A Quinine-Squaramide Catalyzed Enantioselective Vinylogous Mannich Reaction between Benzothiazolimines and γ-Butenolides for Stout Preparation of Chiral N-Benzothiazol Butyrolactones

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1. General experimental information

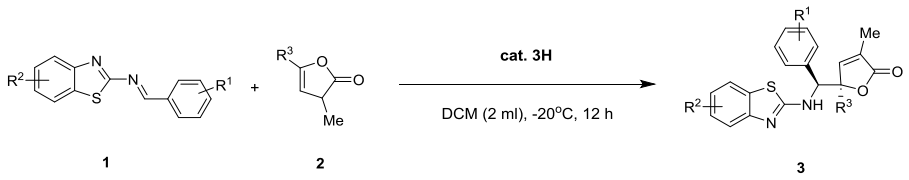
Chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Reactions were monitored by TLC and visualized with ultraviolet light. Flash column chromatography was performed on silica gels (300-400 mesh) eluting with ethyl acetate, dichloromethane and petroleum ether. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker Avance instrument (400 MHz for ^{1}H NMR, 101 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 ($CDCl_3$ at 7.26 ppm), chemical shift, multiplicity (s = singlet, d =doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR chemical shifts arereported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$ at 77.1 ppm). High-resolution mass spectra (HRMS) analyses were obtained with the Thermo ScientificLTQ Orbitrap XL mass spectrometer and 1290 Infinity LC/6460 QQQMS. Enantiomeric excess was determined by HPLC analysis on chiralpak AD-H, IC, Optical rotations were measured on a Perkin-Elmer 241 Polarimeter. Melting points were recorded on a Buchi Melting Point B-545.

2. General procedure for the syntheses of starting materials.

Benzothiazolimines **1a-q** were prepared according to known procedures [1]. Various 3-methyl-5-arylfuran-2(3H)-ones **2a-i** were prepared as reported methods [2,3].

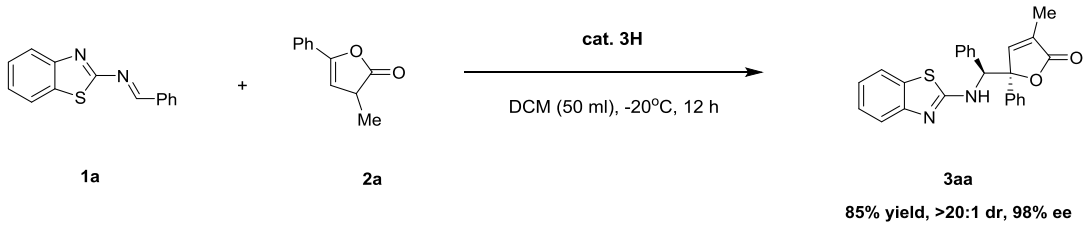
Reference
[1].Q.-J. Ni, X.-X. Song, J.-W. Xiong, G. Raabe, and D. Enders. *Chem. Commun.*, 2015, 51, 1263.
[2]. S. Žari, M. Kudrjashova, T. Pehk, M. Lopp and T. Kanger, *Org. Lett.*, 2014, 16, 1740.
[3]. D. J. Jones, and V. C. Gibson, *Heterocycles*, 2006, 68, 1128.

3. General procedure for the syntheses of compounds 3aa-3qa, 3ab-3ai.



In a vial equipped with a magnetic stirring bar, compound **2** (0.24 mmol, 1.2 equiv), catalyst **3H** (10 mol%) were dissolved in CH_2Cl_2 (2 mL), and the mixture was stirred at -20 ℃ for 10 min. Compound **1** (0.20 mmol, 1.0 equiv) was then added to the reaction vial. After completion of the reaction as indicated by TLC (PE/EA= 3/1), the solvent was evaporated and the crude product was directly purified by flash column chromatography using petroleum ether/ethyl acetate [V (PE/EA= 10/1 to 3/1] as eluent.

4. General procedure for scale-up preparation of 3aa

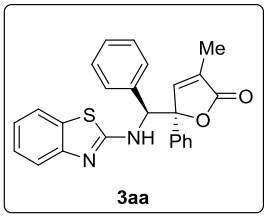


In a vial equipped with a magnetic stirring bar, compound **2a** (1.05g, 6.05 mmol, 1.2 equiv) and catalyst **3H** (10 mol%) were dissolved in CH_2Cl_2 (50 mL, 0.1 M). The mixture was stirred at -20 ℃ for 10 min. Compound **1a** (1.20g, 5.04 mmol, 1.0 equiv) was added to the reaction via. After completion of the reaction as indicated by TLC ((PE/EA= 3/1), the solvent was evaporated

and the crude product was directly purified by flash column chromatography using petroleum ether/ethyl acetate [V(PE/EA= 10/1 to 3/1] as eluent. The product **3aa** (1.76 g, 85% yield, >20:1 dr, 98% ee) was obtained as white solid.

5. Characterization data

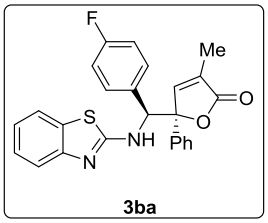
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3aa)



3aa

white solid, 71.7 mg, 87% yield; m.p.:150.3-151.1°C, >20:1 dr, 98% ee, $[\alpha]_D^{20}$= +72.7 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 6.395 min, t_{minor} = 5.473 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 8.0, 3.2 Hz, 4H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.37–7.32 (m, 6H), 7.31– 7.21 (m, 3H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.21 (brs, 1H), 5.53 (s, 1H), 1.69 (s, 3H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.7, 151.9, 149.6, 136.7, 135.9, 130.8, 129.7, 129.0, 128.7, 128.6, 128.5, 128.1, 125.9, 125.5, 125.1, 121.9, 120.9, 120.7, 119.4, 89.9, 65.3, 10.3. HRMS (ESI) m/z calcd for $C_{25}H_{20}N_2O_2SNa^+$ $[M+Na]^+$ 435.1138, found 435.1135.

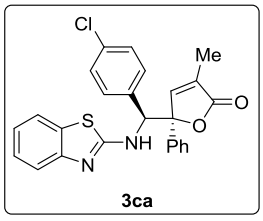
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(4-fluorophenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one(3ba)



3ba

white solid, 76.5 mg, 89% yield; m.p.:122.5-123.0°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +64.5 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 6.838 min, t_{minor} = 5.993 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 4H), 7.37 (t, *J* = 7.2 Hz, 2H), 7.34 – 7.26 (m, 3H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.18 (s, 1H), 7.06 – 6.98 (m, , 3H), 6.01 (s, 1H), 5.51 (s, 1H), 1.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 165.3, 162.7(d, *J* = 249.5 Hz), 151.9, 149.6, 136.5, 131.9, 130.8, 129.8 (d, *J* = 8.1 Hz), 129.7, 129.1, 128.8 126.0, 125.4, 122.1, 120.8, 119.5, 115.5(d, *J* = 21.6 Hz), 89.7, 64.4, 10.4.^{19}F NMR (376 MHz, $CDCl_3$) δ -112.73. HRMS (ESI) m/z calcd for $C_{25}H_{19}FN_2O_2SNa^+$ $[M+Na]^+$ 453.1043, found 453.1040.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(4-chlorophenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ca)

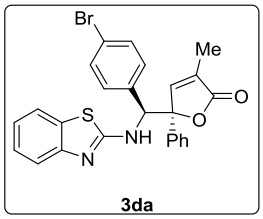


3ca

white solid, 77.6 mg, 87% yield; m.p.:132.6-133.4°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +71.2 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 5.620 min, t_{minor} = 5.093 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 2H), 7.46 – 7.40 (m, 2H), 7.39 – 7.25 (m, 8H), 7.17 (d, *J* = 1.7 Hz, 1H), 7.08 (td, *J* = 7.6, 1.2 Hz, 1H), 6.02 (d, *J* = 8.8 Hz, 1H), 5.53 (d, *J* = 7.6 Hz, 1H), 1.69 (d, *J* = 1.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.2, 165.4, 149.2, 135.7, 135.4, 134.8, 129.9, 129.1, 128.7, 128.6, 127.9, 126.9, 126.0, 122.1, 120.8, 119.5, 89.5, 64.9, 10.3. HRMS (ESI) m/z calcd for $C_{25}H_{19}ClN_2O_2SNa^+$ $[M+Na]^+$ 469.0748,

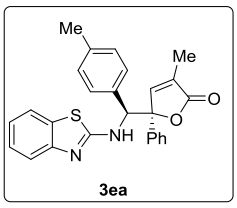
471.0718 , found 469.0744, 471.0710.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(4-bromophenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3da)



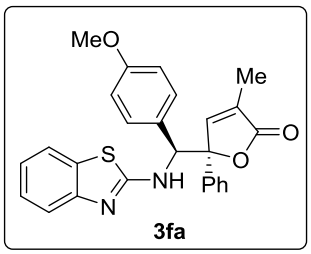
white solid, 81.3 mg, 83% yield; m.p.:123.8-124.6°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +52.2 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 8.995 min, t_{minor} = 7.383 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ7.47 – 7.42 (m, 6H), 7.36 (d, *J* = 7.6 Hz, 2H), 7.34 – 7.29 (m, 1H), 7.24 (td, *J* = 7.6, 1.2 Hz, 1H), 7.21 – 7.16 (m, 3H), 7.05 (td, *J* = 7.6, 1.2 Hz, 1H), 6.03 (s, 1H), 5.51 (s, 1H), 1.71 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 165.2, 151.9, 149.5, 136.4, 135.2, 131.6, 130.8, 129.9, 129.7, 129.1, 128.9, 126.0, 125.4, 122.6, 122.1, 120.8, 119.5, 89.5, 64.5, 10.4. HRMS (ESI) m/z calcd for $C_{25}H_{19}BrN_2O_2SNa^+$ $[M+Na]^+$513.0243 , 515.0222, found 513.0242, 515.0219

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(p-tolyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ea)



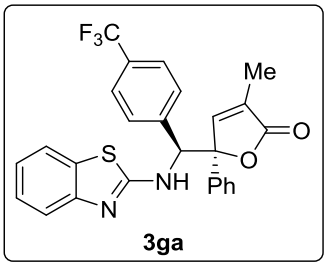
white solid, 74.1 mg, 87% yield; m.p.:151.5-152.2°C, >20:1 dr, 97% ee, $[\alpha]_D^{20}$= +34.6 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 5.647 min, t_{minor} = 4.907 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.45 (m, 4H), 7.43 – 7.36 (m, 2H), 7.36 – 7.31 (m, 1H), 7.29 – 7.23 (m, 1H), 7.23 – 7.16 (m, 3H), 7.14-7.12 (m, 2H), 7.10 – 7.01 (m, 1H), 6.12 – 5.96 (m, 1H), 5.45 (d, *J* = 6.8 Hz, 1H), 2.33 (s, 3H), 1.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.6, 165.7, 152.0, 149.7, 138.4, 136.8, 132.8, 130.8, 129.6, 129.2, 128.9, 128.7, 127.9, 125.9, 125.4, 121.9, 120.7, 119.4, 89.9, 65.1, 21.2, 10.4.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 449.1294, found 449.1293.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(4-methoxyphenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3fa)



white solid, 70.7 mg, 80% yield; m.p.:173.2-174.5°C, >20:1 dr, 98% ee, $[\alpha]_D^{20}$= +17.5 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15.558 min, t_{minor} = 13.403 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.43 (m, 4H), 7.42 – 7.30 (m, 3H), 7.29 – 7.18 (m, 4H), 7.10 – 7.02 (m, 1H), 6.91 – 6.80 (m, 2H), 5.39 (s, 1H), 3.80 (s, 3H), 1.74 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 166.1, 159.7, 151.6, 149.4, 136.8, 130.5, 129.9, 129.3, 128.9, 128.7, 127.7, 125.9, 125.5, 121.9, 120.8, 119.2, 113.8, 90.0, 65.1, 55.3, 10.5.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_3SNa^+$ $[M+Na]^+$ 465.1243, found 465.1242

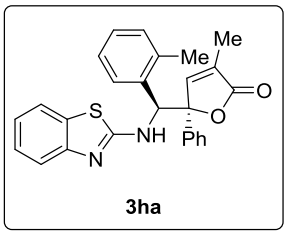
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(4-(trifluoromethyl)phenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ga)



white solid, 78.7 mg, 82% yield; m.p.:140.5-141.3°C, >20:1 dr, 97%

ee, $[\alpha]_D^{20}$= +23.8 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 80/20, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 18.473 min, t_{minor} = 8.505 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.50 – 7.44 (m, 6H), 7.41 – 7.31 (m, 3H), 7.26 – 7.18 (m, 2H), 7.05 (td, *J* = 7.6, 1.2 Hz, 1H), 6.09 (brs, 1H), 5.66 (s, 1H), 1.67 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.2, 165.0, 151.8, 149.4, 140.4, 136.3, 130.9, 130.7 (t, *J* = 32.9 Hz), 130.0, 129.1, 129.0(t, *J* = 16.6 Hz), 128.5, 126.0, 125.4 (d, *J* = 7.5 Hz), 125.3, 122.2, 120.8, 119.5, 89.5, 64.5, 10.4. ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.34. HRMS (ESI) m/z calcd for $C_{26}H_{19}F_3N_2O_2SNa^+$ $[M+Na]^+$ 503.1012, found 503.1010.

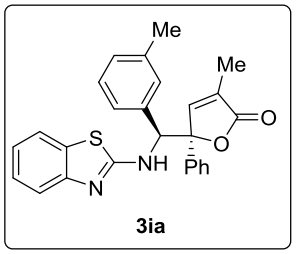
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(o-tolyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ha)



3ha

white solid, 65.6 mg, 77% yield; m.p.:155.2-156.4°C, >20:1 dr, 84% ee, $[\alpha]_D^{20}$= -37.4 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 4.502 min, t_{minor} = 4.017 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.40 (m, 4H), 7.39 – 7.30 (m, 3H), 7.25 – 7.20 (m, 3H), 7.18 – 7.15 (m, 3H), 7.06 – 6.98 (m, 1H), 6.04 (brs, 1H), 5.76 (s, 1H), 2.50 (s, 3H), 1.69 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 165.7, 152.1, 148.3, 136.9, 136.3, 134.6, 130.9, 130.6, 130.3, 128.9, 128.7, 128.4, 126.6, 126.5, 125.9, 125.5, 121.9, 120.7, 119.4, 90.1, 60.8, 20.0, 10.4.
. HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 449.1294, found 449.1293

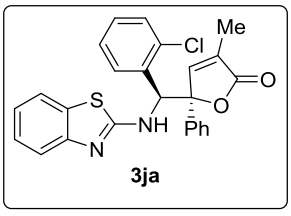
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(m-tolyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one 3ia)



3ia

white solid, 74.1 mg, 87% yield; m.p.:170.2-171.8°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +102.2 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 15.470 min, t_{minor} = 10.515 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.45 (m, 4H), 7.41 – 7.33 (m, 2H), 7.31 (d, *J* = 6.8 Hz, 1H), 7.24 – 7.15 (m, 3H), 7.12 – 6.97 (m, 4H), 6.07 (s, 1H), 5.43 (s, 1H), 2.32 (s, 3H), 1.67 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.8, 152.0, 149.6, 138.2, 136.8, 135.8, 130.8, 129.6, 129.3, 128.9, 128.7, 128.3, 125.9, 125.5, 125.1, 121.9, 120.7, 119.4, 89.9, 65.3, 21.5, 10.4. HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 449.1294, found 449.1293

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(2-chlorophenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ja)

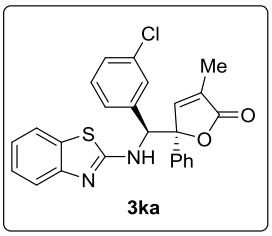


3ja

white solid, 73.1 mg, 82% yield; m.p.:141.3-142.4°C, >20:1 dr, 76% ee, $[\alpha]_D^{20}$= +46.4 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 3.492 min, t_{minor} = 3.722 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.65 – 7.62 (m, 2H), 7.58 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.43 – 7.32 (m, 3H), 7.32 – 7.27 (m, 3H), 7.25 – 7.11 (m, 4H), 6.37 (s, 1H), 1.86 (d, *J* = 1.6 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 165.8,

152.0, 150.9, 136.1, 134.4, 134.4, 130.9, 129.8, 129.5, 129.5, 129.2, 128.4, 128.4, 127.1, 126.0, 125.8, 122.1, 120.9, 119.6, 91.1, 59.5, 10.5. HRMS (ESI) m/z calcd for $C_{25}H_{19}ClN_2O_2SNa^+$ $[M+Na]^+$ 469.0748, 471.0718 , found 469.0744, 471.0710

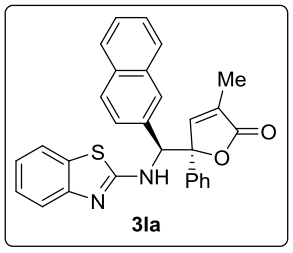
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(3-chlorophenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ka)



3ka

white solid, 77.6 mg, 87% yield; m.p.:135.7-136.5 °C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= -30.5 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 23.815 min, t_{minor} = 15.398 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 13.0, 6.8 Hz, 4H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.22 (m, 7H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.01 (s, 1H), 5.56 (s, 1H), 1.73 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 165.1, 151.9, 149.4, 138.3, 136.4, 134.4, 130.8, 129.9, 129.8, 129.1, 128.9, 128.8, 128.4, 126.0, 126.0, 125.4, 122.1, 120.8, 119.5, 89.6, 64.5, 10.4.HRMS (ESI) m/z calcd for $C_{25}H_{19}ClN_2O_2SNa^+$ $[M+Na]^+$ 469.0748, 471.0718 , found 469.0744, 471.0710

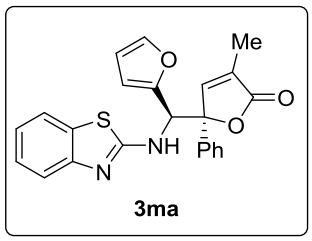
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(naphthalen-2-yl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3la)



3la

white solid, 81.3 mg, 88% yield; m.p.:183.6-184.5°C, >20:1 dr, 97% ee, $[\alpha]_D^{20}$= +92.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 28.315 min, t_{minor} = 16.765 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ7.87 – 7.79 (m, 4H), 7.56 – 7.47 (m, 6H), 7.46 – 7.31 (m, 4H), 7.30 (d, *J* = 3.6 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.18 (d, *J* = 8.4 Hz, 1H), 5.68 (d, *J* = 8.0 Hz, 1H), 1.63 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 165.6, 151.9, 149.5, 136.8, 133.4, 133.2, 132.9, 130.8, 129.7, 129.0, 128.8, 128.3, 128.1, 127.7, 127.6, 126.5, 126.5, 125.9, 125.5, 125.3, 121.9, 120.7, 119.4, 90.0, 65.4, 10.4.HRMS (ESI) m/z calcd for $C_{29}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 485.1294, found 485.1296.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(furan-2-yl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3ma)

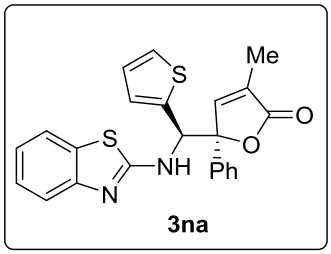


3ma

light yellow solid, 68.3 mg, 85% yield; m.p.:122.8-123.9 °C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +25.8 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 20.057 min, t_{minor} = 17.775 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (t, *J* = 7.2 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 2H), 7.39 – 7.34 (m, 4H), 7.32 – 7.24 (m, 2H), 7.10 (t, *J* = 7.6 Hz, 1H), 6.32 (s, 2H), 5.77 (s, 2H), 1.85 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.4, 151.9, 149.5, 148.5, 142.5, 136.4, 130.8, 130.0, 128.8, 128.7, 125.9, 125.5, 122.2, 120.8, 119.5, 110.7, 109.5, 89.7, 59.3, 10.5. HRMS (ESI) m/z calcd for $C_{23}H_{18}N_2O_3SNa^+$ $[M+Na]^+$425.0930, found 425.0931.

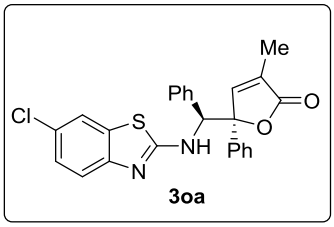
(*S*)-5-((*R*)-(benzo[d]thiazol-2-ylamino)(thiophen-2-yl)methyl)-3-methyl-5-phenylfuran-2(5H)

-one (3na)



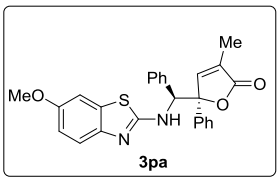
light yellow solid, 72.7 mg, 87% yield; m.p.:128.5-130.1°C, >20:1 dr, $[\alpha]_D^{20}$= +31.9 (c. 1.00, $CHCl_3$); 99% ee, The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 26.125 min, t_{minor} = 17.907 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, *J* = 7.2 Hz, 4H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.34 – 7.20 (m, 4H), 7.09 (d, *J* = 6.0 Hz, 2H), 7.01 – 6.85 (m, 1H), 6.03 (brs, 1H), 5.79 (s, 1H),, 1.79 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.7, 165.3, 151.9, 149.3, 139.1, 136.6, 130.9, 130.0, 128.9, 128.8, 127.3, 126.8, 125.9, 125.8, 125.5, 122.1, 120.8, 119.5, 89.9, 60.7, 10.5. HRMS (ESI) m/z calcd for $C_{23}H_{18}N_2O_2S_2Na^+$ $[M+Na]^+$ 441.0702, found 441.0702.

(*S*)-5-((*S*)-((6-chlorobenzo[d]thiazol-2-yl)amino)(phenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3oa)



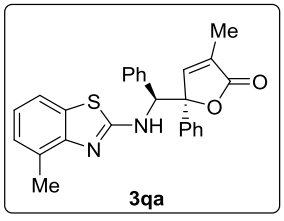
white solid, 76.7 mg, 86% yield; m.p.:175.4-176.8 °C, 7:1 dr, 99% ee, $[\alpha]_D^{20}$= +37.4 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 23.312 min, t_{minor} = 12.643 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.38 (m, 4H), 7.37 – 7.25 (m, 8H), 7.22 – 7.12 (d, *J* = 7.3 Hz, 2H), 6.20 (brs, 1H), 5.52 (s, 1H), 1.65 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.8, 150.6, 149.5, 136.7, 135.8, 132.0, 129.7, 129.0, 128.8, 128.7, 128.5, 128.0, 127.0, 126.3, 125.4, 120.4, 120.0, 89.9, 65.2, 10.3.HRMS (ESI) m/z calcd for $C_{25}H_{19}ClN_2O_2SNa^+$ $[M+Na]^+$ 469.0748, 471.0718 , found 469.0744, 471.0710

(*S*)-5-((*S*)-((6-methoxybenzo[d]thiazol-2-yl)amino)(phenyl)methyl)-3-methyl-5-phenylfuran-2(5H)-one (3pa)



white solid, 70.7 mg, 80% yield; m.p.:168.8-170.1°C, >20:1 dr, 98% ee, $[\alpha]_D^{20}$= +61.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 5.715 min, t_{minor} = 5.982 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 7.6, 1.6 Hz, 2H), 7.35 (q, *J* = 8.8, 7.6 Hz, 4H), 7.31 – 7.28 (m, 4H), 7.19 (d, *J* = 1.6 Hz, 1H), 7.01 (d, *J* = 2.4 Hz, 1H), 6.83 (dd, *J* = 8.8, 2.4 Hz, 1H), 5.89 (s, 1H), 5.45 (d, *J* = 3.6 Hz, 1H), 3.76 (s, 3H), 1.66 (d, *J* = 1.6 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 163.9, 155.4, 149.7, 146.2, 136.8, 136.1, 131.8, 129.6, 128.9, 128.7, 128.5, 128.4, 128.0, 125.5, 119.7, 113.6, 105.2, 89.9, 65.2, 55.9, 10.3.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_3SNa^+$ $[M+Na]^+$ 465.1243, found 465.1242.

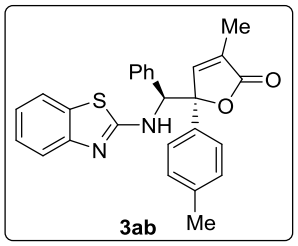
(*S*)-3-methyl-5-((*S*)-((4-methylbenzo[d]thiazol-2-yl)amino)(phenyl)methyl)-5-phenylfuran-2(5H)-one (3qa)



white solid, 68.2 mg, 80% yield; m.p.:164.7-165.4°C, >20:1 dr, 14% ee, $[\alpha]_D^{20}$= -24.8 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 3.657 min, t_{minor} = 3.982 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 1.6 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.32 –

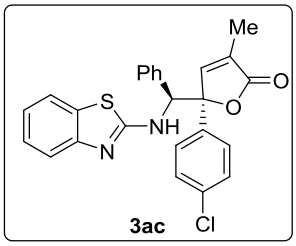
7.30 (m, 4H), 7.29 – 7.19 (m, 6H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 7.6 Hz, 1H), 5.80 (dd, *J* = 12.0, 8.4 Hz, 1H), 5.68 (dd, *J* = 8.4, 2.8 Hz, 1H), 2.69 (s, 3H), 1.85 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.5, 164.9, 150.9, 137.1, 136.0, 130.6, 129.2, 128.8, 128.7, 128.6, 128.2, 128.2, 126.7, 125.4, 122.0, 118.4, 91.3, 64.9, 18.5, 10.6.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 449.1294, found 449.1293.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-3-methyl-5-(p-tolyl)furan-2(5H)-one (3ab)



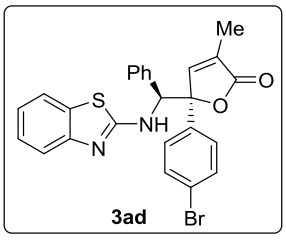
white solid, 77.5 mg, 91% yield; m.p.:158.3-159.2^{o}C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +72.6 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 4.290 min, t_{minor} = 3.882 min);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.34– 7.30 ((m, 5H), 7.29 – 7.23 (m, 1H), 7.24 – 7.17 (m, 3H), 7.09 – 7.03 (m, 1H), 5.48 (s, 1H), 2.34 (s, 3H), 1.68 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.6, 165.8, 151.9, 149.8, 138.7, 136.0, 133.7, 130.8, 129.7, 129.4, 128.5, 128.4, 128.1, 125.9, 125.4, 121.9, 120.7, 119.3, 89.8, 65.3, 21.1, 10.3.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 449.1294, found 449.1293

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(4-chlorophenyl)-3-methylfuran-2(5 H)-one (3ac)



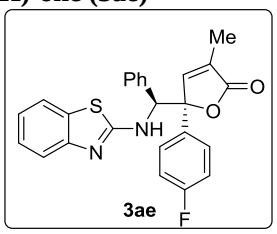
white solid, 76.7.3 mg, 90% yield; m.p.:152.6-153.9 ^{o}C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +57.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 4.300 min, t_{minor} = 3.902 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.48 (m, 4H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.34 – 7.25 (m, 6H), 7.17 (d, *J* = 2.0 Hz, 1H), 7.07 (t, *J* = 7.6 Hz, 1H), 6.19 (d, *J* = 8.0 Hz, 1H), 5.53 (d, *J* = 5.2 Hz, 1H), 1.69 (d, *J* = 1.6 Hz, 3H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 165.5, 151.9, 149.2, 136.0, 135.8, 132.0, 130.8, 129.9, 128.7, 128.5, 128.0, 127.2, 125.9, 122.9, 122.0, 120.8, 119.5, 89.6, 64.8, 10.3. HRMS (ESI) m/z calcd for $C_{25}H_{19}ClN_2O_2SNa^+$ $[M+Na]^+$ 469.0748, 471.0718 , found 469.0744, 471.0710

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(4-bromophenyl)-3-methylfuran-2(5 H)-one (3ad)



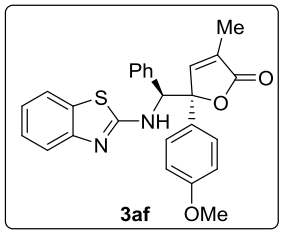
white solid, 85.3 mg, 87% yield; m.p.:142.6-143.3^{o}C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +40.5 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 8.798 min, t_{minor} = 7.190 min); ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 2.0 Hz, 1H), 7.46 (dd, *J* = 7.2, 2.0 Hz, 2H), 7.37 (d, *J* = 2.8 Hz, 1H), 7.36 (d, *J* = 2.8 Hz, 1H), 7.32 – 7.27 (m, 8H), 7.17 (d, *J* = 1.6 Hz, 1H), 6.21 (brs, 1H), 5.49 (s, 1H), 1.64 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.9, 150.9, 149.4, 136.7, 135.7, 132.4, 130.1, 129.7, 129.1, 129.0, 128.8, 128.7, 128.5, 128.1, 128.0, 125.4, 125.1, 123.2, 120.4, 114.3, 104.1, 89.8, 65.3, 10.3. HRMS (ESI) m/z calcd for $C_{25}H_{19}BrN_2O_2SNa^+$ $[M+Na]^+$ 513.0243 , 515.0222, found 513.0242, 515.0219

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(4-fluorophenyl)-3-methylfuran-2(5H)-one (3ae)



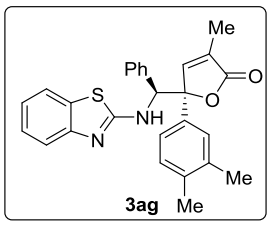
white solid, 73.9 mg, 86% yield; m.p.:135.3-136.7°C, >20:1 dr, 96% ee, $[\alpha]_D^{20}$= +50.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 11.520 min, t_{minor} = 9.752 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.41 (m, 4H), 7.32 – 7.26 (m, 5H), 7.24– 7.22 (m, 1H), 7.19 (s, 1H), 7.07 –7.01 (m, 3H), 6.19 (brs, 1H), 5.53 (d, *J* = 5.2 Hz, 1H), 1.68 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 165.6, 162.7(d, *J* = 249.7 Hz), 151.9, 149.4, 135.9, 132.7, 132.6, 130.8, 129.9, 128.6, 128.5, 128.0, 127.5(d, *J* = 8.3 Hz), 125.9, 122.0, 120.8, 119.4, 115.9(d, *J* = 20.7 Hz), 89.6, 65.1, 10.4.^{19}F NMR (376 MHz, CDCl3) δ -112.70. HRMS (ESI) m/z calcd for $C_{25}H_{19}FN_2O_2SNa^+$ $[M+Na]^+$ 453.1043, found 453.1040.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(4-methoxyphenyl)-3-methylfuran-2(5H)-one (3af)



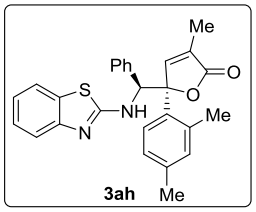
white solid, 77.8 mg, 88% yield; m.p.:157.1-158.4°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +26.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 6.527 min, t_{minor} = 6.072 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.46 (m, 2H), 7.36 (d, *J* = 8.8 Hz, 2H), 7.32 – 7.27 (m, 5H), 7.24 (s, 1H), 7.19 (d, *J* = 1.7 Hz, 1H), 7.04 (td, *J* = 7.6, 1.2 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.11 (d, *J* = 8.4 Hz, 1H), 5.44 (d, *J* = 6.4 Hz, 1H), 3.77 (s, 3H), 1.67 (d, *J* = 1.6 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 172.6, 165.7, 159.8, 152.0, 149.8, 136.0, 130.8, 129.4, 128.5, 128.4, 128.1, 126.9, 125.9, 121.9, 120.7, 119.4, 114.3, 89.7, 65.2, 55.3, 10.3.HRMS (ESI) m/z calcd for $C_{26}H_{22}N_2O_3SNa^+$ $[M+Na]^+$ 465.1243, found 465.1242.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(3,4-dimethylphenyl)-3-methylfuran-2(5H)-one (3ag)



white solid, 72.2 mg, 82% yield; m.p.:163.5-164.8°C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +80.2 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 5.873 min, t_{minor} = 4.990 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.39 –7.35(m, 4H), 7.33 –7.28 (m, 2H) 7.19 –7.14 (m, 2H), 7.07 (d, *J* = 7.2 Hz, 1H), 6.09 (d, *J* = 8.0 Hz, 1H), 5.45 (d, *J* = 6.4 Hz, 1H), 2.27 (s, 3H) 2.24 (s, 3H), 1.66 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.7, 165.8, 152.0, 150.0, 137.4, 137.3, 136.0, 134.0, 130.8, 130.2, 129.2, 128.5, 128.4, 128.1, 126.5, 125.9, 122.7, 121.8, 120.7, 119.3, 89.7, 65.3, 20.0, 19.4, 10.3.HRMS (ESI) m/z calcd for $C_{27}H_{24}N_2O_2SNa^+$ $[M+Na]^+$ 463.1451, found 463.1450.

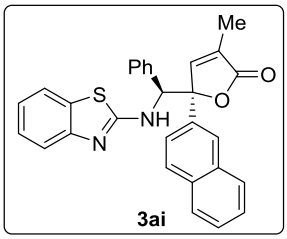
(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-5-(2,4-dimethylphenyl)-3-methylfuran-2(5H)-one (3ah)



white solid, 70.4 mg, 80% yield m.p.:172.3-174.1°C, >20:1 dr, 94% ee, $[\alpha]_D^{20}$= -52.7 (c. 1.00, $CHCl_3$); The ee value was determined by

HPLC (Chiralpak IC, n-hexane/isopropanol = 85/15, flow rate 1.0 mL/min, λ = 254 nm, tmajor = 5.073 min, tminor = 4.573 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.53 (m, 2H), 7.44 – 7.39 (m, 2H), 7.31 – 7.27 (m, 5H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.09 (s, 1H), 7.00 (s, 2H), 5.61 (s, 1H), 5.08 (s, 1H), 2.33 (s, 3H), 2.17 (s, 3H), 1.67 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.0, 166.5, 152.4, 150.7, 139.0, 135.6, 135.4, 130.9, 129.8, 129.3, 128.1, 127.6, 127.4, 127.1, 127.1, 125.7, 125.1, 123.7, 121.0, 119.9, 118.3, 106.7, 63.8, 53.9, 20.2, 20.1.HRMS (ESI) m/z calcd for C27H24N2O2SNa+ [M+Na]+ 463.1451, found 463.1450.

(*S*)-5-((*S*)-(benzo[d]thiazol-2-ylamino)(phenyl)methyl)-3-methyl-5-(naphthalen-2-yl)furan-2(5H)-one (3ai)



white solid, 81.3 mg, 88% yield; m.p.:192.4-193.5^{o}C, >20:1 dr, 99% ee, $[\alpha]_D^{20}$= +51.3 (c. 1.00, $CHCl_3$); The ee value was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 95/5, flow rate 1.0 mL/min, λ = 254 nm, t_{major} = 13.865 min, t_{minor} = 11.890 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.90 – 7.81 (m, 3H), 7.59 – 7.50 (m, 3H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.39 – 7.31 (m, 6H), 7.26 (s, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.23 (s, 1H), 5.64 (s, 1H), 1.71 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 165.7, 149.6, 135.9, 134.0, 133.1, 130.6, 129.8, 128.9, 128.6, 128.5, 128.3, 128.1, 127.7, 126.9, 126.8, 125.9, 124.9, 122.7, 122.0, 120.7, 119.3, 90.0, 65.2, 10.4. HRMS (ESI) m/z calcd for $C_{29}H_{22}N_2O_2SNa^+$ $[M+Na]^+$ 485.1294, found 485.1296.

6.X-ray diffraction parameters and data for 3ac

Table S1: Important crystal data of compound **3ac**

Empirical formula	C51H40Cl4N4O4S2	
Formula weight	978.79	
Temperature/K	293(2)	
Crystal system	monoclinic	
Space group	P21	
a/Å	15.8968(6)	
b/Å	11.2913(4)	
c/Å	16.5092(6)	
α / °	90	
β / °	109.741(4)	
γ / °	90	
Volume/ Å3	2789.17(19)	
Z	2	
ρ calcg/cm3	1.165	
μ /mm−1	2.97	
F(000)	1012	
Crystal size/mm3	0.17 × 0.1 × 0.08	
Radiation	CuKα	(λ = 1.54184)
2Θ range for data collection/°	9.49 to 140.838	

Index ranges	-14 ⩽ h ⩽ 19, -13 ⩽ k ⩽ 10, -20 ⩽ l ⩽ 19
Reflections collected	24007
Independent reflections	9306 [Rint = 0.0461, Rsigma = 0.0588]
Data/restraints/parameters	9306/67/556
Goodness-of-fit on F2	1.027
Final R indexes [I>=2σ (I)]	R1 = 0.0890, wR2 = 0.2295
Final R indexes [all data]	R1 = 0.1093, wR2 = 0.2561
Largest diff. peak/hole / e Å-3	0.60/-0.27
Flack parameter	-0.01(2)

Single crystal of **3ac** was obtained by slow evaporation from PE/DCM (V=4/1) at 25 °C. ORTEP diagram of compound **3ac**, the ellipsoid contour probability levels: 50%

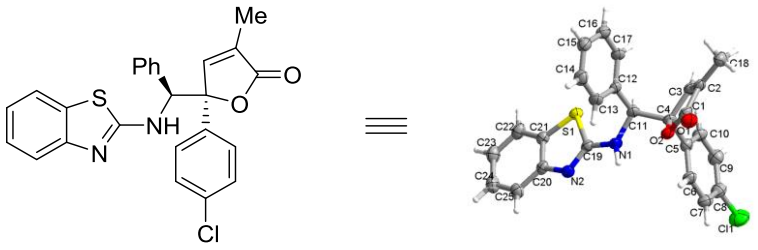
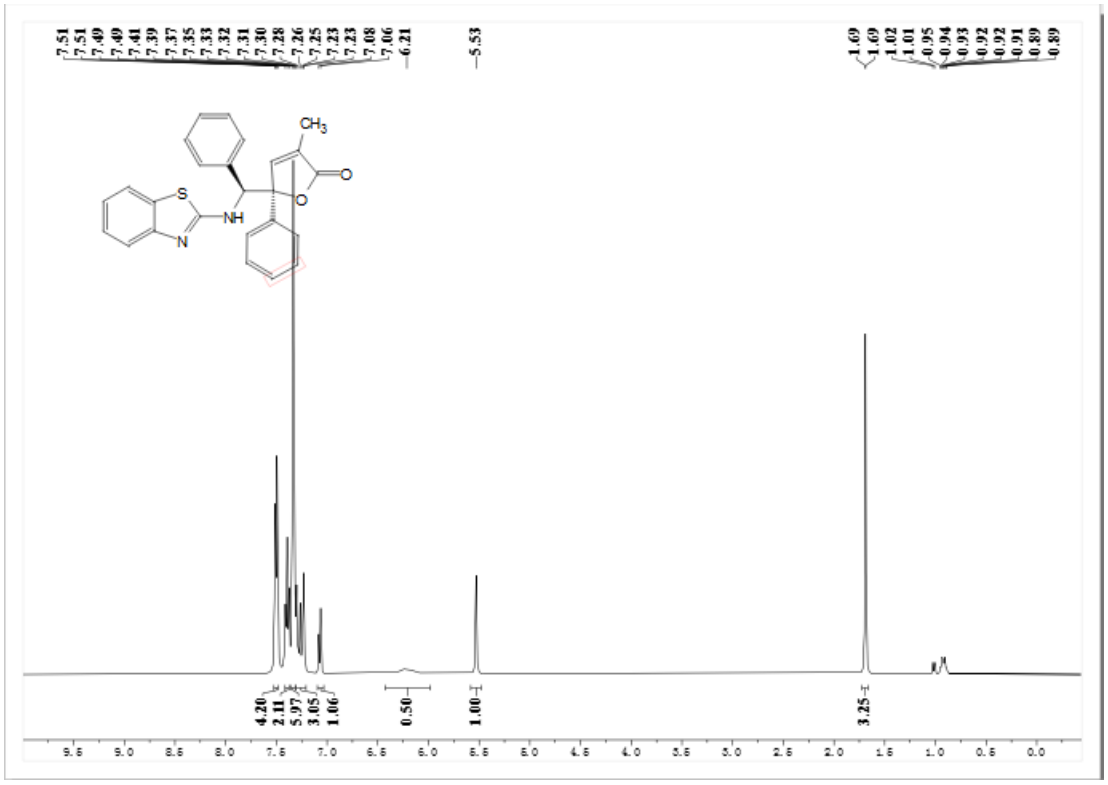


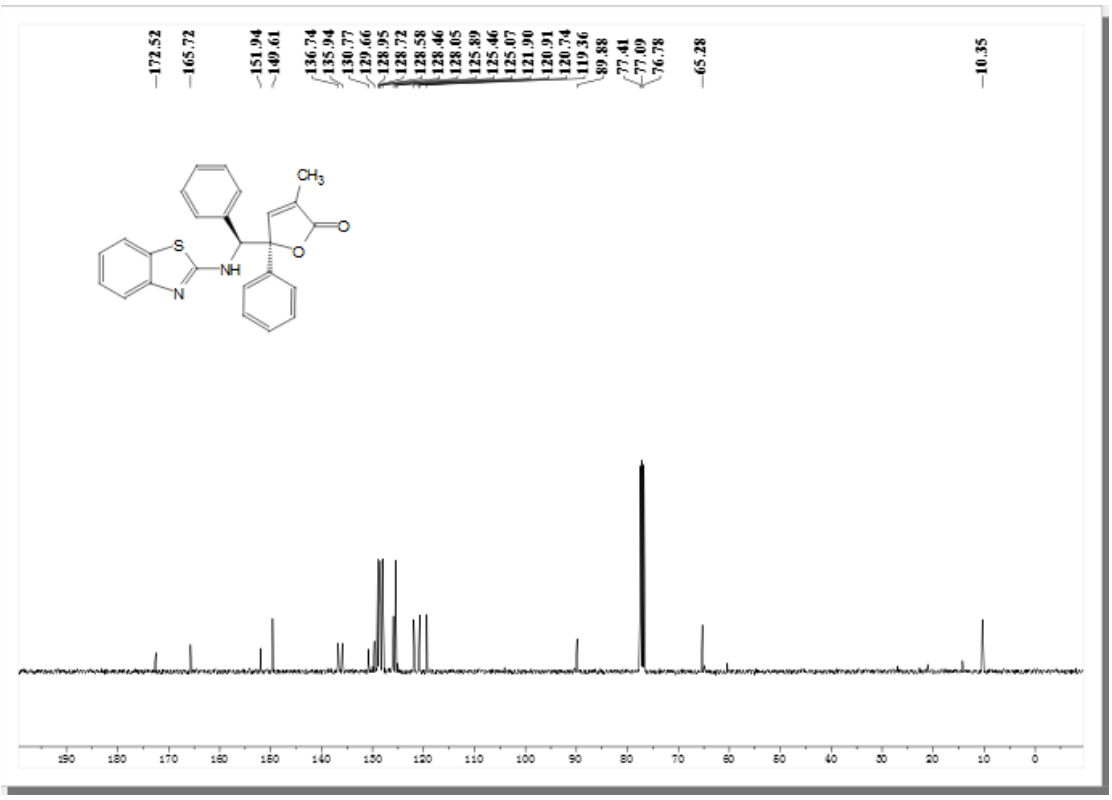
Figure S1. ORTEP plot of compound **3ac**
Thermal ellipsoids are drawn at 50% probability level.

7. HPLC, NMR spectra of products

7.1 NMR spectra

3aa





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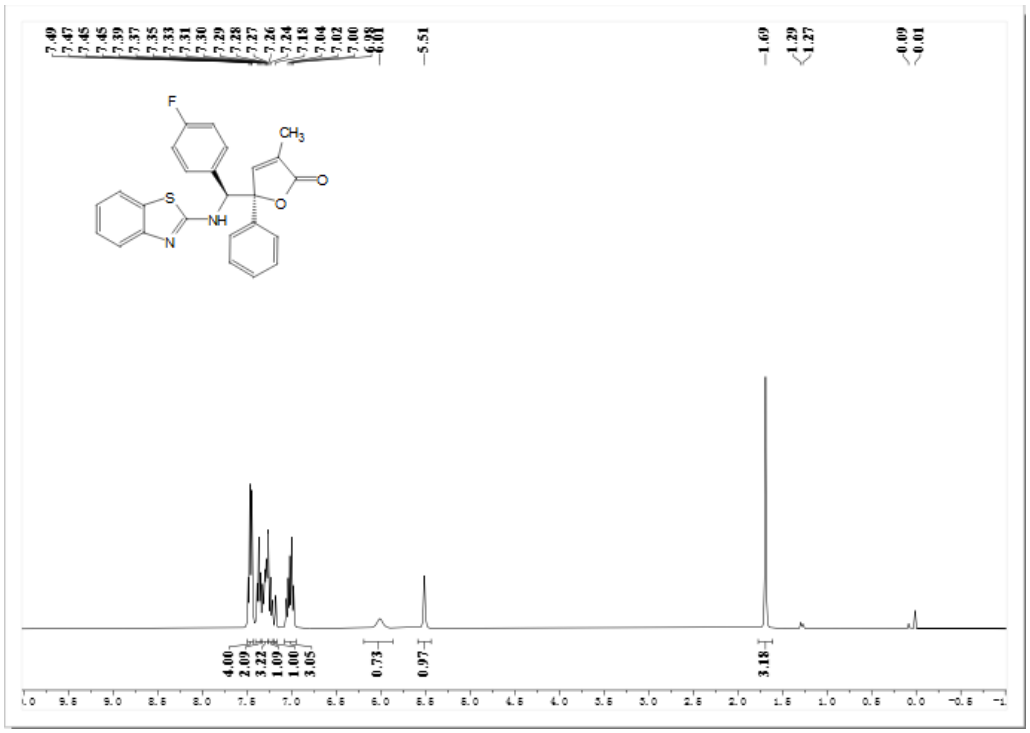
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6.01
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0.01
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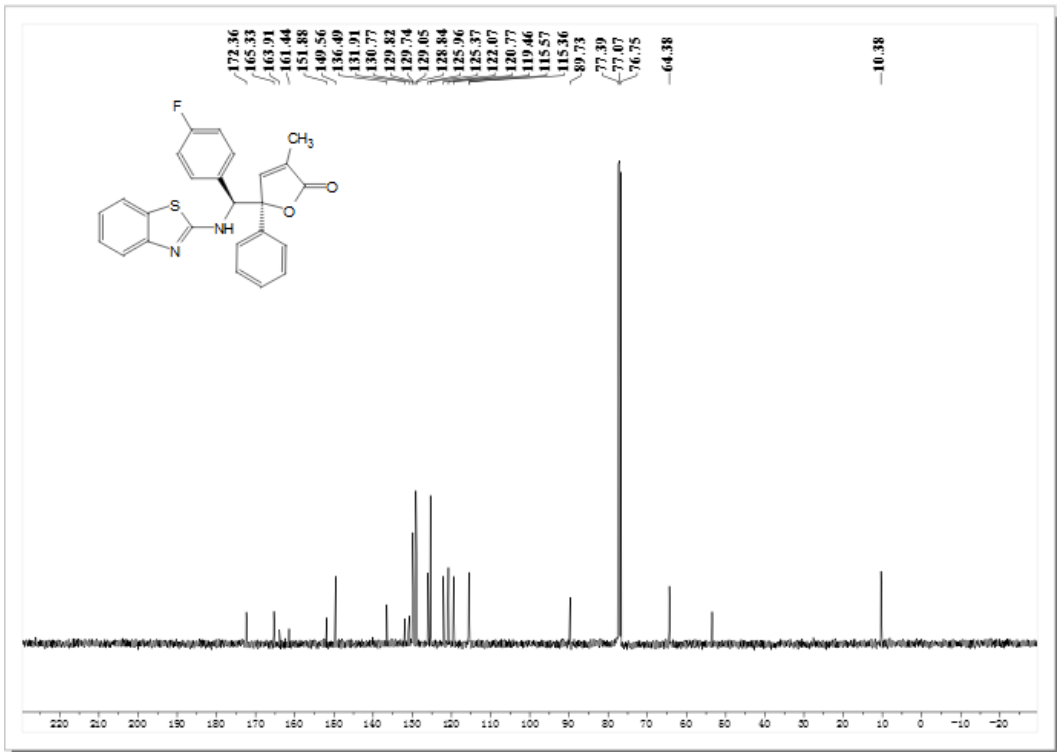
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NH
O
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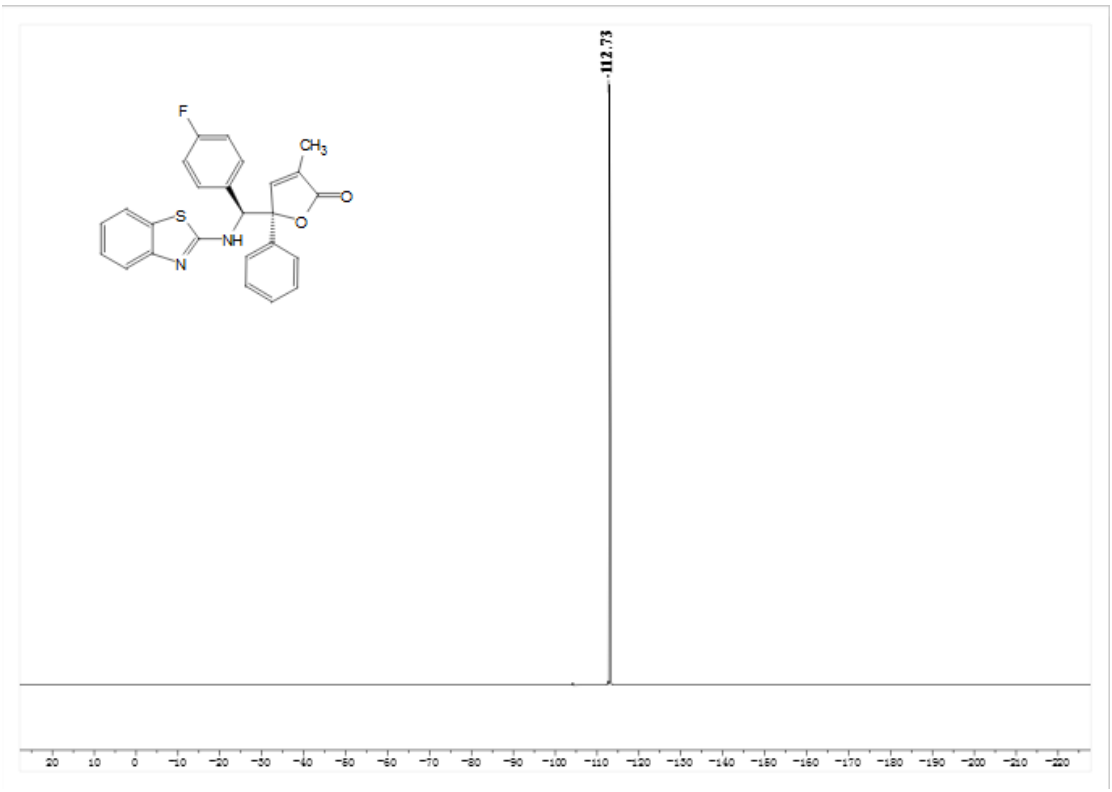
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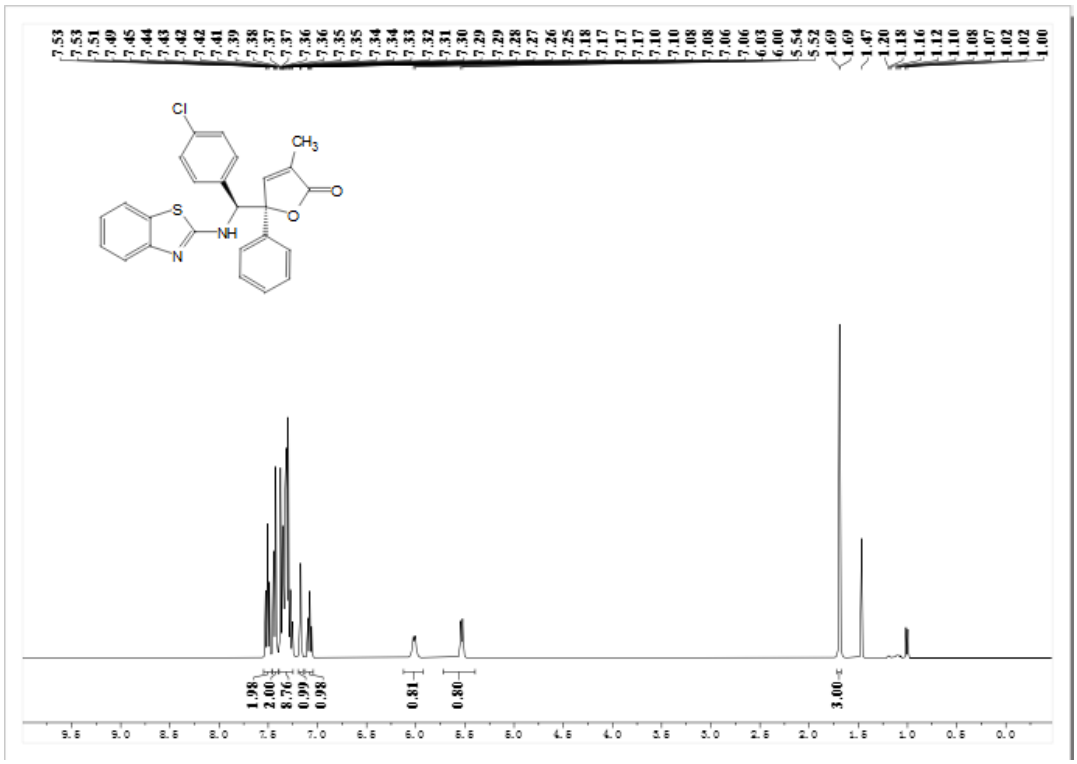
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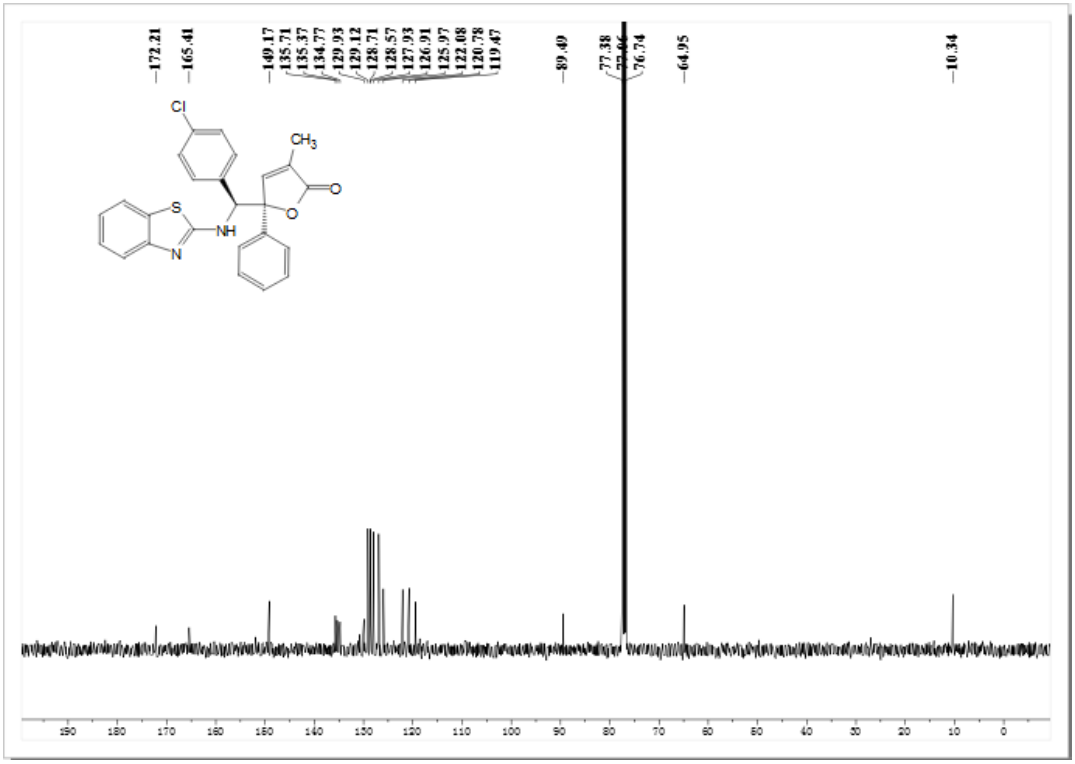
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Cl
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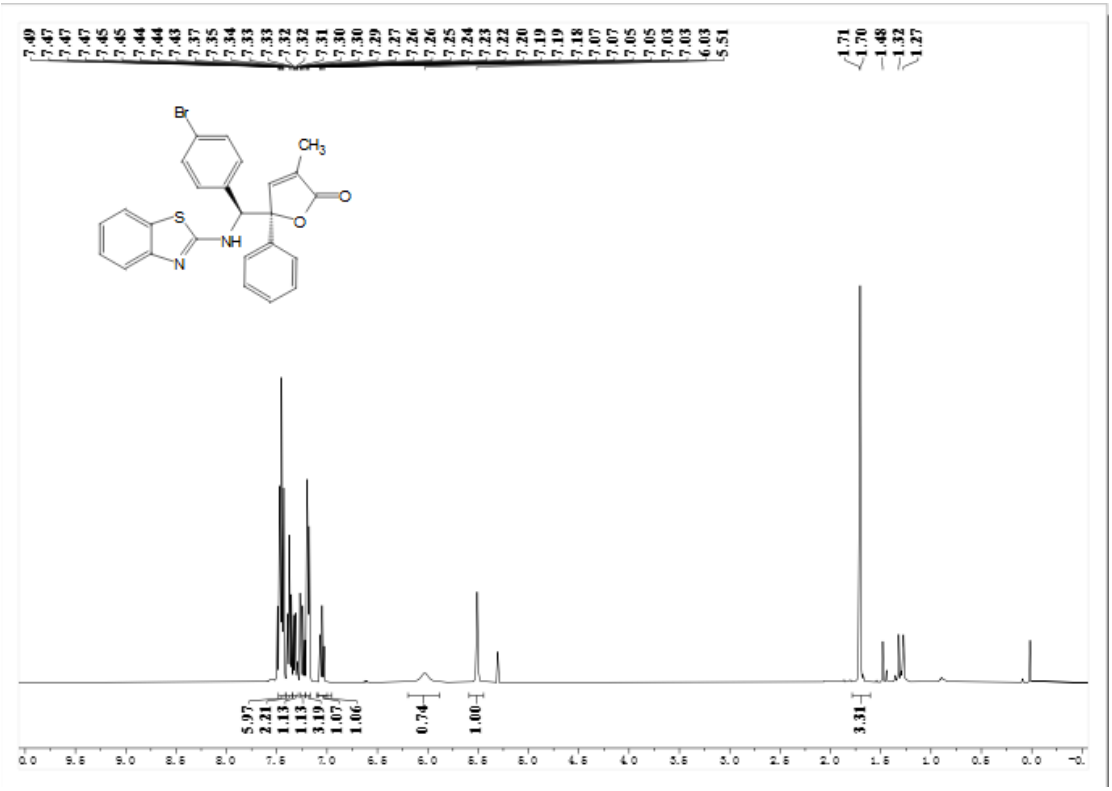
S
NH
N

3da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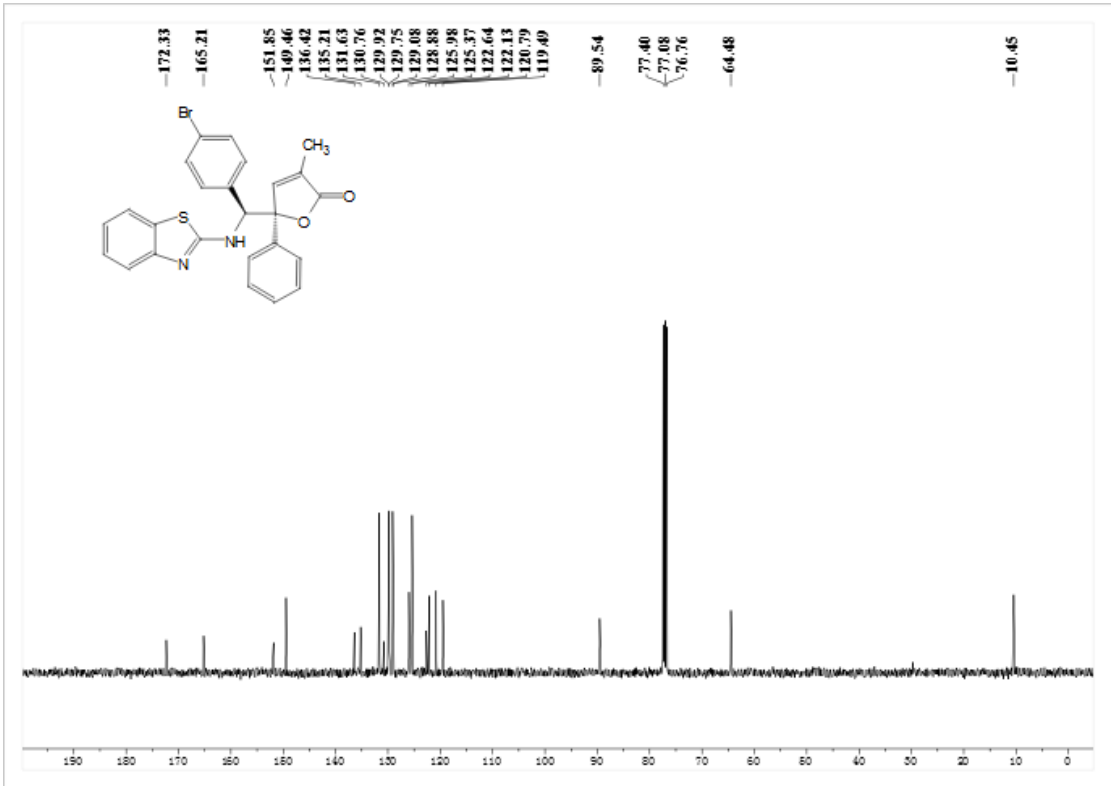
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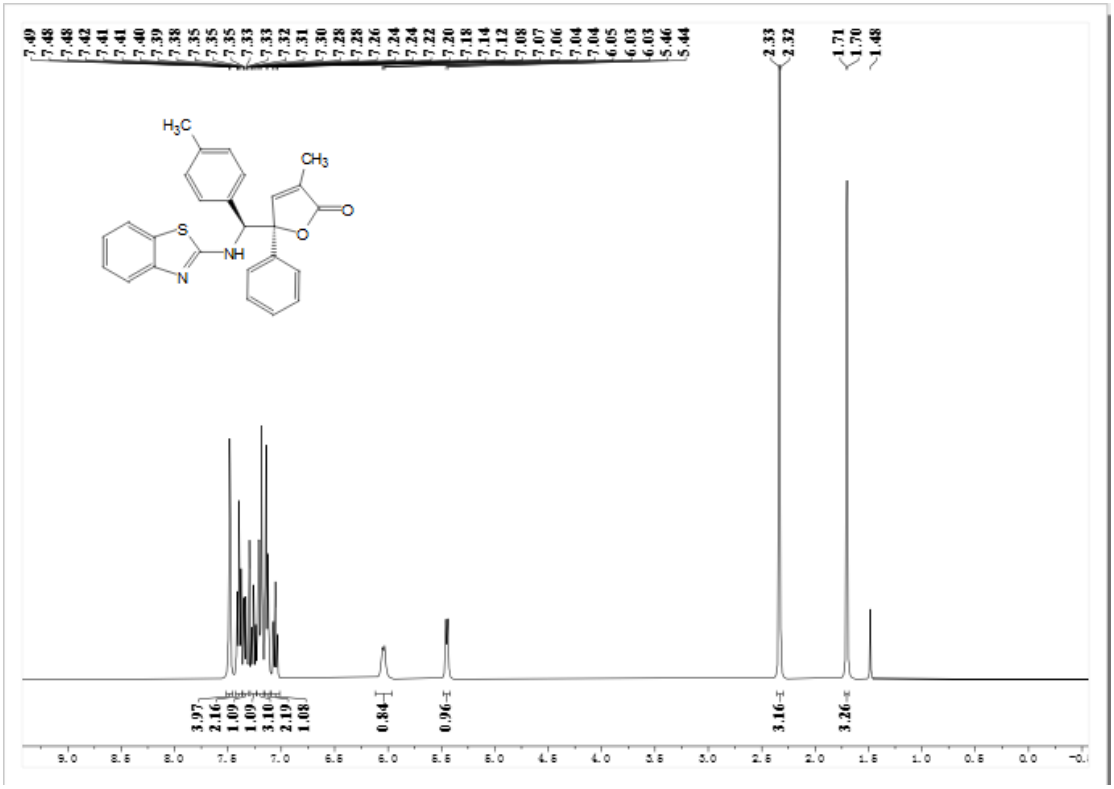
O

3ea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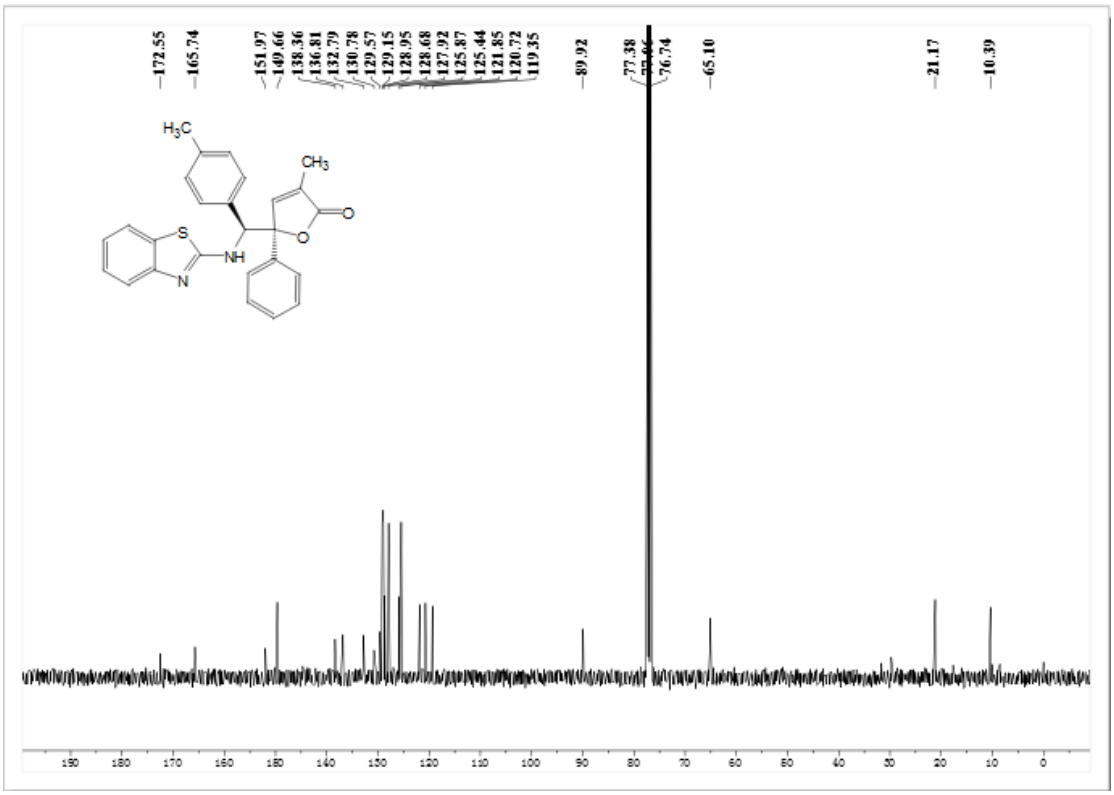
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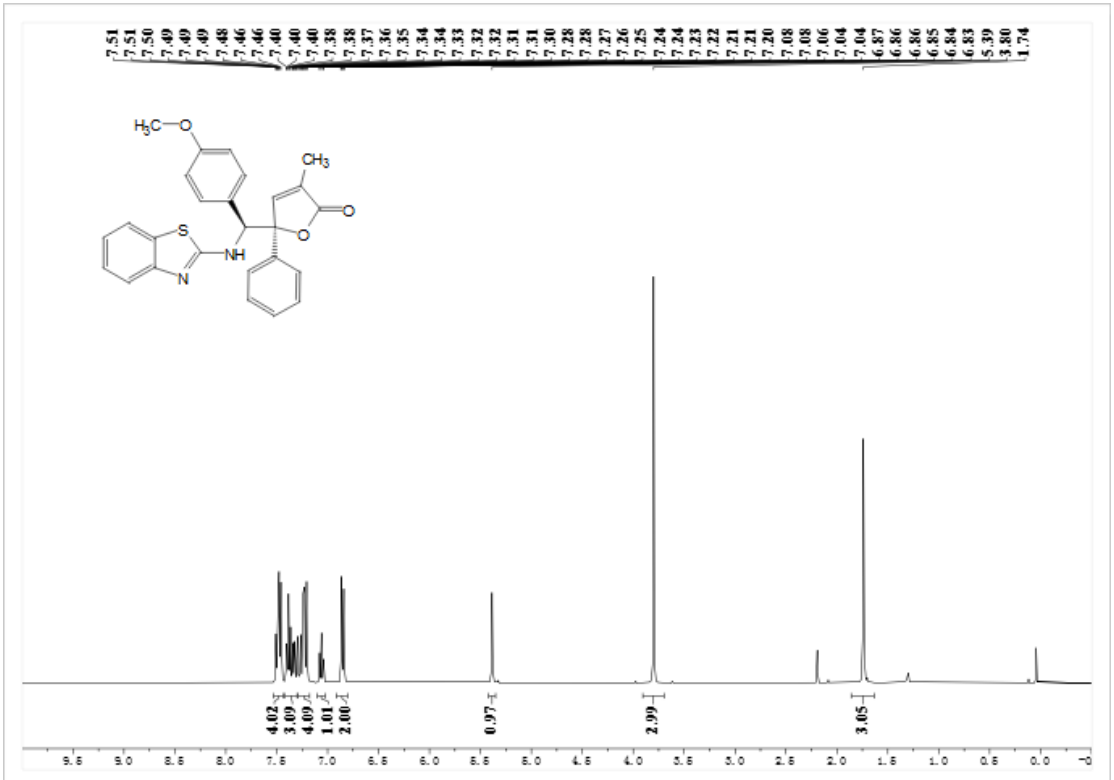
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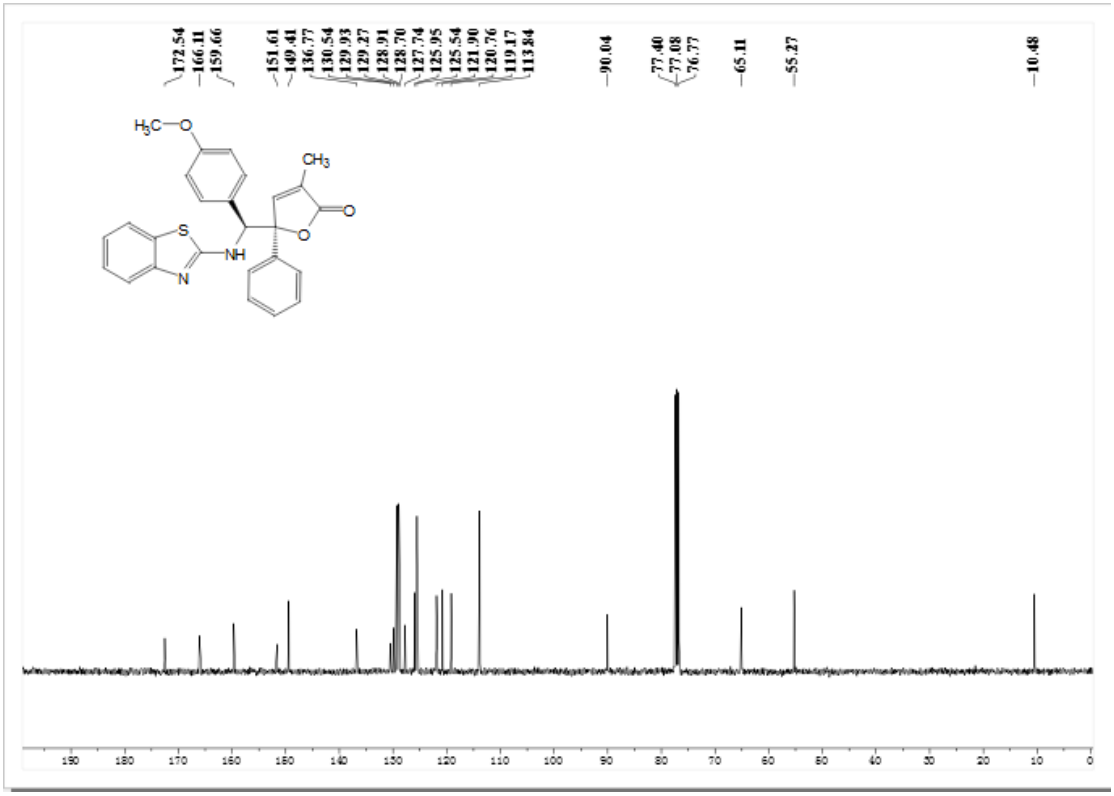
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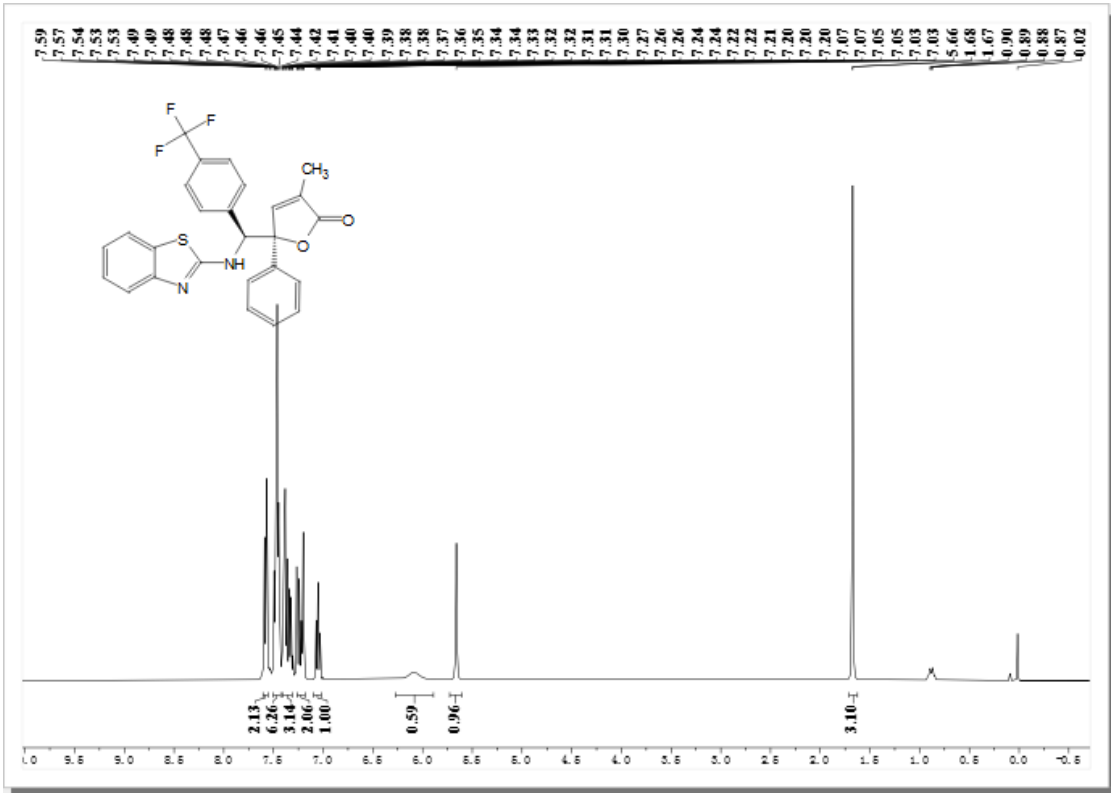
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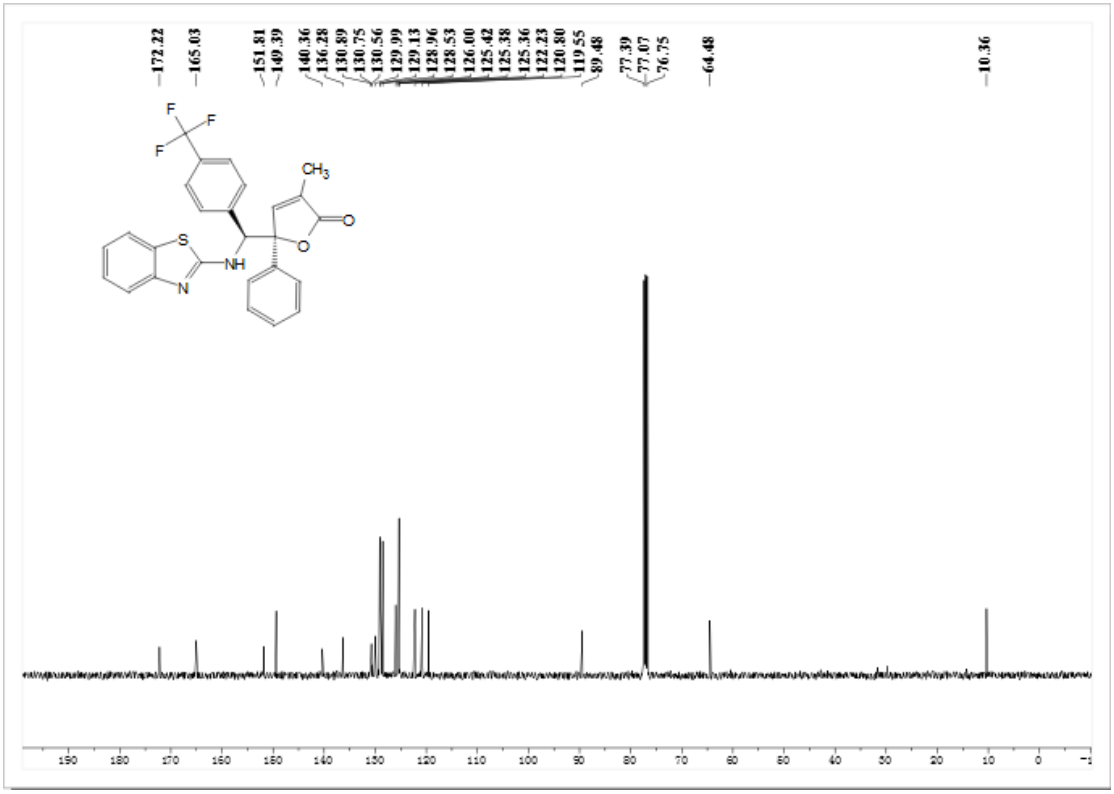
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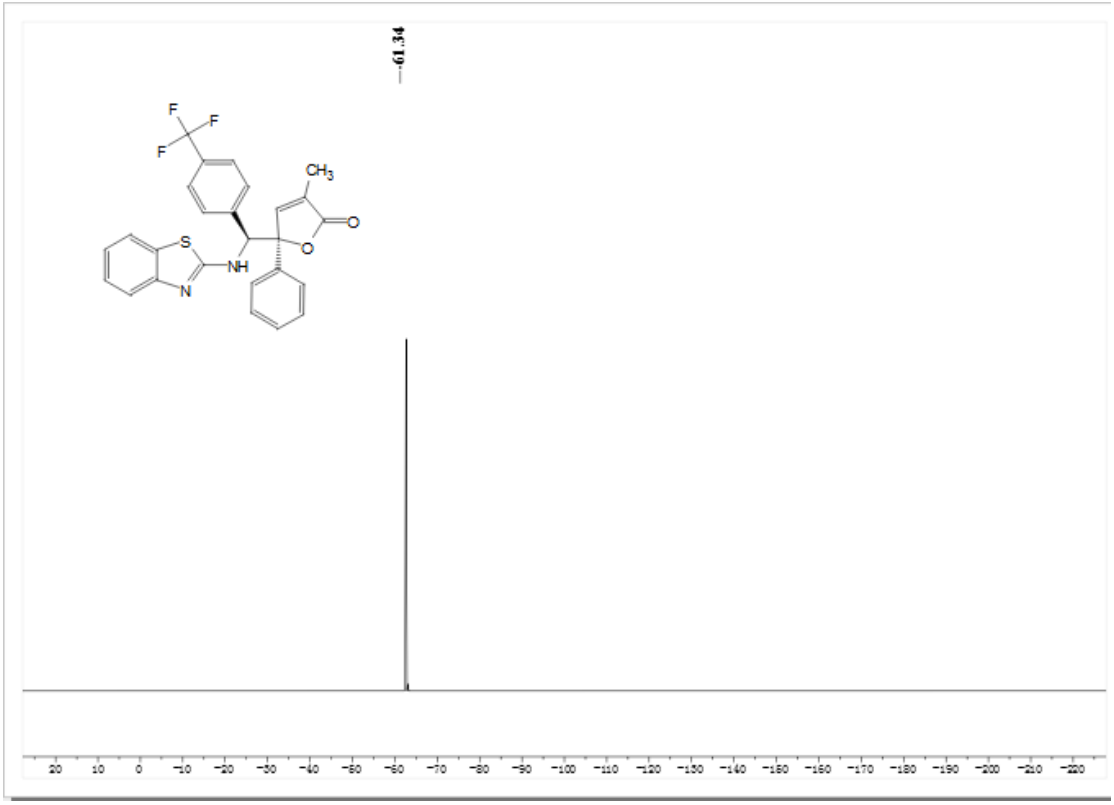
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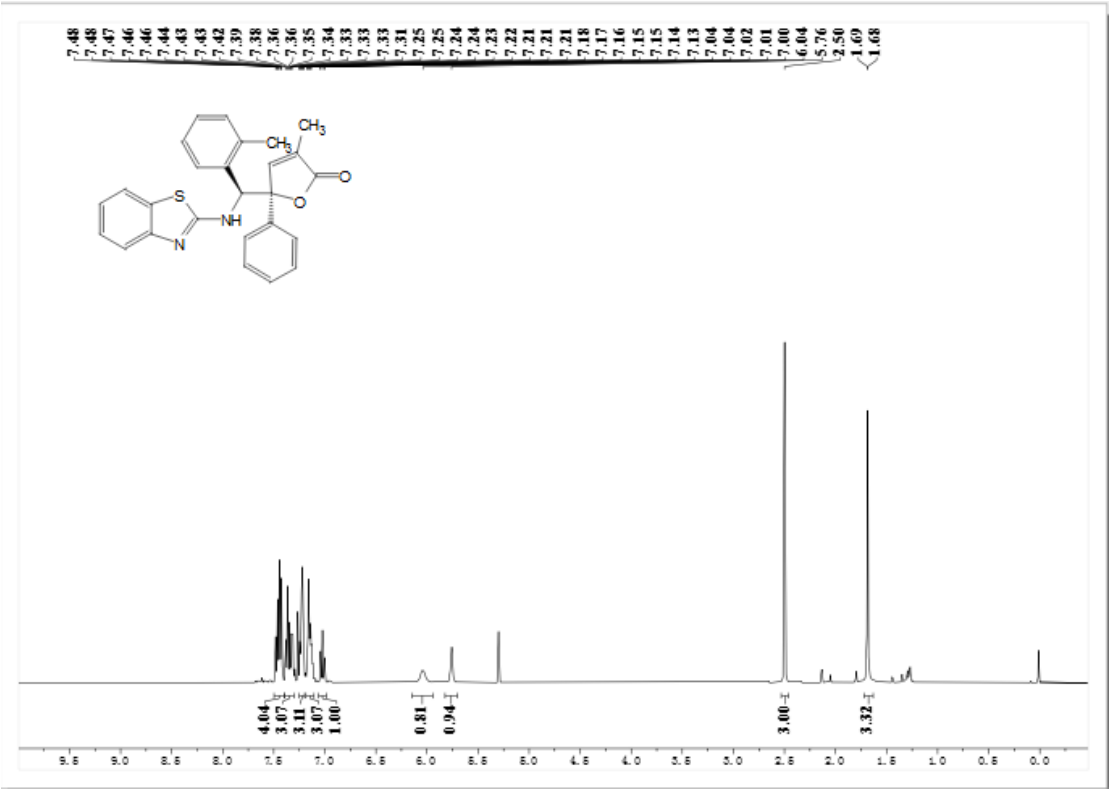
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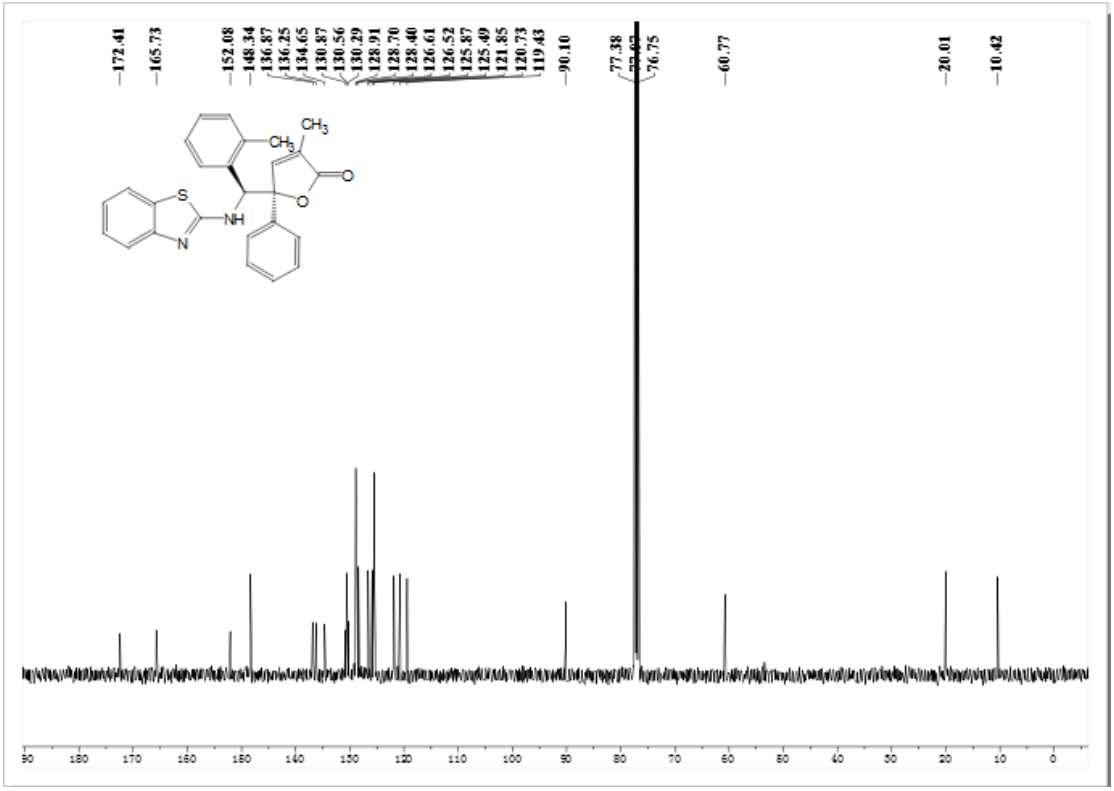
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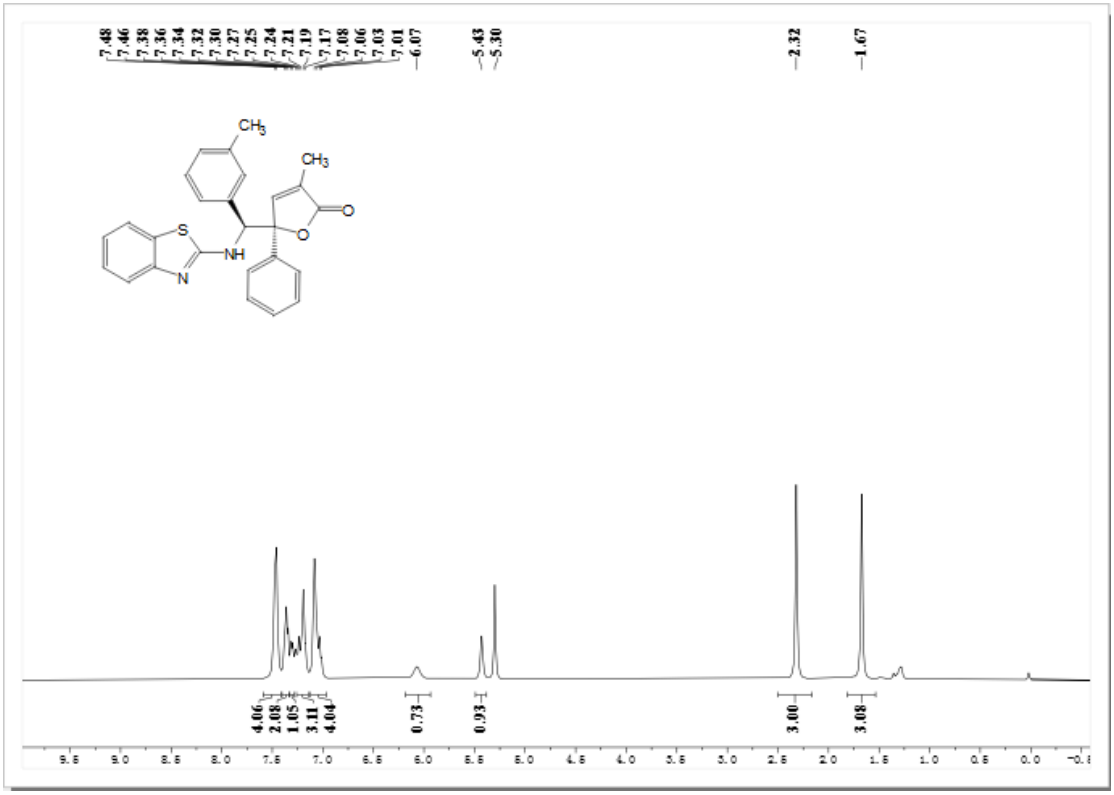
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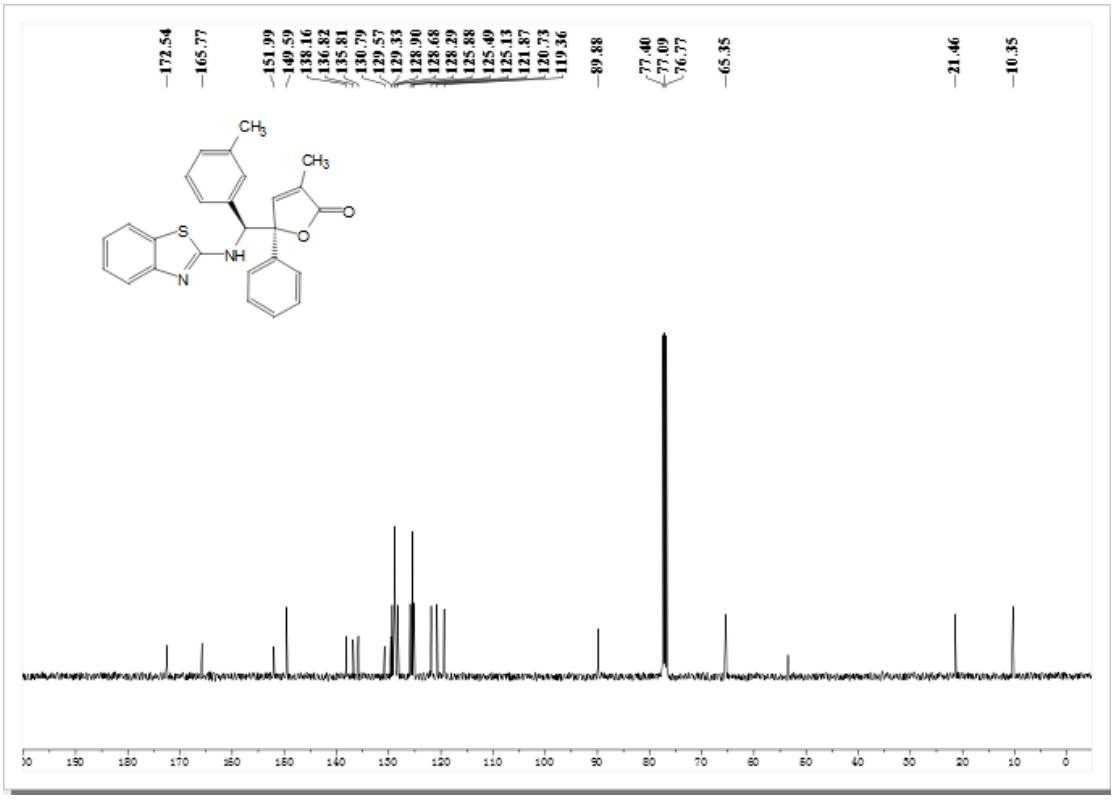
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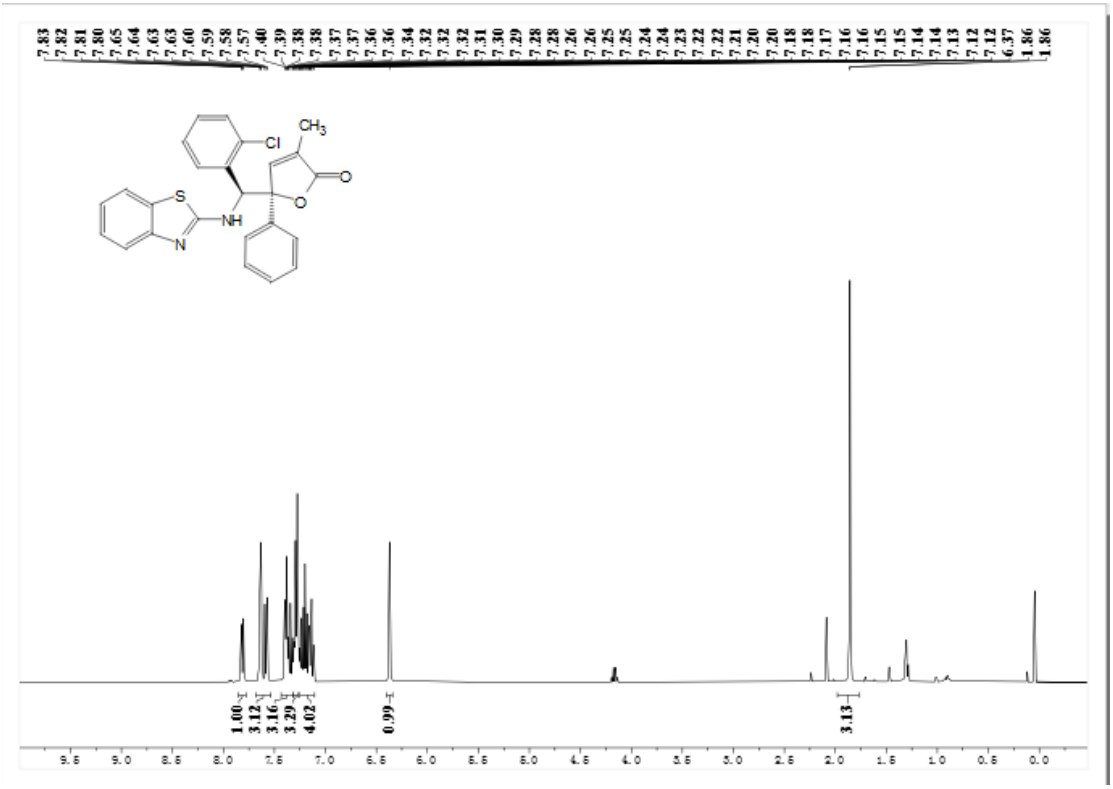
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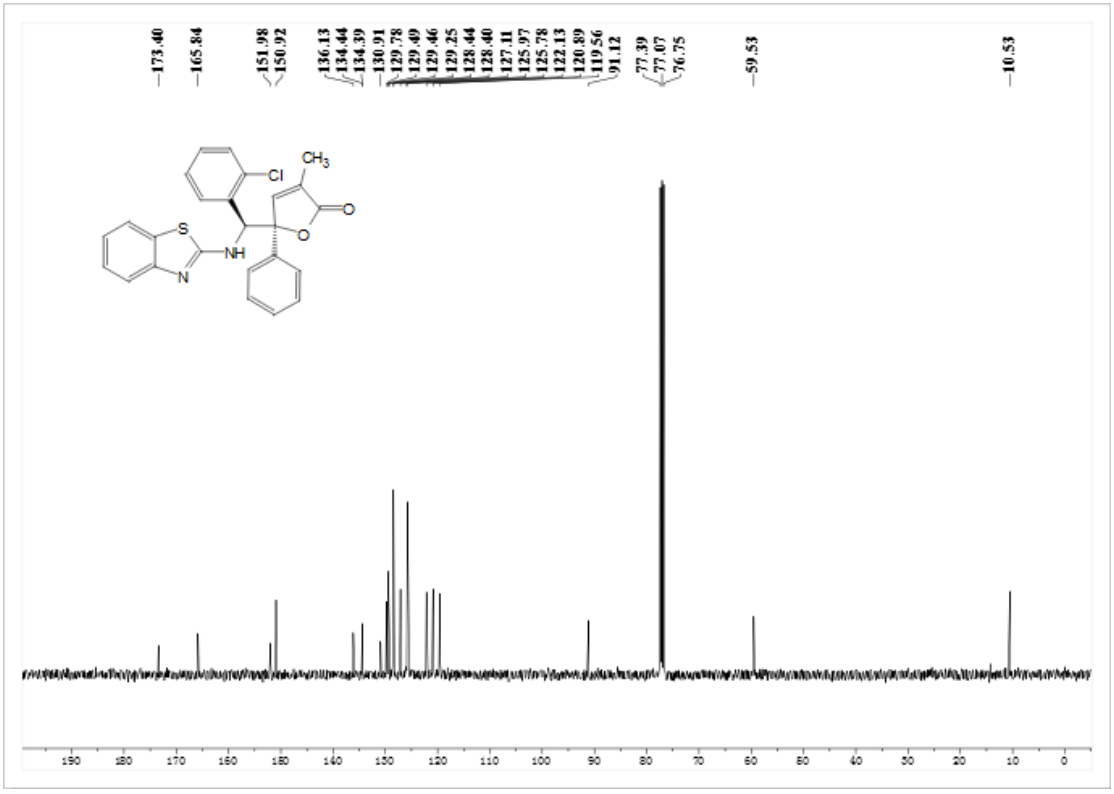
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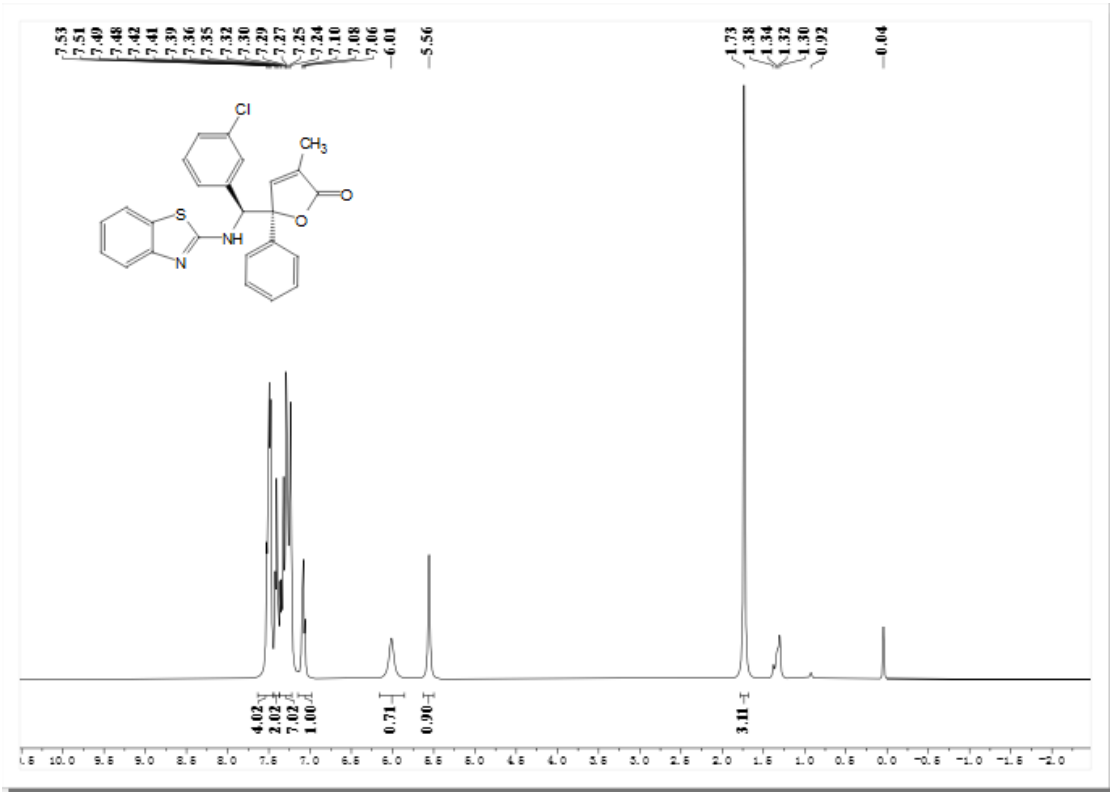
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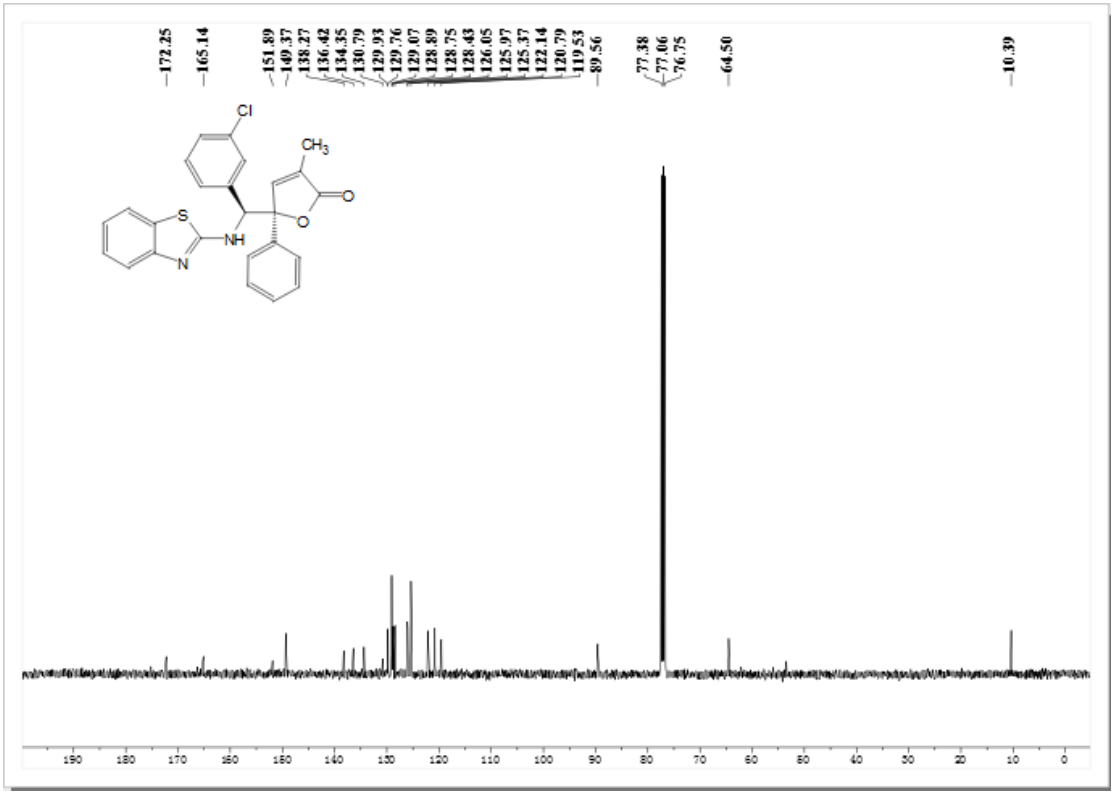
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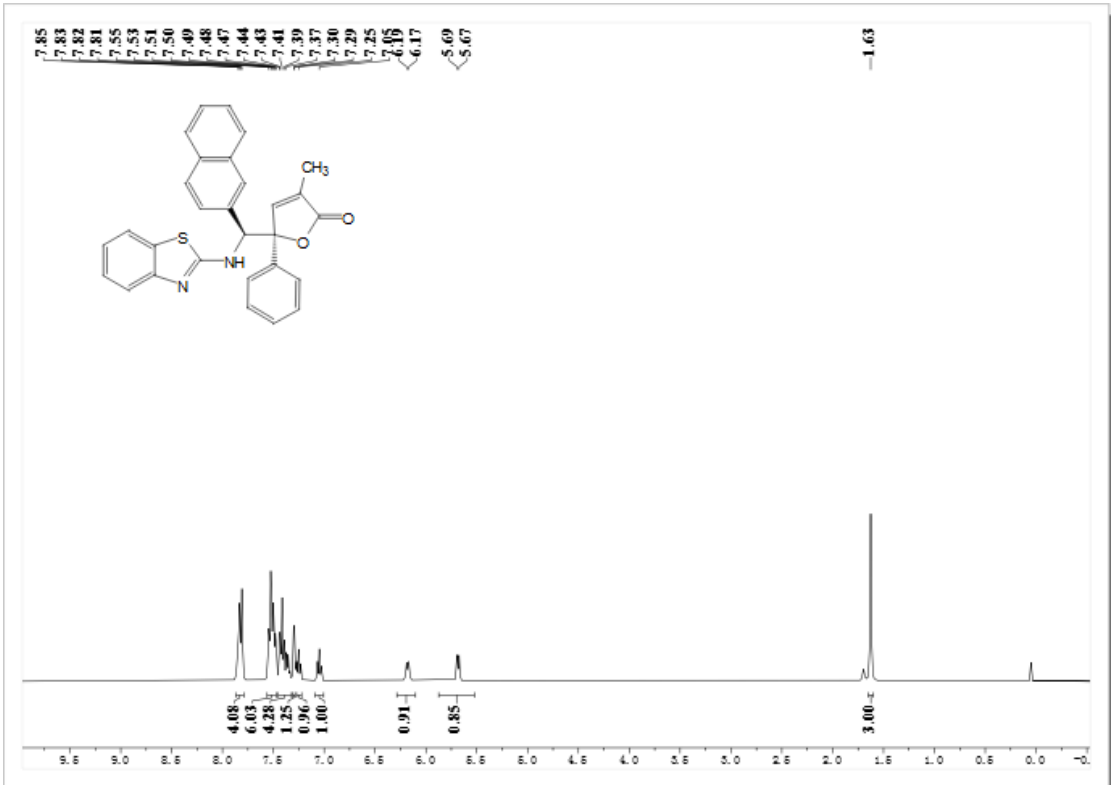
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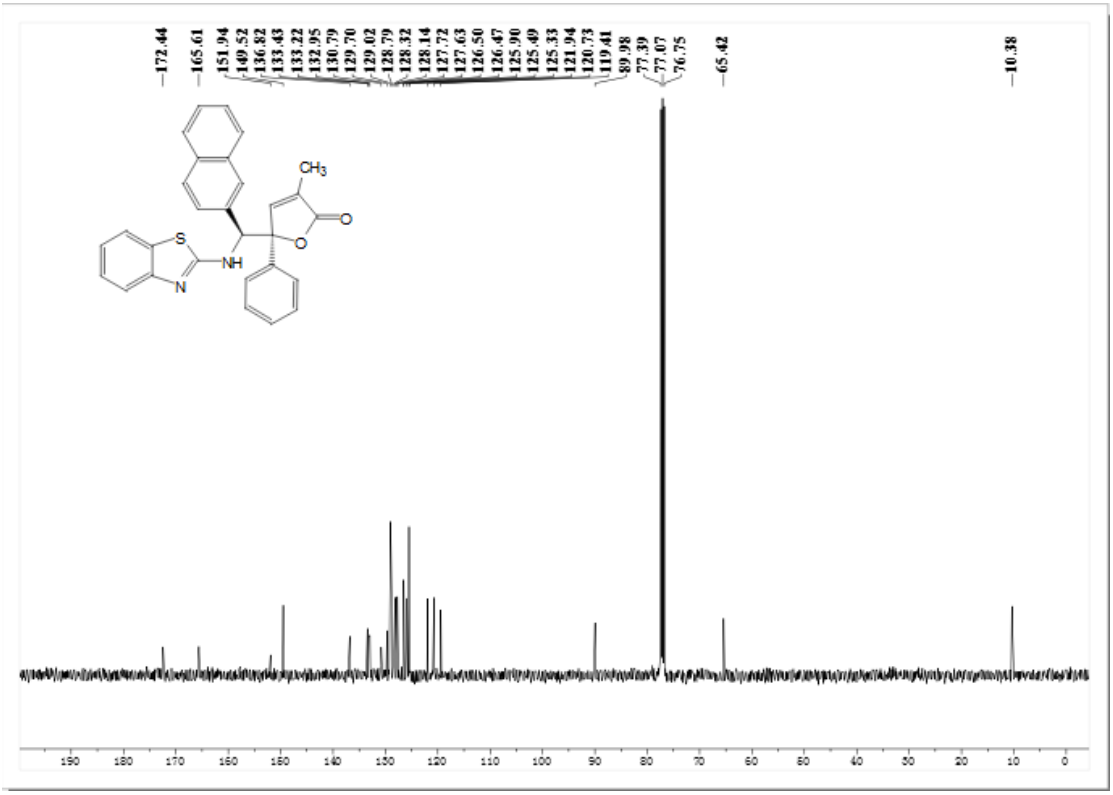
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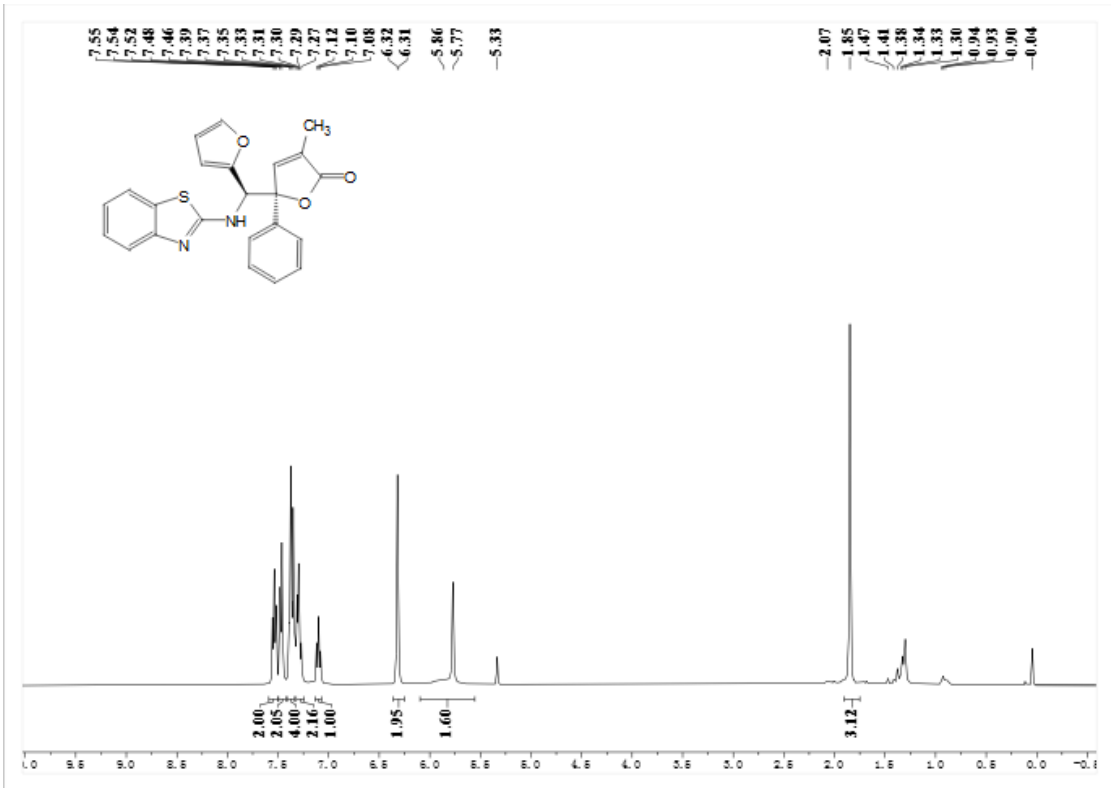
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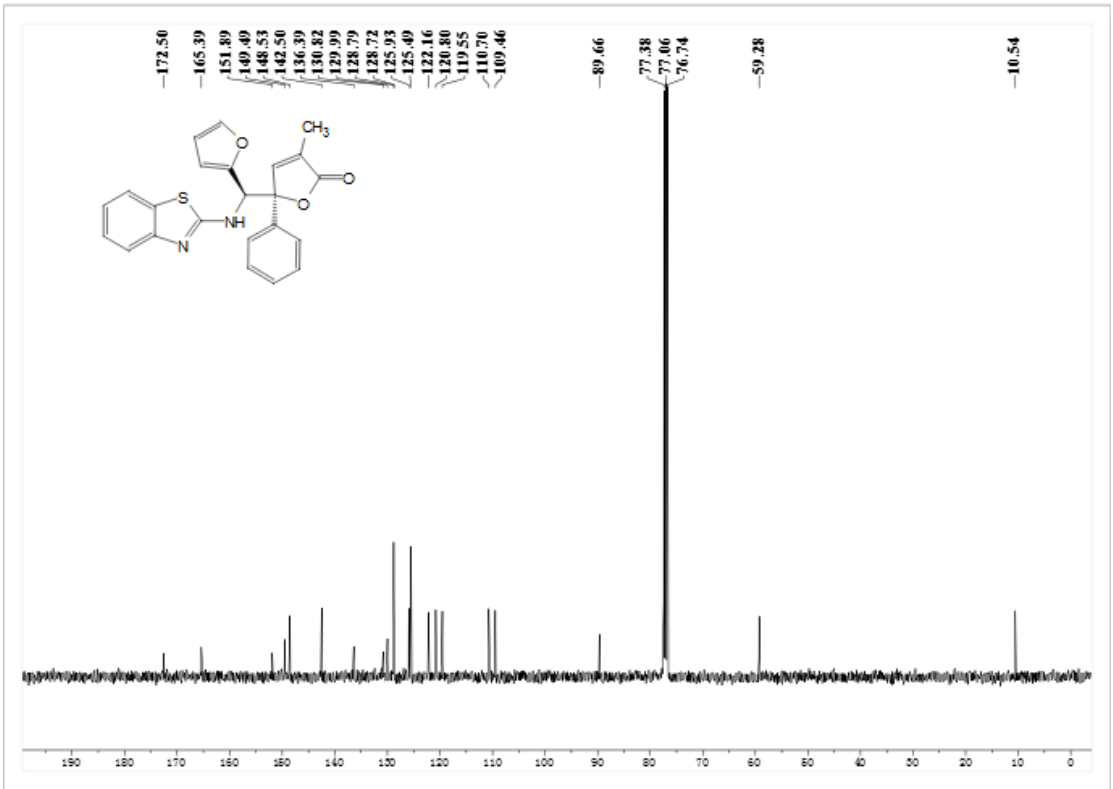
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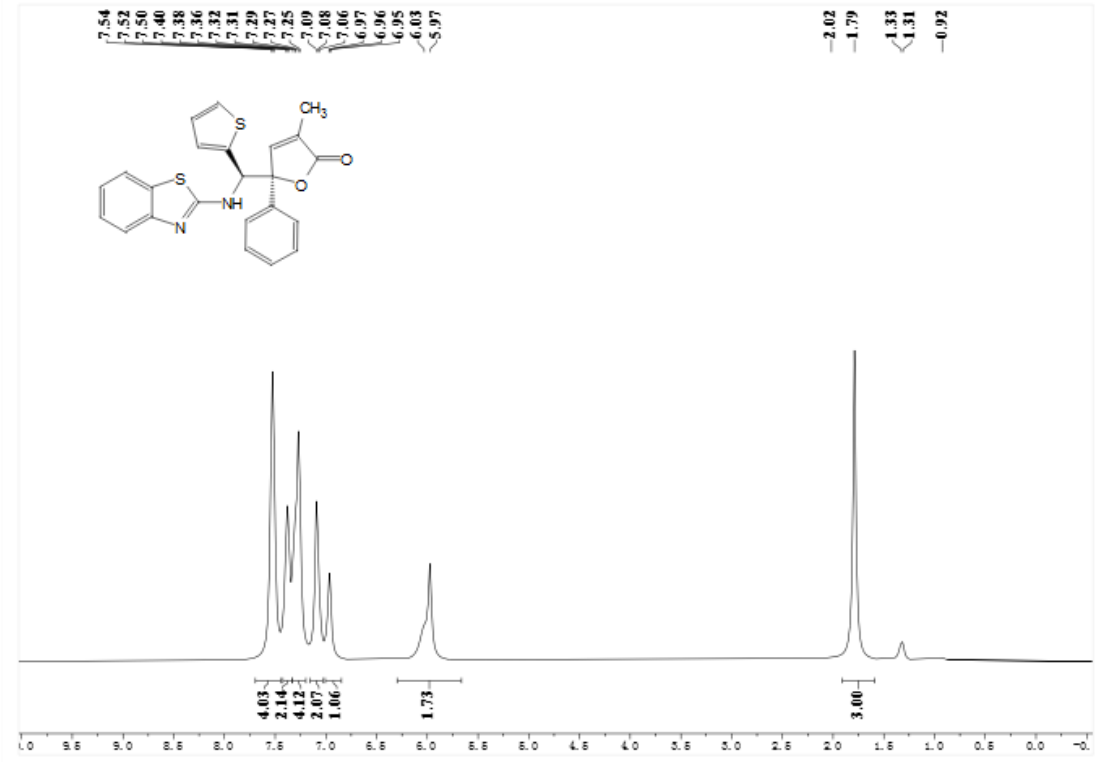
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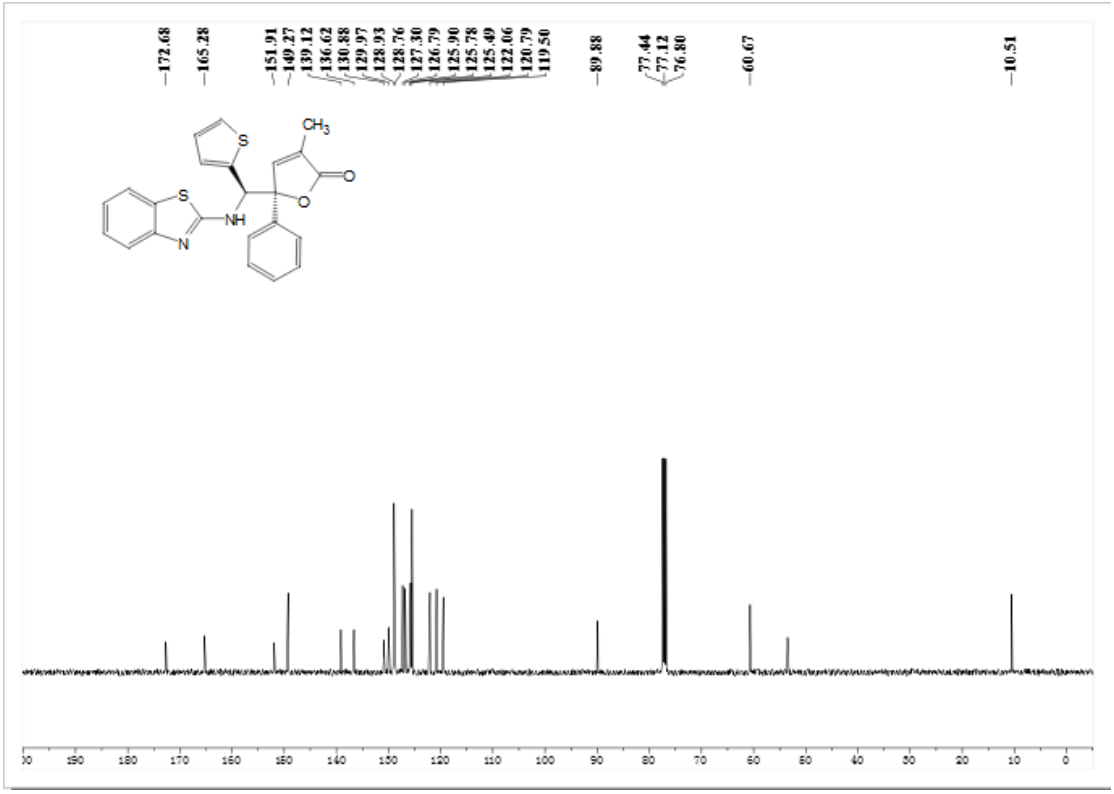
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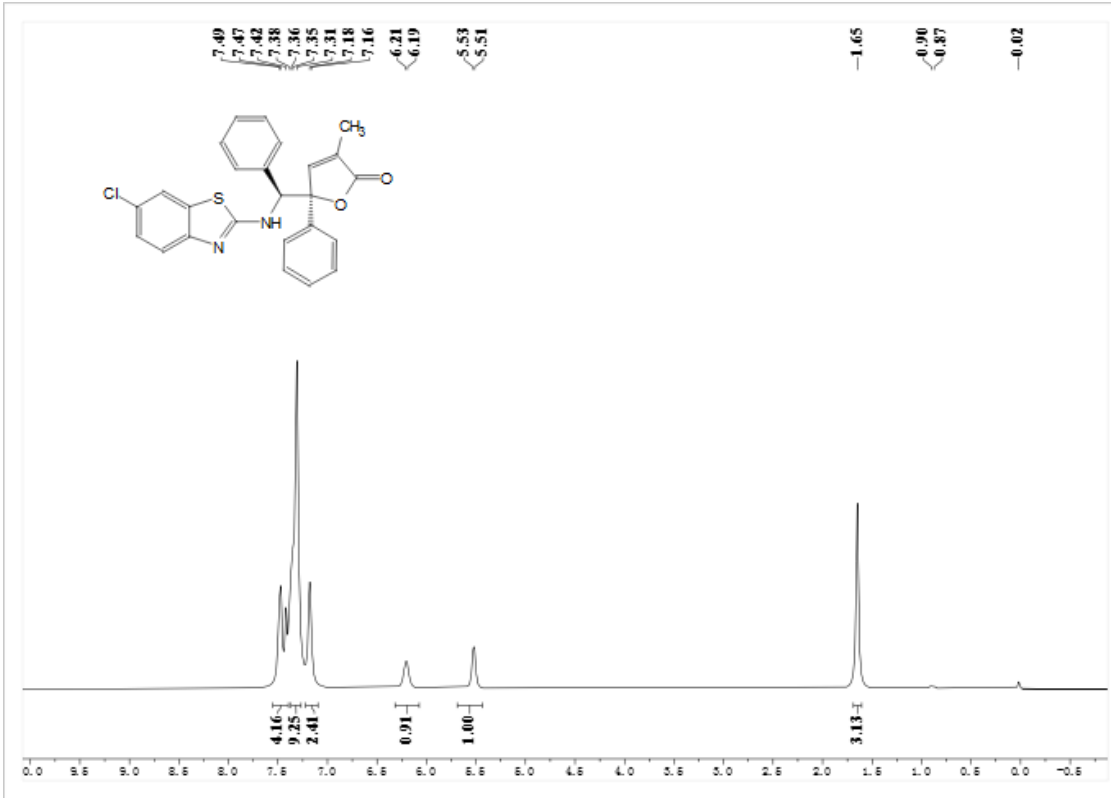
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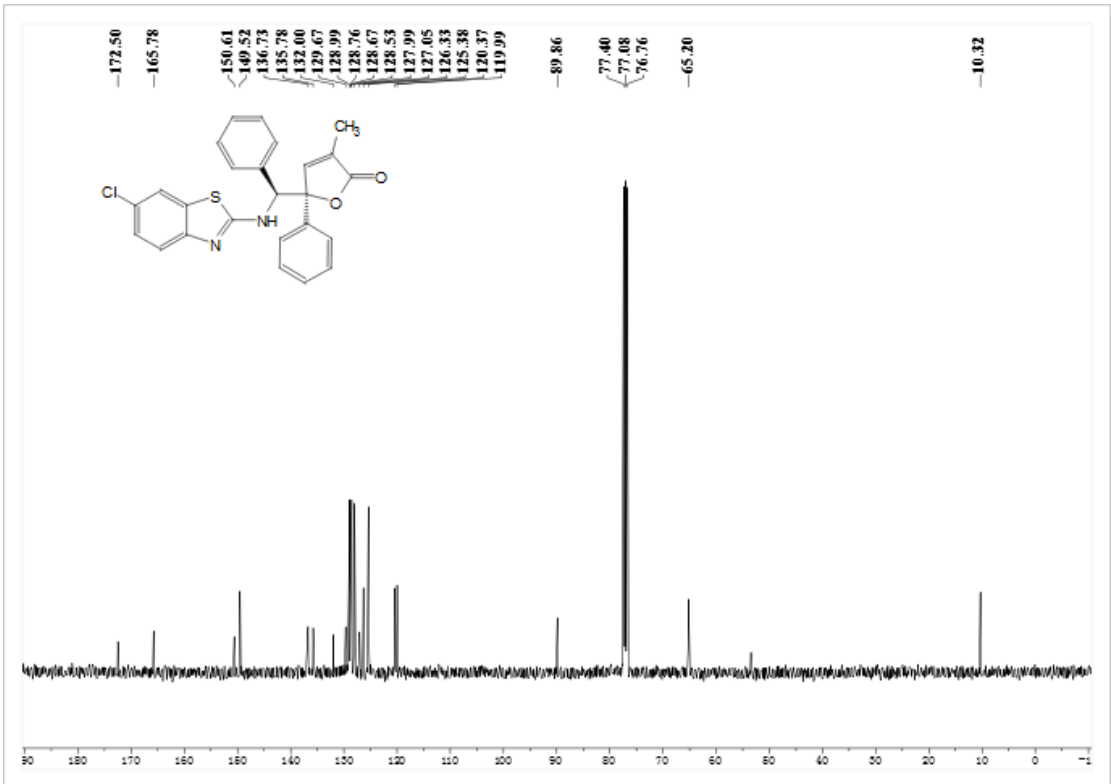
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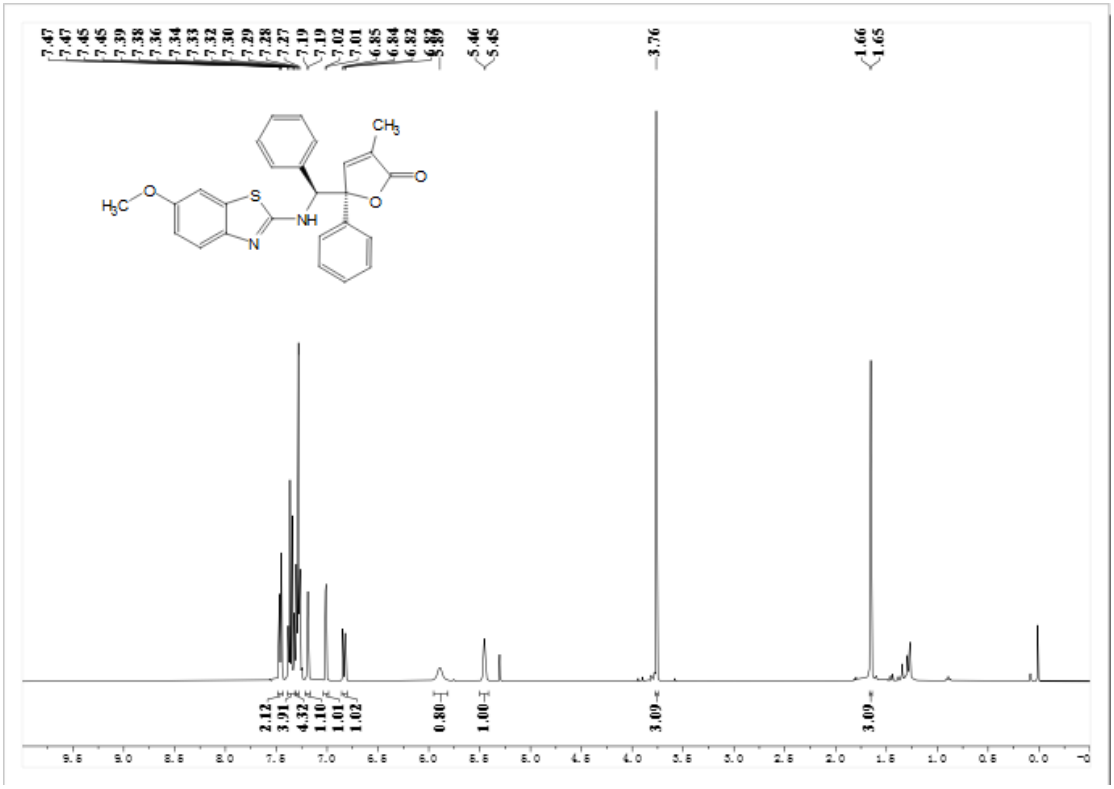
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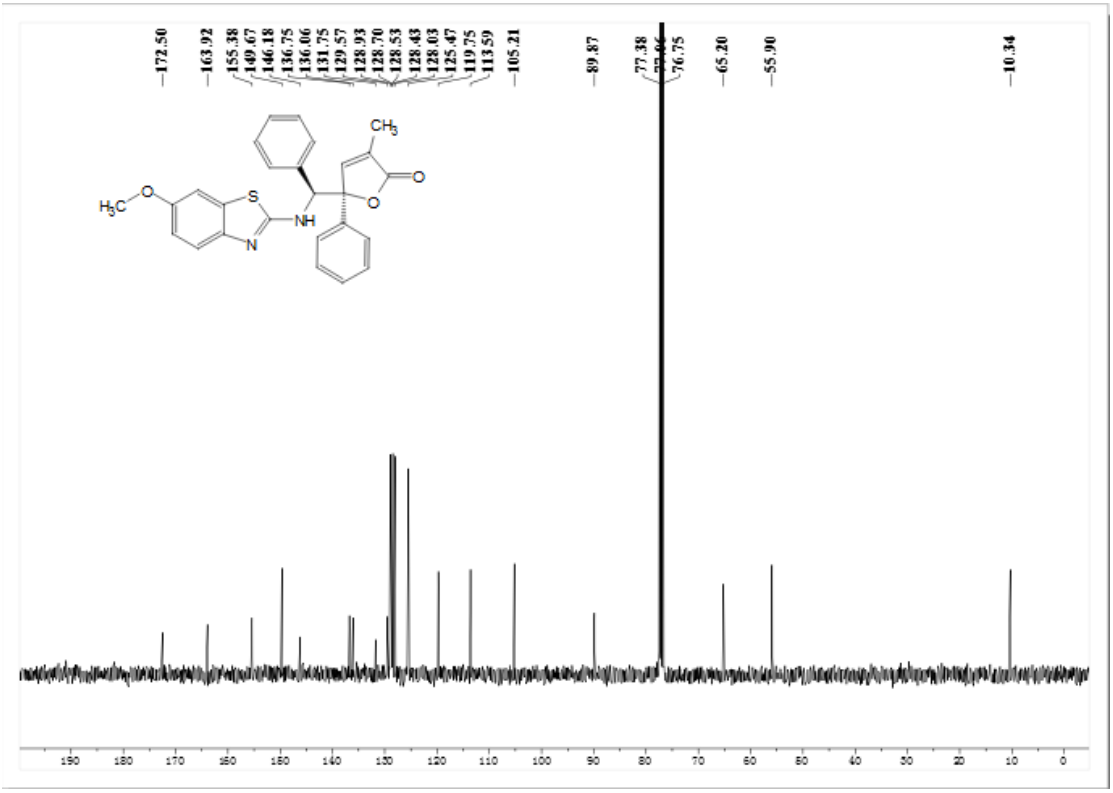
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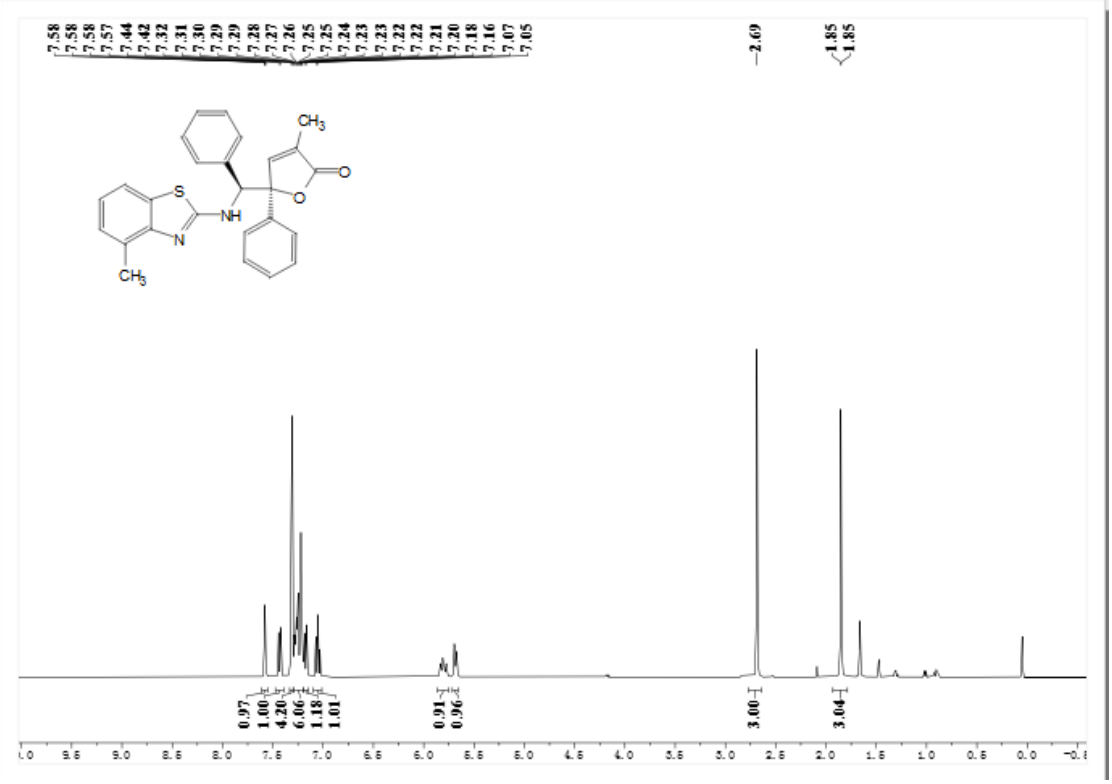
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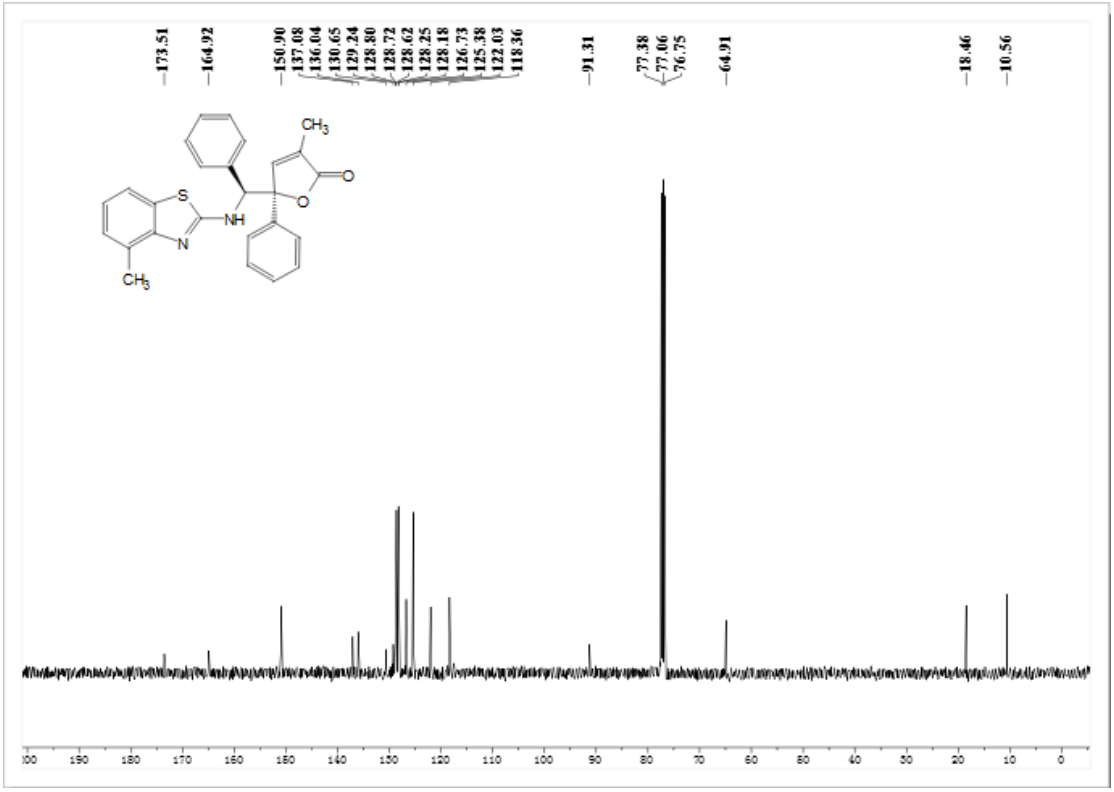
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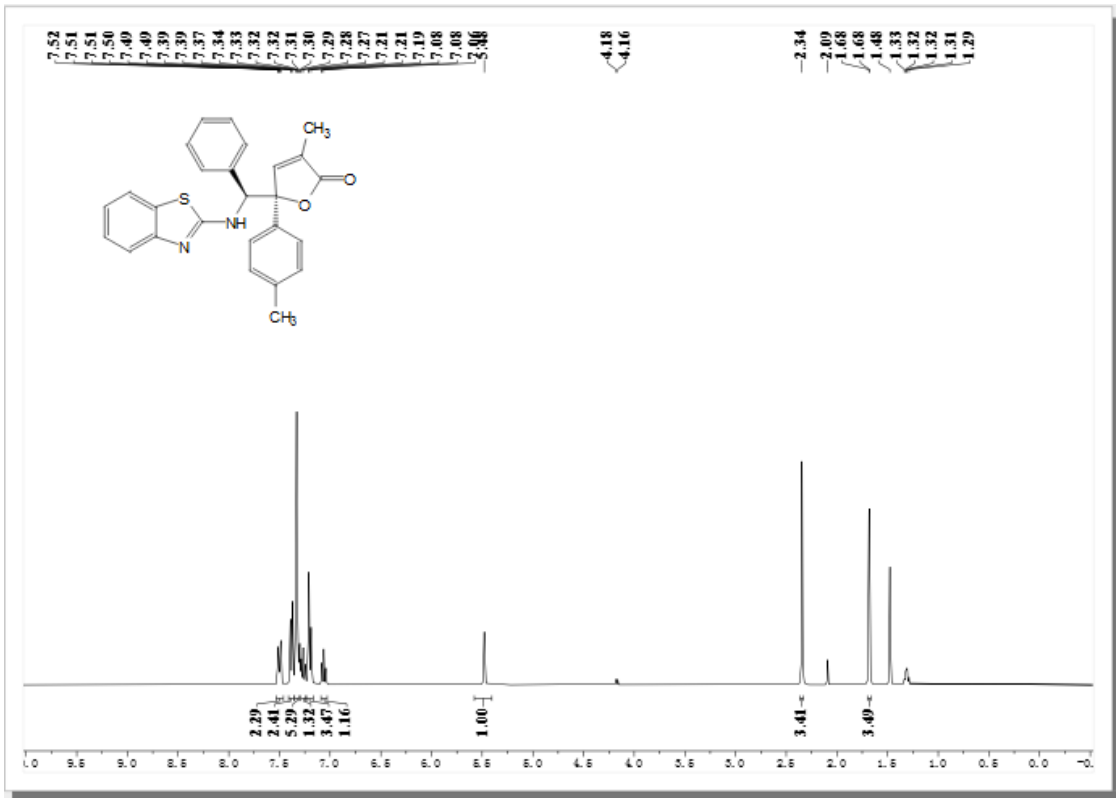
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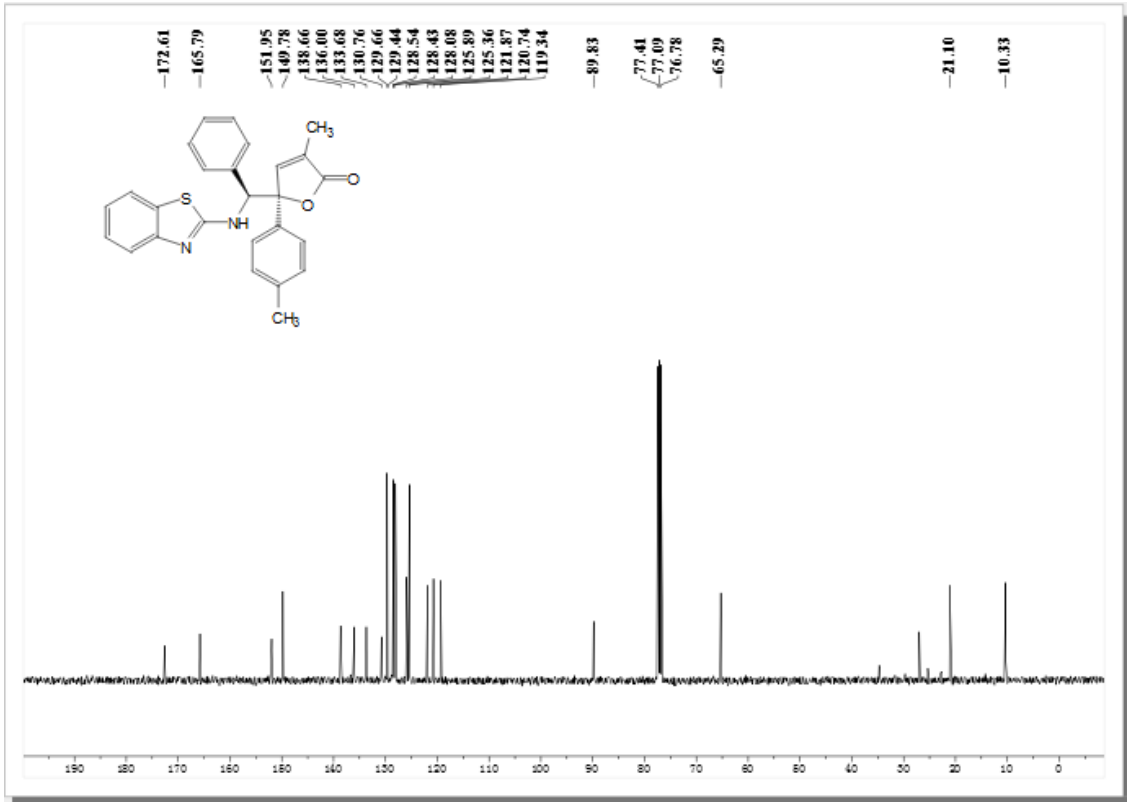
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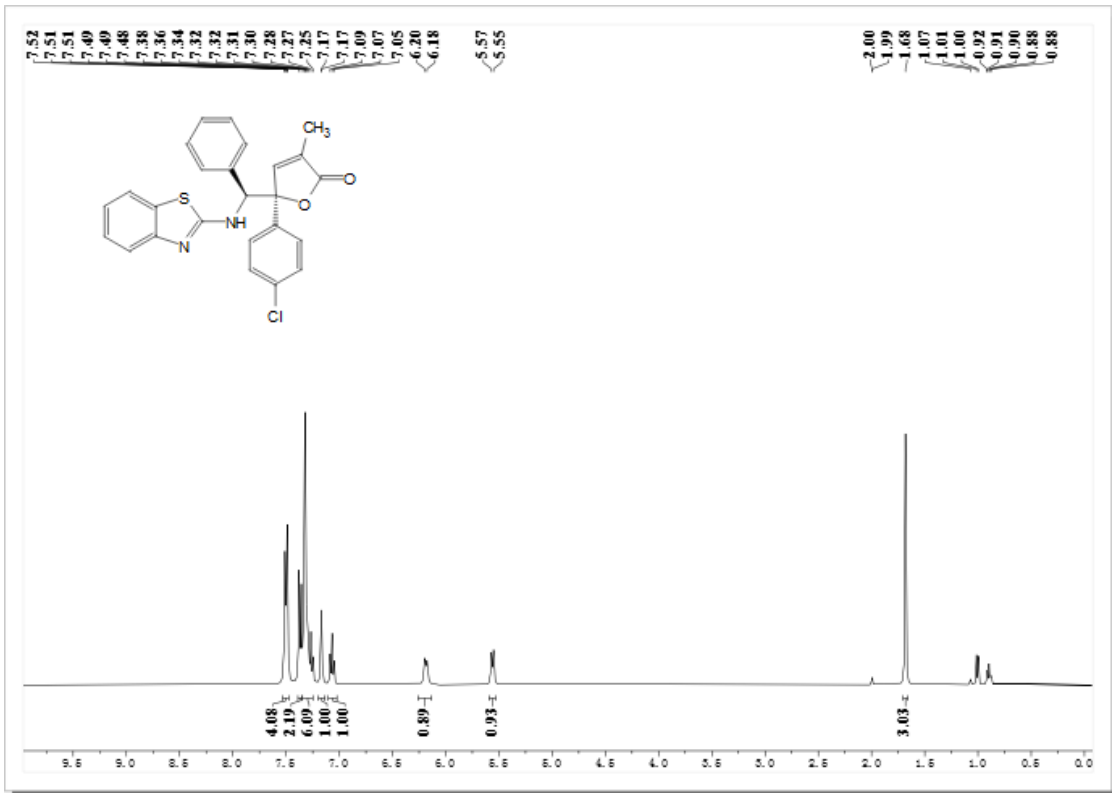
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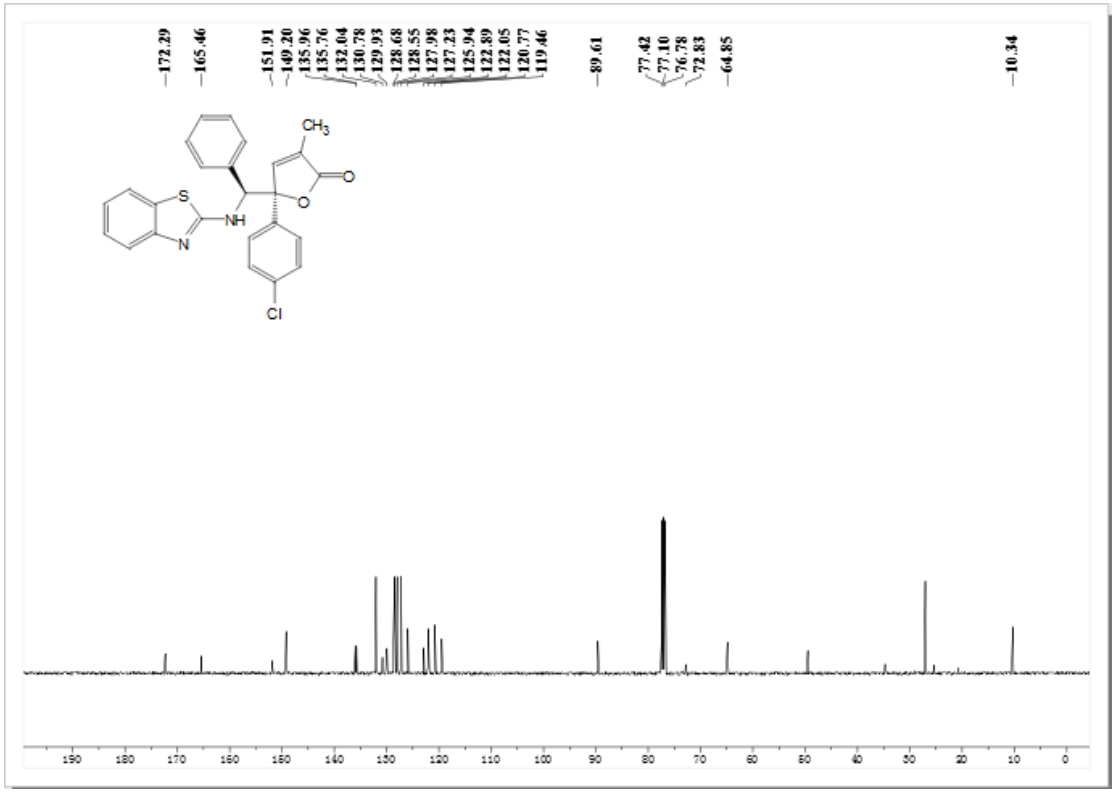
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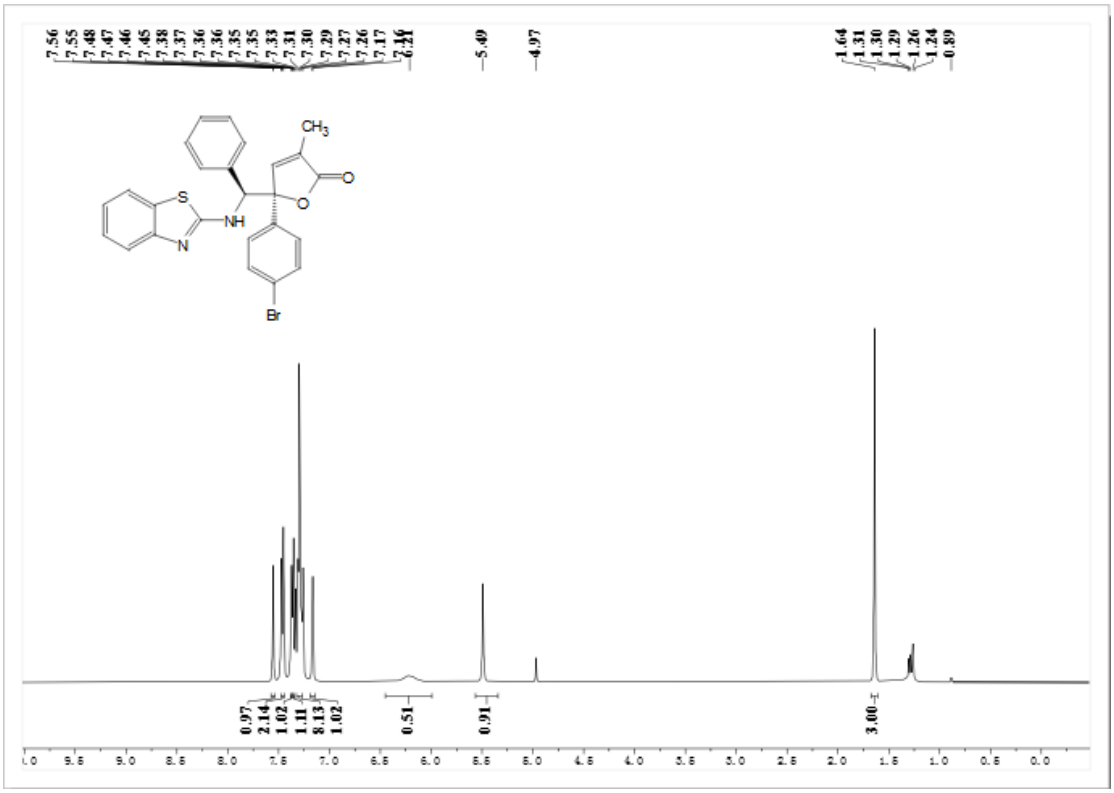
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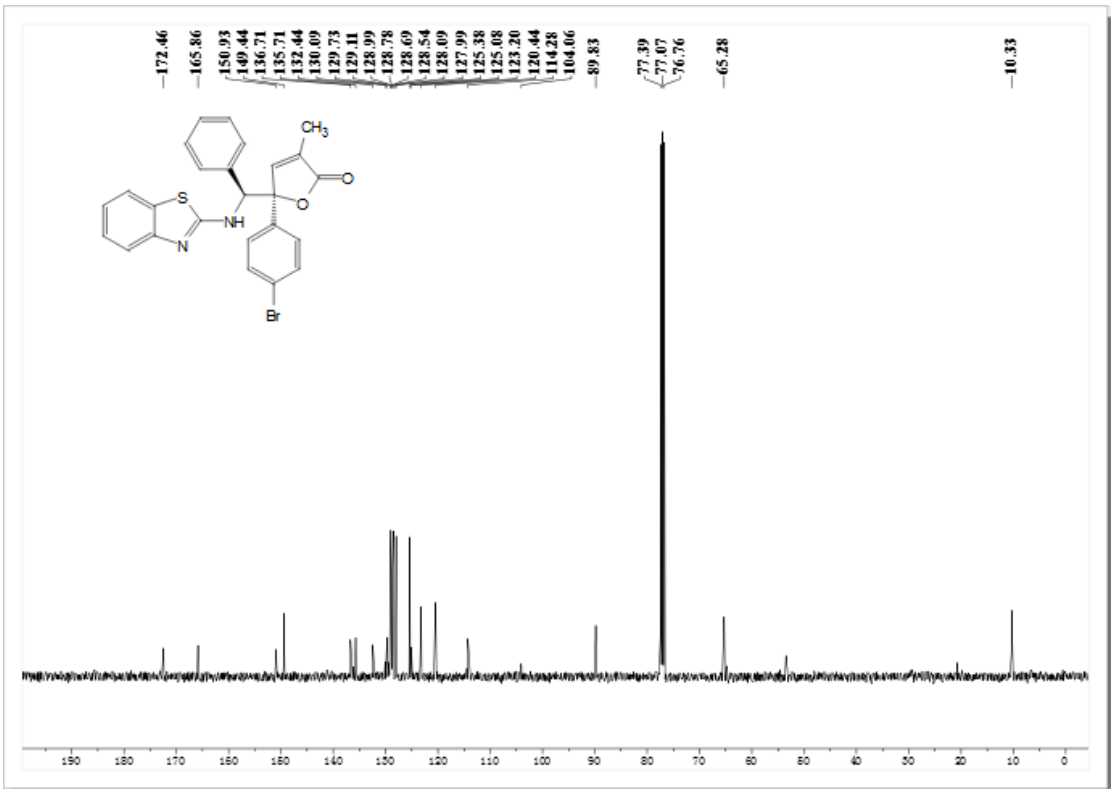
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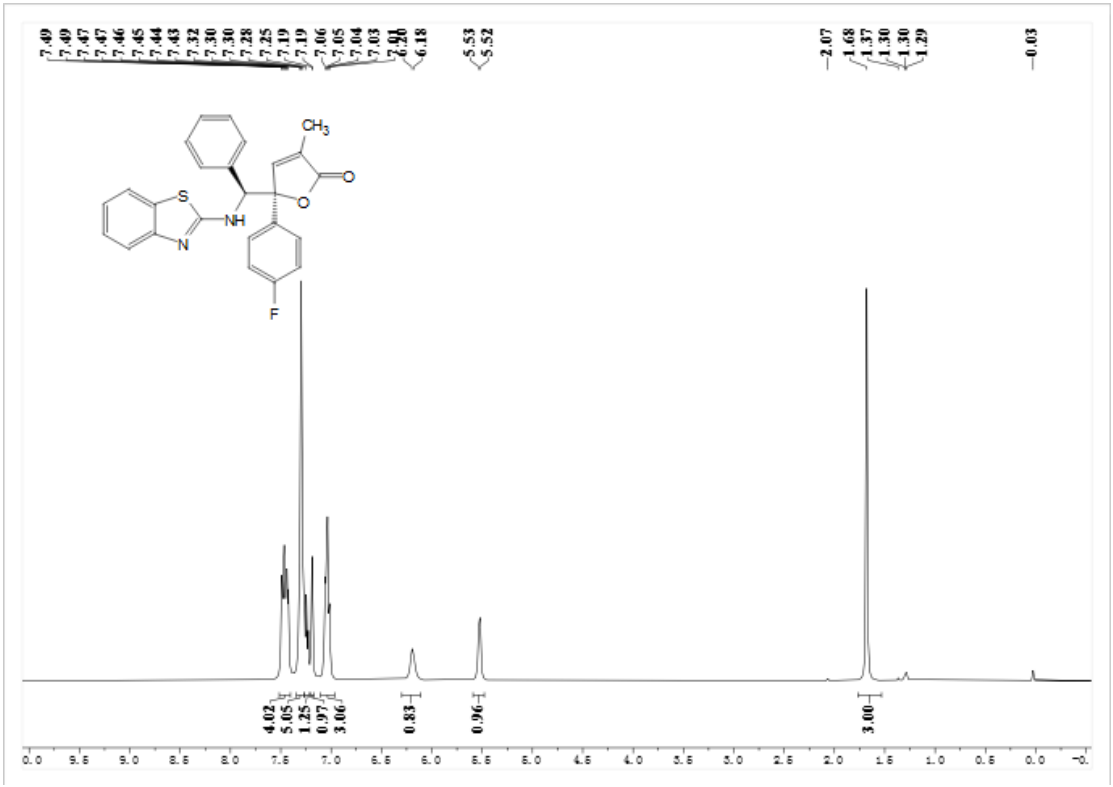
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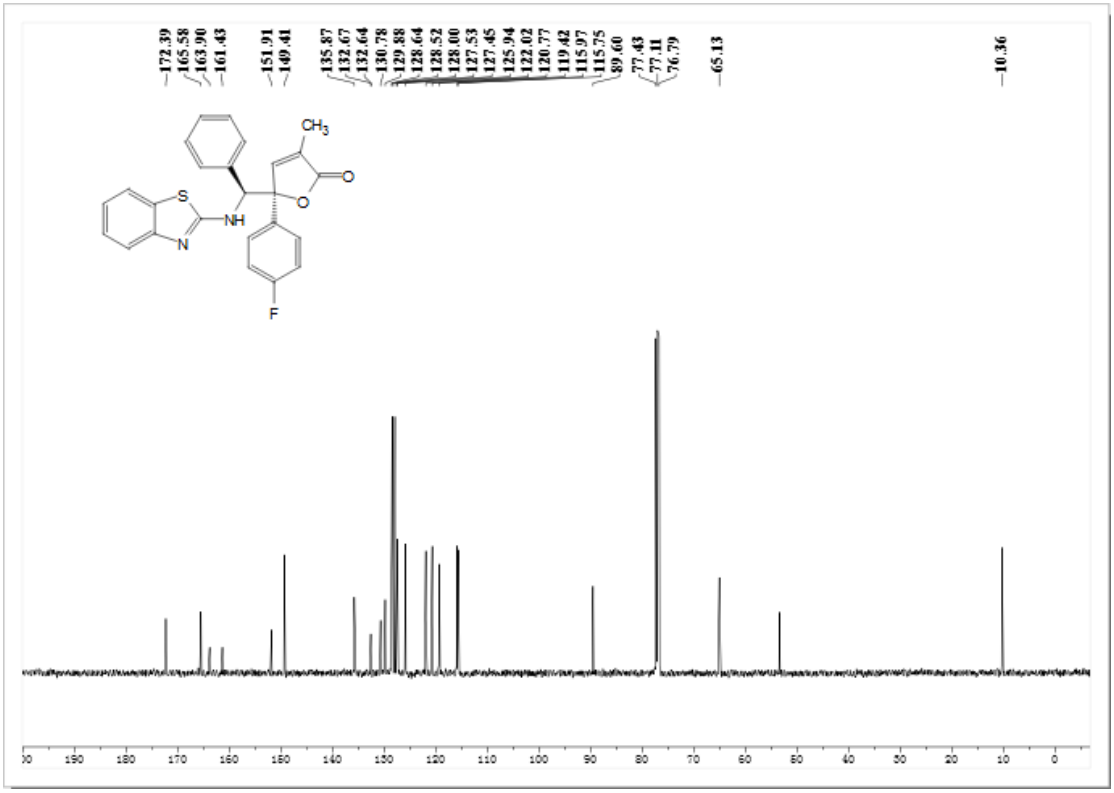
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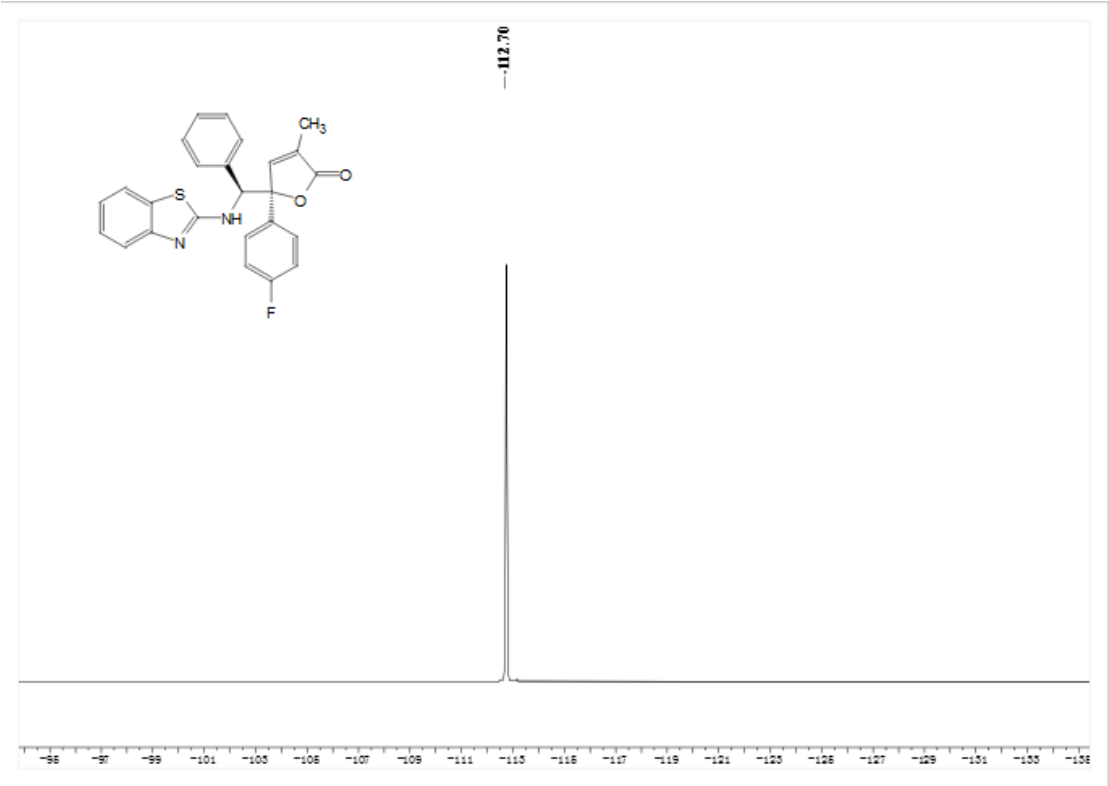
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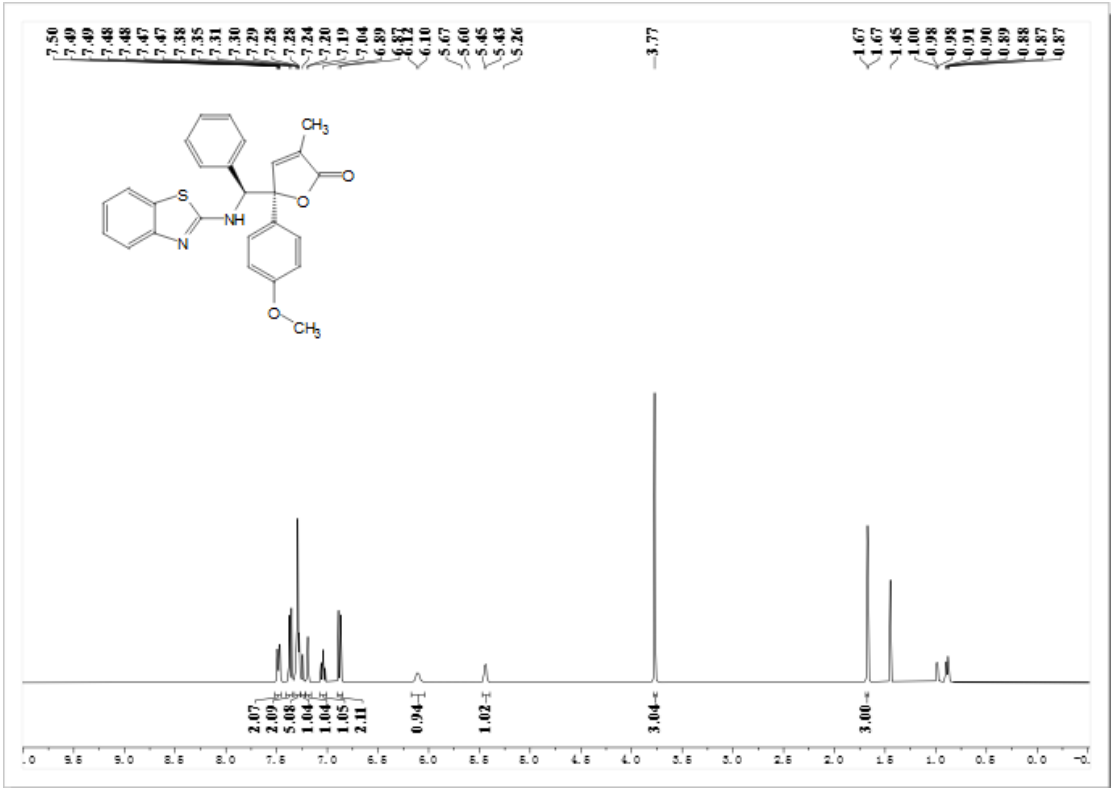
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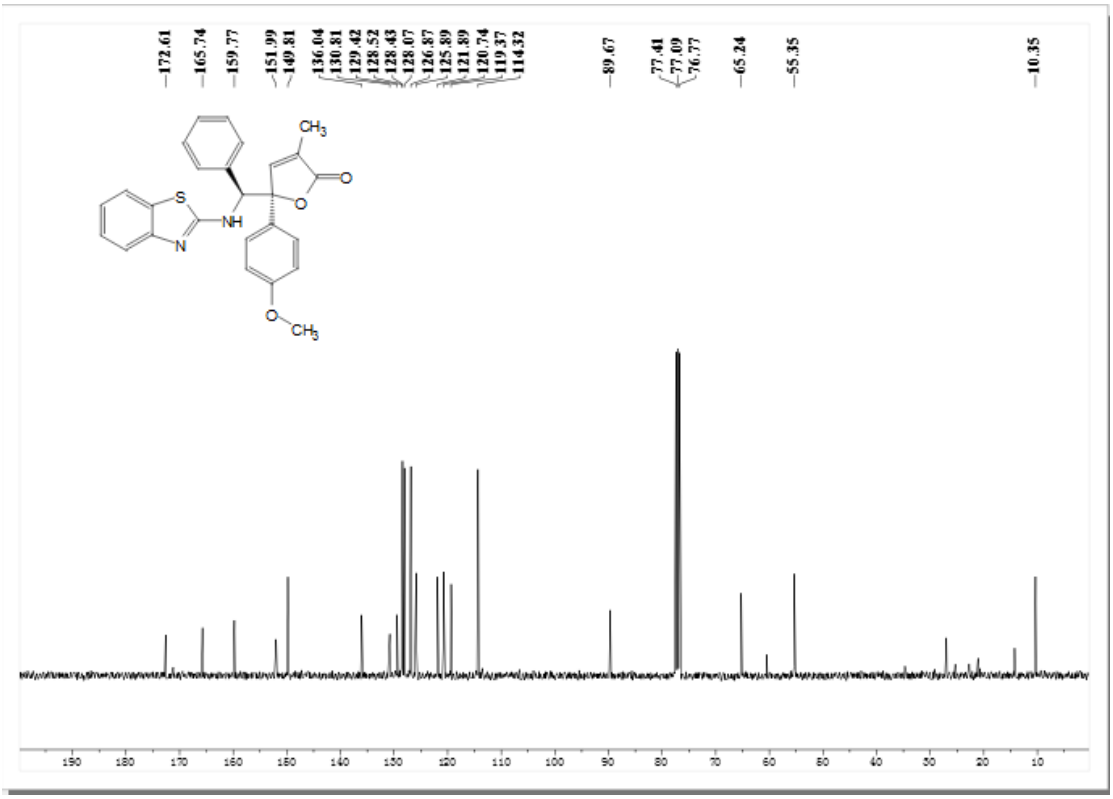
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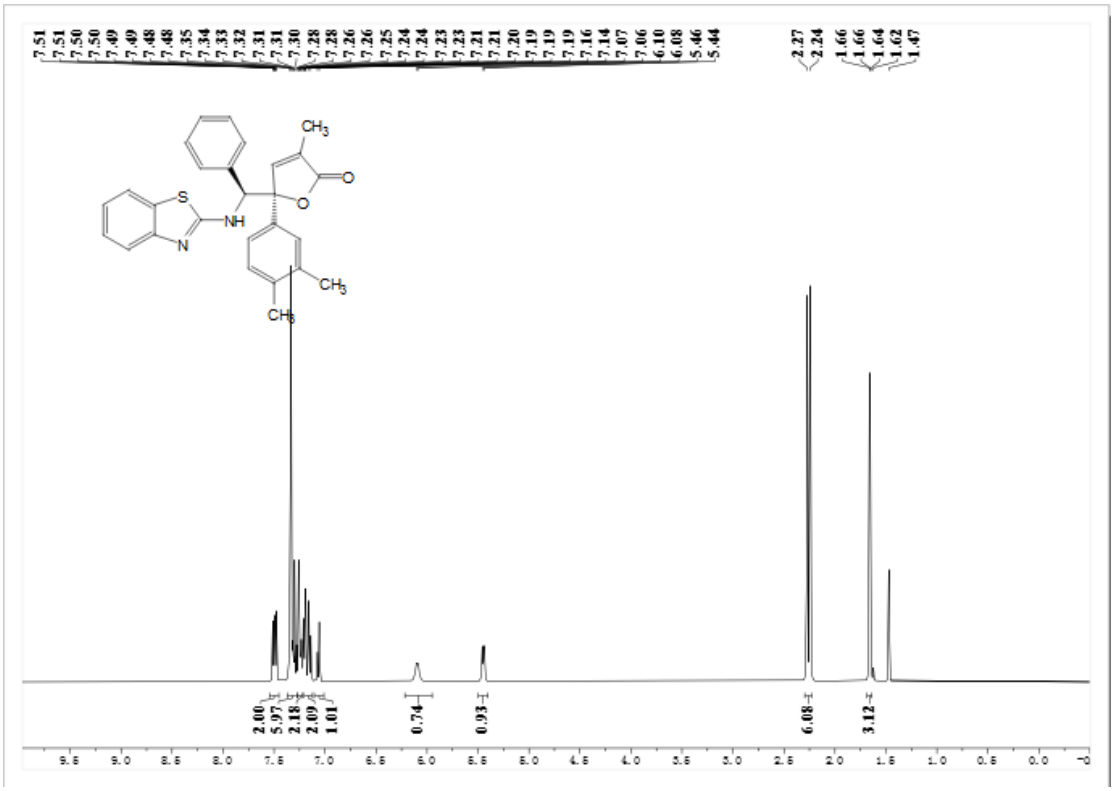
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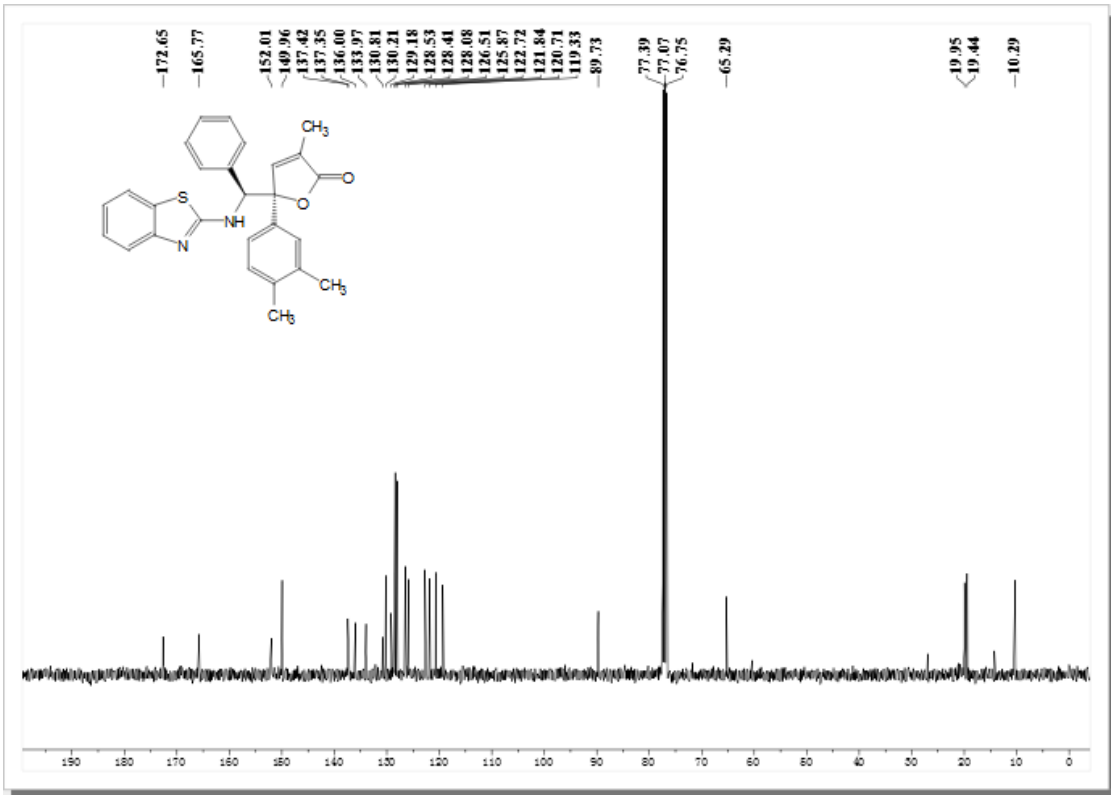
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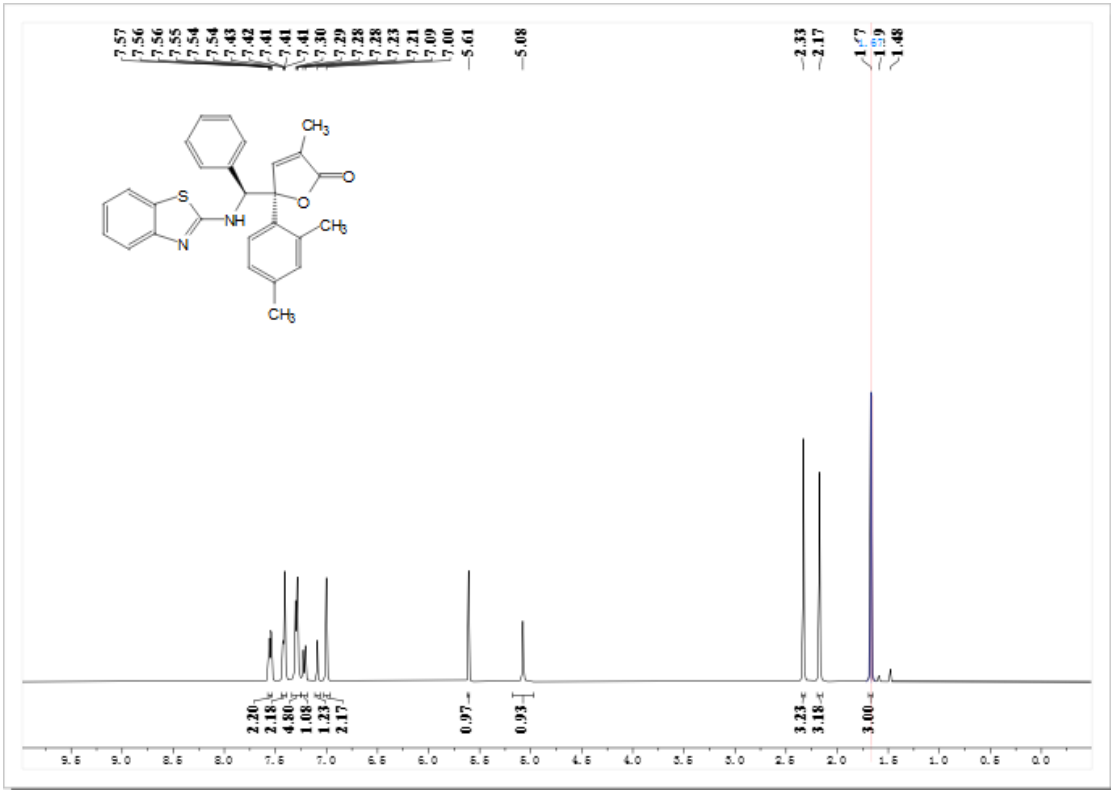
3ah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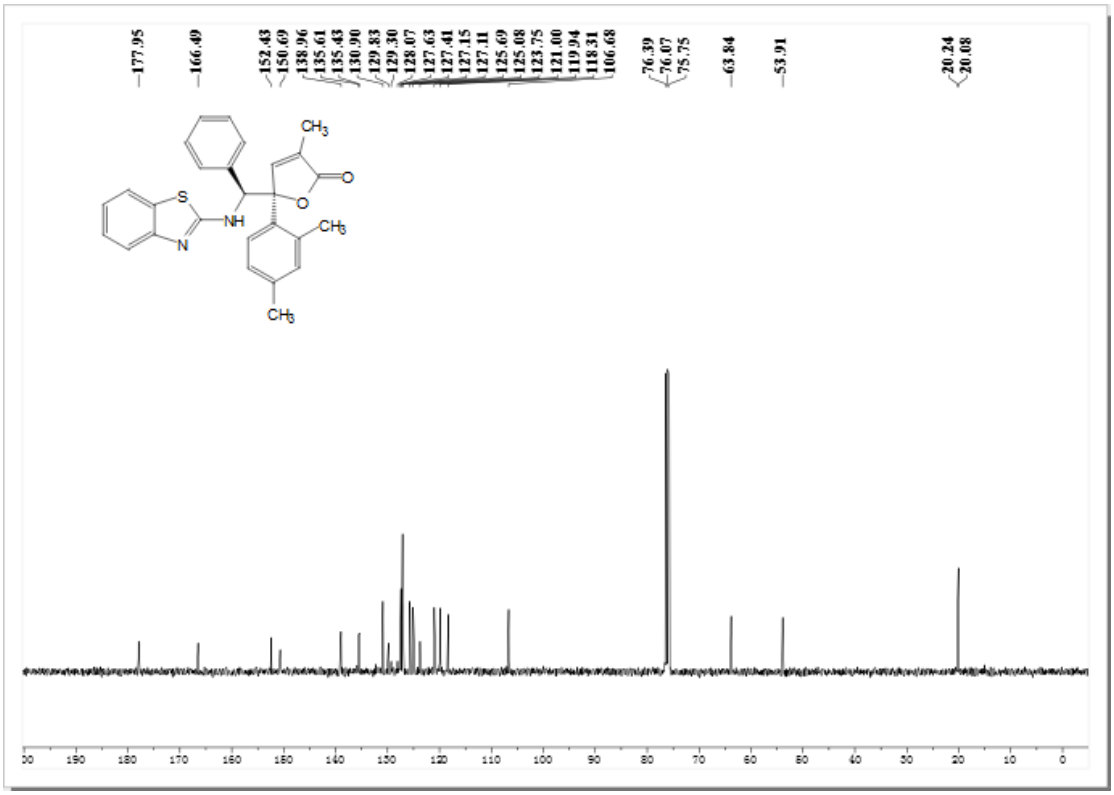
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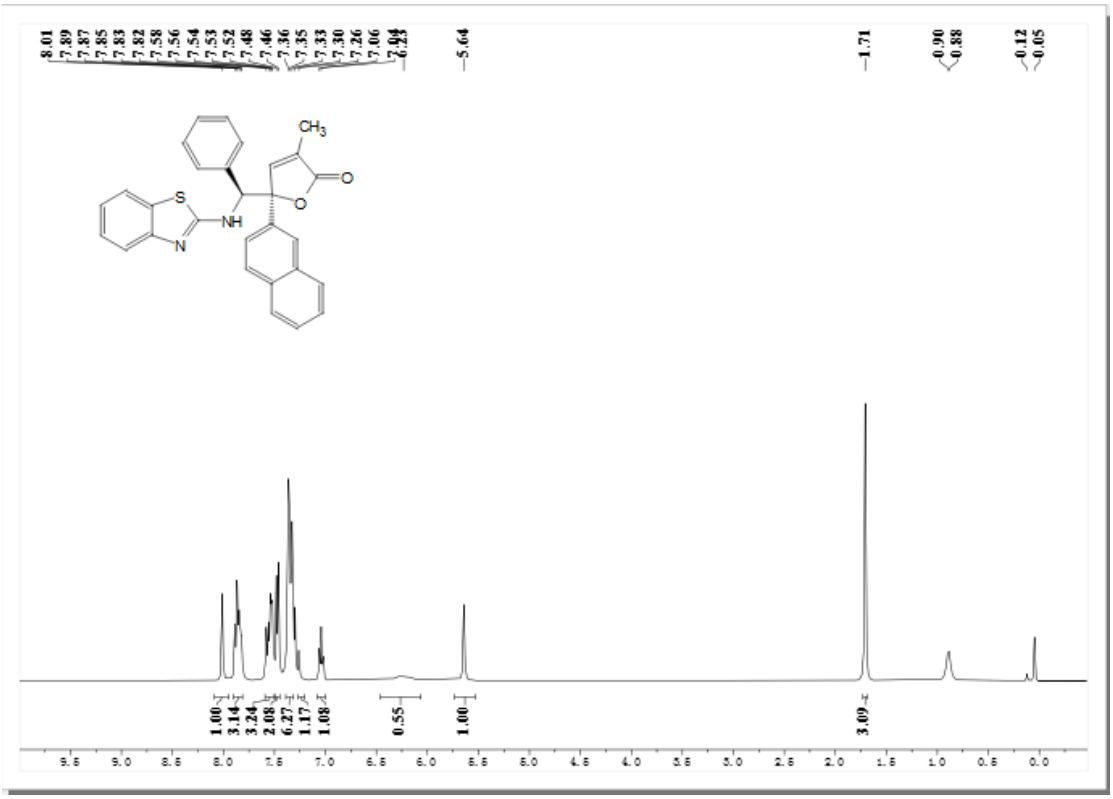
3ai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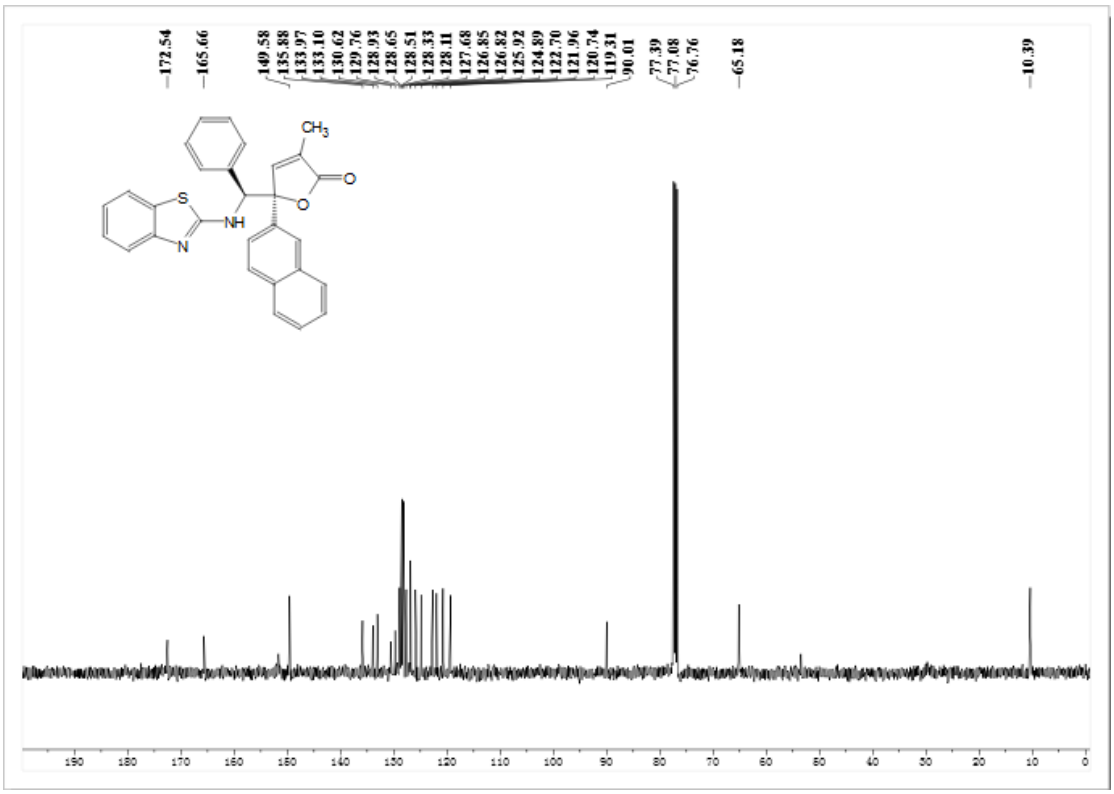
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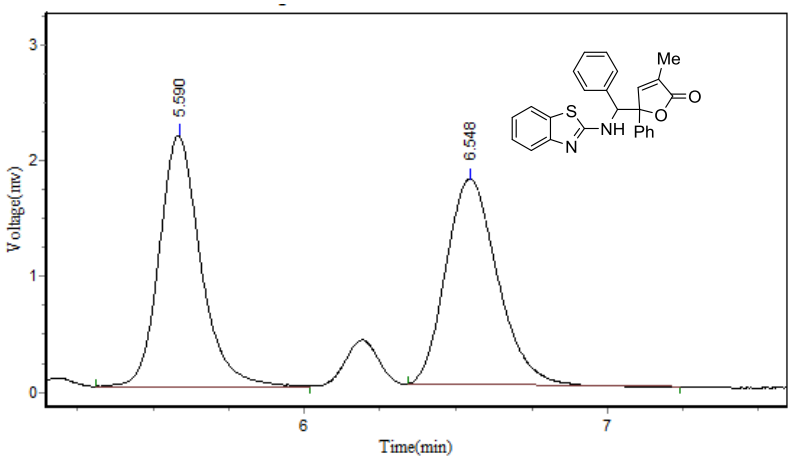
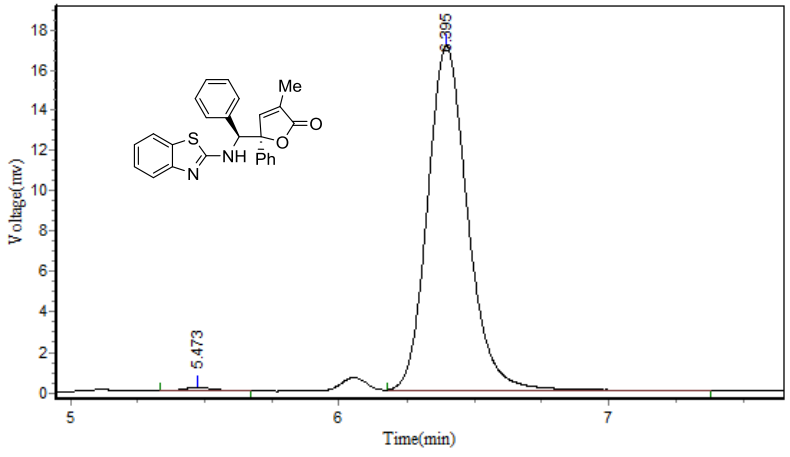
3aa



Results

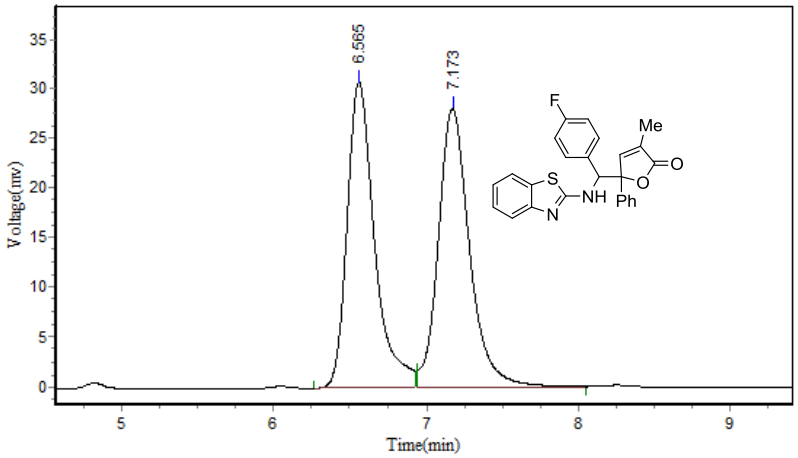
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.590	2162.224	20382.400	48.9888
2		6.548	1775.713	21223.801	51.0111
Total			3937.937	41606.201	100.0000



Results

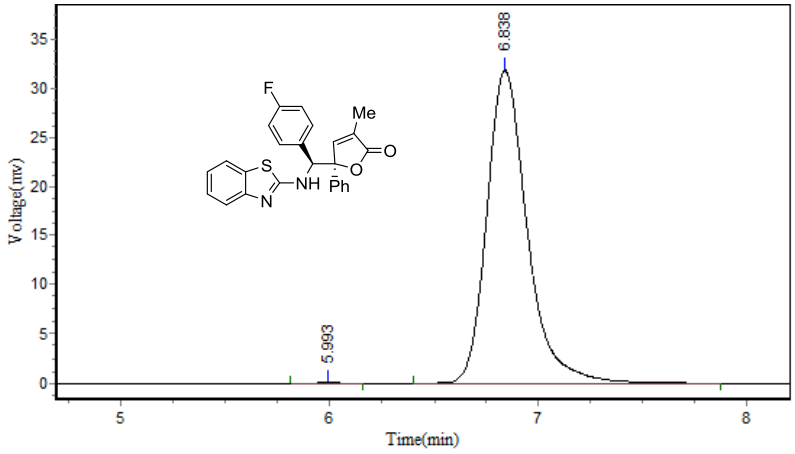
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.473	176.476	1467.900	0.8245
2		6.395	17068.521	176569.094	99.1755
Total			17244.997	178036.994	100.0000

3ba



Results

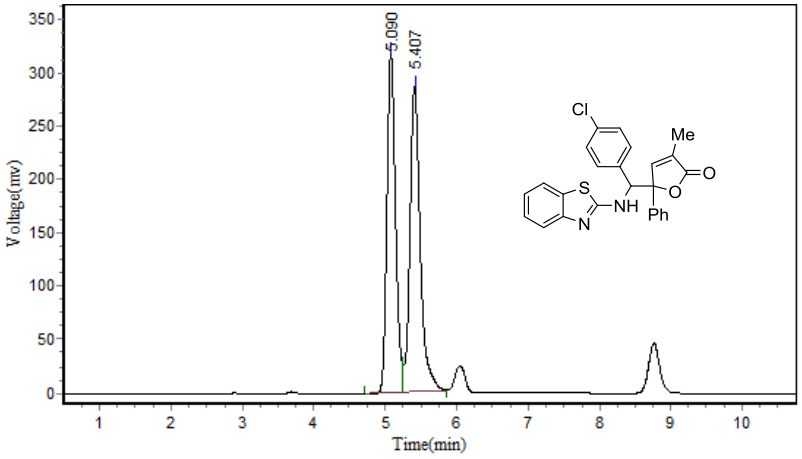
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.565	30718.047	384096.906	49.0605
2		7.173	27994.281	398807.406	50.9395
Total			58712.328	782904.313	100.0000



Results

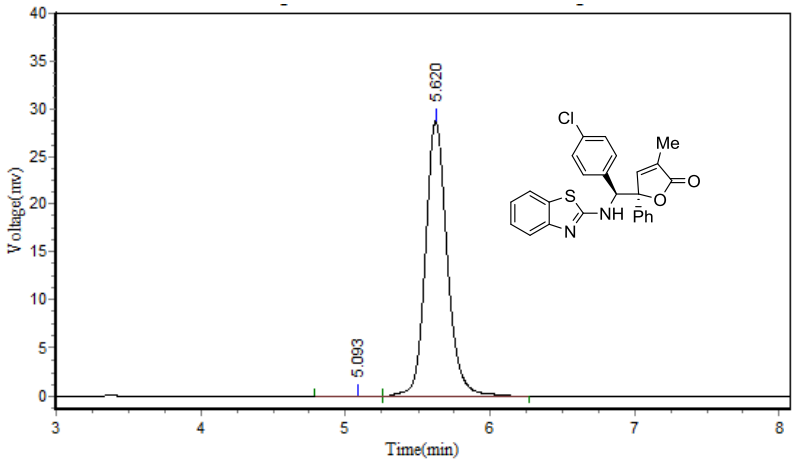
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.993	107.986	957.200	0.2218
2		6.838	31869.359	430657.906	99.7782
Total			31977.345	431615.106	100.0000

3ca



Results

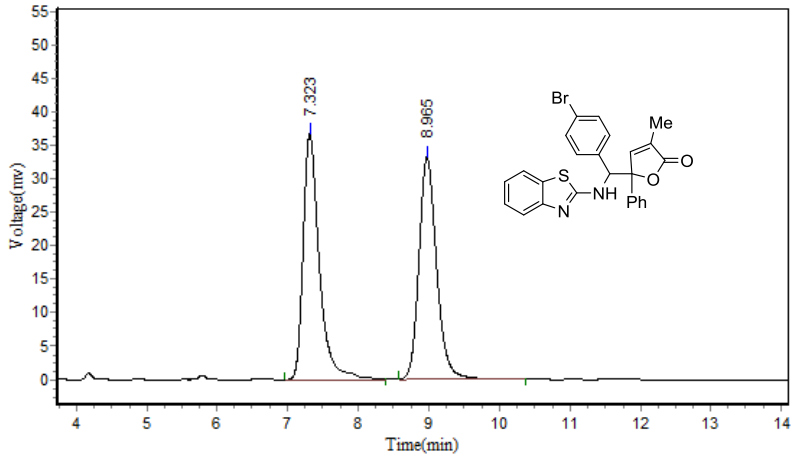
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.090	318101.719	2679002.250	47.7663
2		5.407	287463.000	2929560.000	52.2337
Total			605564.719	5608562.250	100.0000



Results

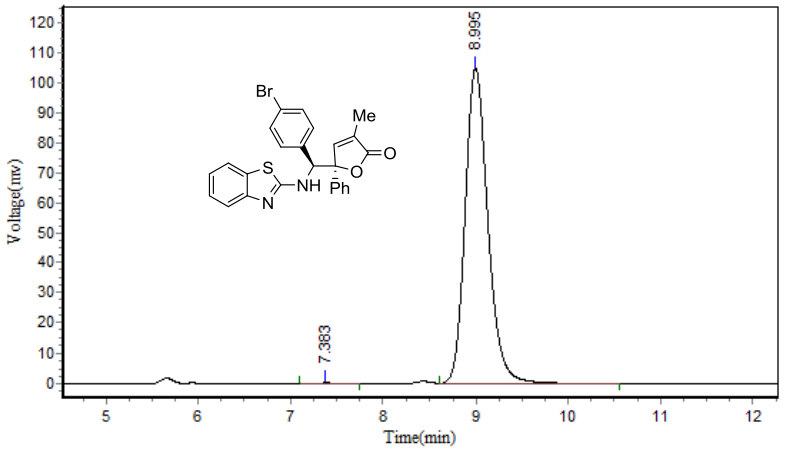
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.093	87.992	776.649	0.2568
2		5.620	28744.328	301633.625	99.7432
Total			28832.320	302410.274	100.0000

3da



Results

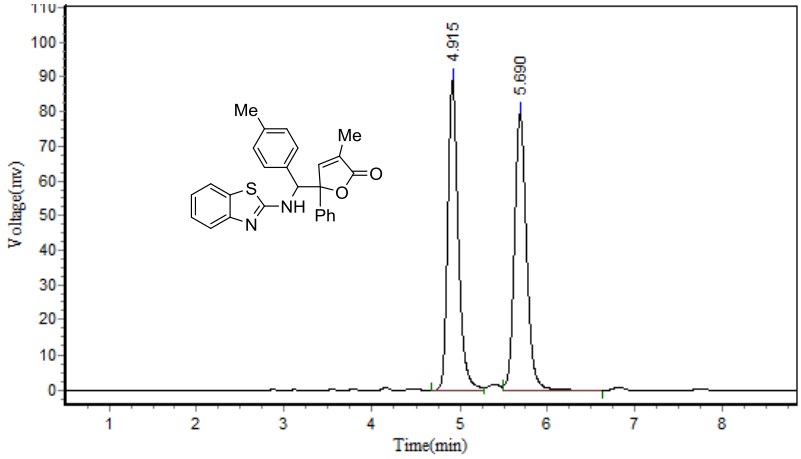
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.323	36642.730	583914.813	50.1213
2		8.965	33078.129	581089.250	49.8787
Total			69720.859	1165004.063	100.0000



Results

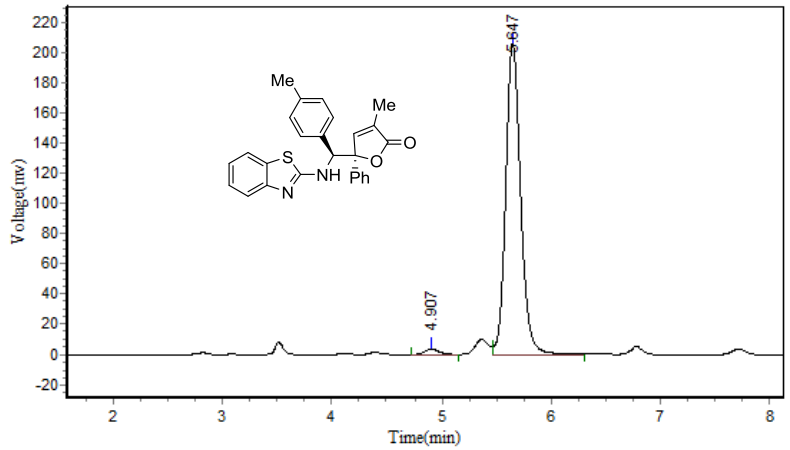
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.383	339.604	6430.181	0.3567
2		8.995	104851.289	1796489.125	99.6433
Total			105190.893	1802919.306	100.0000

3ea



Results

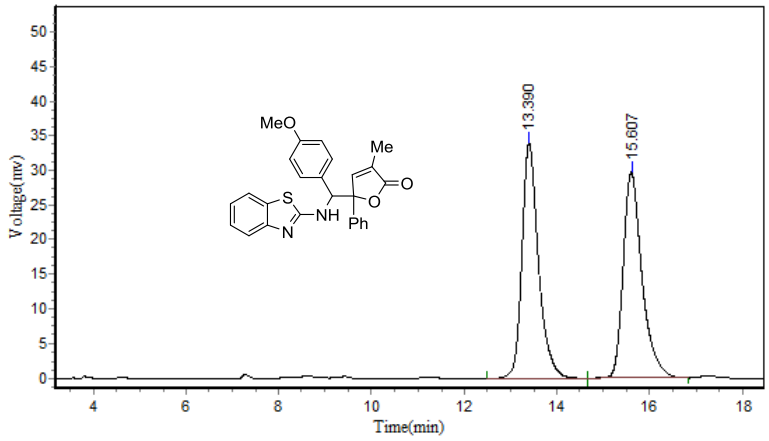
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.915	88748.484	752909.375	49.5915
2		5.690	79226.227	765314.375	50.4085
Total			167974.711	1518223.750	100.0000



Results

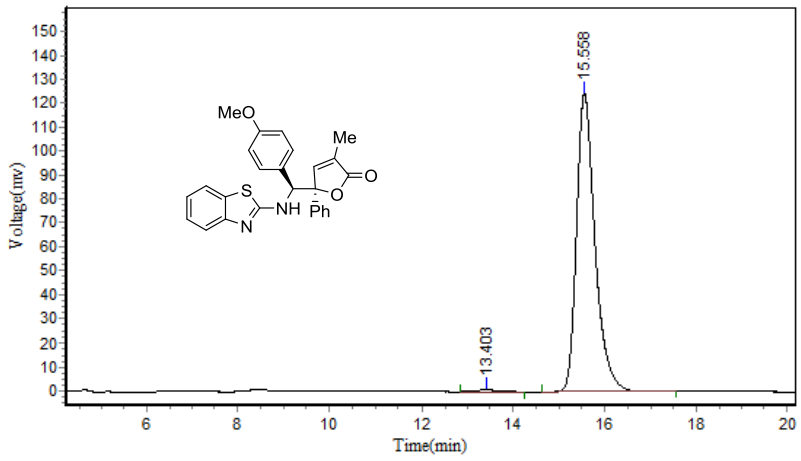
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.907	3562.965	31371.973	1.5846
2		5.647	206039.703	1948463.375	98.4154
Total			209602.668	1979835.348	100.0000

3fa



Results

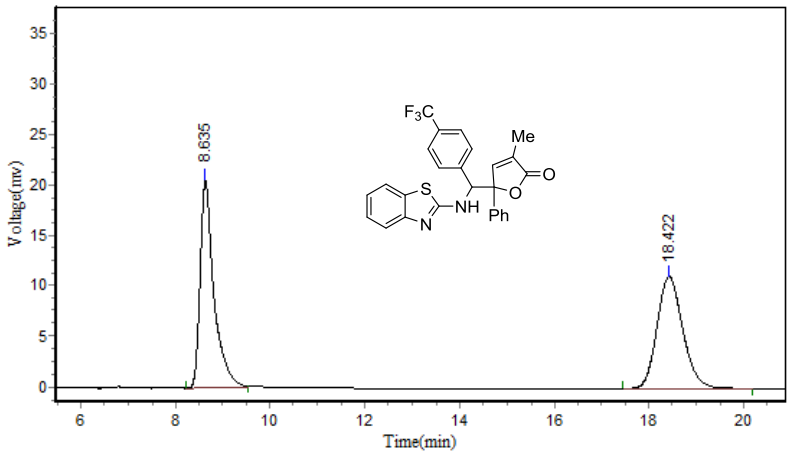
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.390	33942.000	840903.125	50.0400
2		15.607	29665.115	839558.813	49.9600
Total			63607.115	1680461.938	100.0000



Results

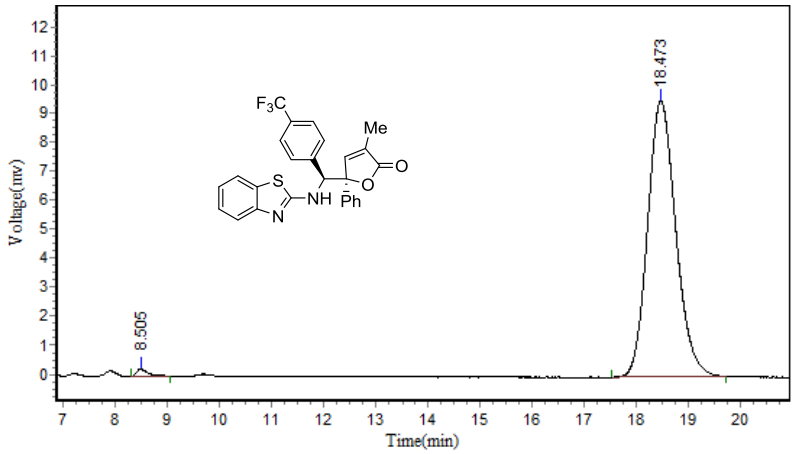
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.403	861.014	21613.199	0.6210
2		15.558	124159.422	3458809.250	99.3790
Total			125020.436	3480422.449	100.0000

3ga



Results

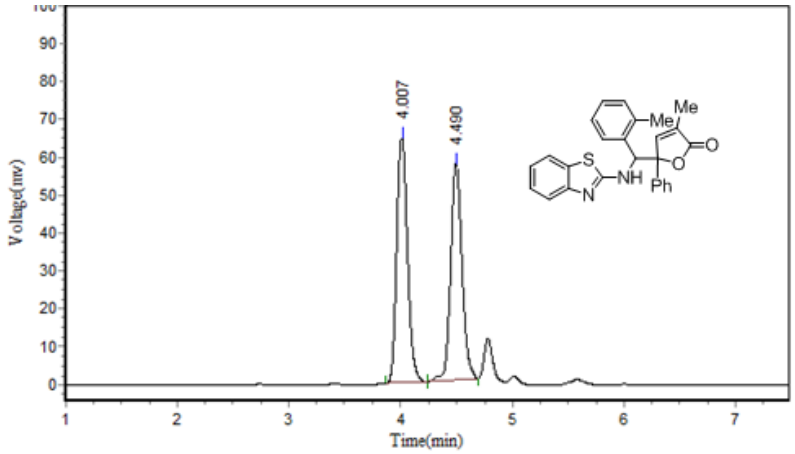
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		8.635	20449.176	414982.469	50.0638
2		18.422	11012.119	413924.875	49.9362
Total			31461.295	828907.344	100.0000



Results

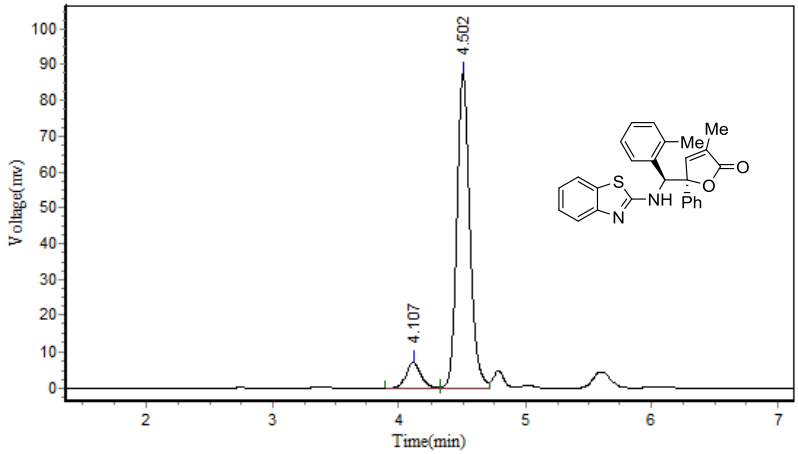
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		8.505	256.803	3740.050	1.0393
2		18.473	9504.573	356122.906	98.9607
Total			9761.376	359862.956	100.0000

3ha



Results

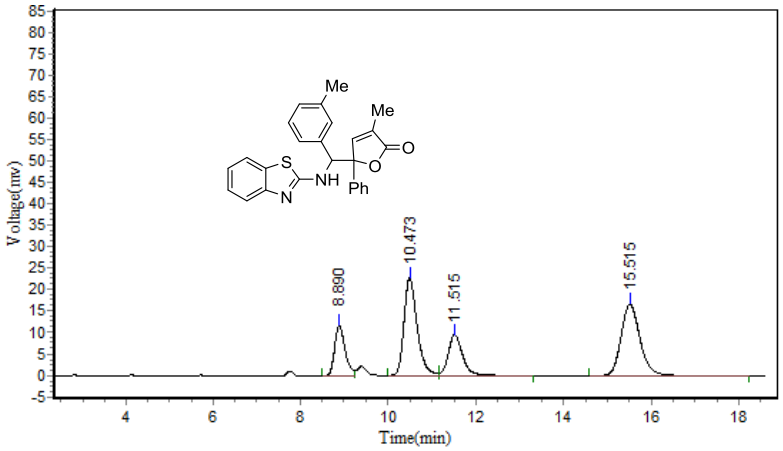
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.007	64832.566	406070.375	48.1166
2		4.490	58376.563	437860.281	51.8834
Total			123209.129	843930.656	100.0000



Results

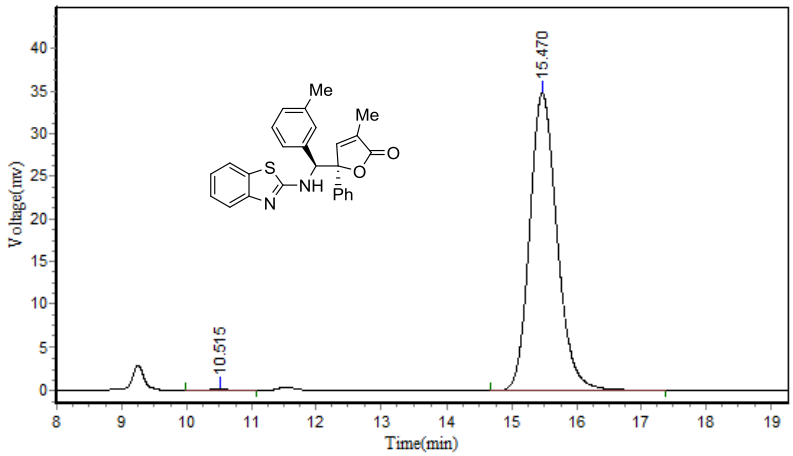
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.107	7167.818	55742.180	8.1843
2		4.502	87405.164	625346.500	91.8157
Total			94572.982	681088.680	100.0000

3ia



Results

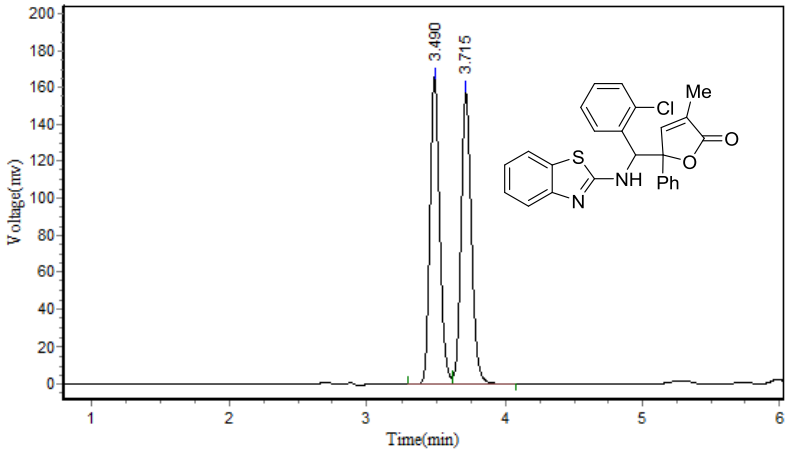
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		8.890	11591.886	192064.828	13.8288
2		10.473	22645.340	477109.563	34.3521
3		11.515	9485.716	220552.953	15.8799
4		15.515	16576.199	499152.500	35.9392
Total			60299.141	1388879.844	100.0000



Results

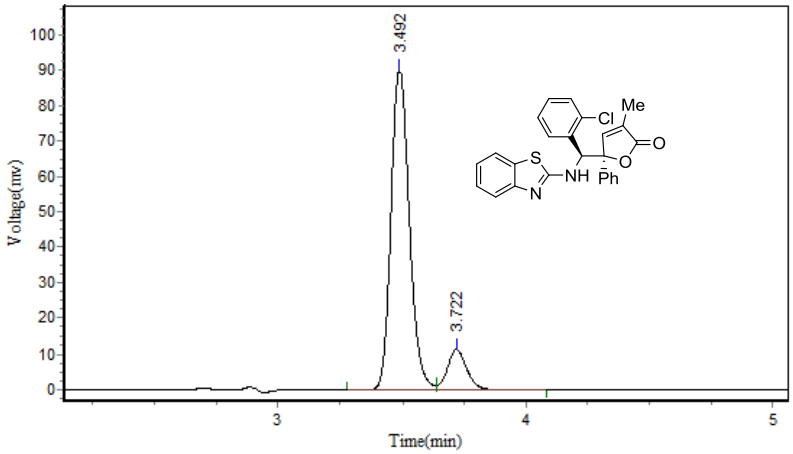
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.515	209.389	5215.250	0.5100
2		15.470	34775.742	1017320.625	99.4900
Total			34985.131	1022535.875	100.0000

3ja



Results

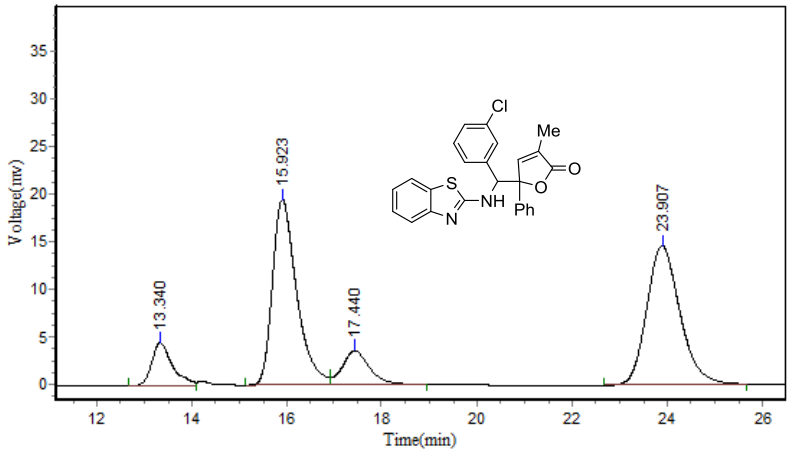
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.490	164819.406	812789.625	49.6504
2		3.715	155524.875	824234.438	50.3496
Total			320344.281	1637024.063	100.0000



Results

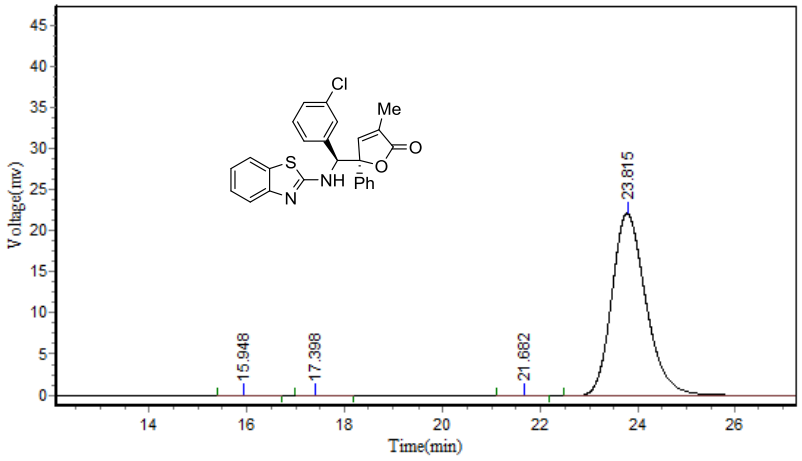
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.492	89533.742	442322.469	87.7281
2		3.722	11335.073	61874.711	12.2719
Total			100868.815	504197.180	100.0000

3ka



Results

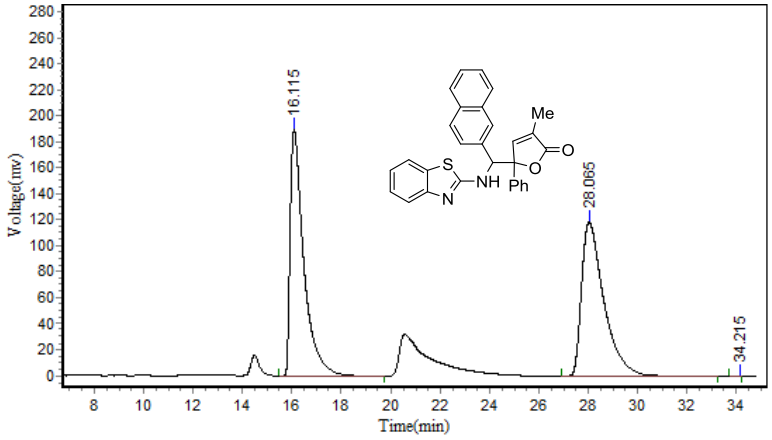
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.340	4372.527	132262.484	7.8315
2		15.923	19471.693	691424.688	40.9403
3		17.440	3602.618	148966.406	8.8205
4		23.907	14598.172	716205.813	42.4077
Total			42045.010	1688859.391	100.0000



Results

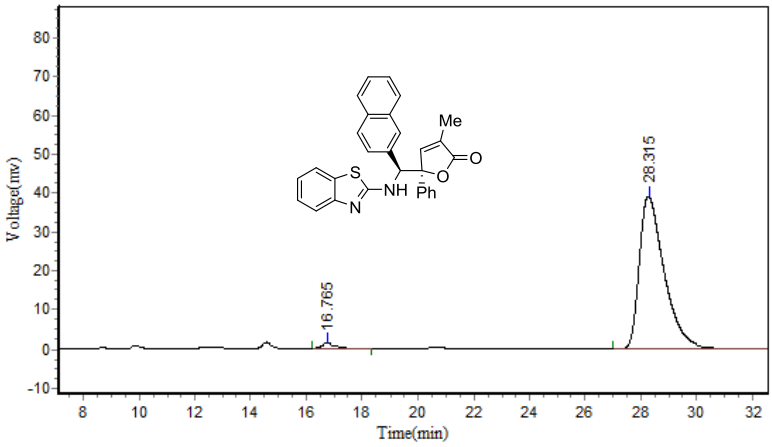
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		15.948	97.478	3320.800	0.3039
2		17.398	25.667	906.800	0.0830
3		21.682	9.000	279.300	0.0256
4		23.815	22127.338	1088205.875	99.5876
Total			22259.482	1092712.775	100.0000

3la



Results

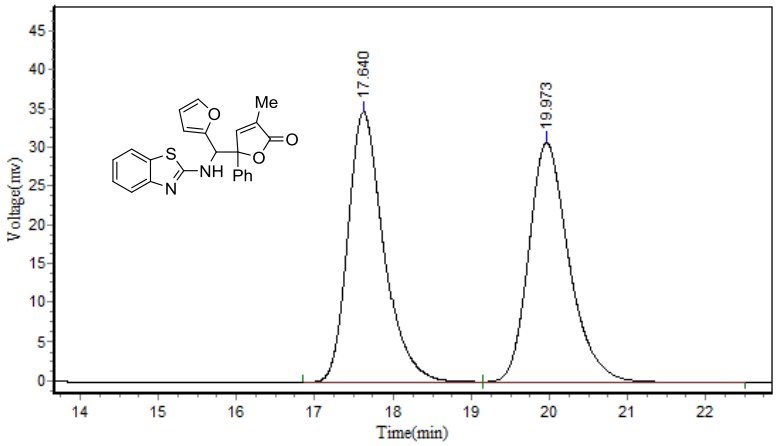
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.115	188732.109	7214528.500	49.4188
2		28.065	118072.328	7384203.000	50.5811
3		34.215	1.200	19.000	0.0001
Total			306805.638	14598750.500	100.0000



Results

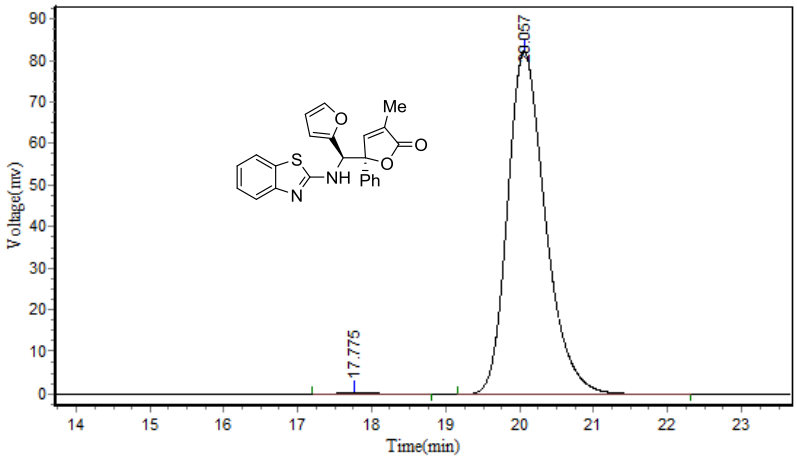
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.765	1041.391	41091.699	1.6737
2		28.315	39098.180	2414083.500	98.3263
Total			40139.571	2455175.199	100.0000

3ma



Results

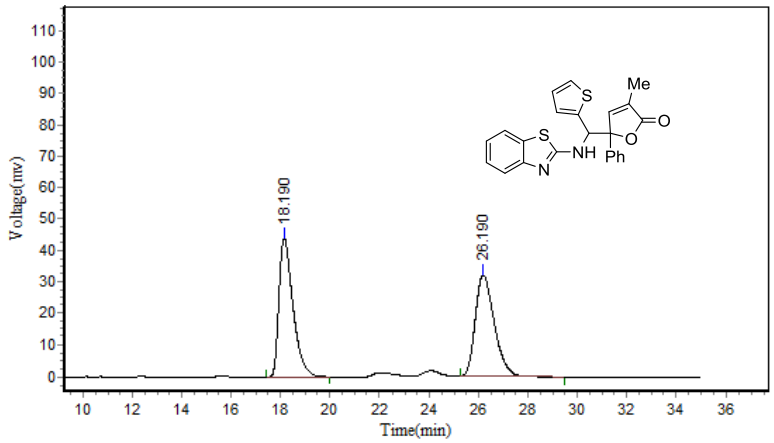
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.640	34744.059	1096935.750	50.0770
2		19.973	30662.920	1093560.500	49.9230
Total			65406.979	2190496.250	100.0000



Results

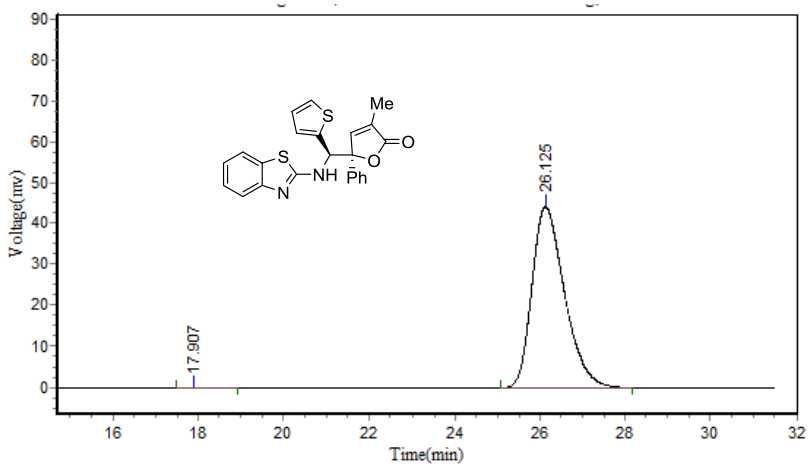
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.775	387.207	13505.450	0.4590
2		20.057	82293.594	2928905.250	99.5410
Total			82680.801	2942410.700	100.0000

3na



Results

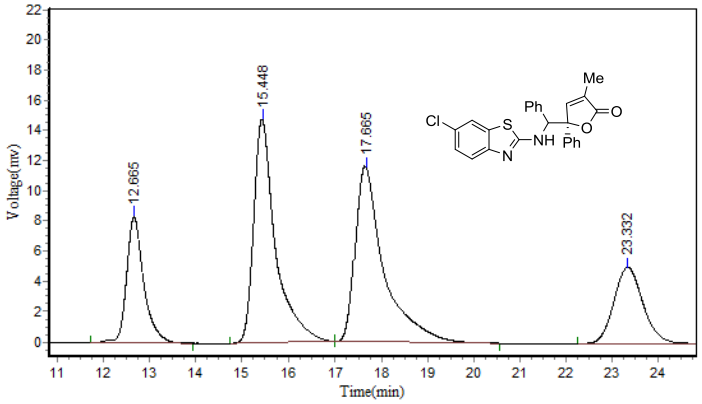
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		18.190	44005.809	1718210.750	49.9124
2		26.190	32144.914	1724242.000	50.0876
Total			76150.723	3442452.750	100.0000



Results

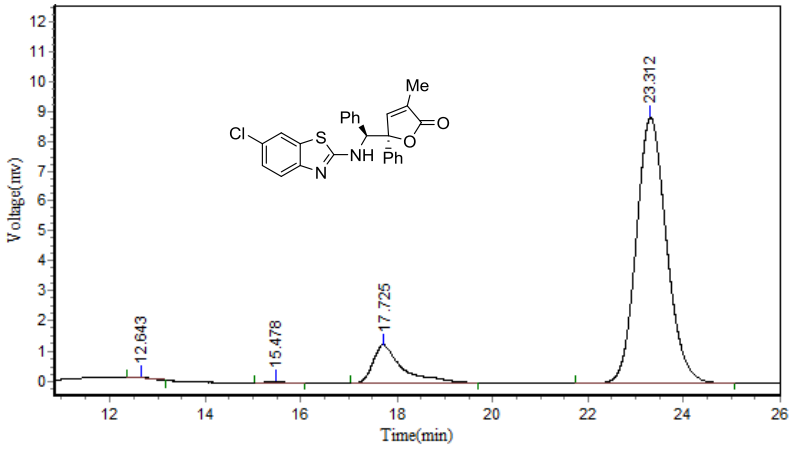
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.907	15.321	740.580	0.0318
2		26.125	43928.492	2327279.000	99.9682
Total			43943.813	2328019.580	100.0000

3oa



Results

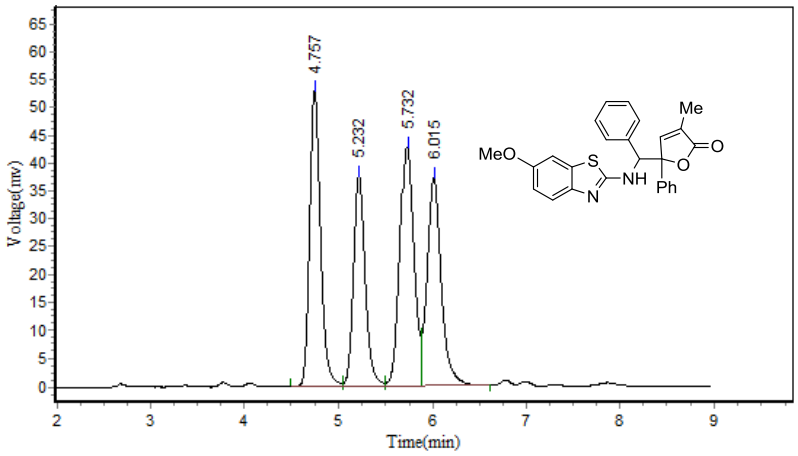
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.665	8298.426	216491.953	15.3854
2		15.448	14742.941	482512.688	34.2907
3		17.665	11579.873	490469.813	34.8562
4		23.332	4990.010	217648.906	15.4676
Total			39611.250	1407123.359	100.0000



Results

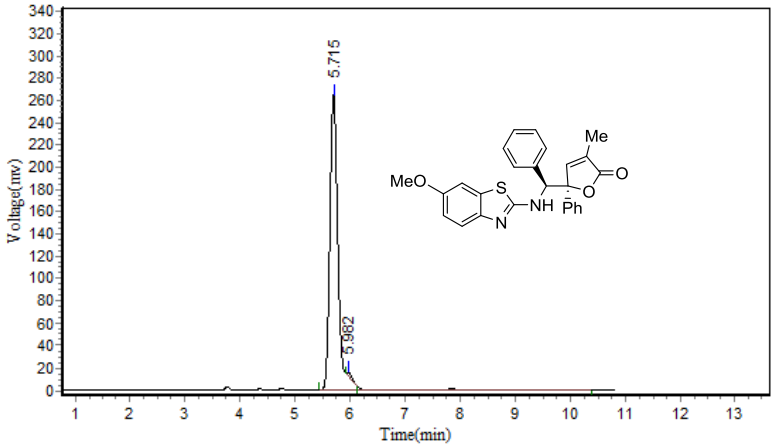
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.643	56.290	1231.400	0.2767
2		15.478	35.632	836.800	0.1880
3		17.725	1252.971	53366.398	11.9921
4		23.312	8873.702	389578.938	87.5432
Total			10218.596	445013.536	100.0000

3pa



Results

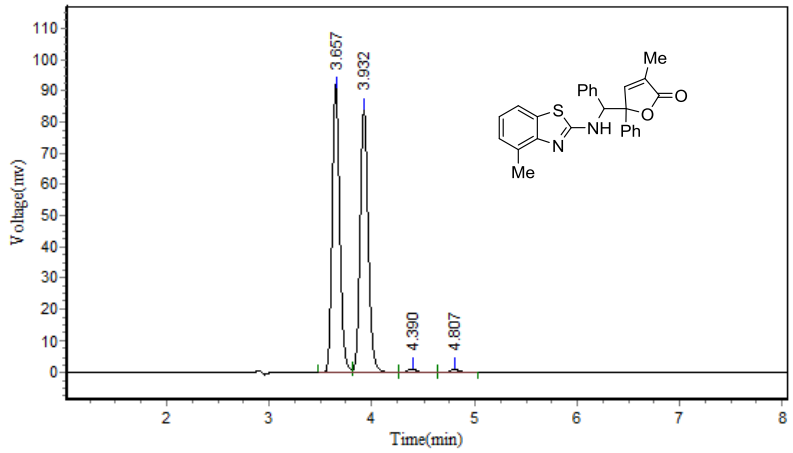
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.757	52852.281	426369.219	26.9697
2		5.232	37619.750	329139.688	20.8195
3		5.732	42879.402	431988.313	27.3252
4		6.015	37246.137	393421.063	24.8856
Total			170597.570	1580918.281	100.0000



Results

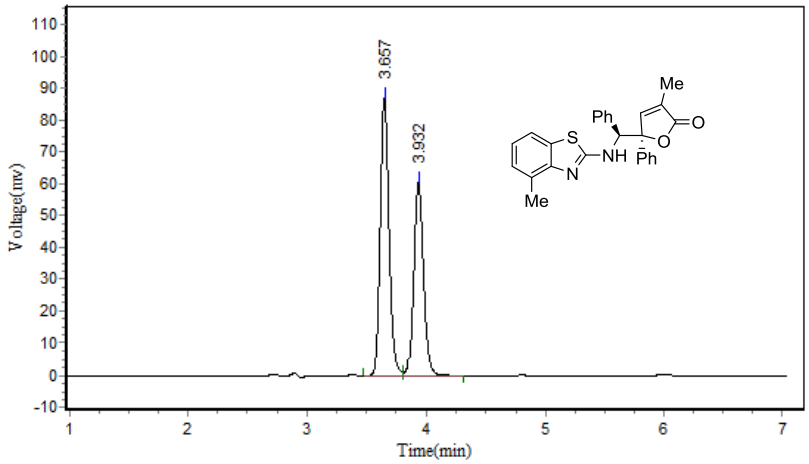
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.715	264031.438	2717820.500	99.1051
2		5.982	3831.750	24539.902	0.8948
Total			267863.188	2742360.402	100.0000

3qa



Results

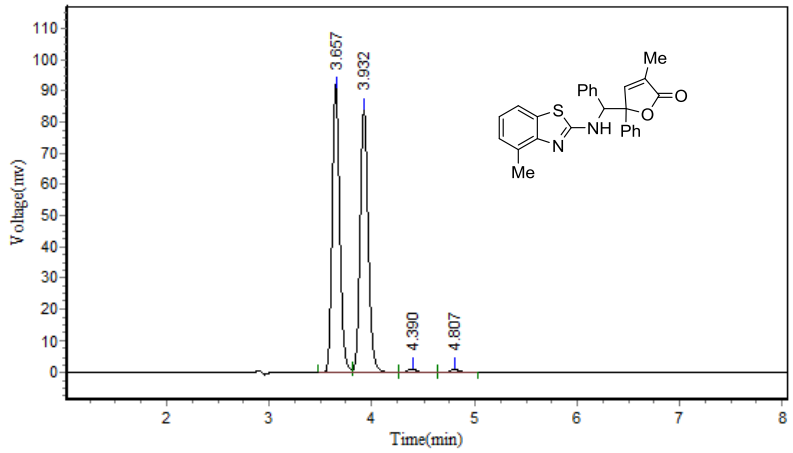
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.657	91448.500	468568.281	49.1732
2		3.932	84129.984	468263.844	49.1412
3		4.390	1295.794	8787.645	0.9222
4		4.807	1036.982	7274.027	0.7634
Total			177911.260	952893.797	100.0000



Results

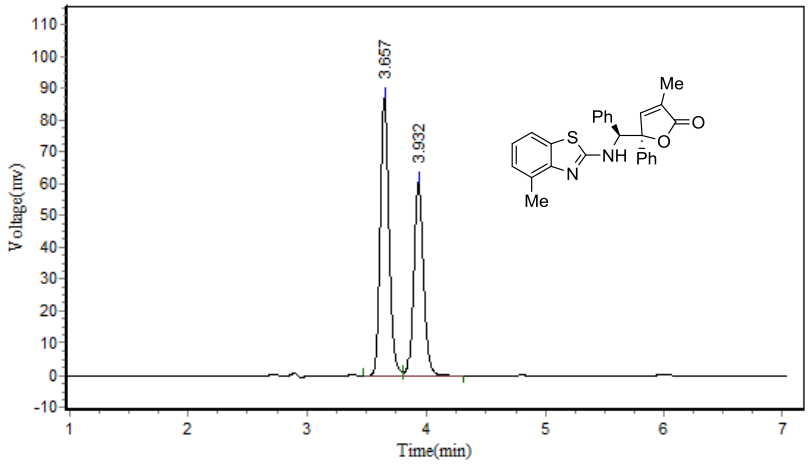
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.657	87119.328	448382.531	56.9726
2		3.932	60104.520	338631.563	43.0274
Total			147223.848	787014.094	100.0000

3qa



Results

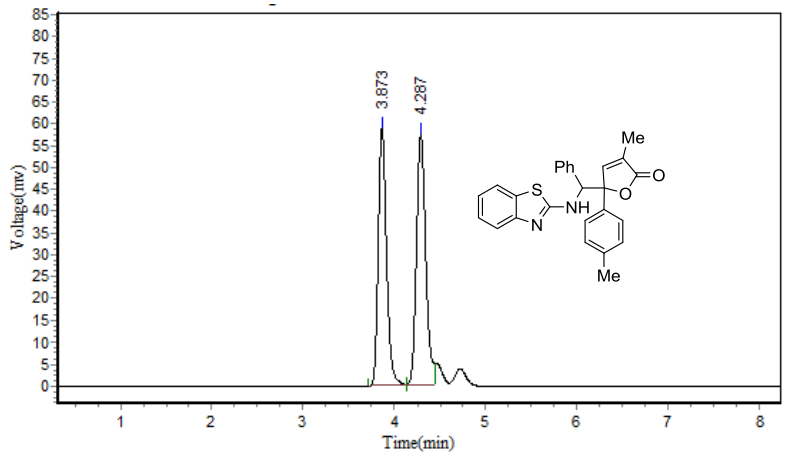
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.657	91448.500	468568.281	49.1732
2		3.932	84129.984	468263.844	49.1412
3		4.390	1295.794	8787.645	0.9222
4		4.807	1036.982	7274.027	0.7634
Total			177911.260	952893.797	100.0000



Results

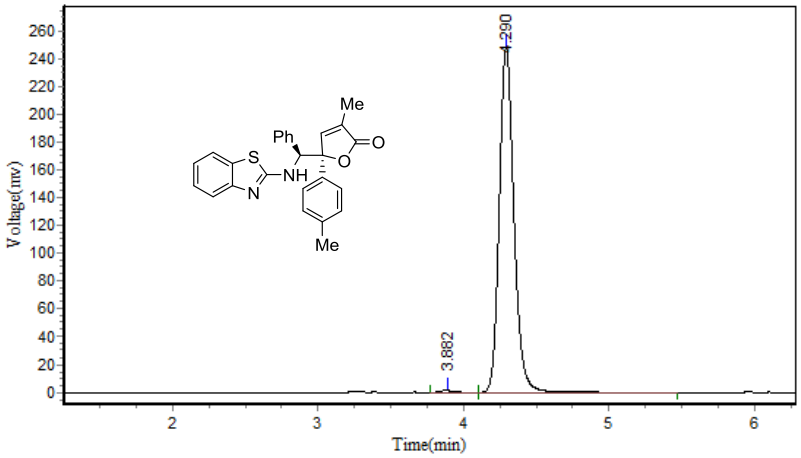
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.657	87119.328	448382.531	56.9726
2		3.932	60104.520	338631.563	43.0274
Total			147223.848	787014.094	100.0000

3ab



Results

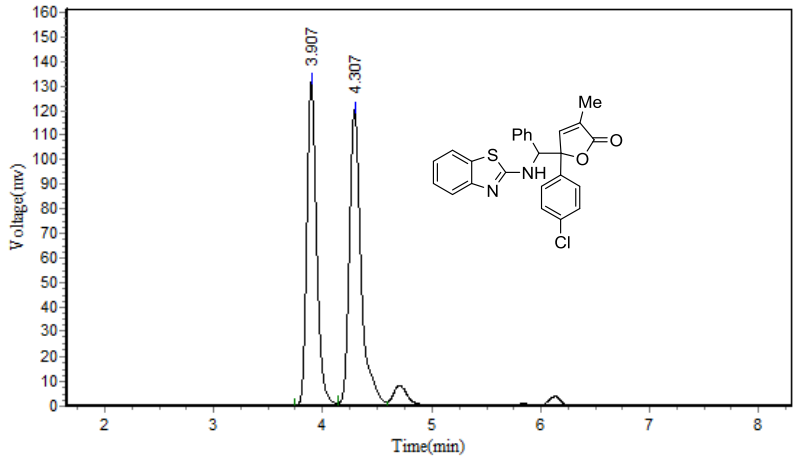
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.873	59124.480	369484.094	47.9306
2		4.287	57354.000	401389.000	52.0694
Total			116478.480	770873.094	100.0000



Results

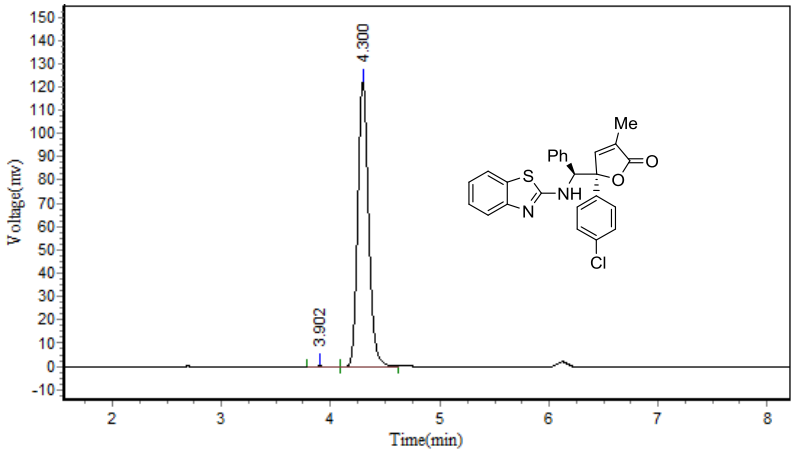
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.882	1426.520	9155.810	0.5201
2		4.290	248690.094	1751185.875	99.4799
Total			250116.614	1760341.685	100.0000

3ac



Results

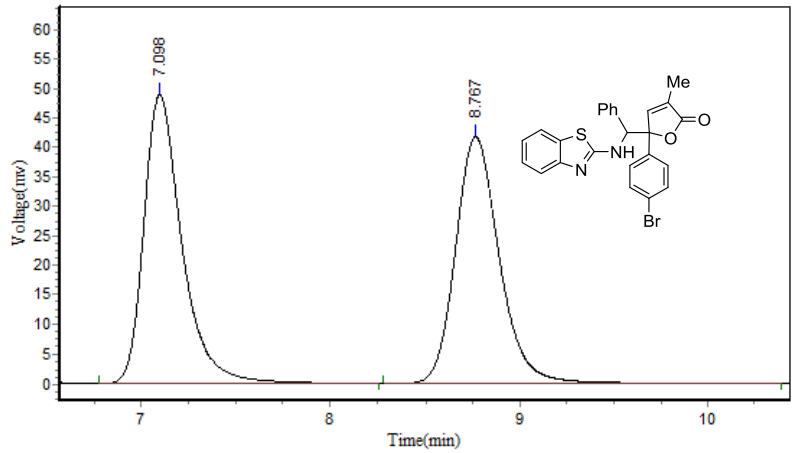
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.907	131240.828	824380.188	45.6768
2		4.307	119933.219	905572.313	50.1754
Total			259655.238	1804813.391	100.0000



Results

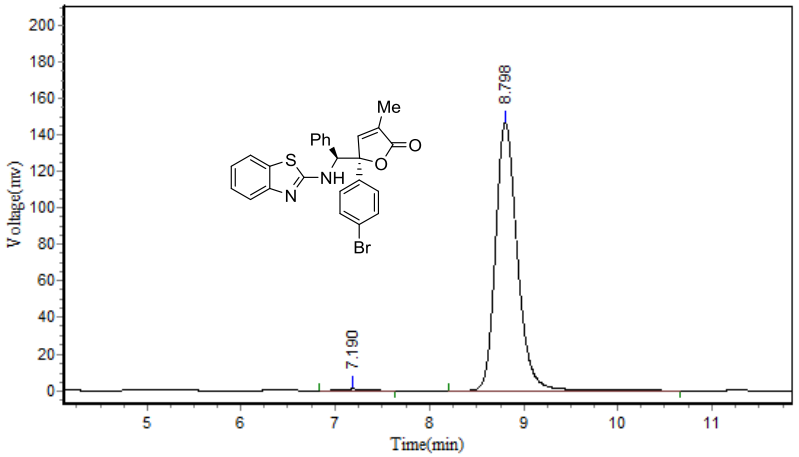
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.902	530.675	3354.475	0.3944
2		4.300	122676.539	847077.938	99.6056
Total			123207.214	850432.413	100.0000

3ad



Results

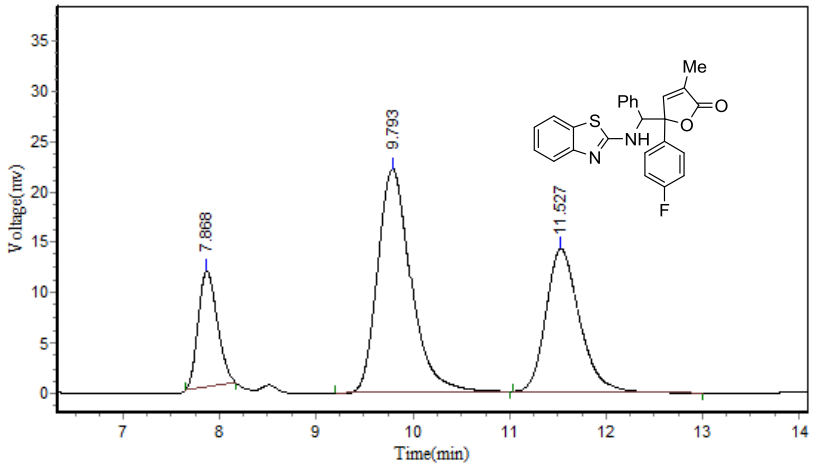
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.098	48841.211	682449.313	50.3877
2		8.767	41631.941	671946.688	49.6123
Total			90473.152	1354396.000	100.0000



Results

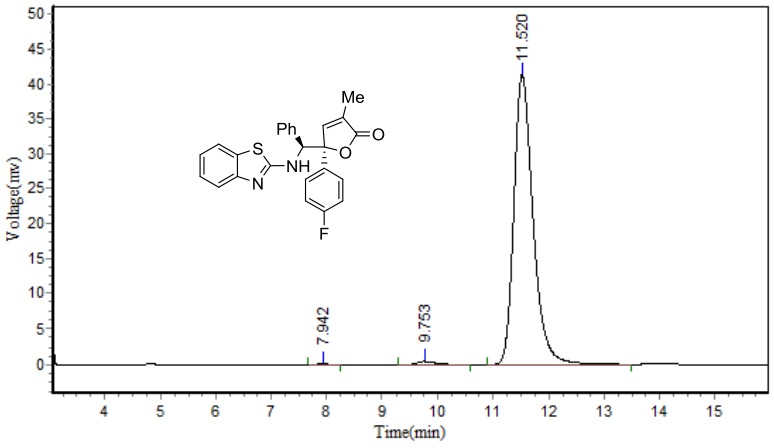
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.190	863.010	13530.850	0.5737
2		8.798	146268.453	2345035.750	99.4263
Total			147131.463	2358566.600	100.0000

3ae



Results

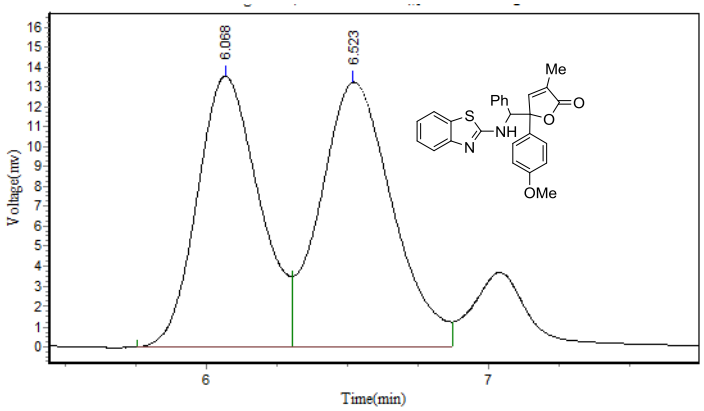
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.868	11467.375	156836.141	15.3149
2		9.793	22267.996	526161.375	51.3790
3		11.527	14123.021	341080.938	33.3061
Total			47858.393	1024078.453	100.0000



Results

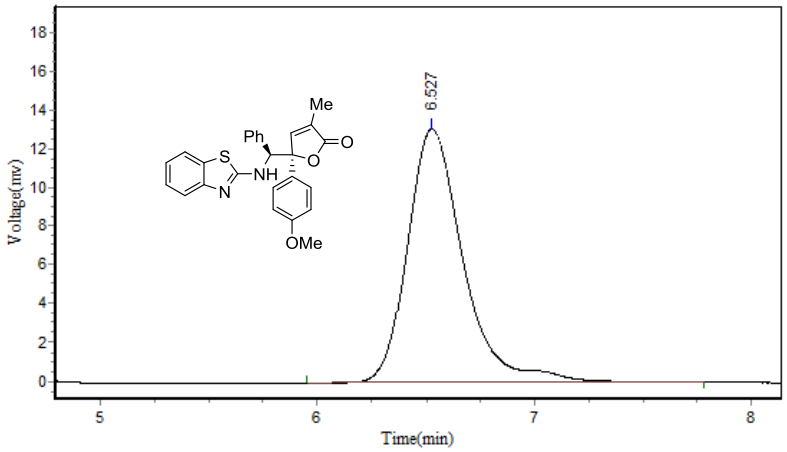
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.942	170.293	1778.250	0.1768
2		9.753	477.539	11365.350	1.1301
3		11.520	41320.012	992572.750	98.6931
Total			41967.844	1005716.350	100.0000

3af



Results

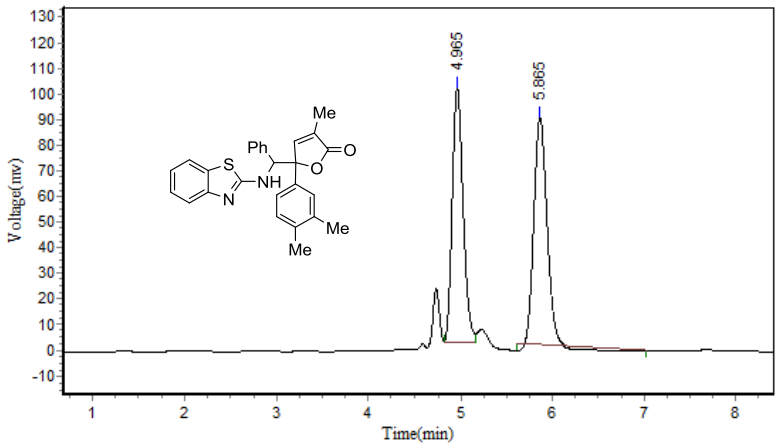
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.068	13549.712	207473.734	46.8842
2		6.523	13257.220	235050.328	53.1158
Total			26806.932	442524.063	100.0000



Results

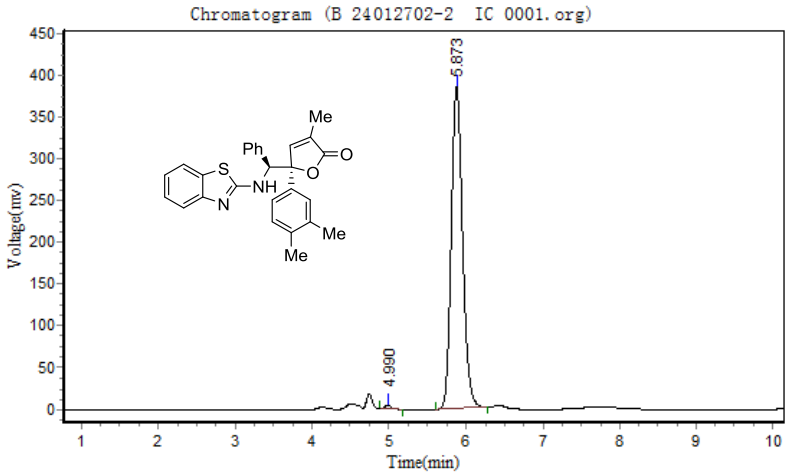
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.527	13100.326	234027.797	100.0000
Total			13100.326	234027.797	100.0000

3ag



Results

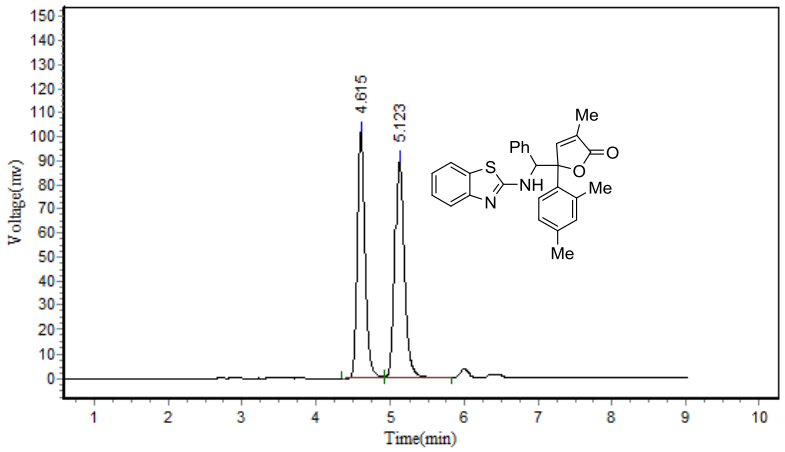
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.965	102447.297	885683.188	48.0509
2		5.865	90596.164	957533.688	51.9491
Total			193043.461	1843216.875	100.0000



Results

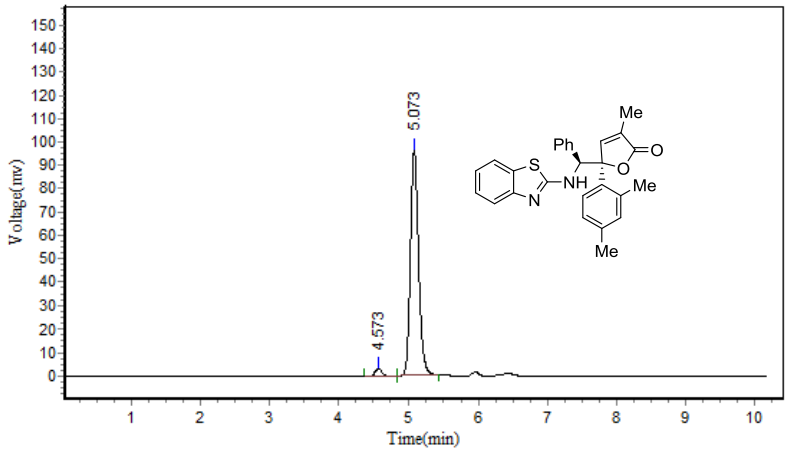
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.990	4181.372	25121.850	0.6338
2		5.873	385666.781	3938353.500	99.3662
Total			389848.153	3963475.350	100.0000

3ah



Results

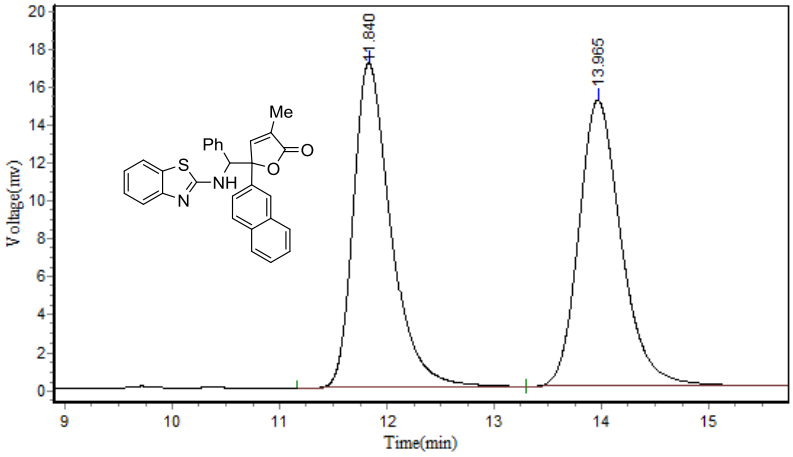
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.615	102133.281	745479.500	49.5813
2		5.123	89738.961	758069.188	50.4187
Total			191872.242	1503548.688	100.0000



Results

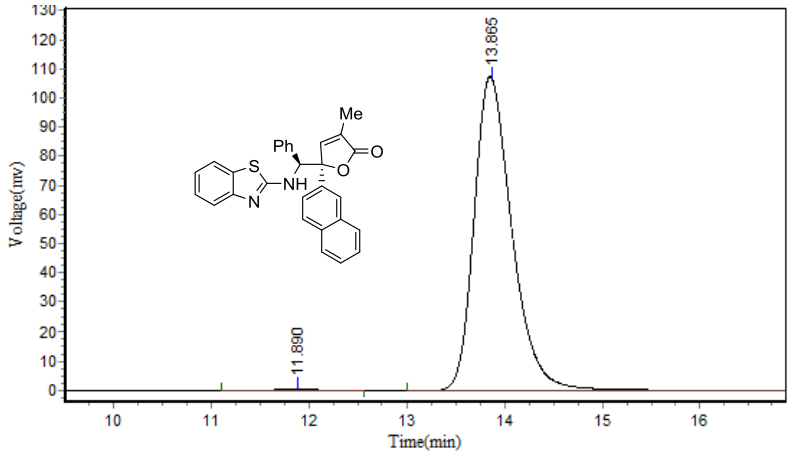
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.573	3194.471	23705.730	2.8733
2		5.073	96785.398	801320.313	97.1267
Total			99979.869	825026.043	100.0000

3ai



Results

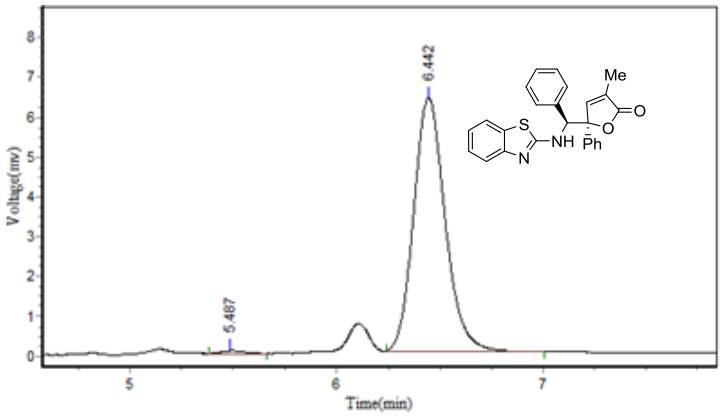
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.840	17109.646	412744.344	49.8736
2		13.965	15054.808	414837.156	50.1264
Total			32164.454	827581.500	100.0000



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.890	497.109	15744.400	0.5420
2		13.865	107114.109	2888993.000	99.4580
Total			107611.218	2904737.400	100.0000

Scale up preparation of 3aa



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.487	73.632	532.450	0.7893
2		6.442	6343.774	66926.805	99.2107
Total			6417.406	67459.255	100.0000