# Enantioselective (3+2) Cycloaddition *via N*-Heterocyclic Carbene-Catalyzed Addition of Homoenolate to Cyclic *N*-Sulfonyl Trifluoromethylated Ketimines: Synthesis of Fused *N*-Heterocycles γ-Lactams

Zhen-Zhen Zhang, Yongna Zhang,* Hui-Xin Duan, Zhuo-Fei Deng and You-Qing Wang*

*Henan Provincial Key Laboratory of Polyoxometalate Chemistry, Provincial Key Laboratory of Natural Medicine and Immuno-Engineering, College of Chemistry and Chemical Engineering, Henan University, Kaifeng, Henan 475004, P. R. China.*
*E-mail: zhangyongna@henu.edu.cn; wyouqing@hotmail.com*

**Supporting Information**

**Table of Contents.**

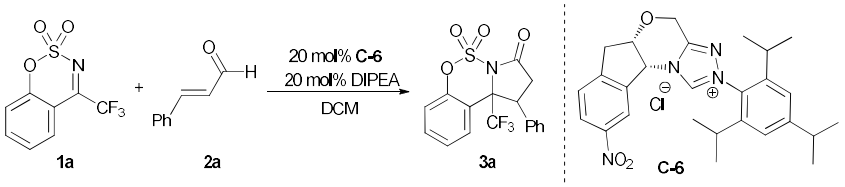
**1. General Information……...……...S1**
**2. More results on the condition optimization of (3+2) cycloaddition reaction..……S1-2**
**3. Preparation of substrates 1 and NHC catalyst C-6…..……S2-5**
**4. General procedure for NHC-catalyzed (3+2) cycloaddition reaction……...S5-11**
**5. Transformations of product 3a……...S11-13**
**6. X-ray structure for compound 3e……...……S13**
**7. Copy of NMR, HRMS and HPLC for new compounds ……..……S14**

## 1. General information

**General:** $^{1}H$ and $^{13}C$ NMR spectra were recorded on Bruker DRX-300 spectrometers, and $^{19}F$ NMR spectra were recorded on Bruker DRX-400 spectrometers. The chemical shifts for $^{1}H$ NMR were recorded in ppm (δ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$, d 7.26 ppm). The chemical shifts for $^{13}C$ NMR were recorded in ppm downfield using the central peak of deuterochloroform (77.0 ppm) as the internal standard. The *ee* values determination was carried out using chiral HPLC with Daicle chiral column on Agilent 1200. Flash column chromatography was performed on silica gel (200-300 mesh). TLC analysis was performed using glass-backed plates coated with 0.2 mm silica. The solvents were distilled from appropriate drying agents prior to use, unless otherwise noted. Cyclic imines were prepared according to the procedures reported in the literature.[1]

## 2. More results on the condition optimization of (3+2) annulation reaction

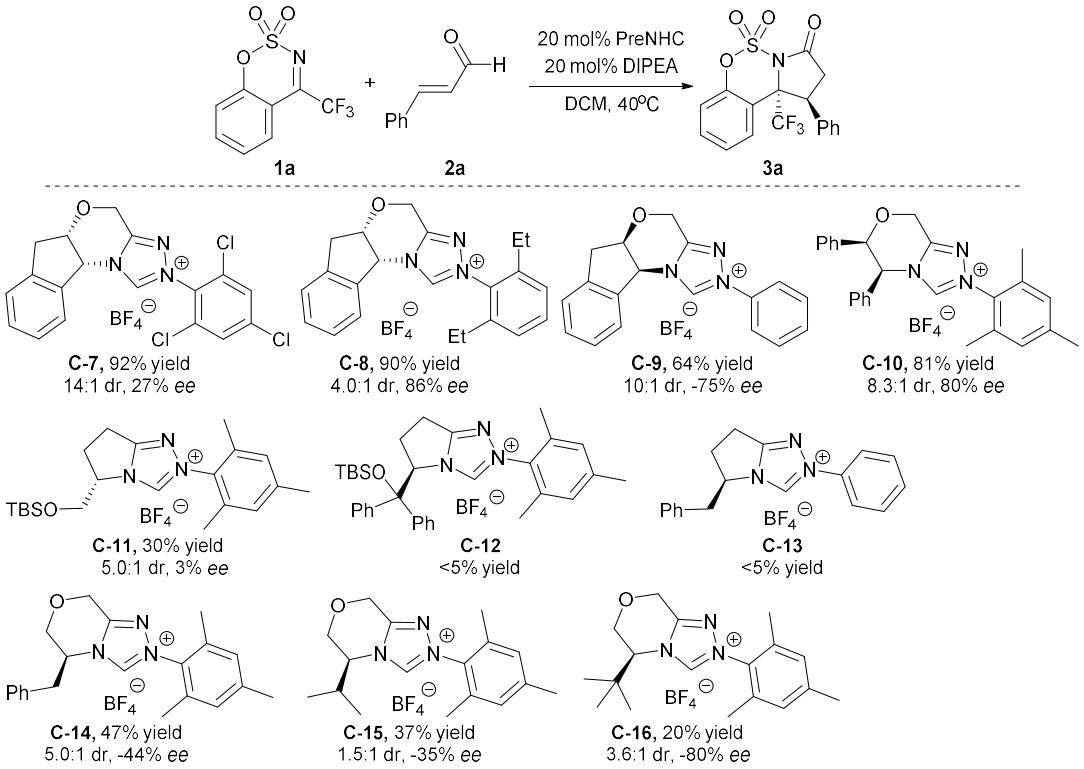
**Table S1** Effect of temperature and concentrations [a]




[1] (a) D. V. Sevenard, M.Vorobyev, V. Y. Sosnovskikh, H. Wessel, O. Kazakova, V. Vogel, N. E. Shevchenko, V. G. Nenajdenko, E. Lork and G.- E. Röschenthaler, *Tetrahedron*, 2009, **65**, 7538; (b) B. H. Brodsky and J. D. Bois, *J. Am. Chem. Soc*., 2005, **127**, 15391; (c) M.-W. Chen, X. C. Mao, Y. Ji, J. j. Yuan, Z. H. Deng and Y. Y. Peng, *Tetrahedron Lett.*, 2019, **60**, 151280.

| Entry | Concentration of **1a** [mol/l] | T [°C] | Yield [%][b] | dr [c] | *ee* [%][d] |
|---|---|---|---|---|---|
| 1 | 0.1 | 40 | 90 | 20:1 | 98 |
| 2 | 0.1 | 25 | 89 | 20:1 | 99 |
| 3 | 0.33 | 25 | 90 | 14:1 | 98 |
| 4 | 0.17 | 25 | 89 | 20:1 | 94 |
| 5 | 0.13 | 25 | 99 | 14:1 | 92 |
| 6 | 0.067 | 25 | 94 | >20:1 | 99 |
| **7** | **0.05** | **25** | **96** | **>20:1** | **99** |

[a] Reaction conditions: **1a** (0.05 mmol), **2a** (0.15 mmol). [b] Isolated yield. [c] Determined by $^1$H NMR. [d] Determined by HPLC using a chiral column.



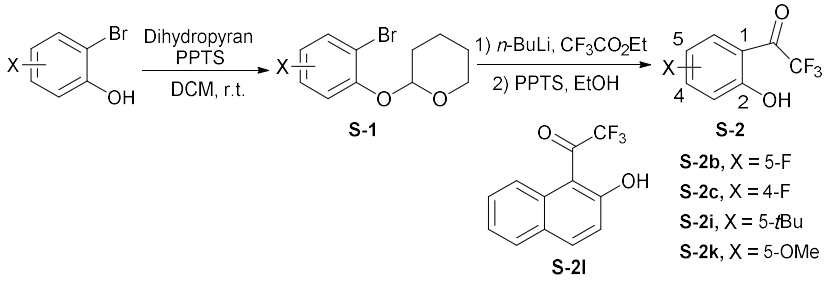

**Scheme S1** Screening for catalysts

## 3. Preparation of substrates 1 and NHC catalyst C-6

The known cyclic imines **1a**[1a], **1d**[1b], **1e**[1c], **1g**[1b], **1h**[1a], **1j**[1b] were prepared according to the modified procedures in the literature.

### 3.1 Preparation of substrates 1

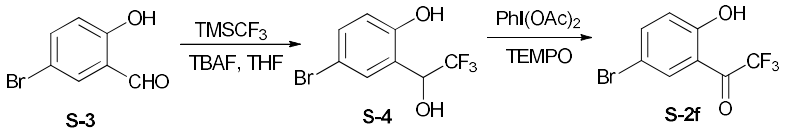
**1) Preparation of 2,2,2-trifluoro-2'-hydroxyacetophenone**



**Typical procedure according to the modified procedure in the literature**[1b]: (**1e** as an example)

**S-1e**: To a solution of 2-bromo-5-chlorophenol (5.0 g, 25 mmol) and dihydropyran (3.8 mL, 42.5 mmol) in $CH_2Cl_2$ (15 ml) was added PPTS (628.2 mg, 2.5 mmol). The resulting mixture was stirred at room temperature for 18 h. The reaction was quenched with $H_2O$, and the product was extracted with $CH_2Cl_2$. The combined organic layer was washed with brine, dried over $Na_2SO_4$ and evaporated. The crude mixture was purified by silica gel column chromatography to afford **S-1** (4.94g, 71% yield) as a colorless oil.

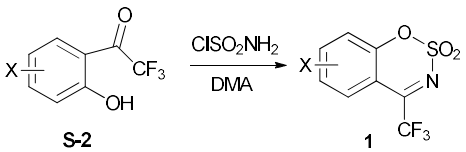
**S-2e:** 1) *n*-Butyllithium (2.5 M in hexanes, 8.2 mL, 21 mmol, 1.2 equiv) was added dropwise to a -78 °C solution of **S-1e** (5.0 g, 17 mmol) in 45 mL of THF. The resulting **S-1e** suspension was stirred at -78 °C for 20 min, then neat ethyl trifluoroacetate (4.0 mL, 34 mmol, 2.0 equiv) was added via syringe. The precipitate dissipated, and the homogenous solution was stirred for 1.5 h at -78 °C. The reaction was quenched at -78 °C by the addition of 20 mL of a 1/2 saturated aqueous $NH_4Cl$. The mixture was transferred to a separatory funnel, the organic layer was collected, and the aqueous layer was extracted with 3 x 15 mL of $Et_2O$. The combined organic extracts were washed with 15 mL saturated aqueous NaCl, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure to afford an oily residue. 2) The unpurified material was dissolved in 20 mL of EtOH and to the solution was added PPTS (427.2 mg, 1.7 mmol). The resulting solution was reflux for 12 h. The mixture was concentrated under reduced pressure, and then was extracted with 3 x 30 mL of ether, dried over $MgSO_4$. Purification by chromatography on silica gel (Pentane) furnished the desired 2,2,2-trifluoro-2'-hydroxyacetophenone as a colorless oil. The determined purity of **S-2e** by nuclear magnetic internal standard method is 52%.




To an ice cold solution of 5-bromosalicylaldehyde **S-3** (1g, 5 mmol) and $TMSCF_3$ (853 mg, 6 mmol, 1.2 equiv) in 8 mL of THF was added 1 mL TBAF (1.0 M in THF, 20 mol%). The solution was reflux for 10 h. Following this time, a 4.4 M aqueous solution of HCl (10 mL) was slowly added. The mixture was stirred for 1 h then diluted with 10 mL of EtOAc, and solid $Na_2CO_3$ was cautiously added. Once effervescence had ceased, the solution was dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification of this material by chromatography on silica gel afforded the desire product **S-4** (731.7 mg, 54%).

To a solution of **S-4** (1.08 g, 4 mmol) in 15 mL of $CH_2Cl_2$ was added TEMPO (0.2 mmol, 31.2 mg) followed by $PhI(OAc)_2$ (2.56 g, 8 mmol). The mixture was stirred for 13 h and quenched by the addition of 1.0 M aqueous $Na_2S_2O_3$. The mixture was transferred to a separatory funnel and the organic layer was collected. The aqueous layer was extracted with 3 x 20 mL of $CH_2Cl_2$. The combined organic fractions were dried over $Na_2SO_4$, and concentrated under reduced pressure. Purification of crude product by chromatography on silica gel afforded the desired product **S-2f** (699.4 mg, 65%).

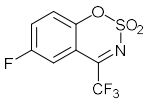
**2) Preparation of cyclic N-sulfonyl trifluoromethylated ketimines 1:**



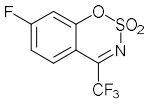

**S-2** **1**

**S-2b, S-2c, S-2f, S-2i, S-2k, S-2l**

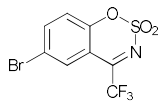
$ClSO_2NH_2$ was prepared by HCOOH and $ClSO_2NCO$ in situ: To an ice cold solution, anhydrous formic acid (1.38g, 30 mmol) was dropwise slowly to the flask of $ClSO_2NCO$ (4.24g, 30 mmol), then stirred at room temperature until there were no bubbles. The resulting solid was $ClSO_2NH_2$, It is directly used in the follow reaction. To a solution of 2-trifluoroacetylphenol **S-2** in DMA was quickly transferred solid $H_2NSO_2Cl$ (3.0 equiv.). **Caution:** a mild exotherm was noted upon combining these reagents. After stirring for 12 h, the reaction was quenched by the addition of 3 mL of $H_2O$ and transferred to a separatory funnel with 10 mL of $Et_2O$. The organic layer was separated, and the aqueous layer was extracted with 3 x 5 mL of $Et_2O$. The combined organic layers were washed successively with 2 x 3 mL of $H_2O$ and 1 x 5 mL of brine, dried over $MgSO_4$, and concentrated under reduced pressure. Purification by chromatography on silica gel afforded the desired cyclic ketimines **1**.



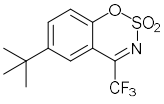

**1b:** 1.83 g, yield: 61%, colorless oil; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.64-7.58 (m, 2H), 7.45 (q, *J* = 4.3 Hz 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 159.2 (d, *J* = 249.1 Hz), 151.1 (d, *J* = 2.5 Hz), 126.3 (d, *J* = 24.1 Hz), 121.5 (d, *J* = 8.1 Hz), 118.1 (q, *J* = 279.6 Hz), 114.8 (q, *J* = 3.3 Hz), 114.5 (q, *J* = 3.3 Hz), 112.1 (d, *J* = 8.5 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$): δ -67.39, -110.88; HRMS (ESI): m/z calculated for $C_8H_4F_4NO_3S^+$ $(M+H)^+$ 269.9843, found: 269.9833.



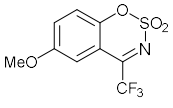

**1c:** 0.78 g, yield: 58%, m.p. 62.1-62.3 °C $^1$H NMR (300 MHz, $CDCl_3$): δ 8.03-7.98 (m, 1H), 7.27-7.21 (m, 1H), 7.17-7.14 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 160.5, 157.8, 154.9 (d, *J* = 3.9 Hz), 139.6 (d, *J* = 11.5 Hz), 117.8 (q, *J* = 278.6 Hz), 115.6 (d, *J* = 3.7 Hz), 114.8 (d, *J* = 26.4 Hz), 103.5 (d, *J* = 17.3 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$): δ -70.31, -70.40, -98.02, -98.12; HRMS (ESI): m/z calculated for $C_8H_4F_4NO_3S^+$ $(M+H)^+$ 269.9843, found 269.9852.



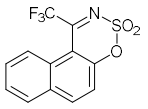

**1f:** 1.83 g, yield: 61%, m.p. 95.7-96.1 °C; $^1$H NMR (300 MHz, CDCl3): δ 8.03 (s, 1H), 7.96 (d, *J* = 8.9 Hz, 1H), 7.33 (d, *J* = 8.9 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl3): δ 161.1 (q, *J* = 37.8 Hz), 153.9, 141.5, 130.8 (q, *J* = 3.5 Hz), 121.3, 119.7, 118.1 (q, *J* = 279.8 Hz), 112.8; $^{19}$F NMR (376 MHz, CDCl3): δ -67.04; HRMS (EI): m/z calculated for $C_8H_3BrF_3NO_3S^+$ $(M)^+$ 328.8964, found: 328.8979.




**1i:** 1.39 g, yield: 41%, m.p. 60.1-60.3 °C $^1$H NMR (300 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.36-7.33 (m, 1H), 1.37 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 162.4 (q, *J* = 37.1 Hz), 152.9, 150.3, 136.5, 124.7 (q, *J* = 3.3 Hz), 118.4 (q, *J* = 279.5 Hz), 119.1, 111.2, 35.0, 31.0; $^{19}$F NMR (376 MHz, $CDCl_3$): δ -66.84; HRMS (ESI): m/z calculated for $C_{12}H_{13}F_3NO_3S$ $(M+H)^+$ 308.0563, found: 308.0567.

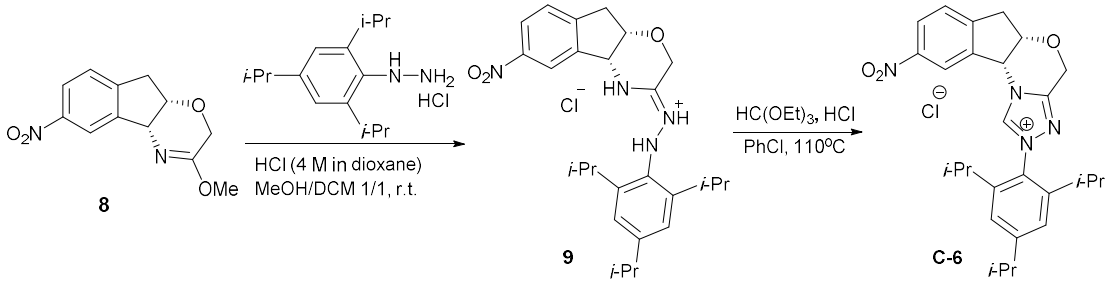



**1k:** 1.09 g, yield: 32%, m.p.109.2-109.4 °C $^1$H NMR (300 MHz, $CDCl_3$): δ 7.41-7.36 (m, 2H), 7.33-7.32 (m, 1H), 3.89 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 162.0 (q, *J* = 37.3 Hz), 157.3, 148.9, 125.6, 120.6, 118.3 (q, *J* = 279.6 Hz), 112.0, 111.2 (q, *J* = 3.3 Hz), 56.1; $^{19}$F NMR (376 MHz, $CDCl_3$): δ -67.14; HRMS (ESI): m/z calculated for $C_9H_7F_3NO_4S^+$ $(M+H)^+$ 282.0042, found 282.0045.



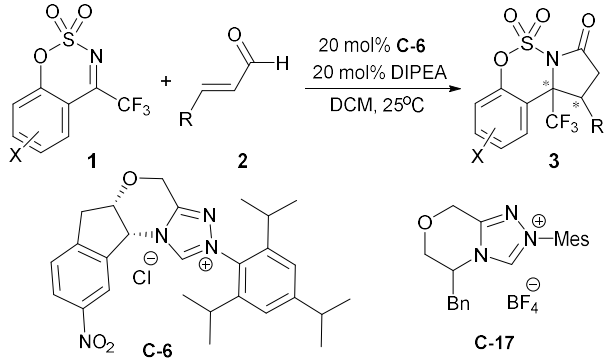
**1l:** 1.82 g, yield: 67%, m.p. 137.2-137.6 °C $^1$H NMR (300 MHz, $CDCl_3$): δ 8.30 (d, *J* = 8.9 Hz, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.82-7.77 (m, 1H), 7.69 (t, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 8.9 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ165.0 (d, *J* = 37.8 Hz), 157.3, 141.2, 131.3, 130.3, 129.7, 128.3, 127.8, 125.8 (q, *J* = 6.3 Hz), 118.9 (q, *J* = 279.9 Hz), 117.1, 110.8; $^{19}$F NMR (376 MHz, $CDCl_3$): δ -64.02; HRMS (ESI): m/z calculated for $C_{12}H_7F_3NO_3S^+$ $(M+H)^+$ 302.0093, found 302.0096.

### 3.2 Preparation of NHC catalyst C-6 [2]




The compound **8** (2.2 mmol) and the hydrazine hydrochloride (2.2 mmol) were dissolved in the mixed solvent of $CH_2Cl_2$ and MeOH (14 mL, v/v, 1/1) under the protection of argon. HCl (4 M in dioxane, 0.055 mL, 0.22 mmol) was added to the solution. The reaction mixture was stirrred at r.t. overnight. The mixture was evaporated to dryness to afford the crude product, which was directly used in the next step without further purification. The crude compound **9** was dissolved in chlorobenzene (6 mL) and then, $HC(OEt)_3$ (3.3 mL) and HCl (4 M in dioxane, 0.5 mL, 2 mmol) were added. The reaction mixture was heated at 110 °C for 0.5 h. The solvent was removed under reduced pressure and the product was purified via flash column chromatography on silica gel ($CH_2Cl_2$/MeOH = 15/1) to afford the triazolium salt as yellow solid 473 mg, 50% yield. $R_f$ = 0.20 (DCM/MeOH, 20:1), $^1$H NMR (300 MHz, $CDCl_3$) δ 12.55 (s, 1H), 8.32 (s, 1H), 8.13 (d, *J* = 8.2 Hz, 1H), 7.40 (d, *J* = 8.3 Hz, 1H), 7.12 (s, 3H), 5.21 (s, 1H), 5.04 (s, 2H), 3.55 (d, *J* = 17.4 Hz, 1H), 3.22 (d, *J* = 17.5 Hz, 1H), 3.15-2.86 (*m*, 1H), 2.64-2.44 (m, 1H), 2.10-1.90 (m, 1H), 1.37 -0.98 (m, 18H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 153.5, 148.8, 147.8 (d, *J* = 7.7 Hz), 146.0, 145.6, 145.1, 138.3, 128.4, 126.0, 124.8, 122.7, 122.2, 119.7, 61.8, 60.3, 37.8, 34.5, 29.1, 28.6, 24.4, 24.1, 23.9, 23.7, 23.4; HRMS (ESI) m/z calcd for $C_{27}H_{33}N_4O_3^+$ $(M-Cl)^+$ 461.2547, found 461.2544.

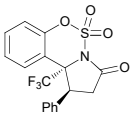
## 4. General procedure for NHC-catalyzed (3+2) cycloaddition reaction



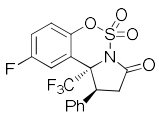

**General procedure:** The solution of DIPEA (0.02 mmol, 20 mol%) in DCM was added to the mixture of NHC cat. **C-6** (0.02 mmol, 20 mol%) and *N*-sulfonyl ketimines **1** (0.1 mmol). Then the

[2] (a) S.-X. Dong, M. Frings, H. C. Cheng, J. Wen, D. Zhang, R. Gerhard and B. Carsten , *J. Am. Chem. Soc*., 2016, **138**, 2166; (b) X.-Y. Chen, Q. Liu, P. Chauhan, S. Li, A. Peuronen, K. Rissanen, E. Jafari and D. Enders, *Angew. Chem. Int. Ed.*, 2017, **56**, 6241.

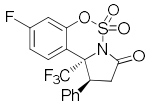
DCM (2 ml) was added, the α,β-unsaturated aldehydes **2** (0.3 mmol) was added. The reaction bottle was sealed and then stirred at 25 °C in showed reaction time. Direct purification of the crude reaction mixture by column chromatography on a silica gel (PE/EA) gave the desired annulation products. The enantiomeric excess was determined by HPLC, dr was determined by $^1$H NMR. Racemic samples for the standard of chiral HPLC spectra were prepared using racemic NHC precursor **C-17** as the catalyst.



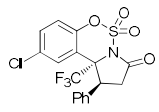
**(1*S*,10b*S*)-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3a):** $R_f$ = 0.48 (PE/EA, 3:1); White solid, 34.3 mg, 89% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 54.7 (*c* 1.5, $CHCl_3$); m.p. 149.9-150.4 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.35-7.27 (m, 1H), 7.22-7.12 (m, 4H), 7.05-6.90 (m, 4H), 4.24 (d, *J* = 8.7 Hz, 1H), 3.41-3.52 (m, 1H), 2.65 (dd, *J* = 18.0, 2.2 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.4, 149.4, 138.7, 131.6, 129.7 (d, *J* = 1.8 Hz), 129.4, 128.4, 127.1, 126.3, 124.5 (q, *J* = 286.7 Hz), 119.4, 118.7, 73.7 (q, *J* =30.0 Hz), 45.4, 38.9 (q, *J* = 2.6 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.19, -112.64; HRMS (ESI) m/z calculated for $C_{17}H_{13}F_3NO_4S^+$ $(M+H)^+$ 384.0512, found 384.0513; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 85/15, 1.0 mL/min, 210 nm): $t_1$ = 14.8 min (major), $t_2$ = 16.9 min.



**(1*S*,10b*S*)-9-fluoro-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3b):** $R_f$ = 0.44 (PE/EA, 3:1); White solid, 32.3 mg, 80% yield, 94% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 36.1 (*c* 1.5, $CHCl_3$); m.p. 203.0-203.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.25-7.17 (m, 3H), 7.17-7.07 (m, 1H), 7.08-6.95 (m, 3H), 6.69 (dd, *J* = 8.7, 2.8 Hz, 1H), 4.20 (d, *J* = 8.7 Hz, 1H), 3.46 (dd, *J* = 18.2, 8.7 Hz, 1H), 2.69 (d, *J* = 18.1 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.6, 159.7 (d, *J* = 247.5 Hz), 145.35 (d, *J* = 3.0 Hz), 138.2, 129.6, 128.8, 127.0, 124.3 (q, *J* = 286.7 Hz), 121.1 (d, *J* = 8.7 Hz), 120.4 (d, *J* = 8.5 Hz), 118.7 (d, *J* = 23.6 Hz) ,116.7 (d, *J* = 26.3 Hz), 73.7 (q, *J* = 32.3 Hz), 45.4, 38.7 (q, *J* = 2.5 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.19, -112.64; HRMS (ESI) m/z calculated for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.0417, found 402.0415; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 85/15, 1.0 mL/min, 210 nm): $t_1$ = 12.3 min (major), $t_2$ = 13.4 min.

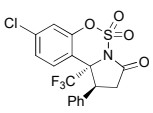


**(1*S*,10b*S*)-8-fluoro-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3c):** $R_f$ = 0.44 (PE/EA, 3:1); White solid, 37.7 mg, 94% yield, 98% *ee*, 17:1 dr, $[\alpha]^{28}_D$ = 69.0 (*c* 1.5, $CHCl_3$); m.p.117.6-118.1 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.25-7.14 (m, 3H), 7.09-6.99 (m, 2H), 6.98-6.84 (m, 2H), 6.78-6.67 (m, 1H), 4.21 (d, *J* = 8.8 Hz, 1H), 3.53-3.38 (m, 1H), 2.67 (dd, *J* = 18.1, 2.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.7, 164.8, 161.4, 150.0 (d, *J* = 11.9 Hz), 138.44, 131.03 (d, *J* = 9.7 Hz), 129.6, 128.6, 127.1, 124.4 (q, *J* = 286.4 Hz), 114.7 (d, *J* = 4.3 Hz), 114.0 (d, *J* = 21.9 Hz), 107.7 (d, *J* = 26.0 Hz), 73.5 (q, *J* = 30.3 Hz), 45.3, 38.7 (q, *J* = 2.4 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.41, -76.69, -105.04; HRMS (ESI) m/z calculated for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.0418, found 402.0414. HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 10.1 min (major), $t_2$ = 11.5 min.

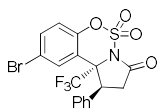


**(1*S*,10b*S*)-9-chloro-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3d):** $R_f$ = 0.56 (PE/EA, 3:1); White solid, 39.1 mg, 93% yield, 98% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -35.0 (*c* 1.5, $CHCl_3$); m.p. 188.0-188.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.51-7.32 (m, 5H), 7.24-7.18 (m, 2H), 7.11 (s, 1H), 4.37 (d, *J* = 8.7 Hz, 1H), 3.63 (dd, *J* = 18.1, 8.7 Hz, 1H), 2.88 (d, *J* = 18.1 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.5, 147.8, 138.1, 132.0, 131.6, 129.9-129.4 (m), 128.8, 127.1, 124.3 (q, *J* =

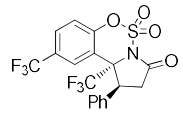
286.8 Hz), 120.7, 120.1, 73.6 (q, *J* = 30.1 Hz), 45.4, 38.5 (q, *J* = 2.3 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.08; HRMS (ESI) m/z calculated for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.0122, found 418.0123; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 9.2 min(major), $t_2$ = 10.3 min.



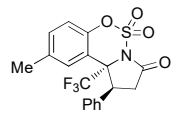
**(1*S*,10b*S*)-8-chloro-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3e):** $R_f$ = 0.34 (PE/EA, 3:1); White solid, 37.1 mg 89% yield, 98% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 44.5 (*c* 1.5, $CHCl_3$); m.p. 185.0-185.6 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.25-7.14 (m, 4H), 7.09-6.94 (m, 3H), 6.88 (d, *J* = 8.6 Hz, 1H), 4.21 (d, *J* = 8.8 Hz, 1H), 3.46 (dd, *J* = 18.2, 8.8 Hz, 1H), 2.67 (d, *J* = 18.1 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.6, 149.5, 138.4, 137.3, 130.4 (d, *J* = 2.0 Hz), 129.6, 128.7, 127.1, 126.7, 124.4 (q, *J* = 286.6 Hz), 120.2-119.8 (m), 117.2, 73.6 (q, *J* = 30.3 Hz), 45.32, 38.7 (q, *J* = 2.5 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.58; HRMS (ESI) m/z calculated for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.0122, found 418.0119; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 9.4 min (major), $t_2$ = 10.7 min.



**(1*S*,10b*S*)-9-bromo-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3f):** $R_f$ = 0.58 (PE/EA, 3:1); White solid, 37.0 mg, 80% yield, 98% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -111.03 (*c* 1.5, $CHCl_3$); m.p. 175.5-176.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.40 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.27-7.18 (m, 3H), 7.10-6.95 (m, 4H), 4.20 (d, *J* = 8.8 Hz, 1H), 3.46 (dd, *J* = 18.3, 8.8 Hz, 1H), 2.71 (d, *J* = 18.2 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.6, 148.3, 138.1, 134.5, 132.6 (d, *J* = 2.0 Hz) , 129.5, 128.8, 127.1, 124.3(q, *J* = 286.7 Hz), 121.0, 120.3, 119.3, 73.5 (q, *J* = 30.4 Hz), 45.3, 38.5 (q, *J* = 2.5 Hz); $^{19}$F NMR (376 MHz, Chloroform-*d*) δ -70.14, -76.13; HRMS (ESI) m/z calcd for $C_{17}H_{11}BrF_3NO_4SNa^+$ $(M+Na)^+$ 483.9437, found 483.9436; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 14.1 min (major), $t_2$ = 16.0 min.

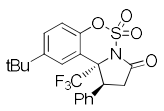


**(1*S*,10b*S*)-1-phenyl-9,10b-bis(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3g):** $R_f$ = 0.48 (PE/EA, 3:1); Light yellow solid, 16.4 mg, 37% yield, 97% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 16.0 (*c* 1.0, $CHCl_3$); m.p. 199.2-200 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.5 Hz, 1H), 7.30-7.16 (m, 5H), 7.02 (s, 2H), 4.25 (d, *J* = 8.8 Hz, 1H), 3.49 (dd, *J* = 18.3, 8.8 Hz, 1H), 2.75 (d, *J* = 18.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.4, 151.5, 138.0, 129.6, 129.0, 128.9, 128.6 (q, *J* = 3.7 Hz), 128.0 (q, *J* = 12.0 Hz), 127.3, 124.3 (q, *J* = 286.6 Hz), 122.5 (q, *J* = 271.0 Hz), 120.3, 119.6, 73.7 (q, *J* = 30.3 Hz), 45.5, 38.4 (q, *J* = 2.4 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -63.01, -76.10; HRMS (ESI) m/z calculated for $C_{18}H_{11}F_6NO_4SNa^+$ $(M+Na)^+$ 474.0205, found 474.0200; HPLC (Chiralpak IGX2-H column, hexane/*i*PrOH = 90/10, 1.0 mL/min, 210 nm): $t_1$ = 17.2 min, $t_2$ = 18.4 min (major).

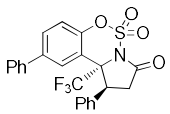


**(1*S*,10b*S*)-9-methyl-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3h):** $R_f$ = 0.34 (PE/EA, 3:1); White solid, 38.9 mg, 98% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 17.2 (*c* 1.0, $CHCl_3$); m.p. 146.3-146.9 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.25-7.13 (m, 3H), 7.11-6.96 (m, 4H), 6.69 (s, 1H), 4.21 (d, *J* = 8.7 Hz, 1H), 3.51-3.38 (m, 1H), 2.73-2.56 (m, 1H), 2.06 (s, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.2, 147.3, 138.9, 136.4, 132.0, 130.2, 129.3, 128.4, 127.2, 124.6 (q, *J* = 2.6 Hz), 119.0, 118.3, 73.7 (q, *J* = 29.7 Hz), 45.4, 38.8 (q, *J* = 2.6 Hz), 20.6; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.37, -76.76; HRMS (ESI) m/z calculated for $C_{18}H_{15}F_3NO_4S^+$ $(M+H)^+$ 398.0668, found 398.0670; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$

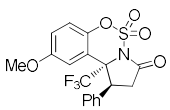
= 14.0 min (major), $t_2$ = 15.6 min.



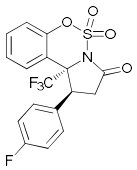
**(1*S*,10b*S*)-9-(tert-butyl)-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3i):** $R_f$ = 0.55 (PE/EA, 3:1); White solid, 42.6 mg, 97% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 12.0 (*c* 1.0, $CHCl_3$); m.p.164.0-164.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 8.7, 2.2 Hz, 1H), 7.25-7.14 (m, 3H), 7.12-7.01 (m, 3H), 6.83 (d, *J* = 2.3 Hz, 1H), 4.25(d, *J* = 8.8 Hz, 1H), 3.59-3.38 (m, 1H), 2.71-2.55 (m, 1H), 0.99 (s, 9H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.3, 149.7, 147.0, 139.2, 129.5, 128.6, 128.2, 127.3, 127.2, 124.6 (q, *J* = 286.2 Hz), 118.8, 118.0, 73.8 (q, *J* = 30.1 Hz), 45.4, 39.0 (q, *J* = 2.5 Hz), 34.5, 30.8; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.54, -77.34; HRMS (ESI) m/z calculated for $C_{21}H_{21}F_3NO_4S^+$ $(M+H)^+$ 440.1138, found 440.1136; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 90/10, 1.0 mL/min, 210 nm): $t_1$ = 11.6min, $t_2$ = 12.6 min (major).



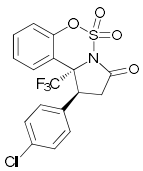
**(1*S*,10b*S*)-1,9-diphenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3j):** $R_f$ = 0.58 (PE/EA, 3:1); White solid, 42.4 mg, 92% yield, 99% *ee*, 17:1 dr, $[\alpha]^{28}_D$ = -71.3 (*c* 1.5, $CHCl_3$); m.p. 89.5-90.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.56-7.47 (m,1H), 7.45-7.37 (m, 3H), 7.36-7.27 (m, 4H), 7.17 (d, *J* = 7.2 Hz, 2H), 7.13-7.03 (m, 3H), 4.33 (d, *J* = 8.8 Hz, 1H), 3.64-3.45 (m, 1H), 2.80-2.62 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.1, 148.6, 140.0, 139.1, 138.6, 130.3, 129.6, 129.1, 128.9, 128.6, 128.2, 127.4, 127.0, 124.6(q, *J* = 286.6 Hz), 119.7, 119.0, 73.7 (q, *J* = 30.2 Hz), 45.5, 38.9 (q, *J* = 2.3 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.28, -76.98; HRMS (ESI) m/z calculated for $C_{23}H_{17}F_3NO_4S^+$ $(M+H)^+$ 460.0825, found 460.0826; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 90/10, 1.0 mL/min, 210 nm): $t_1$ = 17.3 min, $t_2$ = 19.6 min (major).



**(1*S*,10b*S*)-9-methoxy-1-phenyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (3k):** $R_f$ = 0.25 (PE/EA, 3:1); White solid, 40.5 mg, 98% yield, 99% *ee*, >20:1 *dr*, $[\alpha]^{28}_D$ = -17.4 (*c* 1, $CHCl_3$); m.p. 156.5-157.2 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.24-7.17 (m, 3H), 7.13-7.00 (m, 3H), 6.81 (dd, *J* = 9.1, 2.9 Hz, 1H), 6.35 (d, *J* = 2.9 Hz, 1H), 4.22 (d, *J* = 8.7 Hz, 1H), 3.52-3.39 (m, 4H), 2.71-2.57 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.2, 157.1, 142.9, 139.0, 129.5, 128.5, 127.2, 124.5(q, *J* = 286.6 Hz) 120.3, 119.62 117.5, 114.7, 114.6, 73.7 (q, *J* = 30.1 Hz), 55.7, 45.4, 39.0 (q, *J* = 2.3 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.43, -76.95; HRMS (ESI) m/z calculated for. $C_{18}H_{15}F_3NO_5S^+$ $(M+H)^+$ 414.0618, found 414.0619; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 90/10, 1.0 mL/min, 210 nm): $t_1$ = 24.5 min (major), $t_2$ = 26.2 min.

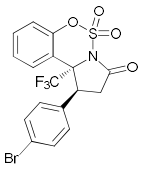


**(1*S*,10b*S*)-1-(4-fluorophenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4b):** $R_f$ = 0.40 (PE/EA, 3:1); White solid, 38.9 mg, 97% yield, 99% *ee*, 17:1 dr, $[\alpha]^{28}_D$ = 66.0 (*c* 1.5, $CHCl_3$); m.p. 146.0-146.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.40-7.29 (m, 1H), 7.17 (d, *J* = 8.3 Hz, 1H), 7.07-6.98 (m, 3H), 6.95-6.80 (m, 3H), 4.25 (d, *J* = 8.7 Hz, 1H), 3.46 (dd, *J* = 18.3, 8.8 Hz, 1H), 2.61 (d, *J* = 18.1 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.8, 162.2 (d, *J* = 249.0 Hz), 149.4, 134.6 (d, *J* = 3.6 Hz), 131.8, 129.6, 128.9, 126.4, 124.4 (q, *J* = 286.6 Hz), 119.6, 118.5, 116.5 (d, *J* = 21.6 Hz), 73.7 (q, *J* = 30.5 Hz), 44.7, 38.9; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.34, -76.61, -112.25; HRMS (ESI) m/z calculated for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.0418, found 402.0419; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 85/15, 1.0 mL/min, 210 nm): $t_1$ = 14.5 min (major), $t_2$ = 16.4 min.

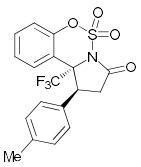


**(1*S*,10b*S*)-1-(4-chlorophenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4c):** $R_f$ = 0.40 (PE/EA, 3:1);

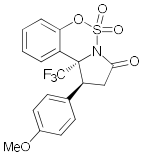
White solid, 40.3 mg, 96% yield, 98% *ee*, 17:1 dr, $[\alpha]^{28}_D$ = 44.8 (*c* 1.5, $CHCl_3$); m.p. 131.0-131.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.42-7.31 (m, 1H), 7.21-7.13 (m, 3H), 7.12-6.78 (m, 4H), 4.24 (d, *J* = 8.6 Hz, 1H), 3.46 (dd, *J* = 18.2, 8.7 Hz, 1H), 2.59 (dd, *J* = 18.0, 2.4 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.7, 149.3, 137.2, 134.4, 131.9, 129.6, 129.6, 128.5, 126.5, 124.4 (q, *J* = 286.8 Hz), 119.6, 118.4, 73.6 (q, *J* = 29.9 Hz), 44.8, 38.8 (q, *J* = 2.8 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.33, -76.71; HRMS (ESI) m/z calculated for $C_{17}H_{11}ClF_3NO_4SNa^+$ $(M+Na)^+$ 439.9942, found 439.9942; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 85/15, 1.0 mL/min, 210 nm): $t_1$ = 13.9 min (major), $t_2$ = 16.5 min.



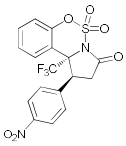
**(1*S*,10b*S*)-1-(4-bromophenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4d):** $R_f$ = 0.39 (PE/EA, 3:1); White solid, 44.9 mg, 97% yield, 99% *ee*, 17:1 dr, $[\alpha]^{28}_D$ = 95.2 (*c* 1.5, $CHCl_3$); m.p. 120.8-121.3 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.41-7.29 (m, 3H), 7.19 (d, *J* = 8.2 Hz, 1H), 7.06 (t, *J* = 7.7 Hz, 1H), 6.92 (d, *J* = 7.5 Hz, 3H), 4.22 (d, *J* = 8.7 Hz, 1H), 3.46 (dd, *J* = 18.1, 8.7 Hz, 1H), 2.59 (dd, *J* = 18.2, 2.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.7, 149.3, 137.7, 132.6, 131.9, 129.6 (d, *J* = 1.5 Hz), 128.7, 126.6, 124.3 (q, *J* = 286.7 Hz), 122.5, 119.6, 118.4, 73.5 (q, *J* = 30.1 Hz), 44.9, 38.7 (q, *J* = 2.5 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.32, -76.75; HRMS (ESI) m/z calculated for $C_{17}H_{12}BrF_3NO_4S^+$ $(M+H)^+$ 461.9617, found 461.9617; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 85/15, 1.0 mL/min, 210 nm): $t_1$ = 14.4 min (major), $t_2$ = 17.7min.



**(1*S*,10b*S*)-1-(p-tolyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4e):** $R_f$ = 0.44 (PE/EA, 3:1); White solid, 38.7 mg, 97% yield, 99% *ee*, 20:1 dr, $[\alpha]^{28}_D$ = 76.7 (*c* 1.5, $CHCl_3$); m.p. 134.0-134.7 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.36-7.27 (m, 1H), 7.15 (d, *J* = 8.3 Hz, 1H), 7.07-6.86 (m, 6H), 4.20 (d, *J* = 8.7 Hz, 1H), 3.44 (dd, *J* = 18.1, 8.7 Hz, 1H), 2.61 (d, *J* = 18.1 Hz, 1H), 2.21 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.2, 149.4, 138.2, 135.7, 131.5, 130.0, 129.8, 127.0, 126.3, 124.6 (q, *J* = 286.4 Hz), 119.4, 118.8, 73.8 (q, *J* = 29.8 Hz), 45.1, 39.0 (q, *J* = 2.6 Hz), 21.0; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.72; HRMS (ESI) m/z calcd for $C_{18}H_{15}F_3NO_4S^+$ $(M+H)^+$ 398.0668, found 398.0669; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 92/8, 1.0 mL/min, 210 nm): $t_1$ = 23.2min, $t_2$ = 24.6 min (major).

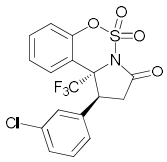


**(1*S*,10b*S*)-1-(4-methoxyphenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4f):** $R_f$ = 0.40 (PE/EA, 3:1); White solid , 37.4 mg, 90% yield, 99% *ee*, 20:1 dr, $[\alpha]^{28}_D$ = 70.0 (*c* 1.5, $CHCl_3$); m.p. 60.0-60.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.37-7.30 (m, 1H), 7.15 (dd, *J* = 8.3, 1.2 Hz, 1H), 7.08-7.00 (m, 1H), 6.95 (d, *J* = 8.9 Hz, 3H), 6.70 (d, *J* = 8.5 Hz, 2H), 4.20 (d, *J* = 8.6 Hz, 1H), 3.70 (s, 3H), 3.52-3.37 (m, 1H), 2.68-2.53 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.3, 159.2, 149.3, 131.5, 130.7, 129.8, 128.3, 126.3, 124.5 (q, *J* = 286.7 Hz), 119.4, 118.8, 114.6, 73.83 (q, *J* = 29.7 Hz), 55.2, 44.7, 39.1; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -70.53, -76.65; HRMS (ESI) m/z calculated for $C_{18}H_{15}F_3NO_5S^+$ $(M+H)^+$ 414.0618, found 414.0612; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 70/30, 1.0 mL/min, 230 nm): $t_1$ = 9.9 min (major), $t_2$ = 11.4 min.

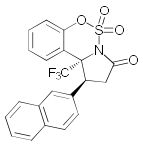


**(1*S*,10b*S*)-1-(4-nitrophenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4g):** $R_f$ = 0.25 (PE/EA, 3:1); Light yellow solid, 35.6 mg 83% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 55.2 (*c* 1.5, $CHCl_3$); m.p. 246.0-246.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.4 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 1H), 7.31-7.18 (m, 3H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 4.39 (d, *J* = 8.7

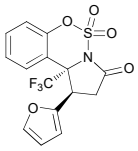
Hz, 1H), 3.52 (dd, *J* = 18.2, 8.7 Hz, 1H), 2.62 (d, *J* = 18.2 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.1, 149.3, 147.5, 145.7, 132.3, 129.2, 128.3, 126.8, 124.6, 124.3 (q, *J* = 286.5 Hz),119.9, 118.0, 73.30 (q, *J* = 30.4 Hz), 45.0, 38.4; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.73; $C_{17}H_{12}F_3N_2O_6S^+$ $(M+H)^+$ 429.0363, found 429.0366; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 70/30, 1.0 mL/min, 254 nm): $t_1$ = 19.5 min (major), $t_2$ = 22.2 min.



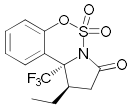
**(1*S*,10b*S*)-1-(3-chlorophenyl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4h):** $R_f$ = 0.50 (PE/EA, 3:1); White solid, 18.1 mg, 43% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 68.6 (*c* 1.5, $CHCl_3$); m.p. 157.5-158.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.41-7.32 (m, 1H), 7.23-7.02 (m, 5H), 6.95 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 1H), 4.21 (d, *J* = 8.7 Hz, 1H), 3.52-3.39 (m, 1H), 2.71-2.55 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.5, 149.4, 140.6, 135.1, 131.9, 131.0, 129.4, 128.7, 128.0, 126.5, 124.4 (q, *J* = 286.5 Hz), 124.6, 119.7, 118.4, 73.6 (q, *J* = 30.4 Hz), 45.1, 38.6; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.62; HRMS (ESI) m/z calculated for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.0122, found 418.0120; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 10.4 min (major), $t_2$ = 11.2 min.



**(1*S*,10b*S*)-1-(naphthalen-2-yl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4j):** $R_f$ = 0.53 (PE/EA, 3:1); Light yellow solid, 42.6 mg, 98% yield, 99% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = 120.1 (*c* 1.5, $CHCl_3$); m.p. 161.5-162.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.77-7.67 (m, 2H), 7.68-7.57 (m, 2H), 7.53-7.40 (m, 2H), 7.25-7.19 (m, 1H), 7.15 (d, *J* = 8.2 Hz, 1H), 7.11-6.95 (m, 2H), 6.87 (t, *J* = 7.7 Hz, 1H), 4.43 (d, *J* = 8.7 Hz, 1H), 3.54 (dd, *J* = 18.1, 8.8 Hz, 1H), 2.69 (d, *J* = 18.1 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.1, 149.4, 136.1, 133.0, 132.6, 131.6, 129.8, 129.7, 127.8, 127.7, 126.9, 126.7, 126.4, 124.6 (q, *J* = 286.6 Hz), 119.4, 118.7, 73.6 (q, *J* = 29.5 Hz), 45.6, 39.0; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -76.89; HRMS (ESI) m/z calculated for $C_{21}H_{15}F_3NO_4S^+$ $(M+H)^+$ 434.0668, found 434.0668; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 14.0 min (major), $t_2$ = 15.3 min.

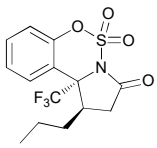


**(1*R*,10b*S*)-1-(furan-2-yl)-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4k):** $R_f$ = 0.38 (PE/EA, 3:1); Light yellow solid, 34.8 mg, 93% yield, 95% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -41.0 (*c* 1.5, $CHCl_3$); m.p. 122.7-123.2 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.41-7.32 (m, 1H), 7.26 (s, 1H), 7.21-7.08 (m, 3H), 6.17-6.07 (m, 2H), 4.40 (d, *J* = 8.6 Hz, 1H), 3.44-3.26 (m, 1H), 2.71 (dd, *J* = 17.8, 2.2 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 170.9, 150.0, 149.1, 142.8, 131.6, 128.0 (d, *J* = 2.0 Hz), 126.4, 124.2 (d, *J* = 287.5 Hz), 119.4, 118.01, 110.5, 108.9, 73.2 (q, *J* = 29.9 Hz), 39.6, 36.2 (d, *J* = 2.6 Hz); $^{19}$F NMR (376 MHz, $CDCl_3$) δ -75.20; HRMS (ESI) m/z calcd for $C_{15}H_{11}F_3NO_5S^+$ $(M+H)^+$ 374.0305, found 374.0306; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 13.7 min (major), $t_2$ = 15.8 min.

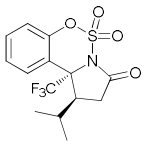


**(1*R*,10b*S*)-1-ethyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4l):** $R_f$ = 0.36 (PE/EA, 3:1); colorless oil, 27.6 mg, 82% yield, 95% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -96.9 (*c* 1.5, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ 7.65-7.48 (m, 1H), 7.45 (d, *J* = 4.6 Hz, 2H), 7.28 (d, *J* = 2.0 Hz, 1H), 3.15-2.89 (m, 2H), 2.63-2.44 (m, 1H), 1.57-1.38 (m, 1H), 1.20-0.97 (m, 1H), 0.89 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.1, 149.8, 131.9, 127.9 (q, *J* = 2.4 Hz), 126.9, 124.6 (q, *J* = 289.0 Hz), 120.0, 117.5, 74.2 (q, *J* = 29.4 Hz), 40.3, 34.06 (q, *J* = 2.7 Hz), 23.2, 10.0; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -72.54, -73.56; HRMS (ESI) m/z calculated for $C_{13}H_{13}F_3NO_4S^+$ $(M+H)^+$ 336.0512,

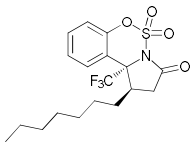
found 336.0512; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 13.6 min (major), $t_2$ = 19.8 min.



**(1*R*,10b*S*)-1-propyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4m):** $R_f$ = 0.36 (PE/EA, 3:1); White solid, 34.7 mg, 99% yield, 94% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -122.5 (*c* 1.5, $CHCl_3$); m.p. 104.0-104.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.62-7.51 (m, 1H), 7.50-7.40 (m, 2H), 7.28 (d, *J* = 2.9 Hz, 1H), 3.13-2.97 (m, 2H), 2.58-2.42 (m, 1H), 1.46-1.14 (m, 3H), 1.13-0.95 (m, 1H), 0.84 (t, *J* = 6.8 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.1, 149.8, 131.9, 127.9 (d, *J* = 2.5 Hz), 126.9, 124.6 (q, *J* = 289.1 Hz), 120.0, 117.5, 74.2 (q, *J* = 29.4 Hz), 38.8, 34.5 (q, *J* = 2.6 Hz), 32.0, 18.8, 13.6; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -73.51; HRMS (ESI) m/z calculated for $C_{14}H_{15}F_3NO_4S^+$ $(M+H)^+$ 350.0668, found 350.0669. HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 12.6min (major), $t_2$ = 18.8 min.

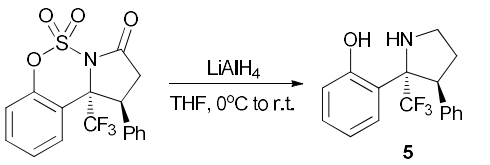


**(1*S*,10b*S*)-1-isopropyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4n):** $R_f$ = 0.36 (PE/EA, 3:1); White solid, 34.6 mg, 99% yield, 96% *ee*, >20:1 dr, $[\alpha]^{28}_D$ = -41.3 (*c* 1.5, $CHCl_3$); m.p. 124.0-124.6 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.62-7.53 (m, 1H), 7.52-7.38 (m, 2H), 7.28 (s, 1H), 3.07-2.86 (m, 2H), 2.53 (dd, *J* = 17.4, 2.4 Hz, 1H), 2.11-1.94 (m, 1H), 0.96 (d, *J* = 6.9 Hz, 3H), 0.58 (d, *J* = 6.7 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 170.8, 149.7, 131.9, 128.0 (q, *J* = 2.6 Hz), 126.7, 124.6 (q, *J* = 287.9 Hz), 120.0, 116.7, 73.8 (q, *J* = 28.9 Hz), 42.9, 30.1 (q, *J* = 2.7 Hz), 27.7, 21.2, 13.9; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -73.88; HRMS (ESI) m/z calcd for $C_{14}H_{15}F_3NO_4S^+$ $(M+H)^+$ 350.0668, found 350.0668; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 92/8, 1.0 mL/min, 210 nm): $t_1$ = 20.6 min (major), $t_2$ = 22.0 min.



**(1*R*,10b*S*)-1-heptyl-10b-(trifluoromethyl)-1,10b-dihydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazin-3(2*H*)-one 5,5-dioxide (4o):** $R_f$ = 0.55 (PE/EA, 3:1); White solid, 33.9 mg, 84% yield, 95% *ee*, >20:1 *dr*, $[\alpha]^{28}_D$ = -44.7 (*c* 1.5, $CHCl_3$); m.p. 84.5-85.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.61-7.52 (m, 1H), 7.50-7.39 (m, 2H), 7.28 (d, *J* = 2.1 Hz, 1H), 3.13-2.95 (m, 2H), 2.59-2.43 (m, 1H), 1.43-1.00 (m, 12H), 0.86 (t, *J* = 6.8 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.1, 149.9, 131.9, 127. 9 (q, *J* = 2.4 Hz), 126.9, 124.6 (d, *J* = 289.0 Hz), 120.0, 117.6, 74.2 (q, *J* = 29.2 Hz), 38.9, 34.6 (q, *J* = 2.4 Hz), 31.5, 30.0, 29.0, 28.9, 25.5, 22.5, 14.0; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -73.51; HRMS (ESI) m/z calculated for $C_{18}H_{23}F_3NO_4S^+$ $(M+H)^+$ 406.1294, found 406.1292; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 95/5, 1.0 mL/min, 210 nm): $t_1$ = 17.6 min (major), $t_2$ = 19.4 min.

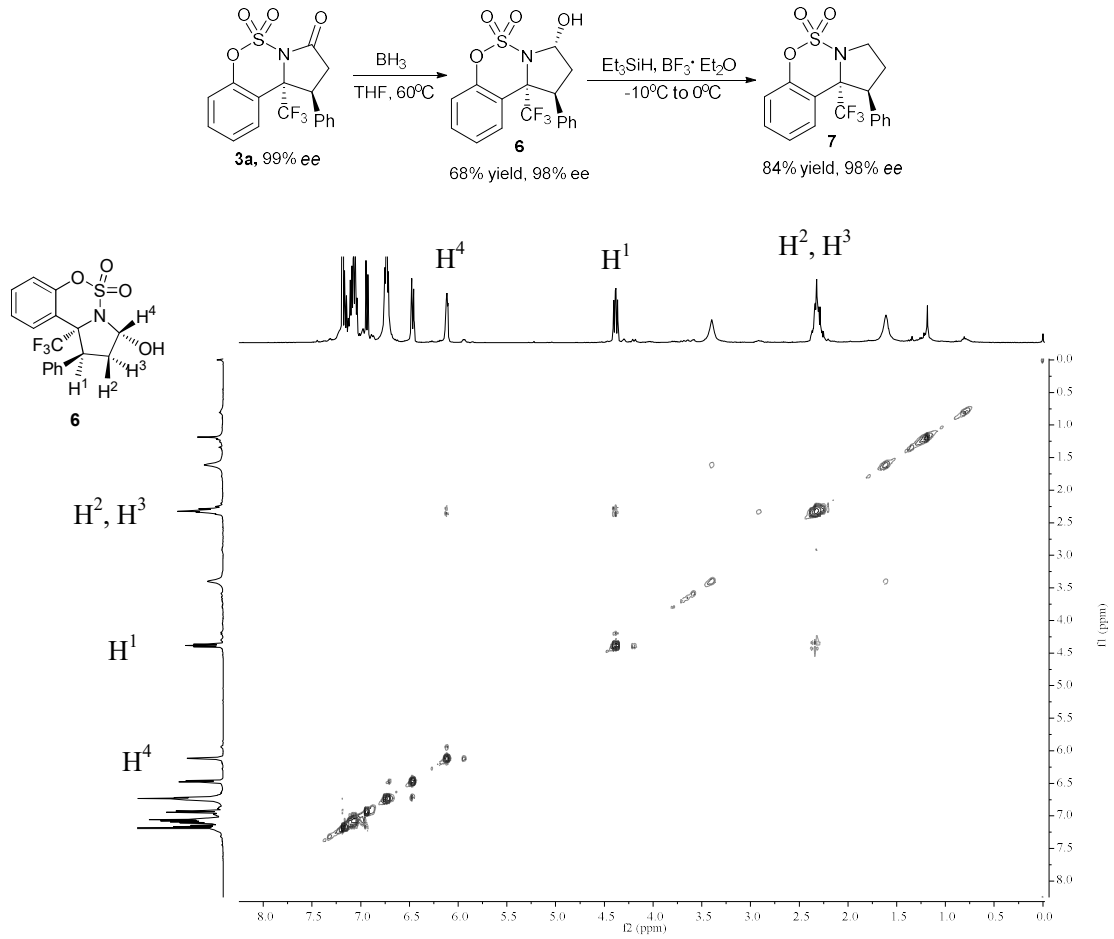
## 5. Transformations of product 3a




**3a**, 99% *ee* — **5**, 55% yield; 98% *ee*

**(4*S*,5*S*)-5-(2-hydroxyphenyl)-4-phenyl-5-(trifluoromethyl)pyrrolidin-2-one (5):** The compound **5** was synthesized according to reported literature. To a suspension of $LiAlH_4$ (1.0 mmol ) in THF (5 mL), **3a** (0.2 mmol) was added dropwise at 0 °C. The reaction mixture was stirred at room temperature for 1 hour, TLC showed one spot. Water was added to destroy the lithium aluminum hydride, the aqueous layer was extracted with EtOAc three times, the combined organic layers were dried and concentrated to provide the crude product. Purification by

chromatography on silica (PE/EA = 5:1) gave the product as white solid (61.7 mg, 55% yield, 98% *ee*). $R_f$ = 0.30 (PE/EA, 3:1); $[\alpha]^{25}_D$ = -21.3 (*c* 1.0, $CHCl_3$); m.p. 52.1-52.6 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.1 Hz, 1H), 7.35-7.23 (m, 3H), 7.20-7.11 (m, 3H), 7.02-6.93 (m, 1H), 6.87 (dd, *J* = 8.1, 1.5 Hz, 1H), 3.79-3.57 (m, 2H), 3.42 (td, *J* = 9.7, 4.3 Hz, 1H), 2.38-2.19 (m, 1H), 2.16-2.04 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 150.5, 135.5, 130.9, 129.7, 128.3, 128.0, 127.5 (d, *J* = 3.1 Hz), 125.8, 124.7 (q, *J* = 287.0 Hz), 119.7, 118.6, 69.72 (q, *J* = 27.3 Hz), 59.6, 50.1, 31.6; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -67.67, -68.76; HRMS (ESI) m/z calculated for $C_{17}H_{17}F_3NO^+$ $(M+H)^+$ 308.1257, found 308.1256; HPLC (Chiralpak IG-H column, hexane/*i*PrOH = 88/12, 1.0 mL/min, 210 nm): $t_1$ = 11.7 min (major), $t_2$ = 47.4 min.




**Scheme S2** 2D-NOESY NMR of the compound **6**

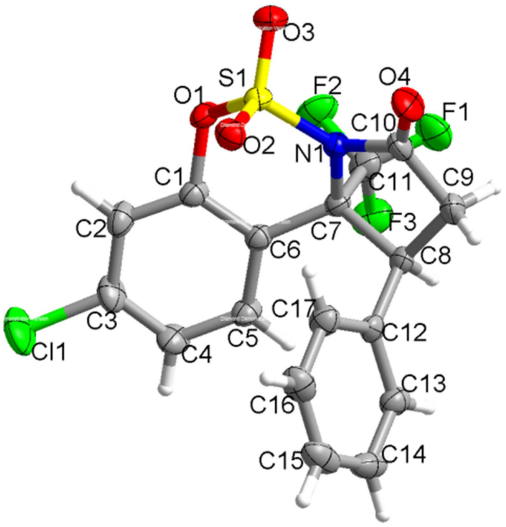
**(1*S*,3*R*,10b*S*)-3-hydroxy-1-phenyl-10b-(trifluoromethyl)-1,2,3,10b-tetrahydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazine 5,5-dioxide (6):** The solution of $BH_3$ (1M in THF, 0.8 ml, 0.8 mmol) in THF (2 mL) was added **3a** (0.2 mmol, 76.6 mg). The reaction mixture was stirred for 4 h at 60 °C. Purification by column chromatography on silica gel (PE/EA = 10:1) afforded the product **6** as white solid (77.3 mg, 68% yield, 98% *ee*). $R_f$ = 0.53 (PE/EA, 3:1); $[\alpha]^{28}_D$ = 42.7 (*c* 1.0, $CHCl_3$); m.p. 45.0-45.5 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.26-7.08 (m, 4H), 7.08-6.97 (m, 1H), 6.91-6.72 (m, 3H), 6.55 (d, *J* = 8.1 Hz, 1H), 6.24-6.16 (m, 1H), 4.46 (t, *J* = 8.3 Hz, 1H), 3.34 (s, 1H), 2.40 (q, *J* = 8.8, 6.6 Hz, 2H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 151.1, 136.1, 130.8, 129.5, 129.2 (d, *J* = 3.4 Hz), 128.3, 128.1, 125.1, 124.8 (q, *J* = 284.8 Hz), 119.3, 116.1, 88.3, 76.3(q, *J* = 29.7 Hz), 52.1, 38.5; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -71.66, -73.83; HRMS (ESI) m/z calculated for

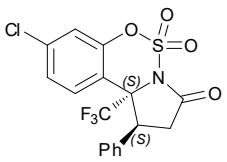
$C_{17}H_{13}F_3NO_3S^+$ $(M\text{-}OH)^+$ 368.0563, found 368.0565; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 80/20, 1.0 mL/min, 210 nm): $t_1$ = 7.6 min (major), $t_2$ = 8.9 min. The relative stereochemistry of compound **6** was determined by 2D-NOESY NMR (**Scheme S2**). No NOE effect was observed for between $H^1$ and $H^4$.

**(1*S*,10b*S*)-1-phenyl-10b-(trifluoromethyl)-1,2,3,10b-tetrahydrobenzo[*e*]pyrrolo[1,2-c][1,2,3]oxathiazine 5,5-dioxide (7):** Compound **6** (0.1 mmol, 38.5mg) was dissolved in DCM (0.5ml) and cooled to -10 °C. $Et_3SiH$ (3.2 eq, 0.32 mmol) was added to the mixture, and then $BF_3{\cdot}Et_2O$ (3.2eq, 0.32mmol) was added slowly. The temperature of the reaction system increased to 0 °C within 30 min and reacted under 0 °C for another 10 min. Purification by column chromatography on silica gel (PE/EA = 10:1) afforded the product **7** as white solid (30.8 mg 84% yield, 98% *ee*). $R_f$ = 0.56 (PE/EA, 3:1); White solid, 30.8 mg, 84% yield, 98% *ee,* $[\alpha]^{28}_D$ = 28.7 (*c* 1.5, $CHCl_3$); m.p. 99.4-100.0 °C; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.21 (d, *J* = 7.2 Hz, 1H), 7.17-7.07 (m, 3H), 7.03 (dd, *J* = 8.2, 1.3 Hz, 1H), 6.88-6.71 (m, 3H), 6.56 (d, *J* = 8.1 Hz, 1H), 4.42-4.29 (m, 1H), 4.18-3.97 (m, 1H), 3.92-3.77 (m, 1H), 2.45-2.30 (m, 1H), 2.32-2.13 (m, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 151.3, 137.0, 130.6, 129.1, 129.0 (d, *J* = 3.4 Hz), 128.3, 127.9, 125.5 (q, *J* = 285.0 Hz), 125.0, 119.3, 117.2, 76.1 (q, *J* = 28.9 Hz), 55.1, 52.4, 31.8; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -67.23, -72.82; HRMS (ESI) m/z calcd for $C_{17}H_{15}F_3NO_3S^+$ $(M+H)^+$ 370.0719, found 370.0719; HPLC (Chiralpak IC-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, 210 nm): $t_1$ = 14.3 min (major), $t_2$ = 15.5 min.

### 6. X-ray structure for compound 3e

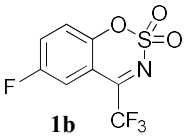
A single crystal of the compound **3e**（CCDC 1919445）was grown from its solution in dichloromethane/isopropanol (1/3), which is suitable for X-ray diffraction analysis. The correctness of the X-ray data and the structure had been checked by using the Check CIF utility on the submission Web site: http://checkcif.iucr.org



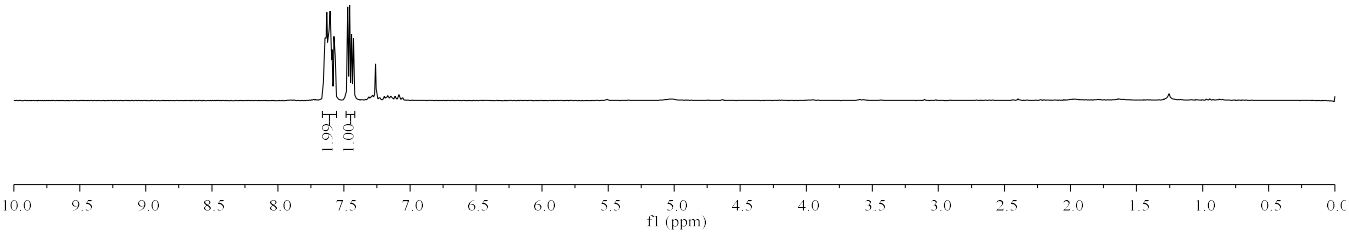



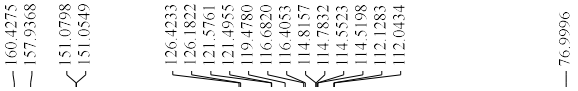

**3e**

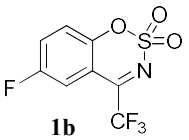
**Copy of NMR for New Compounds**

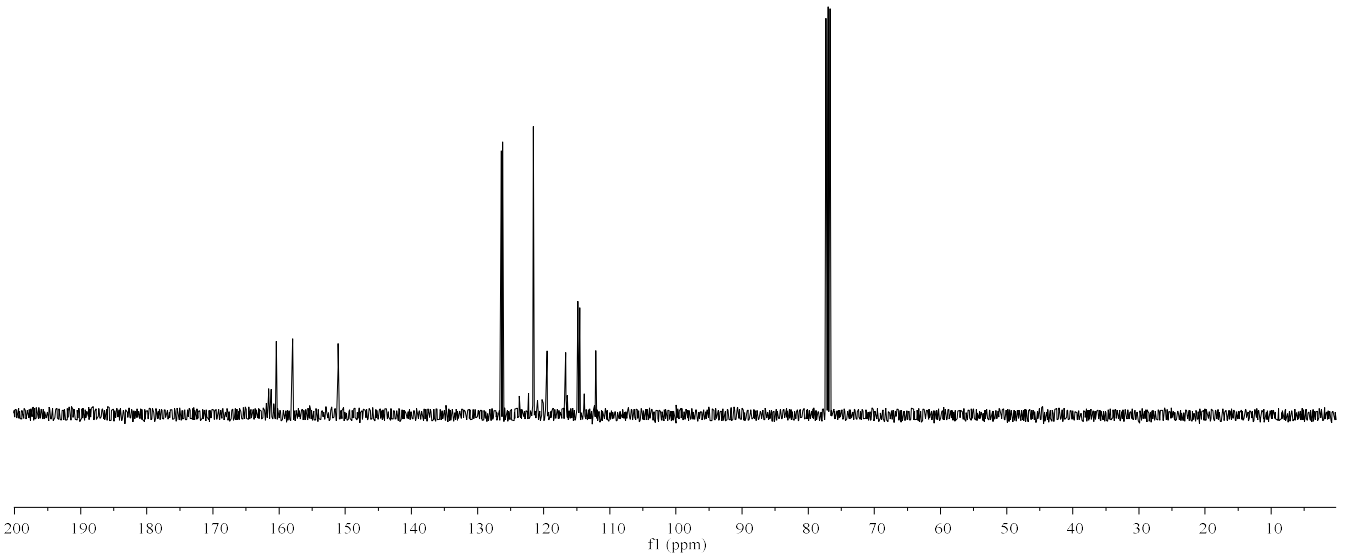

**1b**

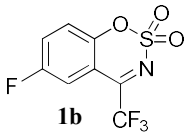


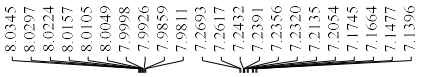

**1b**

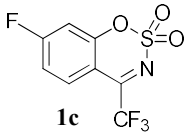



O
O
S
O
N
F
CF3

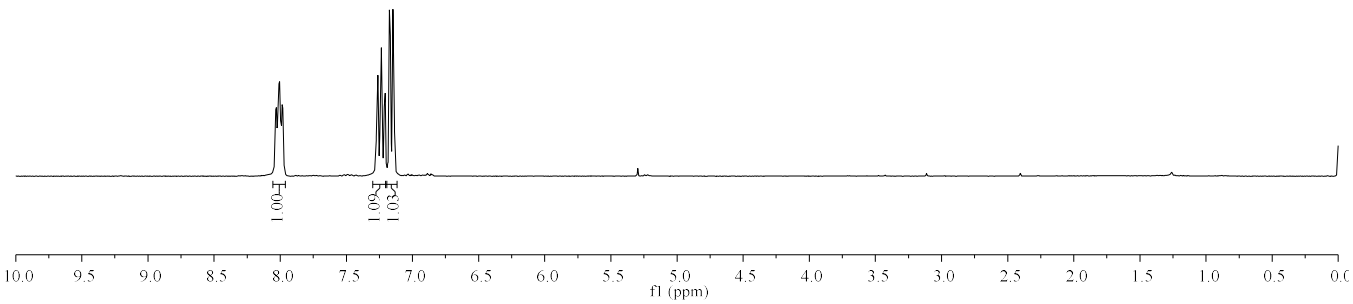
**1b**

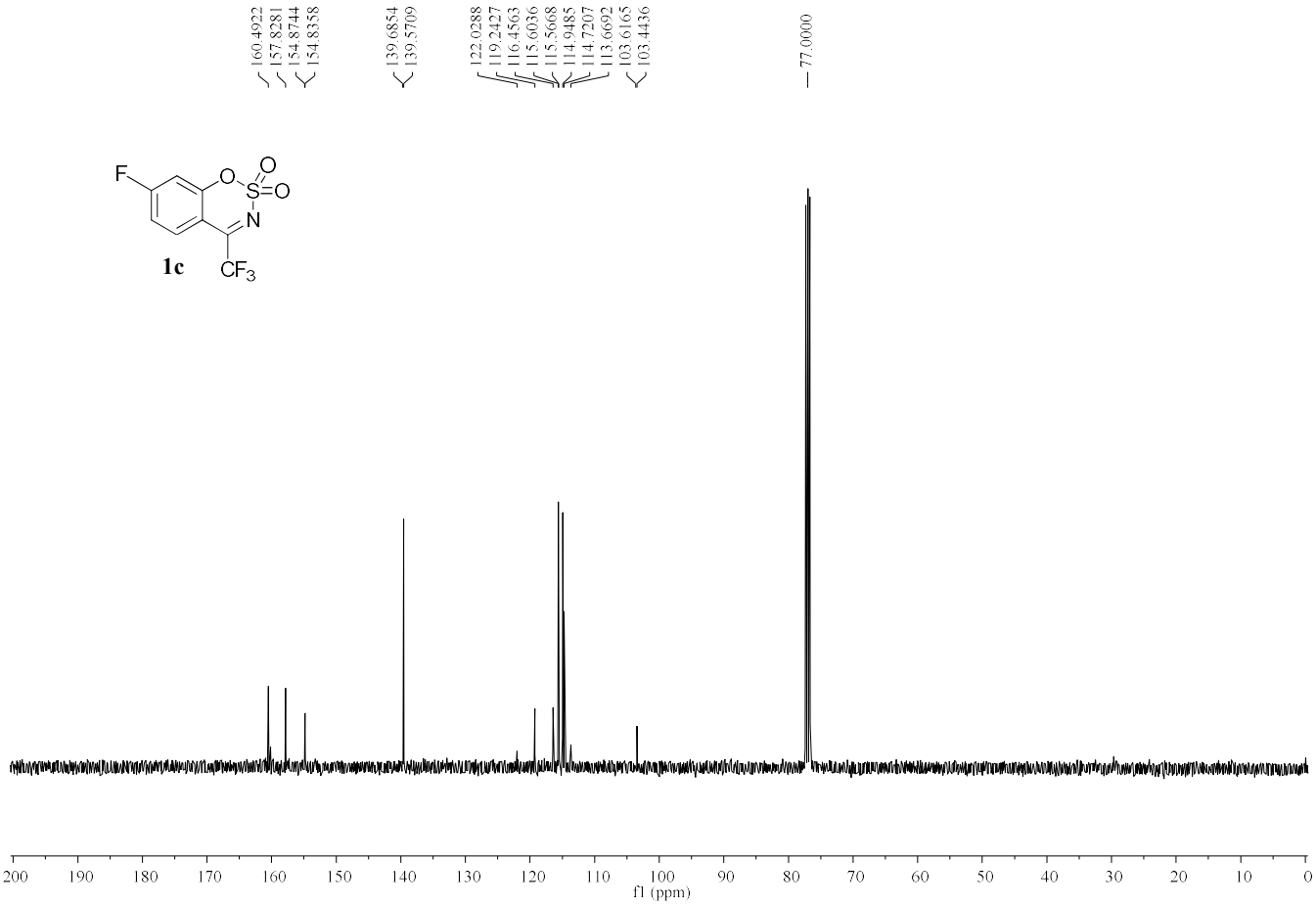

-67.3892
-110.8838
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

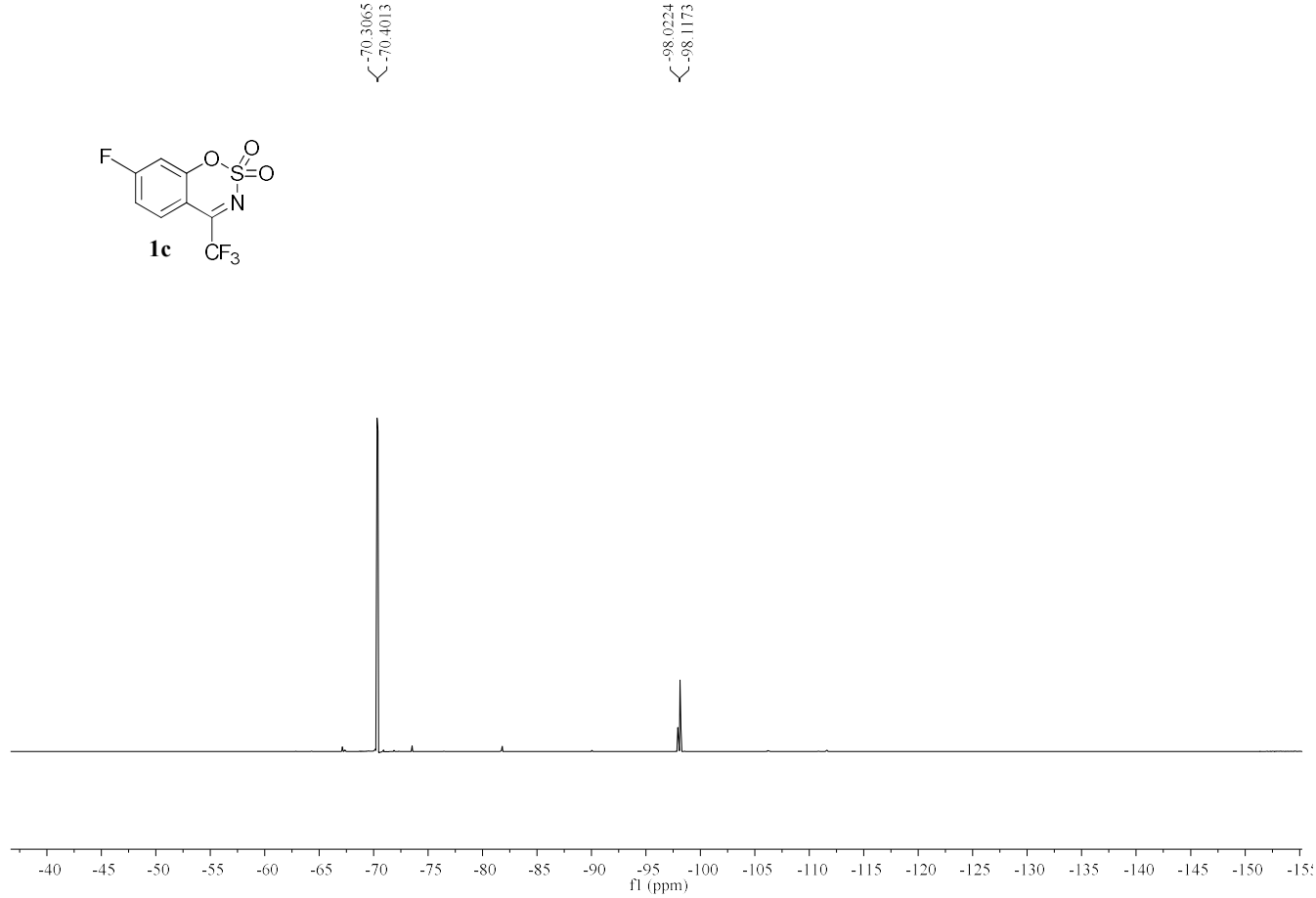

8.0345
8.0297
8.0224
8.0157
8.0105
8.0049
7.9998
7.9926
7.9859
7.9811
7.2693
7.2617
7.2432
7.2391
7.2356
7.2320
7.2135
7.2054
7.1745
7.1664
7.1477
7.1396

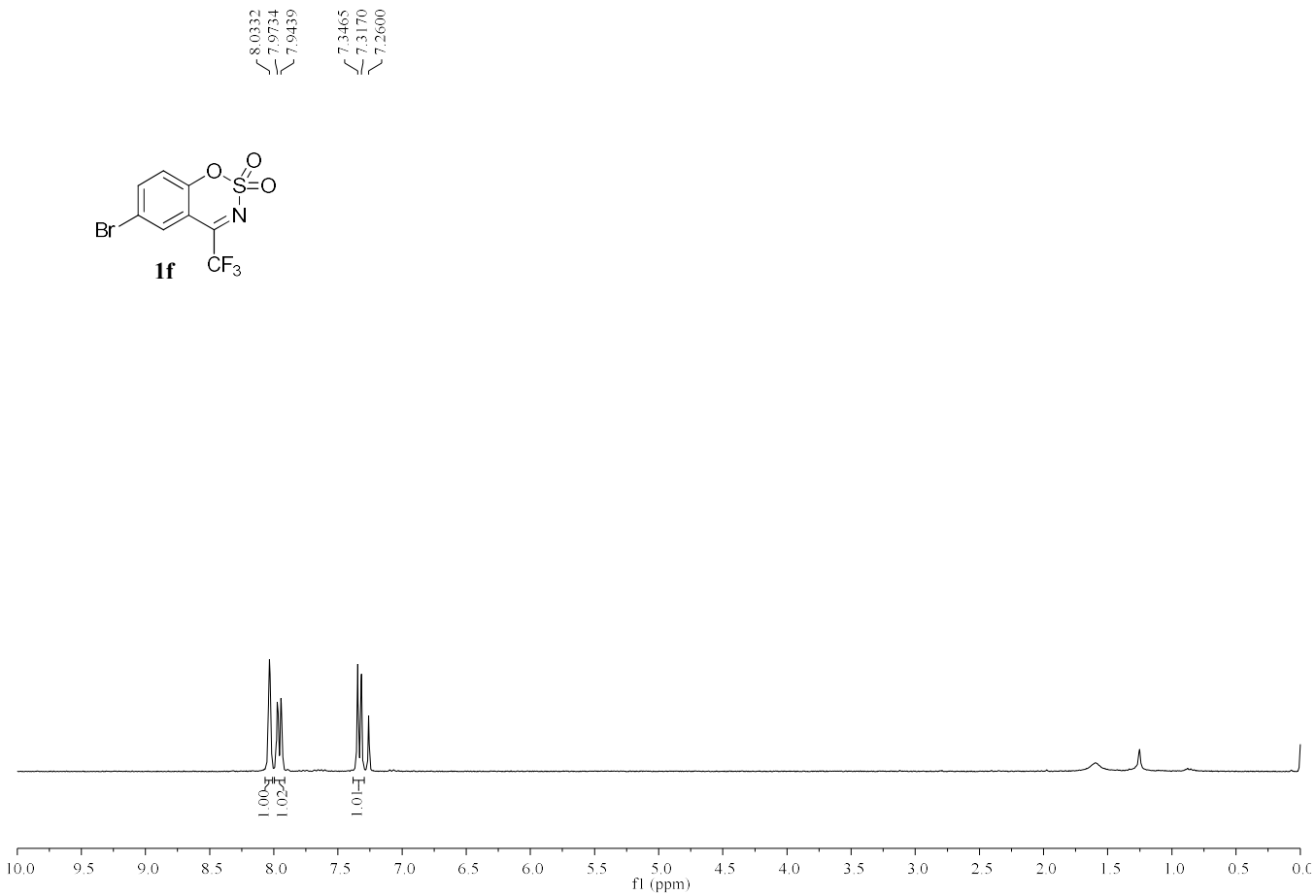

F
O
O
S
O
N
CF3

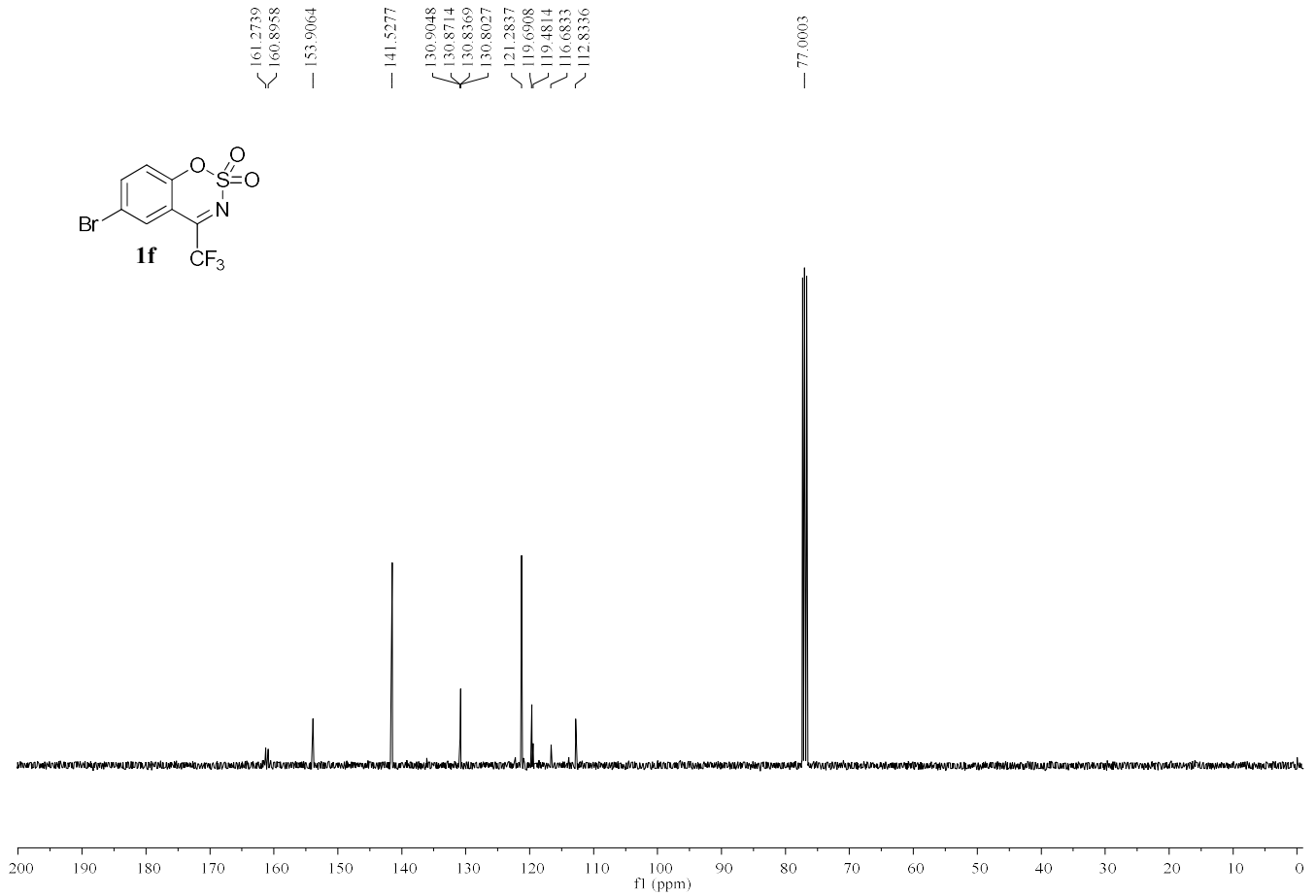
**1c**

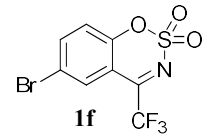

1.00
1.09
1.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


160.4922
157.8281
154.8744
154.8358
139.6854
139.5709
122.0288
119.2427
116.4563
115.6036
115.5668
114.9485
114.7207
113.6692
103.6165
103.4436
77.0000
F
O
S
O
O
N
CF3
1c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-70.3065
-70.4013
-98.0224
-98.1173
F
O
S
O
O
N
CF3
1c
-40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150
f1 (ppm)

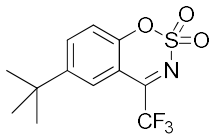

8.0332
7.9734
7.9439
7.3465
7.3170
7.2600
Br
CF3
1f
1.00
1.02
1.01
fl (ppm)


161.2739
160.8958
153.9064
141.5277
130.9048
130.8714
130.8369
130.8027
121.2837
119.6908
119.4814
116.6833
112.8336
77.0003
Br
CF3
1f
fl (ppm)



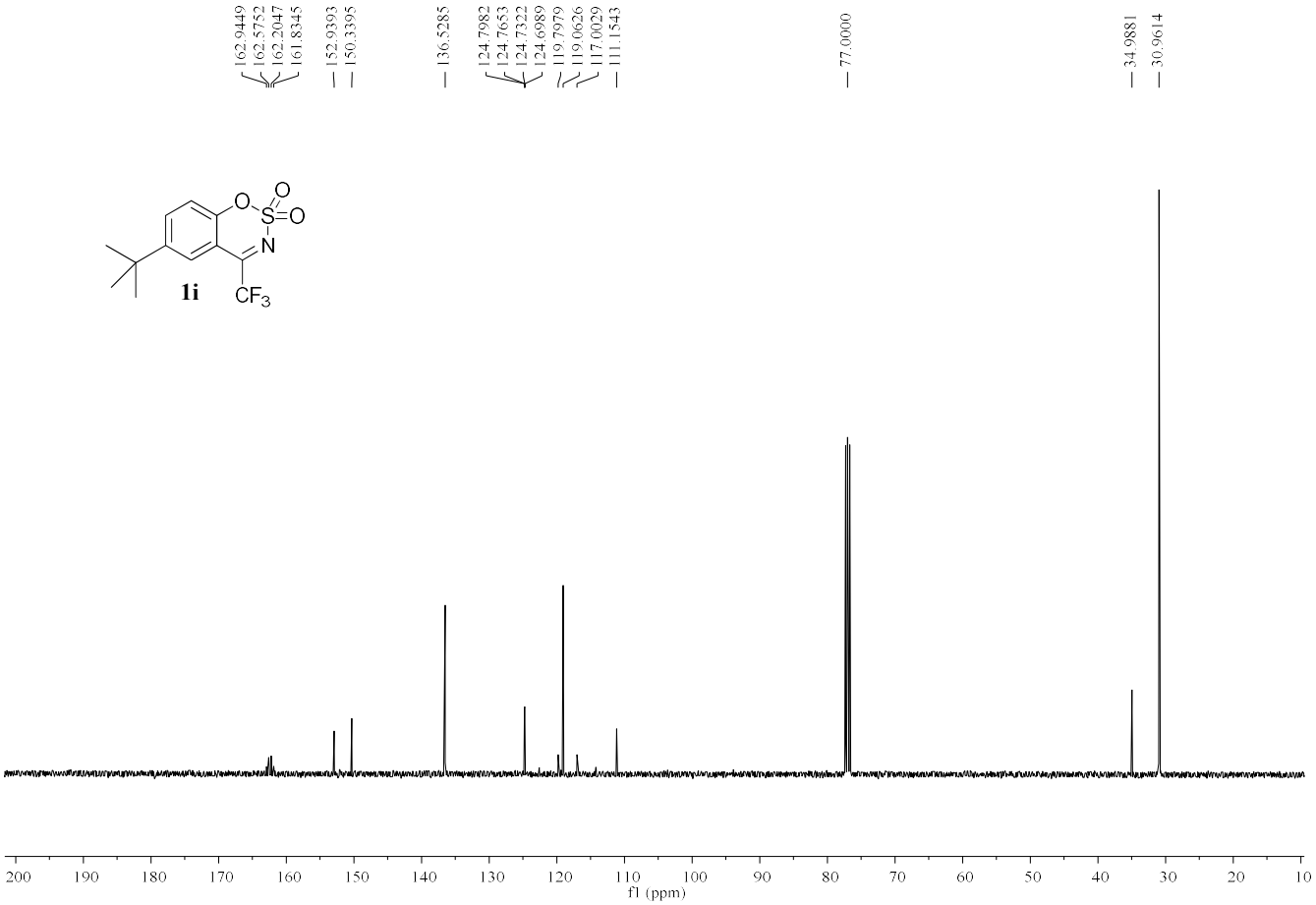
**1f**

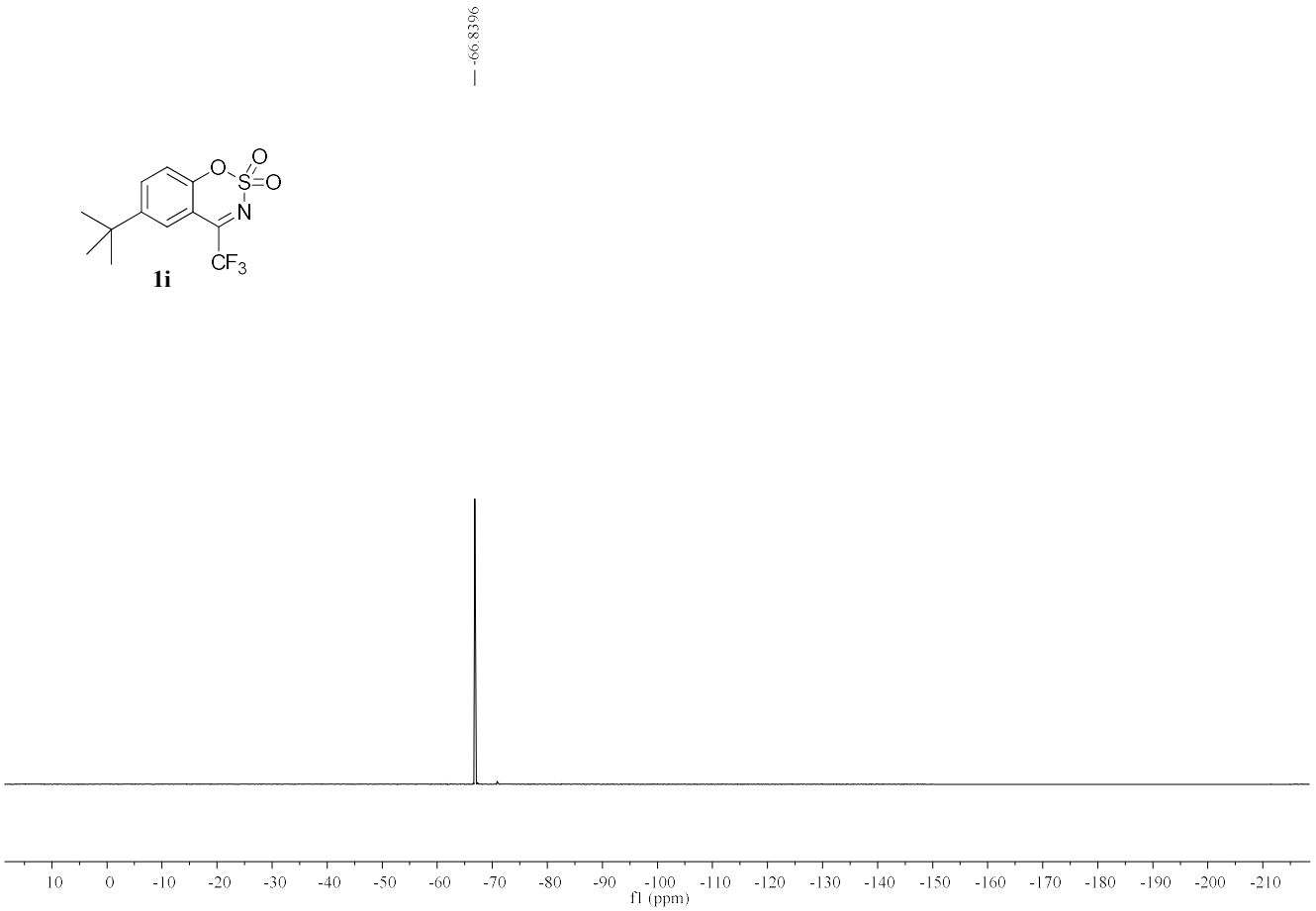

-67.0364
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

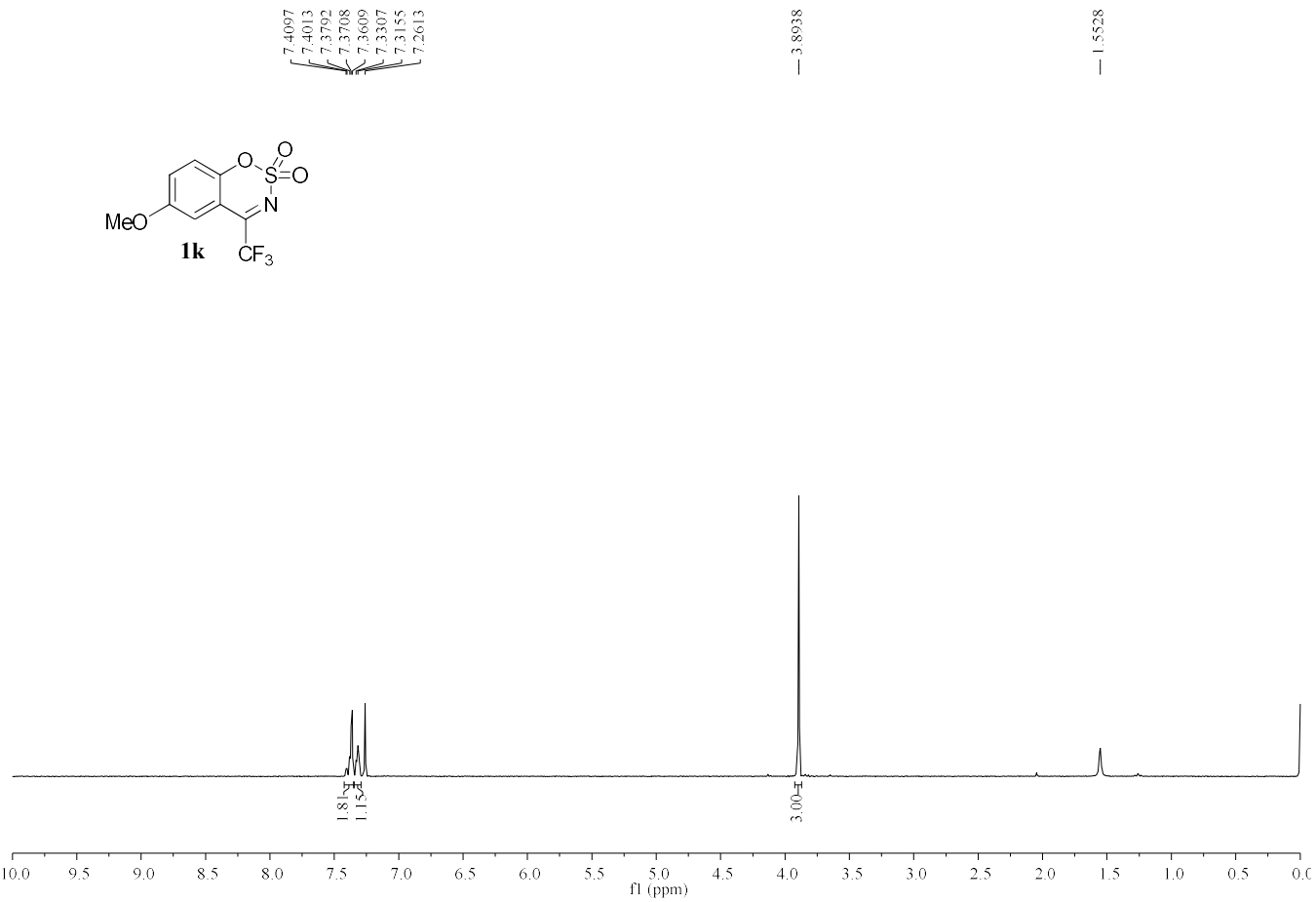


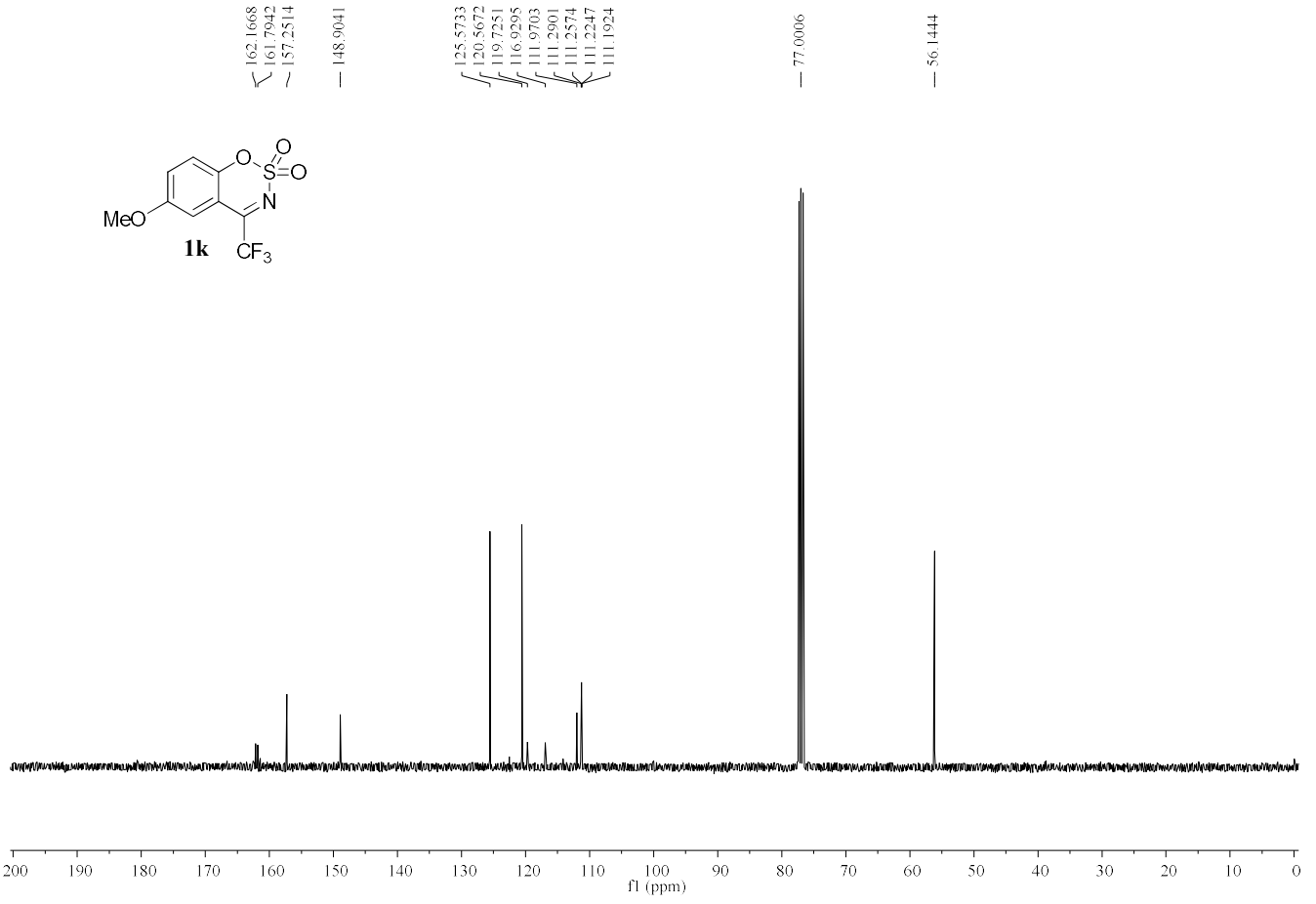
**1i**

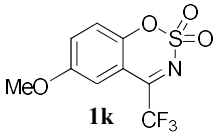

7.8970
7.8854
7.8789
7.8734
7.3634
7.3320
7.2603
1.3713
1.99
1.00
8.90
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


162.9449
162.5752
162.2047
161.8345
152.9393
150.3395
136.5285
124.7982
124.7653
124.7322
124.6989
119.7979
119.0626
117.0029
111.1543
77.0000
34.9881
30.9614
1i
CF3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


-66.8396
1i
CF3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

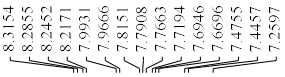

7.4097
7.4013
7.3792
7.3708
7.3609
7.3307
7.3155
7.2613
3.8938
1.5528
MeO
1k
CF3
1.81
1.15
3.00
f1 (ppm)


162.1668
161.7942
157.2514
148.9041
125.5733
120.5672
119.7251
116.9295
111.9703
111.2901
111.2574
111.2247
111.1924
77.0006
56.1444
MeO
1k
CF3
f1 (ppm)

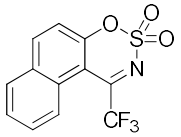

MeO
O
S
O
O
N
CF3

**1k**

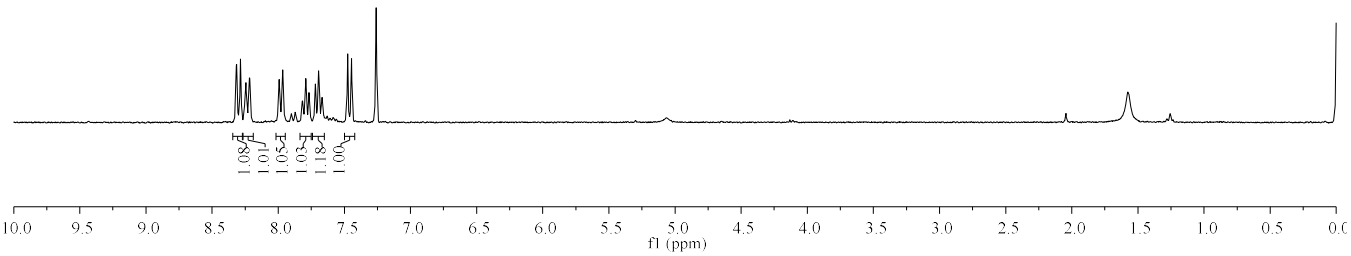

-67.1430
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

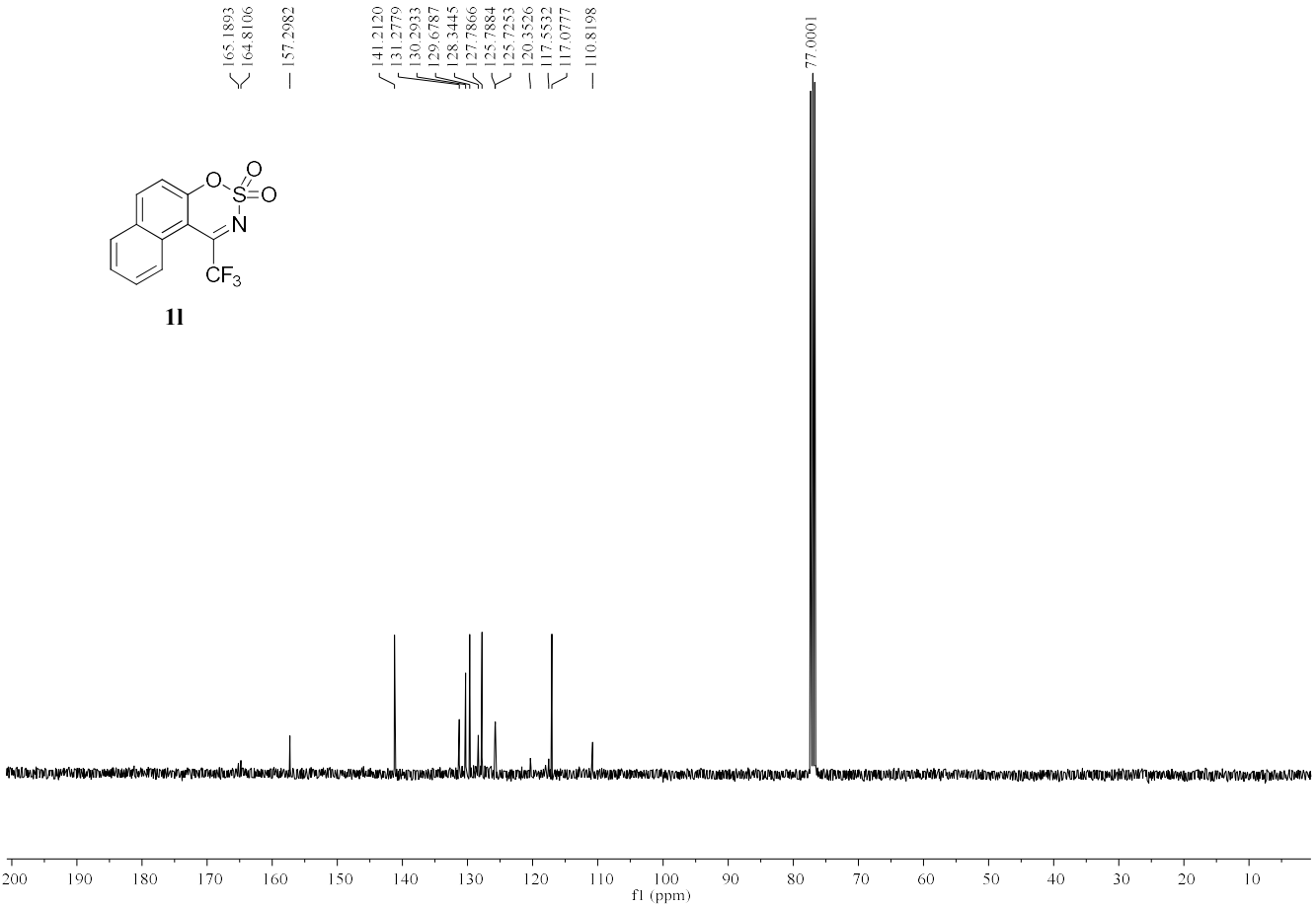

8.3154
8.2855
8.2452
8.2171
7.9931
7.9666
7.8151
7.7908
7.7663
7.7194
7.6946
7.6696
7.4755
7.4457
7.2597


1.5764

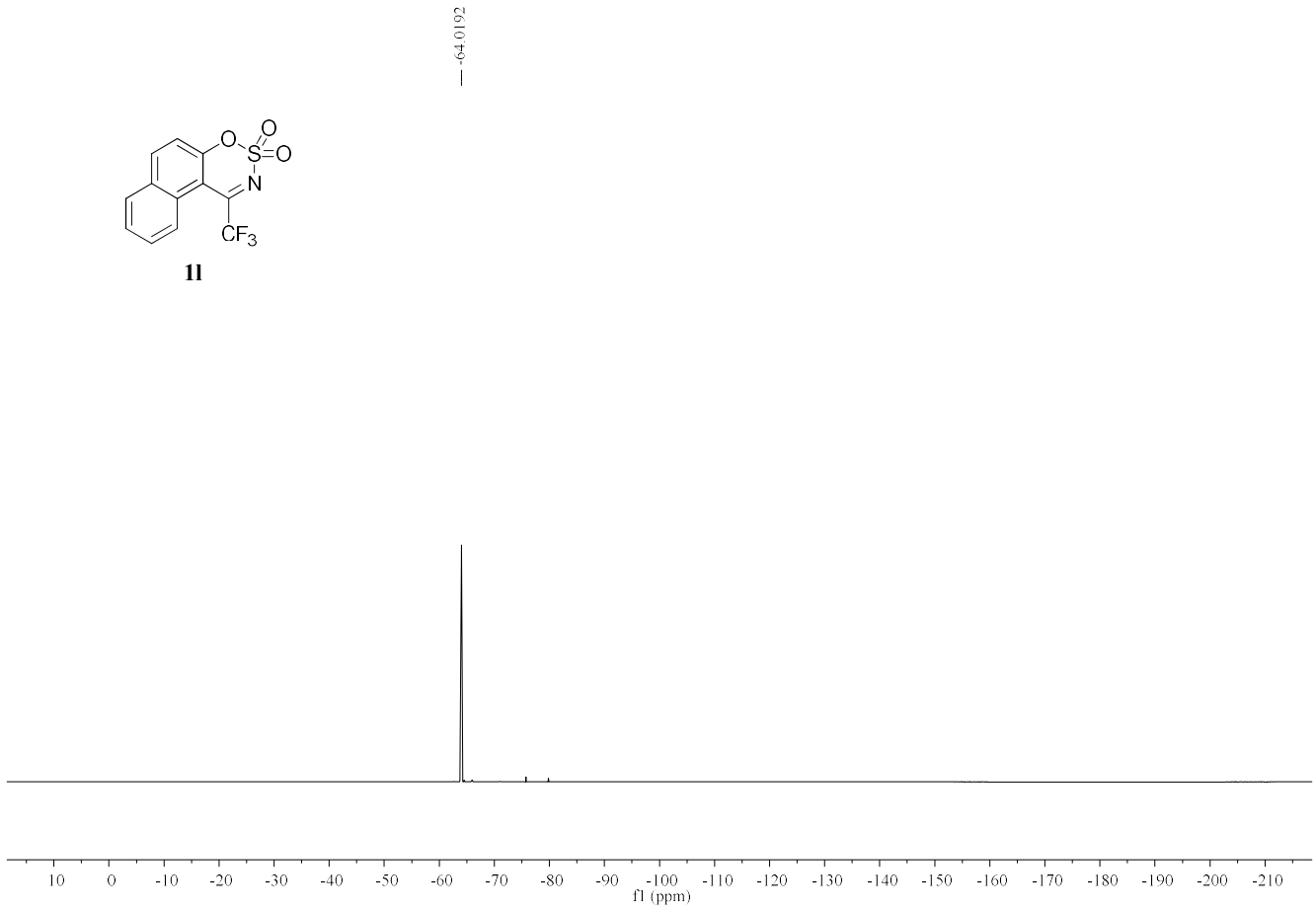

O
S
O
O
N
CF3

**1l**

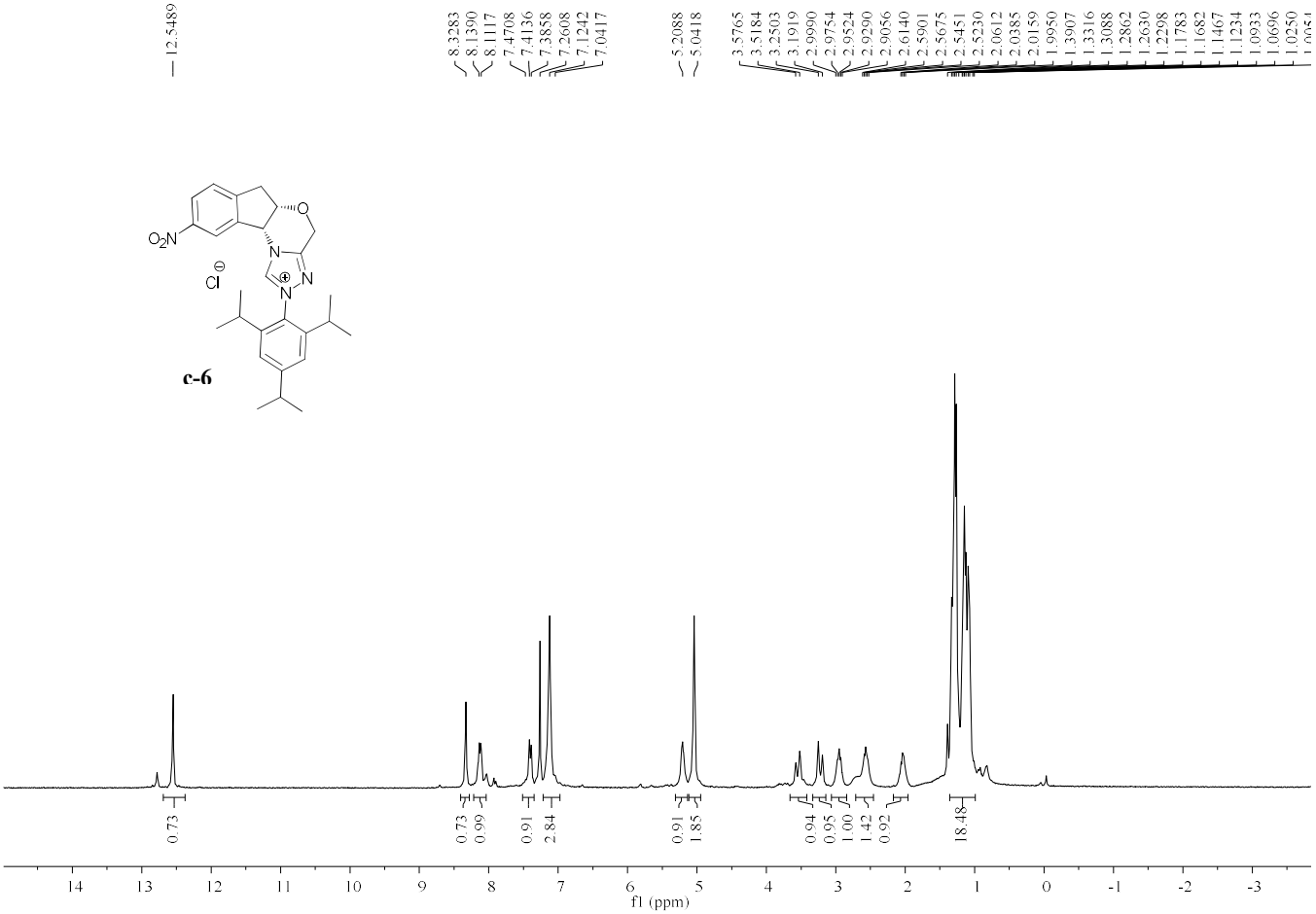

1.08
1.01
1.05
1.03
1.18
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

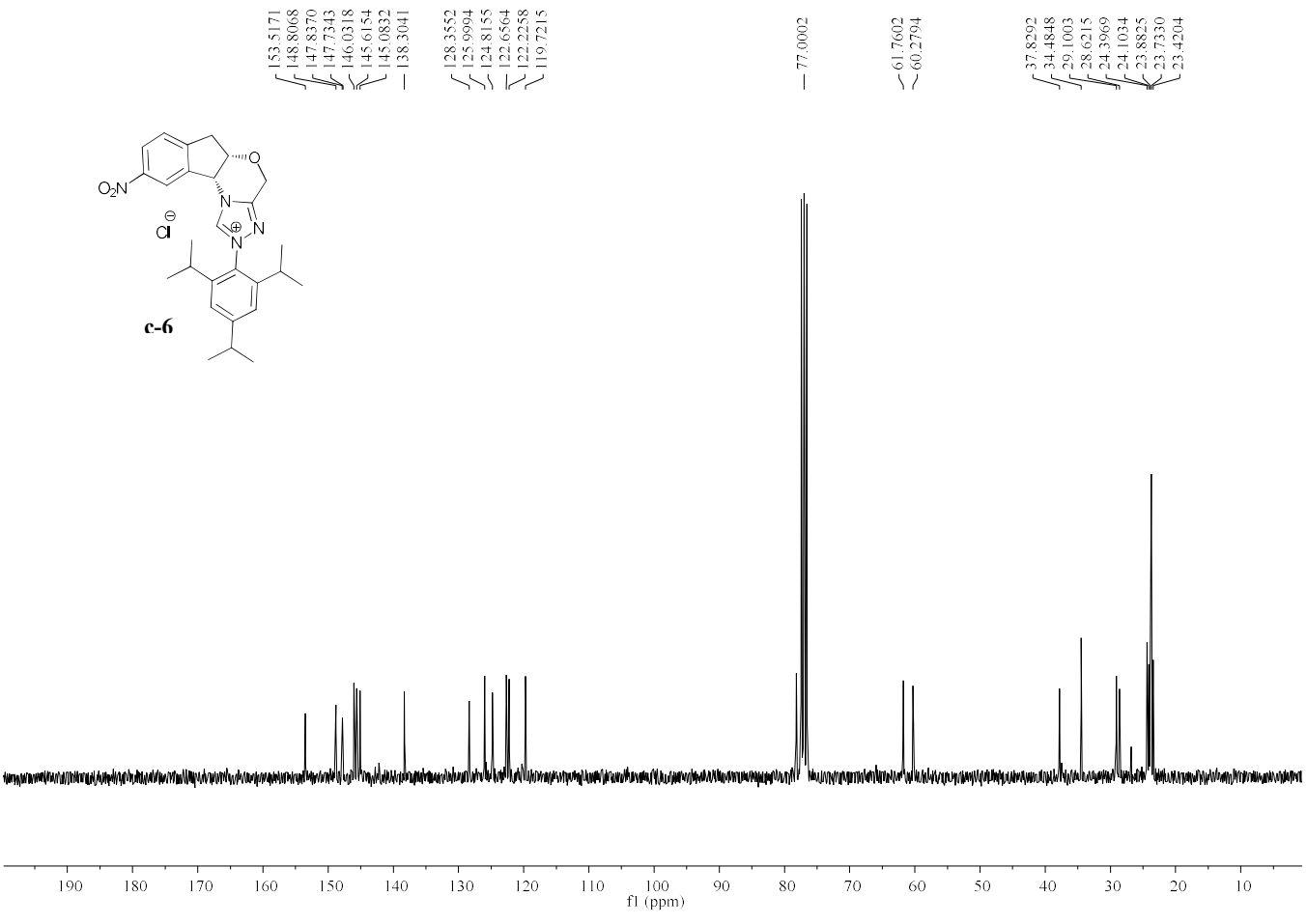

165.1893
164.8106
157.2982
141.2120
131.2779
130.2933
129.6787
128.3445
127.7866
125.7884
125.7253
120.3526
117.5532
117.0777
110.8198
77.0001
O
S
O
N
CF3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

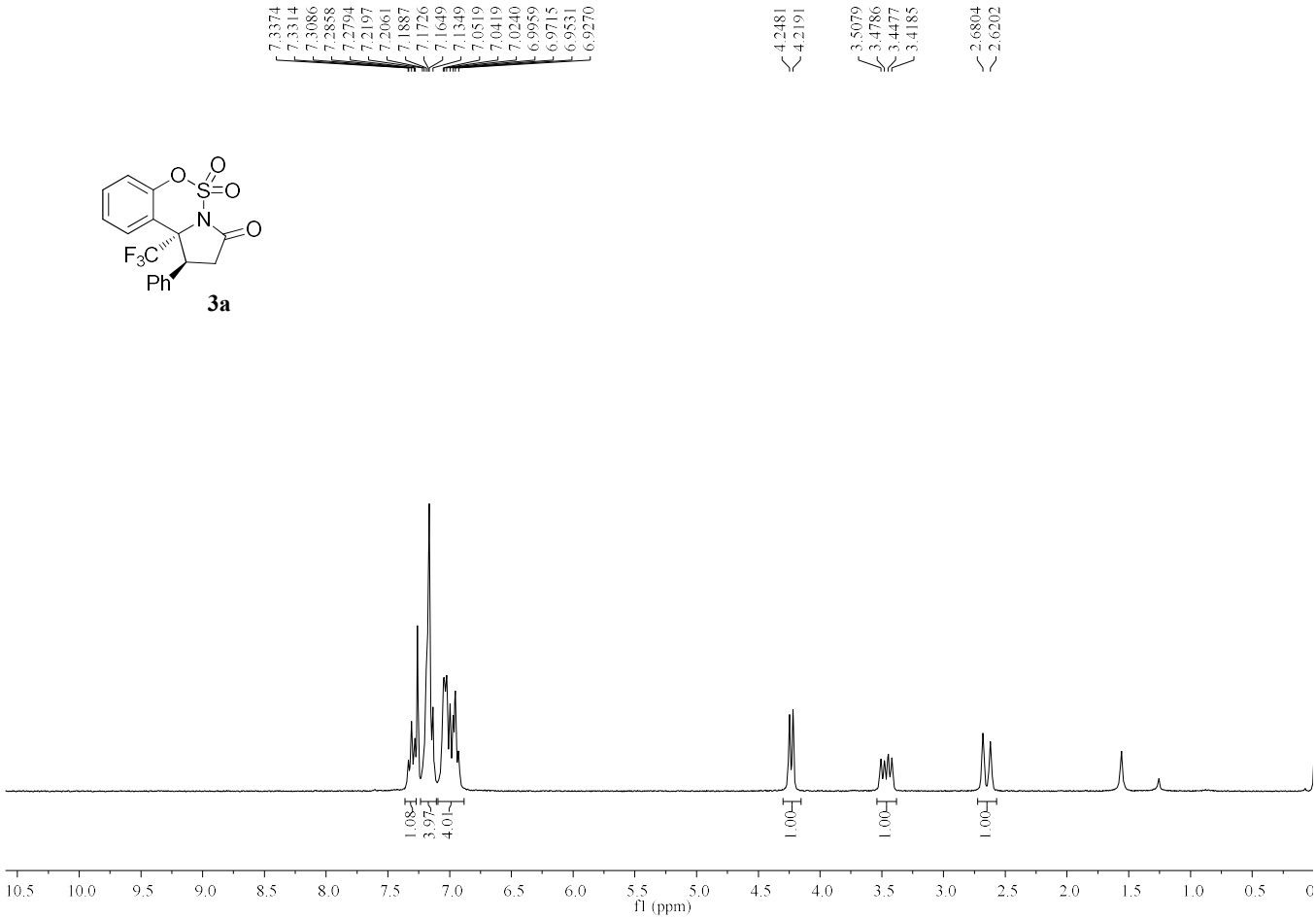
**1l**

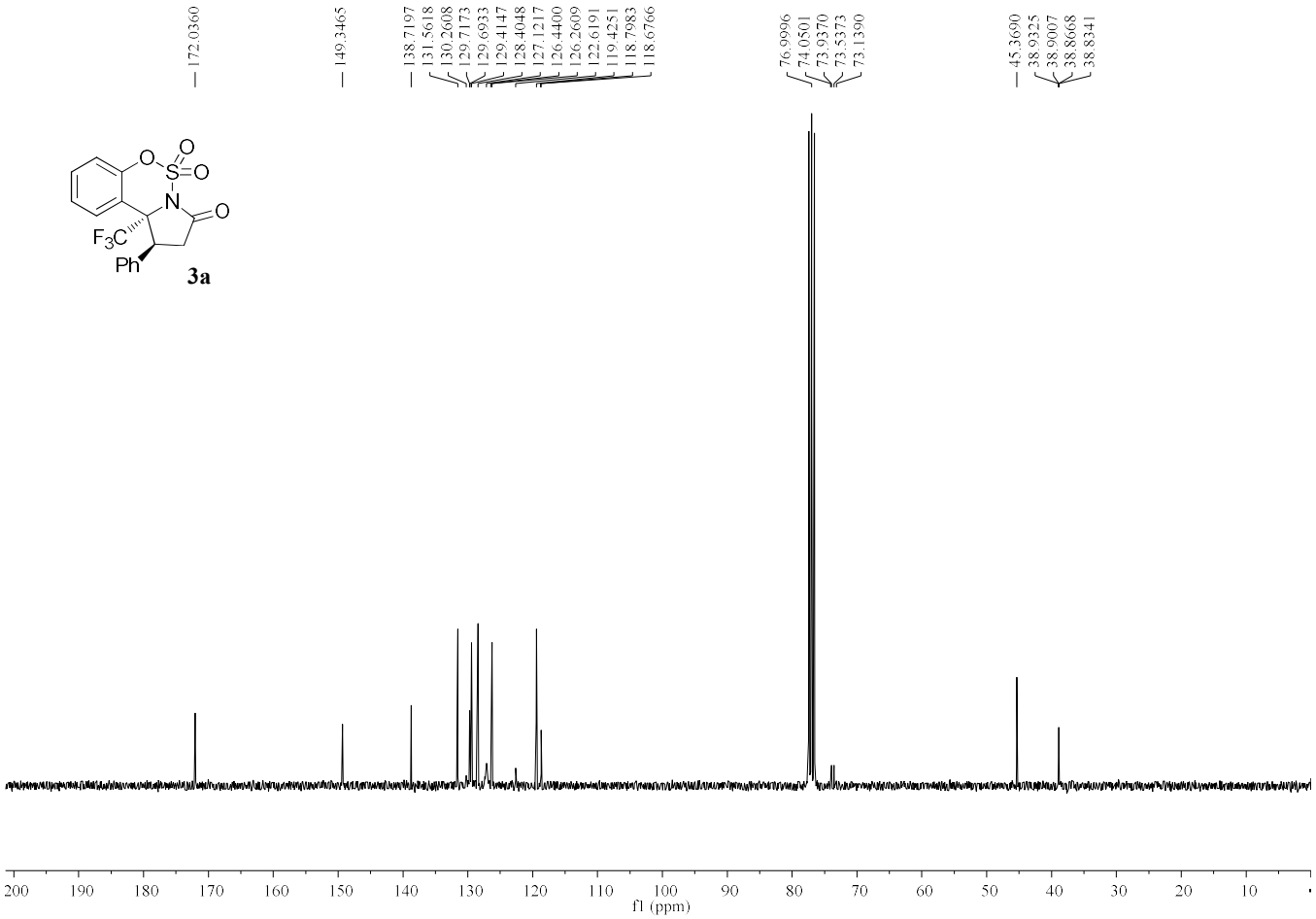

-64.0192
O
S
O
N
CF3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

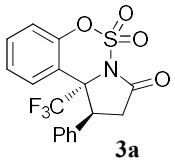
**1l**


12.5489
8.3283
8.1390
8.1117
7.4708
7.4136
7.3858
7.2608
7.1242
7.0417
5.2088
5.0418
3.5765
3.5184
3.2503
3.1919
2.9990
2.9754
2.9524
2.9290
2.9056
2.6140
2.5901
2.5675
2.5451
2.5230
2.0612
2.0385
2.0159
1.9950
1.3907
1.3316
1.3088
1.2862
1.2630
1.2298
1.1783
1.1682
1.1467
1.1234
1.0933
1.0696
1.0250
O2N
Cl
c-6
0.73
0.73
0.99
0.91
2.84
0.91
1.85
0.94
0.95
1.00
1.42
0.92
18.48
f1 (ppm)


153.5171
148.8068
147.8370
147.7343
146.0318
145.6154
145.0832
138.3041
128.3552
125.9994
124.8155
122.6564
122.2258
119.7215
77.0002
61.7602
60.2794
37.8292
34.4848
29.1003
28.6215
24.3969
24.1034
23.8825
23.7330
23.4204
O2N
Cl
c-6
f1 (ppm)

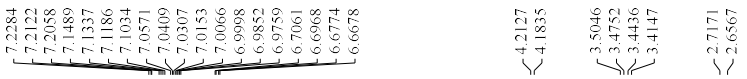

7.3374
7.3314
7.3086
7.2858
7.2794
7.2197
7.2061
7.1887
7.1726
7.1649
7.1349
7.0519
7.0419
7.0240
6.9959
6.9715
6.9531
6.9270
4.2481
4.2191
3.5079
3.4786
3.4477
3.4185
2.6804
2.6202
3a
1.08
3.97
4.01
1.00
1.00
1.00
f1 (ppm)

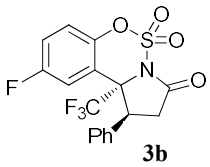

172.0360
149.3465
138.7197
131.5618
130.2608
129.7173
129.6933
129.4147
128.4048
127.1217
126.4400
126.2609
122.6191
119.4251
118.7983
118.6766
76.9996
74.0501
73.9370
73.5373
73.1390
45.3690
38.9325
38.9007
38.8668
38.8341
3a
f1 (ppm)



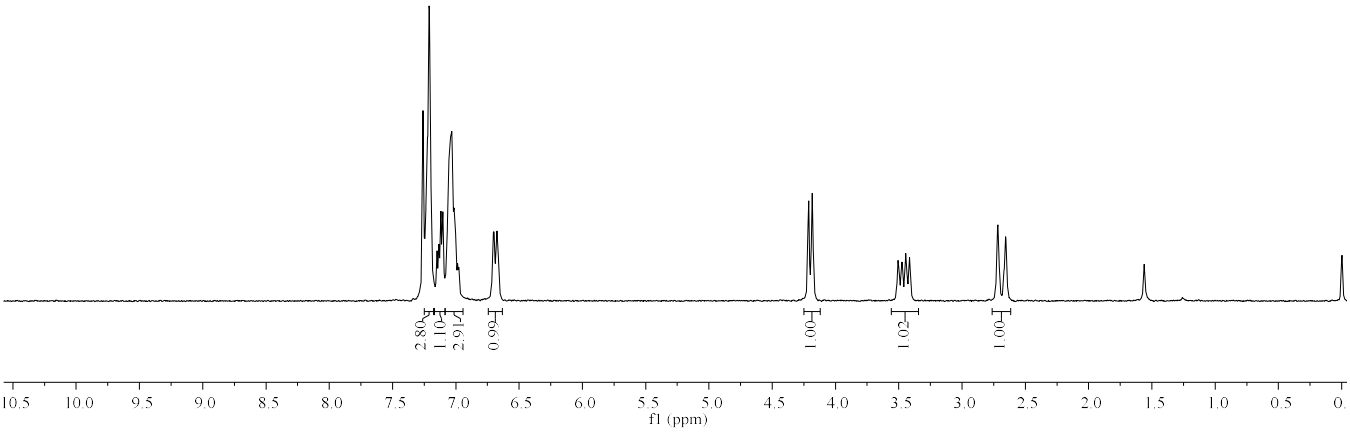
**3a**

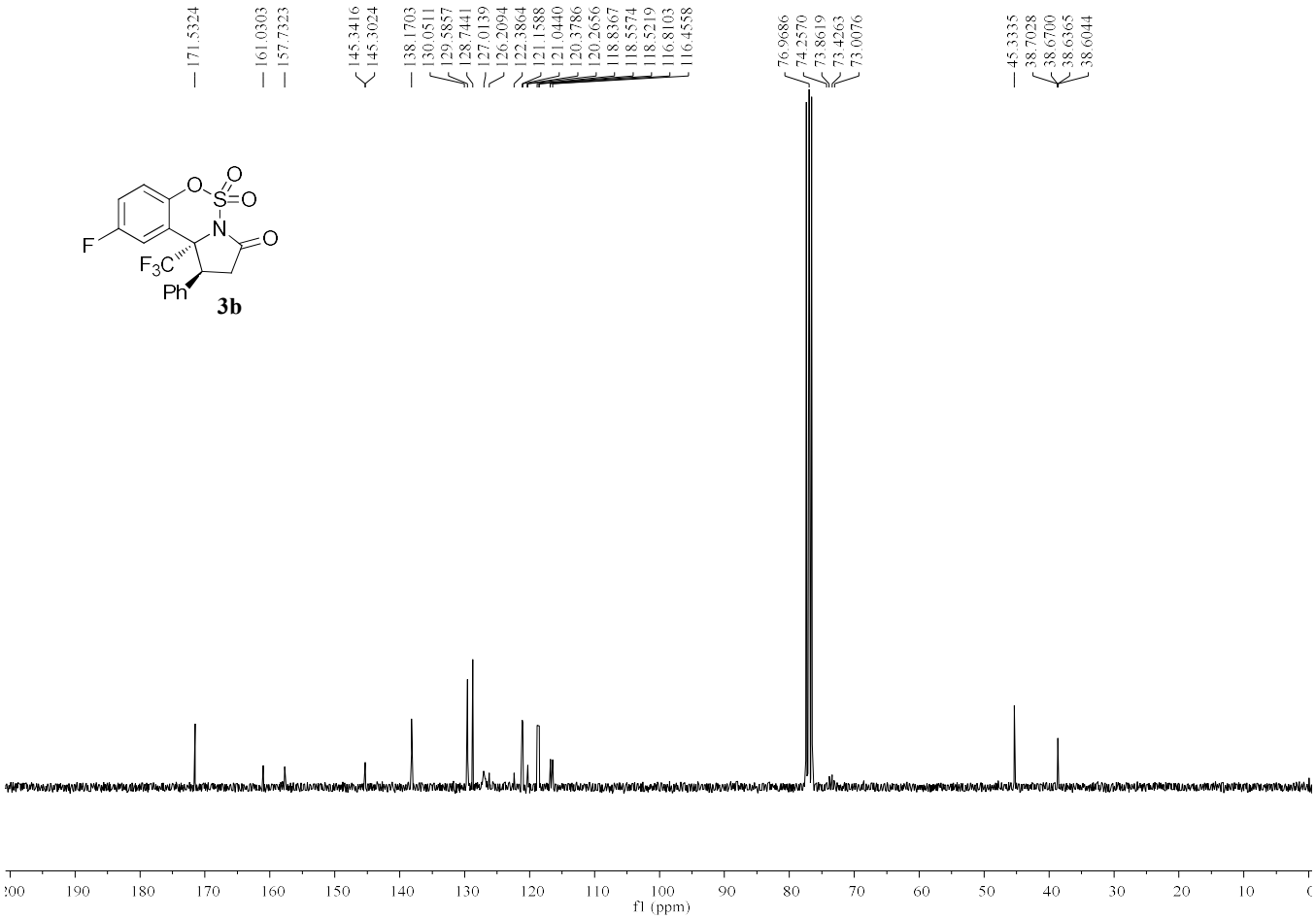

-76.6845
f1 (ppm)

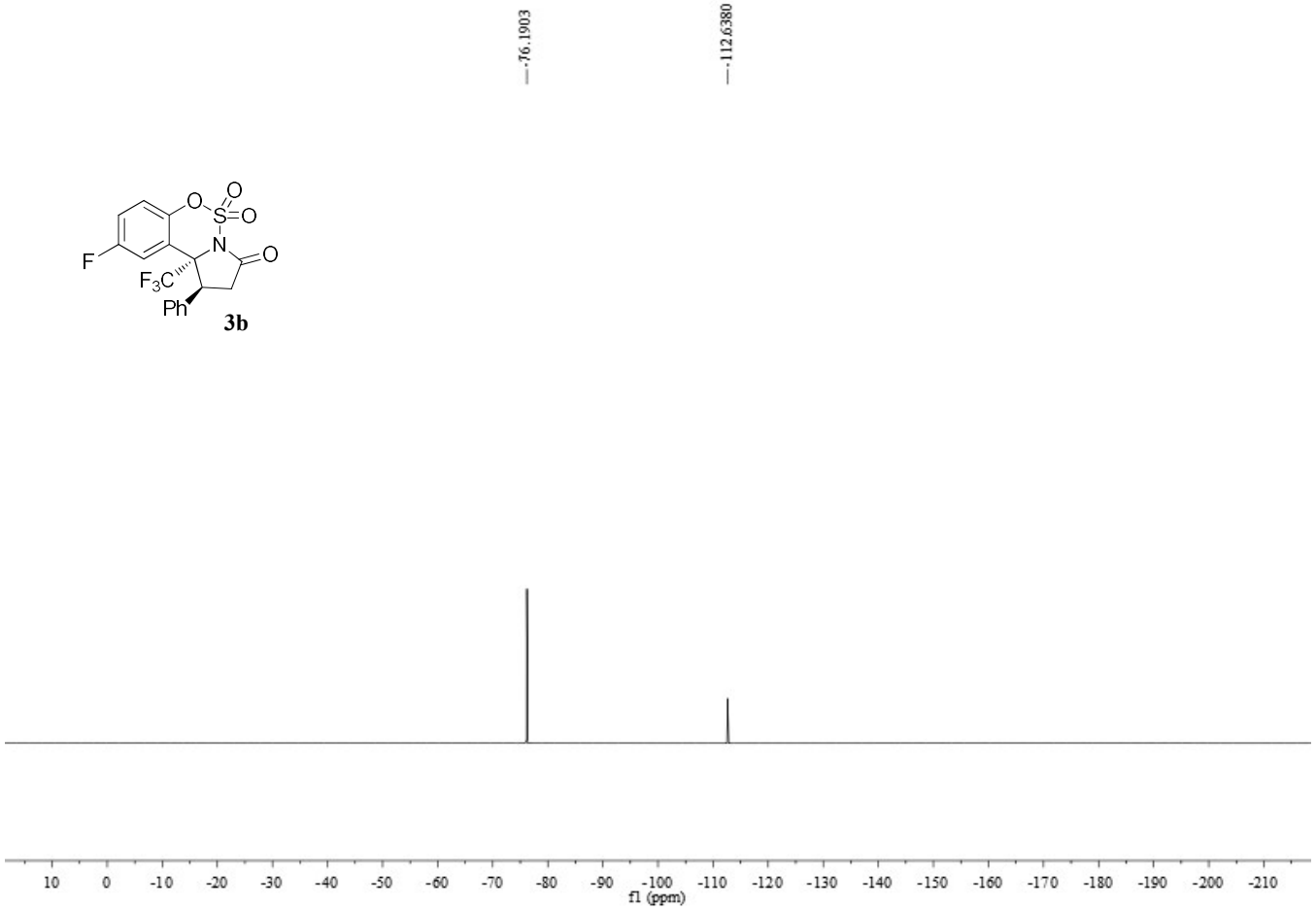

7.2284
7.2122
7.2058
7.1489
7.1337
7.1186
7.1034
7.0571
7.0409
7.0307
7.0153
7.0066
6.9998
6.9852
6.9759
6.7061
6.6968
6.6774
6.6678
4.2127
4.1835
3.5046
3.4752
3.4436
3.4147
2.7171
2.6567

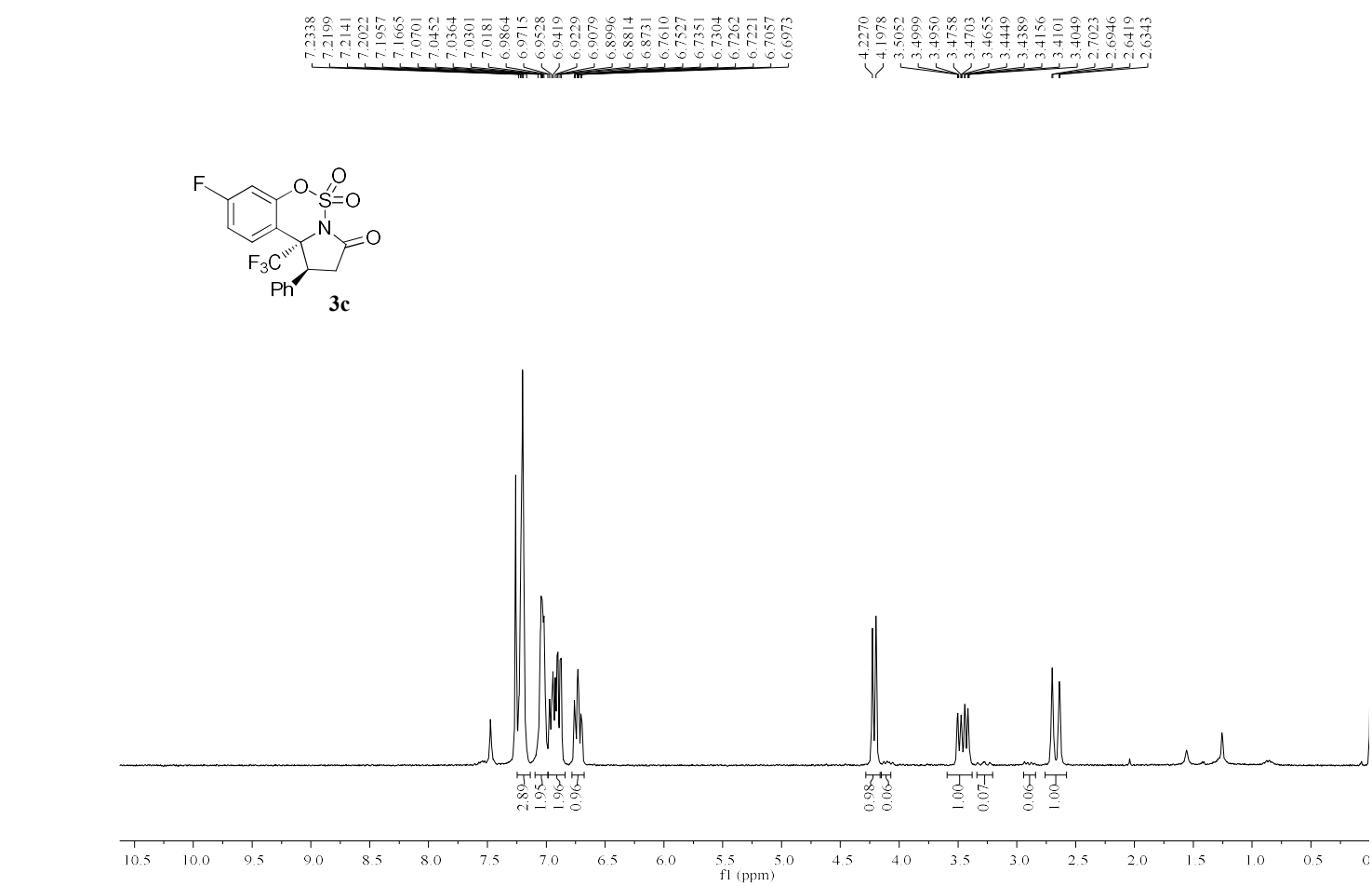


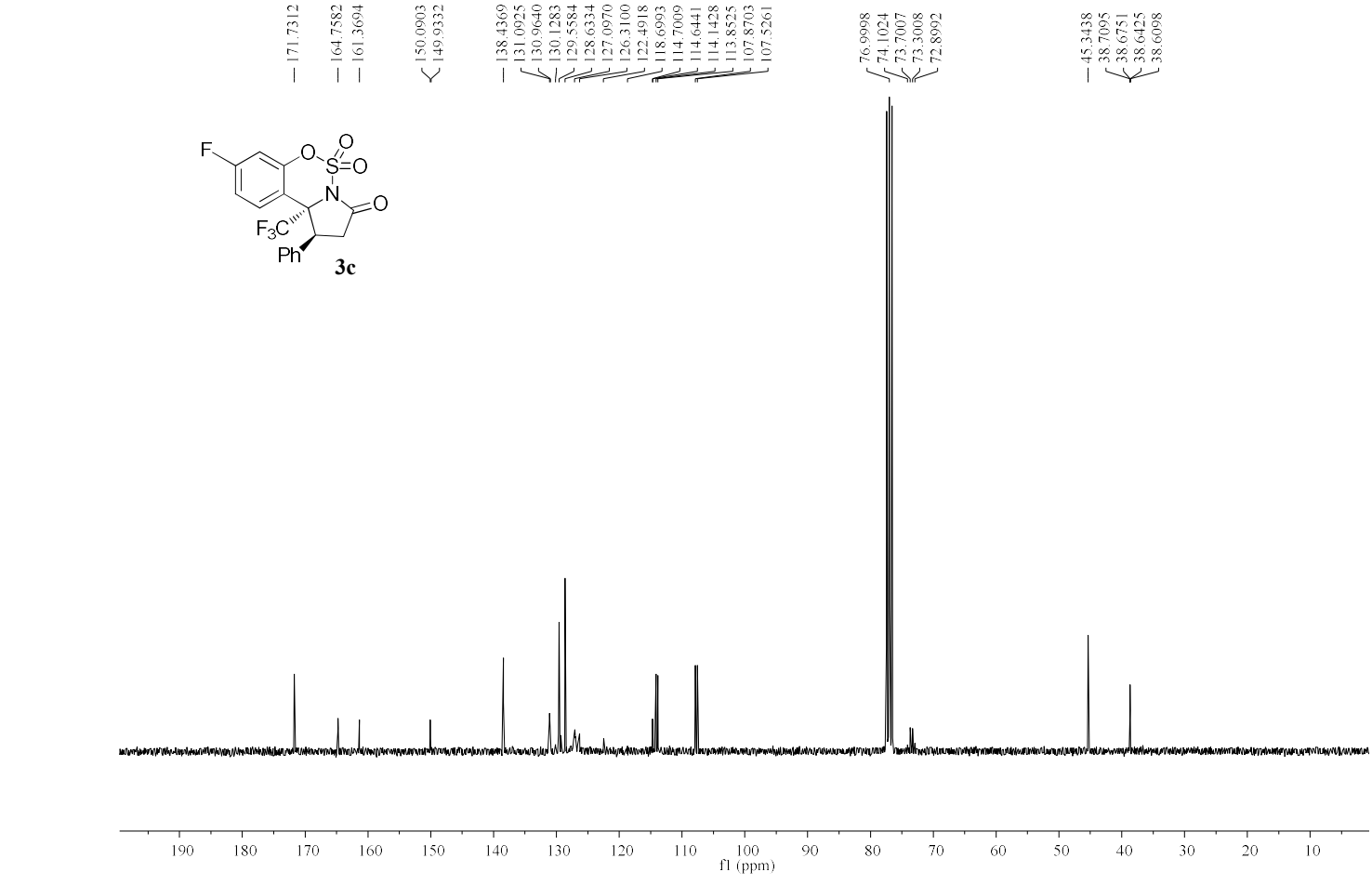
**3b**

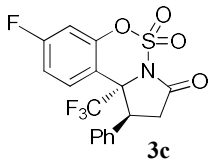

2.80
1.10
2.91
0.99
1.00
1.02
1.00
f1 (ppm)


171.5324
161.0303
157.7323
145.3416
145.3024
138.1703
130.0511
129.5857
128.7441
127.0139
126.2094
122.3864
121.1588
121.0440
120.3786
120.2656
118.8367
118.5574
118.5219
116.8103
116.4558
76.9686
74.2570
73.8619
73.4263
73.0076
45.3335
38.7028
38.6700
38.6365
38.6044
O
S
O
N
O
F
F3C
Ph
3b
f1 (ppm)


-76.1903
-112.6380
O
S
O
N
O
F
F3C
Ph
3b
f1 (ppm)

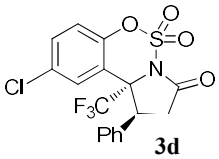

7.2338
7.2199
7.2141
7.2022
7.1957
7.1665
7.0701
7.0452
7.0364
7.0301
7.0181
6.9864
6.9715
6.9528
6.9419
6.9229
6.9079
6.8996
6.8814
6.8731
6.7610
6.7527
6.7351
6.7304
6.7262
6.7221
6.7057
6.6973
4.2270
4.1978
3.5052
3.4999
3.4950
3.4758
3.4703
3.4655
3.4449
3.4389
3.4156
3.4101
3.4049
2.7023
2.6946
2.6419
2.6343
F
O
S
O
O
N
O
F3C
Ph
3c
2.89
1.95
1.96
0.96
0.98
0.06
1.00
0.07
0.06
1.00
f1 (ppm)


171.7312
164.7582
161.3694
150.0903
149.9332
138.4369
131.0925
130.9640
130.1283
129.5584
128.6334
127.0970
126.3100
122.4918
118.6993
114.7009
114.6441
114.1428
113.8525
107.8703
107.5261
76.9998
74.1024
73.7007
73.3008
72.8992
45.3438
38.7095
38.6751
38.6425
38.6098
F
O
S
O
O
N
O
F3C
Ph
3c
f1 (ppm)



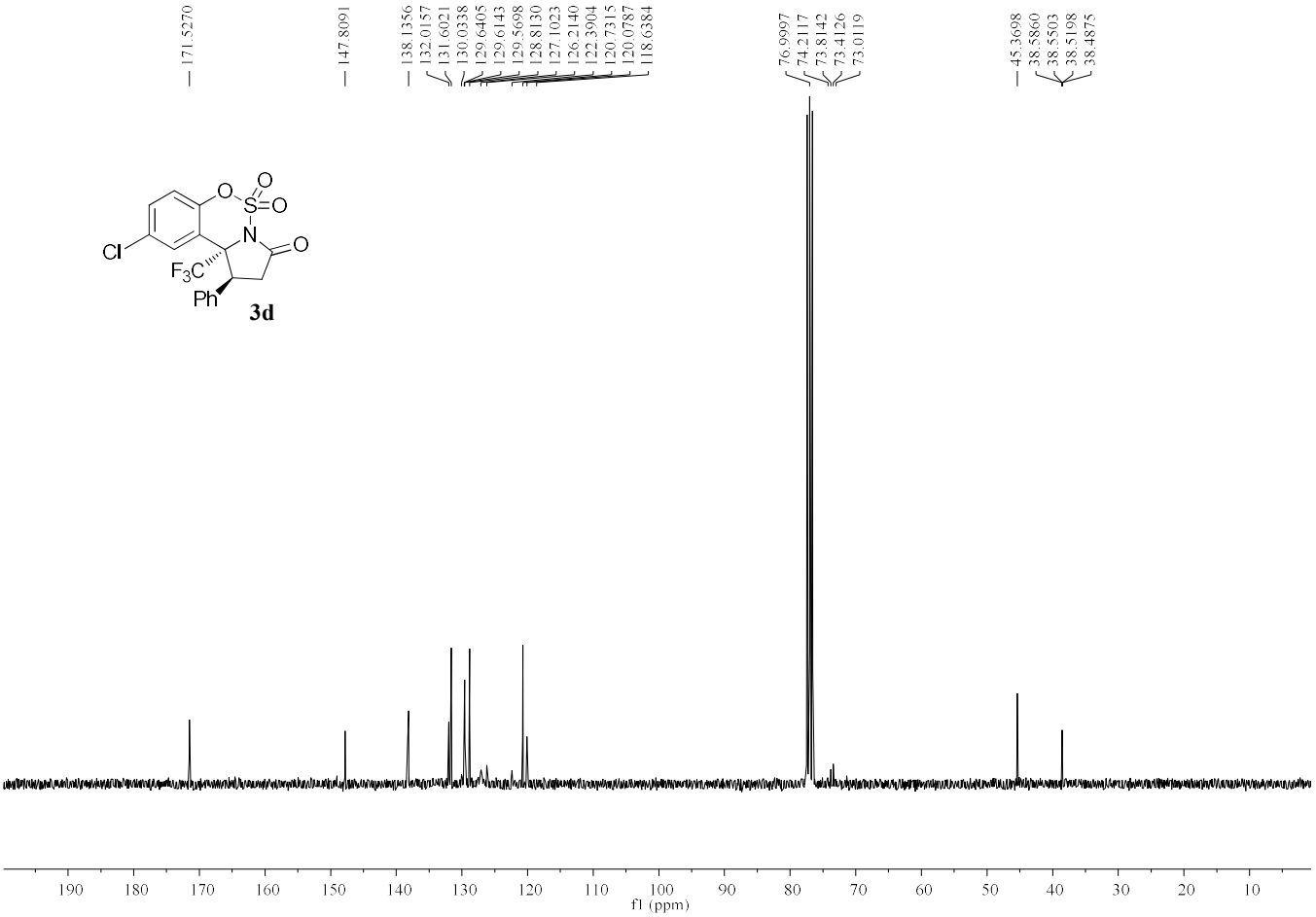
**3c**

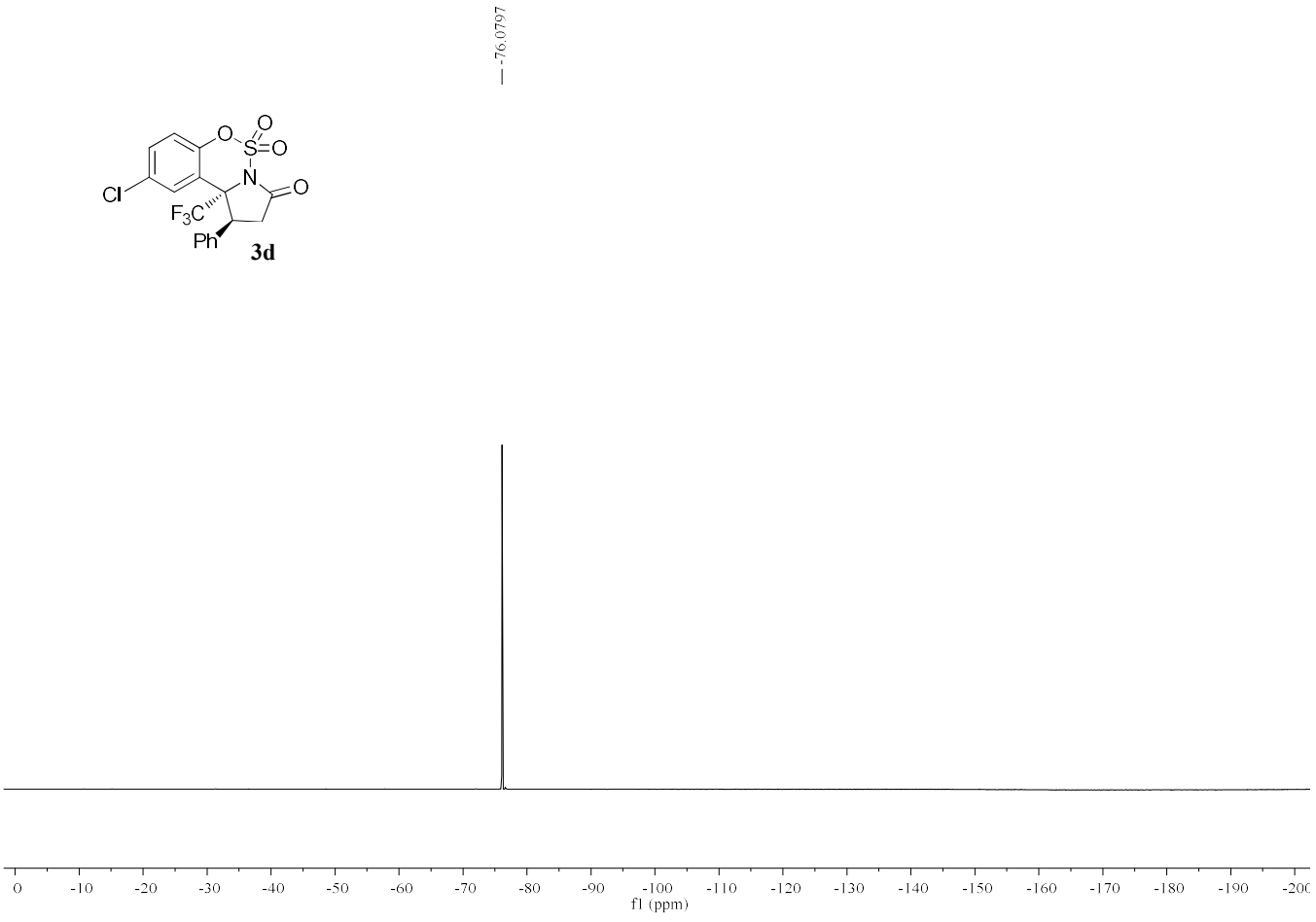

-70.4062
-76.6862
-105.0406
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

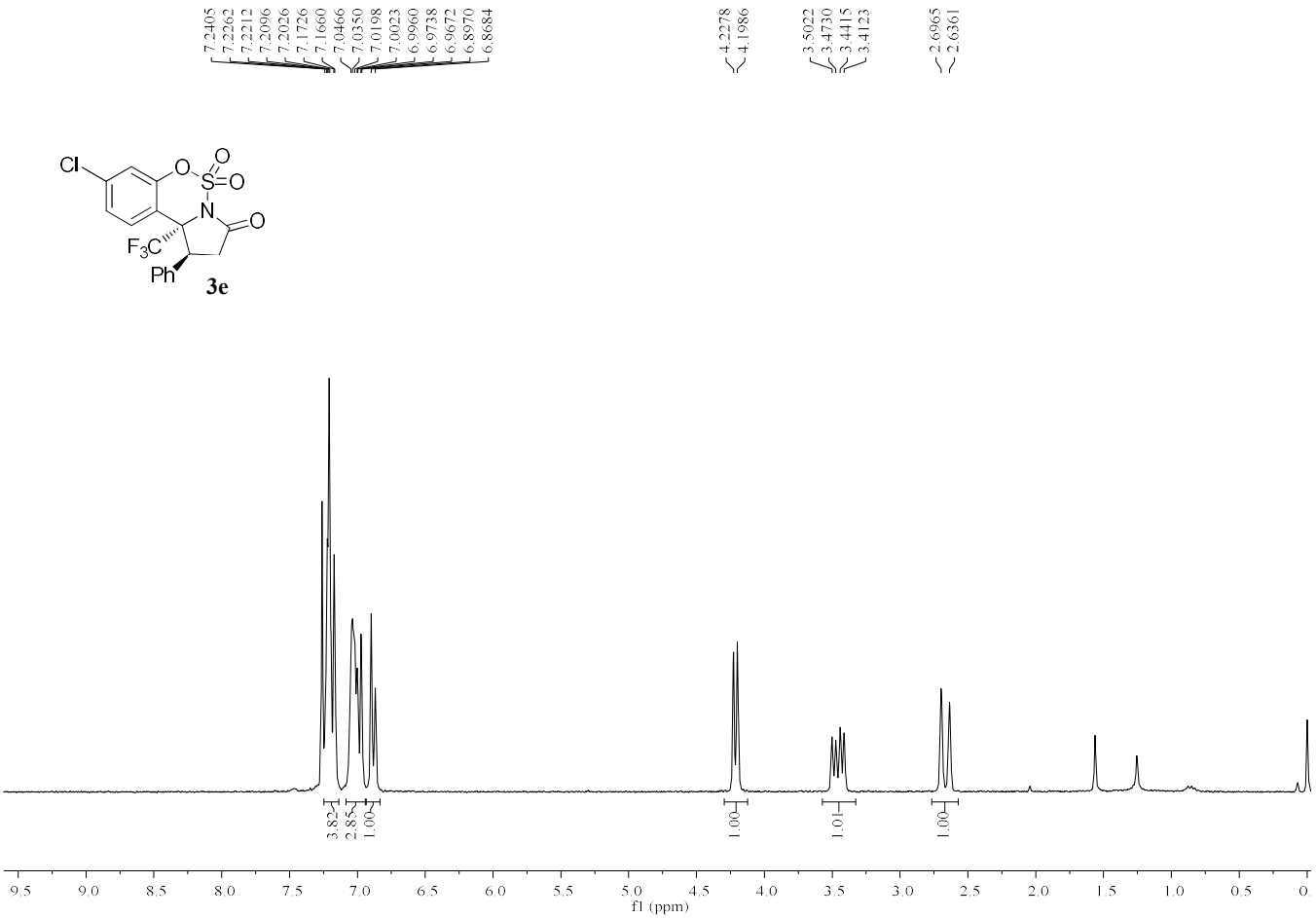


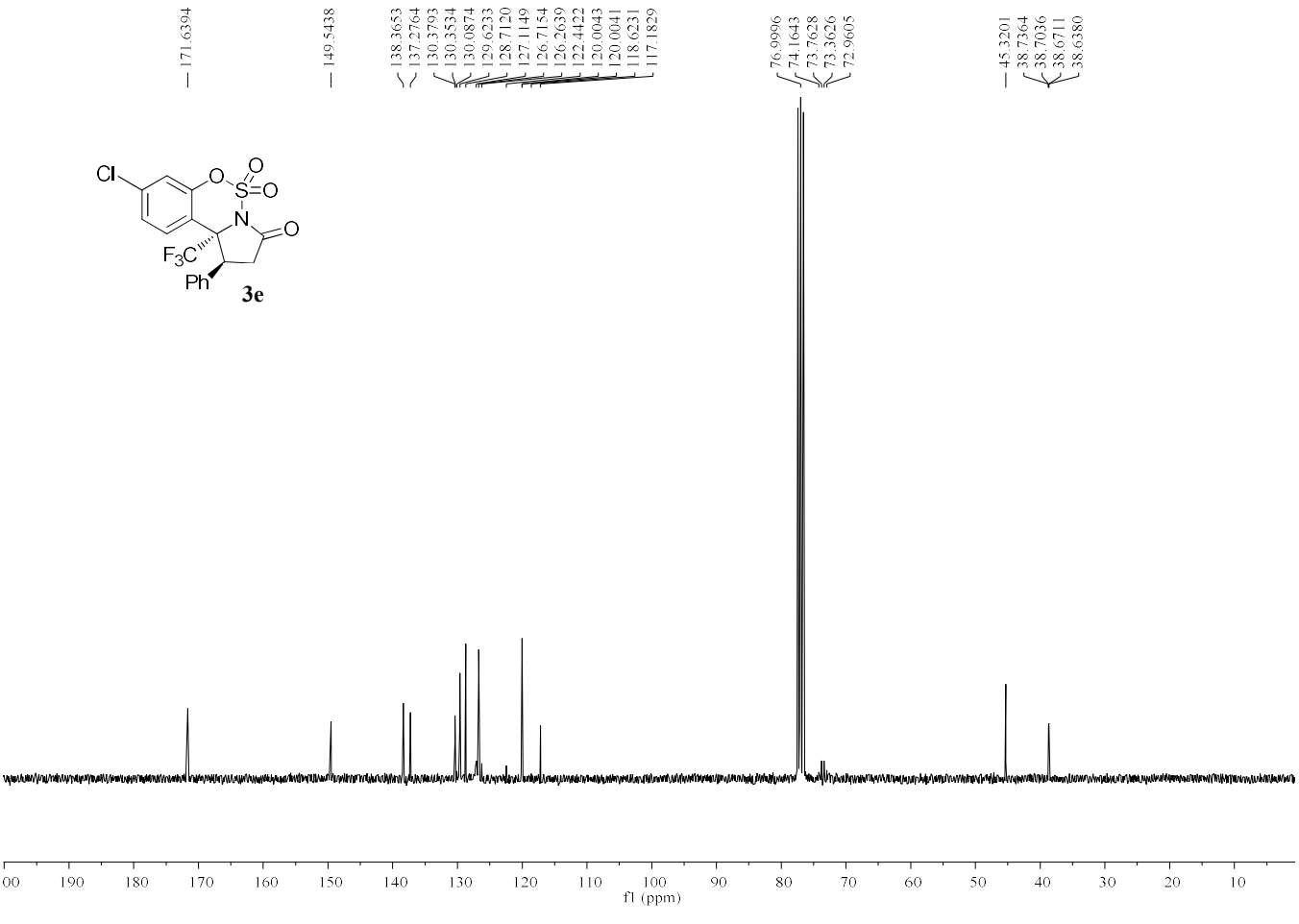
**3d**

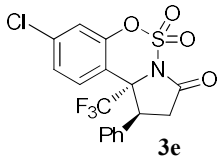

7.2735
7.2604
7.2445
7.2378
7.2189
7.0825
7.0525
7.0281
6.9426
4.2074
4.1782
3.5017
3.4724
3.4410
3.4119
2.7384
2.7304
2.6769
5.88
2.71
0.97
1.00
1.04
1.00
f1 (ppm)


171.5270
147.8091
138.1356
132.0157
131.6021
130.0338
129.6405
129.6143
129.5698
128.8130
127.1023
126.2140
122.3904
120.7315
120.0787
118.6384
76.9997
74.2117
73.8142
73.4126
73.0119
45.3698
38.5860
38.5503
38.5198
38.4875
O
S
O
N
O
Cl
F3C
Ph
3d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


-76.0797
O
S
O
N
O
Cl
F3C
Ph
3d
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

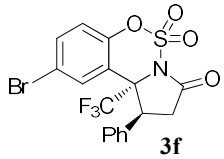

7.2405
7.2262
7.2212
7.2096
7.2026
7.1726
7.1660
7.0466
7.0350
7.0198
7.0023
6.9960
6.9738
6.9672
6.8970
6.8684
4.2278
4.1986
3.5022
3.4730
3.4415
3.4123
2.6965
2.6361
Cl
O
S
O
O
N
O
F3C
Ph
3e
3.82
2.85
1.00
1.00
1.01
1.00
f1 (ppm)


171.6394
149.5438
138.3653
137.2764
130.3793
130.3534
130.0874
129.6233
128.7120
127.1149
126.7154
126.2639
122.4422
120.0043
120.0041
118.6231
117.1829
76.9996
74.1643
73.7628
73.3626
72.9605
45.3201
38.7364
38.7036
38.6711
38.6380
Cl
O
S
O
O
N
O
F3C
Ph
3e
f1 (ppm)

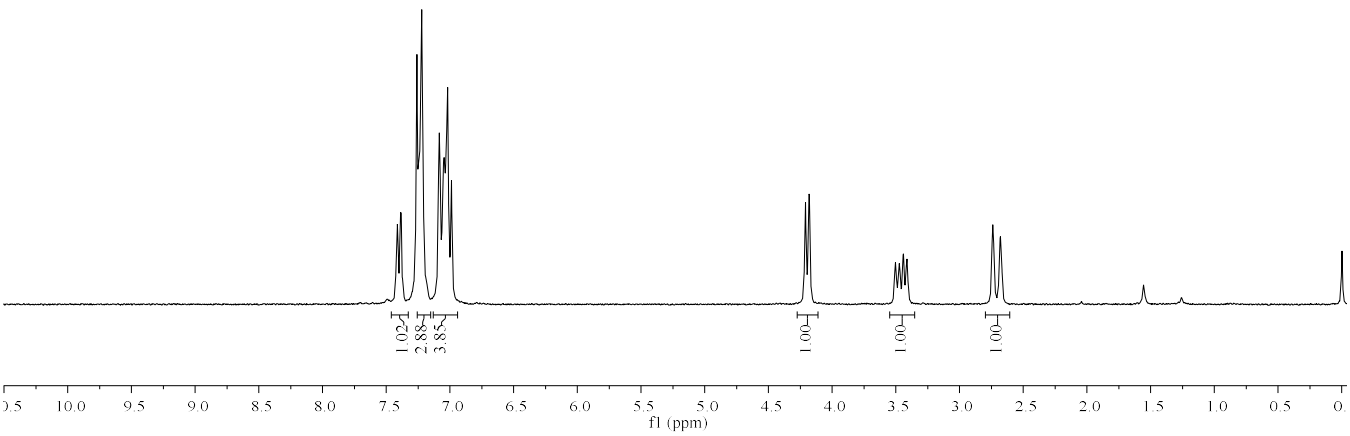


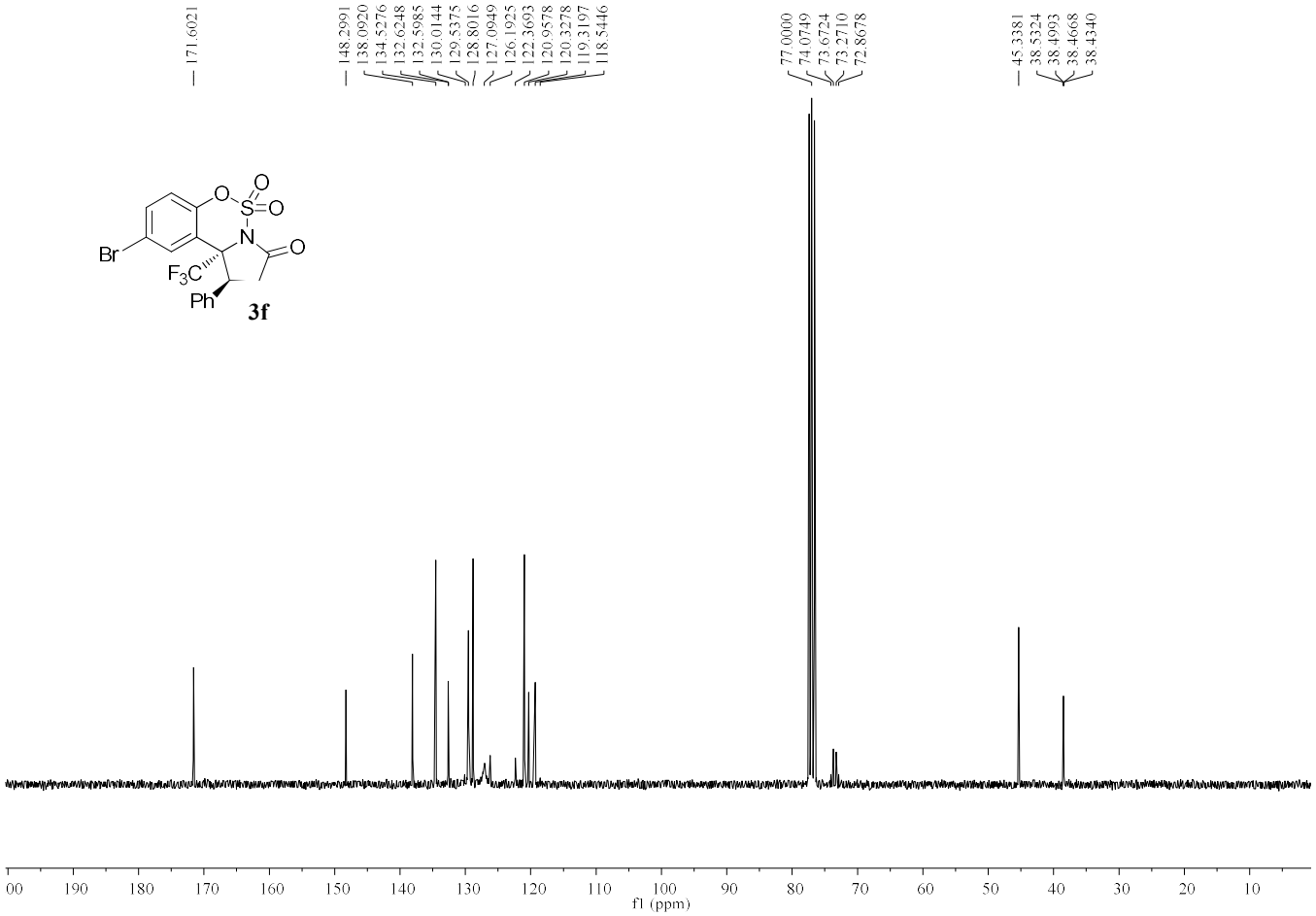
**3e**

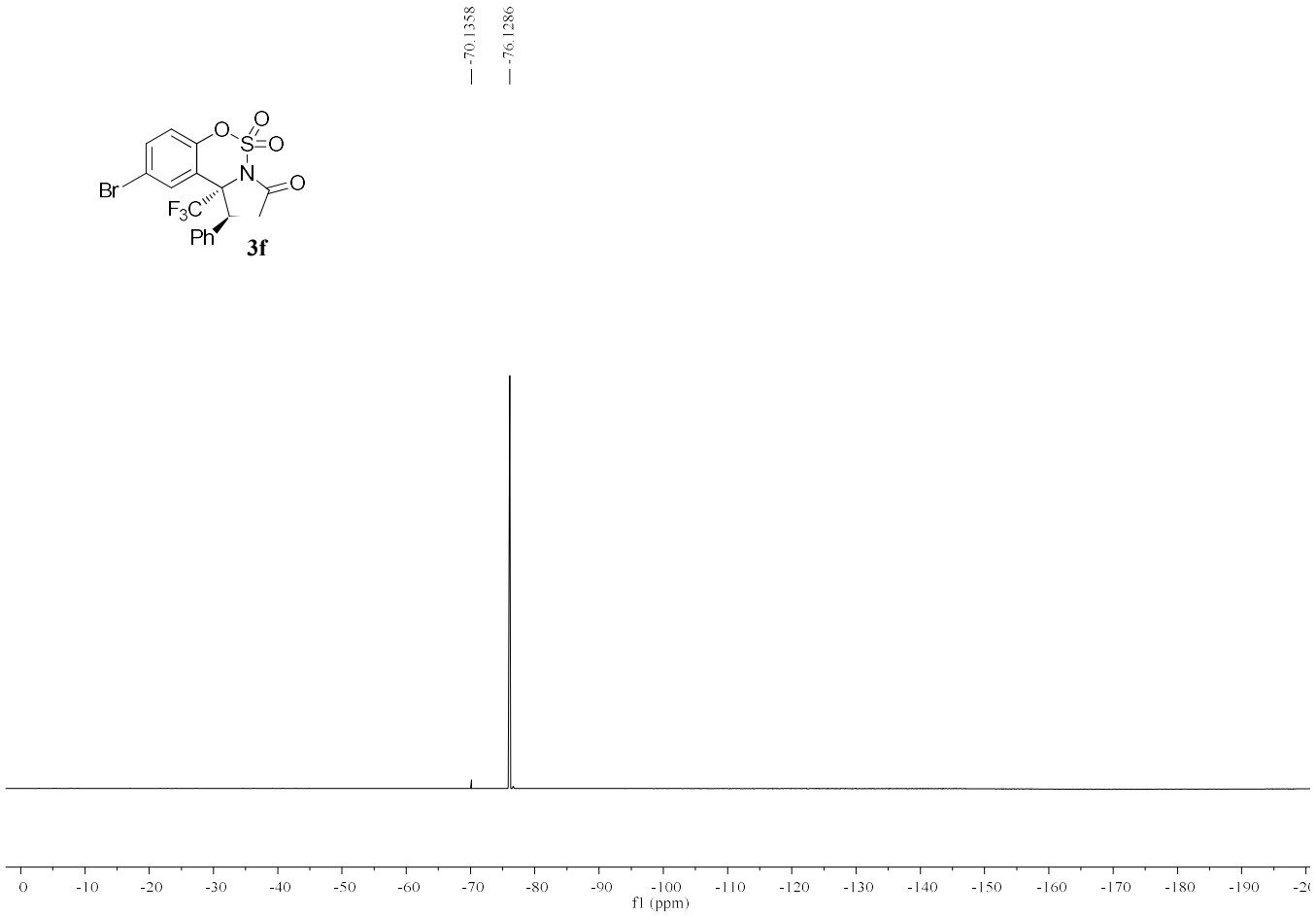

-76.5847
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

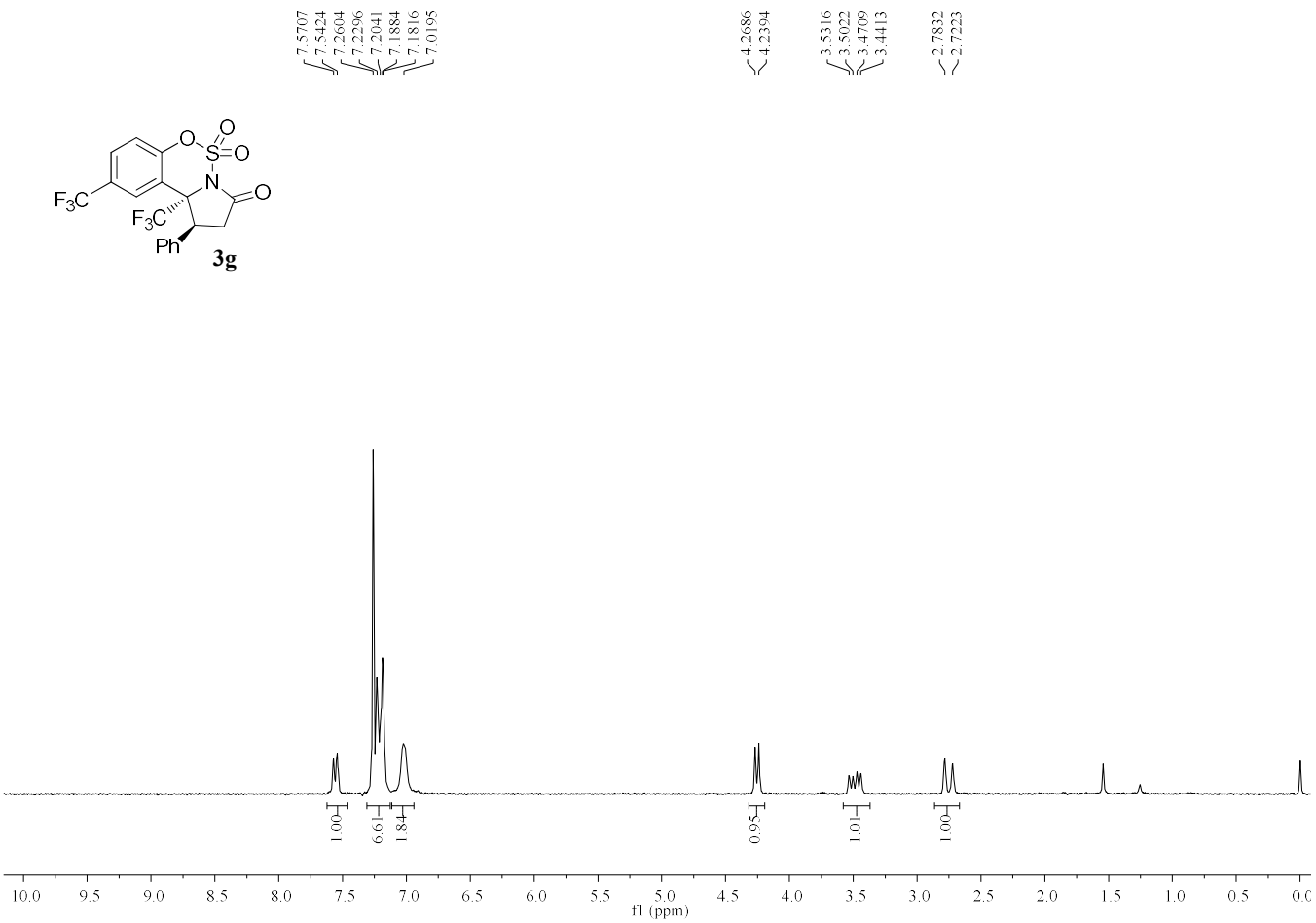


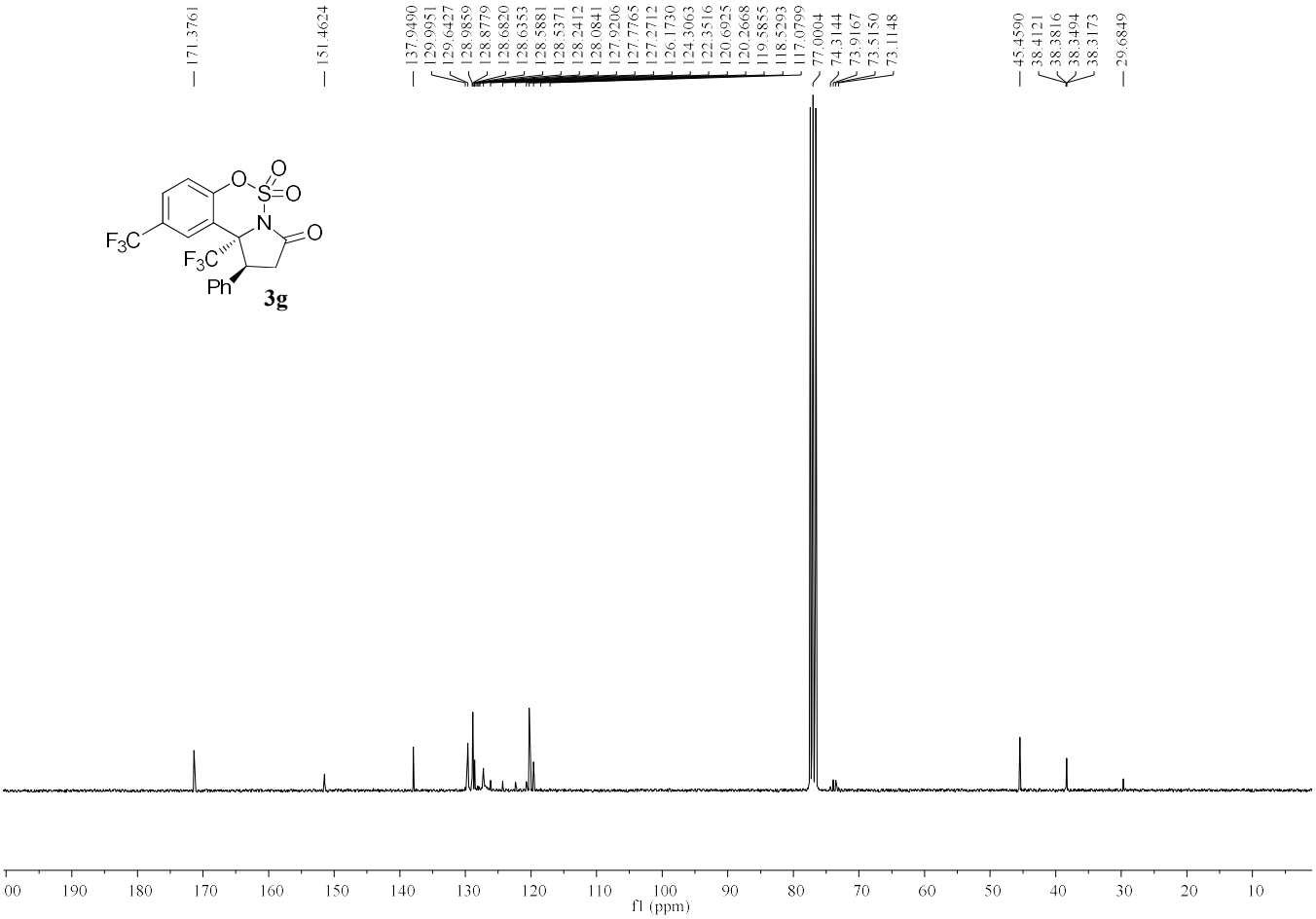
**3f**

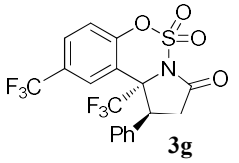

7.4183
7.4107
7.3893
7.3817
7.2459
7.2215
7.2034
7.0839
7.0454
7.0195
6.9886
4.2098
4.1806
3.5028
3.4736
3.4420
3.4127
2.7387
2.6782
1.02
2.88
3.85
1.00
1.00
1.00
f1 (ppm)


171.6021
148.2991
138.0920
134.5276
132.6248
132.5985
130.0144
129.5375
128.8016
127.0949
126.1925
122.3693
120.9578
120.3278
119.3197
118.5446
77.0000
74.0749
73.6724
73.2710
72.8678
45.3381
38.5324
38.4993
38.4668
38.4340
Br
F3C
Ph
O
S
N
3f
f1 (ppm)


-70.1358
-76.1286
Br
F3C
Ph
O
S
N
3f
f1 (ppm)


7.5707
7.5424
7.2604
7.2296
7.2041
7.1884
7.1816
7.0195
4.2686
4.2394
3.5316
3.5022
3.4709
3.4413
2.7832
2.7223
3g
1.00
6.61
1.84
0.95
1.01
1.00
f1 (ppm)

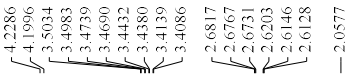

171.3761
151.4624
137.9490
129.9951
129.6427
128.9859
128.8779
128.6820
128.6353
128.5881
128.5371
128.2412
128.0841
127.9206
127.7765
127.2712
126.1730
124.3063
122.3516
120.6925
120.2668
119.5855
118.5293
117.0799
77.0004
74.3144
73.9167
73.5150
73.1148
45.4590
38.4121
38.3816
38.3494
38.3173
29.6849
3g
f1 (ppm)

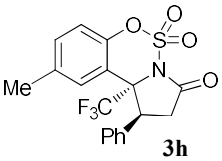


**3g**


-63.0096
-76.1048
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

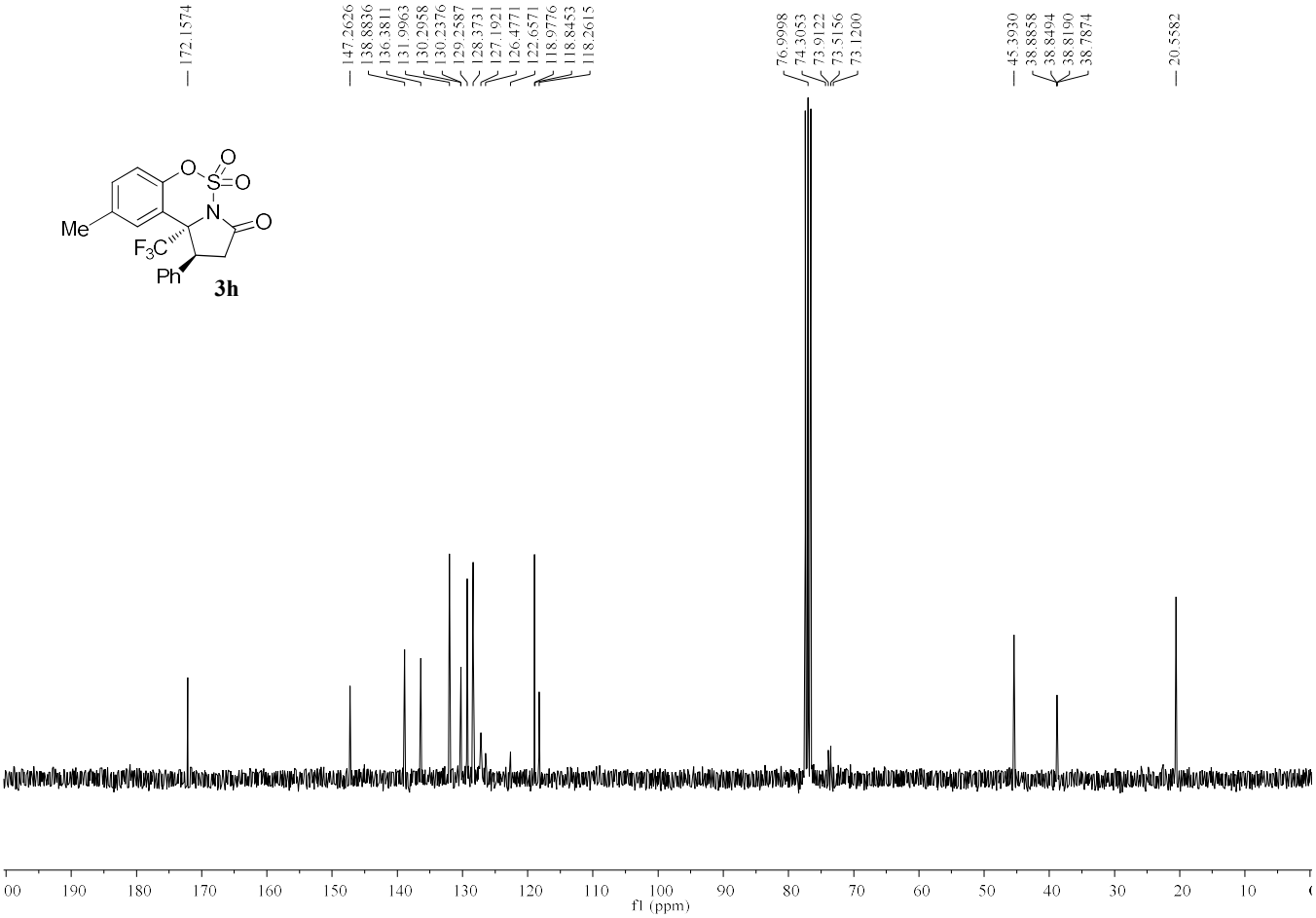

7.2254
7.2141
7.1971
7.1928
7.1740
7.1549
7.1358
7.1307
7.0939
7.0875
7.0656
7.0586
7.0466
7.0258
6.9973
6.6933


4.2286
4.1996
3.5034
3.4983
3.4739
3.4690
3.4432
3.4380
3.4139
3.4086
2.6817
2.6767
2.6731
2.6203
2.6146
2.6128
2.0577

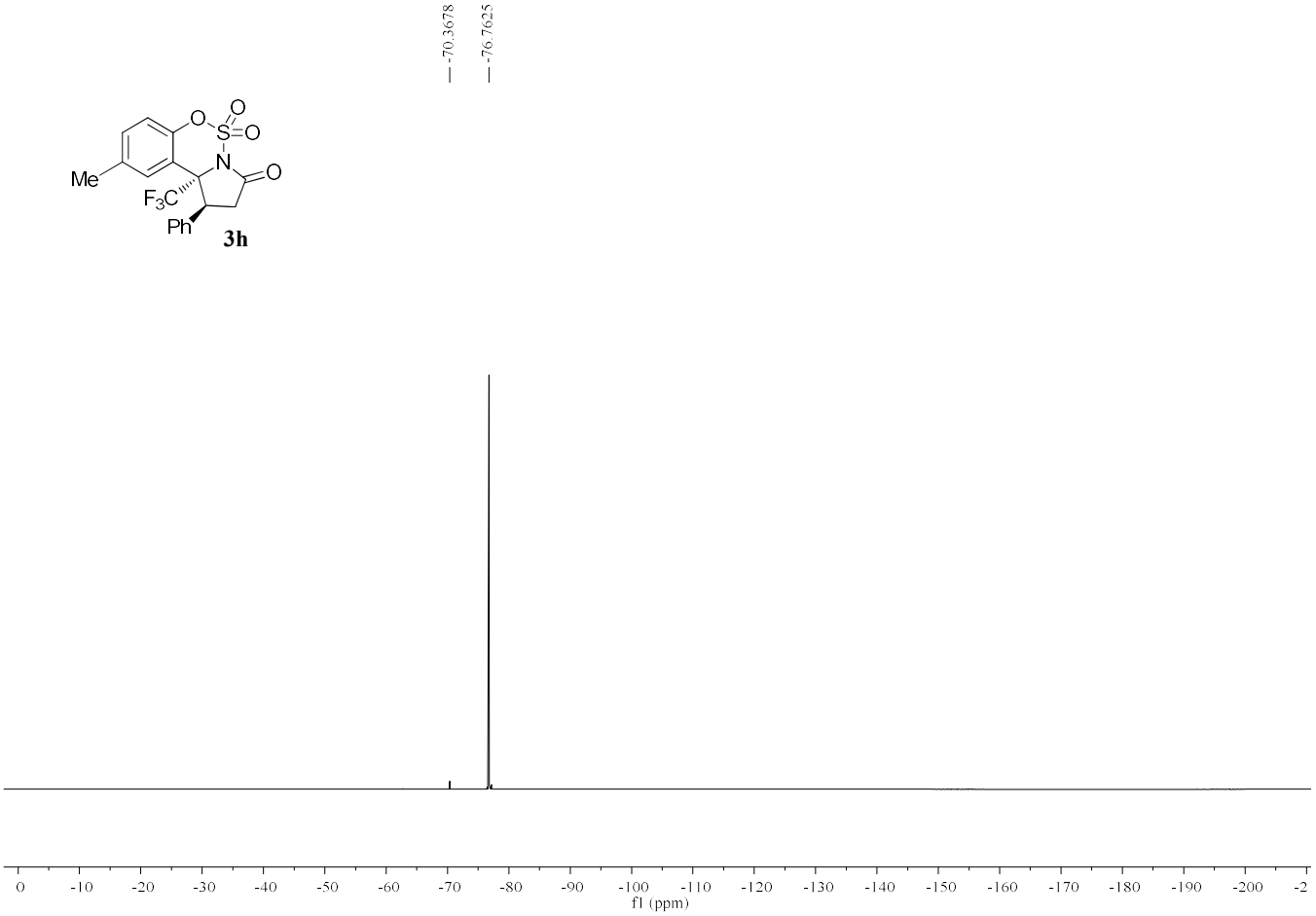


**3h**

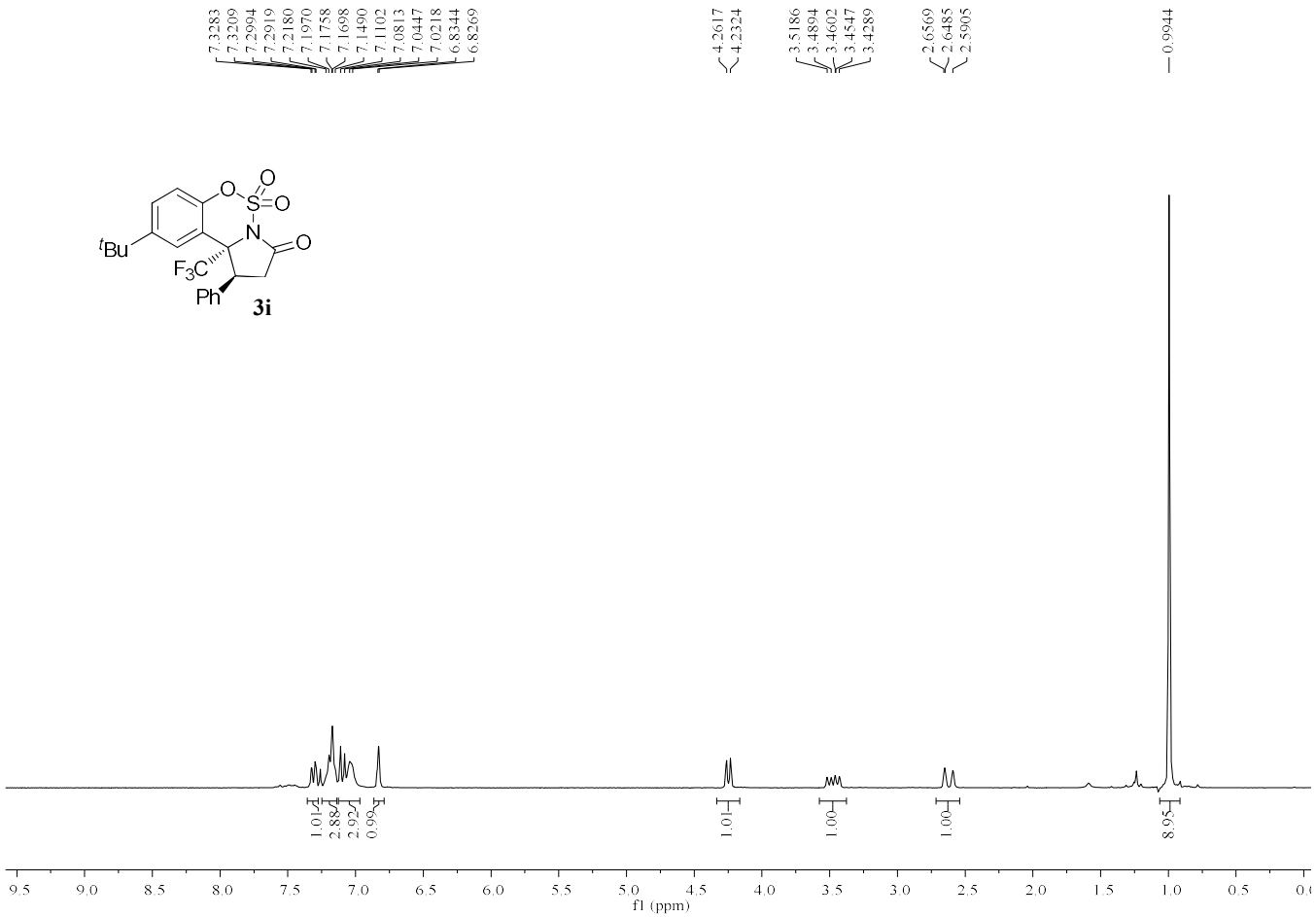

3.03
3.88
0.96
1.00
1.00
0.99
2.91
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


172.1574
147.2626
138.8836
136.3811
131.9963
130.2958
130.2376
129.2587
128.3731
127.1921
126.4771
122.6571
118.9776
118.8453
118.2615
76.9998
74.3053
73.9122
73.5156
73.1200
45.3930
38.8858
38.8494
38.8190
38.7874
20.5582
Me
F3C
Ph
O
S
N
f1 (ppm)

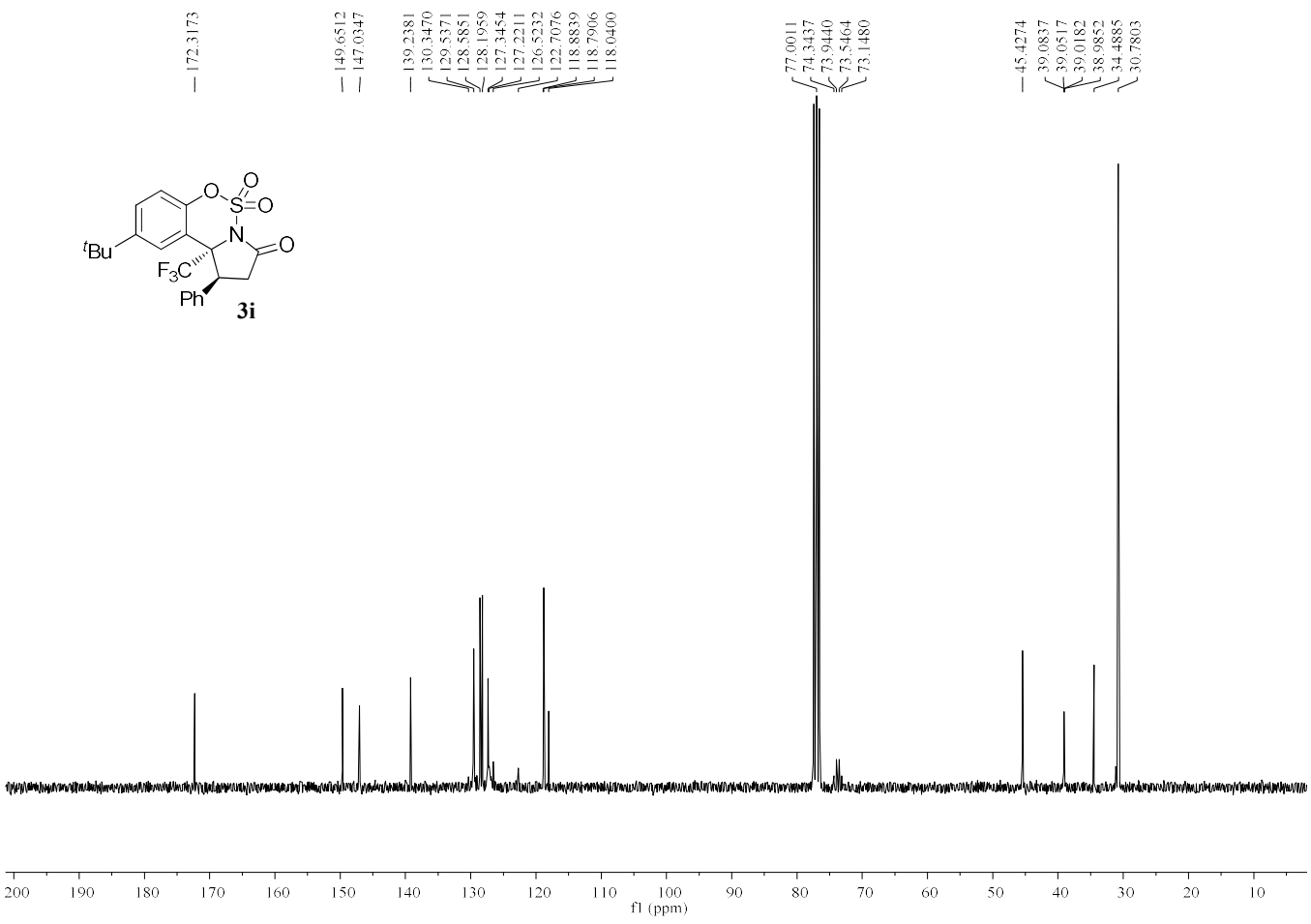
**3h**


-70.3678
-76.7625
Me
F3C
Ph
O
S
N
f1 (ppm)

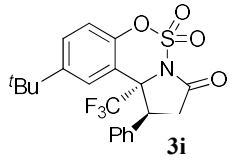
**3h**


7.3283
7.3209
7.2994
7.2919
7.2180
7.1970
7.1758
7.1698
7.1490
7.1102
7.0813
7.0447
7.0218
6.8344
6.8269
4.2617
4.2324
3.5186
3.4894
3.4602
3.4547
3.4289
2.6569
2.6485
2.5905
0.9944
1.01
2.88
2.92
0.99
1.01
1.00
1.00
8.95
f1 (ppm)

**3i**

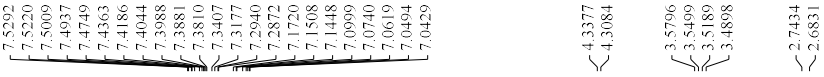

172.3173
149.6512
147.0347
139.2381
130.3470
129.5371
128.5851
128.1959
127.3454
127.2211
126.5232
122.7076
118.8839
118.7906
118.0400
77.0011
74.3437
73.9440
73.5464
73.1480
45.4274
39.0837
39.0517
39.0182
38.9852
34.4885
30.7803
f1 (ppm)

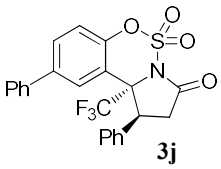
**3i**


tBu
F3C
Ph
O
S
N

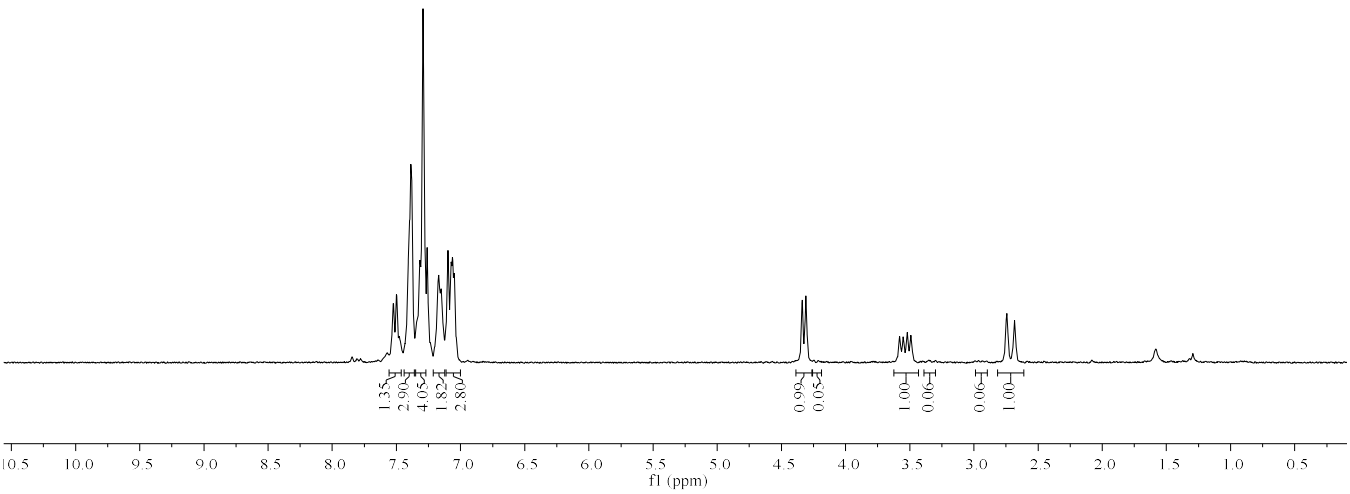
**3i**

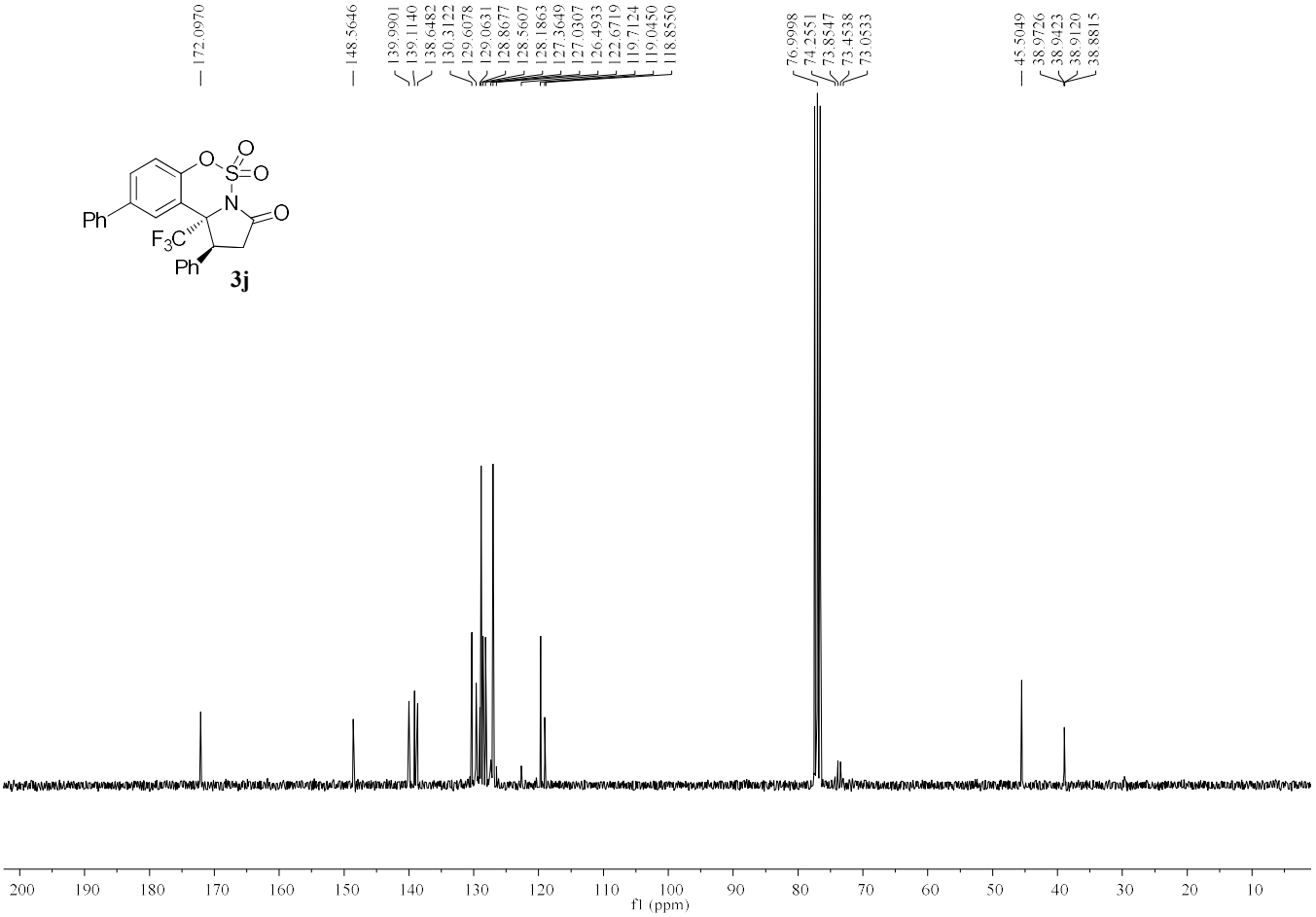

-70.5371
-77.3433
f1 (ppm)

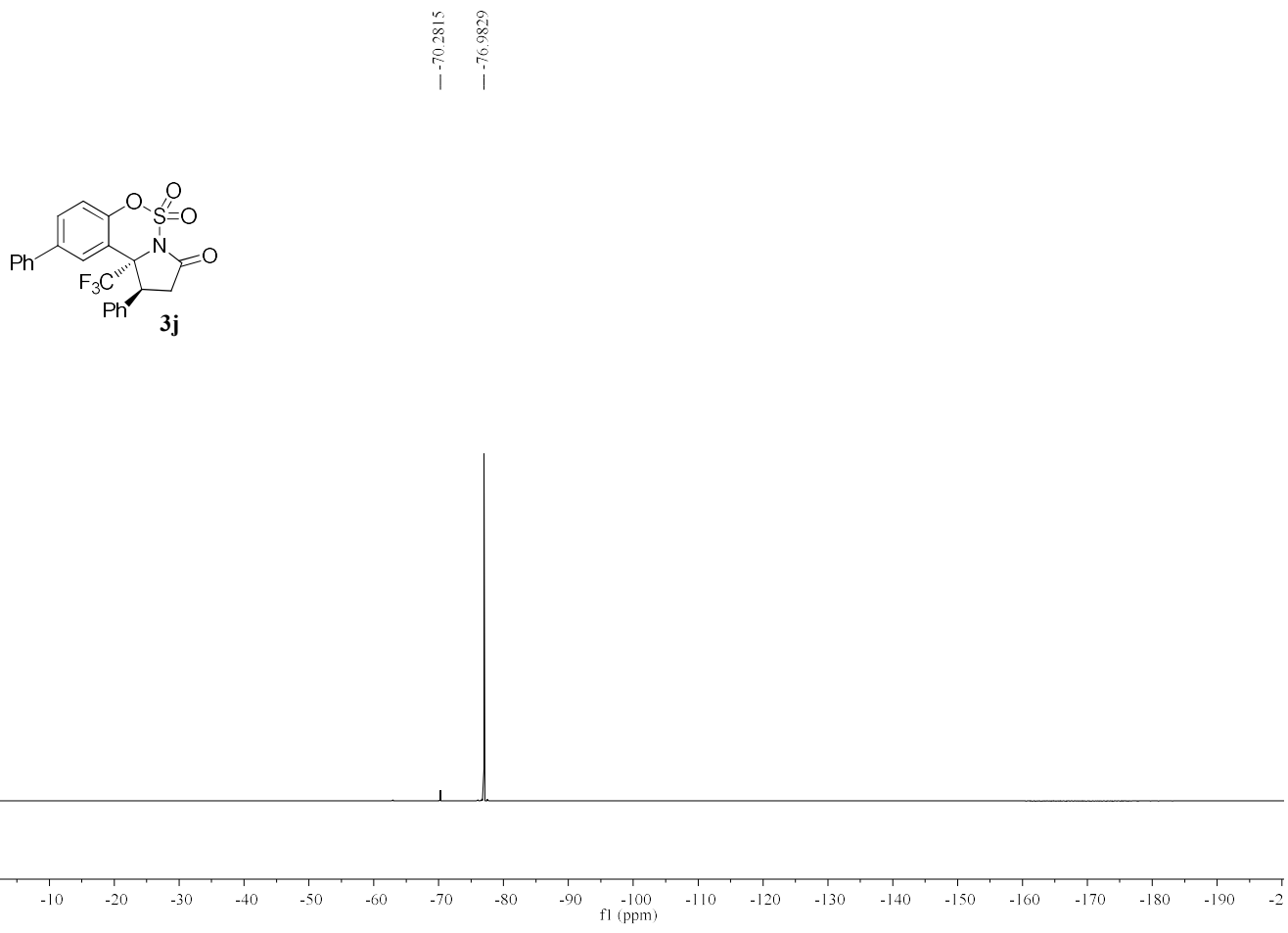

7.5292
7.5220
7.5009
7.4937
7.4749
7.4363
7.4186
7.4044
7.3988
7.3881
7.3810
7.3407
7.3177
7.2940
7.2872
7.1720
7.1508
7.1448
7.0999
7.0740
7.0619
7.0494
7.0429
4.3377
4.3084
3.5796
3.5499
3.5189
3.4898
2.7434
2.6831

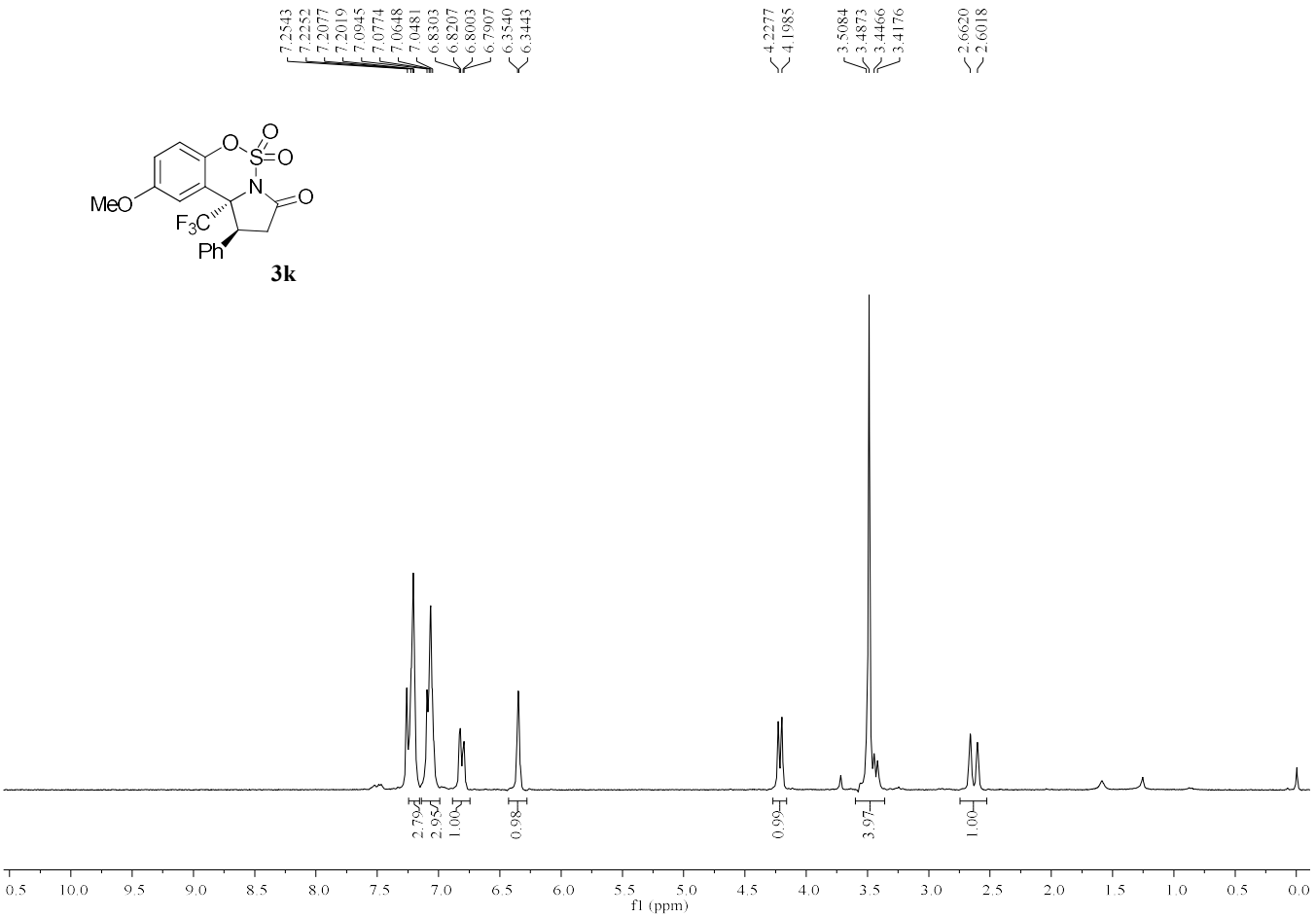

Ph
F3C
Ph
O
S
N

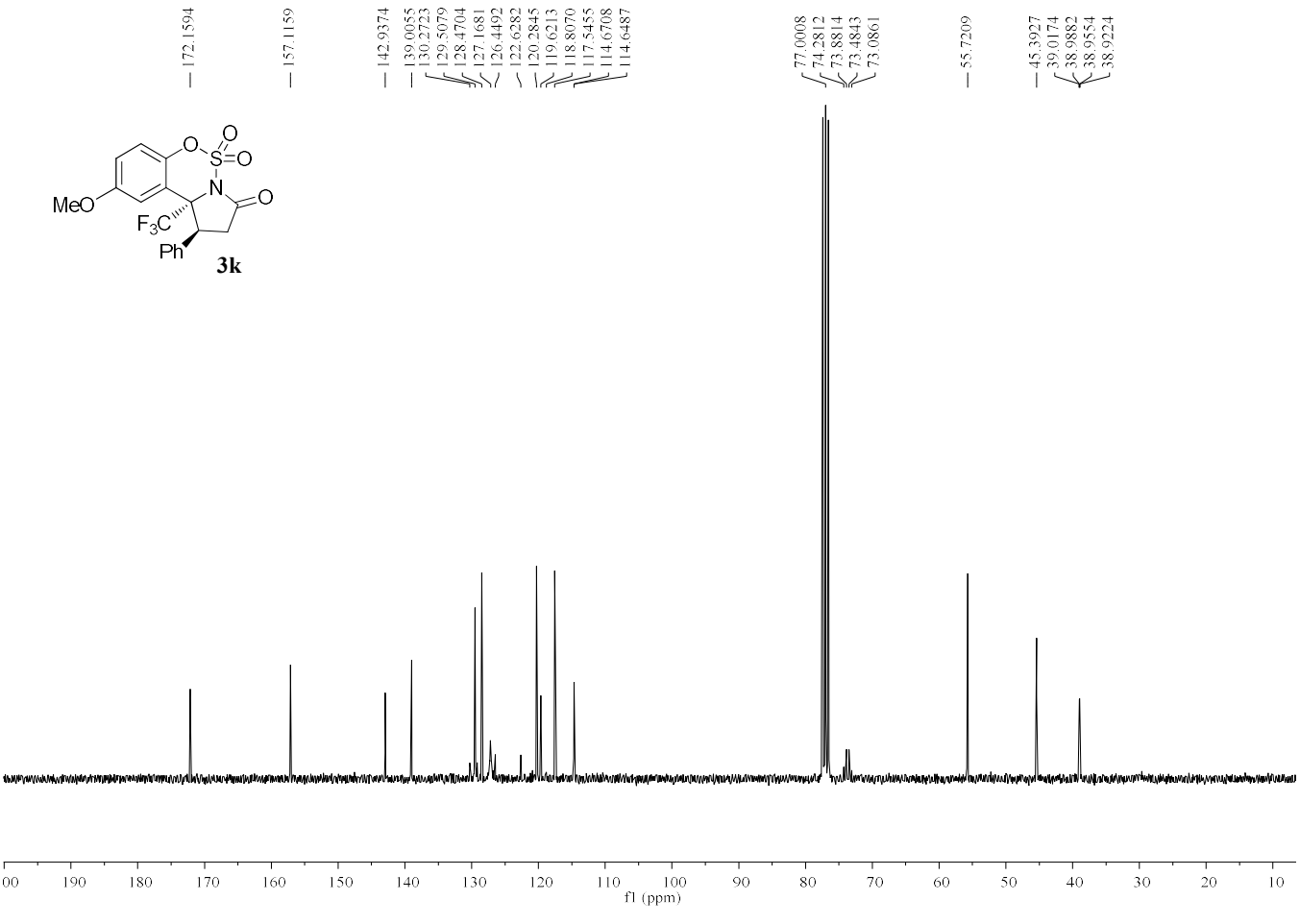
**3j**

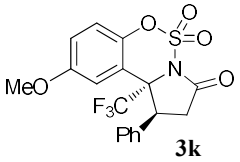

1.35
2.90
4.05
1.82
2.80
0.99
0.05
1.00
0.06
0.06
1.00
f1 (ppm)


172.0970
148.5646
139.9901
139.1140
138.6482
130.3122
129.6078
129.0631
128.8677
128.5607
128.1863
127.3649
127.0307
126.4933
122.6719
119.7124
119.0450
118.8550
76.9998
74.2551
73.8547
73.4538
73.0533
45.5049
38.9726
38.9423
38.9120
38.8815
Ph
F3C
Ph
O
S
O
O
N
O
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


-70.2815
-76.9829
Ph
F3C
Ph
O
S
O
O
N
O
3j
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -2
f1 (ppm)


7.2543
7.2252
7.2077
7.2019
7.0945
7.0774
7.0648
7.0481
6.8303
6.8207
6.8003
6.7907
6.3540
6.3443
4.2277
4.1985
3.5084
3.4873
3.4466
3.4176
2.6620
2.6018
MeO
F3C
Ph
3k
2.794
2.954
1.00
0.98
0.99
3.97
1.00
f1 (ppm)

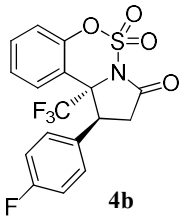

172.1594
157.1159
142.9374
139.0055
130.2723
129.5079
128.4704
127.1681
126.4492
122.6282
120.2845
119.6213
118.8070
117.5455
114.6708
114.6487
77.0008
74.2812
73.8814
73.4843
73.0861
55.7209
45.3927
39.0174
38.9882
38.9554
38.9224
MeO
F3C
Ph
3k
f1 (ppm)


MeO
F3C
Ph
O
S
N

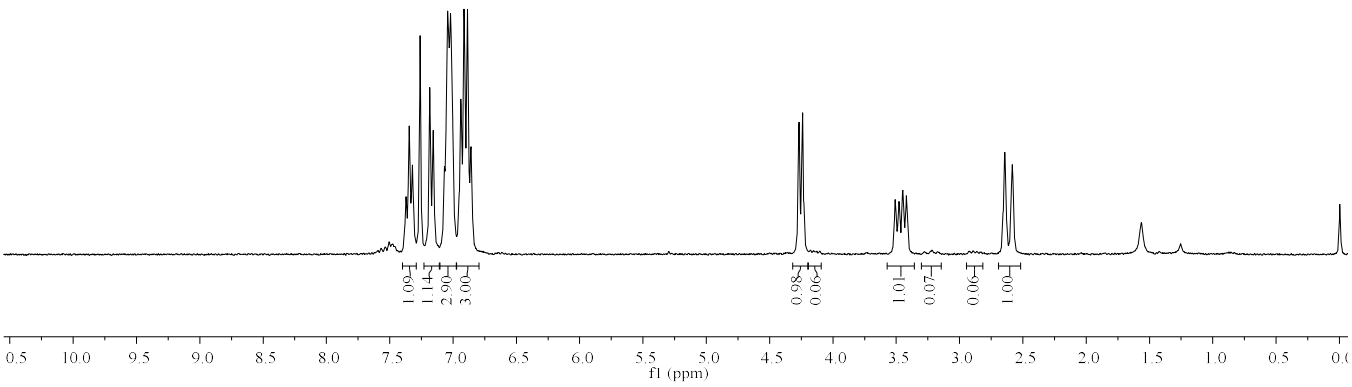
**3k**

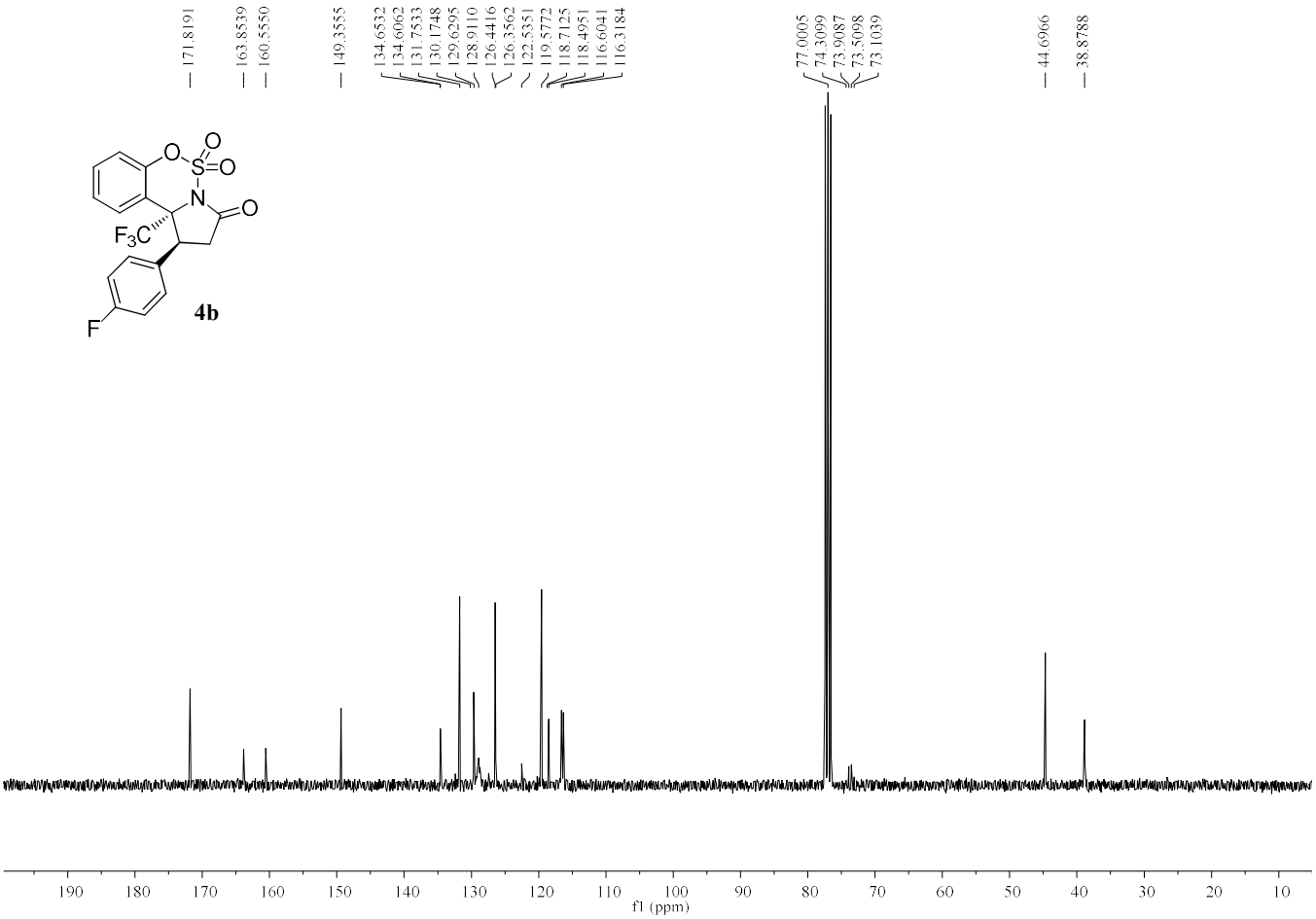

-70.4288
-76.9505
f1 (ppm)

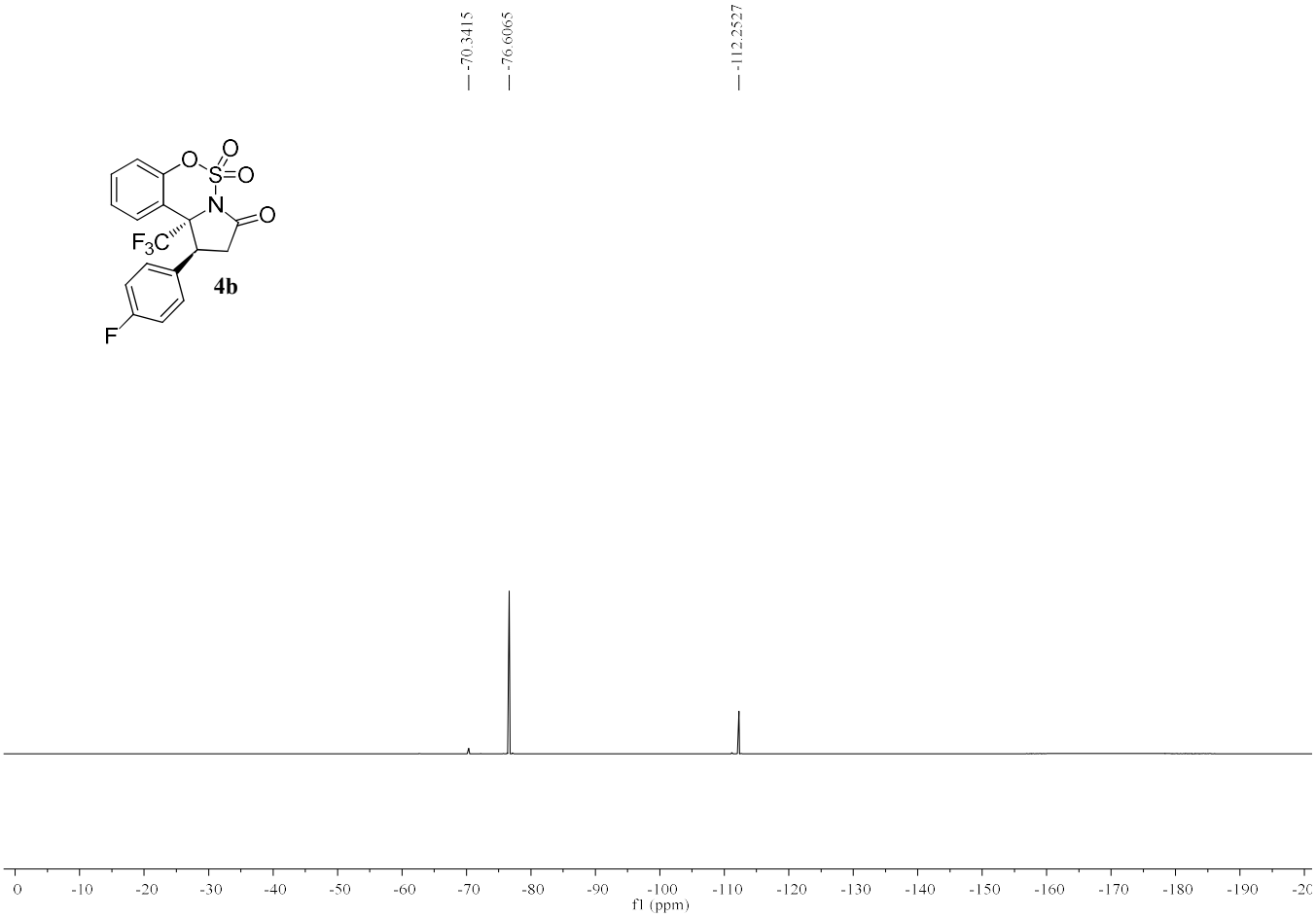

7.3749
7.3694
7.3464
7.3225
7.3170
7.2599
7.1831
7.1556
7.0691
7.0433
7.0203
7.0062
6.9401
6.9135
6.8855
6.8573
4.2684
4.2394
3.5080
3.4790
3.4472
3.4178
2.6433
2.5829

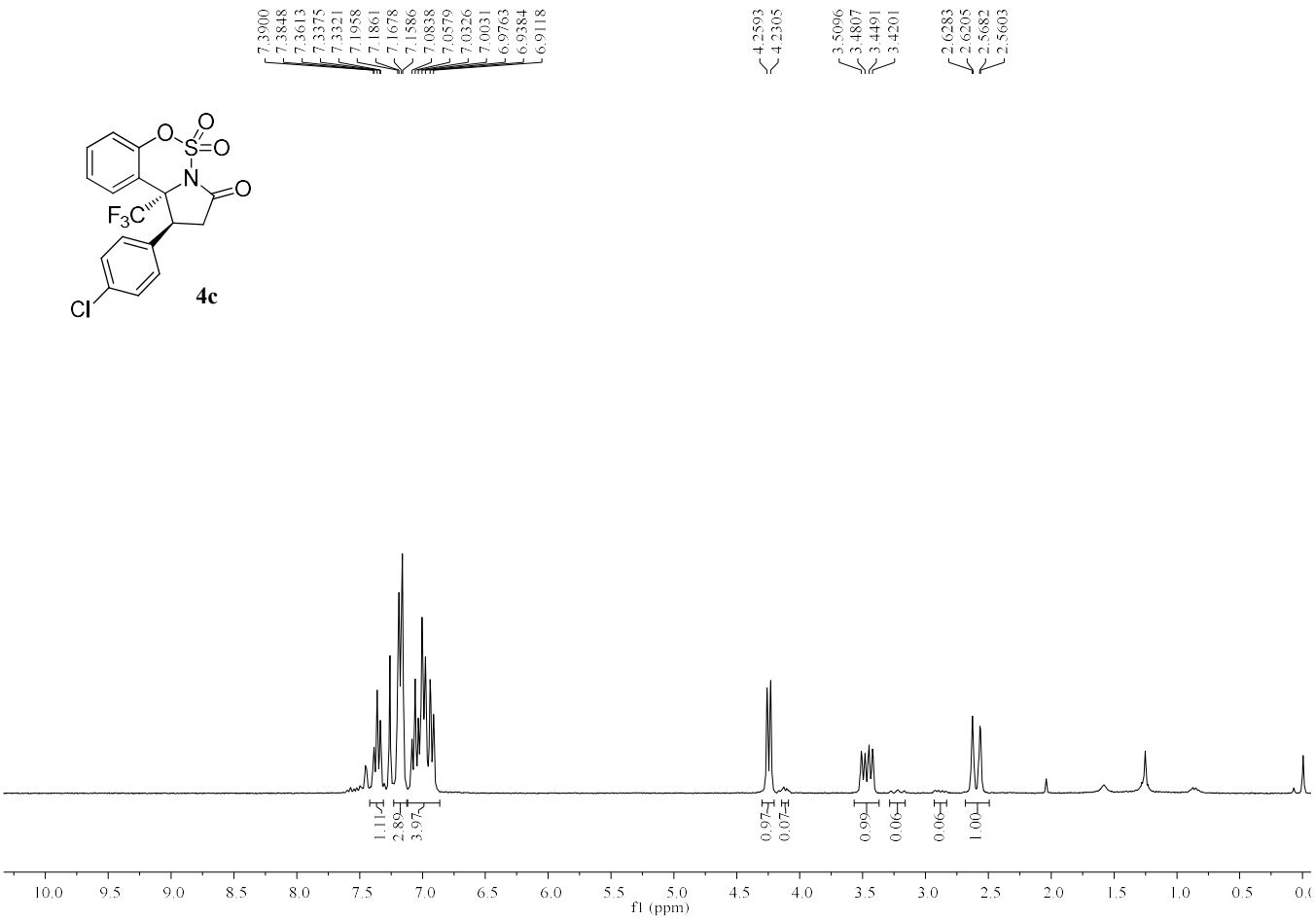

F3C
F
O
S
N

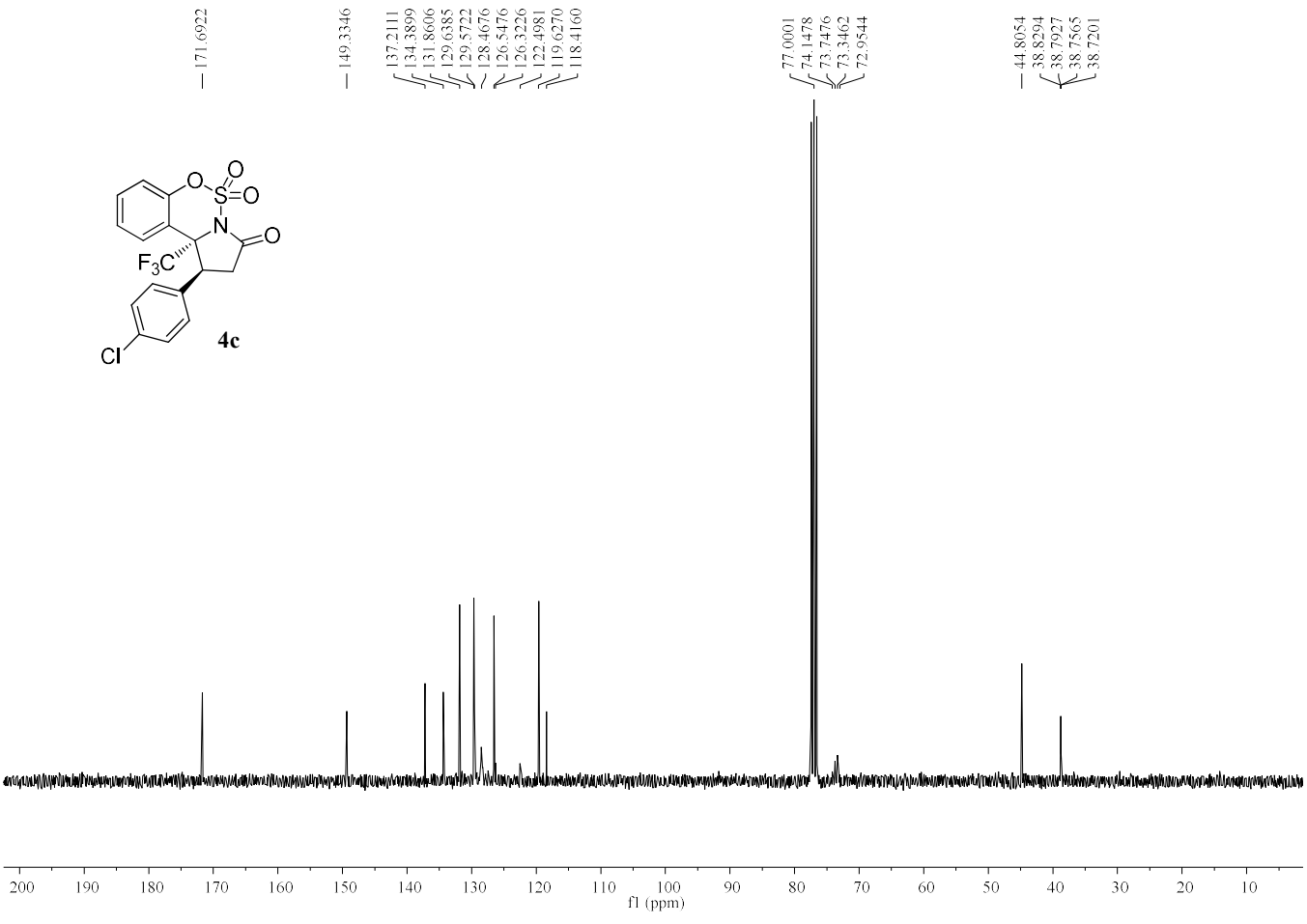
**4b**

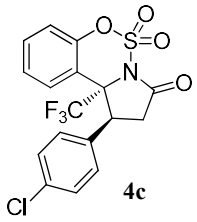

1.09
1.14
2.90
3.00
0.98
0.06
1.01
0.07
0.06
1.00
f1 (ppm)


171.8191
163.8539
160.5550
149.3555
134.6532
134.6062
131.7533
130.1748
129.6295
128.9110
126.4416
126.3562
122.5351
119.5772
118.7125
118.4951
116.6041
116.3184
77.0005
74.3099
73.9087
73.5098
73.1039
44.6966
38.8788
O
S
O
O
N
O
F3C
F
4b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


-70.3415
-76.6065
-112.2527
O
S
O
O
N
O
F3C
F
4b
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

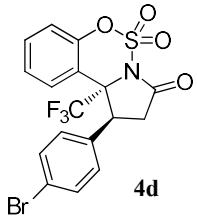

7.3900
7.3848
7.3613
7.3375
7.3321
7.1958
7.1861
7.1678
7.1586
7.0838
7.0579
7.0326
7.0031
6.9763
6.9384
6.9118
4.2593
4.2305
3.5096
3.4807
3.4491
3.4201
2.6283
2.6205
2.5682
2.5603
4c
1.11
2.89
3.97
0.97
0.07
0.99
0.06
0.06
1.00
f1 (ppm)


171.6922
149.3346
137.2111
134.3899
131.8606
129.6385
129.5722
128.4676
126.5476
126.3226
122.4981
119.6270
118.4160
77.0001
74.1478
73.7476
73.3462
72.9544
44.8054
38.8294
38.7927
38.7565
38.7201
4c
f1 (ppm)



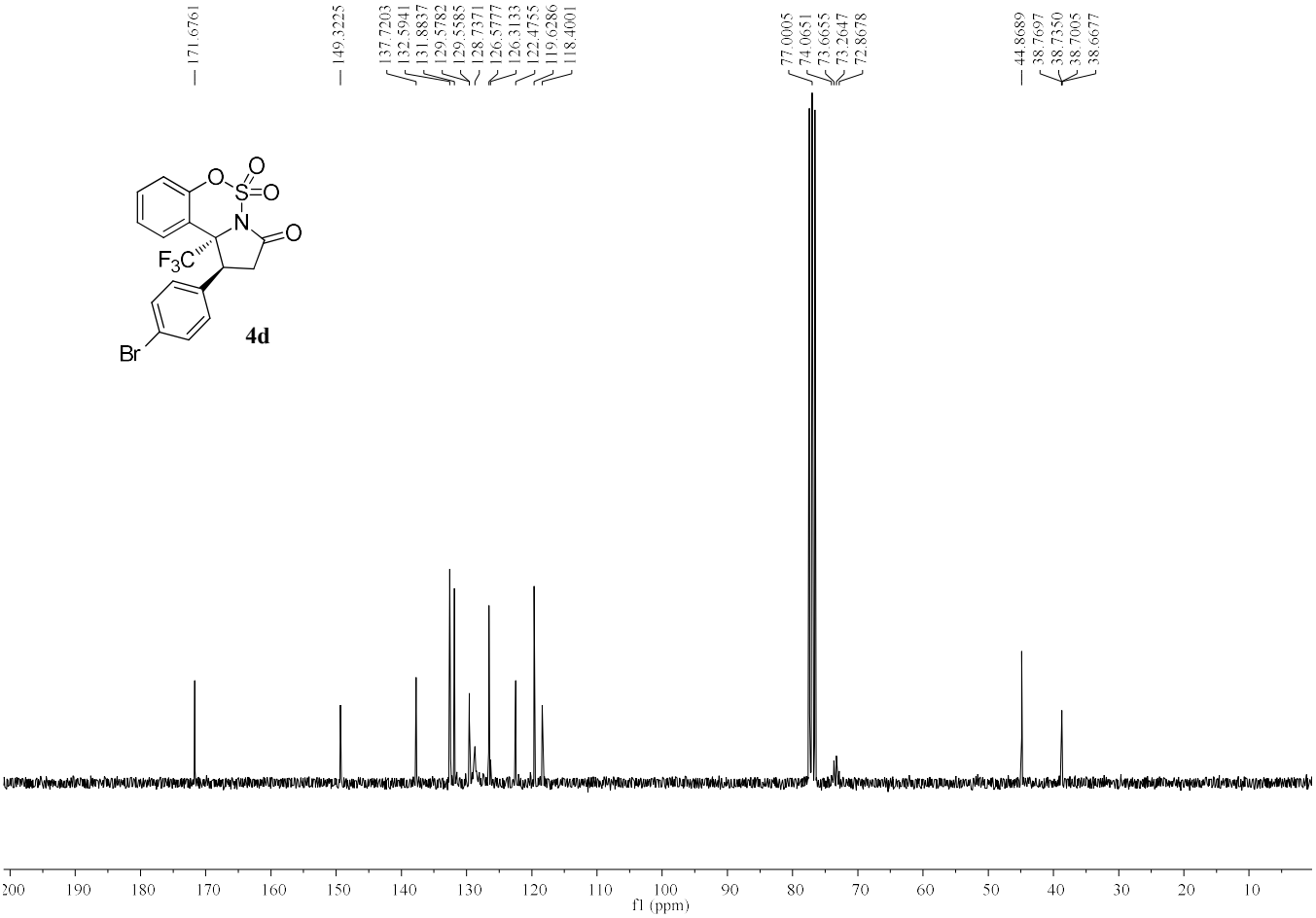
**4c**

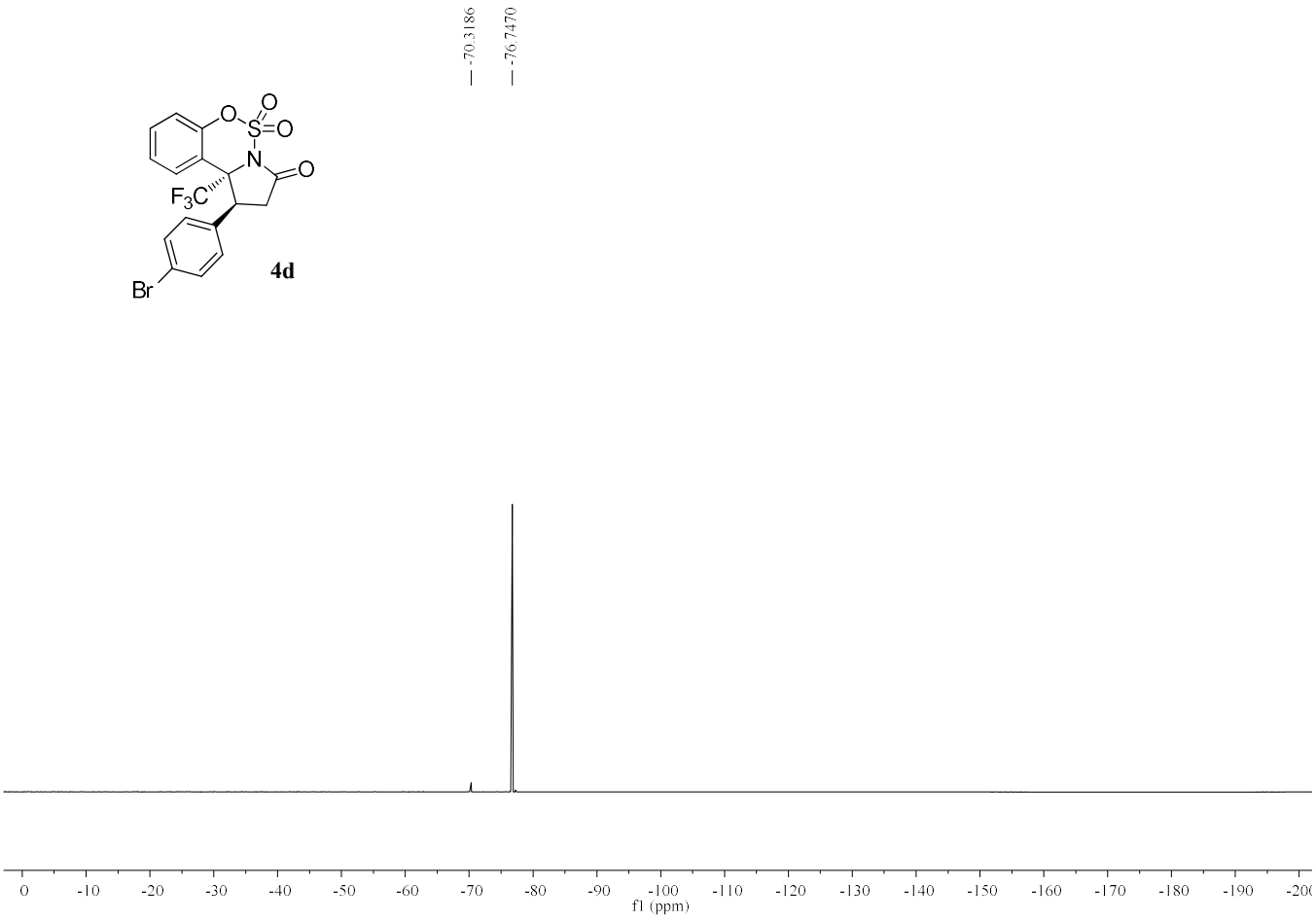

-70.3337
-76.7148
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

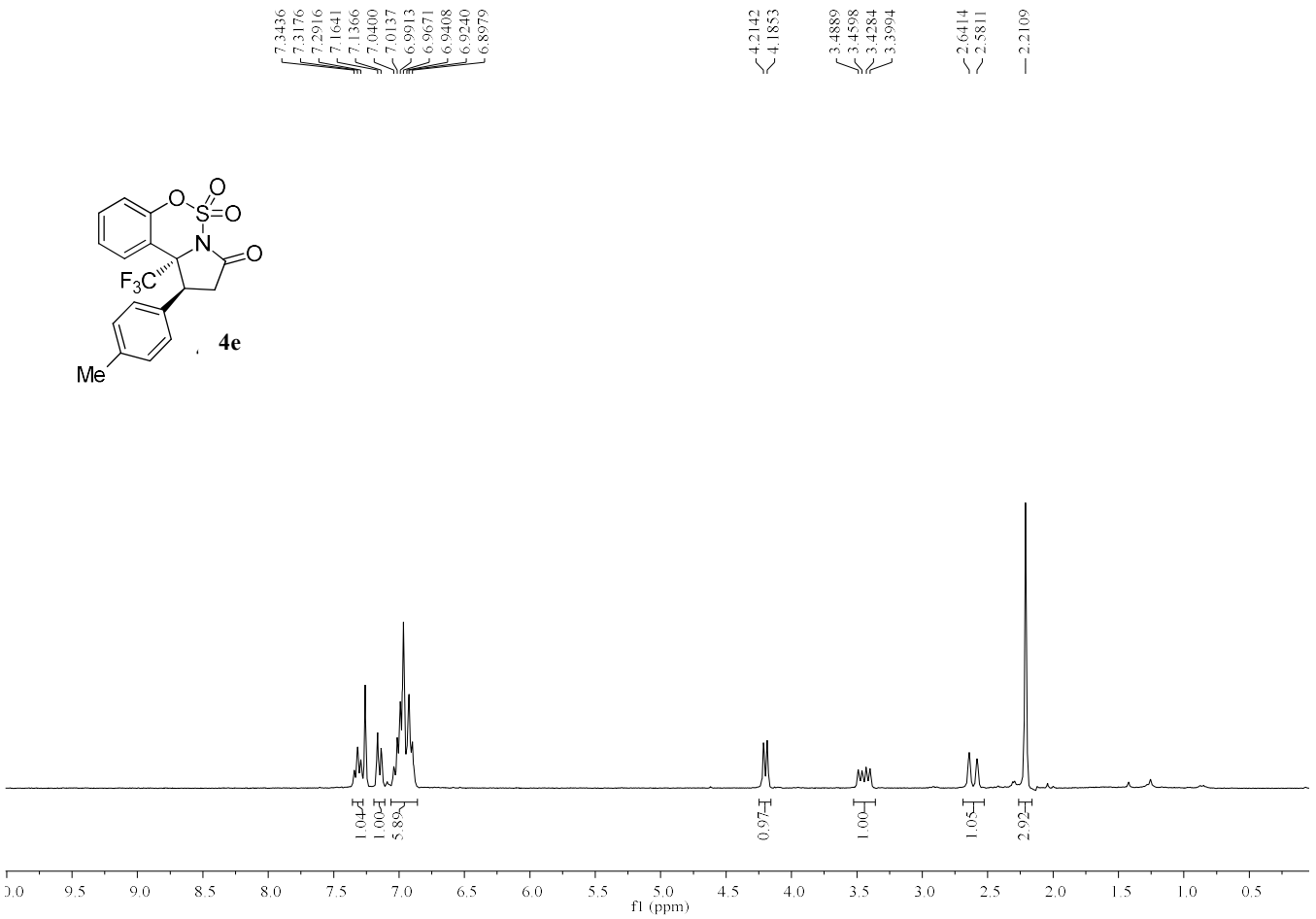


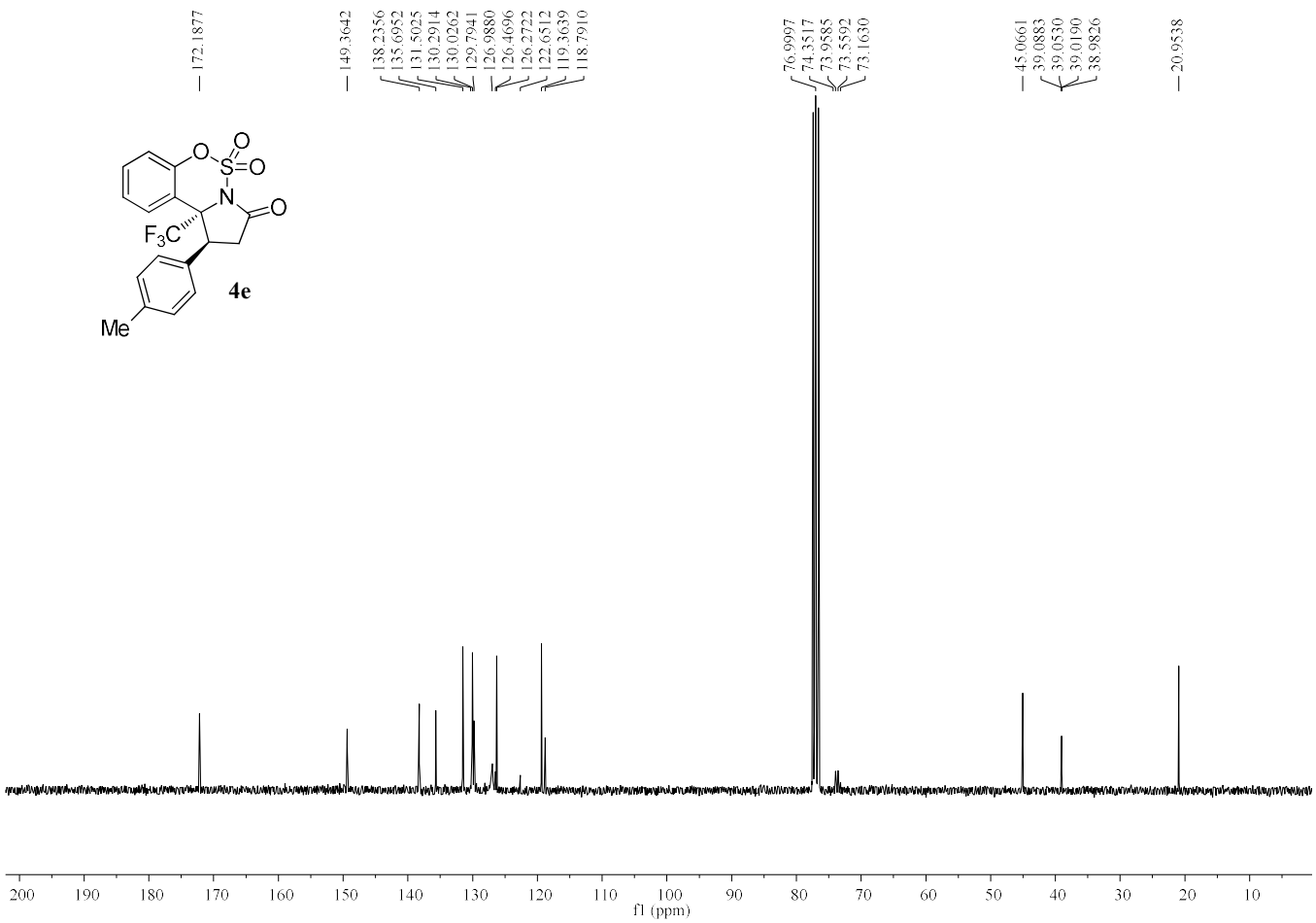
**4d**

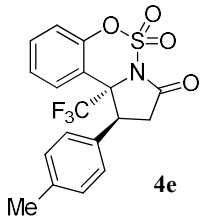

7.3942
7.3891
7.3810
7.3656
7.3432
7.3167
7.2001
7.1728
7.0866
7.0606
7.0352
6.9372
6.9123
6.9040
4.2354
4.2065
3.5037
3.4747
3.4435
3.4146
2.6246
2.6175
2.6111
2.5643
2.5563
2.99
1.00
0.99
2.85
0.99
1.00
0.06
0.06
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


171.6761
149.3225
137.7203
132.5941
131.8837
129.5782
129.5585
128.7371
126.5777
126.3133
122.4755
119.6286
118.4001
77.0005
74.0651
73.6655
73.2647
72.8678
44.8689
38.7697
38.7350
38.7005
38.6677
O
S
O
N
O
F3C
Br
4d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


-70.3186
-76.7470
O
S
O
N
O
F3C
Br
4d
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

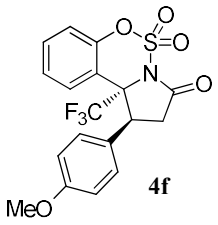

7.3436
7.3176
7.2916
7.1641
7.1366
7.0400
7.0137
6.9913
6.9671
6.9408
6.9240
6.8979
4.2142
4.1853
3.4889
3.4598
3.4284
3.3994
2.6414
2.5811
2.2109
4e
1.04
1.00
5.89
0.97
1.00
1.05
2.92
f1 (ppm)


172.1877
149.3642
138.2356
135.6952
131.5025
130.2914
130.0262
129.7941
126.9880
126.4696
126.2722
122.6512
119.3639
118.7910
76.9997
74.3517
73.9585
73.5592
73.1630
45.0661
39.0883
39.0530
39.0190
38.9826
20.9538
4e
f1 (ppm)



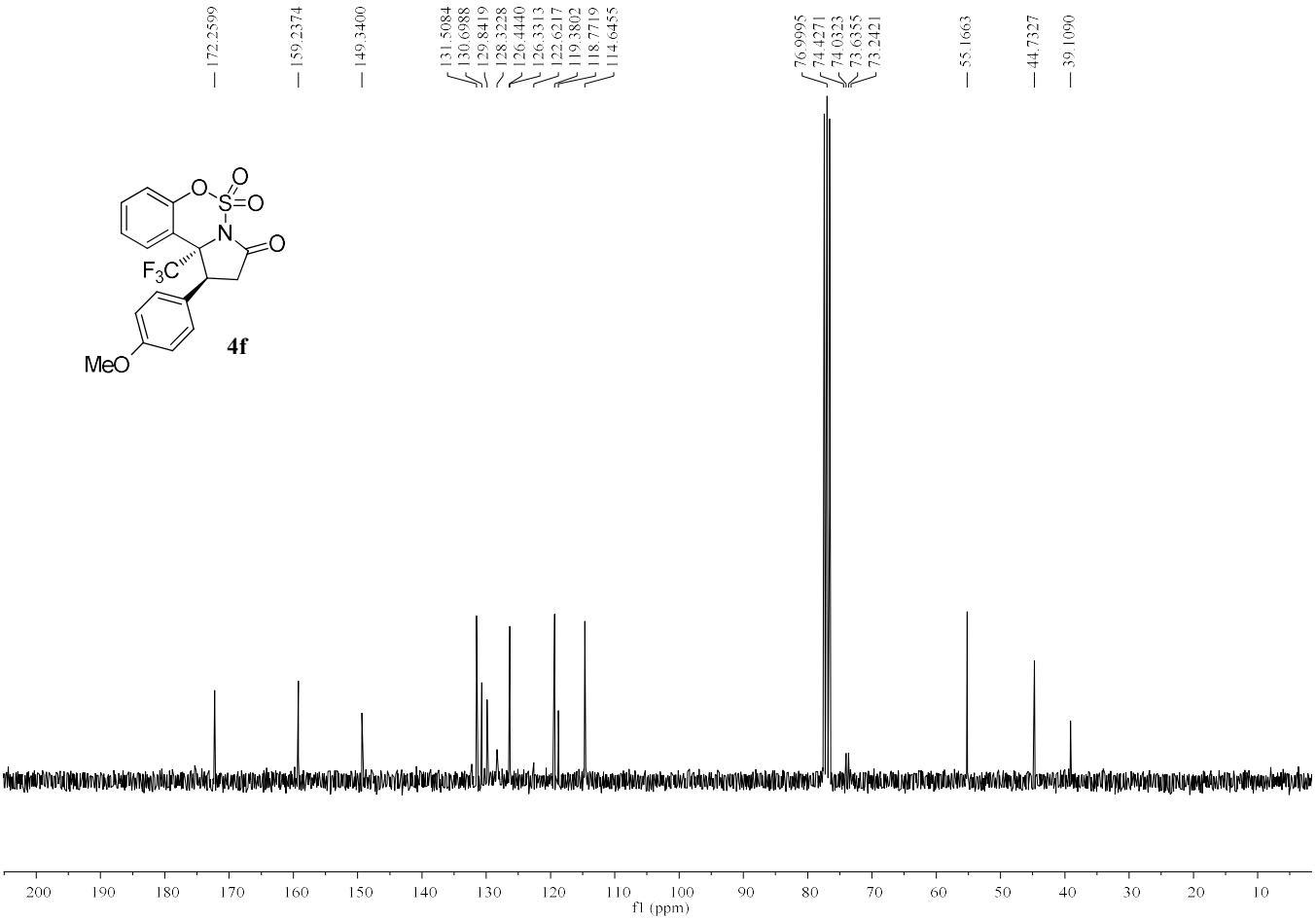
**4e**

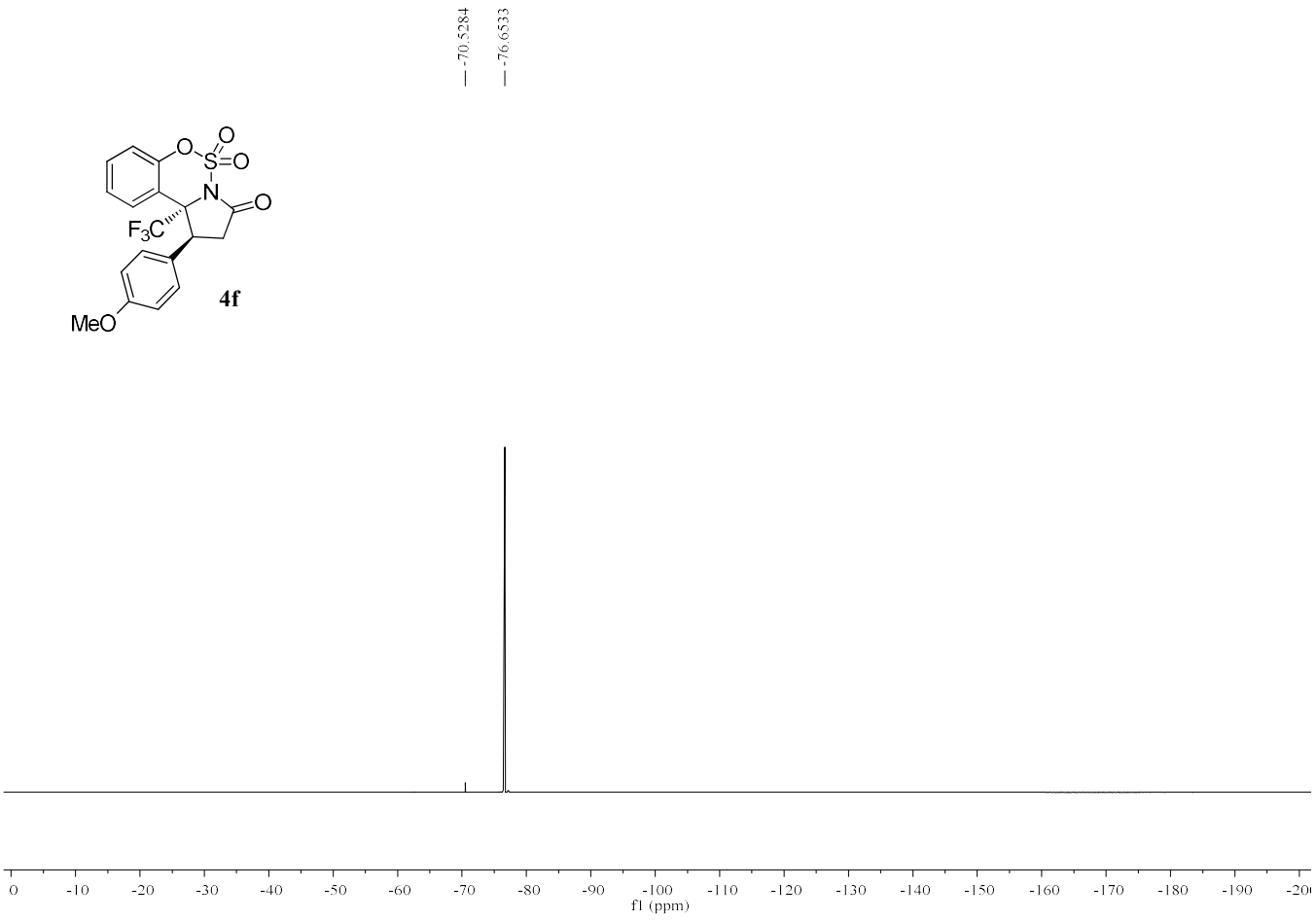

-76.7152
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)
7.3513
7.3460
7.3276
7.3230
7.3182
7.2995
7.2937
7.1682
7.1644
7.1407
7.1367
7.0583
7.0543
7.0314
7.0274
7.0072
7.0034
6.9663
6.9365
6.7110
6.6828
4.2155
4.1867
3.6952
3.4906
3.4613
3.4324
3.4272
3.4012
2.6435
2.6397
2.6346
2.5821
2.5742

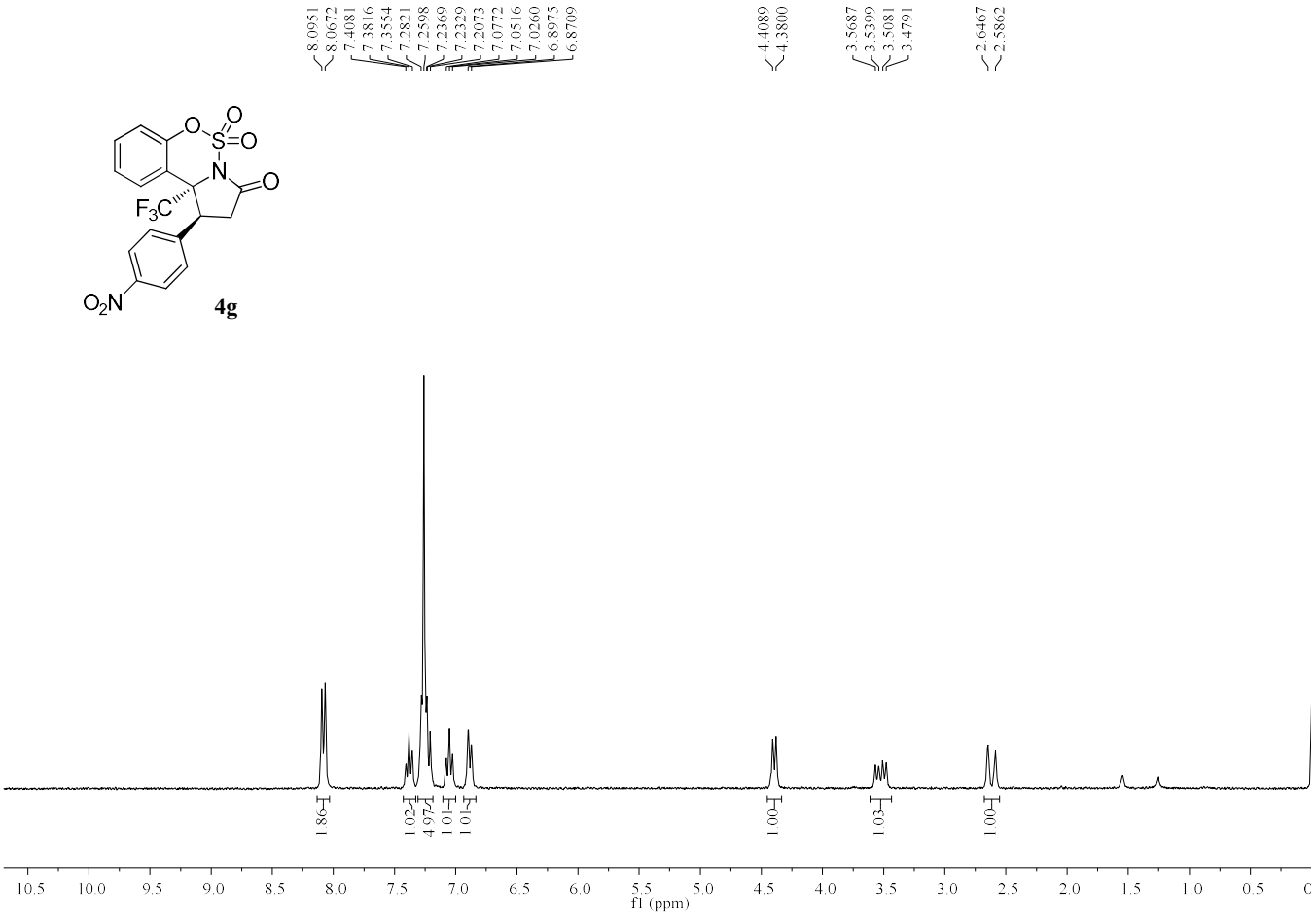


**4f**

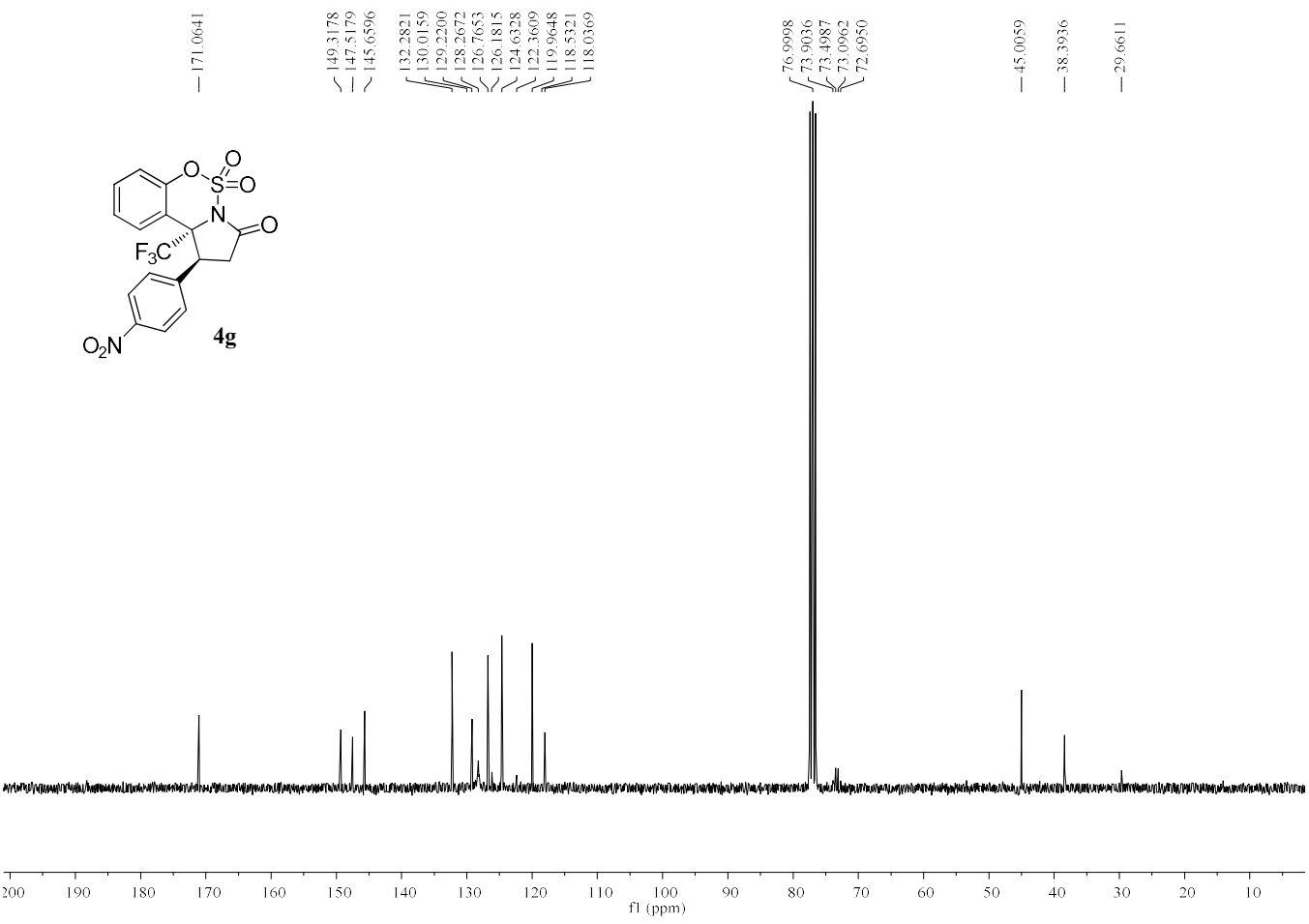

1.07
1.00
1.10
2.94
2.00
1.01
2.96
1.01
1.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)


172.2599
159.2374
149.3400
131.5084
130.6988
129.8419
128.3228
126.4440
126.3313
122.6217
119.3802
118.7719
114.6455
76.9995
74.4271
74.0323
73.6355
73.2421
55.1663
44.7327
39.1090
O
O
S
O
N
O
F3C
MeO
4f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

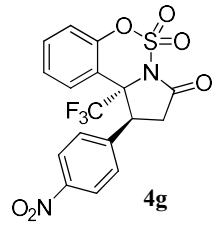

-70.5284
-76.6533
O
O
S
O
N
O
F3C
MeO
4f
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)


8.0951
8.0672
7.4081
7.3816
7.3554
7.2821
7.2598
7.2369
7.2329
7.2073
7.0772
7.0516
7.0260
6.8975
6.8709
4.4089
4.3800
3.5687
3.5399
3.5081
3.4791
2.6467
2.5862
1.86
1.02
4.97
1.01
1.01
1.00
1.03
1.00
f1 (ppm)

**4g**


171.0641
149.3178
147.5179
145.6596
132.2821
130.0159
129.2200
128.2672
126.7653
126.1815
124.6328
122.3609
119.9648
118.5321
118.0369
76.9998
73.9036
73.4987
73.0962
72.6950
45.0059
38.3936
29.6611
f1 (ppm)

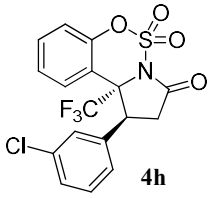
**4g**



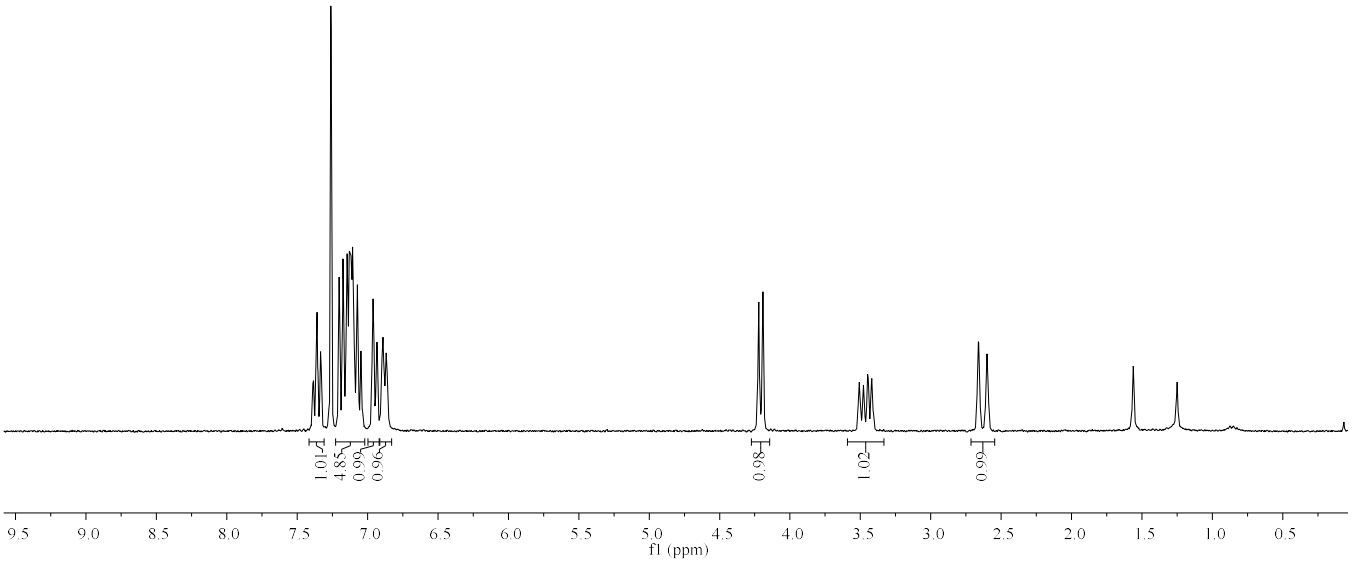
**4g**

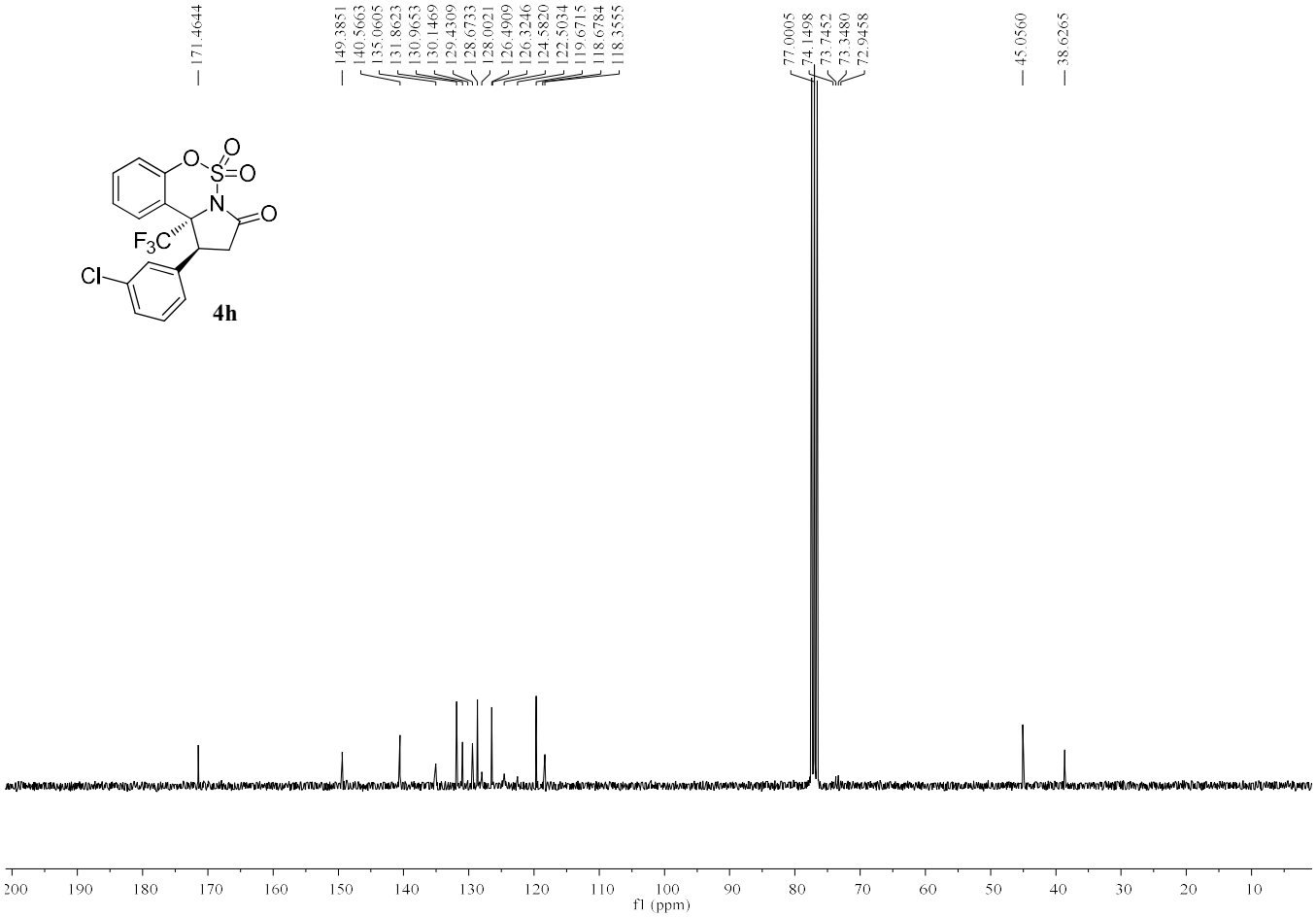

-76.8924
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-2
f1 (ppm)

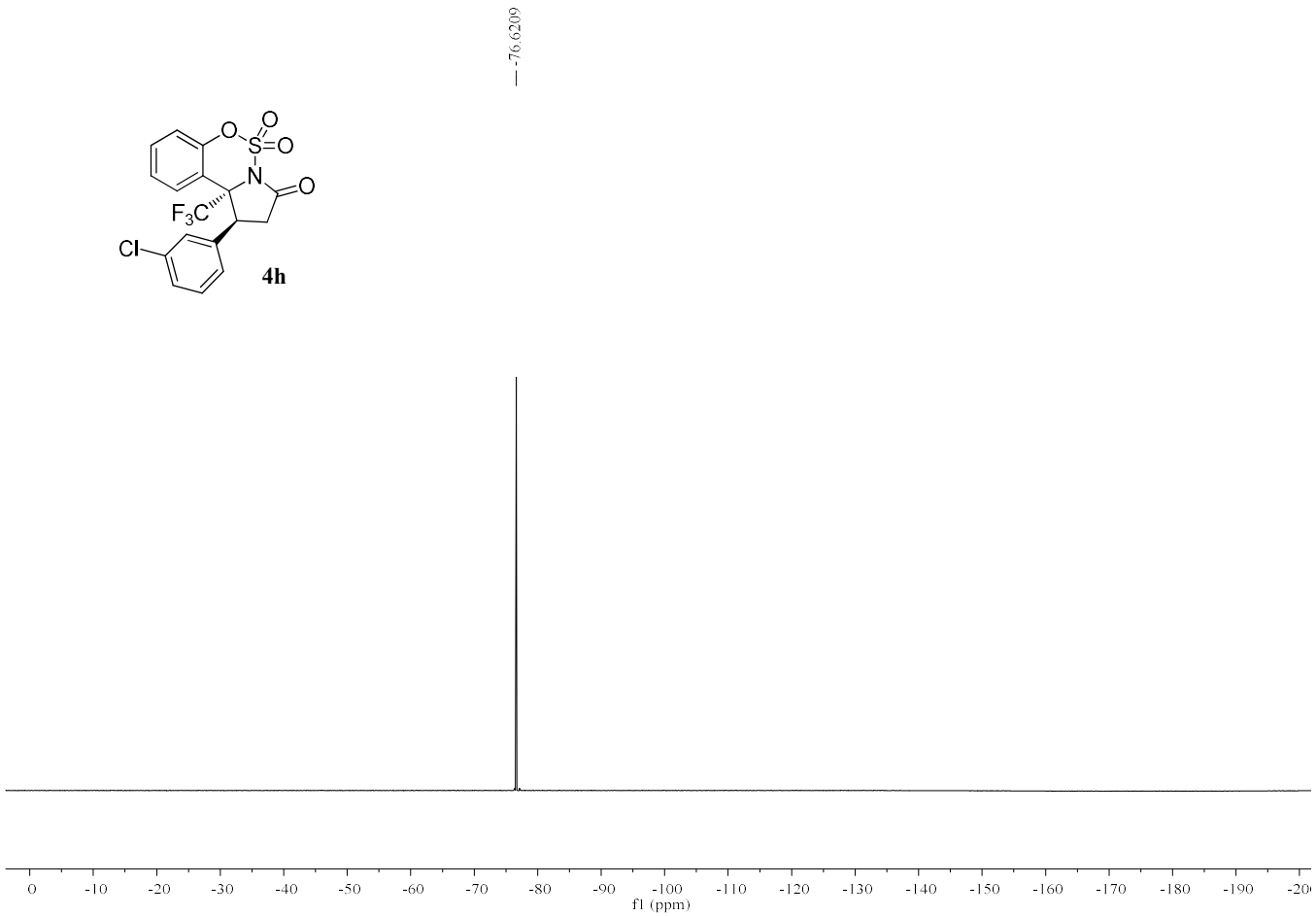

7.3877
7.3826
7.3601
7.3545
7.3352
7.3300
7.2024
7.1981
7.1730
7.1508
7.1458
7.1407
7.1289
7.1181
7.1048
7.0980
7.0937
7.0778
7.0718
7.0676
7.0465
7.0422
6.9602
6.9335
6.8913
6.8662
4.2221
4.1931
3.5095
3.5040
3.4801
3.4748
3.4486
3.4433
3.4198
3.4143
2.6612
2.6048
2.5971

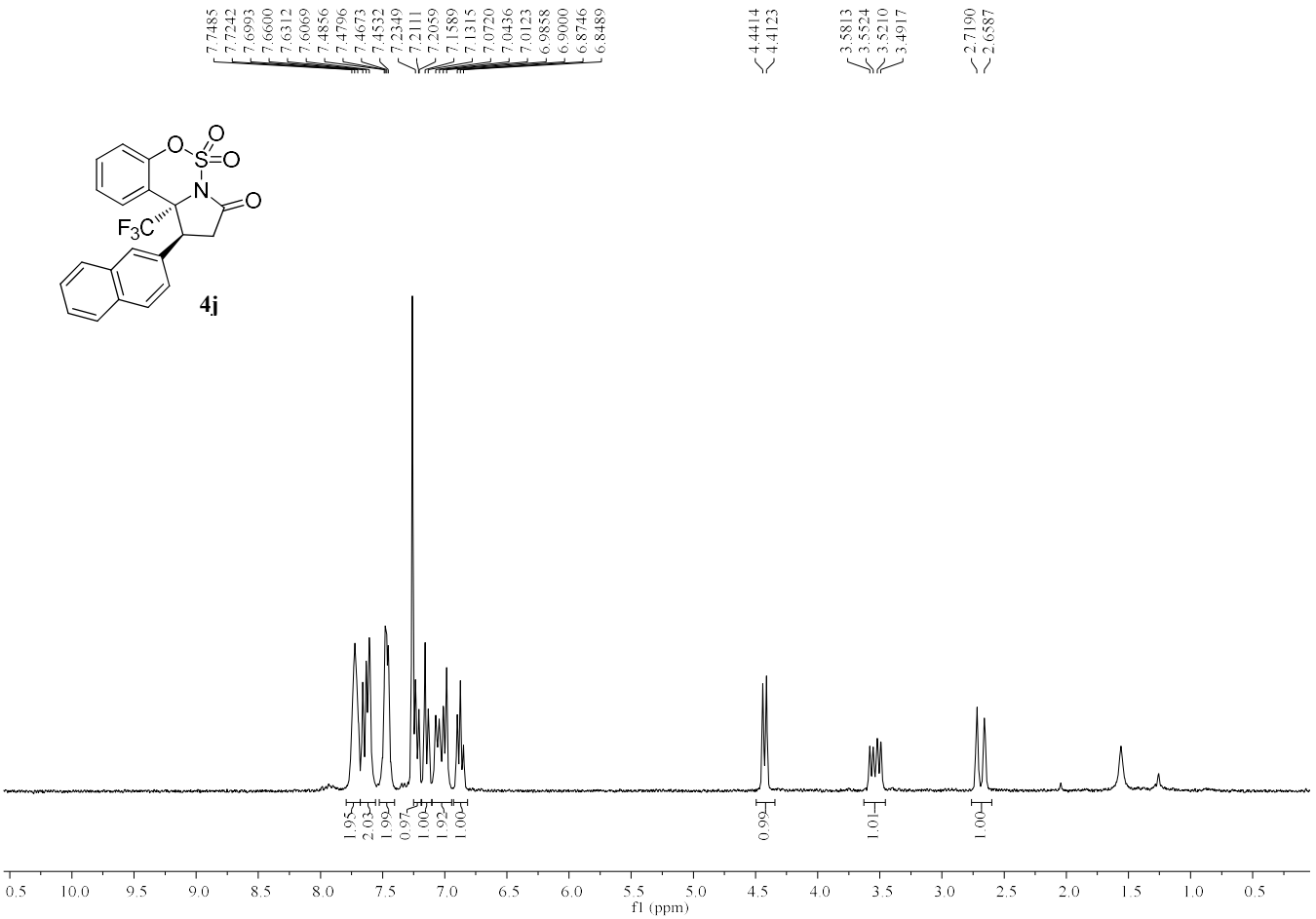


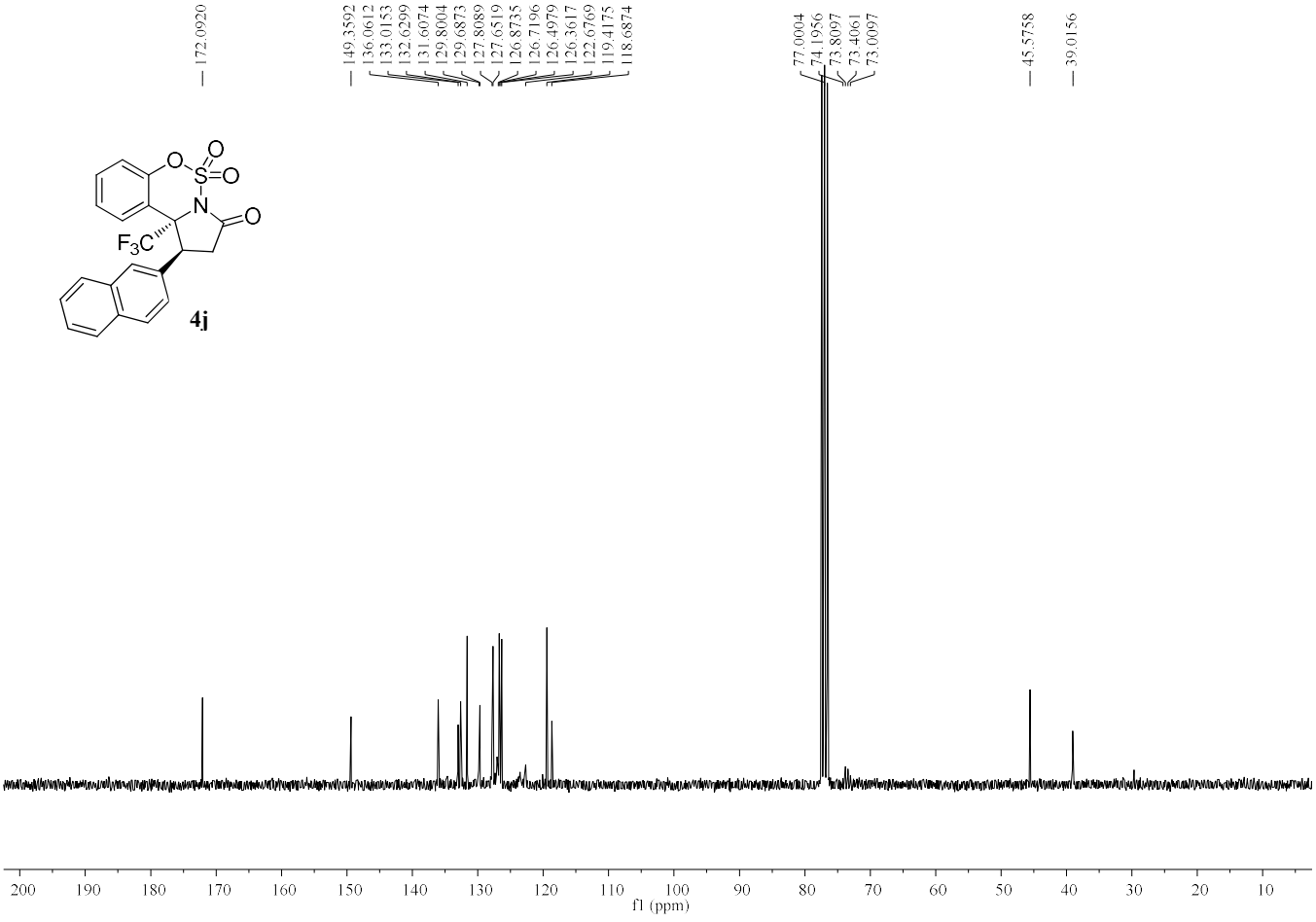
**4h**

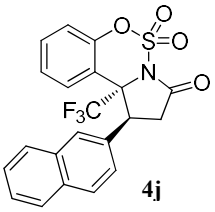

1.01
4.85
0.96
0.96
0.98
1.02
0.99
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


171.4644
149.3851
140.5663
135.0605
131.8623
130.9653
130.1469
129.4309
128.6733
128.0021
126.4909
126.3246
124.5820
122.5034
119.6715
118.6784
118.3555
77.0005
74.1498
73.7452
73.3480
72.9458
45.0560
38.6265
O
S
O
O
N
O
F3C
Cl
4h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


-76.6209
O
S
O
O
N
O
F3C
Cl
4h
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-20
f1 (ppm)


7.7485
7.7242
7.6993
7.6600
7.6312
7.6069
7.4856
7.4796
7.4673
7.4532
7.2349
7.2111
7.2059
7.1589
7.1315
7.0720
7.0436
7.0123
6.9858
6.9000
6.8746
6.8489
4.4414
4.4123
3.5813
3.5524
3.5210
3.4917
2.7190
2.6587
4j
1.95
2.03
1.99
0.97
1.00
1.92
1.00
0.99
1.01
1.00
f1 (ppm)

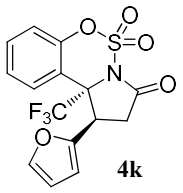

172.0920
149.3592
136.0612
133.0153
132.6299
131.6074
129.8004
129.6873
127.8089
127.6519
126.8735
126.7196
126.4979
126.3617
122.6769
119.4175
118.6874
77.0004
74.1956
73.8097
73.4061
73.0097
45.5758
39.0156
4j
f1 (ppm)



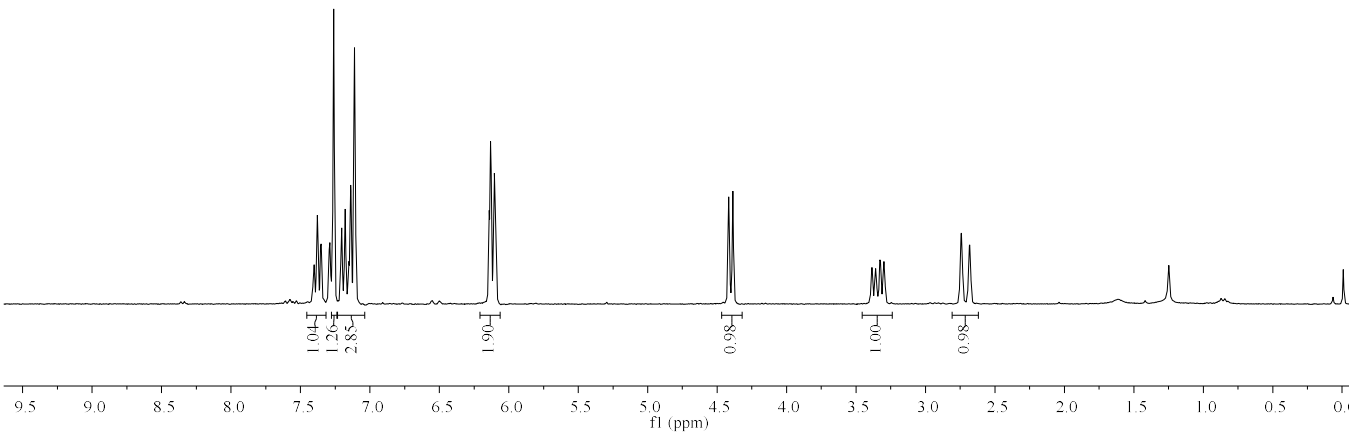
**4j**

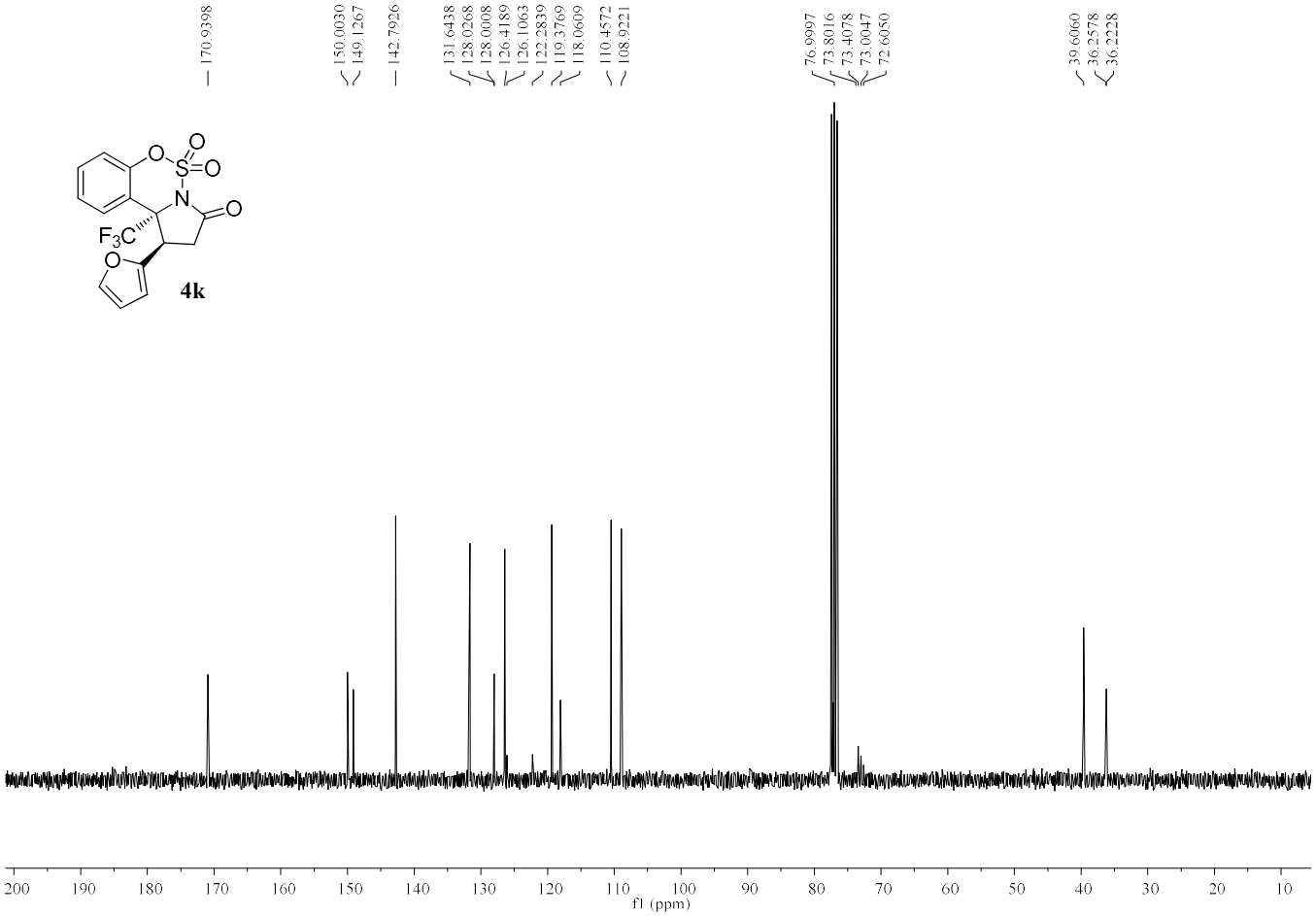

-76.8924
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-2
f1 (ppm)

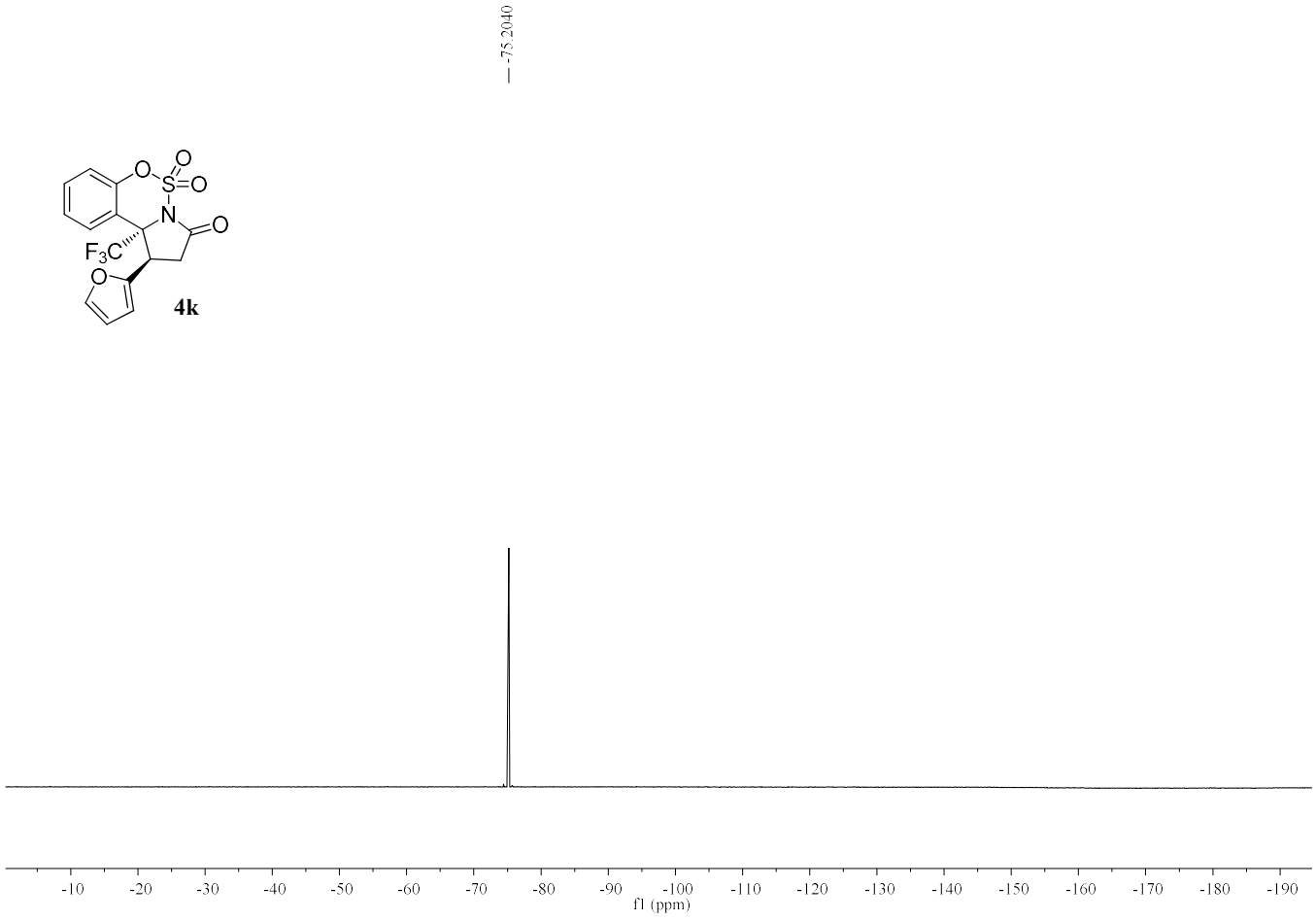

7.4075
7.4019
7.3827
7.3770
7.3555
7.3499
7.2604
7.2061
7.2018
7.1810
7.1765
7.1549
7.1508
7.1402
7.1360
7.1132
7.1078
6.1416
6.1305
6.1094
6.1033
6.0981
6.0923
4.4159
4.3873
3.3886
3.3834
3.3788
3.3599
3.3547
3.3501
3.3301
3.3237
3.3007
3.2956
3.2910
2.7455
2.7384
2.7315
2.6863
2.6788



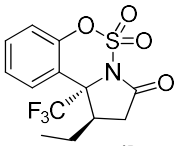
**4k**


1.04
1.26
2.85
1.90
0.98
1.00
0.98
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

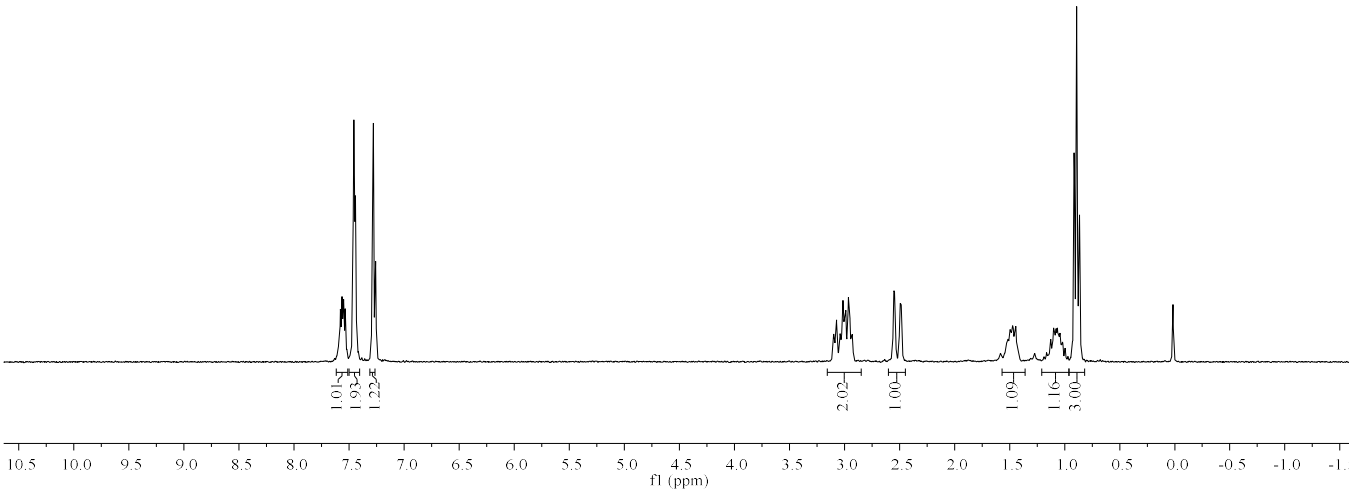

170.9398
150.0030
149.1267
142.7926
131.6438
128.0268
128.0008
126.4189
126.1063
122.2839
119.3769
118.0609
110.4572
108.9221
76.9997
73.8016
73.4078
73.0047
72.6050
39.6060
36.2578
36.2228
4k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

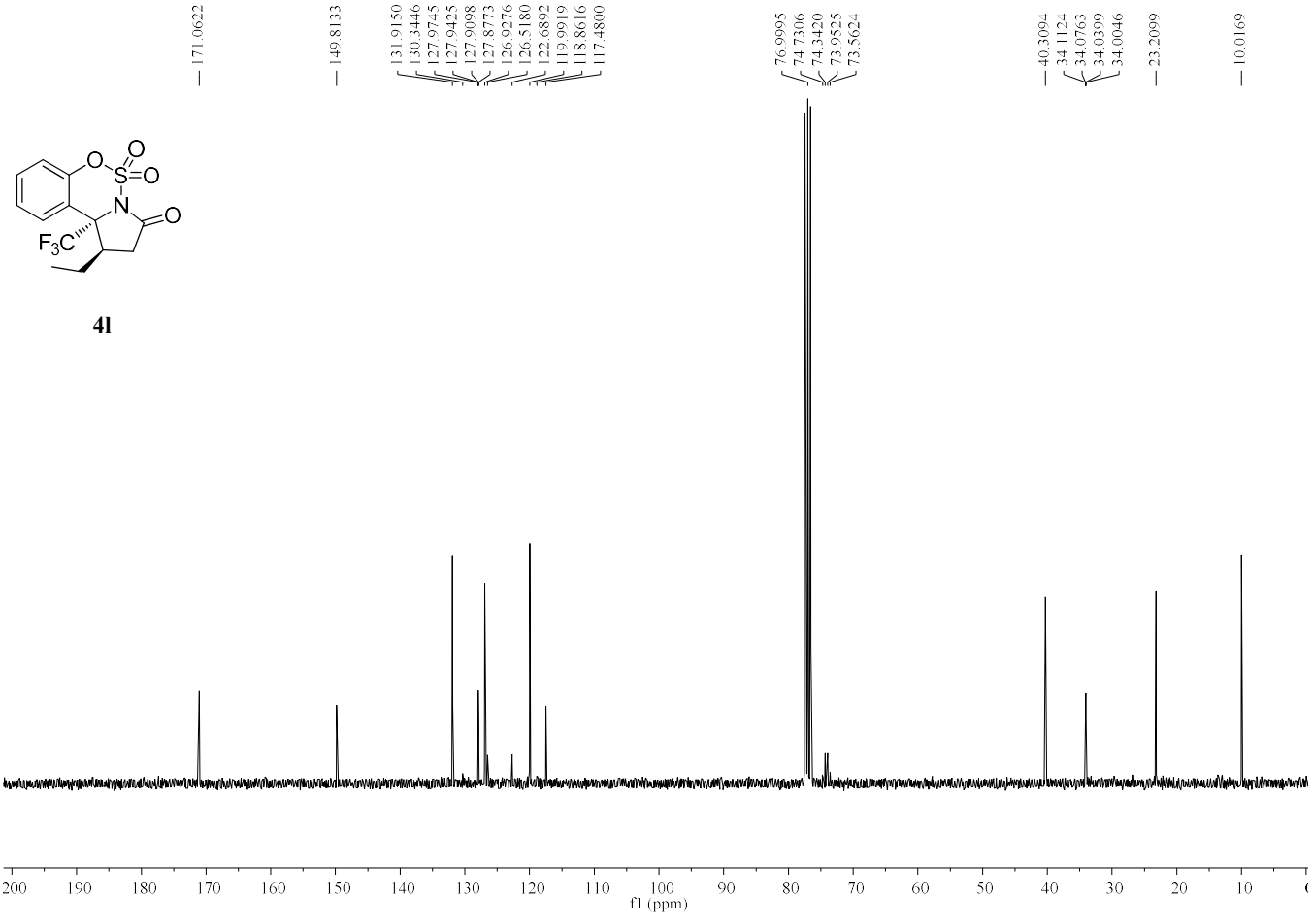

-75.2040
4k
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
f1 (ppm)


7.5935
7.5786
7.5642
7.5505
7.5357
7.5214
7.4576
7.4424
7.2869
7.2803
3.1023
3.0958
3.0779
3.0726
3.0673
3.0392
3.0198
3.0144
3.0092
2.9979
2.9881
2.9730
2.9631
2.9527
2.9380
2.9280
2.5591
2.5516
2.5442
2.5369
2.5006
2.4934
2.4864
2.4793
1.5261
1.5179
1.5018
1.4967
1.4728
1.4443
1.1260
1.1017
1.0896
1.0786
1.0658
1.0542
1.0432
1.0196
0.9968
0.9143
0.8901
0.8658


O
O
S
O
N
O
F3C

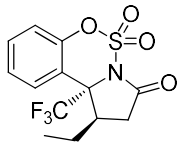
**4l**


1.01
1.93
1.22
2.02
1.00
1.09
1.16
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


171.0622
149.8133
131.9150
130.3446
127.9745
127.9425
127.9098
127.8773
126.9276
126.5180
122.6892
119.9919
118.8616
117.4800
76.9995
74.7306
74.3420
73.9525
73.5624
40.3094
34.1124
34.0763
34.0399
34.0046
23.2099
10.0169
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

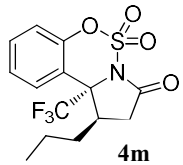

O
O
S
O
N
O
F3C

**4l**

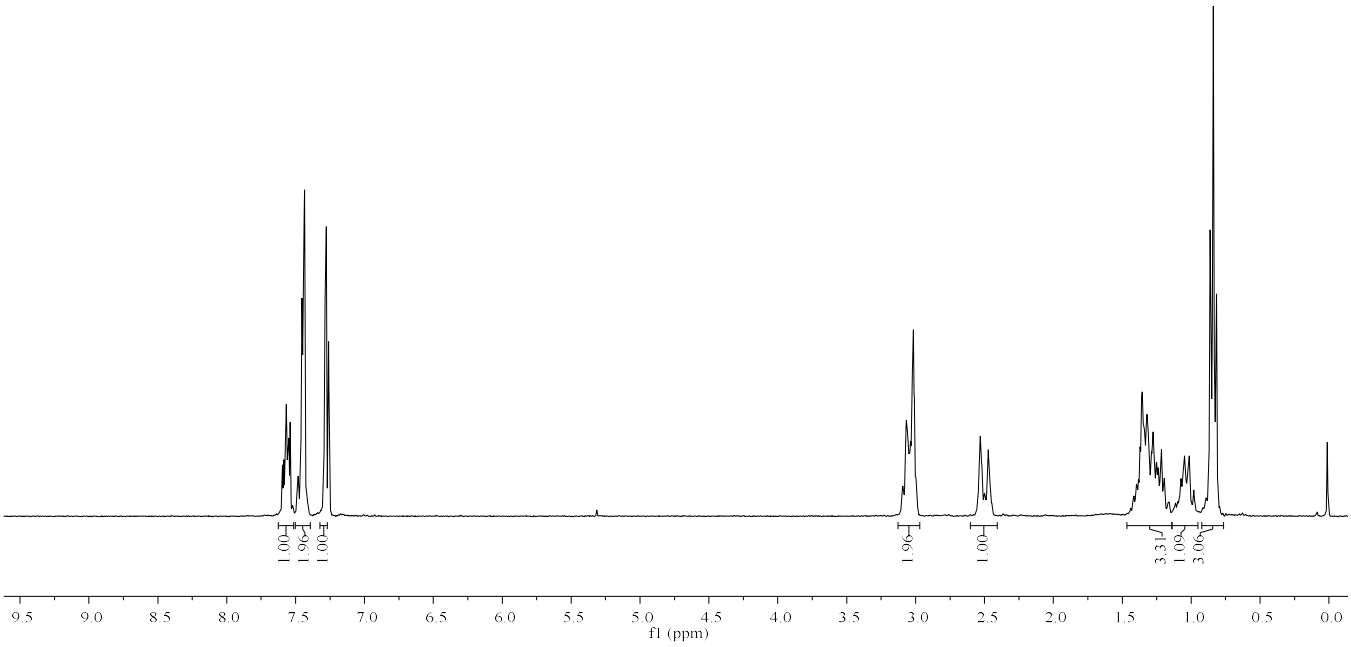


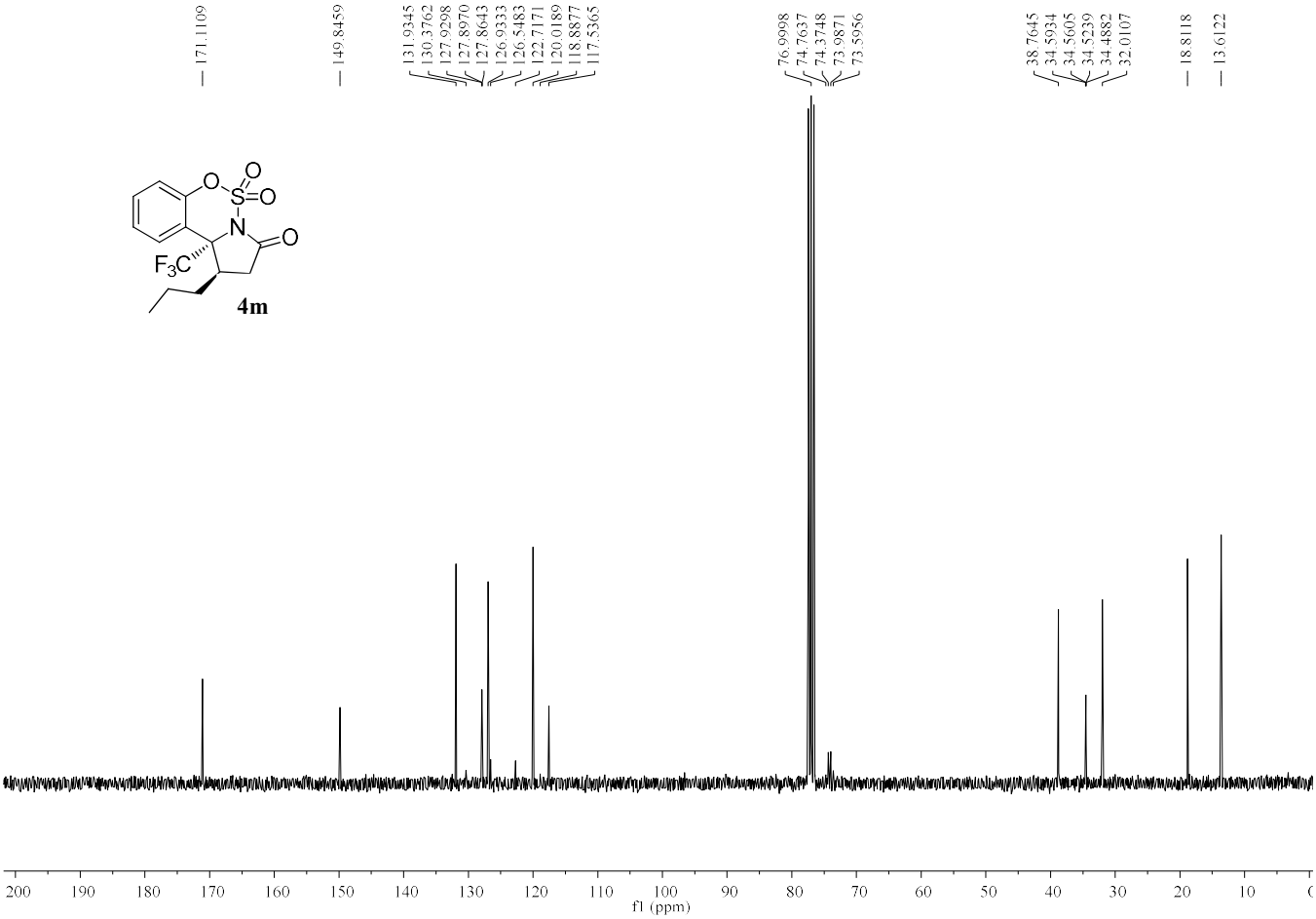
**4l**

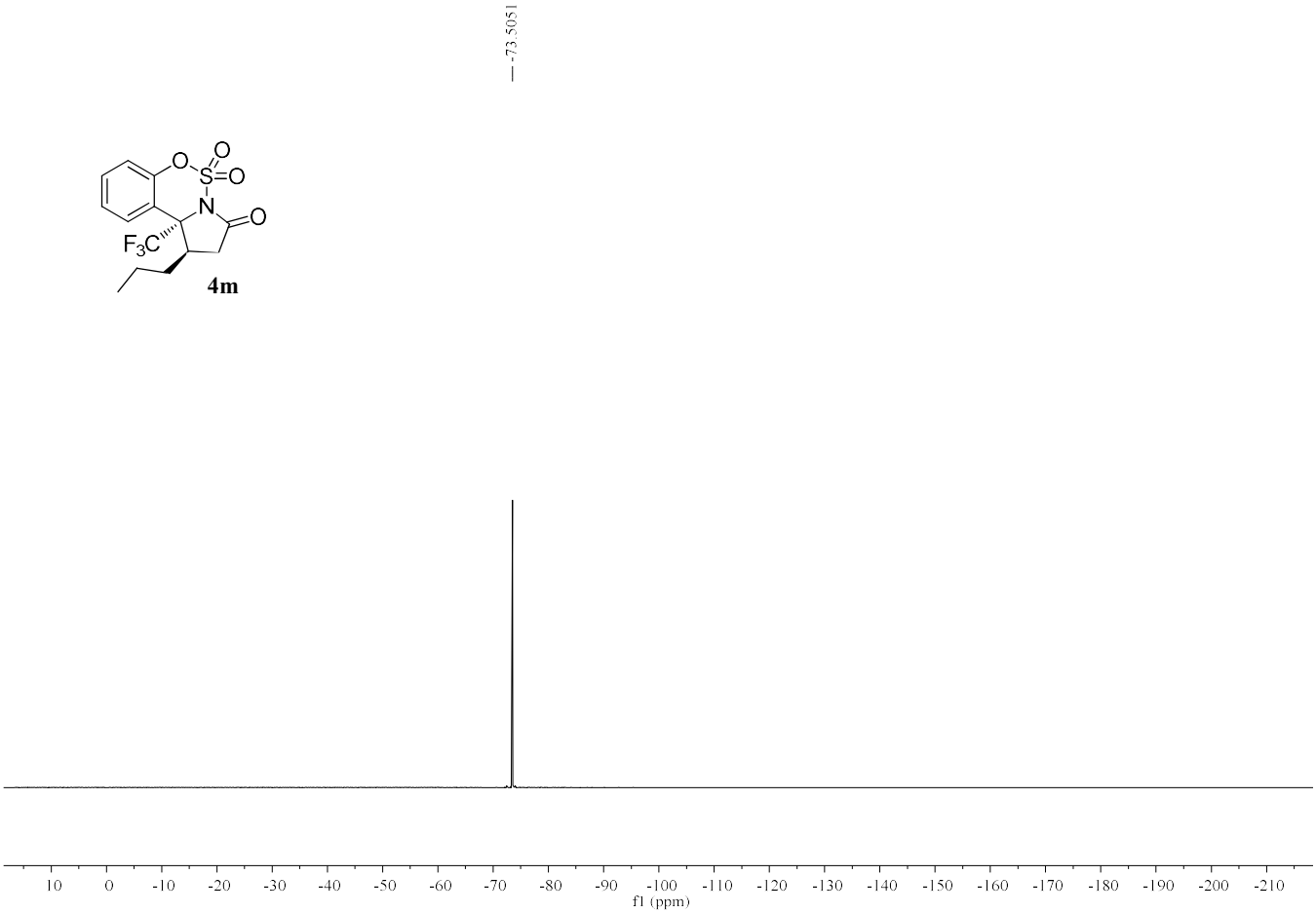

-73.5556
f1 (ppm)
7.5962
7.5864
7.5757
7.5670
7.5582
7.5489
7.5384
7.4840
7.4801
7.4575
7.4535
7.4450
7.4355
7.4172
7.2879
7.2781
3.0936
3.0688
3.0587
3.0504
3.0447
3.0367
3.0241
3.0154
2.9983
2.9916
2.5503
2.5397
2.5303
2.5229
2.5158
2.4989
2.4790
2.4727
2.4653
2.4558
1.4165
1.3960
1.3831
1.3716
1.3603
1.3547
1.3470
1.3423
1.3363
1.3192
1.3048
1.2854
1.2764
1.2671
1.2547
1.2503
1.2403
1.2254
1.2164
1.2019
1.1937
1.1694
1.1596
1.1134
1.0981
1.0742
1.0591
1.0477
1.0222
1.0126
0.9810
0.8612
0.8389
0.8162

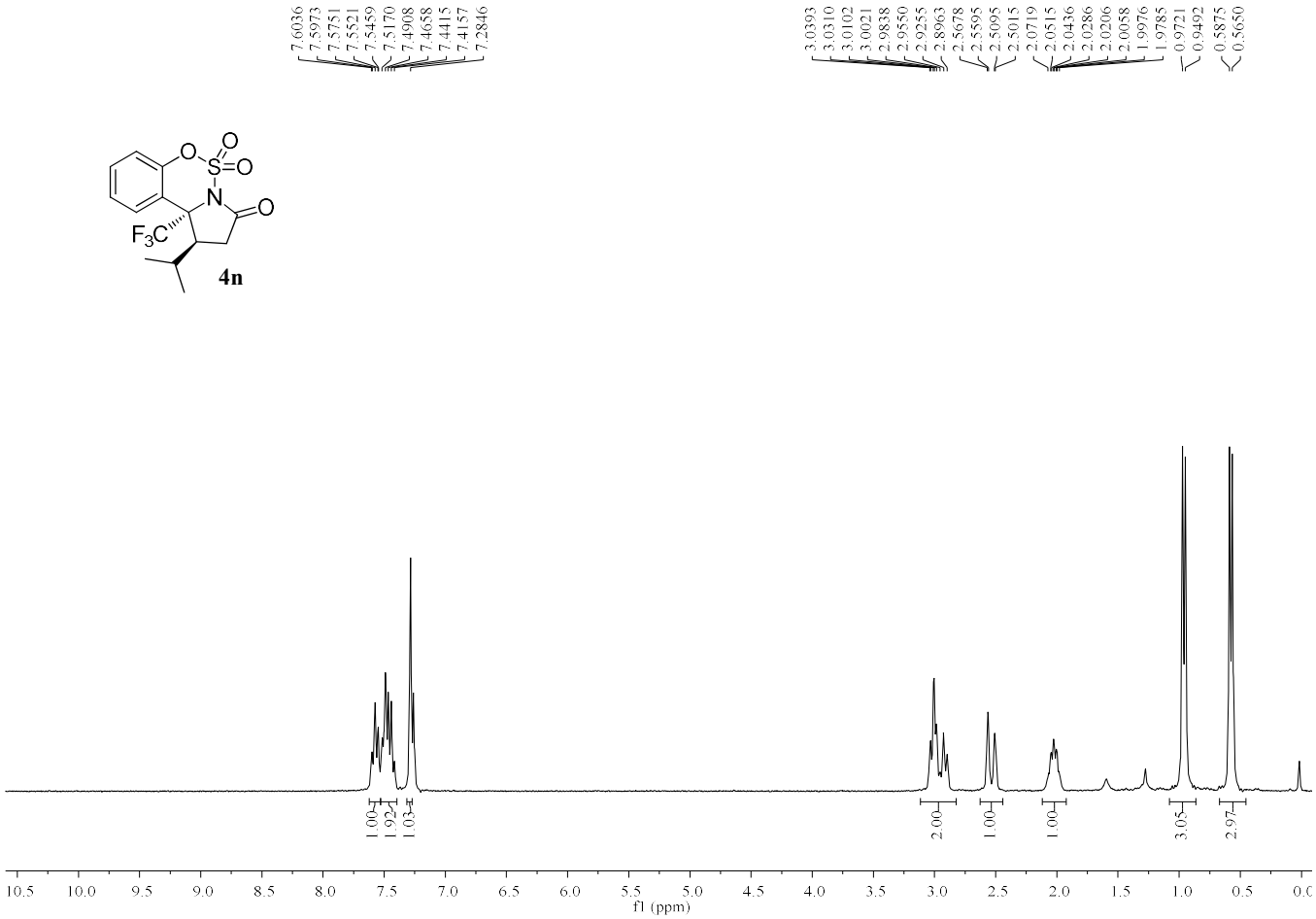


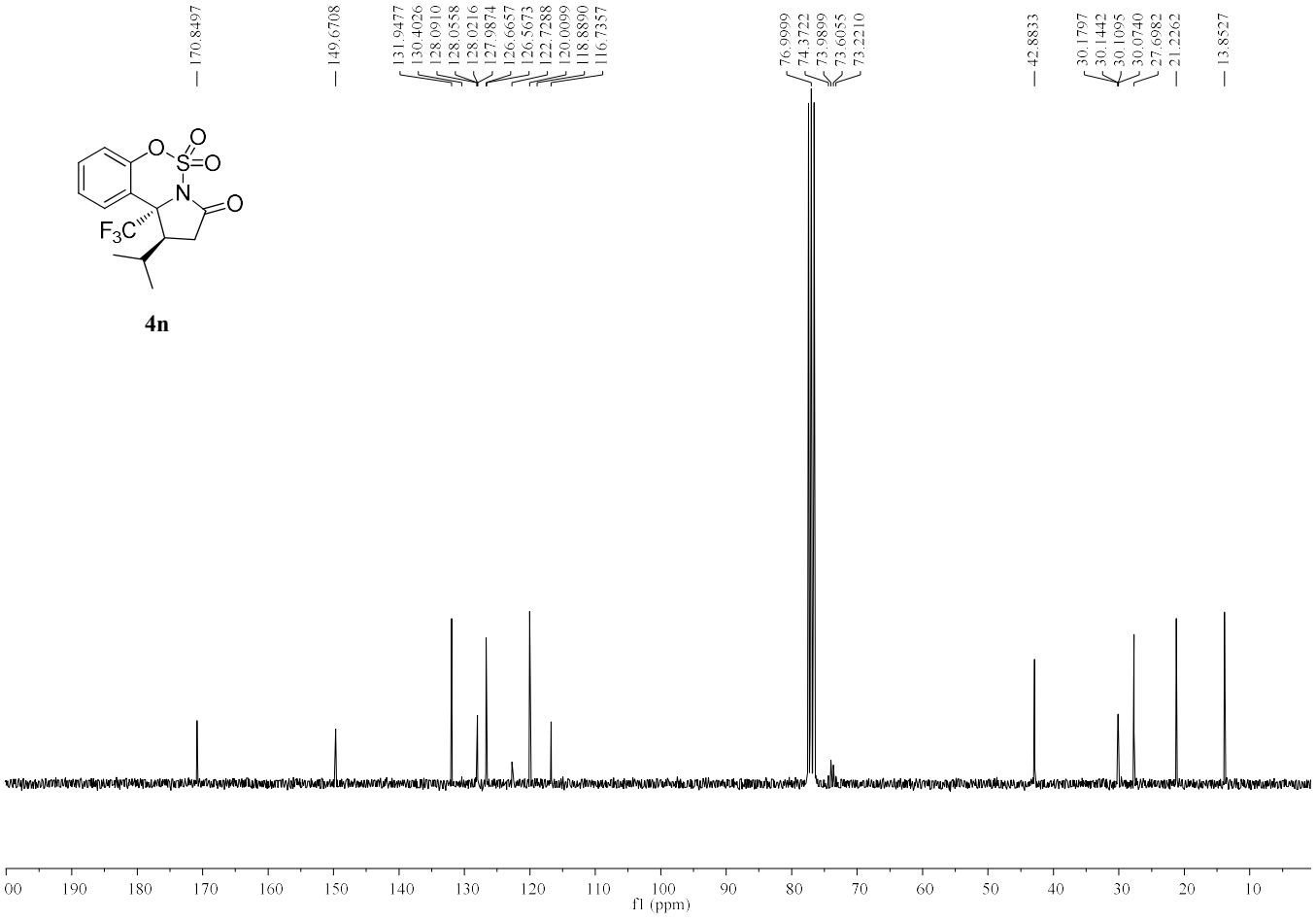
**4m**

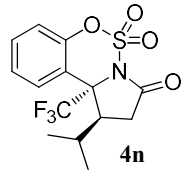

1.00
1.96
1.00
1.96
1.00
3.31
1.09
3.06
f1 (ppm)


171.1109
149.8459
131.9345
130.3762
127.9298
127.8970
127.8643
126.9333
126.5483
122.7171
120.0189
118.8877
117.5365
76.9998
74.7637
74.3748
73.9871
73.5956
38.7645
34.5934
34.5605
34.5239
34.4882
32.0107
18.8118
13.6122
O
O
S
O
N
O
F3C
4m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


-73.5051
O
O
S
O
N
O
F3C
4m
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

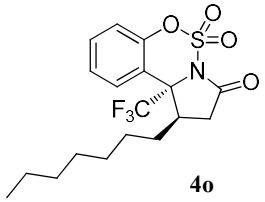

7.6036
7.5973
7.5751
7.5521
7.5459
7.5170
7.4908
7.4658
7.4415
7.4157
7.2846
3.0393
3.0310
3.0102
3.0021
2.9838
2.9550
2.9255
2.8963
2.5678
2.5595
2.5095
2.5015
2.0719
2.0515
2.0436
2.0286
2.0206
2.0058
1.9976
1.9785
0.9721
0.9492
0.5875
0.5650
O
O
S
O
N
O
F3C
4n
1.00
1.92
1.03
2.00
1.00
1.00
3.05
2.97
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.8497
149.6708
131.9477
130.4026
128.0910
128.0558
128.0216
127.9874
126.6657
126.5673
122.7288
120.0099
118.8890
116.7357
76.9999
74.3722
73.9899
73.6055
73.2210
42.8833
30.1797
30.1442
30.1095
30.0740
27.6982
21.2262
13.8527
O
O
S
O
N
O
F3C
4n
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

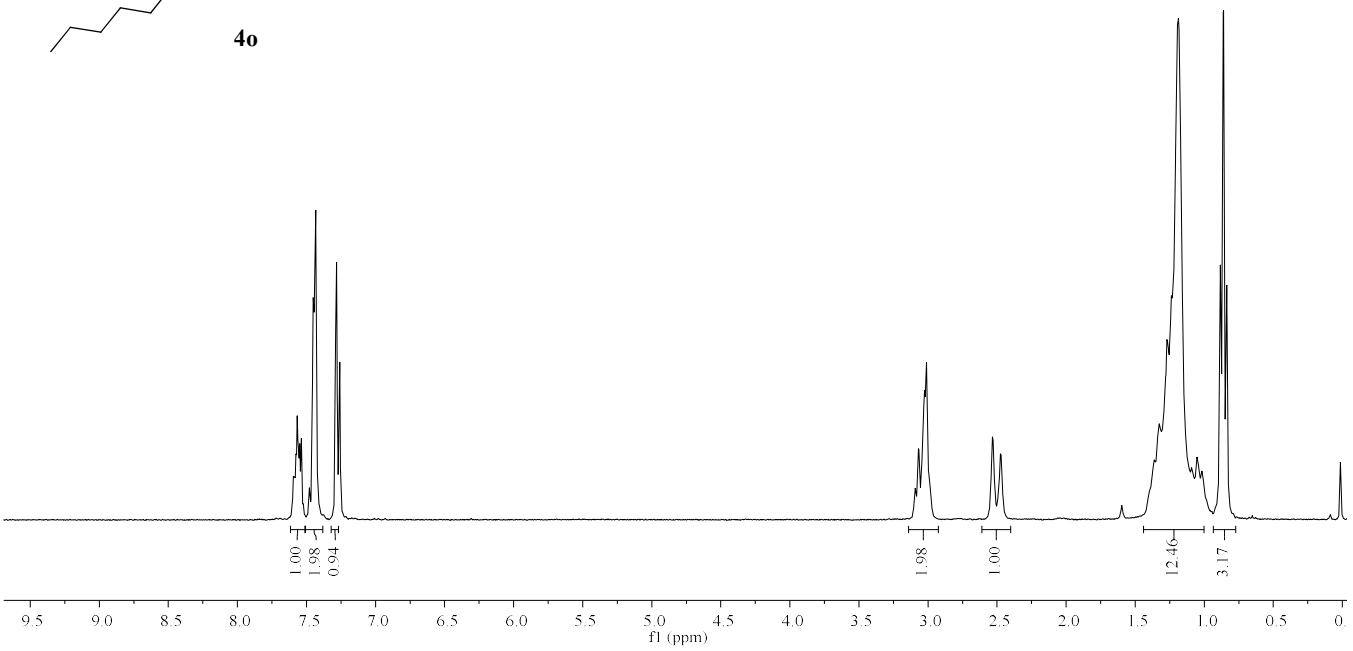


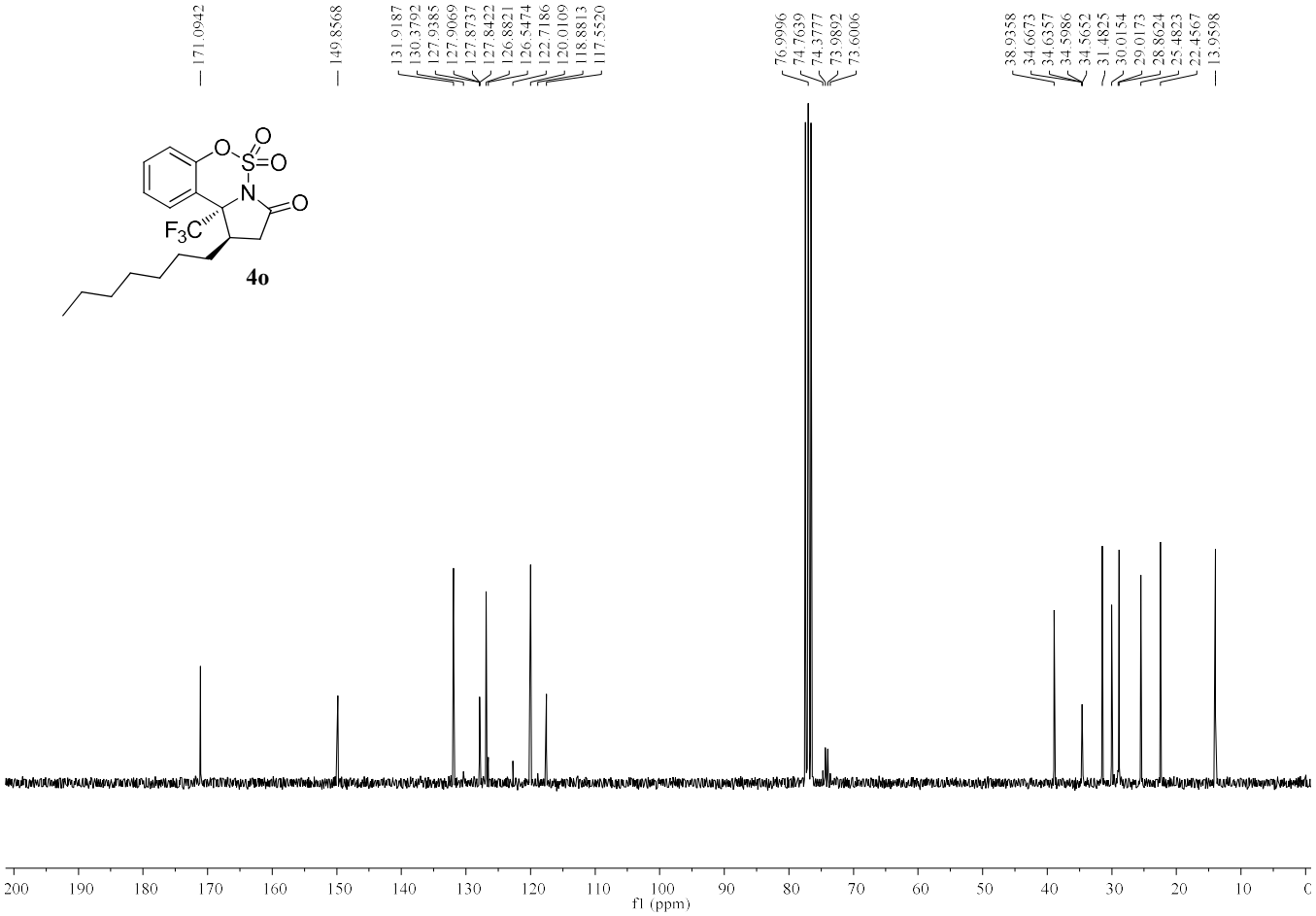
**4n**

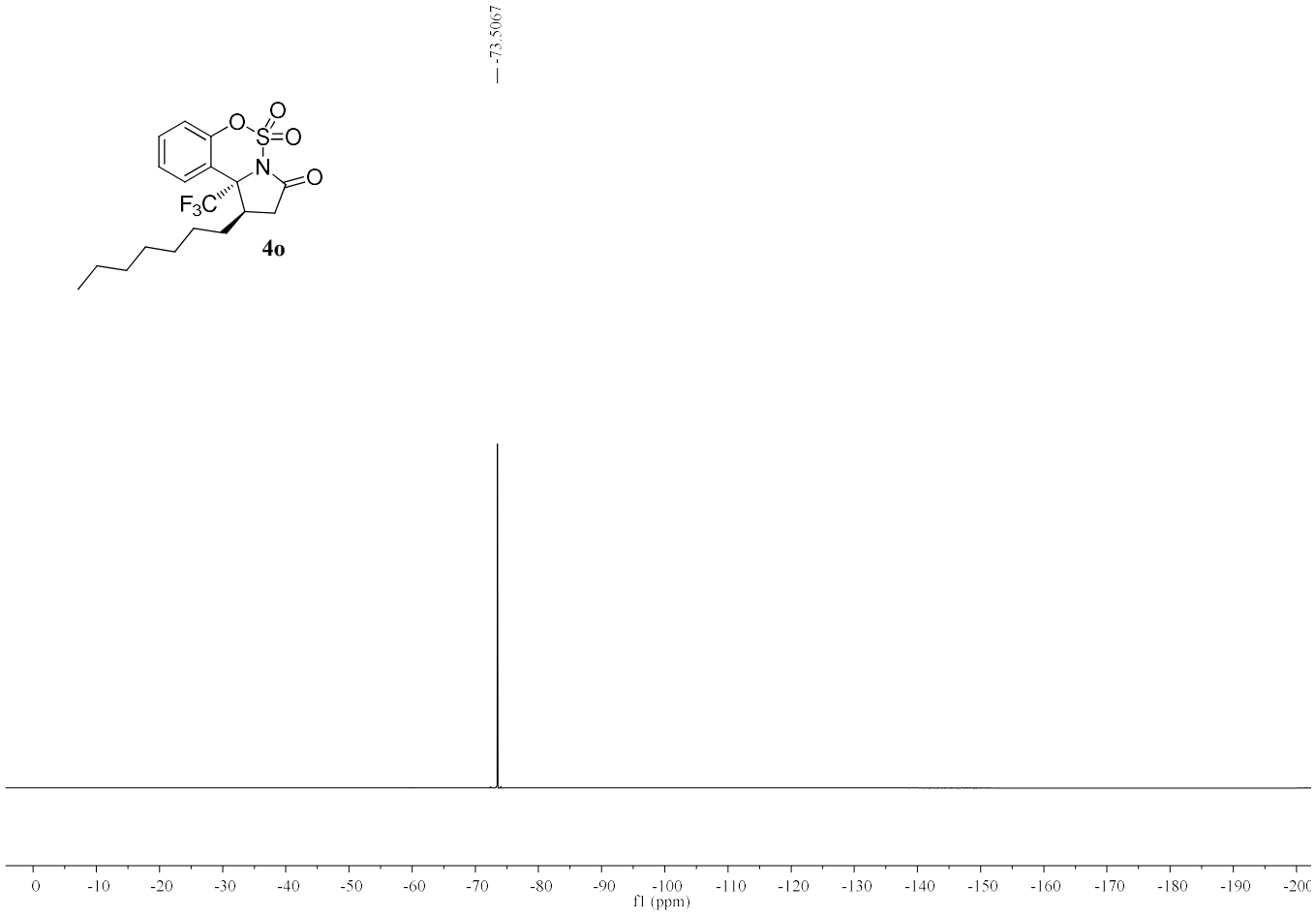

-73.8753
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)
7.5955
7.5853
7.5756
7.5663
7.5569
7.5488
7.5377
7.4791
7.4520
7.4430
7.4332
7.2875
7.2807
3.0939
3.0686
3.0448
3.0268
3.0113
3.0026
2.9872
2.9786
2.5349
2.5266
2.5191
2.4893
2.4778
2.4706
2.4626
1.3686
1.3600
1.3392
1.2777
1.2701
1.2613
1.2386
1.1978
1.1837
1.1670
1.0902
1.0763
1.0526
1.0406
1.0160
1.0037
0.8828
0.8612
0.8378

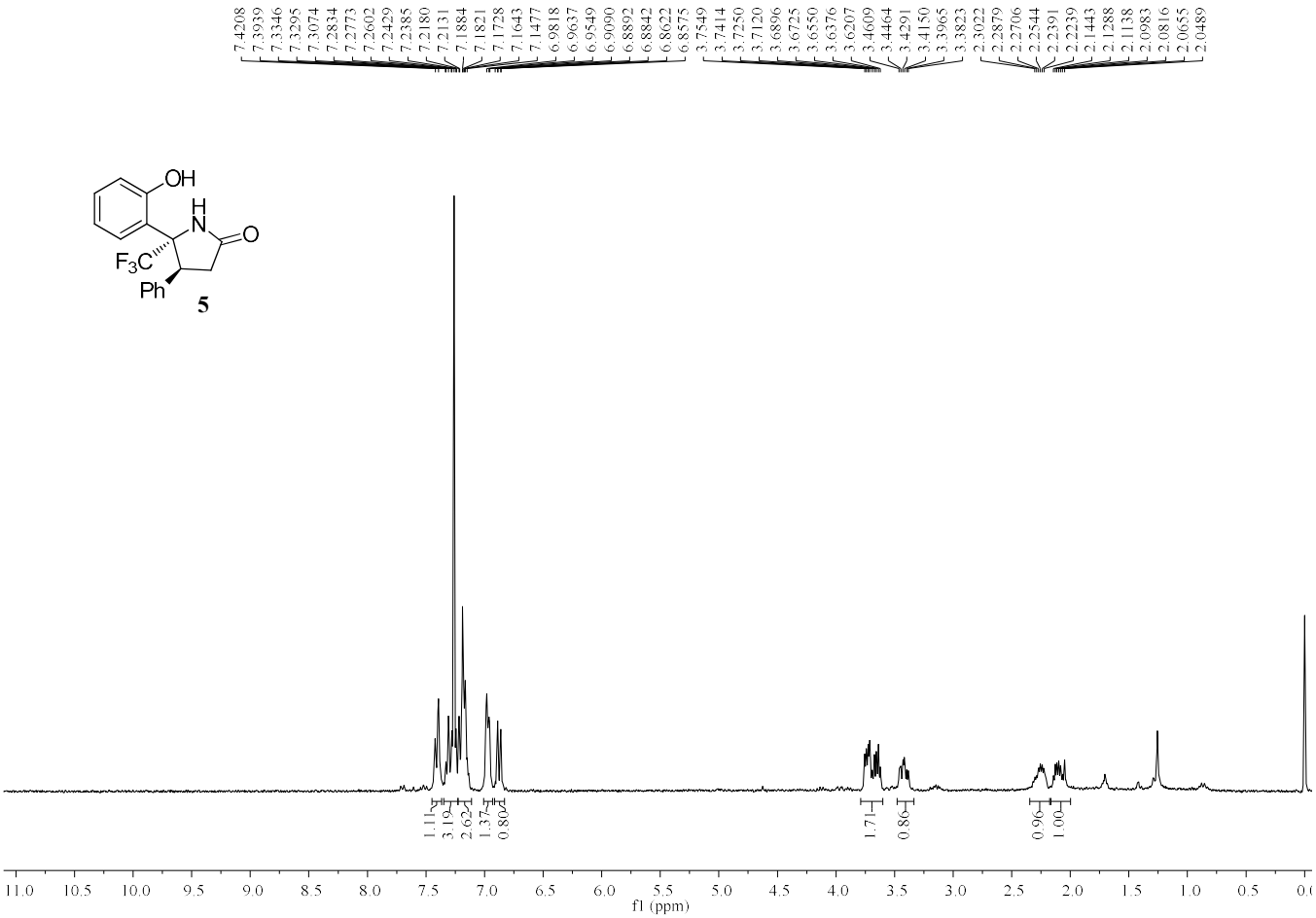


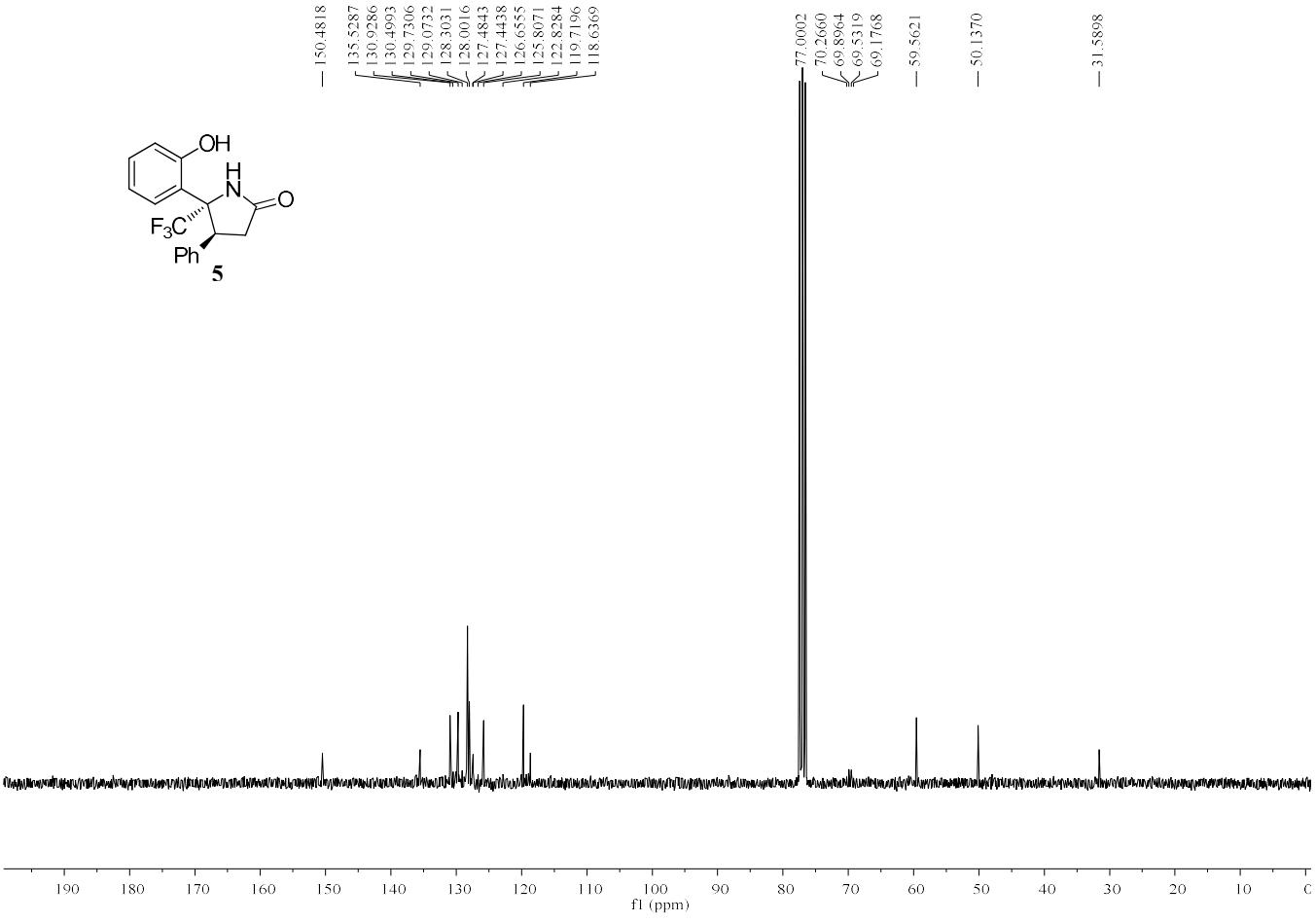
**4o**

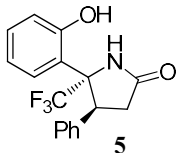

1.00
1.98
0.94
1.98
1.00
12.46
3.17
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


171.0942
149.8568
131.9187
130.3792
127.9385
127.9069
127.8737
127.8422
126.8821
126.5474
122.7186
120.0109
118.8813
117.5520
76.9996
74.7639
74.3777
73.9892
73.6006
38.9358
34.6673
34.6357
34.5986
34.5652
31.4825
30.0154
29.0173
28.8624
25.4823
22.4567
13.9598
O
S
O
O
N
O
F3C
4o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-73.5067
O
S
O
O
N
O
F3C
4o
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

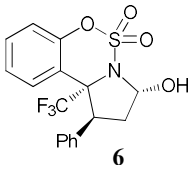

7.4208
7.3939
7.3346
7.3295
7.3074
7.2834
7.2773
7.2602
7.2429
7.2385
7.2180
7.2131
7.1884
7.1821
7.1728
7.1643
7.1477
6.9818
6.9637
6.9549
6.9090
6.8892
6.8842
6.8622
6.8575
3.7549
3.7414
3.7250
3.7120
3.6896
3.6725
3.6550
3.6376
3.6207
3.4609
3.4464
3.4291
3.4150
3.3965
3.3823
2.3022
2.2879
2.2706
2.2544
2.2391
2.2239
2.1443
2.1288
2.1138
2.0983
2.0816
2.0655
2.0489
OH
H
N
O
F3C
Ph
5
1.11
3.19
2.62
1.37
0.80
1.71
0.86
0.96
1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


150.4818
135.5287
130.9286
130.4993
129.7306
129.0732
128.3031
128.0016
127.4843
127.4438
126.6555
125.8071
122.8284
119.7196
118.6369
77.0002
70.2660
69.8964
69.5319
69.1768
59.5621
50.1370
31.5898
OH
H
N
O
F3C
Ph
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

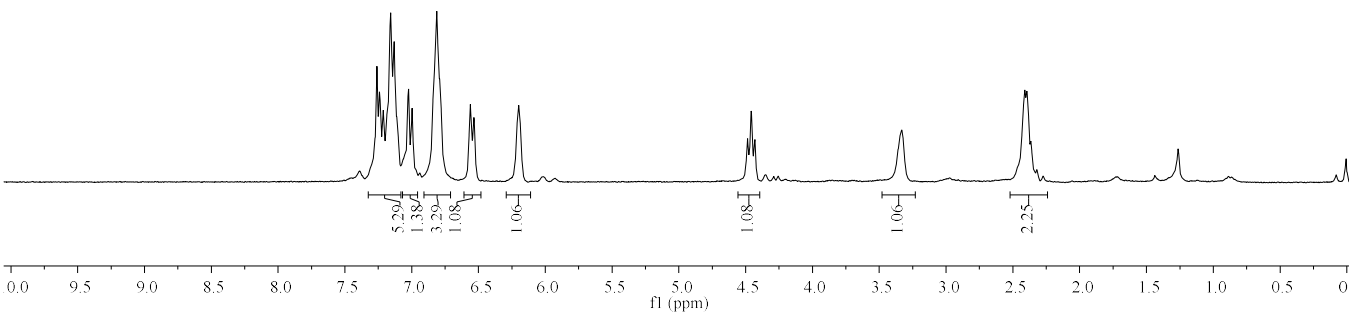

OH
H
N
O
F3C
Ph

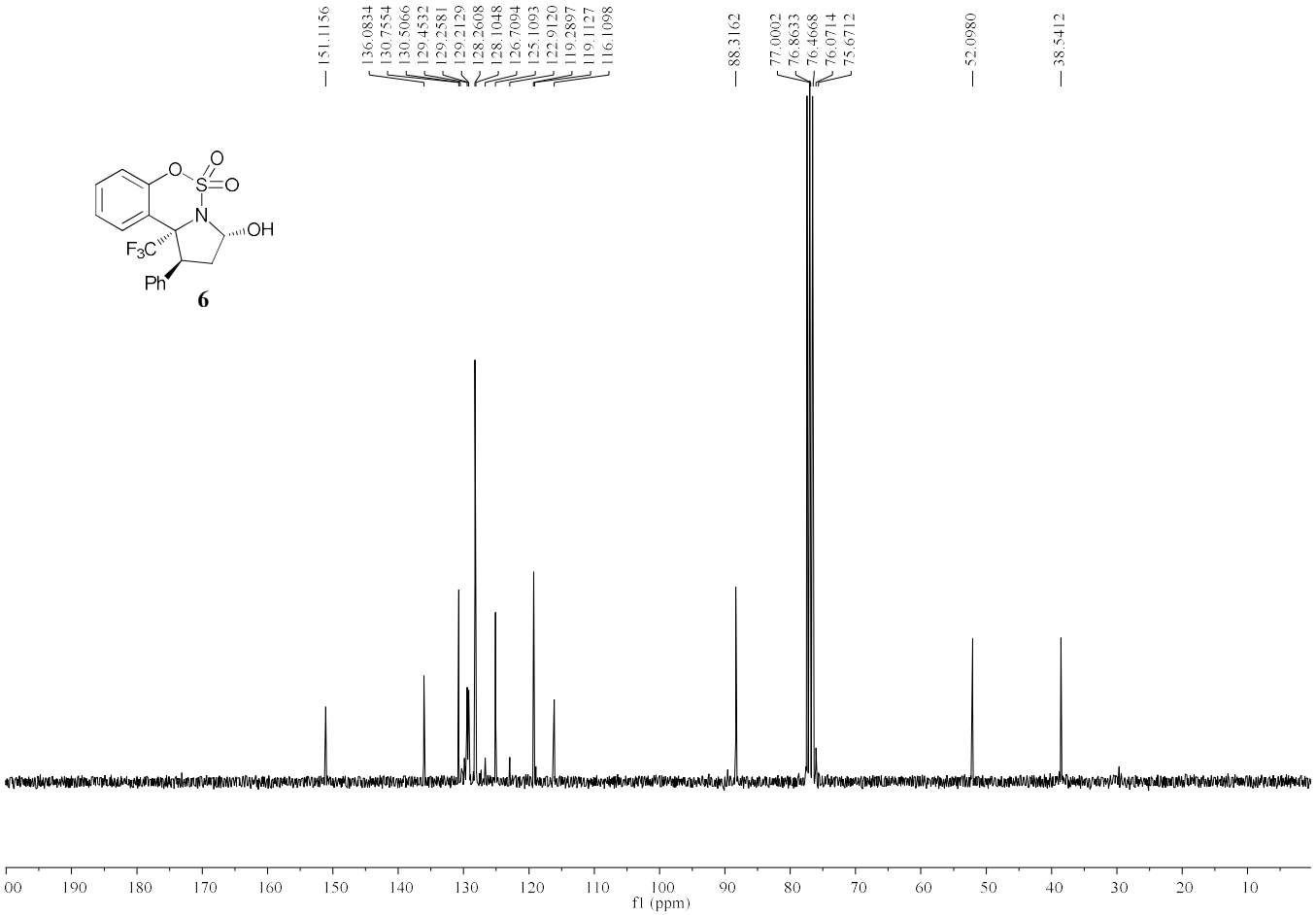
**5**

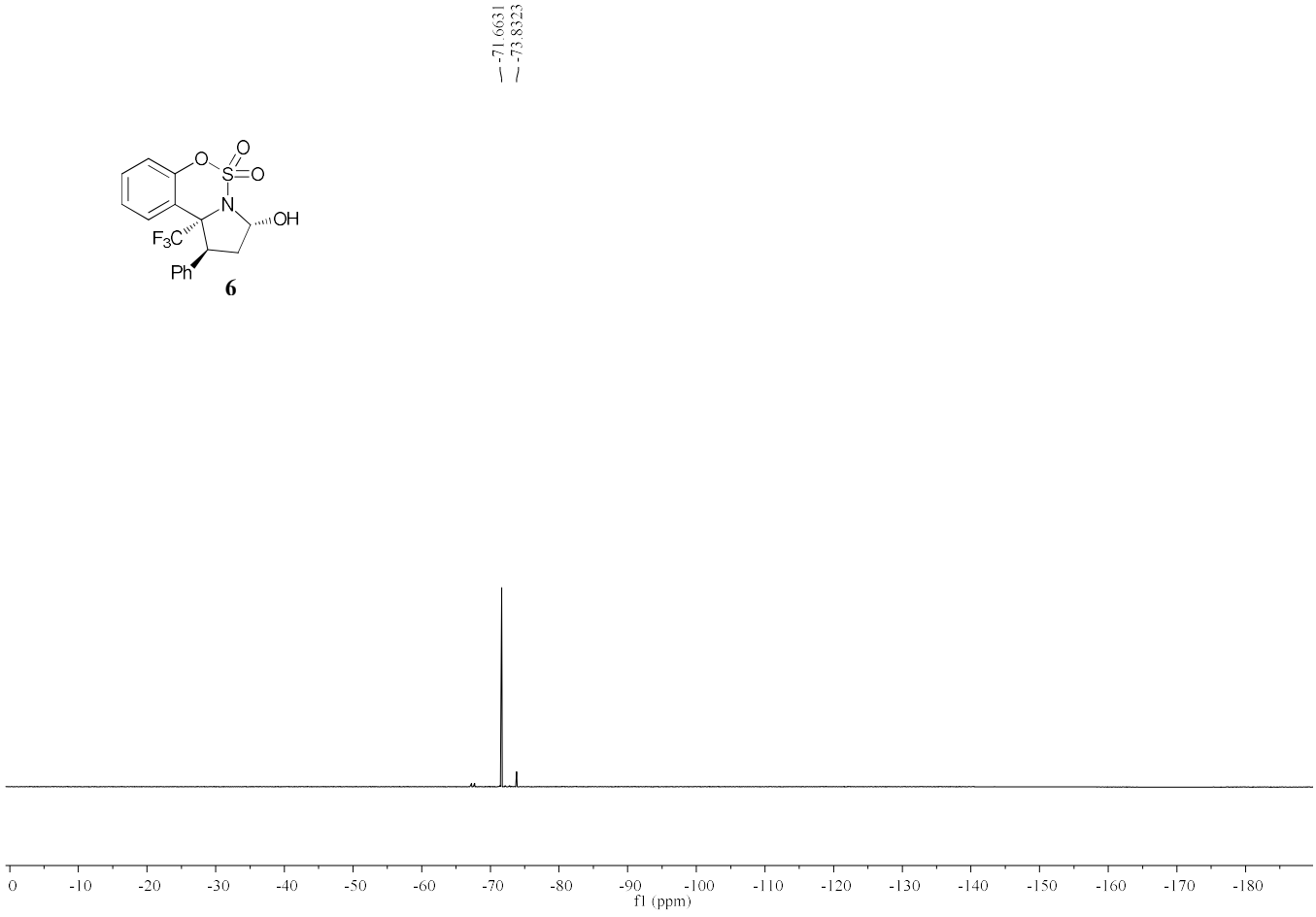

-67.6677
-68.7605
f1 (ppm)
7.2405
7.2127
7.1856
7.1582
7.1308
7.1085
7.0667
7.0511
7.0241
6.9966
6.8359
6.8115
6.7857
6.5613
6.5342
6.2137
6.1984
6.1835
4.4861
4.4585
4.4311
3.3394
3.3277
2.4706
2.4585
2.4278
2.4130
2.3925
2.3803
2.3631

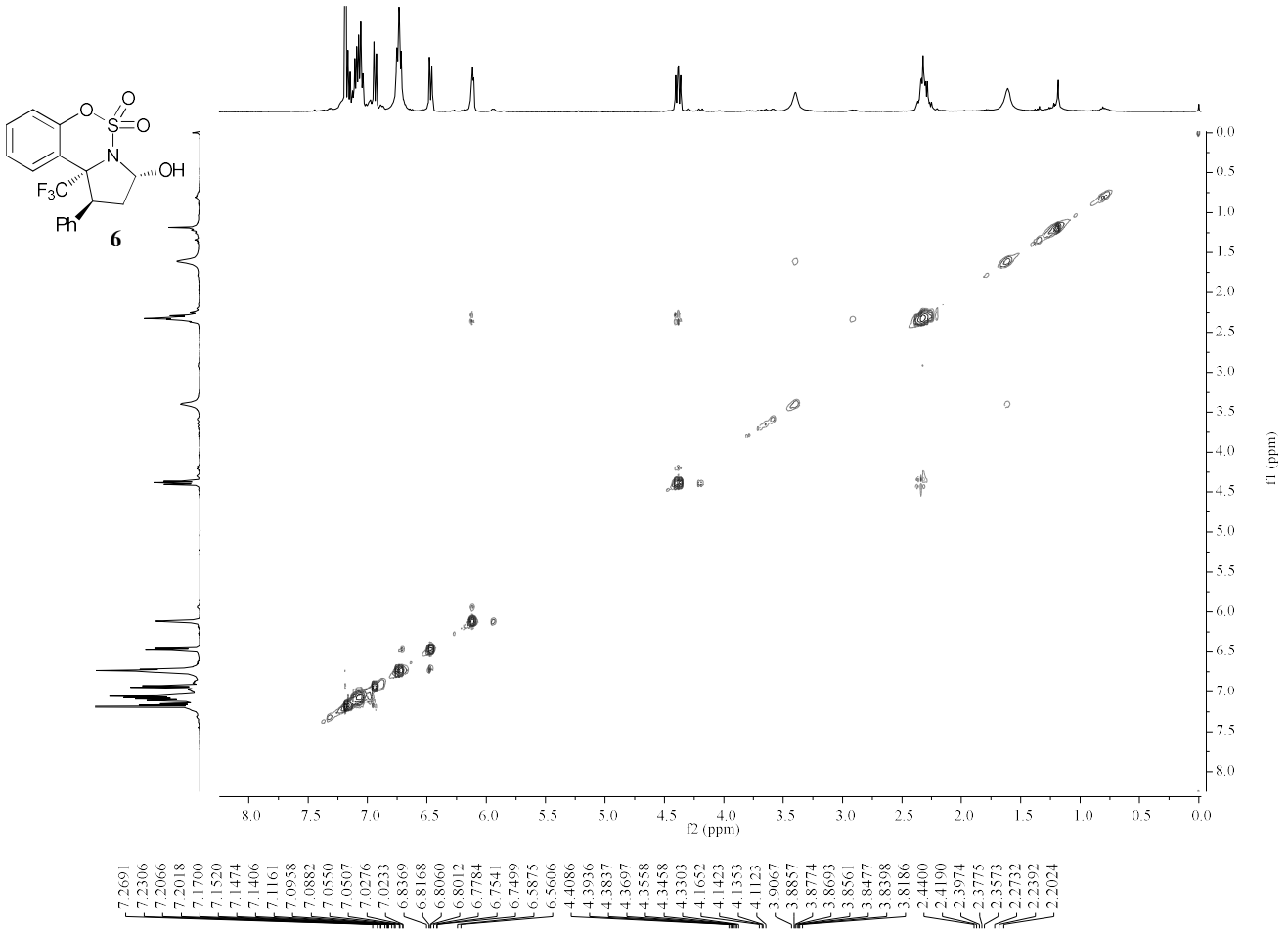

O
O
S
O
N
OH
F3C
Ph

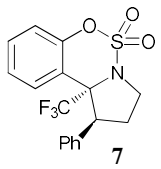
**6**

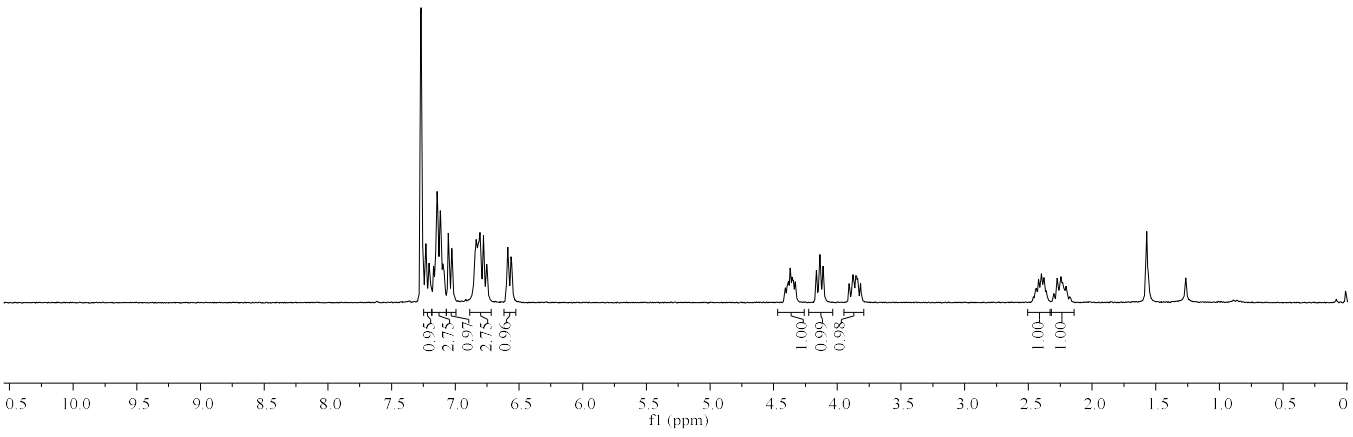

5.29
1.38
3.29
1.08
1.06
1.08
1.06
2.25
f1 (ppm)

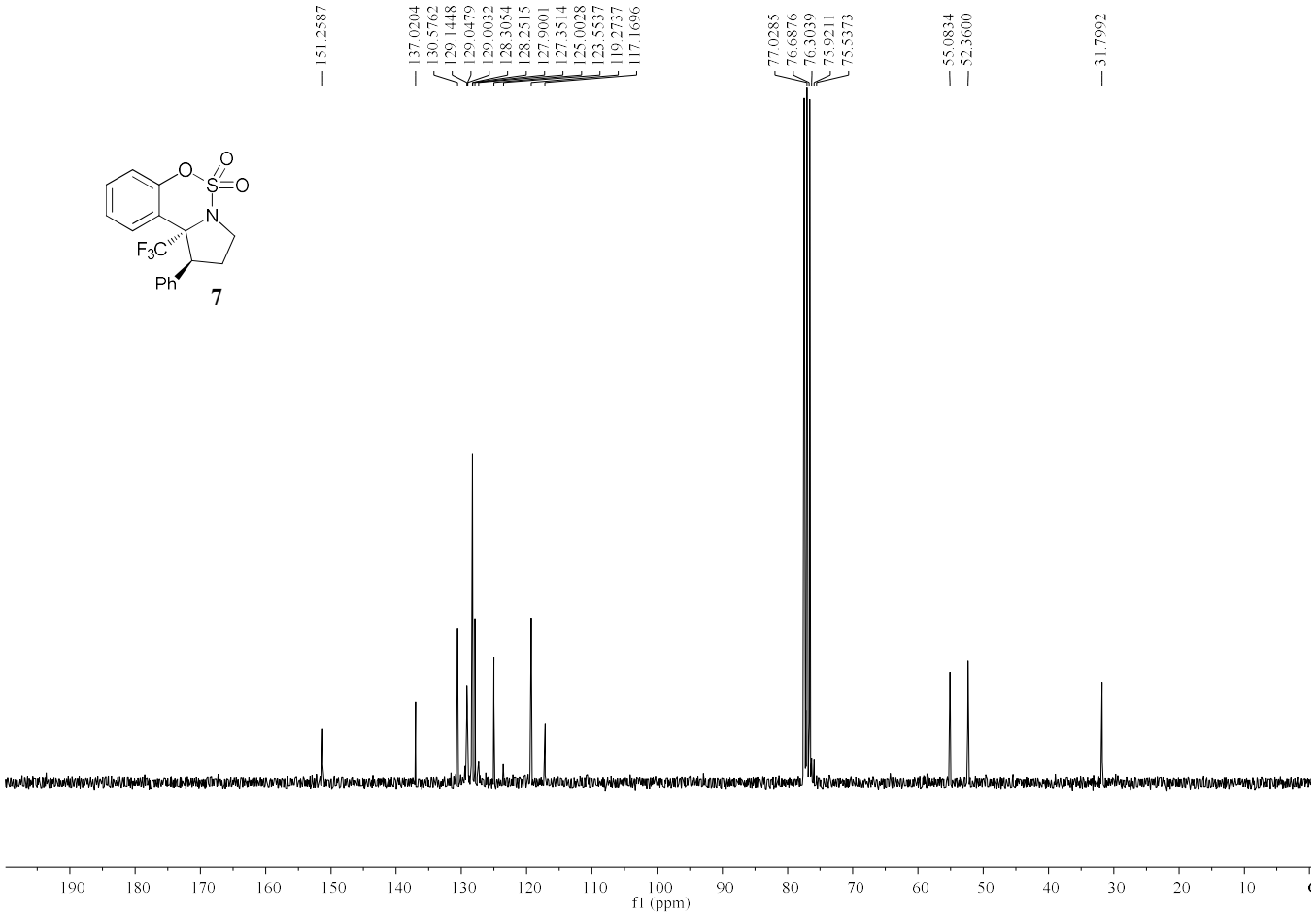

151.1156
136.0834
130.7554
130.5066
129.4532
129.2581
129.2129
128.2608
128.1048
126.7094
125.1093
122.9120
119.2897
119.1127
116.1098
88.3162
77.0002
76.8633
76.4668
76.0714
75.6712
52.0980
38.5412
O
O
S
O
N
OH
F3C
Ph
6
f1 (ppm)

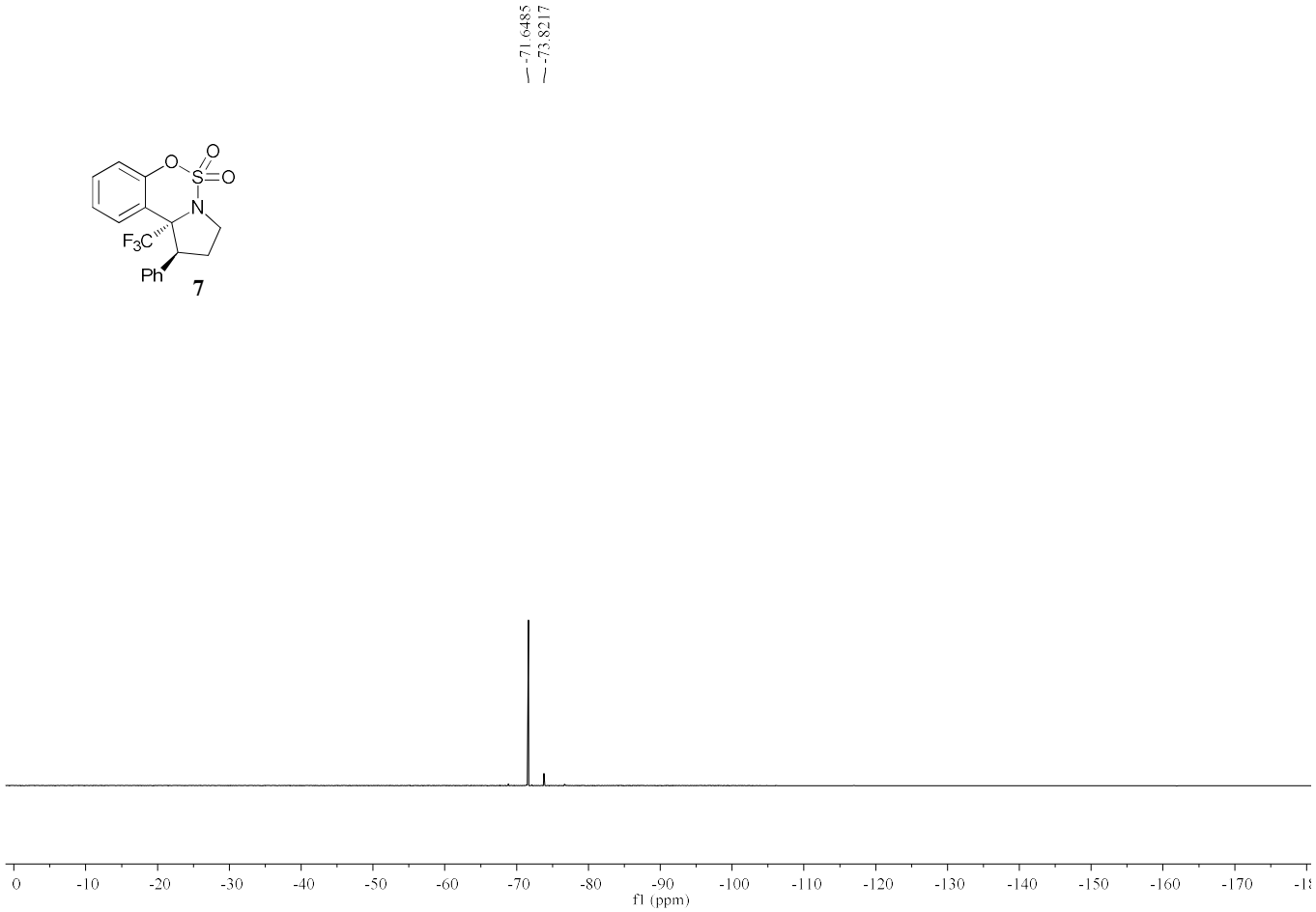

-71.6631
-73.8323
O
O
S
O
N
OH
F3C
Ph
6
f1 (ppm)


O
O
S
O
N
OH
F3C
Ph
6
f2 (ppm)
f1 (ppm)
7.2691
7.2306
7.2066
7.2018
7.1700
7.1520
7.1474
7.1406
7.1161
7.0958
7.0882
7.0550
7.0507
7.0276
7.0233
6.8369
6.8168
6.8060
6.8012
6.7784
6.7541
6.7499
6.5875
6.5606
4.4086
4.3936
4.3837
4.3697
4.3558
4.3458
4.3303
4.1652
4.1423
4.1353
4.1123
3.9067
3.8857
3.8774
3.8693
3.8561
3.8477
3.8398
3.8186
2.4400
2.4190
2.3974
2.3775
2.3573
2.2732
2.2392
2.2024


O
O
S
O
N
F3C
Ph
7

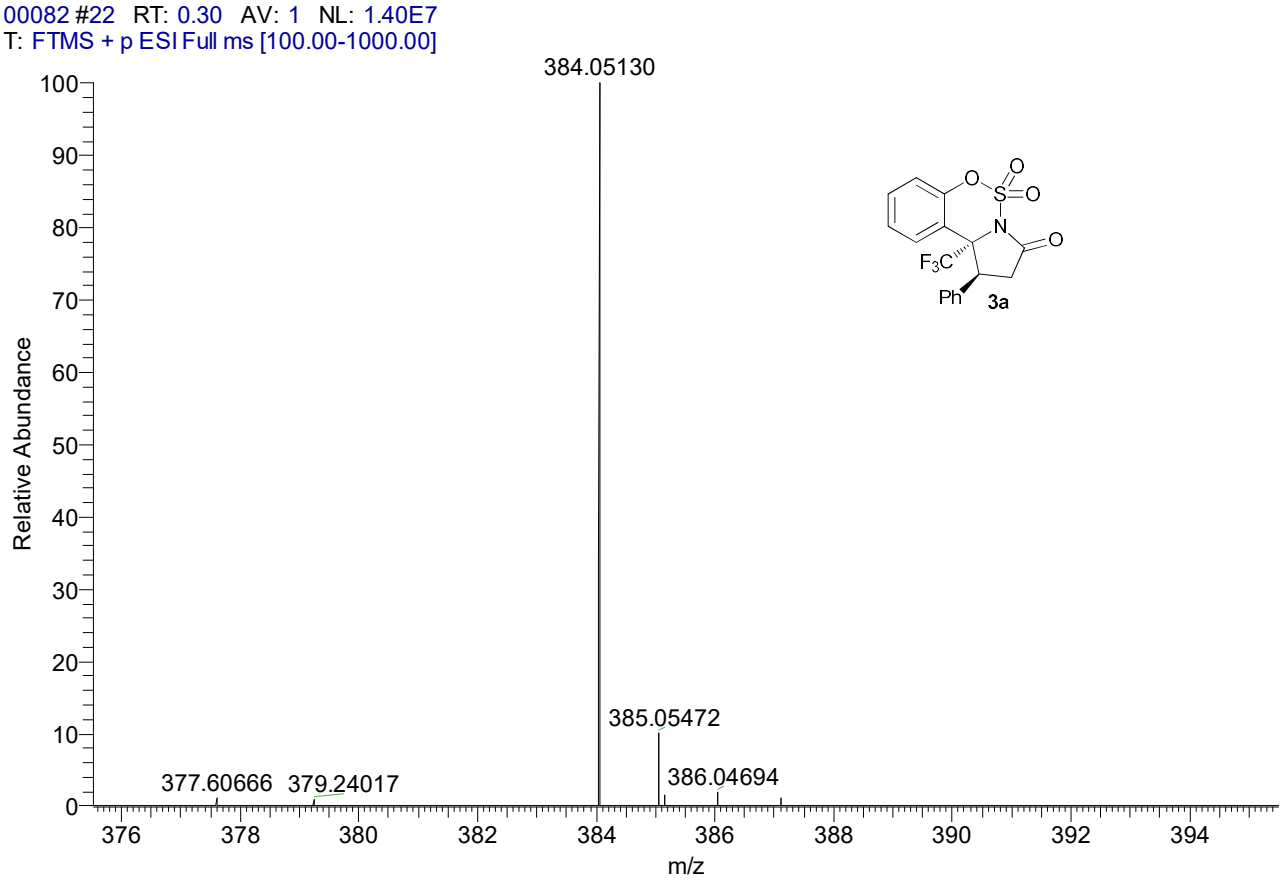

0.95
2.75
0.97
2.75
0.96
1.00
0.99
0.98
1.00
1.00
f1 (ppm)


151.2587
137.0204
130.5762
129.1448
129.0479
129.0032
128.3054
128.2515
127.9001
127.3514
125.0028
123.5537
119.2737
117.1696
77.0285
76.6876
76.3039
75.9211
75.5373
55.0834
52.3600
31.7992
O
O
S
O
N
F3C
Ph
7
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

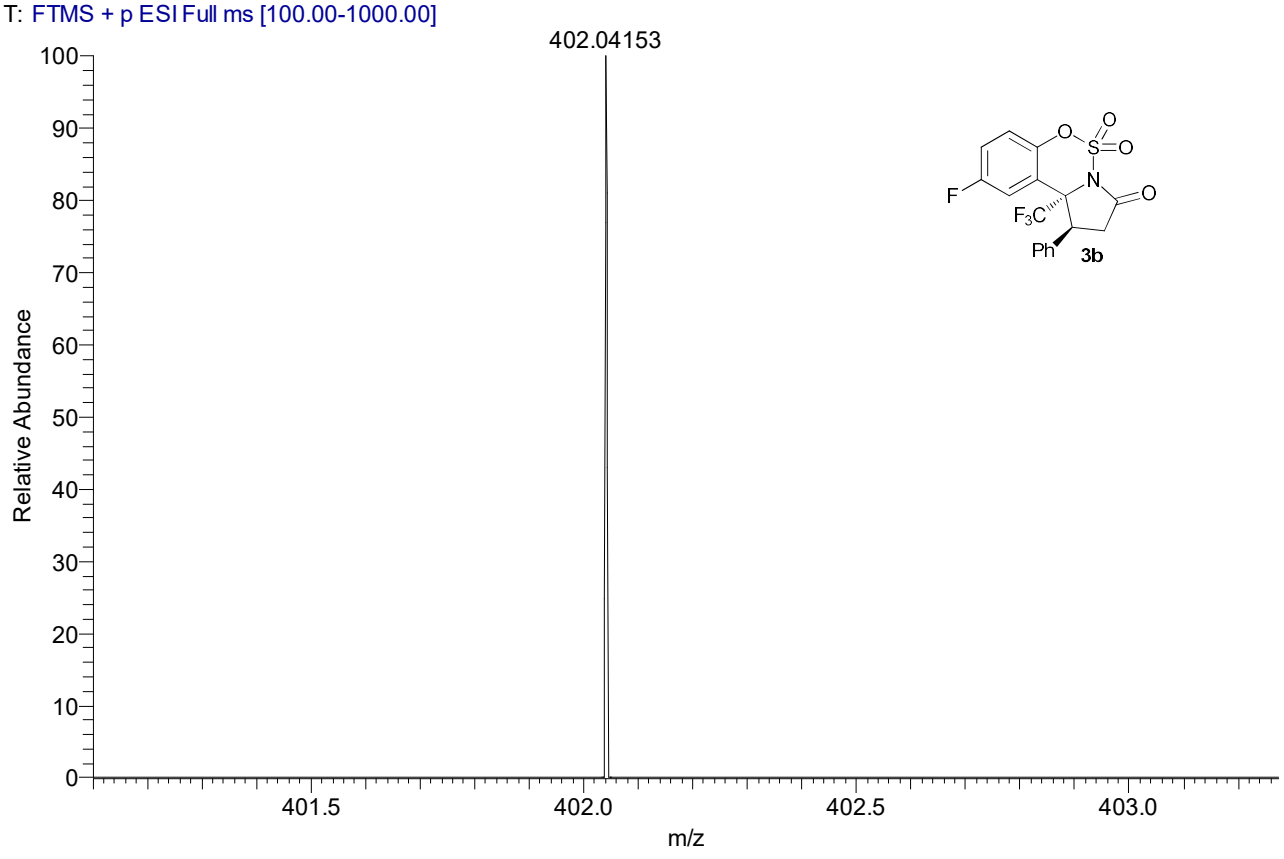

-71.6485
-73.8217
O
O
S
O
N
F3C
Ph
7
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
f1 (ppm)

**Copy of HRMS for New Compounds**

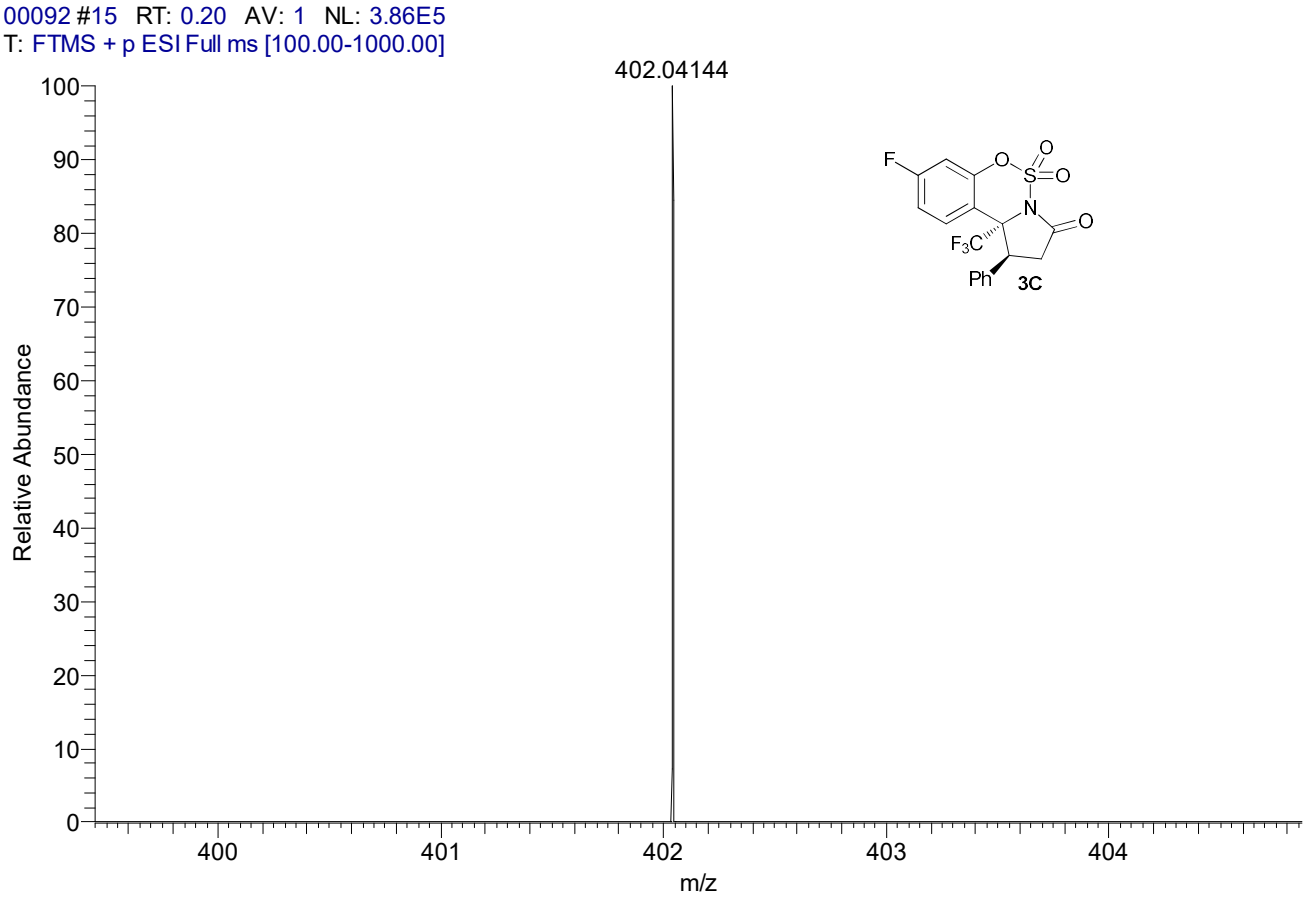
**ZZZ-3-1E.** HRMS (ESI) m/z calcd for $C_{17}H_{13}F_3NO_4S^+$ $(M+H)^+$ 384.05119, found 384.05130.



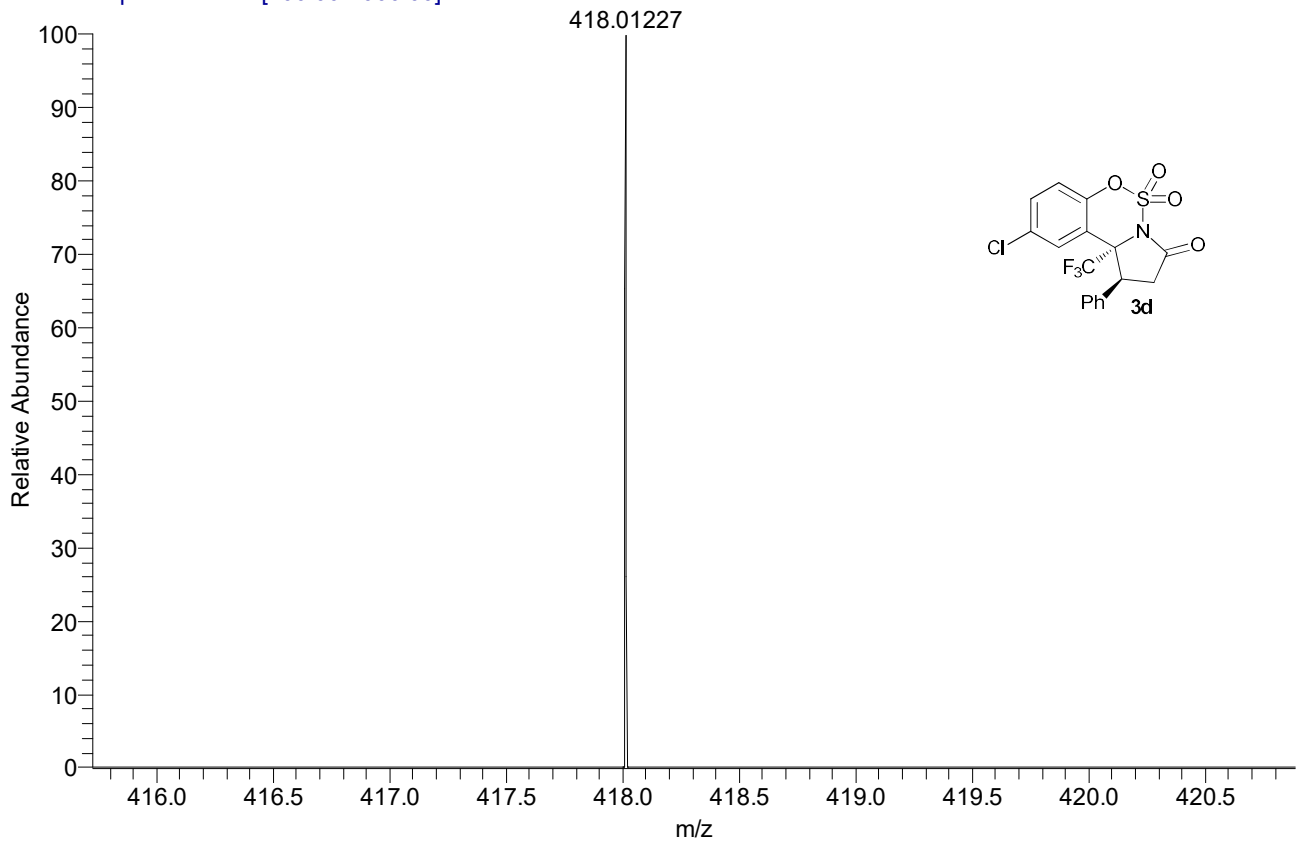

**ZZZ-3-22B.** HRMS (ESI) m/z calcd for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.04177, found 402.04153.



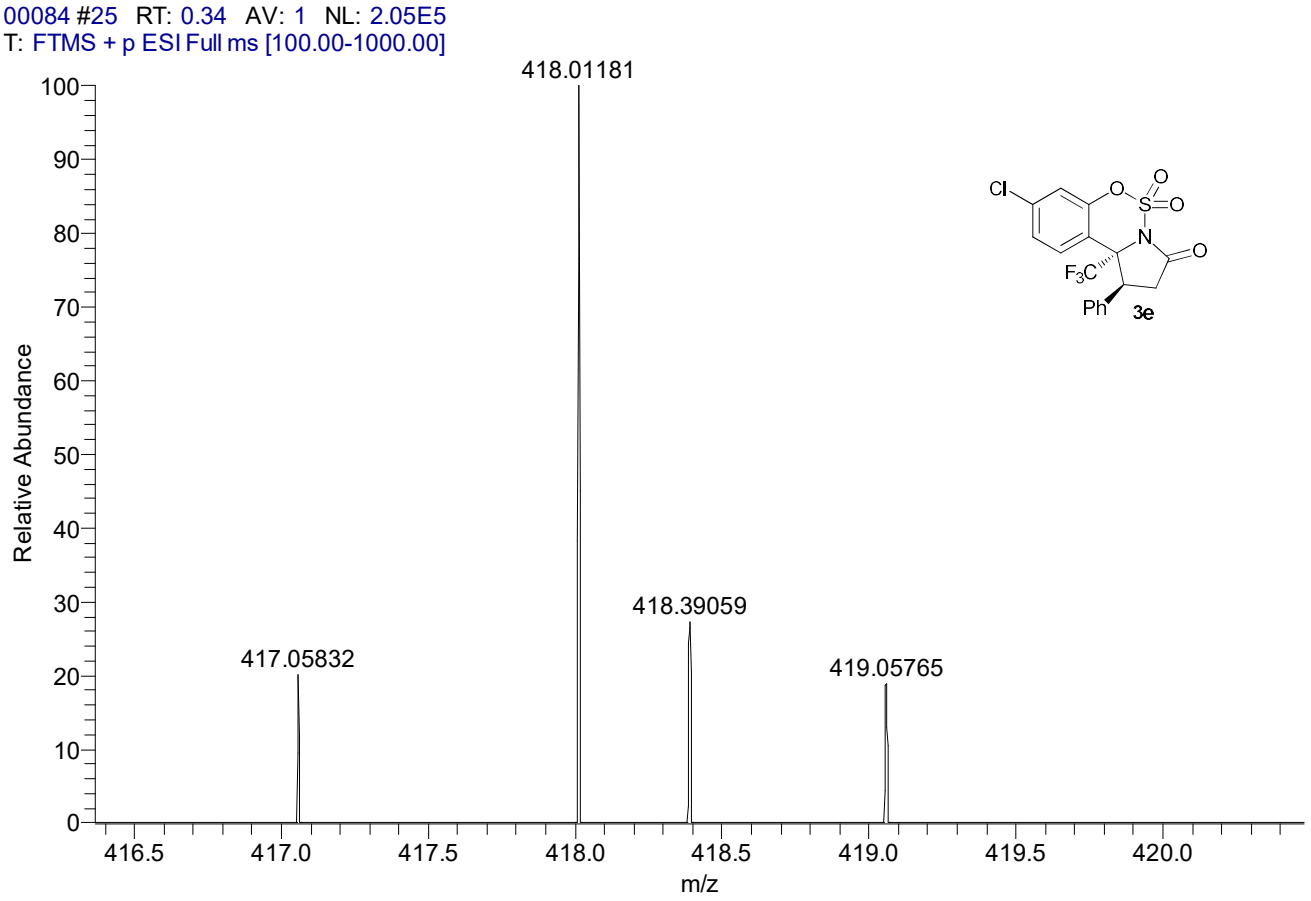
**ZZZ-3-26B.** HRMS (ESI) m/z calcd for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.04177, found 402.04144.



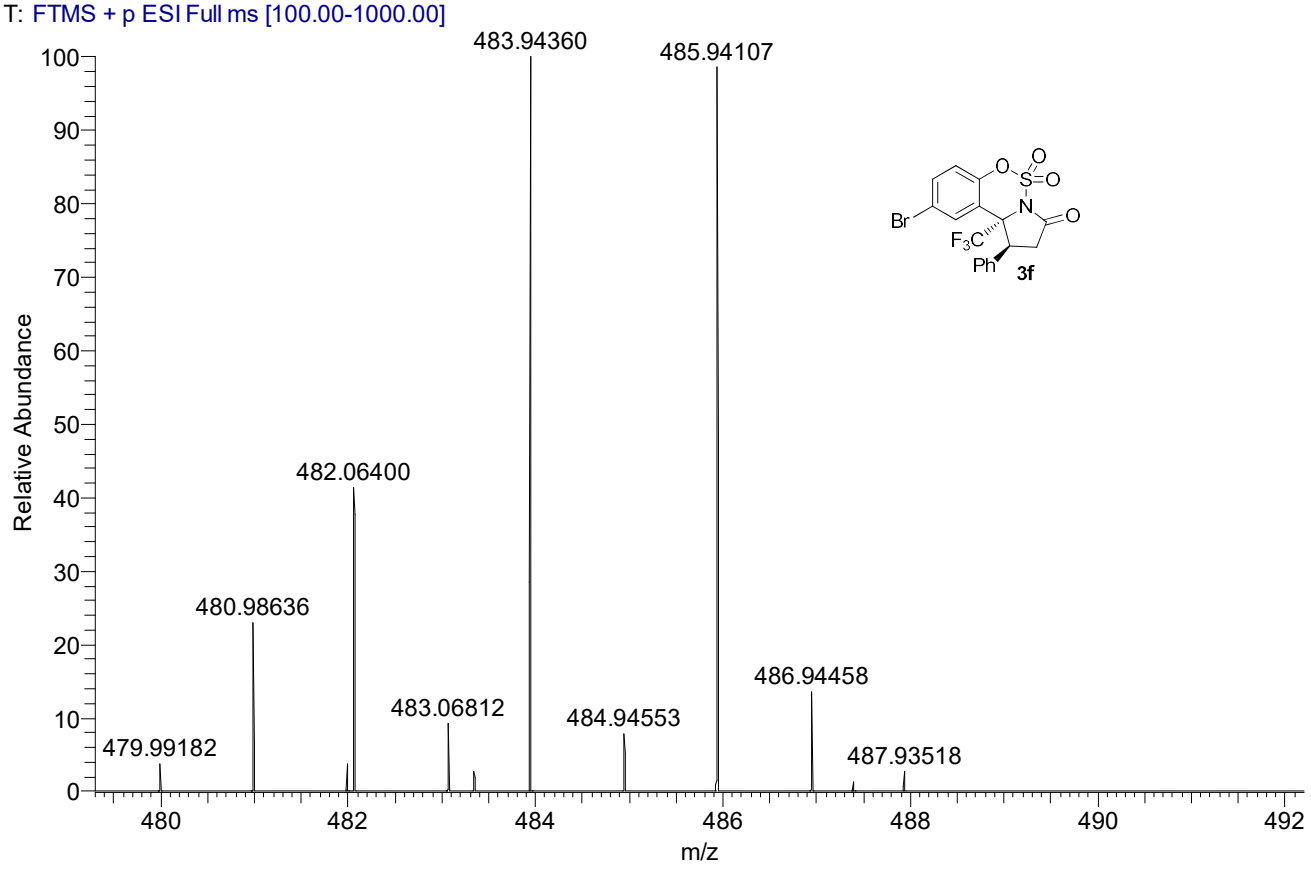

**ZZZ-3-15A.** HRMS (ESI) m/z calcd for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.01222, found 418.01227.



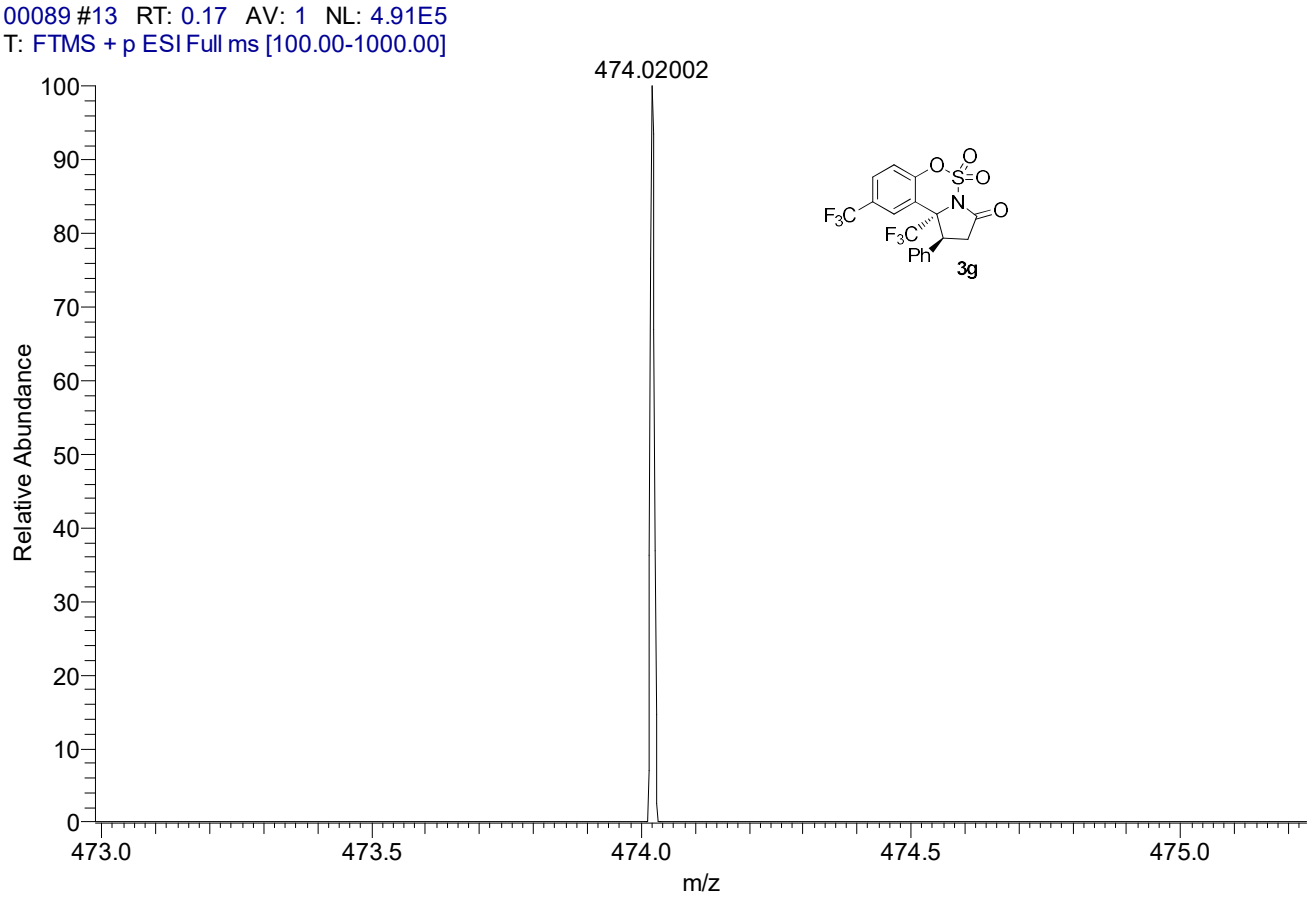
**ZZZ-3-15B.** HRMS (ESI) m/z calcd for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.01222, found 418.01187.



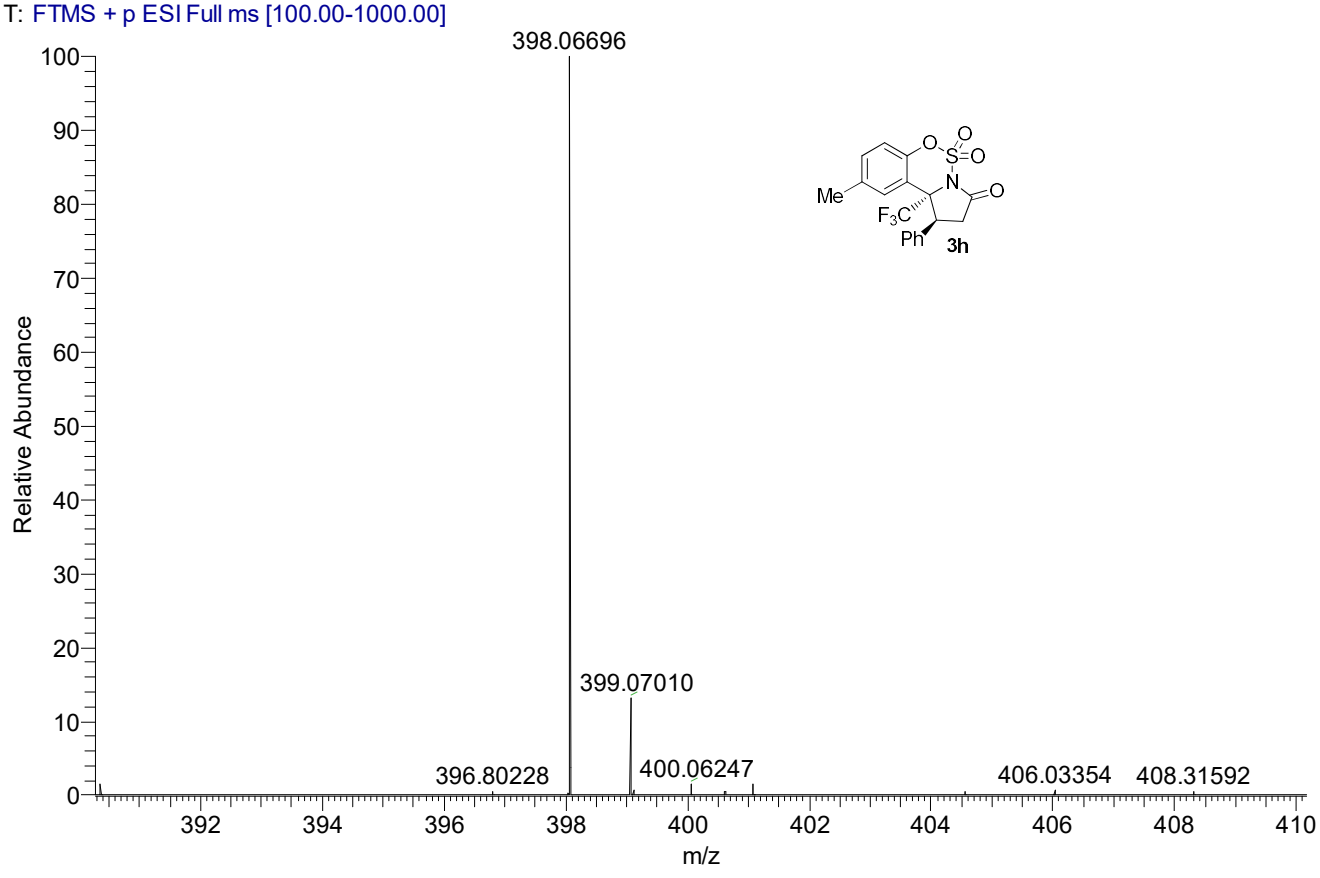

**ZZZ-3-26A.** HRMS (ESI) m/z calcd for $C_{17}H_{11}BrF_3NO_4SNa^+$ $(M+Na)^+$ 483.94365, found 483.94360.



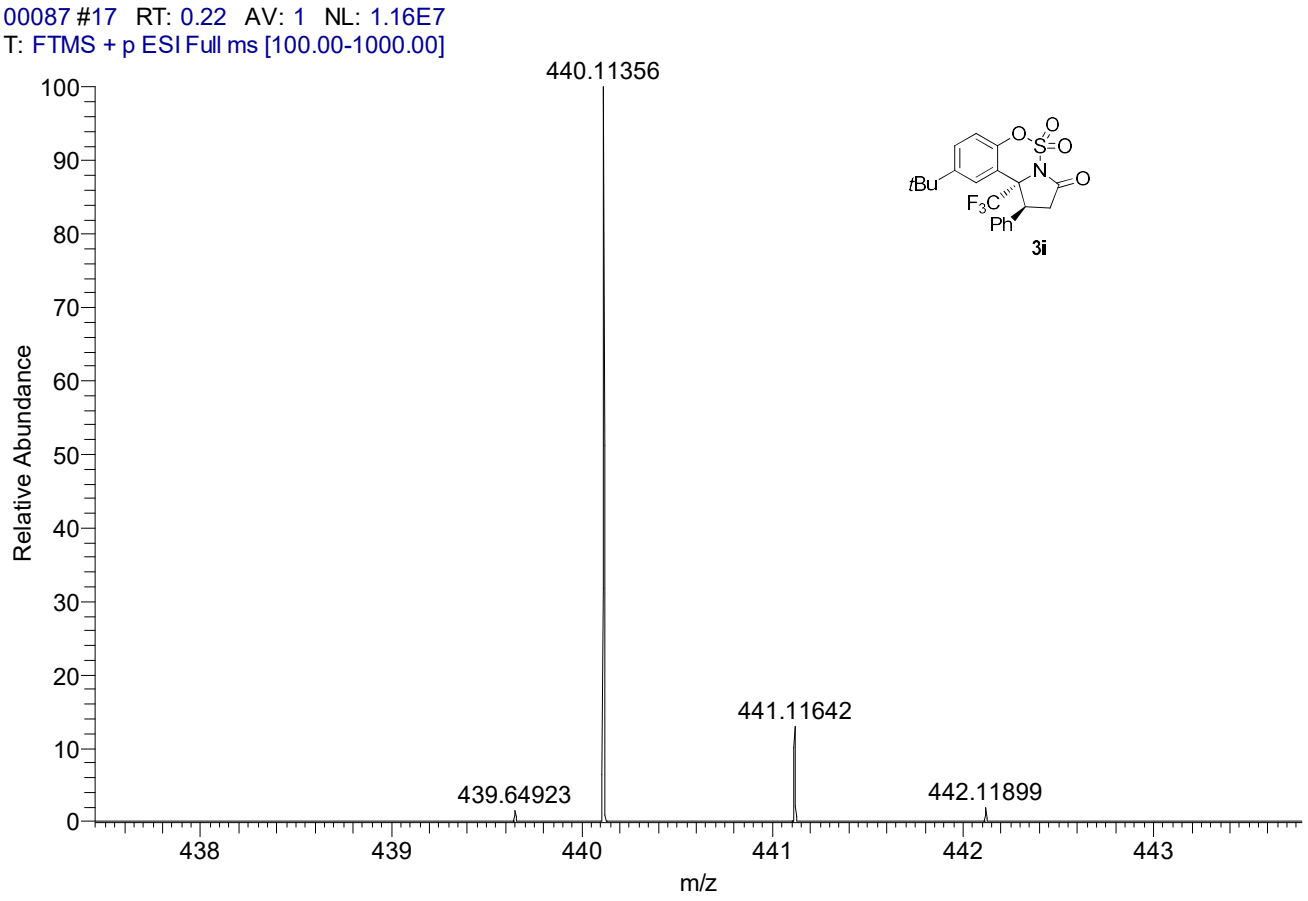
**ZZZ-3-18D.** HRMS (ESI) m/z calcd for $C_{18}H_{11}F_6NO_4SNa^+$ $(M+Na)^+$ 474.02052, found 474.02002.



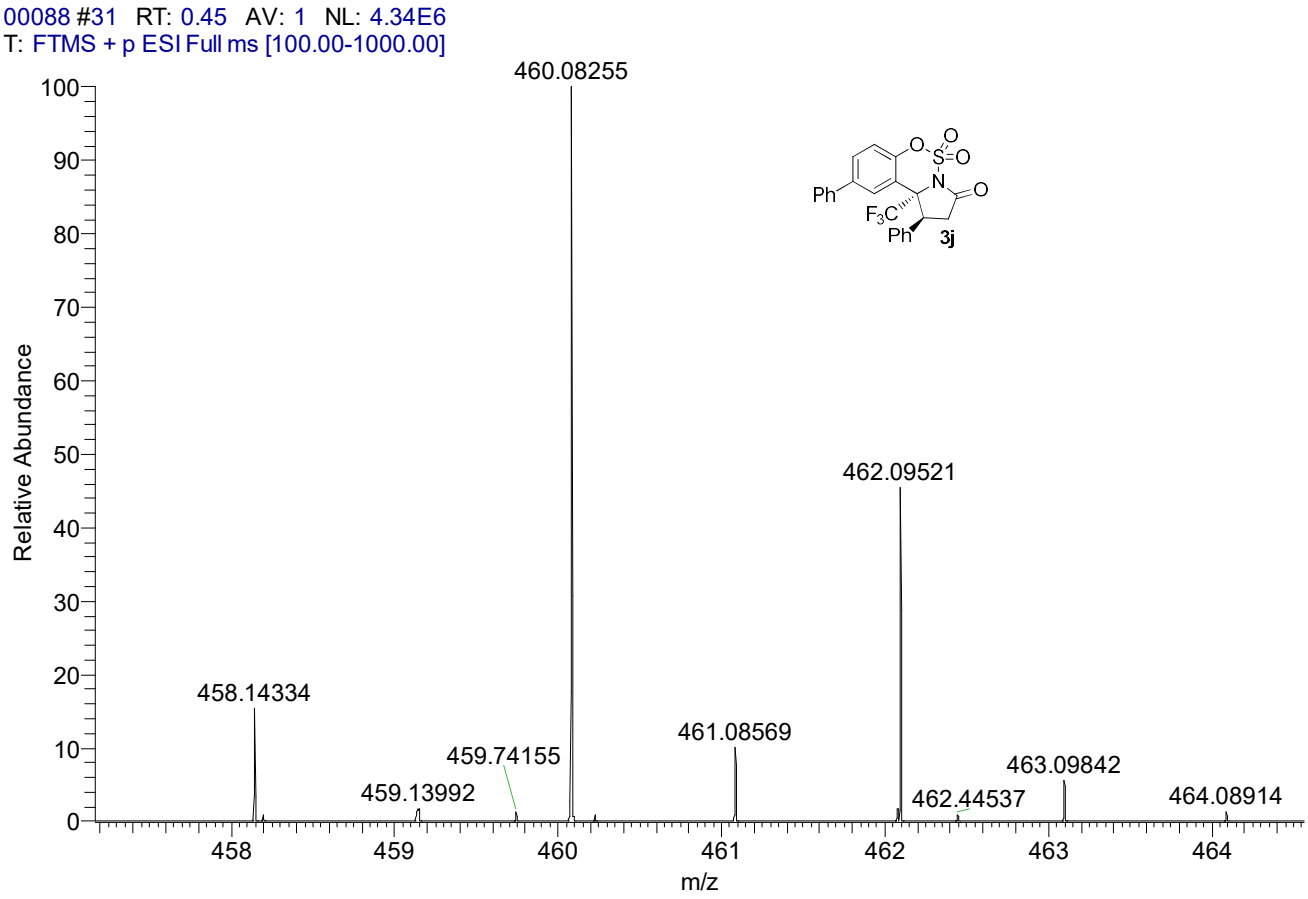

**ZZZ-3-15C.** HRMS (ESI) m/z calcd for $C_{18}H_{15}F_3NO_4S^+$ $(M+H)^+$ 398.06684, found 398.06696.



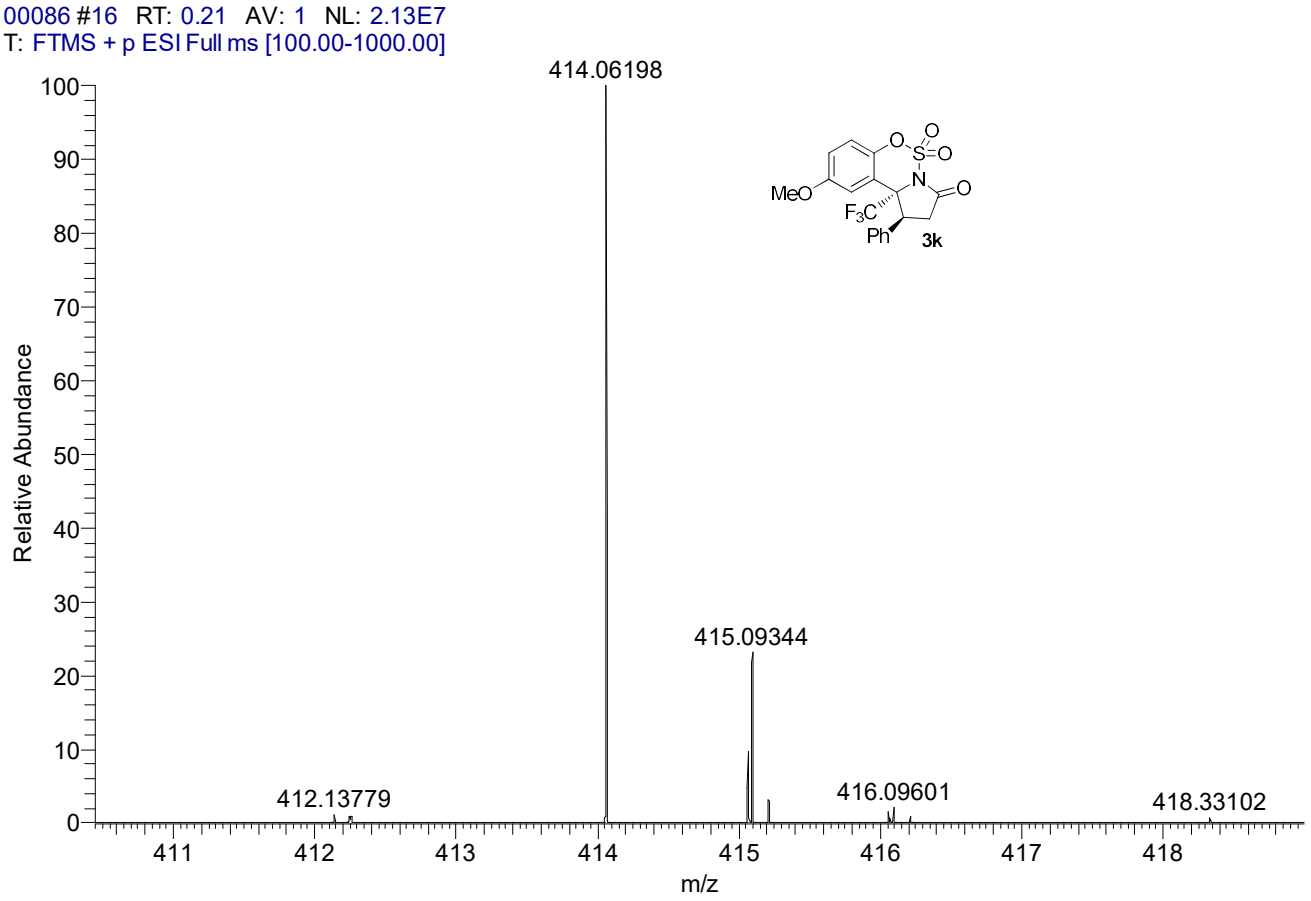
**ZZZ-3-18B.** HRMS (ESI) m/z calcd for $C_{21}H_{21}F_3NO_4S^+$ $(M+H)^+$ 440.11379, found 440.11356.



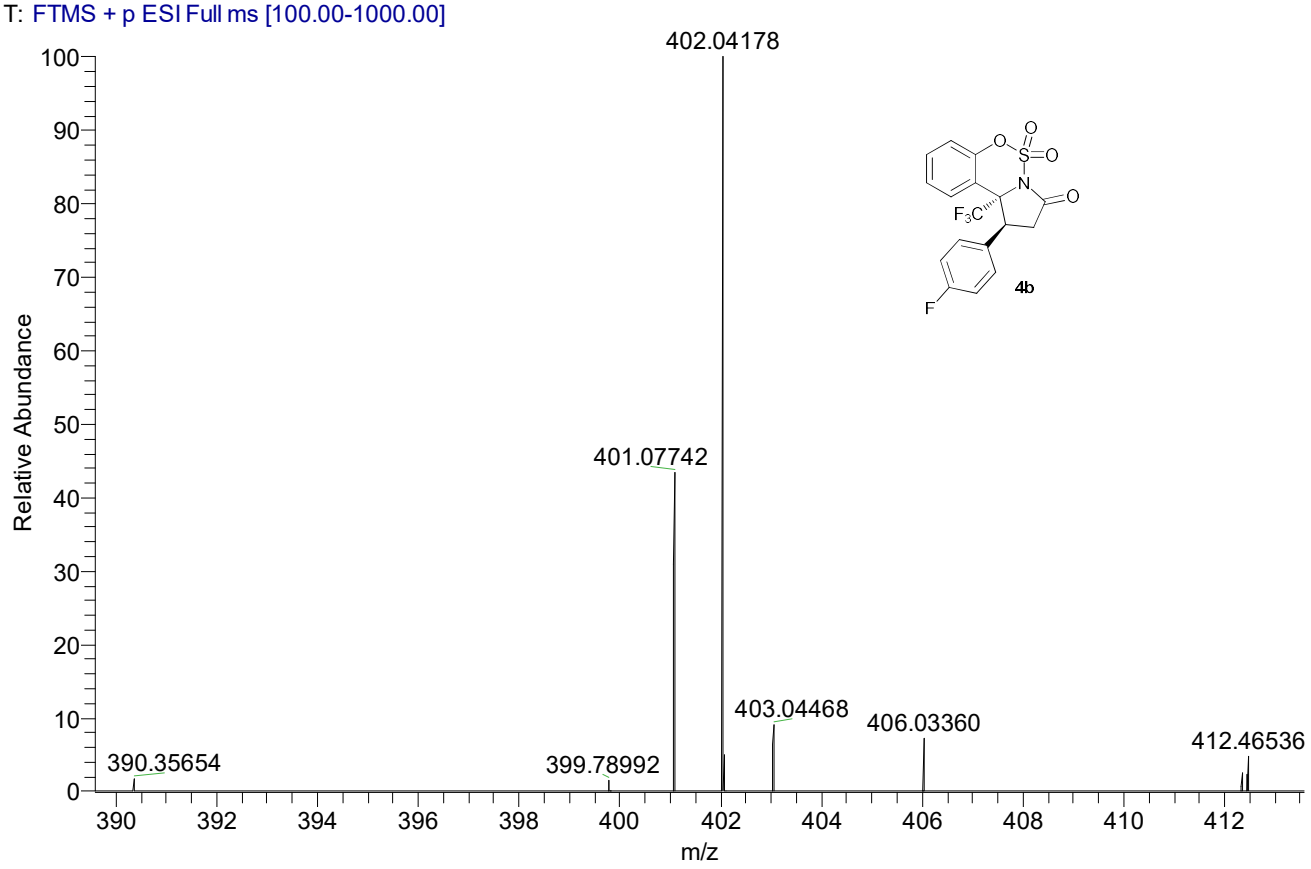

**ZZZ-3-18C.** HRMS (ESI) m/z calcd for $C_{23}H_{17}F_3NO_4S^+$ $(M+H)^+$ 460.08249, found 460.08255.



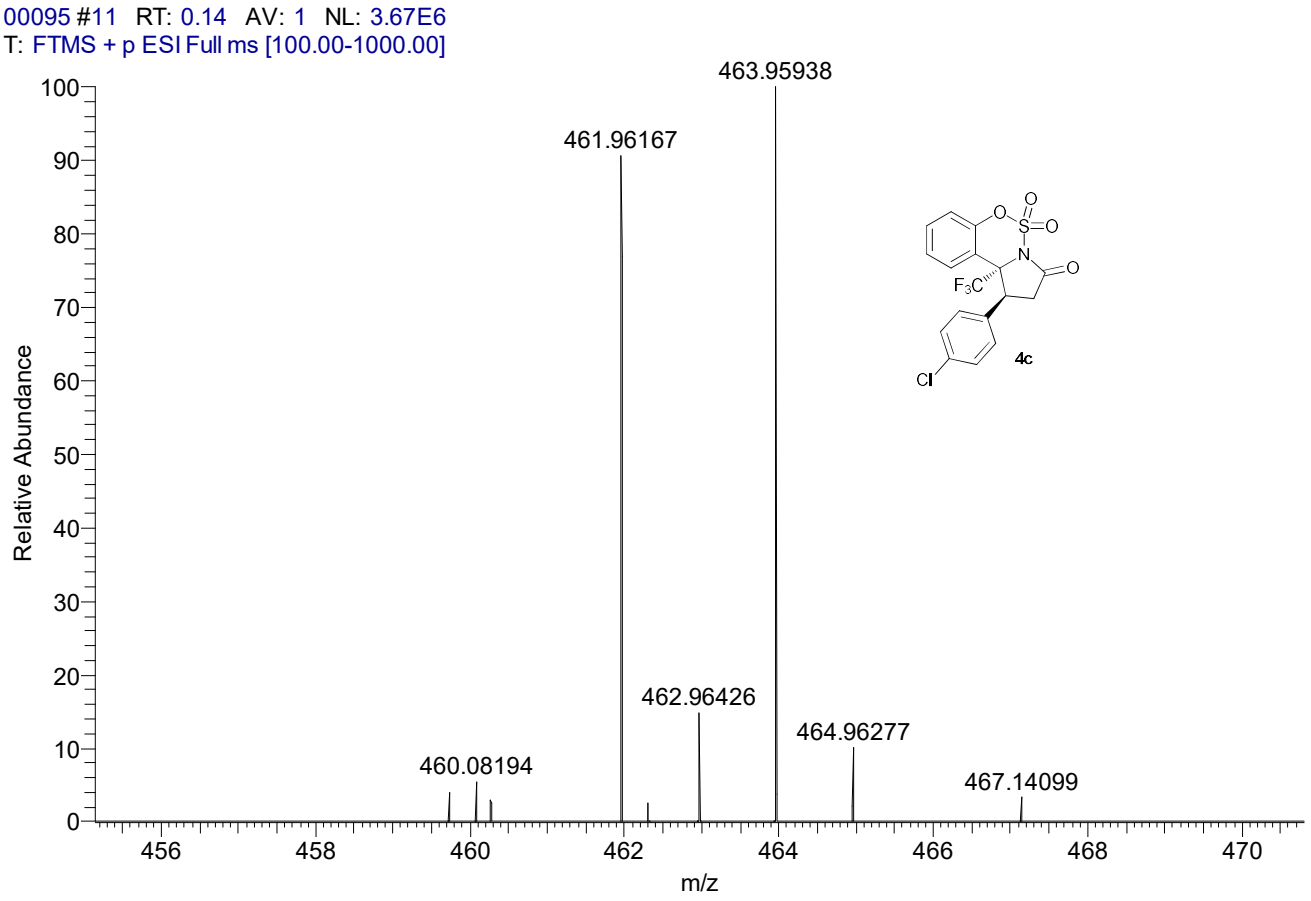
**ZZZ-3-15D.** HRMS (ESI) m/z calcd for $C_{18}H_{15}F_3NO_5S^+$ $(M+H)^+$ 414.06175, found 414.06198.



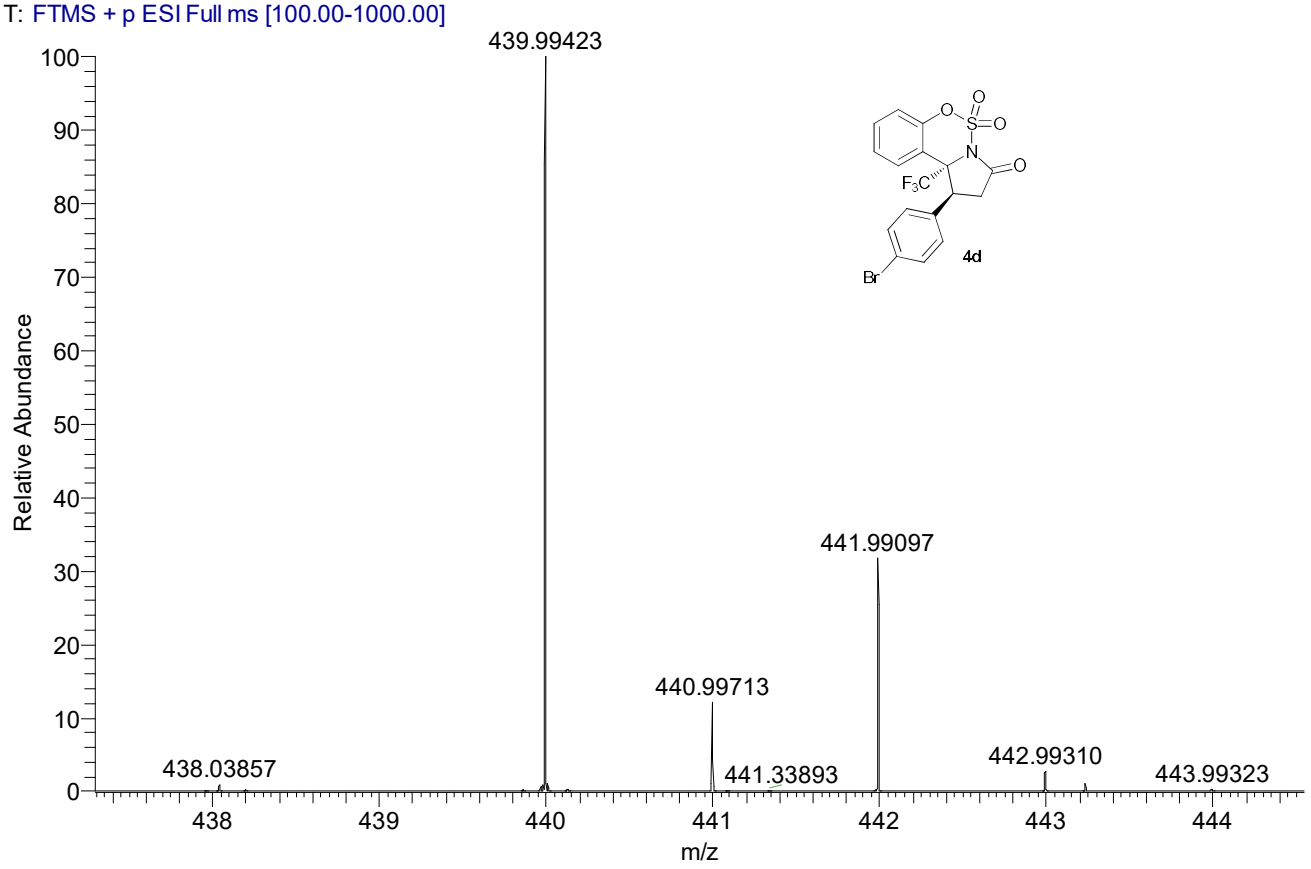

**ZZZ-2-96A.** HRMS (ESI) m/z calcd for $C_{17}H_{12}F_4NO_4S^+$ $(M+H)^+$ 402.04177, found 402.04178.



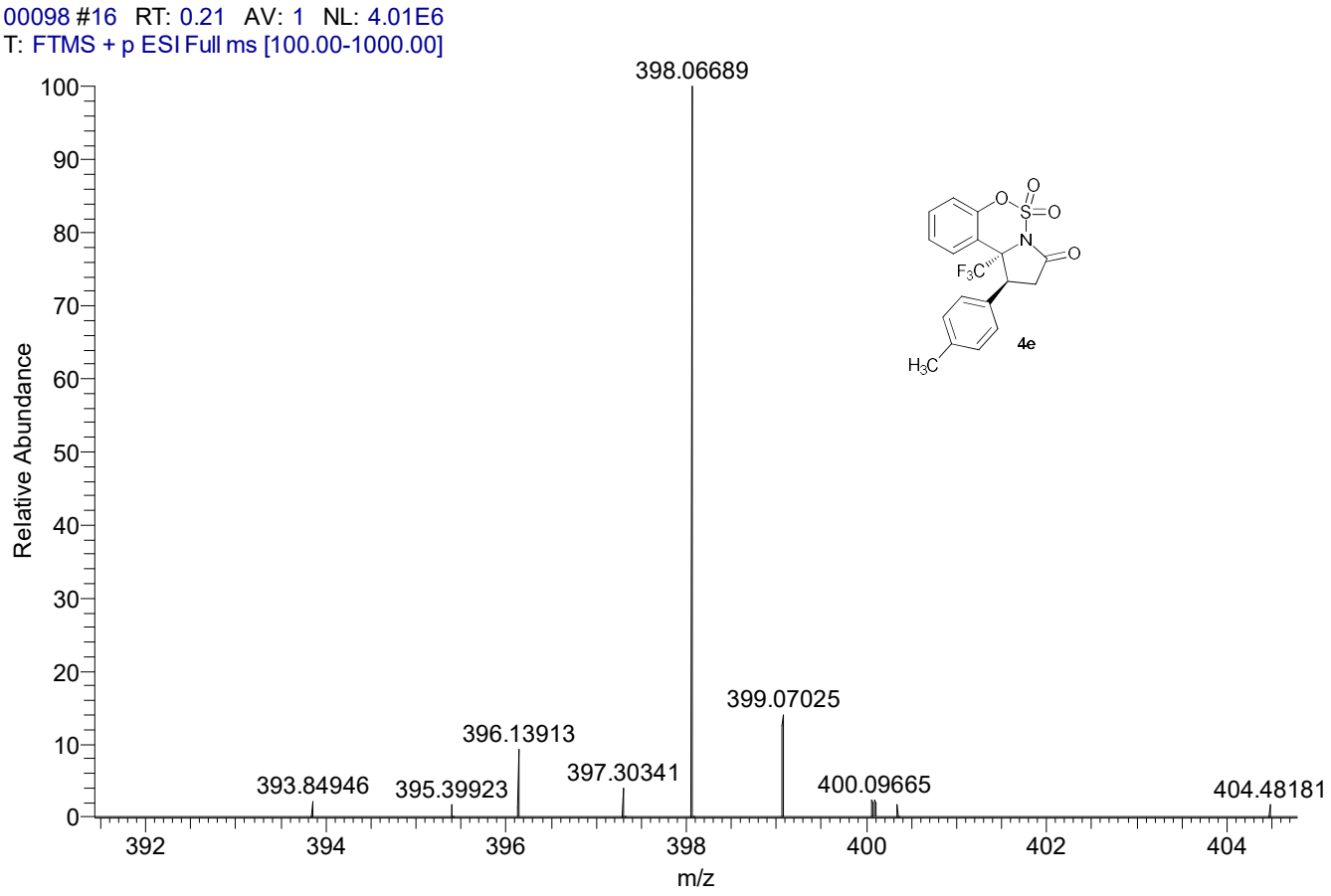
**ZZZ-2-96C.** HRMS (ESI) m/z calcd for $C_{17}H_{12}BrF_3NO_4S^+$ $(M+H)^+$ 461.96170, found 461.96167.



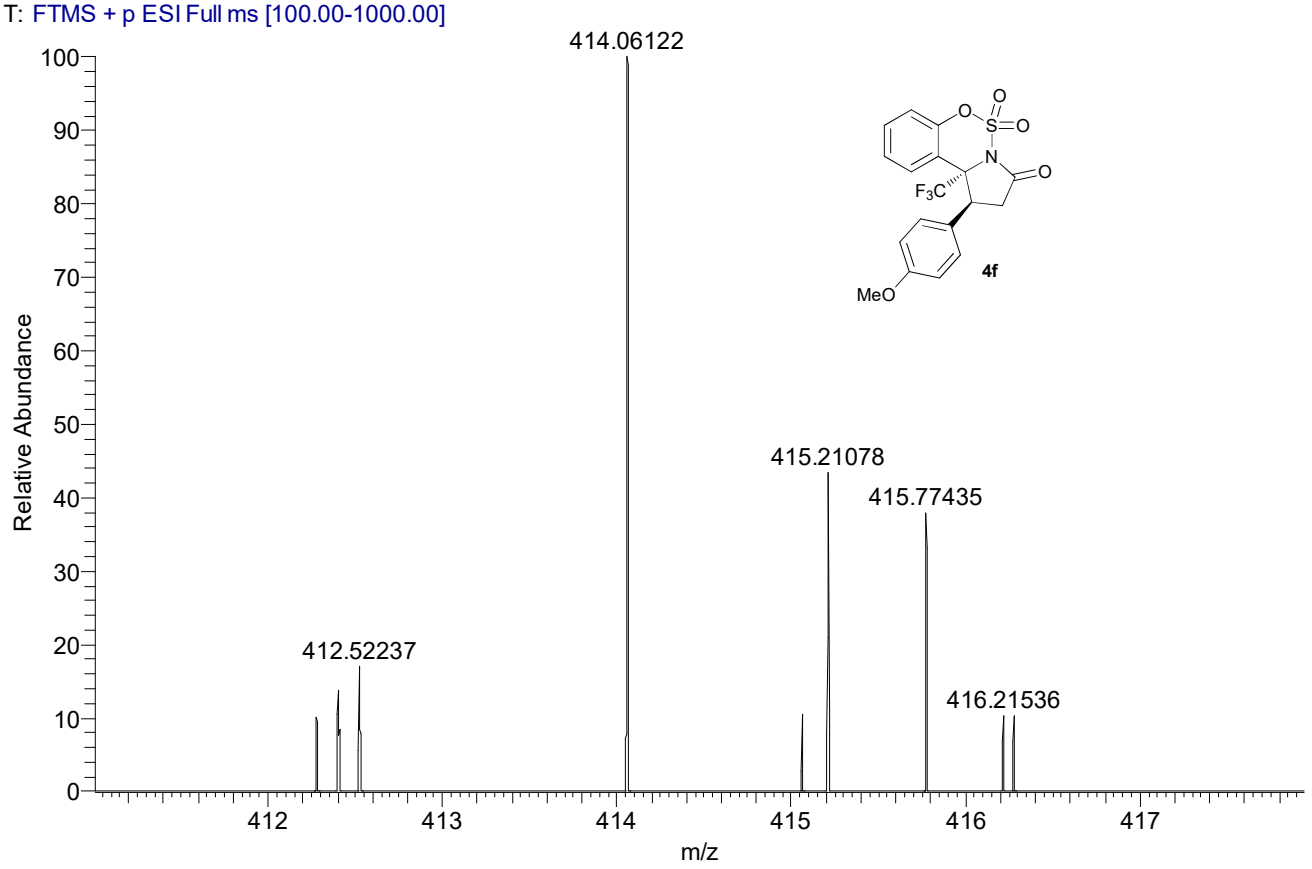

**ZZZ-2-96B.** HRMS (ESI) m/z calcd for $C_{17}H_{11}ClF_3NO_4SNa^+$ $(M+Na)^+$ 439.99416, found 439.99423.



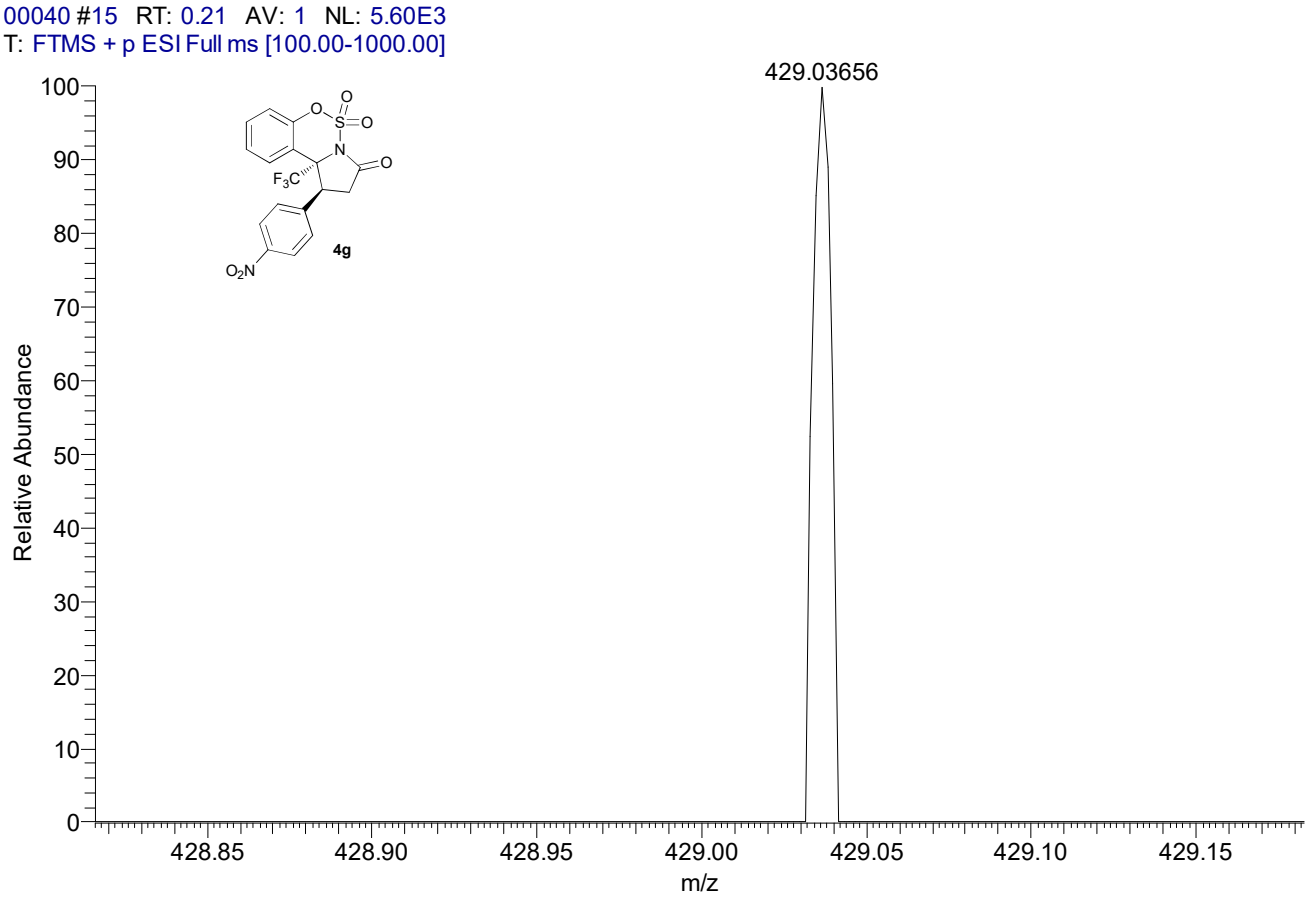
**ZZZ-3-22A.** HRMS (ESI) m/z calcd for $C_{18}H_{15}F_3NO_4S^+$ $(M+H)^+$ 398.06684, found 398.06689.



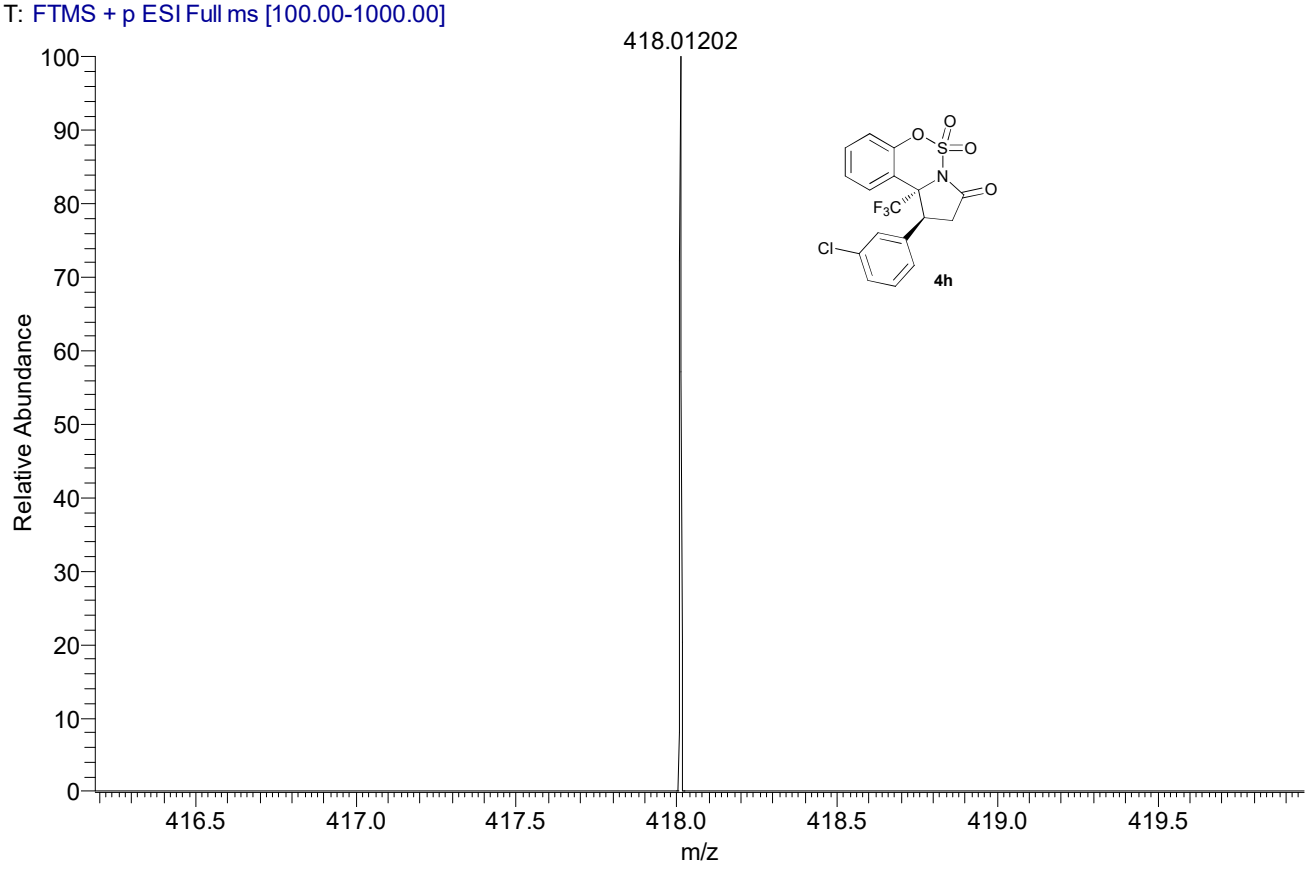

**ZZZ-3-1D.** HRMS (ESI) m/z calcd for $C_{18}H_{15}F_3NO_5S^+$ $(M+H)^+$ 414.06175, found 414.06122.



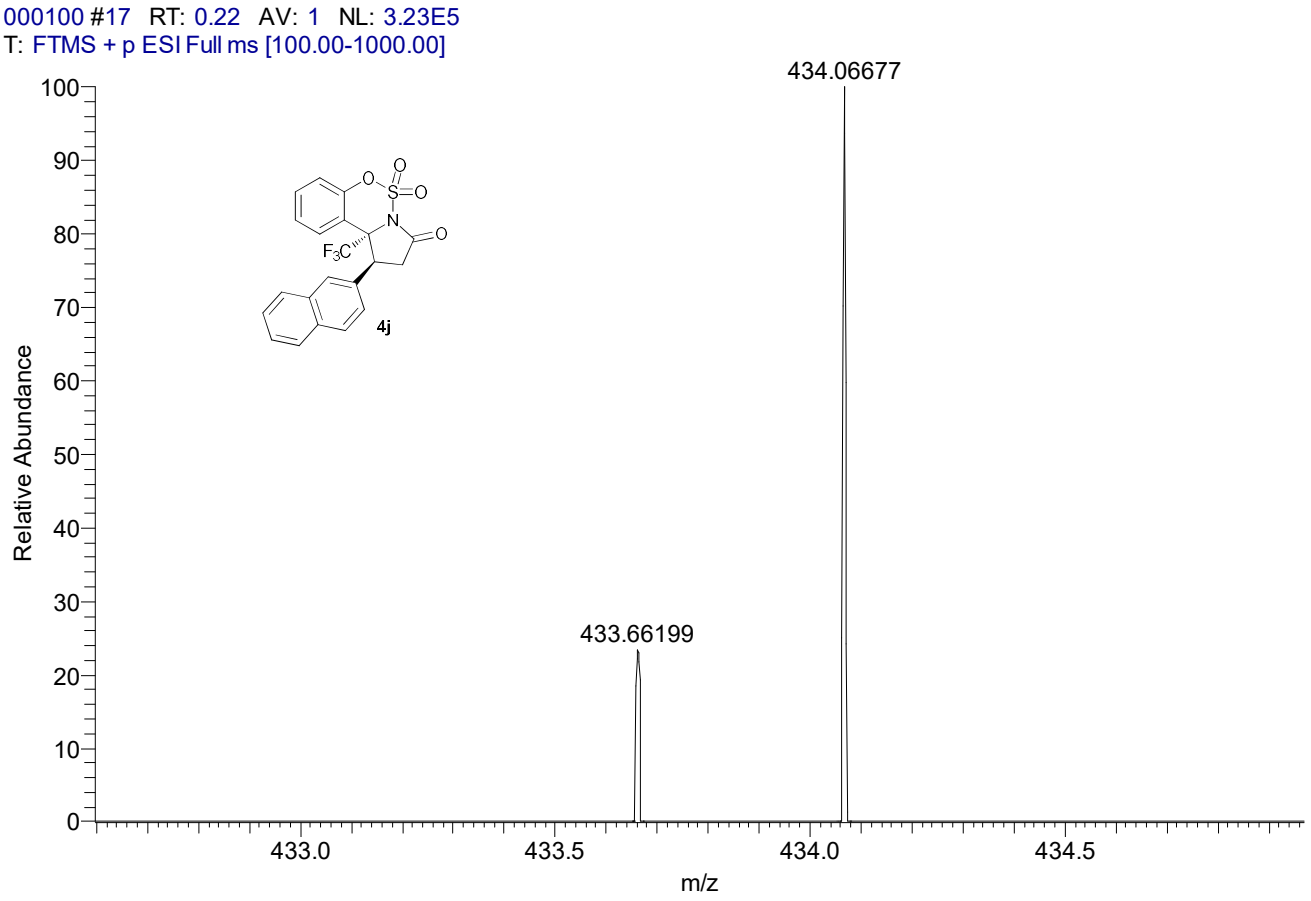
**ZZZ-3-1C.** HRMS (ESI) m/z calcd for $C_{17}H_{12}F_3N_2O_6S^+$ $(M+H)^+$ 429.03627, found 429.03656.



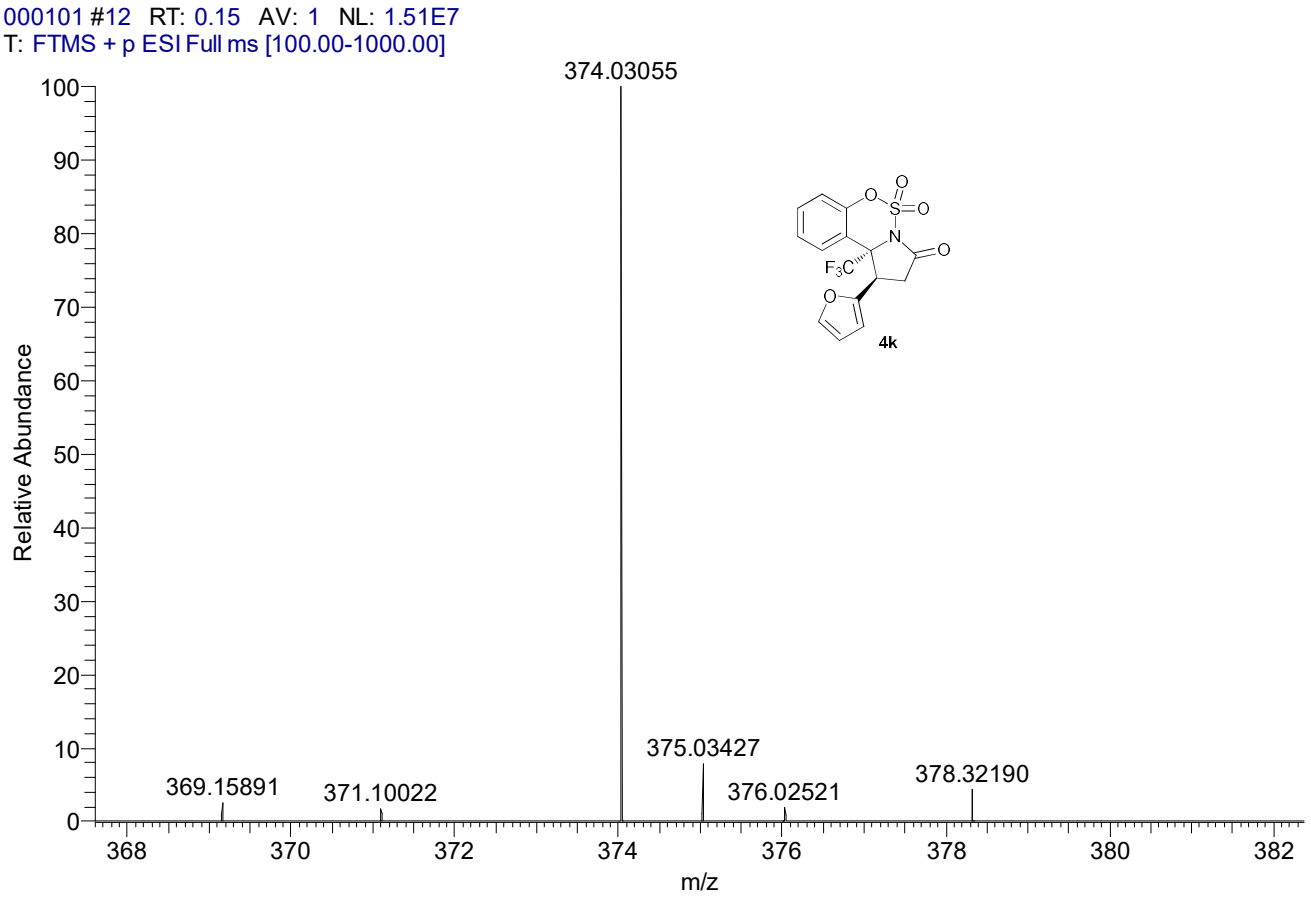

**ZZZ-3-9B.** HRMS (ESI) m/z calcd for $C_{17}H_{12}ClF_3NO_4S^+$ $(M+H)^+$ 418.01222, found 418.01202.



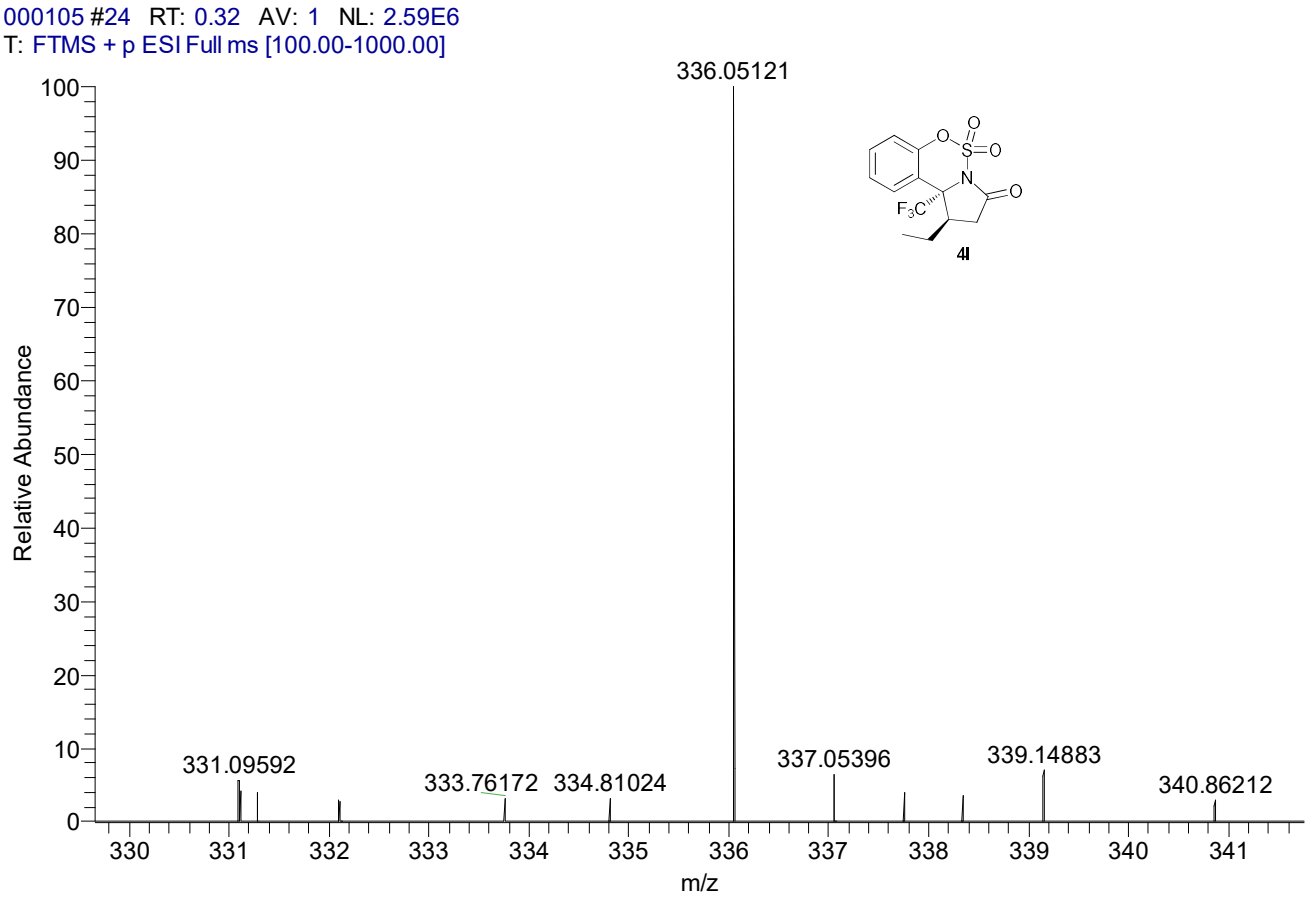
**ZZZ-3-9C.** HRMS (ESI) m/z calcd for $C_{21}H_{15}F_3NO_4S^+$ $(M+H)^+$ 434.06684, found 434.06677.



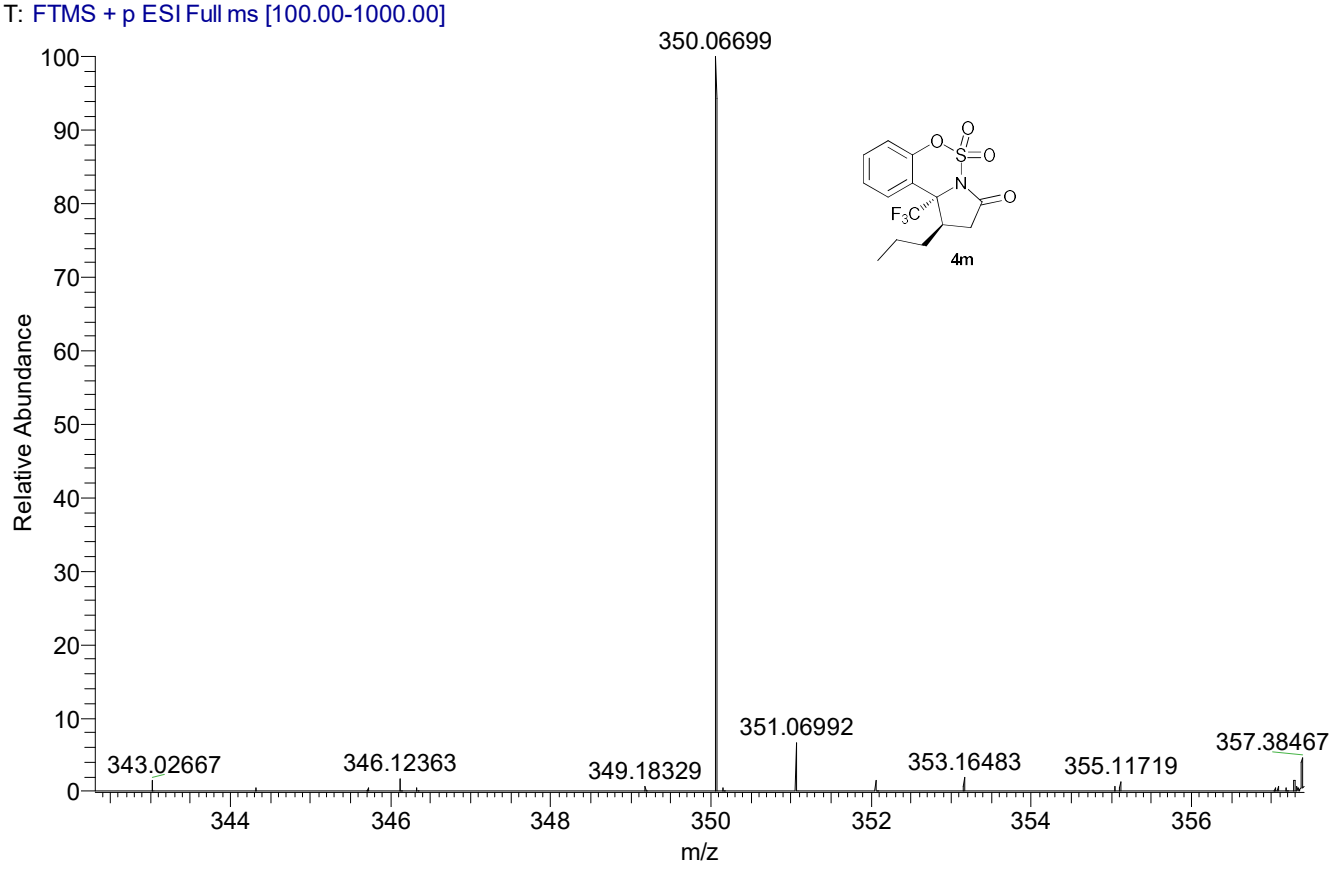

**ZZZ-3-1A.** HRMS (ESI) m/z calcd for $C_{15}H_{11}F_3NO_5S^+$ $(M+H)^+$ 374.03045, found 374.03055.



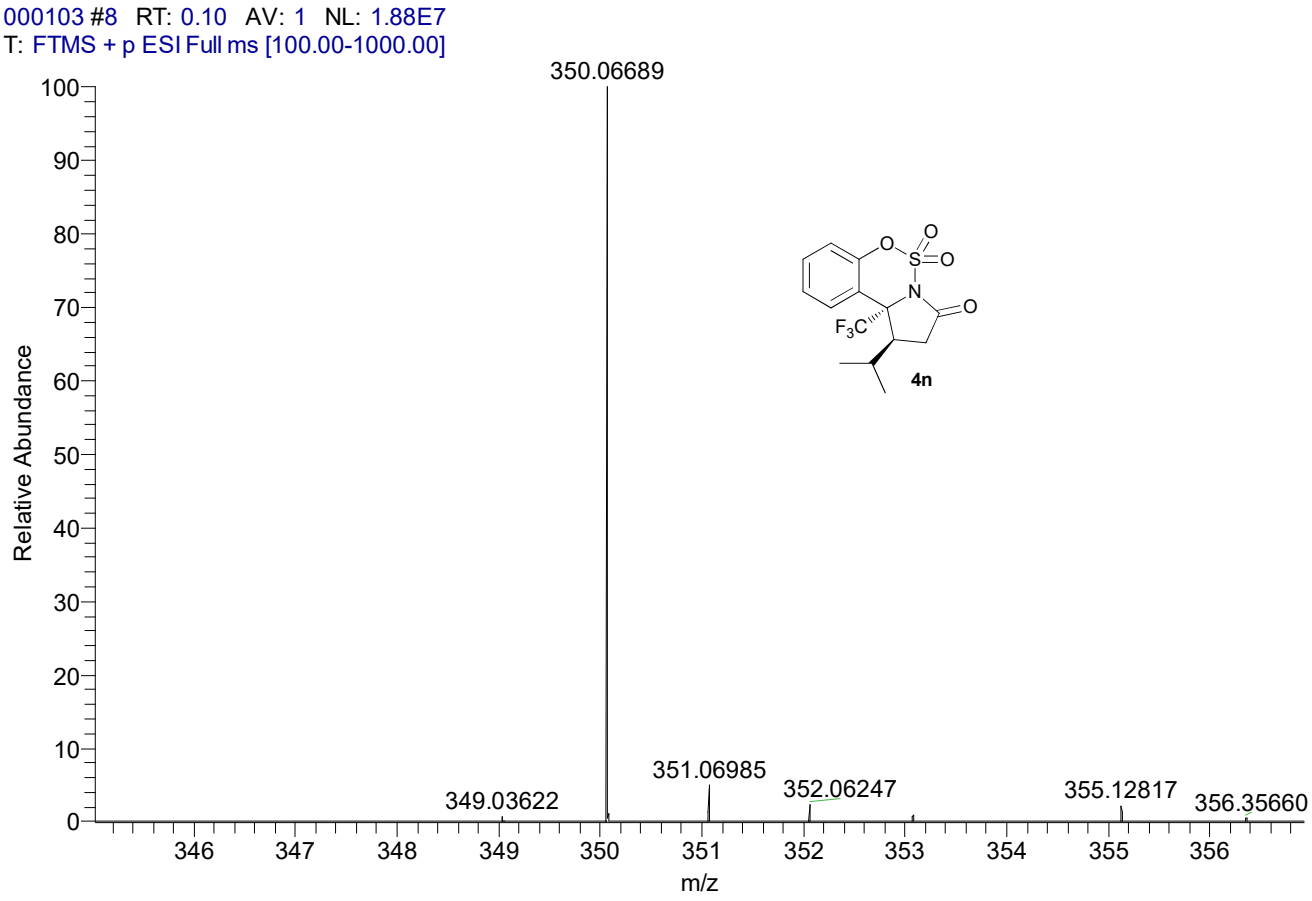
**ZZZ-3-7D.** HRMS (ESI) m/z calcd for $C_{13}H_{13}F_3NO_4S^+$ $(M+H)^+$ 336.05119, found 336.05121.



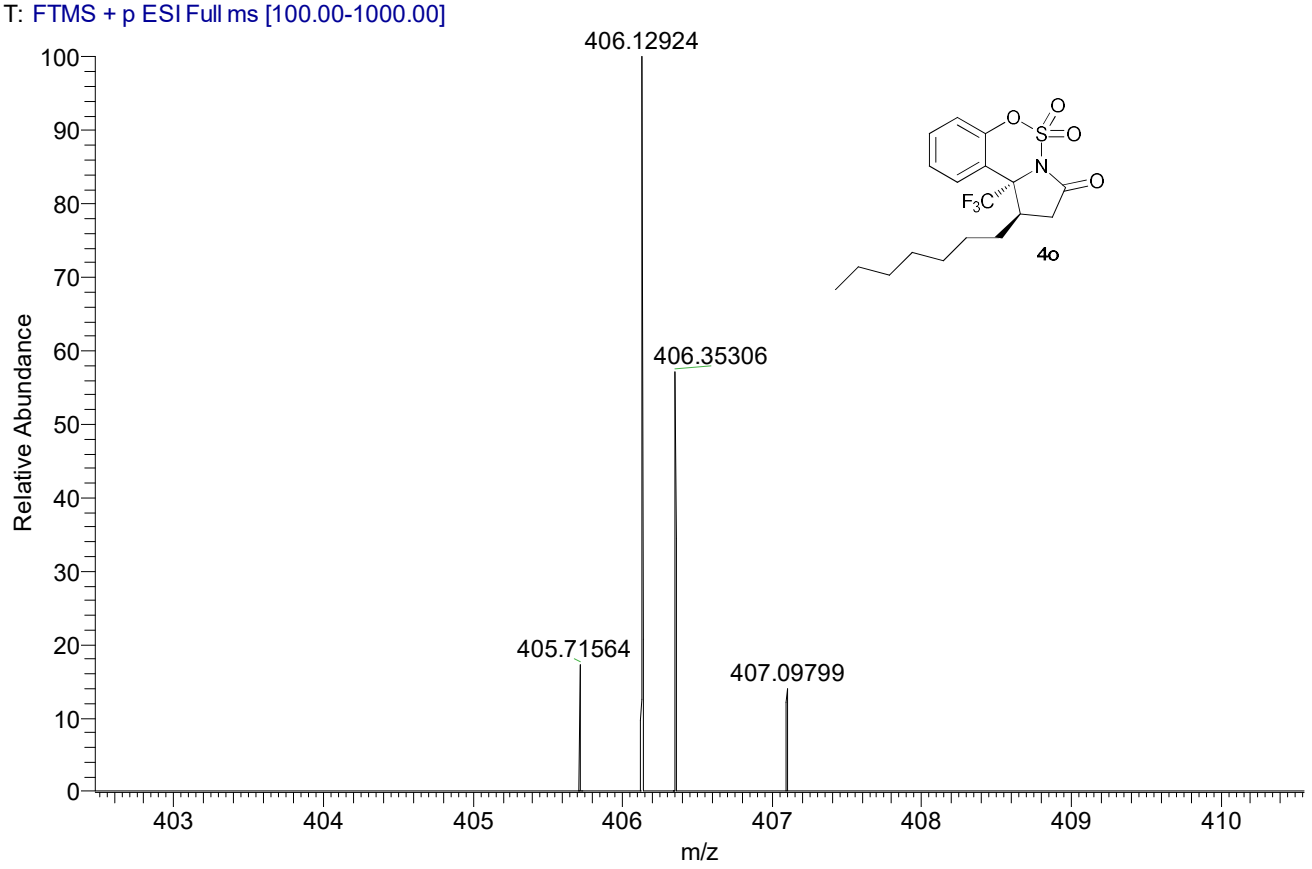

**ZZZ-3-7C.** HRMS (ESI) m/z calcd for $C_{14}H_{15}F_3NO_4S^+$ $(M+H)^+$ 350.06684, found 350.06699.



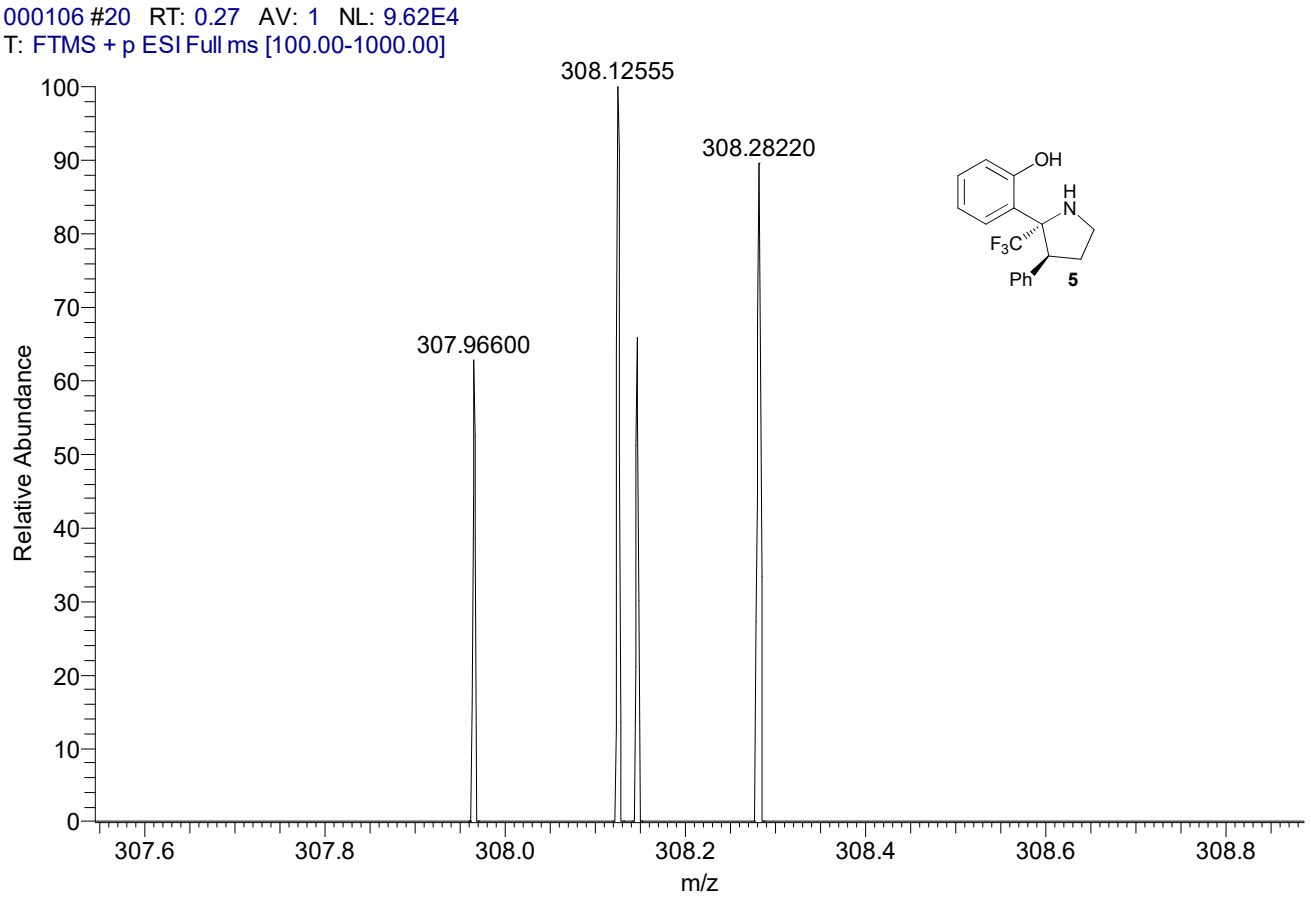
**ZZZ-3-7A.** HRMS (ESI) m/z calcd for $C_{14}H_{15}F_3NO_4S^+$ $(M+H)^+$ 350.06684, found 350.06689.



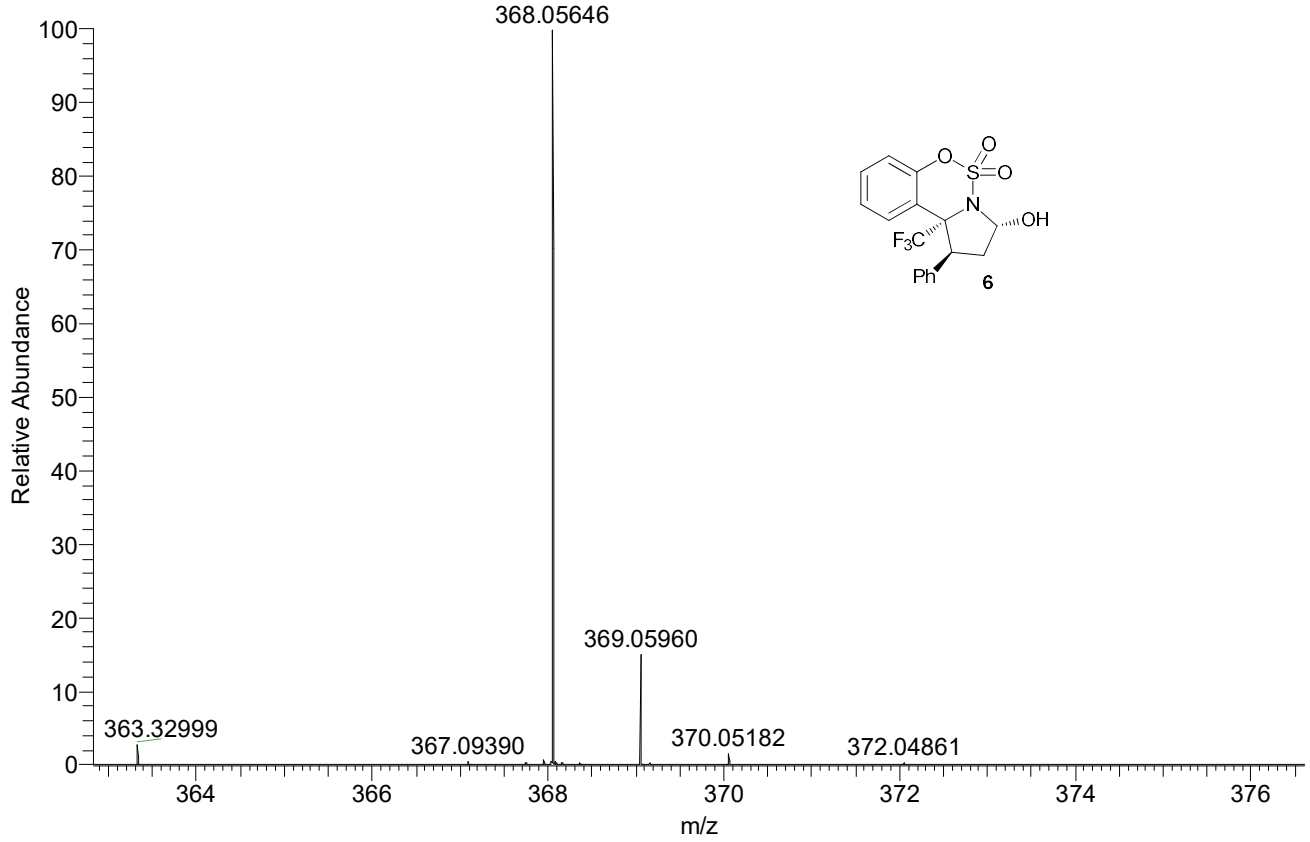

**ZZZ-3-7B.** HRMS (ESI) m/z calcd for $C_{18}H_{23}F_3NO_4S^+$ $(M+H)^+$ 406.12944, found 406.12924.



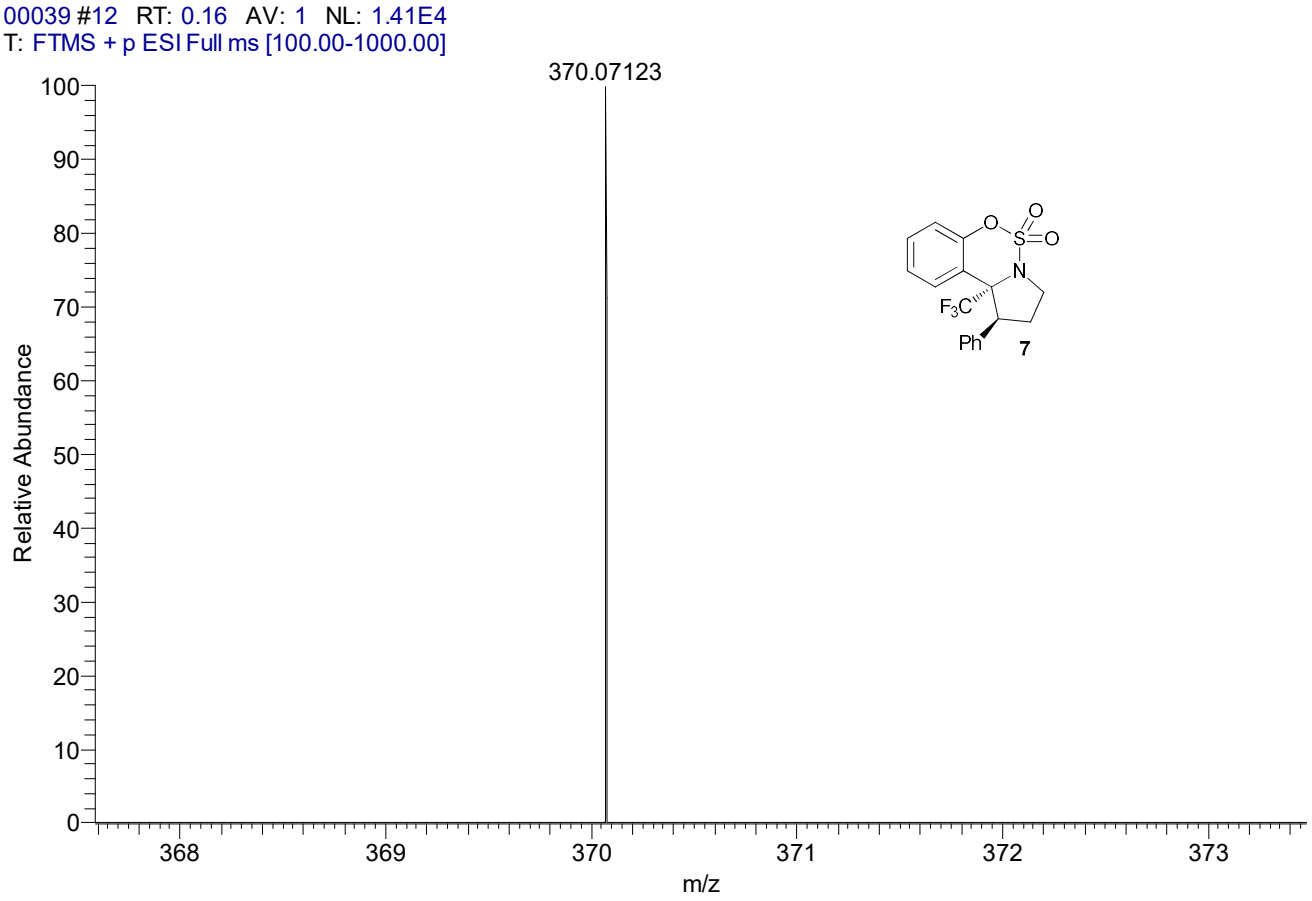
**ZZZ-3-28A.** HRMS (ESI) m/z calcd for $C_{17}H_{17}F_3NO^+$ $(M+H)^+$ 308.12568, found 308.12555.



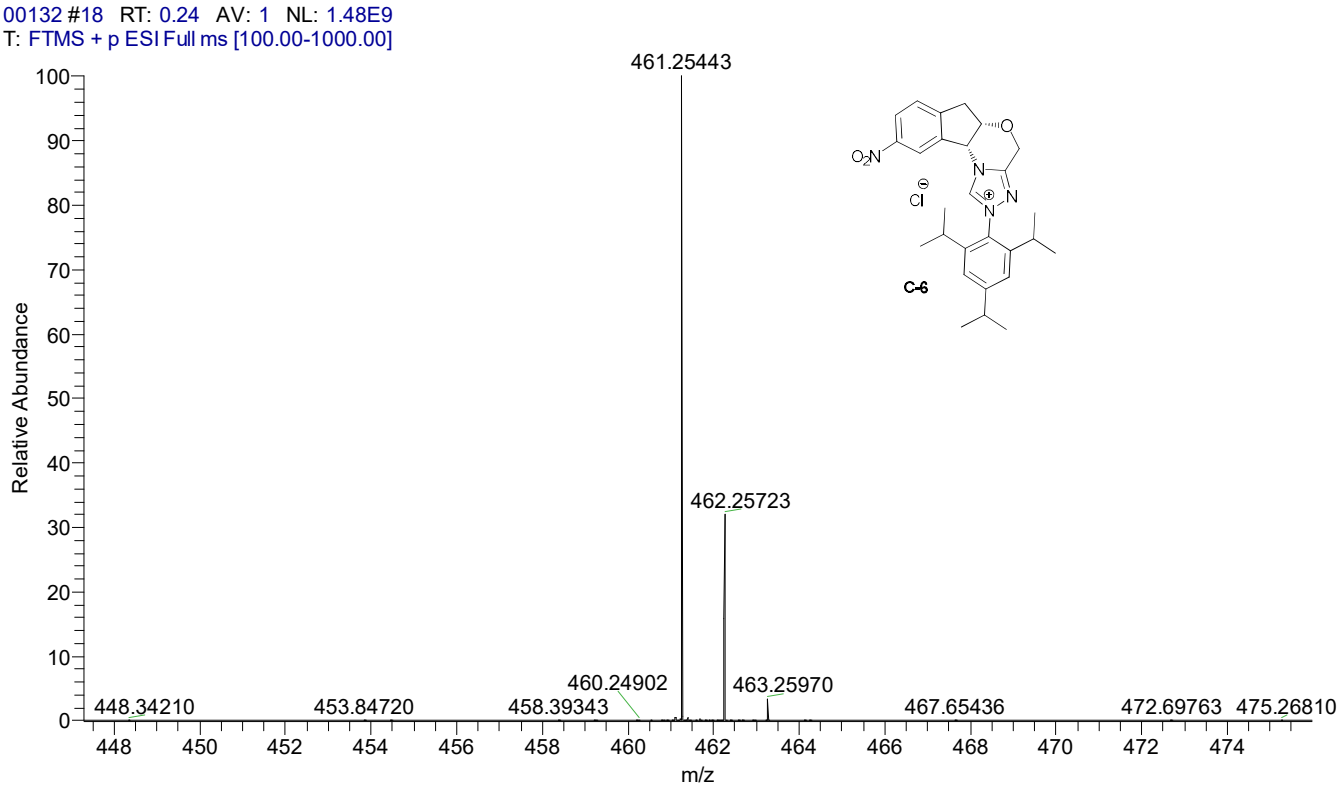

**ZZZ-3-28D.** HRMS (ESI) m/z calcd for $C_{17}H_{13}F_3NO_3S^+$ $(M-OH)^+$ 368.05627, found 368.05646.



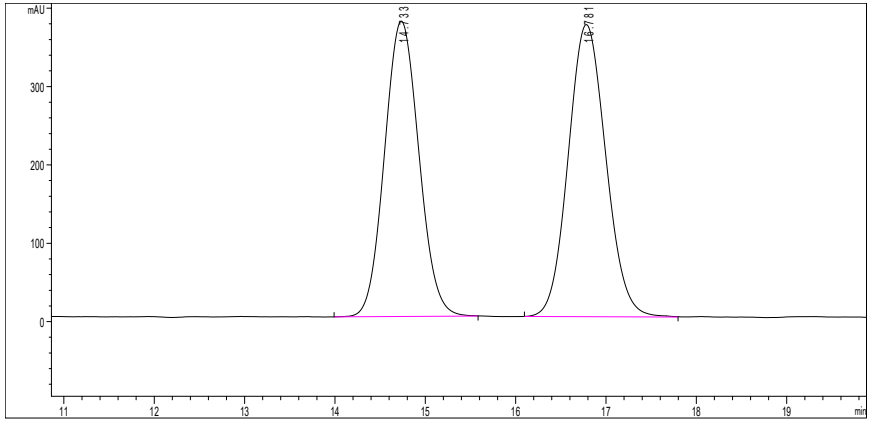
**ZZZ-3-60A.** HRMS (ESI) m/z calcd for $C_{17}H_{15}F_3NO_3S^+$ $(M+H)^+$ 370.07193, found 370.07193.




**ZZZ-2-52.** HRMS (ESI) m/z calcd for $C_{27}H_{33}N_4O_3^+$ $(M-Cl)^+$ 461.25472, found 461.25443.

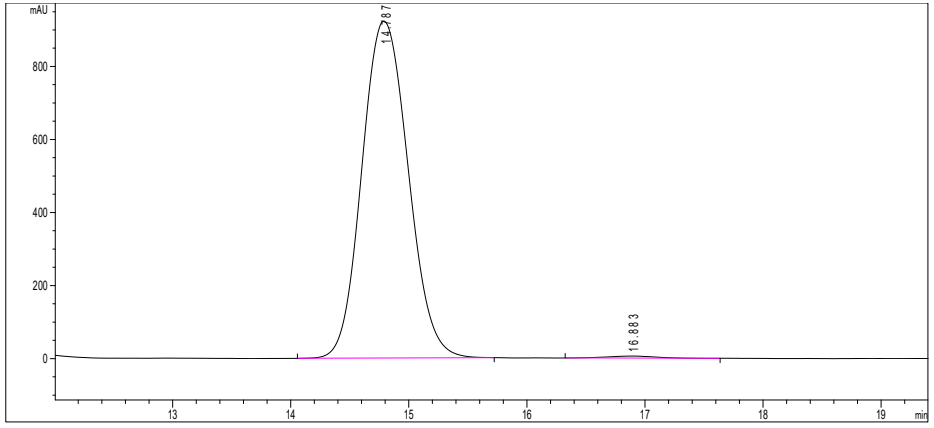


HPLC for racemic and pure enantioenriched sample **3a**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

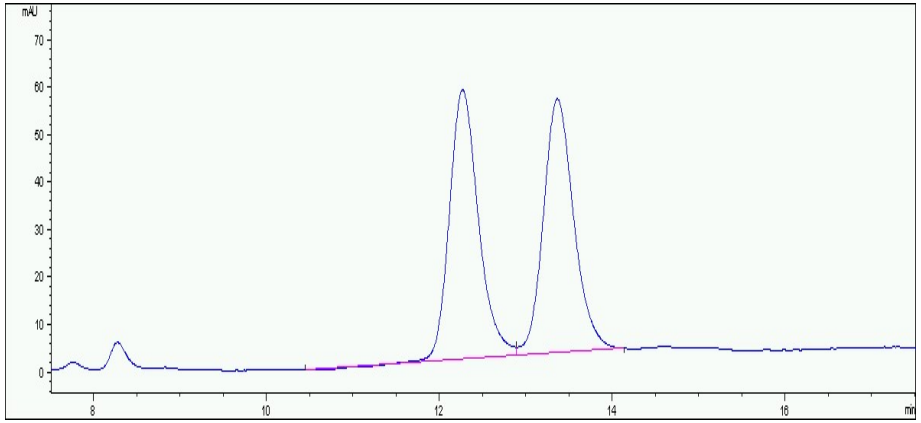
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.733 | BB | 0.4145 | 9974.38086 | 377.22003 |
| 2 | 16.781 | BB | 0.4538 | 1.08014e4 | 373.06921 |



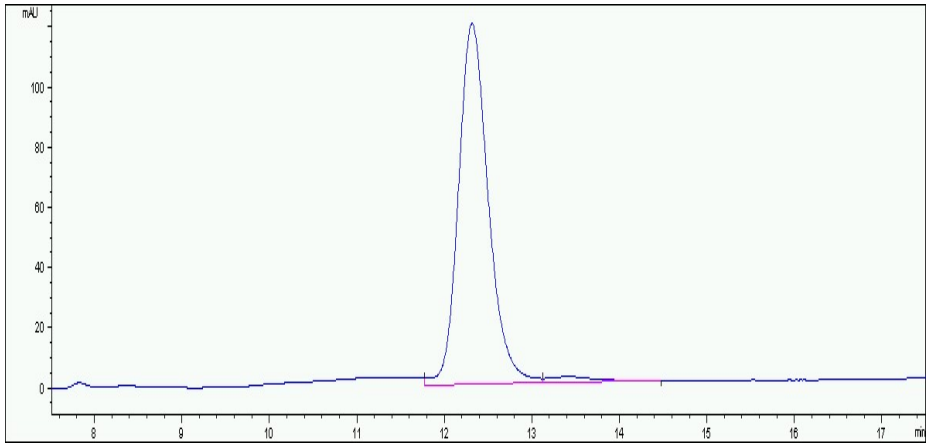

信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.787 | BB | 0.4339 | 2.51610e4 | 923.2721 |
| 2 | 16.883 | BB | 0.3445 | 157.14964 | 5.5473 |

HPLC for racemic and pure enantioenriched sample **3b**

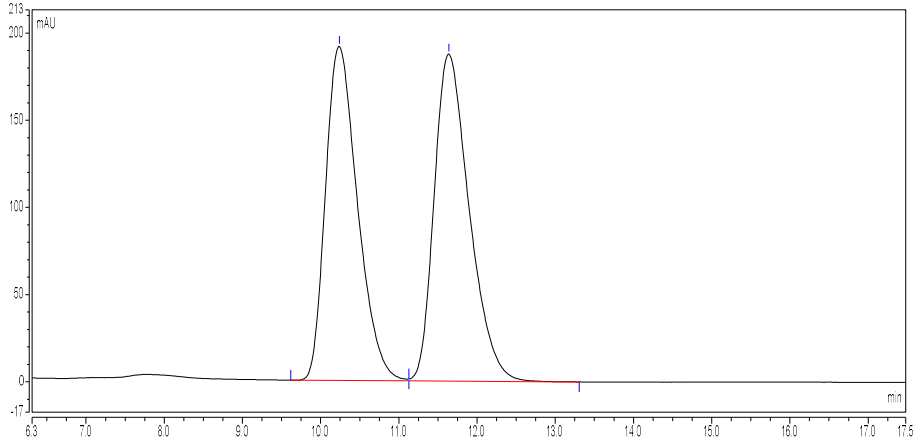



| # | 时间 | 峰面积 | 峰高 | 峰宽 | 对称因 |
|---|---|---|---|---|---|
| 1 | 12.267 | 1344.3 | 56.9 | 0.3586 | 0.797 |
| 2 | 13.362 | 1327.2 | 53.7 | 0.3787 | 0.821 |

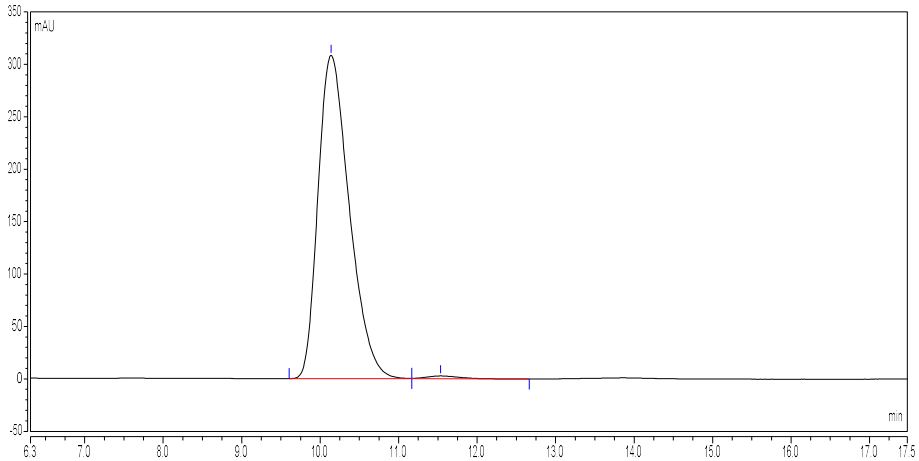



| # | 时间 | 峰面积 | 峰高 | 峰宽 | 对称因 |
|---|---|---|---|---|---|
| 1 | 12.313 | 2860.5 | 120.1 | 0.3653 | 0.799 |
| 2 | 13.402 | 94 | 2.2 | 0.5278 | 0.504 |

HPLC for racemic and pure enantioenriched sample **3c**

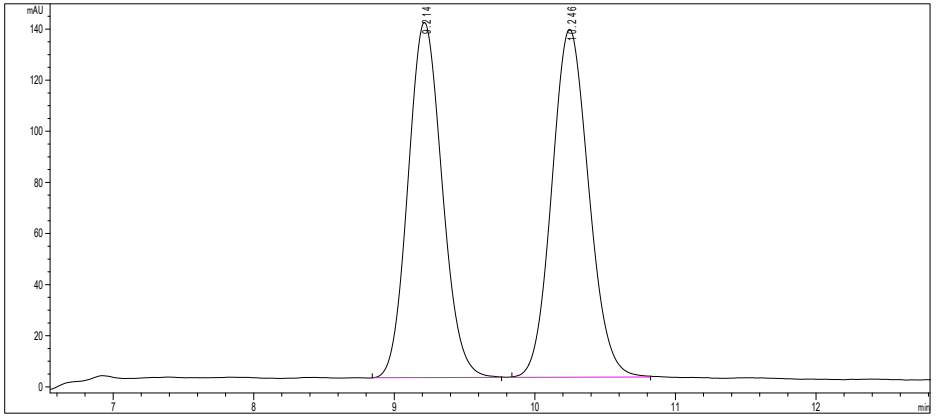



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 10.238 | 48.24 | 90.8264 | 191.61 | 0.440 | 1.32 |



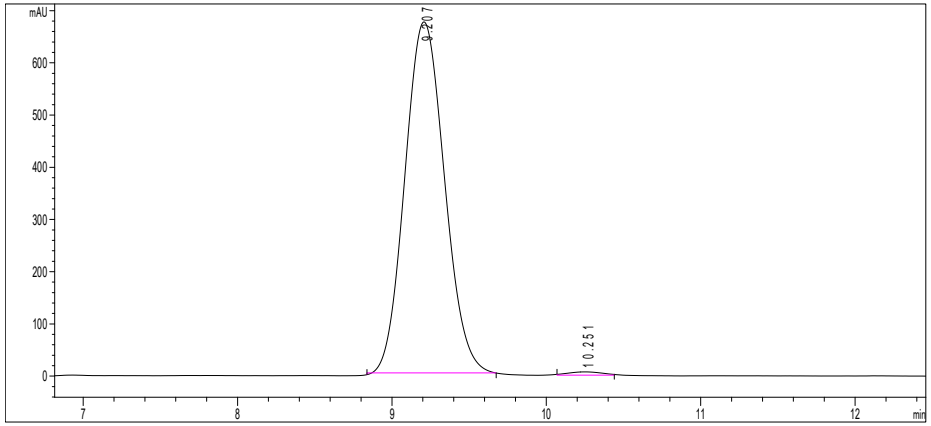

| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 10.138 | 99.05 | 142.4311 | 308.42 | 0.428 | 1.36 |

HPLC for racemic and pure enantioenriched sample **3d**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

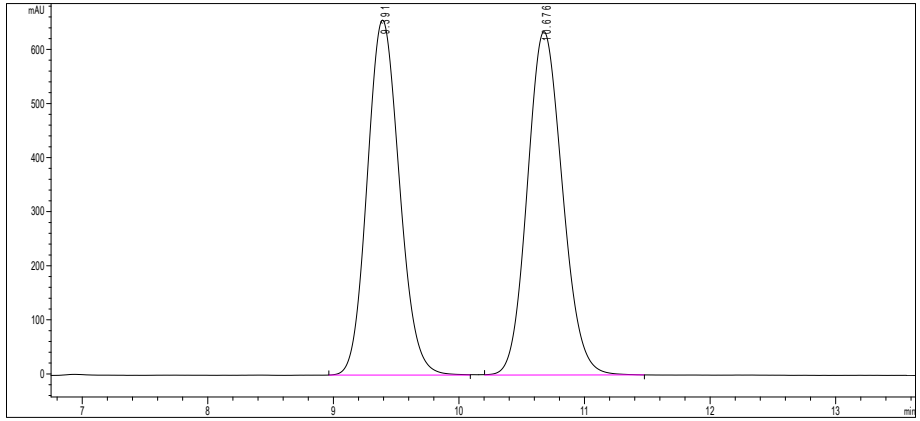
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.214 | BB | 0.2627 | 2345.02075 | 139.0970 |
| 2 | 10.246 | BB | 0.2848 | 2484.55127 | 136.2109 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

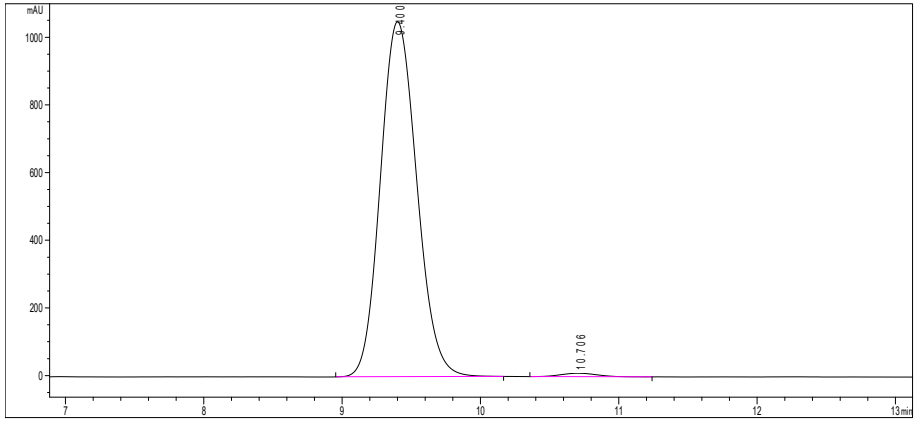
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.207 | MM R | 0.2970 | 1.19929e4 | 672.9564 |
| 2 | 10.251 | MM R | 0.2544 | 96.63966 | 6.3307 |

HPLC for racemic and pure enantioenriched sample **3e**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

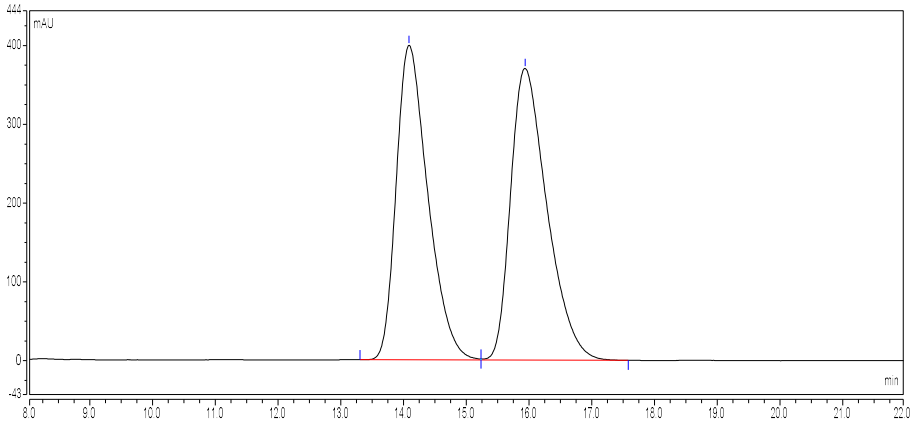
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.391 | BB | 0.2780 | 1.17087e4 | 656.6103 |
| 2 | 10.676 | BB | 0.3027 | 1.23439e4 | 635.2558 |



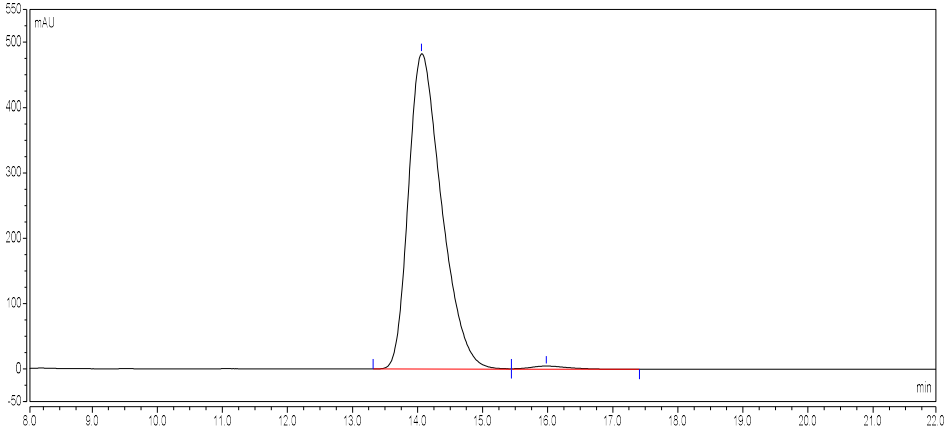

信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.400 | BB | 0.2893 | 1.91947e4 | 1049.73572 |
| 2 | 10.706 | BB | 0.2756 | 185.89107 | 10.24907 |

HPLC for racemic and pure enantioenriched sample **3f**

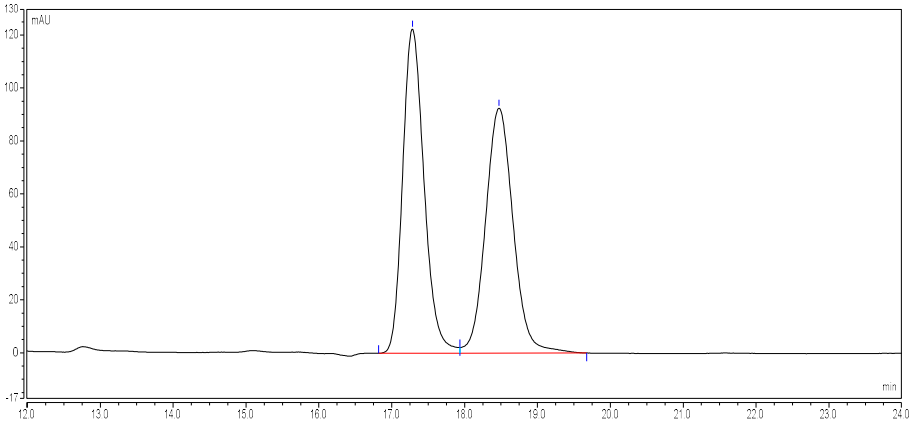



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 14.090 | 48.40 | 233.2890 | 399.16 | 0.538 | 1.38 |

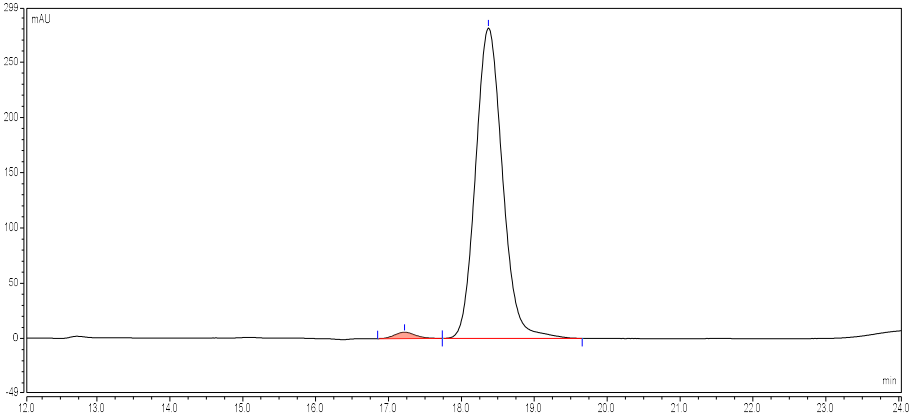



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 14.062 | 98.93 | 283.7720 | 482.64 | 0.541 | 1.40 |

HPLC for racemic and pure enantioenriched sample **3g**

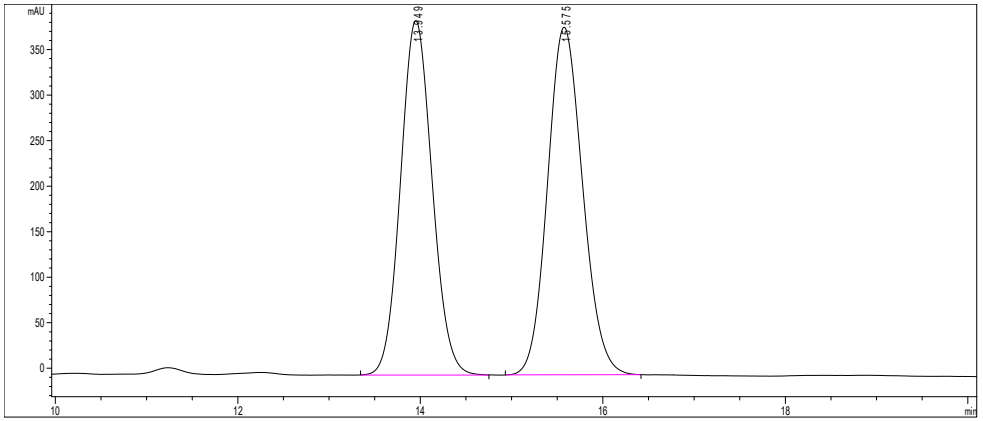



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.283 | 50.66 | 41.7577 | 122.47 | 0.312 | 1.22 |



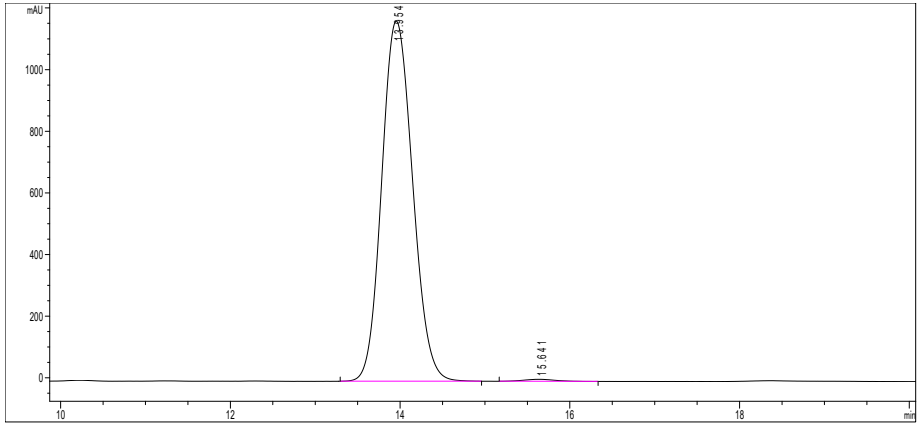

| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.222 | 1.58 | 1.9289 | 5.73 | 0.310 | 1.17 |

HPLC for racemic and pure enantioenriched sample **3h**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

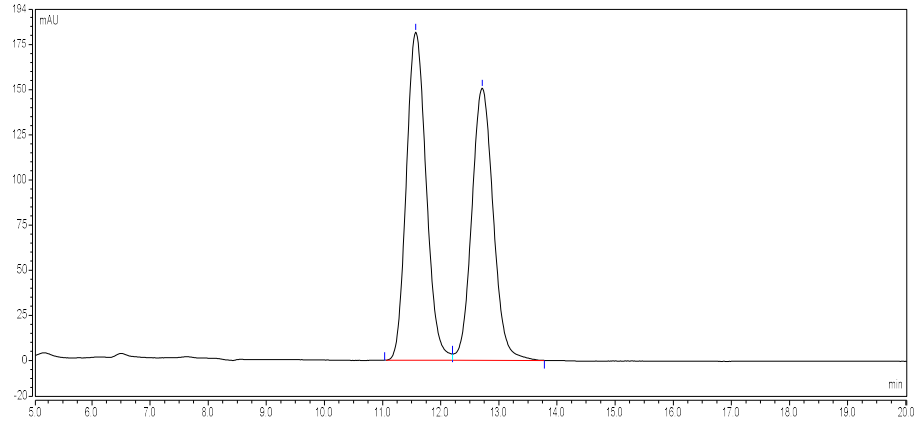
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.949 | BB | 0.3748 | 9370.86426 | 389.3643 |
| 2 | 15.575 | BB | 0.4139 | 1.02106e4 | 381.9708 |



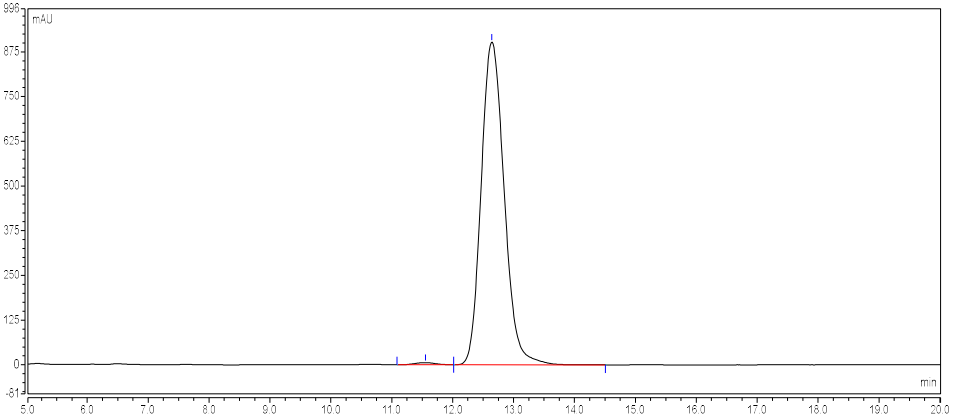

信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.954 | BB | 0.3992 | 2.95731e4 | 1168.6740 |
| 2 | 15.641 | BB | 0.3135 | 167.22672 | 6.5136 |

HPLC for racemic and pure enantioenriched sample **3i**

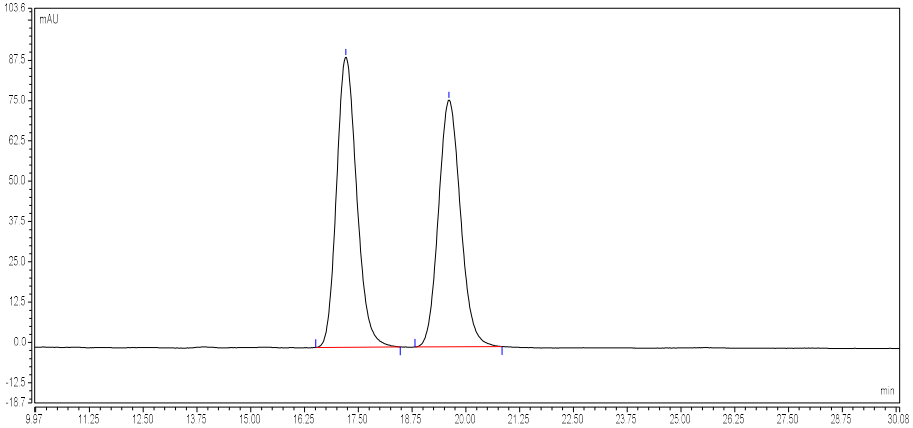



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 11.570 | 52.68 | 71.7763 | 181.84 | 0.368 | 1.15 |

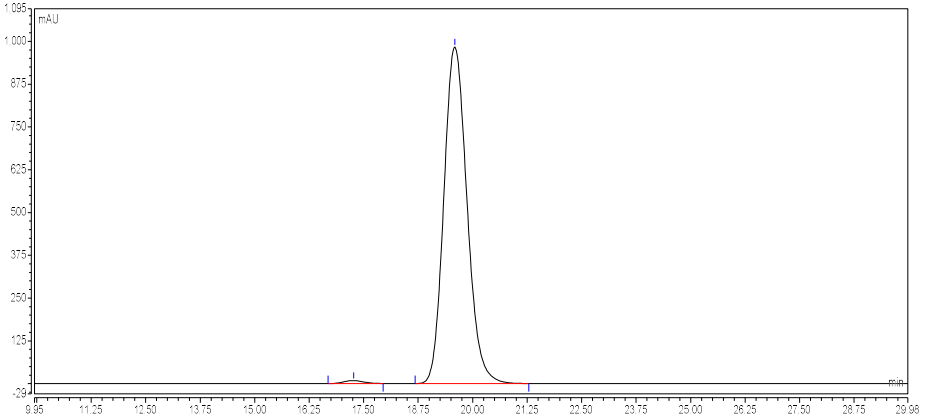



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 11.555 | 0.62 | 2.4636 | 6.19 | 0.373 | n.a. |

HPLC for racemic and pure enantioenriched sample **3j**

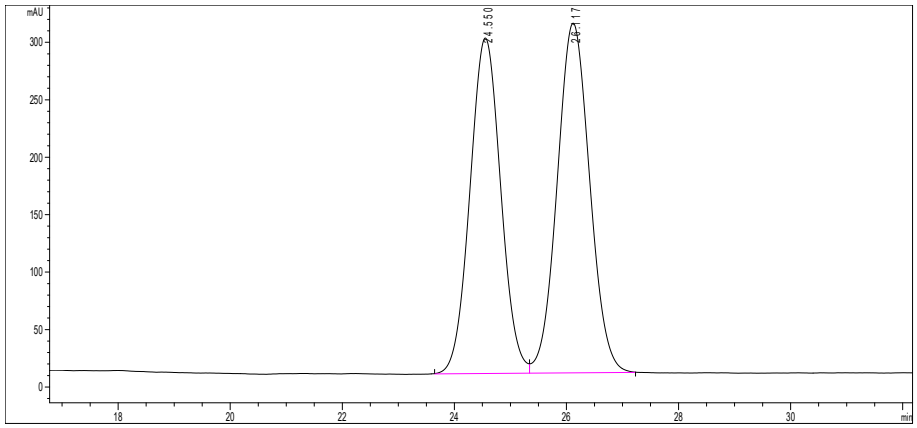



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.210 | 51.82 | 48.9552 | 90.00 | 0.500 | 1.18 |



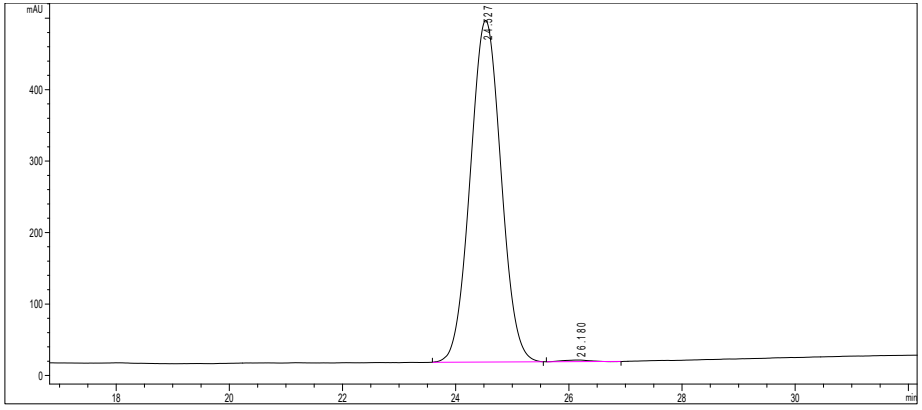

| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.268 | 0.74 | 4.5345 | 8.69 | 0.494 | 1.07 |

HPLC for racemic and pure enantioenriched sample **3k**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

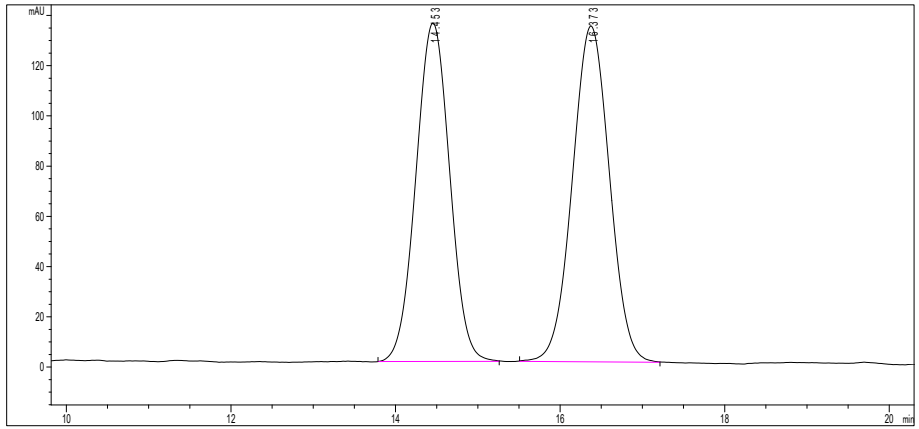
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 24.550 | BV | 0.5968 | 1.11363e4 | 292.1033( |
| 2 | 26.117 | VB | 0.6283 | 1.22725e4 | 304.4364( |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

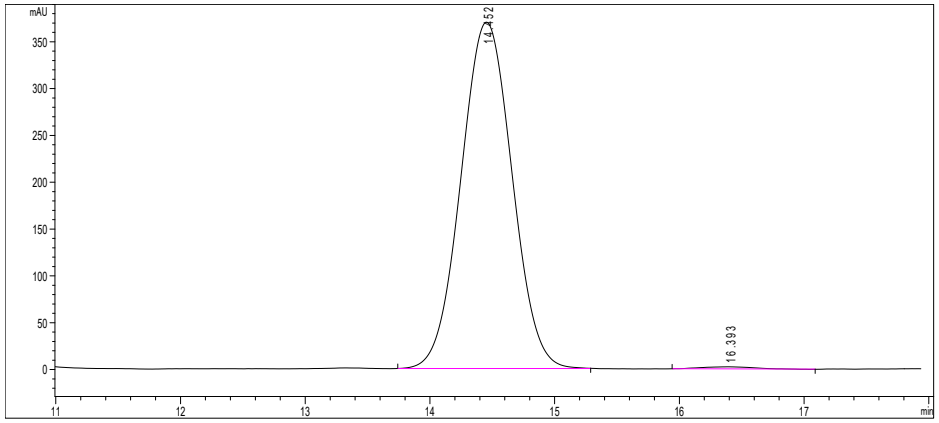
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 24.527 | BB | 0.5912 | 1.80848e4 | 478.2661 |
| 2 | 26.180 | BB | 0.4116 | 79.97885 | 2.3585 |

HPLC for racemic and pure enantioenriched sample **4b**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

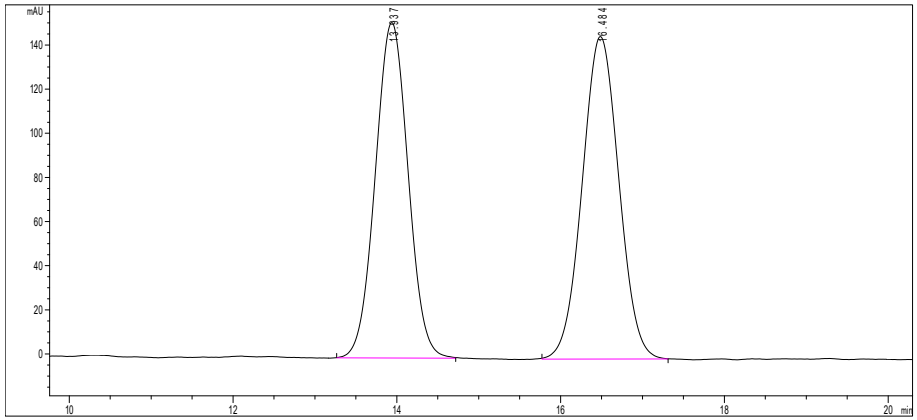
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.453 | BB | 0.4510 | 3848.14404 | 134.8077 |
| 2 | 16.373 | BB | 0.4853 | 4186.27393 | 133.7030 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

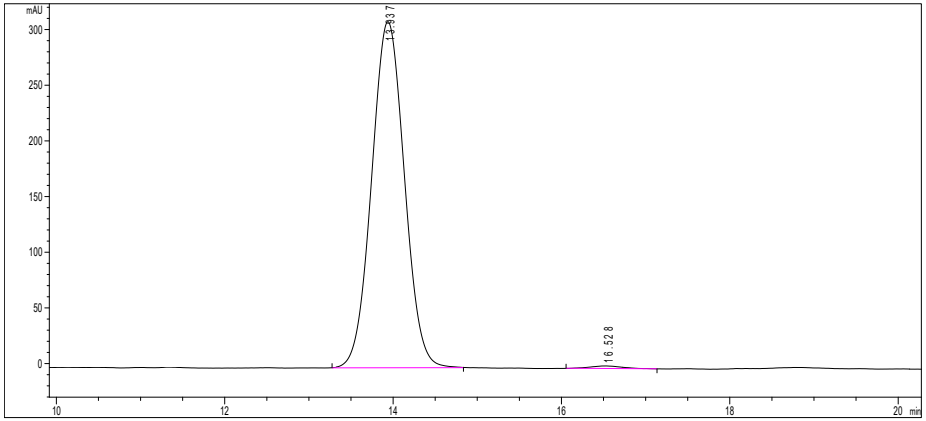
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.452 | BB | 0.4480 | 1.05281e4 | 369.9452 |
| 2 | 16.393 | BB | 0.3587 | 69.37284 | 2.3347 |

HPLC for racemic and pure enantioenriched sample **4c**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

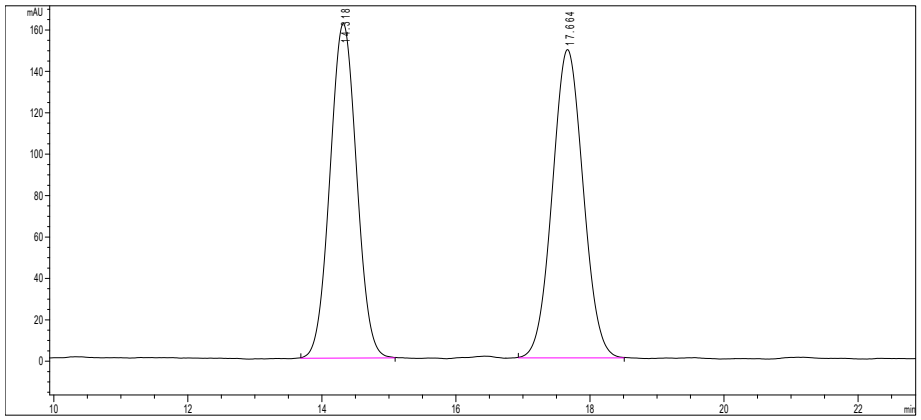
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.937 | BB | 0.4407 | 4231.21484 | 152.0092 |
| 2 | 16.484 | BB | 0.4805 | 4540.80322 | 146.1484 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

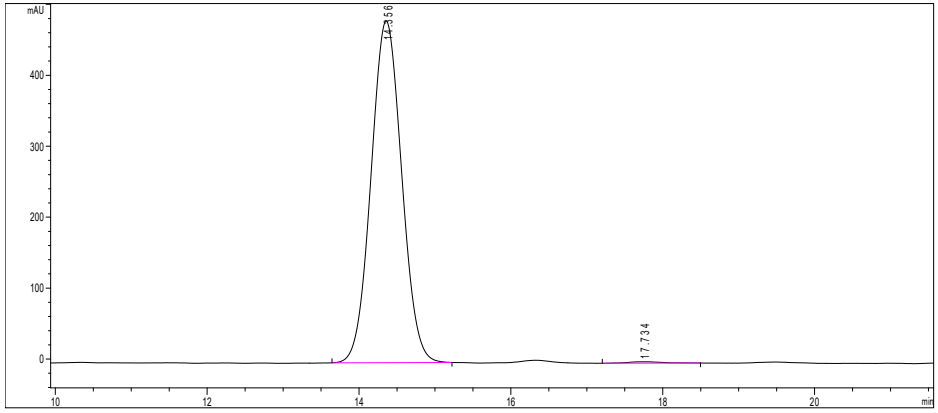
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.937 | BB | 0.4212 | 8401.12305 | 310.9646 |
| 2 | 16.528 | BB | 0.3345 | 66.23709 | 2.3964 |

HPLC for racemic and pure enantioenriched sample **4d**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

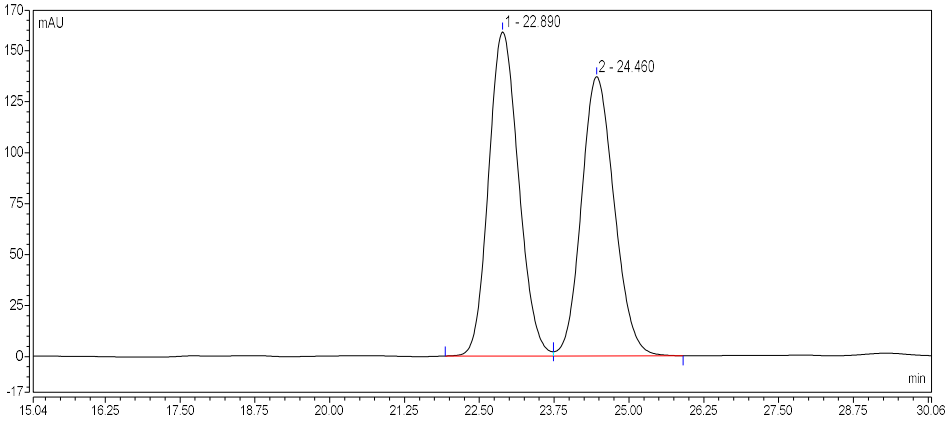
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.318 | BB | 0.4344 | 4468.49072 | 161.6822 |
| 2 | 17.664 | BB | 0.4950 | 4762.43359 | 148.9679 |



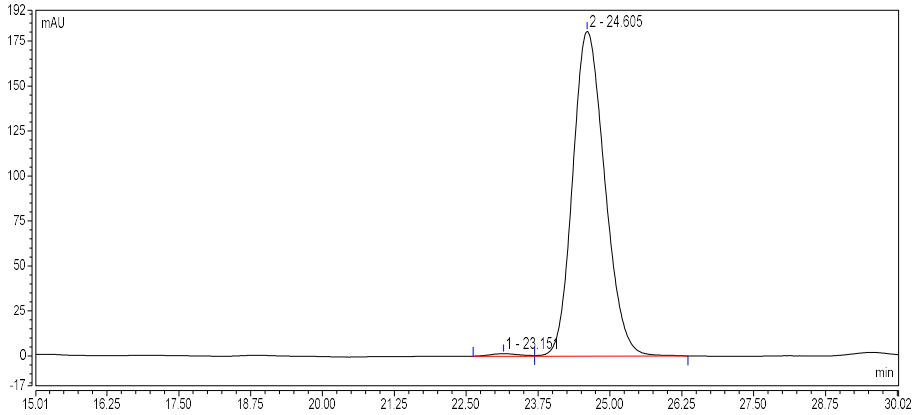

信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.356 | BB | 0.4386 | 1.34872e4 | 481.7459 |
| 2 | 17.734 | BB | 0.3928 | 64.38531 | 1.9622 |

HPLC for racemic and pure enantioenriched sample **4e**

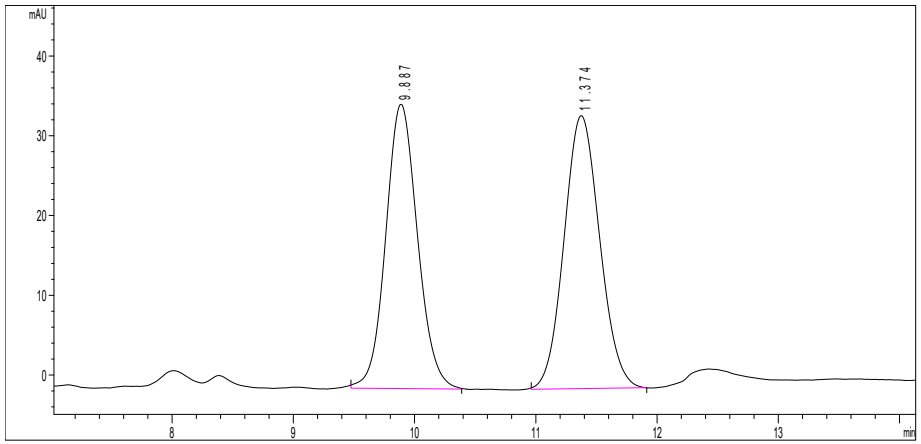



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 22.890 | 51.47 | 92.2570 | 158.93 | 0.542 | 1.13 |



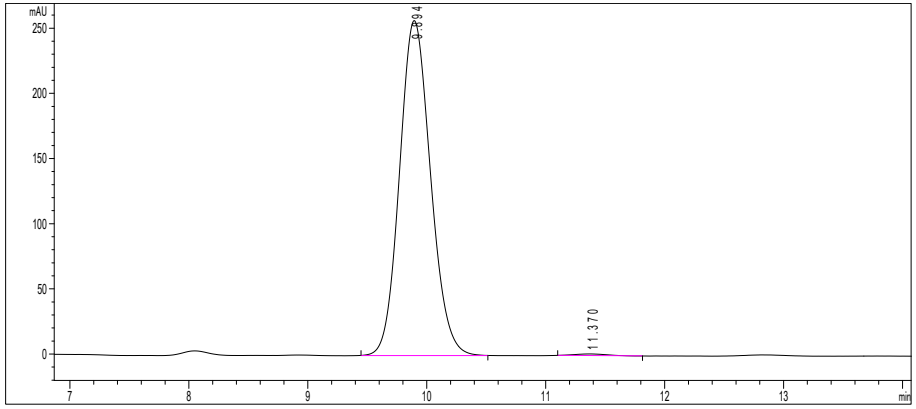

| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 23.151 | 0.71 | 0.8350 | 1.43 | 0.554 | n.a. |

HPLC for racemic and pure enantioenriched sample **4f**




信号 2: DAD1 B, Sig=230,8 Ref=360,100

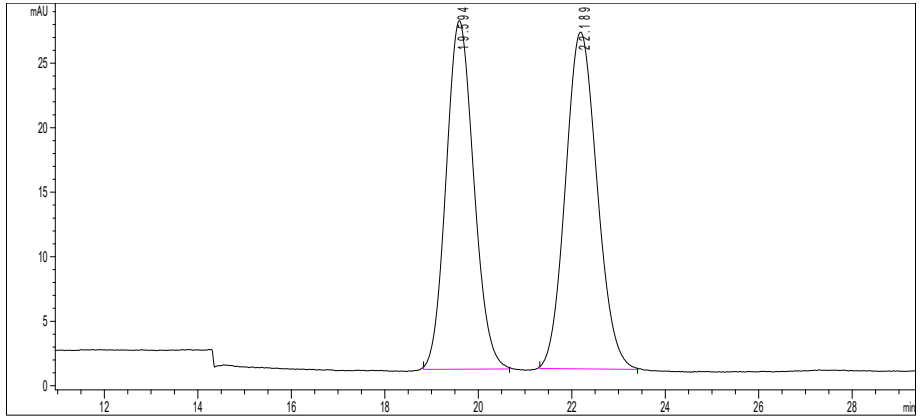
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.887 | BB | 0.2823 | 649.10260 | 35.6726 |
| 2 | 11.374 | BB | 0.3164 | 699.57166 | 34.2412 |




信号 2: DAD1 B, Sig=230,8 Ref=360,100

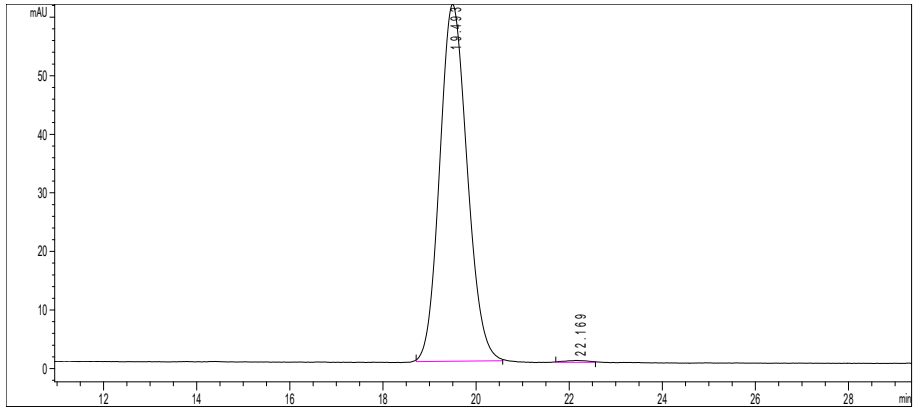
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 9.894 | BB | 0.2814 | 4658.14551 | 257.0625 |
| 2 | 11.370 | BB | 0.2665 | 25.03966 | 1.3128 |

HPLC for racemic and pure enantioenriched sample **4g**




信号 1: DAD1 A, Sig=254,4 Ref=360,100

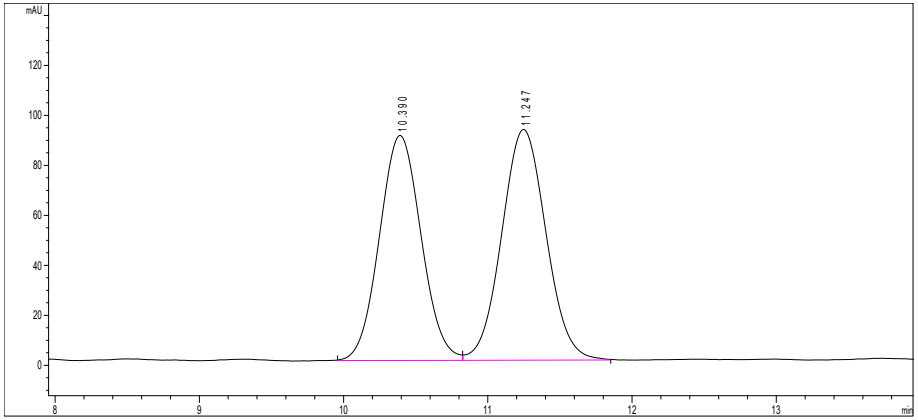
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 19.594 | BB | 0.6025 | 1112.63269 | 27.01707 |
| 2 | 22.189 | BB | 0.6912 | 1229.36792 | 26.11655 |




信号 1: DAD1 A, Sig=254,4 Ref=360,100

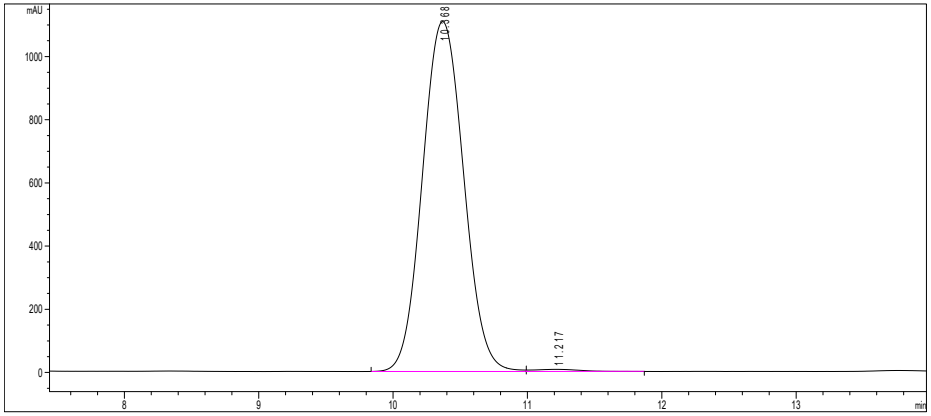
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 19.493 | BB | 0.6337 | 2496.04663 | 60.9549 |
| 2 | 22.169 | MM R | 0.5324 | 14.49009 | 4.53614e- |

HPLC for racemic and pure enantioenriched sample **4h**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

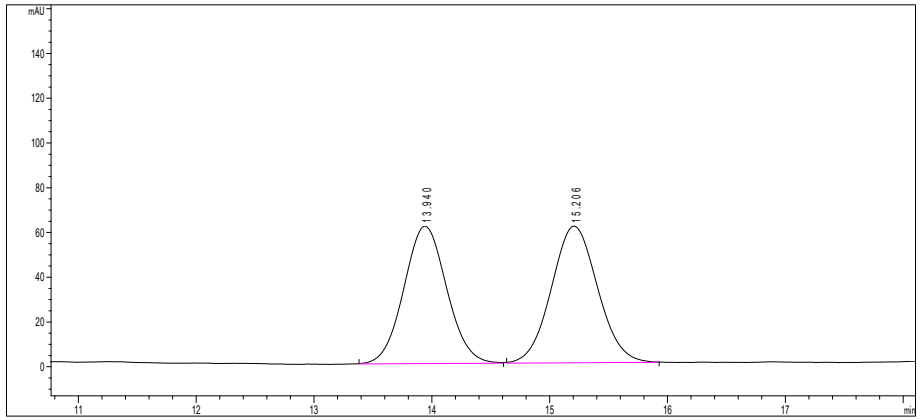
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 10.390 | BV | 0.3181 | 1805.88306 | 90.0153 |
| 2 | 11.247 | VB | 0.3352 | 1971.69714 | 92.3251 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

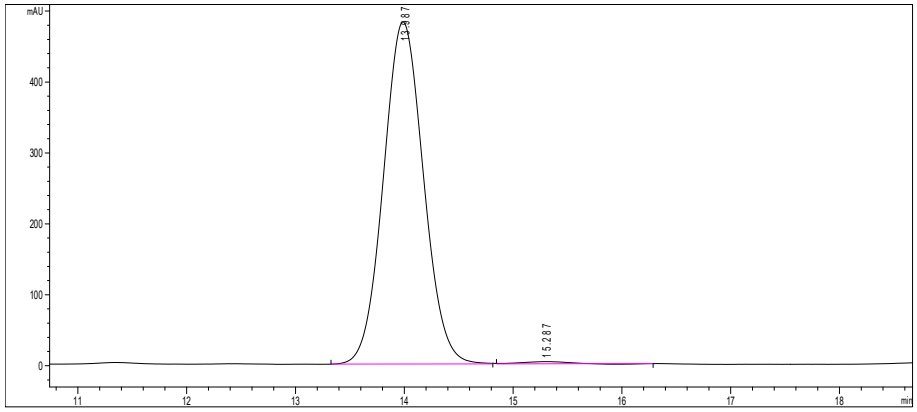
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 10.368 | BV | 0.3363 | 2.35829e4 | 1107.8891 |
| 2 | 11.217 | VB | 0.3234 | 162.23694 | 6.7752 |

HPLC for racemic and pure enantioenriched sample **4j**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

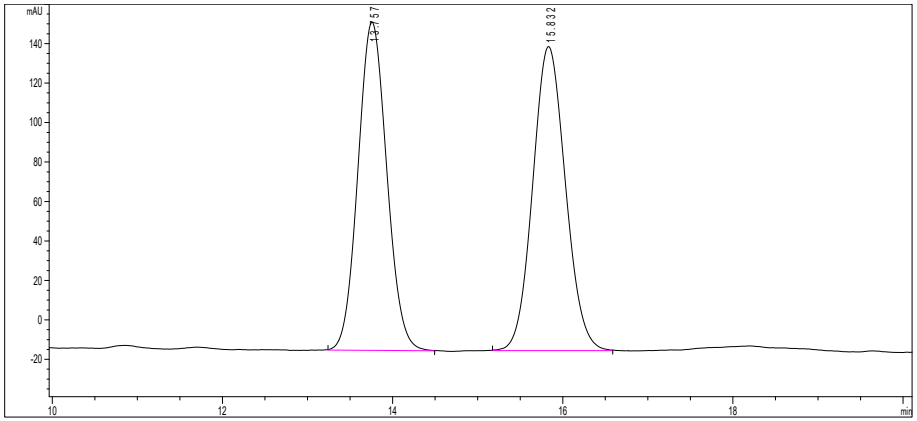
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.940 | BB | 0.3891 | 1555.06543 | 61.4755 |
| 2 | 15.206 | BB | 0.4104 | 1667.90527 | 61.1178 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

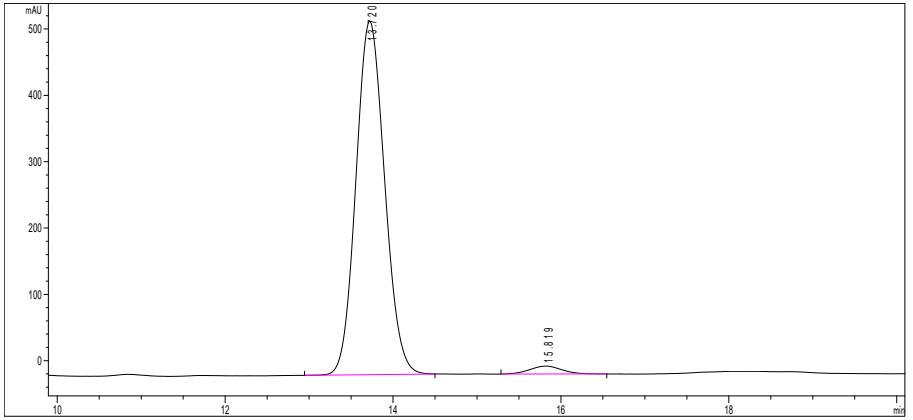
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.987 | BB | 0.3965 | 1.23441e4 | 482.4874 |
| 2 | 15.287 | BB | 0.3179 | 81.16329 | 3.0957 |

HPLC for racemic and pure enantioenriched sample **4k**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

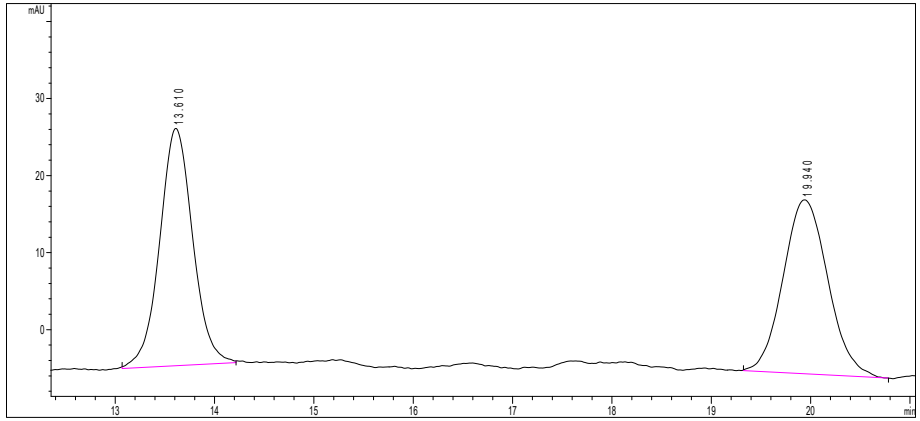
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.757 | BB | 0.3668 | 3921.47534 | 166.4706 |
| 2 | 15.832 | BB | 0.4118 | 4119.40918 | 154.1367 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

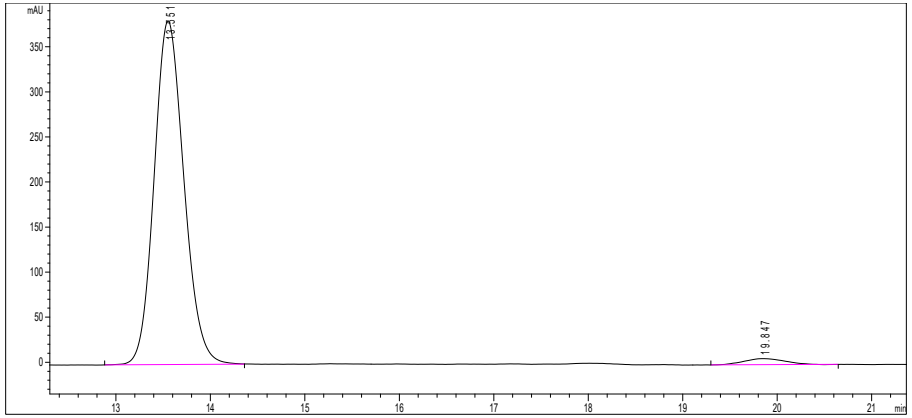
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.720 | BB | 0.3730 | 1.26511e4 | 532.8713 |
| 2 | 15.819 | BB | 0.3489 | 318.81372 | 11.9644 |

HPLC for racemic and pure enantioenriched sample **4l**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

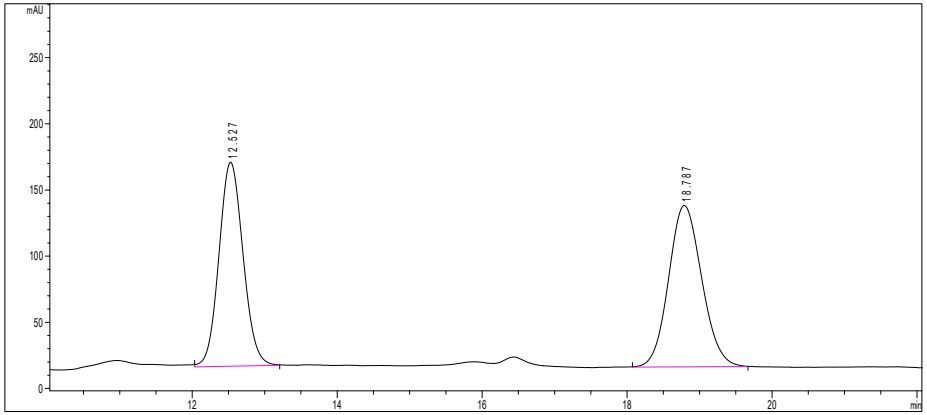
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.610 | BB | 0.3521 | 713.70056 | 30.8240 |
| 2 | 19.940 | BB | 0.4471 | 706.36975 | 22.5712 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

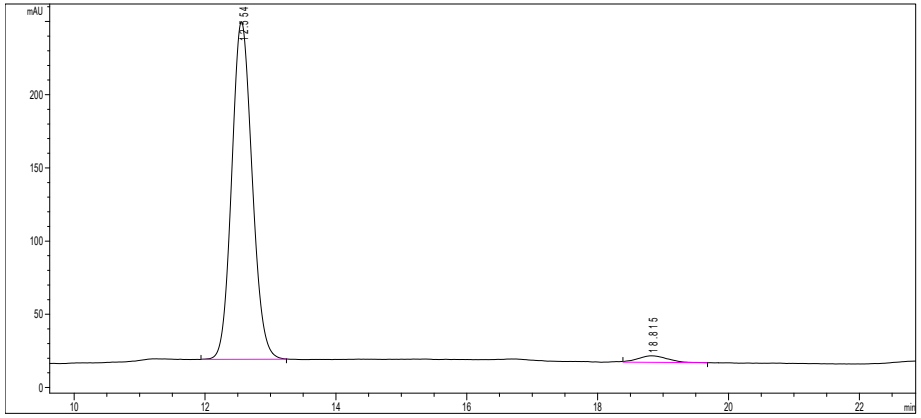
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 13.551 | BB | 0.3390 | 8337.15234 | 381.4297 |
| 2 | 19.847 | BB | 0.3708 | 205.60515 | 6.7606 |

HPLC for racemic and pure enantioenriched sample **4m**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

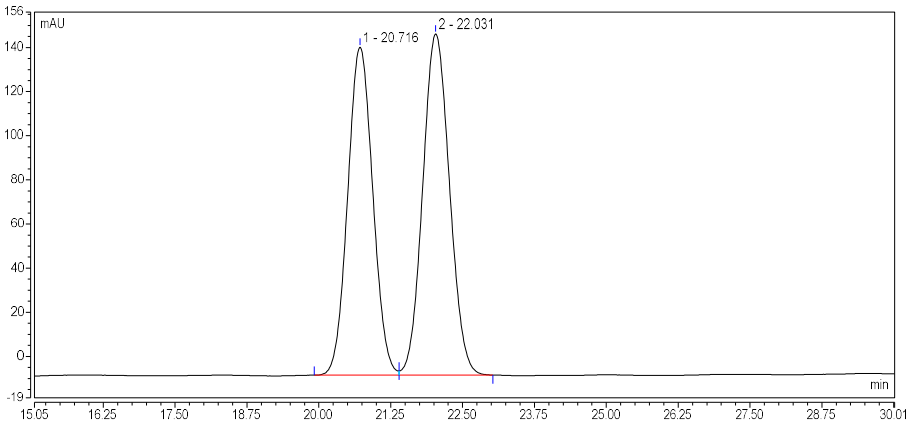
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 12.527 | BB | 0.3596 | 3566.44873 | 154.36166 |
| 2 | 18.787 | BB | 0.4936 | 3888.78491 | 122.08109 |



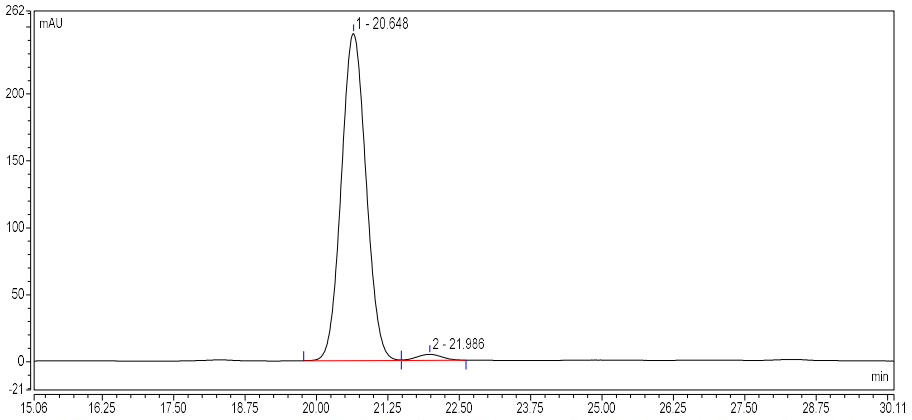

信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 12.554 | BB | 0.3367 | 4997.74609 | 230.6966 |
| 2 | 18.815 | BB | 0.3844 | 142.31018 | 4.4822 |

HPLC for racemic and pure enantioenriched sample**4n**

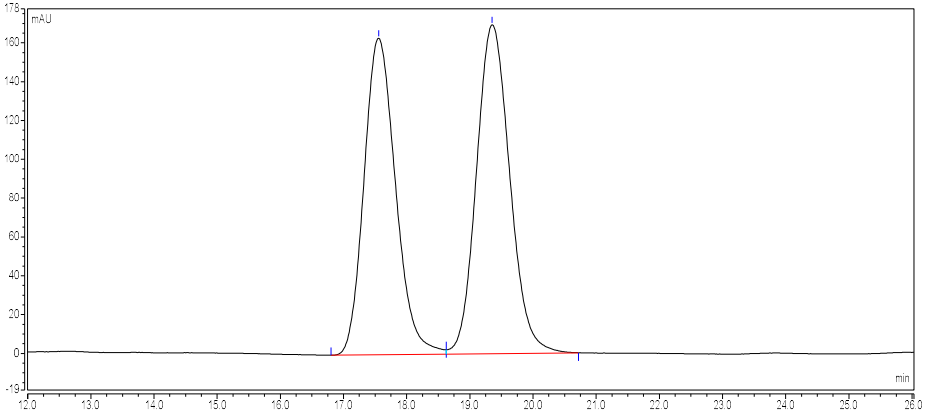



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 20.716 | 49.40 | 75.1893 | 148.62 | 0.475 | 1.05 |

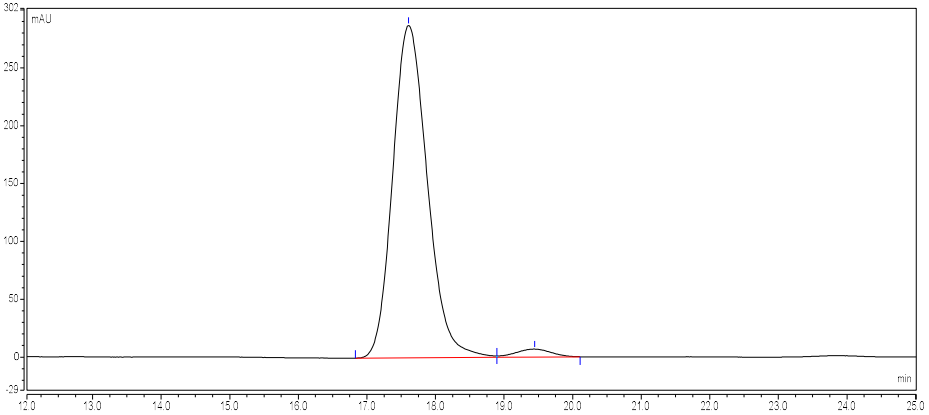



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 20.648 | 98.19 | 122.2400 | 244.16 | 0.469 | 1.05 |

HPLC for racemic and pure enantioenriched sample **4o**

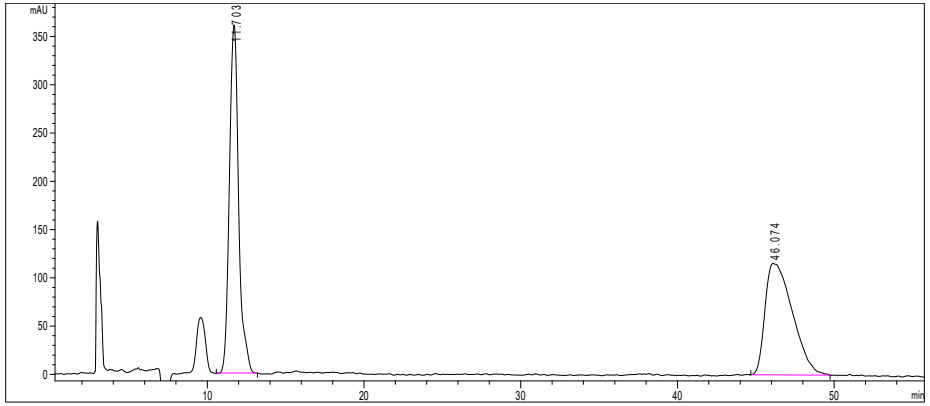



| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.555 | 47.69 | 94.4127 | 163.08 | 0.533 | 1.18 |



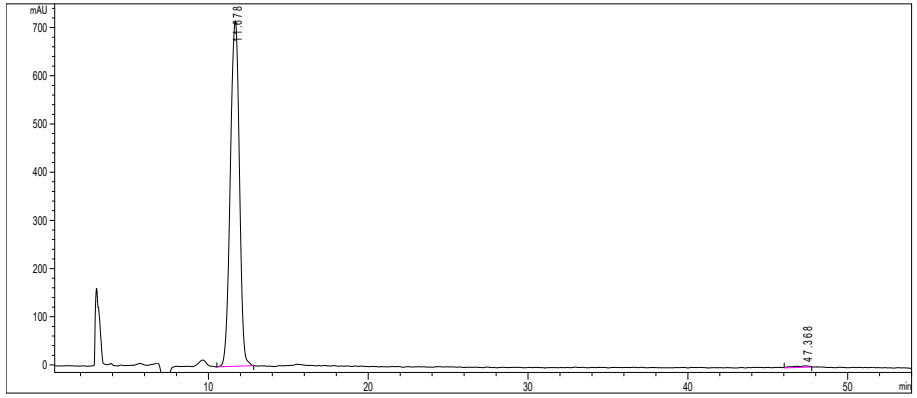

| 峰序号 | 保留时间 min | 相对峰面积 % | 峰面积 mAU*min | 峰高 mAU | 峰宽 (50%) | 不对称度 EP |
|---|---|---|---|---|---|---|
| 1 | 17.608 | 97.58 | 167.8129 | 287.31 | 0.539 | 1.17 |

HPLC for racemic and pure enantioenriched sample **5**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

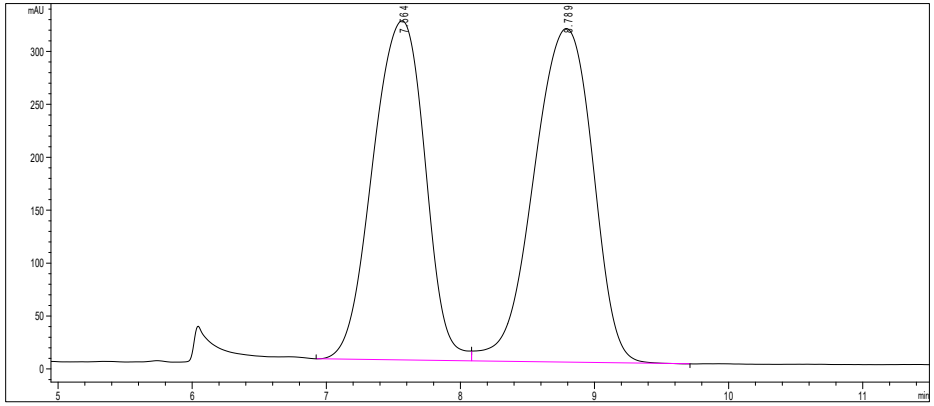
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 11.703 | MM R | 0.6838 | 1.48033e4 | 360.8089 |
| 2 | 46.074 | BB | 1.4303 | 1.40427e4 | 115.6786 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

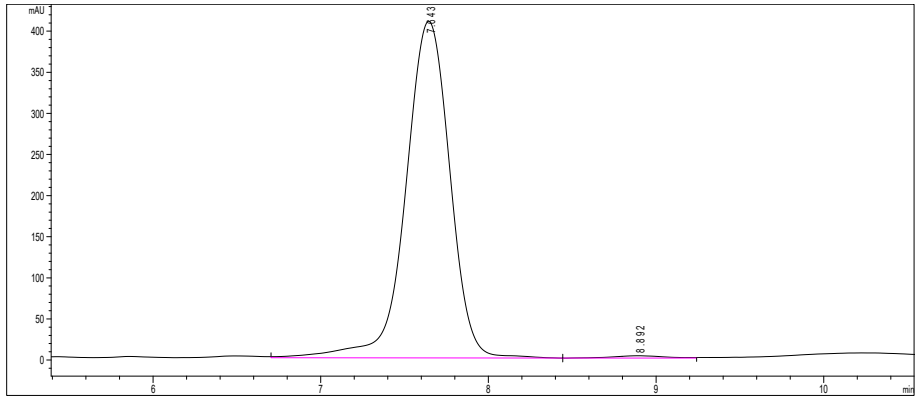
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 11.678 | VB | 0.6301 | 2.82759e4 | 716.9333 |
| 2 | 47.368 | MM R | 1.2978 | 237.00111 | 3.0435 |

HPLC for racemic and pure enantioenriched sample **6**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

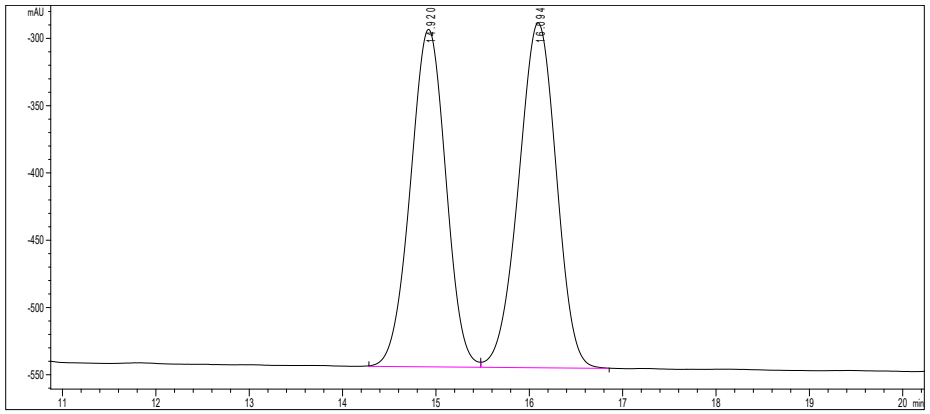
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 7.564 | BV | 0.4398 | 8672.61230 | 320.4353 |
| 2 | 8.789 | VB | 0.4940 | 9620.07910 | 315.2989 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

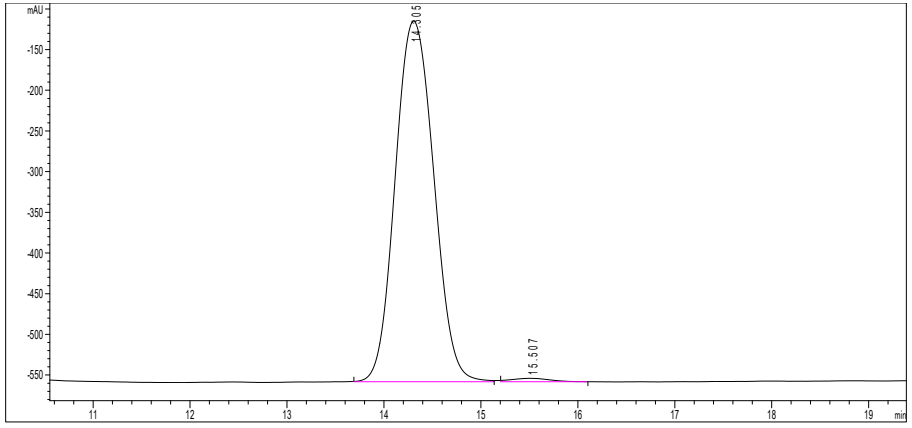
| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 7.643 | VB | 0.2861 | 7519.65869 | 409.6842 |
| 2 | 8.892 | BV | 0.2945 | 65.51433 | 2.9564 |

HPLC for racemic and pure enantioenriched sample **7**




信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.920 | BV | 0.4109 | 6597.92041 | 250.8217 |
| 2 | 16.094 | VB | 0.4391 | 7189.80811 | 256.4325 |




信号 3: DAD1 C, Sig=210,8 Ref=360,100

| 峰 # | 保留时间 [min] | 类型 | 峰宽 [min] | 峰面积 [mAU*s] | 峰高 [mAU] |
|---|---|---|---|---|---|
| 1 | 14.305 | BB | 0.4445 | 1.22544e4 | 443.2915 |
| 2 | 15.507 | BB | 0.3607 | 115.59990 | 4.2311 |