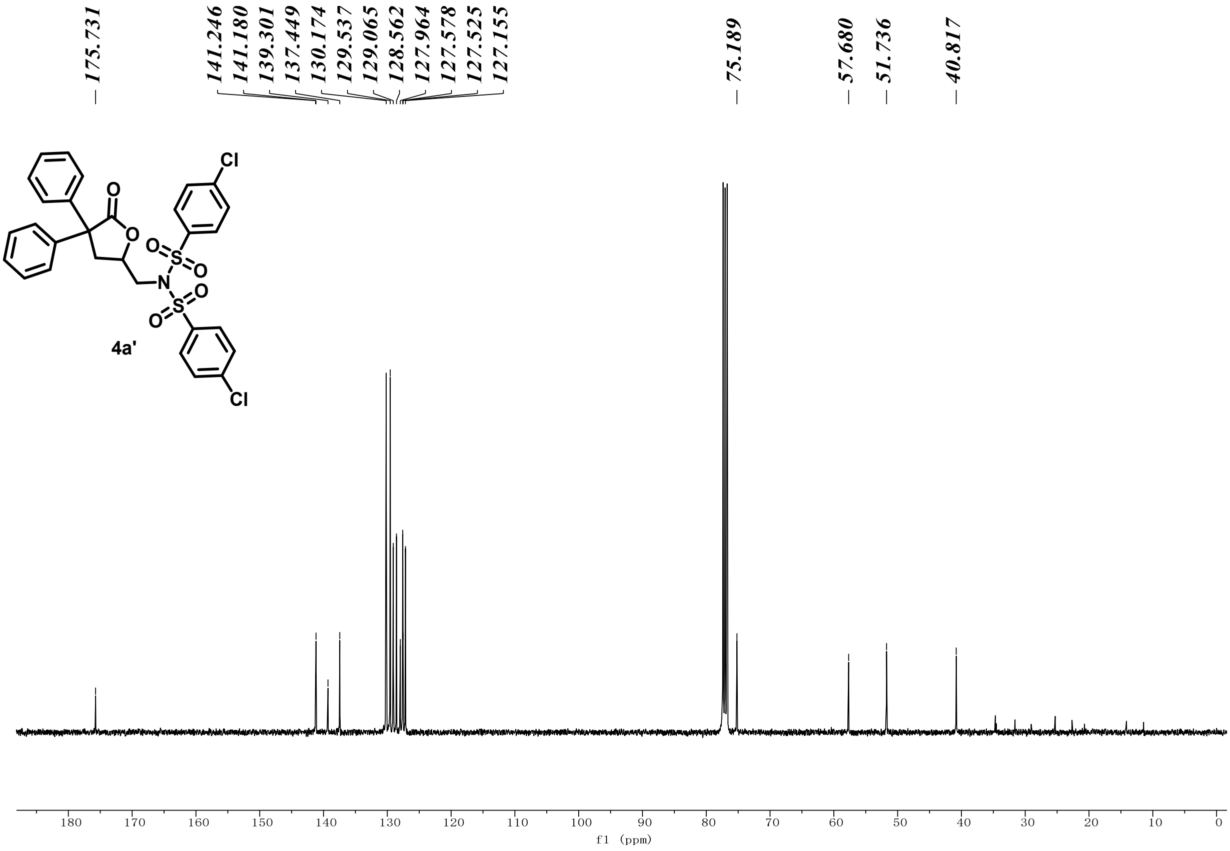
2.542
2.534
2.509
Cl
O
O
O
S
O
N
S
O
O
Cl
4.00
4.00
10.03
1.00
1.09
0.97
0.96
0.97
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)

4a'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57.680
51.736
40.817
Cl
O
O
O
S
O
N
S
O
O
Cl
4a'
180
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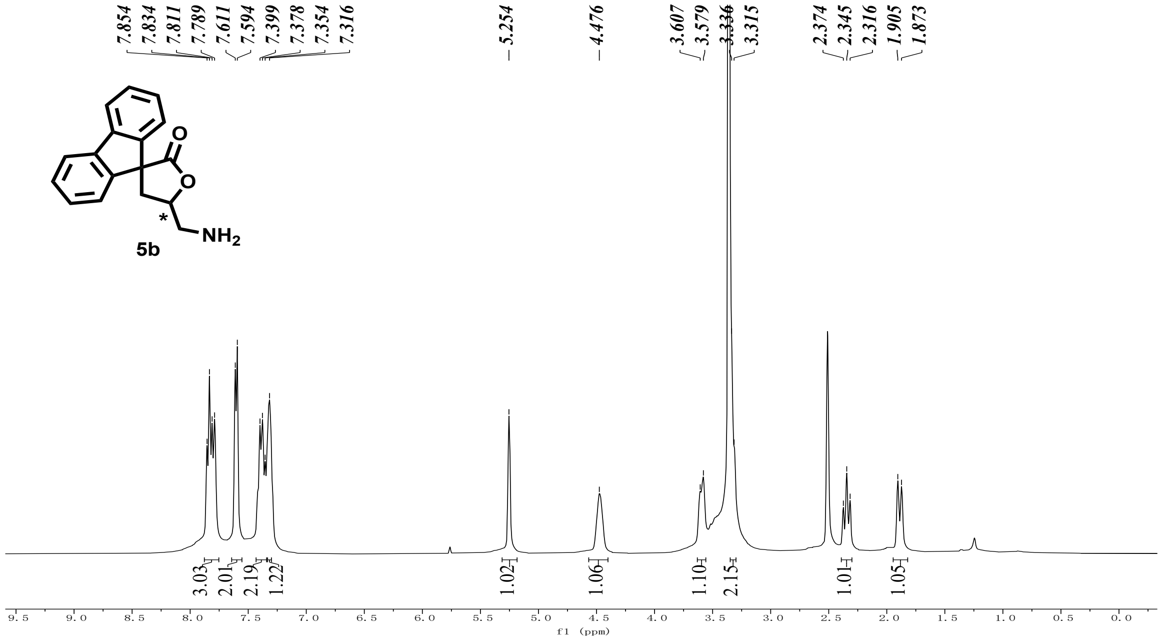
f1 (ppm)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2.316
1.905
1.873
O
O
*
NH2
5b
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2.01
2.19
1.22
1.02
1.06
1.10
2.15
1.01
1.05
9.5
9.0
8.5
8.0
7.5
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6.0
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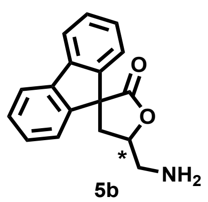
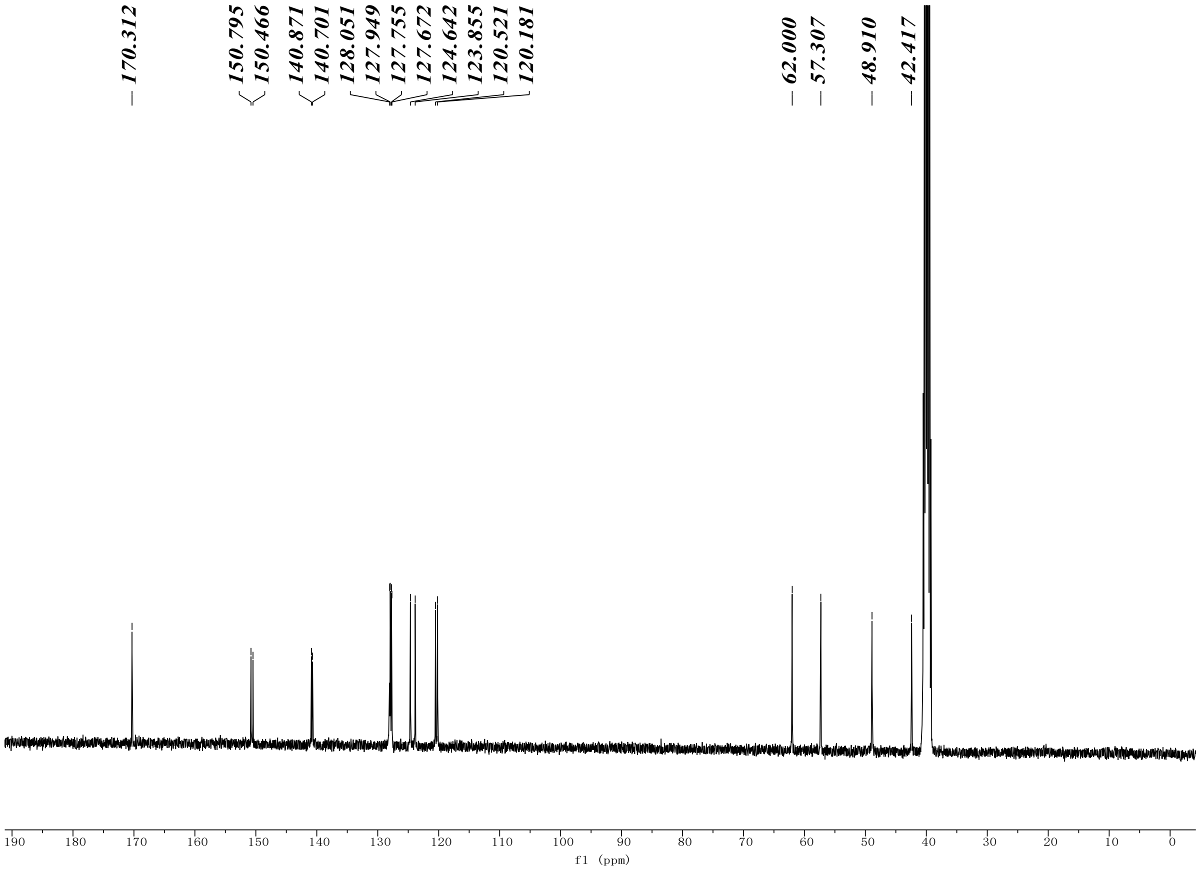
f1 (ppm)



5b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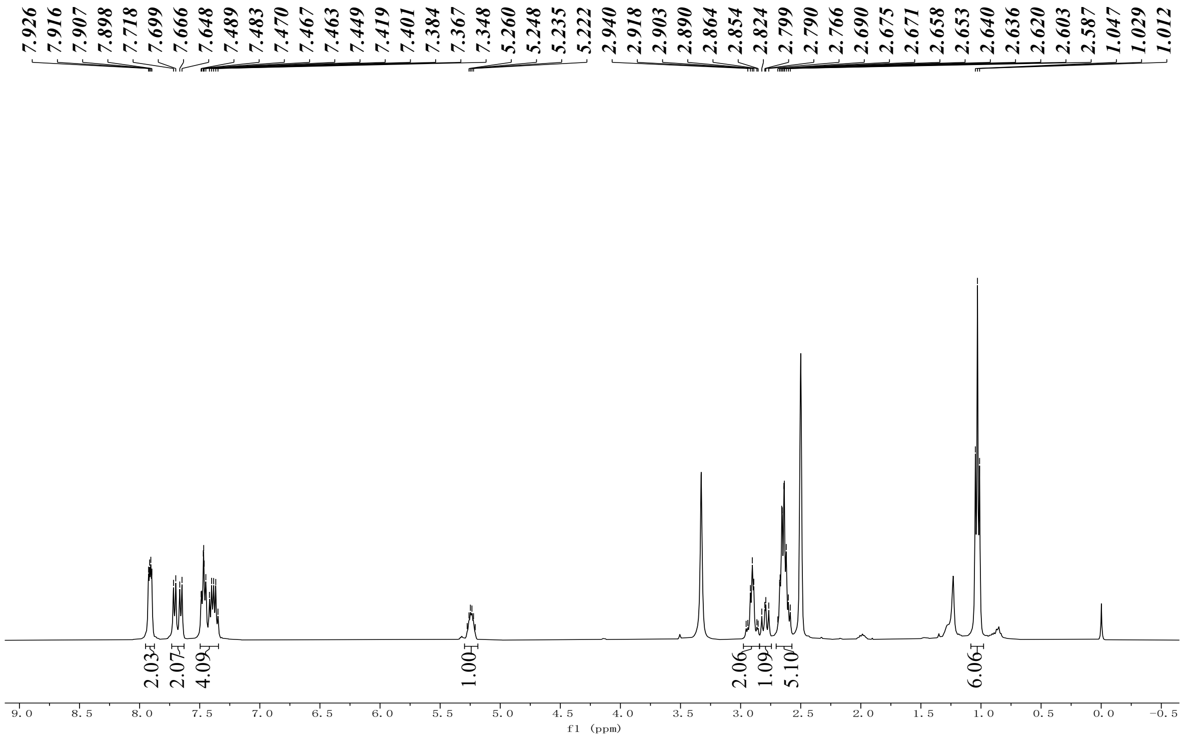
f1 (ppm)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1.00
2.06
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5.10
6.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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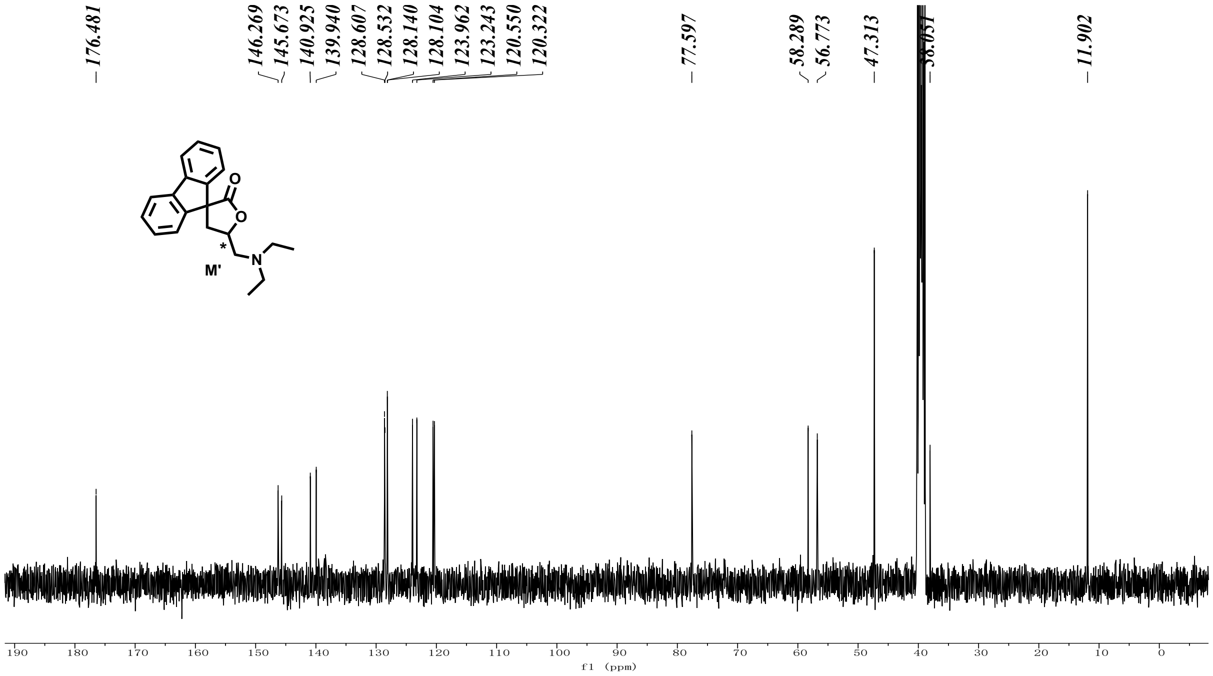
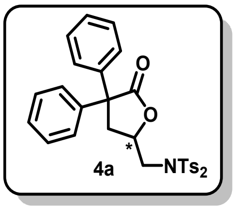
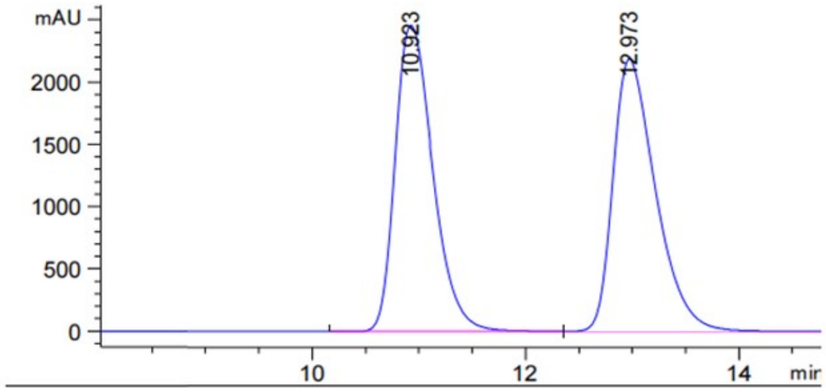


Racemic

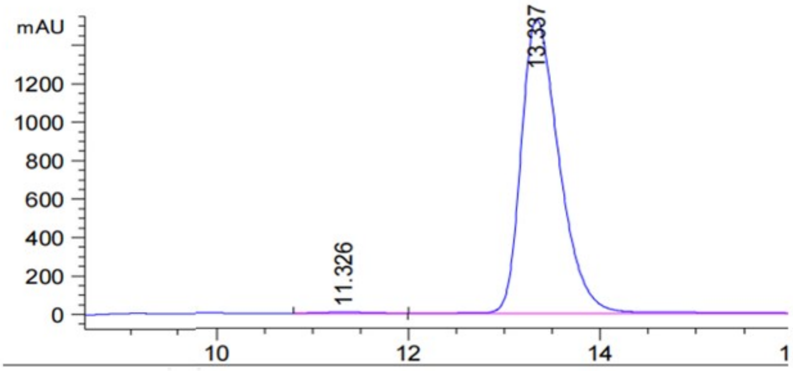




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		10.923	6.17346e4	2493.06470	49.8730
2		12.973	6.20490e4	2221.30762	50.1270
Total			1.23784e5	4714.37231	100.0000

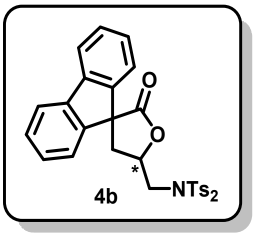
Chiral



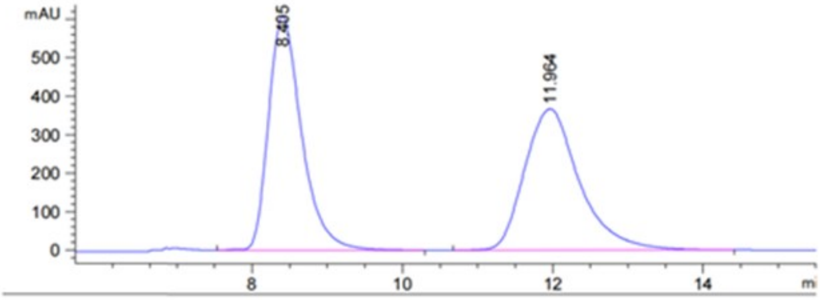
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.326	153.81976	5.98117	0.3758
2		13.337	4.07729e4	1479.59460	99.6242
Total			4.09267e4	1485.57577	100.0000

Racemic



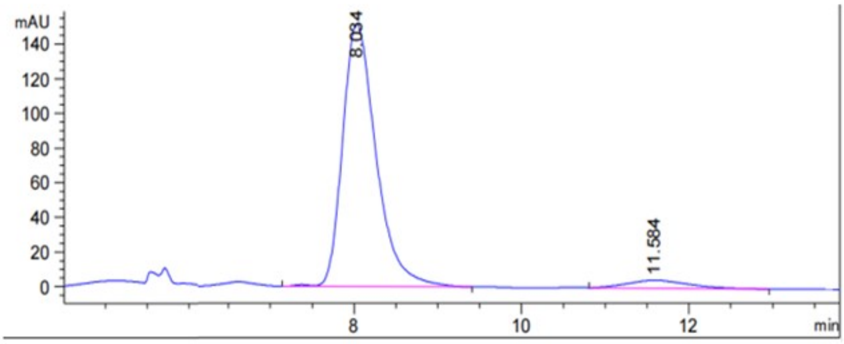
4b



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.405	1.86778e4	607.91687	50.0147
2		11.964	1.86668e4	367.24213	49.9853
Total			3.73446e4	975.15900	100.0000

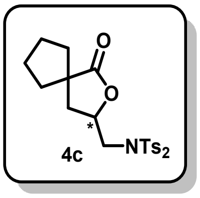
Chiral



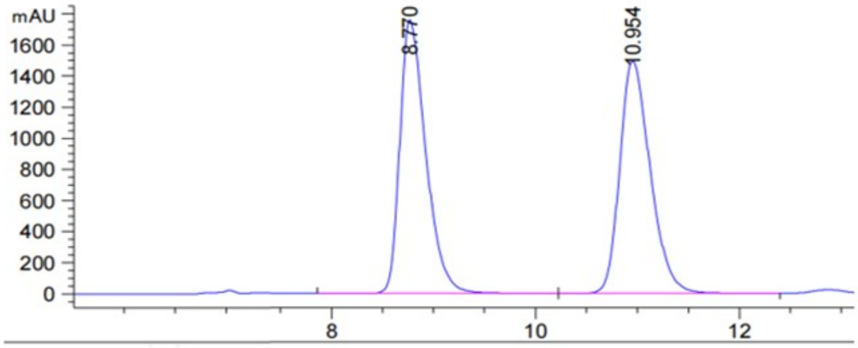
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.034	4225.91895	151.63417	94.8052
2		11.584	231.55588	4.56578	5.1948
Total			4457.47482	156.19995	100.0000

Racemic



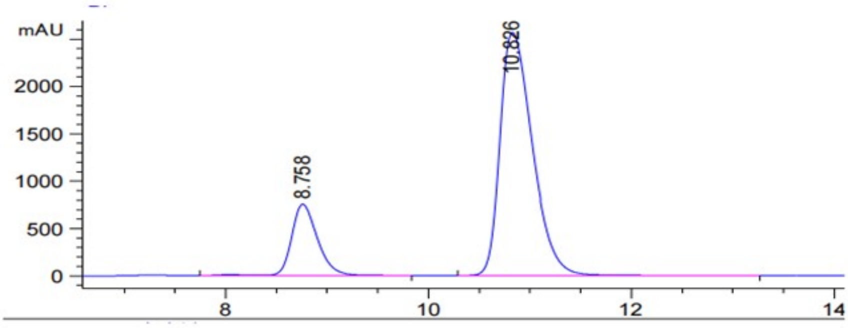
4c



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.770	3.11451e4	1761.72852	50.0003
2		10.954	3.11447e4	1494.63306	49.9997
Total			6.22898e4	3256.36157	100.0000

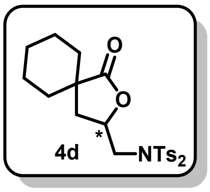
Chiral



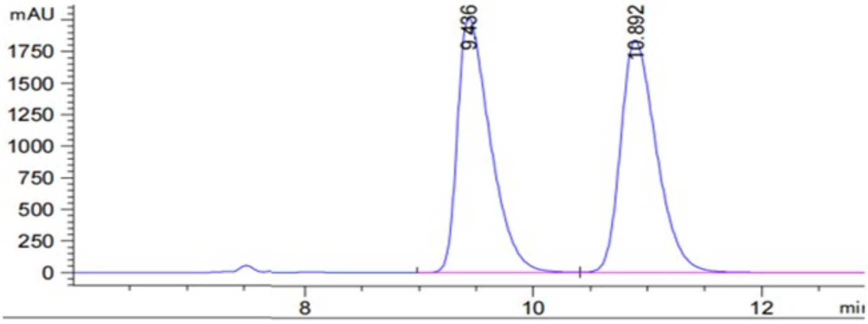
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.758	1.47181e4	833.95251	18.8469
2		10.826	6.33750e4	2854.73047	81.1531
Total			7.80931e4	3688.68298	100.0000

Racemic



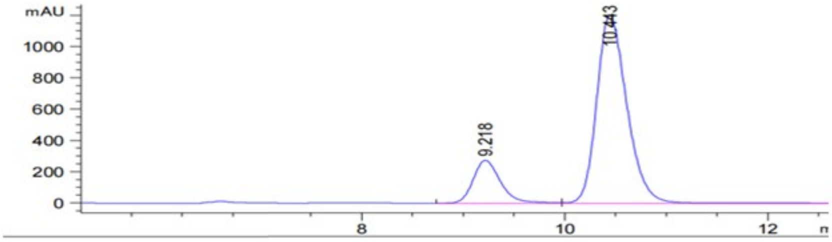
4d



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.436	4.02889e4	2018.62390	49.9317
2		10.892	4.03991e4	1841.14795	50.0683
Total			8.06881e4	3859.77185	100.0000

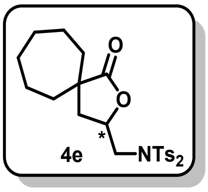
Chiral



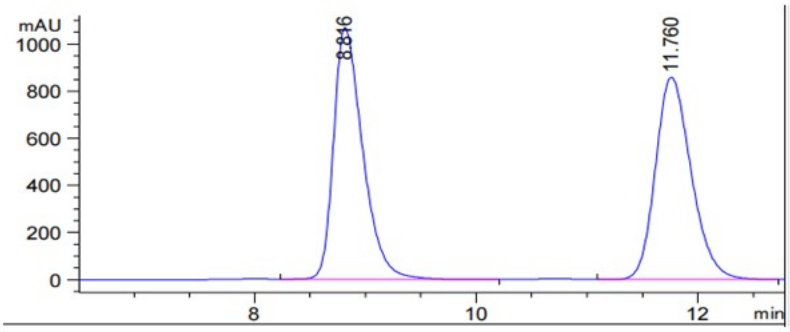
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.218	5169.50244	275.32242	17.2073
2		10.443	2.48730e4	1211.48157	82.7927
Total			3.00425e4	1486.80399	100.0000

Racemic



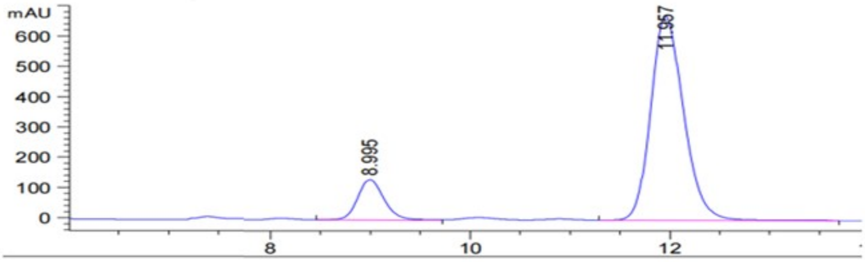
4e



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.816	1.95119e4	1067.37732	50.3603
2		11.760	1.92328e4	858.82690	49.6397
Total			3.87447e4	1926.20422	100.0000

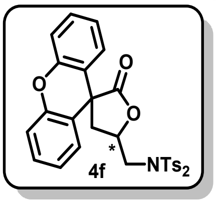
Chiral



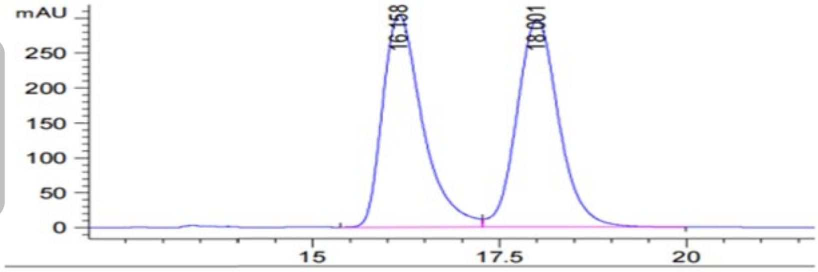
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.995	2433.95898	132.71954	13.6498
2		11.957	1.53975e4	674.55322	86.3502
Total			1.78315e4	807.27277	100.0000

Racemic



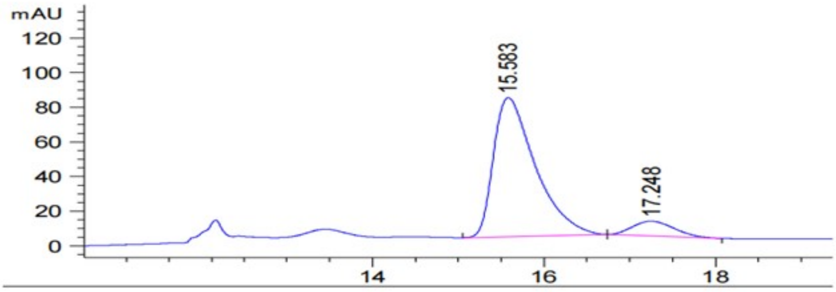
4f



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		16.158	1.13327e4	303.83319	49.4127
2		18.001	1.16021e4	297.63330	50.5873
Total			2.29348e4	601.46649	100.0000

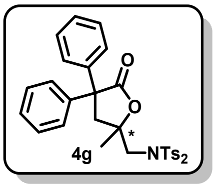
Chiral

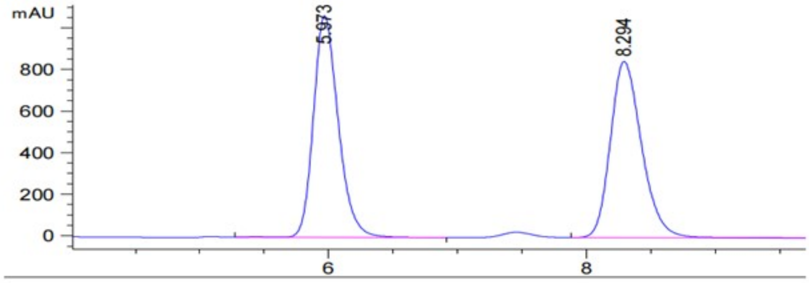


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		15.583	2723.85303	80.21117	90.4615
2		17.248	287.20981	8.52694	9.5385
Total			3011.06284	88.73811	100.0000

Racemic

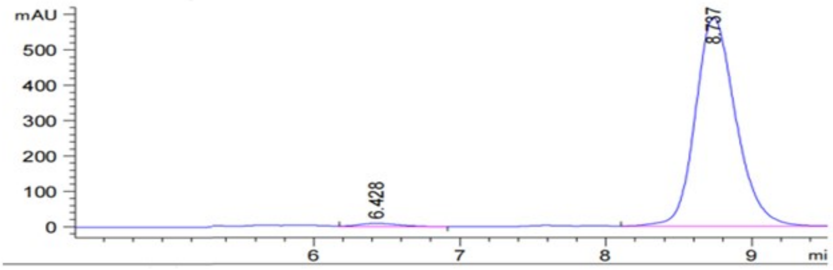




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		5.973	1.43139e4	1066.61292	50.0389
2		8.294	1.42916e4	848.69830	49.9611
Total			2.86055e4	1915.31122	100.0000

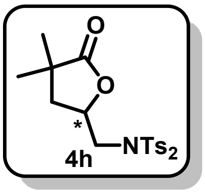
Chiral



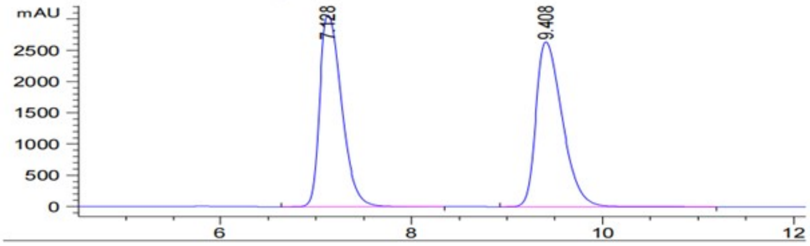
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		6.428	178.92024	9.34496	1.5556
2		8.737	1.13228e4	589.69086	98.4444
Total			1.15018e4	599.03582	100.0000

Racemic



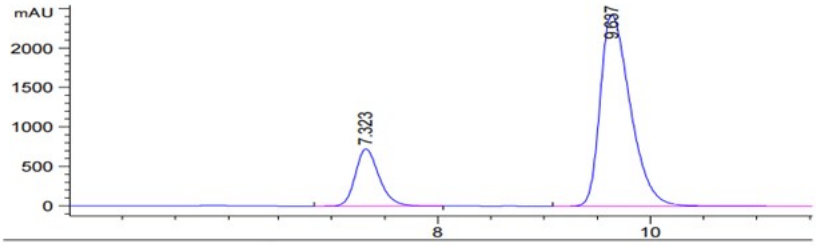
4h



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		7.128	4.90425e4	3066.24170	49.2581
2		9.408	5.05198e4	2639.55176	50.7419
Total			9.95623e4	5705.79346	100.0000

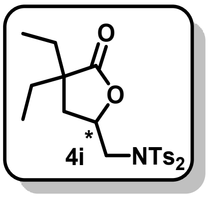
Chiral



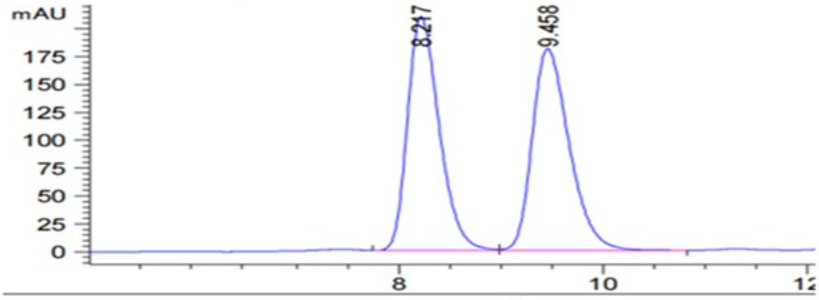
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		7.323	1.12476e4	724.76978	19.0468
2		9.637	4.78051e4	2428.60376	80.9532
Total			5.90528e4	3153.37354	100.0000

Racemic



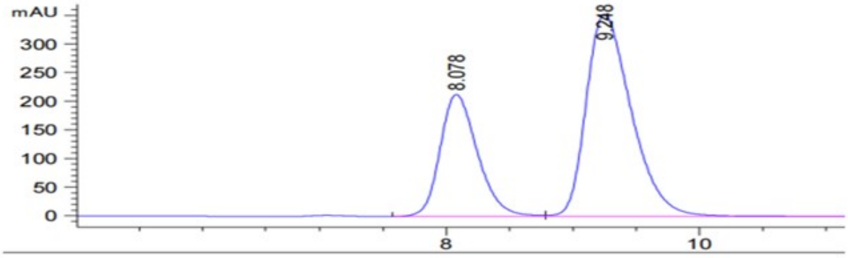
4i



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.217	4527.40869	209.95518	49.8160
2		9.458	4560.84766	181.06865	50.1840
Total			9088.25635	391.02383	100.0000

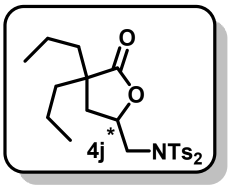
Chiral

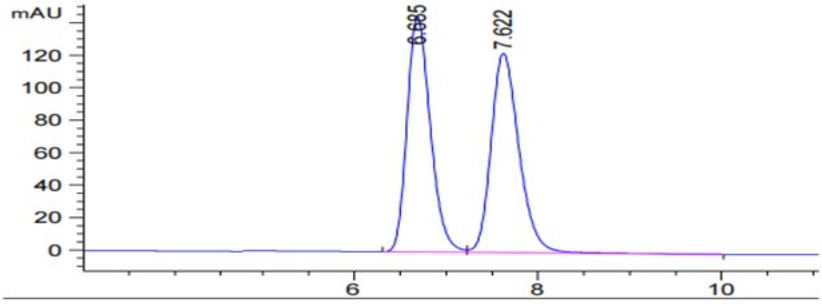


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.078	4382.39648	213.09979	33.3018
2		9.248	8777.25879	353.09375	66.6982
Total			1.31597e4	566.19354	100.0000

Racemic

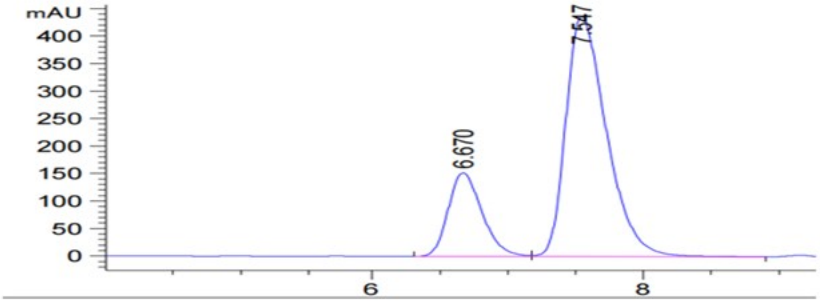




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		6.685	2551.46973	144.98872	49.5584
2		7.622	2596.93799	122.83116	50.4416
Total			5148.40771	267.81989	100.0000

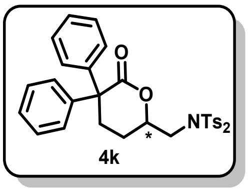
Chiral



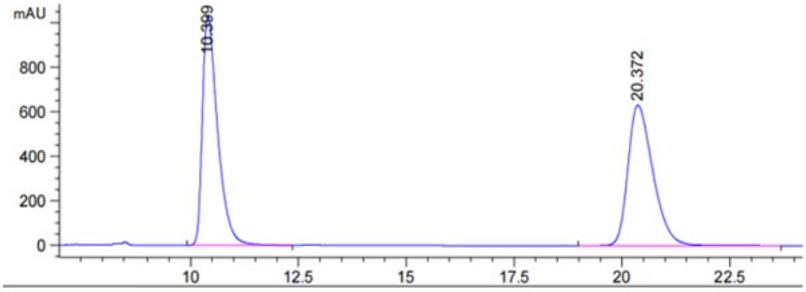
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		6.670	2633.49097	151.98375	22.4532
2		7.547	9095.29004	436.30298	77.5468
Total			1.17288e4	588.28673	100.0000

Racemic



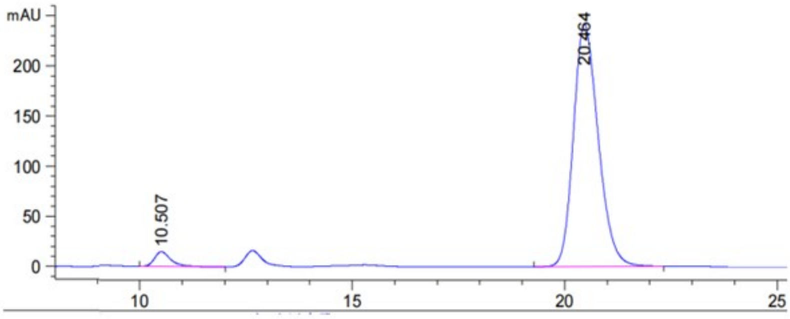
4k



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		10.399	2.53124e4	1032.52991	49.8338
2		20.372	2.54813e4	633.17181	50.1662
Total			5.07937e4	1665.70172	100.0000

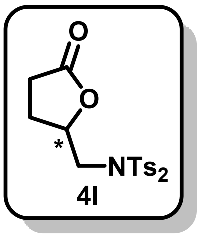
Chiral



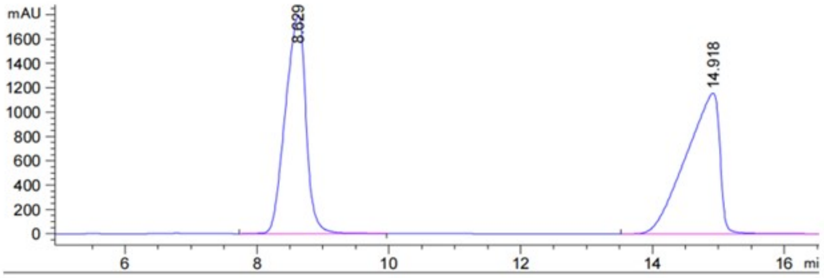
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		10.507	400.17773	15.03325	3.8367
2		20.464	1.00301e4	246.83543	96.1633
Total			1.04303e4	261.86868	100.0000

Racemic



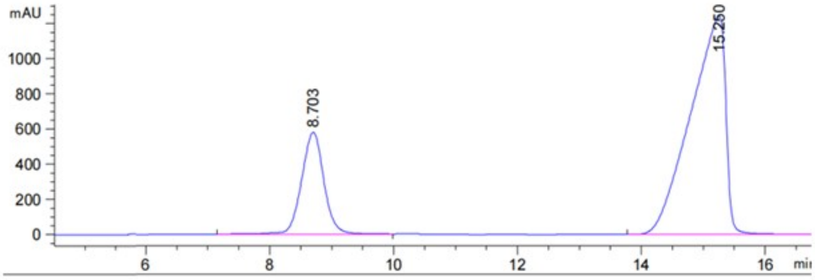
4l



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.629	3.89941e4	1794.40173	48.6661
2		14.918	4. 11318e4	1155.61157	51.3339
Total			8.01259e4	2950.01331	100.0000

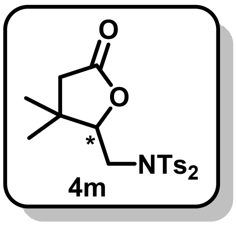
Chiral



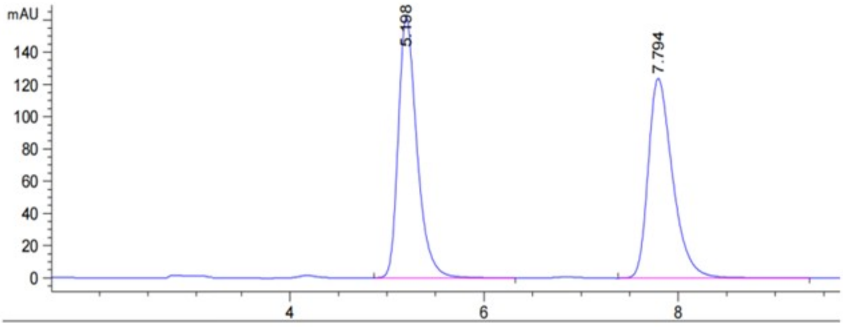
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.703	1.44466e4	578.92560	22.7723
2		15.250	4. 89928e4	1246.74390	77.2277
Total			6.34394e4	1825.66949	100.0000

Racemic



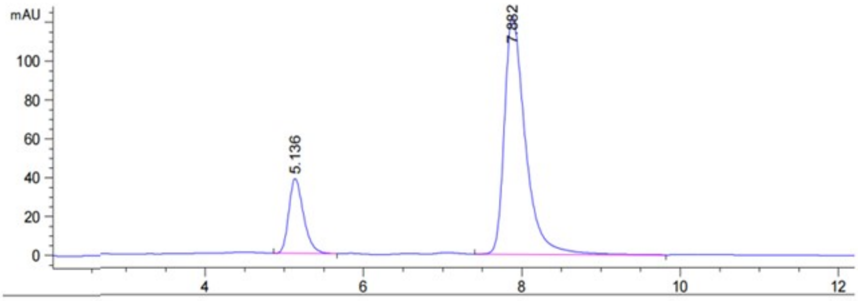
4m



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		5.198	2170.31836	161.43170	49.9773
2		7.794	2172.29321	123.79023	50.0227
Total			4342.61157	285.22193	100.0000

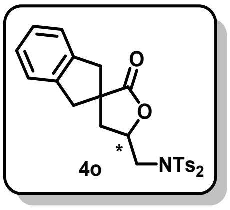
Chiral



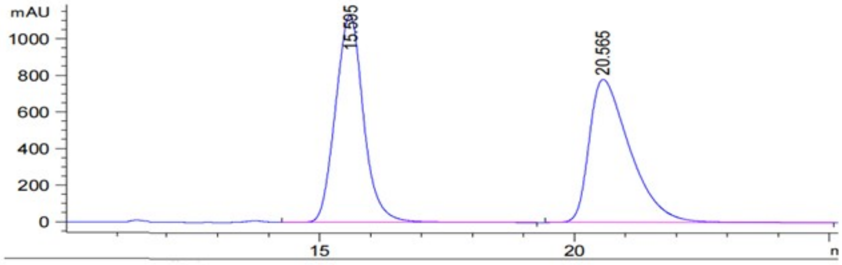
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		5.136	499.69714	38.49426	18.1755
2		7.882	2249.59961	122.21233	81.8245
Total			2749.29675	160.70659	100.0000

Racemic



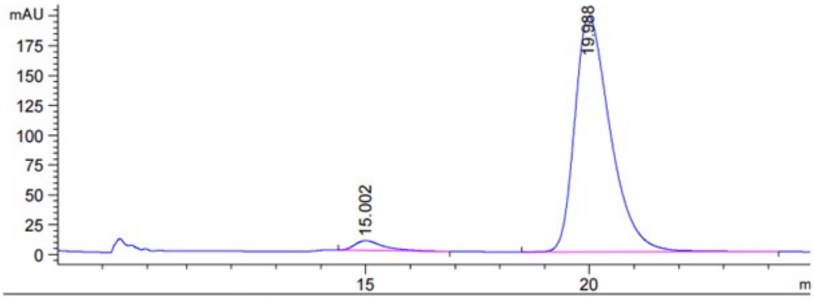
4o



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		15.595	4.33326e4	1130.80396	49.9718
2		20.565	4.33815e4	781.68488	50.0282
Total			8.67141e4	1912.48883	100.0000

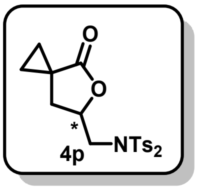
Chiral

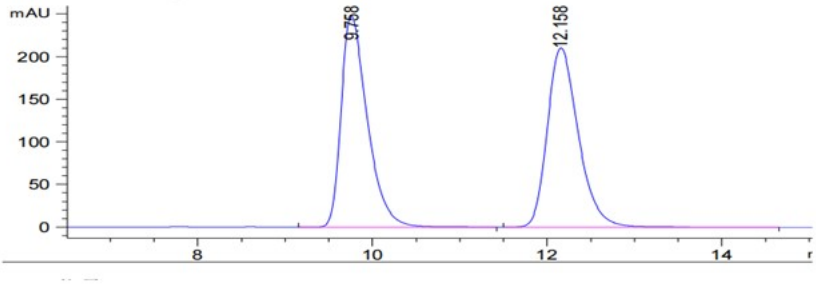


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		15.002	357.03622	8.09039	3.1703
2		19.988	1.09050e4	197.35199	96.8297
Total			1.12620e4	205.44238	100.0000

Racemic

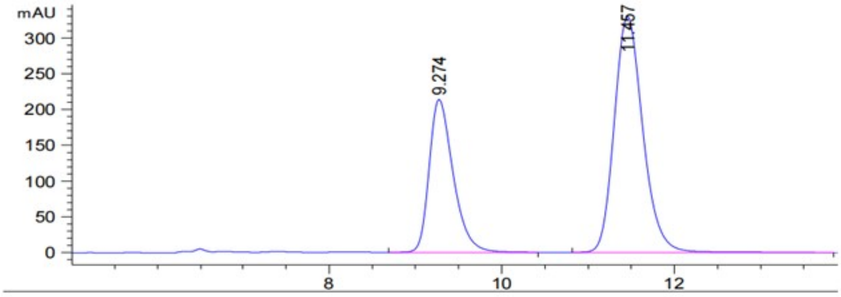




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.758	5188.95898	247.82069	49.8932
2		12.158	5211.17041	210.25146	50.1068
Total			1.04001e4	458.07216	100.0000

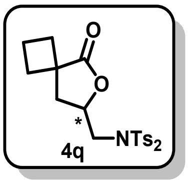
Chiral



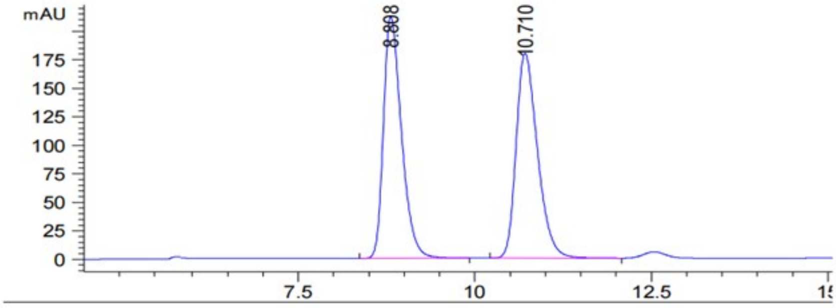
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.274	4127.03760	213.58298	35.3146
2		11.457	7559.45850	329.74402	64.6854
Total			1.16865e4	543.32700	100.0000

Racemic



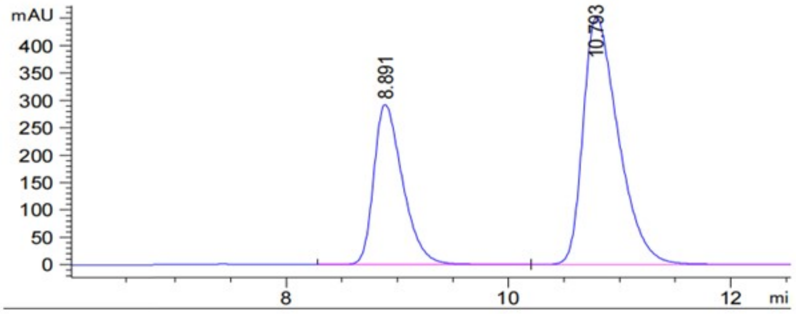
4q



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.808	3753.58521	211.87057	49.8214
2		10.710	3780.49414	179.64853	50.1786
Total			7534.07935	391.51910	100.0000

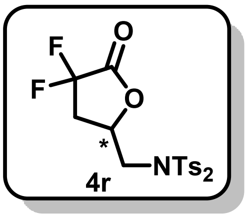
Chiral

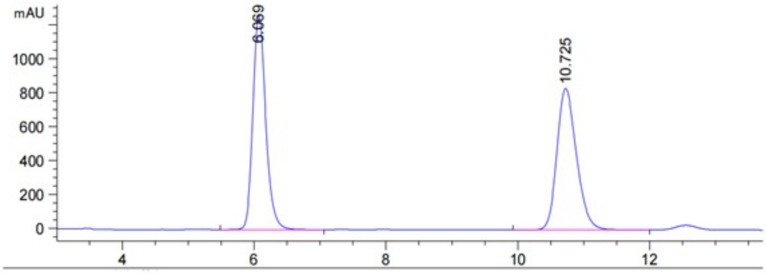


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		8.891	5261.69482	291.21335	34.6082
2		10.793	9941.92676	450.98694	65.3918
Total			1.52036e4	742.20029	100.0000

Racemic

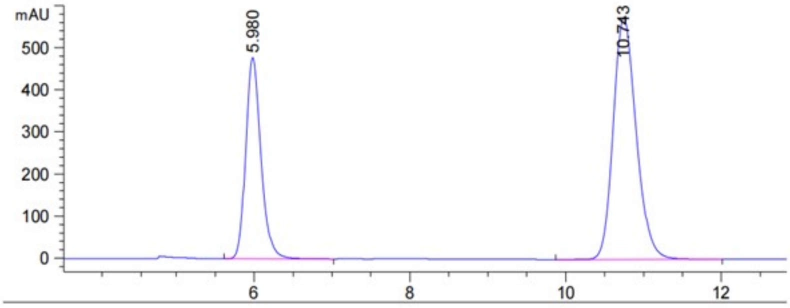

4r



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		6.069	1.71591e4	1263.31030	49.9252
2		10.725	1.72105e4	833.80402	50.0748
Total			3.43696e4	2097.11432	100.0000

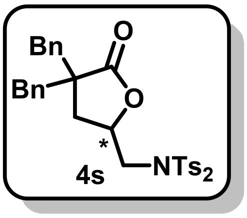
Chiral



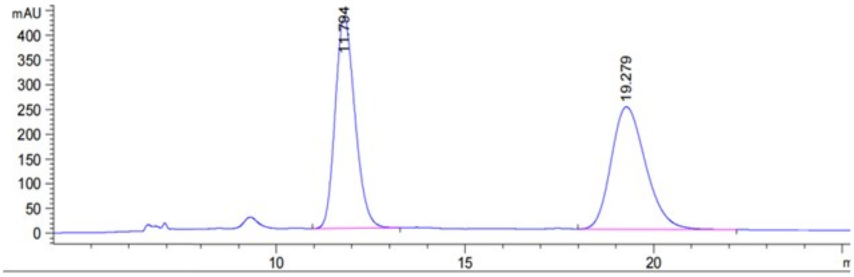
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		5.980	6544.95361	478.14331	35.7359
2		10.743	1.17698e4	574.81445	64.2641
Total			1.83148e4	1052.95776	100.0000

Racemic



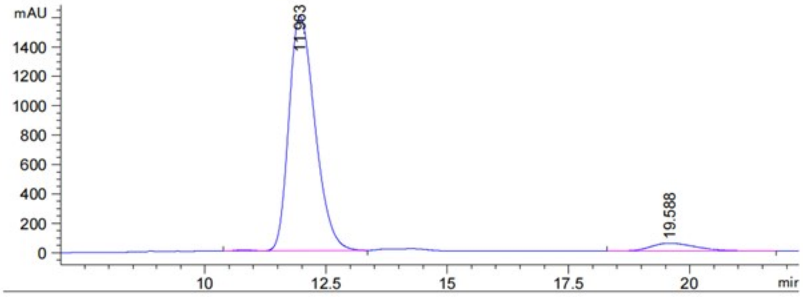
4s



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.794	1.56604e4	428.69846	49.7845
2		19.279	1.57960e4	248.46797	50.2155
Total			3.14565e4	677.16643	100.0000

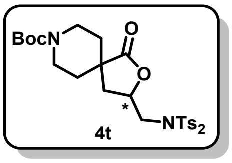
Chiral



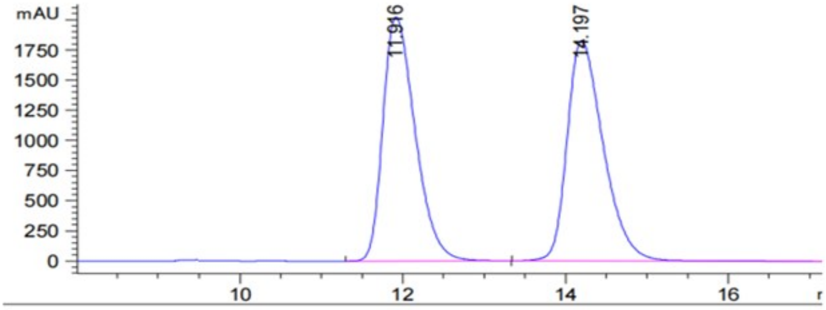
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.963	6.00173e4	1594.40735	94.5600
2		19.588	3452.80420	53.64519	5.4400
Total			6.34701e4	1648.05254	100.0000

Racemic



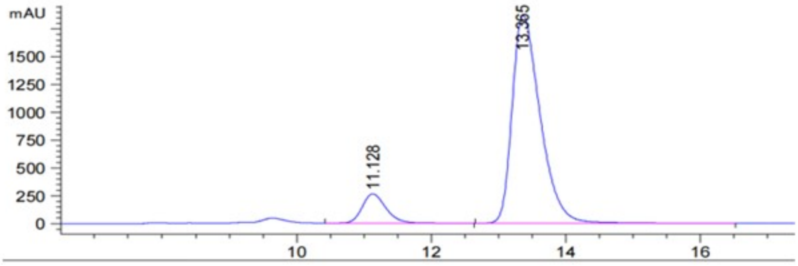
4t



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.916	5.58901e4	2027.41980	49.7109
2		14.197	5.65401e4	1830.91187	50.2891
Total			1.12430e5	3858.33167	100.0000

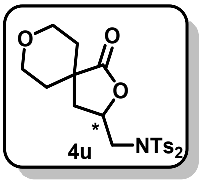
Chiral

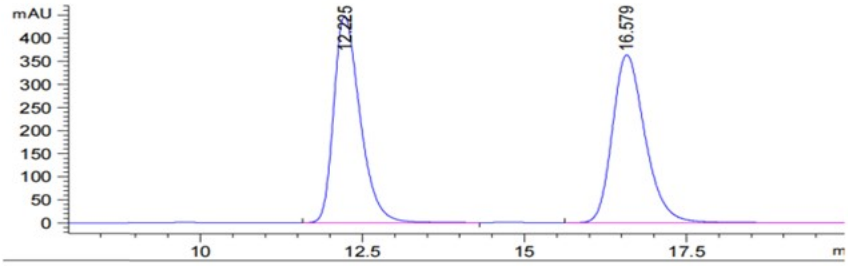


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.128	6452.74170	262.58838	10.6718
2		13.365	5.40129e4	1866.01721	89.3282
Total			6.04656e4	2128.60559	100.0000

Racemic

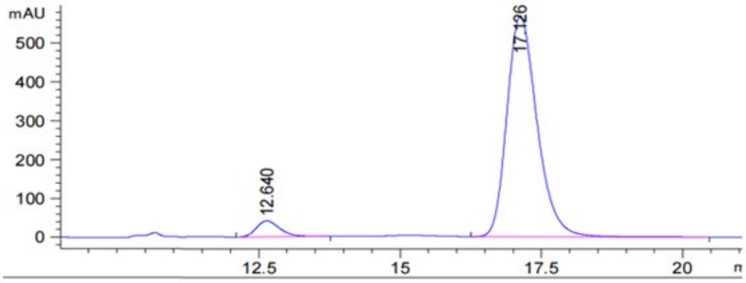




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		12.225	1.26430e4	447.33575	49.7426
2		16.579	1.27739e4	363.48743	50.2574
Total			2.54169e4	810.82318	100.0000

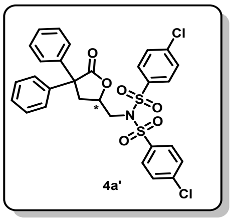
Chiral



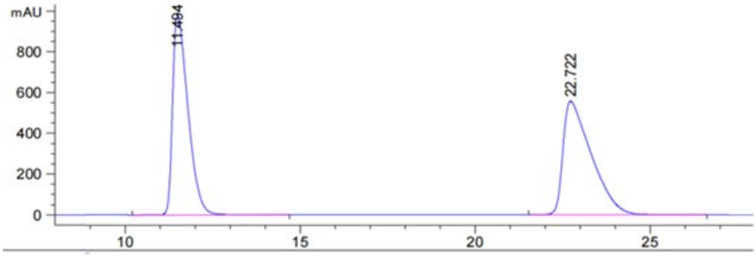
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		12.640	1204.85767	41.48252	5.4013
2		17.126	2.11019e4	568.51923	94.5987
Total			2.23068e4	610.00175	100.0000

Racemic



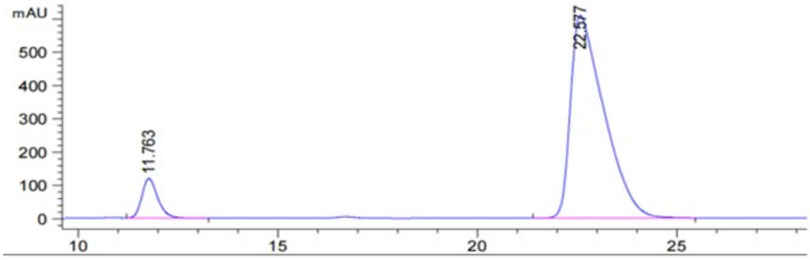
4a'



Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.494	2.98603e4	983.30377	48.7963
2		22.722	3.13335e4	556.94550	51.2037
Total			6.11939e4	1540.24927	100.0000

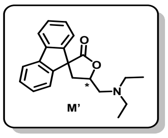
Chiral

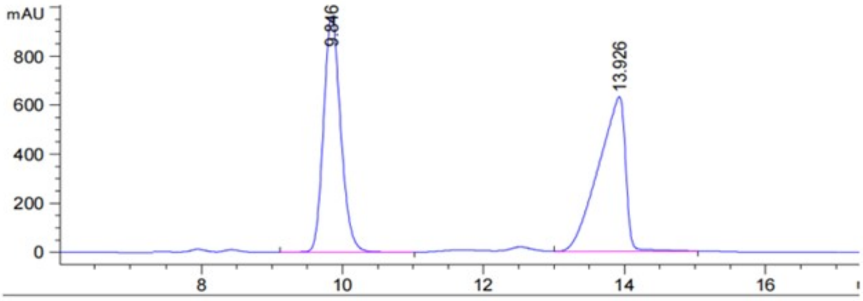


Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		11.763	3287.87085	118.75880	8.4760
2		22.577	3.55027e4	608.95129	91.5240
Total			3.87906e4	727.71009	100.0000

Racemic

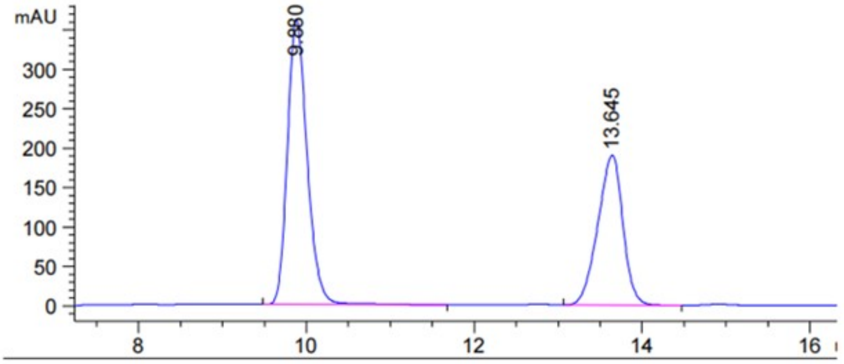




Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.846	1.62986e4	961.23199	49.1056
2		13.926	1.68923e4	630.42877	50.8944
Total			3.31908e4	1591.66077	100.0000

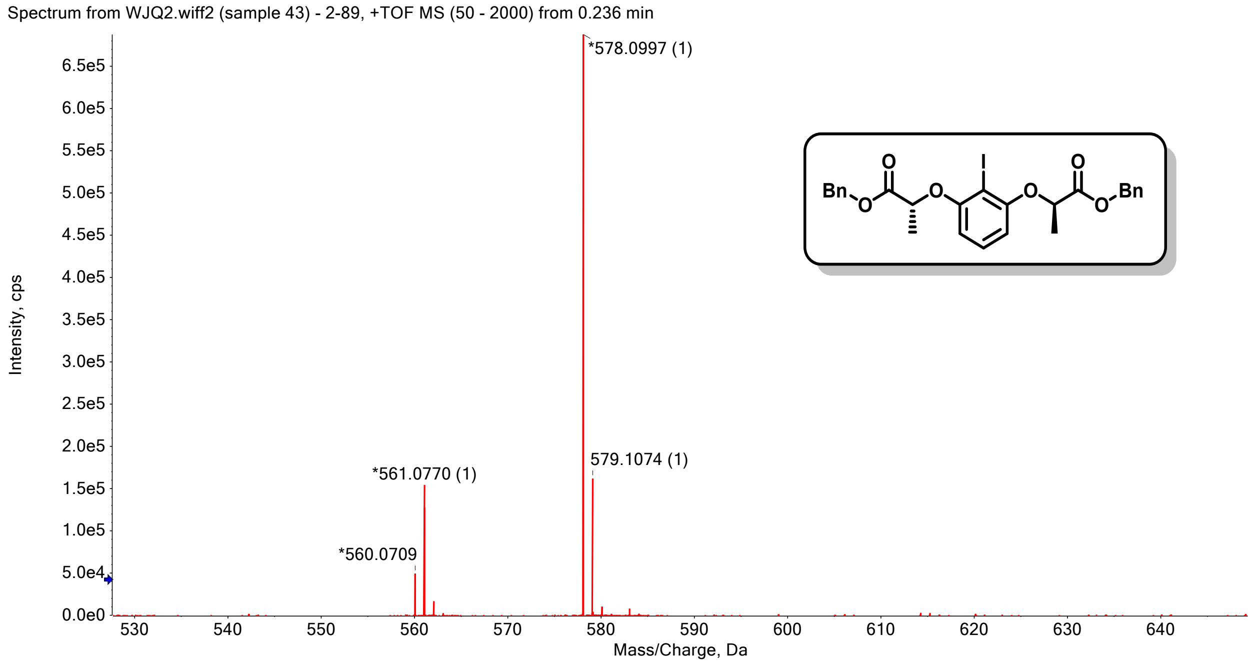
Chiral



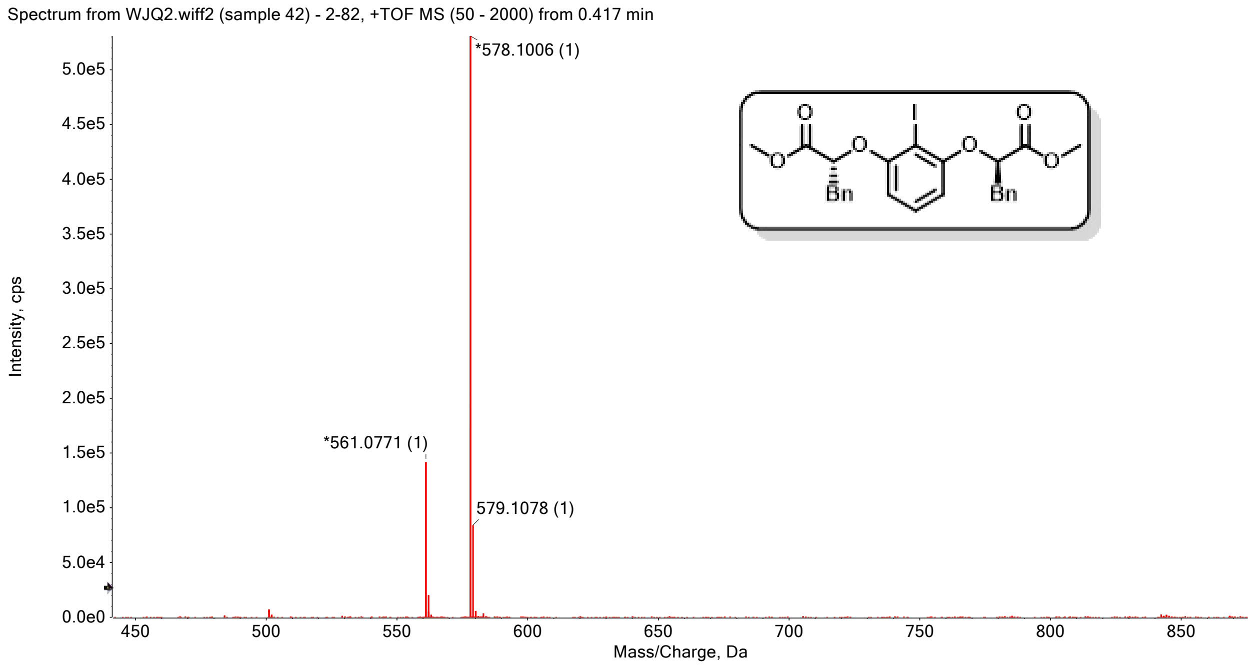
Results

Peak No.	Peak ID	Ret Time	Area	Height	Conc.
1		9.880	5898.05176	361.74747	59.9237
2		13.645	3944.54980	190.24530	40.0763
Total			9842.60156	551.99277	100.0000

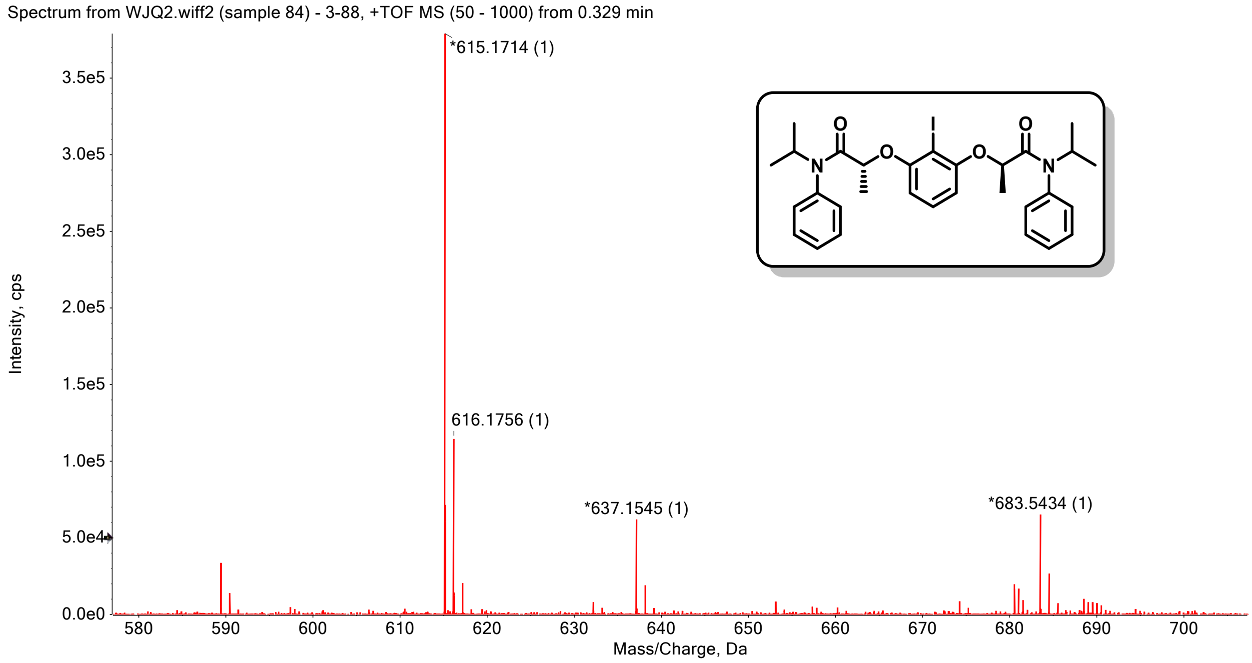
CIC 1 HRMS (ESI) *m/z*: $C_{26}H_{25}IO_6$ $[M + H]^+$ Cal: 561.0769; Found: 561.0770.



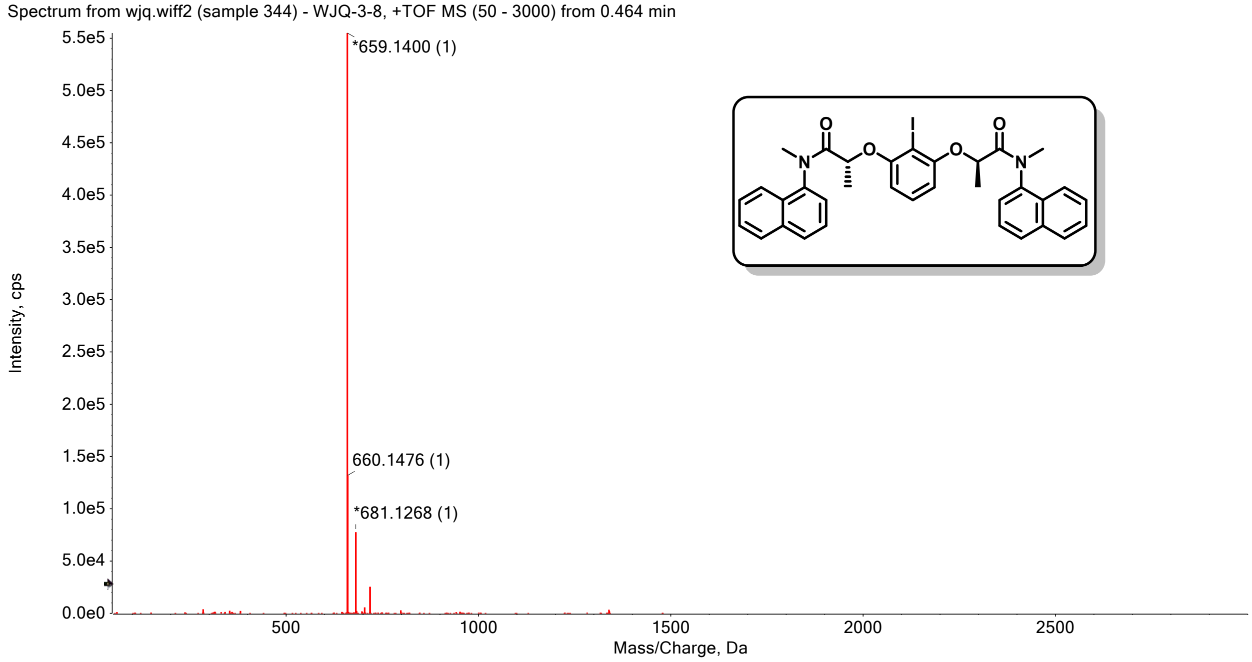
CIC 2 HRMS (ESI) *m/z*: $C_{26}H_{25}IO_6$ $[M + H]^+$ Cal: 561.0769; Found: 561.0771.



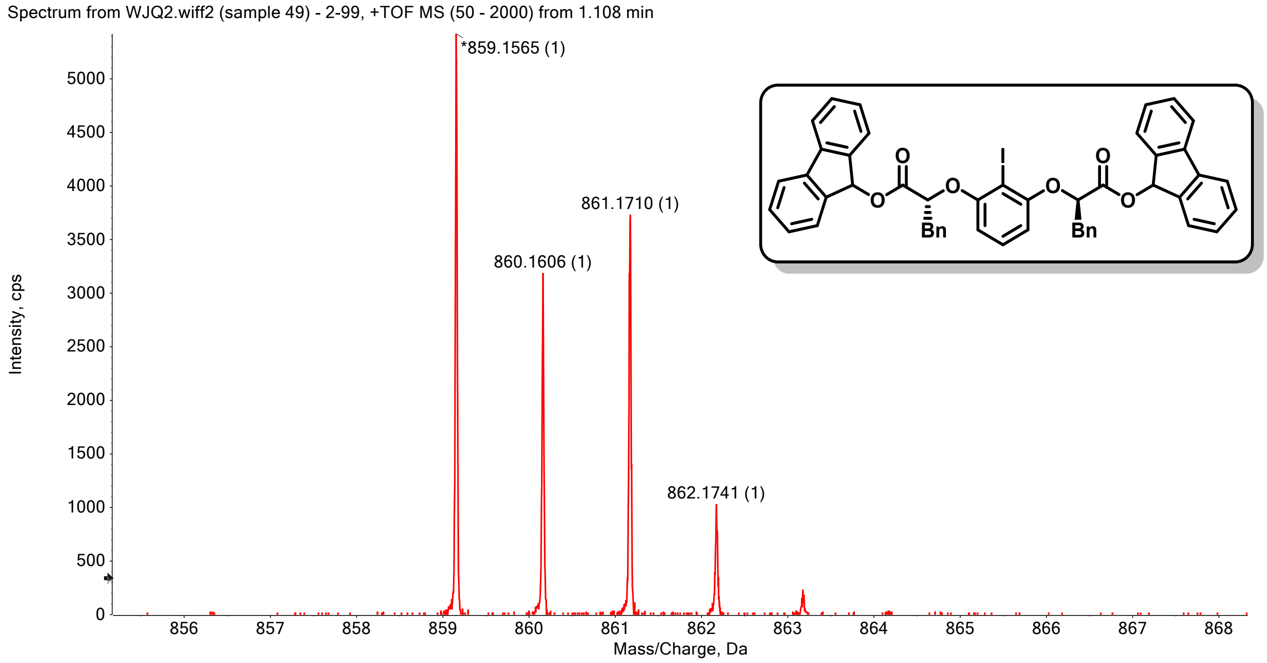
CIC 3 HRMS (ESI) *m*/*z*: $C_{30}H_{35}IN_2O_4$ $[M+H]^+$ Cal: 615.1714; Found: 615.1714.



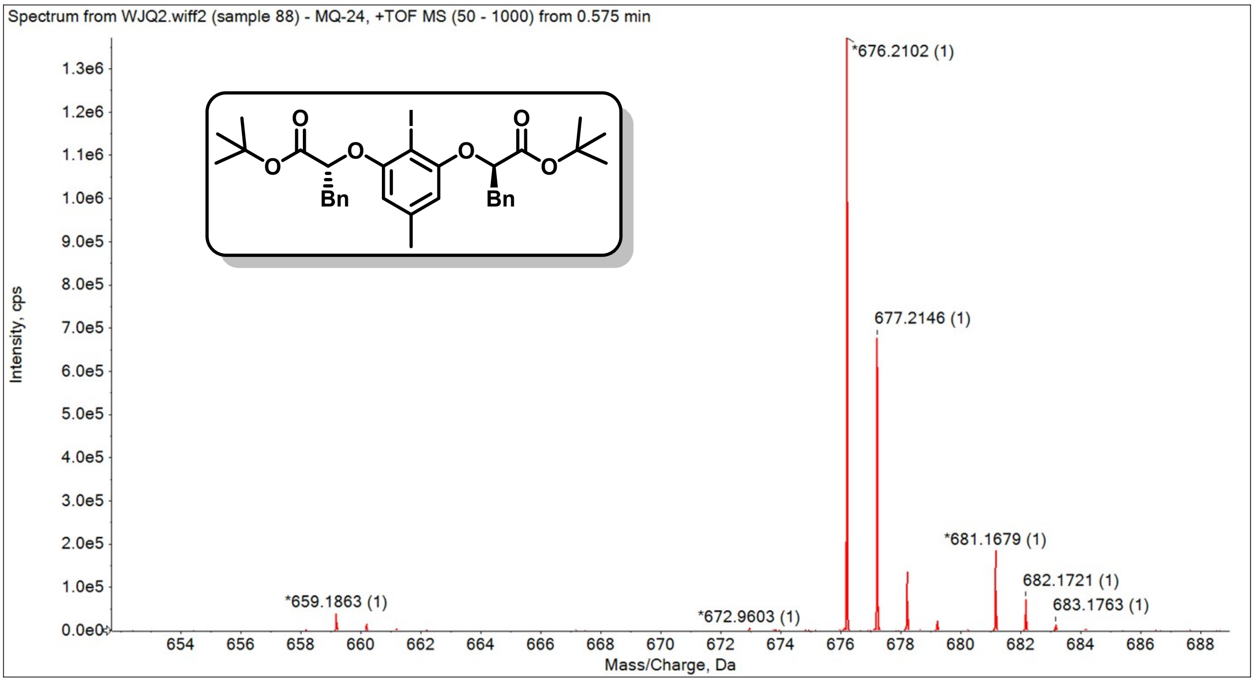
CIC 4 HRMS (ESI) *m*/*z*: $C_{34}H_{31}IN_2O_4$ $[M+H]^+$ Cal: 659.1401; Found: 659.1400.



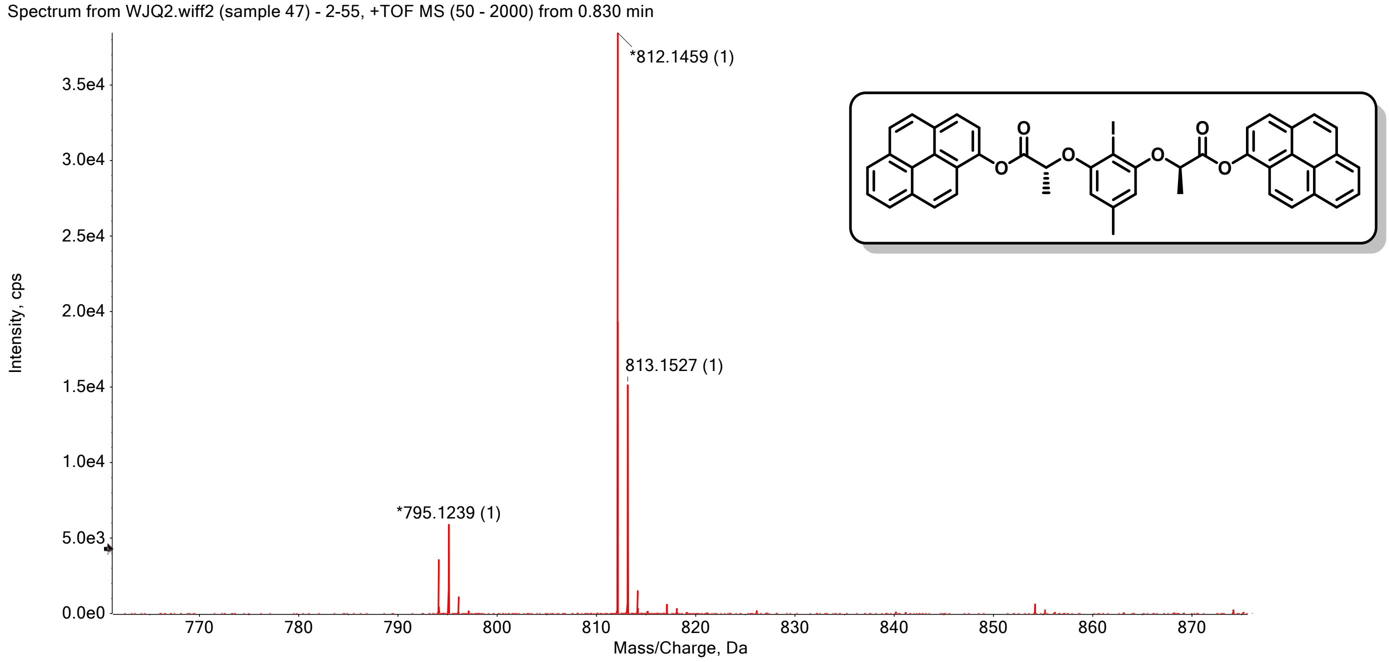
CIC 5 HRMS (ESI) *m/z*: $C_{50}H_{37}IO_6$ $[M + H]^+$ Cal: 861.1708; Found: 861.1710.



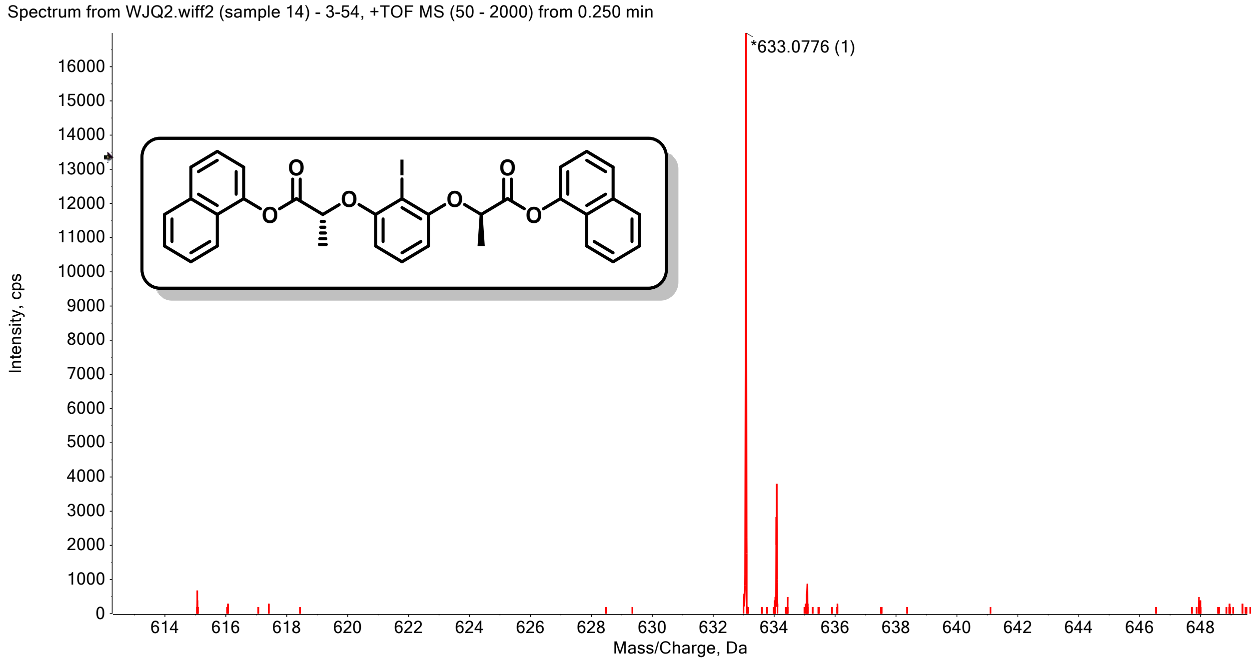
CIC 6 HRMS (ESI) *m/z*: $C_{33}H_{39}IO_6$ $[M + H]^+$ Cal: 659.1864; Found: 659.1863.



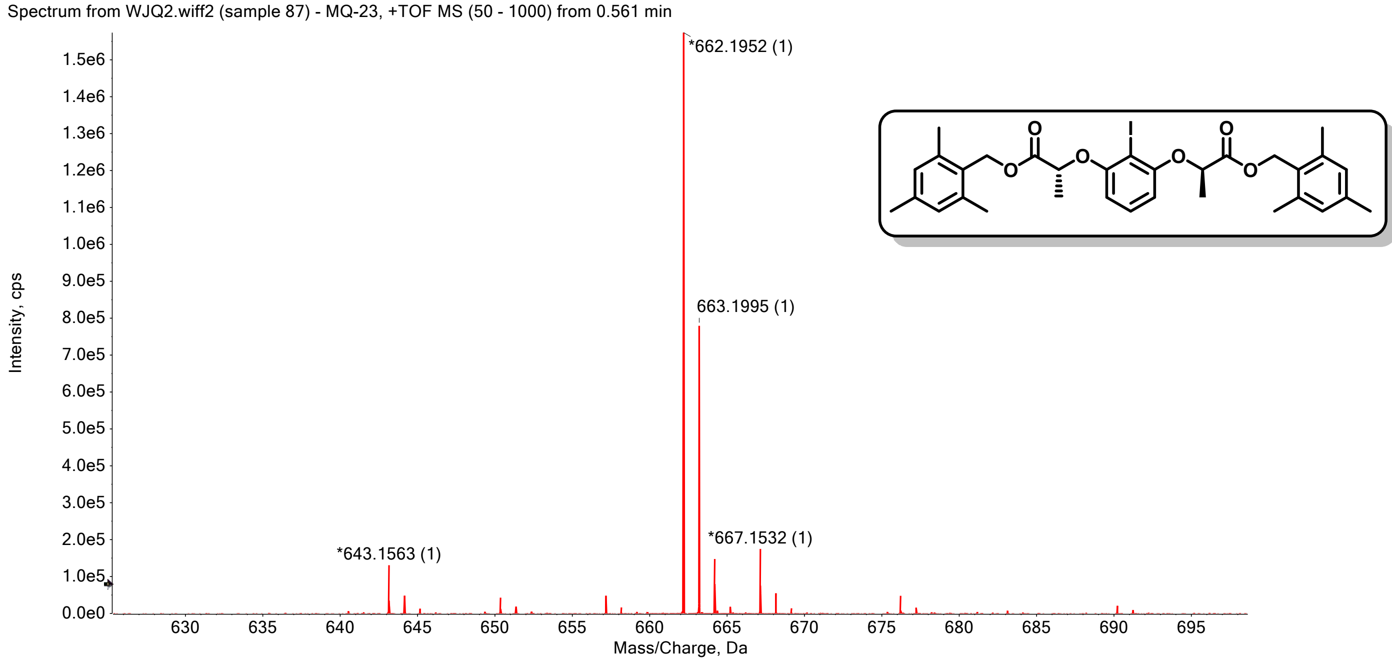
CIC 7 HRMS (ESI) *m/z*: $C_{45}H_{31}IO_6$ $[M + H]^+$ Cal: 795.1238; Found: 795.1239.



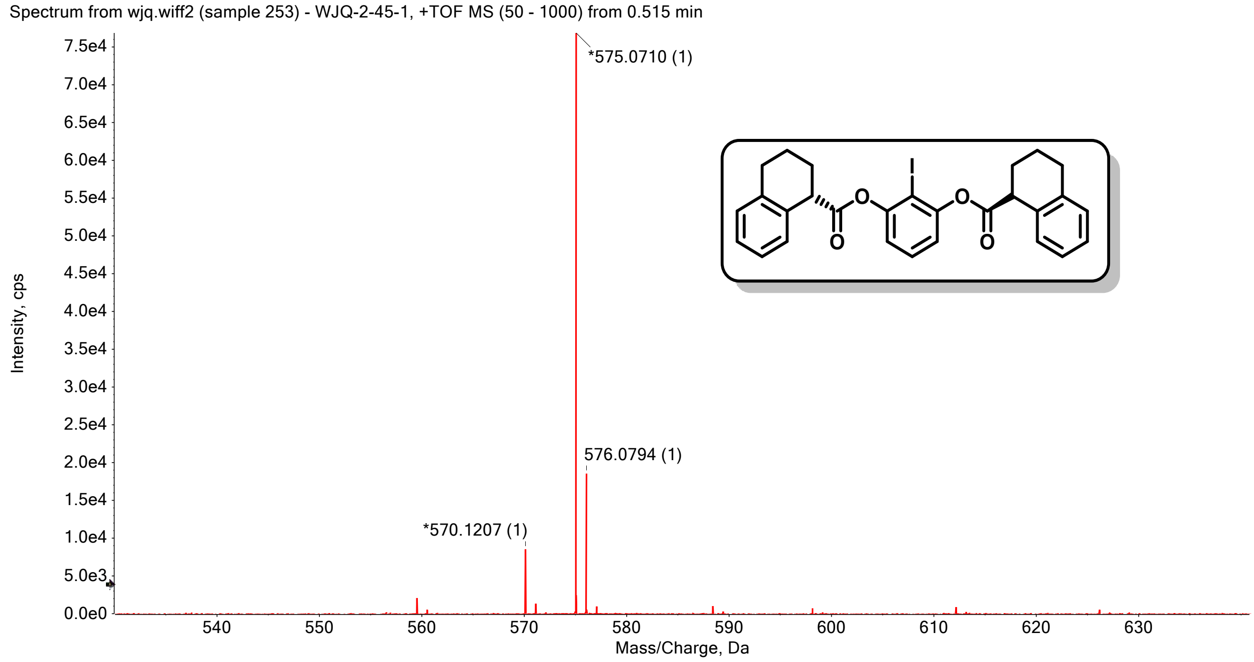
CIC 8 HRMS (ESI) *m/z*: $C_{32}H_{25}IO_6$ $[M + H]^+$ Cal: 633.0769; Found: 633.0776.



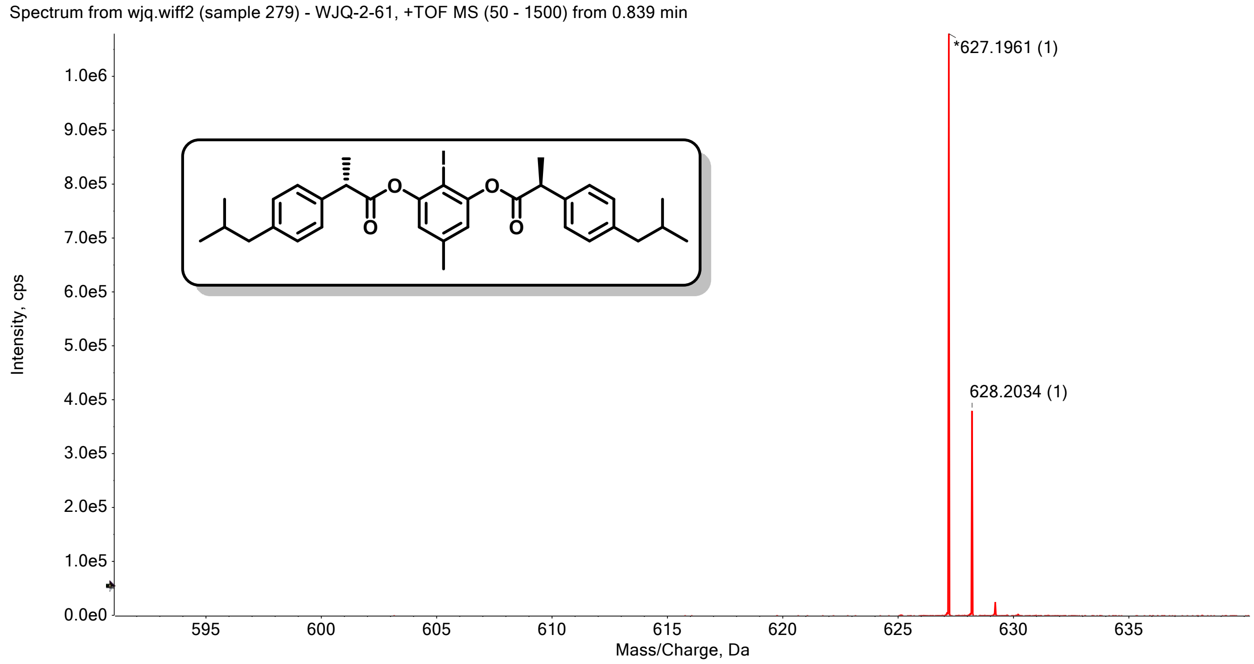
CIC 9 HRMS (ESI) *m/z*: $C_{32}H_{37}IO_6$ [M + Na]$^+$ Cal: 667.1527; Found: 667.1532.



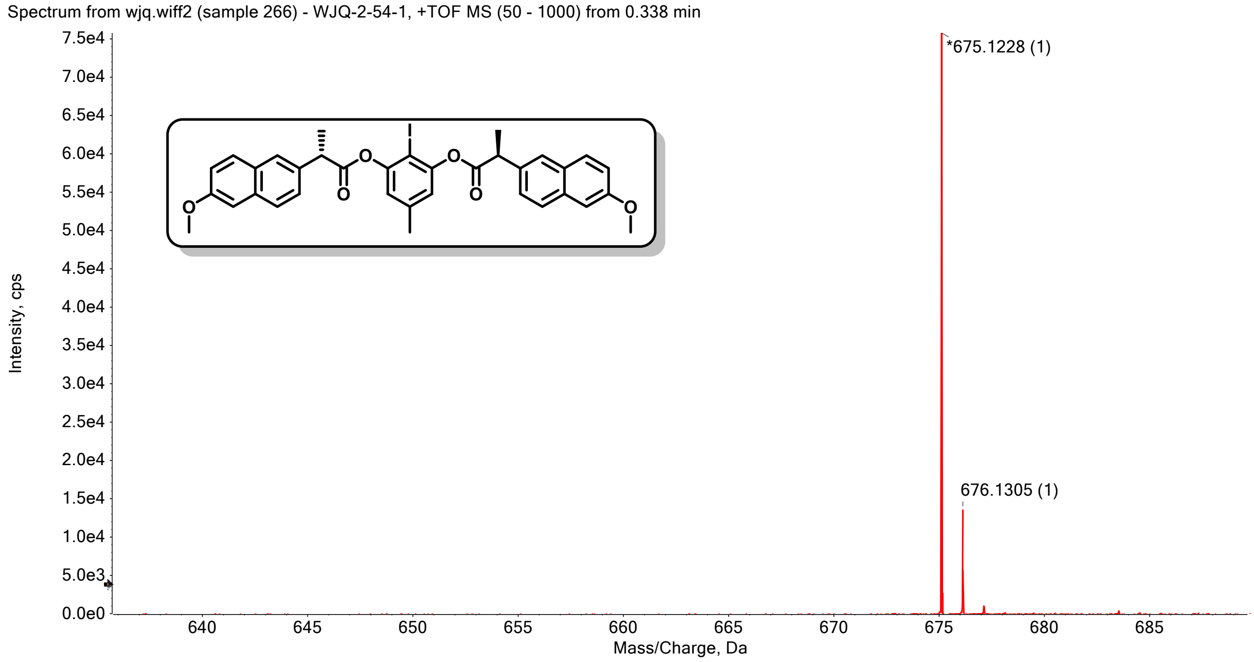
CIC 10 HRMS (ESI) *m/z*: $C_{28}H_{25}IO_4$ [M + Na]$^+$ Cal: 575.0690; Found: 575.0710.



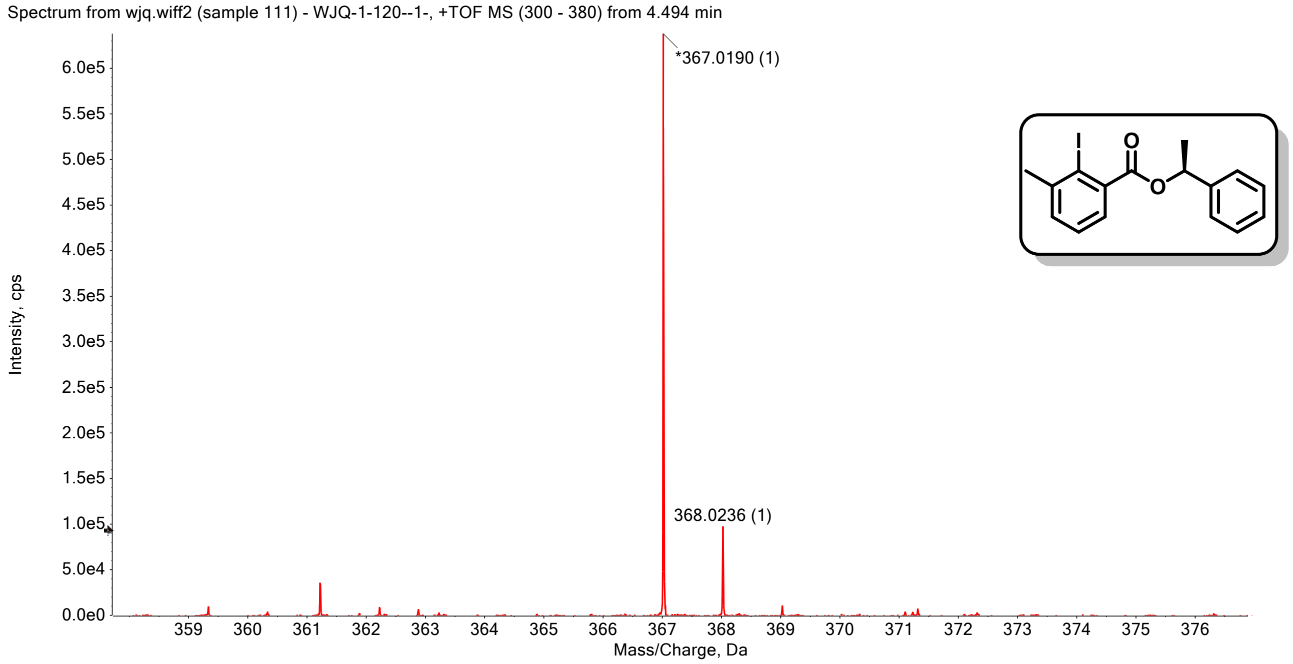
CIC 11 HRMS (ESI) *m/z*: $C_{33}H_{39}IO_4$ $[M + H]^+$ Cal: 627.1966; Found: 627.1961.



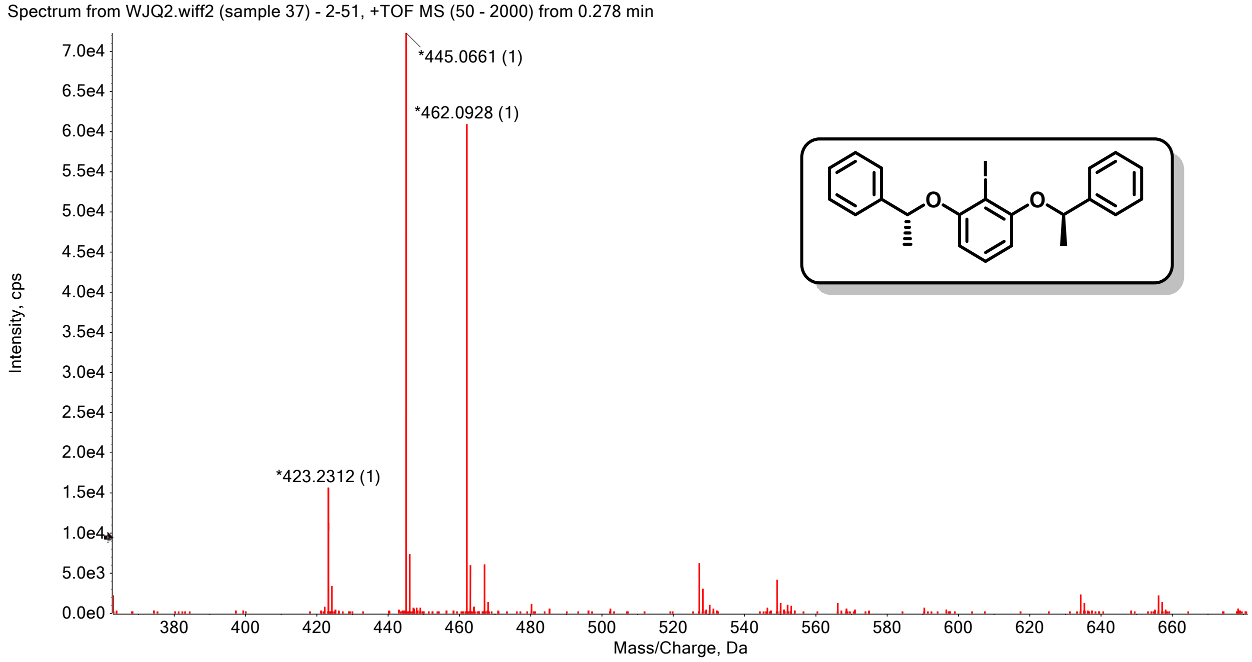
CIC 12 HRMS (ESI) *m/z*: $C_{35}H_{31}IO_6$ $[M + H]^+$ Cal: 675.1238; Found: 675.1228.



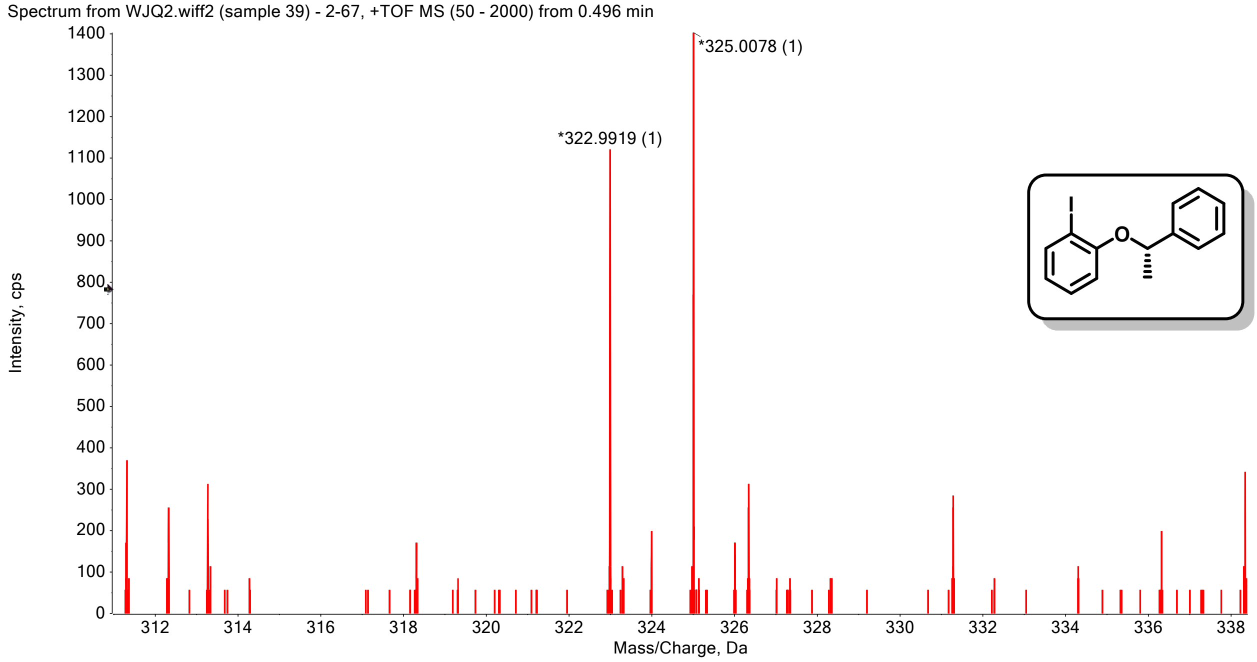
CIC 13 HRMS (ESI) *m/z*: $C_{16}H_{15}IO_2$ [M + H]$^+$ Cal: 367.0189; Found: 367.0190.



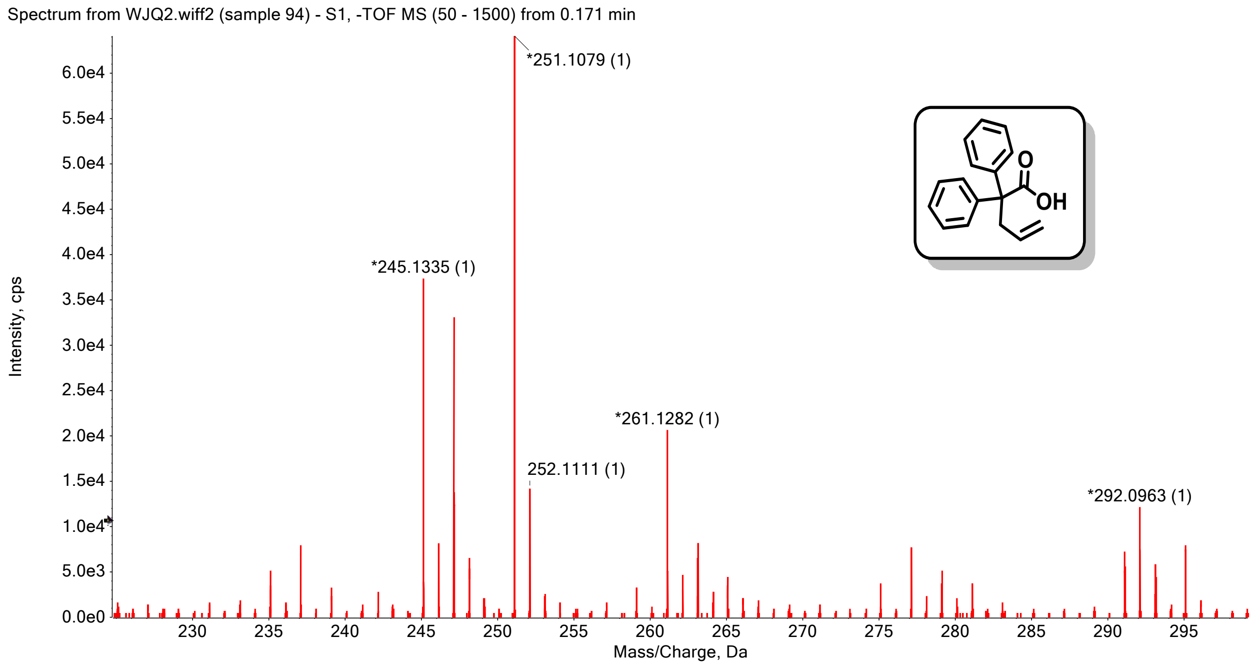
CIC 14 HRMS (ESI) *m/z*: $C_{22}H_{21}IO_2$ [M + H]$^+$ Cal: 445.0659; Found: 445.0661.



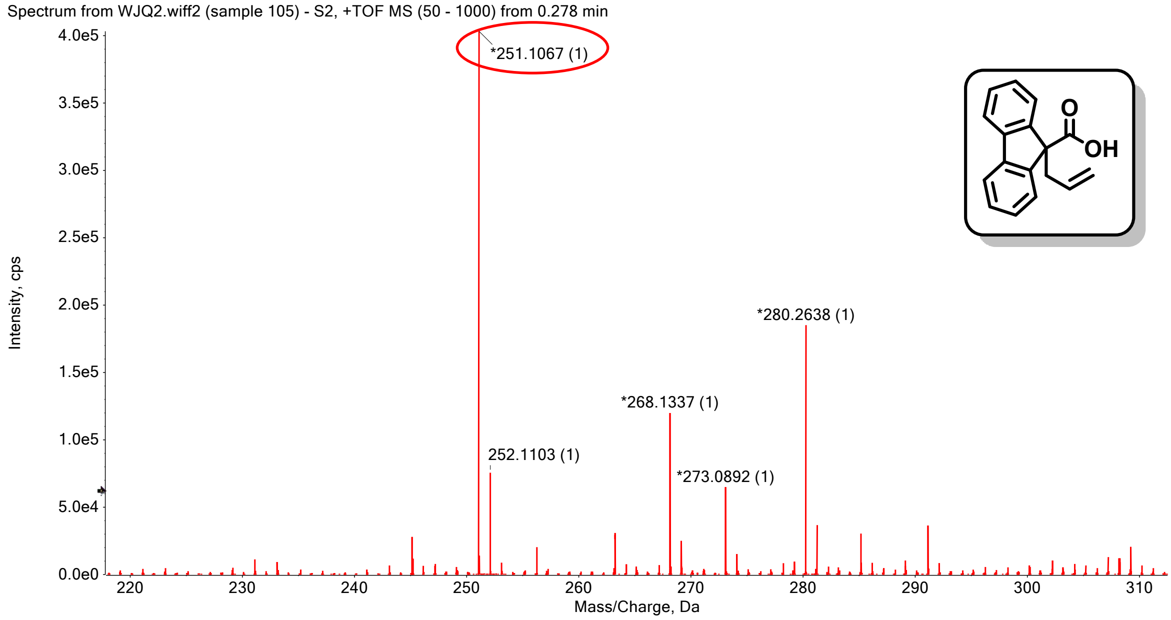
CIC 15 HRMS (ESI) *m/z*: $C_{14}H_{13}IO$ [M + H]$^+$ Cal: 325.0084; Found: 325.0078.



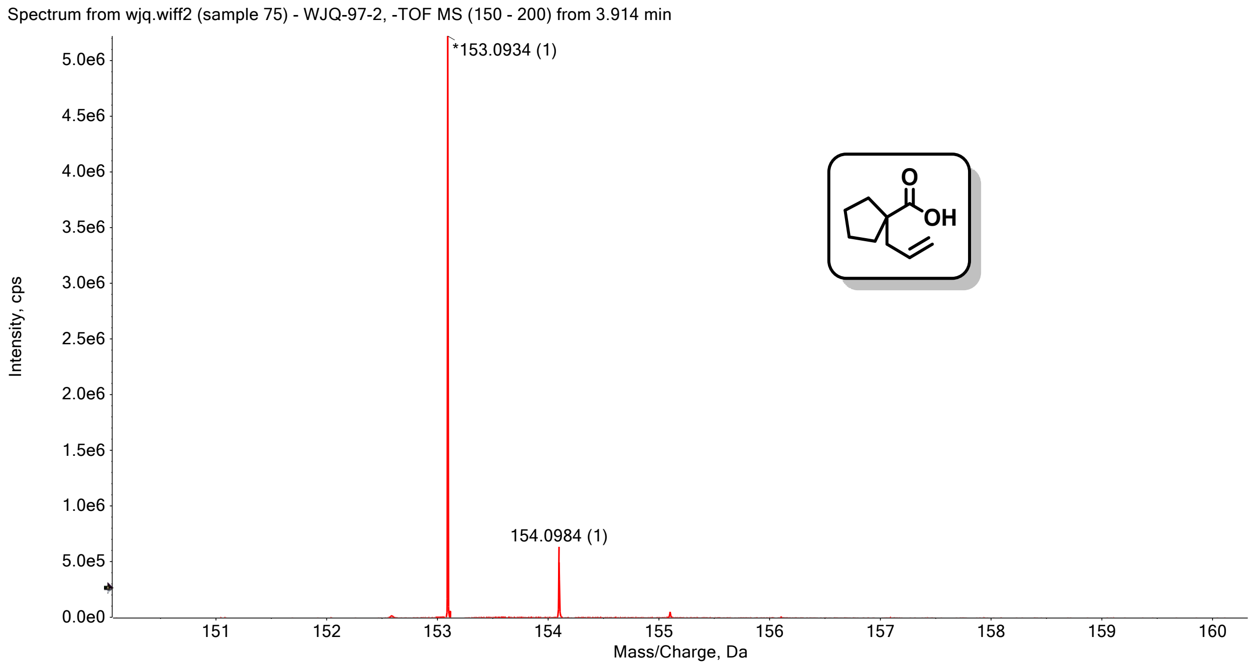
3a HRMS (ESI) *m/z*: $C_{17}H_{16}O_2$ [M−H]$^-$ Cal: 251.1078; Found: 251.1079.



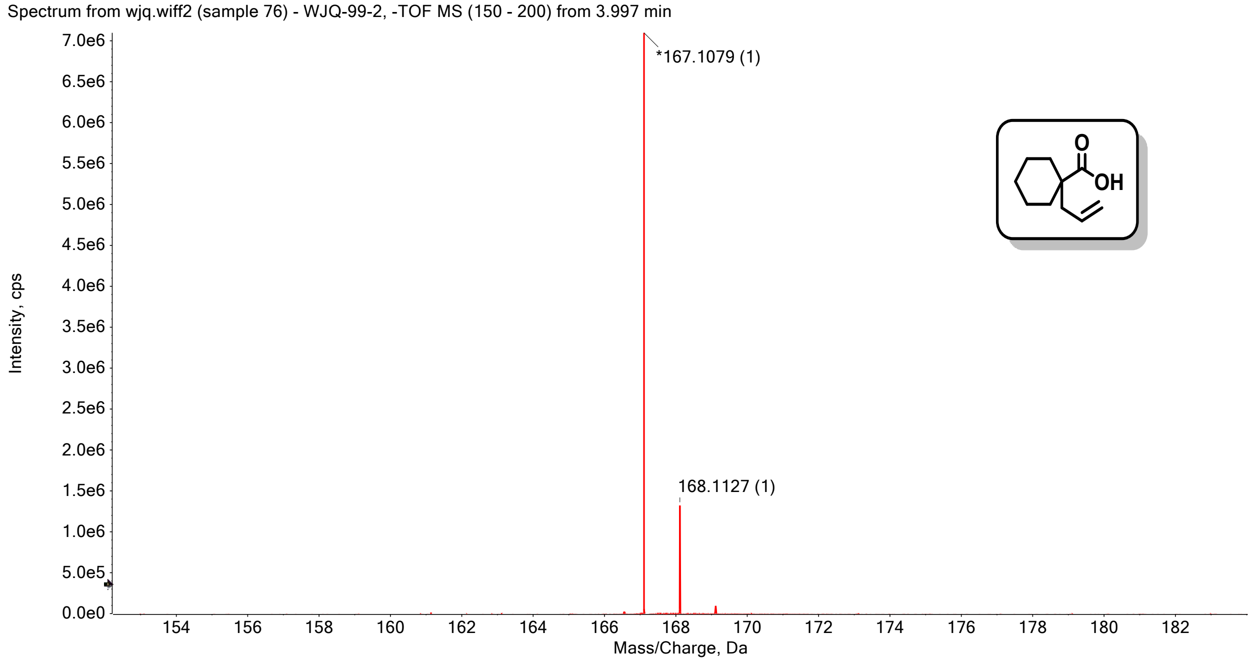
3b HRMS (ESI) *m*/*z*: $C_{17}H_{14}O_2$ $[M+H]^+$ Cal: 251.1067; Found: 251.1067.



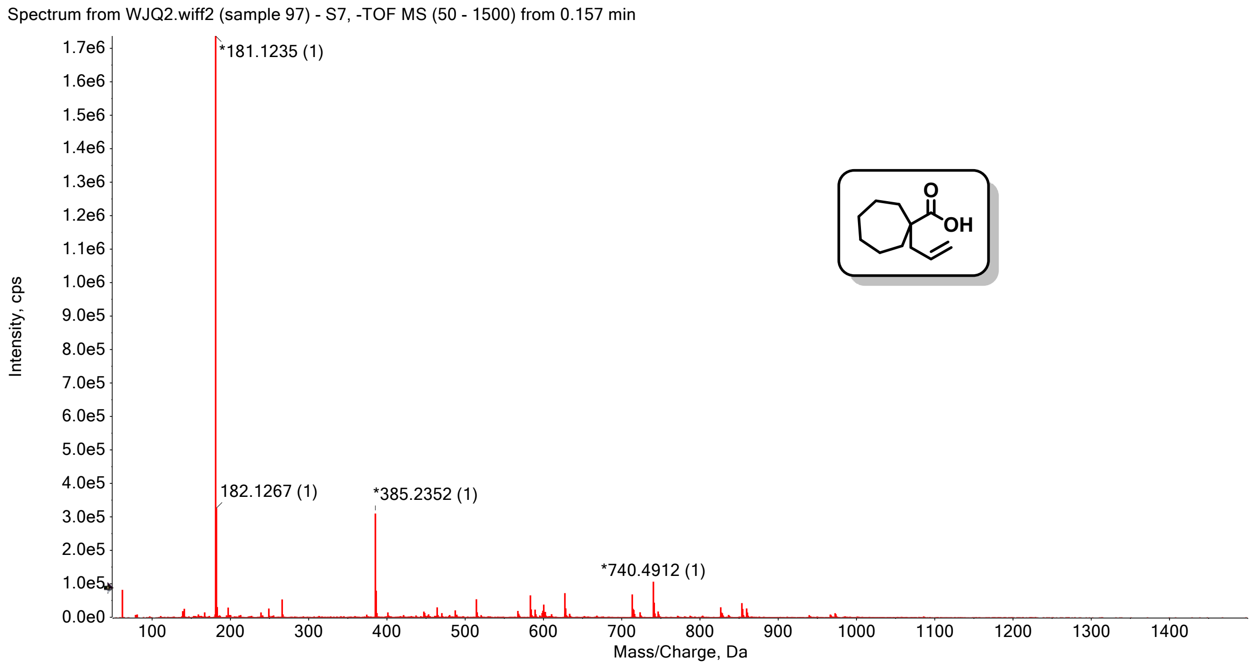
3c HRMS (ESI) *m*/*z*: $C_9H_{14}O_2$ $[M-H]^-$ Cal: 153.0921; Found: 153.0934.



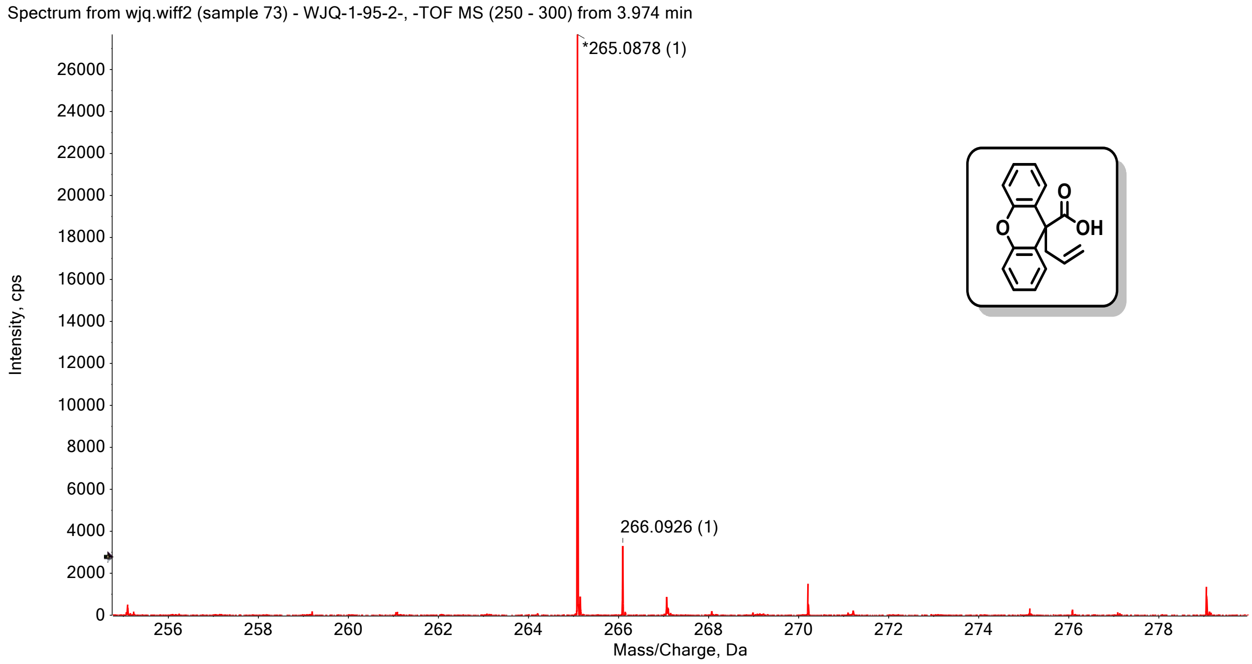
3d HRMS (ESI) *m/z*: $C_{10}H_{16}O_2$ [M—H]$^-$ Cal: 167.1078; Found: 167.1079.



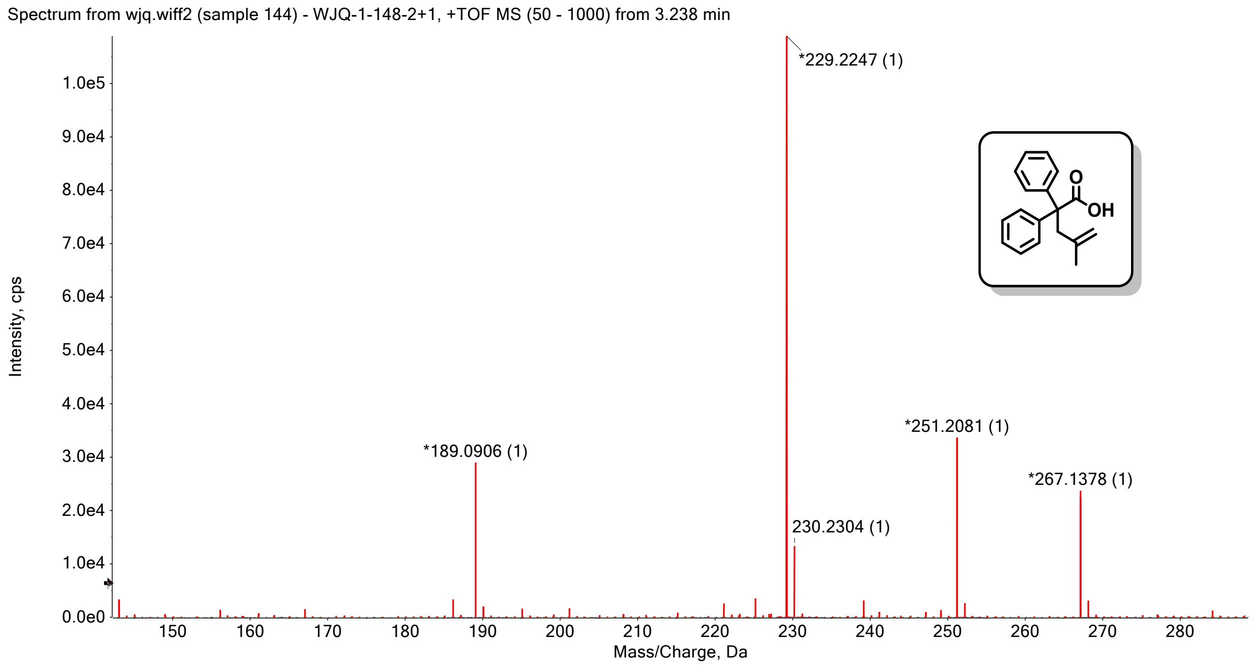
3e HRMS (ESI) *m/z*: $C_{11}H_{18}O_2$ [M—H]$^-$ Cal: 181.1234; Found: 181.1235.



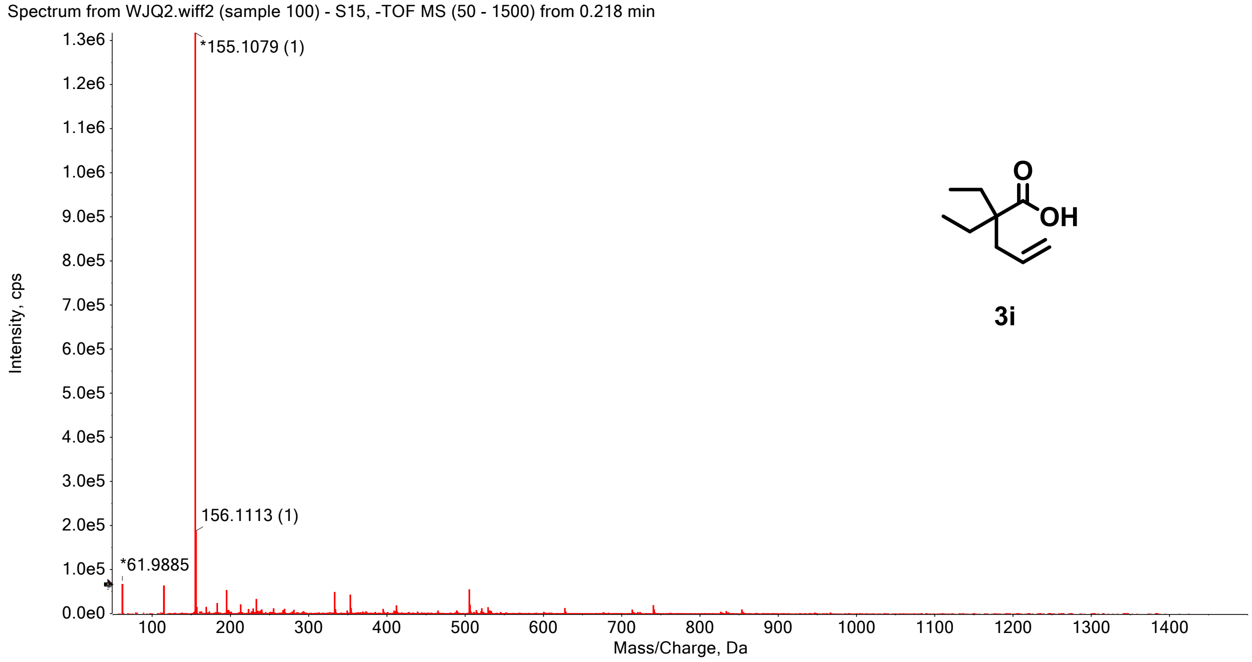
3f HRMS (ESI) *m/z*: $C_{17}H_{14}O_3$ $[M-H]^-$ Cal: 265.0870; Found: 265.0878.



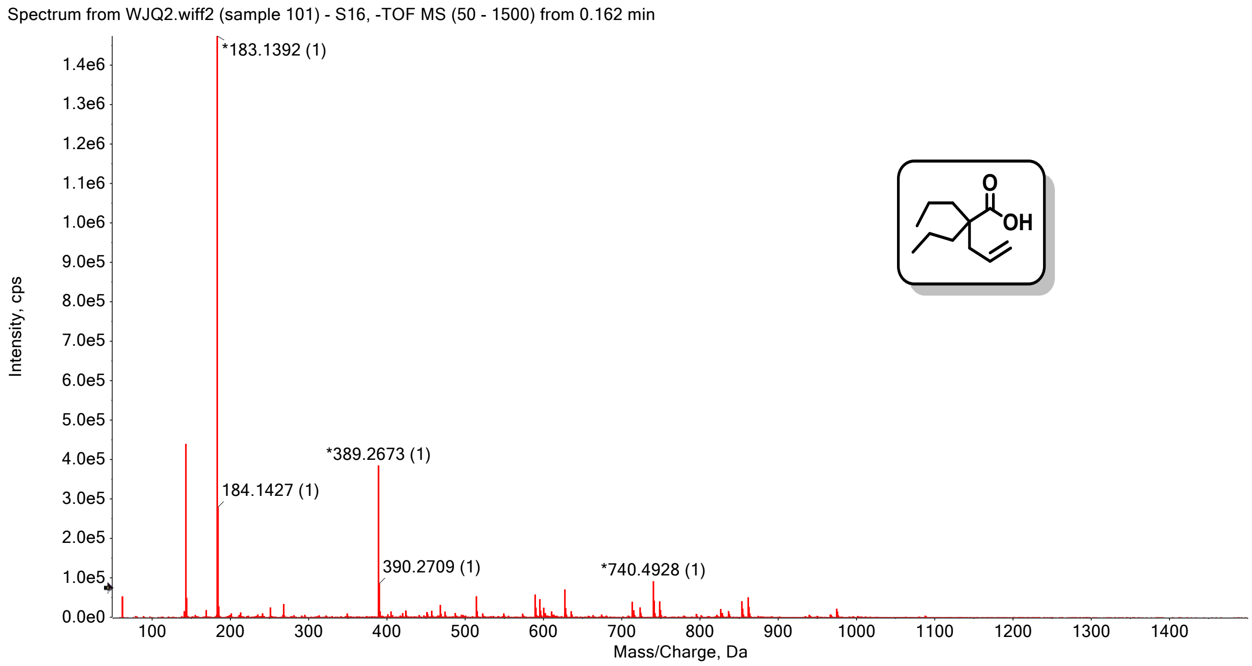
3g HRMS (ESI) *m/z*: $C_{18}H_{18}O_2$ $[M + H]^+$ Cal: 267.1380; Found: 263.1378.



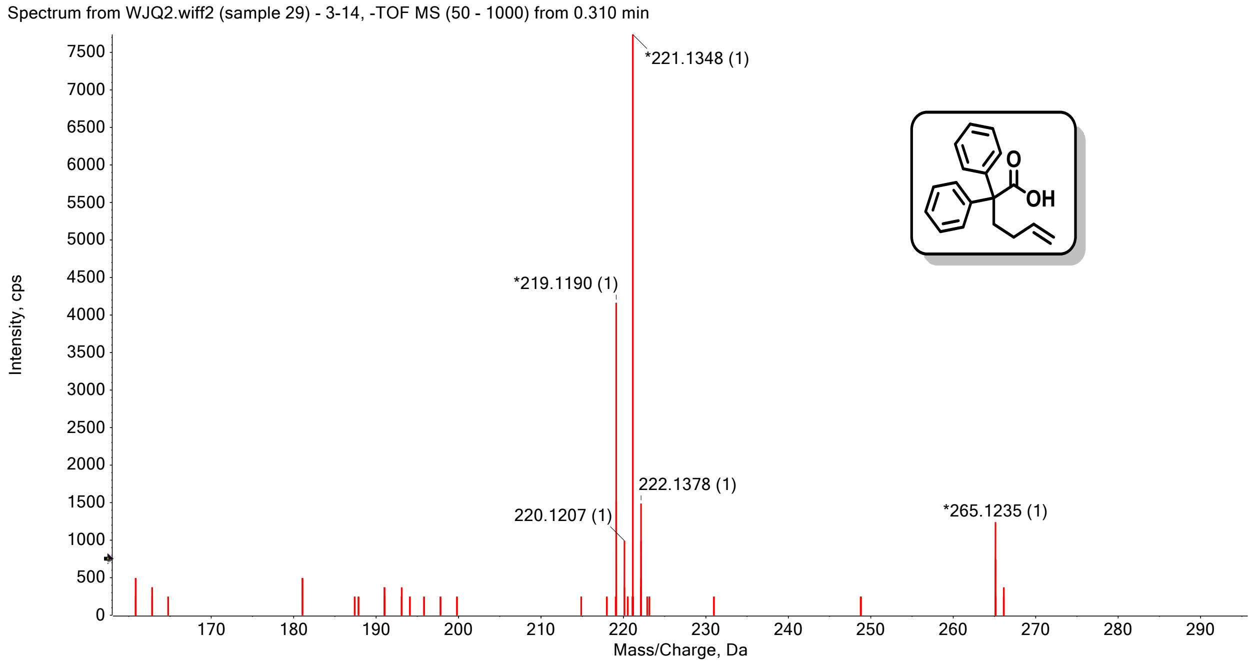
3i HRMS (ESI) *m*/*z*: $C_9H_{16}O_2$ $[M-H]^-$ Cal: 155.1078; Found: 155.1079.



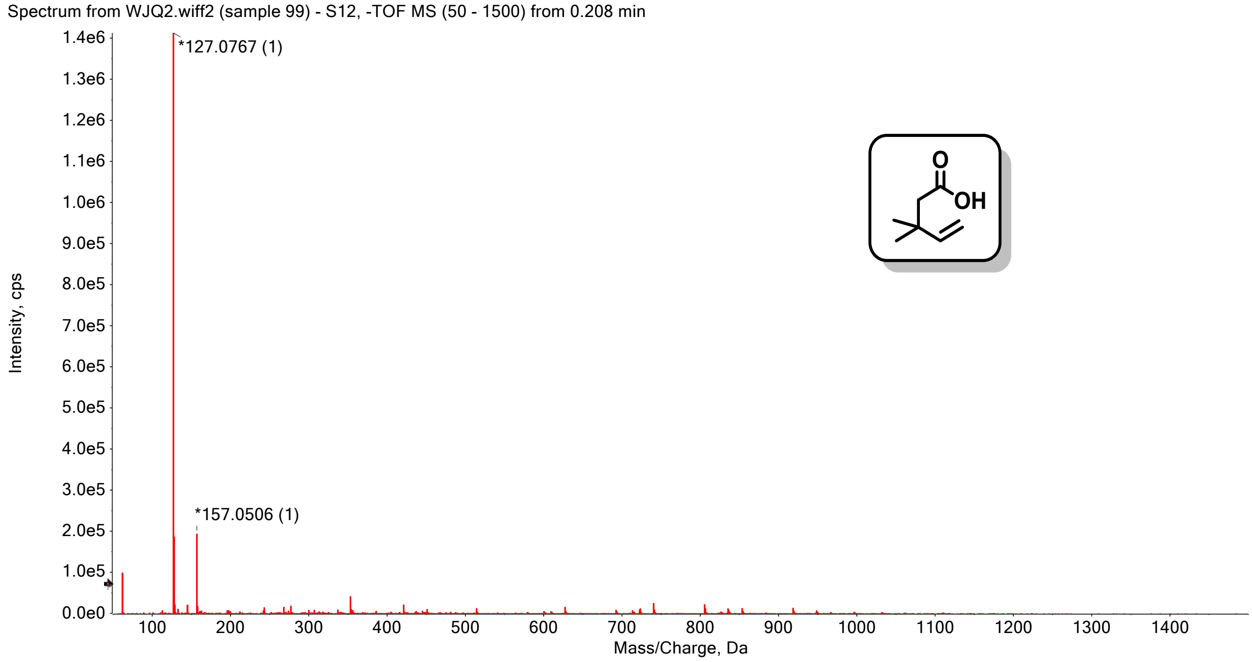
3j HRMS (ESI) *m*/*z*: $C_{11}H_{20}O_2$ $[M-H]^-$ Cal: 183.1391; Found: 183.1392.



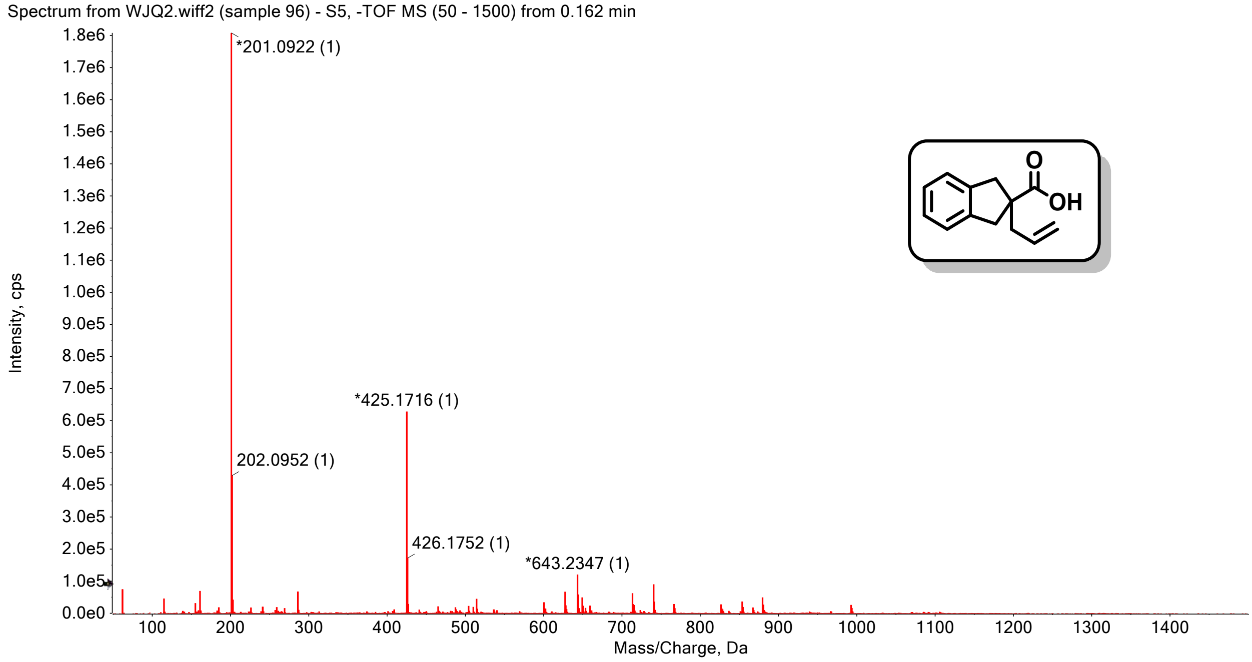
3k HRMS (ESI) *m/z*: $C_{18}H_{18}O_2$ [M−H]$^-$ Cal: 265.1234; Found: 265.1235.



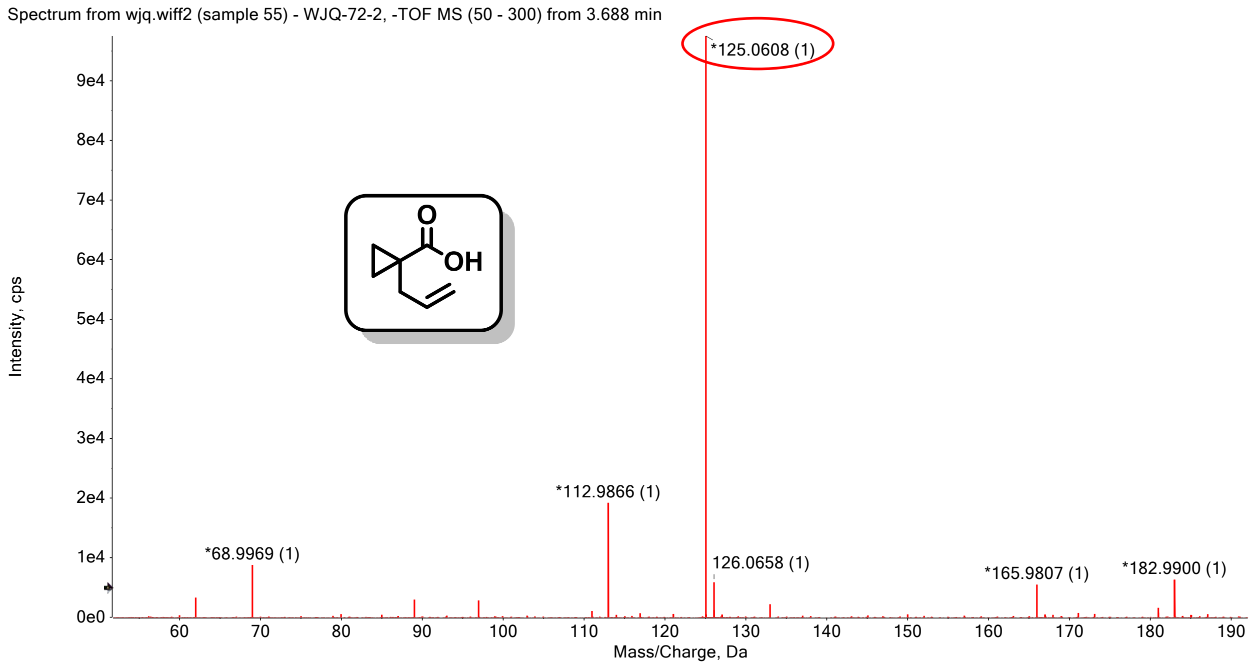
3m HRMS (ESI) *m/z*: $C_7H_{12}O_2$ [M−H]$^-$ Cal: 127.0765; Found: 127.0767.



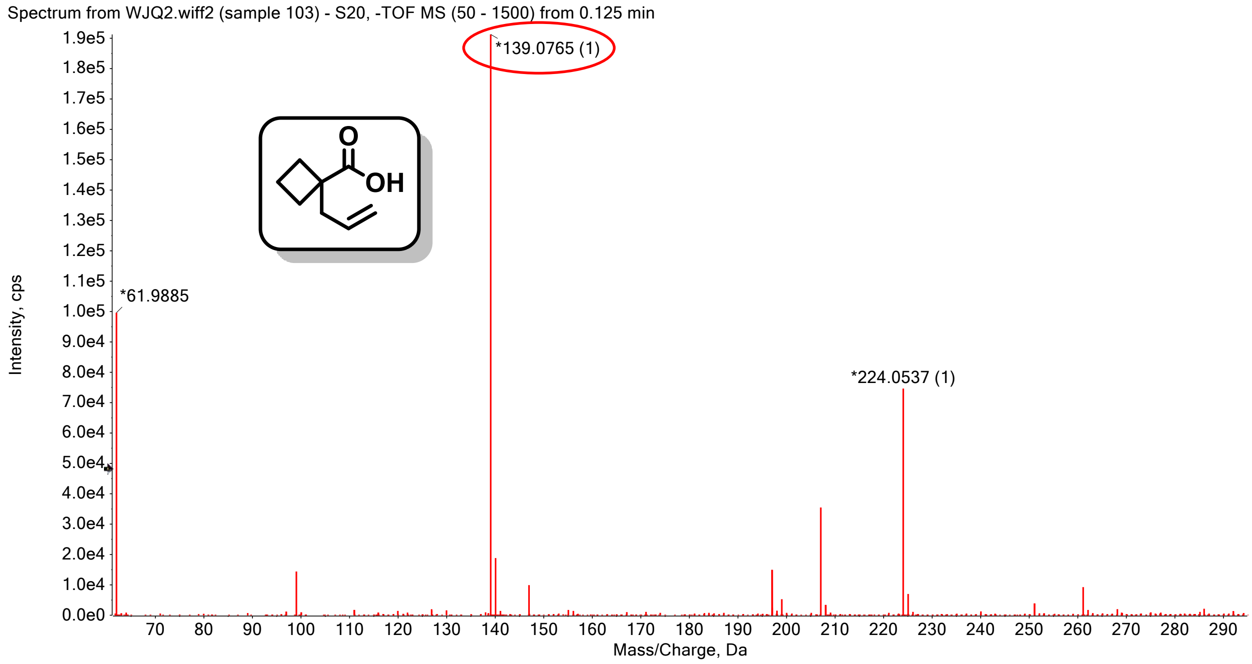
3o HRMS (ESI) *m*/*z*: $C_{13}H_{14}O_2$ $[M-H]^-$ Cal: 201.0921; Found: 201.0922.



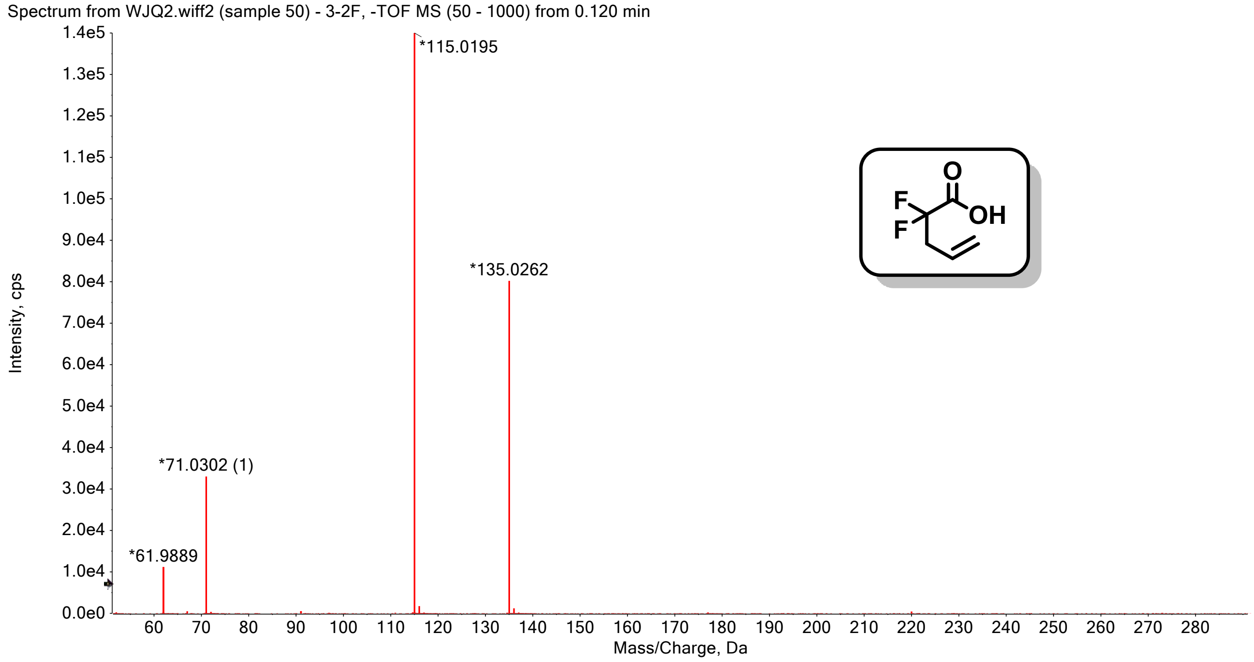
3p HRMS (ESI) *m*/*z*: $C_7H_{10}O_2$ $[M-H]^-$ Cal: 125.0608; Found: 125.0608.



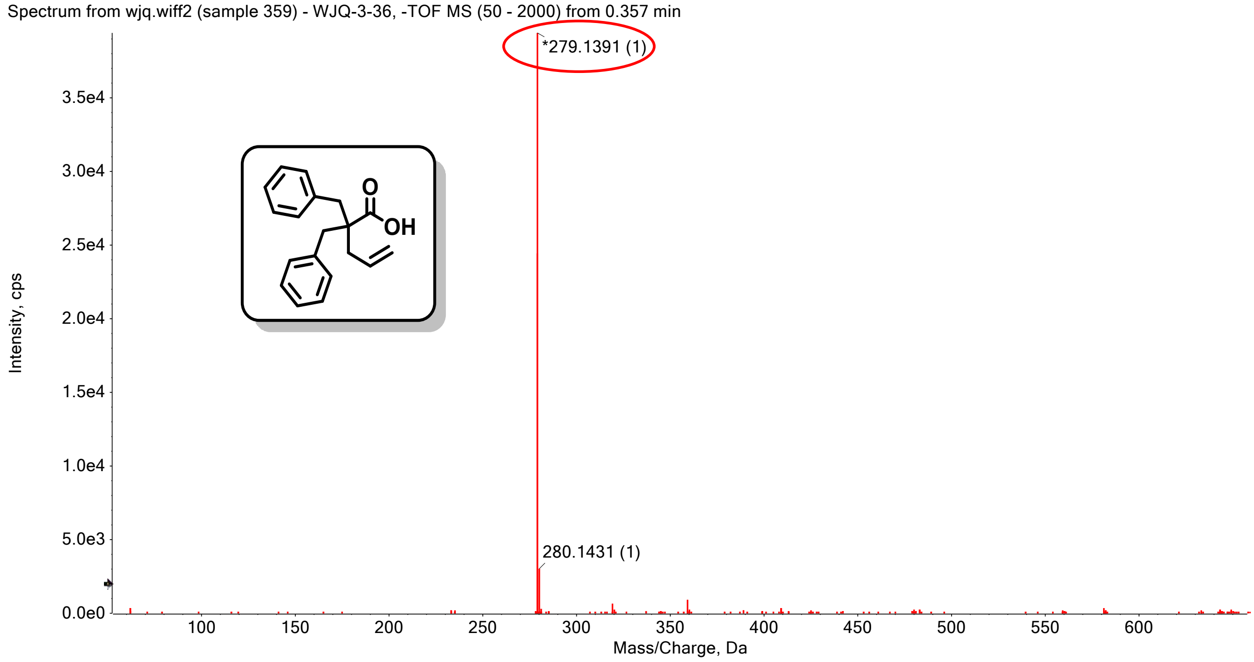
3q HRMS (ESI) *m/z*: $C_8H_{12}O_2$ $[M-H]^-$ Cal: 139.0765; Found: 139.0765.



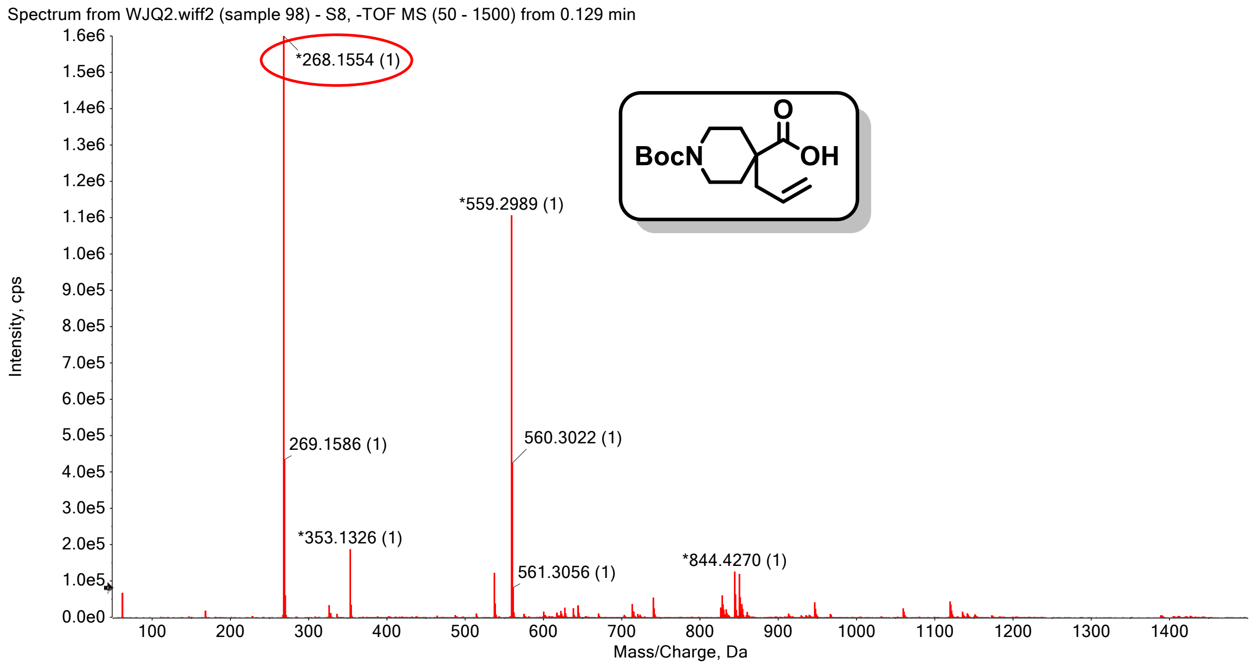
3r HRMS (ESI) *m/z*: $C_5H_6F_2O_2$ $[M-H]^-$ Cal: 135.0263; Found: 135.0262.



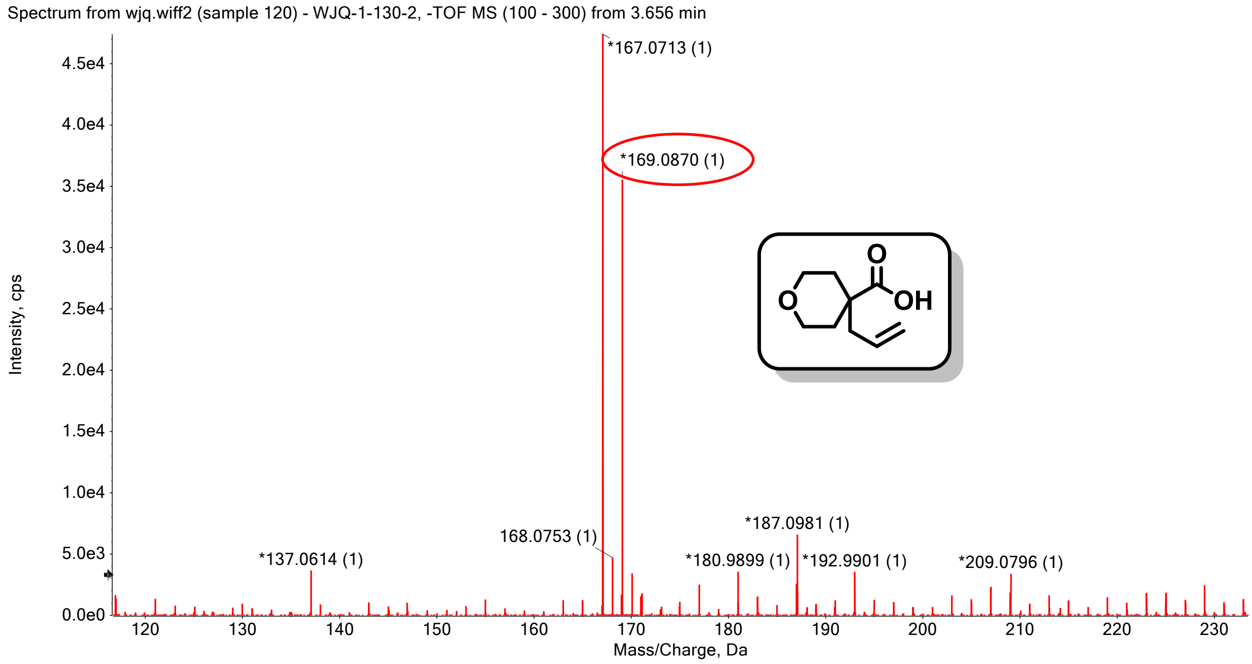
3s HRMS (ESI) *m/z*: $C_{19}H_{20}O_2$ [M−H]$^-$ Cal: 279.1391; Found: 279.1391.



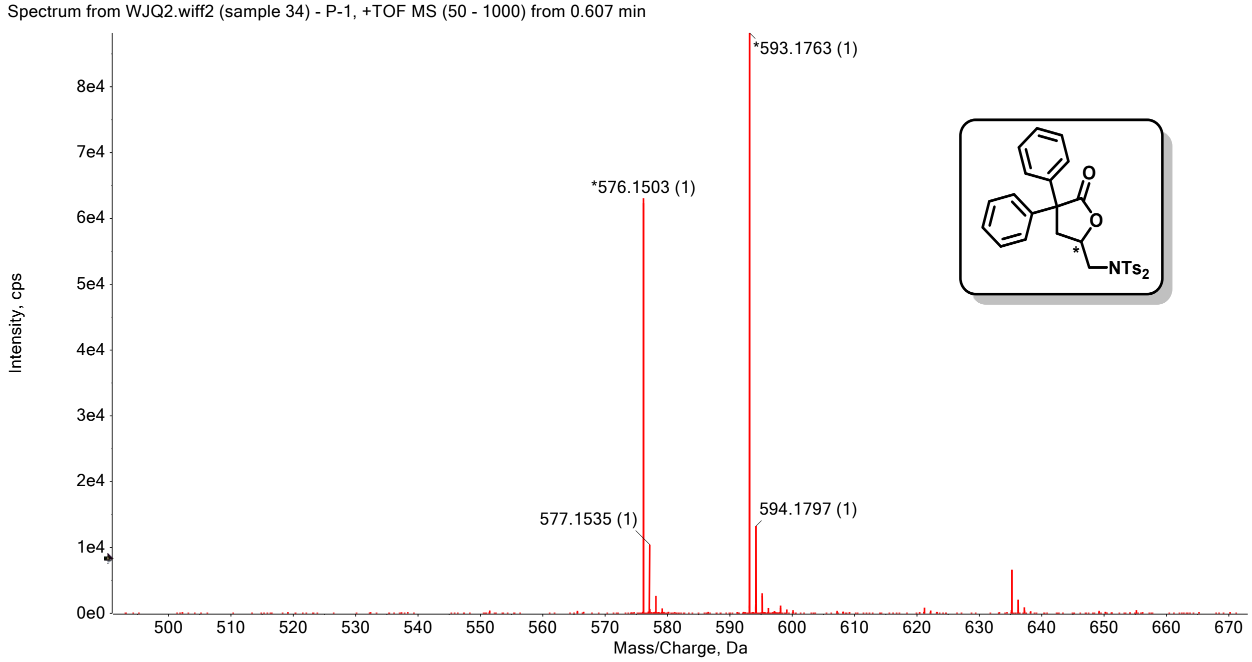
3t HRMS (ESI) *m/z*: $C_{14}H_{23}NO_4$ [M−H]$^-$ Cal: 268.1554; Found: 268.1554.



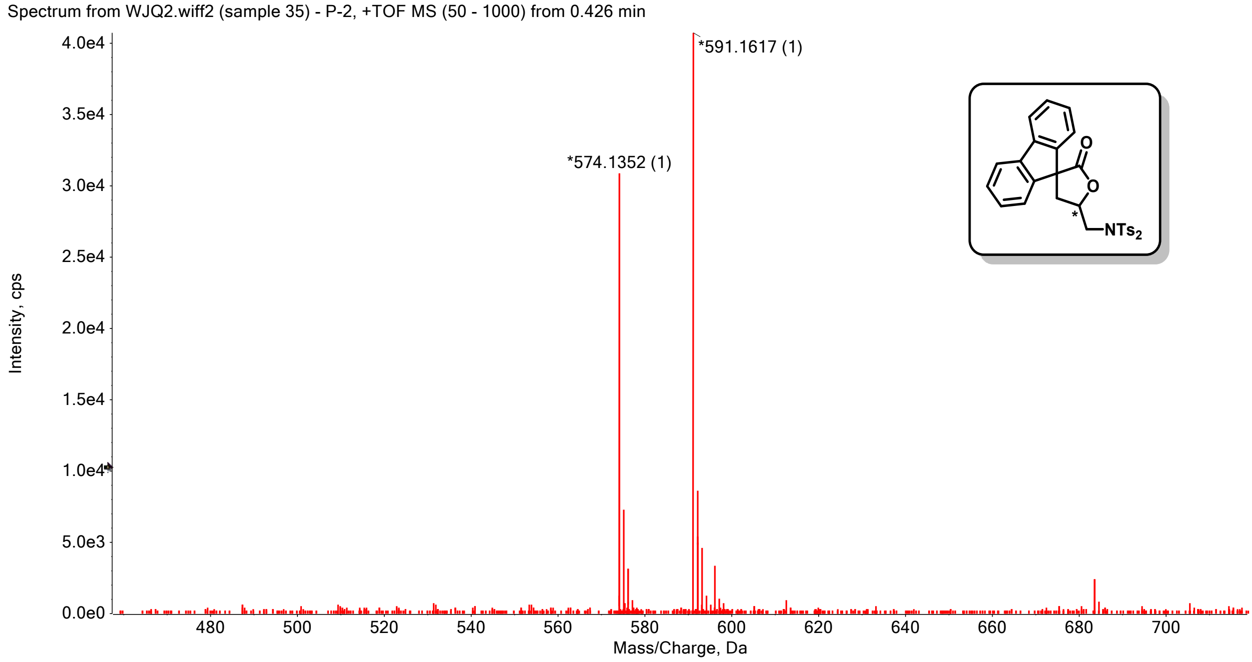
3u HRMS (ESI) *m*/*z*: $C_9H_{14}O_3$ $[M-H]^-$ Cal: 169.0870; Found: 169.0870.



4a HRMS (ESI) *m*/*z*: $C_{31}H_{29}NO_6S_2$ $[M + H]^+$ Calcd for: 576.1509; Found: 576.1503.

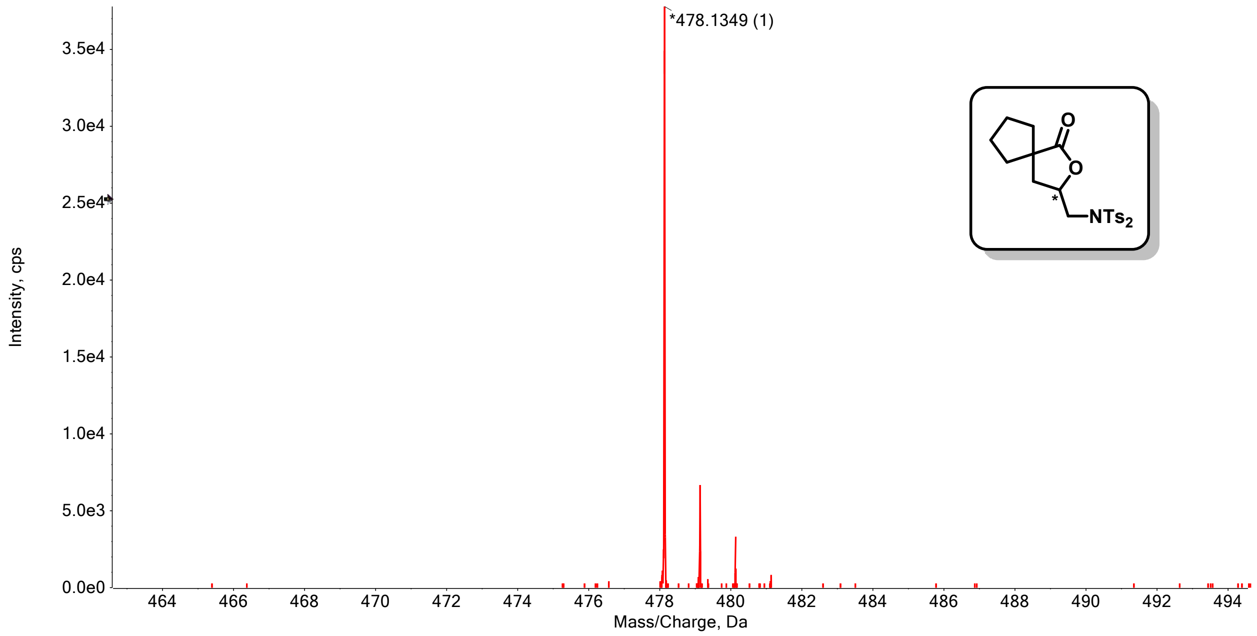


4b HRMS (ESI) *m/z*: $C_{31}H_{27}NO_6S_2$ $[M + H]^+$ Calcd for: 574.1353; Found: 574.1352.



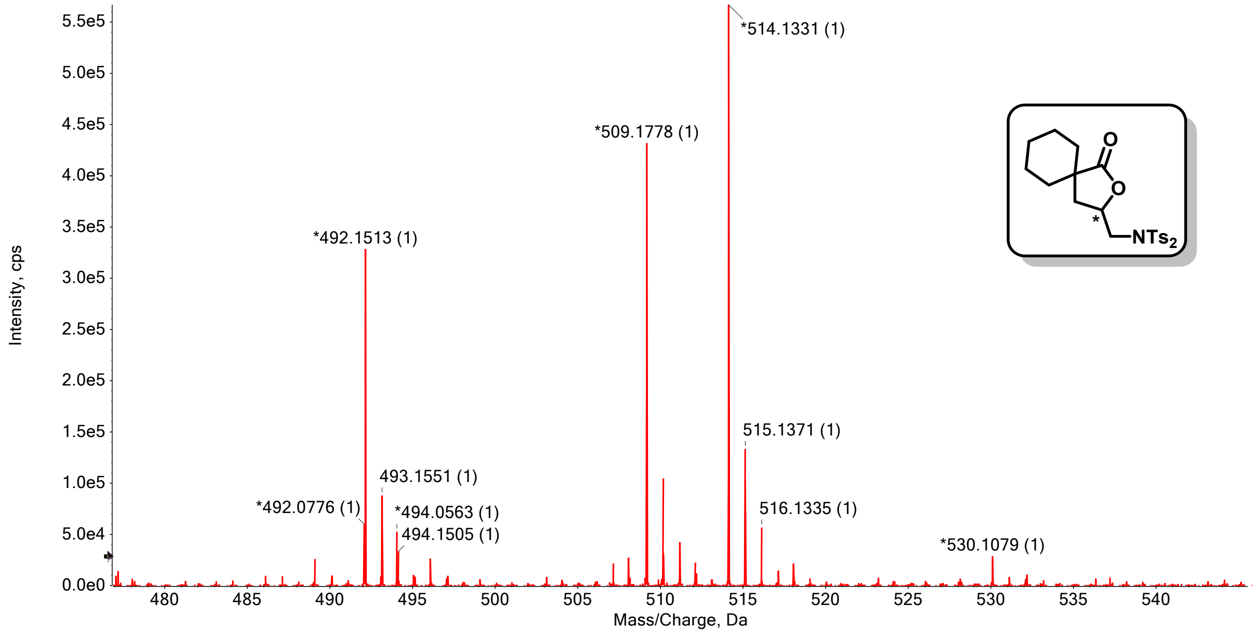
4c HRMS (ESI) *m/z*: $C_{23}H_{27}NO_6S_2$ $[M + H]^+$ Calcd for: 478.1353; Found: 478.1354.

Spectrum from WJQ2.wiff2 (sample 28) - 3-62, +TOF MS (50 - 1000) from 0.454 min

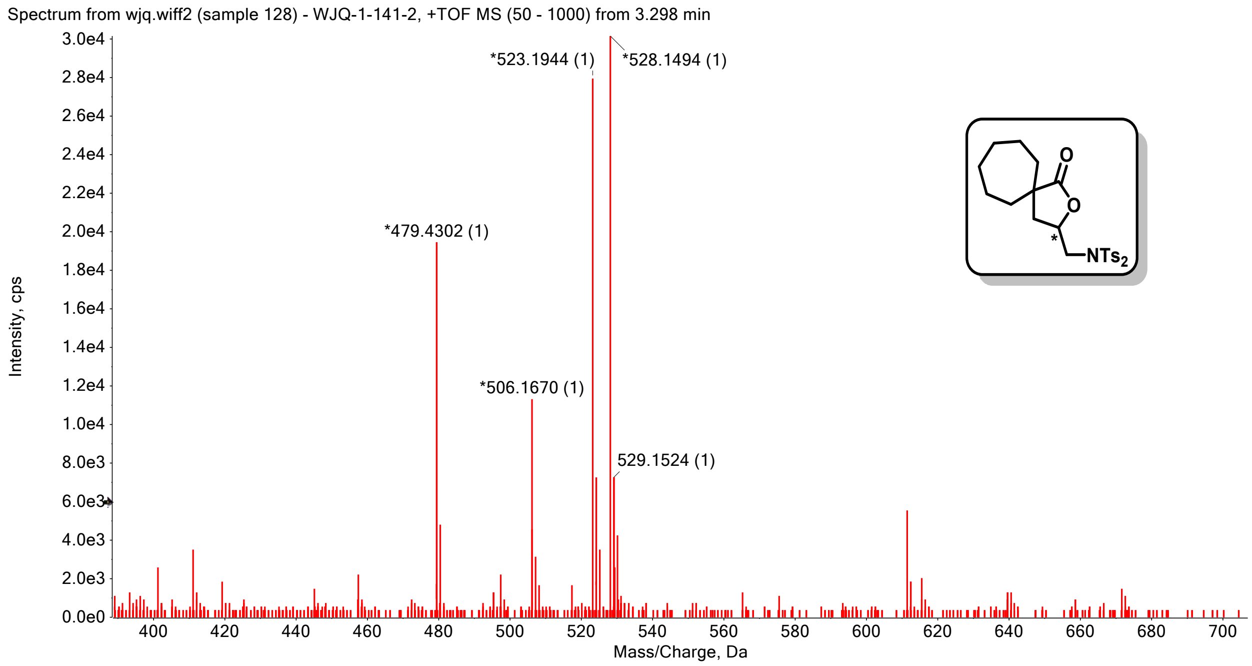


4d HRMS (ESI) *m/z*: $C_{24}H_{29}NO_6S_2$ $[M + H]^+$ Calcd for: 492.1509; Found: 492.1510.

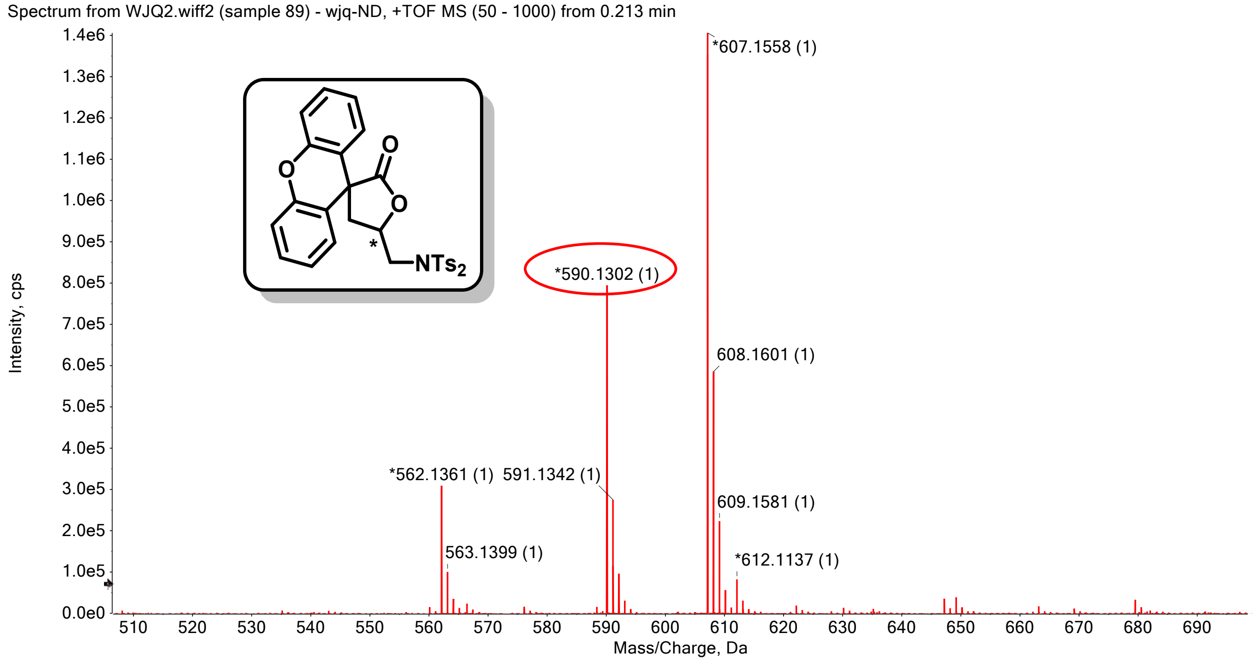
Spectrum from WJQ2.wiff2 (sample 93) - WJQ-P-9, +TOF MS (50 - 1000) from 0.204 min



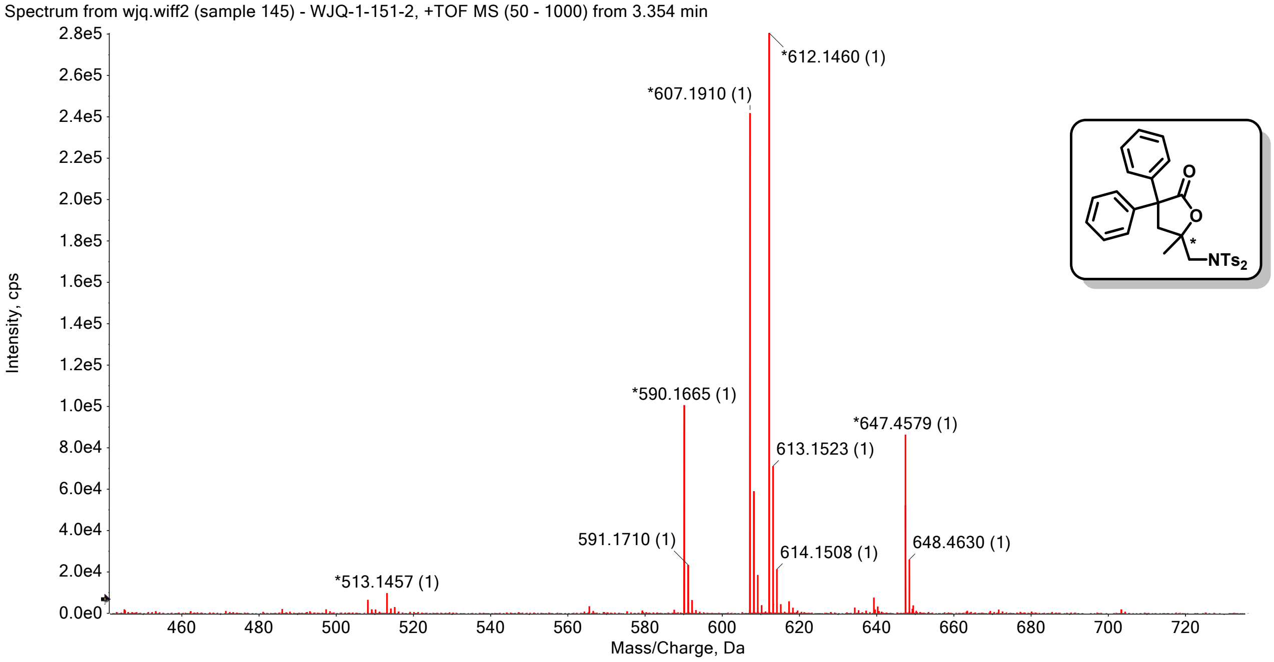
4e HRMS (ESI) *m/z*: $C_{25}H_{31}NO_6S_2$ $[M + H]^+$ Calcd for: 506.1666; Found: 506.1670.



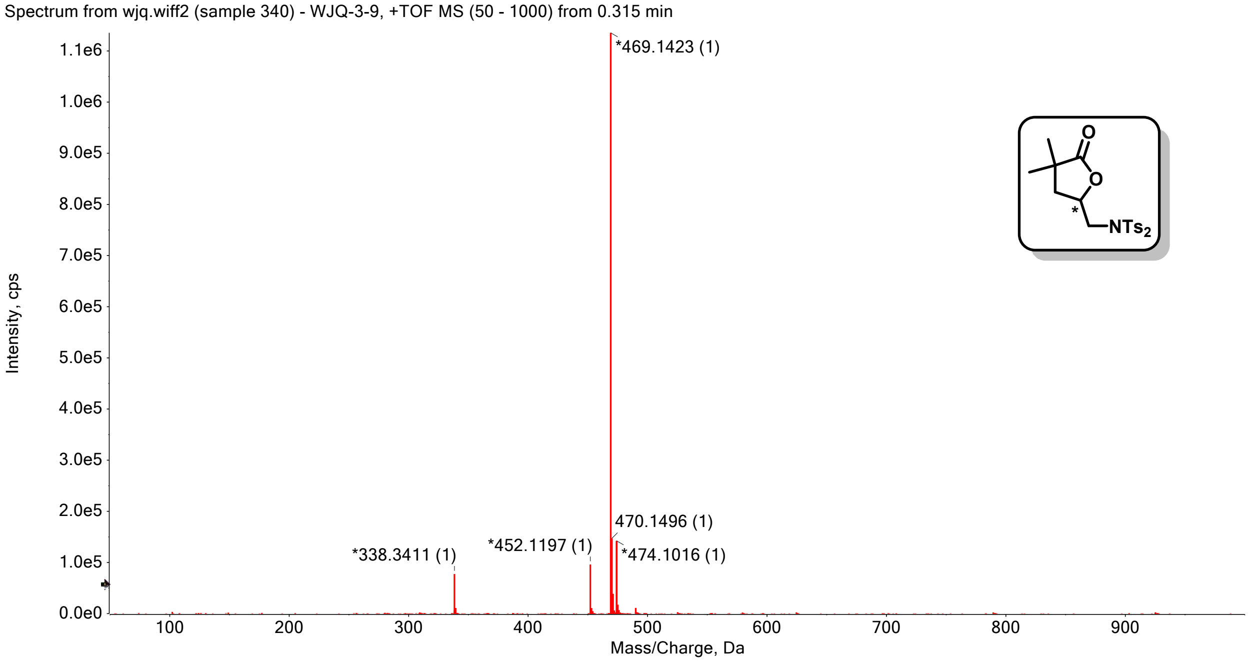
4f HRMS (ESI) *m*/*z*: $C_{31}H_{27}NO_7S_2$ $[M + H]^+$ Calcd for: 590.1302; Found: 590.1302.



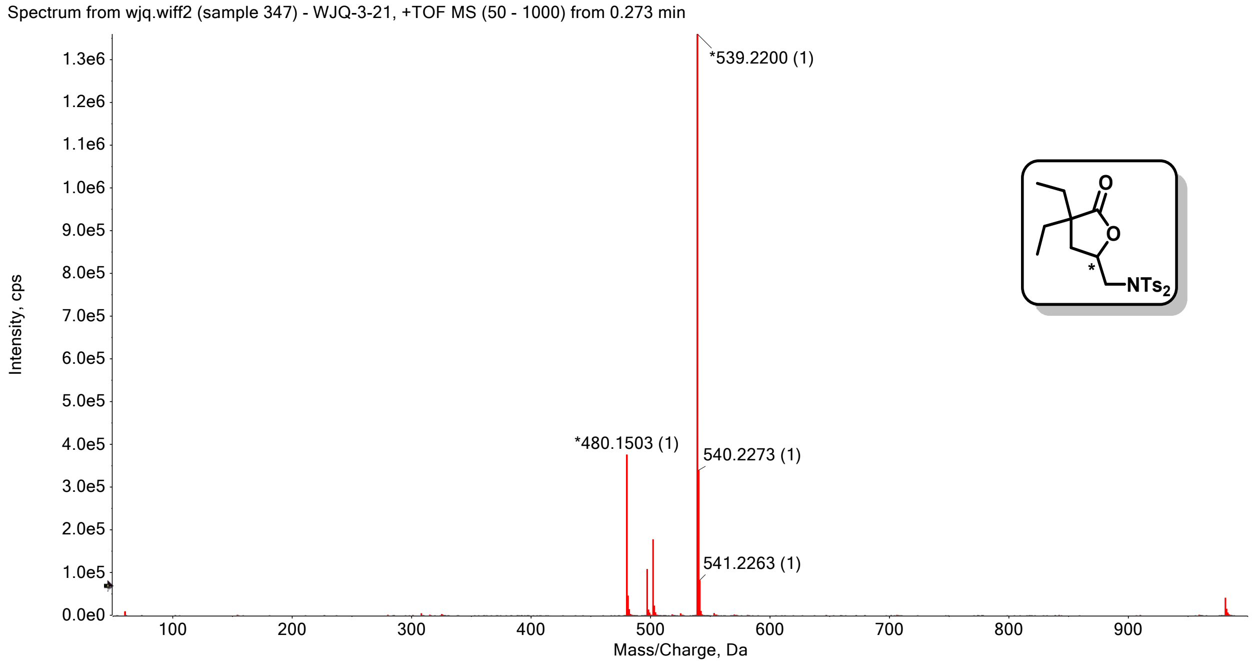
4g HRMS (ESI) *m*/*z*: $C_{32}H_{31}NO_6S_2$ $[M + H]^+$ Calcd for: 590.1666; Found: 590.1660.



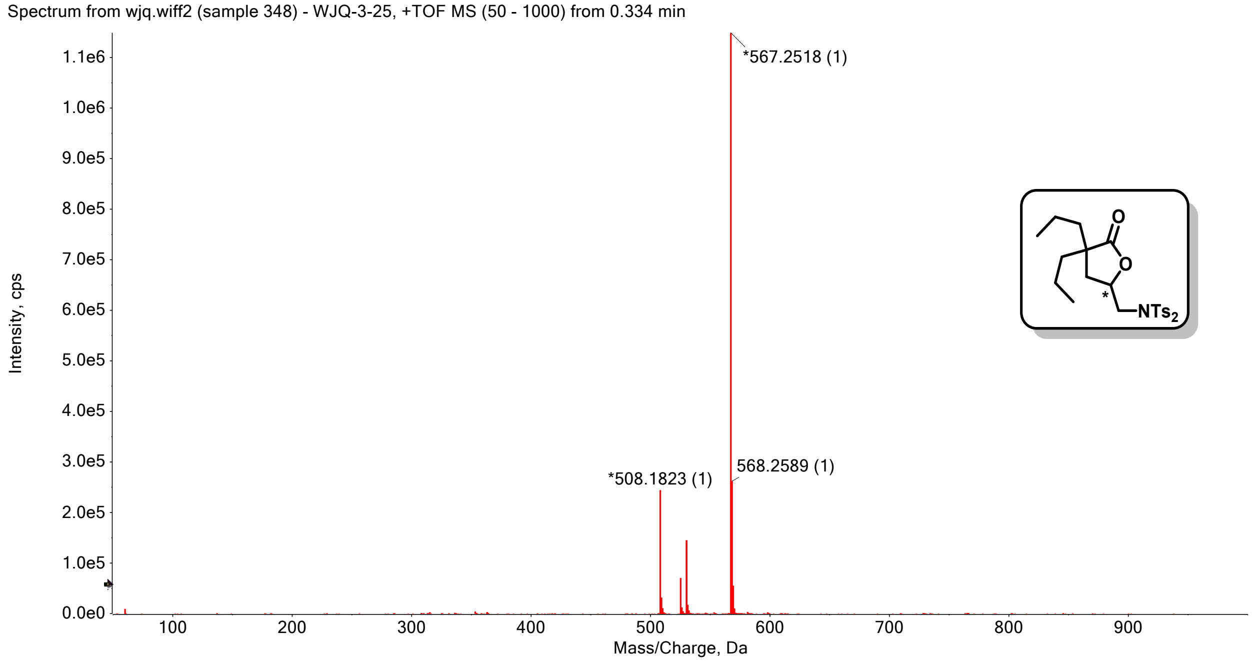
4h HRMS (ESI) *m*/*z*: $C_{21}H_{25}NO_6S_2$ $[M + H]^+$ Calcd for: 452.1196; Found: 452.1191.



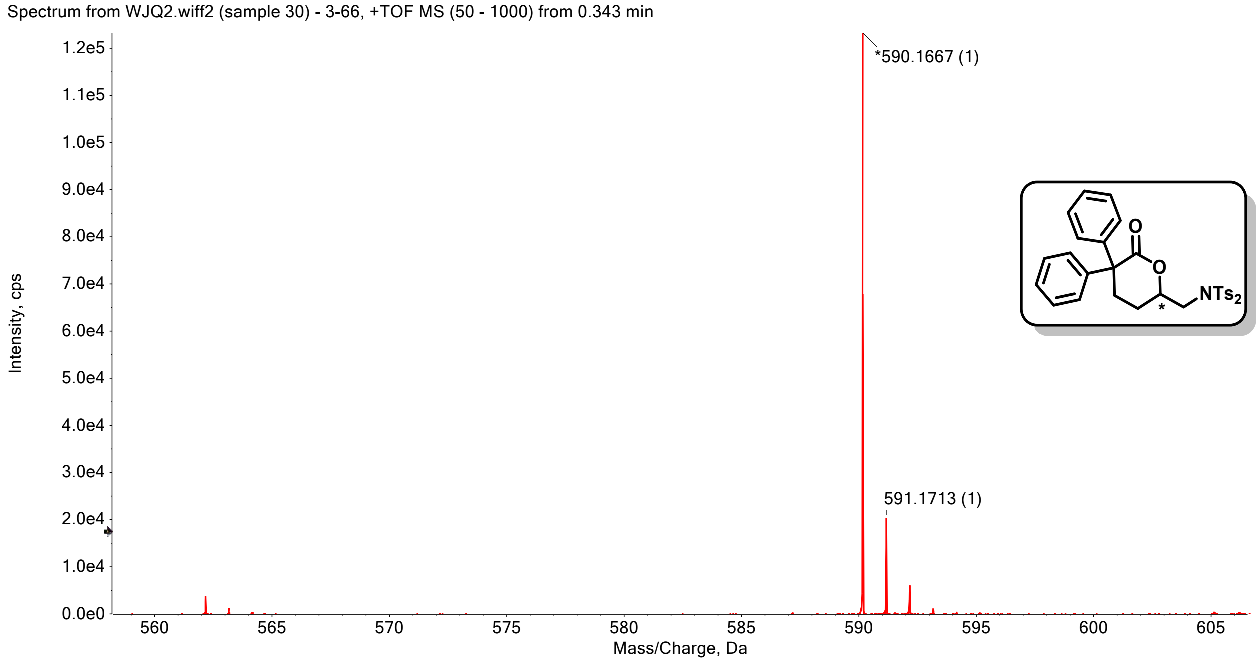
4i HRMS (ESI) *m*/*z*: $C_{23}H_{29}NO_6S_2$ $[M + H]^+$ Calcd for: 480.1509; Found: 480.1516.



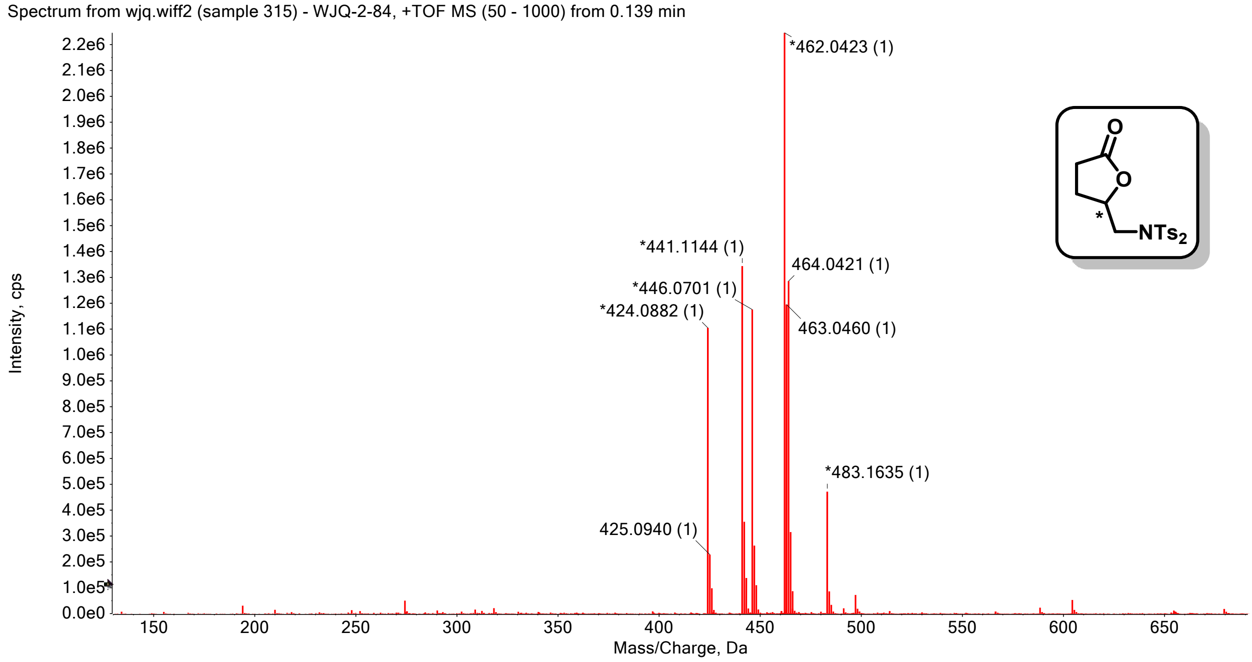
4j HRMS (ESI) *m*/*z*: $C_{25}H_{33}NO_6S_2$ $[M + H]^+$ Calcd for: 508.1822; Found: 508.1823.



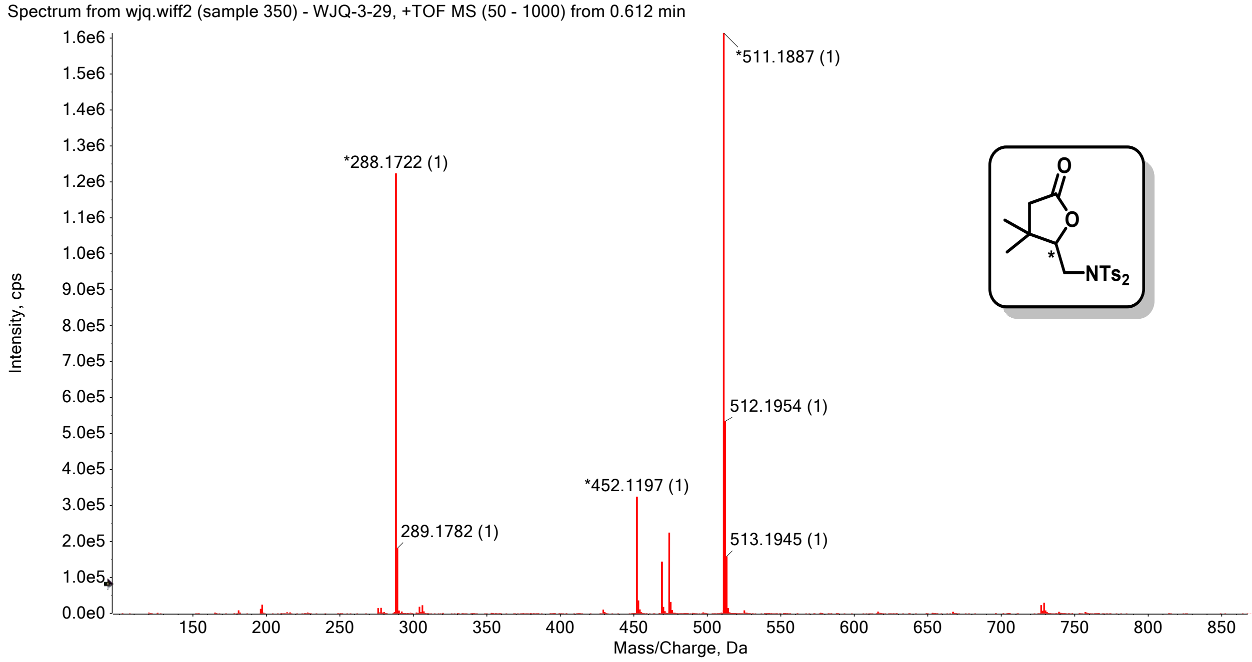
4k HRMS (ESI) *m*/*z*: $C_{32}H_{31}NO_6S_2$ $[M + H]^+$ Calcd for: 590.1666; Found: 590.1667.



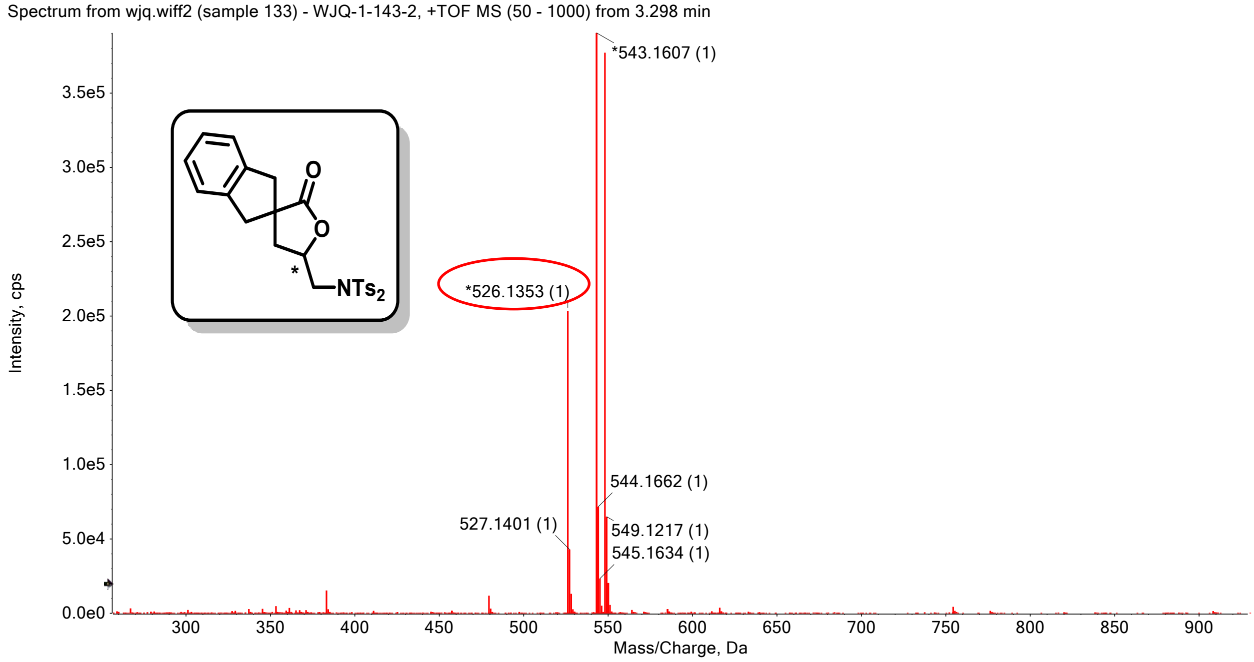
4l HRMS (ESI) *m*/*z*: $C_{19}H_{21}NO_6S_2$ $[M + H]^+$ Calcd for: 424.0883; Found: 424.0882.



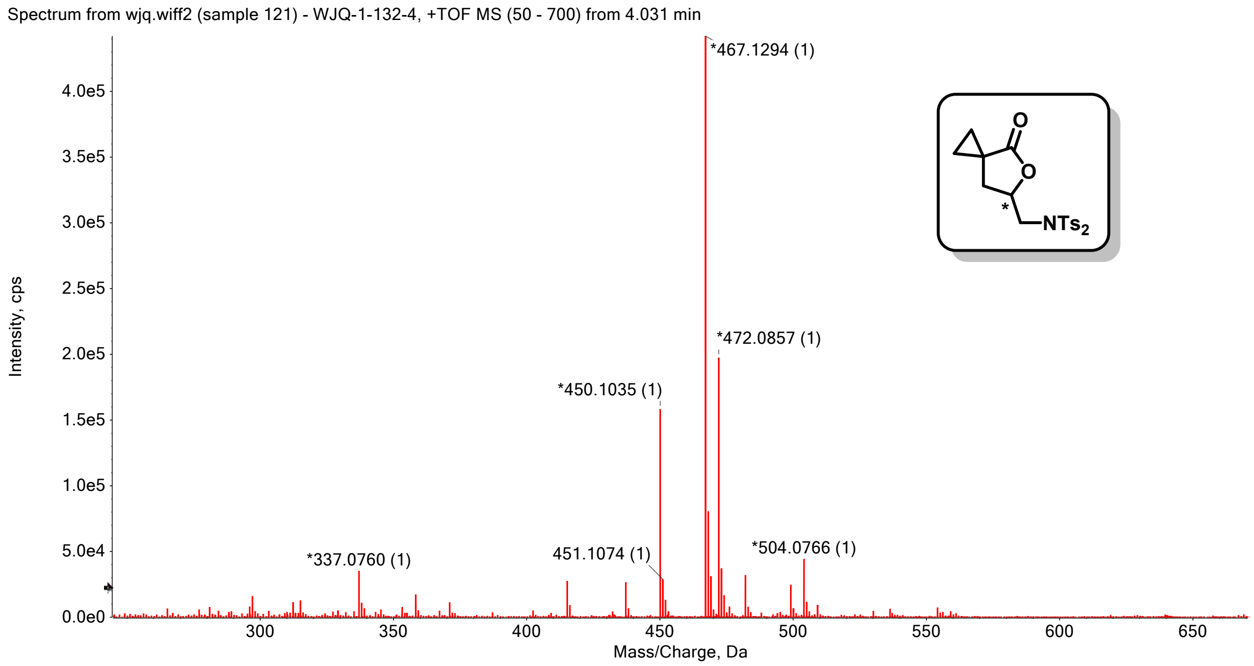
4m HRMS (ESI) *m*/*z*: $C_{21}H_{25}NO_6S_2$ $[M + H]^+$ Calcd for: 452.1196; Found: 452.1197.



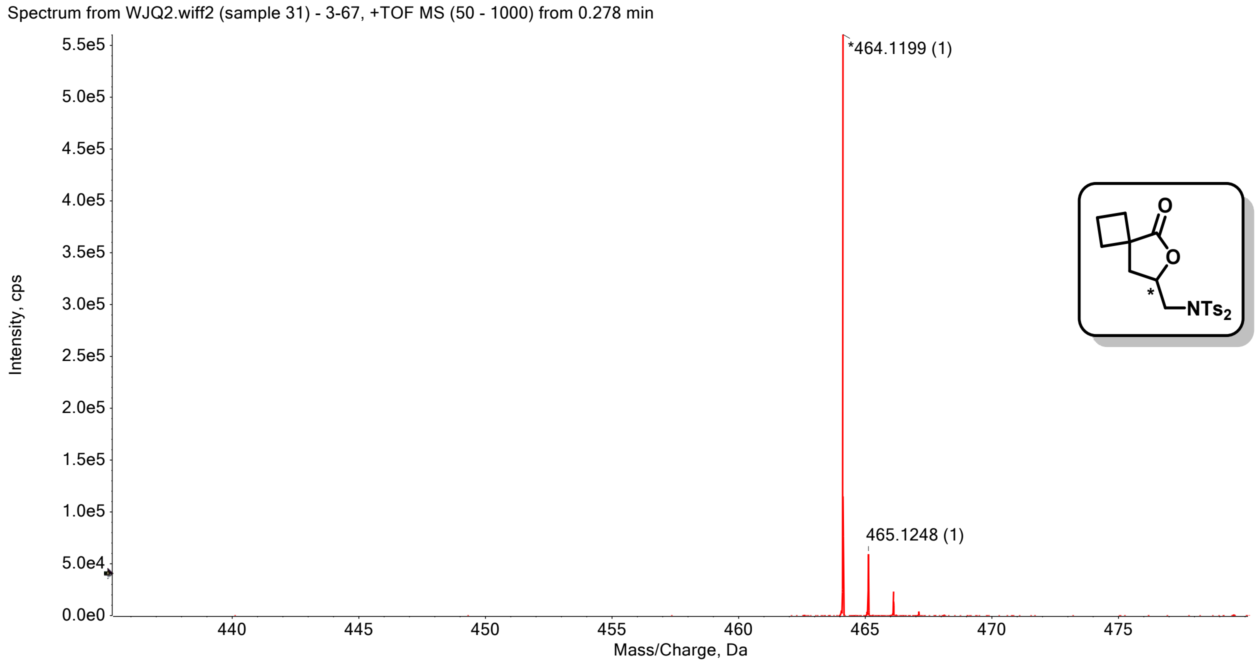
4o HRMS (ESI) *m*/*z*: $C_{27}H_{27}NO_6S_2$ [M + H]$^+$ Calcd for: 526.1353; Found: 526.1353.



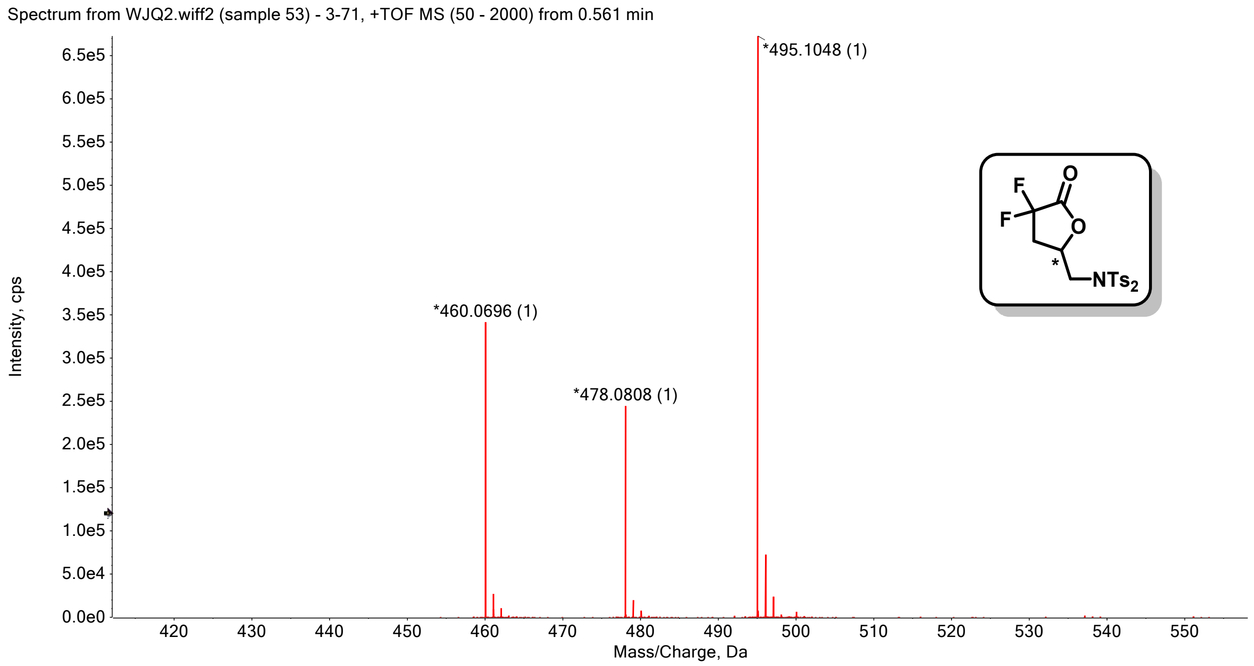
4p HRMS (ESI) *m*/*z*: $C_{21}H_{23}NO_6S_2$ [M + H]$^+$ Calcd for: 450.1040; Found: 450.1035.



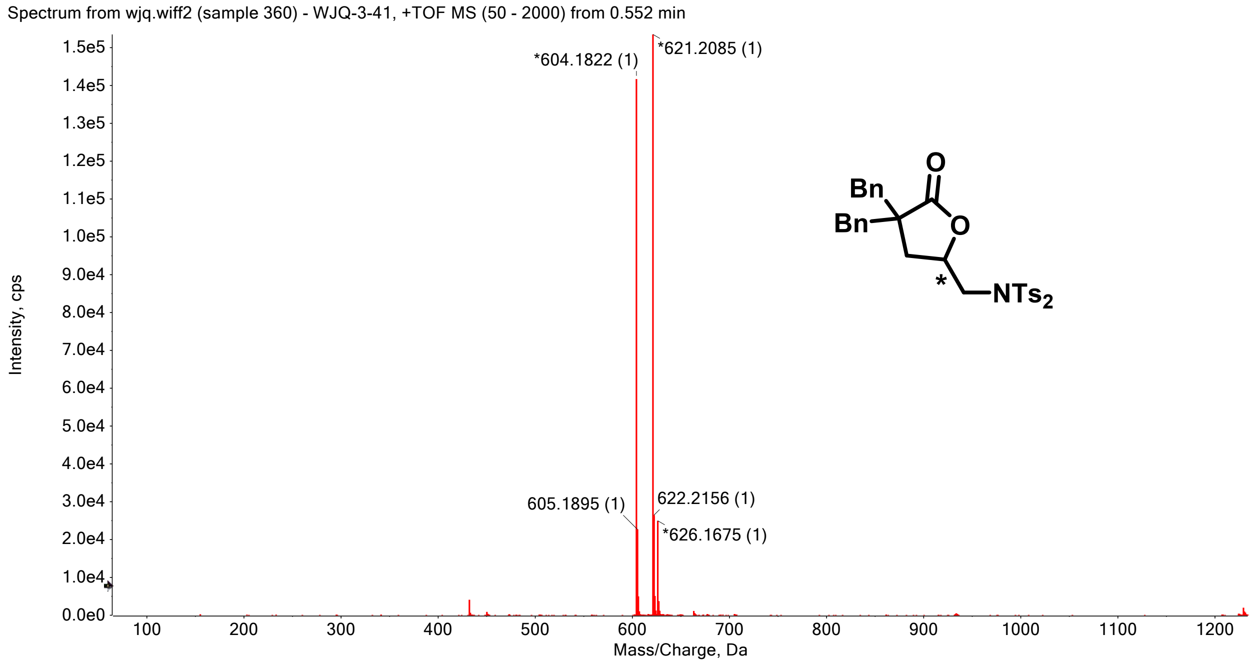
4q HRMS (ESI) *m/z*: $C_{22}H_{25}NO_6S_2$ $[M + H]^+$ Calcd for: 464.1196; Found: 464.1199.



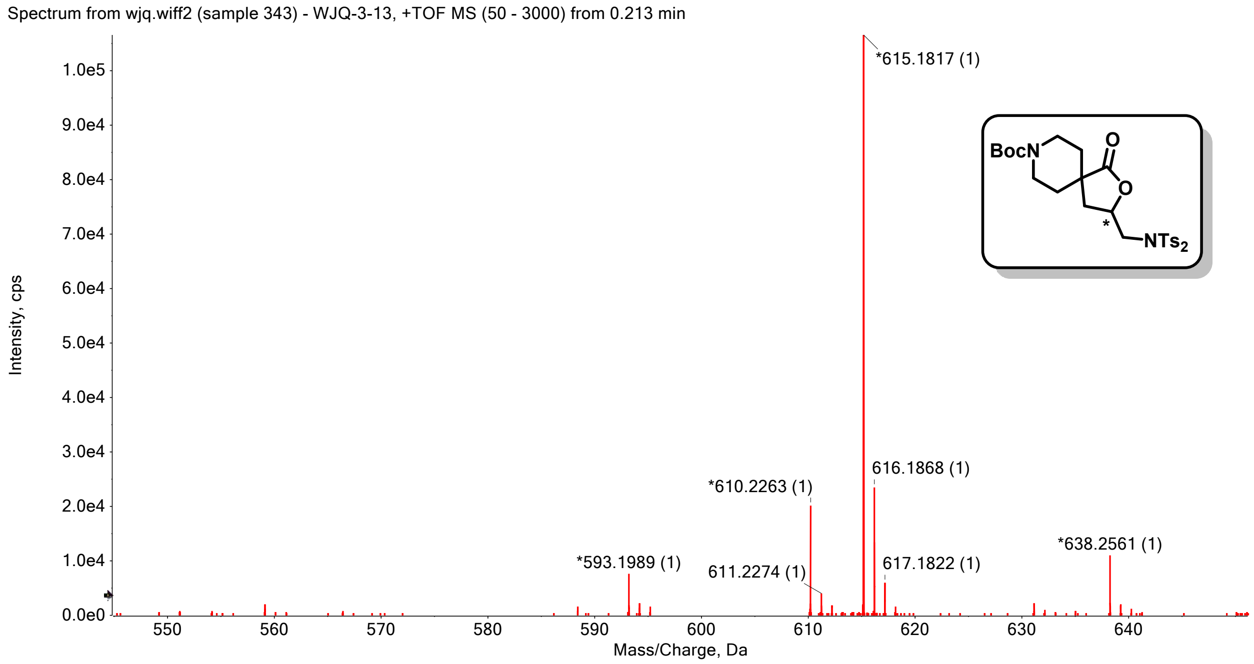
4r HRMS (ESI) *m/z*: $C_{19}H_{19}F_2NO_6S_2$ $[M + H]^+$ Calcd for: 460.0695; Found: 460.0696.



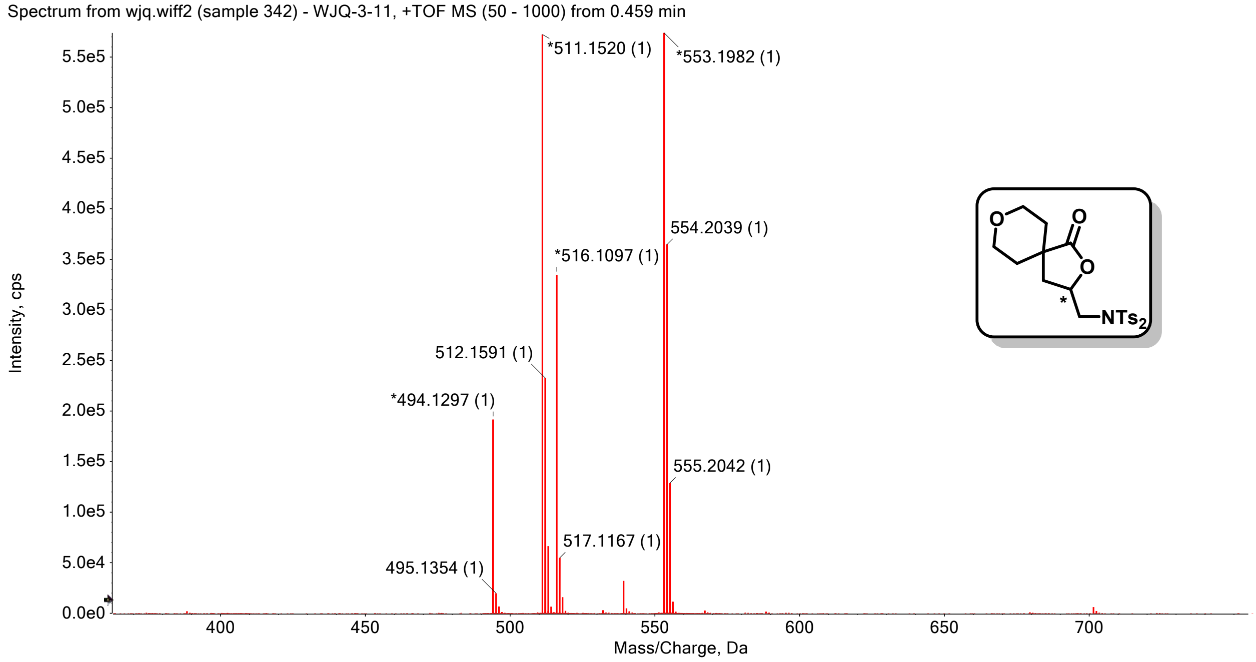
4s



4t HRMS (ESI) *m*/*z*: $C_{28}H_{36}N_2O_8S_2$ $[M+H]^+$ Calcd for: 593.1986; Found: 593.1996.



4u HRMS (ESI) *m*/*z*: $C_{23}H_{27}NO_7S_2$ $[M + H]^+$ Calcd for: 494.1302; Found: 494.1297.



4a’ HRMS (ESI) *m*/*z*: $C_{29}H_{23}Cl_2NO_6S_2$ $[M + H]^+$ Calcd for: 616.0417; Found: 616.0416.

Spectrum from WJQ2.wiff2 (sample 81) - N-Cl, +TOF MS (50 - 2000) from 0.278 min