Supporting Information

Organocatalytic Enantioselective Diels-Alder Reaction between Hydroxymaleimides and in situ Generated Nitrosoalkenes for Direct Preparation of Chiral Hemiketals with 1,2-Oxazine Skeleton

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1 General remarks

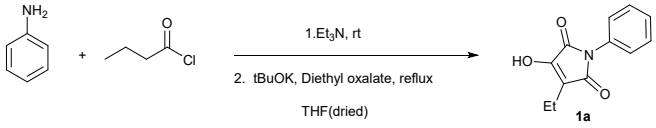
Chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Reactions were monitored by TLC and visualized with ultraviolet light. Flash column chromatography was performed on silica gels (300-400 mesh) eluting with ethyl acetate, dichloromethane and petroleum ether. ^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO-d_6 on a Bruker Avance instrument (600 MHz for ^{1}H NMR,150 MHz for ^{13}C NMR; 300 MHz for ^{1}H NMR, 75 MHz for ^{13}C NMR). ^{1}H NMR chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard (DMSO-d_6 at 2.50 ppm), chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard (DMSO-d_6 at 39.52 ppm). High-resolution mass spectra (HRMS) analyses were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer and 1290 Infinity LC/6460 QQQMS. Enantiomeric excess was determined by HPLC analysis on chiralpak AD-H, IC, AY-H. Optical rotations were measured on a Perkin-Elmer 241 Polarimeter. Melting points were recorded on a Buchi Melting Point B-545.

2 General procedures for the syntheses of compounds 1a-1s

2.1 Hydroxymaleimides:1a-1u and characterization data

1a-1r were prepared according to the literature. [1]

Typical preparation for 3-ethyl-4-hydroxy-1-phenyl-1H-pyrrole-2,5-dione **1a.**



Step-1: A solution of aniline (1.06 g, 10 mmol, 1 equiv.) and triethylamine (1.21 g, 12 mmol, 1.2 equiv.) in 30 mL THF was placed in a round-bottomed flask which was stirred at 25 °C for 10 min, N-butyryl chloride (1.16 g, 11 mmol, 1.1 equiv.) is injected into the sealed bottle by a syringe and stirred at 25 °C for 3 h.

Step-2: Potassium tert-butoxide (2.80 g, 25 mmol, 2.5 equiv.) was slowly added to the reaction flask and stirred at 25 °C for 10 minutes, diethyl oxalate was added (3.65 g, 25 mmol, 2.5 equiv.) and stirred for 10 minutes. The reaction temperature was raised to 70 °C and stirred for another 12 h. After full consumption of the intermediate monitored by TLC. 2 M HCl (25 mL) was added to neutralize the residue to pH = 1. The aqueous solution was extracted with CH_2Cl_2 (3×50 mL). The organic phases were combined, washed with brine and dried over anhydrous Na_2SO_4. After evaporation of solvent under reduced pressure, the crude product was purified by flash column chromatography petroleum ether/ethyl acetate (v(PE/EA) = 15/1 to 4/1) to afford product **1a** (Yellow solid, 1.35g, 67% yield). **^{1}H NMR** (300 MHz, DMSO-d6) δ 13.63 (s, 1H), 7.49-7.40 (m, 2H), 7.37-7.28 (m, 3H), 2.30 (q, J = 7.5 Hz, 2H), 1.08 (t, J = 7.5 Hz, 3H); **^{13}C NMR** (75 MHz, DMSO-d6) δ 171.0, 166.1, 152.5, 131.8, 128.8, 127.2, 126.5, 111.2, 14.3, 12.6. Consistent with previous reports in the literature.[1] Substrates **1b-1p** were prepared by the same procedures. 2-Chloroacetyl chloride is substituted for the preparation of **1s** with similar procedures as for **1a**.

2.1 α-bromoketoxime 2a-2o

α-bromoketoxime were prepared according to the reported methods described in the literatures.[2]

2.3 Catalysts 4a-4d

Catalysts **4** were prepared according to the reported methods described in the literatures.[3]

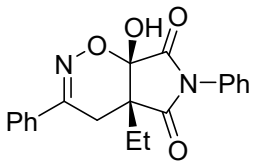
References:

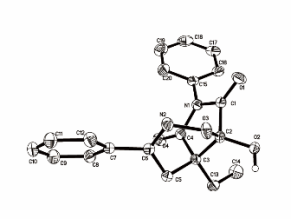
[1] a) Yang, Y.; Ren, H. X.; Chen, F.; Zhang, Z. B.; Zou, Y.; Chen, C.; Song, X. J.; Tian, F.; Peng, L.; Wang, L. X. Organocatalytic Asymmetric Annulation between Hydroxymaleimides and Nitrosoarenes: Stereoselective Preparation of Chiral Quaternary N-Hydroxyindolines. *Org. Lett.* **2017**, *19*, 2805-2808.; (b) Tantray, M. A.; Khan, I.; Hamid, H.; Alam, M. S. Synthesis of aryl anilinomaleimide based derivatives as glycogen synthase kinase-3β inhibitors with potential role as antidepressant agents. *New J. Chem.* **2016**, *40*, 6109-6119.

[2] Wabnitz, T. C.; Saaby, S. Jøgensen, K. A. The first catalytic inverse-electron demand hetero-Diels-Alder reaction of nitrosoalkenes using pyrrolidine as an organocatalyst. *Org. Biomol. Chem*. **2004**, *2*, 828-834.

[3] a) Vakulya, B.; Varga, S.; Csámpai, A. Soós, T. Highly Enantioselective Conjugate Addition of Nitromethane to Chalcones Using Bifunctional Cinchona Organocatalysts. *Org. Lett.* **2005**, *7*, 1967; (b) Zhu, Q.; Lu, Y. Stereocontrolled Creation of All-Carbon Quaternary Stereocenters by Organocatalytic Conjugate Addition of Oxindoles to Vinyl Sulfone. *Angew. Chem. Int. Ed*. **2010**, *49*, 7753-7756; *Angew. Chem*. **2010**, *122*, 7919-7922; (c) Badiola, E.; Fiser, B.; Gomez-Bengoa, E. Enantioselective Construction of Tetrasubstituted Stereogenic Carbons through Brønsted Base Catalyzed Michael Reactions: α′-Hydroxy Enones as Key Enoate Equivalent. *J. Am. Chem. Soc.* **2014**, *136*, 17869-17881.

3 X-ray crystallographic data of compound 3aa, 3an’

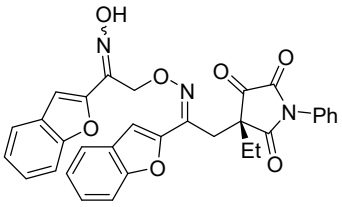


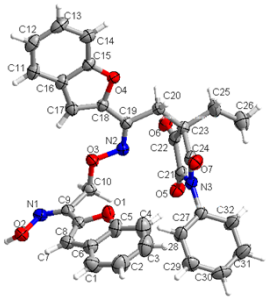


3aa	CCDC 2075420
Identification code	20200668
Empirical formula	C20H18N2O4
Formula weight	350.36
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	P212121
a/Å	7.69091(15)
b/Å	11.2958(2)
c/Å	20.0933(5)
α/°	90
β/°	90
γ/°	90
Volume/Å3	1745.61(6)
Z	4
ρcalcg/cm3	1.333
μ/mm-1	0.773
F(000)	736.0
Crystal size/mm3	0.14 × 0.12 × 0.09
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.802 to 134.09
Index ranges	-9 ≤ h ≤ 6, -13 ≤ k ≤ 13, -24 ≤ l ≤ 22
Reflections collected	12539
Independent reflections	3119 [Rint = 0.0347, Rsigma = 0.0271]
Data/restraints/parameters	3119/0/237
Goodness-of-fit on F2	1.068
Final R indexes [I>=2σ (I)]	R1 = 0.0355, wR2 = 0.0882

Final R indexes [all data]	R1 = 0.0383, wR2 = 0.0913
Largest diff. peak/hole / e Å-3	0.16/-0.25
Flack parameter	-0.04(11)

Single crystal of **3aa** was obtained by slow evaporation from CH_3OH at 25 °C. ORTEP diagram of compound **3aa**, the ellipsoid contour probability levels: 50%.





3an'	CCDC 2075423
Identification code	20210342
Empirical formula	C32H25N3O7
Formula weight	563.55
Temperature/K	293(2)
Crystal system	trigonal
Space group	P31
a/Å	14.1958(4)
b/Å	14.1958(4)
c/Å	29.4228(9)
α/°	90
β/°	90
γ/°	120
Volume/Å3	5135.0(3)
Z	6
ρcalcg/cm3	1.093
μ/mm-1	0.647
F(000)	1764.0
Crystal size/mm3	0.27 × 0.15 × 0.12
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.19 to 134.132

Index ranges	-16 ≤ h ≤ 16, -16 ≤ k ≤ 15, -31 ≤ l ≤ 35
Reflections collected	56519
Independent reflections	11190 [Rint = 0.0423, Rsigma = 0.0281]
Data/restraints/parameters	11190/21/689
Goodness-of-fit on F2	1.073
Final R indexes [I>=2σ (I)]	R1 = 0.0670, wR2 = 0.1940
Final R indexes [all data]	R1 = 0.0745, wR2 = 0.2057
Largest diff. peak/hole / e Å-3	0.46/-0.26
Flack parameter	0.22(11)

Single crystal of **3an'** was obtained by slow evaporation from CH_3CH_2OH at 25 ºC. ORTEP diagram of compound **3an'**, the ellipsoid contour probability levels: 50%.

4 Procedures and characterization data

4.1 Typical procedures for the organocatalytic and enantioselective Diels–Alder reaction between hydroxymaleimides 1a and in situ generated nitrosoalkenes from α-bromoketoxime 2a

A solution of hydroxymaleimide **1a** (21.7 mg, 0.1 mmol, 1.0 equiv.), α-bromoketoxime **2a** (26.0 mg, 0.12 mmol, 1.2 equiv.), and Cat **4b** (3 mg, 5 mol%), Na_2CO_3 (6.4 mg, 0.6 eq) in DCM (2mL, 0.1 M) was stirred at 25 °C. After **1a** was consumed by TLC (24 h, R_f = 0.3, V(PE: EA) = 5:1), the solvent was evaporated and the mixture was directly purified by flash column chromatography petroleum ether/ethyl acetate(V(PE/EA = 10/1 to 5/1) to afford products **3aa** (33.0 mg). Other reactants were operated by the same procedures.

4.2 Scale-up preparation and representative transformation of product 3aa and control experiments.

4.2.1 Scale-up (Scheme S1 a).

3aa: A solution of hydroxymaleimide **1a** (1.1 g, 5.0 mmol, 1.0 equiv.), α-bromoketoxime **2a** (1.28 g, 6 mmol, 1.2 equiv.), and Cat **4b** (0.15 g, 5 mol%), Na_2CO_3 (0.32 g, 0.6 eq) in DCM (50 mL, 0.1 M) was stirred at 25 °C. After **1a** was consumed by TLC (24 h, R_f = 0.3, V(PE: EA) = 5:1), the solvent was evaporated and the mixture was directly purified by flash column chromatography petroleum ether/ethyl acetate(V(PE/EA = 10/1 to 5/1) to afford products **3aa** (1.6 g, 90% yield, 97 % ee).

4.2.2 **control experiments** (Scheme S1 b-d)

Scheme S1 b:

A solution of product **3aa** (35 mg, 0.1 mmol, 1.0 equiv.), α-bromoketoxime **2a** (26.0 mg, 0.12 mmol, 1.2 equiv.), and Na_2CO_3 (6.4 mg, 0.6mmol) in DCM (1mL, 0.1 M) was stirred at 25 °C. The reaction was monitored by TLC, and no new product was detected

Scheme S1 c:

A solution of product **3aa** (35 mg, 0.1 mmol, 1 equiv.), α-bromoketoxime **2m** (24.0 mg, 0.12 mmol, 1.2 equiv.), and Na_2CO_3 (6.4 mg, 0.6mmol) in DCM (1mL, 0.1 M) was stirred at 25 °C. The reaction was monitored by TLC, and no new product was detected.

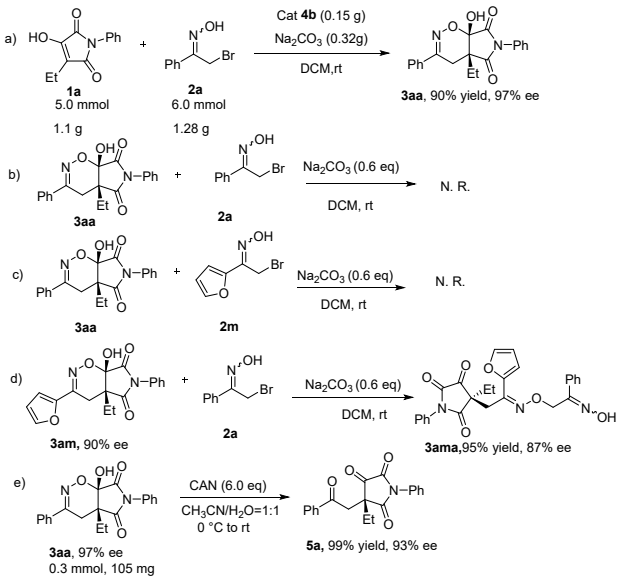
Scheme S1 d:

A solution of product **3am** (34 mg, 0.1 mmol, 1 equiv.), α-bromoketoxime **2a** (26.0 mg, 0.12 mmol, 1.2 equiv.), and Na_2CO_3 (6.4 mg, 0.6 mmol) in DCM (1mL, 0.1 M) was stirred at 25 °C. After **3am** was consumed by TLC (24 h, R_f = 0.2, V(PE: EA) = 5:1), the solvent was evaporated and the mixture was directly purified by column chromatography petroleum ether/ethyl acetate(V(PE/EA = 5/1) to afford products **3ama** (45mg, 95% yield, 87 % ee).

4.2.3 Representative transformations (Scheme S1 e)

A solution of product **3aa** (105 mg, 0.3 mmol, 1.0 equiv.), Cerium Ammonium Nitrate (986 mg, 1.8 mmol, 6.0 equiv.) in 8 mL solvent (V(CH_3CN:H_2O)= 1:1) was stirred at 0 °C. After **3aa** was consumed by TLC (10 min, R_f = 0.4, V(PE: EA) = 6:1). The reaction solution was extracted with DCM (8 mL×3), concentrated under reduced pressure, and column chromatography to obtain the target product **5a**. (99.5 mg,99% yield, 93% ee).

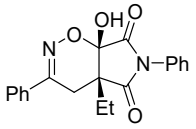
Scheme S 1 Scaled-up preparation, control experiments and representative transformation of the product 3aa.



4.3 Characterization data

(**3aa**):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3,6-diphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA)= 10/1 to 5/1] to afford white solid, 94% yield, 97% ee; mp = 176.1-176.9 °C; [α] D^{20} = +257.8 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 17 min);

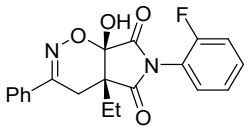
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.85 (s, 1H), 7.86-7.70 (m, 2H), 7.57-7.43 (m, 6H), 7.30-7.18 (m, 2H), 3.17 (d, *J* = 16.5 Hz, 1H), 2.83 (d, *J* = 16.5 Hz, 1H), 2.07-1.78 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.8, 164.2, 133.2, 131.0, 130.9, 129.4, 129.1, 128.9, 126.6, 126.1, 96.7, 50.2, 27.4, 26.6, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{18}N_2O_4H^+$ 351.1339, found 351.1346.

(**3ba**):

(4a*R*,7a*S*)-4a-ethyl-6-(2-fluorophenyl)-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA)= 10/1 to 5/1] to afford white solid, 92% yield, 96% ee; mp = 182.7-183.5 °C; [α] D^{20} = +201.9 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 17 min, t_{major} = 20 min);

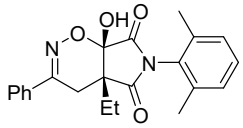
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.96 (s, 1H), 7.80 (d, *J* = 7.1 Hz, 2H), 7.61-7.28 (m, 7H), 3.18 (d, *J* = 16.4 Hz, 1H), 2.85 (d, *J* = 16.4 Hz, 1H), 2.07-1.75 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 175.6, 171.3, 164.6, 156.7 (d, *J* = 251.0 Hz), 133.2, 131.9 (d, *J* = 8.0 Hz), 130.9, 129.6, 128.9, 126.1, 125.4, 118.6 (d, *J* = 13.6 Hz), 116.7 (d, *J* = 19.1 Hz), 97.0, 50.9, 27.3, 26.8, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}FN_2O_4H^+$ 369.1245, found 369.1250.

(**3ca**):

(4a*R*,7a*S*)-6-(2,6-dimethylphenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 96% ee; mp = 198.8-200.1 °C; [α] D^{20} = +166.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24 min, t_{minor} = 28 min);

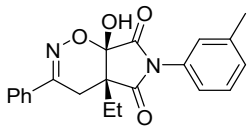
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.94 (s, 1H), 7.91-7.73 (m, 2H), 7.60-7.43 (m, 3H), 7.33-7.08 (m, 3H), 3.22 (d, *J* = 16.2 Hz, 1H), 2.82 (d, *J* = 16.3 Hz, 1H), 2.05 (s, 3H), 2.02-1.85 (m, 2H), 1.81 (s, 3H), 1.06 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.0, 171.8, 165.1, 135.7, 135.4, 132.9, 131.0, 129.5, 128.8, 128.5, 128.5, 126.1, 97.7, 50.9, 27.0, 26.5, 17.4, 17.1, 9.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{22}N_2O_4H^+$ 379.1652, found 379.1660

(**3da**):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-phenyl-6-(o-tolyl)-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 96% yield, 99% ee; mp = 154.6-155.2 °C; [α] D^{20} = +186.7 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 25 min, t_{major} = 27 min);

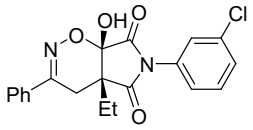
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.84 (s, 1H), 7.89-7.67 (m, 2H), 7.57-7.34 (m, 4H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.02 (d, *J* = 9.9 Hz, 1H), 3.16 (d, *J* = 16.5 Hz, 1H), 2.82 (d, *J* = 16.5 Hz, 1H), 2.33 (s, 3H), 2.03-1.79 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.1, 171.6, 163.8, 138.8, 133.0, 130.7, 130.6, 129.5, 128.9, 128.6, 126.7, 125.8, 123.4, 96.4, 49.9, 27.2, 26.3, 20.4, 9.0.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_4H^+$ 365.1496, found 365.1531

(**3ea**):

(4a*R*,7a*S*)-6-(3-chlorophenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 97% yield, 96% ee; mp = 165.8-166.7 °C; [α] D^{20} = +221.6 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5 min, t_{major} = 6 min);

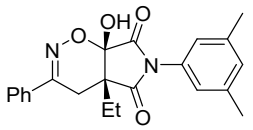
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.87 (s, 1H), 7.93-7.70 (m, 2H), 7.63-7.39 (m, 6H), 7.33-7.22 (m, 1H), 3.16 (d, *J* = 16.6 Hz, 1H), 2.86 (d, *J* = 16.6 Hz, 1H), 2.03-1.75 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.7, 164.4, 133.7, 133.5, 132.5, 131.3, 131.2, 129.4, 129.2, 126.9, 126.3, 125.8, 96.9, 50.4, 27.6, 26.6, 9.5.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}ClN_2O_4H^+$ 385.0950, found 385.0951

(**3fa**):

(4a*R*,7a*S*)-6-(3,5-dimethylphenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione3,5



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 95% ee; mp =177.5-179.1 °C; [α] D^{20} = +201.6 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24 min, t_{minor} = 25 min);

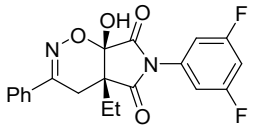
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.82 (s, 1H), 7.80 (d, *J* = 5.7 Hz, 2H), 7.59-7.43 (m, 3H), 7.09 (s, 1H), 6.82 (s, 2H), 3.14 (d, *J* = 16.6 Hz, 1H), 2.81 (d, *J* = 16.6 Hz, 1H), 2.28 (s, 6H), 2.00-1.81 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.9, 164.0, 138.8, 133.3, 131.0, 130.9, 130.5, 128.9, 126.1, 124.2, 96.7, 50.1, 27.5, 26.6, 20.6, 9.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{22}N_2O_4H^+$ 379.1652, found 379.1661

(**3ga**):

(4a*R*,7a*S*)-6-(3,5-difluorophenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 94% ee; mp = 150.7-151.9 °C; [α] D^{20}= +192.2 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 11 min, t_{major} = 24 min);

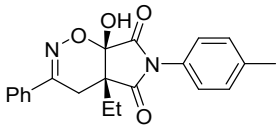
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.86 (s, 1H), 7.89-7.73 (m, 2H), 7.56-7.39 (m, 4H), 7.27-7.11 (m, 2H), 3.14 (d, *J* = 16.6 Hz, 1H), 2.87 (d, *J* = 16.7 Hz, 1H), 1.99-1.78 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 175.7, 171.0, 163.7, 162.2 (dd, *J* = 246.8, 14.5 Hz), 133.2, 133.1 (d, *J* = 12.6 Hz), 133.0 (d, *J* = 17.4 Hz), 130.9, 128.9, 126.1, 110.7 (d, *J* = 27.8 Hz), 110.7 (d, *J* = 9.3 Hz), 105.3, 105.0, 104.6, 96.4, 50.0, 27.4, 26.1, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{16}F_2N_2O_4H^+$ 387.1151, found 387.1160

(**3ha**):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-phenyl-6-(p-tolyl)-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 92% yield, 96% ee; mp = 163.7-165.6 °C; [α] D^{20} = +208.3 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14min, t_{minor} = 18 min);

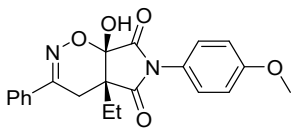
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.81 (s, 1H), 7.86-7.73 (m, 2H), 7.57-7.42 (m, 3H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 3.15 (d, *J* = 16.5 Hz, 1H), 2.82 (d, *J* = 16.5 Hz, 1H), 2.34 (s, 3H), 2.01-1.78 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.9, 164.2, 138.7, 133.2, 130.9, 129.8, 128.9, 128.4, 126.4, 126.0, 96.7, 50.1, 27.4, 26.6, 20.7, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_4H^+$ 365.1496, found 365.1505

(**3ia**):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-6-(4-methoxyphenyl)-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 95% yield, 96% ee; mp = 142.7-143.8 °C; [α] D^{20} = +180.8 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 19 min, t_{minor}= 26 min);

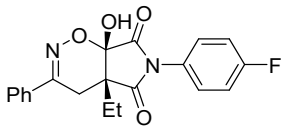
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.79 (s, 1H), 7.84-7.73 (m, 2H), 7.53-7.44 (m, 3H), 7.17-7.10 (m, 2H), 7.08-7.02 (m, 2H), 3.78 (s, 3H), 3.14 (d, *J* = 16.4 Hz, 1H), 2.81 (d, *J* = 16.5 Hz, 1H), 1.99-1.82 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.8, 172.3, 164.7, 159.7, 133.5, 131.1, 129.1, 128.2, 126.3, 123.8, 114.8, 97.0, 55.7, 50.4, 27.7, 27.0, 9.5.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_5H^+$ 381.1445, found 381.1451

(**3ja**):

(4a*R*,7a*S*)-4a-ethyl-6-(4-fluorophenyl)-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 96% ee; mp = 169.8-170.1 °C; [α] D^{20} = +240.3 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14 min, t_{minor} = 17 min);

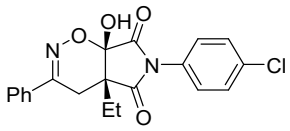
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.85 (s, 1H), 7.88-7.71 (m, 2H), 7.55-7.45 (m, 3H), 7.41-7.28 (m, 4H), 3.16 (d, *J* = 16.5 Hz, 1H), 2.84 (d, *J* = 16.5 Hz, 1H), 2.02-1.76 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.7, 164.3, 161.8 (d, *J* = 246.5 Hz), 133.2, 130.9, 129.0, 128.9, 127.2 (d, *J* = 3.1 Hz), 126.1, 116.4 (d, *J* = 23.1 Hz), 96.7, 50.2, 27.4, 26.6, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}FN_2O_4H^+$ 369.1245, found 369.1250

(**3ka**):

(4a*R*,7aS)-6-(4-chlorophenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 96% ee; mp = 158.7-159.9 °C; [α] D^{20} = +220.4 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 19 min);

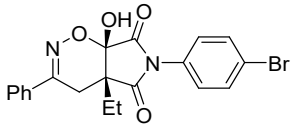
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.87 (s, 1H), 7.79 (dd, *J* = 7.8, 1.9 Hz, 2H), 7.63-7.56 (m, 2H), 7.53-7.42 (m, 3H), 7.35-7.26 (m, 2H), 3.16 (d, *J* = 16.6 Hz, 1H), 2.83 (d, *J* = 16.6 Hz, 1H), 2.00-1.75 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.1, 171.5, 164.1, 133.6, 133.2, 130.9, 129.8, 129.5, 128.9, 128.4, 126.1, 96.7, 50.1, 27.4, 26.5, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}ClN_2O_4H^+$ 385.0950, found 385.0961

(**3la**):

(4a*R*,7a*S*)-6-(4-bromophenyl)-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 94% ee; mp = 169.7-170.7 ºC; [α] D^{20} = +144.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} =16 min, t_{minor} = 21 min);

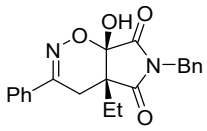
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.86 (s, 1H), 7.82-7.68 (m, 4H), 7.55-7.42 (m, 3H), 7.29-7.19 (m, 2H), 3.16 (d, *J* = 16.6 Hz, 1H), 2.83 (d, *J* = 16.6 Hz, 1H), 2.04-1.77 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.1, 171.5, 164.1, 133.2, 132.4, 130.9, 130.2, 128.9, 128.7, 126.0, 122.1, 96.6, 50.1, 27.4, 26.4, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}BrN_2O_4H^+$ 429.0444, found 429.0447

(**3ma**):

(4a*R*,7a*S*)-6-benzyl-4a-ethyl-7a-hydroxy-3-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 94% yield, 95% ee; mp = 164.7-166.5 ºC; [α] D^{20} = +128.4 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} =20 min, t_{major} = 25 min);

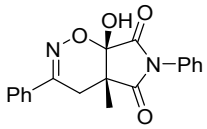
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.73 (s, 1H), 7.69 (d, *J* = 6.5 Hz, 2H), 7.59-7.36 (m, 3H), 7.31-7.09 (m, 5H), 4.63 (s, 2H), 3.05 (d, *J* = 15.9 Hz, 1H), 2.74 (d, *J* = 16.0 Hz, 1H), 1.92-1.65 (m, 2H), 0.80 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.9, 172.4, 165.9, 135.3, 133.0, 130.9, 128.8, 128.5, 127.6, 127.3, 126.1, 97.4, 50.7, 41.5, 27.4, 26.7, 9.2

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_4H^+$ 365.1496, found 365.1503

(3na):

(4a*R*,7a*S*)-7a-hydroxy-4a-methyl-3,6-diphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 99% yield, 98% ee; mp = 190.2-192.1 °C; [α] D^{20} = +150.3 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} =21 min, t_{major} = 24 min);

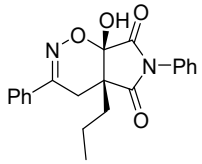
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.72 (s, 1H), 7.88-7.73 (m, 2H), 7.57-7.42 (m, 6H), 7.29-7.21 (m, 2H), 3.13 (d, *J* = 15.7 Hz, 1H), 2.91 (d, *J* = 15.7 Hz, 1H), 1.39 (s, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 177.1, 171.4, 166.6, 133.1, 131.1, 131.1, 129.2, 129.0, 128.9, 126.9, 126.2, 97.3, 47.2, 29.2, 18.4.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{19}H_{16}N_2O_4H^+$ 337.1183, found 337.1191

(3oa):

(4a*R*,7a*S*)-7a-hydroxy-3,6-diphenyl-4a-propyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 94% yield, 96% ee; mp = 183.3-184.8 °C; [α] D^{20} = +170.4 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14 min, t_{minor} = 16 min);

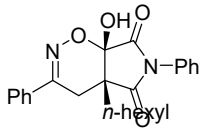
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.84 (s, 1H), 7.91-7.69 (m, 2H), 7.57-7.38 (m, 6H), 7.31-7.10 (m, 2H), 3.16 (d, *J* = 16.5 Hz, 1H), 2.84 (d, *J* = 16.4 Hz, 1H), 1.94-1.69 (m, 2H), 1.56-1.23 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.8, 164.4, 133.2, 131.0, 130.9, 129.4, 129.1, 128.9, 126.6, 126.0, 96.8, 50.0, 36.4, 26.9, 17.8, 14.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_4H^+$ 365.1496, found 365.1506.

(**3pa**):

(4a*R*,7a*S*)-4a-hexyl-7a-hydroxy-3,6-diphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 86% yield, 95% ee; mp = 132.7-133.7 °C; [α] D^{20} = +173.5 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 10 min, t_{minor} = 11 min);

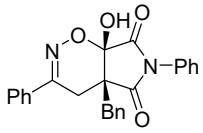
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.85 (s, 1H), 7.80 (d, *J* = 5.5 Hz, 2H), 7.56-7.41 (m, 6H), 7.22 (d, *J* = 6.8 Hz, 2H), 3.17 (d, *J* = 16.5 Hz, 1H), 2.84 (d, *J* = 16.5 Hz, 1H), 1.96-1.72 (m, 2H), 1.50-1.17 (m, 8H), 0.89-0.76 (m, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.8, 164.4, 133.2, 131.0, 130.9, 129.4, 129.0, 128.9, 126.5, 126.1, 96.8, 49.9, 34.3, 30.9, 29.0, 27.0, 24.1, 22.0, 13.9. [α] D^{20}

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{26}N_2O_4H^+$ 407.1965, found 407.1973.

(**3qa**):

(4a*R*,7a*S*)-4a-benzyl-7a-hydroxy-3,6-diphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 97% yield, 95% ee; mp = 169.7-170.7 °C; [α] D^{20} = +335.0 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 16 min, t_{minor} = 19 min);

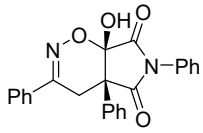
^{1}H NMR (300 MHz, DMSO-d_6) δ 9.25 (s, 1H), 7.92-7.72 (m, 2H), 7.54-7.46 (m, 3H), 7.42-7.35 (m, 3H), 7.29 (s, 5H), 6.72-6.56 (m, 2H), 3.42 (d, *J* = 14.2 Hz, 3H), 3.30 (d, *J* = 17.5 Hz, 1H), 3.10 (d, *J* = 13.5 Hz, 1H), 2.94 (d, *J* = 17.5 Hz, 1H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 175.1, 171.1, 161.1, 135.0, 133.5, 130.7, 130.6, 130.5, 129.1, 128.9, 128.8, 128.4, 127.4, 126.3, 125.9, 95.7, 50.8, 25.7.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{20}N_2O_4H^+$ 413.1496, found 413.1504.

(**3ra**):

(4a*S*,7a*S*)-7a-hydroxy-3,4a,6-triphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 5/1] to afford white solid, 98% yield, 84% ee; mp = 118.2-120.1 °C; [α] D^{20} = +74.5 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 9 min, t_{major} = 13 min);

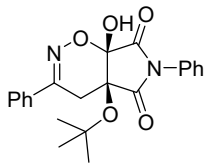
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.95 (s, 1H), 7.94-7.85 (m, 2H), 7.58-7.39 (m, 11H), 7.28 (d, *J* = 6.8 Hz, 2H), 3.70-3.51 (m, 2H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 175.5, 171.9, 167.7, 135.4, 132.7, 131.3, 131.0, 129.5, 129.3, 129.1, 128.5, 128.1, 127.7, 126.7, 126.3, 98.4, 56.7, 28.0.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{18}N_2O_4H^+$ 399.1339, found 399.1345.

(3sa):

(4a*S*,7a*S*)-4a-(tert-butoxy)-7a-hydroxy-3,6-diphenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



White solid, 91% yield, >99% ee; mp = 128.7-129.9 °C; [α] D^{20} = +234.8 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 15min, t_{major} = 20 min); eluent: v(petroleum ether/ethyl acetate)= 10/1 to 5/1

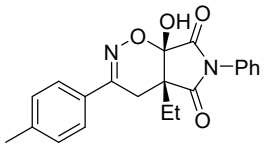
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.72 (s, 1H), 7.82 (d, *J* = 5.9 Hz, 2H), 7.62-7.47 (m, 6H), 7.30 (d, *J* = 7.6 Hz, 2H), 3.51 (d, *J* = 17.4 Hz, 1H), 3.14 (d, *J* = 17.4 Hz, 1H), 1.33 (s, 9H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 174.0, 170.8, 160.2, 133.1, 131.0, 130.8, 129.5, 129.1, 128.8, 126.1, 94.6, 78.8, 75.8, 29.5, 29.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{22}N_2O_5H^+$ 395.1602, found 395.1601.

(3ab):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-6-phenyl-3-(p-tolyl)-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 83% yield, 97% ee; mp = 184.3-185.2 °C; [α] D^{20} = +229.5 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 18 min, t_{minor} = 20 min);

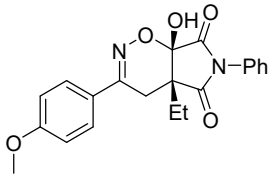
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.79 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.56-7.42 (m, 3H), 7.33-7.19 (m, 4H), 3.13 (d, *J* = 16.4 Hz, 1H), 2.80 (d, *J* = 16.4 Hz, 1H), 2.34 (s, 3H), 2.02-1.77 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.8, 164.5, 140.8, 131.0, 130.4, 129.4, 129.3, 129.0, 126.6, 126.0, 96.8, 50.3, 27.2, 26.7, 20.9, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_4H^+$ 365.1496, found 365.1503.

(3ac):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-(4-methoxyphenyl)-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 87% yield, 96% ee; mp = 161.9-163.7 °C; [α] D^{20} = +264.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 11 min, t_{major} = 15 min);

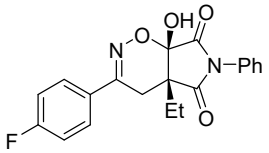
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.75 (s, 1H), 7.76 (d, *J* = 8.9 Hz, 2H), 7.59-7.34 (m, 3H), 7.21 (d, *J* = 6.8 Hz, 2H), 7.03 (d, *J* = 8.9 Hz, 2H), 3.80 (s, 3H), 3.12 (d, *J* = 16.1 Hz, 1H), 2.80 (d, *J* = 16.1 Hz, 1H), 2.04-1.74 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.9, 164.5, 161.4, 131.0, 129.3, 129.0, 127.7, 126.6, 125.3, 114.2, 96.8, 55.3, 50.4, 27.1, 26.9, 9.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_5H^+$ 381.1445, found 381.1454.

(**3ad**):

(4a*R*,7a*S*)-4a-ethyl-3-(4-fluorophenyl)-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 87% yield, 95% ee; mp = 181.2-182.3 °C; [α] D^{20} = +210.9 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 17 min, t_{minor} = 18 min);

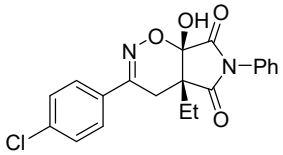
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.86 (s, 1H), 7.96-7.75 (m, 2H), 7.61-7.40 (m, 3H), 7.39-7.11 (m, 4H), 3.16 (d, *J* = 16.7 Hz, 1H), 2.83 (d, *J* = 16.7 Hz, 1H), 2.00-1.80 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.7, 163.7 (d, *J* = 248.5 Hz), 163.1, 131.0, 129.8 (d, *J* = 2.7 Hz), 129.4, 129.1, 128.5 (d, *J* = 8.7 Hz), 126.6, 115.9 (d, *J* = 21.9 Hz), 96.6, 50.1, 27.5, 26.4, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}FN_2O_4H^+$ 369.1245, found 369.1258.

(**3ae**):

(4a*R*,7a*S*)-3-(4-chlorophenyl)-4a-ethyl-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 95% yield, 95% ee; mp = 135.8-137.6 °C; [α] D^{20} = +215.8 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 19 min, t_{minor} = 21 min);

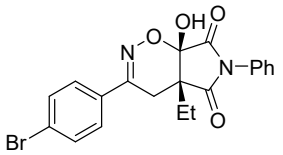
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.90 (s, 1H), 8.00-7.72 (m, 2H), 7.61-7.42 (m, 5H), 7.24 (d, *J* = 6.7 Hz, 2H), 3.17 (d, *J* = 16.8 Hz, 1H), 2.82 (d, *J* = 16.9 Hz, 1H), 2.08-1.72 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.2, 171.7, 162.8, 135.7, 132.1, 131.0, 129.4, 129.1, 128.9, 127.9, 126.6, 96.6, 49.9, 27.7, 26.1, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}ClN_2O_4H^+$ 385.0950, 387.0928, found 385.0957, 387.0936.

(**3af**):

(4a*R*,7a*S*)-3-(4-bromophenyl)-4a-ethyl-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 89% yield,95% ee; mp = 144.8-145.9 °C; [α] D^{20} = +208.9 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 20 min, t_{minor} = 22 min);

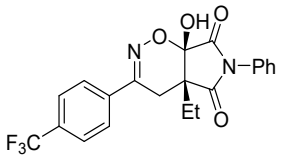
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.88 (s, 1H), 7.85-7.64 (m, 4H), 7.57-7.43 (m, 3H), 7.27-7.16 (m, 2H), 3.16 (d, *J* = 16.8 Hz, 1H), 2.82 (d, *J* = 16.8 Hz, 1H), 2.05-1.75 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.2, 171.6, 162.9, 132.4, 131.8, 130.9, 129.3, 129.0, 128.0, 126.6, 124.5, 96.6, 49.9, 27.6, 26.0, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}BrN_2O_4H^+$ 429.0444; 431.0427, found 429.0453, 431.0435.

(3ag):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-6-phenyl-3-(4-(trifluoromethyl)phenyl)-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 91% yield, 95% ee; mp = 173.1-174.4 °C; [α] D^{20} = +205.5 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 16 min, t_{major} = 17 min);

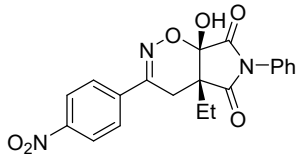
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.97 (s, 1H), 8.03 (d, *J* = 8.2 Hz, 2H), 7.85 (d, *J* = 8.2 Hz, 2H), 7.61-7.44 (m, 3H), 7.31-7.21 (m, 2H), 3.23 (d, *J* = 17.0 Hz, 1H), 2.87 (d, *J* = 17.1 Hz, 1H), 2.04-1.83 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, DMSO-d_6) δ 176.2, 171.6, 162.2, 137.2, 131.0, 130.7 (q, *J* = 32.6, 32.1 Hz), 129.4, 129.2, 129.1, 127.3, 127.0, 126.6, 125.8 (q, *J* = 3.5 Hz), 124.0 (d, *J* = 272.4 Hz), 96.6, 49.7, 27.9, 25.8, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}F_3N_2O_4H^+$ 419.1213, found 419.1223.

(**3ah):**

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-(4-nitrophenyl)-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 86% yield, 93% ee; mp = 184.8-186.8 °C; [α] D^{20} = +215.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 50/50, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 8 min, t_{major} = 9 min);

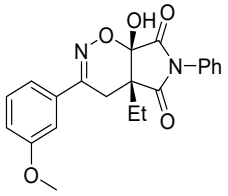
^{1}H NMR (300 MHz, DMSO-d_6) δ 9.05 (s, 1H), 8.31 (d, *J* = 8.5 Hz, 2H), 8.08 (d, *J* = 8.5 Hz, 2H), 7.57-7.43 (m, 3H), 7.27 (d, *J* = 7.0 Hz, 2H), 3.26 (d, *J* = 17.3 Hz, 1H), 2.89 (d, *J* = 17.3 Hz, 1H), 2.07-1.76 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.0, 171.4, 161.3, 148.7, 139.3, 130.9, 129.4, 129.1, 127.4, 126.6, 123.9, 96.6, 49.5, 28.1, 25.5, 9.0.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{17}N_3O_6H^+$ 396.1190, found 396.1197.

(**3ai**):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-(3-methoxyphenyl)-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 97% yield, 95% ee; mp = 159.2-160.6 °C; [α] D^{20} = +126.6 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 16 min, t_{major} = 17 min);

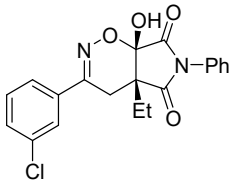
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.85 (s, 1H), 7.56-7.33 (m, 5H), 7.32-7.28 (m, 1H), 7.27-7.21 (m, 2H), 7.13-7.05 (m, 1H), 3.80 (s, 3H), 3.15 (d, *J* = 16.6 Hz, 1H), 2.82 (d, *J* = 16.6 Hz, 1H), 2.07-1.69 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.3, 171.8, 164.0, 159.4, 134.6, 131.0, 130.0, 129.4, 129.1, 126.6, 118.5, 116.7, 111.1, 96.7, 55.3, 50.2, 27.5, 26.7, 9.2.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_5H^+$ 381.1445, found 381.1451.

(**3aj**):

(4a*R*,7a*S*)-3-(3-chlorophenyl)-4a-ethyl-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 89% yield, 94% ee; mp = 166.2-167.7 °C; [α] D^{20} = +208.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 12 min, t_{minor} = 13 min);

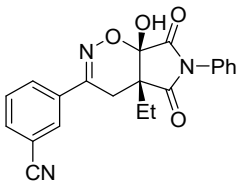
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.91 (s, 1H), 7.88-7.70 (m, 2H), 7.63-7.46 (m, 5H), 7.31-7.20 (m, 2H), 3.19 (d, *J* = 17.0 Hz, 1H), 2.83 (d, *J* = 17.1 Hz, 1H), 2.01-1.81 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.1, 171.6, 162.2, 135.4, 133.7, 131.0, 130.8, 130.6, 129.4, 129.1, 126.6, 125.7, 124.7, 96.6, 49.8, 27.8, 25.9, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}N_2O_5H^+$ 385.0950; 387.0928, found385.0961; 387.0941.

(**3ak**):

3-((4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-5,7-dioxo-6-phenyl-4,4a,5,6,7,7a-hexahydropyrrolo[3,4-*e*][1,2]oxazin-3-yl)benzonitrile



It was purified by flash chromatography [V(PE/EA) = 10/1 to 5/1] to afford white solid, 89% yield, 95% ee; mp = 198.4-200.2 °C; [α] D^{20} = +301.9 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 26 min, t_{minor} = 29 min);

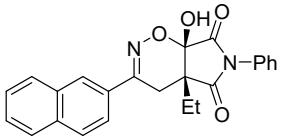
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.96 (s, 1H), 8.29 (s, 1H), 8.15 (d, *J* = 9.0 Hz, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.75-7.64 (m, 1H), 7.56-7.43 (m, 3H), 7.26 (d, *J* = 7.8 Hz, 2H), 3.24 (d, *J* = 17.3 Hz, 1H), 2.87 (d, *J* = 17.4 Hz, 1H), 1.92 (q, *J* = 7.3 Hz, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.1, 171.5, 161.2, 134.5, 134.3, 131.0, 130.5, 130.1, 129.9, 129.4, 129.1, 126.7, 118.3, 112.2, 96.4, 49.5, 28.1, 25.3, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}N_3O_4H^+$ 376.1292, found 376.1302.

(3al):

(4a*R*,7a*S*)-4a-ethyl-7a-hydroxy-3-(naphthalen-2-yl)-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 8/1] to afford white solid, 93% yield, 96% ee; mp = 153.7-156.5 °C; [α] D^{20} = +361.5 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 22 min, t_{minor} = 26 min);

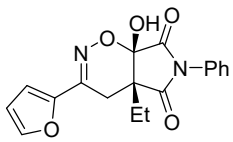
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.91 (s, 1H), 8.44 (s, 1H), 8.15-7.88 (m, 4H), 7.67-7.40 (m, 5H), 7.34-7.12 (m, 2H), 3.34 (d, *J* = 16.6 Hz, 1H), 2.99 (d, *J* = 16.5 Hz, 1H), 2.19-1.65 (m, 2H), 1.02 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.4, 171.8, 163.9, 133.9, 132.6, 131.0, 130.5, 129.4, 129.1, 128.8, 128.4, 127.6, 126.9, 126.7, 126.7, 122.5, 96.8, 50.2, 27.6, 26.3, 9.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{20}N_2O_4H^+$ 401.1496, found 401.1500.

(3am):

(4a*R*,7a*S*)-4a-ethyl-3-(furan-2-yl)-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 5/1 to 2/1] to afford white solid, 21% yield, 97% ee; mp = 178.9-182.3 °C; [α] D^{20} = +127.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 13 min, t_{major} = 16 min);

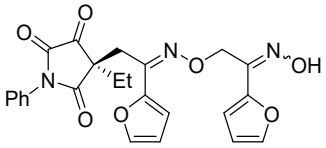
^{1}H NMR (300 MHz, DMSO-d_6) δ 9.15 (s, 1H), 7.95 (s, 1H), 7.75 (d, J = 3.5 Hz, 1H), 7.54-7.45 (m, 3H), 7.36-7.27 (m, 2H), 6.85-6.69 (m, 1H), 3.51 (d, J = 18.3 Hz, 1H), 3.19 (d, J = 18.3 Hz, 1H), 1.99 (dq, J = 11.5, 6.7 Hz, 2H), 0.99 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.2, 168.7, 145.4, 144.6, 132.1, 131.1, 129.2, 129.1, 126.9, 114.7, 112.6, 98.7, 51.2, 33.7, 25.0, 8.9.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{18}H_{16}N_2O_5H^+$ 341.1132, found 341.1143.

(3am'):

(*R*)-4-ethyl-4-((2*Z*)-2-(furan-2-yl)-2-((2-(furan-2-yl)-2-(hydroxyimino)ethoxy)imino)ethyl)-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 8/1] to afford white solid, 80% yield, 90% ee (1.2 eq: 98% yield, 90% ee); mp = 105.1-107.0 °C; [α] D^{20} = +124.7 (c 0.5, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 21 min); eluent: v(petroleum ether/ethyl acetate)= 10/1 to 8/1

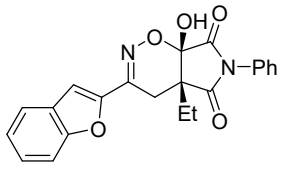
^{1}H NMR (300 MHz, DMSO-d_6) δ 12.09 (s, 1H), 7.89 (s, 1H), 7.77 (s, 1H), 7.64-7.42 (m, 5H), 7.34 (d, J = 3.5 Hz, 1H), 7.03 (d, J = 3.6 Hz, 1H), 6.73-6.56 (m, 2H), 4.99-4.75 (m, 2H), 3.46 (d, J = 4.0 Hz, 2H), 2.08 (q, J = 7.4 Hz, 2H), 1.01 (t, J = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 194.4, 174.9, 160.3, 144.9, 144.4, 143.3, 143.2, 142.1, 140.9, 131.0, 129.4, 129.1, 126.3, 118.9, 117.3, 112.6, 112.1, 73.6, 50.6, 34.0, 28.3, 8.3.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{18}H_{16}N_2O_5H^+$ 464.1452, found 464.1461.

(3an):

(4a*R*,7a*S*)-3-(benzofuran-2-yl)-4a-ethyl-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 8/1 to 5/1] to afford white solid, 77% yield,94% ee; mp = 189.7-190.1 °C; [α] D^{20} = +197.0 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24 min, t_{minor} = 38 min);

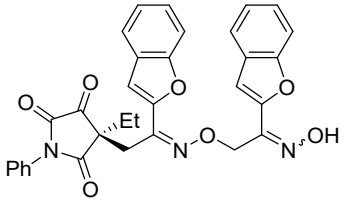
^{1}H NMR (300 MHz, DMSO-d_6) δ 8.96 (s, 1H), 7.81-7.60 (m, 3H), 7.57-7.42 (m, 4H), 7.37-7.22 (m, 3H), 3.21 (d, J = 16.4 Hz, 1H), 2.93 (d, J = 16.6 Hz, 1H), 2.11-1.72 (m, 2H), 0.99 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 176.0, 171.4, 156.3, 154.9, 148.7, 130.9, 129.3, 129.1, 127.3, 126.9, 126.6, 123.7, 122.2, 111.6, 110.2, 97.2, 49.6, 27.5, 25.4, 9.1.

HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{18}N_2O_5H^+$ 391.1289, found 391.1288.

(3an'):

(*R*)-4-((2*Z*)-2-(benzofuran-2-yl)-2-((2-(benzofuran-2-yl)-2-(hydroxyimino)ethoxy)imino) ethyl)-4-ethyl-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 8/1] to afford white solid, 33% yield, 90% ee (1.2 eq: 95% yield, 90% ee); mp = 205.6-207.1 °C; [α] D^{20} = +171.2 (c 0.5, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 20 min);

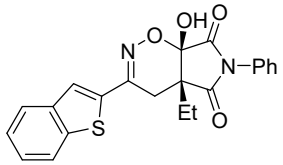
^{1}H NMR (300 MHz, DMSO-d_6) δ 12.63 (s, 1H), 7.83 (s, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.71-7.36 (m, 7H), 7.35-7.19 (m, 2H), 5.26-5.02 (m, 2H), 3.61 (d, *J* = 3.5 Hz, 2H), 2.14 (q, *J* = 7.4 Hz, 2H), 1.04 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 206.1, 194.6, 174.9, 160.3, 153.1, 152.7, 145.4, 144.2, 143.1, 141.6, 131.0, 129.5, 129.2, 127.8, 127.5, 127.2, 126.6, 126.4, 123.9, 123.5, 122.7, 122.5, 114.7, 113.4, 111.7, 111.4, 74.4, 50.8, 34.1, 28.5, 8.5.

HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{32}H_{25}N_3O_7H^+$ 564.1765, found 564.1766.

(**3ao**):

(4a*R*,7a*S*)-3-(benzo[b]thiophen-2-yl)-4a-ethyl-7a-hydroxy-6-phenyl-4a,7a-dihydropyrrolo[3,4-*e*][1,2]oxazine-5,7(4*H*,6*H*)-dione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 8/1] to afford white solid, 73% yield, 94% ee; mp = 167.8-169.1 °C; [α] D^{20} = +203.0 (c 0.2, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 23 min, t_{minor} = 28 min);

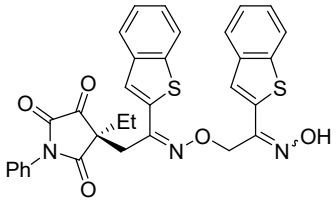
^{1}H NMR (600 MHz, DMSO-d_6) δ 8.98 (s, 1H), 8.17 (s, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.91 (d, *J* = 7.5 Hz, 1H), 7.55-7.42 (m, 5H), 7.25 (d, *J* = 7.7 Hz, 2H), 3.34 (d, *J* = 16.6 Hz, 1H), 2.99 (d, *J* = 16.6 Hz, 1H), 1.98-1.88 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (150 MHz, DMSO-d_6) δ 176.0, 171.4, 160.2, 139.6, 138.9, 136.3, 131.0, 129.4, 129.1, 127.1, 126.7, 126.6, 125.0, 124.8, 122.7, 97.2, 49.6, 27.6, 25.8, 9.1.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{18}N_2O_4S\ H^+$ 407.1072, found 407.1060.

(3ao'):

(*R*)-4-((2*Z*)-2-(benzo[b]thiophen-2-yl)-2-((2-(benzo[b]thiophen-2-yl)-2(hydroxyimino)ethoxy)imino)ethyl)-4-ethyl-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA) = 8/1 to 4/1] to afford white solid, 28% yield, 96% ee (1.2 eq: 96% yield, 90% ee); mp = 115.4-116.4 °C; [α] D^{20} = +202.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 41 min, t_{minor} = 44 min);

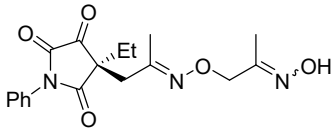
^{1}H NMR (300 MHz, DMSO-d_6) δ 12.67 (s, 1H), 8.20 (s, 1H), 8.04-7.73 (m, 5H), 7.64-7.37 (m, 9H), 5.28-5.03 (m, 2H), 3.89-3.65 (m, 2H), 2.16 (q, *J* = 7.4 Hz, 2H), 1.11 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 212.0, 192.4, 177.9, 164.1, 162.4, 159.0, 158.4, 154.5, 154.2, 148.5, 147.6, 146.8, 146.7, 146.6, 146.5, 144.1, 143.8, 143.7, 143.3, 142.4, 142.0, 141.9, 139.5, 93.4, 68.4, 53.6, 45.9, 25.8.

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{32}H_{25}N_3O_5S_2H^+$ 596.1318, found 596.1308.

(3ap'):

(*R*)-4-ethyl-4-((2*E*)-2-((2-(hydroxyimino)propoxy)imino)propyl)-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA/DCM) = 10/1/1 to 5/1/1] to afford white solid, 78% yield, 84% ee; mp = 131.1-131.9 °C; [α] D^{20} = +19.9 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 13.6 min, t_{minor} = 14.6 min);

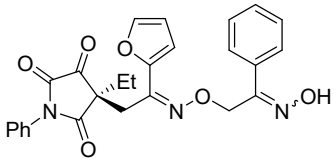
^{1}H NMR (300 MHz, DMSO-d_6) δ 10.85 (s, 1H), 7.69-7.26 (m, 5H), 4.39-4.16 (m, 2H), 3.00 (s, 2H), 1.94 (q, *J* = 7.5 Hz, 2H), 1.81 (s, 3H), 1.65 (s, 3H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 194.6 , 175.0 , 160.2 , 155.1 , 151.8 , 131.1 , 129.3 , 129.1 , 126.3 , 75.0 , 50.6 , 28.1 , 14.3 , 11.3 , 8.2 .

HRMS(ESI) m/z: $[M+H]^+$ calcd for $C_{18}H_{21}N_3O_5H^+$ 360.1554, found 360.1556.

(3ama):

(*R*)-4-ethyl-4-((2*Z*)-2-(furan-2-yl)-2-((2-(hydroxyimino)-2-phenylethoxy)imino)ethyl)-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA) = 5/1] to afford white solid, 95% yield, 87% ee; mp = 116.0-117.7 °C; [α] D^{20} = +66.1 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 29 min, t_{minor} =39 min);

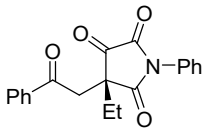
^{1}H NMR (300 MHz, DMSO-d_6) δ 11.40 (s, 1H), 7.84 (s, 1H), 7.63-7.48 (m, 5H), 7.45-7.29 (m, 5H), 6.80 (d, J = 3.6 Hz, 1H), 6.62-6.45 (m, 1H), 4.98-4.64 (m, 2H), 3.41-3.32 (m, 2H), 2.05 (q, J = 7.3 Hz, 2H), 0.99 (t, J = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 194.4 , 174.8 , 160.3 , 150.7 , 144.9 , 143.2 , 142.4 , 131.6 , 131.0 , 129.4 , 129.2 , 128.9 , 128.3 , 128.1 , 126.5 , 126.3 , 118.9 , 112.5 , 75.9 , 50.6 , 34.0 , 28.3 , 8.3 .

HRMS(ESI) m/z[M+H]$^+$ calcd for $C_{26}H_{23}N_3O_6$ H$^+$ 474.1660, found 474.1663.

(**5**):

(*R*)-4-ethyl-4-(2-oxo-2-phenylethyl)-1-phenylpyrrolidine-2,3,5-trione



It was purified by flash chromatography [V(PE/EA) = 10/1 to 8/1] to afford yellow solid, 99% yield, 93% ee; mp = 117.0-118.5 ^{o}C; [α] D^{20} = +42.6 (c 1.0, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 12 min, t_{minor} = 13 min); eluent: v(petroleum ether/ethyl acetate)= 8/1

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.16-7.95 (m, 2H), 7.75-7.66 (m, 1H), 7.65-7.49 (m, 5H), 7.43-7.36 (m, 2H), 4.16 (q, J = 19.2 Hz, 2H), 2.13-1.99 (m, 2H), 1.04 (t, J = 7.4 Hz, 3H).

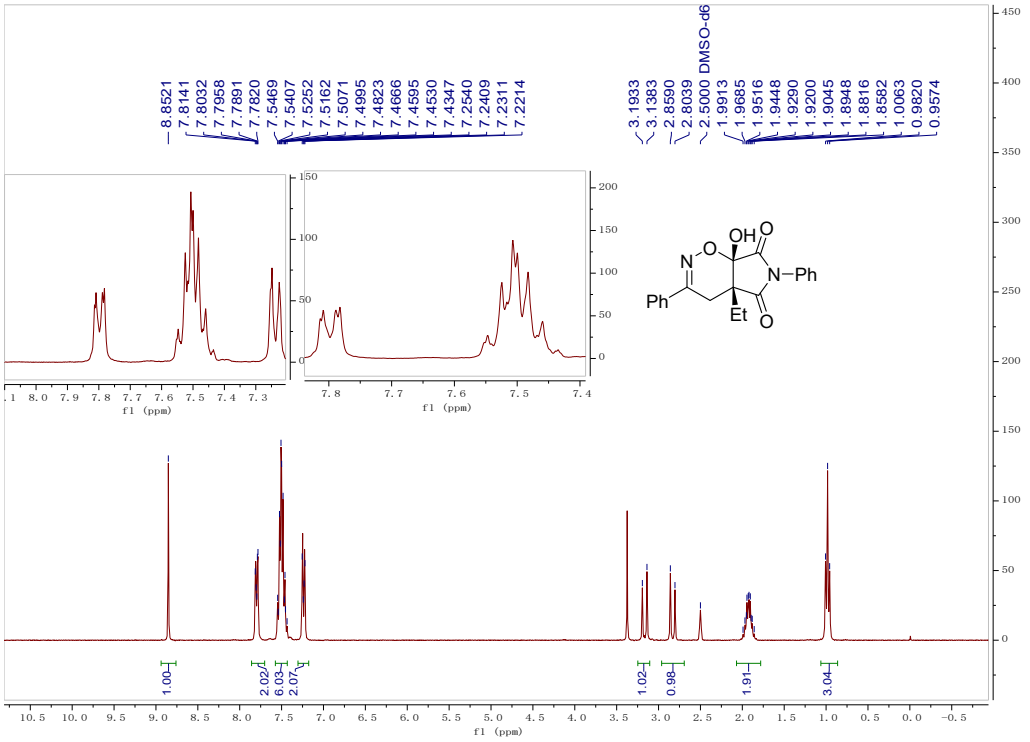
^{13}C NMR (75 MHz, DMSO-d_6) δ 198.9, 196.1, 175.5, 160.0, 134.7, 134.0, 131.2, 129.4, 129.3, 129.0, 128.8, 126.6, 50.7, 45.7, 26.7, 8.5.

HRMS(ESI) m/z: [M+ Na]$^+$ calcd for $C_{20}H_{17}NO_4Na^+$ 358.1050, found 358.1050.

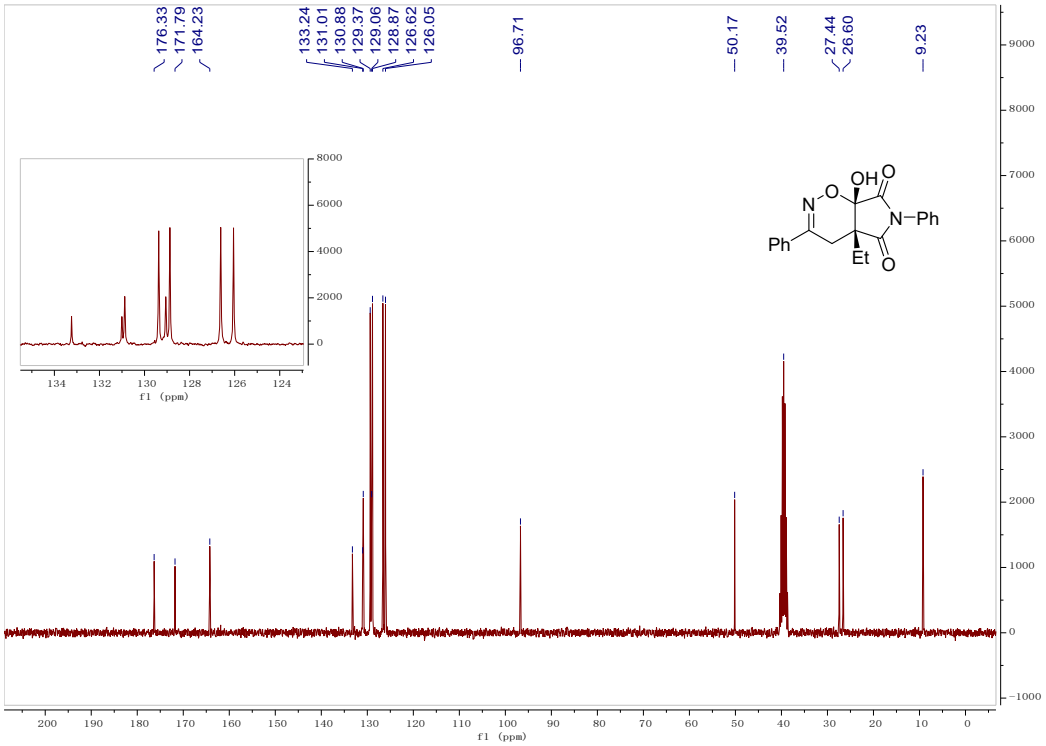
5 HPLC, NMR and HRMS spectra of products

3aa

^{1}H NMR (300 MHz, DMSO-d_6)

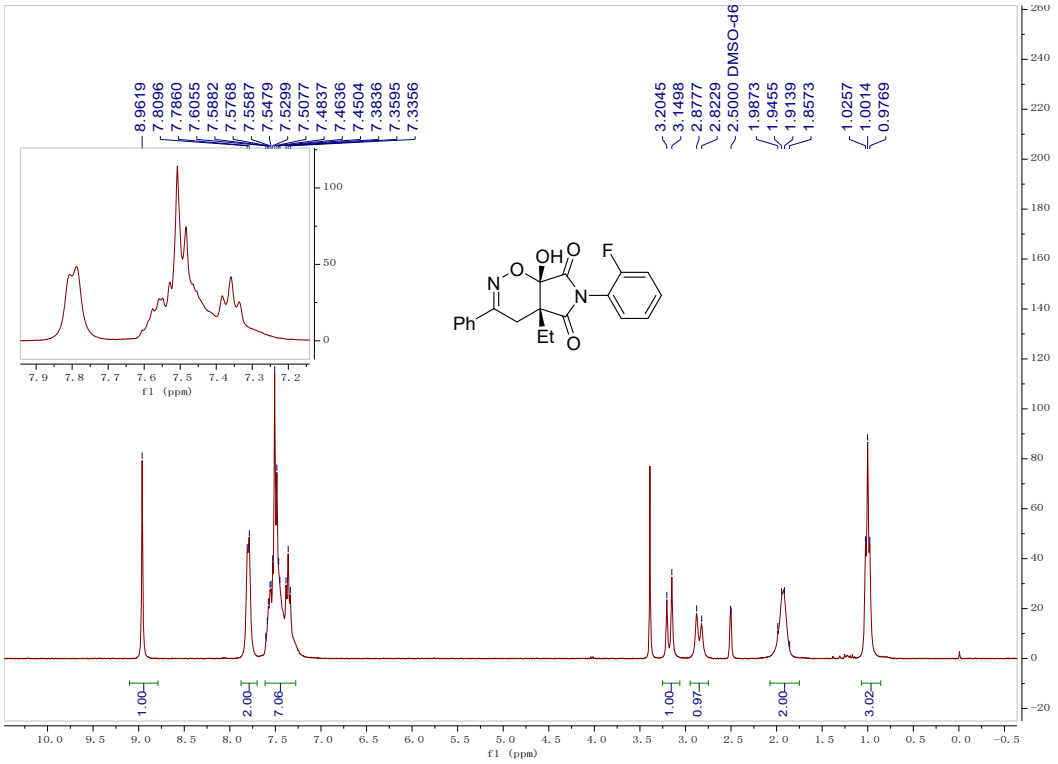


^{13}C NMR (75 MHz, DMSO-d_6)

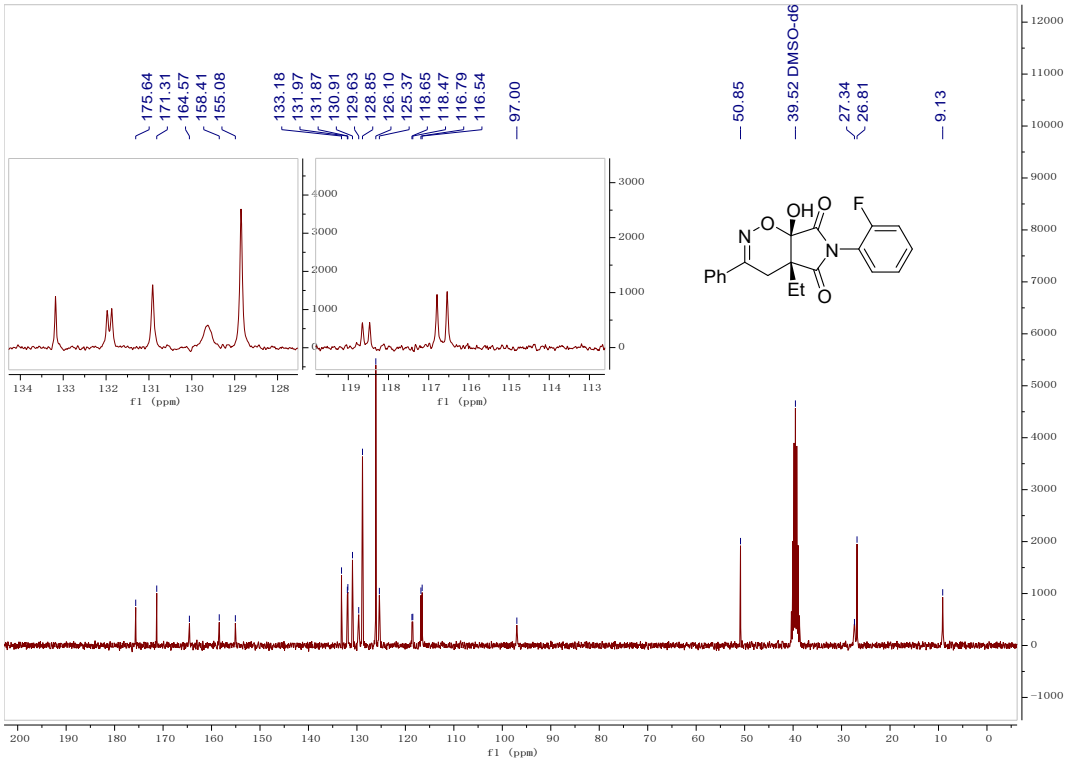


3ba

^{1}H NMR (300 MHz, DMSO-d_6)

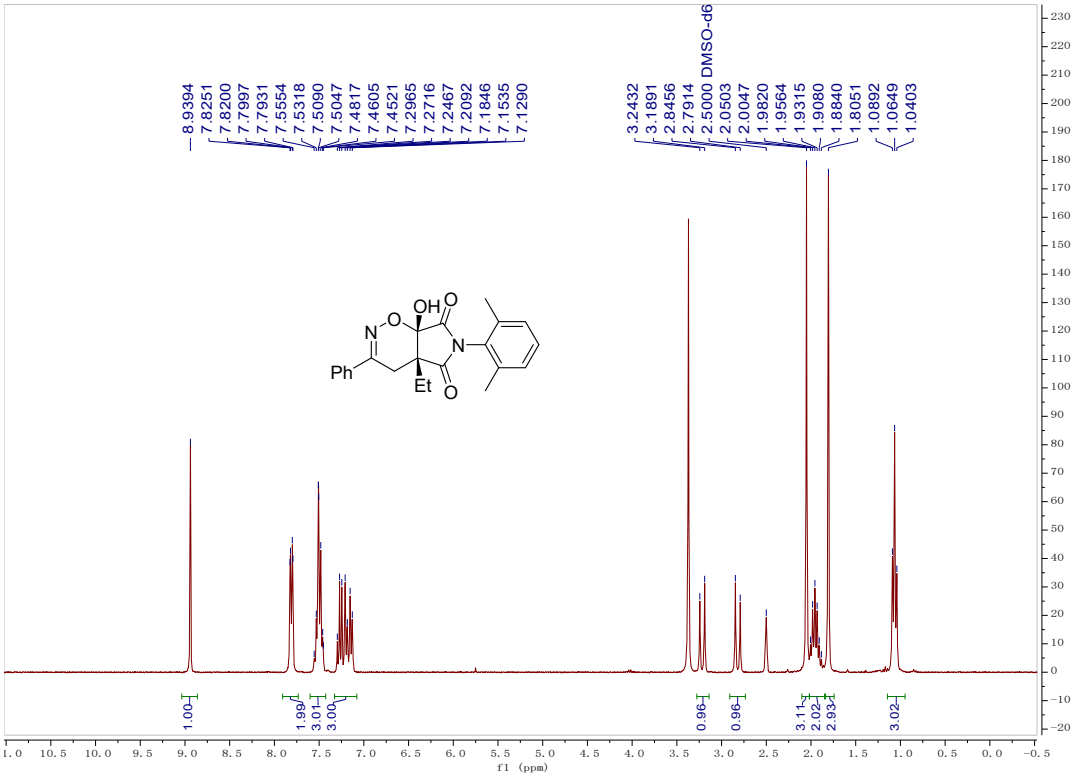


^{13}C NMR (75 MHz, DMSO-d_6)

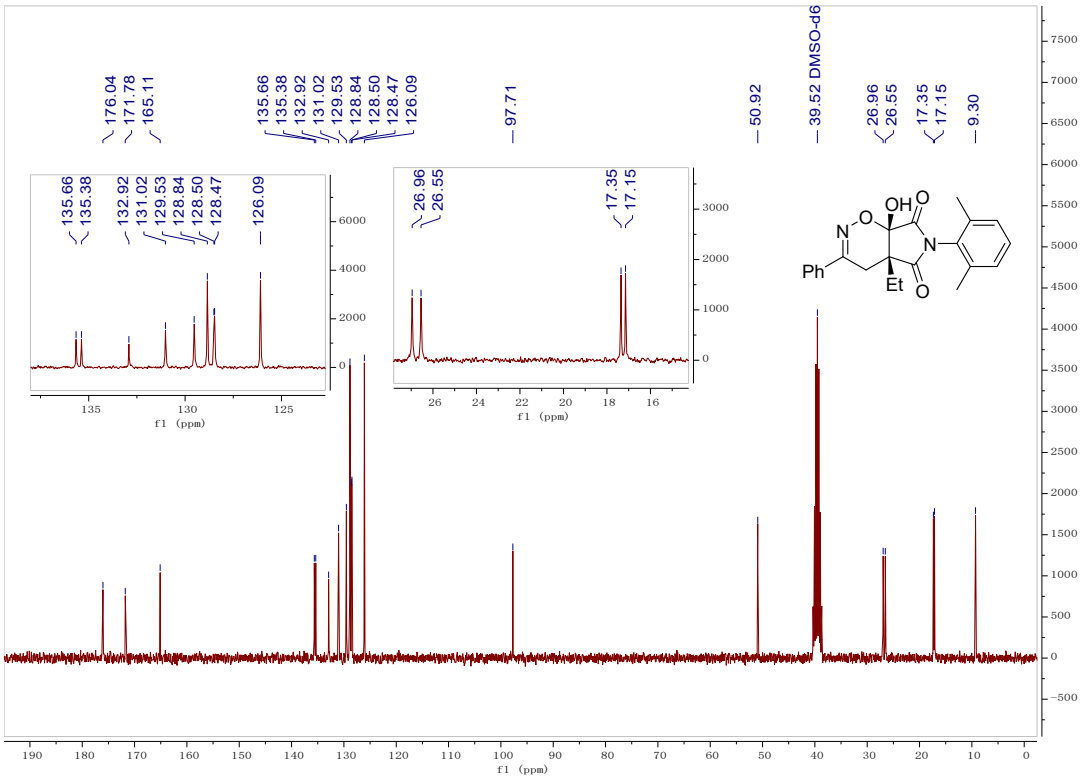


3ca

^{1}H NMR (300 MHz, DMSO-d_6)

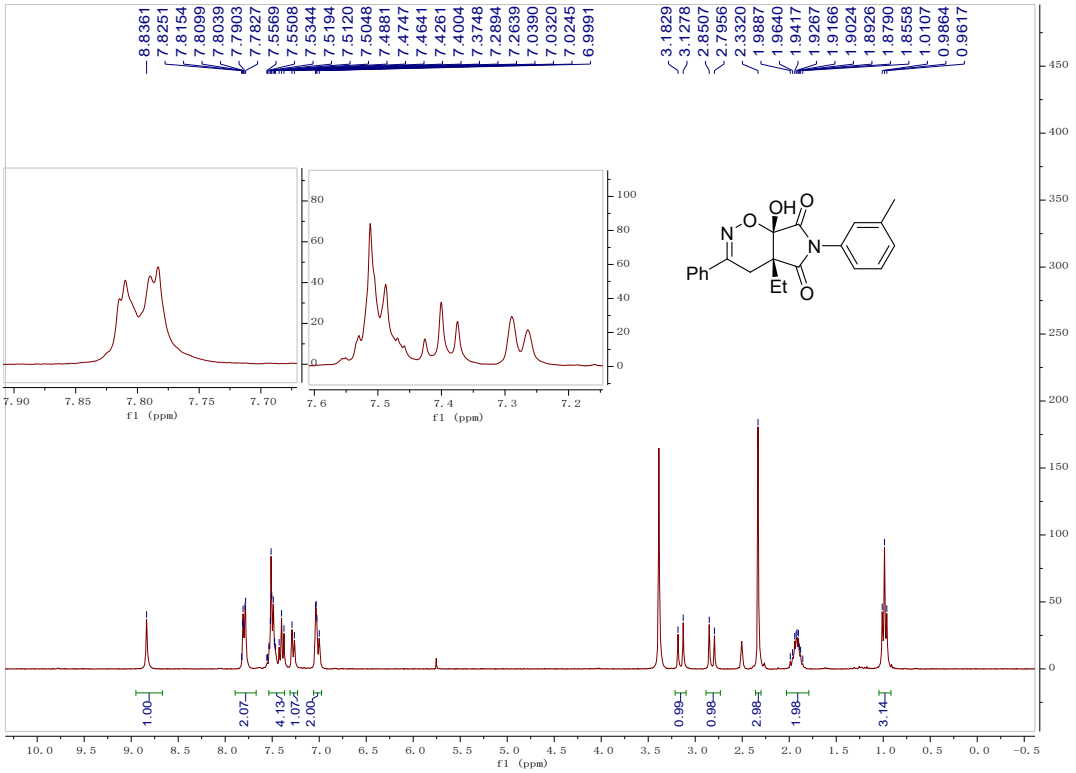


^{13}C NMR (75 MHz, DMSO-d_6)

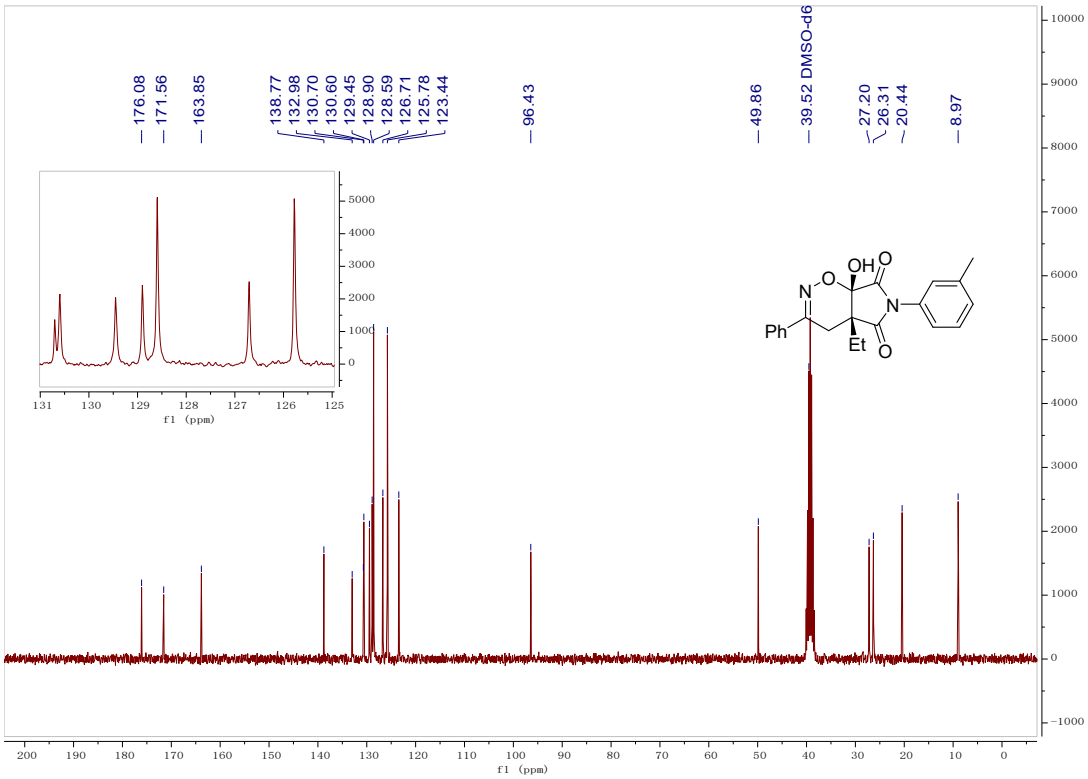


3da

^{1}H NMR (300 MHz, DMSO-d_6)

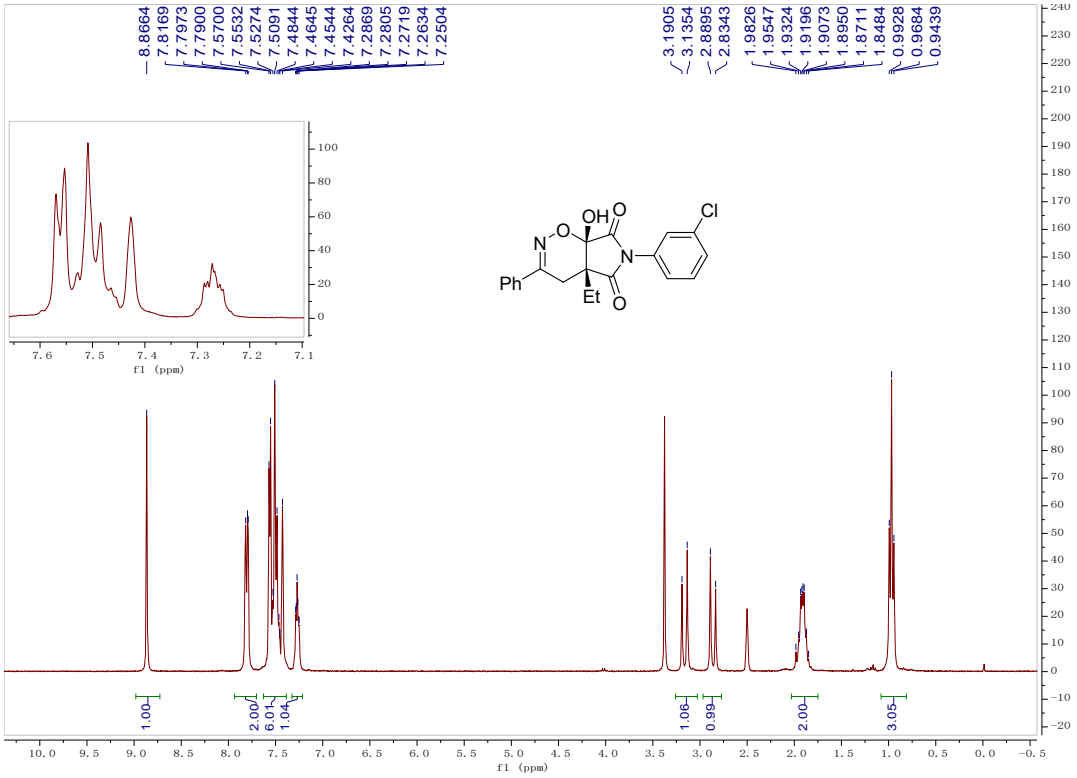


^{13}C NMR (75 MHz, DMSO-d_6)

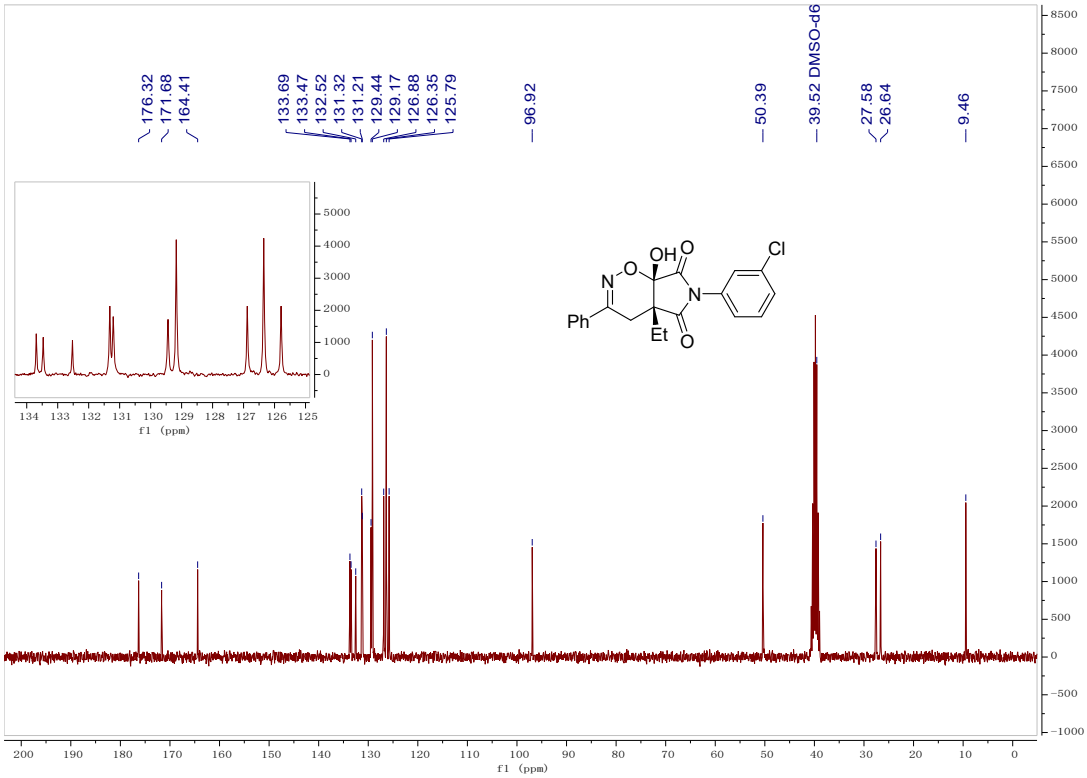


3ea

^{1}H NMR (300 MHz, DMSO-d_6)

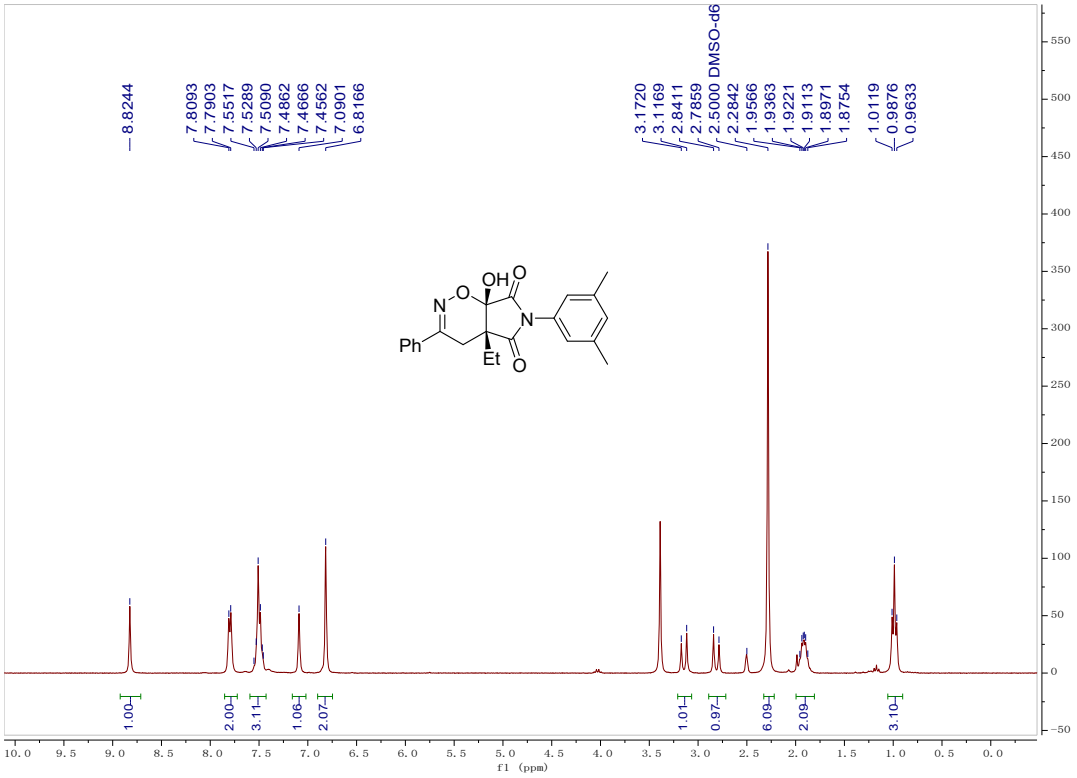


^{13}C NMR (75 MHz, DMSO-d_6)

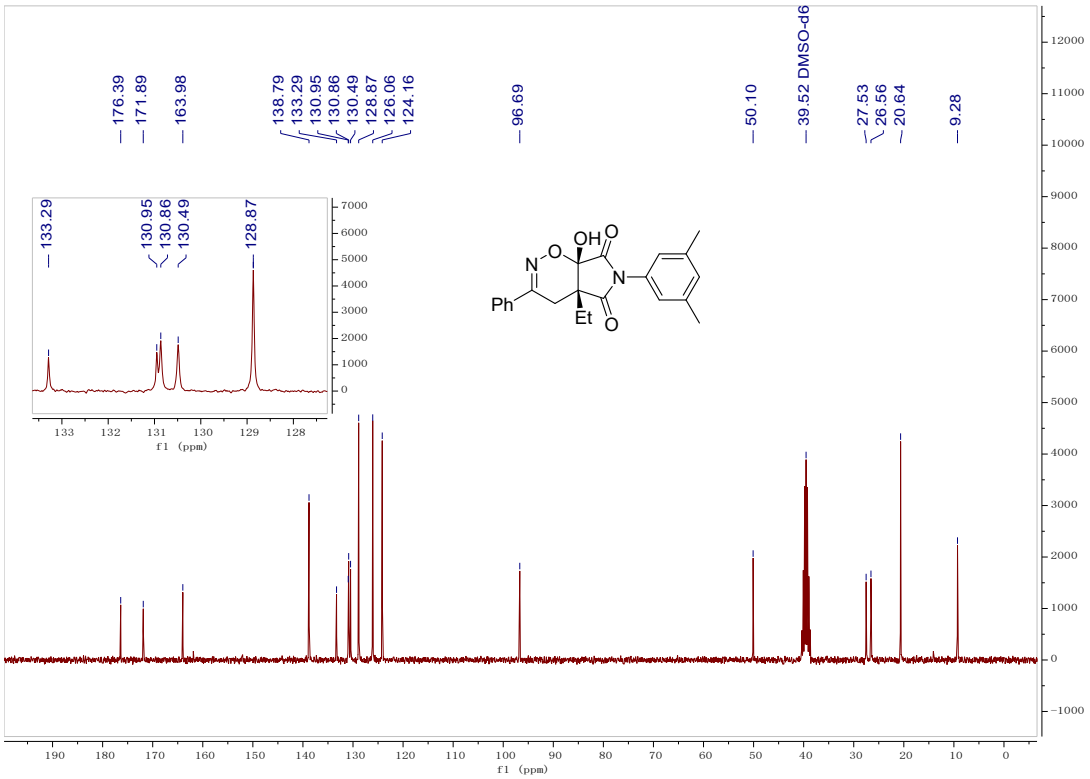


3fa

^{1}H NMR (300 MHz, DMSO-d_6)

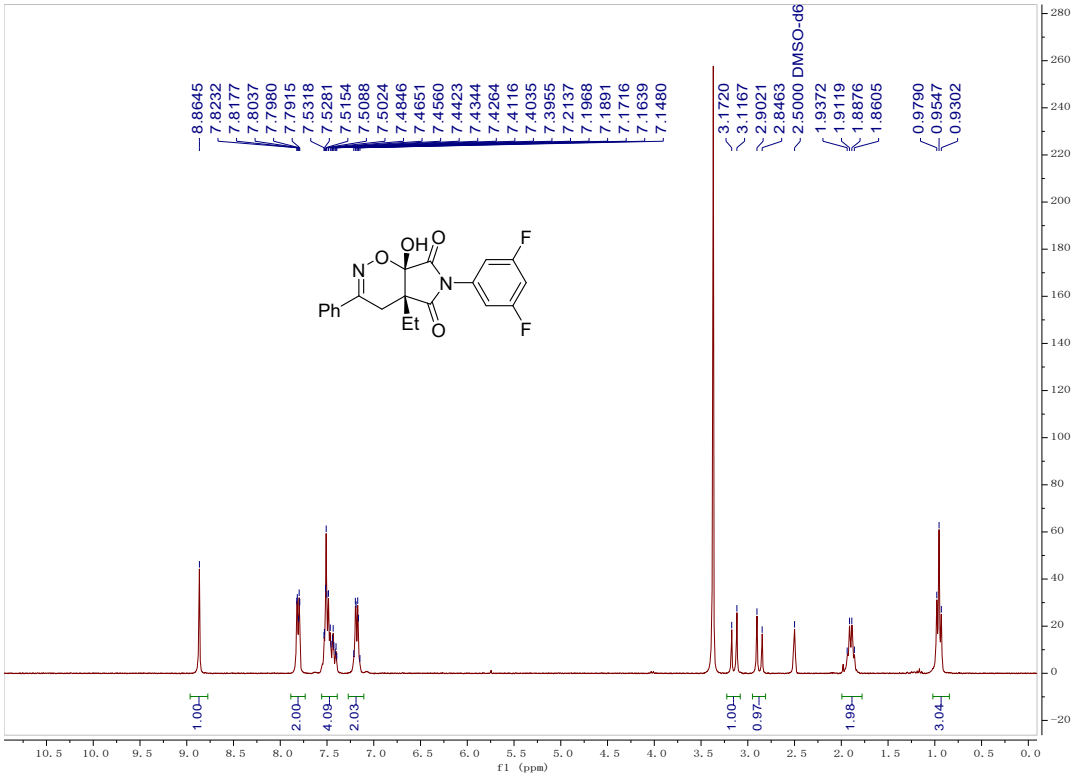


^{13}C NMR (75 MHz, DMSO-d_6)

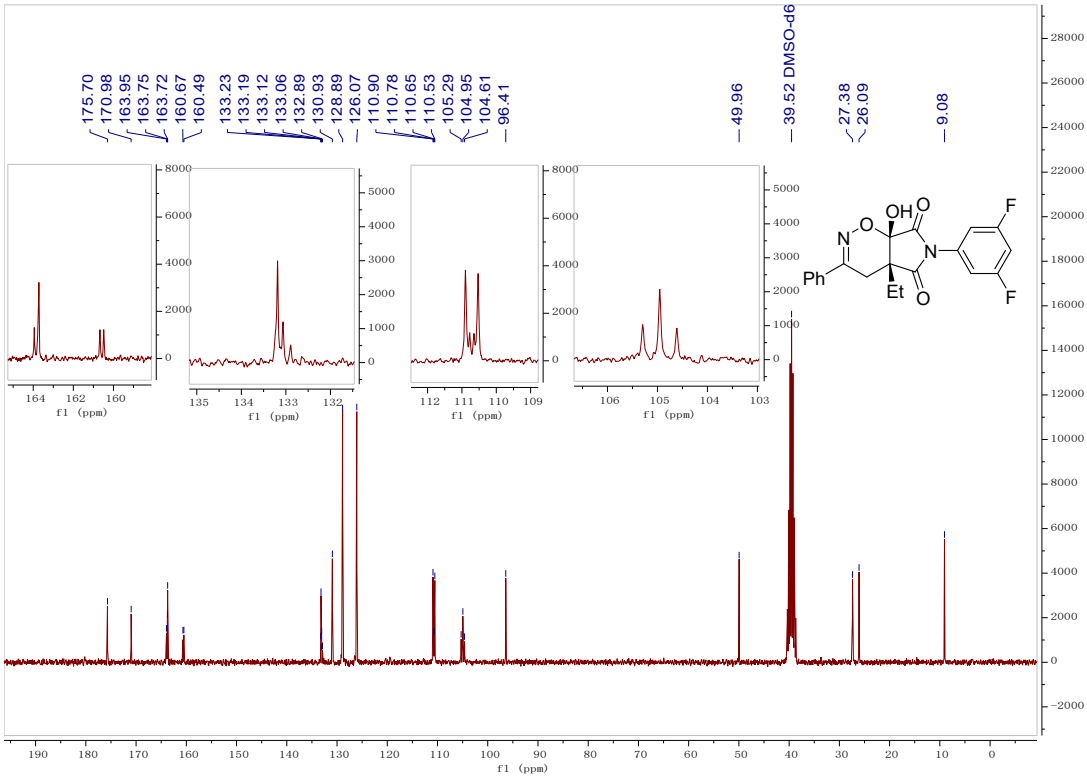


3ga

^{1}H NMR (300 MHz, DMSO-d_6)

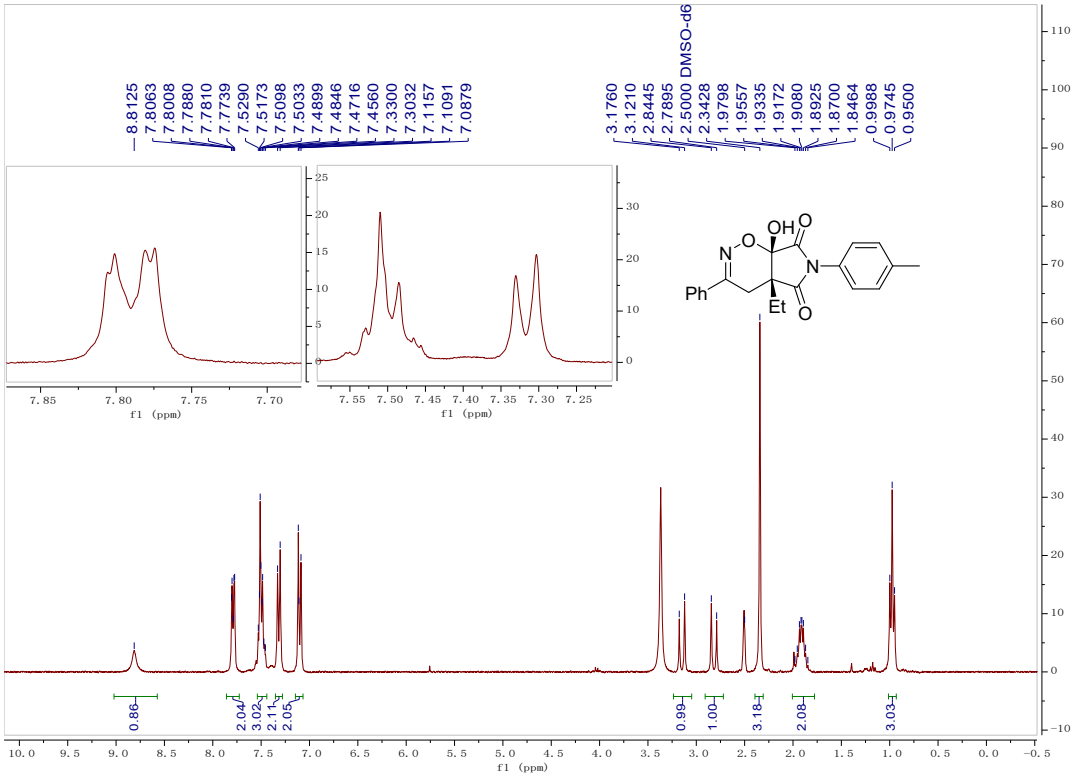


^{13}C NMR (75 MHz, DMSO-d_6)

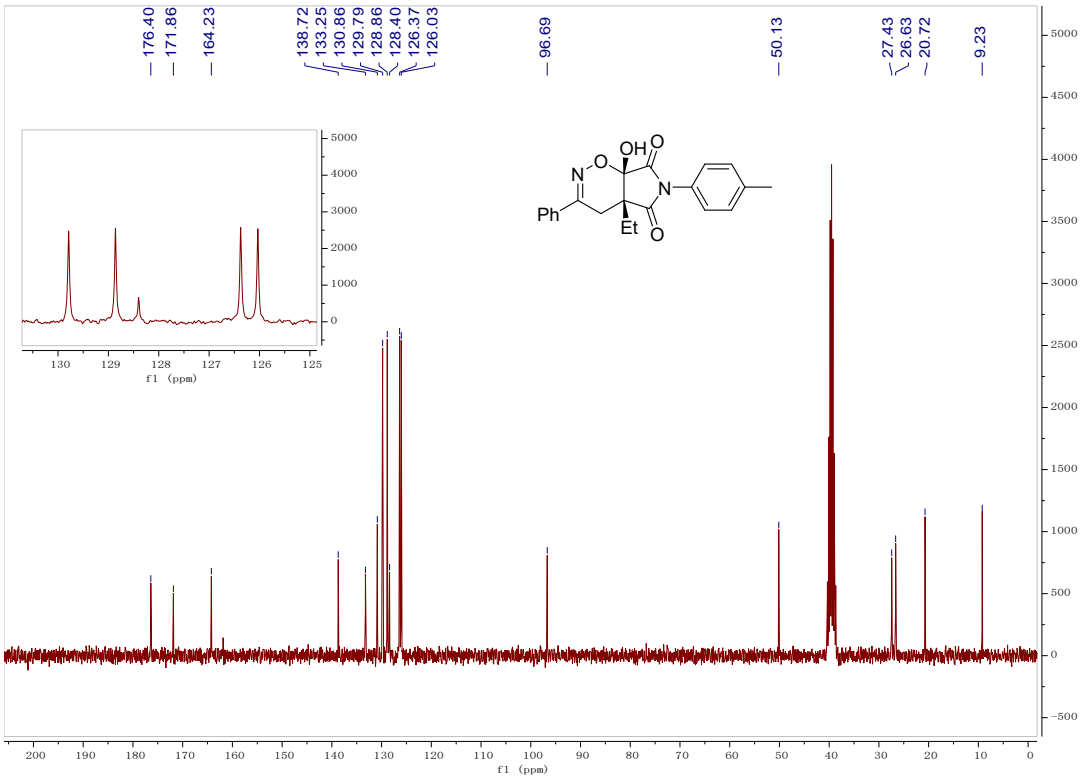


3ha

^{1}H NMR (300 MHz, DMSO-d_6)

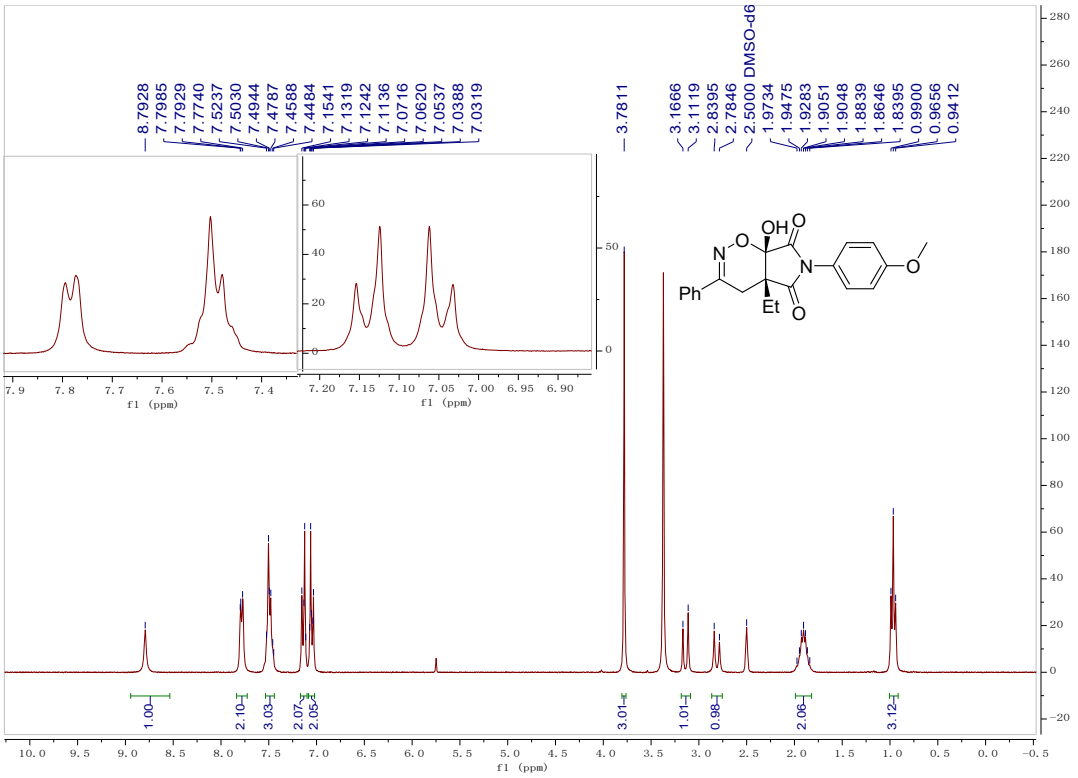


^{13}C NMR (75 MHz, DMSO-d_6)

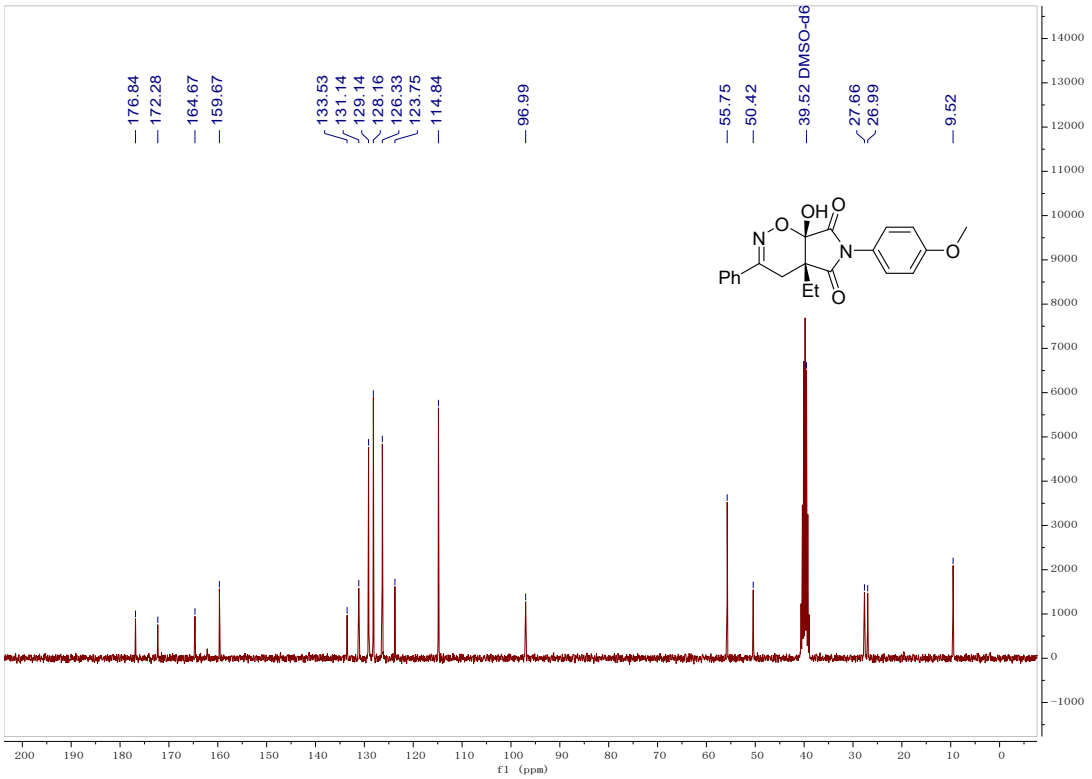


3ia

^{1}H NMR (300 MHz, DMSO-d_6)

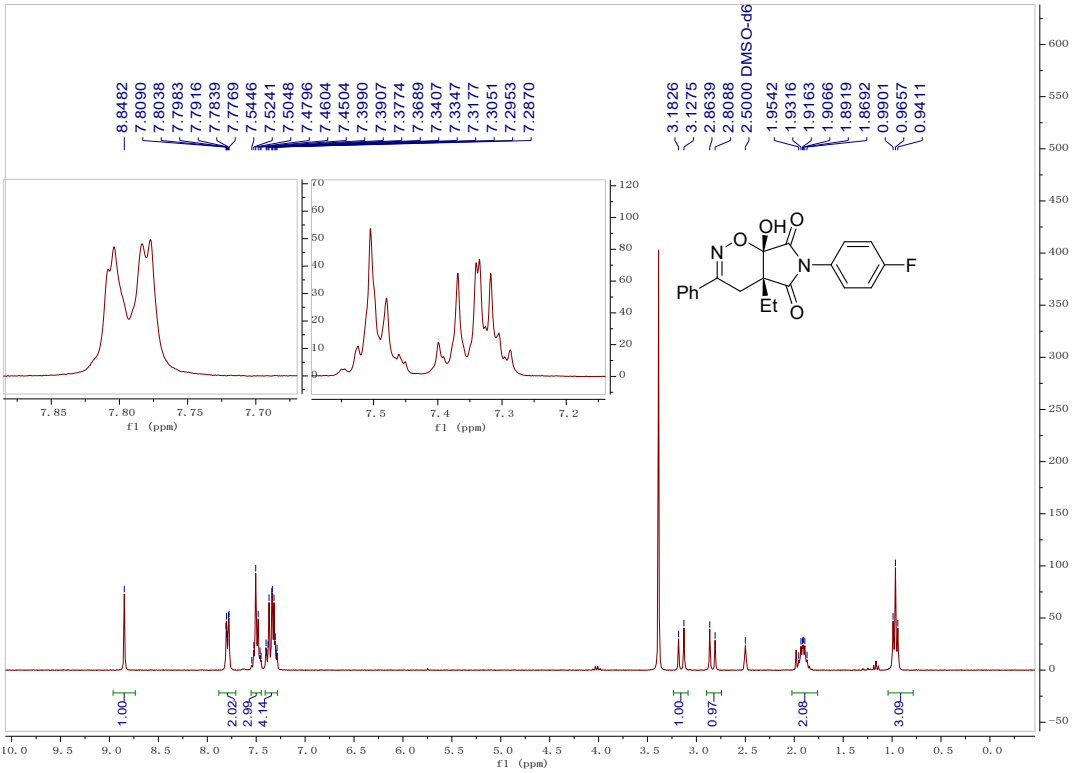


^{13}C NMR (75 MHz, DMSO-d_6)

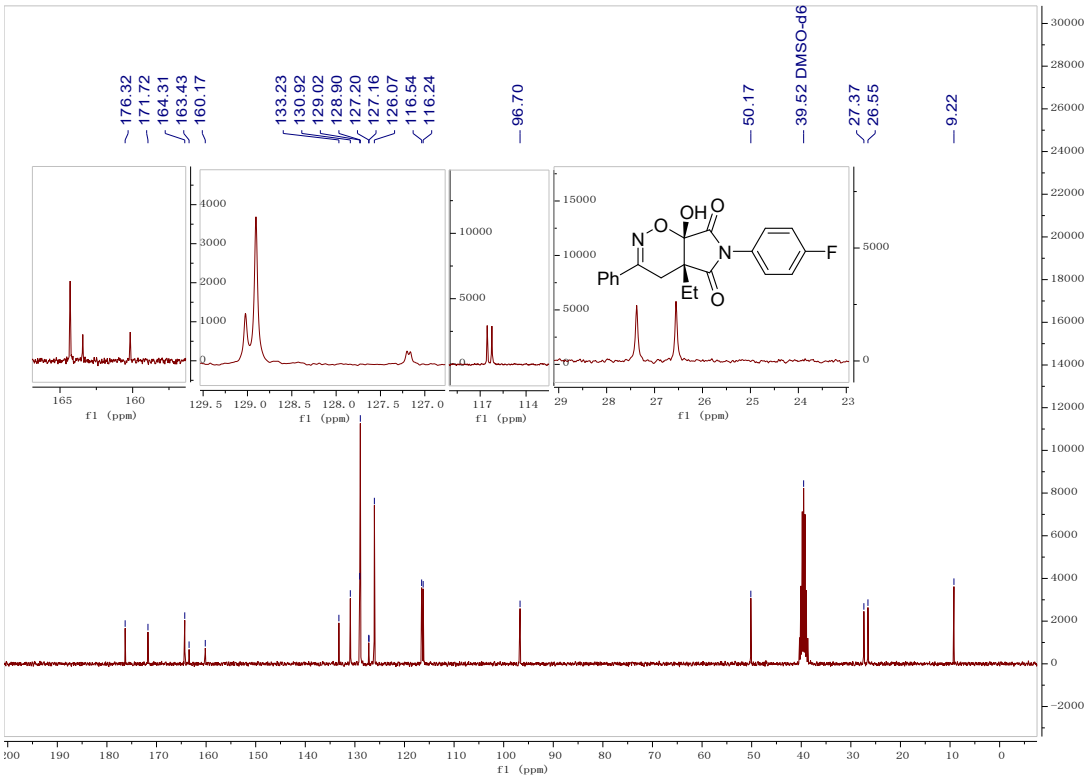


3ja

^{1}H NMR (300 MHz, DMSO-d_6)

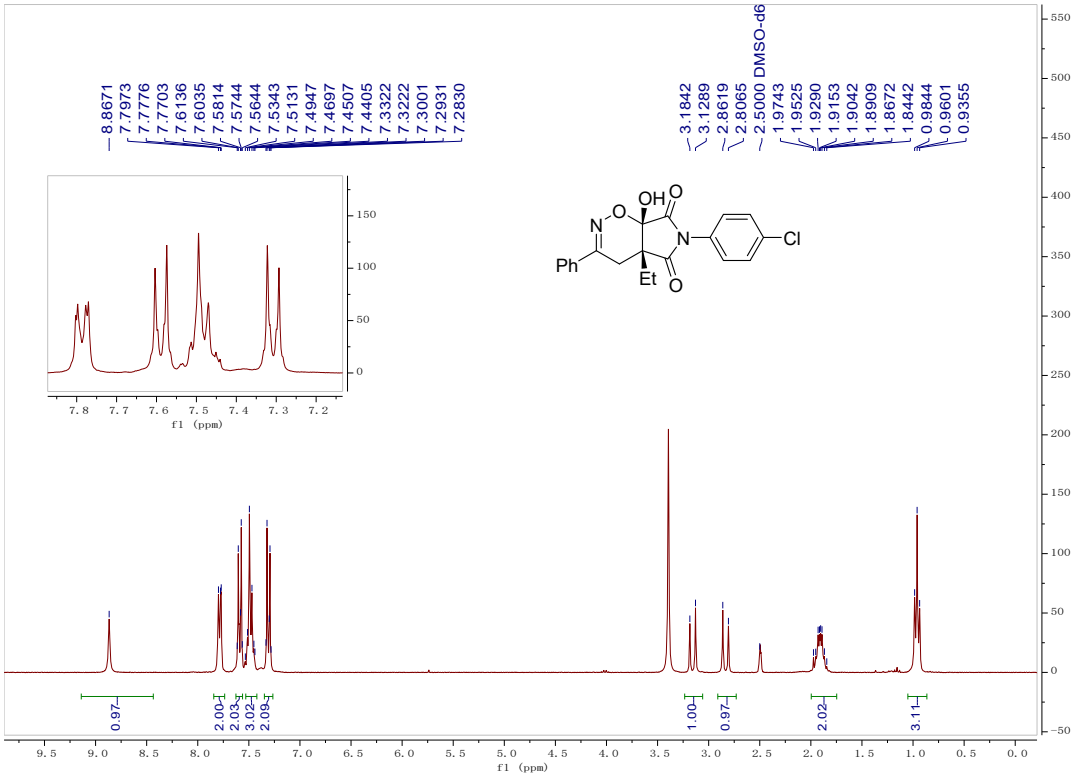


^{13}C NMR (75 MHz, DMSO-d_6)

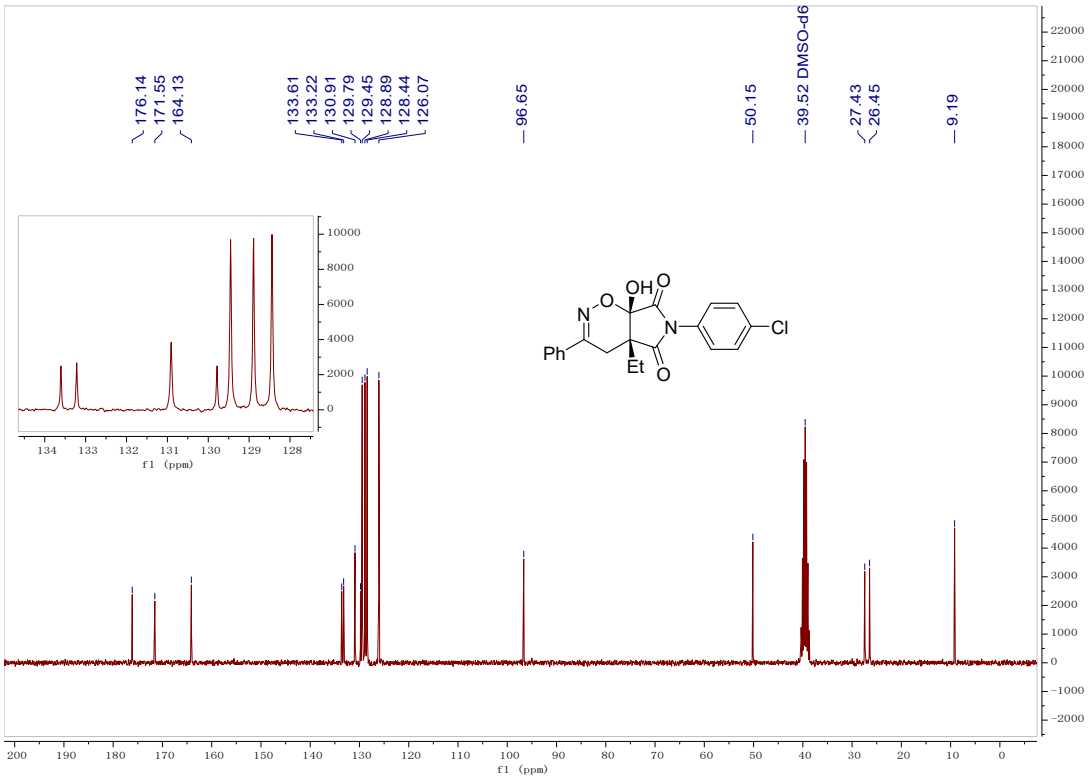


3ka

^{1}H NMR (300 MHz, DMSO-d_6)

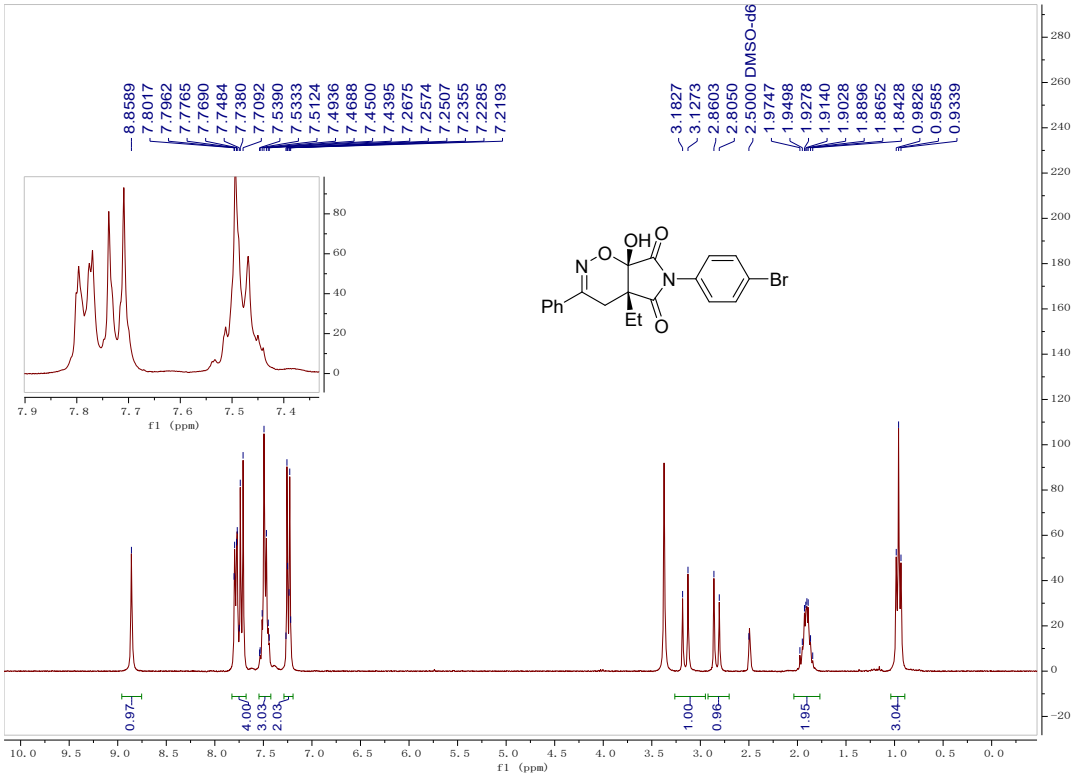


^{13}C NMR (75 MHz, DMSO-d_6)

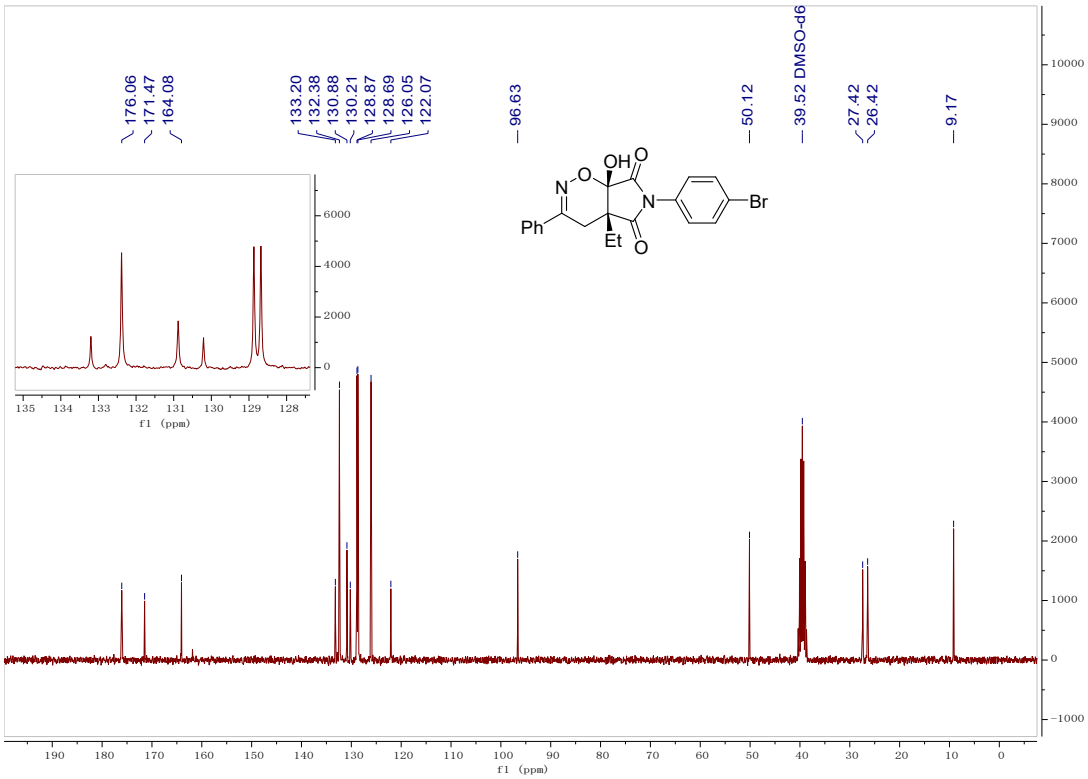


3la

^{1}H NMR (300 MHz, DMSO-d_6)

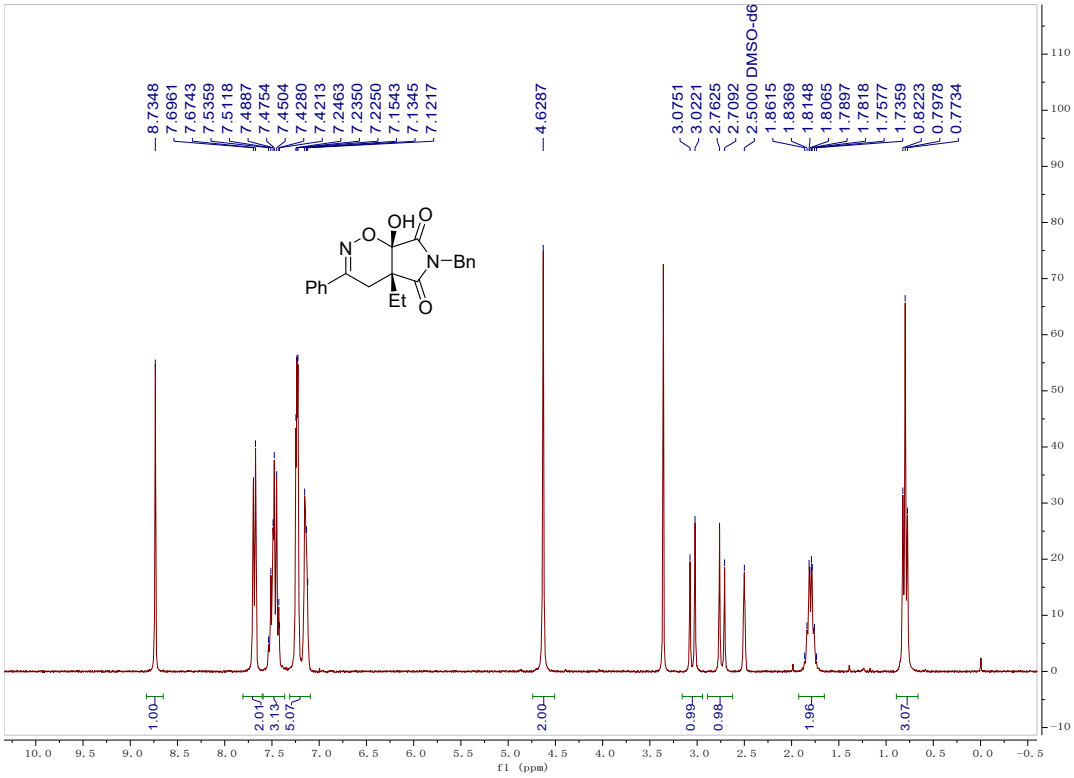


^{13}C NMR (75 MHz, DMSO-d_6)

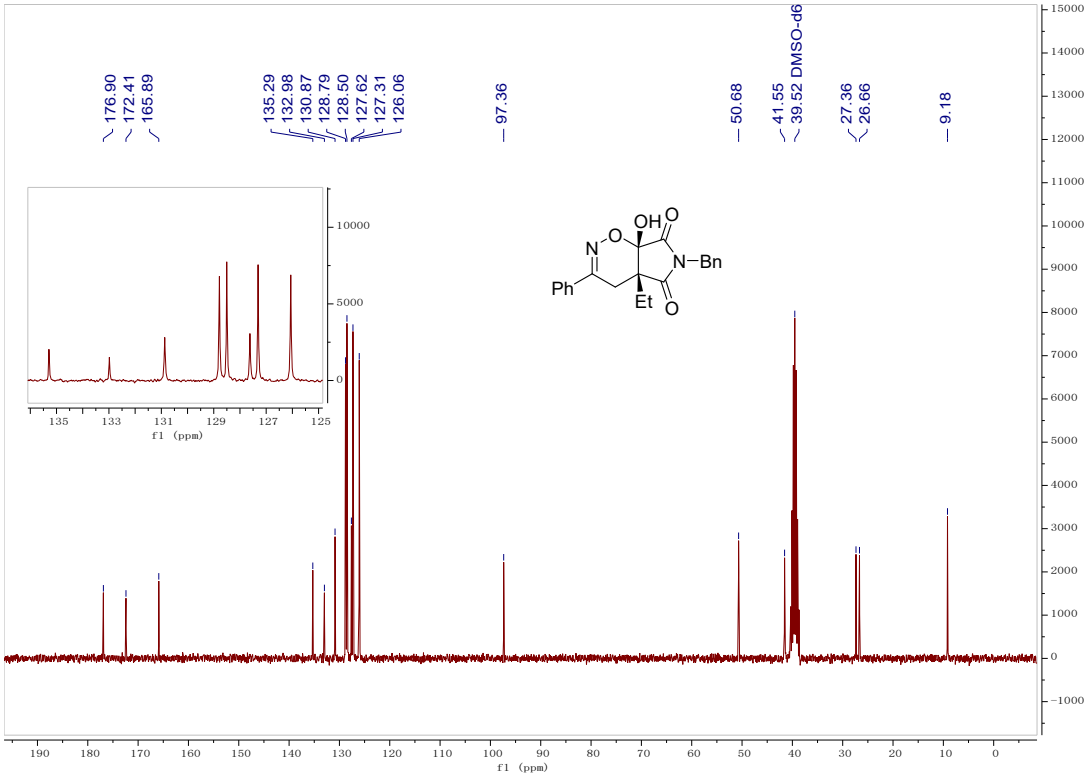


3ma

^{1}H NMR (300 MHz, DMSO-d_6)

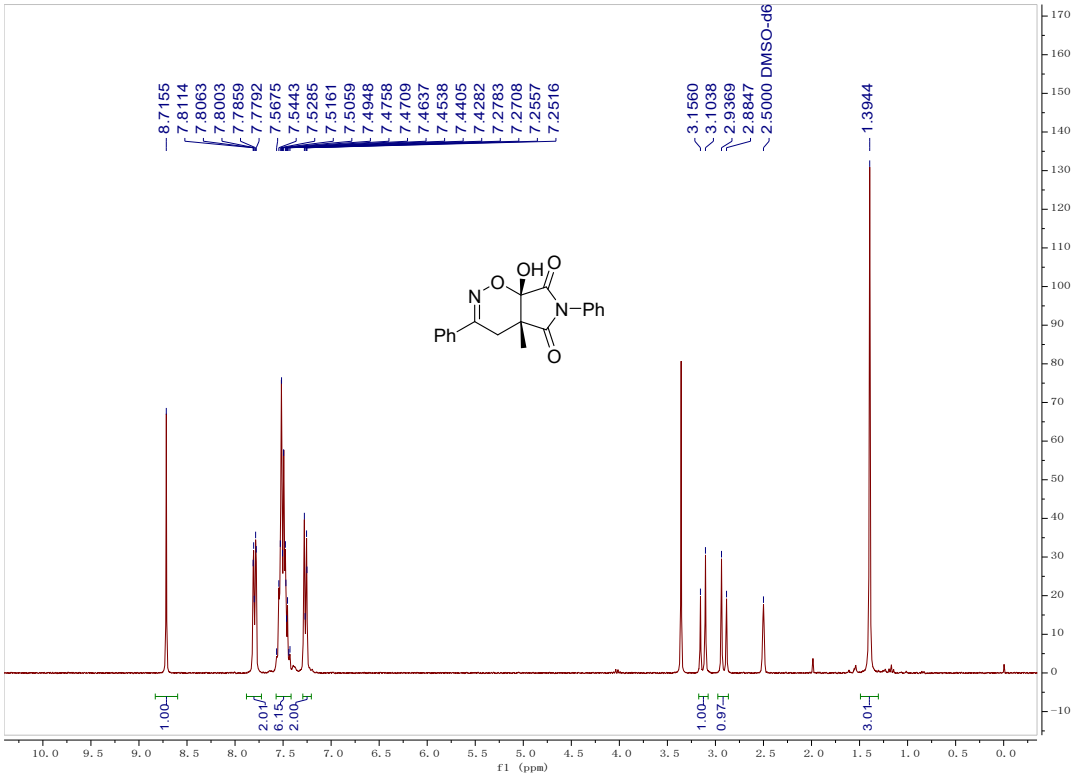


^{13}C NMR (75 MHz, DMSO-d_6)

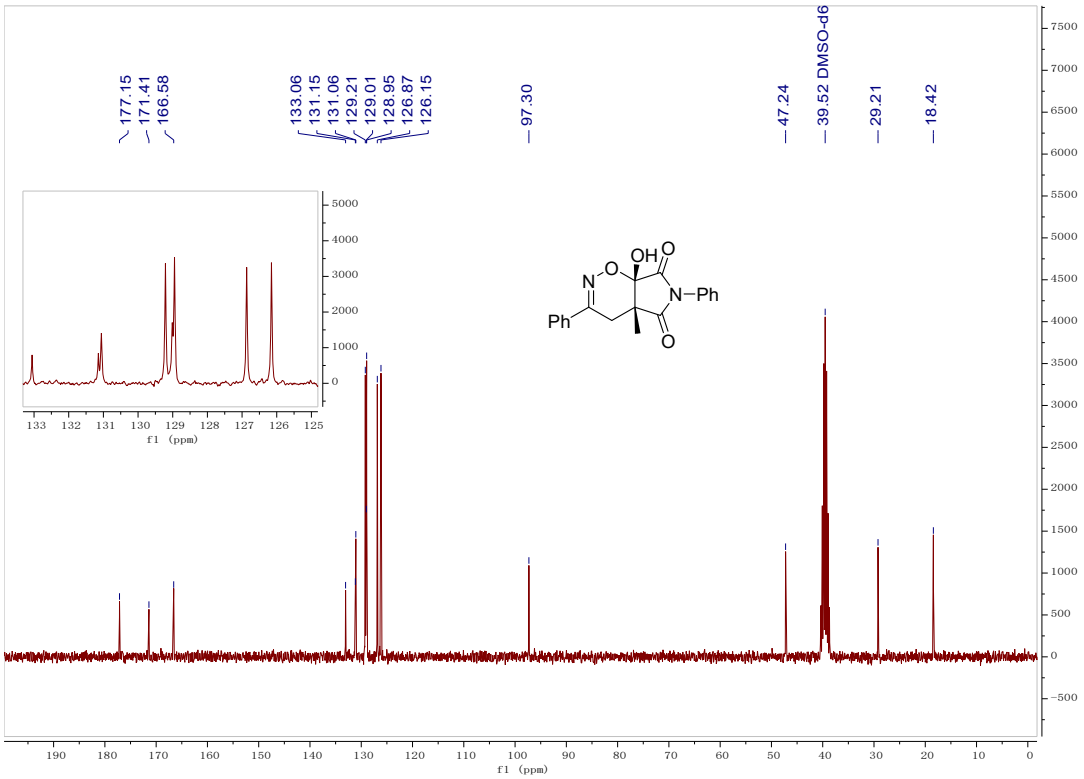


3na

[1]H NMR (300 MHz, DMSO-d_6)

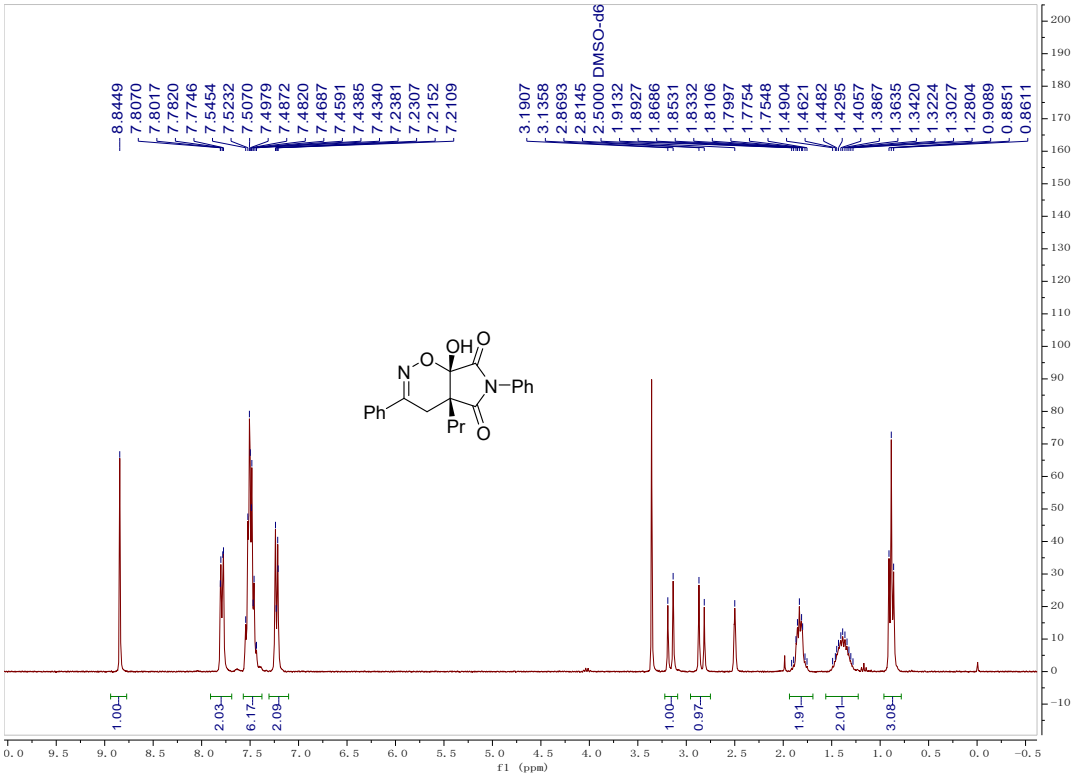


[13]C NMR (75 MHz, DMSO-d_6)

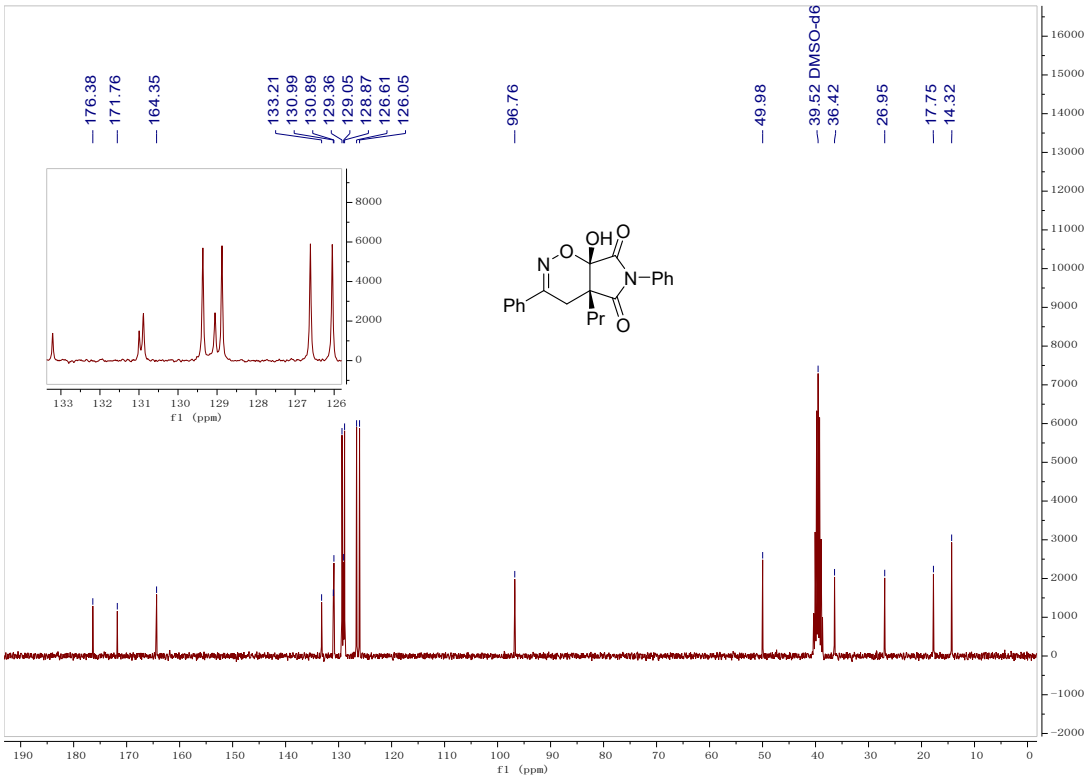


3oa

^{1}H NMR (300 MHz, DMSO-d_6)

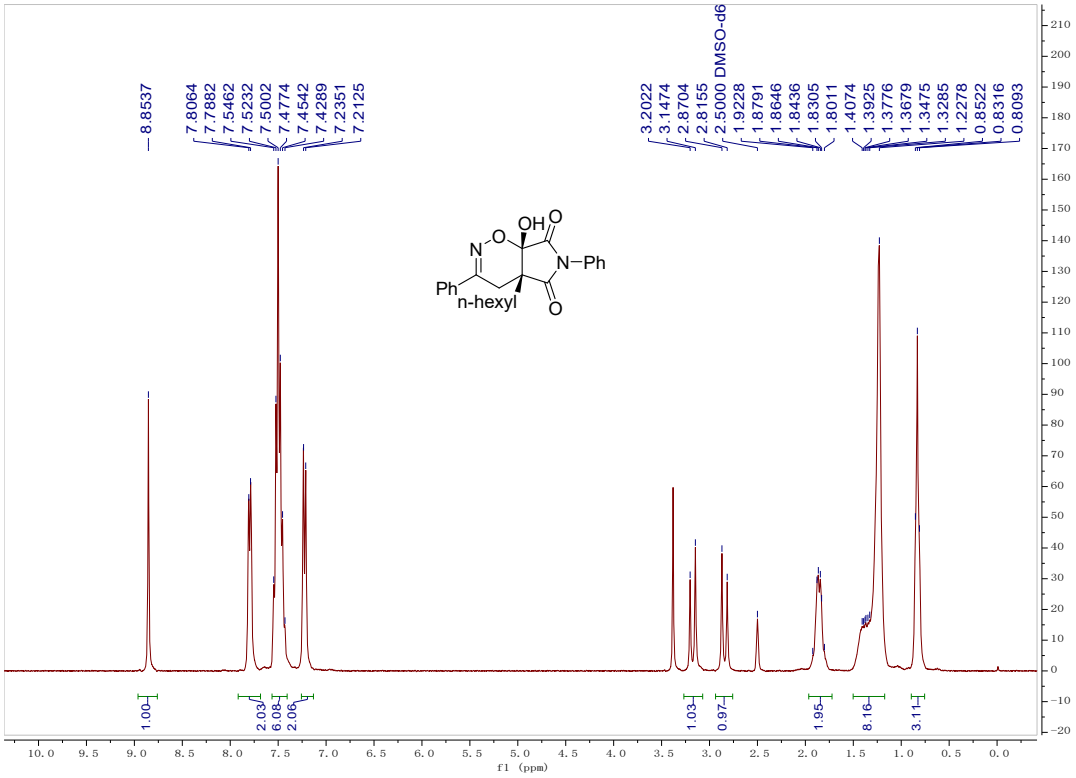


^{13}C NMR (75 MHz, DMSO-d_6)

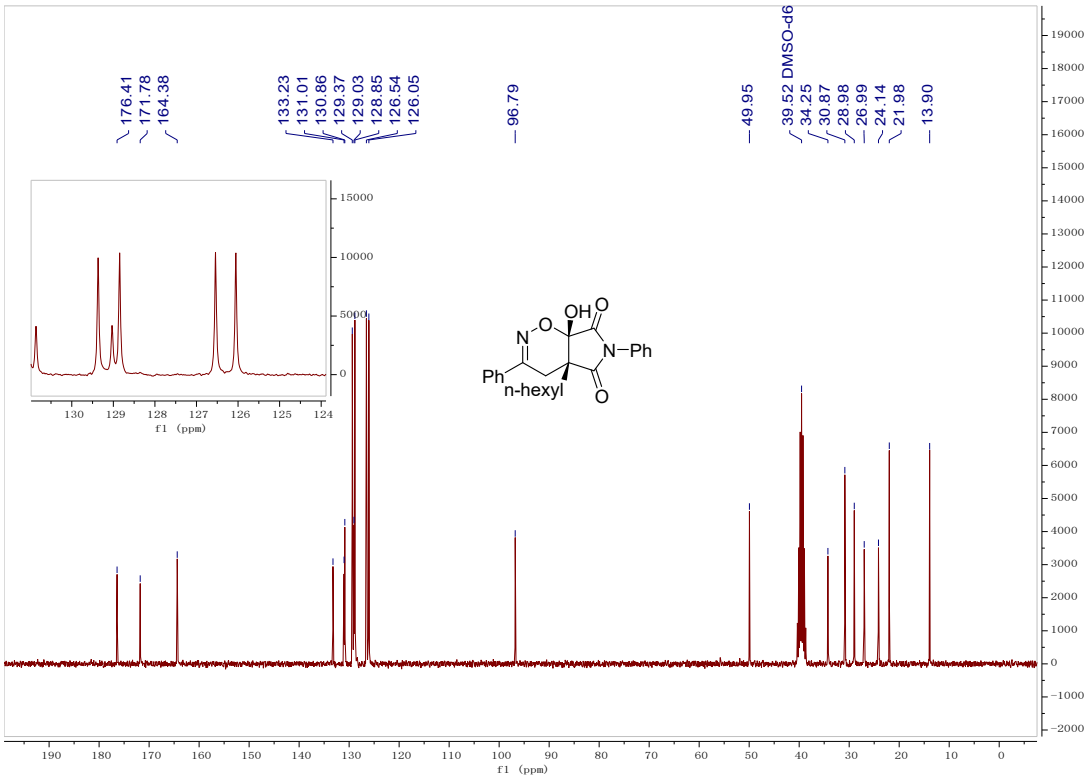


3pa

^{1}H NMR (300 MHz, DMSO-d_6)

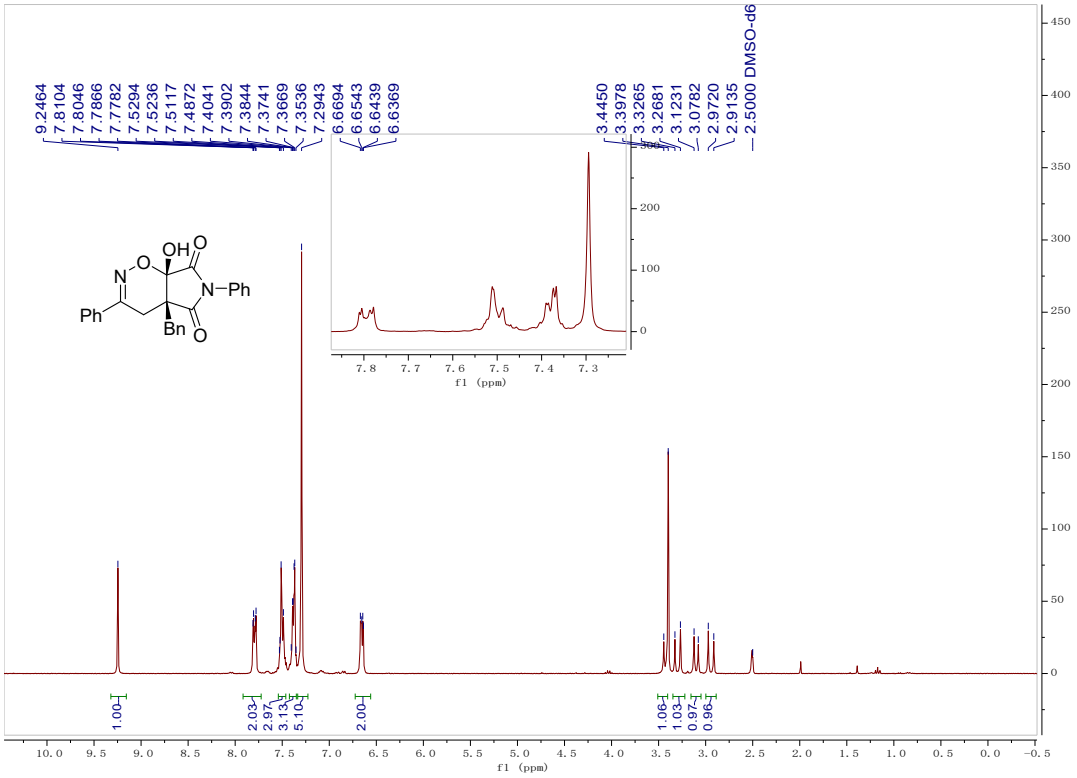


^{13}C NMR (75 MHz, DMSO-d_6)

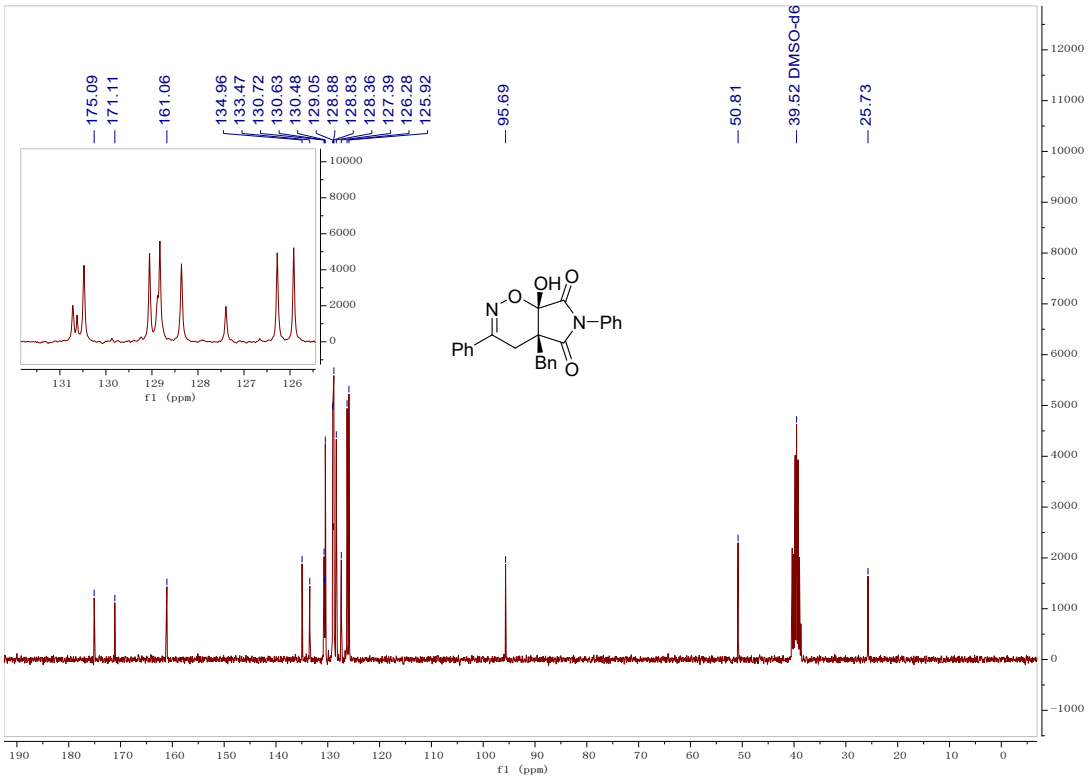


3qa

^{1}H NMR (300 MHz, DMSO-d_6)

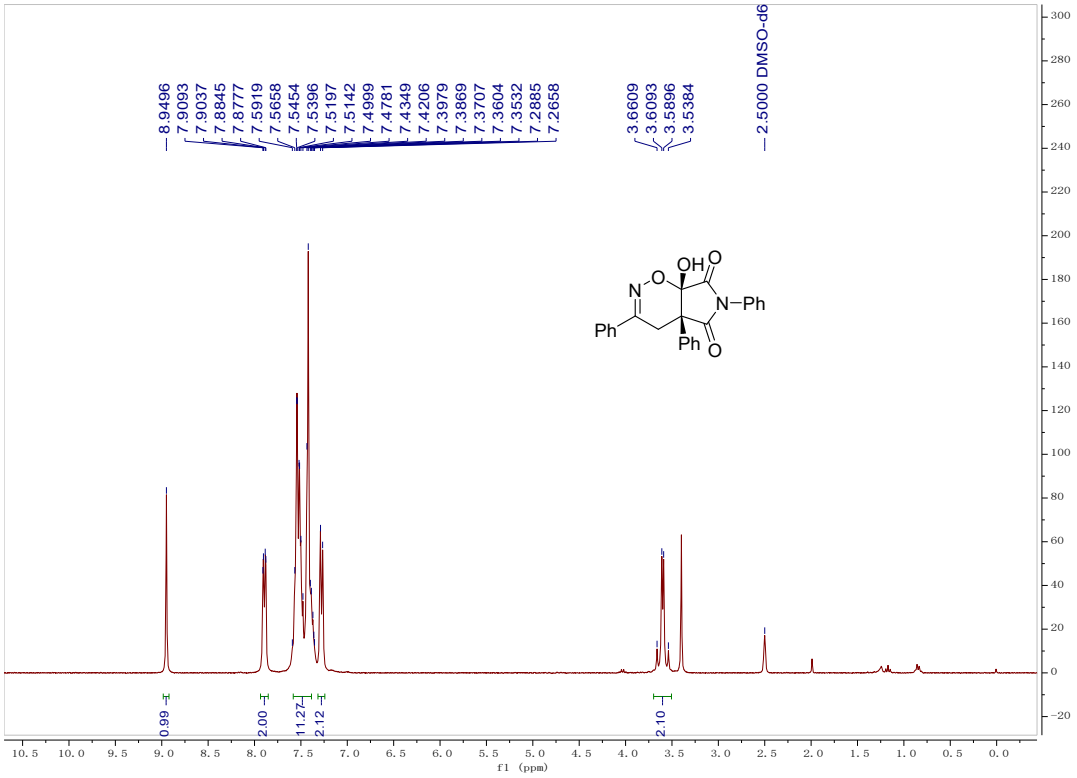


^{13}C NMR (75 MHz, DMSO-d_6)

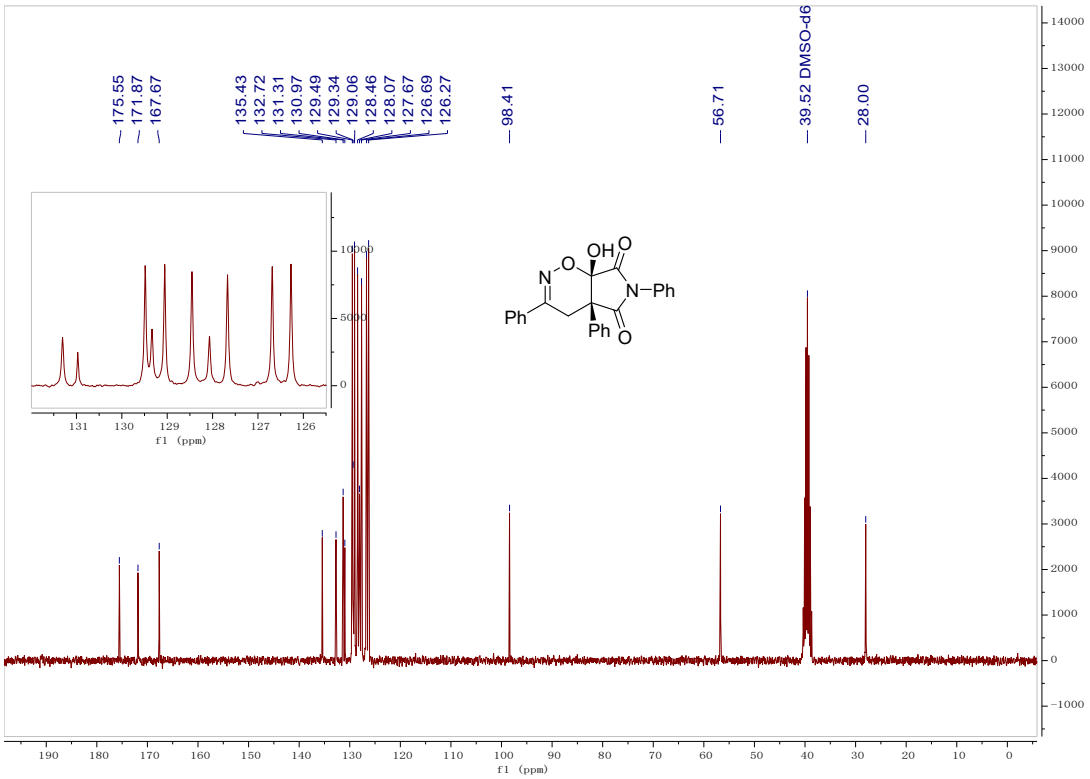


3ra

^{1}H NMR (300 MHz, DMSO-d_6)

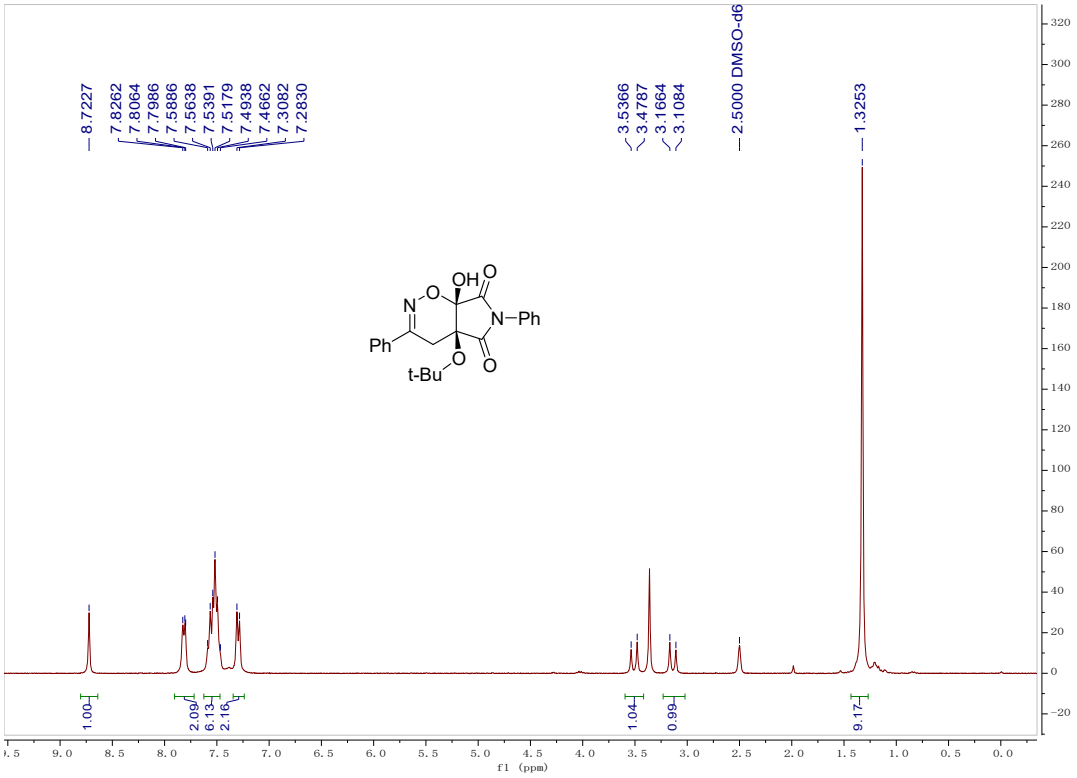


^{13}C NMR (75 MHz, DMSO-d_6)

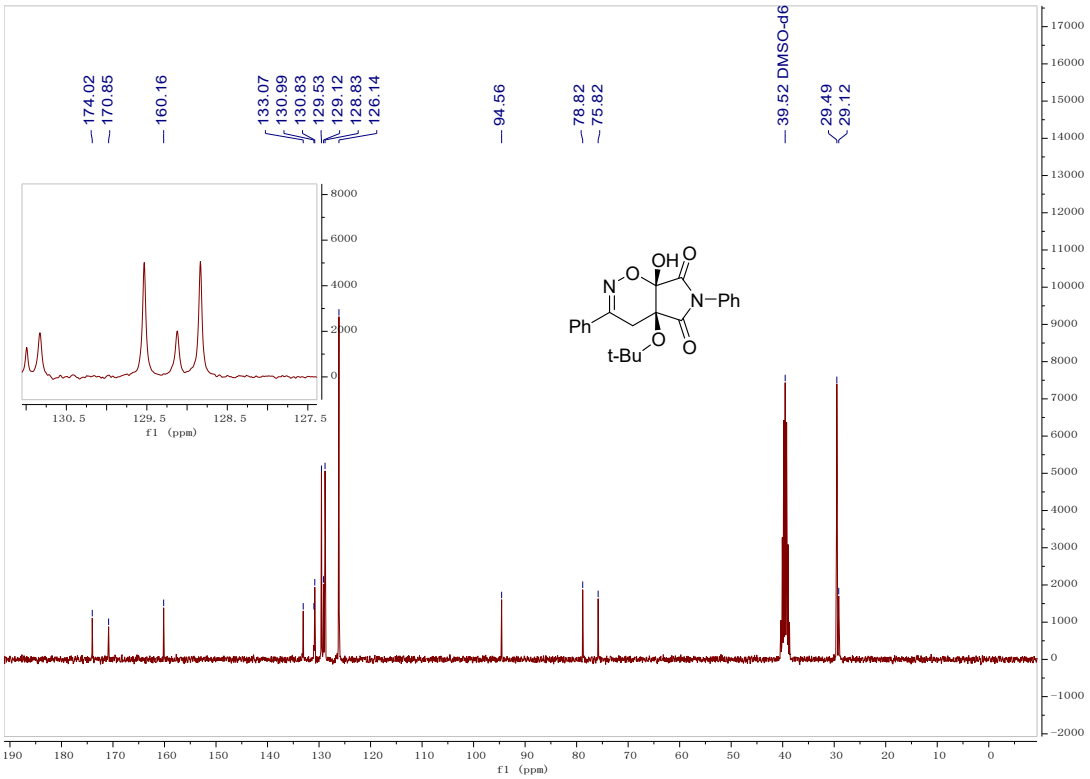


3sa

^{1}H NMR (300 MHz, DMSO-d_6)

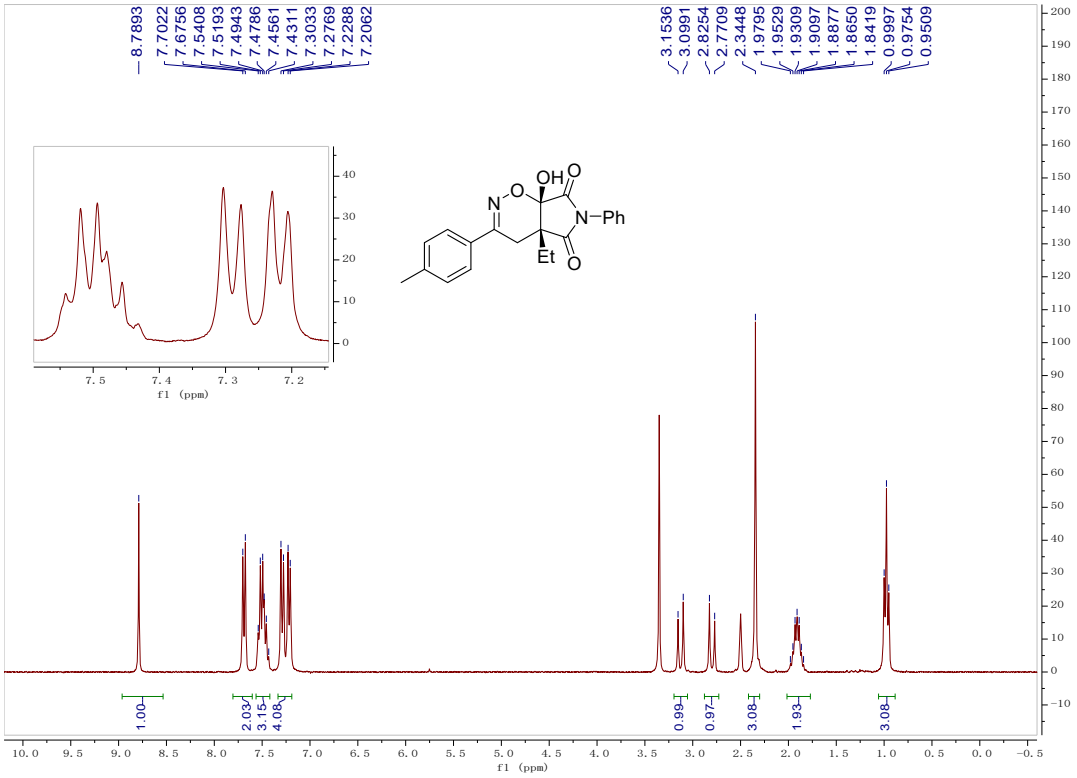


^{13}C NMR (75 MHz, DMSO-d_6)

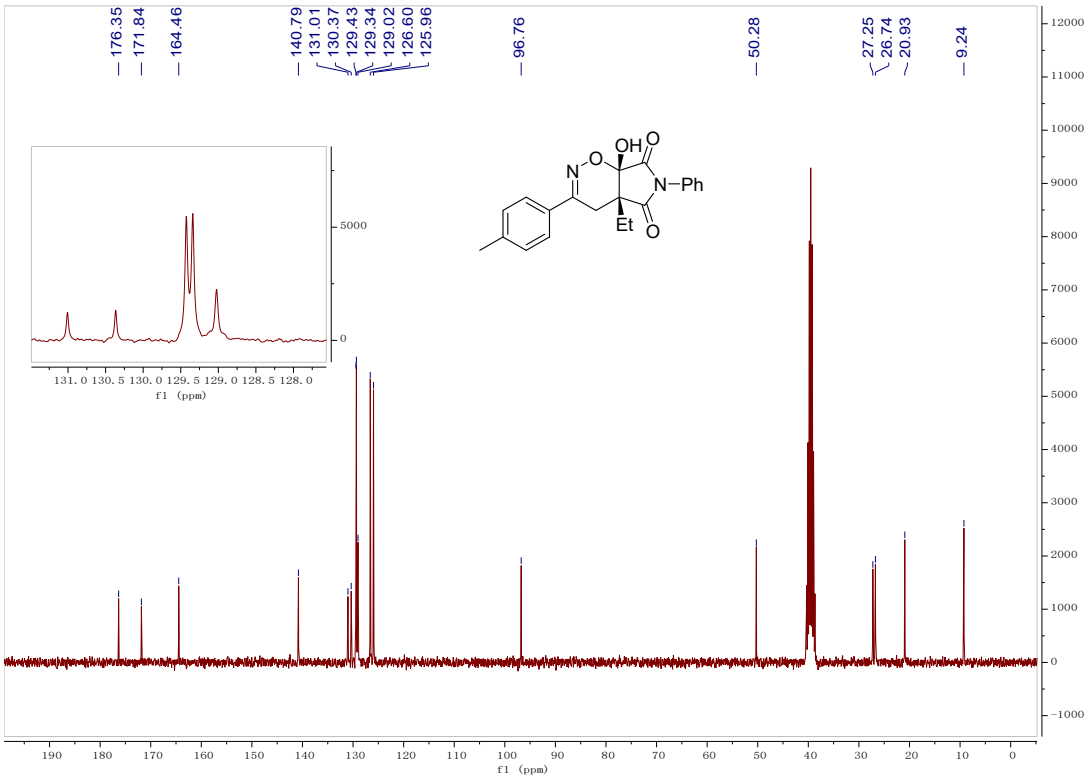


3ab

^{1}H NMR (300 MHz, DMSO-d_6)

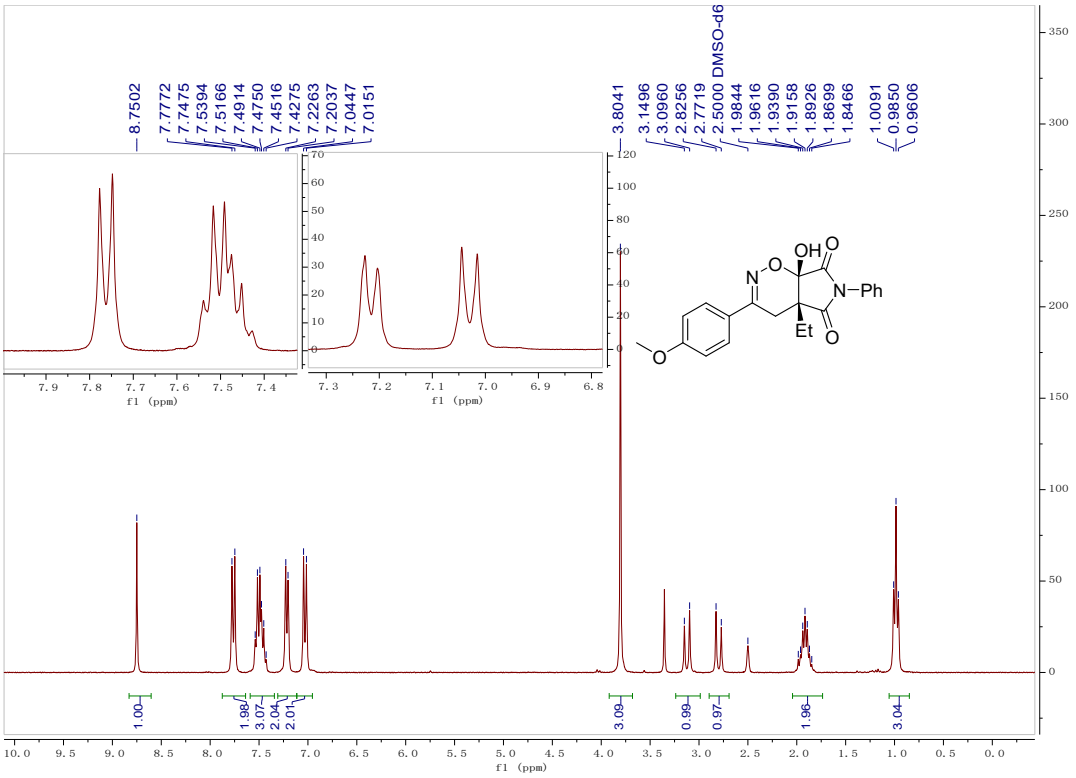


^{13}C NMR (75 MHz, DMSO-d_6)

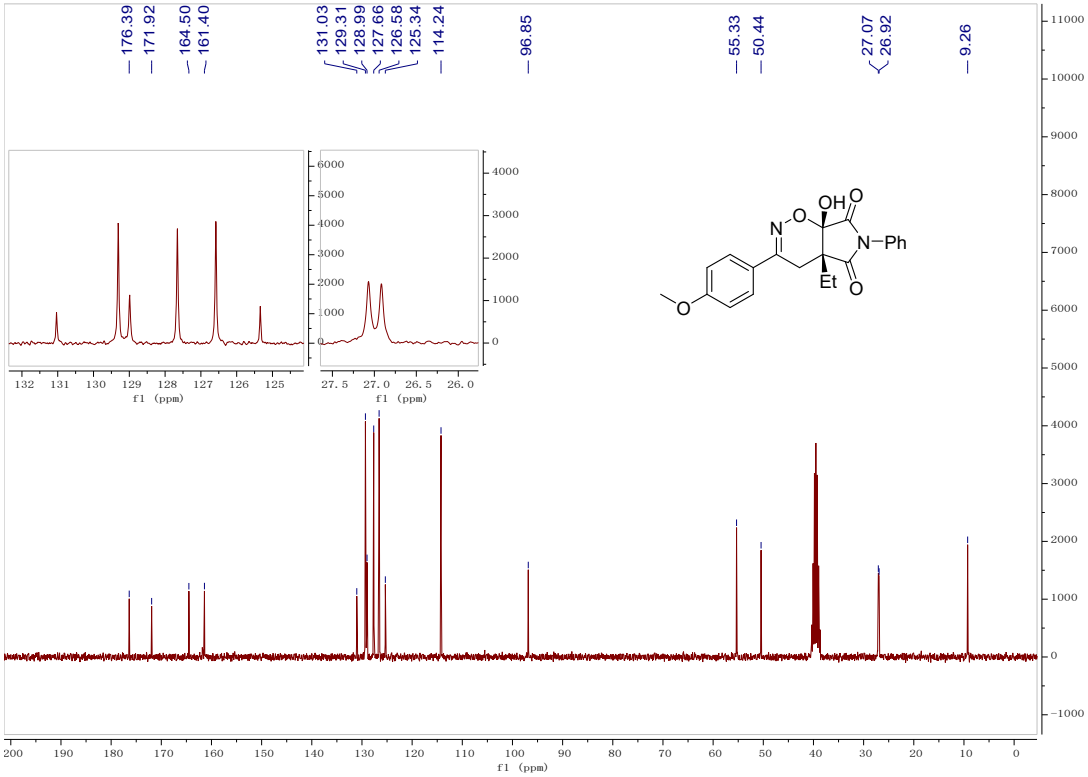


3ac

^{1}H NMR (300 MHz, DMSO-d_6)

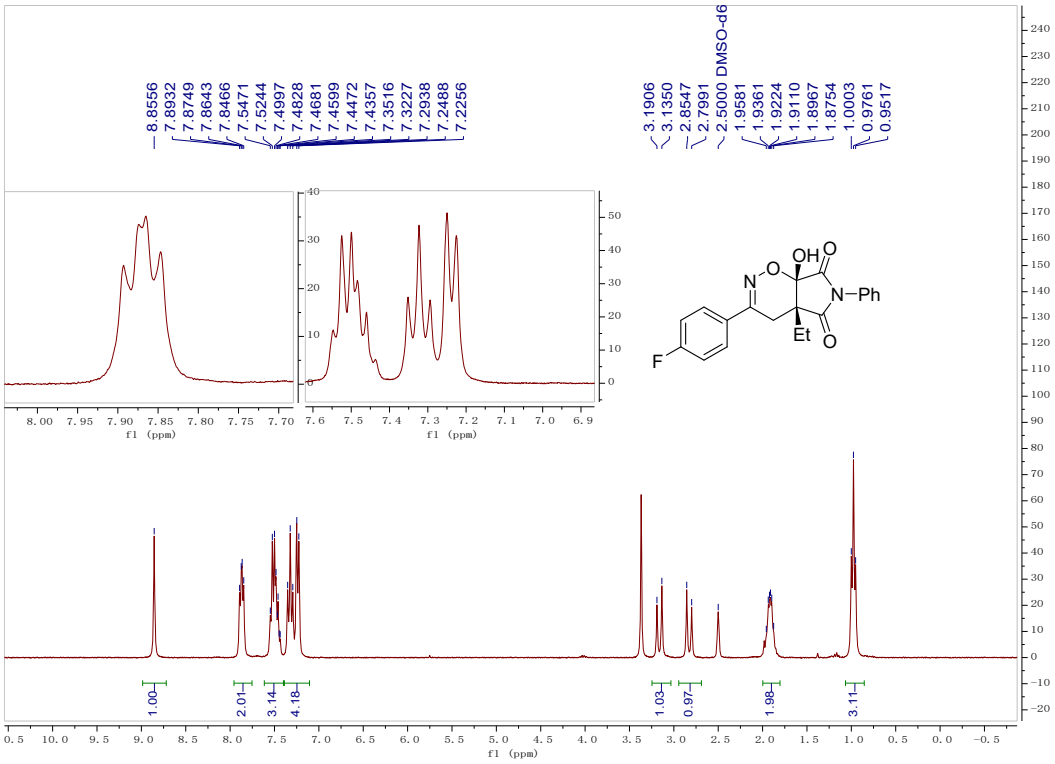


^{13}C NMR (75 MHz, DMSO-d_6)

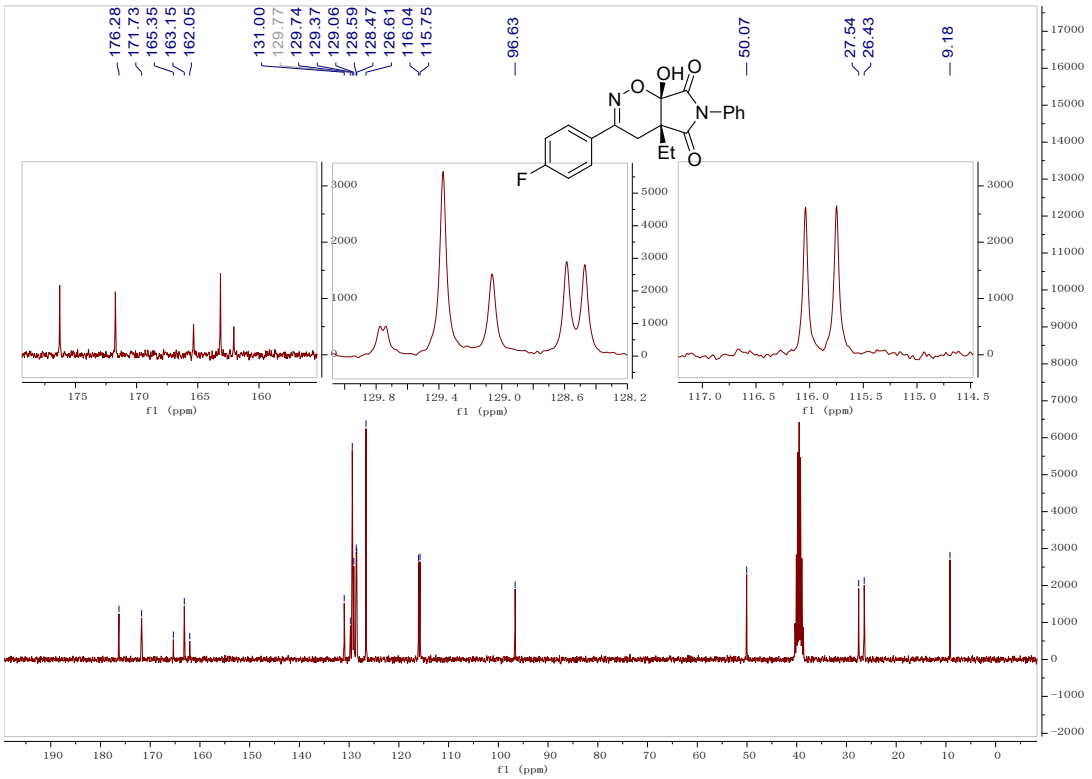


3ad

^{1}H NMR (300 MHz, DMSO-d_6)

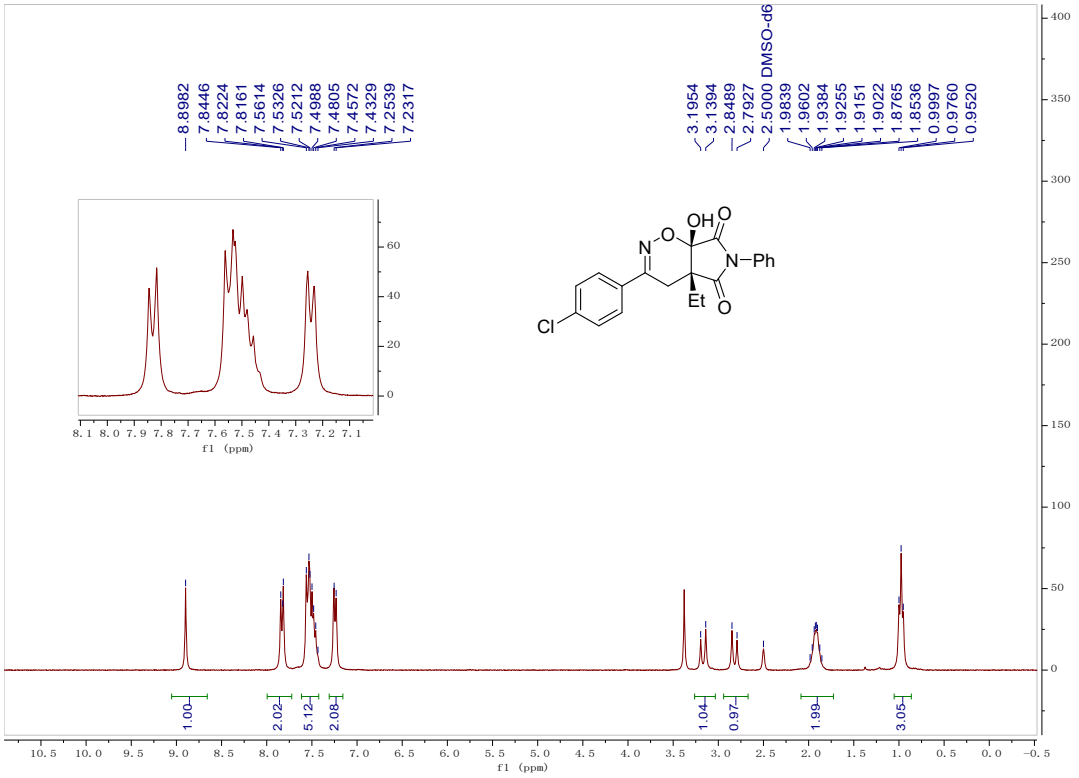


^{13}C NMR (75 MHz, DMSO-d_6)

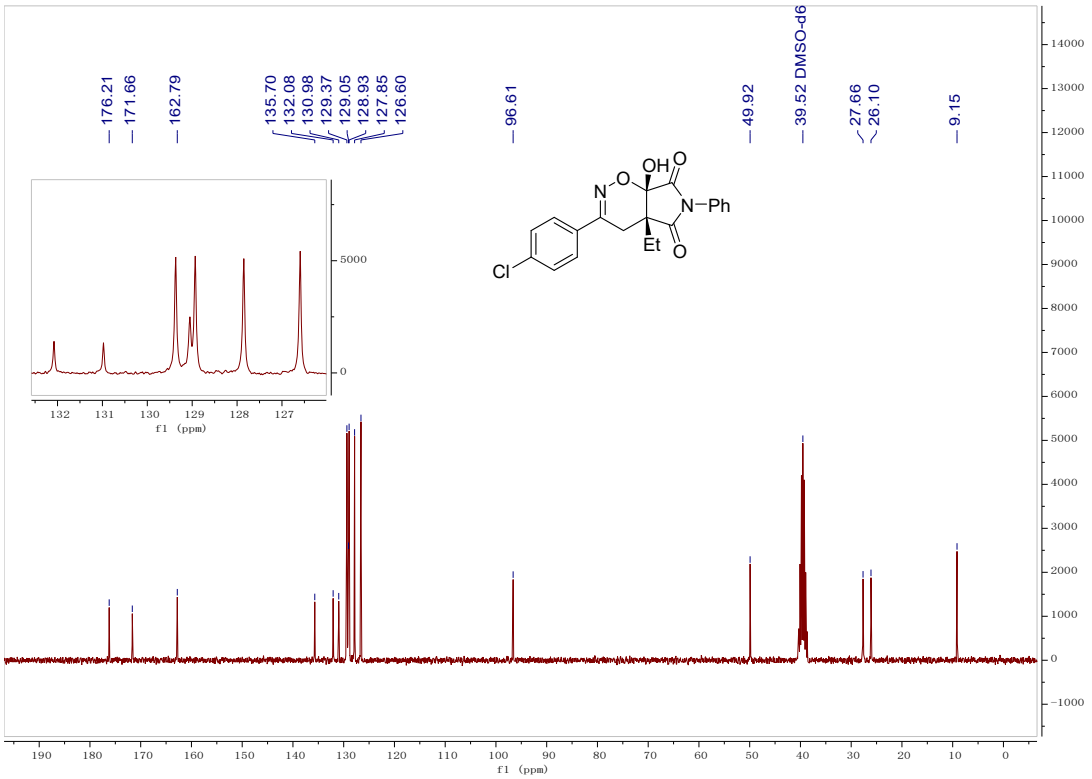


3ae

^{1}H NMR (300 MHz, DMSO-d_6)

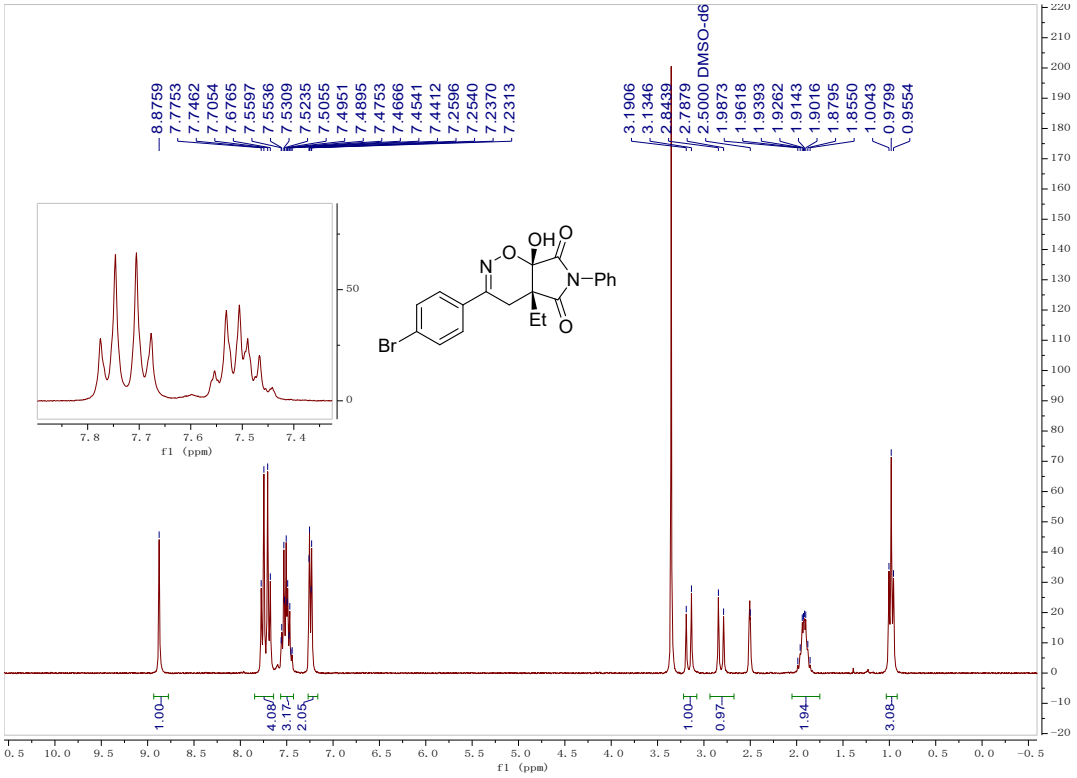


^{13}C NMR (75 MHz, DMSO-d_6)

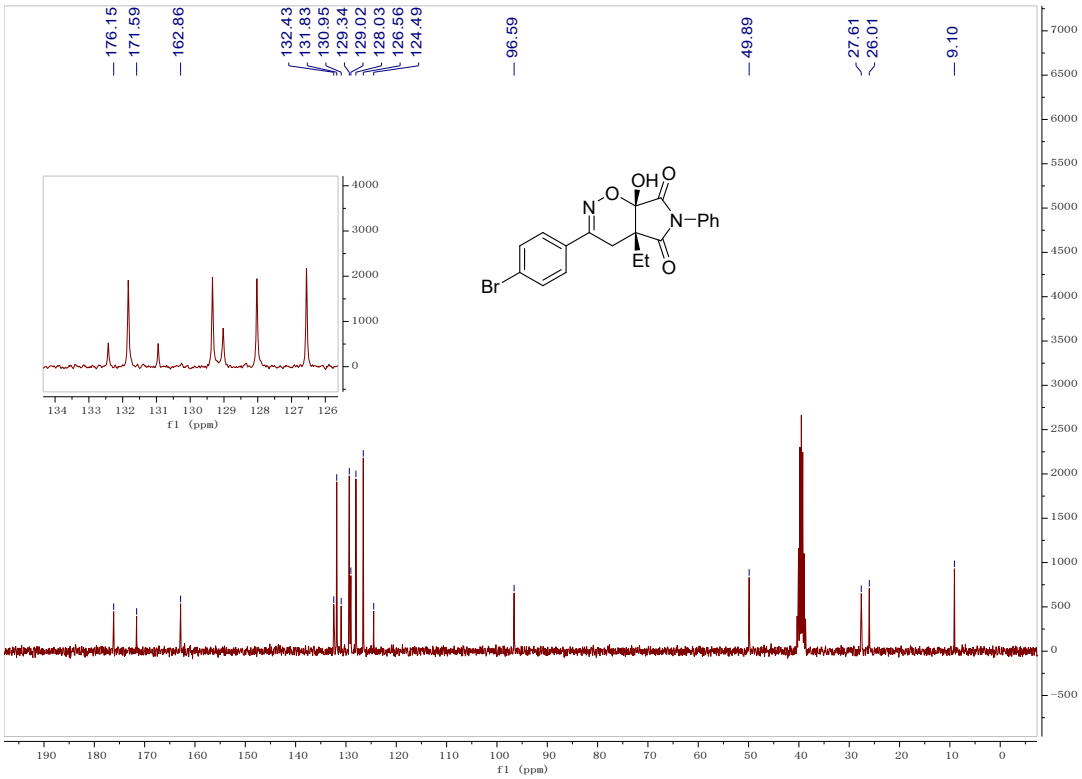


3af

^{1}H NMR (300 MHz, DMSO-d_6)

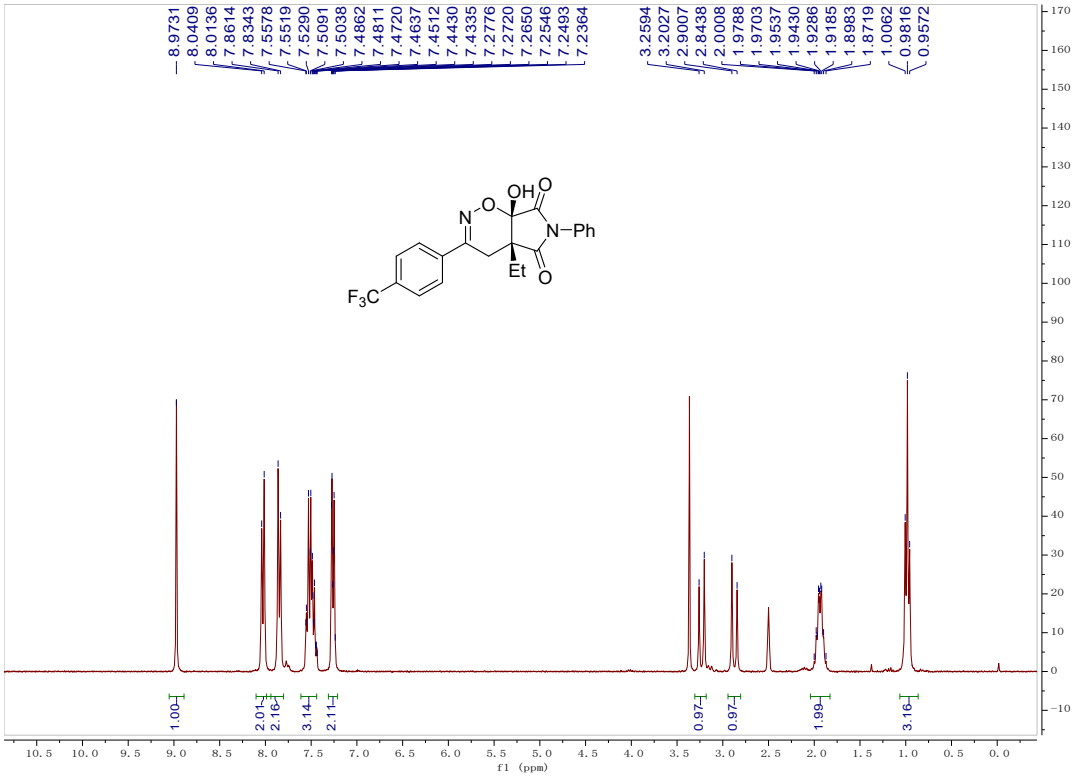


^{13}C NMR (75 MHz, DMSO-d_6)

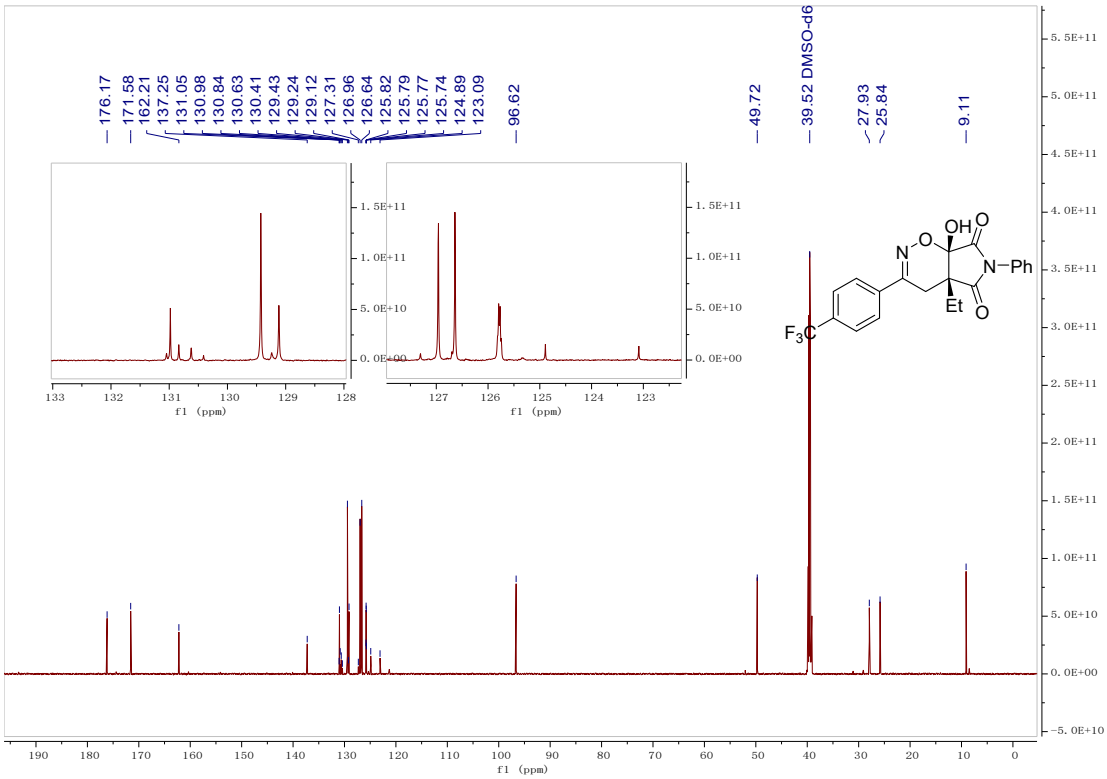


3ag

^{1}H NMR (300 MHz, DMSO-d_6)

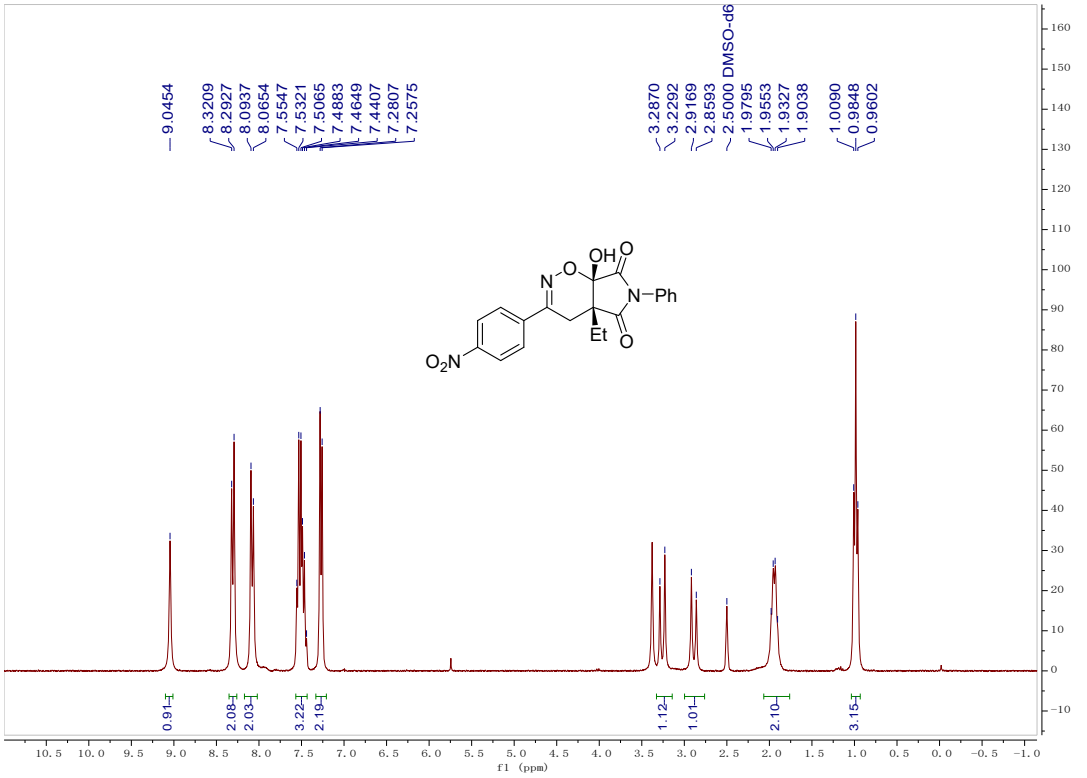


^{13}C NMR (75 MHz, DMSO-d_6)

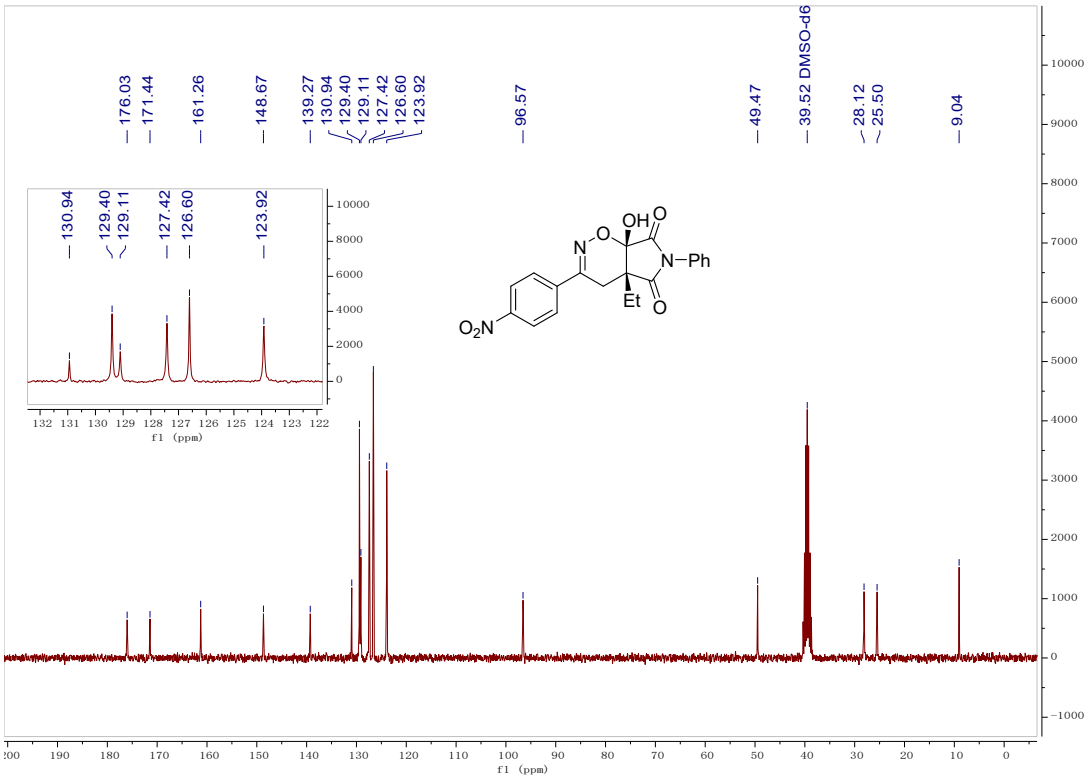


3ah

^{1}H NMR (300 MHz, DMSO-d_6)

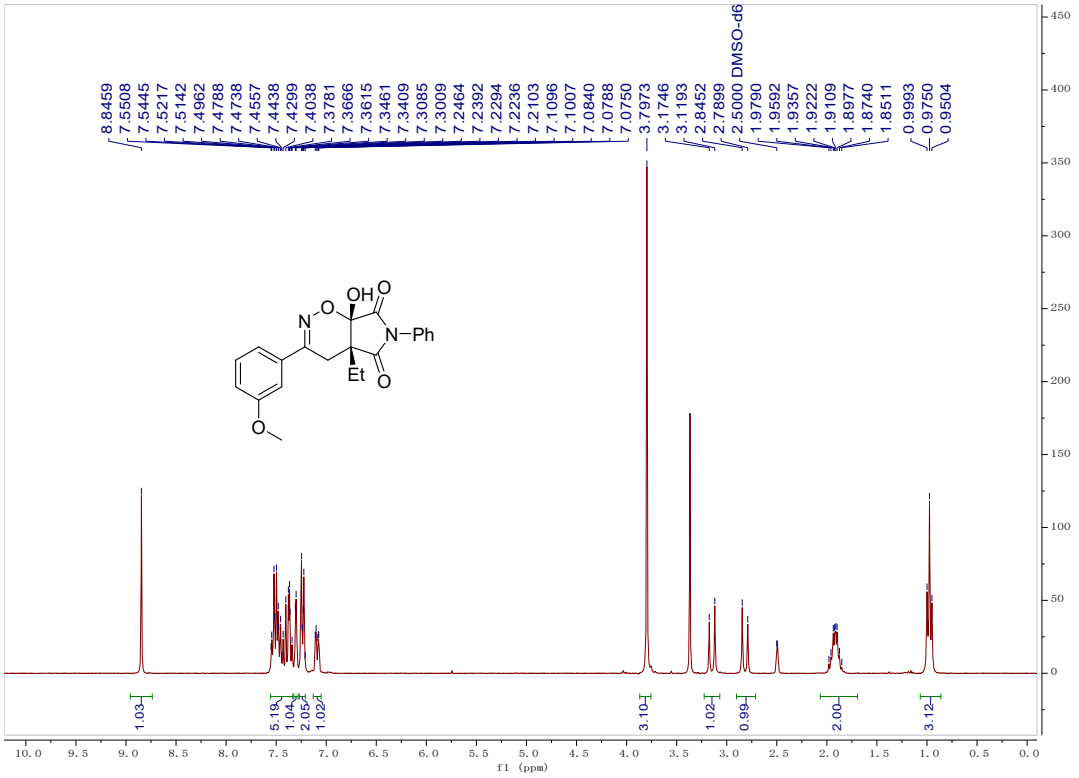


^{13}C NMR (75 MHz, DMSO-d_6)

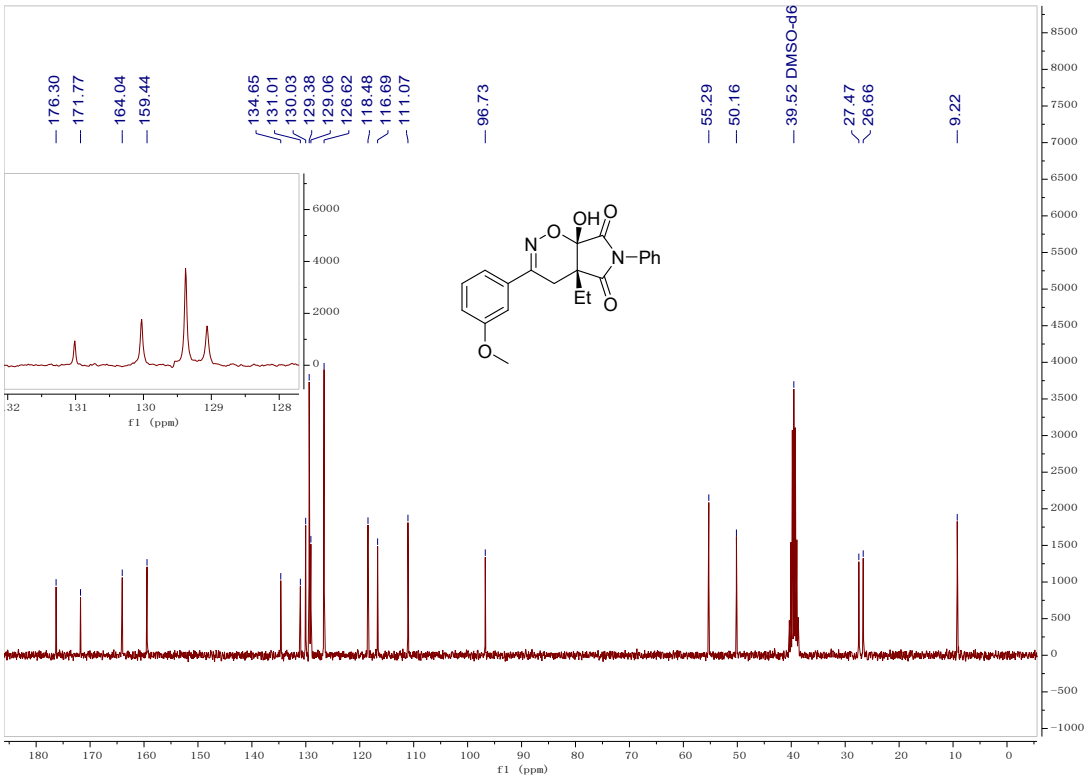


3ai

^{1}H NMR (300 MHz, DMSO-d_6)

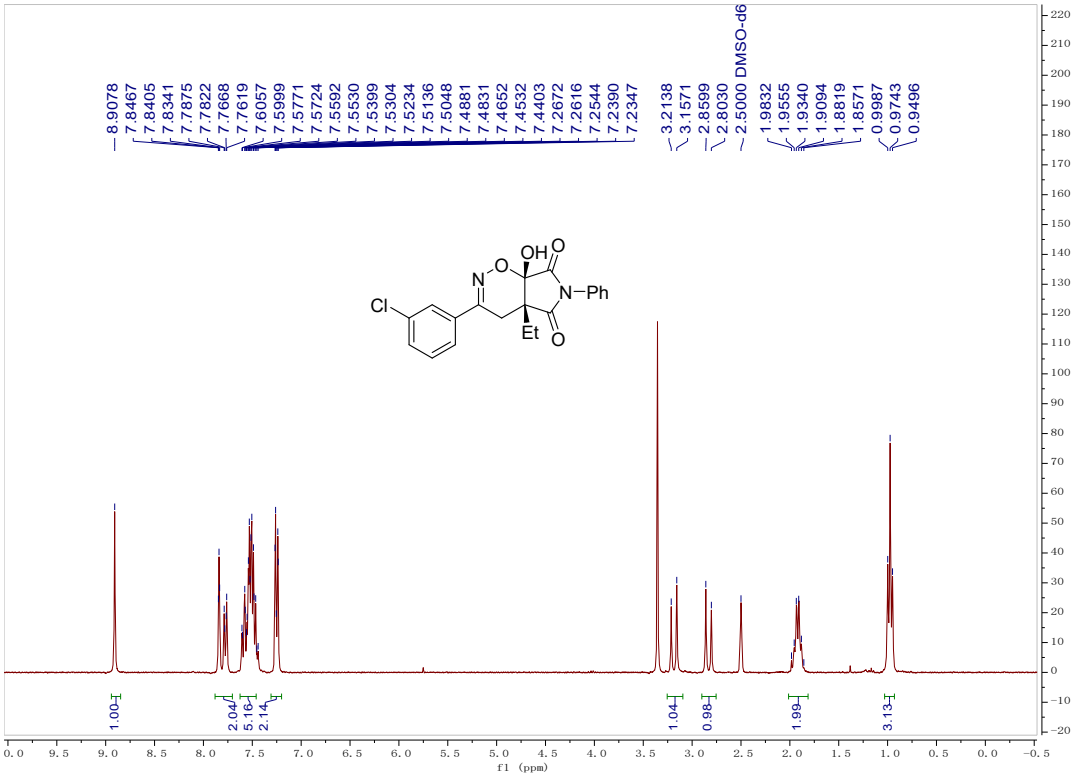


^{13}C NMR (75 MHz, DMSO-d_6)

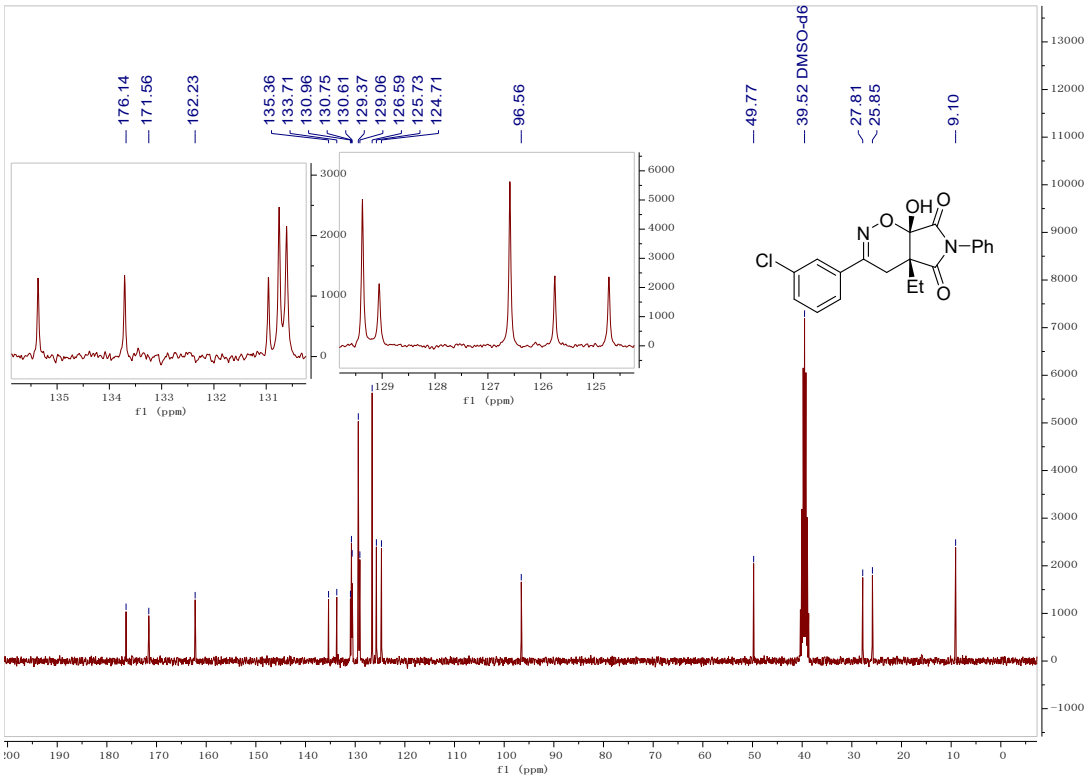


3aj

^{1}H NMR (300 MHz, DMSO-d_6)

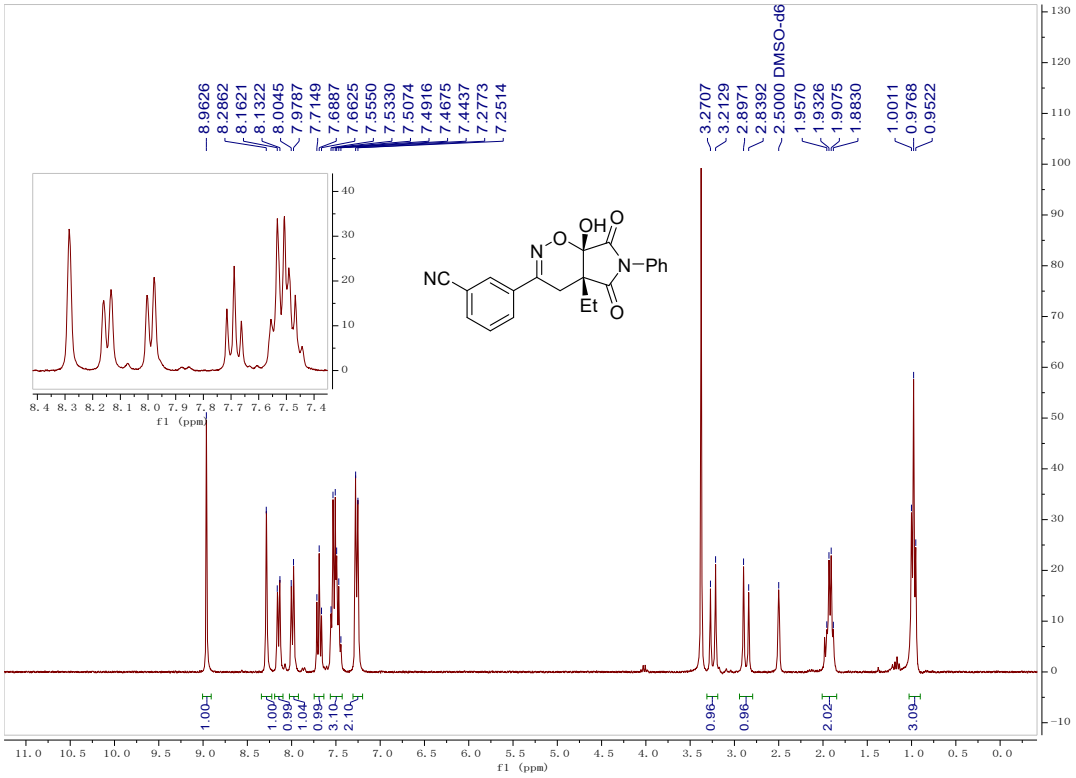


^{13}C NMR (75 MHz, DMSO-d_6)

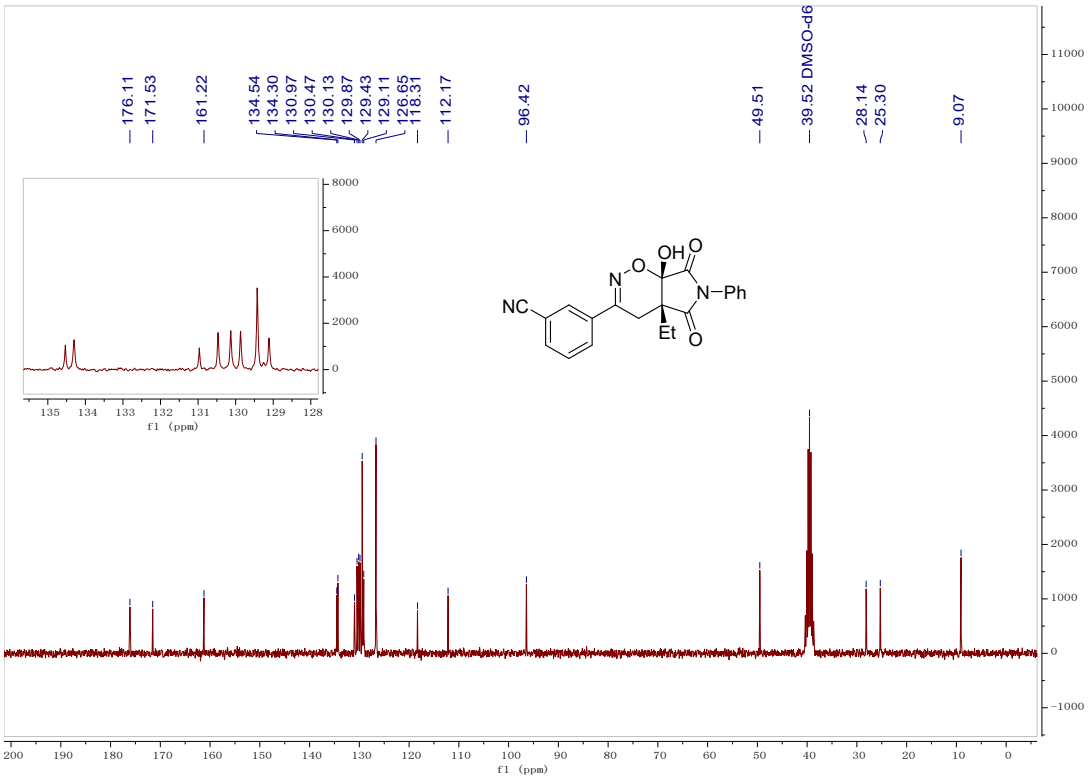


3ak

^{1}H NMR (300 MHz, DMSO-d_6)

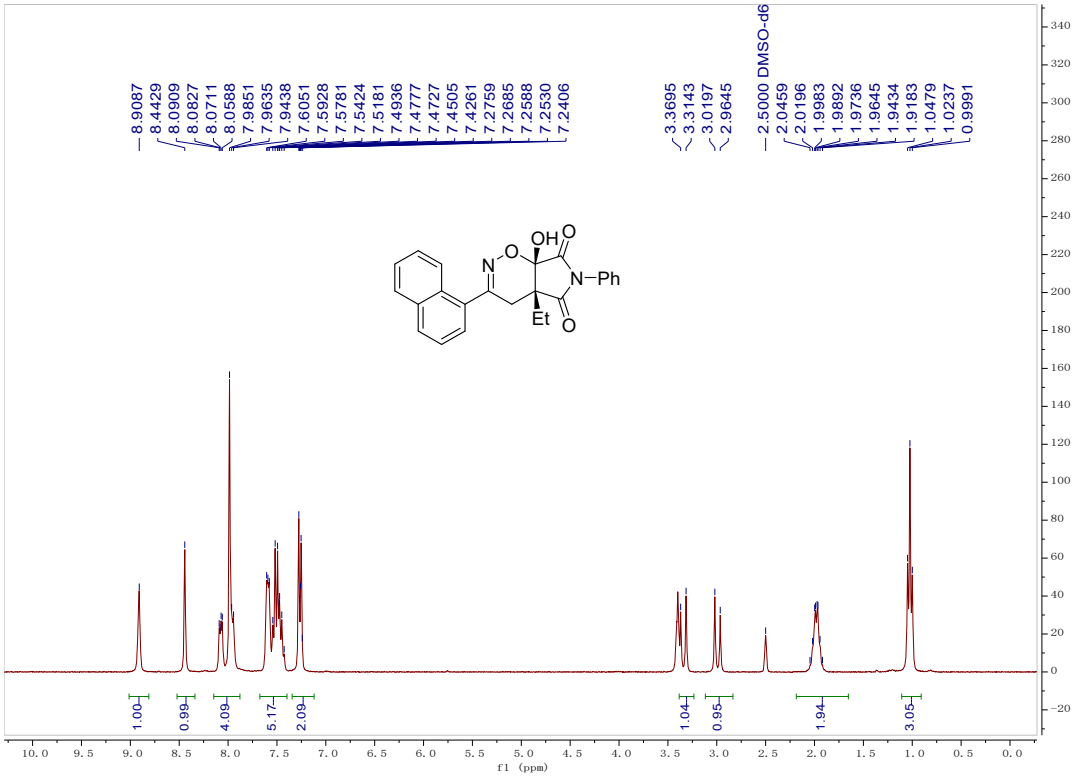


^{13}C NMR (75 MHz, DMSO-d_6)

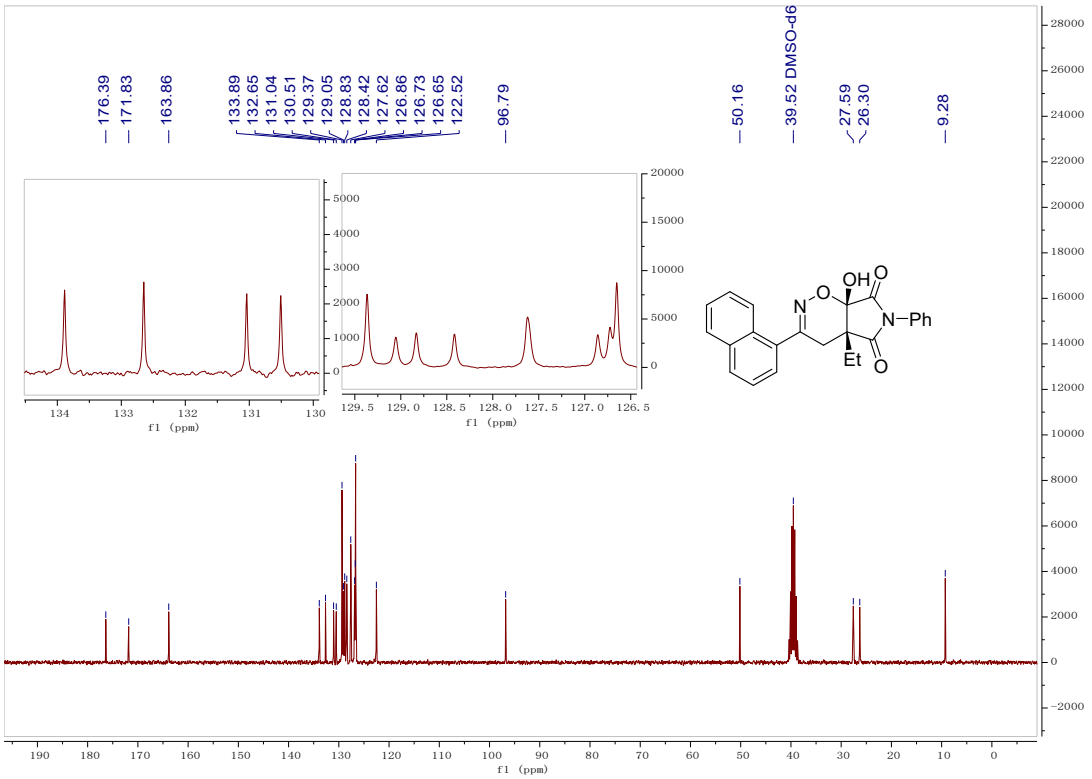


3al

^{1}H NMR (300 MHz, DMSO-d_6)

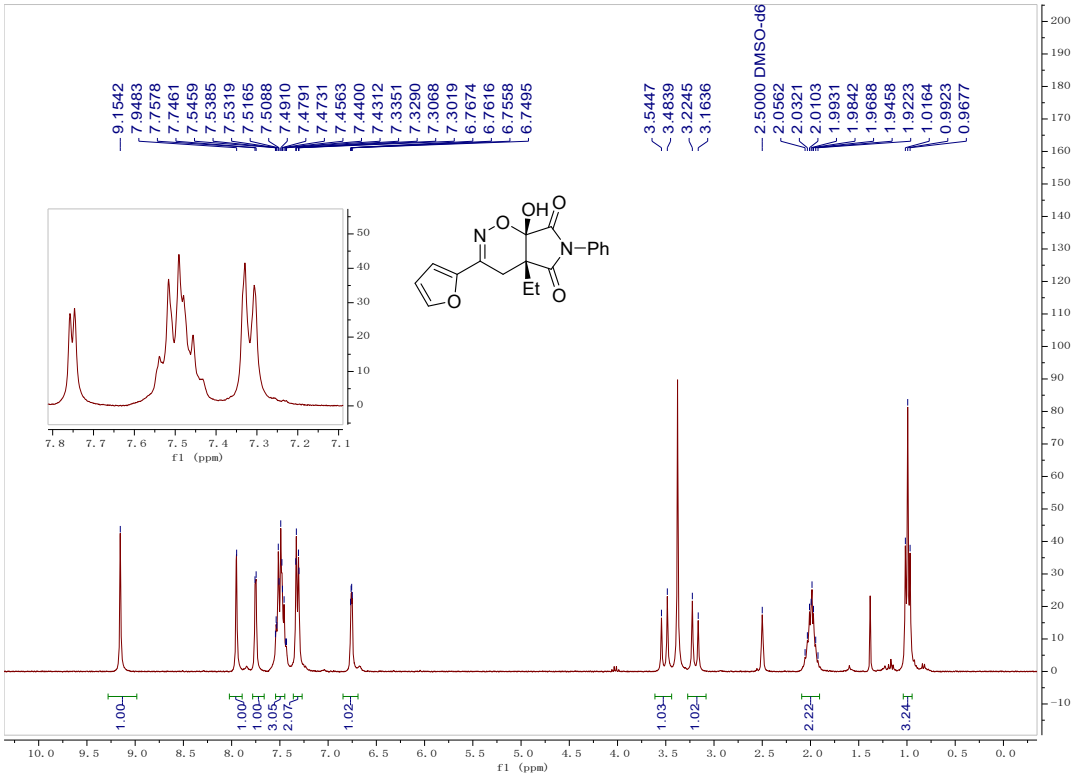


^{13}C NMR (75 MHz, DMSO-d_6)

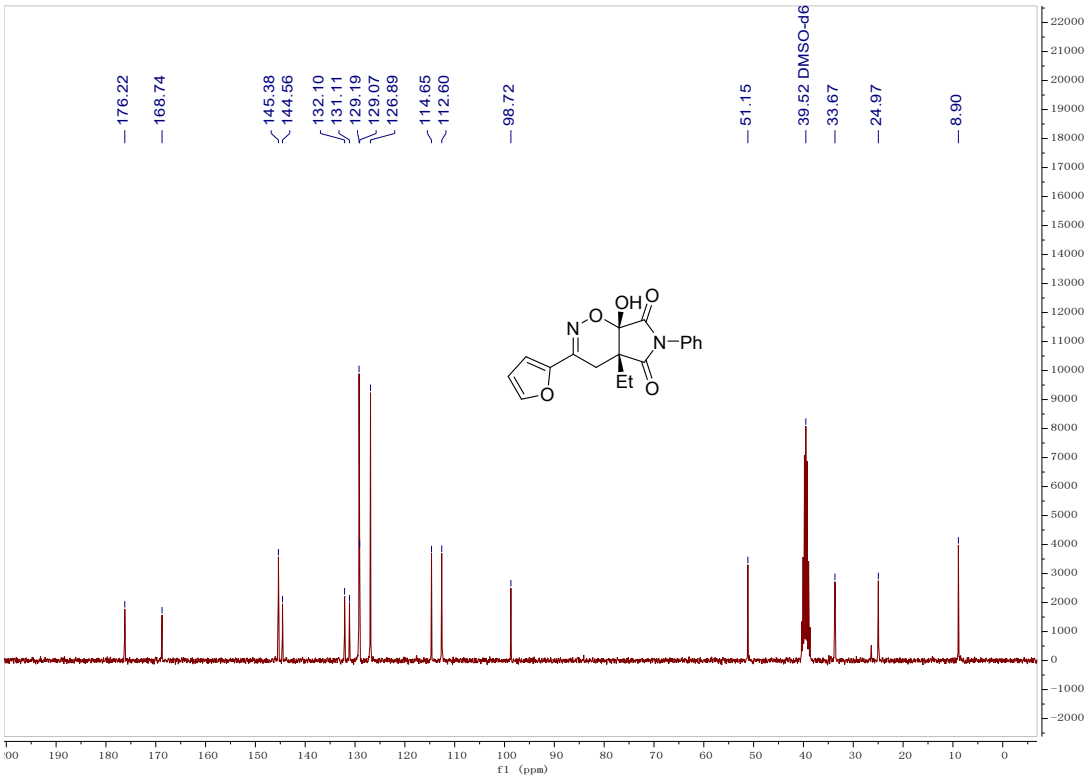


3am

[1]H NMR (300 MHz, DMSO-d_6)

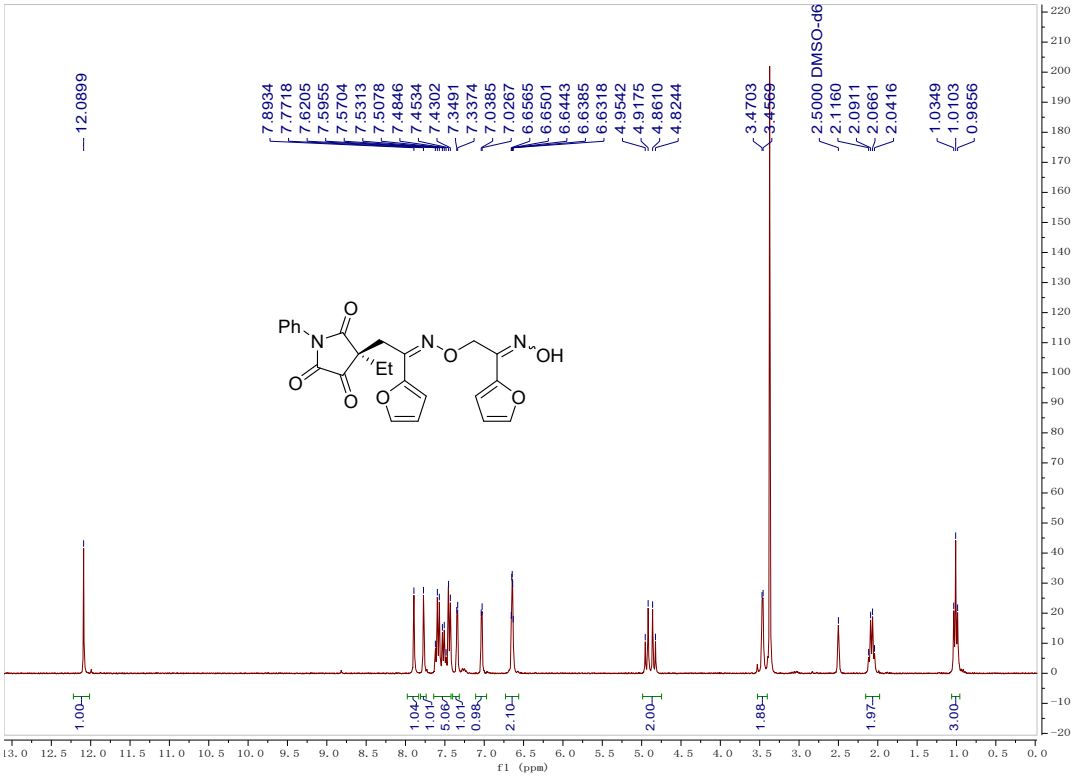


[13]C NMR (75 MHz, DMSO-d_6)

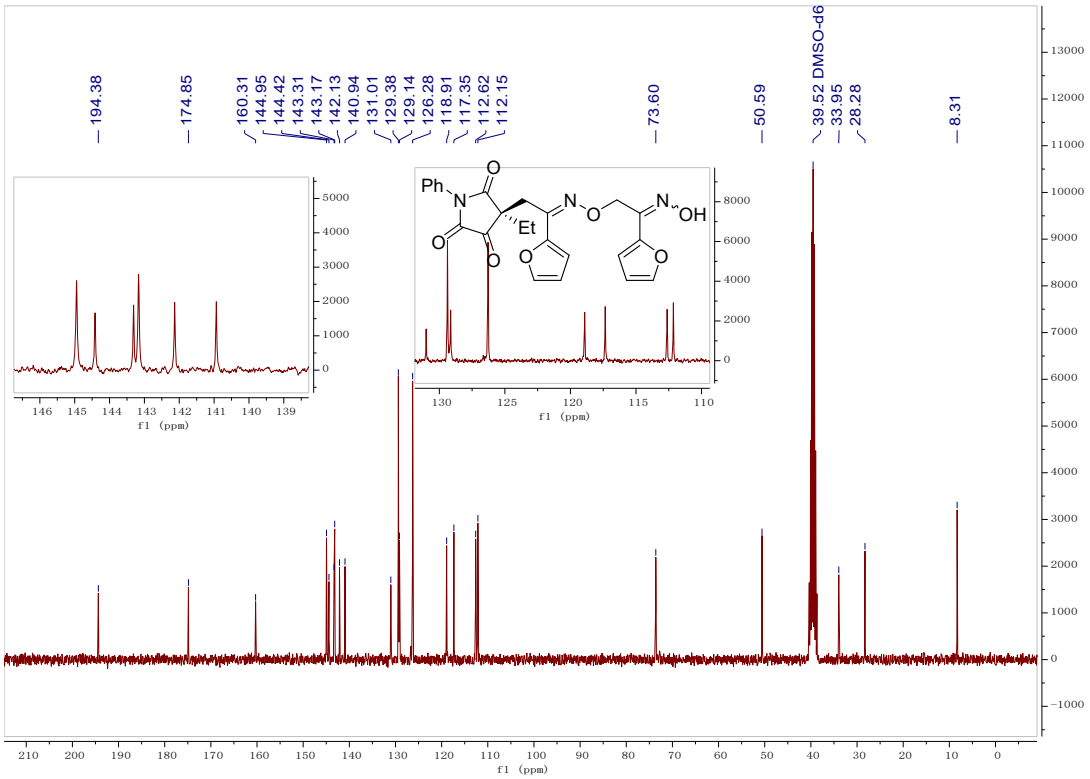


3am'

^{1}H NMR (300 MHz, DMSO-d_6)

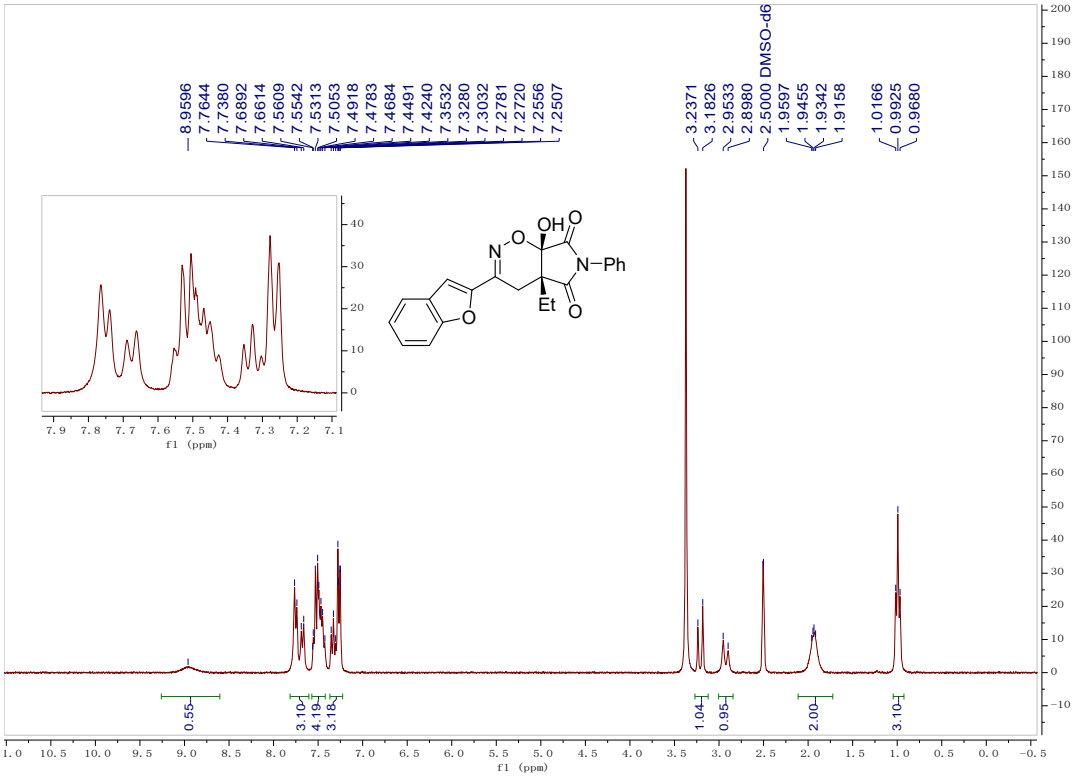


^{13}C NMR (75 MHz, DMSO-d_6)

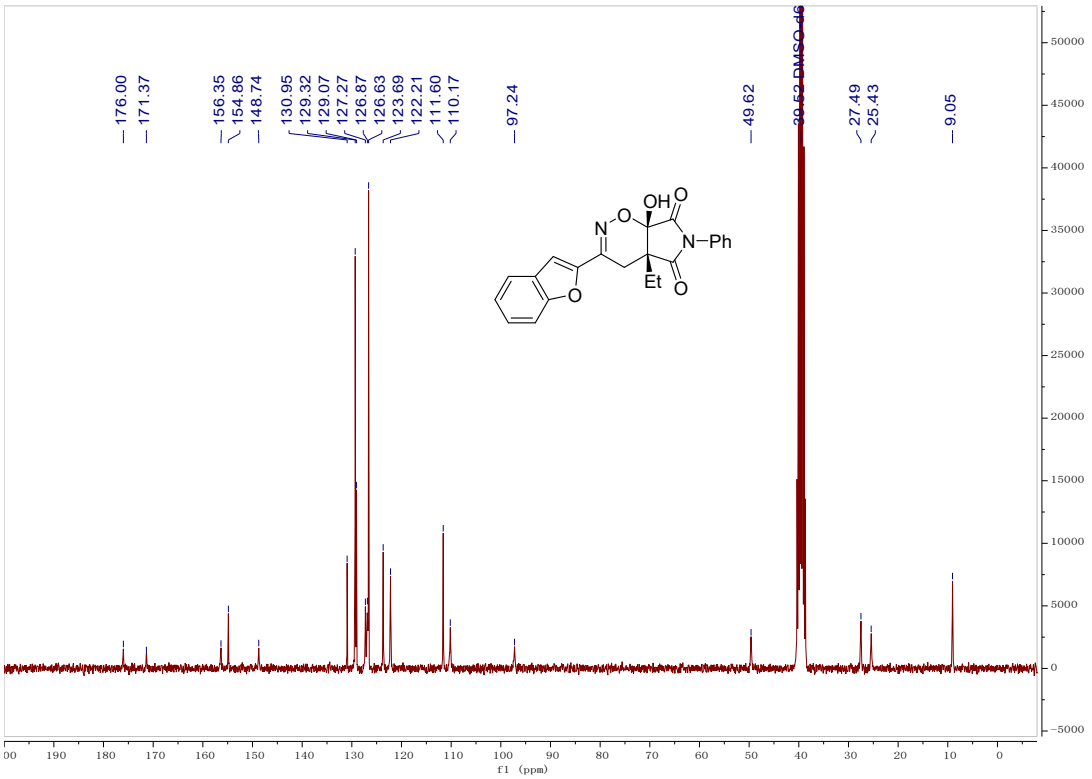


3an

^{1}H NMR (300 MHz, DMSO-d_6)

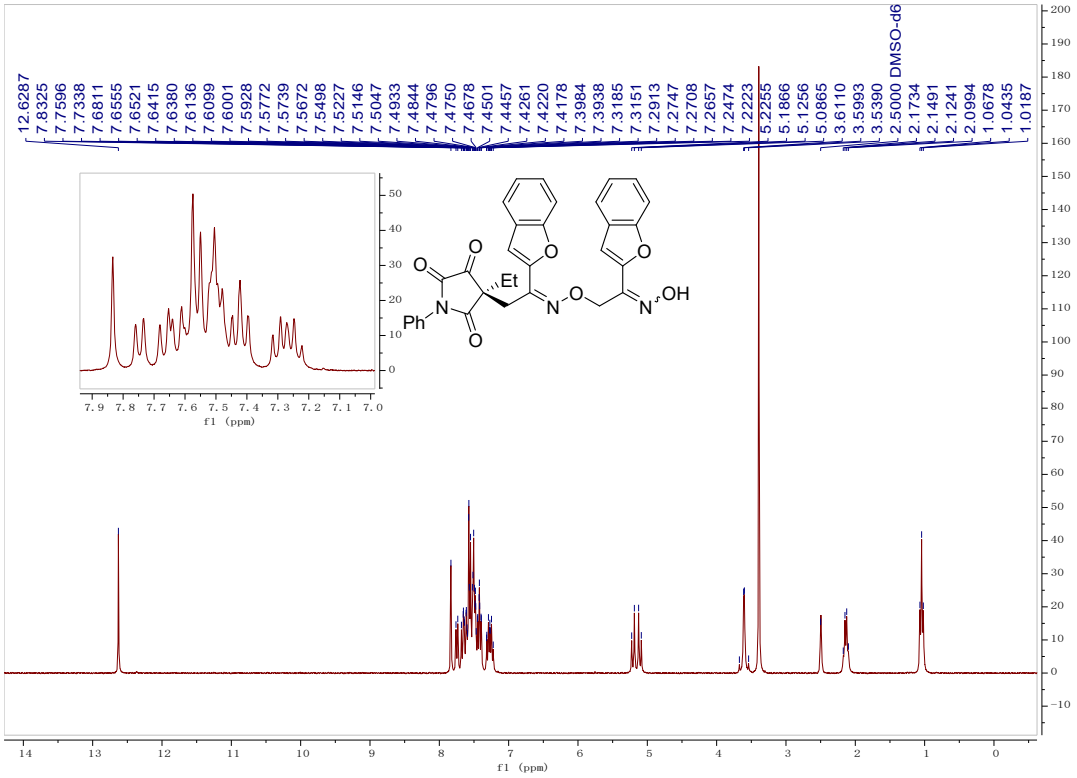


^{13}C NMR (75 MHz, DMSO-d_6)

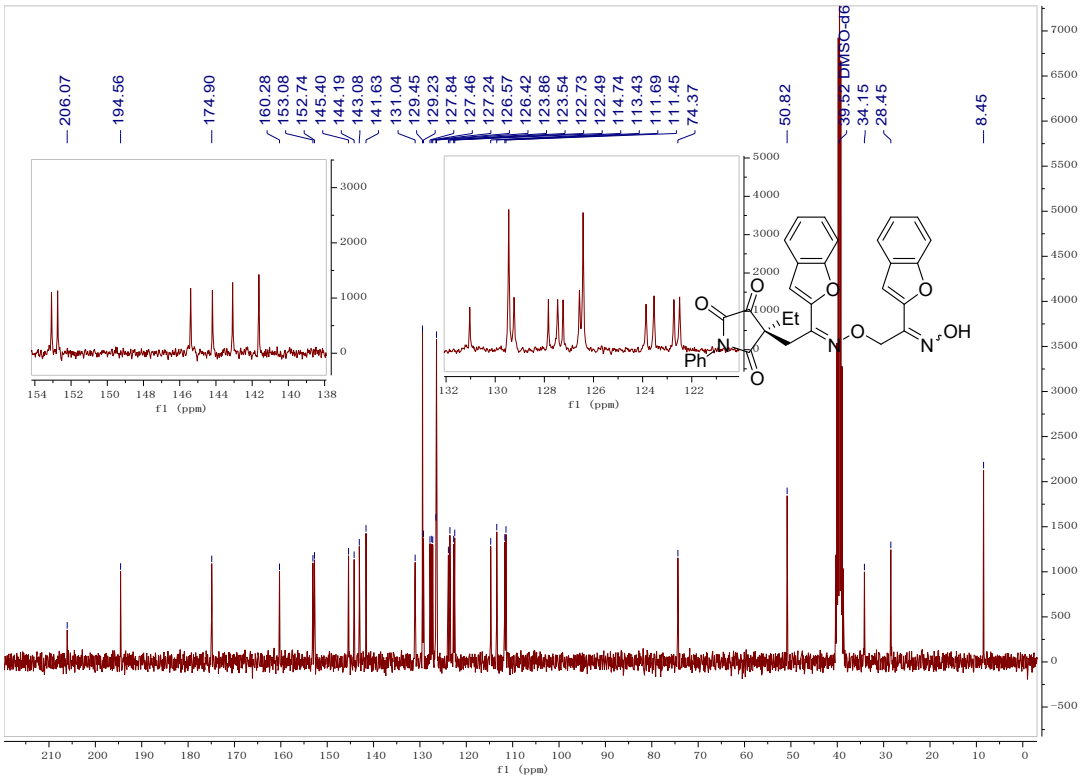


3an'

^{1}H NMR (300 MHz, DMSO-d_6)

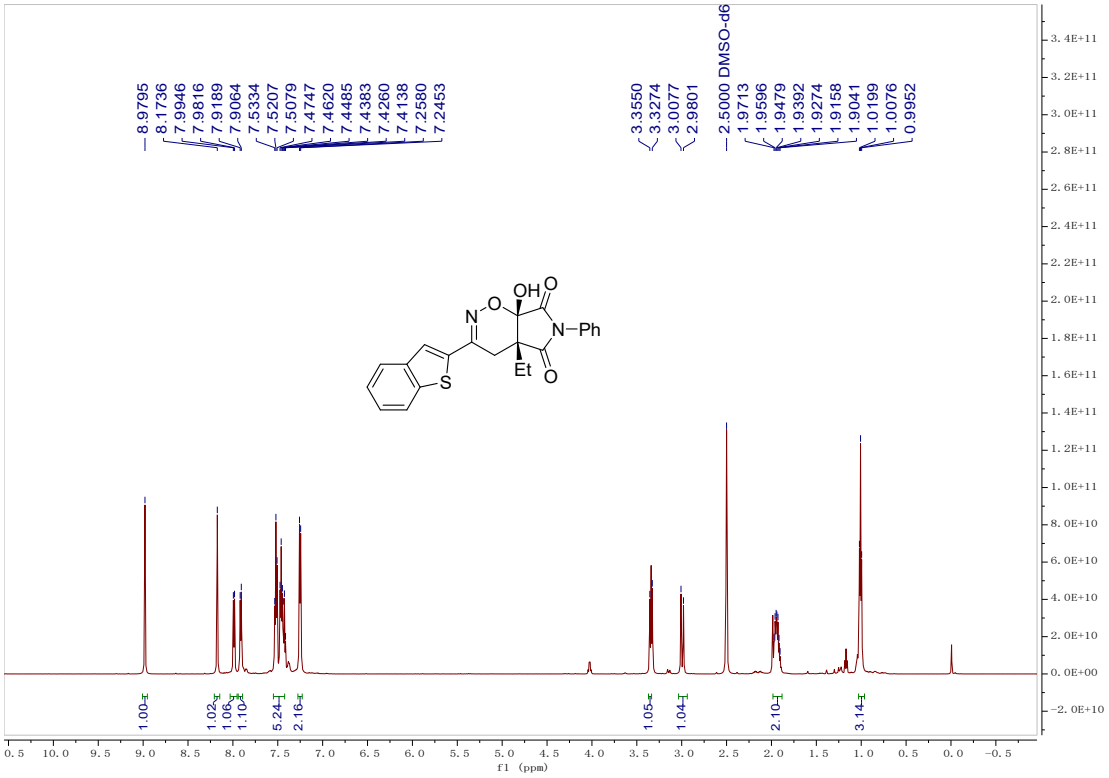


^{13}C NMR (75 MHz, DMSO-d_6)

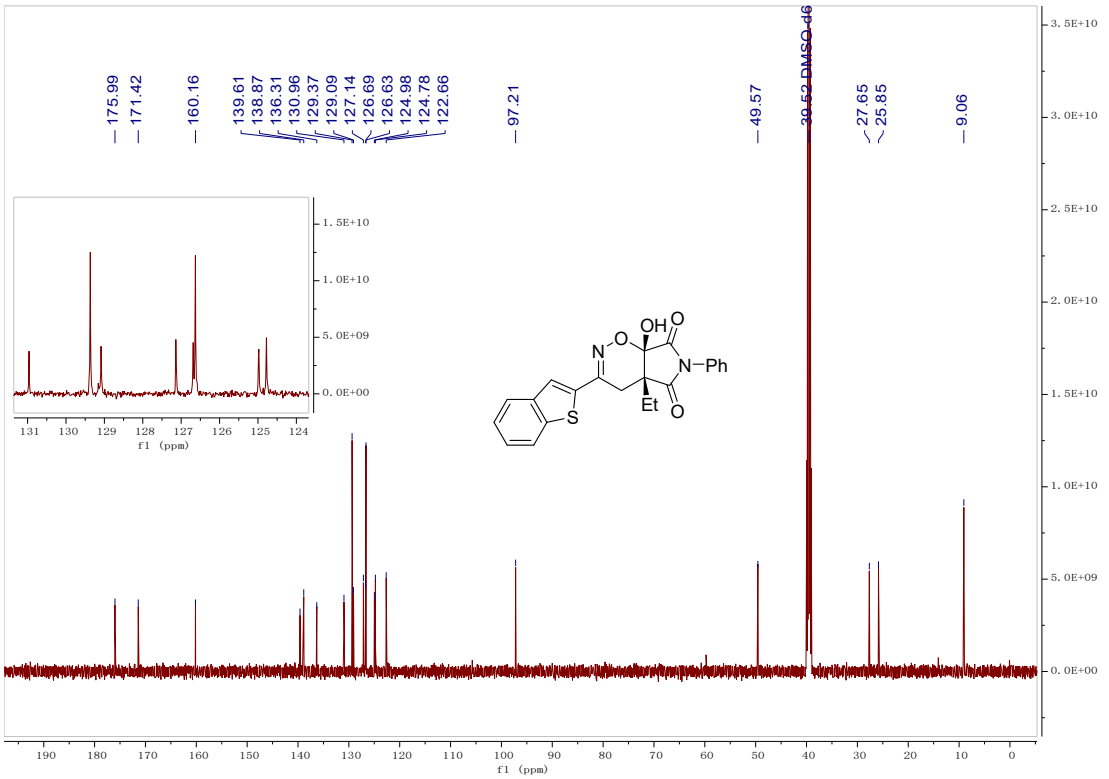


3ao

^{1}H NMR (600 MHz, DMSO-d_6)

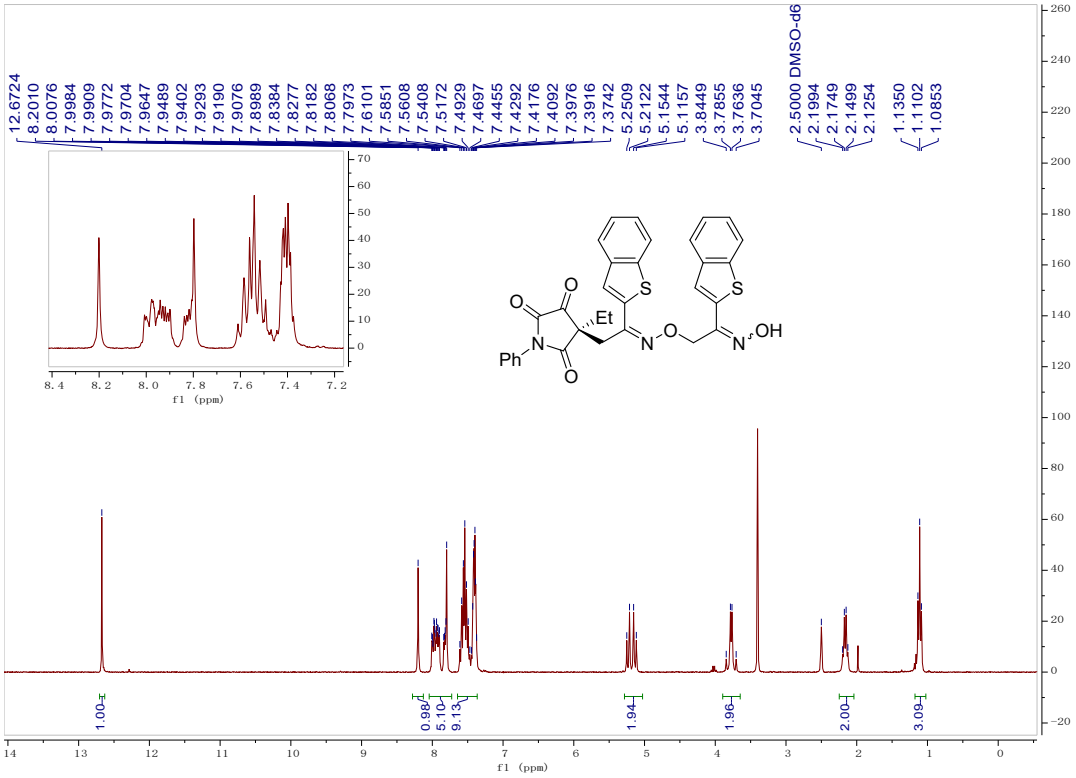


^{13}C NMR (150 MHz, DMSO-d_6)

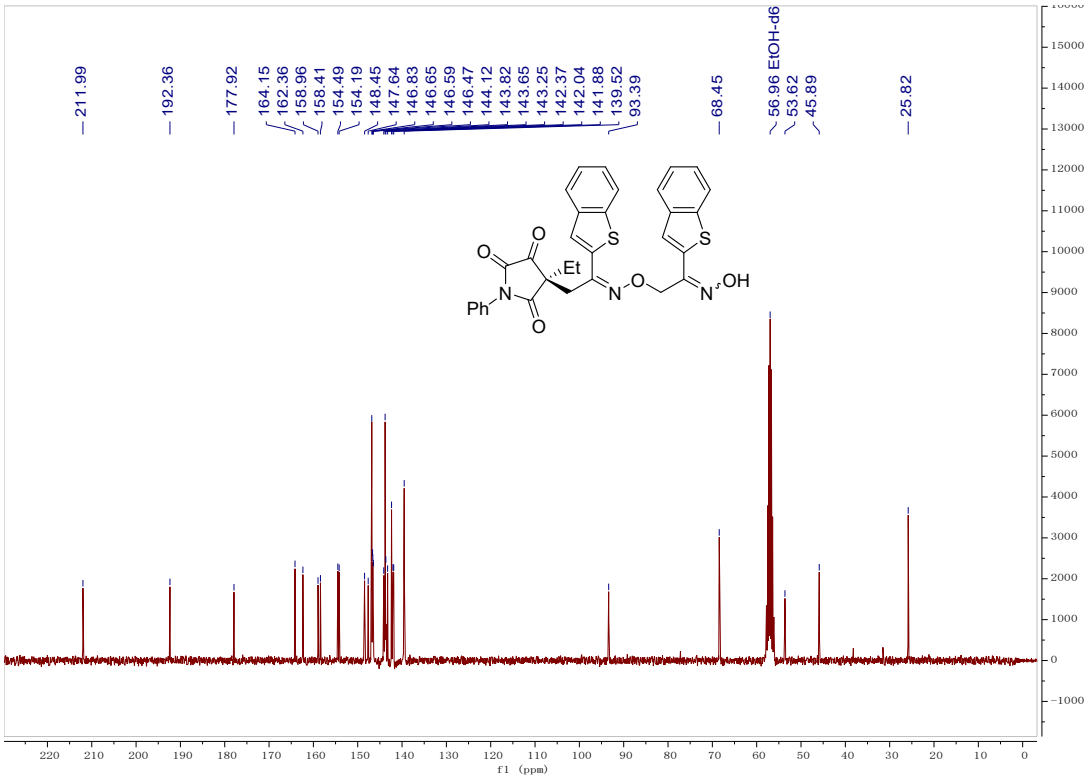


3ao'

^{1}H NMR (300 MHz, DMSO-d_6)

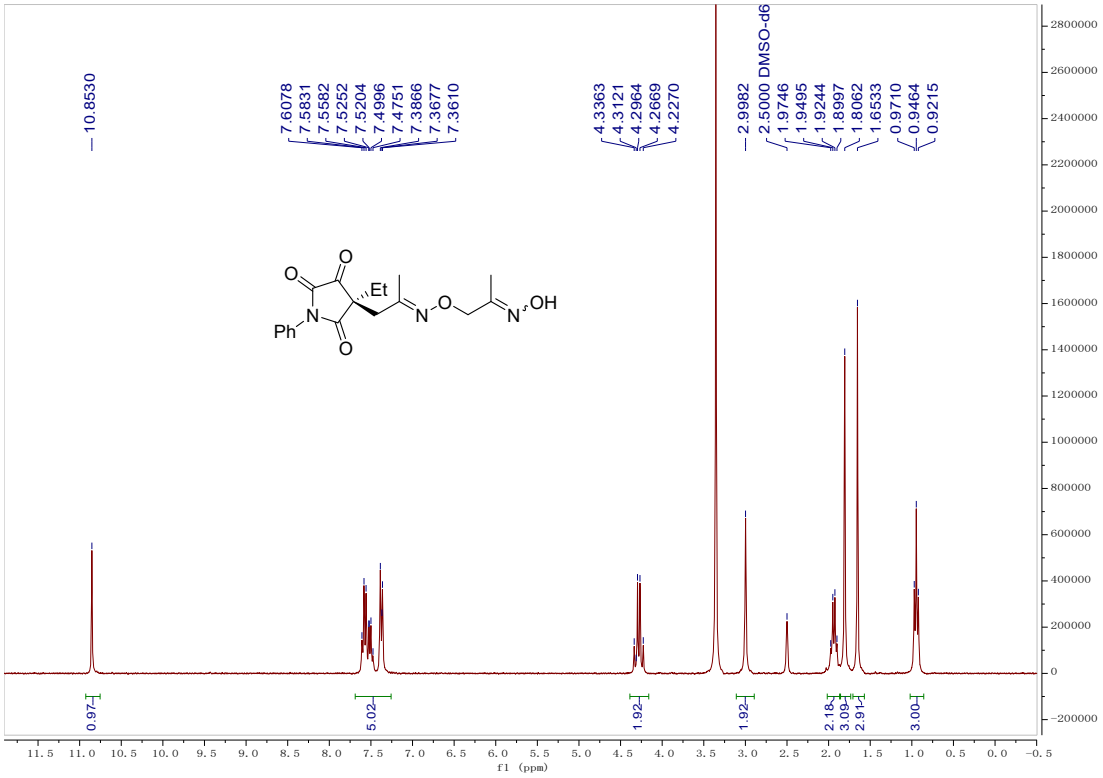


^{13}C NMR (75 MHz, DMSO-d_6)

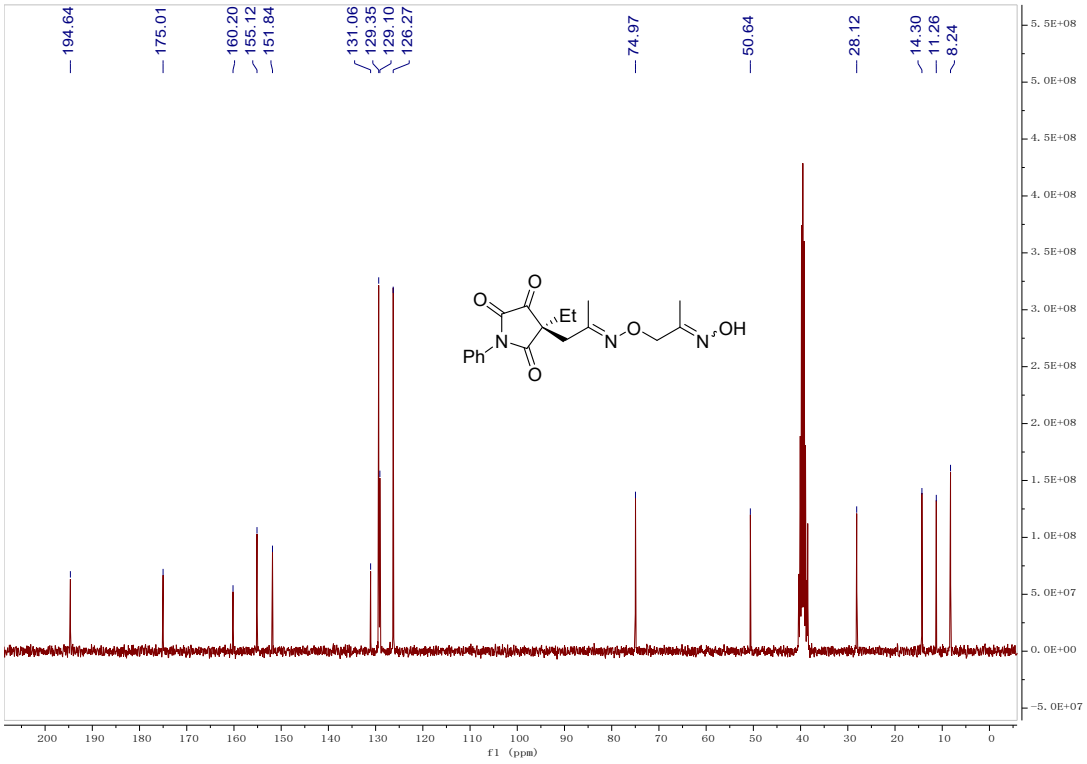


3ap'

^{1}H NMR (300 MHz, DMSO-d_6)

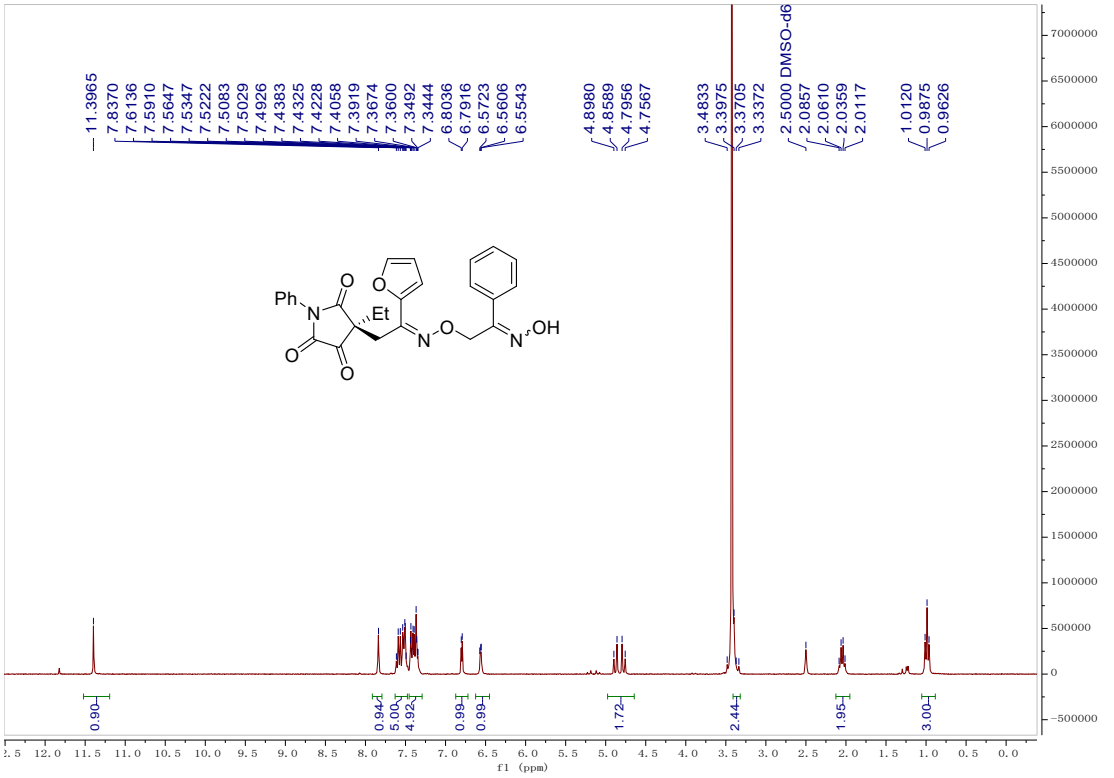


^{13}C NMR (75 MHz, DMSO-d_6)

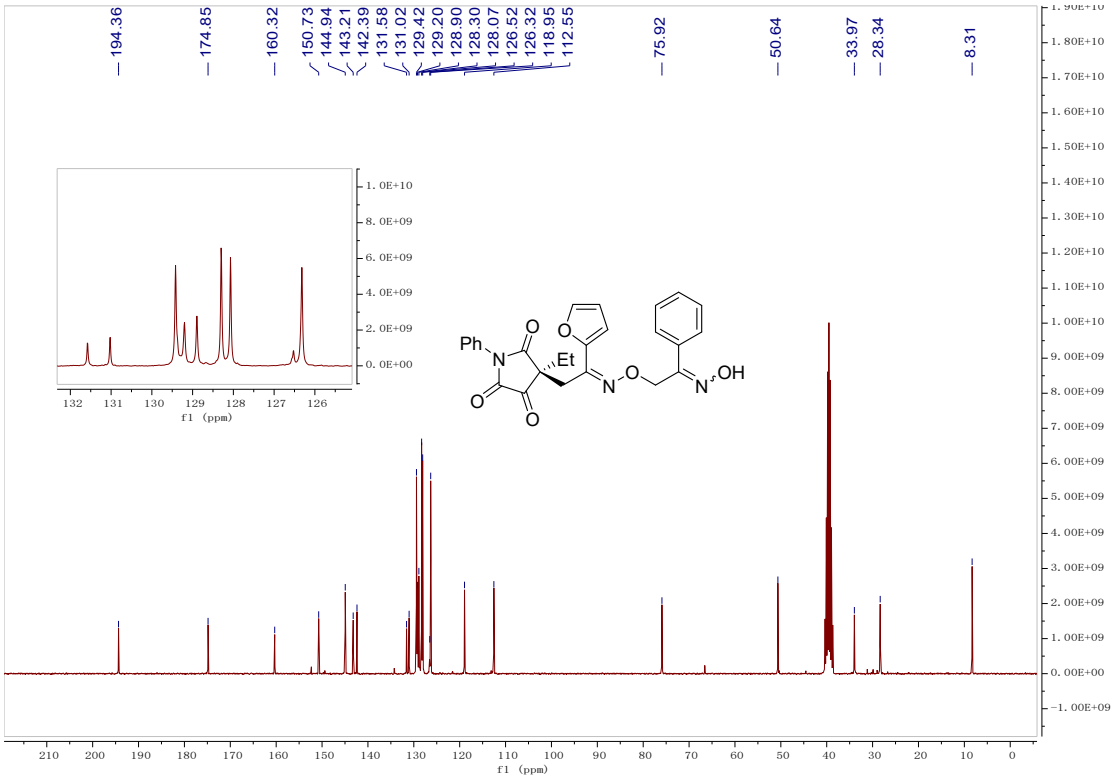


3ama

^{1}H NMR (300 MHz, DMSO-d_6)

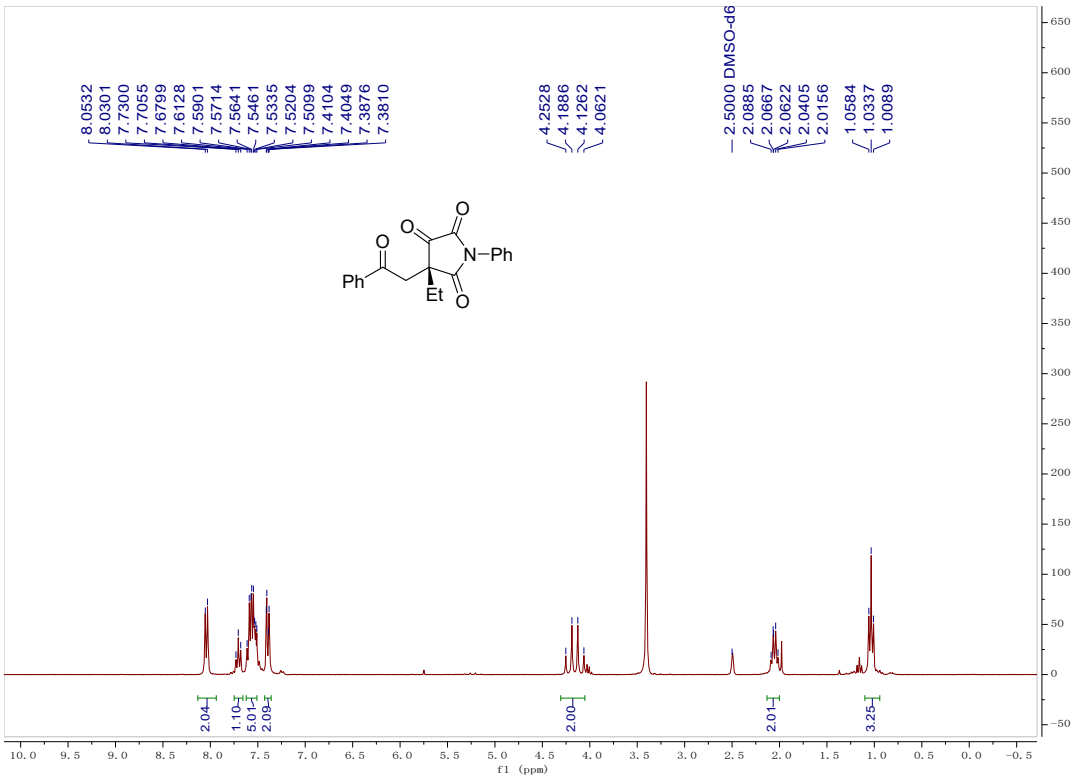


^{13}C NMR (75 MHz, DMSO-d_6)

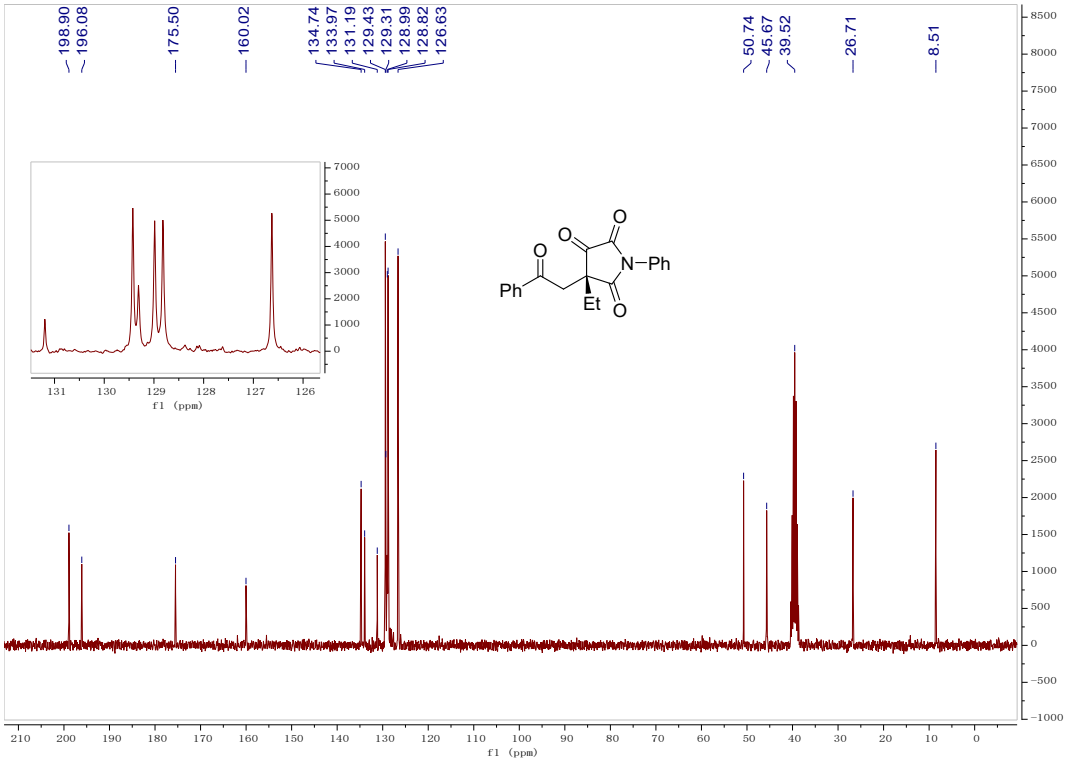


5

^{1}H NMR (300 MHz, DMSO-d_6)

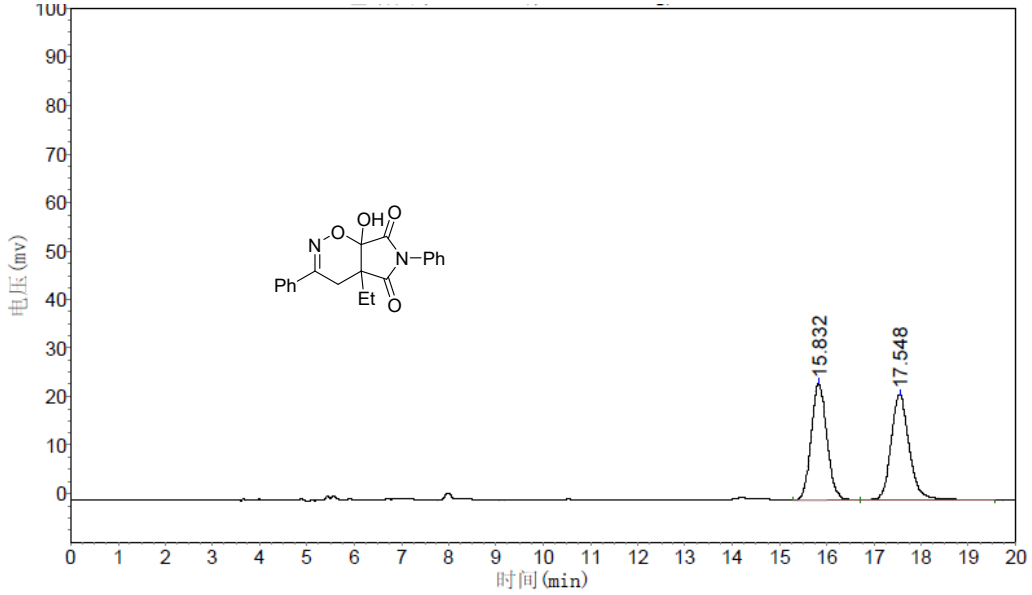


^{13}C NMR (75 MHz, DMSO-d_6)

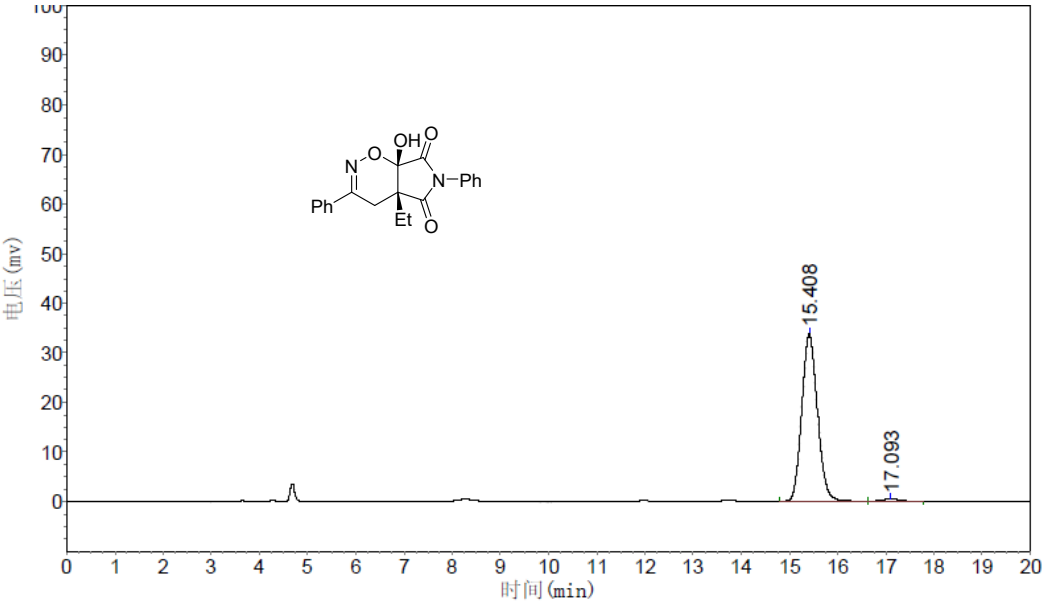


HPLC

3aa



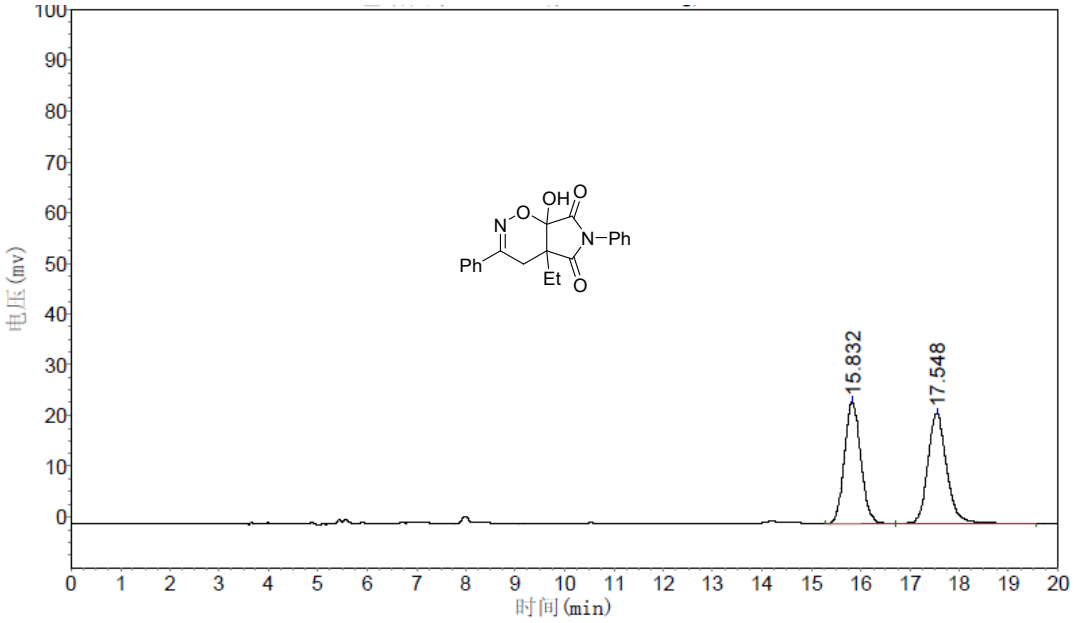
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.827	24066.148	557999.688	49.1340
2	17.548	21693.217	577668.438	50.8660



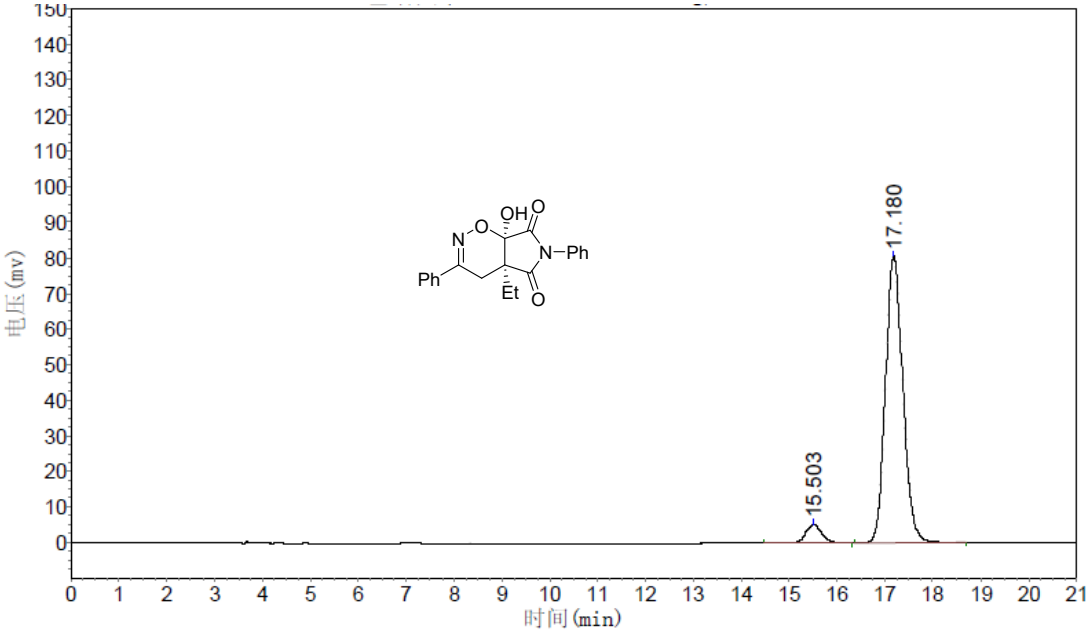
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.408	33863.457	760736.688	98.5613
2	17.093	454.692	11104.500	1.4387

HPLC conditions: Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 17 min

3aa'



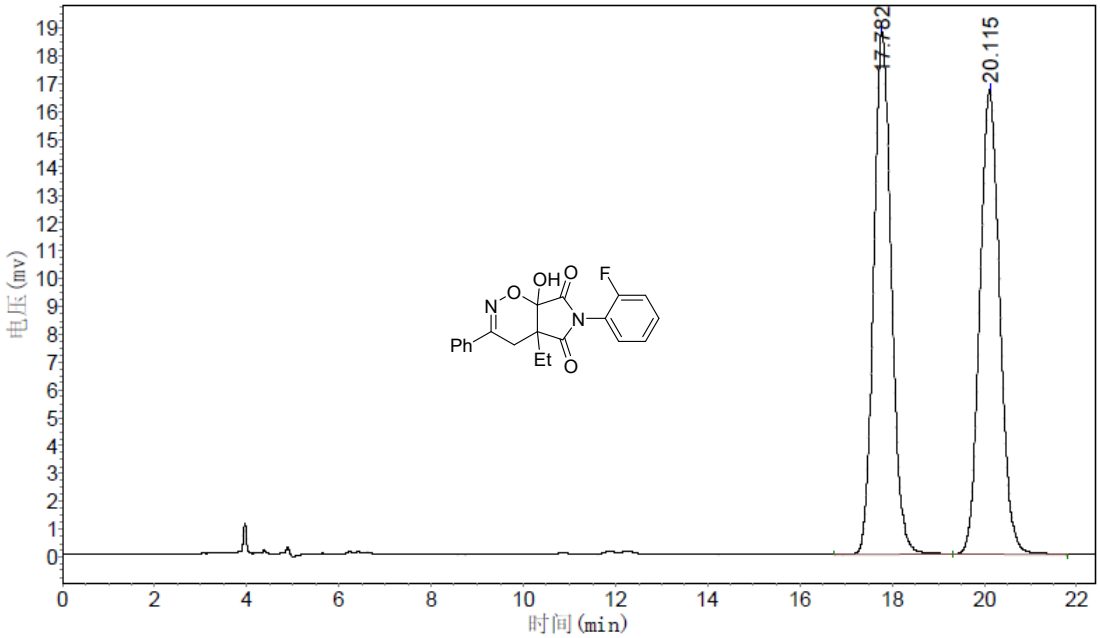
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.832	24066.148	557999.688	49.1340
2	17.548	21693.217	577668.438	50.8660



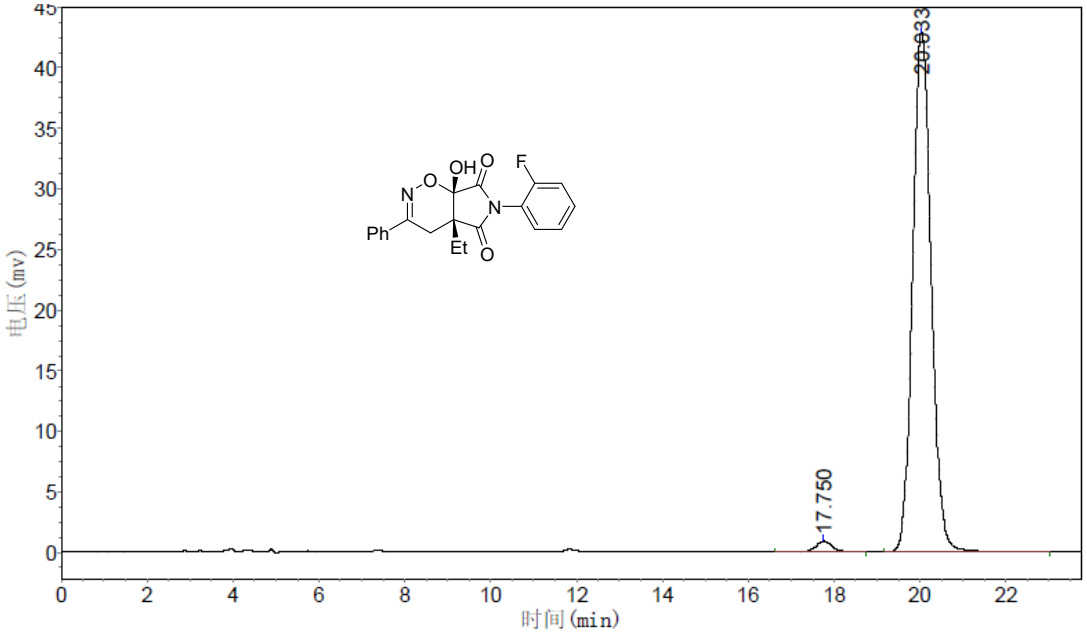
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.503	5139.938	113870.164	5.2945
2	17.180	80435.602	2036871.250	94.7055

HPLC conditions: Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 17 min

3ba



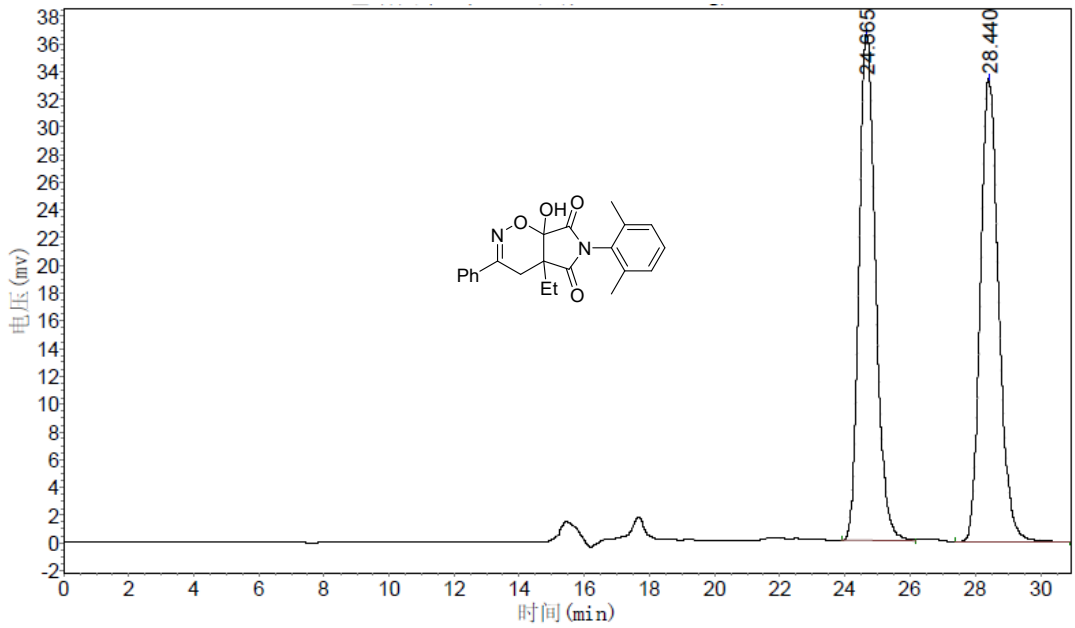
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	17.782	18778.156	503208.000	50.0330
2	20.115	16692.918	502543.313	49.9670



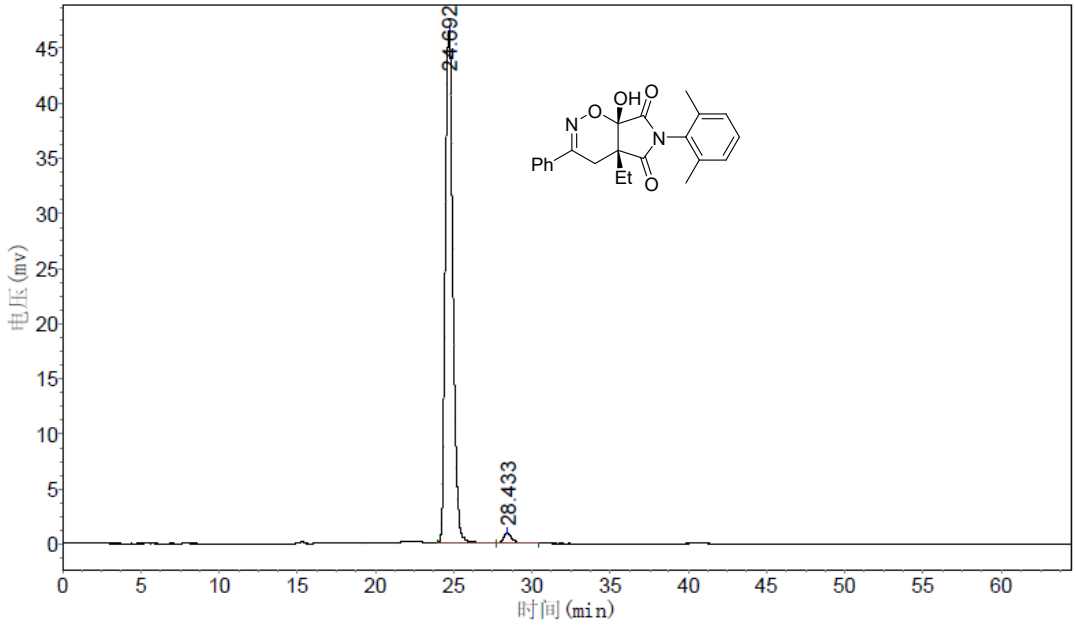
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	17.750	903.929	24355.000	1.8576
2	20.033	42840.961	1286759.375	98.1424

HPLC conditions: Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 17 min, t_{major} = 20 min

3ca



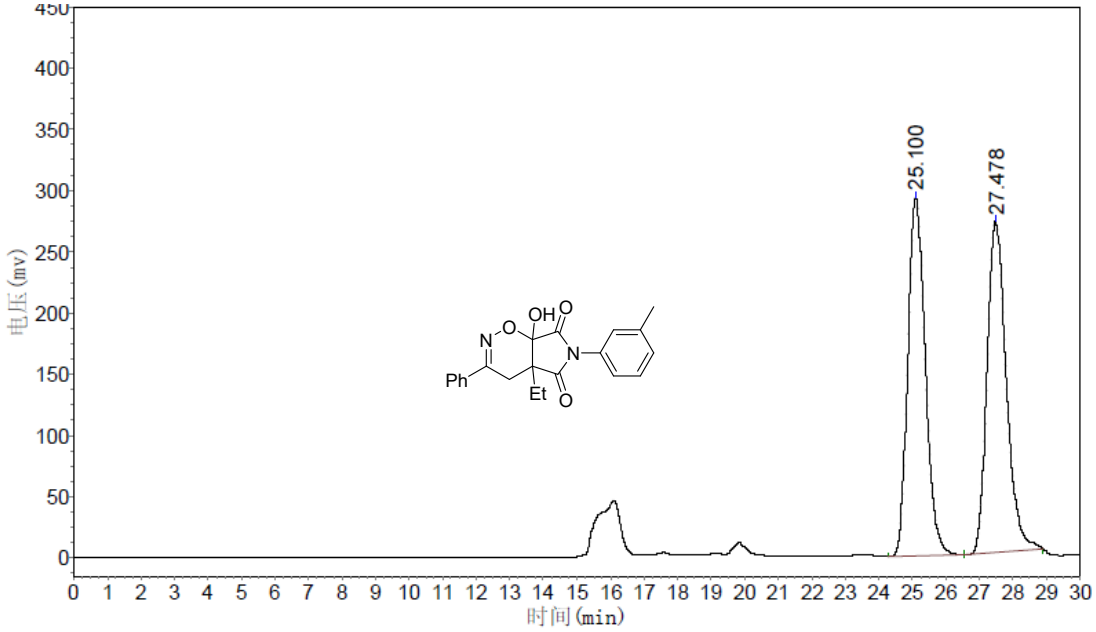
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	24.665	36545.426	1298136.000	49.7389
2	28.440	33384.840	1304495.625	49.9826



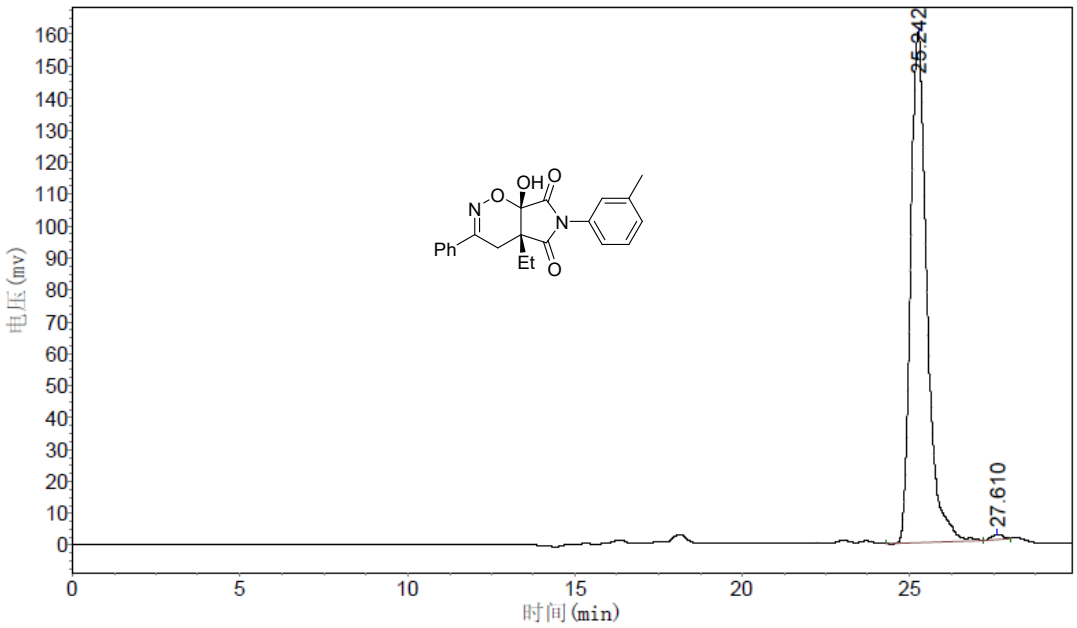
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	24.692	46528.074	1543716.500	97.7531
2	28.433	950.273	35482.434	2.2469

HPLC conditions: Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24 min, t_{minor} = 28 min

3da



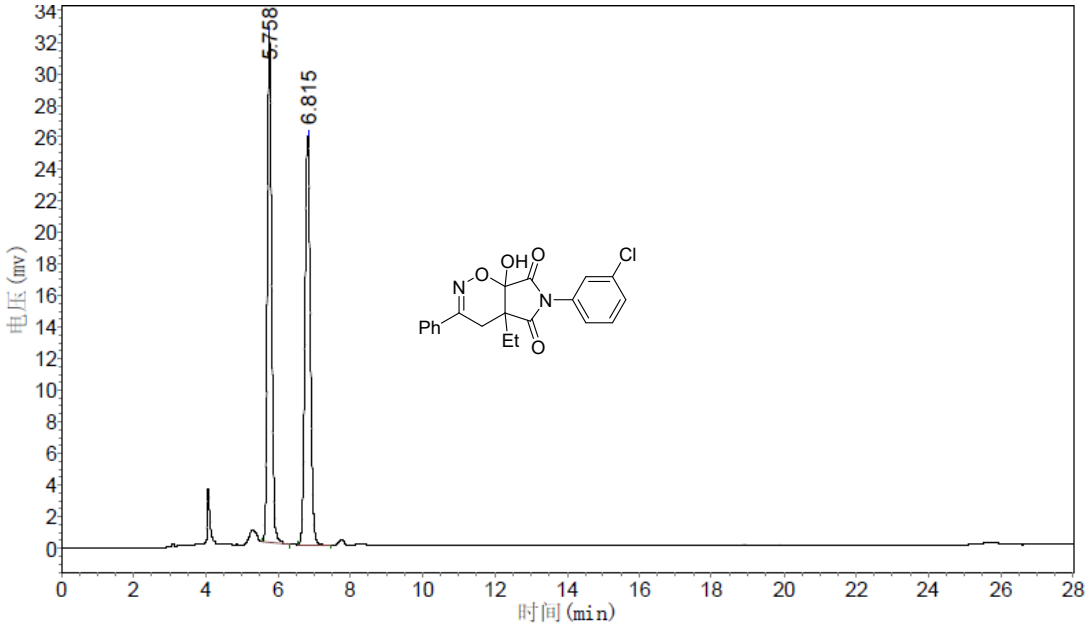
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	25.100	293124.344	10581355.000	49.6069
2	27.478	270466.375	10749064.000	50.3931



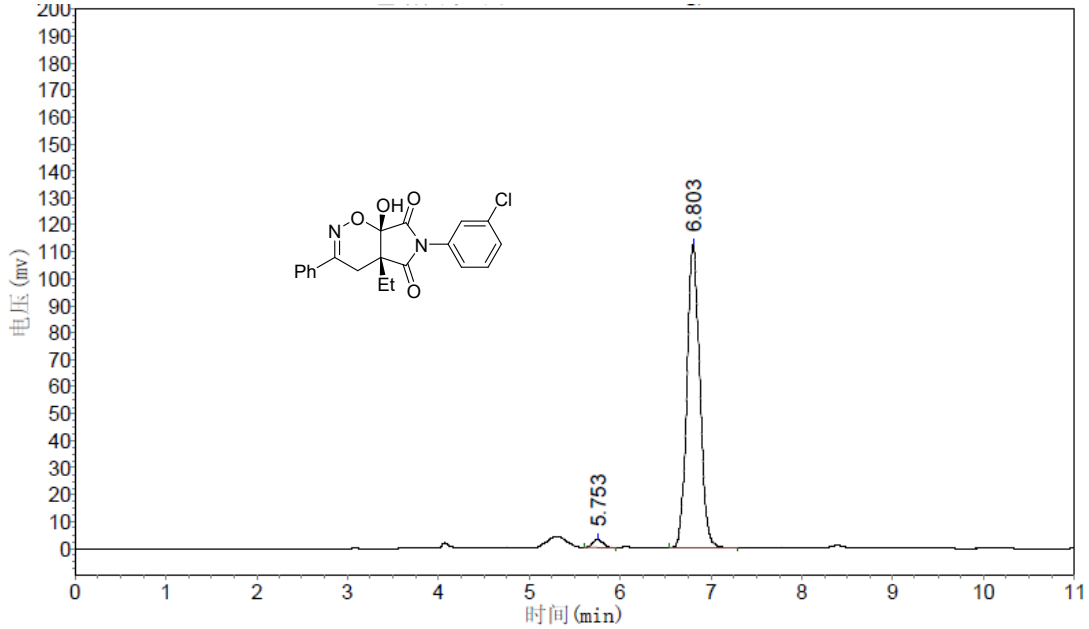
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	25.242	159787.891	5398044.500	99.3486
2	27.610	1512.630	35393.914	0.6514

HPLC conditions: Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 25 min, t_{minor} = 27 min

3ea



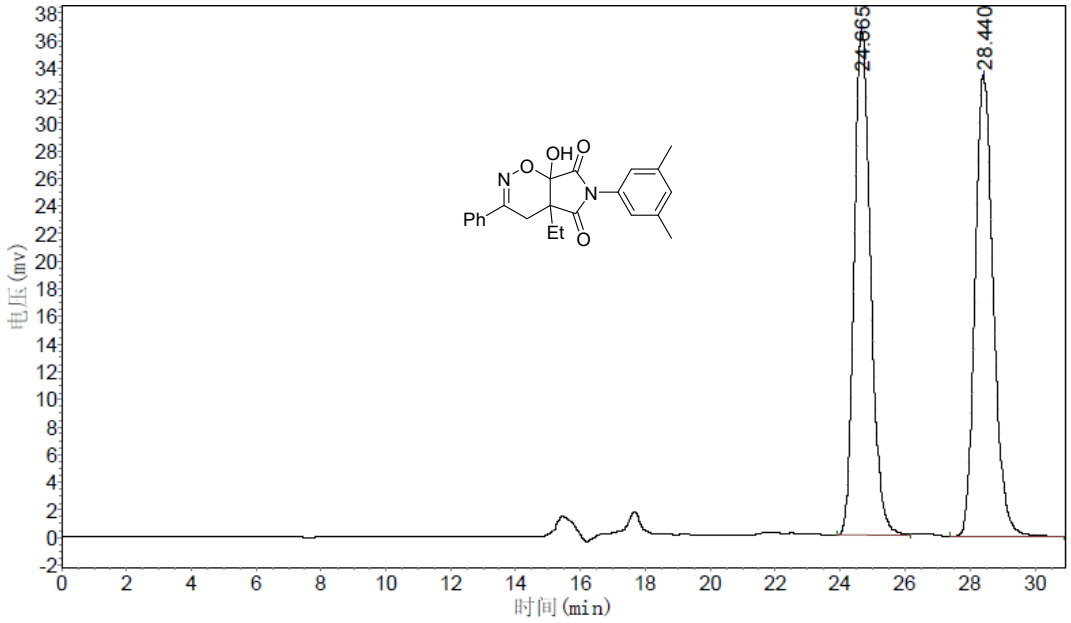
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.758	32299.521	267806.688	51.2212
2	6.815	25835.373	255037.000	48.7788



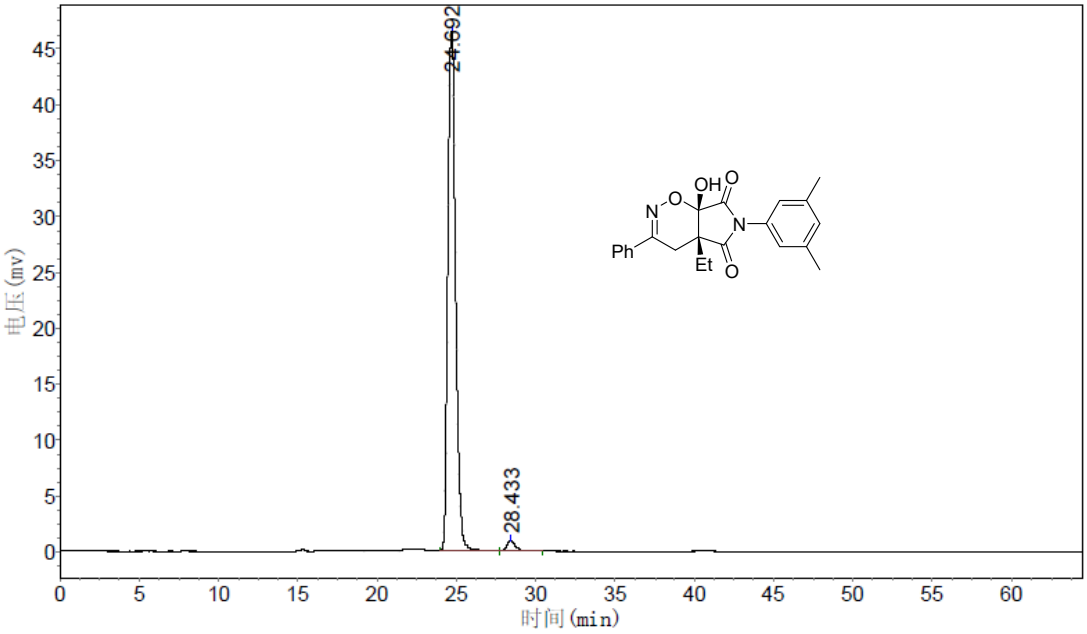
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.753	2956.296	22936.852	2.0261
2	6.803	112480.641	1109129.750	97.9739

Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5 min, t_{major} = 6 min

3fa



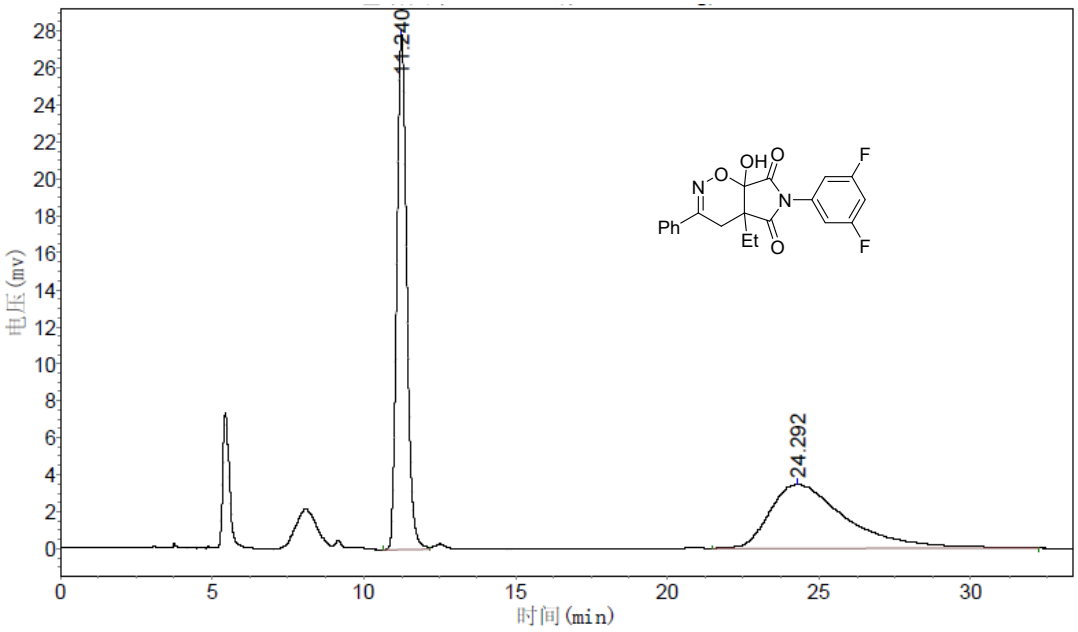
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	24.665	36545.426	1298136.000	49.7389
2	28.440	33384.840	1304495.625	49.9826



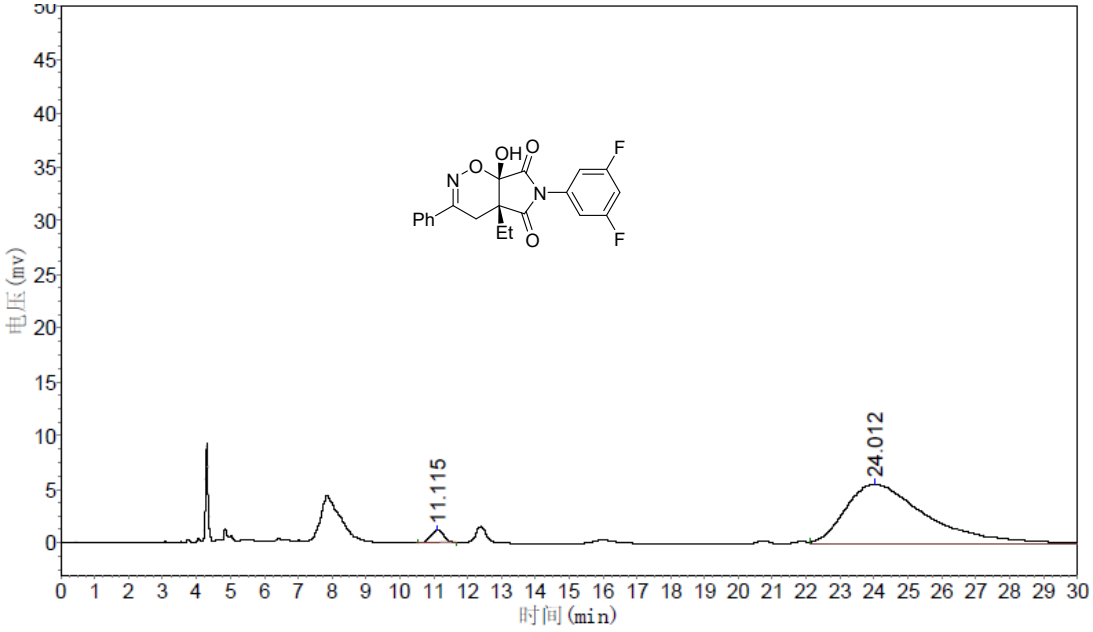
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	24.692	46528.074	1543716.500	97.7531
2	28.433	950.273	35482.434	2.2469

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24min, t_{minor} = 28 min

3ga



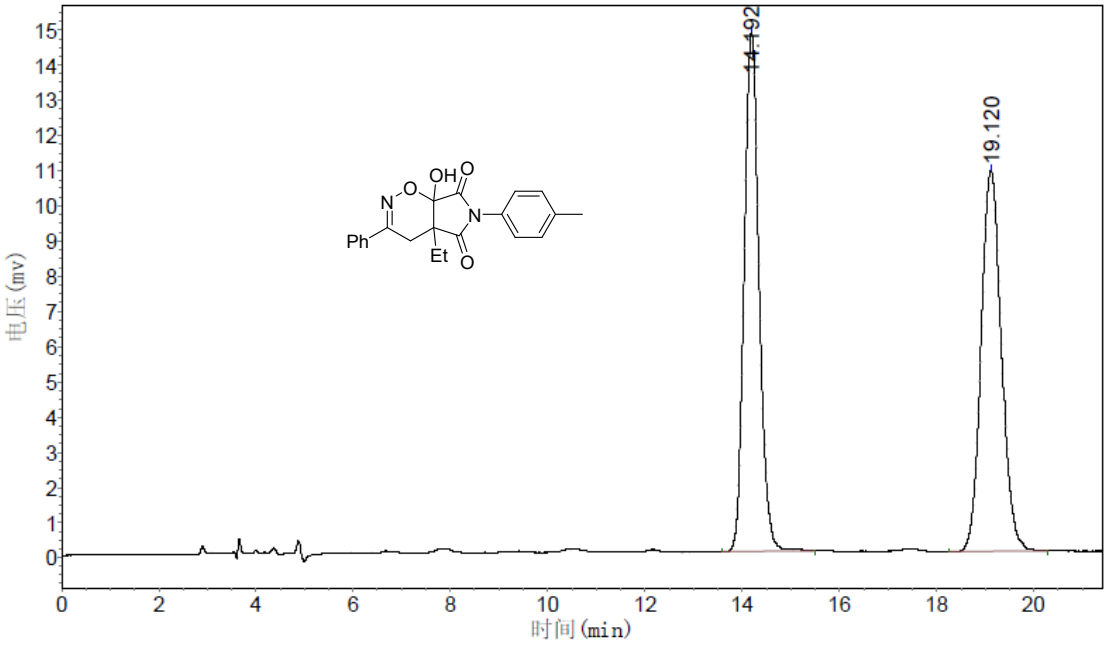
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	11.240	27906.396	630051.750	50.8120
2	24.292	3464.418	609915.750	49.1880



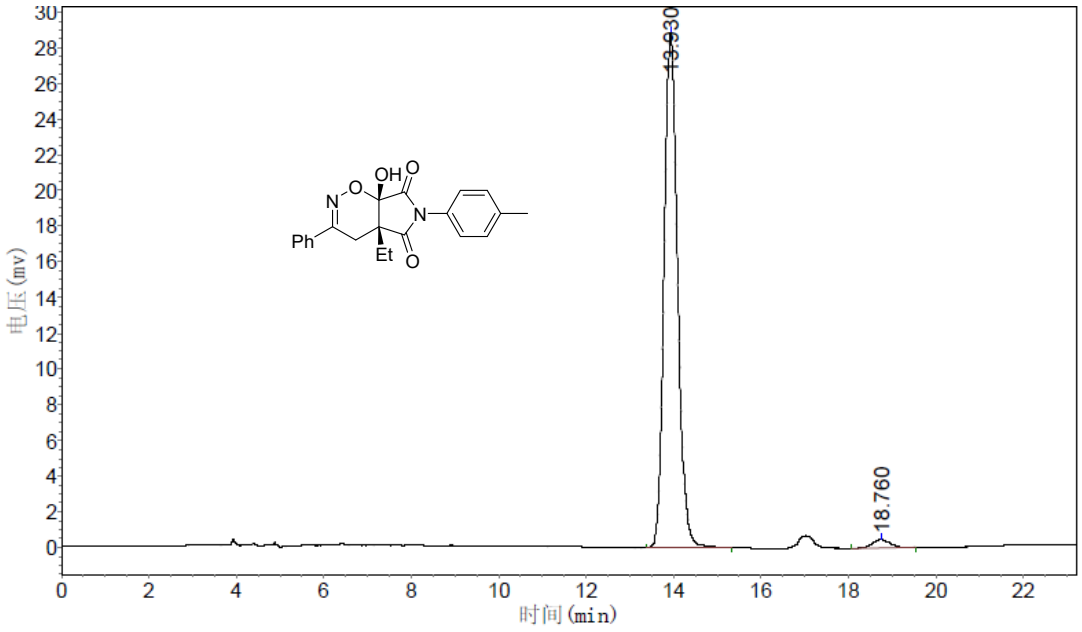
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	11.115	1124.944	29053.533	3.0614
2	24.012	5494.825	919965.500	96.9386

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 11 min, t_{major} = 24 min

3ha



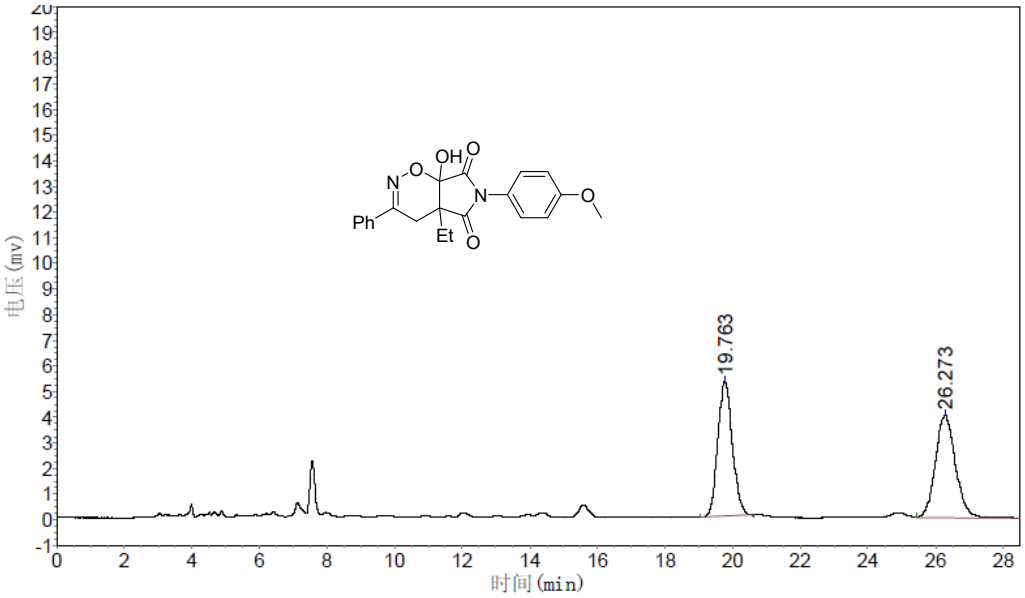
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	14.192	1124.944	29053.533	50.0500
2	19.120	10817.121	314046.219	49.9500



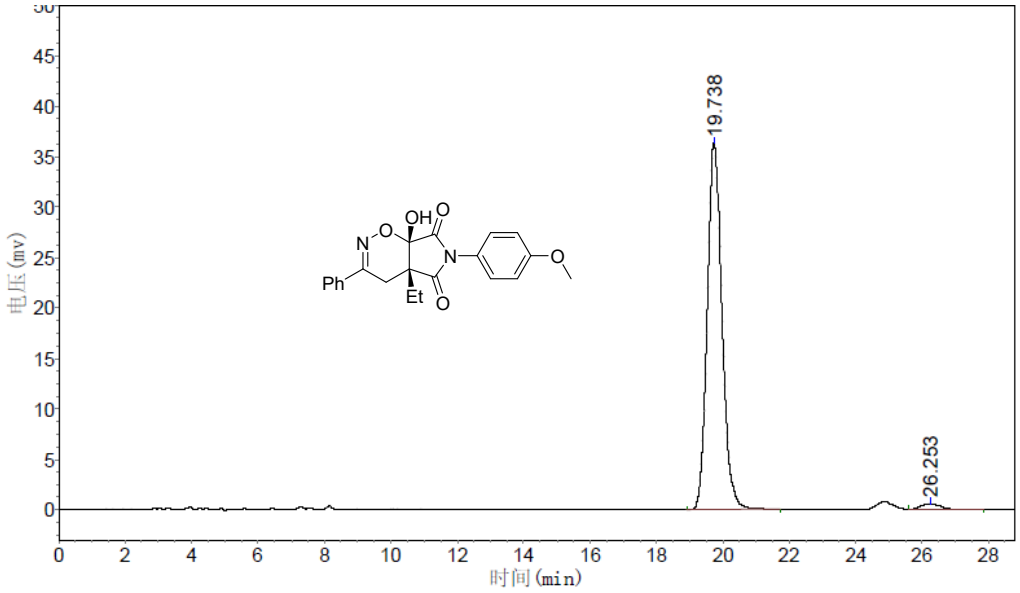
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.930	28870.078	616349.875	97.8330
2	18.760	487.956	13651.851	2.1670

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14min, t_{minor} = 18 min

3ia



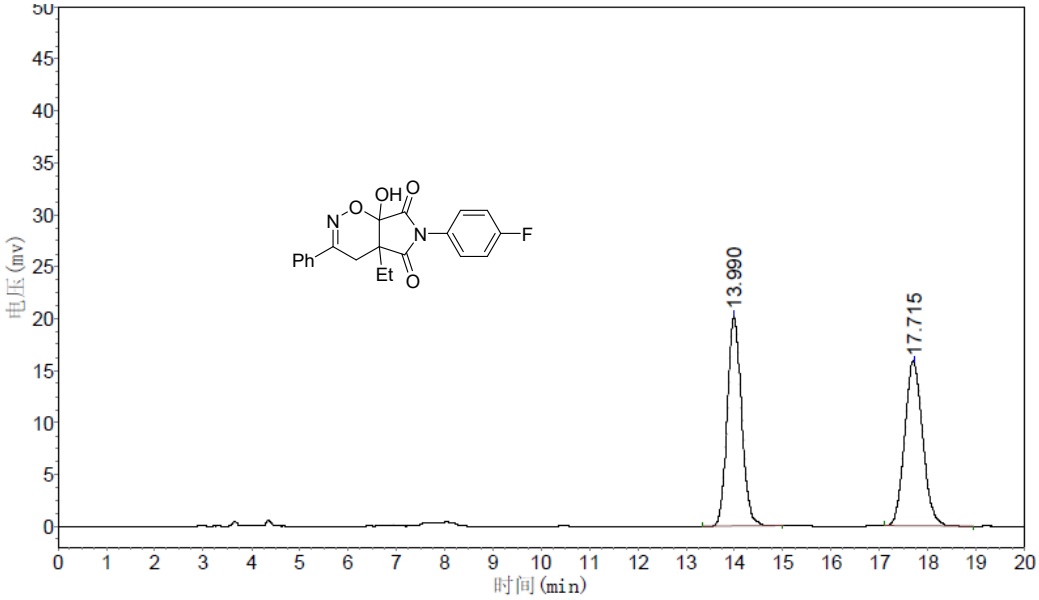
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	19.763	5247.229	157341.094	49.3942
2	26.273	3957.042	161200.422	50.6058



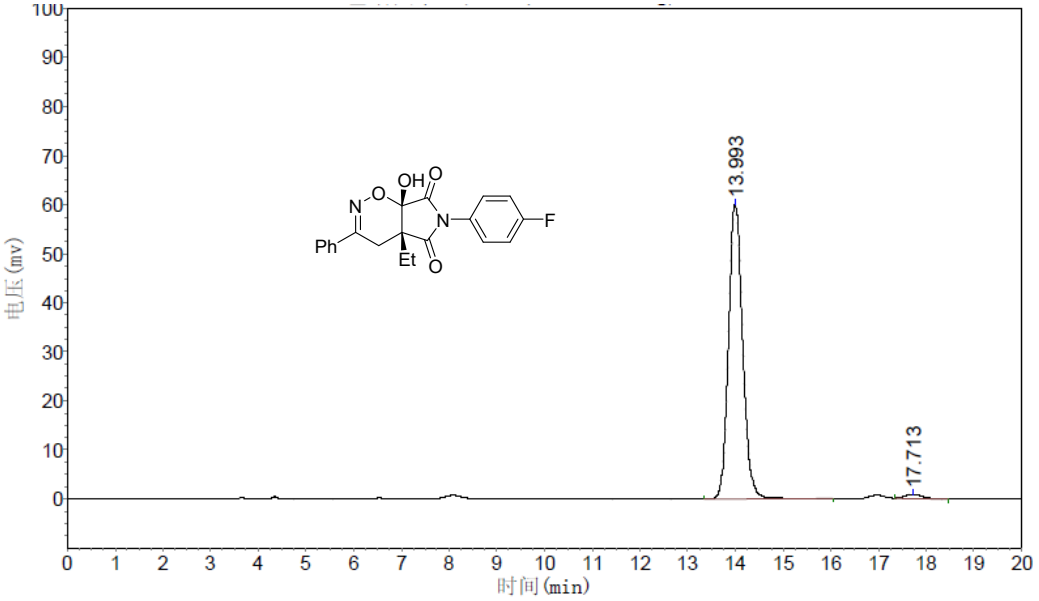
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	19.738	36346.707	1120485.875	97.8678
2	26.253	590.034	24411.162	2.1322

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 19 min, t_{minor}= 26 min

3ja



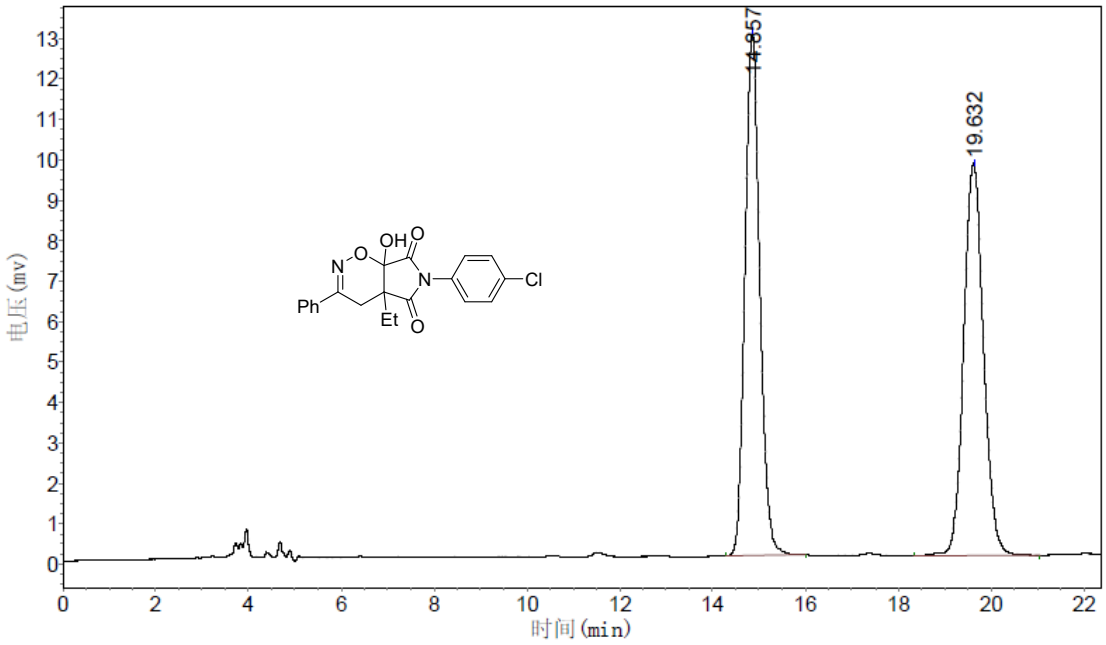
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.990	20232.000	413568.313	49.9276
2	17.715	15846.039	414768.000	50.0724



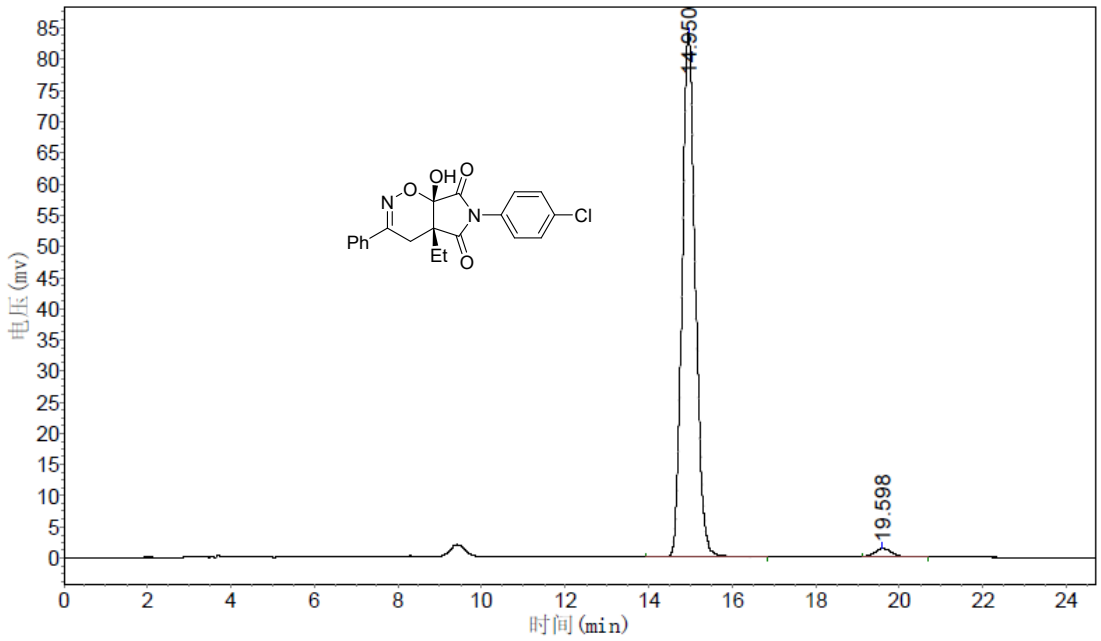
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.993	60052.121	1236301.250	98.1654
2	17.713	889.894	23105.029	1.8346

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14 min, t_{minor} = 17 min

3ka



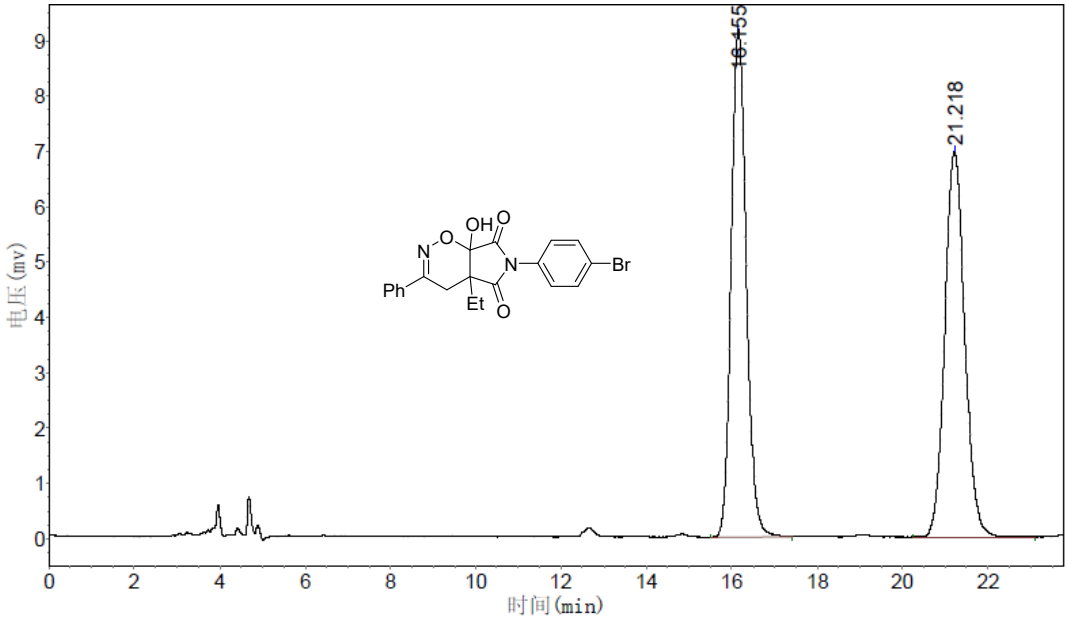
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	14.857	12925.391	288402.250	50.0325
2	19.632	9671.908	288027.813	49.9675



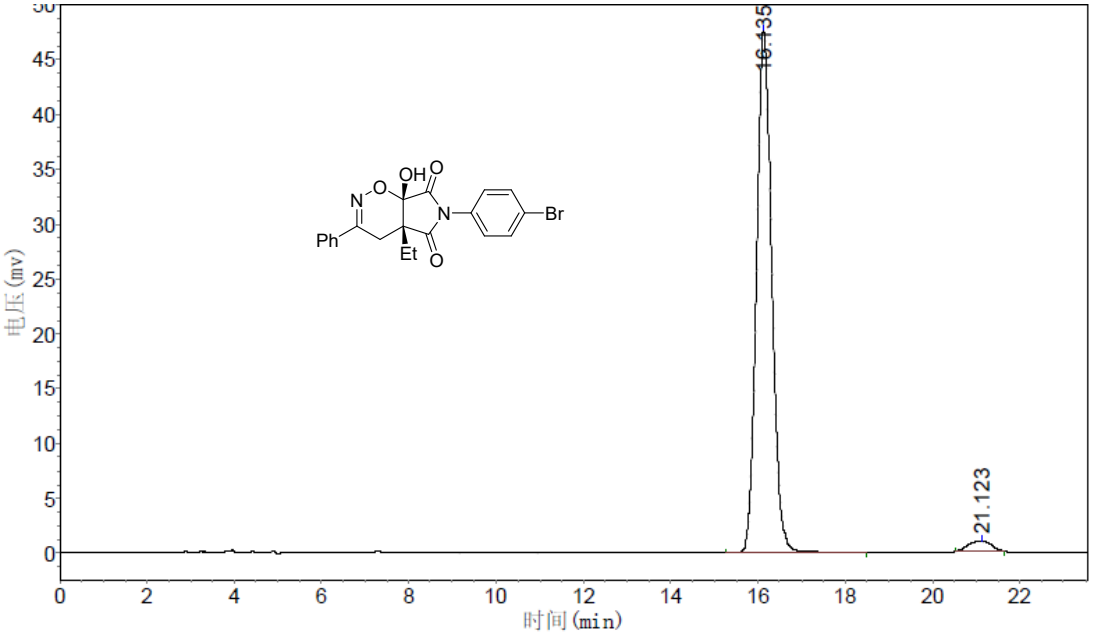
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	14.950	84045.117	1869203.250	97.7897
2	19.598	1454.430	42248.543	2.2103

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 19 min

3la



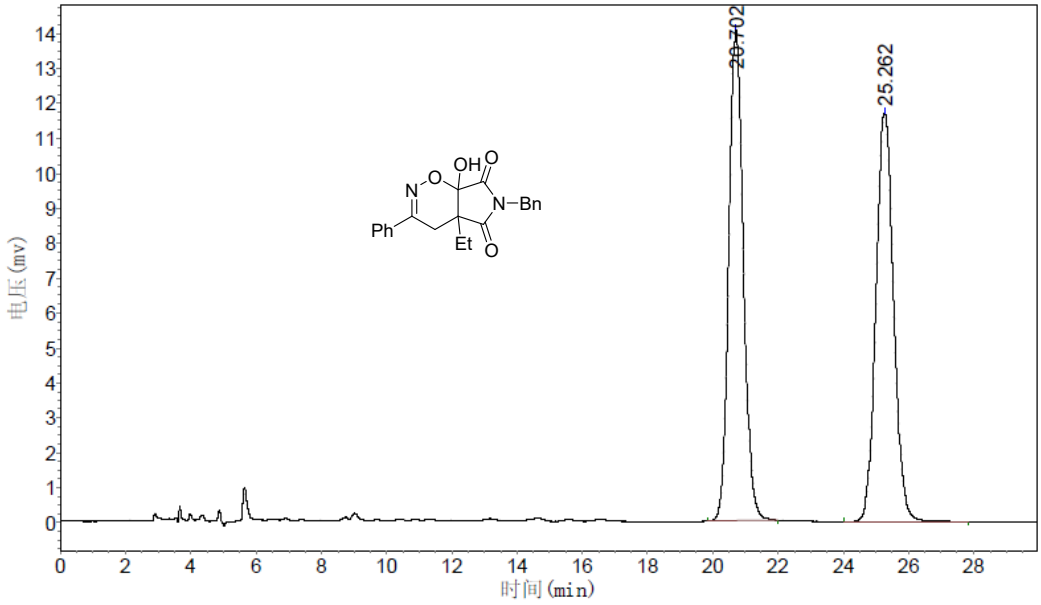
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.155	9169.237	225605.906	49.8334
2	21.218	6961.228	227114.594	50.1666



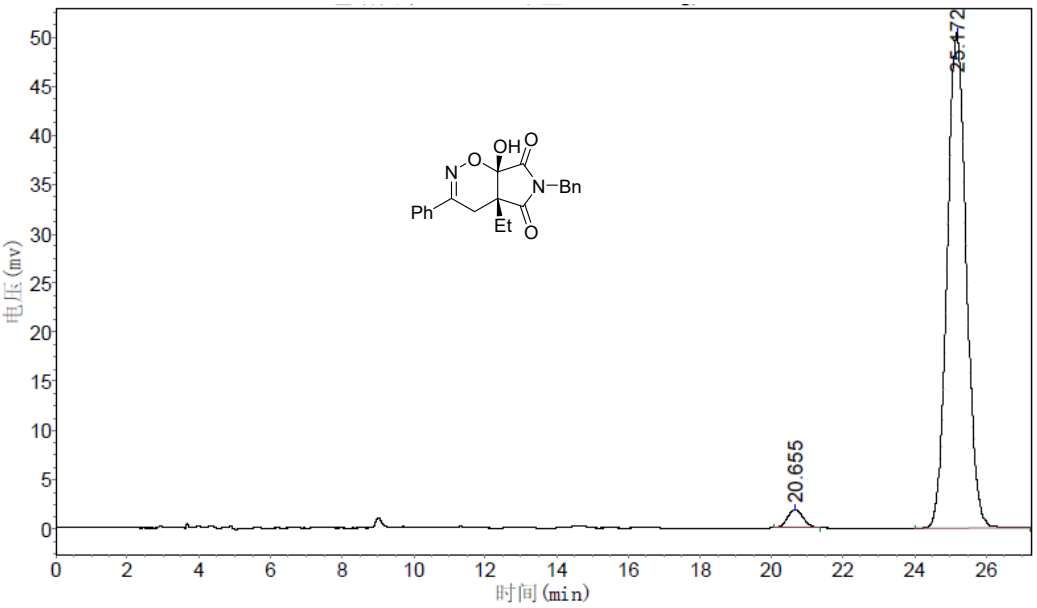
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.135	47616.211	1157612.000	97.0603
2	21.123	930.371	35060.375	2.9396

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} =16 min, t_{minor} = 21 min

3ma



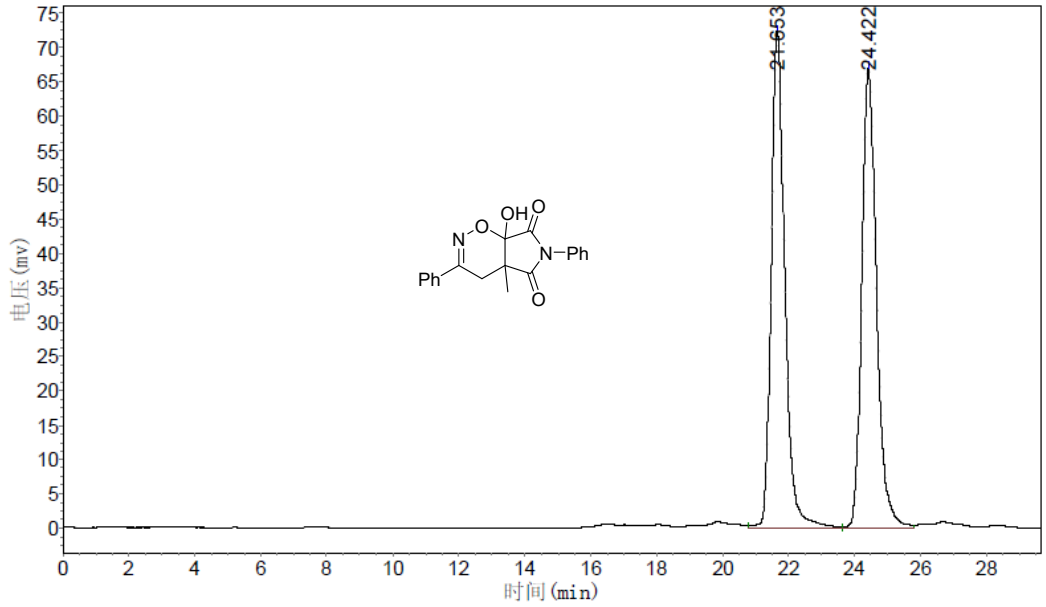
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	20.702	14060.308	435329.594	49.8453
2	25.262	11684.464	438031.688	50.1547



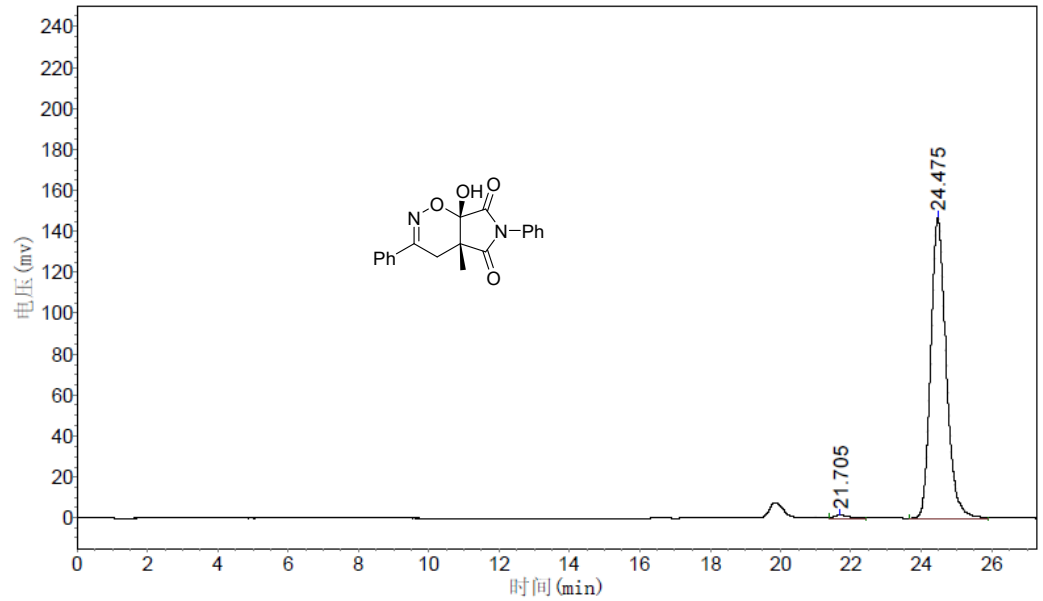
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	20.655	1677.673	44997.938	2.3719
2	25.172	50170.496	1852096.000	97.6281

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} =20 min, t_{major} = 25 min

3na



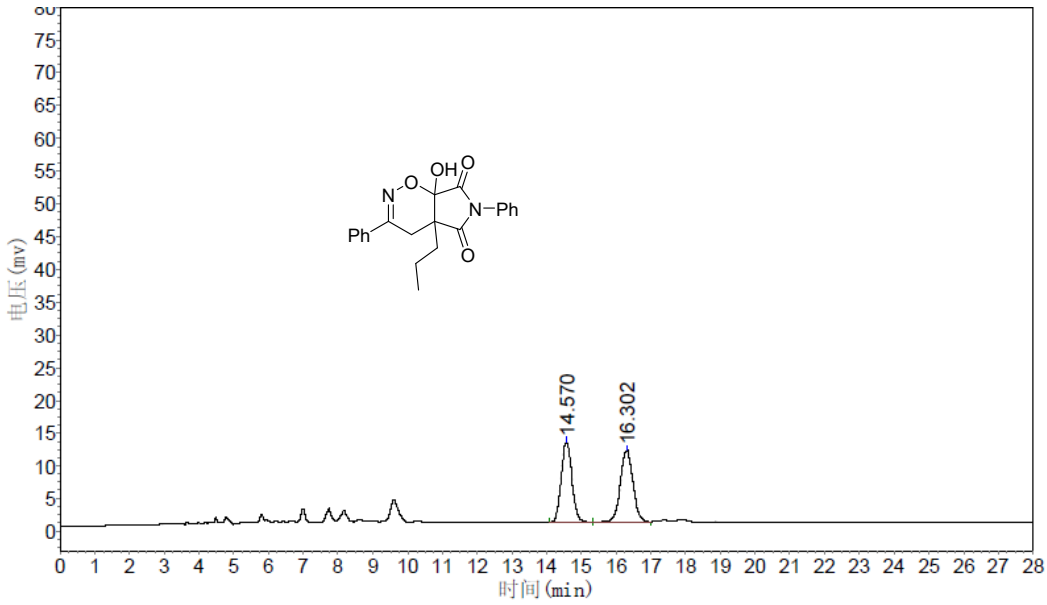
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	21.653	72252.688	2048823.875	49.7021
2	24.422	67011.359	2073383.750	50.2979



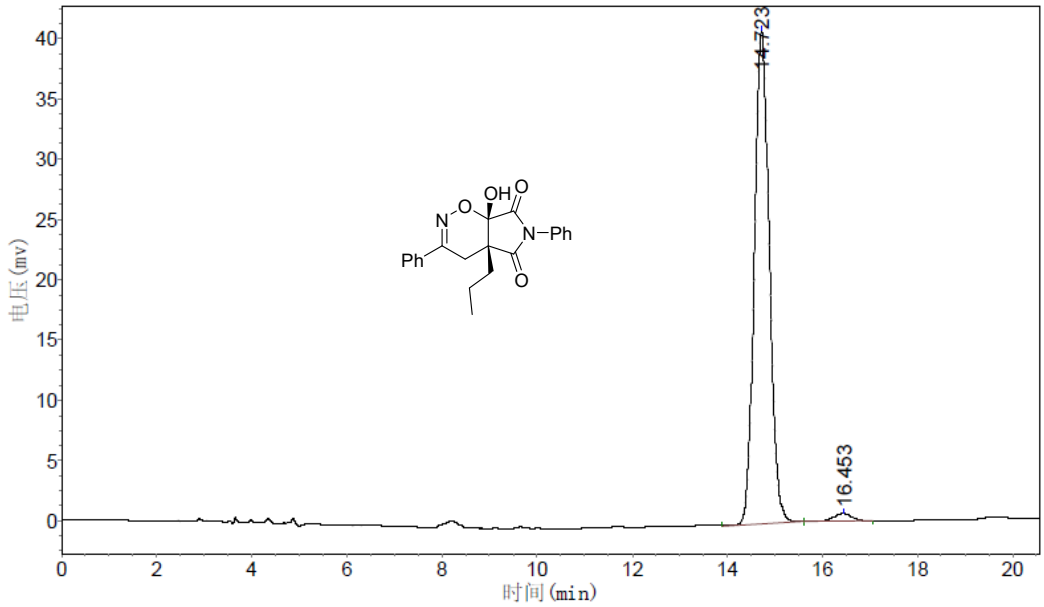
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	21.705	1283.919	36266.129	0.8015
2	24.475	146443.328	4457390.000	98.5066

Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} =21 min, t_{major} = 24 min.

3oa



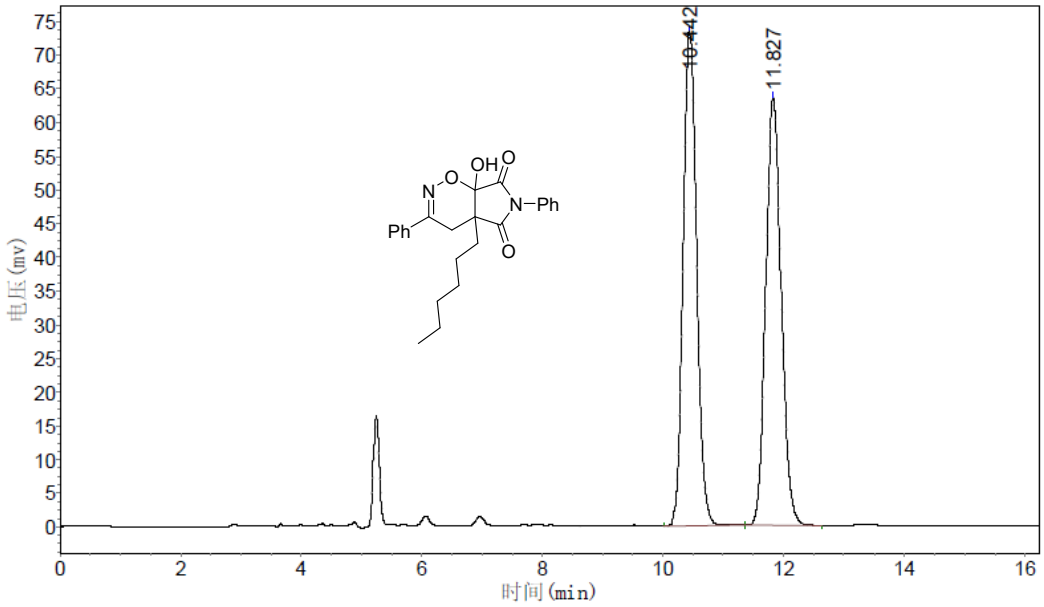
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	14.570	12128.607	261522.797	49.2072
2	16.302	10836.230	269949.438	50.7928



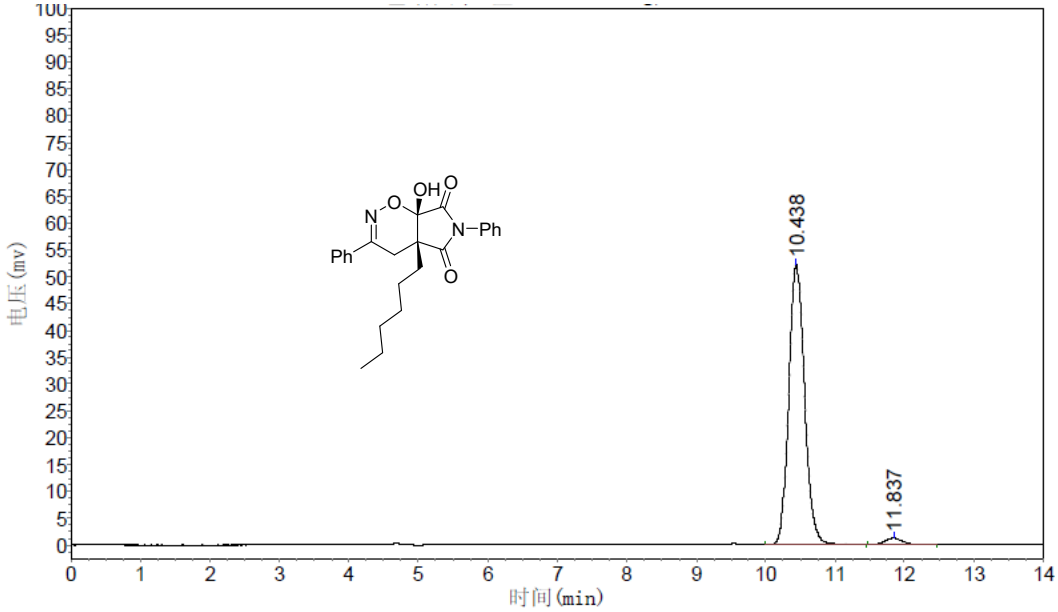
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	14.723	40848.457	890202.313	98.2208
2	16.453	602.189	16124.916	1.7791

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 14 min, t_{minor} = 16 min

3pa



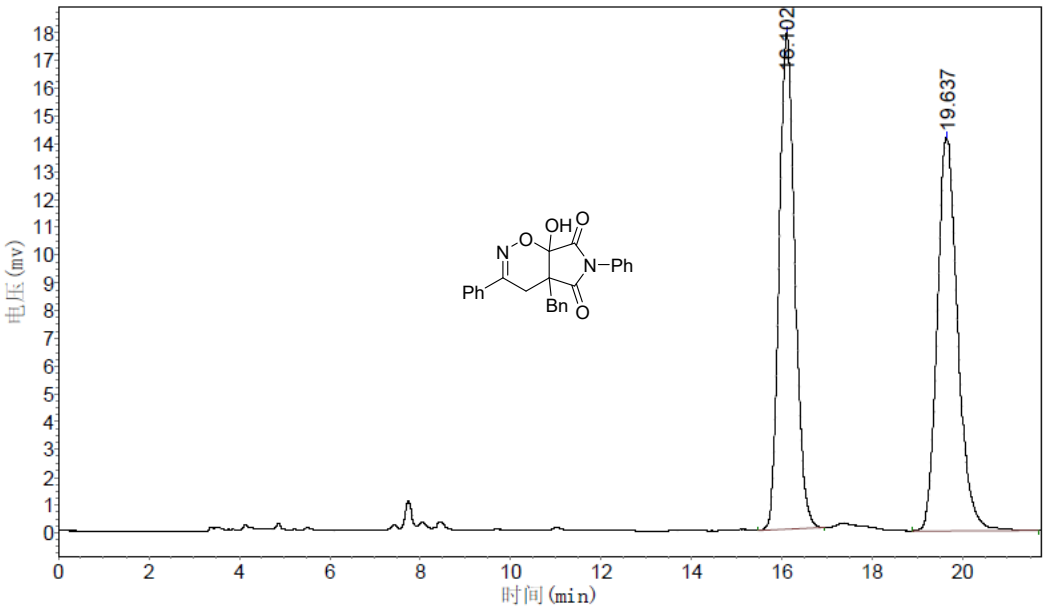
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	10.442	73451.016	1139727.125	49.9140
2	11.827	63522.500	1137818.500	50.0860



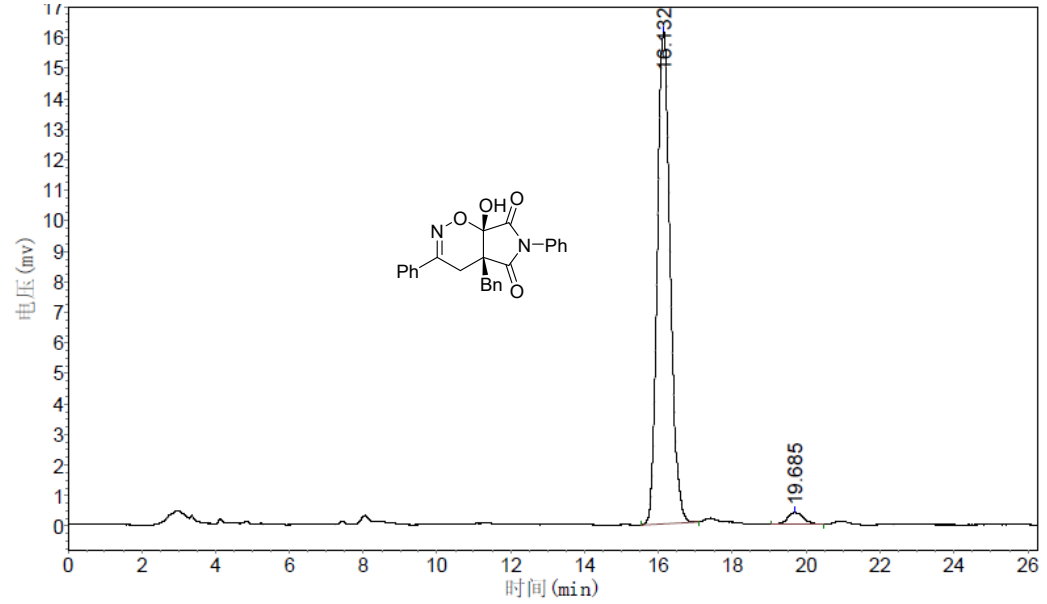
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	10.438	52272.051	828559.438	97.4072
2	11.837	1191.046	22054.381	2.5928

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 10 min, t_{minor} = 11 min

3qa



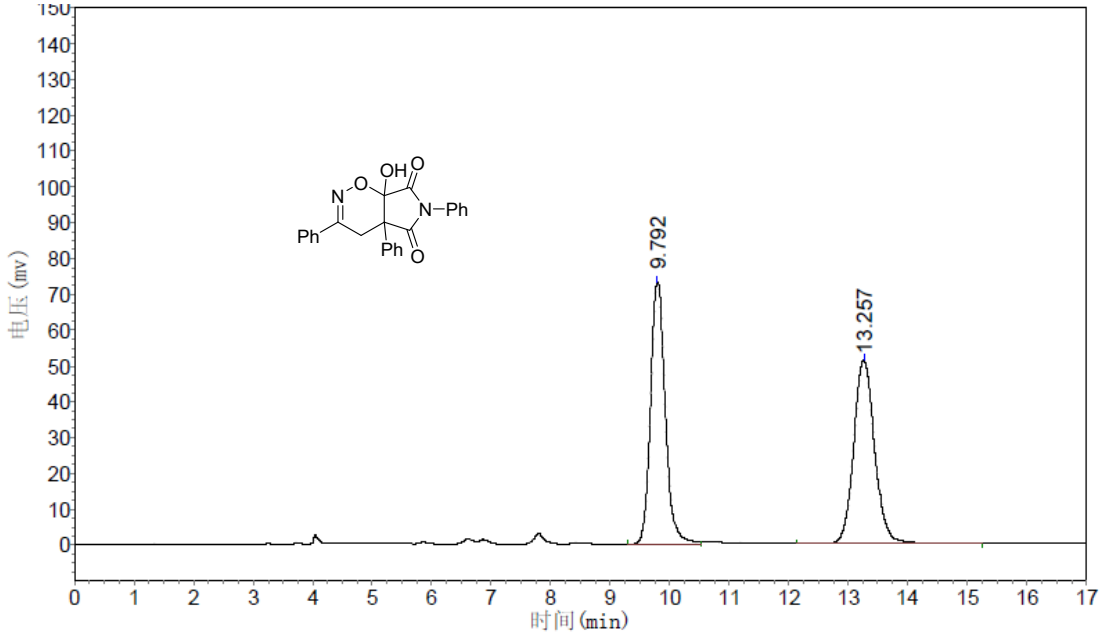
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.102	17908.777	431216.813	49.4127
2	19.637	14155.213	441466.813	50.5873



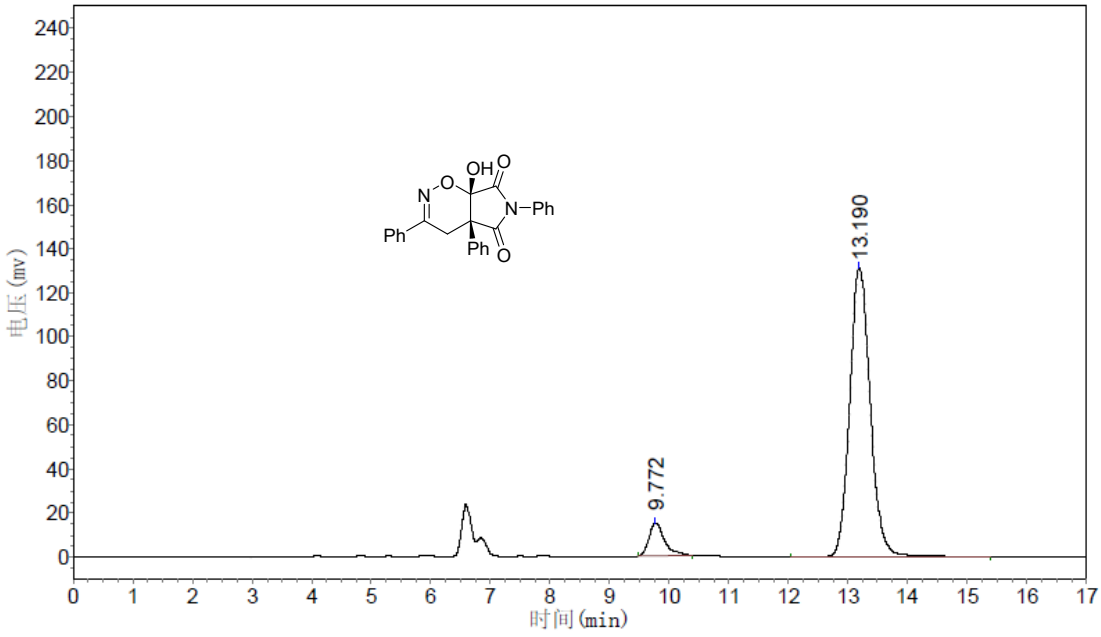
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.132	16121.605	391280.375	97.7128
2	19.685	356.221	9158.818	2.2872

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 16 min, t_{minor} = 19 min

3ra



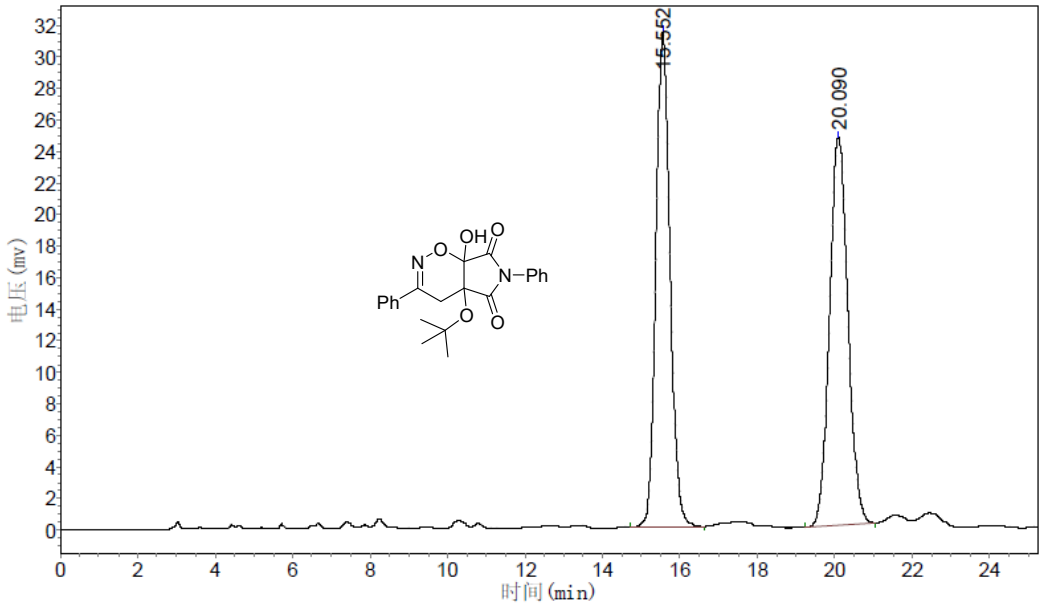
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	9.792	73104.258	1246569.375	49.8132
2	13.257	51301.516	1255917.125	50.1868



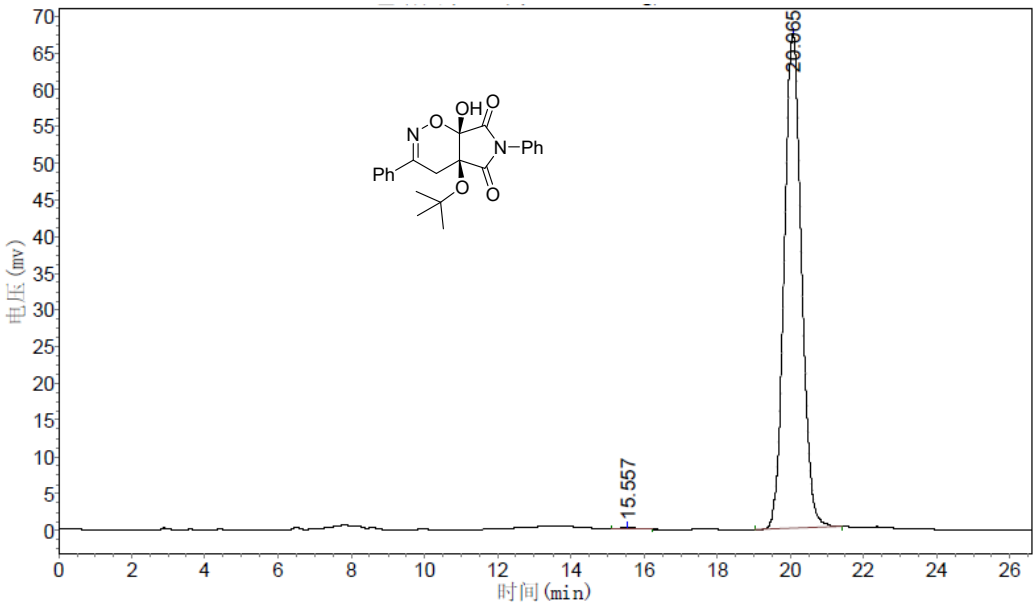
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	9.772	15180.345	285753.531	8.3869
2	13.190	131079.125	3121401.250	91.6131

Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 9 min, t_{major} = 13 min

3sa



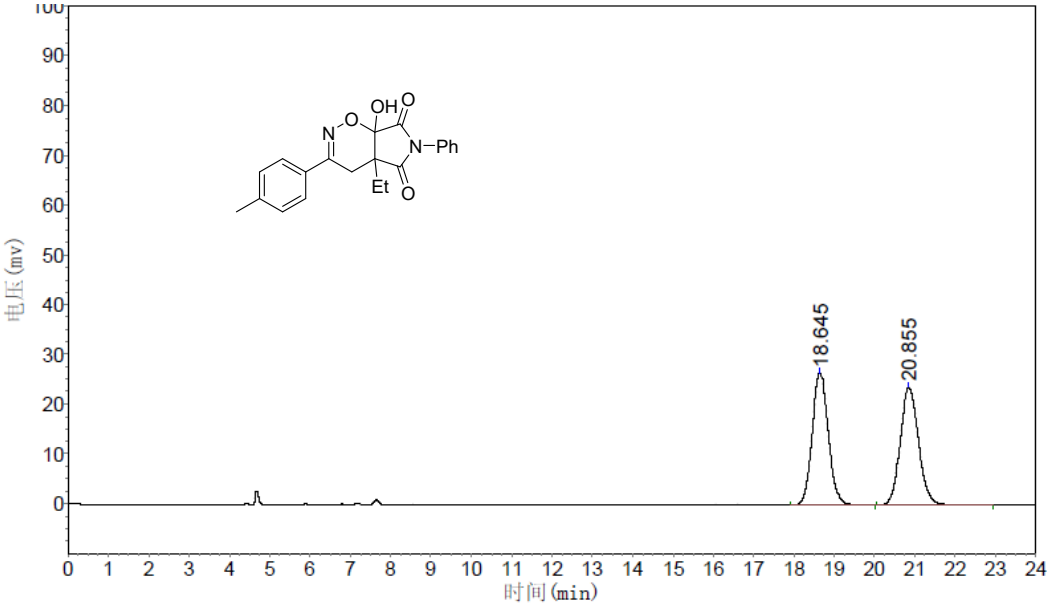
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.552	31398.766	793393.500	49.4143
2	20.090	24620.063	812199.875	50.5857



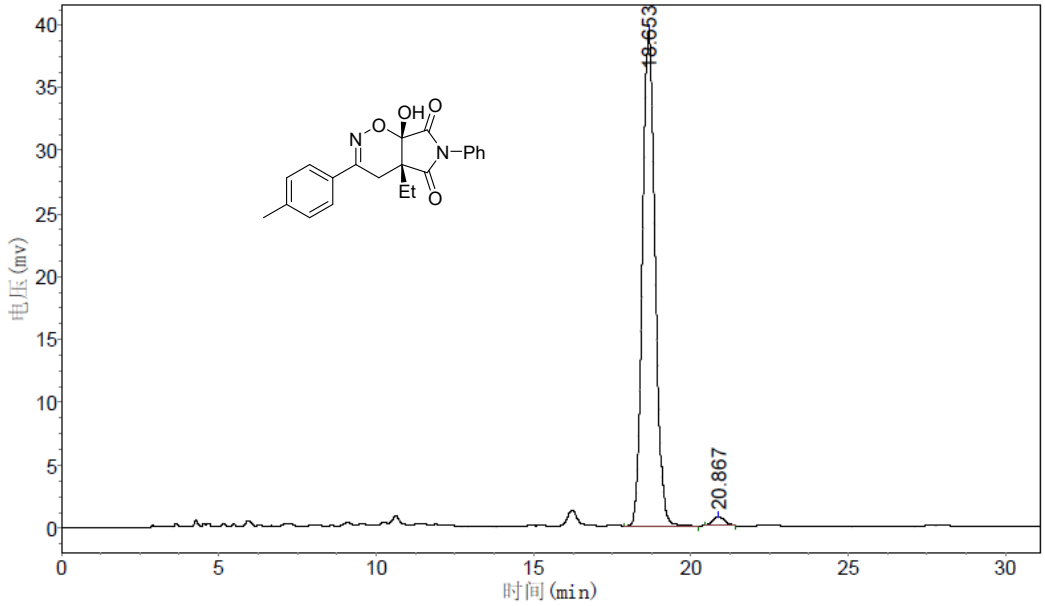
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.557	313.556	7675.550	0.3473
2	20.065	67253.539	2202280.750	99.6527

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 15min, t_{major} = 20 min

3ab



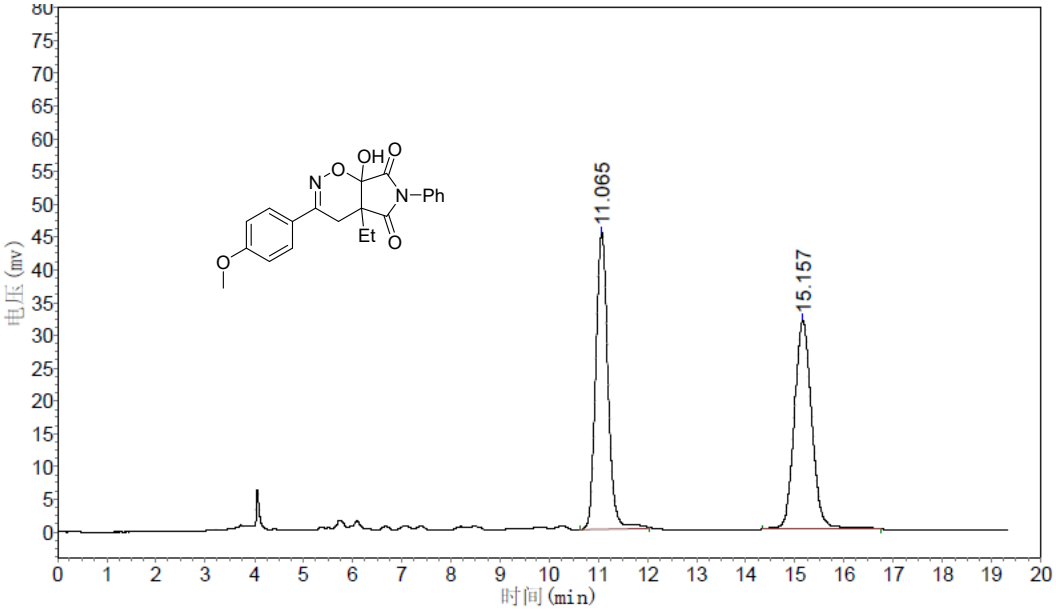
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	18.645	26470.850	737368.875	50.1576
2	20.855	23466.025	732733.875	49.8424



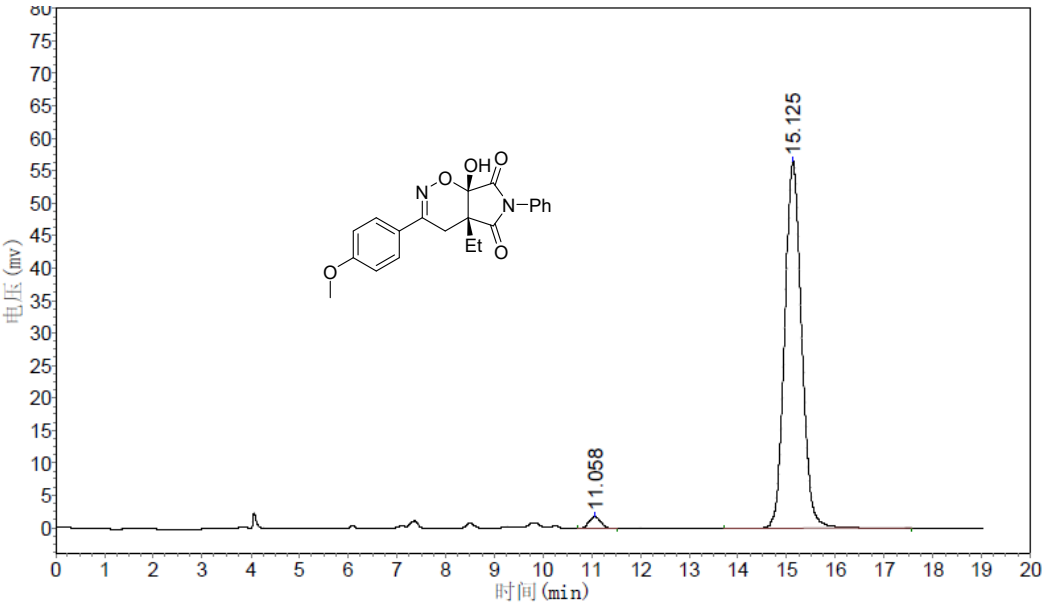
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	18.653	39438.063	1098108.125	98.5491
2	20.867	624.001	16167.441	1.4509

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 18 min, t_{minor} = 20 min

3ac



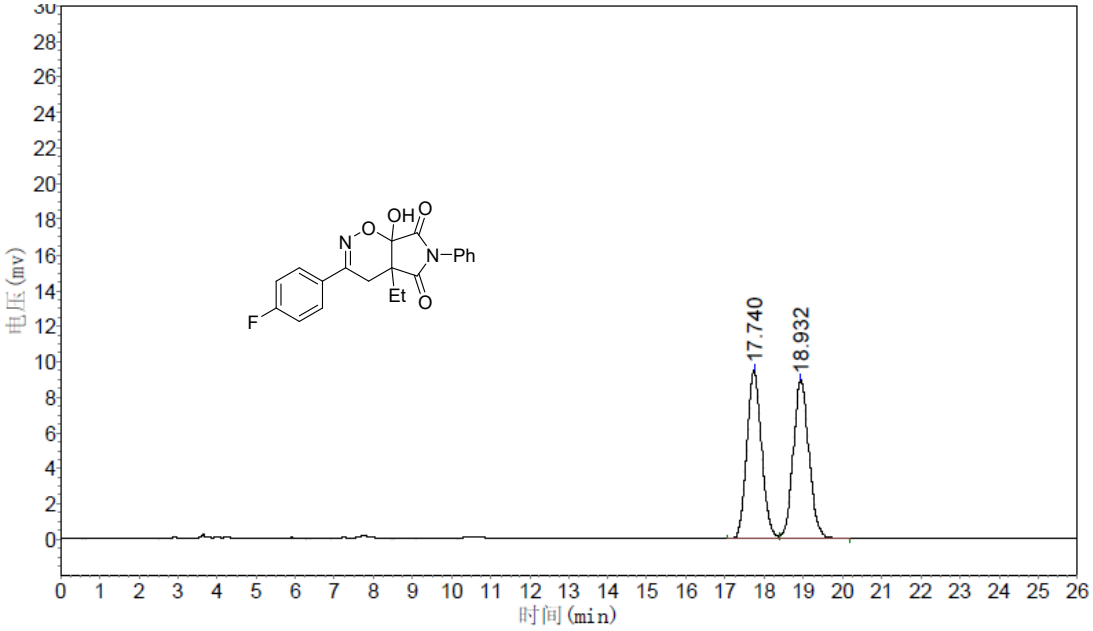
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	11.065	45270.203	774898.125	50.0481
2	15.157	31945.744	773409.125	49.9519



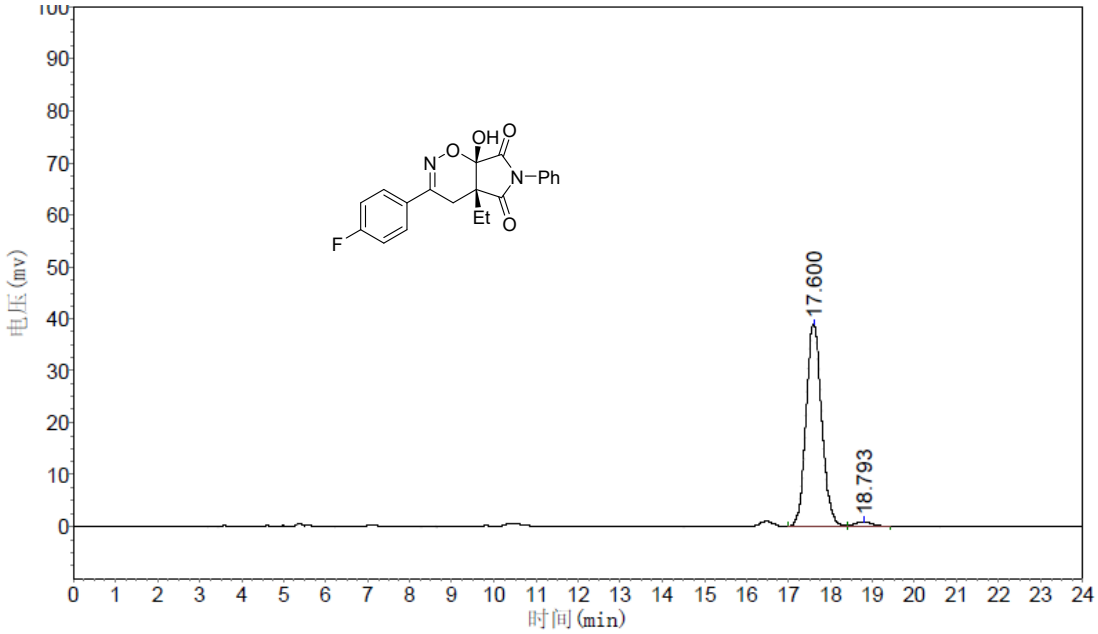
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	11.058	1769.000	30196.000	2.1706
2	15.125	56402.270	1360957.250	97.8294

Chiralpak IC, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 11 min, t_{major} = 15 min

3ad



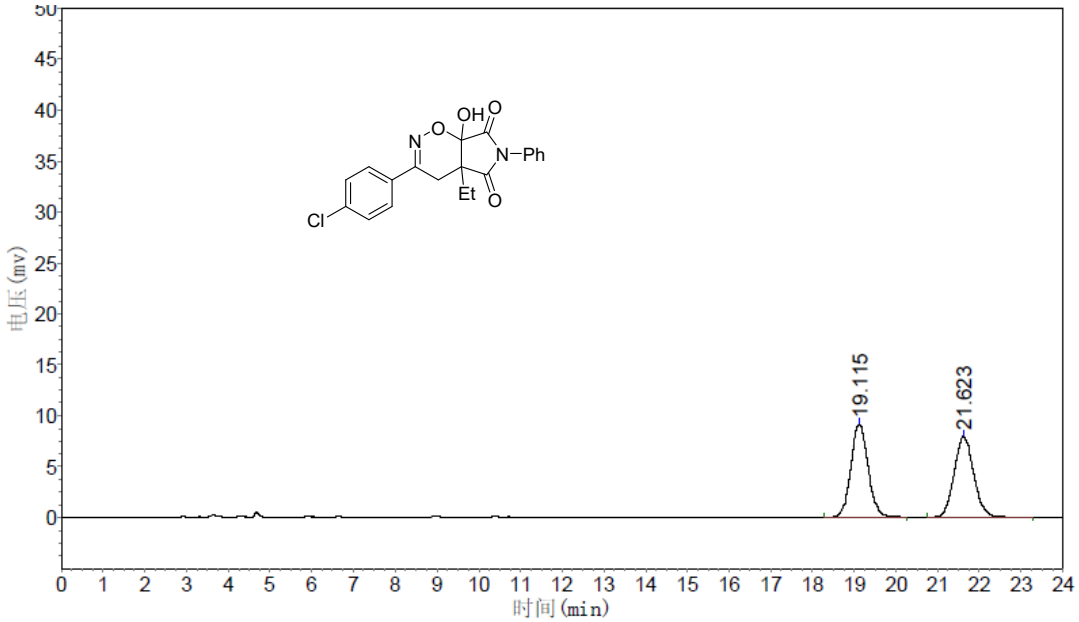
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	17.740	9493.085	247566.344	49.8704
2	18.932	8897.257	248853.469	50.1296



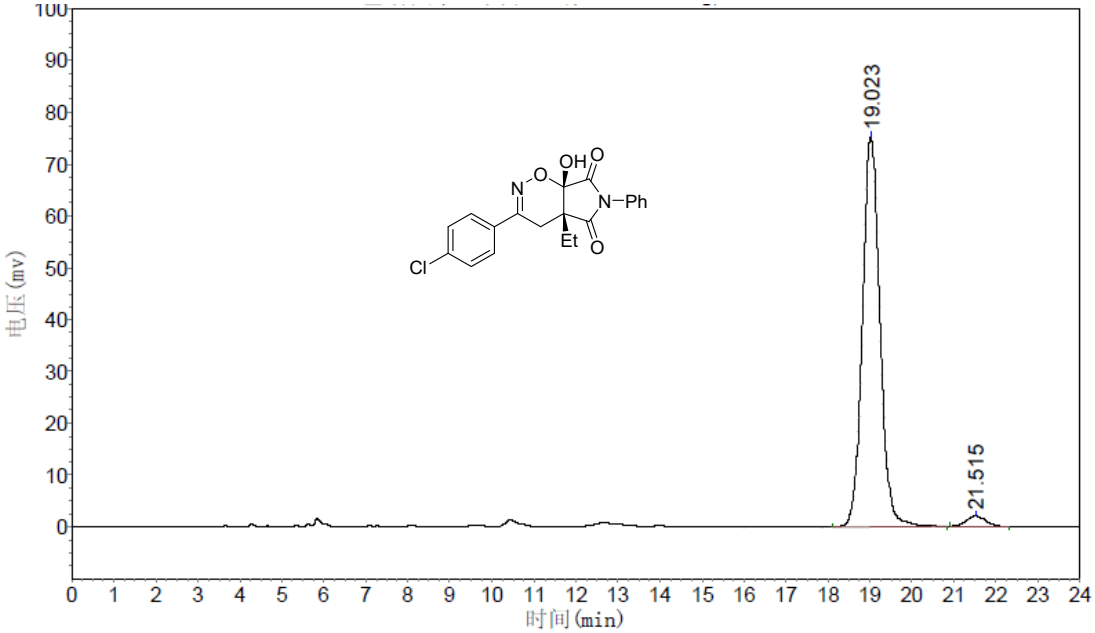
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	17.600	38782.379	1006869.375	97.5698
2	18.793	876.622	25078.043	2.4302

Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 17 min, = 18 min

3ae



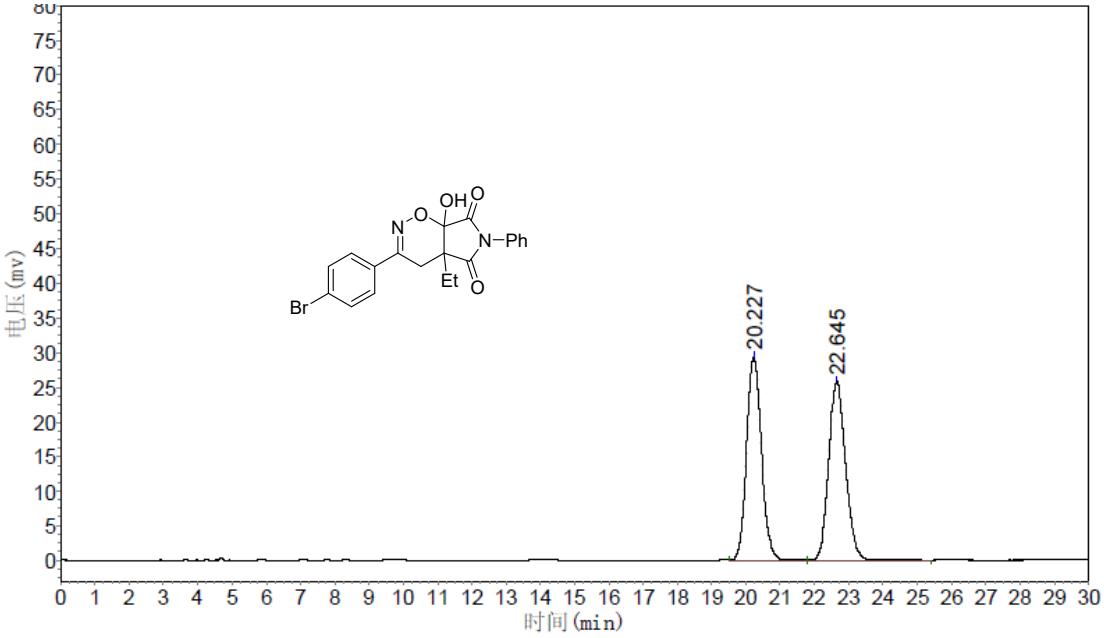
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	19.115	9086.468	260287.656	50.4270
2	21.623	7900.000	255880.000	49.5730



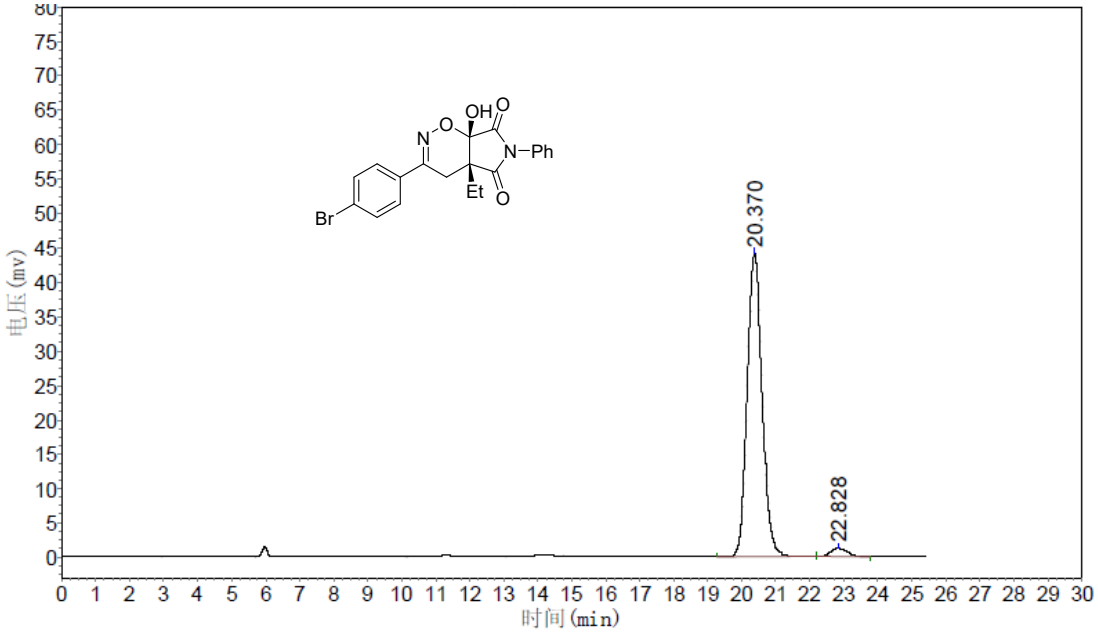
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	19.023	75157.156	2174424.000	97.3020
2	21.515	1908.526	60292.898	2.6980

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 19 min, t_{minor} = 21 min

3af



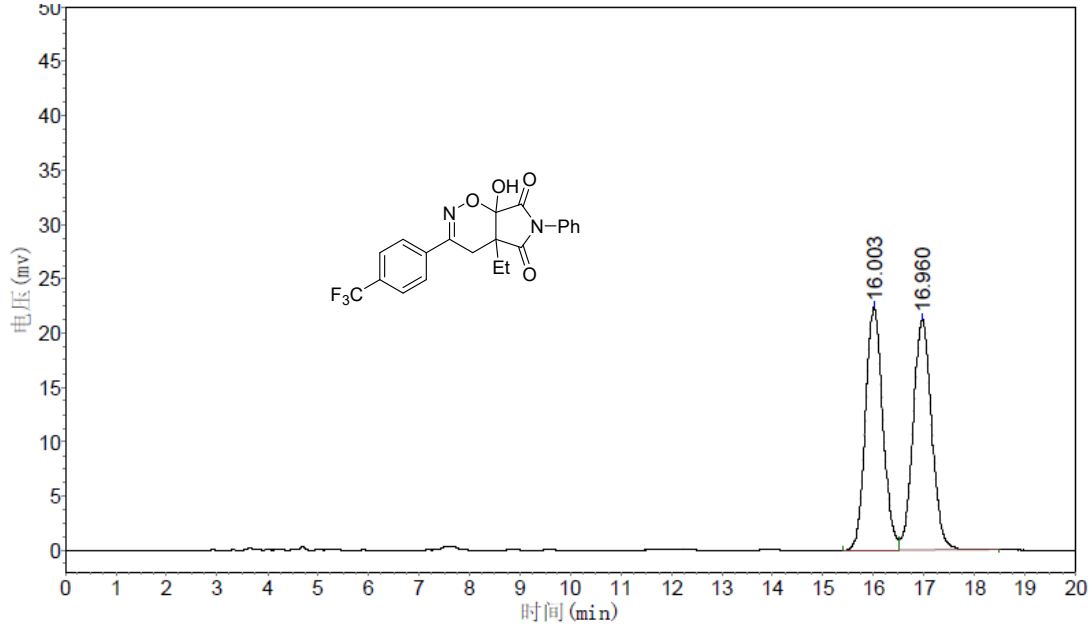
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	20.227	29232.371	882371.813	49.9642
2	22.645	25734.191	883636.375	50.0358



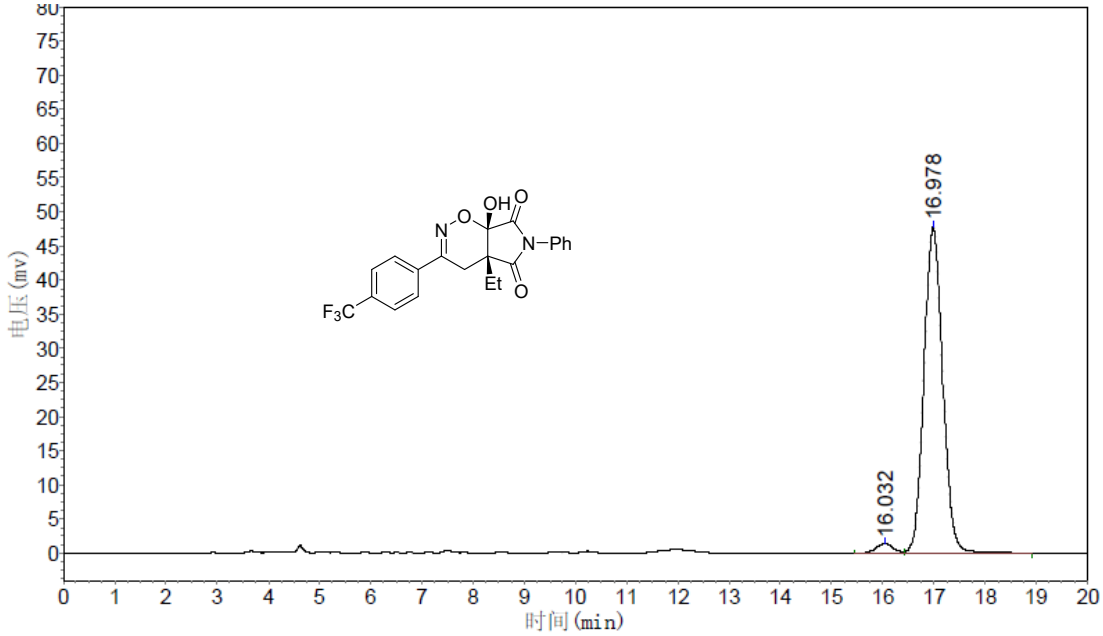
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	20.370	44029.871	1344894.750	97.3948
2	22.828	1046.405	35974.094	2.6052

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 20 min, t_{minor} = 22 min

3ag



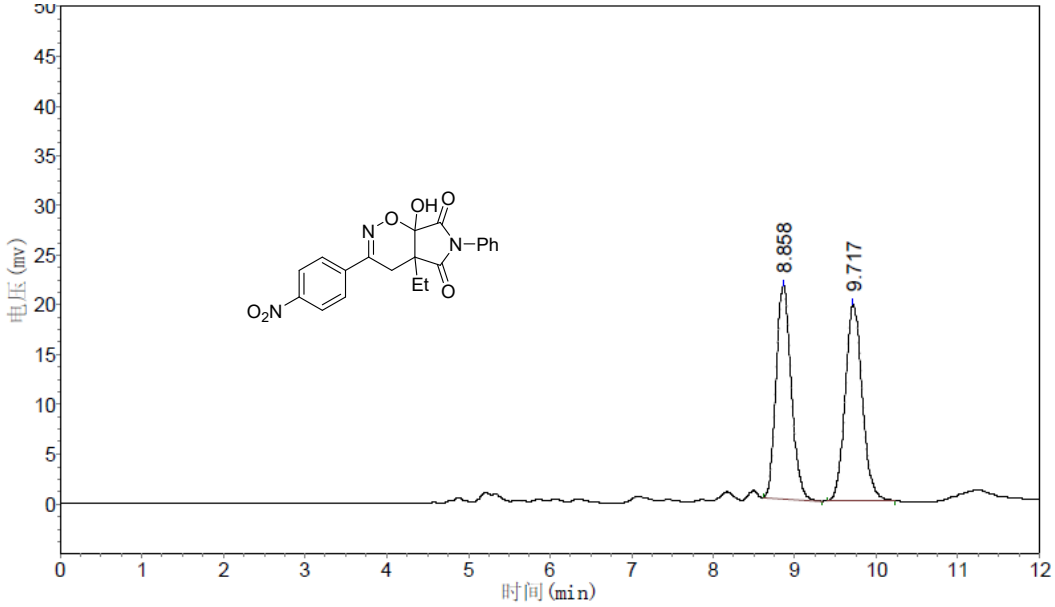
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.003	22316.473	530962.313	49.6120
2	16.960	21236.707	539268.063	50.3880



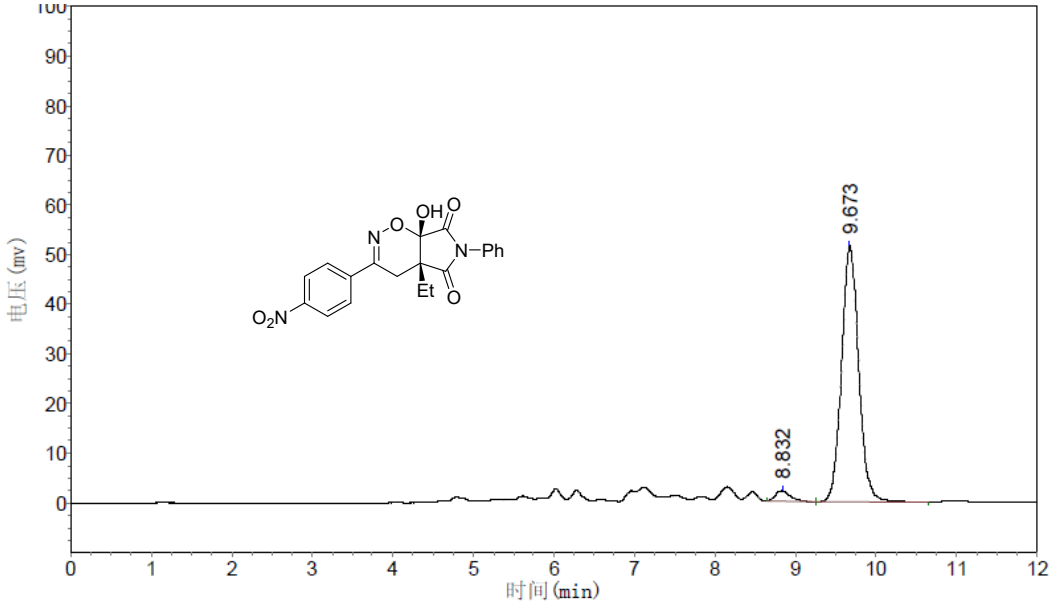
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.032	1412.961	33307.188	2.6622
2	16.978	47826.738	1217798.750	97.3378

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 16 min, t_{major} = 17 min

3ah



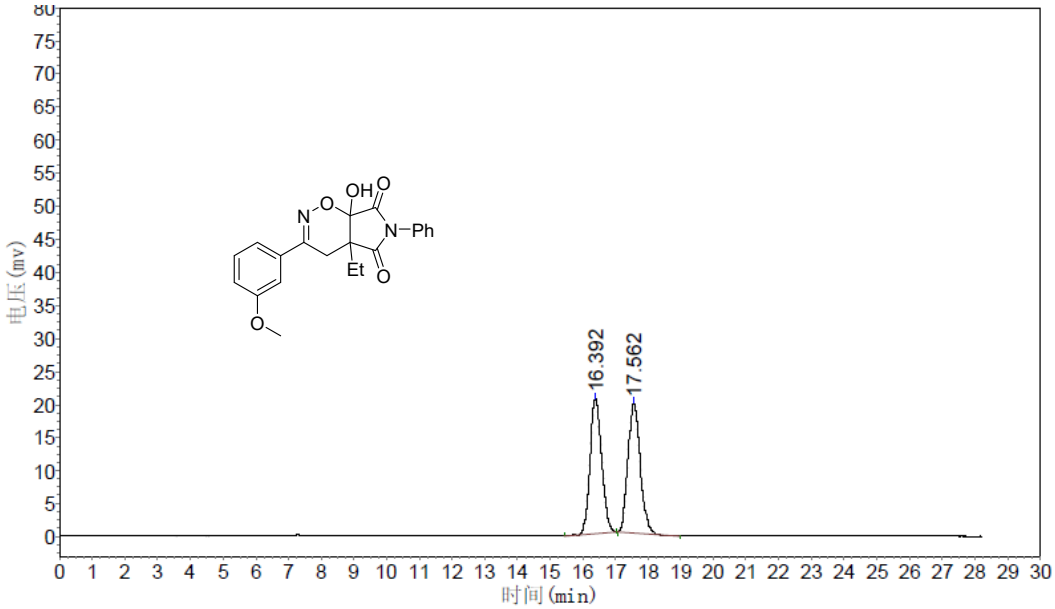
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	8.858	21475.223	277353.344	48.9156
2	9.717	19766.707	289650.906	51.0844



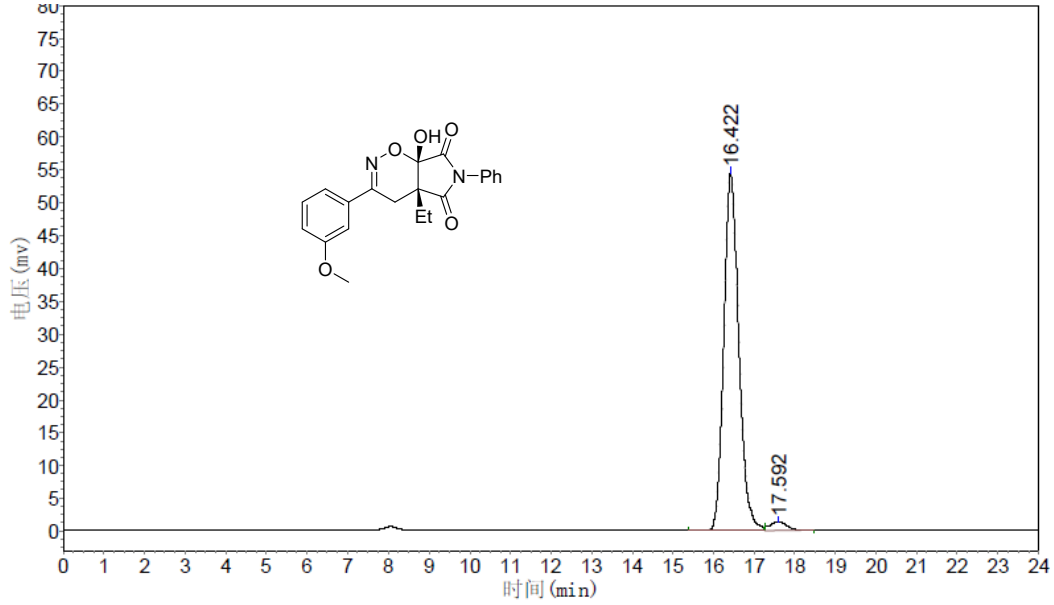
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	8.832	2048.630	26801.600	3.3951
2	9.673	51449.855	762610.375	96.6049

Chiralpak IC, n-hexane/isopropanol = 50/50, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 8 min, t_{major} = 9 min

3ai



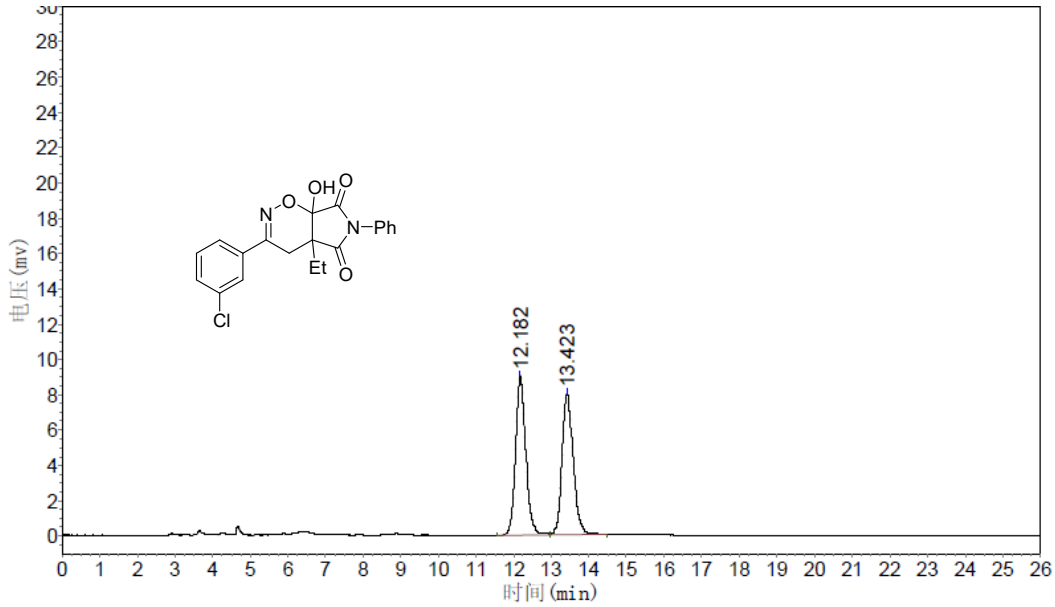
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.392	20801.037	536615.125	49.4914
2	17.562	20134.582	547644.188	50.5086



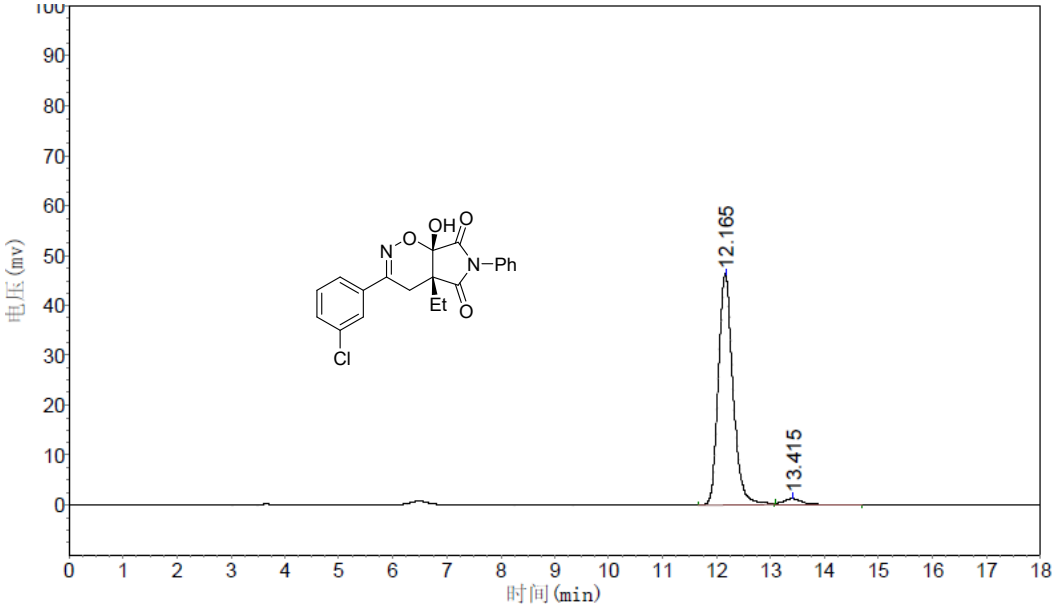
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.422	54378.328	1393724.500	97.5171
2	17.592	1203.044	35485.555	2.4829

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, tminor = 16 min, tmajor = 17 min

3aj



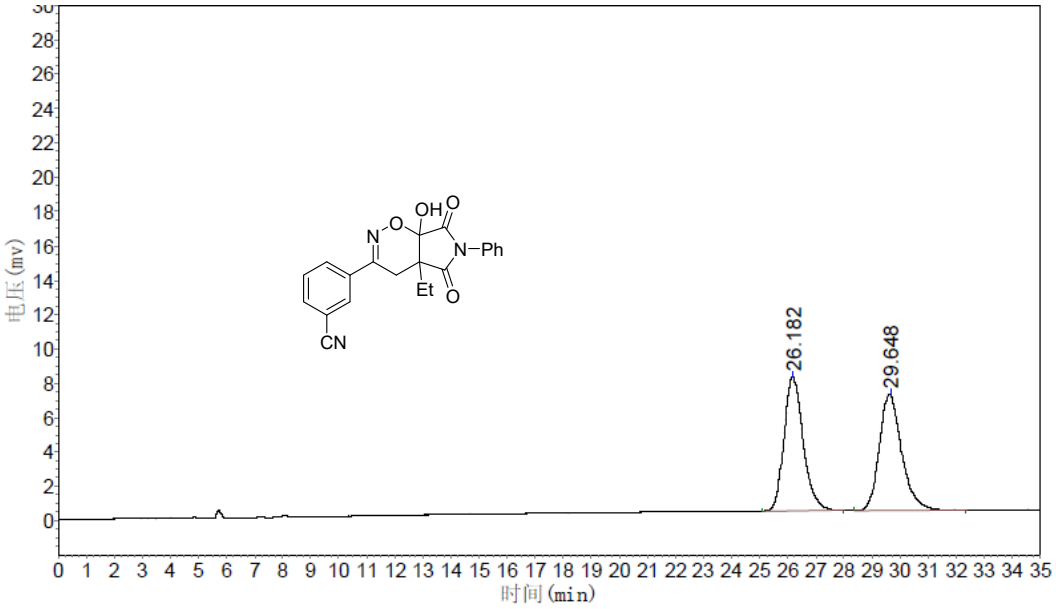
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.182	9020.145	172430.453	50.8485
2	13.423	7974.020	166675.609	49.1515



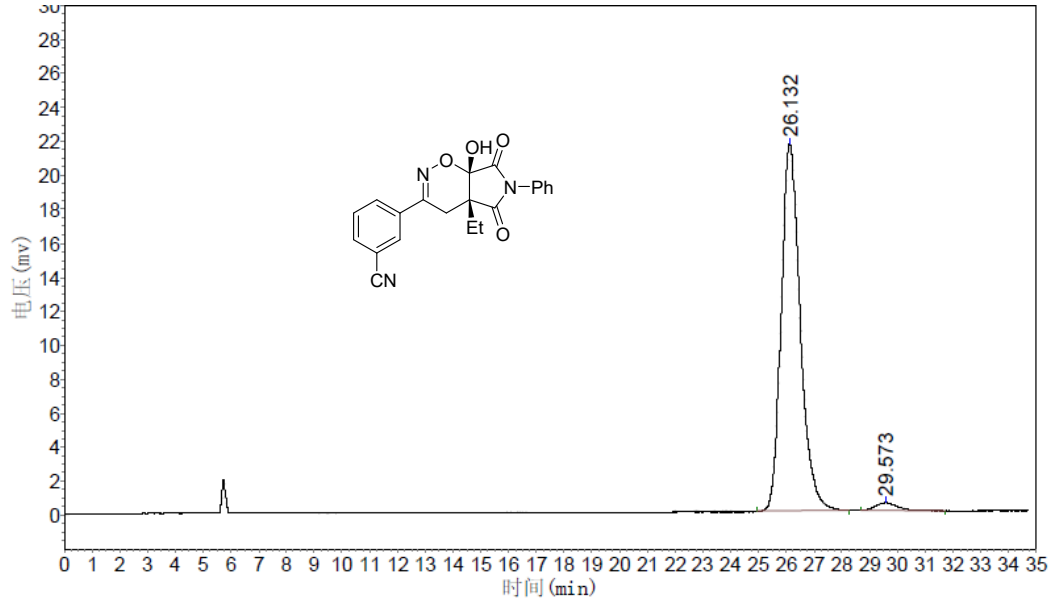
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.165	46104.246	868950.563	96.7914
2	13.415	1152.799	28805.061	3.2086

Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 12 min, t_{minor} = 13 min

3ak



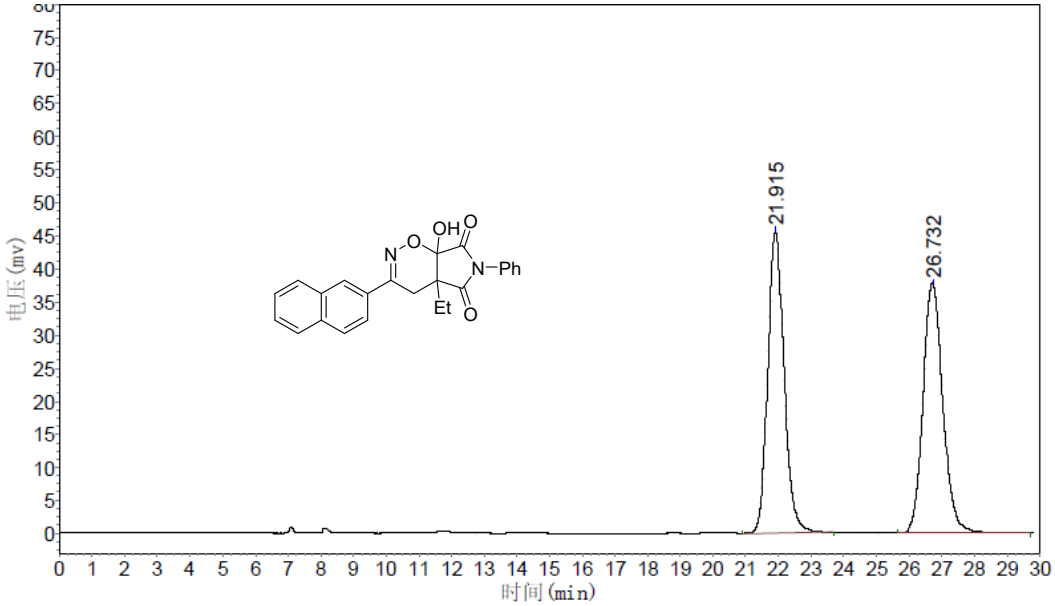
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	26.182	7823.649	373985.000	50.0594
2	29.648	6761.462	373097.000	49.9406



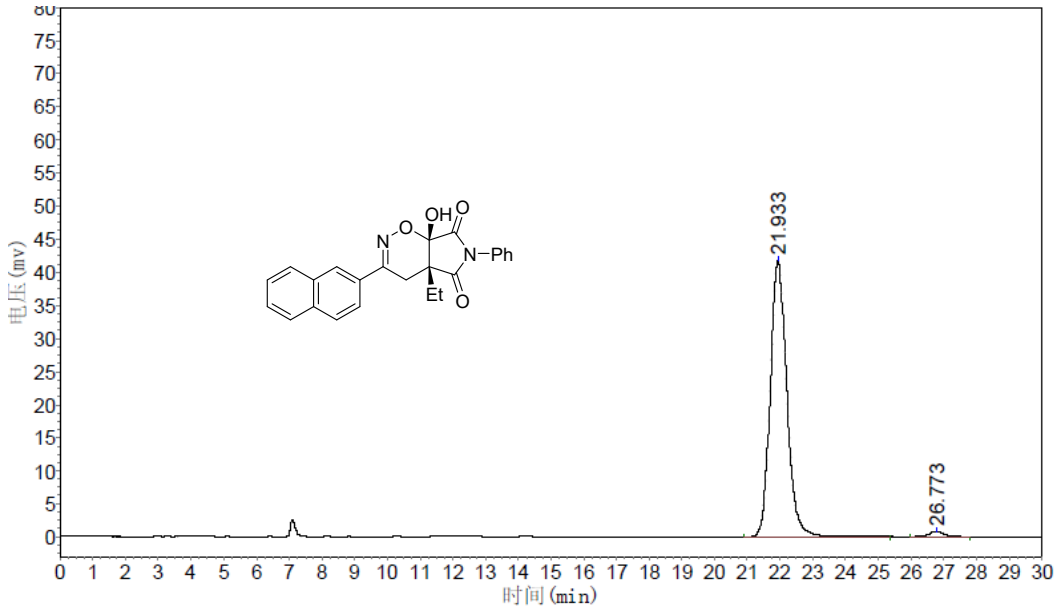
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	26.132	21614.488	1031086.250	97.5946
2	29.573	478.637	25413.500	2.4054

Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 26 min, t_{minor} = 29 min

3al



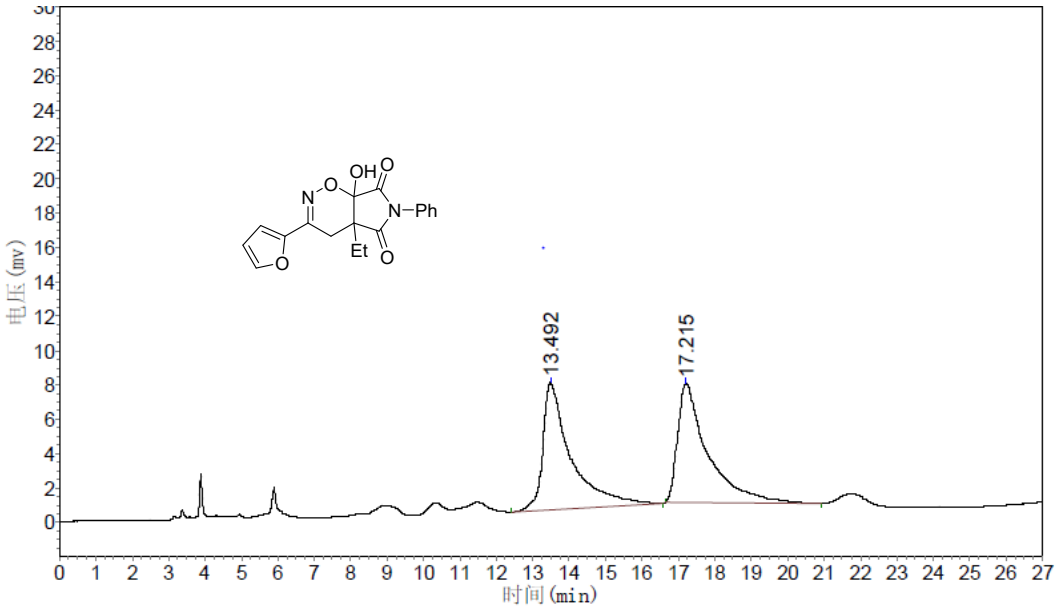
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	21.915	45490.969	1578999.250	49.8418
2	26.732	37647.688	1589025.750	50.1582



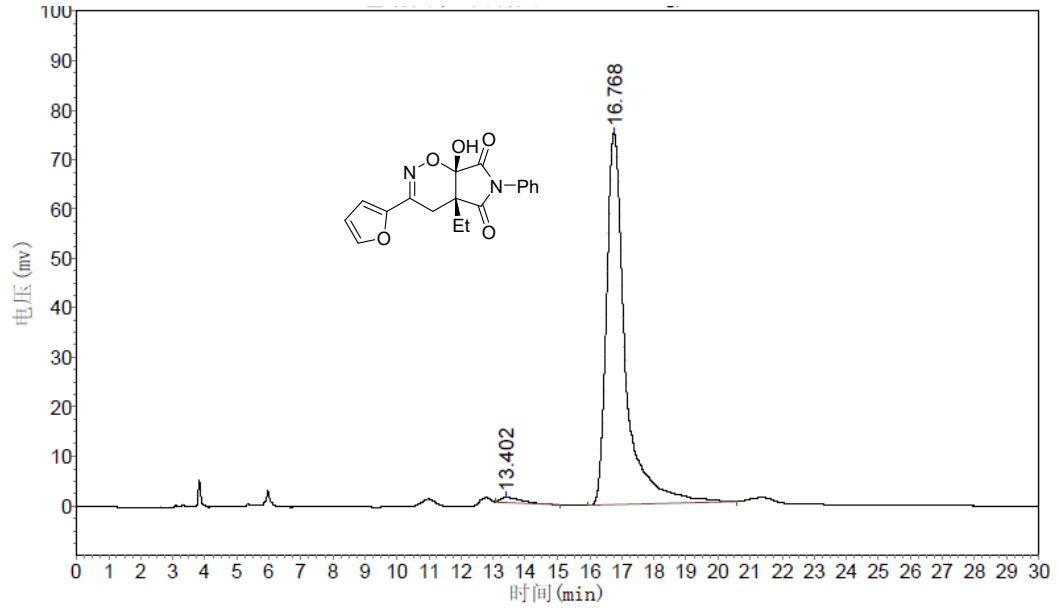
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	21.933	41652.414	1471149.375	98.0886
2	26.773	698.556	28668.031	1.9114

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 22 min, t_{minor} = 26 min

3am



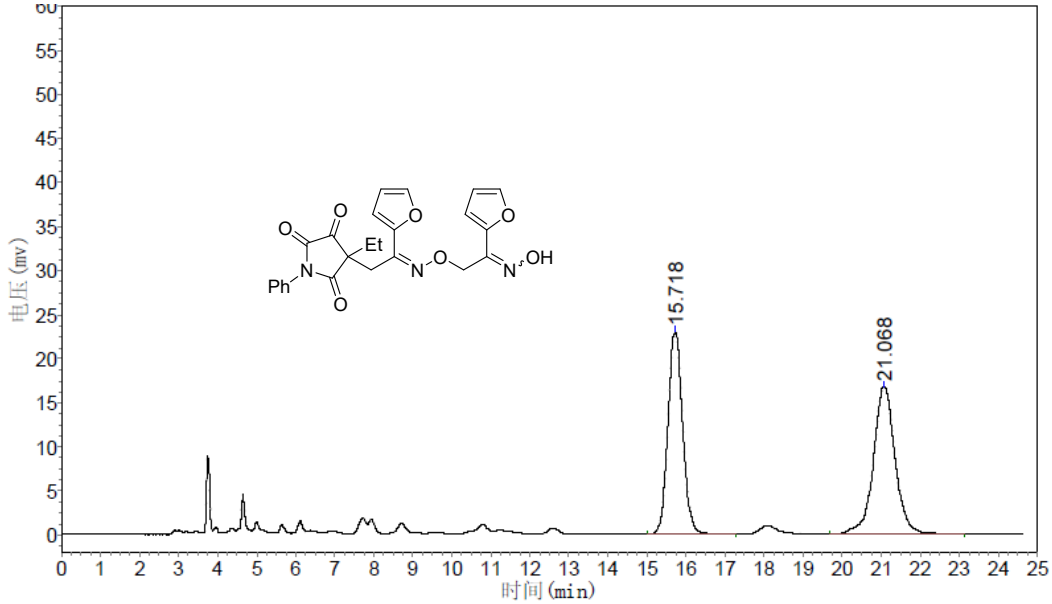
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.492	7505.857	429690.031	49.3515
2	17.215	7199.009	440981.813	50.6484



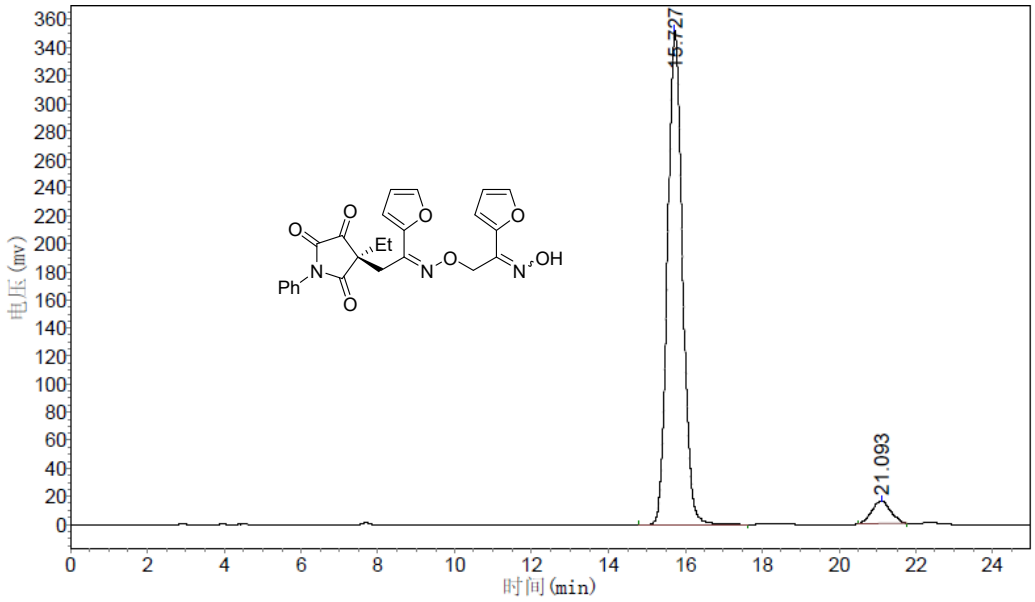
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.402	1123.190	49357.797	1.4926
2	16.768	75325.406	3257410.750	98.5074

Chiralpak IC, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 13 min, t_{major} = 16 min

3am'



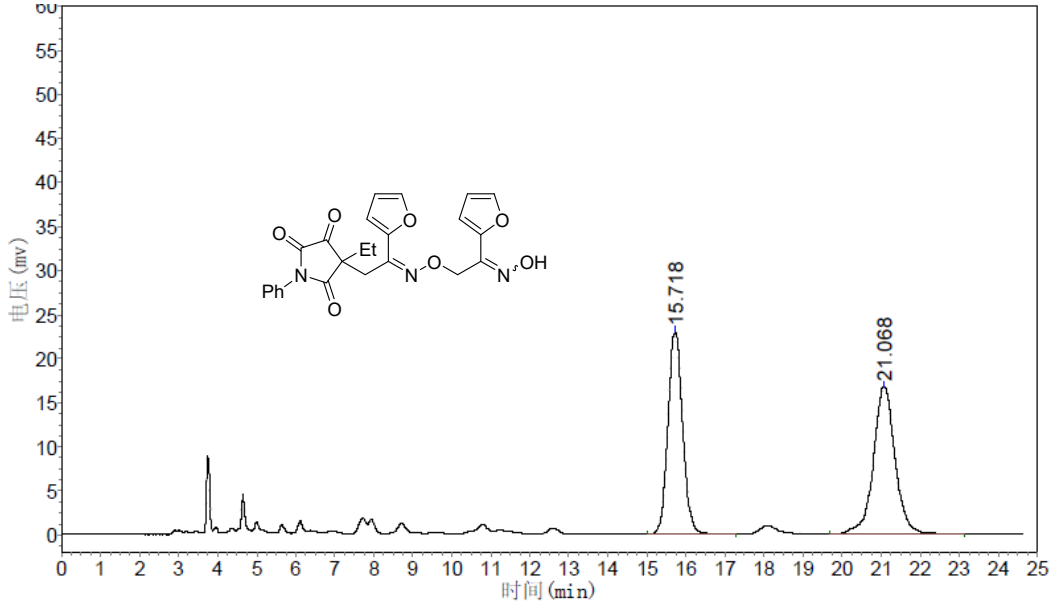
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.718	22908.596	593093.500	49.2011
2	21.068	16486.066	612353.688	50.7989



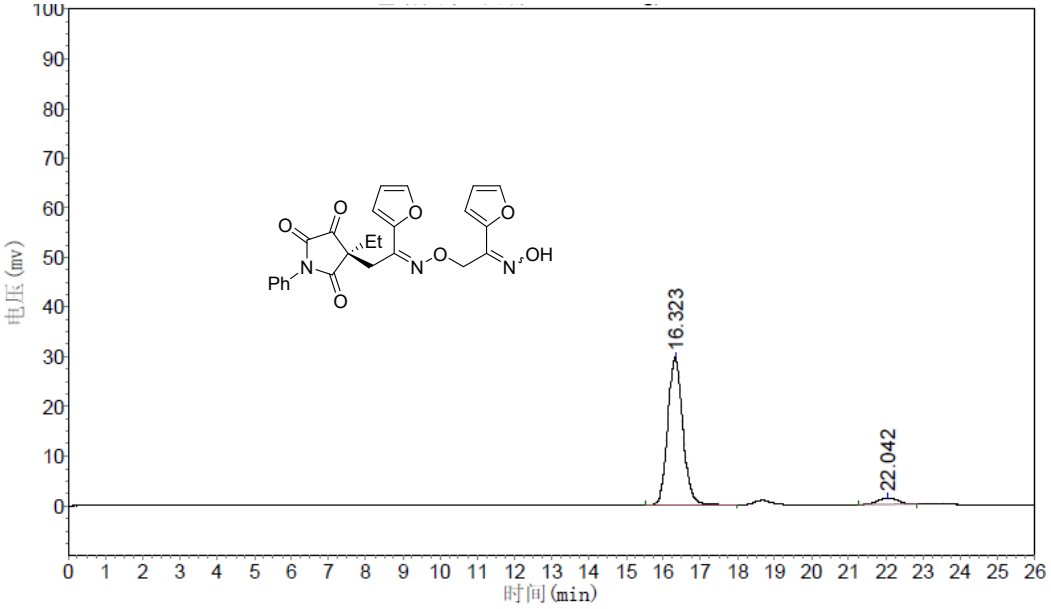
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.727	351788.094	9526318.000	94.6968
2	21.093	15538.790	533495.313	5.3032

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 21 min

3am' (2.2eq)



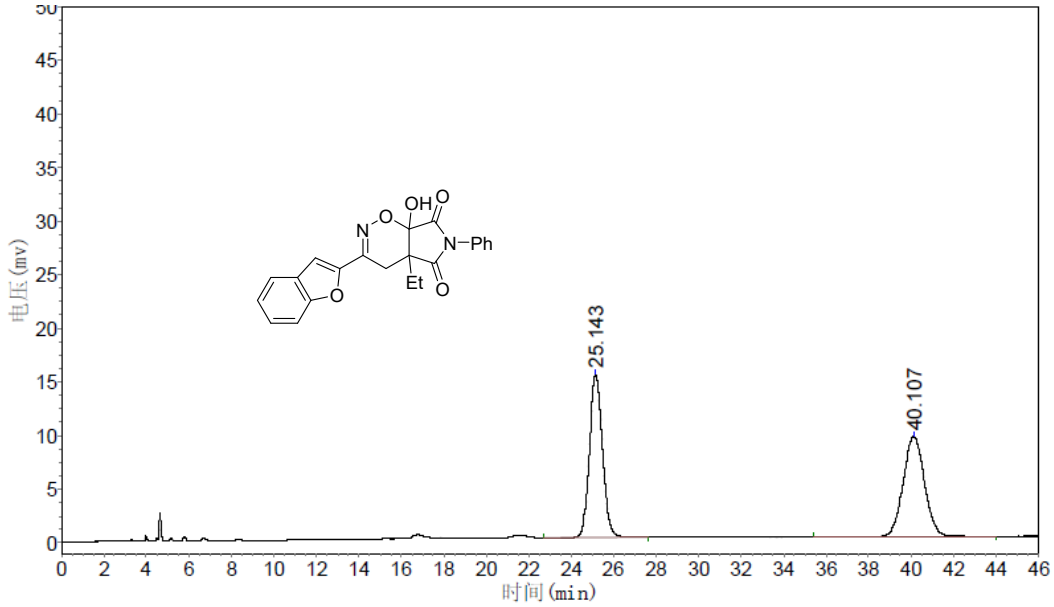
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.718	22908.596	593093.500	49.2011
2	21.068	16486.066	612353.688	50.7989



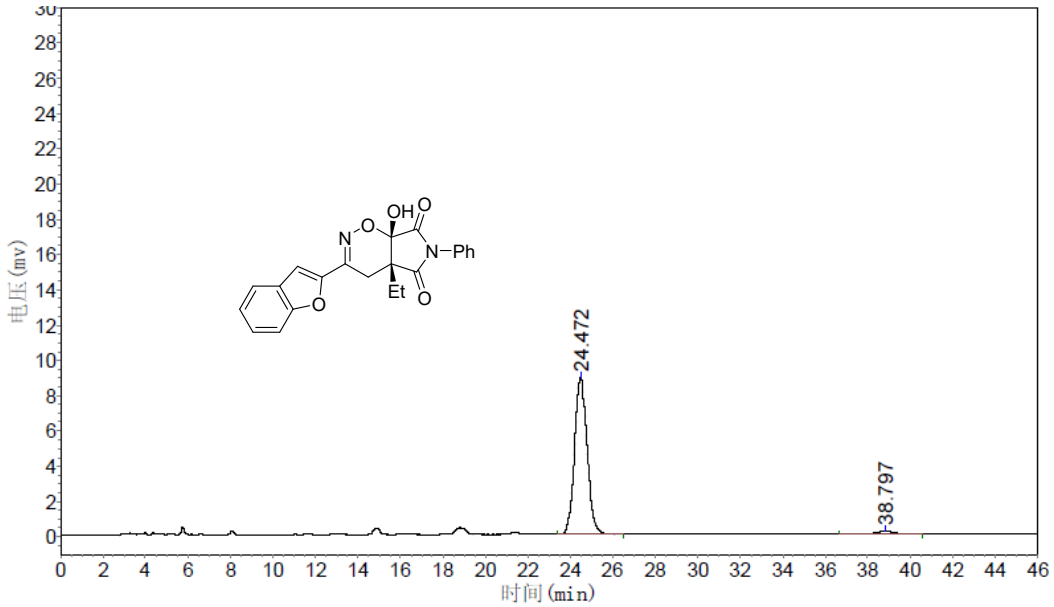
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	16.323	29690.545	834943.250	94.6072
2	22.042	1285.830	47593.207	5.3928

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 16 min, t_{minor} = 22 min

3an



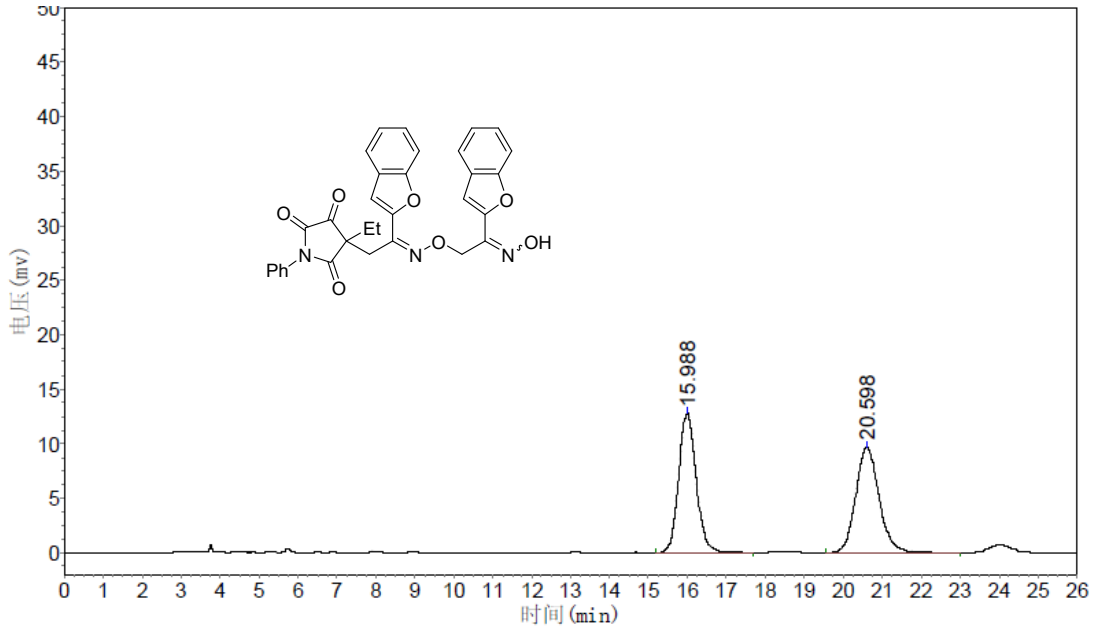
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	25.143	15193.055	646266.688	49.9665
2	40.107	9298.064	647132.313	50.0335



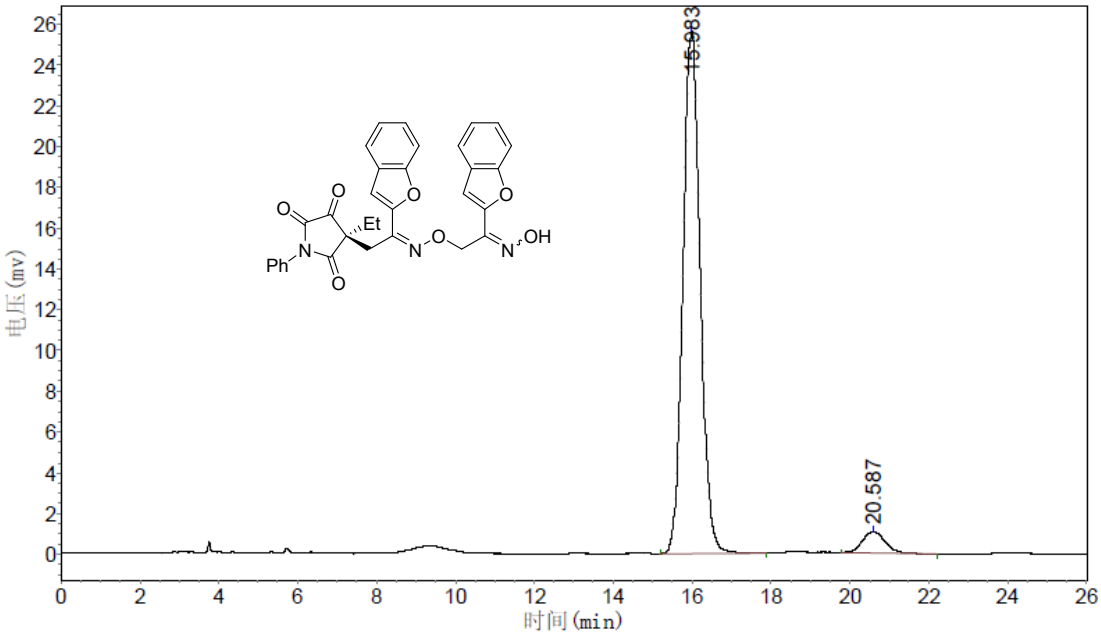
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	24.472	8858.015	367551.094	96.9037
2	38.797	173.653	11744.149	3.0963

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 24 min, t_{minor} = 38 min

3an'



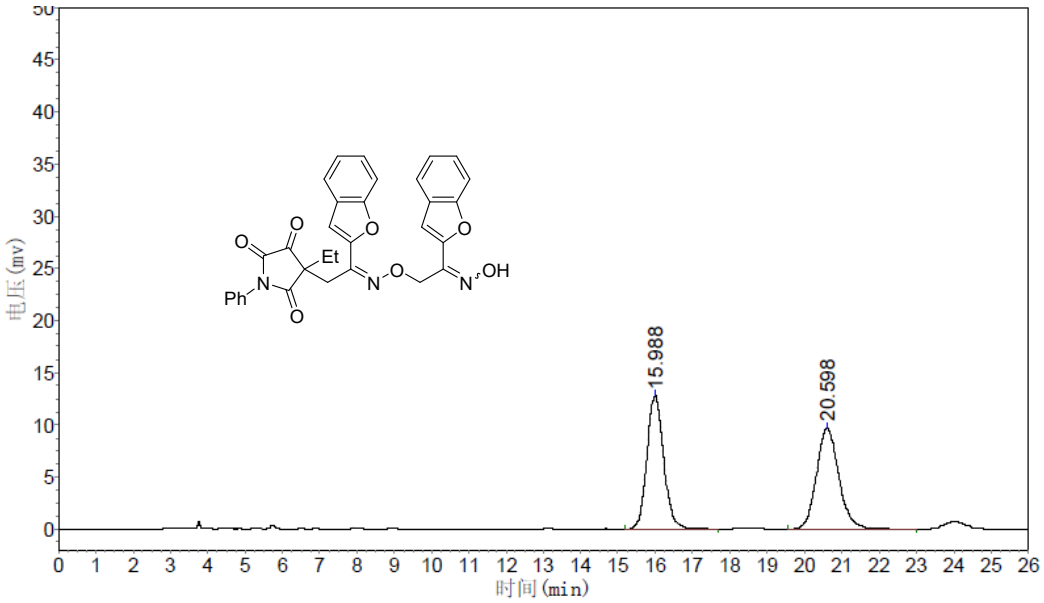
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.988	12740.798	395449.313	49.8862
2	20.598	9663.707	397253.438	50.1138



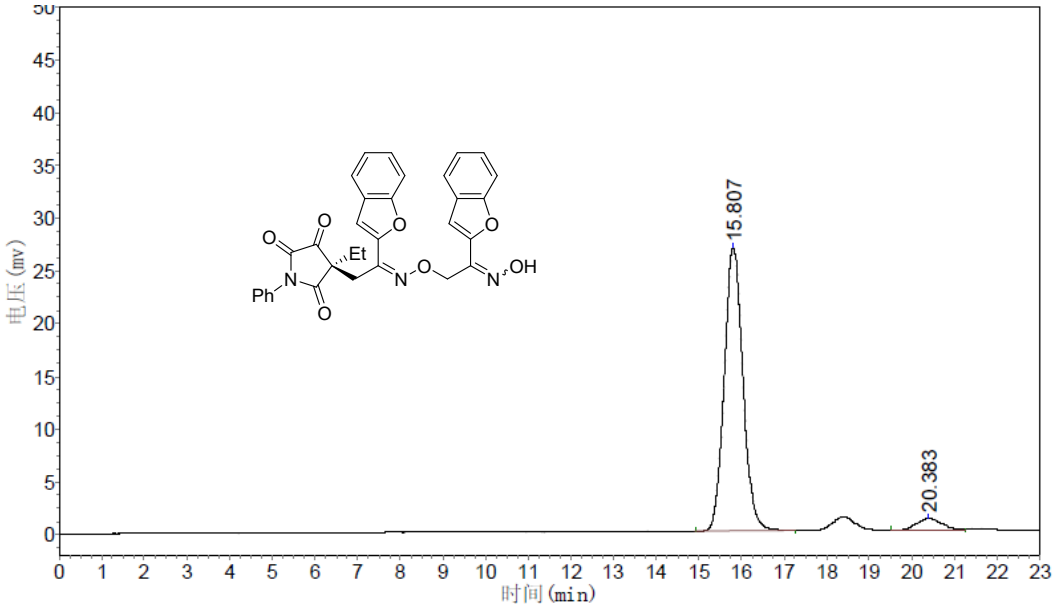
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.983	25609.830	778847.938	95.3503
2	20.587	1018.711	37980.340	4.6497

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 20 min

3an'（2.2eq）



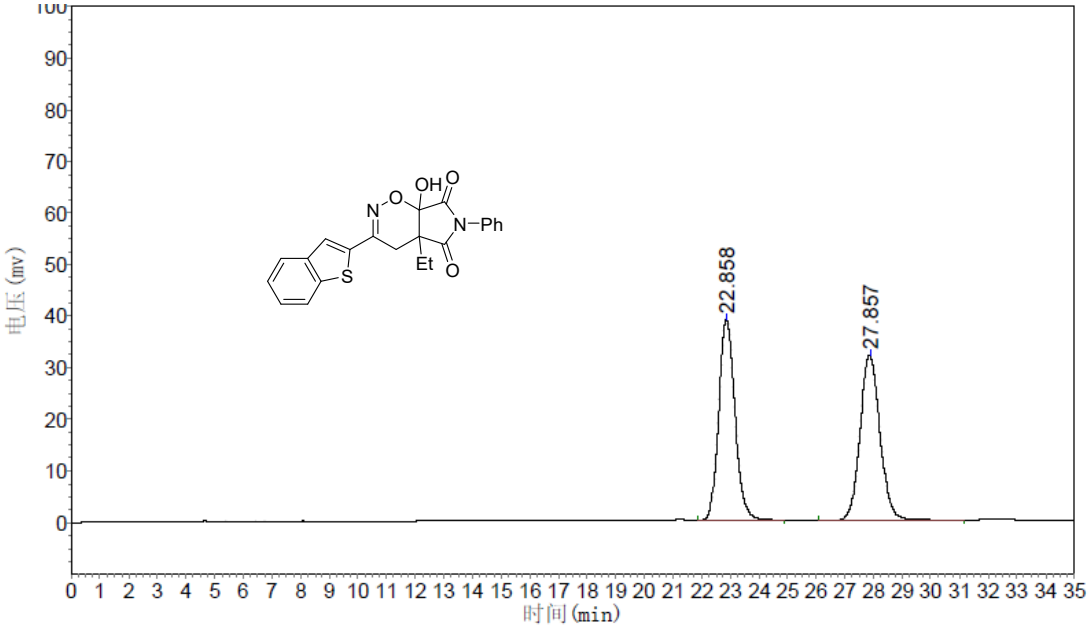
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.988	12740.798	395449.313	49.8862
2	20.598	9663.707	397253.438	50.1138



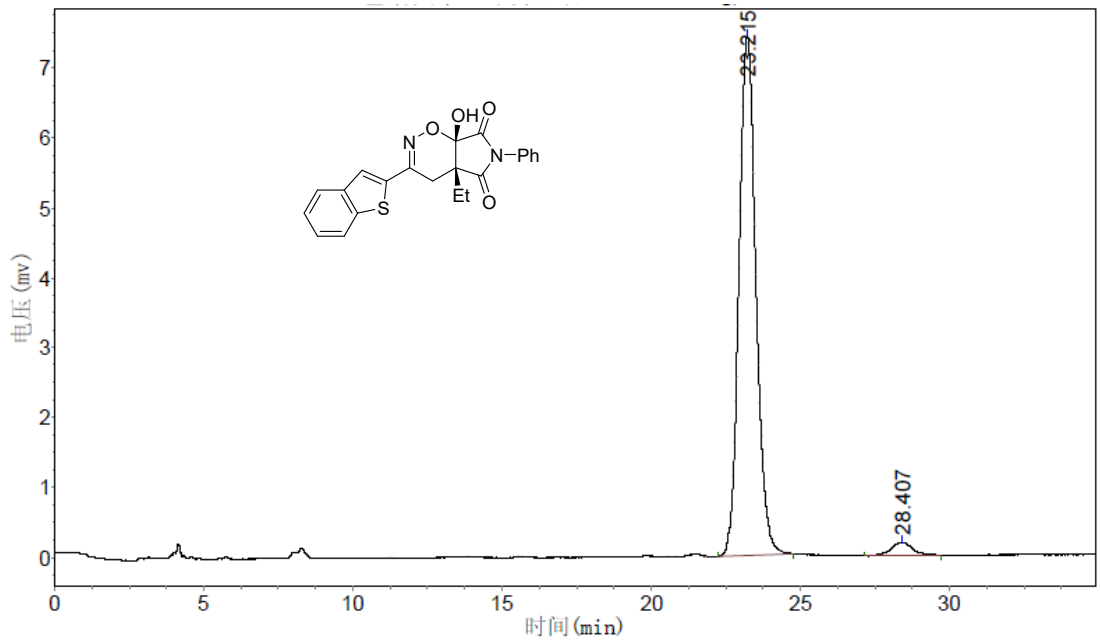
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.807	26816.297	816345.563	95.0340
2	20.383	1059.722	42657.996	4.9660

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15 min, t_{minor} = 20 min

3ao



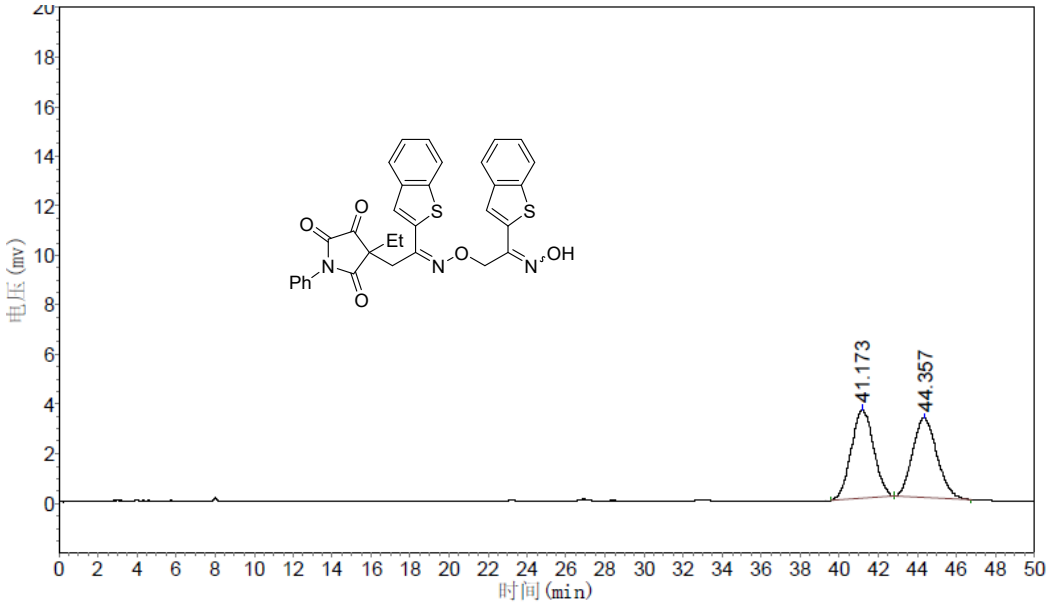
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	22.858	38872.797	1528147.625	49.9328
2	27.857	31845.869	1532259.000	50.0672



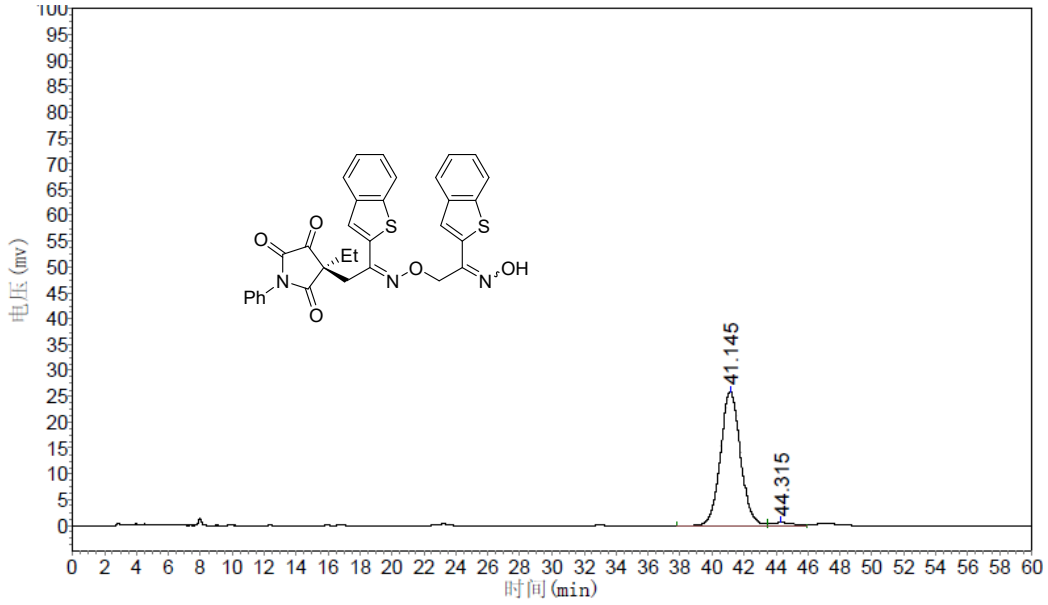
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	23.215	7425.785	292571.813	96.8746
2	28.407	193.519	9439.000	3.1254

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 23 min, t_{minor} = 28 min

3ao'



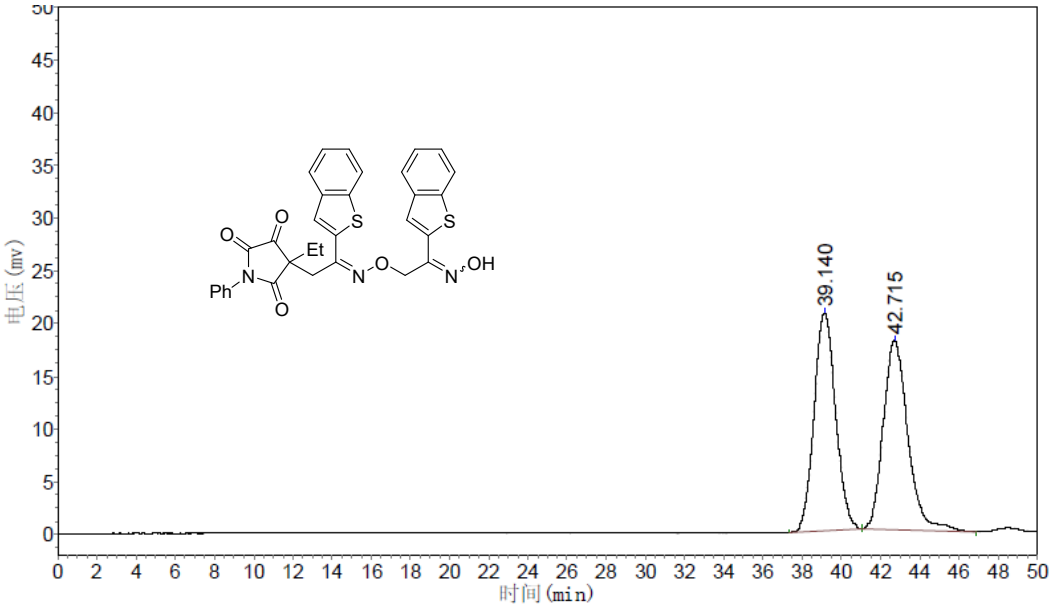
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	41.173	3568.592	284154.156	50.5807
2	44.357	3183.152	277629.813	49.4193



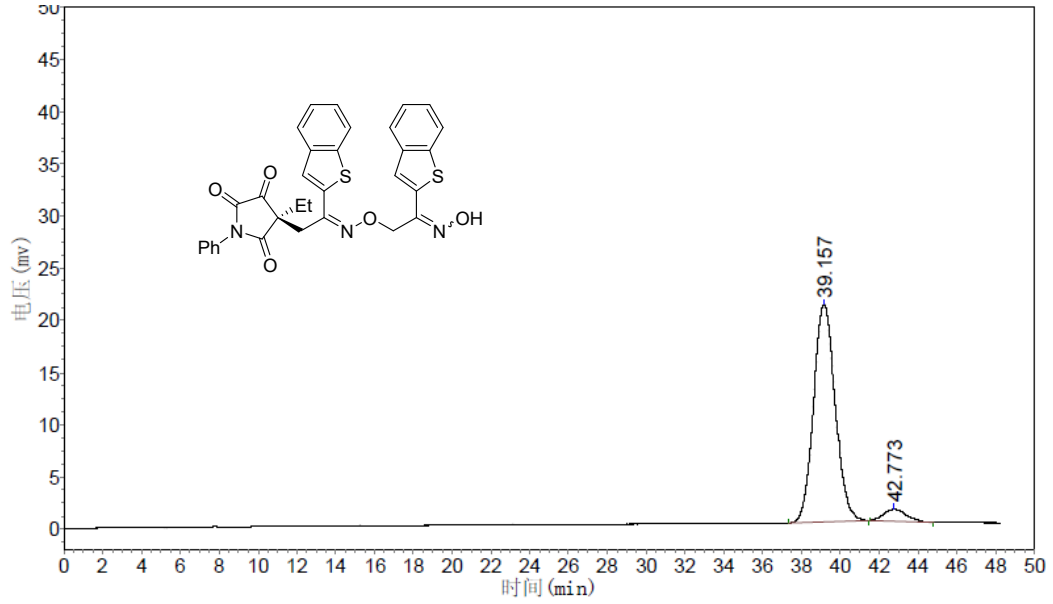
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	41.145	25870.180	2228276.000	97.7426
2	44.315	545.500	51463.750	2.2574

Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 41 min, t_{minor} = 44 min

3ao' (2.2 eq)



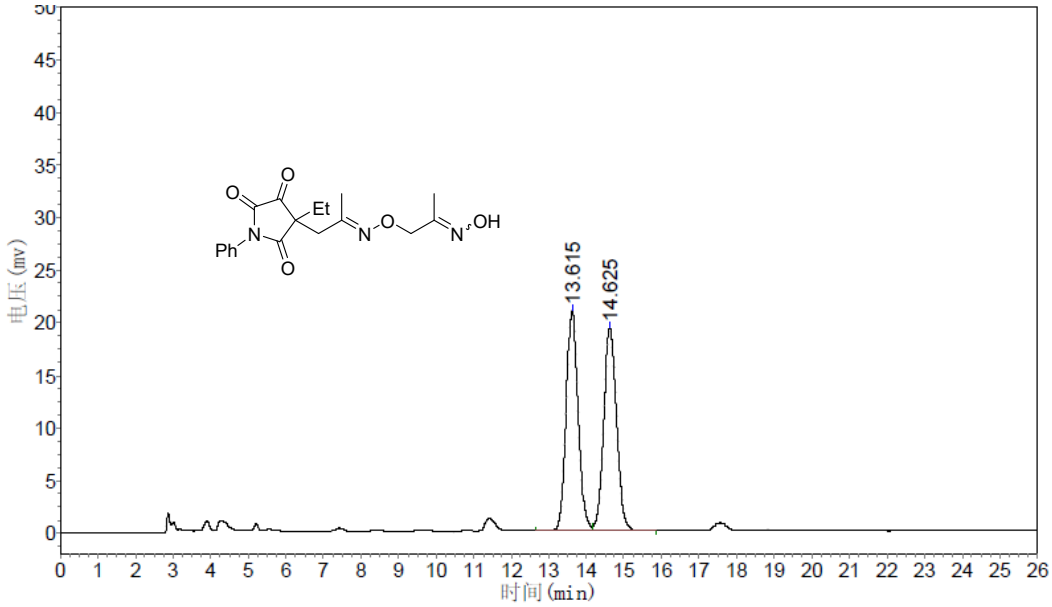
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	39.140	20662.900	1575071.375	50.2131
2	42.715	17914.256	1561702.125	49.7869



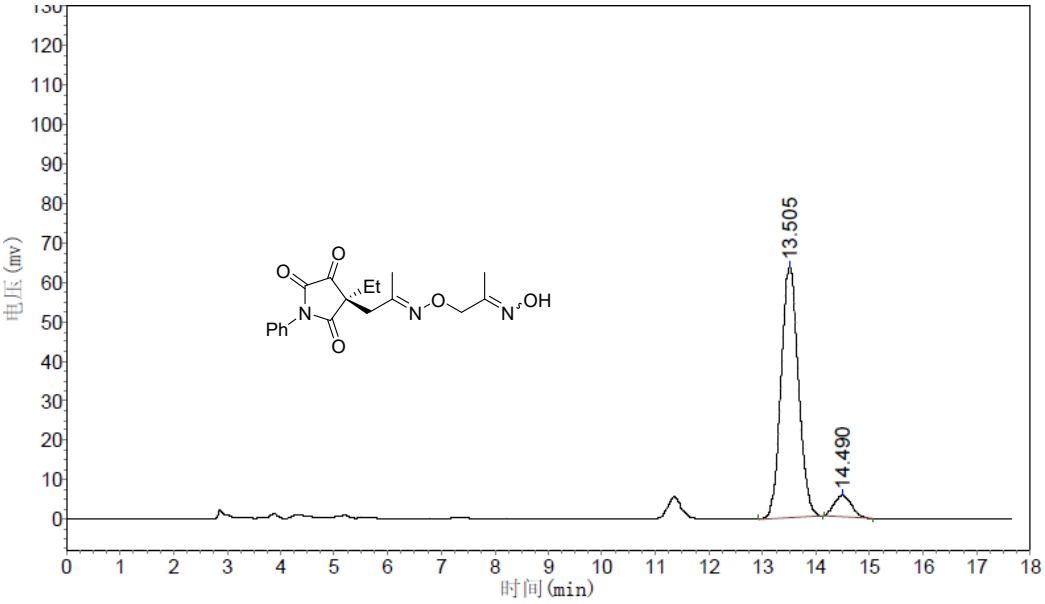
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	39.157	20868.396	1615189.750	94.8490
2	42.773	1136.342	87715.914	5.1510

Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 39 min, t_{minor} = 42 min

3ap'

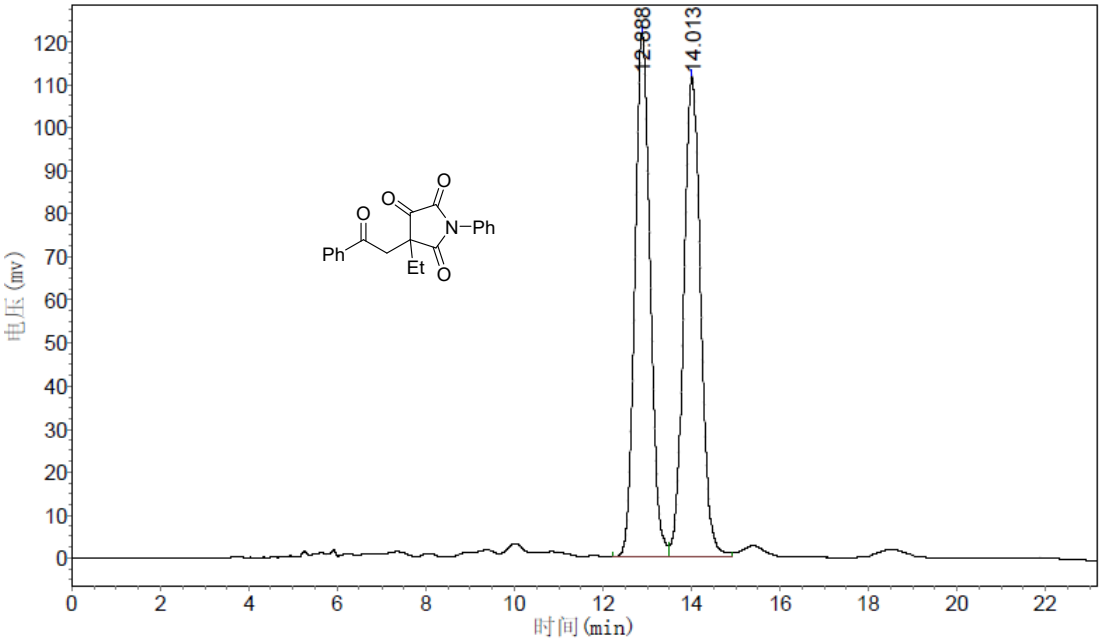


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.615	20931.334	469013.219	50.3902
2	14.625	19290.162	461748.750	49.6098

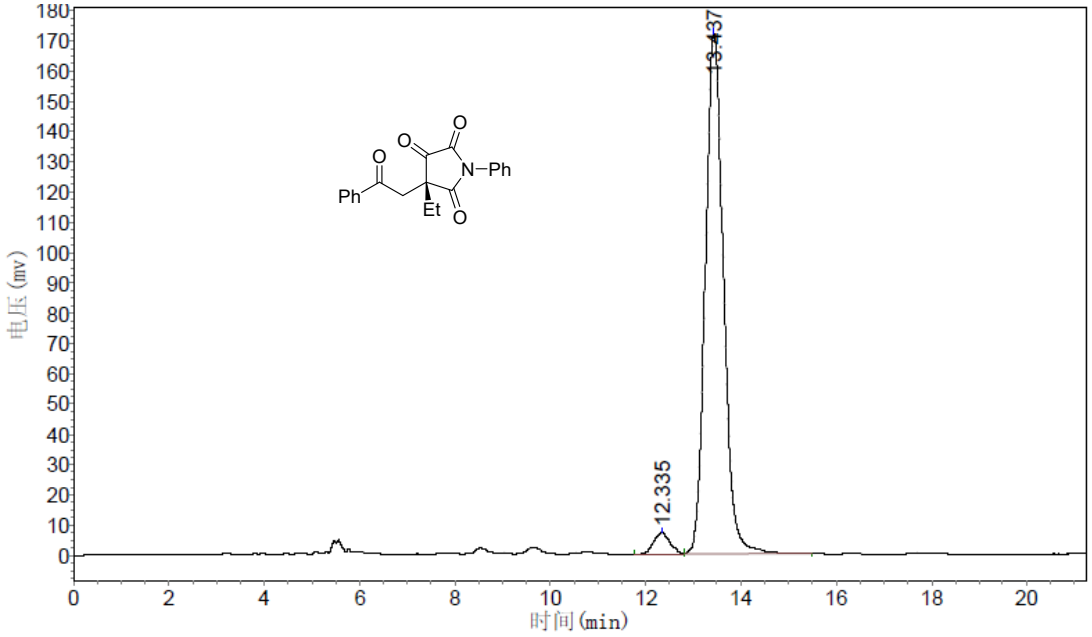


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	13.505	63459.309	1428135.000	92.0713
2	14.490	5396.768	122983.711	7.9287

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 13 min, t_{minor} = 14 min



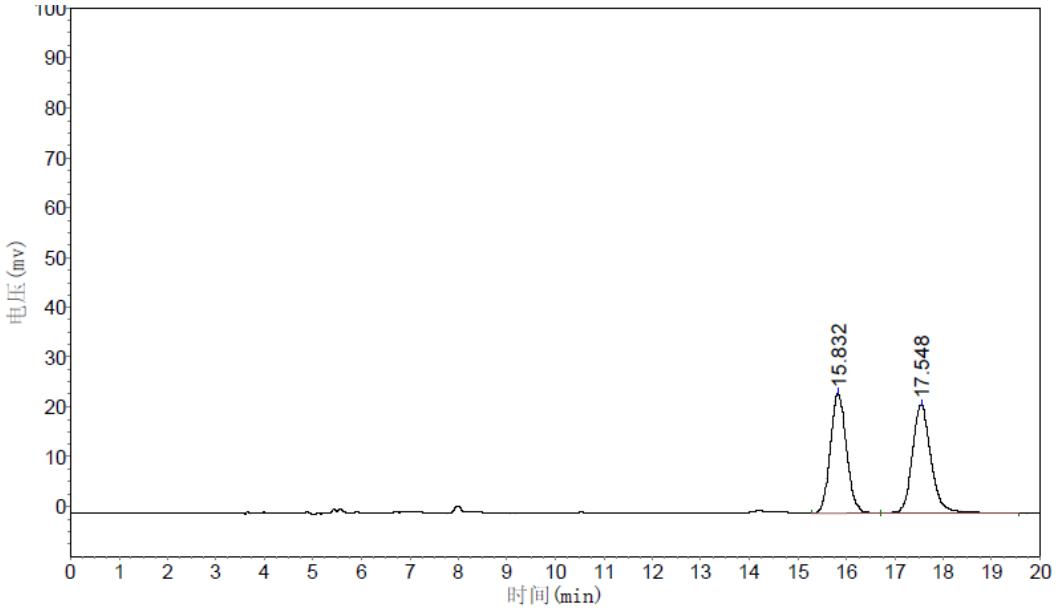
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.888	121920.891	2934003.750	49.5593
2	14.013	111816.977	2986185.000	50.4407



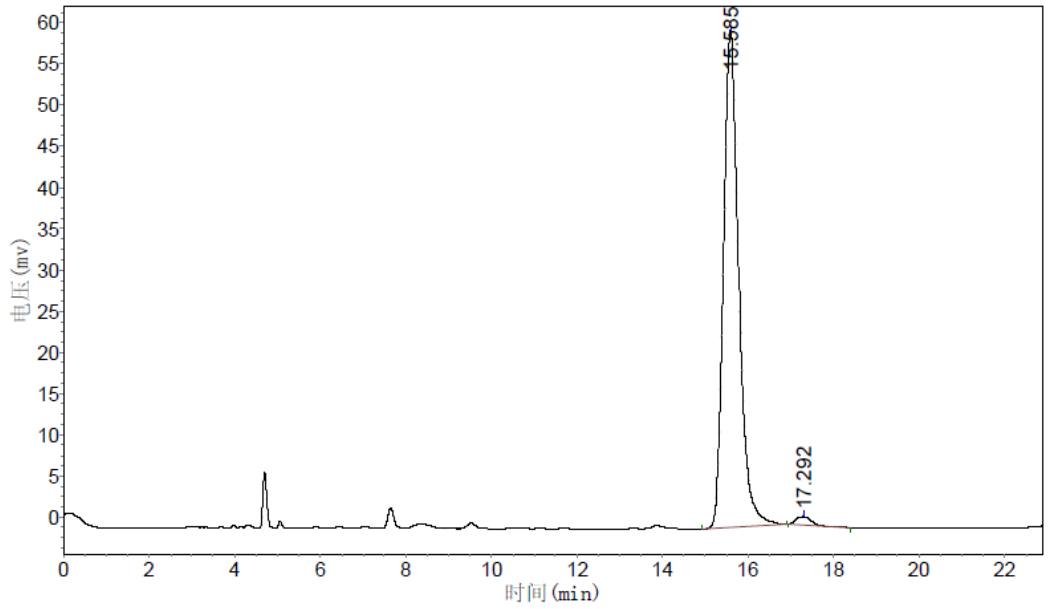
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.335	6860.317	162957.672	3.4655
2	13.437	171715.078	4539268.500	96.5345

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 12 min, t_{minor} = 14 min

3aa Scale up



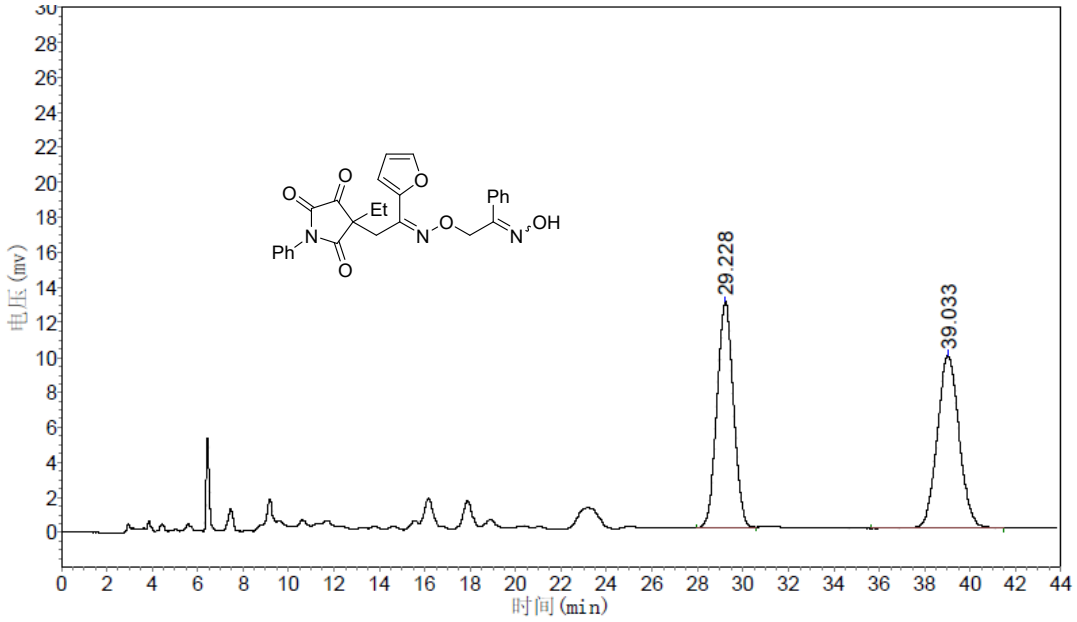
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.827	24015.346	553793.438	49.1216
2	17.540	21669.410	573600.313	50.8784



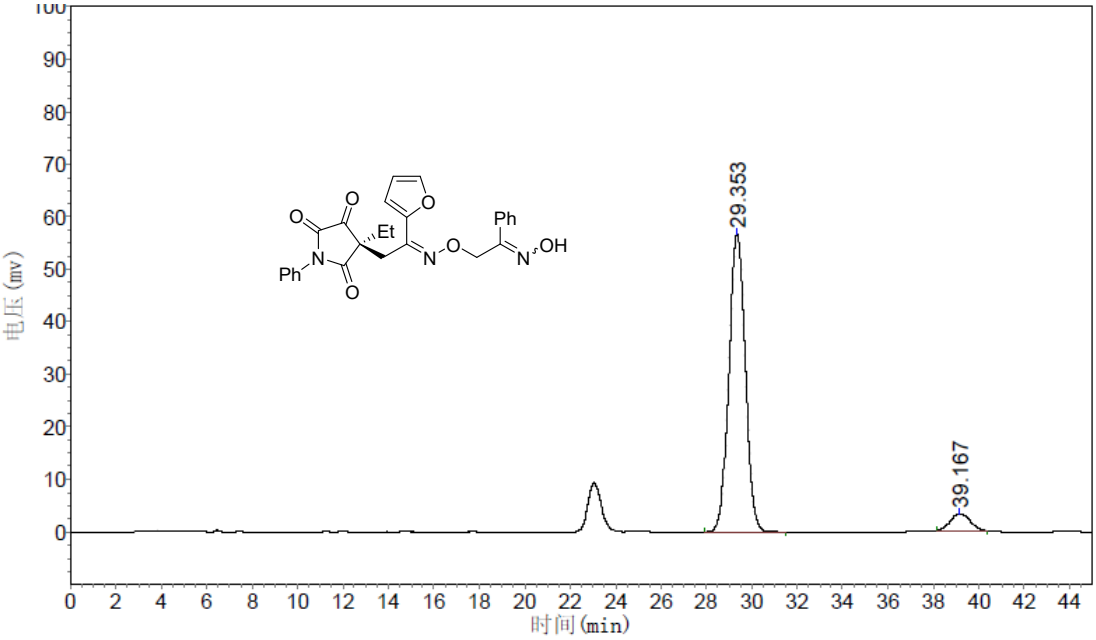
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	15.585	60205.543	1484715.250	98.3510
2	17.292	1065.370	24894.047	1.6490

Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 12 min, t_{minor} = 14 min

3ama



Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	29.228	12901.671	658688.875	49.7667
2	39.033	9864.483	664864.000	50.2333



Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	29.353	56601.680	2835231.250	93.4743
2	39.167	3187.604	197934.781	6.5257

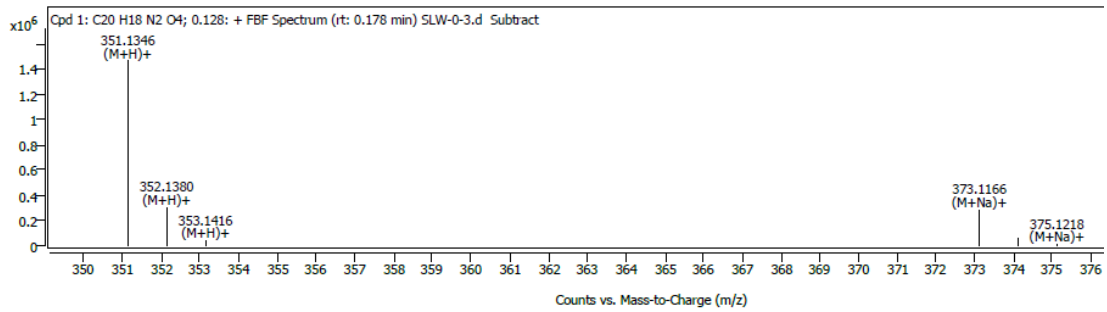
Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 29 min, t_{minor} = 39 min.

HRMS

3aa

Cpd. 1: C20 H18 N2 O4

Compound Spectra



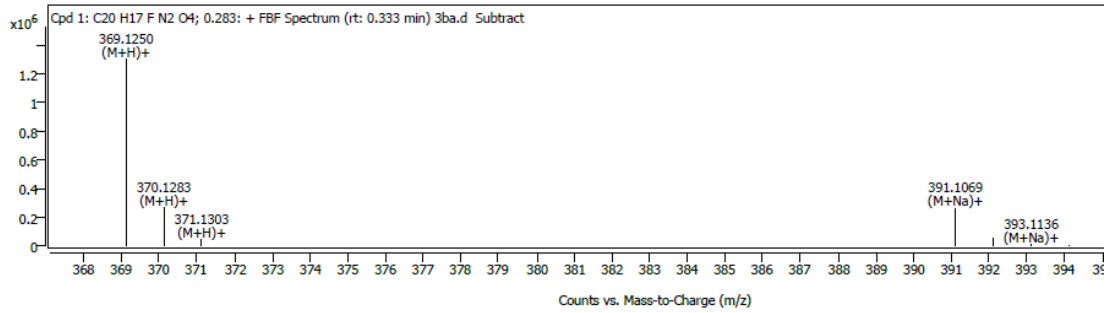
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
351.1346	351.1339	1.94	1466728	100.00	100.00	(M+H)+	1
352.1380	352.1371	2.57	305606	20.84	22.73	(M+H)+	1
353.1416	353.1398	5.21	39123	2.67	3.29	(M+H)+	1
373.1166	373.1159	2.05	286719	100.00	100.00	(M+Na)+	1
374.1195	374.1191	1.24	63905	22.29	22.72	(M+Na)+	1
375.1218	375.1217	0.25	11113	3.88	3.28	(M+Na)+	1

3ba

Cpd. 1: C20 H17 F N2 O4

Compound Spectra



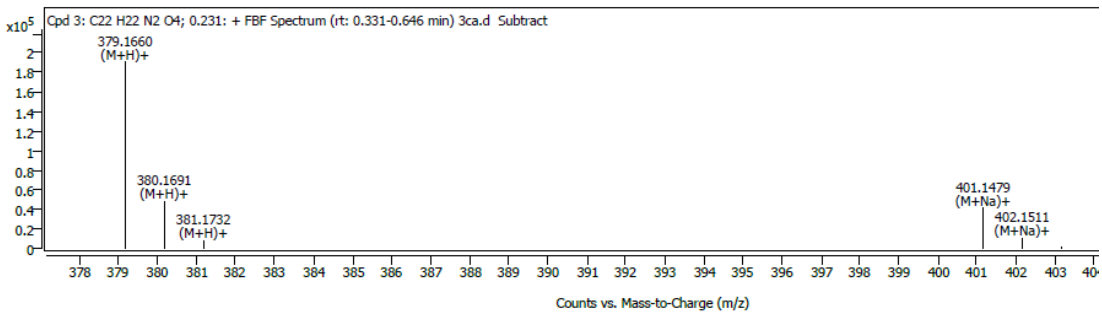
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
369.1250	369.1245	1.34	1305243	100.00	100.00	(M+H)+	1
370.1283	370.1277	1.66	265474	20.34	22.72	(M+H)+	1
371.1303	371.1303	-0.06	41421	3.17	3.28	(M+H)+	1
391.1069	391.1065	1.17	260796	100.00	100.00	(M+Na)+	1
392.1102	392.1096	1.47	56995	21.85	22.71	(M+Na)+	1
393.1136	393.1123	3.28	8237	3.16	3.28	(M+Na)+	1
394.1141	394.1149	-1.99	601	0.23	0.36	(M+Na)+	1

3ca

Cpd. 3: C22 H22 N2 O4

Compound Spectra



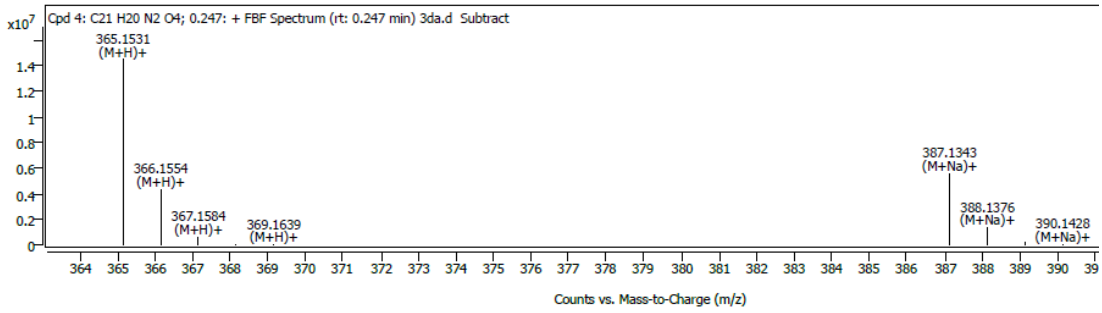
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
379.1660	379.1652	1.93	190943	100.00	100.00	(M+H)+	1
380.1691	380.1684	1.81	48435	25.37	24.94	(M+H)+	1
381.1732	381.1712	5.41	8204	4.30	3.80	(M+H)+	1
401.1479	401.1472	1.91	41242	100.00	100.00	(M+Na)+	1
402.1511	402.1504	1.80	10534	25.54	24.93	(M+Na)+	1
403.1595	403.1531	15.81	1987	4.82	3.80	(M+Na)+	1

3da

Cpd. 4: C21 H20 N2 O4

Compound Spectra



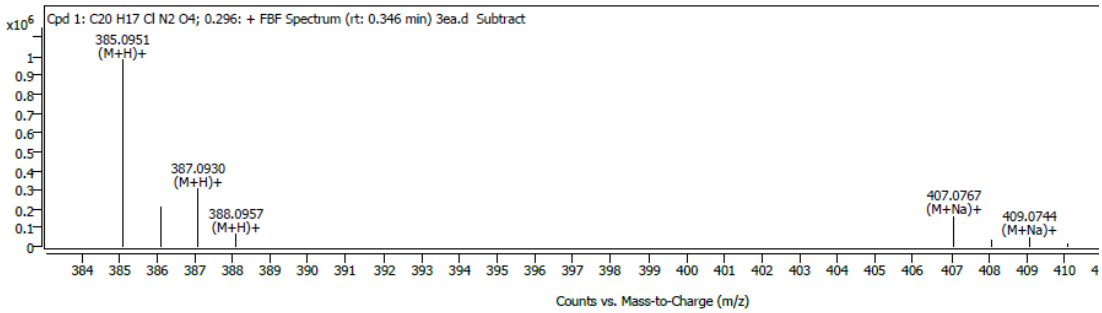
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
365.1531	365.1496	9.49	14485329	100.00	100.00	(M+H)+	1
366.1554	366.1528	7.17	4337080	29.94	23.84	(M+H)+	1
367.1584	367.1555	7.90	642679	4.44	3.54	(M+H)+	1
368.1615	368.1581	9.39	69734	0.48	0.39	(M+H)+	1
369.1639	369.1607	8.85	8334	0.06	0.03	(M+H)+	1
387.1343	387.1315	7.25	5592626	100.00	100.00	(M+Na)+	1
388.1376	388.1347	7.29	1339836	23.96	23.83	(M+Na)+	1
389.1405	389.1374	7.99	187830	3.36	3.54	(M+Na)+	1
390.1428	390.1400	7.05	23067	0.41	0.39	(M+Na)+	1

3ea

Cpd. 1: C20 H17 Cl N2 O4

Compound Spectra



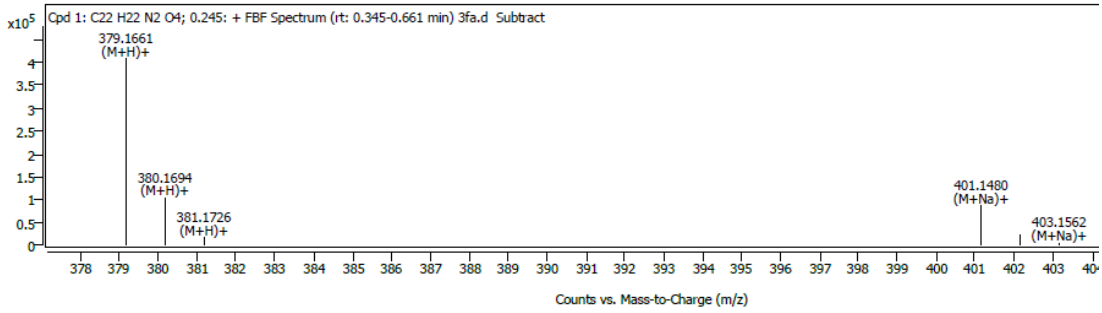
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
385.0951	385.0950	0.36	980203	100.00	100.00	(M+H)+	1
386.0982	386.0981	0.02	210063	21.43	22.72	(M+H)+	1
387.0930	387.0928	0.35	302280	30.84	35.28	(M+H)+	1
388.0957	388.0956	0.41	63714	6.50	7.63	(M+H)+	1
407.0767	407.0769	-0.57	159523	100.00	100.00	(M+Na)+	1
408.0802	408.0801	0.32	35306	22.13	22.71	(M+Na)+	1
409.0744	409.0748	-0.85	48942	30.68	35.28	(M+Na)+	1
410.0768	410.0775	-1.76	12693	7.96	7.62	(M+Na)+	1

3fa

Cpd. 1: C22 H22 N2 O4

Compound Spectra



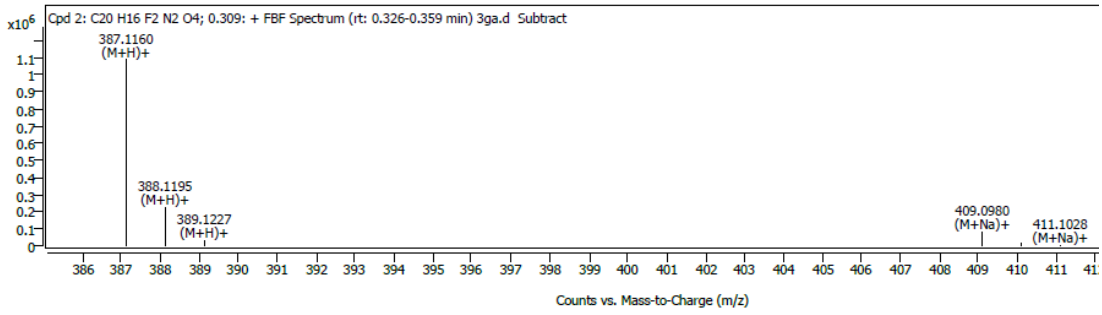
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
379.1661	379.1652	2.41	407673	100.00	100.00	(M+H)+	1
380.1694	380.1684	2.43	102397	25.12	24.94	(M+H)+	1
381.1726	381.1712	3.68	16032	3.93	3.80	(M+H)+	1
401.1480	401.1472	2.12	85297	100.00	100.00	(M+Na)+	1
402.1512	402.1504	1.96	21956	25.74	24.93	(M+Na)+	1
403.1562	403.1531	7.61	3702	4.34	3.80	(M+Na)+	1

3ga

Cpd. 2: C20 H16 F2 N2 O4

Compound Spectra



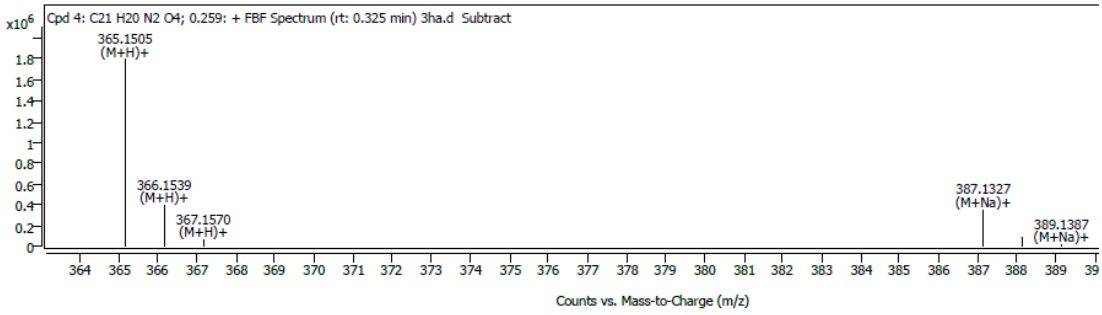
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
387.1160	387.1151	2.36	1089990	100.00	100.00	(M+H)+	1
388.1195	388.1183	3.13	226278	20.76	22.71	(M+H)+	1
389.1227	389.1209	4.61	31078	2.85	3.28	(M+H)+	1
409.0980	409.0970	2.46	82372	100.00	100.00	(M+Na)+	1
410.1012	410.1002	2.42	17717	21.51	22.70	(M+Na)+	1
411.1028	411.1029	-0.17	3467	4.21	3.28	(M+Na)+	1

3ha

Cpd. 4: C21 H20 N2 O4

Compound Spectra



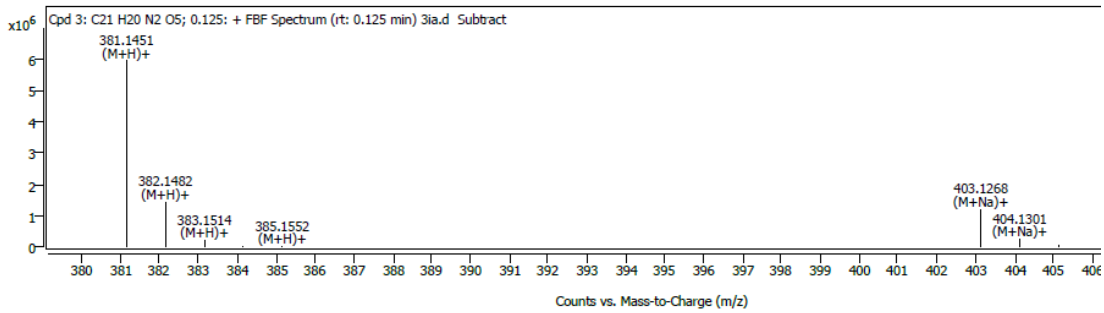
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
365.1505	365.1496	2.54	1799427	100.00	100.00	(M+H)+	1
366.1539	366.1528	3.08	392277	21.80	23.84	(M+H)+	1
367.1570	367.1555	4.17	60760	3.38	3.54	(M+H)+	1
387.1327	387.1315	2.96	351917	100.00	100.00	(M+Na)+	1
388.1359	388.1347	3.13	82926	23.56	23.83	(M+Na)+	1
389.1387	389.1374	3.33	14209	4.04	3.54	(M+Na)+	1

3ia

Cpd. 3: C21 H20 N2 O5

Compound Spectra



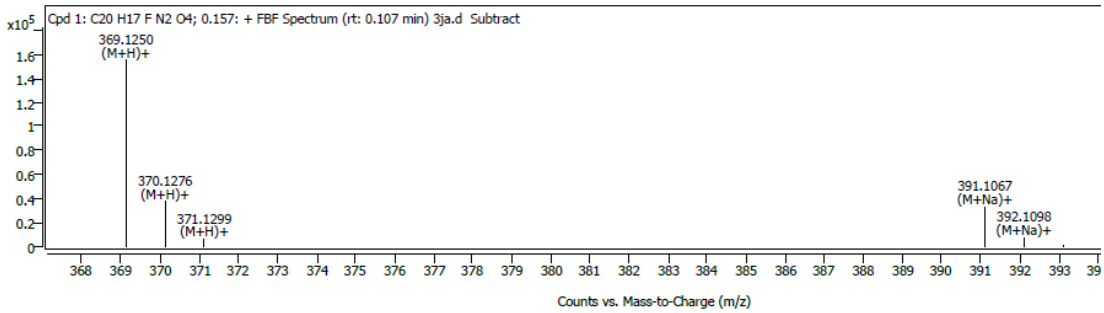
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
381.1451	381.1445	1.62	5935420	100.00	100.00	(M+H)+	1
382.1482	382.1477	1.21	1434329	24.17	23.88	(M+H)+	1
383.1514	383.1503	2.84	200238	3.37	3.75	(M+H)+	1
384.1542	384.1529	3.32	25788	0.43	0.44	(M+H)+	1
385.1552	385.1554	-0.47	3014	0.05	0.04	(M+H)+	1
403.1268	403.1264	0.77	1185478	100.00	100.00	(M+Na)+	1
404.1301	404.1296	1.13	242022	20.42	23.86	(M+Na)+	1
405.1326	405.1322	1.00	43437	3.66	3.75	(M+Na)+	1

3ja

Cpd. 1: C20 H17 F N2 O4

Compound Spectra



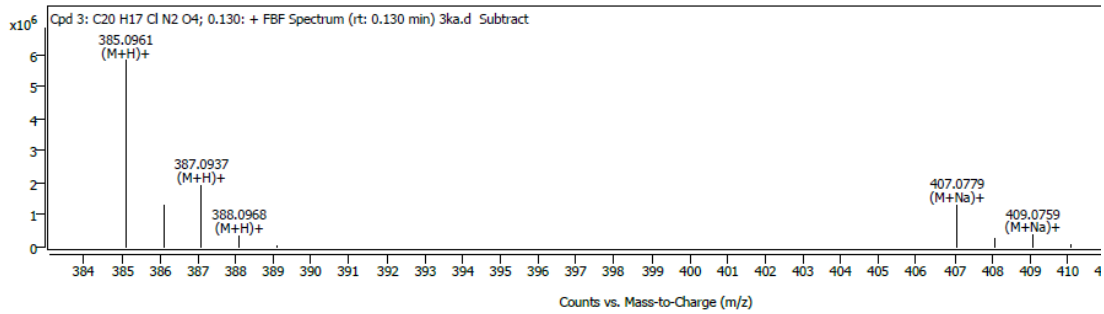
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
369.1250	369.1245	1.25	154941	100.00	100.00	(M+H)+	1
370.1276	370.1277	-0.18	37512	24.21	22.72	(M+H)+	1
371.1299	371.1303	-1.21	6232	4.02	3.28	(M+H)+	1
391.1067	391.1065	0.53	33304	100.00	100.00	(M+Na)+	1
392.1098	392.1096	0.46	7890	23.69	22.71	(M+Na)+	1
393.1180	393.1123	14.60	1434	4.30	3.28	(M+Na)+	1

3ka

Cpd. 3: C20 H17 Cl N2 O4

Compound Spectra



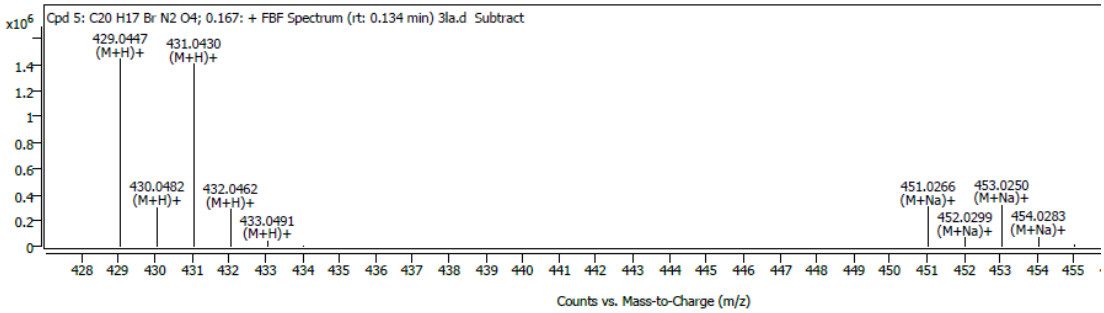
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
385.0961	385.0950	3.02	5812792	100.00	100.00	(M+H)+	1
386.0991	386.0981	2.49	1298354	22.34	22.72	(M+H)+	1
387.0937	387.0928	2.18	1935741	33.30	35.28	(M+H)+	1
388.0968	388.0956	3.13	369646	6.36	7.63	(M+H)+	1
389.0997	389.0981	4.06	57948	1.00	1.08	(M+H)+	1
407.0779	407.0769	2.32	1310898	100.00	100.00	(M+Na)+	1
408.0813	408.0801	2.91	273993	20.90	22.71	(M+Na)+	1
409.0759	409.0748	2.72	390126	29.76	35.28	(M+Na)+	1
410.0785	410.0775	2.33	86828	6.62	7.62	(M+Na)+	1

3la

Cpd. 5: C20 H17 Br N2 O4

Compound Spectra



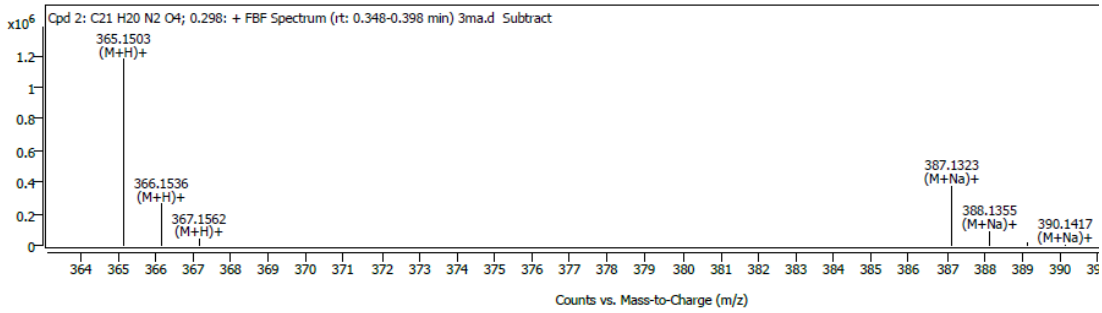
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
429.0447	429.0444	0.61	1438729	100.00	99.44	(M+H)+	1
430.0482	430.0476	1.33	296304	20.59	22.59	(M+H)+	1
431.0430	431.0427	0.80	1400069	97.31	100.00	(M+H)+	1
432.0462	432.0457	1.09	283631	19.71	22.33	(M+H)+	1
433.0491	433.0483	1.79	39378	2.74	3.21	(M+H)+	1
434.0513	434.0509	1.08	4918	0.34	0.35	(M+H)+	1
451.0266	451.0264	0.55	302789	96.77	99.44	(M+Na)+	1
452.0299	452.0296	0.74	64832	20.72	22.58	(M+Na)+	1
453.0250	453.0246	0.80	312895	100.00	100.00	(M+Na)+	1
454.0283	454.0276	1.43	70129	22.41	22.32	(M+Na)+	1
455.0296	455.0302	-1.32	10739	3.43	3.21	(M+Na)+	1

3ma

Cpd. 2: C21 H20 N2 O4

Compound Spectra



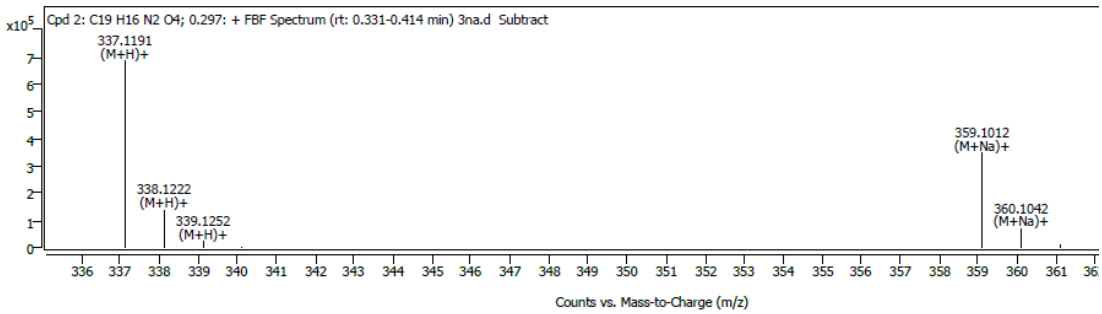
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
365.1503	365.1496	1.96	1174798	100.00	100.00	(M+H)+	1
366.1536	366.1528	2.32	262504	22.34	23.84	(M+H)+	1
367.1562	367.1555	2.12	40384	3.44	3.54	(M+H)+	1
387.1323	387.1315	2.03	373488	100.00	100.00	(M+Na)+	1
388.1355	388.1347	1.87	90130	24.13	23.83	(M+Na)+	1
389.1385	389.1374	2.84	16459	4.41	3.54	(M+Na)+	1
390.1417	390.1400	4.34	2359	0.63	0.39	(M+Na)+	1

3na

Cpd. 2: C19 H16 N2 O4

Compound Spectra



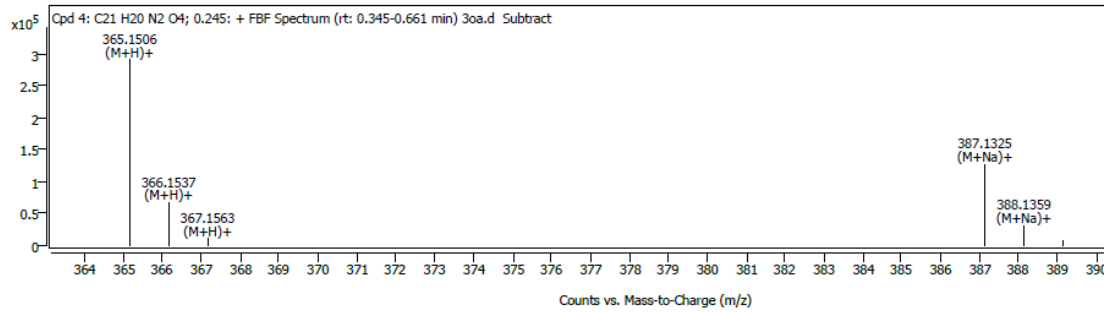
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
337.1191	337.1183	2.56	684575	100.00	100.00	(M+H)+	1
338.1222	338.1215	2.11	136554	19.95	21.63	(M+H)+	1
339.1252	339.1241	3.24	22046	3.22	3.05	(M+H)+	1
340.1256	340.1266	-3.07	2751	0.40	0.32	(M+H)+	1
359.1012	359.1002	2.77	346486	100.00	100.00	(M+Na)+	1
360.1042	360.1034	2.32	67819	19.57	21.62	(M+Na)+	1
361.1069	361.1060	2.37	10559	3.05	3.05	(M+Na)+	1

3oa

Cpd. 4: C21 H20 N2 O4

Compound Spectra



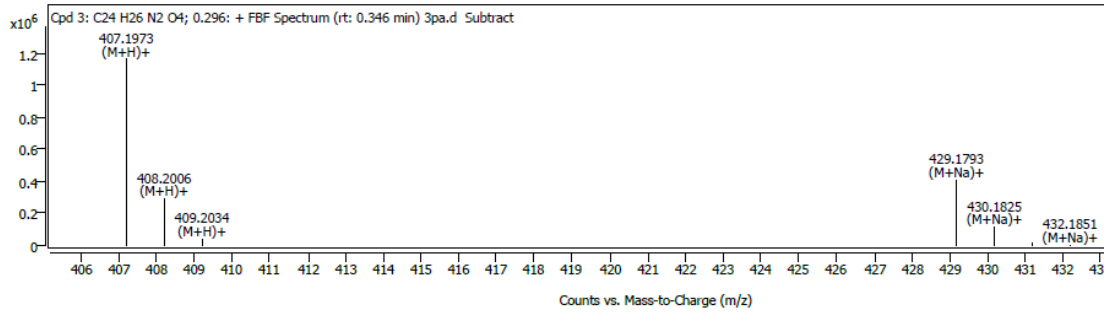
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
365.1506	365.1496	2.71	291235	100.00	100.00	(M+H)+	1
366.1537	366.1528	2.61	67169	23.06	23.84	(M+H)+	1
367.1563	367.1555	2.27	11191	3.84	3.54	(M+H)+	1
387.1325	387.1315	2.39	127311	100.00	100.00	(M+Na)+	1
388.1359	388.1347	2.97	31549	24.78	23.83	(M+Na)+	1
389.1399	389.1374	6.34	8226	6.46	3.54	(M+Na)+	1

3pa

Cpd. 3: C24 H26 N2 O4

Compound Spectra



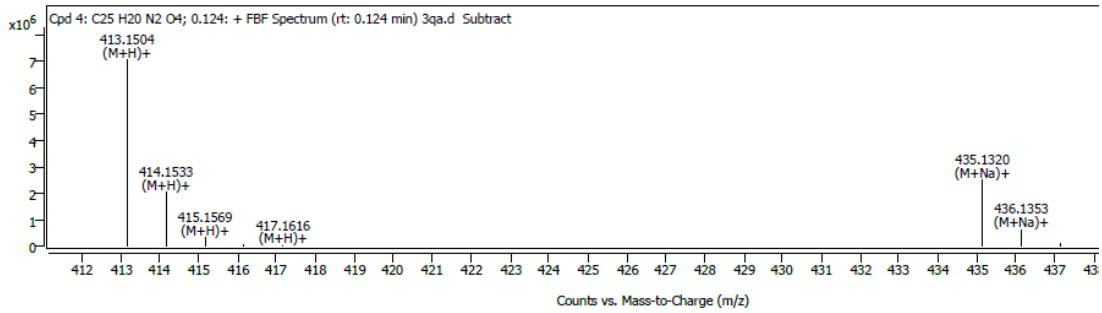
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
407.1973	407.1965	1.89	1160467	100.00	100.00	(M+H)+	1
408.2006	408.1998	1.97	291407	25.11	27.15	(M+H)+	1
409.2034	409.2026	2.17	43597	3.76	4.37	(M+H)+	1
429.1793	429.1785	1.96	408871	100.00	100.00	(M+Na)+	1
430.1825	430.1817	1.83	115653	28.29	27.14	(M+Na)+	1
431.1845	431.1845	0.03	17387	4.25	4.36	(M+Na)+	1
432.1851	432.1872	-4.73	2888	0.71	0.52	(M+Na)+	1

3qa

Cpd. 4: C25 H20 N2 O4

Compound Spectra



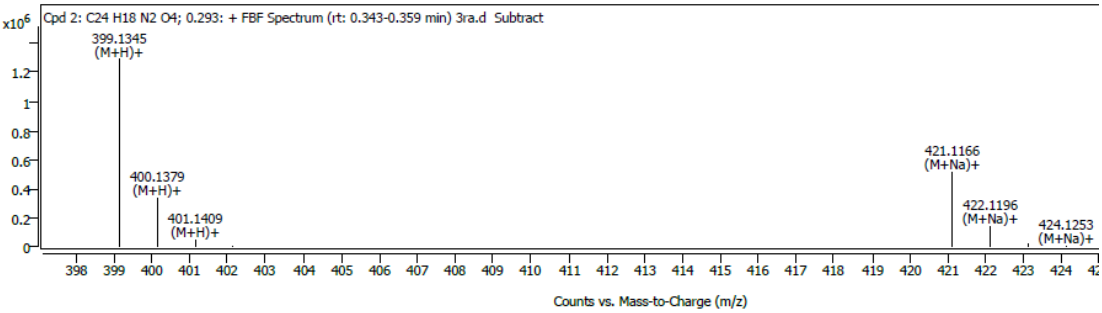
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
413.1504	413.1496	2.09	7019094	100.00	100.00	(M+H)+	1
414.1533	414.1528	1.26	2027023	28.88	28.16	(M+H)+	1
415.1569	415.1556	3.10	314341	4.48	4.64	(M+H)+	1
416.1600	416.1583	3.97	40667	0.58	0.56	(M+H)+	1
417.1616	417.1610	1.51	2394	0.03	0.05	(M+H)+	1
435.1320	435.1315	1.02	2491953	100.00	100.00	(M+Na)+	1
436.1353	436.1347	1.33	628781	25.23	28.15	(M+Na)+	1
437.1381	437.1376	1.16	102995	4.13	4.64	(M+Na)+	1

3ra

Cpd. 2: C24 H18 N2 O4

Compound Spectra



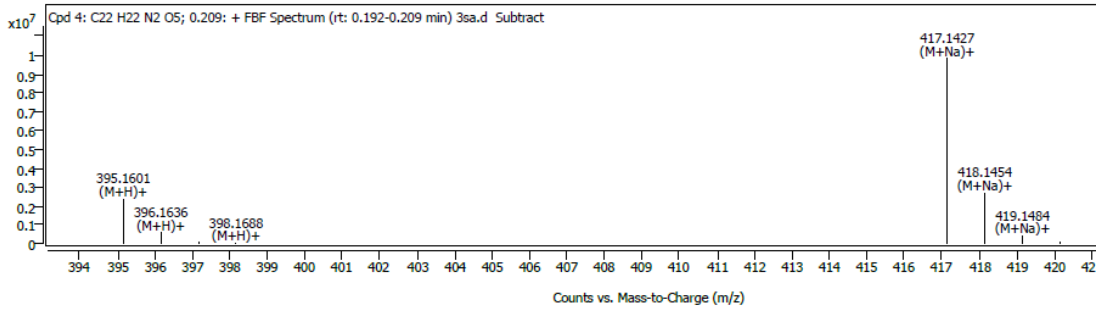
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
399.1345	399.1339	1.36	1290503	100.00	100.00	(M+H)+	1
400.1379	400.1371	1.89	335058	25.96	27.06	(M+H)+	1
401.1409	401.1399	2.46	47413	3.67	4.34	(M+H)+	1
402.1446	402.1426	4.93	6487	0.50	0.51	(M+H)+	1
421.1166	421.1159	1.63	515190	100.00	100.00	(M+Na)+	1
422.1196	422.1191	1.16	140328	27.24	27.05	(M+Na)+	1
423.1226	423.1219	1.65	20526	3.98	4.34	(M+Na)+	1
424.1253	424.1246	1.79	3067	0.60	0.51	(M+Na)+	1

3sa

Cpd. 4: C22 H22 N2 O5

Compound Spectra



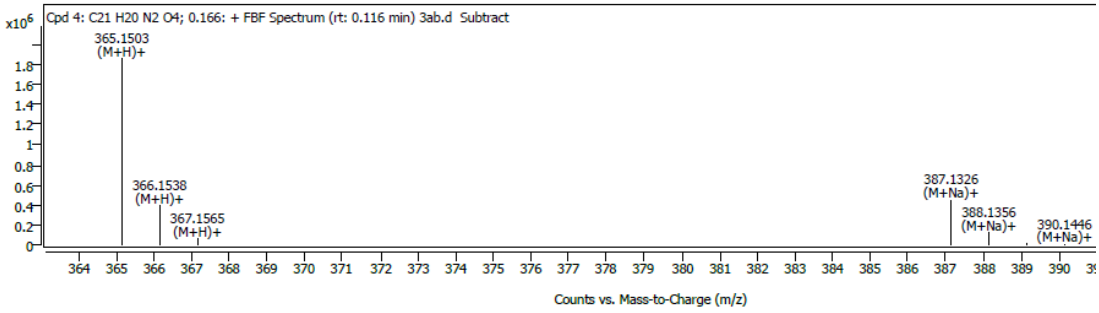
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
395.1601	395.1601	-0.14	2376497	100.00	100.00	(M+H)+	1
396.1636	396.1634	0.51	574685	24.18	24.98	(M+H)+	1
397.1663	397.1660	0.72	87088	3.66	4.02	(M+H)+	1
398.1688	398.1686	0.35	9983	0.42	0.48	(M+H)+	1
417.1427	417.1421	1.37	9825860	100.00	100.00	(M+Na)+	1
418.1454	418.1453	0.20	2701282	27.49	24.97	(M+Na)+	1
419.1484	419.1479	1.00	392317	3.99	4.01	(M+Na)+	1
420.1486	420.1506	-4.70	56345	0.57	0.48	(M+Na)+	1

3ab

Cpd. 4: C21 H20 N2 O4

Compound Spectra



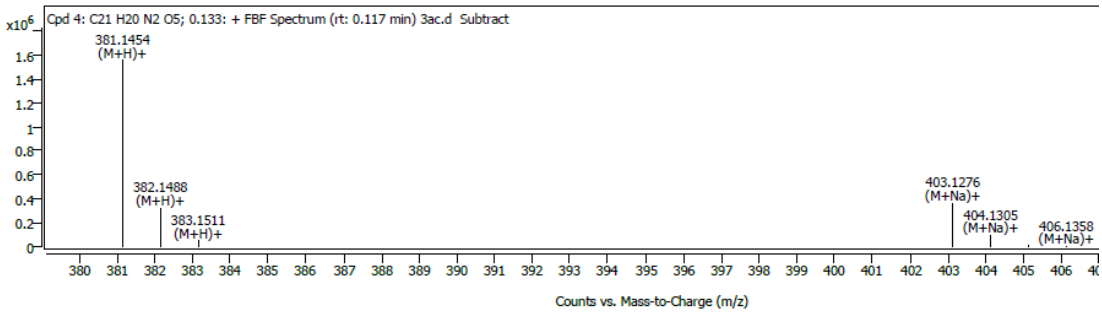
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
365.1503	365.1496	1.86	1856117	100.00	100.00	(M+H)+	1
366.1538	366.1528	2.83	391077	21.07	23.84	(M+H)+	1
367.1565	367.1555	2.76	59297	3.19	3.54	(M+H)+	1
387.1326	387.1315	2.84	449037	100.00	100.00	(M+Na)+	1
388.1356	388.1347	2.21	119930	26.71	23.83	(M+Na)+	1
389.1385	389.1374	2.77	20687	4.61	3.54	(M+Na)+	1
390.1446	390.1400	11.60	1192	0.27	0.39	(M+Na)+	1

3ac

Cpd. 4: C21 H20 N2 O5

Compound Spectra



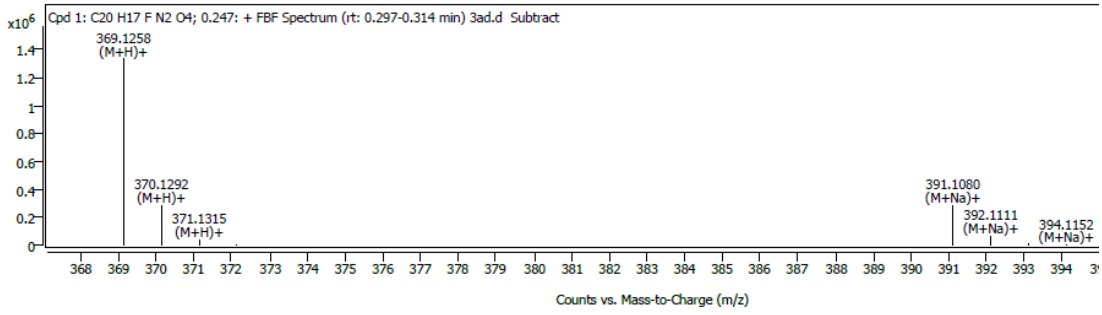
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
381.1454	381.1445	2.48	1551675	100.00	100.00	(M+H)+	1
382.1488	382.1477	2.99	321081	20.69	23.88	(M+H)+	1
383.1511	383.1503	2.11	50835	3.28	3.75	(M+H)+	1
403.1276	403.1264	2.97	363550	100.00	100.00	(M+Na)+	1
404.1305	404.1296	2.09	92654	25.49	23.86	(M+Na)+	1
405.1336	405.1322	3.46	14261	3.92	3.75	(M+Na)+	1
406.1358	406.1348	2.33	1667	0.46	0.44	(M+Na)+	1

3ad

Cpd. 1: C20 H17 F N2 O4

Compound Spectra



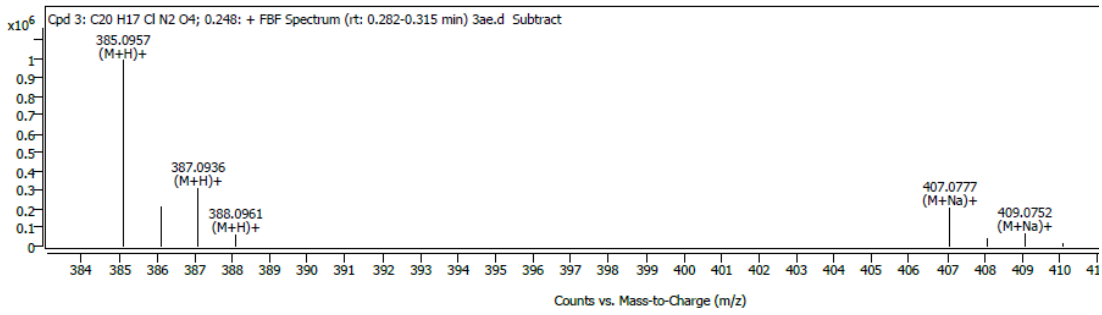
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
369.1258	369.1245	3.42	1333334	100.00	100.00	(M+H)+	1
370.1292	370.1277	3.95	284303	21.32	22.72	(M+H)+	1
371.1315	371.1303	3.23	38683	2.90	3.28	(M+H)+	1
372.1334	372.1329	1.22	4646	0.35	0.36	(M+H)+	1
391.1080	391.1065	3.93	285079	100.00	100.00	(M+Na)+	1
392.1111	392.1096	3.85	63914	22.42	22.71	(M+Na)+	1
393.1132	393.1123	2.36	7775	2.73	3.28	(M+Na)+	1
394.1152	394.1149	0.78	1308	0.46	0.36	(M+Na)+	1

3ae

Cpd. 3: C20 H17 Cl N2 O4

Compound Spectra



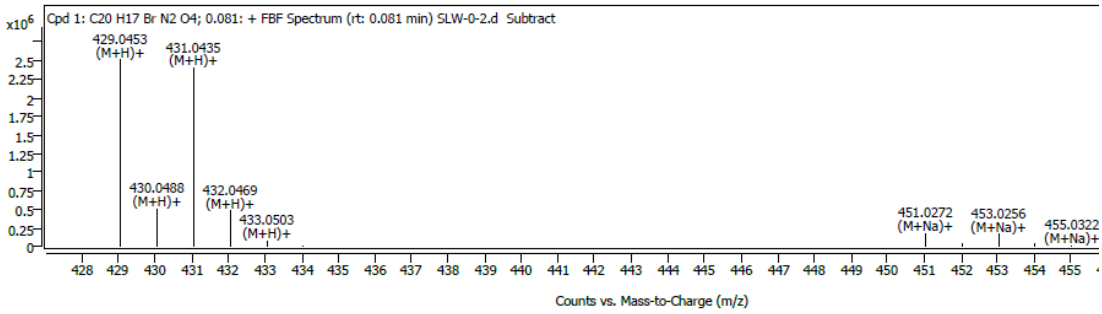
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
385.0957	385.0950	1.82	992260	100.00	100.00	(M+H)+	1
386.0988	386.0981	1.82	207875	20.95	22.72	(M+H)+	1
387.0936	387.0928	1.96	310207	31.26	35.28	(M+H)+	1
388.0961	388.0956	1.29	63190	6.37	7.63	(M+H)+	1
407.0777	407.0769	1.98	204303	100.00	100.00	(M+Na)+	1
408.0806	408.0801	1.30	44278	21.67	22.71	(M+Na)+	1
409.0752	409.0748	1.02	67937	33.25	35.28	(M+Na)+	1
410.0782	410.0775	1.69	14046	6.87	7.62	(M+Na)+	1

3af

Cpd. 1: C20 H17 Br N2 O4

Compound Spectra



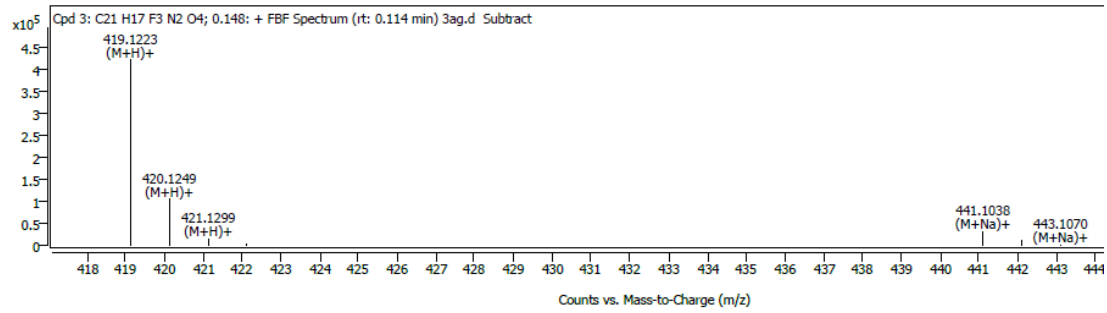
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
429.0453	429.0444	2.07	2510871	100.00	99.44	(M+H)+	1
430.0488	430.0476	2.79	502556	20.02	22.59	(M+H)+	1
431.0435	431.0427	1.99	2397408	95.48	100.00	(M+H)+	1
432.0469	432.0457	2.84	476720	18.99	22.33	(M+H)+	1
433.0503	433.0483	4.52	73401	2.92	3.21	(M+H)+	1
434.0517	434.0509	1.92	11108	0.44	0.35	(M+H)+	1
451.0272	451.0264	1.75	169996	100.00	99.44	(M+Na)+	1
452.0310	452.0296	3.11	36238	21.32	22.58	(M+Na)+	1
453.0256	453.0246	2.22	164436	96.73	100.00	(M+Na)+	1
454.0283	454.0276	1.55	38180	22.46	22.32	(M+Na)+	1
455.0322	455.0302	4.24	4771	2.81	3.21	(M+Na)+	1

3ag

Cpd. 3: C21 H17 F3 N2 O4

Compound Spectra



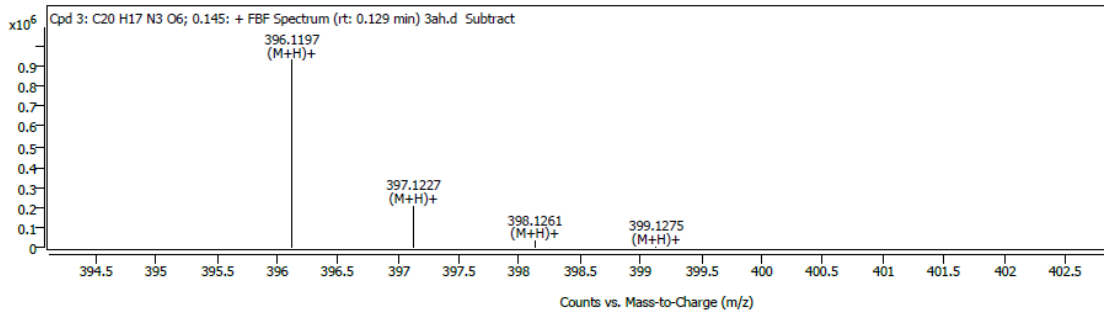
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
419.1223	419.1213	2.42	422257	100.00	100.00	(M+H)+	1
420.1249	420.1245	0.98	105402	24.96	23.80	(M+H)+	1
421.1299	421.1272	6.39	15265	3.62	3.53	(M+H)+	1
422.1331	422.1298	7.72	2548	0.60	0.39	(M+H)+	1
441.1038	441.1033	1.19	32696	100.00	100.00	(M+Na)+	1
442.1068	442.1065	0.77	10665	32.62	23.79	(M+Na)+	1
443.1070	443.1091	-4.83	1803	5.51	3.53	(M+Na)+	1

3ah

Cpd. 3: C20 H17 N3 O6

Compound Spectra



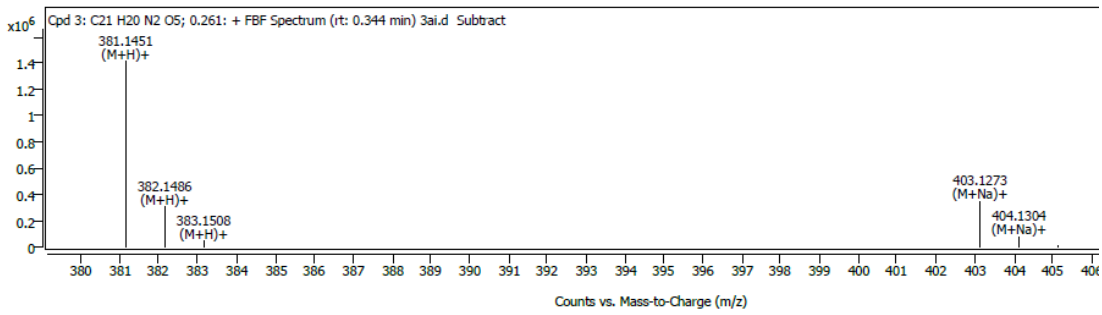
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
396.1197	396.1190	1.76	930224	100.00	100.00	(M+H)+	1
397.1227	397.1221	1.50	204059	21.94	23.16	(M+H)+	1
398.1261	398.1246	3.94	31346	3.37	3.80	(M+H)+	1
399.1275	399.1271	0.93	4808	0.52	0.47	(M+H)+	1

3ai

Cpd. 3: C21 H20 N2 O5

Compound Spectra



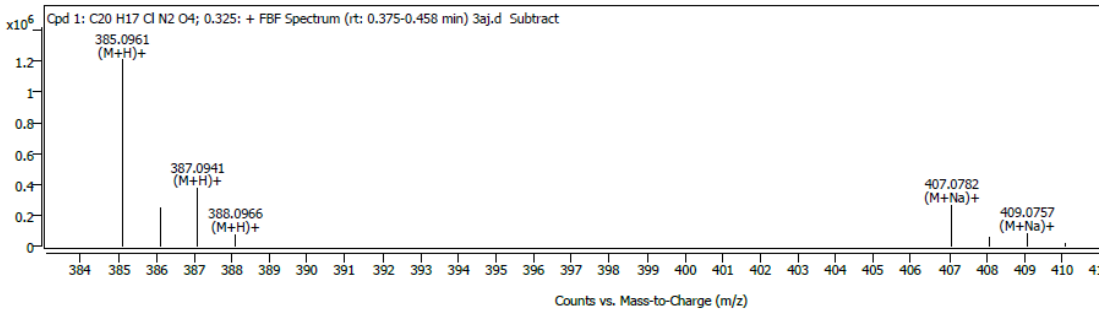
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
381.1451	381.1445	1.62	1416530	100.00	100.00	(M+H)+	1
382.1486	382.1477	2.26	306534	21.64	23.88	(M+H)+	1
383.1508	383.1503	1.38	46138	3.26	3.75	(M+H)+	1
403.1273	403.1264	2.04	349851	100.00	100.00	(M+Na)+	1
404.1304	404.1296	1.98	78082	22.32	23.86	(M+Na)+	1
405.1336	405.1322	3.28	14605	4.17	3.75	(M+Na)+	1

3aj

Cpd. 1: C20 H17 Cl N2 O4

Compound Spectra



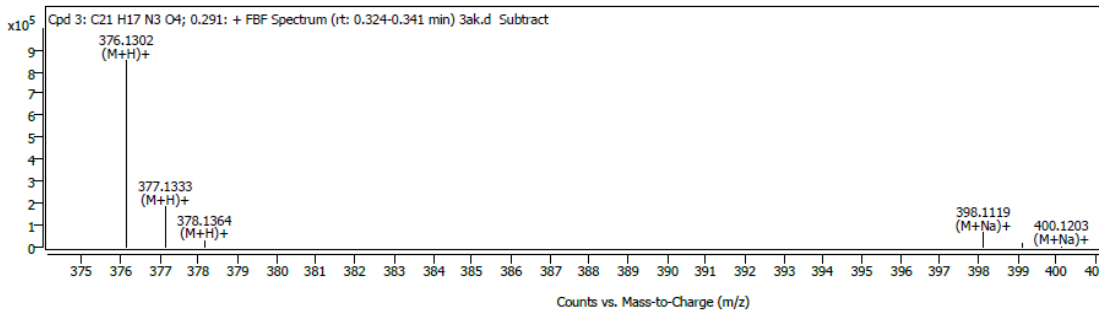
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
385.0961	385.0950	2.99	1209765	100.00	100.00	(M+H)+	1
386.0993	386.0981	3.07	252885	20.90	22.72	(M+H)+	1
387.0941	387.0928	3.35	374455	30.95	35.28	(M+H)+	1
388.0966	388.0956	2.74	77003	6.37	7.63	(M+H)+	1
407.0782	407.0769	3.18	265412	100.00	100.00	(M+Na)+	1
408.0812	408.0801	2.84	59384	22.37	22.71	(M+Na)+	1
409.0757	409.0748	2.39	85743	32.31	35.28	(M+Na)+	1
410.0784	410.0775	2.14	20516	7.73	7.62	(M+Na)+	1

3ak

Cpd. 3: C21 H17 N3 O4

Compound Spectra



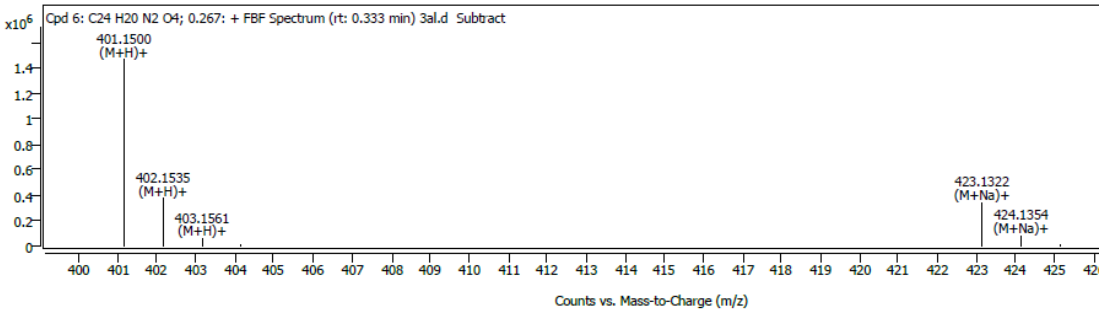
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
376.1302	376.1292	2.63	851064	100.00	100.00	(M+H)+	1
377.1333	377.1323	2.66	184575	21.69	24.17	(M+H)+	1
378.1364	378.1349	3.93	28320	3.33	3.62	(M+H)+	1
398.1119	398.1111	1.82	67162	100.00	100.00	(M+Na)+	1
399.1149	399.1142	1.77	16041	23.88	24.16	(M+Na)+	1
400.1203	400.1169	8.54	2650	3.95	3.61	(M+Na)+	1

3al

Cpd. 6: C24 H20 N2 O4

Compound Spectra



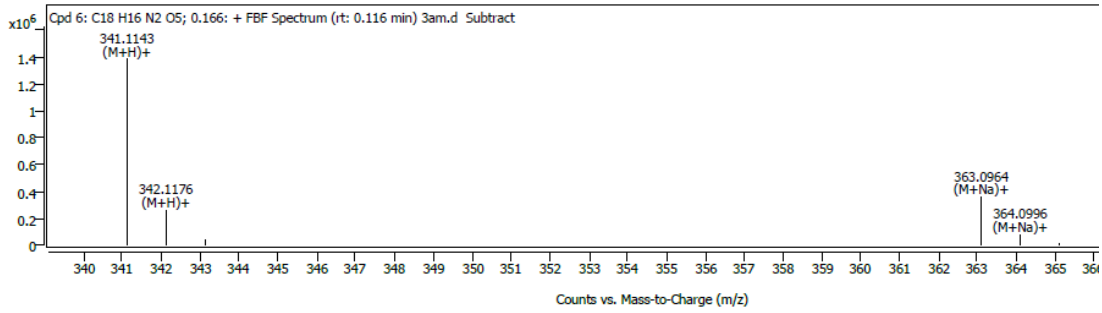
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
401.1500	401.1496	1.14	1464729	100.00	100.00	(M+H)+	1
402.1535	402.1528	1.69	374857	25.59	27.08	(M+H)+	1
403.1561	403.1556	1.24	58139	3.97	4.35	(M+H)+	1
404.1584	404.1583	0.23	7844	0.54	0.52	(M+H)+	1
423.1322	423.1315	1.48	344077	100.00	100.00	(M+Na)+	1
424.1354	424.1347	1.47	84440	24.54	27.07	(M+Na)+	1
425.1380	425.1375	1.15	14292	4.15	4.34	(M+Na)+	1

3am

Cpd. 6: C18 H16 N2 O5

Compound Spectra



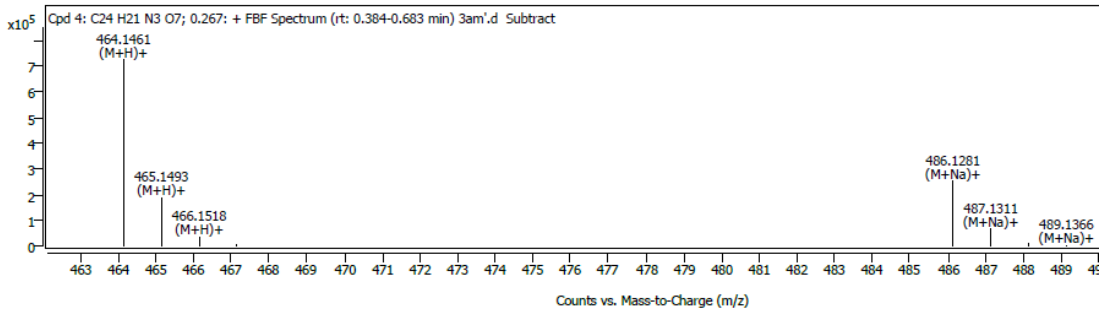
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
341.1143	341.1132	3.25	1385281	100.00	100.00	(M+H)+	1
342.1176	342.1164	3.72	258615	18.67	20.58	(M+H)+	1
343.1208	343.1188	5.79	42447	3.06	3.04	(M+H)+	1
363.0964	363.0951	3.51	356436	100.00	100.00	(M+Na)+	1
364.0996	364.0983	3.66	79219	22.23	20.57	(M+Na)+	1
365.1021	365.1008	3.74	13041	3.66	3.04	(M+Na)+	1

3am'

Cpd. 4: C24 H21 N3 O7

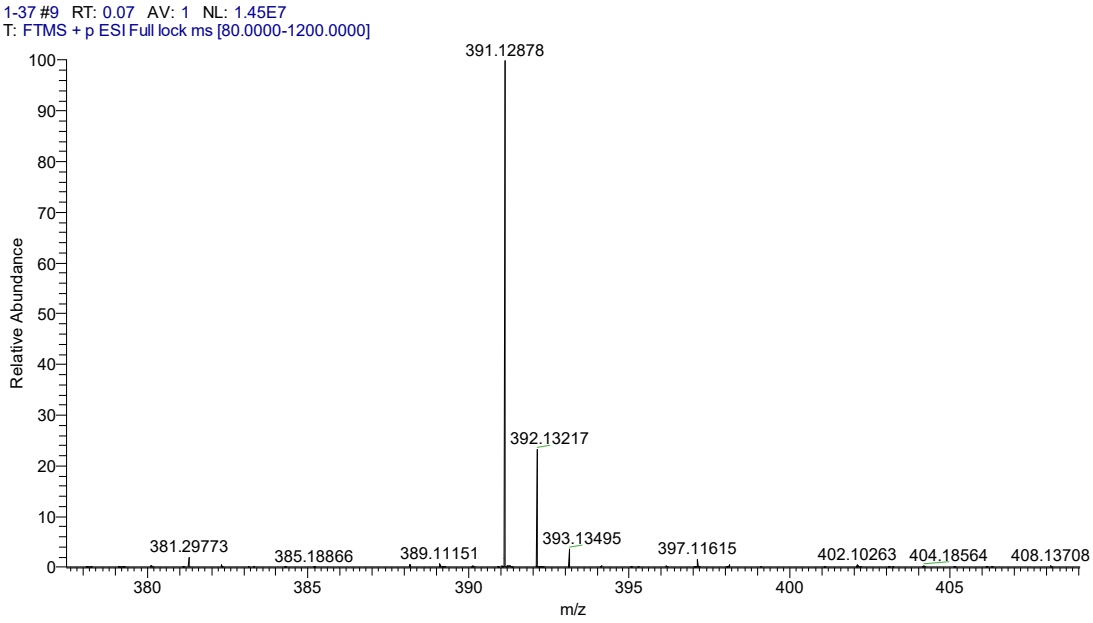
Compound Spectra



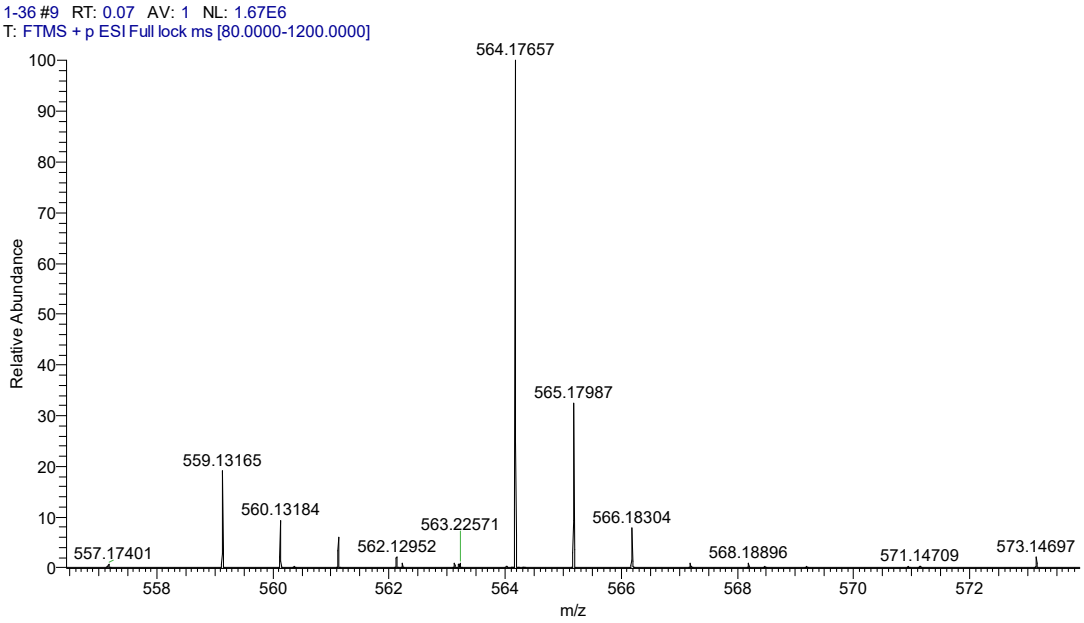
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
464.1461	464.1452	1.98	723080	100.00	100.00	(M+H)+	1
465.1493	465.1484	2.06	189164	26.16	27.57	(M+H)+	1
466.1518	466.1509	1.87	34461	4.77	5.10	(M+H)+	1
467.1547	467.1535	2.68	5071	0.70	0.71	(M+H)+	1
486.1281	486.1272	1.98	251756	100.00	100.00	(M+Na)+	1
487.1311	487.1303	1.64	66188	26.29	27.56	(M+Na)+	1
488.1338	488.1329	1.85	12034	4.78	5.09	(M+Na)+	1
489.1366	489.1354	2.33	1831	0.73	0.71	(M+Na)+	1

3an


1-37 #9 RT: 0.07 AV: 1 NL: 1.45E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
391.12878
392.13217
381.29773
385.18866
389.11151
393.13495
397.11615
402.10263
404.18564
408.13708
Relative Abundance
m/z

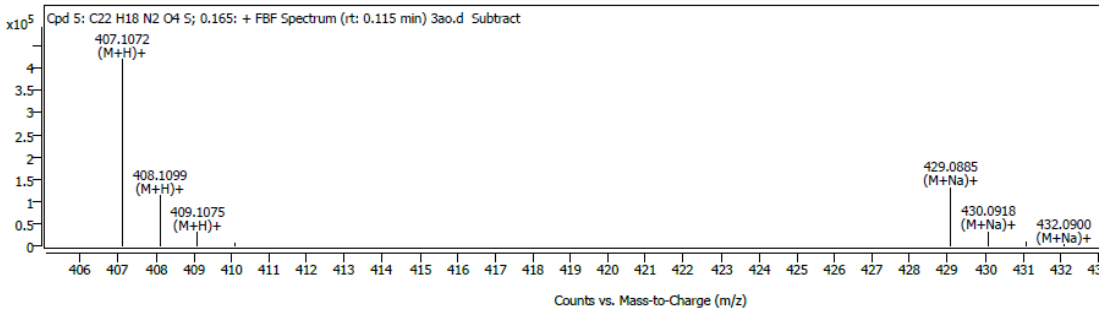
3an'


1-36 #9 RT: 0.07 AV: 1 NL: 1.67E6
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
564.17657
565.17987
559.13165
560.13184
563.22571
566.18304
557.17401
562.12952
568.18896
571.14709
573.14697
Relative Abundance
m/z

3ao

Cpd. 5: C22 H18 N2 O4 S

Compound Spectra



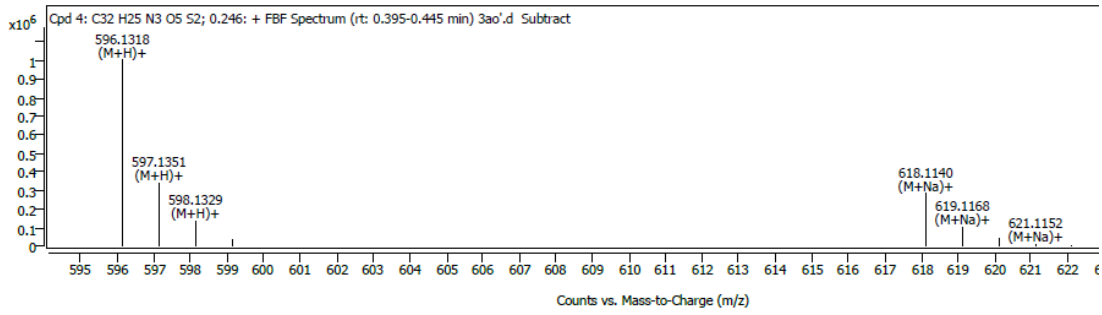
Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
407.1072	407.1060	2.93	418326	100.00	100.00	(M+H)+	1
408.1099	408.1091	1.93	112496	26.89	25.69	(M+H)+	1
409.1075	409.1065	2.48	29766	7.12	8.46	(M+H)+	1
410.1082	410.1077	1.07	6707	1.60	1.57	(M+H)+	1
429.0885	429.0879	1.39	130840	100.00	100.00	(M+Na)+	1
430.0918	430.0910	1.82	32390	24.76	25.67	(M+Na)+	1
431.0910	431.0884	5.90	8706	6.65	8.46	(M+Na)+	1
432.0900	432.0897	0.83	2292	1.75	1.57	(M+Na)+	1

3ao'

Cpd. 4: C32 H25 N3 O5 S2

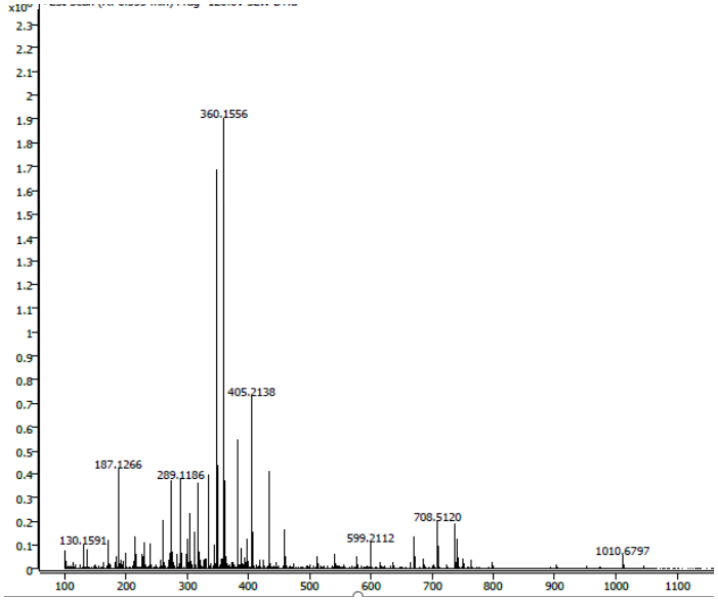
Compound Spectra



Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
596.1318	596.1308	1.69	1002052	100.00	100.00	(M+H)+	1
597.1351	597.1339	2.12	339375	33.87	37.77	(M+H)+	1
598.1329	598.1314	2.59	133642	13.34	16.92	(M+H)+	1
599.1334	599.1323	1.76	35982	3.59	4.52	(M+H)+	1
618.1140	618.1128	1.90	283023	100.00	100.00	(M+Na)+	1
619.1168	619.1158	1.62	101742	35.95	37.76	(M+Na)+	1
620.1146	620.1133	2.09	38391	13.56	16.91	(M+Na)+	1
621.1152	621.1143	1.55	10246	3.62	4.52	(M+Na)+	1
622.1152	622.1138	2.24	2323	0.82	1.05	(M+Na)+	1

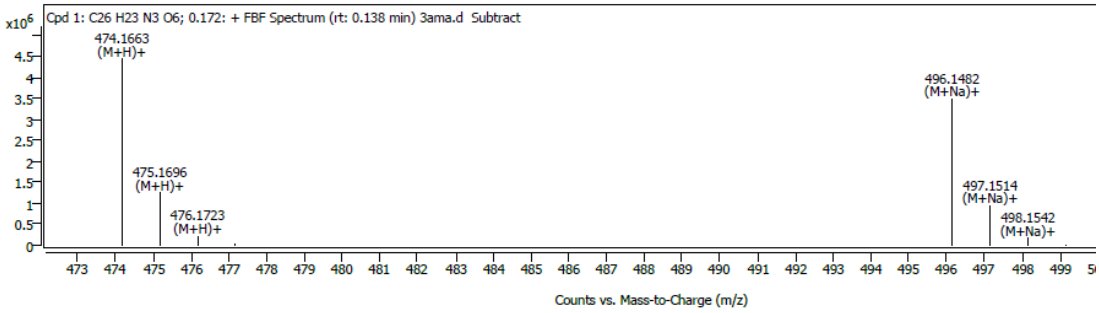
3ap'



3ama

Cpd. 1: C26 H23 N3 O6

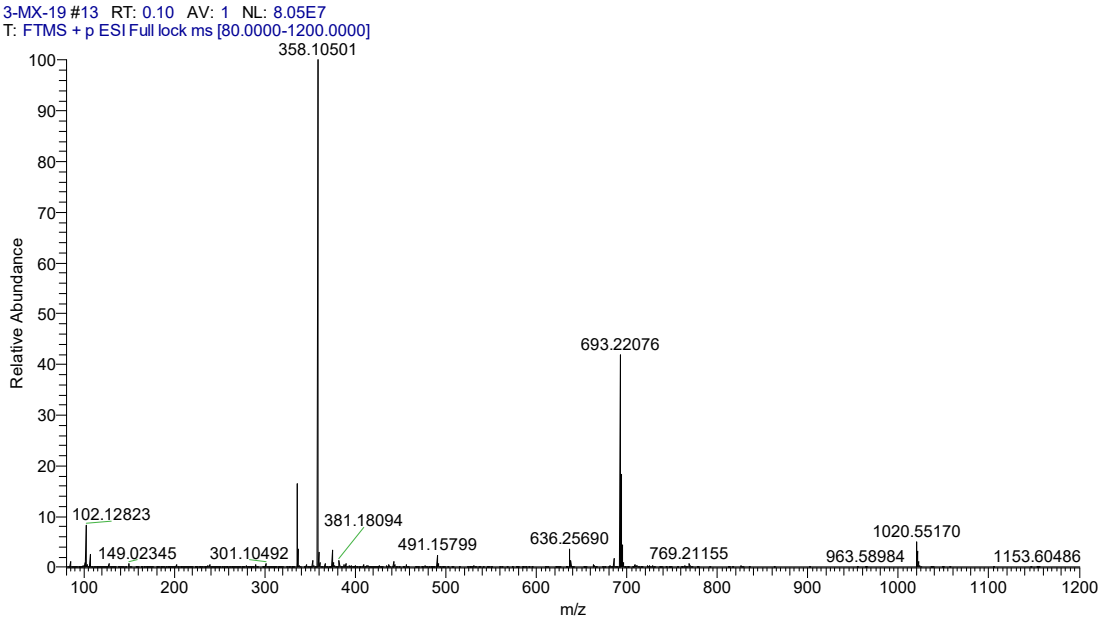
Compound Spectra



Spectrum Peaks

m/z	m/z (Calc)	Diff (ppm)	Abund	Height %	Height % (Calc)	Ion Species	Z
474.1663	474.1660	0.77	4446637	100.00	100.00	(M+H)+	1
475.1696	475.1691	1.09	1255418	28.23	29.72	(M+H)+	1
476.1723	476.1718	1.12	217840	4.90	5.50	(M+H)+	1
477.1760	477.1744	3.36	31387	0.71	0.76	(M+H)+	1
496.1482	496.1479	0.61	3486447	100.00	100.00	(M+Na)+	1
497.1514	497.1511	0.70	942748	27.04	29.71	(M+Na)+	1
498.1542	498.1537	1.01	173778	4.98	5.49	(M+Na)+	1
499.1569	499.1564	1.15	20192	0.58	0.76	(M+Na)+	1

5


3-MX-19 #13 RT: 0.10 AV: 1 NL: 8.05E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
Relative Abundance
m/z
358.10501
693.22076
102.12823
149.02345
301.10492
381.18094
491.15799
636.25690
769.21155
963.58984
1020.55170
1153.60486