

Supporting Information

Pd-Catalyzed Atroposelective C–H Allylation and Alkenylation: Access to Enantioenriched Atropisomers Featuring Pentatomic Heteroaromatics

Hao-Ming Chen,^{†,§} Shuo Zhang,^{‡,§} Gang Liao,[‡] Qi-Jun Yao,[‡] Xue-Tao Xu,[†] Kun Zhang,[†] Bing-Feng Shi^{*,‡}

[†]School of Biotechnology and Health Sciences, Wuyi University, Jiangmen 529020, China

[‡]Department of Chemistry, Zhejiang University, Hangzhou 310027, China

*Email: bfshi@zju.edu.cn

[§]These authors contributed equally to this work

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1. Experimental Section

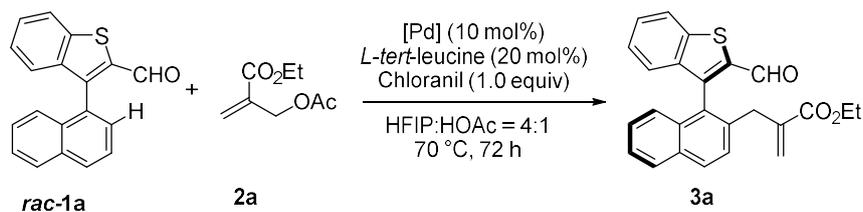
1.1 Optimization of Reaction Conditions

Table S1. Screening of Additives for Enantioselective Allylation^a

rac-1a + **2a** $\xrightarrow[\text{HFIP:HOAc} = 4:1, 70\text{ }^\circ\text{C}, 72\text{ h}]{\text{Pd(OAc)}_2 (10\text{ mol}\%), \text{L-tert-leucine} (20\text{ mol}\%), \text{Additives} (1.0\text{ equiv})}$ **3a**

Entry	Additives	Yield (%) ^b	ee (%) ^c
1.	BQ	44	97
2.	<i>p</i> -Toluquinone	42	20
3.	Duroquinone	47	5
4.	Naphthoquinone	27	84
5.	2-Chloronaphthoquinone	--	--
6.	Menadione	33	23
7.	2,6-Dichloquinone	50	99
8.	2-Methoxyquinone	52	97
9.	Chloranil	55 (48)	96

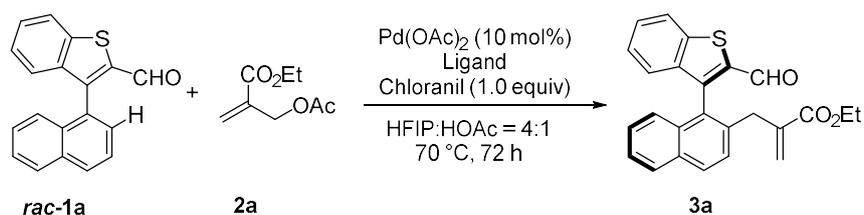
^aReaction conditions: **rac-1a** (0.1 mmol), **2a** (0.3 mmol), Pd(OAc)₂ (10 mol%), *L-tert-leucine* (20 mol%), additives (1.0 equiv), HFIP:HOAc = 4:1 (1 mL), 70 °C, air, 72 h. ^bDetermined by ¹HNMR spectroscopy using 1,3,5-trimethoxybenzene as the internal standard. ^cThe ee value was determined by chiral HPLC.

Table S2. Screening of Palladium Catalyst for Enantioselective Allylation^a

Entry	[Pd]	Yield (%) ^b	ee (%) ^c
1.	Pd(OAc) ₂	55	96
2.	Pd(TFA) ₂	50	96
3.	Pd(dba) ₂	48	96
4.	Pd ₂ (dba) ₃	49	96
5.	Pd(OTf) ₂	--	--
6.	PdBr ₂	--	--
7.	Pd(OTs) ₂ (MeCN) ₂	36	98
8.	Pd(acac) ₂	28	98
9.	Pd(NMe) ₄ (OTf) ₂	40	97
10.	Pd(dbm) ₂	39	97

^aReaction conditions: *rac*-1a (0.1 mmol), 2a (0.3 mmol), Pd (10 mol%), *L*-tert-leucine (20 mol%), chloranil (1.0 equiv), HFIP:HOAc = 4:1 (1 mL), 70 °C, air, 72 h. ^bDetermined by ¹HNMR spectroscopy using 1,3,5-trimethoxybenzene as the internal standard. ^cThe ee value was determined by chiral HPLC.

Table S3. Screening of Ligands for Enantioselective Allylation^a



Entry	Ligand (mol%)	Yield (%) ^b	ee (%) ^c
1	L1 (20)	55	96
2	L2 (30)	26	53
3	L3 (20)	trace	--
4	L4 (30)	31	73
5	L5 (20)	25	69
6	L6 (20)	trace	--
7	L7 (20)	43	44
8	L8 (20)	13	--
<i>g</i> ^d	--	--	--

^aReaction conditions: **rac-1a** (0.1 mmol), **2a** (0.3 mmol), Pd (10 mol%), ligand (mol%), chloranil (1.0 equiv), HFIP:HOAc = 4:1 (1.0 mL), 70 °C, air, 72 h. ^bDetermined by ¹HNMR spectroscopy using 1,3,5-trimethoxybenzene as the internal standard. ^cThe ee value was determined by chiral HPLC. ^dWithout Ligand.

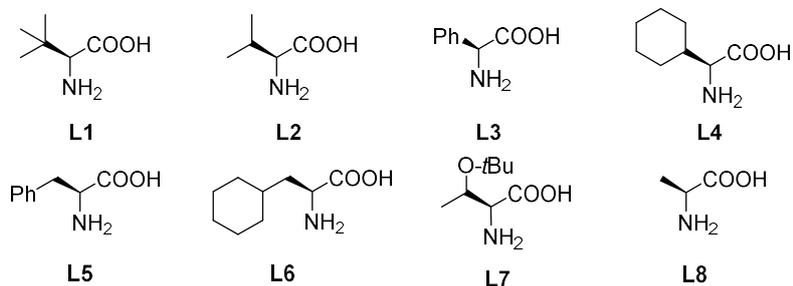
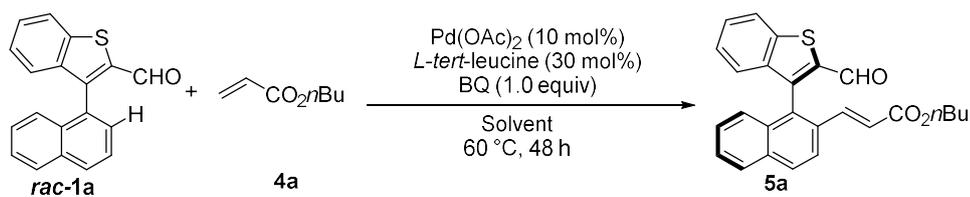


Table S4. Screening of Solvents for Enantioselective Alkenylation^a



Entry	Solvent	Yield (%) ^b	ee (%) ^c
1	HOAc	37	97
2	TFE	trace	--
3	HFIP	trace	--
4	DCE	trace	--
5	THF	trace	--
6	Toluene	trace	--
7	TFE:HOAc = 9:1	49	99
8	TFE:HOAc = 4:1	50	99
9	HFIP:HOAc = 9:1	52	99
10	HFIP:HOAc = 4:1	54	98
11	HFIP:HOAc = 1:1	54	97

^aReaction conditions: **rac-1a** (0.1 mmol), **4a** (0.3 mmol), Pd(OAc)₂ (10 mol%), *L-tert-leucine* (30 mol%), BQ (1.0 equiv), solvent (1 mL), 60 °C, air, 48 h. ^bDetermined by ¹HNMR spectroscopy using 1,3,5-trimethoxybenzene as the internal standard. ^cThe ee value was determined by chiral HPLC.

1.2 Pd(II)-Catalyzed Asymmetric C-H Allylation and Alkenylation

Table S5. Substrates of Biaryls

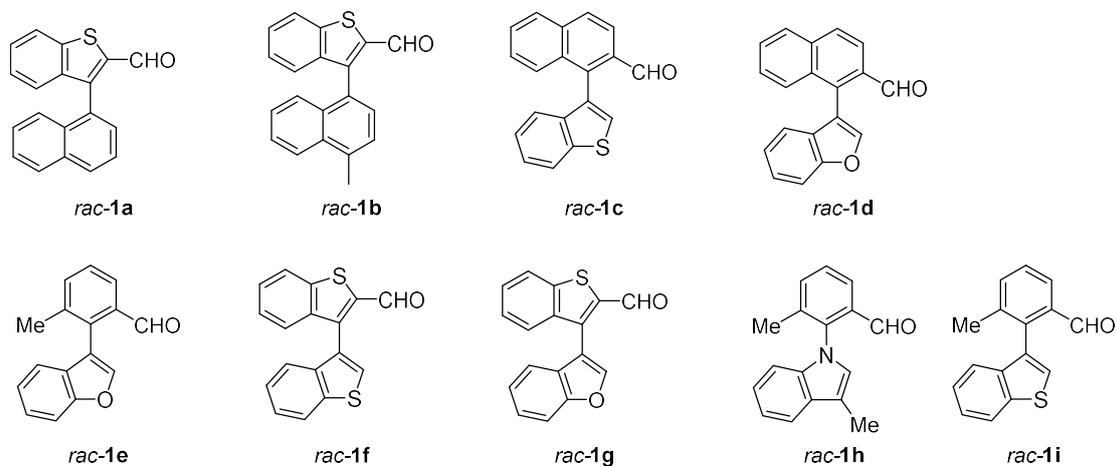
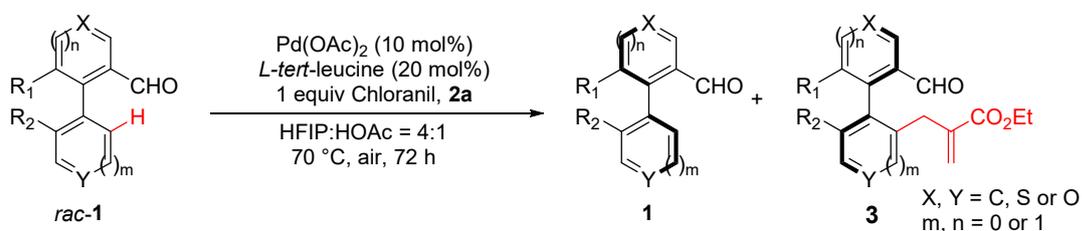


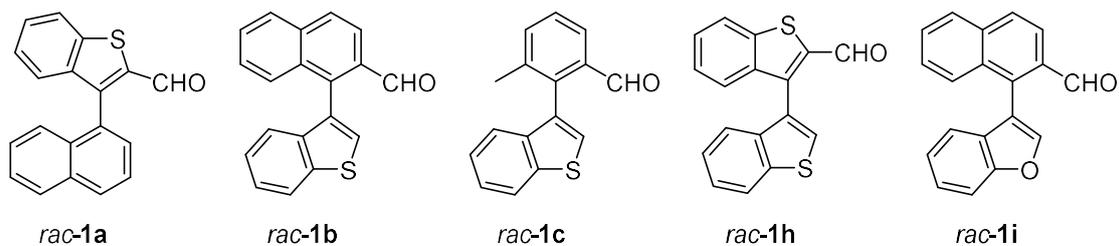
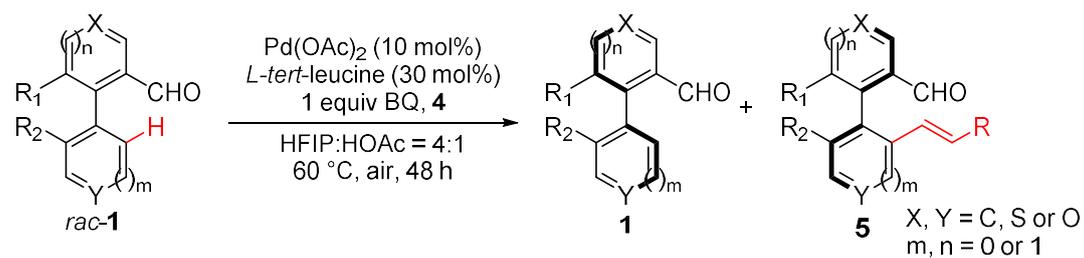
Table S6. Scope of Pd-Catalyzed C-H Allylation



entry	1	3	1		3		<i>s</i> ^c
			yield (%) ^a	ee (%) ^b	yield (%) ^a	ee (%) ^b	
1	1a	3a	47	30	48	96	66
2	1b	3b	32	27	41	96	64
3	1c	3c	/	/	60	88	/
4	1d	3d	/	/	83	78	/
5	1e	1e	/	/	61	94	/
6	1f	1f	/	/	77	92	/

^aIsolated yield. ^bThe ee value was determined by chiral HPLC. ^c $s = \ln[(1-C)(1-ee_1)]/\ln[(1-C)(1+ee_1)]$, $C = ee_1/(ee_1+ee_3)$.

Table S7. Scope of Pd-Catalyzed C–H Alkenylation



entry	1	5	1		5		s^c
			yield (%) ^a	ee (%) ^b	yield (%) ^a	ee (%) ^b	
1	1a	5a	37	67	51	98	200
2	1b	5b	24	32	58	90	26
3	1c	5c	30	70	52	90	40
4	1a	5d	43	58	49	99	363
5	1a	5e	41	63	50	99	383
6	1a	5f	45	41	54	97	98
7	1a	5g	48	72	45	96	106
8	1h	5h	/	/	70	98	/
9	1i	5i	/	/	50	75	/

^aIsolated yield. ^bThe ee value was determined by chiral HPLC. ^c $s = \ln[(1-C)(1-ee_1)]/\ln[(1-C)(1+ee_1)]$, $C = ee_1/(ee_1+ee_5)$.

2. NMR Spectra

3a

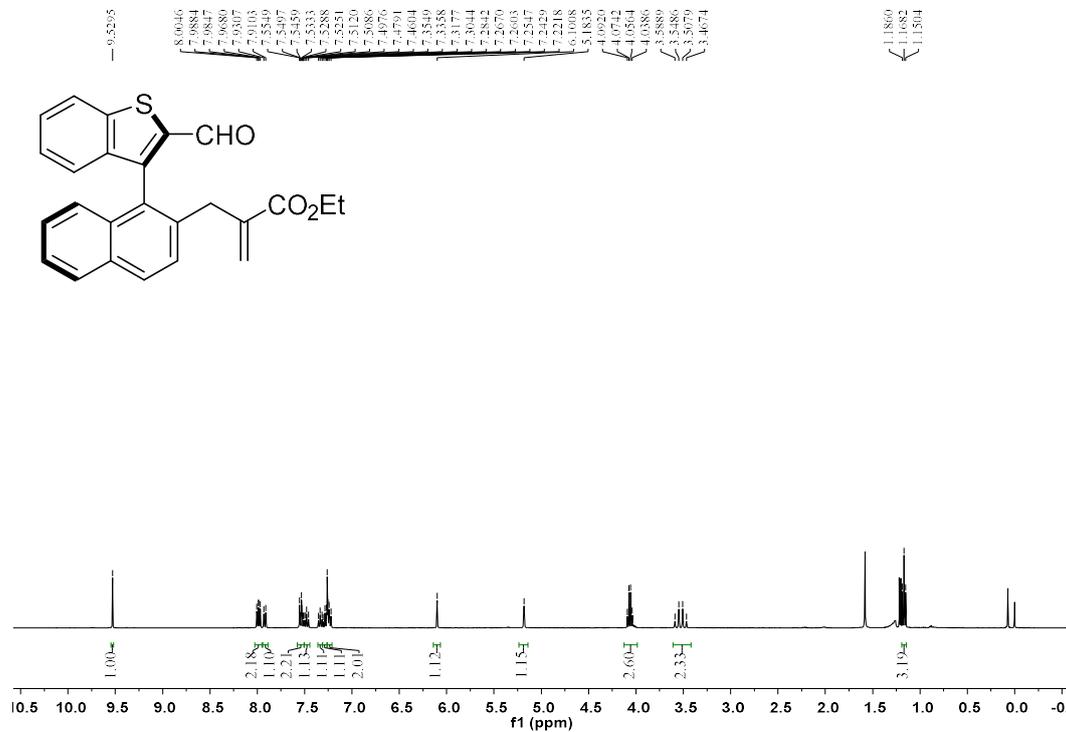


Figure S1 ¹H NMR Spectrum of 3a in CDCl₃

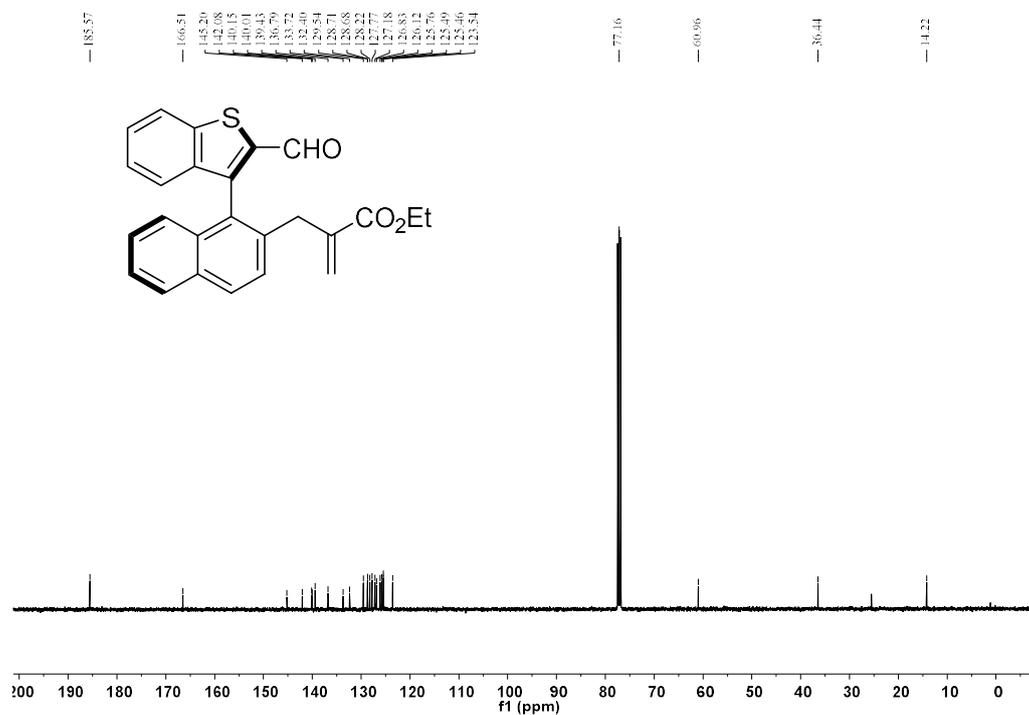


Figure S2 ¹³C NMR Spectrum of 3a in CDCl₃

3b

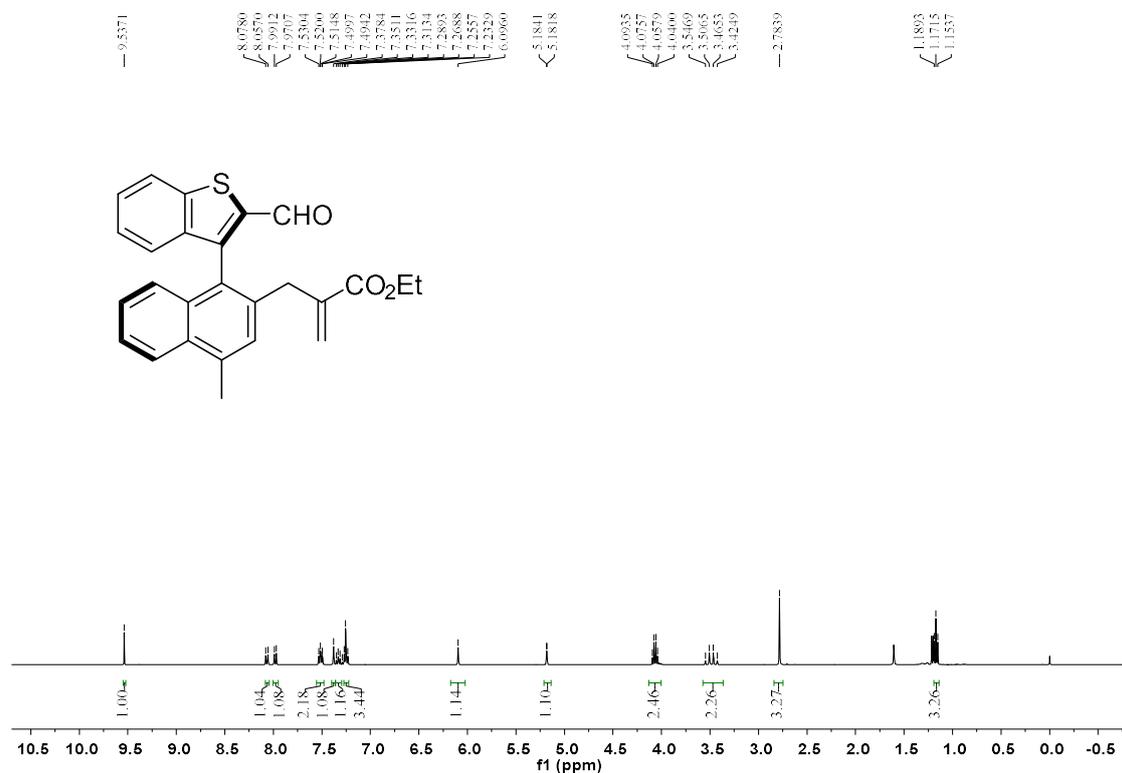


Figure S3 ¹H NMR Spectrum of 3b in CDCl₃

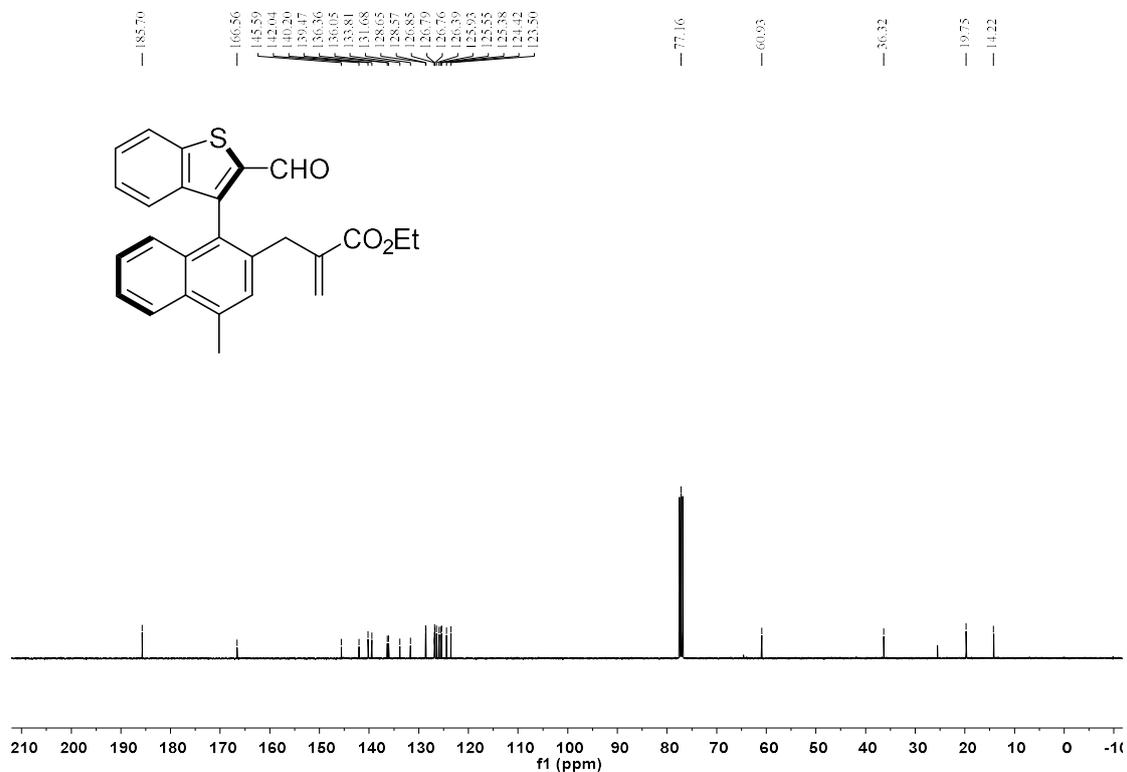


Figure S4 ¹³C NMR Spectrum of 3b in CDCl₃

3c

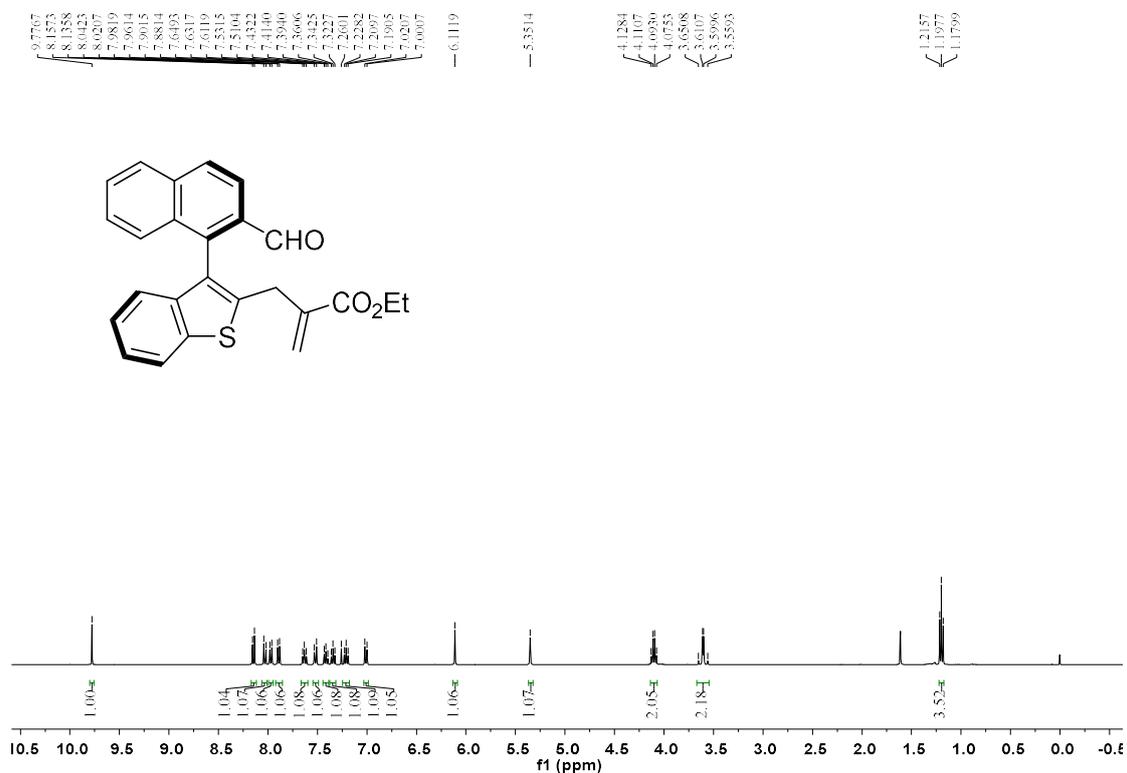


Figure S5 ¹H NMR Spectrum of 3c in CDCl₃

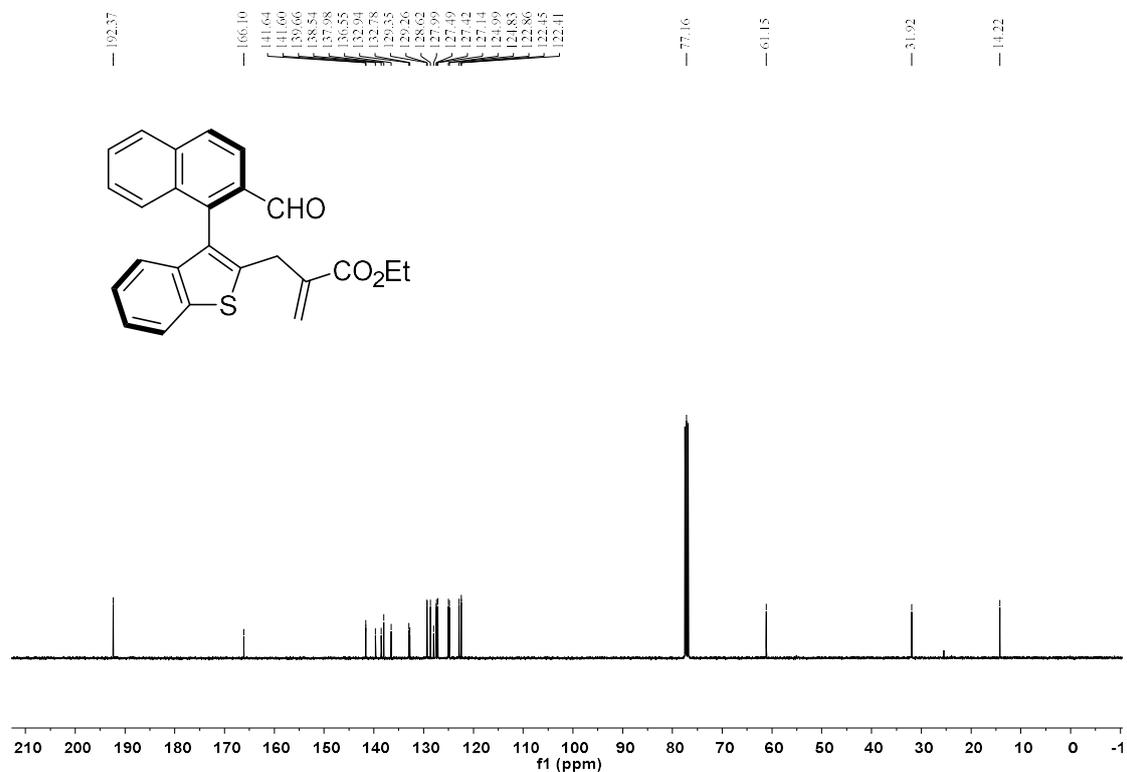


Figure S6 ¹³C NMR Spectrum of 3c in CDCl₃

3d

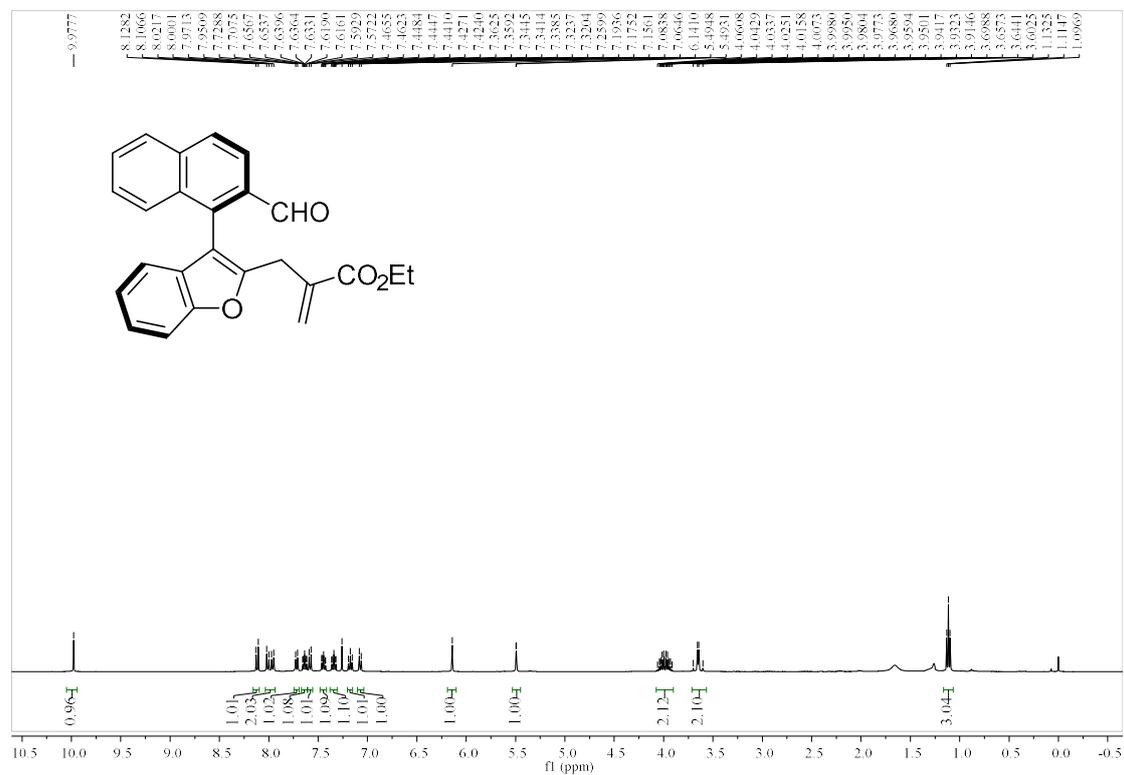


Figure S7 ^1H NMR Spectrum of 3d in CDCl_3

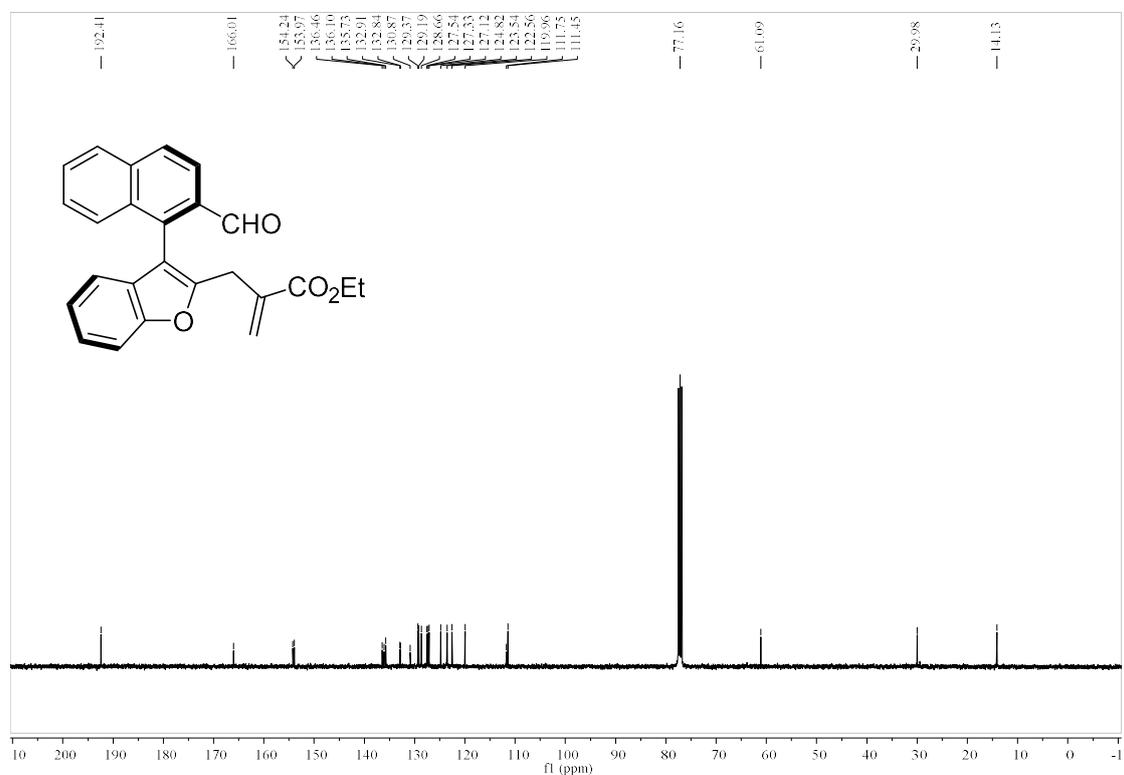


Figure S8 ^{13}C NMR Spectrum of 3d in CDCl_3

3e

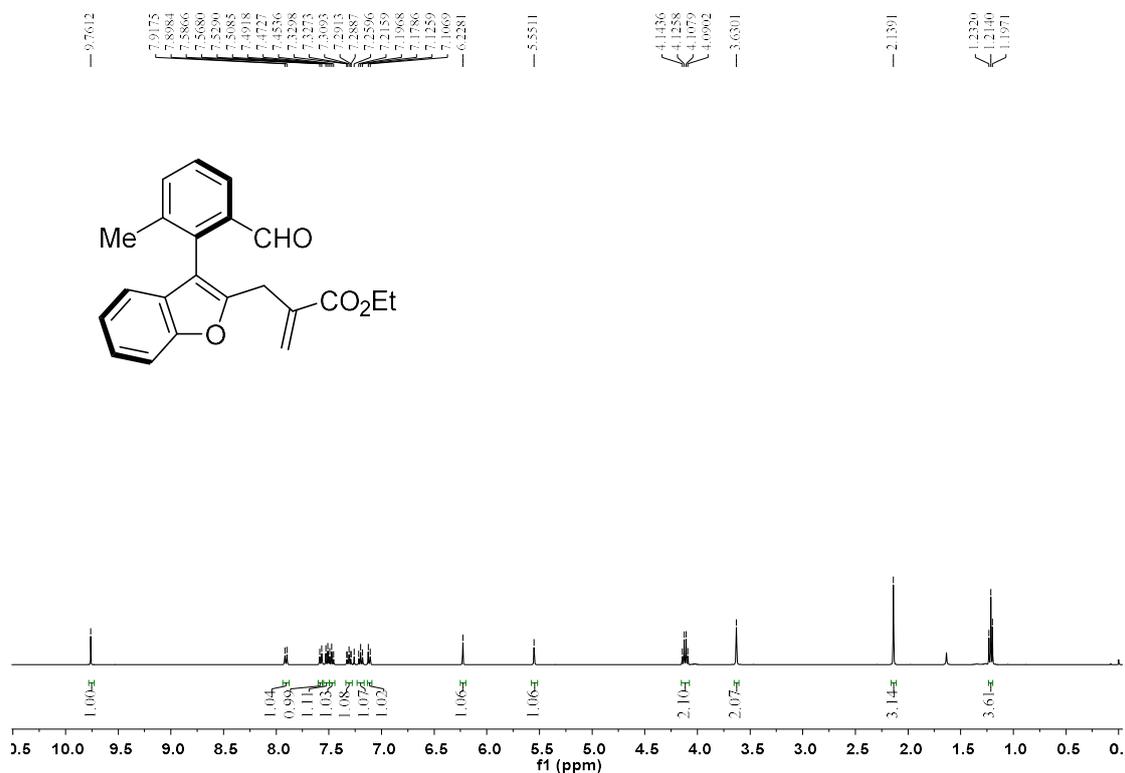


Figure S9 ¹H NMR Spectrum of 3e in CDCl₃

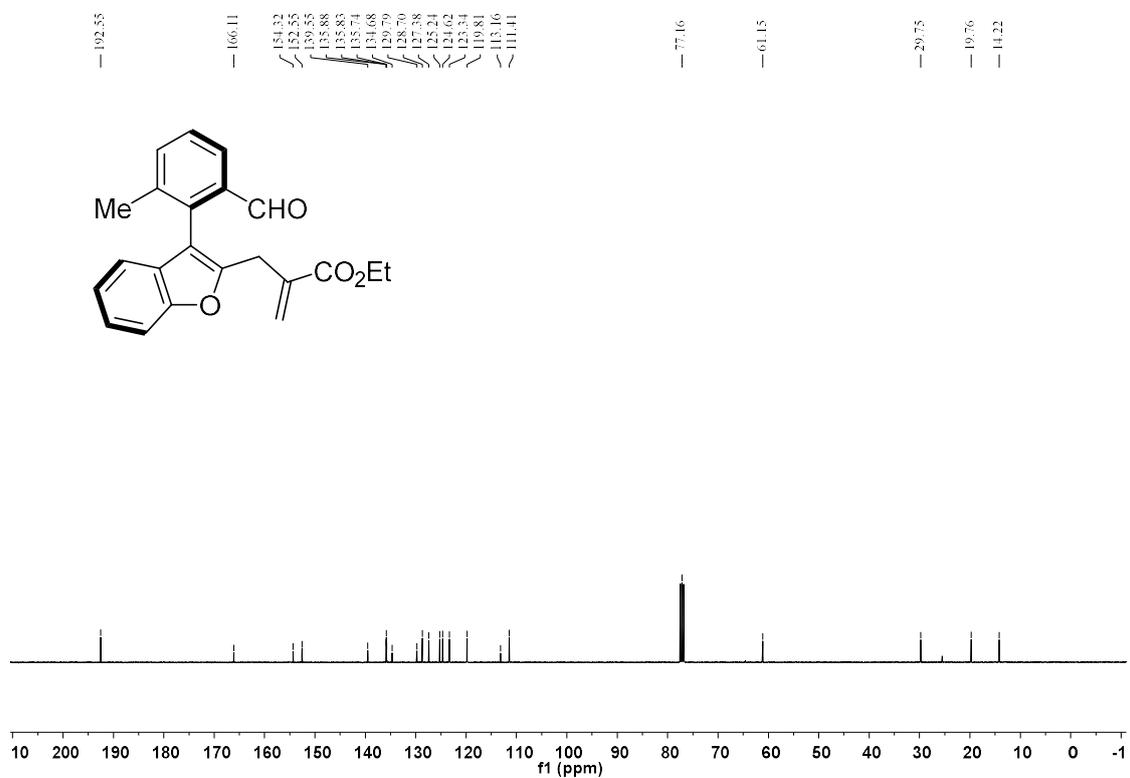


Figure S10 ¹³C NMR Spectrum of 3e in CDCl₃

3f

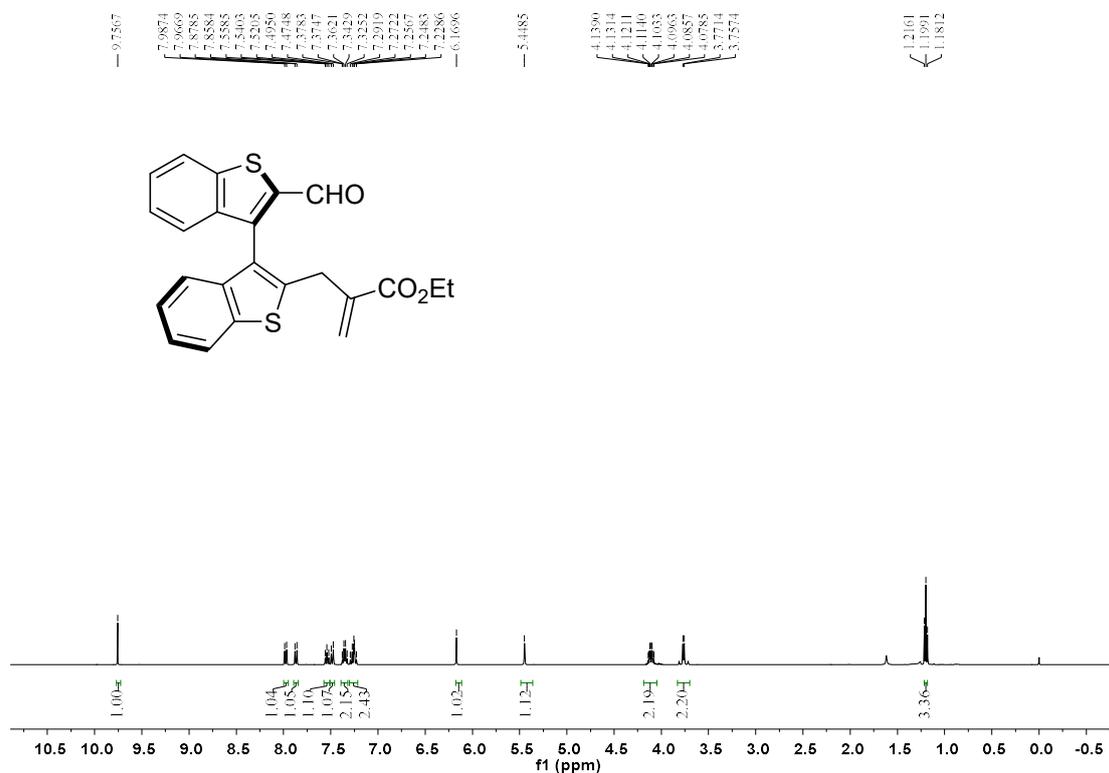


Figure S11 ¹H NMR Spectrum of 3f in CDCl₃

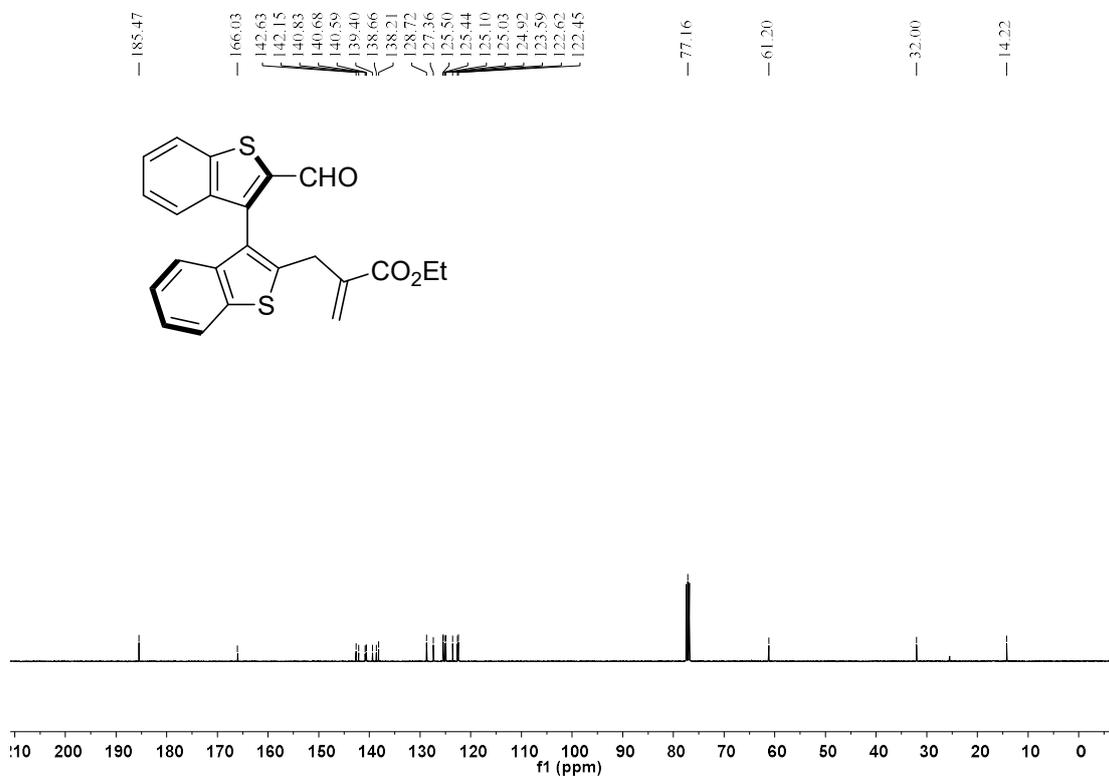
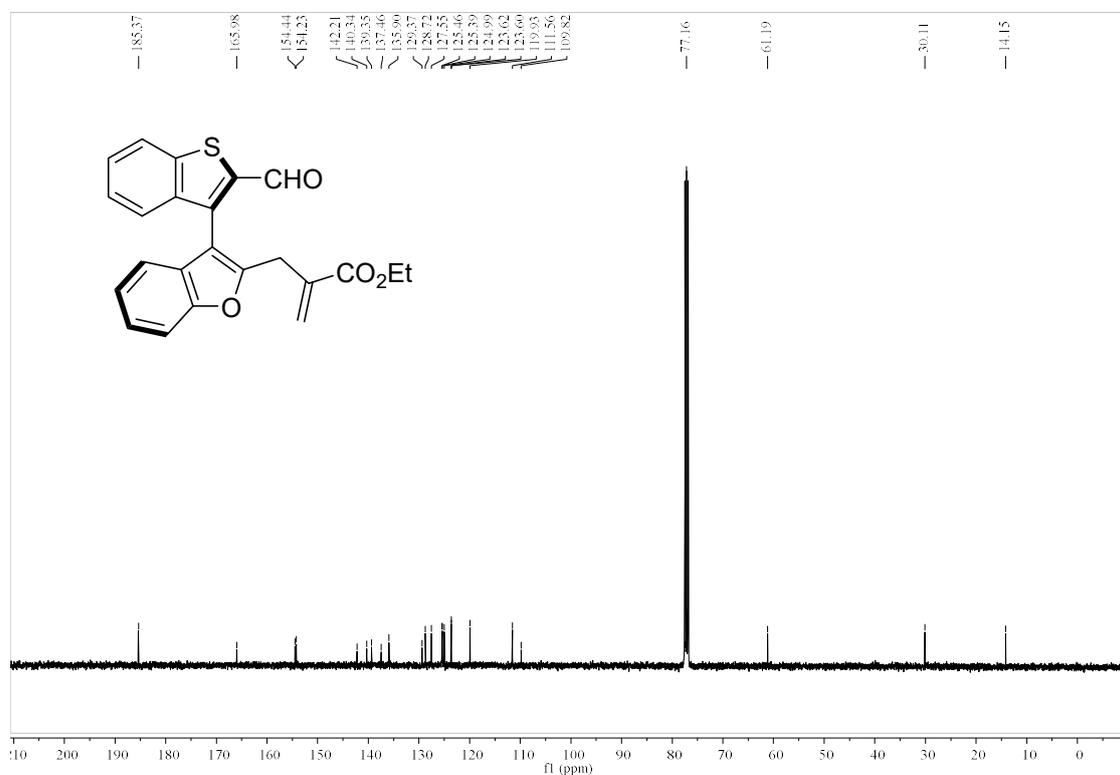
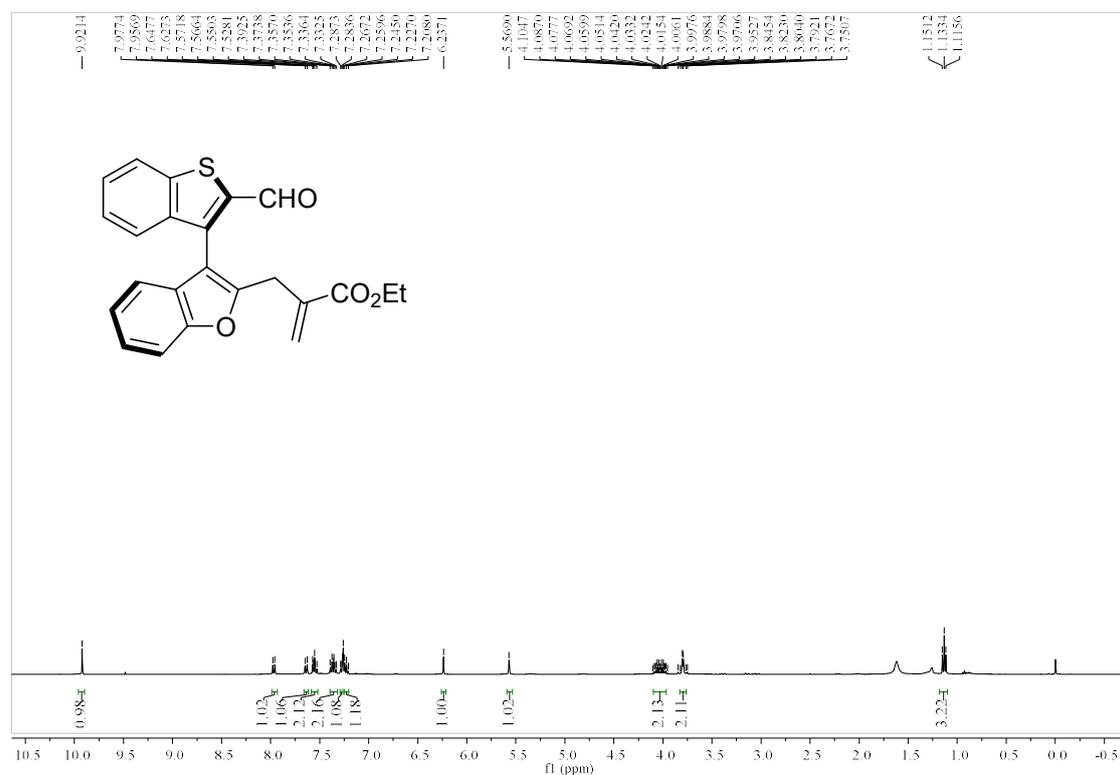


Figure S12 ¹³C NMR Spectrum of 3f in CDCl₃

3g



5a

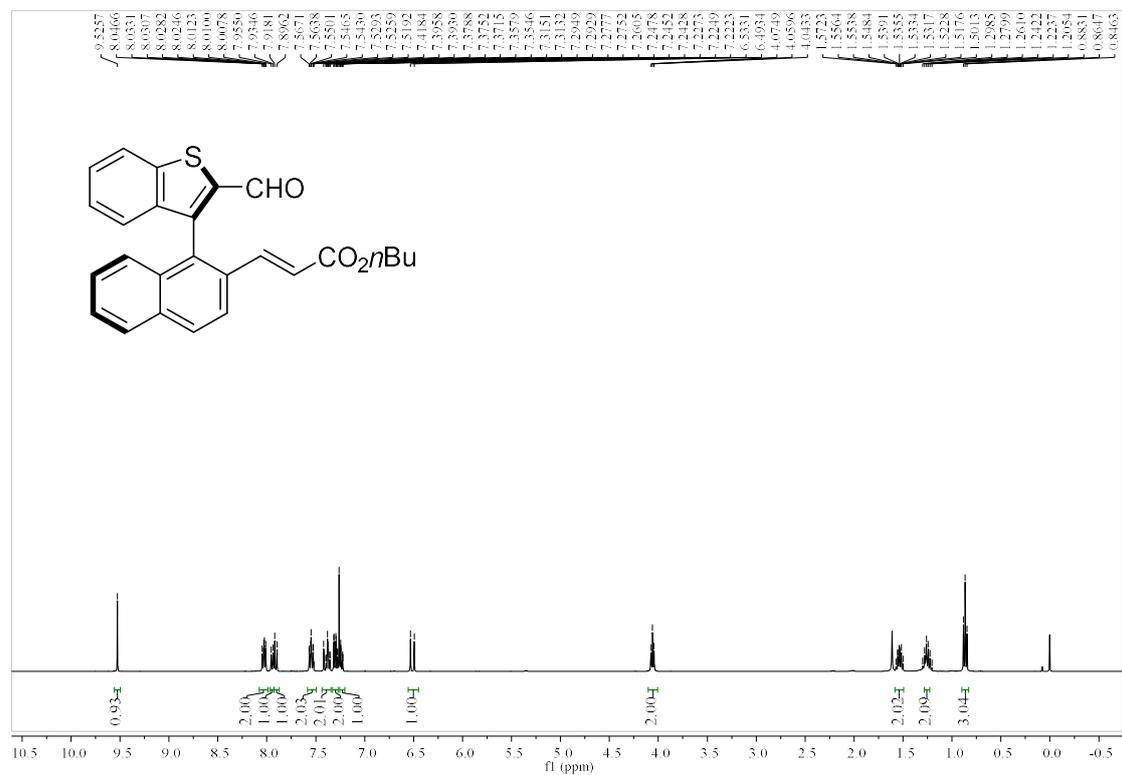


Figure S15 ¹H NMR Spectrum of 5a in CDCl₃

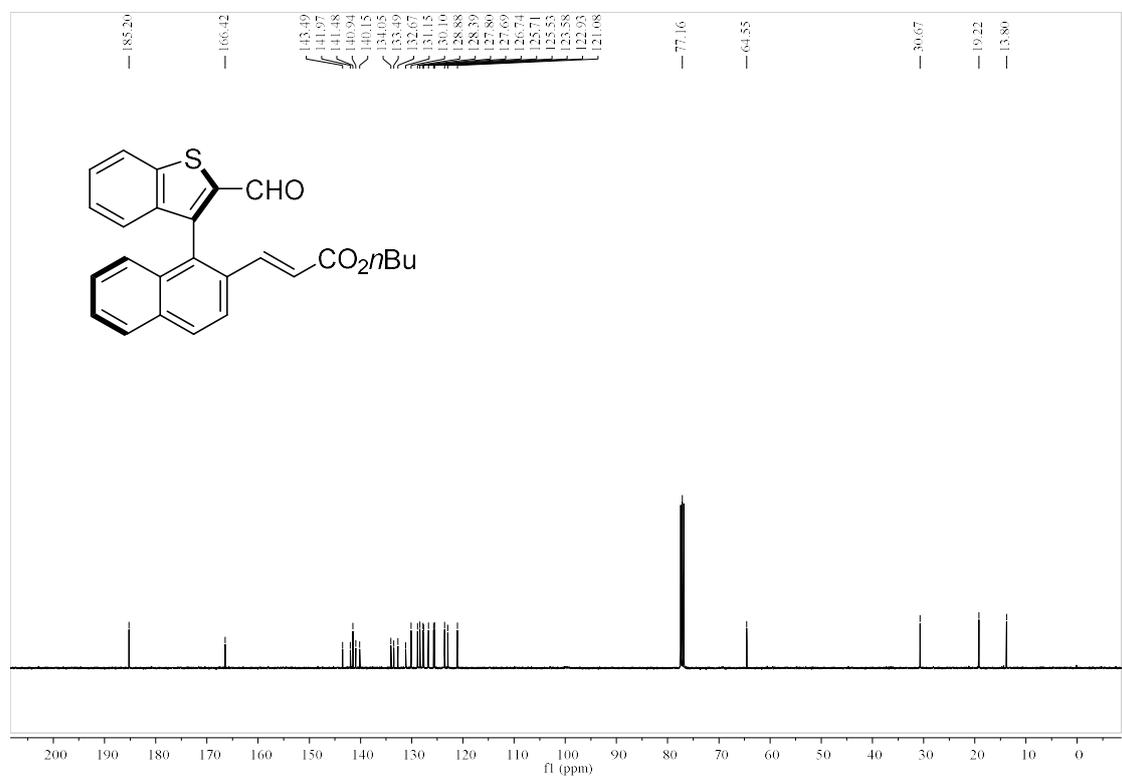


Figure S16 ¹³C NMR Spectrum of 5a in CDCl₃

5b

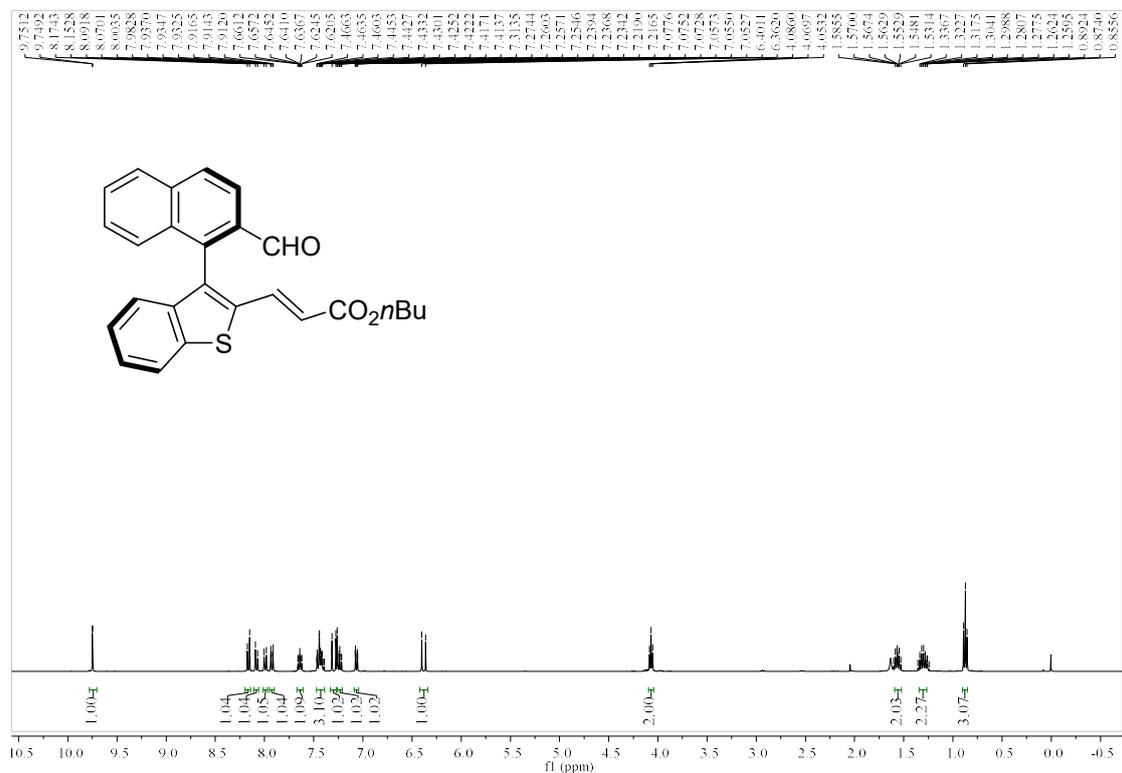


Figure S17 ¹H NMR Spectrum of 5b in CDCl₃

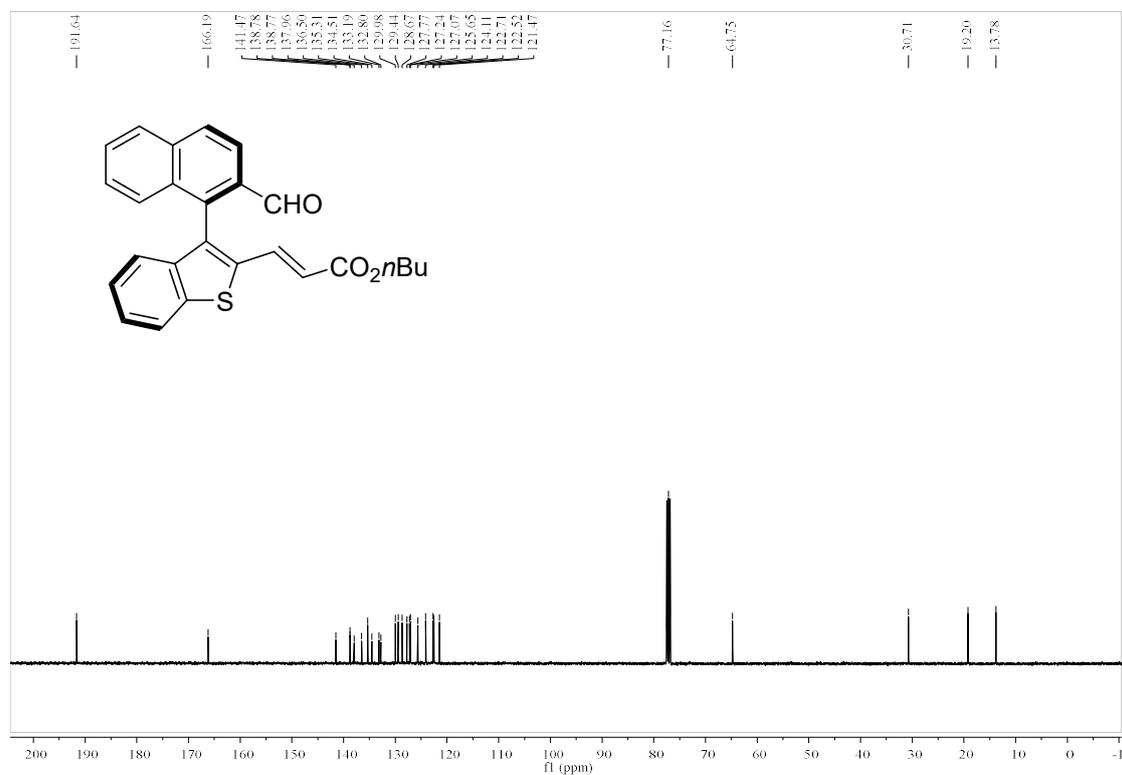


Figure S18 ¹³C NMR Spectrum of 5b in CDCl₃

5c

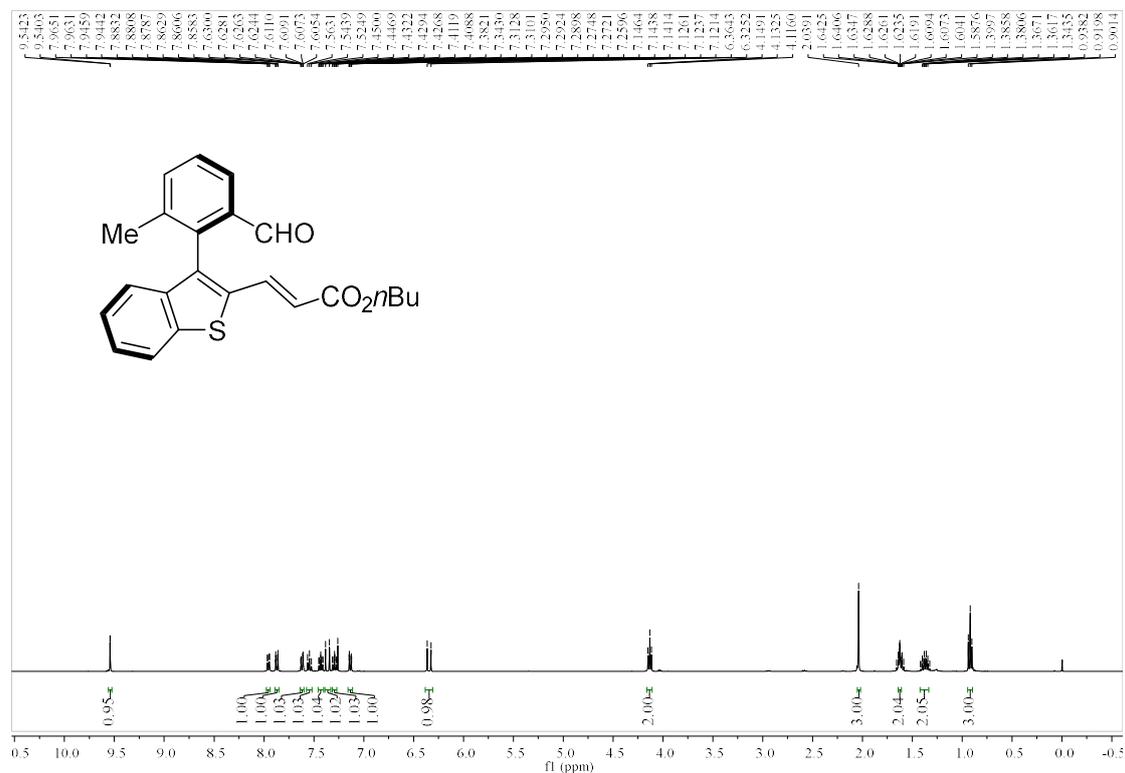


Figure S19 ¹H NMR Spectrum of 5c in CDCl₃

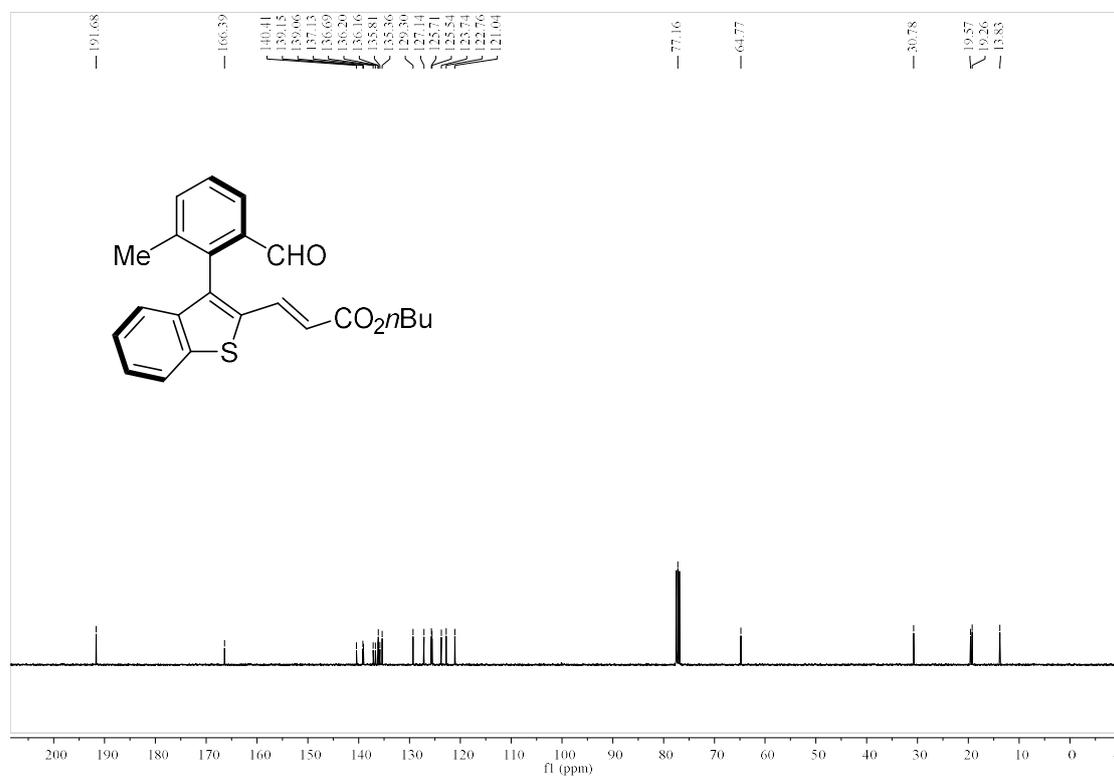


Figure S20 ¹³C NMR Spectrum of 5c in CDCl₃

5d

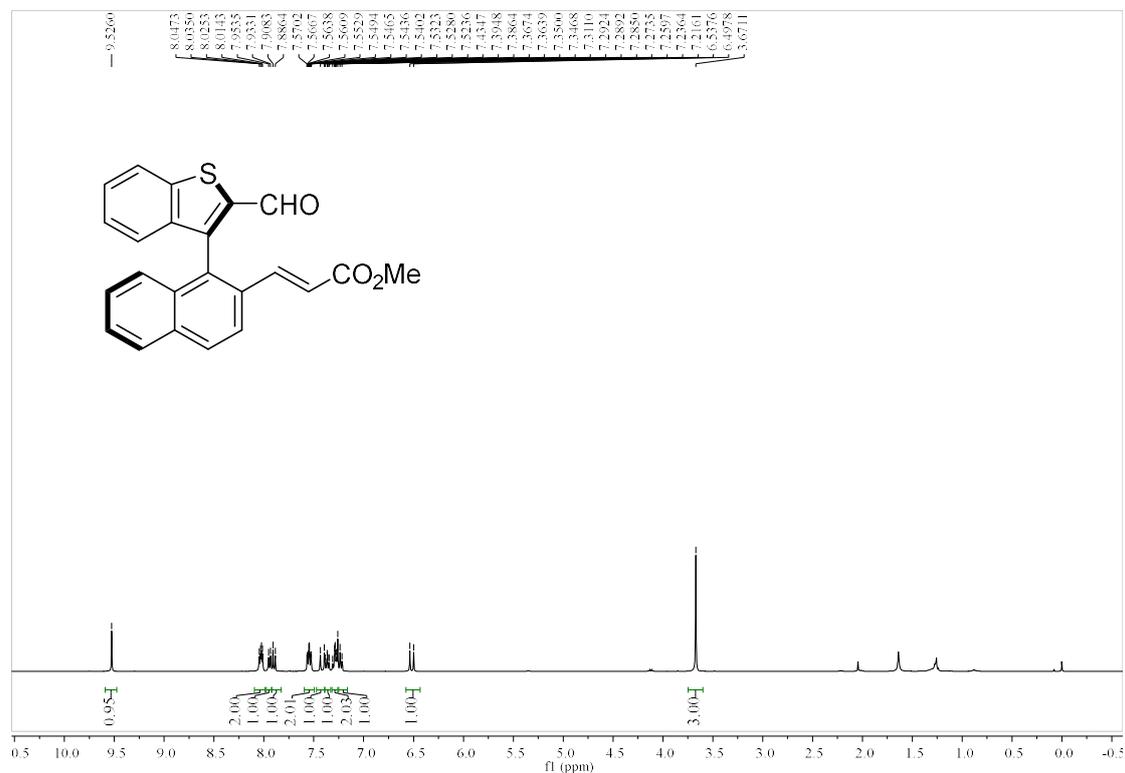


Figure S21 ¹H NMR Spectrum of 5d in CDCl₃

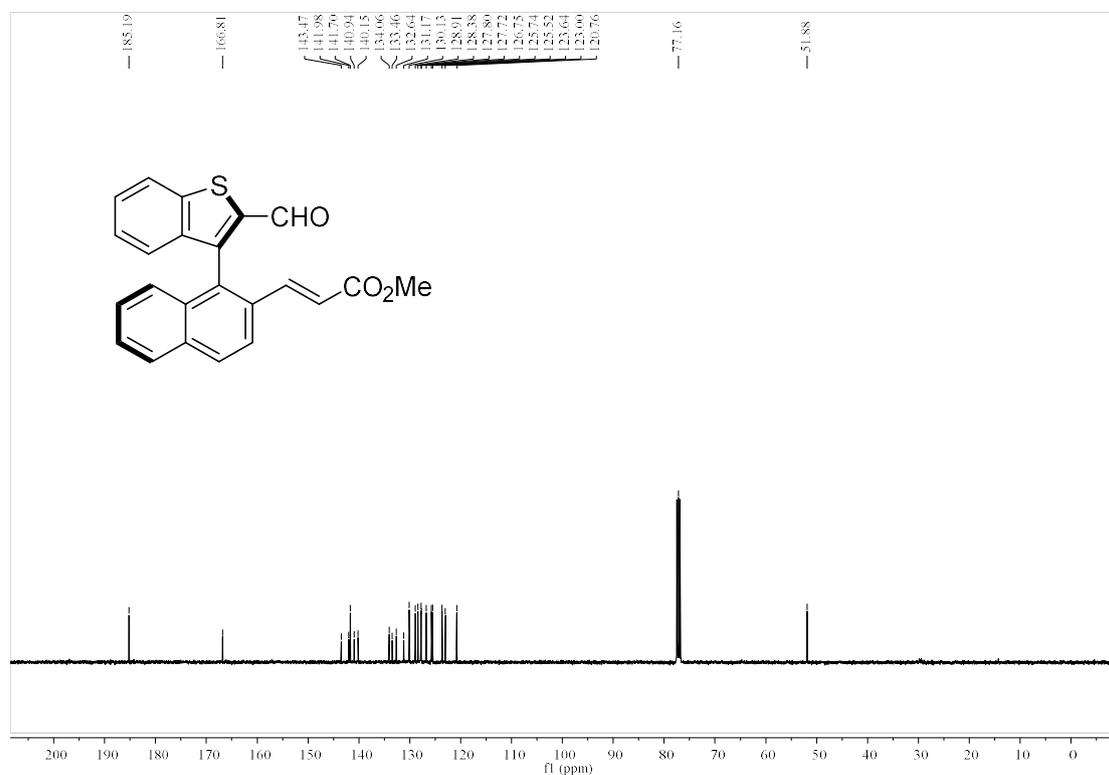


Figure S22 ¹³C NMR Spectrum of 5d in CDCl₃

5e

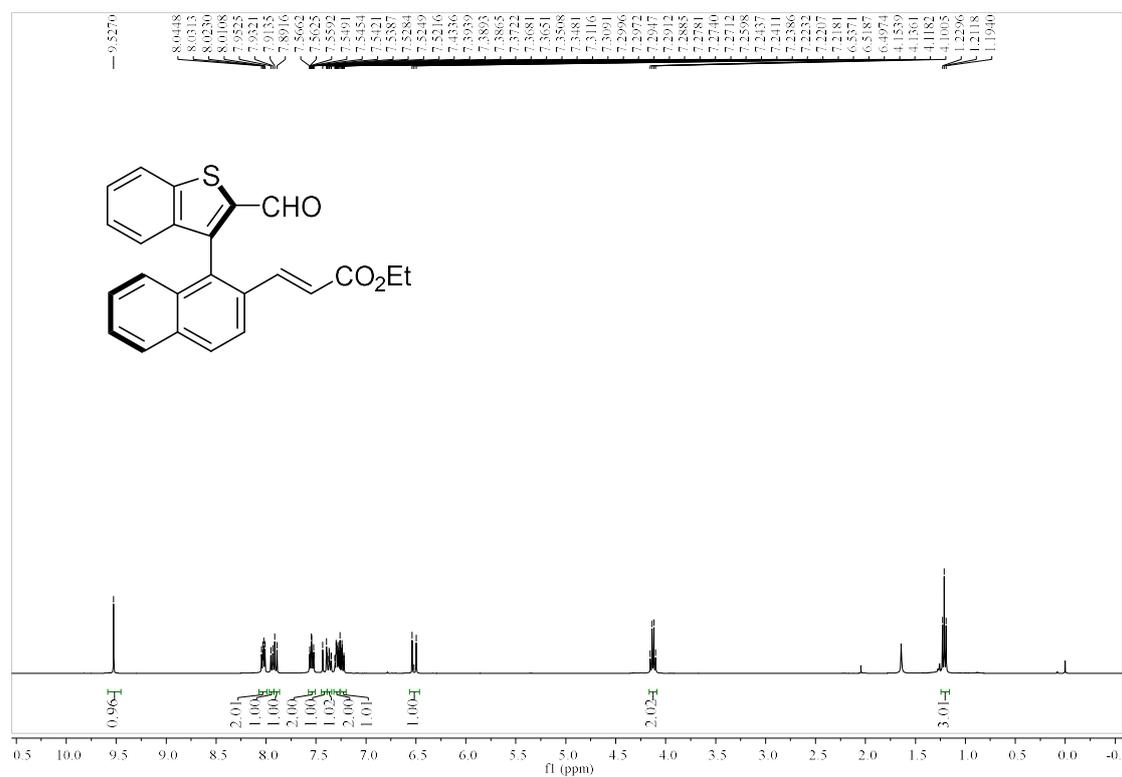


Figure S23 ¹H NMR Spectrum of 5e in CDCl₃

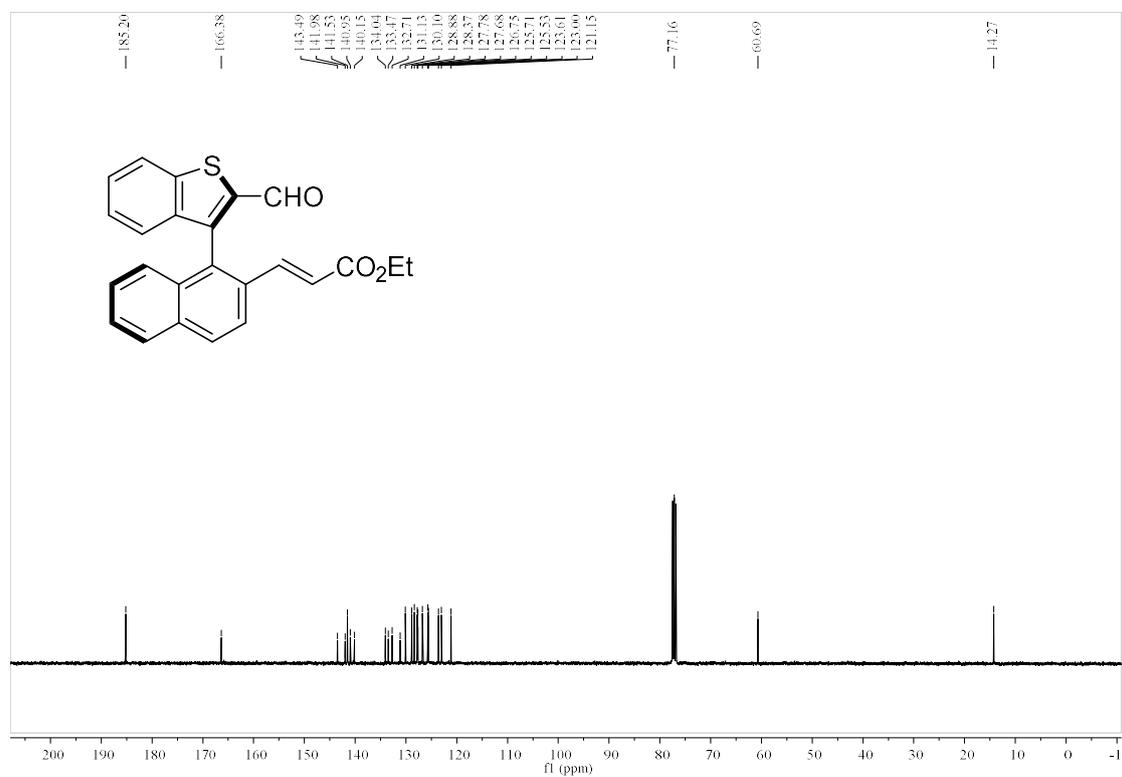


Figure S24 ¹³C NMR Spectrum of 5e in CDCl₃

5f

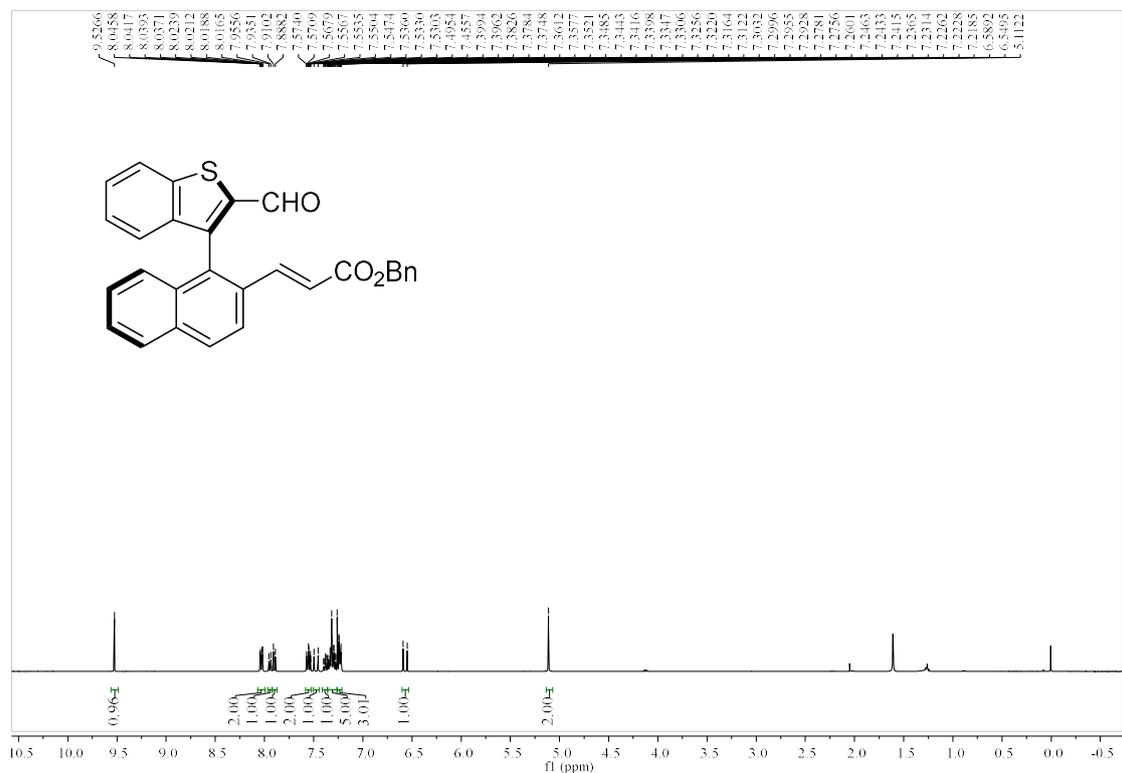


Figure S25 ¹H NMR Spectrum of 5f in CDCl₃

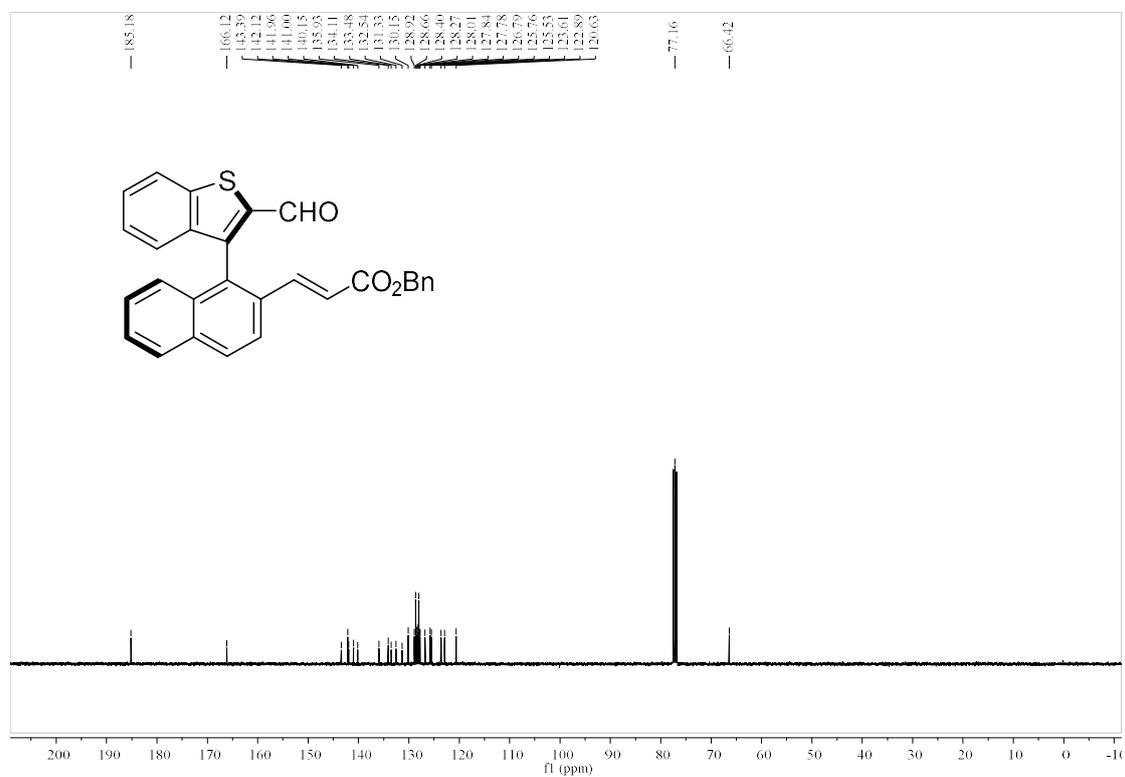


Figure S26 ¹³C NMR Spectrum of 5f in CDCl₃

5g

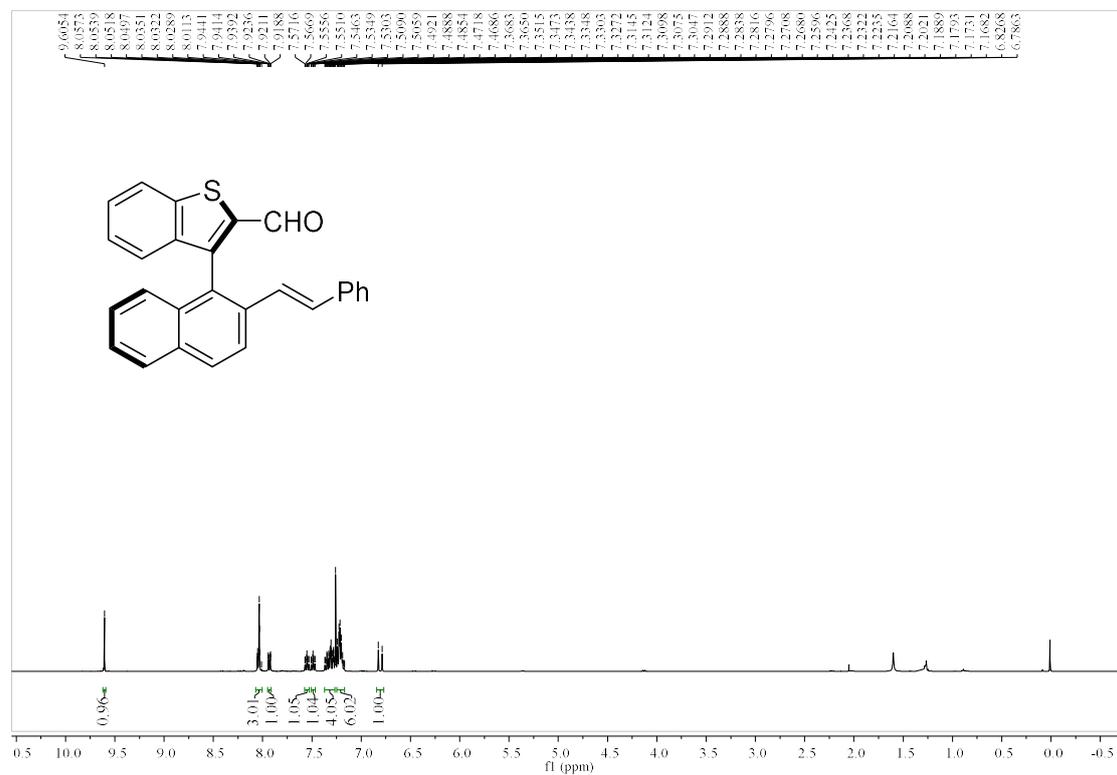


Figure S27 ¹H NMR Spectrum of 5g in CDCl₃

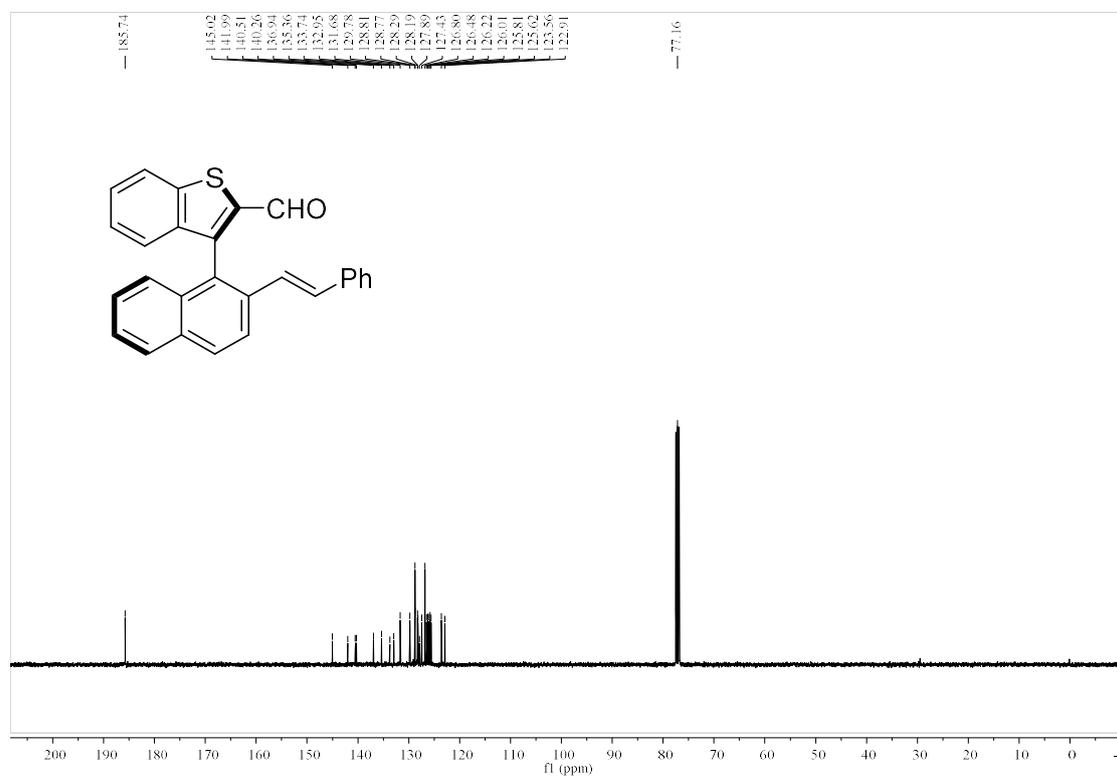


Figure S28 ¹³C NMR Spectrum of 5g in CDCl₃

5h

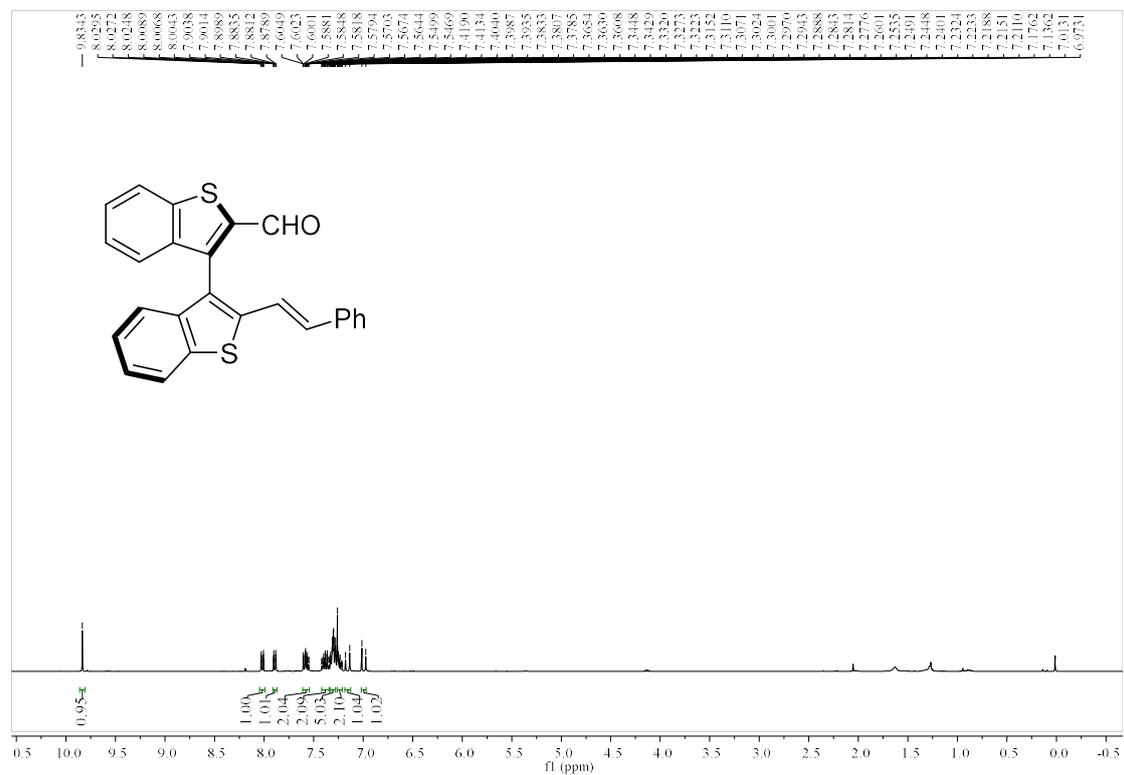


Figure S29 ¹H NMR Spectrum of 5h in CDCl₃

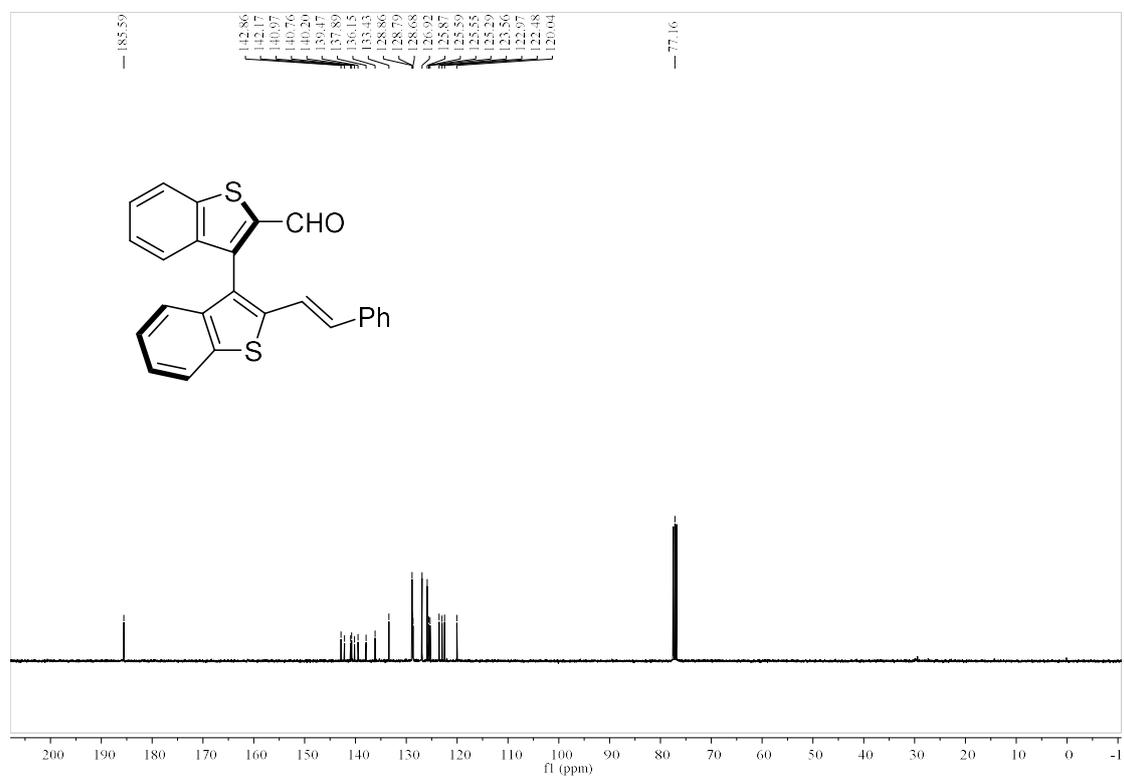


Figure S30 ¹³C NMR Spectrum of 5h in CDCl₃

6a

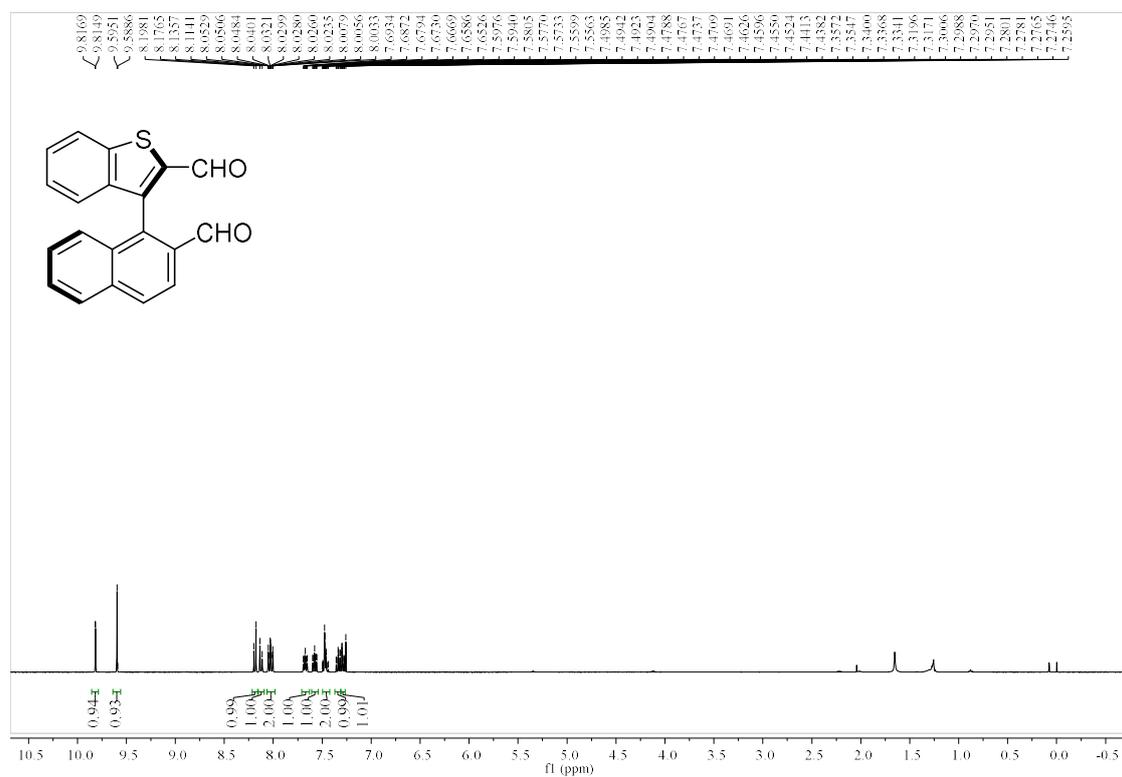


Figure S33 ¹H NMR Spectrum of 6a in CDCl₃

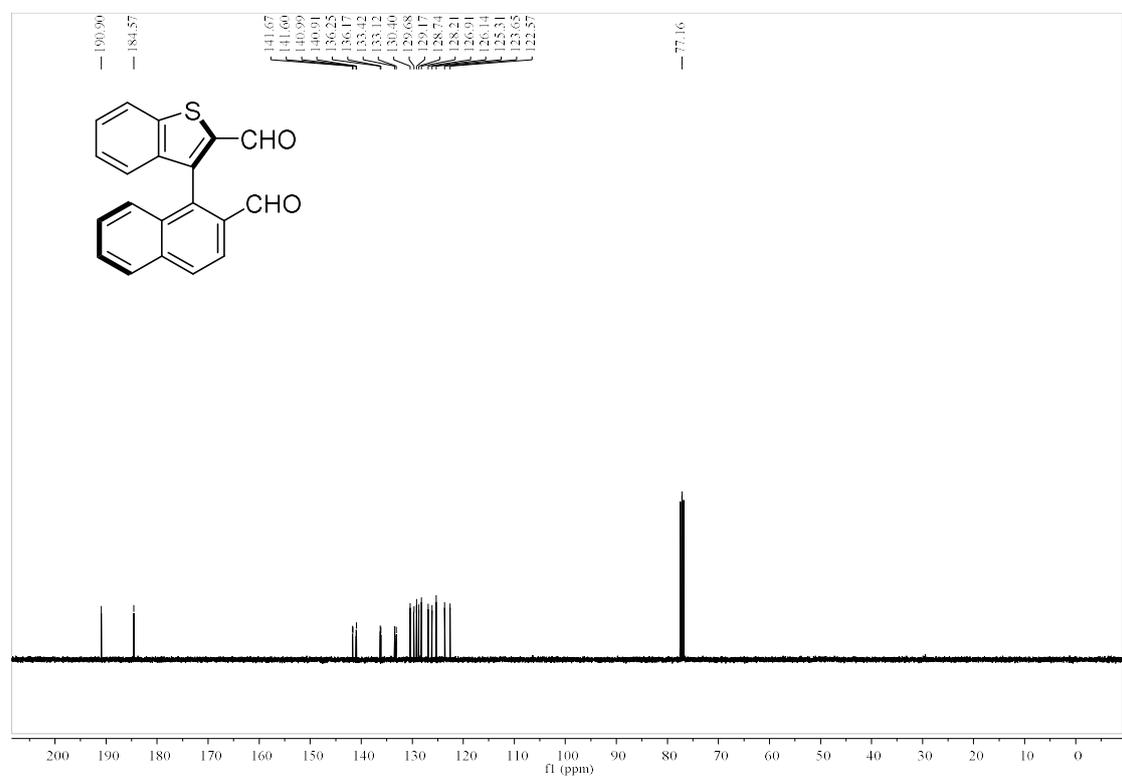


Figure S34 ¹³C NMR Spectrum of 6a in CDCl₃

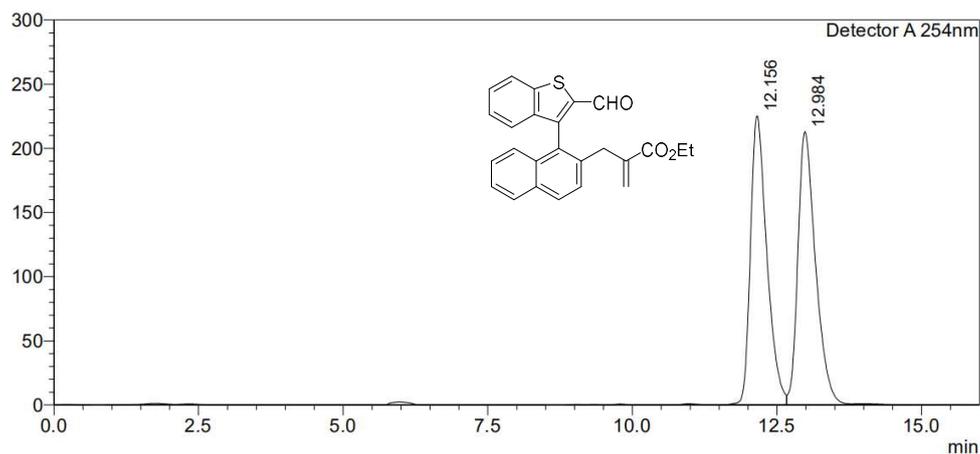
3. Copies of HPLC analysis

3a: AD-H, Hex/*i*PrOH = 92:8, rate = 0.5 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

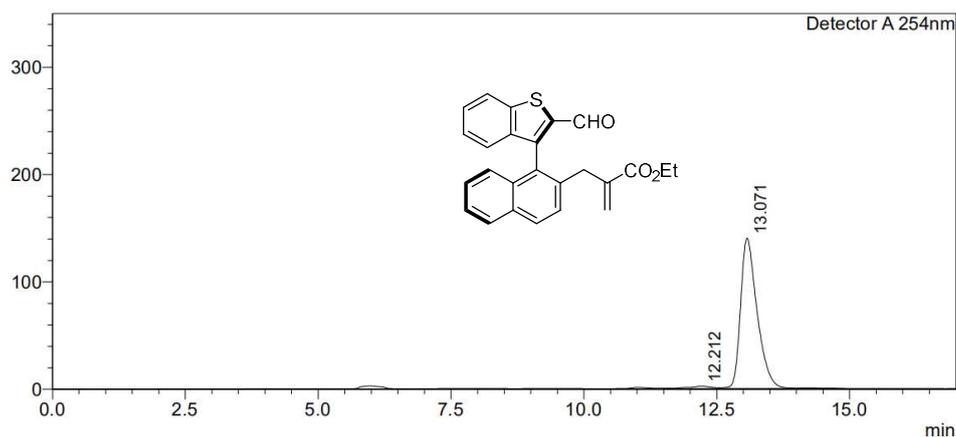
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.156	4466475	225357	49.445			
2	12.984	4566760	213059	50.555		V	
Total		9033235	438416				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.212	66720	2261	2.147			
2	13.071	3041550	139873	97.853		V	
Total		3108270	142134				

Figure S35 HPLC Data of 3a

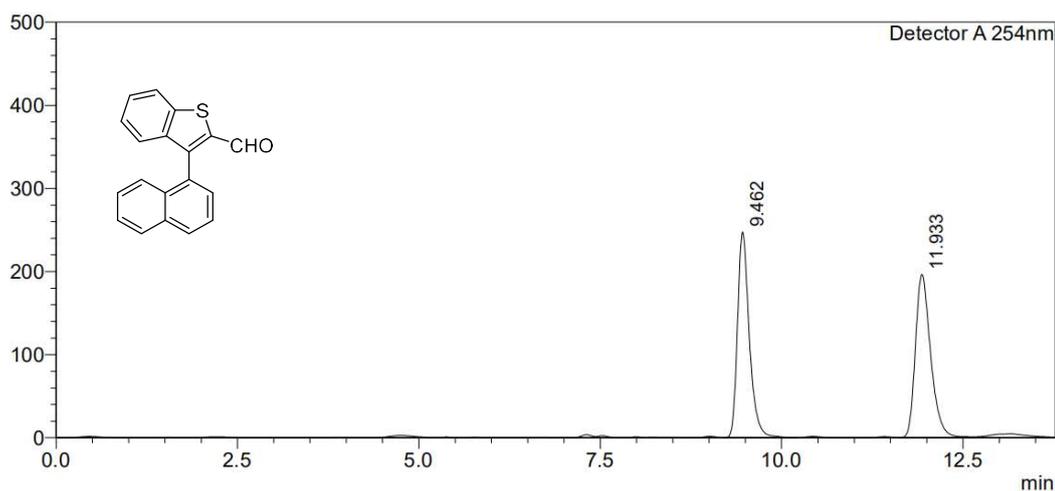
Table S6, entry 1

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

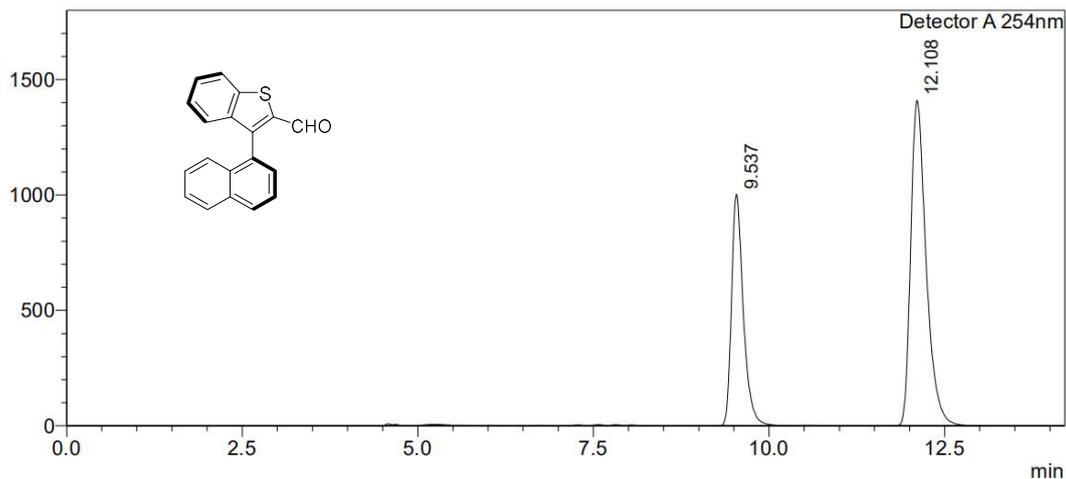
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

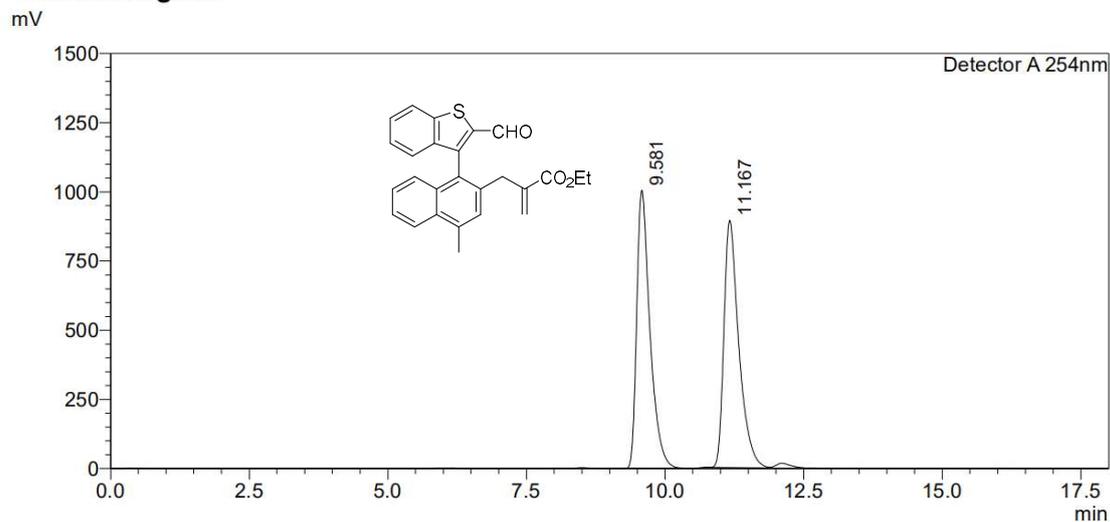
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.537	11732020	1001672	35.249		M	
2	12.108	21551142	1409057	64.751		M	
Total		33283163	2410729				

Figure S36 HPLC Data of 1a in Table 5, entry 1

3b: AD-H, Hex/*i*PrOH = 98:2, rate = 0.5 mL/min, 254 nm

Racemate

<Chromatogram>

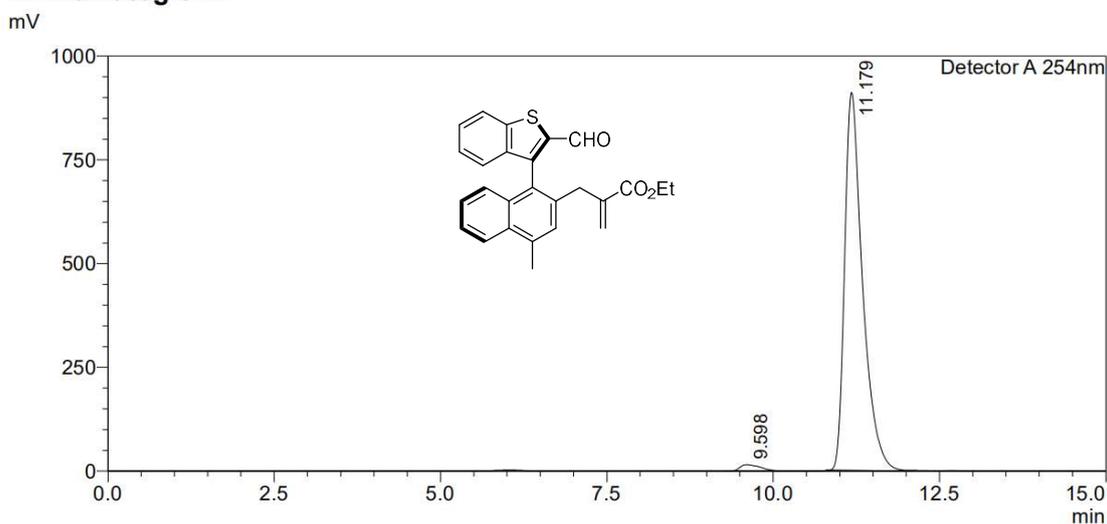


<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.581	16553048	1003566	49.854		M	
2	11.167	16649875	892536	50.146		M	
Total		33202922	1896103				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.598	386630	15734	2.203			
2	11.179	17160846	910371	97.797		M	
Total		17547476	926105				

Figure S37 HPLC Data of 3b

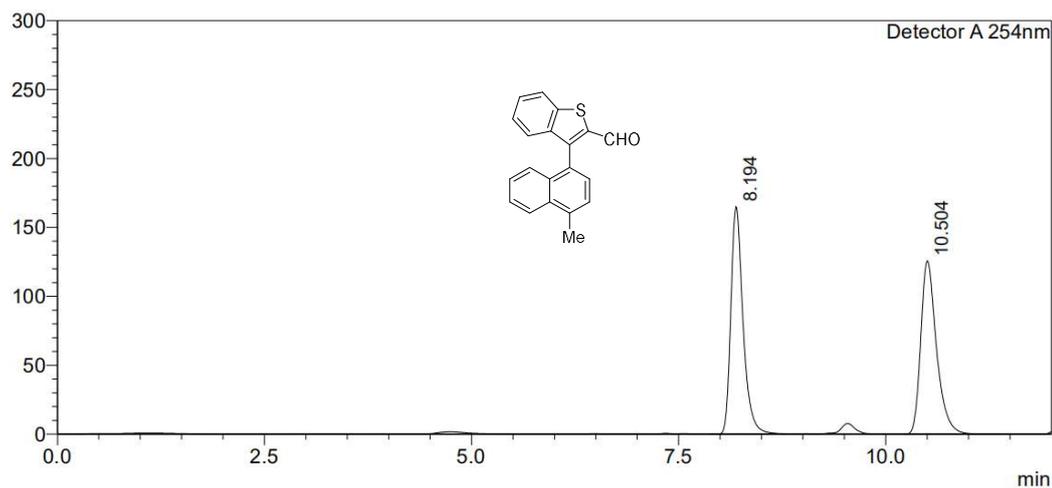
Table S6, entry 2

1b: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

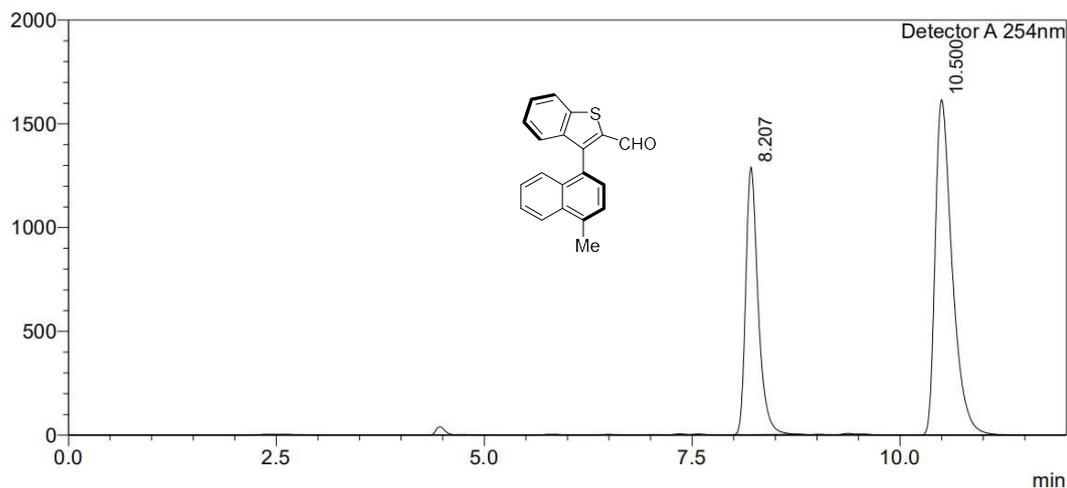
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.194	1682375	164946	50.100		M	
2	10.504	1675628	125809	49.900			
Total		3358003	290756				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.207	13297289	1287980	36.577		M	
2	10.500	23056879	1615839	63.423		M	
Total		36354168	2903819				

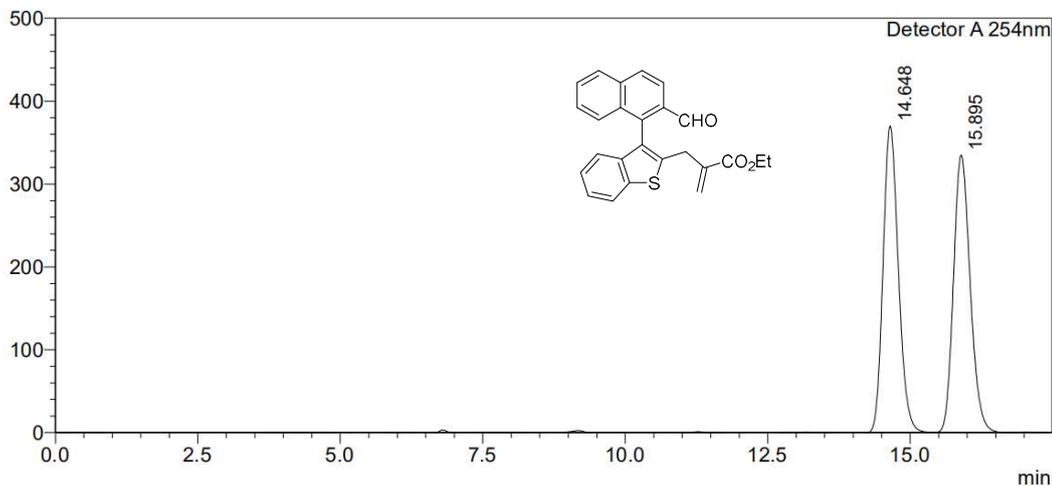
Figure S38 HPLC Data of **1b** Table 5, entry 2

3c: AD-H, Hex/*i*PrOH = 95:5, rate = 0.7 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

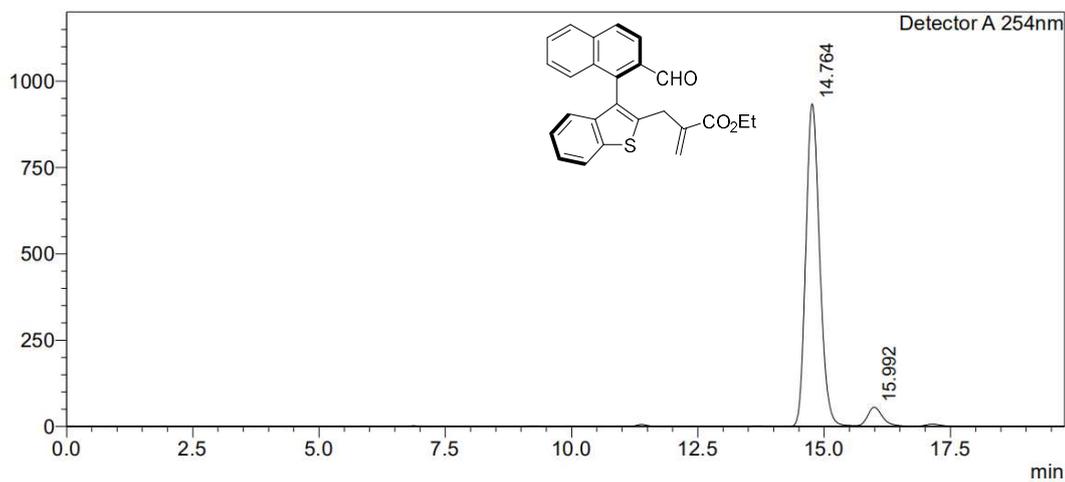
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.648	6936580	370469	50.125			
2	15.895	6902069	335457	49.875		V	
Total		13838649	705926				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.764	17818081	933939	93.830			
2	15.992	1171616	55456	6.170		V	
Total		18989698	989395				

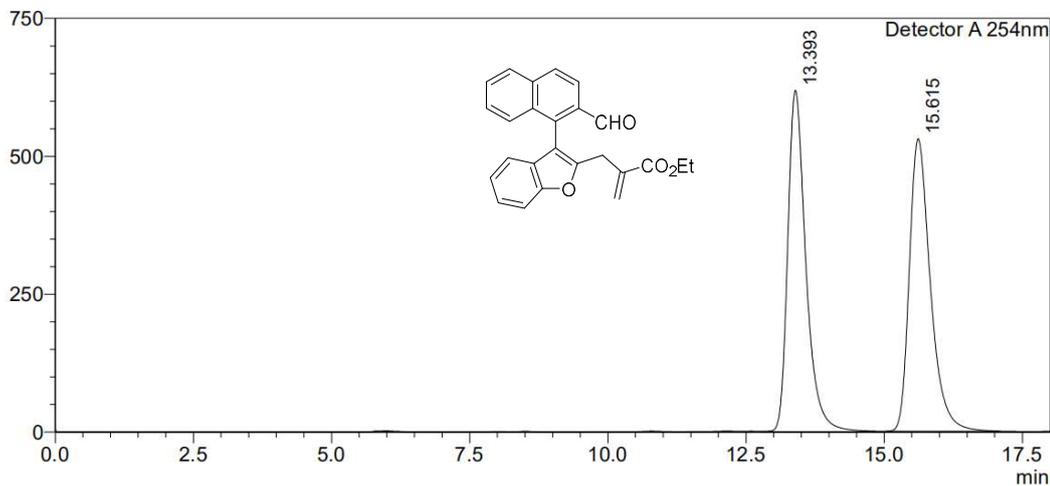
Figure S39 HPLC Data of 3c

3d: OD-H, Hex/*i*PrOH = 92:8, rate = 0.2 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

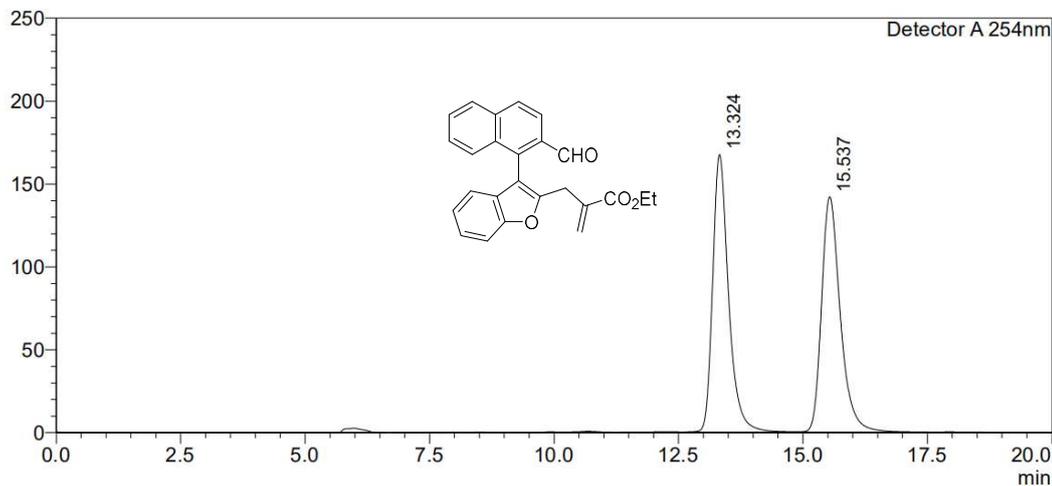
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.393	14127896	618723	50.120			
2	15.615	14060346	530413	49.880		M	
Total		28188241	1149136				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.324	3714710	167293	50.017			
2	15.537	3712210	141850	49.983		V	
Total		7426920	309143				

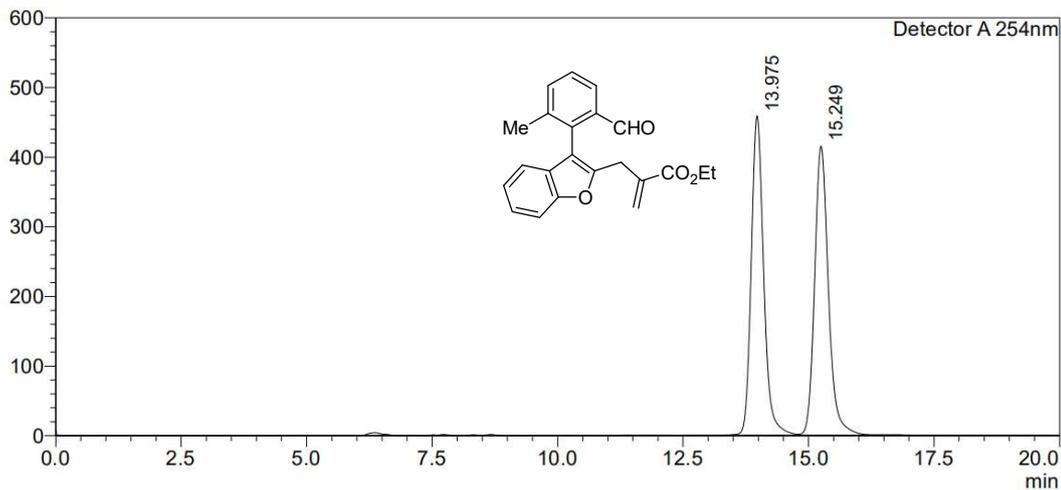
Figure S40 HPLC Data of 3d

3e: IC, Hex/*i*PrOH = 98:2, rate = 0.5 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

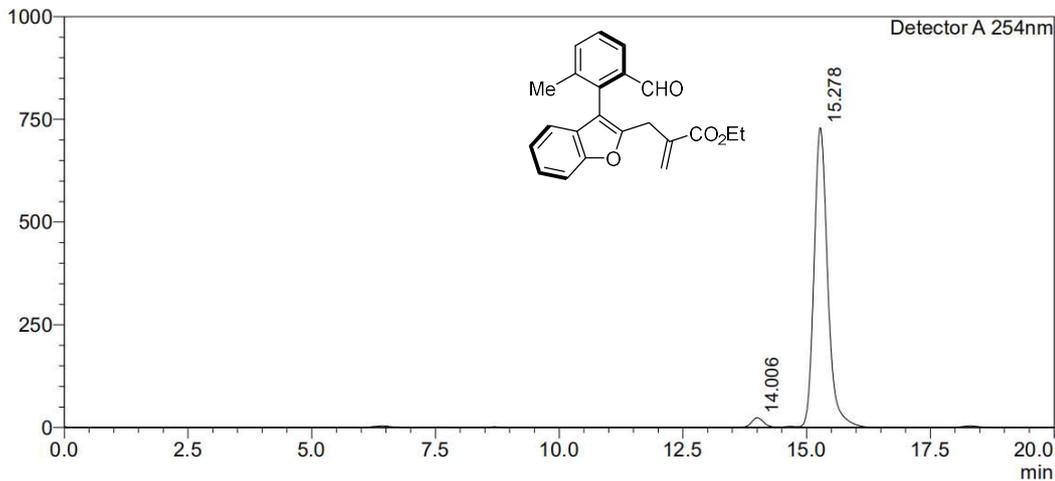
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.975	7882515	457767	49.778			
2	15.249	7952923	414538	50.222		V	
Total		15835437	872304				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

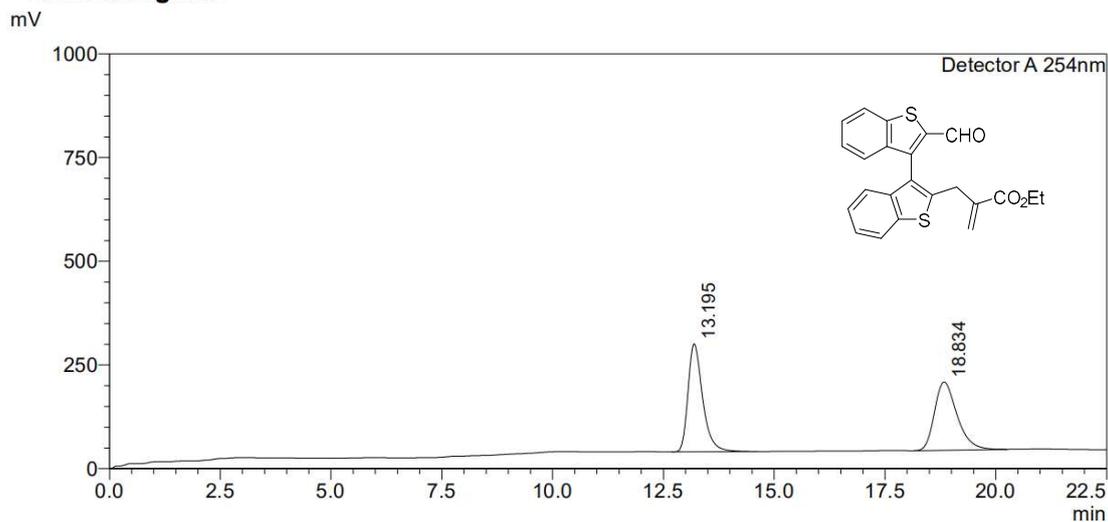
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.006	426710	24038	2.944			
2	15.278	14069788	729444	97.056			
Total		14496498	753482				

Figure S41 HPLC Data of 3e

3f: OD-H, Hex/*i*PrOH = 98:2, rate = 0.5 mL/min, 254 nm

Racemate

<Chromatogram>



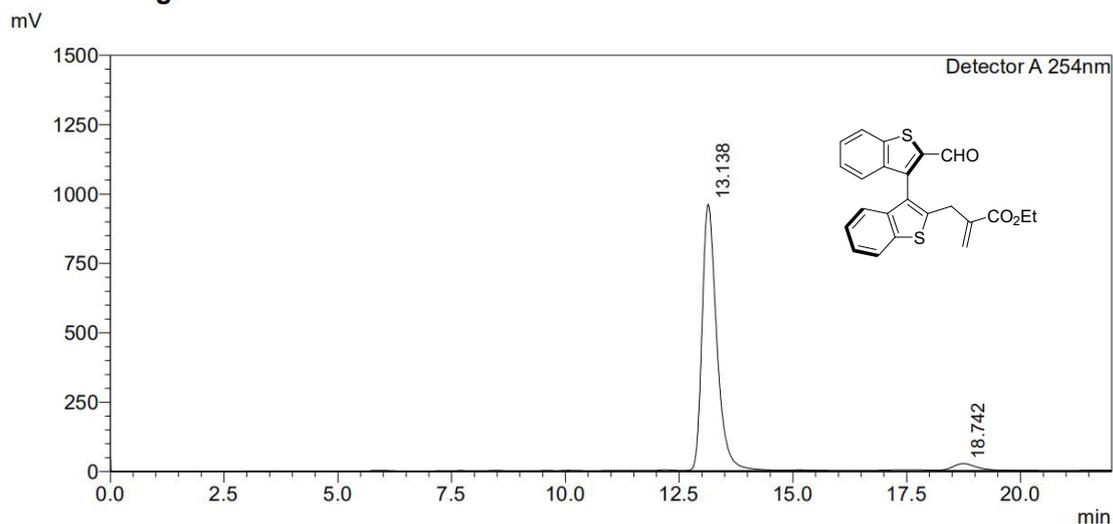
<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.195	6008636	260180	50.798			
2	18.834	5819882	164379	49.202			
Total		11828518	424558				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.138	21541870	958944	96.144			
2	18.742	864016	24538	3.856			
Total		22405887	983482				

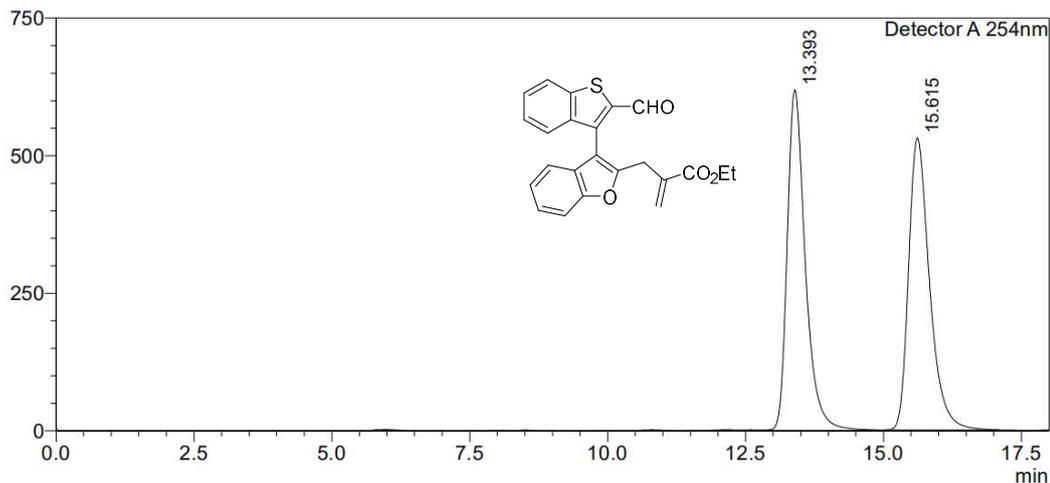
Figure S42 HPLC Data of 3f

3g: OD-H, Hex/*i*PrOH = 98:2, rate = 0.4 mL/min, 254 nm

Racemate

<Chromatogram>

mV



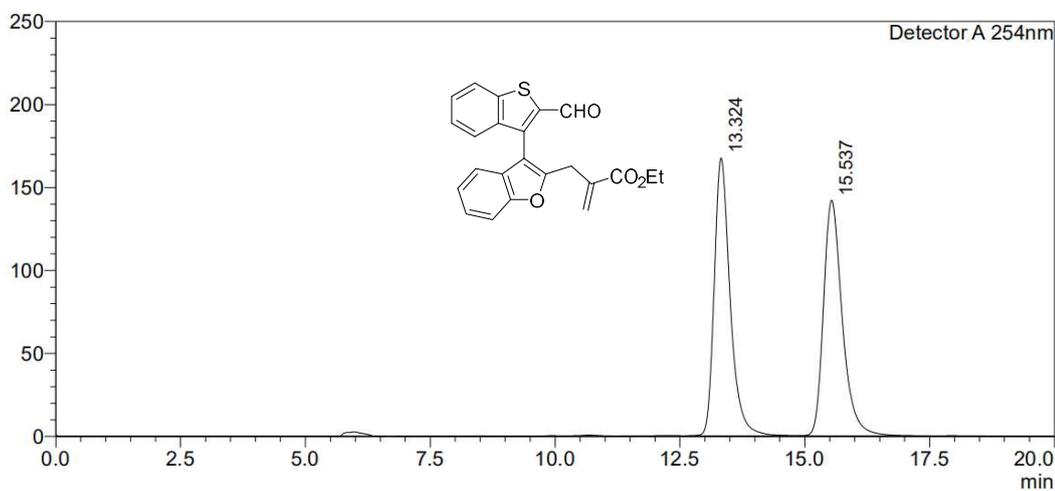
<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.393	14127896	618723	50.120			
2	15.615	14060346	530413	49.880		M	
Total		28188241	1149136				

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.324	3714710	167293	50.017			
2	15.537	3712210	141850	49.983		V	
Total		7426920	309143				

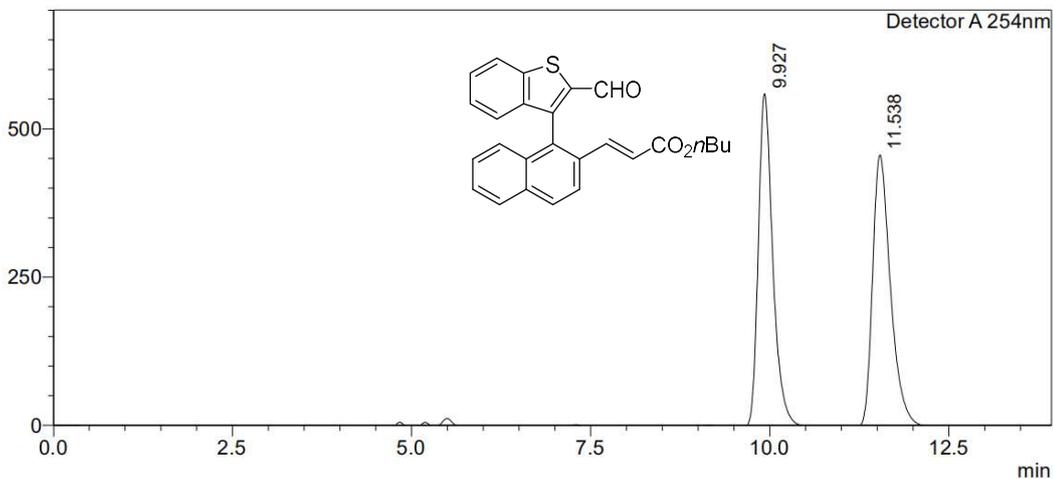
Figure S43 HPLC Data of 3g

5a: IB, Hex/*i*PrOH = 85:15, rate = 0.9 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

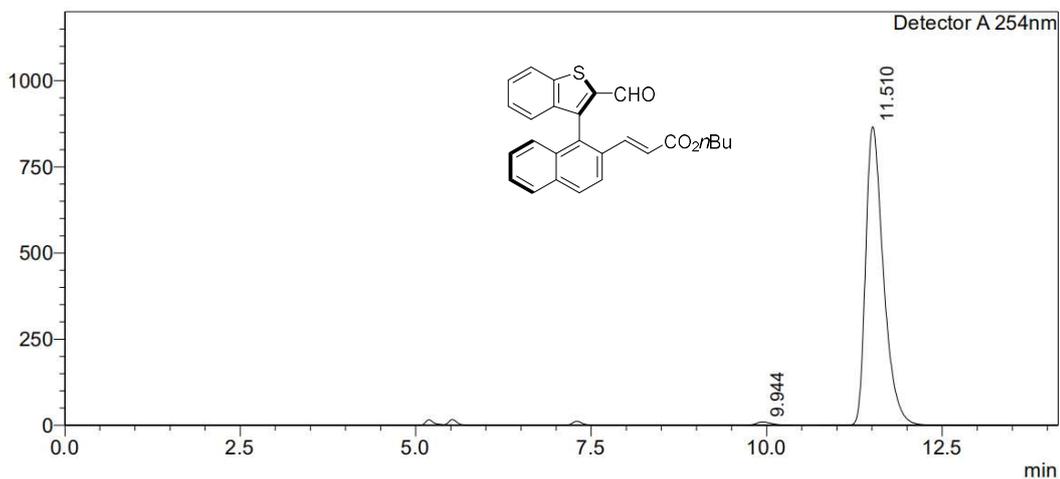
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.927	7964400	562418	49.609			
2	11.538	8090103	459445	50.391			
Total		16054503	1021863				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.944	138510	9464	0.898		M	
2	11.510	15293981	866508	99.102		M	
Total		15432491	875972				

Figure S44 HPLC Data of 5a

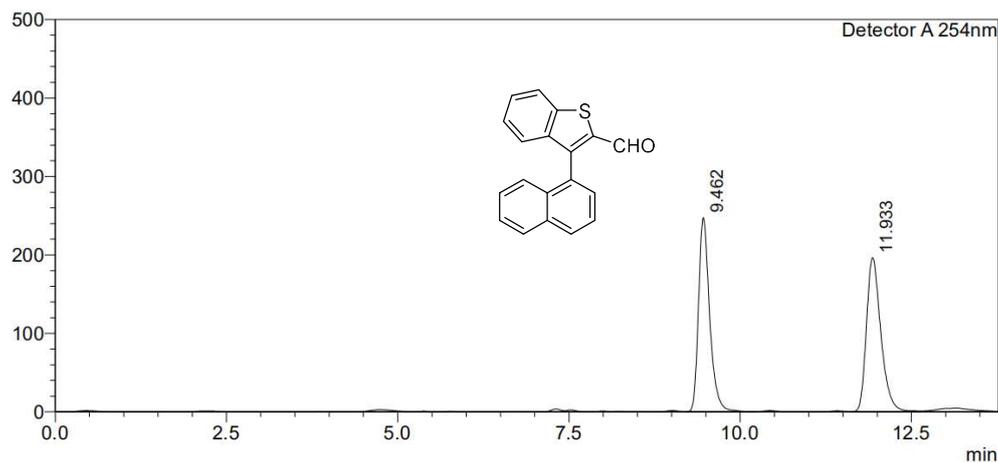
Table S7, entry 1

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

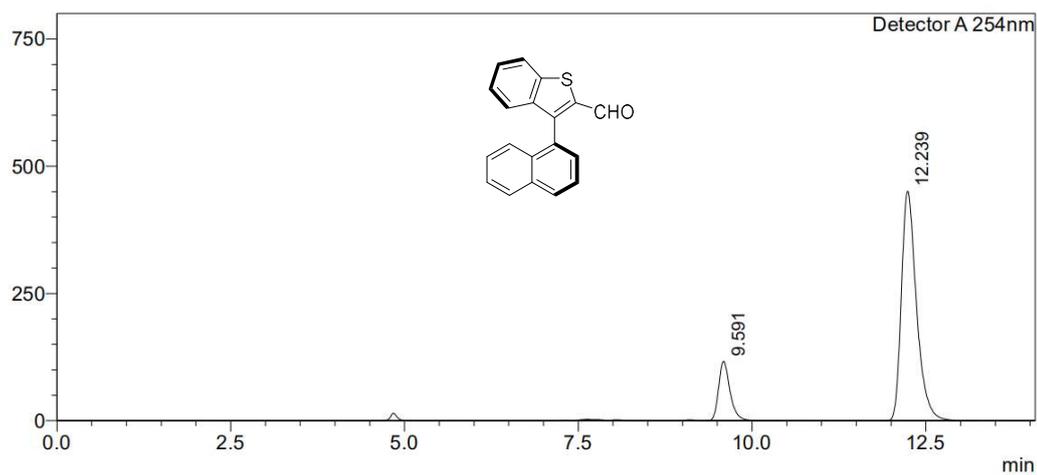
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.591	1356046	116964	16.590			
2	12.239	6817828	450940	83.410		M	
Total		8173874	567904				

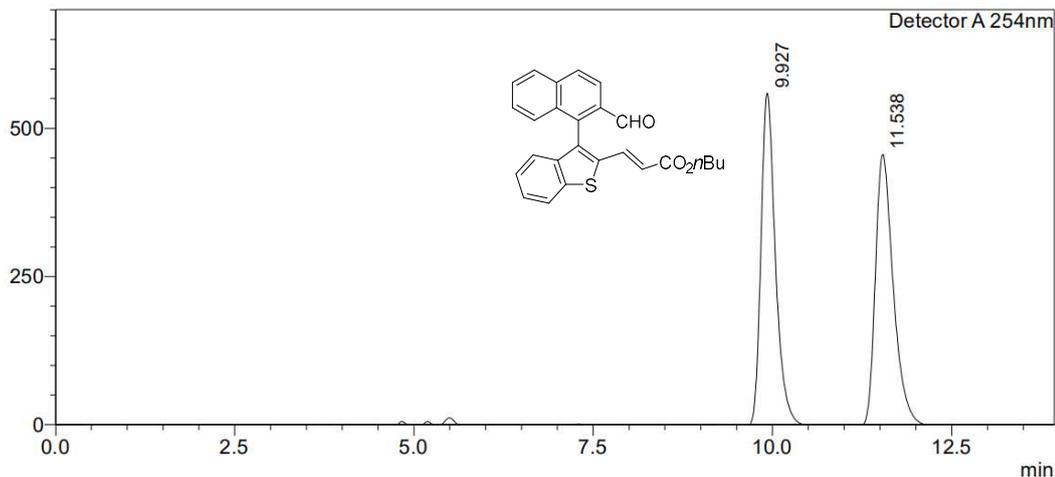
Figure S45 HPLC Data of 1a in Table 6, entry 1

5b: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

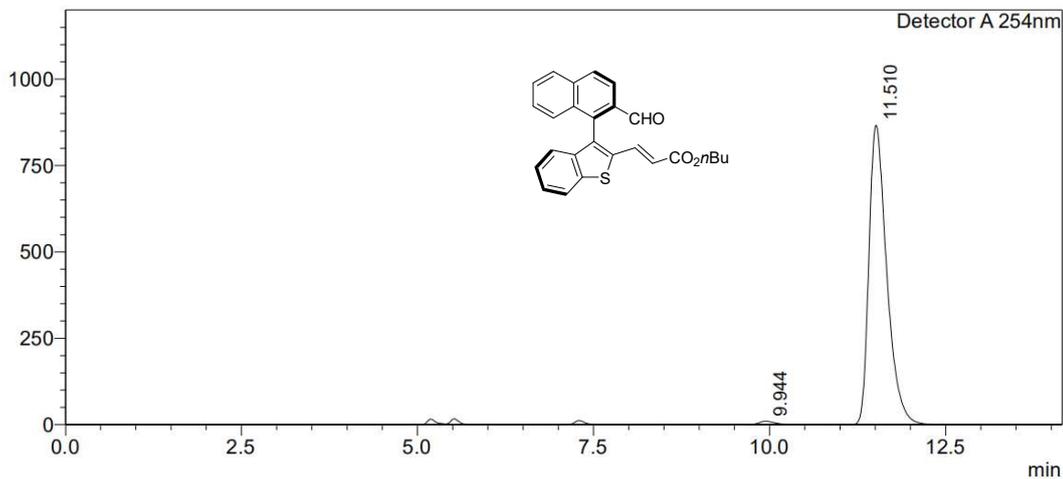
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.927	7964400	562418	49.609			
2	11.538	8090103	459445	50.391			
Total		16054503	1021863				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.944	138510	9464	0.898		M	
2	11.510	15293981	866508	99.102		M	
Total		15432491	875972				

Figure S46 HPLC Data of 5b

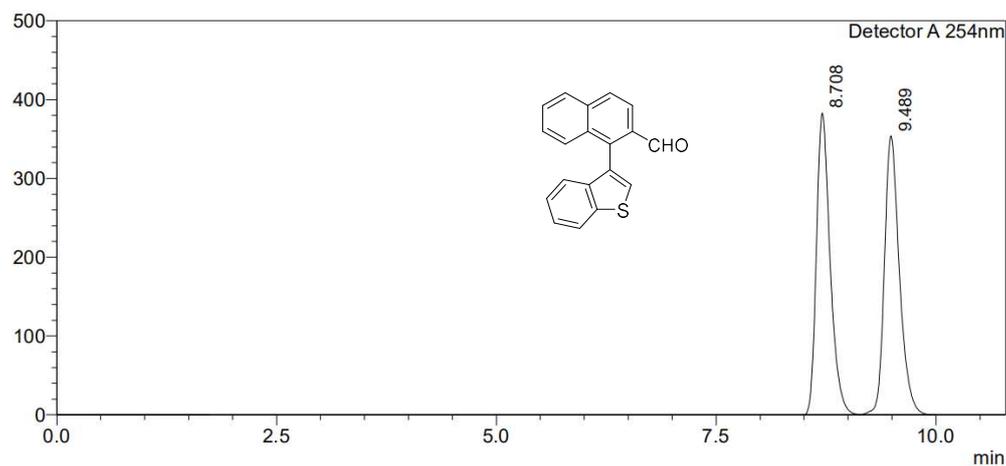
Table S7, entry 2

1b: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

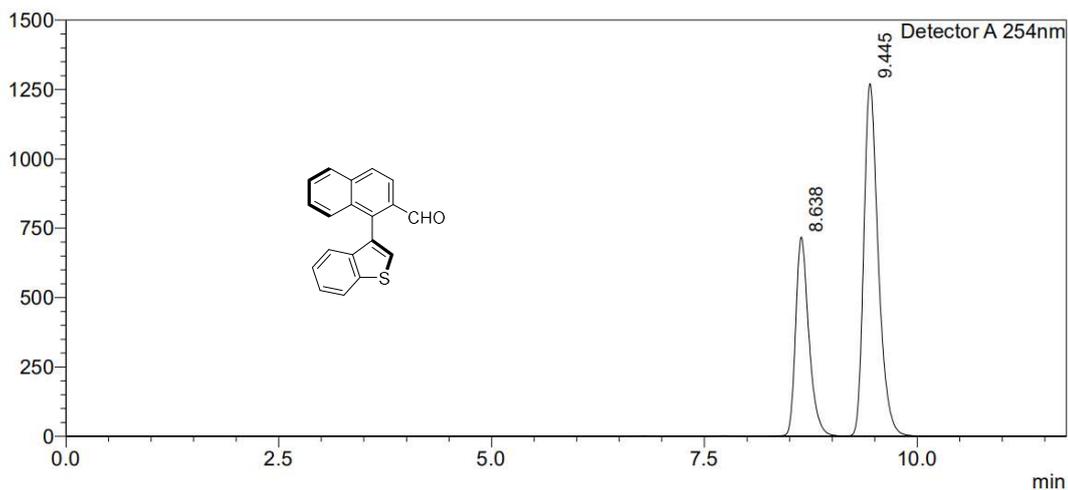
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.708	4188988	383595	49.624			
2	9.489	4252517	354527	50.376		V	
Total		8441505	738122				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.638	7789911	717703	34.061			
2	9.445	15080799	1270350	65.939		V	
Total		22870710	1988053				

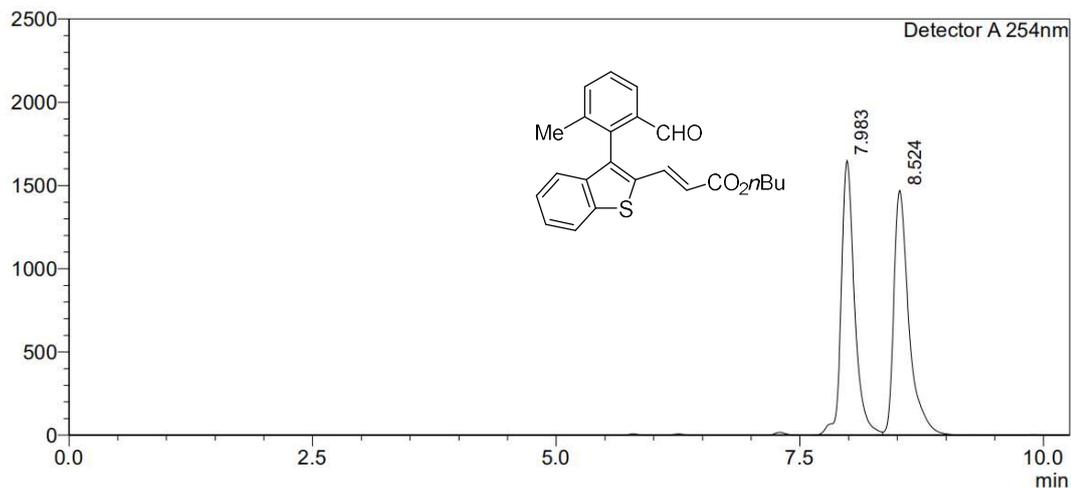
Figure S47 HPLC Data of **1b** in Table 6, entry 2

5c: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

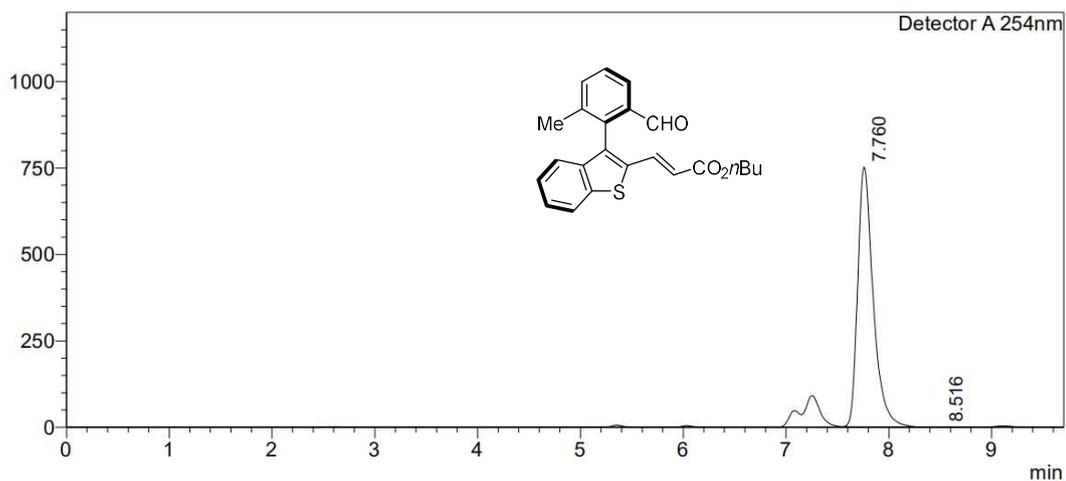
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.983	15709028	1650227	49.552			
2	8.524	15993292	1470147	50.448		V	
Total		31702321	3120373				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.760	7876941	750538	99.940		M	
2	8.516	4716	577	0.060			
Total		7881657	751115				

Figure S48 HPLC Data of 5c

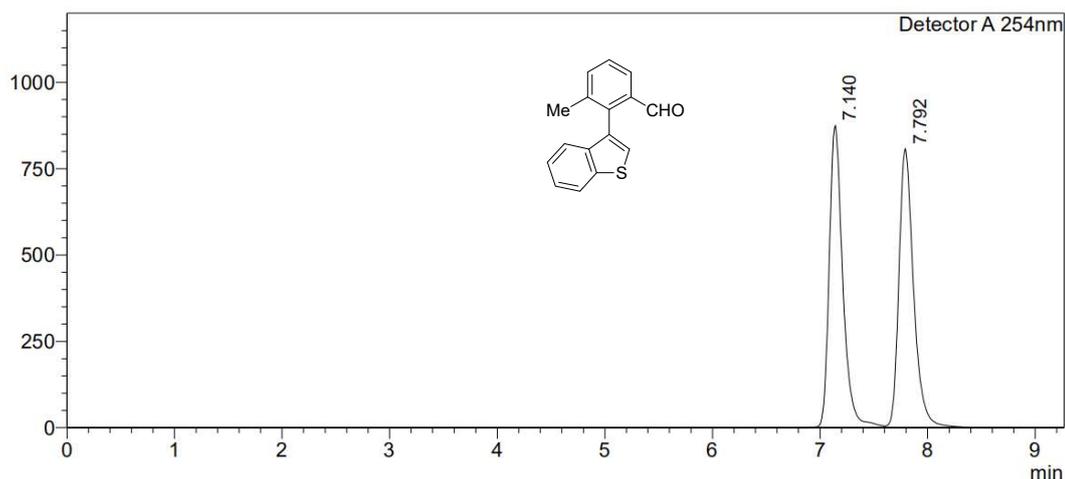
Table S7, entry 3

1c: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

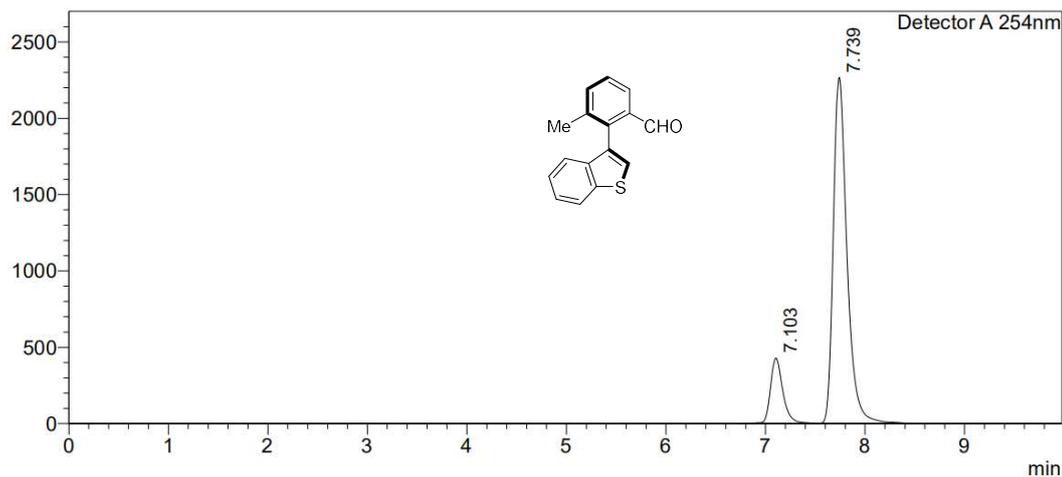
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.140	7668095	873955	50.062			
2	7.792	7649195	806709	49.938		V	
Total		15317291	1680664				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.103	3775785	430092	14.970			
2	7.739	21447091	2263303	85.030		M	
Total		25222877	2693395				

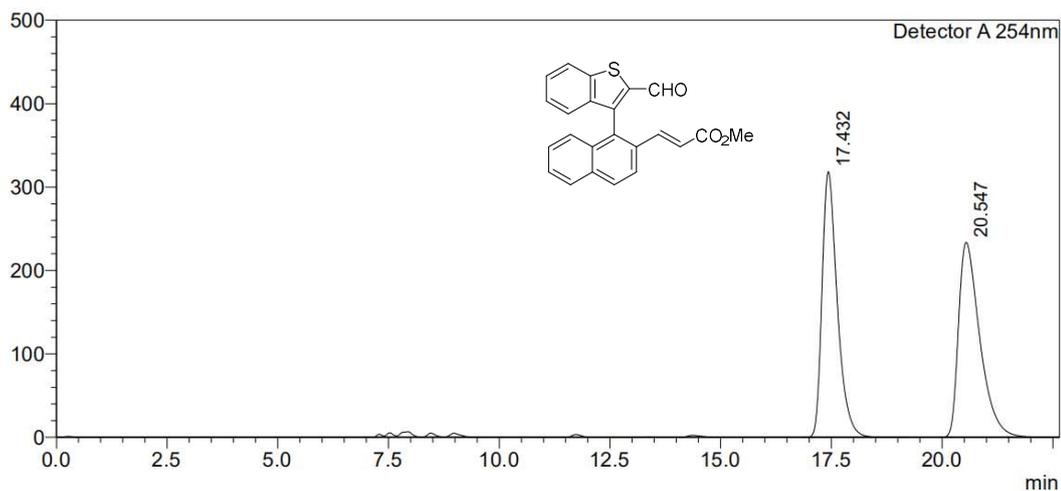
Figure 49 HPLC Data of 1c in Table 6, entry 3

5d: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

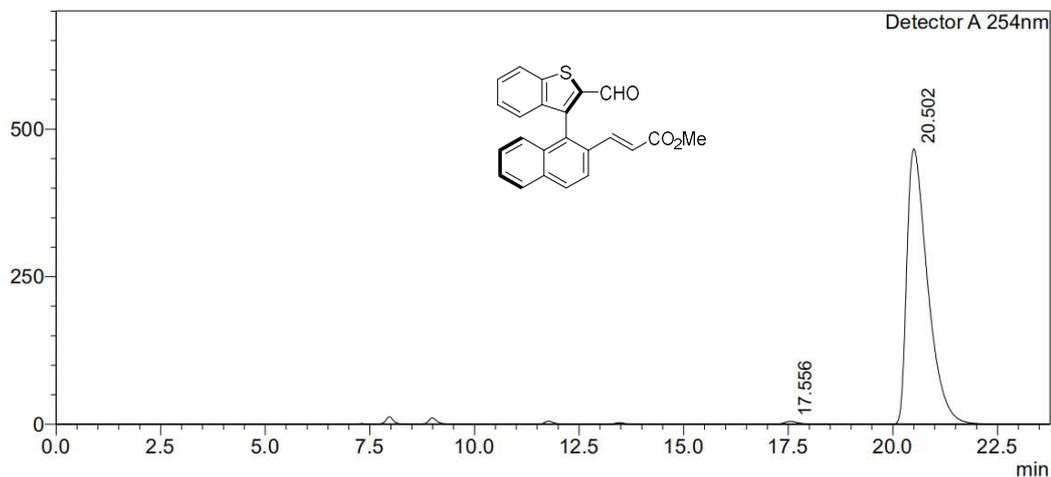
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.432	7624511	318478	49.413			
2	20.547	7805634	233543	50.587			
Total		15430145	552020				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.556	115715	4992	0.710			
2	20.502	16175828	466238	99.290			
Total		16291543	471229				

Figure S50 HPLC Data of 5d

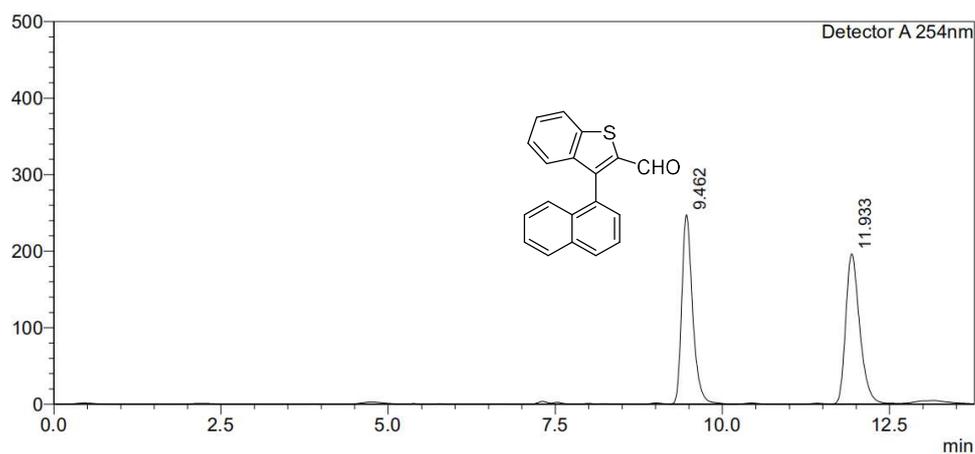
Table S7, entry 4

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

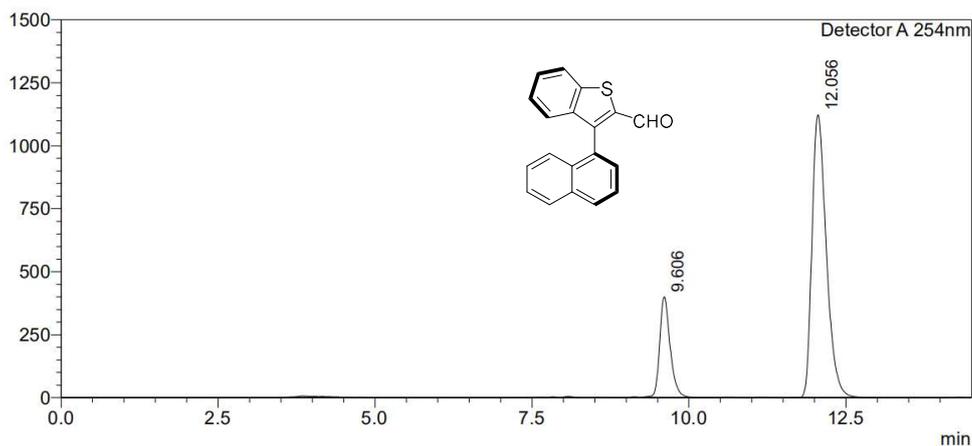
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.606	4712469	400079	20.879			
2	12.056	17857715	1121977	79.121		M	
Total		22570184	1522056				

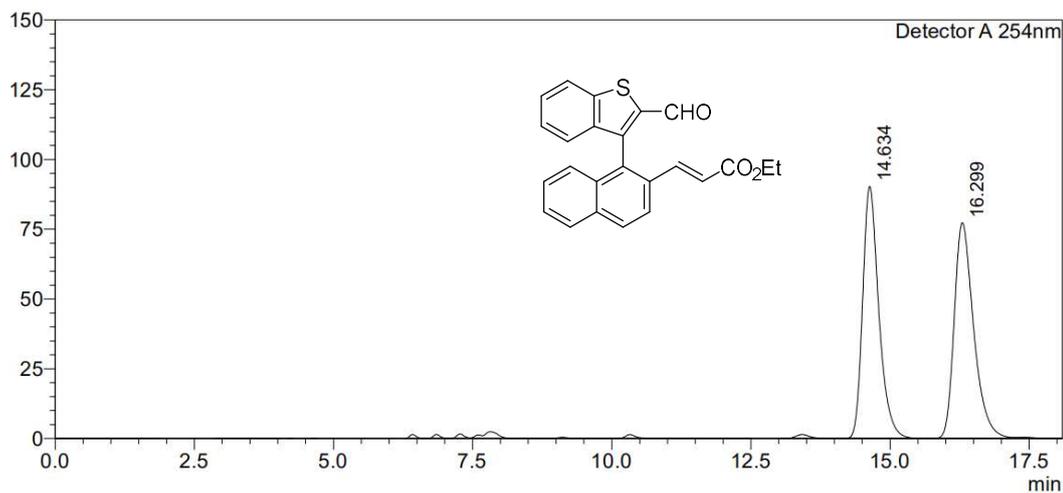
Figure S51 HPLC Data of 1a in Table 6, entry 4

5e: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

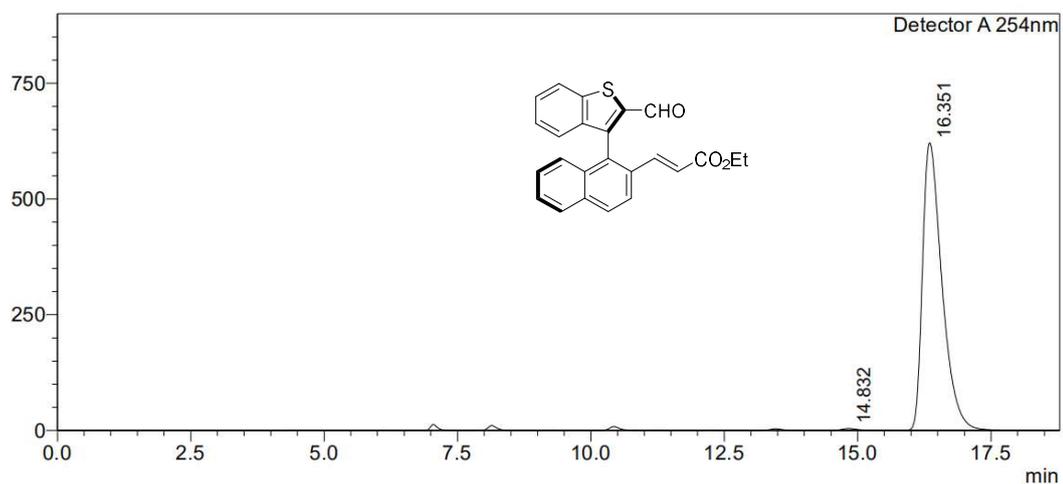
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.634	1830786	90454	49.058			
2	16.299	1901121	77460	50.942		V	
Total		3731907	167914				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.832	81128	4069	0.511			
2	16.351	15788750	621177	99.489			
Total		15869878	625246				

Figure S52 HPLC Data of 5e

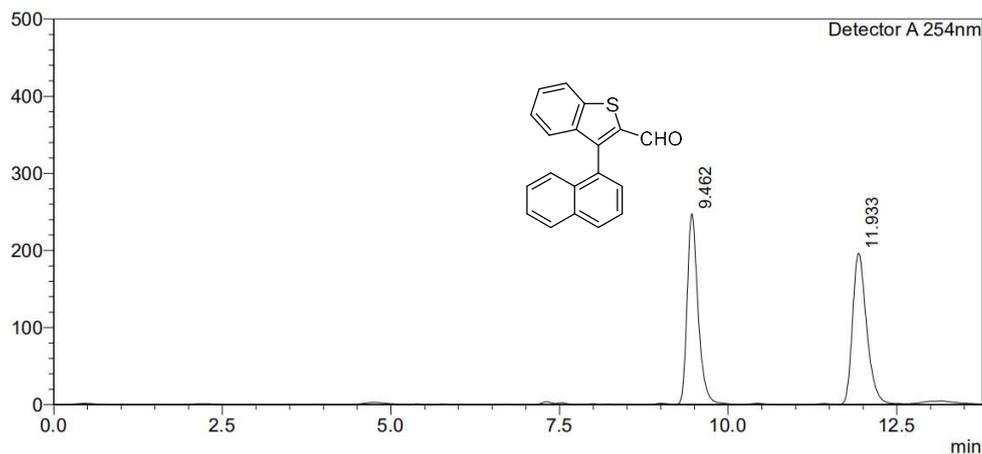
Table S7, entry 5

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

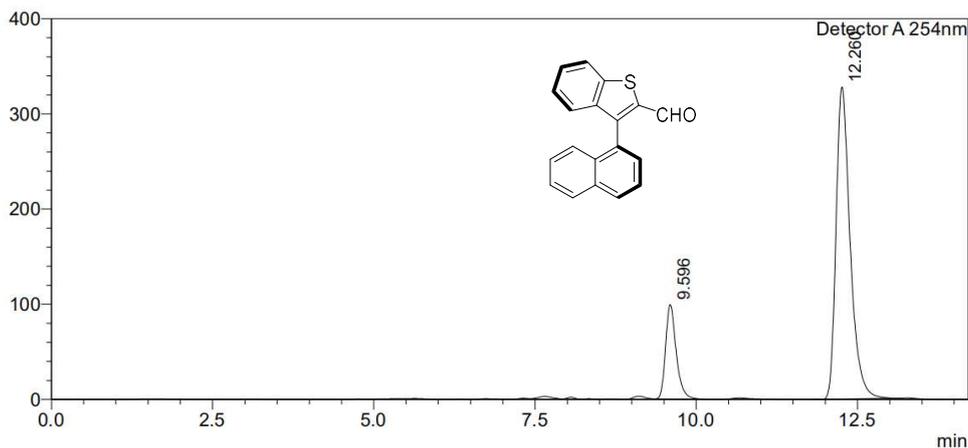
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

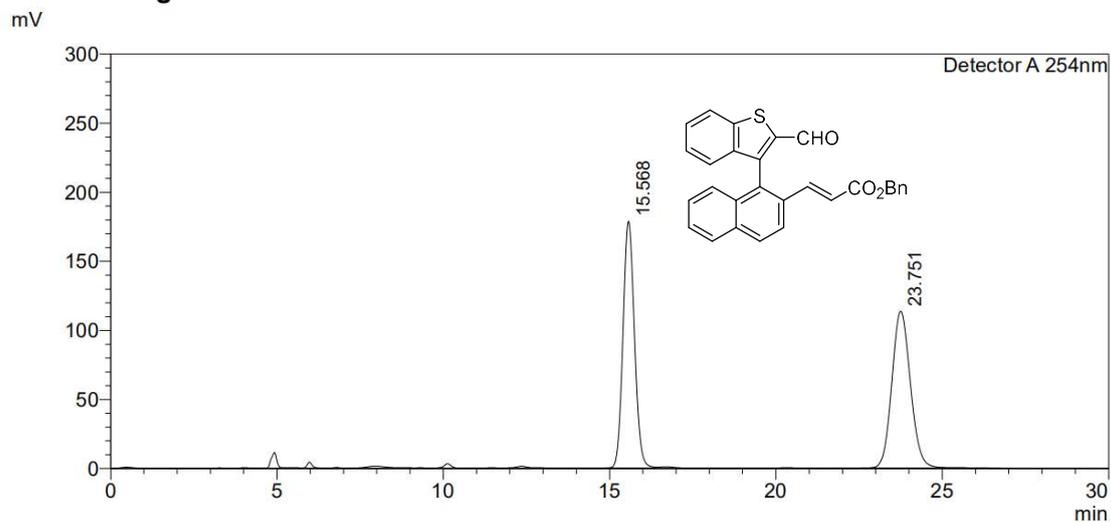
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.596	1133709	99125	18.545		M	
2	12.260	4979543	327995	81.455		M	
Total		6113252	427119				

Figure S53 HPLC Data of 1a in Table 6, entry 5

5f: IC, Hex/*i*PrOH = 80:20, rate = 1.0 mL/min, 254 nm

Racemate

<Chromatogram>

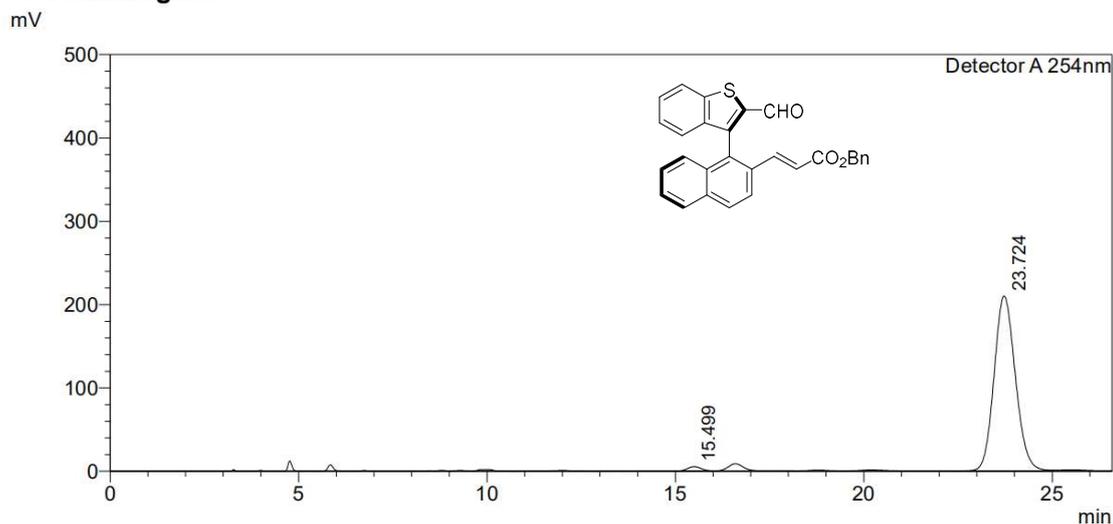


<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.568	4303608	178586	49.274			
2	23.751	4430352	113503	50.726			
Total		8733960	292088				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.499	131702	5243	1.571			
2	23.724	8251520	209713	98.429			
Total		8383222	214956				

Figure S54 HPLC Data of 5f

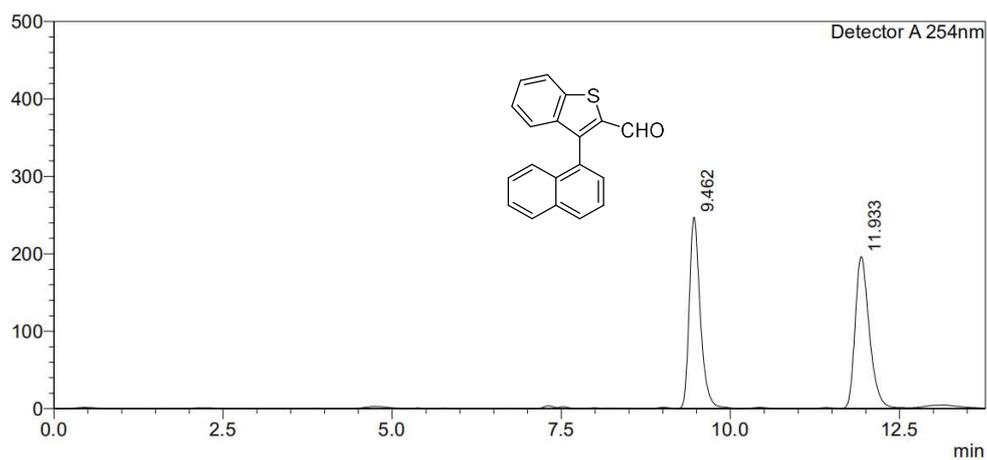
Table S7, entry 6

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

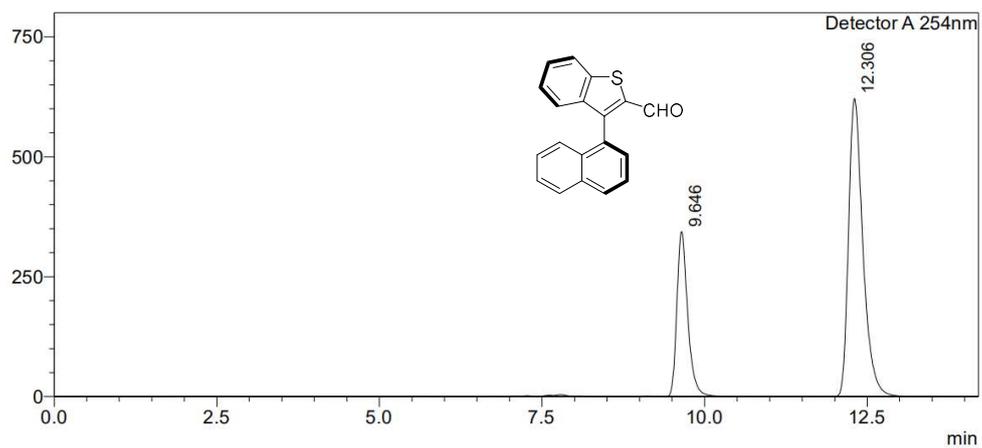
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

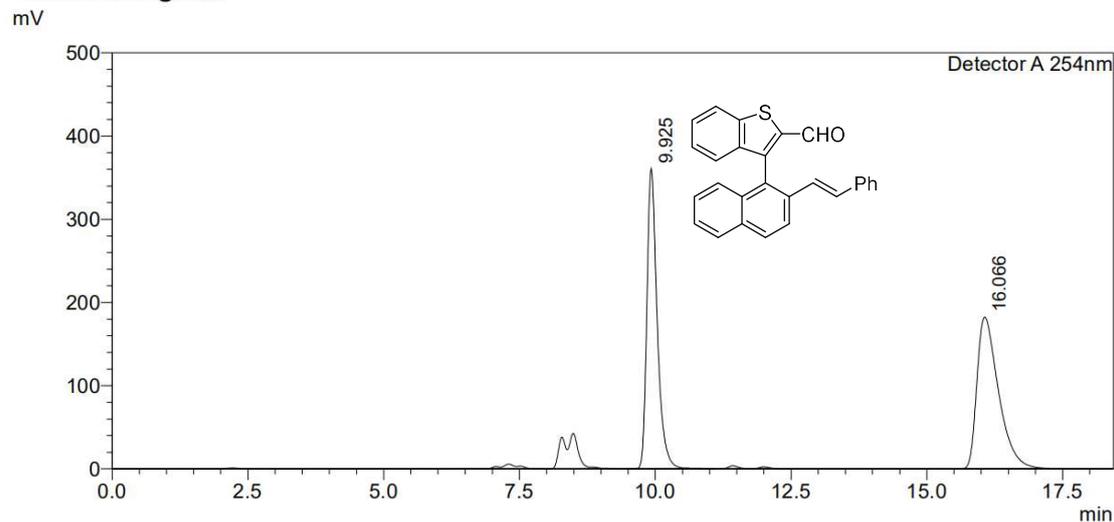
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.646	3990775	343198	29.442		M	
2	12.306	9563898	621471	70.558			
Total		13554673	964669				

Figure S55 HPLC Data of 1a in Table 6, entry 6

5g: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

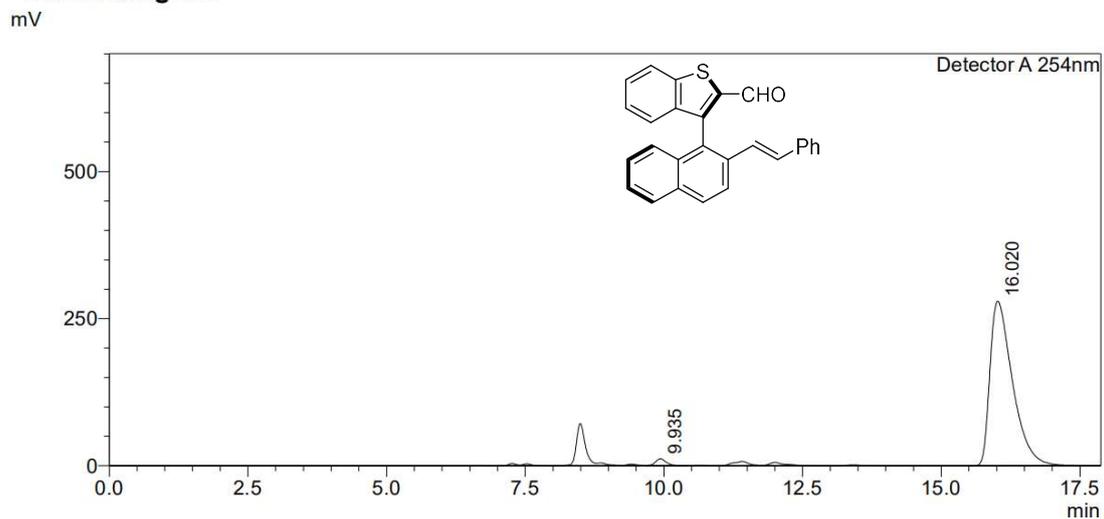


<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.925	4763167	360811	48.705		M	
2	16.066	5016435	182090	51.295		M	
Total		9779602	542901				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.935	169507	11967	2.095			
2	16.020	7922160	279478	97.905			
Total		8091667	291445				

Figure S56 HPLC Data of 5g

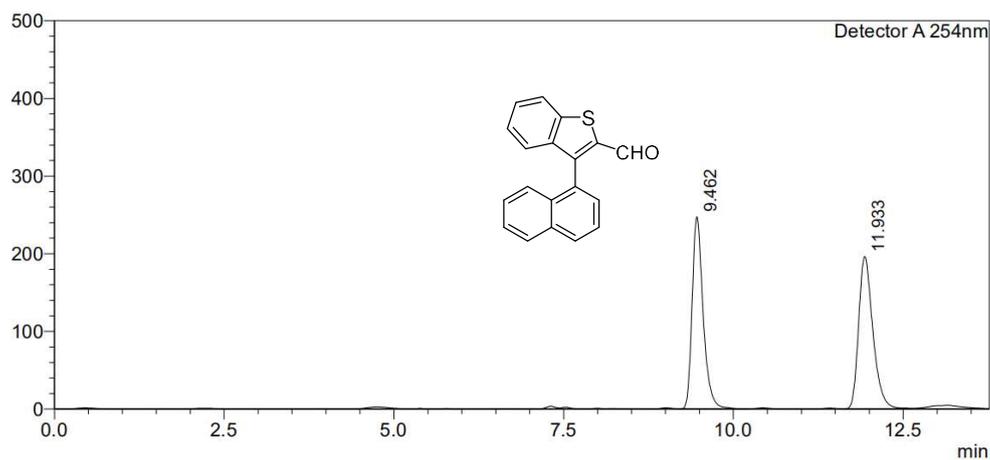
Table S7, entry 7

1a: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

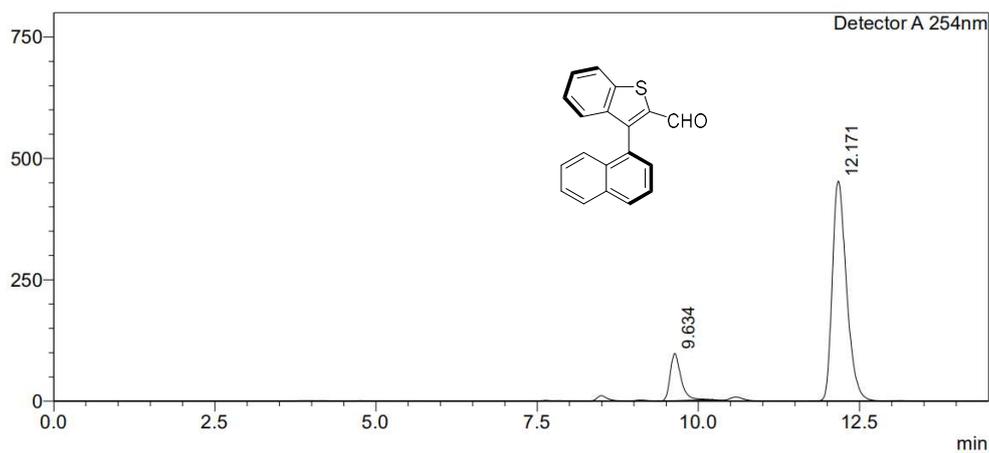
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.462	2811476	247628	49.483			
2	11.933	2870258	196246	50.517			
Total		5681734	443874				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

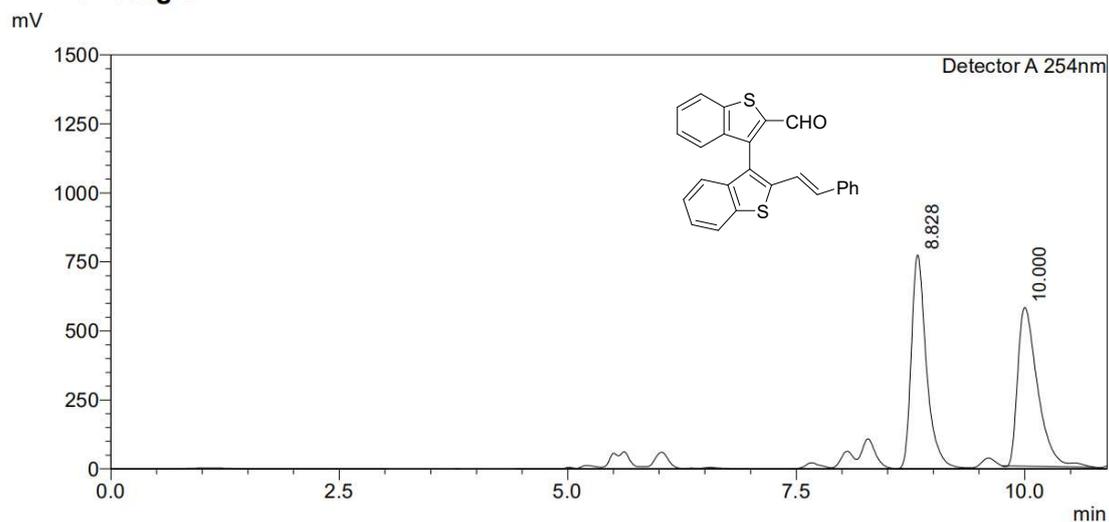
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.634	1135208	97356	14.049		M	
2	12.171	6944899	452563	85.951		M	
Total		8080107	549918				

Figure S57 HPLC Data of 1a in Table 6, entry 7

5h: IB, Hex/*i*PrOH = 90:10, rate =0.8 mL/min, 254 nm

Racemate

<Chromatogram>

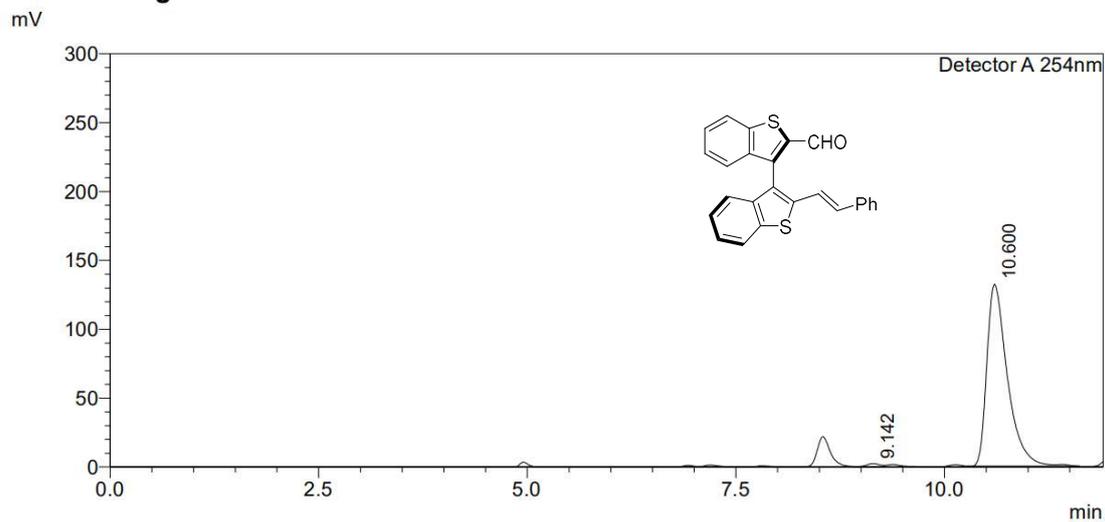


<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.828	8899171	773805	50.406			
2	10.000	8755648	575480	49.594		M	
Total		17654820	1349285				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.142	28612	2456	1.243			
2	10.600	2272662	131965	98.757		M	
Total		2301273	134421				

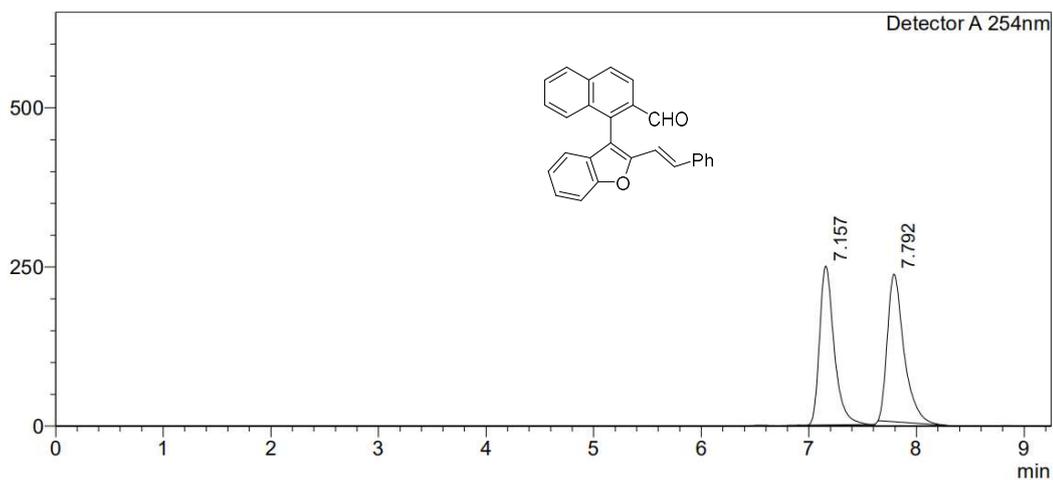
Figure S58 HPLC Data of 5h

5i: IB, Hex/*i*PrOH = 90:10, rate = 0.8 mL/min, 254 nm

Racemate

<Chromatogram>

mV



<Peak Table>

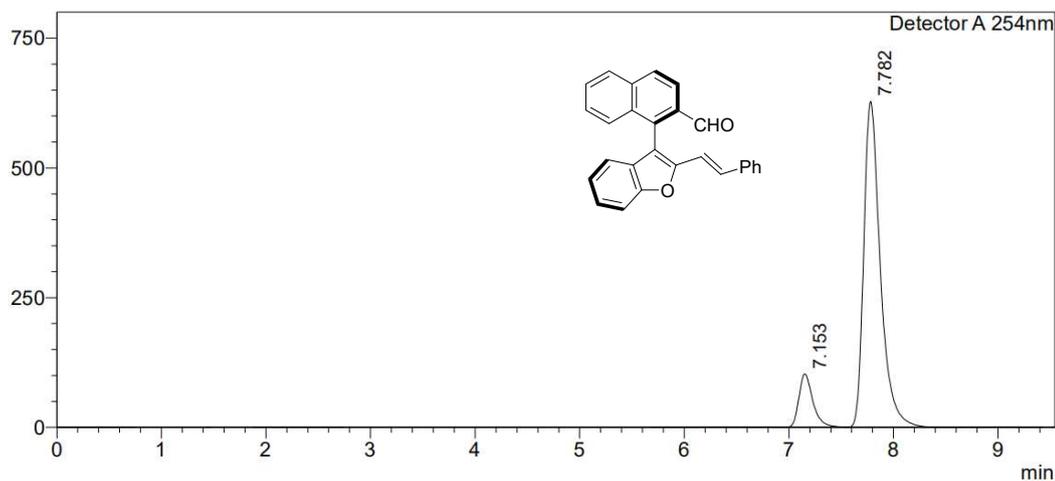
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.157	2344520	249694	48.381		M	
2	7.792	2501411	231970	51.619		M	
Total		4845930	481664				

Chiral

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

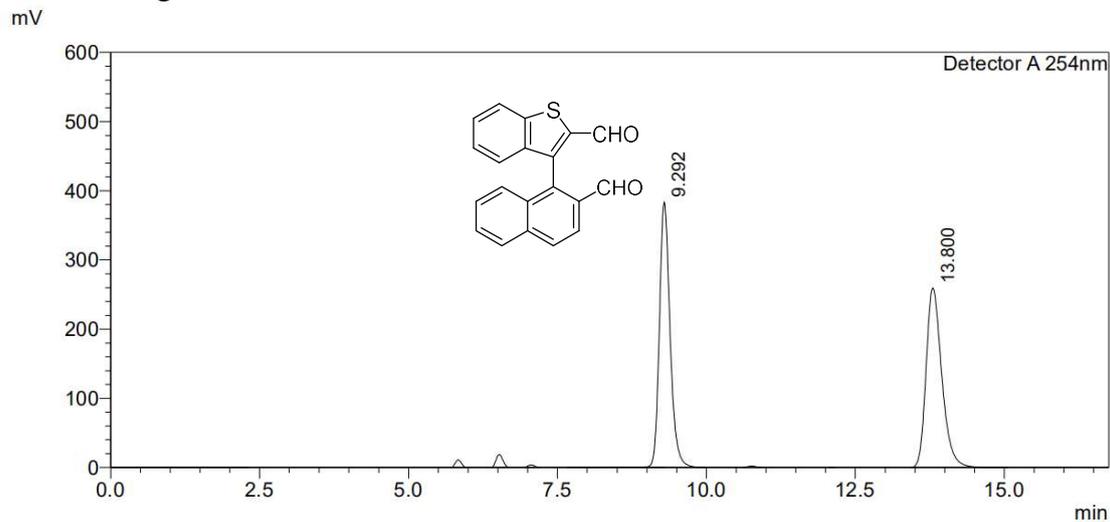
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.153	988027	103772	12.361			
2	7.782	7005182	629019	87.639		V	
Total		7993209	732791				

Figure S59 HPLC Data of 5i

6a: IA, Hex/*i*PrOH = 85:15, rate = 1.0 mL/min, 254 nm

Racemate

<Chromatogram>

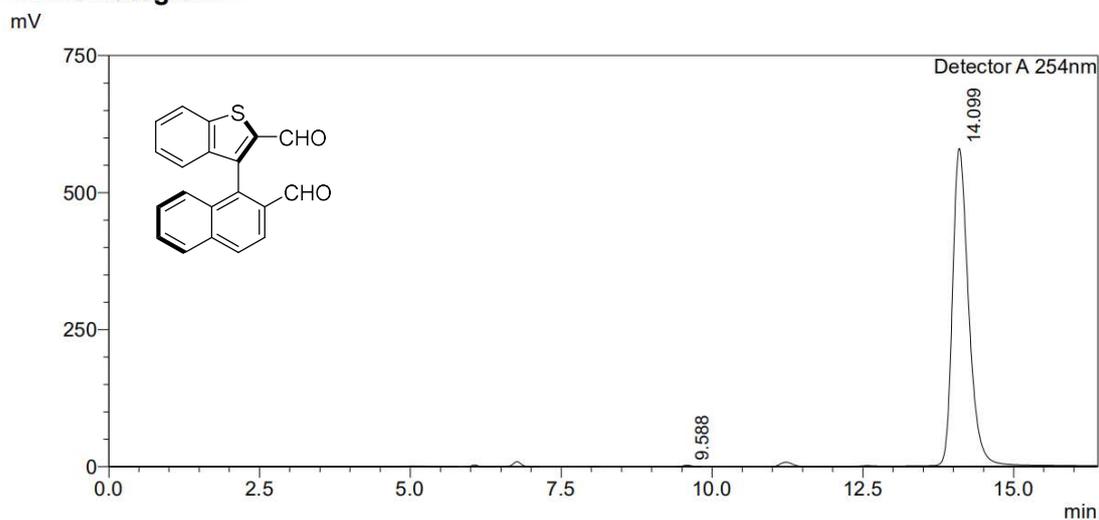


<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.292	4771677	383015	49.937		M	
2	13.800	4783755	260096	50.063		M	
Total		9555432	643111				

Chiral

<Chromatogram>



<Peak Table>

Detector A 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.588	47811	3716	0.439			
2	14.099	10854640	579484	99.561			
Total		10902451	583200				

Figure S60 HPLC Data of 6a