

Supporting Information

Electrochemical oxygenation of sulfides with water or molecular oxygen: switchable preparation of sulfones and sulfoxides

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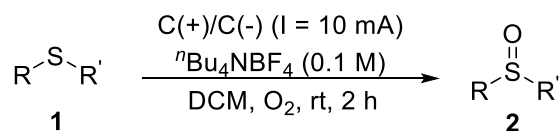
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(A) Typical Experimental Procedure

(a) General

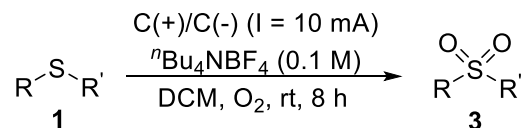
The ^1H and ^{13}C NMR spectra were recorded in CDCl_3 solvent on a NMR spectrometer using TMS as internal standard. HRMS was measured on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry. Melting points are uncorrected. The instrument for electrolysis is DC power source (PM3005B) (made in China). Cyclic voltammograms were obtained on a CHI 605E potentiostat. The anode electrode is graphite electrodes (Φ 6mm \times 80mm) and cathode electrode is graphite electrodes (Φ 6mm \times 80mm).

(b) General procedures for electrochemical oxidation of sulfides to prepare sulfoxides



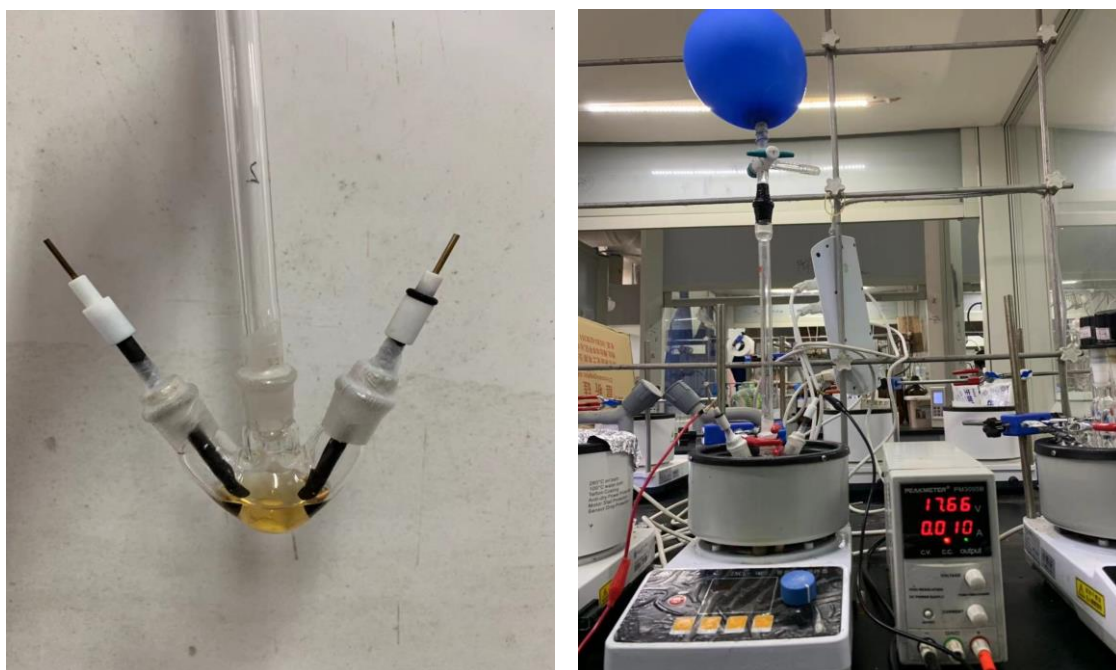
To an undivided three-necked bottle (10 mL) were added **1** (0.2 mmol), ${}^n\text{Bu}_4\text{NBF}_4$ (0.1 M) and DCM (4 mL). The bottle was equipped with graphite rod electrode as cathode and graphite rod electrode as anode under oxygen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 2 h until complete consumption of **1** as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with EtOAc (3 \times 10 mL). The combined organic layer was dried with Na_2SO_4 , filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **2**.

(c) General procedures for electrochemical oxidation of sulfides to prepare sulfones



To an undivided three-necked bottle (10 mL) were added **1** (0.2 mmol), ${}^n\text{Bu}_4\text{NBF}_4$ (0.1 M) and DCM (4 mL). The bottle was equipped with graphite rod electrode as cathode and graphite rod electrode as anode under oxygen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 8 h until complete consumption of **1** as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with EtOAc (3×10 mL). The combined organic layer was dried with Na_2SO_4 , filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.

(d) Experimental device



(e) Cyclic voltammograms device and Cyclic voltammogram analysis

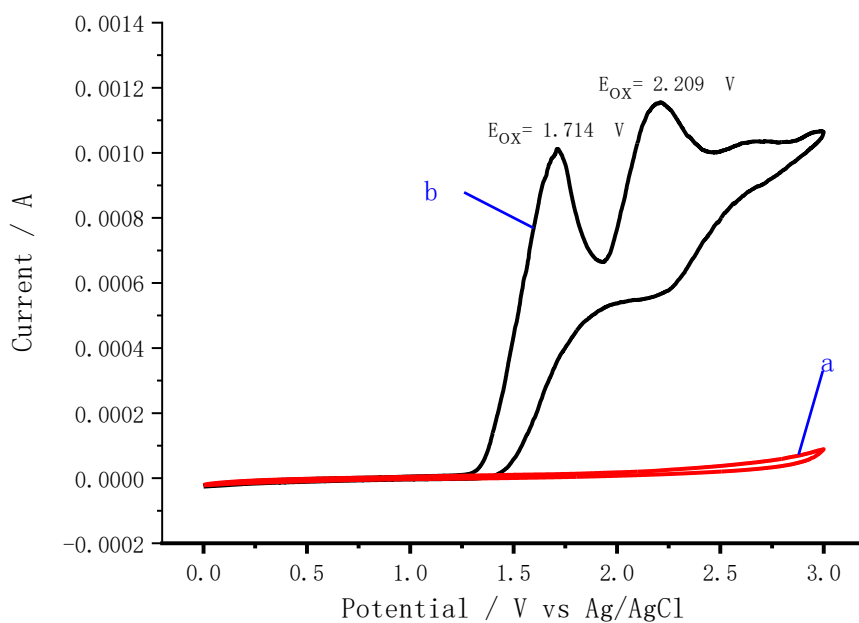
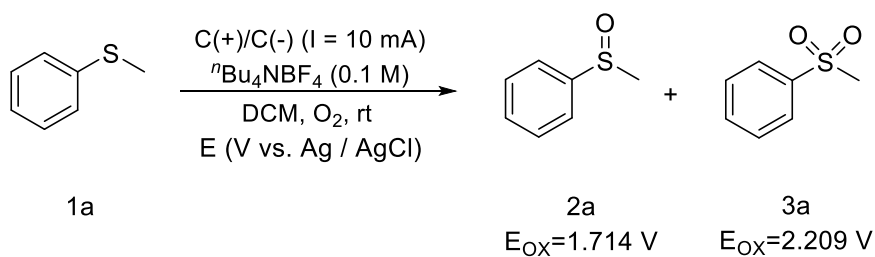
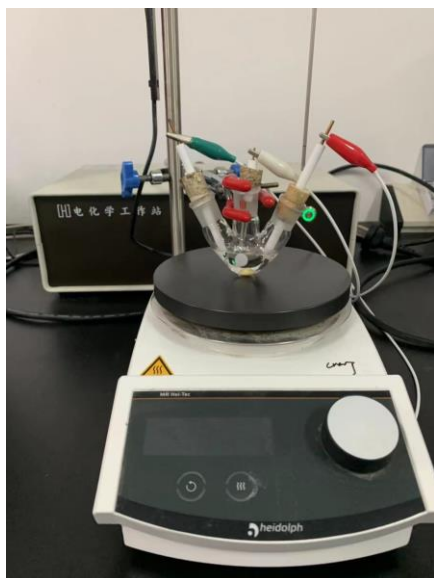
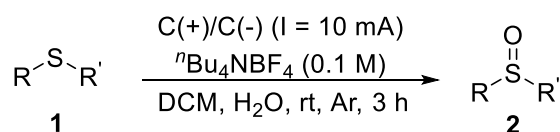


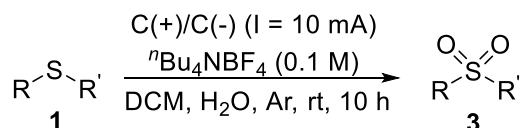
Figure S1. Cyclic voltammogram curves. Using GC disk as working electrode, Pt slice, and Ag/AgCl as counter and reference electrode at 100 mV/s scan rate. **Curve a:** ${}^n\text{Bu}_4\text{NBF}_4$ (0.1 M) and CH_3CN (6 mL); **Curve b:** 1a (0.05 M), ${}^n\text{Bu}_4\text{NBF}_4$ (0.1 M) and CH_3CN (6 mL).

(B) Water as oxygen source for selective synthesis of sulfoxides and sulfones



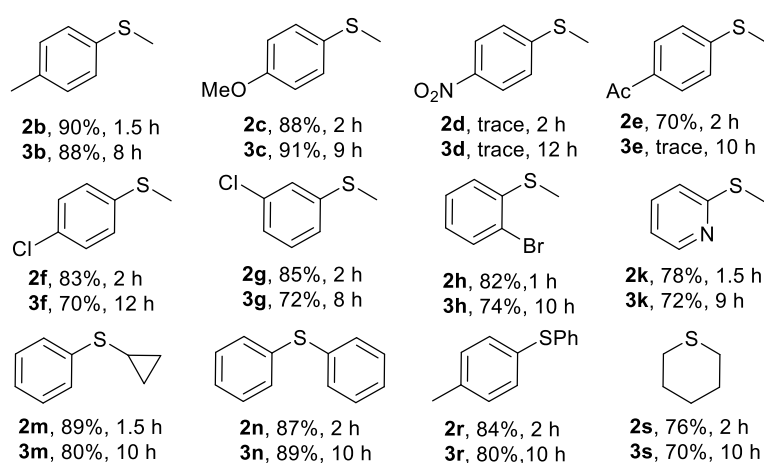
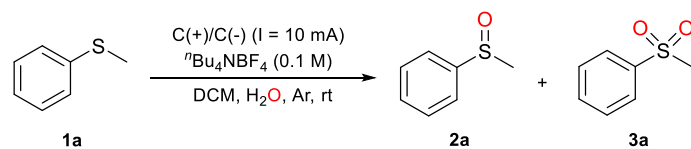
To an undivided three-necked bottle (10 mL) were added **1** (0.2 mmol), H₂O (30eq), ⁿBu₄NBF₄ (0.1 M) and DCM (4 mL). The bottle was equipped with graphite rod electrode as cathode and graphite rod electrode as anode under argon. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 3 h until complete consumption of **1** as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with EtOAc (3×10 mL). The combined organic layer was dried with Na₂SO₄, filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **2**.

(c) General procedures for electrochemical oxidation of sulfides to prepare sulfones



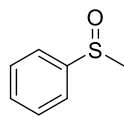
To an undivided three-necked bottle (10 mL) were added **1** (0.2 mmol), H₂O (30eq), ⁿBu₄NBF₄ (0.1 M) and DCM (4 mL). The bottle was equipped with graphite rod electrode as cathode and graphite rod electrode as anode under argon. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 10 h until complete consumption of **1** as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was extracted with

EtOAc (3×10 mL). The combined organic layer was dried with Na₂SO₄, filtered and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.



Reaction conditions: undivided cell, graphite rod anode, graphite plate cathode, constant current= 10 mA, **1a** (0.2 mmol), H₂O (30 equiv.), *n*Bu₄NBF₄ (0.1 M), DCM (4 ml), Ar (1 atm), room temperature.

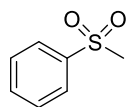
(C) Analytical data



(methylsulfinyl)benzene(2a)⁽⁵⁾

26.0 mg, 93% yield; Yellow oil;

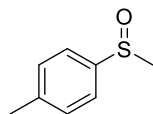
¹H NMR (500 MHz, CDCl₃) δ : 7.66 (d, J = 7.5 Hz, 2H), 7.55-7.51 (m, 3H), 2.73 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ : 145.7, 131.0, 129.3, 123.5, 43.9; LRMS (EI, 70eV) (*m/z*, %) 140 (M⁺, 90), 125 (100), 97 (77), 81 (8).



(methylsulfonyl)benzene (3a)⁽⁵⁾

28.1 mg, 90% yield; Yellow soild;

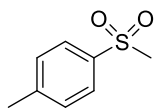
¹H NMR (500 MHz, CDCl₃) δ : 7.96 (d, J = 7.5 Hz, 2H), 7.67 (t, J = 7.5 Hz, 1H), 7.58 (t, J = 7.5 Hz, 2H), 3.06 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ : 140.5, 133.6, 130.9, 129.39, 127.3, 44.4; LRMS (EI, 70eV) (*m/z*, %) 156 (M⁺, 56), 141 (60), 94 (100), 89 (28).



1-methyl-4-(methylsulfinyl)benzene (2b)⁽⁶⁾

30.2 mg, 98% yield; Yellow soild;

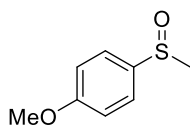
¹H NMR (500 MHz, CDCl₃) δ : 7.54 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 2.71 (s, 3H), 2.42 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ : 142.5, 141.5, 130.0, 123.5, 43.9, 21.3; LRMS (EI, 70eV) (*m/z*, %) 154 (M⁺, 63), 139 (100), 111 (16), 91 (38).



1-methyl-4-(methylsulfonyl)benzene (3b) ⁽⁶⁾

32.6 mg, 96% yield; Yellow solid;

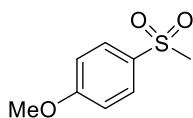
¹H NMR (500 MHz, CDCl₃) δ: 7.82 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 3.04 (s, 3H), 2.45 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 144.5, 137.6, 129.8, 127.2, 44.5, 21.5; LRMS (EI, 70eV) (*m/z*, %) 170 (M⁺, 30), 155 (28), 107 (29), 91 (100).



1-methoxy-4-(methylsulfonyl)benzene (2c) ⁽³⁾

29.9 mg, 88% yield; White solid;

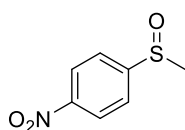
¹H NMR (500 MHz, CDCl₃) δ: 7.87 (d, J = 8.5 Hz, 2H), 7.03 (d, J = 8.5 Hz, 2H), 3.89 (s, 3H), 3.03 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 163.7, 132.3, 129.6, 114.5, 55.7, 44.9; LRMS (EI, 70eV) (*m/z*, %) 170 (M⁺, 26), 155 (100), 123 (13), 92 (8).



1-methoxy-4-(methylsulfonyl)benzene (3c) ⁽²⁾

33.5 mg, 90% yield; Colorless solid;

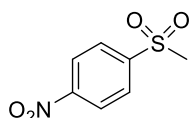
¹H NMR (500 MHz, CDCl₃) δ: 7.87 (d, J = 8.5 Hz, 2H), 7.03 (d, J = 9.0 Hz, 2H), 3.89 (s, 3H), 3.04 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 163.6, 132.1, 129.4, 114.39, 55.6, 44.7; LRMS (EI, 70eV) (*m/z*, %) 186 (M⁺, 78), 171 (100), 123 (64) 107 (70).



1-(methylsulfinyl)-4-nitrobenzene (2d) ⁽⁵⁾

35.2mg, 95% yield; Colorless solid ;

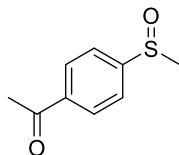
¹H NMR (500 MHz, CDCl₃) δ: 8.40 (d, J = 8.5 Hz, 2H), 7.85 (d, J = 8.5 Hz, 2H), 2.81 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 153.2, 149.5, 124.6, 124.4, 43.8; LRMS (EI, 70eV) (*m/z*, %) 185 (M⁺, 100), 170 (44), 140 (34), 112 (20).



1-(methylsulfonyl)-4-nitrobenzene (3d) ⁽⁵⁾

35.0 mg, 87% yield; Colorless solid ;

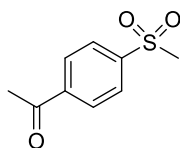
¹H NMR (500 MHz, CDCl₃) δ: 8.44 (d, J = 9.0 Hz, 2H), 8.17 (d, J = 9.0 Hz, 2H), 3.13 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ:150.8, 145.9, 128.9, 124.6, 44.3; LRMS (EI, 70eV) (*m/z*, %) 201 (M⁺, 18), 186 (24), 139 (100), 122 (54).



1-(4-(methylsulfinyl)phenyl)ethan-1-one (2e) ⁽³⁾

29.1 mg, 80% yield; White solid;

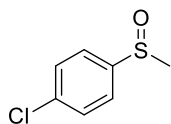
¹H NMR (500 MHz, CDCl₃) δ: 8.11 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 2H), 2.77 (s, 3H), 2.66 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 197.0, 150.9, 139.0, 129.1, 123.7, 43.8, 26.8; LRMS (EI, 70eV) (*m/z*, %) 182 (M⁺, 66), 167 (100), 152 (76), 139 (27).



1-(4-(methylsulfonyl)phenyl)ethan-1-one (3e) ⁽²⁾

32.9 mg, 83% yield; Colorless solid;

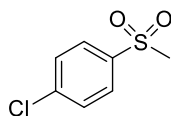
^1H NMR (500 MHz, CDCl_3) δ : 8.10 (m, 4H), 3.10 (s, 3H), 2.68 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 196.6, 144.1, 140.8, 129.1, 127.7, 44.2, 26.9; LRMS (EI, 70eV) (m/z , %) 198 (M^+ , 15), 183 (100), 121 (54), 91 (12).



1-chloro-4-(methylsulfinyl)benzene (2f)⁽³⁾

29.9 mg, 86% yield; Yellow solid;

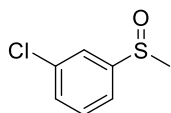
^1H NMR (500 MHz, CDCl_3) δ : 7.60 (d, $J = 8.5$ Hz, 2H), 7.52 (d, $J = 9.0$ Hz, 2H), 2.72 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 144.2, 137.2, 129.6, 124.9, 44.02, . LRMS (EI, 70eV) (m/z , %) 176 (M^{++2} , 48), 174 (M^+ , 58), 159 (100), 131 (46), 111 (26).



1-chloro-4-(methylsulfonyl)benzene (3f)⁽²⁾

30.4 mg, 80% yield; Colorless solid;

^1H NMR (500 MHz, CDCl_3) δ : 7.89 (d, $J = 8.5$ Hz, 2H), 7.56 (d, $J = 8.5$ Hz, 2H), 3.07 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 140.2, 138.9, 129.5, 128.7, 44.3; LRMS (EI, 70eV) (m/z , %) 192 (M^{++2} , 36) 190 (M^+ , 40), 175 (48), 127 (48), 111 (100).

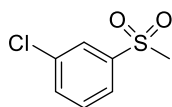


1-chloro-3-(methylsulfinyl)benzene (2g)⁽²⁾

31.3 mg, 90% yield; Yellow oil;

^1H NMR (500 MHz, CDCl_3) δ : 7.67 (s, 1H), 7.51-7.49 (m, 1H), 7.47 (d, $J = 5.0$ Hz,

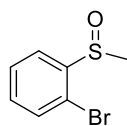
2H), 2.75 (s, 3H).; ^{13}C NMR (125 MHz, CDCl_3) δ : 147.8, 135.7, 131.2, 130.5, 123.6, 121.6, 44.0; LRMS (EI, 70eV) (m/z , %) 176 (M^{+2} , 60) 174 (M^{+} , 80), 159 (100), 131 (71) 111 (45).



1-chloro-3-(methylsulfonyl)benzene (3g) ⁽²⁾

29.6 mg, 78% yield; Yollow Soild;

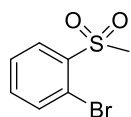
^1H NMR (500 MHz, CDCl_3) δ : 7.94 (s, 1H), 7.84 (d, $J = 8.0$ Hz, 1H), 7.64 (d, $J = 7.5$ Hz, 1H), 7.54 (t, $J = 7.5$ Hz, 1H), 3.08 (s, 3H).; ^{13}C NMR (125 MHz, CDCl_3) δ : 142.1, 135.5, 133.8, 130.7, 127.4, 125.4, 44.3; LRMS (EI, 70eV) (m/z , %) 190 (M^{+} , 43), 175 (36), 128 (54), 111 (100).



1-bromo-2-(methylsulfinyl)benzene (2h) ⁽²⁾

34.9 mg, 80% yield; Yollow oil;

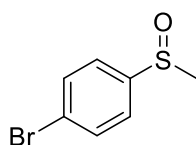
H NMR (500 MHz, CDCl_3) δ : 7,95 (d, $J = 7.5$ Hz 1H), 7.61-7.56 (m, 2H), 7.39 (t, $J = 6.5$ Hz 1H), 2.83 (s, 3H).; ^{13}C NMR (125 MHz, CDCl_3) δ : 145.3, 132.9, 132.2, 128.7, 125.6, 118.4, 41.9; LRMS (EI, 70eV) (m/z , %) 220 (M^{+2} , 88) 218 (M^{+} , 68), 205 (100), 139 (52), 96 (74).



1-bromo-2-(methylsulfonyl)benzene (3h) ⁽²⁾

36.5 mg, 78% yield; Yollow Soild;

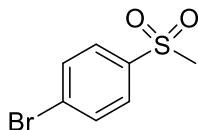
^1H NMR (500 MHz, CDCl_3) δ : 8.20 (d, $J = 7.5$ Hz, 1H), 7.78 (d, $J = 7.5$ Hz, 1H), 7.52 (m, 2H), 3.29 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 139.6, 135.4, 134.7, 131.1, 128.1, 120.6, 42.3; LRMS (EI, 70eV) (m/z , %) 236 ($\text{M}^+ + 2$, 56) 234 (M^+ , 74), 172 (52), 155 (100), 143 (22).



1-bromo-4-(methylsulfinyl)benzene (2i) ⁽³⁾

36.6 mg, 84%; White solid;

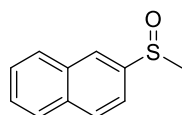
^1H NMR (500 MHz, CDCl_3) δ : 7.67 (d, $J = 8.5$ Hz, 2H), 7.53 (d, $J = 8.0$ Hz, 2H), 2.73 (s, 3H).; ^{13}C NMR (125 MHz, CDCl_3) δ : 144.7, 132.5, 125.4, 125.1, 43.8; LRMS (EI, 70eV) (m/z , %) 220 ($\text{M}^+ + 2$, 38) 218 (M^+ , 54), 205 (100), 175 (17), 96 (48).



1-bromo-4-(methylsulfonyl)benzene (3i) ⁽²⁾

37.4 mg, 80% yield; White solid ;

^1H NMR (500 MHz, CDCl_3) δ : 7.82 (d, $J = 8.5$ Hz, 2H), 7.72 (d, $J = 8.5$ Hz, 2H), 3.06 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 139.5, 132.6, 128.9, 128.9, 44.4. LRMS (EI, 70eV) (m/z , %) 236 ($\text{M}^+ + 2$, 48) 234 (M^+ , 54), 221 (52), 173 (56), 143 (100).

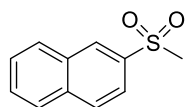


2-(methylsulfinyl)naphthalene (2j) ⁽³⁾

31.9 mg, 84% yield; Yellow solid;

^1H NMR (500 MHz, CDCl_3) δ : 8.22 (s, 1H), 7.99 (d, $J = 8.5$ Hz, 1H), 7.94 (t, $J = 5.5$

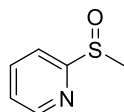
Hz 1H), 7.91 (t, J = 4.0 Hz 1H), 7.62-7.58 (m, 3H), 2.79 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 142.7, 134.4, 132.9, 129.6, 128.5, 128.0, 127.7, 127.3, 124.0, 119.4, 43.7; LRMS (EI, 70eV) (*m/z*, %) 190 (M⁺, 62), 175 (100), 147 (45), 115 (50).



2-(methylsulfonyl)naphthalene (3j) ⁽⁶⁾

21.4 mg, 52% yield; Yellow Solid;

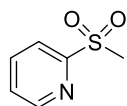
¹H NMR (500 MHz, CDCl₃) δ: 8.52 (s, 1H), 7.99 (t, J = 9.0 Hz, 2H), 7.93-7.89 (m, 2H), 7.67 (t, J = 7.0 Hz, 1H), 7.62 (t, J = 7.0 Hz, 1H), 3.12 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 137.4, 135.3, 132.2, 129.8, 129.4, 129.3, 129.0, 128.0, 127.8, 122.13, 44.6; LRMS (EI, 70eV) (*m/z*, %) 206 (M⁺, 54), 191 (18), 127 (100), 115 (30).



2-(methylsulfonyl)pyridine (2k) ⁽³⁾

21.3 mg, 76% yield; Yellow oil;

¹H NMR (500 MHz, CDCl₃) δ: 8.63 (d, J = 4.0 Hz, 1H), 8.04 (d, J = 8.0 Hz, 1H), 7.96 (t, J = 7.5 Hz, 1H), 7.41-7.38 (m, 1H), 2.86 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 165.9, 149.5, 138.1, 124.6, 119.2, 41.3; LRMS (EI, 70eV) (*m/z*, %) 141 (M⁺, 53), 125 (24), 93 (100), 80 (8).

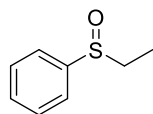


2-(methylsulfonyl)pyridine (3k) ⁽⁴⁾

23.4 mg, 75% yield; Yellow oil;

¹H NMR (500 MHz, CDCl₃) δ: 8.75 (d, J = 4.5 Hz, 1H), 8.10 (d, J = 7.5 Hz, 1H), 7.99

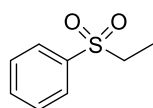
(t, J = 7.5 Hz, 1H), 7.60-7.57 (m, 1H), 3.25 (s, 3H). δ : 157.7, 149.9, 138.2, 127.4, 120.9, 39.9; LRMS (EI, 70eV) (m/z , %) 157 (M^+ , 2), 95 (80), 93 (100), 80 (2).



(ethylsulfinyl)benzene (2l) ⁽³⁾

27.1 mg, 88% yield; Colorless oil;

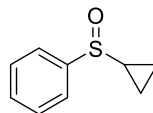
^1H NMR (500 MHz, CDCl_3) δ : 7.61 (d, J = 7.0 Hz, 2H), 7.52 (d, J = 9.0 Hz, 3H), 2.93-2.75 (m, 2H), 1.19 (d, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 143.2, 130.8, 129.1, 124.1, 50.2, 5.9; LRMS (EI, 70eV) (m/z , %) 154 (M^+ , 302), 126 (100), 97 (23), 81 (3).



(ethylsulfonyl)benzene (3l) ⁽⁵⁾

28.9 mg, 85% yield; Yellow oil;

^1H NMR (500 MHz, CDCl_3) δ : 7.91 (d, J = 7.5 Hz, 2H), 7.67 (t, J = 7.5 Hz, 1H), 7.58 (t, J = 8.0 Hz, 2H), 3.15-3.10 (m, 2H), 1.28 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 138.4, 133.6, 129.2, 128.1, 50.5, 7.3; LRMS (EI, 70eV) (m/z , %) 170 (M^+ , 53), 141 (35), 94 (100), 81 (1).

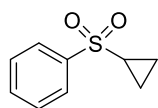


(cyclopropylsulfinyl)benzene (2m) ⁽³⁾

31.5 mg, 95% yield; Yellow oil;

^1H NMR (500 MHz, CDCl_3) δ : 7.68-7.66 (m, 2H), 7.54-7.50 (m, 3H), 2.29-2.24 (m, 1H), 1.27-1.22 (m, 2H), 1.06-1.01 (m, 1H), 0.98-0.92 (m, 1H); ^{13}C NMR (125 MHz,

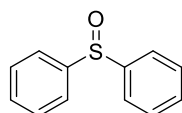
CDCl₃) δ : 144.8, 130.9, 129.1, 123.9, 33.7, 3.3, 2.7; LRMS (EI, 70eV) (*m/z*, %) 166 (M⁺, 26), 125 (100), 117 (40), 97 (27).



(cyclopropylsulfonyl)benzene (3m)⁽²⁾

31.3 mg, 86% yield; Yellow oil;

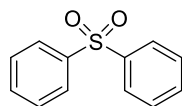
¹H NMR (500 MHz, CDCl₃) δ : 7.91-7.89 (m, 2H), 7.64 (t, J = 7.5 Hz, 1H), 7.56 (t, J = 8.0 Hz, 2H), 2.50-2.45 (m, 1H), 1.36-1.32 (m, 2H), 1.0-1.01 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ : 140.6, 133.3, 129.1, 127.4, 32.8, 5.8; LRMS (EI, 70eV) (*m/z*, %) 182 (M⁺, 55), 141 (100), 117 (46), 91 (16).



Sulfinyldibenzene (2n)⁽²⁾

37.2 mg, 92% yield; Yellow soild;

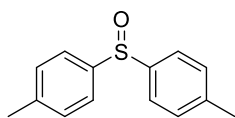
¹H NMR (500 MHz, CDCl₃) δ : 7.64 (d, J = 7.5 Hz, 4H), 7.43 (d, J = 6.5 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ : 145.4, 130.9, 129.2, 124.6; LRMS (EI, 70eV) (*m/z*, %) 202 (M⁺, 100), 173 (21), 154 (72), 109 (82).



Sulfonyldibenzene (3n)⁽¹⁾

39.2 mg, 90% yield; Yellow soild;

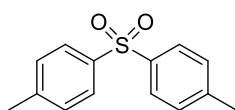
¹H NMR (500 MHz, CDCl₃) δ : 7.95 (d, J = 7.5 Hz, 4H), 7.56 (t, J = 7.0 Hz, 2H), 7.50 (t, J = 7.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ : 141.5, 133.1, 129.2, 127.6; LRMS (EI, 70eV) (*m/z*, %) 218 (M⁺, 24), 152 (5), 125 (100), 97 (22).



4,4'-sulfinylbis(methylbenzene) (2o)⁽²⁾

41.4 mg, 90% yield; White solid;

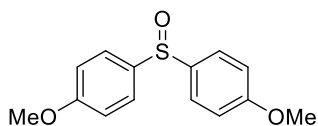
¹H NMR (500 MHz, CDCl₃) δ: 7.51 (d, J = 8.0 Hz, 4H), 7.24 (d, J = 8.0 Hz, 4H), 2.34 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ: 142.6, 141.3, 129.8, 124.7, 21.3; LRMS (EI, 70eV) (*m/z*, %) 230 (M⁺, 64), 182 (100), 123 (86), 91 (52).



4,4'-sulfonylbis(methylbenzene) (3o)⁽¹⁾

39.4 mg, 80% yield; White solid;

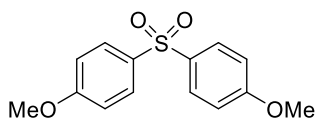
¹H NMR (500 MHz, CDCl₃) δ: 7.81 (d, J = 8.0 Hz, 4H), 7.28 (d, J = 8.0 Hz, 4H), 2.38 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ: 143.9, 139.0, 129.8, 127.5, 21.5; LRMS (EI, 70eV) (*m/z*, %) 246 (M⁺, 32), 139 (100), 107 (12), 91 (35).



4,4'-sulfinylbis(methoxybenzene) (2p)⁽²⁾

46.3 mg, 89% yield; Yellow solid;

¹H NMR (500 MHz, CDCl₃) δ: 7.53 (d, J = 8.5 Hz, 4H), 6.95 (d, J = 8.5 Hz, 4H), 3.80 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ: 161.7, 136.8, 126.7, 114.6, 55.4; LRMS (EI, 70eV) (*m/z*, %) 262 (M⁺, 17), 214 (100), 199 (48), 123 (68).

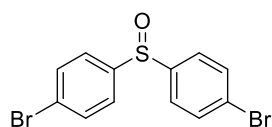


4,4'-sulfonylbis(methoxybenzene) (3p)⁽¹⁾

43.4 mg, 78% yield; Yellow solid;

^1H NMR (500 MHz, CDCl_3) δ : 8.10 (d, $J = 39.5$ Hz, 4H), 3.10 (s, 3H), 2.68 (s, 3H);

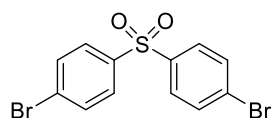
^{13}C NMR (125 MHz, CDCl_3) δ : 196.6, 144.1, 140.8, 129.1, 127.7, 44.2, 26.9; LRMS (EI, 70eV) (m/z , %) 278 (M^+ , 60), 155 (96), 123 (100), 107 (17).



4,4'-sulfinylbis(bromobenzene) (2q) ⁽²⁾

60.9 mg, 85% yield; White solid;

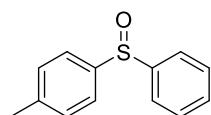
^1H NMR (500 MHz, CDCl_3) δ : 7.60-7.59 (m, 4H), 7.51-7.49 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 144.3, 132.6, 126.0, 125.8; LRMS (EI, 70eV) (m/z , %) 360 (M^+ , 38), 312 (88), 281 (82), 108 (100).



4,4'-sulfonylbis(bromobenzene) (3q) ⁽¹⁾

62.8 mg, 84% yield; Colorless solid;

^1H NMR (500 MHz, CDCl_3) δ : 7.80-7.77 (m, 4H), 7.65 (d, $J = 8.5$ Hz, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 140.2, 132.7, 129.2, 128.8; LRMS (EI, 70eV) (m/z , %) 376 (M^+ , 28), 205 (100), 167 (22), 149 (12).

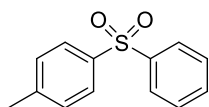


1-methyl-4-(phenylsulfinyl)benzene (2r) ⁽²⁾

37.2 mg, 86% yield; White solid;

^1H NMR (500 MHz, CDCl_3) δ : 7.63-7.61 (m, 2H), 7.53 (d, $J = 8.5$ Hz, 2H), 7.44-7.39

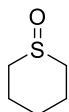
(m, 3H), 7.23 (d, J = 8.0 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 145.6, 142.3, 141.4, 130.7, 129.8, 129.1, 124.7, 124.4; LRMS (EI, 70eV) (m/z , %) 216 (M^+ , 100), 168 (92), 123 (70), 107 (74).



1-methyl-4-(phenylsulfonyl)benzene (3r)⁽¹⁾

38.5 mg, 83% yield; Colorless solid;

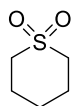
^1H NMR (500 MHz, CDCl_3) δ : 7.94-7.92 (m, 2H), 7.83 (d, J = 8.0 Hz, 2H), 7.54 (t, J = 7.5 Hz, 1H), 7.49 (t, J = 7.5 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 2.39 (s, 3H).; ^{13}C NMR (125 MHz, CDCl_3) δ : 144.1, 142.0, 138.6, 133.0, 129.9, 129.2, 127.7, 127.5, 21.5; LRMS (EI, 70eV) (m/z , %) 232 (M^+ , 76), 139 (96), 107 (100), 91 (38).



tetrahydro-2H-thiopyran 1-oxide (2s)⁽⁵⁾

18.4mg, 78% yield; Yellow Solid;

^1H NMR (500 MHz, CDCl_3) δ : 2.89 (t, J = 11.0 Hz, 2H), 2.78-2.74 (m, 2H), 2.26-2.23 (m, 2H), 1.68-1.65 (m, 2H), 1.61-1.58 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 48.8, 24.5, 18.9; LRMS (EI, 70eV) (m/z , %) 118 (M^+ , 100), 101 (34), 90 (24), 82 (2).

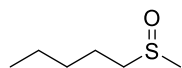


tetrahydro-2H-thiopyran 1,1-dioxide (3s)⁽⁵⁾

18.2 mg, 68% yield; White solid;

^1H NMR (500 MHz, CDCl_3) δ : 3.0 (t, J = 6.0 Hz, 4H), 2.12-2.07 (m, 4H), 1.67-1.64 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 52.1, 24.2, 23.7; LRMS (EI, 70eV) (m/z , %) 134

(M⁺, 100), 118 (38), 106 (32), 80 (14).



2-(methylsulfonyl)pyridine (2t)

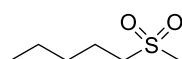
14.6 mg, 62% yield; Yellow oil;

¹H NMR (500 MHz, CDCl₃) δ: 2.77-2.72 (m, 1H), 2.67-2.61 (m, 1H), 2.57 (s, 3H), 1.80-1.73 (m, 2H), 1.48-1.42 (m, 2H), 1.40-1.37 (m, 2H), 0.92 (t, J = 7.0 Hz, 3H).;

¹³C NMR (125 MHz, CDCl₃) δ: 54.7, 38.5, 30.8, 22.2, 22.2, 13.7; LRMS (EI, 70eV)

(*m/z*, %) 135(M⁺, 2), 117 (100), 103 (2), 80 (1); HRMS *m/z* (ESI) for calcd C₆H₁₄OS

[M+H]⁺ 135.0838, found 135.0838.



1-(methylsulfonyl)pentane (3t)

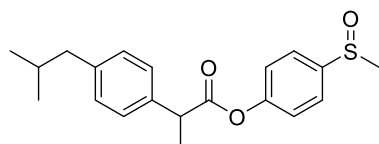
16.0 mg, 60% yield; Yellow oil;

¹H NMR (500 MHz, CDCl₃) δ: ¹H NMR (500 MHz, CDCl₃) : 3.03-3.00 (m, 2H), 2.90 (s, 3H), 1.88-1.82 (m, 2H), 1.47-1.41 (m, 2H), 1.40-1.34 (m, 2H), 0.93 (t, J = 7.5

Hz, 3H).; ¹³C NMR (125 MHz, CDCl₃) : 54.6, 40.3, 30.3, 22.0, 22.0, 13.6; LRMS (EI,

70eV) (*m/z*, %) 151 (M⁺, 2), 135 (13), 94 (16), 81 (100); HRMS *m/z* (ESI) for calcd

C₆H₁₄O₂S [M+H]⁺ 151.0787, found 151.0787.

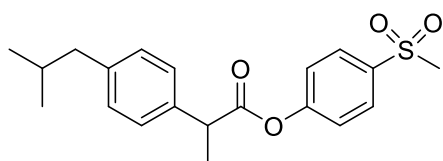


4-(methylsulfonyl)phenyl 2-(4-isobutylphenyl)propanoate (4a)

51.6 mg, 75% yield; Yellow solid; mp: 169.0-171.2 °C (uncorrected);

¹H NMR (500 MHz, CDCl₃) δ: 7.63 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H),

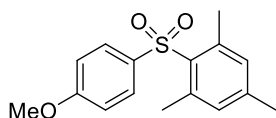
7.18-7.14 (m, 4H), 3.97-3.93 (m, 1H), 2.70 (s, 3H), 2.47 (d, J = 7.0 Hz, 2H), 1.89-1.84 (m, 1H), 1.61 (d, J = 7.0 Hz, 3H), 0.91 (d, J = 6.5 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ: 172.8, 152.9, 142.6, 141.0, 136.7, 129.6, 127.1, 124.8, 122.6, 45.2, 45.0, 44.0, 30.1, 22.3, 18.4; LRMS (EI, 70eV) (*m/z*, %) 344 (M⁺, 8), 188 (32), 161 (100), 140 (24); HRMS *m/z* (ESI) for calcd C₂₀H₂₄O₃S [M+H]⁺ 345.1519, found 345.1518.



4-(methylsulfonyl)phenyl 2-(4-isobutylphenyl)propanoate (4b)

29.5 mg, 41% yield; White solid; mp; 193.2-195.0 °C (uncorrected);

¹H NMR (500 MHz, CDCl₃) δ: 7.63 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.18-7.14 (m, 4H), 3.97-3.93 (m, 1H), 2.70 (s, 3H), 2.47 (d, J = 7.0 Hz, 2H), 1.89-1.84 (m, 1H), 1.61 (d, J = 7.0 Hz, 3H), 0.91 (d, J = 6.5 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ: 172.8, 152.9, 142.6, 141.0, 136.7, 129.6, 127.1, 124.8, 122.6, 45.2, 45.0, 44.0, 30.1, 22.3, 18.4; LRMS (EI, 70eV) (*m/z*, %) 360 (M⁺, 1), 161 (100), 117 (16), 91 (10); HRMS *m/z* (ESI) for calcd C₂₀H₂₄O₄S [M+H]⁺ 361.1468, found 361.1464.

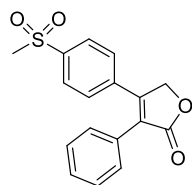


2-((4-methoxyphenyl)sulfonyl)-1,3,5-trimethylbenzene (4c)

37.7 mg, 65% yield; Yellow oil;

¹H NMR (500 MHz, CDCl₃) δ: 7.73 (d, J = 8.5 Hz, 2H), 6.93 (d, J = 7.0 Hz, 4H), 3.83 (s, 3H), 2.60 (s, 6H), 2.28 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 162.7, 143.0,

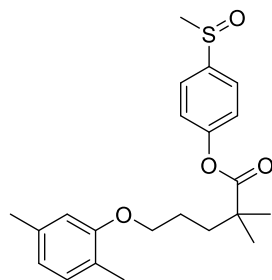
139.7, 135.2, 134.5, 132.1, 128.4, 114.0, 55.6, 22.8, 20.9; LRMS (EI, 70eV) (*m/z*, %) 290 (M^+ , 46), 272 (84), 165 (100), 91 (82).



4-(4-(methylsulfonyl)phenyl)-3-phenylfuran-2(5H)-one (4d)⁽⁴⁾

35.8 mg, 57% yield; White solid;

¹H NMR (500 MHz, CDCl₃) δ : 7.92 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.40 (s, 5H), 5.20 (s, 2H), 3.08 (s, 3H).; ¹³C NMR (125 MHz, CDCl₃) δ : 172.5, 153.4, 141.9, 136.2, 129.5, 129.1, 129.1, 129.0, 129.0, 128.5, 128.1, 70.3, 44.3; LRMS (EI, 70eV) (*m/z*, %) 314 (M^+ , 48), 257 (68), 131 (100), 103 (30).

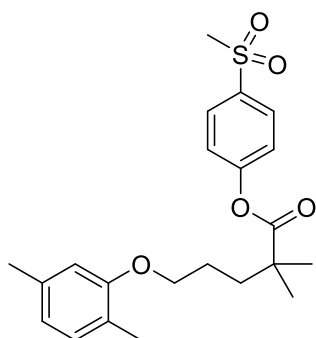


4-(methylsulfinyl)phenyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4e)

47.3 mg, 61% yield; Brown oil;

¹H NMR (500 MHz, CDCl₃) δ : 7.65 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 7.0 Hz, 1H), 6.67 (d, *J* = 7.0 Hz, 1H), 6.62 (s, 1H), 3.99 (t, *J* = 5.5 Hz, 2H), 2.72 (s, 3H), 2.30 (s, 3H), 2.17 (s, 3H), 1.92-1.86 (m, 4H), 1.38 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ : 176.0, 156.8, 153.1, 142.7, 136.5, 130.4, 124.9, 123.6, 122.8, 120.9, 112.0, 67.6, 44.2, 42.6, 37.1, 25.3, 25.1, 15.8; LRMS (EI, 70eV) (*m/z*, %) 388 (M^+ , 1), 272 (13), 233 (15), 83 (100); HRMS *m/z* (ESI) for calcd C₂₂H₂₈O₄S [$M+H$]⁺

389.1781, found 389.1786.



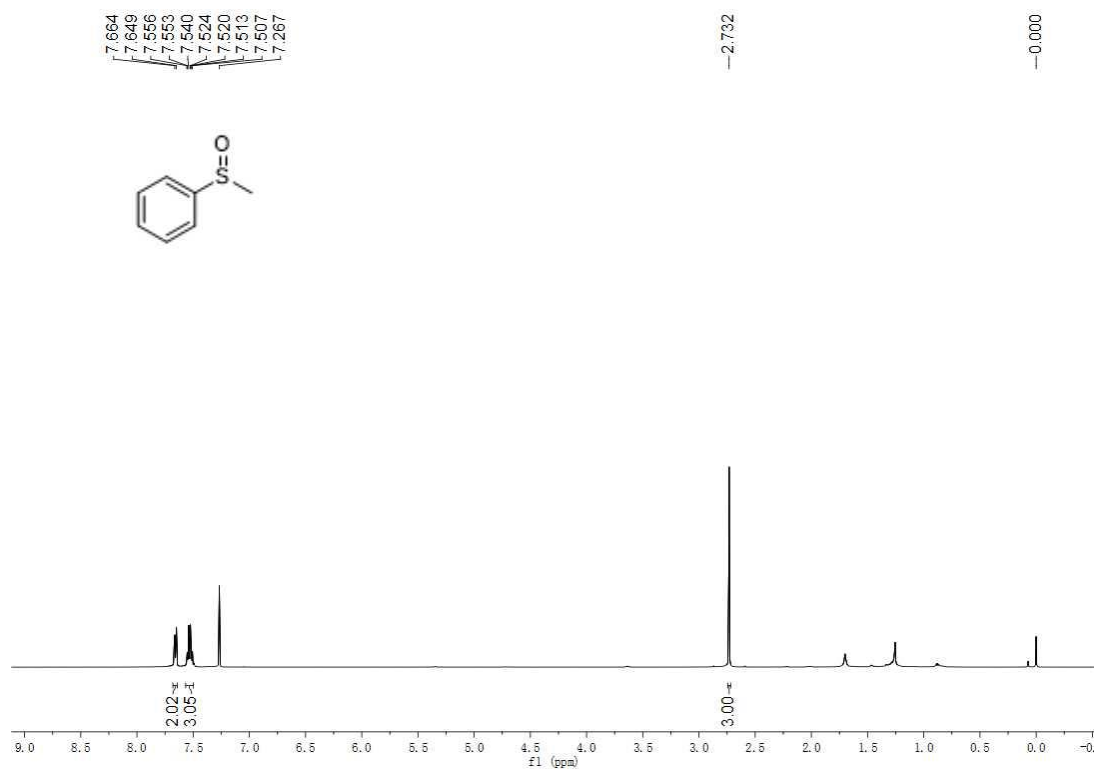
4-(methylsulfonyl)phenyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4f)

29.1 mg, 36% yield, Brown oil;

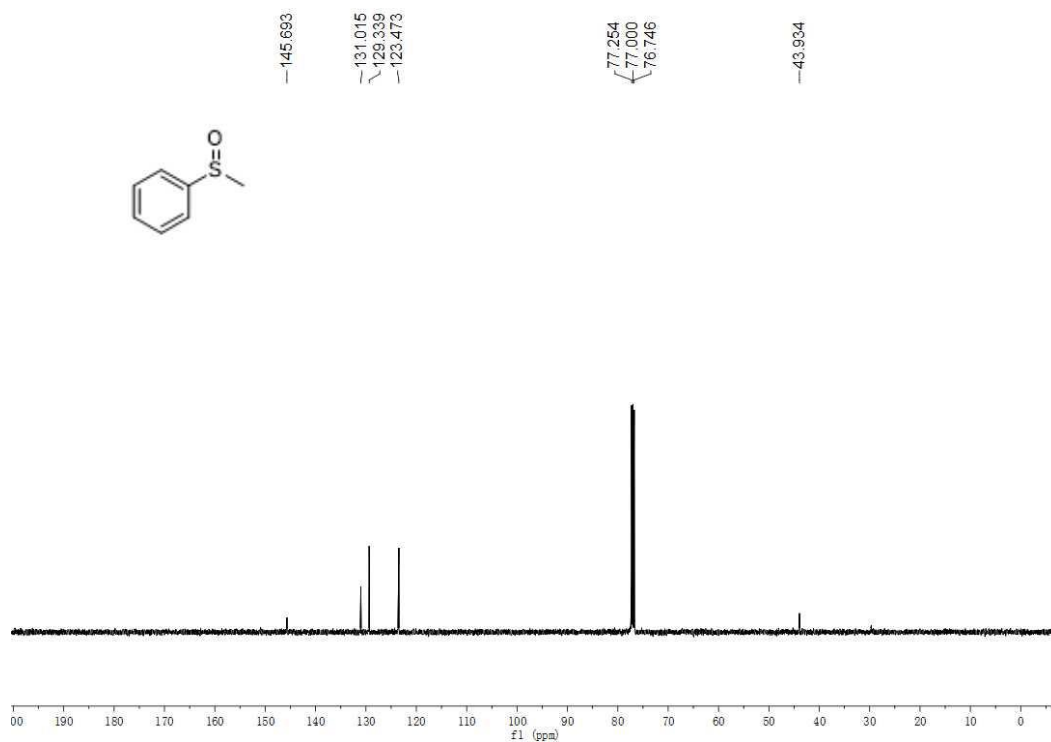
^1H NMR (500 MHz, CDCl_3) δ : 7.95 (d, $J = 8.0$ Hz, 2H), 7.23 (d, $J = 8.5$ Hz, 2H), 7.00 (d, $J = 7.5$ Hz, 1H), 6.67 (d, $J = 7.5$ Hz, 1H), 6.62 (s, 1H), 3.99 (t, $J = 5.5$ Hz, 2H), 3.05 (s, 3H), 2.30 (s, 3H), 2.16 (s, 3H), 1.92-1.84 (m, 4H), 1.39 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ : 175.6, 156.66, 155.06, 137.6, 136.4, 130.3, 129.0, 123.4, 122.6, 120.8, 111.8, 67.4, 44.5, 42.6, 36.9, 25.1, 25.0, 21.3, 15.7; LRMS (EI, 70eV) (m/z , %) 404 (M^+ , 1), 283 (30), 122 (11), 83 (100); HRMS m/z (ESI) for calcd $\text{C}_{22}\text{H}_{28}\text{O}_5\text{S}$ $[\text{M}+\text{H}]^+$ 405.1730, found 405.1728.

(D) Spectra

(methylsulfinyl)benzene (2a)

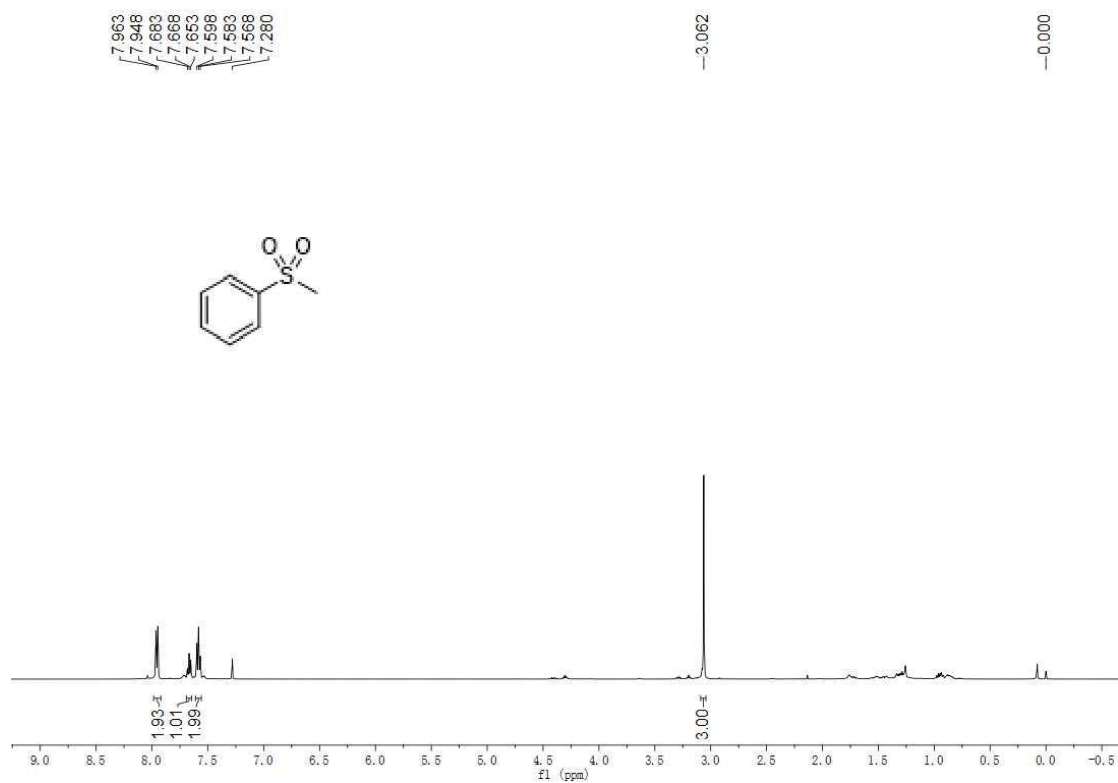


¹H NMR (500 MHz, CDCl₃)

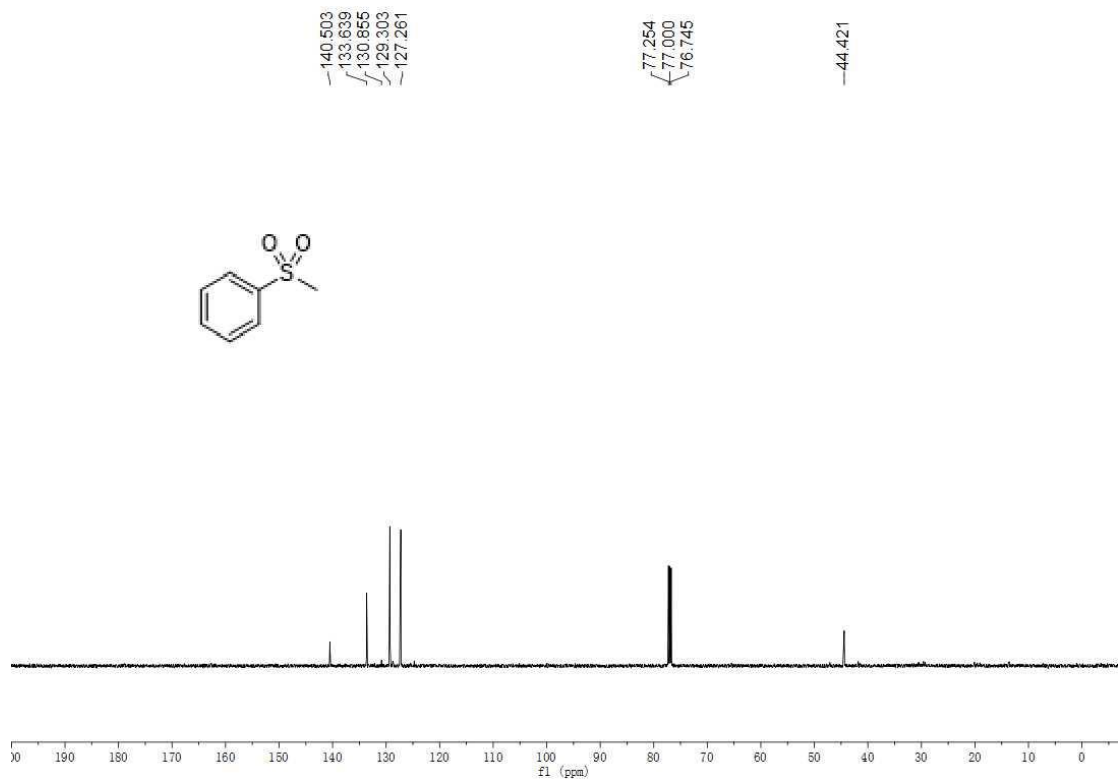


¹³C NMR (125 MHz, CDCl₃)

(methylsulfonyl)benzene (3a)

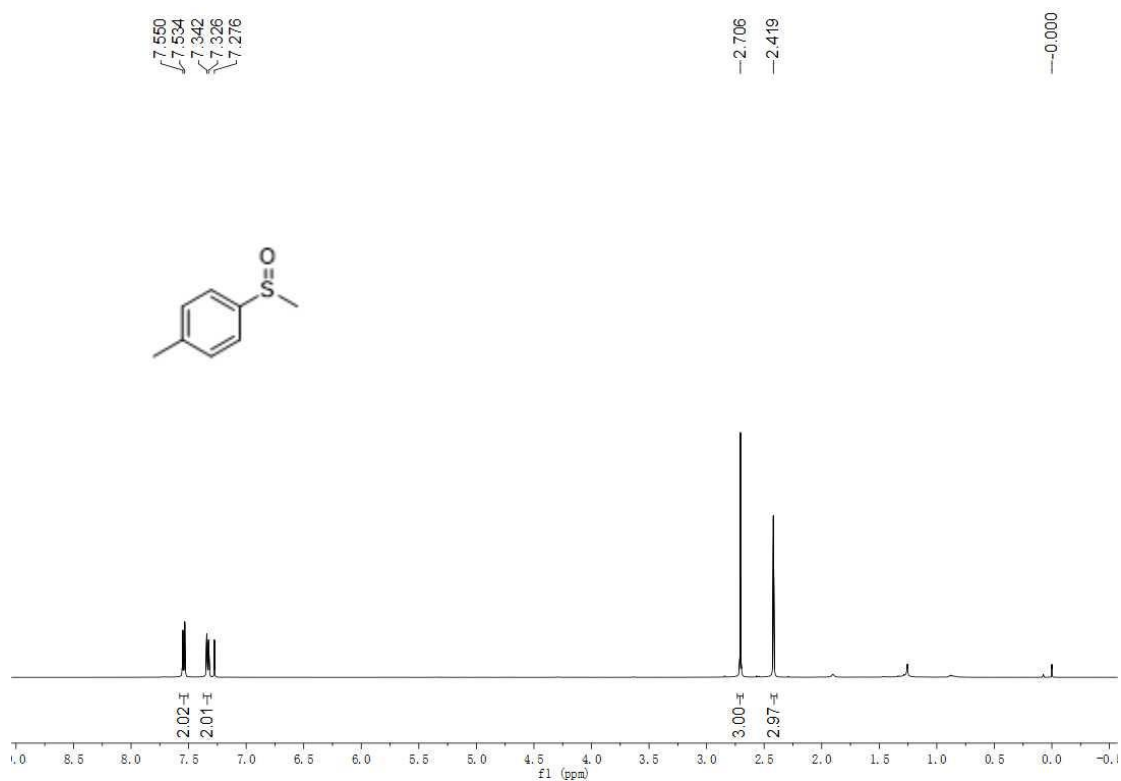


¹H NMR (500 MHz, CDCl₃)

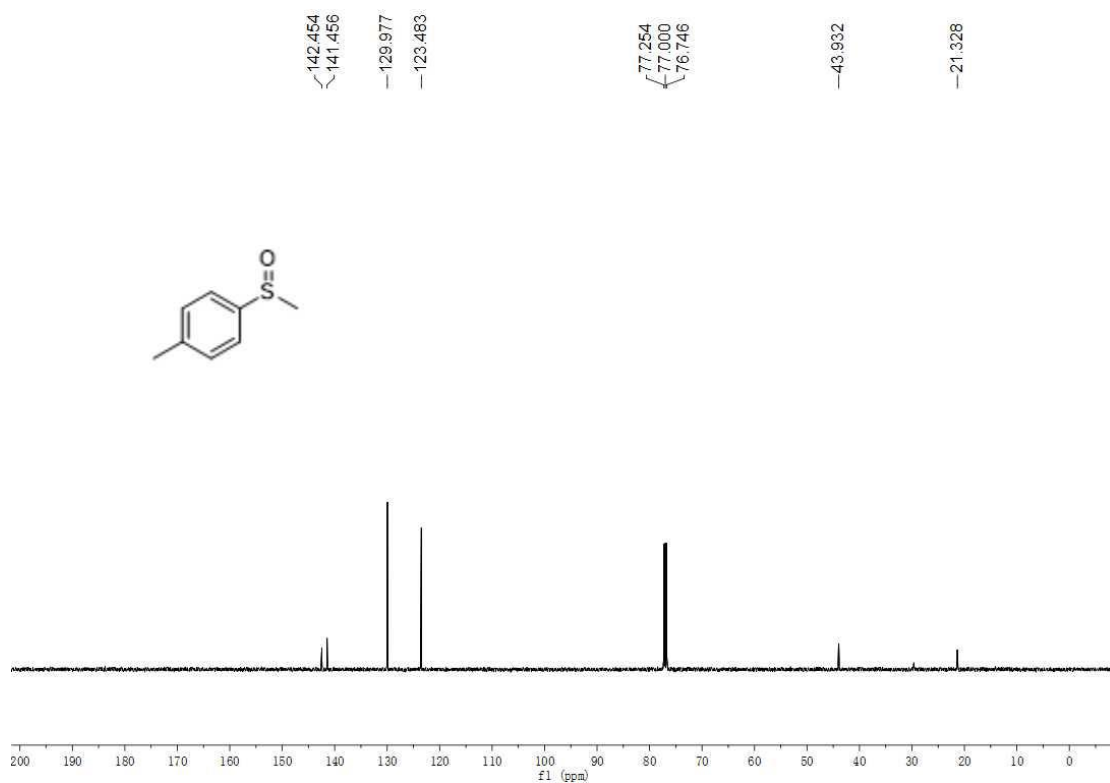


¹³C NMR (125 MHz, CDCl₃)

1-methyl-4-(methylsulfinyl)benzene (2b)

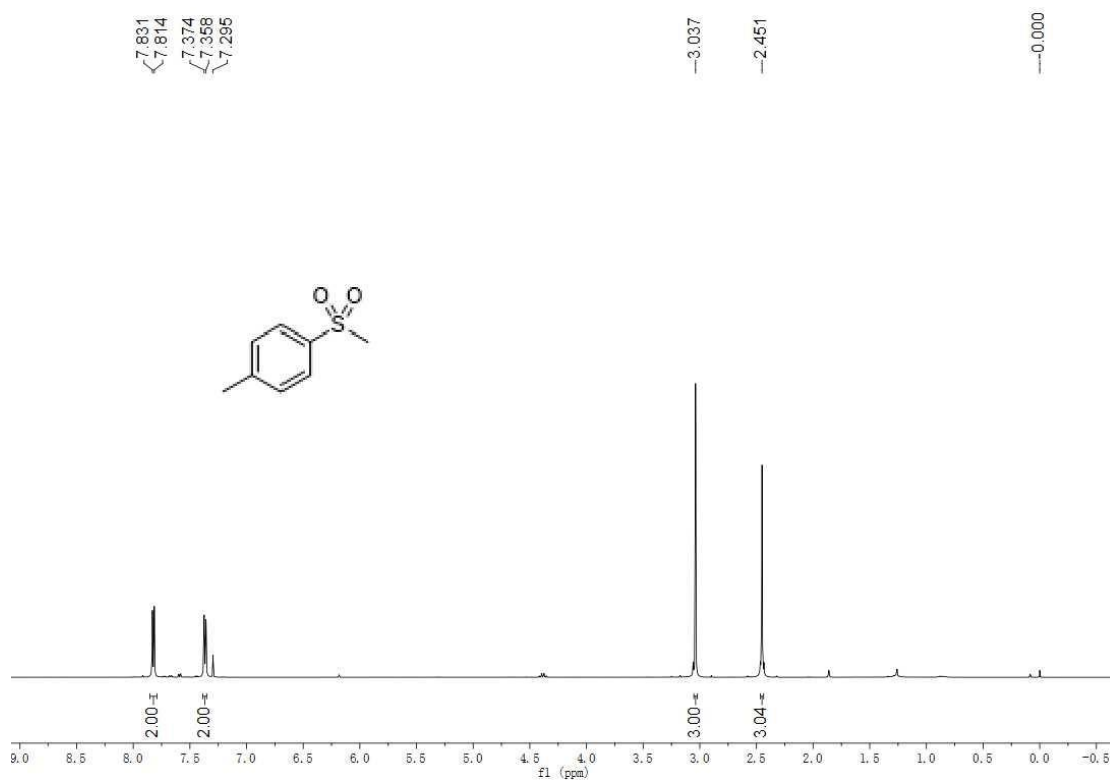


¹H NMR (500 MHz, CDCl₃)

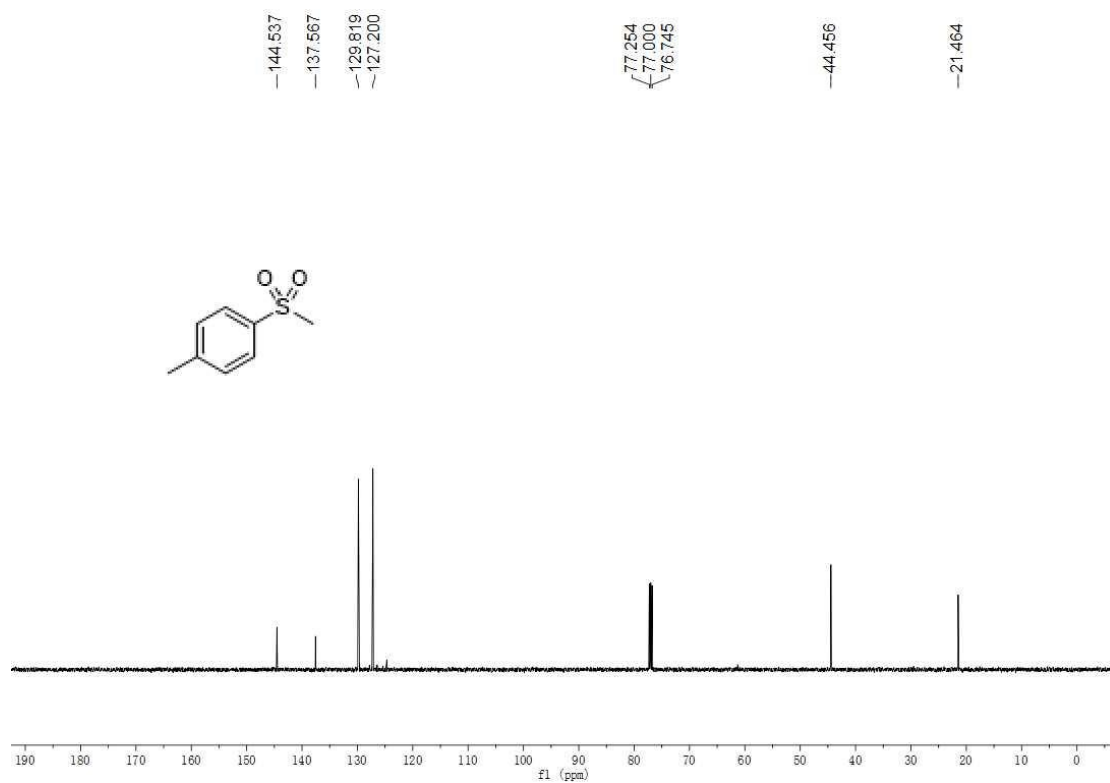


¹³C NMR (125 MHz, CDCl₃)

1-methyl-4-(methylsulfonyl)benzene (3b)

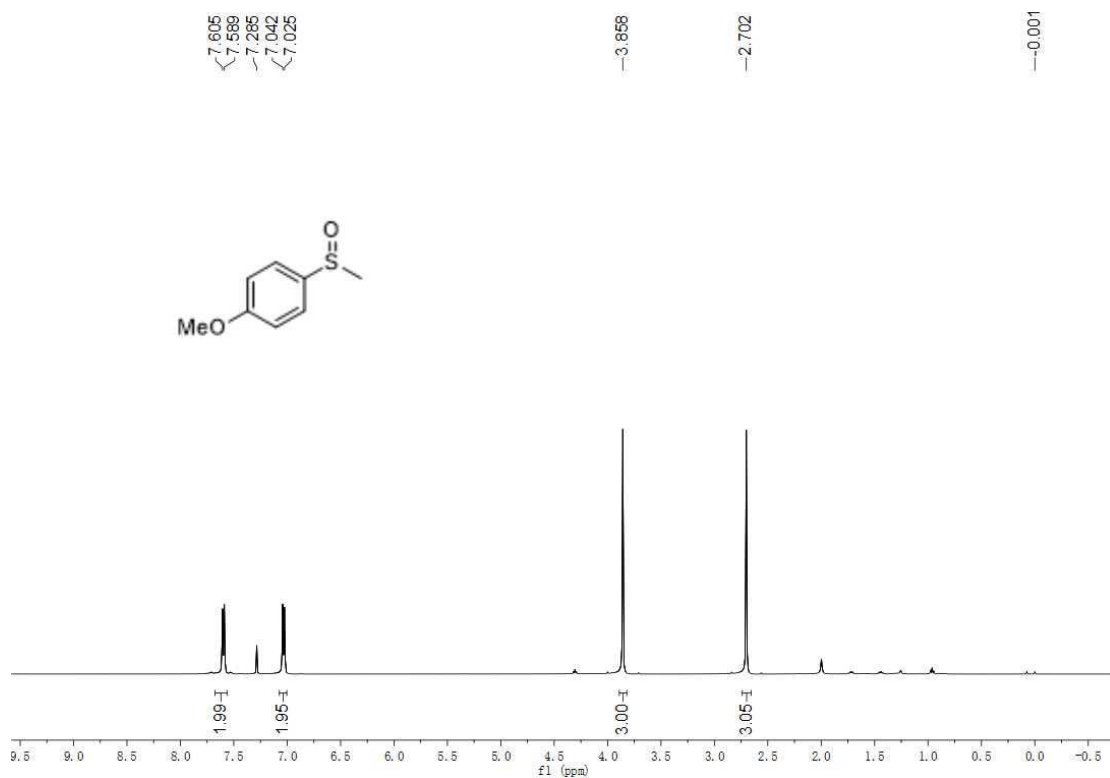


¹H NMR (500 MHz, CDCl₃)

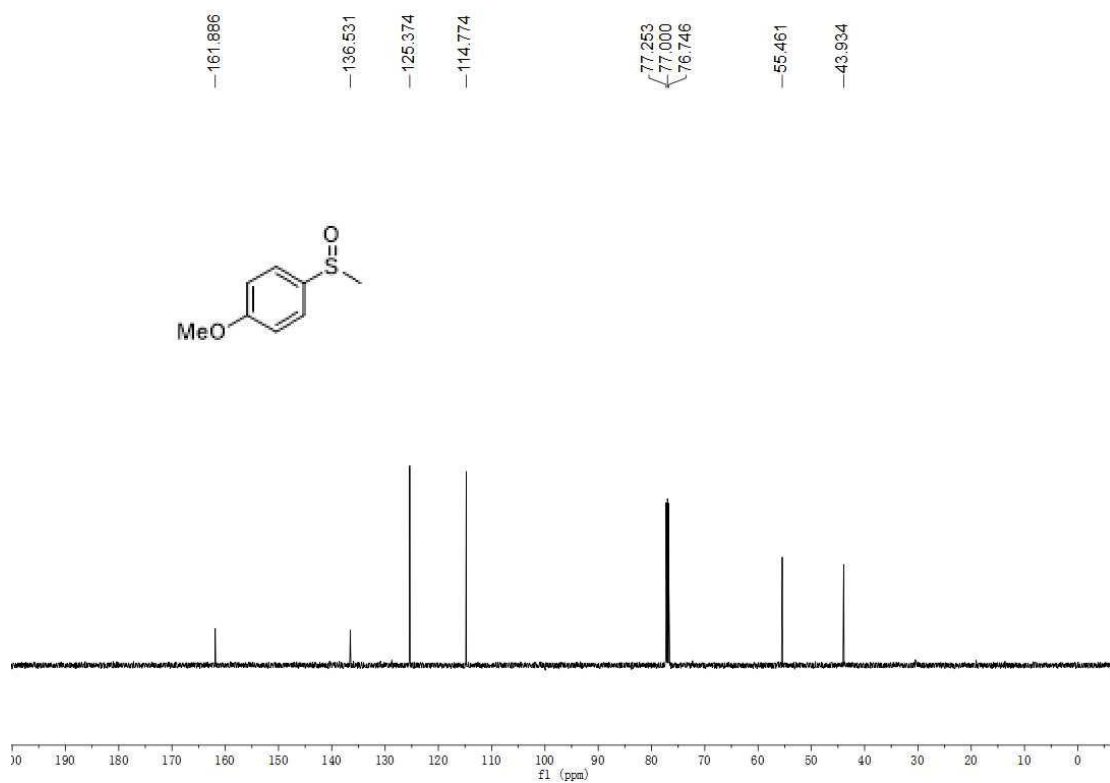


¹³C NMR (125 MHz, CDCl₃)

1-methoxy-4-(methylsulfinyl)benzene (2c)

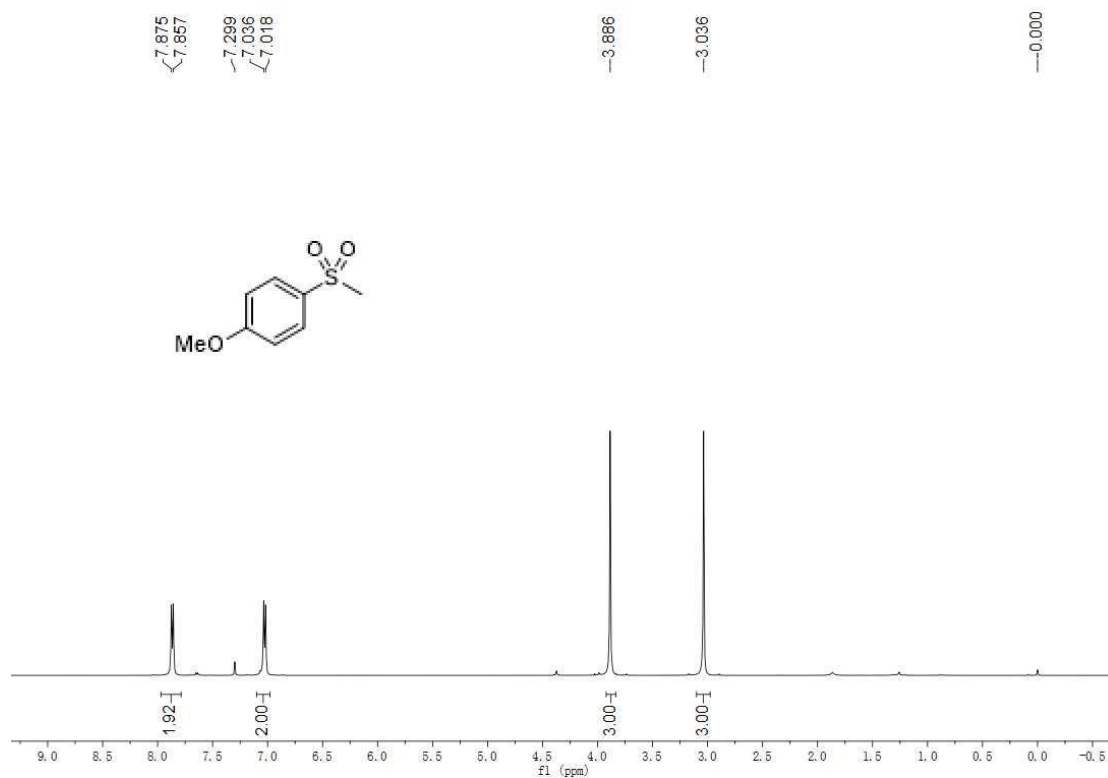


¹H NMR (500 MHz, CDCl₃)

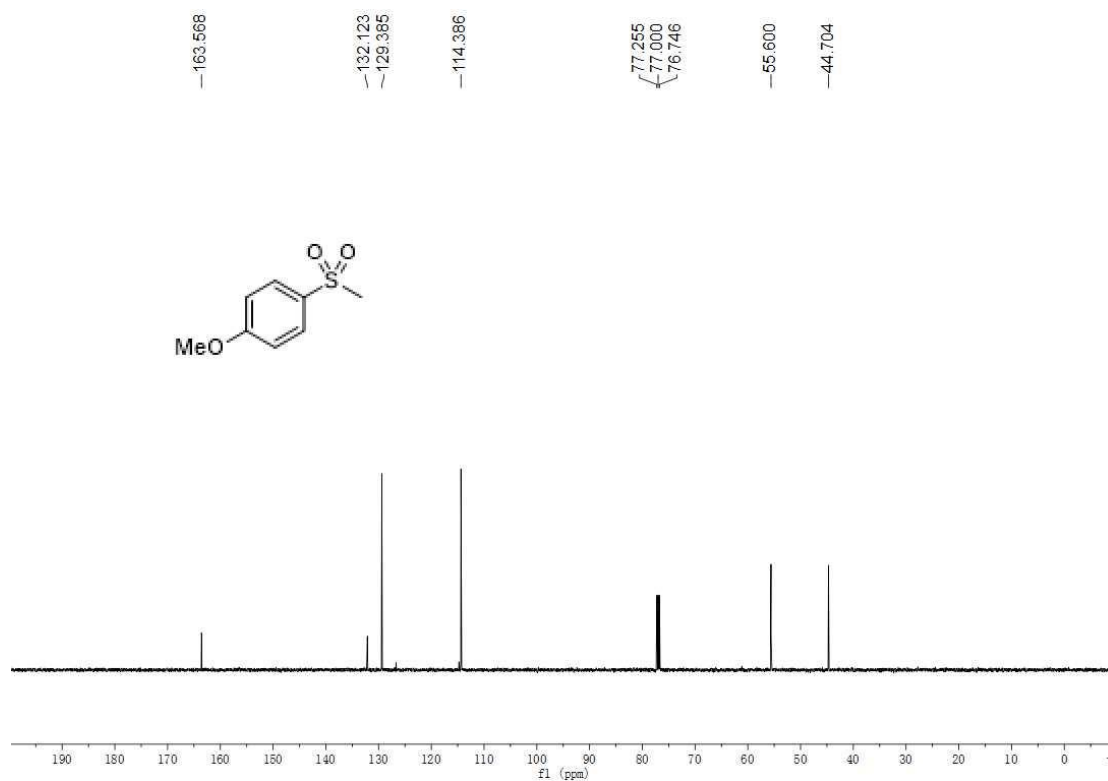


¹³C NMR (125 MHz, CDCl₃)

1-methoxy-4-(methylsulfonyl)benzene (3c)

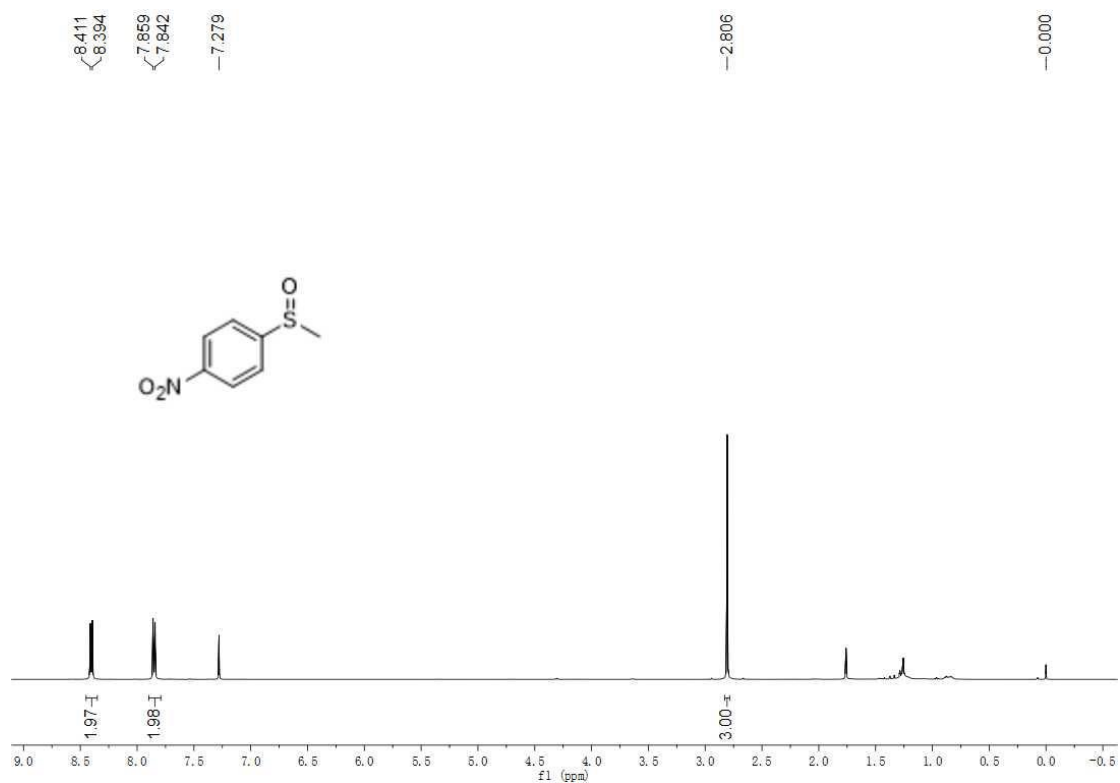


¹H NMR (500 MHz, CDCl₃)

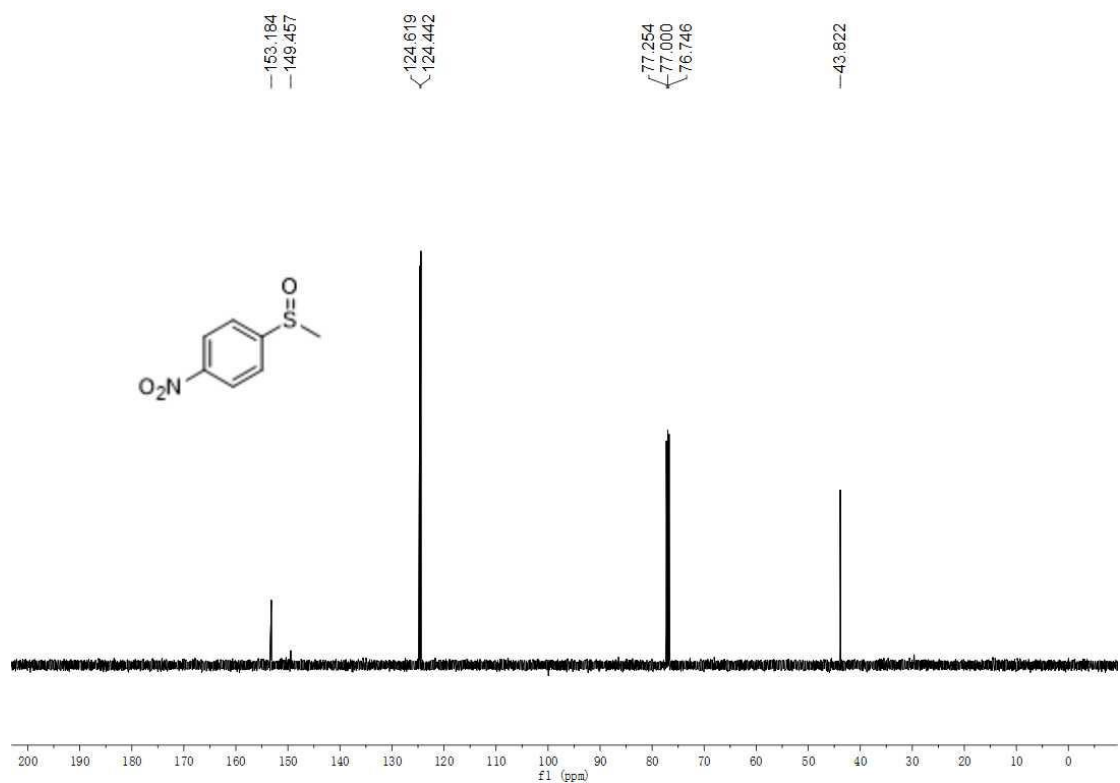


¹³C NMR (125 MHz, CDCl₃)

1-(methylsulfinyl)-4-nitrobenzene (2d)

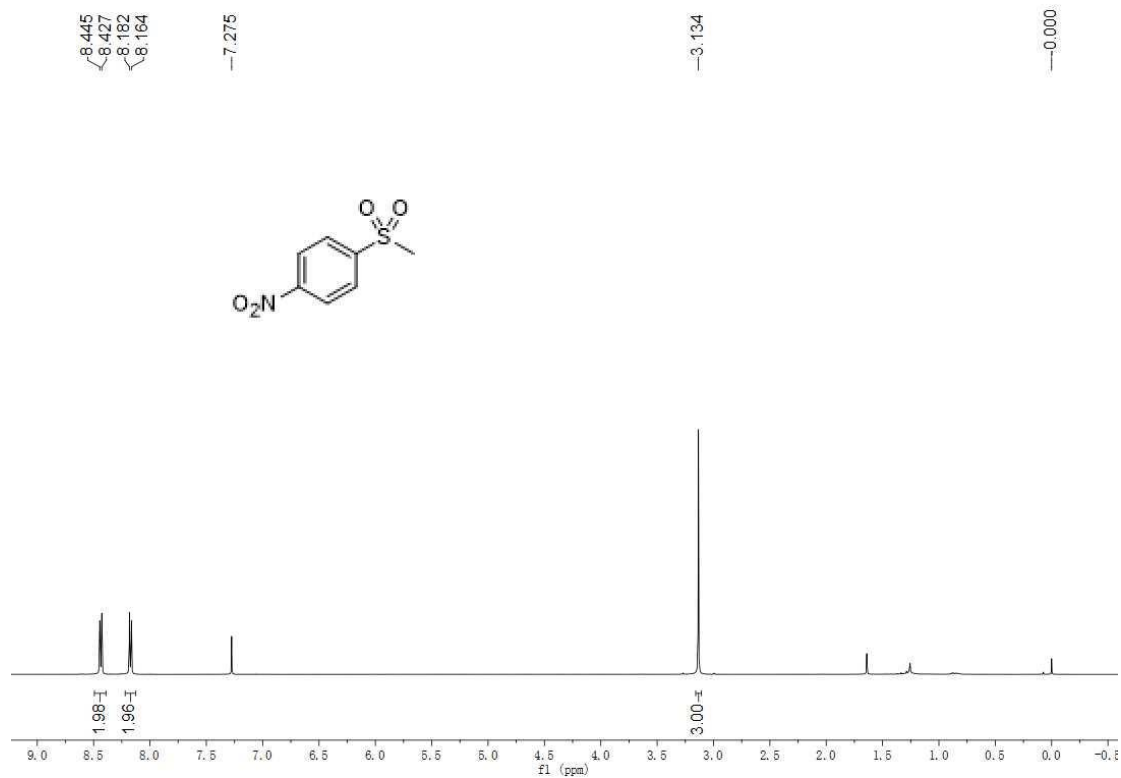


¹H NMR (500 MHz, CDCl₃)

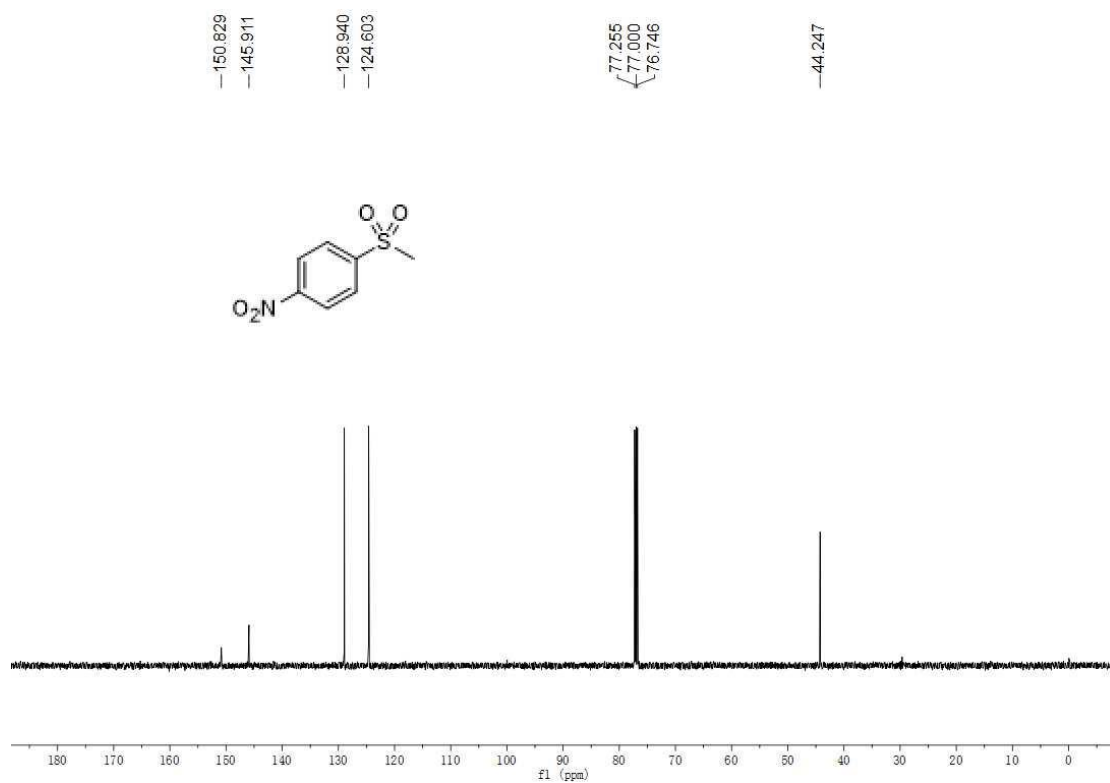


¹³C NMR (125 MHz, CDCl₃)

1-(methylsulfonyl)-4-nitrobenzene (3d)

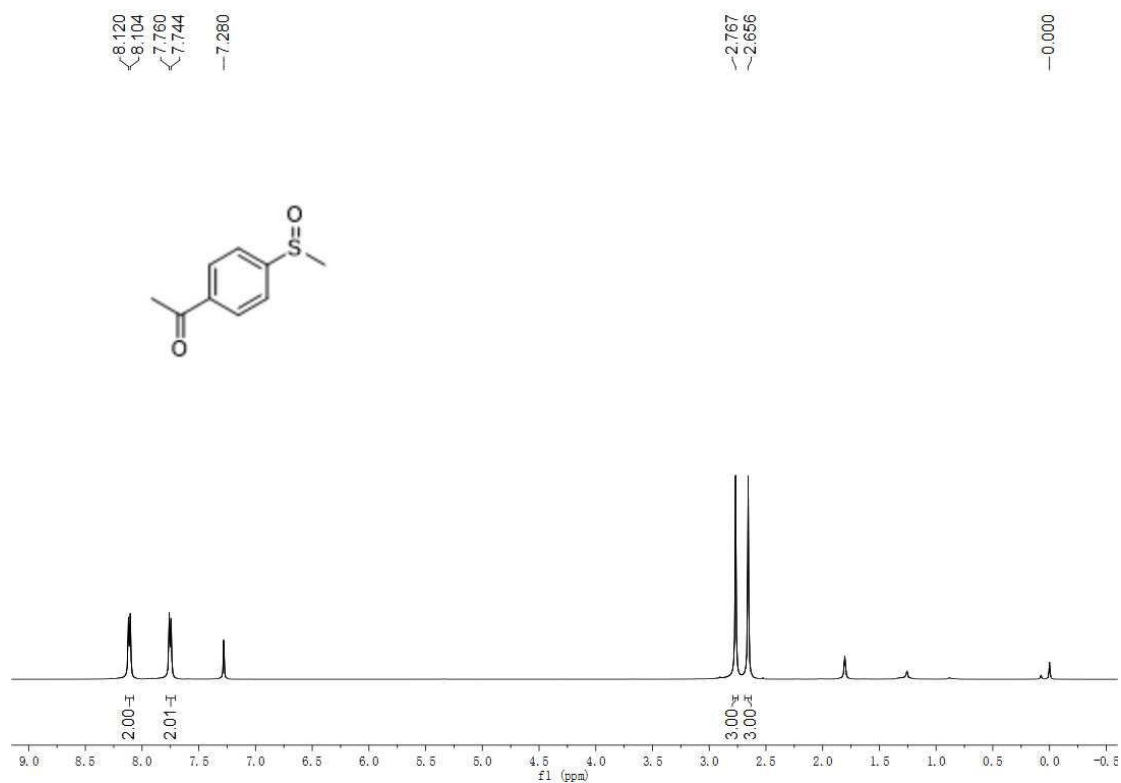


¹H NMR (500 MHz, CDCl₃)

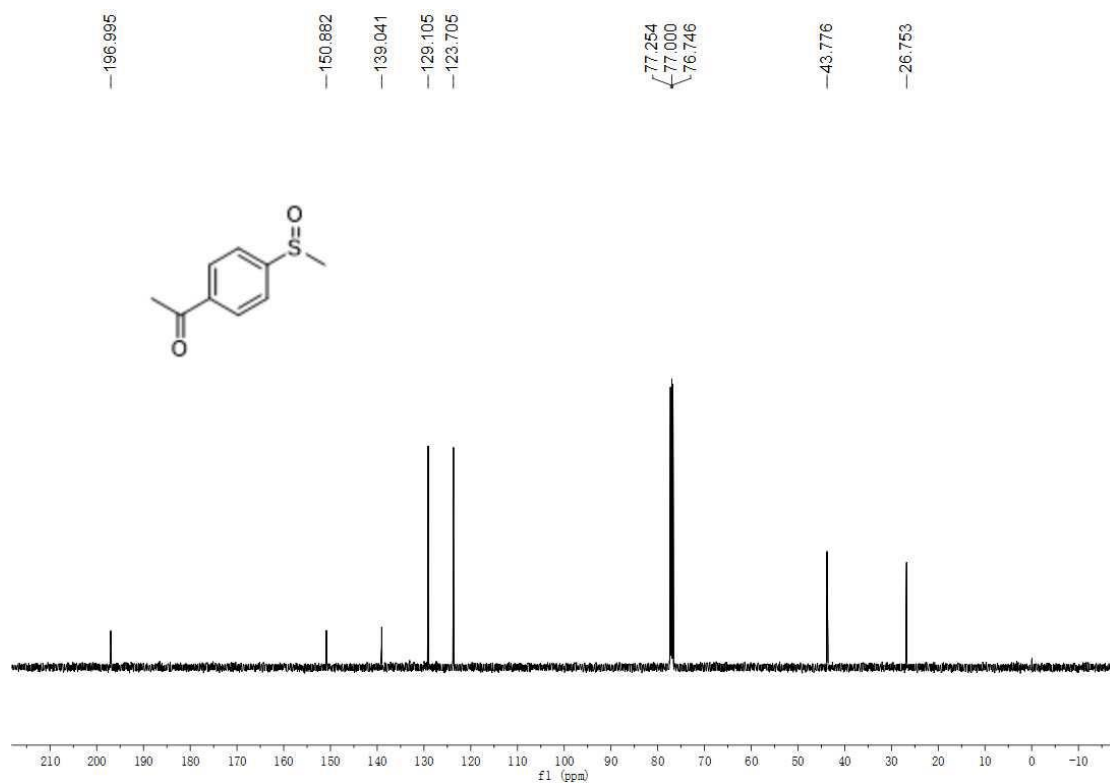


¹³C NMR (125 MHz, CDCl₃)

1-(methylsulfonyl)-4-nitrobenzen (2e)

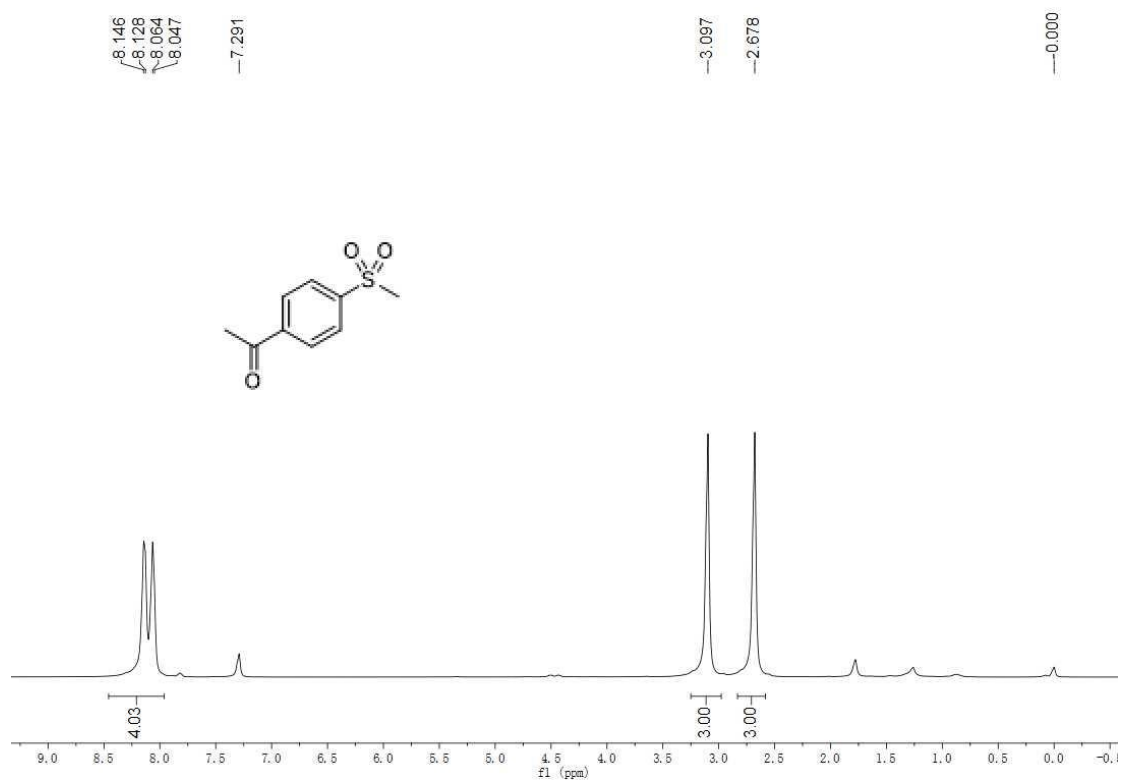


¹H NMR (500 MHz, CDCl₃)

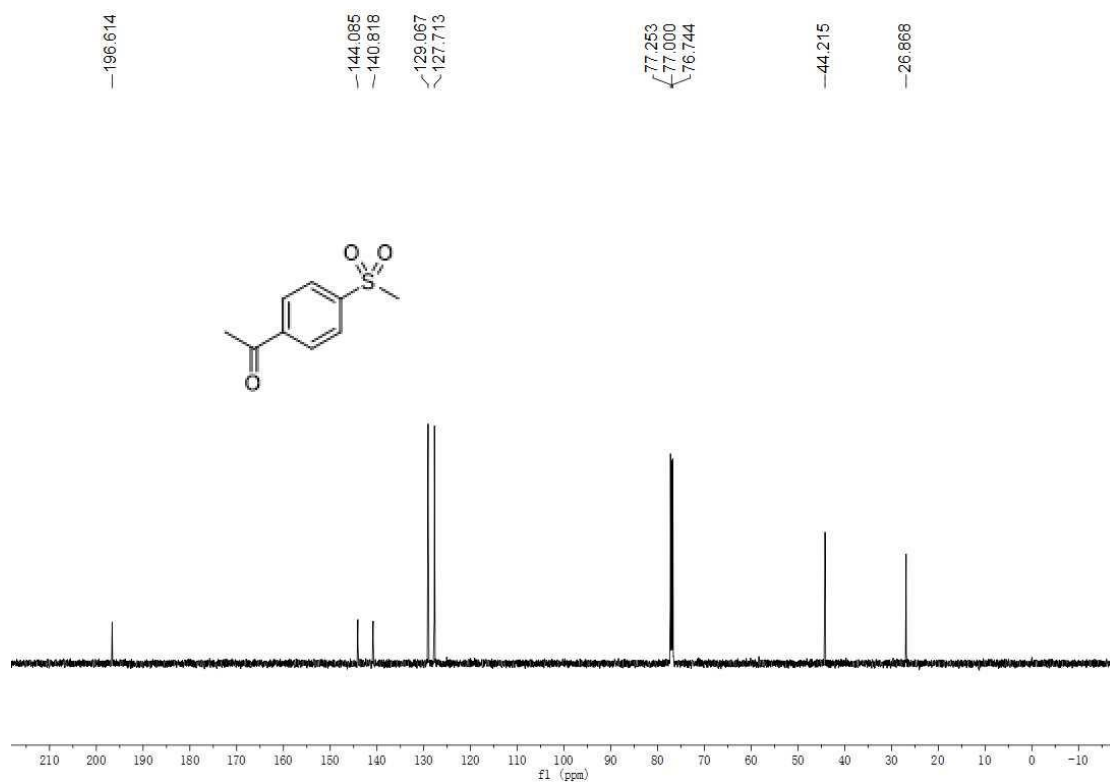


¹³C NMR (125 MHz, CDCl₃)

1-(4-(methylsulfonyl)phenyl)ethan-1-one (3e)

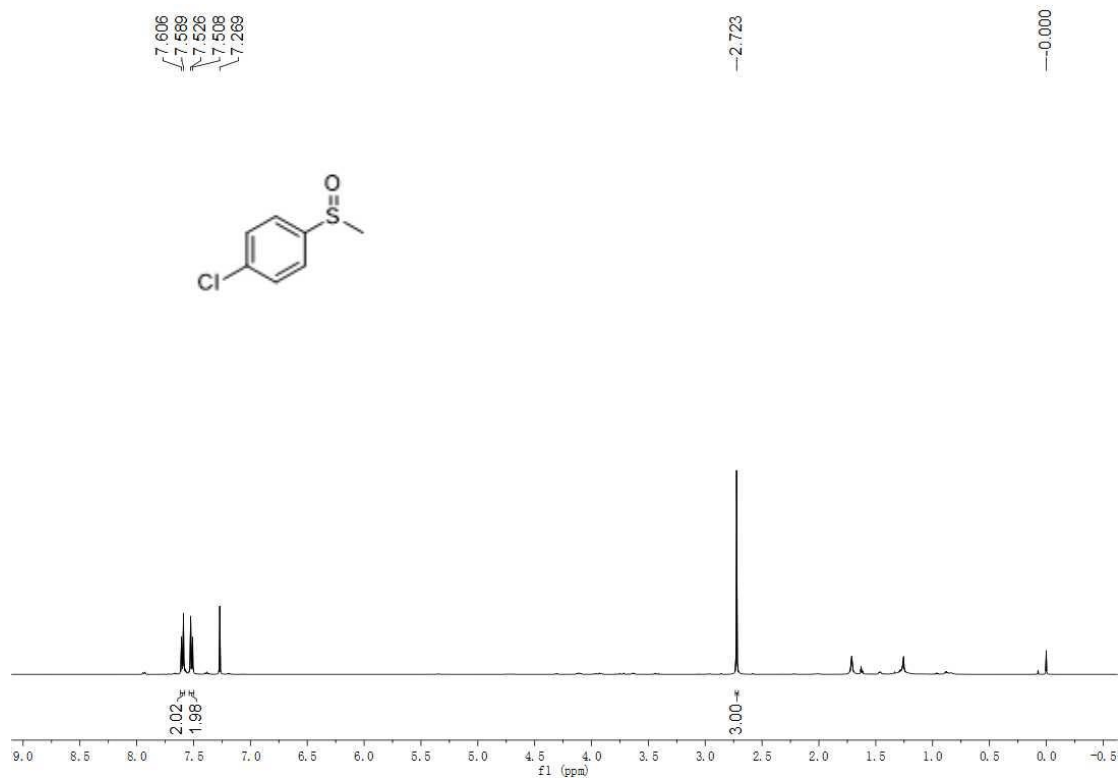


¹H NMR (500 MHz, CDCl₃)

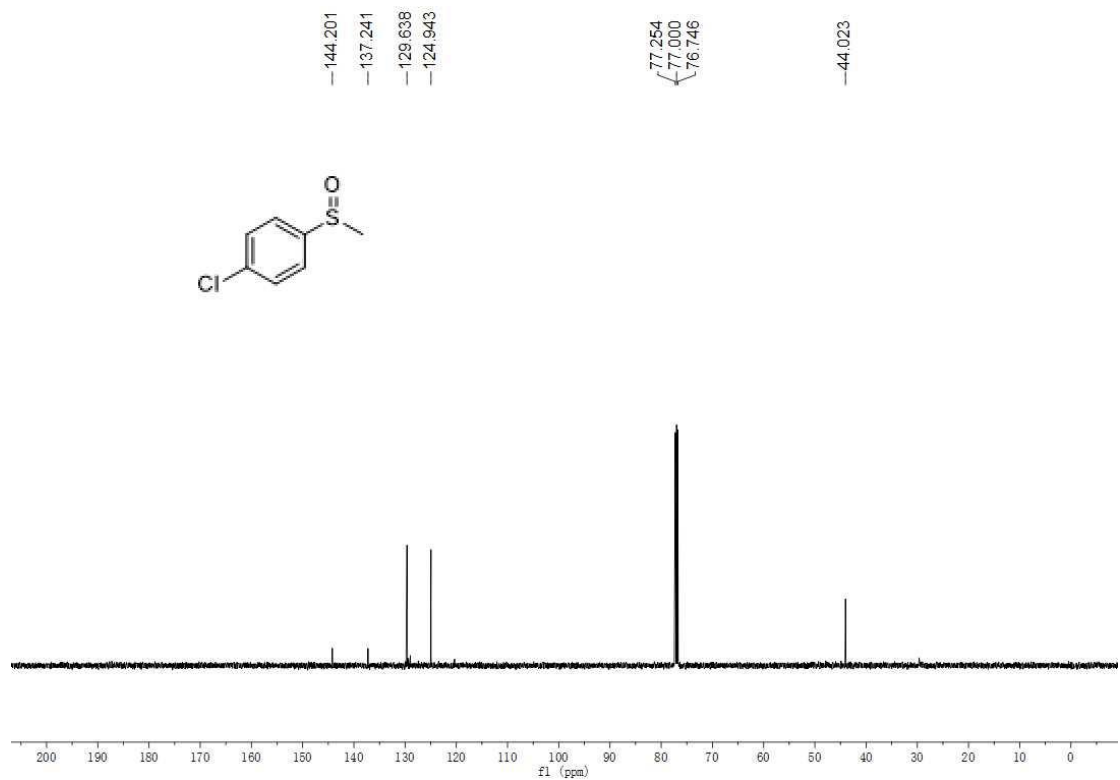


¹³C NMR (125 MHz, CDCl₃)

1-chloro-4-(methylsulfinyl)benzene (2f)



¹H NMR (500 MHz, CDCl₃)

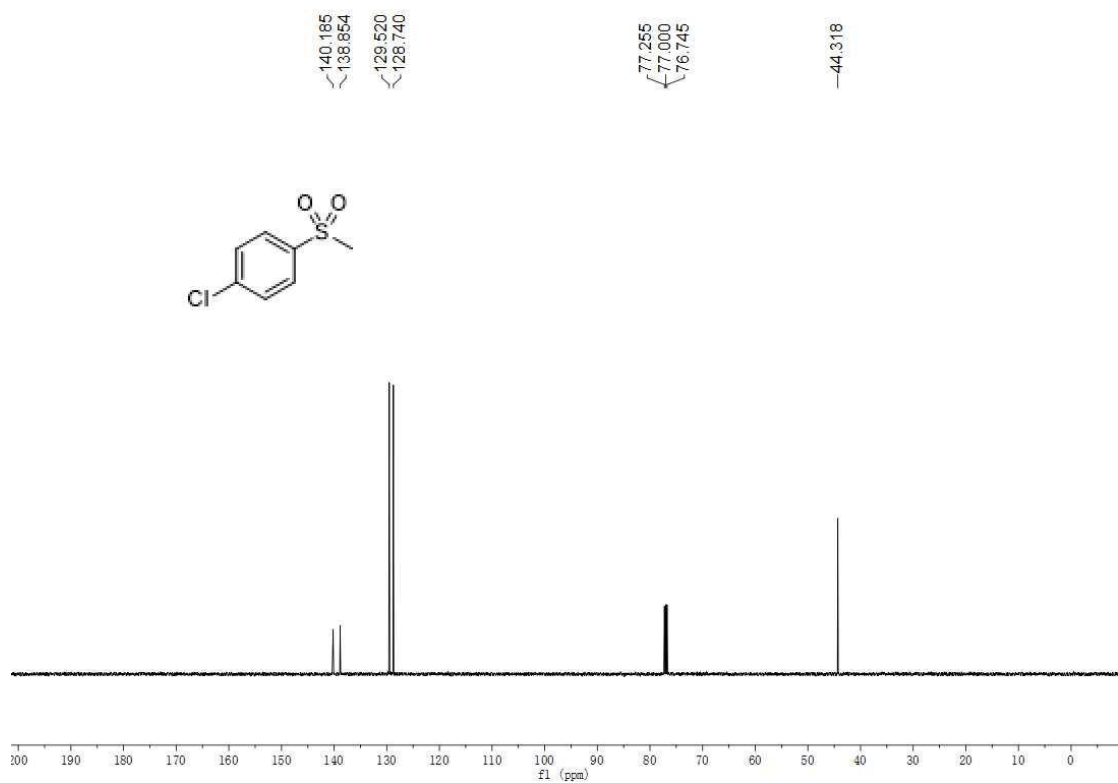


¹³C NMR (125 MHz, CDCl₃)

1-chloro-4-(methylsulfonyl)benzene (3f)

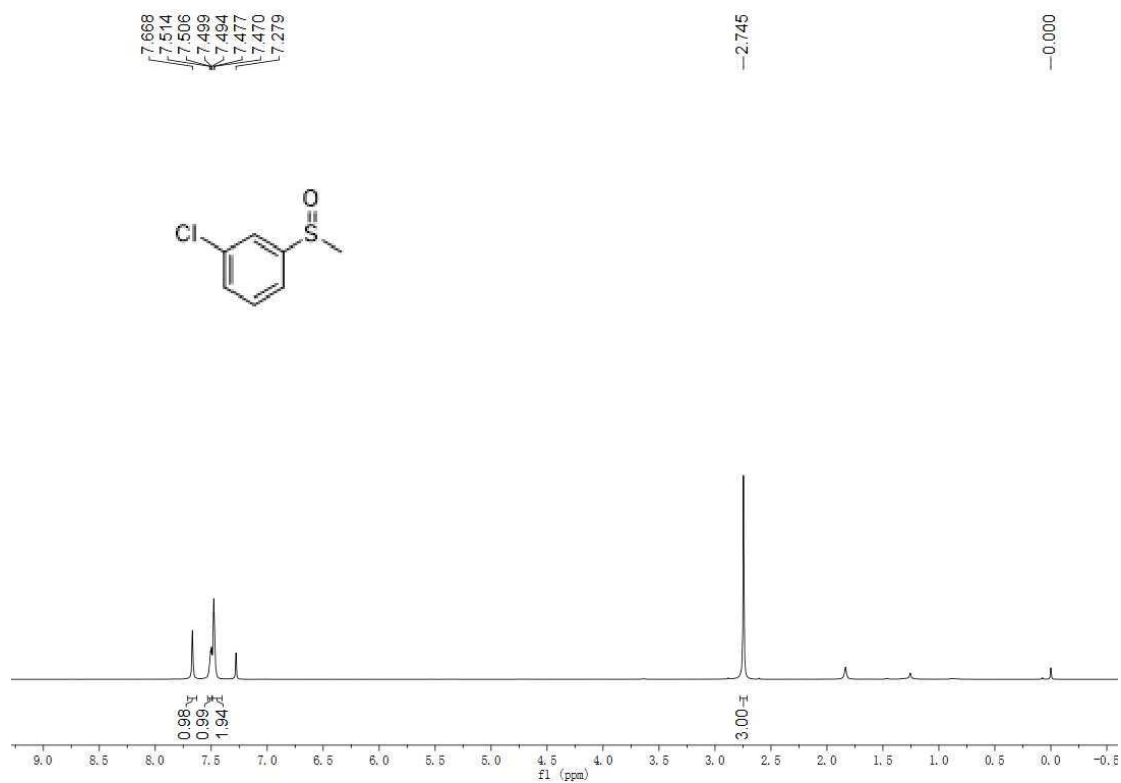


¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

1-chloro-3-(methylsulfinyl)benzene (2g)

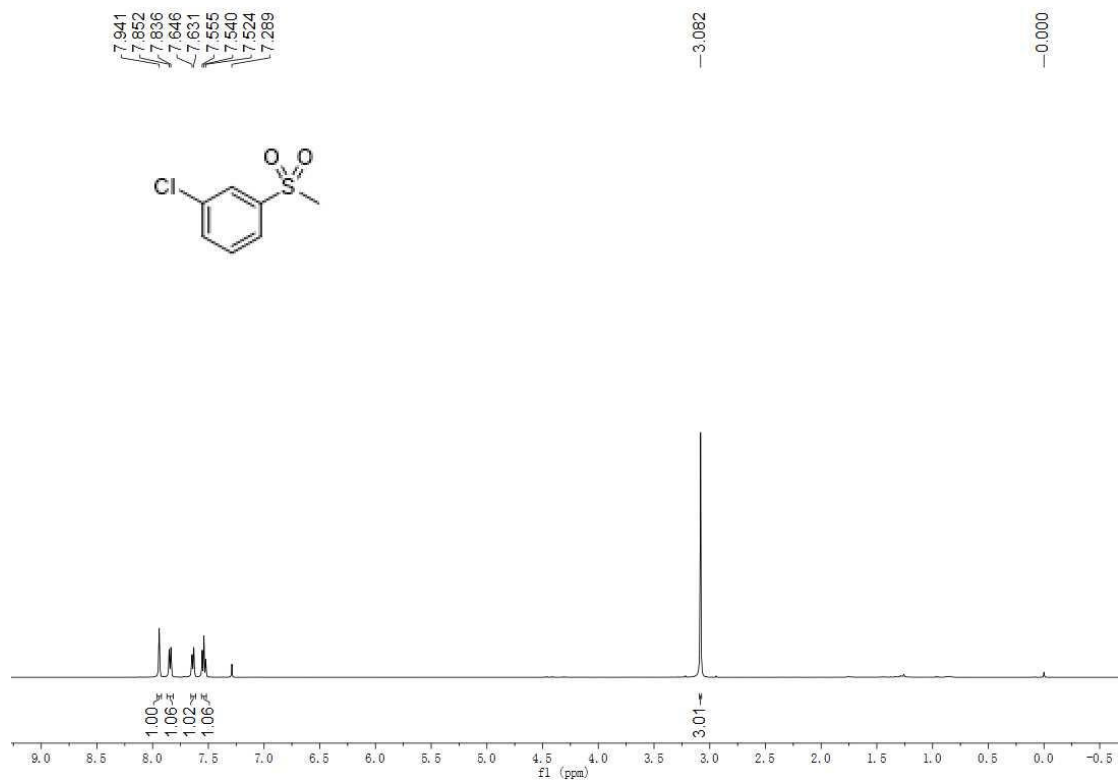


¹H NMR (500 MHz, CDCl₃)

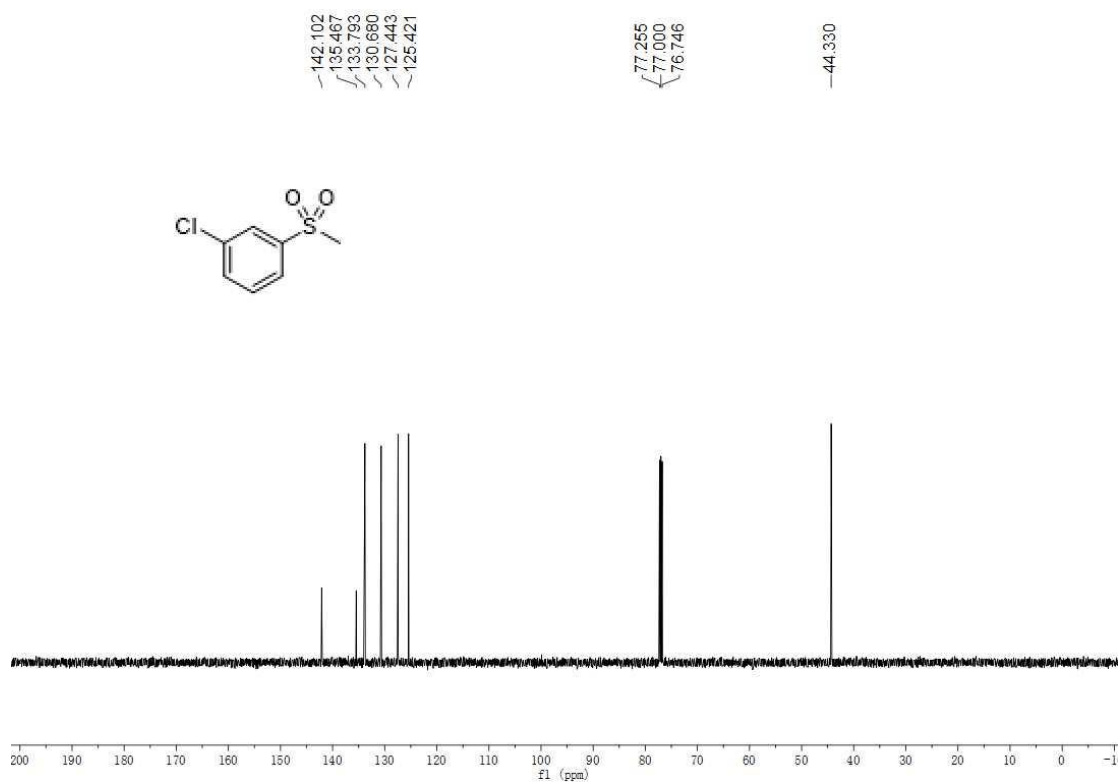


¹³C NMR (125 MHz, CDCl₃)

1-chloro-3-(methylsulfonyl)benzene (3g)

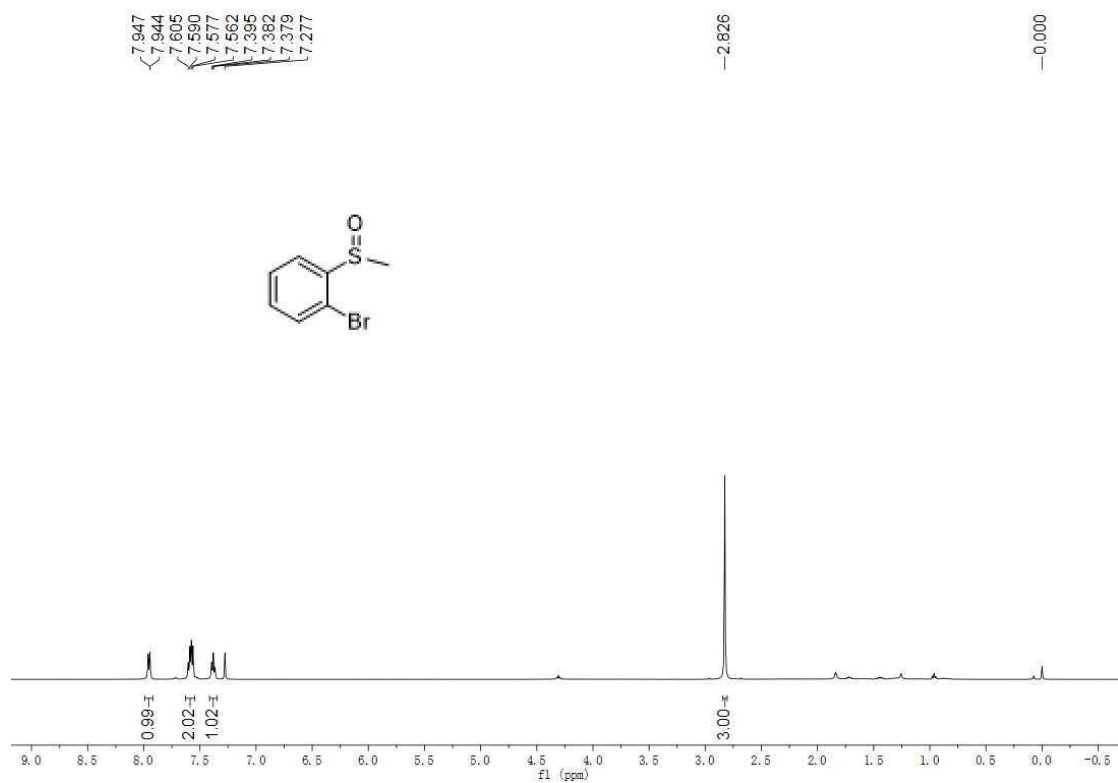


¹H NMR (500 MHz, CDCl₃)

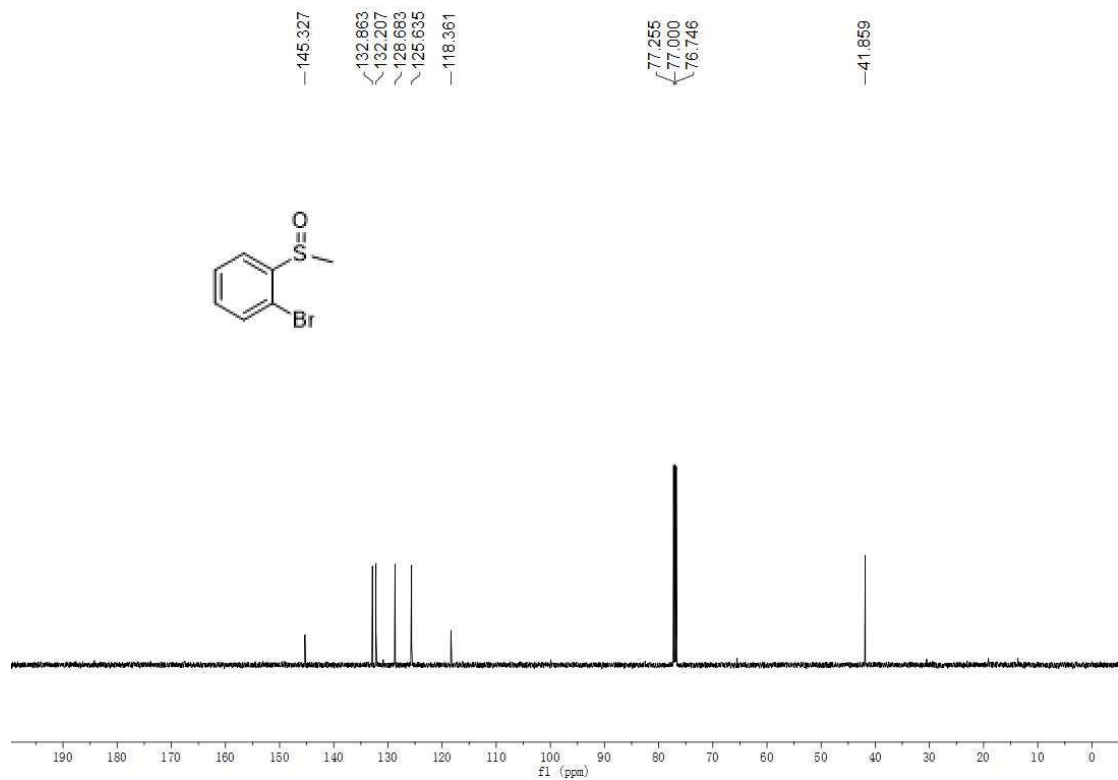


¹³C NMR (125 MHz, CDCl₃)

1-bromo-2-(methylsulfinyl)benzene (2h)



¹H NMR (500 MHz, CDCl₃)

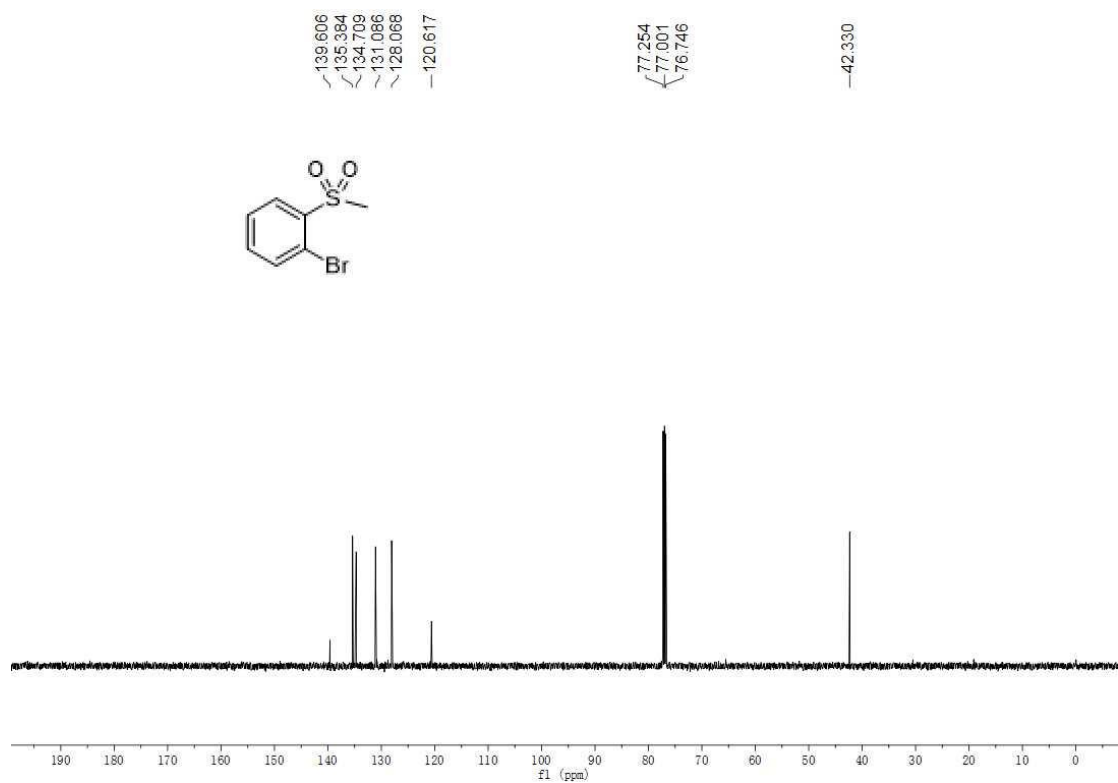


¹³C NMR (125 MHz, CDCl₃)

1-bromo-2-(methylsulfonyl)benzene (3h)

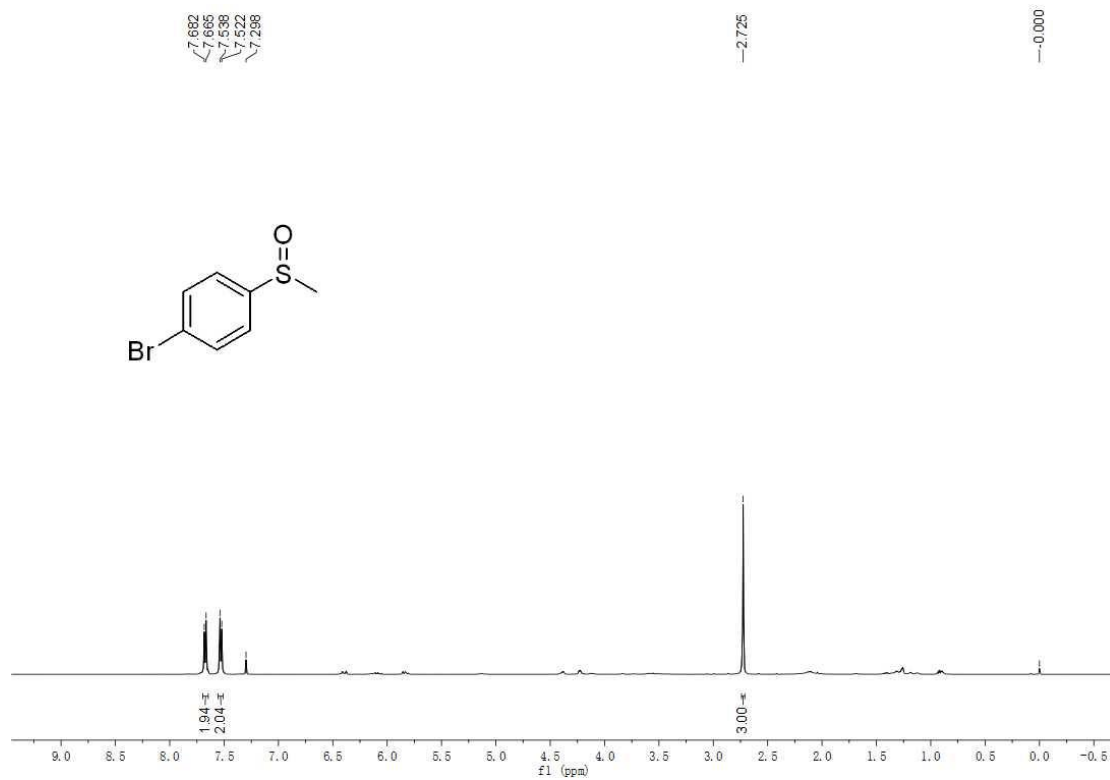


¹H NMR (500 MHz, CDCl₃)

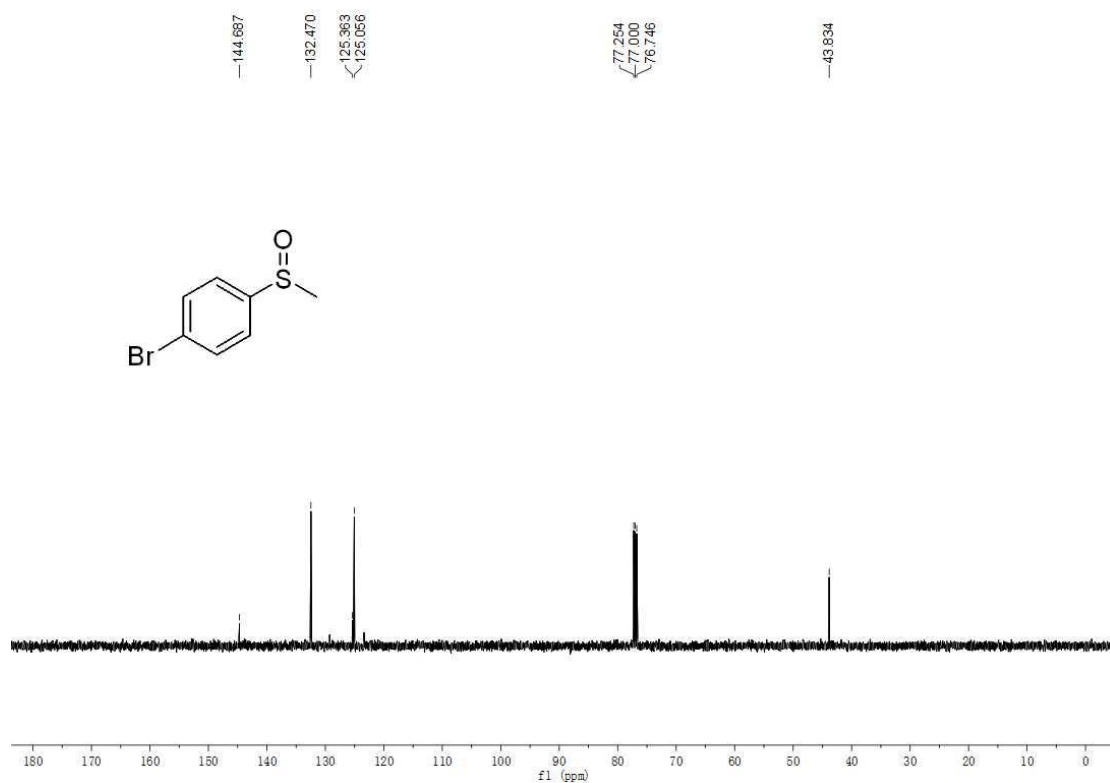


¹³C NMR (125 MHz, CDCl₃)

1-bromo-4-(methylsulfinyl)benzene (2i)

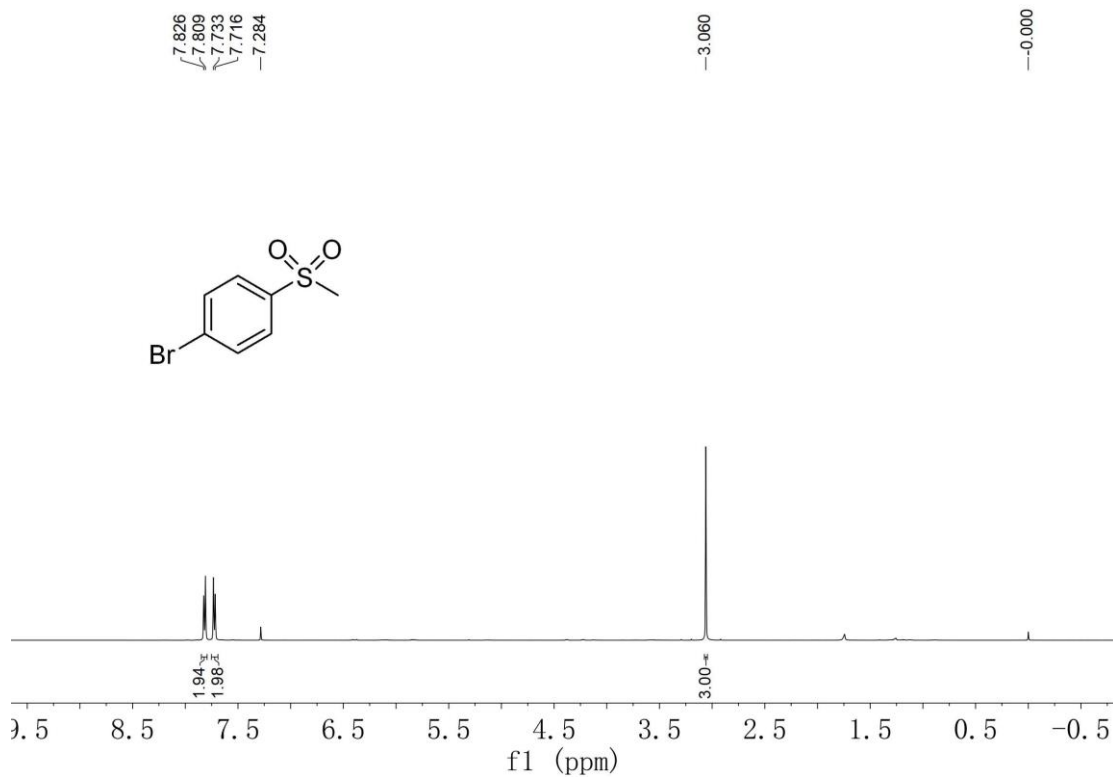


¹H NMR (500 MHz, CDCl₃)

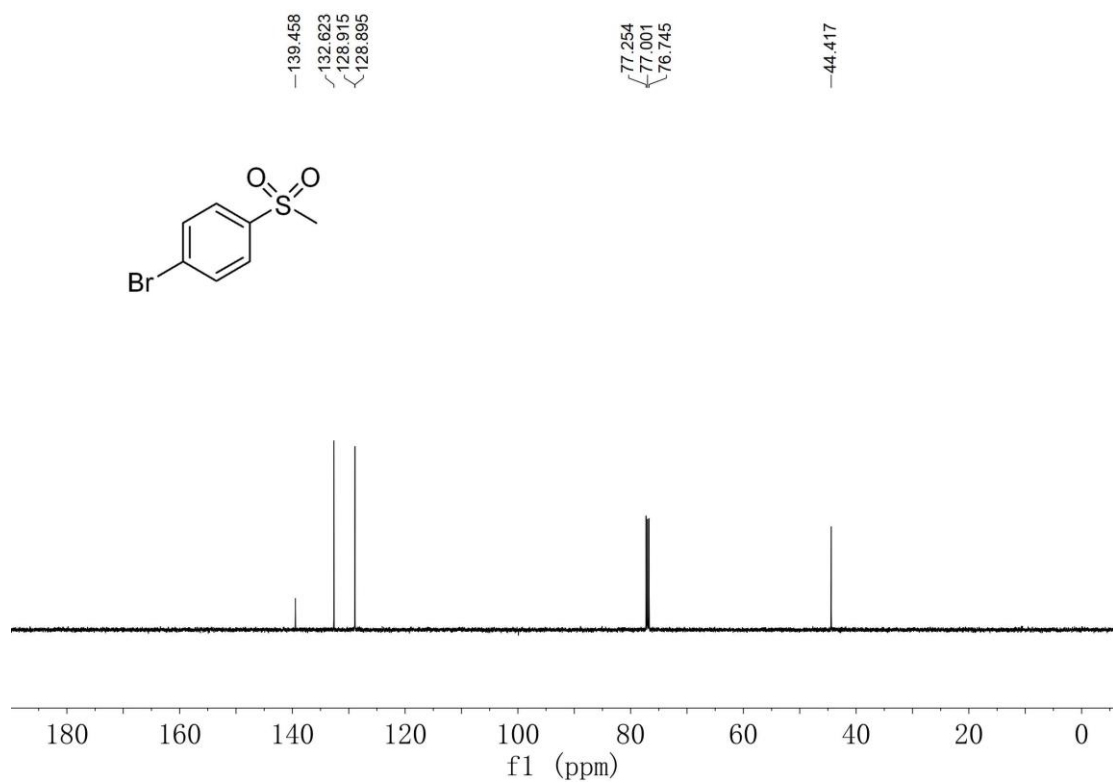


¹³C NMR (125 MHz, CDCl₃)

1-bromo-4-(methylsulfonyl)benzene (3i)

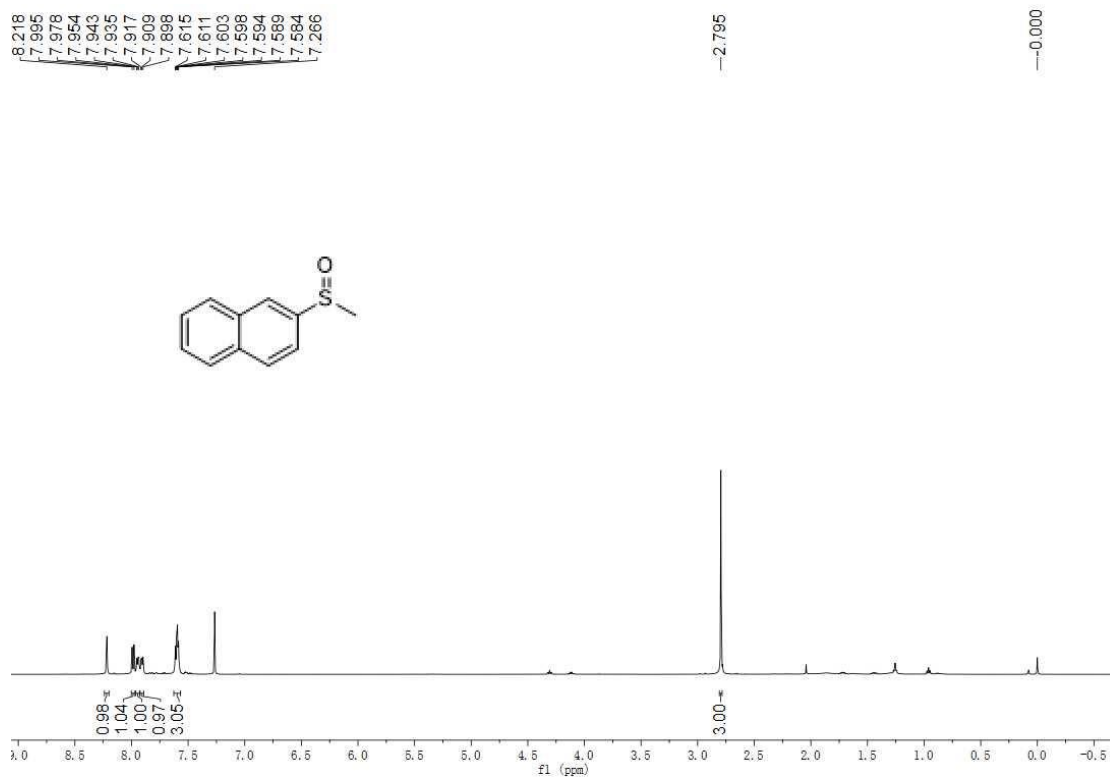


¹H NMR (500 MHz, CDCl₃)

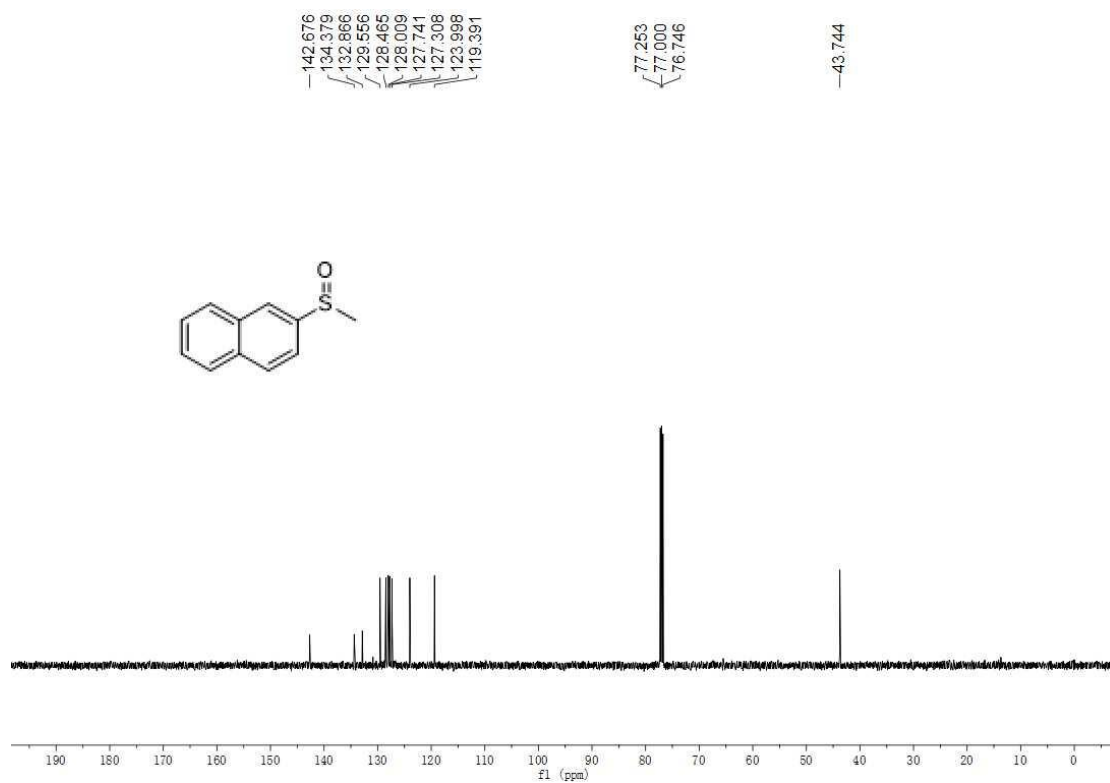


¹³C NMR (125 MHz, CDCl₃)

2-(methylsulfinyl)naphthalene (2j)

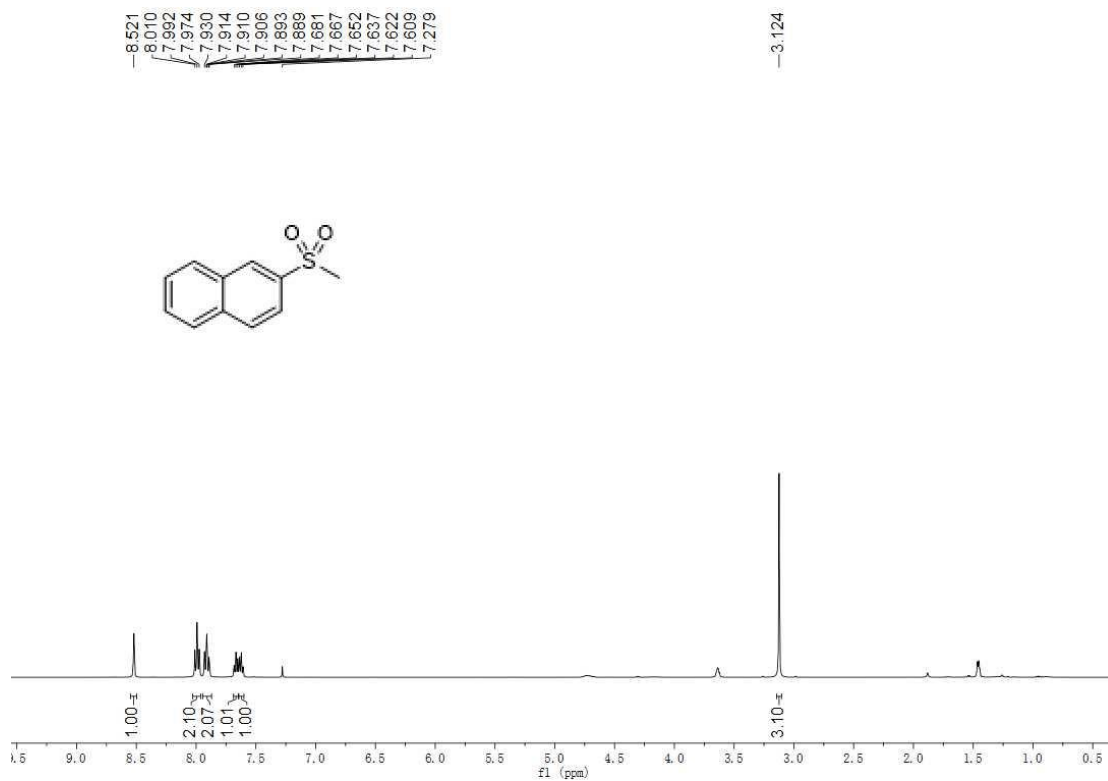


¹H NMR (500 MHz, CDCl₃)

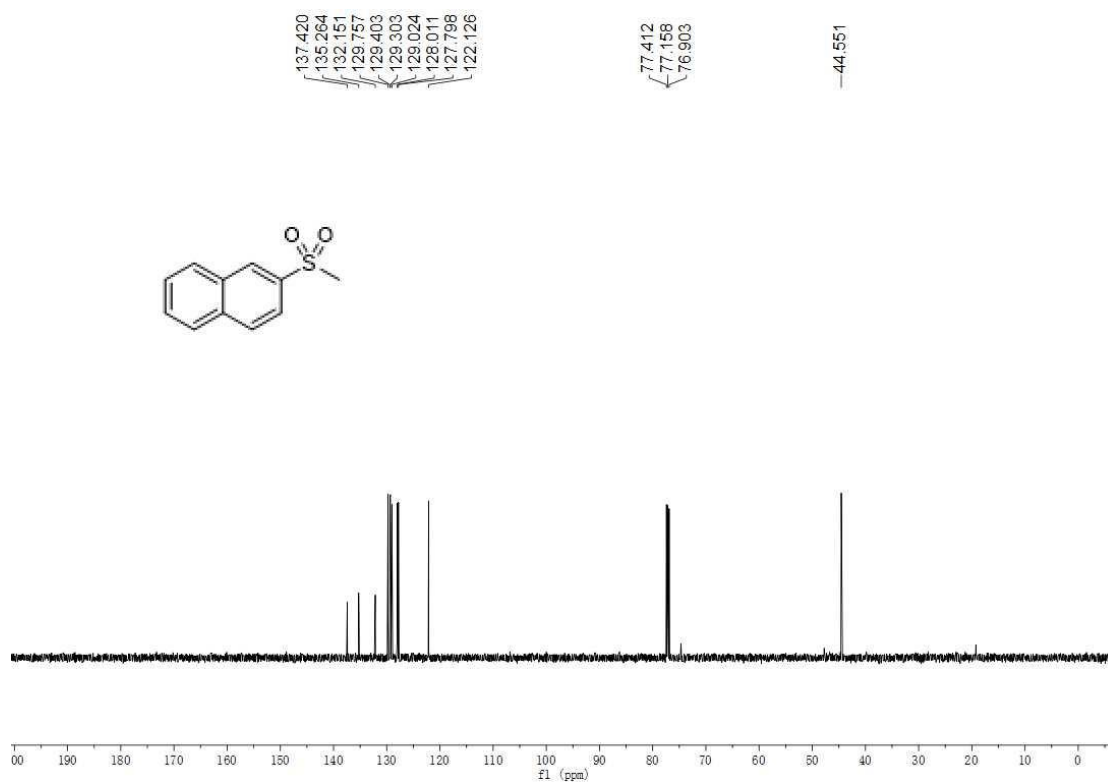


¹³C NMR (125 MHz, CDCl₃)

2-(methylsulfonyl)naphthalene (3j)

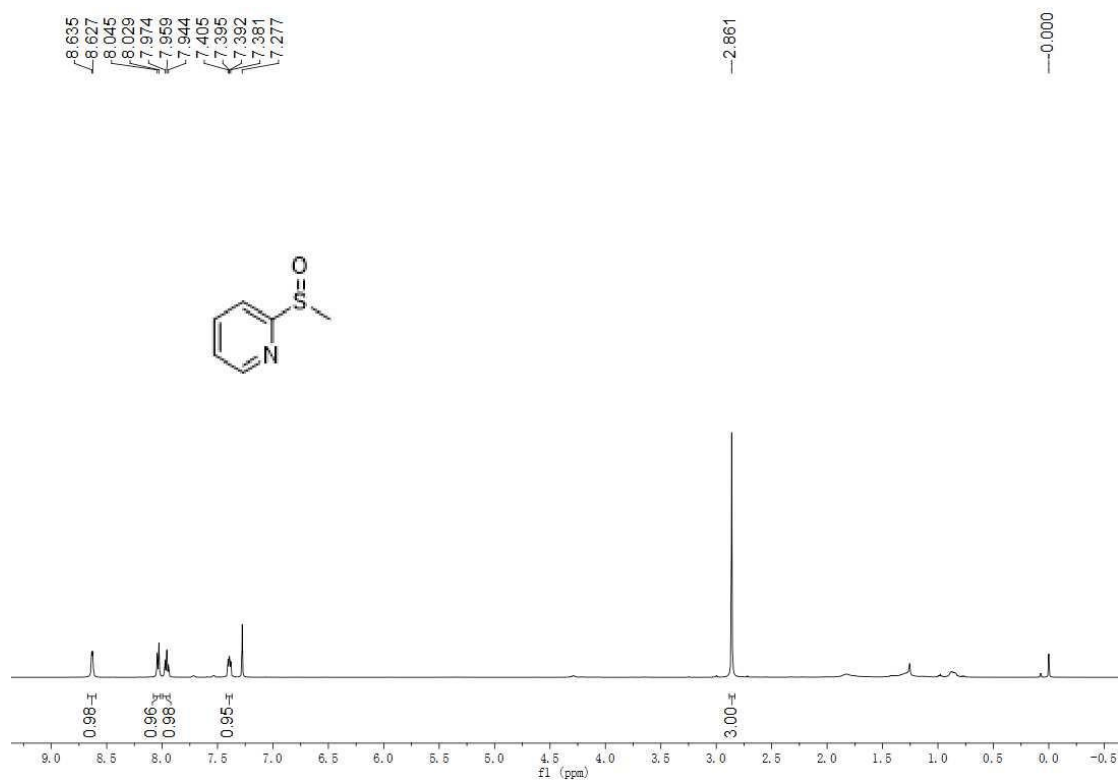


¹H NMR (500 MHz, CDCl₃)

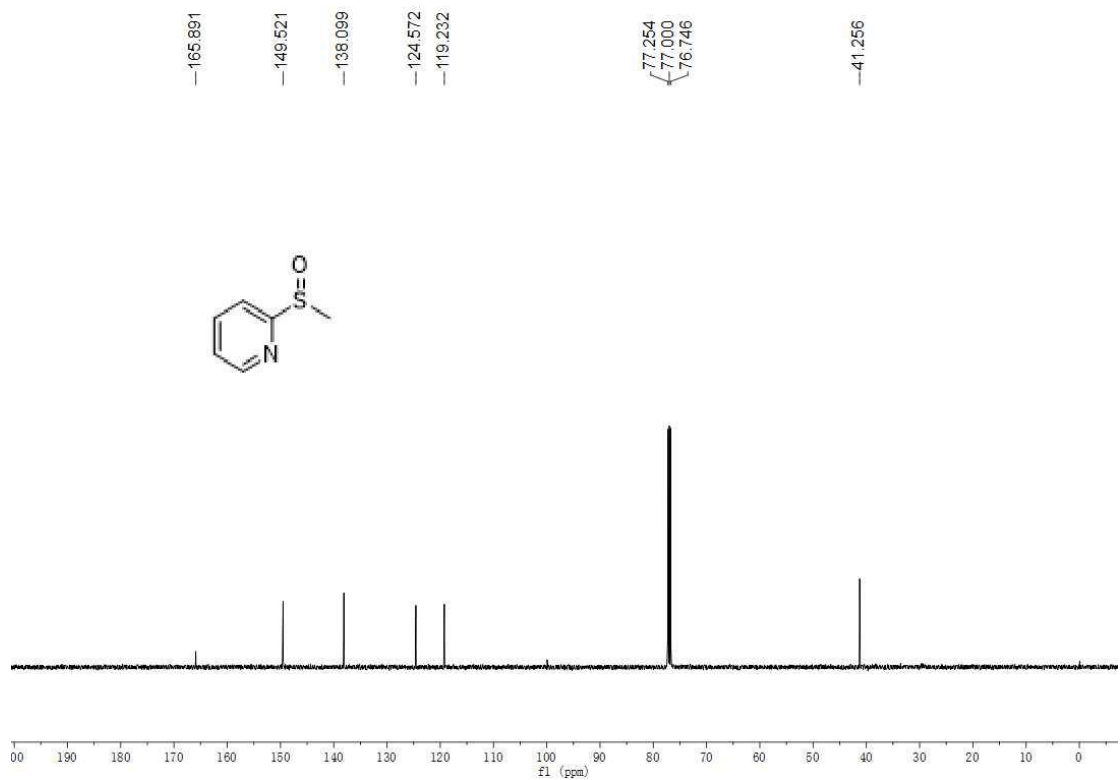


¹³C NMR (125 MHz, CDCl₃)

2-(methylsulfinyl)pyridine (2k)

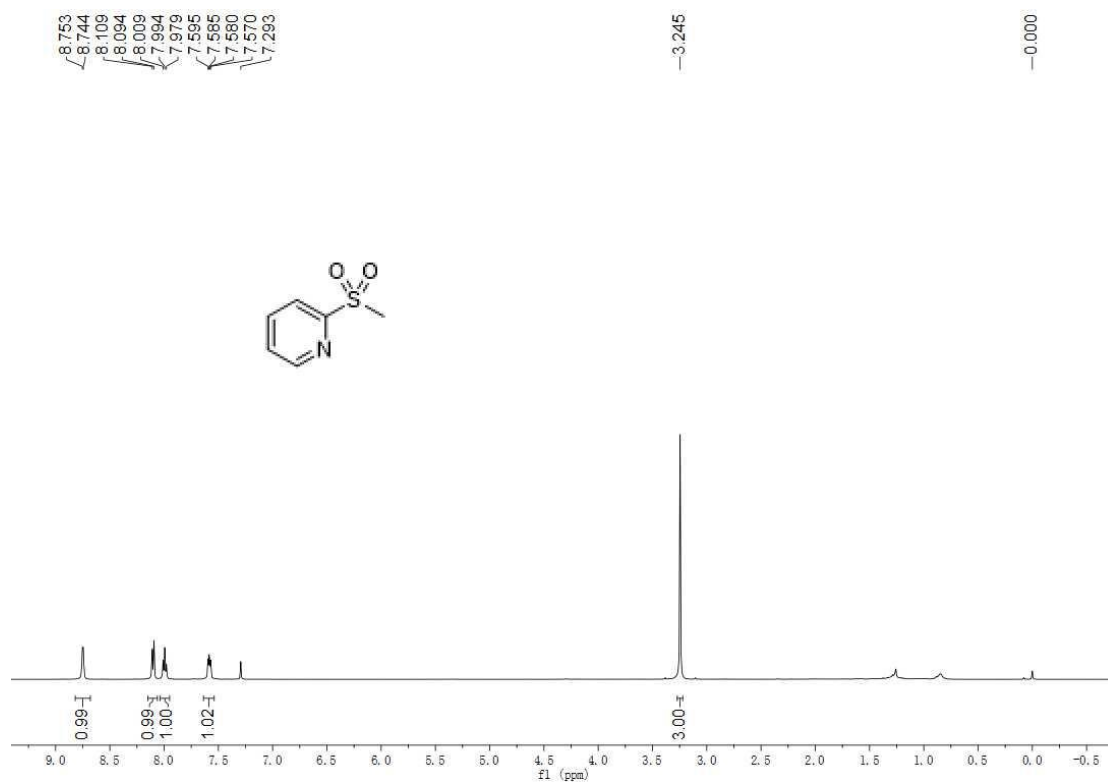


¹H NMR (500 MHz, CDCl₃)

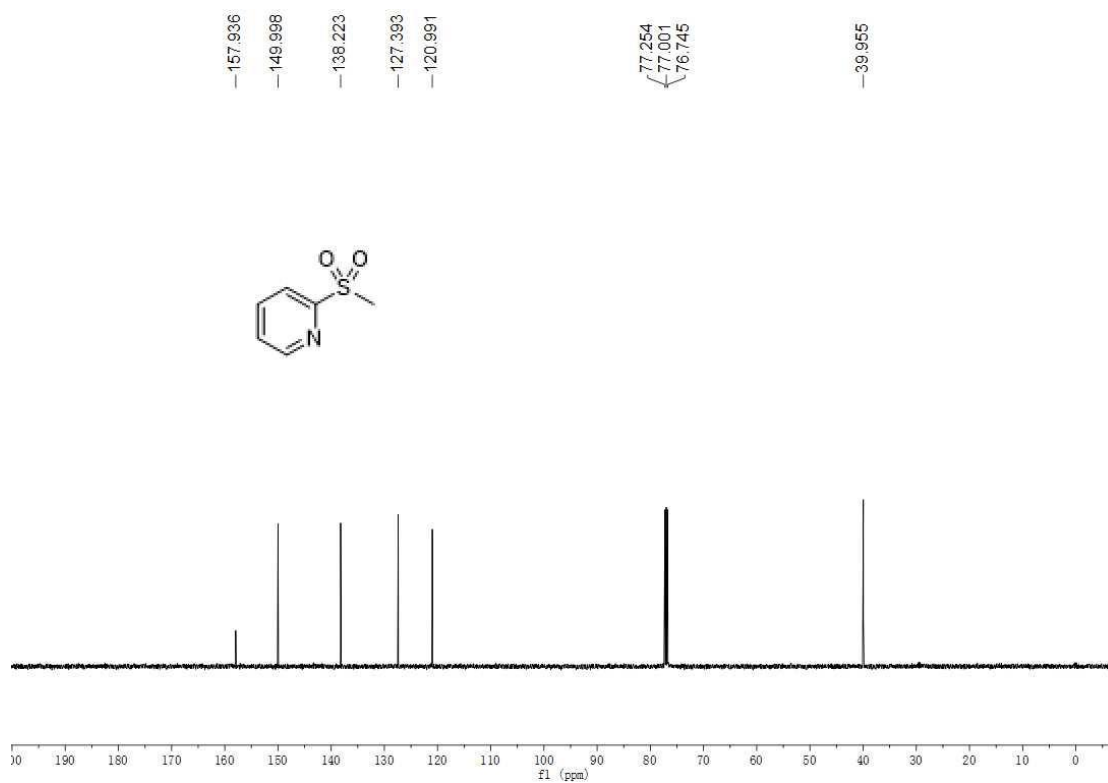


¹³C NMR (125 MHz, CDCl₃)

2-(methylsulfonyl)pyridine (3k)

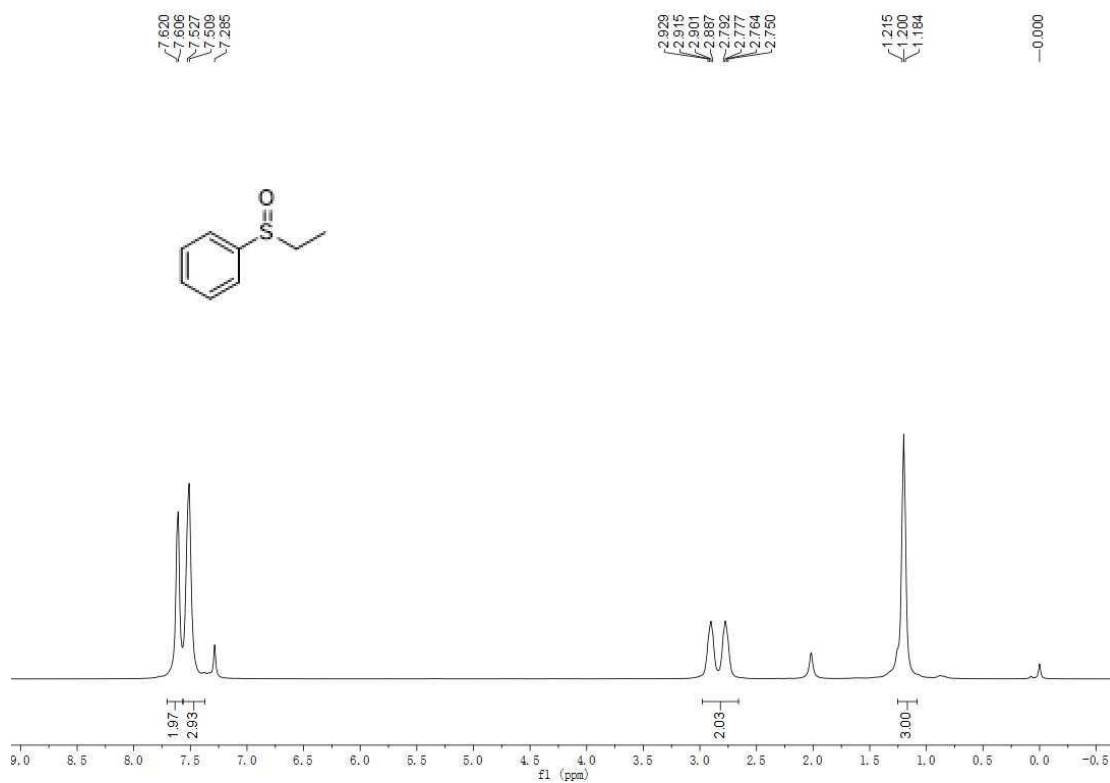


¹H NMR (500 MHz, CDCl₃)

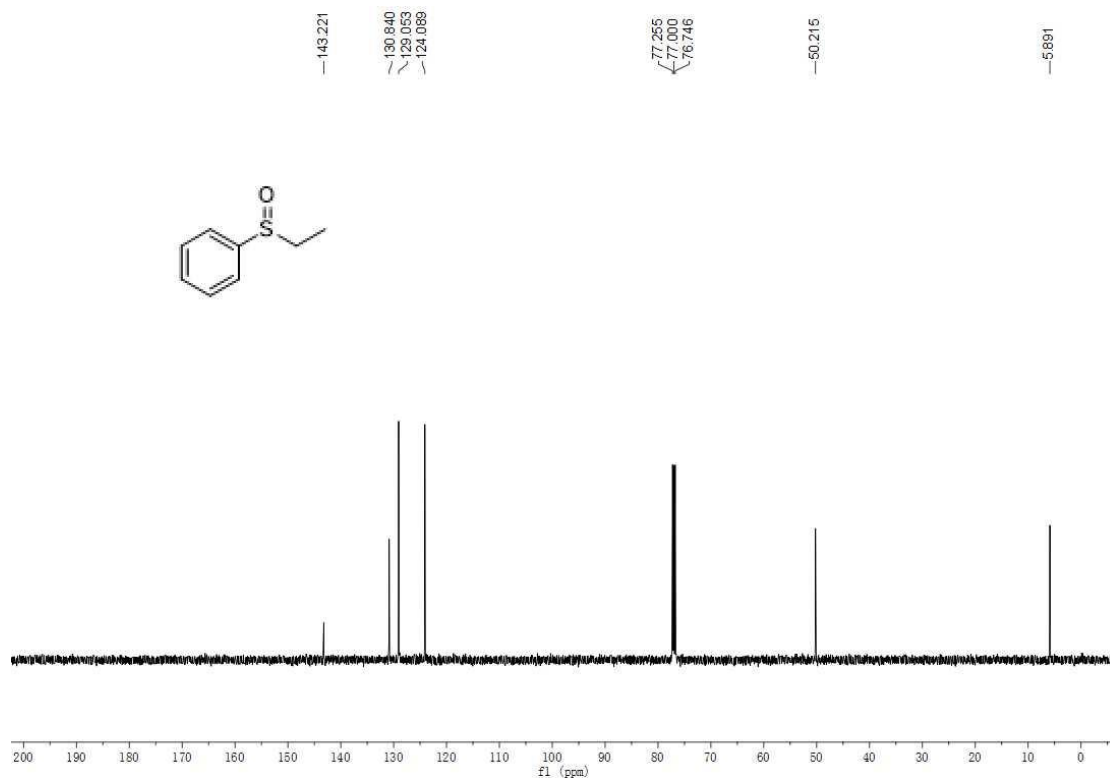


¹³C NMR (125 MHz, CDCl₃)

(ethylsulfinyl)benzene (2l)

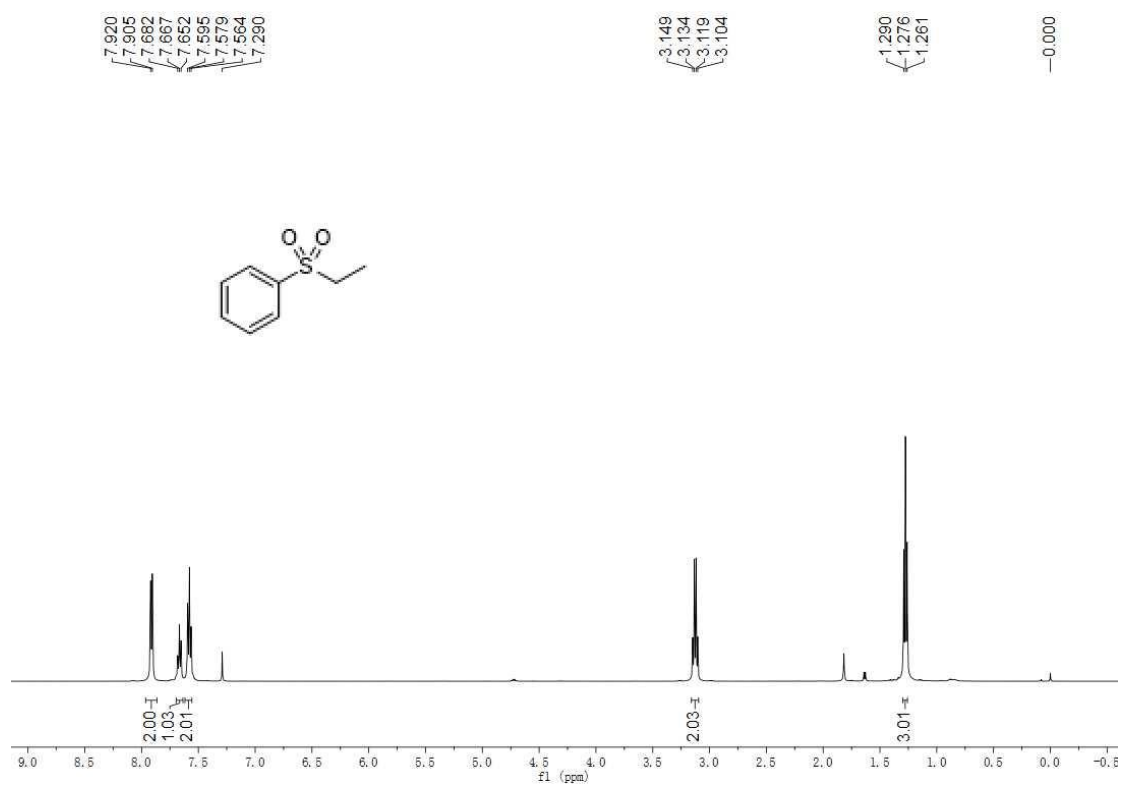


¹H NMR (500 MHz, CDCl₃)

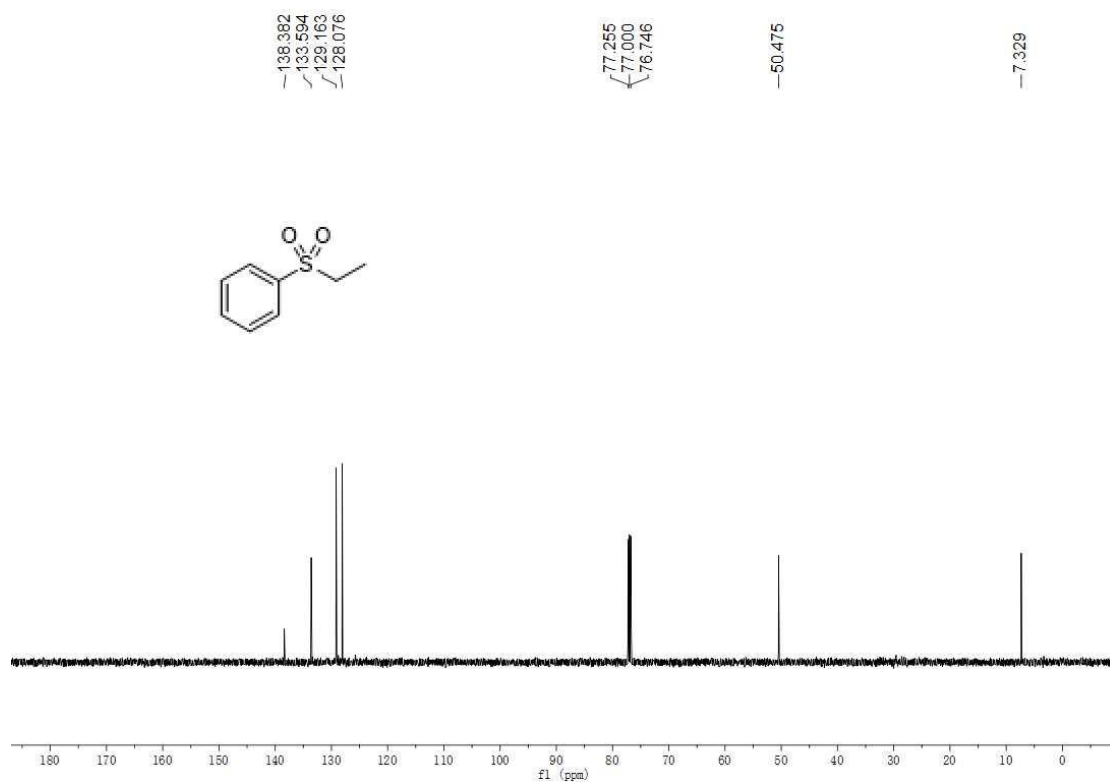


¹³C NMR (125 MHz, CDCl₃)

(ethylsulfonyl)benzene (3l)

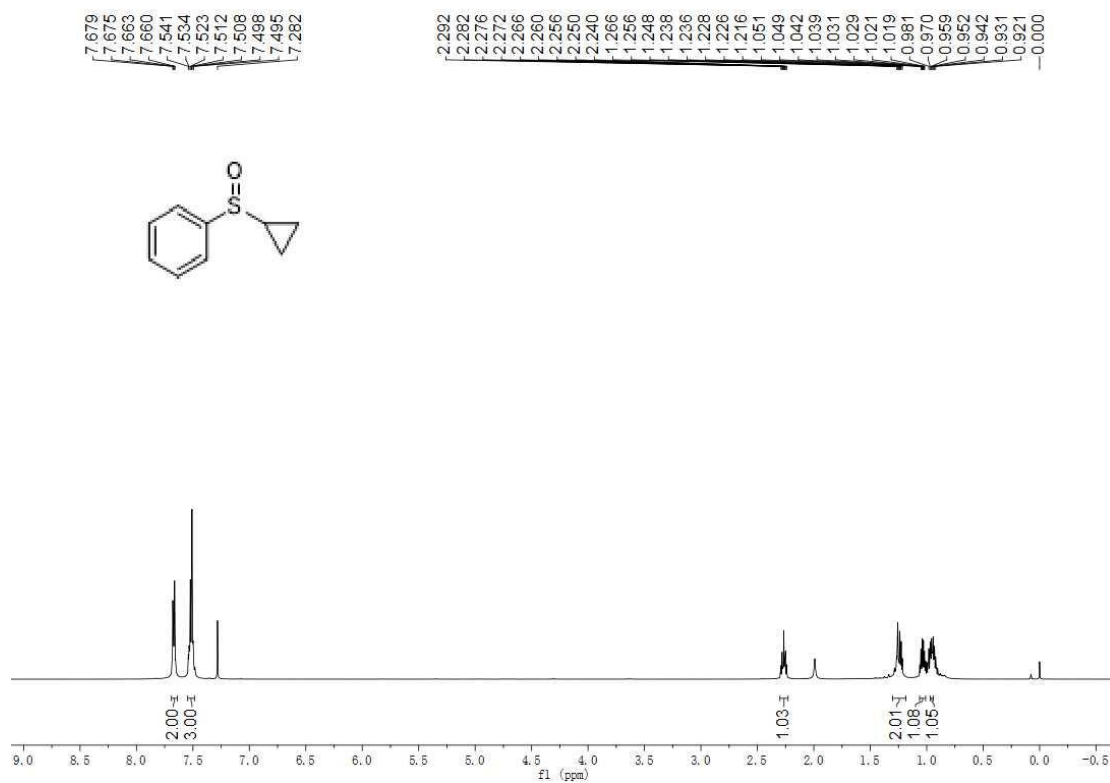


¹H NMR (500 MHz, CDCl₃)

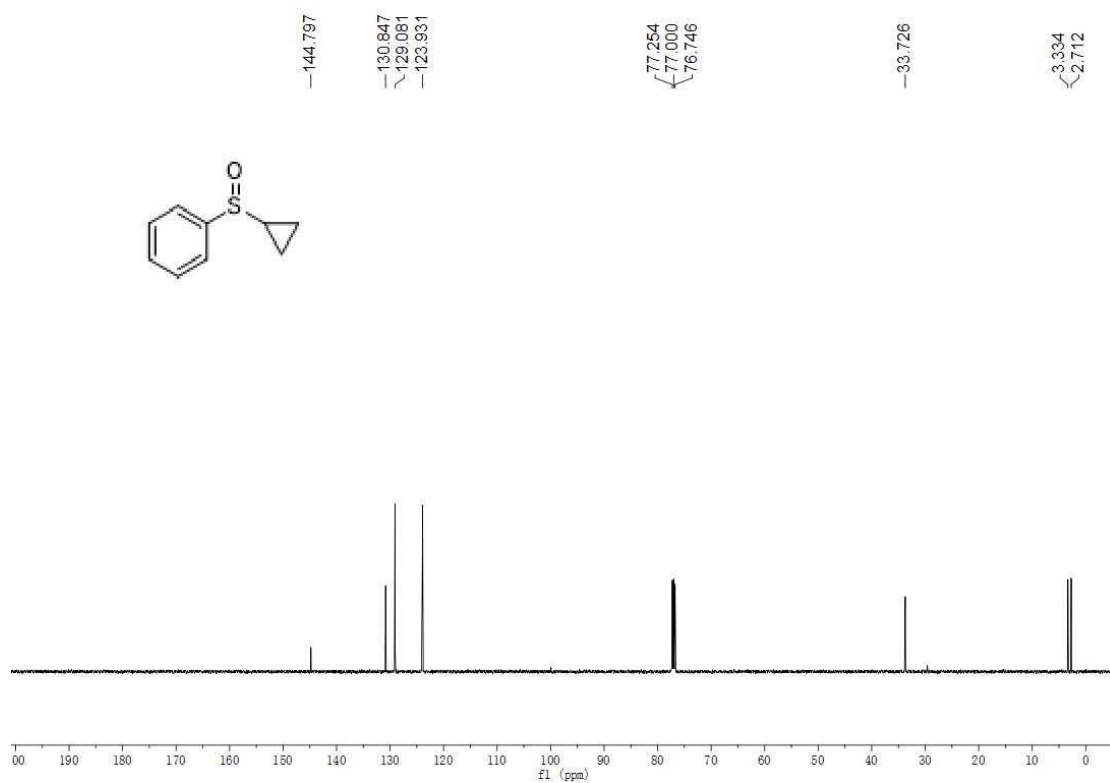


¹³C NMR (125 MHz, CDCl₃)

(ethylsulfonyl)benzene (2m)

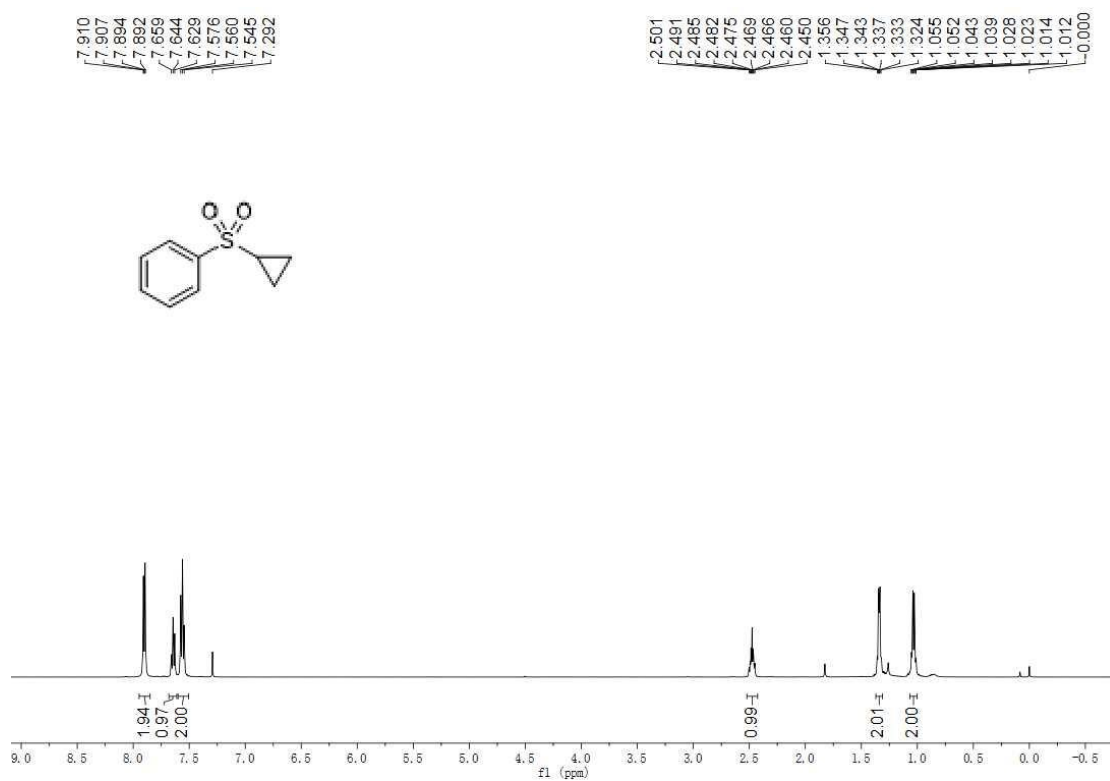


¹H NMR (500 MHz, CDCl₃)

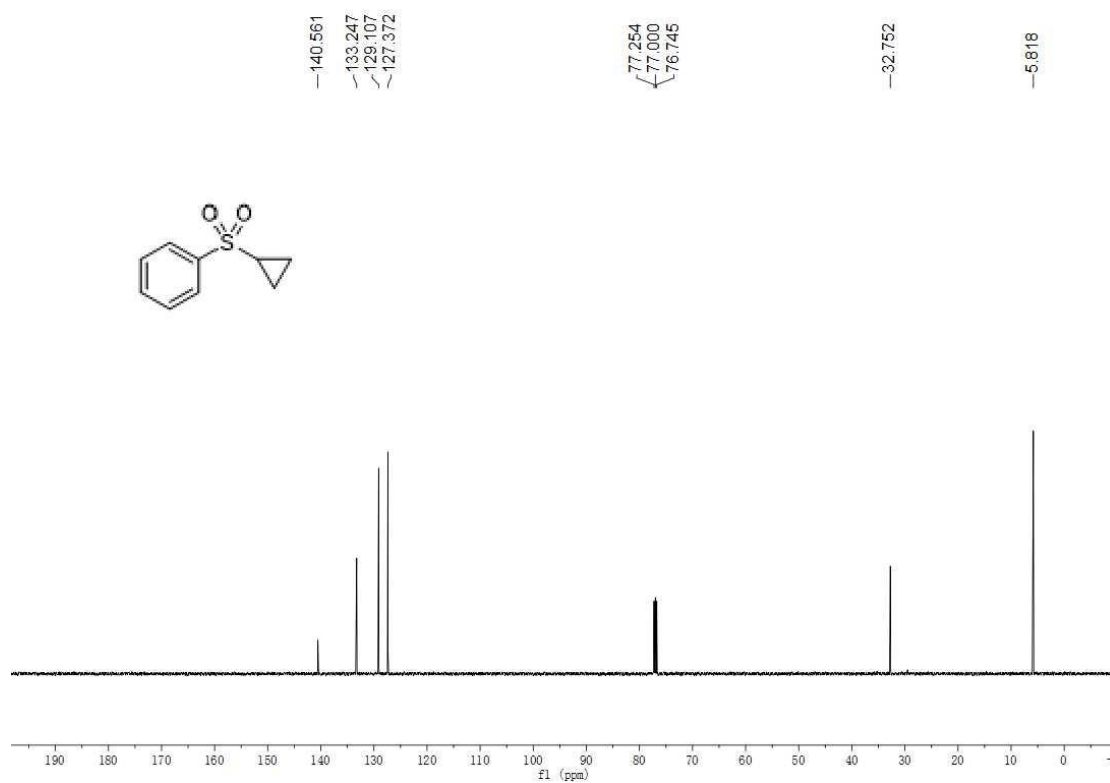


¹³C NMR (125 MHz, CDCl₃)

(cyclopropylsulfonyl)benzene (3m)

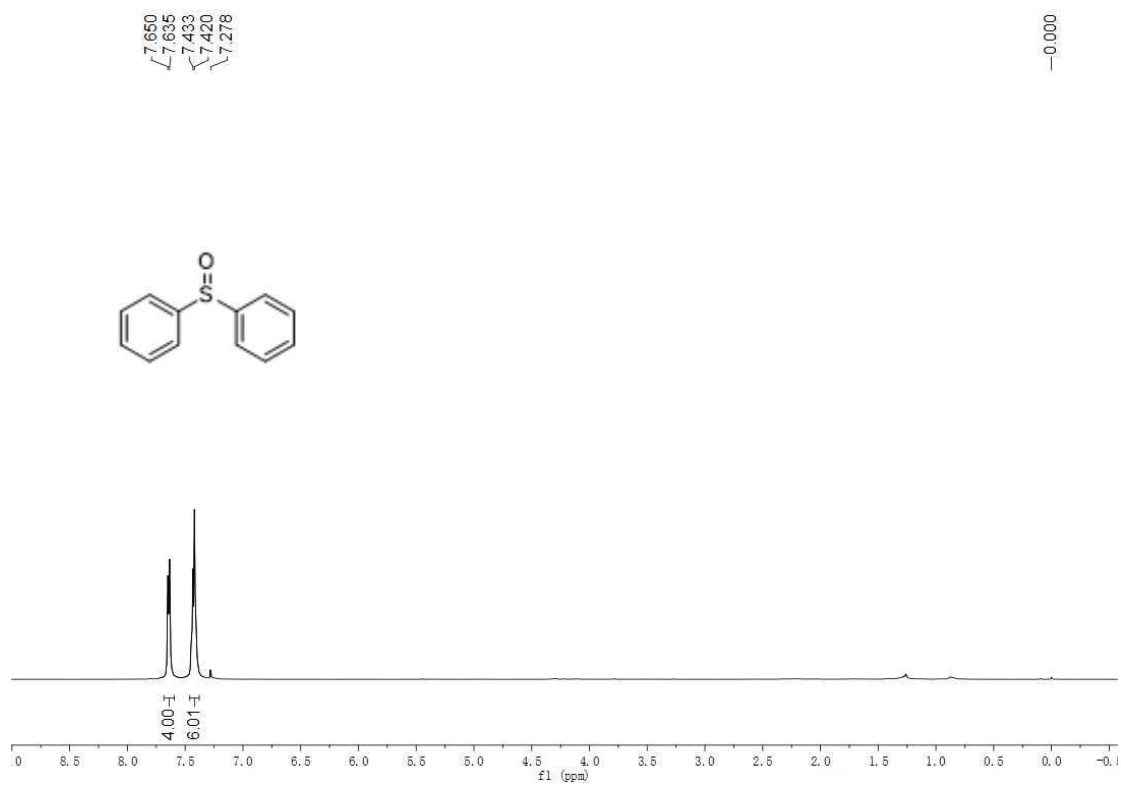


¹H NMR (500 MHz, CDCl₃)

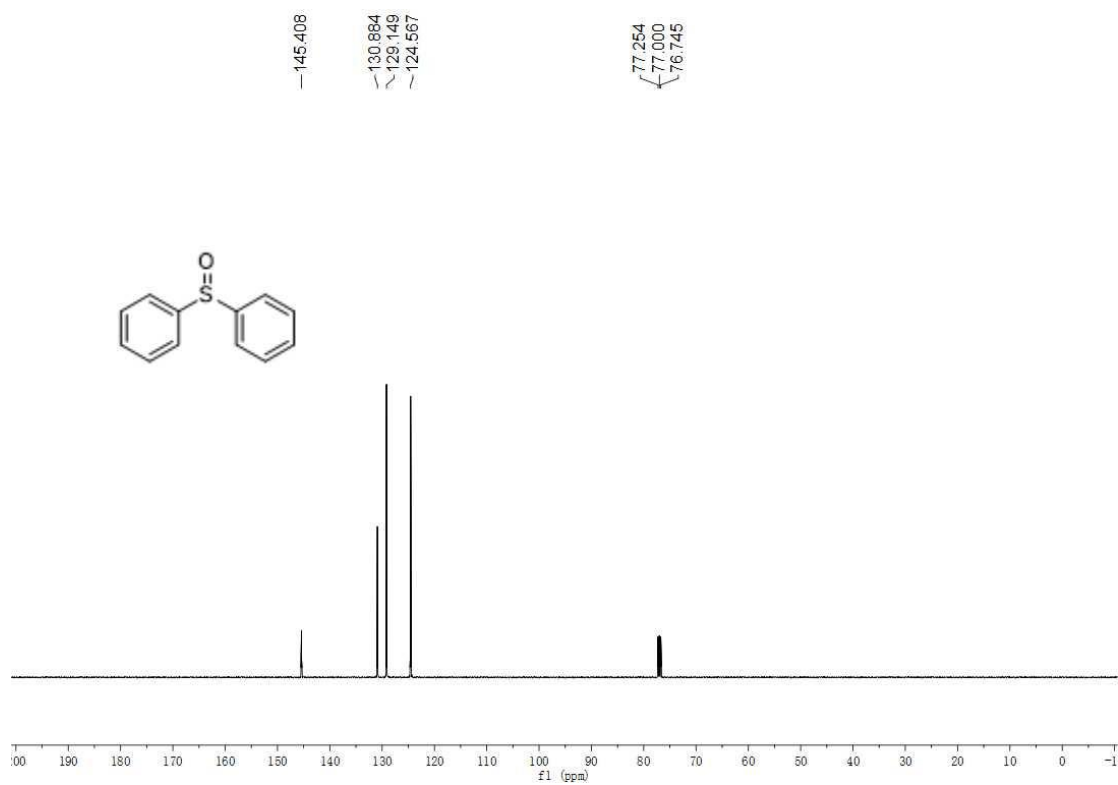


¹³C NMR (125 MHz, CDCl₃)

Sulfinyldibenzene (2n)

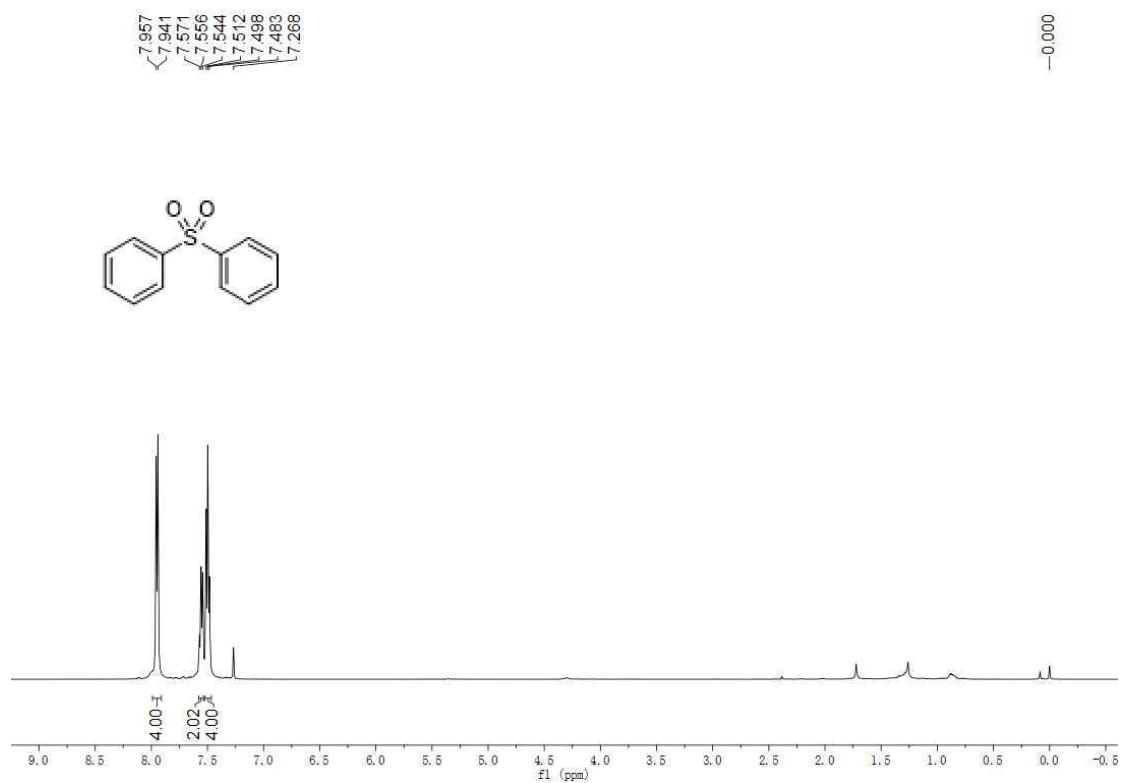


¹H NMR (500 MHz, CDCl₃)

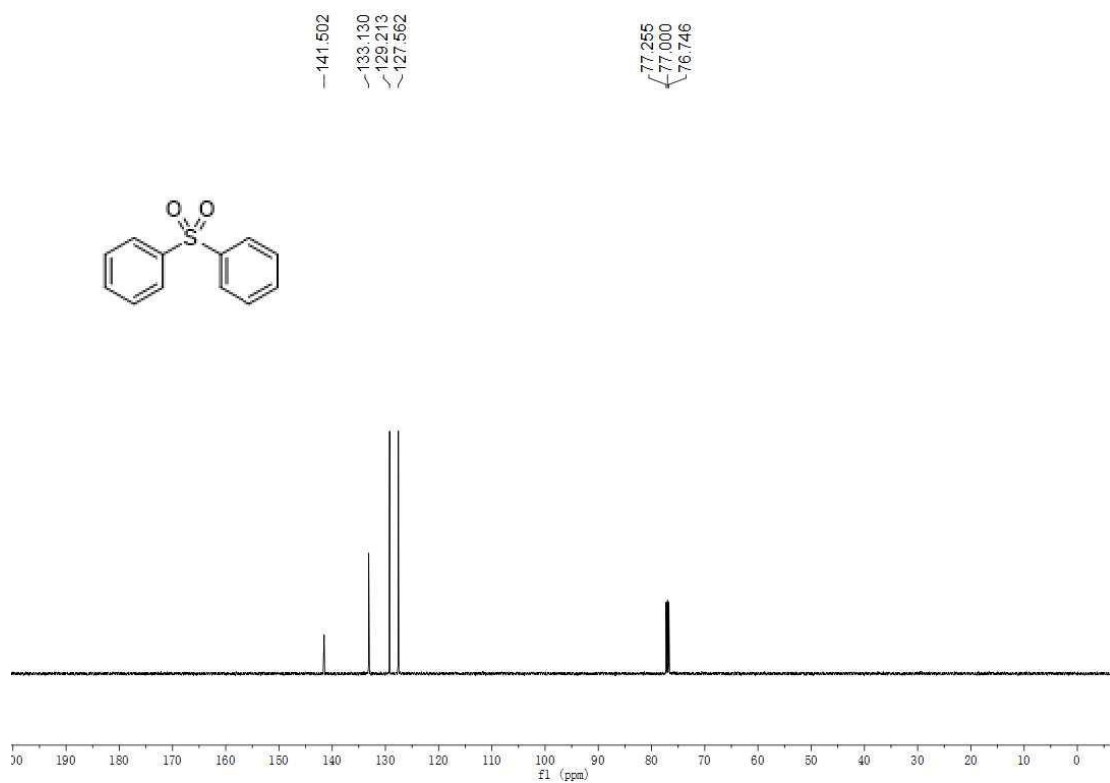


¹³C NMR (125 MHz, CDCl₃)

Sulfonyldibenzene (3n)

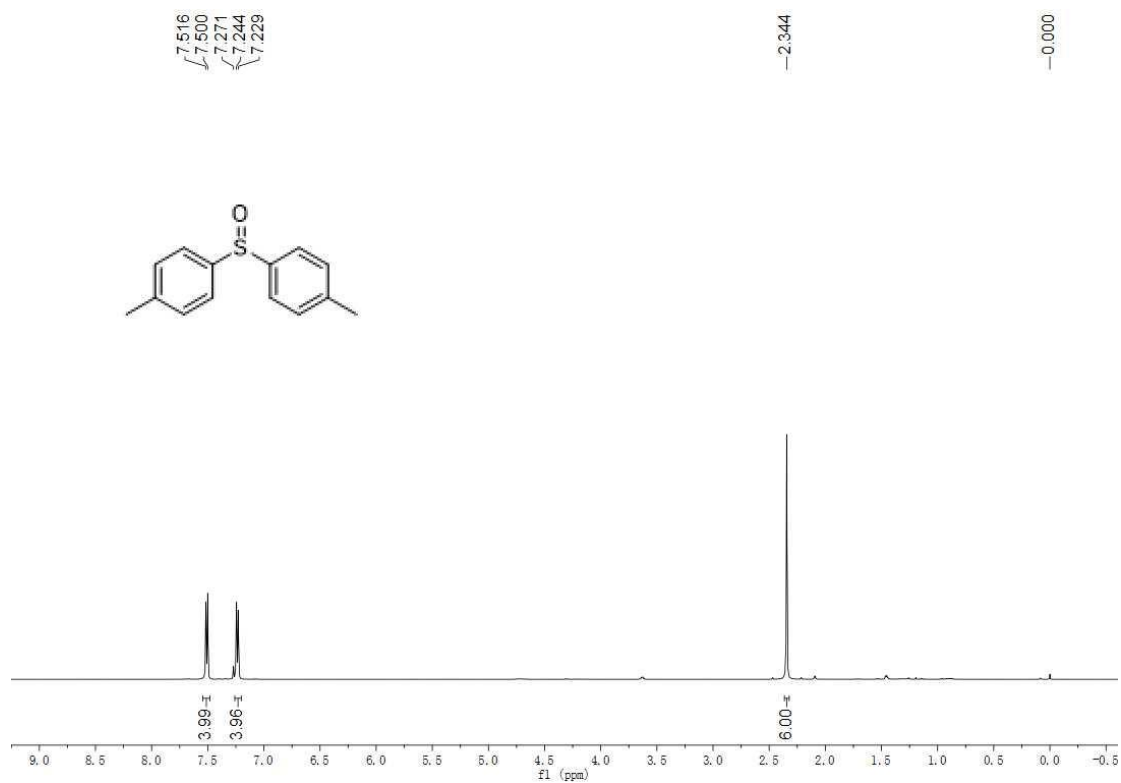


¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfinylbis(methylbenzene) (2o)

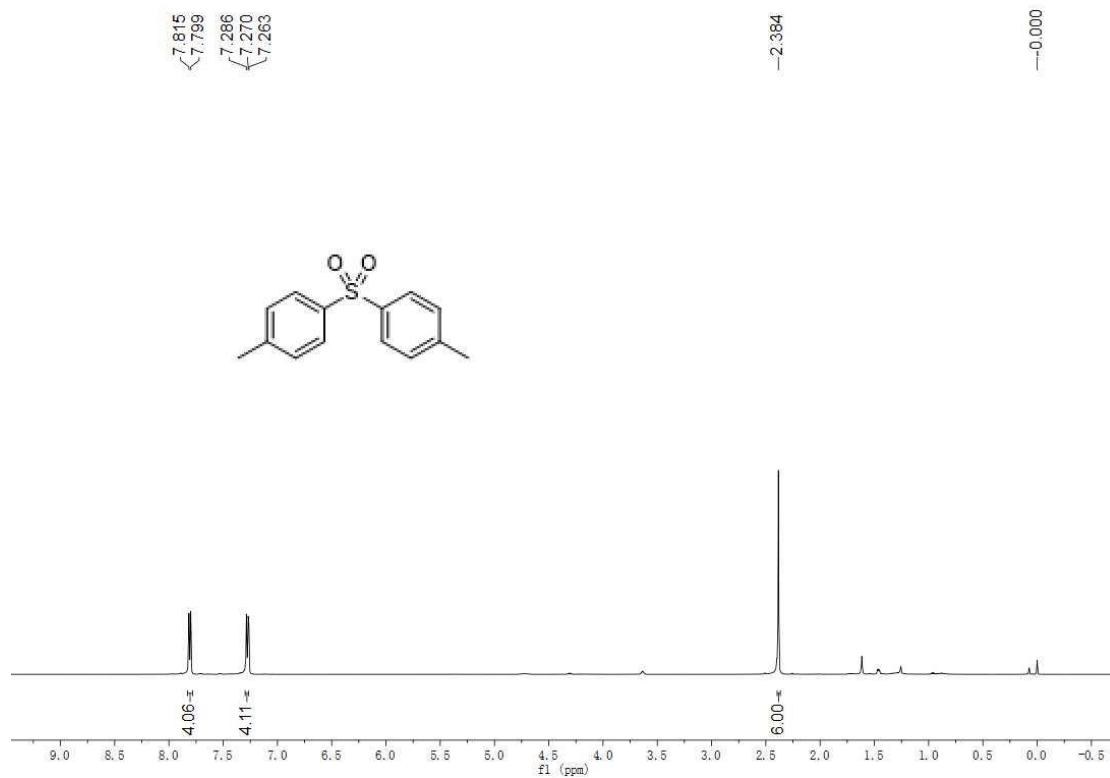


¹H NMR (500 MHz, CDCl₃)

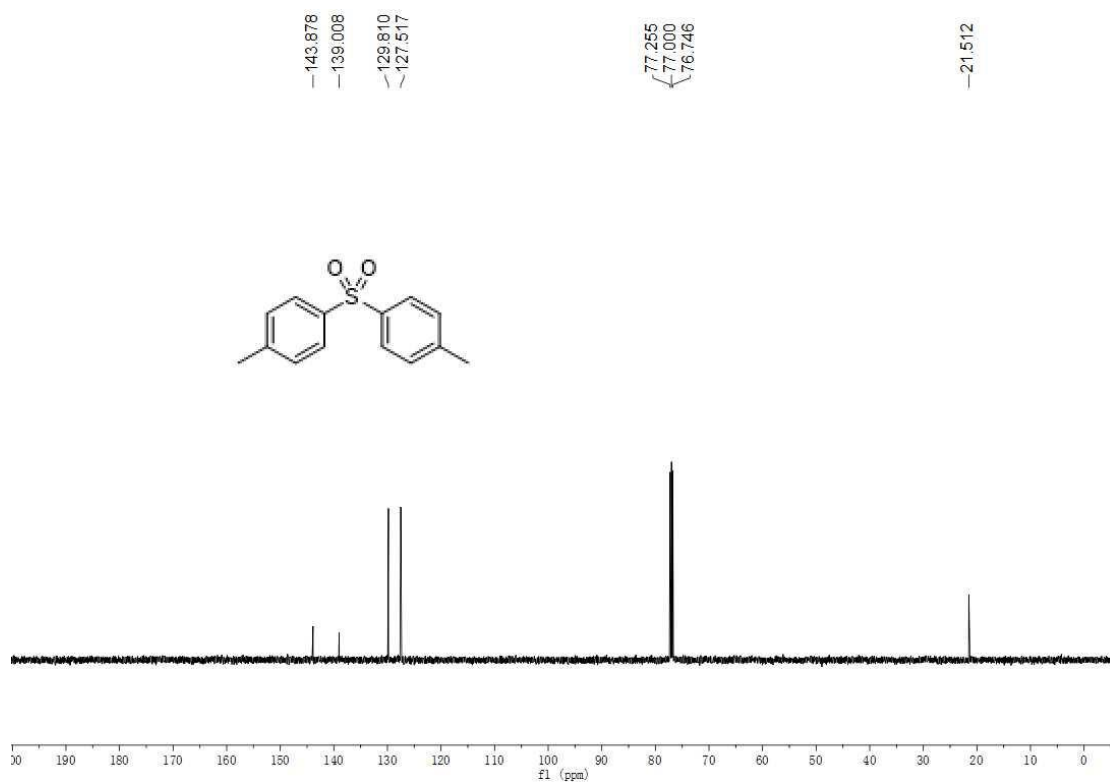


¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfonylbis(methylbenzene) (3o)

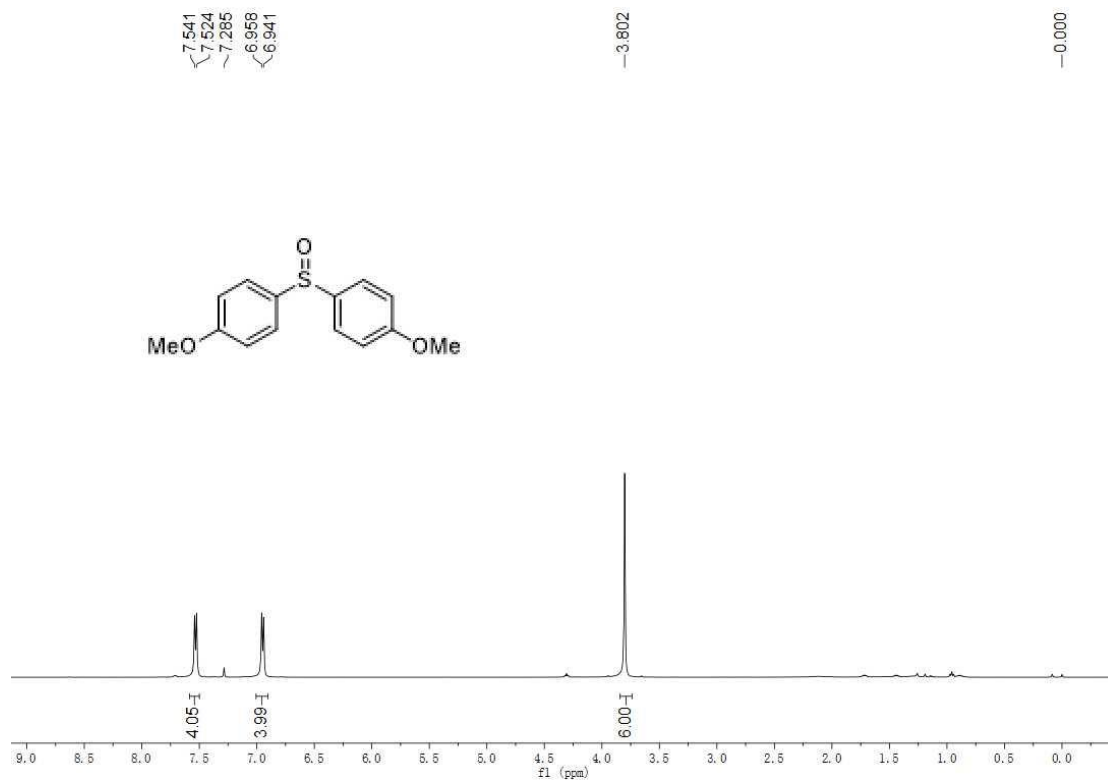


¹H NMR (500 MHz, CDCl₃)

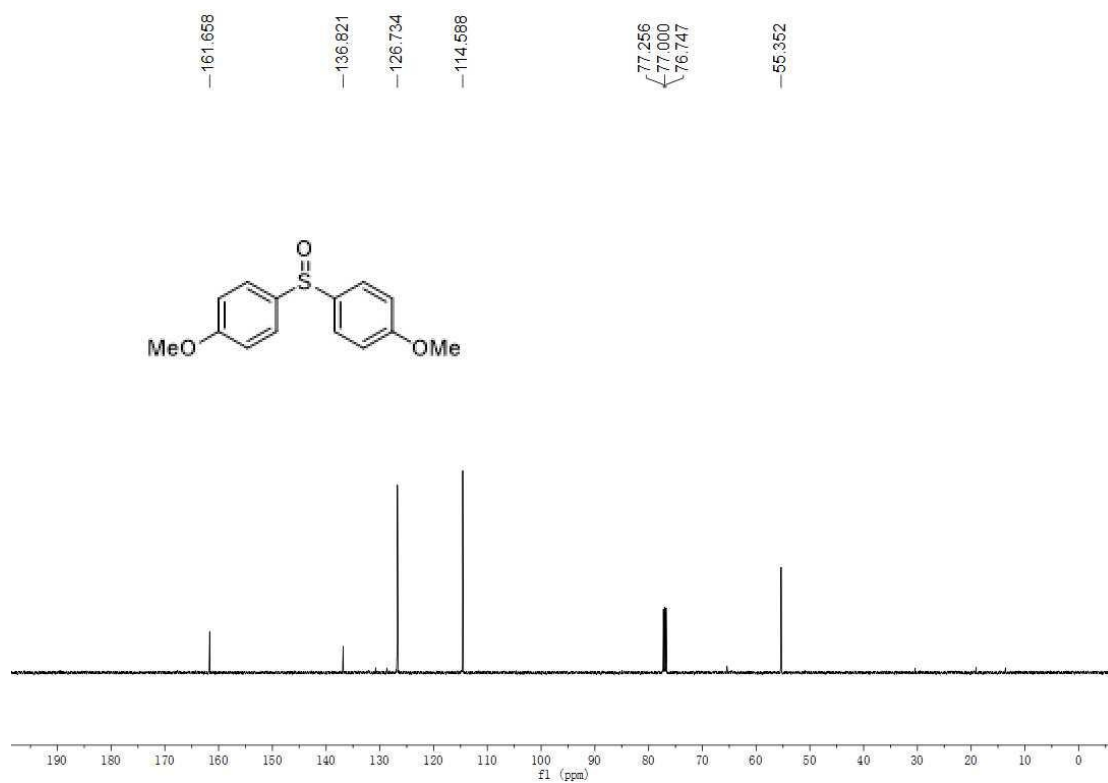


¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfinylbis(methoxybenzene) (2p)



¹H NMR (500 MHz, CDCl₃)

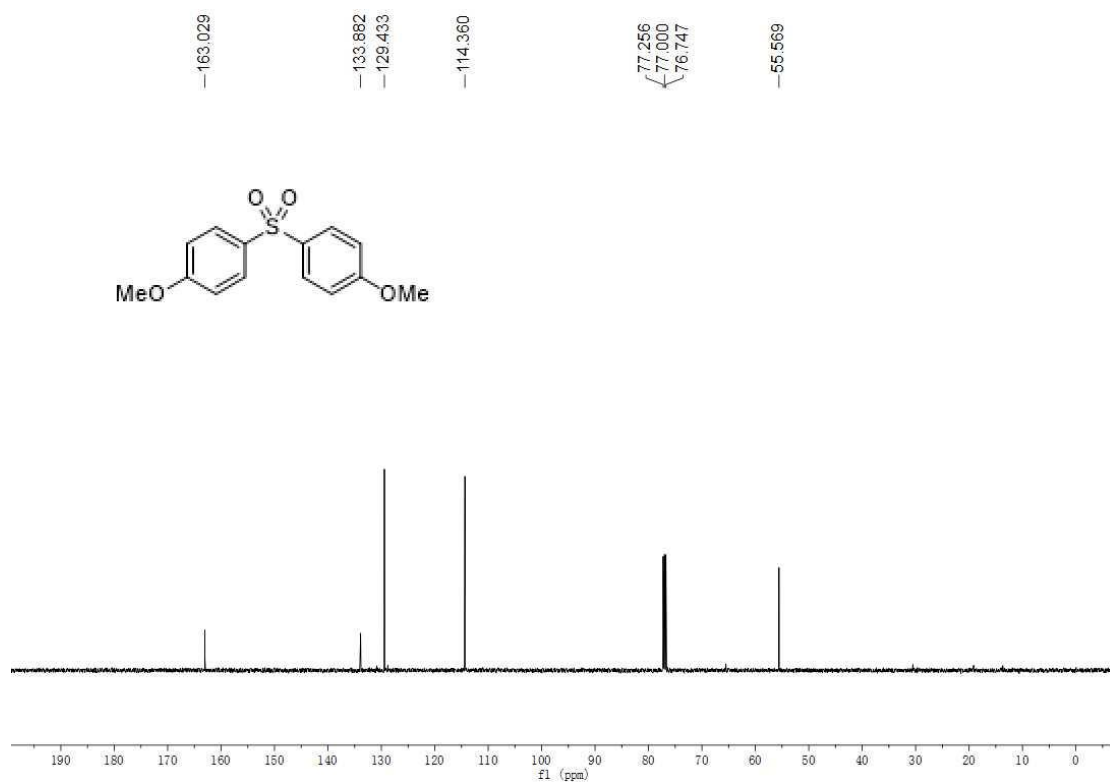


¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfonylbis(methoxybenzene) (3p)



¹H NMR (500 MHz, CDCl₃)

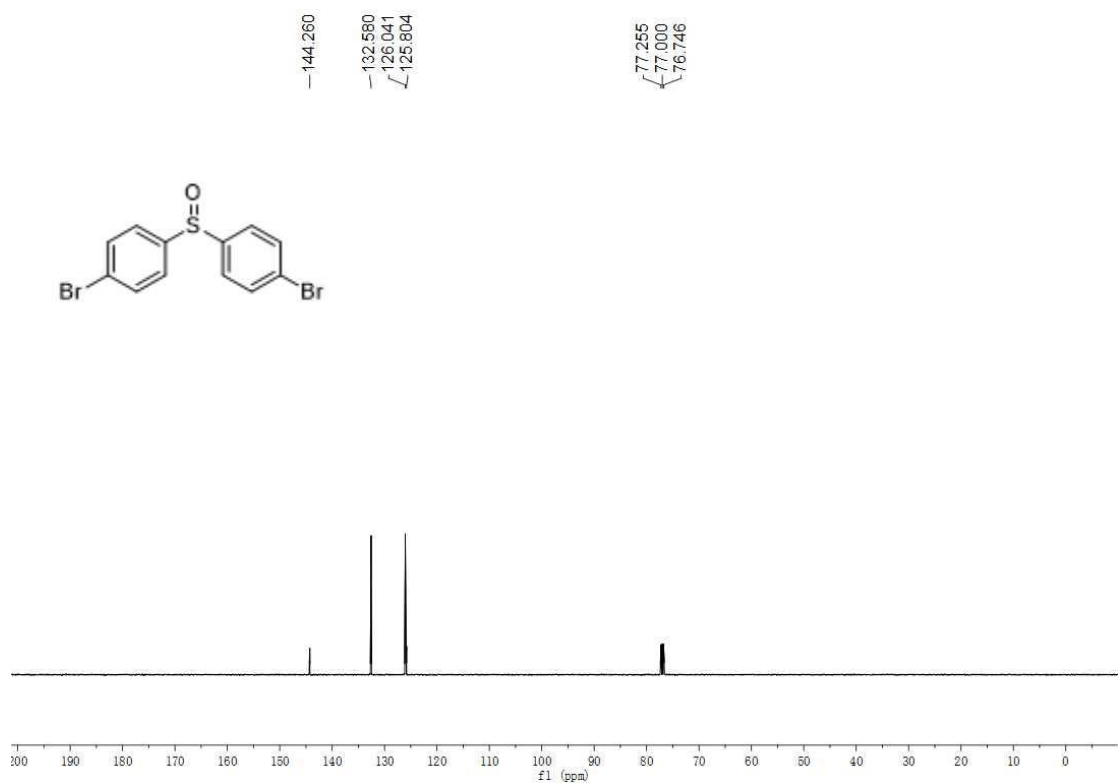


¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfinylbis(bromobenzene) (2q)

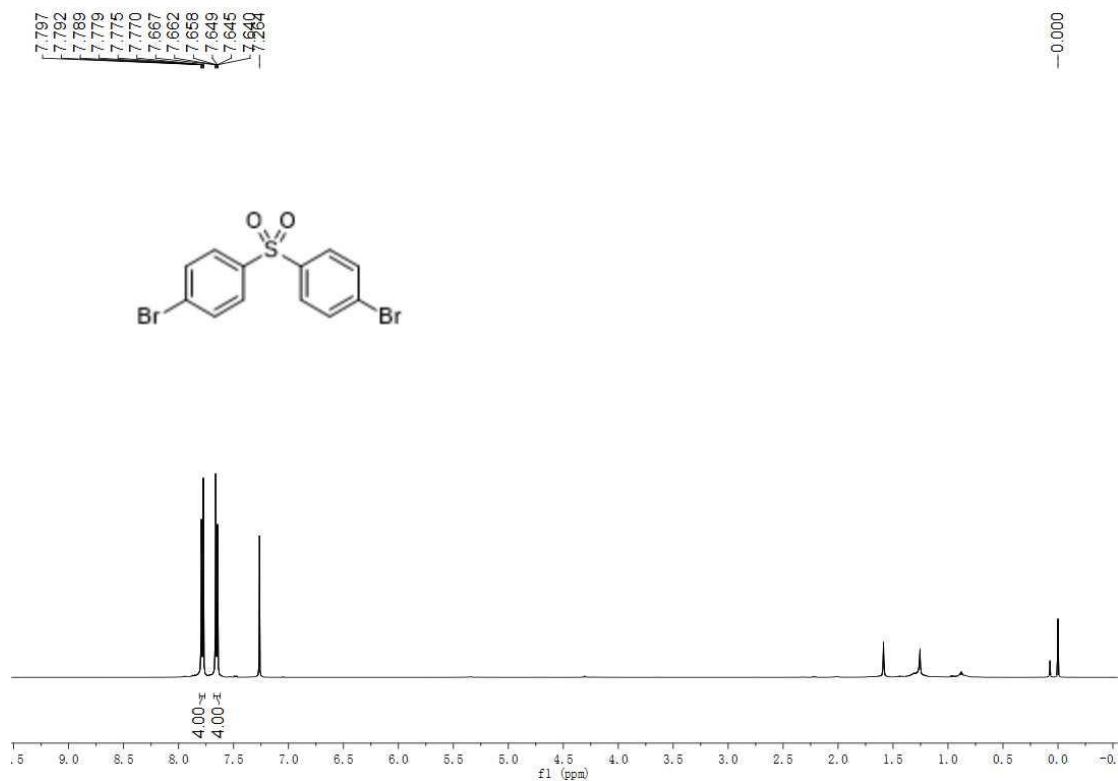


¹H NMR (500 MHz, CDCl₃)

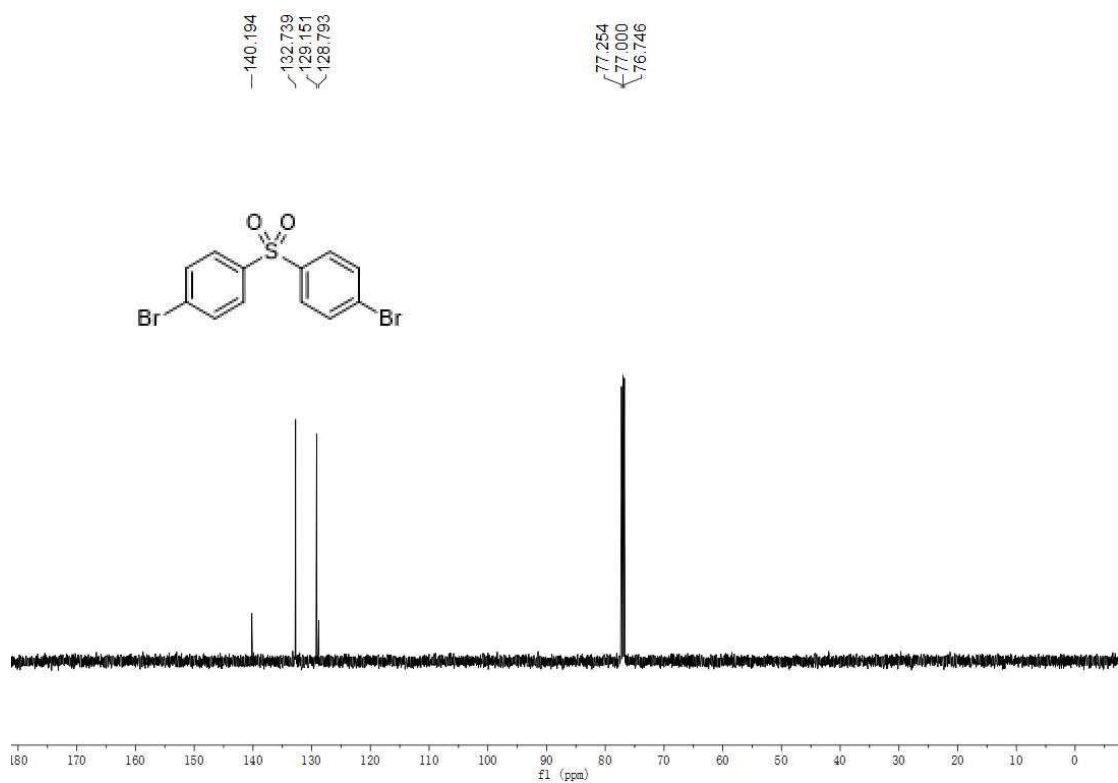


¹³C NMR (125 MHz, CDCl₃)

4,4'-sulfonylbis(bromobenzene) (3q)

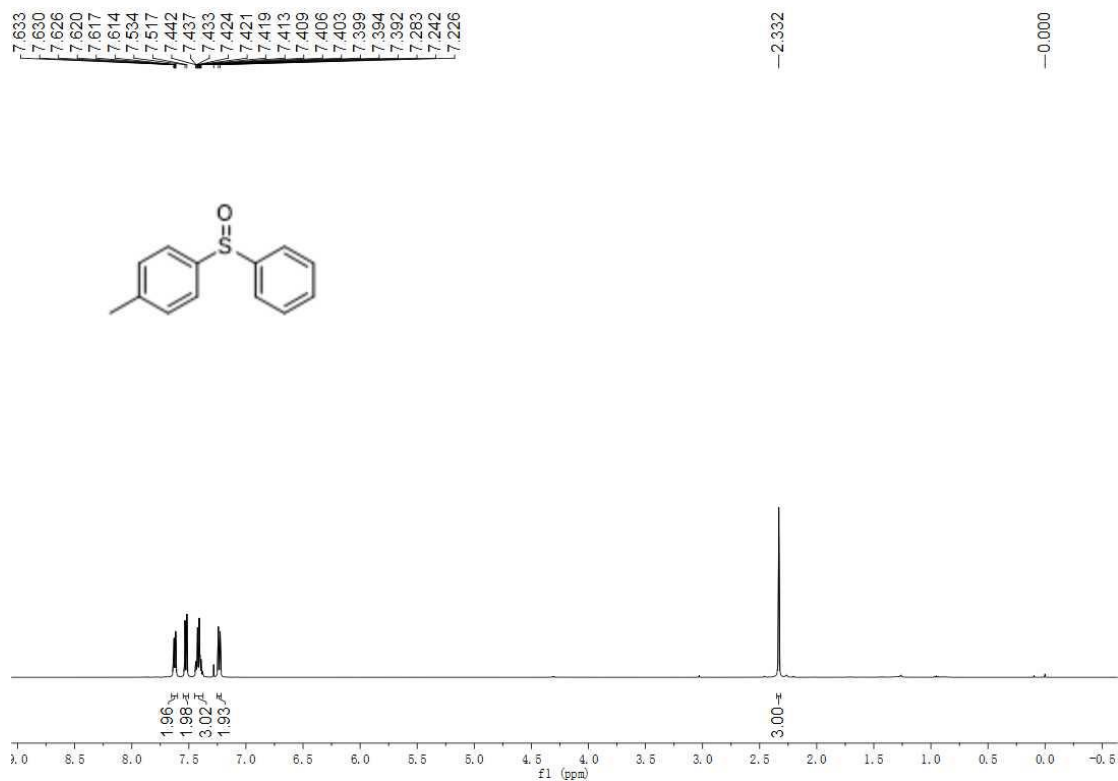


¹H NMR (500 MHz, CDCl₃)

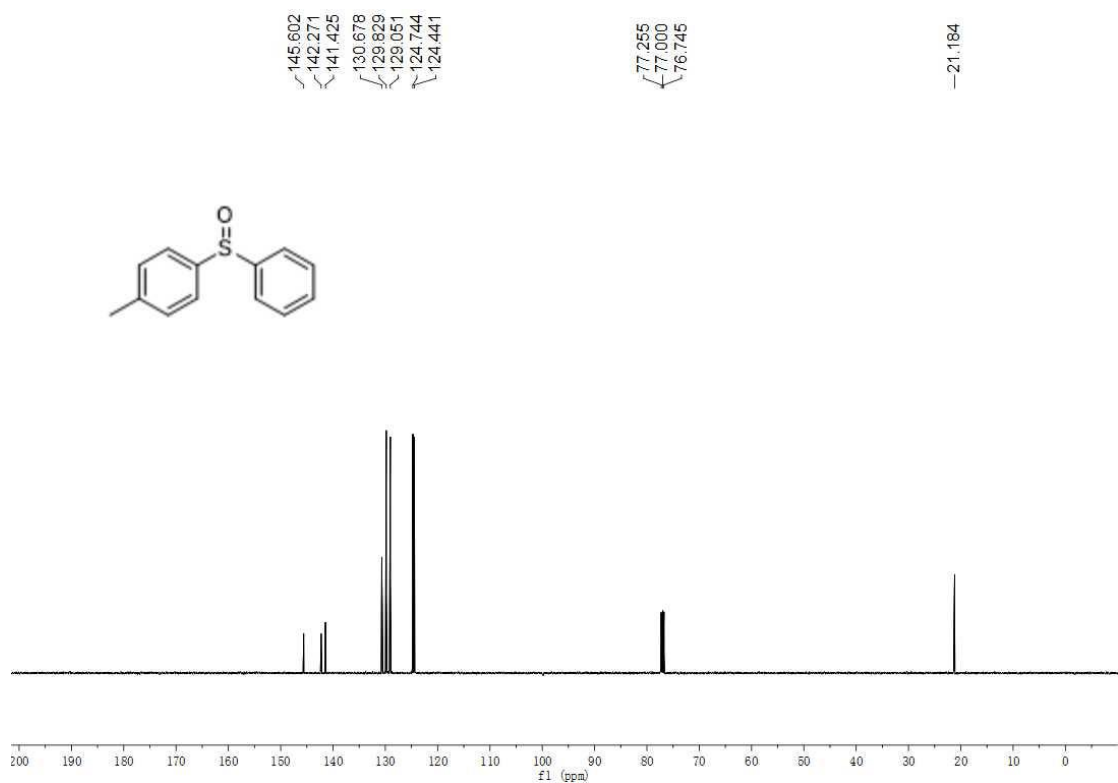


¹³C NMR (125 MHz, CDCl₃)

1-methyl-4-(phenylsulfinyl)benzene (2r)



¹H NMR (500 MHz, CDCl₃)

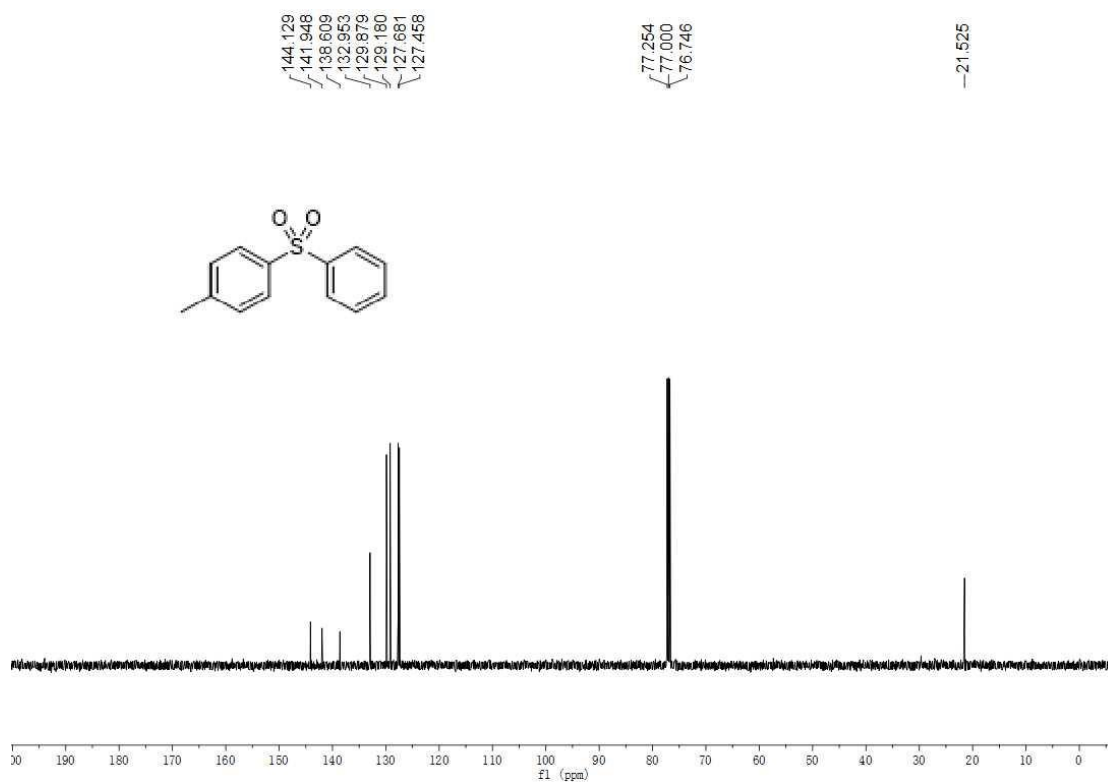


¹³C NMR (125 MHz, CDCl₃)

1-methyl-4-(phenylsulfonyl)benzene (3r)

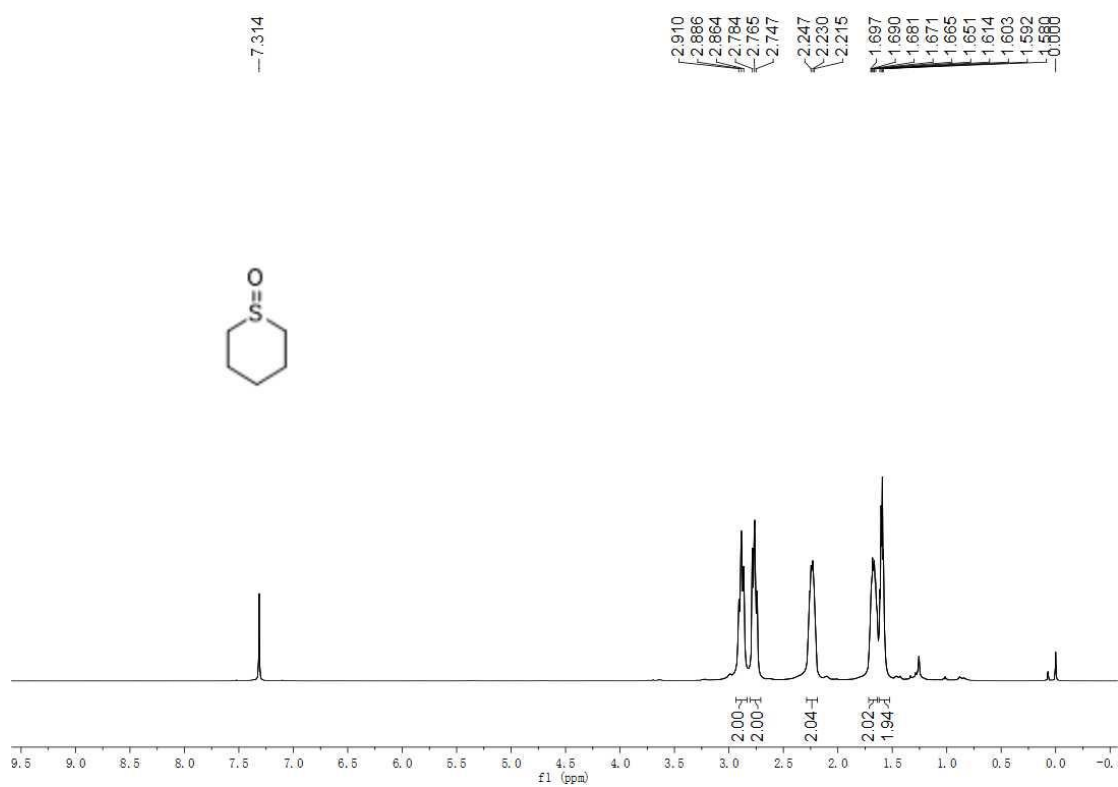


¹H NMR (500 MHz, CDCl₃)

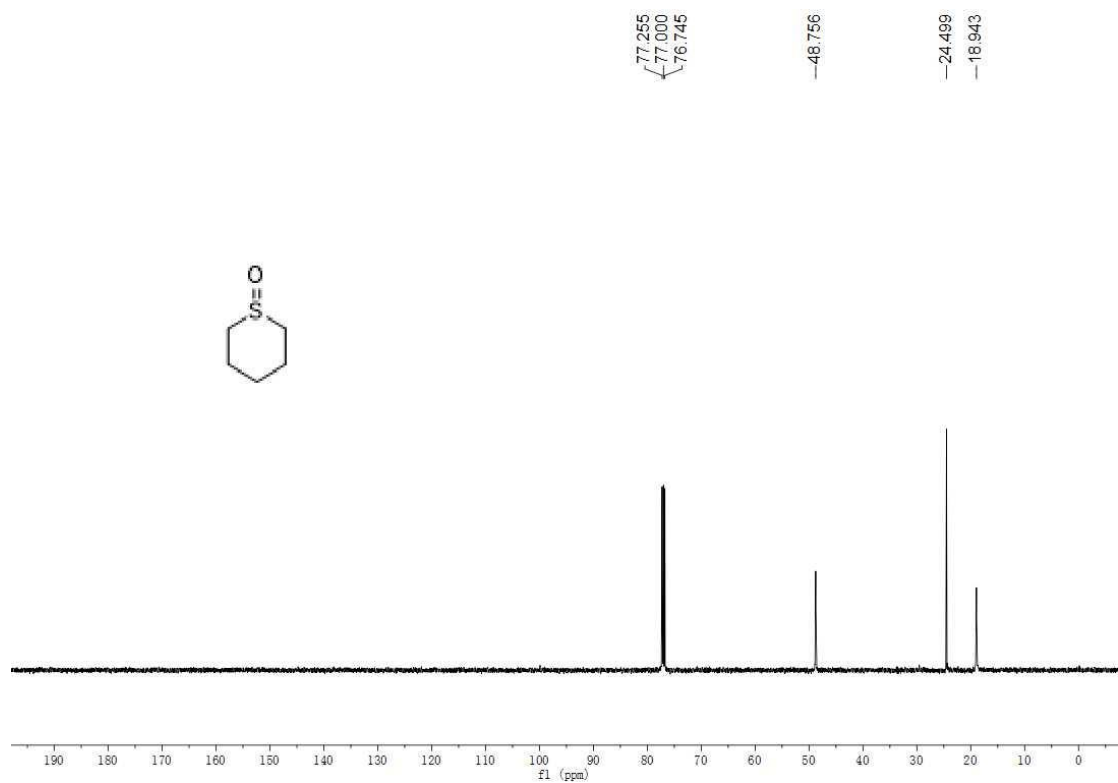


¹³C NMR (125 MHz, CDCl₃)

tetrahydro-2H-thiopyran 1-oxide (2s)

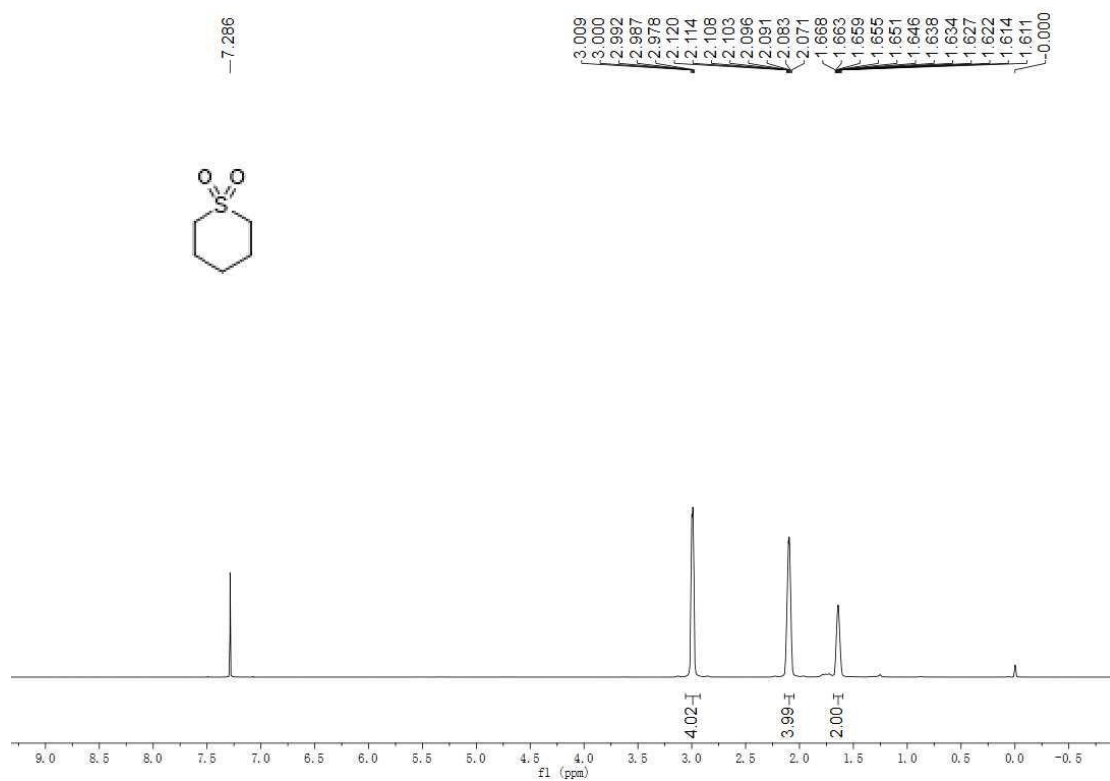


¹H NMR (500 MHz, CDCl₃)

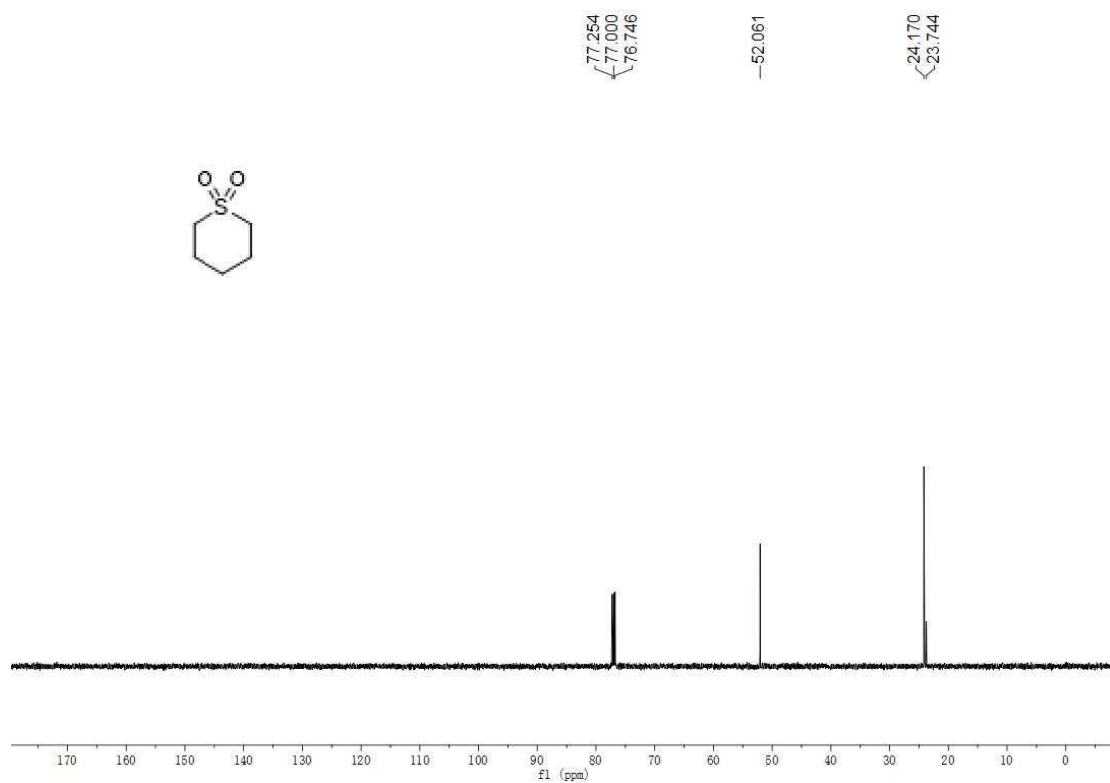


¹³C NMR (125 MHz, CDCl₃)

tetrahydro-2H-thiopyran 1,1-dioxide (3s)

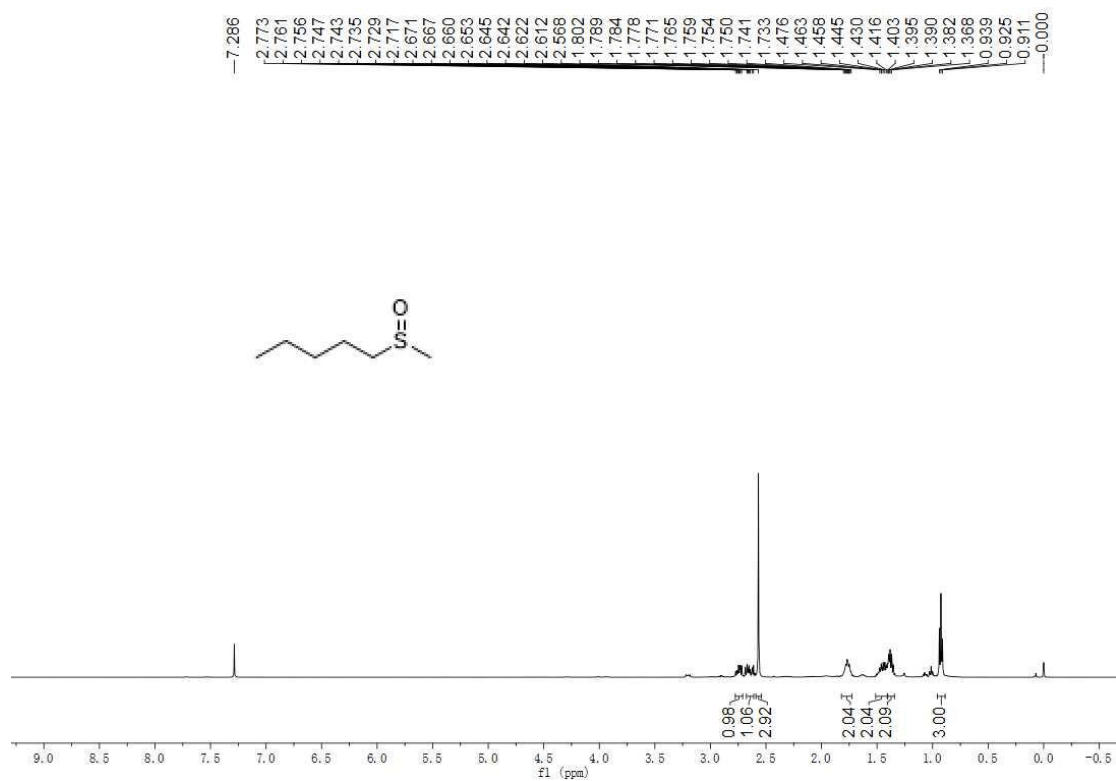


¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

2-(methylsulfonyl)pyridine (2t)

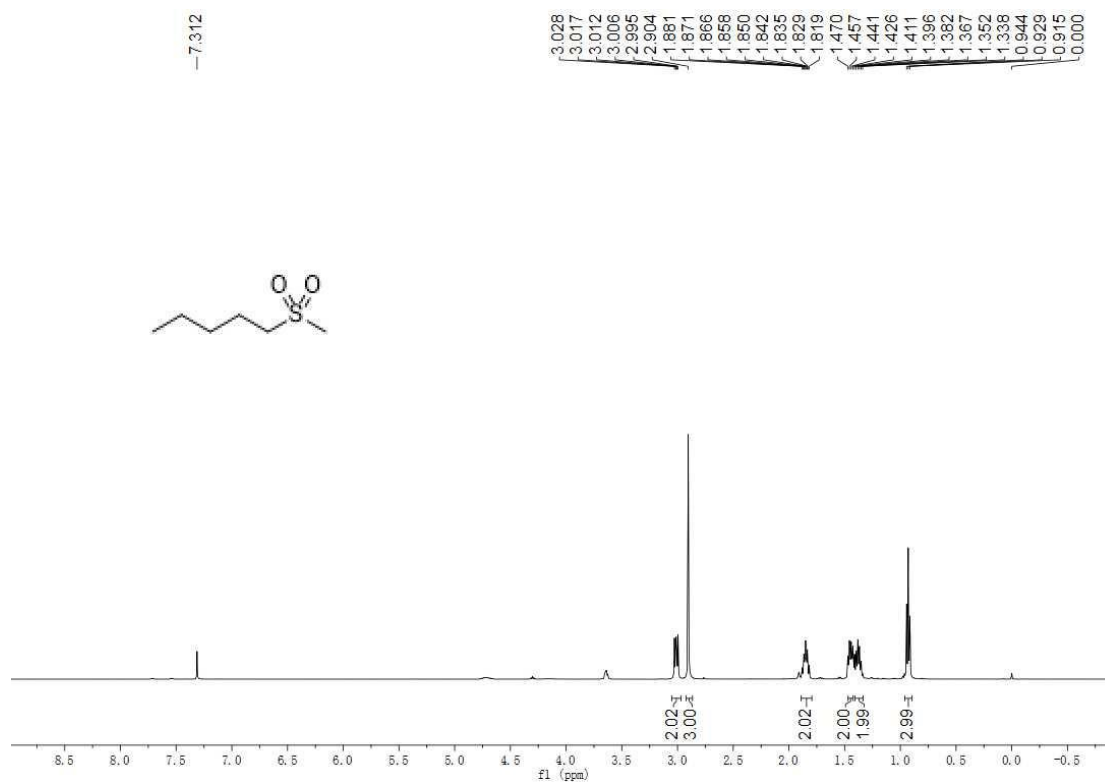


¹H NMR (500 MHz, CDCl₃)

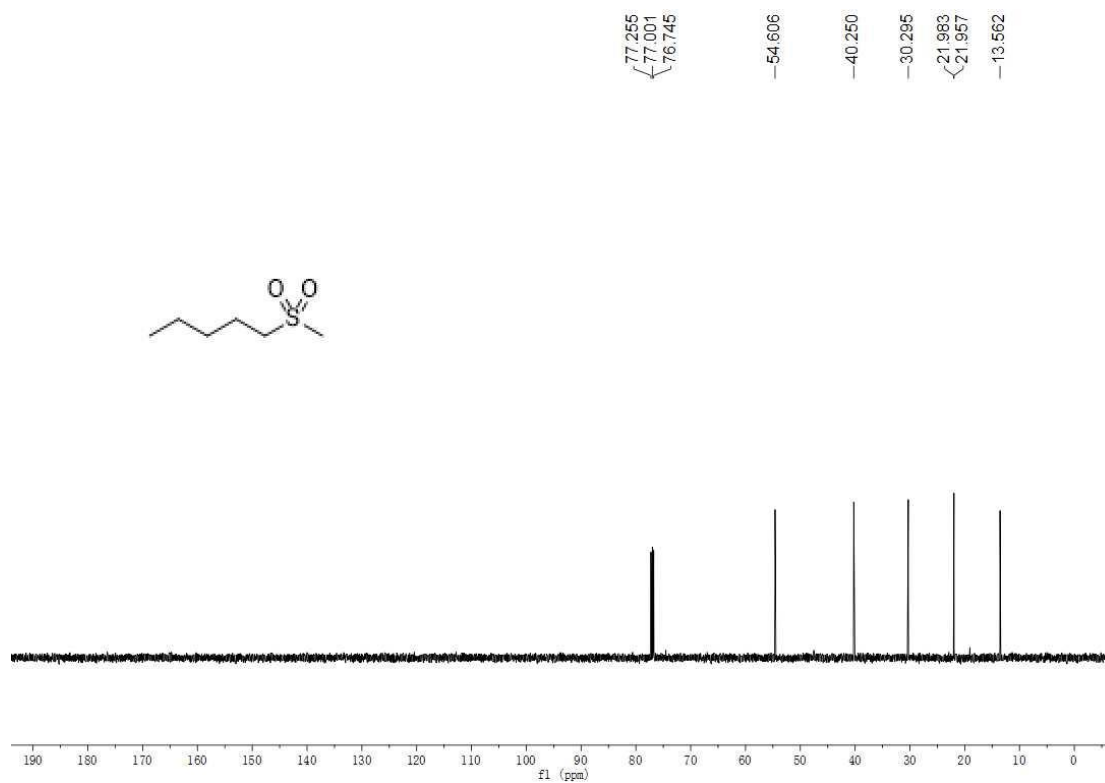


¹³C NMR (125 MHz, CDCl₃)

1-(methylsulfonyl)pentane (3t)

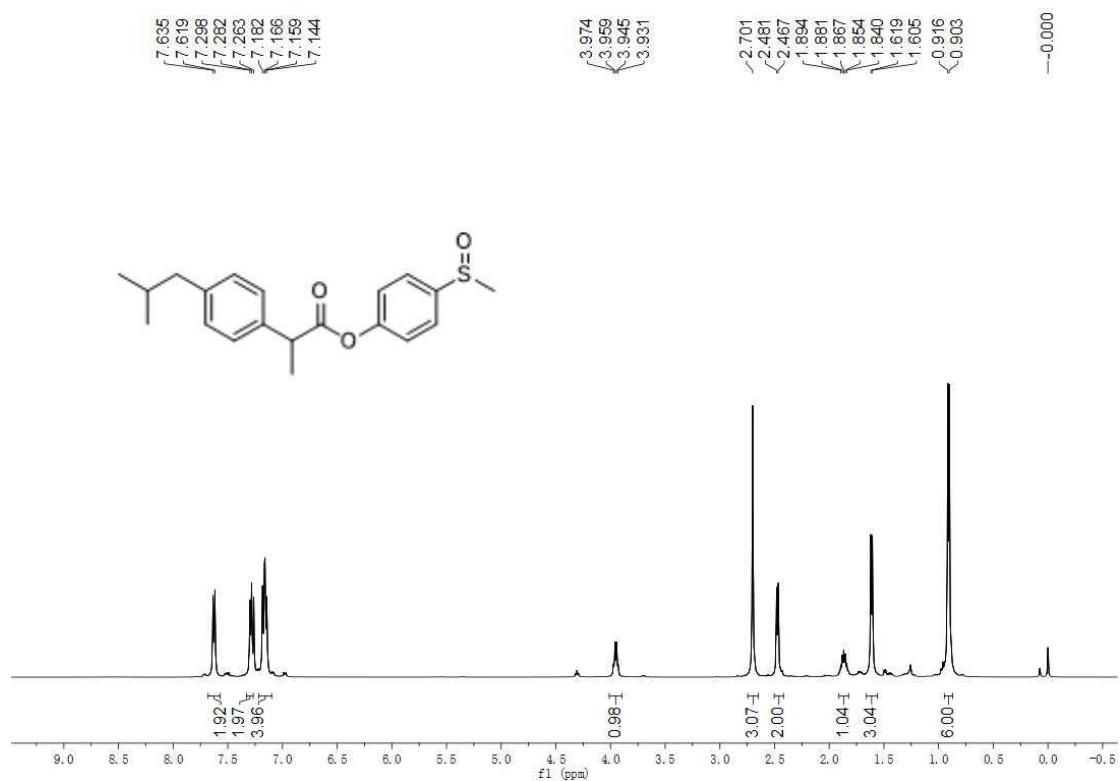


$^1\text{H NMR}$ (500 MHz, CDCl_3)

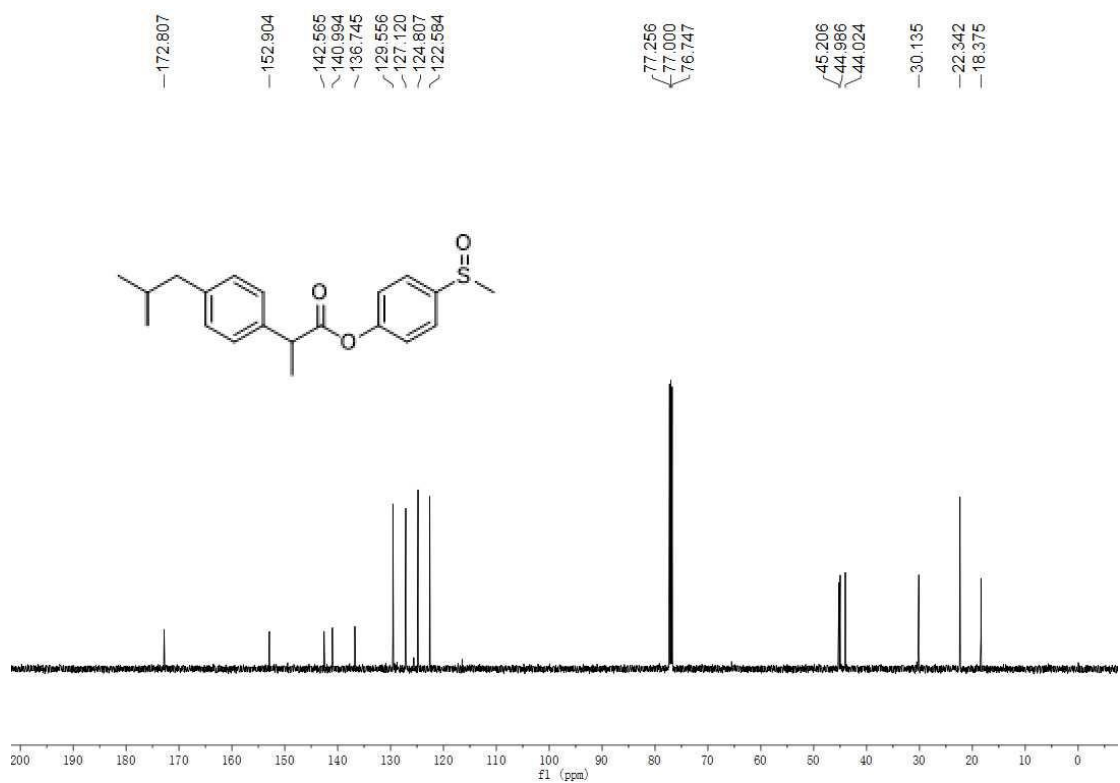


$^{13}\text{C NMR}$ (125 MHz, CDCl_3)

4-(methylsulfinyl)phenyl 2-(4-isobutylphenyl)propanoate (4a)

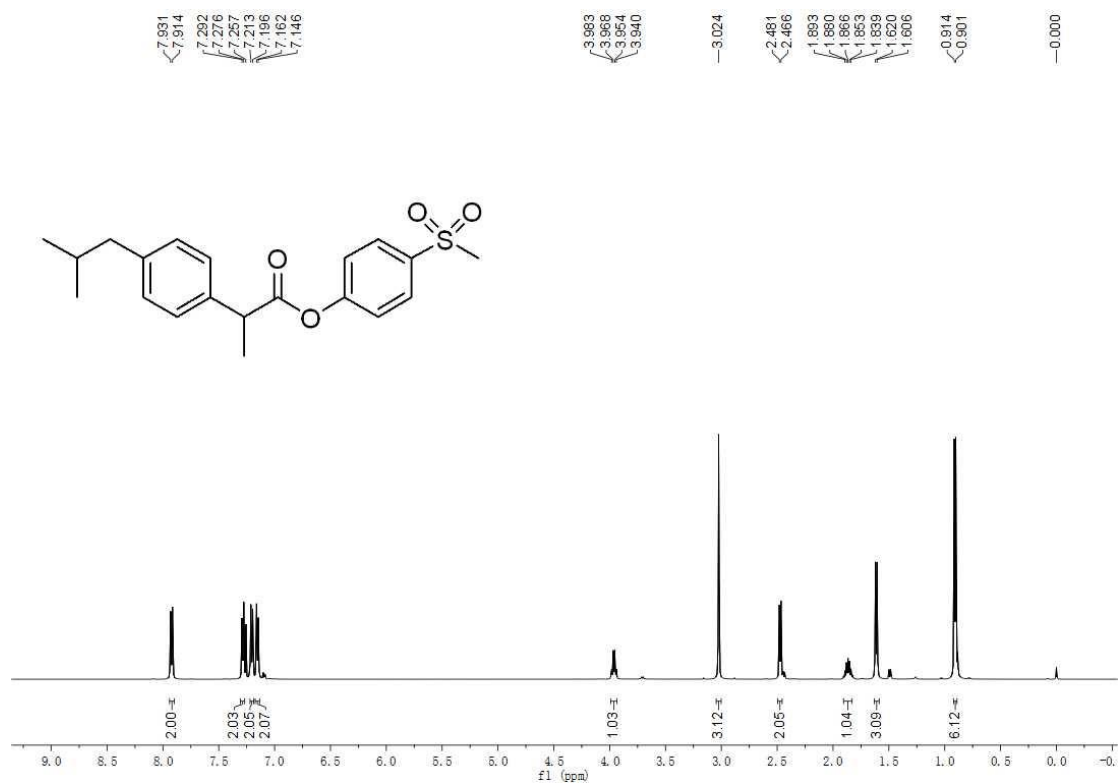


¹H NMR (500 MHz, CDCl₃)

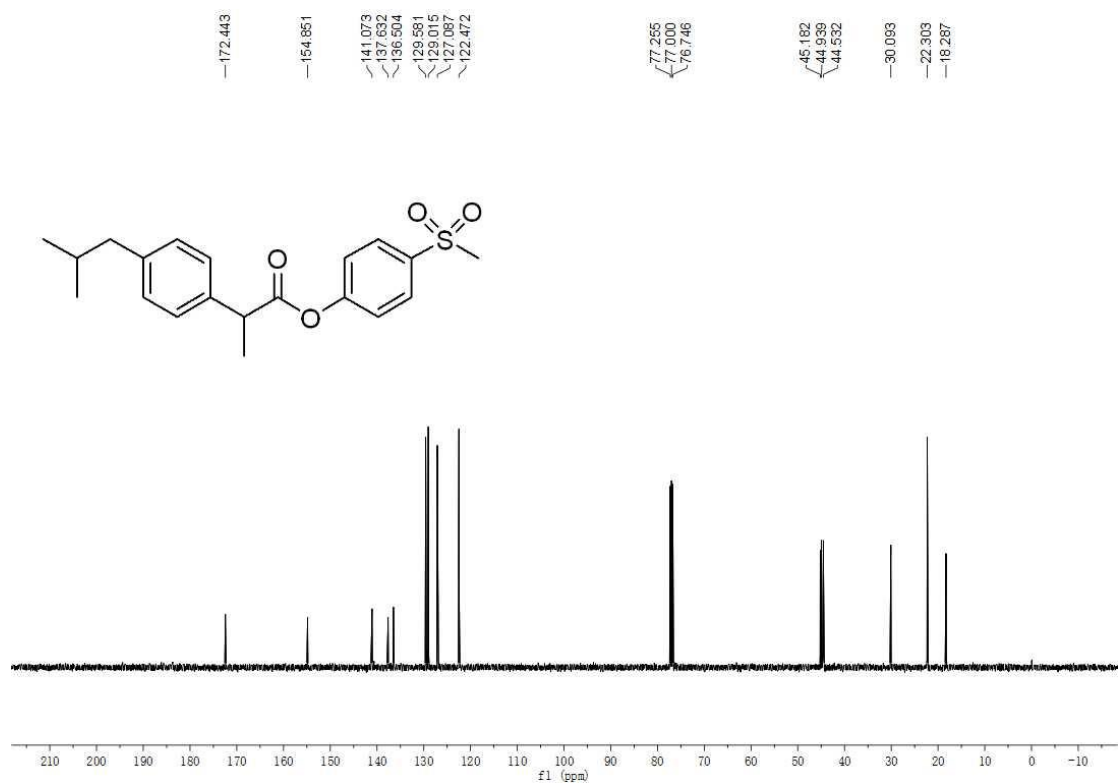


¹³C NMR (125 MHz, CDCl₃)

4-(methylsulfonyl)phenyl 2-(4-isobutylphenyl)propanoate (4b)

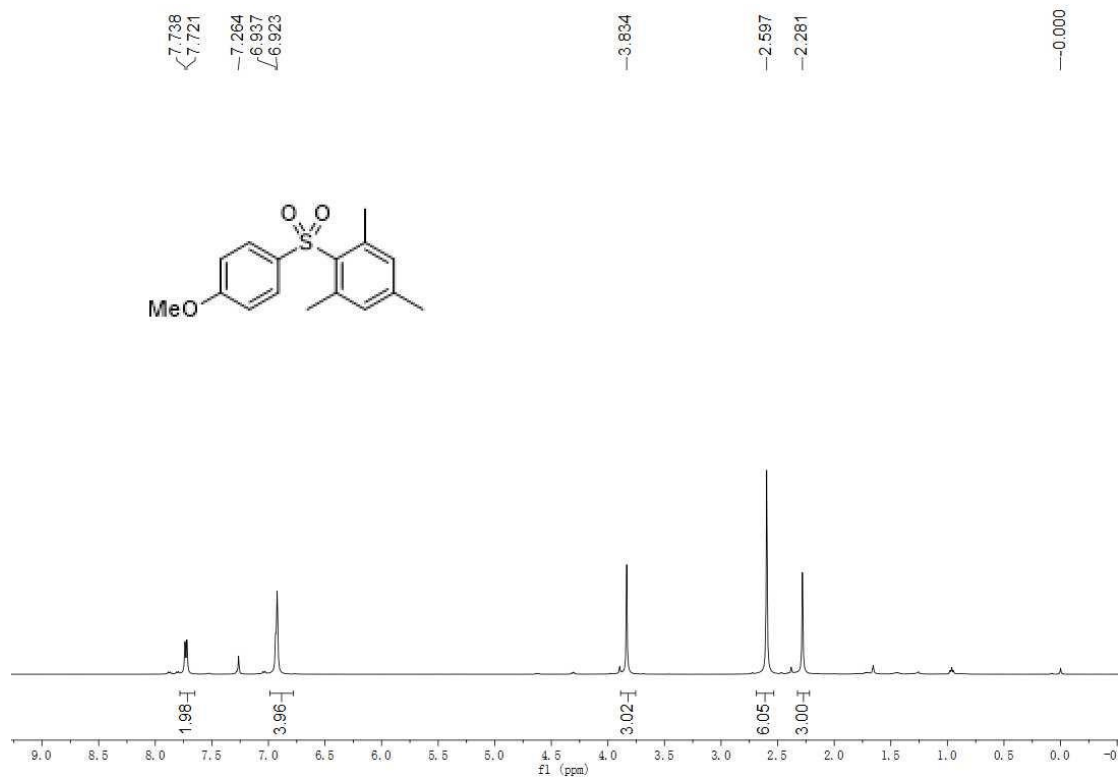


¹H NMR (500 MHz, CDCl₃)

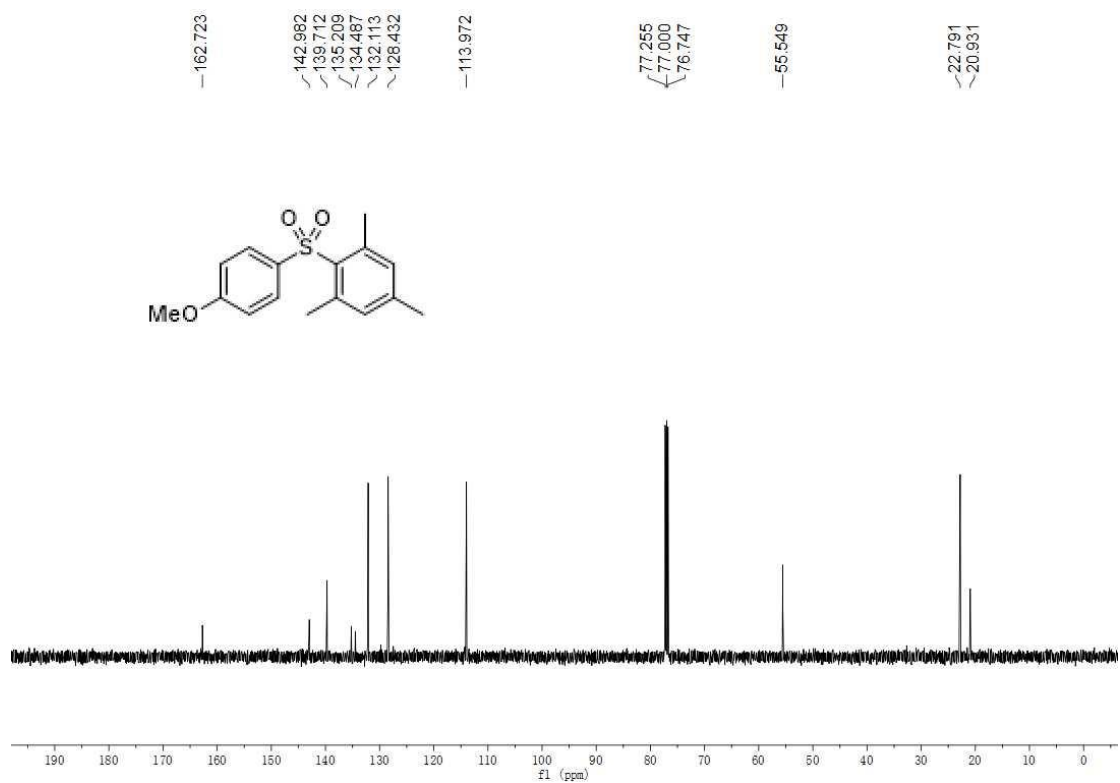


¹³C NMR (125 MHz, CDCl₃)

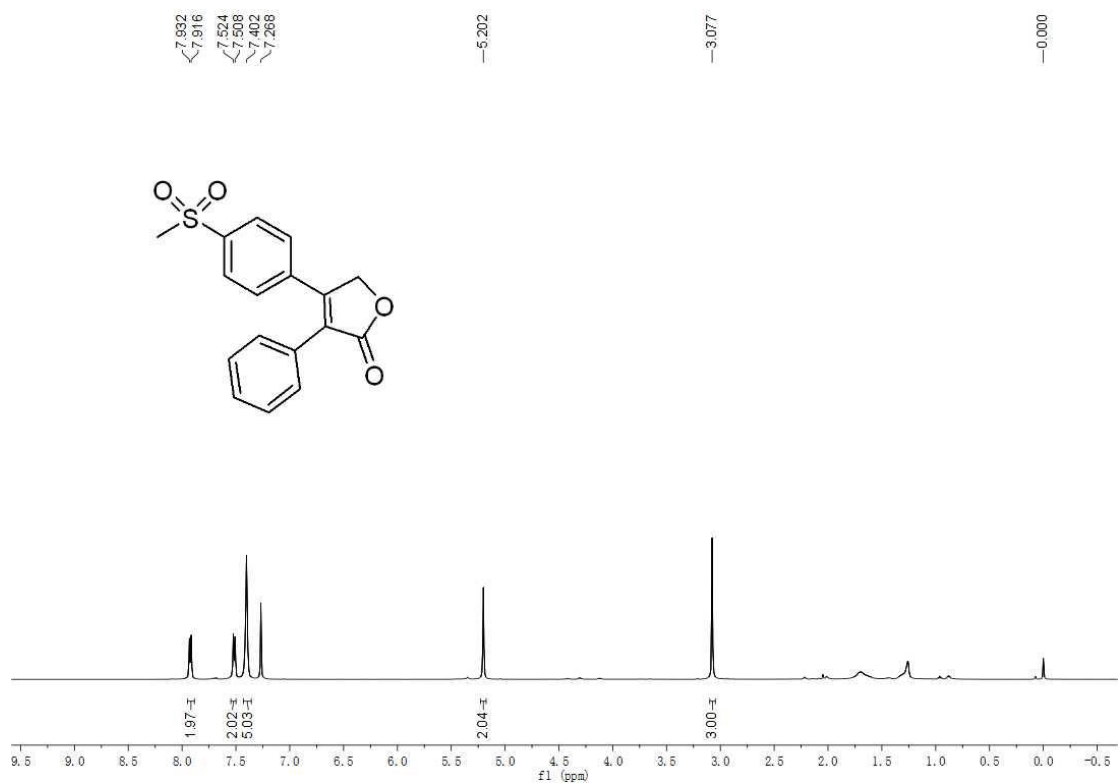
2-((4-methoxyphenyl)sulfonyl)-1,3,5-trimethylbenzene (4c)



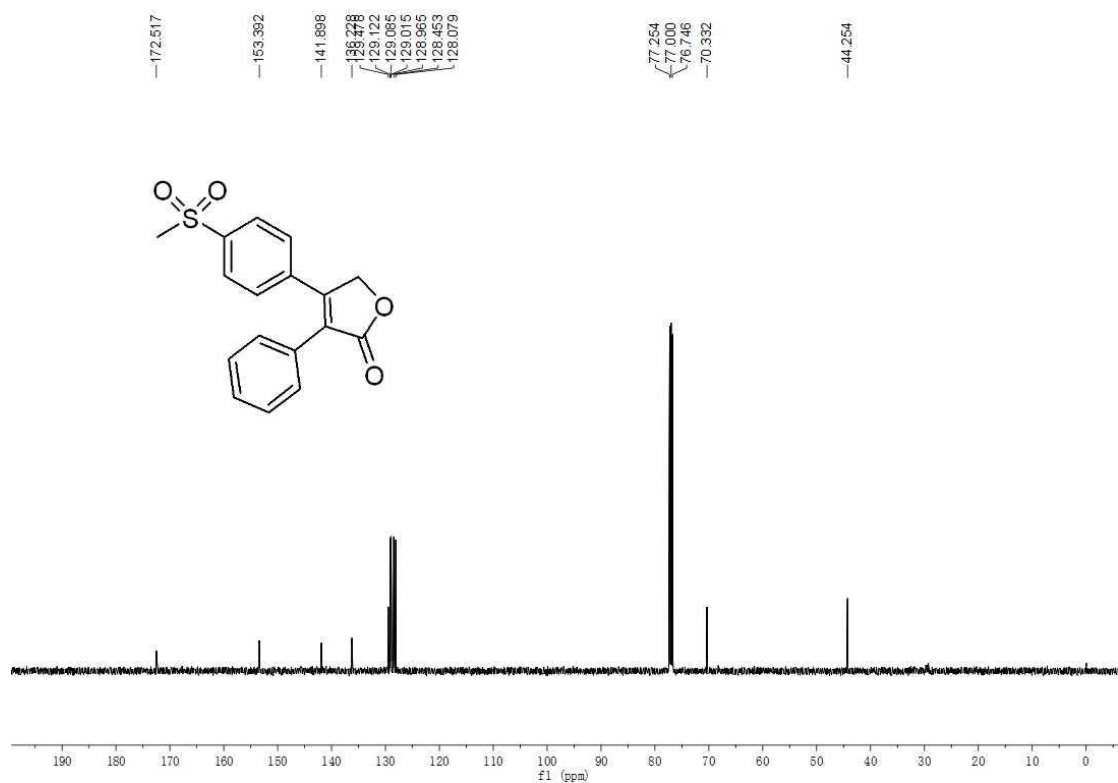
¹H NMR (500 MHz, CDCl₃)



4-(4-(methylsulfonyl)phenyl)-3-phenylfuran-2(5H)-one (4d)

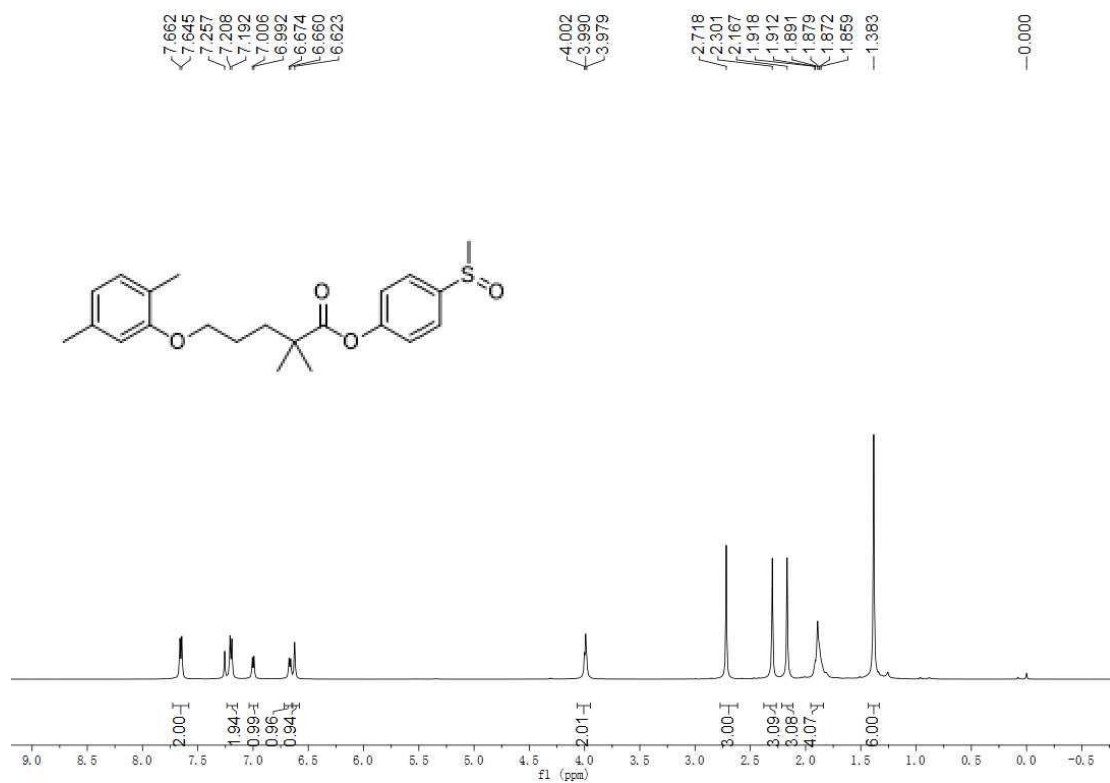


¹H NMR (500 MHz, CDCl₃)

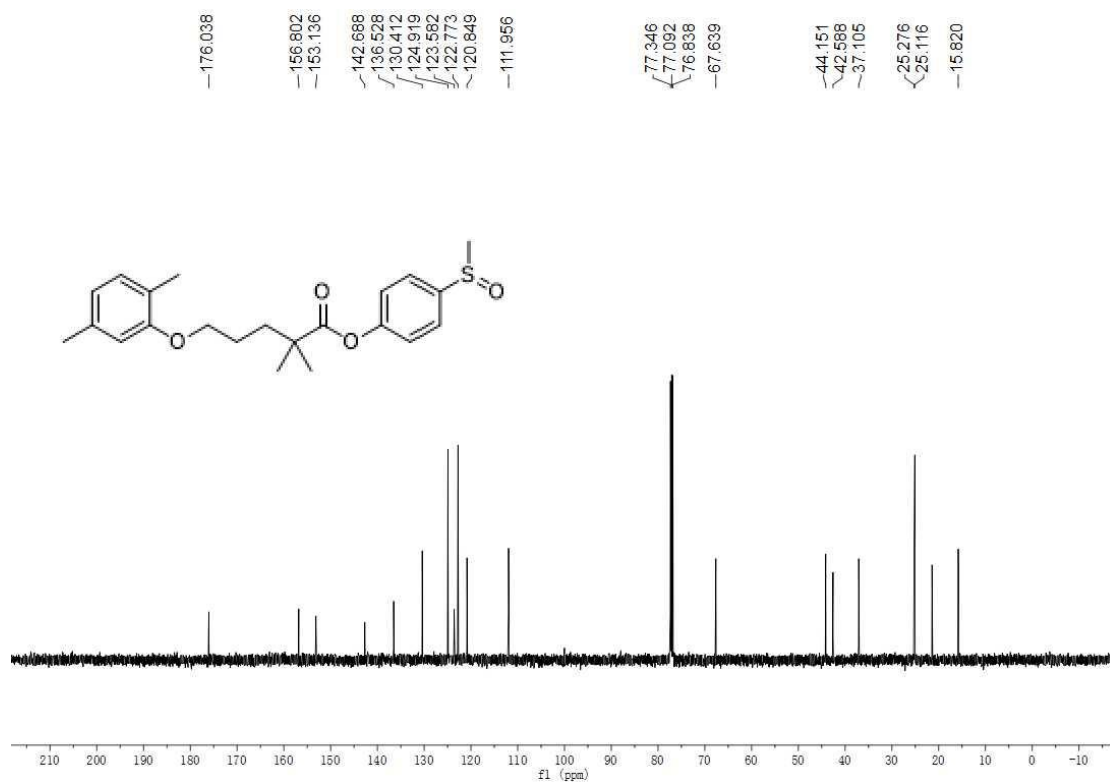


¹³C NMR (125 MHz, CDCl₃)

4-(methylsulfinyl)phenyl5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4e)

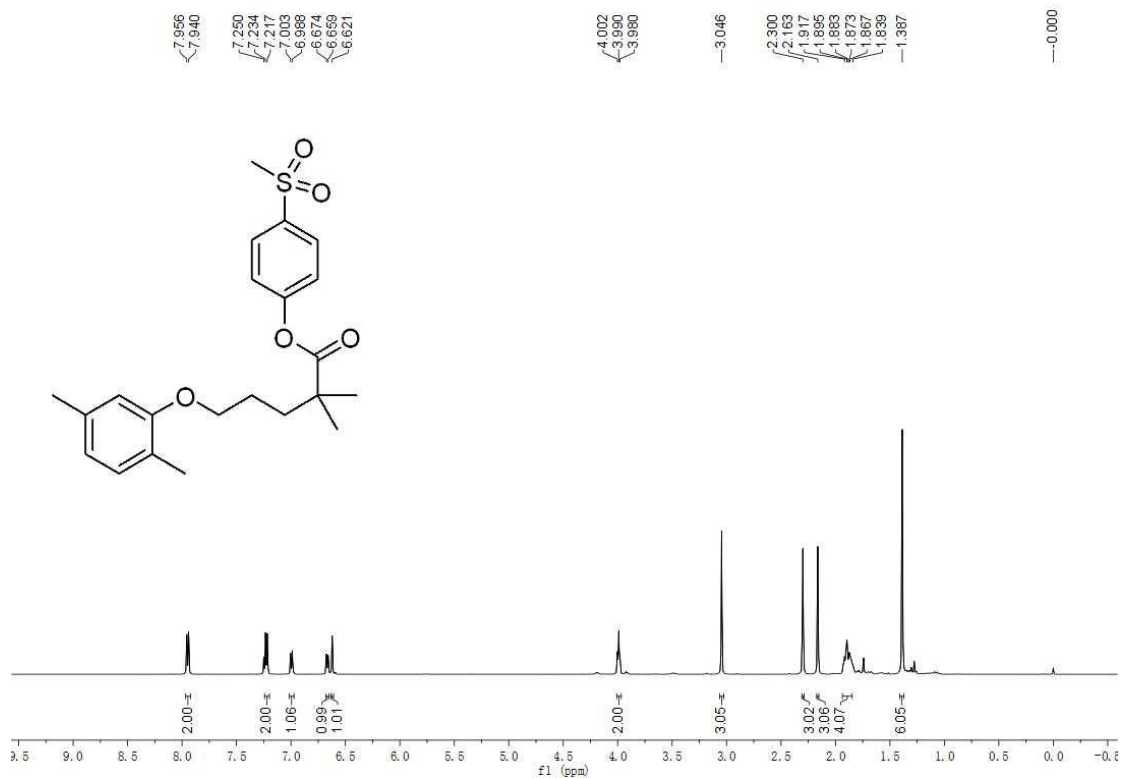


¹H NMR (500 MHz, CDCl₃)

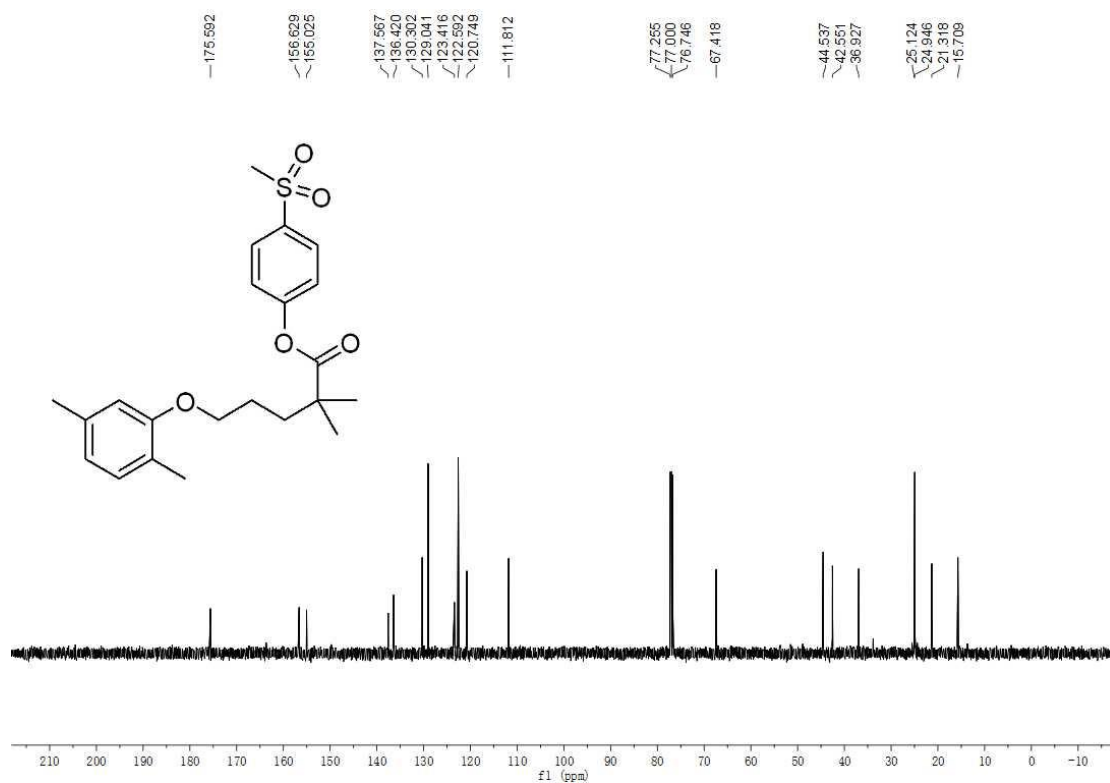


¹³C NMR (125 MHz, CDCl₃)

4-(methylsulfonyl)phenyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4f)



¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

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