

**Asymmetric Organocatalytic Vinylogous Mannich Reaction of
3-Methyl-5-arylfuran-2(3*H*)-ones with *N*-(2-pyridinesulfonyl)
Imines: Enantioselective Synthesis of δ -Amino γ,γ -Disubstituted
Butenolides**

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Supporting Information

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1. General experimental information

Reagents were purchased from commercial sources and were used as received unless mentioned otherwise. Reactions were monitored by TLC. ^1H NMR and ^{13}C NMR spectra were recorded in CDCl_3 and $\text{DMSO}-d_6$. ^1H 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, $\text{DMSO}-d_6$ at 2.50 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, 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 (CDCl_3 at 77.20 ppm, $\text{DMSO}-d_6$ at 39.51 ppm). Melting points of major diastereoisomer were recorded on a Büchi Melting Point B-545. Specific rotation of major diastereoisomer were determined by Perkin Elmer 341

2. General procedure for the synthesis of starting materials.

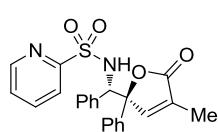
N-(2-pyridinesulfonyl)imines **1a-o** were prepared according to known literature procedures.^{1,2} Various 3-methyl-5-arylfuran-2(3*H*)-ones **2a-e** were prepared according to the literature.^{3,4}

References:

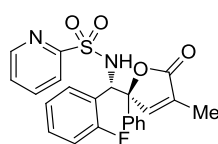
1. S. Diltz, G. Aguirre, F. Ortega, P. J. Walsh, *Tetrahedron:Asymmetry*. 1997, **8**, 3559.
2. S. Morales, F. G. Guijarro, J. L. G. Ruano, M. B. Cid, *J. Am. Chem. Soc.* 2014, **136**, 1082.
3. S. Žari, M. Kudrjashova, T. Pehk, M. Lopp and T. Kanger, *Org. Lett.*, 2014, **16**, 1740.
4. D. J. Jones, and V. C. Gibson, *Heterocycles*, 2006, **68**, 1128.

3. General procedure for the synthesis of compounds 3a-r.

In an ordinary vial equipped with a magnetic stirring bar, the compounds **2** (0.15 mmol, 1.5 equiv), catalyst **B** (5 mol%) were dissolved in mesitylene (2 mL), and then the mixture was stirred -10 °C for the 10 min. Then compounds **2** (0.10 mmol, 1.0 equiv) was added to the reaction system. After completion of the reaction as indicated by TLC, the major diastereoisomer of **3** were isolated by flash chromatography on silica gel (petroleum ether/ ethyl acetate = 10/1~2/1).

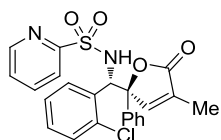


***N*-(*(S)*-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(phenyl)methyl)pyridine-2-sulfonamide (3a)** White solid; 35.1 mg, 84% yield; 88:12 dr, 95:5 er; $[\alpha]_D^{20} = -73.7$ (c. 0.97, CHCl_3); m.p. 207.2-208.3 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_R = 33.4$ min (minor), 37.0 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.6 (s, 3H), 5.03 (d, $J = 9.7$ Hz, 1H), 5.85 (d, $J = 9.6$ Hz, 1H), 6.81-6.89 (m, 2H), 7.02 (m, 3H), 7.15-7.24 (m, 2H), 7.27-7.37 (m, 5H), 7.48-7.57 (m, 2H), 8.36 (d, $J = 4.5$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.3, 64.5, 89.4, 121.5, 125.6, 126.1, 127.7, 127.9, 128.0, 128.5, 128.6, 129.8, 134.7, 136.1, 137.3, 149.3, 149.7, 157.0, 172.2; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 443,1036, found: 443.1040.

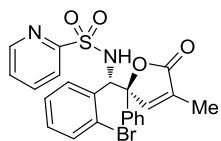


***N*-(*(S)*-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methyl)pyridine-2-sulfonamide (3b)**. White solid; 27.3 mg, 62% yield; 66:34 dr, 94:6 er; $[\alpha]_D^{20} = -61.2$ (c. 1.37, CHCl_3); m.p. 176.9-177.3 °C;

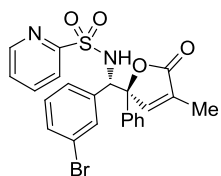
HPLC (AD-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 20.6 min (minor), 28.4 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.59 (s, 3H), 5.45 (d, J = 9.7 Hz, 1H), 5.82 (d, J = 9.8 Hz, 1H), 6.79-6.90 (m, 2H), 6.97 (m, 1H), 7.05-7.15 (m, 1H), 7.18-7.25 (m, 2H), 7.28-7.40 (m, 5H), 7.54-7.63 (m, 2H), 8.34 (d, J = 4.7 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.2, 57.6, 89.6, 114.7 (d, J = 21.7 Hz, 1C), 121.4, 122.4 (d, J = 12.7 Hz, 1C), 124.0 (d, J = 3.7 Hz, 1C), 125.3, 126.2, 128.6, 128.7, 128.8, 129.9 (d, J = 9.0 Hz, 1C), 136.0, 137.4, 148.7 (d, J = 2.2 Hz, 1C), 149.8, 156.8, 157.8, 159.4 (d, J = 244.5 Hz, 1C), 172.0; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{FN}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 461.0942, found: 461.0950.



***N*-((*S*)-(2-chlorophenyl))((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methylpyridine-2-sulfonamide (3c).** White solid; 24.5 mg, 54% yield; 60:40 dr, 94:6 er; $[\alpha]_D^{20}$ = -88.0 (c. 1.23, CHCl_3); m.p. 194.6-195.2 °C; HPLC (IA-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 18.2 min (minor), 19.7 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.52 (s, 3H), 5.75 (d, J = 9.4 Hz, 1H), 5.83 (d, J = 9.3 Hz, 1H), 6.95-7.11 (m, 3H), 7.15-7.25 (m, 3H), 7.30-7.40 (m, 3H), 7.41-7.49 (m, 2H), 7.51-7.64 (m, 2H), 8.33 (d, J = 4.6 Hz, 1H). ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.1, 59.8, 89.7, 121.4, 125.1, 126.1, 126.9, 128.6, 128.9, 129.0, 129.3, 129.6, 132.9, 136.2, 137.3, 148.6, 149.8, 156.6, 172.1; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{ClN}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 477.0646, found: 477.0651.

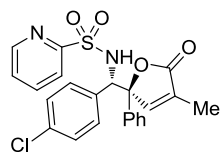


***N*-((*S*)-(2-bromophenyl))((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methylpyridine-2-sulfonamide (3d).** White solid; 30.2 mg, 66% yield; 57:43 dr, 94:6 er; $[\alpha]_D^{20}$ = -51.0 (c. 1.51, CHCl_3); m.p. 177.6-178.4 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 24.6 min (major), 28.0 min (minor); ^1H NMR (300 MHz, Chloroform-*d*) δ (major + minor diastereoisomer) 1.52 (s, 3H), 1.66 (s, 1H), 5.53-5.66 (m, 0.5H), 5.74 (d, J = 9.3 Hz, 1H), 5.86 (d, J = 9.0 Hz, 1H), 6.77-7.23 (m, 6.5H), 7.27-7.42 (m, 5.2H), 7.46 (d, J = 6.6 Hz, 2H), 7.51-7.65 (m, 2.6H), 7.69 (td, J = 7.8, 1.7 Hz, 0.3H), 7.87 (d, J = 8.2 Hz, 0.3H), 8.25 (d, J = 4.1 Hz, 0.2H), 8.33 (d, J = 4.2 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major + minor diastereoisomer) 10.1, 10.6, 60.3, 62.5, 89.8, 90.2, 121.5, 121.8, 123.8, 125.1, 125.8, 126.1, 126.3, 127.5, 128.1, 128.3, 128.6, 128.8, 129.0, 129.4, 129.5, 129.6, 132.3, 134.5, 136.3, 137.3, 137.5, 148.7, 149.5, 149.8, 150.5, 156.6, 172.1, 172.2; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{BrN}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 521.0141, found: 521.0155.



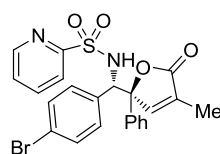
***N*-((*S*)-(3-bromophenyl))((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methylpyridine-2-sulfonamide (3e).** White solid; 36.9 mg, 74% yield; 86:14 dr, 91:9 er; $[\alpha]_D^{20}$ = -39.5 (c. 1.85, CHCl_3); m.p. 181.7-182.2 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 17.9 min (minor), 21.5 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.69 (s, 3H), 4.95 (d, J = 9.7 Hz, 1H), 5.70 (d, J = 9.6 Hz, 1H), 6.83 (d, J = 7.8 Hz, 1H), 6.88-7.00 (m, 2H), 7.10-7.29 (m, 4H), 7.29-7.44 (m, 5H), 7.49-7.67 (m, 2H), 8.42 (d, J = 4.5 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.4, 64.0, 89.0, 121.5, 121.9, 125.7, 126.4, 128.8, 128.9, 129.5, 130.2, 131.0,

131.2, 135.6, 137.0, 137.4, 149.0, 149.9, 156.8, 171.9; HRMS (ESI) calcd. for $C_{23}H_{19}BrN_2NaO_4S$ $[M + Na]^+$ 521.0141, found: 521.0150.



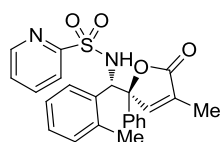
***N*-((*S*)-(4-chlorophenyl))((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methylpyridine-2-sulfonamide (3f).** White solid; 41.1 mg, 90% yield; 87:13 dr, 94:6 er; $[\alpha]_D^{20} = -49.6$ (c. 2.06, $CHCl_3$); m.p. 177.3-178.1 °C;

HPLC (AD-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_R = 27.1$ min (minor), 43.2 min (major); 1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.66 (s, 3H), 5.02 (d, $J = 9.6$ Hz, 1H), 5.97 (d, $J = 9.6$ Hz, 1H), 6.83 (d, $J = 8.5$ Hz, 2H), 7.00 (d, $J = 8.4$ Hz, 2H), 7.20-7.35 (m, 7H), 7.51-7.69 (m, 2H), 8.38 (d, $J = 4.6$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.4, 63.8, 89.2, 121.5, 125.6, 126.2, 128.0, 128.6, 128.7, 129.2, 130.1, 133.6, 133.9, 135.8, 137.5, 149.0, 149.8, 156.9, 172.0; HRMS (ESI) calcd. for $C_{23}H_{19}ClN_2NaO_4S$ $[M + Na]^+$ 477.0646, found: 477.0645.



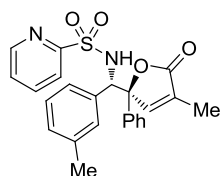
***N*-((*S*)-(4-bromophenyl))((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methylpyridine-2-sulfonamide (3g).** White solid; 39.9 mg, 80% yield; 86:14 dr, 94:6 er; $[\alpha]_D^{20} = -36.6$ (c. 1.99, $CHCl_3$); m.p. 113.4-114.2 °C;

HPLC (AD-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_R = 31.5$ min (minor), 51.0 min (major); 1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.66 (s, 3H), 5.01 (d, $J = 9.2$ Hz, 1H), 5.93 (d, $J = 9.3$ Hz, 1H), 6.76 (d, $J = 8.4$ Hz, 2H), 7.15 (d, $J = 8.3$ Hz, 2H), 7.20-7.25 (m, 1H), 7.25-7.36 (m, 6H), 7.51-7.64 (m, 2H), 8.38 (d, $J = 4.7$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.4, 63.9, 89.1, 121.5, 122.1, 125.6, 126.2, 128.6, 128.7, 130.1, 131.0, 134.1, 135.7, 137.5, 149.0, 149.8, 156.9, 172.0; HRMS (ESI) calcd. for $C_{23}H_{19}BrN_2NaO_4S$ $[M + Na]^+$ 521.0141, found: 521.0162.



***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(*o*-tolyl)methylpyridine-2-sulfonamide (3h).** White solid; 32.0 mg, 74% yield; 87:13 dr, 93:7 er; $[\alpha]_D^{20} = -62.7$ (c. 1.60, $CHCl_3$); m.p. 178.8-179.7 °C; HPLC (IA-H, *i*-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major

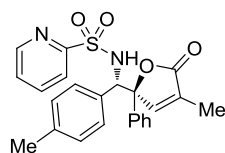
diastereoisomer: $t_R = 24.1$ min (minor), 27.3 min (major); 1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.67 (s, 3H), 2.20 (s, 3H), 5.31 (d, $J = 9.8$ Hz, 1H), 5.78 (d, $J = 9.8$ Hz, 1H), 6.65 (d, $J = 7.9$ Hz, 1H), 6.75-6.85 (m, 1H), 6.86-6.98 (m, 2H), 7.13-7.23 (m, 1H), 7.25-7.29 (m, 6H), 7.47-7.58 (m, 2H), 8.34 (dt, $J = 4.7, 1.3$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.4, 19.7, 59.5, 89.6, 121.1, 125.7, 125.8, 126.1, 126.7, 127.9, 128.6, 130.0, 130.3, 133.2, 135.6, 136.0, 137.3, 148.4, 149.7, 156.9, 172.1; HRMS (ESI) calcd. for $C_{24}H_{22}N_2NaO_4S$ $[M + Na]^+$ 457.1192, found: 457.1203.



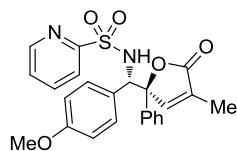
***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(*m*-tolyl)methylpyridine-2-sulfonamide (3i).** White solid; 38.6 mg, 89% yield; 83:17 dr, 91:9 er; $[\alpha]_D^{20} = -59.8$ (c. 1.93, $CHCl_3$); m.p. 177.3-177.8 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major

diastereoisomer: $t_R = 18.0$ min (minor), 21.5 min (major); 1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.62 (s, 3H), 2.10 (s, 3H), 4.96 (d, $J = 9.1$ Hz, 1H), 5.79 (d, $J = 9.1$ Hz, 1H), 6.54-6.72 (m, 2H), 6.79-6.97 (m, 2H), 7.15-7.24 (m, 2H), 7.27-7.37 (m,

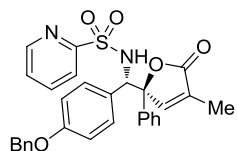
5H), 7.49-7.60 (m, 2H), 8.38 (d, $J = 4.6$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform- d) δ (major diastereoisomer) 10.3, 21.1, 64.4, 89.4, 121.5, 124.8, 125.6, 126.1, 127.8, 128.3, 128.5, 128.6, 128.7, 129.7, 134.5, 136.2, 137.2, 137.5, 149.3, 149.6, 157.0, 172.2; HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 457.1192, found: 457.1193.



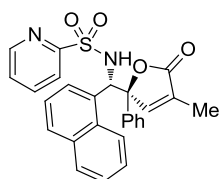
***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(*p*-tolyl)methyl)pyridine-2-sulfonamide (3j).** White solid; 35.9 mg, 83% yield; 87:13 dr, 93:7 er; $[\alpha]_{\text{D}}^{20} = -55.7$ (c. 1.79, CHCl_3); m.p. 162.4-163.2 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_{\text{R}} = 30.0$ min (minor), 44.7 min (major); ^1H NMR (300 MHz, Chloroform- d) δ (major diastereoisomer) 1.62 (s, 3H), 2.17 (s, 3H), 4.98 (d, $J = 9.8$ Hz, 1H), 5.84 (d, $J = 9.7$ Hz, 1H), 6.73 (d, $J = 8.2$ Hz, 2H), 6.80 (d, $J = 8.0$ Hz, 2H), 7.15-7.24 (m, 2H), 7.25-7.38 (m, 5H), 7.47-7.58 (m, 2H), 8.37 (dt, $J = 4.7, 1.2$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform- d) δ (major diastereoisomer) 10.3, 20.9, 64.2, 89.5, 121.5, 125.6, 125.9, 127.6, 128.4, 128.5, 128.6, 129.7, 131.7, 136.2, 137.2, 137.6, 149.3, 149.7, 157.0, 172.3; HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 457.1192, found: 457.1181.



***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methyl)pyridine-2-sulfonamide (3k).** White solid; 29.1 mg, 65% yield; 76:24 dr, 91:9 er; $[\alpha]_{\text{D}}^{20} = -48.4$ (c. 1.46, CHCl_3); m.p. 166.9-167.3 °C; HPLC (IA-H, *i*-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_{\text{R}} = 28.8$ min (minor), 34.1 min (major); ^1H NMR (300 MHz, Chloroform- d) δ (major diastereoisomer) 1.64 (s, 3H), 3.7 (s, 3H), 4.97 (d, $J = 9.5$ Hz, 1H), 5.74 (d, $J = 9.3$ Hz, 1H), 6.53 (d, $J = 8.7$ Hz, 2H), 6.76 (d, $J = 8.6$ Hz, 2H), 7.20-7.33 (m, 7H), 7.50-7.59 (m, 2H), 8.39 (d, $J = 4.4$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform- d) δ (major diastereoisomer) 10.3, 55.1, 64.0, 89.6, 113.3, 121.5, 125.7, 126.1, 126.9, 128.5, 128.7, 128.9, 129.7, 136.2, 137.3, 149.4, 149.8, 157.1, 159.1, 172.3; HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{NaO}_5\text{S}$ $[\text{M} + \text{Na}]^+$ 473.1142, found: 473.1135.

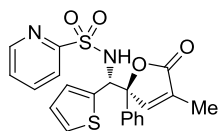


***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methyl)pyridine-2-sulfonamide (3l).** White solid; 33.2 mg, 63% yield; 88:12 dr, 95:5 dr; $[\alpha]_{\text{D}}^{20} = -24.3$ (c. 1.66, CHCl_3); m.p. 154.2-155.0 °C; HPLC (IB-H, *i*-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_{\text{R}} = 18.4$ min (major), 23.1 min (minor); ^1H NMR (300 MHz, Chloroform- d) δ (major diastereoisomer) 1.64 (s, 3H), 4.87-5.02 (m, 3H), 5.72 (d, $J = 9.7$ Hz, 1H), 6.60 (d, $J = 8.7$ Hz, 2H), 6.74 (d, $J = 8.7$ Hz, 2H), 7.08-7.15 (m, 1H), 7.20 (d, $J = 1.5$ Hz, 1H), 7.28-7.43 (m, 10H), 7.44-7.53 (m, 2H), 8.32 (dt, $J = 4.6, 1.1$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform- d) δ (major diastereoisomer) 10.3, 29.7, 64.0, 69.7, 89.6, 114.3, 121.5, 125.7, 126.0, 127.1, 127.3, 128.0, 128.5, 128.7, 128.9, 129.7, 136.1, 136.7, 137.3, 149.4, 149.7, 157.0, 158.1, 172.3; HRMS (ESI) calcd. for $\text{C}_{30}\text{H}_{26}\text{N}_2\text{NaO}_5\text{S}$ $[\text{M} + \text{Na}]^+$ 549.1455, found: 549.1472.

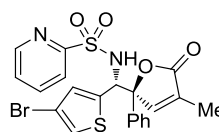


***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(naphthalen-1-yl)methyl)pyridine-2-sulfonamide (3m).** White solid; 41.5 mg, 88% yield; 88:12 dr, 80:20 er; $[\alpha]_{\text{D}}^{20} = -74.8$ (c. 2.07, CHCl_3); m.p. 143.5-144.6 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0

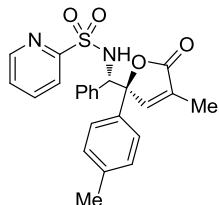
mL/min, λ = 254 nm) major diastereoisomer: t_R = 46.0 min (major), 50.7 min (minor); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.40 (s, 3H), 6.02 (s, 2H), 6.94-7.01 (m, 1H), 7.04 (d, J = 7.2 Hz, 1H), 7.08-7.16 (m, 2H), 7.20 (td, J = 7.7, 1.5 Hz, 1H), 7.27-7.42 (m, 4H), 7.42-7.51 (m, 3H), 7.51-7.62 (m, 2H), 7.73 (d, J = 8.0 Hz, 1H), 8.01 (d, J = 8.6 Hz, 1H), 8.15 (d, J = 4.2 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.1, 58.3, 89.8, 121.3, 121.6, 124.7, 125.3, 125.6, 125.8, 126.7, 128.6, 128.7, 128.9, 129.4, 130.7, 130.8, 133.1, 136.4, 136.8, 148.8, 149.4, 156.6, 172.2; HRMS (ESI) calcd. for $\text{C}_{27}\text{H}_{22}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 493.1192, found: 493.1198.



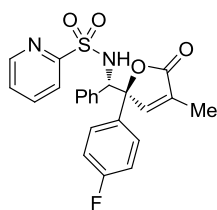
***N*-((*R*)-((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(thiophen-2-yl)methyl)pyridine-2-sulfonamide (3n).** White solid; 25.2 mg, 59% yield; 45:55 dr, 90:10 er; $[\alpha]_D^{20}$ (minor diastereoisomer) = -55.9 (c. 1.26, CHCl_3); m.p. (minor diastereoisomer) 204.2-205.1 $^\circ\text{C}$; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm) minor diastereoisomer: t_R = 29.8 min (minor), 37.0 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (minor diastereoisomer) 1.69 (s, 3H), 5.34 (d, J = 9.8 Hz, 1H), 5.66 (d, J = 9.8 Hz, 1H), 6.60 (d, J = 3.5 Hz, 1H), 6.64 (dd, J = 4.9, 3.7 Hz, 1H), 7.00 (dd, J = 5.0, 1.0 Hz, 1H), 7.20 (d, J = 1.4 Hz, 1H), 7.23-7.29 (m, 1H), 7.29-7.43 (m, 5H), 7.56-7.69 (m, 2H), 8.42 (d, J = 3.8 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (minor diastereoisomer) 10.4, 60.1, 89.4, 121.4, 125.6, 125.8, 126.2, 126.3, 127.0, 128.6, 128.7, 130.1, 136.0, 137.4, 137.6, 148.8, 149.8, 157.0, 172.3; HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{18}\text{N}_2\text{NaO}_4\text{S}_2$ $[\text{M} + \text{Na}]^+$ 449.0600, found: 449.0588.



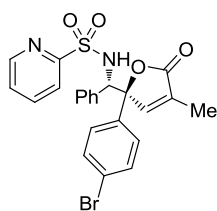
***N*-((*R*)-(4-bromothiophen-2-yl)((*S*)-4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)methyl)pyridine-2-sulfonamide (3o).** White solid; 21.6 mg, 43% yield; 50:50 dr, 80:20 er; $[\alpha]_D^{20}$ = -71.1 (c. 1.23, CHCl_3); m.p. 173.9-174.2 $^\circ\text{C}$; HPLC (IB-H, *i*-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 12.6 min (major), 16.2 min (minor); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.74 (s, 3H), 5.28 (d, J = 9.6 Hz, 1H), 5.83 (d, J = 9.6 Hz, 1H), 6.49 (s, 1H), 6.91 (d, J = 1.3 Hz, 1H), 7.21-7.25 (m, 1H), 7.28-7.42 (m, 6H), 7.61-7.73 (m, 2H), 8.45 (d, J = 5.1 Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.5, 59.9, 89.0, 109.0, 121.5, 123.2, 125.6, 126.5, 128.8, 128.9, 129.9, 130.5, 135.6, 137.5, 139.2, 148.5, 149.8, 156.8, 172.0; HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{17}\text{BrN}_2\text{NaO}_4\text{S}_2$ $[\text{M} + \text{Na}]^+$ 526.9705, found: 526.9725.



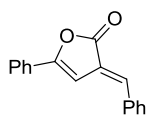
***N*-((*S*)-((*S*)-4-methyl-5-oxo-2-(*p*-tolyl)-2,5-dihydrofuran-2-yl)(phenyl)methyl)pyridine-2-sulfonamide (3p).** White solid; 34.2 mg, 79% yield; 90:10 dr, 94:6 er; $[\alpha]_D^{20}$ = -79.6 (c. 1.71, CHCl_3); m.p. 185.2-185.8 $^\circ\text{C}$; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, λ = 254 nm) major diastereoisomer: t_R = 22.6 min (minor), 26.4 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.59 (s, 3H), 2.32 (s, 3H), 5.01 (s, 1H), 5.90 (s, 1H), 6.80-6.91 (m, 2H), 6.94-7.07 (m, 3H), 7.07-7.14 (m, 2H), 7.15-7.25 (m, 4H), 7.46-7.58 (m, 2H), 8.29-8.47 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.2, 21.0, 64.4, 89.4, 121.5, 125.5, 126.0, 127.7, 127.9, 128.0, 129.3, 129.5, 129.9, 133.0, 134.8, 137.2, 138.4, 149.5, 149.7, 157.0, 172.3; HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 457.1192, found: 457.1183.



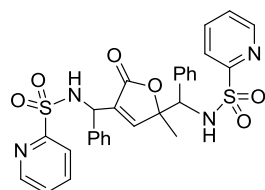
N-((S)-((S)-2-(4-fluorophenyl)-4-methyl-5-oxo-2,5-dihydrofuran-2-yl)(phenyl)methyl)pyridine-2-sulfonamide (3q). White solid; 37.5 mg, 86% yield; 83:17 dr, 93:7 er; $[\alpha]_D^{20} = -42.5$ (c. 1.87, CHCl_3); m.p. 180.8-181.2 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_R = 18.3$ min (minor), 23.3 min (major); ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ (major + minor diastereoisomer) 1.45 (s, 3.0H), 1.66 (s, 0.6H), 4.98 (d, $J = 10.1$ Hz, 0.2H), 5.13 (d, $J = 10.4$ Hz, 1.0H), 6.96-7.09 (m, 5.1H), 7.09-7.21 (m, 3.8H), 7.30-7.36 (m, 4.3 Hz, 1.5H), 7.37-7.43 (m, 0.3H), 7.46-7.56 (m, 3.0H), 7.57-7.63 (m, 0.3H), 7.67-7.77 (m, 2.1H), 7.78-7.83 (m, 0.2H), 7.84-7.89 (m, 0.2H), 8.27 (d, $J = 4.5$ Hz, 1H), 8.38 (d, $J = 4.3$ Hz, 0.2H), 8.83 (d, $J = 10.3$ Hz, 1H), 8.96 (d, $J = 10.1$ Hz, 0.2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$) δ (major diastereoisomer) 9.9, 63.7, 89.7, 115.9 (d, $J = 21.4$ Hz, 1C), 126.1, 127.2, 127.3, 128.1, 128.2, 128.3, 128.7, 133.9 (d, $J = 2.9$ Hz, 1C), 136.1, 149.2, 150.8, 157.4, 161.6 (d, $J = 242.7$ Hz, 1C), 172.3; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{FN}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 461.0942, found: 461.0935.



N-((S)-((S)-2-(4-bromophenyl)-4-methyl-5-oxo-2,5-dihydrofuran-2-yl)(phenyl)methyl)pyridine-2-sulfonamide (3r). White solid; 34.2 mg, 69% yield; 80:20 dr, 90:10 er; $[\alpha]_D^{20} = -75.2$ (c. 1.71, CHCl_3); m.p. 115.6-116.2 °C; HPLC (AD-H, *i*-propanol/*n*-hexane = 25/75, flow rate = 1.0 mL/min, $\lambda = 254$ nm) major diastereoisomer: $t_R = 21.5$ min (minor), 27.9 min (major); ^1H NMR (300 MHz, Chloroform-*d*) δ (major diastereoisomer) 1.60 (s, 3H), 5.01 (d, $J = 9.9$ Hz, 1H), 5.92 (d, $J = 9.9$ Hz, 1H), 6.86-6.97 (m, 2H), 7.02-7.12 (m, 3H), 7.12-7.18 (m, 1H), 7.18-7.31 (m, 3H), 7.35-7.45 (m, 2H), 7.51-7.64 (m, 2H), 8.37 (d, $J = 4.5$ Hz, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ (major diastereoisomer) 10.2, 64.4, 89.1, 121.4, 122.7, 126.2, 127.3, 127.6, 128.1, 128.2, 130.1, 131.7, 134.7, 135.4, 137.4, 148.7, 149.8, 156.9, 171.9; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{BrN}_2\text{NaO}_4\text{S}$ $[\text{M} + \text{Na}]^+$ 521.0141, found: 521.0130.

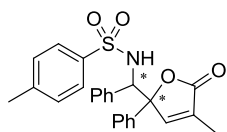


(E)-3-benzylidene-5-phenylfuran-2(3H)-one (5). Yellow solid; m.p. 156.8-157.6 °C; ^1H NMR (300 MHz, Chloroform-*d*) δ 6.94 (s, 1H), 7.36-7.54 (m, 7H), 7.60-7.68 (m, 2H), 7.73-7.81 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 99.8, 125.3, 125.4, 128.0, 128.8, 129.1, 130.1, 130.2, 130.5, 135.1, 135.4, 156.9, 169.3; HRMS (ESI) calcd. for $\text{C}_{17}\text{H}_{12}\text{NaO}_2$ $[\text{M} + \text{Na}]^+$ 271.0730, found: 271.0729.



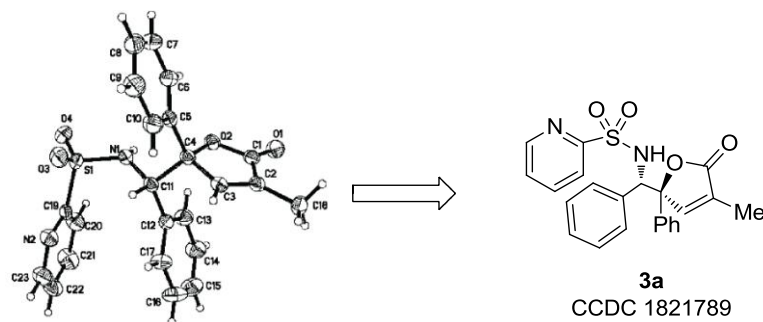
N,N'-((5-methyl-2-oxo-2,3-dihydrofuran-3,3-diyl)bis(phenylmethylene))bis(pyridine-2-sulfonamide) (6). White solid; m.p. 169.2-169.8 °C; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ (major + minor diastereoisomer) 1.26 (s, 3H), 1.31 (s, 0.6H), 4.80 (d, $J = 9.9$ Hz, 1H), 4.85 (d, $J = 10.0$ Hz, 0.2H), 5.03 (d, $J = 8.3$ Hz, 0.2H), 5.15 (d, $J = 8.0$ Hz, 1H), 6.22 (d, $J = 7.6$ Hz, 0.4H), 6.70-6.81 (m, 2H), 6.87-6.96 (m, 0.4H), 6.96-7.10 (m, 7.2H), 7.10-7.18 (m, 2H), 7.31-7.40 (m, 1.2H), 7.47-7.50 (m, 0.2H), 7.50-7.56 (m, 2H), 7.57-7.62 (m, 0.2H), 7.65-7.72 (m, 1.2H), 7.73-7.86 (m, 2.4H), 7.87-7.95 (m, 1H), 7.95-8.04 (m, 0.2H), 8.30 (d, $J = 4.6$ Hz, 0.2H), 8.34 (d, $J = 4.4$ Hz, 1H), 8.57 (d, $J = 4.4$ Hz, 1H), 8.62 (d, $J = 4.5$ Hz, 0.2H), 8.65-8.80 (m, 2H), 8.81-8.95 (m, 0.4H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$) δ (major + minor diastereoisomer) 21.4, 22.1, 53.2, 53.3, 61.6, 62.1, 87.5, 88.3, 121.5, 126.5, 126.7, 126.8, 127.0, 127.1, 127.2, 127.3, 127.4, 127.6, 127.9, 128.0, 128.1, 132.9, 136.2, 136.4, 137.7, 137.8, 137.9, 138.0, 138.4, 138.6, 149.3, 149.5,

149.8, 150.0, 153.7, 154.3, 157.3, 157.4, 157.7, 157.9, 169.6, 170.2; HRMS (ESI) calcd. for $C_{29}H_{26}N_4NaO_6S_2$ $[M + Na]^+$ 613.1186, found: 613.1184.



4-methyl-N-((4-methyl-5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)(phenyl)methyl)benzenesulfonamide (6). White solid; 39.1 mg, 92% yield; 87:13 dr, 86.5:13.5 er; $[\alpha]_D^{20} = -52.1$ (c. 1.92, $CHCl_3$); m.p. 185.7-186.4 °C; 1H NMR (300 MHz, $DMSO-d_6$) δ (major diastereoisomer) 1.42 (s, 3H), 2.18 (s, 3H), 5.11 (d, $J = 10.5$ Hz, 1H), 6.94 (d, $J = 8.2$ Hz, 2H), 7.00-7.08 (m, 3H), 7.11-7.22 (m, 4H), 7.29-7.38 (m, 3H), 7.46-7.54 (m, 2H), 7.62-7.68 (m, 1H), 8.52 (d, $J = 10.5$ Hz, 1H); ^{13}C NMR (75 MHz, $DMSO-d_6$) δ (major diastereoisomer) 9.9, 20.8, 63.3, 90.3, 125.7, 125.9, 126.2, 127.1, 127.2, 127.9, 128.2, 128.7, 129.3, 135.8, 137.8, 138.3, 141.6, 151.4, 172.5; HRMS (ESI) calcd. for $C_{25}H_{23}NNaO_4S$ $[M + Na]^+$ 456.1240, found: 456.1244.

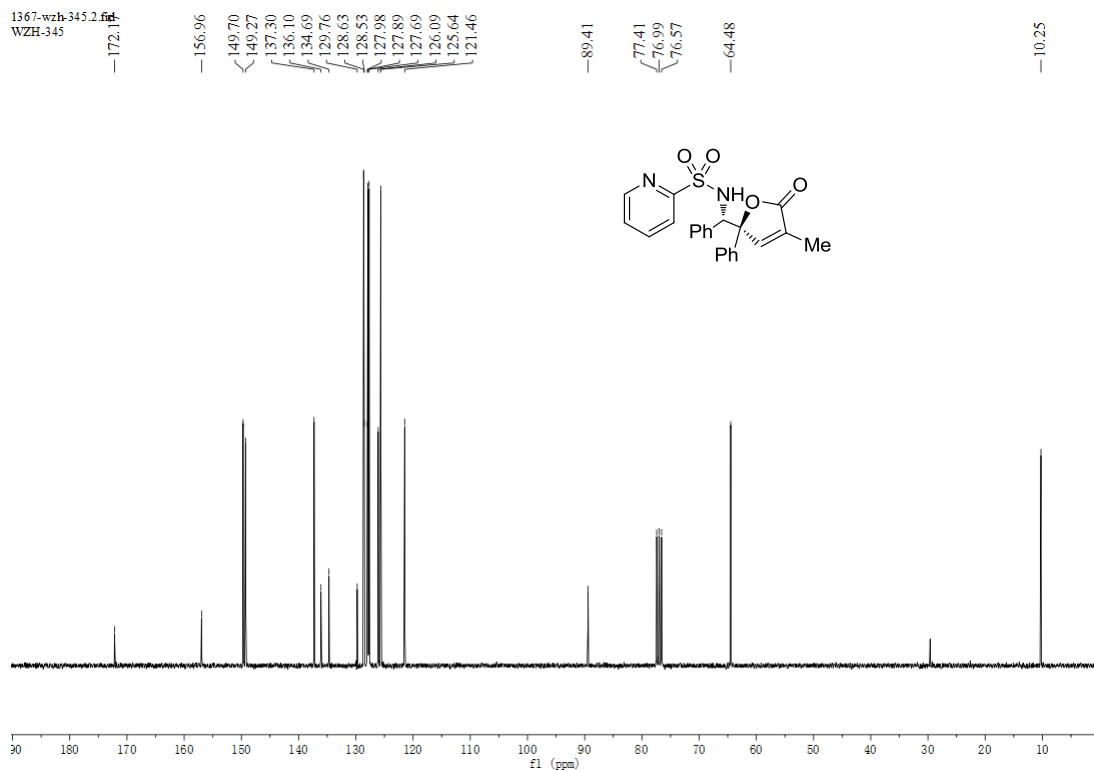
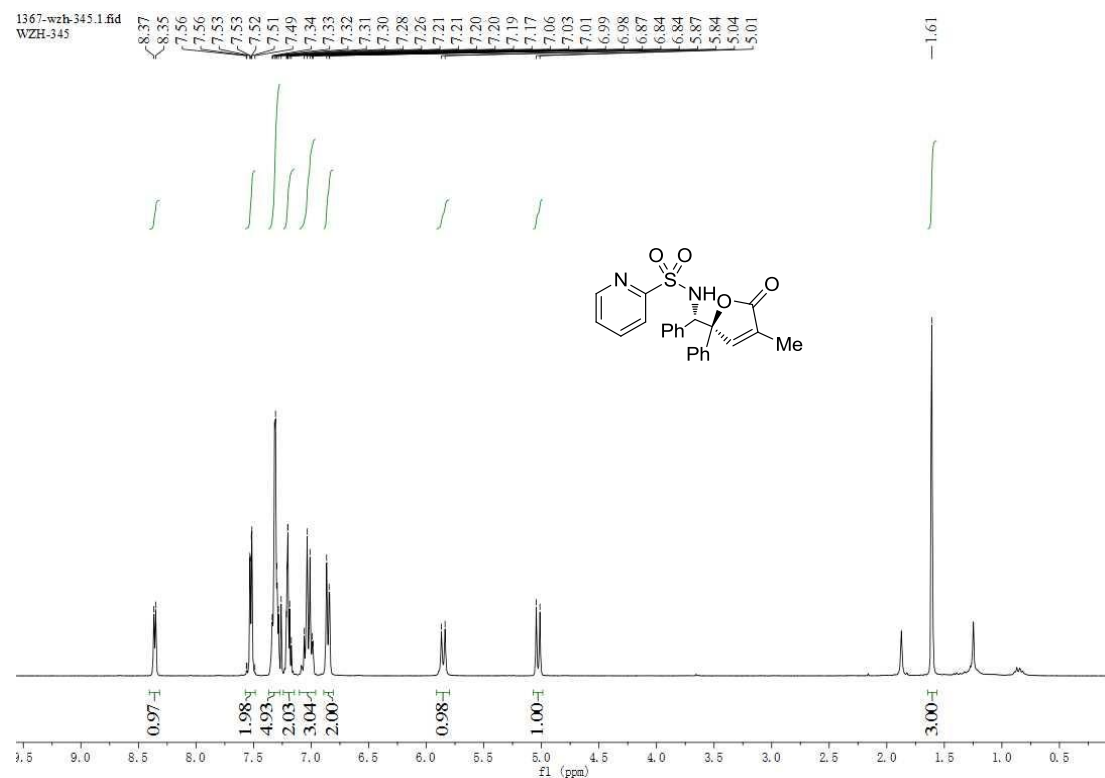
4. Crystal data and structure refinement for 3a.

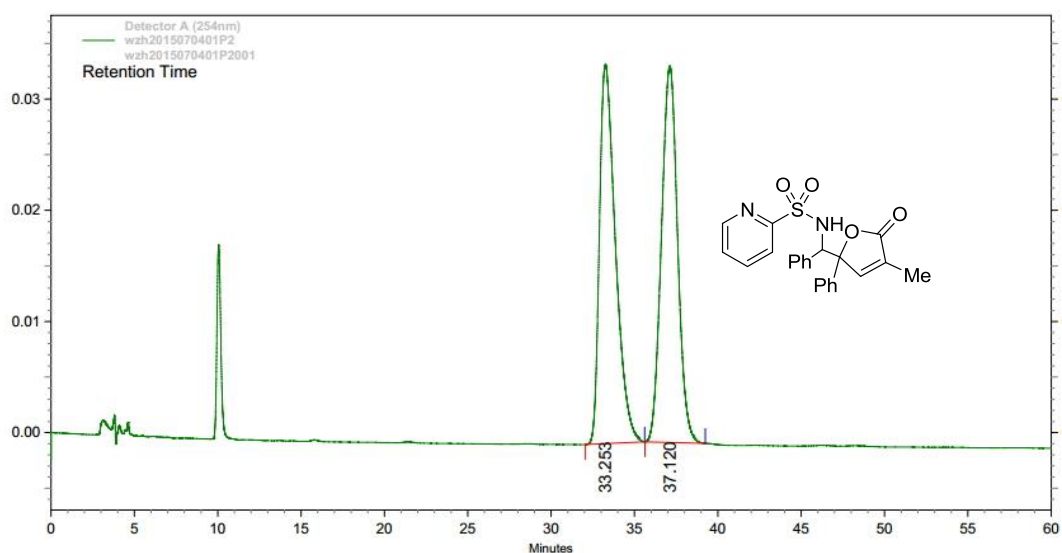


Identification code	3a
Empirical formula	4(C ₂₃ H ₂₀ N ₂ O ₄ S ₁), CH ₃ OH, 2(H ₂ O)
Formula weight	1749.95
Temperature/K	293(2)
Crystal system	triclinic
Space group	P1
a/Å	11.3575(4)
b/Å	12.9479(6)
c/Å	15.7889(5)
α/°	81.909(3)
β/°	88.938(3)
γ/°	72.873(3)
Volume/Å ³	2196.14(15)
Z	1
ρ _{calc} /cm ³	1.323
μ/mm ⁻¹	1.615
F(000)	918.0
Crystal size/mm ³	0.24 × 0.19 × 0.15
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	7.216 to 134.158
Index ranges	-13 ≤ h ≤ 13, -13 ≤ k ≤ 15, -18 ≤ l ≤ 18
Reflections collected	31604
Independent reflections	14303 [R _{int} = 0.0368, R _{sigma} = 0.0432]
Data/restraints/parameters	14303/111/1204
Goodness-of-fit on F ²	1.026
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0458, wR ₂ = 0.1139
Final R indexes [all data]	R ₁ = 0.0528, wR ₂ = 0.1208
Largest diff. peak/hole / e Å ⁻³	0.23/-0.29
Flack parameter	-0.001(8)

5. ^1H , ^{13}C NMR and HPLC spectra for compounds 3a-r, 5, 6 and 8.

^1H NMR, ^{13}C NMR and HPLC of 3a



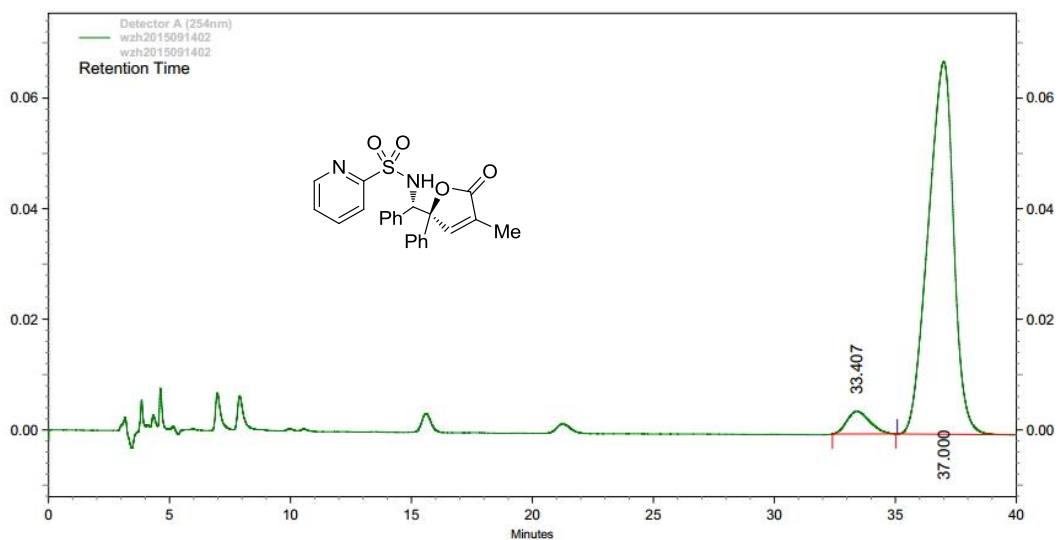


Detector
A

(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	33.253	34076	50.16	2271733	49.97
2	37.120	33864	49.84	2274341	50.03

Totals		67940	100.00	4546074	100.00
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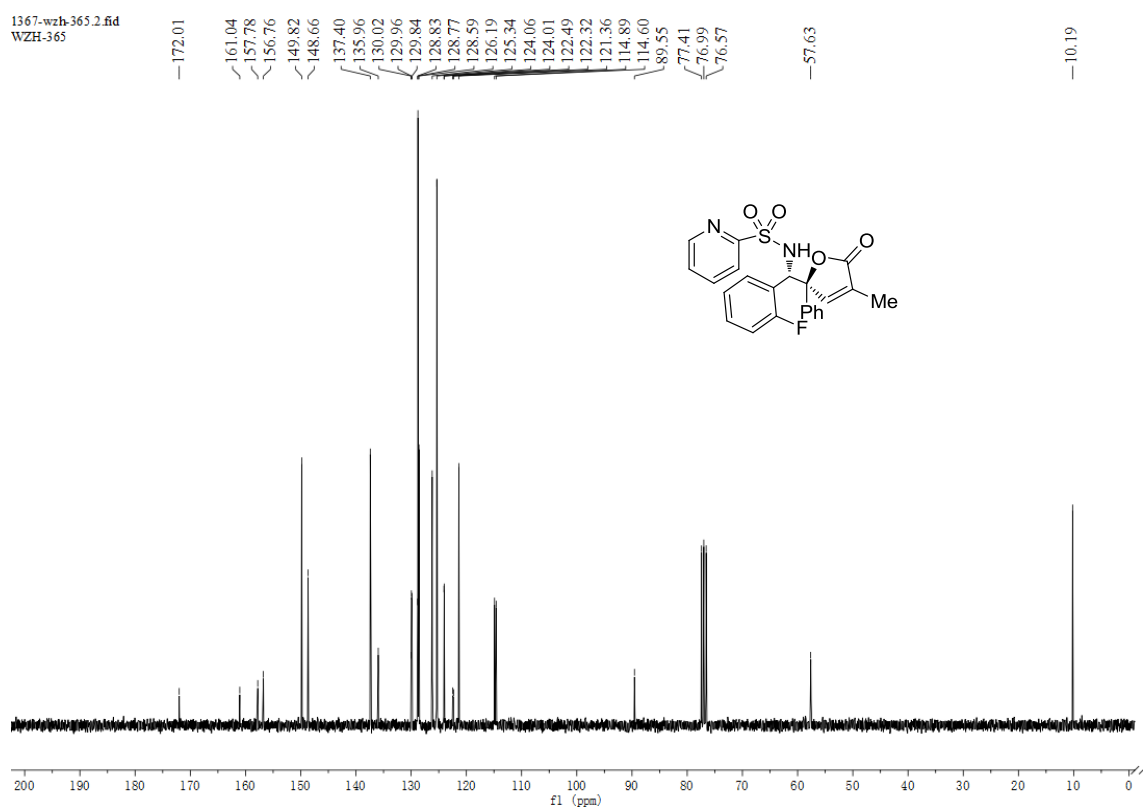
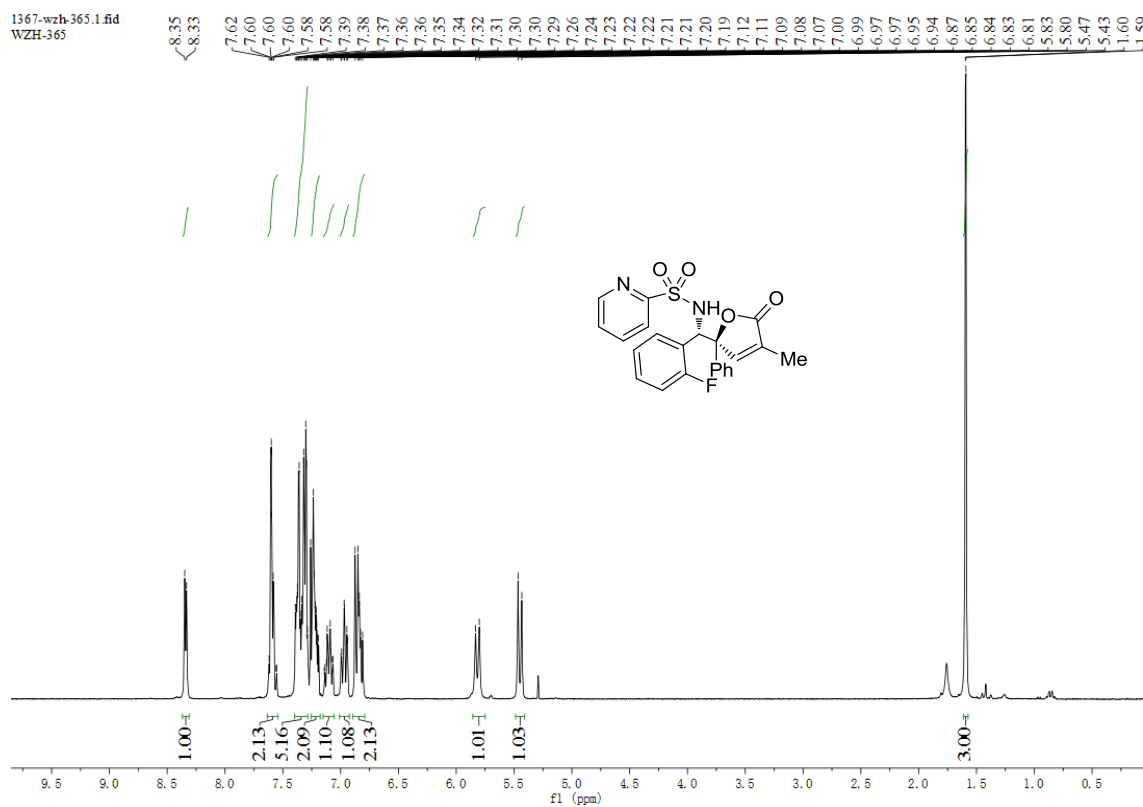
Detector
A

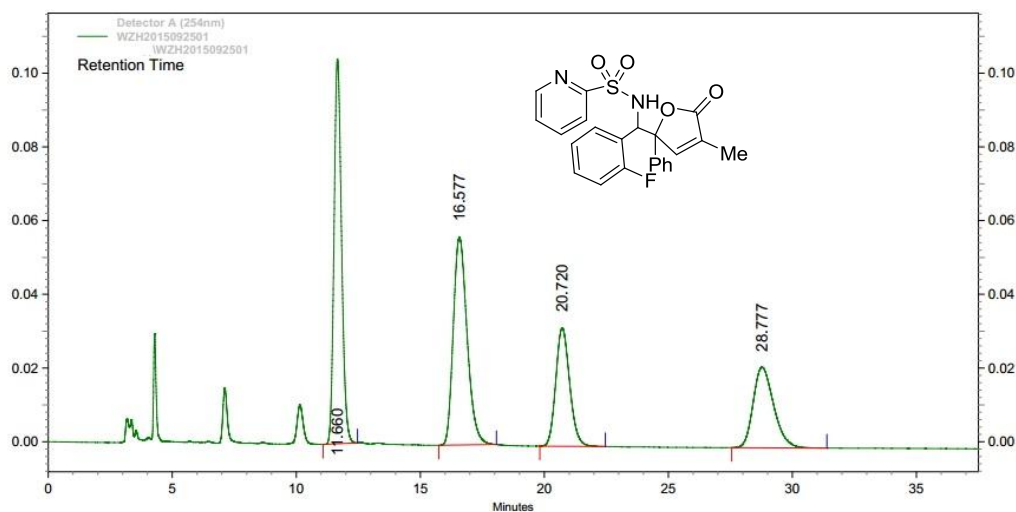
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	33.407	4094	5.73	270566	5.20
2	37.000	67410	94.27	4929369	94.80

Totals		71504	100.00	5199935	100.00
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¹H NMR, ¹³C NMR and HPLC of 3b

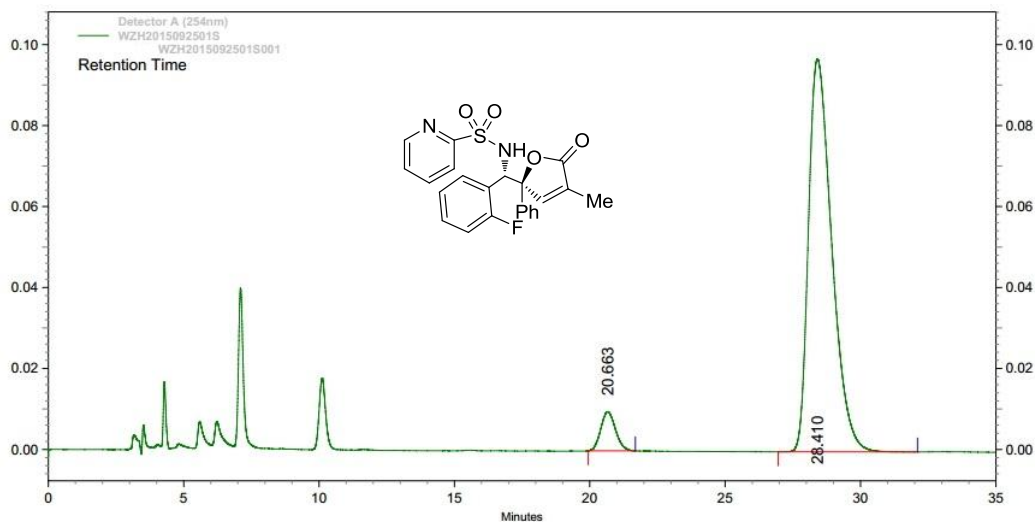




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	11.660	104422	48.58	2194434	31.84
2	16.577	56482	26.28	2189787	31.77
3	20.720	32162	14.96	1253272	18.18
4	28.777	21877	10.18	1255023	18.21

Totals		214943	100.00	6892516	100.00
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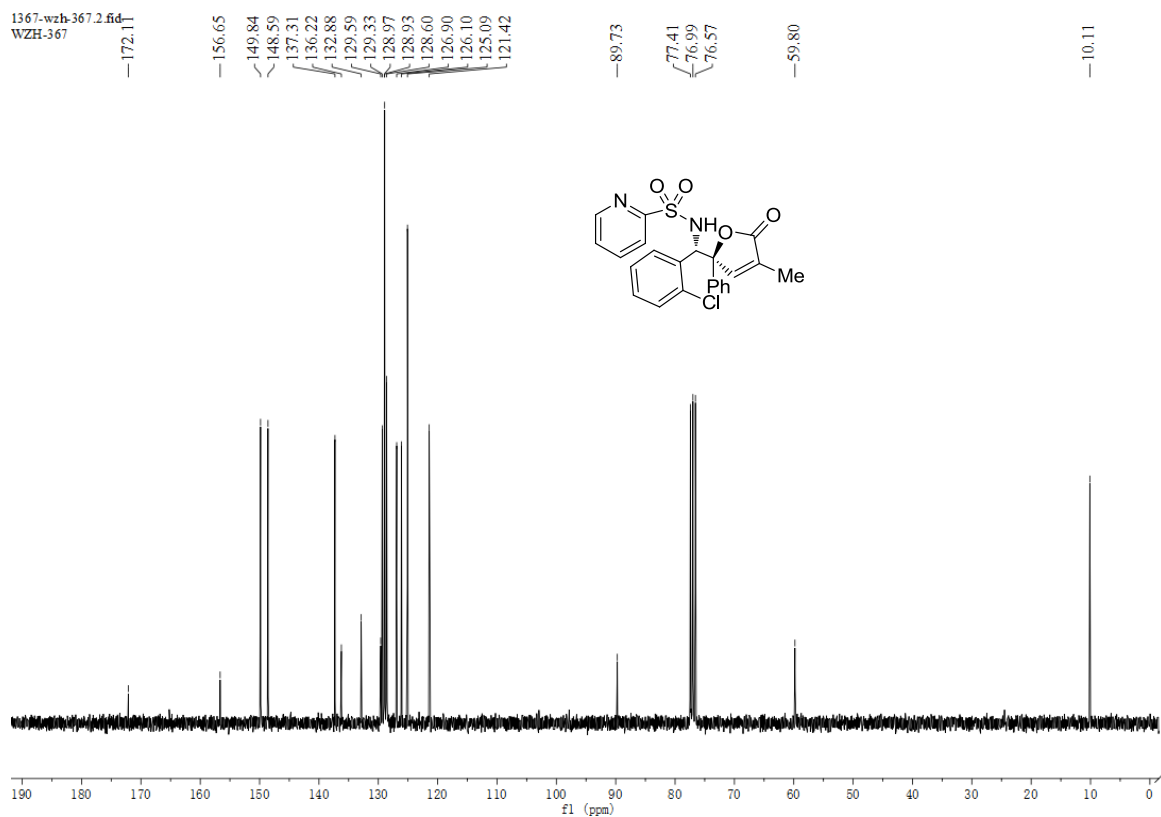
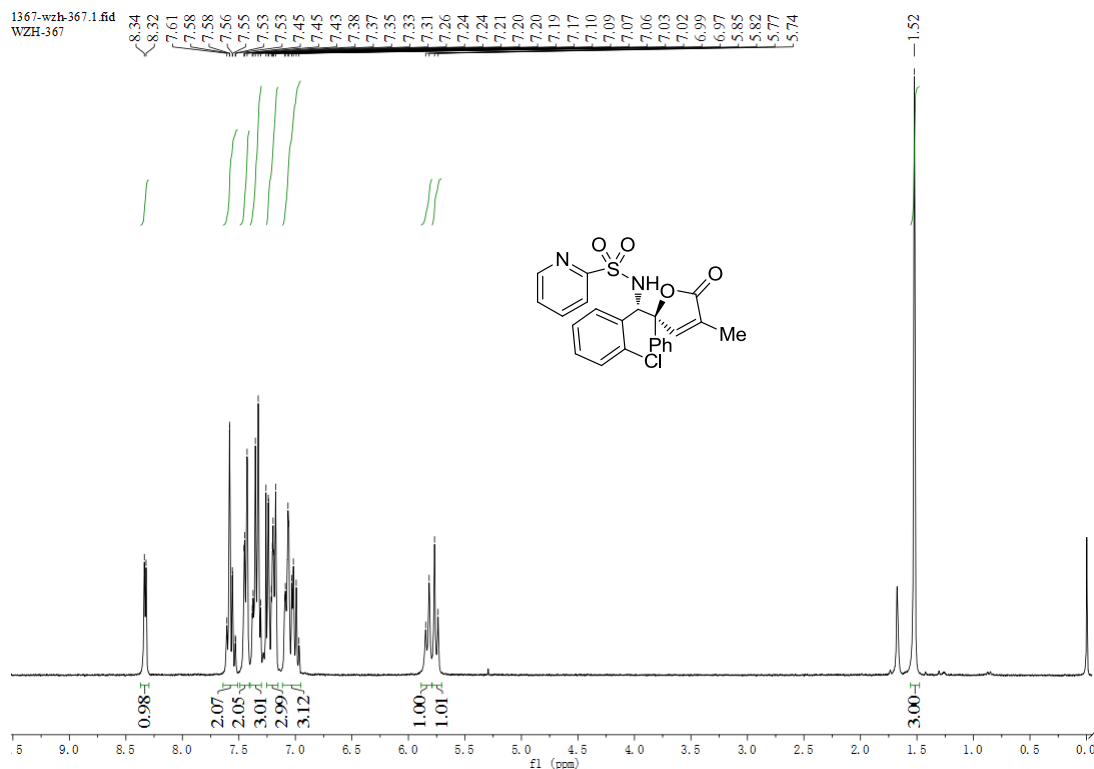


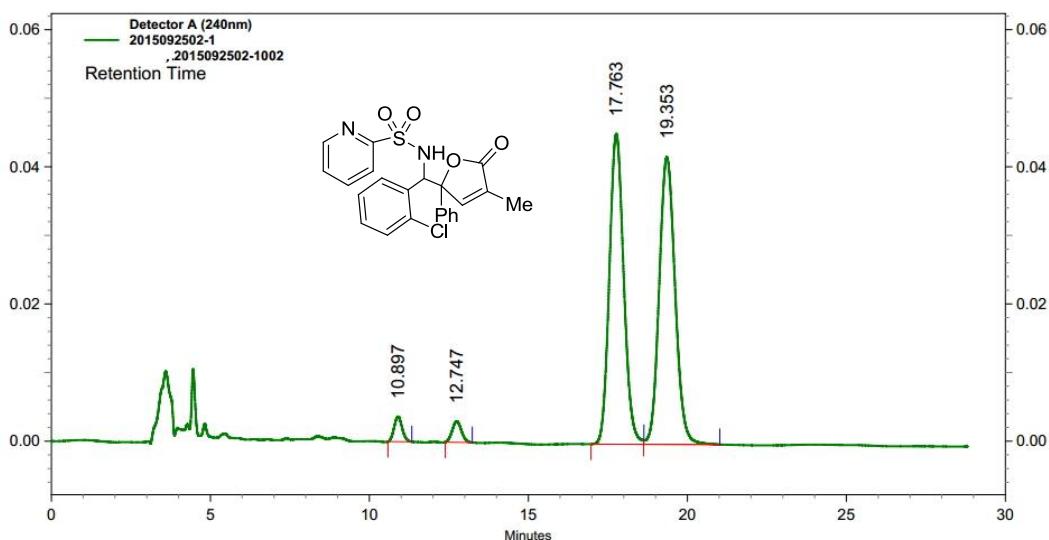
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	20.663	9664	9.05	366611	5.95
2	28.410	97084	90.95	5793879	94.05

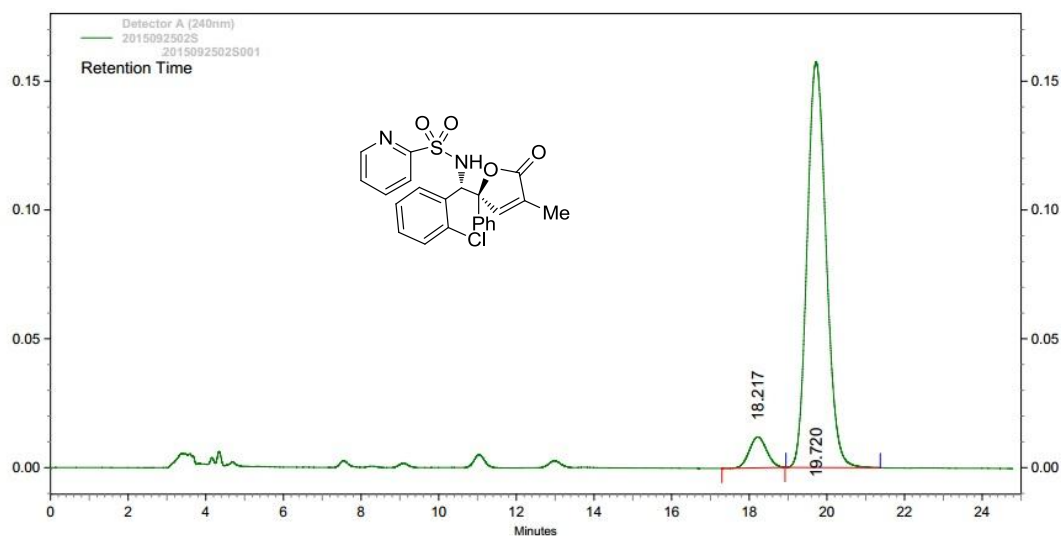
Totals		106748	100.00	6160490	100.00
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¹H NMR, ¹³C NMR and HPLC of 3c



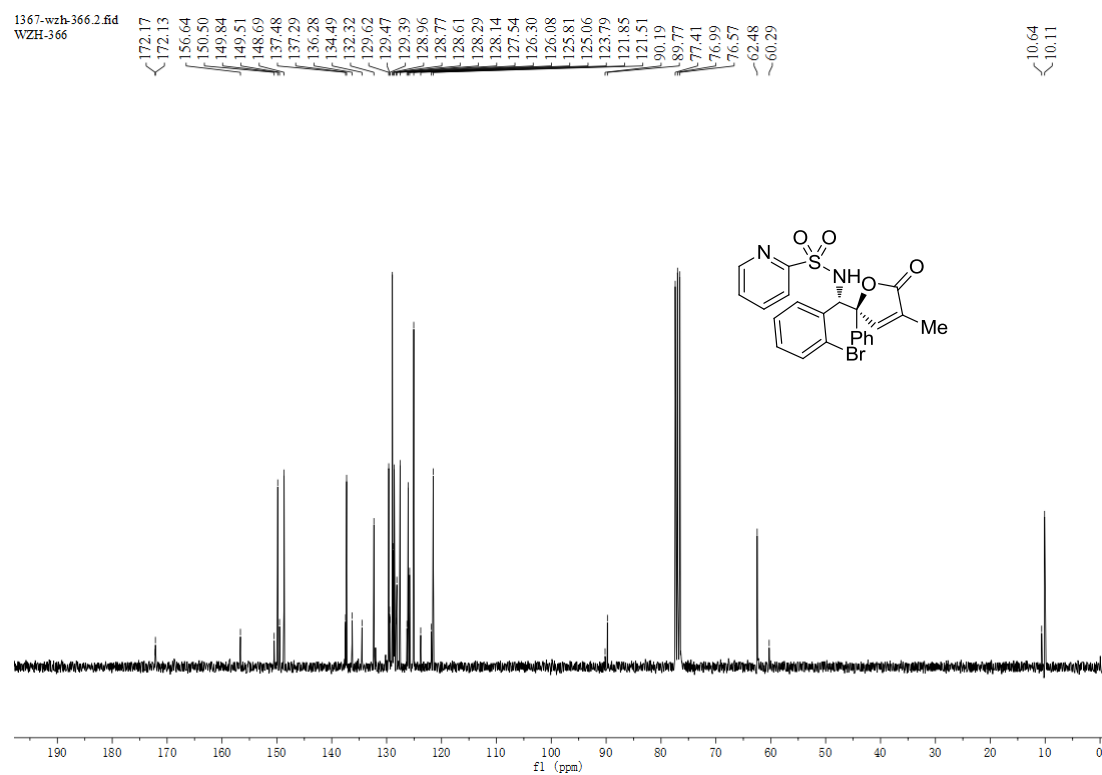
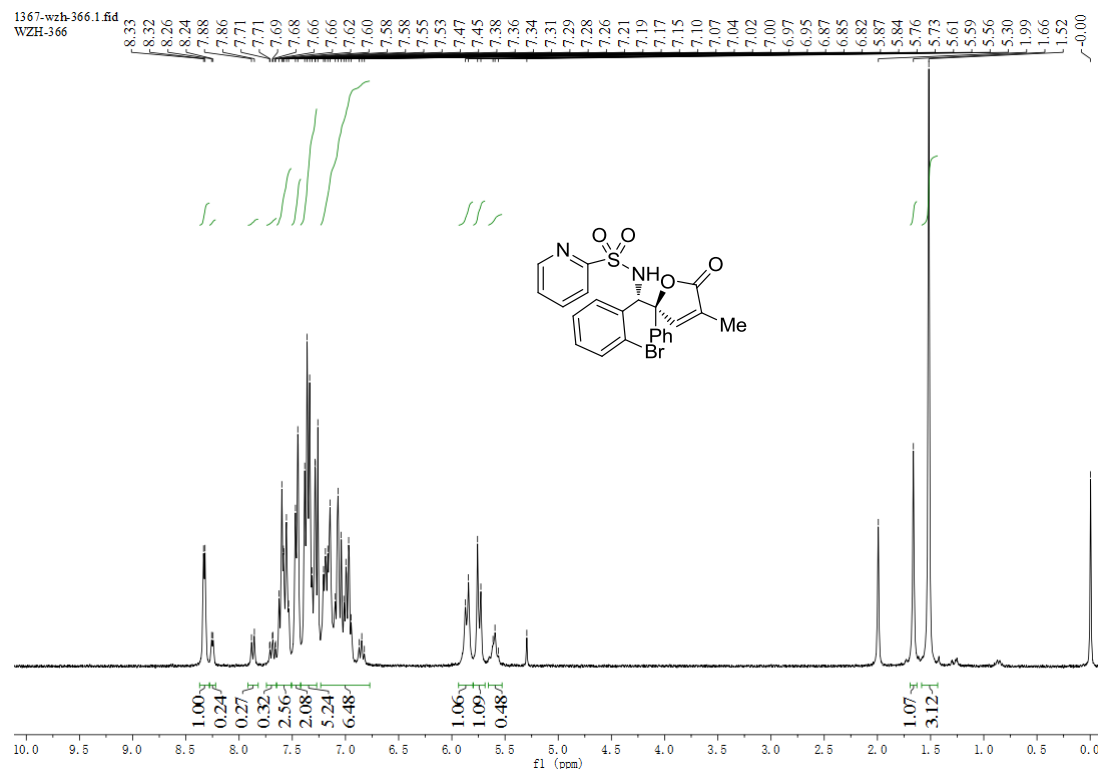


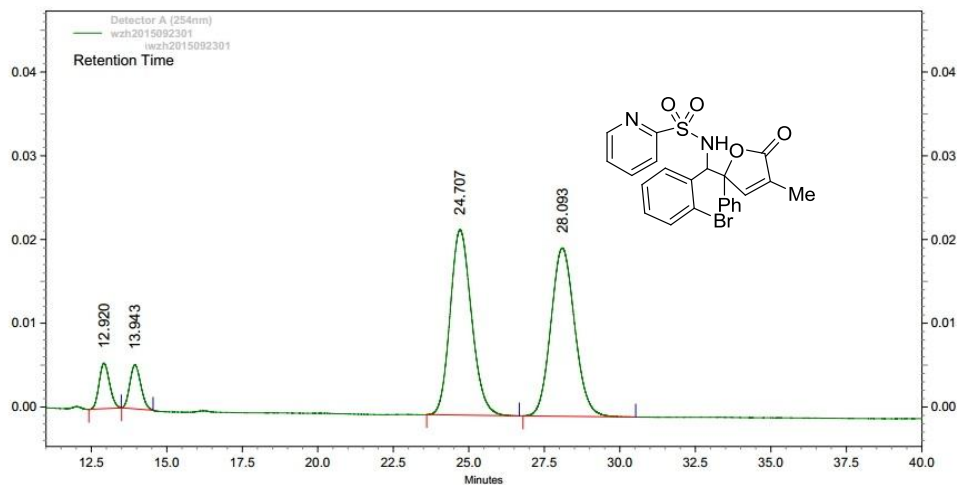
Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	10.897	3657	3.90	64419	2.15
2	12.747	3051	3.25	61837	2.06
3	17.763	45261	48.22	1418487	47.23
4	19.353	41901	44.64	1458454	48.56
Totals		93870	100.00	3003197	100.00



Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	18.217	11983	7.07	373652	6.53
2	19.720	157521	92.93	5351972	93.47
Totals		169504	100.00	5725624	100.00

¹H NMR, ¹³C NMR and HPLC of 3d

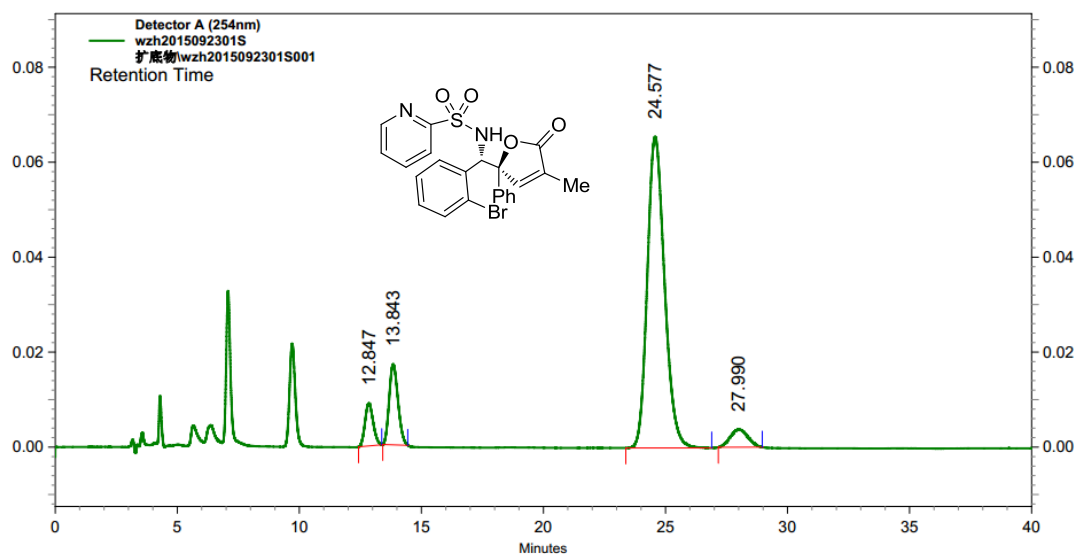




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.920	5446	10.29	131468	5.34
2	13.943	5267	9.95	132848	5.39
3	24.707	22124	41.78	1093680	44.40
4	28.093	20111	37.98	1105410	44.87

Totals		52948	100.00	2463406	100.00
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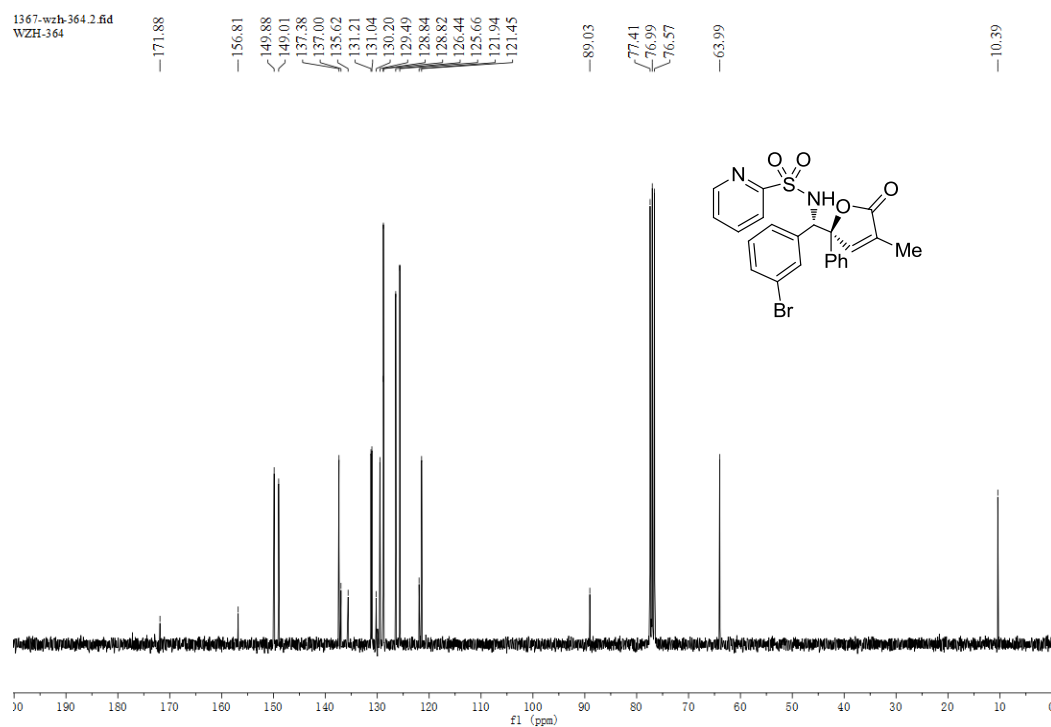
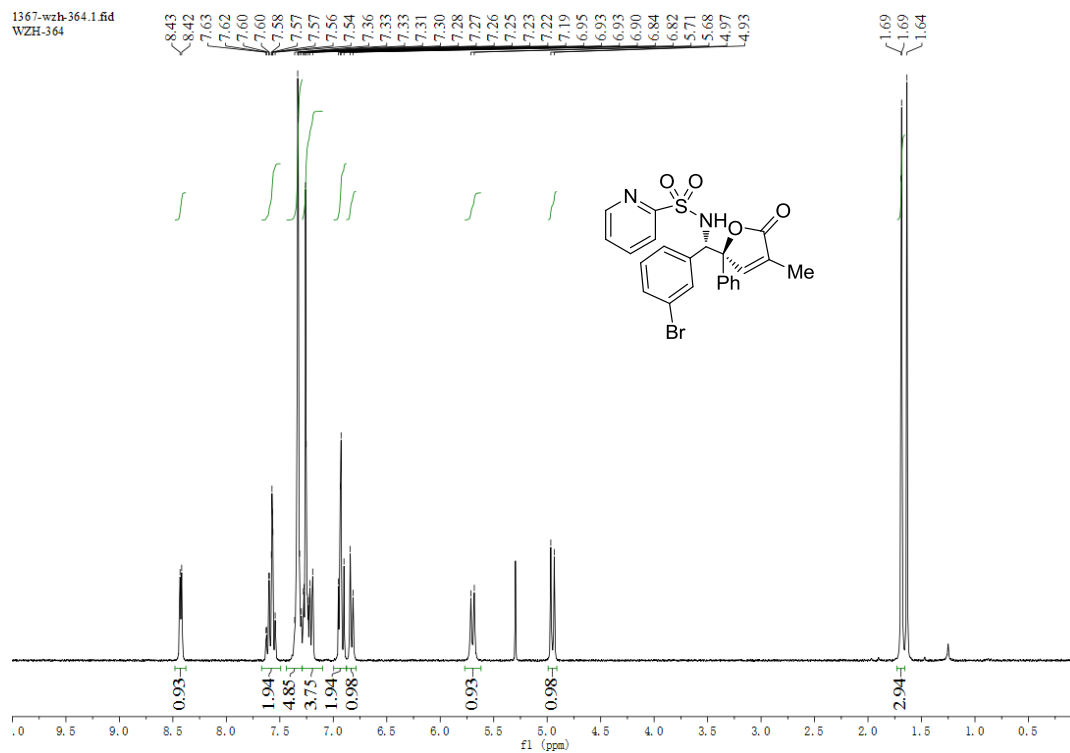


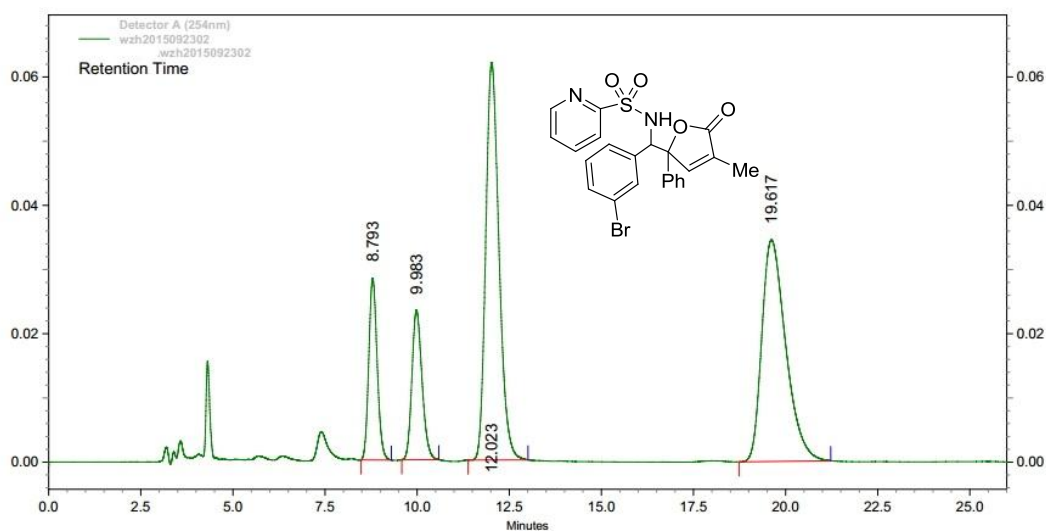
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.847	8966	9.42	208509	5.17
2	13.843	16983	17.84	426844	10.58
3	24.577	65525	68.83	3212856	79.63
4	27.990	3726	3.91	186514	4.62

Totals		95200	100.00	4034723	100.00
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¹H NMR, ¹³C NMR and HPLC of 3e

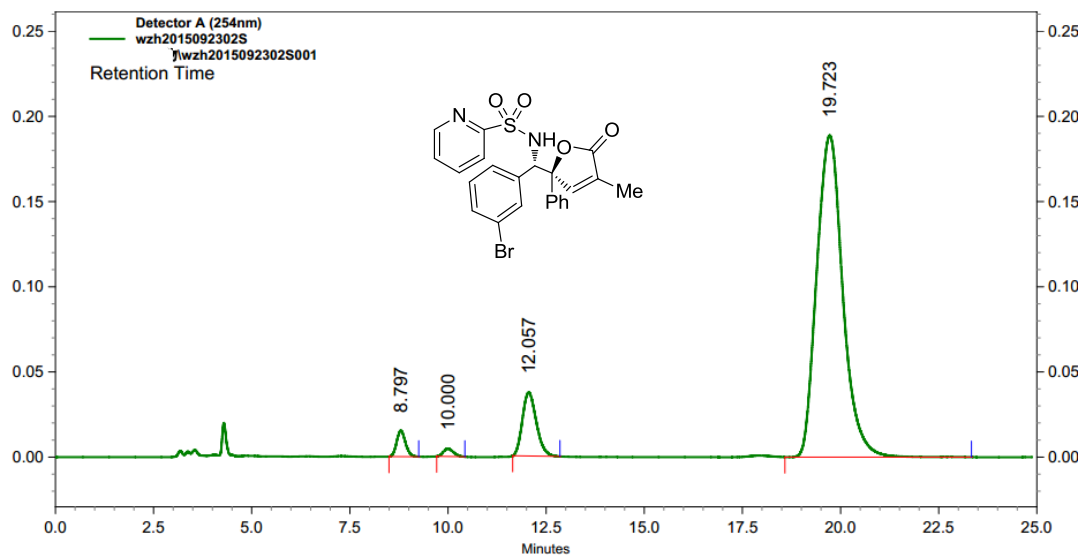




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	8.793	28347	19.14	448887	11.22
2	9.983	23290	15.73	446190	11.15
3	12.023	61938	41.82	1548696	38.69
4	19.617	34526	23.31	1558620	38.94

Totals		148101	100.00	4002393	100.00
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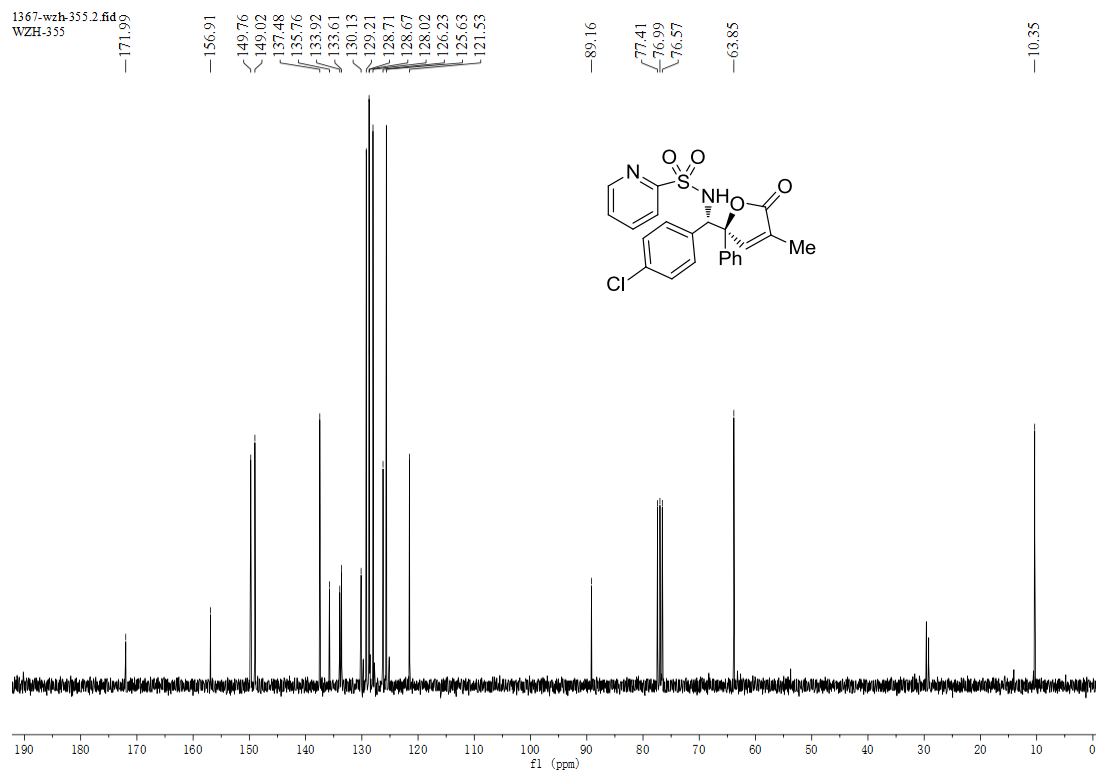
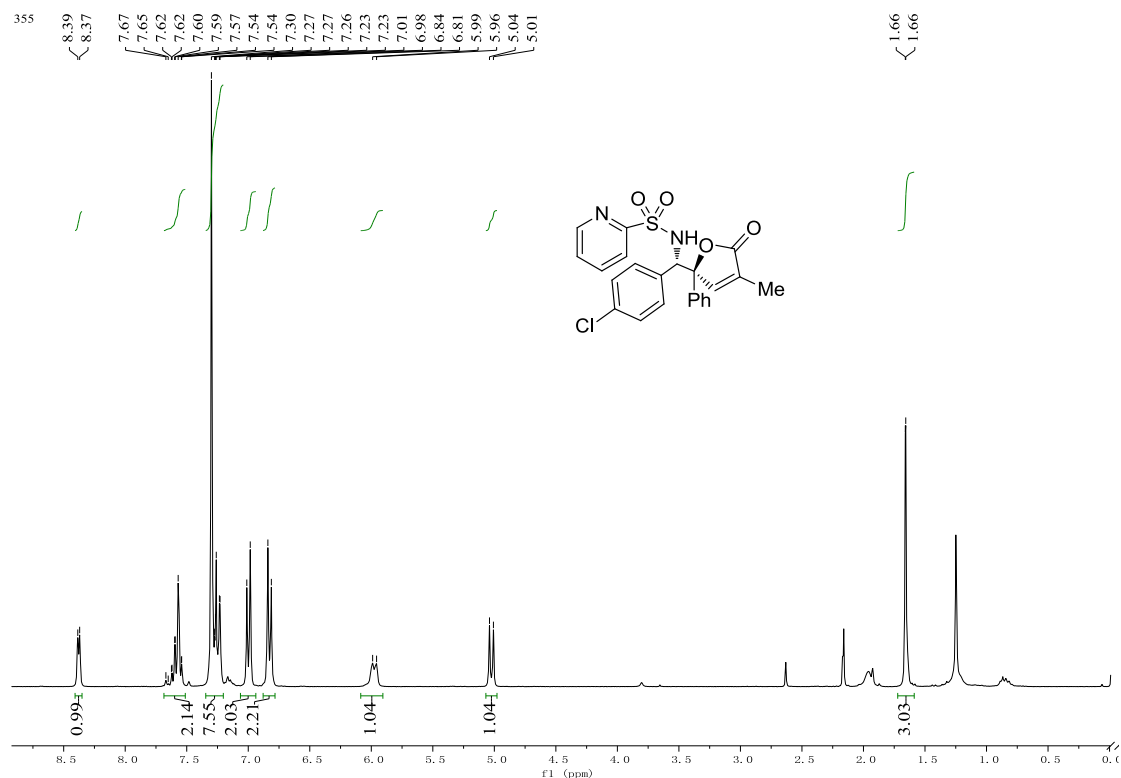


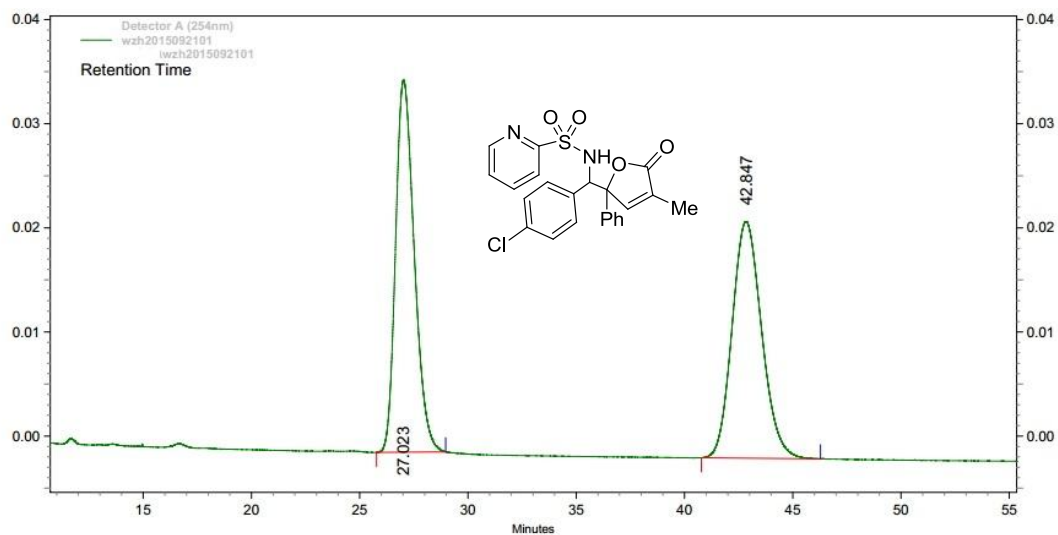
Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	8.797	15285	6.21	241420	2.42
2	10.000	4569	1.86	83473	0.84
3	12.057	37297	15.16	924178	9.27
4	19.723	188845	76.77	8718909	87.47

Totals		245996	100.00	9967980	100.00
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¹H NMR, ¹³C NMR and HPLC of 3f

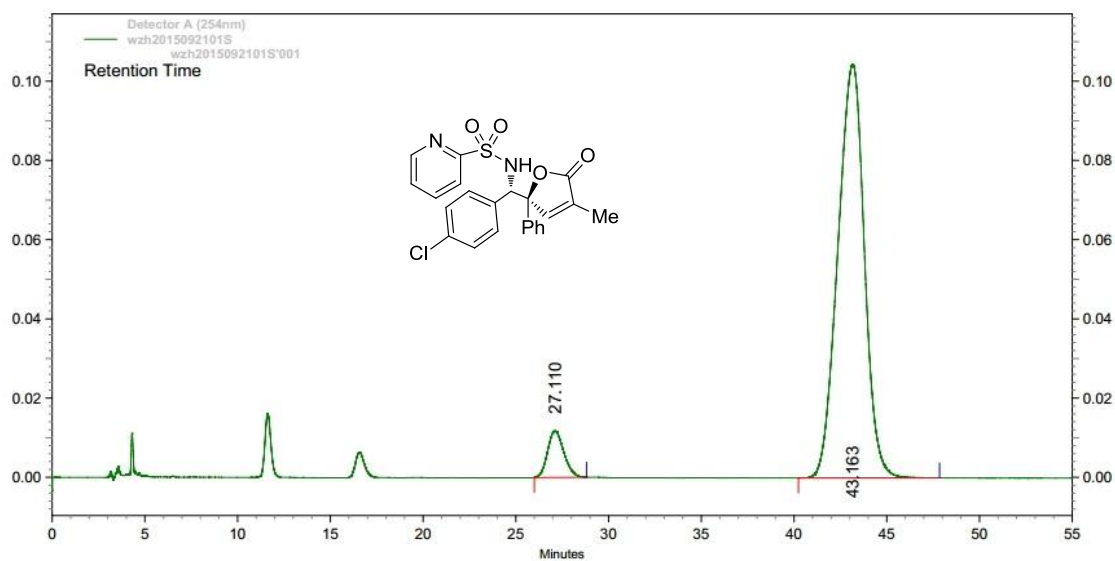




Detector A
(254nm)

PK #	Retention Time	Height	Height Percent	Area	Area Percent
1	27.023	35739	61.13	2142929	49.83
2	42.847	22727	38.87	2157158	50.17

Totals		58466	100.00	4300087	100.00
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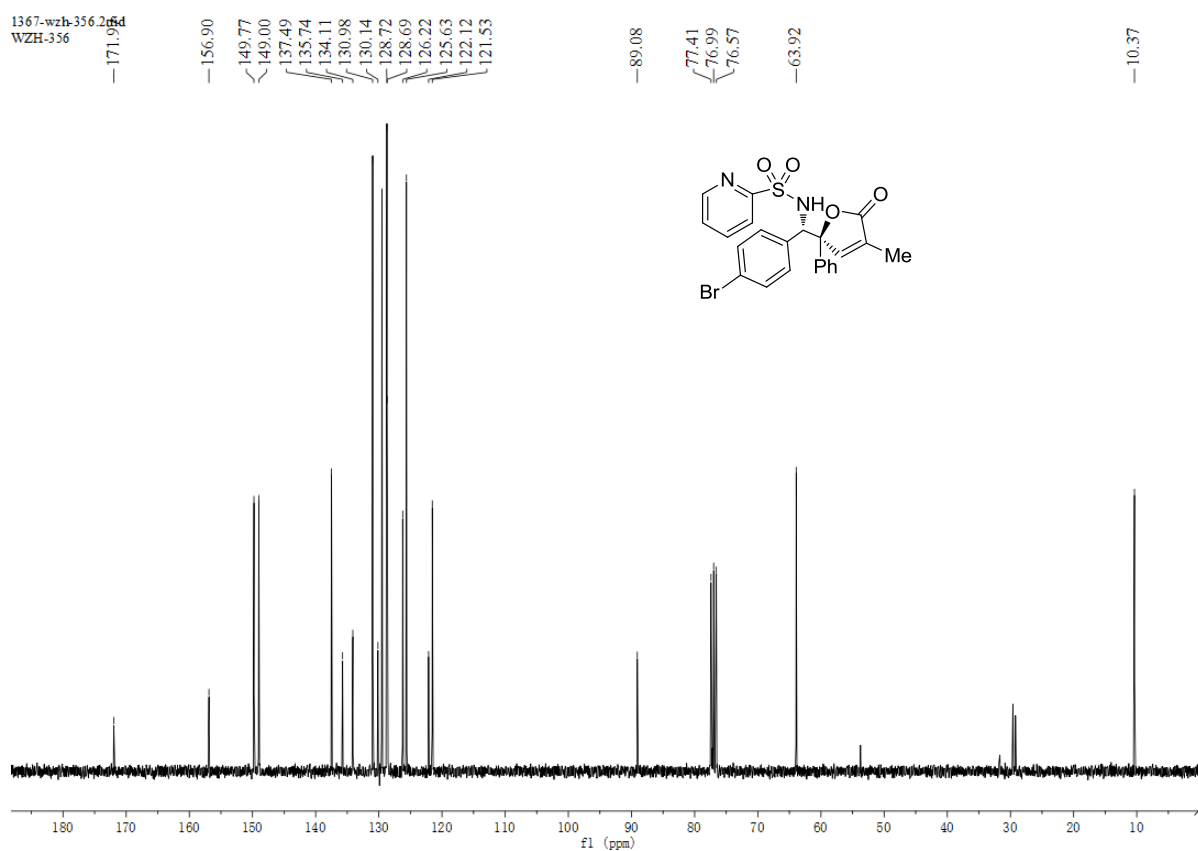
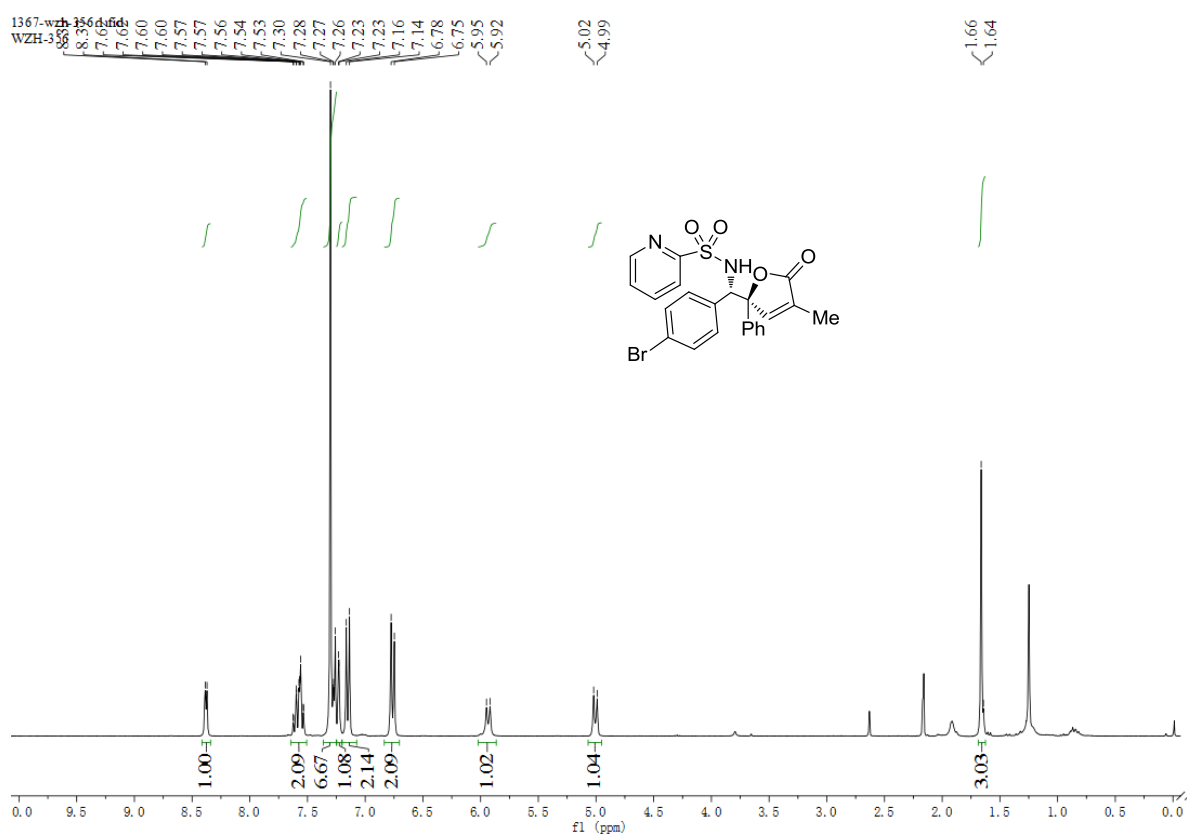


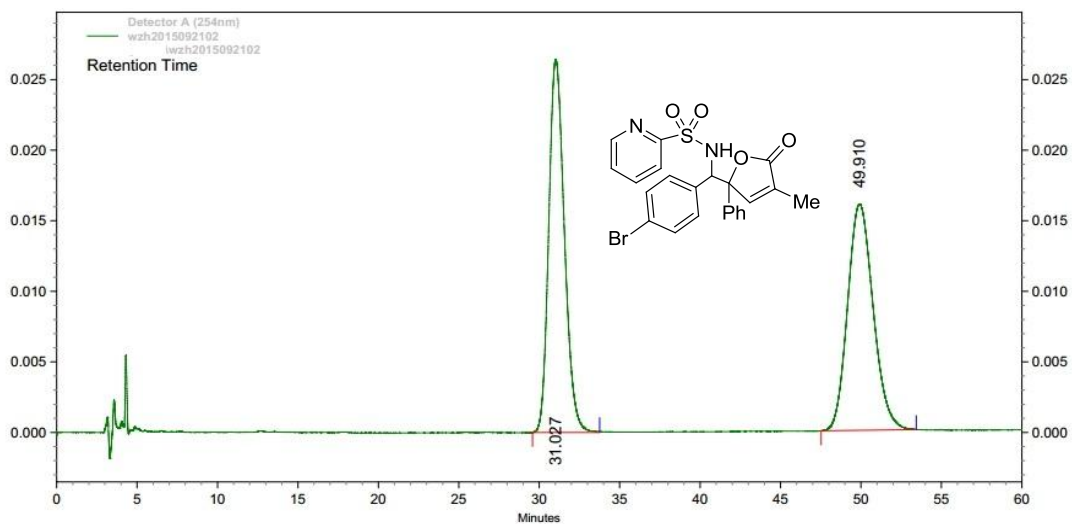
Detector A
(254nm)

PK #	Retention Time	Height	Height Percent	Area	Area Percent
1	27.110	11740	10.10	713549	6.44
2	43.163	104504	89.90	10368030	93.56

Totals		116244	100.00	11081579	100.00
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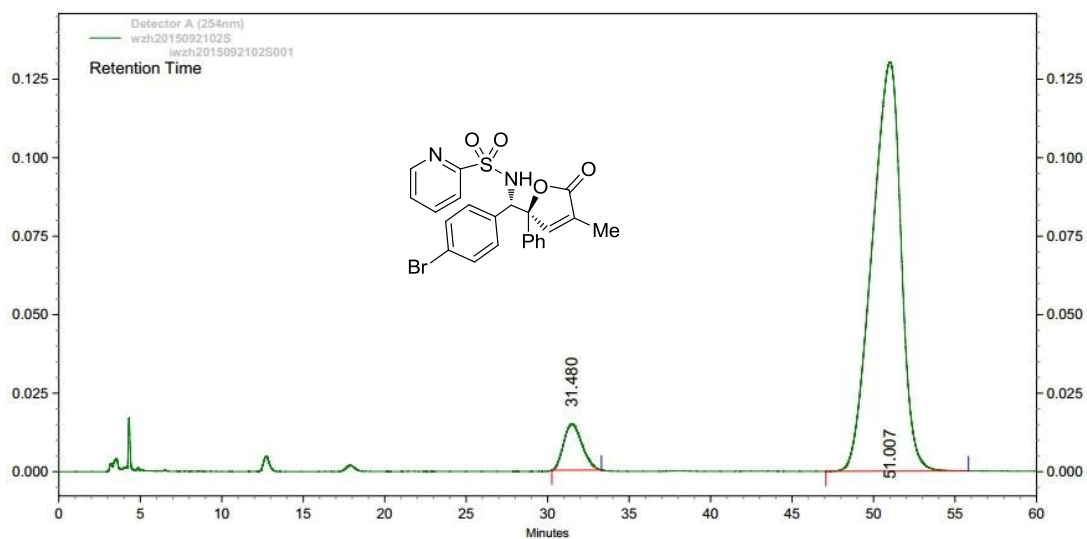
¹H NMR, ¹³C NMR and HPLC of 3g





Detector A
(254nm)

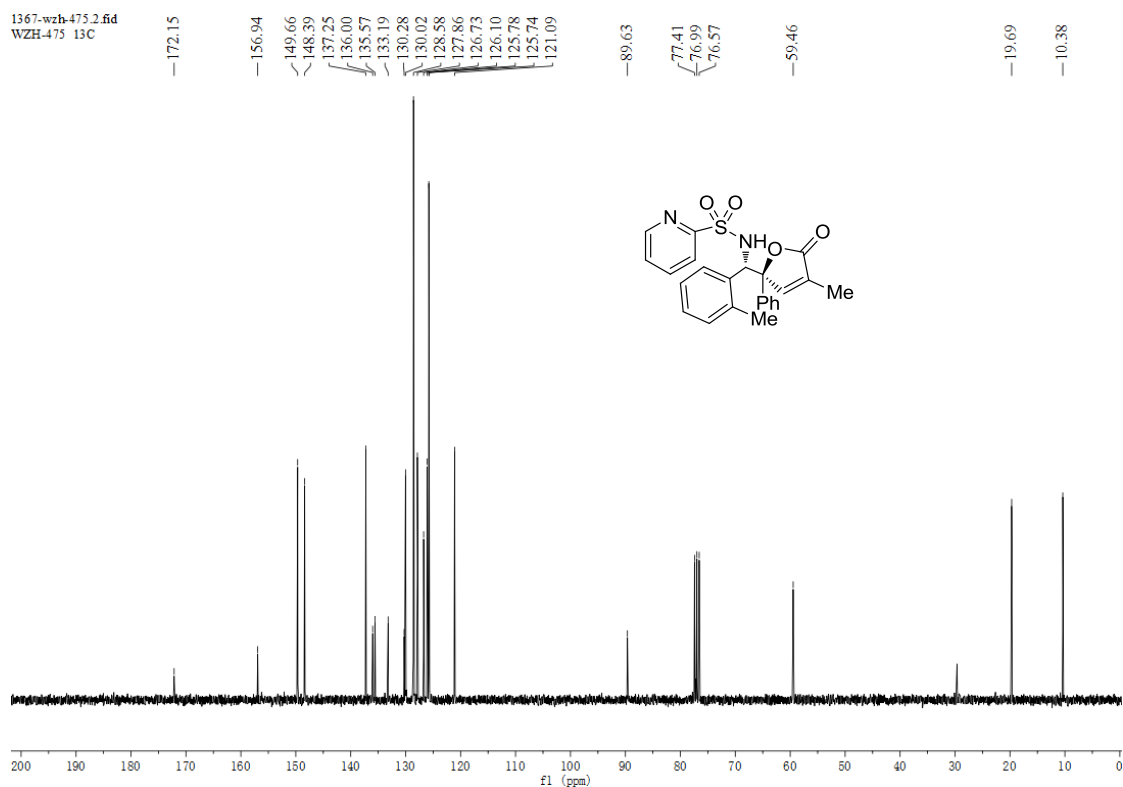
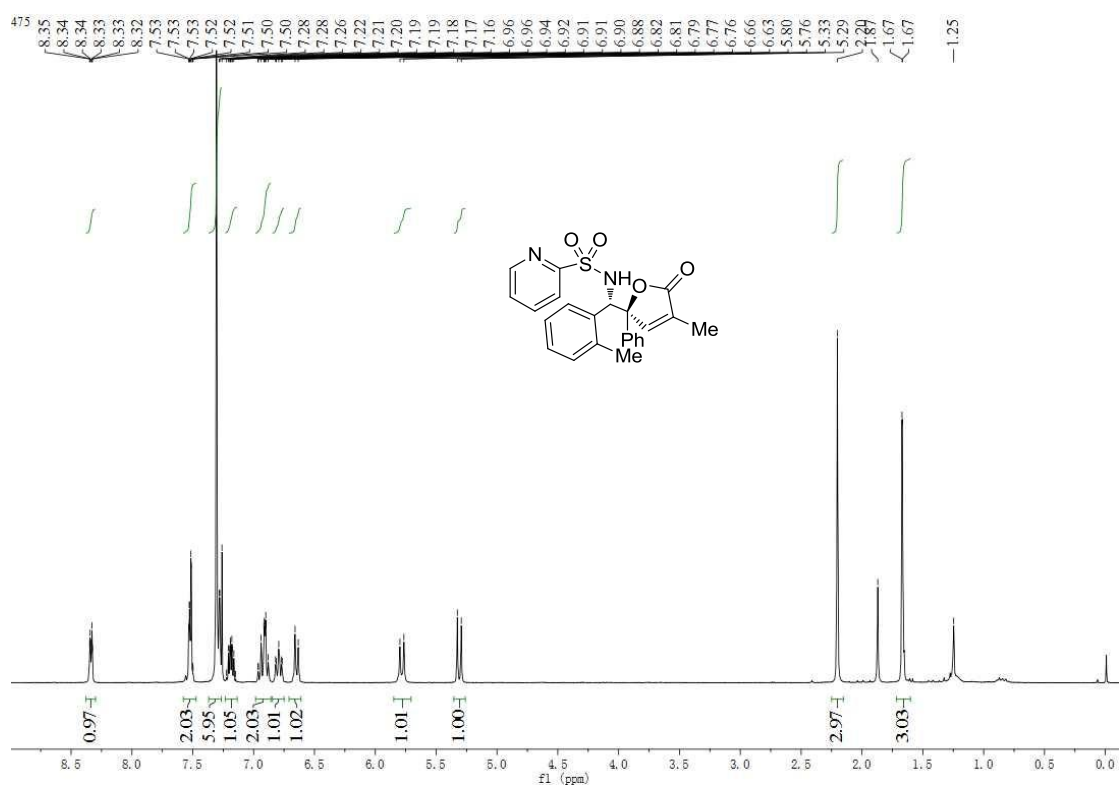
Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	31.027	26445	62.24	1789740	50.27
2	49.910	16043	37.76	1770605	49.73
Totals		42488	100.00	3560345	100.00

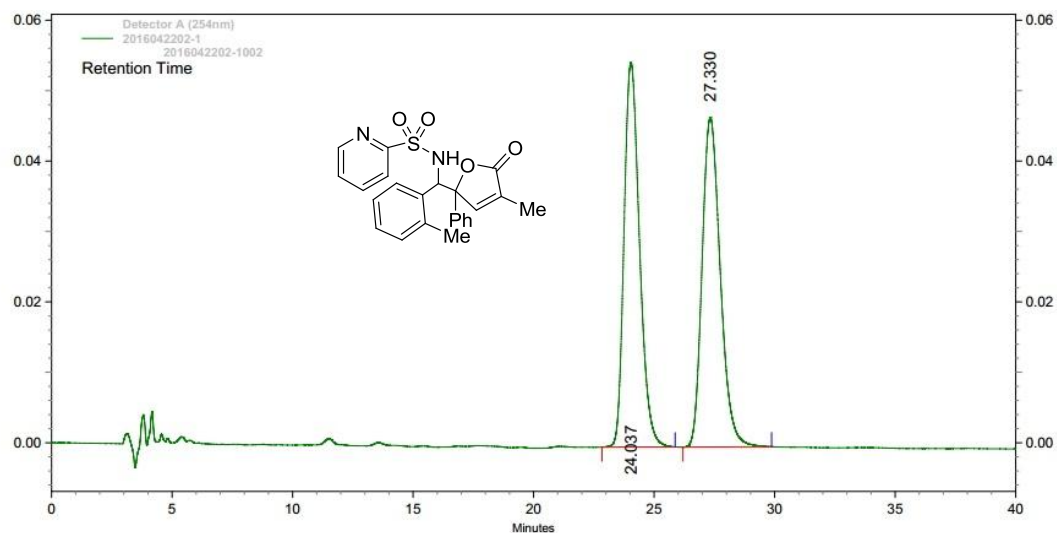


Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	31.480	14693	10.13	1159714	6.73
2	51.007	130366	89.87	16076452	93.27
Totals		145059	100.00	17236166	100.00

¹H NMR, ¹³C NMR and HPLC of 3h

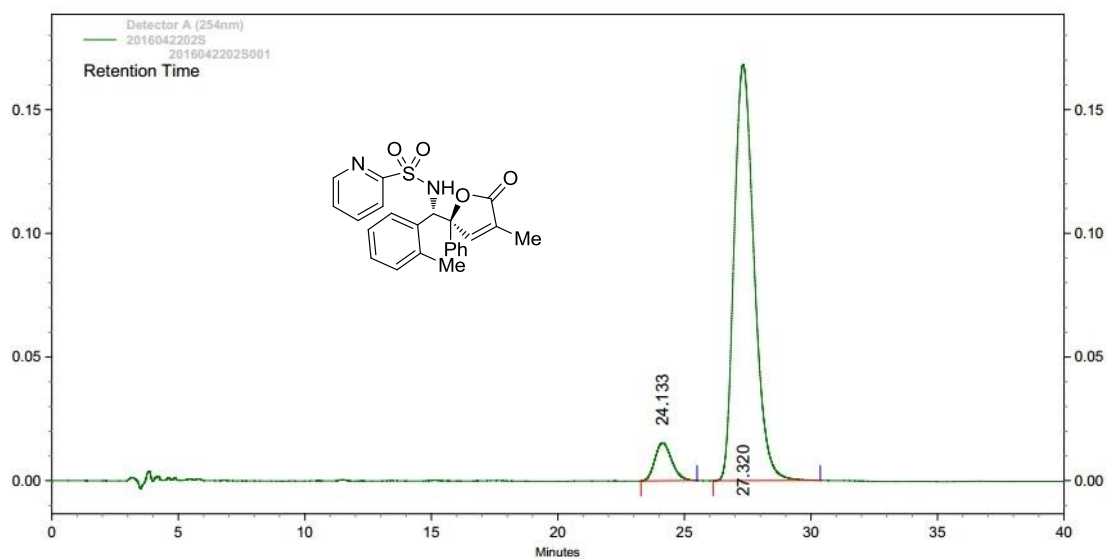




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	24.037	54665	53.90	2436116	49.80
2	27.330	46757	46.10	2455649	50.20

Totals		101422	100.00	4891765	100.00
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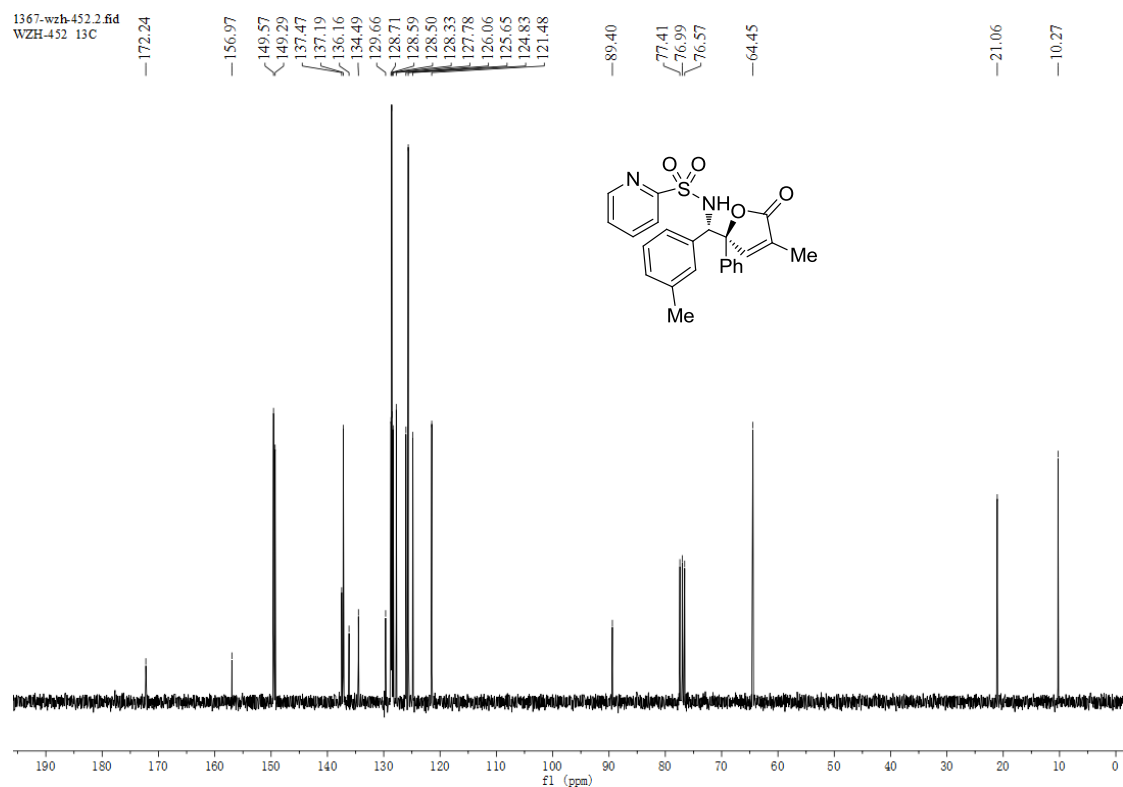
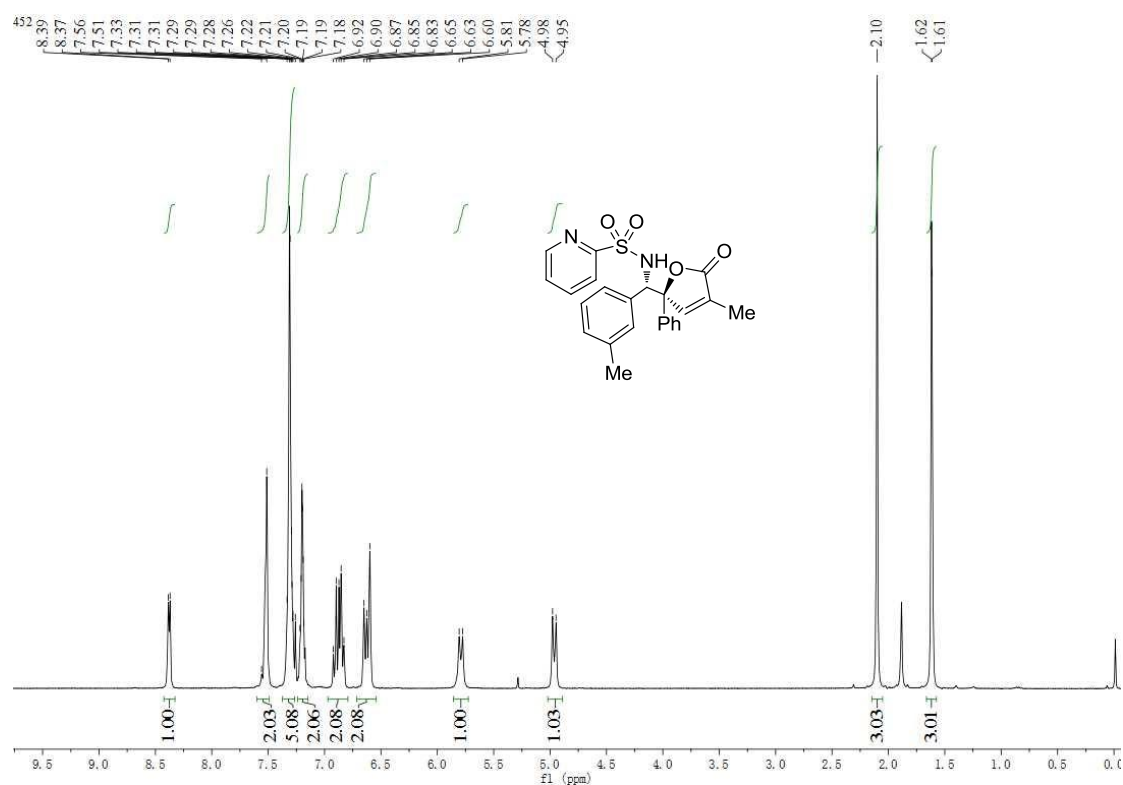


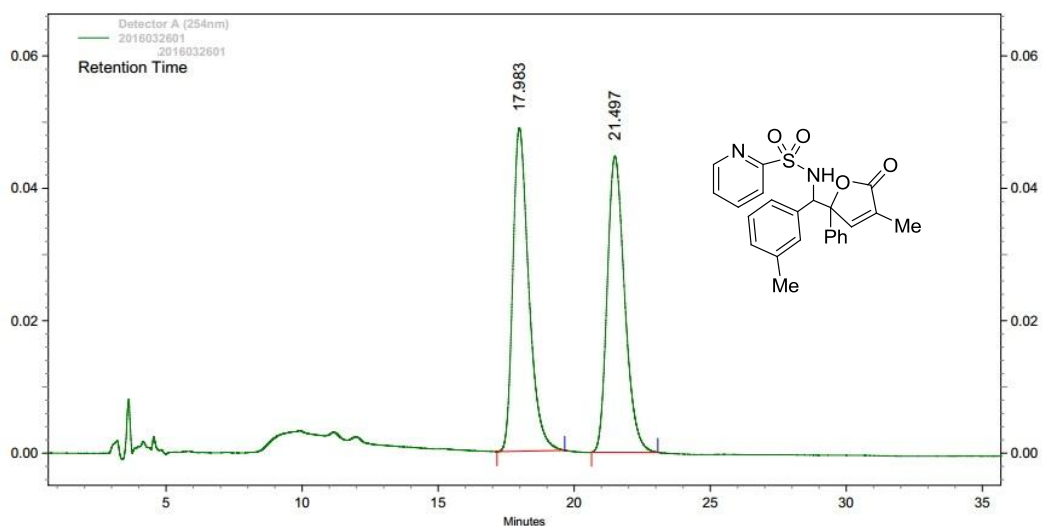
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	24.133	15387	8.38	708841	7.14
2	27.320	168296	91.62	9222813	92.86

Totals		183683	100.00	9931654	100.00
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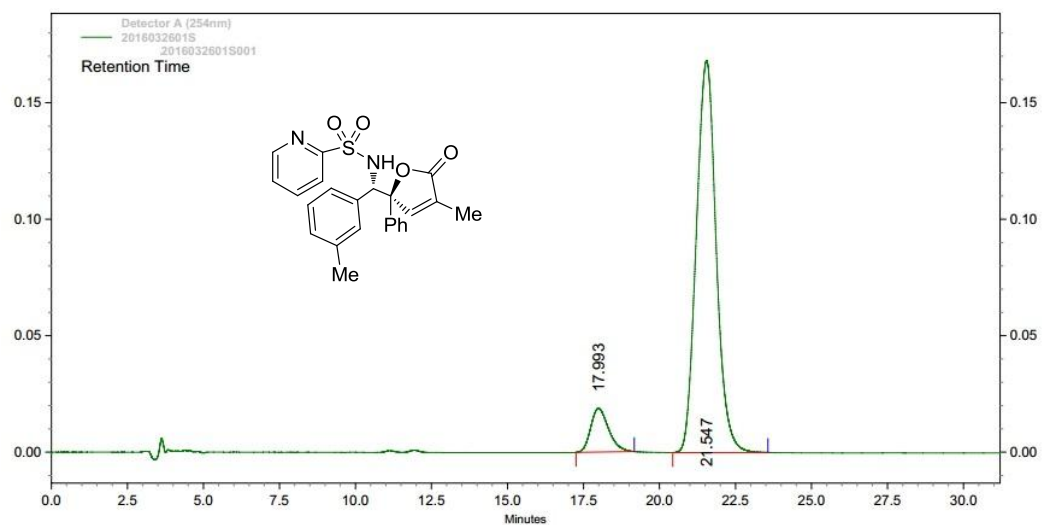
¹H NMR, ¹³C NMR and HPLC of 3i





Detector A
(254nm)

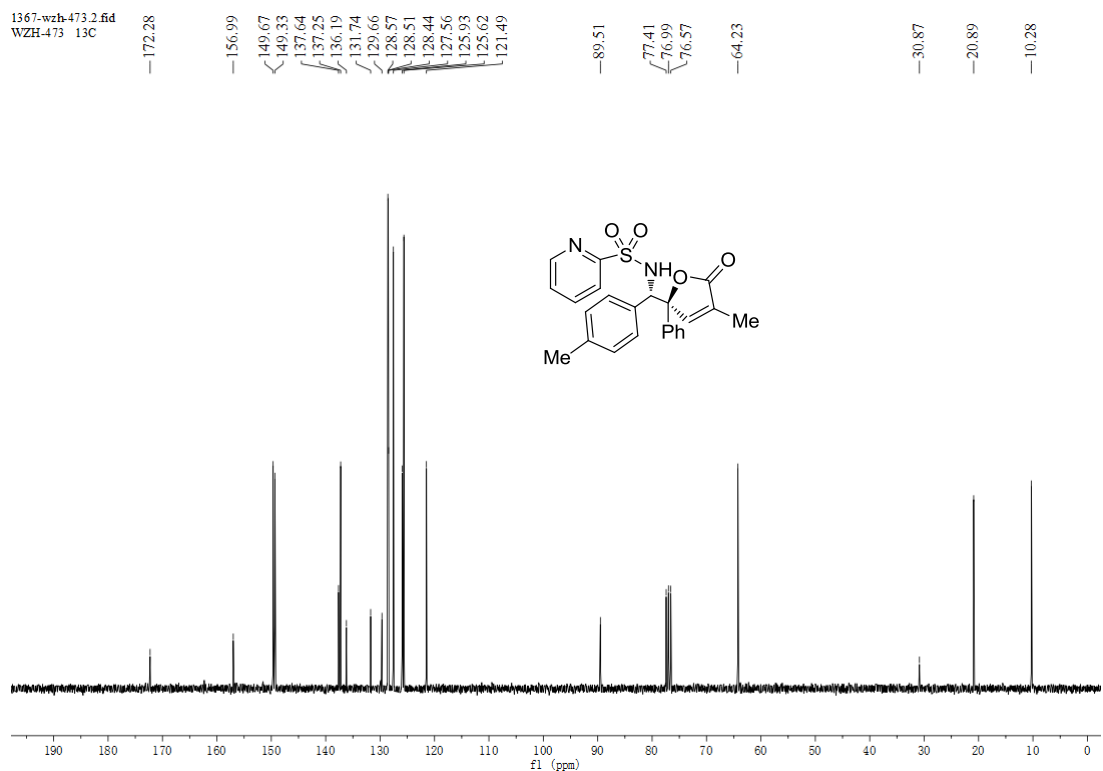
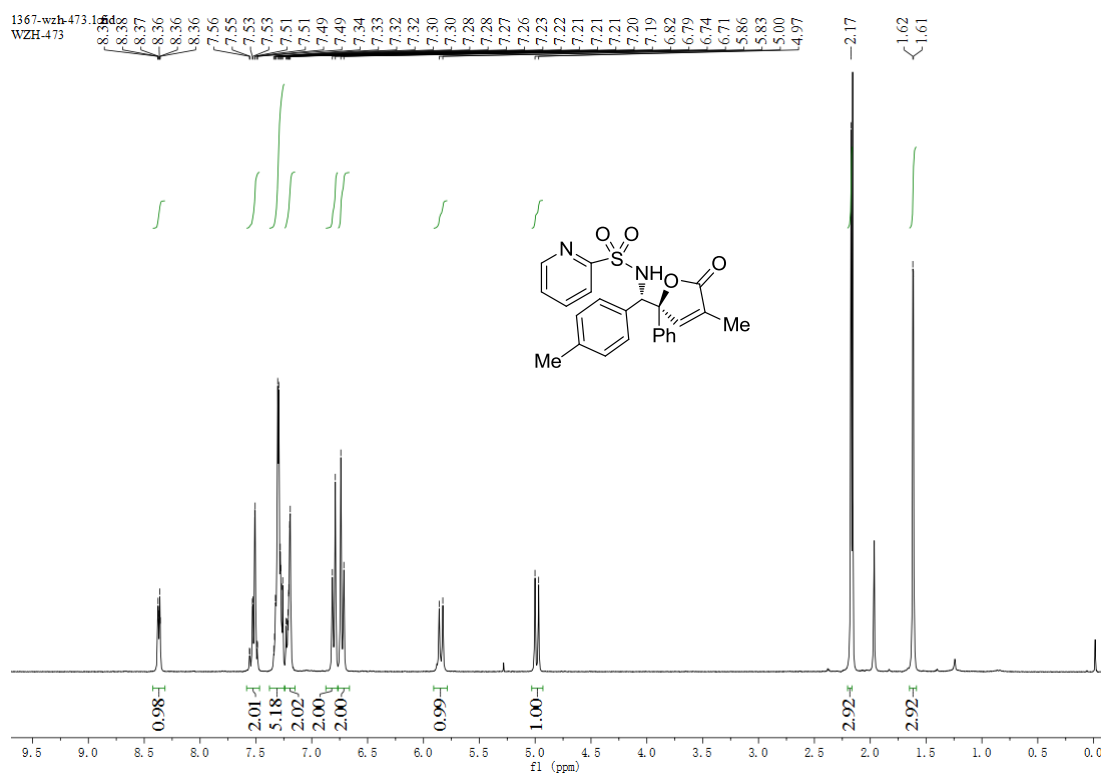
Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	17.983	48886	52.22	1988780	49.95
2	21.497	44726	47.78	1992982	50.05
Totals		93612	100.00	3981762	100.00

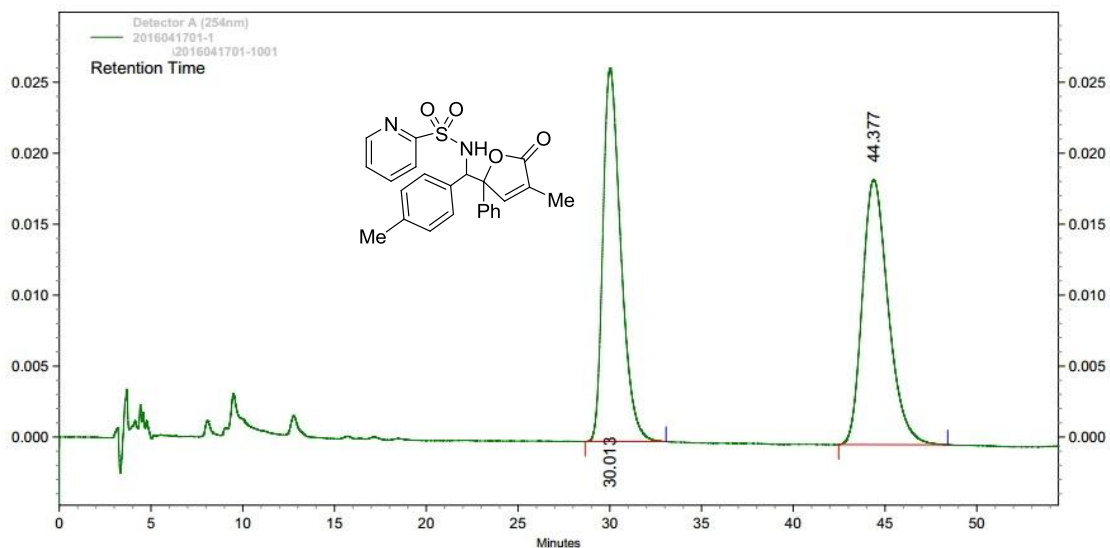


Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	17.993	18803	10.05	769784	9.16
2	21.547	168313	89.95	7635880	90.84
Totals		187116	100.00	8405664	100.00

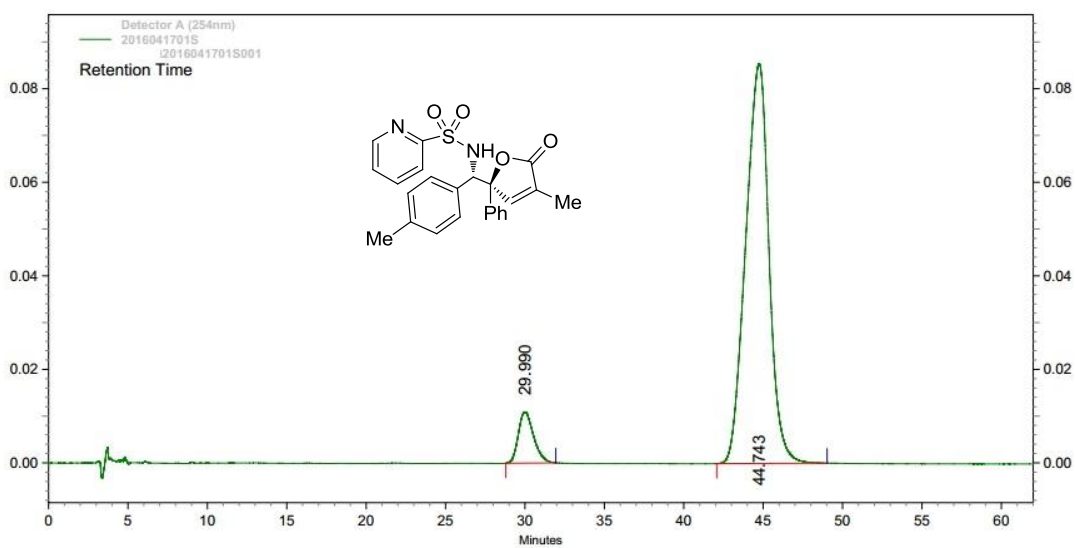
¹H NMR, ¹³C NMR and HPLC of 3j





Detector A
(254nm)

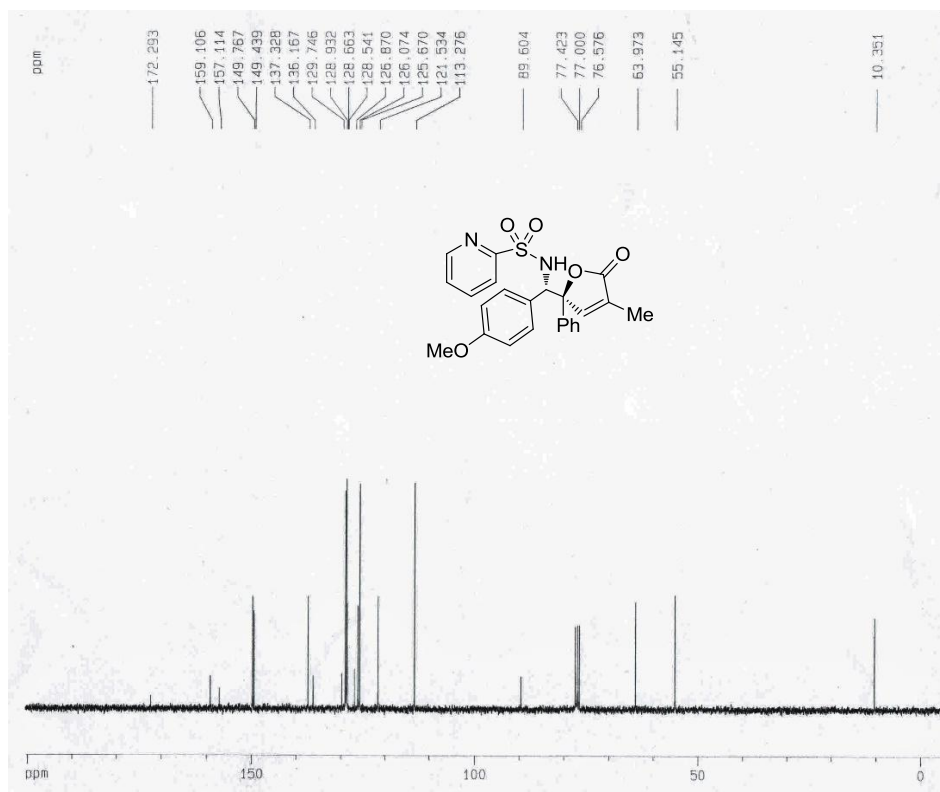
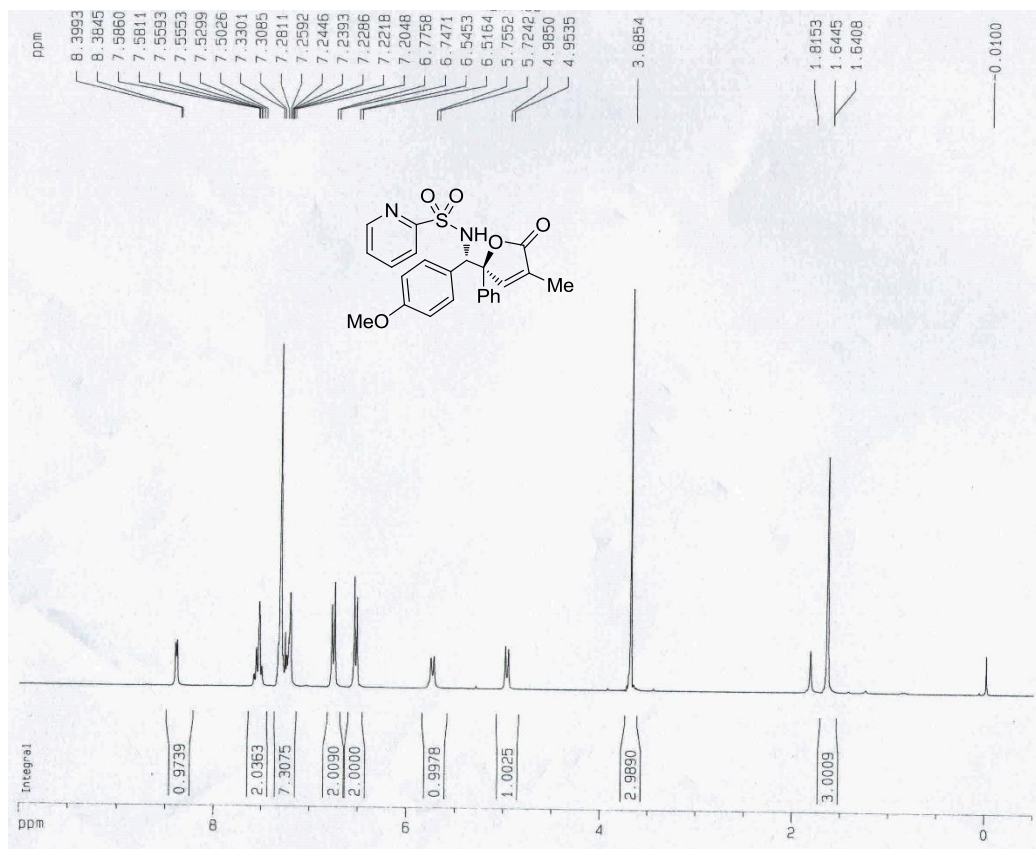
Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	30.013	26299	58.46	1785514	49.32
2	44.377	18689	41.54	1834441	50.68
Totals		44988	100.00	3619955	100.00

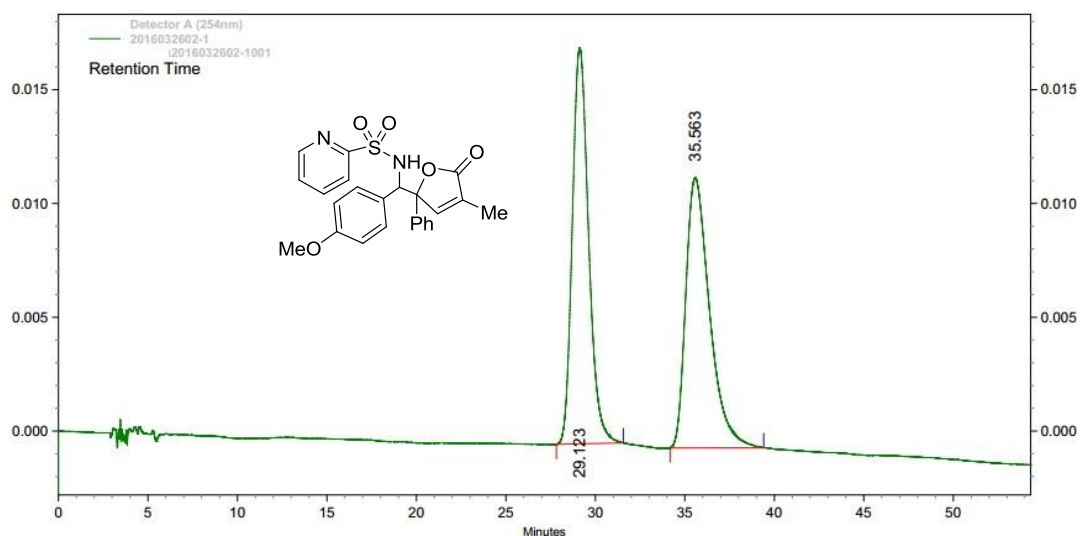


Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	29.990	10969	11.37	728507	7.73
2	44.743	85527	88.63	8698614	92.27
Totals		96496	100.00	9427121	100.00

¹H NMR, ¹³C NMR and HPLC of 3k

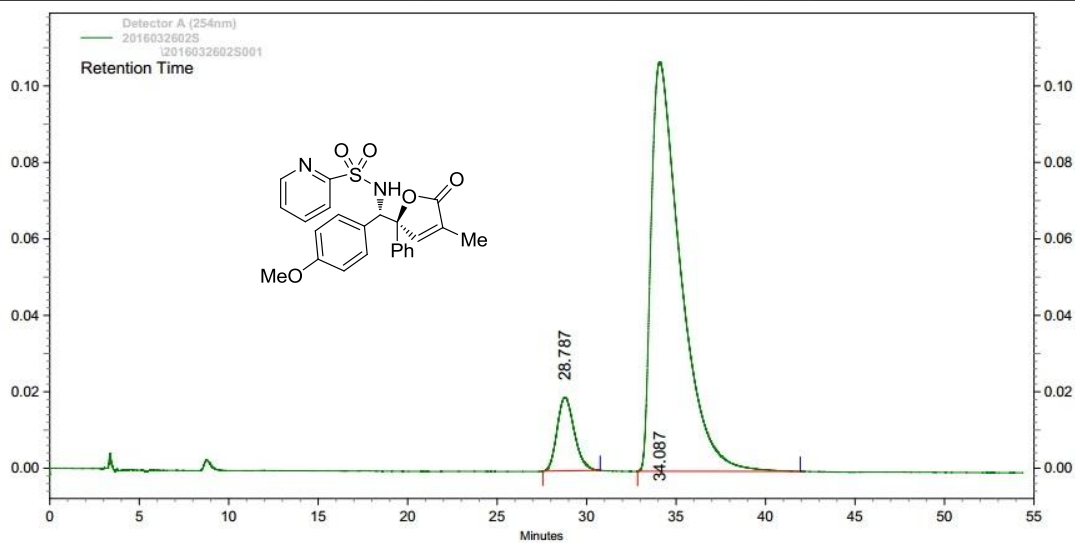




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	29.123	17420	59.46	1101192	50.14
2	35.563	11876	40.54	1095256	49.86

Totals		29296	100.00	2196448	100.00
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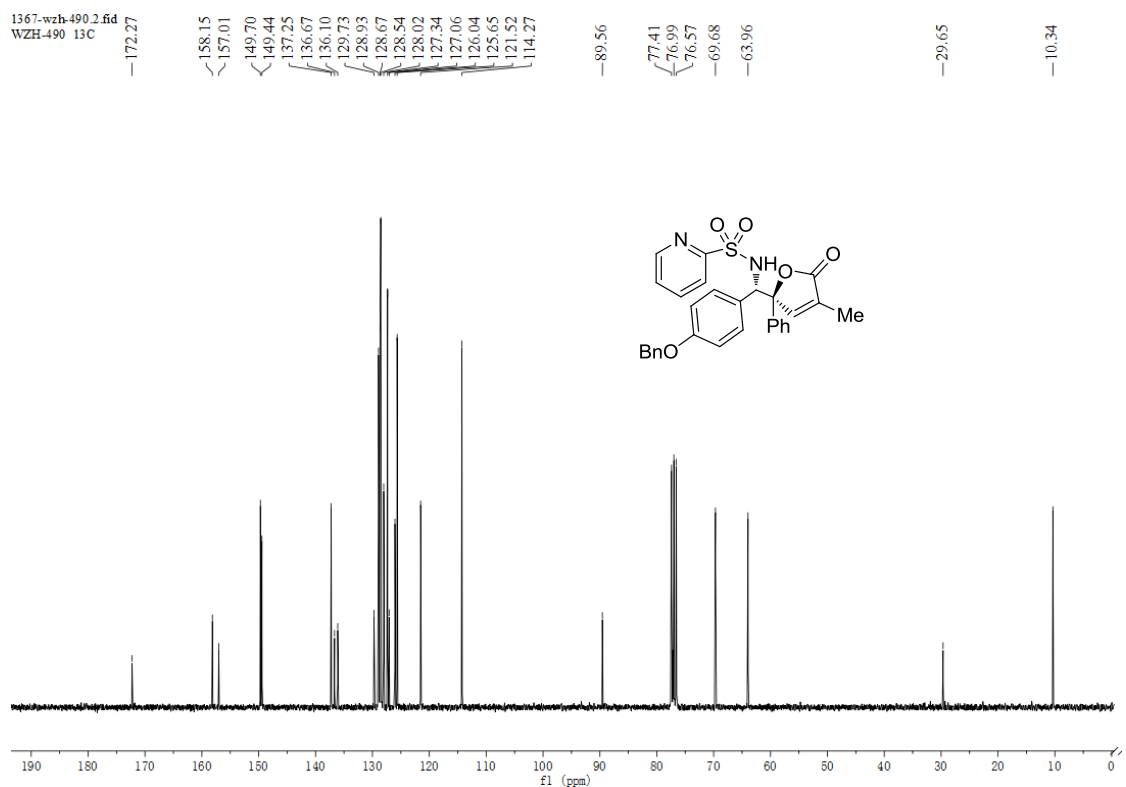
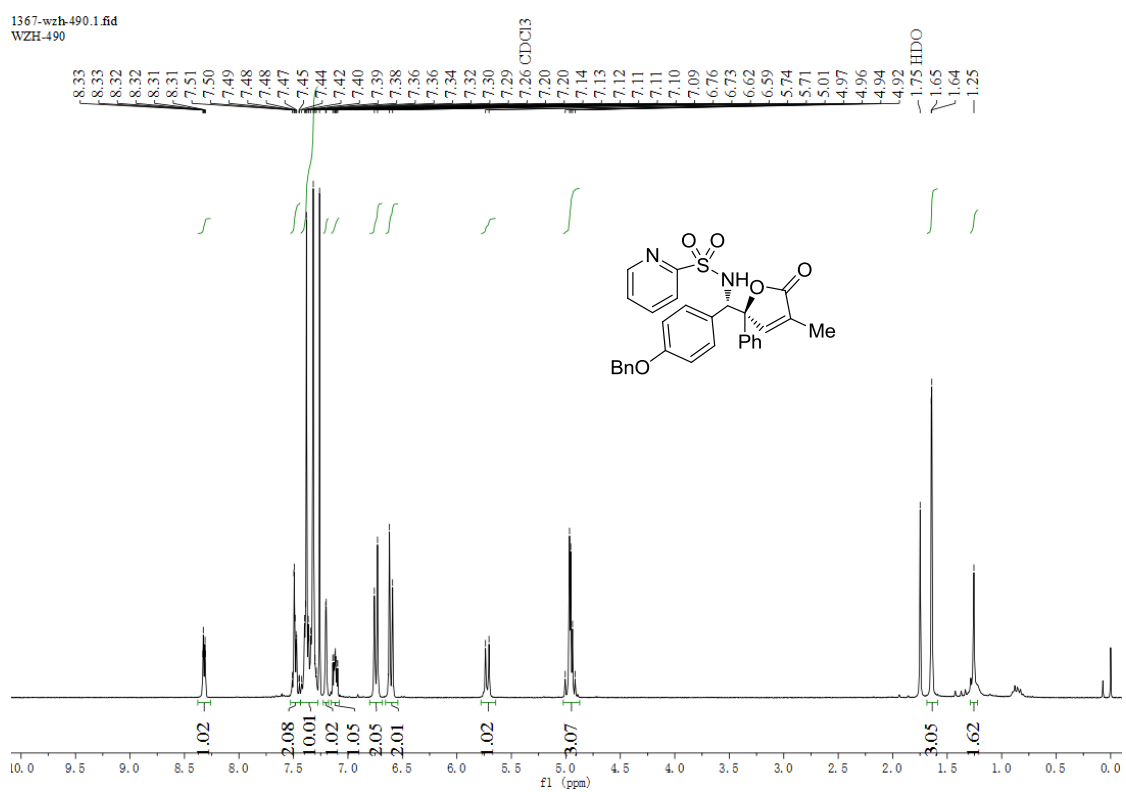


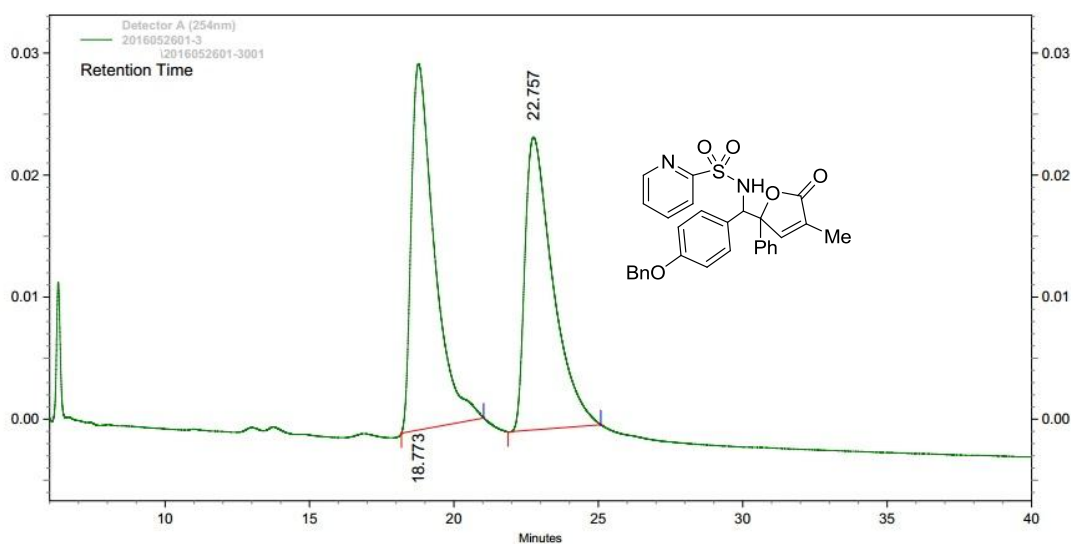
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	28.787	19228	15.22	1284422	9.52
2	34.087	107144	84.78	12214494	90.49

Totals		126372	100.00	13498916	100.00
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¹H NMR, ¹³C NMR and HPLC of 3l

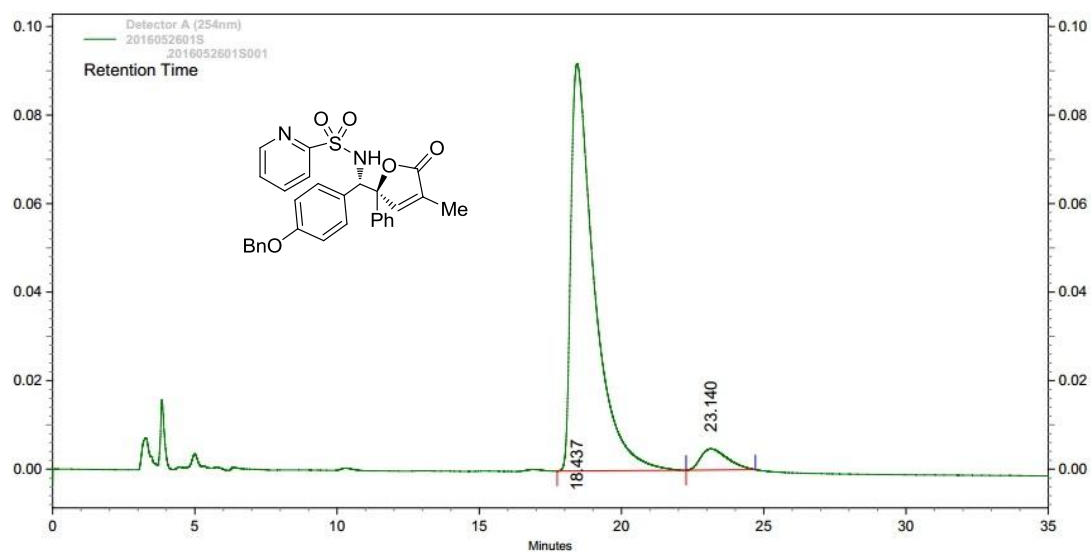




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	18.773	30008	55.56	1671661	50.90
2	22.757	23999	44.44	1612668	49.10

Totals		54007	100.00	3284329	100.00
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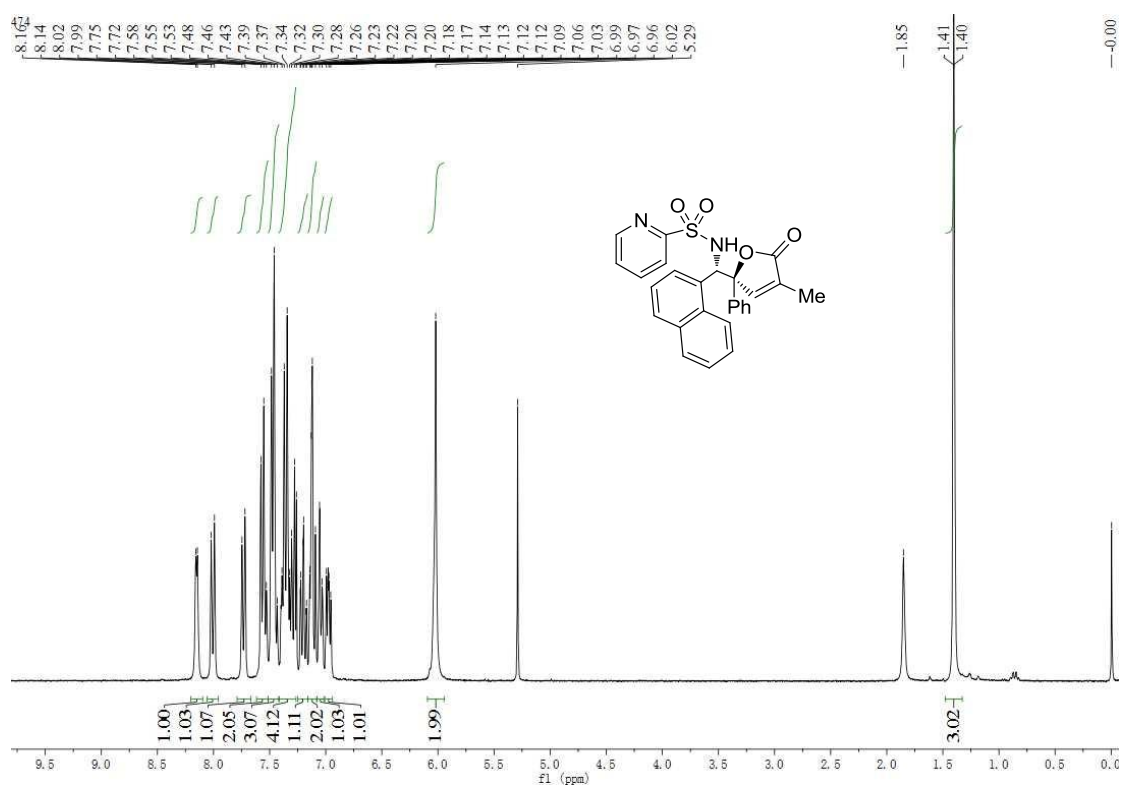


Detector A
(254nm)

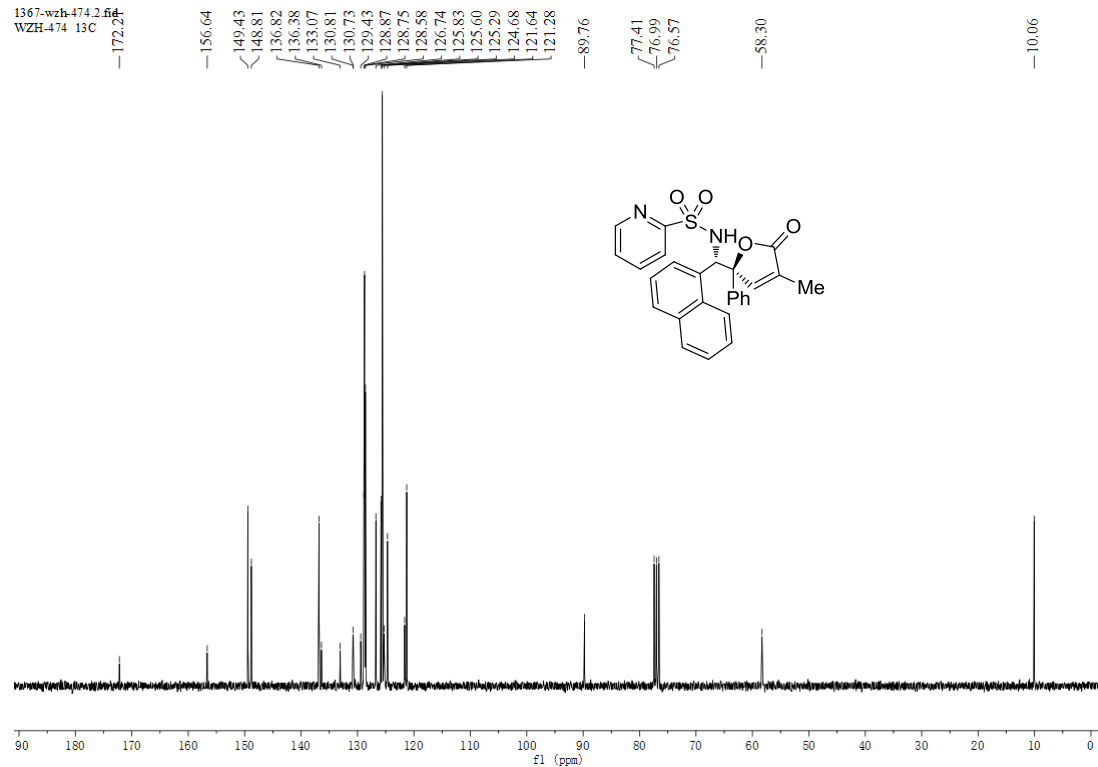
Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	18.437	92053	95.03	5226965	94.34
2	23.140	4815	4.97	313523	5.66

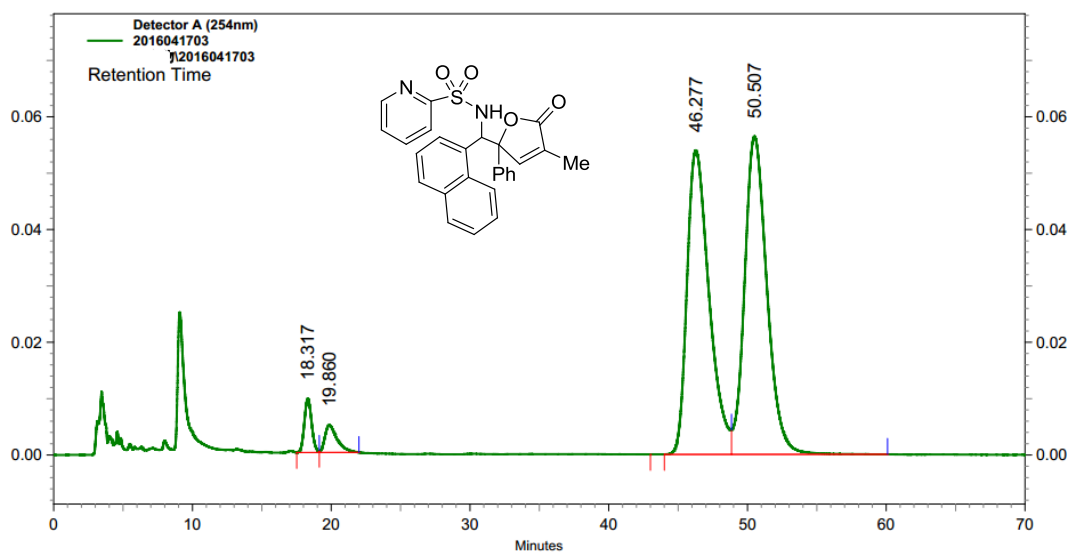
Totals		96868	100.00	5540488	100.00
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¹H NMR, ¹³C NMR and HPLC of 3m



1367-wzh-474.2.fid
WZH-474 13C

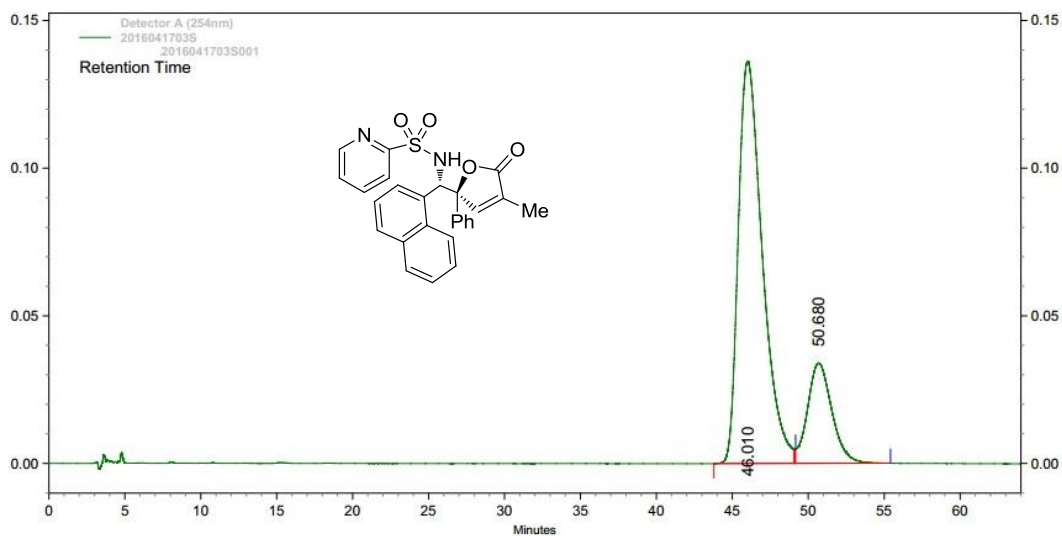




Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	18.317	9587	7.68	350095	1.85
2	19.860	4857	3.89	297209	1.57
3	46.277	53958	43.22	5966505	31.55
4	50.507	56435	45.21	12296093	65.02

Totals		124837	100.00	18909902	100.00
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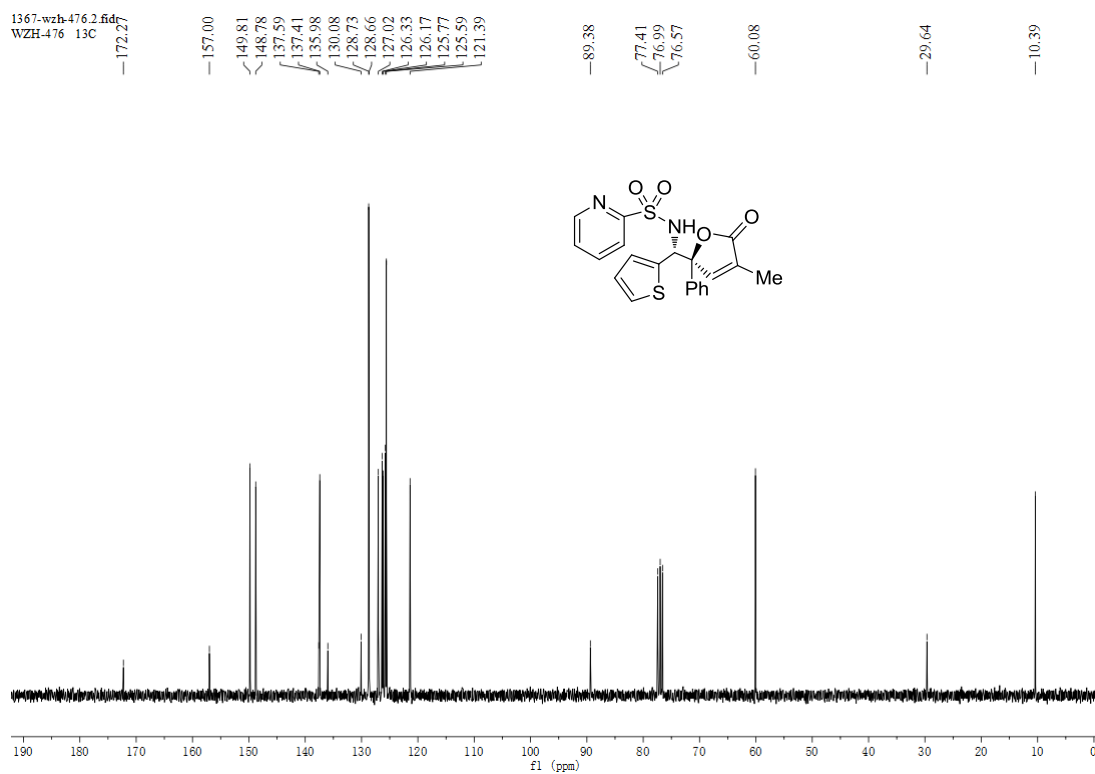
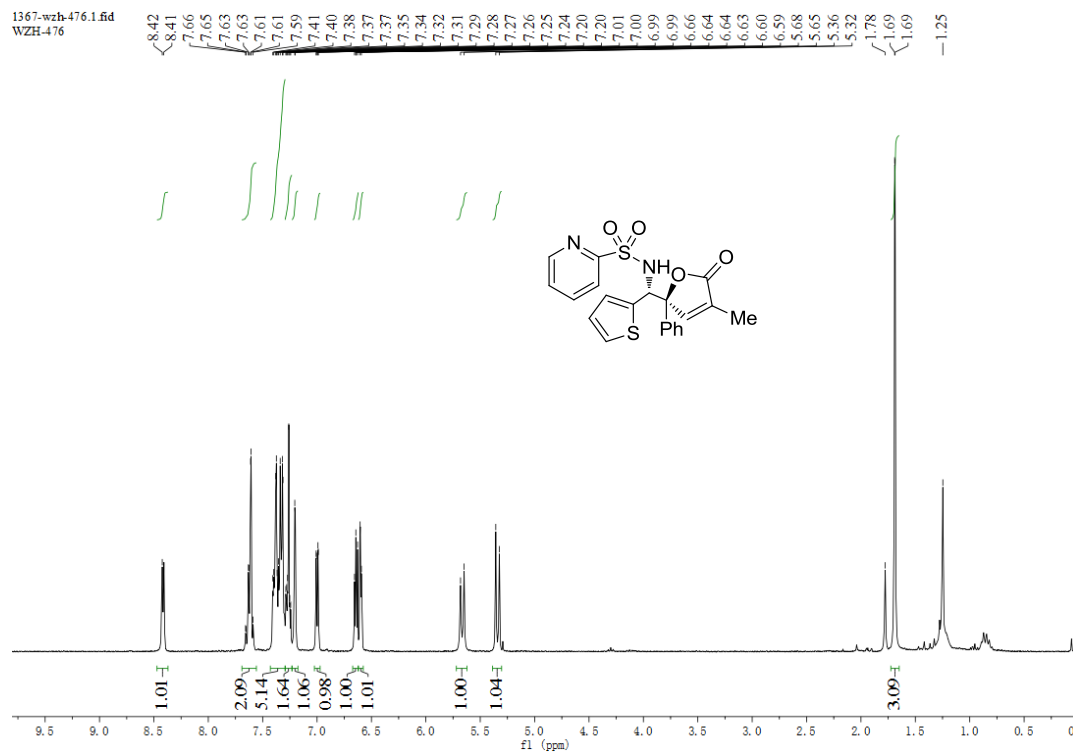


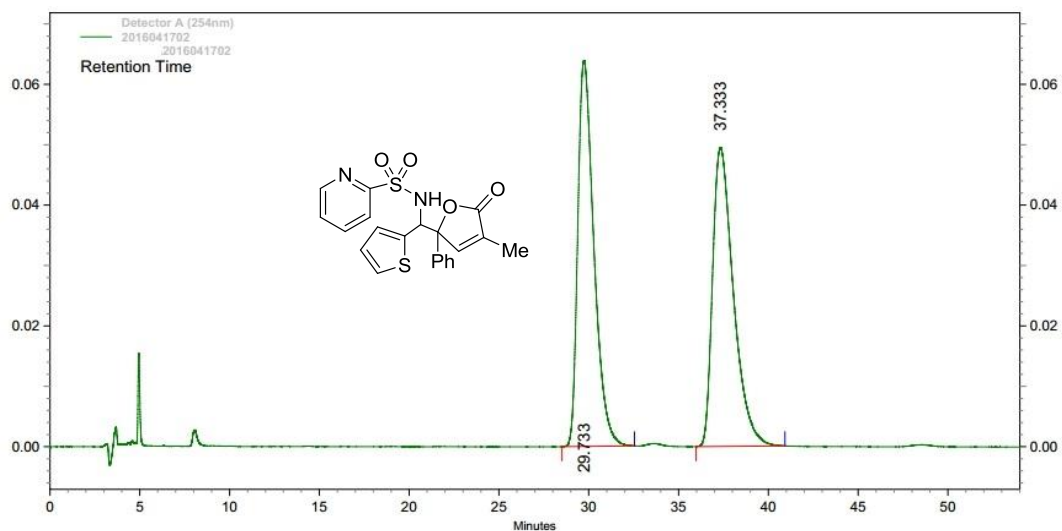
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	46.010	136275	80.05	15058673	79.87
2	50.680	33967	19.95	3796396	20.13

Totals		170242	100.00	18855069	100.00
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¹H NMR, ¹³C NMR and HPLC of 3n

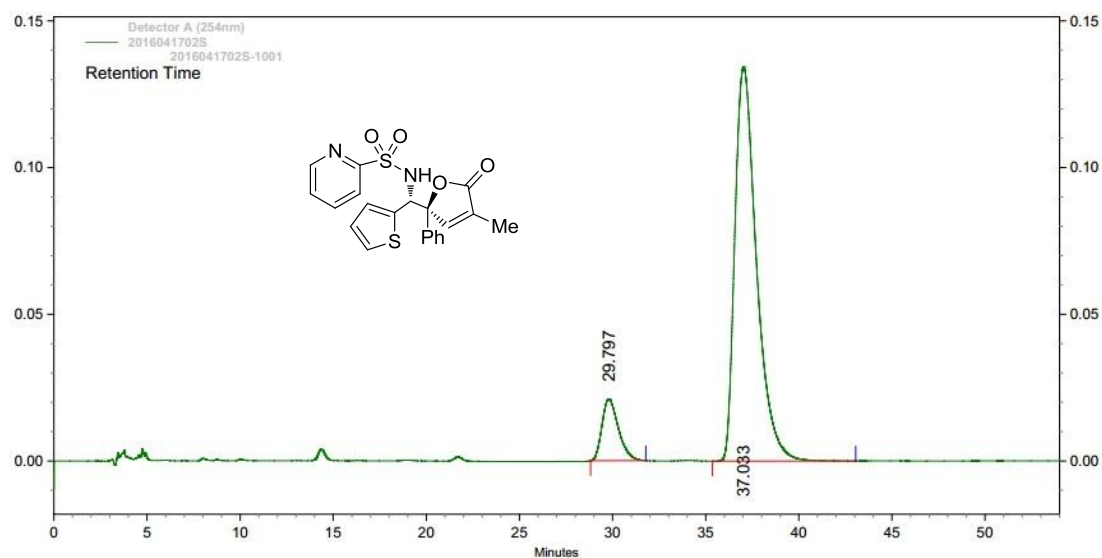




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	29.733	63922	56.37	3987756	50.02
2	37.333	49466	43.63	3984481	49.98

Totals		113388	100.00	7972237	100.00
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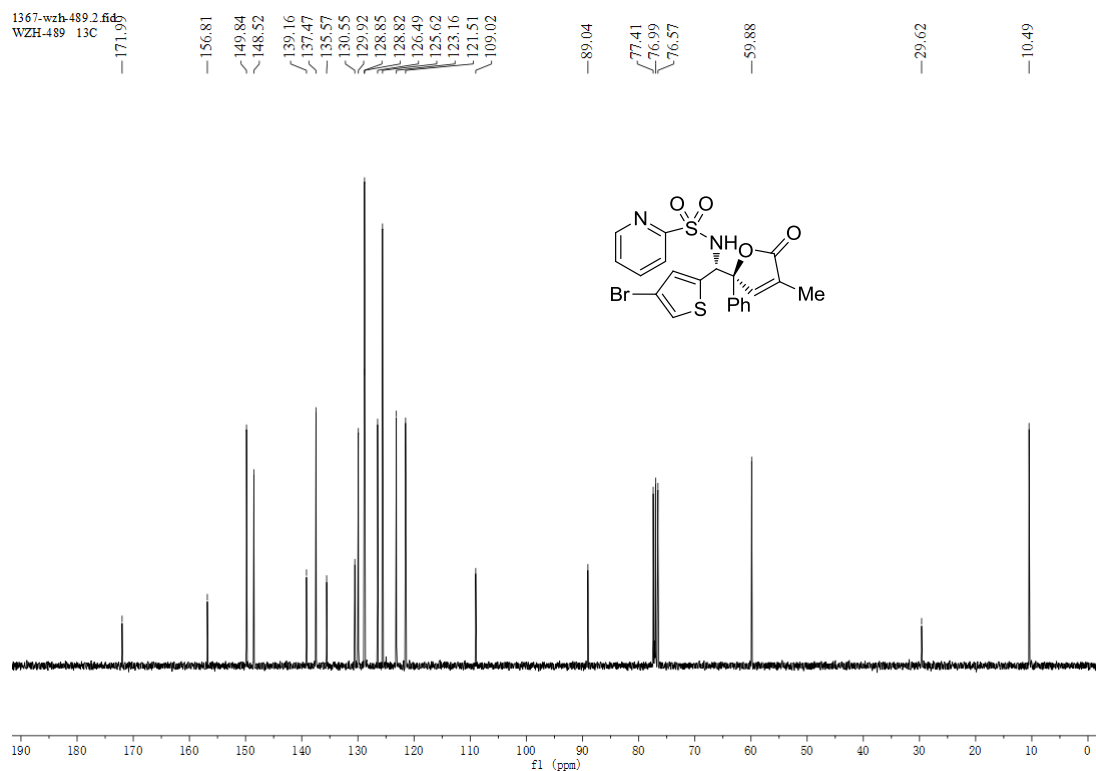
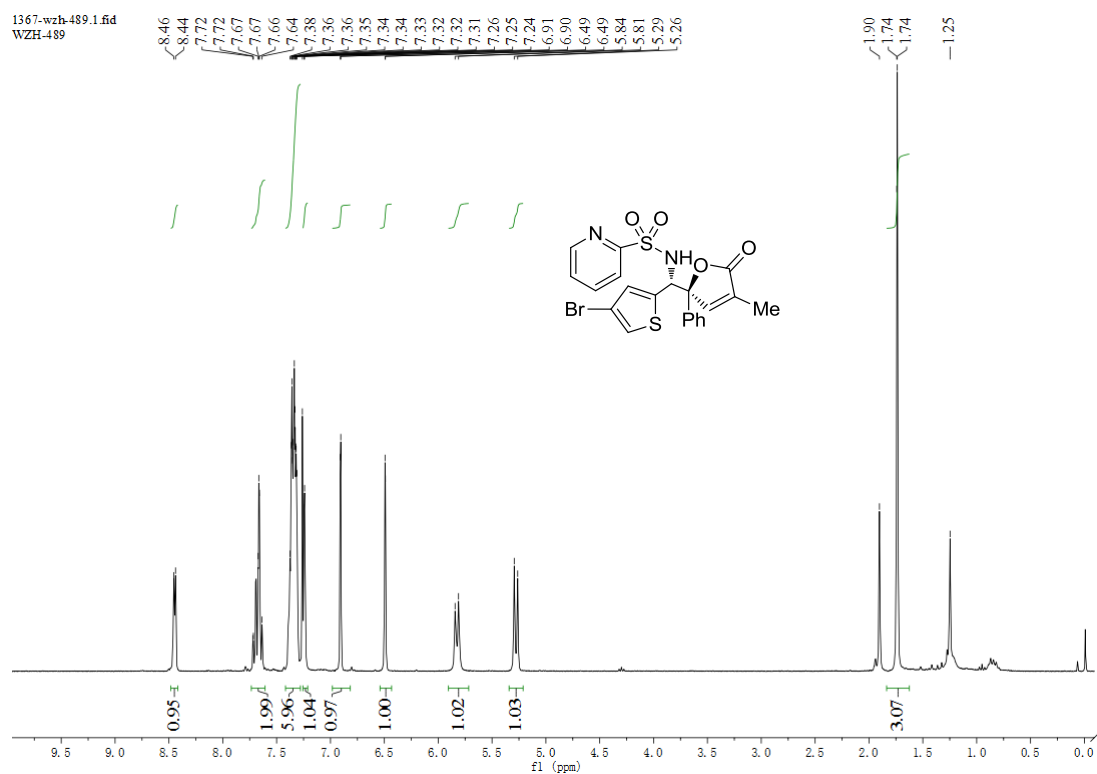


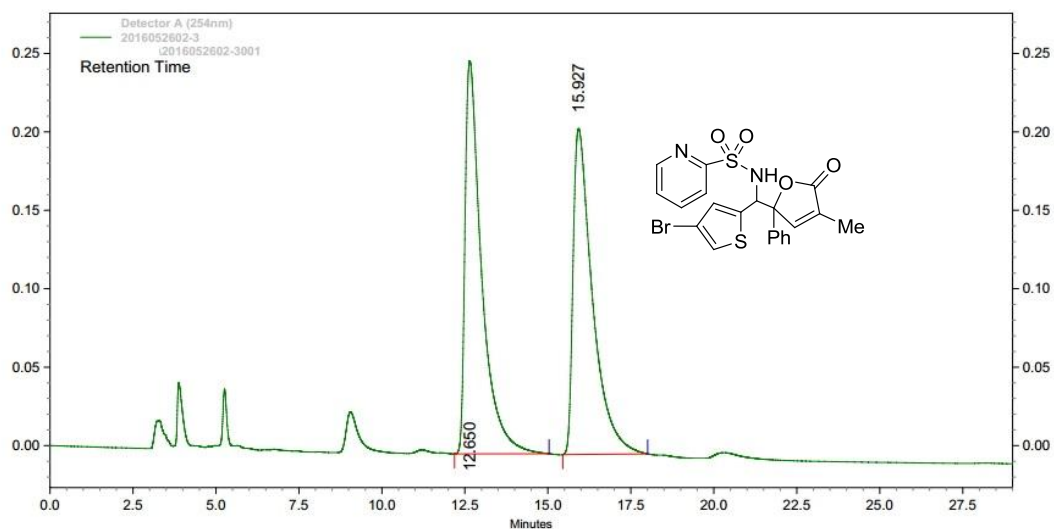
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	29.797	20982	13.50	1292097	10.65
2	37.033	134489	86.50	10834749	89.35

Totals		155471	100.00	12126846	100.00
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¹H NMR, ¹³C NMR and HPLC of 3o

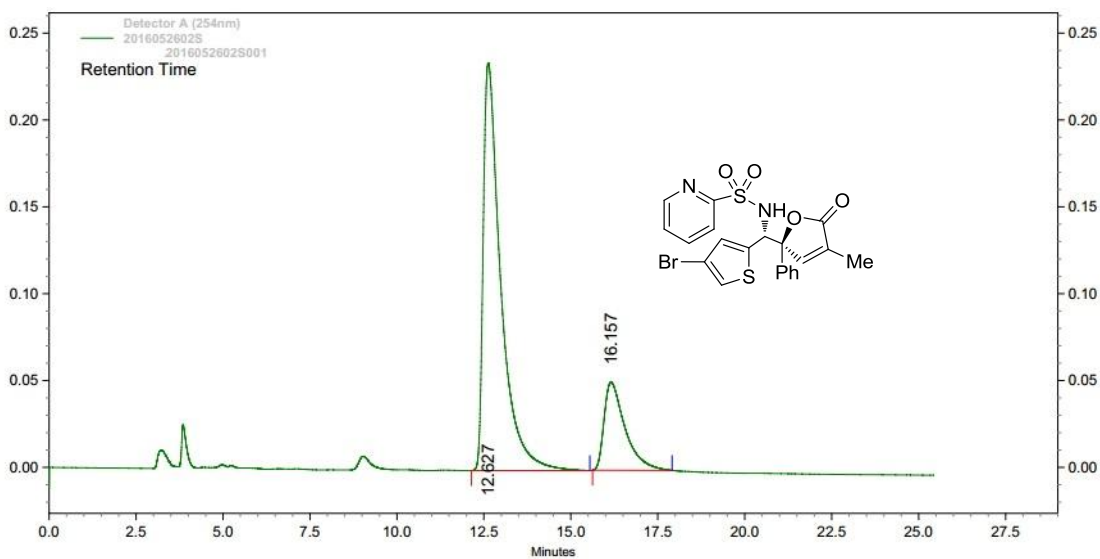




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.650	250667	54.67	8727476	50.75
2	15.927	207804	45.33	8469146	49.25

Totals		458471	100.00	17196622	100.00
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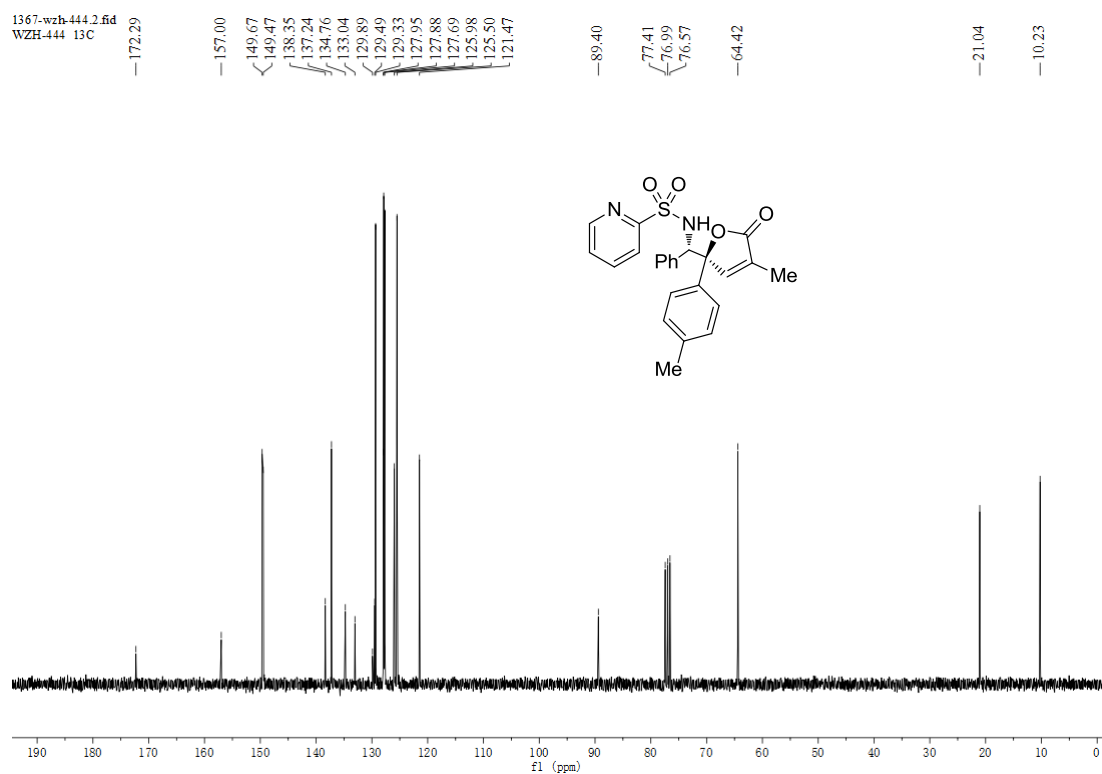
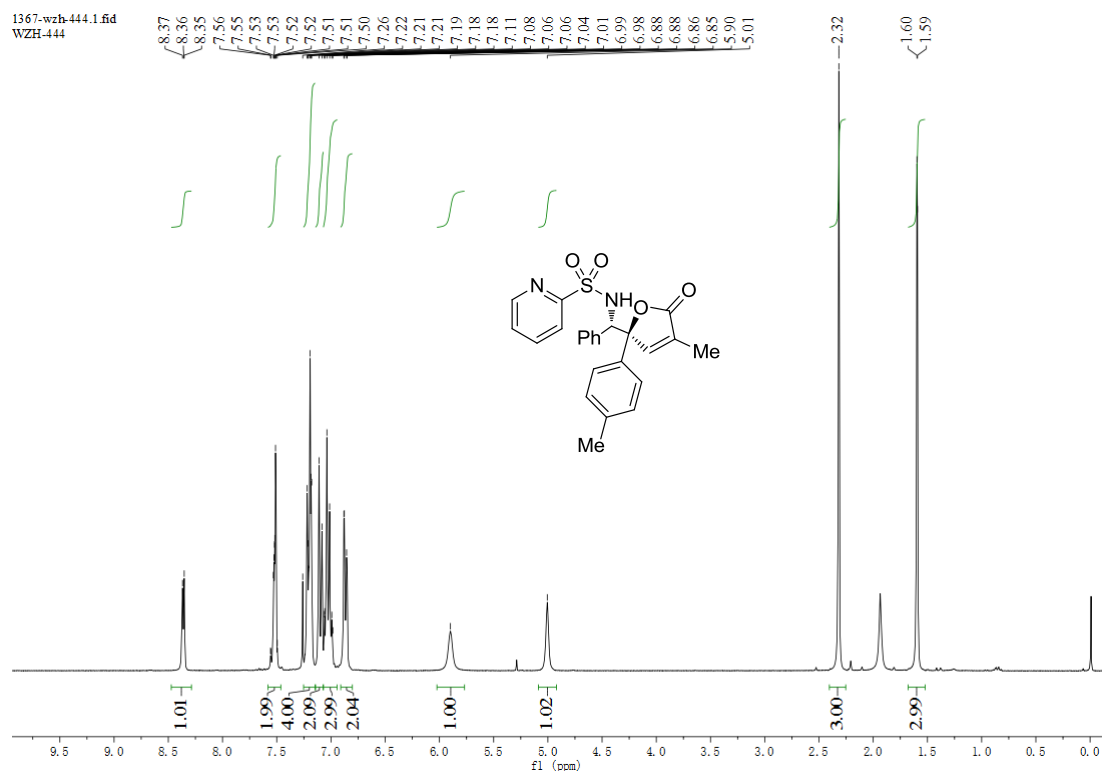


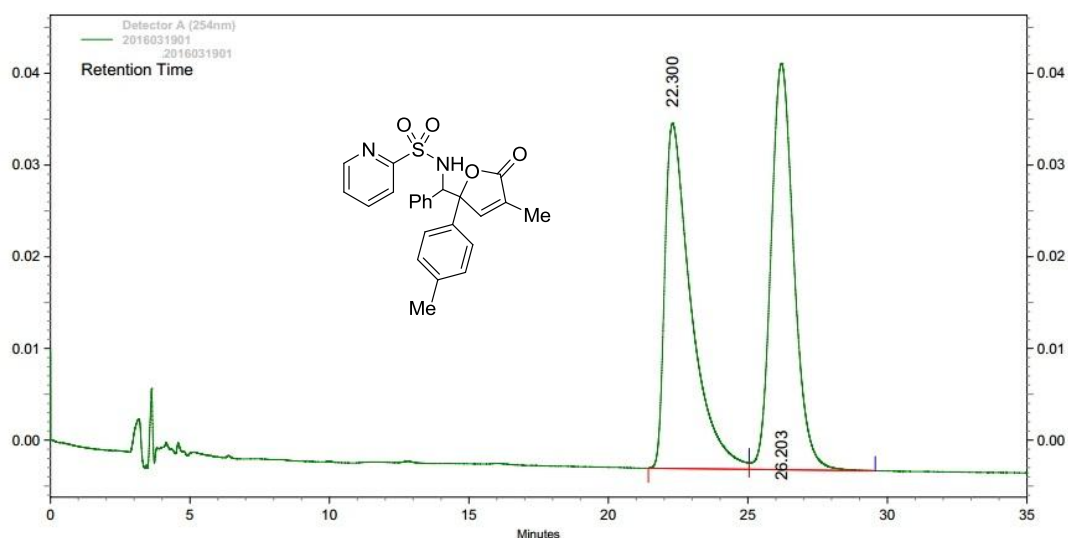
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.627	234732	82.23	8233658	79.48
2	16.157	50727	17.77	2126136	20.52

Totals		285459	100.00	10359794	100.00
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¹H NMR, ¹³C NMR and HPLC of 3p

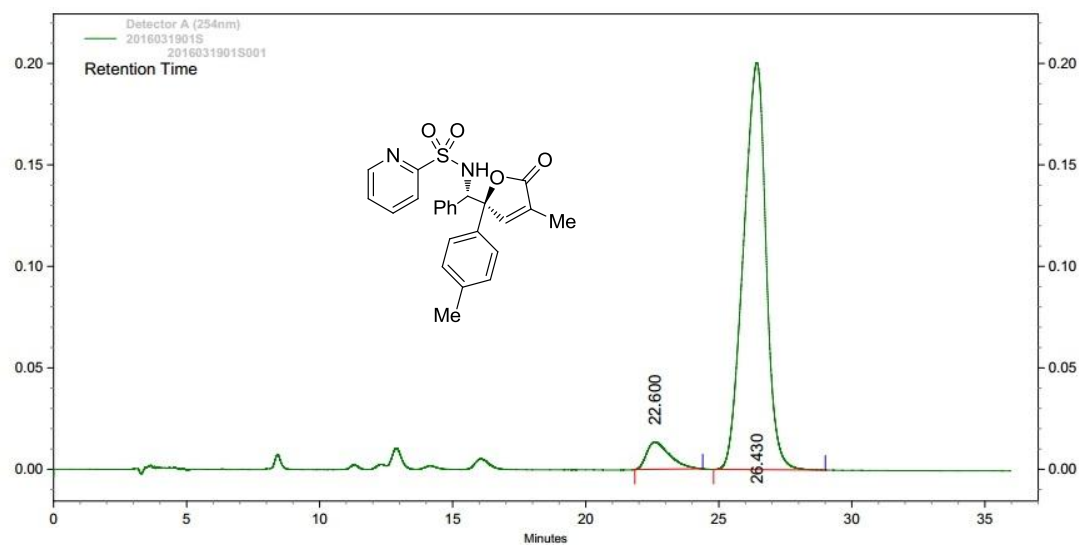




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	22.300	37625	45.92	2460494	49.36
2	26.203	44314	54.08	2524243	50.64

Totals		81939	100.00	4984737	100.00
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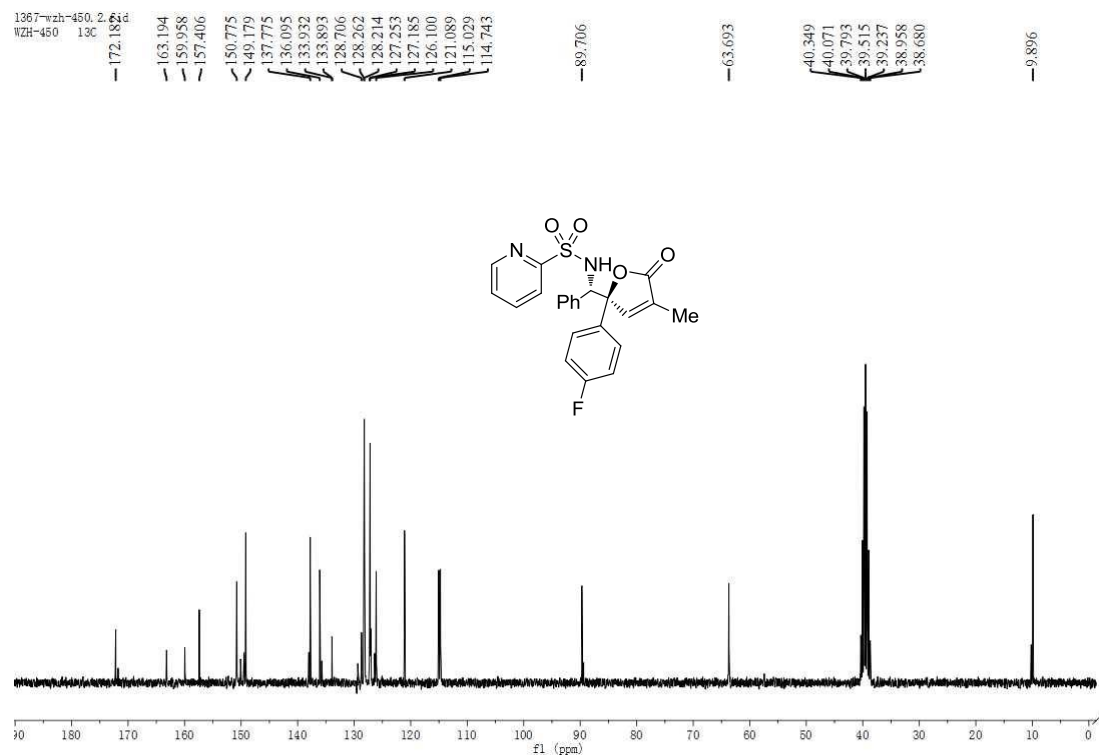
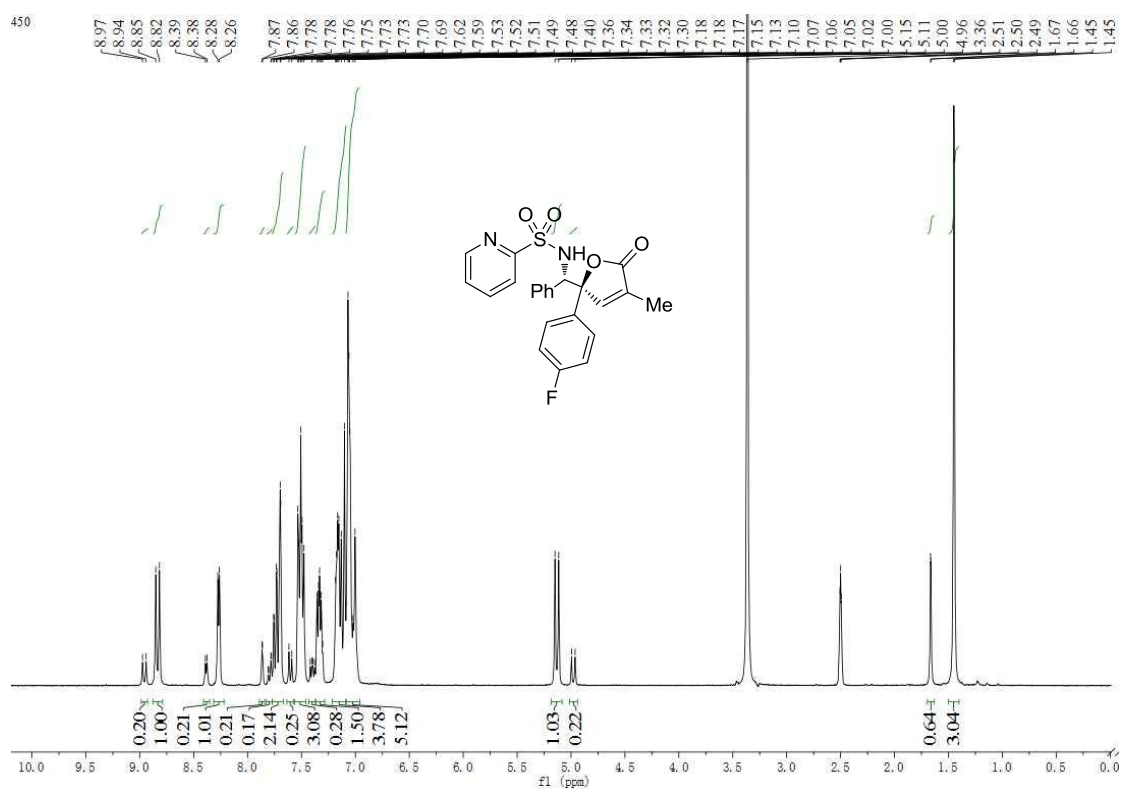


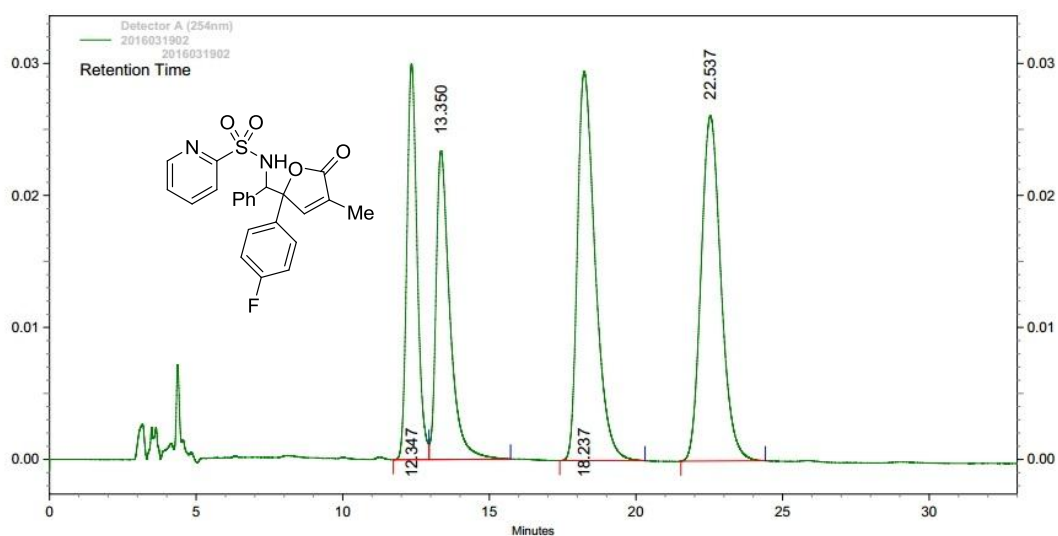
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	22.600	13435	6.28	807926	6.42
2	26.430	200596	93.72	11775455	93.58

Totals		214031	100.00	12583381	100.00
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¹H NMR, ¹³C NMR and HPLC of 3q

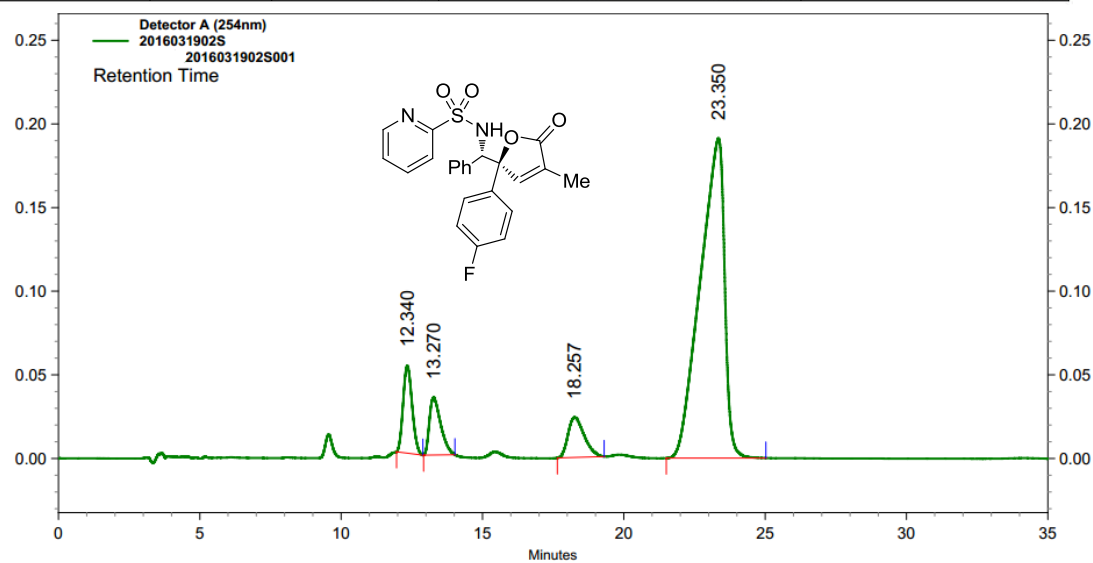




Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.347	29996	27.52	730805	18.41
2	13.350	23373	21.44	767091	19.32
3	18.237	29472	27.04	1238446	31.20
4	22.537	26152	23.99	1233222	31.07

Totals		108993	100.00	3969564	100.00
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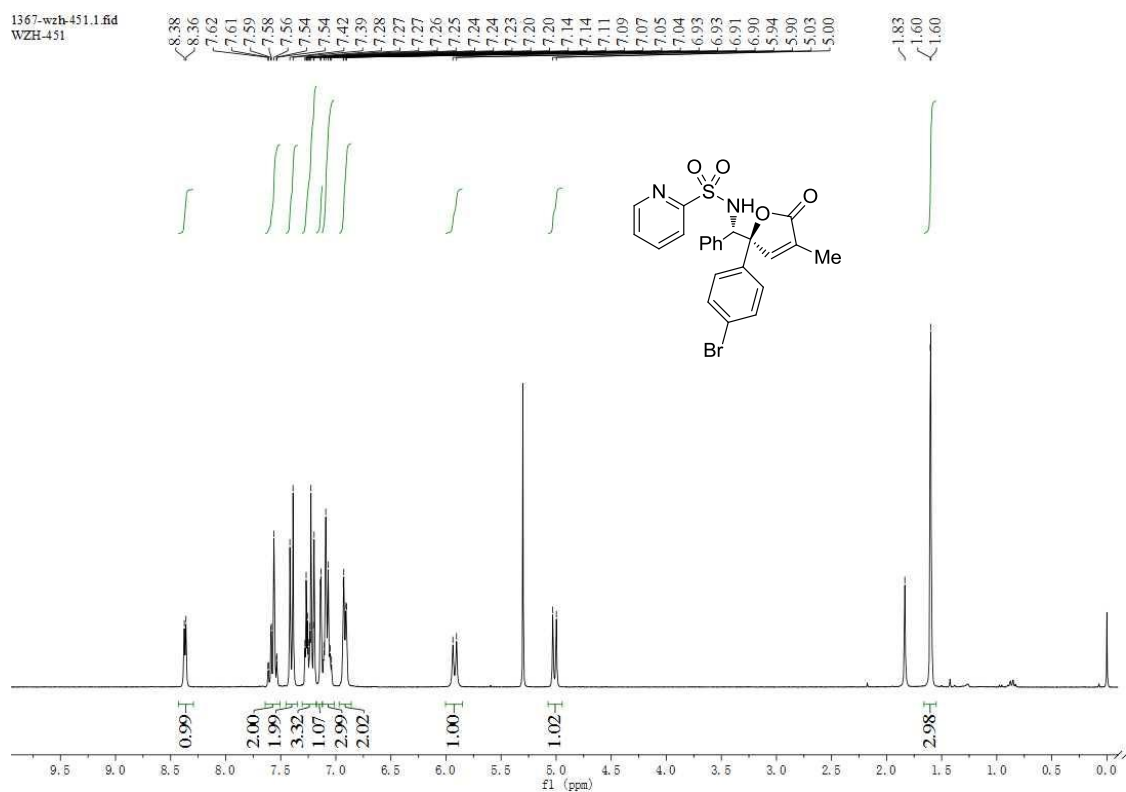
Detector A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	12.340	52317	17.34	1166823	8.05
2	13.270	34312	11.37	955719	6.60
3	18.257	23952	7.94	954882	6.59
4	23.350	191179	63.35	11408705	78.76

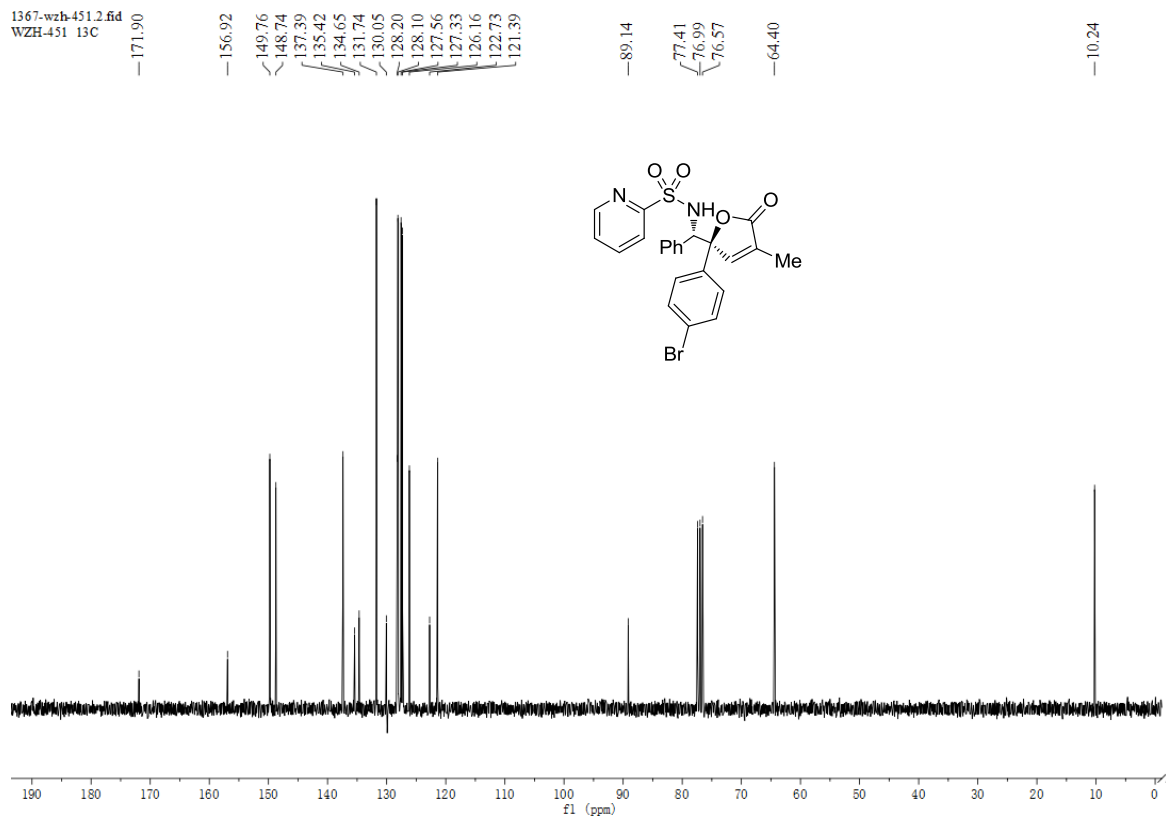
Totals		301760	100.00	14486129	100.00
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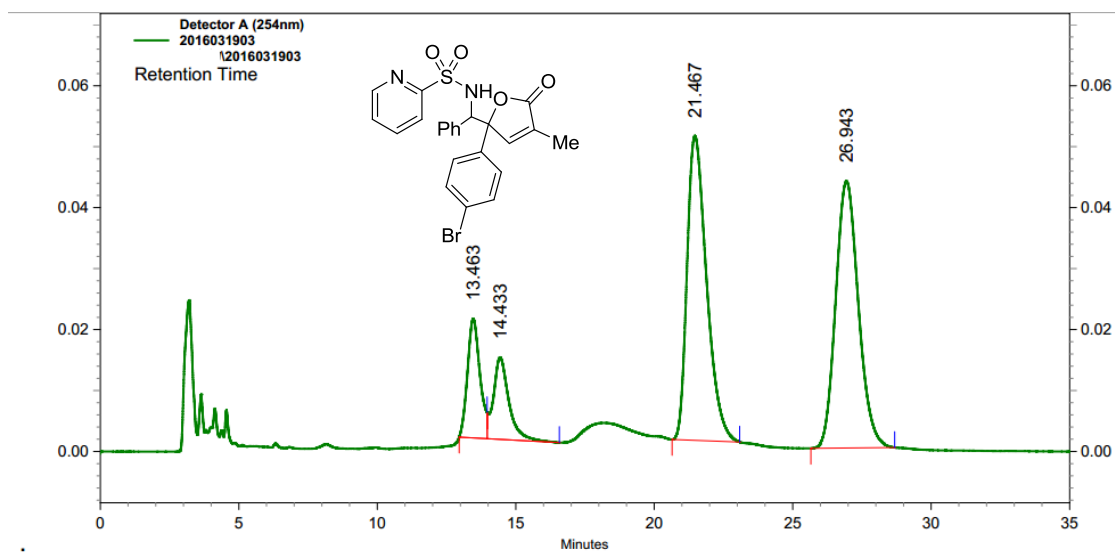
¹H NMR, ¹³C NMR and HPLC of 3r

1367-wzh-451.1.fid
WZH-451

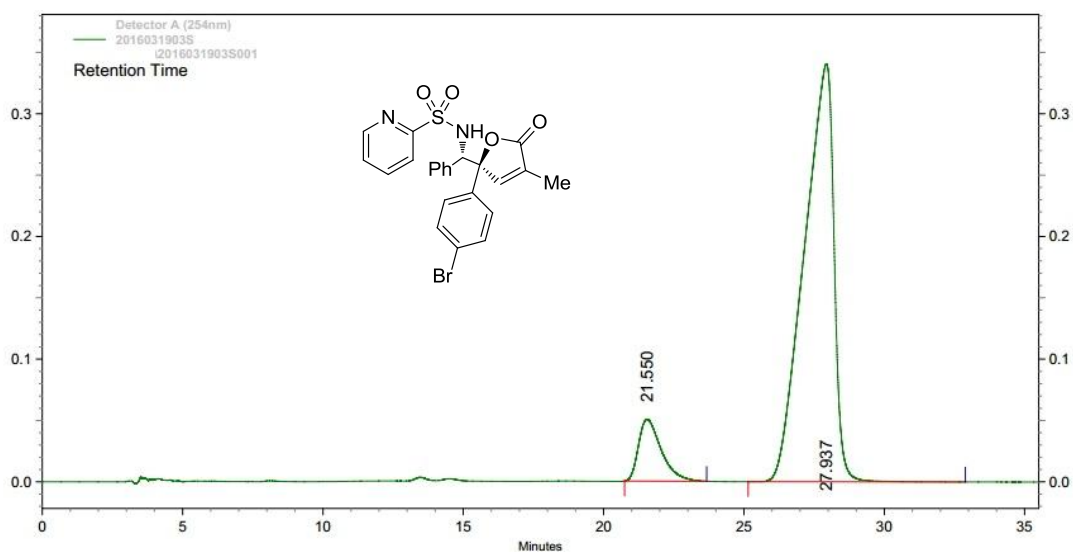


1367-wzh-451.2.fid
WZH-451 13C



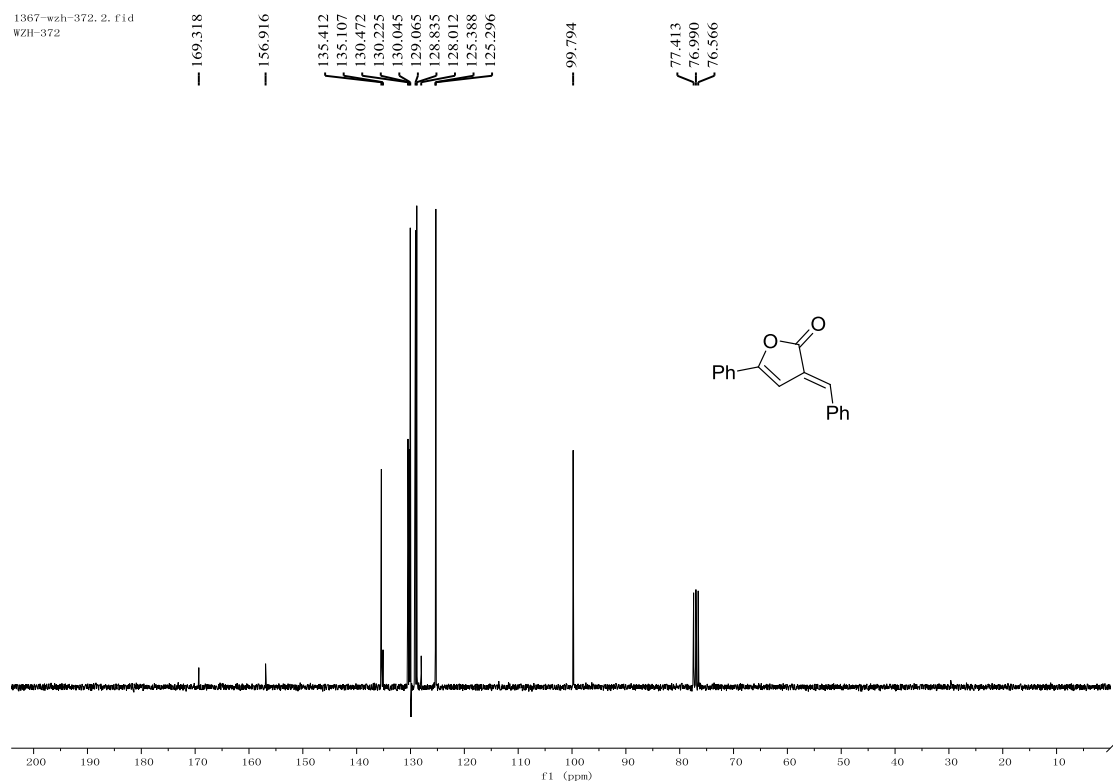
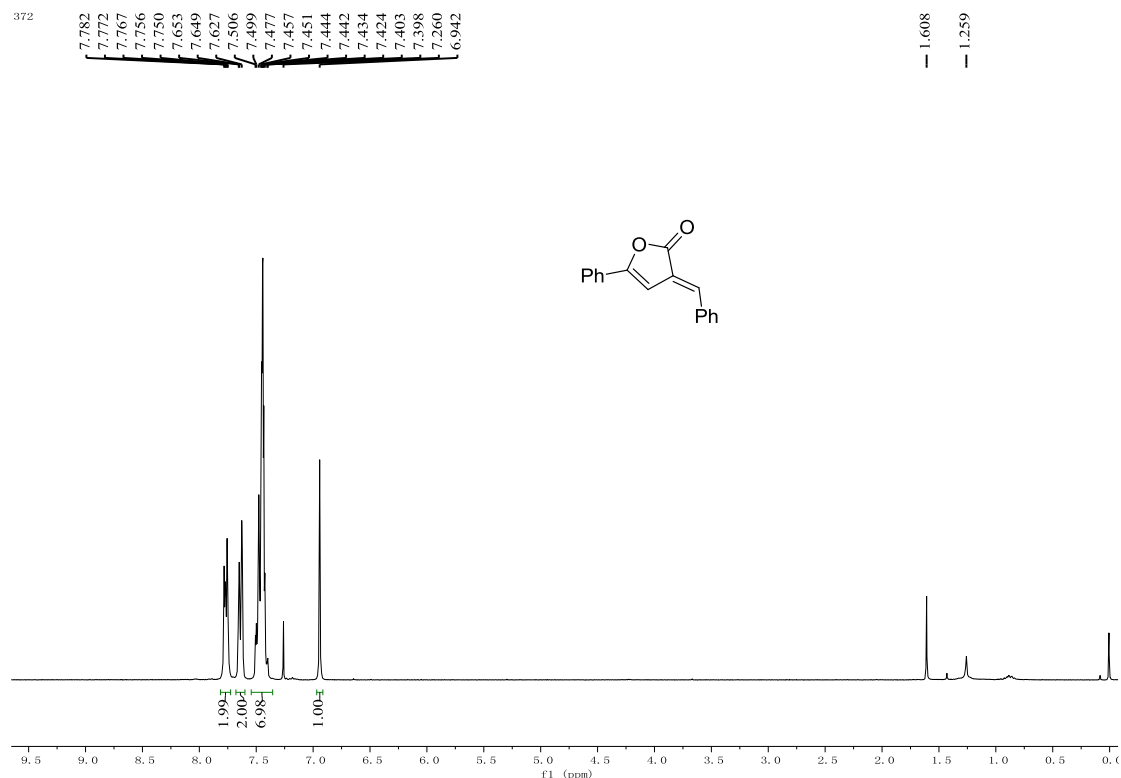


Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	13.463	19561	15.43	609355	10.02
2	14.433	13467	10.62	523798	8.61
3	21.467	49976	39.42	2450588	40.30
4	26.943	43769	34.53	2496676	41.06
Totals		126773	100.00	6080417	100.00

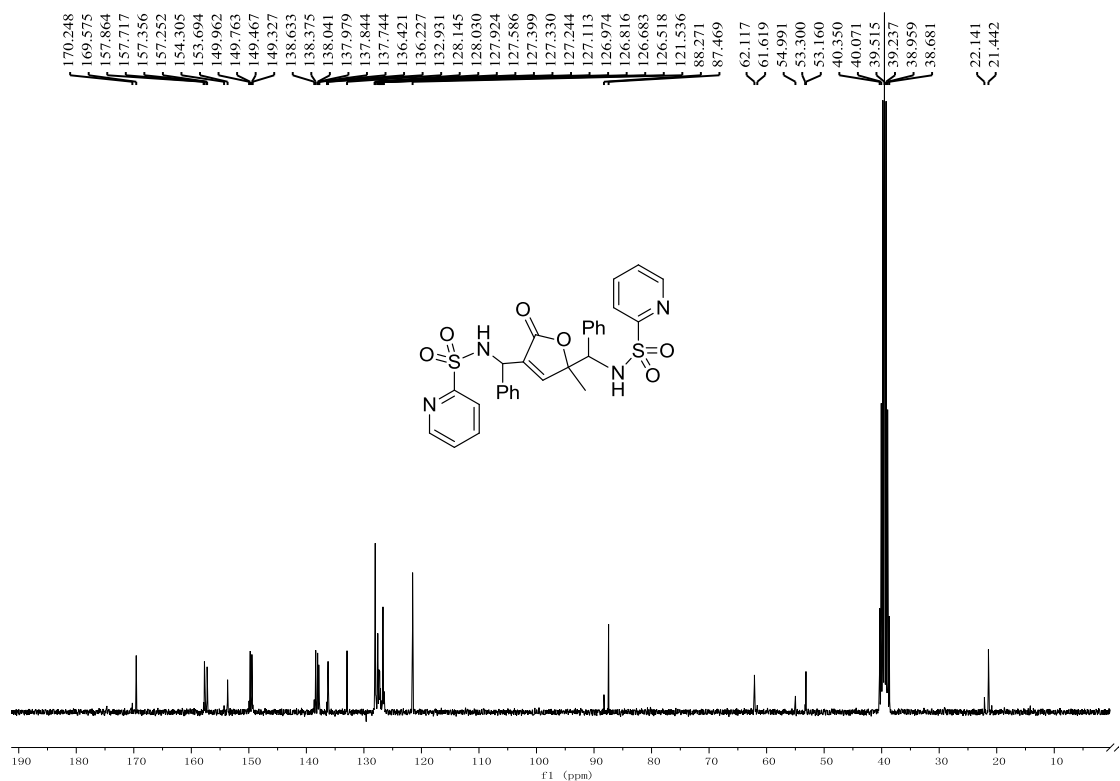
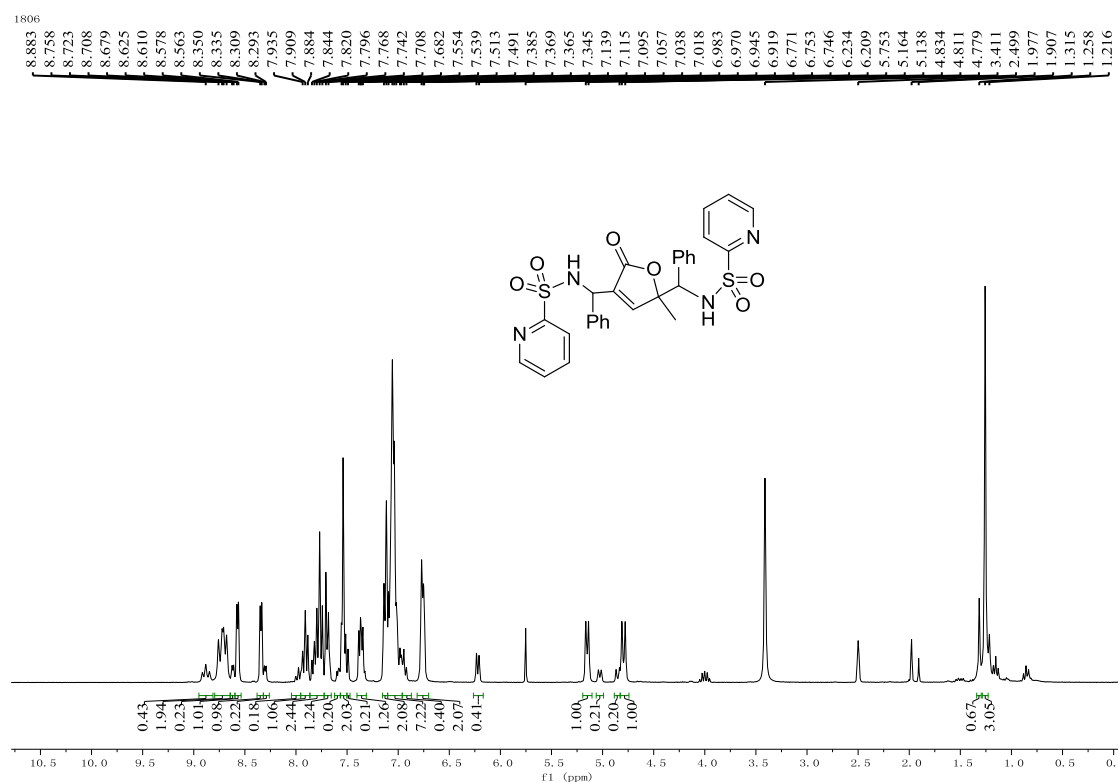


Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	21.550	50502	12.91	2844543	10.21
2	27.937	340681	87.09	25008244	89.79
Totals		391183	100.00	27852787	100.00

¹H NMR, ¹³C NMR of 5

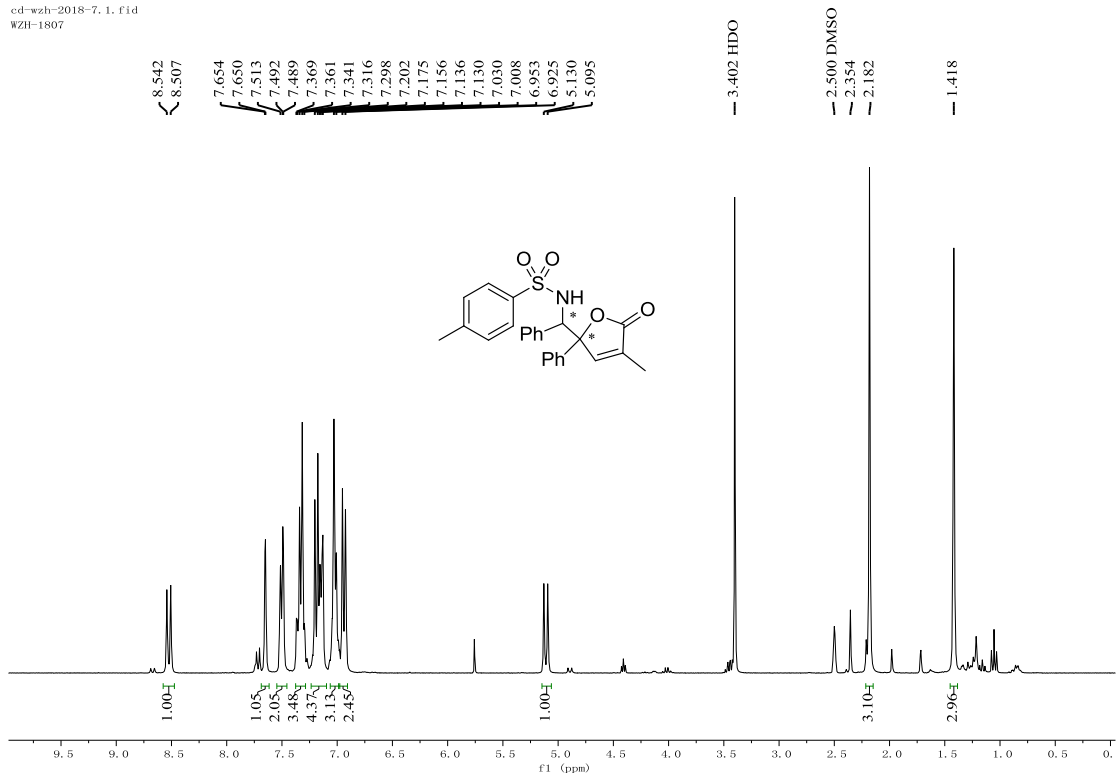


¹H NMR, ¹³C NMR of 6

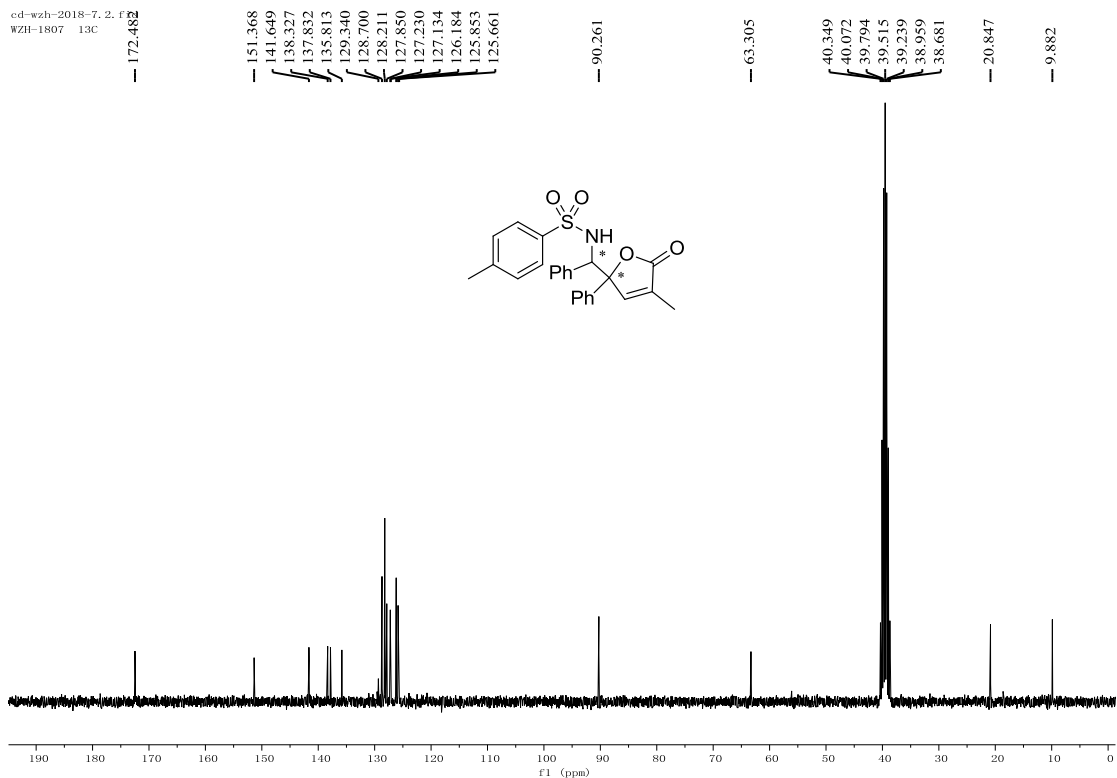


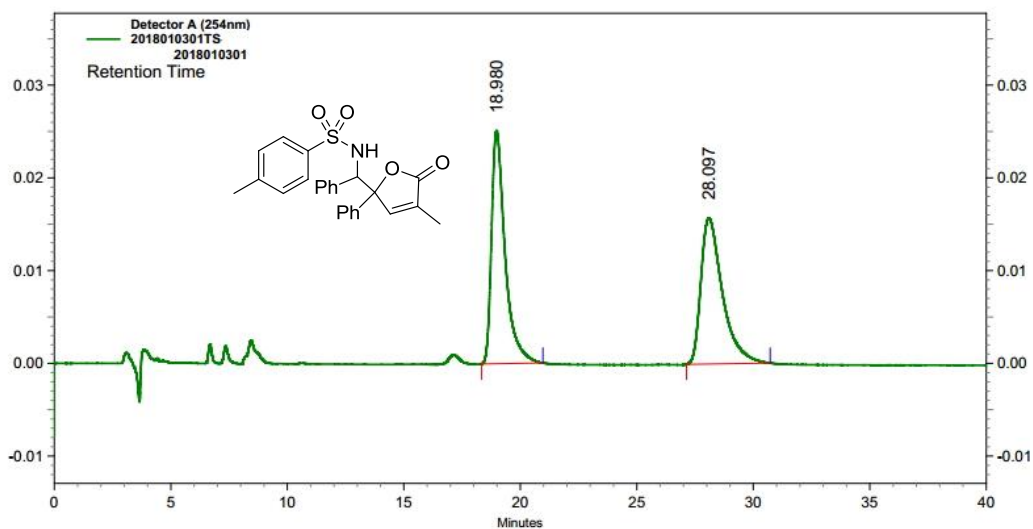
¹H NMR, ¹³C NMR and HPLC of 8

cd-wzh-2018-7.1.fid
WZH-1807



cd-wzh-2018-7.2.fid
WZH-1807 13C

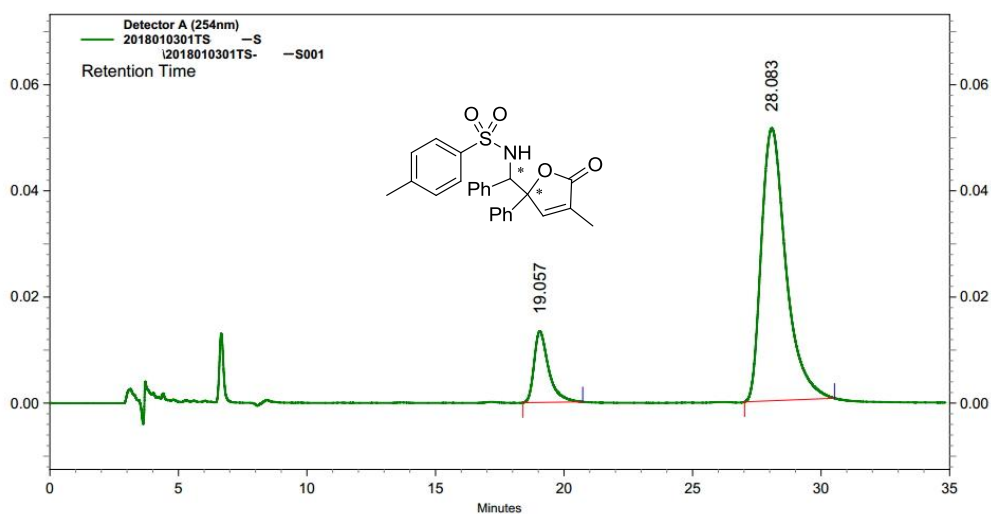




Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	18.980	25133	61.45	1017049	50.16
2	28.097	15766	38.55	1010714	49.84

Totals		40899	100.00	2027763	100.00
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Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	19.057	13415	20.71	527347	13.38
2	28.083	51351	79.29	3413615	86.62

Totals		64766	100.00	3940962	100.00
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