Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

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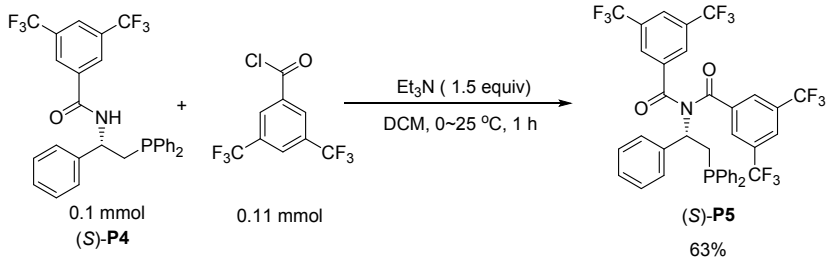
1. General Information

Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere; materials obtained from commercial suppliers were used directly without further purification. The $[\alpha]_D$ was recorded using PolAAr 3005 High Accuracy Polarimeter. ^{1}H NMR spectra, ^{13}C NMR spectra, ^{31}P NMR spectra and ^{19}F NMR spectra were recorded on a Bruker 400 (or 500) MHz spectrometer in chloroform-d_3. Chemical shifts (in ppm) were referenced to tetramethylsilane ($\delta = 0$ ppm) in $CDCl_3$ as an internal standard. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ ($\delta = 77.00$ ppm). The data is being reported as (s = singlet, d = doublet, dd = doublet of doublet, t = triplet, m = multiplet or unresolved, br = broad signal, coupling constant(s) in Hz, integration). Noteworthy, splitting signals between ^{13}C nucleus and ^{13}P nucleus in some chiral phosphine catalysts were difficult to distinguish and these ^{13}C NMR signals were reported as singlet entirely.

Trichloromethane ($CHCl_3$), dichloromethane, dichloroethane and ethyl acetate were freshly distilled from CaH_2; tetrahydrofuran (THF), toluene and ether were dried with sodium benzophenone and distilled before use.

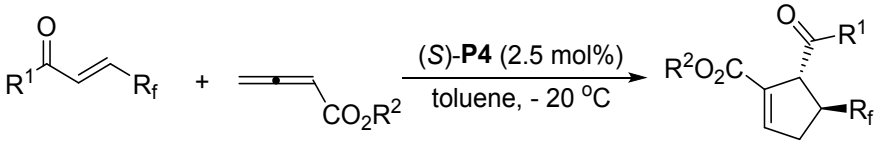
Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed on silica gel 60 (particle size 200-400 mesh ASTM, purchased from Yantai, China) and eluted with petroleum ether/ethyl acetate.

2. Synthetic Procedure and Data for Chiral Phosphine Catalyst (*S*)-P5



Under the protection of Ar, 3,5-Bis(trifluoromethyl)benzoyl chloride (1.1 eq.) was added slowly to the solution of (*S*)-**P4**[1] and Et_3N in DCM (1 mL) at 0 °C. This reaction mixture was then stirred at 25 °C for another 1 h, after completion of the reaction, the solvent was then removed in vacuo and the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired (*S*)-**P5**; white solid; $[\alpha]_D^{20}$ = – 211.2 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.61 (br, 6H), 7.56–7.54 (m, 2H), 7.47–7.43 (m, 2H), 7.39–7.21 (m, 11H), 5.94–5.88 (m, 1H), 3.70–3.62 (m, 1H), 2.95–2.89 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.56, 139.33, 139.07 (d, *J* = 7.50 Hz), 137.46 (d, *J* = 9.10 Hz), 136.14 (d, *J* = 11.80 Hz), 133.50 (d, *J* = 20.00 Hz), 132.32 (q, *J* = 34.3 Hz), 132.23 (d, *J* = 17.90 Hz), 129.75, 128.94 (d, *J* = 7.40 Hz), 128.83, 128.79, 128.69, 128.66, 128.63, 128.41 (br), 127.97, 124.98 (br), 122.16 (q, *J* = 271.50 Hz), 59.56 (d, *J* = 18.00 Hz), 32.59 (d, *J* = 13.30 Hz); ^{31}P NMR (121.5 MHz, $CDCl_3$) δ = -23.06 ppm; HRMS (ESI) m/z calcd. for $C_{38}H_{24}F_{12}NNaO_3P$ $[M+Na]^+$ = 824.1194, found = 824.1194.

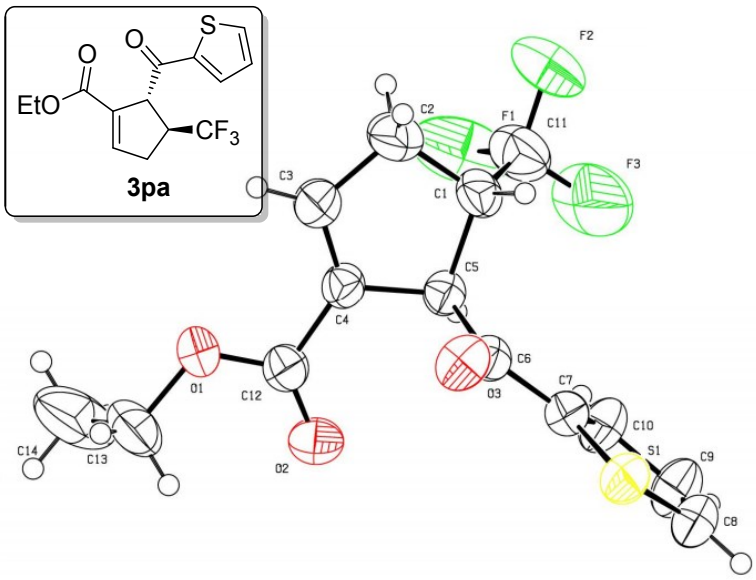
3. Typical Procedure for the Enantioselective [3+2] Cycloadditions of Allenes with β–Perfluoroalkyl α,β–Enones



1 **2** **3**

Under Ar, a stirred solution of **1**[2] (0.2 mmol) and (*S*)-**P4** (0.005 mmol) in toluene (2 mL) was cooled to -20 °C. Subsequently, **2** (0.24 mmol) was added in one portion. The reaction mixture was stirred for another 0.5 h, the solvents were removed in vacuo and the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired cycloaddition product **3**.

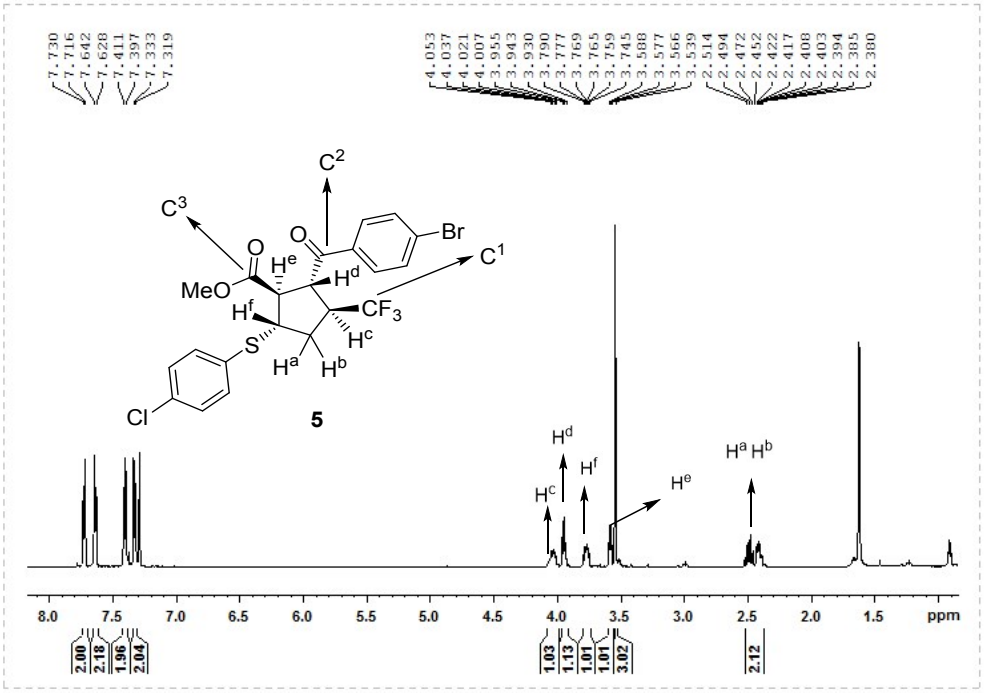
4. X-ray Crystal Structure for 3pa



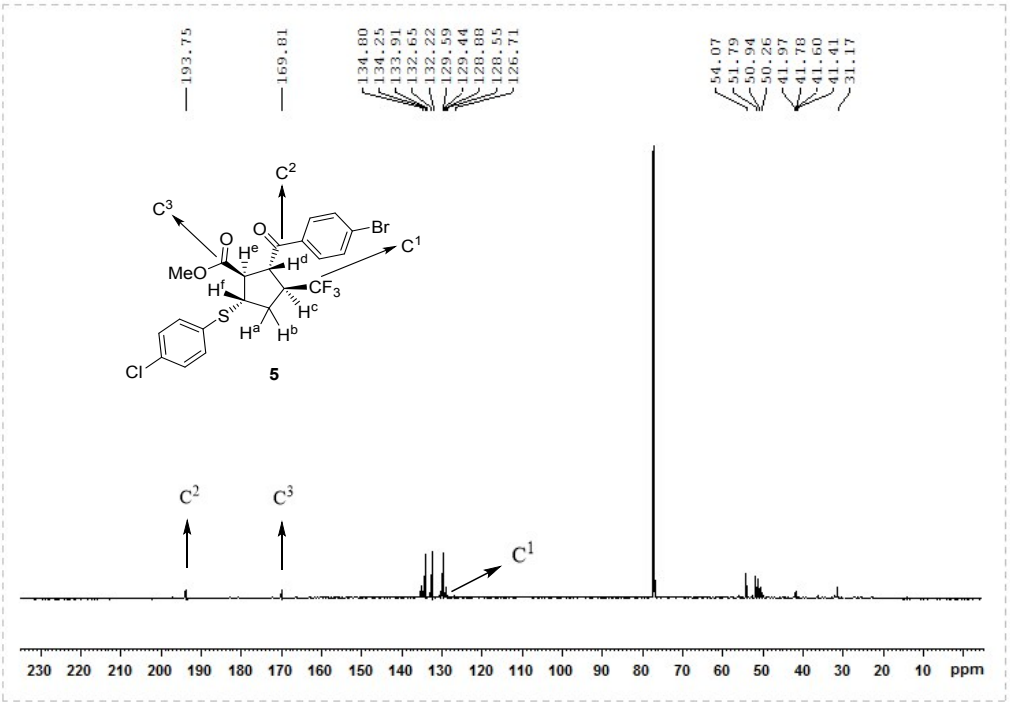
5. The assignment of the stereochemistry of the compound 5

In order to confirm the structure of the compound 5, 2D NMR (HSQC, HMBC and NOE) analysis was carried out. According to these NMR spectra, the the stereochemistry of the compound 5 was validate (NOE of H^d and H^e was not obsearved; NOE of H^e and H^f was not obsearved).

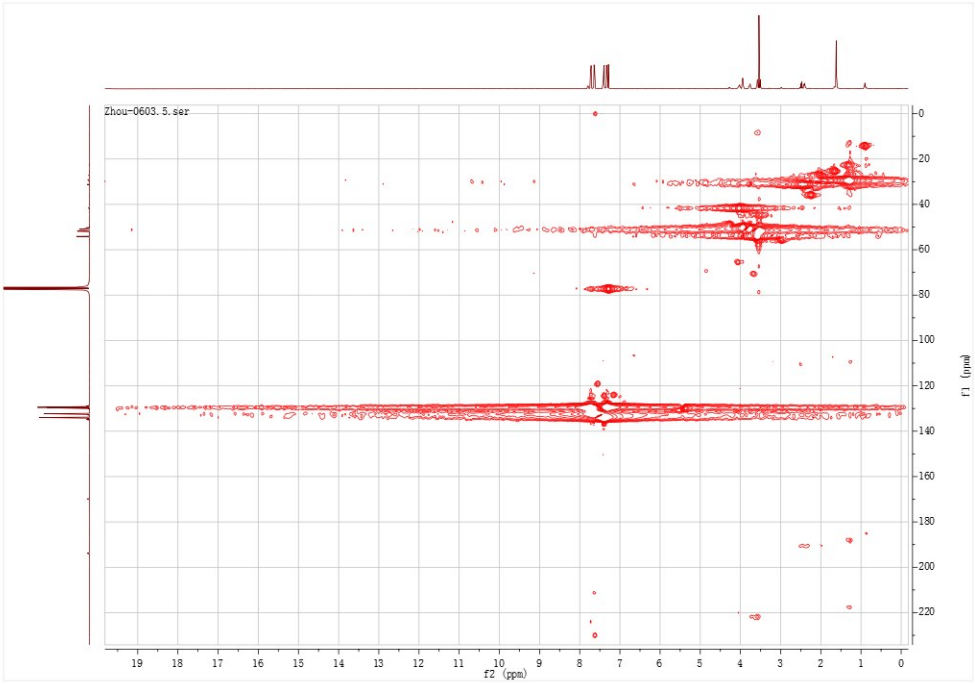
(a) ^{1}H NMR spectra for 5



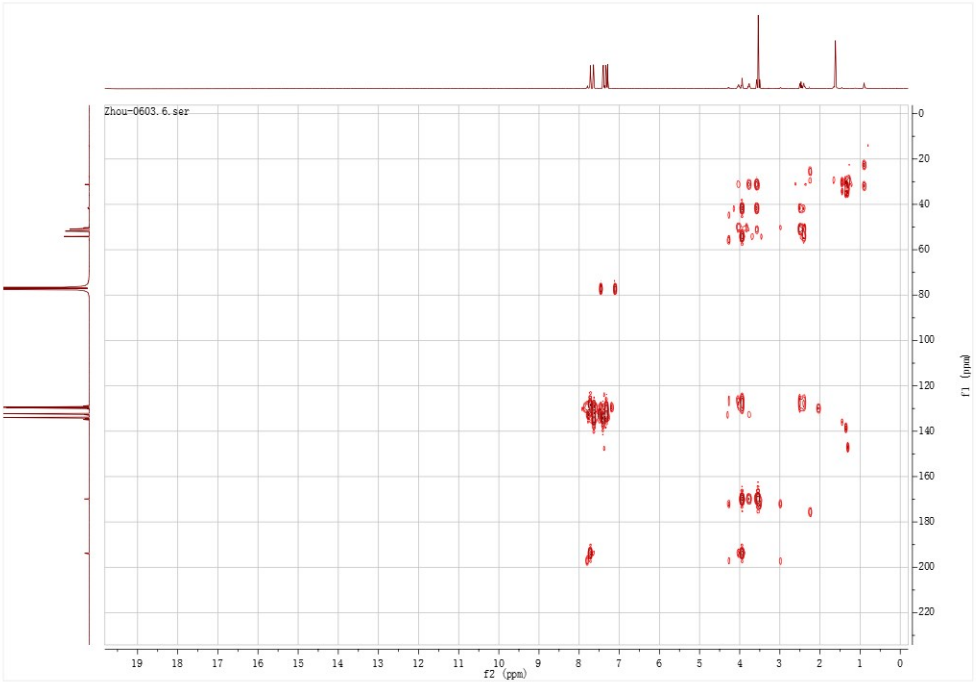
(b) ^{13}C NMR spectra for 5



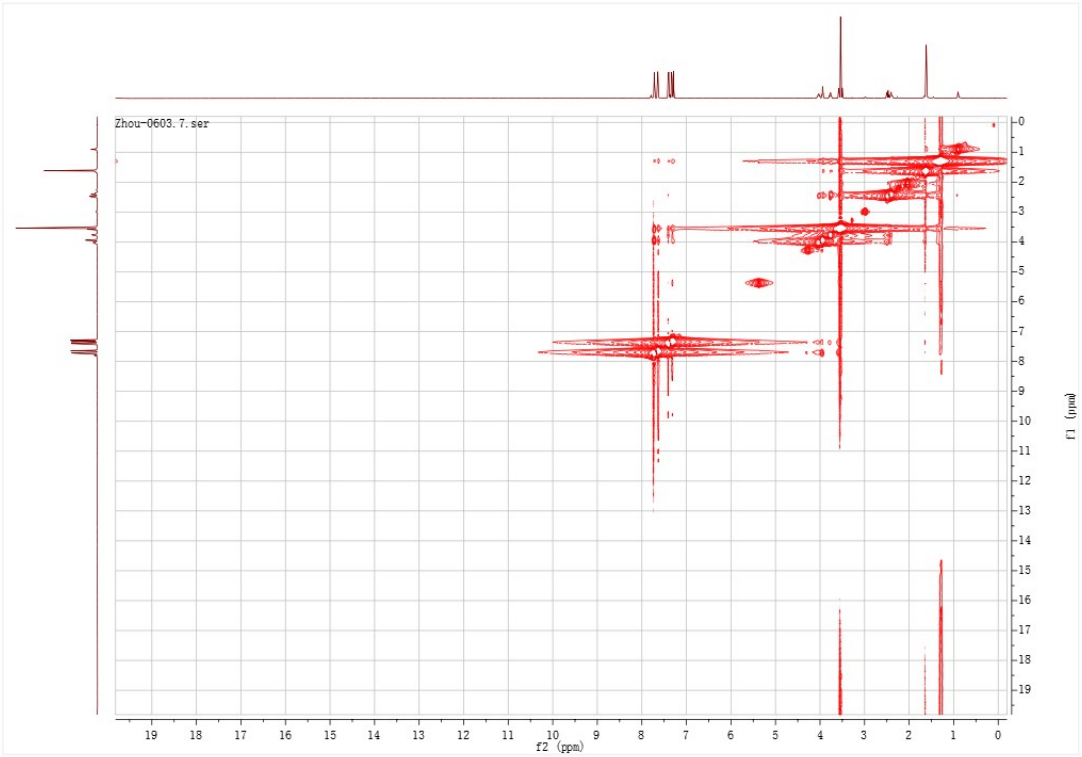
(c) HSQC spectra for 5



(d) HMBC spectra for 5

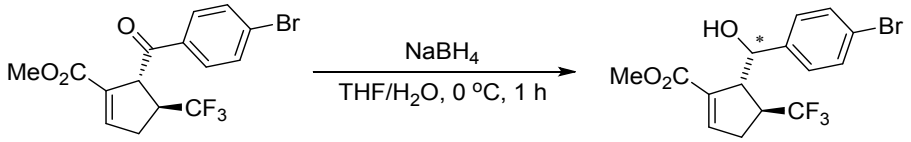


(e) NOE spectra for 5



6. Experimental Procedure for the Transformations of 3gb

Experimental Procedure for the Selective Reduction of 3gb

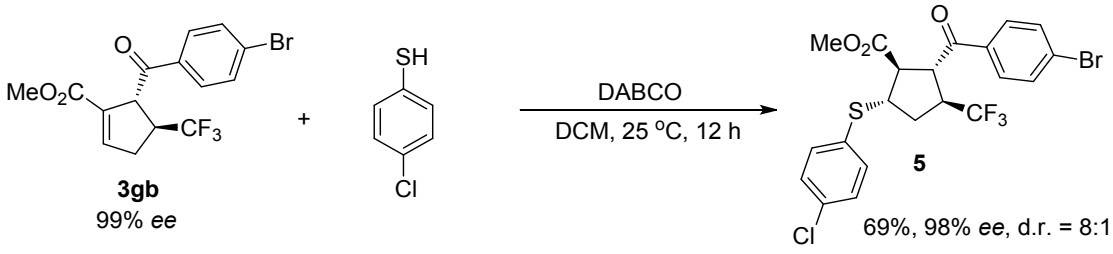


3gb
99% *ee*

4
96%, 99% *ee*, d.r. = 4:1

Under Ar, a stirred solution of **3gb** (0.2 mmol) in THF/H_2O (3/1, 2 mL) was added $NaBH_4$ (0.1 mmol). The mixture was stirred at 0 °C for 1 h. After completion of the reaction, 20 mL EtOAc was added. The organic layers was separated and dried over $MgSO_4$ and the solvents were removed in vacuo. Finally, the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired **4** in 96% yield without loss of enantiopurity (d.r. = 4:1).

Experimental Procedure for the Michael Addition of 3gb with 4-Chlorothiophenol

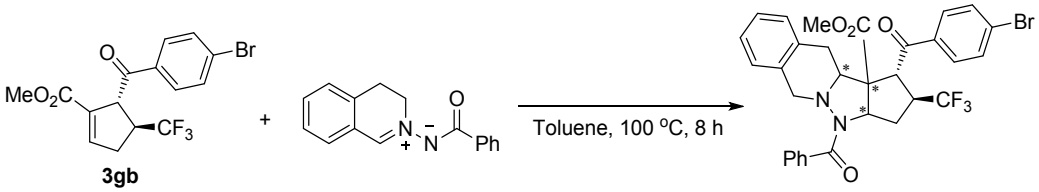


3gb
99% *ee*

5
69%, 98% *ee*, d.r. = 8:1

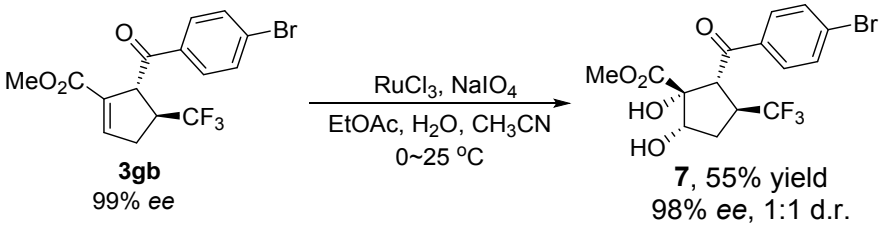
A stirred solution of **3gb** (0.2 mmol) in DCM (2 mL) was added 4-chlorothiophenol (0.40 mmol), DABCO (0.20 mmol). The mixture was stirred at r.t. for 12 h. After completion of the reaction, the reaction mixture was directly applied to a silica gel chromatography column to afford the desired **5** in 69% yield without loss of enantiopurity (d.r. = 8:1).

Experimental Procedure for the Dipolar Cycloaddition of 3gb



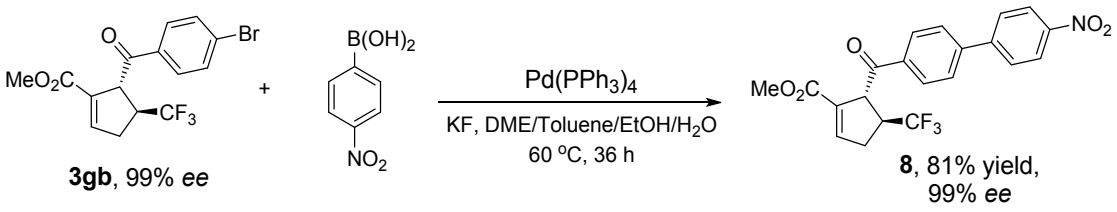
A stirred solution of **3gb** (0.2 mmol) in toluene (2 mL) was added benzoyl(3,4-dihydroisoquinolin-2-ium-2-yl)amide (0.4 mmol). The mixture was stirred at 100 °C for 8 h. After completion of the reaction, the reaction mixture was directly applied to a silica gel chromatography column to afford the desired **6** in 72% yield without loss of enantiopurity (d.r. > 20:1).

Experimental Procedure for the Dihydroxylation of 3gb



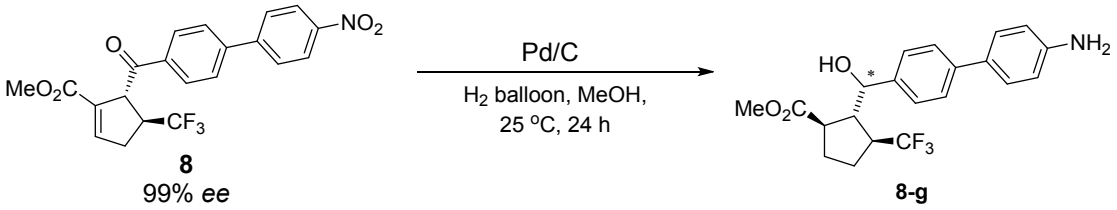
$NaIO_4$ (73 mg, 0.34 mmol, 1.3 equiv) and water (0.21 mL) were added to a 25 mL flask. After the $NaIO_4$ had dissolved, the solution was cooled to 0 °C, and H_2SO_4 (6 drops of a 2 N solution) and then $RuCl_3•_3H_2O$ (5 mg) were added. The solution was stirred for 5 min, and then EtOAc (0.37 mL) was added. The solution was stirred for an additional 5 min, and then CH_3CN (0.73 mL) was added. The solution was stirred for 5 more minutes, and then a solution of the substrate (0.26 mmol) in EtOAc (0.55 mL) was added in one portion. The solution was stirred for 6 min at 0 °C, and then it was transferred into a solution of 10% $NaHCO_3$ (1.8 mL) and saturated Na_2SO_3 (4.3 mL). The solution was stirred for 30 min, and then it was extracted with EtOAc (3x25mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated. The crude diol was chromatographed on silica gel (EtOAc/petroleum ether) to afford the desired **7** in 55% yield without loss of enantiopurity (d.r. = 1:1).

Experimental Procedure for the Suzuki Coulpling Reaction of 3gb with 4-Nitrophenylboronic Acid



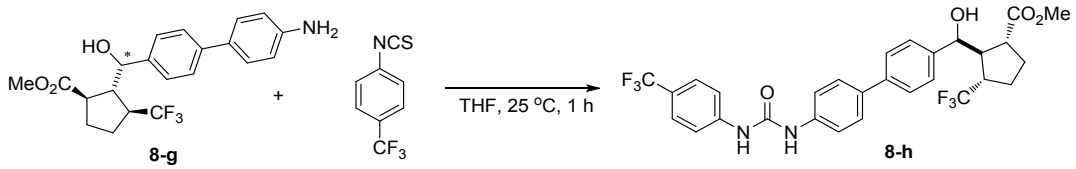
Under Ar, a stirred solution of **3gb** (2.0 mmol) in dimethoxyethane/Toluene/ EtOH/H_2O (10/1/6/3, 15 mL) was added KF (4.0 mmol), $Pd(PPh_3)_4$ (0.1 mmol). The mixture was stirred at 60 °C for 36 h. After completion of the reaction, 50 mL EtOAc was added. The organic layers was separated and dried over $MgSO_4$ and the solvents were removed in vacuo. Finally, the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired **8** in 81% yield without loss of enantiopurity.

Experimental Procedure for the Reduction of 8



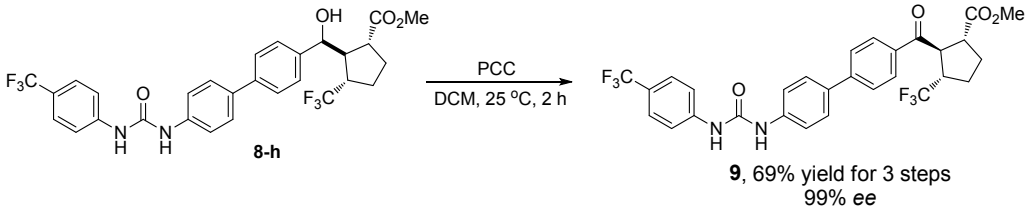
To a solution of **8** (1 mmol) in MeOH (4 mL) was added 10% Pd/C (150 mg) at room temperature. The reaction flask was evacuated twice under reduced pressure, and a H_2 balloon was placed on the top. After stirring at room temperature for 24 h, the mixture was filtered through celite and the filtrate was concentrated and the residue was directly used in the next step.

Experimental Procedure for the Reaction of 8-g with 4-(Trifluoro-methyl) phenyl isocyanate



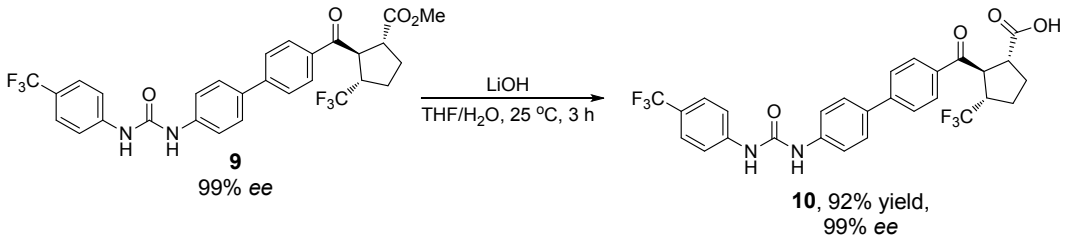
To a solution of **8-g** in THF (5 mL) was added 4-(trifluoromethyl)phenyl isocyanate (1.1 mmol). The mixture was stirred at room temperature for 1 h. After completion of the reaction, the reaction mixture was concentrated and the residue was directly used in the next step.

Experimental Procedure for the PCC Oxidation of 8-h



To a solution of **8-h** in DCM (5 mL) was added PCC (2.0 mmol) and silica gel (500 mg) at room temperature. After stirring at room temperature for 2 h, the mixture was filtered through celite and the filtrate was concentrated, the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired **9** in 69% yield (three steps)without loss of enantiopurity (d.r. > 20:1).

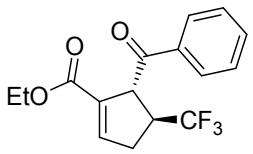
Experimental Procedure for the Hydrolysis of 9



A stirred solution of **9** (0.2 mmol) in THF/H_2O (4/1, 5 mL) was added LiOH (1 mmol). After stirring at room temperature for 3 h, 4 N HCl was added dropwise untill the pH < 7. Subsequently, 50 mL EtOAc was added and the organic layers was separated. Then the organic layers was dried over $MgSO_4$ and the solvents were removed in vacuo. Finally, the residue was directly purified by silica gel chromatography using petroleum ether/EtOAc as the eluent to afford the desired **10** in 92% yield without loss of

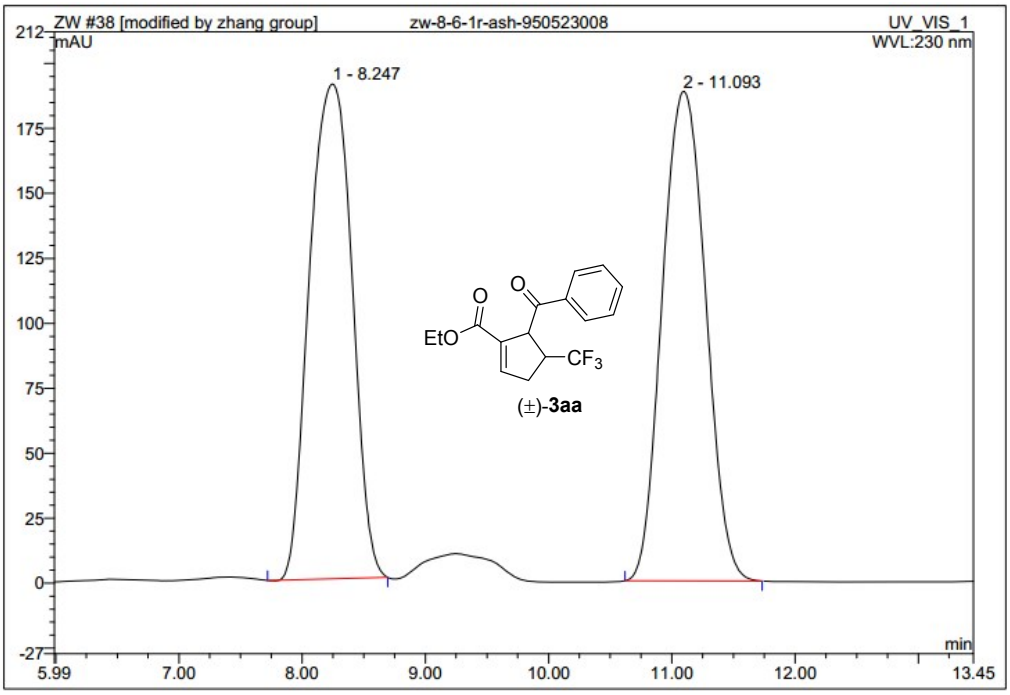
enantiopurity.

7. Data and HPLC Spectra for 3, 4, 5, 6, 7, 8, 9, 10

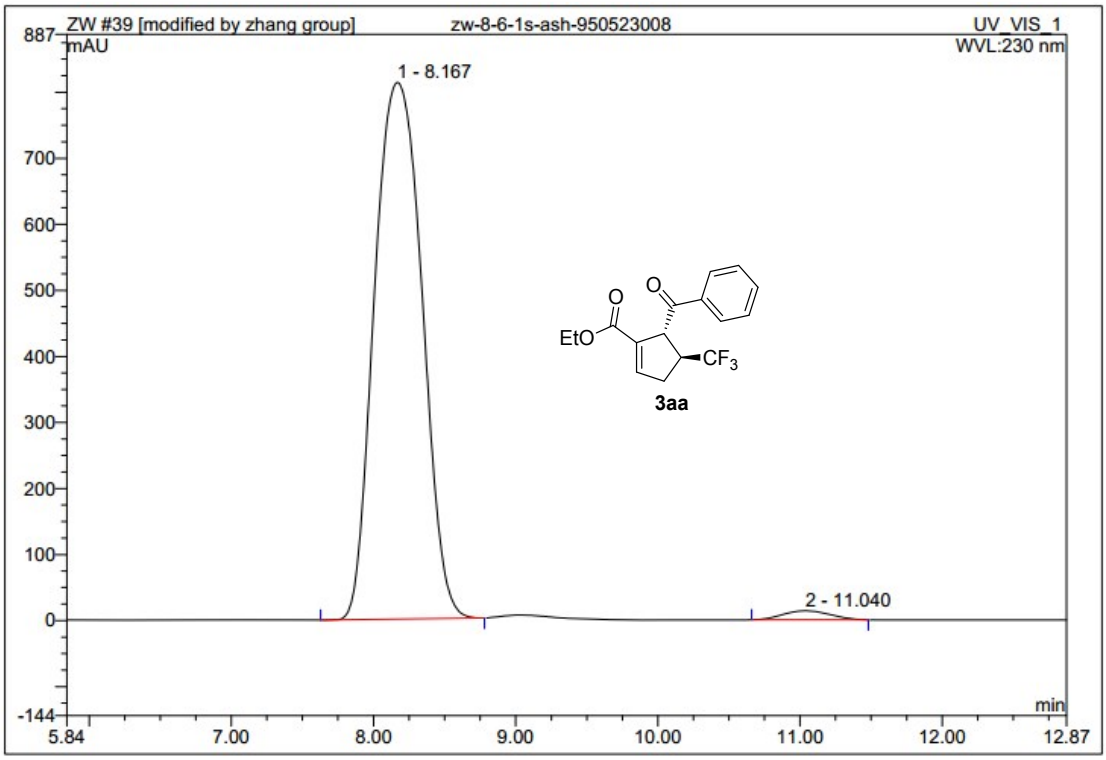


3aa

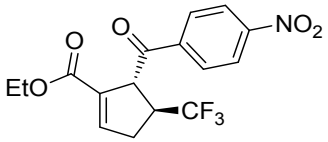
3aa; colorless oil; $[\alpha]_D^{20}$ = + 29.2 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 8.07–8.05 (m, 2H), 7.63–7.59 (m, 1H), 7.52–7.48 (m, 2H), 6.96–6.95 (m, 1H), 5.06–5.04 (m, 1H), 4.10–3.99 (m, 2H), 3.31–3.19 (m, 1H), 3.04–2.95 (m, 1H), 2.81–2.74 (m, 1H), 1.05 (t, *J* = 6.80 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.09, 163.03, 143.50, 136.35, 135.79, 133.58, 128.76, 128.64, 127.15 (q, *J* = 275.90 Hz), 60.78, 50.52 (d, *J* = 1.80 Hz), 46.74 (q, *J* = 28.00 Hz), 33.33 (d, *J* = 2.40 Hz), 13.74; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.64 ppm; Enantiomeric excess: 97%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 8.17 min, second peak: t_R = 11.04 min; HRMS (ESI) m/z calcd. for $C_{16}H_{15}F_3NaO_3$ $[M+Na]^+$ = 335.0865, found = 335.0868.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.25	n.a.	190.443	76.149	49.72	n.a.	BMB*
2	11.09	n.a.	188.418	76.993	50.28	n.a.	BMB*
Total:			378.861	153.142	100.00	0.000	

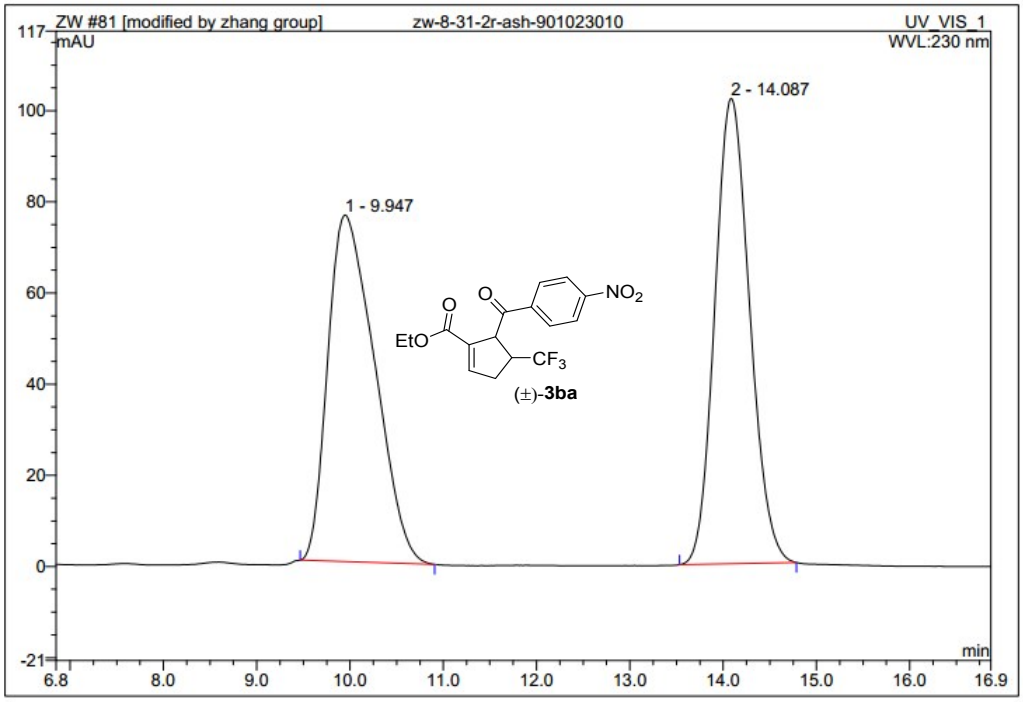


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.17	n.a.	812.538	315.340	98.37	n.a.	BMB*
2	11.04	n.a.	13.610	5.238	1.63	n.a.	BMB*
Total:			826.147	320.578	100.00	0.000	

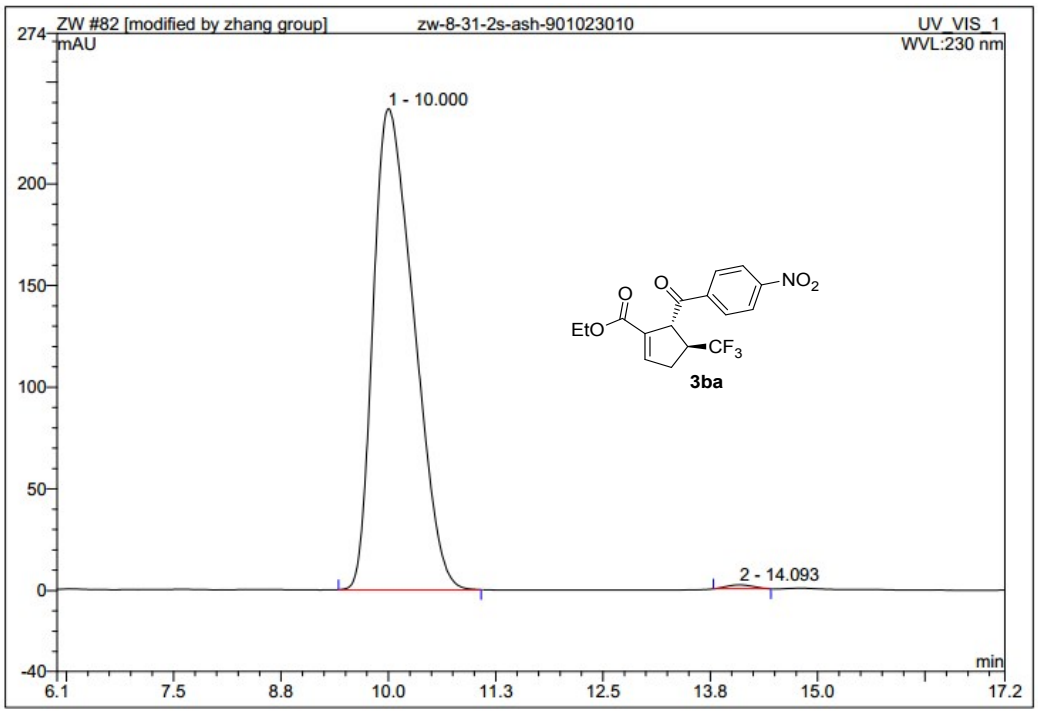


3ba

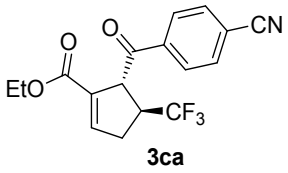
3ba; colorless oil; $[\alpha]_D^{20}$ = + 110.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.37–8.35 (m, 2H), 8.23–8.20 (m, 2H), 6.97 (d, *J* = 1.05 Hz, 1H), 5.04–5.02 (m, 1H), 4.13–4.04 (m, 2H), 3.38–3.28 (m, 1H), 3.08–3.01 (m, 1H), 2.86–2.80 (m, 1H), 1.12 (t, *J* = 7.50 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.27, 162.92, 150.47, 143.80, 141.15, 135.52, 129.67, 126.90 (q, *J* = 275.88 Hz), 123.83, 61.07, 50.07 (d, *J* = 1.50 Hz), 46.97 (q, *J* = 28.25 Hz), 33.33 (d, *J* = 2.25 Hz), 13.88; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.36 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 10.00 min, second peak: t_R = 14.09 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}F_3NNaO_5$ $[M+Na]^+$ = 380.0716, found = 380.0726.



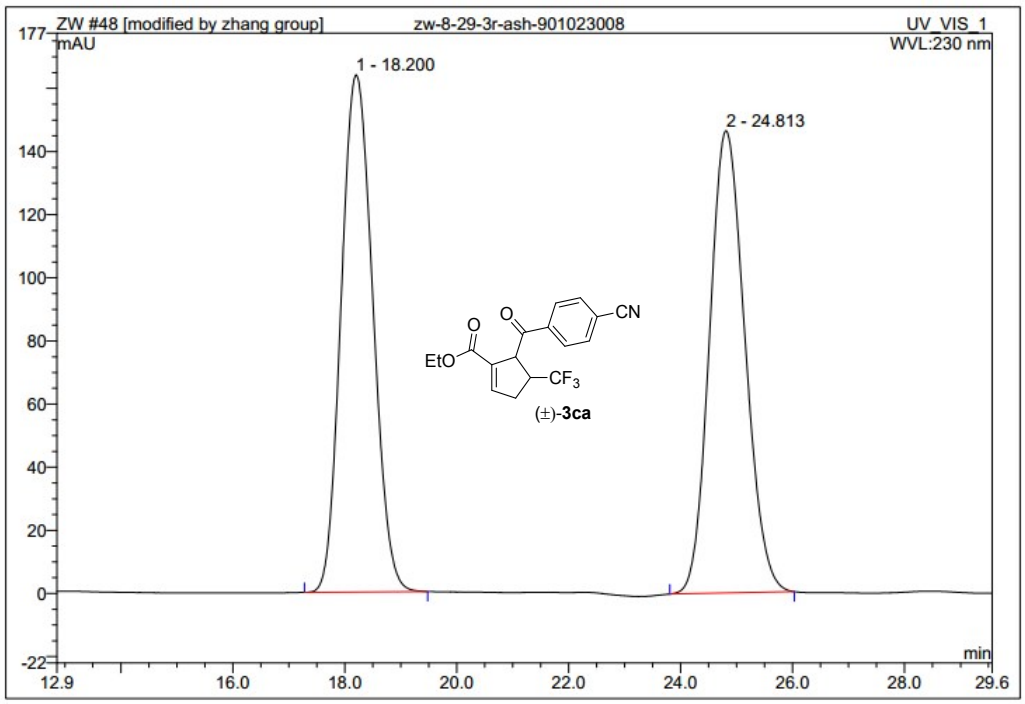
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.95	n.a.	76.054	44.100	49.97	n.a.	BMB*
2	14.09	n.a.	102.141	44.151	50.03	n.a.	BMB*
Total:			178.195	88.250	100.00	0.000	



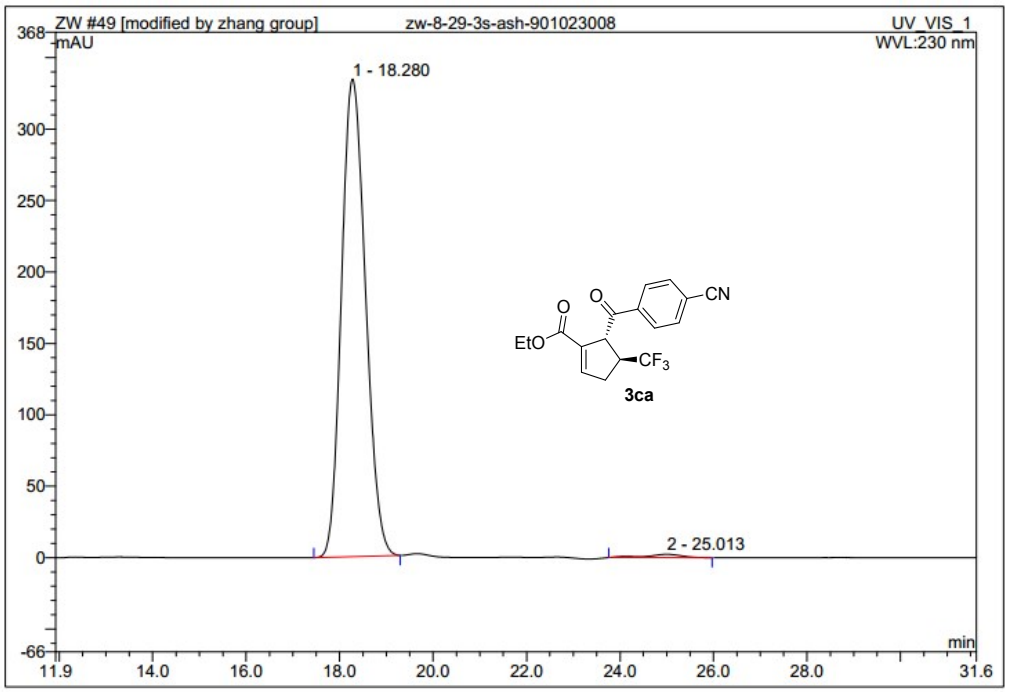
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.00	n.a.	236.591	129.437	99.47	n.a.	BMB*
2	14.09	n.a.	1.921	0.685	0.53	n.a.	BMB*
Total:			238.512	130.122	100.00	0.000	



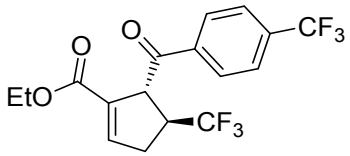
3ca; colorless oil; $[\alpha]_D^{20}$ = + 119.7 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.16–8.14 (m, 2H), 7.83–7.81 (m, 2H), 6.97 (d, *J* = 1.00 Hz, 1H), 5.01–4.99 (m, 1H), 4.12–4.03 (m, 2H), 3.35–3.25 (m, 1H), 3.06–3.00 (m, 1H), 2.84–2.79 (m, 1H), 1.11 (t, *J* = 7.50 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.36, 162.90, 143.76, 139.59, 135.51, 132.48, 129.03, 126.90 (q, *J* = 275.88 Hz), 117.80, 116.70, 61.02, 50.78, 46.91 (q, *J* = 28.25 Hz), 33.33, 13.84; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.39 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 18.28 min, second peak: t_R = 25.01 min; HRMS (ESI) m/z calcd. for $C_{17}H_{14}F_3NNaO_3$ $[M+Na]^+$ = 360.0818, found = 360.0821.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	18.20	n.a.	163.841	107.152	49.98	n.a.	BMB*
2	24.81	n.a.	146.444	107.246	50.02	n.a.	BMB*
Total:			310.285	214.398	100.00	0.000	

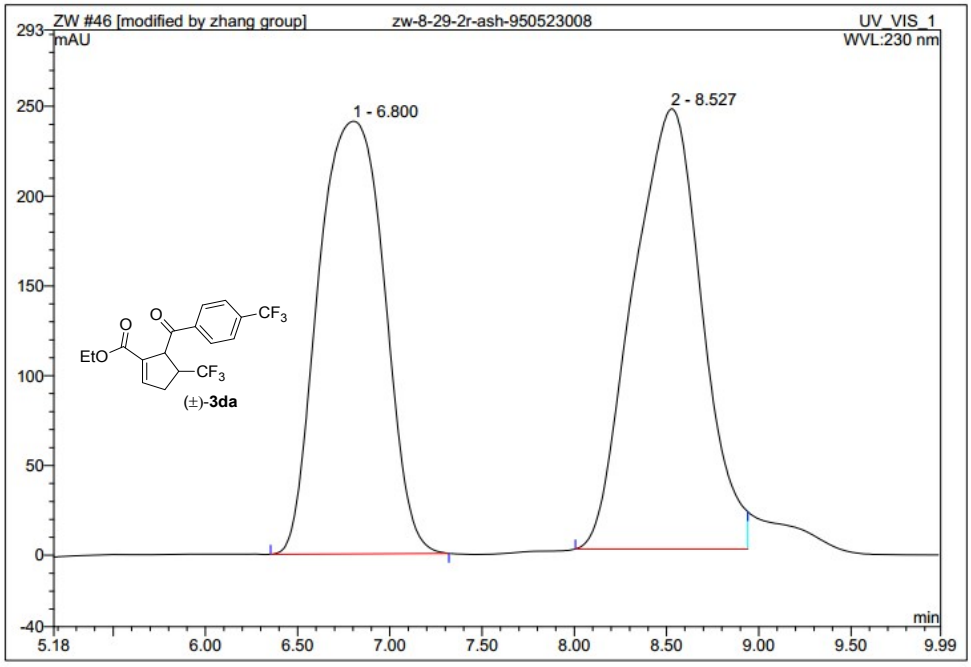


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	18.28	n.a.	334.312	198.783	98.97	n.a.	BMB*
2	25.01	n.a.	2.334	2.073	1.03	n.a.	BMB*
Total:			336.646	200.856	100.00	0.000	

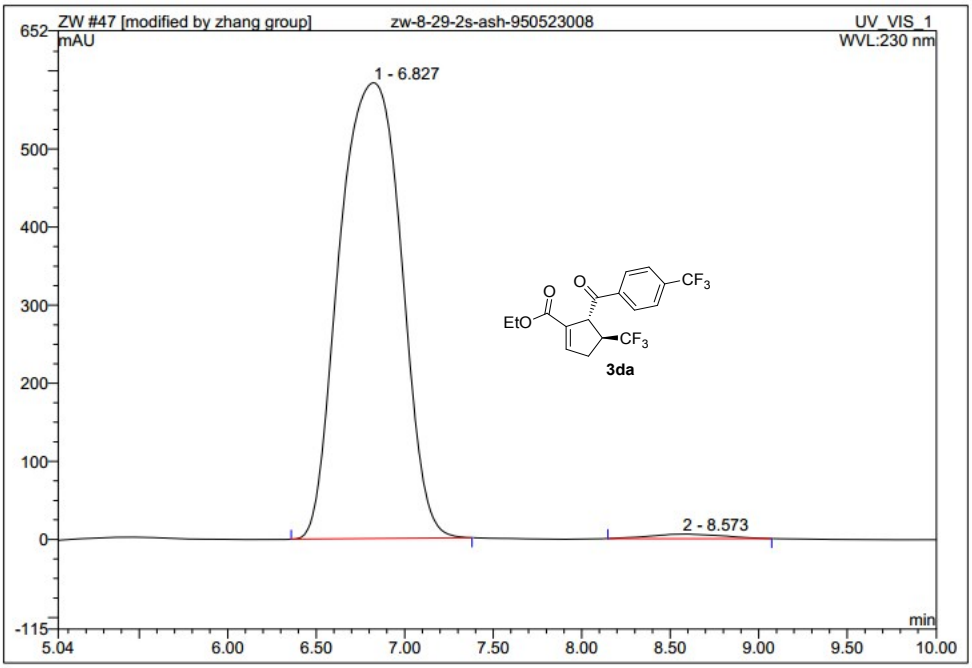


3da

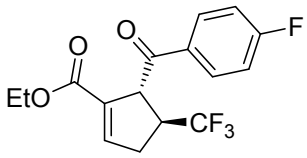
3da; colorless oil; $[\alpha]_D^{20}$ = + 165.9 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.18 (d, *J* = 8.00 Hz, 2H), 7.79 (d, *J* = 8.50 Hz, 2H), 6.98 (s, 1H), 5.04 (d, *J* = 3.50 Hz, 1H), 4.13–4.04 (m, 2H), 3.35–3.26 (m, 1H), 3.07–3.00 (m, 1H), 2.84–2.79 (m, 1H), 1.10 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.56, 162.97, 143.68, 139.25, 135.65, 134.74 (q, *J* = 32.50 Hz), 129.04, 127.01 (q, *J* = 275.88 Hz), 125.73 (q, *J* = 3.63 Hz), 123.53 (q, *J* = 271.00 Hz), 60.98, 50.83 (q, *J* = 1.63 Hz), 46.91 (q, *J* = 28.25 Hz), 33.34 (d, *J* = 2.50 Hz), 13.81; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -63.26, -71.52 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 6.83 min, second peak: t_R = 8.57 min; HRMS (ESI) m/z calcd. for $C_{17}H_{14}F_6NaO_3$ $[M+Na]^+$ = 403.0739, found = 403.0745.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.80	n.a.	240.974	100.008	48.43	n.a.	BMB*
2	8.53	n.a.	245.114	106.476	51.57	n.a.	BM *
Total:			486.088	206.484	100.00	0.000	

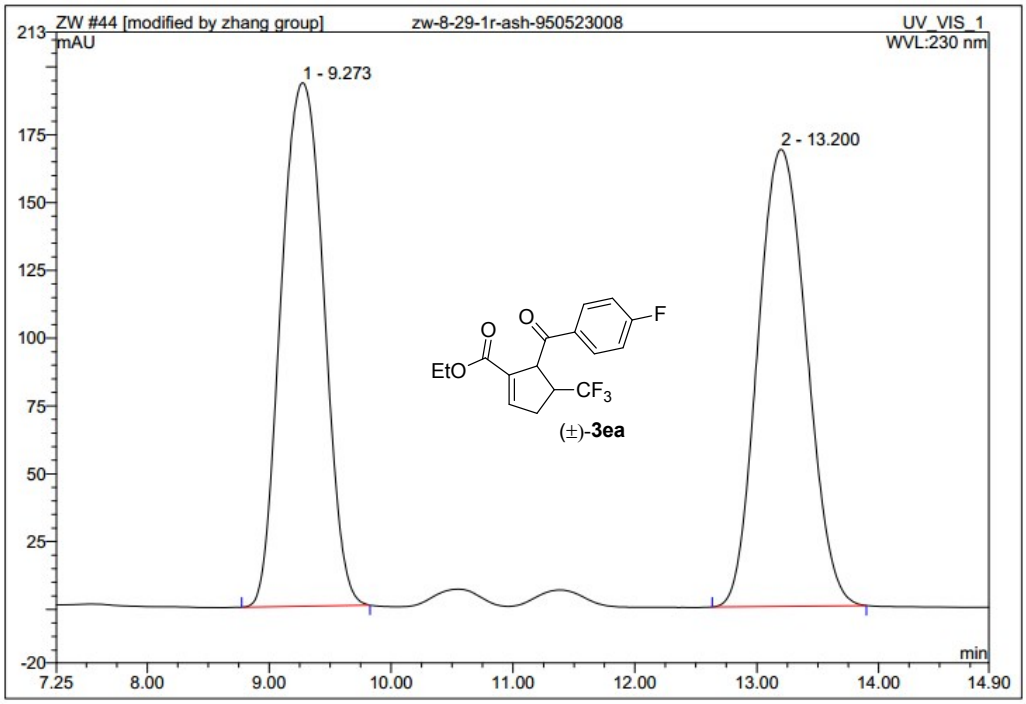


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.83	n.a.	583.750	237.463	98.85	n.a.	BMB*
2	8.57	n.a.	5.846	2.757	1.15	n.a.	BMB*
Total:			589.596	240.220	100.00	0.000	

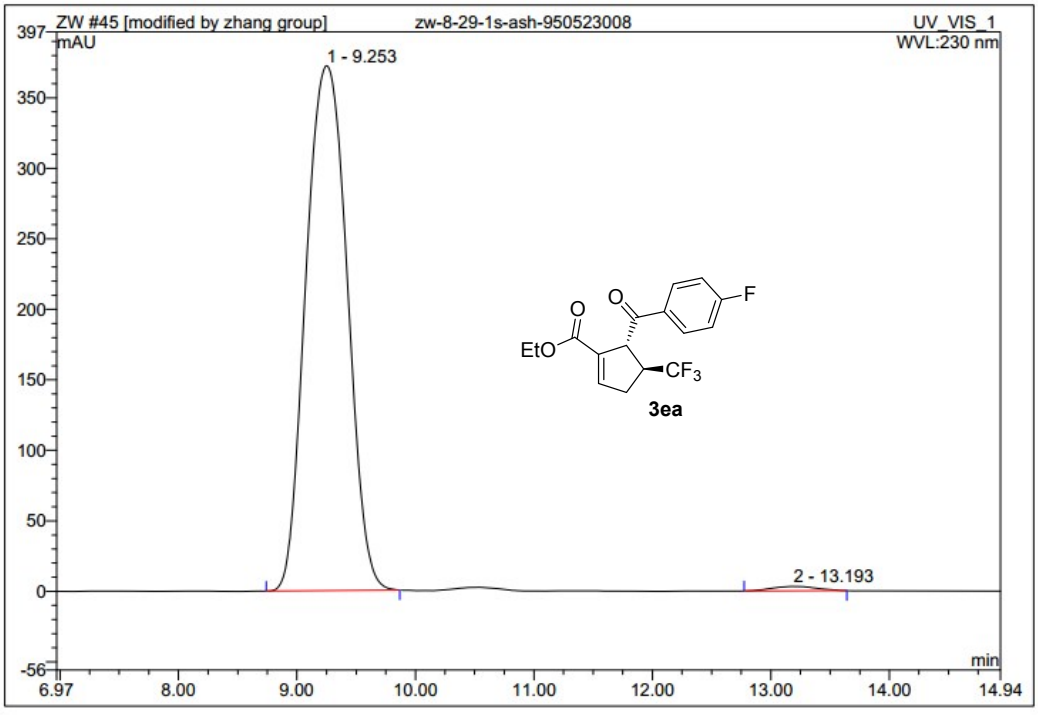


3ea

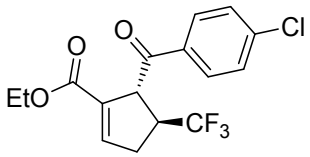
3ea; colorless oil; $[\alpha]_D^{20}$ = + 176.0 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 8.13–8.08 (m, 2H), 7.21–7.15 (m, 2H), 6.96–6.95 (m, 1H), 5.01–4.98 (m, 1H), 4.12–4.01 (m, 2H), 3.31–3.19 (m, 1H), 3.05–2.96 (m, 1H), 2.81–2.74 (m, 1H), 1.08 (t, *J* = 7.20 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.59, 166.10 (d, *J* = 254.20 Hz), 163.02, 143.57, 135.67, 132.81 (d, *J* = 2.90 Hz), 131.50 (d, *J* = 9.80 Hz), 127.10 (q, *J* = 276.00 Hz), 115.81 (d, *J* = 21.80 Hz), 60.84, 50.49 (d, *J* = 1.90 Hz), 46.77 (q, *J* = 28.20 Hz), 33.34 (d, *J* = 2.40 Hz), 13.81; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.62, -104.32 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.25 min, second peak: t_R = 13.19 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}F_4NaO_3$ $[M+Na]^+$ = 353.0771, found = 353.0775.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.27	n.a.	193.000	78.358	50.01	n.a.	BMB*
2	13.20	n.a.	168.538	78.321	49.99	n.a.	BMB*
Total:			361.538	156.679	100.00	0.000	

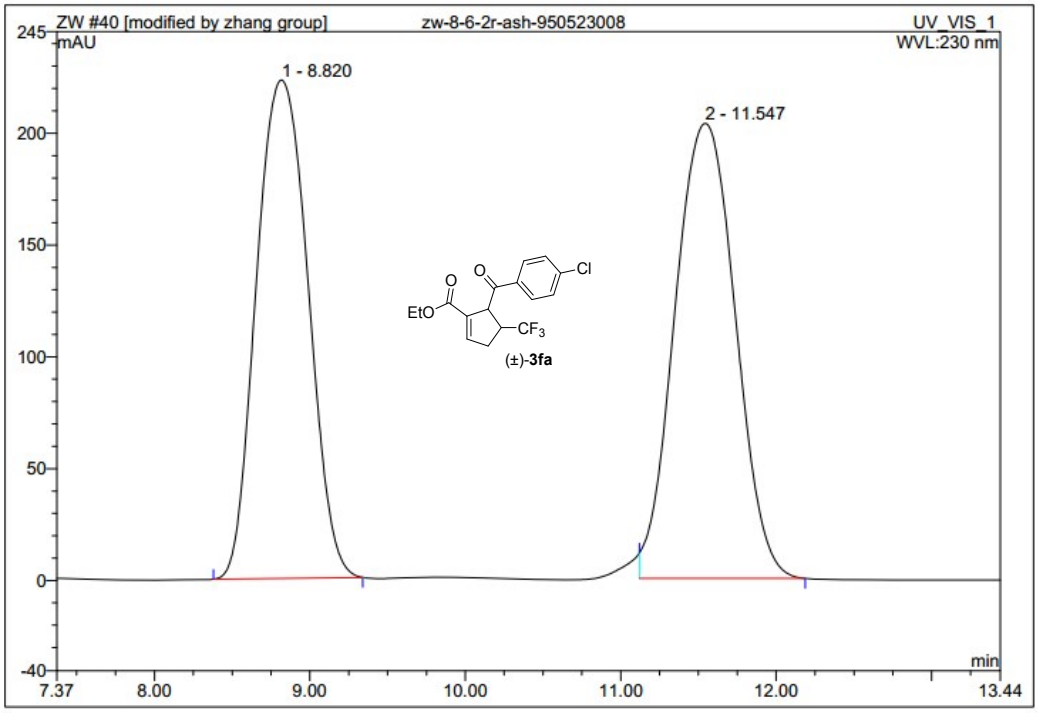


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.25	n.a.	372.201	150.532	99.10	n.a.	BMB*
2	13.19	n.a.	3.142	1.367	0.90	n.a.	BMB*
Total:			375.343	151.898	100.00	0.000	

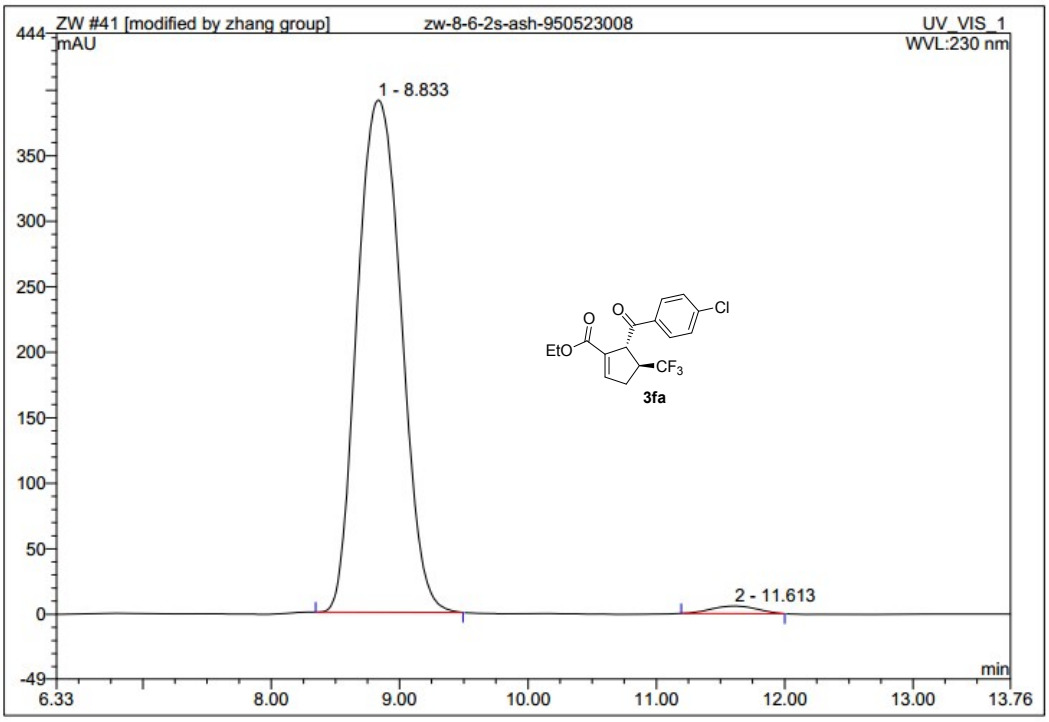


3fa

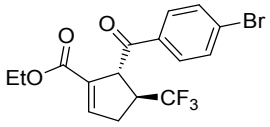
3fa; colorless oil; $[\alpha]_D^{20}$ = + 216.6 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.50 Hz, 2H), 7.49 (d, *J* = 8.50 Hz, 2H), 6.96 (s, 1H), 5.00–4.99 (m, 1H), 4.14–4.03 (m, 2H), 3.31–3.22 (m, 1H), 3.04–2.98 (m, 1H), 2.81–2.76 (m, 1H), 1.10 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.03, 162.96, 143.58, 140.16, 135.65, 134.74, 130.15, 128.98, 127.06 (q, *J* = 276.13 Hz), 60.86, 50.50 (d, *J* = 1.38 Hz), 46.79 (q, *J* = 28.00 Hz), 33.32 (q, *J* = 2.25 Hz), 13.82; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.57 ppm; Enantiomeric excess: 97%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 8.83 min, second peak: t_R = 11.61 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}ClF_3NaO_3$ $[M+Na]^+$ = 369.0476, found = 369.0482.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.82	n.a.	222.863	85.794	48.21	n.a.	BMB*
2	11.55	n.a.	203.562	92.148	51.79	n.a.	MB*
Total:			426.425	177.941	100.00	0.000	

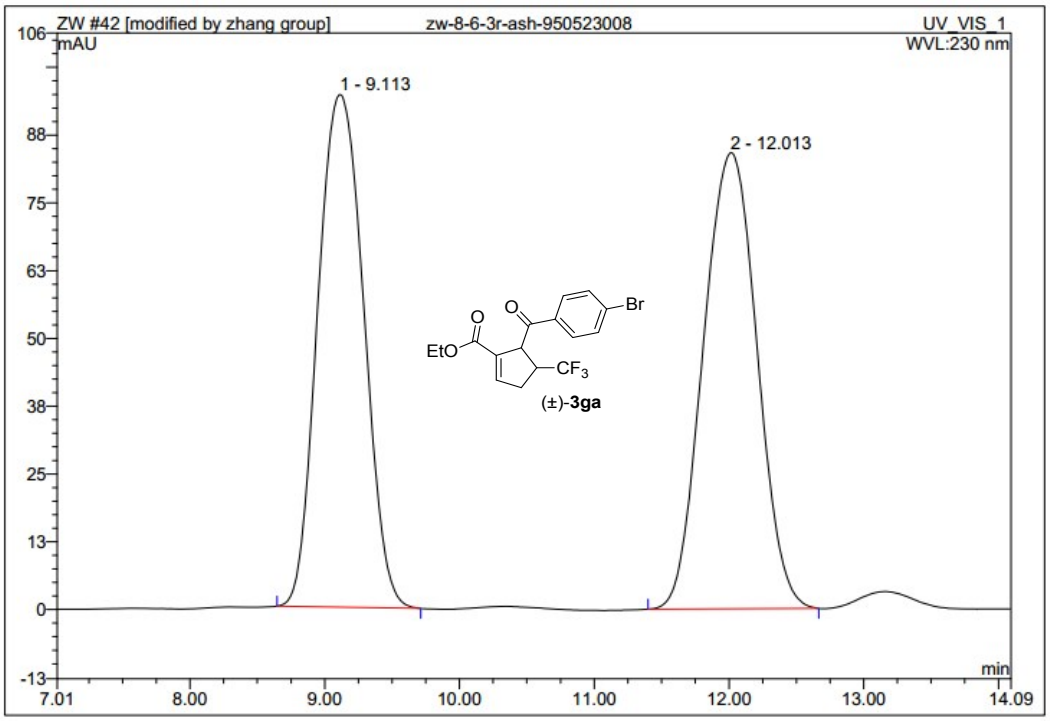


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.83	n.a.	391.038	154.905	98.48	n.a.	BMB*
2	11.61	n.a.	5.762	2.397	1.52	n.a.	BMB*
Total:			396.799	157.302	100.00	0.000	

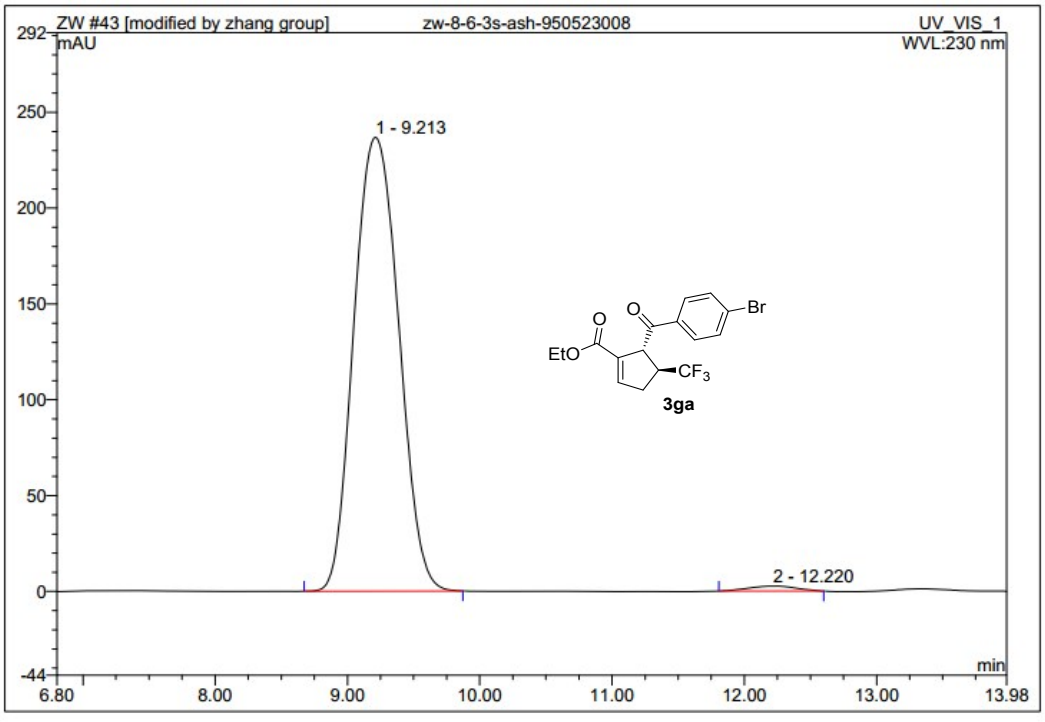


3ga

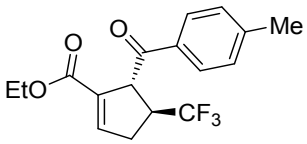
3ga; colorless oil; $[\alpha]_D^{20}$ = + 176.9 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.95–7.93 (m, 2H), 7.68–7.65 (m, 2H), 6.96 (d, *J* = 1.00 Hz, 1H), 5.00–4.98 (m, 1H), 4.14–4.03 (m, 2H), 3.31–3.21 (m, 1H), 3.04–2.98 (m, 1H), 2.82–2.76 (m, 1H), 1.10 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.25, 162.96, 143.58, 135.64, 135.15, 131.99, 130.24, 128.96, 127.05 (q, *J* = 275.75 Hz), 60.87, 50.48 (d, *J* = 1.50 Hz), 46.79 (q, *J* = 28.38 Hz), 33.32 (d, *J* =2.38 Hz), 13.83; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.55 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.21 min, second peak: t_R = 12.22 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}BrF_3NaO_3$ $[M+Na]^+$ = 412.9971, found = 412.9969.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.11	n.a.	94.562	37.252	48.96	n.a.	BMB*
2	12.01	n.a.	84.126	38.834	51.04	n.a.	BMB*
Total:			178.688	76.086	100.00	0.000	

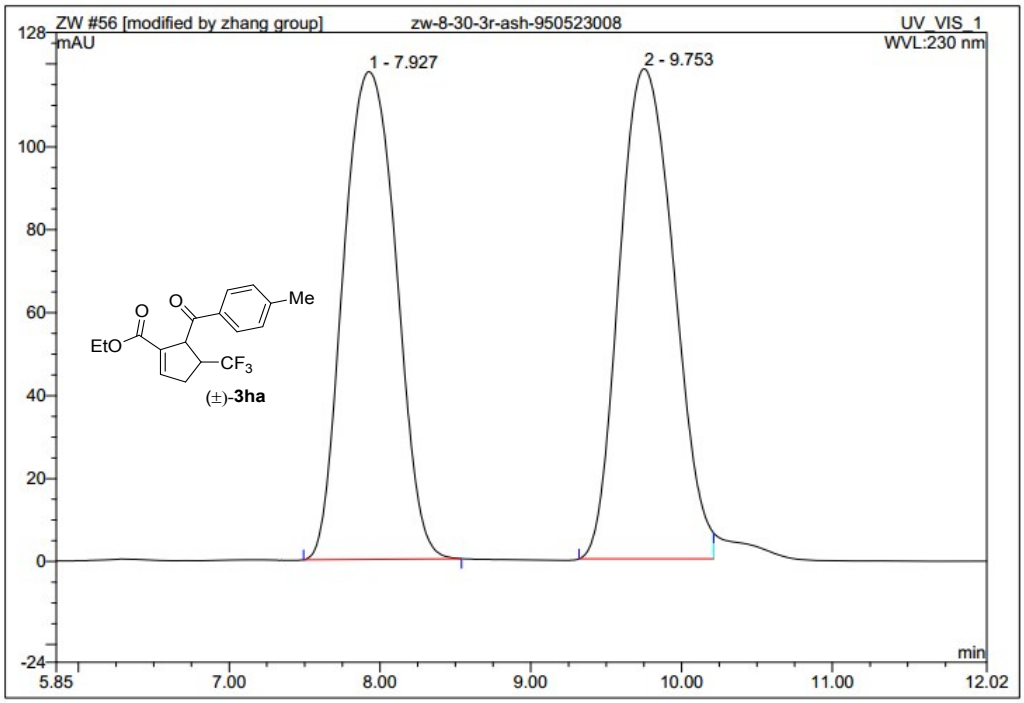


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.21	n.a.	236.854	93.181	98.83	n.a.	BMB*
2	12.22	n.a.	2.654	1.105	1.17	n.a.	BMB*
Total:			239.508	94.286	100.00	0.000	

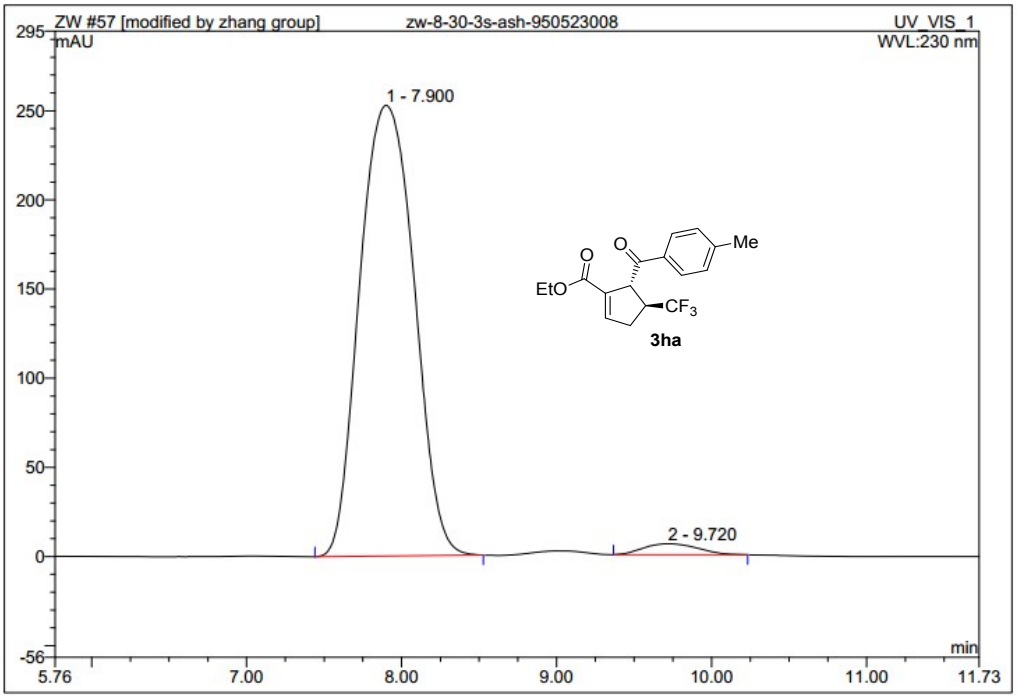


3ha

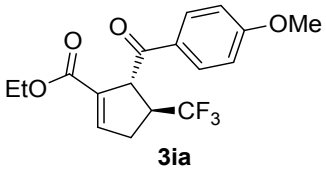
3ha; colorless oil; $[\alpha]_D^{20}$ = + 239.6 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.50 Hz, 2H), 7.31 (d, *J* = 8.00 Hz, 2H), 6.96 (d, *J* = 1.00 Hz, 1H), 5.05–5.03 (m, 1H), 4.12–4.03 (m, 2H), 3.30–3.20 (m, 1H), 3.04–2.97 (m, 1H), 2.81–2.75 (m, 1H), 2.45 (s, 3H), 1.08 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.56, 163.06, 144.56, 143.37, 135.87, 133.82, 129.36, 128.94, 127.20 (q, *J* = 276.13 Hz), 60.73, 50.41 (d, *J* = 1.50 Hz), 46.71 (q, *J* = 28.00 Hz), 33.33 (d, *J* =2.38 Hz), 21.64, 13.83; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.66 ppm; Enantiomeric excess: 95%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 7.90 min, second peak: t_R = 9.72 min; HRMS (ESI) m/z calcd. for $C_{17}H_{17}F_3NaO_3$ $[M+Na]^+$ = 349.1022, found = 349.1023.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.93	n.a.	117.614	47.856	49.05	n.a.	BMB*
2	9.75	n.a.	118.220	49.705	50.95	n.a.	BM *
Total:			235.834	97.561	100.00	0.000	

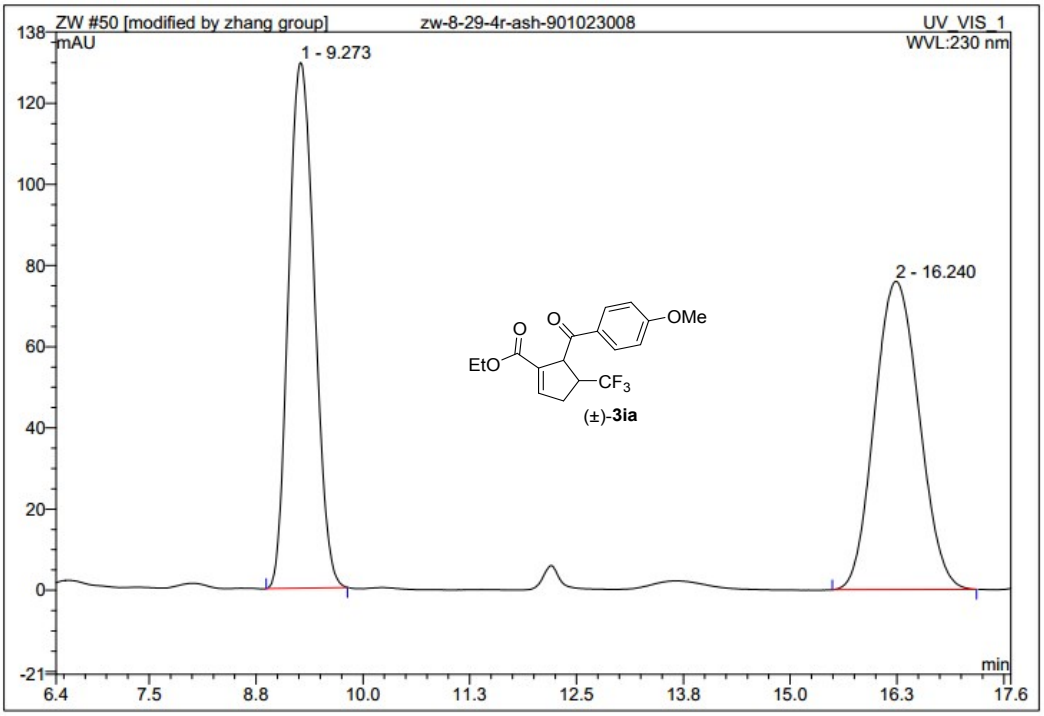


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.90	n.a.	252.792	103.093	97.63	n.a.	BMB*
2	9.72	n.a.	6.142	2.503	2.37	n.a.	BMB*
Total:			258.934	105.597	100.00	0.000	

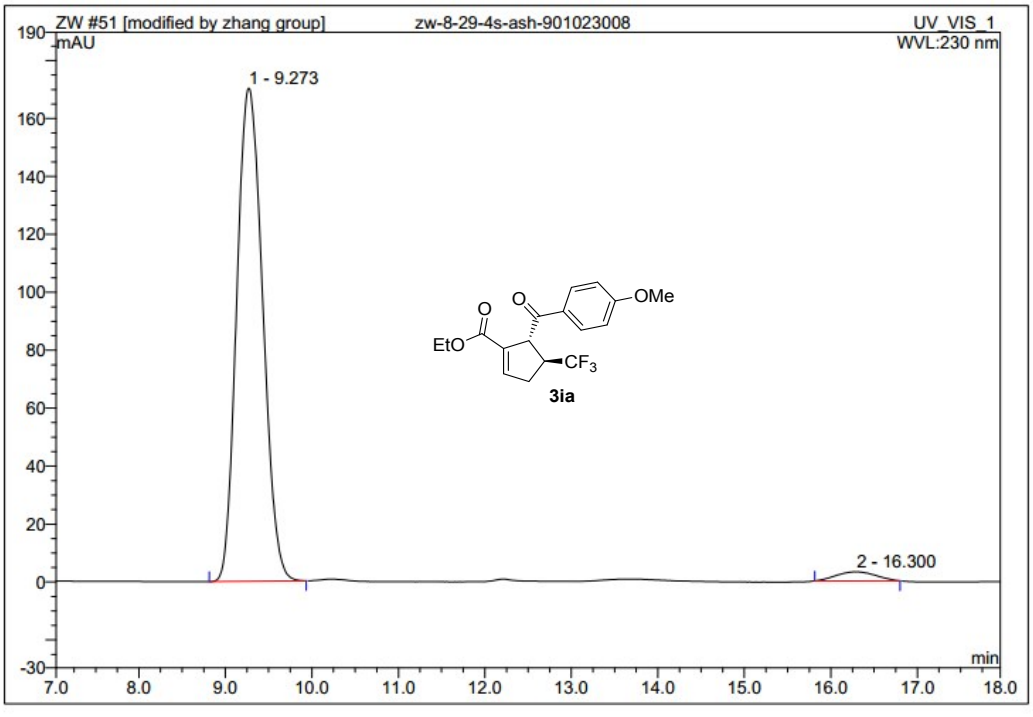


3ia

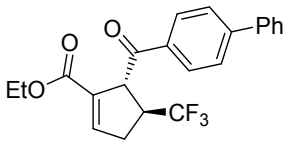
3ia; colorless oil; $[\alpha]_D^{20}$ = + 242.7 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 8.08–8.04 (m, 2H), 6.99–6.94 (m, 3H), 5.01–5.00 (m, 1H), 4.12–4.00 (m, 2H), 4.12–3.88 (s, 3H), 3.29–3.17 (m, 1H), 3.03–2.95 (m, 1H), 2.79–2.72 (m, 1H), 1.08 (t, *J* = 7.20 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.30, 163.98, 163.10, 143.34, 135.85, 131.20, 129.25, 127.23 (q, *J* = 275.80 Hz), 113.83, 60.69, 55.44, 50.20 (d, *J* = 1.50 Hz), 46.67 (q, *J* = 28.10 Hz), 33.34 (q, *J* =2.60 Hz), 13.81; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.69 ppm; Enantiomeric excess: 95%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.27 min, second peak: t_R = 16.30 min; HRMS (ESI) m/z calcd. for $C_{17}H_{17}F_3NaO_4$ $[M+Na]^+$ = 365.0971, found = 365.0981.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.27	n.a.	129.473	45.390	49.71	n.a.	BMB*
2	16.24	n.a.	75.916	45.923	50.29	n.a.	BMB*
Total:			205.389	91.313	100.00	0.000	

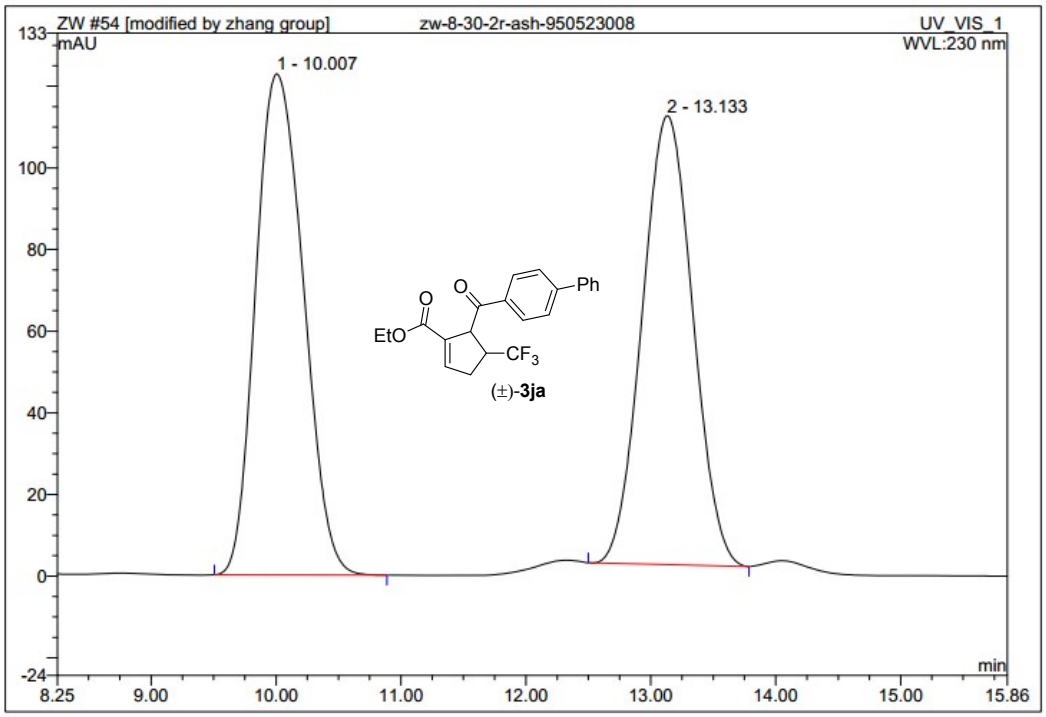


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.27	n.a.	170.201	59.091	97.28	n.a.	BMB*
2	16.30	n.a.	3.128	1.652	2.72	n.a.	BMB*
Total:			173.329	60.744	100.00	0.000	

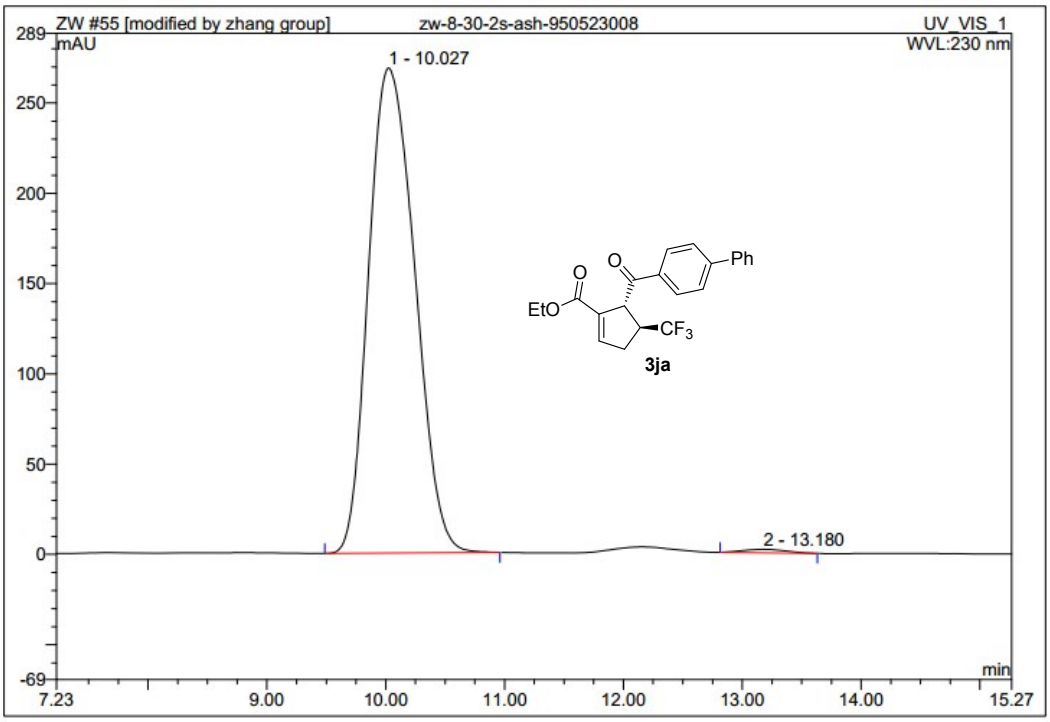


3ja

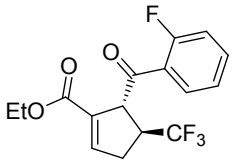
3ja; colorless oil; $[\alpha]_D^{20}$ = + 254.3 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.18–8.17 (m, 2H), 7.77–7.75 (m, 2H), 7.68–7.66 (m, 2H), 7.52–7.49 (m, 2H), 7.45–7.42 (m, 1H), 6.99 (d, *J* = 1.00 Hz, 1H), 5.12–5.11 (m, 1H), 4.15–4.06 (m, 2H), 3.36–3.27 (m, 1H), 3.08–3.01 (m, 1H), 2.85–2.79 (m, 1H), 1.11 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.61, 163.08, 146.25, 143.47, 139.73, 135.86, 135.02, 129.39, 128.92, 128.28, 127.29, 127.18, 126.68 (q, *J* = 274.25 Hz), 60.81, 50.34 (d, *J* = 1.38 Hz), 46.77 (q, *J* = 28.00 Hz), 33.37 (q, *J* = 2.50 Hz), 13.81; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.54 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 10.02 min, second peak: t_R = 13.18 min; HRMS (ESI) m/z calcd. for $C_{22}H_{19}F_3NaO_3$ $[M+Na]^+$ = 411.1178, found = 411.1182.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.01	n.a.	122.735	54.253	51.30	n.a.	BMB*
2	13.13	n.a.	109.974	51.498	48.70	n.a.	BMB*
Total:			232.709	105.751	100.00	0.000	



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.03	n.a.	268.595	120.971	99.33	n.a.	BMB*
2	13.18	n.a.	1.992	0.813	0.67	n.a.	BMB*
Total:			270.587	121.783	100.00	0.000	



3ka

3ka; colorless oil; $[\alpha]_D^{20}$ = + 271.8 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.88–8.85 (m, 1H), 7.59–7.54 (m, 1H), 7.28–7.25 (m, 1H), 7.21–7.17 (m, 1H), 6.94–6.93 (m, 1H), 5.08–5.05 (m, 1H), 4.12–4.02 (m, 2H), 3.41–3.31 (m, 1H), 3.02–2.96 (m, 1H), 2.80–2.74 (m, 1H), 1.09 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 198.73 (d, *J* = 4.00 Hz), 162.99, 161.66 (d, *J* = 254.13 Hz), 143.36, 135.90, 134.90 (d, *J* = 9.00 Hz), 131.01 (d, *J* = 1.75 Hz), 126.95 (q, *J* = 275.63 Hz), 125.72 (d, *J* = 11.50 Hz), 124.38 (d, *J* = 3.25 Hz), 116.86 (d, *J* = 23.75 Hz), 60.79, 54.71–54.63 (m), 46.78 (q, *J* = 28.25 Hz), 33.19 (q, *J* = 2.63 Hz), 13.73; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.76, -110.04 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak OJ-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 7.47 min, second peak: t_R = 8.14 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}F_4NaO_3$ $[M+Na]^+$

= 353.0771, found = 353.0779.

SHIMADZU LabSolutions 分析报告

<样品信息>

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样品ID	:		
数据文件名	: zw-2f-rac--ojh950510.lcd		
方法文件名	: OJ-H-50%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/1 18:37:55	分析者	: System Administrator
处理日期	: 2020/6/1 18:50:40	处理者	: System Administrator

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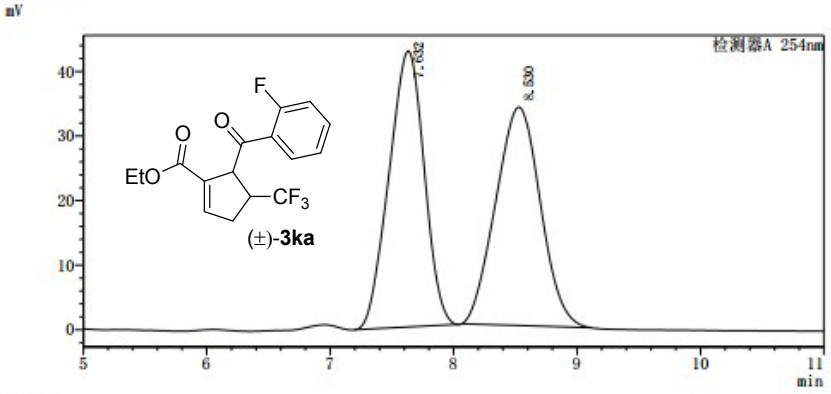

<峰表>

检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	7.632	847938	42737	49.937		M	
2	8.530	850062	33810	50.063		M	
总计		1698000	76547				

SHIMADZU LabSolutions 分析报告

<样品信息>

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样品ID	:		
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进样体积	: 1 uL		
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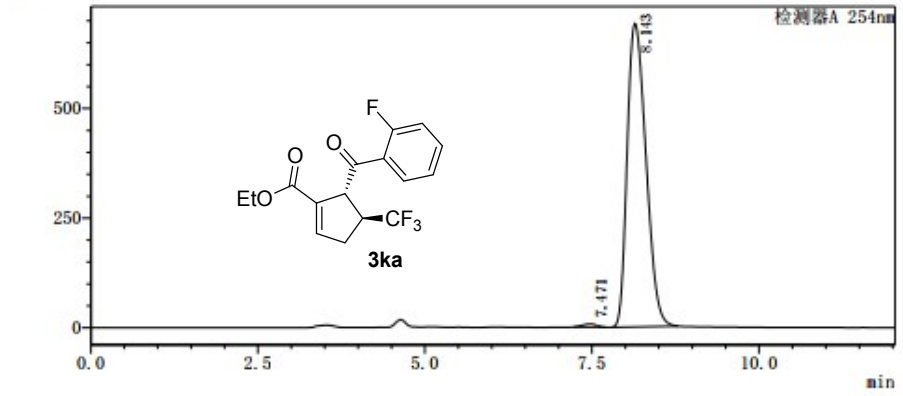
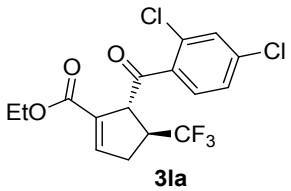


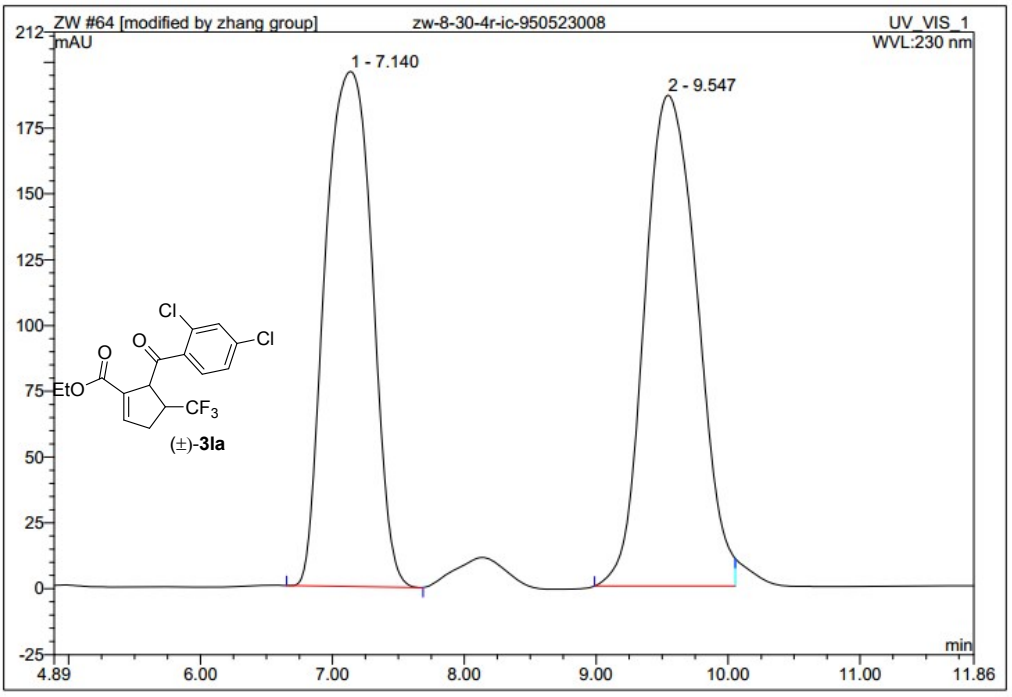
<峰表>

检测器A 254nm

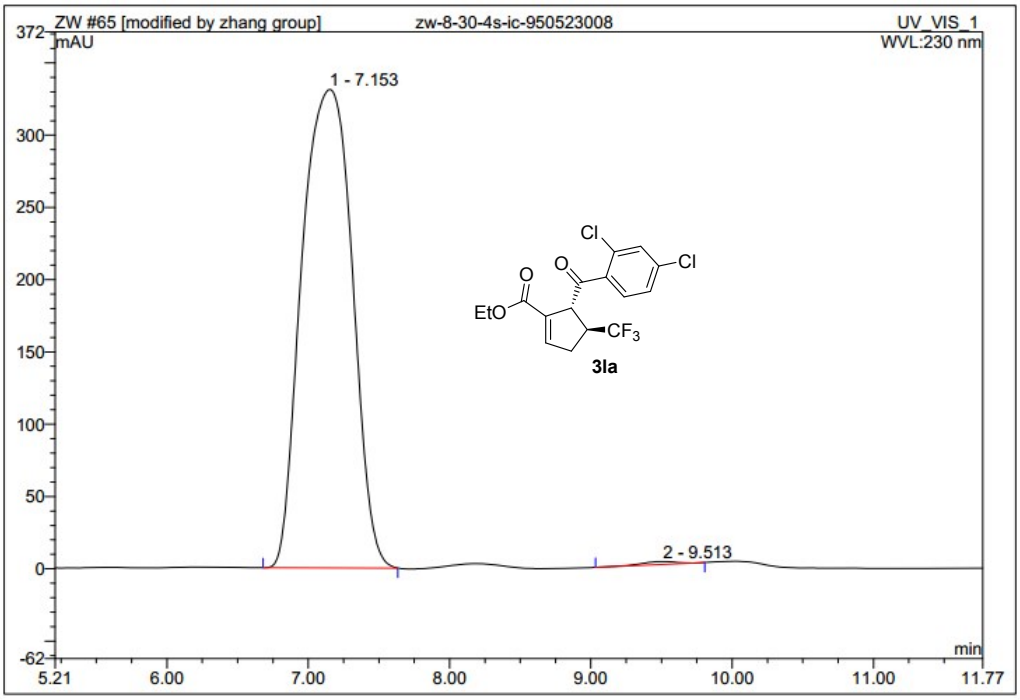
峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	7.471	102905	6898	0.755		M	
2	8.143	13519307	691748	99.245		M	
总计		13622211	698646				



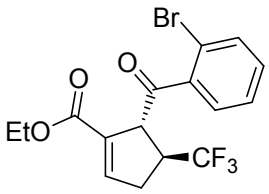
3la; colorless oil; $[\alpha]_D^{20}$ = + 203.8 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.68 (d, *J* = 8.50 Hz, 1H), 7.50 (d, *J* = 2.00 Hz, 1H), 7.38–7.36 (m, 1H), 6.93 (d, *J* = 1.00 Hz, 1H), 4.84–4.83 (m, 1H), 4.18–4.06 (m, 2H), 3.42–3.30 (m, 1H), 3.05–2.99 (m, 1H), 2.80–2.75 (m, 1H), 1.19 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.84, 163.03, 143.82, 137.80, 136.42, 134.85, 132.72, 130.71, 130.26, 127.07, 126.94 (q, *J* = 276.13 Hz), 61.00, 55.32 (q, *J* = 1.38 Hz), 46.50 (q, *J* = 28.50 Hz), 33.32 (d, *J* = 2.50 Hz), 13.93; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -72.20 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak IC, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 7.15 min, second peak: t_R = 9.51 min; HRMS (ESI) m/z calcd. for $C_{16}H_{13}Cl_2F_3NaO_3$ $[M+Na]^+$ = 403.0086, found = 403.0093.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.14	n.a.	195.682	80.683	48.29	n.a.	BMB*
2	9.55	n.a.	186.523	86.392	51.71	n.a.	BM *
Total:			382.204	167.075	100.00	0.000	

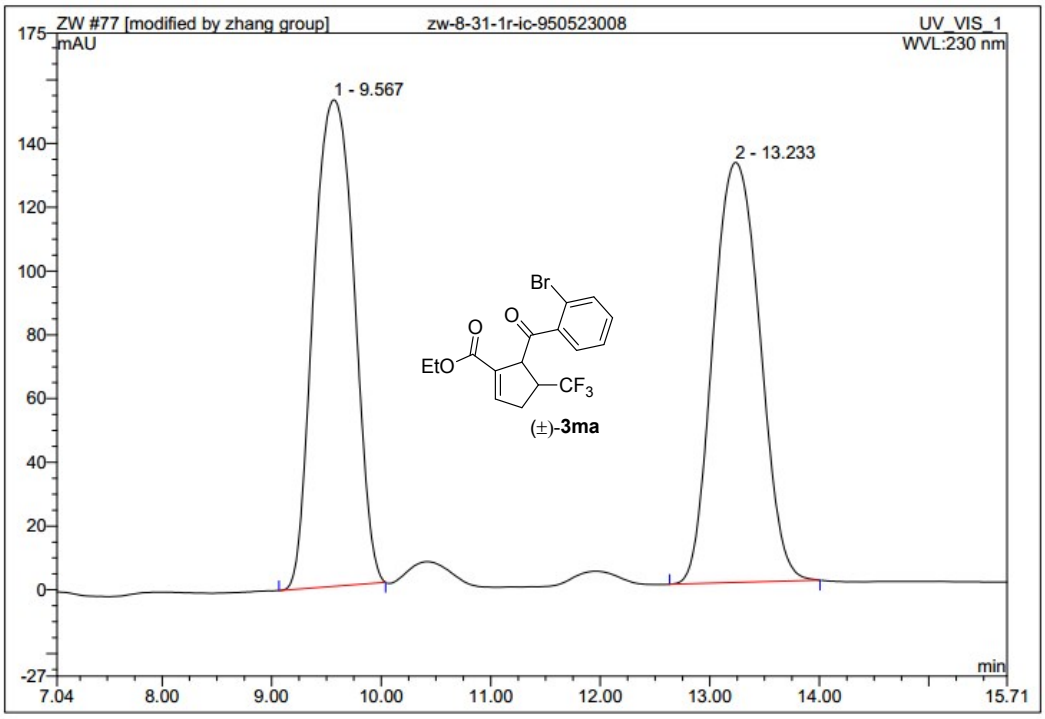


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.15	n.a.	331.017	136.989	99.60	n.a.	BMB*
2	9.51	n.a.	1.924	0.552	0.40	n.a.	BMB*
Total:			332.941	137.542	100.00	0.000	

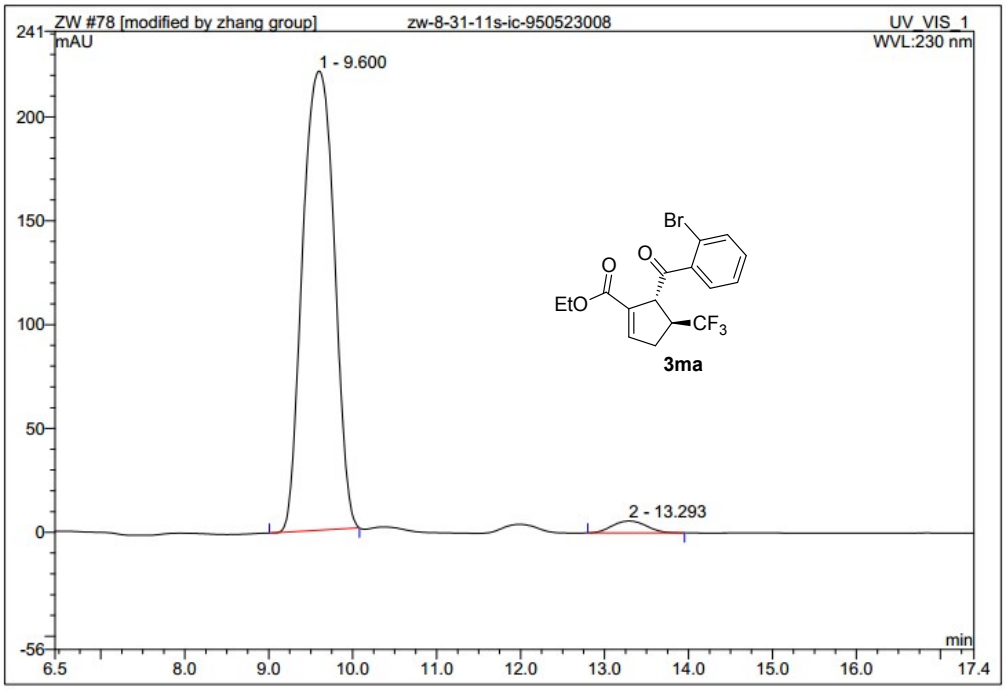


3ma

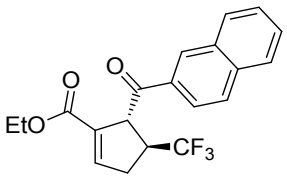
3ma; colorless oil; $[\alpha]_D^{20}$ = + 240.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.69–7.66 (m, 2H), 7.44–7.41 (m, 1H), 7.36–7.33 (m, 1H), 6.94 (s, 1H), 4.84 (s, 1H), 4.18–4.03 (m, 2H), 3.44–3.35 (m, 1H), 3.06–2.99 (m, 1H), 2.79–2.76 (m, 1H), 1.17 (t, *J* = 7.50 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 200.97, 163.07, 143.76, 140.06, 134.69, 133.99, 132.12, 128.97, 127.20, 127.05 (q, *J* = 276.13 Hz), 119.68, 60.92, 55.56 (d, *J* = 1.25 Hz), 44.97 (q, *J* = 28.50 Hz), 33.29 (q, *J* = 2.50 Hz), 13.90; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -72.47 ppm; Enantiomeric excess: 94%, determined by HPLC (Chiralpak IC, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.60 min, second peak: t_R = 13.29 min; HRMS (ESI) m/z calcd. for $C_{16}H_{14}BrF_3NaO_3$ $[M+Na]^+$ = 412.9971, found = 412.9969.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.57	n.a.	152.612	65.437	50.13	n.a.	BMB*
2	13.23	n.a.	131.791	65.105	49.87	n.a.	BMB*
Total:			284.403	130.542	100.00	0.000	

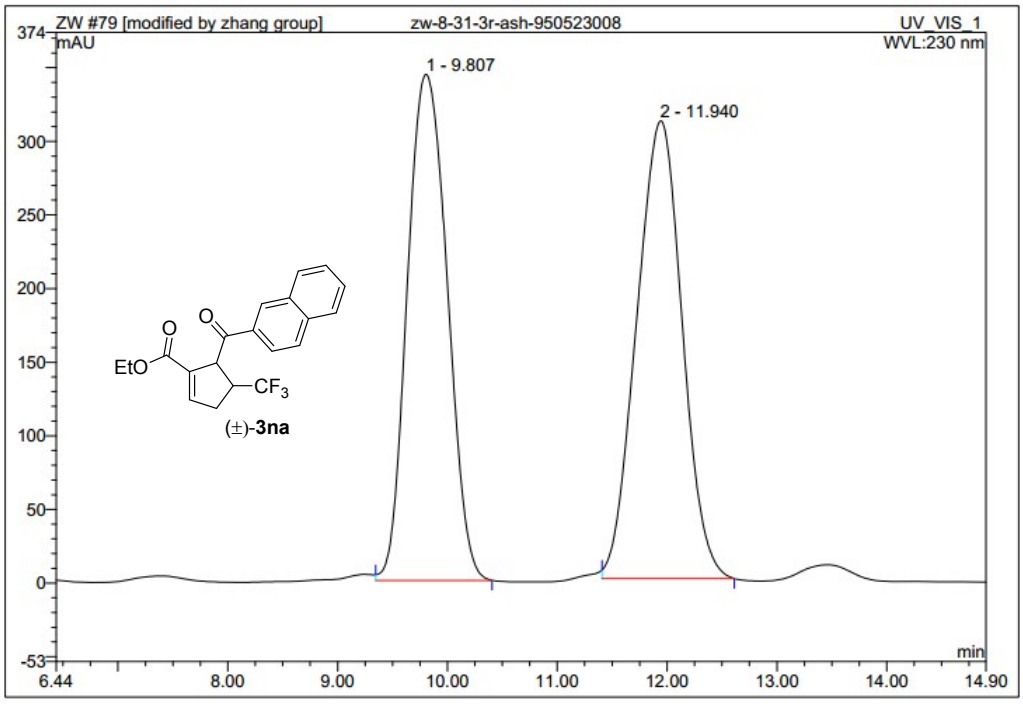


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.60	n.a.	221.053	96.678	97.26	n.a.	BMB*
2	13.29	n.a.	5.759	2.726	2.74	n.a.	BMB*
Total:			226.812	99.405	100.00	0.000	

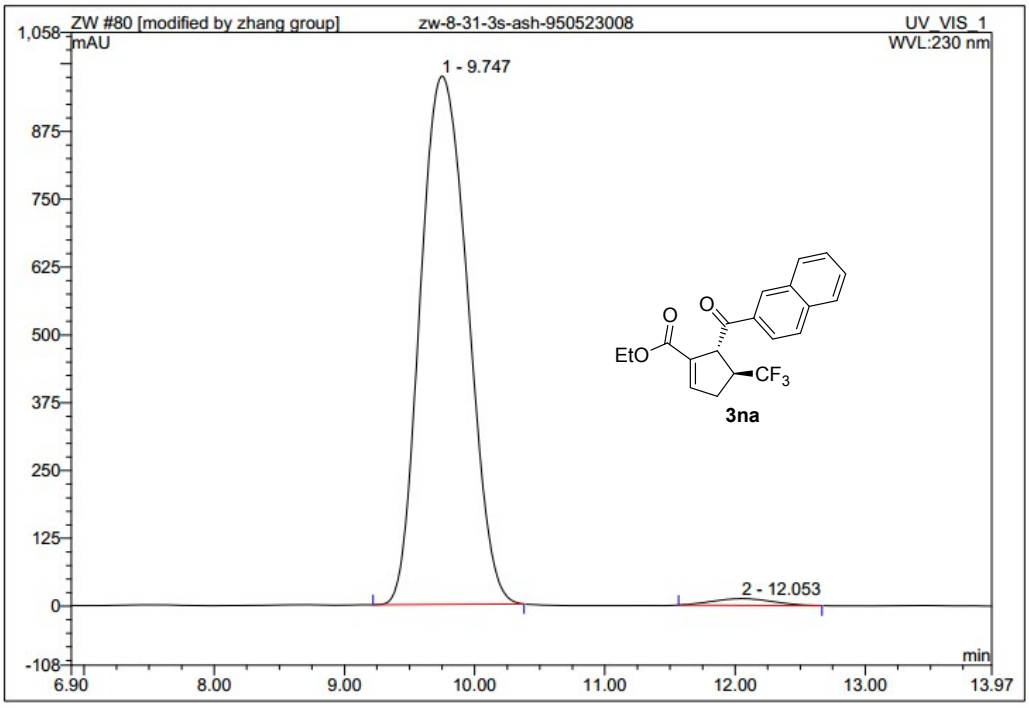


3na

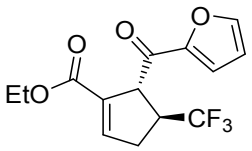
3na; colorless oil; $[\alpha]_D^{20}$ = + 50.8 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.66 (s, 1H), 8.13–8.11 (m, 1H), 8.03 (d, *J* = 8.00 Hz, 1H), 7.96–7.90 (m, 2H), 7.65–7.57 (m, 2H), 7.01 (s, 1H), 5.26–5.24 (m, 1H), 4.11–4.02 (m, 2H), 3.39–3.29 (m, 1H), 3.08–3.02 (m, 1H), 2.86–2.80 (m, 1H), 1.04 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.88, 163.05, 143.52, 135.84, 135.78, 133.64, 132.45, 130.80, 129.82, 128.74, 128.53, 127.68, 127.22 (q, *J* = 276.00 Hz), 126.77, 124.21, 60.75, 50.62 (d, *J* = 1.25 Hz), 46.81 (q, *J* = 28.00 Hz), 33.35 (q, *J* = 2.50 Hz), 13.77; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.52 ppm; Enantiomeric excess: 97%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.74 min, second peak: t_R = 12.05 min; HRMS (ESI) m/z calcd. for $C_{20}H_{17}F_3NaO_3$ $[M+Na]^+$ = 385.1022, found = 385.1022.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.81	n.a.	343.679	144.758	49.20	n.a.	MB*
2	11.94	n.a.	310.966	149.437	50.80	n.a.	MB*
Total:			654.645	294.195	100.00	0.000	

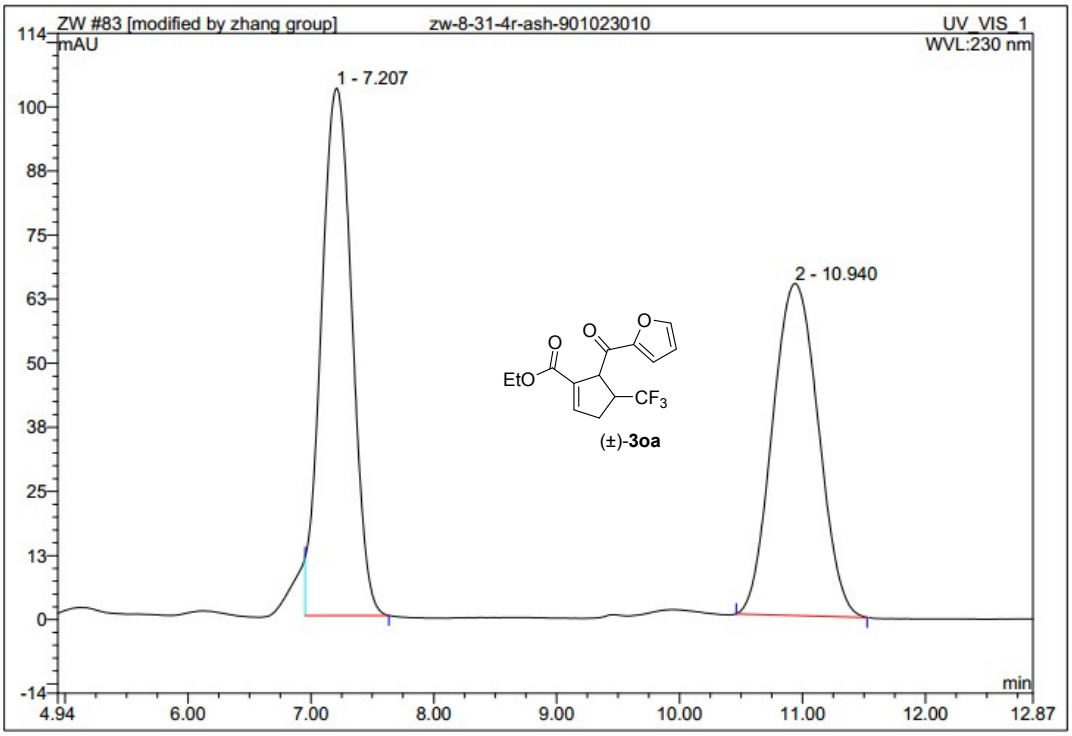


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.75	n.a.	973.641	409.494	98.40	n.a.	BMB*
2	12.05	n.a.	12.339	6.646	1.60	n.a.	BMB*
Total:			985.980	416.139	100.00	0.000	

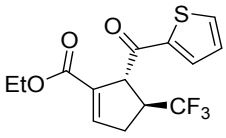


3oa

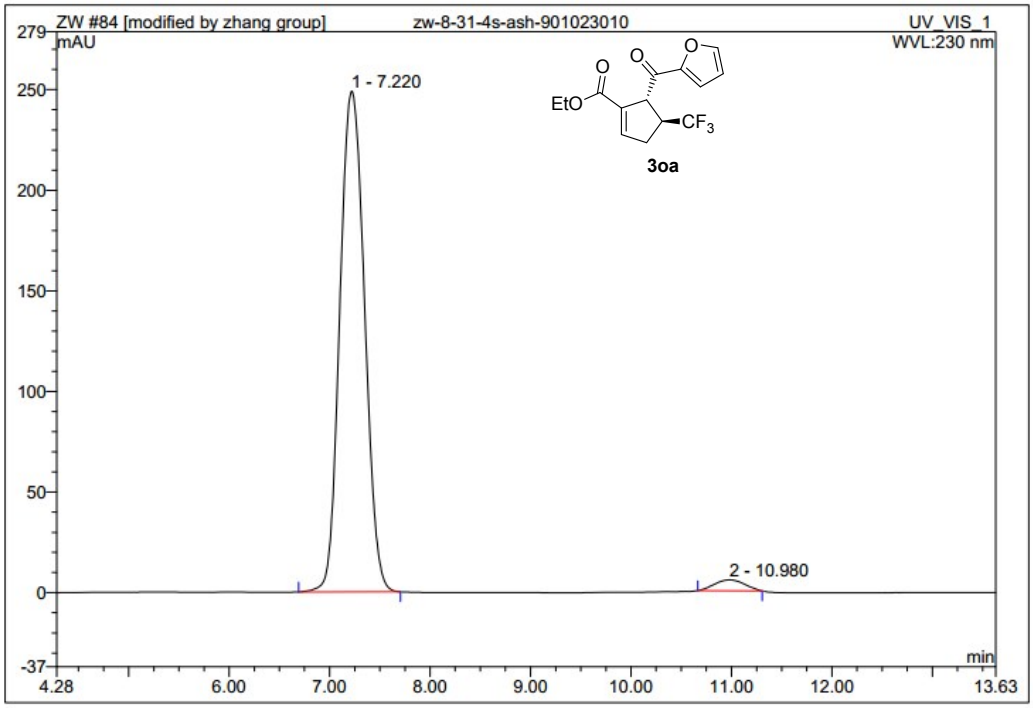
3oa; white solid; $[\alpha]_D^{20}$ = + 196.7 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.68–7.67 (m, 1H), 7.37 (d, *J* = 4.00 Hz, 1H), 6.94 (d, *J* = 1.00 Hz, 1H), 6.61–6.60 (m, 1H), 4.83–4.82 (m, 1H), 4.13–4.04 (m, 2H), 3.33–3.23 (m, 1H), 3.02–2.96 (m, 1H), 2.80–2.74 (m, 1H), 1.11 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.83, 162.89, 152.00, 147.44, 143.75, 135.06, 127.00 (q, *J* = 275.63 Hz), 119.00, 112.62, 60.75, 51.43 (q, *J* = 2.00 Hz), 46.34 (q, *J* = 28.25 Hz), 33.30–33.26 (m), 13.77; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.84 ppm; Enantiomeric excess: 95%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 7.22 min, second peak: t_R = 10.98 min; HRMS (ESI) m/z calcd. for $C_{14}H_{13}F_3NaO_4$ $[M+Na]^+$ = 325.0658, found = 325.0662.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.21	n.a.	102.803	29.701	51.57	n.a.	MB*
2	10.94	n.a.	64.718	27.892	48.43	n.a.	BMB*
Total:			167.521	57.593	100.00	0.000	

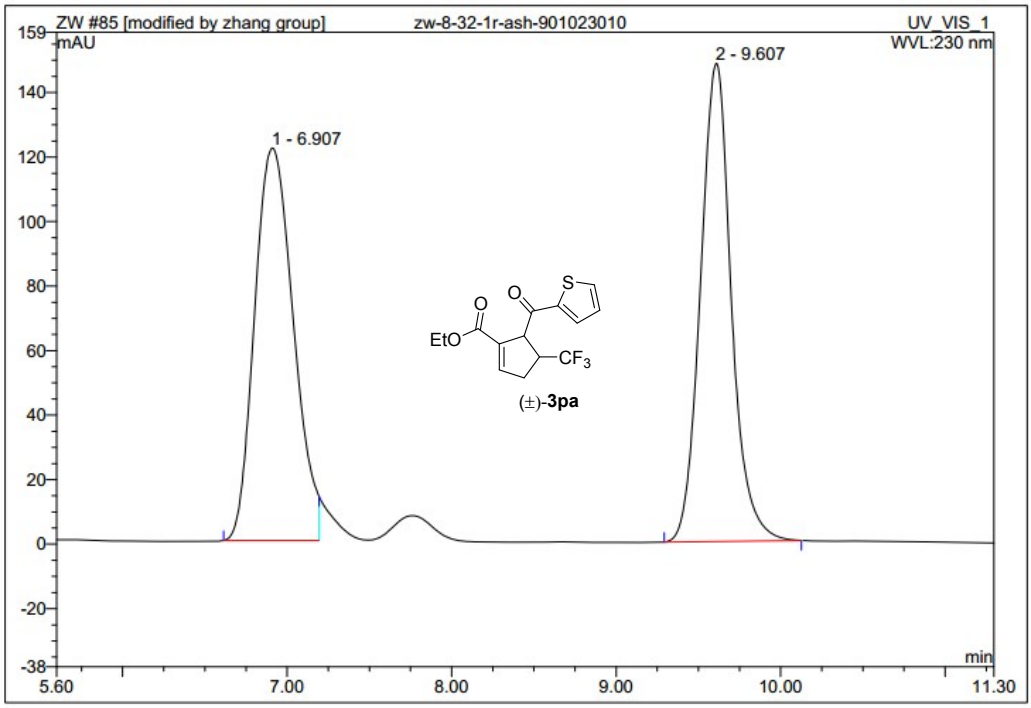


3pa

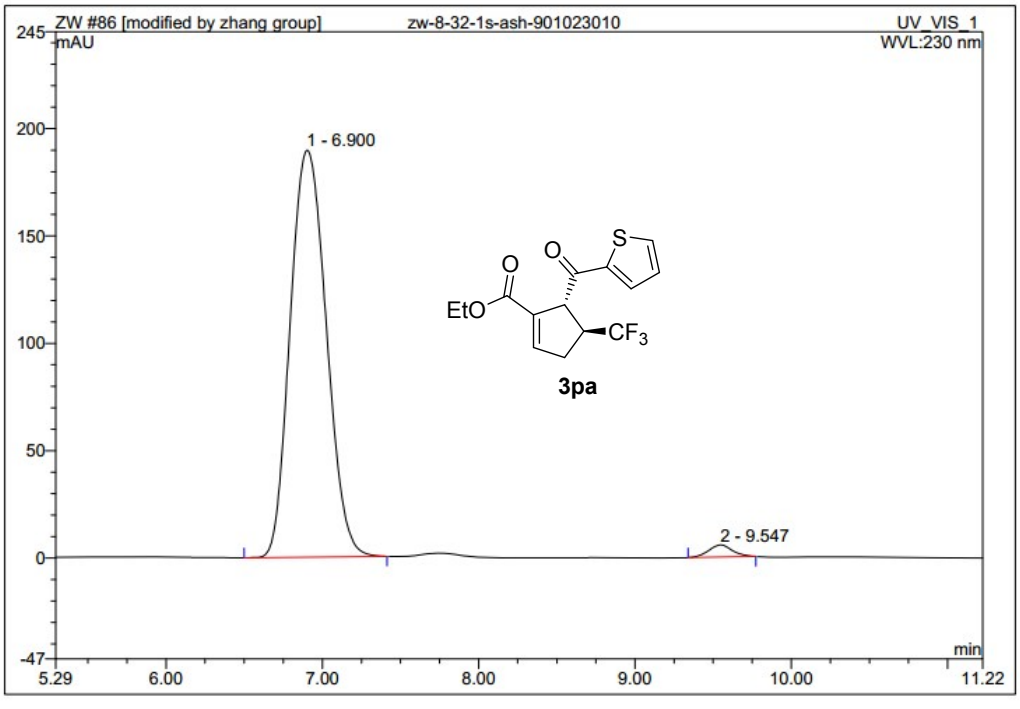


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.22	n.a.	248.799	70.782	97.43	n.a.	BMB*
2	10.98	n.a.	5.404	1.869	2.57	n.a.	BMB*
Total:			254.203	72.651	100.00	0.000	

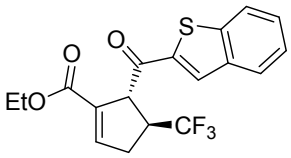
3pa; white solid; $[\alpha]_D^{20}$ = + 221.6 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.92–7.91 (m, 1H), 7.74–7.73 (m, 1H), 7.20–7.18 (m, 1H), 6.97 (m, 1H), 4.84–4.82 (m, 1H), 4.14–4.04 (m, 2H), 3.34–3.24 (m, 1H), 3.04–2.97 (m, 1H), 2.80–2.75 (m, 1H), 1.10 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.05, 162.90, 143.77, 143.40, 135.18, 133.32, 128.37, 127.08 (q, *J* = 275.88 Hz), 60.76, 52.49 (d, *J* = 1.50 Hz), 46.57 (q, *J* = 28.25 Hz), 33.35 (q, *J* = 2.38 Hz), 13.70; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.75 ppm; Enantiomeric excess: 96%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 6.90 min, second peak: t_R = 9.54 min; HRMS (ESI) m/z calcd. for $C_{14}H_{13}F_3NaO_3S$ $[M+Na]^+$ = 341.0430, found = 341.0431.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.91	n.a.	121.679	33.181	50.75	n.a.	BM *
2	9.61	n.a.	148.187	32.200	49.25	n.a.	BMB*
Total:			269.866	65.381	100.00	0.000	

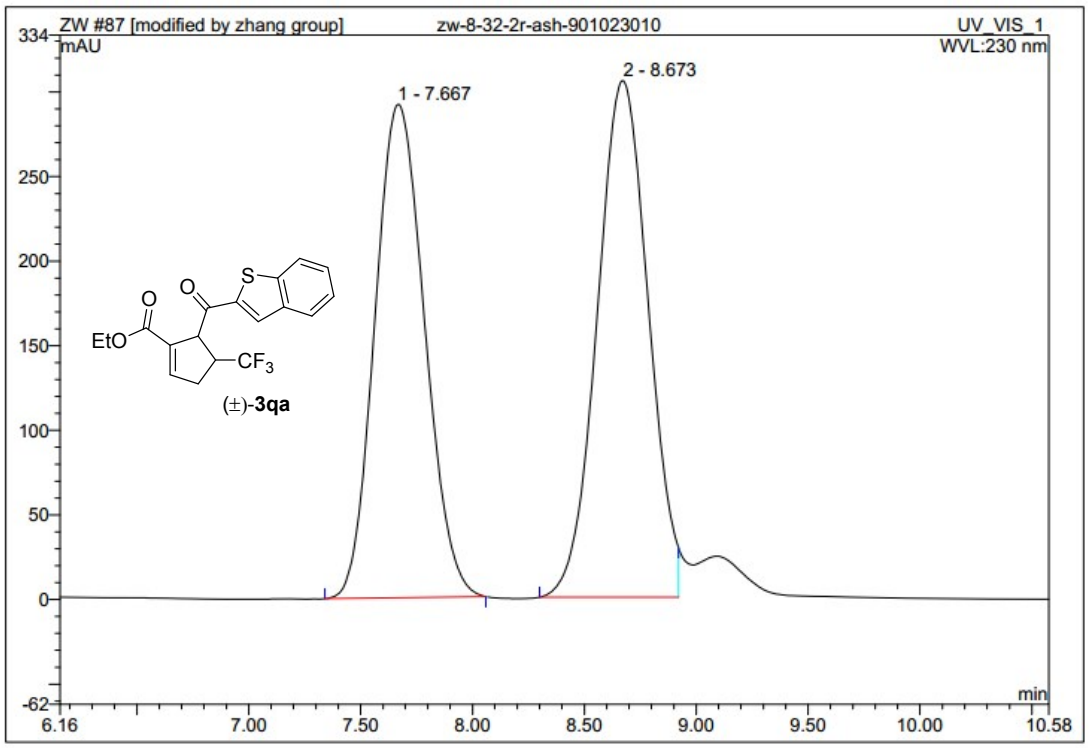


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.90	n.a.	189.594	50.747	98.06	n.a.	BMB*
2	9.55	n.a.	5.710	1.002	1.94	n.a.	BMB*
Total:			195.304	51.750	100.00	0.000	

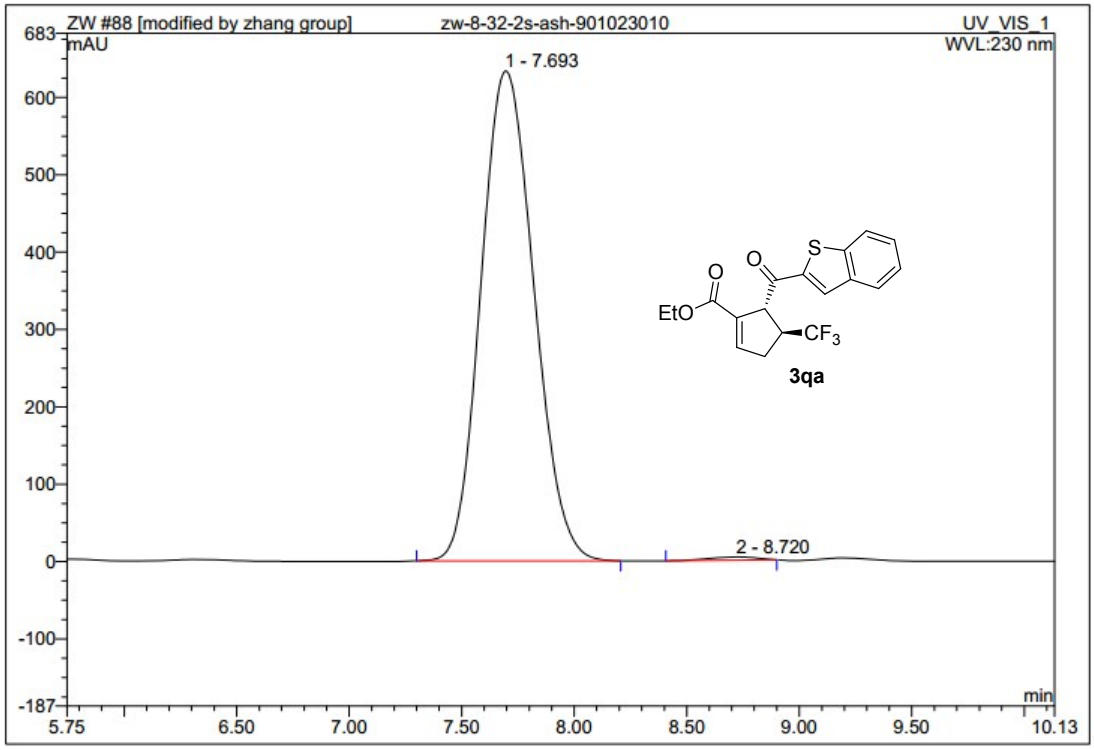


3qa

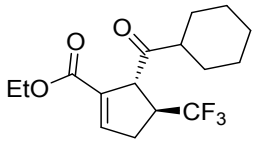
3qa; white solid; $[\alpha]_D^{20}$ = + 229.9 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.19 (s, 1H), 7.96 (d, *J* = 7.50 Hz, 1H), 7.90 (d, *J* = 8.00 Hz, 1H), 7.52–7.48 (m, 1H), 7.46–7.43 (m, 1H), 7.00 (s, 1H), 5.00–4.98 (m, 1H), 4.15–4.05 (m, 2H), 3.40–3.31 (m, 1H), 3.08–3.01 (m, 1H), 2.85–2.80 (m, 1H), 1.10 (t, *J* = 7.00 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.75, 162.89, 143.87, 143.16, 142.81, 139.09, 135.19, 130.62, 127.76, 127.09 (q, *J* = 276.00 Hz), 122.89, 60.86, 52.20 (q, *J* = 1.50 Hz), 46.66 (q, *J* = 28.25 Hz), 33.38 (d, *J* = 2.25 Hz), 13.78; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.62 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 7.69 min, second peak: t_R = 8.72 min; HRMS (ESI) m/z calcd. for $C_{18}H_{15}F_3NaO_3S$ $[M+Na]^+$ = 391.0586, found = 391.0592.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.67	n.a.	291.724	75.043	48.20	n.a.	BMB*
2	8.67	n.a.	305.393	80.637	51.80	n.a.	BM *
Total:			597.118	155.681	100.00	0.000	



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.69	n.a.	633.062	172.611	99.39	n.a.	BMB*
2	8.72	n.a.	4.276	1.053	0.61	n.a.	BMB*
Total:			637.338	173.664	100.00	0.000	



3ra

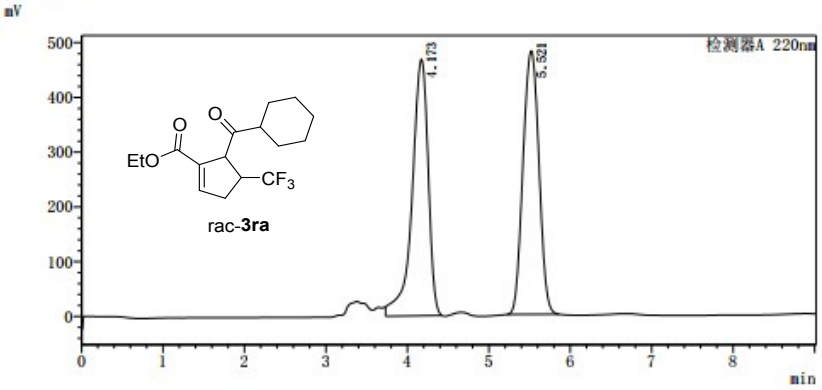
3ra; colorless oil; $[\alpha]_D^{20}$ = + 92.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 6.86 (s, 1H), 4.27–4.16 (m, 3H), 3.17–3.05 (m, 1H), 2.96–2.78 (m, 2H), 2.73–2.68 (m, 1H), 1.98–1.92 (m, 2H), 1.85–1.81 (m, 2H), 1.72–1.69 (m, 1H), 1.45–1.24 (m, 8H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 212.77, 163.36, 143.41, 135.32, 127.23 (q, *J* = 275.88 Hz), 60.86, 54.22, 51.06, 45.38 (d, *J* = 35.0 Hz), 33.32, 28.29, 28.02, 28.82, 25.79, 25.47, 14.13; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -72.47 ppm; Enantiomeric excess: 61%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 4.16 min, second peak: t_R = 5.52 min; HRMS (ESI) m/z calcd. for $C_{16}H_{21}F_3NaO_3$ $[M+Na]^+$ = 341.1335, found = 341.0339.

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: huanjijirac-ash950510		
样品ID	:		
数据文件名	: huanjijirac-ash9505.lcd		
方法文件名	: AS-H-60%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/5/30 12:56:27	分析者	: System Administrator
处理日期	: 2020/5/30 13:05:28	处理者	: System Administrator

〈色谱图〉



〈峰表〉

检测器A 220nm

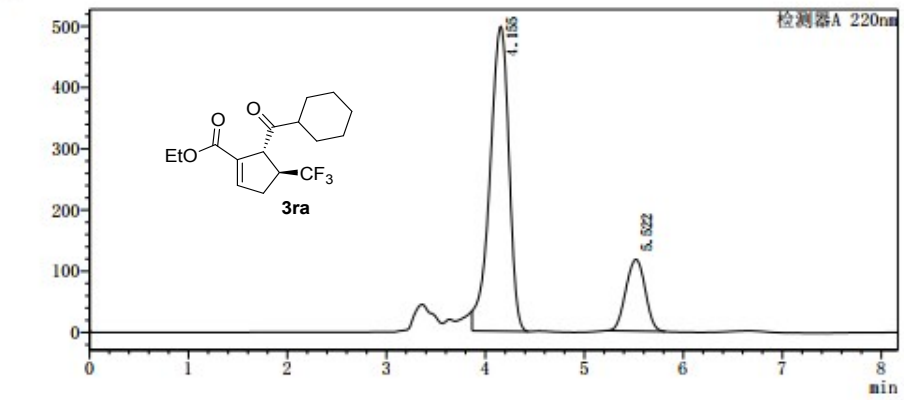
峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	4.173	6418732	468423	49.510		M	
2	5.521	6545768	481073	50.490		M	
总计		12964500	949496				

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: huanjijichiral-ash950510		
样品ID	:		
数据文件名	: huanjijichiral-ash950510.lcd		
方法文件名	: AS-H-60%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/5/30 13:08:17	分析者	: System Administrator
处理日期	: 2020/5/30 13:16:27	处理者	: System Administrator

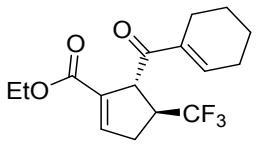
〈色谱图〉



〈峰表〉

检测器A 220nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	4.155	6611005	497612	80.596		M	
2	5.522	1591677	116964	19.404		M	
总计		8202682	614576				



3sa

3sa; colorless oil; $[\alpha]_D^{20}$ = + 57.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.19 (s, 1H), 6.91 (s, 1H), 4.30–4.22 (m, 2H), 4.15–4.10 (m, 1H), 3.98–3.92 (m, 1H), 3.43–3.38 (m, 1H), 2.45–2.32 (m, 3H), 1.67–1.63 (m, 5H), 1.35–1.27 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 196.62, 163.13, 149.25, 139.65, 139.56, 130.64, 126.20 (q, *J* = 281.4 Hz), 60.86, 50.99 (d, *J* = 27.75 Hz), 47.91, 33.43, 26.13, 23.32, 21.80, 21.50; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.62 ppm; Enantiomeric excess: 84%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 7.44 min, second peak: t_R = 13.57 min; HRMS (ESI) m/z calcd. for $C_{16}H_{19}F_3NaO_3$ $[M+Na]^+$ = 339.1179, found = 339.1188.

SHIMADZU LabSolutions 分析报告

<样品信息>

样品名	: zw-xiji-ash-9505230		
样品ID	:		
数据文件名	: ZW.lcd		
方法文件名	: OD-H-95%-40min.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/5/29 16:33:44	分析者	: System Administrator
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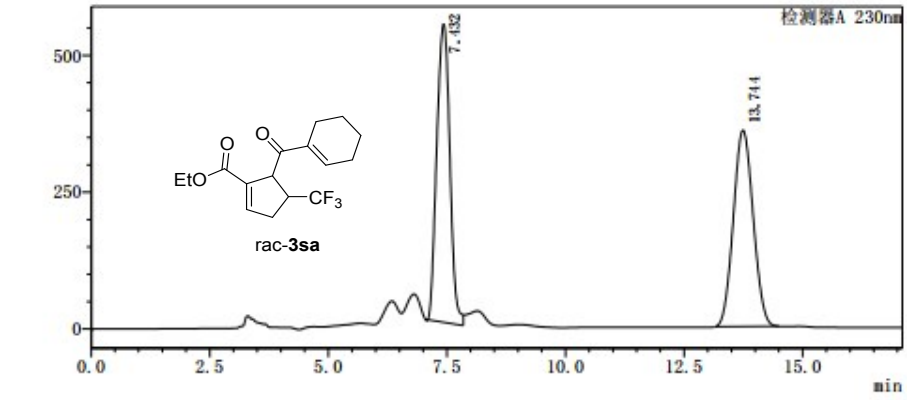
<色谱图>



<峰表>

检测器A 230nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	7.432	10438172	545480	50.076		M	
2	13.744	10406451	358881	49.924		M	
总计		20844622	904361				

SHIMADZU LabSolutions 分析报告

<样品信息>

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样品ID	:		
数据文件名	: zw-xijichiral-ash-9505230s.lcd		
方法文件名	: OD-H-95%-40min.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
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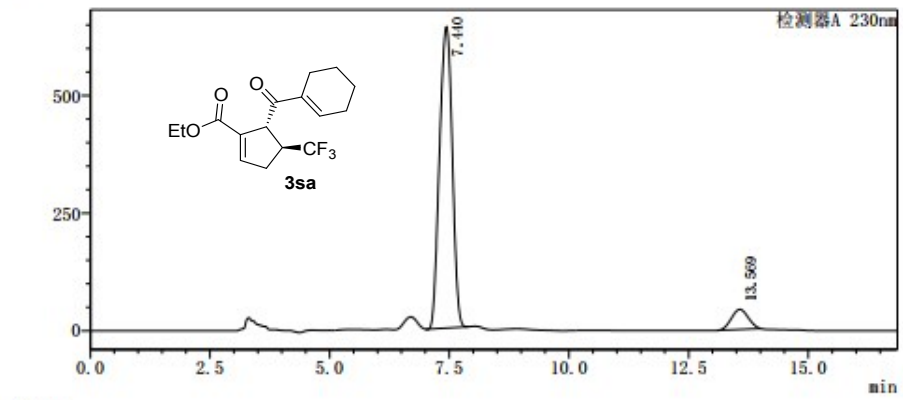
<色谱图>

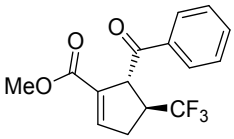


3sa

<峰表>

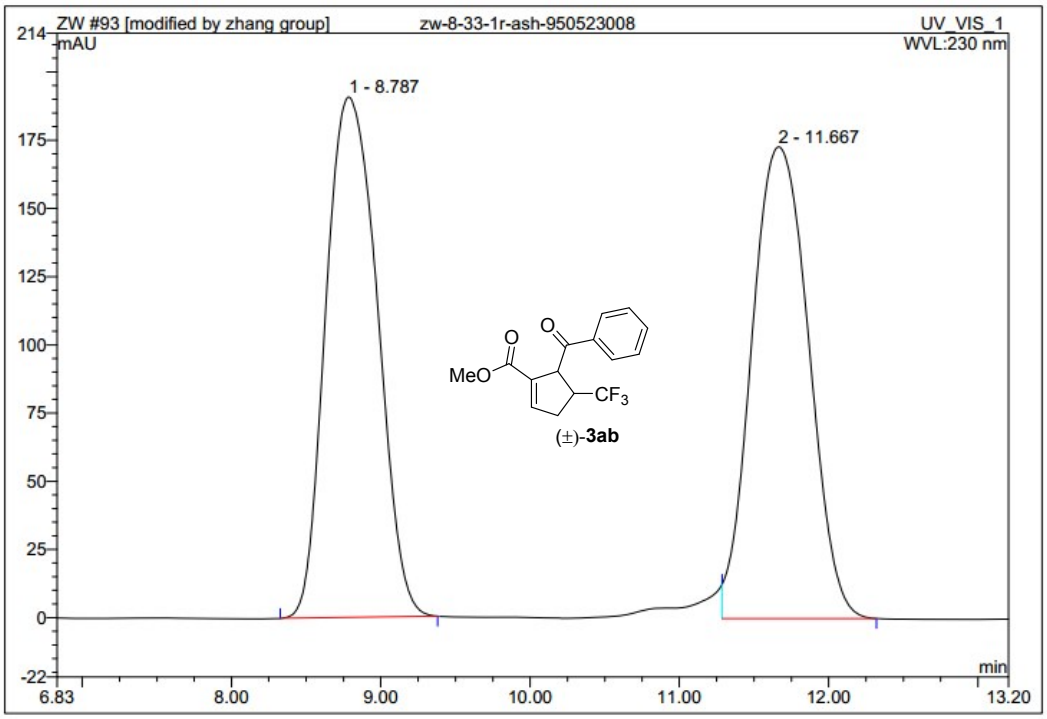
检测器A 230nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	7.440	11876682	640416	91.958		M	
2	13.569	1038591	42533	8.042		M	
总计		12915273	682949				

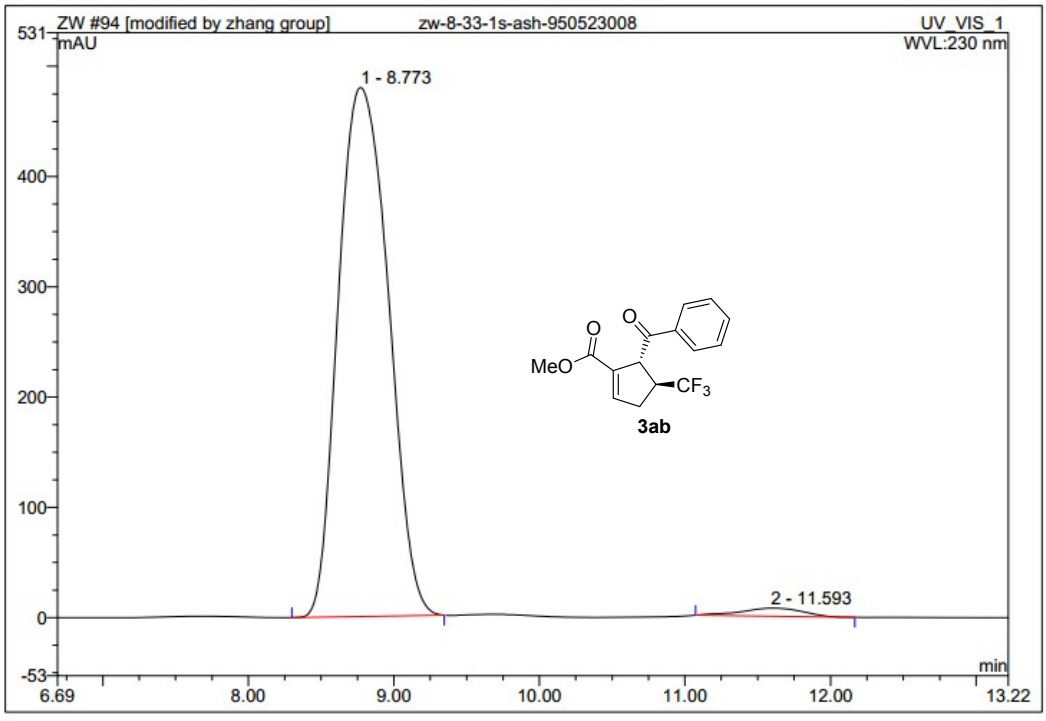


3ab

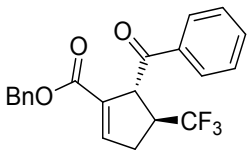
3ab; colorless oil; $[\alpha]_D^{20}$ = + 127.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.08–8.07 (m, 2H), 7.65–7.61 (m, 1H), 7.54–7.51 (m, 2H), 6.97 (d, *J* = 0.50 Hz, 1H), 5.06–5.05 (m, 1H), 3.63 (s, 3H), 3.29–3.20 (m, 1H), 3.04–2.98 (m, 1H), 2.82–2.77 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.87, 163.52, 143.67, 136.23, 135.44, 133.60, 128.85, 128.67, 127.13 (q, *J* = 276.25 Hz), 51.73, 50.70 (q, *J* = 1.50 Hz), 46.57 (q, *J* = 28.13 Hz), 33.39 (d, *J* = 2.50 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.75 ppm; Enantiomeric excess: 96%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 8.77 min, second peak: t_R = 11.59 min; HRMS (ESI) m/z calcd. for $C_{15}H_{13}F_3NaO_3$ $[M+Na]^+$ = 321.0709, found = 321.0710.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.79	n.a.	190.719	76.261	49.90	n.a.	BMB*
2	11.67	n.a.	172.895	76.561	50.10	n.a.	MB*
Total:			363.614	152.822	100.00	0.000	

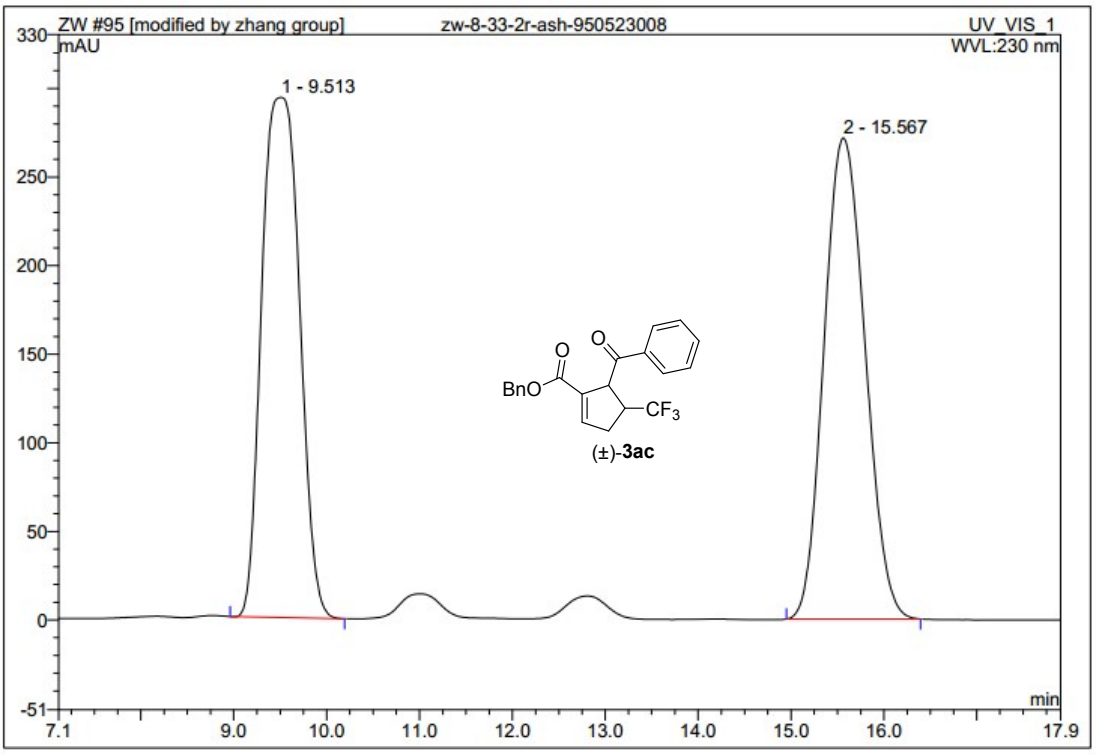


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.77	n.a.	479.825	191.059	98.15	n.a.	BMB*
2	11.59	n.a.	7.317	3.603	1.85	n.a.	BMB*
Total:			487.142	194.662	100.00	0.000	

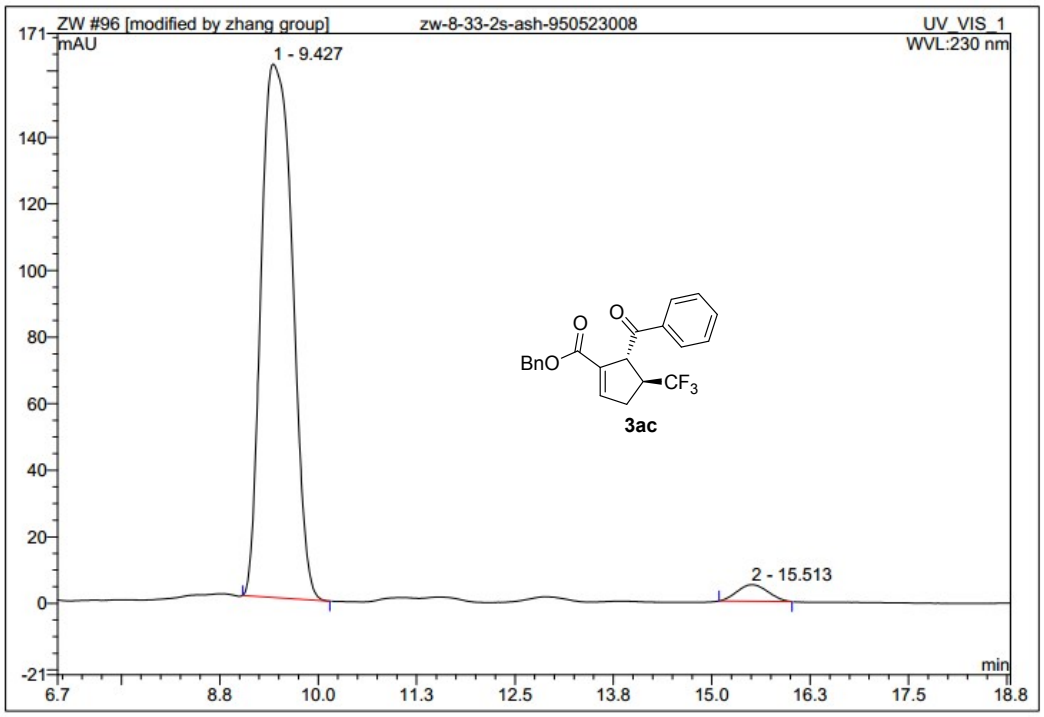


3ac

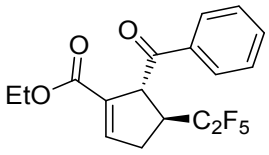
3ac; colorless oil; $[\alpha]_D^{20}$ = + 83.3 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.07–8.05 (m, 2H), 7.64–7.60 (m, 1H), 7.51–7.47 (m, 2H), 7.31–7.27 (m, 3H), 7.20–7.18 (m, 2H), 7.04 (s, 1H), 5.10–5.08 (m, 3H), 3.32–3.22 (m, 1H), 3.06–2.99 (m, 1H), 2.83–2.77 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.87, 162.79, 144.20, 136.16, 135.41, 135.20, 133.57, 128.75, 128.62, 128.39, 128.14, 128.10, 124.89 (q, *J* = 275.88 Hz), 66.54, 50.51 (d, *J* = 1.38 Hz), 46.70 (q, *J* = 28.25 Hz), 33.37 (q, *J* = 2.38 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.59 ppm; Enantiomeric excess: 94%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 9.42 min, second peak: t_R = 15.51 min; HRMS (ESI) m/z calcd. for $C_{21}H_{17}F_3NaO_3$ $[M+Na]^+$ = 397.1022, found = 397.1025.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.51	n.a.	293.579	138.461	49.90	n.a.	BMB*
2	15.57	n.a.	271.545	139.009	50.10	n.a.	BMB*
Total:			565.124	277.470	100.00	0.000	

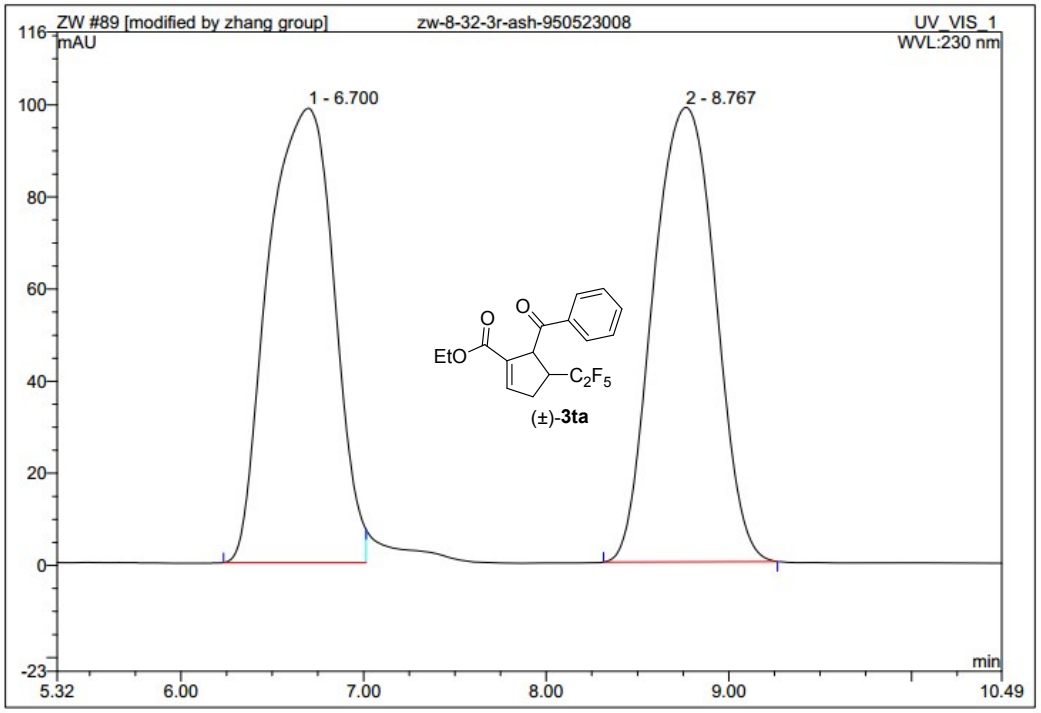


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	9.43	n.a.	160.270	73.644	97.05	n.a.	BMB*
2	15.51	n.a.	4.961	2.238	2.95	n.a.	BMB*
Total:			165.231	75.882	100.00	0.000	

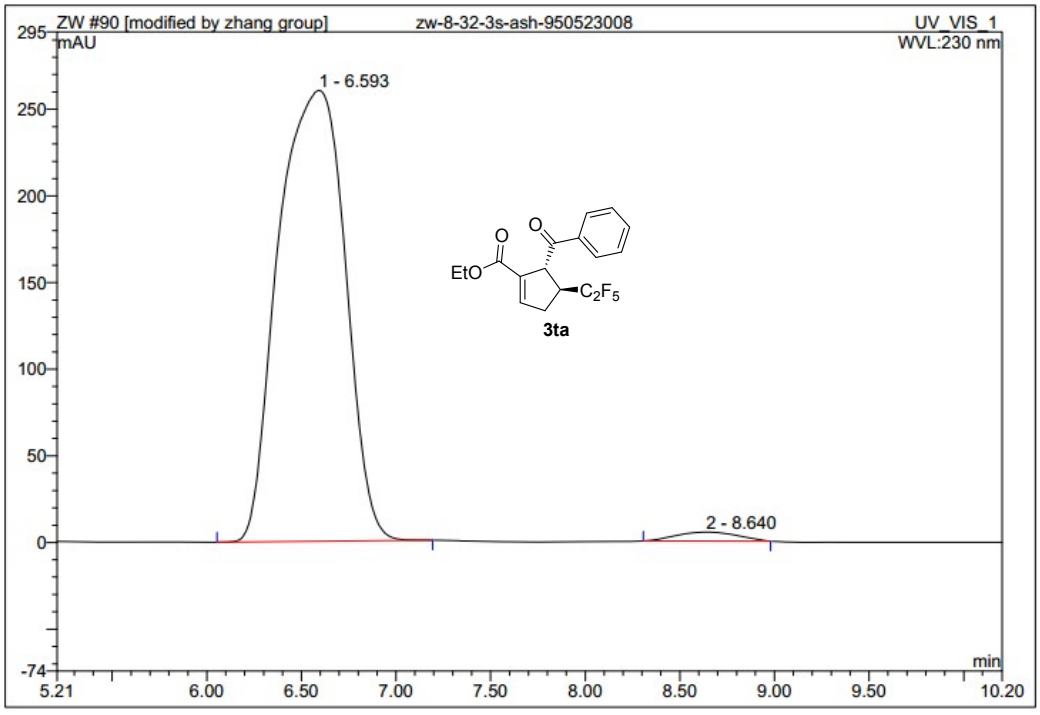


3ta

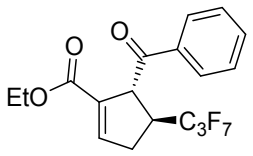
3ta; colorless oil; $[\alpha]_D^{20}$ = + 221.4 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.00 Hz, 2H), 7.63–7.60 (m, 1H), 7.52–7.49 (m, 2H), 6.96–6.95 (m, 1H), 5.24–5.22 (m, 1H), 4.02 (q, *J* = 7.00 Hz, 2H), 3.55–3.44 (m, 1H), 2.99–2.93 (m, 1H), 2.83–2.78 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 200.74, 162.90, 143.44, 136.65, 135.93, 133.45, 128.75, 128.51, 122.31 (t, *J* = 36.25 Hz), 120.04 (t, *J* = 36.13 Hz), 115.95–115.03 (m), 113.92–113.03 (m), 60.80, 49.41, 45.50 (t, *J* = 21.13 Hz), 32.71, 13.60; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -83.10, -113.19, -114.14, -123.53, -124.48 ppm; Enantiomeric excess: 96%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 6.59 min, second peak: t_R = 8.64 min; HRMS (ESI) m/z calcd. for $C_{17}H_{15}F_5NaO_3$ $[M+Na]^+$ = 385.0834, found = 385.0838.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.70	n.a.	98.663	41.224	51.40	n.a.	BM *
2	8.77	n.a.	98.681	38.977	48.60	n.a.	BMB*
Total:			197.344	80.202	100.00	0.000	

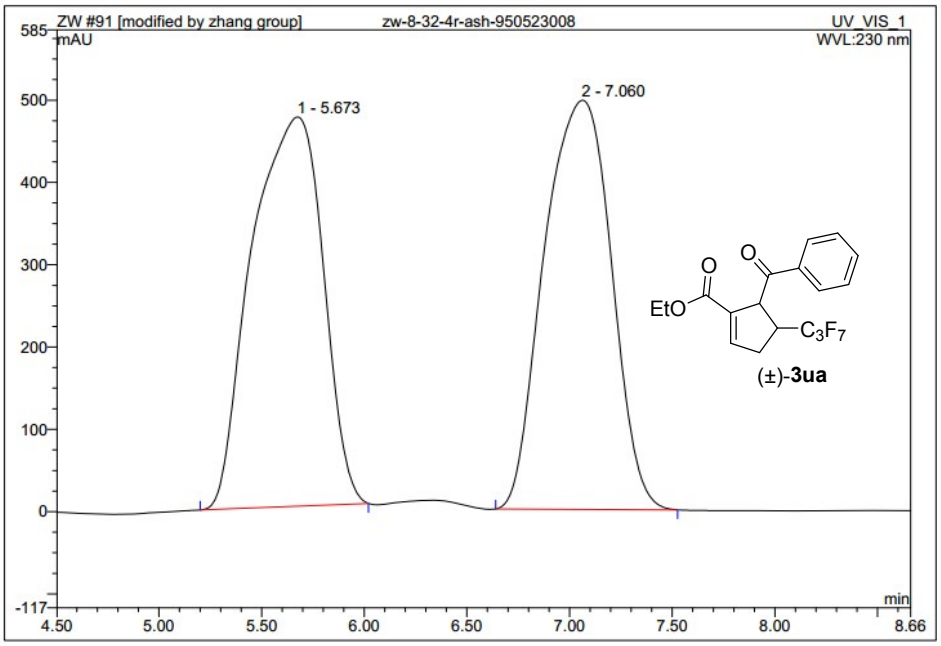


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.59	n.a.	260.159	106.719	98.23	n.a.	BMB*
2	8.64	n.a.	5.120	1.924	1.77	n.a.	BMB*
Total:			265.279	108.643	100.00	0.000	

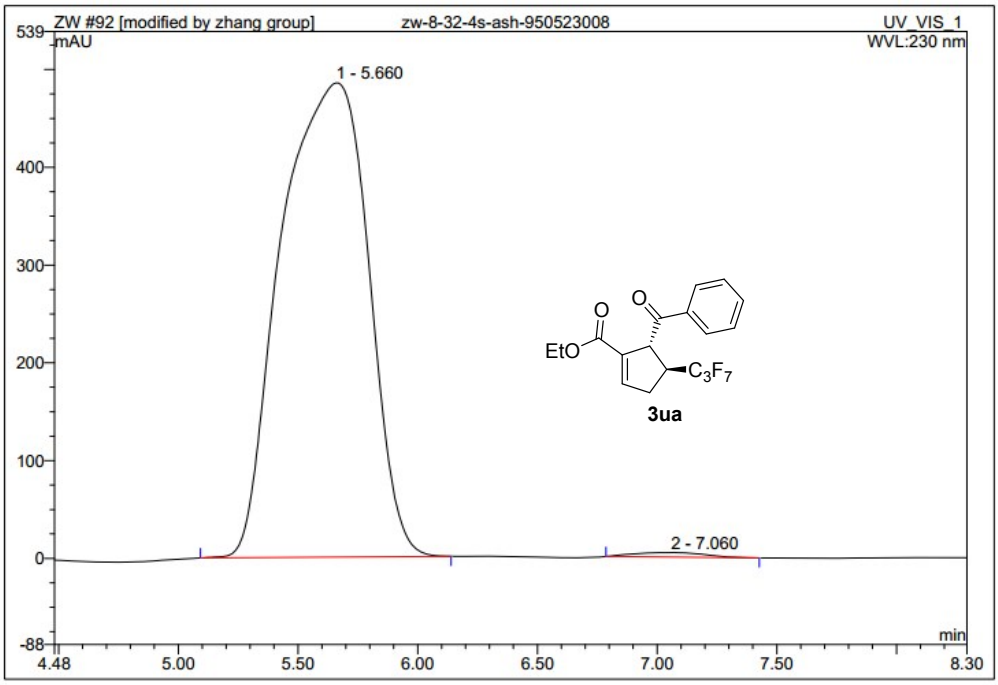


3ua

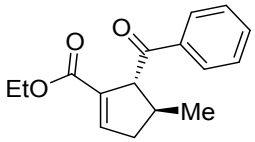
3ua; colorless oil; $[\alpha]_D^{20}$ = + 123.0 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.10–8.08 (m, 2H), 7.63–7.60 (m, 1H), 7.53–7.50 (m, 2H), 6.97–6.95 (m, 1H), 5.26–5.24 (m, 1H), 4.05–3.99 (m, 2H), 3.65–3.53 (m, 1H), 3.01–2.95 (m, 1H), 2.85–2.80 (m, 1H), 0.99 (t, *J* = 7.50 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 200.78, 162.91, 143.53, 136.67, 135.84, 133.45, 128.75, 128.53, 121.02–120.74 (m), 119.53 (t, *J* = 31.38 Hz), 118.73 (t, *J* = 33.88 Hz), 117.77–117.24 (m), 116.44 (t, *J* = 33.50 Hz), 115.48 (t, *J* = 31.25 Hz), 114.43–113.89 (m), 111.58–110.69 (m), 109.47–108.58 (m), 107.37–106.48 (m), 60.82, 49.42, 45.98 (t, *J* = 21.25 Hz), 32.74 (t, *J* = 3.50 Hz), 13.58; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -83.10–-80.75 (m), -109.77–-109.92 (m), -110.75–-110.90 (m), -120.24–-120.31 (m), -121.18–-121.32 (m), -124.32–-126.50 (m); Enantiomeric excess: 98%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 5.66 min, second peak: t_R = 7.06 min; HRMS (ESI) m/z calcd. for $C_{18}H_{15}F_7NaO_3$ $[M+Na]^+$ = 435.0802, found = 435.0803.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.67	n.a.	473.105	190.930	50.24	n.a.	BMB*
2	7.06	n.a.	497.240	189.070	49.76	n.a.	BMB*
Total:			970.345	379.999	100.00	0.000	



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.66	n.a.	484.988	204.856	99.24	n.a.	BMB*
2	7.06	n.a.	4.566	1.562	0.76	n.a.	BMB*
Total:			489.554	206.418	100.00	0.000	



3va

3va[3]; colorless oil; $[\alpha]_D^{20}$ = + 16.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.04–8.02 (m, 2H), 7.62–7.58 (m, 1H), 7.52–7.48 (m, 2H), 7.01–7.00 (s, 1H), 4.43–4.42 (m, 1H), 4.16–4.09 (m, 2H), 2.93–2.85 (m, 1H), 2.60–2.51 (m, 1H), 2.24–2.17 (m, 1H), 1.24 (d, *J* = 7.2 Hz, 3H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 201.13, 164.31, 145.25, 136.99, 135.36, 132.98, 128.55, 60.35, 59.09, 40.96, 38.45, 21.59, 14.01; Enantiomeric excess: 22% (13/1 ratio of regioisomers), determined by HPLC (Chiralpak AD-H, hexane/*i*-PrOH = 95/05; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 10.60 min, second peak: t_R = 11.78 min.

SHIMADZU LabSolutions 分析报告

<样品信息>

样品名	: zw-Me-adh-950510rac3		
样品ID	:		
数据文件名	: zw-Me-adh-950510rac3.lcd		
方法文件名	: AS-H-40%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/3 20:25:07	分析者	: System Administrator
处理日期	: 2020/6/3 20:43:07	处理者	: System Administrator

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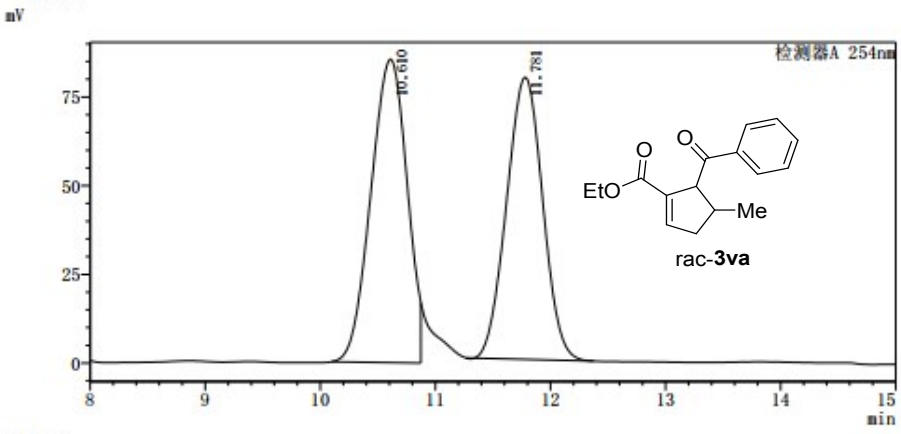

<峰表>

检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	10.610	1860496	85482	51.733		M	
2	11.781	1735868	79509	48.267		M	
总计		3596364	164991				

SHIMADZU LabSolutions 分析报告

<样品信息>

样品名	: zw-Me-adh-950510rac2		
样品ID	:		
数据文件名	: zw-Me-adh-950510rac2.lcd		
方法文件名	: AS-H-40%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/3 20:10:32	分析者	: System Administrator
处理日期	: 2020/6/3 20:24:39	处理者	: System Administrator

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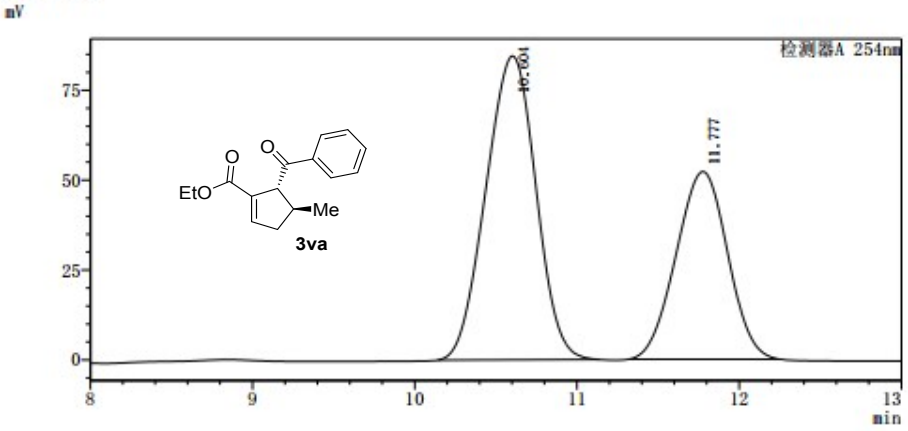
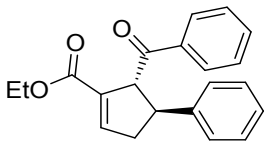


<峰表>

检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	10.604	1814125	84496	61.134		M	
2	11.777	1153344	52186	38.866		M	
总计		2967470	136682				



3wa

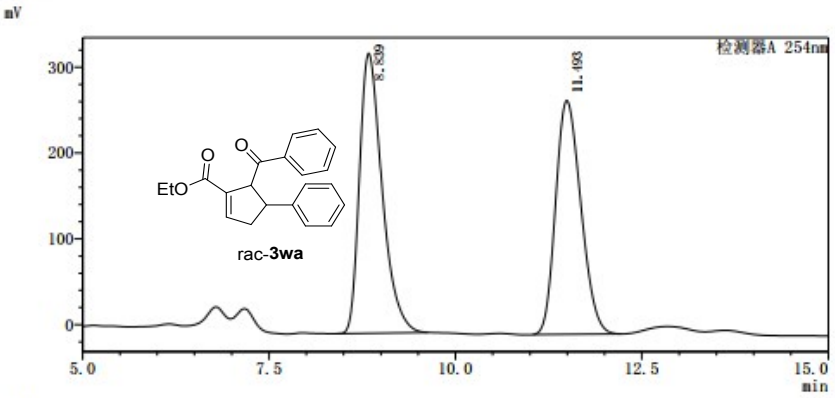
3wa[3]; colorless oil; $[\alpha]_D^{20}$ = + 14.5 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.80 (d, *J* = 8.0 Hz, 2H), 7.55–7.51 (m, 1H), 7.40–7.28 (m, 5H), 7.23–7.21 (m, 2H), 7.13–7.12 (m, 1H), 4.90–4.89 (m, 1H), 4.19–4.09 (m, 2H), 3.62–3.57 (m, 1H), 3.26–3.18 (m, 1H), 2.77–2.71 (m, 1H), 1.18 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 200.74, 163.99, 145.05, 144.93, 136.50, 135.64, 133.03, 128.89, 128.75, 128.40, 126.98, 126.80, 60.50, 60.24, 48.88, 42.11, 14.00; Enantiomeric excess: 11% (5/1 ratio of regioisomers), determined by HPLC (Chiralpak OD-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 8.54 min, second peak: t_R = 11.28 min.

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: zw-cha-rac1--odh901010		
样品ID	:		
数据文件名	: zw-cha-rac1--odh901010.lcd		
方法文件名	: OD-H-20%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/2 10:50:47	分析者	: System Administrator
处理日期	: 2020/6/2 11:07:08	处理者	: System Administrator

〈色谱图〉



〈峰表〉

检测器A 254nm

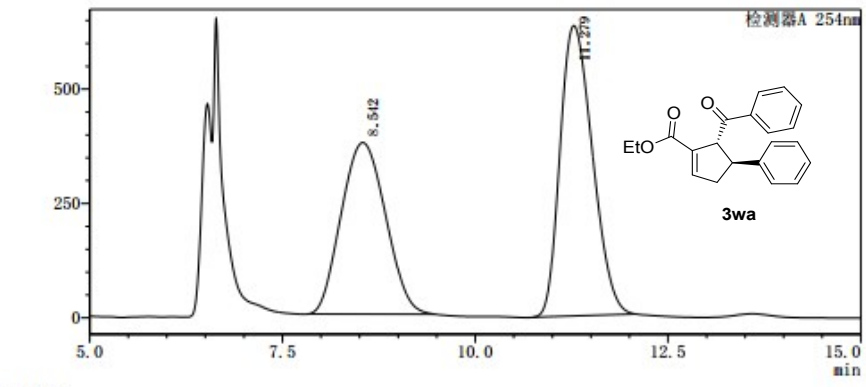
峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	8.839	6862895	325972	51.627		M	
2	11.493	6430425	272262	48.373		M	
总计		13293321	598234				

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: zw-cha-chiral1--odh901010		
样品ID	:		
数据文件名	: zw-cha-chiral--odh901010.lcd		
方法文件名	: OD-H-20%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/2 11:08:03	分析者	: System Administrator
处理日期	: 2020/6/2 11:23:09	处理者	: System Administrator

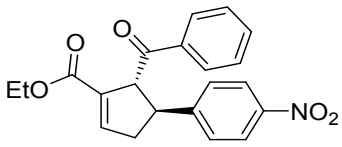
〈色谱图〉



〈峰表〉

检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	8.542	15038535	375135	44.750		M	
2	11.279	18567349	634615	55.250		M	
总计		33605884	1009750				



3xa

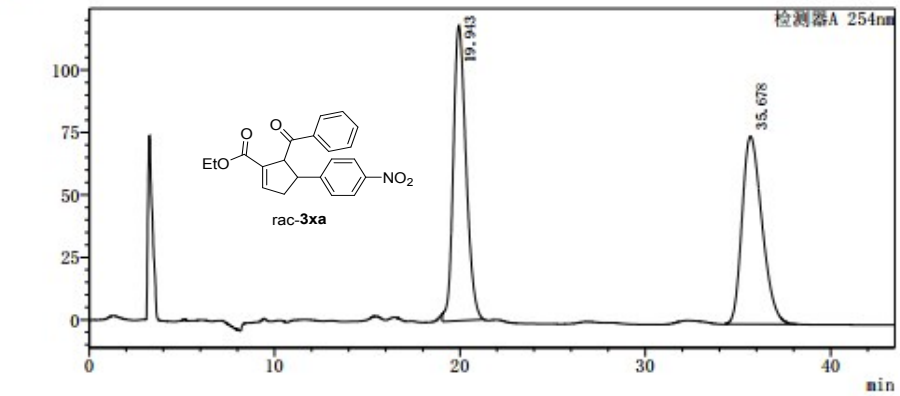
3xa[3]; yellow oil; $[\alpha]_D^{20}$ = + 18.4 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.19 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.59–7.55 (m, 1H), 7.43–7.37 (m, 4H), 7.13–7.12 (m, 1H), 4.90–4.89 (m, 1H), 4.21–4.09 (m, 2H), 3.75–3.70 (m, 1H), 3.32–3.24 (m, 1H), 2.76–2.70 (m, 1H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.92, 163.63, 152.25, 147.01, 144.48, 136.25, 135.75, 133.46, 128.65, 128.62, 127.72, 124.27, 60.75, 59.57, 48.46, 41.79; Enantiomeric excess: 6% (20/1 ratio of regioisomers), determined by HPLC (Chiralpak OD-H, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 19.81 min, second peak: t_R = 35.40 min.

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: zw-xioji-rac-odh901010		
样品ID	:		
数据文件名	: zw-xioji-rac-odh901010.lcd		
方法文件名	: OD-H-20%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
分析日期	: 2020/6/2 11:24:12	分析者	: System Administrator
处理日期	: 2020/6/2 12:07:40	处理者	: System Administrator

〈色谱图〉



〈峰表〉

检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	19.943	5637949	118223	49.979		M	
2	35.678	5642639	75220	50.021		M	
总计		11280587	193443				

SHIMADZU LabSolutions 分析报告

〈样品信息〉

样品名	: zw-xioji-chiral-odh901010		
样品ID	:		
数据文件名	: zw-xioji-chiral-odh901010.lcd		
方法文件名	: OD-H-20%.lcm		
批处理文件名	:		
样品瓶号	: 1-1	样品类型	: 未知
进样体积	: 1 uL		
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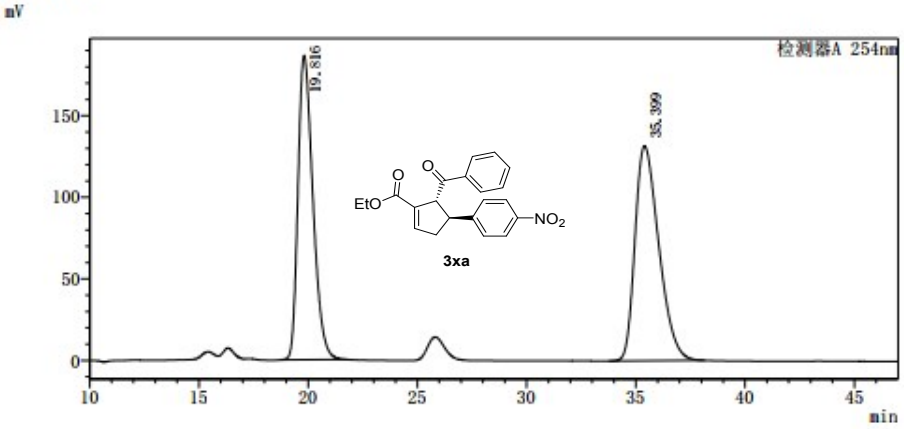
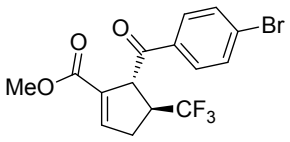
〈色谱图〉



〈峰表〉

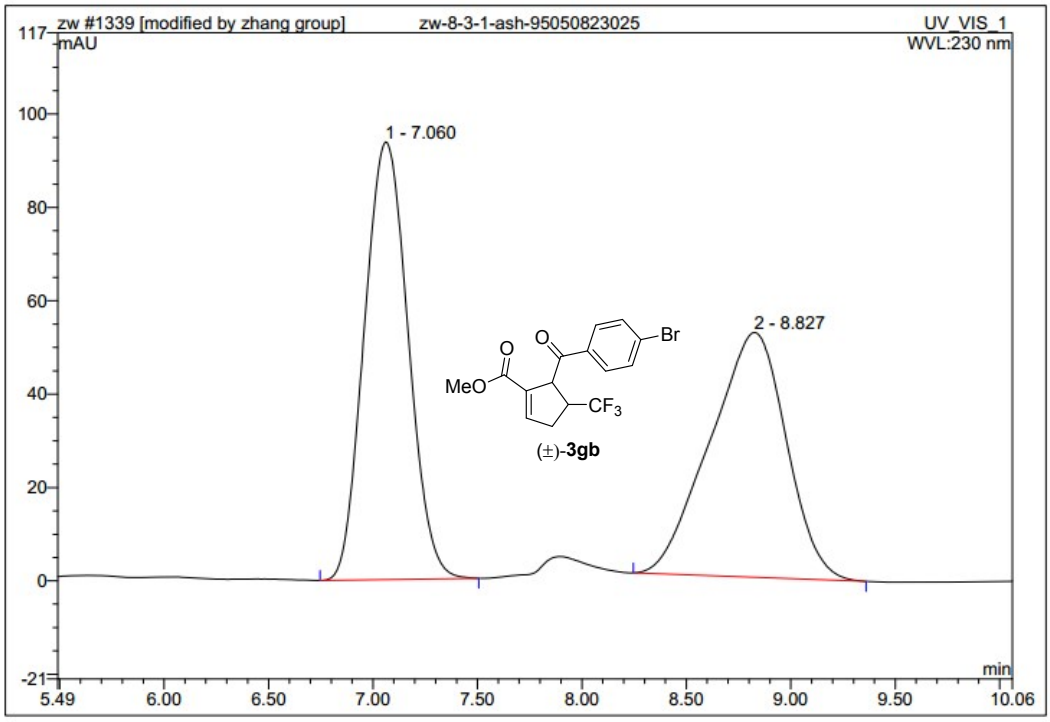
检测器A 254nm

峰号	保留时间	面积	高度	浓度	浓度单位	标记	化合物名
1	19.816	8865749	186491	47.009		M	
2	35.399	9994044	131622	52.991		M	
总计		18859793	318113				

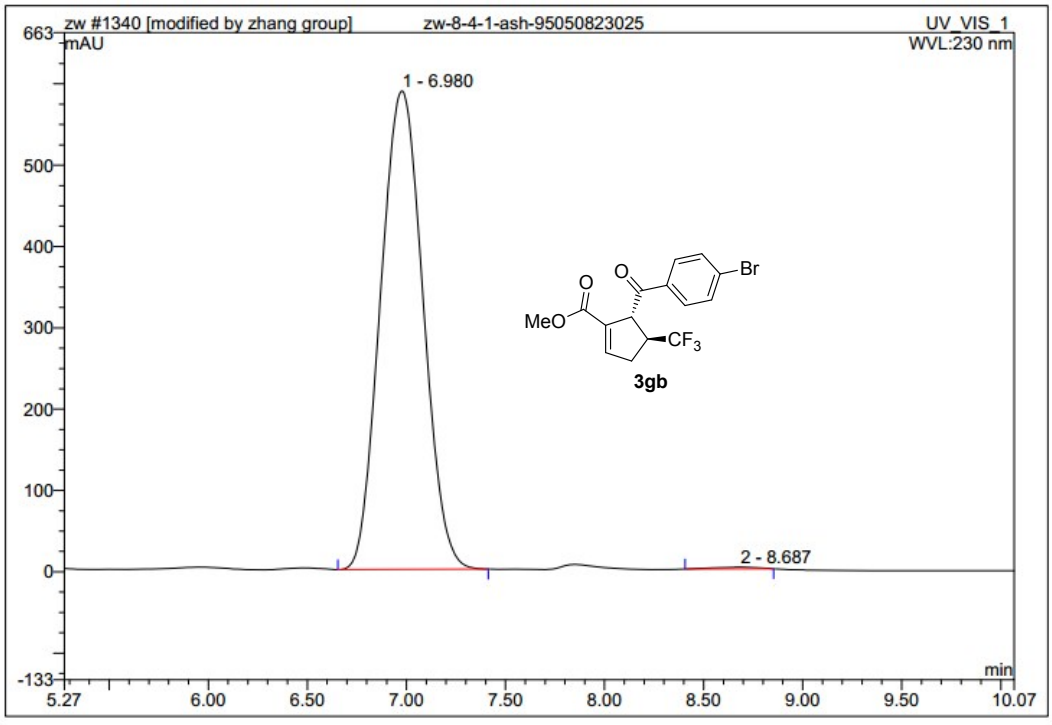


3gb

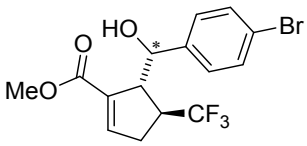
3gb; colorless oil; $[\alpha]_D^{20}$ = + 136.2 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.80 Hz, 2H), 7.65 (d, *J* = 8.40 Hz, 2H), 6.94 (s, 1H), 4.97–4.96 (m, 1H), 3.62 (s, 1H), 3.30–3.18 (m, 1H), 3.04–2.96 (m, 1H), 2.81–2.75 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.08, 163.46, 143.75, 135.34, 135.11, 132.04, 130.25, 129.01, 127.06 (q, *J* = 275.90 Hz), 51.81, 50.69, 46.70 (q, *J* = 28.20 Hz), 33.43; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.65 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 6.98 min, second peak: t_R = 8.69 min; HRMS (ESI) m/z calcd. for $C_{15}H_{12}BrF_3NaO_3$ $[M+Na]^+$ = 398.9814, found = 398.9815.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.06	n.a.	93.744	22.854	50.78	n.a.	BMB*
2	8.83	n.a.	52.465	22.156	49.22	n.a.	BMB*
Total:			146.209	45.010	100.00	0.000	

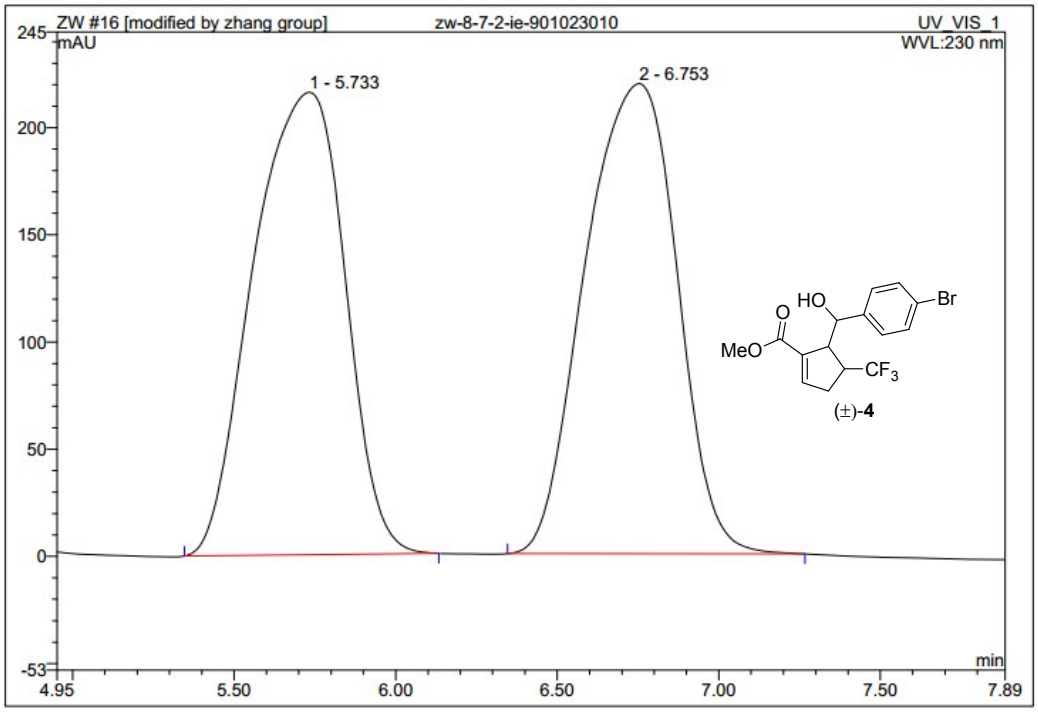


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	6.98	n.a.	588.359	146.351	99.64	n.a.	BMB*
2	8.69	n.a.	2.044	0.536	0.36	n.a.	BMB*
Total:			590.402	146.886	100.00	0.000	

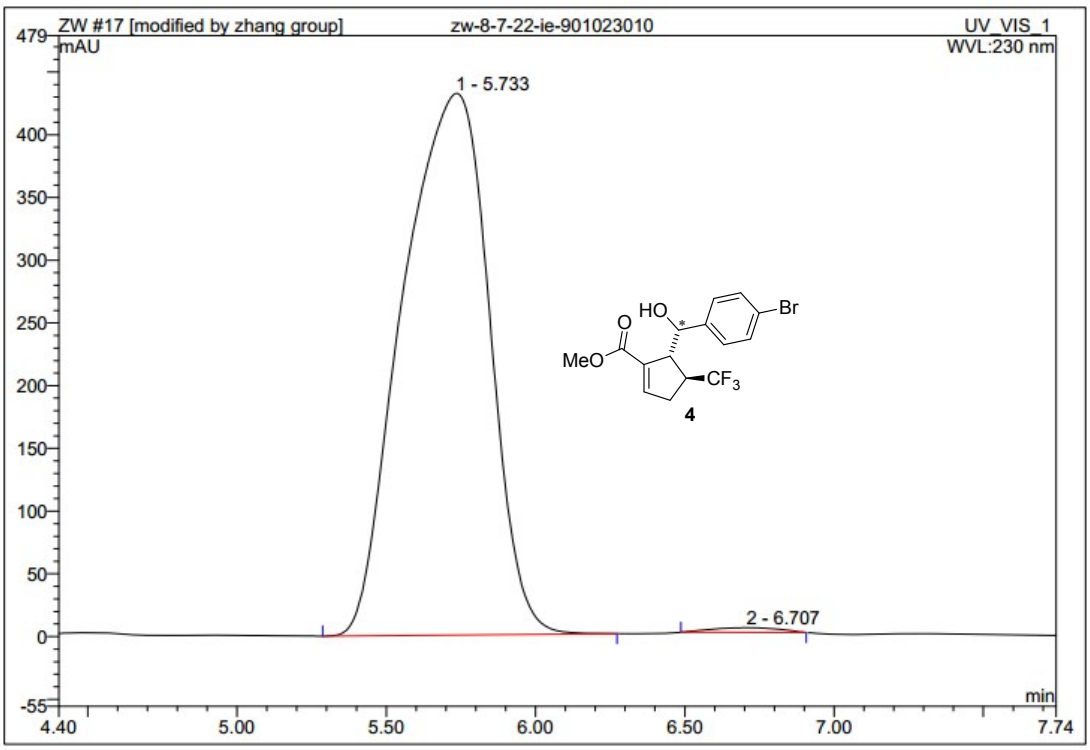


4

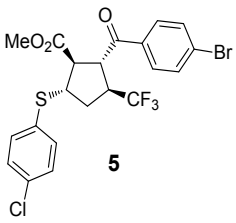
4; colorless oil; $[\alpha]_D^{20}$ = + 51.3 (*c* = 0.33, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.40 Hz, 2H), 7.10 (d, *J* = 8.40 Hz, 2H), 6.66 (s, 1H), 5.04–5.02 (m, 1H), 3.75 (s, 3H), 3.58–3.57 (m, 1H), 3.32–3.30 (m, 1H), 2.96–2.86 (m, 1H), 2.46–2.39 (m, 1H), 2.15–2.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.36, 144.56, 139.48, 135.03, 131.13, 128.11, 127.65 (q, *J* = 276.60 Hz), 121.70, 73.32, 52.85, 51.95 (q, *J* = 1.80 Hz), 42.14 (q, *J* = 27.20 Hz), 32.54; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -73.70 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak IE, hexane/*i*-PrOH = 90/10; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 5.73 min, second peak: t_R = 6.71 min; HRMS (ESI) m/z calcd. for $C_{15}H_{14}BrF_3NaO_3$ $[M+Na]^+$ = 400.9971, found = 400.9975.



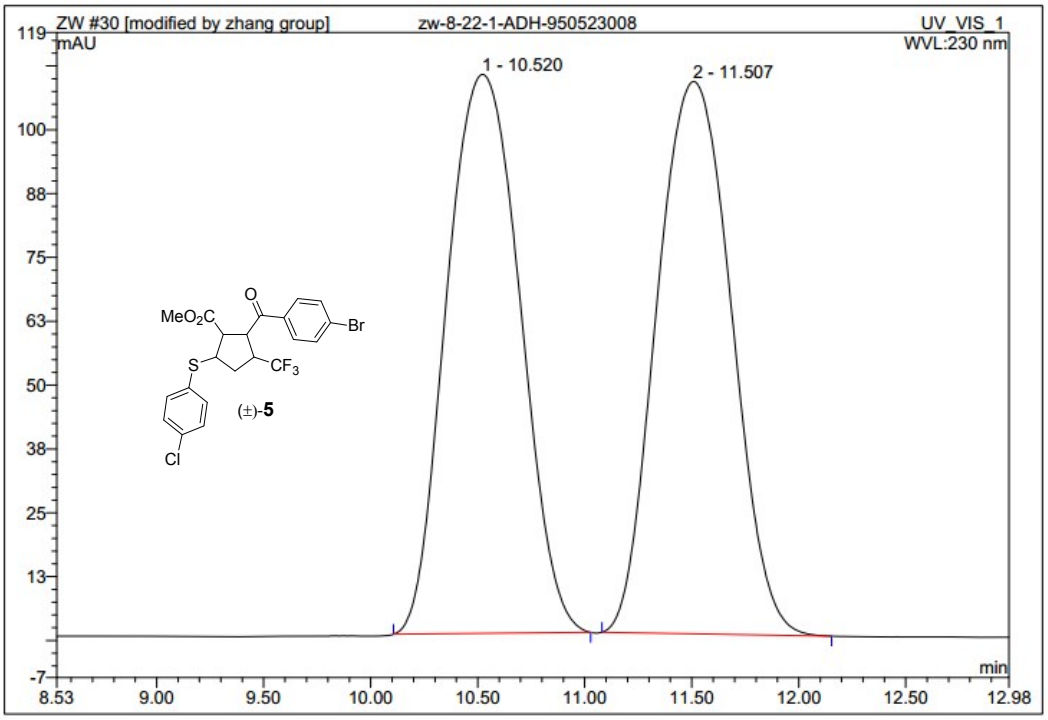
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.73	n.a.	215.748	71.191	49.50	n.a.	BMB*
2	6.75	n.a.	219.296	72.619	50.50	n.a.	BMB*
Total:			435.043	143.810	100.00	0.000	



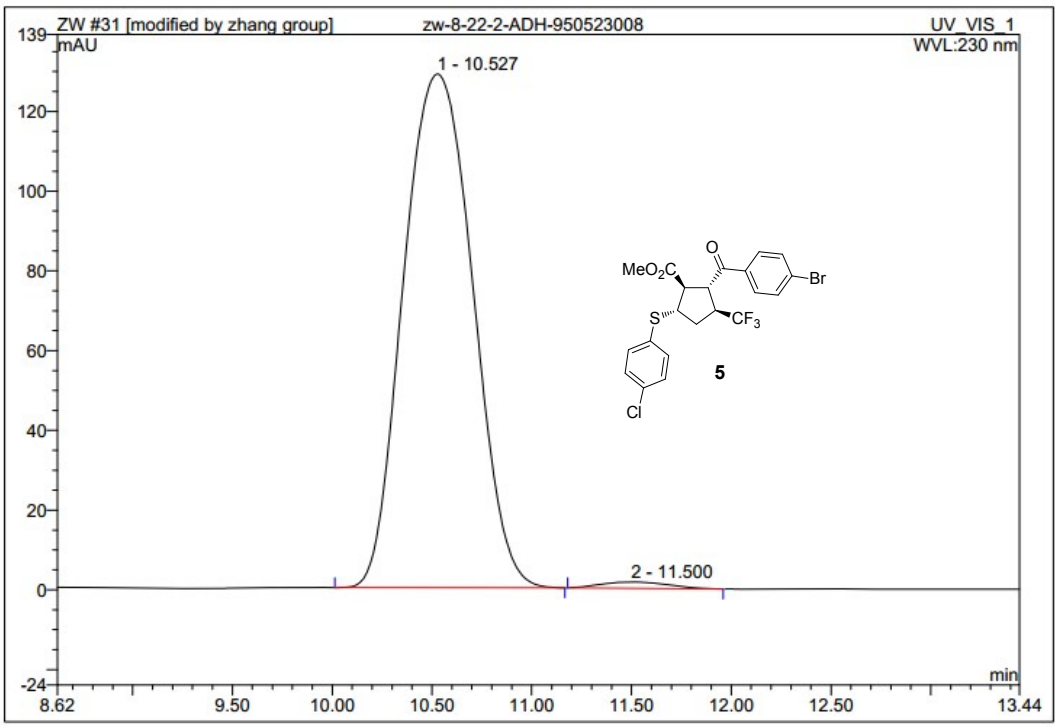
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	5.73	n.a.	431.687	145.003	99.32	n.a.	BMB*
2	6.71	n.a.	3.691	0.994	0.68	n.a.	BMB*
Total:			435.379	145.997	100.00	0.000	



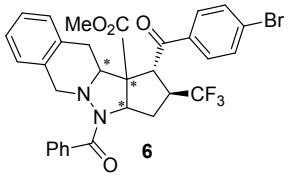
5; white solid; $[\alpha]_D^{20}$ = -17.9 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.50 Hz, 2H), 7.64 (d, *J* = 8.50 Hz, 2H), 7.38 (d, *J* = 8.50 Hz, 2H), 7.31–7.29 (m, 2H), 4.29–4.26 (m, 1H), 3.95–3.91 (m, 1H), 3.56–3.50 (m, 4H), 3.01–2.97 (m, 1H), 2.45–2.39 (m, 1H), 2.17–2.11 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 197.03, 171.97, 134.56, 134.48 (q, *J* = 1.75 Hz), 132.17, 130.94, 130.05, 129.42, 129.33, 126.96 (q, *J* = 276.25 Hz), 55.68, 52.47, 49.74, 49.02, 44.72 (q, *J* = 28.25 Hz), 32.78; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -70.47 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak AD-H, hexane/*i*-PrOH = 95/05; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 10.53 min, second peak: t_R = 11.50 min; HRMS (ESI) m/z calcd. for $C_{21}H_{17}BrClF_3NaO_3S$ $[M+Na]^+$ = 542.9615, found = 542.9613.



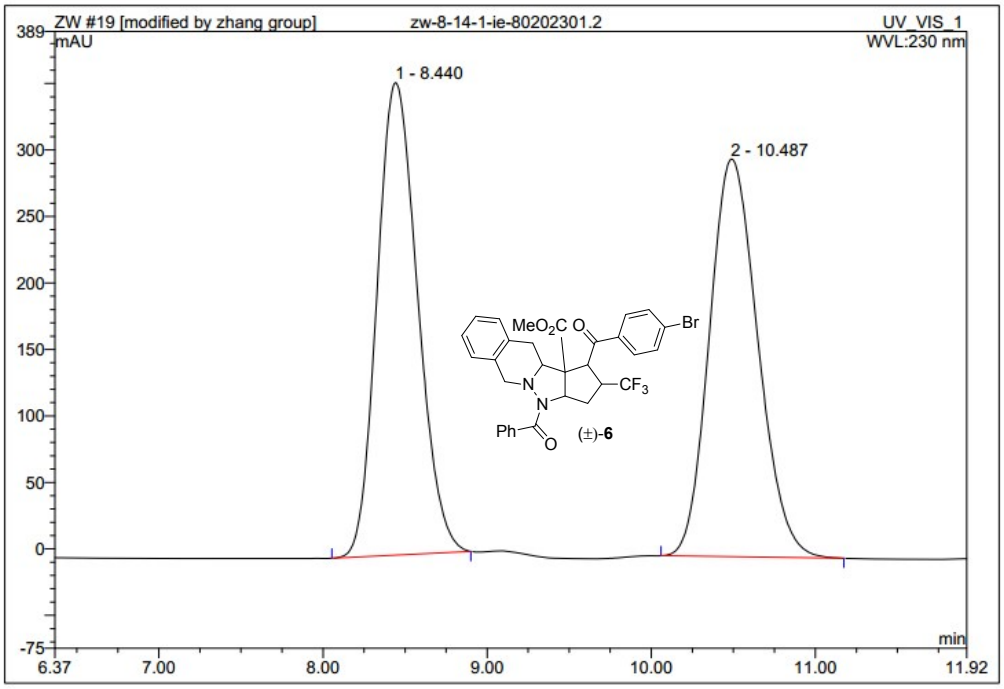
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.52	n.a.	109.462	43.572	49.90	n.a.	BMB*
2	11.51	n.a.	108.151	43.746	50.10	n.a.	BMB*
Total:			217.613	87.318	100.00	0.000	



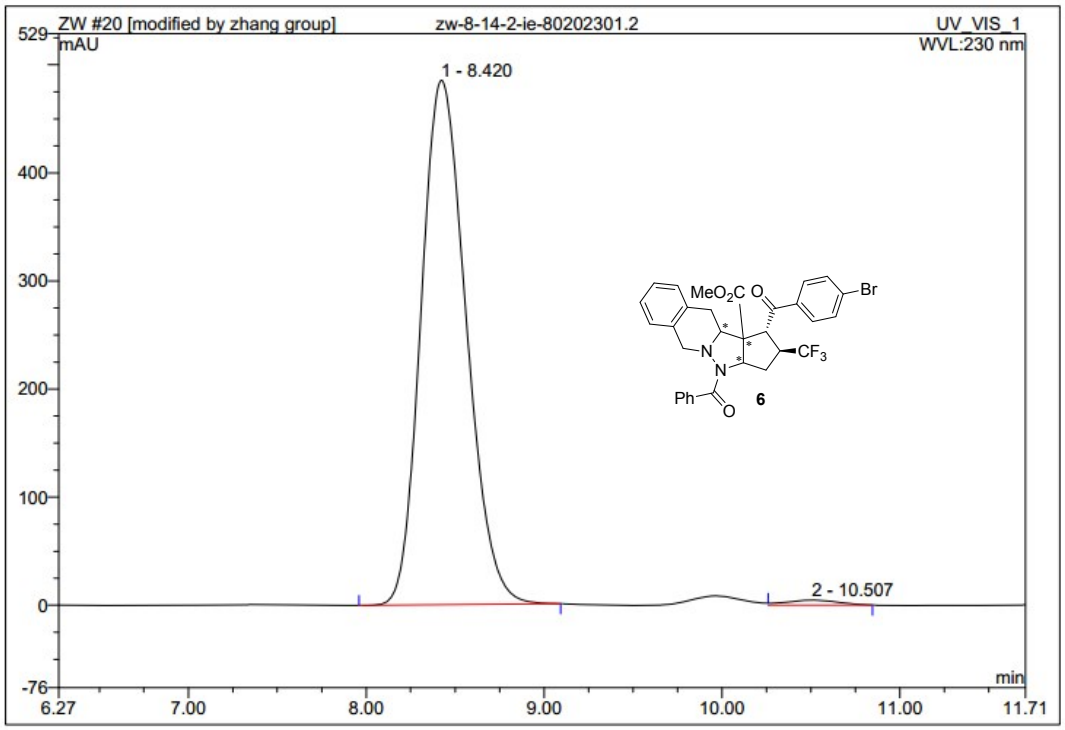
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.53	n.a.	128.849	51.848	98.85	n.a.	BMB*
2	11.50	n.a.	1.552	0.605	1.15	n.a.	BMB*
Total:			130.400	52.453	100.00	0.000	



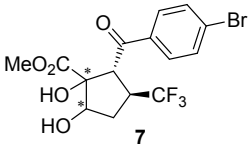
6; white solid; $[\alpha]_D^{20}$ = + 33.9 (*c* = 0.33, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.50 Hz, 2H), 8.05–8.03 (m, 2H), 7.76 (d, *J* = 8.50 Hz, 2H), 7.52–7.49 (m, 1H), 7.44–7.41 (m, 2H), 7.05–6.97 (m, 2H), 6.82–6.79 (m, 1H), 6.45 (d, *J* = 8.00 Hz, 1H), 5.80 (s, 1H), 5.29 (d, *J* = 6.00 Hz, 1H), 4.73 (d, *J* = 12.00 Hz, 1H), 3.65–3.55 (m, 2H), 3.28–3.24 (m, 1H), 3.02 (s, 3H), 2.76–2.69 (m, 2H), 2.58–2.53 (m, 1H), 2.17–2.10 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.72, 172.75, 169.40, 136.16, 134.42, 133.72, 132.23, 131.52, 130.78, 130.71, 129.68, 129.24, 128.16, 127.71, 127.14, 126.84, 126.32 (q, *J* = 276.63 Hz), 125.32, 72.73, 67.21, 63.06, 51.95, 49.17, 47.34 (q, *J* = 27.88 Hz), 46.92, 28.96; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -69.11 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak IE, hexane/*i*-PrOH = 80/20; flow rate 1.2 ml/min; 25 °C; 230 nm), first peak: t_R = 8.42 min, second peak: t_R = 10.50 min; HRMS (ESI) m/z calcd. for $C_{31}H_{26}BrF_3N_2NaO_4$ $[M+Na]^+$ = 649.0920, found = 649.0920.



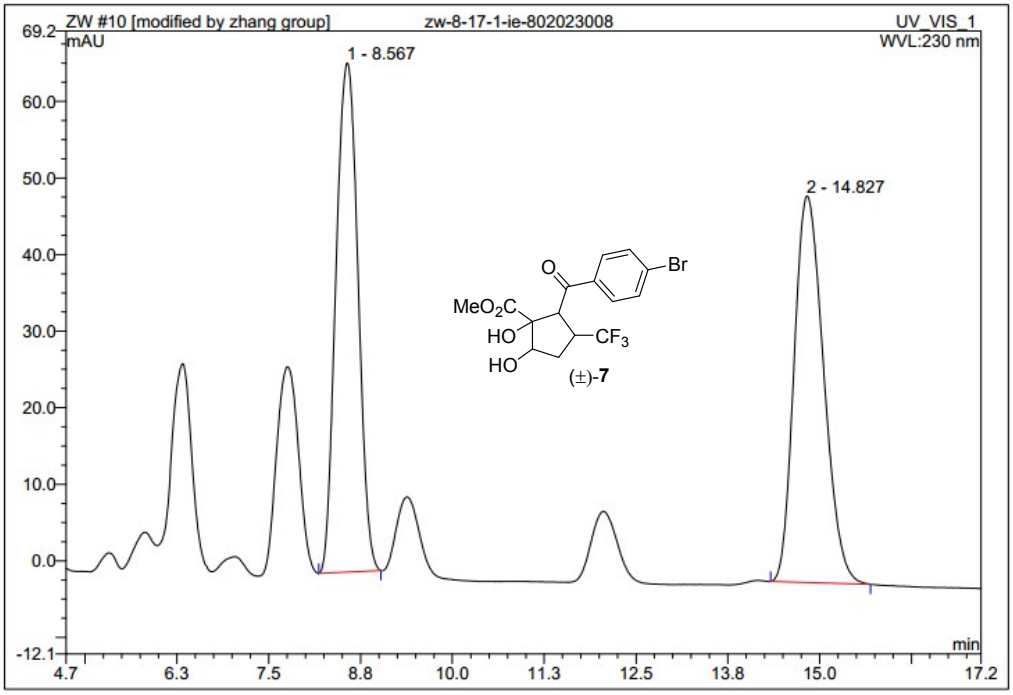
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.44	n.a.	355.434	101.596	49.83	n.a.	BMB*
2	10.49	n.a.	299.135	102.291	50.17	n.a.	BMB*
Total:			654.569	203.887	100.00	0.000	



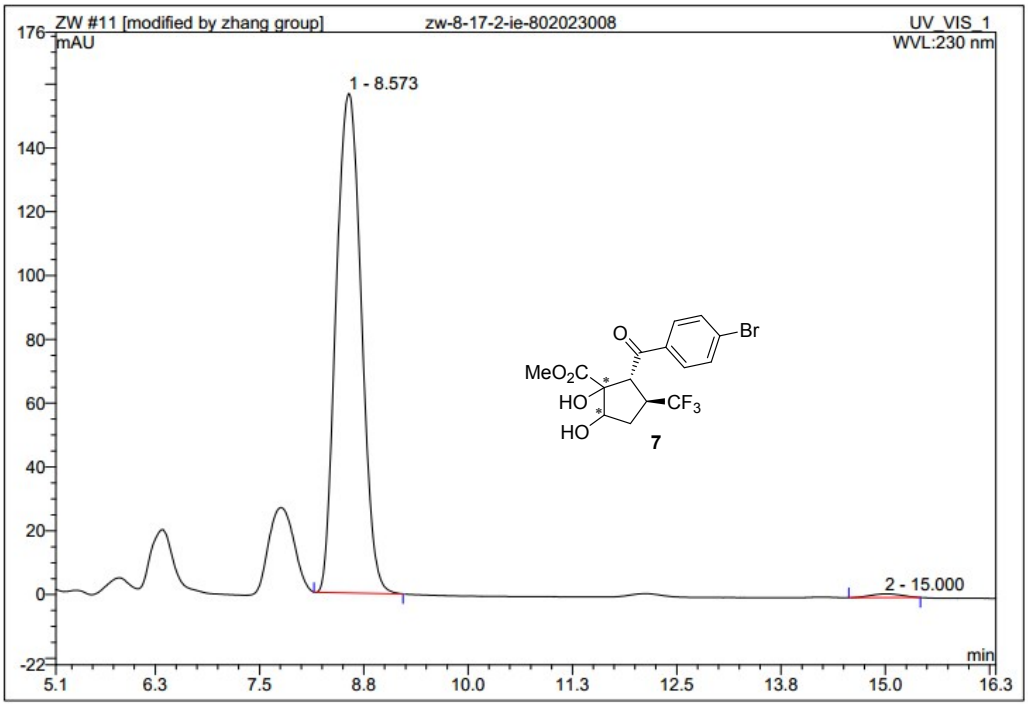
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.42	n.a.	485.124	142.567	98.92	n.a.	BMB*
2	10.51	n.a.	4.633	1.556	1.08	n.a.	MB*
Total:			489.757	144.123	100.00	0.000	



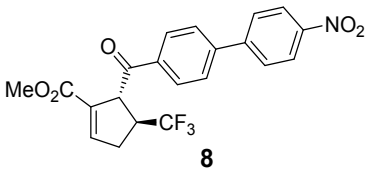
7; colorless oil; $[\alpha]_D^{20} = -57.1$ ($c = 0.33$, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, $J = 8.40$ Hz, 2H), 7.66–7.59 (m, 2H), 4.22 (br, 1H), 4.16 (d, $J = 8.80$ Hz, 1H), 3.64–3.57 (m, 2H), 3.49 (s, 3H), 2.40–2.34 (m, 1H), 2.11–2.02 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.49, 172.60, 135.61, 132.15, 130.10, 129.42, 126.70 (q, $J = 275.80$ Hz), 83.65, 75.41, 54.63, 53.34, 39.91 (q, $J = 28.80$ Hz), 31.35; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -70.92 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak IE, hexane/*i*-PrOH = 80/20; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: $t_R = 8.57$ min, second peak: $t_R = 15.00$ min; HRMS (ESI) m/z calcd. for $C_{15}H_{14}BrF_3NaO_5$ $[M+Na]^+$ = 432.9869, found = 432.9873.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.57	n.a.	66.494	23.058	49.57	n.a.	BMB*
2	14.83	n.a.	50.451	23.457	50.43	n.a.	BMB*
Total:			116.945	46.515	100.00	0.000	

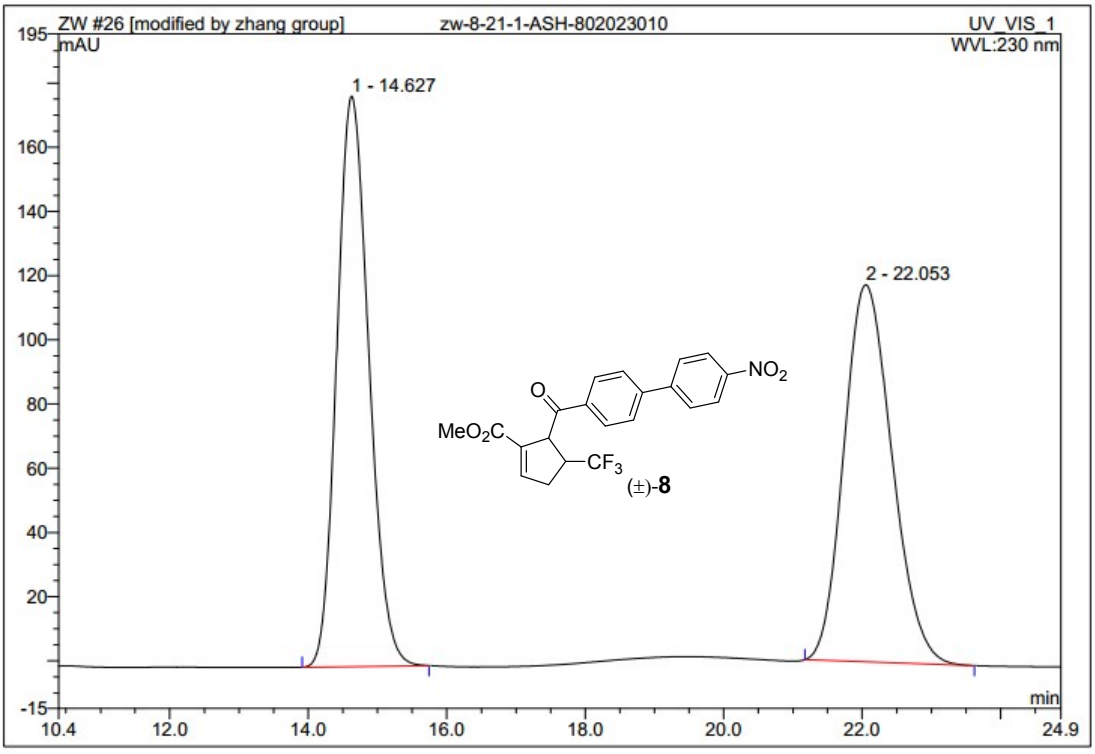


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.57	n.a.	156.589	54.708	99.08	n.a.	BMB*
2	15.00	n.a.	1.184	0.506	0.92	n.a.	BMB*
Total:			157.773	55.215	100.00	0.000	

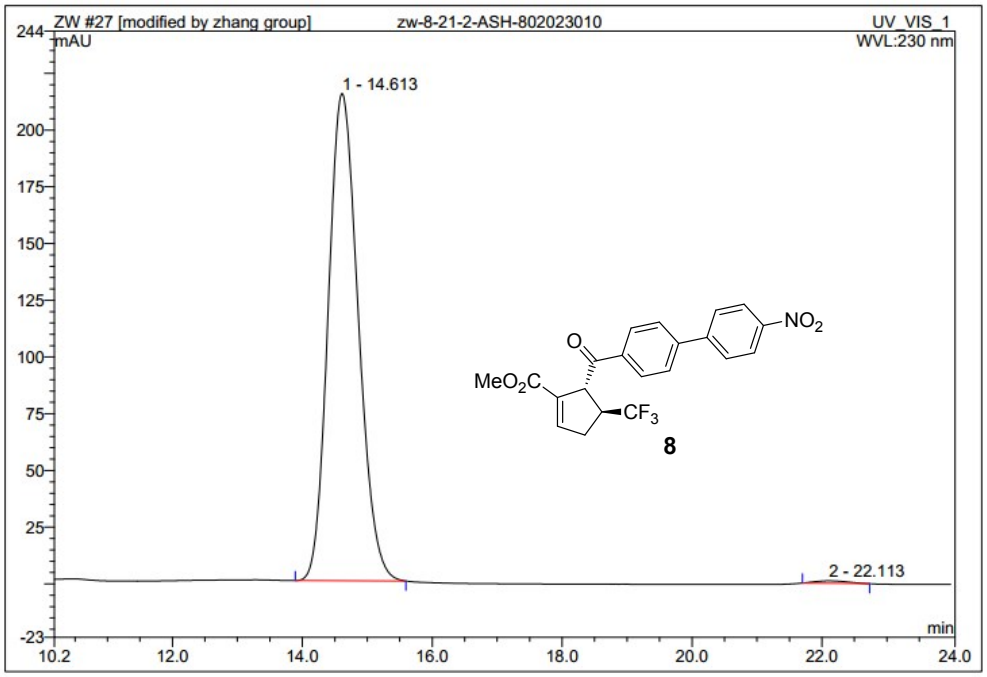


8

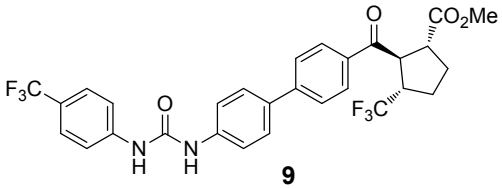
8; yellow solid; $[\alpha]_D^{20} = -16.6$ ($c = 0.33$, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.37–8.34 (m, 2H), 8.20 (d, $J = 8.50$ Hz, 2H), 7.83–7.77 (m, 4H), 6.99 (s, 1H), 5.10–5.08 (m, 1H), 3.66 (s, 3H), 3.35–3.25 (m, 1H), 3.08–3.01 (m, 1H), 2.86–2.80 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.47, 163.57, 147.65, 146.07, 143.79, 143.55, 136.30, 135.41, 129.57, 128.10, 127.72, 127.09 (q, $J = 276.00$ Hz), 124.21, 51.87, 50.76 (q, $J = 1.38$ Hz), 46.71 (q, $J = 28.00$ Hz), 33.46 (d, $J = 2.13$ Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -71.55 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak AS-H, hexane/*i*-PrOH = 80/20; flow rate 1.0 ml/min; 25 °C; 230 nm), first peak: t_R = 14.61 min, second peak: t_R = 22.11 min; HRMS (ESI) m/z calcd. for $C_{21}H_{16}F_3NNaO_5$ $[M+Na]^+$ = 442.0813, found = 442.0873.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	14.63	n.a.	177.682	95.153	50.19	n.a.	BMB*
2	22.05	n.a.	117.463	94.436	49.81	n.a.	BMB*
Total:			295.144	189.589	100.00	0.000	

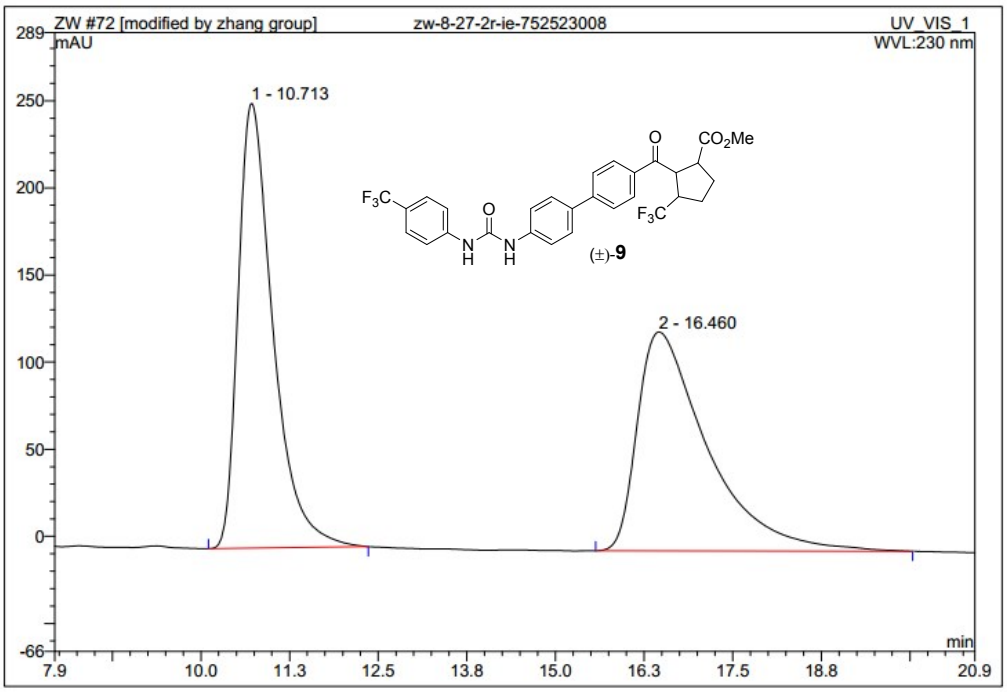


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	14.61	n.a.	214.758	115.001	99.45	n.a.	BMB*
2	22.11	n.a.	1.102	0.640	0.55	n.a.	BMB*
Total:			215.860	115.642	100.00	0.000	

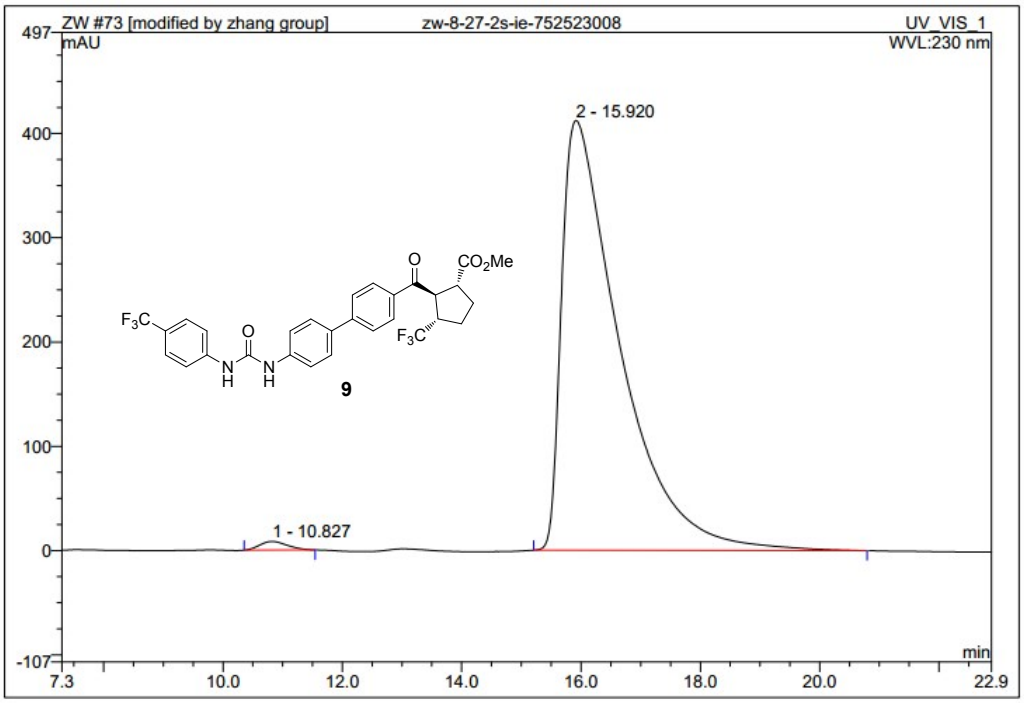


9

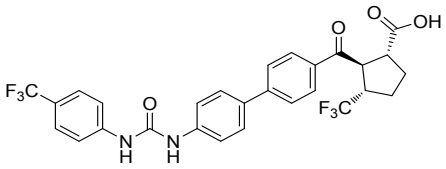
9; white solid; $[\alpha]_D^{20} = -285.3$ ($c = 0.33$, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ 8.19 (s, 1H), 8.07 (s, 1H), 8.02 (d, $J = 8.50$ Hz, 2H), 7.45 (d, $J = 8.50$ Hz, 2H), 7.38 (d, $J = 8.50$ Hz, 2H), 7.33 (d, $J = 8.50$ Hz, 2H), 7.25–7.23 (m, 4H), 4.43–4.40 (m, 1H), 3.64 (s, 3H), 3.38–3.28 (m, 1H), 3.19–3.14 (m, 1H), 2.19–2.09 (m, 3H), 2.05–1.97 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.49, 173.80, 154.00, 145.04, 141.05, 137.71, 135.31, 134.38, 129.35, 127.77, 127.40 (q, $J = 276.00$ Hz), 126.69, 126.30, 126.07 (q, $J = 3.50$ Hz), 125.76, 125.50, 125.23, 123.87 (q, $J = 269.88$ Hz), 121.34, 119.83, 52.20, 49.69, 48.48, 46.50 (q, $J = 27.63$ Hz), 29.35, 25.87; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -62.10, -70.14 ppm; Enantiomeric excess: 98%, determined by HPLC (Chiralpak IE, hexane/*i*-PrOH = 75/25; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 10.82 min, second peak: t_R = 15.92 min; HRMS (ESI) m/z calcd. for $C_{29}H_{24}F_6N_2NaO_4$ $[M+Na]^+ = 601.1532$, found = 601.1531.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.71	n.a.	255.339	145.322	50.28	n.a.	BMB*
2	16.46	n.a.	125.722	143.708	49.72	n.a.	BMB*
Total:			381.061	289.029	100.00	0.000	

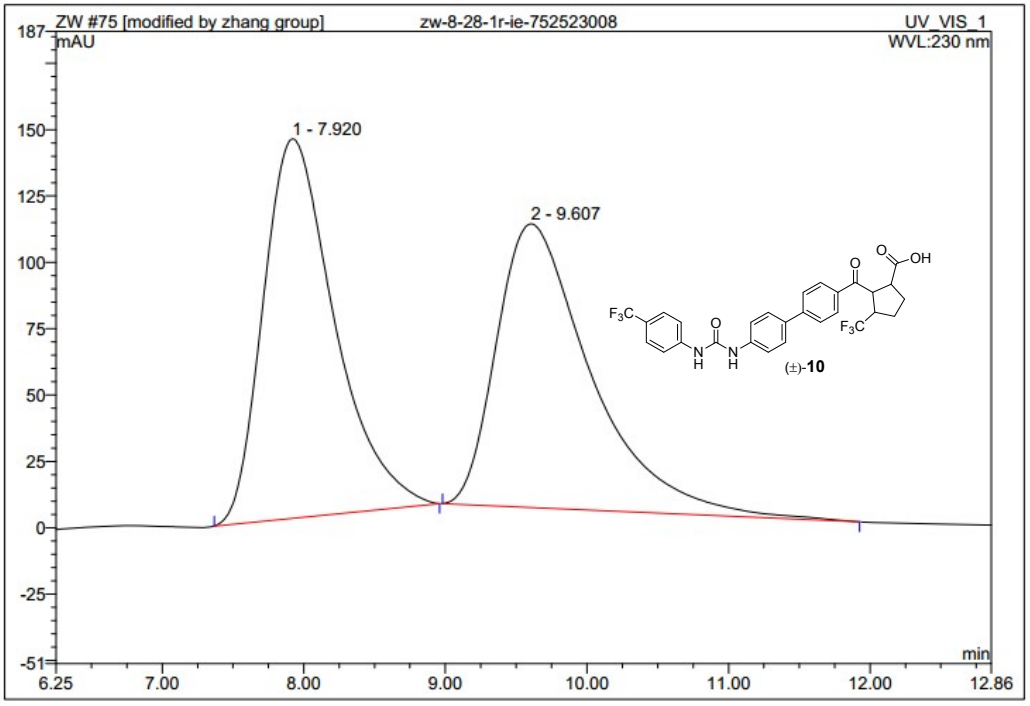


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.83	n.a.	8.301	4.446	0.95	n.a.	BMB*
2	15.92	n.a.	412.210	464.225	99.05	n.a.	BMB*
Total:			420.511	468.671	100.00	0.000	

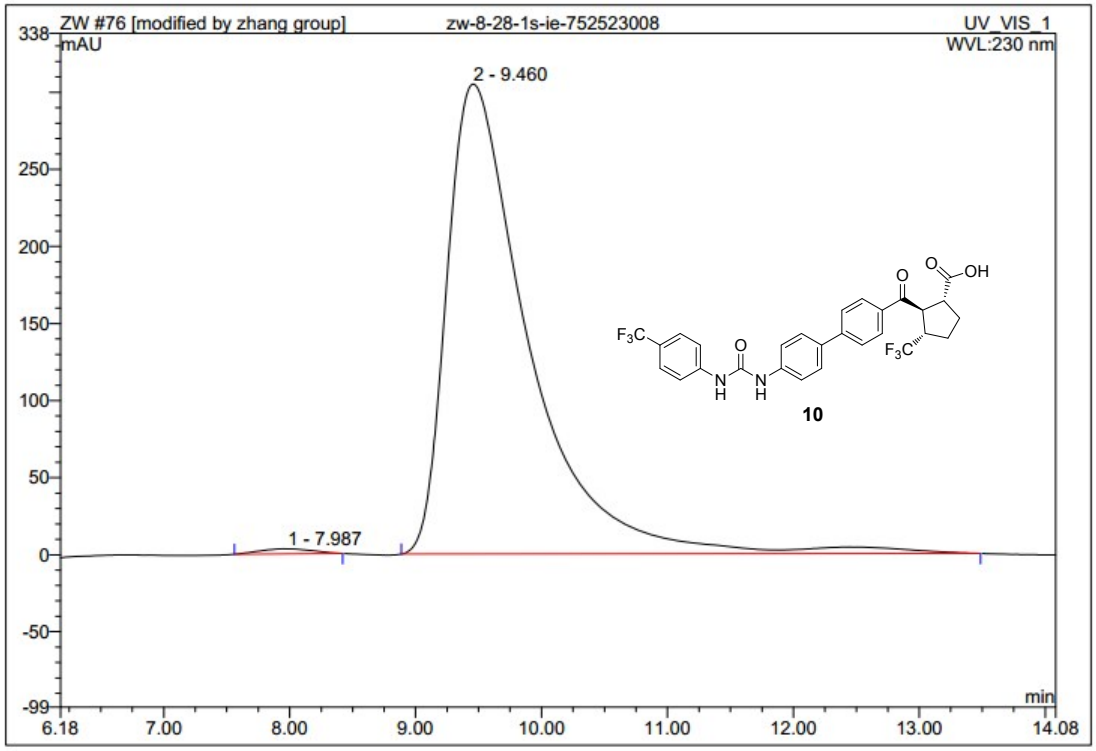


10

10; white solid; $[\alpha]_D^{20} = -237.3$ ($c = 0.33$, CH_2Cl_2); ^{1}H NMR (500 MHz, CD_3COCD_3): δ 8.62 (s, 1H), 8.48 (s, 1H), 8.18 (d, J = 8.50 Hz, 2H), 7.87–7.80 (m, 4H), 7.77–7.72 (m, 4H), 7.66 (d, J = 8.00 Hz, 2H), 4.55–4.52 (m, 1H), 3.53–3.43 (m, 1H), 3.22–3.18 (m, 1H), 2.27–2.14 (m, 3H), 2.04–1.97 (m, 1H); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 198.00, 173.44, 151.69, 145.00, 142.98, 139.60 (q, J = 3.00 Hz), 133.90, 132.86, 128.92, 127.11, 127.48 (q, J = 275.25 Hz), 126.05, 125.50 (q, J = 3.75 Hz), 124.24 (q, J = 268.88 Hz), 123.16–122.34 (m), 118.71–118.57 (m), 117.79–117.67 (m), 48.99, 47.89, 45.68 (q, J = 27.25 Hz), 28.89, 25.30 (q, J = 1.38 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ = -62.17, -70.47 ppm; Enantiomeric excess: 99%, determined by HPLC (Chiralpak IE, hexane/*i*-PrOH = 75/25; flow rate 0.8 ml/min; 25 °C; 230 nm), first peak: t_R = 7.98 min, second peak: t_R = 9.46 min.



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.92	n.a.	142.940	83.506	50.25	n.a.	BMB*
2	9.61	n.a.	106.836	82.664	49.75	n.a.	BMB*
Total:			249.776	166.170	100.00	0.000	



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.99	n.a.	3.285	1.592	0.68	n.a.	BMB*
2	9.46	n.a.	304.927	231.161	99.32	n.a.	BMB*
Total:			308.212	232.753	100.00	0.000	

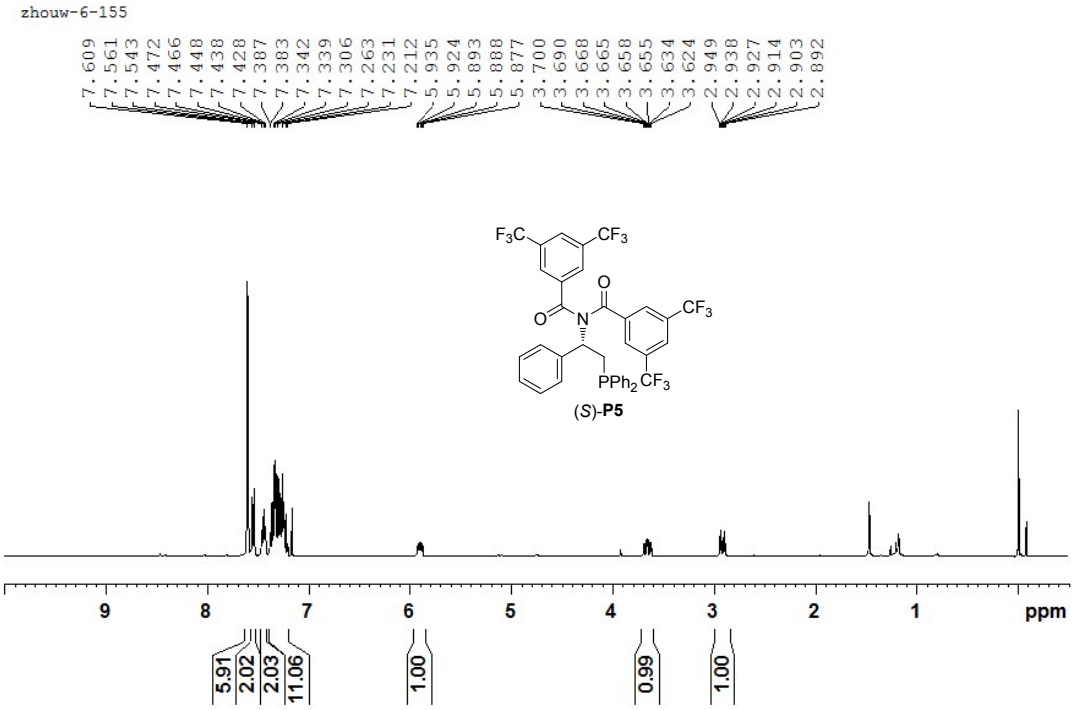
8. References

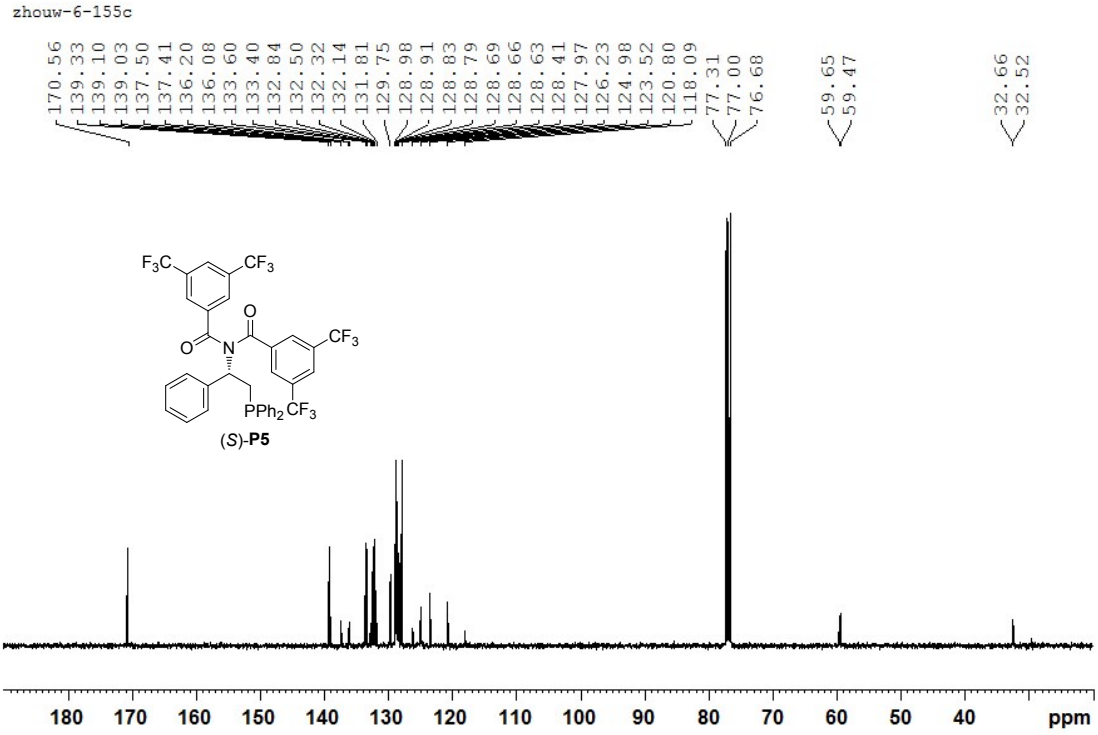
[1] a) X. Su, W. Zhou, Y. Li, J. Zhang, *Angew. Chem. Int. Ed*. **2015**, *54*, 6874; *Angew. Chem.* **2015**, *127*, 6978; b) W. Zhou, X. Su. M. Tao, C. Zhu, Q. Zhao, J. Zhang, *Angew. Chem. Int. Ed*. **2015**, *54*, 14853; *Angew. Chem.* **2015**, *127*, 15066; c) W. Zhou, P. Chen, M. Tao, X. Su, Q. Zhao, J. Zhang, *Chem. Commun*. **2016**, *52*, 7612; d) P. Chen, X. Su, W. Zhou, Y. Xiao, J. Zhang, *Tetrahedron* **2016**, *72*, 2700.

[2] T. Yamazaki, T. Kawasaki-Takasuka, A. Furuta, S. Sakamoto, *Tetrahedron*, **2009**, *65*, 5945.

[3] A. Voituriez, A. Panossian, N. Fleury-Brégeot, P. Retailleau, A. Marinetti, *J. Am. Chem. Soc.* **2008**, *130*, 14030.

9. ^{1}H, ^{13}C, ^{31}P and ^{19}F NMR Spectra





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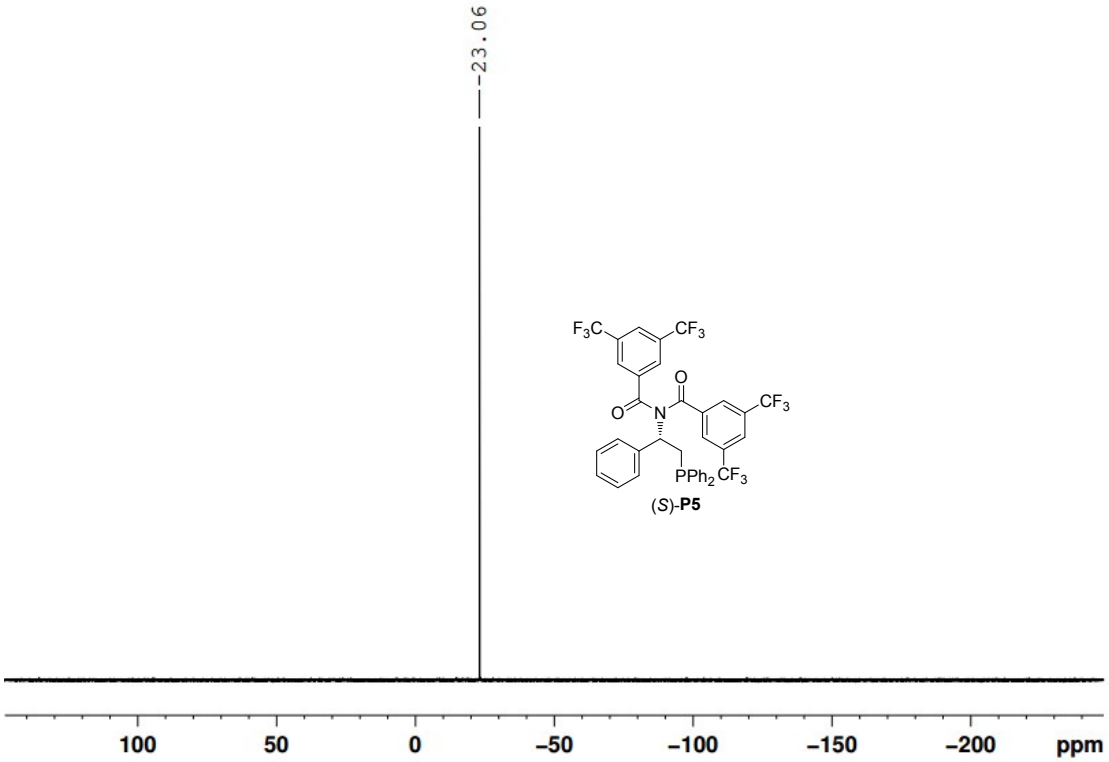

(*S*)-**P5**

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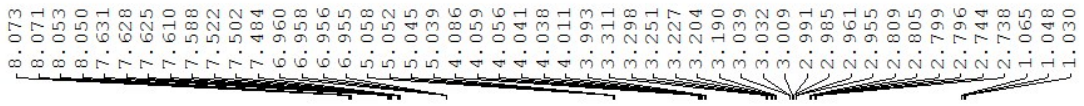

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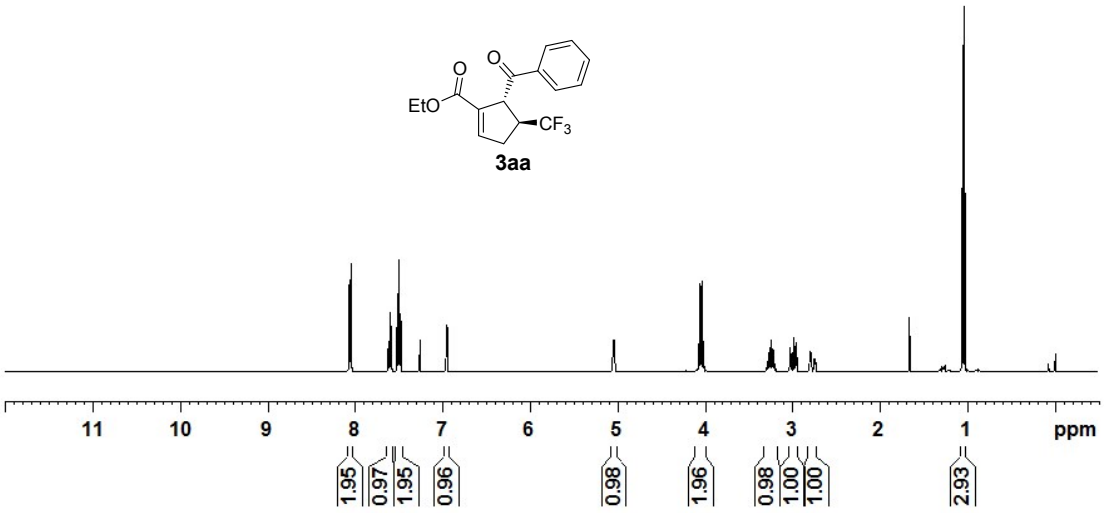

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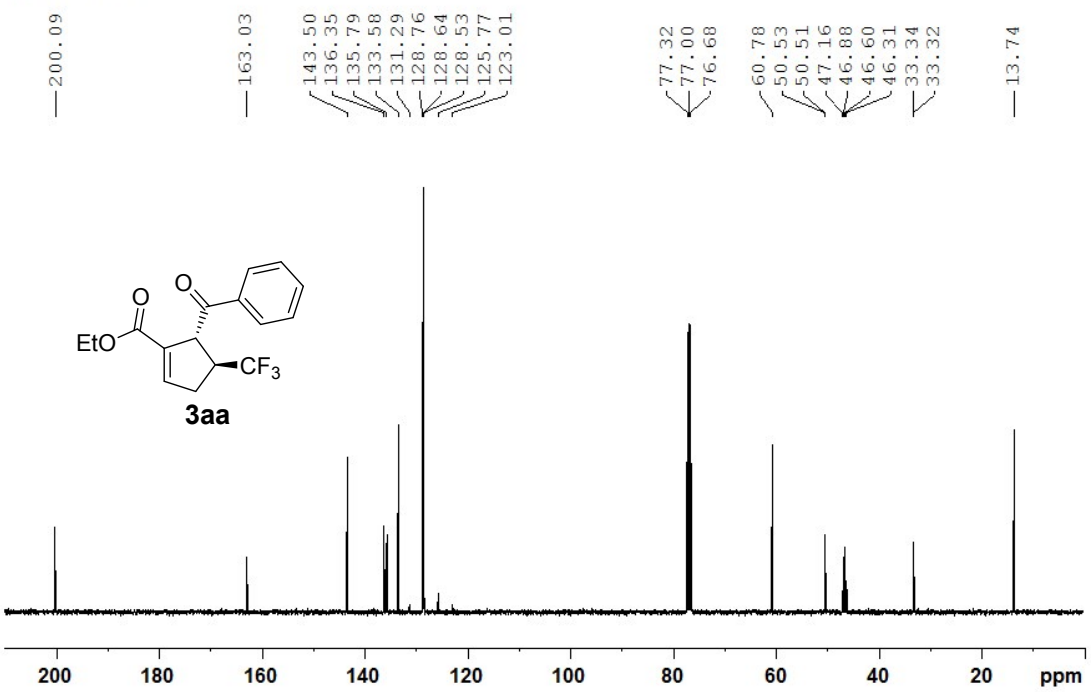

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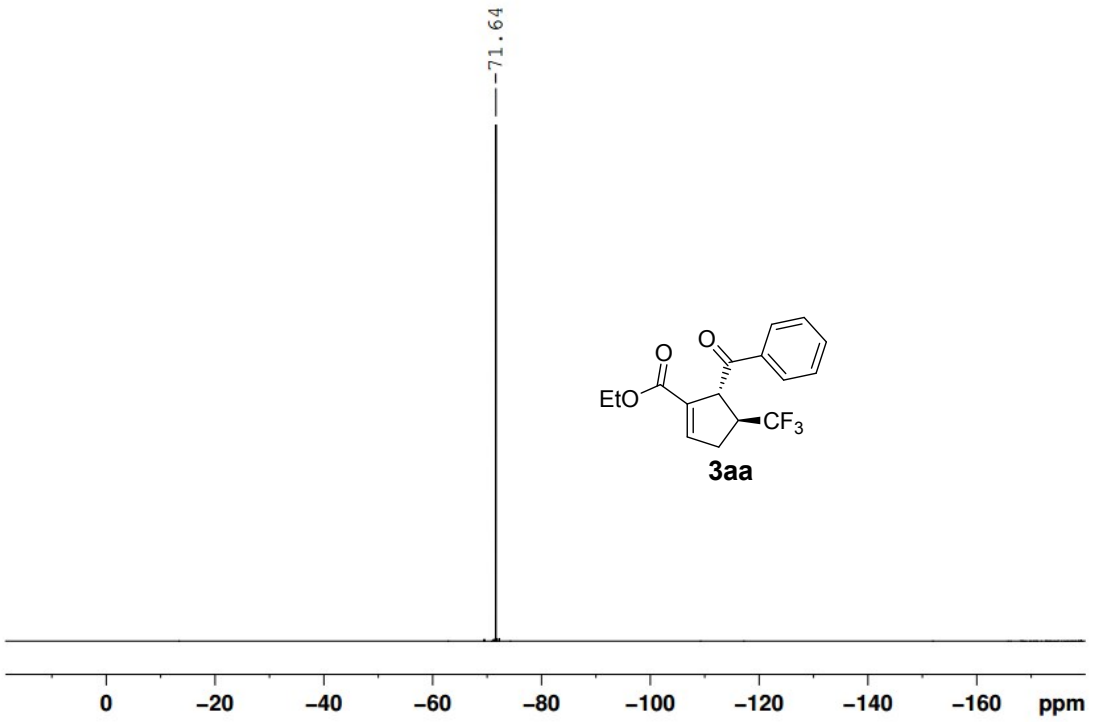

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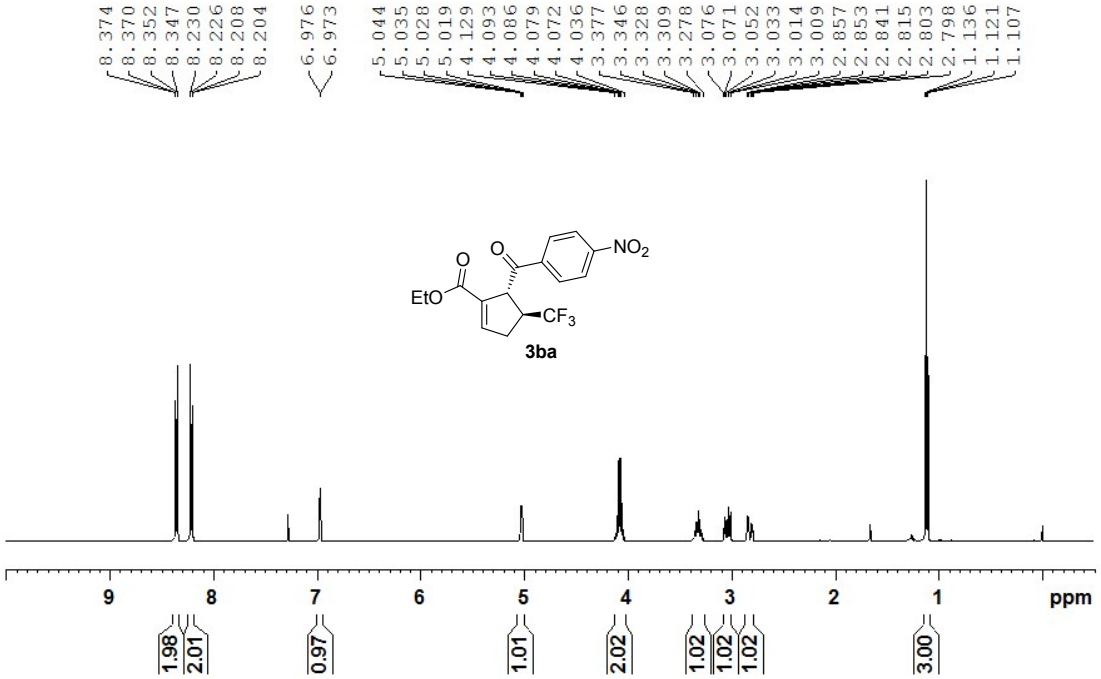

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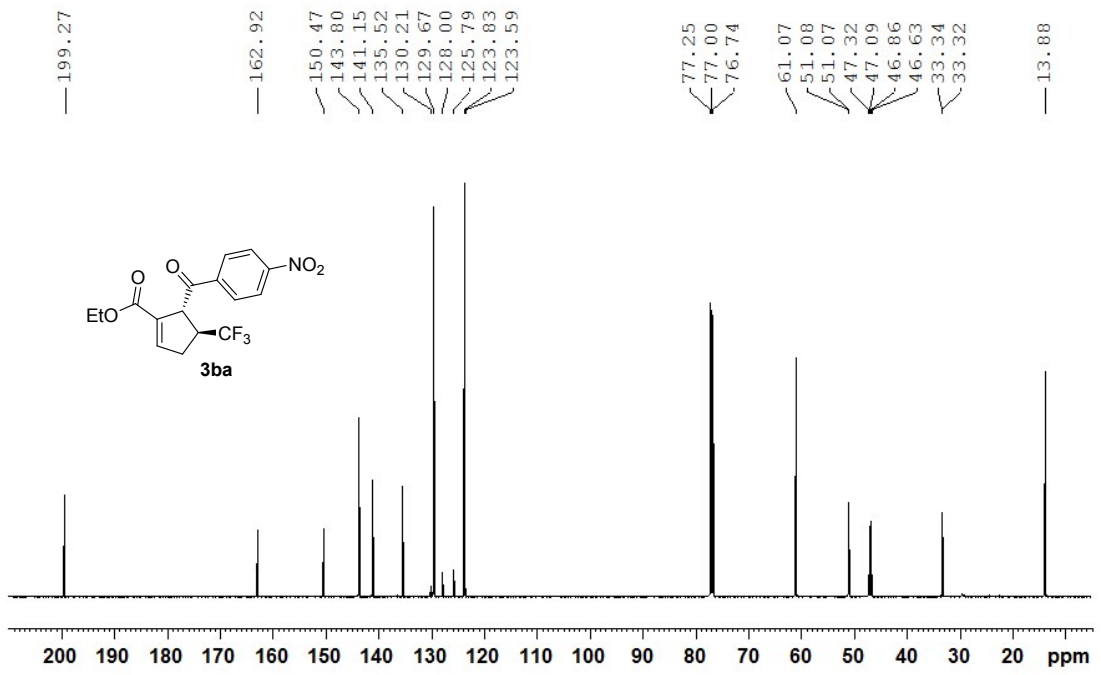

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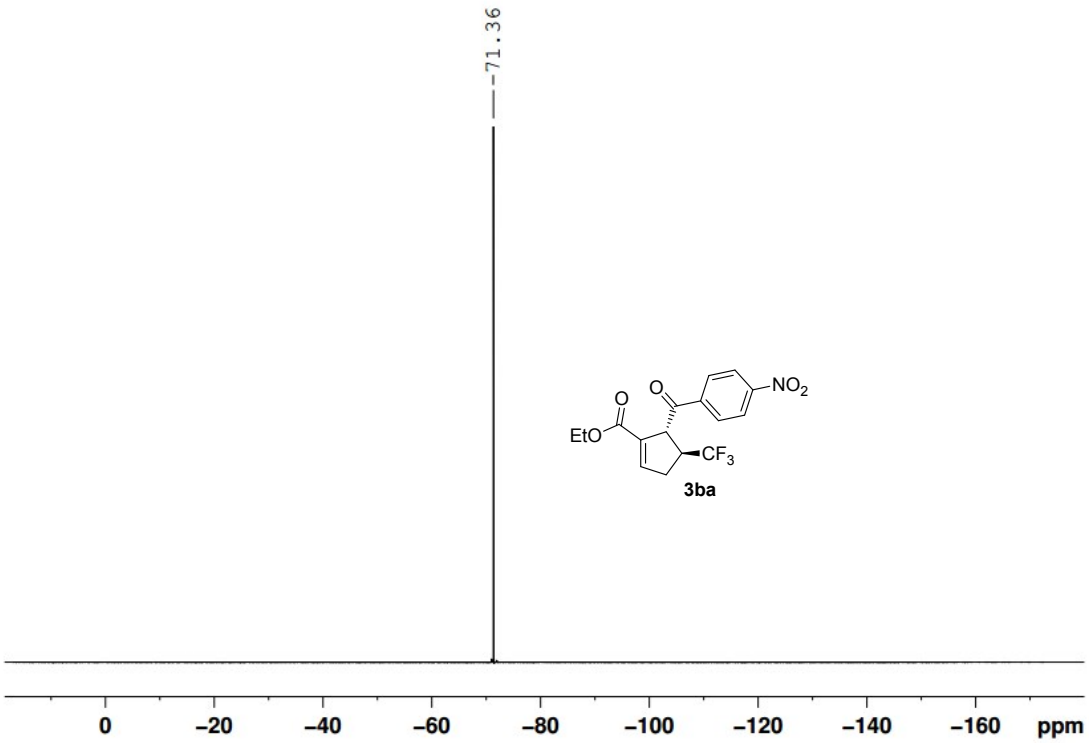

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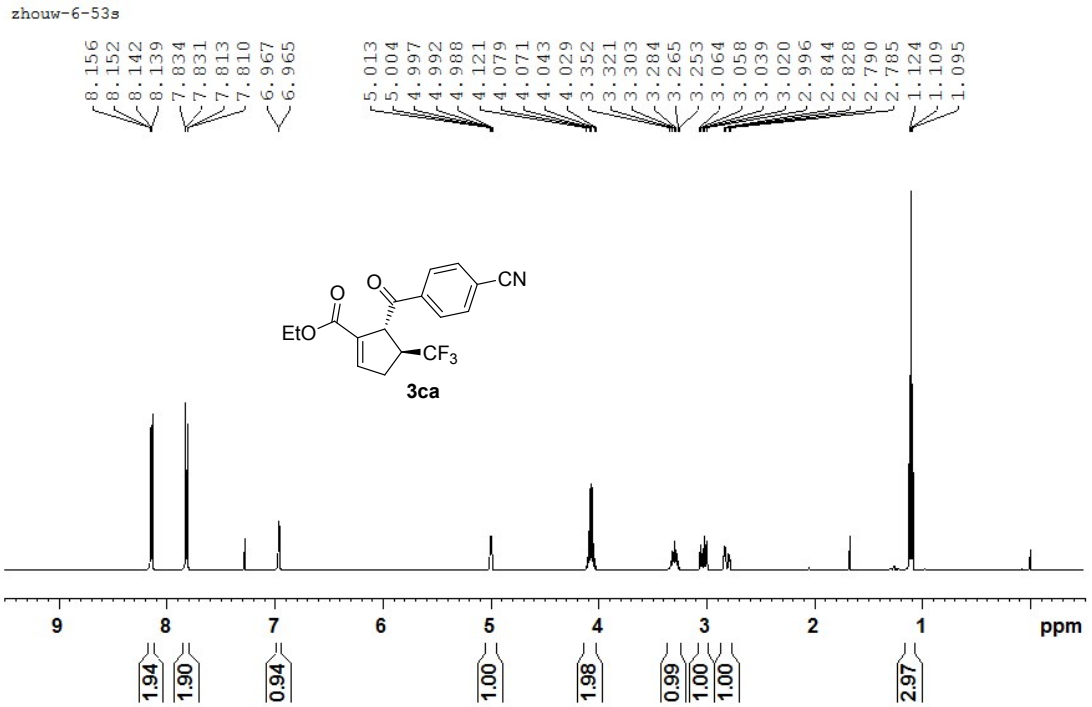

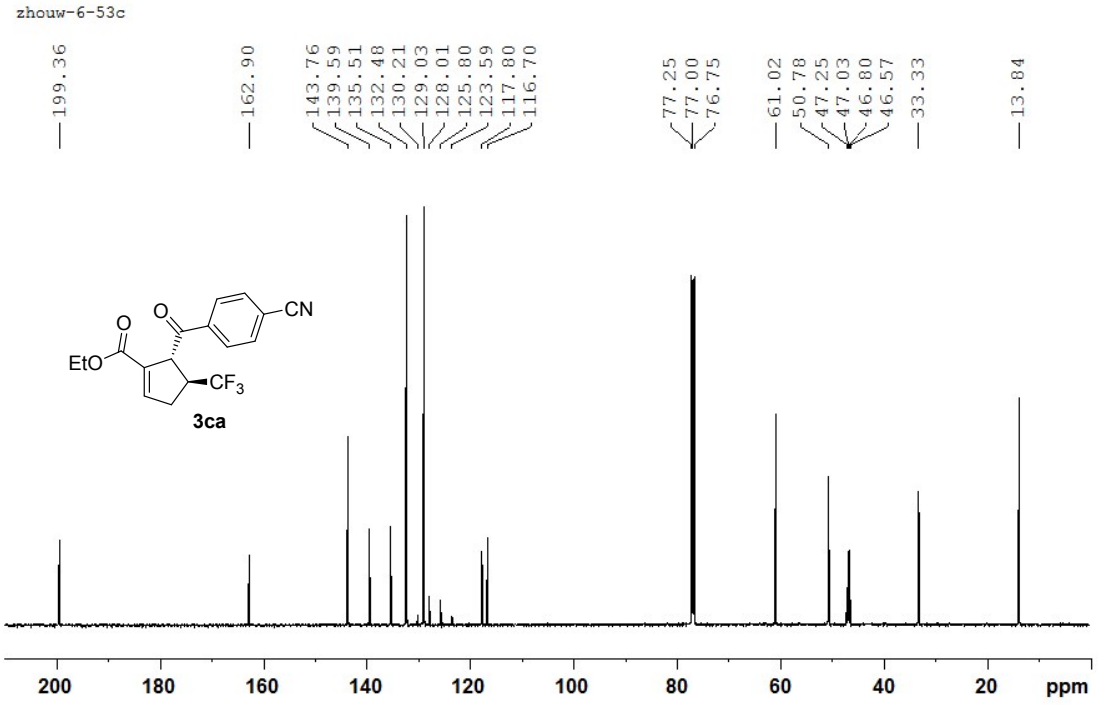
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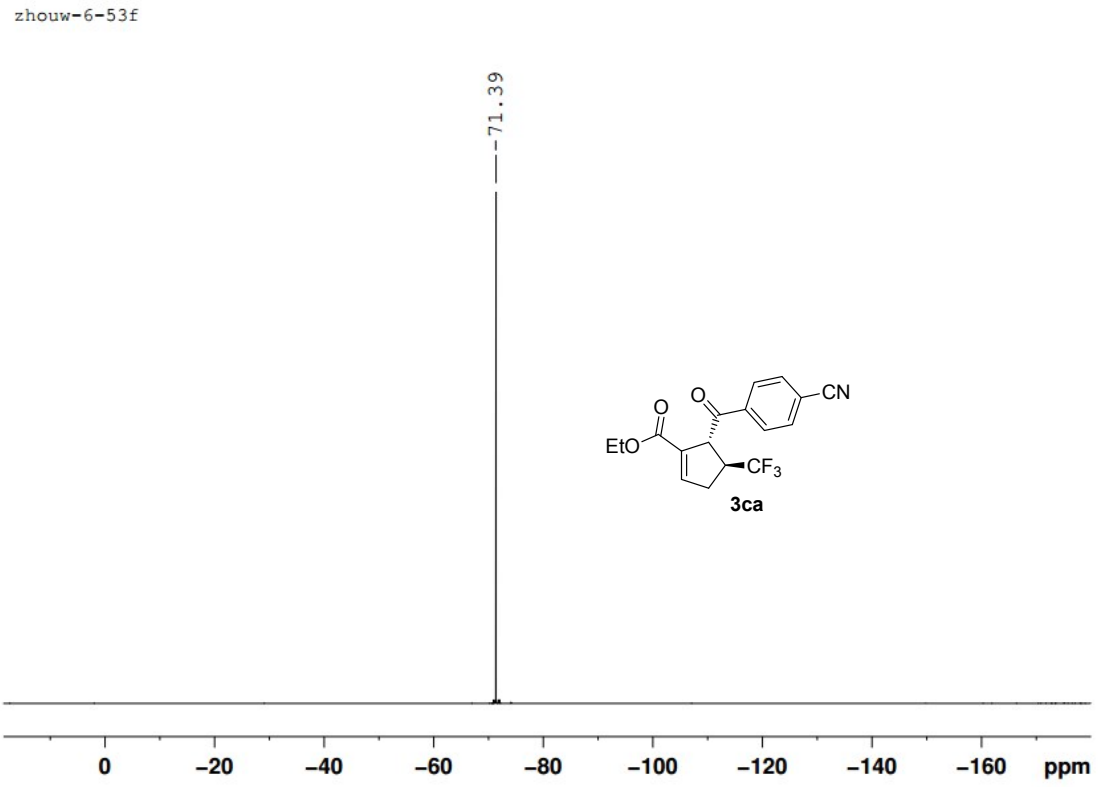
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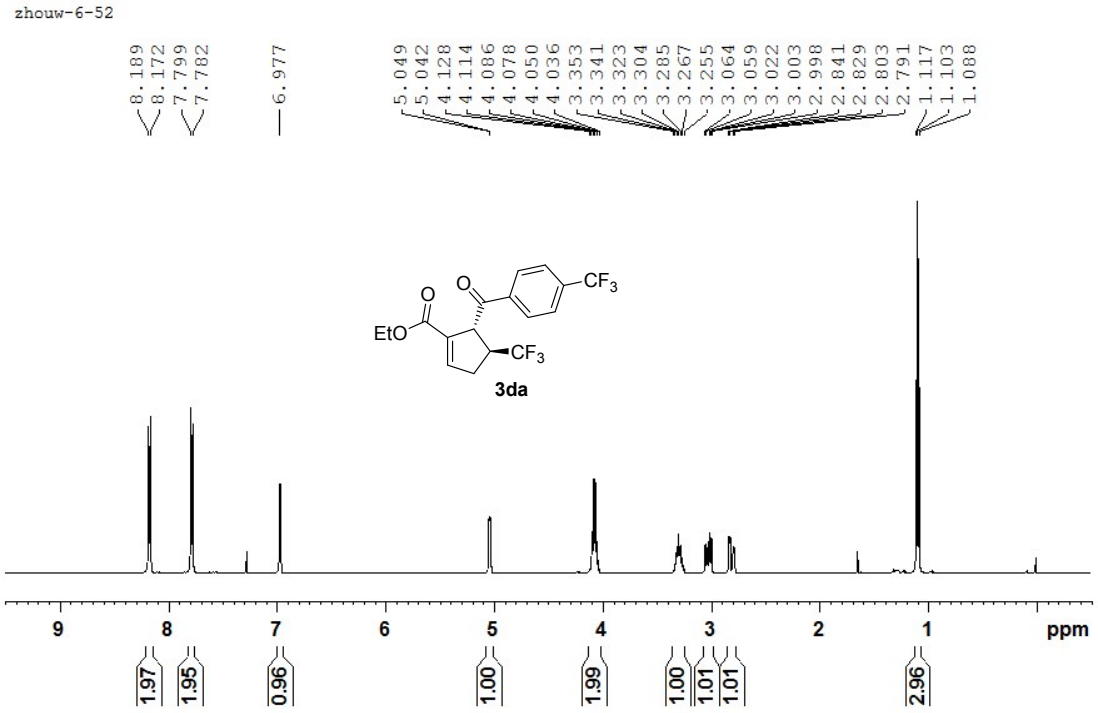
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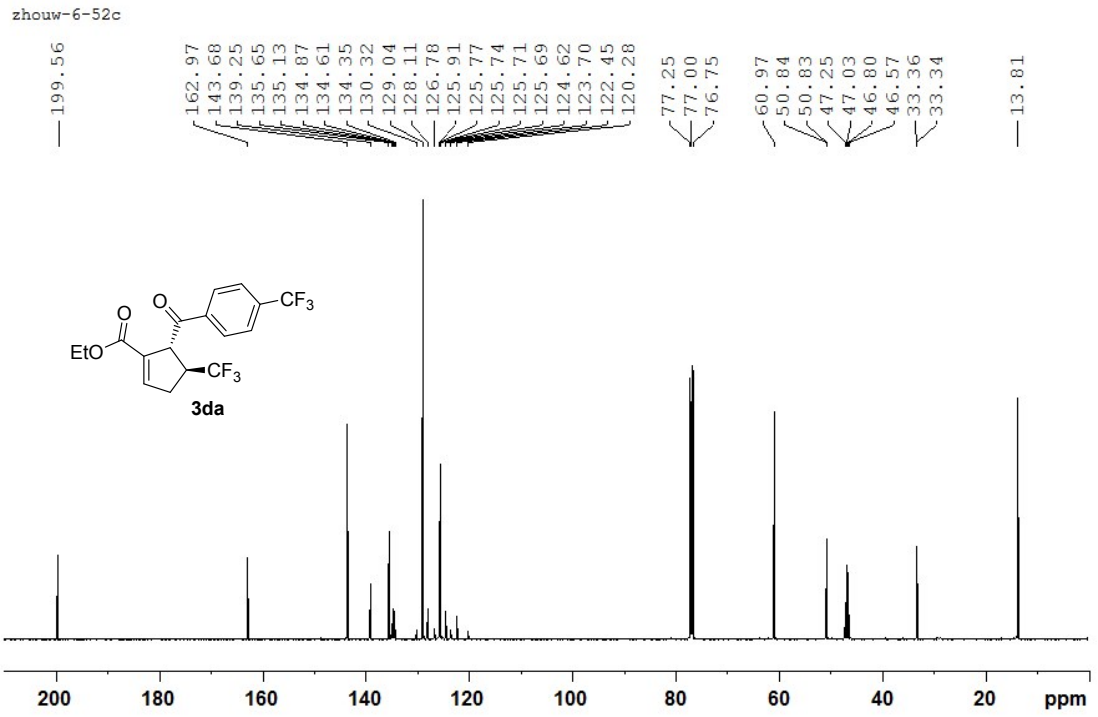
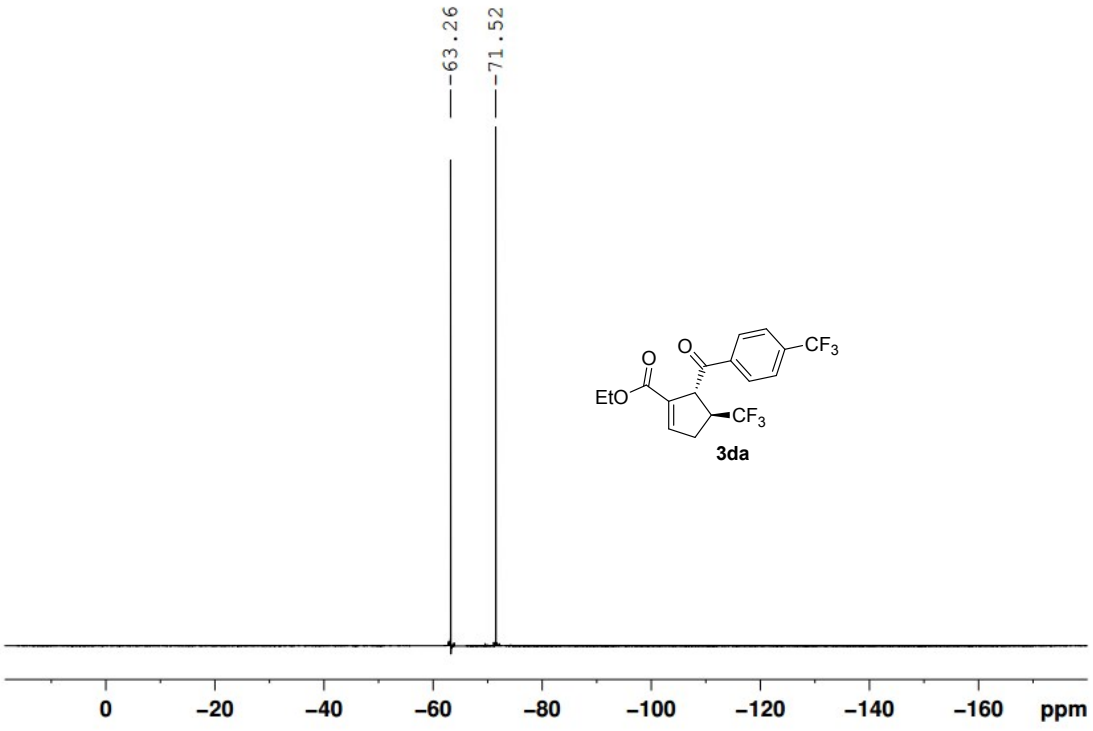
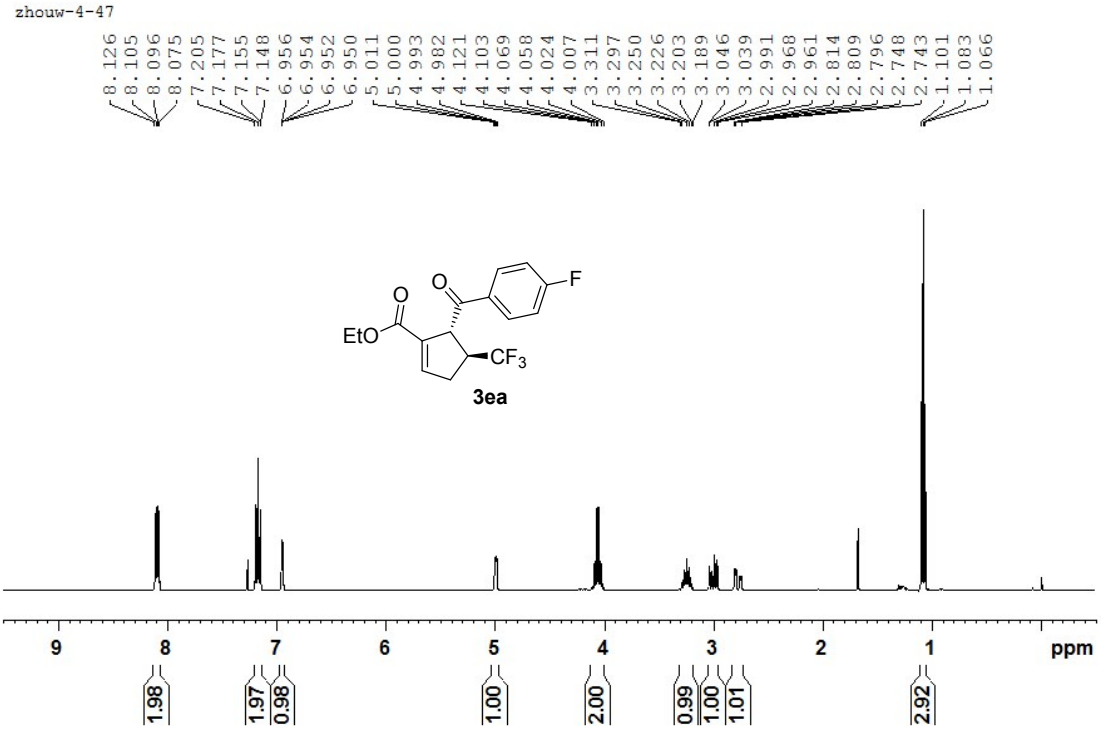
CF3
CF3
3da
9
8
7
6
5
4
3
2
1
ppm
1.97
1.95
0.96
1.00
1.99
1.00
1.01
1.01
2.96


zhouw-6-52c
199.56
162.97
143.68
139.25
135.65
135.13
134.87
134.61
134.35
130.32
129.04
128.11
126.78
125.91
125.77
125.74
125.71
125.69
124.62
123.70
122.45
120.28
77.25
77.00
76.75
60.97
50.84
50.83
47.25
47.03
46.80
46.57
33.36
33.34
13.81
EtO
CF3
CF3
3da
200
180
160
140
120
100
80
60
40
20
ppm

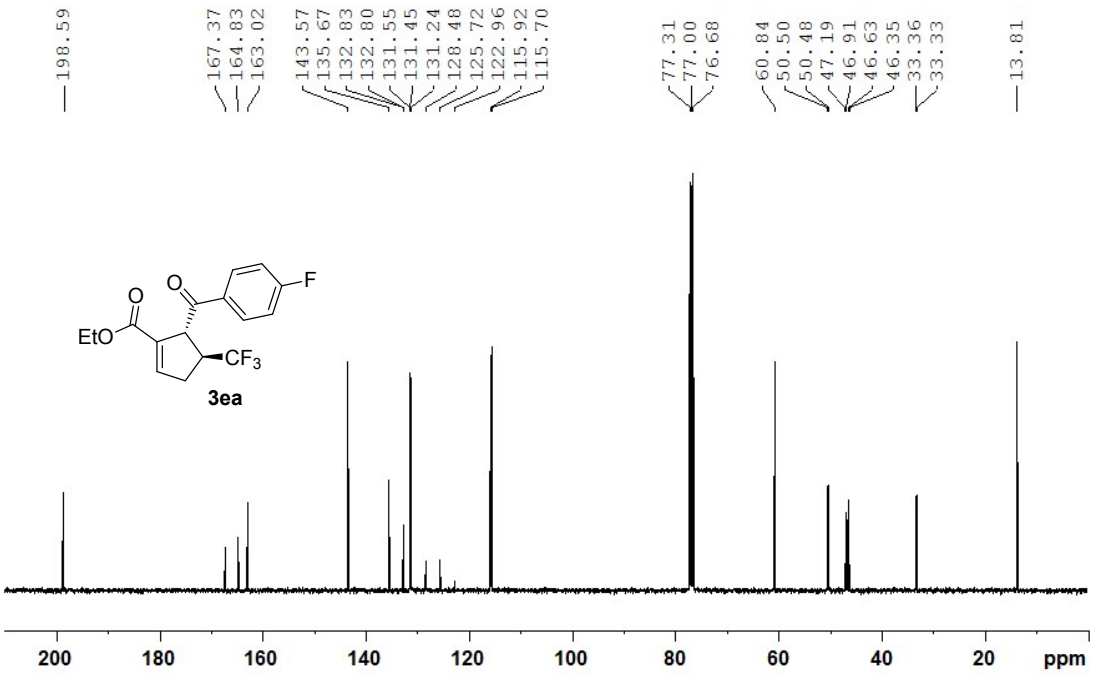
zhouw-6-52f



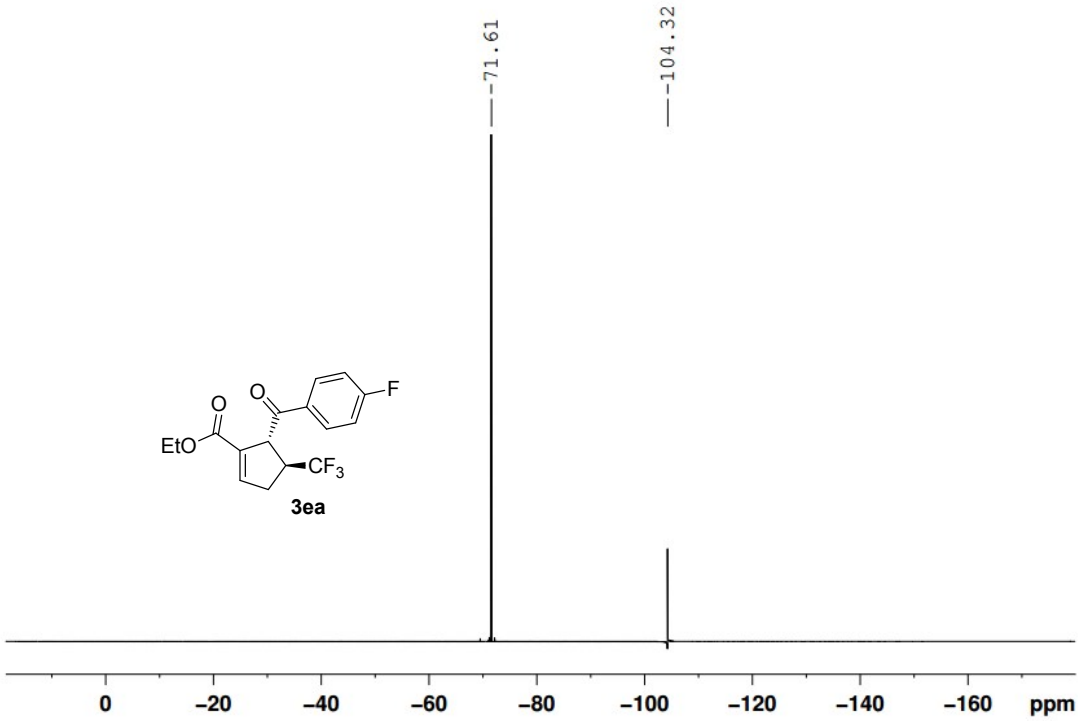
zhouw-4-47

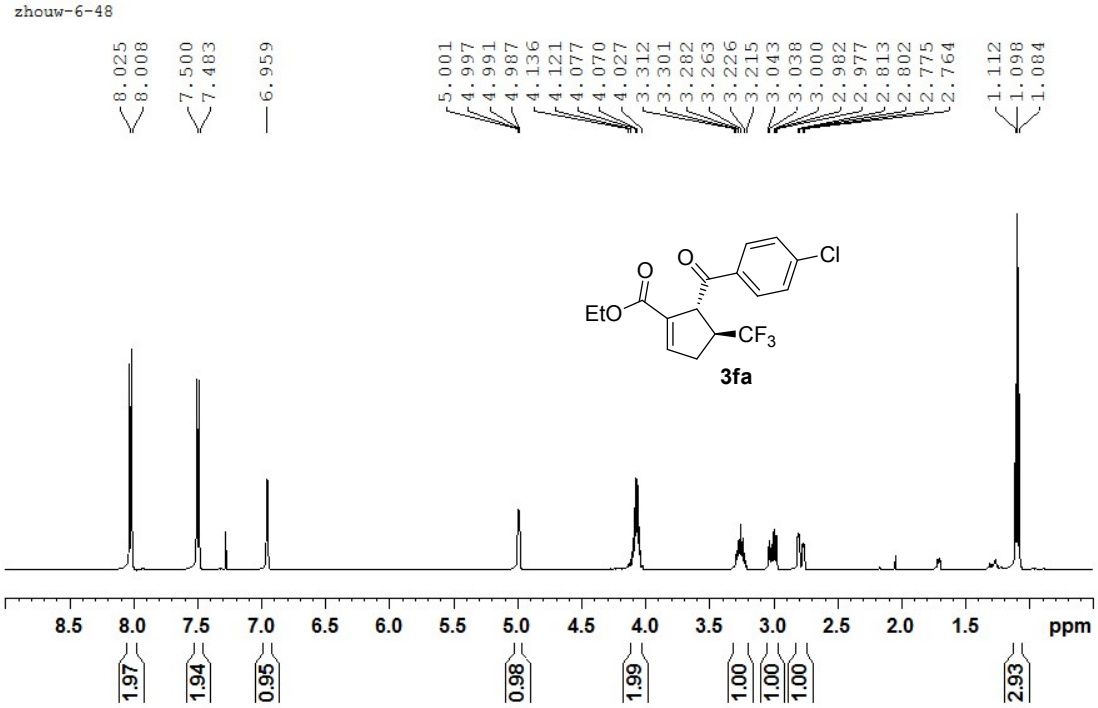


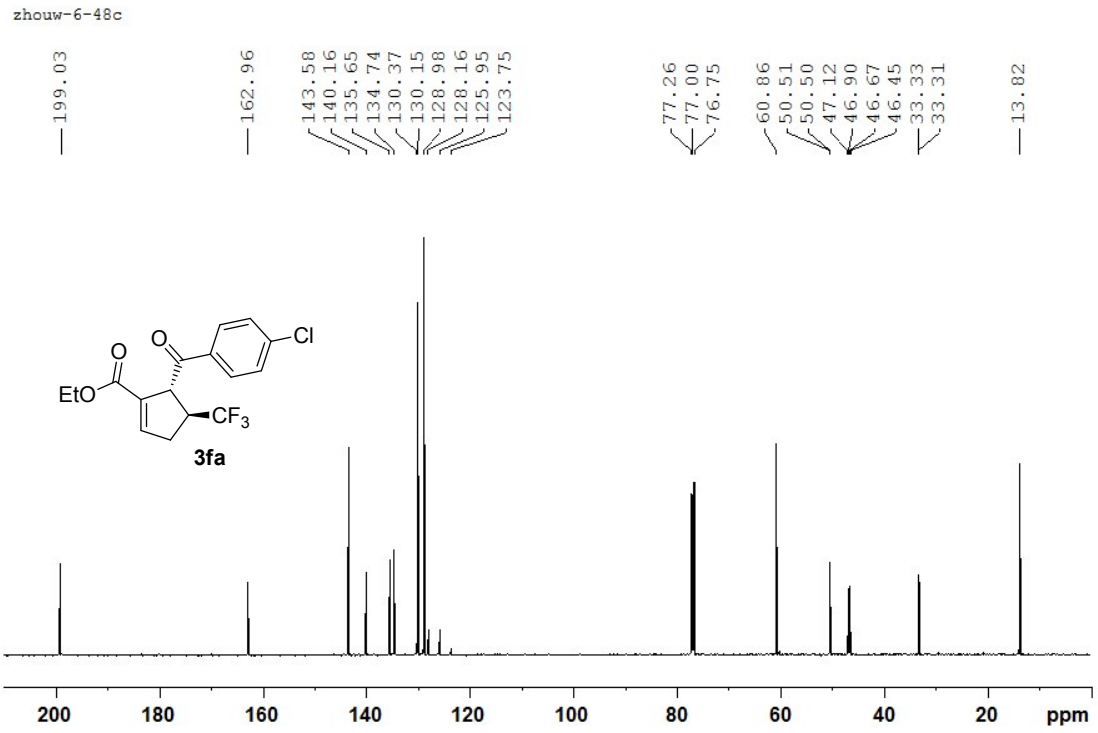
zhouw-6-47c



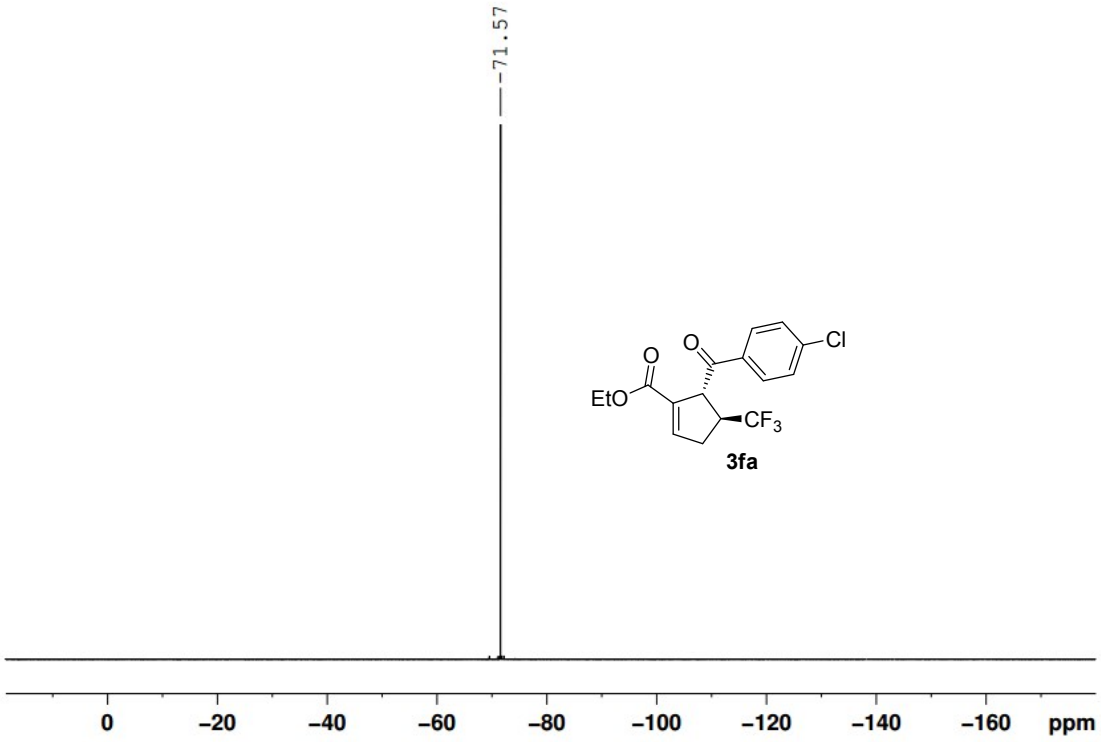
zhouw-6-47f



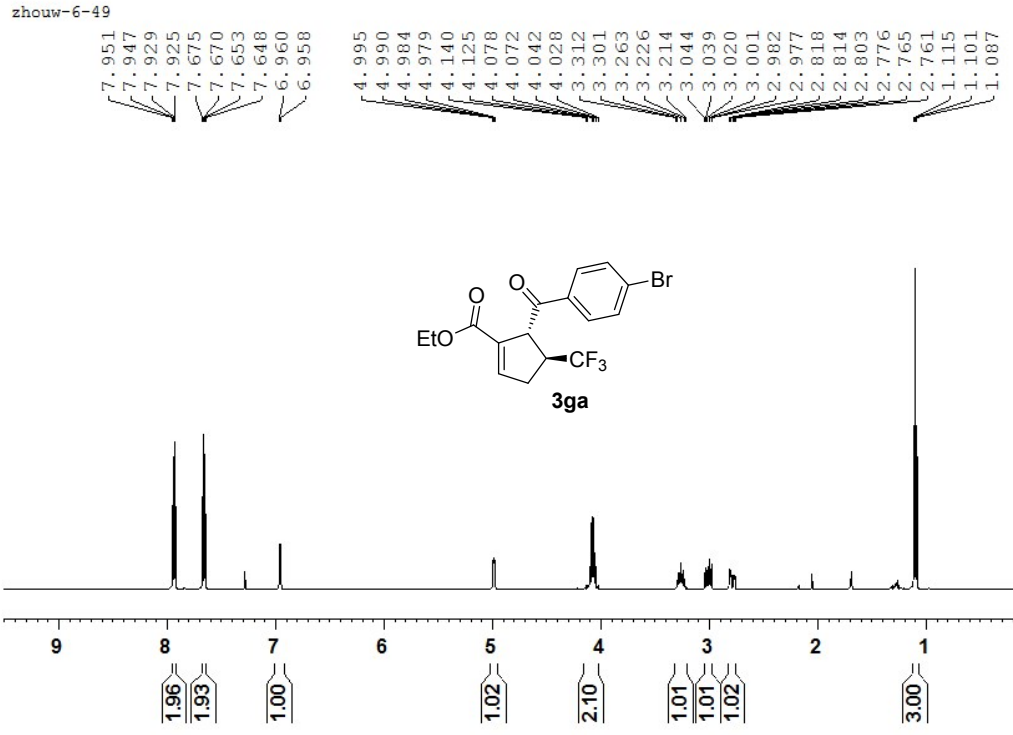

zhouw-6-48
8.025
8.008
7.500
7.483
6.959
5.001
4.997
4.991
4.987
4.136
4.121
4.077
4.070
4.027
3.312
3.301
3.282
3.263
3.226
3.215
3.043
3.038
3.000
2.982
2.977
2.813
2.802
2.775
2.764
1.112
1.098
1.084
O
O
Cl
EtO
CF3
3fa
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.97
1.94
0.95
0.98
1.99
1.00
1.00
1.00
2.93


zhouw-6-48c
199.03
162.96
143.58
140.16
135.65
134.74
130.37
130.15
128.98
128.16
125.95
123.75
77.26
77.00
76.75
60.86
50.51
50.50
47.12
46.90
46.67
46.45
33.33
33.31
13.82
O
O
Cl
EtO
CF3
3fa
200
180
160
140
120
100
80
60
40
20
ppm

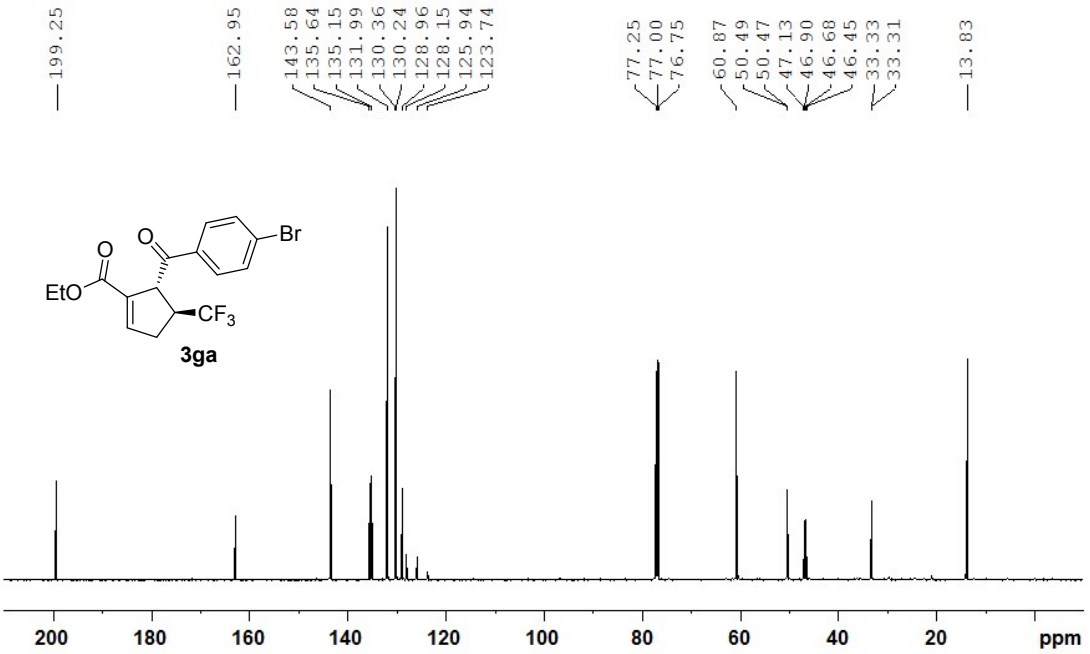
zhouw-6-48f



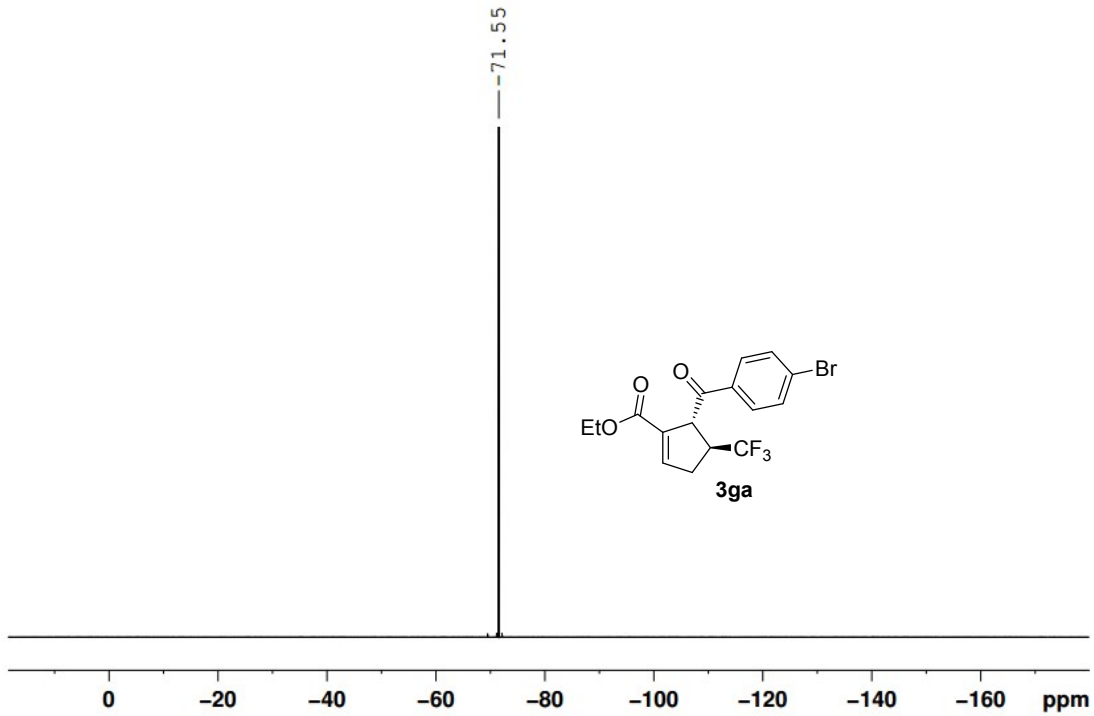
zhouw-6-49

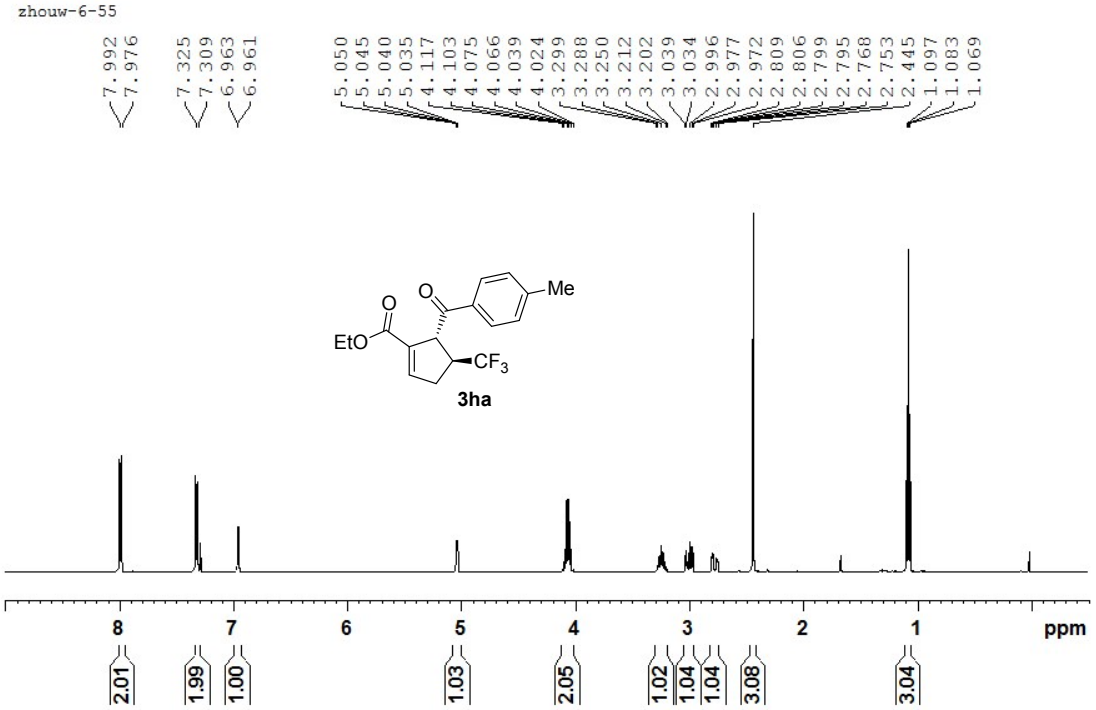


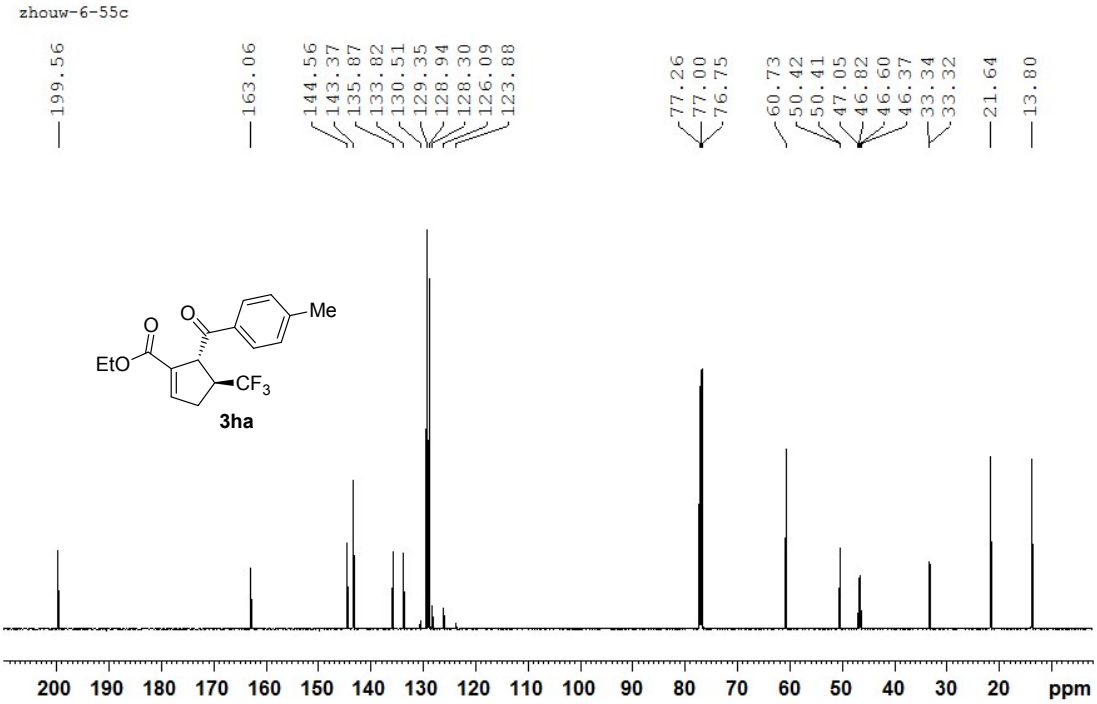
zhouw-6-49c



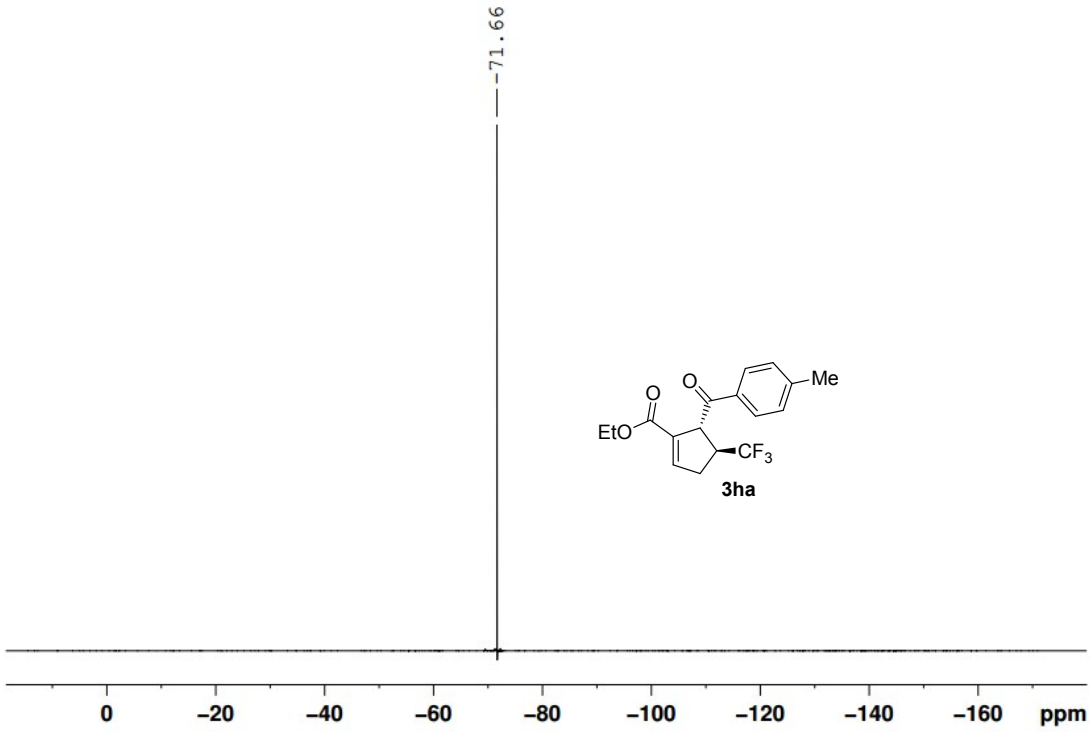
zhouw-6-49f



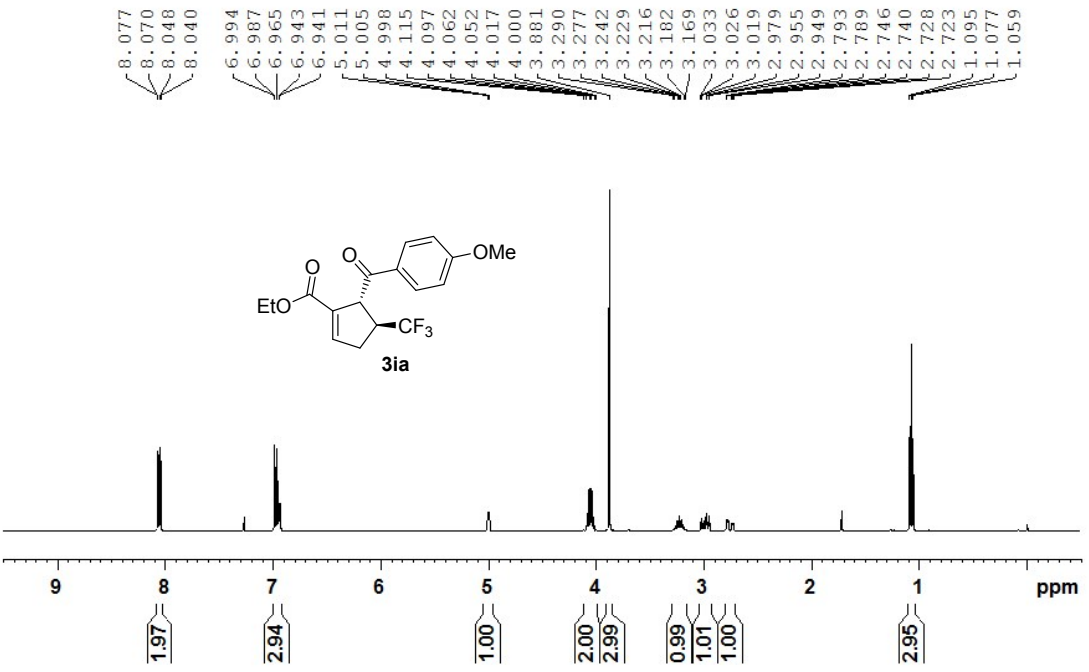

zhouw-6-55
7.992
7.976
7.325
7.309
6.963
6.961
5.050
5.045
5.040
5.035
4.117
4.103
4.075
4.066
4.039
4.024
3.299
3.288
3.250
3.212
3.202
3.039
3.034
2.996
2.977
2.972
2.809
2.806
2.799
2.795
2.768
2.753
2.445
1.097
1.083
1.069
O
O
Me
EtO
CF3
3ha
8
7
6
5
4
3
2
1
ppm
2.01
1.99
1.00
1.03
2.05
1.02
1.04
1.04
3.08
3.04

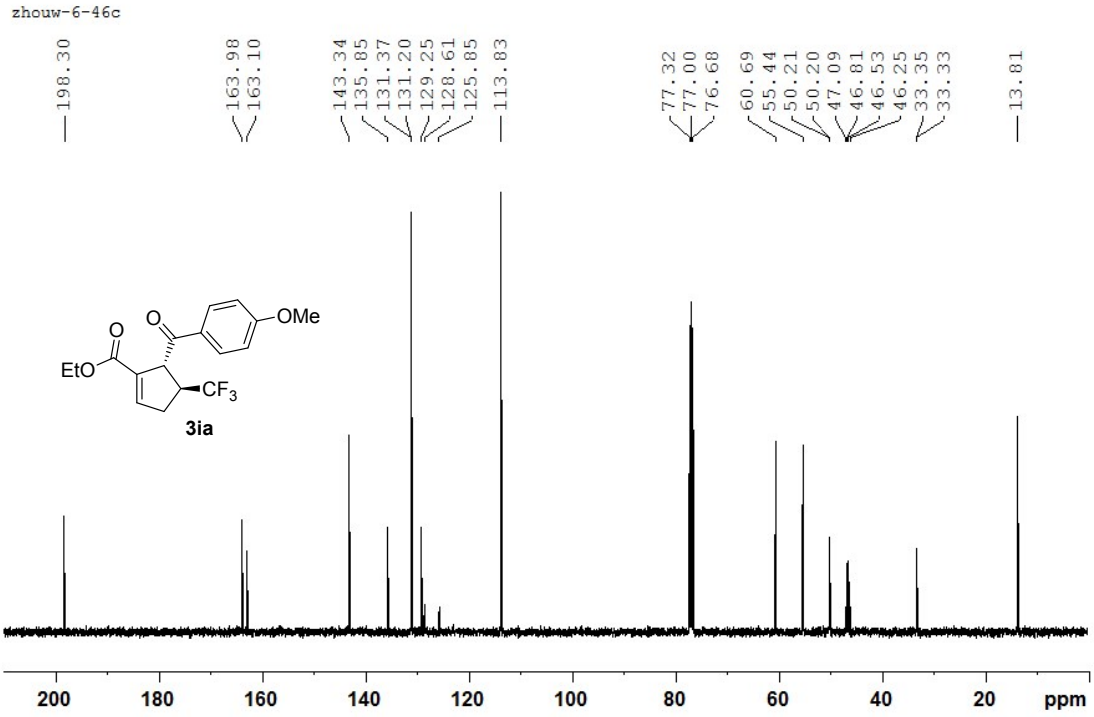

zhouw-6-55c
199.56
163.06
144.56
143.37
135.87
133.82
130.51
129.35
128.94
128.30
126.09
123.88
77.26
77.00
76.75
60.73
50.42
50.41
47.05
46.82
46.60
46.37
33.34
33.32
21.64
13.80
O
O
Me
EtO
CF3
3ha
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

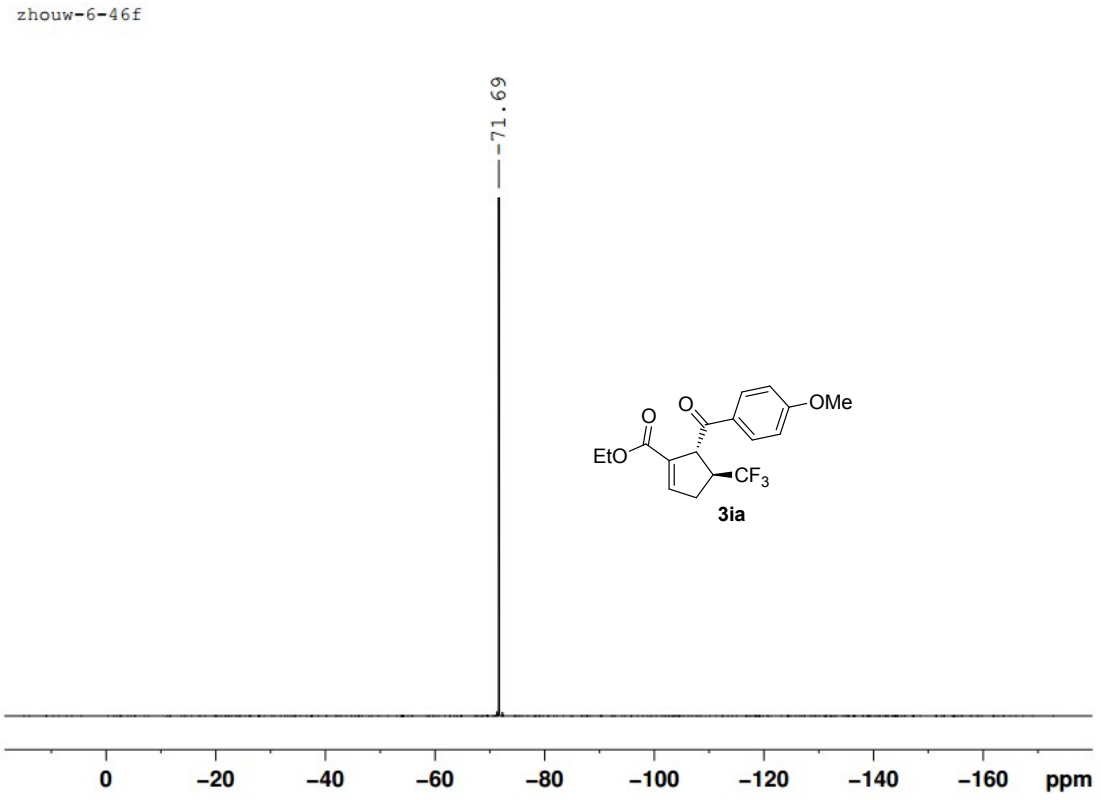
zhouw-6-55f

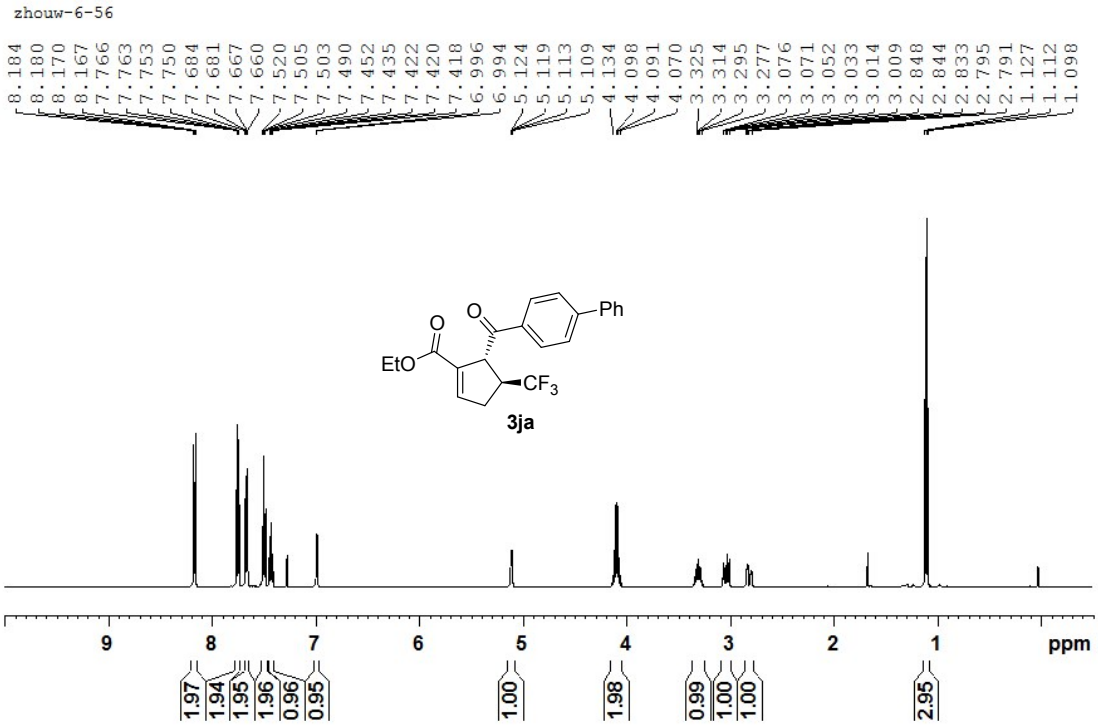


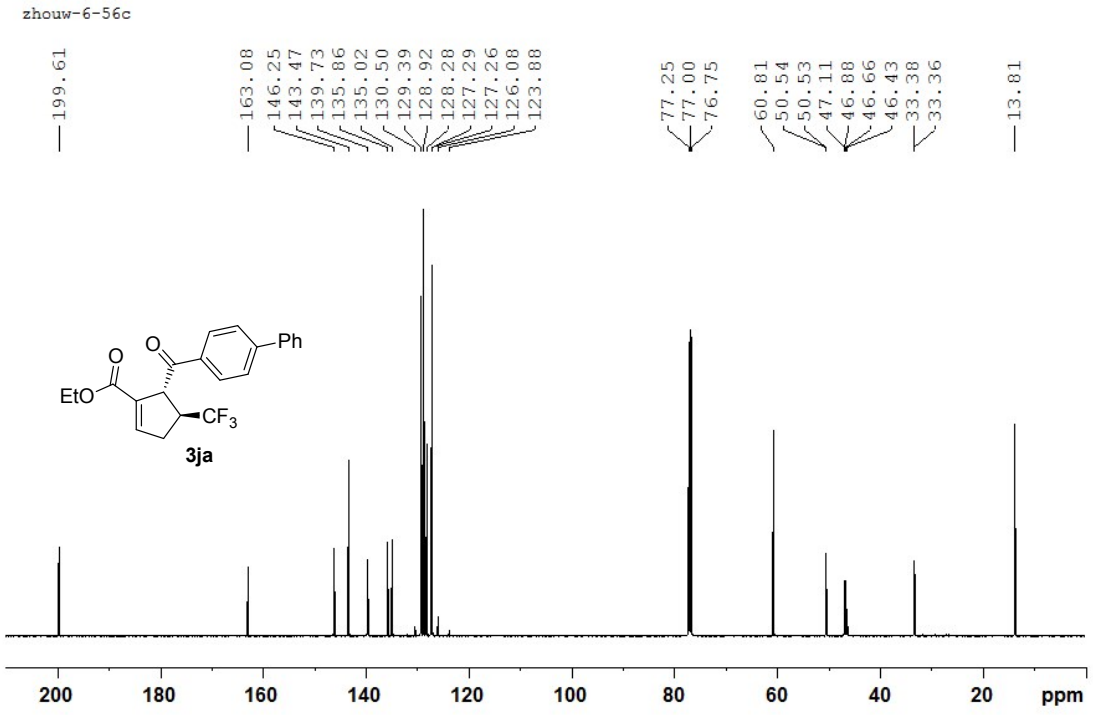
zhouw-6-46



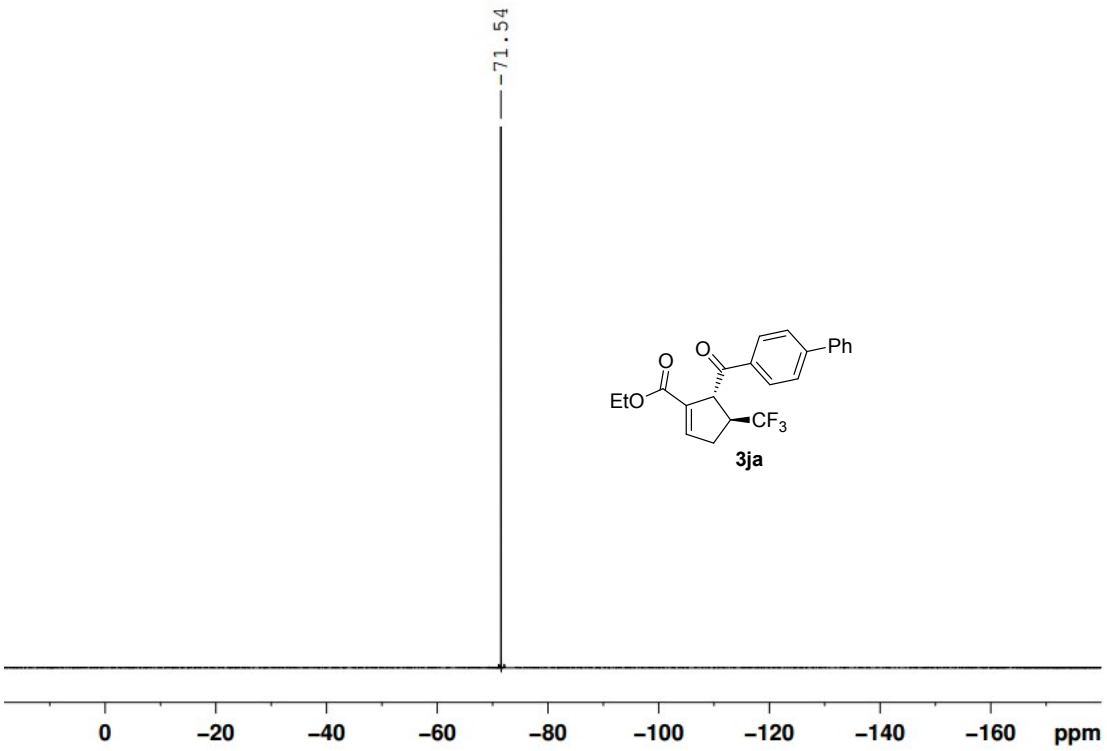

zhouw-6-46c
198.30
163.98
163.10
143.34
135.85
131.37
131.20
129.25
128.61
125.85
113.83
77.32
77.00
76.68
60.69
55.44
50.21
50.20
47.09
46.81
46.53
46.25
33.35
33.33
13.81
O
O
OMe
EtO
CF3
3ia
200
180
160
140
120
100
80
60
40
20
ppm


zhouw-6-46f
-71.69
O
O
OMe
EtO
CF3
3ia
0
-20
-40
-60
-80
-100
-120
-140
-160
ppm

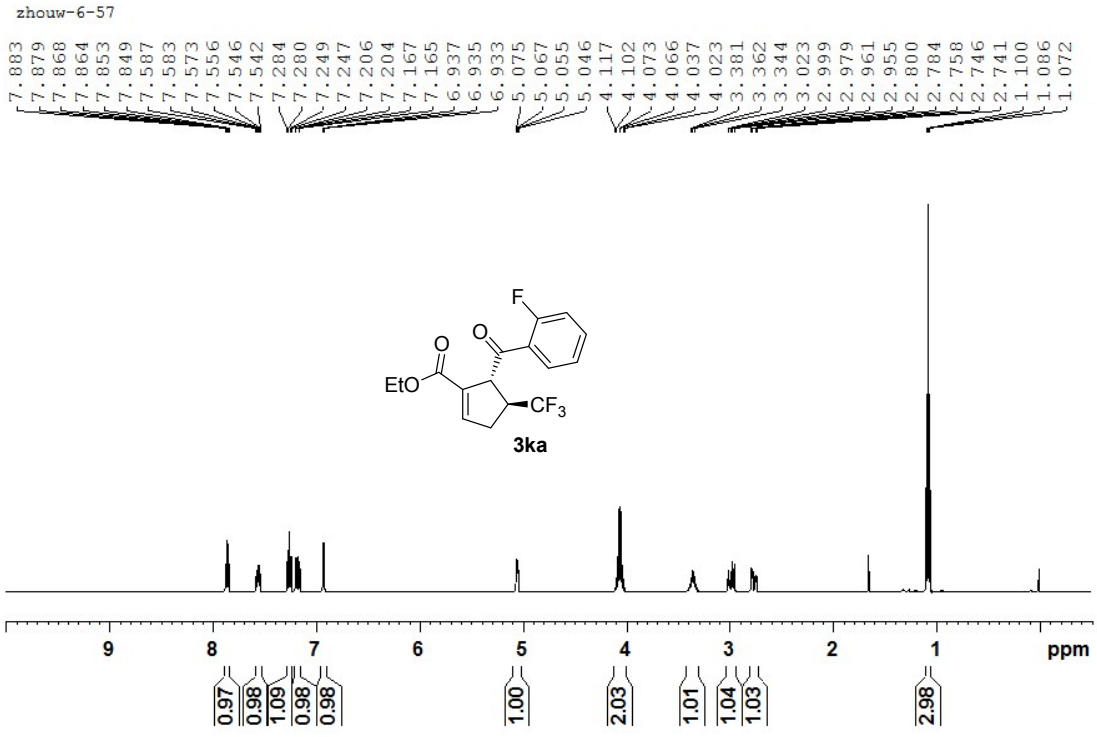

zhouw-6-56
8.184
8.180
8.170
8.167
7.766
7.763
7.753
7.750
7.684
7.681
7.667
7.660
7.520
7.505
7.503
7.490
7.452
7.435
7.422
7.420
7.418
6.996
6.994
5.124
5.119
5.113
5.109
4.134
4.098
4.091
4.070
3.325
3.314
3.295
3.277
3.076
3.071
3.052
3.033
3.014
3.009
2.848
2.844
2.833
2.795
2.791
1.127
1.112
1.098
O
O
EtO
Ph
CF3
3ja
9
8
7
6
5
4
3
2
1
ppm
1.97
1.94
1.95
1.96
0.96
0.95
1.00
1.98
0.99
1.00
1.00
2.95

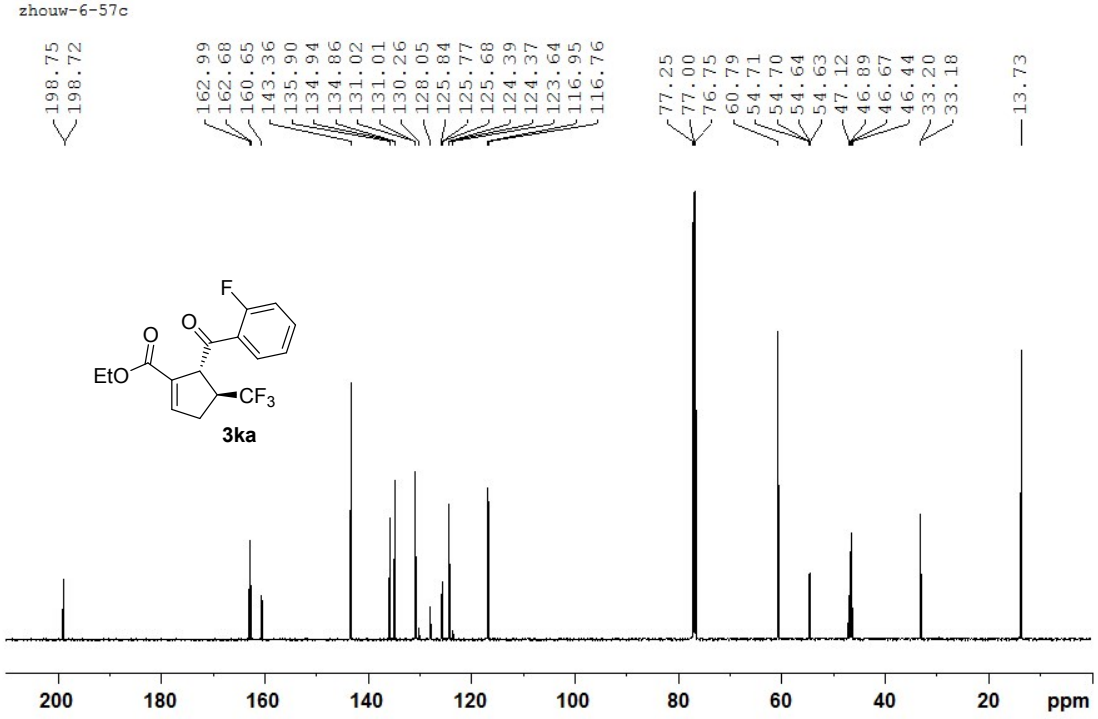

zhouw-6-56c
199.61
163.08
146.25
143.47
139.73
135.86
135.02
130.50
129.39
128.92
128.28
127.29
127.26
126.08
123.88
77.25
77.00
76.75
60.81
50.54
50.53
47.11
46.88
46.66
46.43
33.38
33.36
13.81
O
O
EtO
Ph
CF3
3ja
200
180
160
140
120
100
80
60
40
20
ppm

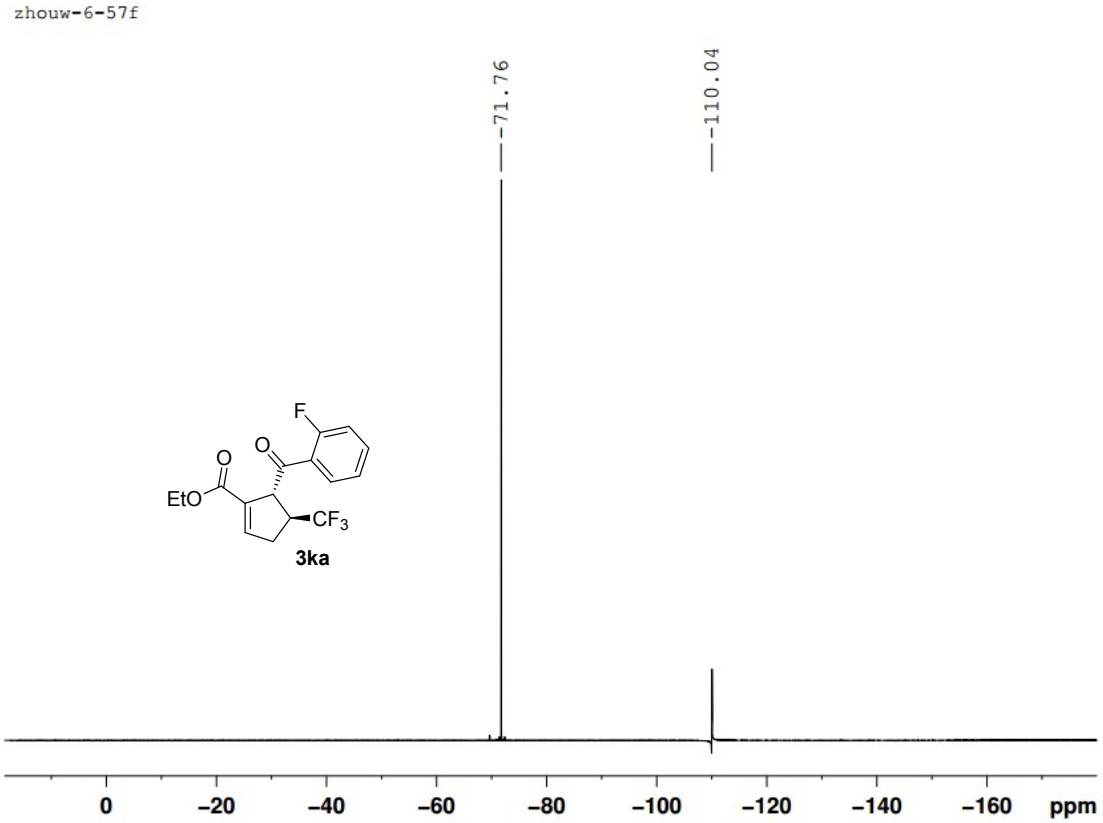
zhouw-6-56f



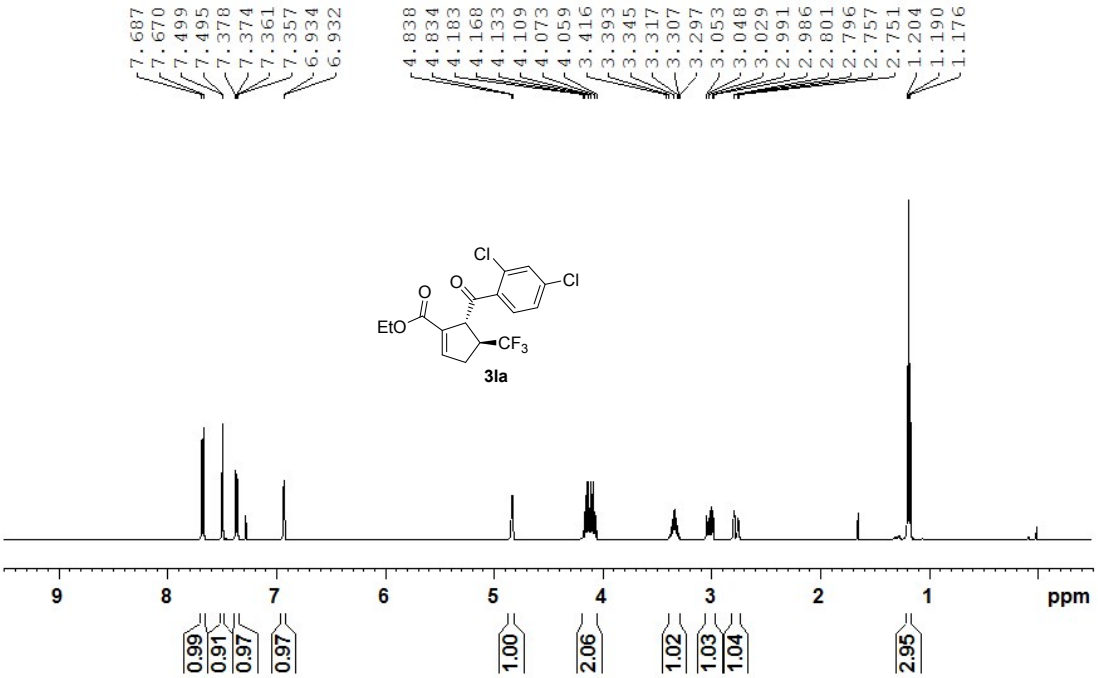
zhouw-6-57



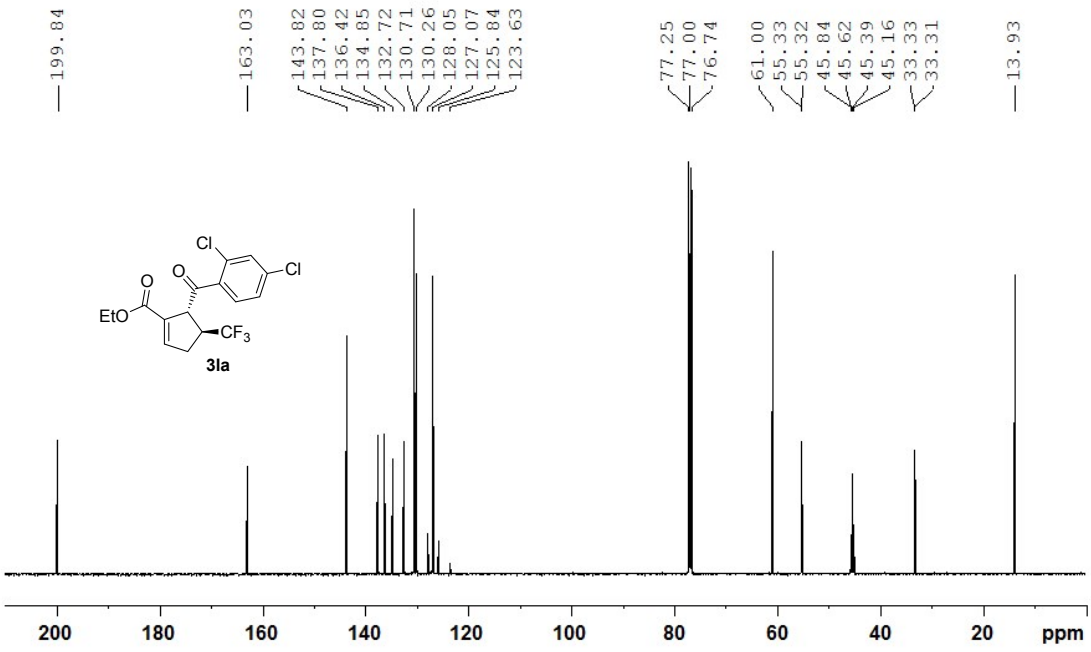

zhouw-6-57c
198.75
198.72
162.99
162.68
160.65
143.36
135.90
134.94
134.86
131.02
131.01
130.26
128.05
125.84
125.77
125.68
124.39
124.37
123.64
116.95
116.76
77.25
77.00
76.75
60.79
54.71
54.70
54.64
54.63
47.12
46.89
46.67
46.44
33.20
33.18
13.73
F
O
O
EtO
CF3
3ka
200
180
160
140
120
100
80
60
40
20
ppm


zhouw-6-57f
-71.76
-110.04
F
O
O
EtO
CF3
3ka
0
-20
-40
-60
-80
-100
-120
-140
-160
ppm

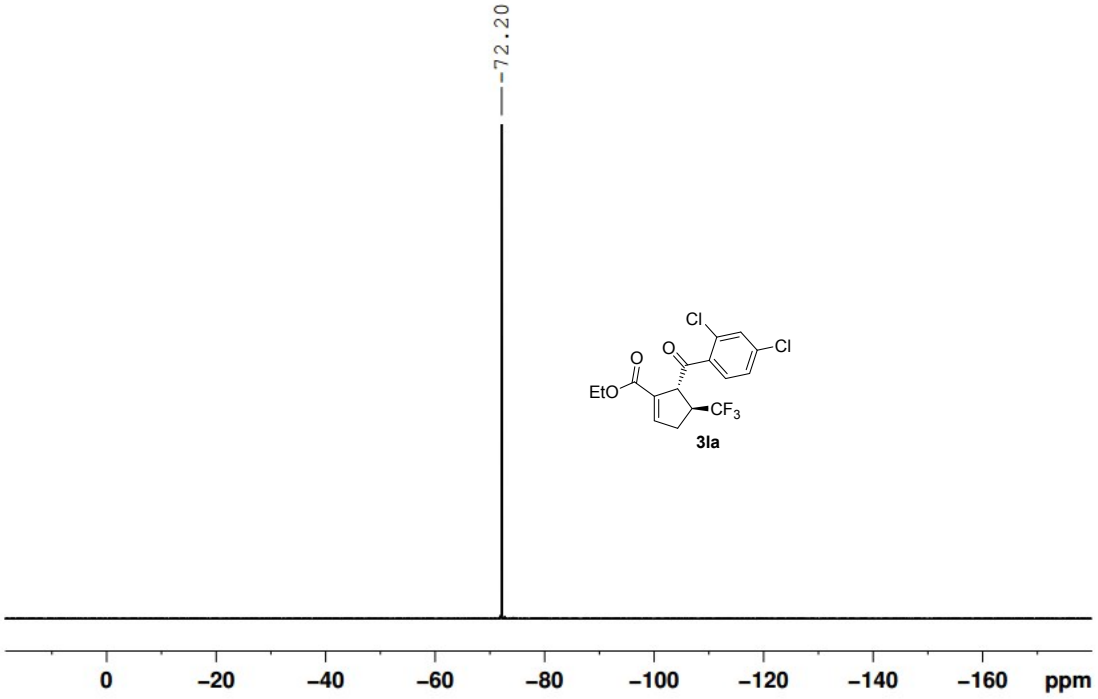
zhouw-6-58



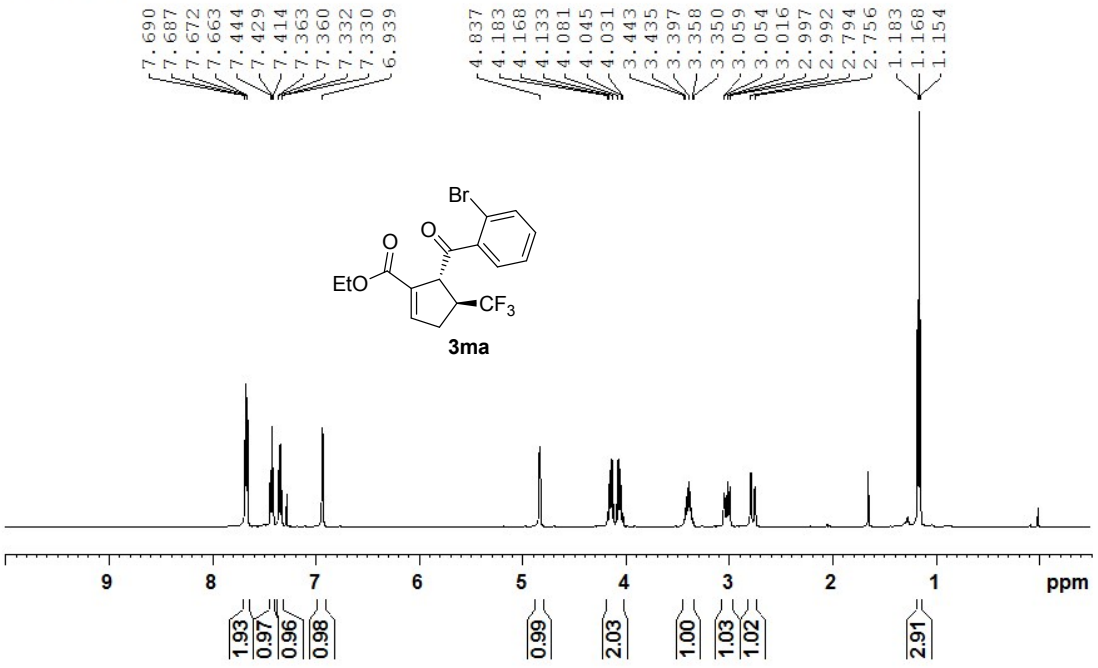
zhouw-6-58c



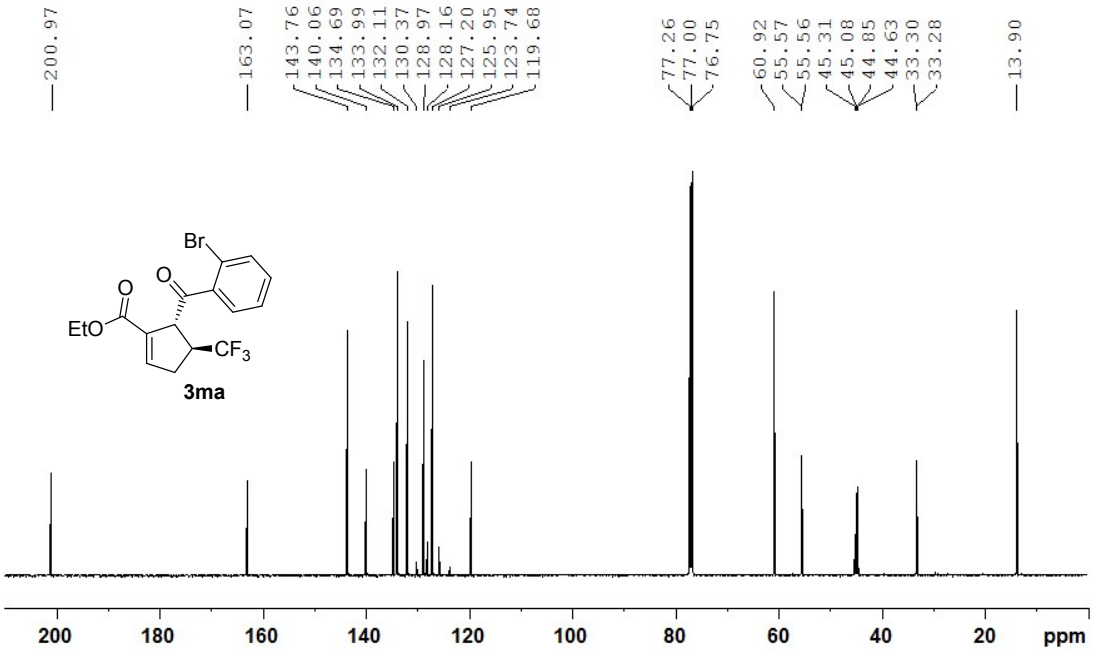
zhouw-6-58f



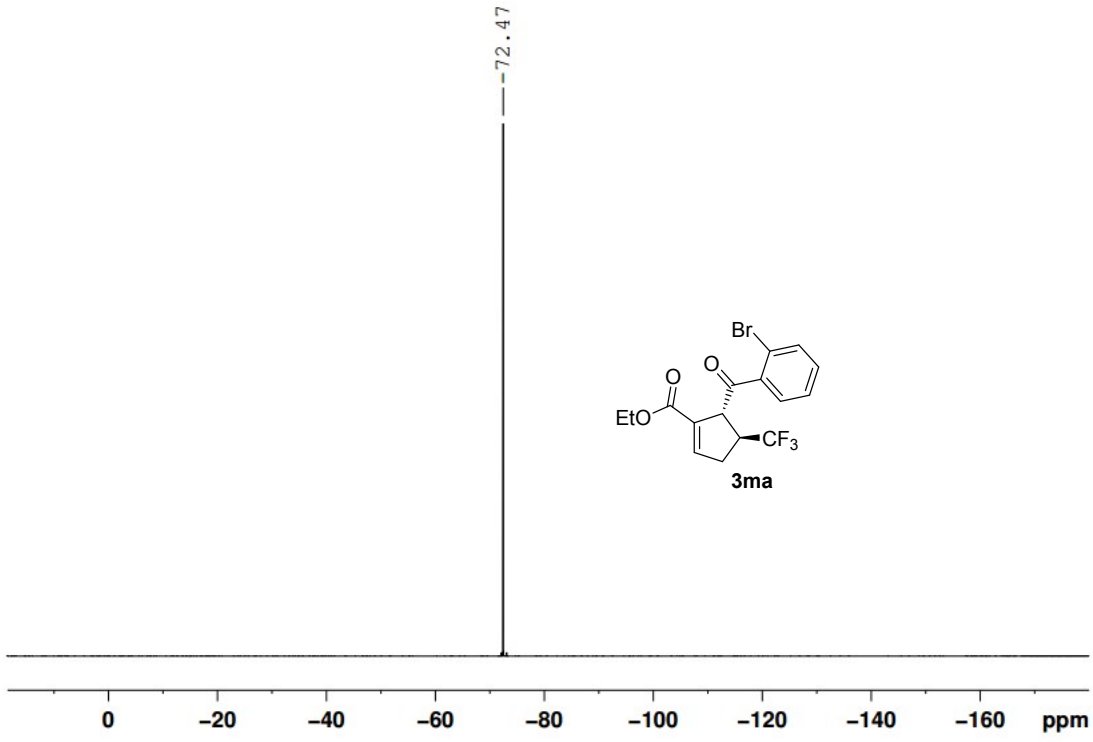
zhouw-6-59

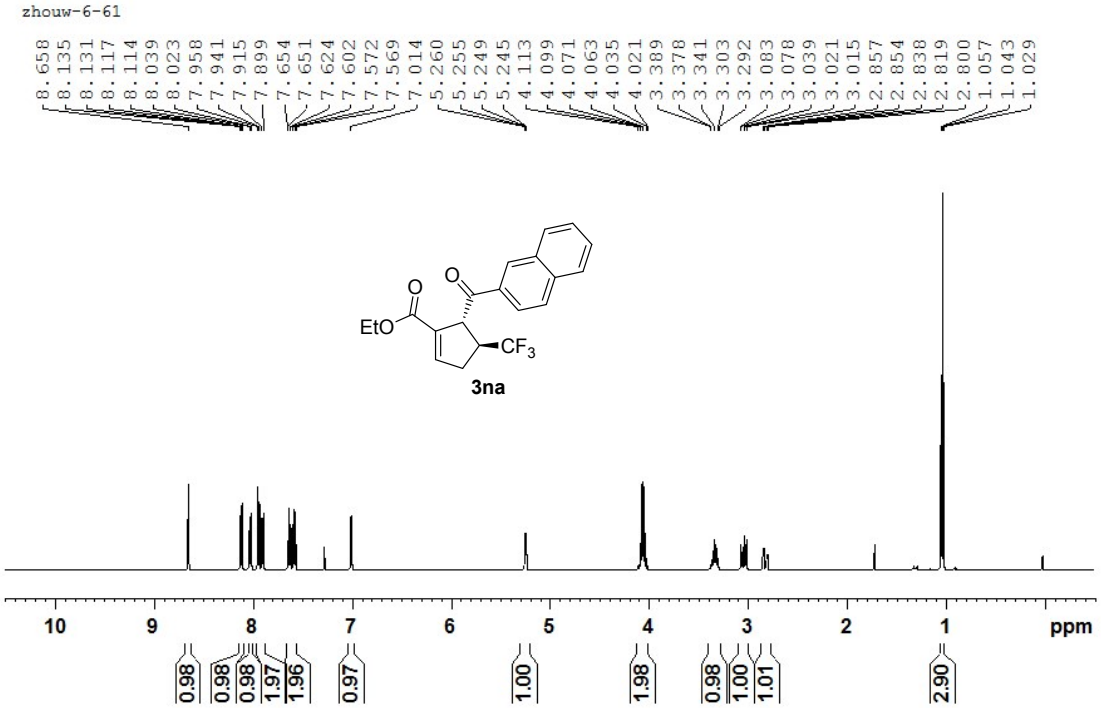


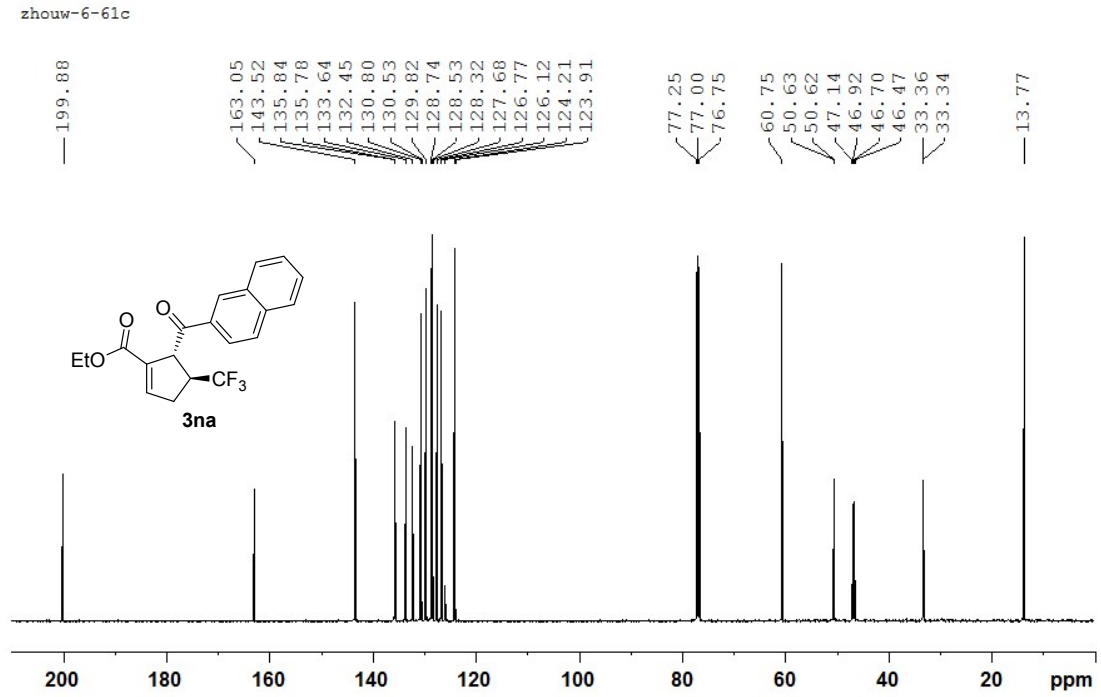
zhouw-6-59c



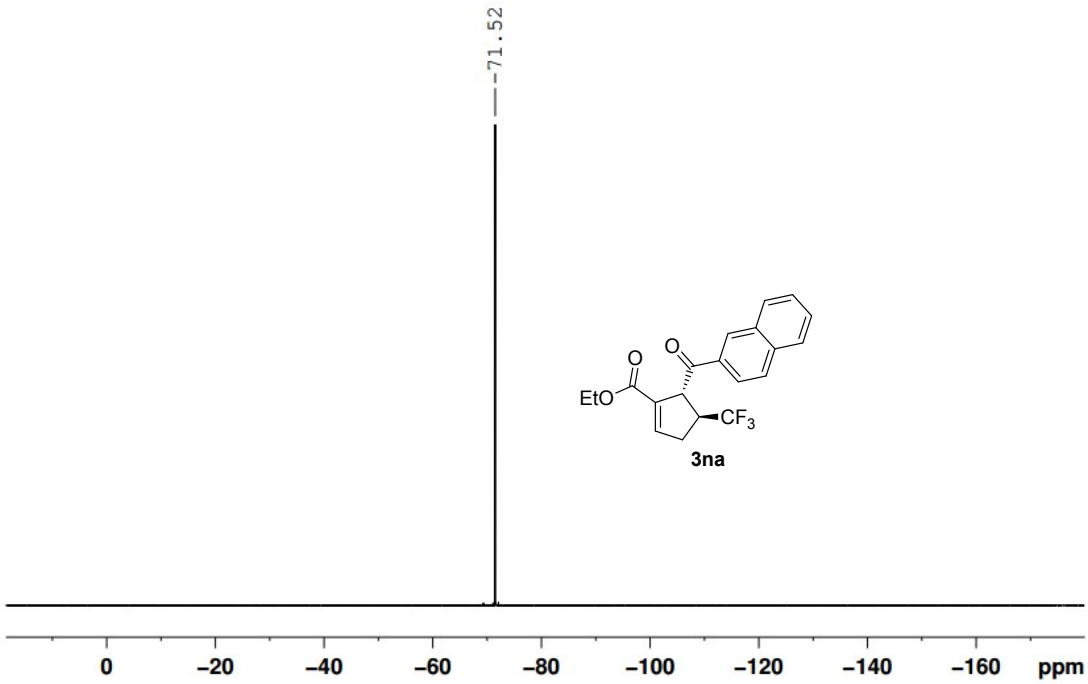
zhouw-6-59f



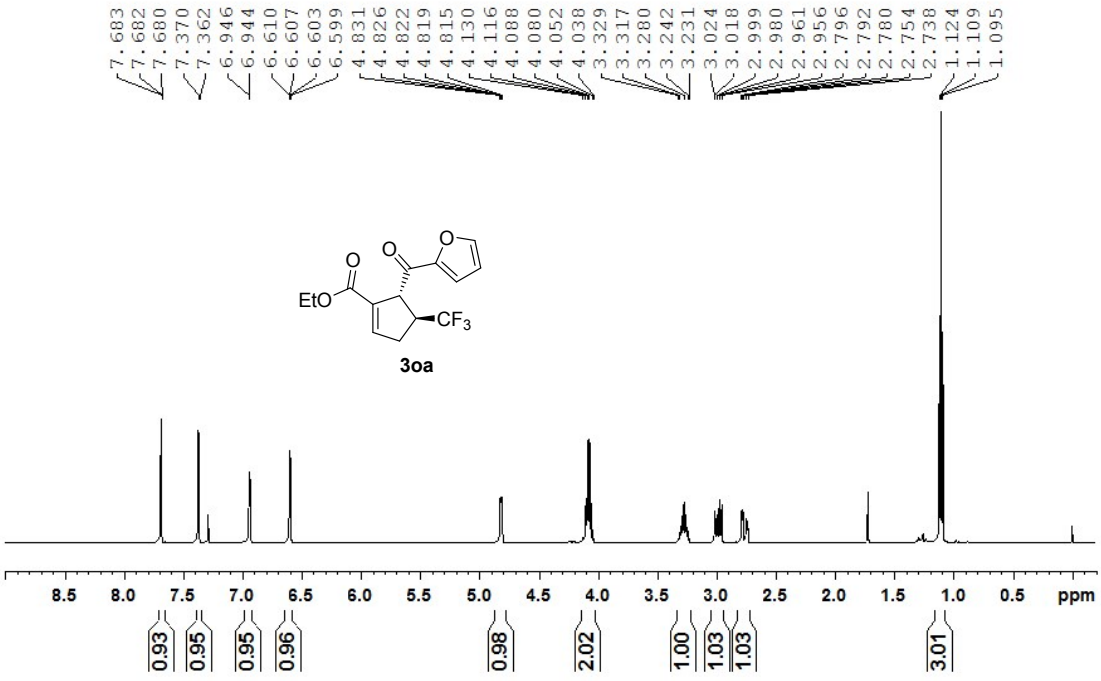

zhouw-6-61
8.658
8.135
8.131
8.117
8.114
8.039
8.023
7.958
7.941
7.915
7.899
7.654
7.651
7.624
7.602
7.572
7.569
7.014
5.260
5.255
5.249
5.245
4.113
4.099
4.071
4.063
4.035
4.021
3.389
3.378
3.341
3.303
3.292
3.083
3.078
3.039
3.021
3.015
2.857
2.854
2.838
2.819
2.800
1.057
1.043
1.029
O
O
EtO
CF3
3na
10
9
8
7
6
5
4
3
2
1
ppm
0.98
0.98
0.98
1.97
1.96
0.97
1.00
1.98
0.98
1.00
1.01
2.90

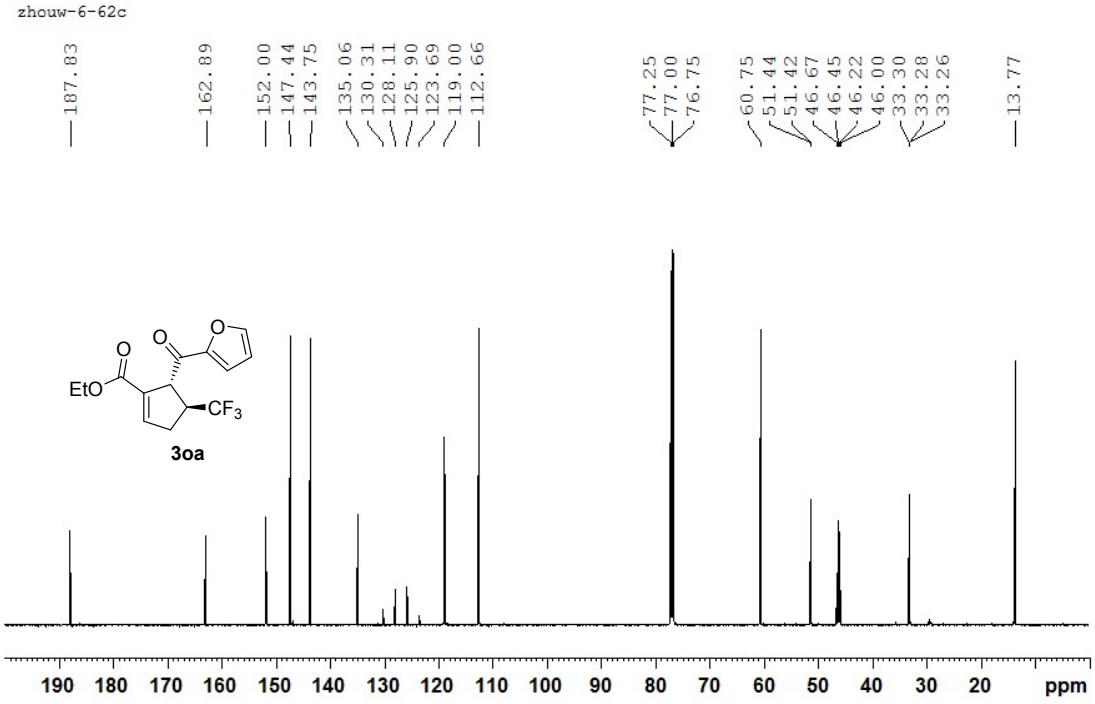

zhouw-6-61c
199.88
163.05
143.52
135.84
135.78
133.64
132.45
130.80
130.53
129.82
128.74
128.53
128.32
127.68
126.77
126.12
124.21
123.91
77.25
77.00
76.75
60.75
50.63
50.62
47.14
46.92
46.70
46.47
33.36
33.34
13.77
O
O
EtO
CF3
3na
200
180
160
140
120
100
80
60
40
20
ppm

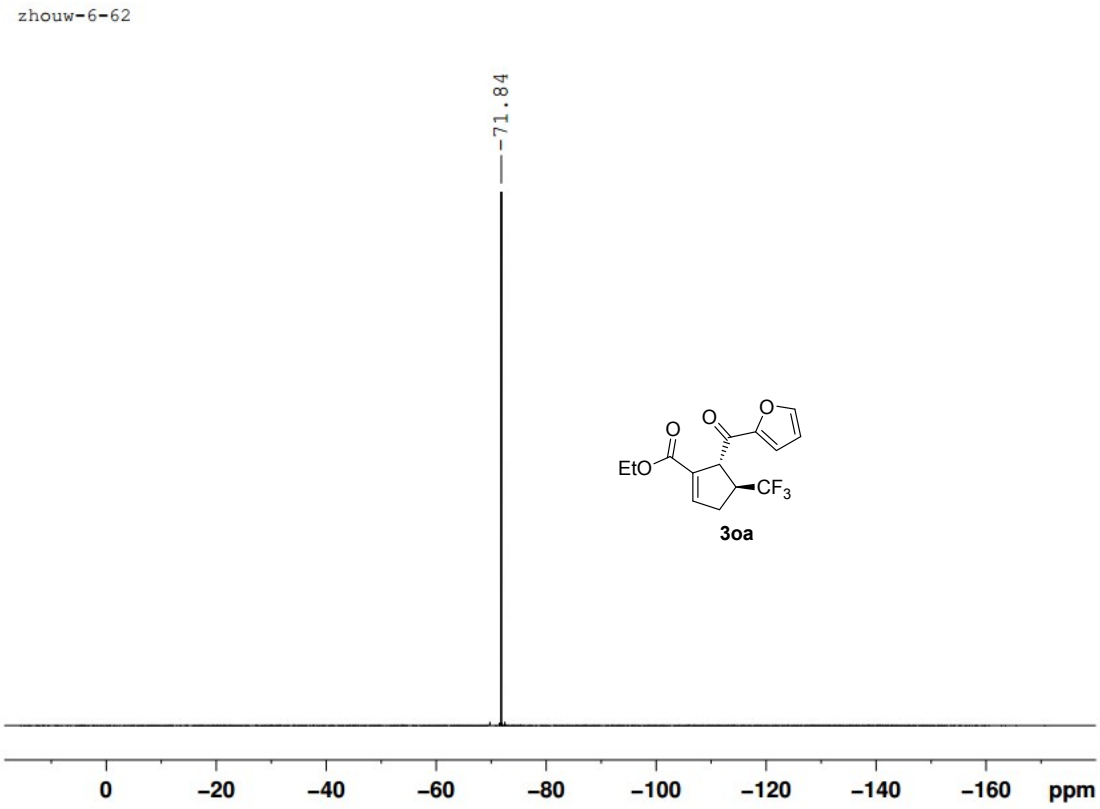
zhouw-6-61f



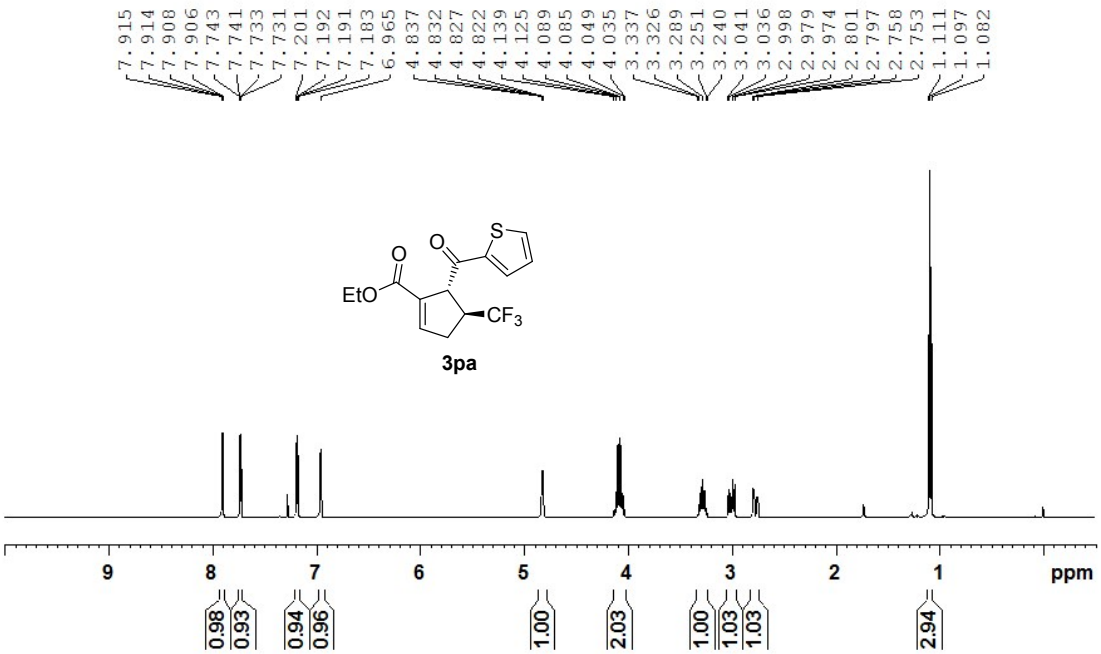
zhouw-6-62



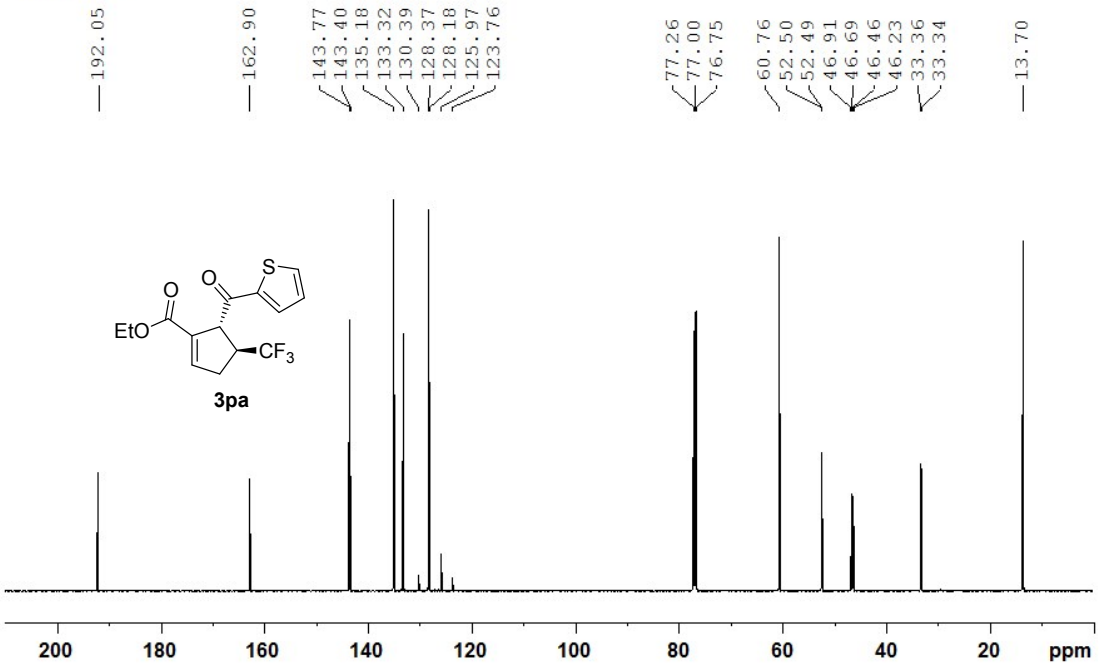

zhouw-6-62c
187.83
162.89
152.00
147.44
143.75
135.06
130.31
128.11
125.90
123.69
119.00
112.66
77.25
77.00
76.75
60.75
51.44
51.42
46.67
46.45
46.22
46.00
33.30
33.28
33.26
13.77
EtO
CF3
3oa
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


zhouw-6-62
-71.84
EtO
CF3
3oa
0 -20 -40 -60 -80 -100 -120 -140 -160 ppm

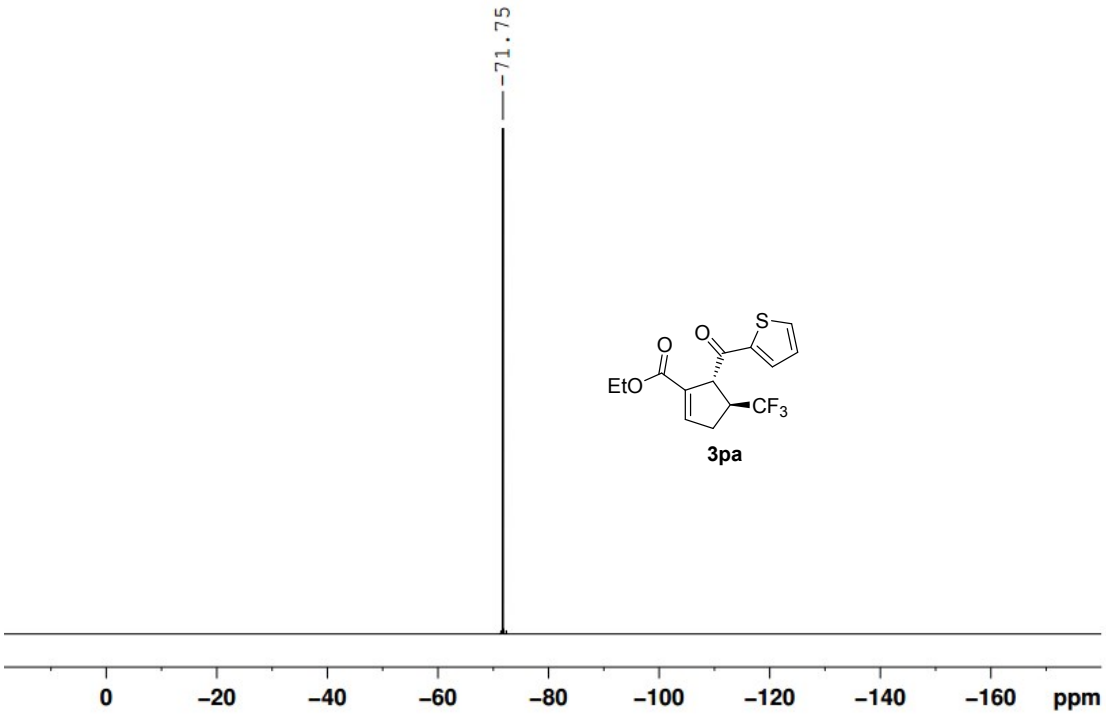
zhouw-6-63



zhouw-6-63c



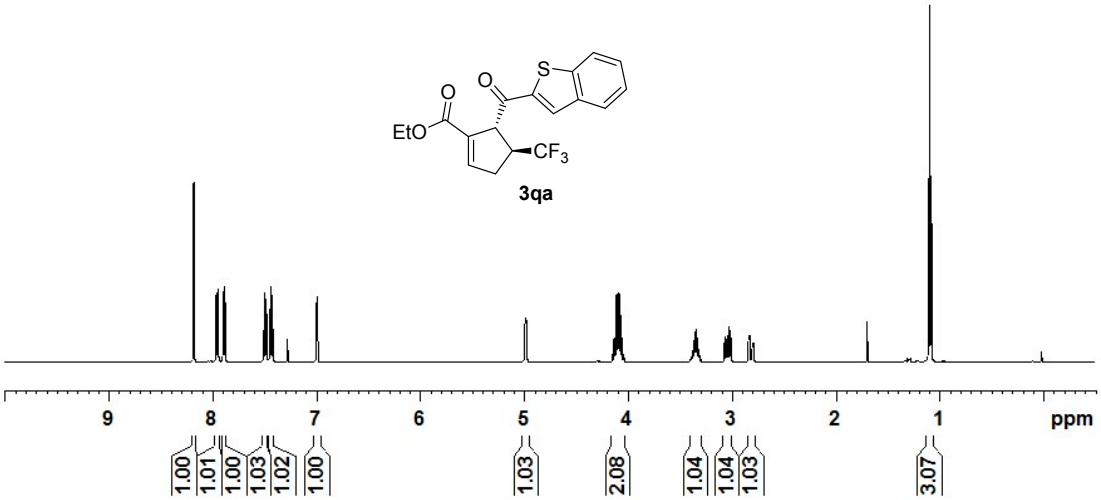
zhouw-6-63f



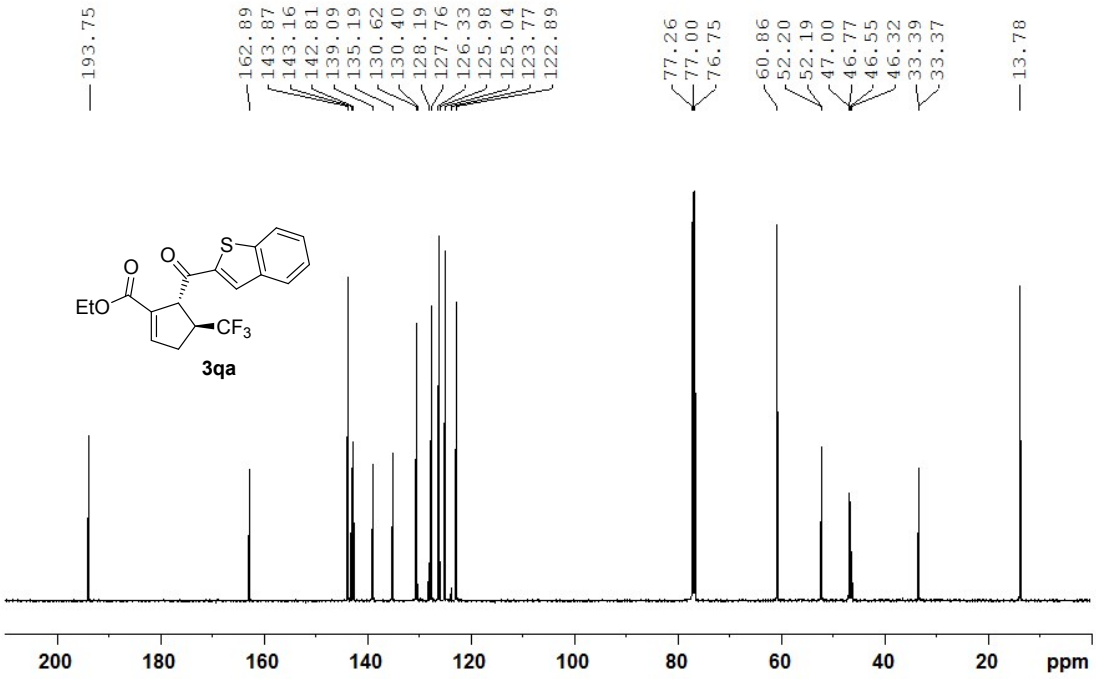
3pa

zhouw-6-64

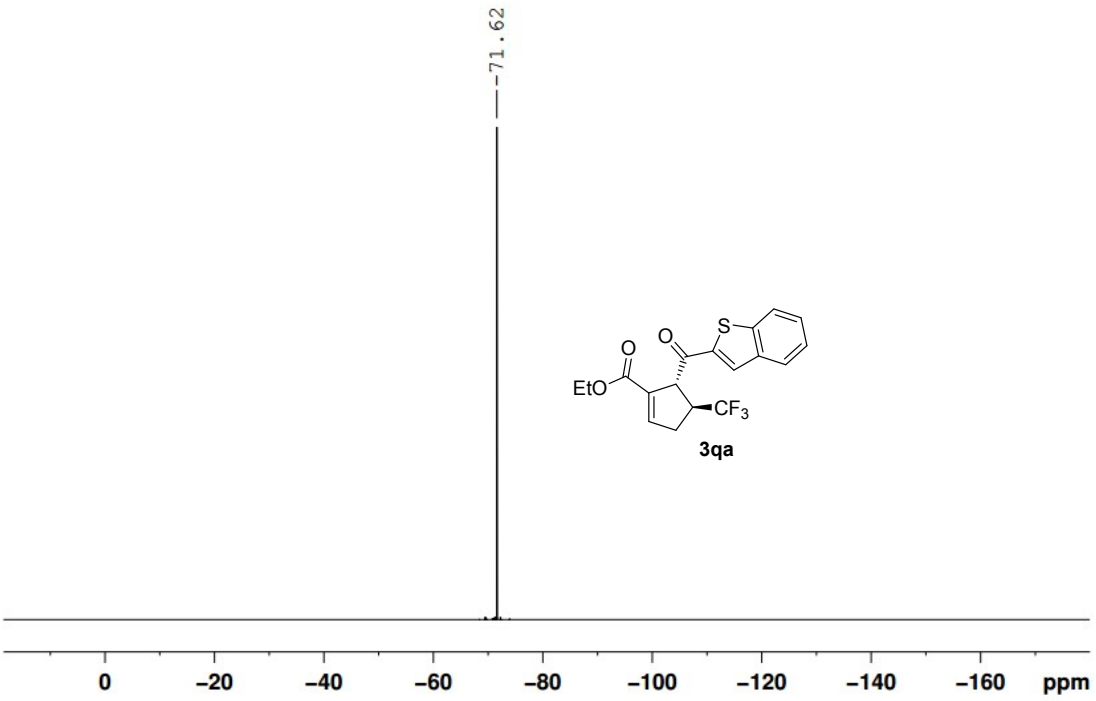
3qa

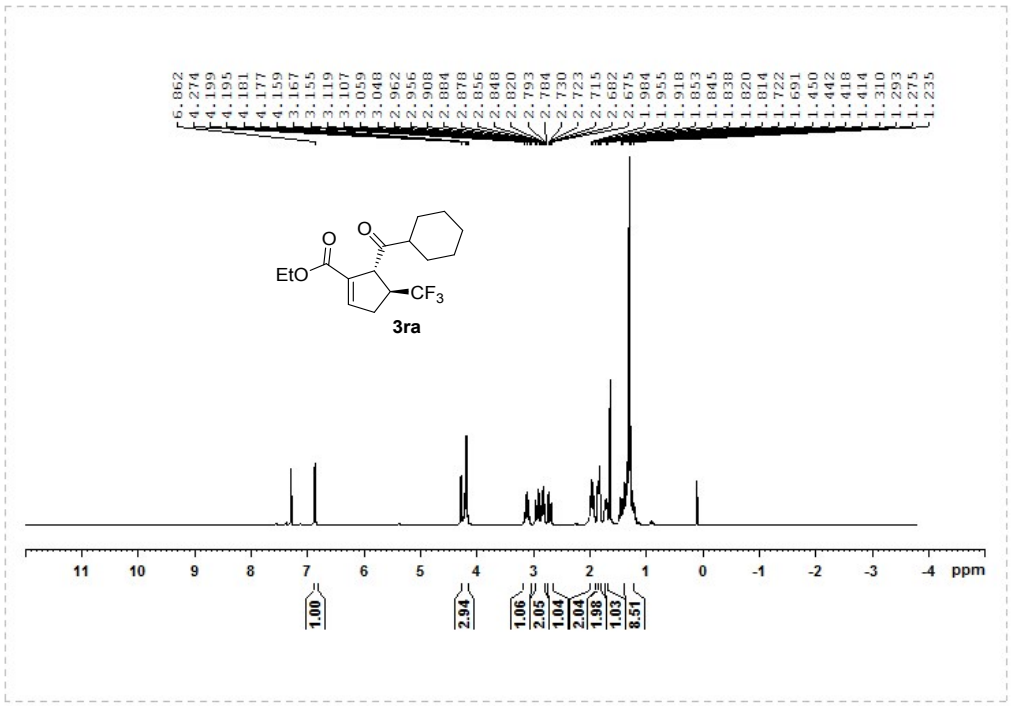


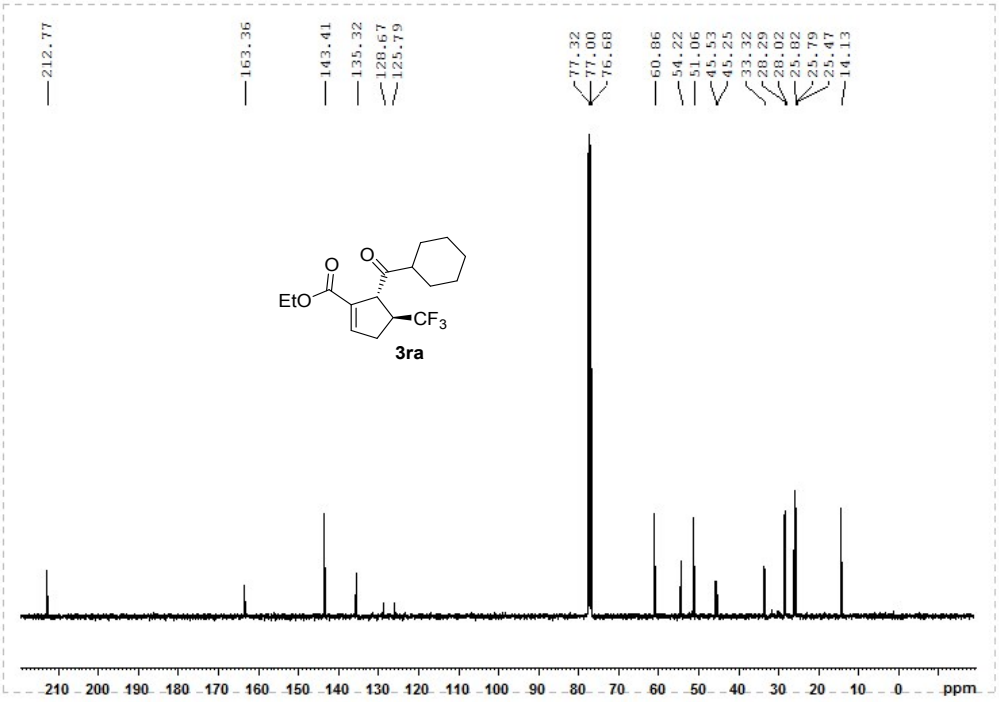
zhouw-6-64c

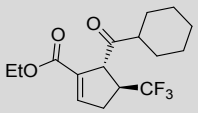


zhouw-6-64f

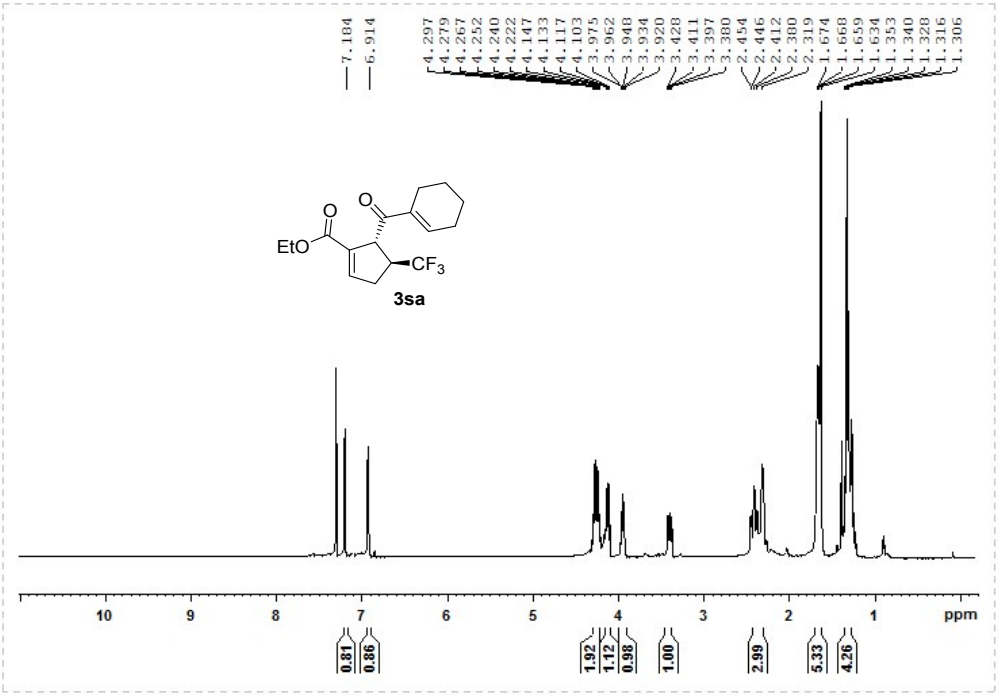



6.862
4.274
4.199
4.195
4.181
4.177
4.159
3.167
3.155
3.119
3.107
3.059
3.048
2.962
2.956
2.908
2.884
2.878
2.856
2.848
2.820
2.793
2.784
2.730
2.723
2.715
2.682
2.675
1.984
1.955
1.918
1.853
1.845
1.838
1.820
1.814
1.722
1.691
1.450
1.442
1.418
1.414
1.310
1.293
1.275
1.235
EtO
O
O
CF3
3ra
11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 ppm
1.00
2.94
1.06
2.05
1.04
2.04
1.98
1.03
8.51

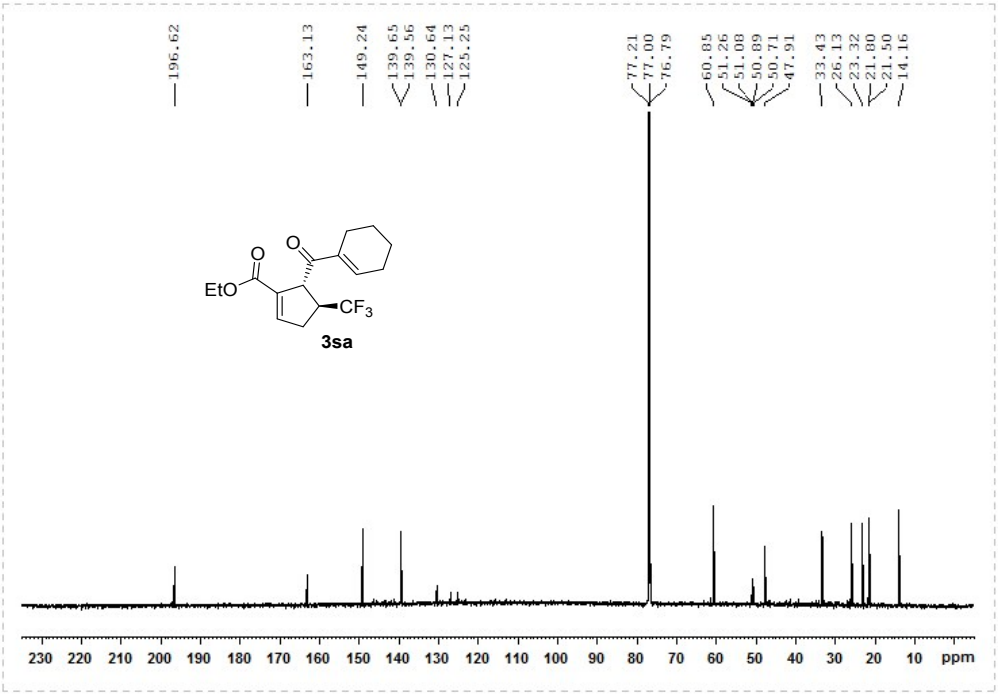

212.77
163.36
143.41
135.32
128.67
125.79
77.32
77.00
76.68
60.86
54.22
51.06
45.53
45.25
33.32
28.29
28.02
25.82
25.79
25.47
14.13
EtO
O
O
CF3
3ra
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

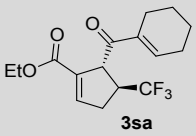

O
O
EtO
CF3

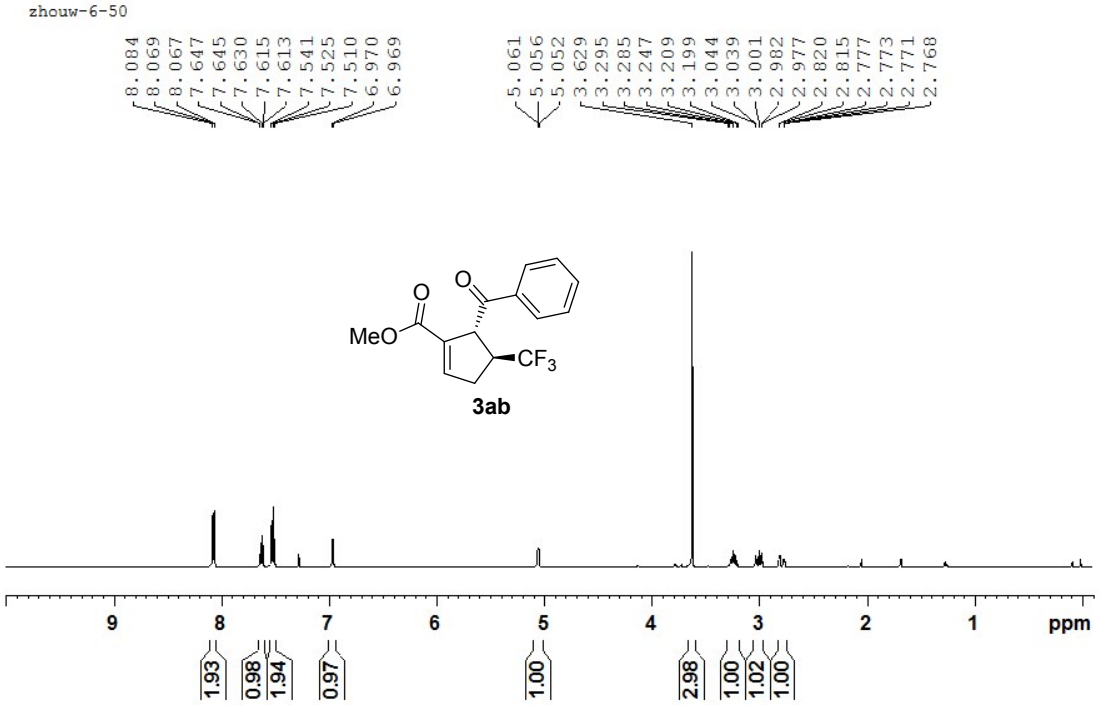
3ra

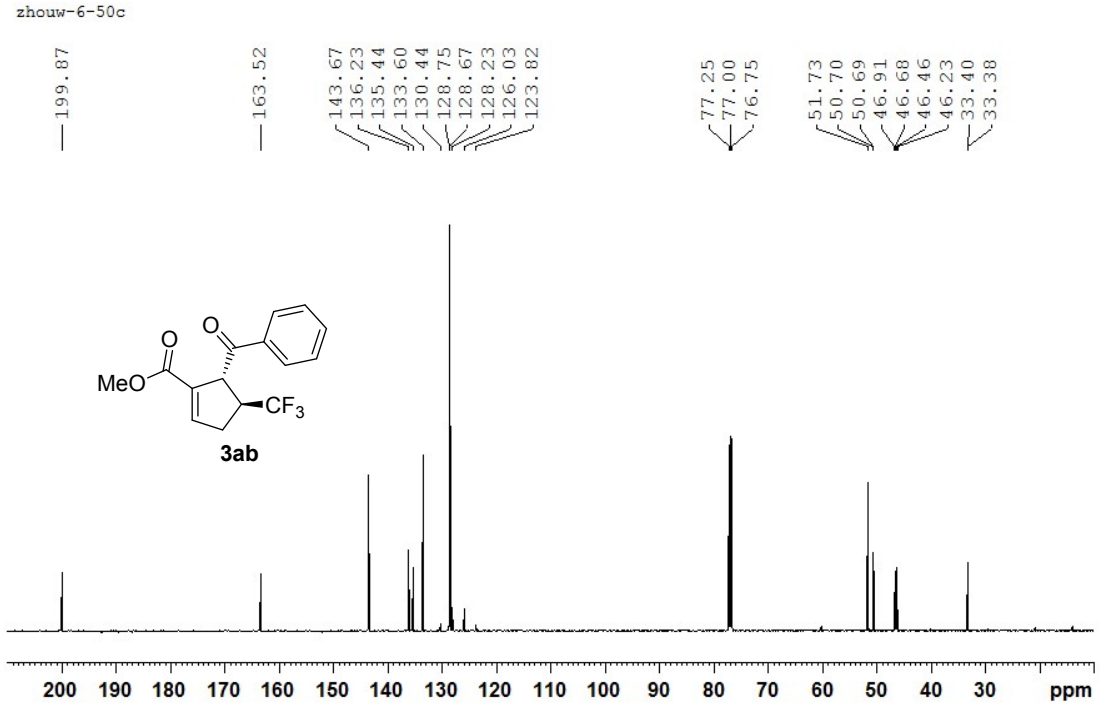

7.184
6.914
4.297
4.279
4.267
4.252
4.240
4.222
4.147
4.133
4.117
4.103
3.975
3.962
3.948
3.934
3.920
3.428
3.411
3.397
3.380
2.454
2.446
2.412
2.380
2.319
1.674
1.668
1.659
1.634
1.353
1.340
1.328
1.316
1.306
O
O
EtO
CF3
10
9
8
7
6
5
4
3
2
1
ppm
0.81
0.86
1.92
1.12
0.98
1.00
2.99
5.33
4.26

3sa

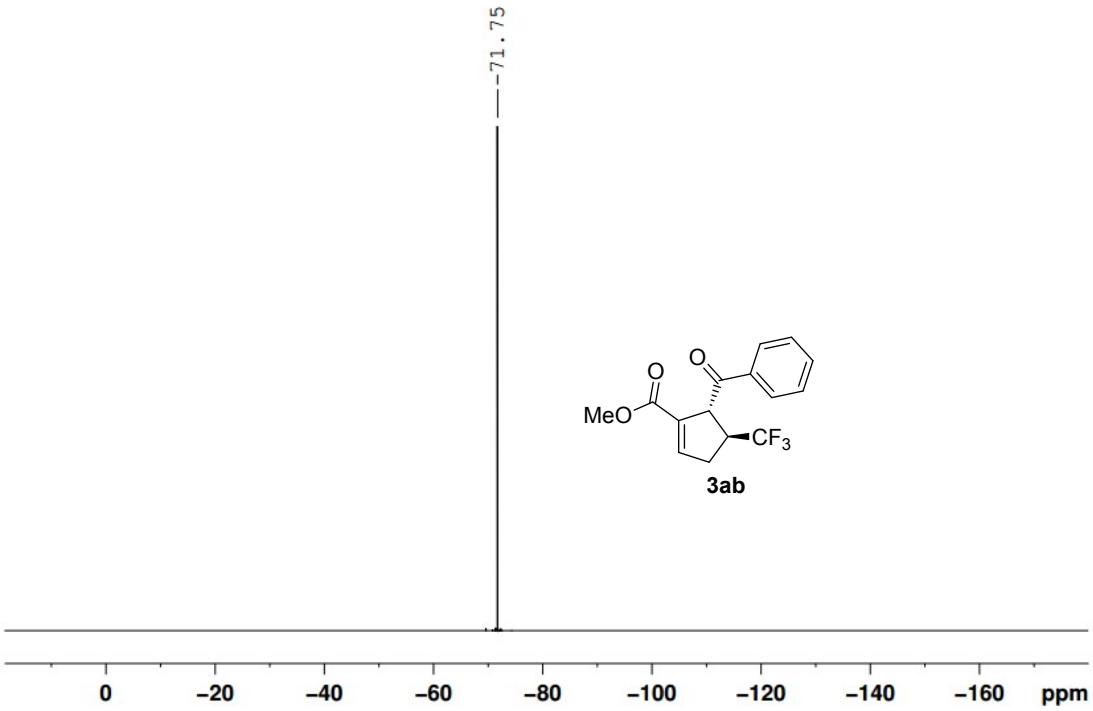

196.62
163.13
149.24
139.65
139.56
130.64
127.13
125.25
77.21
77.00
76.79
60.85
51.26
51.08
50.89
50.71
47.91
33.43
26.13
23.32
21.80
21.50
14.16
O
O
EtO
CF3
3sa
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


O
O
EtO
CF3
3sa

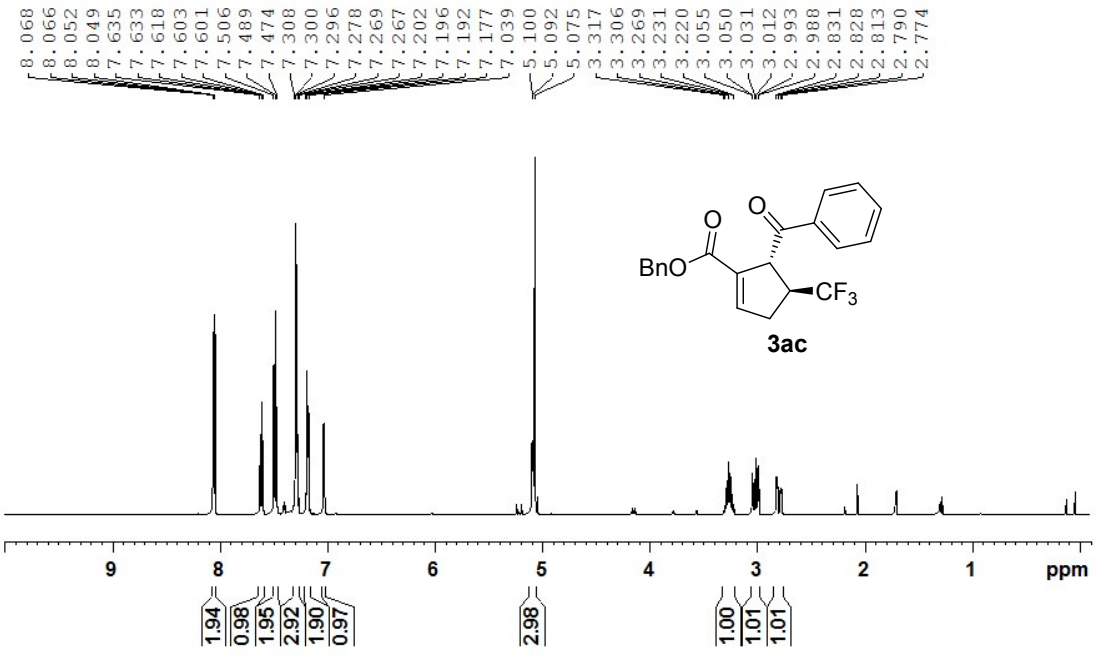

zhouw-6-50
8.084
8.069
8.067
7.647
7.645
7.630
7.615
7.613
7.541
7.525
7.510
6.970
6.969
5.061
5.056
5.052
3.629
3.295
3.285
3.247
3.209
3.199
3.044
3.039
3.001
2.982
2.977
2.820
2.815
2.777
2.773
2.771
2.768
MeO
CF3
3ab
9
8
7
6
5
4
3
2
1
ppm
1.93
0.98
1.94
0.97
1.00
2.98
1.00
1.02
1.00


zhouw-6-50c
199.87
163.52
143.67
136.23
135.44
133.60
130.44
128.75
128.67
128.23
126.03
123.82
77.25
77.00
76.75
51.73
50.70
50.69
46.91
46.68
46.46
46.23
33.40
33.38
MeO
CF3
3ab
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

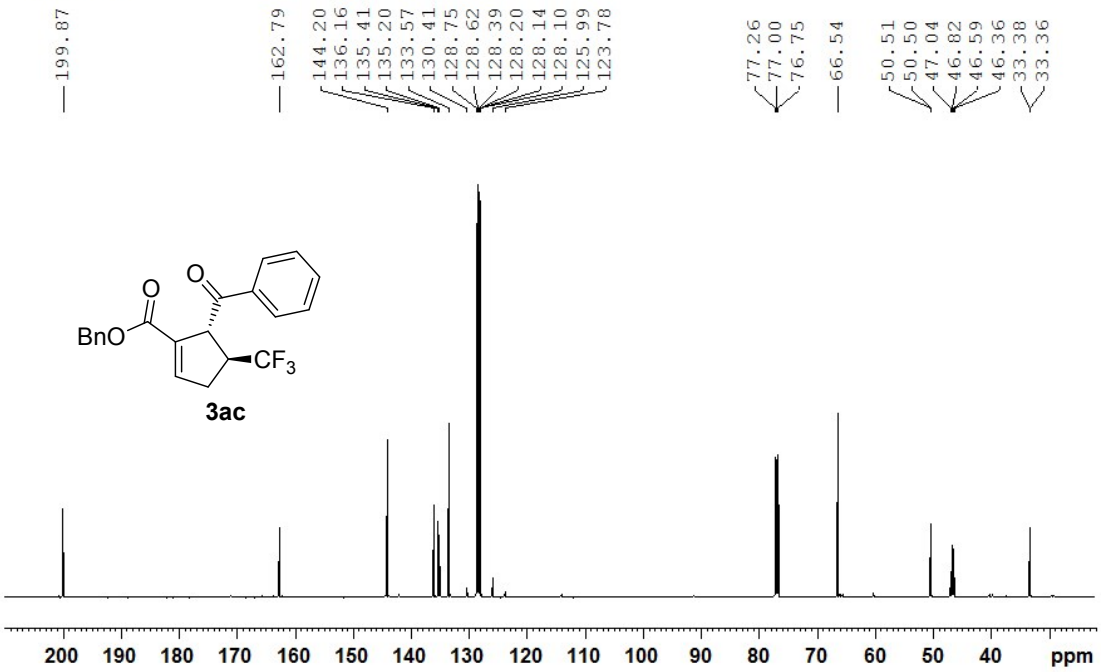
zhouw-6-50f



zhouw-6-51

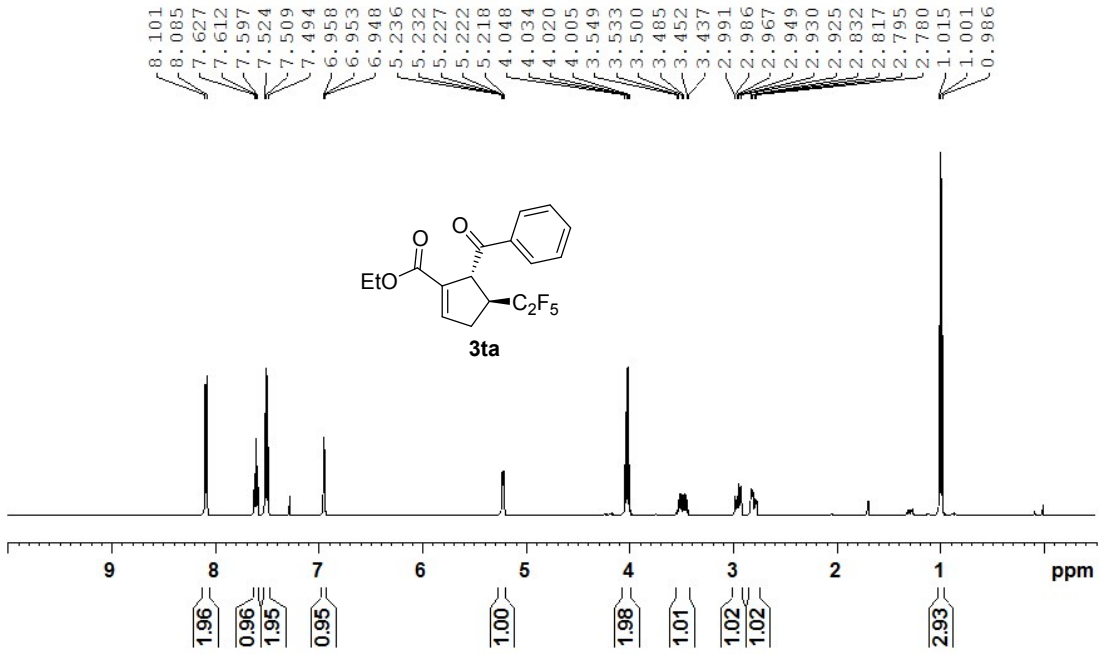


zhouw-6-51c

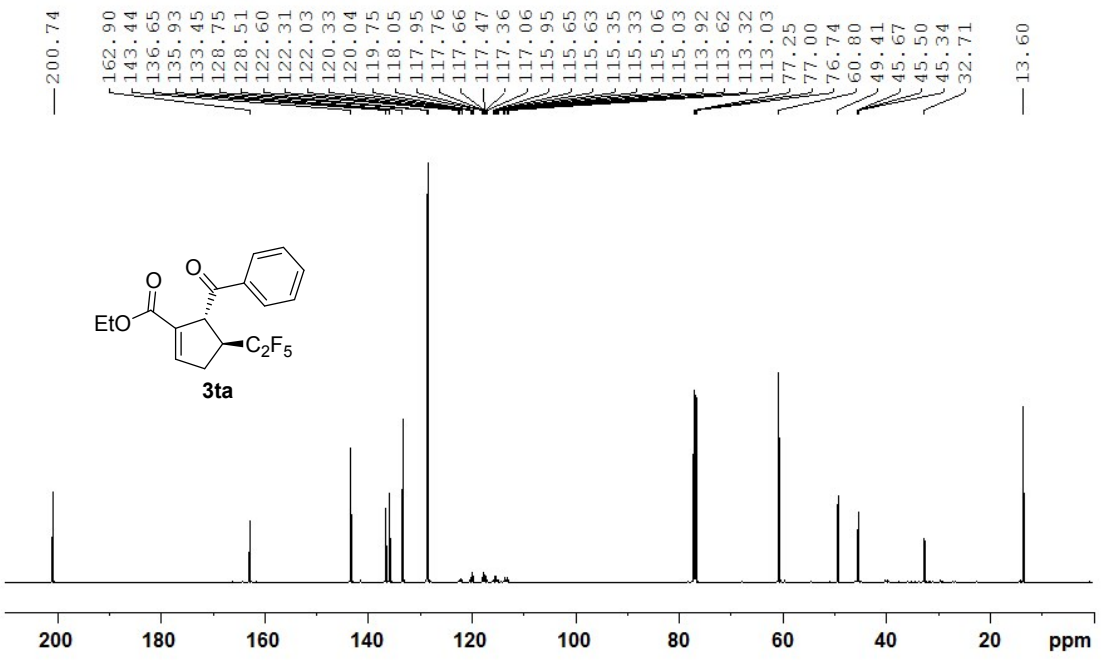


zhouw-6-51f

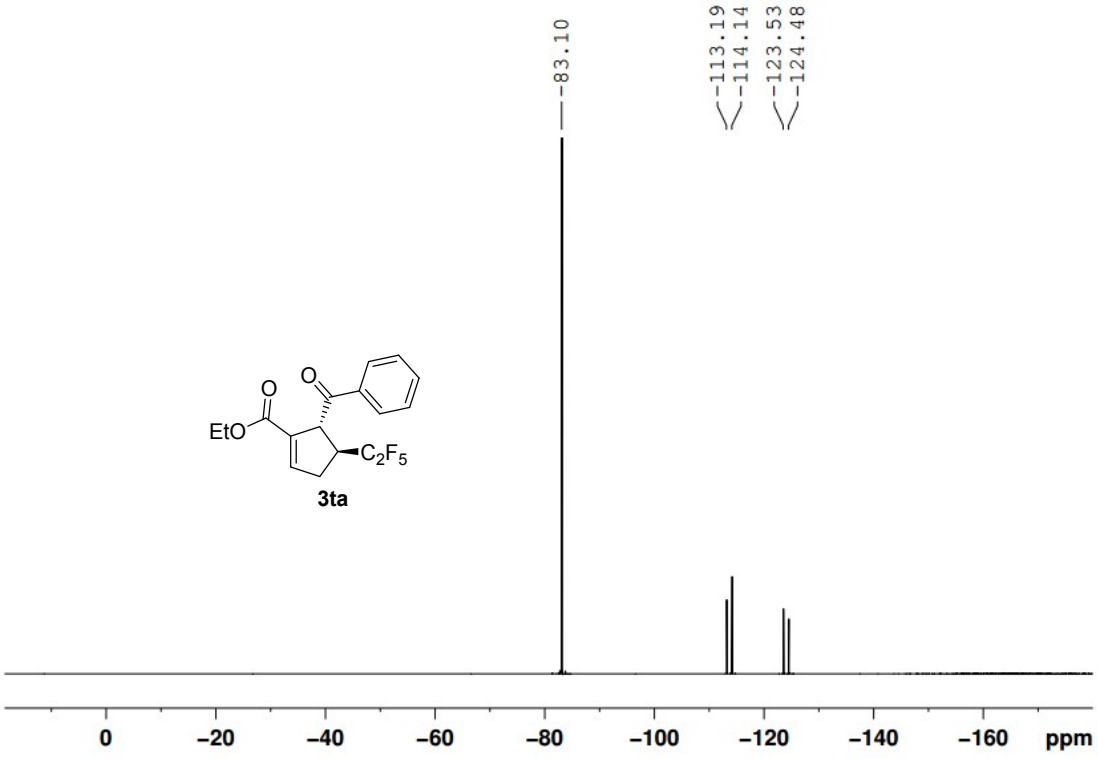
zhouw-6-65ss



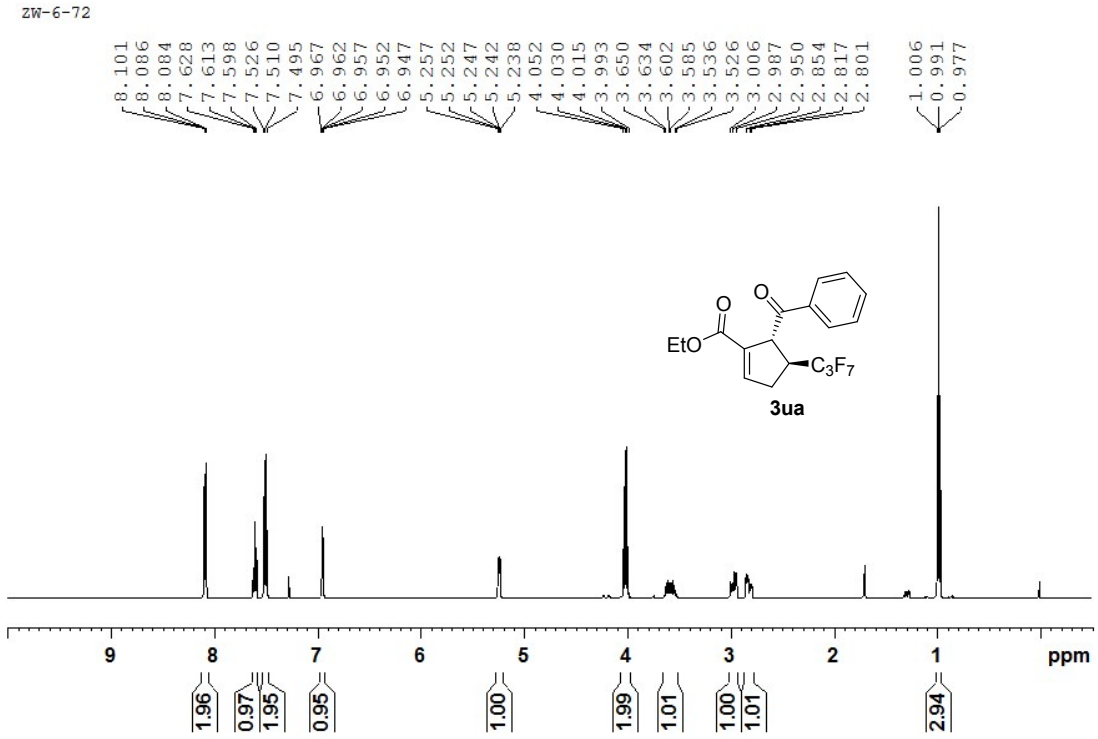
zhouw-6-65cs

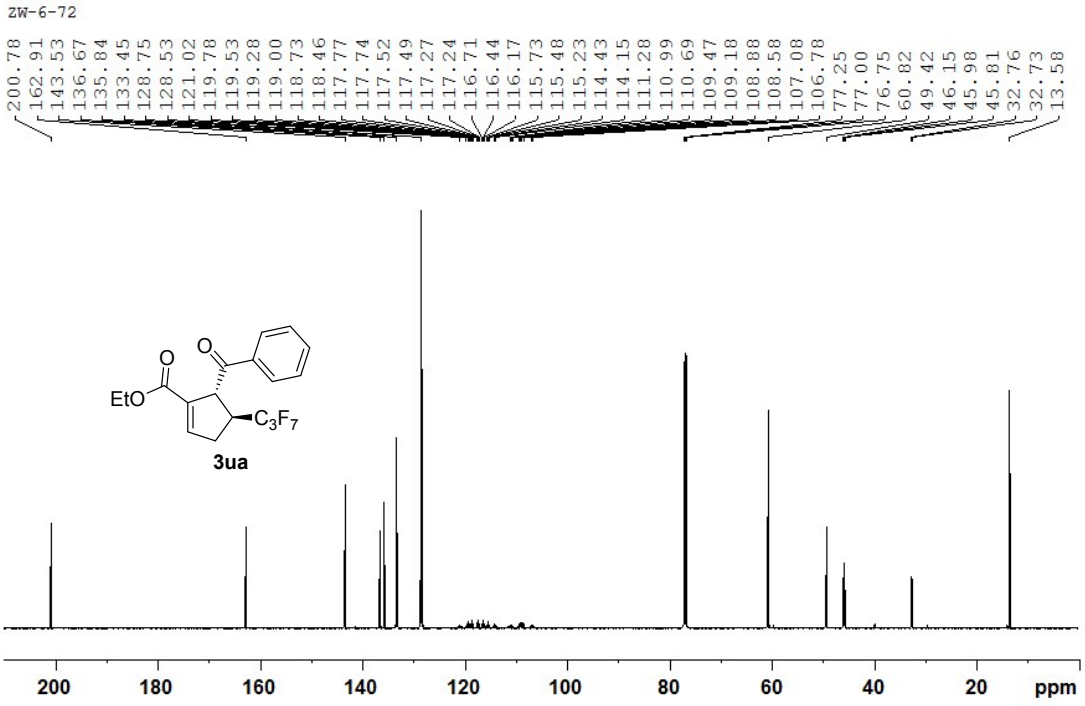


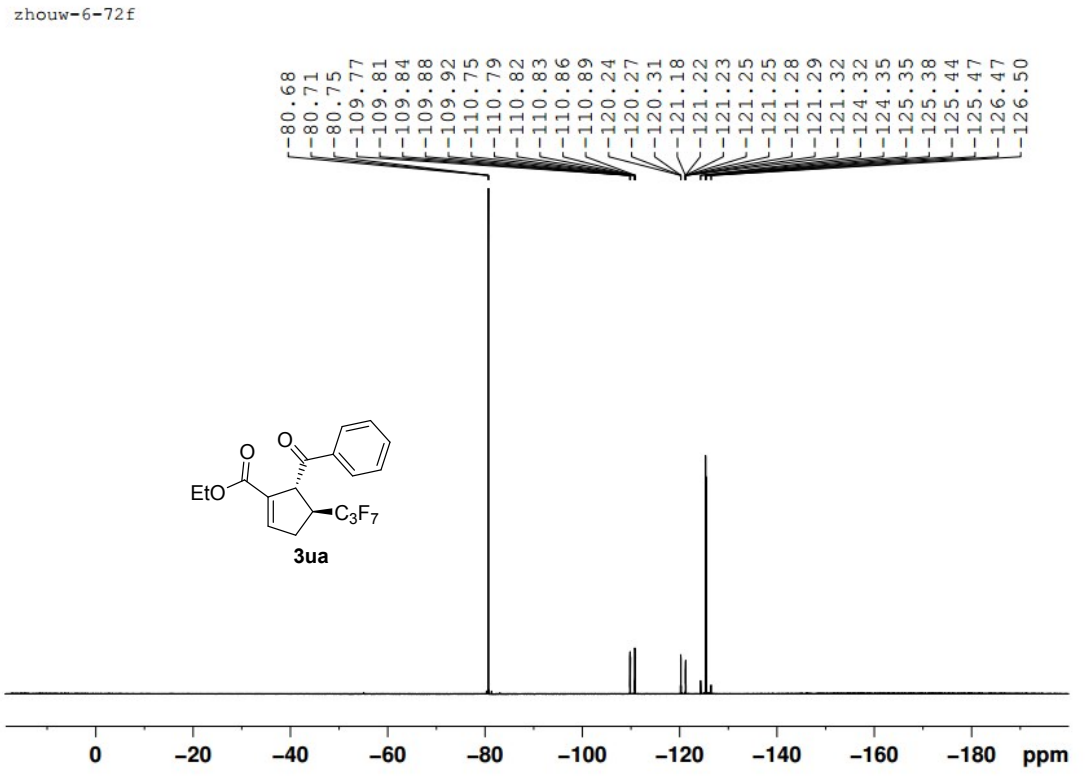
zhouw-6-65f

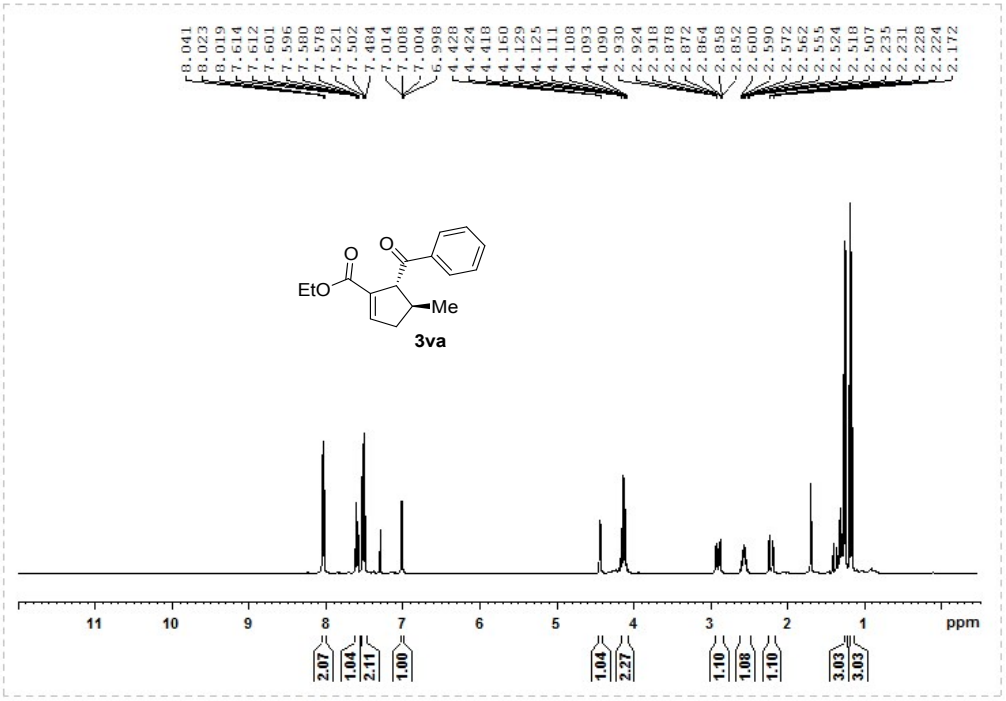


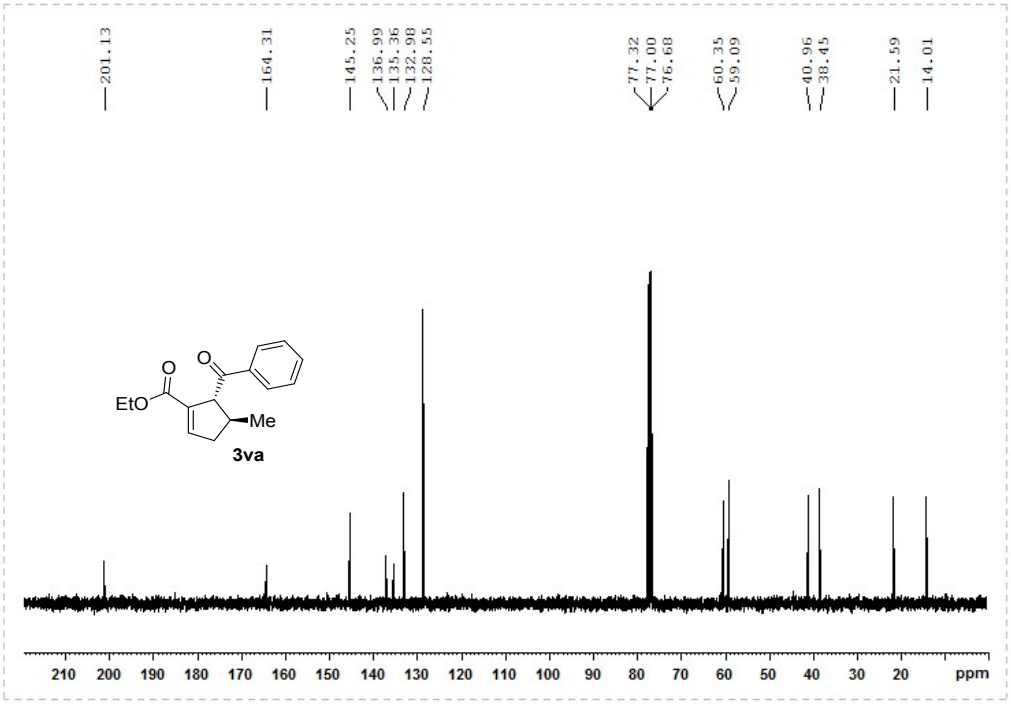
ZW-6-72

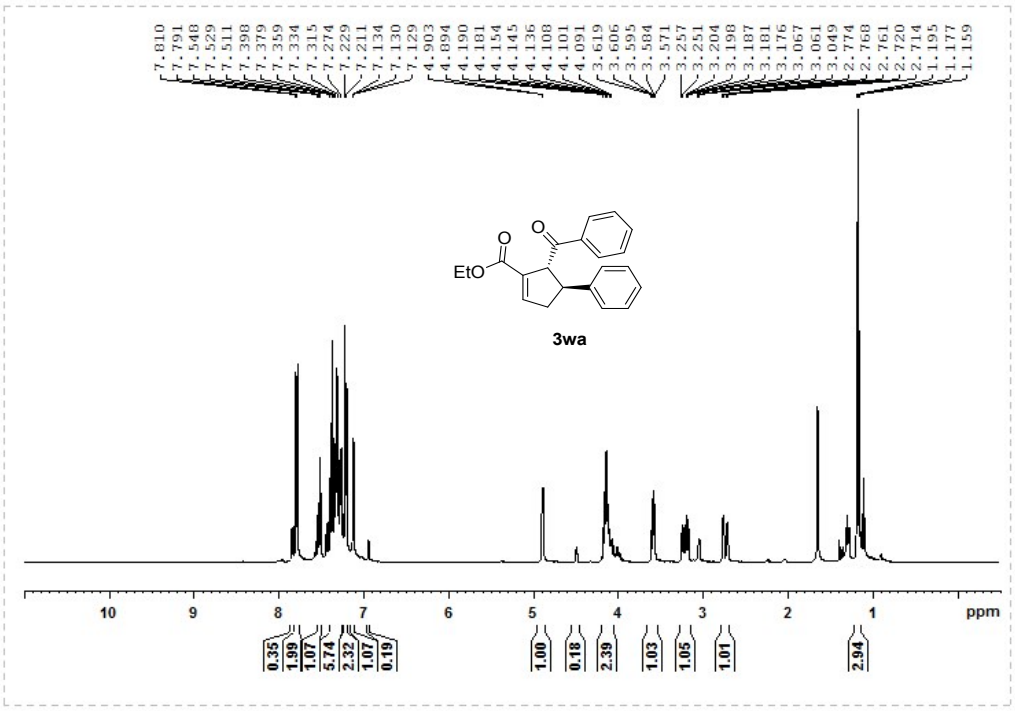


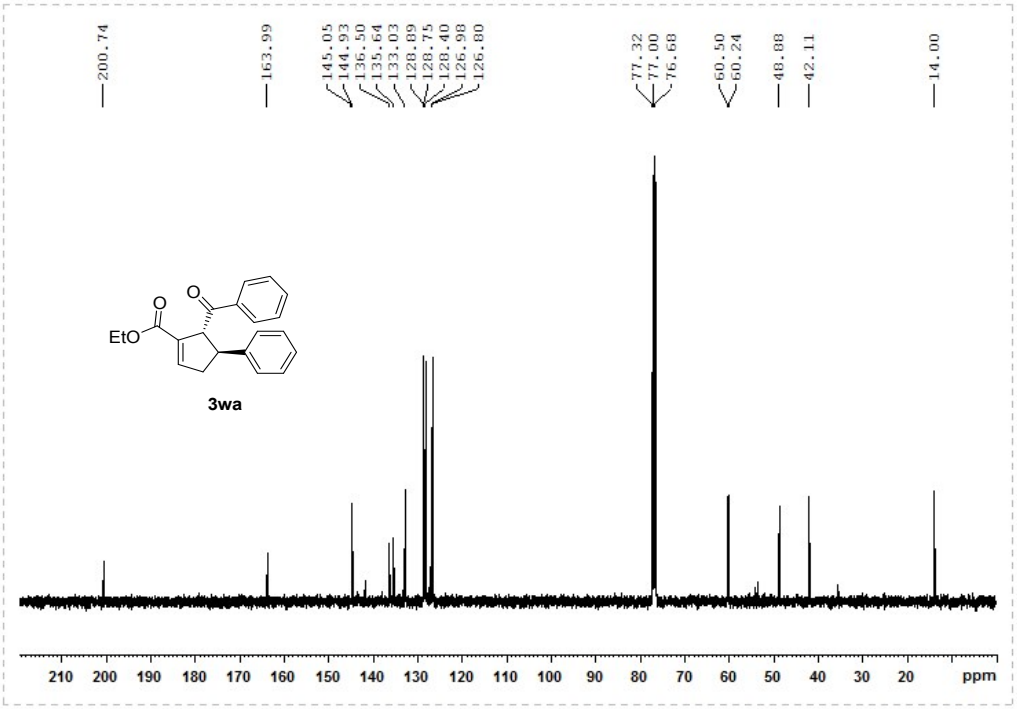

zw-6-72
200.78
162.91
143.53
136.67
135.84
133.45
128.75
128.53
121.02
119.78
119.53
119.28
119.00
118.73
118.46
117.77
117.74
117.52
117.49
117.27
117.24
116.71
116.44
116.17
115.73
115.48
115.23
114.43
114.15
111.28
110.99
110.69
109.47
109.18
108.88
108.58
107.08
106.78
77.25
77.00
76.75
60.82
49.42
46.15
45.98
45.81
32.76
32.73
13.58
EtO
O
O
C_3F_7
3ua
200
180
160
140
120
100
80
60
40
20
ppm

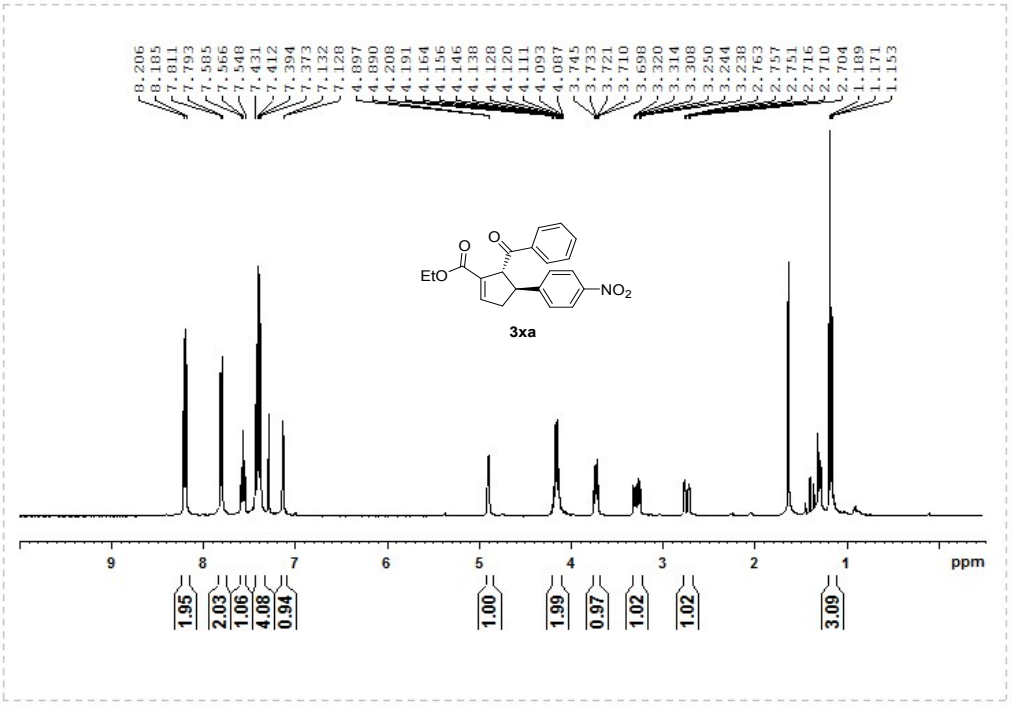

zhouw-6-72f
80.68
80.71
80.75
109.77
109.81
109.84
109.88
109.92
110.75
110.79
110.82
110.83
110.86
110.89
120.24
120.27
120.31
121.18
121.22
121.23
121.25
121.25
121.28
121.29
121.32
124.32
124.35
125.35
125.38
125.44
125.47
126.47
126.50
EtO
O
O
C_3F_7
3ua
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

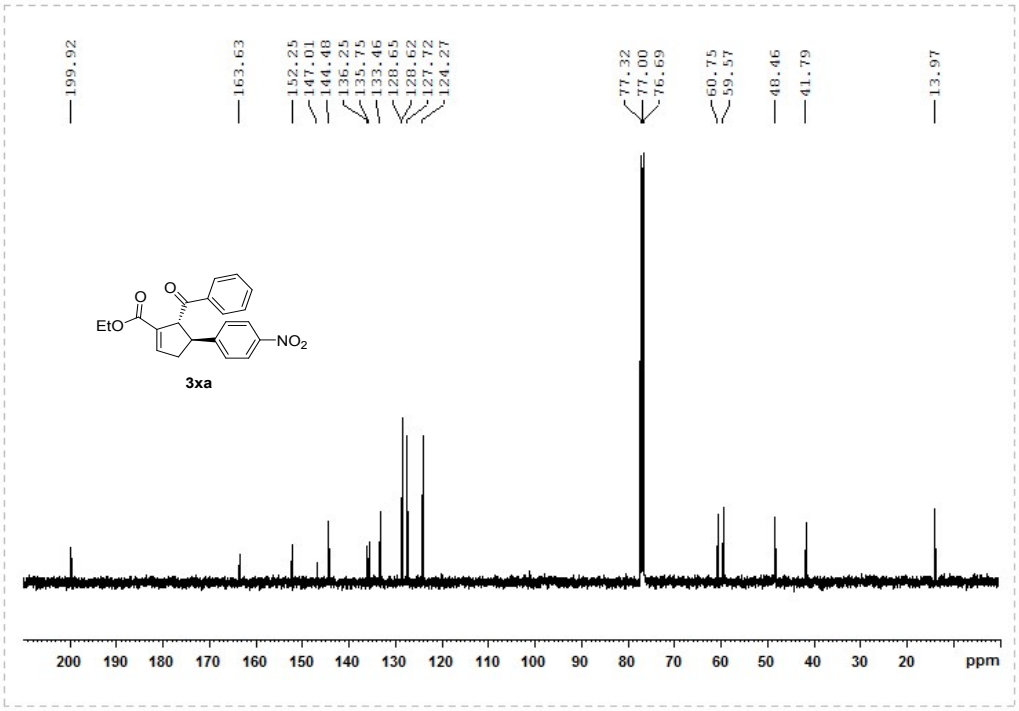

8.041
8.023
8.019
7.614
7.612
7.601
7.596
7.580
7.578
7.521
7.502
7.484
7.014
7.008
7.004
6.998
4.428
4.424
4.418
4.160
4.129
4.125
4.111
4.108
4.093
4.090
2.930
2.924
2.918
2.878
2.872
2.864
2.858
2.852
2.600
2.590
2.572
2.562
2.555
2.524
2.518
2.507
2.235
2.231
2.228
2.224
2.172
O
O
EtO
Me
3va
11
10
9
8
7
6
5
4
3
2
1
ppm
2.07
1.04
2.11
1.00
1.04
2.27
1.10
1.08
1.10
3.03
3.03


201.13
164.31
145.25
136.99
135.36
132.98
128.55
77.32
77.00
76.68
60.35
59.09
40.96
38.45
21.59
14.01
O
O
EtO
Me
3va
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

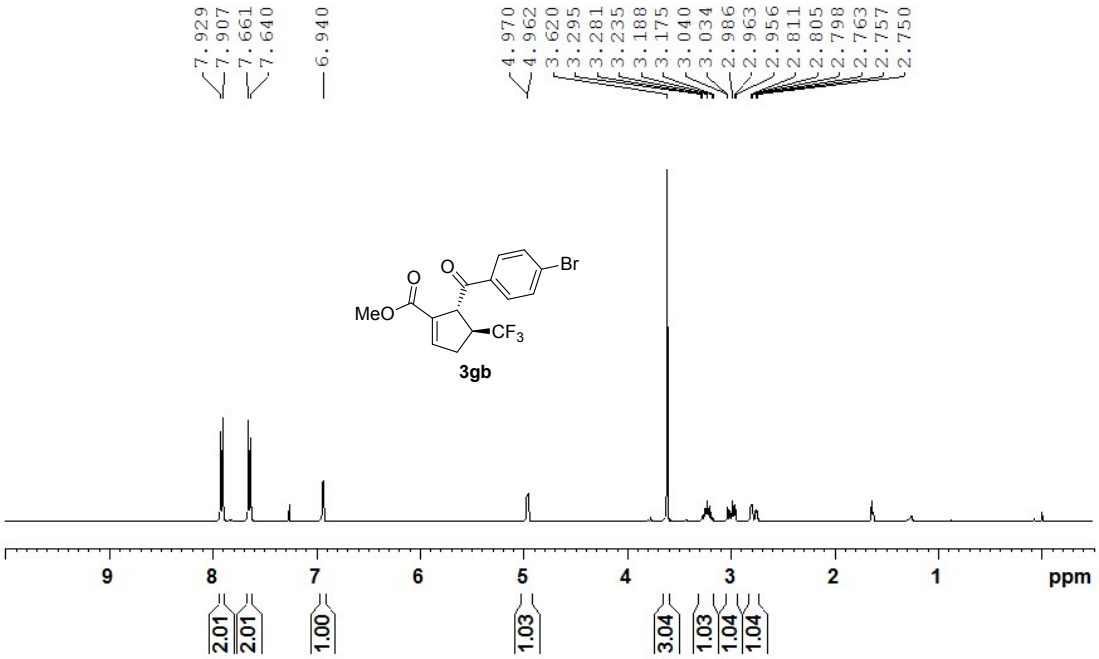

7.810
7.791
7.548
7.529
7.511
7.398
7.379
7.359
7.334
7.315
7.274
7.229
7.211
7.134
7.130
7.129
4.903
4.894
4.190
4.181
4.154
4.145
4.136
4.108
4.101
4.091
3.619
3.606
3.595
3.584
3.571
3.257
3.251
3.204
3.198
3.187
3.181
3.176
3.067
3.061
3.049
2.774
2.768
2.761
2.720
2.714
1.195
1.177
1.159
O
O
EtO
3wa
10
9
8
7
6
5
4
3
2
1
ppm
0.35
1.99
1.07
5.74
2.32
1.07
0.19
1.00
0.18
2.39
1.03
1.05
1.01
2.94


200.74
163.99
145.05
144.93
136.50
135.64
133.03
128.89
128.75
128.40
126.98
126.80
77.32
77.00
76.68
60.50
60.24
48.88
42.11
14.00
O
O
EtO
3wa
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

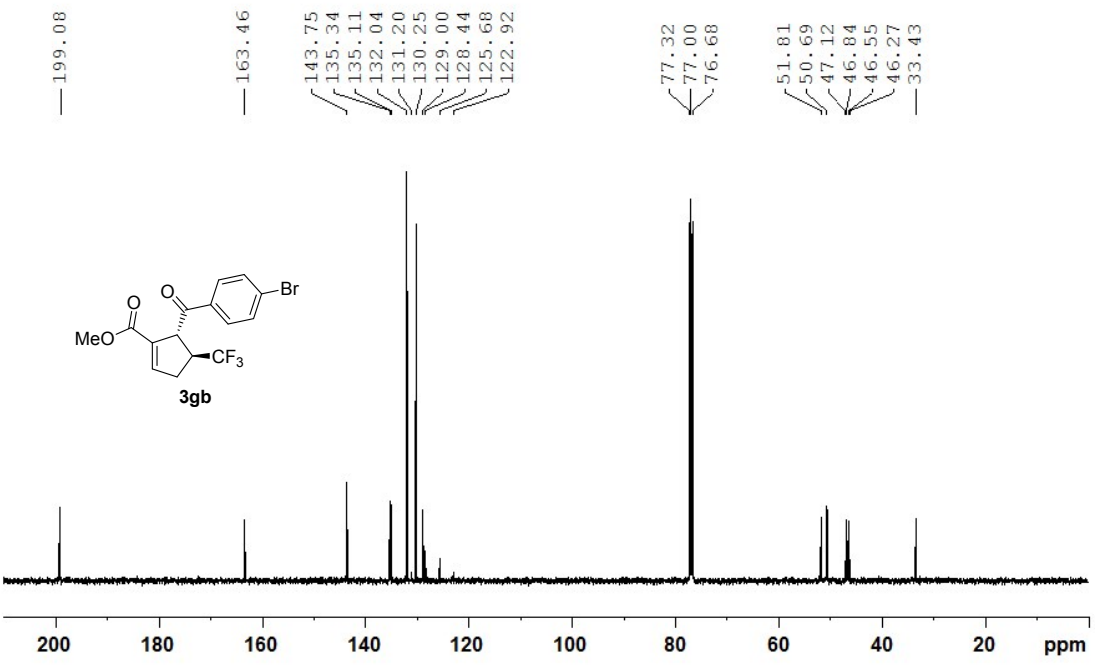

8.206
8.185
7.811
7.793
7.585
7.566
7.548
7.431
7.412
7.394
7.373
7.132
7.128
4.897
4.890
4.208
4.191
4.164
4.156
4.146
4.138
4.128
4.120
4.111
4.093
4.087
3.745
3.733
3.721
3.710
3.698
3.320
3.314
3.308
3.250
3.244
3.238
2.763
2.757
2.751
2.716
2.710
2.704
1.189
1.171
1.153
EtO
O
O
NO2
3xa
9
8
7
6
5
4
3
2
1
ppm
1.95
2.03
1.06
4.08
0.94
1.00
1.99
0.97
1.02
1.02
3.09


199.92
163.63
152.25
147.01
144.48
136.25
135.75
133.46
128.65
128.62
127.72
124.27
77.32
77.00
76.69
60.75
59.57
48.46
41.79
13.97
EtO
O
O
NO2
3xa
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

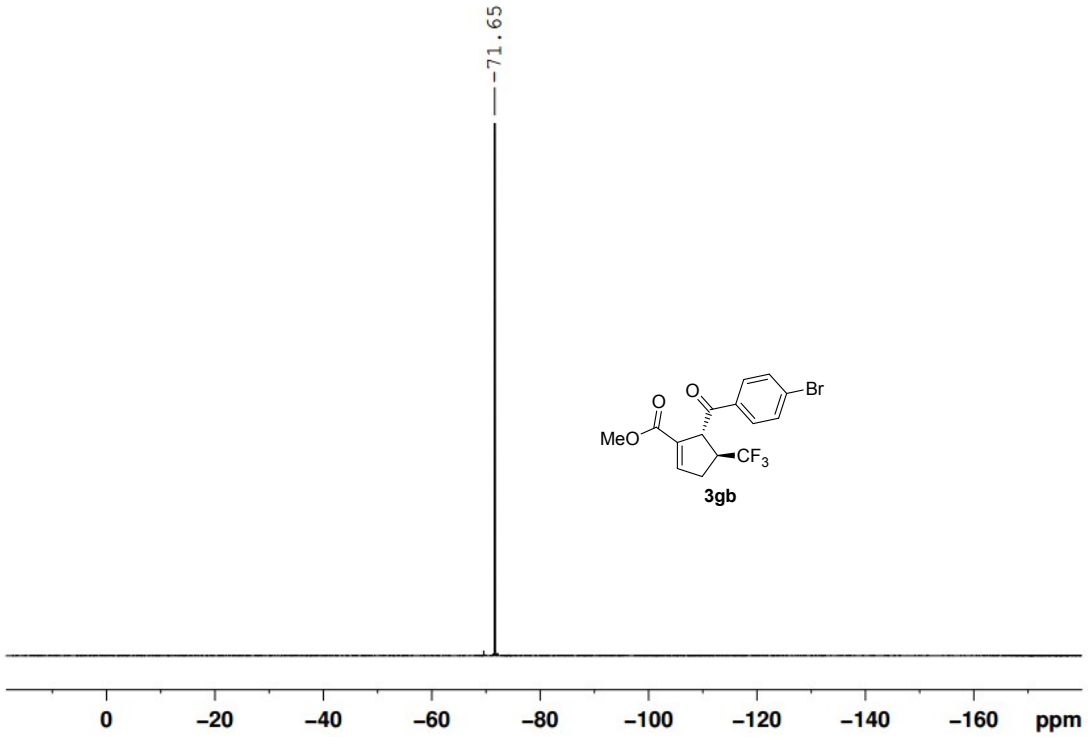
zhouwei-6-121



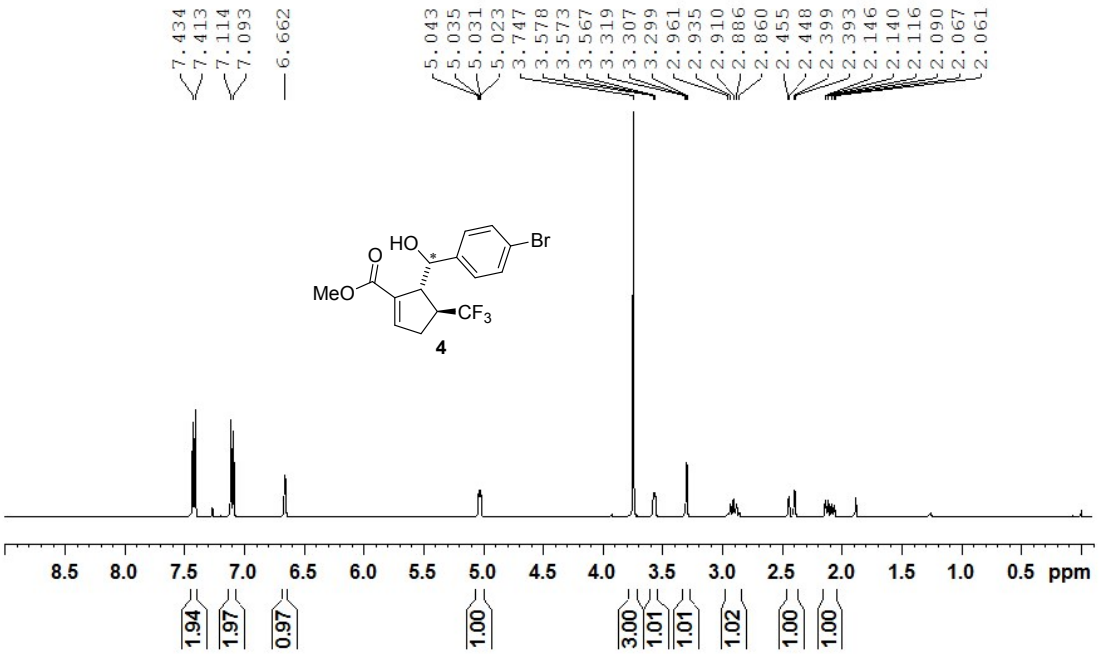
zhouwei-6-121c



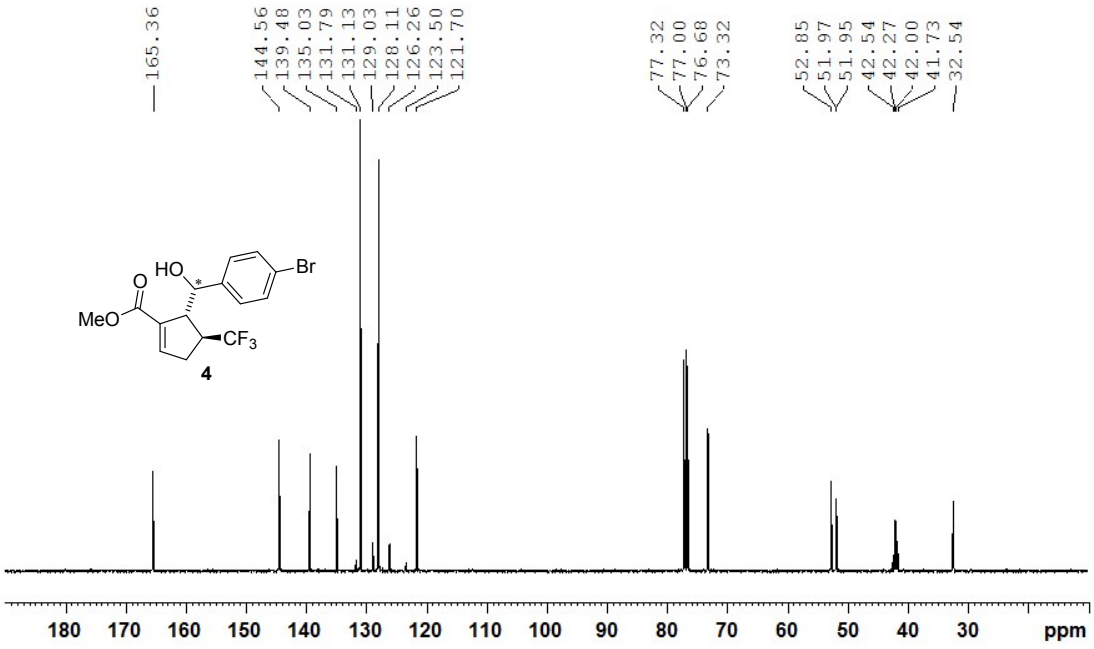
zhouwei-6-121f



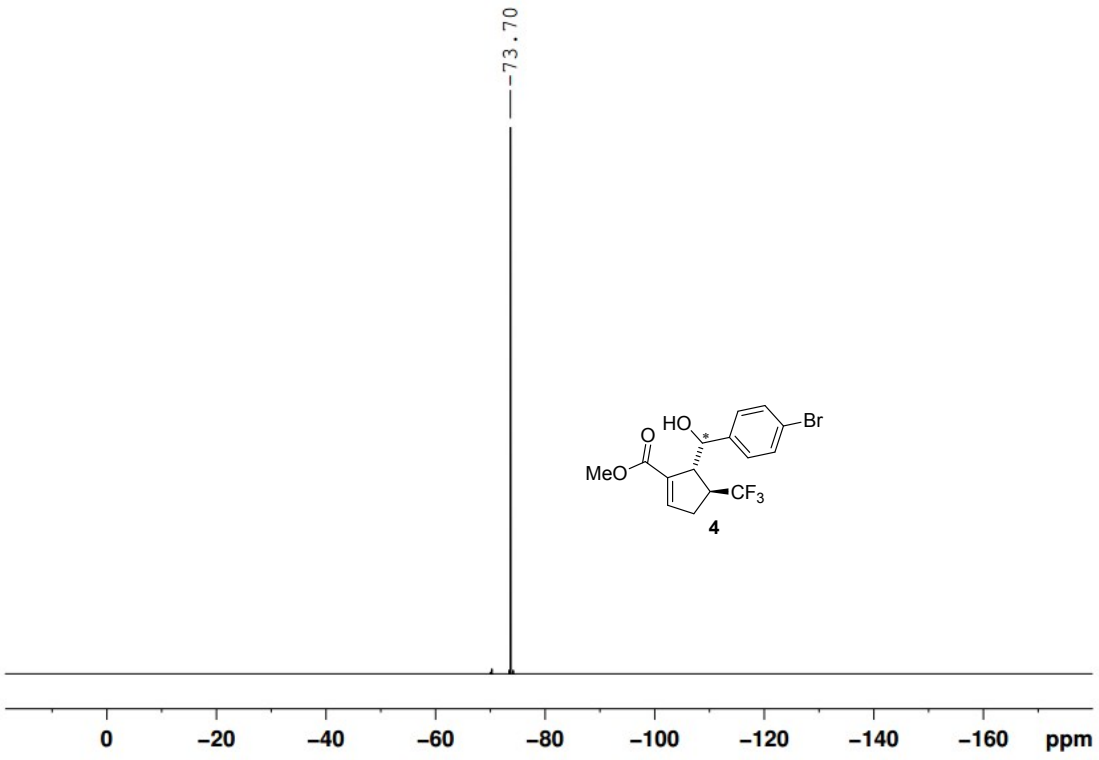
zhouw-6-136

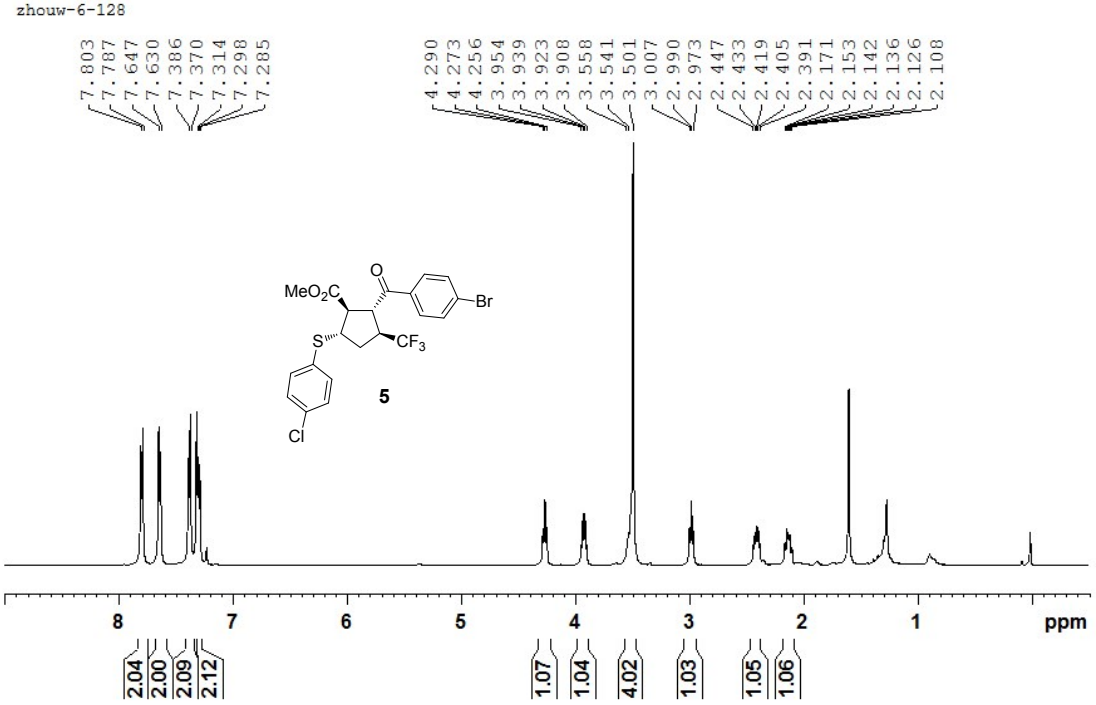


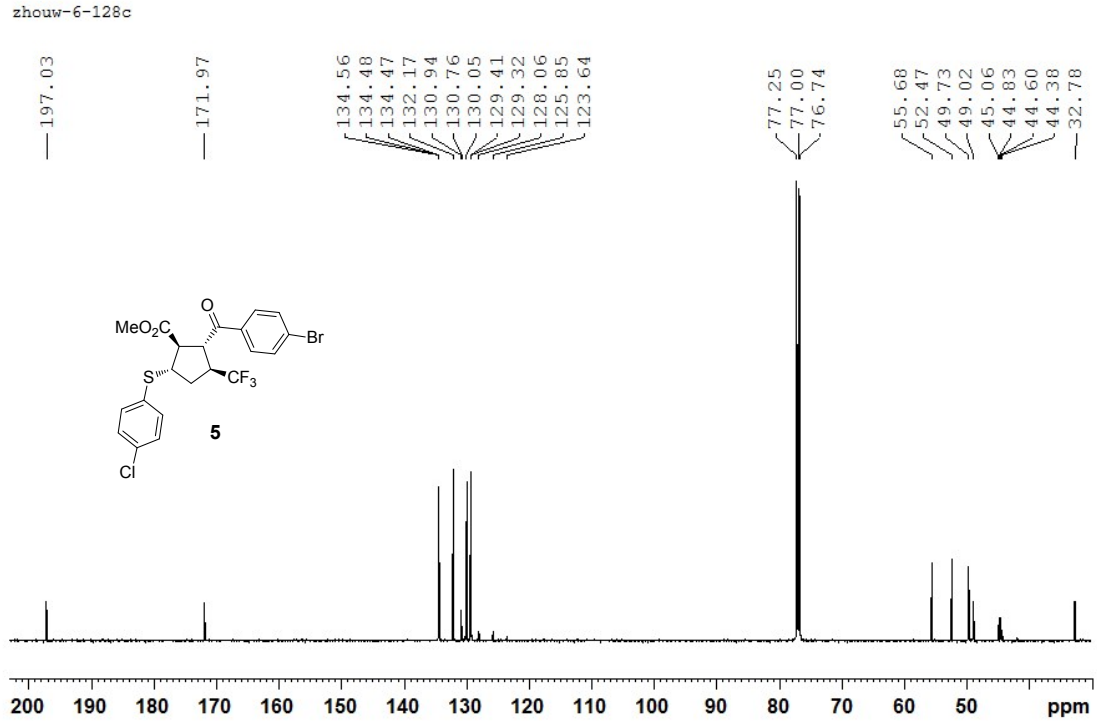
zhouw-6-136c



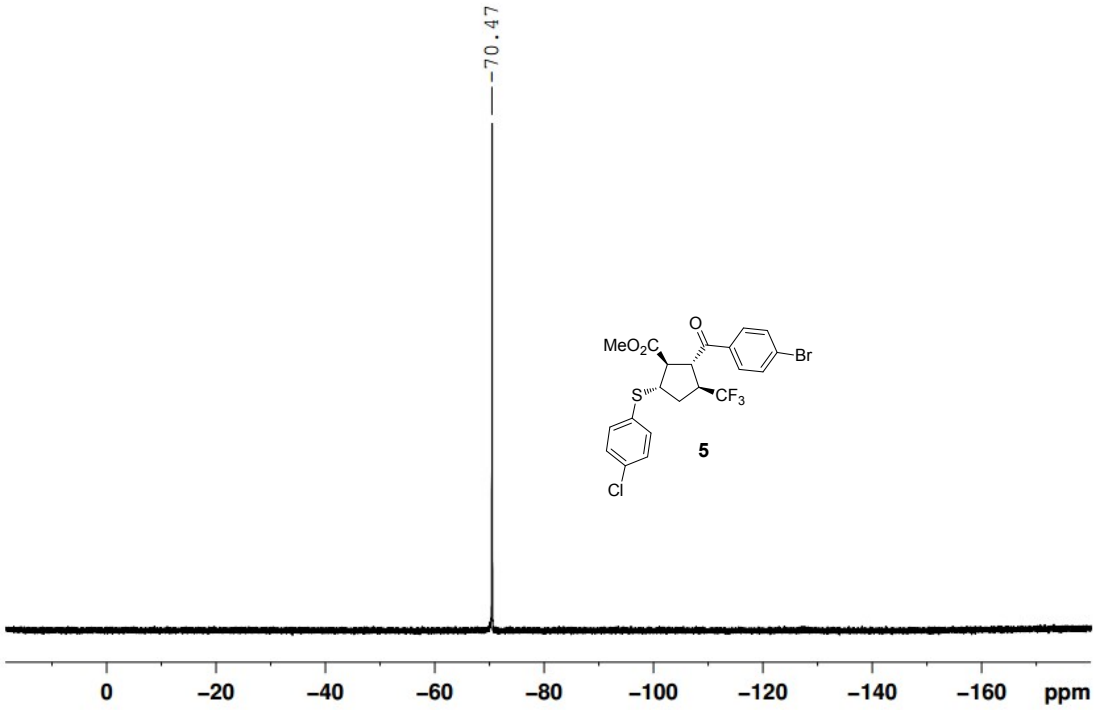
zhouw-6-136f



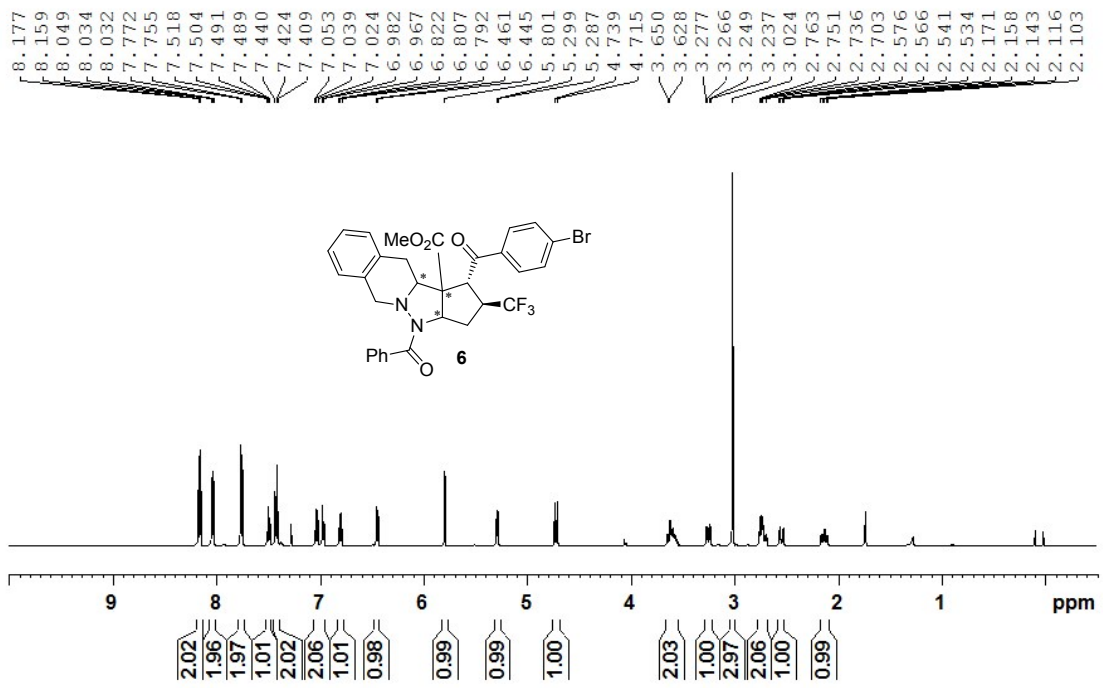

zhouw-6-128
7.803
7.787
7.647
7.630
7.386
7.370
7.314
7.298
7.285
4.290
4.273
4.256
3.954
3.939
3.923
3.908
3.558
3.541
3.501
3.007
2.990
2.973
2.447
2.433
2.419
2.405
2.391
2.171
2.153
2.142
2.136
2.126
2.108
MeO2C
O
Br
S
CF3
Cl
5
8
7
6
5
4
3
2
1
ppm
2.04
2.00
2.09
2.12
1.07
1.04
4.02
1.03
1.05
1.06


zhouw-6-128c
197.03
171.97
134.56
134.48
134.47
132.17
130.94
130.76
130.05
129.41
129.32
128.06
125.85
123.64
77.25
77.00
76.74
55.68
52.47
49.73
49.02
45.06
44.83
44.60
44.38
32.78
MeO2C
O
Br
S
CF3
Cl
5
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
ppm

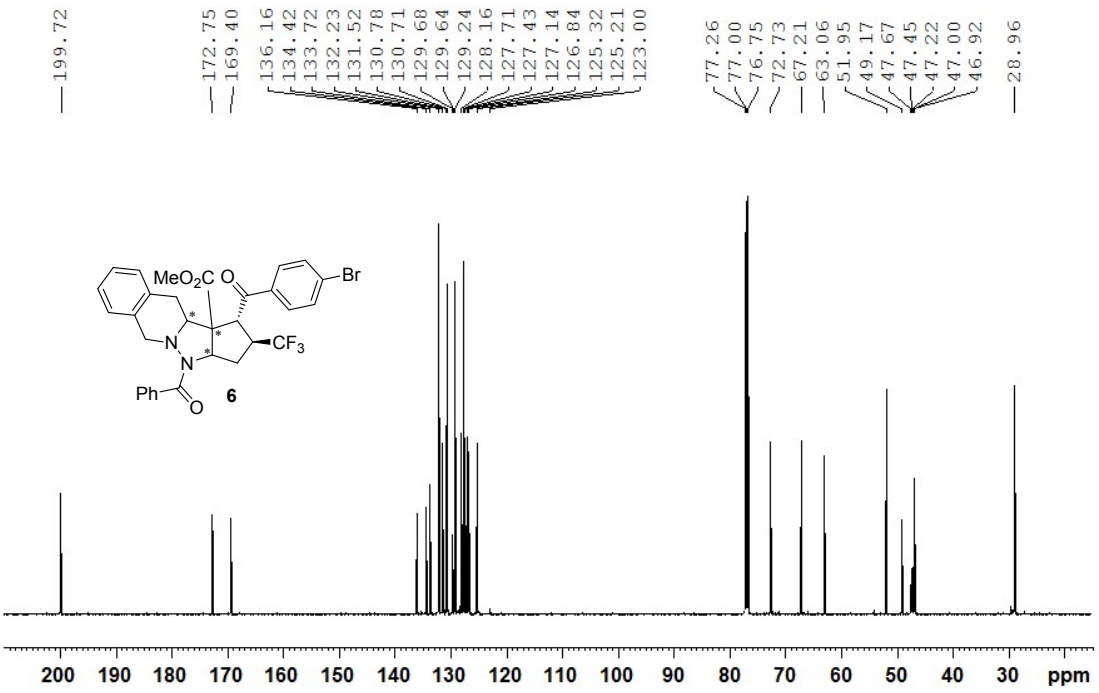
zhouw-6-128f



zhouw-6-134



zhouw-6-134c



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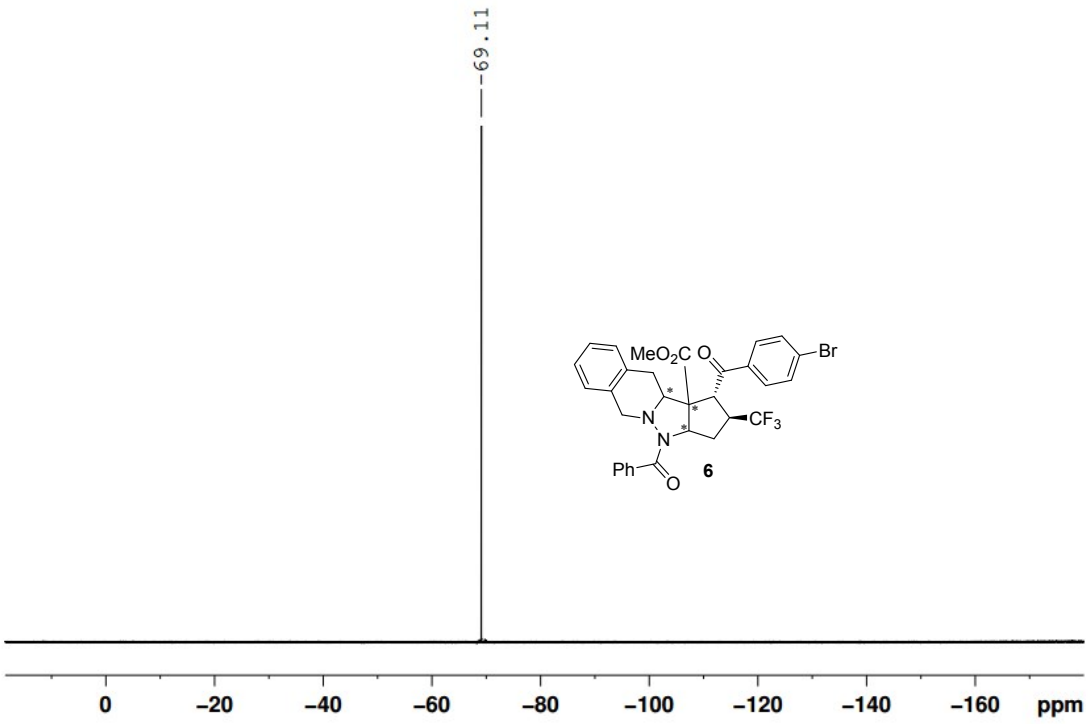
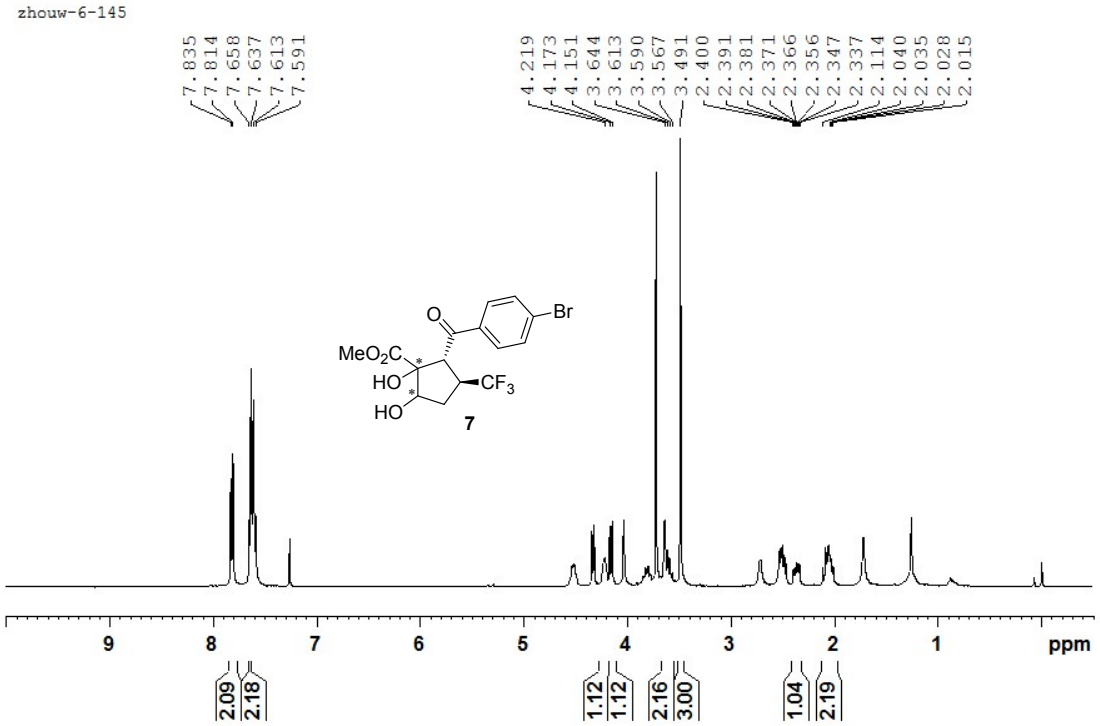
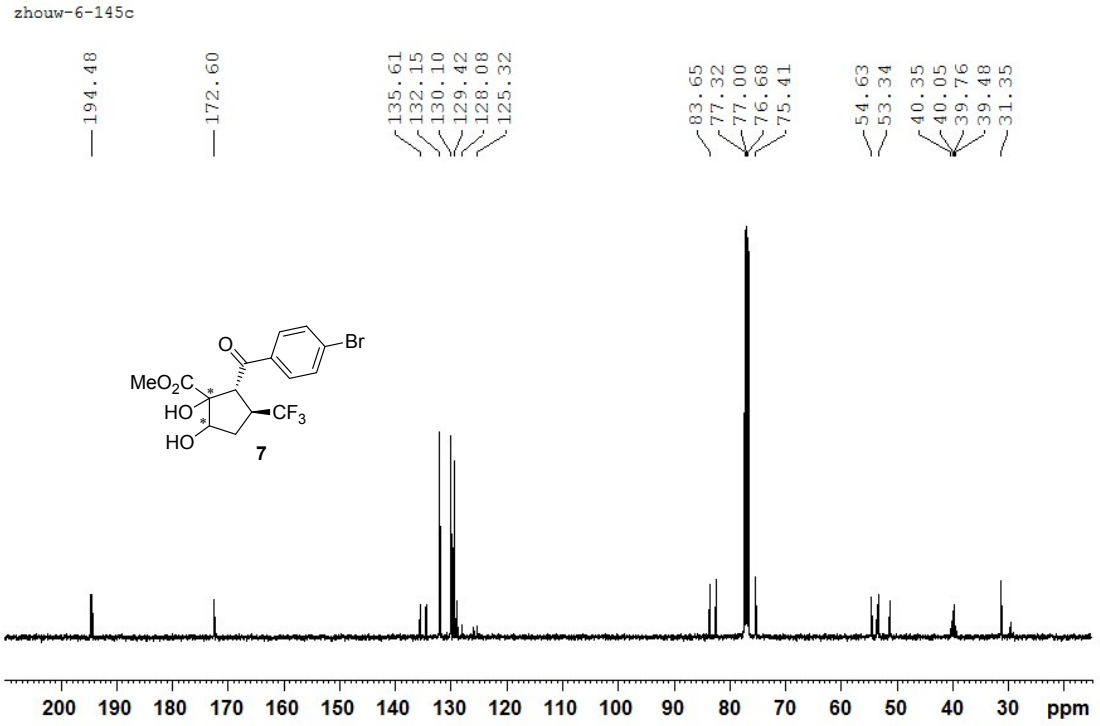
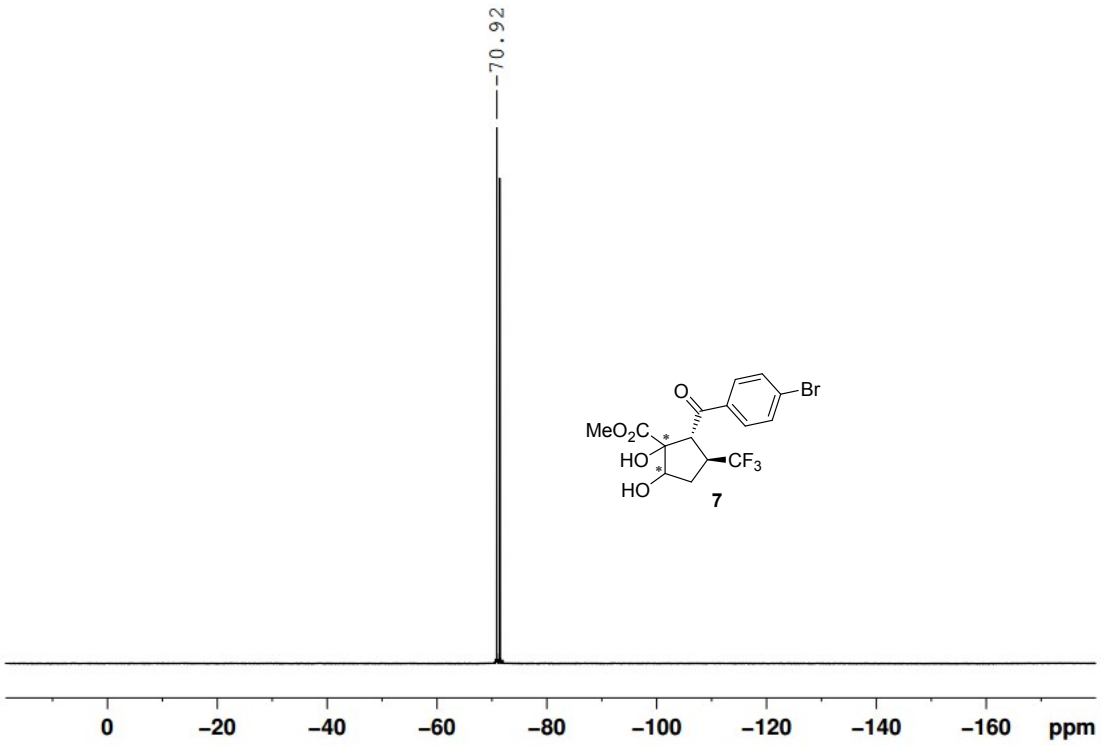


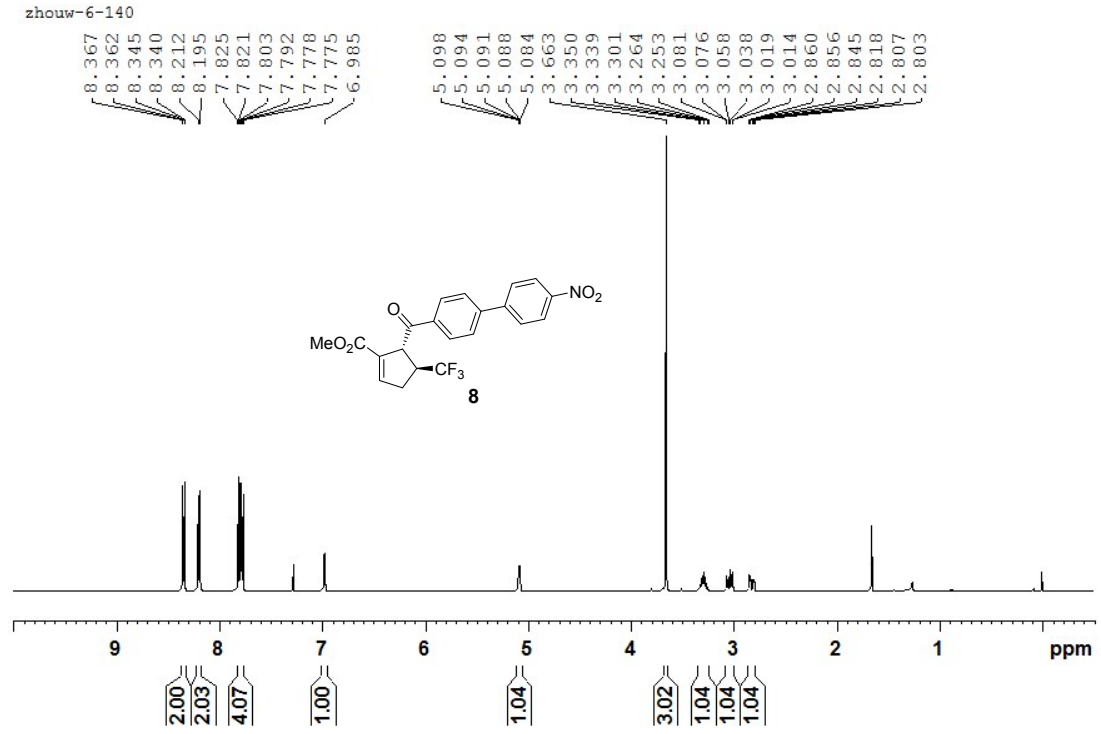

zhouw-6-145
7.835
7.814
7.658
7.637
7.613
7.591
4.219
4.173
4.151
3.644
3.613
3.590
3.567
3.491
2.400
2.391
2.381
2.371
2.366
2.356
2.347
2.337
2.114
2.040
2.035
2.028
2.015
Br
O
MeO2C
HO
HO
CF3
7
9
8
7
6
5
4
3
2
1
ppm
2.09
2.18
1.12
1.12
2.16
3.00
1.04
2.19

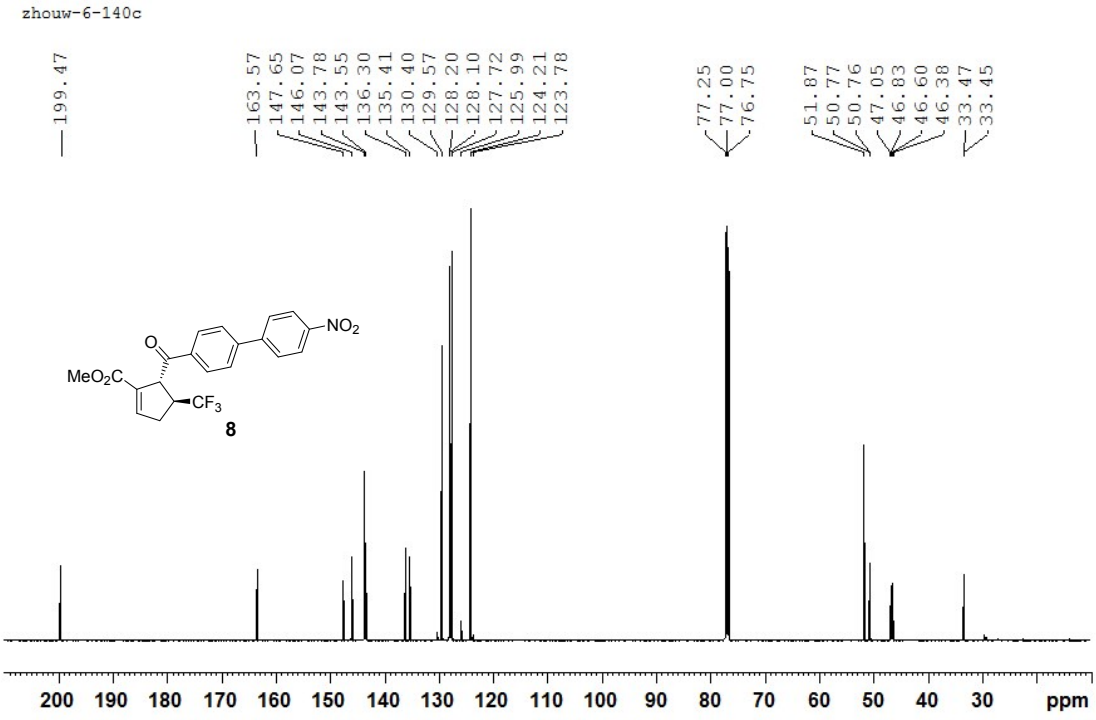

zhouw-6-145c
194.48
172.60
135.61
132.15
130.10
129.42
128.08
125.32
83.65
77.32
77.00
76.68
75.41
54.63
53.34
40.35
40.05
39.76
39.48
31.35
Br
O
MeO2C
HO
HO
CF3
7
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

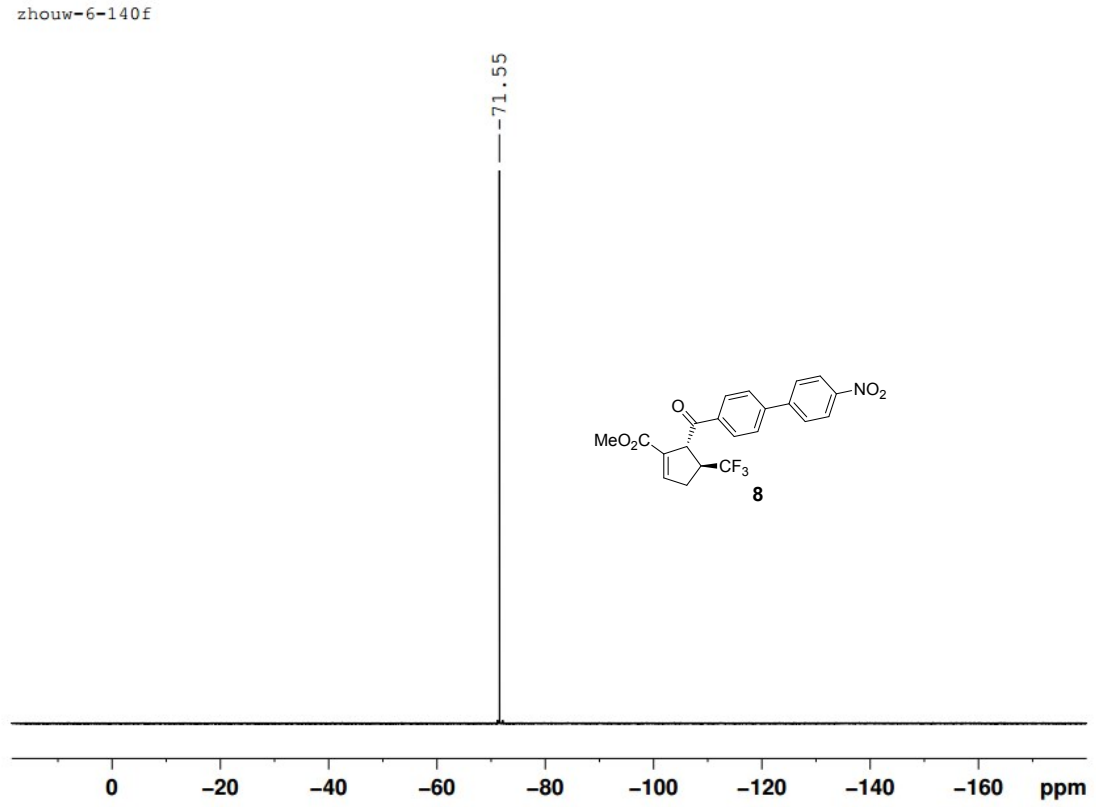
zhouw-6-145f



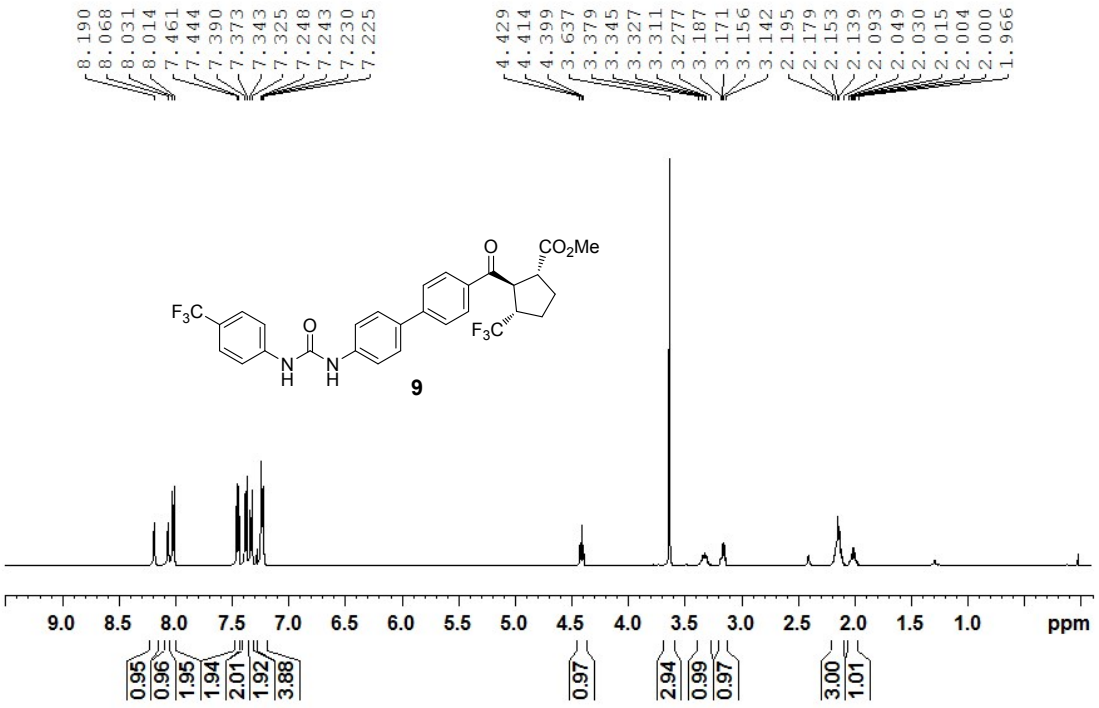
zhouw-6-140



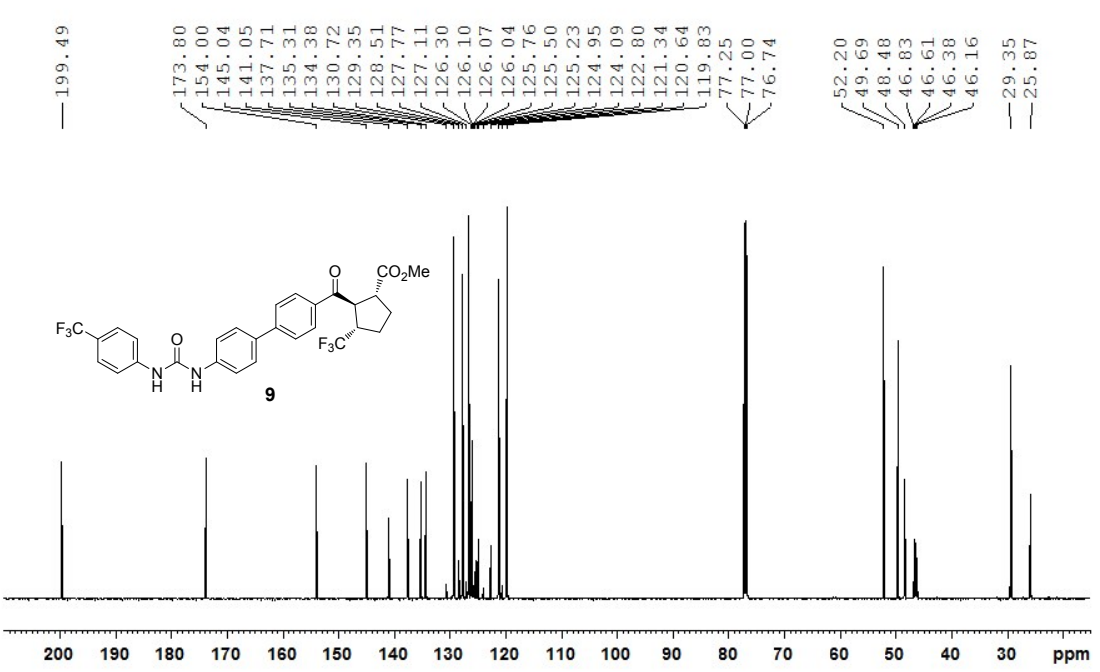

zhouw-6-140c
199.47
163.57
147.65
146.07
143.78
143.55
136.30
135.41
130.40
129.57
128.20
128.10
127.72
125.99
124.21
123.78
77.25
77.00
76.75
51.87
50.77
50.76
47.05
46.83
46.60
46.38
33.47
33.45
NO2
O
MeO2C
CF3
8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


zhouw-6-140f
-71.55
NO2
O
MeO2C
CF3
8
0 -20 -40 -60 -80 -100 -120 -140 -160 ppm

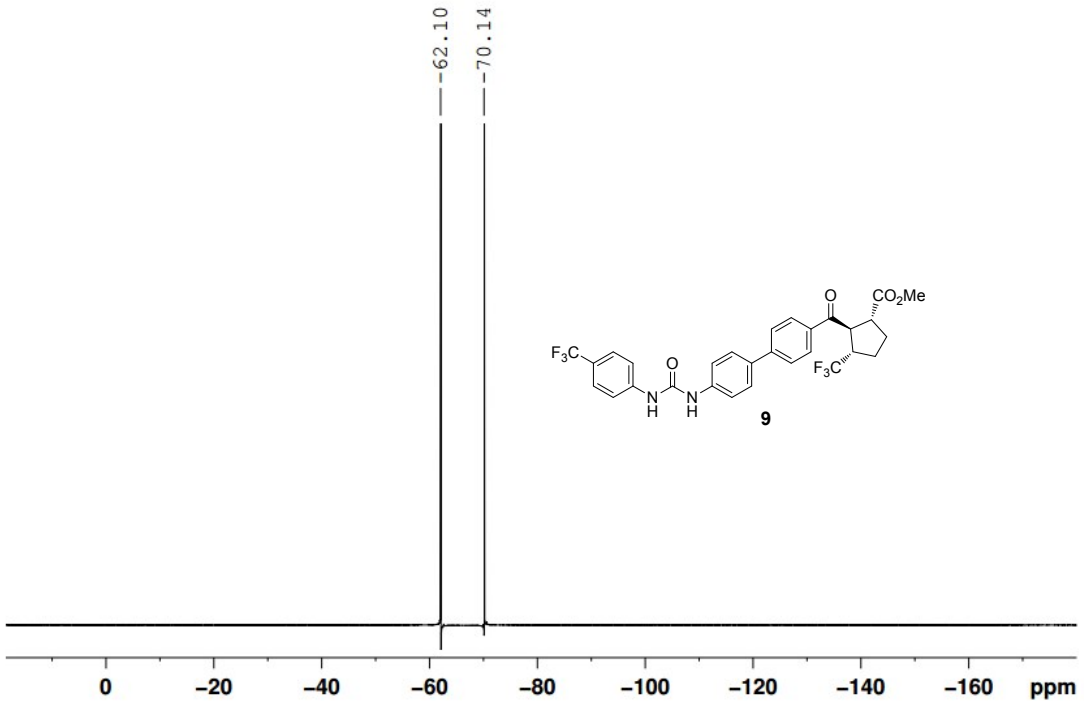
zhouw-6-146



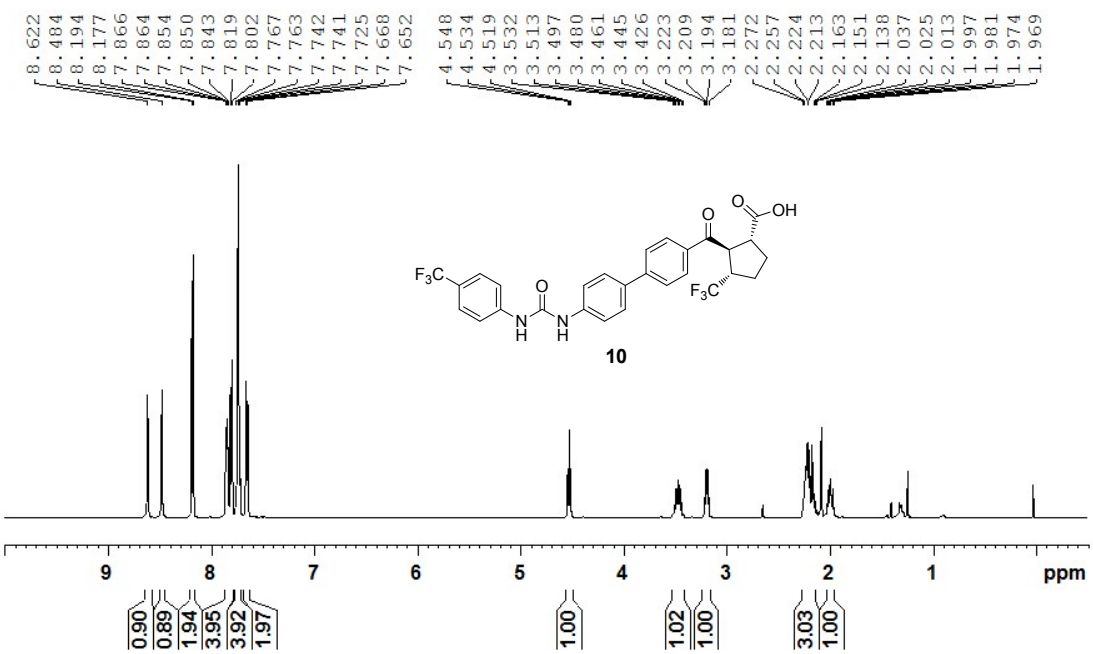
zhouw-6-146c



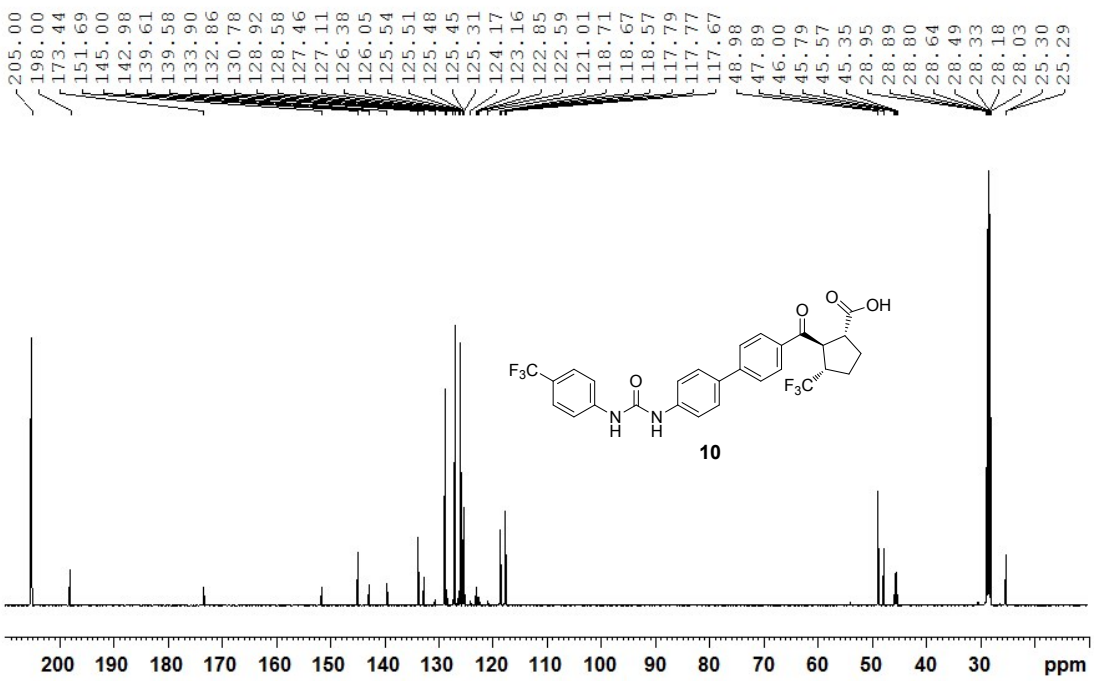
zhouw-6-146f



zhouw-6-147

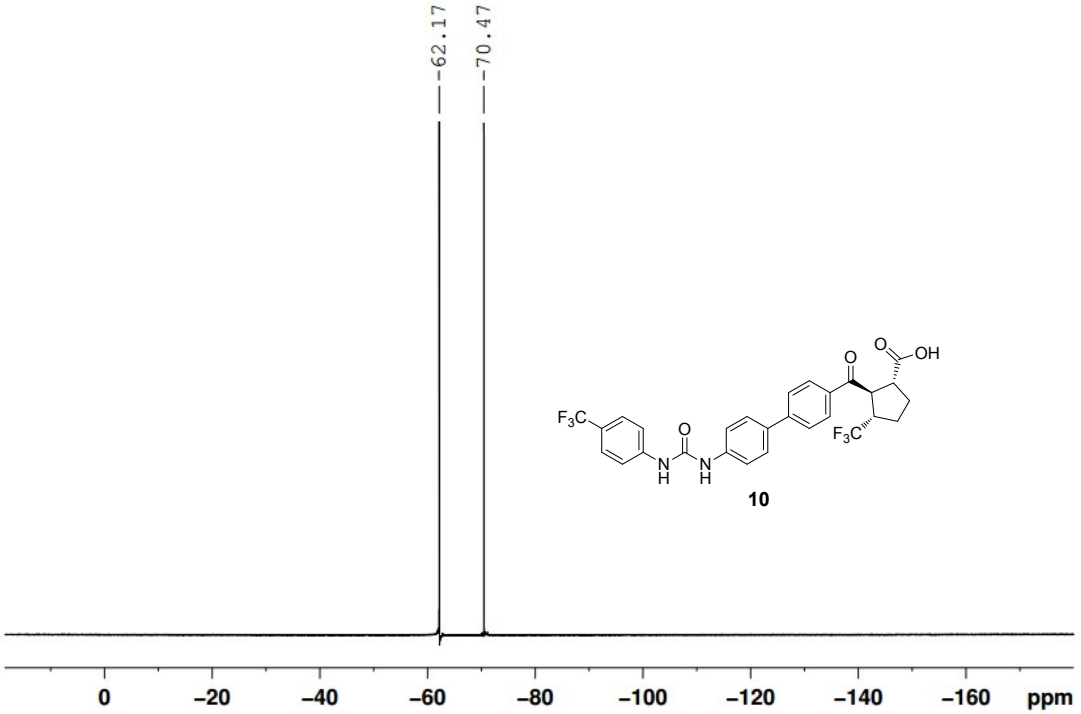


zhouw-6-147c



10

zhouw-6-147f



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