

Facile Synthesis of α -Sulfonyl Ketoximes from Alkenes Using Sodium Sulfite and NaNO_2 in Water

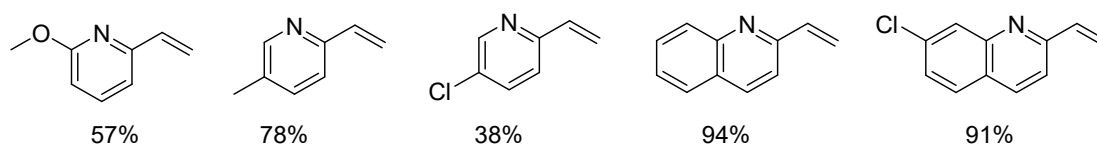
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Experimental Section

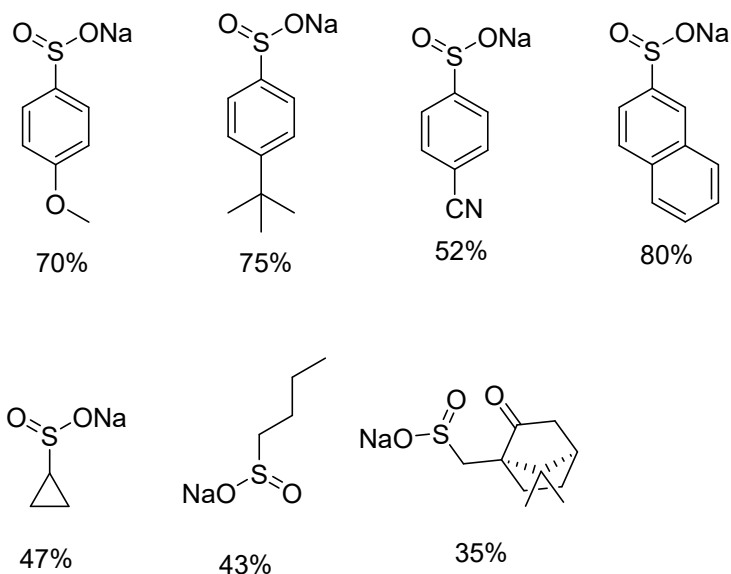
General Information: Commercially available reagents were used as received. Solvents were commercially available and used without further purification. Column chromatographic purifications of the compounds were performed using silica gel (mesh 200–300) and petroleum ether –ethyl acetate mixtures as eluent unless otherwise specified. NMR spectra were recorded on a 400 MHz instrument at 25 °C. The chemical shift values are reported in parts per million (ppm) with respect to residual trichloromethane (7.26 ppm for ^1H and 77.16 ppm for ^{13}C). The peak patterns are designated as follows: s: singlet; d: doublet; t: triplet; q: quartet; m: multiplet; dd: doublet of doublets; td: triplet of doublets; br s: broad singlet. The coupling constants (J) are reported in hertz (Hz). All melting points were taken on a WRX-4 melting point apparatus (Shanghai Yice) and were uncorrected.

Typical Procedure for the Synthesis of 1¹¹: vinyl pyridine was prepared by heating $\text{Ph}_3\text{PCH}_2\text{I}$ (5 mmol, 1010 mg), pyridine aldehyde (4 mmol) and K_2CO_3 (5 mmol), in 10 mL of 1,4-dioxane at 100 °C for 2 h. After the reaction finished, the reaction mixture was diluted with H_2O (25 mL) and extracted with ethyl acetate (3 × 25 mL). The organic extracts were dried over anhydrous Na_2SO_4 . After filtration and evaporation of the solvents under reduced pressure, the crude product was purified by column chromatography on silica gel to afford desired product.



Typical Procedure for the Synthesis of sodium 4-methoxybenzenesulfinate²¹: Sodium 4-methoxybenzenesulfinate was prepared by heating 2.08 g of sodium hydrogen sulfite, 2.05 g of 4-methoxybenzenesulfonyl chloride, and 1.68 g of sodium bicarbonate in 20 mL of water at 100 °C for 12 h. After cooling to room temperature, water was removed under vacuum. Recrystallization of the residue in ethanol produced a white solid; yield: 1.36 g (70%).

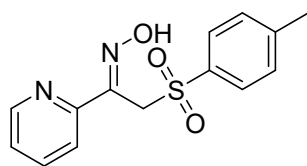
Similarly, other sodium arylsulfonates were prepared from their corresponding sulfonyl chlorides.



General procedure for preparation synthesis of Compounds 3 and 5: alkene (**1** or **4**, 1 mmol), Sodium sulfinate **2** (1.5 mmol), NaNO_2 (4 mmol) and HBF_4 (2 mmol) were added in H_2O (5 mL) was stirred at $80\text{ }^\circ\text{C}$ under air for 1 h. After the reaction finished, the reaction mixture was diluted with H_2O (10 mL) and extracted with ethyl acetate ($3 \times 15\text{ mL}$). The organic extracts were dried over anhydrous Na_2SO_4 . After filtration and evaporation of the solvents under reduced pressure, the crude product was purified by column chromatography on silica gel to afford desired product.

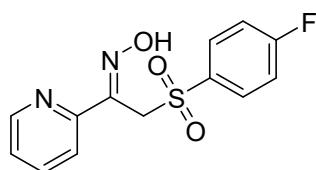
Gram-scale Experimental Procedure for Compound 3a and 5a: 2-vinylpyridine **1a** or styrene **4a** (10 mmol), sodium p-tolylsulfinate **1** (15 mmol), NaNO_2 (40 mmol) and HBF_4 (20 mmol) were added in H_2O (50 mL) was stirred at $80\text{ }^\circ\text{C}$ under air for 2 h. After the reaction finished, the reaction mixture was diluted with H_2O (50 mL) and extracted with ethyl acetate ($3 \times 100\text{ mL}$). The organic extracts were dried over anhydrous Na_2SO_4 . After filtration and evaporation of the solvents under reduced pressure, the crude product was purified by column chromatography on silica gel to afford desired product. A comparable yield could also be obtained by the following treatment method: first, the reaction mixture was cooled and the product was precipitated, followed by filtration and washing with petroleum ether and ethyl acetate (30 ml, 3:1) to obtain the pure product.

Characterization of synthesized compounds:



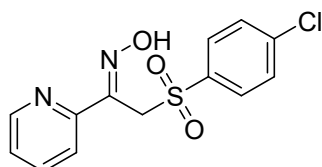
(Z)-1-(pyridin-2-yl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3a** (270 mg; yield 93%), white solid; m.p.174-175 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.16 (s, 1H), 8.39 (dt, *J*=4.8 Hz, 1.4 Hz, 1H), 8.00 – 7.68 (m, 2H), 7.70 – 7.50 (m, 2H), 7.48 – 7.21 (m, 3H), 4.97 (s, 2H), 2.35 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 152.20, 148.53, 146.88, 144.29, 137.61, 136.89, 129.52, 127.86, 123.87, 120.21, 50.70, 21.15. HRMS calc. C₁₄H₁₄N₂O₃S (M+Na⁺): 313.0617, found: 313.0623.



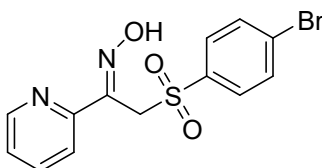
(Z)-2-((4-fluorophenyl)sulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3b** (276 mg; yield 94%), white solid; m.p.127-128 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.18 (s, 1H), 8.40 (dt, *J*=4.8 Hz, 1.4 Hz, 1H), 7.97 – 7.64 (m, 4H), 7.64 – 7.18 (m, 3H), 5.02 (s, 2H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 166.71, 164.20, 152.39, 148.89, 147.03, 137.18 (d, *J*_{C-F} = 27.0 Hz), 137.01, 131.58 (d, *J*_{C-F} = 9.0 Hz), 122.44 (d, *J*_{C-F} = 381.0 Hz), 116.62 (d, *J*_{C-F} = 23.0 Hz), 50.98. HRMS calc. C₁₃H₁₁FN₂O₃S (M+Na⁺): 317.0372, found: 317.0378.



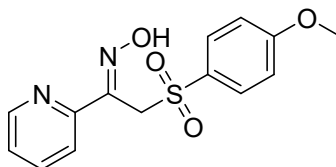
(Z)-2-((4-chlorophenyl)sulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3c** (254 mg; yield 82%), white solid; m.p.155-156 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.21 (s, 1H), 8.41 (dt, *J*=4.8 Hz, 1.4 Hz, 1H), 7.88 – 7.69 (m, 4H), 7.67 – 7.51 (m, 2H), 7.34 (q, *J*=4.5 Hz, 1H), 5.05 (s, 2H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 151.94, 148.46, 146.54, 139.10, 138.87, 136.89, 129.90, 129.14, 123.91, 120.12, 50.52. C₁₃H₁₁ClN₂O₃S (M+Na⁺): 333.0077, found: 333.0080.



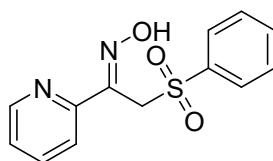
(Z)-2-((4-bromophenyl)sulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3d** (336 mg; yield 95%), white solid; m.p.168-169 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.22 (s, 1H), 8.41 (dt, *J*=4.9 Hz, 1.5 Hz, 1H), 7.83 – 7.71 (m, 4H), 7.72 – 7.62 (m, 2H), 7.35 (ddd, *J*=6.3 Hz, 4.9 Hz, 2.3 Hz, 1H), 5.03 (s, 2H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 151.95, 148.52, 146.57, 139.54, 136.97, 132.15, 129.98, 128.04, 123.97, 120.17, 50.54. C₁₃H₁₁BrN₂O₃S (M+H⁺): 354.9752,356.9732, found: 354.9755,356.9734.



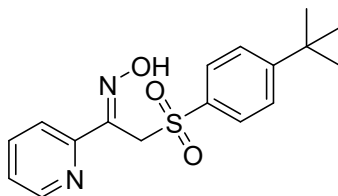
(Z)-2-((4-methoxyphenyl)sulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3e** (208 mg; yield 68%), white solid; m.p.149-150 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.13 (s, 1H), 8.54 – 8.28 (m, 1H), 7.85 – 7.72 (m, 2H), 7.70 – 7.57 (m, 2H), 7.33 (ddd, *J*=6.8 Hz, 4.9 Hz, 2.1 Hz, 1H), 7.09 – 6.94 (m, 2H), 4.97 (s, 2H), 3.82 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 163.20, 152.21, 148.49, 146.92, 136.82, 131.90, 130.13, 123.81, 120.17, 114.17, 55.77, 50.75. C₁₄H₁₄N₂O₄S (M+Na⁺): 329.0572, found: 329.0576.



(Z)-2-(phenylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

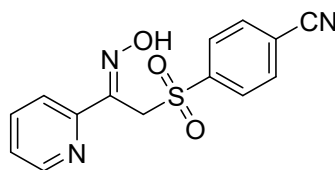
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3f** (262 mg; yield 95%), white solid; m.p.178-179 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.18 (s, 1H), 8.39 (d, *J*=4.9 Hz, 1H), 7.87 – 7.71 (m, 4H), 7.67 (t, *J*=7.4 Hz, 1H), 7.54 (t, *J*=7.7 Hz, 2H), 7.33 (td, *J*=5.2 Hz, 3.1 Hz, 1H), 5.02 (s, 2H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 152.06, 148.45, 146.68, 140.38, 136.84, 133.72, 129.01, 127.76, 123.88, 120.07, 50.52. C₁₃H₁₂N₂O₃S (M+Na⁺): 299.0466, found: 299.0468.



(Z)-2-((4-*tert*-butyl)phenyl)sulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

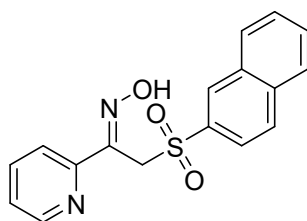
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3g** (156 mg; yield 47%), white solid; m.p.182-183 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.22 (s, 1H), 8.30 (dt, *J*=5.0 Hz, 1.4 Hz, 1H), 7.75 (dd, *J*=6.5 Hz, 1.7 Hz, 2H), 7.62 (d, *J*=8.5 Hz, 2H), 7.54 – 7.41 (m, 2H), 7.28 (ddd,

$J=6.8$ Hz, 4.8 Hz, 2.3 Hz, 1H), 4.97 (s, 2H), 1.27 (s, 9H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) $\delta = 156.70$, 151.98 , 148.34 , 146.70 , 137.35 , 136.73 , 127.68 , 125.79 , 123.73 , 120.07 , 50.49 , 34.91 , 30.76 . $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_3\text{S}$ ($\text{M}+\text{Na}^+$): 355.1092 , found: 355.1093 .



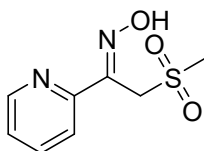
(Z)-4-((2-(hydroxyimino)-2-(pyridin-2-yl)ethyl)sulfonyl)benzonitrile

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3h** (271 mg; yield 90%), white solid; m.p. 172 - 173 °C: ^1H NMR (400 MHz, $\text{DMSO-}d_6$) $\delta = 12.23$ (s, 1H), 8.39 (d, $J=4.9$ Hz, 1H), 8.05 (d, $J=8.1$ Hz, 2H), 7.94 (d, $J=8.1$ Hz, 2H), 7.80 (t, $J=2.4$ Hz, 2H), 7.36 (td, $J=5.1$ Hz, 3.1 Hz, 1H), 5.09 (s, 2H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) $\delta = 151.38$, 148.12 , 145.96 , 143.93 , 136.63 , 132.77 , 128.43 , 123.69 , 119.71 , 117.44 , 115.76 , 49.98 . $\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_3\text{S}$ ($\text{M}+\text{H}^+$): 302.0599 , found: 302.0607 .



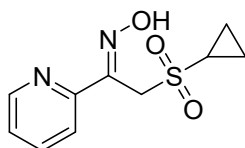
(Z)-2-(naphthalen-2-ylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3i** (209 mg; yield 64%), white solid; m.p. 189 - 190 °C: ^1H NMR (400 MHz, $\text{DMSO-}d_6$) $\delta = 12.16$ (s, 1H), 8.42 (d, $J=1.9$ Hz, 1H), 8.31 (dt, $J=4.8$ Hz, 1.4 Hz, 1H), 8.24 – 7.97 (m, 3H), 7.87 – 7.77 (m, 2H), 7.77 – 7.68 (m, 2H), 7.65 (ddd, $J=8.2$ Hz, 6.9 Hz, 1.3 Hz, 1H), 7.21 (ddd, $J=7.3$ Hz, 4.8 Hz, 1.3 Hz, 1H), 5.13 (s, 2H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) $\delta = 152.12$, 148.34 , 146.77 , 137.53 , 136.76 , 134.78 , 131.53 , 129.53 , 129.40 , 129.30 , 129.05 , 127.85 , 127.57 , 123.76 , 122.98 , 120.15 , 50.62 . $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$ ($\text{M}+\text{H}^+$): 327.0803 , found: 327.0808 .



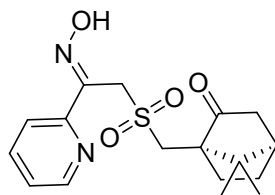
(Z)-2-(methylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3j** (169 mg; yield 79%), white solid; m.p. 80 - 81 °C: ^1H NMR (400 MHz, $\text{DMSO-}d_6$) $\delta = 12.51$ (s, 1H), 8.62 (d, $J=4.9$ Hz, 1H), 8.03 – 7.68 (m, 2H), 7.42 (t, $J=6.2$ Hz, 1H), 4.88 (s, 2H), 3.07 (s, 3H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) $\delta = 152.35$, 148.69 , 147.56 , 137.02 , 124.18 , 120.12 , 49.21 , 43.74 . $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3\text{S}$ ($\text{M}+\text{H}^+$): 215.0490 , found: 215.0486 .



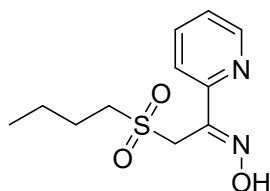
(Z)-2-(cyclopropylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3k** (206 mg; yield 86%), white solid; m.p. 92-93 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.47 (s, 1H), 8.61 (dt, *J*=4.7 Hz, 1.4 Hz, 1H), 8.21 – 7.55 (m, 2H), 7.41 (ddd, *J*=7.3 Hz, 4.8 Hz, 1.4 Hz, 1H), 4.91 (s, 2H), 2.70 (tt, *J*=7.3 Hz, 5.6 Hz, 1H), 1.06 – 0.62 (m, 4H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 152.55, 148.77, 147.39, 137.10, 124.21, 120.23, 48.46, 32.05, 4.87. C₁₀H₁₂N₂O₃S (M+Na⁺): 263.0466, found: 263.0473.



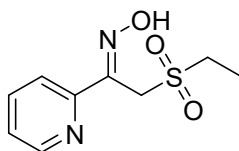
(1S,4R)-1-(((Z)-2-(hydroxyimino)-2-(pyridin-2-yl)ethyl)sulfonyl)methyl)-7,7-dimethylbicyclo[2.2.1]heptan-2-one

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3l** (129 mg; yield 37%), white solid; m.p. 105-106 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.54 (s, 1H), 8.60 (ddd, *J*=4.9, 1.8 Hz, 1.1 Hz, 1H), 8.05 – 7.68 (m, 2H), 7.41 (ddd, *J*=7.3 Hz, 4.9 Hz, 1.4 Hz, 1H), 5.08 (d, *J*=13.4 Hz, 1H), 4.92 (d, *J*=13.4 Hz, 1H), 3.72 (d, *J*=15.0 Hz, 1H), 3.20 (d, *J*=14.9 Hz, 1H), 2.45 – 2.13 (m, 2H), 2.14 – 1.80 (m, 3H), 1.59 (ddd, *J*=13.9 Hz, 9.4 Hz, 4.7 Hz, 1H), 1.38 (ddd, *J*=12.8 Hz, 9.3 Hz, 3.9 Hz, 1H), 0.95 (s, 3H), 0.79 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 214.75, 152.94, 149.01, 148.07, 137.43, 124.55, 120.56, 59.05, 52.74, 50.83, 48.35, 42.49, 42.25, 26.83, 25.05, 19.91, 19.78. C₁₇H₂₂N₂O₄S (M+H⁺): 351.1379, found: 351.1384.



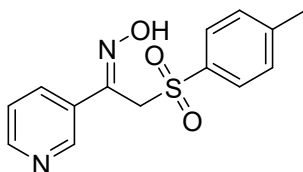
(Z)-2-(butylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3m** (163 mg; yield 64%), white solid; m.p. 85-86 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.52 (s, 1H), 8.60 (dt, *J*=4.8 Hz, 1.4 Hz, 1H), 8.06 – 7.75 (m, 2H), 7.41 (ddd, *J*=7.3 Hz, 4.9 Hz, 1.3 Hz, 1H), 4.83 (s, 2H), 3.24 – 3.01 (m, 2H), 1.69 (ddd, *J*=11.9 Hz, 10.1 Hz, 6.5 Hz, 2H), 1.35 (dt, *J*=14.7 Hz, 7.4 Hz, 2H), 0.86 (t, *J*=7.4 Hz, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 152.49, 148.73, 147.59, 137.11, 124.27, 120.21, 54.70, 47.38, 23.79, 21.13, 13.57. C₁₁H₁₆N₂O₃S (M+H⁺): 257.0960, found: 257.0964.



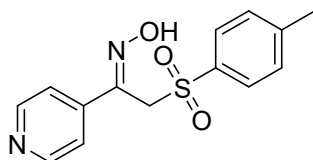
(Z)-2-(ethylsulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3n** (189 mg; yield 83%), white solid; m.p.83-84 °C:¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.51 (s, 1H), 8.61 (d, *J*=4.9 Hz, 1H), 8.01 – 7.66 (m, 2H), 7.40 (dd, *J*=7.4 Hz, 4.9 Hz, 1H), 4.84 (s, 2H), 3.16 (q, *J*=7.4 Hz, 2H), 1.26 (t, *J*=7.4 Hz, 3H).¹³C NMR (101 MHz, DMSO-*d*₆) δ = 152.46, 148.71, 147.56, 137.04, 124.20, 120.14, 49.57, 46.76, 6.55. C₉H₁₂N₂O₃S (M+H⁺):229.0647, found:229.0949.



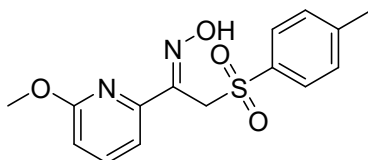
(Z)-1-(pyridin-3-yl)-2-tosylethan-1-one oxime [3]

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3o** (220 mg; yield 76%), white solid; m.p.170-171 °C:¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.07 (s, 1H), 8.80 (d, *J*=2.3 Hz, 1H), 8.55 (dd, *J*=4.8 Hz, 1.5 Hz, 1H), 8.12 – 7.75 (m, 1H), 7.62 (d, *J*=8.0 Hz, 2H), 7.44 – 7.21 (m, 3H), 4.98 (s, 2H), 2.37 (s, 3H).¹³C NMR (101 MHz, DMSO-*d*₆) δ = 149.72, 147.37, 144.62, 143.90, 136.53, 133.81, 130.44, 129.64, 127.97, 123.31, 51.04, 21.15. C₁₄H₁₄N₂O₃S (M+H⁺):291.0803, found:291.0807.



(Z)-1-(pyridin-4-yl)-2-tosylethan-1-one oxime

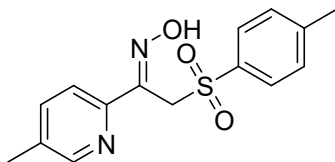
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3p** (246 mg; yield 85%), white solid; m.p.176-177 °C:¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.40 (s, 1H), 8.55 (d, *J*=5.3 Hz, 2H), 7.62 (dd, *J*=12.8 Hz, 6.7 Hz, 4H), 7.36 (d, *J*=7.9 Hz, 2H), 4.94 (s, 2H), 2.37 (s, 3H).¹³C NMR (101 MHz, DMSO-*d*₆) δ = 149.80, 144.81, 144.40, 141.96, 136.61, 129.74, 128.08, 120.74, 50.92, 21.23. C₁₄H₁₄N₂O₃S (M+H⁺):291.0803, found:291.0808.



(Z)-1-(6-methoxy-pyridin-2-yl)-2-tosylethan-1-one oxime

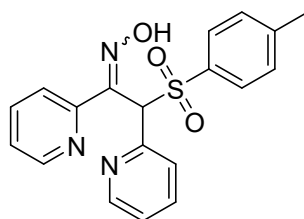
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3q** (278 mg;

yield 87%), white solid; m.p.155-156 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.15 (s, 1H), 7.68 (dd, *J*=8.1 Hz, 7.5 Hz, 1H), 7.65 – 7.52 (m, 2H), 7.46 – 7.20 (m, 3H), 6.74 (dd, *J*=8.2 Hz, 0.8 Hz, 1H), 4.94 (s, 2H), 3.71 (s, 3H), 2.34 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 162.46, 149.74, 146.49, 144.23, 139.66, 137.53, 129.46, 127.75, 112.98, 110.42, 52.85, 50.85, 21.07. C₁₅H₁₆N₂O₄S (M+Na⁺):343.0728, found:343.0738.



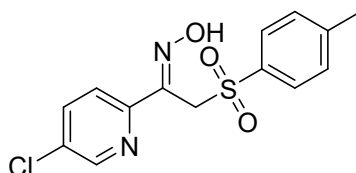
(Z)-1-(5-methylpyridin-2-yl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3r** (285 mg; yield 94%), white solid; m.p.147-148 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.01 (s, 1H), 8.24 (dt, *J*=2.1 Hz, 0.9 Hz, 1H), 7.66 (dd, *J*=8.0 Hz, 0.9 Hz, 1H), 7.64 – 7.58 (m, 3H), 7.36 – 7.29 (m, 2H), 4.96 (s, 2H), 2.37 (s, 3H), 2.29 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 149.61, 148.60, 146.70, 144.13, 137.61, 137.15, 133.30, 129.40, 127.79, 119.60, 50.66, 21.08, 17.75. C₁₅H₁₆N₂O₃S (M+H⁺):305.0960, found:305.0965.



1,2-di(pyridin-2-yl)-2-tosylethan-1-one oxime

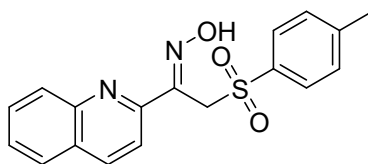
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3s** (209 mg; yield 57%, *Z*:*E*=4.5:1), white solid; m.p.179-180 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.48 (s, 1H), 8.62 – 8.56 (m, 1H), 8.44 – 8.37 (m, 1H), 7.98 (d, *J*=8.0 Hz, 1H), 7.93 (d, *J*=8.0 Hz, 1H), 7.87 (dd, *J*=7.5 Hz, 1.8 Hz, 1H), 7.79 (tt, *J*=7.8 Hz, 2.9 Hz, 1H), 7.44 (d, *J*=8.0 Hz, 2H), 7.38 – 7.33 (m, 2H), 7.26 (d, *J*=8.0 Hz, 2H), 6.69 (s, 1H), 2.36 (s, 1H), 2.32 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 151.40, 148.92, 148.78, 146.87, 144.56, 136.40, 136.26, 135.49, 129.51, 128.74, 128.31, 126.75, 126.01, 124.29, 123.81, 72.72, 21.10. C₁₉H₁₇N₃O₃S (M+H⁺):368.1069, found:368.1073.



(Z)-1-(5-chloropyridin-2-yl)-2-tosylethan-1-one oxime

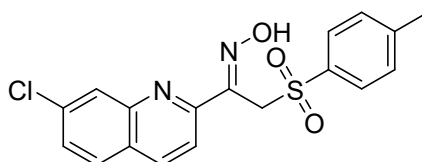
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3t** (259 mg; yield 80%), white solid; m.p.160-161 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.31 (s, 1H), 8.42 (dd, *J*=2.5 Hz, 0.8 Hz, 1H), 7.90 (dd, *J*=8.6 Hz, 2.5 Hz, 1H), 7.76 (dd, *J*=8.6 Hz, 0.8 Hz, 1H), 7.69 – 7.51 (m, 2H), 7.43 – 7.24 (m, 2H), 4.93 (s, 2H), 2.36 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 150.71, 146.97, 145.96,

144.33, 137.32, 136.65, 131.03, 129.48, 127.88, 121.32, 50.54, 21.08. $C_{14}H_{13}ClN_2O_3S$ ($M+H^+$):325.0414, found:325.0417.



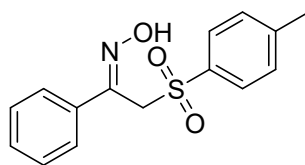
(Z)-1-(quinolin-2-yl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3u** (268 mg; yield 79%), white solid; m.p.189-190 °C: 1H NMR (400 MHz, Chloroform-*d*) δ = 8.83 (s, 1H), 8.02 (d, J =8.7 Hz, 1H), 7.89 (d, J =8.6 Hz, 1H), 7.71 (t, J =8.2 Hz, 2H), 7.69 – 7.57 (m, 3H), 7.48 (ddd, J =8.1 Hz, 6.8 Hz, 1.3 Hz, 1H), 6.96 (d, J =8.0 Hz, 2H), 5.24 (s, 2H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ = 155.64, 149.41, 146.91, 144.43, 137.33, 136.45, 129.71, 129.51, 129.32, 128.45, 128.04, 127.53, 127.29, 118.33, 51.06, 21.39. $C_{18}H_{16}N_2O_3S$ ($M+H^+$):341.0960, found:341.0965.



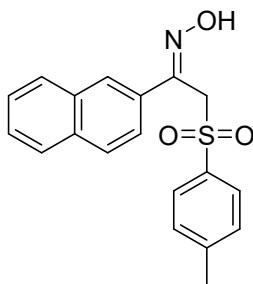
(Z)-1-(7-chloroquinolin-2-yl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **3v** (299 mg; yield 80%), white solid; m.p.175-176 °C: 1H NMR (400 MHz, DMSO-*d*₆) δ = 12.62 (s, 1H), 8.33 (dd, J =8.8 Hz, 0.8 Hz, 1H), 7.95 (dd, J =17.2 Hz, 8.7 Hz, 2H), 7.64 – 7.55 (m, 3H), 7.52 (d, J =2.1 Hz, 1H), 7.21 – 7.15 (m, 2H), 5.07 (s, 2H), 2.19 (s, 3H). ^{13}C NMR (101 MHz, DMSO-*d*₆) δ = 152.99, 146.99, 146.50, 144.04, 137.76, 136.66, 134.12, 129.71, 129.35, 127.81, 127.61, 127.52, 125.99, 118.02, 50.59, 20.85. $C_{18}H_{15}ClN_2O_3S$ ($M+H^+$):375.0570, found:375.0574.



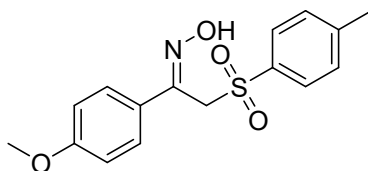
(Z)-1-phenyl-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5a** (271 mg; yield 94%), white solid; m.p.179-180 °C: 1H NMR (400 MHz, DMSO-*d*₆) δ = 11.80 (s, 1H), 7.74 – 7.51 (m, 4H), 7.35 (d, J =7.3 Hz, 5H), 4.91 (s, 2H), 2.37 (s, 3H). ^{13}C NMR (101 MHz, DMSO-*d*₆) δ = 145.71, 144.43, 136.88, 134.59, 129.54, 129.01, 128.26, 127.94, 126.44, 51.44, 21.15. $C_{15}H_{15}NO_3S$ ($M+Na^+$):312.0670, found:312.0677.



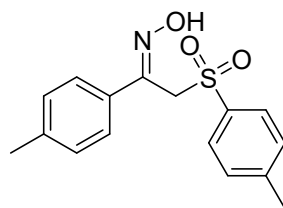
(Z)-1-(naphthalen-2-yl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5b** (298 mg; yield 88%), white solid; m.p.183-184 °C:¹H NMR (400 MHz, Chloroform-*d*) δ = 8.63 (s, 1H), 7.94 (d, J =1.2 Hz, 1H), 7.85 – 7.75 (m, 4H), 7.74 – 7.67 (m, 2H), 7.55 – 7.45 (m, 2H), 7.14 (d, J =8.0 Hz, 2H), 4.86 (s, 2H), 2.26 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 148.00, 145.10, 136.56, 133.91, 132.98, 131.01, 129.57, 128.85, 128.60, 128.45, 127.69, 127.31, 127.25, 126.65, 123.35, 52.78, 21.61. C₁₉H₁₇NO₃S (M+Na⁺):362.0827, found:362.0831.



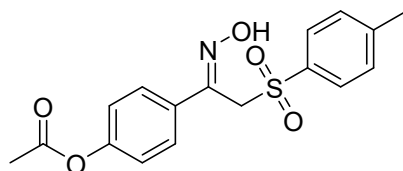
(Z)-1-(4-methoxyphenyl)-2-tosylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5c** (306 mg; yield 96%), white solid; m.p.165-166 °C:¹H NMR (400 MHz, Chloroform-*d*) δ = 8.75 (s, 1H), 7.78 – 7.70 (m, 2H), 7.64 – 7.56 (m, 2H), 7.26 (d, J =8.1 Hz, 2H), 6.94 – 6.86 (m, 2H), 4.74 (s, 2H), 3.86 (s, 3H), 2.40 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 161.05, 147.38, 144.95, 136.63, 129.53, 128.56, 128.19, 126.26, 114.10, 55.46, 52.80, 21.71. C₁₆H₁₇NO₄S (M+Na⁺):342.0776, found:342.0767.



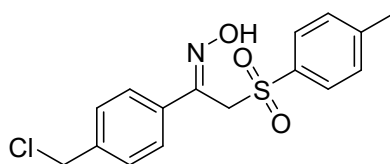
(Z)-1-(*p*-tolyl)-2-tosylethan-1-one oxime [3]

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5d** (254 mg; yield 84%), white solid; m.p.175-176 °C:¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.64 (s, 1H), 7.68 – 7.58 (m, 2H), 7.53 (dt, J =8.2 Hz, 1.6 Hz, 2H), 7.35 (d, J =7.9 Hz, 2H), 7.15 (d, J =8.0 Hz, 2H), 4.87 (s, 2H), 2.38 (s, 3H), 2.31 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 145.55, 144.33, 138.51, 136.88, 131.80, 129.44, 128.78, 127.87, 126.32, 51.44, 21.08, 20.80.



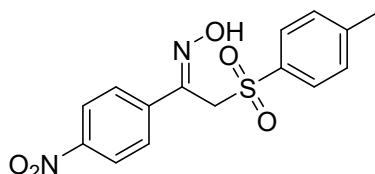
(Z)-4-(1-(hydroxyimino)-2-tosylethyl)phenyl acetate

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5e** (291 mg; yield 81%), white solid; m.p.173-174 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.87 (s, 1H), 7.73 – 7.67 (m, 2H), 7.67 – 7.62 (m, 2H), 7.36 (d, *J*=8.2 Hz, 2H), 7.15 – 7.09 (m, 2H), 4.92 (s, 2H), 2.37 (s, 3H), 2.28 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 169.27, 151.19, 145.15, 144.65, 136.87, 132.27, 129.68, 128.02, 127.68, 121.81, 51.52, 21.22, 21.00. C₁₇H₁₇NO₅S (M+Na⁺):370.0725, found:370.0730.



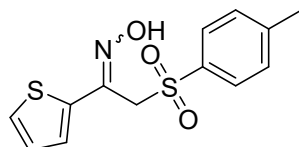
(Z)-1-(4-(chloromethyl)phenyl)-2-tosylethan-1-one oxime [4]

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5f** (256 mg; yield 76%), white solid; m.p.168-169 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 11.84 (s, 1H), 7.67 – 7.55 (m, 4H), 7.43 – 7.37 (m, 2H), 7.34 (d, *J*=8.1 Hz, 2H), 4.91 (s, 2H), 4.77 (s, 2H), 2.37 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 145.34, 144.40, 138.31, 136.74, 134.52, 129.49, 128.70, 127.90, 126.66, 51.33, 45.74, 21.11. C₁₆H₁₆ClNO₃S (M+Na⁺):360.0437, found:360.0441.



(Z)-1-(4-nitrophenyl)-2-tosylethan-1-one oxime

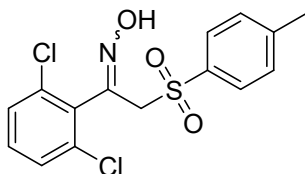
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5g** (260 mg; yield 78%), yellow solid; m.p.87-88 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.39 (s, 1H), 8.19 (d, *J*=8.6 Hz, 2H), 7.91 (d, *J*=8.6, 2H), 7.63 (d, *J*=7.9, 2H), 7.35 (d, *J*=8.0 Hz, 2H), 5.02 (s, 2H), 2.36 (s, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ = 147.46, 144.74, 144.74, 140.88, 136.52, 129.67, 128.06, 127.64, 123.43, 51.17, 21.13. C₁₅H₁₄N₂O₅S (M+Na⁺):357.0521, found:357.0526.



1-(thiophen-2-yl)-2-tosylethan-1-one oxime

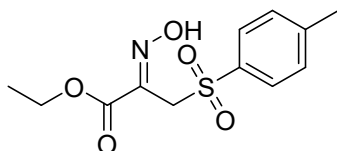
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5h** (138 mg; yield 47%, E:Z=1.06:1), white solid; m.p.100-101 °C: ¹H NMR (400 MHz, DMSO-*d*₆) δ = 12.48 (s,

1H), 11.75 (s, 1H), 7.67 (ddd, $J=26.7$ Hz, 19.8 Hz, 4.5 Hz, 6H), 7.55 – 7.28 (m, 6H), 7.17 – 6.93 (m, 2H), 4.85 (s, 2H), 4.74 (s, 2H), 2.39 (d, $J=2.0$, 6H). ^{13}C NMR (101 MHz, DMSO- d_6) δ = 144.96, 144.81, 142.34, 140.23, 138.87, 137.25, 136.44, 131.37, 131.13, 131.04, 130.09, 130.01, 128.78, 128.62, 128.42, 127.74, 127.63, 125.91, 59.54, 52.57, 21.58, 21.55. $\text{C}_{13}\text{H}_{13}\text{NO}_3\text{S}_2$ ($\text{M}+\text{Na}^+$):318.0235, found:318.0239.



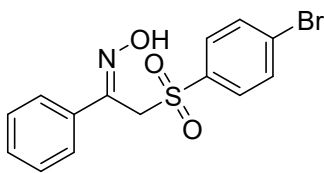
1-(2,6-dichlorophenyl)-2-tosylethan-1-one oxime [5]

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5i** (138 mg; yield 47%, Z:E=13:1), white solid; m.p.78-79 °C: ^1H NMR (400 MHz, DMSO- d_6) δ = 11.96 (s, 1H), 7.93 – 7.76 (m, 2H), 7.58 – 7.27 (m, 5H), 4.58 (s, 2H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, DMSO- d_6) δ = 144.37, 141.82, 136.15, 132.82, 131.43, 131.18, 129.56, 128.12, 127.98, 58.95, 21.07. $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NO}_3\text{S}$ ($\text{M}+\text{Na}^+$):379.9891, found:379.9891.



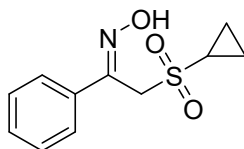
ethyl (Z)-2-(hydroxyimino)-3-tosylpropanoate [5]

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5j** (262 mg; yield 92%), white solid; m.p.97-98 °C: ^1H NMR (400 MHz, Chloroform- d) δ = 10.94 (s, 1H), 7.86 – 7.61 (m, 2H), 7.31 (d, $J=8.2$ Hz, 2H), 4.57 (s, 2H), 4.17 (q, $J=7.1$ Hz, 2H), 2.40 (s, 3H), 1.23 (t, $J=7.1$, 3H). ^{13}C NMR (101 MHz, Chloroform- d) δ = 162.09, 145.28, 141.08, 136.38, 129.79, 128.22, 62.54, 51.41, 21.60, 13.86. $\text{C}_{12}\text{H}_{15}\text{NO}_5\text{S}$ ($\text{M}+\text{Na}^+$):308.0569, found:308.0572.



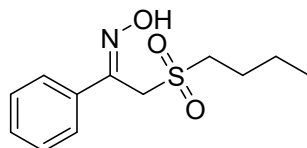
(Z)-2-((4-bromophenyl)sulfonyl)-1-phenylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5k** (261 mg; yield 74%), white solid; m.p.157-158 °C: ^1H NMR (400 MHz, DMSO- d_6) δ = 11.81 (s, 1H), 7.81 – 7.74 (m, 2H), 7.66 (dq, $J=9.7$ Hz, 2.6 Hz, 4H), 7.40 – 7.33 (m, 3H), 4.97 (s, 2H). ^{13}C NMR (101 MHz, DMSO- d_6) δ = 145.46, 138.74, 134.40, 132.11, 130.00, 129.05, 128.27, 128.18, 126.41, 51.37. $\text{C}_{14}\text{H}_{12}\text{BrNO}_3\text{S}$ ($\text{M}+\text{Na}^+$):375.9619,377.9598, found:375.9624,377.9607.



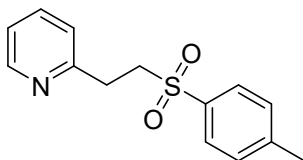
(Z)-2-(cyclopropylsulfonyl)-1-phenylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5l** (162 mg; yield 68%), white solid; m.p.115-116 °C: ¹H NMR (400 MHz, Chloroform-*d*) δ = 9.49 (s, 1H), 7.82 – 7.66 (m, 2H), 7.49 – 7.31 (m, 3H), 4.70 (s, 2H), 2.52 (tt, *J*=8.0 Hz, 4.8 Hz, 1H), 1.24 – 1.14 (m, 2H), 0.99 – 0.87 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 148.39, 133.89, 130.24, 128.88, 126.74, 50.76, 31.60, 5.48. C₁₁H₁₃NO₃S (M+Na⁺):262.0514, found:262.0510.



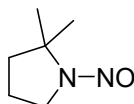
(Z)-2-(butylsulfonyl)-1-phenylethan-1-one oxime

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 1:1) to give the product **5m** (234 mg; yield 92%), white solid; m.p.112-113 °C: ¹H NMR (400 MHz, Chloroform-*d*) δ = 9.92 (s, 1H), 7.73 (dq, *J*=7.9 Hz, 2.5 Hz, 1.8 Hz, 2H), 7.43 – 7.36 (m, 3H), 4.59 (s, 2H), 3.24 – 2.97 (m, 2H), 1.96 – 1.71 (m, 2H), 1.39 (h, *J*=7.3 Hz, 2H), 0.89 (t, *J*=7.4 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 148.30, 133.84, 130.17, 128.78, 126.74, 54.68, 50.09, 23.65, 21.68, 13.59. C₁₂H₁₇NO₃S (M+Na⁺):278.0827, found:278.0832.



2-(2-tosylethyl)pyridine [6]

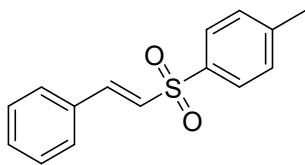
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 3:1) to give the product **3aa** (250 mg; yield 96%), white solid; m.p.67-68 °C: ¹H NMR (400 MHz, Chloroform-*d*) δ = 8.48 – 8.32 (m, 1H), 7.85 – 7.70 (m, 2H), 7.56 (td, *J*=7.7 Hz, 1.8 Hz, 1H), 7.32 (d, *J*=8.0 Hz, 2H), 7.22 – 7.05 (m, 2H), 3.68 – 3.51 (m, 2H), 3.31 – 3.12 (m, 2H), 2.42 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 157.25, 149.43, 144.78, 136.80, 136.08, 129.98, 128.21, 123.35, 121.97, 55.34, 30.96, 21.71. C₁₄H₁₅NO₂S (M+H⁺):262.0896, found:262.0906.



2,2-dimethyl-1-nitrosopyrrolidine

The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 15:1) to give the product **6** (58 mg; yield 45%), colorless liquid: ¹H NMR (400 MHz, Chloroform-*d*) δ = 3.71 – 3.45 (m, 2H), 2.02 – 1.84 (m, 4H), 1.52 (s, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 63.80, 45.58, 39.19, 27.97, 19.65. C₆H₁₂N₂O

(M+H⁺):129.1028, found:129.1029.



(*E*)-1-methyl-4-(styrylsulfonyl)benzene [7]

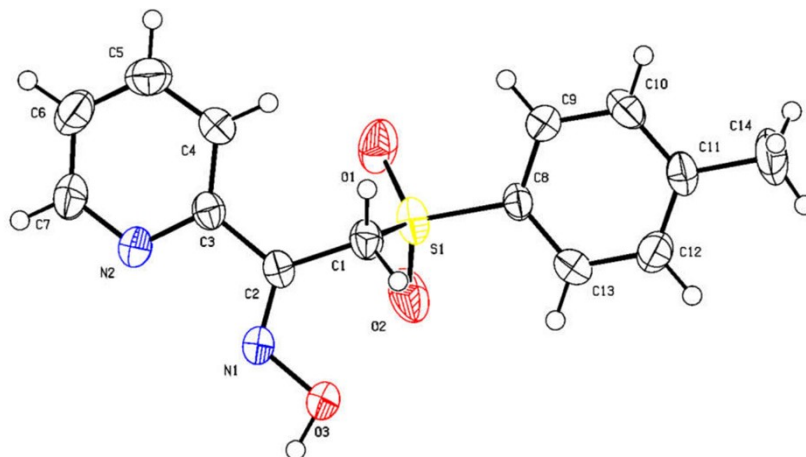
The title compound was prepared according to the general working procedure and purified by column chromatography (petroleum ether / ethyl acetate = 10:1) to give the product **5p'** (147 mg; yield 57%), white solid: ¹H NMR (400 MHz, Chloroform-*d*) δ = 7.92 – 7.74 (m, 2H), 7.66 (d, *J*=15.4 Hz, 1H), 7.56 – 7.43 (m, 2H), 7.44 – 7.31 (m, 5H), 6.85 (d, *J*=15.4 Hz, 1H), 2.43 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ = 144.54, 142.06, 137.79, 132.53, 131.24, 130.10, 129.19, 128.65, 127.83, 127.67, 21.76.

References

- [1] T. Mandal, S. Mallick, N. Kumari and S. De Sarkar, *Org. Lett.* **2022**, *24*, 8452-8457.
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- [3] X.-Q. Chu, D. Ge, T.-P. Loh and Z.-L. Shen, *Organic Chemistry Frontiers*. **2019**, *6*, 835-840.
- [4] J. Yang, Y.-Y. Liu, R.-J. Song, Z.-H. Peng and J.-H. Li, *Adv. Synth. Catal.* **2016**, *358*, 2286-2292.
- [5] N. Liu, P. Yin, Y. Chen, Y. Deng and L. He, *Eur. J. Org. Chem.* **2012**, *2012*, 2711-2714.
- [6] Y. Wang, Y. Zhao, C. Cai, L. Wang and H. Gong, *Organic Letters*. **2021**, *23*, 8296-8301.
- [7] M. Pramanik, K. Choudhuri and P. Mal, *Asian Journal of Organic Chemistry*. **2019**, *8*, 144-150.

Crystal Structure

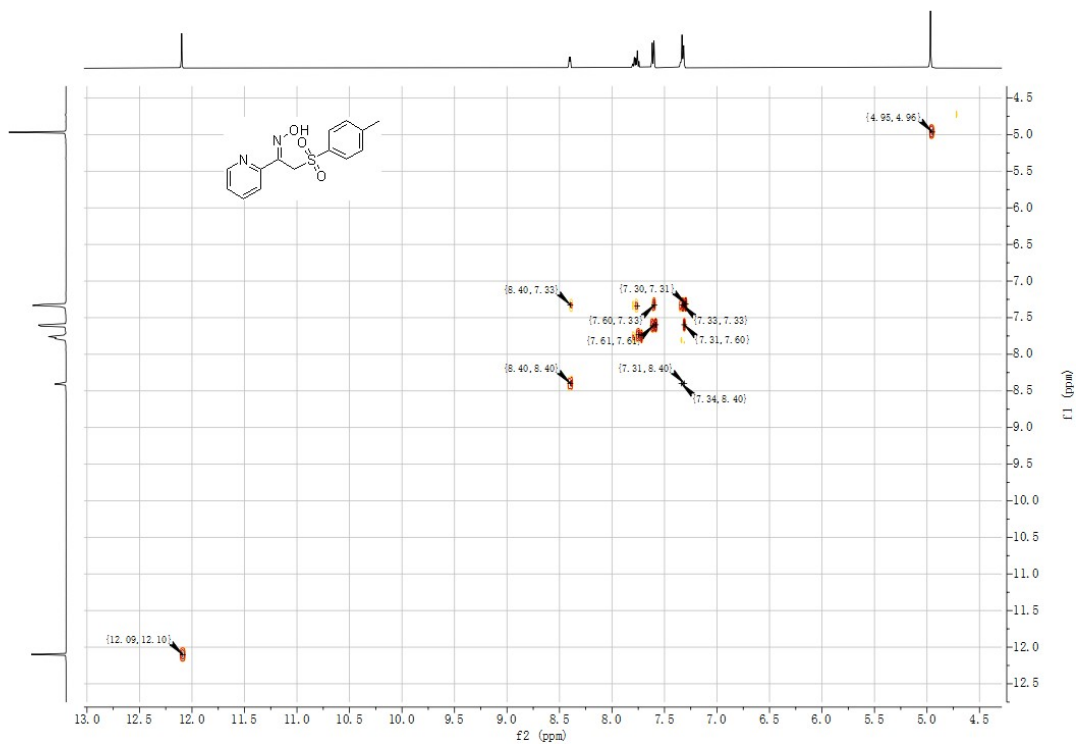
The Product **3a** crystals were grown in ethyl acetate and petroleum ether mixed solvent. The structures are given below. The corresponding .cif file has been uploaded separately as supporting information.



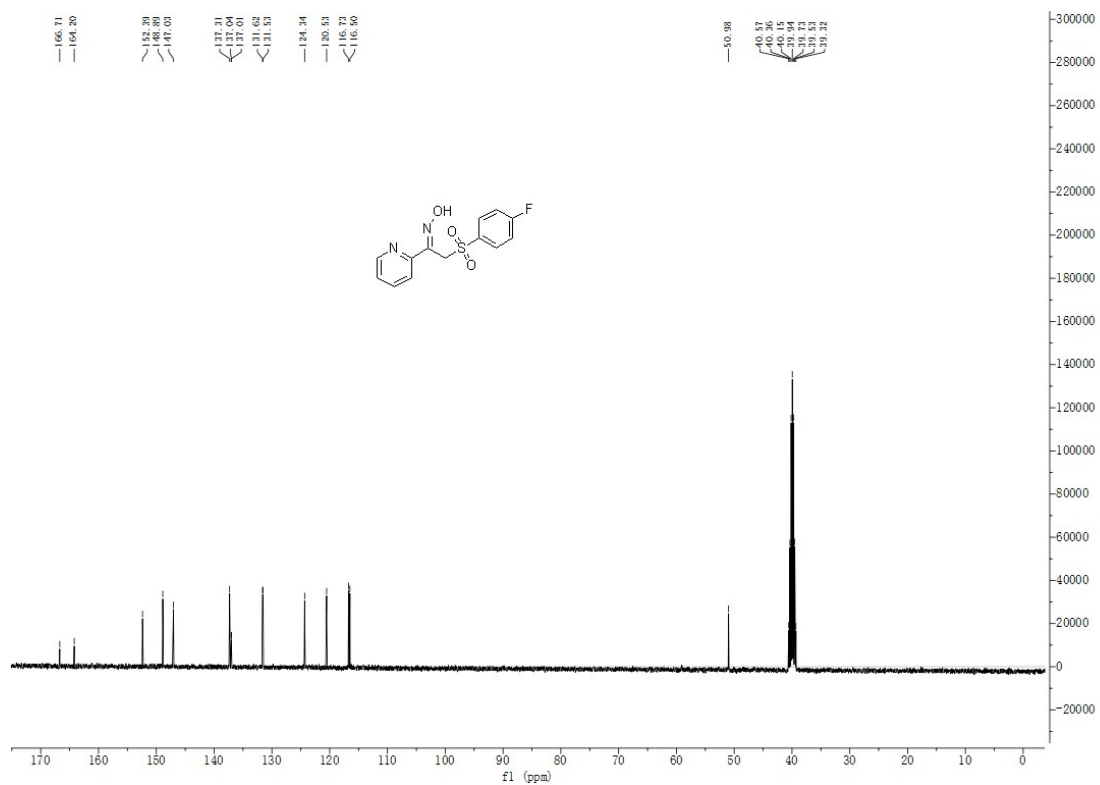
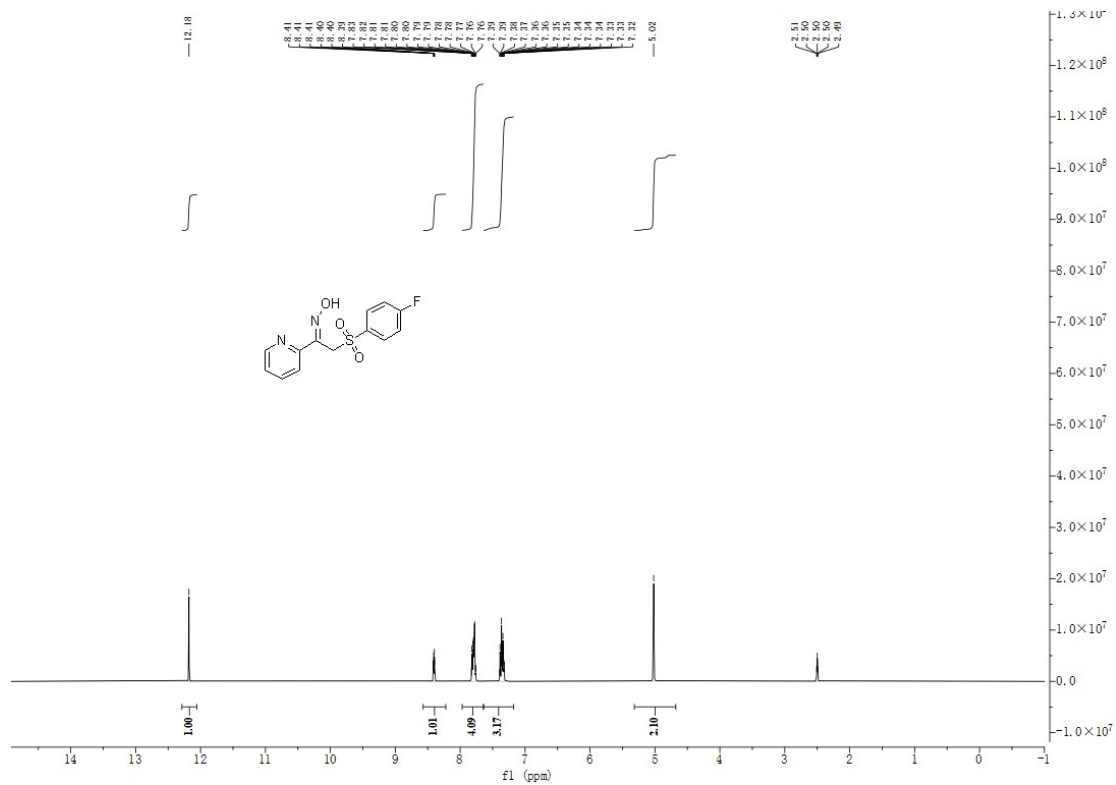
CCDC-2363463

Empirical formula	C ₁₄ H ₁₄ N ₂ O ₃ S
Formula weight	290.33
Temperature/K	293(2)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	15.053(3)
b/Å	5.5099(9)
c/Å	16.412(2)
α/°	90
β/°	90.360(14)
γ/°	90
Volume/Å ³	1361.1(4)
Z	4
ρ _{calc} /cm ³	1.417
μ/mm ⁻¹	0.246
F(000)	608.0
Crystal size/mm ³	0.16 × 0.14 × 0.09
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	4.964 to 51.996
Index ranges	-18 ≤ h ≤ 13, -6 ≤ k ≤ 6, -20 ≤ l ≤ 16
Reflections collected	5321
Independent reflections	2675 [R _{int} = 0.0598, R _{sigma} = 0.1125]

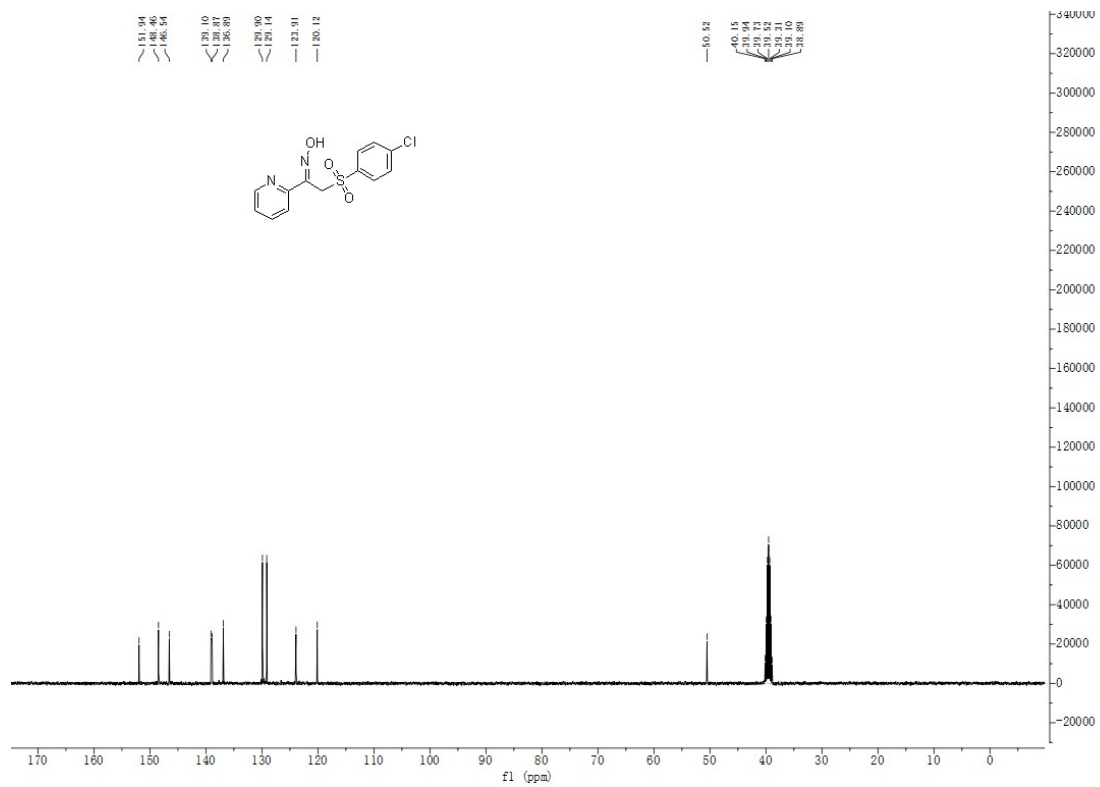
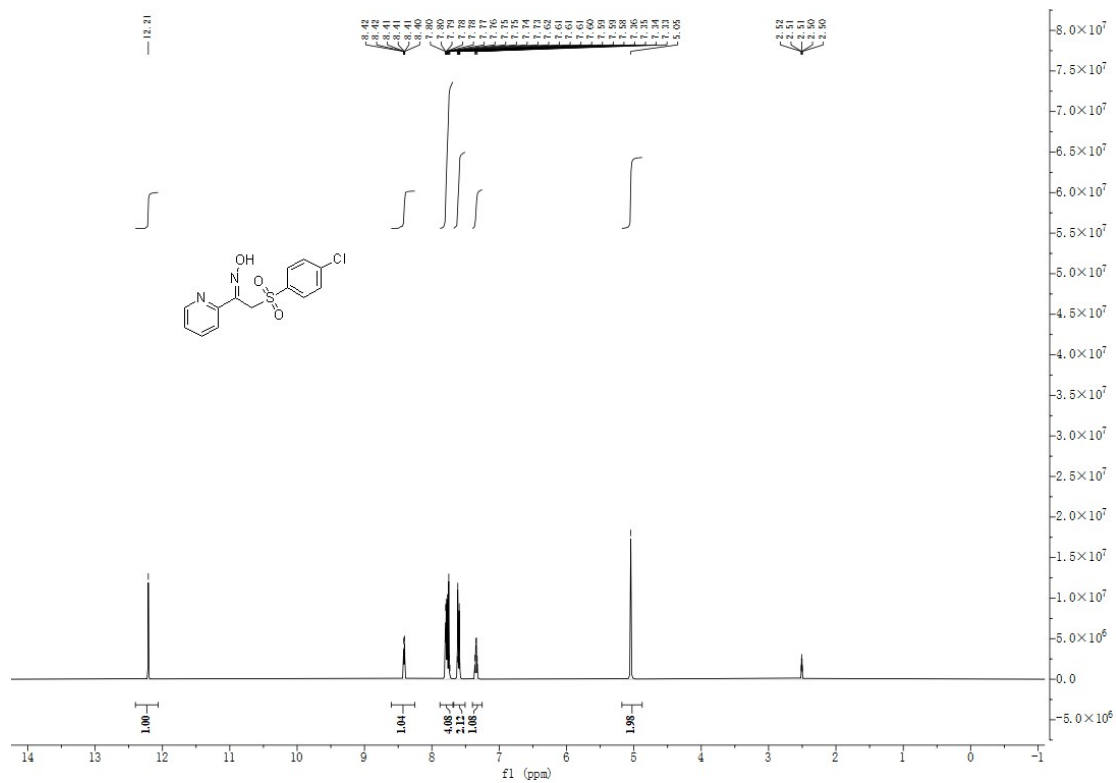
Data/restraints/parameters	2675/1/186
Goodness-of-fit on F^2	1.038
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0713$, $wR_2 = 0.1063$
Final R indexes [all data]	$R_1 = 0.1436$, $wR_2 = 0.1329$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.25/-0.26



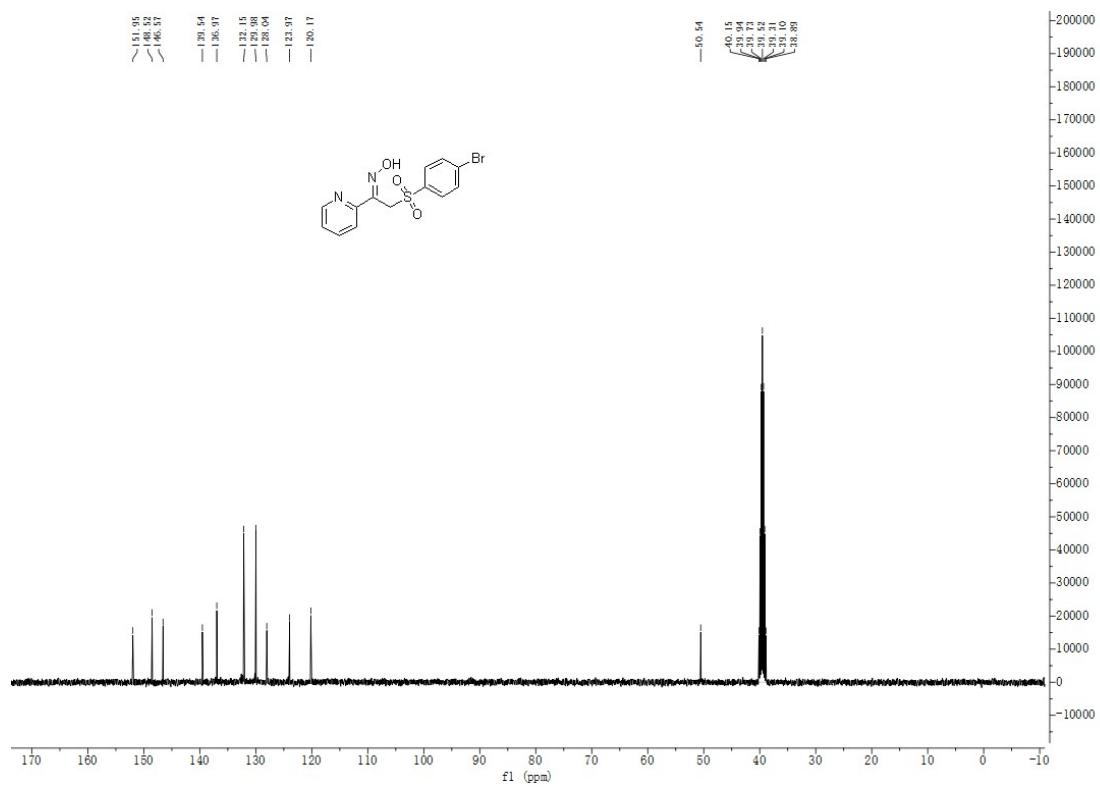
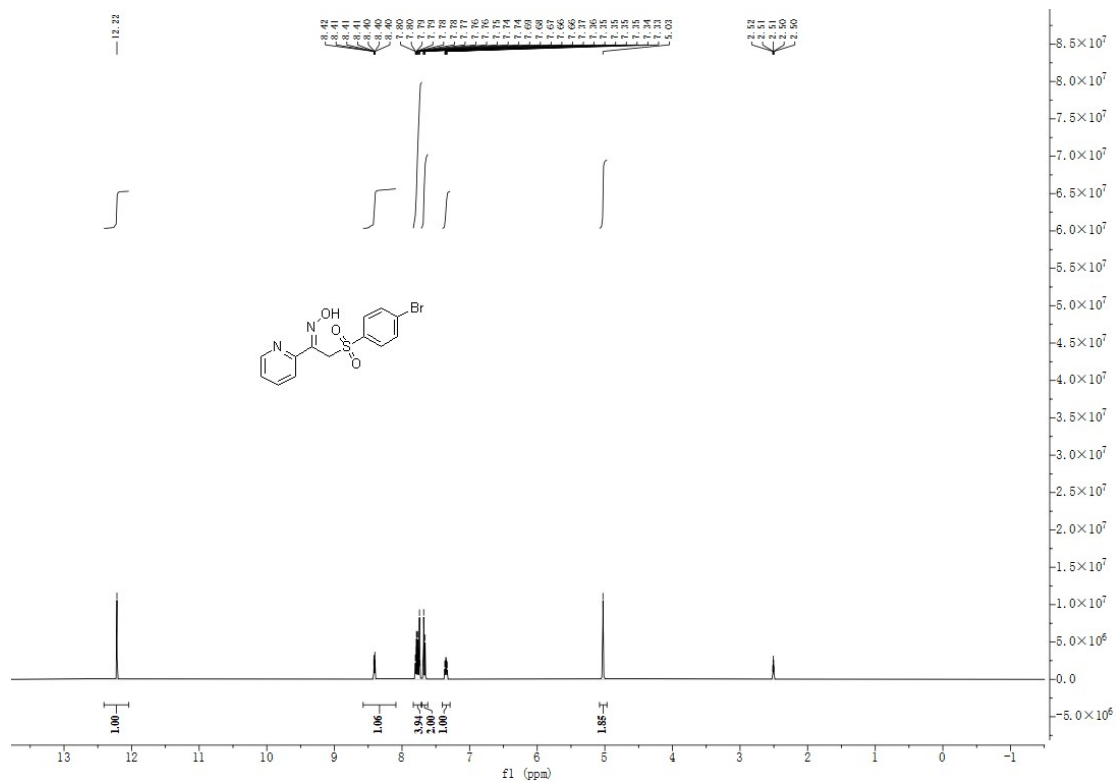
3a ¹H-¹H COSY (500 MHz, DMSO-*d*₆)



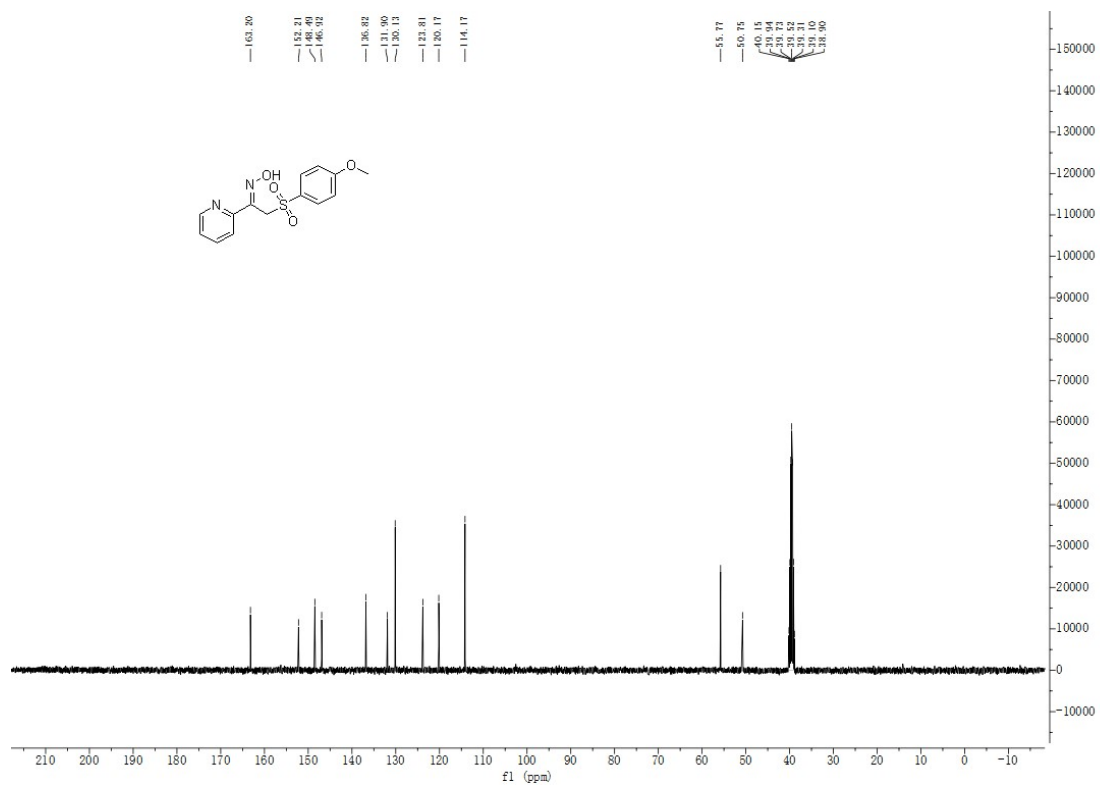
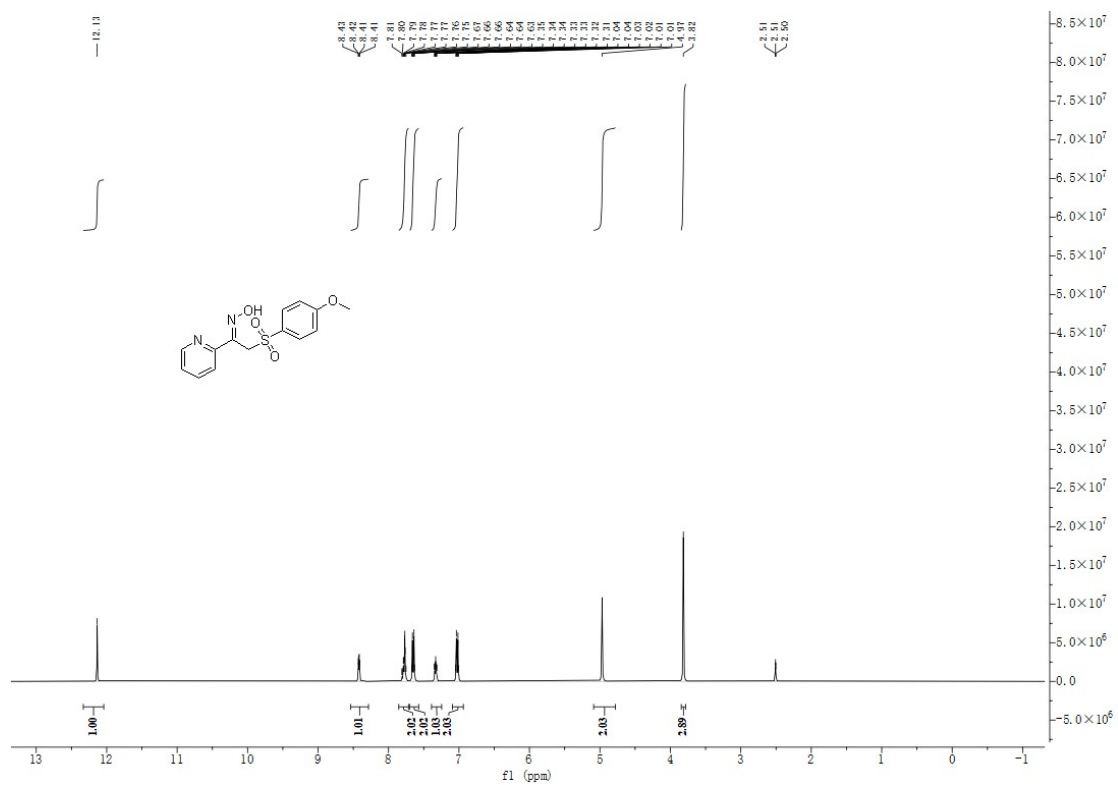
3b ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



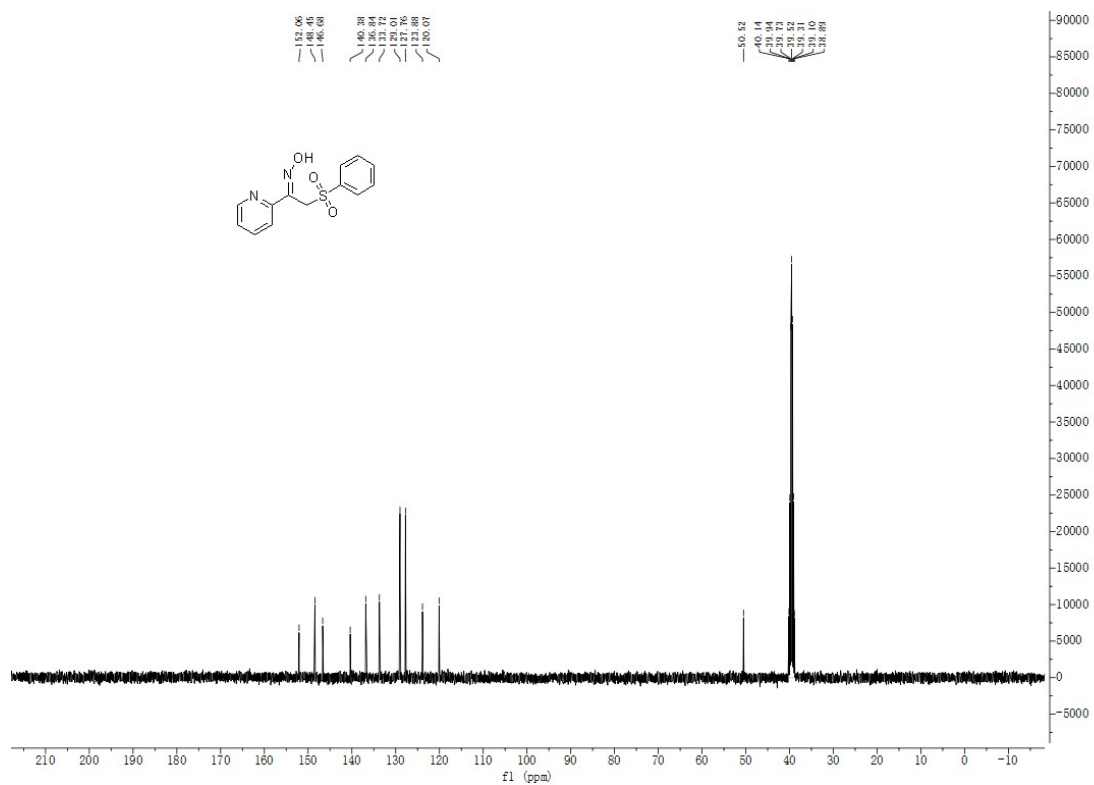
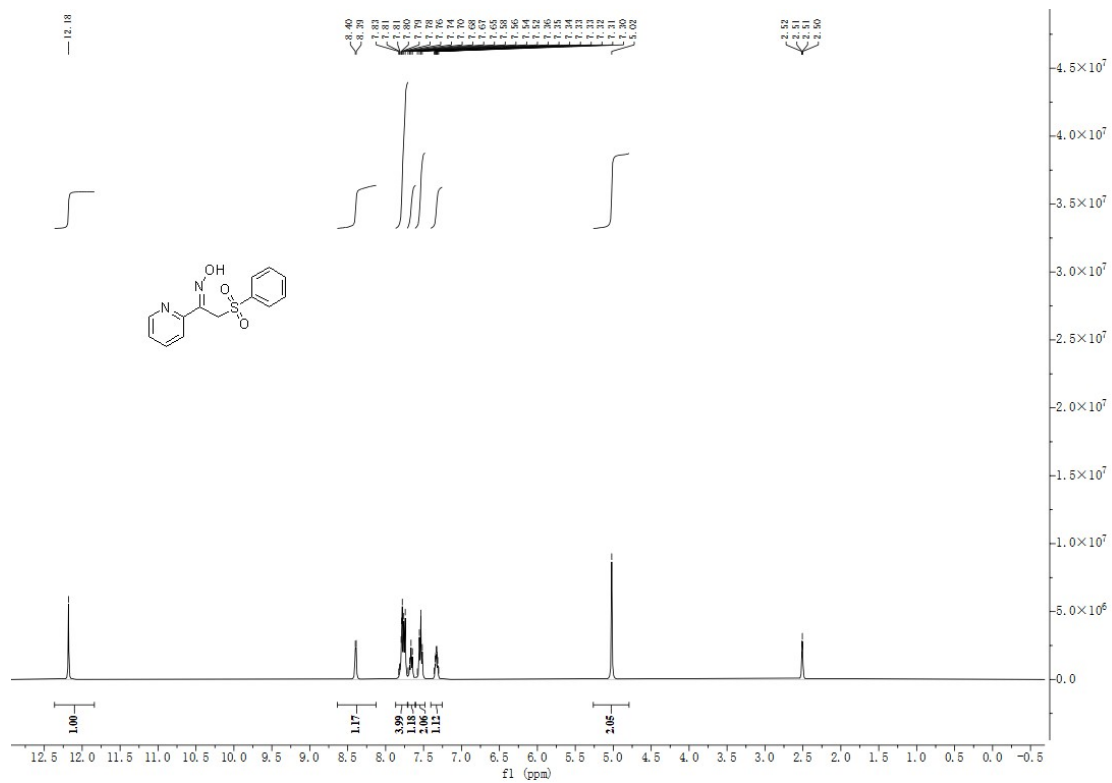
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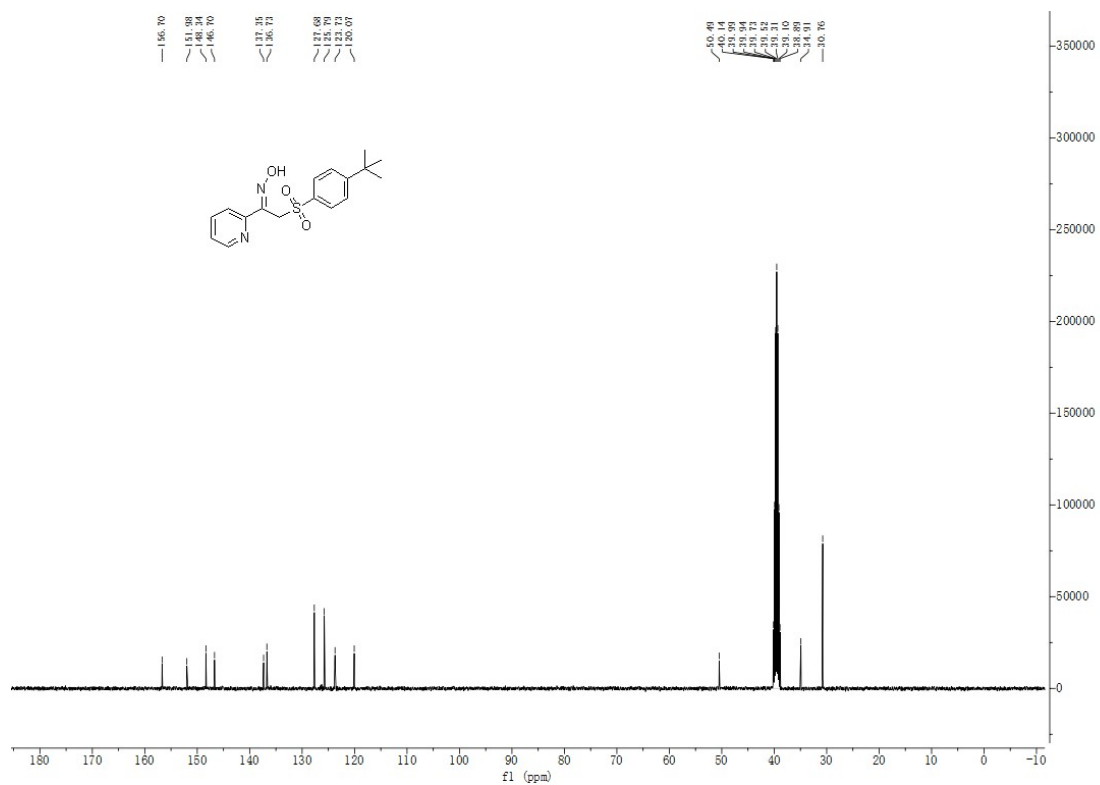
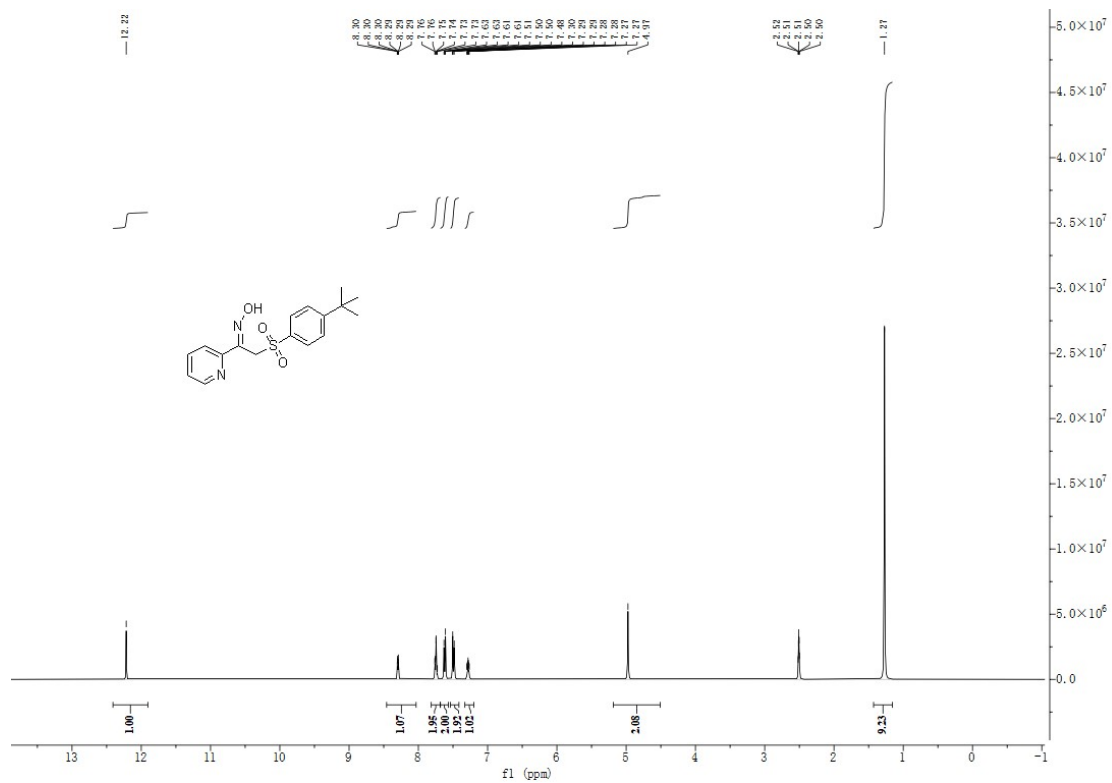
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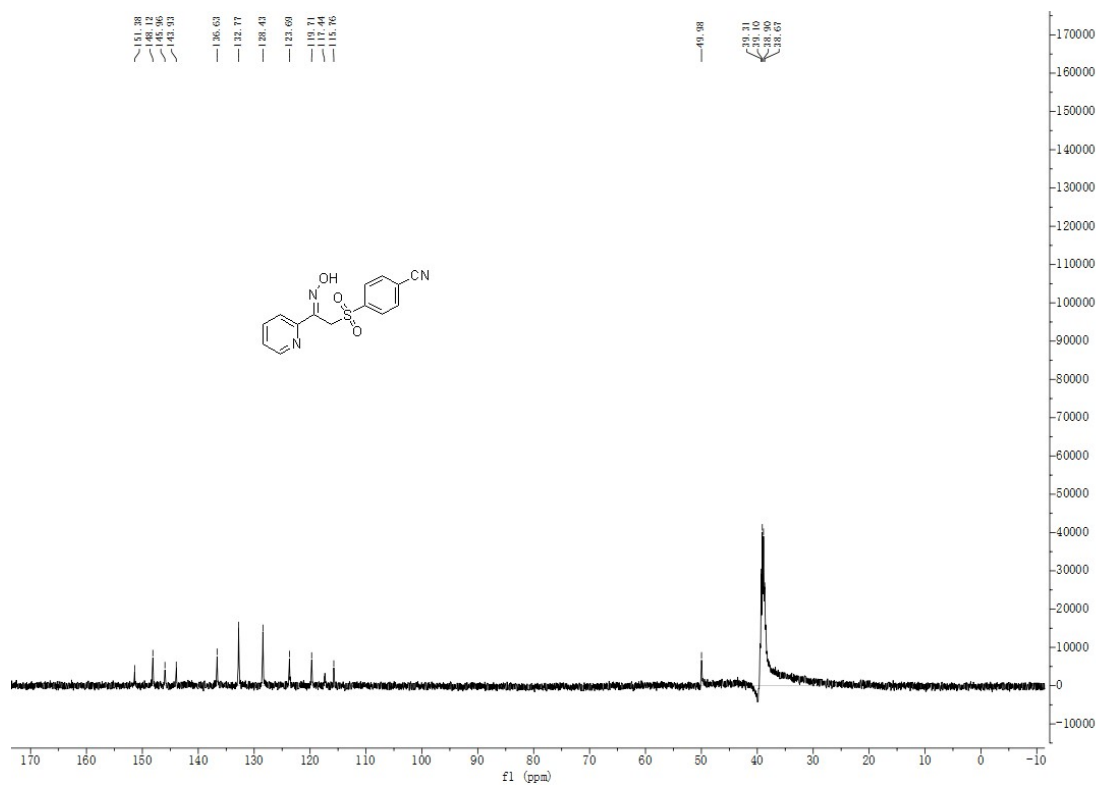
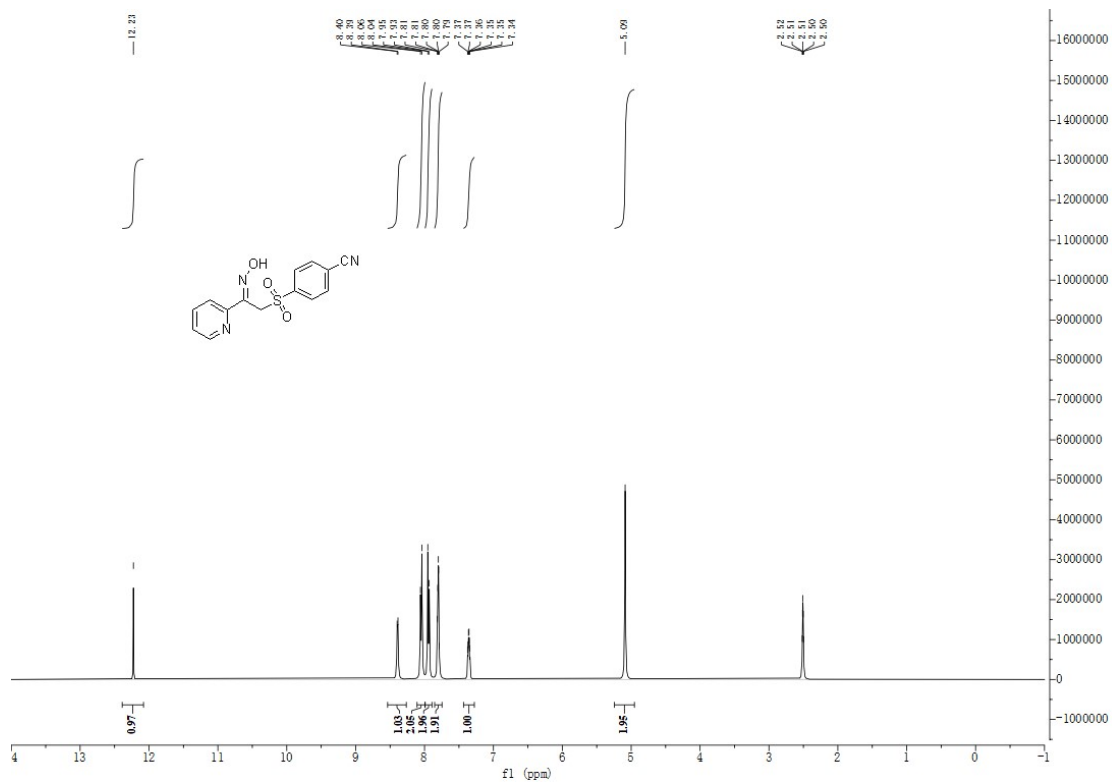
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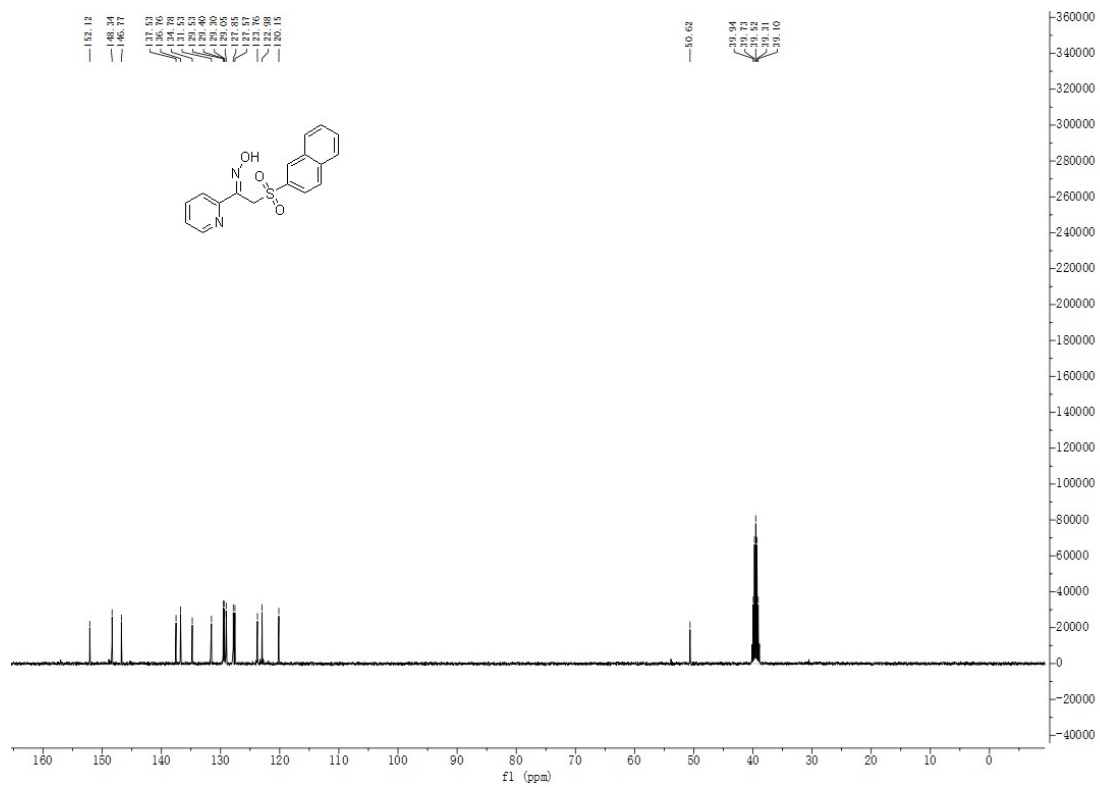
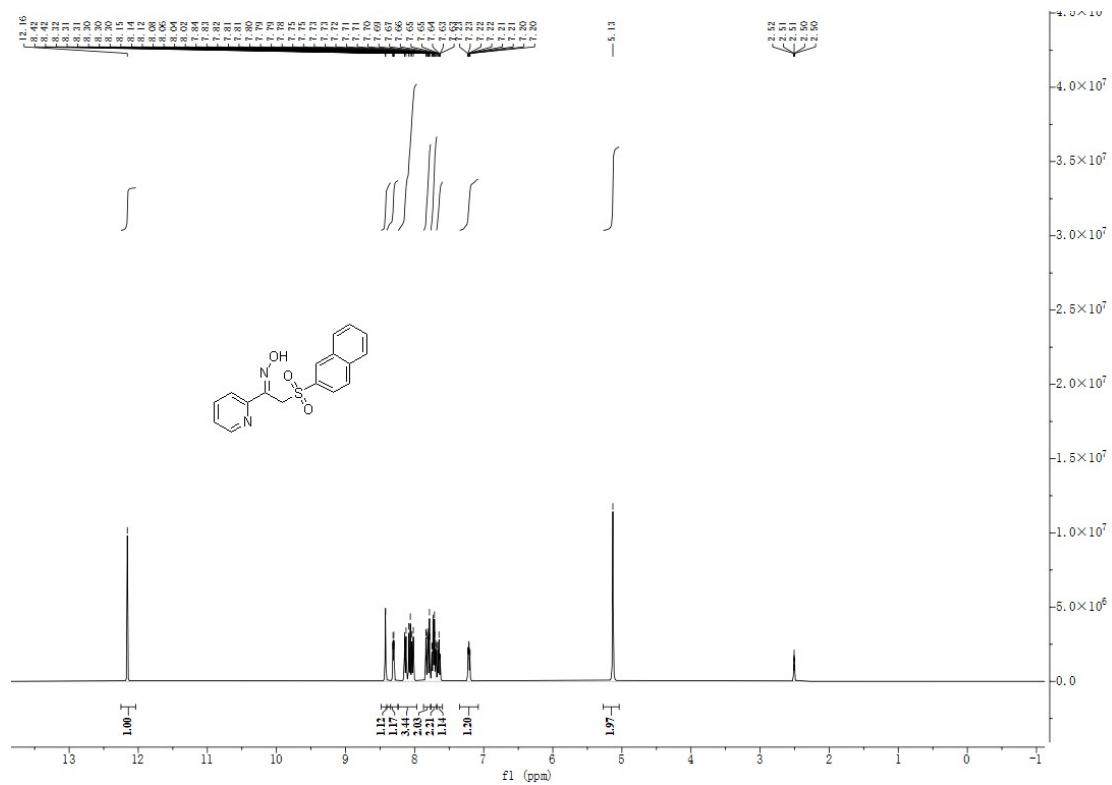
3f $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$), $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$)



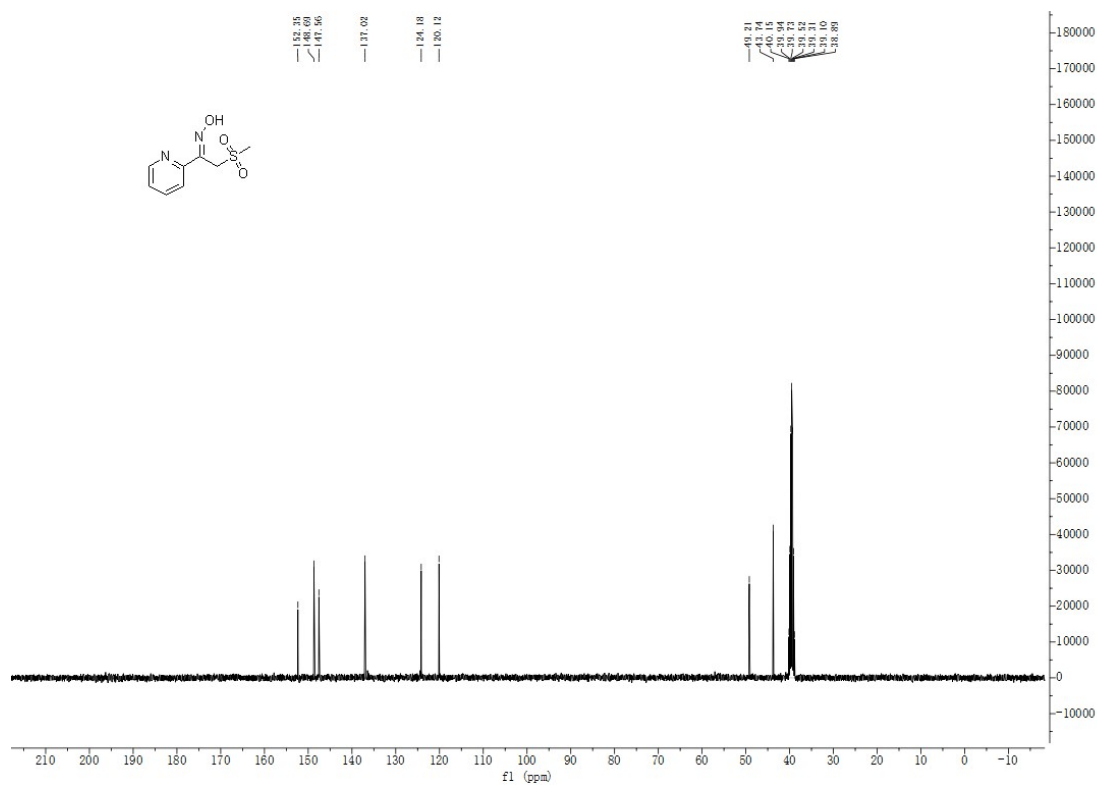
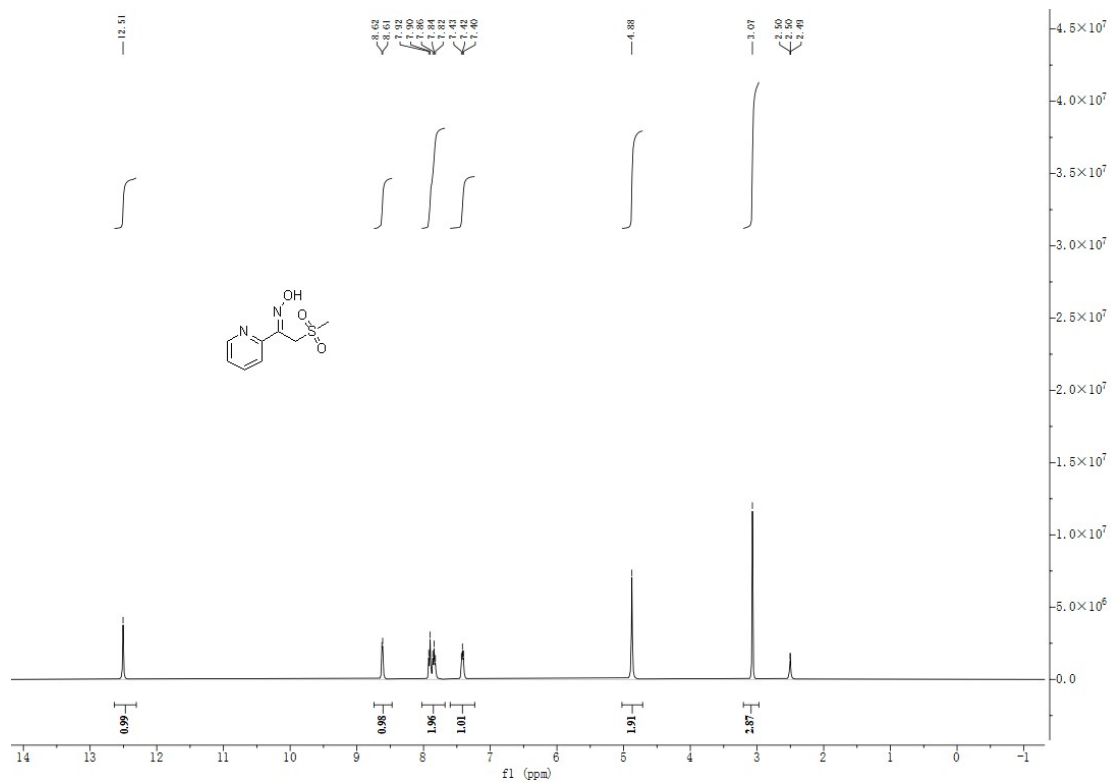
3g ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



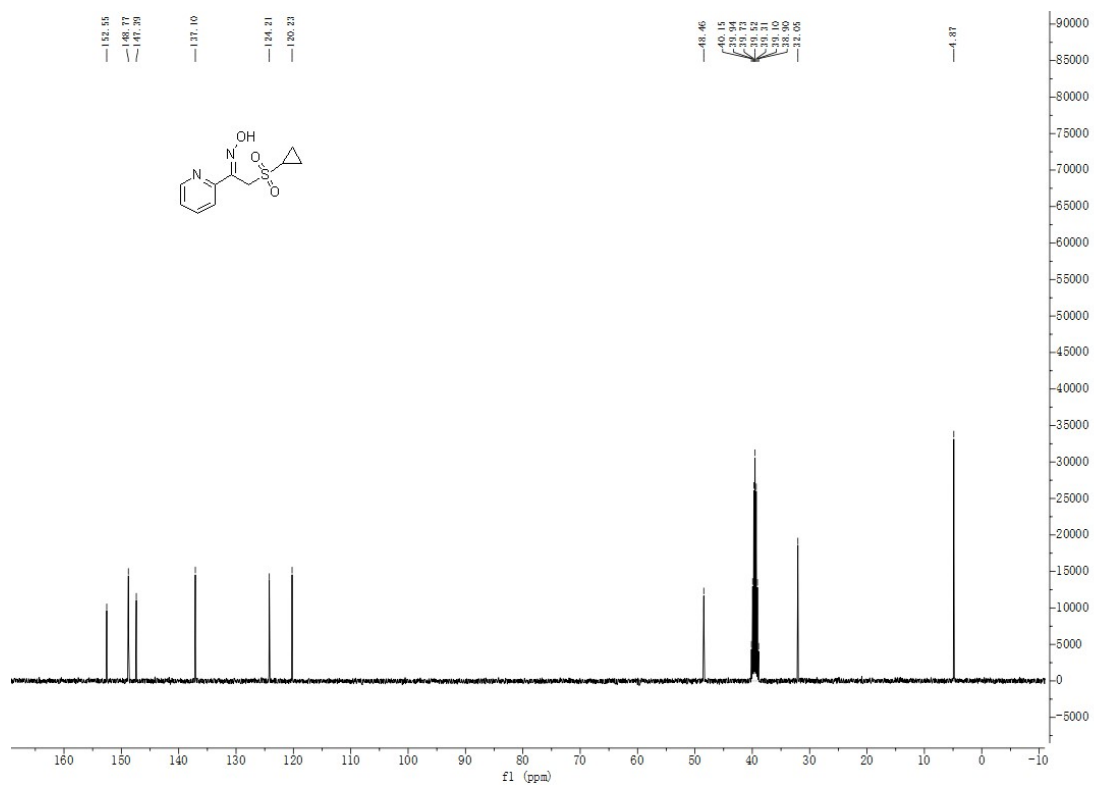
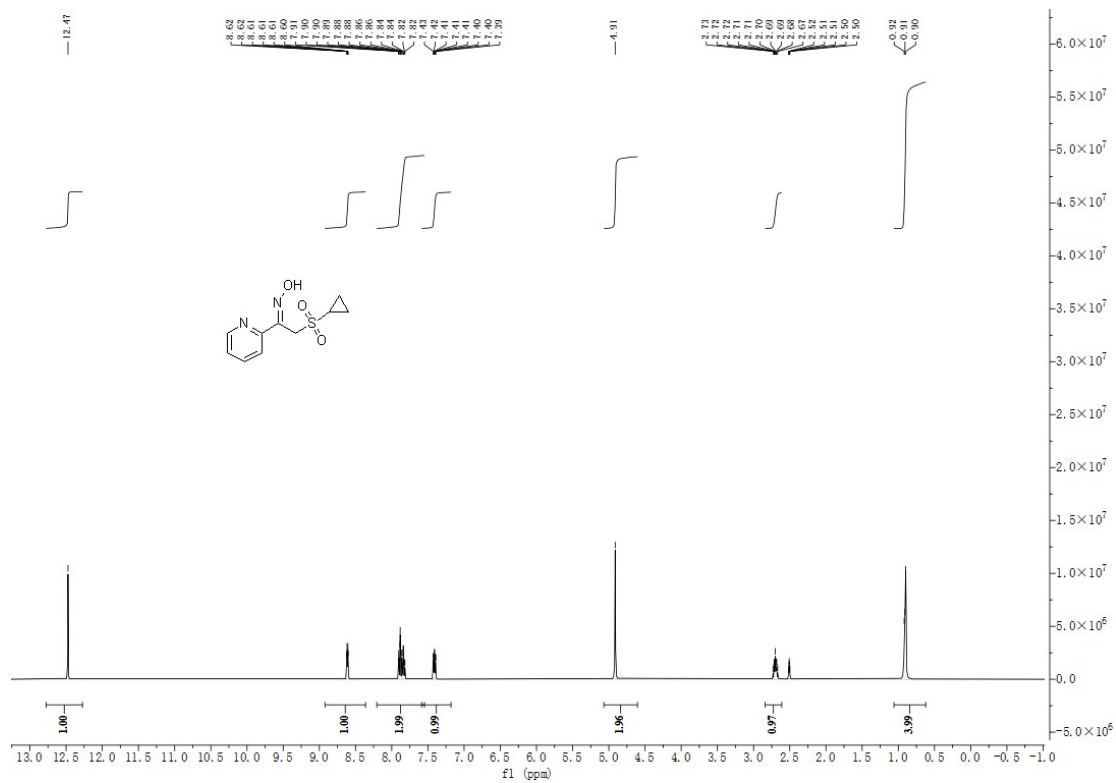
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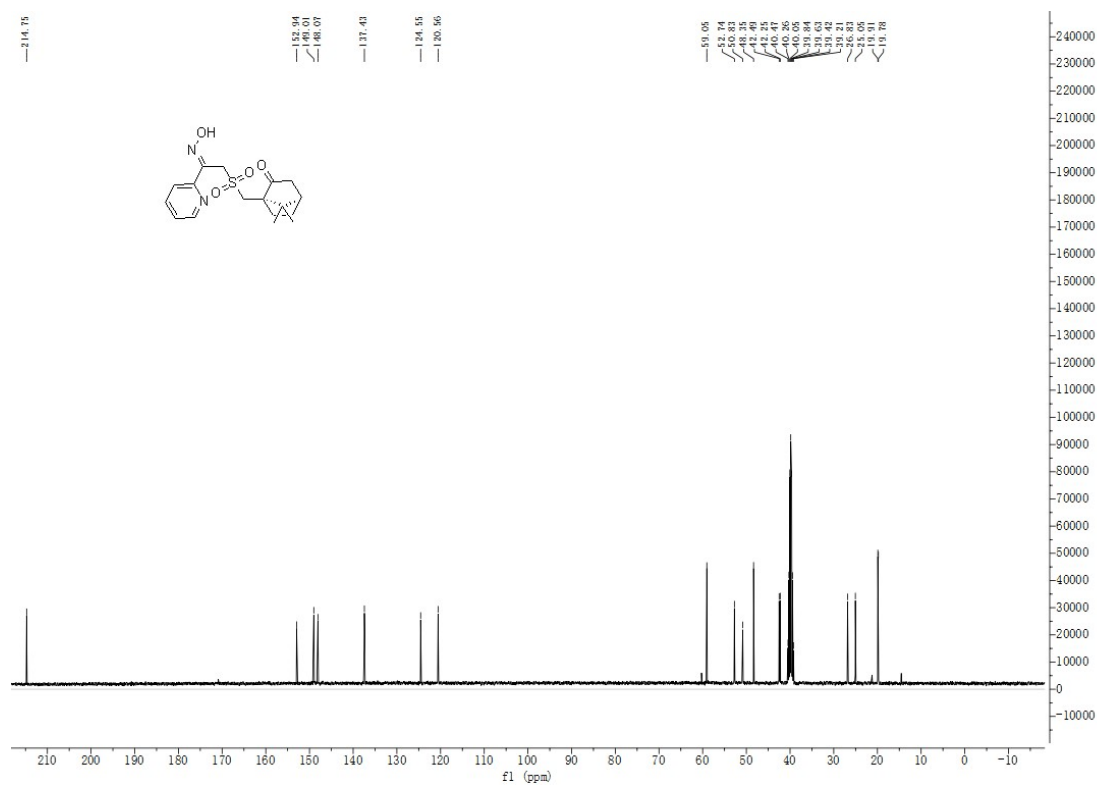
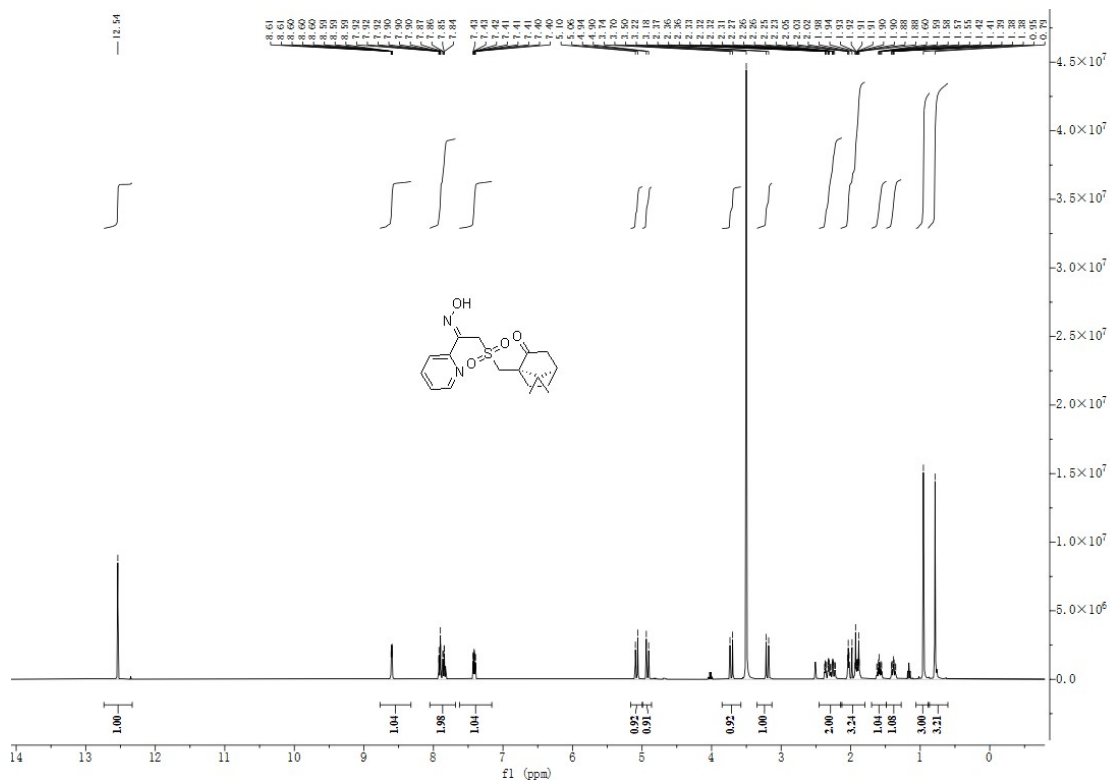
3i $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$), $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$)



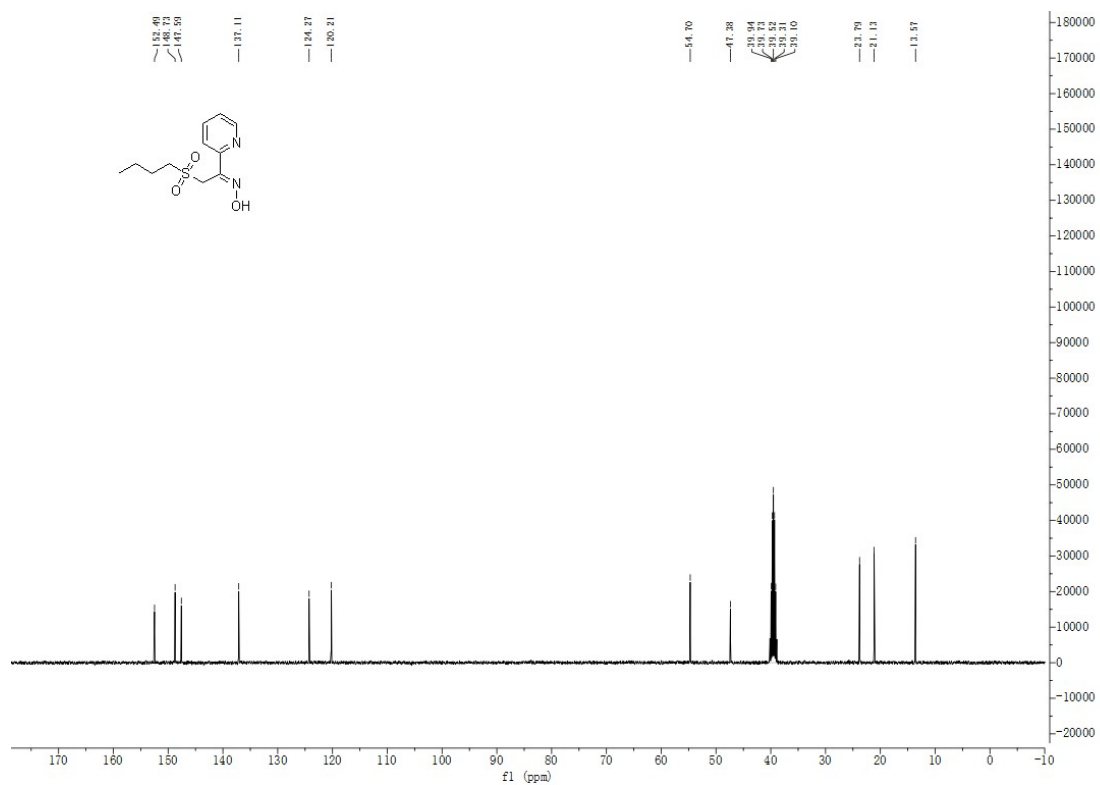
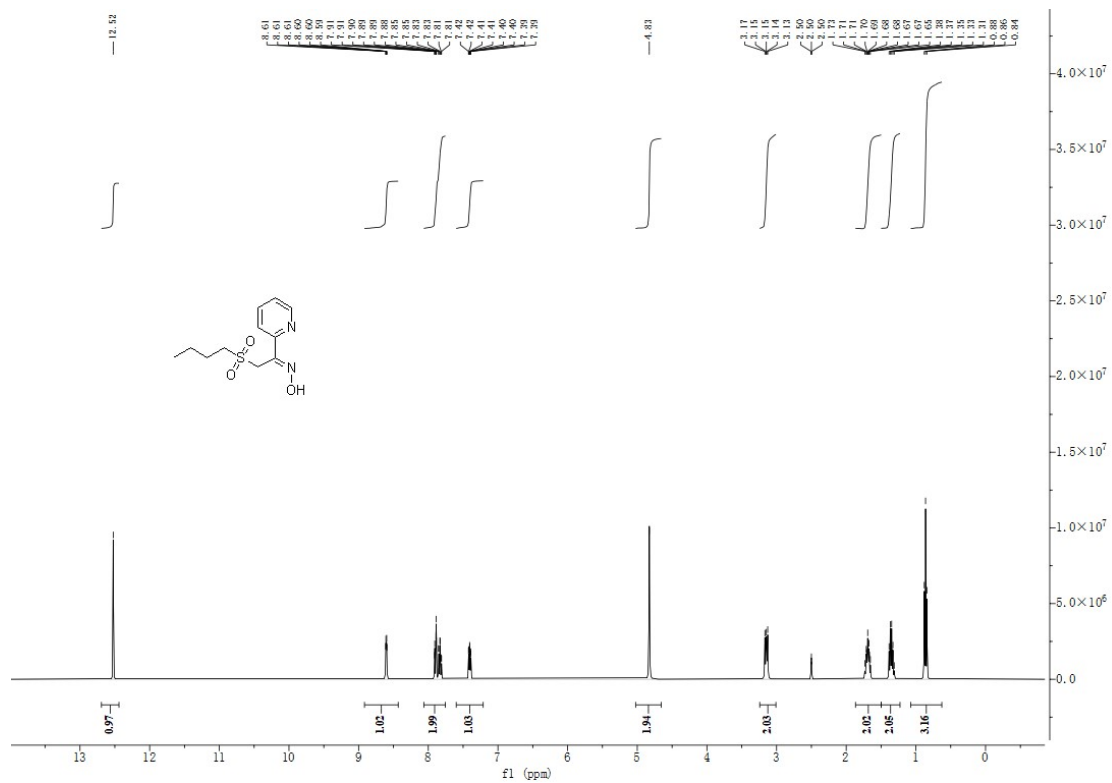
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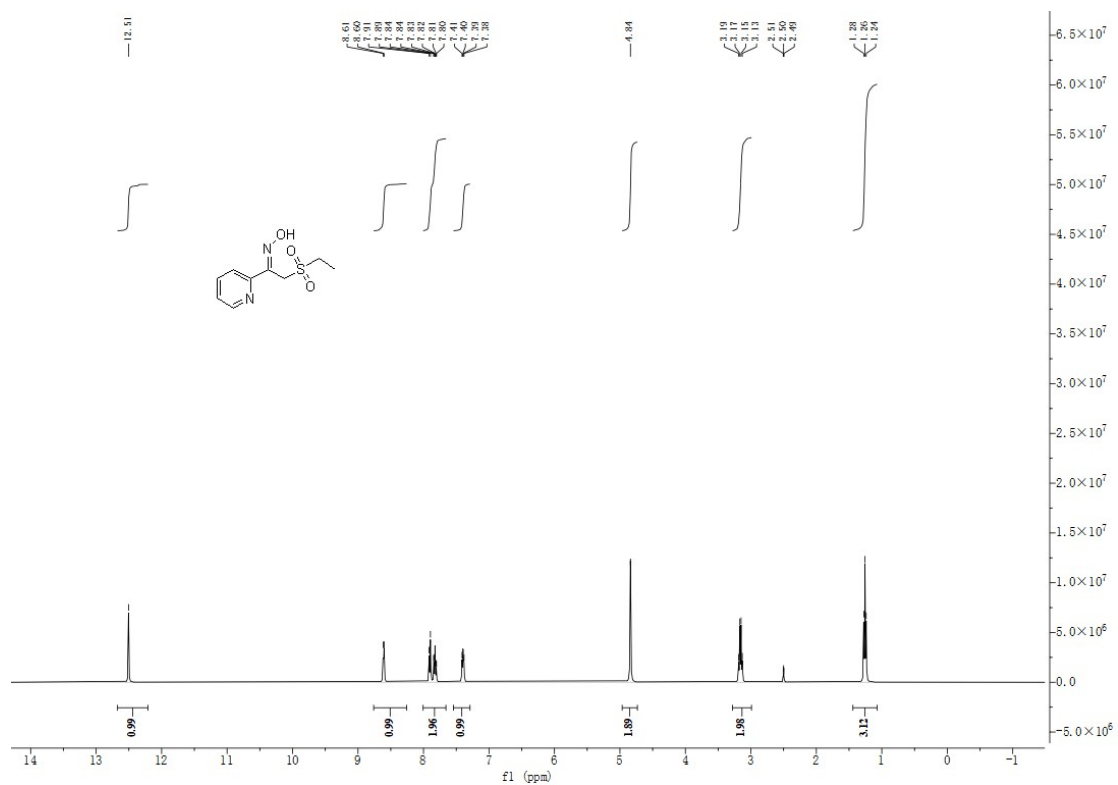
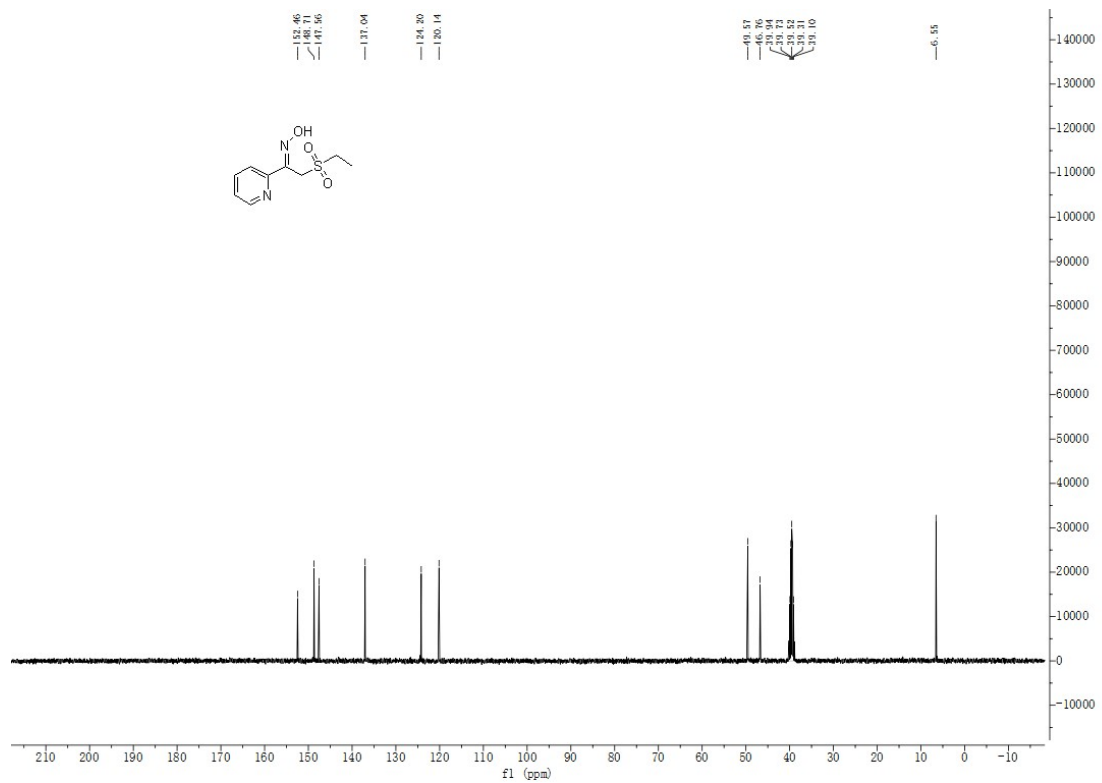
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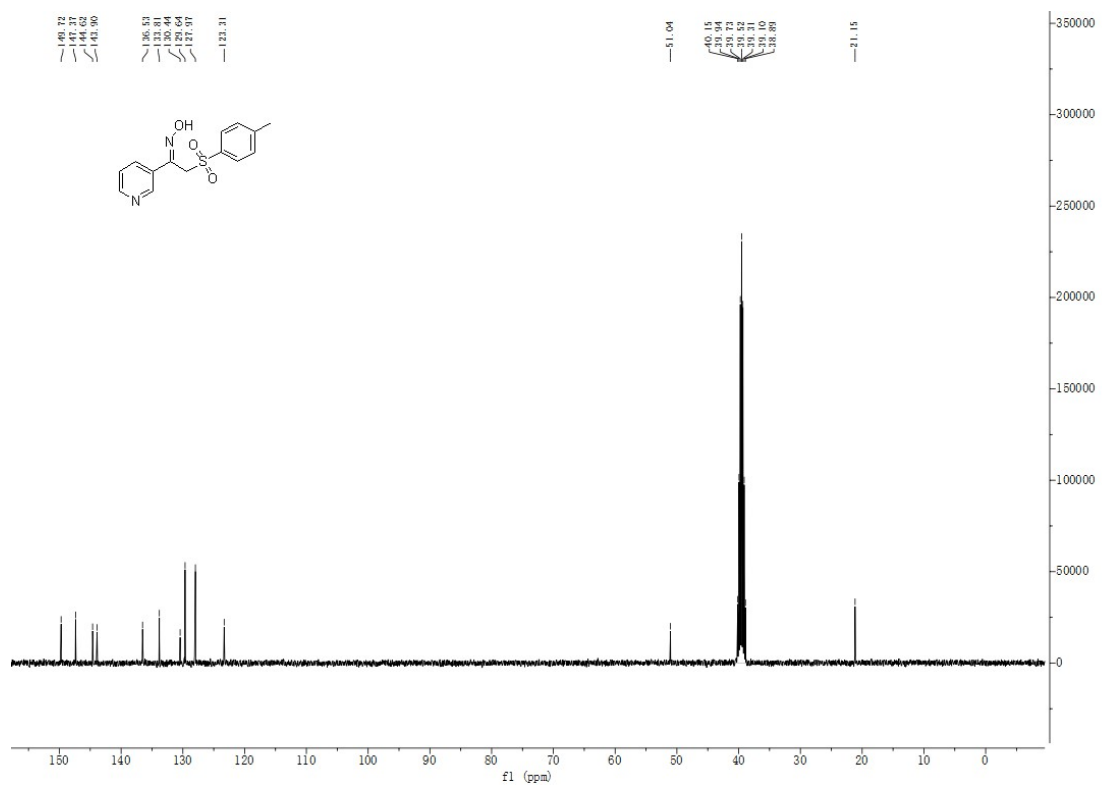
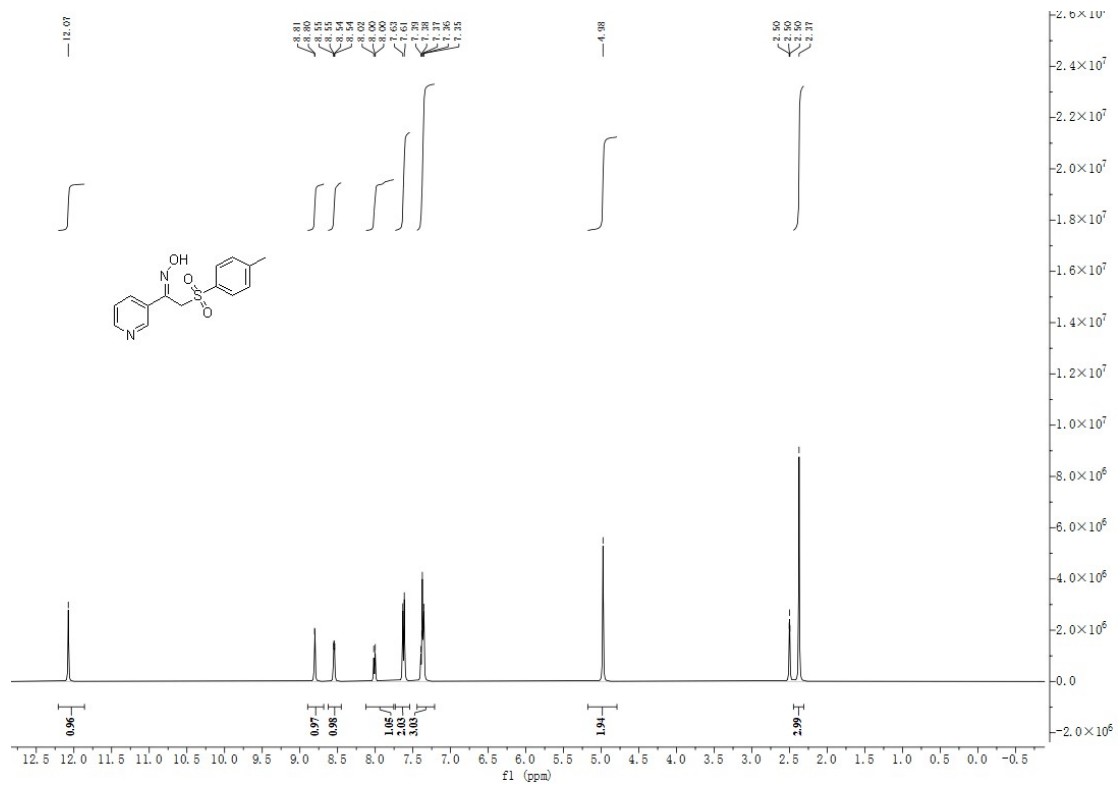
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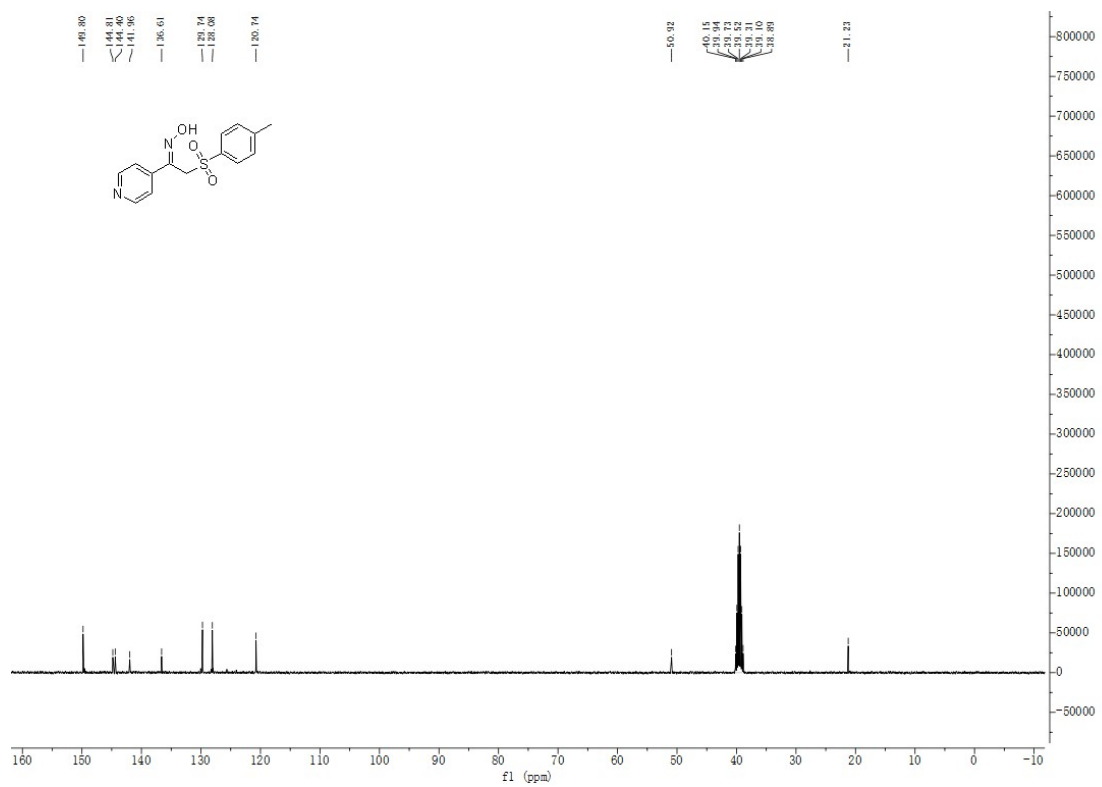
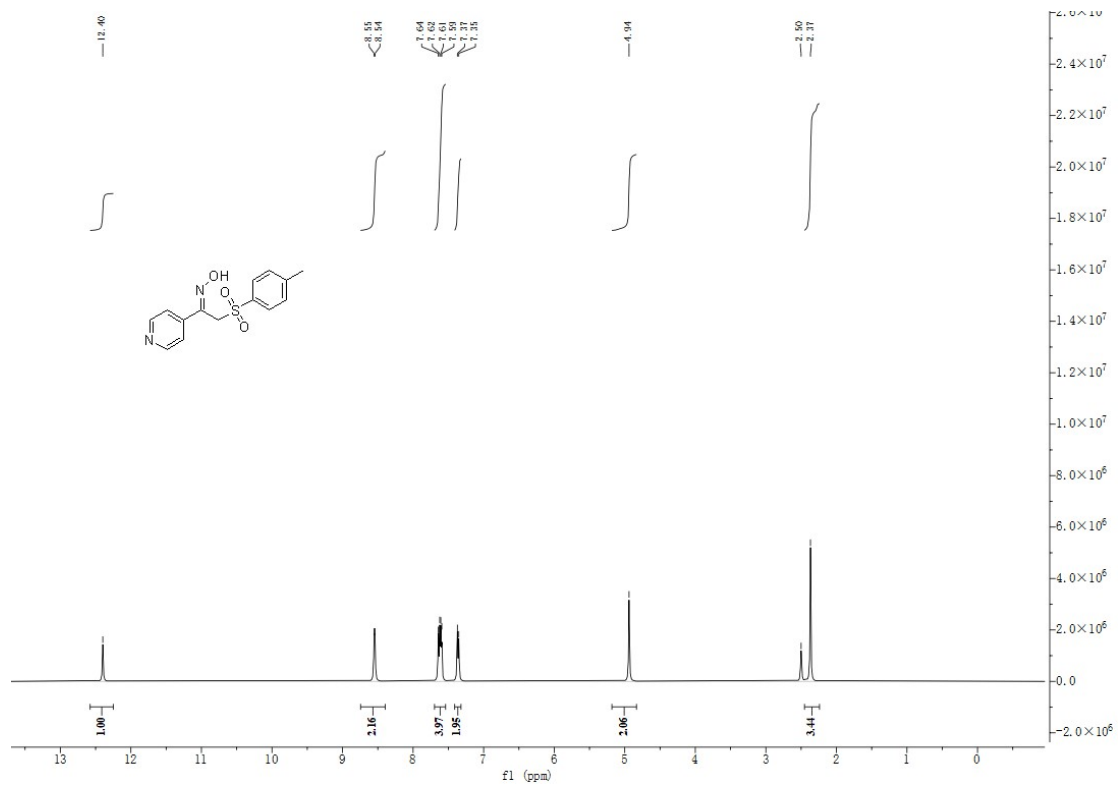
3m ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



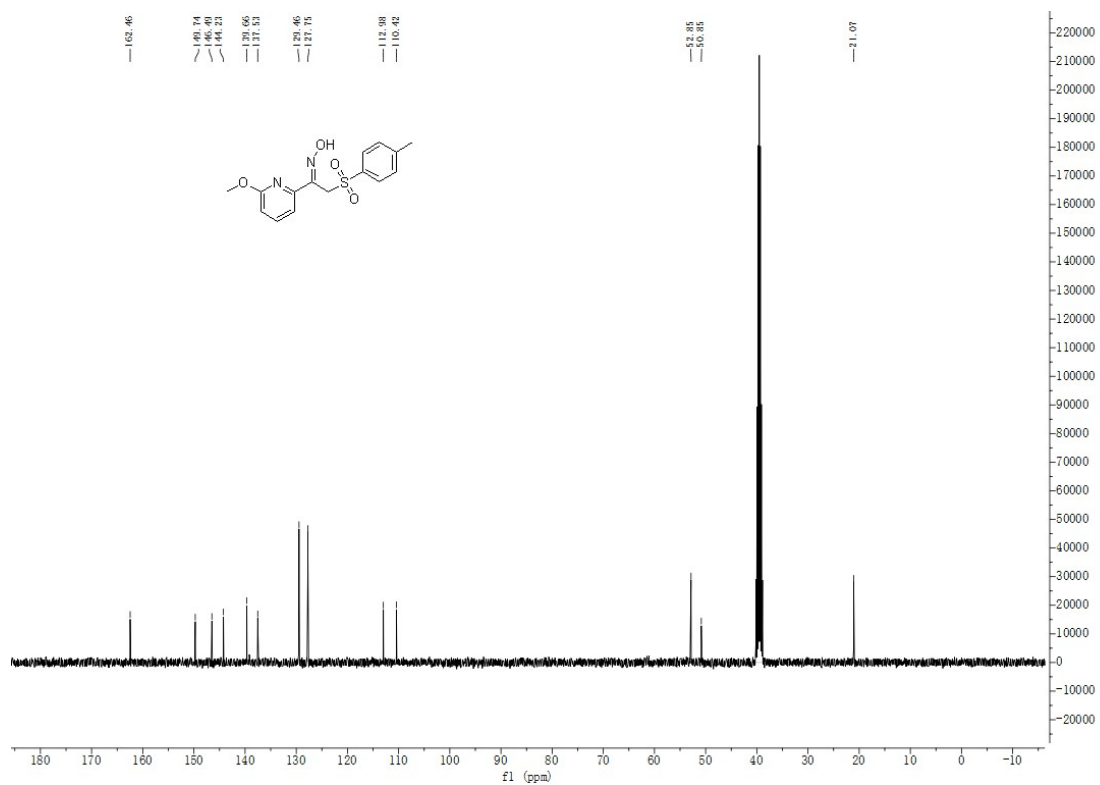
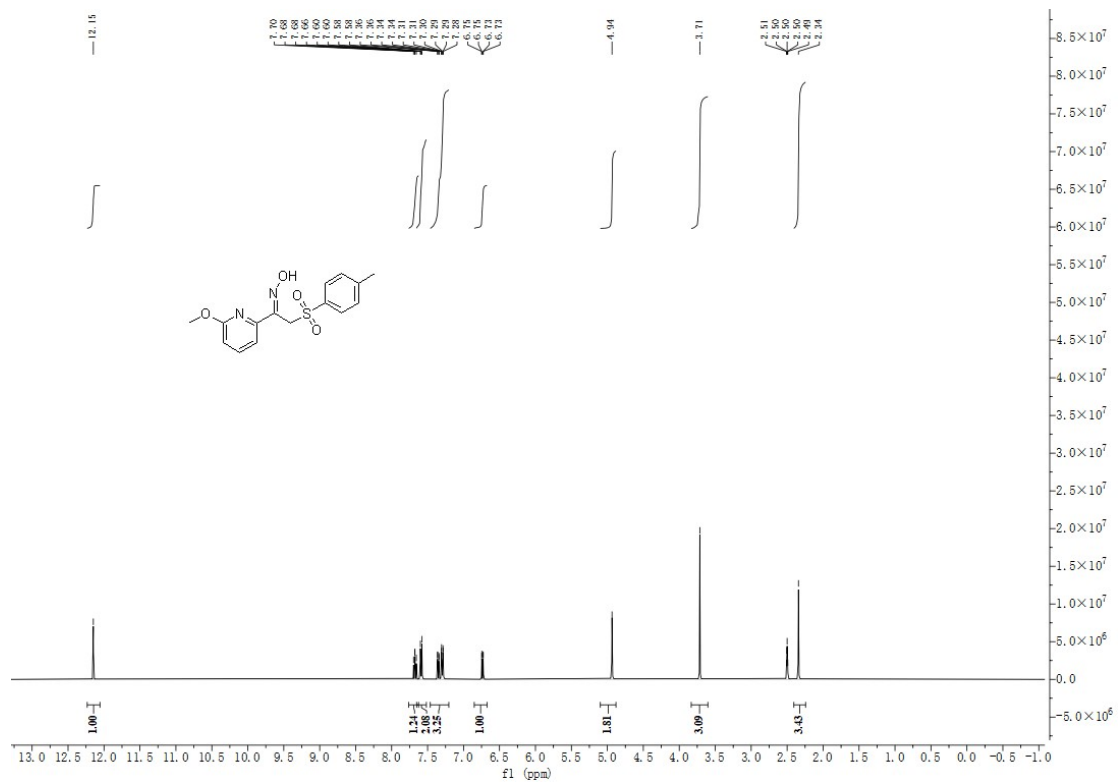
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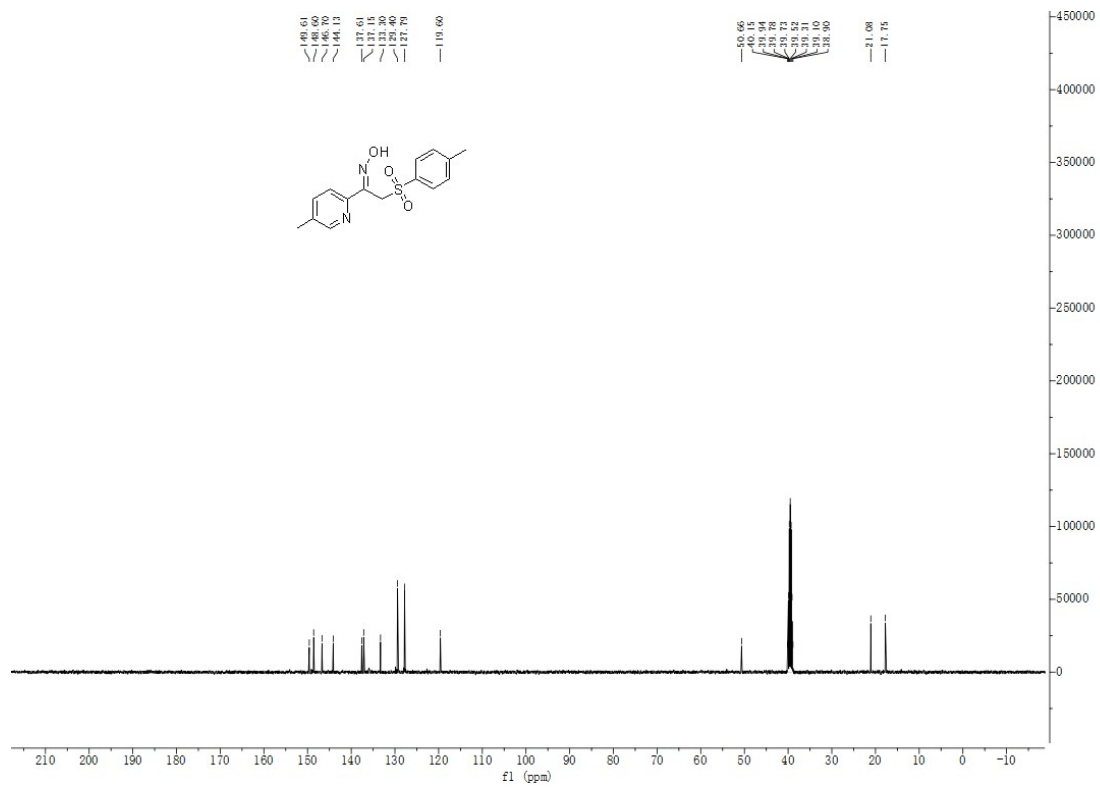
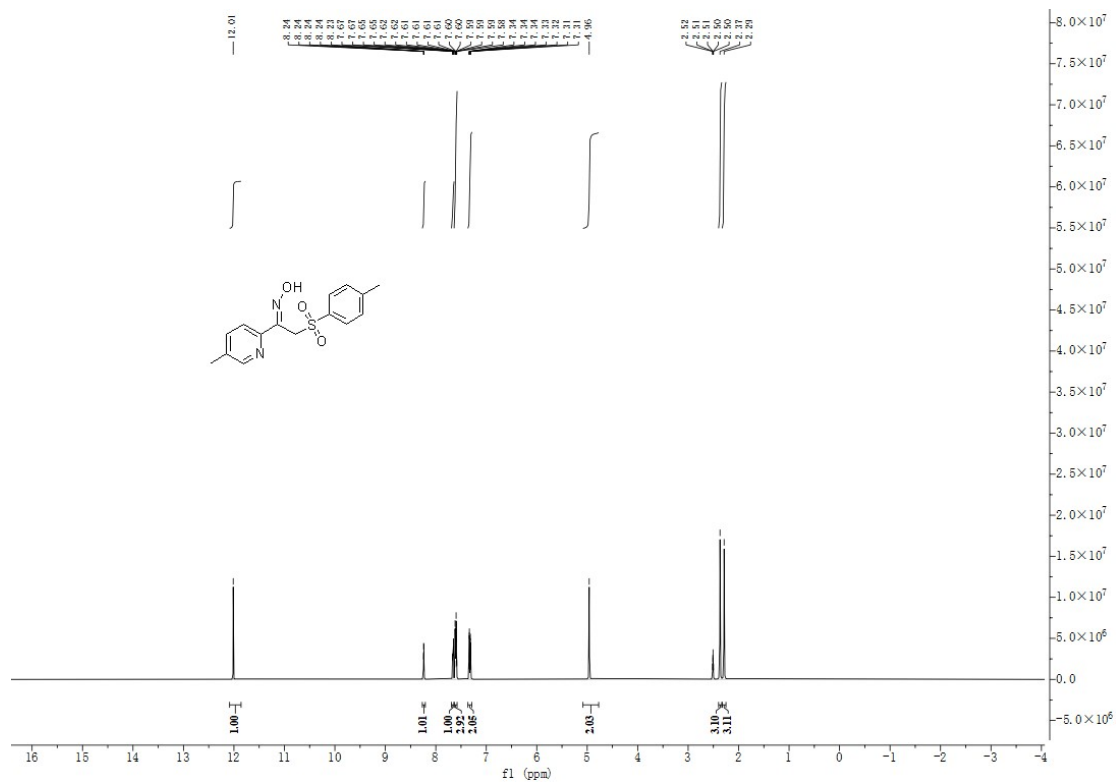
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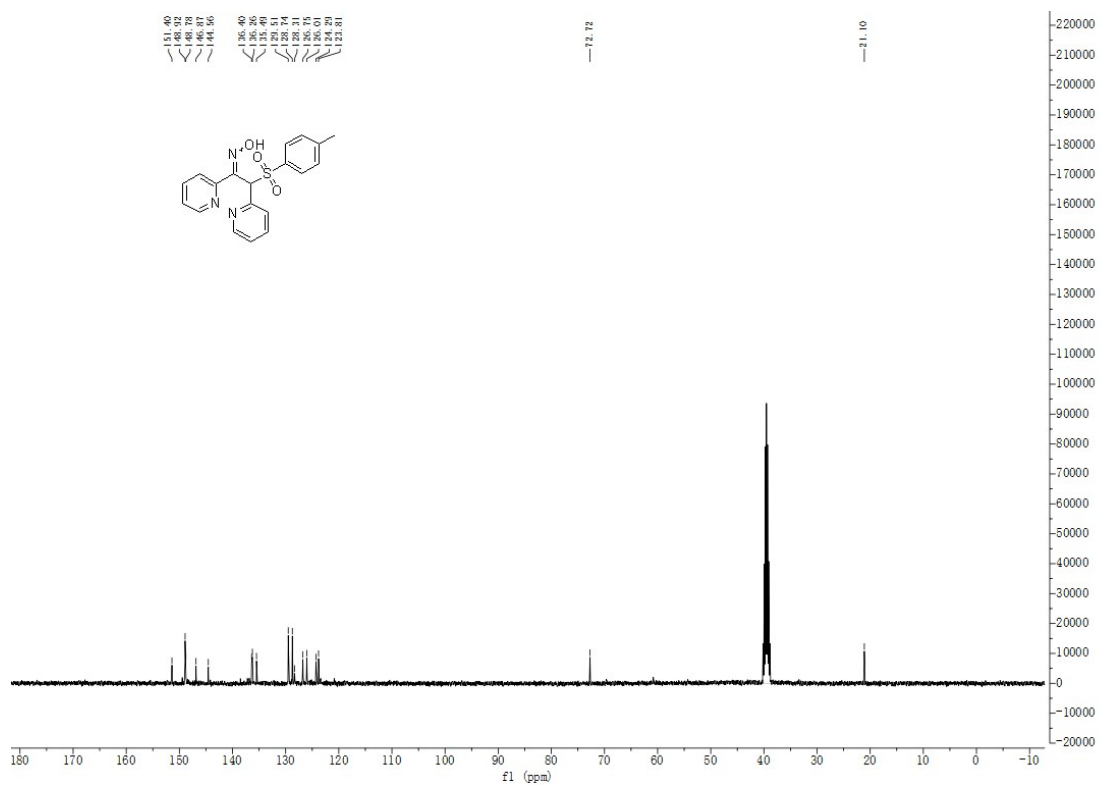
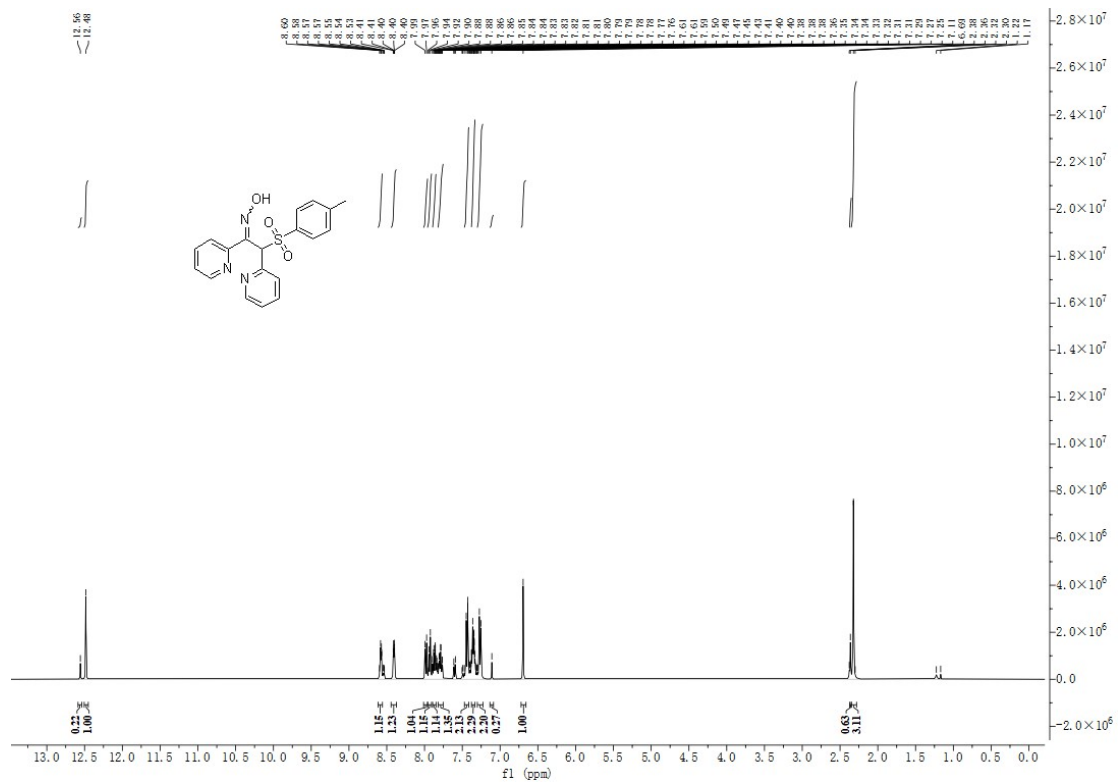
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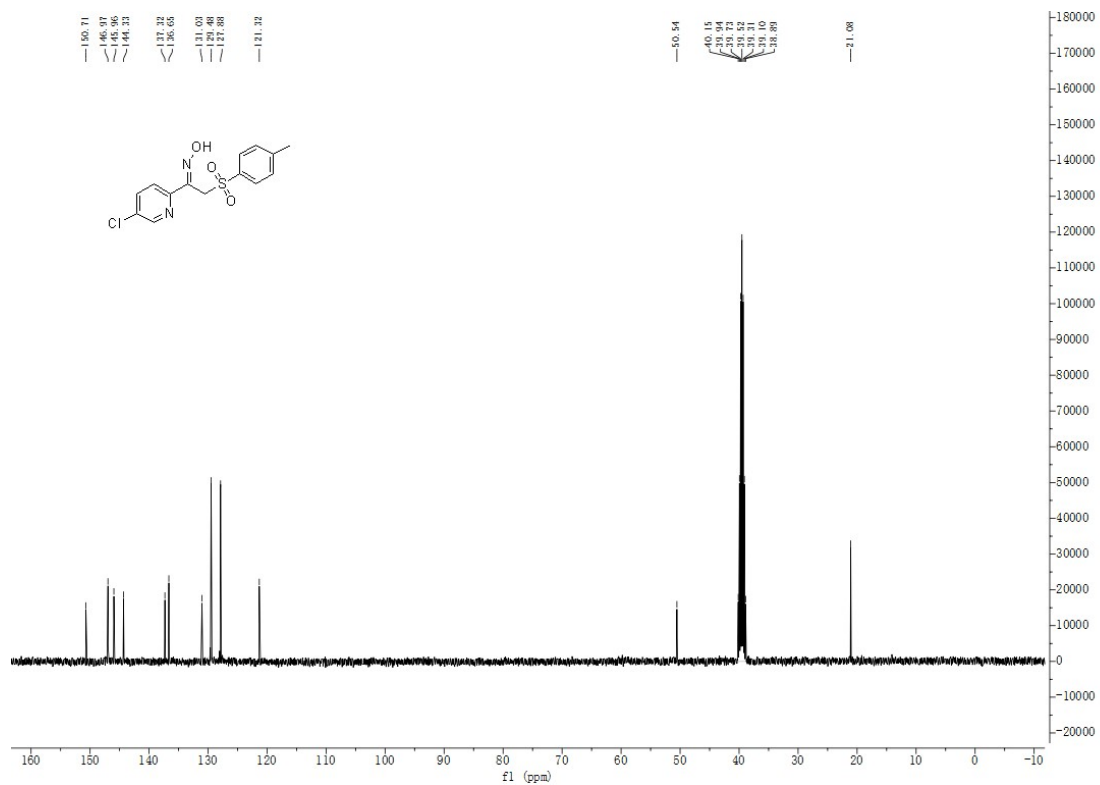
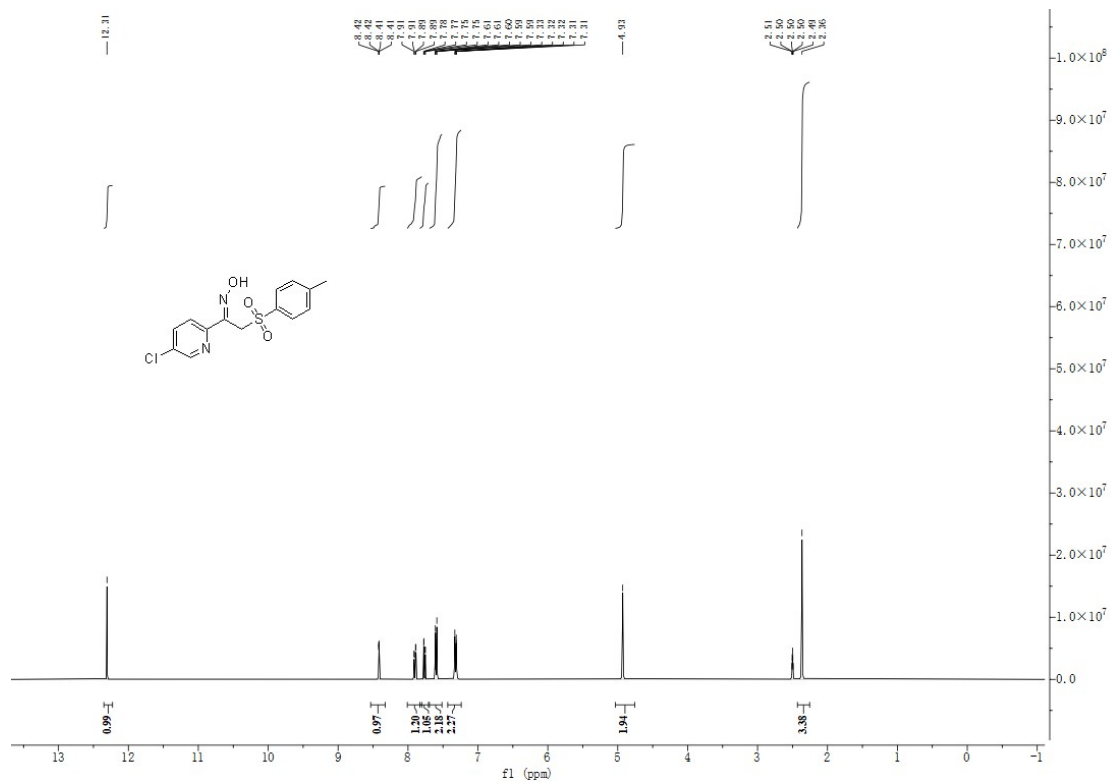
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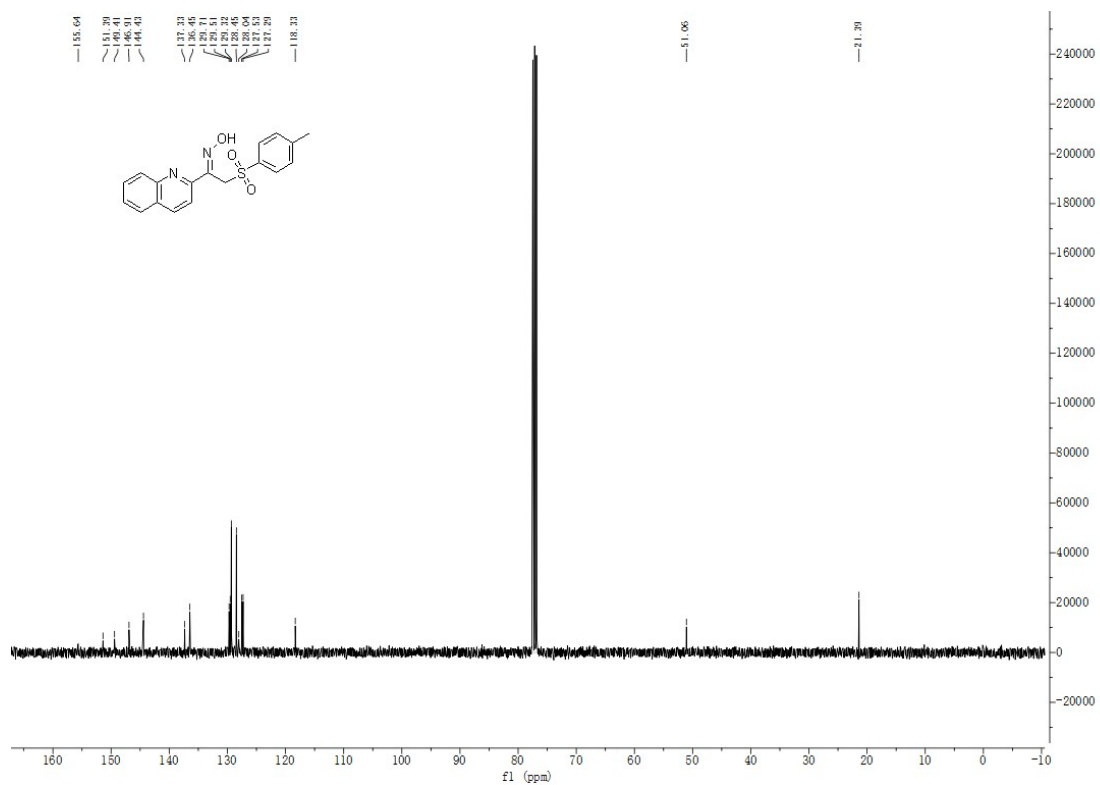
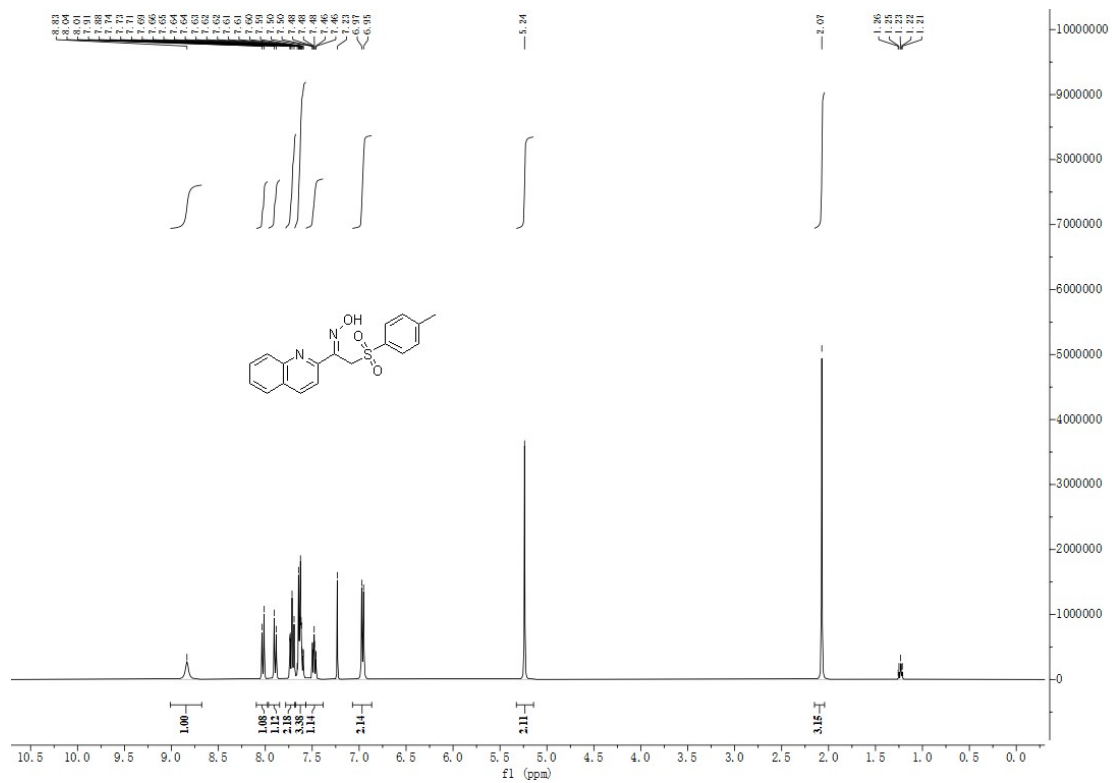
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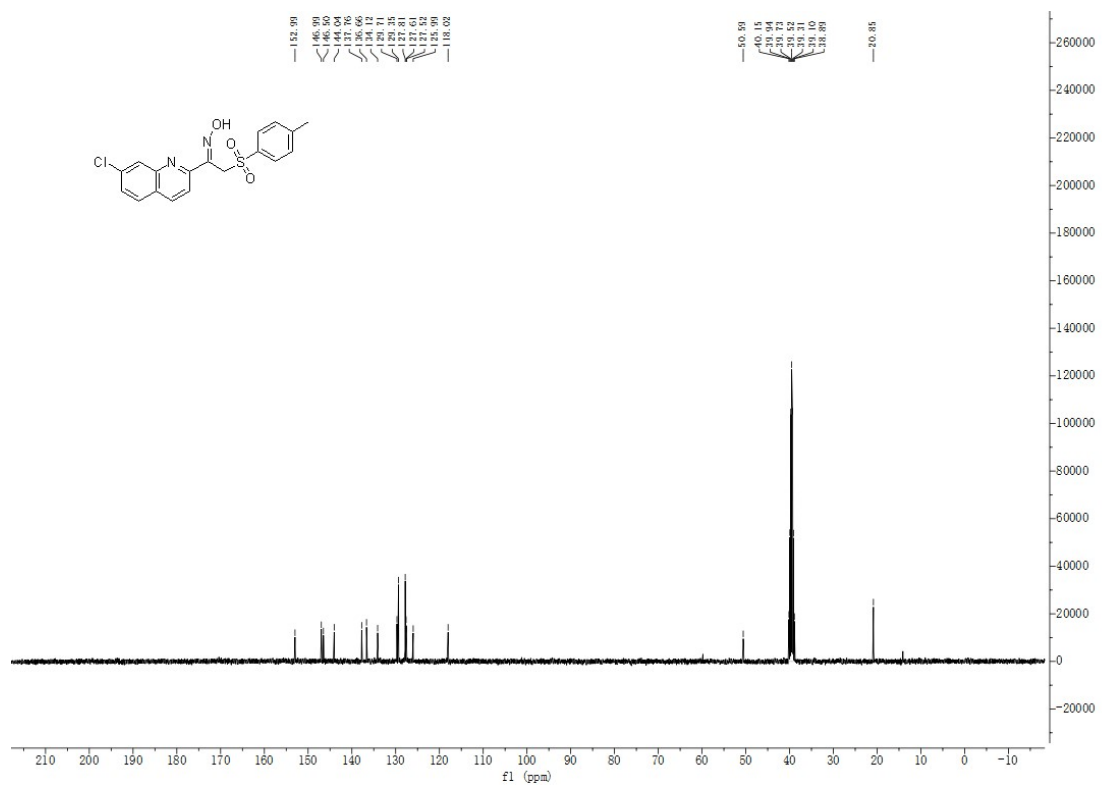
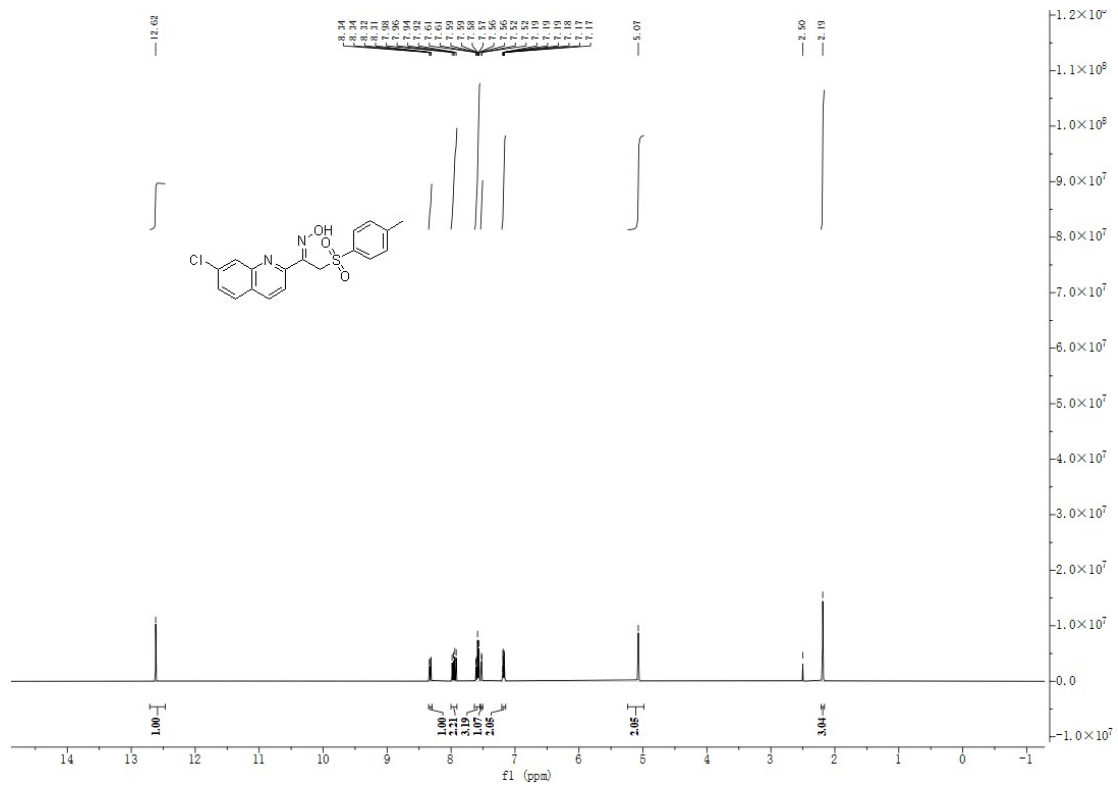
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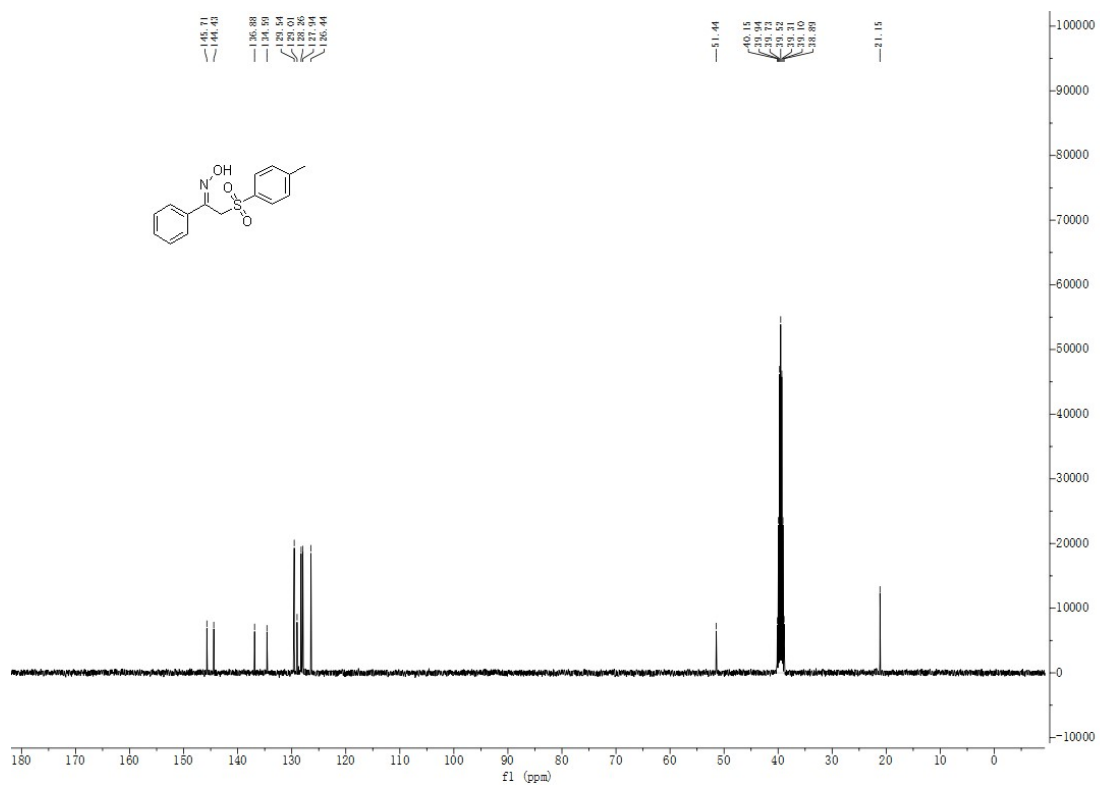
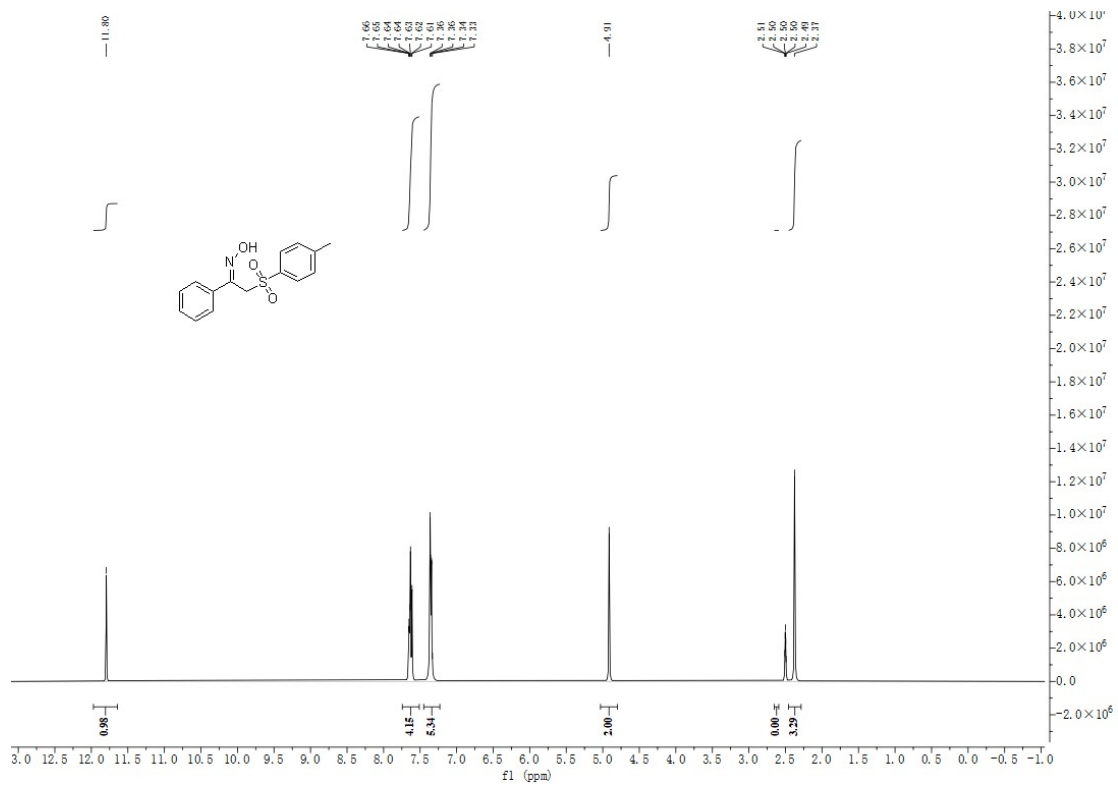
3t $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$), $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$)



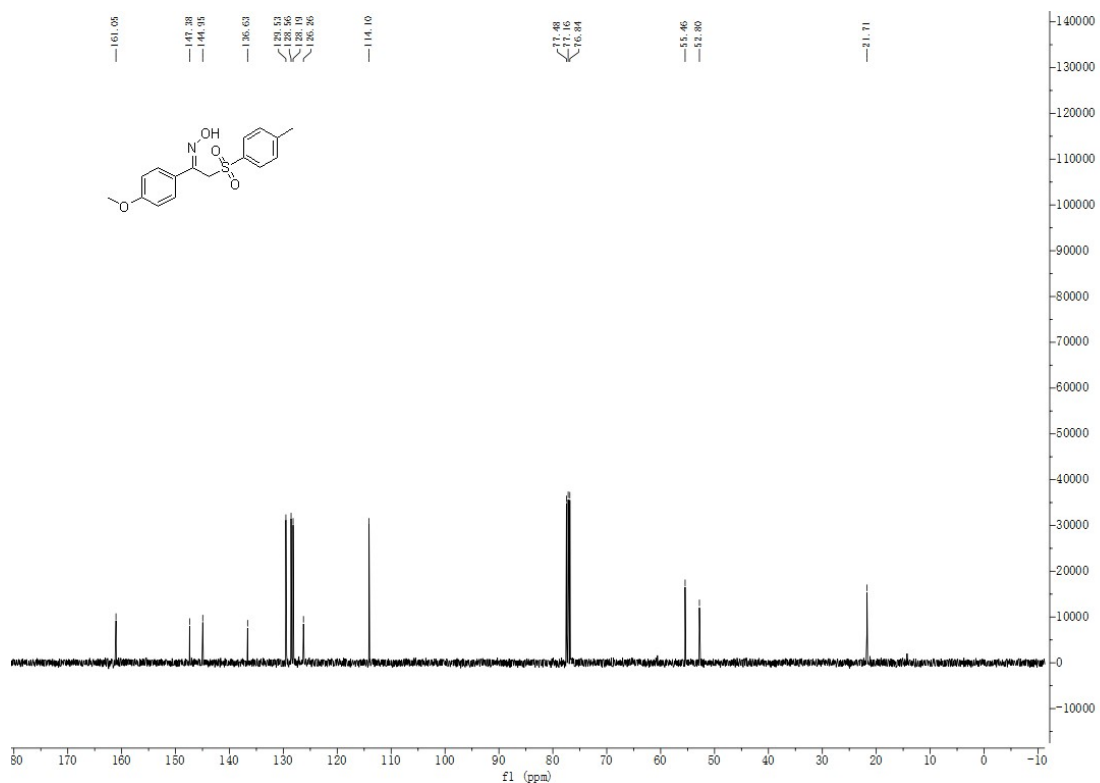
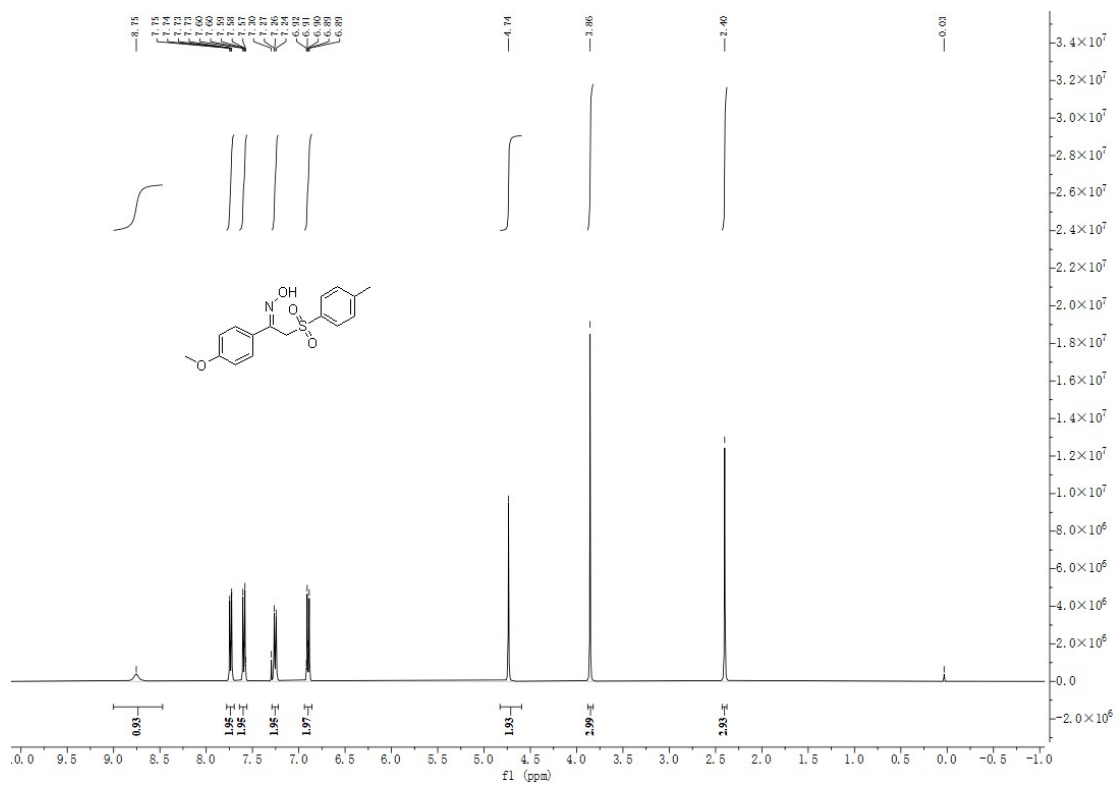
3u ¹H NMR (400 MHz, Chloroform-*d*), ¹³C NMR (101 MHz, Chloroform-*d*)



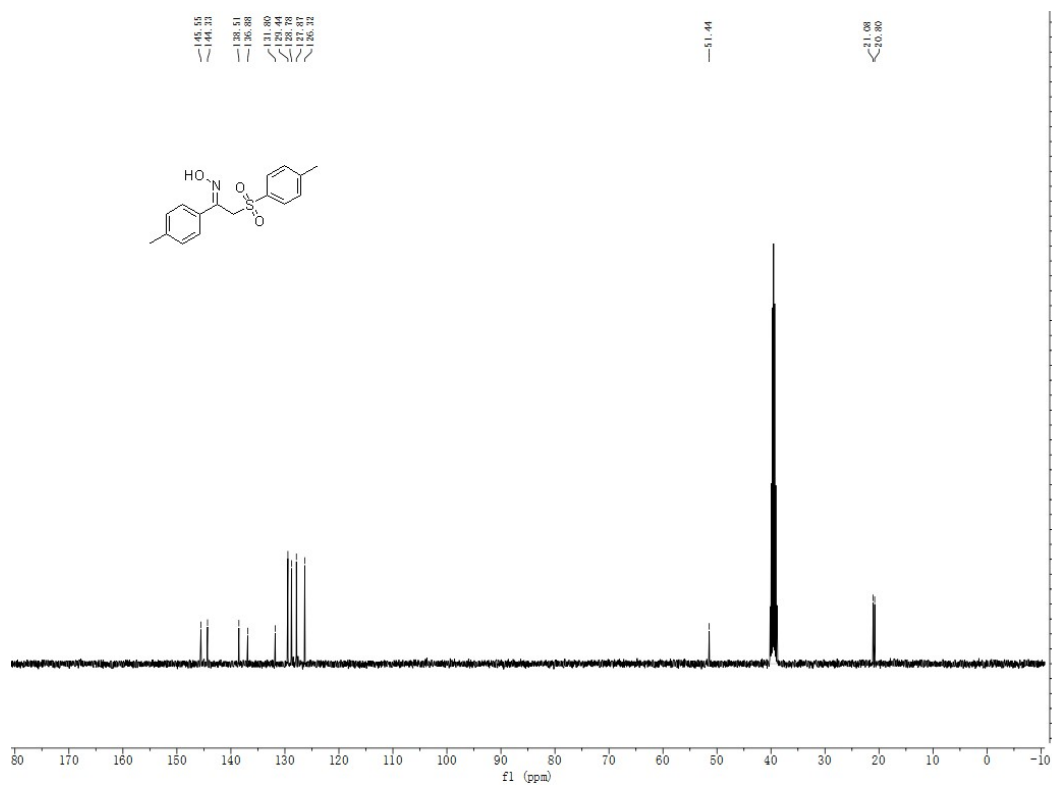
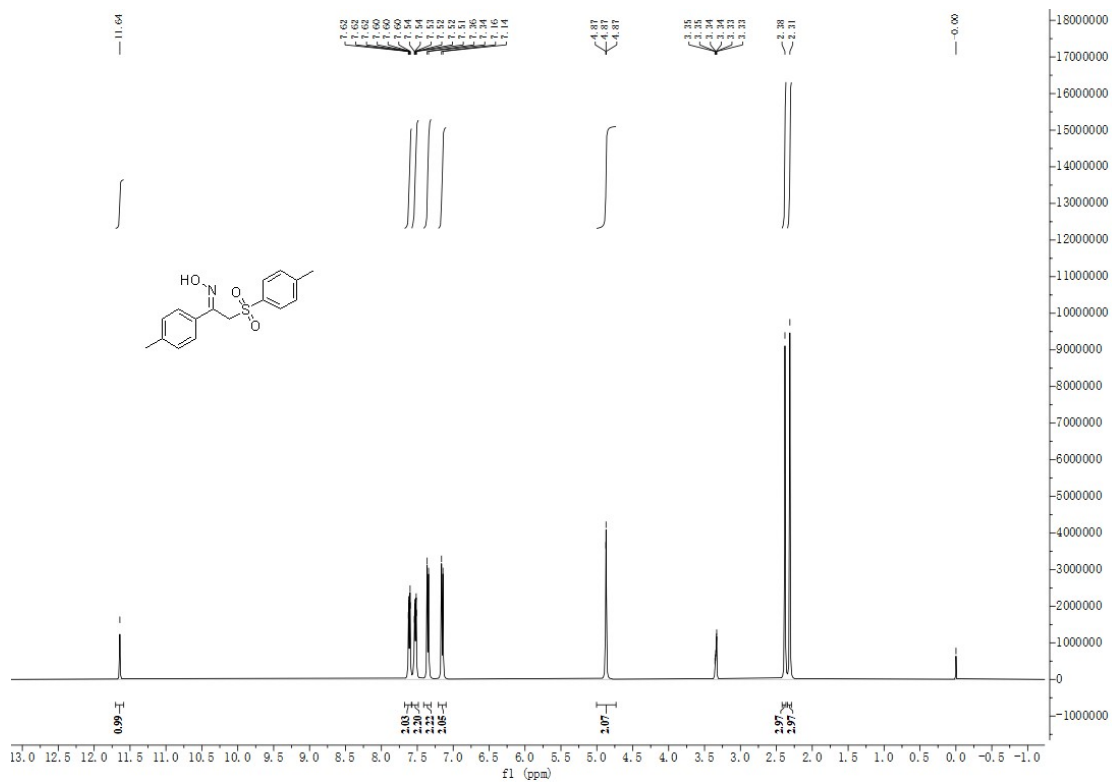
3v ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



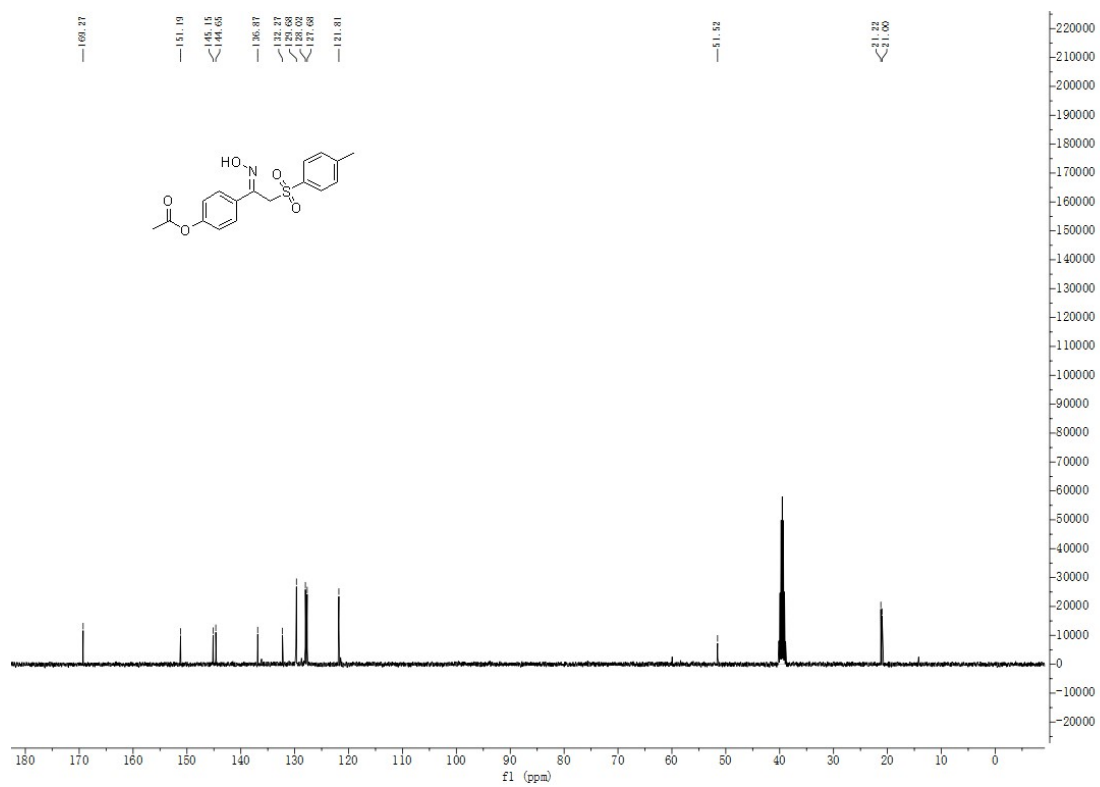
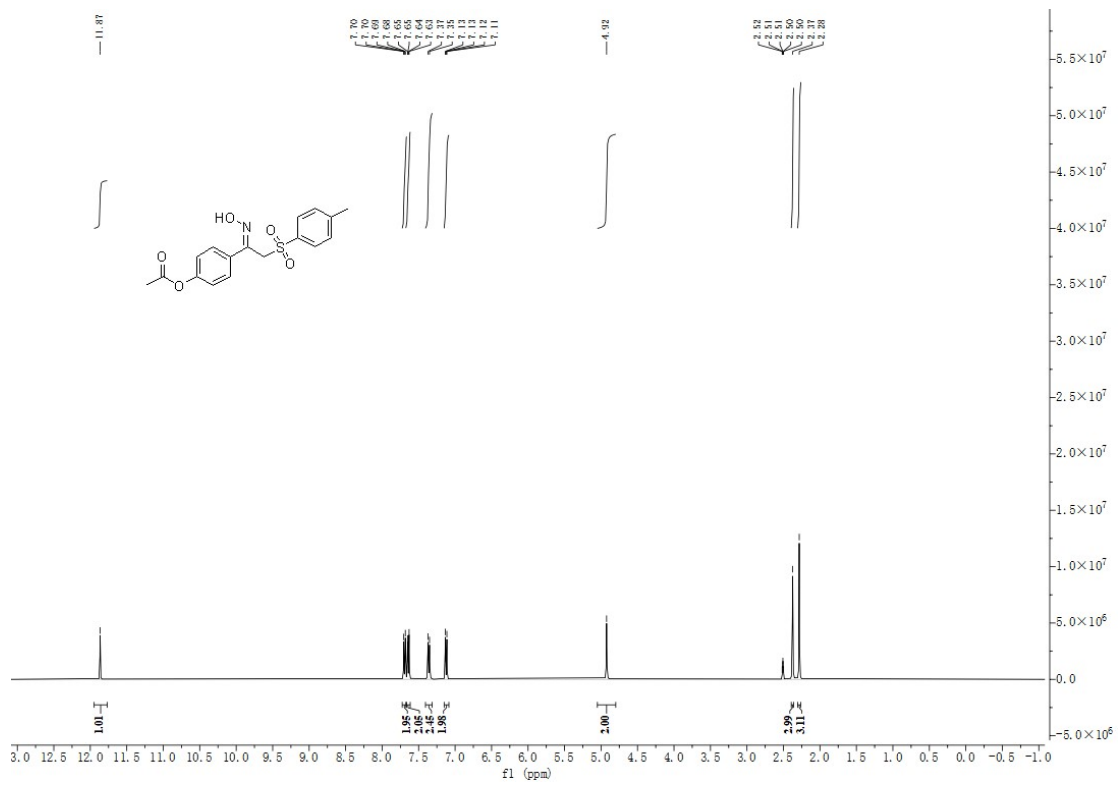
5a ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



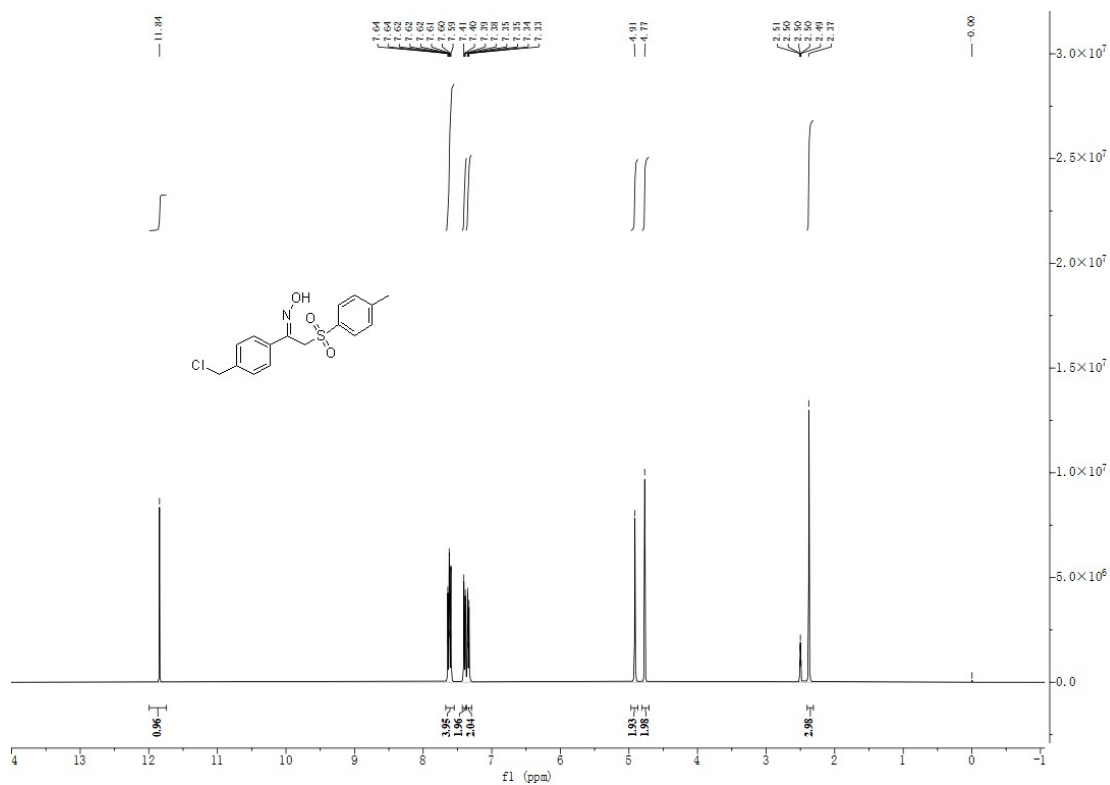
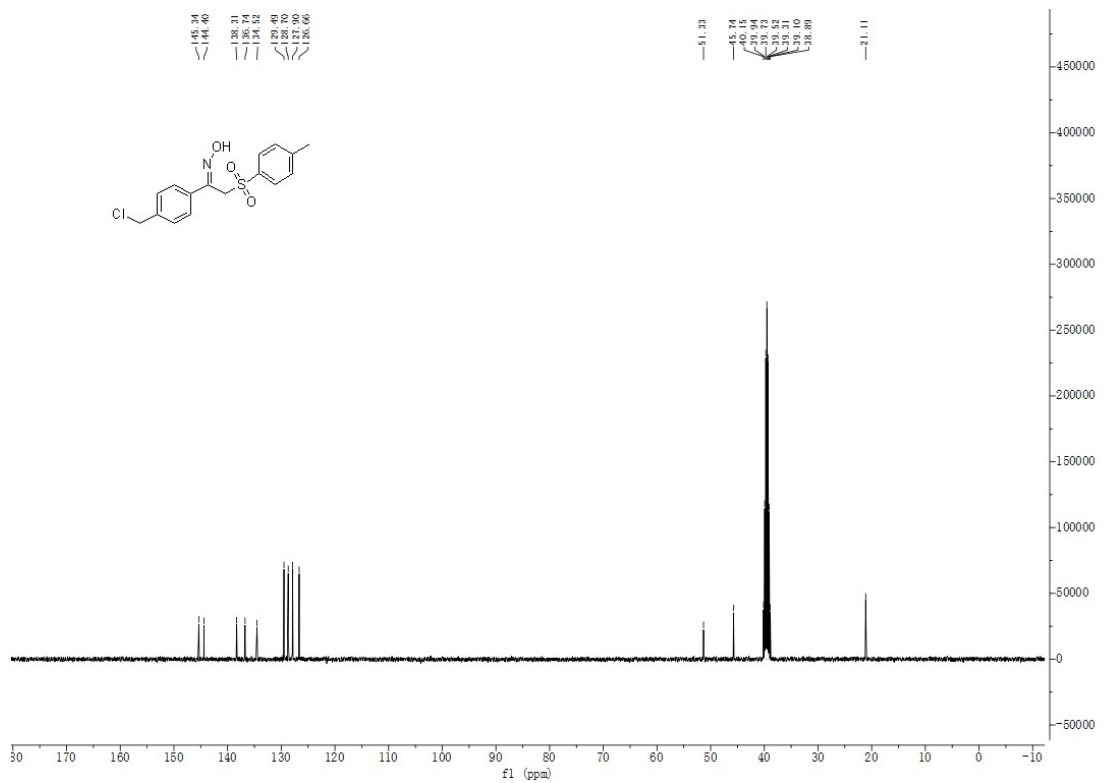
5c ^1H NMR (400 MHz, Chloroform-*d*), ^{13}C NMR (101 MHz, Chloroform-*d*)



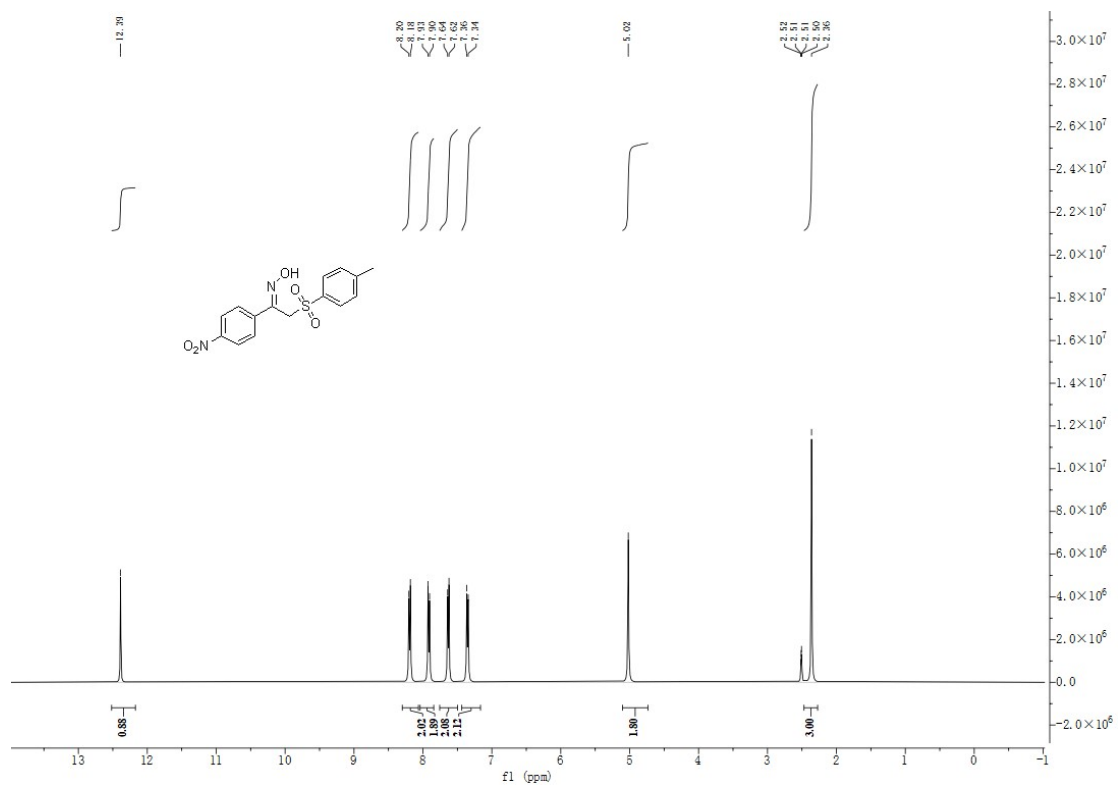
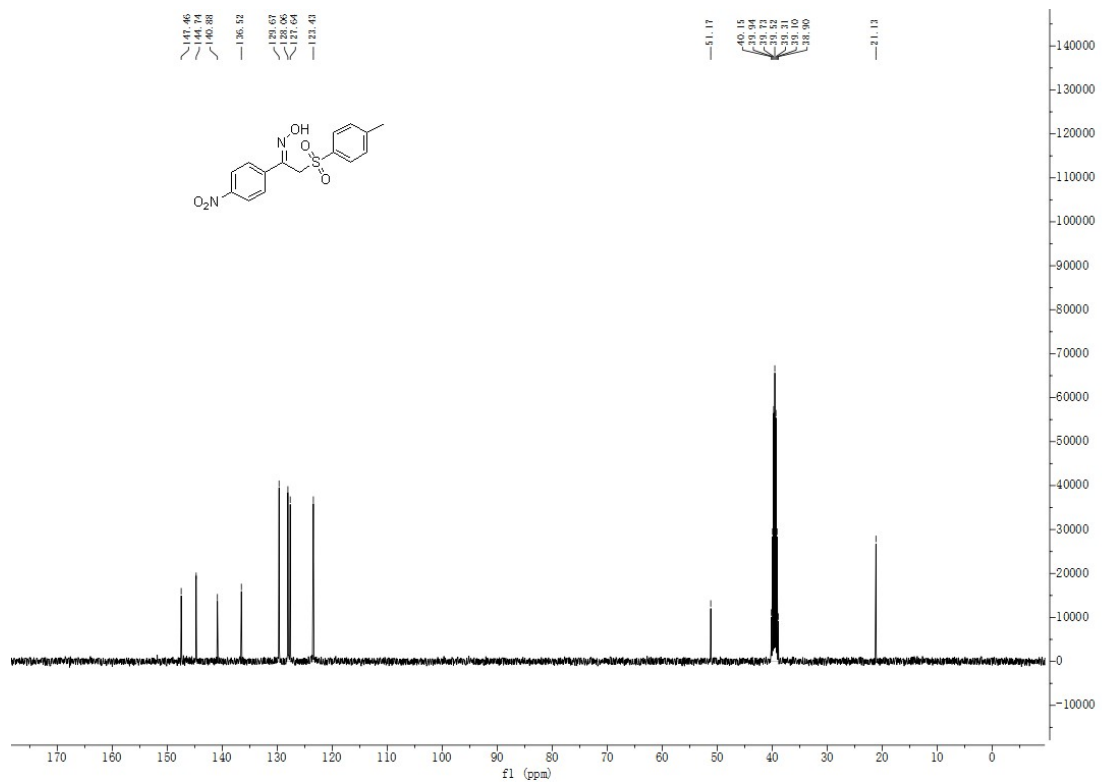
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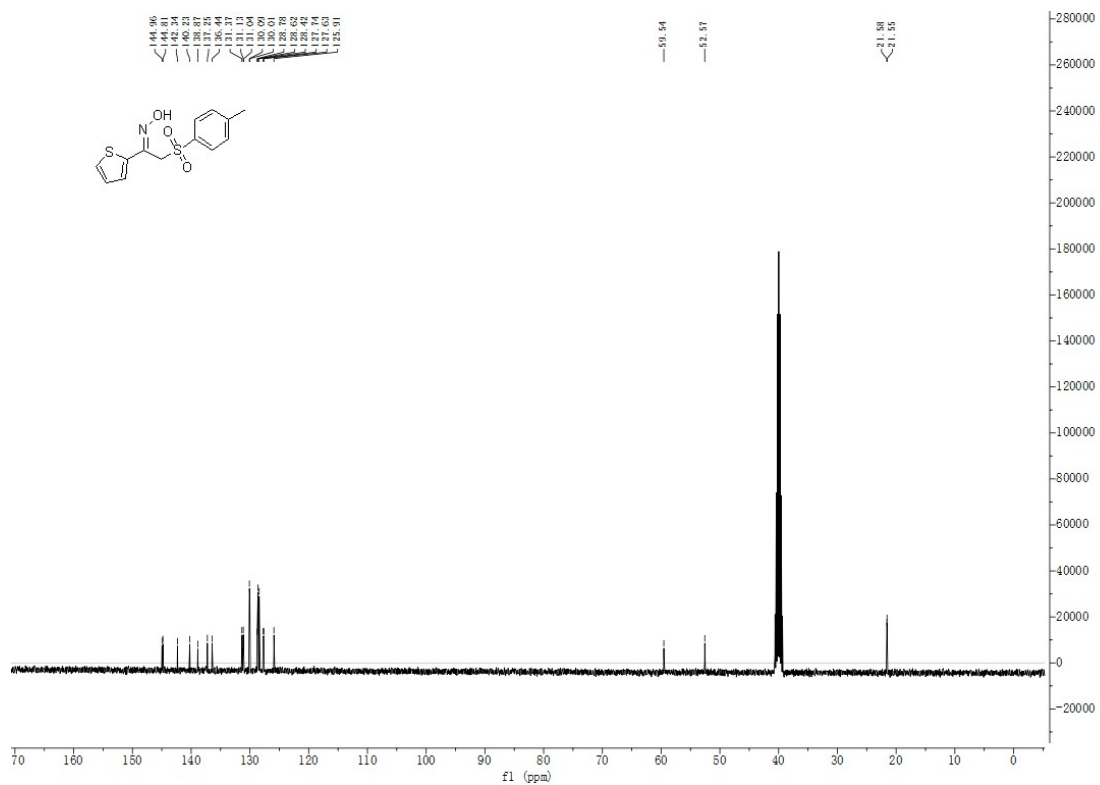
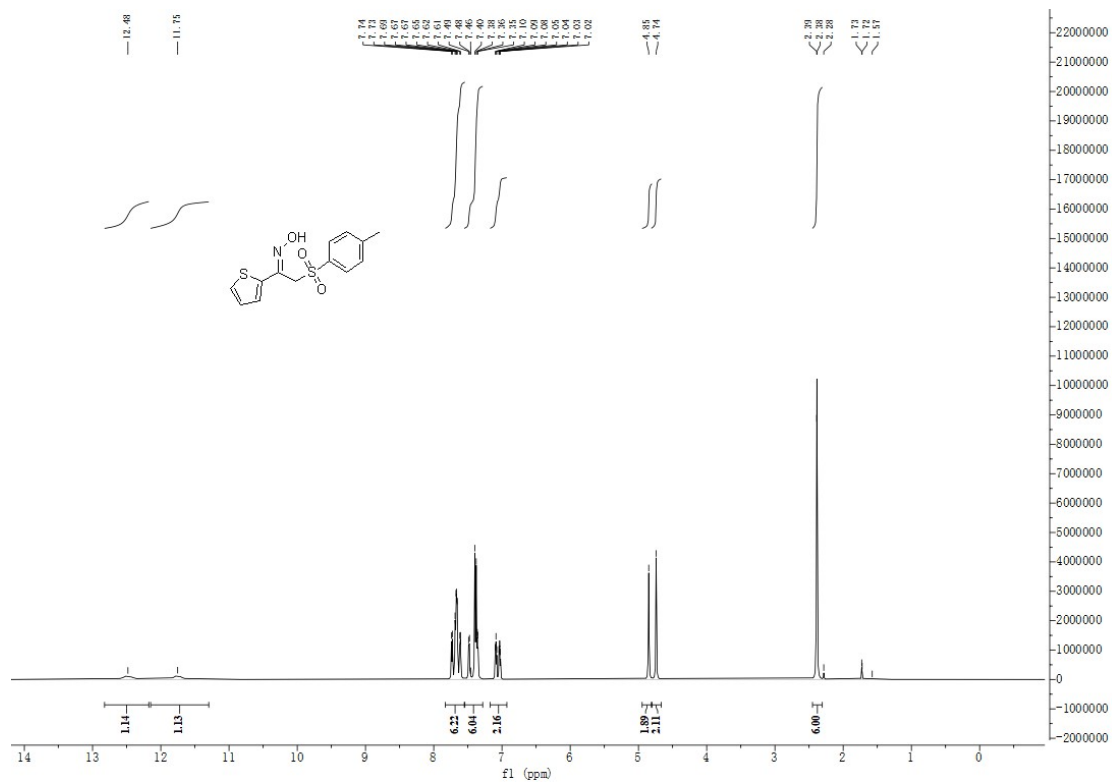
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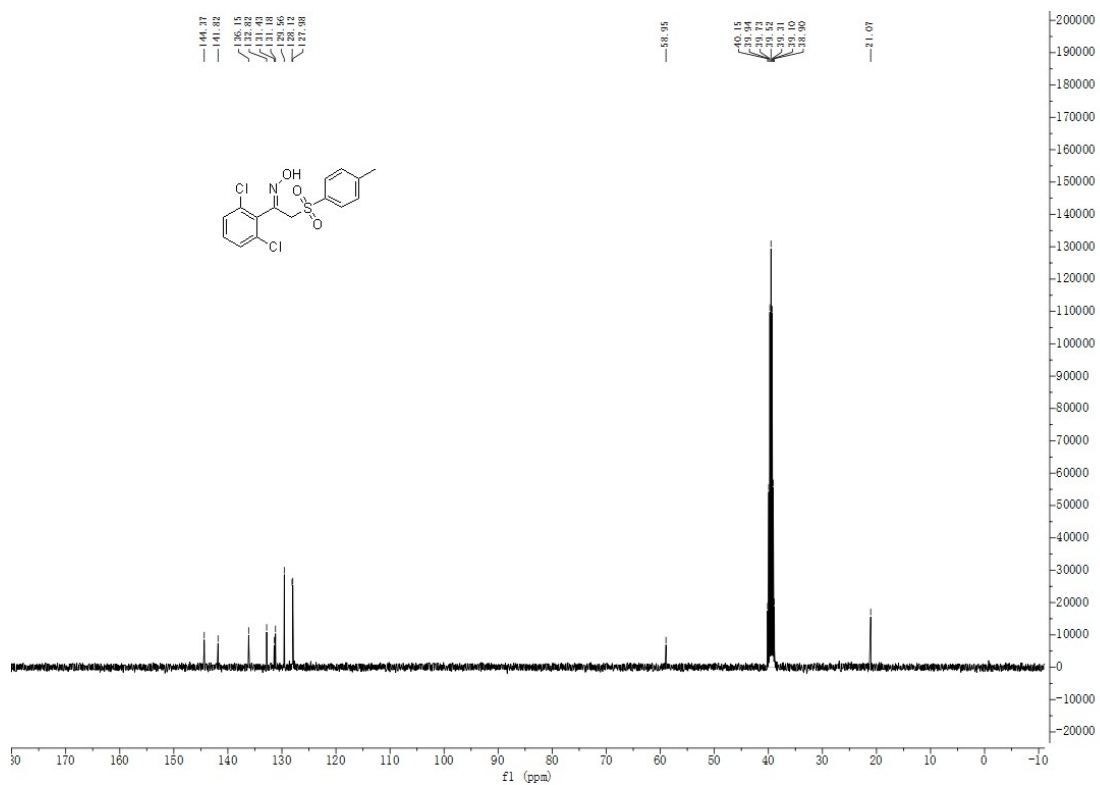
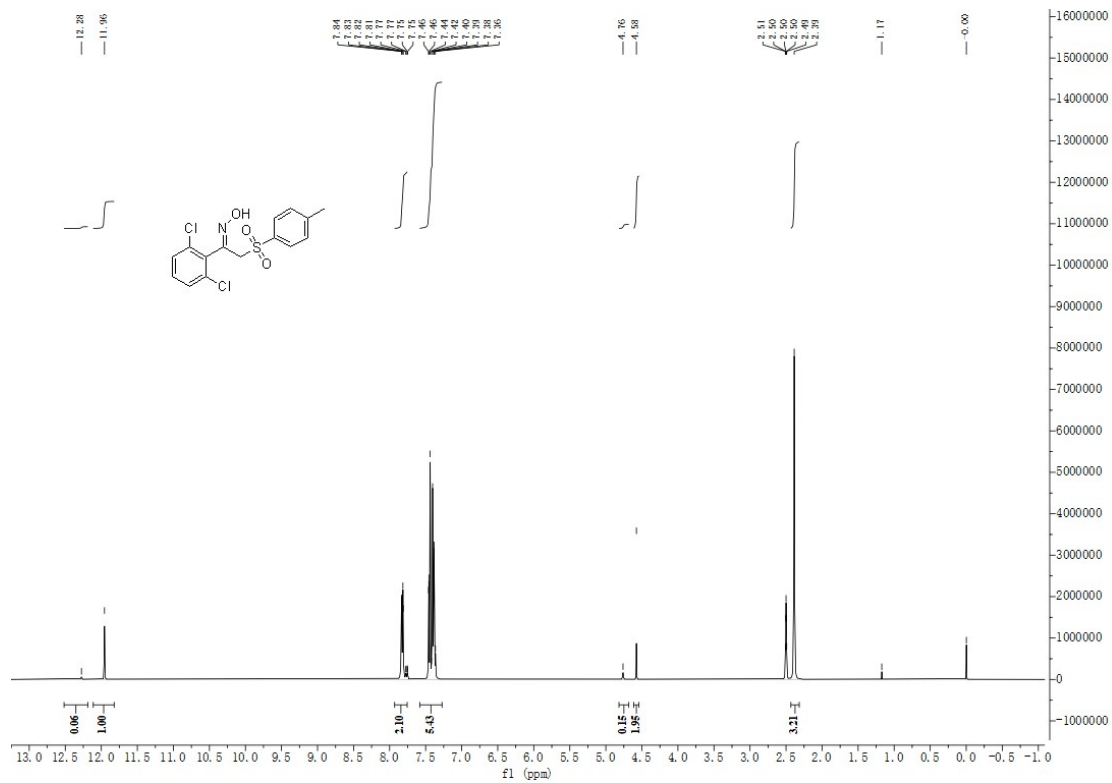
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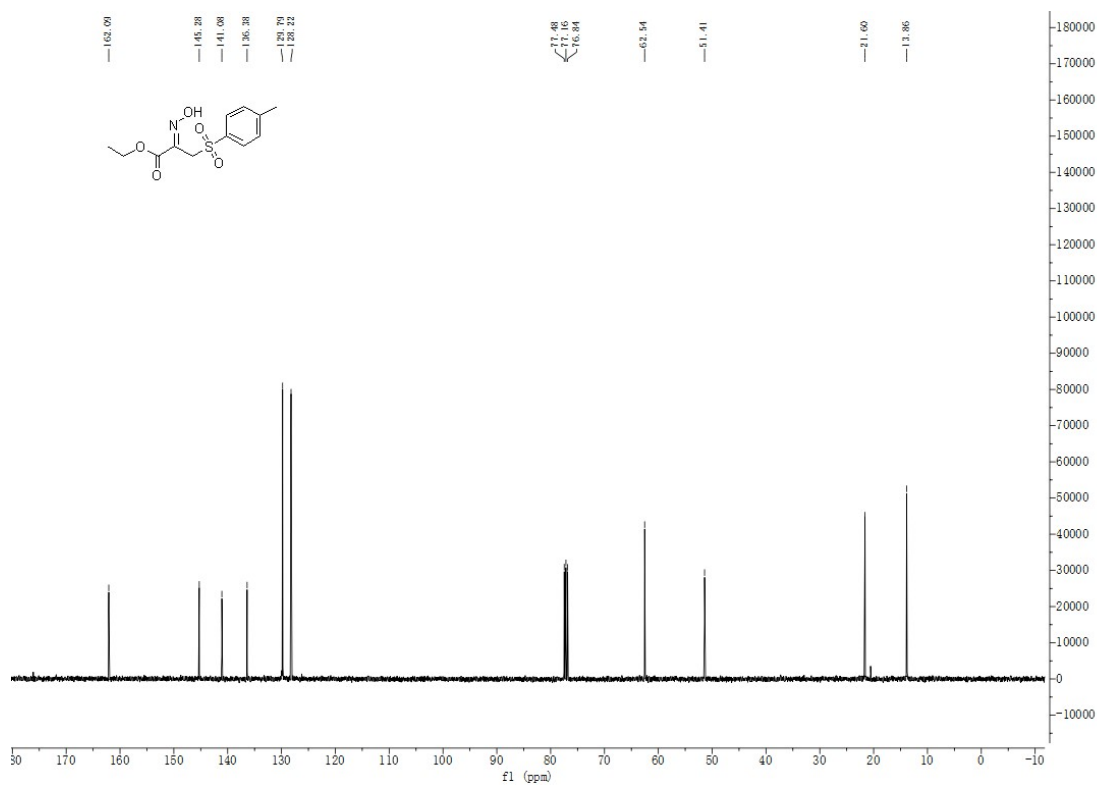
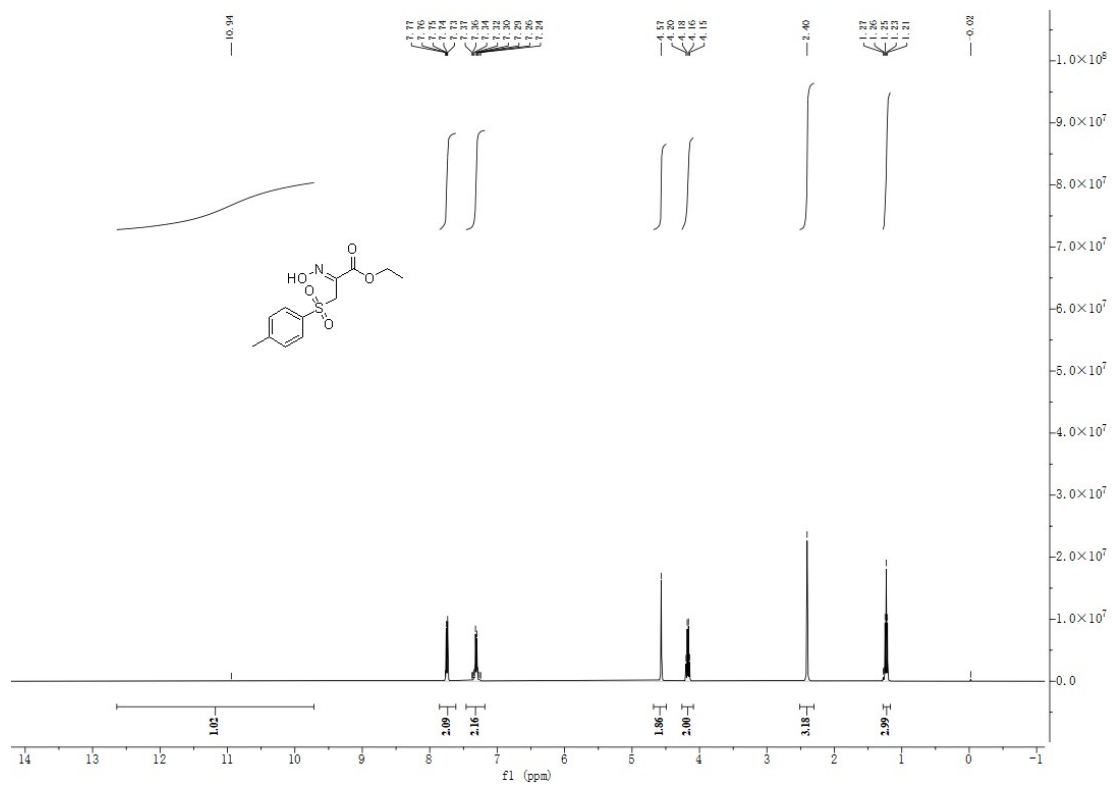
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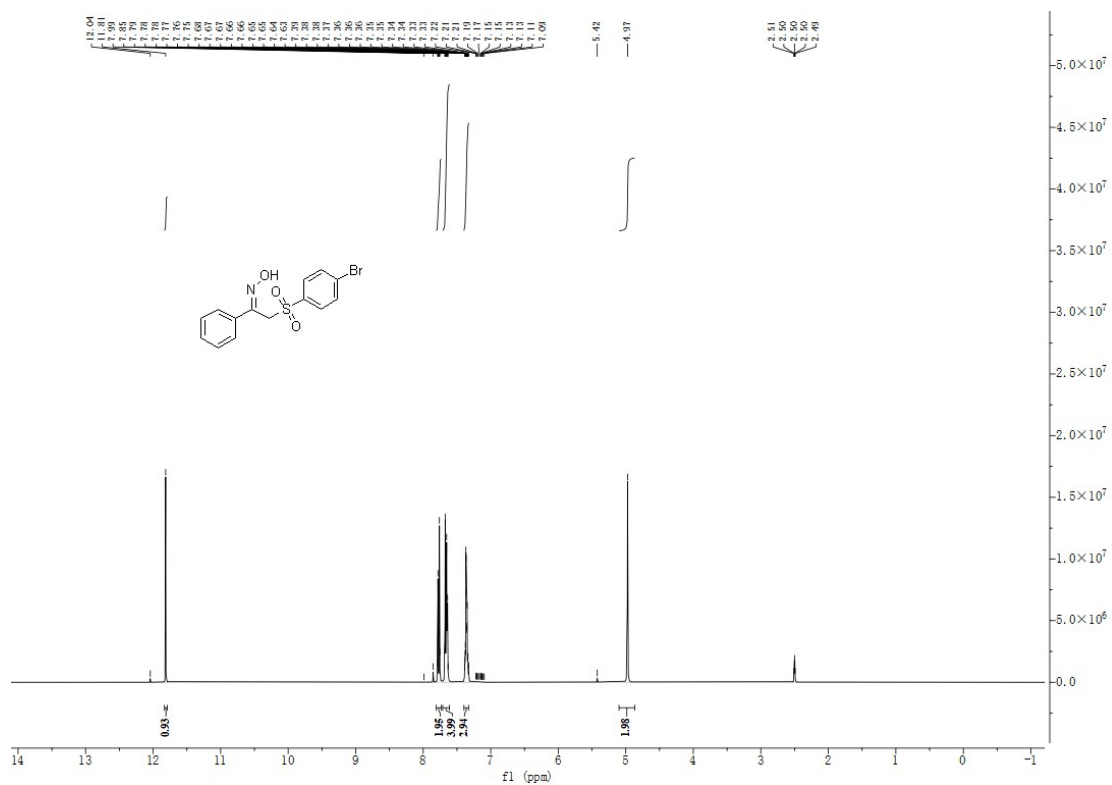
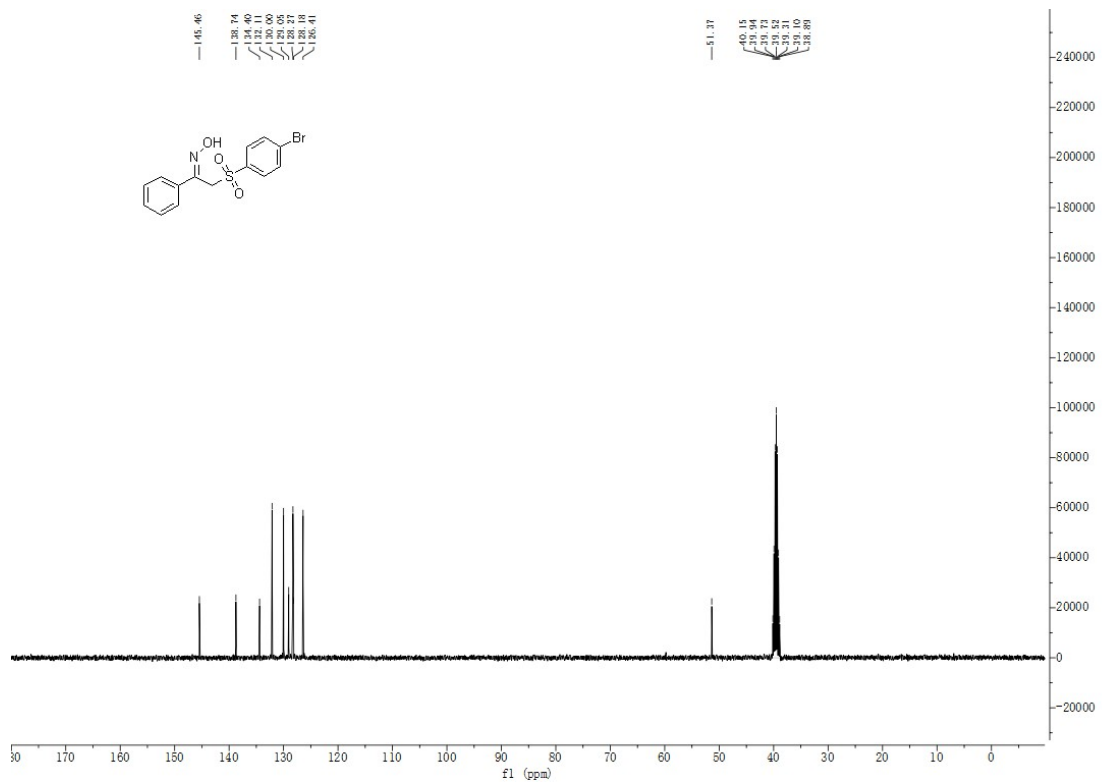
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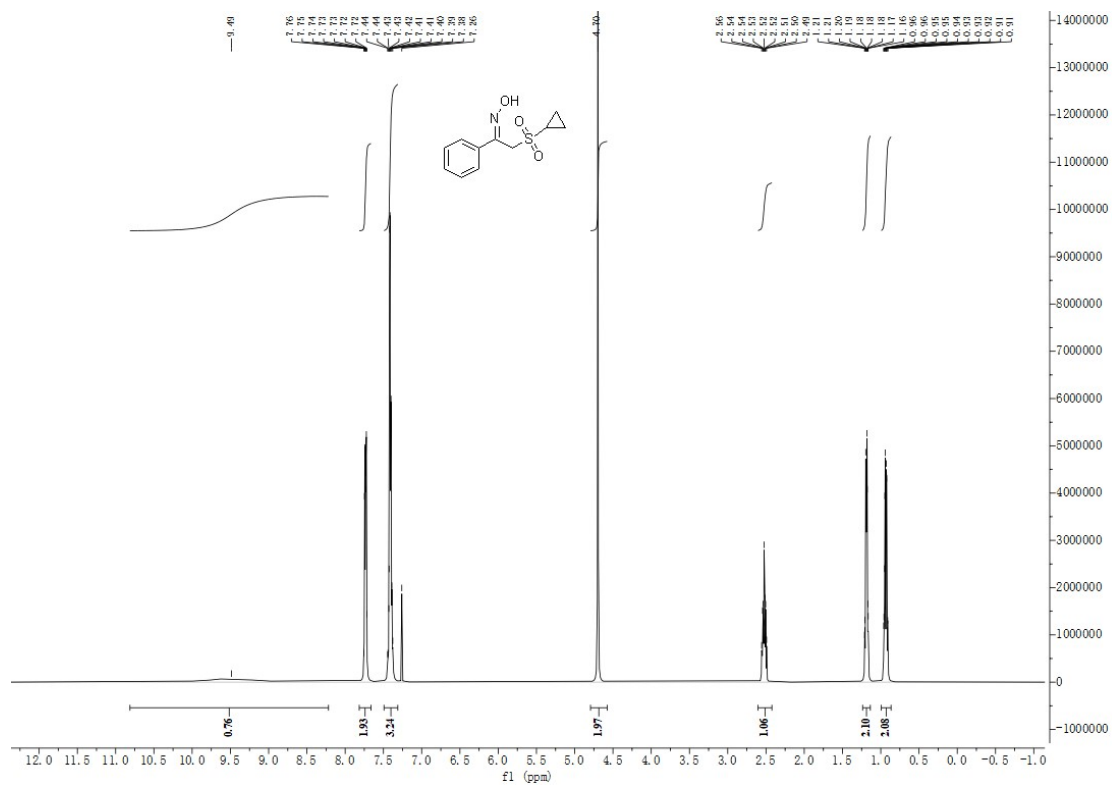
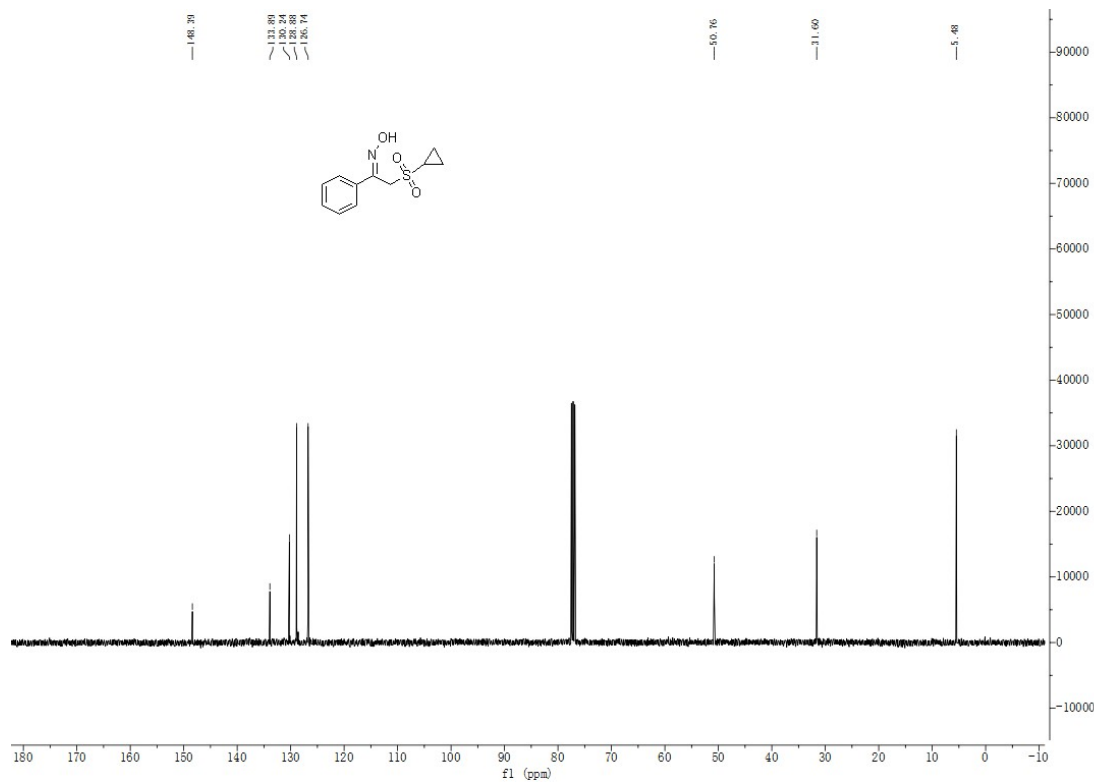
5i ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



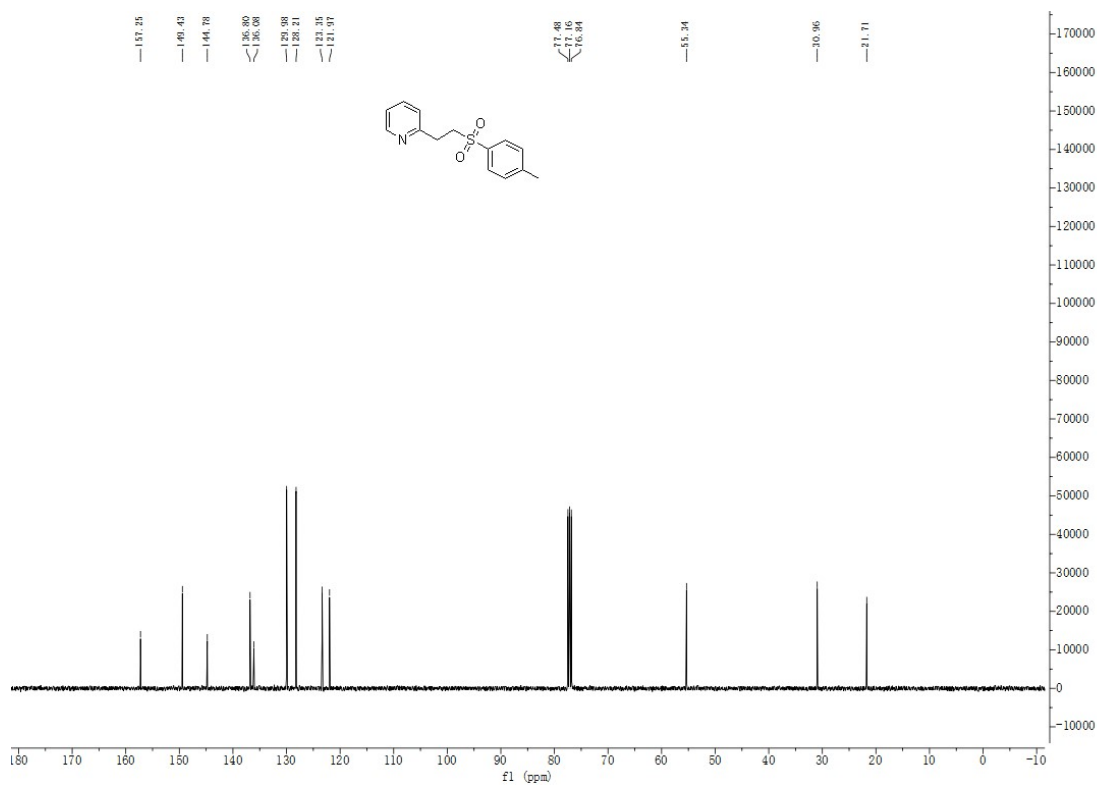
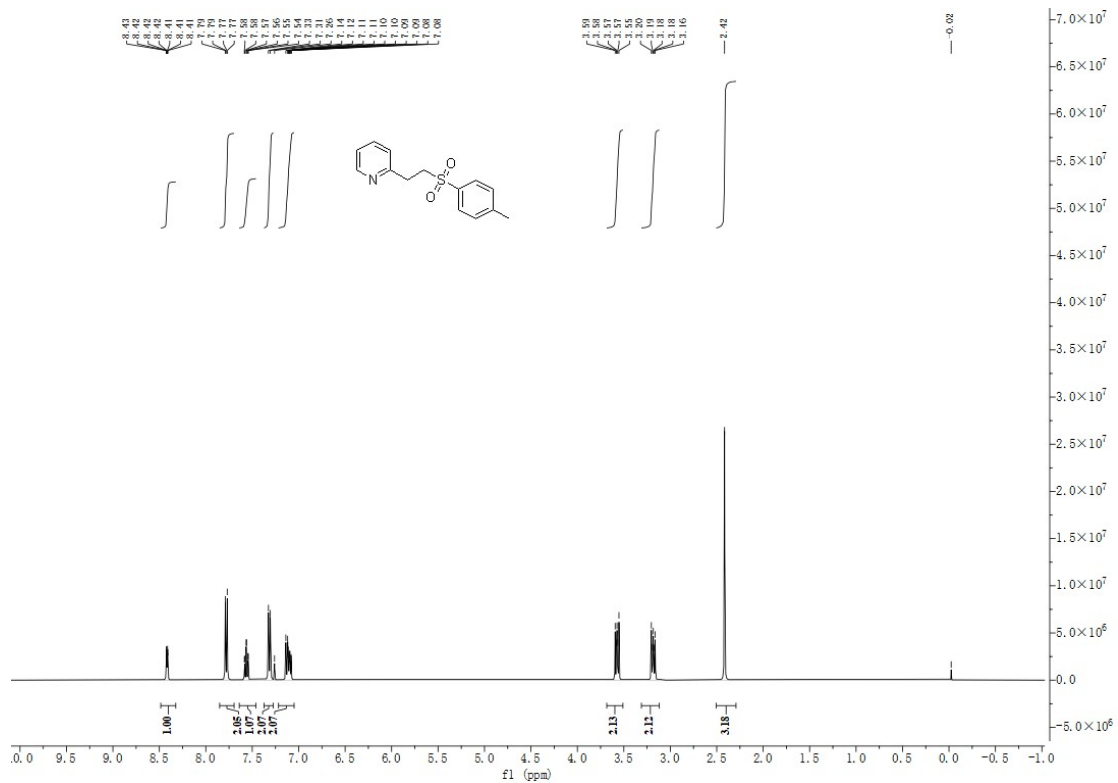
5j ^1H NMR (400 MHz, Chloroform-*d*), ^{13}C NMR (101 MHz, Chloroform-*d*)



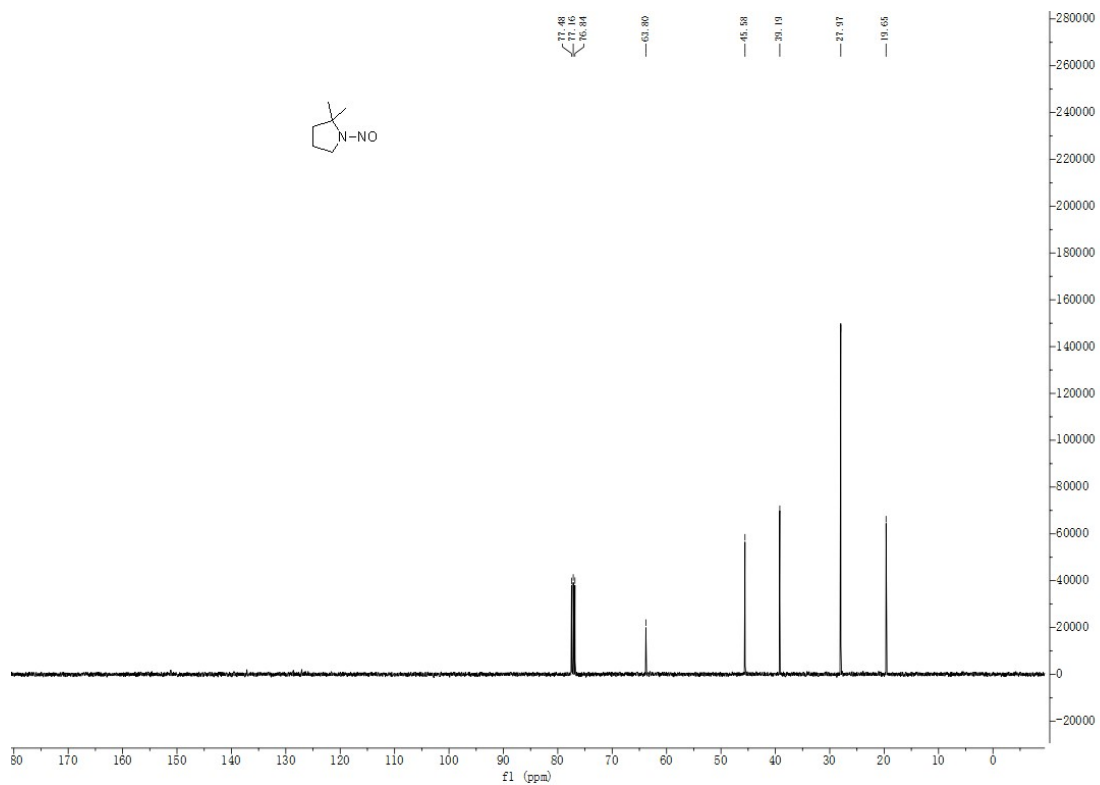
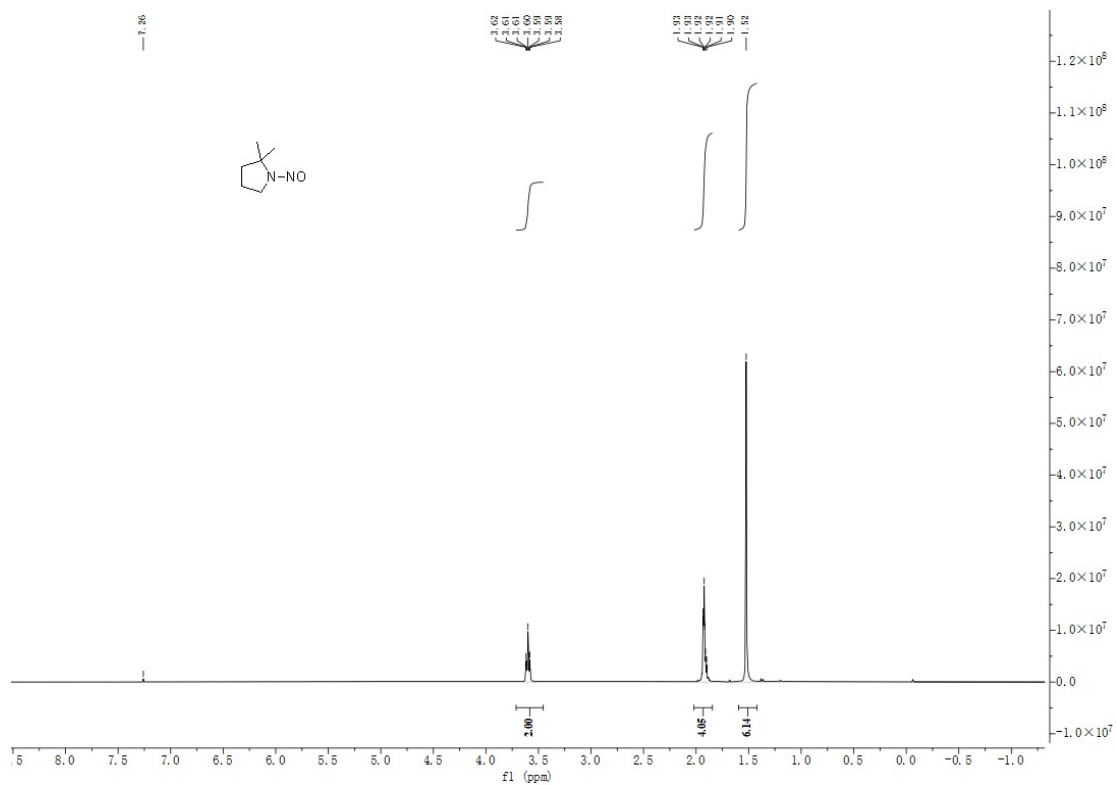
5k ¹H NMR (400 MHz, DMSO-*d*₆), ¹³C NMR (101 MHz, DMSO-*d*₆)



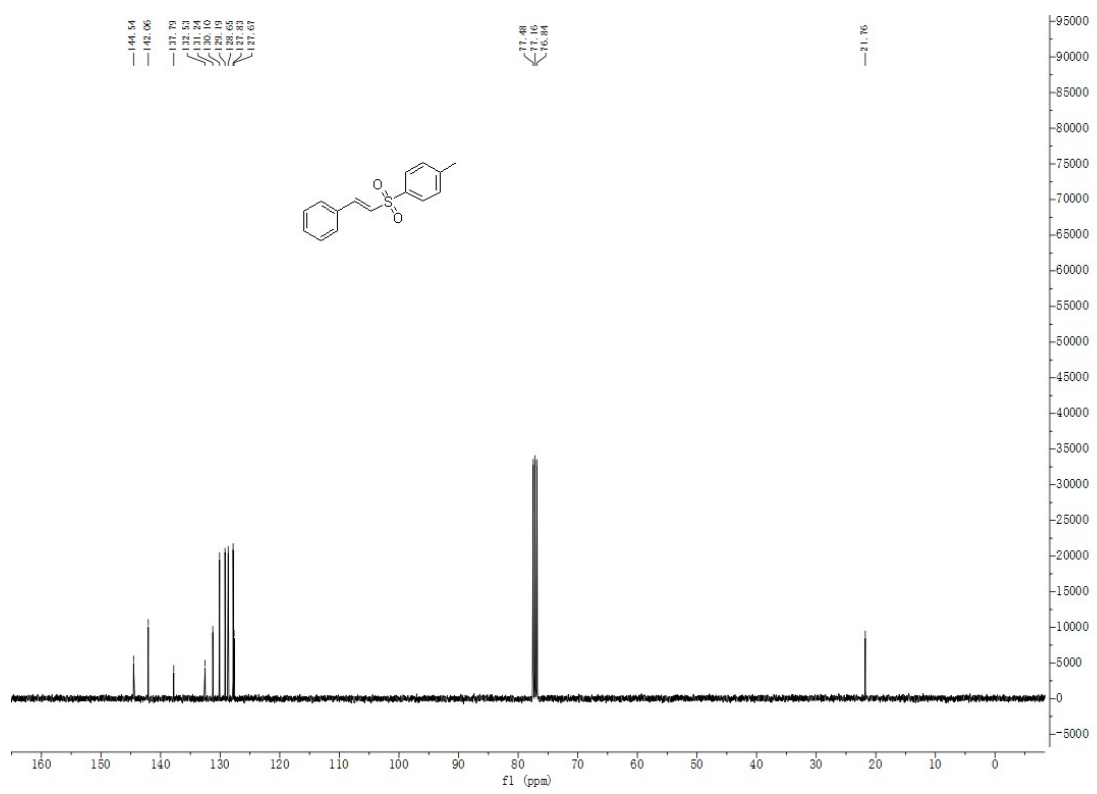
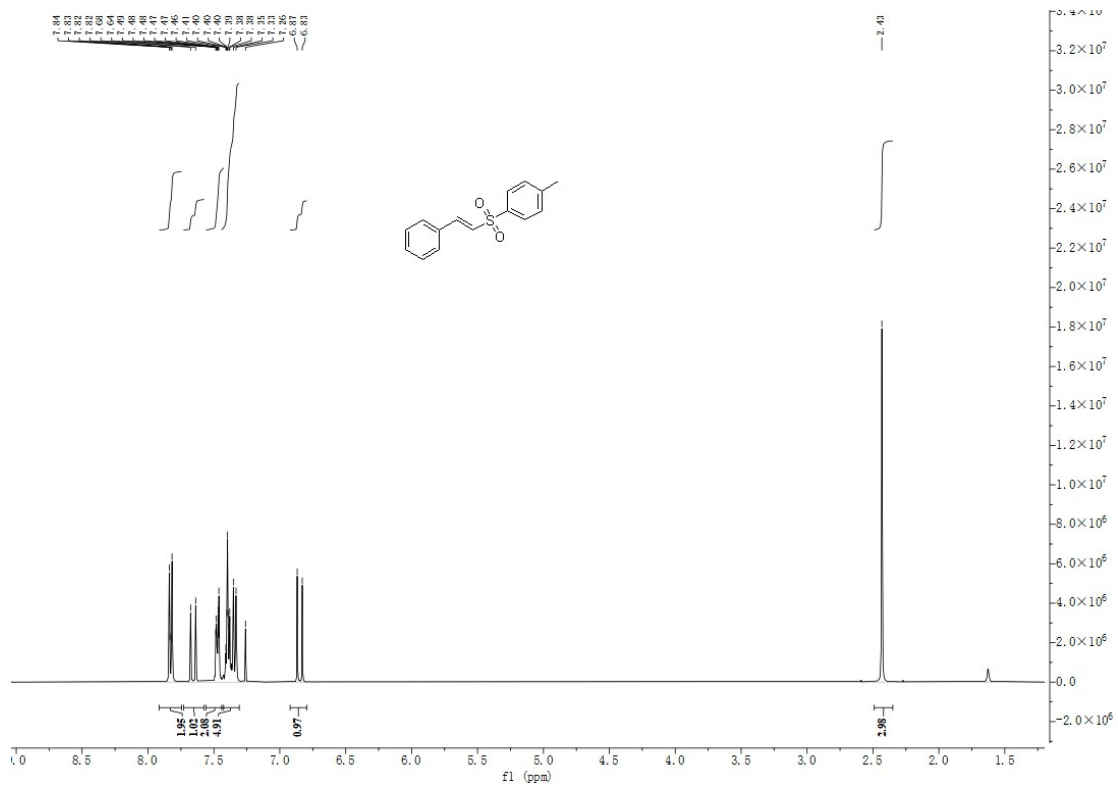
51 ^1H NMR (400 MHz, Chloroform-*d*), ^{13}C NMR (101 MHz, Chloroform-*d*)



3aa ¹H NMR (400 MHz, Chloroform-*d*), ¹³C NMR (101 MHz, Chloroform-*d*)



6 ^1H NMR (400 MHz, Chloroform-*d*), ^{13}C NMR (101 MHz, Chloroform-*d*)



5p' ¹H NMR (400 MHz, Chloroform-*d*), ¹³C NMR (101 MHz, Chloroform-*d*)