



Supporting Information

for

Circumventing Mukaiyama oxidation: selective S–O bond formation via sulfenamide–alcohol coupling

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Beilstein J. Org. Chem. **2026**, 22, 158–166. doi:10.3762/bjoc.22.9

Experimental procedures, characterization data and copies of spectra

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(A) General information

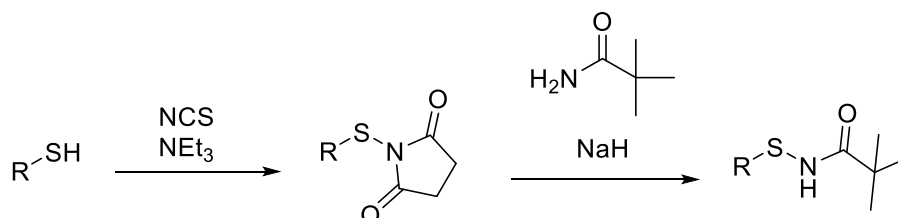
Chemicals and solvents were purchased from commercial suppliers and used as received unless noted. All products were purified by flash chromatography on silica gel. The chemical yields referred are isolated products. ^1H NMR and ^{13}C NMR spectra were recorded on 400 MHz Bruker spectrometers. Chemical shifts of ^1H NMR spectra were reported in parts per million relative to the TMS peak (δ 0.0). Chemical shifts of ^{13}C NMR spectra were reported relative to CDCl_3 (δ 77.16). The used abbreviations are as follows: s (singlet), d (doublet), t (triplet), quart. (quartet), quint. (quintet), m (multiplet), br (broad). Multiplets which arise from accidental equality of coupling constants of magnetically non-equivalent protons are marked as virtual (*virt.*). High-resolution mass spectra (HRMS) data were measured on an ESI-microTOF II. Melting points were measured on a SGW® X-4B and are not corrected. HPLC analyses were performed using a chiral stationary phase (ChiralPak IA, ChiralPak IB, ChiralPak IC, UV detection, Daicel Chemical Industries) employing *n*-hexane/*i*PrOH as eluents. Reactions were monitored by TLC analysis using silica gel 60 Å GF-254 thin layer plates and compounds were visualized with a UV light at 254 nm or 365 nm. Further visualization was achieved by staining with iodine, or KMnO_4 followed by heating on a hot plate. Flash column chromatography was performed on silica gel 60 Å, 10–40 μm .

The substrates were prepared following literature procedures ¹⁻³ which are provided in section B.

(B) Procedures for the synthesis of substrates

The sulfenamides were prepared following the literature procedure reported by our groups. ¹

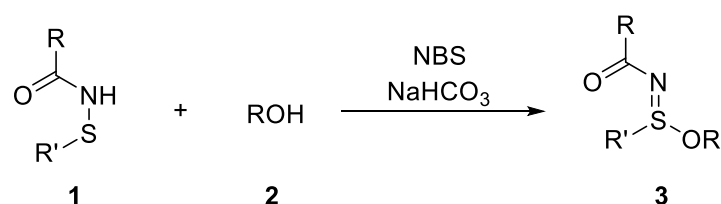
General scheme for the two-step synthesis of sulfenamides from *N*-thiosuccinimides.



General procedure for the synthesis of *N*-thiosuccinimides: To a stirred solution of the indicated thiol (5.0 mmol, 1.0 equiv) in DCM (0.25 M) under nitrogen was added *N*-chlorosuccinimide at room temperature. After stirring at room temperature for one hour, a solution of triethylamine (5.0 mmol, 1.0 equiv) in DCM (0.7 M) was added dropwise over 30 minutes. The reaction mixture was heated to 40 °C overnight. After cooling to room temperature, the reaction mixture was diluted with H_2O , and extracted 3× with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4 , and concentrated. The crude residue was purified through column chromatography.

General procedure for the synthesis of sulfenamides: To a flame-dried round-bottomed flask under nitrogen and equipped with an addition funnel and stirring bar was added pivalamide (1.0 mmol, 1.0 equiv). THF (0.15 M) was added and the solution was cooled to 0 °C. NaH (2.5 equiv) was added, then the solution was warmed to room temperature and stirred for 30 minutes. The addition funnel was charged with a solution of the indicated thiosuccinimide (1.0 mmol, 1.0 equiv) in THF (0.3 M), which was added dropwise over one hour. Upon completion of the addition, the reaction mixture was stirred at room temperature for 30 minutes, then quenched with saturated aqueous ammonium chloride and extracted 3× with ethyl acetate. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated. The crude residue was purified through column chromatography.

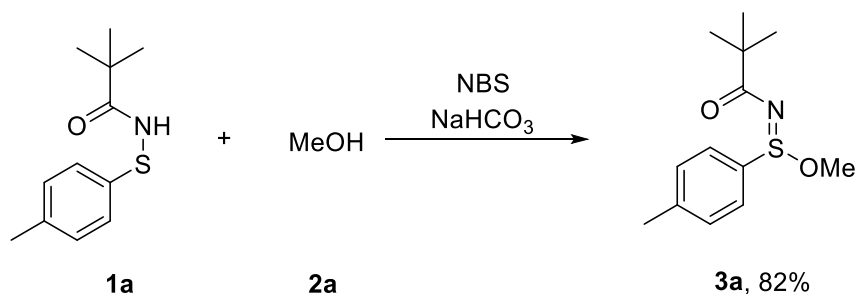
(C) Procedures for the synthesis of products



General procedure 1: A solution of sulfenamides **1** (0.15 mmol) and NaHCO₃ (0.225 mmol, 1.5 equiv) was prepared in ROH **2** (1.5 mL). To this, NBS (0.18 mmol, 1.2 equiv) was added at room temperature. The mixture was stirred at room temperature for 0.5 hours. Following this, the reaction was quenched using a saturated NH₄Cl solution. The resulting mixture was then extracted with ethyl acetate (EtOAc). The combined organic phases were subsequently washed with brine, dried over Na₂SO₄, and the solvents were evaporated. The residual crude material was then subjected to column chromatography, yielding the desired product.

General procedure 2: A solution of sulfenamides **1** (0.15 mmol), ROH **2** (10 equiv) and NaHCO₃ (0.225 mmol, 1.5 equiv) was prepared in DCM (1.5 mL). To this, NBS (0.18 mmol, 1.2 equiv) was added at room temperature. The mixture was stirred at room temperature for 0.5 hours. Following this, the reaction was quenched using a saturated NH₄Cl solution. The resulting mixture was then extracted with ethyl acetate (EtOAc). The combined organic phases were subsequently washed with brine, dried over Na₂SO₄, and the solvents were evaporated. The residual crude material was then subjected to column chromatography, yielding the desired product.

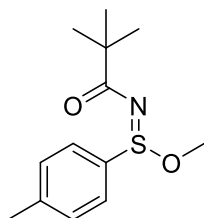
(D) Scale-up reaction



A solution of sulfenamides **1a** (670.0 mg, 3 mmol) and NaHCO₃ (378.0 mg, 1.5 equiv, 4.5 mmol) was prepared in MeOH (25 mL). To this, NBS (640.7 mg, 1.2 equiv, 3.6 mmol) was added at room temperature. The mixture was stirred at room temperature for 1.0 hour. The reaction was quenched with saturated aqueous NH₄Cl, and the resulting mixture was extracted with EtOAc (3 × 20 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography to afford compound **3a** (623.3 mg, 82% yield).

(E) Analytical data of products

Methyl *N*-(pivaloyl)-4-methylphenylsulfinimide (3a**)**



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

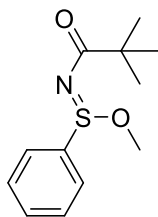
A colorless oil, 37.6 mg, 99% yield.

TLC: R_f = 0.52 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.79 – 7.69 (m, 2H), 7.33 – 7.23 (m, 2H), 3.47 (s, 3H), 2.37 (s, 3H), 1.23 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 193.0, 143.6, 133.0, 130.1, 128.0, 51.8, 41.3, 28.4, 21.7. Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)phenylsulfinimide (3b)



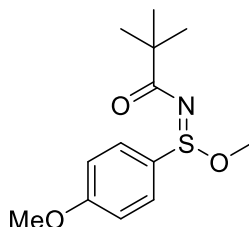
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 34.8 mg, 97% yield.

TLC: R_f = 0.65 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.84 (m, 2H), 7.57 – 7.45 (m, 3H), 3.50 (s, 3H), 1.24 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-4-methoxyphenylsulfinimide (3c)



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 34.3 mg, 85% yield.

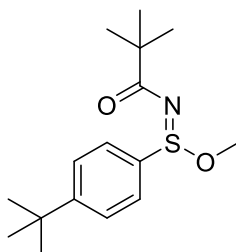
TLC: R_f = 0.42 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.78 (m, 2H), 7.01 – 6.95 (m, 2H), 3.81 (s, 3H), 3.45 (s, 3H), 1.23 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 192.8, 163.3, 130.0, 127.2, 114.8, 55.8, 51.5, 41.3, 28.4.

HRMS (ESI+): m/z calcd for $\text{C}_{13}\text{H}_{19}\text{NO}_3\text{SNa}^+$ $[\text{M}+\text{Na}]^+$: 292.0978, found: 292.0977. Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-4-*tert*-butylphenylsulfinimide (3d)



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 38.1 mg, 86% yield.

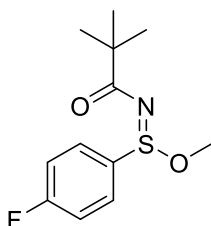
TLC: R_f = 0.52 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.85 (m, 2H), 7.63 – 7.57 (m, 2H), 3.59 (s, 3H), 1.37 (s, 9H), 1.32 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 193.0, 156.6, 133.0, 127.9, 126.5, 52.1, 41.3, 35.3, 31.3, 28.4.

HRMS (ESI $^{+}$): m/z calcd for $\text{C}_{16}\text{H}_{25}\text{NO}_2\text{SNa}^{+}$ $[\text{M}+\text{Na}]^{+}$: 318.1498, found: 318.1498.

Methyl *N*-(pivaloyl)-4-fluorophenylsulfonimide (3e)



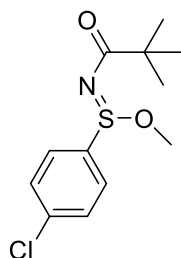
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 32.0 mg, 83% yield.

TLC: R_f = 0.66 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.85 (m, 2H), 7.23 – 7.14 (m, 2H), 3.50 (s, 3H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-4-chlorophenylsulfonimide (3f)



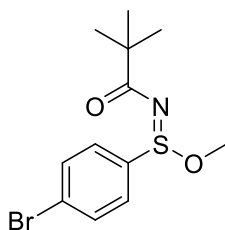
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.5 mg, 89% yield.

TLC: R_f = 0.63 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.77 (m, 2H), 7.50 – 7.45 (m, 2H), 3.51 (s, 3H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-4-bromophenylsulfonimide (3g)



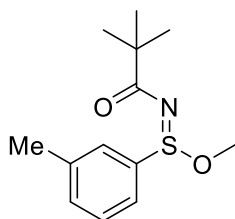
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 39.1 mg, 82% yield.

TLC: R_f = 0.55 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.71 (m, 2H), 7.66 – 7.60 (m, 2H), 3.51 (s, 3H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-3-methylphenylsulfonimide (3h)



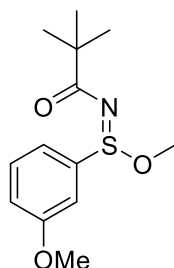
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 29.3 mg, 77% yield.

TLC: R_f = 0.65 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.71 – 7.61 (m, 2H), 7.41 – 7.29 (m, 2H), 3.50 (s, 3H), 2.39 (s, 3H), 1.24 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-3-methoxyphenylsulfonimide (3i)



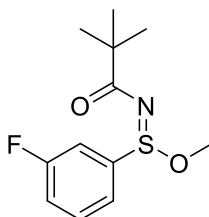
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.8 mg, 91% yield.

TLC: R_f = 0.65 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.44 (m, 1H), 7.42 – 7.38 (m, 2H), 7.08 – 7.02 (m, 1H), 3.81 (s, 3H), 3.49 (s, 3H), 1.24 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-3-fluorophenylsulfonimide (3j)



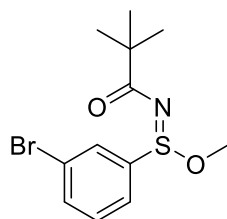
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 34.7 mg, 90% yield.

TLC: R_f = 0.68 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.68 – 7.59 (m, 2H), 7.53 – 7.45 (m, 1H), 7.27 – 7.20 (m, 1H), 3.52 (s, 3H), 1.24 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-3-bromophenylsulfonimide (3k)



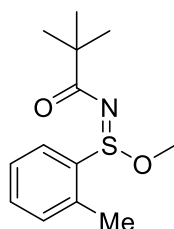
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.8 mg, 77% yield.

TLC: R_f = 0.55 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.72 (m, 2H), 7.66 – 7.61 (m, 2H), 3.51 (s, 3H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-2-methylphenylsulfonimide (3l)



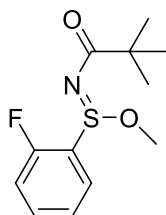
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 34.6 mg, 91% yield.

TLC: R_f = 0.65 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.42 – 8.35 (m, 1H), 7.54 – 7.48 (m, 1H), 7.46 – 7.39 (m, 1H), 7.36 – 7.31 (m, 1H), 3.58 (s, 3H), 2.61 (s, 3H), 1.32 (s, 5H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-2-fluorophenylsulfonimide (3m)



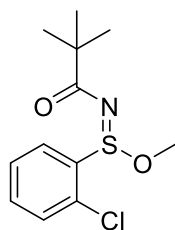
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 35.5 mg, 92% yield.

TLC: R_f = 0.73 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.27 – 8.20 (m, 1H), 7.57 – 7.50 (m, 1H), 7.34 – 7.27 (m, 1H), 7.19 – 7.11 (m, 1H), 3.66 (s, 3H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-2-chlorophenylsulfinimide (3n)



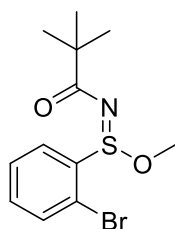
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 35.7 mg, 87% yield.

TLC: R_f = 0.67 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.51 – 8.44 (m, 1H), 7.60 – 7.48 (m, 3H), 3.81 (s, 3H), 1.31 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-2-bromophenylsulfinimide (3o)



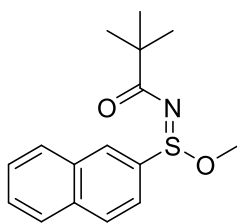
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 43.4 mg, 91% yield.

TLC: R_f = 0.78 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.40 – 8.34 (m, 1H), 7.66 – 7.59 (m, 1H), 7.50 – 7.45 (m, 1H), 7.42 – 7.35 (m, 1H), 3.74 (s, 3H), 1.22 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-2-naphthylsulfinimide (3p)



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 30.4 mg, 70% yield.

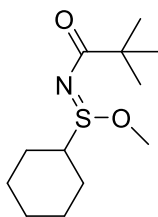
TLC: R_f = 0.65 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.43 – 8.38 (m, 1H), 7.96 – 7.89 (m, 2H), 7.88 – 7.81 (m, 2H), 7.61 – 7.51 (m, 2H), 3.52 (s, 3H), 1.28 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 193.0, 135.2, 133.3, 132.7, 129.7, 129.4, 129.3, 128.9, 128.1, 127.6, 123.1, 52.2, 41.4, 28.4.

HRMS (ESI⁺): m/z calcd for $\text{C}_{16}\text{H}_{19}\text{NO}_2\text{SNa}^+$ [$\text{M}+\text{Na}$]⁺: 312.1029, found: 312.1029.

Methyl *N*-(pivaloyl)-cyclohexylsulfonimide (3q)



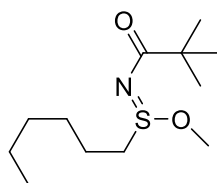
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 25.8 mg, 70% yield.

TLC: R_f = 0.68 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 3.82 (s, 3H), 3.00–2.88 (m, 1H), 2.10–1.95 (m, 2H), 1.88–1.74 (m, 2H), 1.67–1.59 (m, 1H), 1.46–1.21 (m, 5H), 1.15 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-hexylsulfonimide (3r)



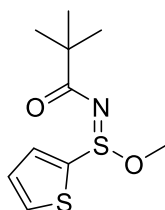
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 26.0 mg, 70% yield.

TLC: R_f = 0.67 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 3.82 (s, 3H), 3.05–2.87 (m, 2H), 1.71–1.59 (m, 2H), 1.41–1.32 (m, 2H), 1.28–1.22 (m, 4H), 1.14 (s, 9H), 0.87–0.79 (m, 4H). Analytical data in agreement with the literature.¹

Methyl *N*-(pivaloyl)-thiophensulfonimide (3s)



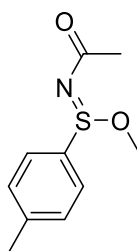
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.1 mg, 98% yield.

TLC: R_f = 0.53 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.70 (m, 1H), 7.65 – 7.59 (m, 1H), 7.27 – 7.19 (m, 1H), 3.55 (s, 3H), 1.30 (s, 9H). Analytical data in agreement with the literature.¹

Methyl *N*-(acetyl)-4-methylphenylsulfonimide (3t)



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

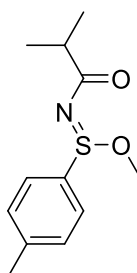
A colorless oil, 28.8 mg, 91% yield.

TLC: R_f = 0.29 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.76–7.69 (m, 2H), 7.33–7.27 (m, 2H), 3.52 (s, 3H), 2.37 (s, 3H), 2.19 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 185.0, 143.9, 132.3, 130.3, 127.9, 52.6, 25.9, 21.7. Analytical data in agreement with the literature. ¹

Methyl *N*-(isobutyryl)-4-methylphenylsulfonimide (3u)



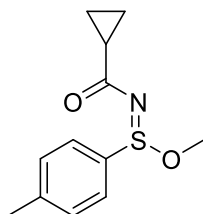
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 28.7 mg, 80% yield.

TLC: R_f = 0.54 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.85–7.80 (m, 2H), 7.41–7.35 (m, 2H), 3.60 (s, 3H), 2.86–2.70 (m, 1H), 2.46 (s, 3H), 1.27 (d, J = 2.8 Hz, 3H), 1.25 (d, J = 2.9 Hz, 3H). Analytical data in agreement with the literature. ¹

Methyl *N*-(cyclopropanecarbonyl)-4-methylphenylsulfonimide (3v)



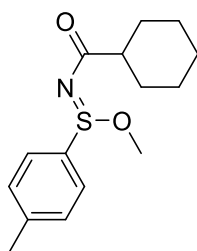
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 28.8 mg, 81% yield.

TLC: R_f = 0.36 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.79 – 7.70 (m, 2H), 7.35 – 7.25 (m, 2H), 3.52 (s, 3H), 2.37 (s, 3H), 1.92 – 1.80 (m, 1H), 1.10 – 0.92 (m, 2H), 0.83 – 0.70 (m, 2H). Analytical data in agreement with the literature. ¹

Methyl N-(cyclohexanecarbonyl)-4-methylphenylsulfinimide (3w)



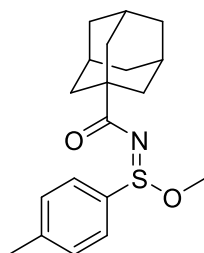
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.0 mg, 86% yield.

TLC: R_f = 0.57 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.78 (m, 2H), 7.41 – 7.35 (m, 2H), 3.58 (s, 3H), 2.54 – 2.46 (m, 1H), 2.45 (s, 3H), 2.09 – 1.98 (m, 2H), 1.85 – 1.75 (m, 2H), 1.72 – 1.65 (m, 1H), 1.61 – 1.48 (m, 2H), 1.40 – 1.20 (m, 4H). Analytical data in agreement with the literature.¹

Methyl N-(adamantane-1-carbonyl)-4-methylphenylsulfinimide (3x)



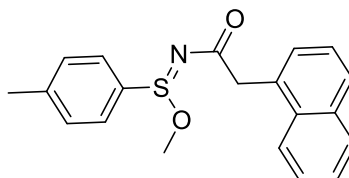
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 43.8 mg, 88% yield.

TLC: R_f = 0.64 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.80 (m, 2H), 7.41 – 7.35 (m, 2H), 3.55 (s, 3H), 2.46 (s, 3H), 2.09 – 2.01 (m, 9H), 1.79 – 1.73 (m, 6H). Analytical data in agreement with the literature.¹

Methyl N-[2-(naphthalen-1-yl)acetyl]-4-methylphenylsulfinimide (3y)



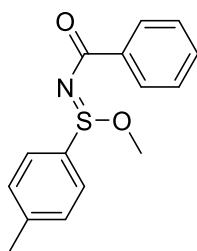
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 50.1 mg, 99% yield.

TLC: R_f = 0.38 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.16 – 8.11 (m, 1H), 7.79 – 7.74 (m, 1H), 7.70 – 7.66 (m, 1H), 7.57 – 7.50 (m, 2H), 7.44 – 7.40 (m, 2H), 7.40 – 7.32 (m, 2H), 7.21 – 7.16 (m, 3H), 4.20 (s, 2H), 3.38 (s, 3H), 2.32 (s, 3H). Analytical data in agreement with the literature.¹

Methyl N-(benzoyl)-4-methylphenylsulfonimide (3z)



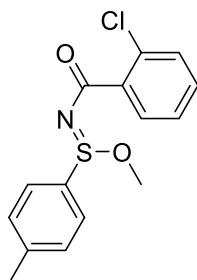
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 32.8 mg, 80% yield.

TLC: R_f = 0.51 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.27 – 8.19 (m, 2H), 7.90 – 7.84 (m, 2H), 7.48 – 7.41 (m, 1H), 7.40 – 7.32 (m, 4H), 3.56 (s, 3H), 2.40 (s, 3H). Analytical data in agreement with the literature.¹

Methyl N-(2-chlorobenzoyl)-4-methylphenylsulfonimide (3a')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 40.2 mg, 87% yield.

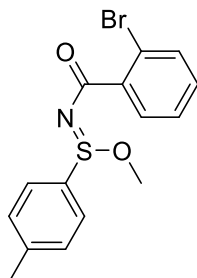
TLC: R_f = 0.43 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.96 – 7.86 (m, 3H), 7.47 – 7.43 (m, 1H), 7.42 – 7.39 (m, 2H), 7.38 – 7.29 (m, 2H), 3.68 (s, 3H), 2.47 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 178.9, 144.2, 136.9, 132.4, 131.9, 130.9, 130.9, 130.6, 130.3, 128.2, 126.5, 52.6, 21.7.

HRMS (ESI⁺): m/z calcd for $\text{C}_{15}\text{H}_{14}\text{ClINO}_2\text{SNa}^+$ $[\text{M}+\text{Na}]^+$: 330.0326, found: 330.0326.

Methyl N-(2-bromobenzoyl)-4-methylphenylsulfonimide (3b')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 46.5 mg, 88% yield.

TLC: R_f = 0.37 (PE/EA = 3:1).

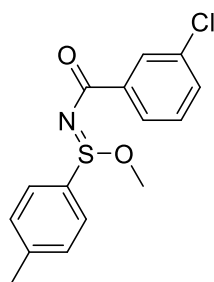
¹H NMR (400 MHz, CDCl₃) δ 7.96 – 7.89 (m, 2H), 7.88 – 7.82 (m, 1H), 7.67 – 7.62 (m, 1H), 7.44 – 7.39 (m, 2H), 7.39 – 7.33 (m, 1H), 7.28 – 7.22 (m, 1H), 3.69 (s, 3H), 2.47 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 179.5, 144.2, 139.0, 133.8, 131.8, 131.0, 130.7, 130.3, 128.2, 127.1, 120.6, 52.7, 21.7.

HRMS (ESI+): m/z calcd for C₁₅H₁₄⁸¹BrNO₂SNa⁺ [M+Na]⁺: 375.9800, found: 375.9801.

HRMS (ESI+): m/z calcd for C₁₅H₁₄⁷⁹BrNO₂SNa⁺ [M+Na]⁺: 373.9821, found: 373.9823.

Methyl N-(3-chlorobenzoyl)-4-methylphenylsulfonimide (3c')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 43.9 mg, 95% yield.

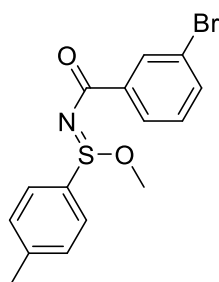
TLC: *R*_f = 0.55 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 8.29 (t, *J* = 1.8 Hz, 1H), 8.20 – 8.15 (m, 1H), 7.99 – 7.89 (m, 2H), 7.52 – 7.48 (m, 1H), 7.47 – 7.42 (m, 2H), 7.42 – 7.36 (m, 1H), 3.65 (s, 3H), 2.49 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 177.1, 144.3, 137.8, 134.1, 132.2, 131.8, 130.4, 129.7, 129.4, 128.1, 127.7, 52.5, 21.7.

HRMS (ESI+): m/z calcd for C₁₅H₁₄ClNO₂SNa⁺ [M+Na]⁺: 330.0326, found: 330.0327.

Methyl N-(3-bromobenzoyl)-4-methylphenylsulfonimide (3d')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 42.8 mg, 81% yield.

TLC: *R*_f = 0.54 (PE/EA = 3:1).

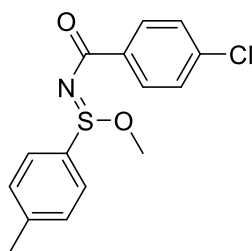
¹H NMR (400 MHz, CDCl₃) δ 8.48 – 8.41 (m, 1H), 8.27 – 8.18 (m, 1H), 7.99 – 7.89 (m, 2H), 7.69 – 7.61 (m, 1H), 7.49 – 7.41 (m, 2H), 7.38 – 7.30 (m, 1H), 3.65 (s, 3H), 2.49 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 176.9, 144.3, 138.0, 134.7, 132.6, 132.2, 130.4, 129.7, 128.1, 128.1, 122.2, 52.5, 21.7.

HRMS (ESI+): m/z calcd for C₁₅H₁₄⁸¹BrNO₂SNa⁺ [M+Na]⁺: 375.9800, found: 375.9800.

HRMS (ESI+): m/z calcd for C₁₅H₁₄⁷⁹BrNO₂SNa⁺ [M+Na]⁺: 373.9821, found: 373.9822.

Methyl N-(4-chlorobenzoyl)-4-methylphenylsulfinimide (3e')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.9 mg, 80% yield.

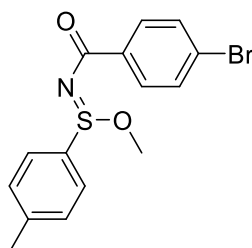
TLC: R_f = 0.50 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 8.34 – 8.16 (m, 2H), 7.99 – 7.87 (m, 2H), 7.47 – 7.38 (m, 4H), 3.65 (s, 3H), 2.49 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 177.4, 144.2, 138.1, 134.4, 132.4, 131.0, 130.3, 128.3, 128.0, 52.5, 21.7.

HRMS (ESI⁺): m/z calcd for C₁₅H₁₄ClNO₂SNa⁺ [M+Na]⁺: 330.0326, found: 330.0326.

Methyl N-(4-bromobenzoyl)-4-methylphenylsulfinimide (3f')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 47.6 mg, 90% yield.

TLC: R_f = 0.41 (PE/EA = 3:1).

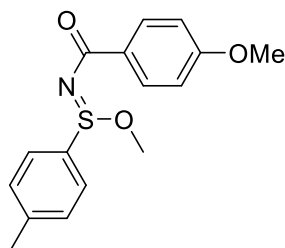
¹H NMR (400 MHz, CDCl₃) δ 8.12 – 8.05 (m, 1H), 7.88 – 7.81 (m, 1H), 7.52 – 7.47 (m, 1H), 7.39 – 7.31 (m, 3H), 3.56 (s, 3H), 2.40 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 177.6, 144.2, 134.9, 132.4, 131.3, 131.2, 130.3, 128.1, 126.8, 52.5, 21.7.

HRMS (ESI⁺): m/z calcd for C₁₅H₁₄⁸¹BrNO₂SNa⁺ [M+Na]⁺: 375.9800, found: 375.9800.

HRMS (ESI⁺): m/z calcd for C₁₅H₁₄⁷⁹BrNO₂SNa⁺ [M+Na]⁺: 373.9821, found: 373.9821.

Methyl N-(4-methoxybenzoyl)-4-methylphenylsulfinimide (3g')



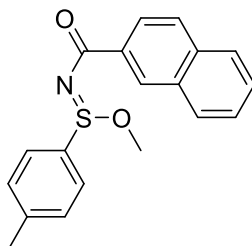
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 45.0 mg, 99% yield.

TLC: R_f = 0.39 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.32 – 8.25 (m, 2H), 7.98 – 7.91 (m, 2H), 7.45 – 7.39 (m, 2H), 6.99 – 6.90 (m, 2H), 3.88 (s, 3H), 3.64 (s, 3H), 2.48 (s, 3H). Analytical data in agreement with the literature.¹

Methyl *N*-(2-Naphthoyl)-4-methylphenylsulfonimide (3h')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 37.4 mg, 77% yield.

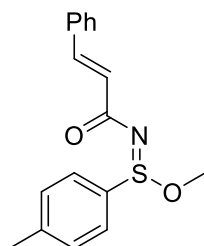
TLC: R_f = 0.38 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 8.83 – 8.73 (m, 1H), 8.31 – 8.24 (m, 1H), 7.95 – 7.87 (m, 3H), 7.85 – 7.76 (m, 2H), 7.50 – 7.40 (m, 2H), 7.38 – 7.31 (m, 2H), 3.60 (s, 3H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 178.6, 144.0, 135.3, 133.3, 132.8, 132.7, 130.5, 130.3, 129.5, 128.1, 127.8, 127.7, 127.6, 126.3, 126.1, 52.5, 21.7.

HRMS (ESI⁺): m/z calcd for $\text{C}_{19}\text{H}_{17}\text{NO}_2\text{SNa}^+ [\text{M}+\text{Na}]^+$: 346.0872, found: 346.0872.

Methyl *N*-(cinnamoyl)-4-methylphenylsulfonimide (3i')



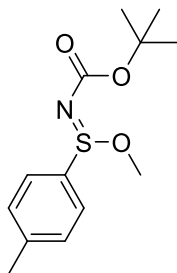
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 33.7 mg, 75% yield.

TLC: R_f = 0.52 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.87 (m, 2H), 7.81 (d, J = 15.9 Hz, 1H), 7.64 – 7.53 (m, 2H), 7.45 – 7.31 (m, 5H), 6.85 (d, J = 15.9 Hz, 1H), 3.67 (s, 3H), 2.48 (s, 3H). Analytical data in agreement with the literature.¹

Methyl *N*-(*tert*-butoxycarbonyl)-4-methylphenylsulfonimide (3j')



This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 36.4 mg, 90% yield.

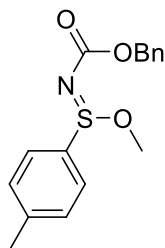
TLC: R_f = 0.63 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7.78 (m, 2H), 7.39 – 7.32 (m, 2H), 3.51 (s, 3H), 2.44 (s, 3H), 1.55 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 164.0, 143.8, 132.3, 130.1, 128.0, 80.4, 50.8, 28.4, 21.7.

HRMS (ESI⁺): m/z calcd for C₁₃H₁₉NO₃SN⁺ [M+Na]⁺: 292.0978, found: 292.0978.

Methyl *N*-[(benzyloxy)carbonyl]-4-methylphenylsulfonimide (3k')



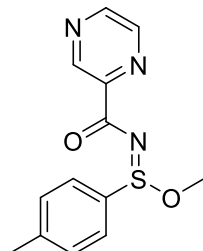
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 44.6 mg, 98% yield.

TLC: R_f = 0.50 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.44 (m, 2H), 7.40 – 7.34 (m, 5H), 7.34 – 7.31 (m, 1H), 5.26 (d, J = 12.4 Hz, 1H), 5.24 (d, J = 12.4 Hz, 1H), 3.51 (s, 3H), 2.45 (s, 3H). Analytical data in agreement with the literature.¹

Methyl *N*-(pyrazine-2-carbonyl)-4-methylphenylsulfonimide (3l')



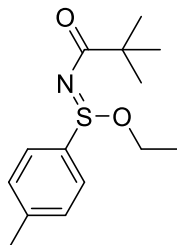
This compound was synthesized following *General procedure 1*, in which MeOH served as both the solvent and the alcohol reactant.

A colorless oil, 40.9 mg, 99% yield.

TLC: R_f = 0.59 (DCM/MeOH = 20:1).

¹H NMR (400 MHz, CDCl₃) δ 9.61 – 9.49 (m, 1H), 8.78 – 8.67 (m, 2H), 8.02 – 7.89 (m, 2H), 7.49 – 7.38 (m, 2H), 3.72 (s, 3H), 2.48 (s, 3H). Analytical data in agreement with the literature.¹

Ethyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3m')



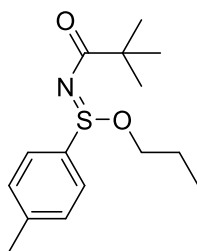
This compound was synthesized following *General procedure 1*, in which EtOH served as both the solvent and the alcohol reactant.

A colorless oil, 32.5 mg, 81% yield.

TLC: *R*_f = 0.33 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.79 – 7.71 (m, 2H), 7.34 – 7.21 (m, 2H), 4.28 – 4.06 (m, 1H), 3.96 – 3.70 (m, 1H), 2.37 (s, 3H), 1.22 (s, 9H). Analytical data in agreement with the literature.¹

Propyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3n')



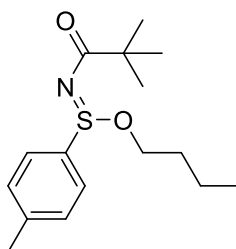
This compound was synthesized following *General procedure 1*, in which *n*-PrOH served as both the solvent and the alcohol reactant.

A colorless oil, 38.0 mg, 90% yield.

TLC: *R*_f = 0.87 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.90 – 7.77 (m, 2H), 7.46 – 7.31 (m, 2H), 4.13 (dt, *J* = 9.9, 6.6 Hz, 1H), 3.79 (dt, *J* = 9.9, 6.6 Hz, 1H), 2.45 (s, 3H), 1.71 – 1.55 (m, 2H), 1.31 (s, 9H), 0.89 (t, *J* = 7.4 Hz, 3H). Analytical data in agreement with the literature.¹

Butyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3o')



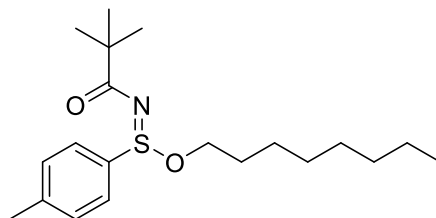
This compound was synthesized following *General procedure 1*, in which *n*-BuOH served as both the solvent and the alcohol reactant.

A colorless oil, 39.4 mg, 89% yield.

TLC: R_f = 0.83 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.80 (m, 2H), 7.39 – 7.34 (m, 2H), 4.17 (dt, J = 9.9, 6.6 Hz, 1H), 3.82 (dt, J = 9.9, 6.6 Hz, 1H), 2.46 (s, 3H), 1.66 – 1.56 (m, 2H), 1.31 (s, 9H), 0.88 (t, J = 7.4 Hz, 3H). Analytical data in agreement with the literature.¹

Octyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3p')



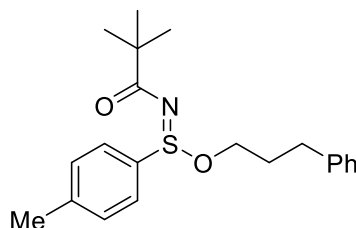
This compound was synthesized following *General procedure 2*, with 2.0 equiv of 1-octanol as the alcohol substrate.

A colorless oil, 36.9 mg, 70% yield.

TLC: R_f = 0.85 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.81 – 7.69 (m, 2H), 7.33 – 7.22 (m, 2H), 4.07 (s, 1H), 3.77 – 3.68 (m, 1H), 2.37 (s, 3H), 1.58 – 1.48 (m, 2H), 1.22 (s, 9H), 1.21 – 1.04 (m, 10H), 0.79 (t, J = 6.8 Hz, 3H). Analytical data in agreement with the literature.¹

3-Phenylpropyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3q')



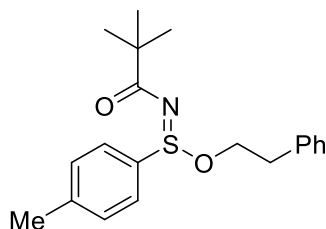
This compound was synthesized following *General procedure 2*, with 10.0 equiv of 3-phenyl-1-propanol as the alcohol substrate.

A colorless oil, 40.8 mg, 76% yield.

TLC: R_f = 0.76 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.71 (m, 2H), 7.31 – 7.25 (m, 2H), 7.19 – 7.15 (m, 2H), 7.13 – 7.06 (m, 1H), 7.06 – 7.01 (m, 2H), 4.13 – 4.04 (m, 1H), 3.76 (dt, J = 10.0, 6.3 Hz, 1H), 2.58 – 2.52 (m, 2H), 2.37 (s, 3H), 1.90 – 1.81 (m, 2H), 1.22 (s, 9H). Analytical data in agreement with the literature.¹

Phenethyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3r')



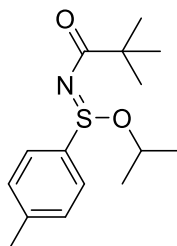
This compound was synthesized following *General procedure 2*, with 10.0 equiv of phenethyl alcohol as the alcohol substrate.

A colorless oil, 42.8 mg, 83% yield.

TLC: R_f = 0.80 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.59 – 7.52 (m, 2H), 7.22 – 7.14 (m, 6H), 7.10 – 7.04 (m, 2H), 4.34 – 4.26 (m, 1H), 4.00 – 3.92 (m, 1H), 2.84 (t, J = 7.0 Hz, 2H), 2.34 (s, 3H), 1.22 (s, 9H). Analytical data in agreement with the literature.¹

Isopropyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3s')



This compound was synthesized following *General procedure 2*, with 10.0 equiv of iPrOH as the alcohol substrate.

A colorless oil, 33.3 mg, 79% yield.

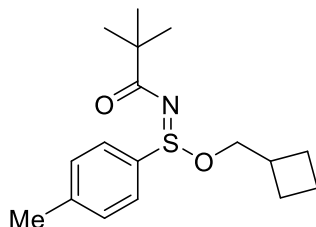
TLC: R_f = 0.83 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.79 (m, 2H), 7.44 – 7.33 (m, 2H), 5.05 – 4.85 (m, 1H), 2.45 (s, 2H), 1.41 (d, J = 6.3 Hz, 3H), 1.30 (s, 9H), 1.22 (d, J = 6.3 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 193.1, 143.1, 134.8, 130.0, 127.7, 75.3, 41.0, 28.4, 23.8, 23.5, 21.6.

Analytical data in agreement with the literature.¹

Cyclobutylmethyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3t')



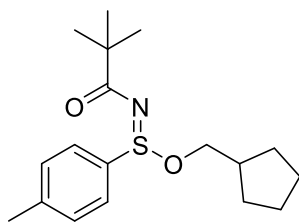
This compound was synthesized following *General procedure 2*, with 10.0 equiv of cyclobutanemethanol as the alcohol substrate.

A colorless oil, 25.4 mg, 55% yield.

TLC: R_f = 0.56 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.76 – 7.70 (m, 2H), 7.31 – 7.24 (m, 2H), 4.10 – 4.04 (m, 1H), 3.72 – 3.65 (m, 1H), 2.56 – 2.46 (m, 1H), 2.37 (s, 3H), 2.00 – 1.87 (m, 2H), 1.85 – 1.71 (m, 2H), 1.69 – 1.56 (m, 2H), 1.23 (s, 9H). Analytical data in agreement with the literature.¹

Cyclopentylmethyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3u')



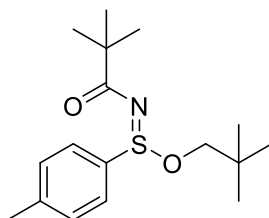
This compound was synthesized following *General procedure 2*, with 10.0 equiv of cyclopentanemethanol as the alcohol substrate.

A colorless oil, 38.1 mg, 79% yield.

TLC: R_f = 0.37 (PE/EA = 10:1).

^1H NMR (400 MHz, CDCl_3) δ 7.84 – 7.77 (m, 2H), 7.38 – 7.31 (m, 2H), 4.04 (dd, J = 9.7, 7.2 Hz, 1H), 3.64 (dd, J = 9.7, 7.1 Hz, 1H), 2.43 (s, 3H), 2.24 – 2.09 (m, 1H), 1.79 – 1.62 (m, 2H), 1.59 – 1.44 (m, 4H), 1.29 (s, 9H), 1.25 – 1.08 (m, 2H). Analytical data in agreement with the literature.¹

Neopentyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3v')



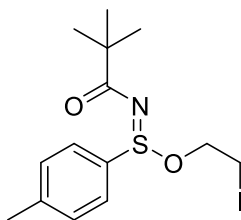
This compound was synthesized following *General procedure 2*, with 10.0 equiv of neopentyl alcohol as the alcohol substrate.

A colorless oil, 42.2 mg, 91% yield.

TLC: R_f = 0.86 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.92 – 7.74 (m, 2H), 7.43 – 7.28 (m, 2H), 3.81 (d, J = 9.4 Hz, 1H), 3.37 (d, J = 9.4 Hz, 1H), 2.43 (s, 3H), 1.28 (s, 9H), 0.86 (s, 9H). Analytical data in agreement with the literature.¹

2-Iodoethyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3w')



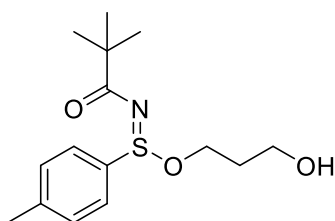
This compound was synthesized following *General procedure 2*, with 10.0 equiv of 2-iodoethanol as the alcohol substrate.

A colorless oil, 23.0 mg, 39% yield.

TLC: R_f = 0.74 (PE/EA = 3:1).

^1H NMR (400 MHz, CDCl_3) δ 7.92 – 7.83 (m, 2H), 7.40 – 7.32 (m, 2H), 4.44 – 4.34 (m, 1H), 4.14 – 4.04 (m, 1H), 3.33 – 3.19 (m, 2H), 2.45 (s, 3H), 1.29 (s, 9H). Analytical data in agreement with the literature.¹

3-Hydroxypropyl *N*-(pivaloyl)-4-methylphenylsulfonimide (3x')



This compound was synthesized following *General procedure 2*.

When the reaction was conducted on a 0.10 mmol scale of sulfenamide using 2.5 equiv each of alcohol, NBS, and NaHCO₃, the product was obtained as a colorless oil (10.4 mg, 35% yield).

Under modified conditions with 2.0 equiv of alcohol, 1.2 equiv of NBS, and 1.5 equiv of NaHCO₃, the product was isolated as a colorless oil (19.0 mg, 64% yield).

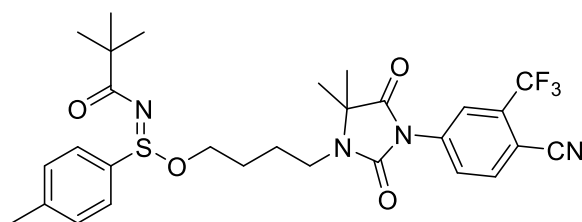
TLC: *R*_f = 0.34 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 7.79 – 7.72 (m, 2H), 7.33 – 7.26 (m, 2H), 4.23 – 4.13 (m, 1H), 4.12 – 4.04 (m, 1H), 3.66 – 3.50 (m, 2H), 2.37 (s, 3H), 1.83 – 1.66 (m, 3H), 1.23 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 194.1, 143.7, 133.8, 130.2, 127.7, 64.9, 58.3, 41.2, 32.6, 28.4, 21.7.

Analytical data in agreement with the literature.¹

4-(3-(4-Cyano-3-(trifluoromethyl)phenyl)-5,5-dimethyl-2,4-dioxoimidazolidin-1-yl)butyl *N*-(pivaloyl)-4-methylphenylsulfonimide (4)



Following *General procedure 2*, the reaction was carried out on a 0.1 mmol scale with RU58841 (1.2 equiv) as the alcohol substrate.

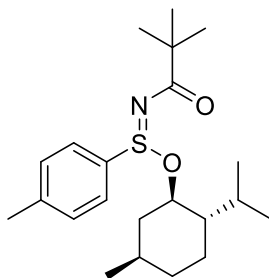
A colorless oil, 31.2 mg, 53% yield.

TLC: *R*_f = 0.41 (PE/EA = 3:1).

¹H NMR (400 MHz, CDCl₃) δ 8.17 – 8.10 (m, 1H), 8.01 – 7.96 (m, 1H), 7.92 – 7.88 (m, 1H), 7.84 – 7.78 (m, 2H), 7.38 – 7.31 (m, 2H), 4.18 – 4.09 (m, 1H), 3.86 – 3.74 (m, 1H), 3.39 – 3.30 (m, 2H), 2.43 (s, 3H), 1.51 (d, *J* = 5.5 Hz, 6H), 1.27 (s, 9H).

¹⁹F NMR (376 MHz, CDCl₃) δ -62.0. Analytical data in agreement with the literature.¹

(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl *N*-(pivaloyl)-4-methylphenylsulfonimide (5)



Following *General procedure 2*, the reaction was carried out on a 1.34 mmol scale with L-menthol (1.0 equiv) as the alcohol substrate in DCM (5.0 mL).

A colorless oil, 337.0 mg, 67% yield.

TLC: R_f = 0.61 (PE/EA = 10:1).

Major diastereomer (5-1)

^1H NMR (400 MHz, CDCl_3) δ 7.80 – 7.75 (m, 2H), 7.35 – 7.30 (m, 2H), 4.63 – 4.54 (m, 1H), 2.52 – 2.45 (m, 1H), 2.42 (s, 3H), 2.09 – 1.97 (m, 1H), 1.71 – 1.51 (m, 4H), 1.27 (s, 9H), 1.23 – 1.14 (m, 1H), 1.03 – 1.00 (m, 1H), 0.95 (d, J = 6.5 Hz, 3H), 0.91 – 0.83 (m, 1H), 0.81 (d, J = 7.1 Hz, 3H), 0.69 (d, J = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 193.2, 142.9, 135.1, 130.0, 127.8, 82.5, 48.4, 43.0, 40.9, 34.2, 31.7, 25.5, 23.4, 22.3, 21.6, 21.0, 16.1.

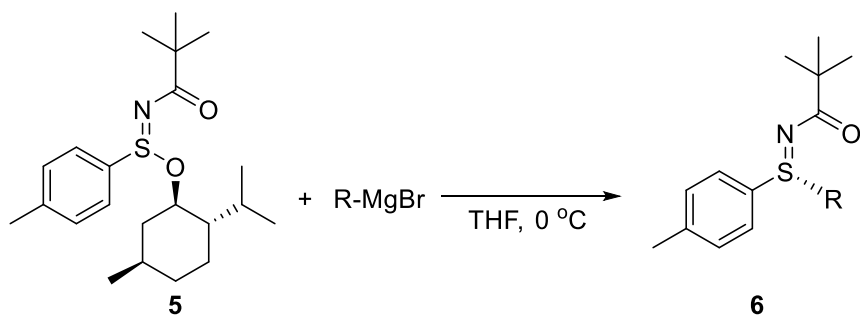
HRMS (ESI $^+$): m/z calcd for $\text{C}_{22}\text{H}_{35}\text{NO}_2\text{SNa}^+$ $[\text{M}+\text{Na}]^+$: 400.2281, found: 400.2281.

Minor diastereomer (5-2)

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.77 (m, 2H), 7.37 – 7.30 (m, 2H), 4.57 – 4.47 (m, 1H), 2.43 (s, 3H), 2.09 – 1.98 (m, 2H), 1.70 – 1.60 (m, 4H), 1.57 – 1.44 (m, 1H), 1.27 (s, 9H), 1.13 – 1.02 (m, 2H), 0.92 – 0.81 (m, 9H).

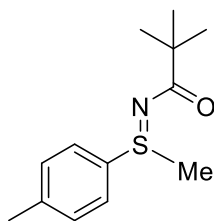
^{13}C NMR (101 MHz, CDCl_3) δ 192.9, 143.2, 135.6, 130.0, 127.4, 84.0, 48.6, 42.9, 41.0, 34.0, 32.0, 28.3, 25.7, 23.1, 22.2, 21.7, 21.2, 15.9.

HRMS (ESI $^+$): m/z calcd for $\text{C}_{22}\text{H}_{35}\text{NO}_2\text{SNa}^+$ $[\text{M}+\text{Na}]^+$: 400.2281, found: 400.2280.



To a flame-dried round-bottomed flask under argon atmosphere were added sulfilimide ester **5** (0.1 mmol, 1.0 equiv) and anhydrous THF (sufficient to achieve 0.02 M concentration). The mixture was cooled to 0 °C, and a solution of the corresponding R-MgBr (0.2 mmol, 2.0 equiv) was added dropwise under argon. The reaction was stirred at 0 °C overnight. Upon completion, the reaction was quenched with saturated aqueous NH_4Cl , extracted with EtOAc, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography to afford compound **6**.

***N*-(Methyl(*p*-tolyl)- λ^4 -sulfanylidene)pivalamide (6a)**



A colorless oil, 20.9 mg, 88% yield.

TLC: R_f = 0.41 (DCM/MeOH = 20:1).

^1H NMR (400 MHz, CDCl_3) δ 7.58 – 7.52 (m, 2H), 7.27 – 7.22 (m, 2H), 2.68 (s, 3H), 2.33 (s, 3H), 1.16 (s, 9H). Analytical data in agreement with the literature.²

Chiral HPLC chromatogram of *rac*-**6a**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C):

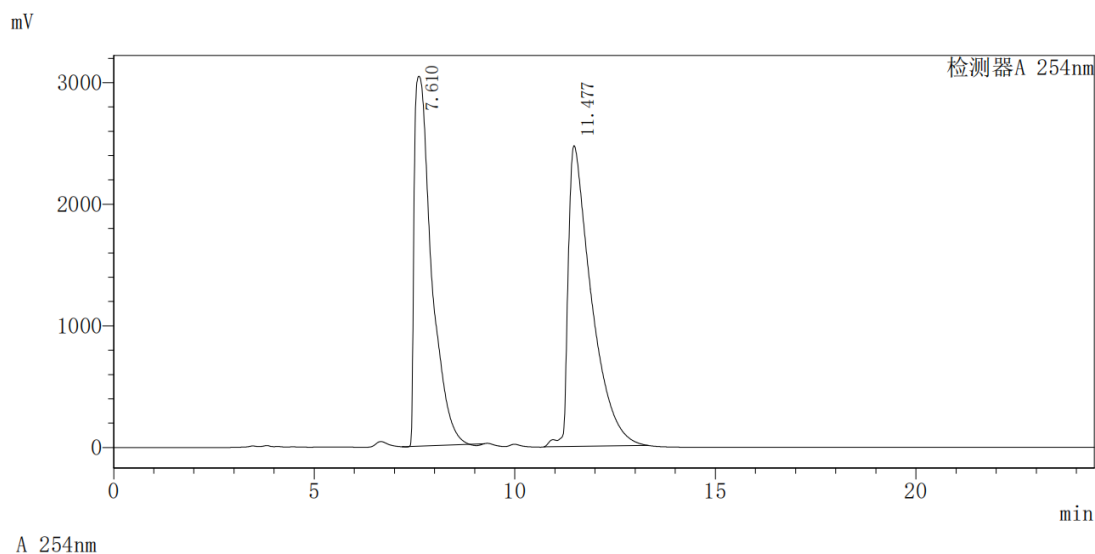


Figure S1: HPLC chromatogram of racemic compound 6a

Chiral HPLC chromatogram of **6a-1** derived from **major diastereomer 5-1**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C).

The enantiomeric excess (ee) of compound **6a-1** is 92%.

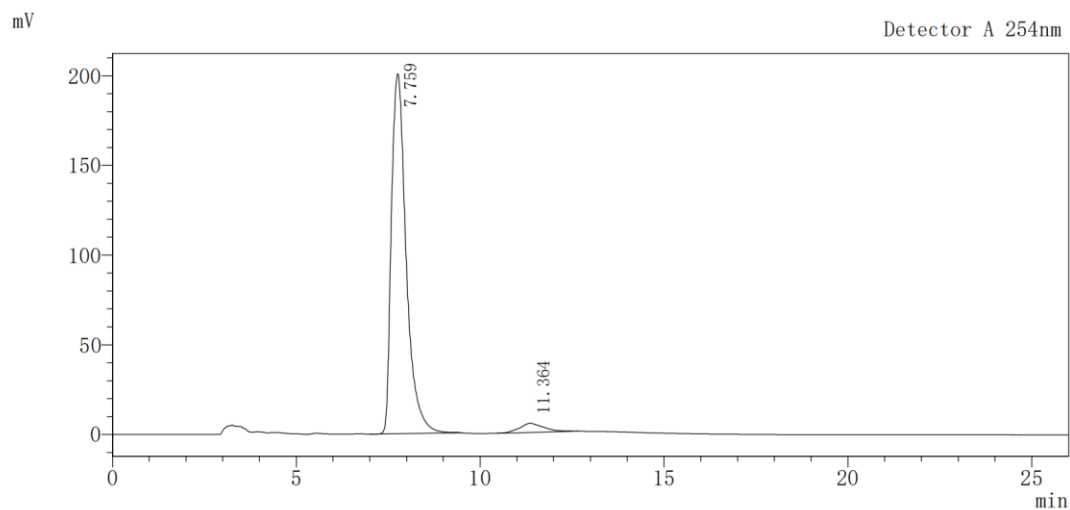


Figure S2: HPLC chromatogram of compound 6a-1

Chiral HPLC chromatogram of **6a-2** derived from **minor diastereomer 5-2**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C).

The enantiomeric excess (ee) of compound **6a-2** is 66%.

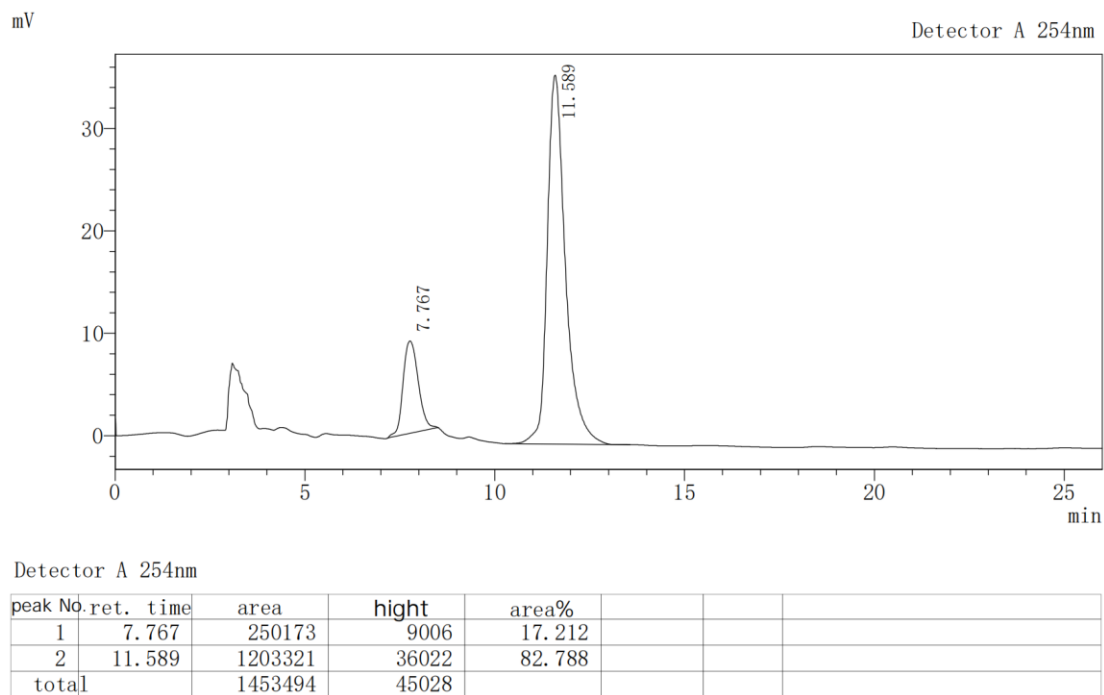
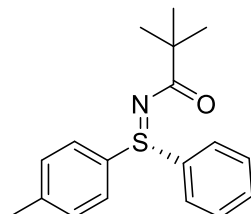


Figure S3: HPLC chromatogram of compound 6a-2

(*R*)-*N*-(Phenyl(*p*-tolyl)- λ^4 -sulfanylidene)pivalamide (6b)



A colorless oil, 25.5 mg, 85% yield.

TLC: R_f = 0.75 (DCM/MeOH = 20:1).

^1H NMR (400 MHz, CDCl_3) δ 7.67 – 7.60 (m, 2H), 7.58 – 7.50 (m, 2H), 7.42 – 7.31 (m, 3H), 7.23 – 7.11 (m, 2H), 2.28 (s, 3H), 1.21 (s, 9H). Analytical data in agreement with the literature.³

Chiral HPLC chromatogram of *rac*-**6b**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C).

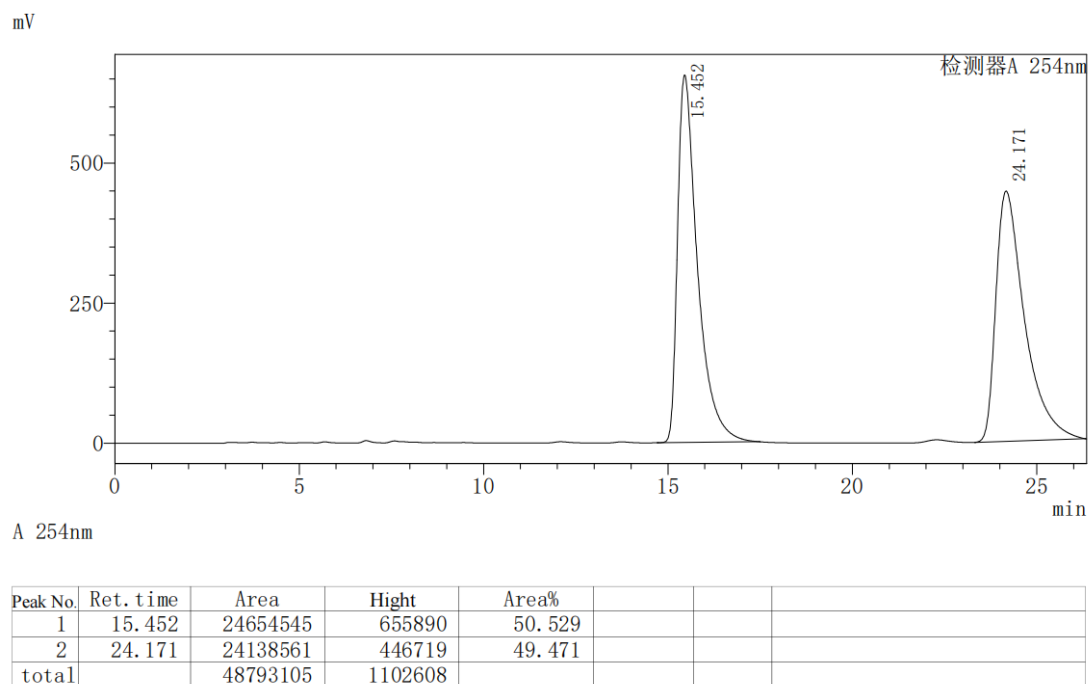


Figure S4: HPLC chromatogram of racemic compound 6b

Chiral HPLC chromatogram of **6b-1** derived from **Major diastereomer 5-1**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C).

The enantiomeric excess (ee) of compound **6b-1** is 93%.

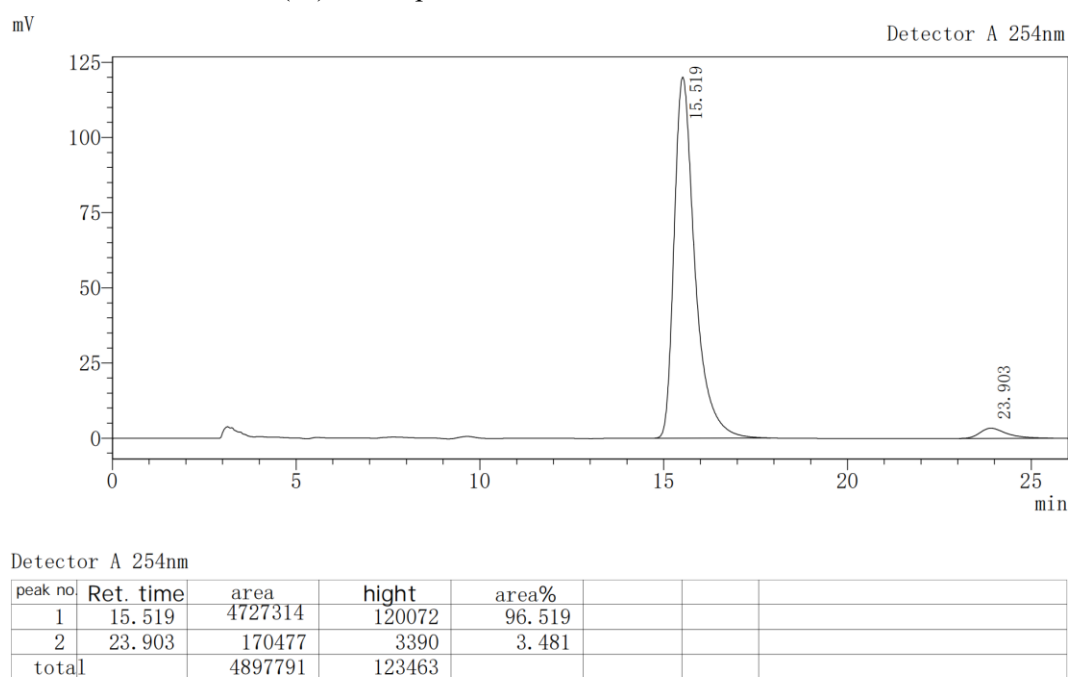


Figure S5: HPLC chromatogram of compound 6b-1

Chiral HPLC chromatogram of **6b-2** derived from **Minor diastereomer 5-2**: HPLC using a Chiralpak IA column (*n*-hexane/*i*PrOH 90:10, flow rate = 1.0 mL/min, λ = 254 nm, 25 °C).

The enantiomeric excess (ee) of compound **6b-2** is 65%.

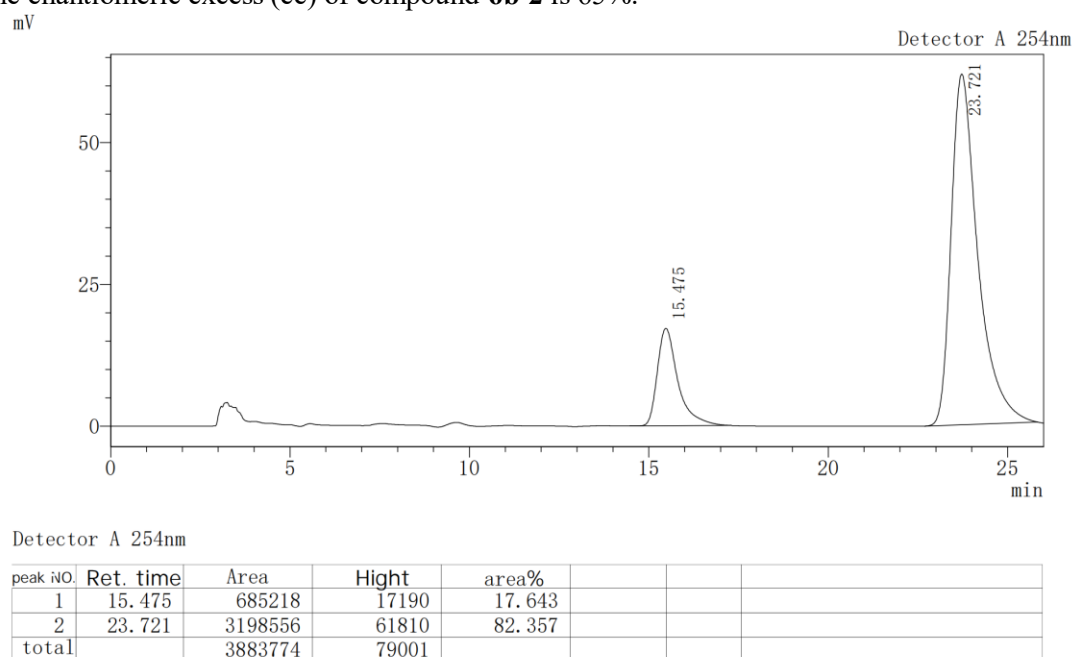


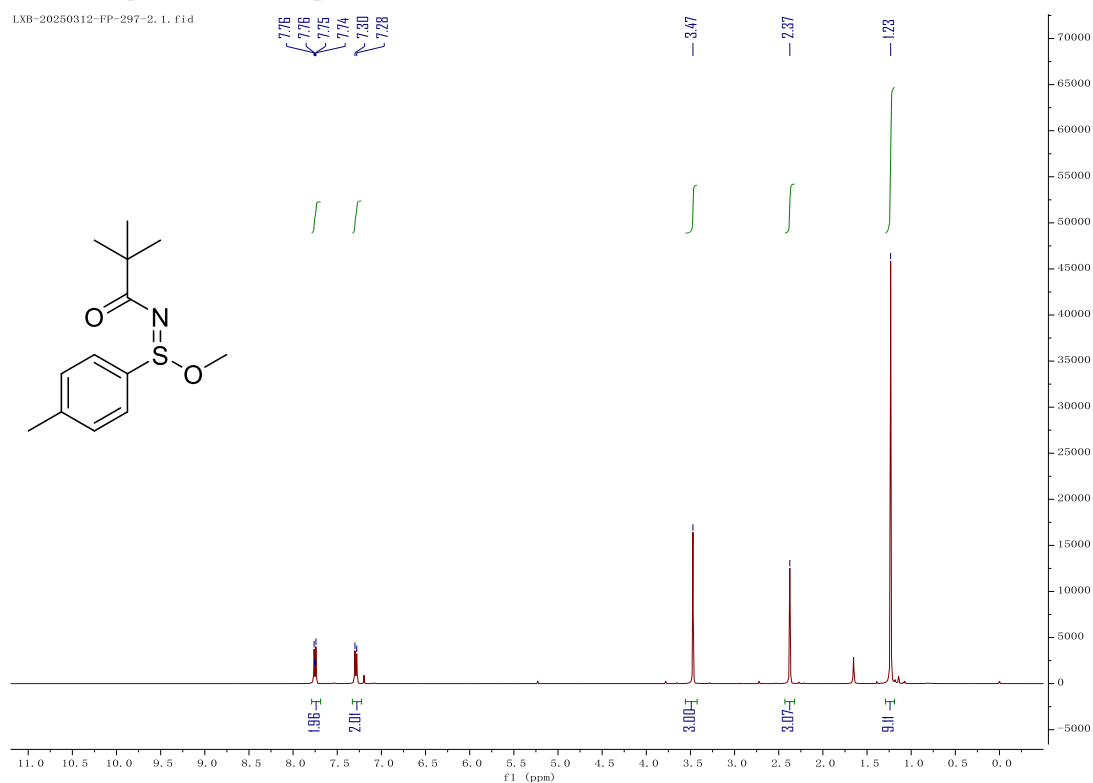
Figure S6: HPLC chromatogram of compound 6b-2

(F) References

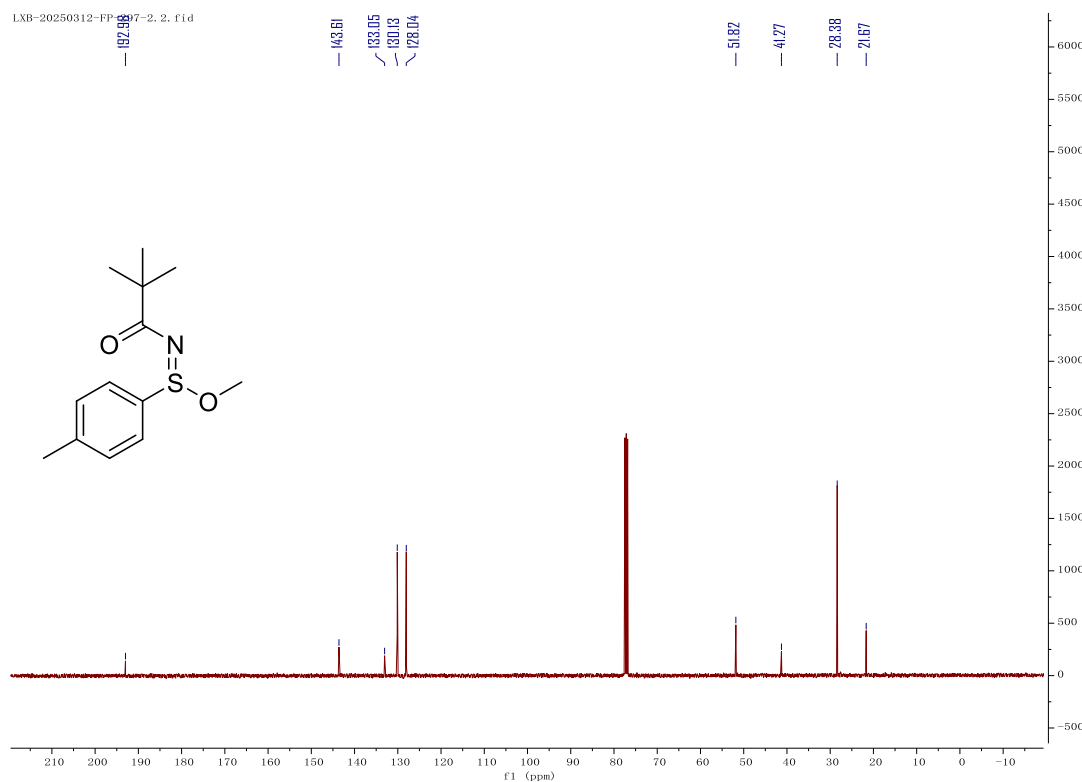
1. X. Lu, G. Huang, J. Ye, M. A. Bashir, J. Su, K. Yang, F. Liang, X. Xu, *Org. Lett.* **2023**, 25, 2151.
2. G. Huang, X. Lu, K. Yang, X. Xu, *Org. Lett.* **2023**, 25, 3173.
3. G. Huang, X. Lu, F. Liang, *Org. Lett.* **2023**, 25, 3179.

(G) Copies of NMR spectra (^1H , ^{13}C , ^{19}F)

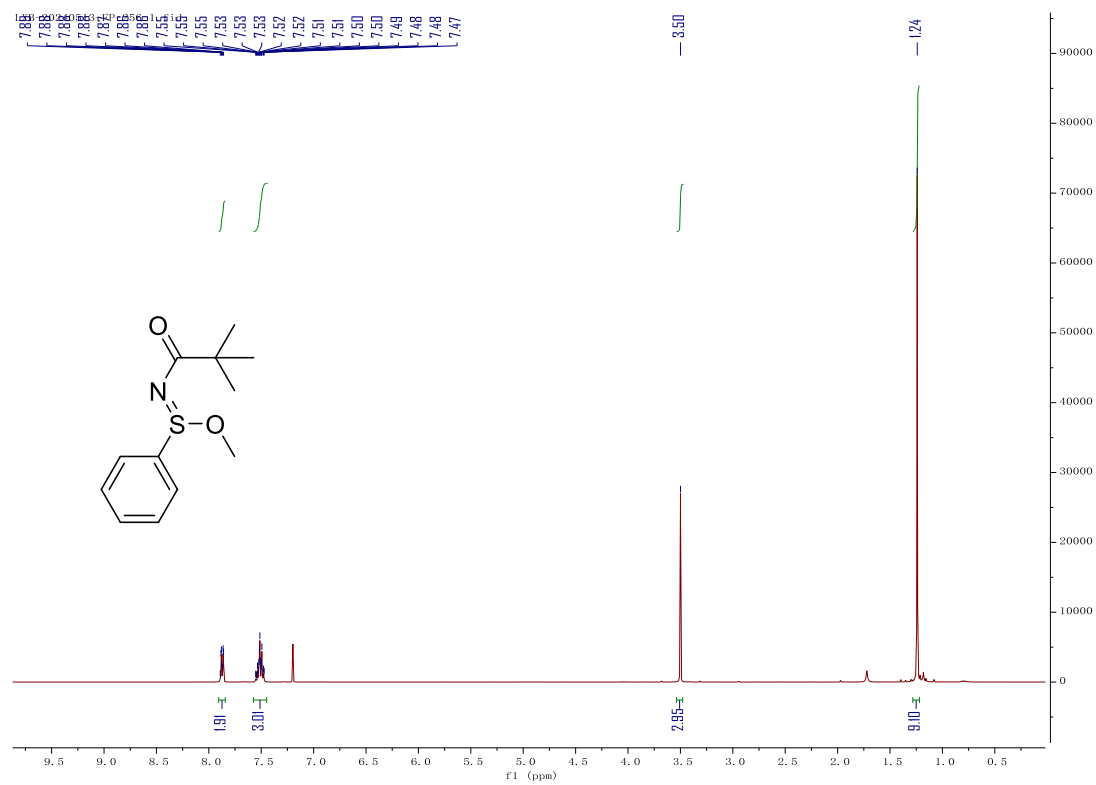
^1H NMR spectrum for compound **3a** (in CDCl_3 , 400 MHz)



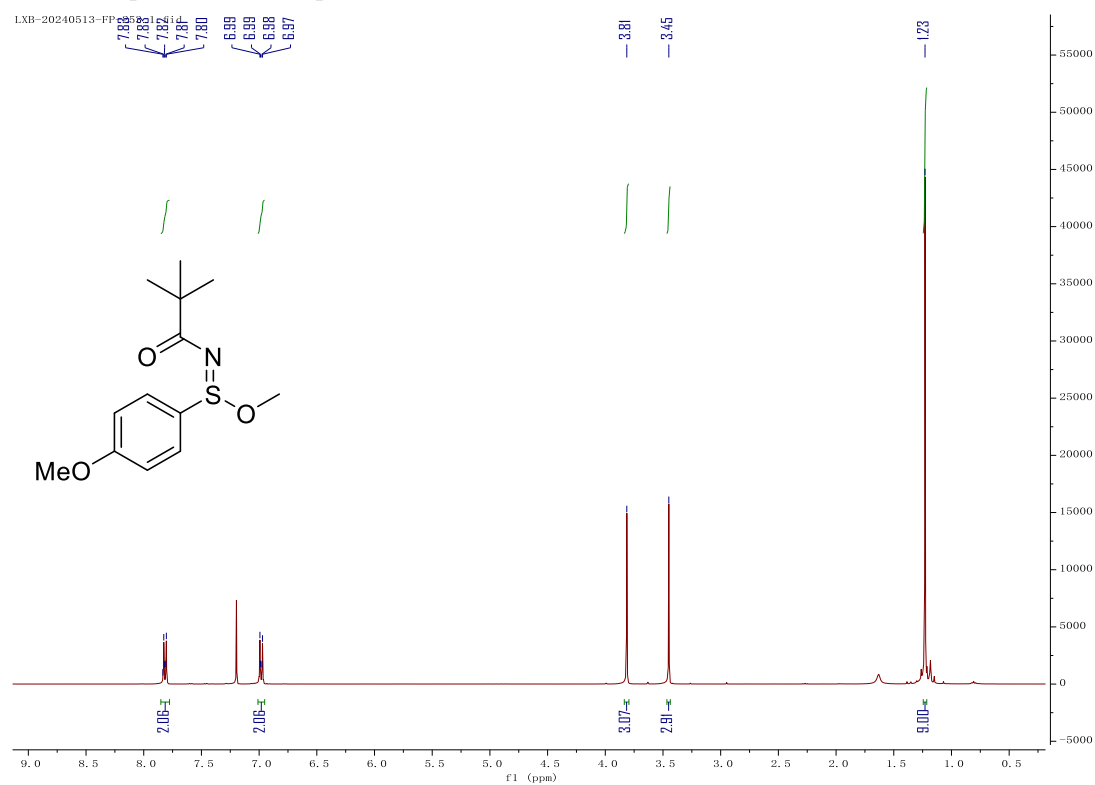
^{13}C NMR spectrum for compound **3a** (in CDCl_3 , 101 MHz)



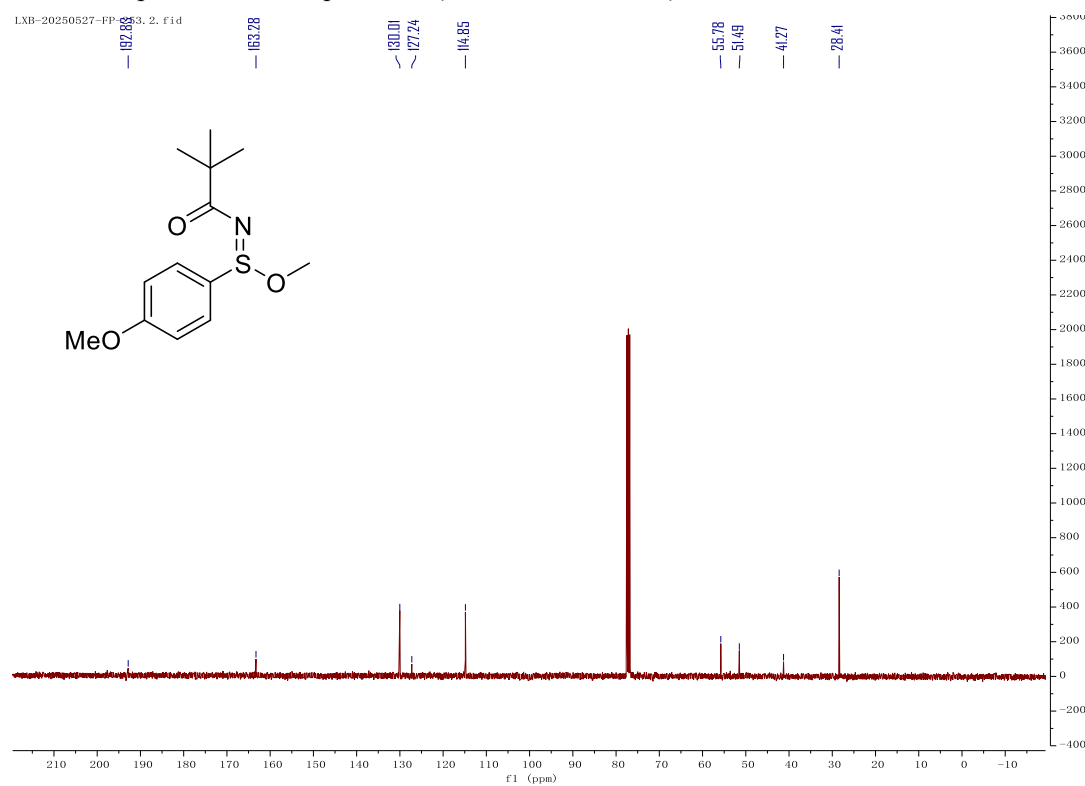
¹H NMR spectrum for compound **3b** (in CDCl₃, 400 MHz)



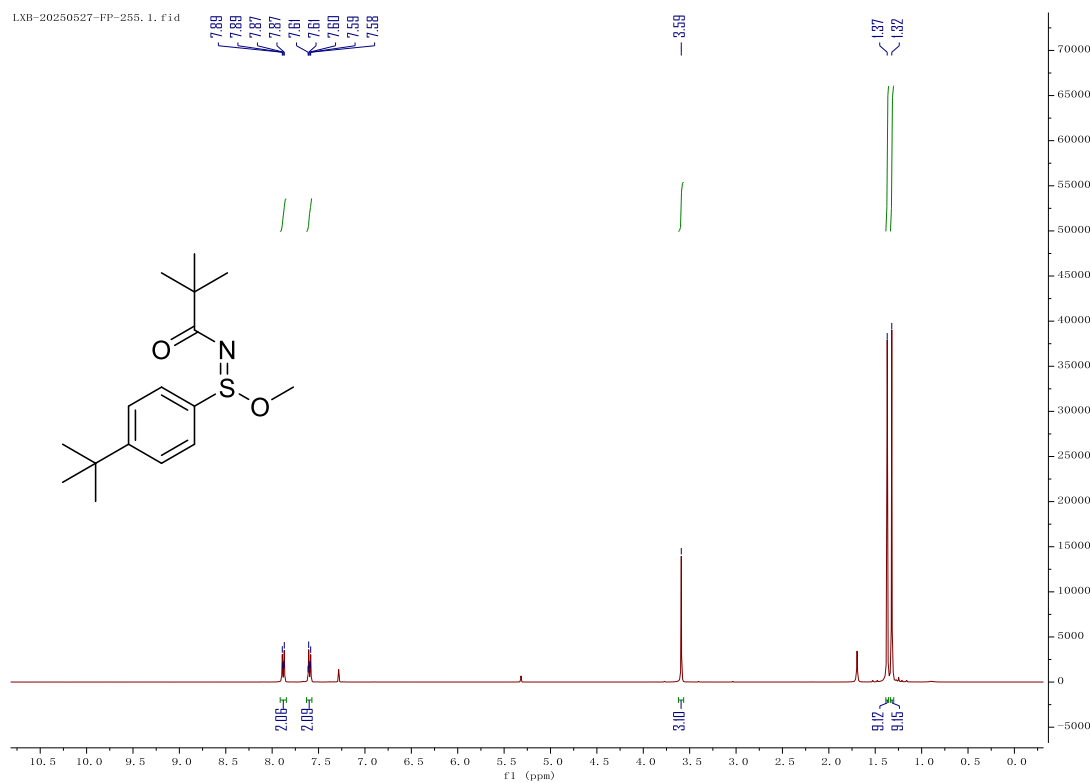
¹H NMR spectrum for compound **3c** (in CDCl₃, 400 MHz)



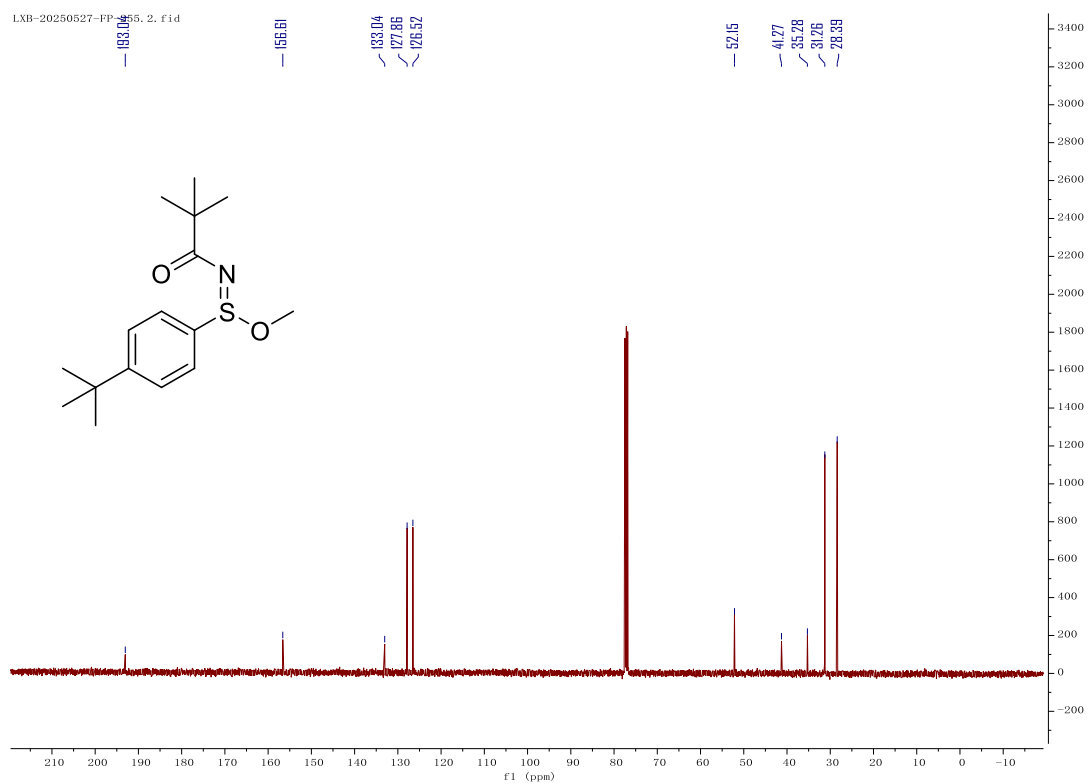
¹³C NMR spectrum for compound **3c** (in CDCl₃, 101 MHz)



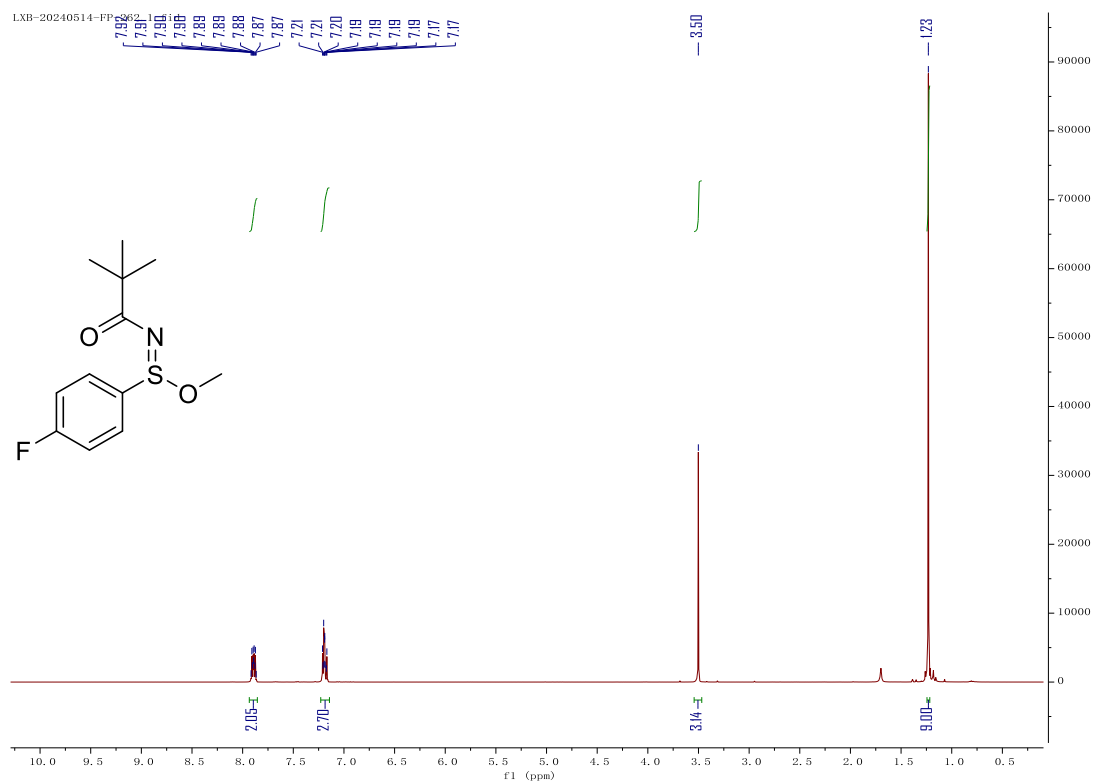
¹H NMR spectrum for compound **3d** (in CDCl₃, 400 MHz)



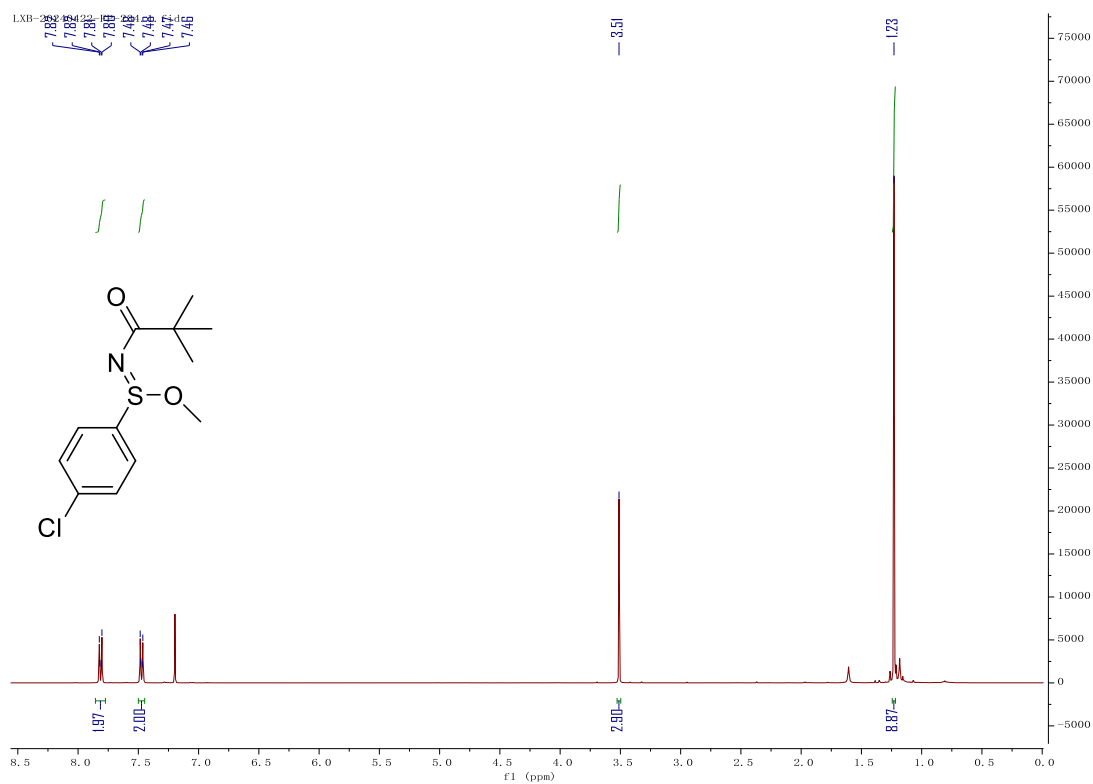
¹³C NMR spectrum for compound **3d** (in CDCl₃, 101 MHz)



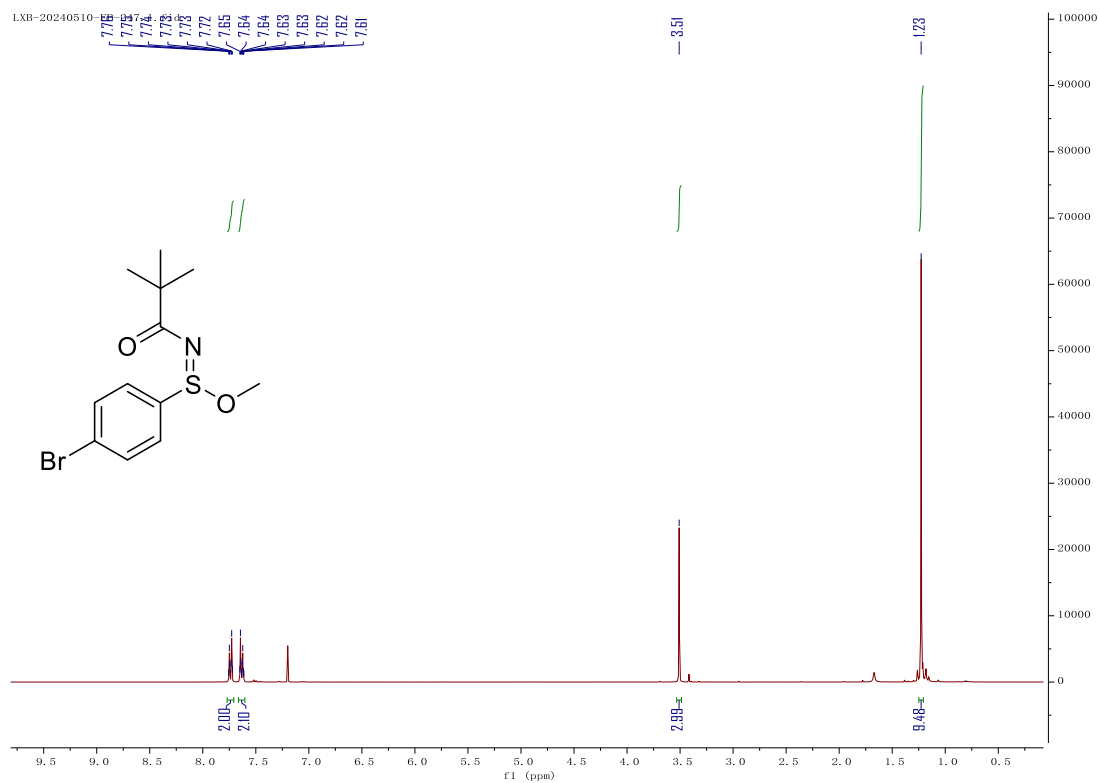
¹H NMR spectrum for compound **3e** (in CDCl₃, 400 MHz)



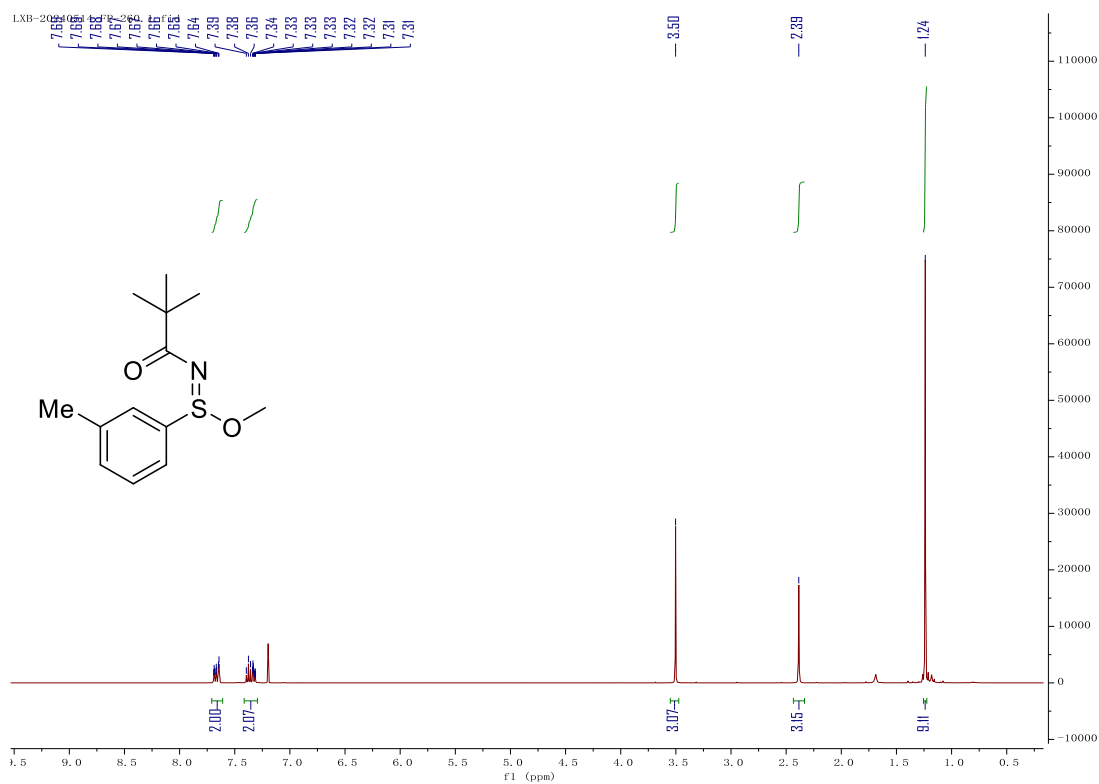
¹H NMR spectrum for compound **3f** (in CDCl₃, 400 MHz)



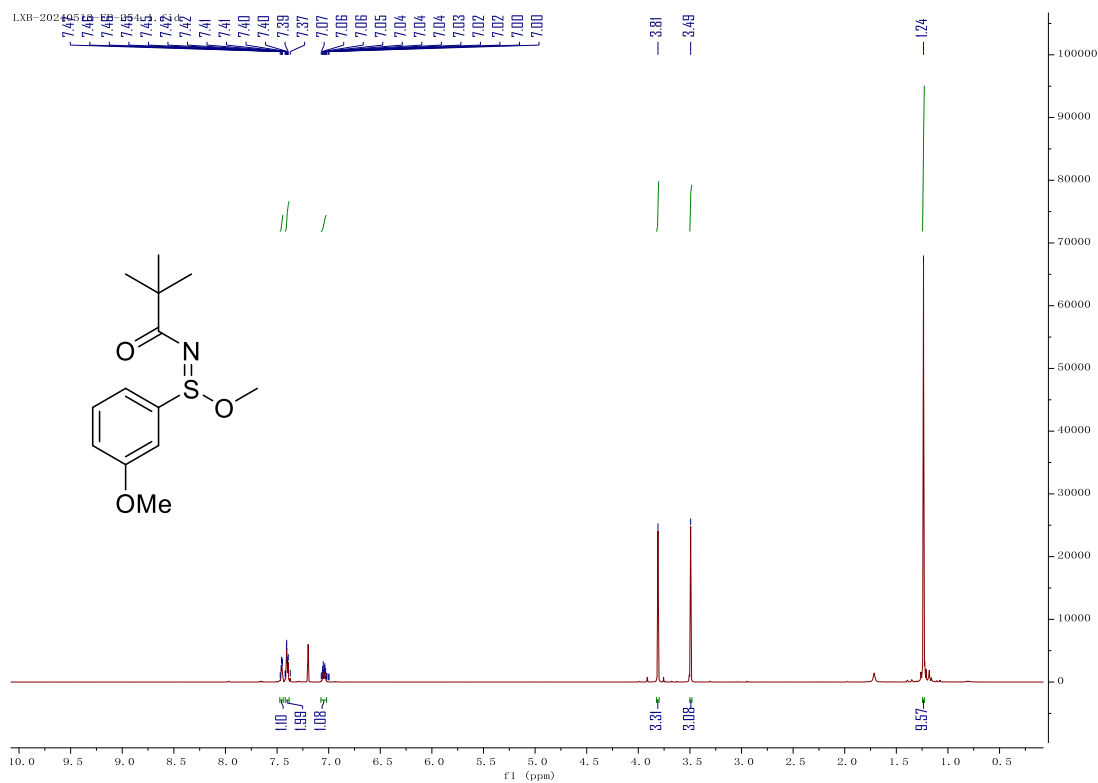
¹H NMR spectrum for compound **3g** (in CDCl₃, 400 MHz)



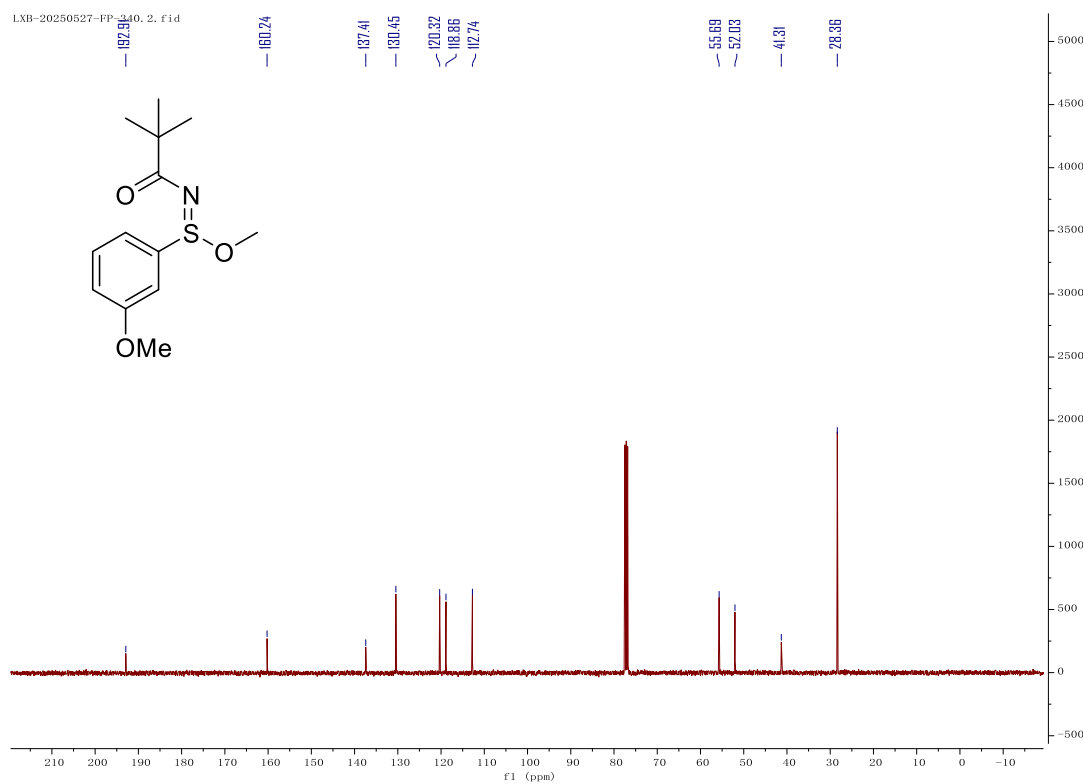
¹H NMR spectrum for compound **3h** (in CDCl₃, 400 MHz)



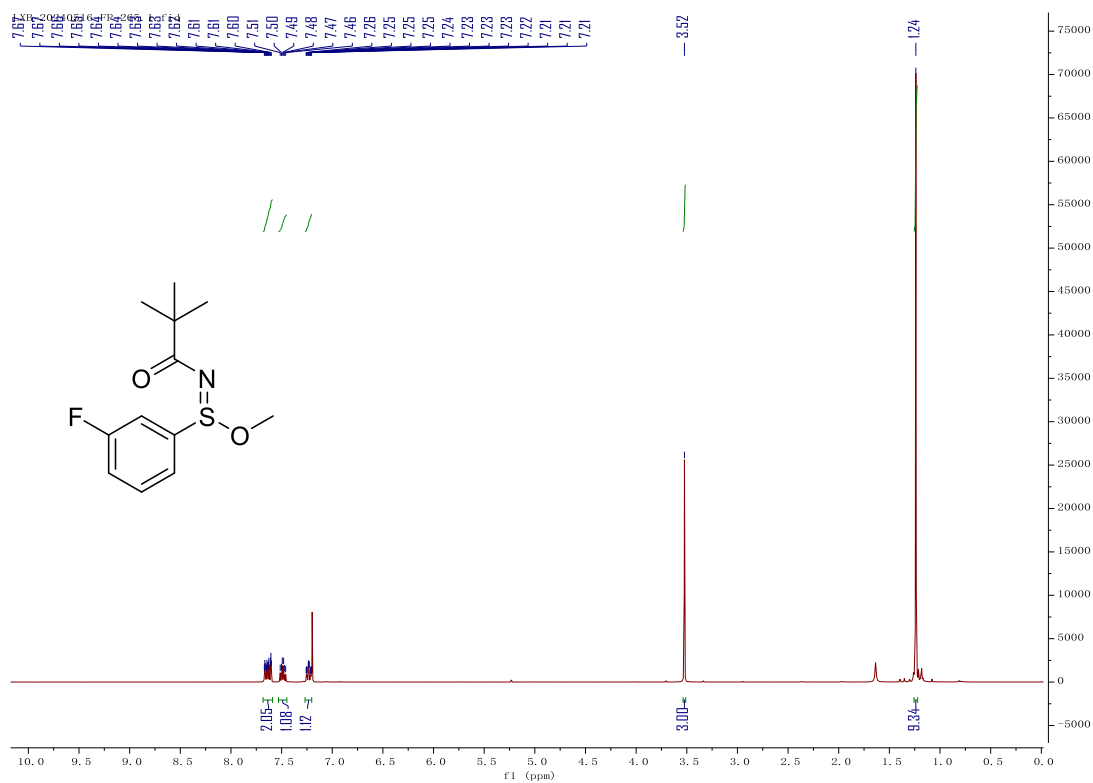
¹H NMR spectrum for compound **3i** (in CDCl₃, 400 MHz)



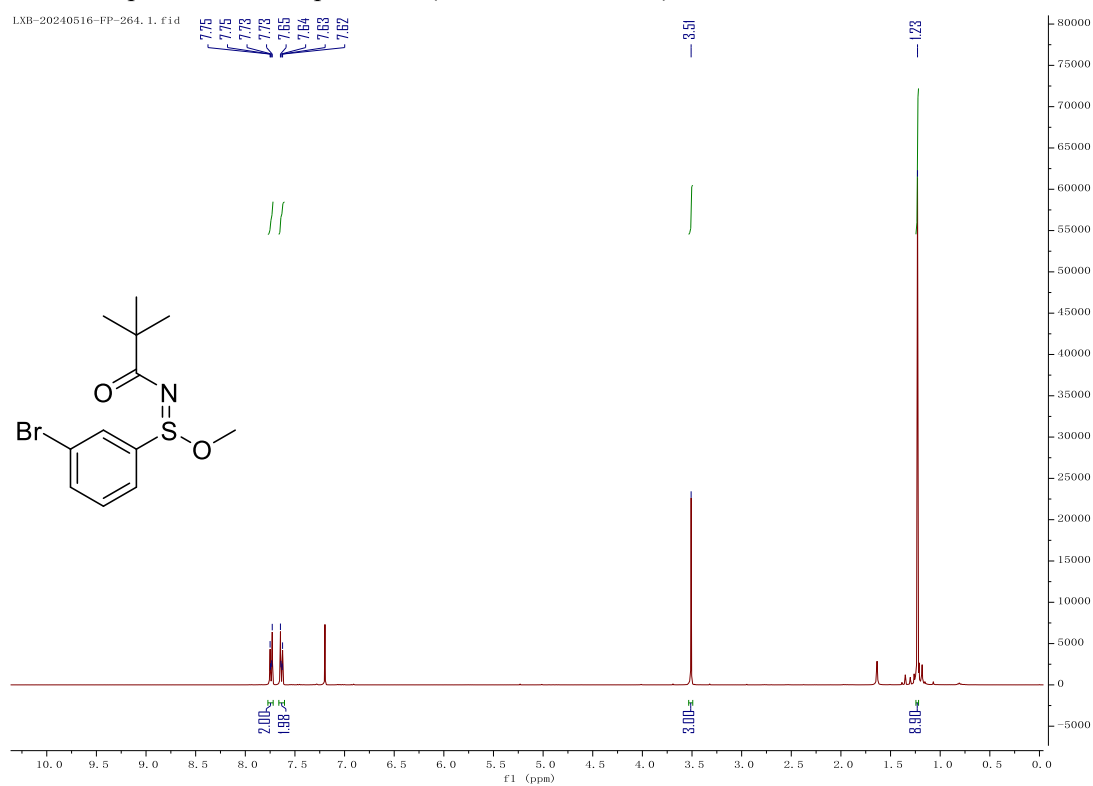
¹³C NMR spectrum for compound **3i** (in CDCl₃, 101 MHz)



¹H NMR spectrum for compound **3j** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **3k** (in CDCl₃, 400 MHz)



Chemical structure: CC(C)(C)C(=O)N=S(OC)c1ccc(C)cc1

¹H NMR spectrum (ppm):

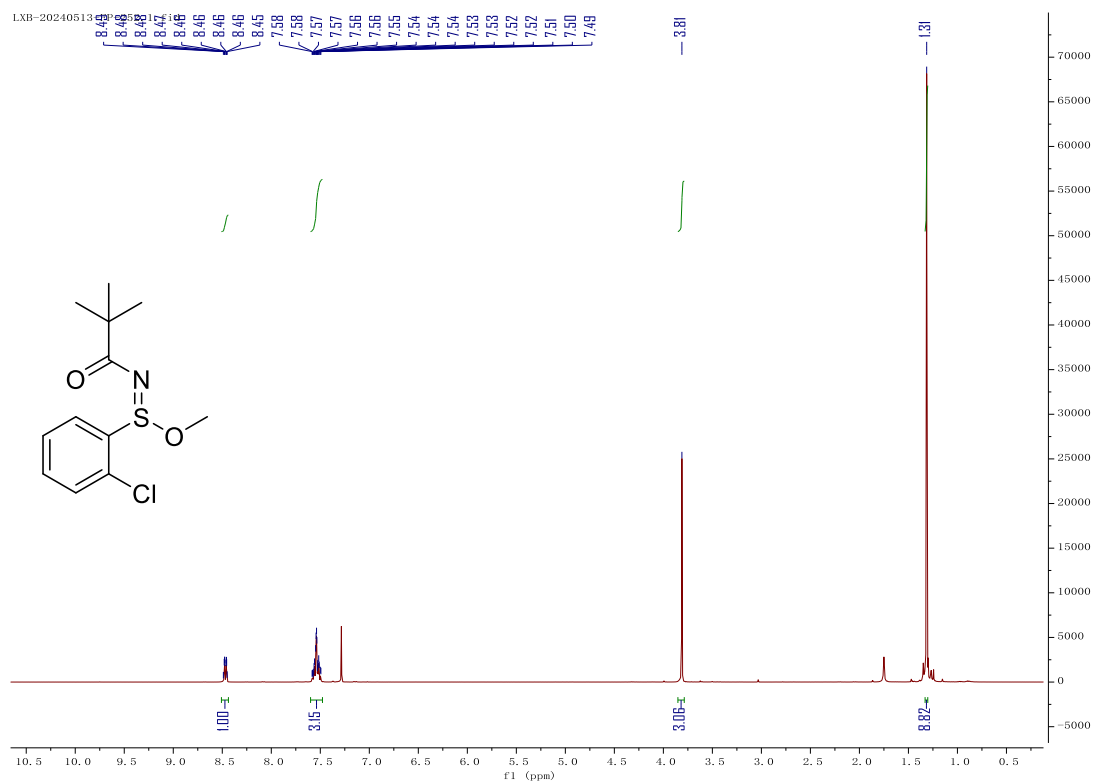
- 8.35, 8.34, 8.33, 8.37, 8.37, 8.37 (aromatic protons, integration 0.98)
- 7.52, 7.51, 7.51, 7.49, 7.49, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.34, 7.33, 7.32 (aromatic protons, integration 1.01, 1.03, 0.98)
- 3.58 (tert-butyl methyls, integration 3.01)
- 2.61 (methoxy singlet, integration 3.03)
- 1.32 (methyl singlet, integration 9.00)

CC(C)(C)C(=O)N=S(c1ccccc1F)OC

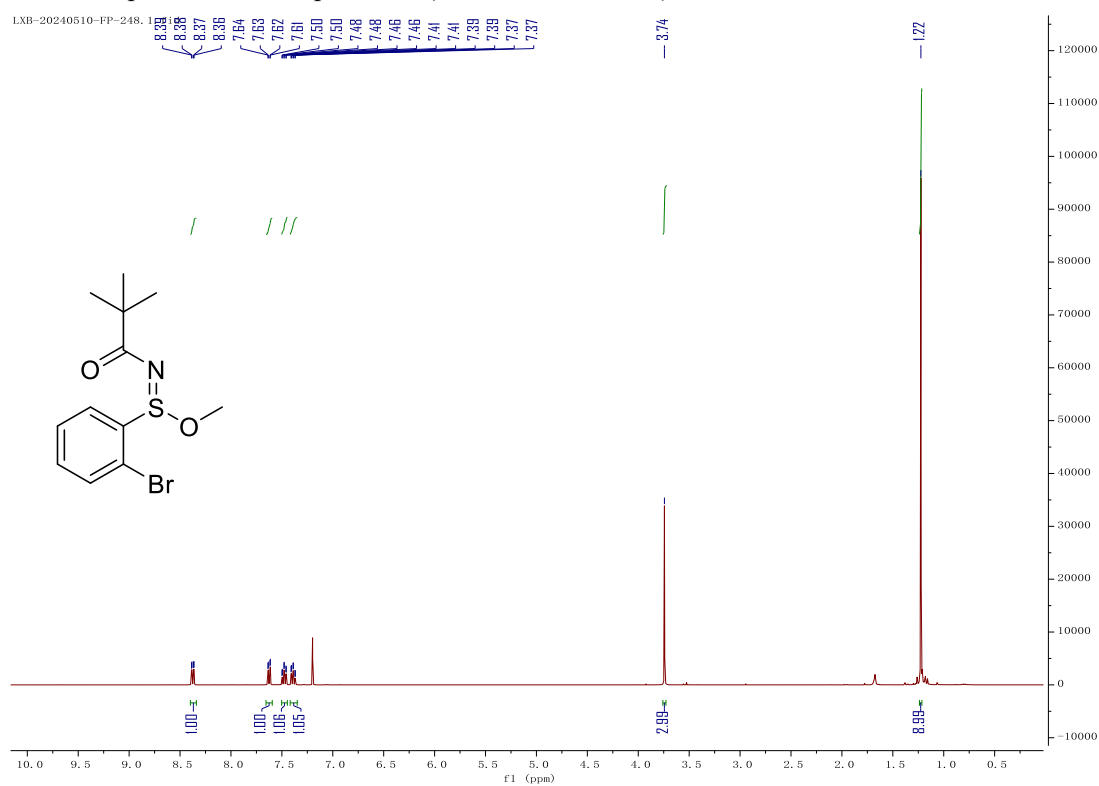
¹H NMR spectrum (CDCl₃) showing peaks in the aromatic region (7.0-8.3 ppm), a methoxy singlet (3.8 ppm), a tert-butyl singlet (1.2 ppm), and a solvent peak (1.5 ppm). Integration values are provided for the main signals.

Chemical Shift (ppm)	Integration
8.28, 8.26, 8.24, 8.22, 8.20	0.99
7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.32, 7.30, 7.30, 7.28, 7.18, 7.16, 7.15, 7.13, 7.13	1.01, 1.04, 1.01
3.86	3.00
1.23	9.29

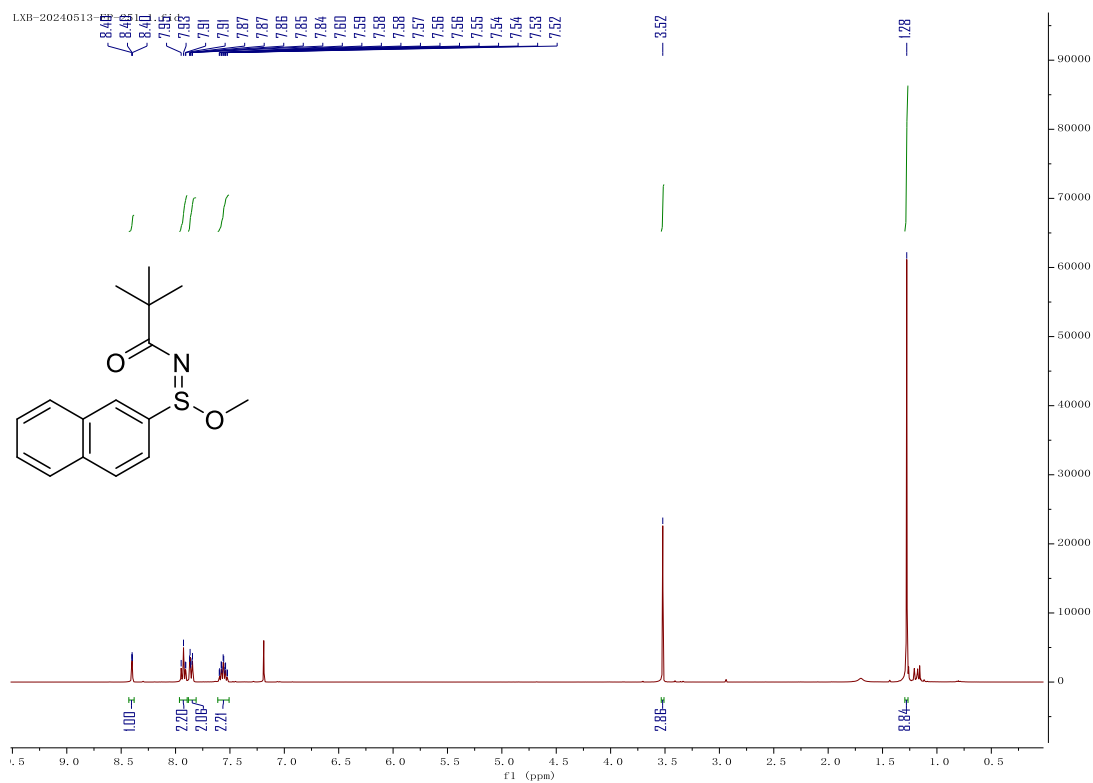
¹H NMR spectrum for compound **3n** (in CDCl₃, 400 MHz)



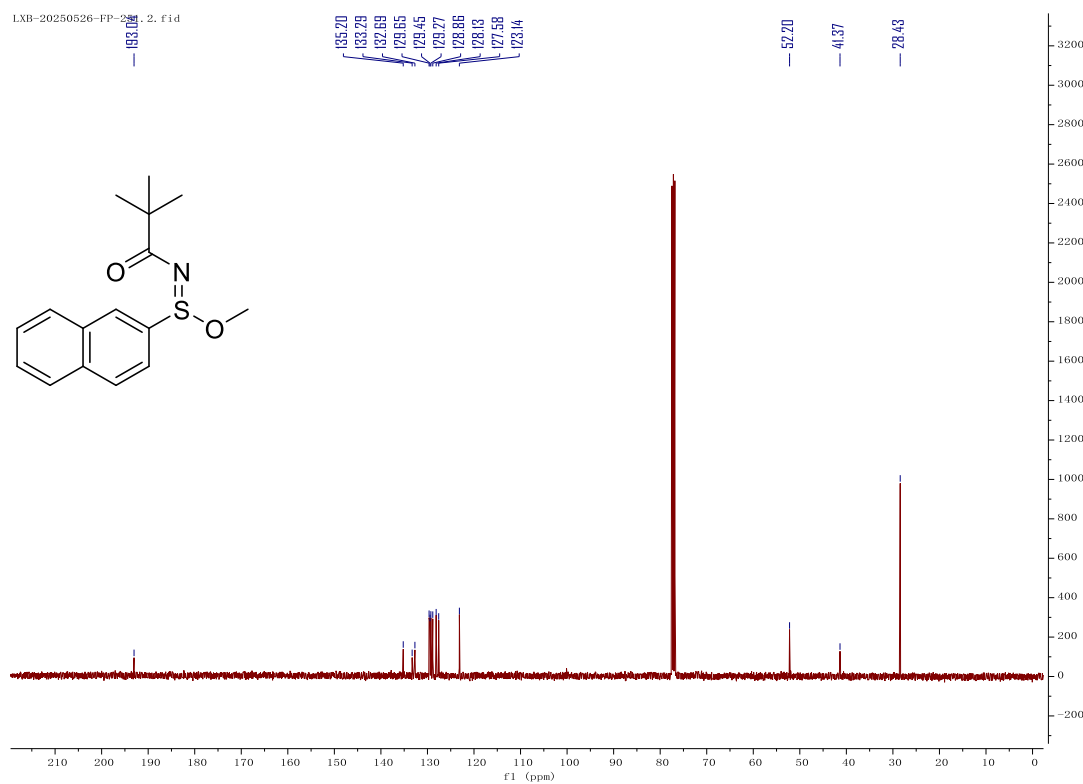
¹H NMR spectrum for compound **3o** (in CDCl₃, 400 MHz)



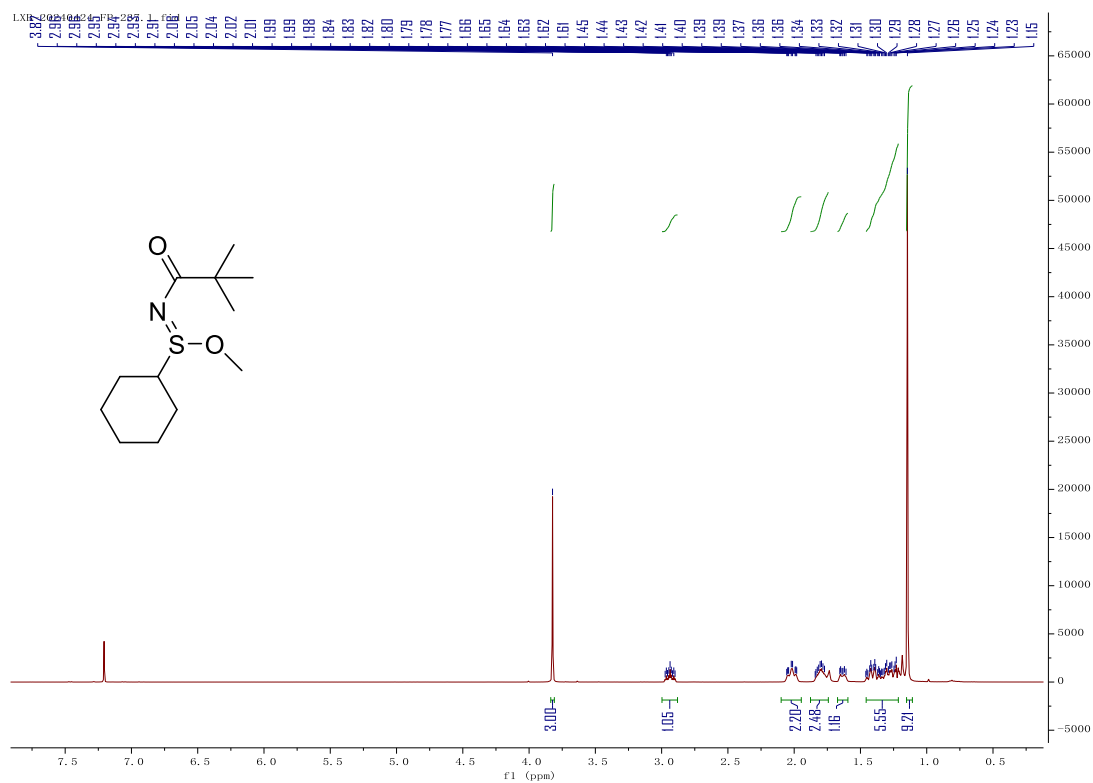
¹H NMR spectrum for compound **3p** (in CDCl₃, 400 MHz)



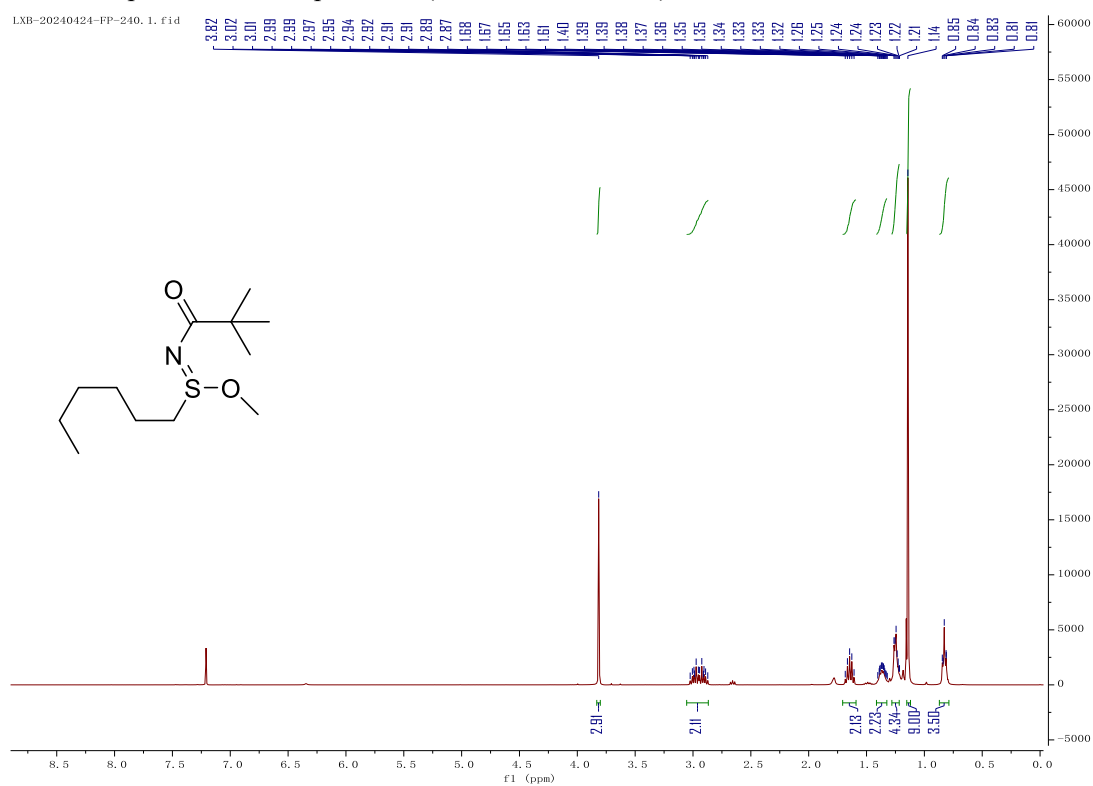
¹³C NMR spectrum for compound **3p** (in CDCl₃, 101 MHz)



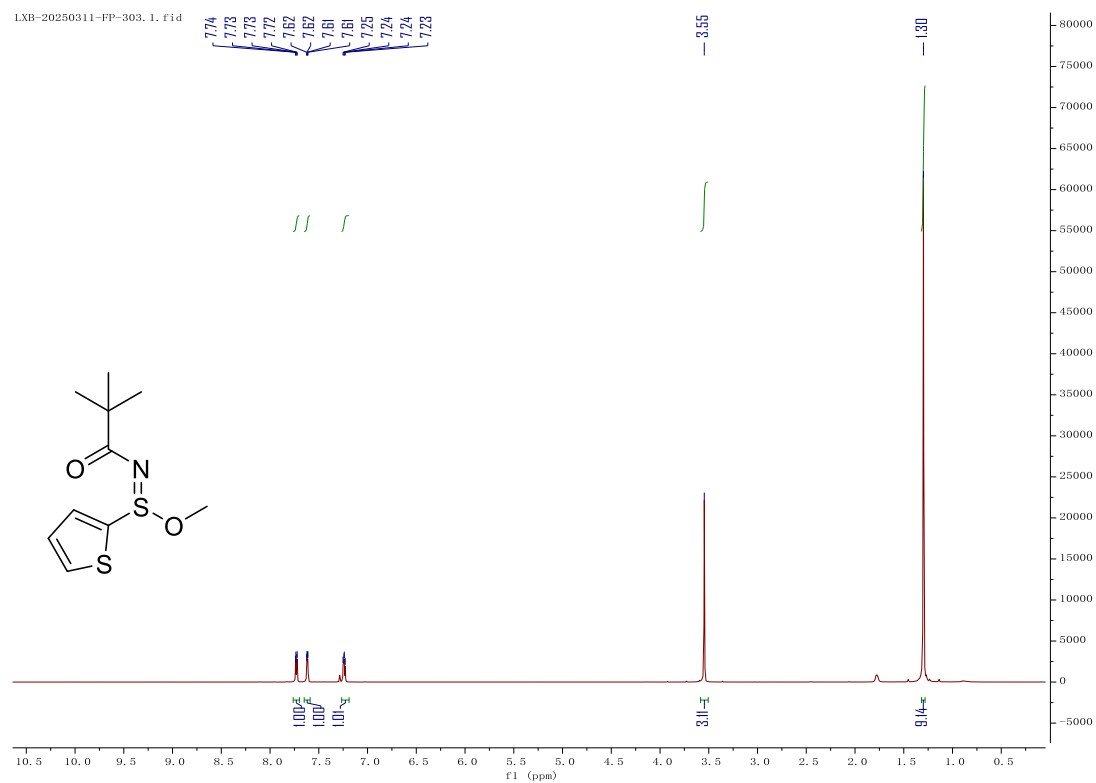
¹H NMR spectrum for compound **3q** (in CDCl₃, 400 MHz)



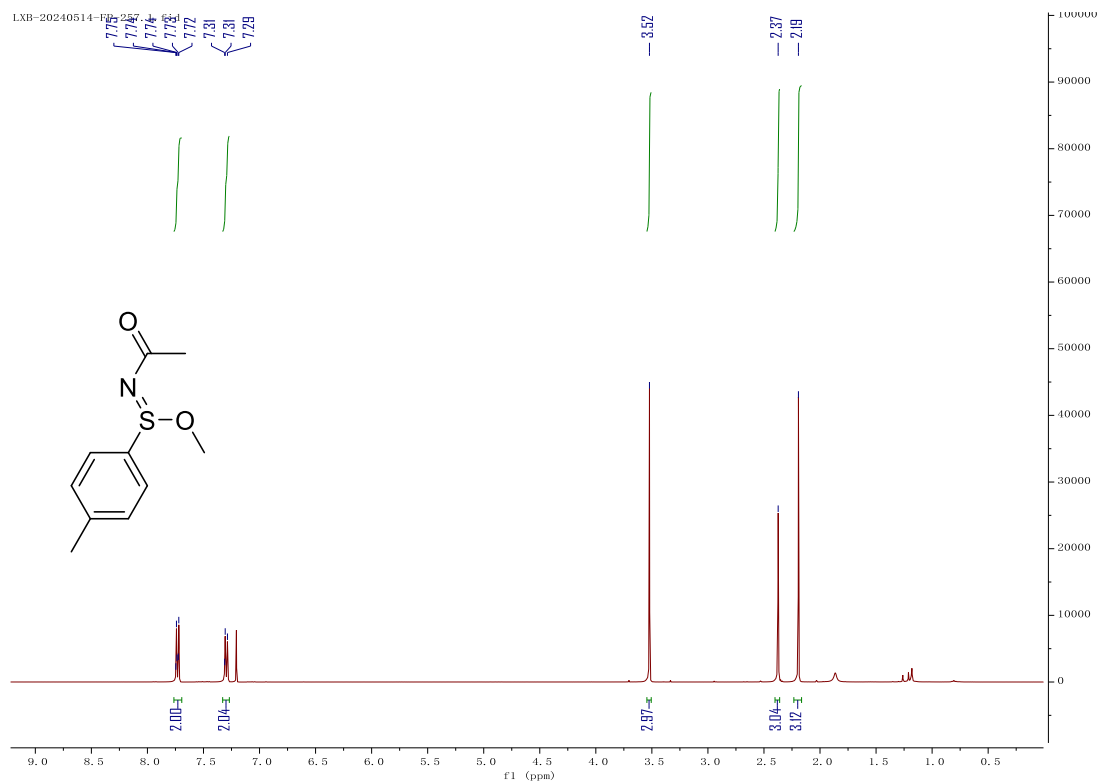
¹H NMR spectrum for compound **3r** (in CDCl₃, 400 MHz)



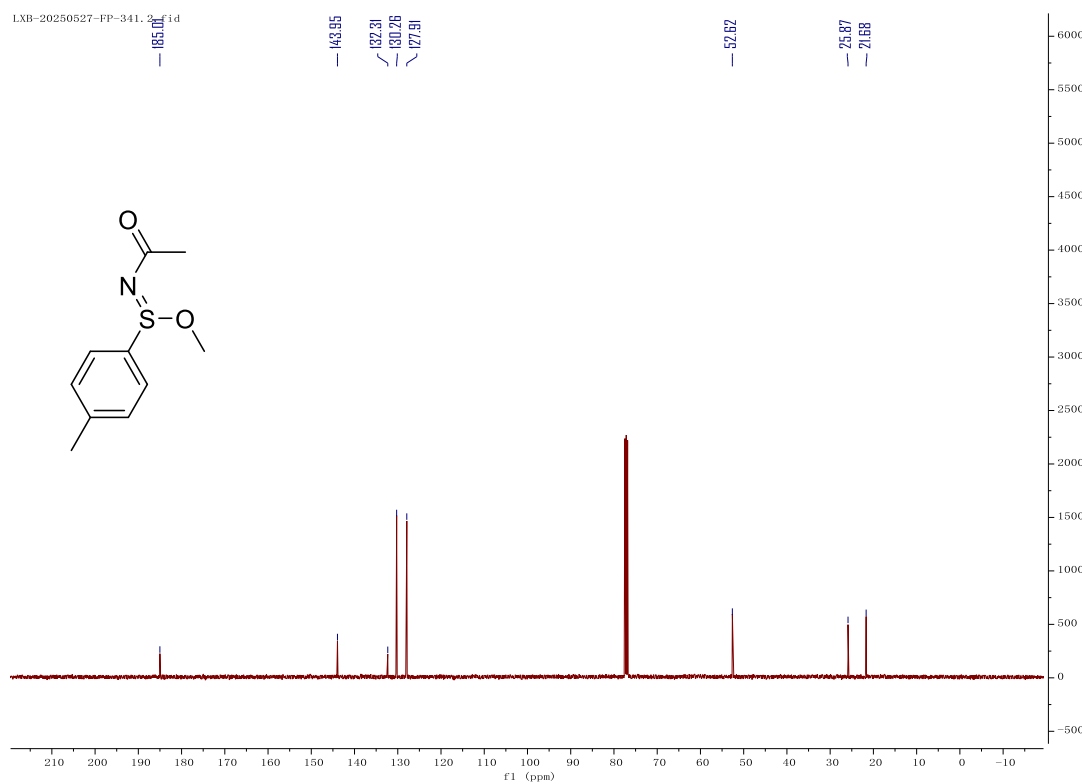
¹H NMR spectrum for compound **3s** (in CDCl₃, 400 MHz)



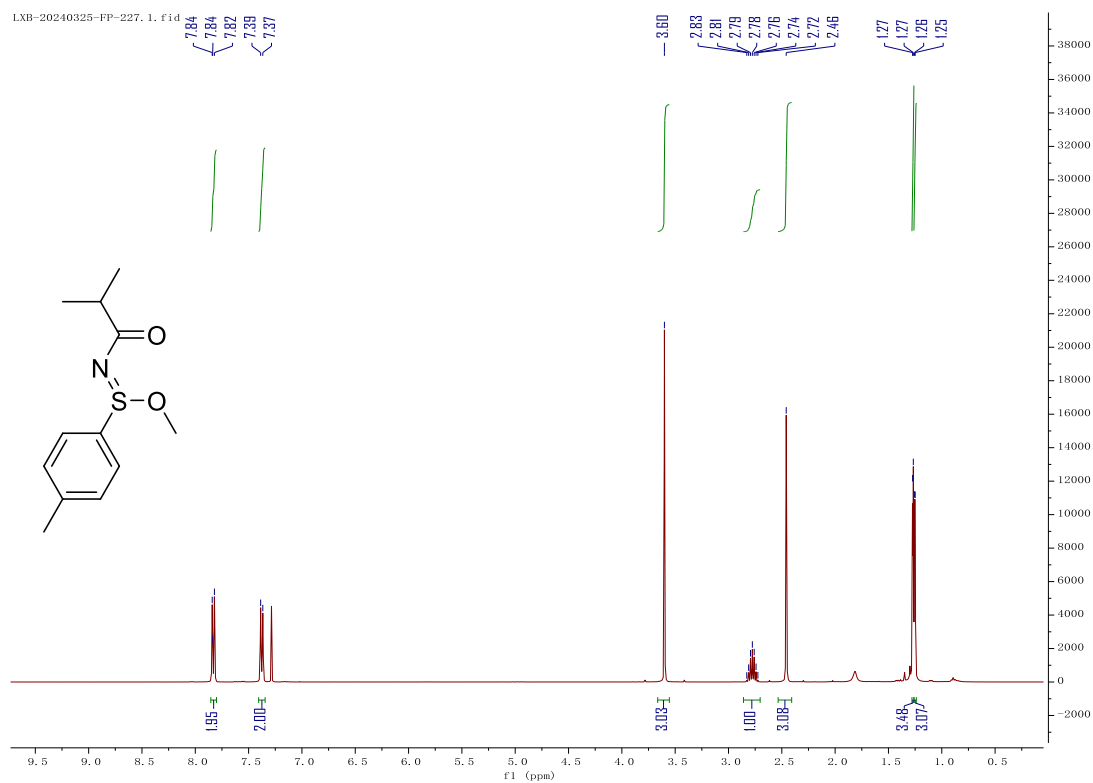
¹H NMR spectrum for compound **3t** (in CDCl₃, 400 MHz)



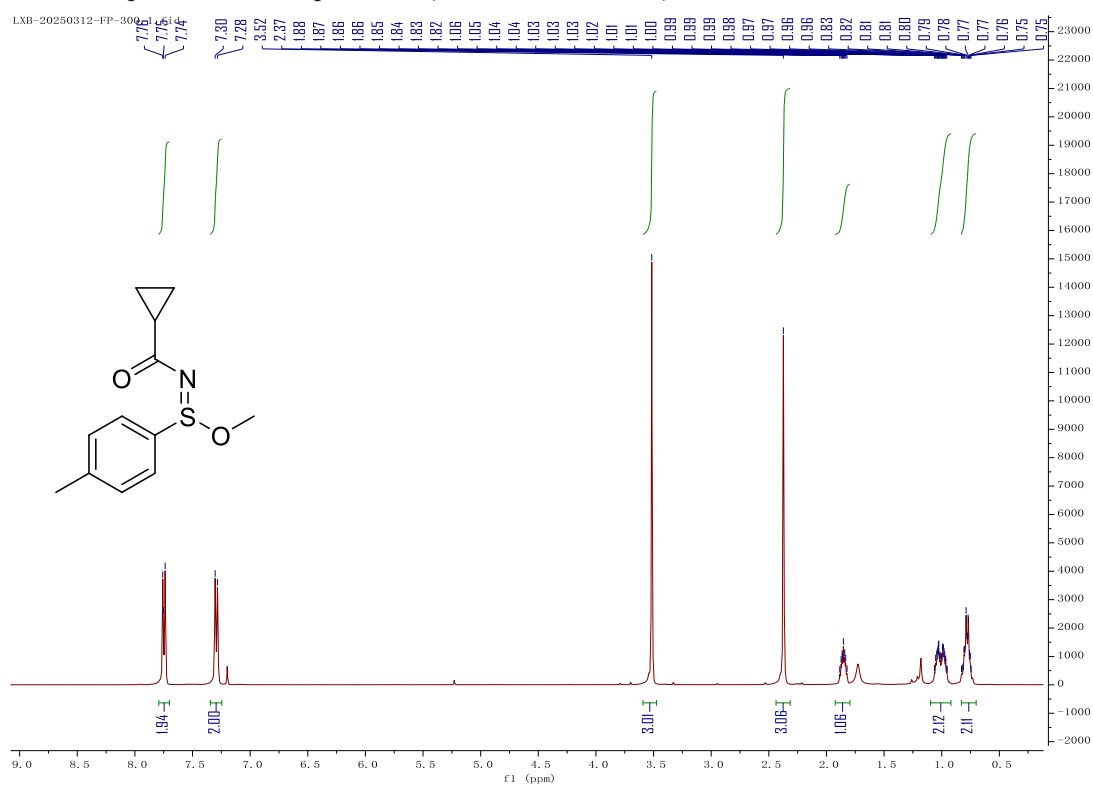
¹³C NMR spectrum for compound **3t** (in CDCl₃, 101 MHz)



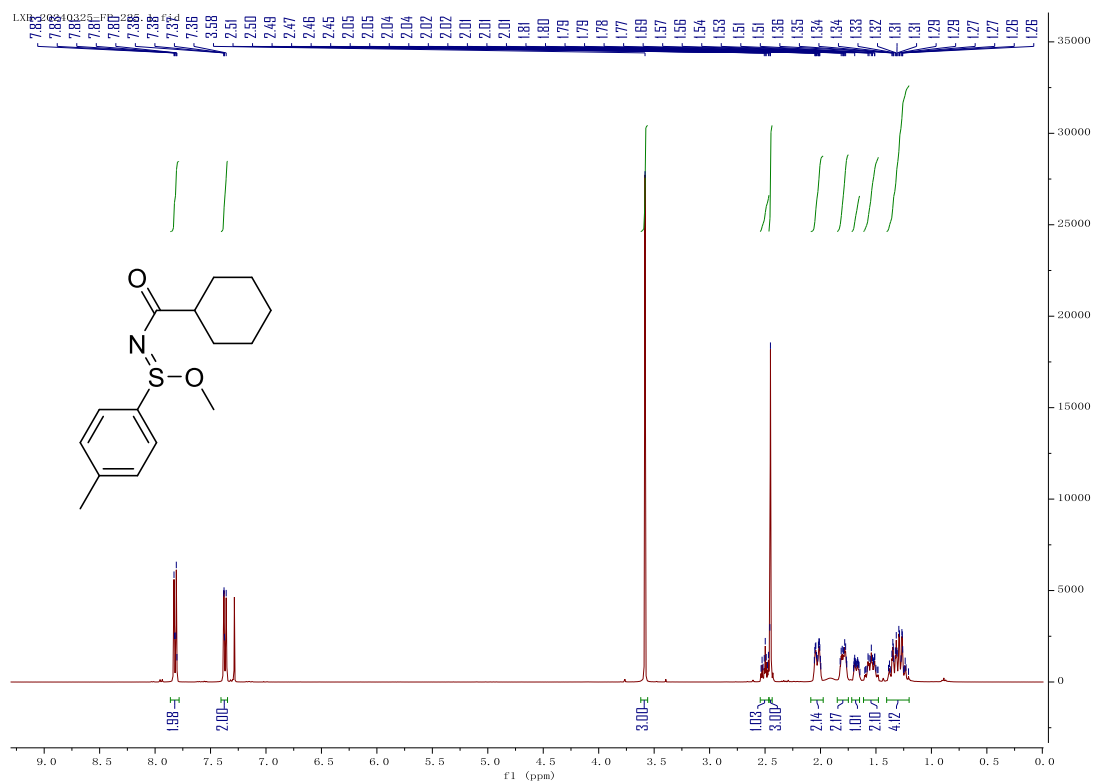
¹H NMR spectrum for compound **3u** (in CDCl₃, 400 MHz)



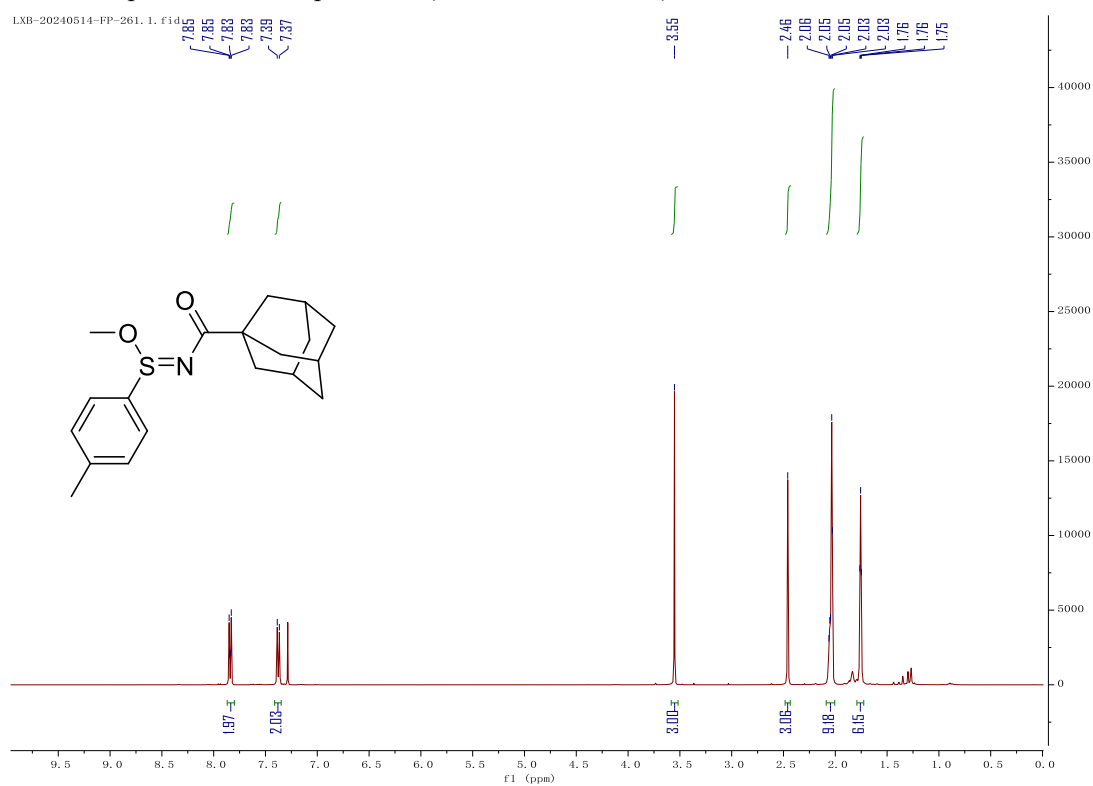
¹H NMR spectrum for compound **3v** (In CDCl₃, 400 MHz)



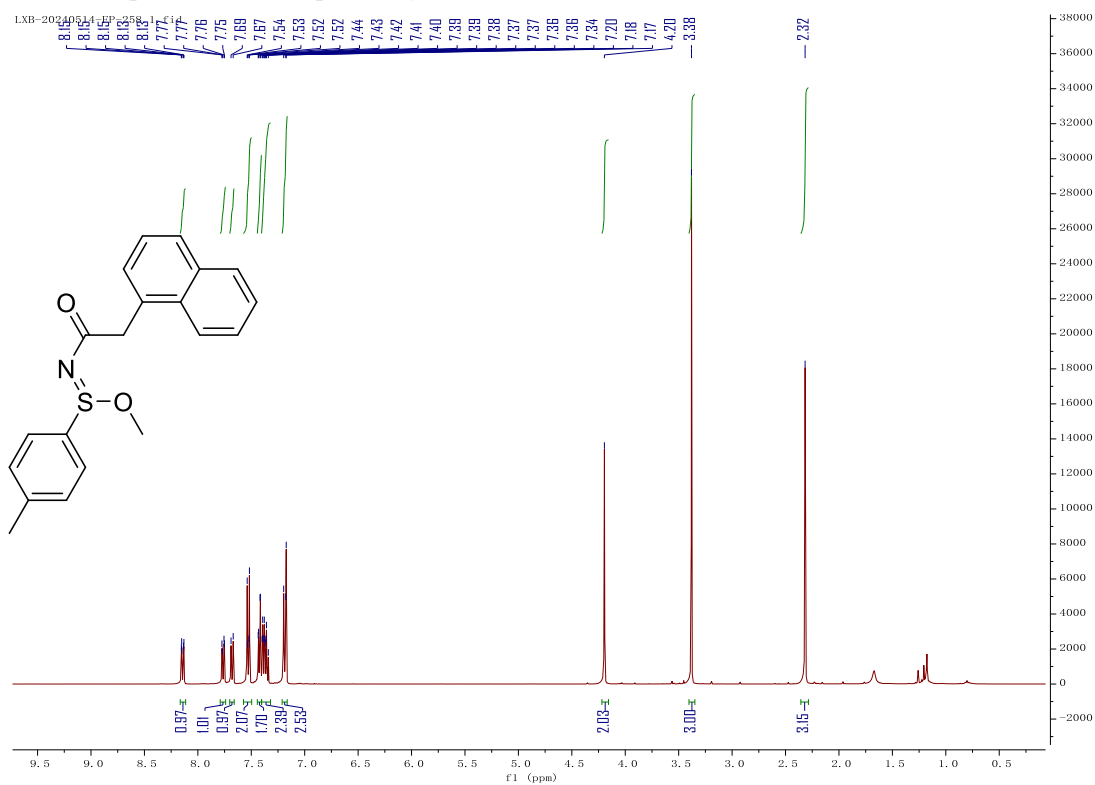
¹H NMR spectrum for compound **3w** (in CDCl₃, 400 MHz)



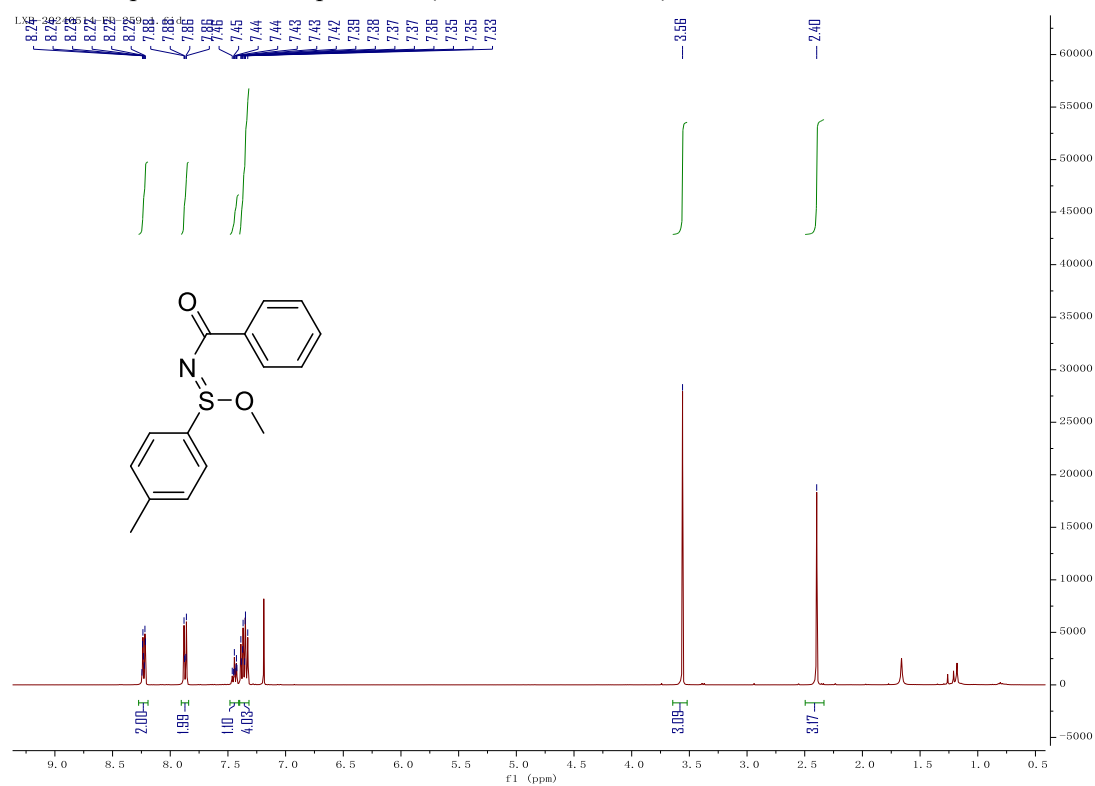
¹H NMR spectrum for compound **3x** (in CDCl₃, 400 MHz)



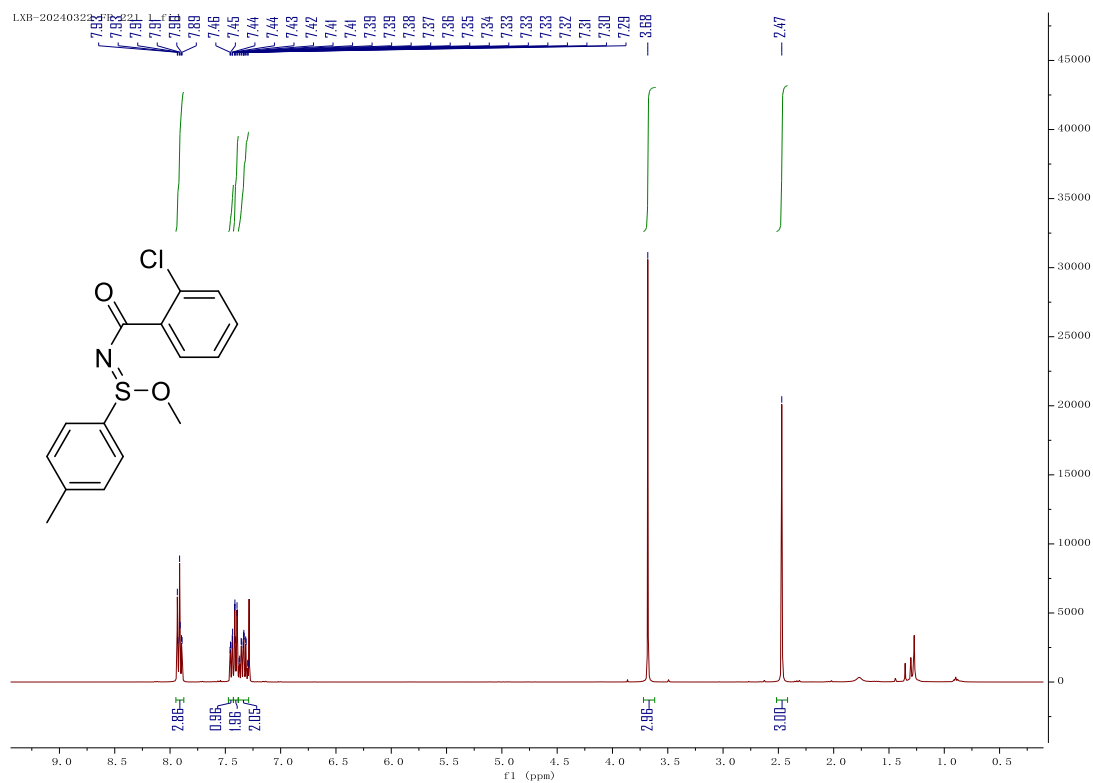
¹H NMR spectrum for compound **3y** (in CDCl₃, 400 MHz)



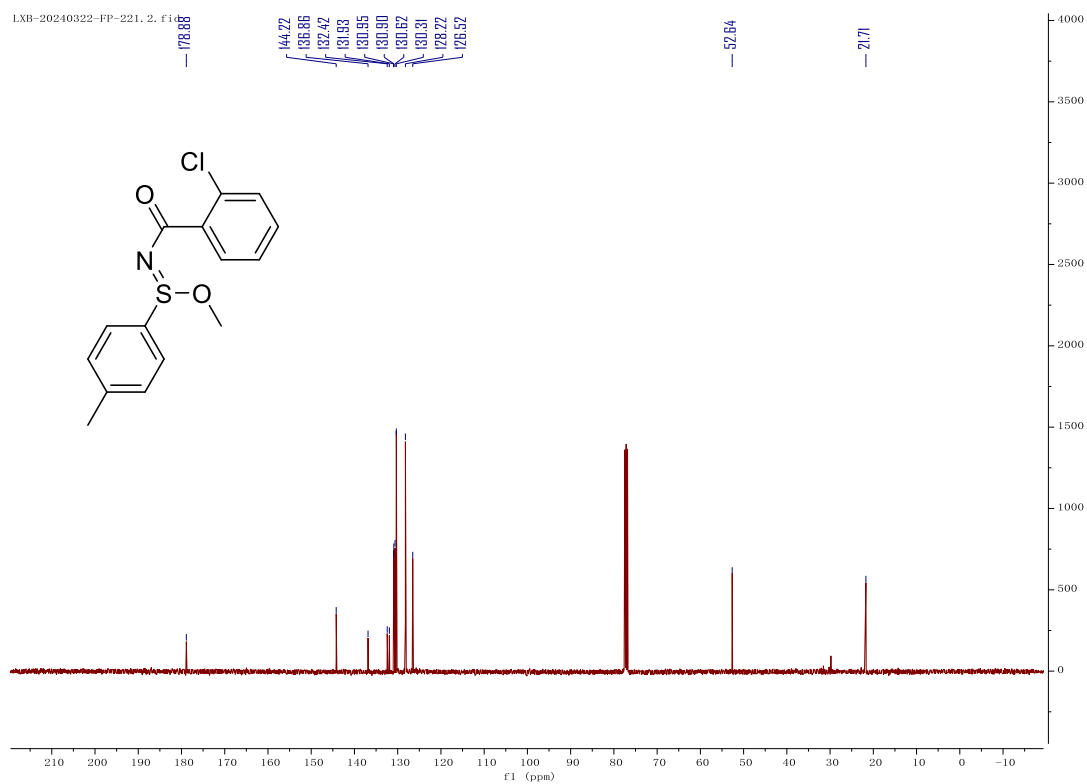
¹H NMR spectrum for compound **3z** (in CDCl₃, 400 MHz)



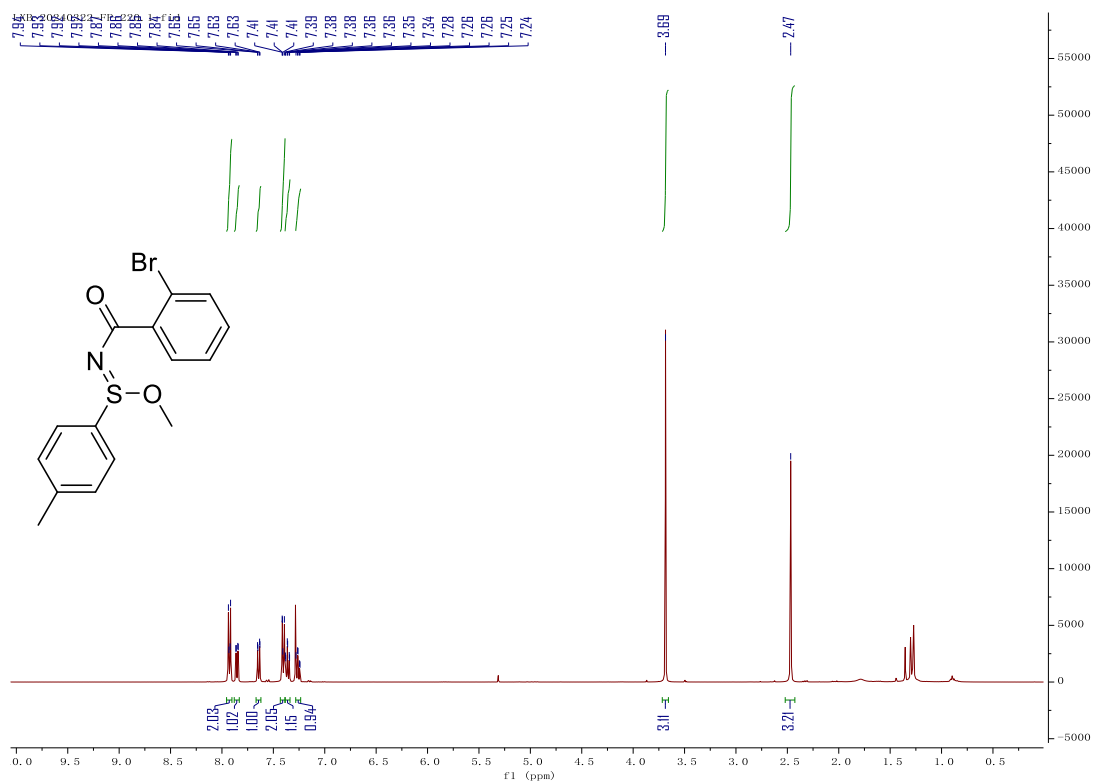
¹H NMR spectrum for compound **3a'** (in CDCl₃, 400 MHz)



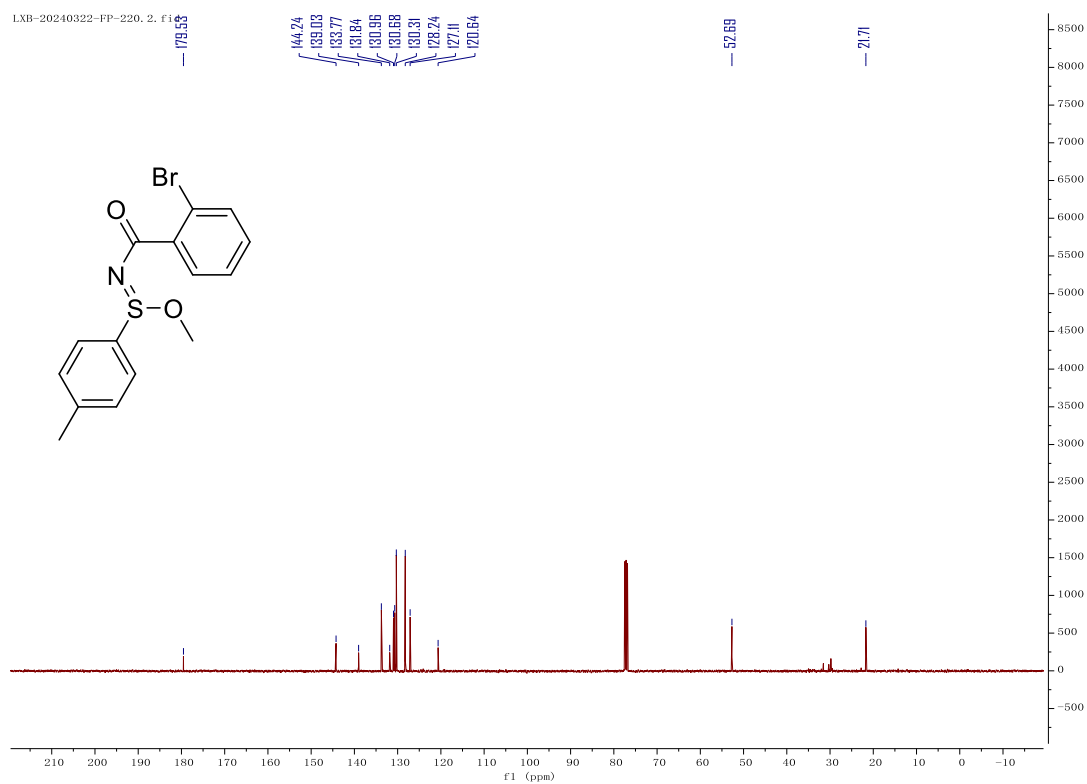
¹³C NMR spectrum for compound **3a'** (in CDCl₃, 101 MHz)



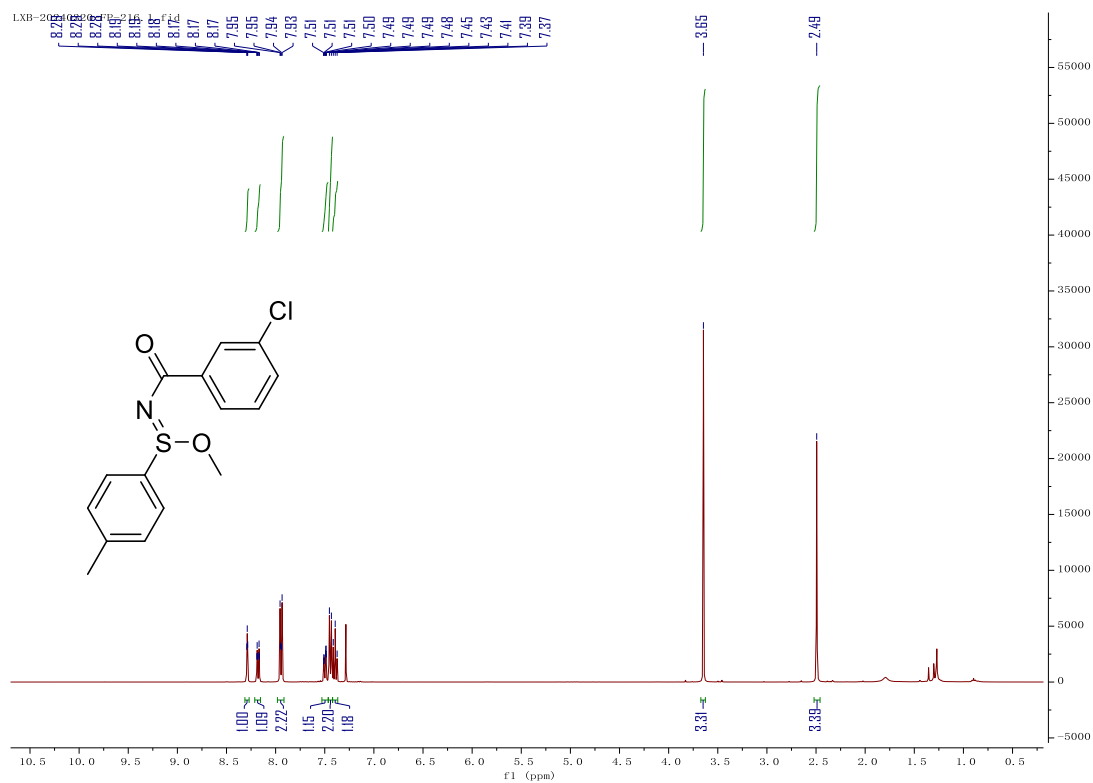
^1H NMR spectrum for compound **3b'** (in CDCl_3 , 400 MHz)



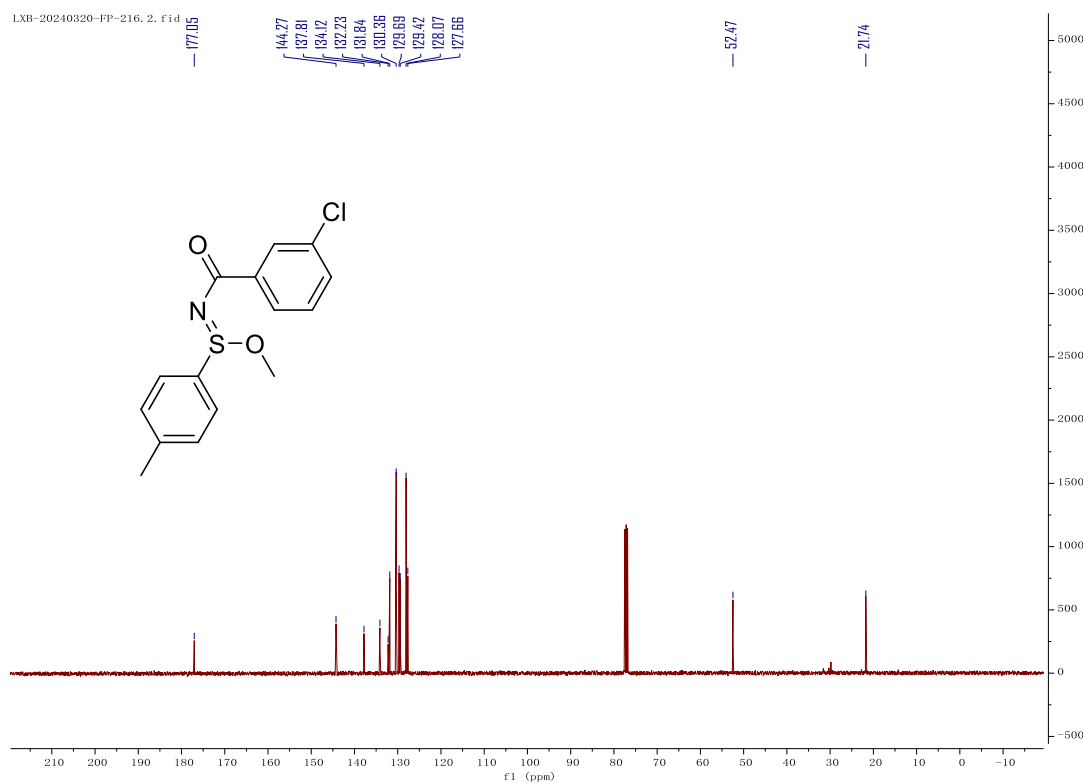
^{13}C NMR spectrum for compound **3b'** (in CDCl_3 , 101 MHz)



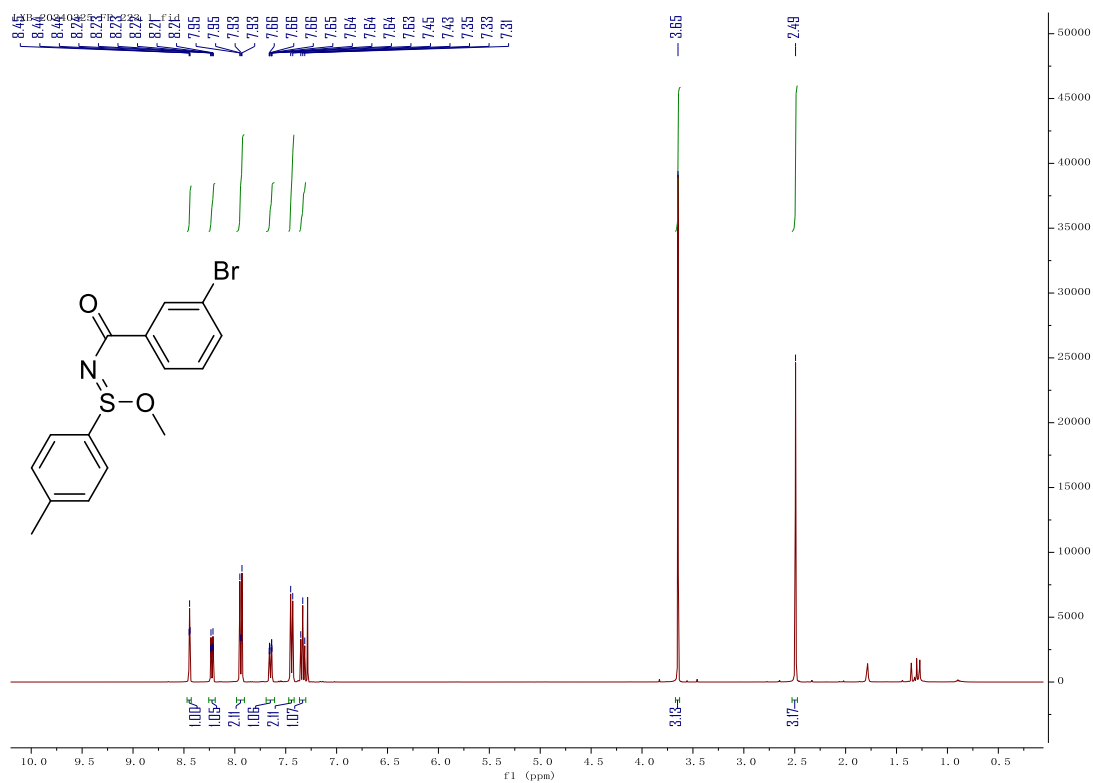
¹H NMR spectrum for compound **3c'** (in CDCl₃, 400 MHz)



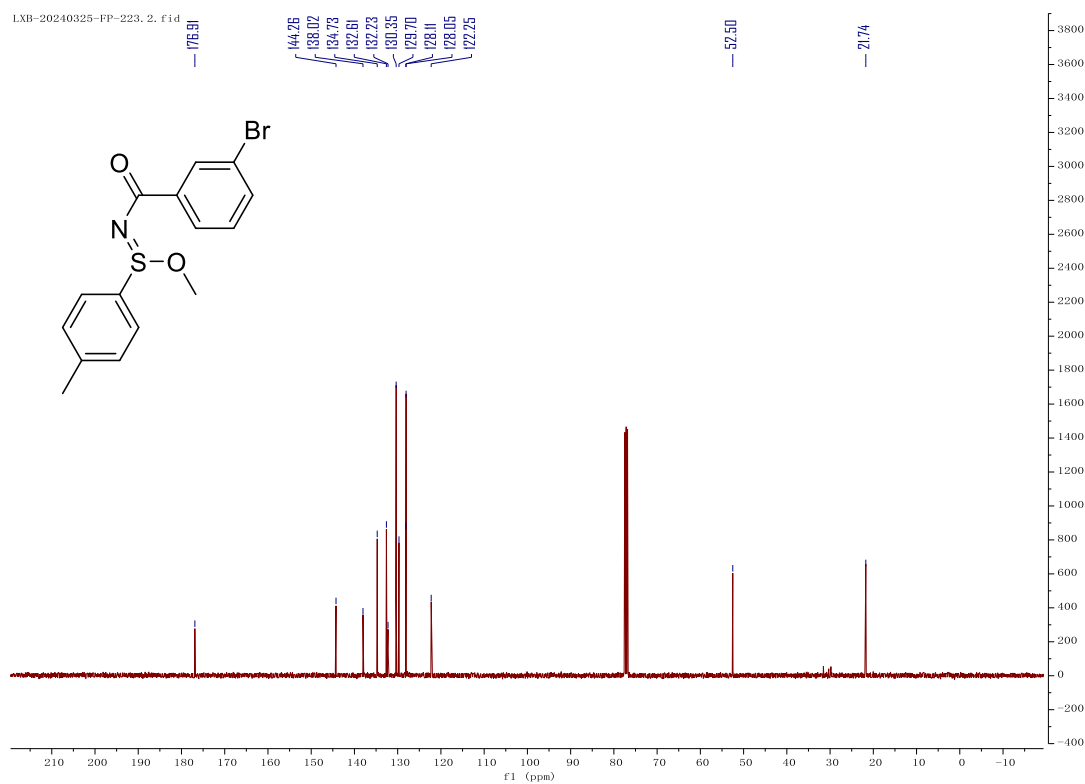
¹³C NMR spectrum for compound **3c'** (in CDCl₃, 101 MHz)



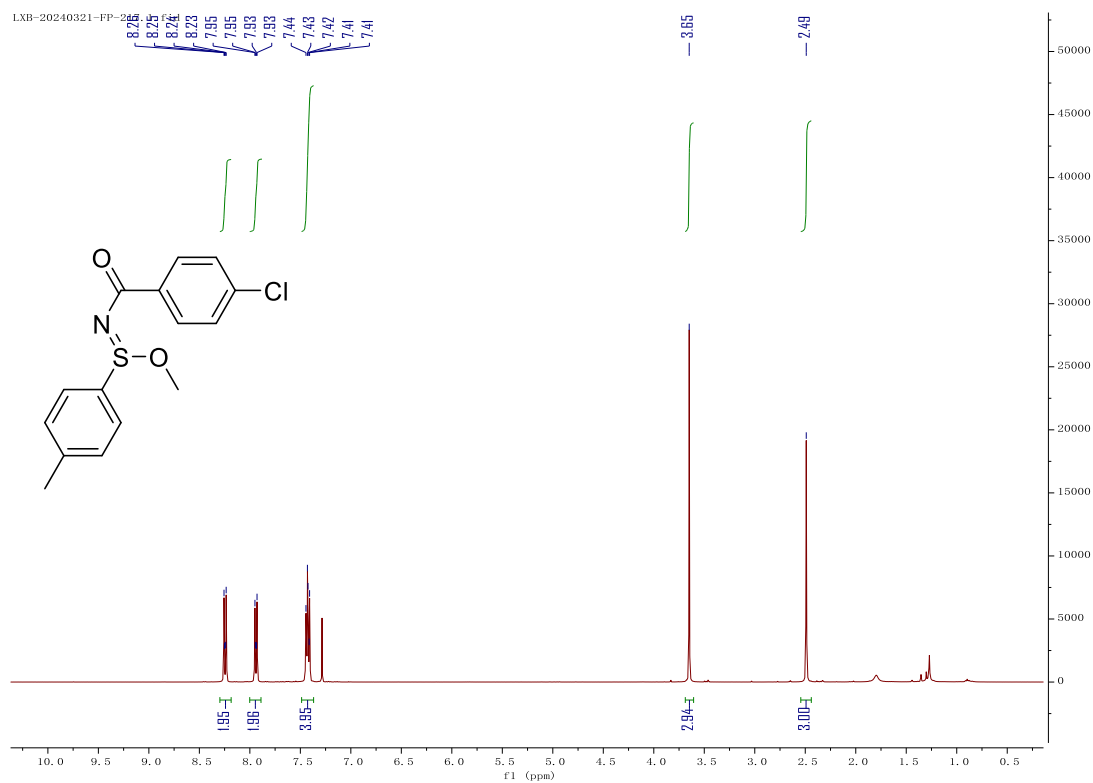
¹H NMR spectrum for compound **3d'** (in CDCl₃, 400 MHz)



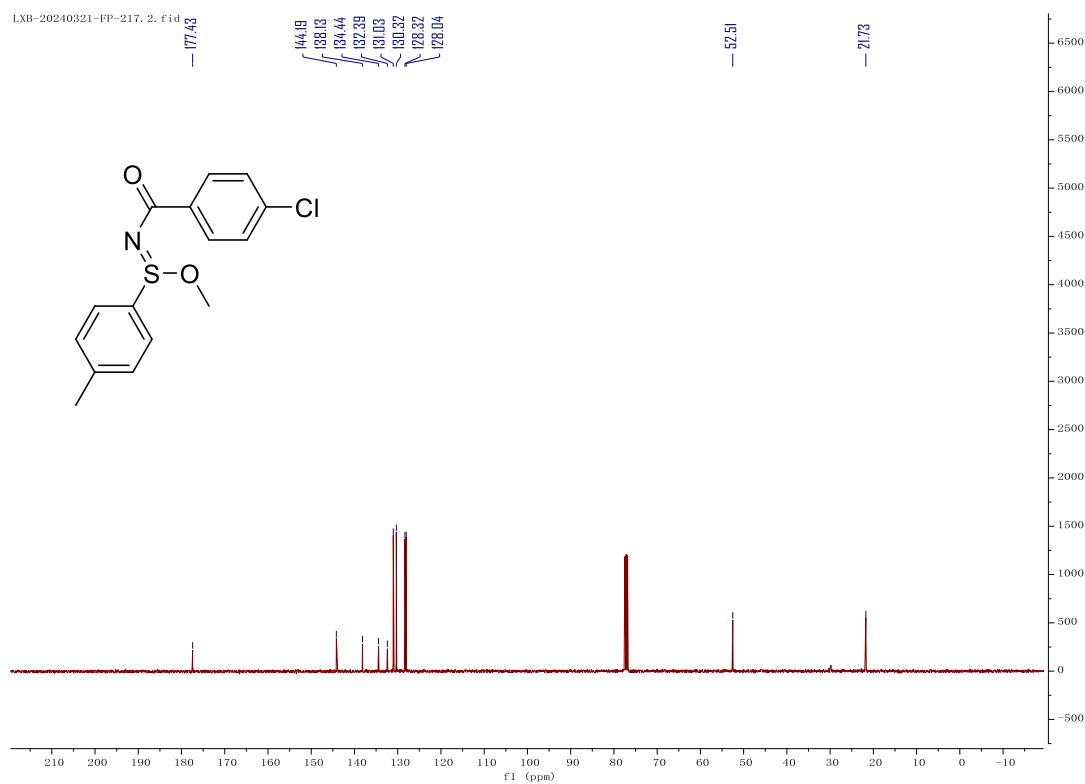
¹³C NMR spectrum for compound **3d'** (in CDCl₃, 101 MHz)



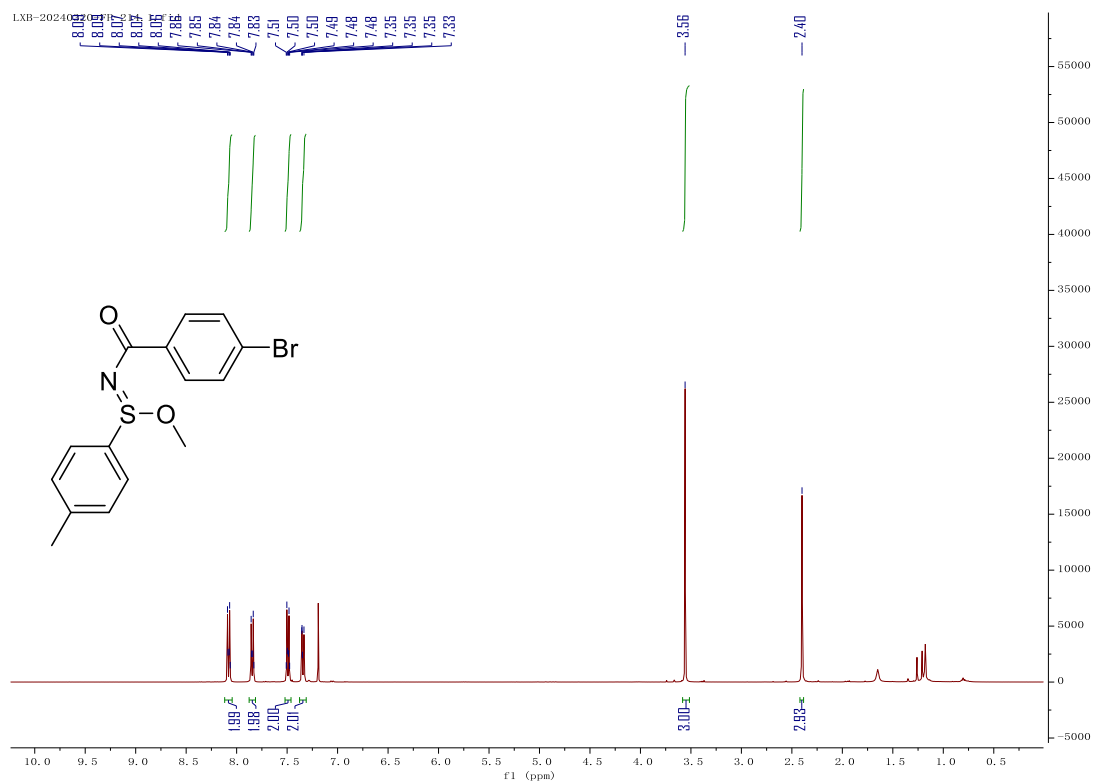
¹H NMR spectrum for compound **3e'** (in CDCl₃, 400 MHz)



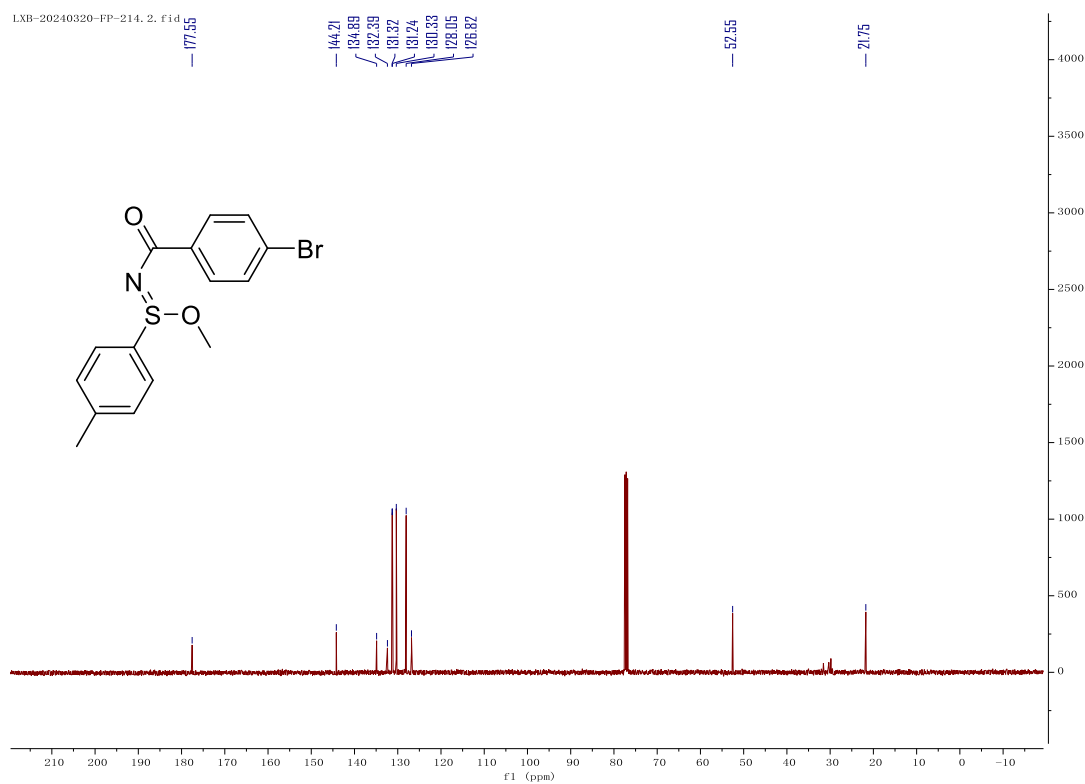
¹³C NMR spectrum for compound **3e'** (in CDCl₃, 101 MHz)



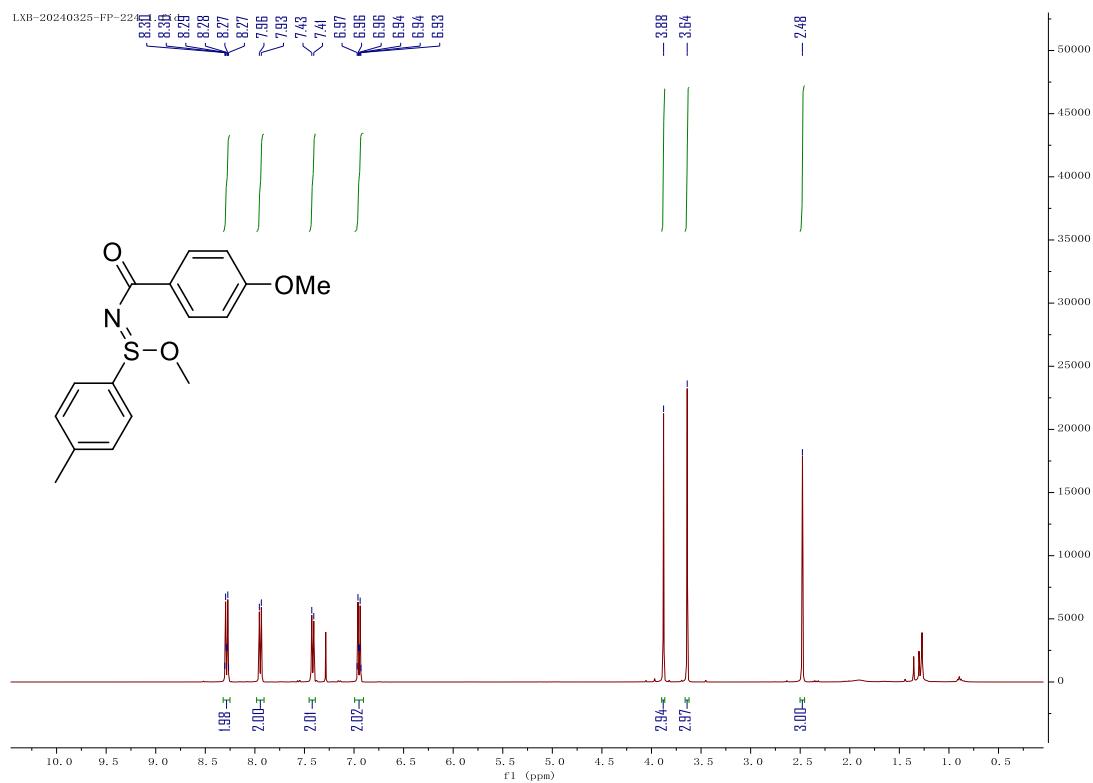
^1H NMR spectrum for compound **3f'** (in CDCl_3 , 400 MHz)



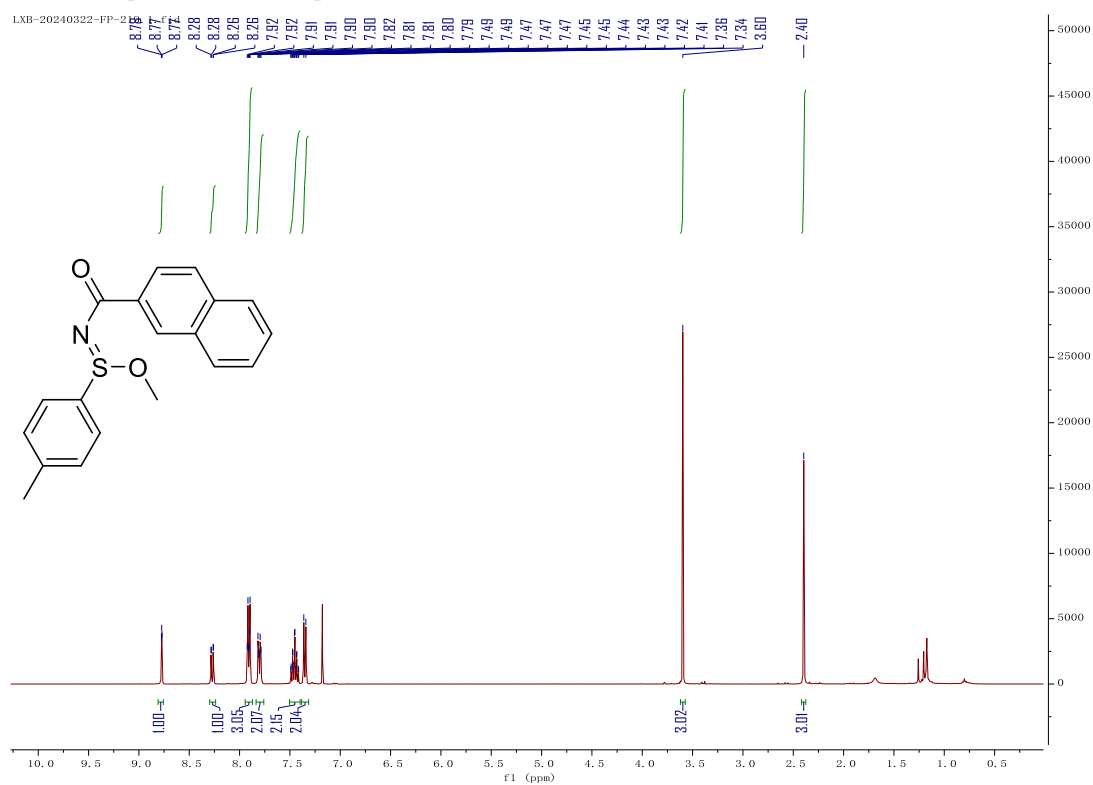
^{13}C NMR spectrum for compound **3f'** (in CDCl_3 , 101 MHz)



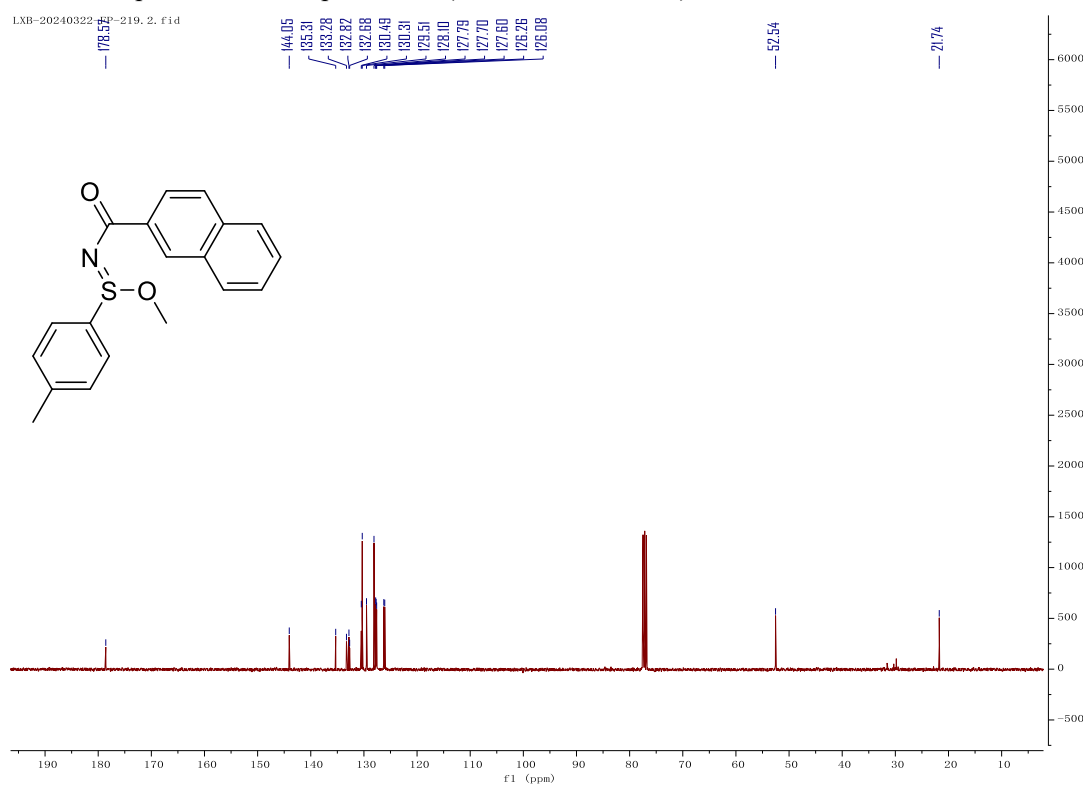
¹H NMR spectrum for compound **3g'** (in CDCl₃, 400 MHz)



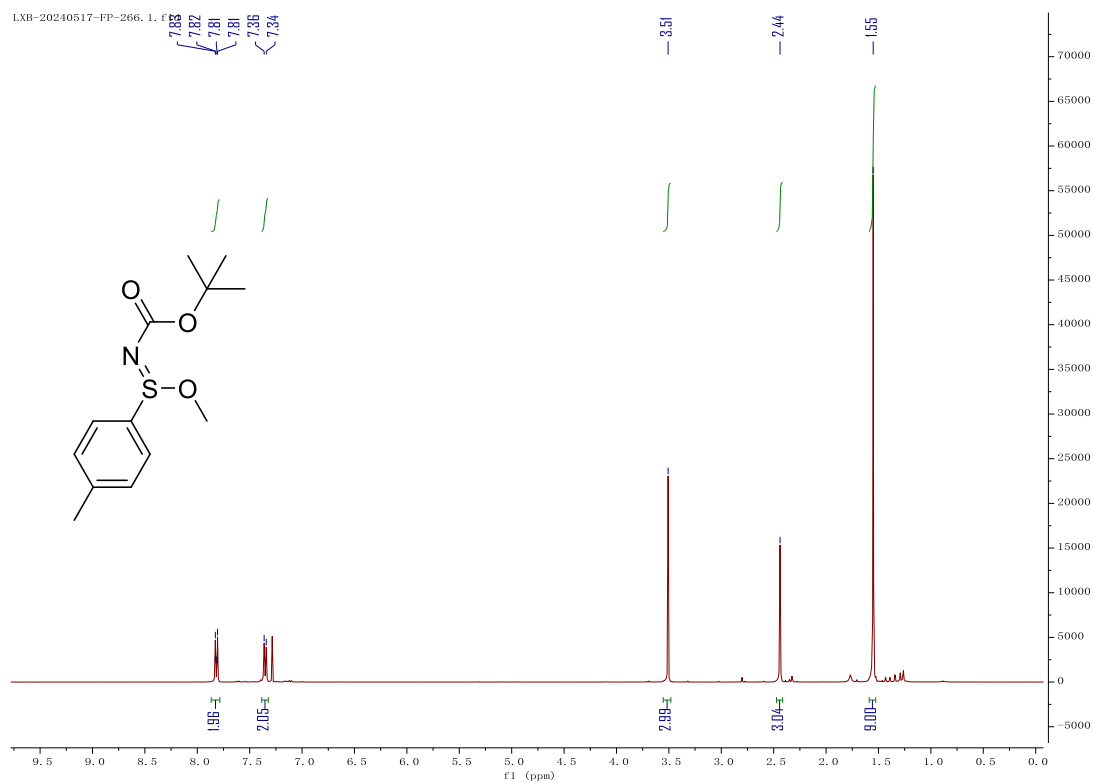
¹H NMR spectrum for compound **3h'** (in CDCl₃, 400 MHz)



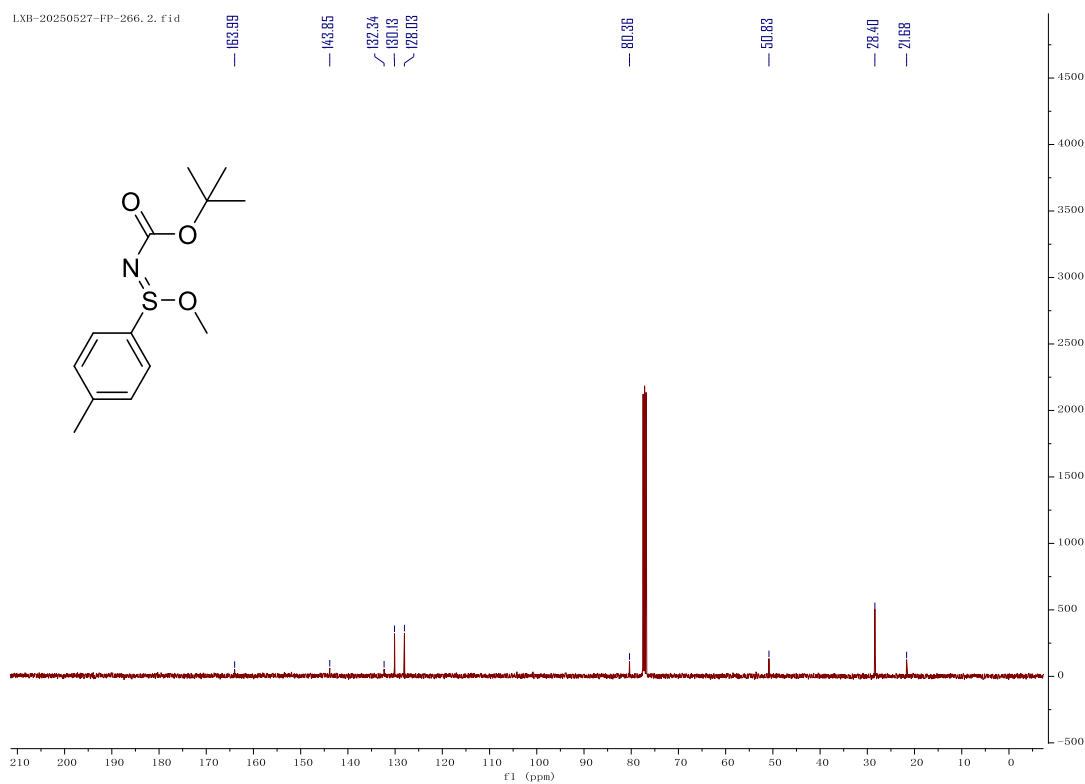
¹³C NMR spectrum for compound **3h'** (in CDCl₃, 101 MHz)



¹H NMR spectrum for compound **3j'** (in CDCl₃, 400 MHz)



¹³C NMR spectrum for compound **3j'** (in CDCl₃, 101 MHz)



Chemical Structure: 4-methyl-2-((benzyloxycarbonyl)imino)phenyl methyl sulfone

¹H NMR Data (CDCl₃):

Chemical Shift (ppm)	Integration
7.78, 7.75, 7.62, 7.48, 7.47, 7.45, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32	1.91, 1.91, 3.69, 0.94
5.28, 5.25, 5.24, 5.21	0.98, 1.00
3.51	2.95
2.45	2.91
1.2	-

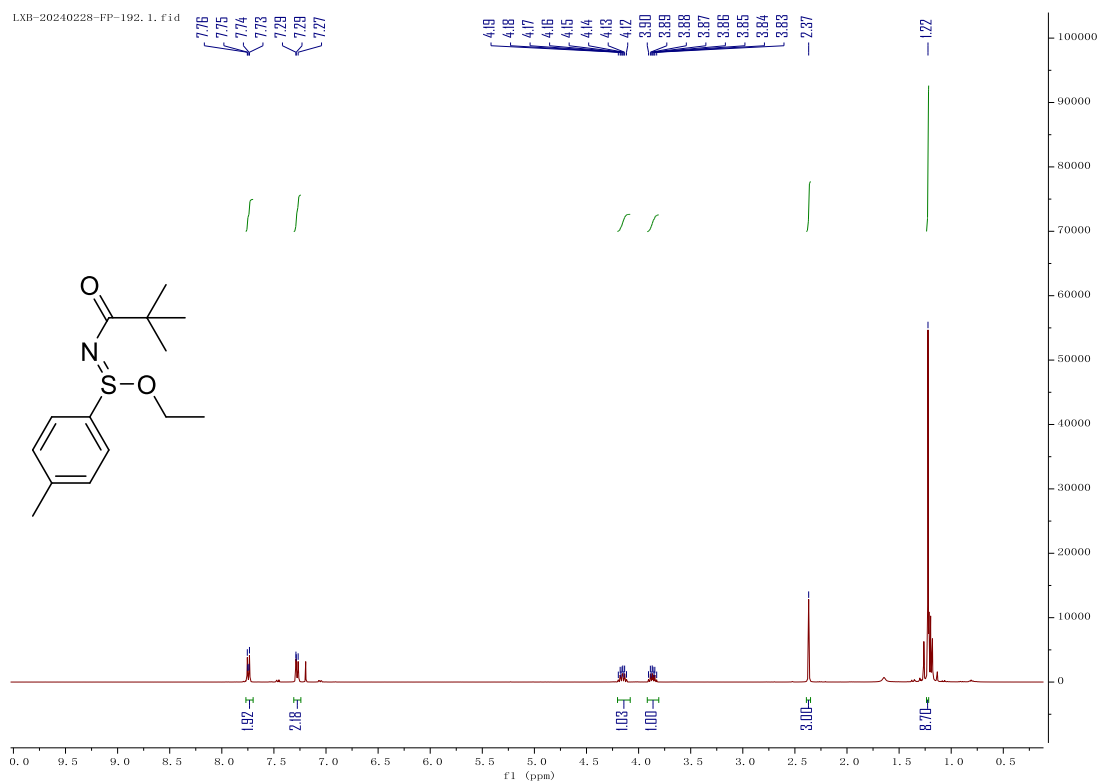
LXB-20250312

Chemical structure: Cc1ccc(cc1)S(=O)(=O)c2nc(C(=O)c3ccncc3)cc2

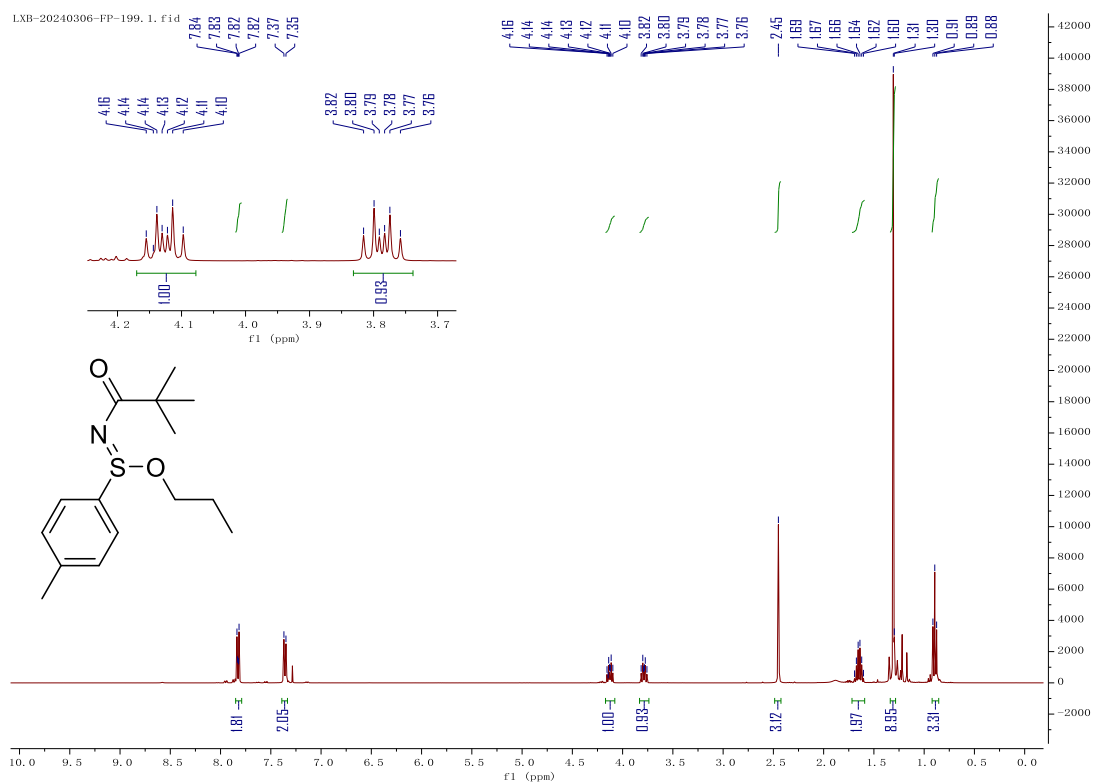
¹H NMR spectrum (ppm):

- 9.544, 9.538, 9.532, 9.526, 9.520, 9.514, 9.508, 9.502, 9.496, 9.490, 9.484, 9.478, 9.472, 9.466, 9.460, 9.454, 9.448, 9.442, 9.436, 9.430, 9.424, 9.418, 9.412, 9.406, 9.400, 9.394, 9.388, 9.382, 9.376, 9.370, 9.364, 9.358, 9.352, 9.346, 9.340, 9.334, 9.328, 9.322, 9.316, 9.310, 9.304, 9.298, 9.292, 9.286, 9.280, 9.274, 9.268, 9.262, 9.256, 9.250, 9.244, 9.238, 9.232, 9.226, 9.220, 9.214, 9.208, 9.202, 9.196, 9.190, 9.184, 9.178, 9.172, 9.166, 9.160, 9.154, 9.148, 9.142, 9.136, 9.130, 9.124, 9.118, 9.112, 9.106, 9.100, 9.094, 9.088, 9.082, 9.076, 9.070, 9.064, 9.058, 9.052, 9.046, 9.040, 9.034, 9.028, 9.022, 9.016, 9.010, 9.004, 9.000, 8.994, 8.988, 8.982, 8.976, 8.970, 8.964, 8.958, 8.952, 8.946, 8.940, 8.934, 8.928, 8.922, 8.916, 8.910, 8.904, 8.900, 8.894, 8.888, 8.882, 8.876, 8.870, 8.864, 8.858, 8.852, 8.846, 8.840, 8.834, 8.828, 8.822, 8.816, 8.810, 8.804, 8.800, 8.794, 8.788, 8.782, 8.776, 8.770, 8.764, 8.758, 8.752, 8.746, 8.740, 8.734, 8.728, 8.722, 8.716, 8.710, 8.704, 8.700, 8.694, 8.688, 8.682, 8.676, 8.670, 8.664, 8.658, 8.652, 8.646, 8.640, 8.634, 8.628, 8.622, 8.616, 8.610, 8.604, 8.600, 8.594, 8.588, 8.582, 8.576, 8.570, 8.564, 8.558, 8.552, 8.546, 8.540, 8.534, 8.528, 8.522, 8.516, 8.510, 8.504, 8.500, 8.494, 8.488, 8.482, 8.476, 8.470, 8.464, 8.458, 8.452, 8.446, 8.440, 8.434, 8.428, 8.422, 8.416, 8.410, 8.404, 8.400, 8.394, 8.388, 8.382, 8.376, 8.370, 8.364, 8.358, 8.352, 8.346, 8.340, 8.334, 8.328, 8.322, 8.316, 8.310, 8.304, 8.300, 8.294, 8.288, 8.282, 8.276, 8.270, 8.264, 8.258, 8.252, 8.246, 8.240, 8.234, 8.228, 8.222, 8.216, 8.210, 8.204, 8.200, 8.194, 8.188, 8.182, 8.176, 8.170, 8.164, 8.158, 8.152, 8.146, 8.140, 8.134, 8.128, 8.122, 8.116, 8.110, 8.104, 8.100, 8.094, 8.088, 8.082, 8.076, 8.070, 8.064, 8.058, 8.052, 8.046, 8.040, 8.034, 8.028, 8.022, 8.016, 8.010, 8.004, 8.000, 7.994, 7.988, 7.982, 7.976, 7.970, 7.964, 7.958, 7.952, 7.946, 7.940, 7.934, 7.928, 7.922, 7.916, 7.910, 7.904, 7.900, 7.894, 7.888, 7.882, 7.876, 7.870, 7.864, 7.858, 7.852, 7.846, 7.840, 7.834, 7.828, 7.822, 7.816, 7.810, 7.804, 7.800, 7.794, 7.788, 7.782, 7.776, 7.770, 7.764, 7.758, 7.752, 7.746, 7.740, 7.734, 7.728, 7.722, 7.716, 7.710, 7.704, 7.700, 7.694, 7.688, 7.682, 7.676, 7.670, 7.664, 7.658, 7.652, 7.646, 7.640, 7.634, 7.628, 7.622, 7.616, 7.610, 7.604, 7.600, 7.594, 7.588, 7.582, 7.576, 7.570, 7.564, 7.558, 7.552, 7.546, 7.540, 7.534, 7.528, 7.522, 7.516, 7.510, 7.504, 7.500, 7.494, 7.488, 7.482, 7.476, 7.470, 7.464, 7.458, 7.452, 7.446, 7.440, 7.434, 7.428, 7.422, 7.416, 7.410, 7.404, 7.400, 7.394, 7.388, 7.382, 7.376, 7.370, 7.364, 7.358, 7.352, 7.346, 7.340, 7.334, 7.328, 7.322, 7.316, 7.310, 7.304, 7.300, 7.294, 7.288, 7.282, 7.276, 7.270, 7.264, 7.258, 7.252, 7.246, 7.240, 7.234, 7.228, 7.222, 7.216, 7.210, 7.204, 7.200, 7.194, 7.188, 7.182, 7.176, 7.170, 7.164, 7.158, 7.152, 7.146, 7.140, 7.134, 7.128, 7.122, 7.116, 7.110, 7.104, 7.100, 7.094, 7.088, 7.082, 7.076, 7.070, 7.064, 7.058, 7.052, 7.046, 7.040, 7.034, 7.028, 7.022, 7.016, 7.010, 7.004, 7.000, 6.994, 6.988, 6.982, 6.976, 6.970, 6.964, 6.958, 6.952, 6.946, 6.940, 6.934, 6.928, 6.922, 6.916, 6.910, 6.904, 6.900, 6.894, 6.888, 6.882, 6.876, 6.870, 6.864, 6.858, 6.852, 6.846, 6.840, 6.834, 6.828, 6.822, 6.816, 6.810, 6.804, 6.800, 6.794, 6.788, 6.782, 6.776, 6.770, 6.764, 6.758, 6.752, 6.746, 6.740, 6.734, 6.728, 6.722, 6.716, 6.710, 6.704, 6.700, 6.694, 6.688, 6.682, 6.676, 6.670, 6.664, 6.658, 6.652, 6.646, 6.640, 6.634, 6.628, 6.622, 6.616, 6.610, 6.604, 6.600, 6.594, 6.588, 6.582, 6.576, 6.570, 6.564, 6.558, 6.552, 6.546, 6.540, 6.534, 6.528, 6.522, 6.516, 6.510, 6.504, 6.500, 6.494, 6.488, 6.482, 6.476, 6.470, 6.464, 6.458, 6.452, 6.446, 6.440, 6.434, 6.428, 6.422, 6.416, 6.410, 6.404, 6.400, 6.394, 6.388, 6.382, 6.376, 6.370, 6.364, 6.358, 6.352, 6.346, 6.340, 6.334, 6.328, 6.322, 6.316, 6.310, 6.304, 6.300, 6.294, 6.288, 6.282, 6.276, 6.270, 6.264, 6.258, 6.252, 6.246, 6.240, 6.234, 6.228, 6

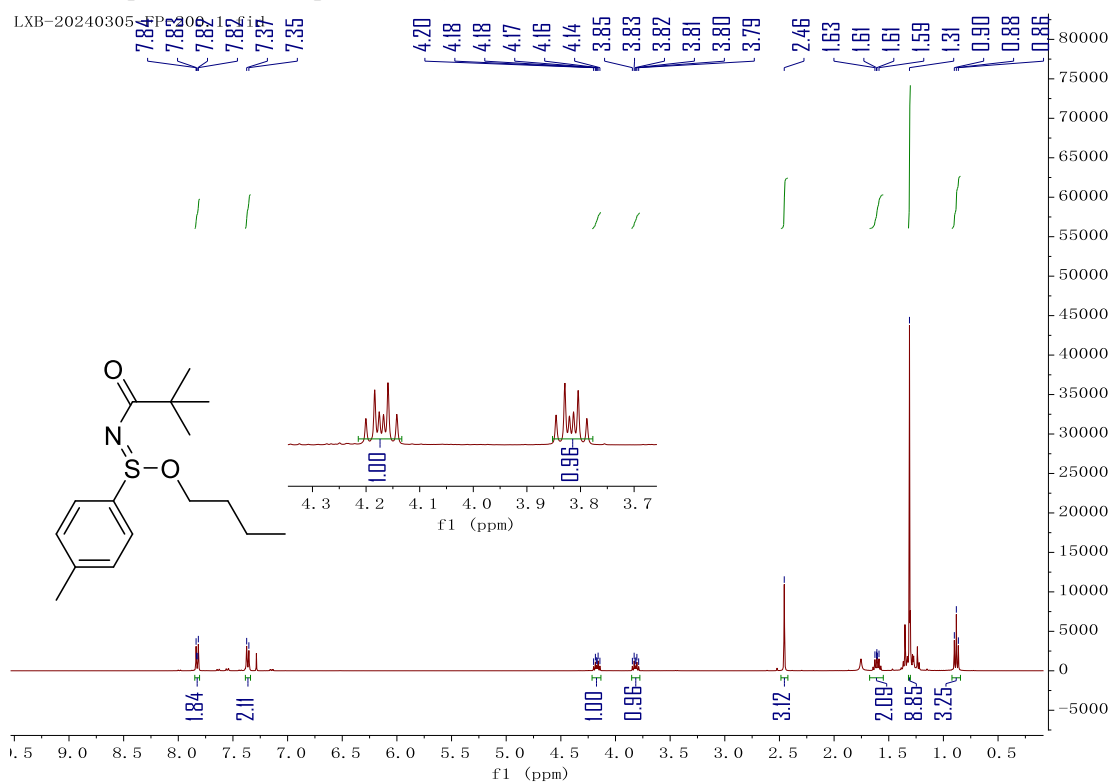
¹H NMR spectrum for compound **3m'** (in CDCl₃, 400 MHz)



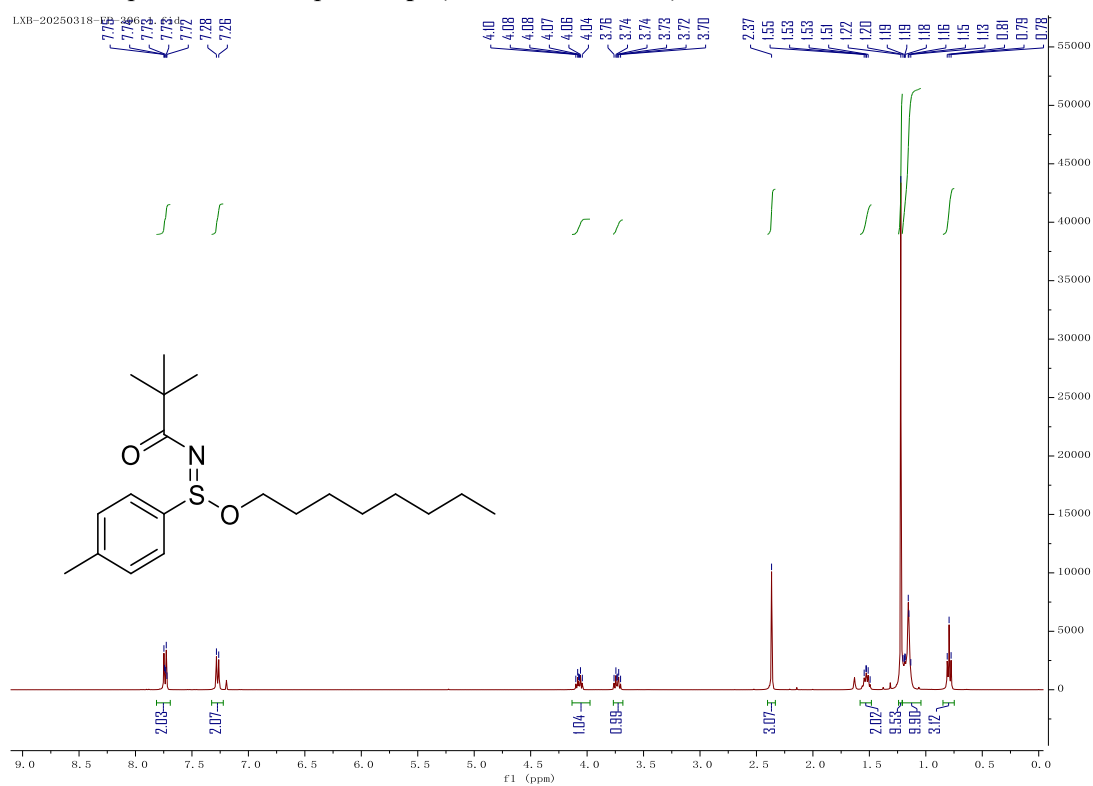
¹H NMR spectrum for compound **3n'** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **3o'** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **3p'** (in CDCl₃, 400 MHz)



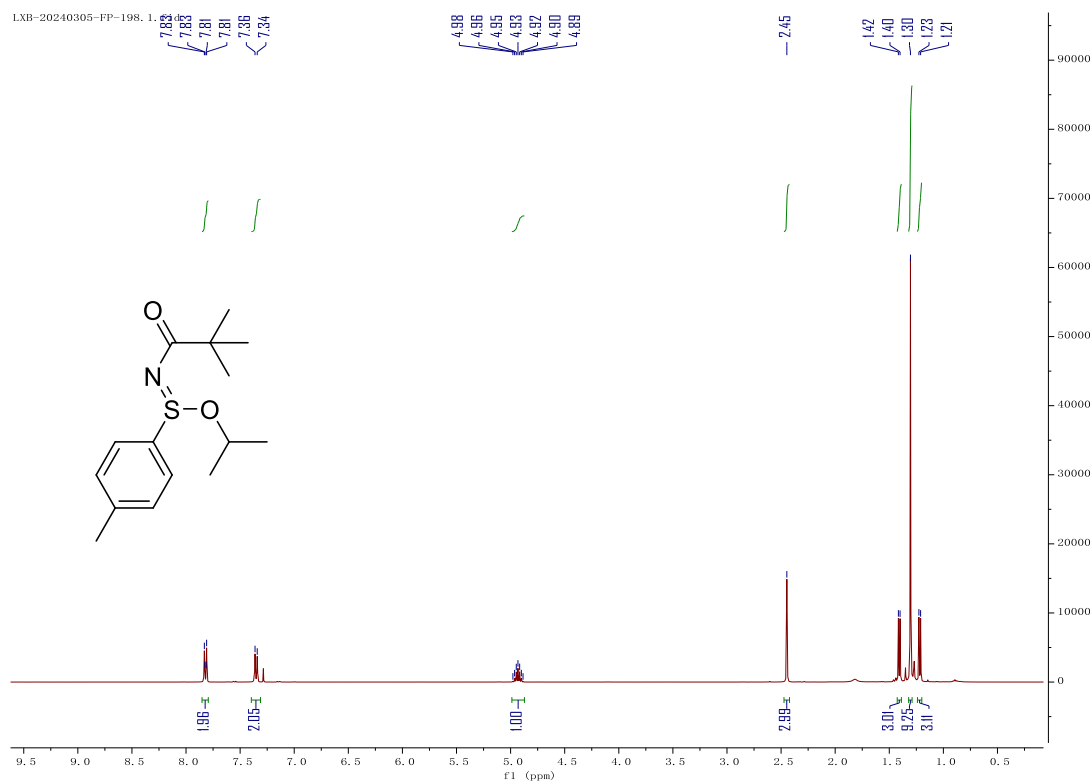
Chemical structure: CC(C)(C)C(=O)N(S(=O)(=O)OCCCC1=CC=CC=C1)C2=CC=C(C)C=C2

¹H NMR spectrum (400 MHz, CDCl₃) showing peaks from 0.5 to 9.5 ppm. The spectrum includes a large peak at 1.22 ppm (9H, t-butyl), a doublet at 1.85 ppm (3H, CH₃), a multiplet at 2.37 ppm (2H, CH₂), a multiplet at 2.53 ppm (2H, CH₂), a multiplet at 3.73 ppm (2H, CH₂), a multiplet at 4.06 ppm (2H, CH₂), and aromatic signals between 7.0 and 7.8 ppm. Integration values are provided for several peaks: 1.90, 2.00, 1.93, 1.12, 1.89, 1.00, 0.88, 1.86, 2.85, 2.00, and 8.87.

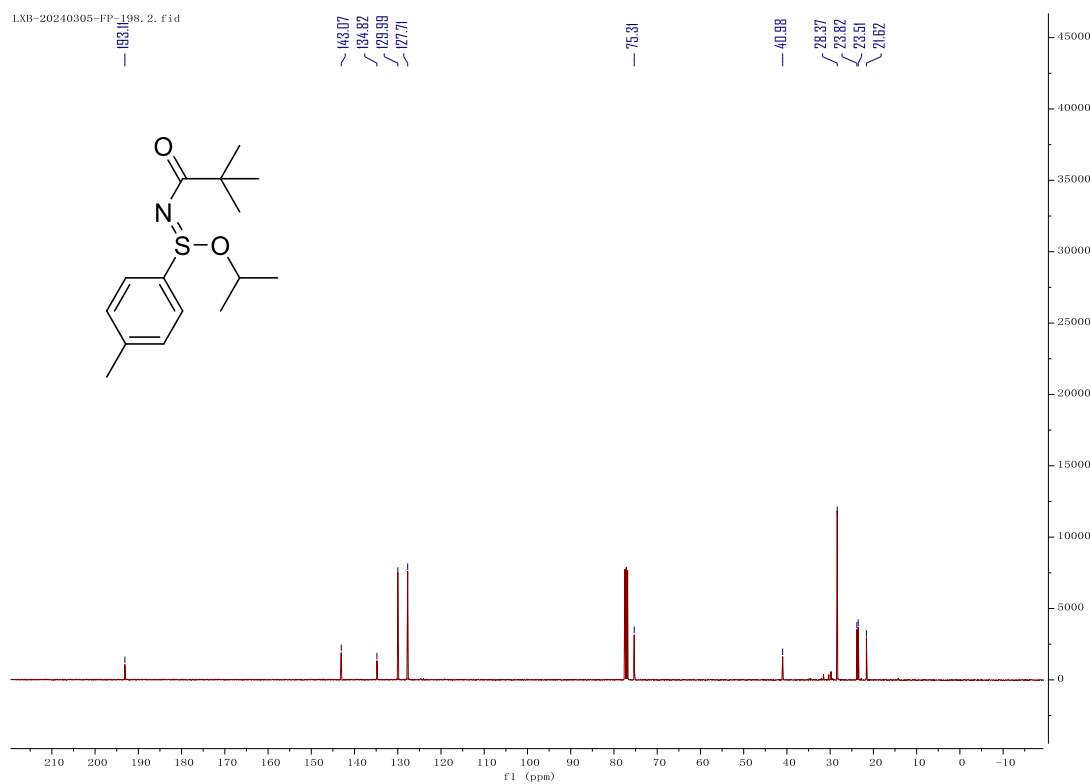
CC(C)(C)C(=O)N=S(c1ccc(C)cc1)OCCc2ccccc2

¹H NMR spectrum (400 MHz, CDCl₃) of 1-(4-methylphenyl)-2-(2-phenylethoxy)propan-1-one oxime. The spectrum shows peaks from 0.5 to 10.0 ppm. Integration values are provided below the peaks: 1.95, 5.57, 2.01, 1.02, 0.89, 1.98, 3.06, and 9.00. A list of chemical shifts (delta) is shown at the top: 7.56, 7.56, 7.55, 7.55, 7.21, 7.20, 7.20, 7.19, 7.18, 7.18, 7.17, 7.16, 7.15, 7.08, 7.07, 7.06, 7.06, 7.06, 4.33, 4.31, 4.31, 4.30, 4.29, 4.27, 3.99, 3.97, 3.96, 3.96, 3.95, 3.93, 2.86, 2.84, 2.83, 2.34, and 1.22.

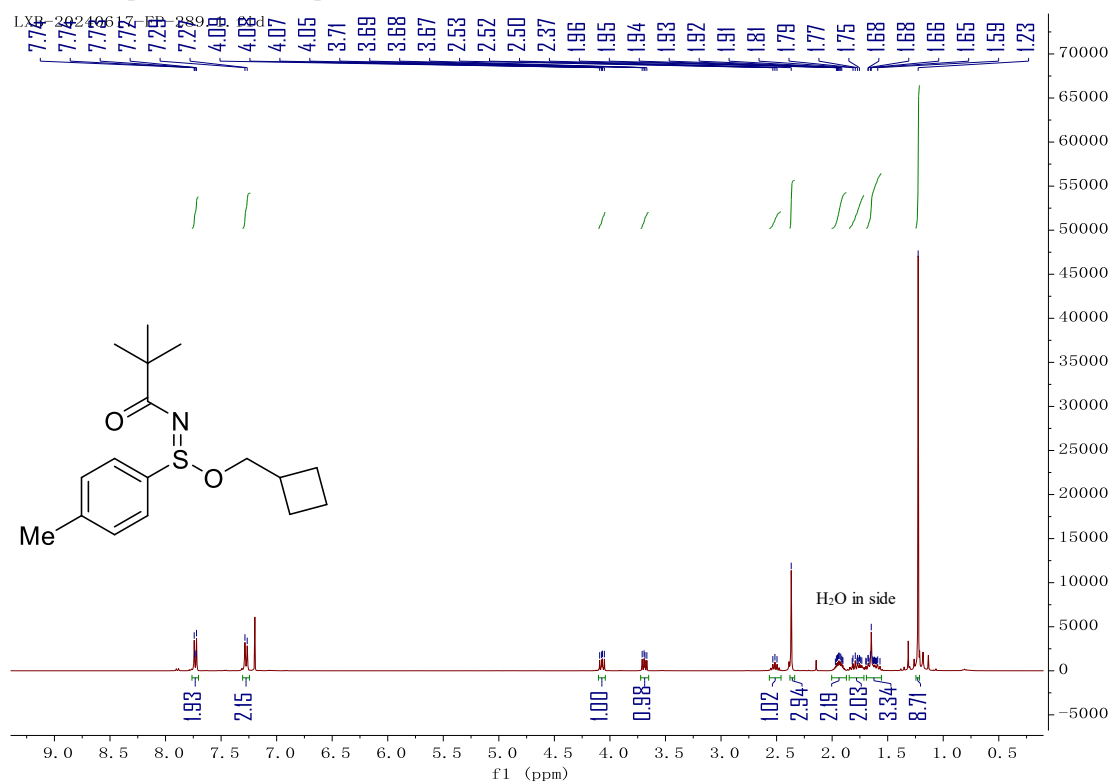
¹H NMR spectrum for compound **3s'** (in CDCl₃, 400 MHz)



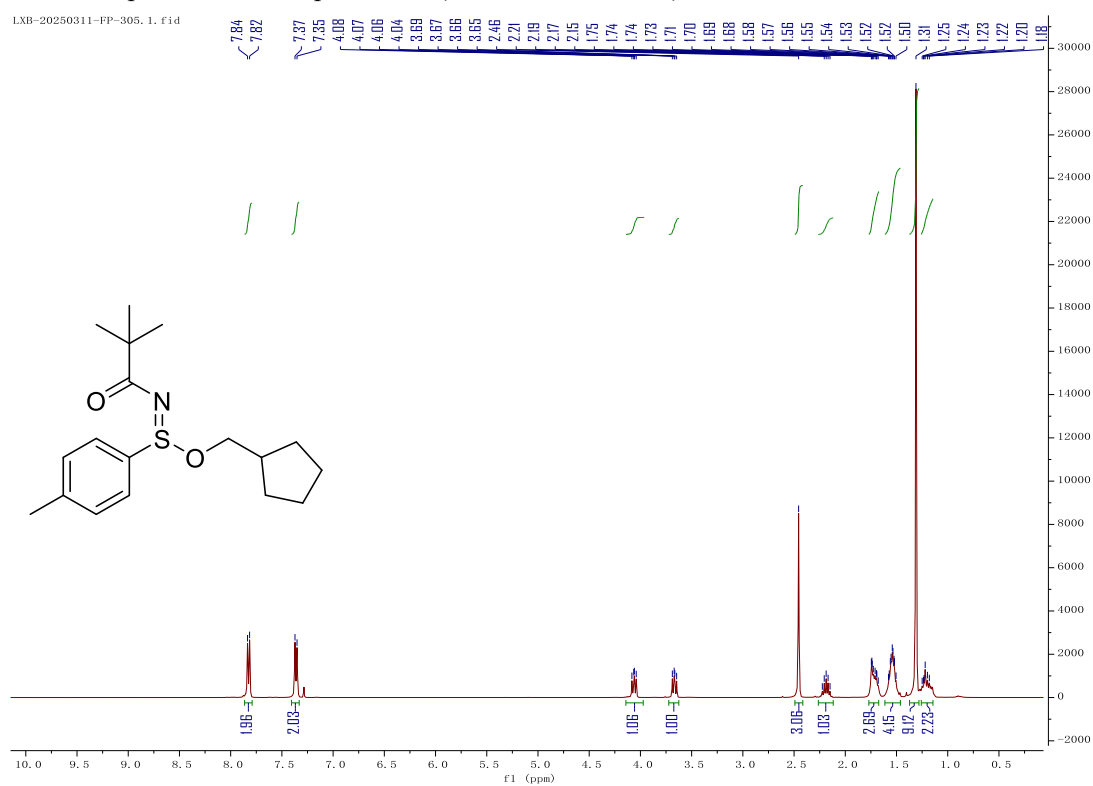
¹³C NMR spectrum for compound **3s'** (in CDCl₃, 101 MHz)



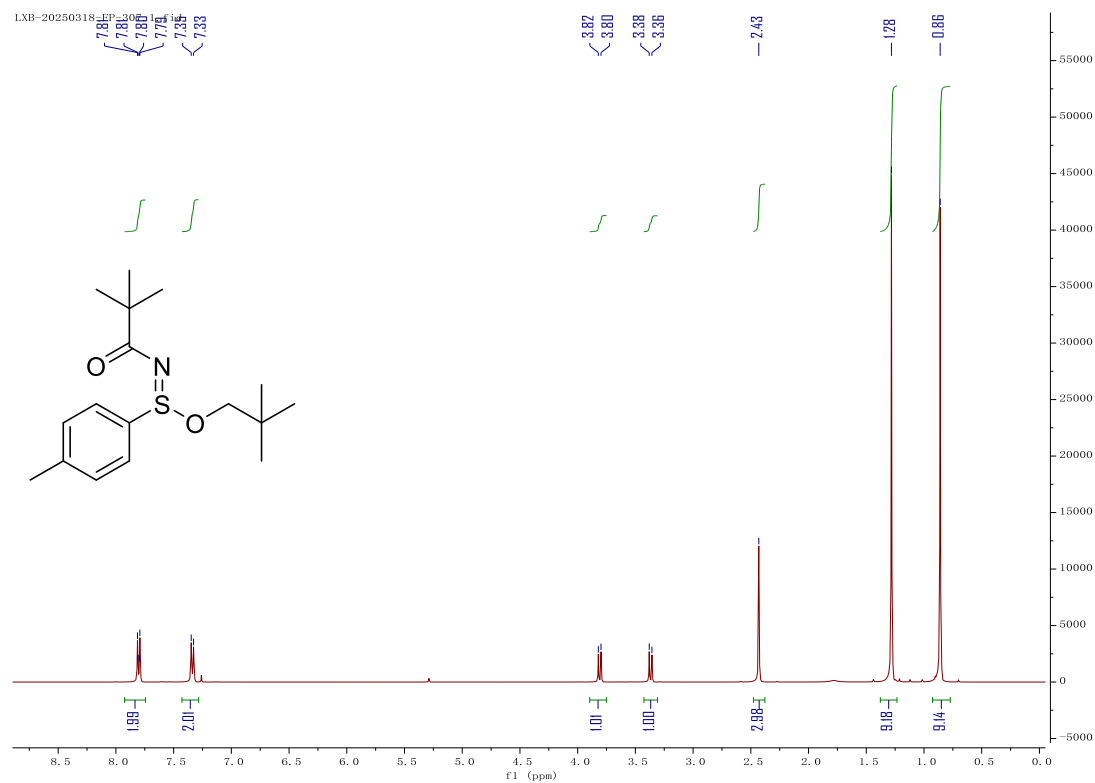
¹H NMR spectrum for compound **3t'** (in CDCl₃, 400 MHz)



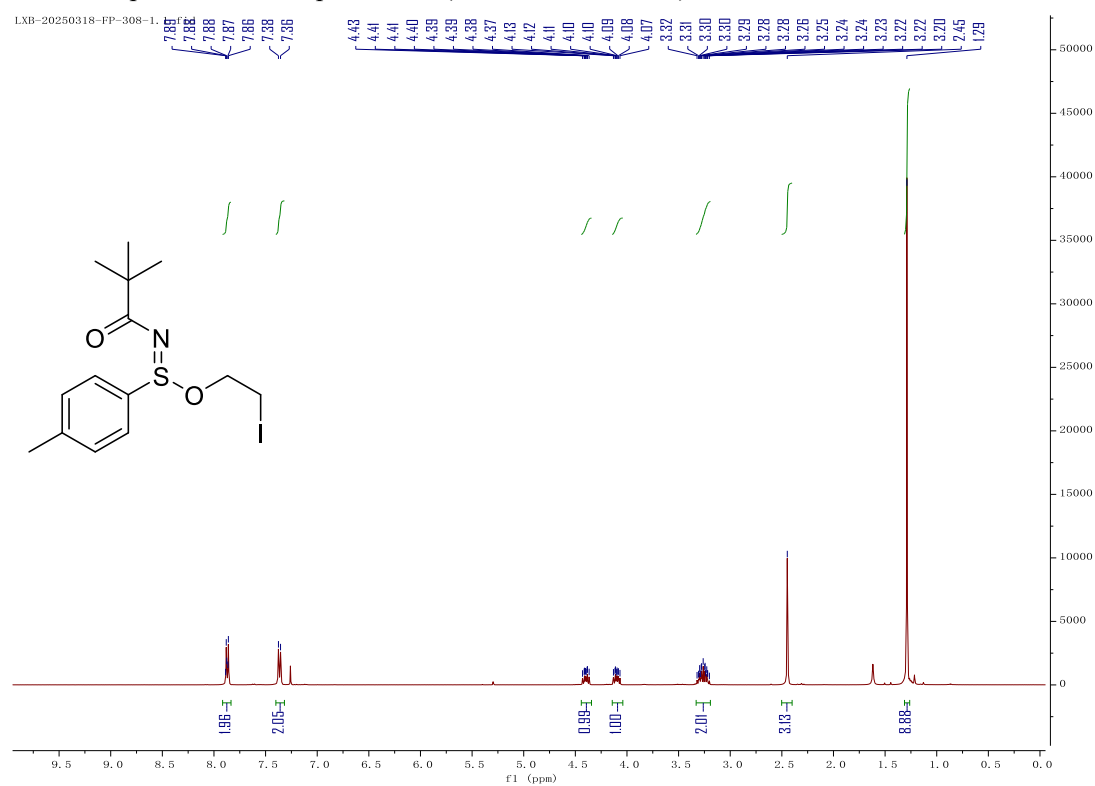
¹H NMR spectrum for compound **3u'** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **3v'** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **3w'** (in CDCl₃, 400 MHz)



Chemical structure: CC(C)(C)C(=O)N=S(OCCCO)c1ccc(C)cc1

¹H NMR spectrum (ppm):

- 7.76, 7.75, 7.74, 7.73, 7.72, 7.71, 7.70, 7.69, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.61, 7.60, 7.59, 7.58, 7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.50, 7.49, 7.48, 7.47, 7.46, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.28, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98, 6.97, 6.96, 6.95, 6.94, 6.93, 6.92, 6.91, 6.90, 6.89, 6.88, 6.87, 6.86, 6.85, 6.84, 6.83, 6.82, 6.81, 6.80, 6.79, 6.78, 6.77, 6.76, 6.75, 6.74, 6.73, 6.72, 6.71, 6.70, 6.69, 6.68, 6.67, 6.66, 6.65, 6.64, 6.63, 6.62, 6.61, 6.60, 6.59, 6.58, 6.57, 6.56, 6.55, 6.54, 6.53, 6.52, 6.51, 6.50, 6.49, 6.48, 6.47, 6.46, 6.45, 6.44, 6.43, 6.42, 6.41, 6.40, 6.39, 6.38, 6.37, 6.36, 6.35, 6.34, 6.33, 6.32, 6.31, 6.30, 6.29, 6.28, 6.27, 6.26, 6.25, 6.24, 6.23, 6.22, 6.21, 6.20, 6.19, 6.18, 6.17, 6.16, 6.15, 6.14, 6.13, 6.12, 6.11, 6.10, 6.09, 6.08, 6.07, 6.06, 6.05, 6.04, 6.03, 6.02, 6.01, 6.00, 5.99, 5.98, 5.97, 5.96, 5.95, 5.94, 5.93, 5.92, 5.91, 5.90, 5.89, 5.88, 5.87, 5.86, 5.85, 5.84, 5.83, 5.82, 5.81, 5.80, 5.79, 5.78, 5.77, 5.76, 5.75, 5.74, 5.73, 5.72, 5.71, 5.70, 5.69, 5.68, 5.67, 5.66, 5.65, 5.64, 5.63, 5.62, 5.61, 5.60, 5.59, 5.58, 5.57, 5.56, 5.55, 5.54, 5.53, 5.52, 5.51, 5.50, 5.49, 5.48, 5.47, 5.46, 5.45, 5.44, 5.43, 5.42, 5.41, 5.40, 5.39, 5.38, 5.37, 5.36, 5.35, 5.34, 5.33, 5.32, 5.31, 5.30, 5.29, 5.28, 5.27, 5.26, 5.25, 5.24, 5.23, 5.22, 5.21, 5.20, 5.19, 5.18, 5.17, 5.16, 5.15, 5.14, 5.13, 5.12, 5.11, 5.10, 5.09, 5.08, 5.07, 5.06, 5.05, 5.04, 5.03, 5.02, 5.01, 5.00, 4.99, 4.98, 4.97, 4.96, 4.95, 4.94, 4.93, 4.92, 4.91, 4.90, 4.89, 4.88, 4.87, 4.86, 4.85, 4.84, 4.83, 4.82, 4.81, 4.80, 4.79, 4.78, 4.77, 4.76, 4.75, 4.74, 4.73, 4.72, 4.71, 4.70, 4.69, 4.68, 4.67, 4.66, 4.65, 4.64, 4.63, 4.62, 4.61, 4.60, 4.59, 4.58, 4.57, 4.56, 4.55, 4.54, 4.53, 4.52, 4.51, 4.50, 4.49, 4.48, 4.47, 4.46, 4.45, 4.44, 4.43, 4.42, 4.41, 4.40, 4.39, 4.38, 4.37, 4.36, 4.35, 4.34, 4.33, 4.32, 4.31, 4.30, 4.29, 4.28, 4.27, 4.26, 4.25, 4.24, 4.23, 4.22, 4.21, 4.20, 4.19, 4.18, 4.17, 4.16, 4.15, 4.14, 4.13, 4.12, 4.11, 4.10, 4.09, 4.08, 4.07, 4.06, 4.05, 4.04, 4.03, 4.02, 4.01, 4.00, 3.99, 3.98, 3.97, 3.96, 3.95, 3.94, 3.93, 3.92, 3.91, 3.90, 3.89, 3.88, 3.87, 3.86, 3.85, 3.84, 3.83, 3.82, 3.81, 3.80, 3.79, 3.78, 3.77, 3.76, 3.75, 3.74, 3.73, 3.72, 3.71, 3.70, 3.69, 3.68, 3.67, 3.66, 3.65, 3.64, 3.63, 3.62, 3.61, 3.60, 3.59, 3.58, 3.57, 3.56, 3.55, 3.54, 3.53, 3.52, 3.51, 3.50, 3.49, 3.48, 3.47, 3.46, 3.45, 3.44, 3.43, 3.42, 3.41, 3.40, 3.39, 3.38, 3.37, 3.36, 3.35, 3.34, 3.33, 3.32, 3.31, 3.30, 3.29, 3.28, 3.27, 3.26, 3.25, 3.24, 3.23, 3.22, 3.21, 3.20, 3.19, 3.18, 3.17, 3.16, 3.15, 3.14, 3.13, 3.12, 3.11, 3.10, 3.09, 3.08, 3.07, 3.06, 3.05, 3.04, 3.03, 3.02, 3.01, 3.00, 2.99, 2.98, 2.97, 2.96, 2.95, 2.94, 2.93, 2.92, 2.91, 2.90, 2.89, 2.88, 2.87, 2.86, 2.85, 2.84, 2.83, 2.82, 2.81, 2.80, 2.79, 2.78, 2.77, 2.76, 2.75, 2.74, 2.73, 2.72, 2.71, 2.70, 2.69, 2.68, 2.67, 2.66, 2.65, 2.64, 2.63, 2.62, 2.61, 2.60, 2.59, 2.58, 2.57, 2.56, 2.55, 2.54, 2.53, 2.52, 2.51, 2.50, 2.49, 2.48, 2.47, 2.46, 2.45, 2.44, 2.43, 2.42, 2.41, 2.40, 2.39, 2.38, 2.37, 2.36, 2.35, 2.34, 2.33, 2.32, 2.31, 2.30, 2.29, 2.28, 2.27, 2.26, 2.25, 2.24, 2.23, 2.22, 2.21, 2.20, 2.19, 2.18, 2.17, 2.16, 2.15, 2.14, 2.13, 2.12, 2.11, 2.10, 2.09, 2.08, 2.07, 2.06, 2.05, 2.04, 2.03, 2.02, 2.01, 2.00, 1.99, 1.98, 1.97, 1.96, 1.95, 1.94, 1.93, 1.92, 1.91, 1.90, 1.89, 1.88, 1.87, 1.86, 1.85, 1.84, 1.83, 1.82, 1.81, 1.80, 1.79, 1.78, 1.77, 1.76, 1.75, 1.74, 1.73, 1.72, 1.71, 1.70, 1.69, 1.68, 1.67, 1.66, 1.65, 1.64, 1.63, 1.62, 1.61, 1.60, 1.59, 1.58, 1.57, 1.56, 1.55, 1.54, 1.53, 1.52, 1.51, 1.50, 1.49, 1.48, 1.47, 1.46, 1.45, 1.44, 1.43, 1.42, 1.41, 1.40, 1

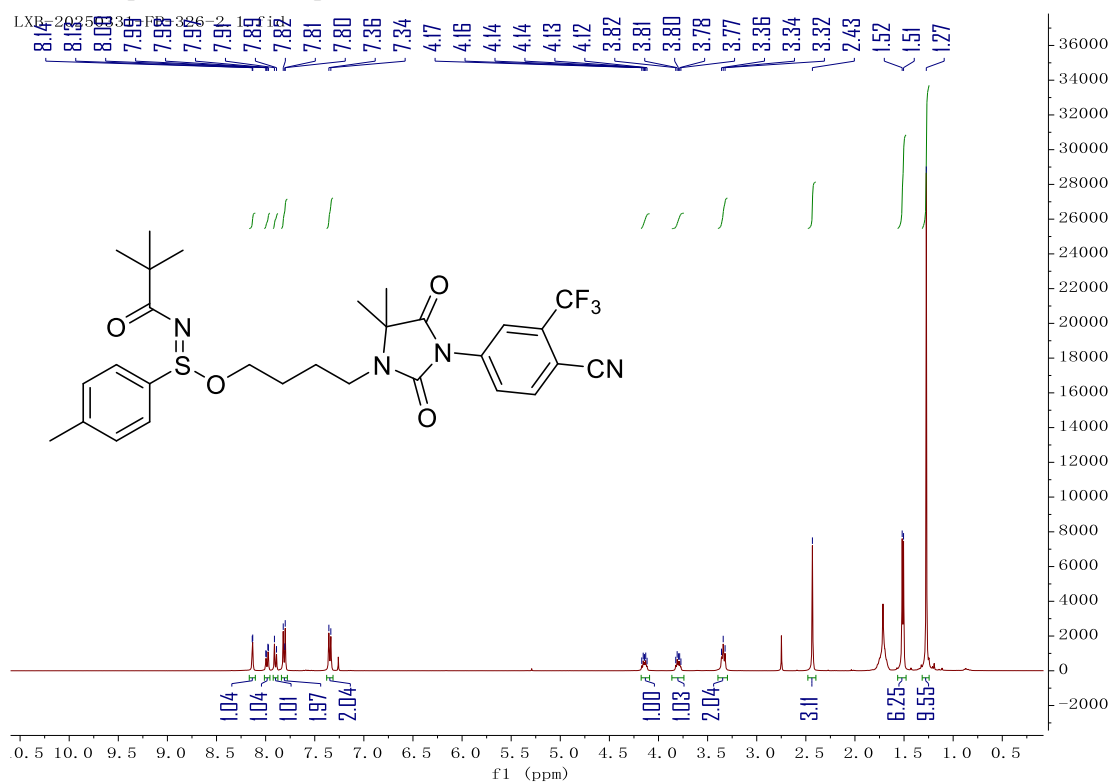
LXB-20250325-FFA-20-2. 2. f1d

Chemical structure: CC1=CC=C(C=C1)C(=O)N(SOCCCCO)C(C)(C)C

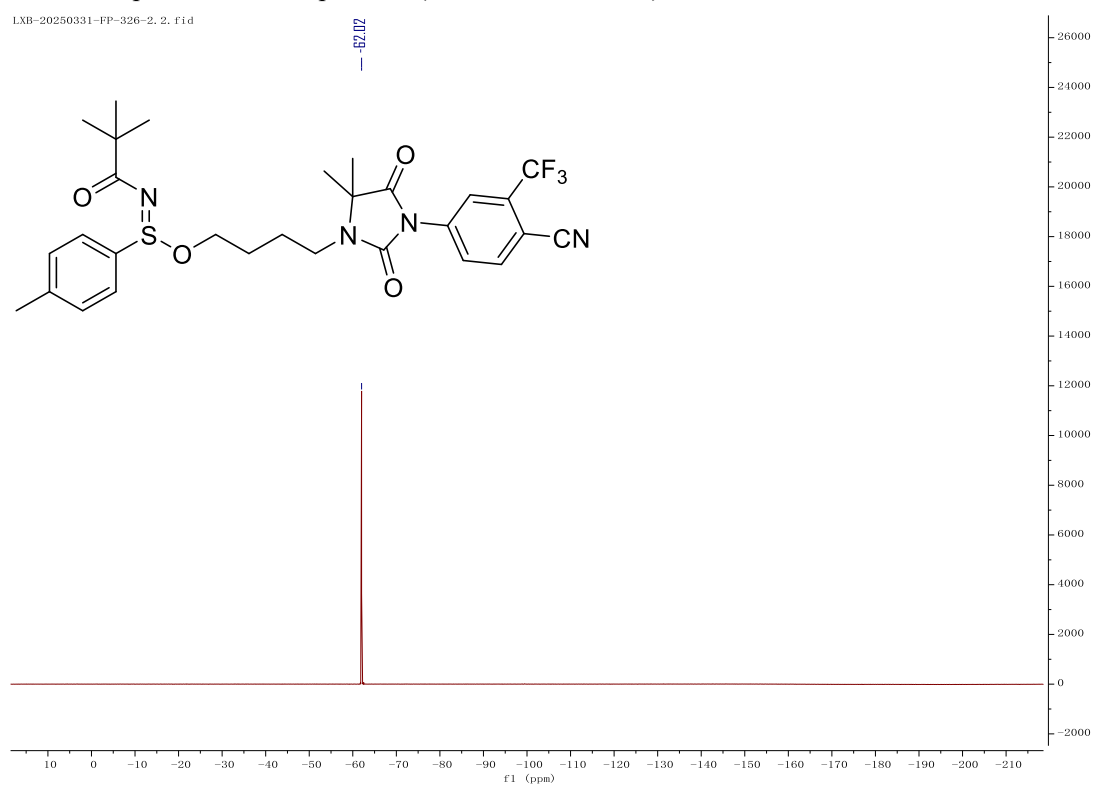
¹H NMR spectrum (ppm):

- 7.27
- 7.19
- 6.83
- 6.77
- 6.32
- 6.20
- 3.77
- 3.60
- 3.27
- 3.19
- 2.19
- 0.00

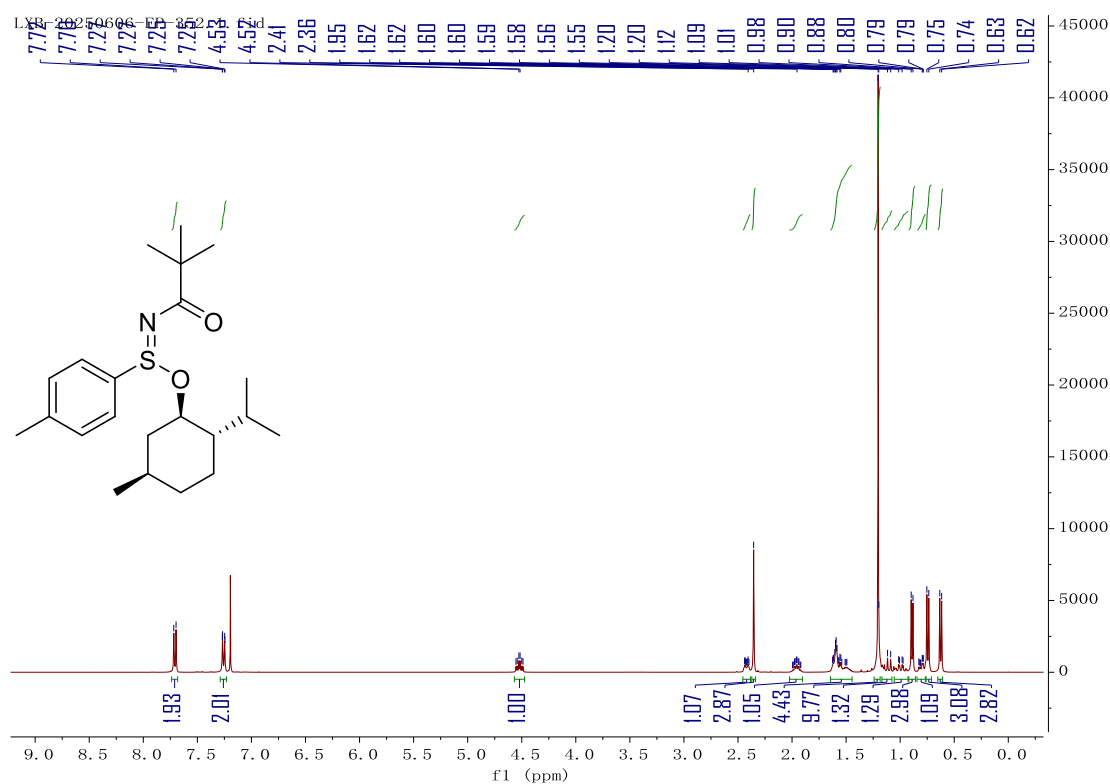
¹H NMR spectrum for compound **4** (in CDCl₃, 400 MHz)



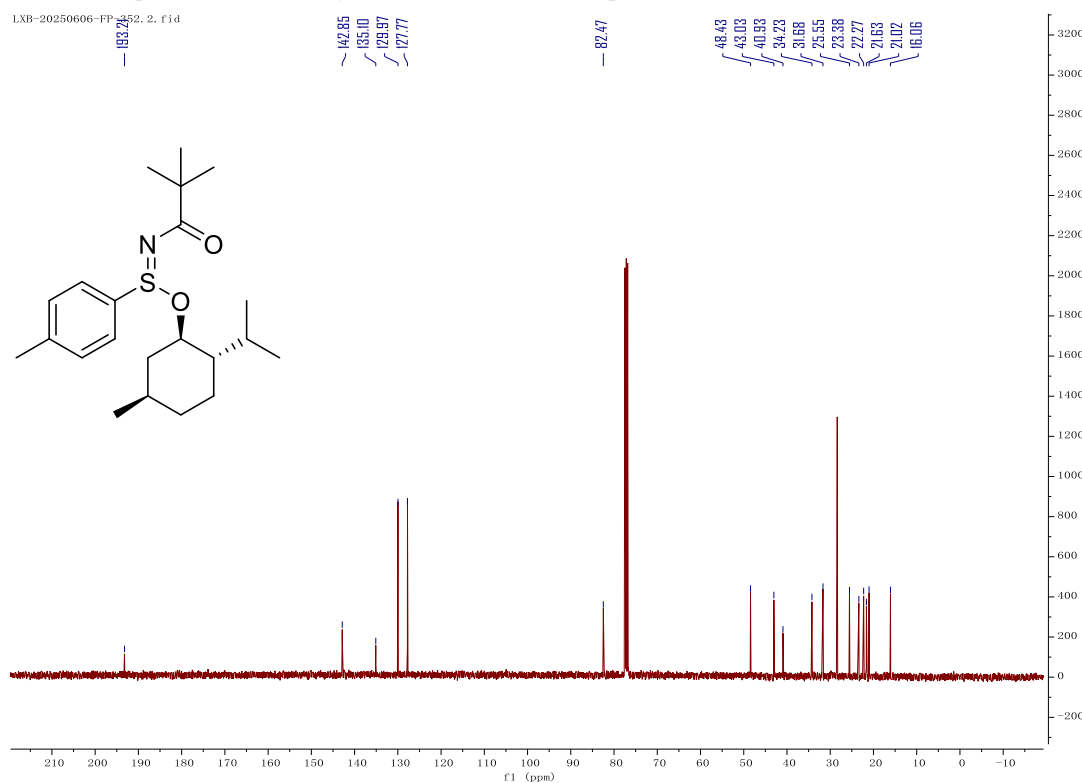
¹⁹F NMR spectrum for compound **4** (in CDCl₃, 376 MHz)



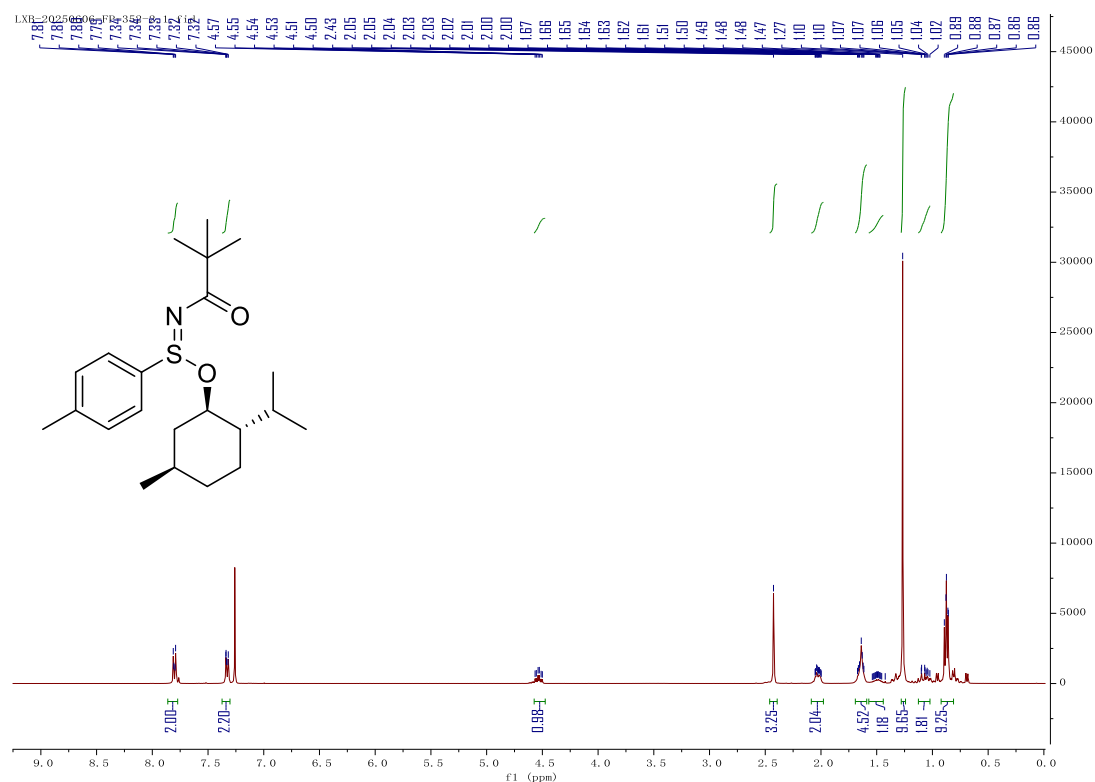
^1H NMR spectrum of the major diastereomer of compound **5** (in CDCl_3 , 400 MHz).



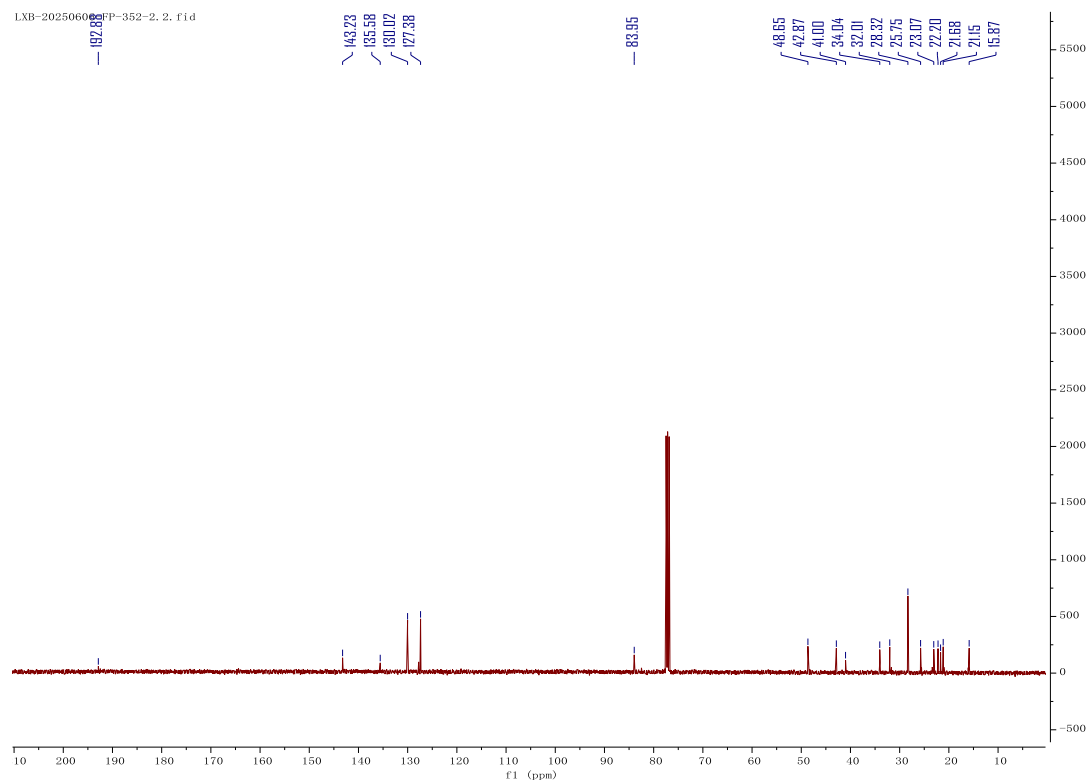
^{13}C NMR spectrum of the major diastereomer of compound **5** (in CDCl_3 , 101 MHz)



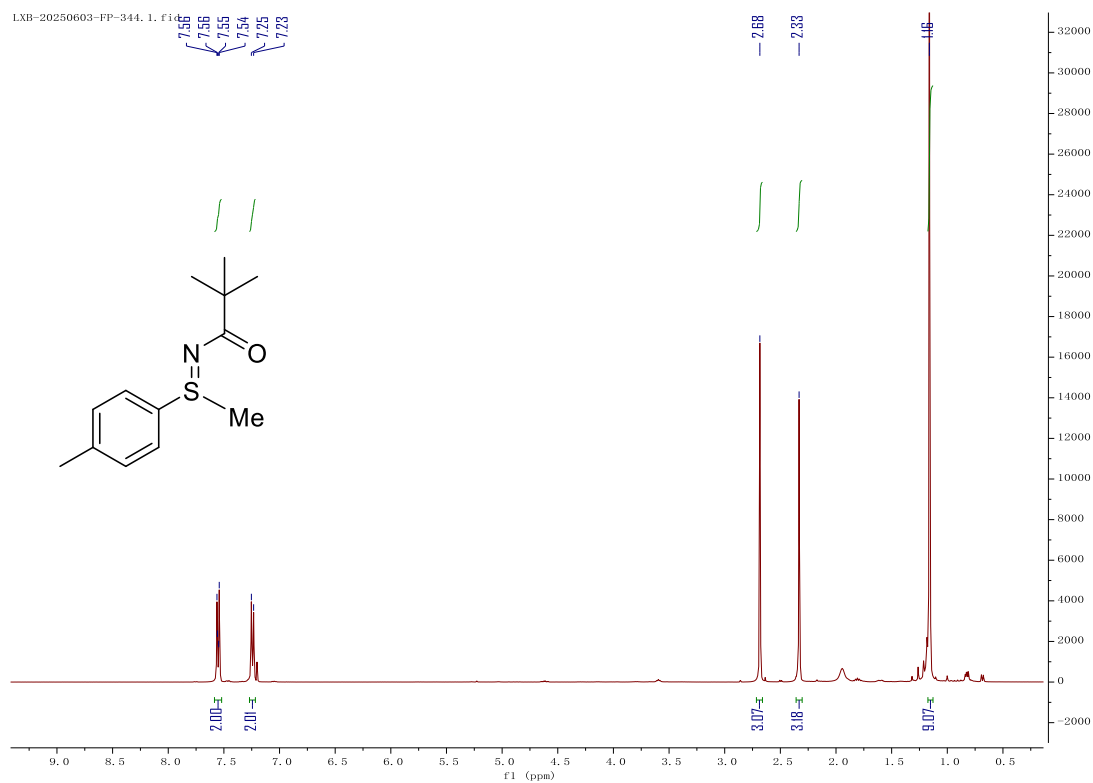
^1H NMR spectrum of the minor diastereomer of compound **5** (in CDCl_3 , 400 MHz).



^{13}C NMR spectrum of the minor diastereomer of compound **5** (in CDCl_3 , 101 MHz)



¹H NMR spectrum for compound **6a** (in CDCl₃, 400 MHz)



¹H NMR spectrum for compound **6b** (in CDCl₃, 400 MHz)

