Electrochemical sulfonylation of alkenes with sulfonyl hydrazides: a metal- and oxidant-free protocol for the synthesis of (*E*)-vinyl sulfones in water

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Supporting Information

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1. General information

Unless otherwise noted, all solvents and reagents were obtained commercially and used without further purification. Analytical thin layer chromatography (TLC) plates and the silica gel (200−300 mesh) for column chromatography were phased from Qingdao Haiyang Chemical and Special Silica Gel Co, Ltd.

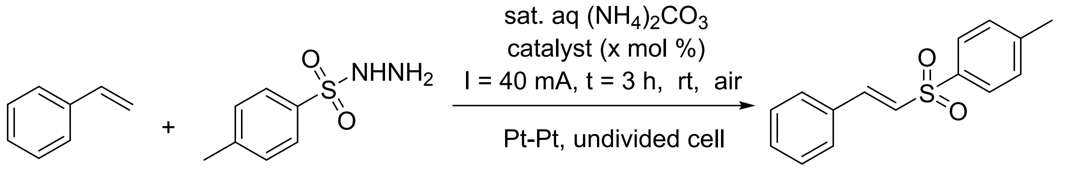
Proton nuclear magnetic resonance (^{1}H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectroscopy were performed on Bruker Advance III-400 spectrometers (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR). Chemical shifts of ^{1}H NMR and ^{13}C NMR spectra were reported as in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0 ppm) and relative to the signal of chloroform-d (δ **7.26** ppm for ^{1}H NMR and δ **77.2** ppm for ^{13}C NMR). Multiplicities were given as: s (singlet); br s (broad singlet); d (doublet); t (triplet); q (quartet); m (multiplets), etc. The number of protons (n) for a given resonance was indicated by nH.

Electrolysis reactions were conducted using a Model QJ3005T (32V) DC power supply purchased from Ningbo Jiuyuan Electronic Co., Ltd., China.
Cyclic voltammetry (CV) analysis was performed on CHI660E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China), using a glassy carbon electrode (GCE) (d = 3 mm) as working electrode, a Pt wire as counter electrode, and a saturated calomel electrode (SCE) as a reference electrode. Cyclic voltammograms were recorded at 100 mV/s scan rate. The pH value was performed on PHB-3 pH Pocket Tester (Shanghai San-Xin Co, Ltd., China).

2. General procedure for the optimization of reaction conditions

2.1 Catalyst screening

Table S1 Catalyst screening [a]



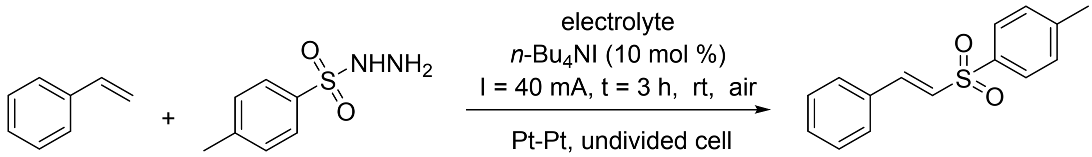
1a **2a** **3a**

Entry	Catalyst	Catalyst loading (mol %)	Yield [%] [b]
1	n-Bu_4NI	10	88
2	NH_4I	10	77
3	KI	10	75
4	I_2	5	56
5	KIO_3	10	43
6	$PhI(OAc)_2$	10	48
7	n-Bu_4NBr	10	68
8	n-Bu_4NCl	10	54
9	n-Bu_4NI	5	79

[a] Standard conditions: **1a** (0.5 mmol), **2a** (1.0 mmol), catalyst (10 mol %), sat. aq $(NH_4)_2CO_3$ (5.0 mL) as electrolyte, Pt foils (1.0 × 1.5 cm^2) as anode and cathode, undivided cell, constant current = 40 mA, 3 h, room temperature. [b] The yield of the product was determined by ^1H NMR spectroscopy.

2.2 Electrolyte screening

Table S2 Electrolyte screening [a]



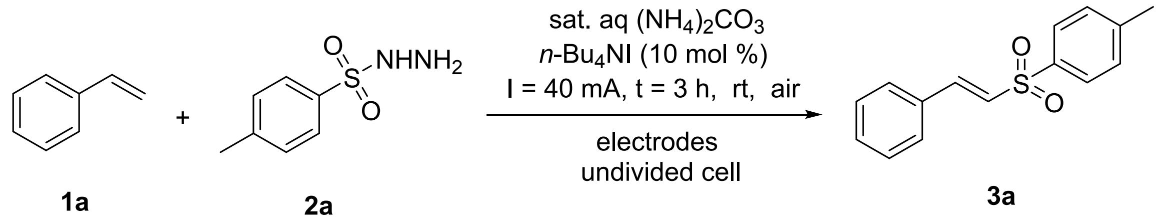
1a **2a** **3a**

Entry	Electrolyte	Yield [%] [b]
1	sat. aq $(NH_4)_2CO_3$	88
2	sat. aq NH_4HCO_3	53
3	sat. aq $(NH_4)_3PO_4$	69
4	sat. aq K_2CO_3	67
5	sat. aq CS_2CO_3	75
6	sat. aq KOH	49

[a] Standard conditions: **1a** (0.5 mmol), **2a** (1.0 mmol), n-Bu_4NI (10 mol %), electrolyte (5.0 mL), Pt foils (1.0 × 1.5 cm^2) as anode and cathode, undivided cell, constant current = 40 mA, 3 h, room temperature. [b] The yield of the product was determined by ^1H NMR spectroscopy.

2.3 Electrodes screening

Table S3 Electrodes screening [a]



Entry	Electrodes	Yield [%] [b]
1	Pt - Pt	88
2[c]	Pt - C	64
3[d]	Pt - RVC	57
4[c]	C - C	72
5[d]	RVC - RVC	70

[a] Standard conditions: **1a** (0.5 mmol), **2a** (1.0 mmol), *n*-Bu_4NI (10 mol %), sat. aq $(NH_4)_2CO_3$ (5.0 mL) as electrolyte, Pt foils (1.0 × 1.5 cm^2) as anode and cathode, undivided cell, constant current = 40 mA, 3 h, room temperature. [b] The yield of the product was determined by ^1H NMR spectroscopy. [c] Graphite rods (diameter: 0.5cm, height: 1.78 cm). [d] Reticulated vitreous carbon RVC (100 PPI, 1.5 cm × 1cm × 0.2 cm)

3. General procedure for the vinyl sulfones

Into a round bottom flask was added **1** (0.5 mmol, 1.0 equiv), **2** (1.0 mmol, 2.0 equiv), *n*-Bu_4NI (10 mol %) and sat. aq $(NH_4)_2CO_3$ (5mL).The resulting solution was electrolyzed with a pair of Pt foil (1.0×1.5 cm^2) as electrodes at constant current (40 mA) at ambient temperature for 3h (8.95 F/mol), The reaction mixture was extracted with ethyl acetate (3×15 mL). The combined organic layer was washed with brine (10 mL), dried over $MgSO_4$. The concentrated residue was purified by column chromatography on a silica gel to afford the pure product **3**.

4. The gram scale synthesis of the product 3a

Procedure for **3a**. To a round-bottomed flask (150 mL) was added **1a** (6 mmol, 1.0 equiv), **2a** (14 mmol, 2.5 equiv), *n*-Bu_4NI (10 mol %) and sat. aq $(NH_4)_2CO_3$ (80mL). The reaction flask was equipped with Pt foils as anode (2.0 × 3.0 cm^2) and cathode (2.0 × 3.0 cm^2). The solution was electrolyzed under a constant current at ambient temperature (40 mA) for 28 h (6.96 F/mol). After electrolysis, the reaction mixture was extracted with ethyl acetate (3 × 80 mL). The combined organic layer was washed with brine (80 mL) and dried over $MgSO_4$, filtered and concentrated. The resulting mixture was purified by silica gel column chromatography to afford **3a** in the yield of 72%.

5. Cyclic voltammograms

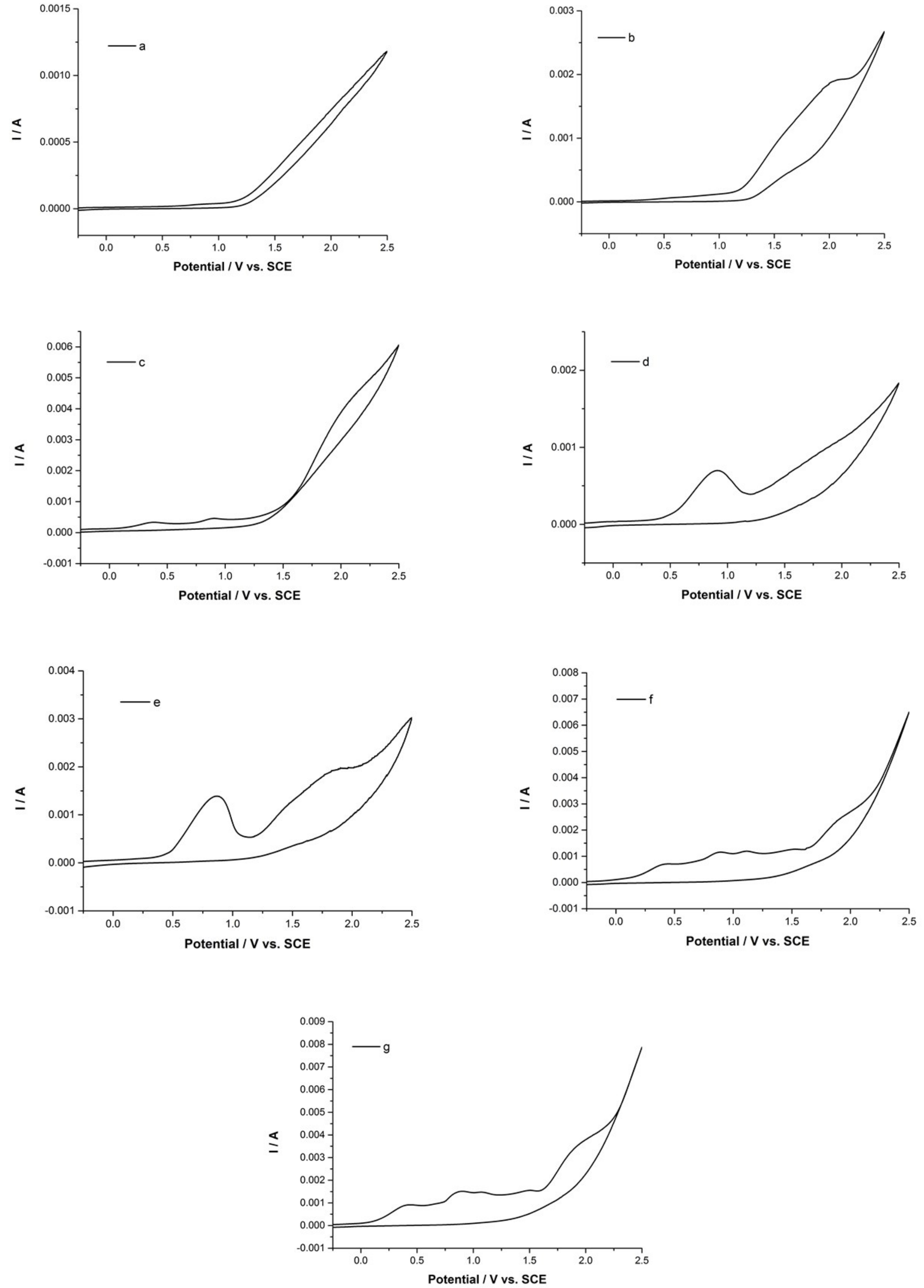
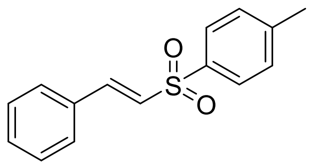


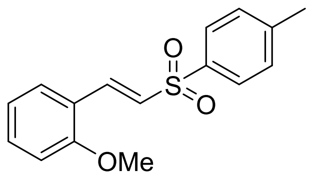
Figure 1. Cyclic voltammograms of related compounds in sat.aq $(NH_4)_2CO_3$, Using a glassy carbon electrode as working electrode (d = 3 mm), a Pt wire as counter electrode, and a saturated calomel electrode (SCE) as a reference electrode, at 100 mV/s scan rate: (a) none; (b) **1a** (0.005 M); (c) **2a** (0.005 M); (d) *n*-Bu_4NI (0.001 M); (e) **1a** (0.005 M) and *n*-Bu_4NI (0.001 M); (f) **2a** (0.005 M) and *n*-Bu_4NI (0.001 M); (g) **1a** (0.005 M), **2a** (0.005 M) and *n*-Bu_4NI (0.001 M).

6. Spectroscopic data of products



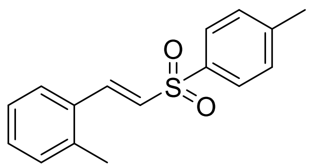
3a

(*E*)-1-Methyl-4-(styrylsulfonyl)benzene (3a).[1] The crude product was purified by column chromatography on silica gel to give **3a** as a white solid (105.9 mg, 82% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.0 Hz, 1H), 7.65 (d, J = 16.0 Hz, 1H), 7.47 – 7.46 (m, 2H), 7.39 –7.33 (m, 5H), 6.86 (d, J = 16.0 Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.50, 142.03, 137.86, 132.55, 131.21, 130.08, 129.16, 128.62, 127.80, 127.76, 21.70.



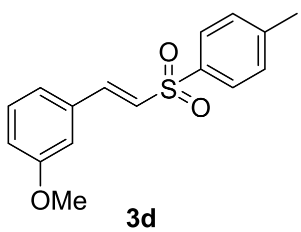
3b

(*E*)-1-methoxy-2-(2-tosylvinyl)benzene (3b).[2] The crude product was purified by column chromatography on silica gel to give **3b** as a colourless oil (116.8 mg, 81% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.81 (m, 3H), 7.41 – 7.31 (m, 4H), 7.06 (d, J = 16.0 Hz, 1H), 6.96 – 6.90 (m, 2H), 3.86 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.93, 144.16, 138.40, 138.13, 132.51, 130.81, 129.98, 128.36, 127.76, 121.42, 120.89, 111.38, 55.62, 21.70.



3c

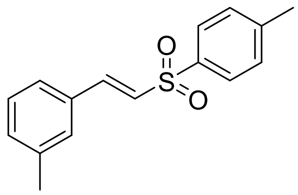
(*E*)-1-methyl-2-(2-tosylvinyl)benzene (3c).[1] The crude product was purified by column chromatography on silica gel to give **3c** as a pale yellow solid solid (106.3 mg, 78% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 12.0 Hz, 1H), 7.79 (d, J = 8.0 Hz, 2H), 7.38 (d, J = 8.0 Hz, 1H), 7.30 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.0 Hz, 1H), 7.17 – 7.11 (m, 2H), 6.74 (d, J = 16.0 Hz, 1H), 2.40 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.45, 139.68, 138.19, 137.88, 131.44, 131.11, 130.92, 130.07, 128.67, 127.79, 126.94, 126.56, 21.69, 19.83.



3d

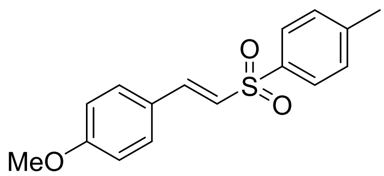
(*E*)-1-methoxy-3-(2-tosylvinyl)benzene (3d).[2] The crude product was purified by column chromatography on silica gel to give **3d** as a colourless oil (115.4 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 12.0 Hz, 2H), 7.62 (d, J = 16.0 Hz, 1H), 7.34 (d, J = 8.0 Hz, 2H), 7.29 – 7.27 (m, 1H), 7.06 (d, J = 8.0 Hz, 1H), 6.96 – 6.93 (m, 2H), 6.84 (d, J = 16.0 Hz, 1H), 3.80 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.12, 144.56, 142.02, 137.88, 133.91, 130.22, 130.12, 128.06, 127.86, 121.30, 117.17, 113.47, 55.49, 21.75.



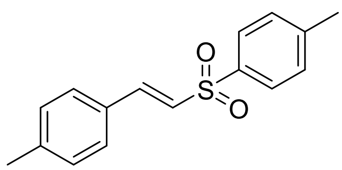
3e

(*E*)-1-methyl-3-(2-tosylvinyl)benzene (3e).[2] The crude product was purified by column chromatography on silica gel to give **3e** as a pale yellow solid (102.2 mg, 75% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.0 Hz, 2H), 7.64 (d, J = 12.0 Hz, 1H), 7.35 (d, J = 8.0 Hz, 2H), 7.29 – 7.28 (m, 3H), 7.24 – 7.22 (m, 1H), 6.87 (d, J = 16.0 Hz, 1H), 2.44 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.42, 142.21, 138.89, 137.99, 132.51, 132.03, 130.05, 129.19, 129.04, 127.78, 127.52, 125.86, 21.69, 21.34.



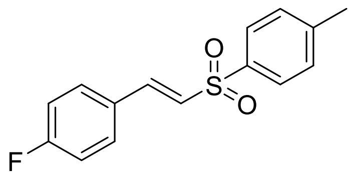
3f

(*E*)-1-methoxy-4-(2-tosylvinyl)benzene (3f).[1] The crude product was purified by column chromatography on silica gel to give **3f** as a white solid (126.9 mg, 88% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.0 Hz, 2H), 7.59 (d, J = 16.0 Hz, 1H), 7.40 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 6.87 (d, J = 12.0 Hz, 2H), 6.71 (d, J = 12.0 Hz, 1H), 3.80 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.07, 144.20, 141.81, 138.28, 130.38, 129.97, 127.61, 125.11, 124.91, 114.57, 55.50, 21.64.



3g

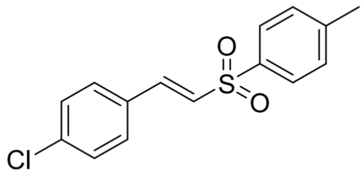
(*E*)-1-methyl-4-((4-methylstyryl)sulfonyl)benzene (3g).[1] The crude product was purified by column chromatography on silica gel to give **3g** as a white solid (113.1 mg, 83% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.0 Hz, 2H), 7.63 (d, J = 160 Hz, 1H), 7.37 – 7.32 (m, 4H), 7.18 (d, J = 8.0 Hz, 2H), 6.80 (d, J = 16.0 Hz, 1H), 2.43 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.38, 142.13, 141.85, 138.12, 130.06, 129.91, 129.85, 128.66, 127.78, 126.58, 21.72, 21.63.



3h

(*E*)-1-fluoro-4-(2-tosylvinyl)benzene (3h).[2] The crude product was purified by column chromatography on silica gel to give **3h** as a colourless oil (92.6 mg, 67% yield).

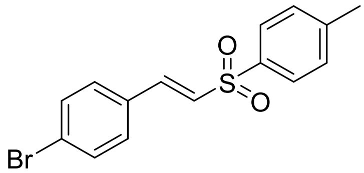
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 16.0 Hz, 1H), 7.48 – 7.45 (m, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.08 – 7.04 (m, 2H), 6.79 (d, J = 16.0 Hz, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.42 (d, $J_{C\text{-}F}$ = 251.0 Hz), 144.60, 140.73, 137.81, 130.68 (d, $J_{C\text{-}F}$ = 9.0 Hz), 130.13, 128.87 (d, $J_{C\text{-}F}$ = 3.0 Hz), 127.83, 127.58 (d, $J_{C\text{-}F}$ = 2.0 Hz), 116.41 (d, $J_{C\text{-}F}$ = 2.2 Hz), 21.72.



3i

(*E*)-1-chloro-4-(2-tosylvinyl)benzene (3i).[1] The crude product was purified by column chromatography on silica gel to give **3i** as a white solid (109.8 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.60 (d, J = 16.0 Hz, 1H), 7.42 – 7.34 (m, 6H), 6.83 (d, J = 16.0 Hz, 1H), 2.43 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.73, 140.58, 137.68, 137.28, 131.13, 130.19, 129.85, 129.53, 128.44, 127.92, 21.78.

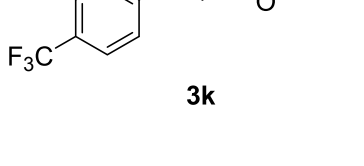


3j

(*E*)-1-bromo-4-(2-tosylvinyl)benzene (3j). [2]

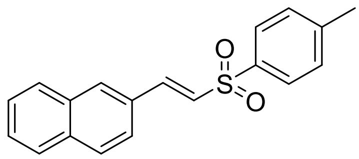
The crude product was purified by column chromatography on silica gel to give **3j** as a white solid (118.1 mg, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.59 (d, J = 16.0 Hz, 1H), 7.52 (d, J = 8.0 Hz, 2H), 7.34 (t, J = 8.0 Hz, 4H), 6.85 (d, J = 12.0 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.76, 140.66, 137.67, 132.52, 131.58, 130.21, 130.03, 128.57, 127.95, 125.69, 21.80



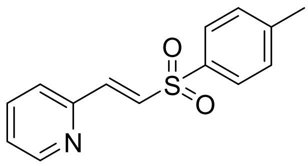
3k

(*E*)-1-methyl-4-((4-(trifluoromethyl)styryl)sulfonyl)benzene (3k).[1] The crude product was purified by column chromatography on silica gel to give **3k** as a white solid (89.8mg, 55% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8.0 Hz, 2H), 7.67 (d, J = 16.0 Hz, 1H), 7.65 – 7.57 (m, 4H), 7.35 (d, J = 8.0 Hz, 2H), 6.97 (d, J = 16.0 Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.93, 139.99, 137.30, 136.01, 132.55 (q, $J_{C\text{-}F}$ = 32.0 Hz), 130.56, 130.21, 128.82, 127.96, 126.09 (q, $J_{C\text{-}F}$ = 4.0 Hz), 123.75 (q, $J_{C\text{-}F}$ = 271.0 Hz).



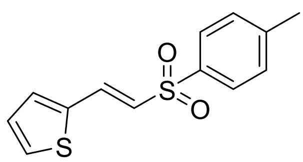
3l

(*E*)-2-(2-tosylvinyl)naphthalene (3l).[2] The crude product was purified by column chromatography on silica gel to give **3l** as a yellow solid (114.1 mg, 74% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.80 (m, 7H), 7.55 – 7.52 (m, 3H), 7.35 (d, J = 8.0 Hz, 2H), 6.96 (d, J = 16.0 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.55, 142.15, 137.98, 134.60, 133.26, 130.98, 130.14, 130.05, 129.08, 128.81, 127.96, 127.89, 127.81, 127.12, 123.58, 21.77.



3m

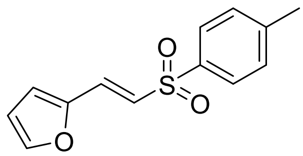
(*E*)-2-(2-tosylvinyl)pyridine (3m).[1] The crude product was purified by column chromatography on silica gel to give **3m** as a yellow oil (75.2mg, 58% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 2H), 7.75 – 7.71 (m, 1H), 7.62 (d, J = 16.0 Hz, 1H), 7.44 (d, J = 16.0 Hz, 1H), 7.40 (d, J = 4.0 Hz, 1H), 7.34 (d, J = 8.0 Hz, 2H), 7.30 – 7.27 (m, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.20, 150.36, 144.72, 140.17, 137.38, 137.18, 132.29, 130.10, 128.02, 125.46, 125.06, 21.71.



3n

(***E*)-2-(2-tosylvinyl)thiophene (3n).**[2] The crude product was purified by column chromatography on silica gel to give **3n** as a brown solid (80.7 mg, 61% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 16.0 Hz, 1H), 7.41 (d, J = 4.0 Hz, 1H), 7.33 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 12.0 Hz, 1H), 7.06 – 7.04 (m, 1H), 6.63 (d, J = 16.0 Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.50, 138.03, 137.18, 134.73, 132.39,

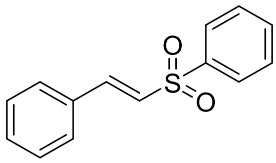
130.11, 129.98, 128.44, 127.77, 125.97, 21.73.



3o

(*E*)-2-(2-tosylvinyl)furan (3o).[2] The crude product was purified by column chromatography on silica gel to give **3o** as a white solid (84.5 mg, 68% yield).

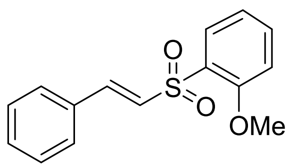
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.0 Hz, 2H), 7.46 (s, 1H), 7.40 (d, *J* = 16.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.73 (d, *J* = 12.0 Hz, 1H), 6.68 (d, *J* = 4.0 Hz, 1H), 6.47 – 6.46 (m, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.92, 145.69, 144.45, 138.07, 130.08, 128.56, 127.74, 125.28, 116.83, 112.70, 21.73.



4a

(*E*)-(2-(phenylsulfonyl)vinyl)benzene (4a).[3] The crude product was purified by column chromatography on silica gel to give **4a** as a pale yellow solid (99.0 mg, 81% yield).

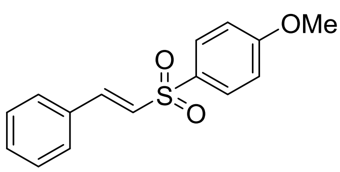
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.94 (m, 2H), 7.69 (d, *J* = 16.0 Hz, 1H), 7.62 – 7.60 (m, 1H), 7.56 – 7.53 (m, 2H), 7.49 – 7.47 (m, 2H), 7.41 – 7.38 (m, 3H), 6.87 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.62, 140.85, 133.52, 132.47, 131.35, 129.47, 129.21, 128.70, 127.76, 127.43.



4b

(*E*)-1-methoxy-2-(styrylsulfonyl)benzene (4b).[2] The crude product was purified by column chromatography on silica gel to give **4b** as a yellow oil (105.6 mg, 77% yield).

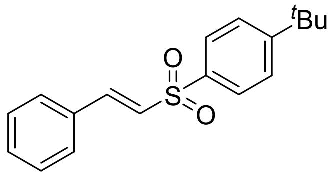
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 – 8.02 (m, 1H), 7.69 (d, *J* = 12.0 Hz, 1H), 7.58 – 7.54 (m, 1H), 7.50 – 7.49 (m, 2H), 7.40 – 7.39 (m, 3H), 7.16 – 7.08 (m, 2H), 7.01 (d, *J* = 8.0 Hz, 1H), 3.96 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.48, 143.43, 135.50, 132.98, 131.09, 129.60, 129.17, 128.80, 128.60, 127.12, 120.86, 112.57, 56.42.



4c

(*E*)-1-methoxy-4-(styrylsulfonyl)benzene (4c).[3] The crude product was purified by column chromatography on silica gel to give **4c** as a white oil (118.0 mg, 86% yield).

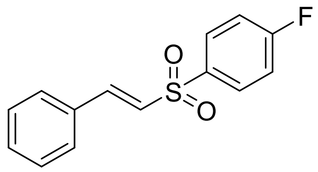
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 16.0 Hz, 1H), 7.46 – 7.44 (m, 2H), 7.38 – 7.35 (m, 3H), 7.00 (d, *J* = 12.0 Hz, 2H), 6.89 – 6.84 (m, 1H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.67, 141.43, 132.58, 132.28, 131.09, 129.95, 129.12, 128.55, 128.08, 114.67, 55.77.



4d

(*E*)-1-*tert*-butyl-4-(styrylsulfonyl)benzene (4d). [3] The crude product was purified by column chromatography on silica gel to give **4d** as a white solid (103.7 mg, 69% yield).

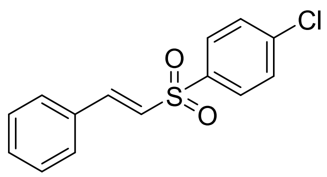
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 16.0 Hz, 1H), 7.55 (d, *J* = 8.0Hz, 2H), 7.47 (d, *J* = 4.0 Hz, 2H), 7.39 – 7.37 (m, 3H), 6.87 (d, *J* = 16.0 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.46, 142.09, 137.75, 132.57, 131.23, 129.18, 128.66, 127.74, 127.66, 126.50, 35.35, 31.18.



4e

(*E*)-1-fluoro-4-(styrylsulfonyl)benzene (4e). [3] The crude product was purified by column chromatography on silica gel to give **4e** as a pale yellow solid (85.3 mg, 65% yield).

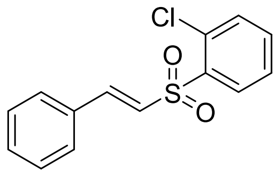
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.95 (m, 2H), 7.68 (d, *J* = 16.0 Hz, 1H), 7.49 – 7.47 (m, 2H), 7.43 – 7.37 (m, 3H), 7.26 – 7.19 (m, 2H), 6.86 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.74 (d, $J_{C\text{-}F}$ = 255.0 Hz), 142.82, 136.93 (d, $J_{C\text{-}F}$ = 3.0 Hz), 132.34, 131.47, 130.64 (d, *J* = 10.0 Hz), 129.25, 128.74, 127.24, 116.77 (d, *J* = 23.0 Hz).



4f

(***E*)-1-chloro-4-(styrylsulfonyl)benzene (4f).** [3] The crude product was purified by column chromatography on silica gel to give **4f** as a brown solid (101.8 mg, 73% yield).

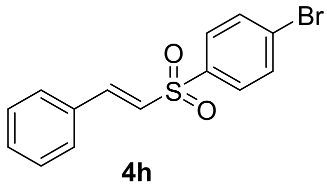
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.0 Hz, 2H), 7.59 (d, *J* = 16.0 Hz, 1H), 7.41 – 7.37 (m, 4H), 7.31 – 7.28 (m, 3H), 6.73 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.10, 140.07, 139.35, 132.23, 131.46, 129.70, 129.20, 129.18, 128.71, 126.95.



4g

(*E*)-1-chloro-2-(styrylsulfonyl)benzene (4g). [2] The crude product was purified by column chromatography on silica gel to give **4g** as a pale yellow solid (89.2 mg, 64% yield).

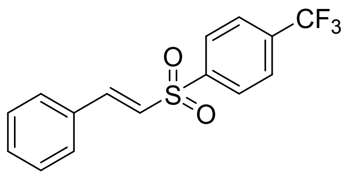
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.0 Hz, 1H), 7.77 (d, J = 16.0 Hz, 1H), 7.53 – 7.39 (m, 8H), 7.08 (d, J = 12.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.44, 138.43, 134.66, 132.95, 132.50, 132.03, 131.57, 130.85, 129.27, 128.85, 127.60, 125.47.



4h

(*E*)-1-bromo-4-(styrylsulfonyl)benzene (4h). [3] The crude product was purified by column chromatography on silica gel to give **4h** as a white solid (109.9 mg, 68% yield).

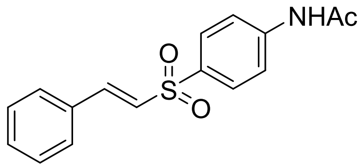
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 8.0 Hz, 2H), 8.09 – 8.05 (m, 3H), 7.86 (d, J = 8.0 Hz, 2H), 7.81 – 7.76 (m, 3H), 7.22 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.25, 139.96, 132.80, 132.32, 131.57, 129.36, 129.29, 128.79, 126.97.



4i

(*E*)-1-(styrylsulfonyl)-4-(trifluoromethyl)benzene (4i). [3] The crude product was purified by column chromatography on silica gel to give **4i** as a pale yellow solid (95.3 mg, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.0 Hz, 2H), 7.81 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 16.0 Hz, 1H), 7.51 – 7.49 (m, 2H), 7.43 – 7.38 (m, 3H), 6.88 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.54 (d, $J_{C\text{-}F}$ = 1.0 Hz), 144.20, 135.12 (q, $J_{C\text{-}F}$ = 33.0 Hz), 132.16, 131.75, 129.30, 128.87, 128.37, 126.61 (q, $J_{C\text{-}F}$ = 3.0 Hz), 126.43, 123.30 (q, J = 272.0 Hz).

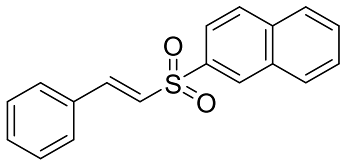


4j

(*E*)-*N*-(4-(styrylsulfonyl)phenyl)acetamide (4j). [3]

The crude product was purified by column chromatography on silica gel to give **4j** as a white solid (85.9 mg, 57% yield).

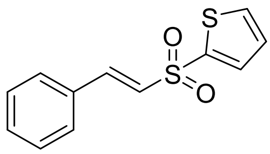
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 16.0 Hz, 1H), 7.47 (d, *J* = 4.0 Hz, 2H), 7.39 (d, *J* = 4.0 Hz, 3H), 6.85 (d, *J* = 12.0 Hz, 1H), 2.19 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.62, 143.25, 142.39, 134.75, 132.31, 131.43, 129.23, 128.93, 128.71, 127.28, 119.85, 24.73.



4k

(*E*)-2-(styrylsulfonyl)naphthalene (**4k**). [3] The crude product was purified by column chromatography on silica gel to give **4k** as a white solid (103.1 mg, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.00– 7.97 (m, 2H), 7.91 – 7.88 (m, 2H), 7.75 (d, *J* = 16.0 Hz, 1H), 7.67 – 7.59 (m, 2H), 7.50 – 7.47 (m, 2H), 7.40 – 7.35 (m, 3H), 6.94 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.70, 137.66, 135.29, 132.52, 132.44, 131.34, 129.81, 129.51, 129.36, 129.32, 129.21, 128.72, 128.10, 127.79, 127.49, 122.69.



4l

(*E*)-2-(styrylsulfonyl)thiophene (**4l**).[3] The crude product was purified by column chromatography on silica gel to give **4l** as a yellow solid (85.1 mg, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.66 (m, 3H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.43 – 7.38 (m, 3H), 7.14 (t, *J* = 4.0 Hz, 1H), 6.97 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.41, 142.33, 134.08, 133.63, 132.41, 131.46, 129.27, 128.78, 128.19, 128.05.

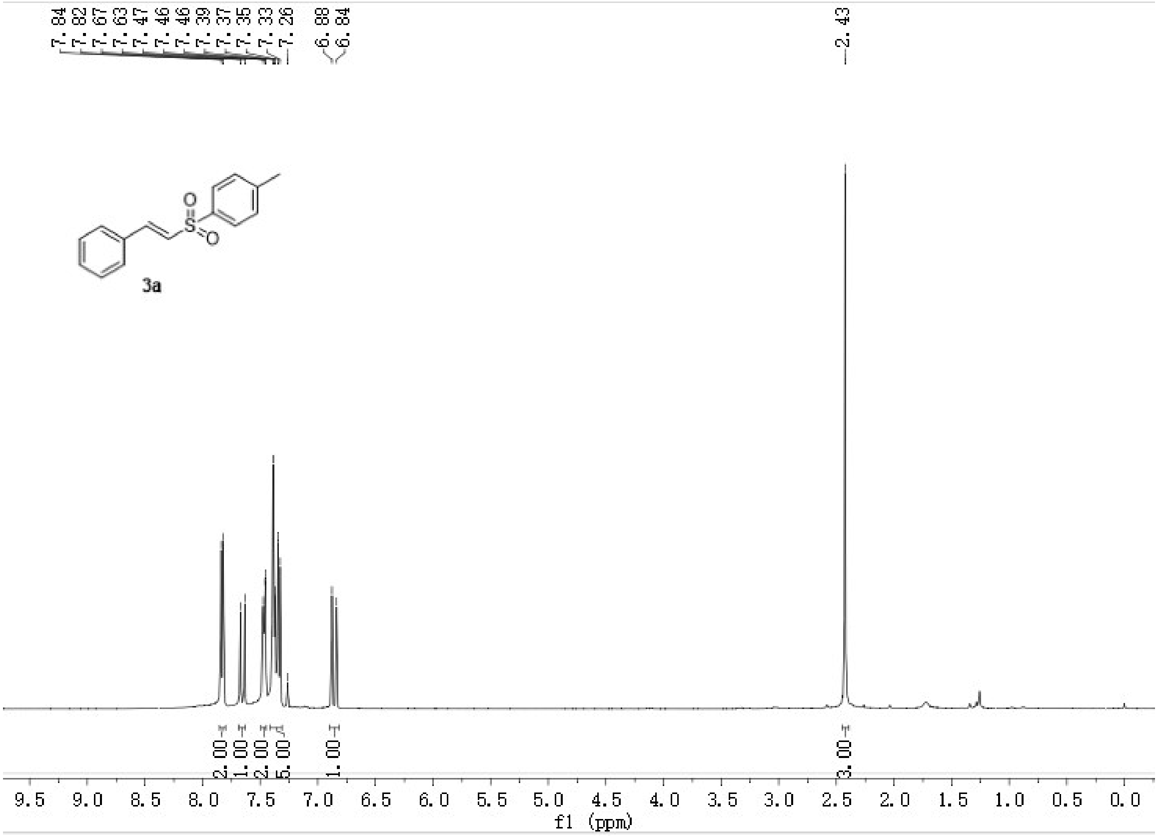
7. References

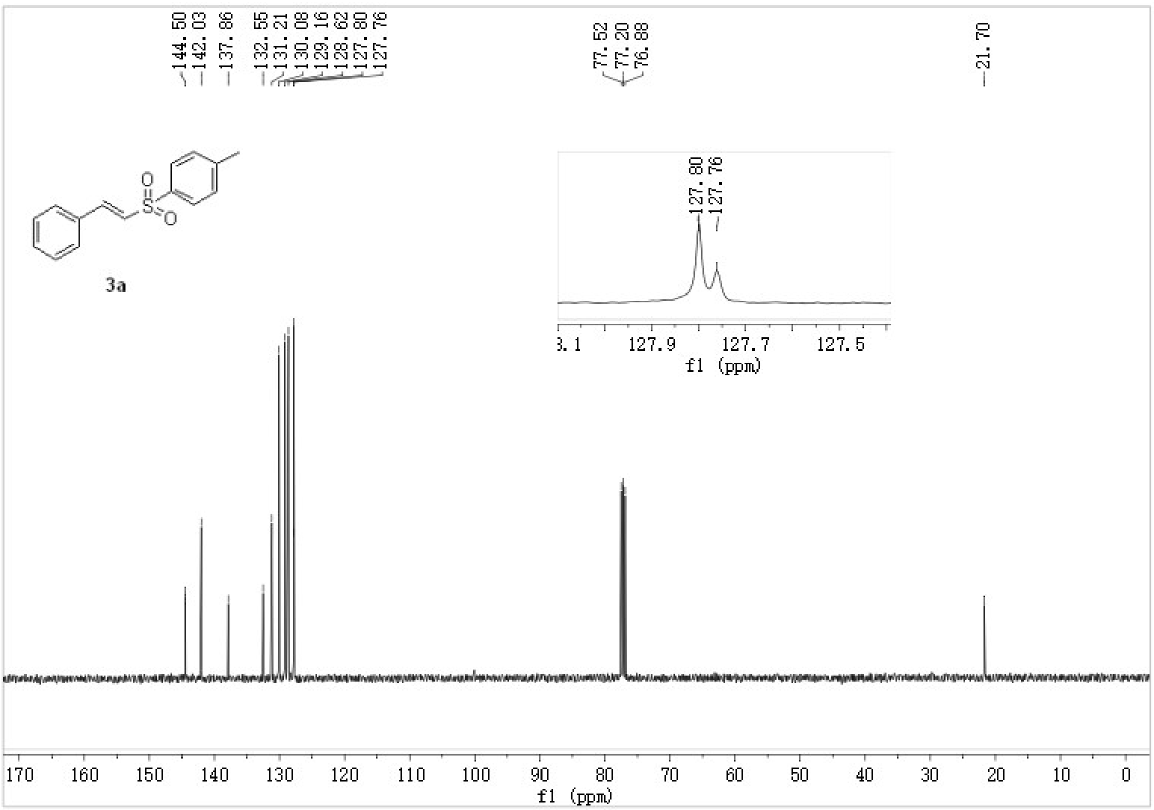
1. M. Pramanik, K. Choudhuri, P. Mal, *Asian J. Org. Chem.*, 2019, **8**, 144.

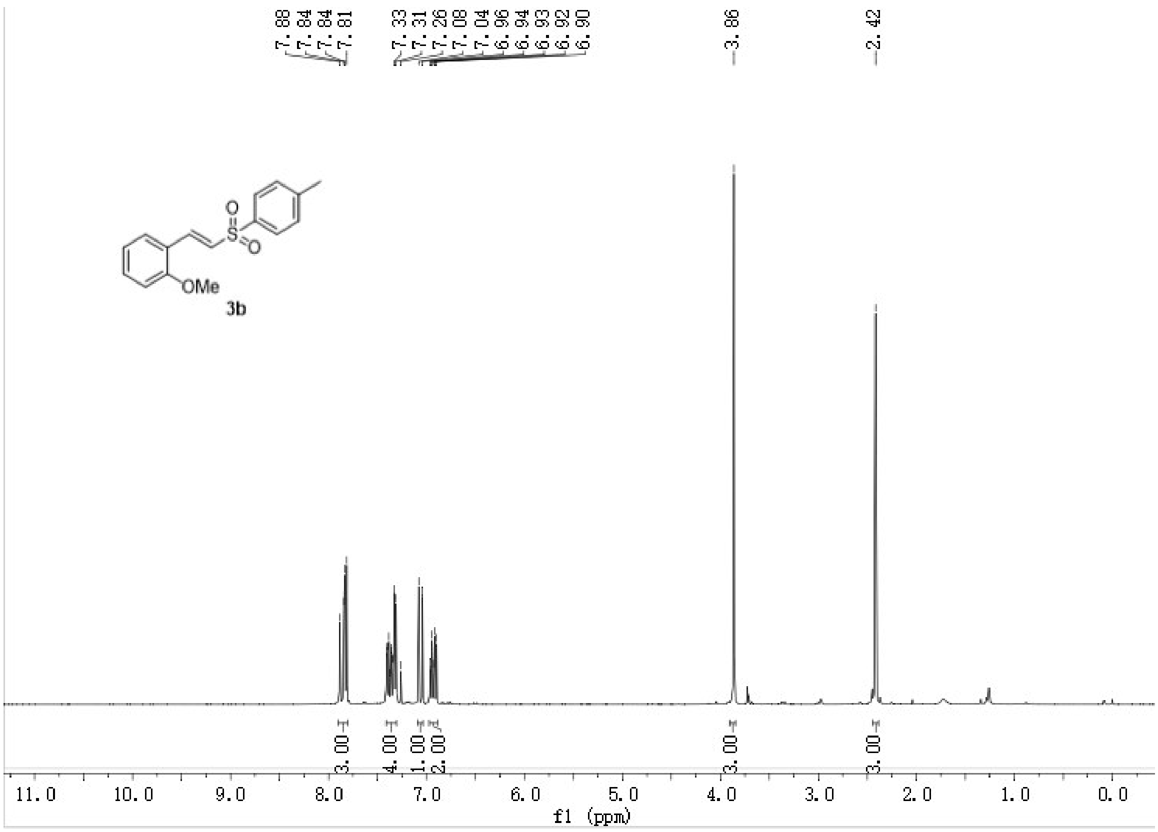
2. Y. Zhao, Y.-L. Lai, K.-S. Du, D.-Z. Lin, J.-M. Huang, *J. Org. Chem.,* 2017, **82**, 9655.

3. S. Cai, Y. Xu, D. Chen, L. Li, Q. Chen, M. Huang, W. Weng, *Org. Lett.,* 2016, **18**, 2990.

8. Copies of NMR spectra of products



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4.00
1.00
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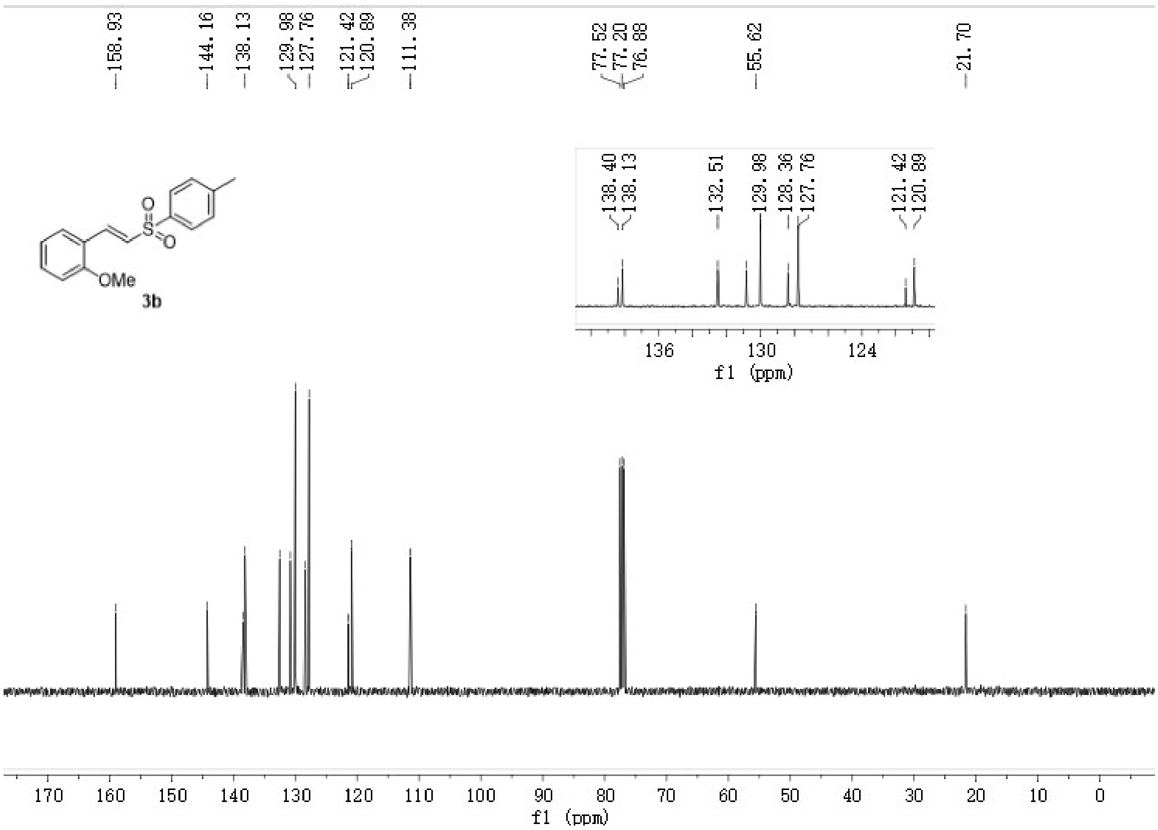
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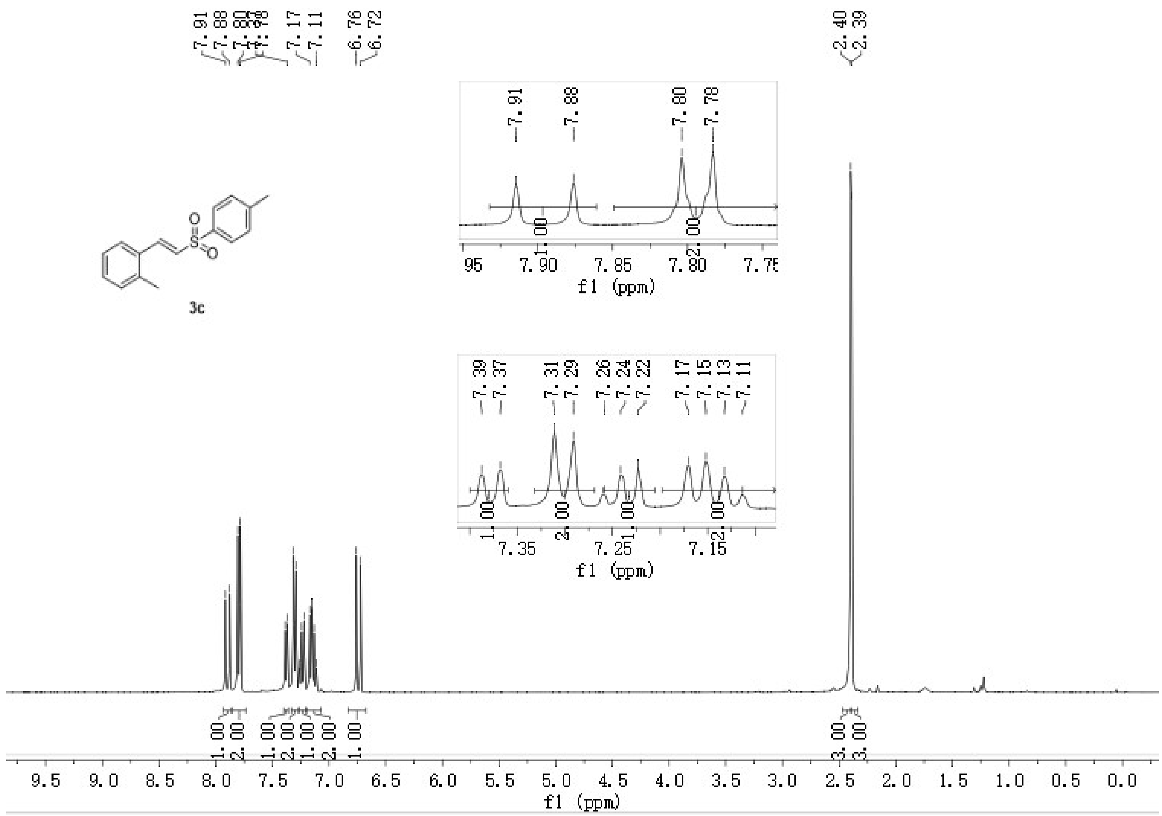
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3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
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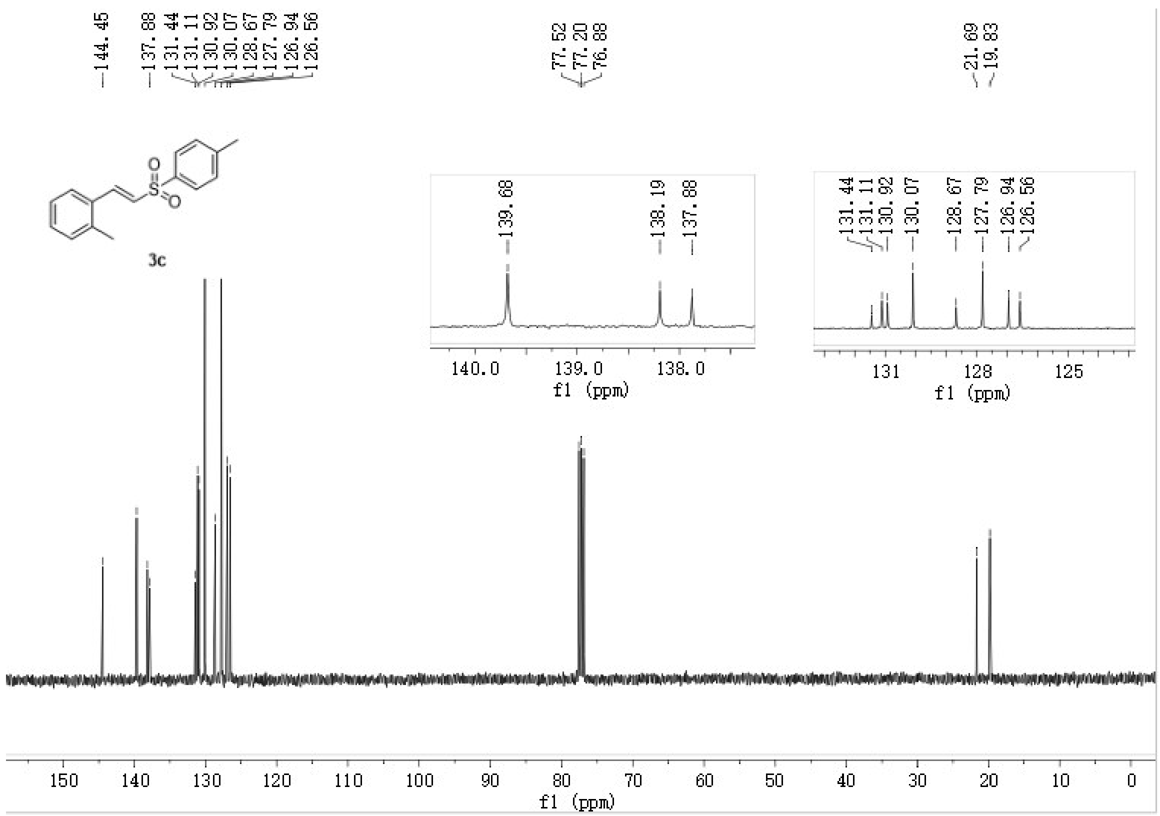
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19.83
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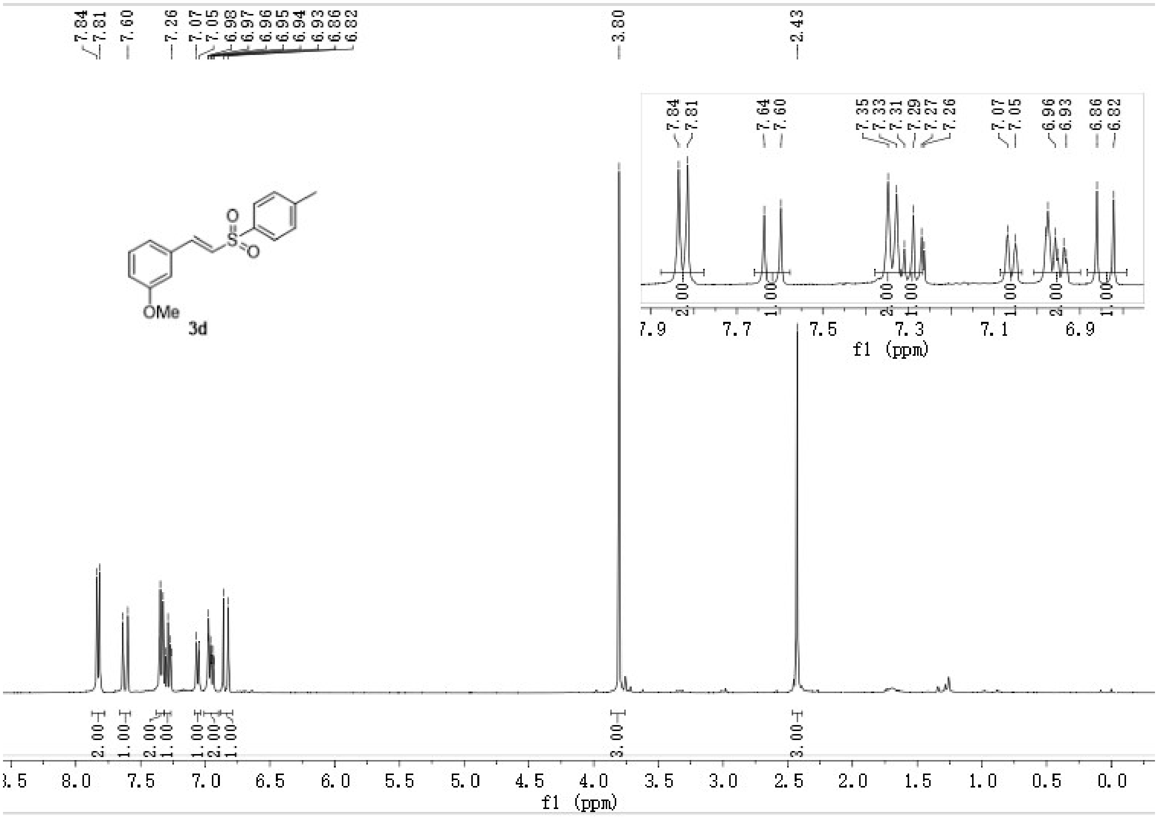

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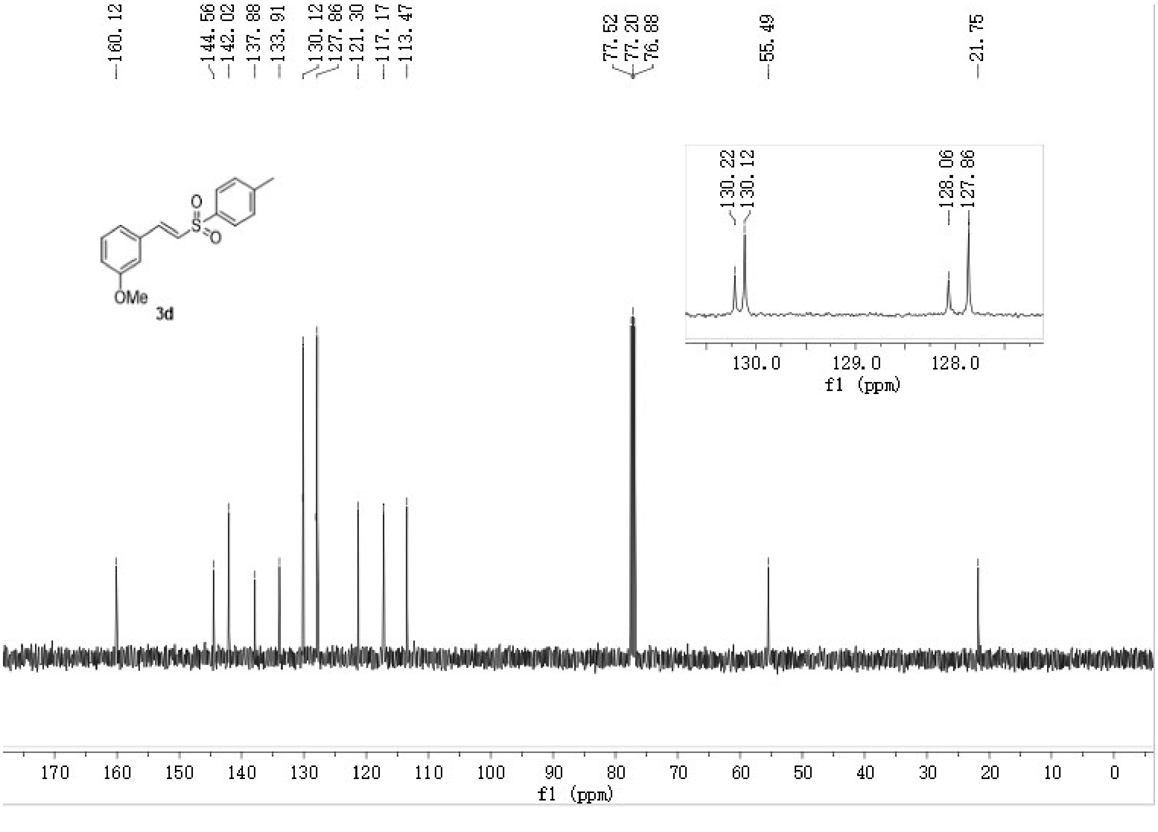
f1 (ppm)



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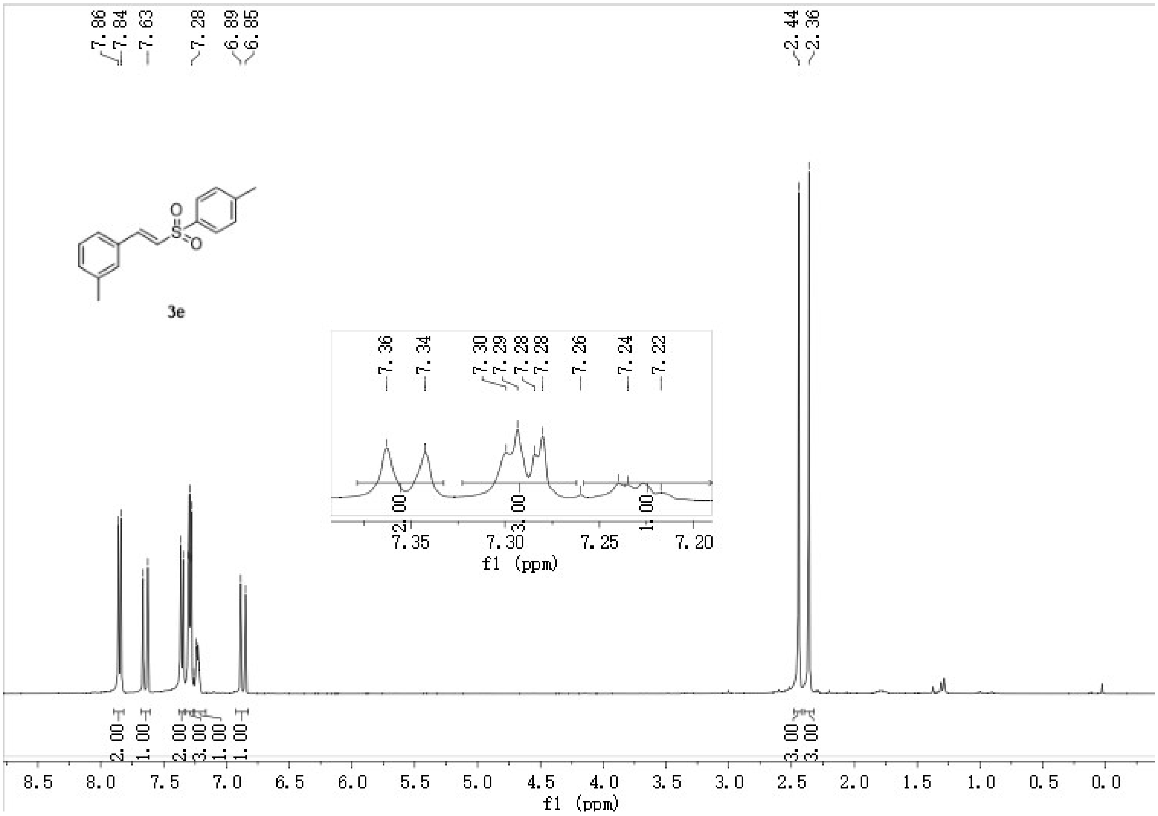
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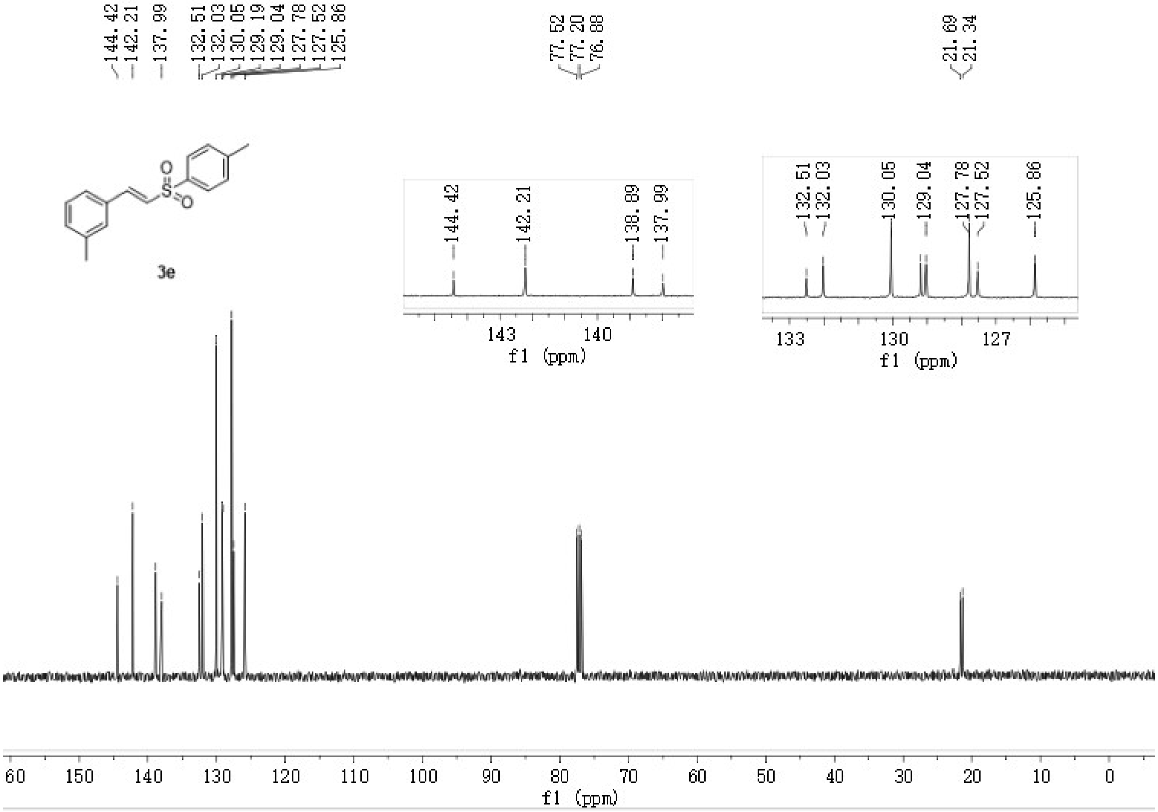
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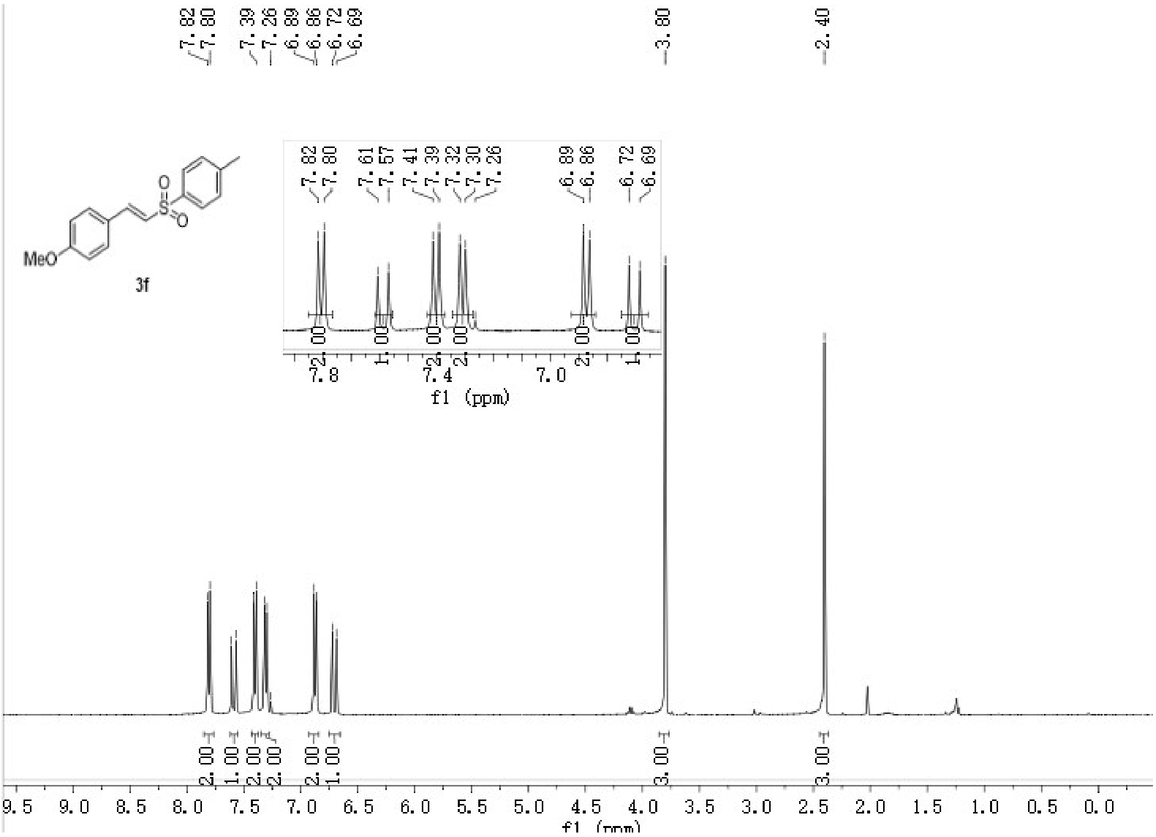
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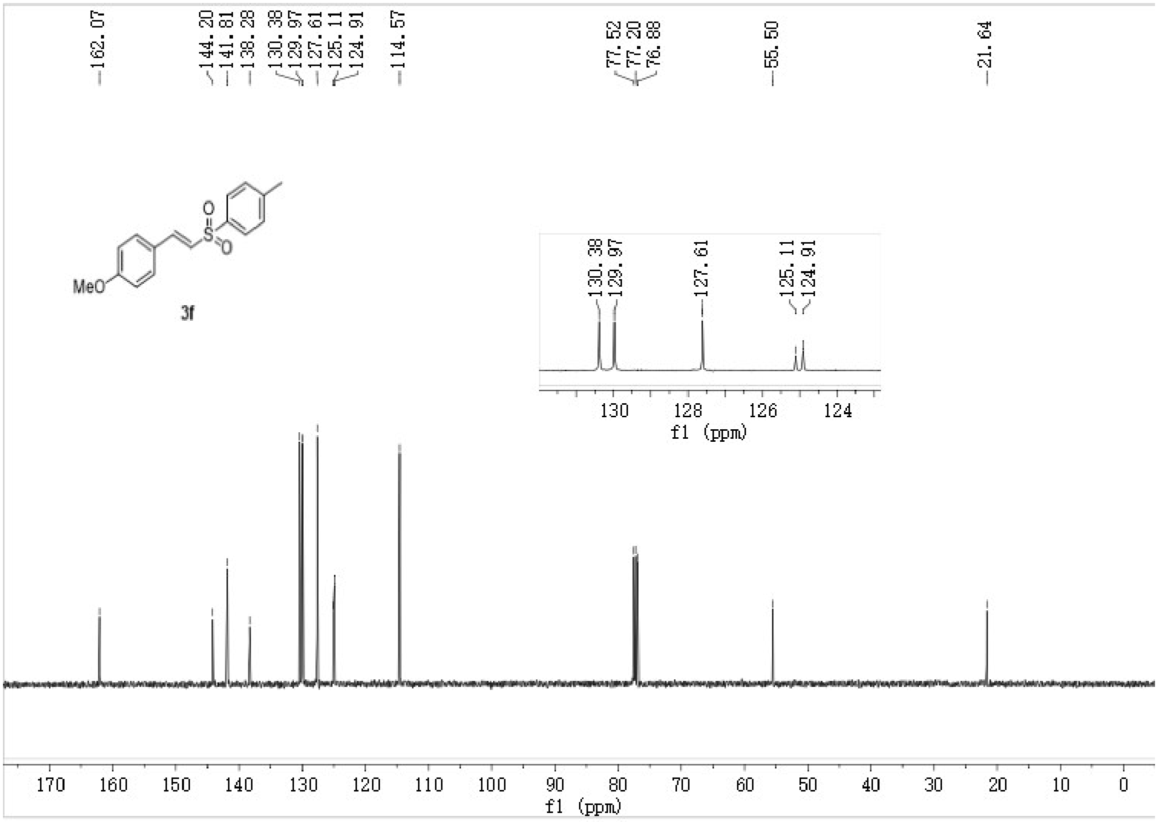
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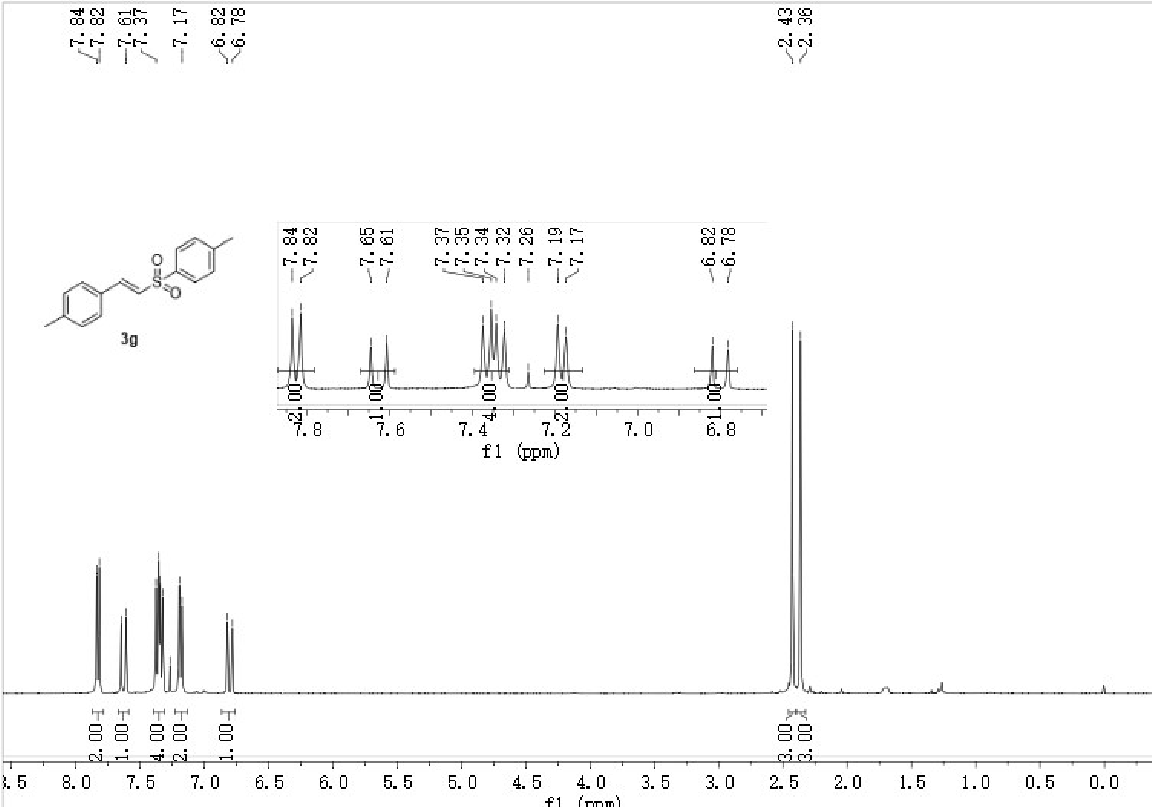
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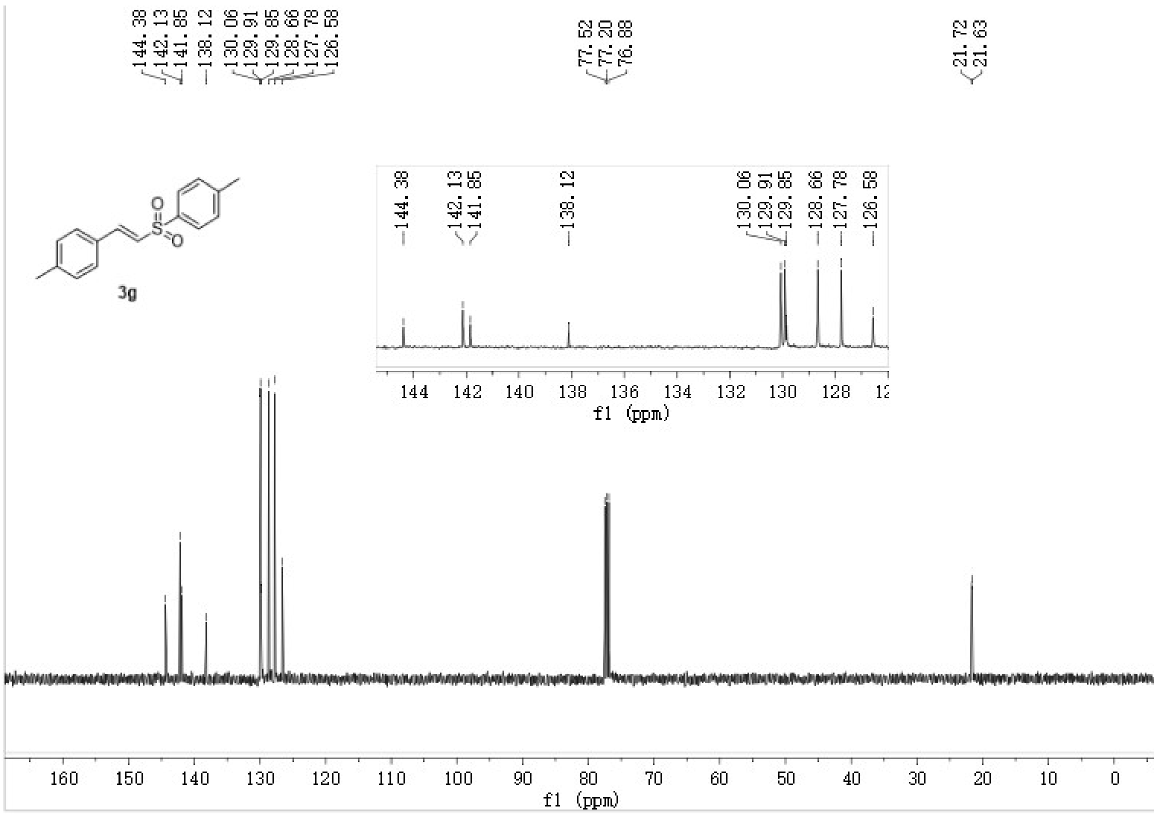
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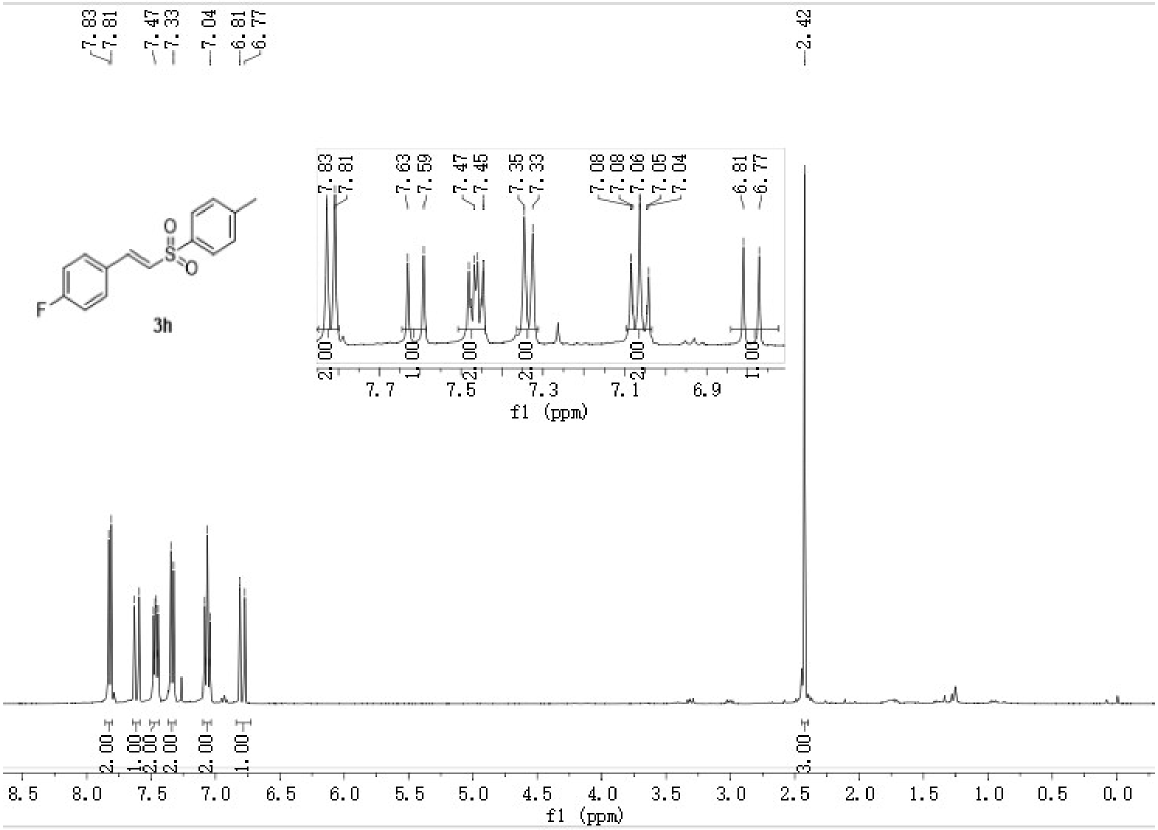
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1.0
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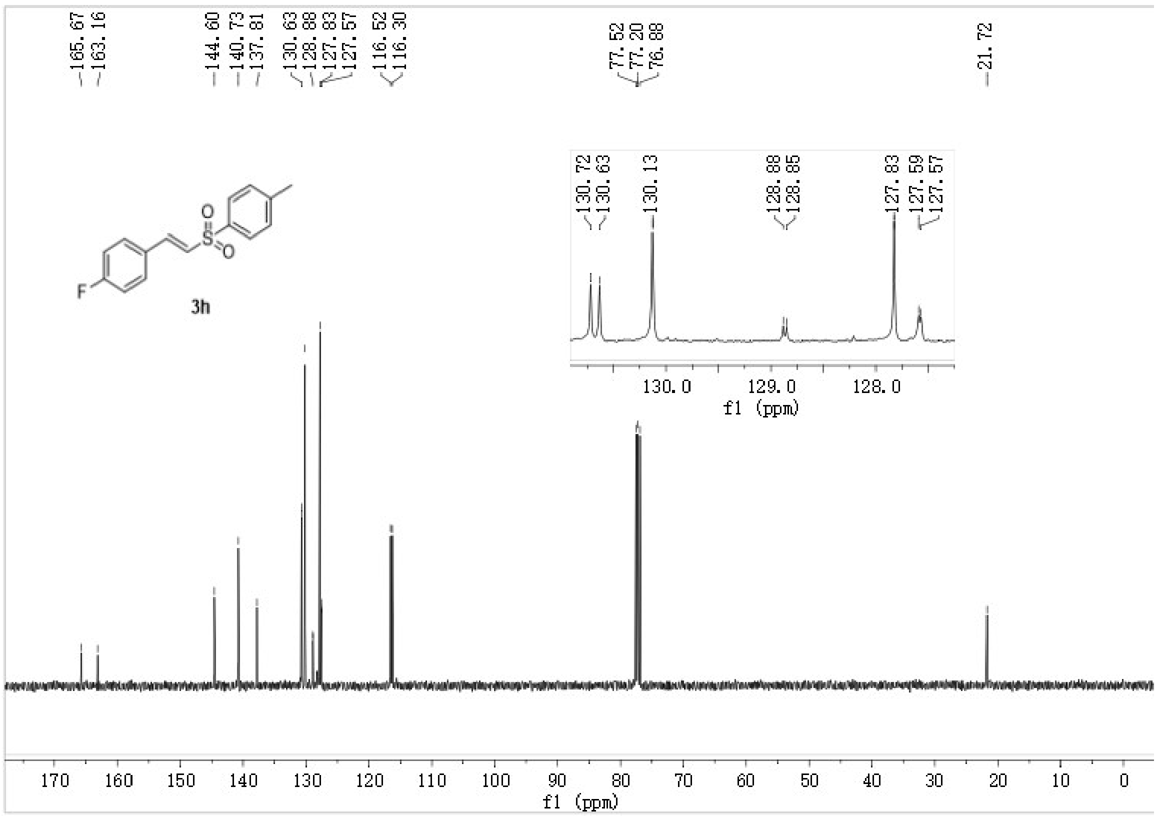
0.0
f1 (ppm)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128.88
127.83
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116.30
77.52
77.20
76.88
21.72
130.72
130.63
130.13
128.88
128.85
127.83
127.59
127.57
F
3h
130.0
129.0
128.0
f1 (ppm)
170
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140
130
120
110
100
90
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70
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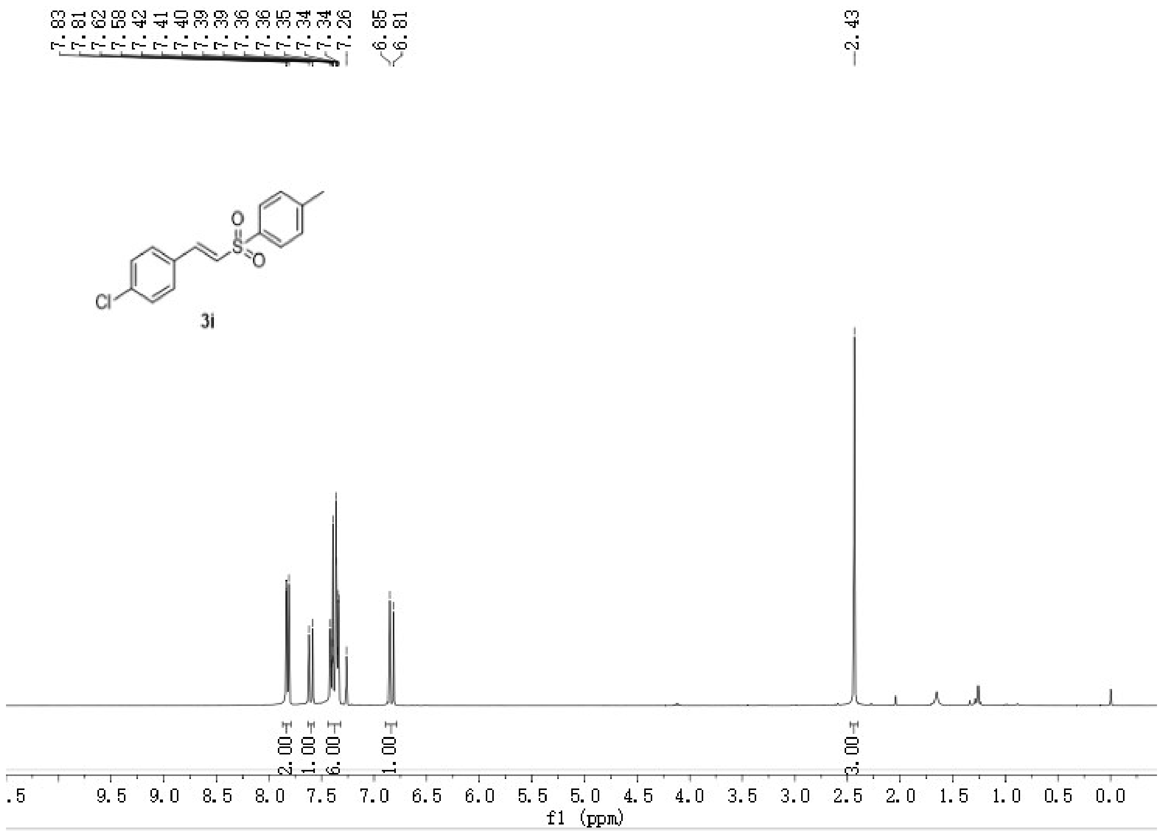
0
f1 (ppm)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7.41
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7.39
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7.36
7.36
7.35
7.34
7.34
7.26
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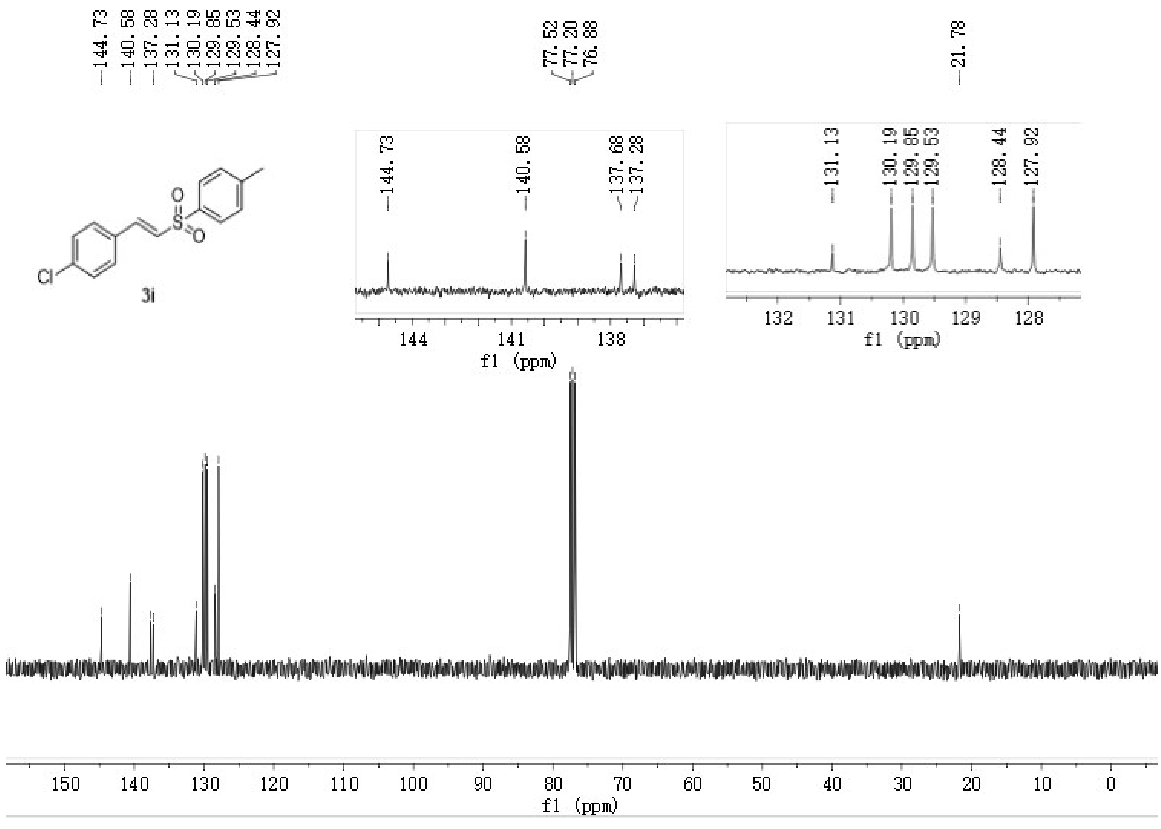
S
O
O
3i
2.00
1.00
6.00
1.00
3.00
f1 (ppm)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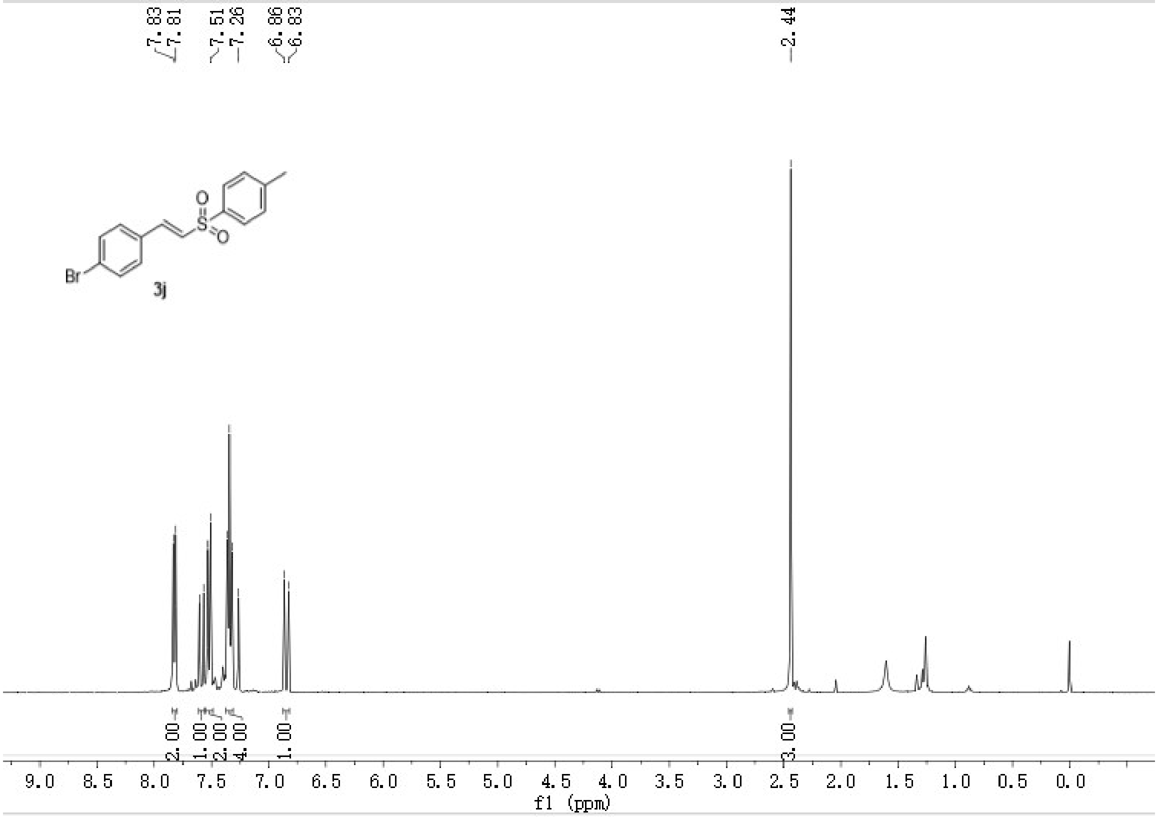
128.44
127.92
77.52
77.20
76.88
21.78
Cl
S
O
O
3i
144.73
140.58
137.68
137.28
f1 (ppm)
131.13
130.19
129.85
129.53
128.44
127.92
f1 (ppm)
f1 (ppm)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6.83
2.44
Br
3j
2.00
1.00
2.00
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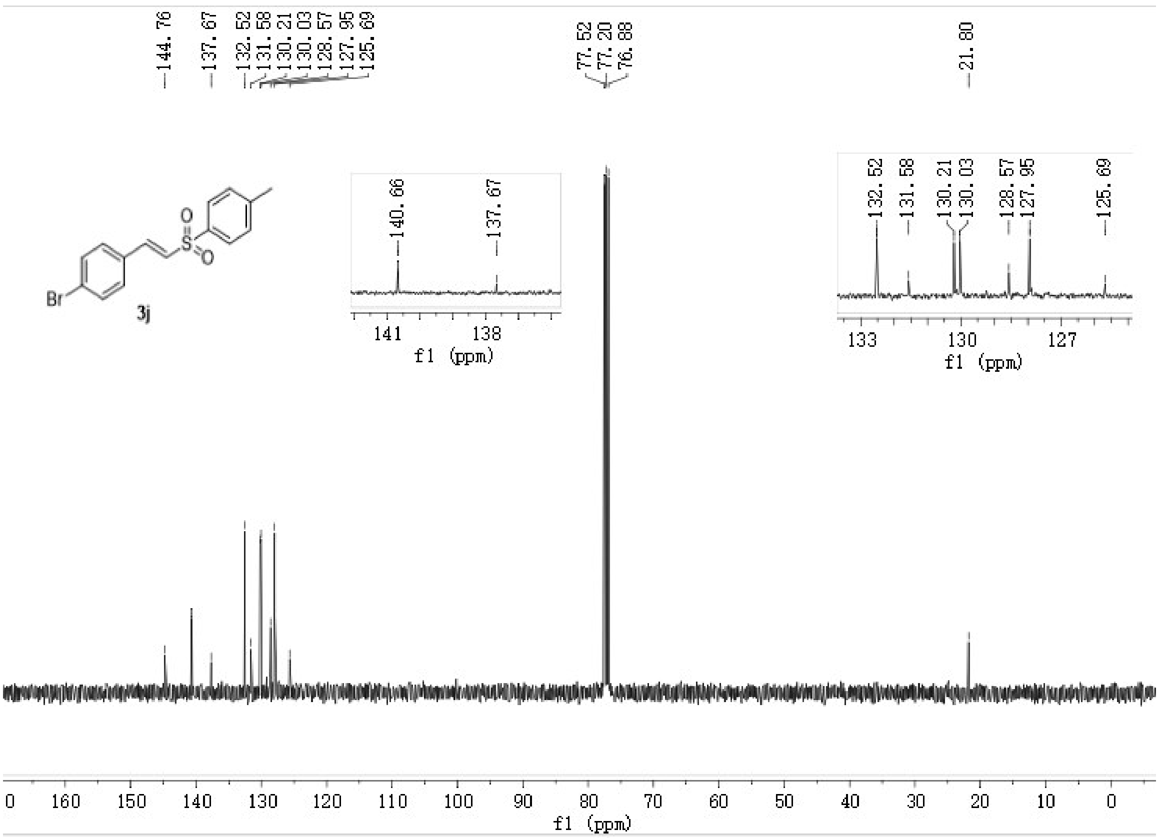
1.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.52
77.20
76.88
21.80
Br
3j
140.66
137.67
141
138
f1 (ppm)
132.52
131.58
130.21
130.03
128.57
127.95
125.69
133
130
127
f1 (ppm)
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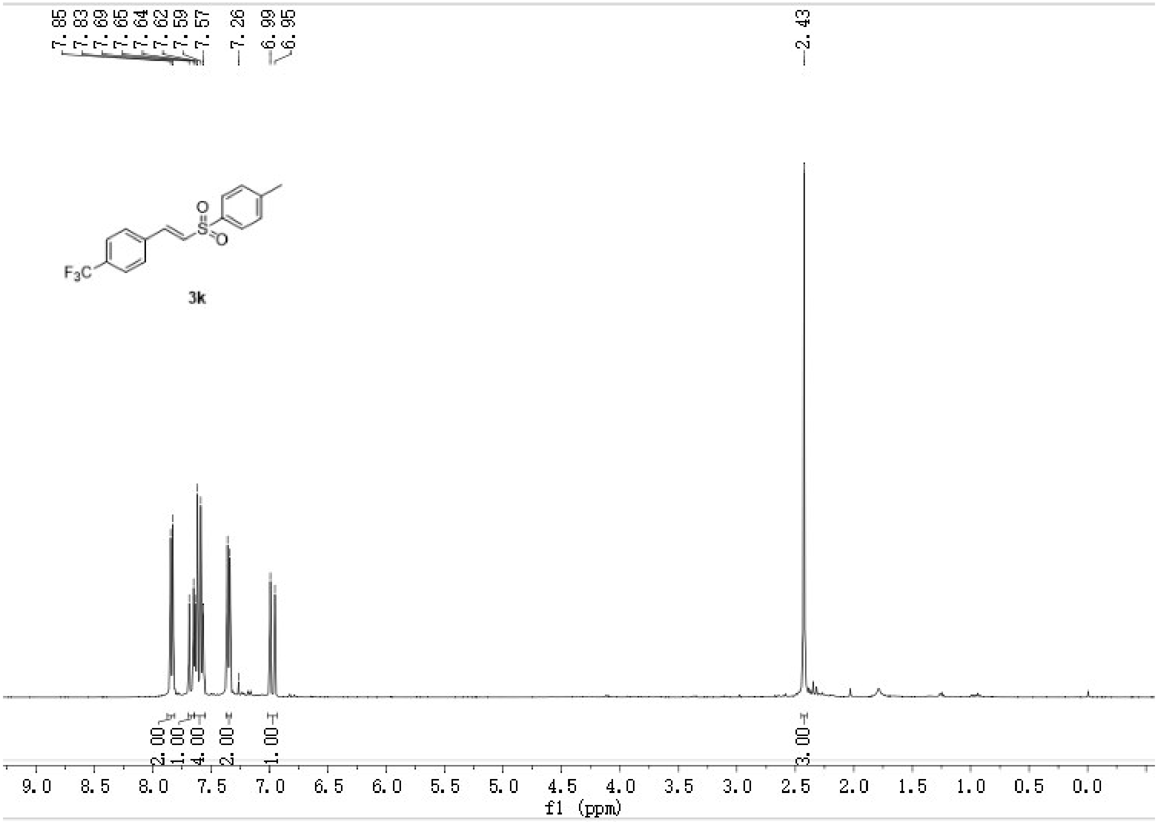
f1 (ppm)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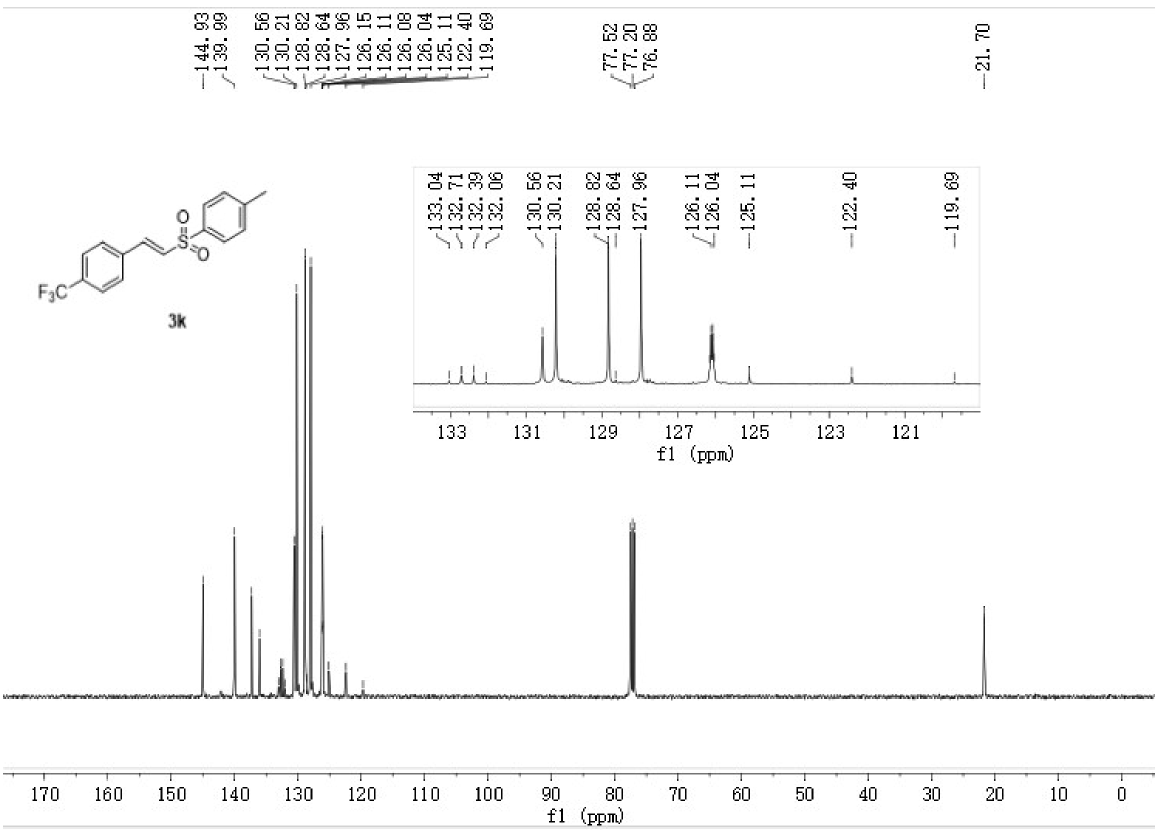
7.62
7.59
7.57
7.26
6.99
6.95
2.43
F3C
3k
2.00
1.00
4.00
2.00
1.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.64
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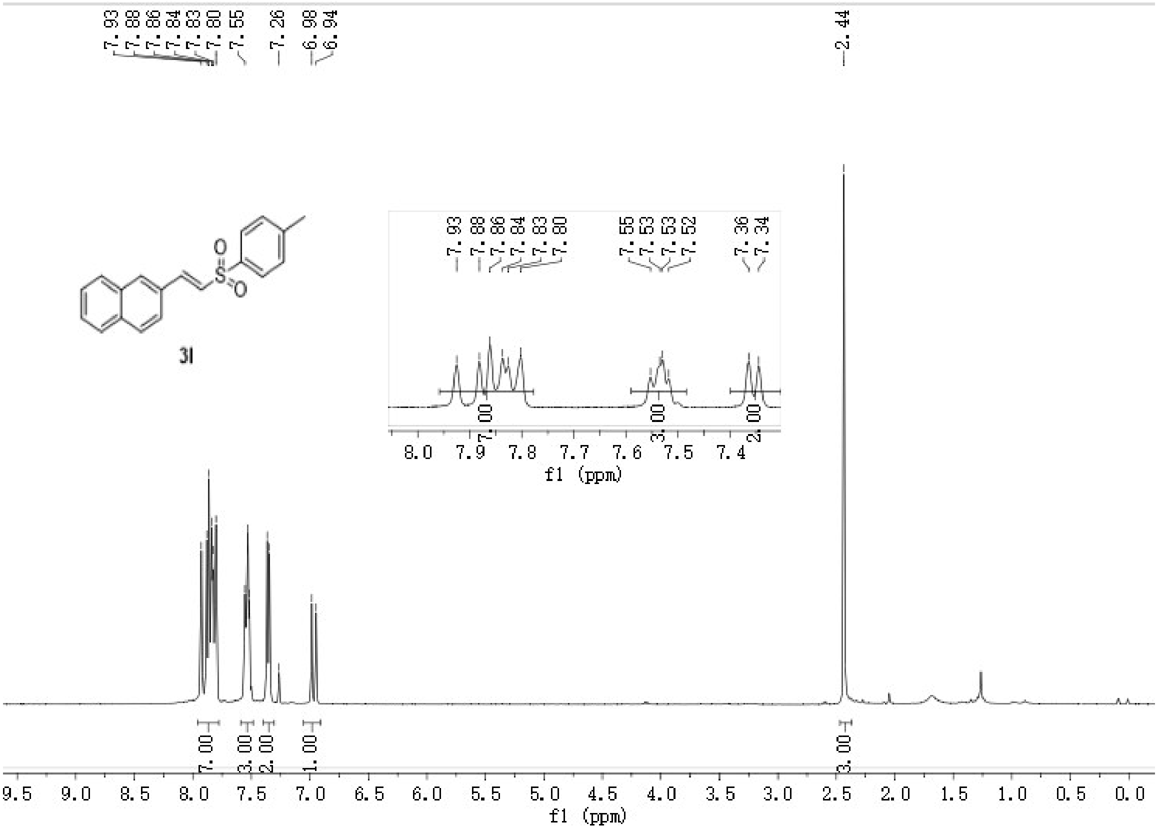
126.04
125.11
122.40
119.69
77.52
77.20
76.88
21.70
133.04
132.71
132.39
132.06
130.56
130.21
128.82
128.64
127.96
126.11
126.04
125.11
122.40
119.69
133 131 129 127 125 123 121
f1 (ppm)
F3C
3k
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.36
7.34
3l
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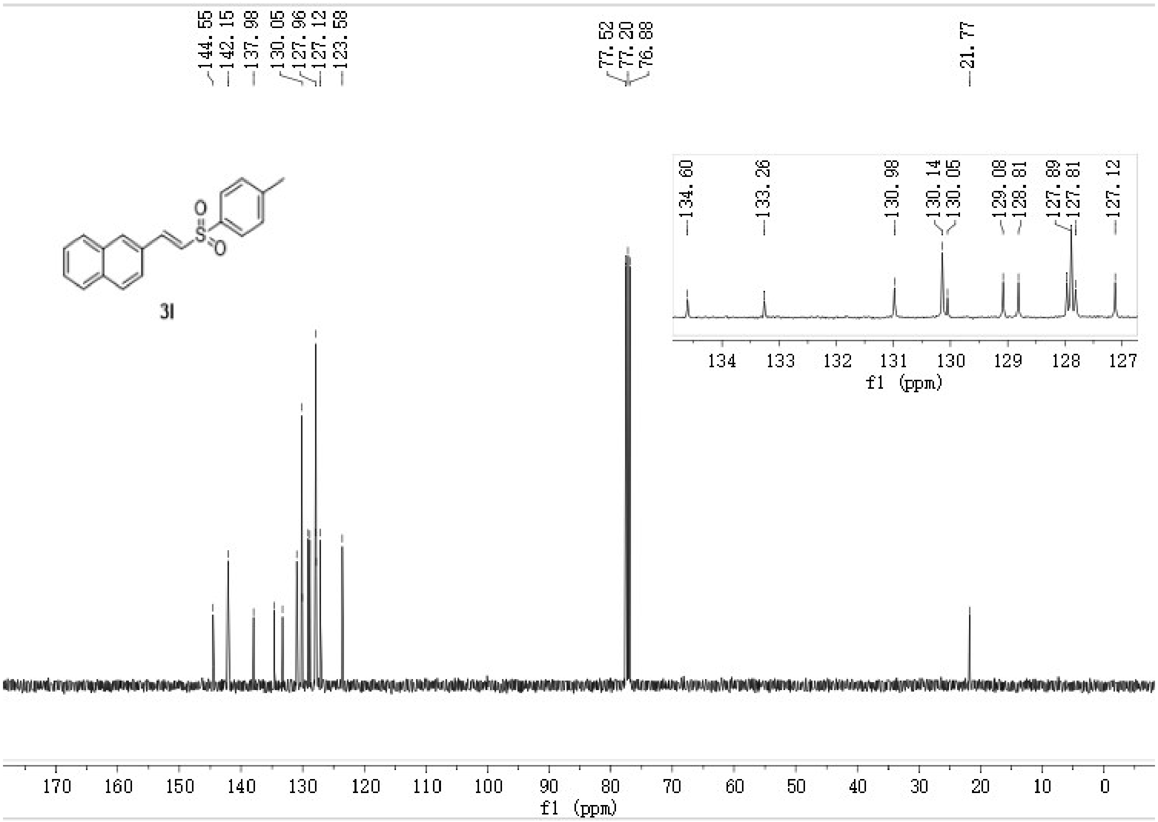
3.00
2.00
1.00
3.00
f1 (ppm)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130.05
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127.12
123.58
77.52
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76.88
21.77
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129.08
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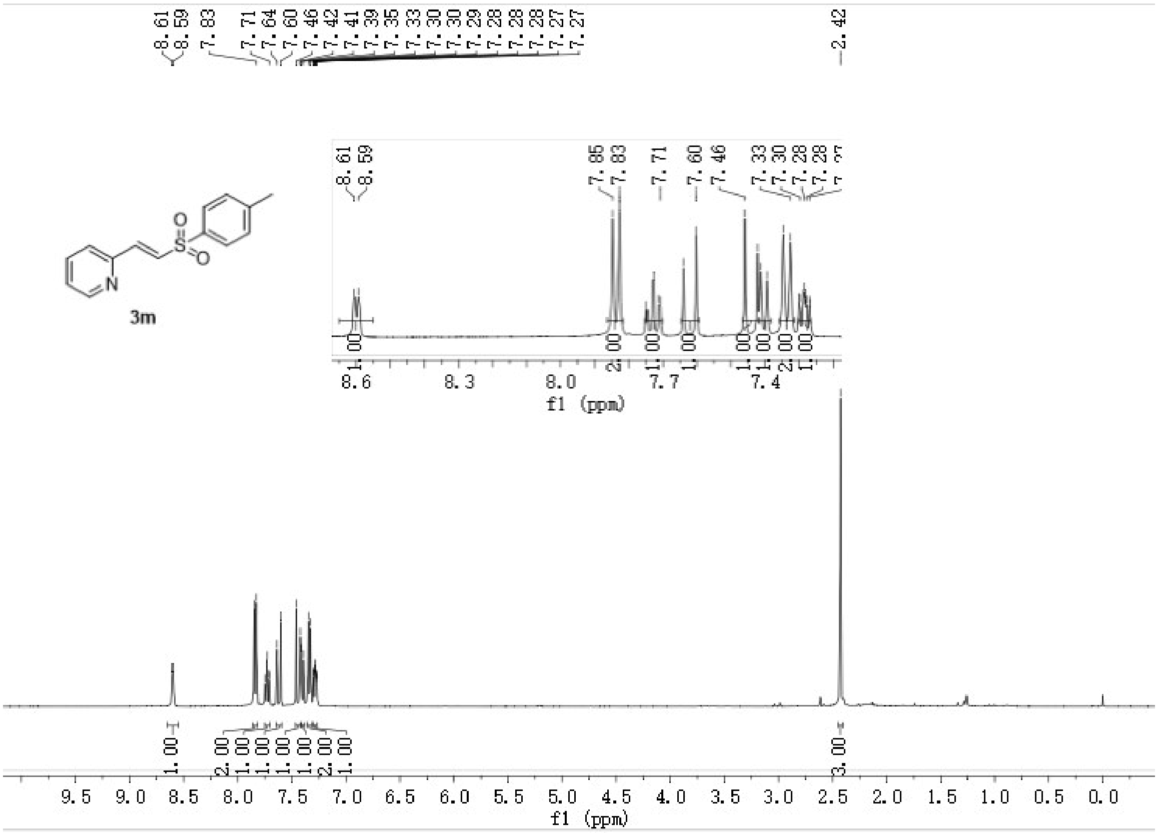
127.12
3l
f1 (ppm)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7.42
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7.39
7.35
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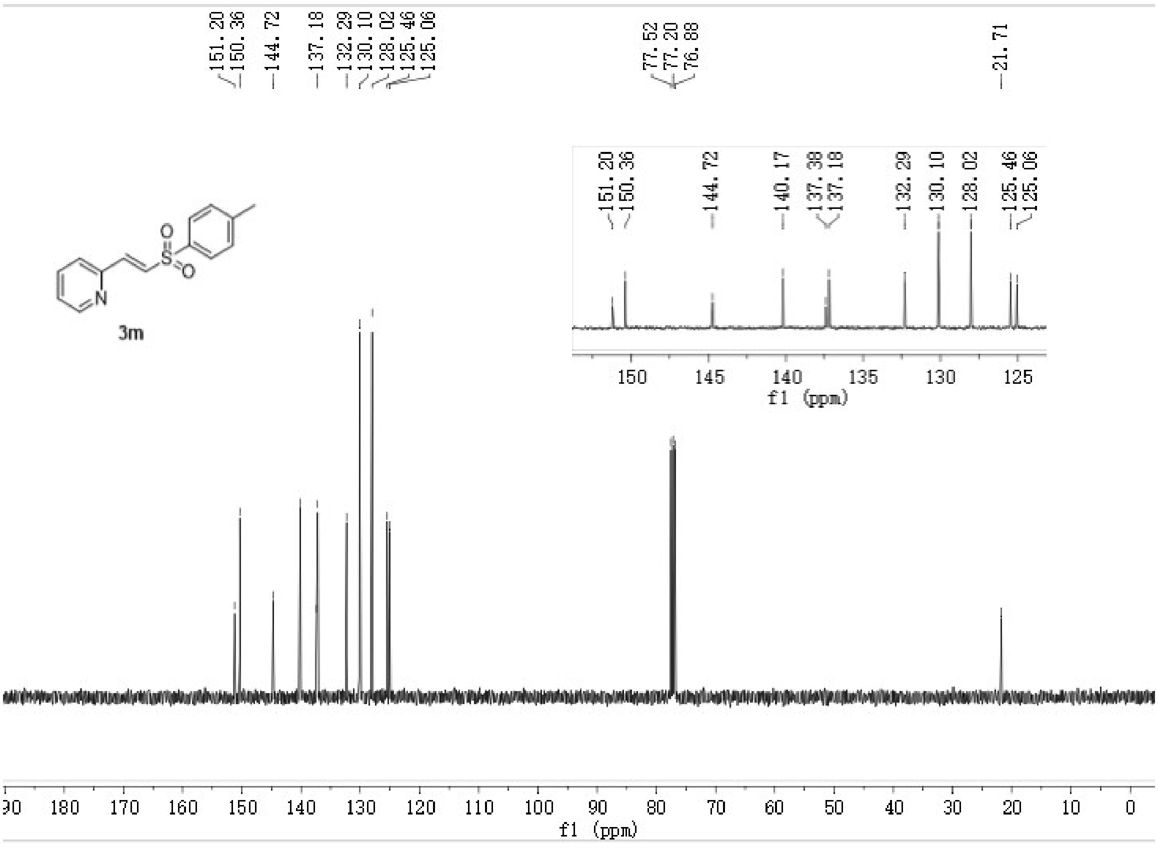
7.29
7.28
7.28
7.28
7.27
7.27
2.42
3m
f1 (ppm)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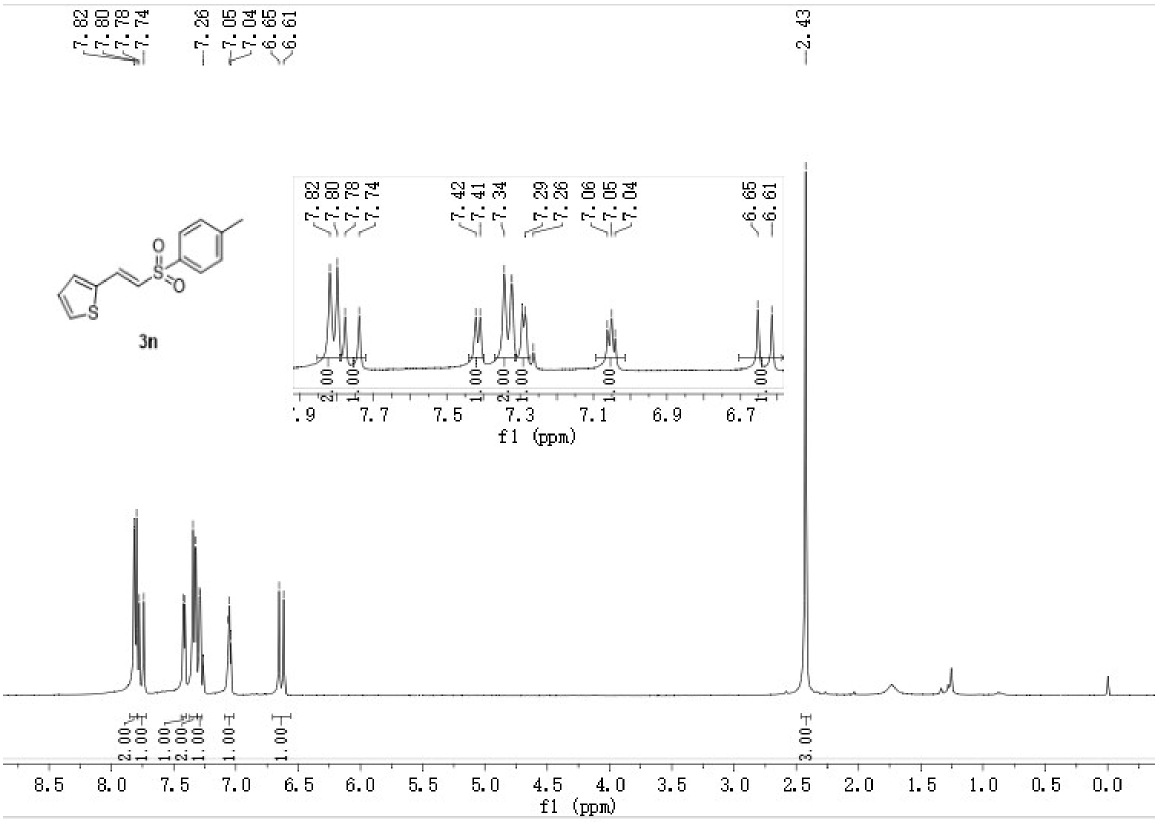
125.06
77.52
77.20
76.88
21.71
140.17
137.38
3m
f1 (ppm)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7.78
7.74
7.26
7.05
7.04
6.65
6.61
2.43
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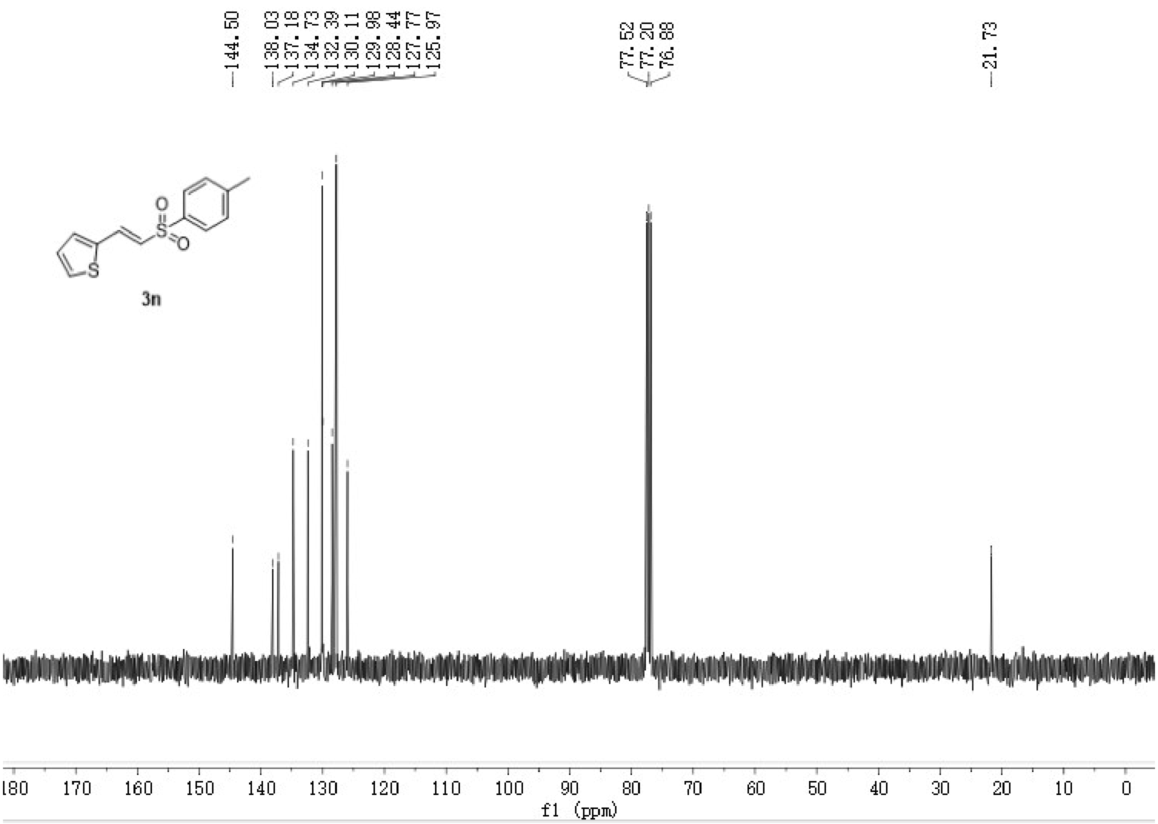
7.41
7.34
7.29
7.26
7.06
7.05
7.04
6.65
6.61
3n
f1 (ppm)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134.73
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130.11
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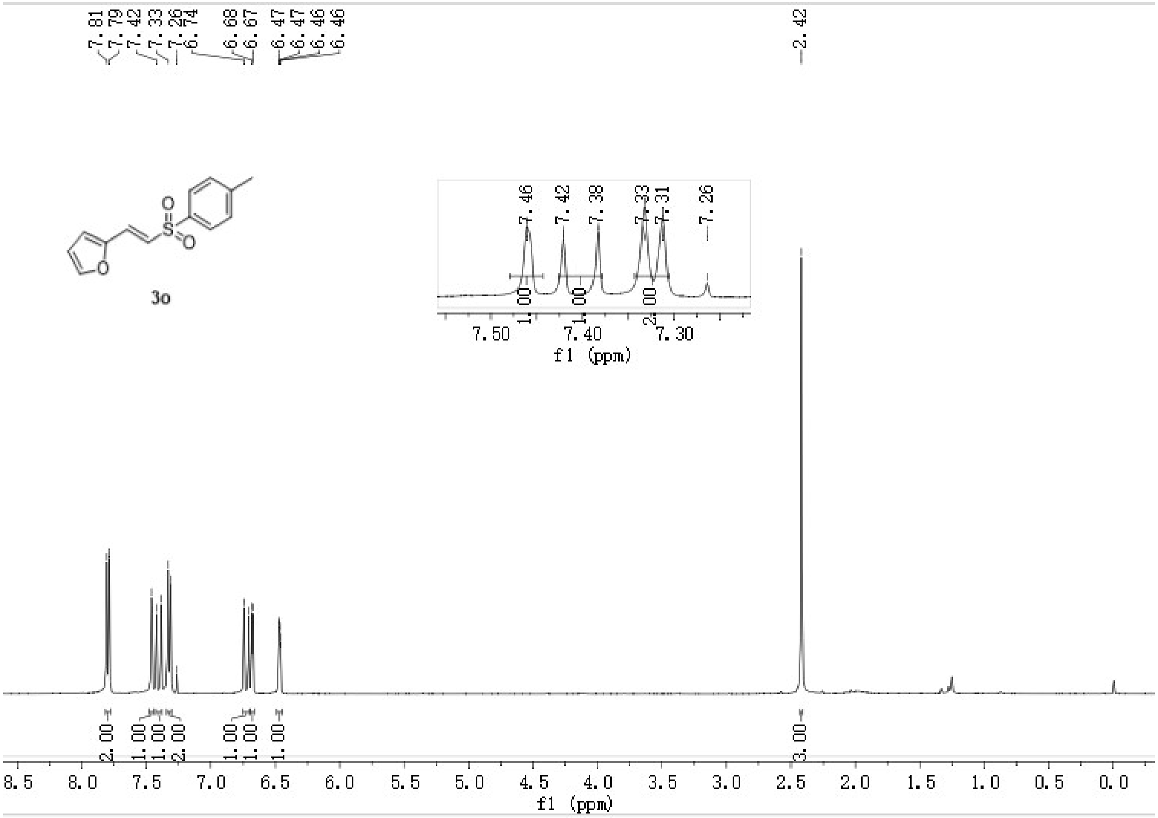
77.52
77.20
76.88
21.73
3n
f1 (ppm)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6.47
6.46
6.46
2.42
3o
7.46
7.42
7.38
7.33
7.31
7.26
1.00
1.00
2.00
7.50
7.40
7.30
f1 (ppm)
2.00
1.00
1.00
2.00
1.00
1.00
1.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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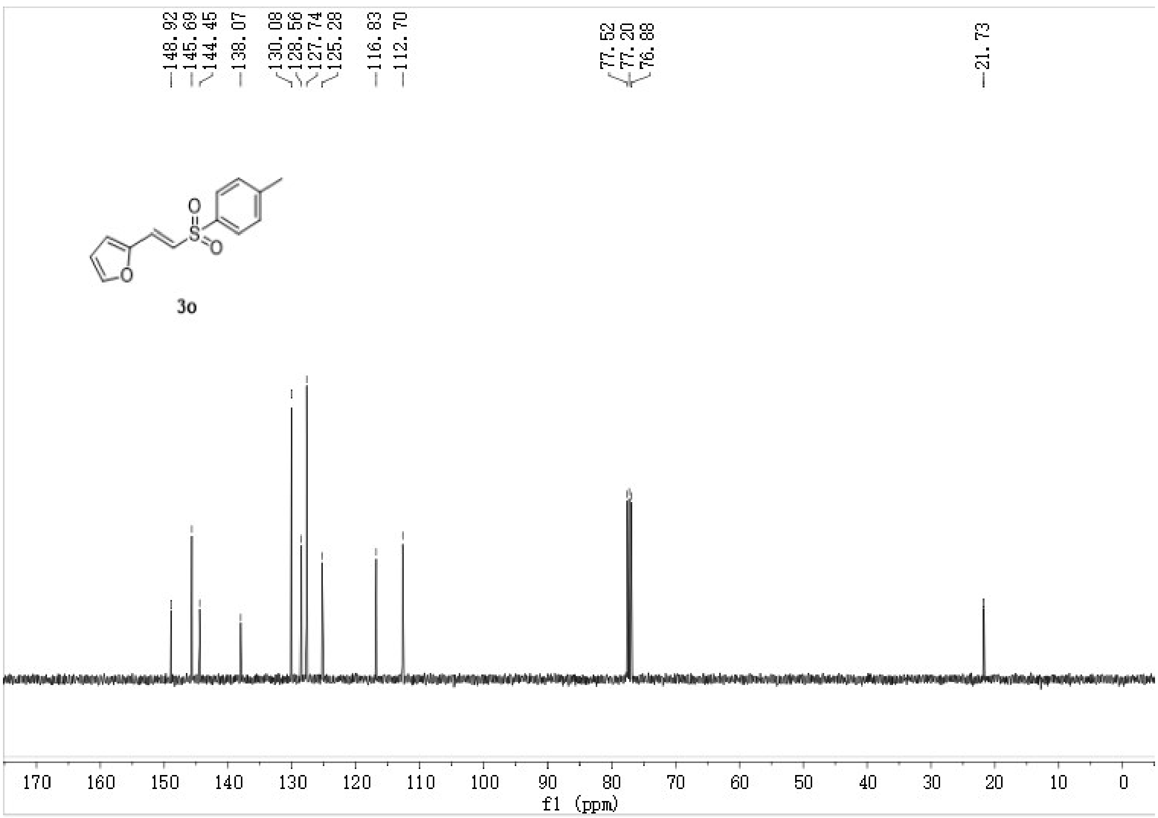
0.0
f1 (ppm)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112.70
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21.73
3o
170
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150
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130
120
110
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90
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50
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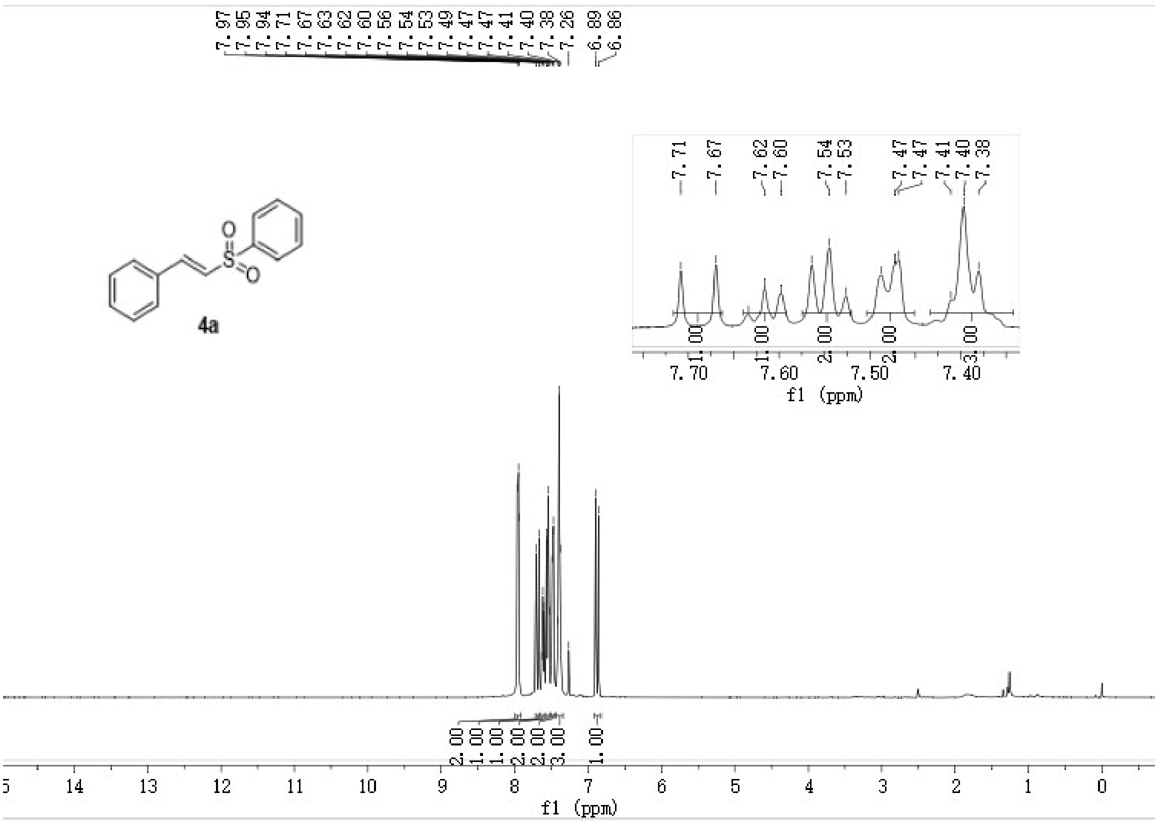
10
0
f1 (ppm)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7.60
7.56
7.54
7.53
7.49
7.47
7.47
7.41
7.40
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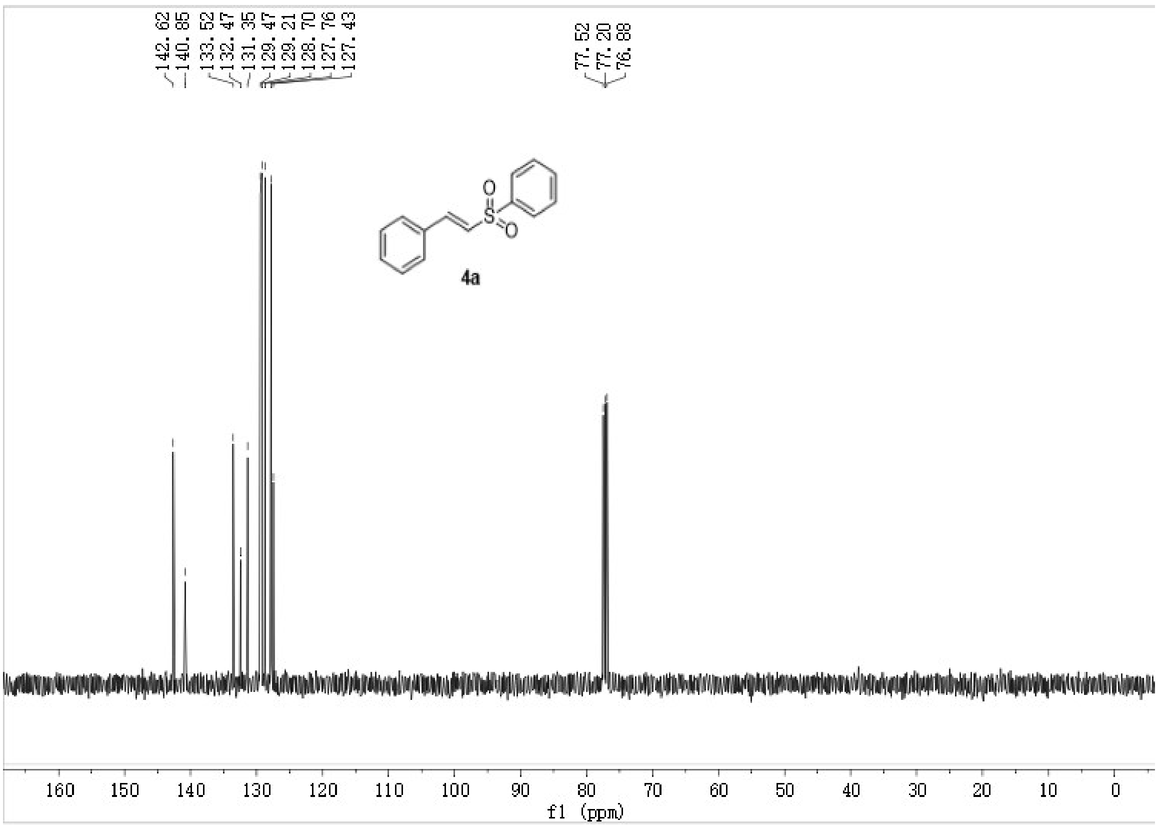
6.89
6.86
4a
7.70
7.60
7.50
7.40
f1 (ppm)
f1 (ppm)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77.52
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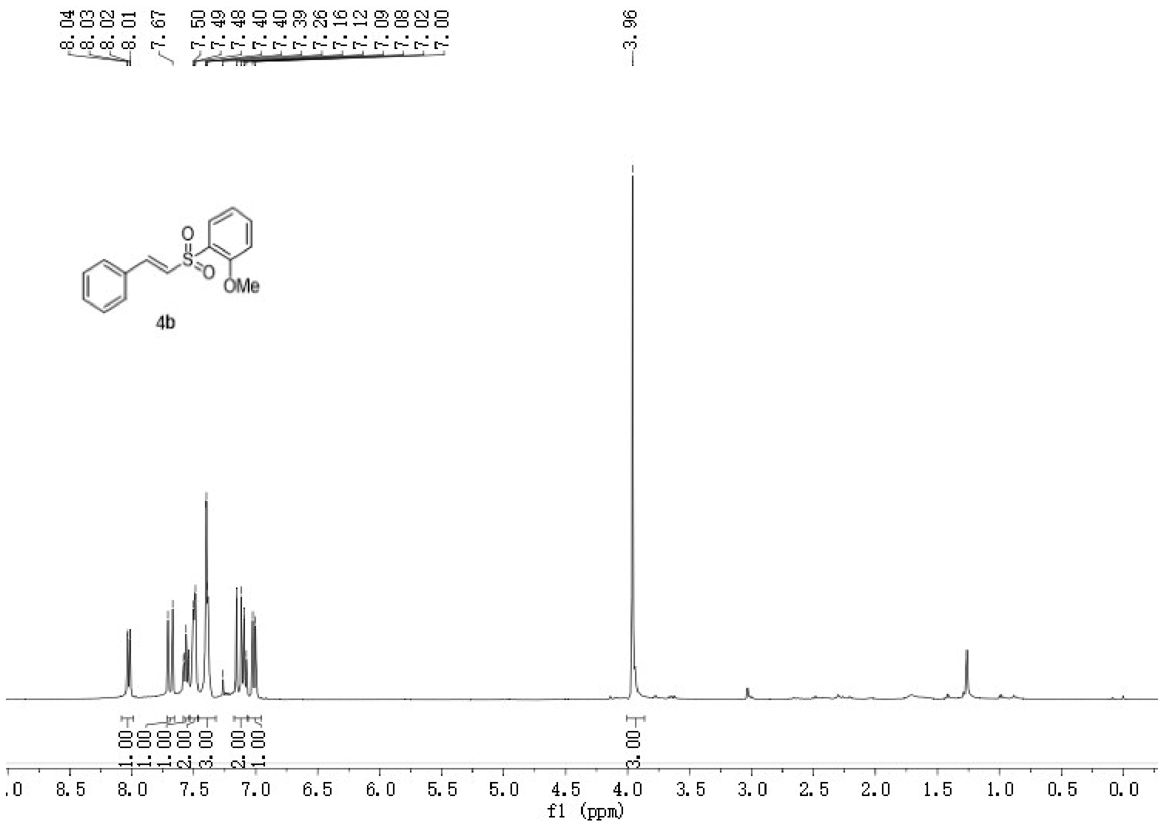
76.88
4a
f1 (ppm)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7.48
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7.40
7.39
7.26
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7.12
7.09
7.08
7.02
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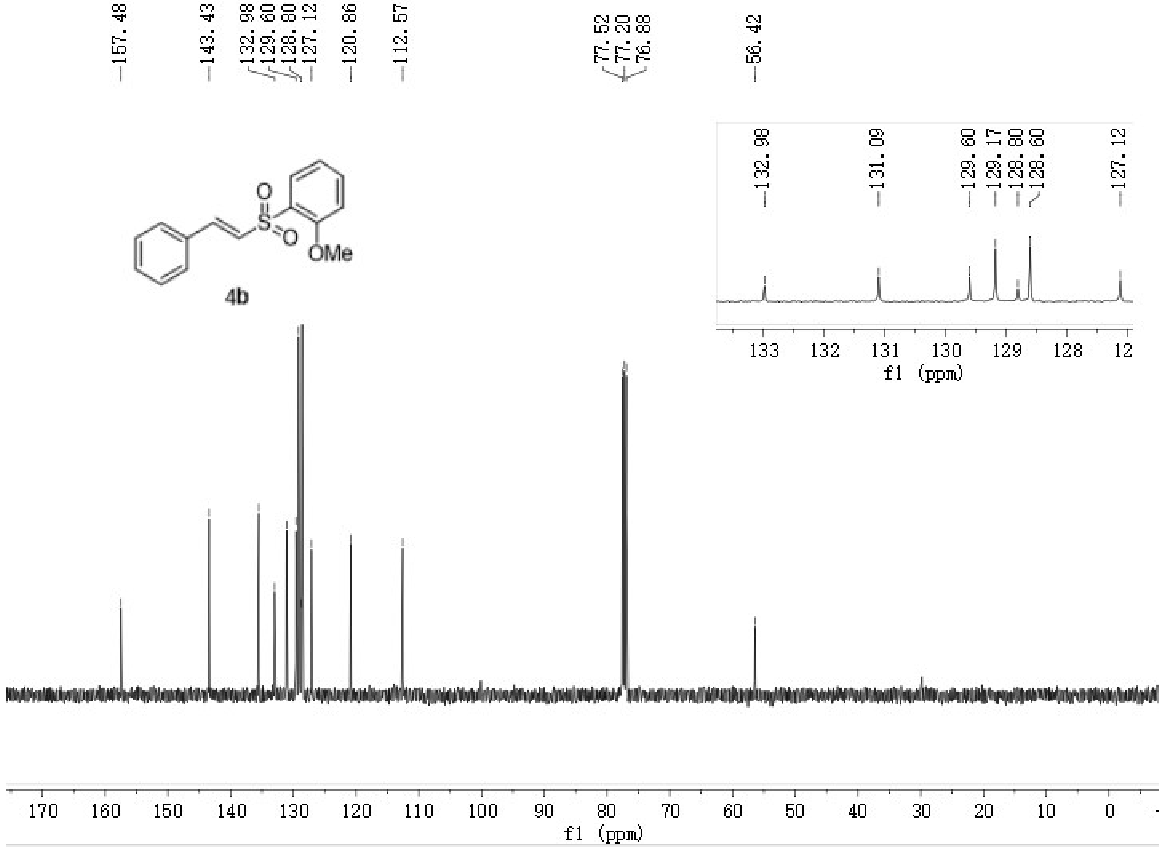
1.00
3.00
f1 (ppm)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112.57
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77.20
76.88
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OMe
4b
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131.09
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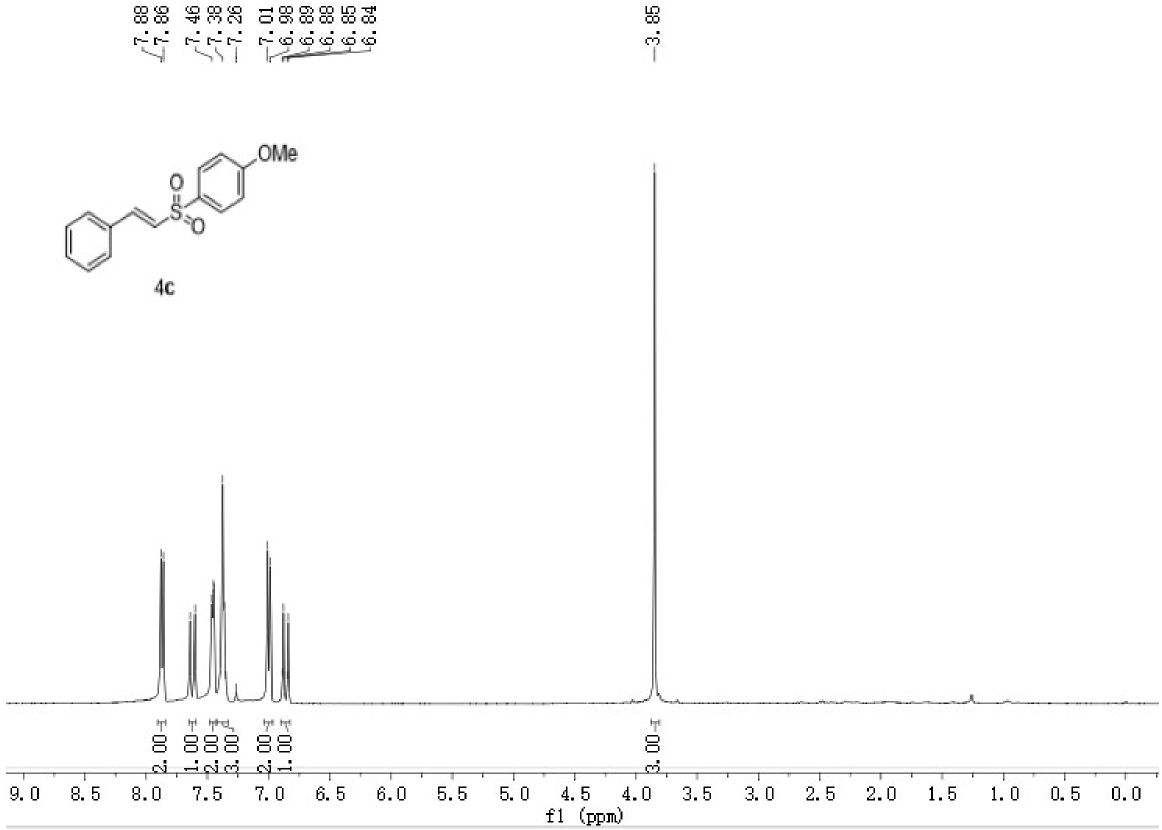
128.80
128.60
127.12
f1 (ppm)
f1 (ppm)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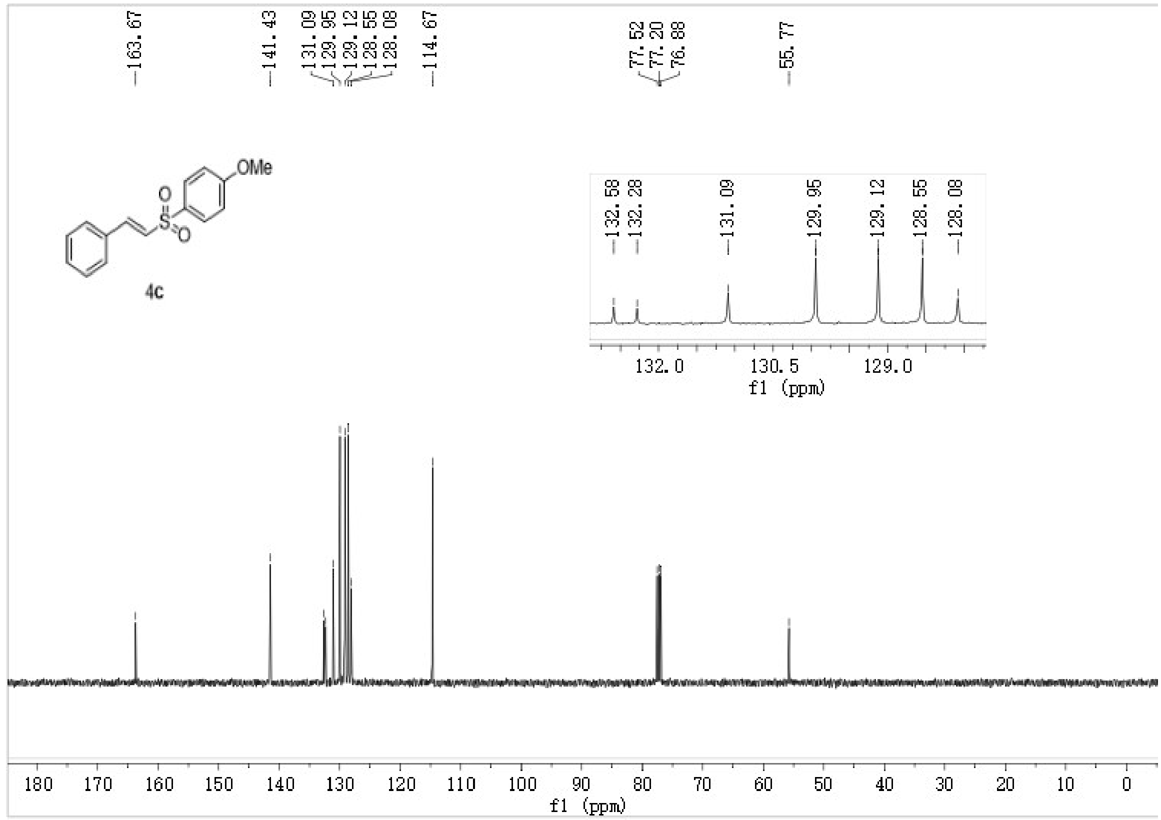
7.38
7.26
7.01
6.98
6.89
6.88
6.85
6.84
3.85
OMe
O
S
O
4c
2.00
1.00
2.00
3.00
2.00
1.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.55
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77.52
77.20
76.88
55.77
OMe
O
S
O
4c
132.58
132.28
131.09
129.95
129.12
128.55
128.08
132.0 130.5 129.0
f1 (ppm)
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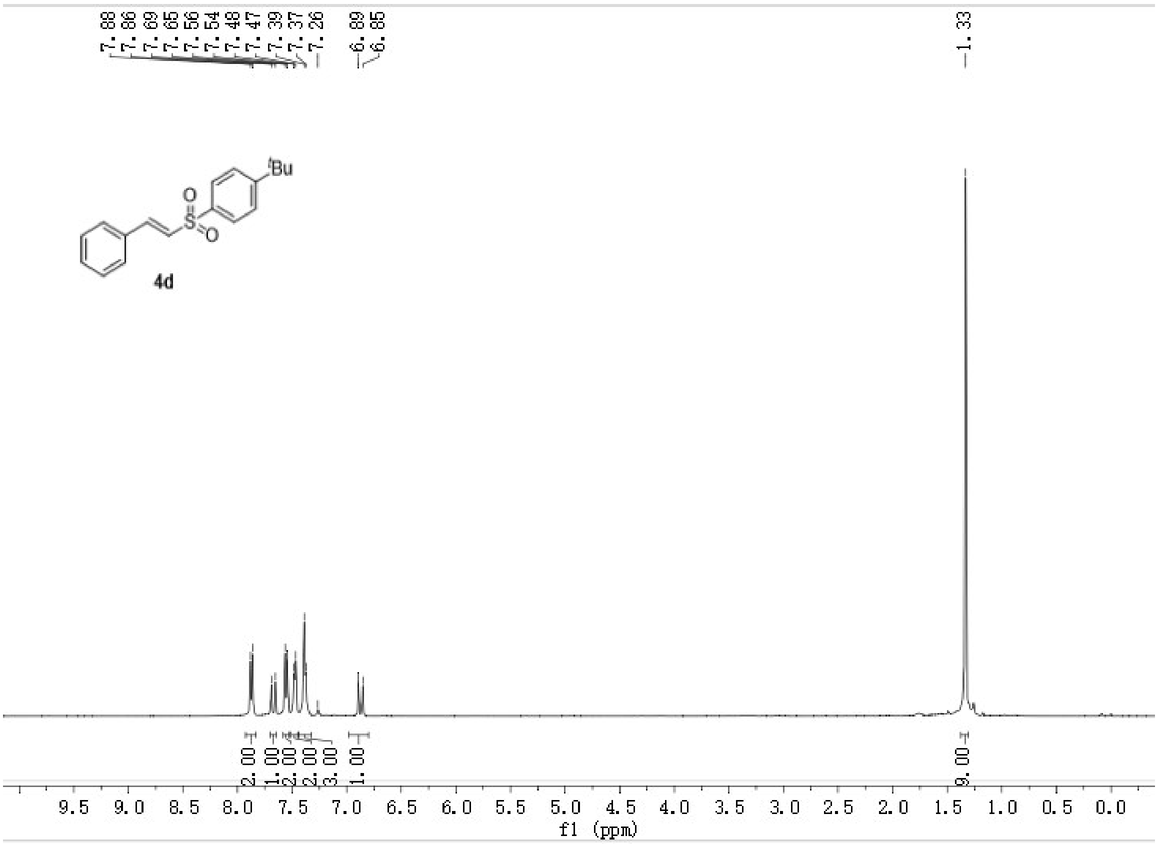
f1 (ppm)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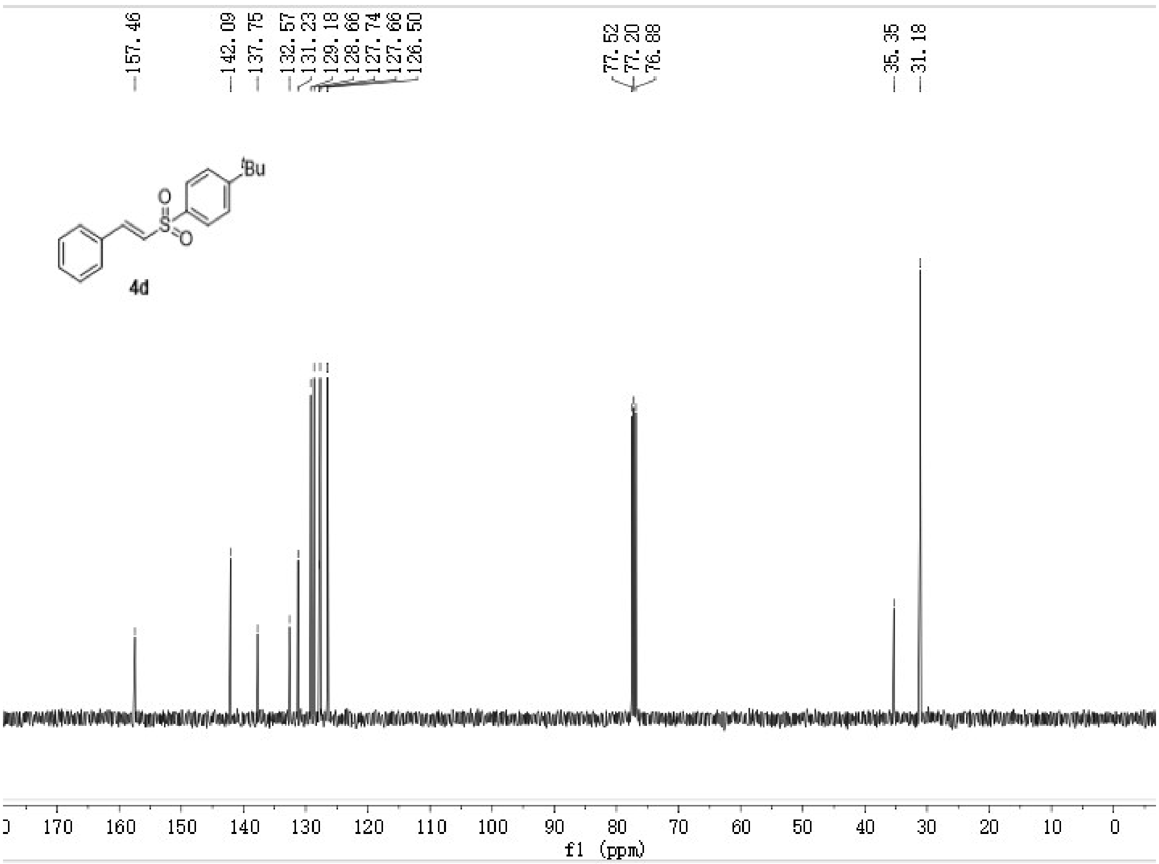
7.69
7.65
7.56
7.54
7.48
7.47
7.39
7.37
7.26
6.89
6.85
1.33
tBu
O
S
O
4d
2.00
1.00
2.00
2.00
3.00
1.00
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.74
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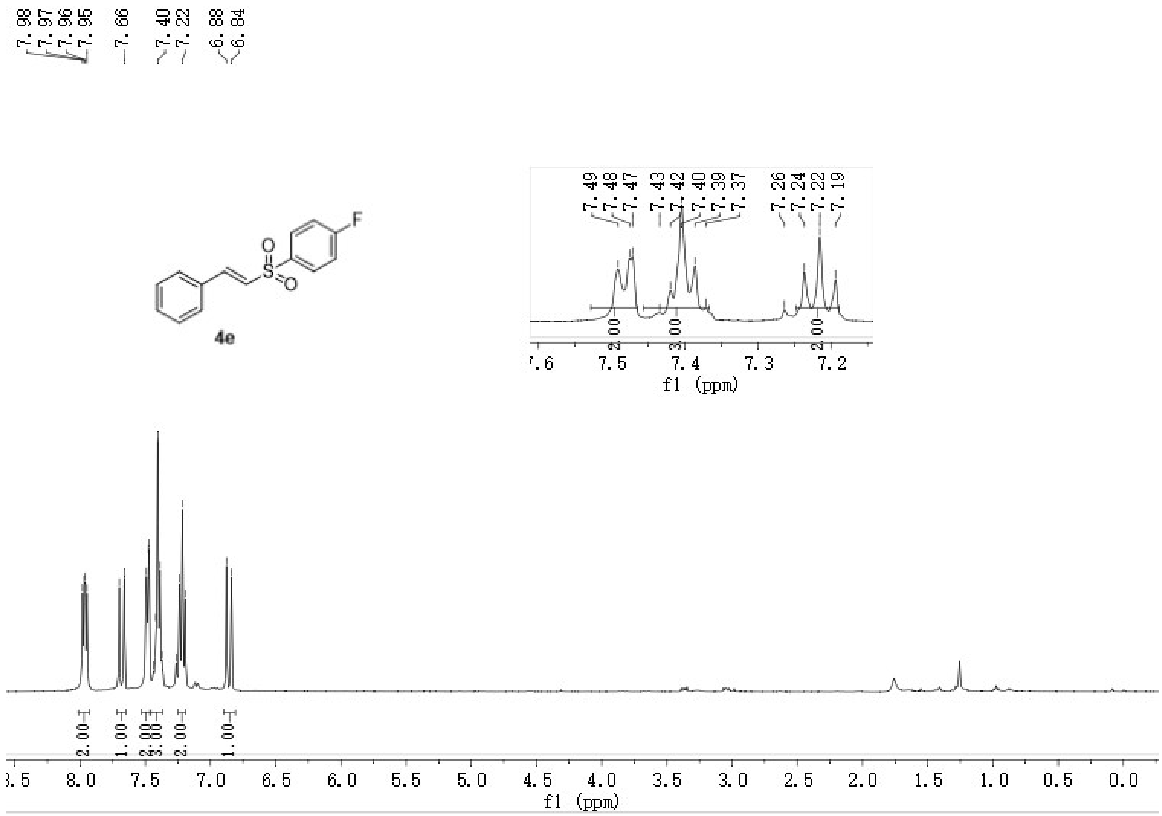
126.50
77.52
77.20
76.88
35.35
31.18
tBu
O
S
O
4d
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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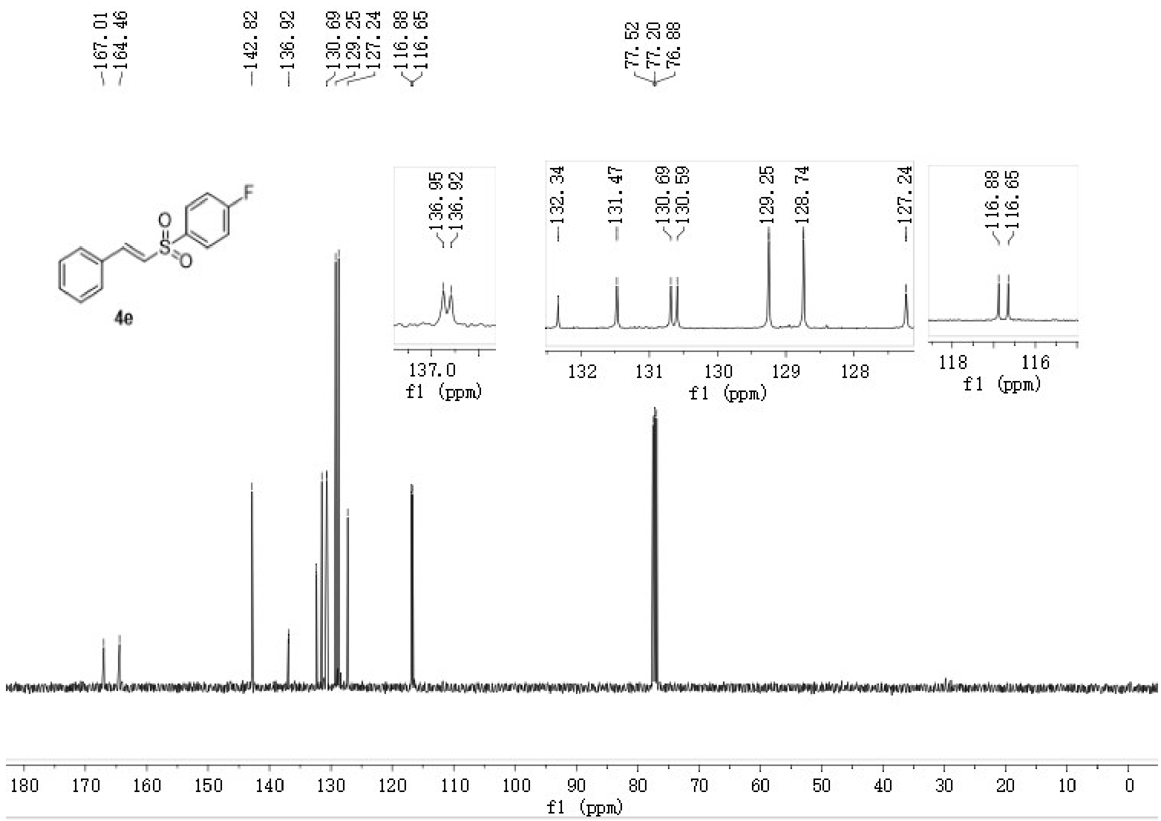
F
S
O
O
4e
7.49 7.48 7.47 7.43 7.42 7.40 7.39 7.37 7.26 7.24 7.22 7.19
2.00 3.00 2.00
f1 (ppm)
2.00 1.00 2.00 3.00 2.00 1.00
f1 (ppm)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F
S
O
O
4e
136.95 136.92
f1 (ppm)
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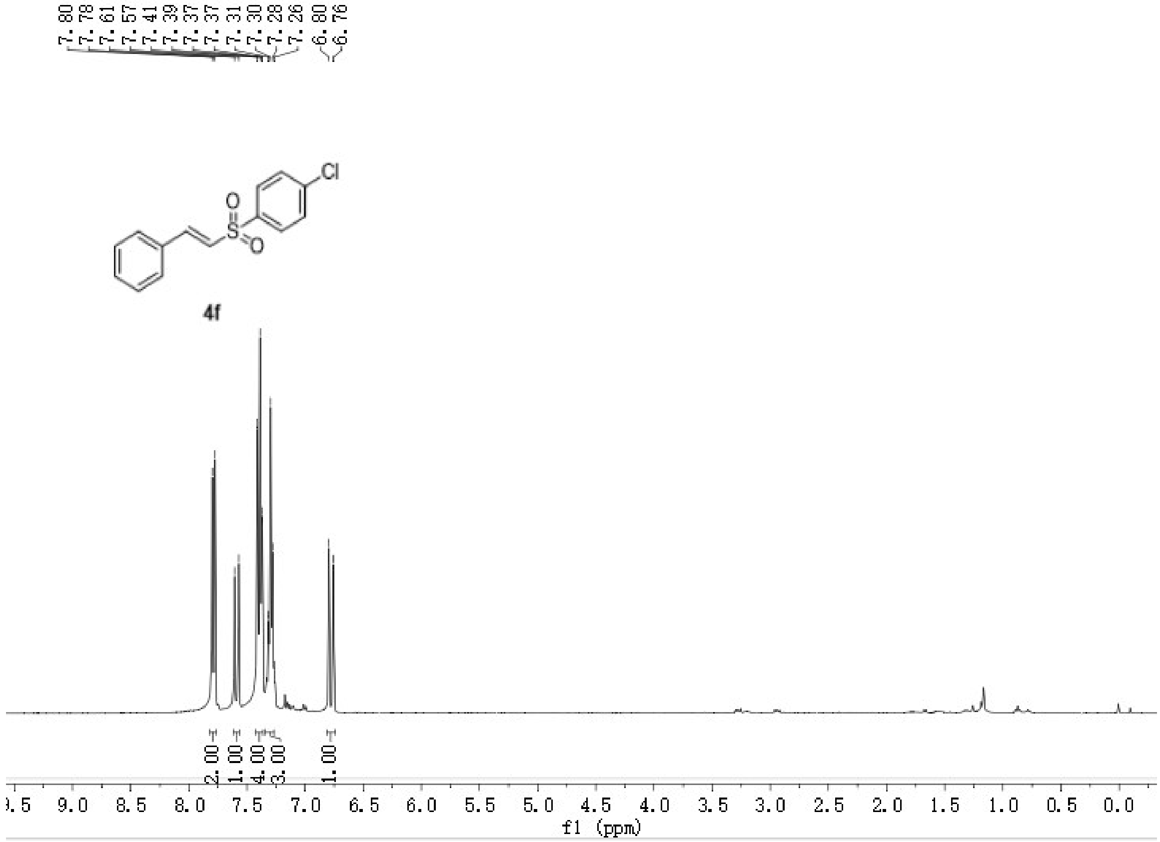
f1 (ppm)
116.88 116.65
f1 (ppm)
f1 (ppm)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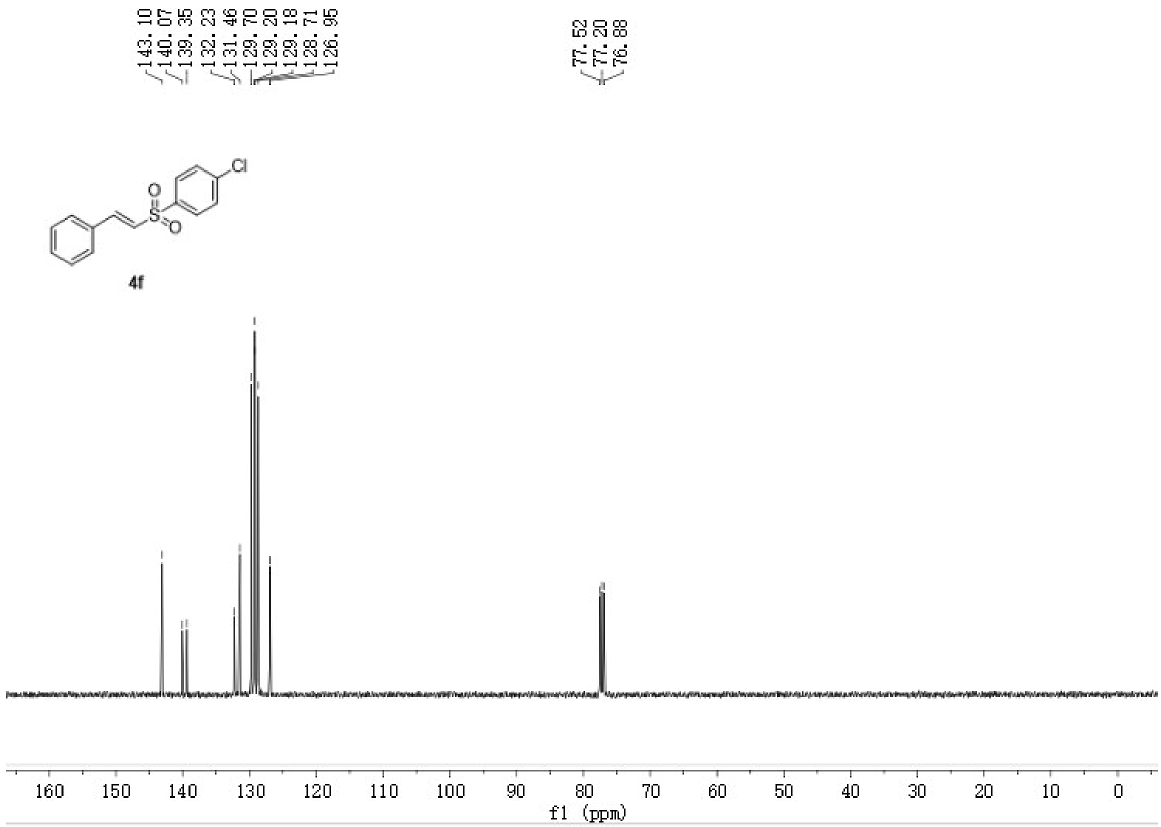
7.30
7.28
7.26
6.80
6.76
Cl
O
S
O
4f
2.00
1.00
4.00
3.00
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.20
129.18
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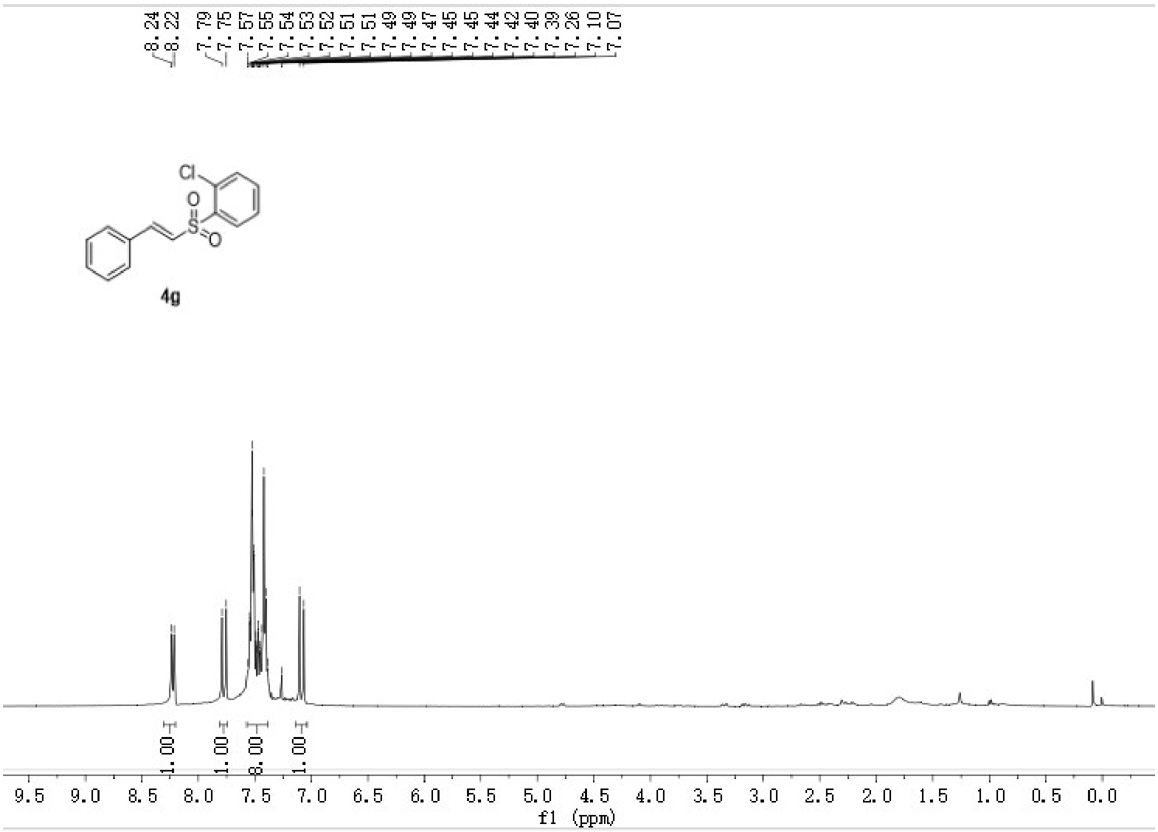
126.95
77.52
77.20
76.88
Cl
O
S
O
4f
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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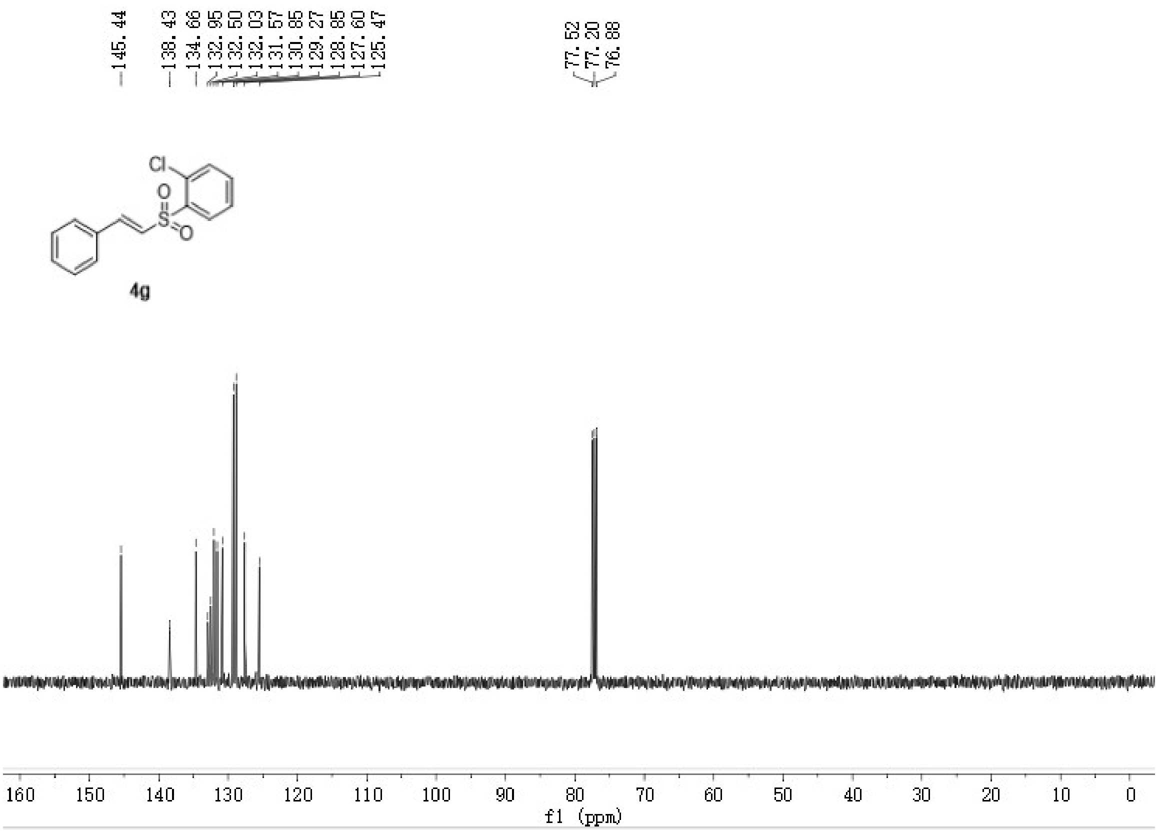
7.45
7.45
7.44
7.42
7.40
7.39
7.26
7.10
7.07
Cl
O
S
O
4g
1.00
1.00
8.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.85
127.60
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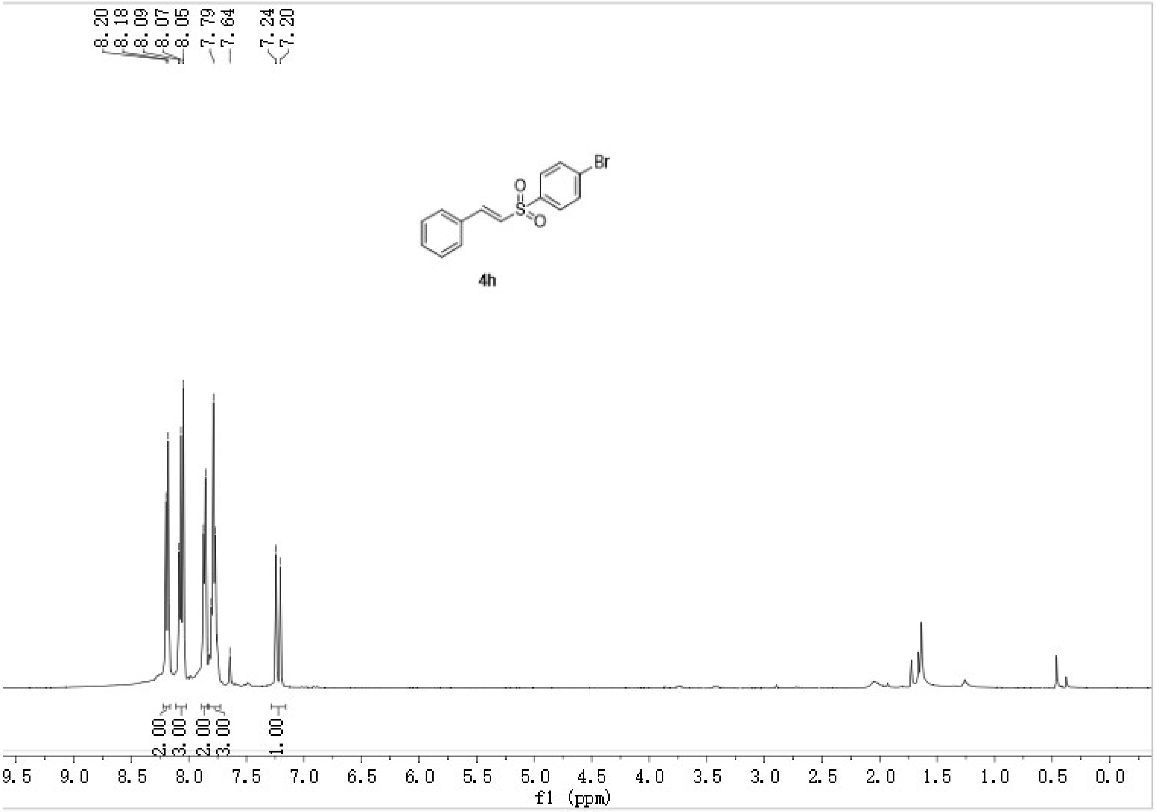
77.52
77.20
76.88
Cl
O
S
O
4g
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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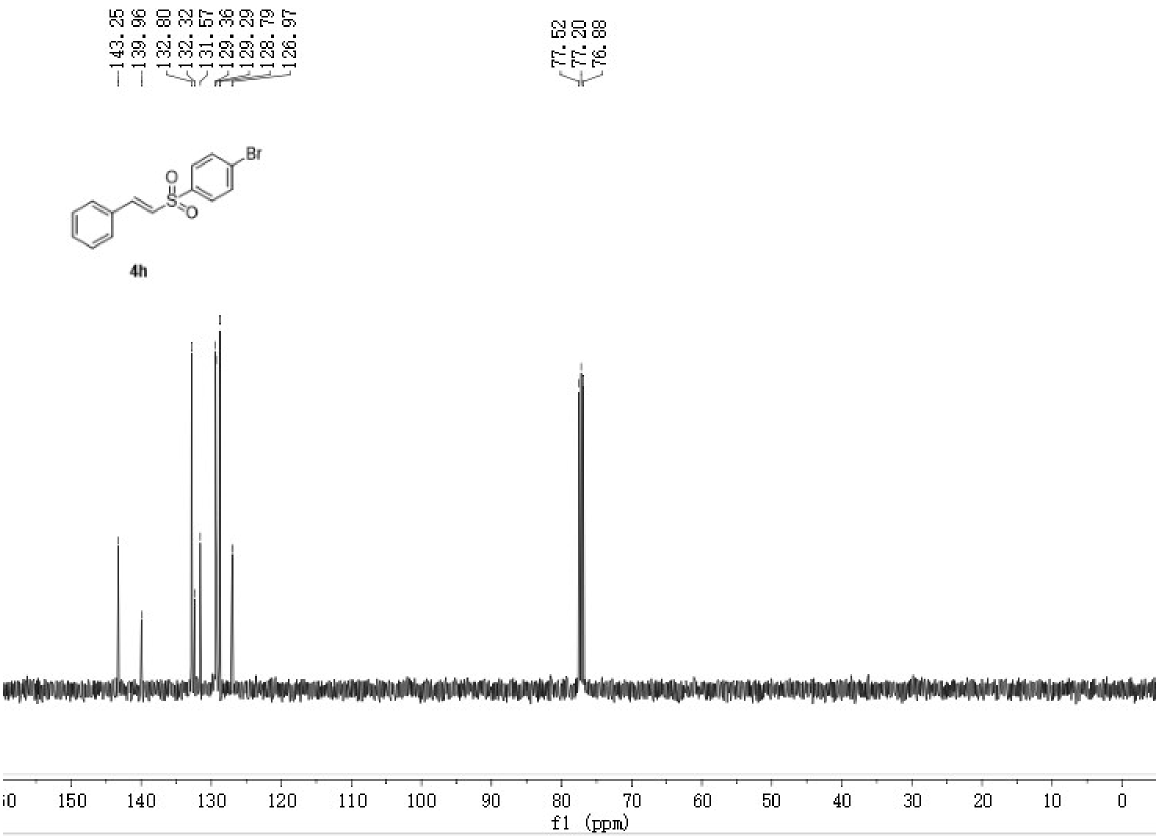
8.07
8.05
7.79
7.64
7.24
7.20
Br
O
S
O
4h
2.00
3.00
2.00
3.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.79
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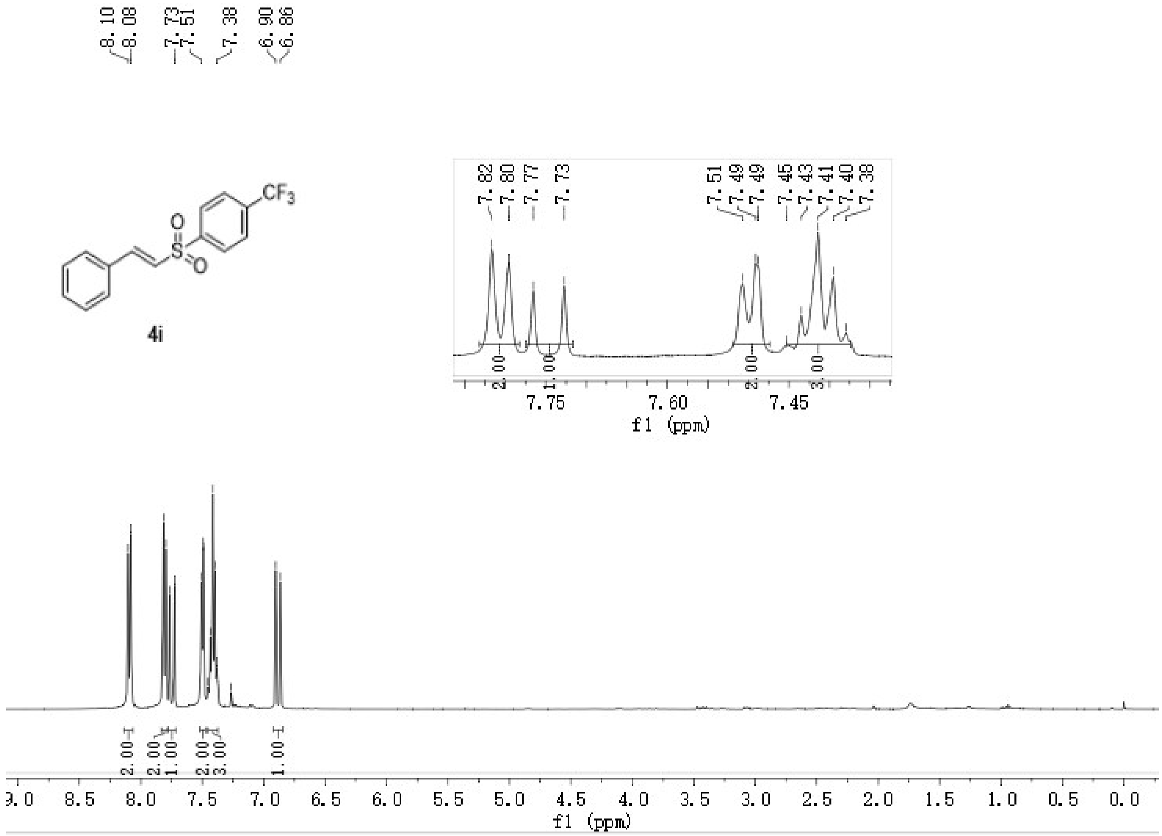
76.88
Br
O
S
O
4h
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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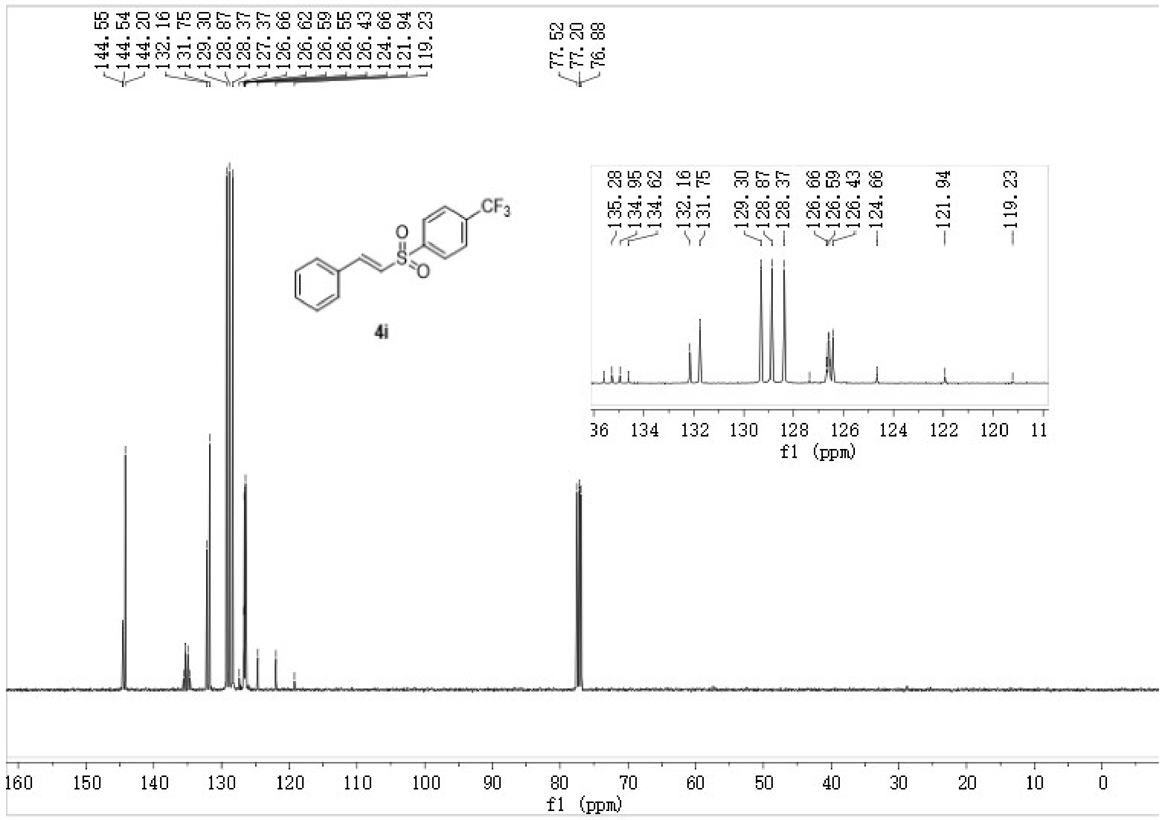
7.49
7.49
7.45
7.43
7.41
7.40
7.38
CF_3
4i
7.75
7.60
7.45
f1 (ppm)
2.00
2.00
1.00
2.00
3.00
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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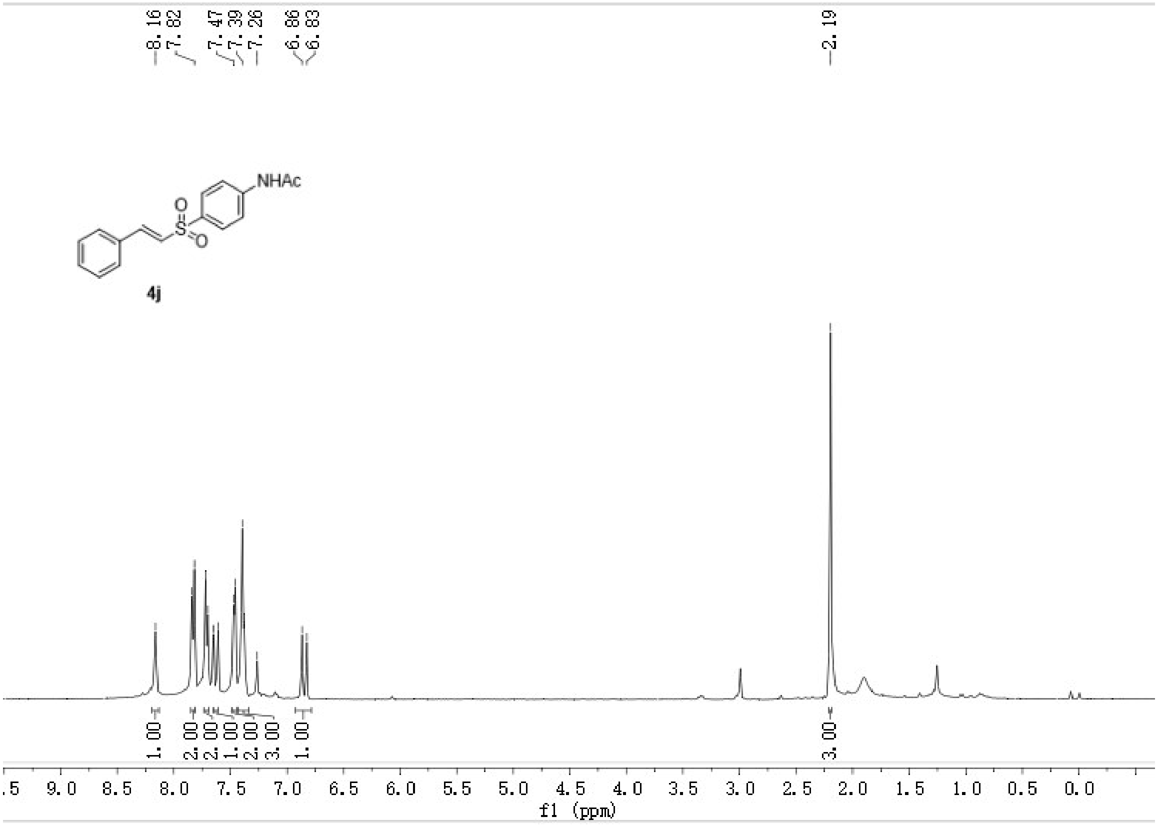
144.54
144.20
132.16
131.75
129.30
128.87
128.37
127.37
126.66
126.62
126.59
126.55
126.43
124.66
121.94
119.23
77.52
77.20
76.88
135.28
134.95
134.62
132.16
131.75
129.30
128.87
128.37
126.66
126.59
126.43
124.66
121.94
119.23
CF_3
4i
136 134 132 130 128 126 124 122 120 118
f1 (ppm)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.47
7.39
7.26
6.86
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4j
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2.00
2.00
1.00
2.00
3.00
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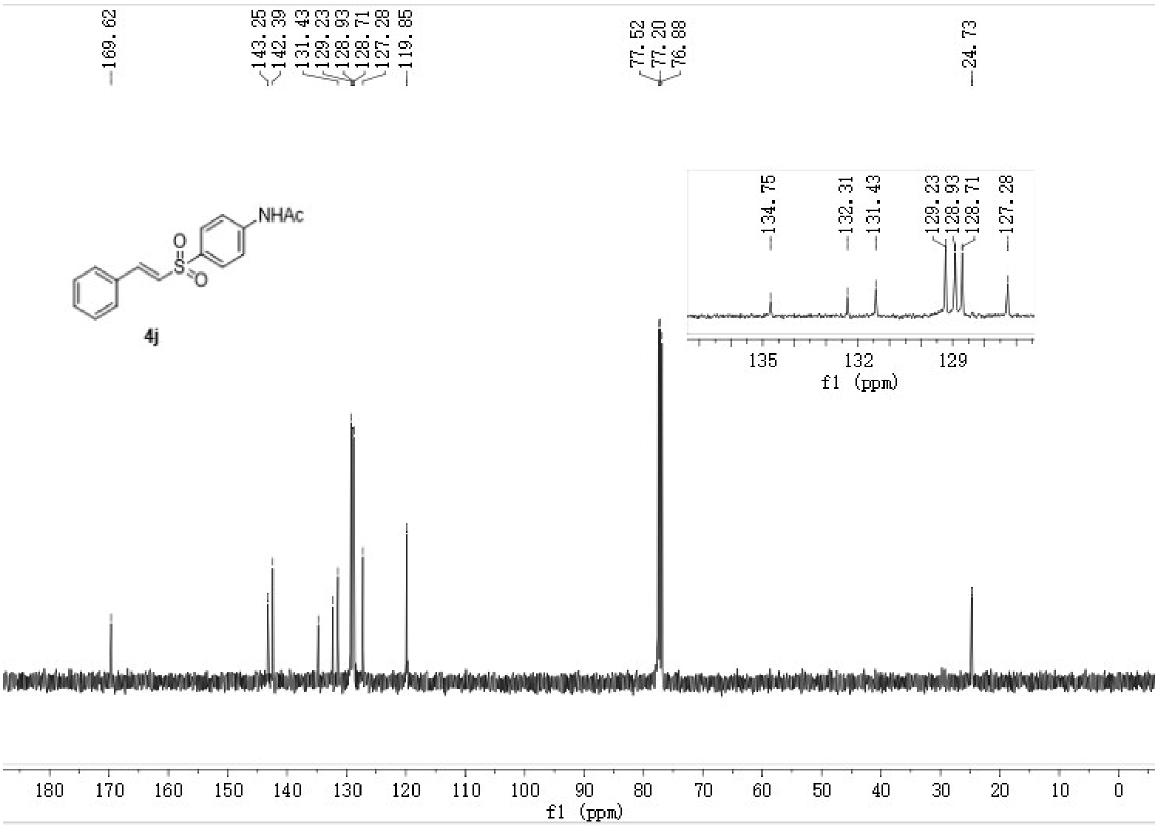
3.00
f1 (ppm)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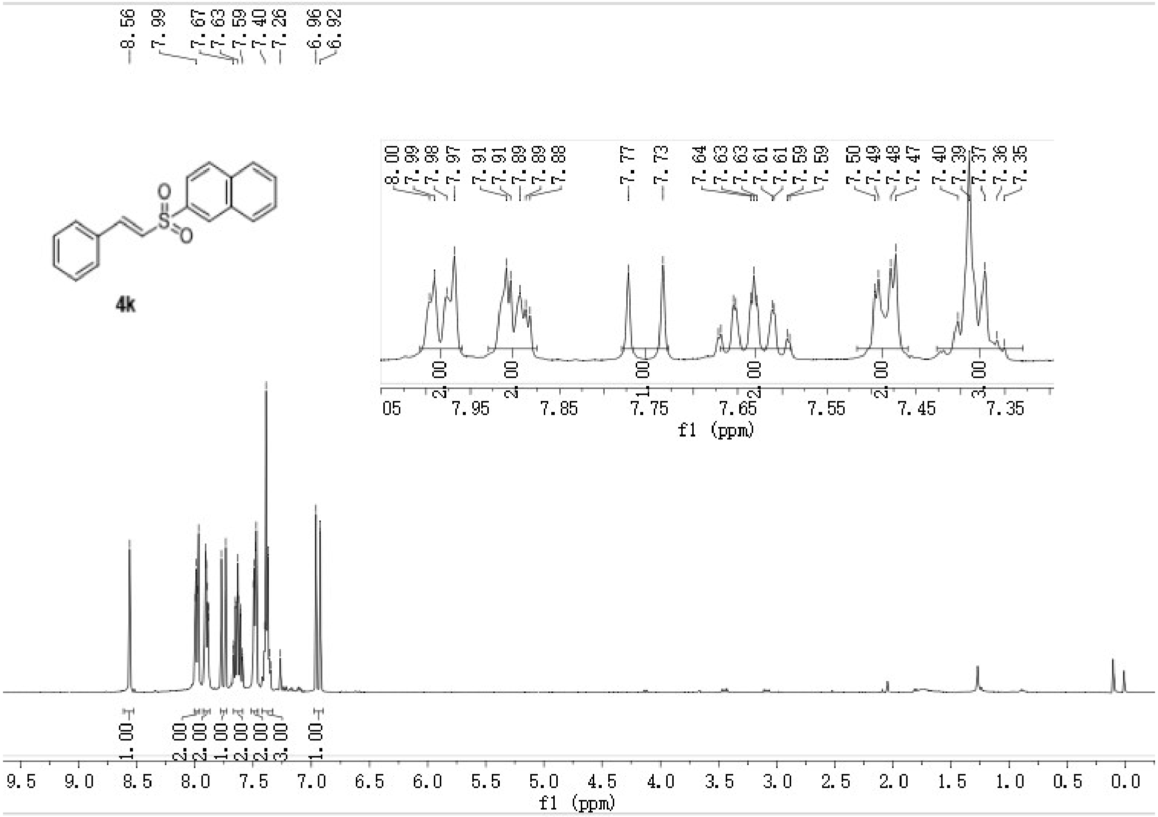
143.25
142.39
131.43
129.23
128.93
128.71
127.28
119.85
77.52
77.20
76.88
24.73
134.75
132.31
131.43
129.23
128.93
128.71
127.28
NHAc
4j
f1 (ppm)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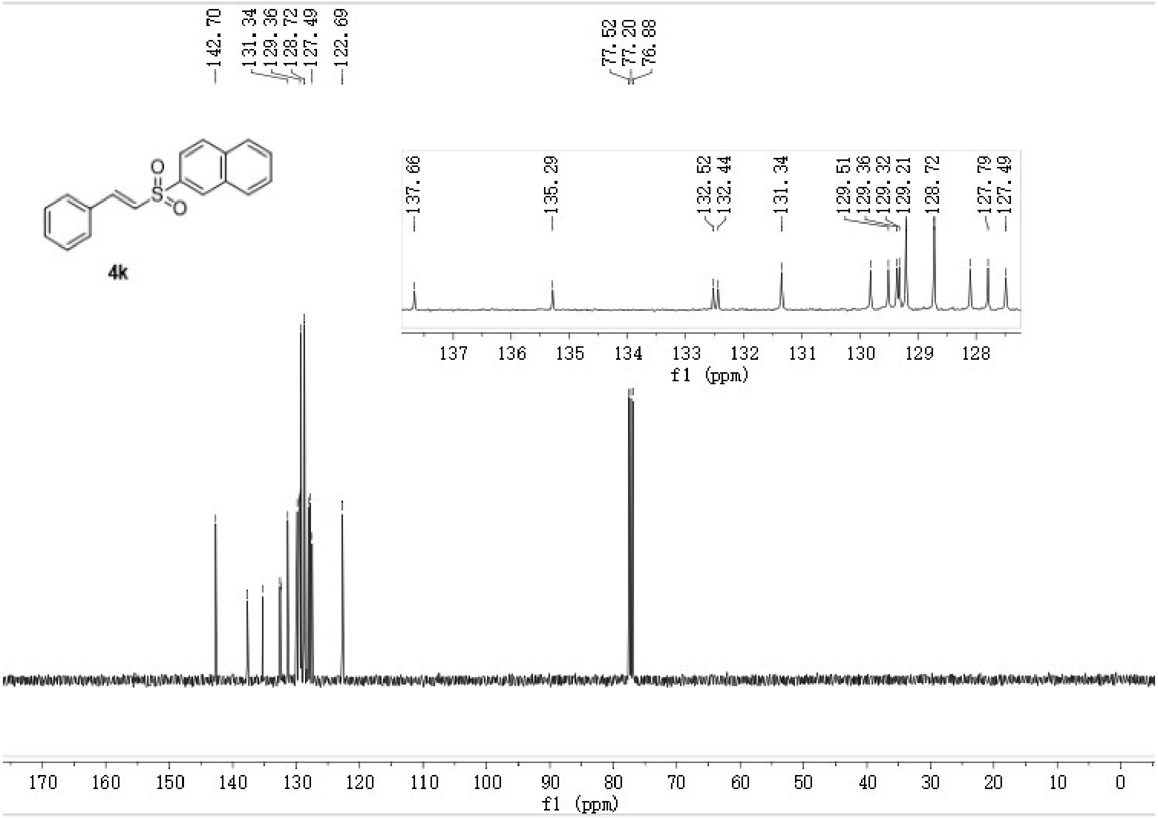
7.63
7.59
7.40
7.26
6.96
6.92
4k
8.00
7.99
7.98
7.97
7.91
7.91
7.89
7.88
7.77
7.73
7.64
7.63
7.63
7.61
7.61
7.59
7.59
7.50
7.49
7.48
7.47
7.40
7.39
7.37
7.36
7.35
f1 (ppm)
f1 (ppm)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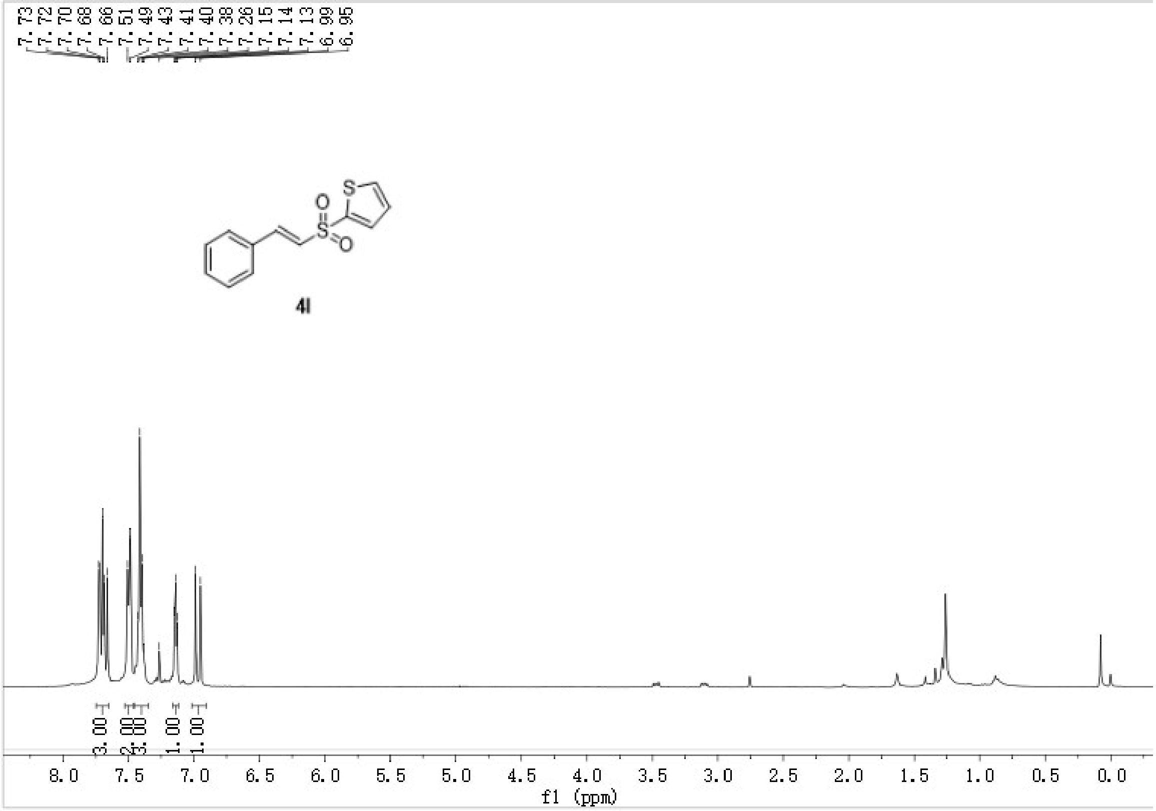
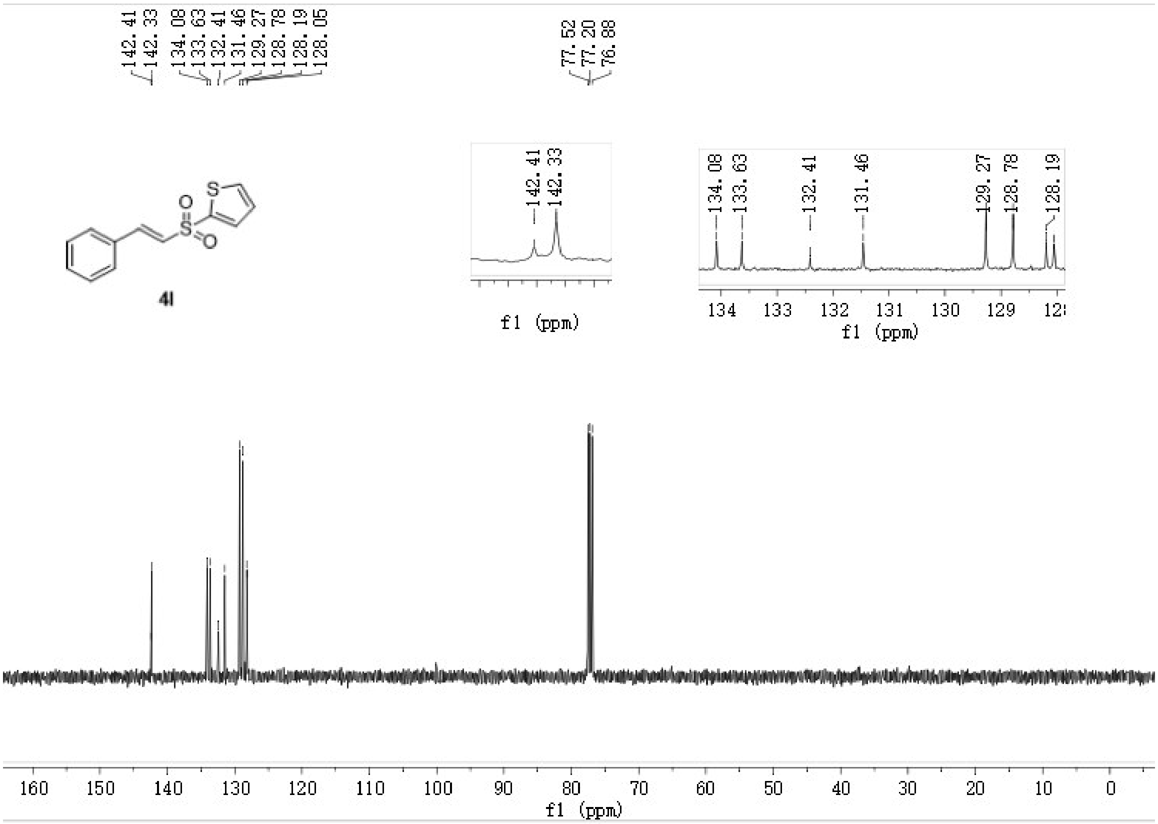
131.34
129.36
128.72
127.49
122.69
77.52
77.20
76.88
4k
137.66
135.29
132.52
132.44
131.34
129.51
129.36
129.32
129.21
128.72
127.79
127.49
f1 (ppm)
f1 (ppm)


7.73
7.72
7.70
7.68
7.66
7.51
7.49
7.43
7.41
7.40
7.38
7.26
7.15
7.14
7.13
6.99
6.95
4l
3.00
2.00
3.00
1.00
1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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142.33
134.08
133.63
132.41
131.46
129.27
128.78
128.19
128.05
77.52
77.20
76.88
4l
f1 (ppm)
134 133 132 131 130 129
f1 (ppm)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)