Asymmetric [3+2] Cycloaddition of Alkynes with Oxiranes by Selective C-C Bond Cleavage of Epoxide: Highly Efficient Synthesis of Chiral Furan Derivatives

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Contents:

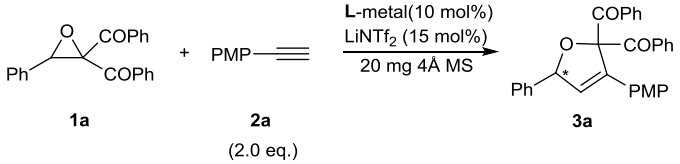
1. General remarks

^1H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26; DMSO, δ = 2.49). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration and assignment. ^{13}C NMR spectra were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0; DMSO, δ = 39.6). Melting points (m.p.) were measured on electrothermal digital melting point apparatus and were uncorrected. Enantiomeric excesses (ee) were determined by HPLC analysis using the corresponding commercial chiral column as stated in the experimental procedures at 23 °C. Optical rotations were reported as follows: $[\alpha]^T_D$ (c g/100 mL, in ethyl acetate). HRMS was recorded on a commercial apparatus (ESI Source). All catalytic reactions were run in dried glassware using standard techniques. CH_2Cl_2, DCE, $CHCl_2CHCl_2$was distilled over CaH_2. Toluene and THF were freshly distilled from sodium metal prior to use. All liquid alkynes were commercial aviable.

2. Typical procedure for catalytic asymmetric [3+2] cycloaddition of oxiranes and alkynes

N,*N*'-dioxide **L2** (7.7 mg, 0.11 mmol), $Ni(ClO_4)_2$ $6H_2O$ (3.7 mg, 0.01 mmol), $LiNTf_2$ (15 mol%) and 4 Å MS (20.0 mg) were stirred in 0.5 mL $CHCl_2CHCl_2$ at 35 °C under N2 atmosphere for 0.5 h, then 1-ethynyl-4-methoxybenzene (26.0 μL, 0.2 mmol) was added. After the temperature decreased to 30 ℃, (3-phenyloxirane-2,2-diyl)bis(phenylmethanone) **1a** (32.8 mg, 0.1 mmol) was added. The mixture was stirred at 30 ℃ for 24 h. The reaction mixture was purified by flash chromatography (petroleum ether: ethyl acetate = 5:1) on silica gel to afford the desired product.

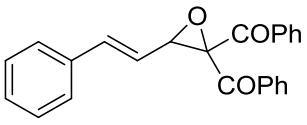
3. Investigation of the molar ratio of L6 to $Ni(ClO_4)_2$ $6H_2O$



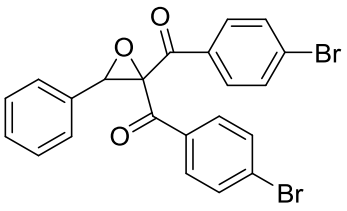
entry	ligand	**L**/metal	yield (%)[b]	*ee* (%)[c]
1	**L6**	1:1	93	88
2	**L6**	1.1:1	93	92
3	**L6**	1.2:1	70	92
4	**L6**	1.5:1	28	90

[a] Unless otherwise noted, all reactions were carried out with **L**-metal (10 mol%), **1a** (0.10 mmol), **2a** (0.20 mmol, 2.0 eq.), LiNTf2 (15 mol%), and 20 mg 4Å MS in $CHCl_2CHCl_2$ (0.5 mL) under N_2 at 30 °C for 24 h. [b] Isolated yield. [c] Determined by chiral HPLC analysis.

4. Characterization of the oxiranes[1]

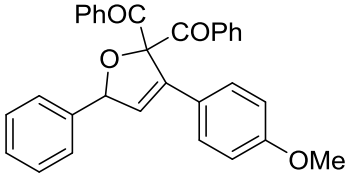


(E)-(3-styryloxirane-2,2-diyl)bis(phenylmethanone): Yellow solid; M.P.: 66-68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 – 8.02 (m, 4H), 7.60 – 7.51 (m, 2H), 7.49 – 7.40 (m, 4H), 7.00 (d, *J* = 15.9 Hz, 1H), 5.88 – 5.75 (m, 1H), 4.31 (dd, *J* = 8.5, 1.9 Hz, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 192.29, 192.10, 138.83, 135.34, 134.87, 134.46, 134.35, 129.93, 129.83, 128.83, 128.80, 128.67, 126.91, 120.79, 72.62, 63.10 ppm.



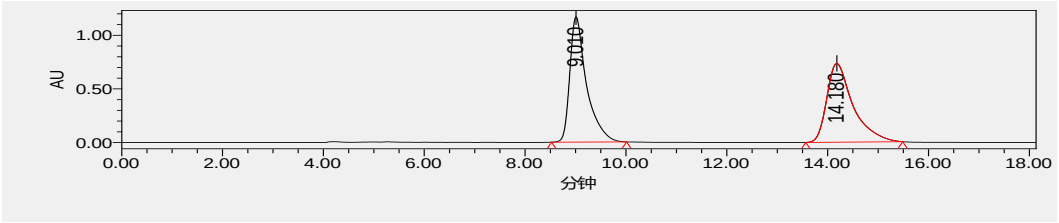
(3-phenyloxirane-2,2-diyl)bis((4-bromophenyl)methanone): White solid; M.P.: 58-60 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.98 (m, 2H), 7.85 – 7.75 (m, 2H), 7.69 – 7.58 (m, 2H), 7.54 – 7.46 (m, 2H), 7.32 – 7.24 (m, 5H), 4.64 (s, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 191.03, 190.02, 133.15, 132.70, 132.28, 132.04, 131.91, 131.57, 131.14, 130.34, 130.04, 129.19, 128.59, 126.20, 73.80, 62.68 ppm.

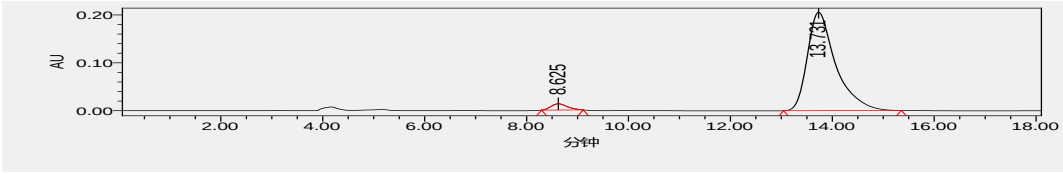
5. Characterization of the products



(3-(4-methoxyphenyl)-5-phenyl-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3a: [1] [2] White solid; M.P.: 56-58 °C; 93% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{20}_D$ = +268.37 (c 0.72, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.63 min, t_r (major) = 13.73 min, 93% ee; ^{1}H NMR (400 MHz, $CDCl_3$): 8.05 – 7.98 (m, 2H), 7.94 – 7.88 (m, 2H), 7.61 – 7.53 (m, 1H), 7.49 – 7.42 (m, 5H), 7.33 – 7.27 (m, 2H), 7.27 – 7.23 (m, 5H),

6.82 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 2 Hz, 1H), 6.17 (d, *J* = 2 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.58, 196.56, 159.77, 141.46, 138.90, 135.99, 134.79, 133.13, 132.73, 130.08, 129.83, 129.27, 128.61, 128.56, 128.42, 128.38, 128.14, 127.18, 124.39, 113.70, 101.63, 88.98, 55.24 ppm.





Retention Time	Area	% Area
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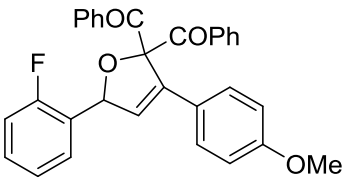
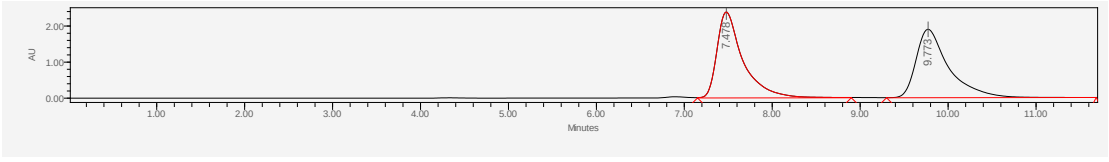
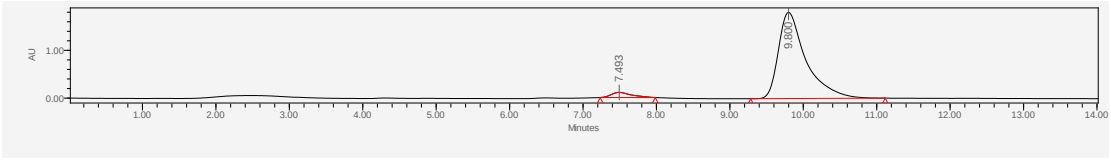
13.731	7928970	96.45

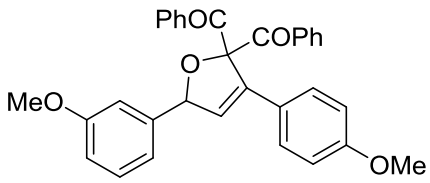


(5-(2-fluorophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3b: Viscous oil in 68% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{15}_D$ = +261.09 (c 0.59, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 7.49 min, t_r (major) = 9.80 min, 92% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.97 – 7.92 (m, 2H), 7.61 – 7.56 (m, 1H), 7.52 – 7.45 (m, 5H), 7.38 – 7.33 (m, 2H), 7.24 – 7.17 (m, 1H), 7.15 – 7.09 (m, 1H), 7.03 – 6.93 (m, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.62 (t, *J* = 1.6 Hz, 1H), 6.51 (d, *J* = 1.3 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.42, 196.22, 160.99, 159.78, 158.54, 141.30, 135.87, 134.68, 133.21, 132.84, 130.06, 129.80, 129.77, 129.68, 129.23, 128.58, 128.24, 127.94, 127.90, 127.05, 126.32, 126.19, 124.39, 124.36, 124.20, 115.33, 115.12, 113.68, 101.39, 82.65, 82.61, 55.23 ppm; EI-HRMS: Calcd for $C_{31}H_{23}FO_4$ $[M+Na]^+$ 501.1473, Found 501.1474.

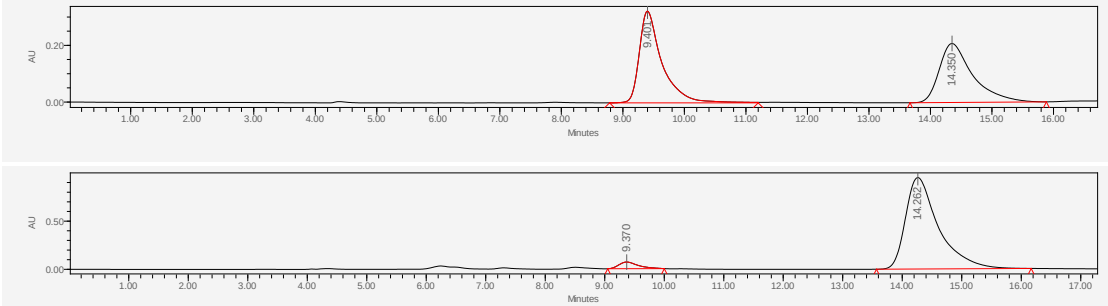




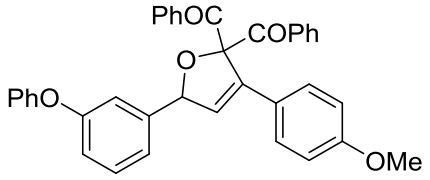
Retention Time	Area	% Area
7.493	2062473	4.20
9.800	46998560	95.80



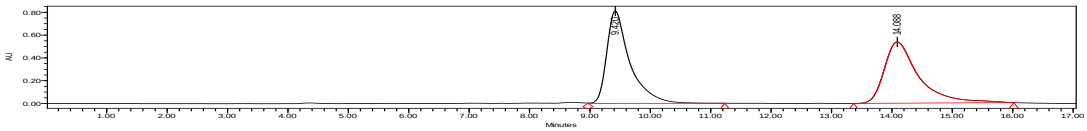
(5-(3-methoxyphenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3c: [2] White solid; M.P.: 38-40 °C; 99% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{21}_{D}$ = +293.43(c 0.97, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.37 min, t_r (major) = 14.26 min, 92% ee; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 – 7.99 (m, 2H), 7.97 – 7.91 (m, 2H), 7.61 – 7.54 (m, 1H), 7.49 – 7.42 (m, 5H), 7.35 – 7.29 (m, 2H), 7.20 – 7.13 (m, 1H), 6.86 – 6.80 (m, 3H), 6.79 – 6.74(m, 2H), 6.53 (d, *J* = 2 Hz, 1H), 6.17 (d, *J* = 1.6 Hz, 1H), 3.76 (s, 3H), 3.59 (s, 3H) ppm.; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.70, 196.31, 159.85, 159.77, 141.25, 140.58, 135.98, 134.59, 133.19, 132.79, 130.16, 129.81, 129.55, 129.23, 128.58, 128.27, 128.21, 124.37, 119.14, 114.80, 113.70, 111.53, 101.63, 88.91, 55.24, 55.06 ppm; EI-HRMS: Calcd for $C_{32}H_{26}O_5$ $[M+K]^+$ 529.1412, Found 529.1418.

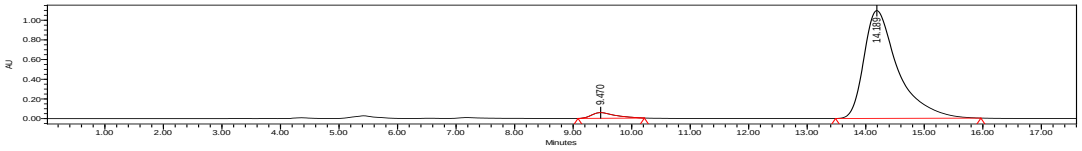


Retention Time	Area	% Area
9.370	1564403	4.12
14.262	36424859	95.88



(3-(4-methoxyphenyl)-5-(3-phenoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3d: [2] White solid; M.P.: 42-44 °C; 90% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{18}_{D}$ = +267.10 (c 0.99, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.47 min, t_r (major) = 14.19 min, 93% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.98 (m, 2H), 7.91 – 7.85 (m, 2H), 7.61 – 7.54 (m, 1H), 7.49 – 7.42 (m, 5H), 7.33 – 7.27 (m, 2H), 7.25 – 7.20 (m, 1H), 7.09 (t, *J* = 7.8 Hz, 1H); 6.99 (d, *J* = 8.0 Hz, 1H), 6.95 – 6.86 (m, 4H), 6.82 (d, *J* = 8.8 Hz,, 2H), 6.54 (d, *J* = 1.6 Hz, 1H), 6.14 (d, *J* = 1.6 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.38, 196.37, 159.81, 157.39, 156.97, 141.71, 141.01, 135.90, 134.68, 133.16, 132.75, 130.05, 130.01, 129.84, 129.76, 129.27, 128.56, 128.14, 127.86, 124.29, 123.32, 121.69, 118.84, 118.70, 117.52, 113.70, 101.58, 88.41, 55.24 ppm; EI-HRMS: Calcd for $C_{37}H_{28}O_5$ $[M+K]^+$ 591.1568, Found 591.1572.





Retention Time	Area	% Area
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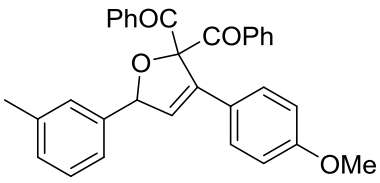
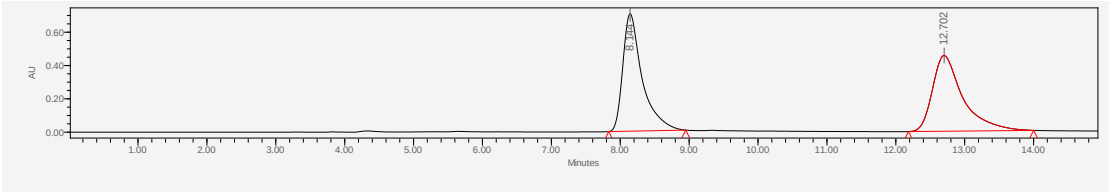
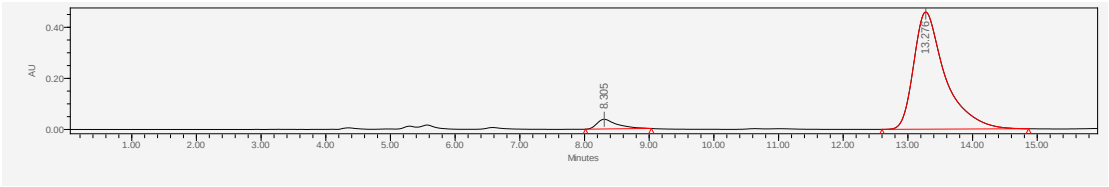
14.189	44165450	96.46

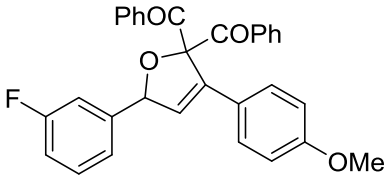


(3-(4-methoxyphenyl)-5-(m-tolyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3e: [2] Viscous oil in 97% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{21}_{D}$ = +244.81 (c 0.92, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.31 min, t_r (major) = 13.28 min, 90% ee; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 – 8.00 (m, 2H), 7.96 – 7.89 (m, 2H), 7.61 – 7.54 (m, 1H), 7.50 – 7.42 (m, 5H), 7.34 – 7.29 (m, 2H), 7.19 – 7.13 (m, 1H), 7.08 – 7.02 (m, 3H), 6.83 (d, *J* = 9.2 Hz, 2H), 6.54 (d, *J* = 2 Hz, 1H), 6.13 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H), 3.09 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.55, 196.75, 159.74, 141.35, 138.79, 138.27, 135.91, 134.86, 133.14, 132.73, 130.11, 129.80, 129.34, 129.14, 128.55, 128.50, 128.43, 128.14, 127.92, 124.45, 124.22, 113.70, 101.57, 88.96, 55.25, 21.39 ppm; EI-HRMS: Calcd for $C_{32}H_{26}O_4$ $[M+K]^+$ 497.1723, Found 497.1734.



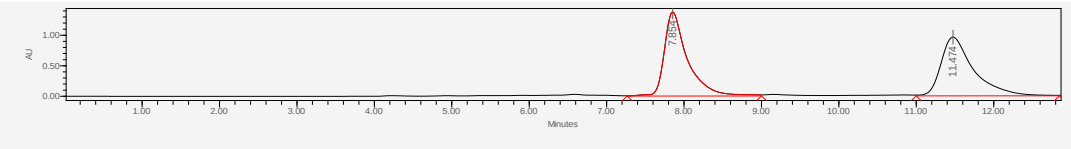


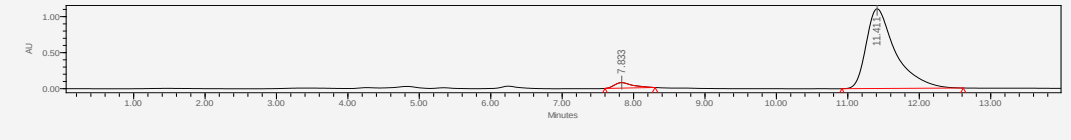
Retention Time	Area	% Area
8.305	834163	5.12
13.276	15457581	94.88



(5-(3-fluorophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3f: Viscous oil in 57% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{18}_{D}$ = +239.71 (c 0.61, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 7.83 min, t_r (major) = 11.41 min, 92% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.98 (m, 2H), 7.94 – 7.88 (m, 2H), 7.62 – 7.56 (m, 1H), 7.51 – 7.45 (m, 5H), 7.37 – 7.30 (m, 2H), 7.25 – 7.18 (m, 1H), 7.07 – 7.01 (m, 1H), 6.98 – 6.89 (m, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 2.0 Hz, 1H), 6.17 (d, *J* = 1.2 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.30, 196.50, 164.06, 161.61, 159.87, 141.87, 141.53, 141.47, 135.84, 134.66, 133.24, 132.90, 130.24, 130.16, 130.02, 129.86,

129.27, 128.60, 128.23, 127.57, 124.11, 122.54, 122.51, 115.36, 115.15, 114.12, 113.90, 113.73, 101.60, 88.13, 88.11, 55.25 ppm; EI-HRMS: Calcd for $C_{31}H_{23}FO_4$ $[M+K]^+$ 517.1212, Found 517.1217.





Retention Time	Area	% Area
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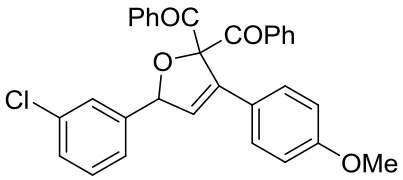
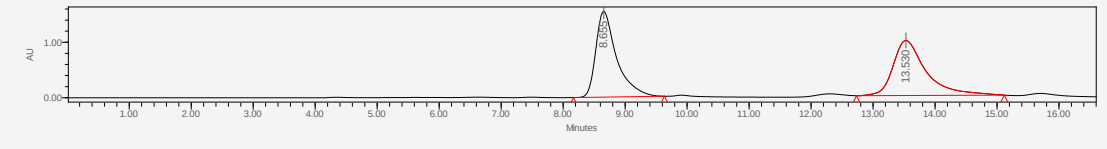
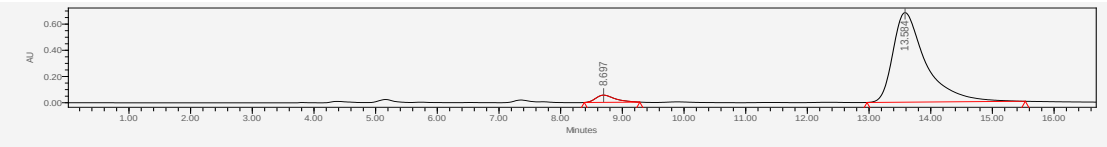
11.411	31428082	95.86

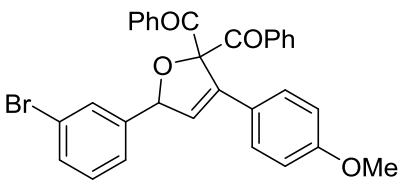


(5-(3-chlorophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3g: White solid; M.P.: 44-46 ^oC; 67% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{15}_D$ = +199.36 (c 0.62, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.70 min, t_r (major) = 13.58 min, 91% ee; ^1H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.97 (m, 2H), 7.91 – 7.85 (m, 2H), 7.60 – 7.55 (m, 1H), 7.50 – 7.44 (m, 5H), 7.35 – 7.30 (m, 2H), 7.23 – 7.16 (m, 3H), 7.14 – 7.09 (m, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 2.0 Hz, 1H), 6.12 (d, *J* = 1.6 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.12, 196.63, 159.89, 142.05, 140.93, 135.79, 134.73, 134.44, 133.20, 132.88, 129.98, 129.94, 129.84, 129.29, 128.57, 128.48, 128.21, 127.41, 127.23, 125.10, 124.10, 113.74, 101.59, 88.03, 55.25 ppm; EI-HRMS: Calcd for $C_{31}H_{23}{}^{34.9689}ClO_4$ $[M+Na]^+$ 517.1177, Found 517.1177; Calcd for $C_{31}H_{23}{}^{36.9659}ClO_4$ $[M+Na]^+$ 519.1147, Found 517.1166.



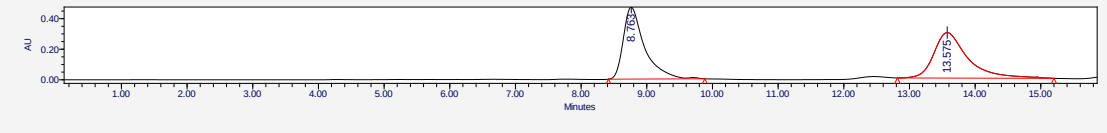


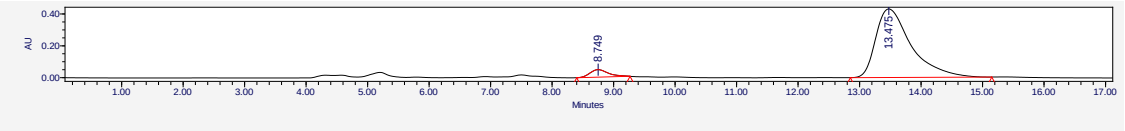
Retention Time	Area	% Area
8.697	1197214	4.54
13.584	25195099	95.46



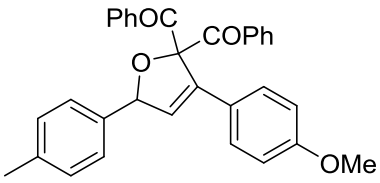
(5-(3-bromophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3h: [2] White solid; M.P.: 57-59 ^oC; 81% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{20}_D$ = +176.89 (c 0.87, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0

mL/min, 254 nm), t_r (minor) = 8.75 min, t_r (major) = 13.48 min, 89% ee; ^{1}H NMR (400 MHz, $CDCl_3$): 8.01–7.98 (m, 2H), 7.91–7.85 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49–7.44 (m, 5H), 7.40–7.31 (m, 4H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* = 1.6 Hz, 1H), 6.13 (d, *J* = 1.6 Hz, 1H), 3.08 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.09, 196.65, 159.89, 142.09, 141.17, 135.77, 134.73, 133.21, 132.90, 131.41, 130.24, 130.13, 129.98, 129.84, 129.30, 128.57, 128.23, 127.37, 125.57, 124.10, 122.61, 113.75, 101.58, 87.96, 55.26 ppm; EI-HRMS: Calcd for $C_{31}H_{23}{}^{78.9183}BrO_4$ $[M+K]^+$ 577.0411, Found 577.0408; Calcd for $C_{31}H_{23}{}^{80.9163}BrO_4$ $[M+K]^+$ 579.0391, Found 570.0398.

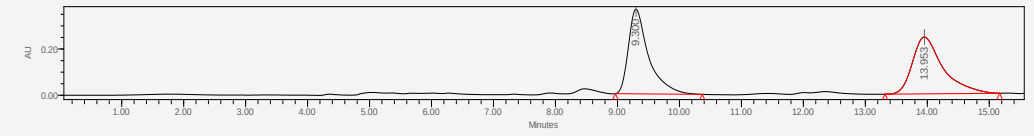


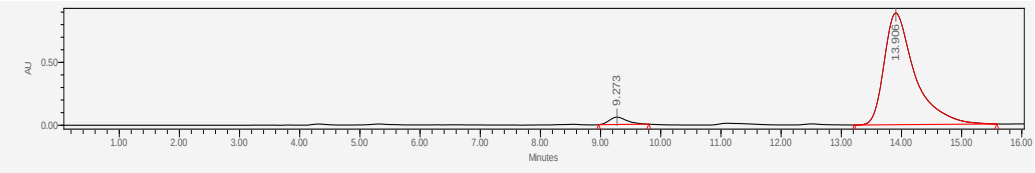


Retention Time	Area	% Area
8.749	978026	5.37
13.475	17222993	94.63



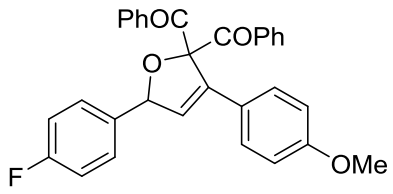
(3-(4-methoxyphenyl)-5-(p-tolyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3i: [2] White solid; M.P.: 50-52 ºC; 95% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{20}_D$ = +360.52 (c 0.93, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.27 min, t_r (major) = 13.91 min, 92% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.8 Hz, 2H), 7.94 (d, *J* = 7.8 Hz, 2H), 7.61 – 7.56 (m, 1H), 7.51 – 7.43 (m, 5H), 7.35 – 7.29 (m, 2H), 7.19 – 7.14 (m, 2H), 7.12 – 7.06 (m, 2H), 6.87 – 6.80 (d, *J* = 8.8 Hz, 2H), 6.54 (s, 1H), 6.16 (s, 1H), 3.78 (s, 3H), 2.30 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.72, 196.57, 159.74, 141.38, 138.30, 136.03, 135.92, 134.80, 133.11, 132.69, 130.11, 129.82, 129.29, 129.27, 128.59, 128.55, 128.13, 127.33, 124.47, 113.69, 101.56, 88.96, 55.24, 21.17 ppm; EI-HRMS: Calcd for $C_{32}H_{26}O_4$ $[M+K]^+$ 513.1463, Found 513.1463.



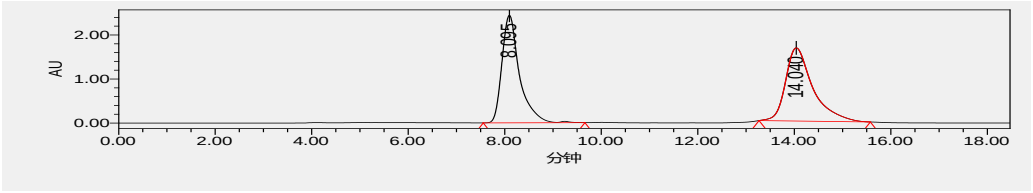


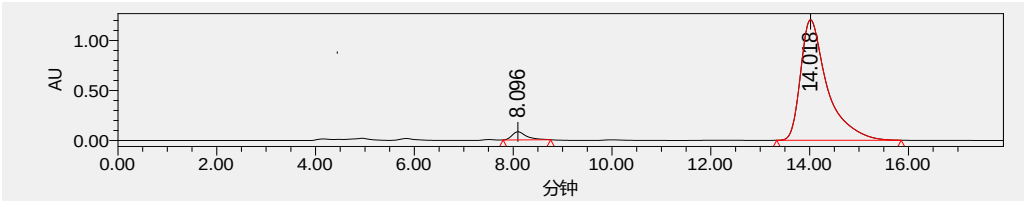
Retention Time	Area	% Area
9.273	1205586	3.69

13.906	31426598	96.31

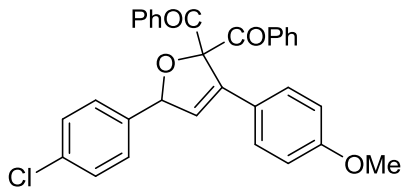


(5-(4-fluorophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3j: White solid; M.P.: 43-45 °C; 85% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{16}_{D}$ = +291.93 (c 0.82, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.10 min, t_r (major) = 14.02 min, 93% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 – 7.98 (m, 2H), 7.93 – 7.87 (m, 2H), 7.63 – 7.55 (m, 1H), 7.50 – 7.44 (m, 5H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.25 – 7.20 (m, 2H), 6.95 (t, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 9.2 Hz, 2H), 6.52 (d, *J* = 2.0 Hz, 1H), 6.16 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.49, 196.54, 163.93, 161.47, 159.82, 141.76, 135.91, 134.76, 134.73, 134.62, 133.20, 132.79, 130.00, 129.84, 129.20, 129.16, 129.08, 128.58, 128.16, 127.92, 124.15, 115.61, 115.40, 113.71, 101.49, 88.31, 55.25 ppm; EI-HRMS: Calcd for $C_{31}H_{23}FO_4$ $[M+Na]^+$ 501.1473, Found 501.1475.

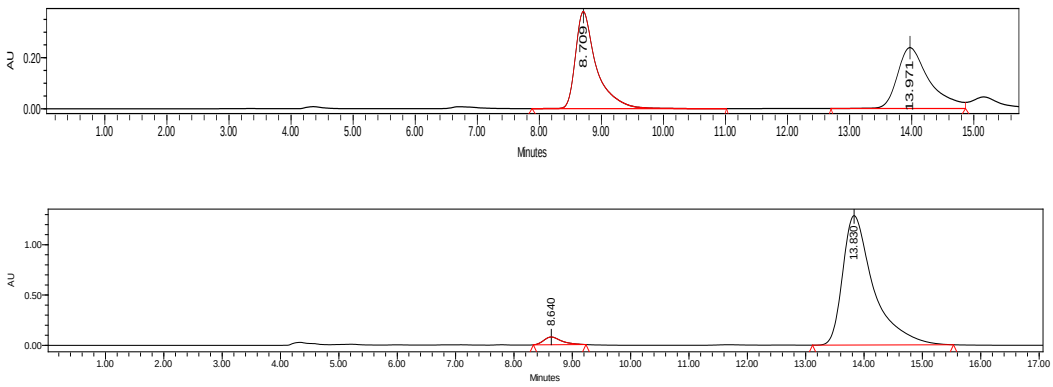




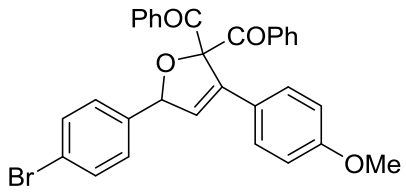
Retention Time	Area	% Area
8.096	1650723	3.53
14.018	45072595	96.47



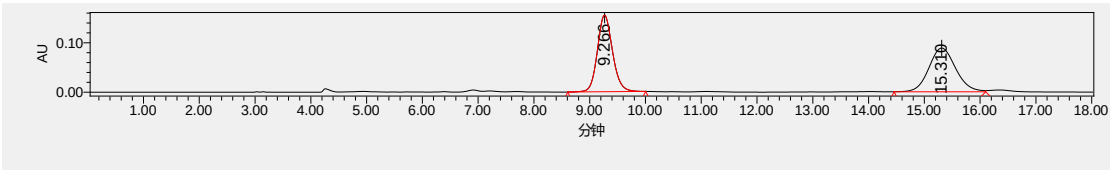
(5-(4-chlorophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3k: White solid; M.P.: 45-47 °C; 95% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{15}_{D}$ = +228.42 (c 0.94, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.64 min, t_r (major) = 13.83 min, 93% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.97 (m, 2H), 7.93 – 7.86 (m, 2H), 7.62 – 7.56 (m, 1H), 7.50 – 7.44 (m, 5H), 7.33 (t, *J* = 8.2 Hz, 2H), 7.25 – 7.15 (m, 4H), 6.83 (d, *J* = 9.2 Hz, 2H), 6.51 (d, *J* = 2.0 Hz, 1H), 6.15 (d, *J* = 1.6 Hz, 1H), 3.77 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.41, 196.44, 159.86, 141.89, 137.42, 135.90, 134.59, 134.25, 133.21, 132.83, 130.00, 129.84, 129.19, 128.77, 128.58, 128.19, 127.64, 124.09, 113.72, 101.54, 88.23, 55.25 ppm; EI-HRMS: $C_{31}H_{23}{}^{34.9689}ClO_4$ $[M+Na]^+$ 517.1177, Found 517.1185.

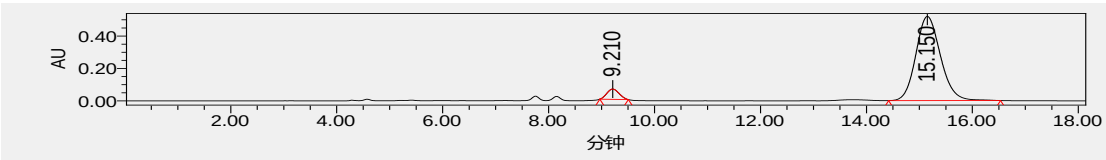


Retention Time	Area	% Area
8.640	1617608	3.31
13.830	47223543	96.69

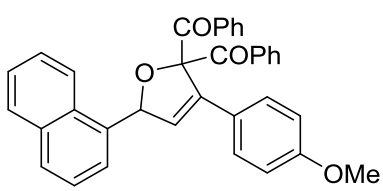


(5-(4-bromophenyl)-3-(4-methoxyphenyl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3l: [1][2] White solid; M.P.: 48-50 ºC; 90% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{11}_{D}$ = +251.71 (c 0.64, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.21 min, t_r (major) = 15.15 min, 90% ee; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 – 7.97 (m, 2H), 7.91 – 7.85 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49 – 7.43 (m, 5H), 7.40 – 7.36 (m, 2H), 7.35 – 7.29 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.50 (d, *J* = 2 Hz, 1H), 6.12 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.41, 196.44, 159.88, 141.93, 137.96, 135.92, 134.62, 133.22, 132.85, 131.74, 130.01, 129.86, 129.20, 128.88, 128.59, 128.21, 127.59, 124.10, 122.47, 113.74, 101.57, 88.27, 55.25 ppm; EI-HRMS: Calcd for $C_{31}H_{23}{}^{78.9183}BrO_4$ $[M+Na]^+$ 561.0672, Found 561.0675; Calcd for $C_{31}H_{23}{}^{80.9163}BrO_4$ $[M+Na]^+$ 563.0651, Found 563.0665.



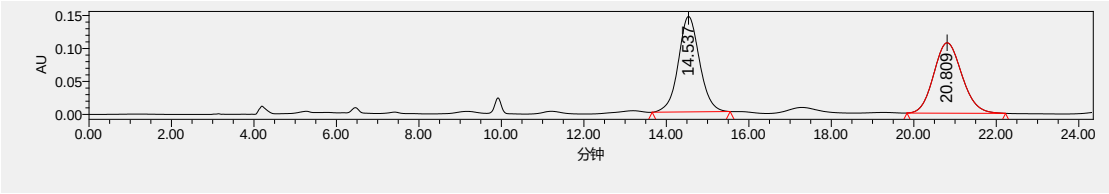


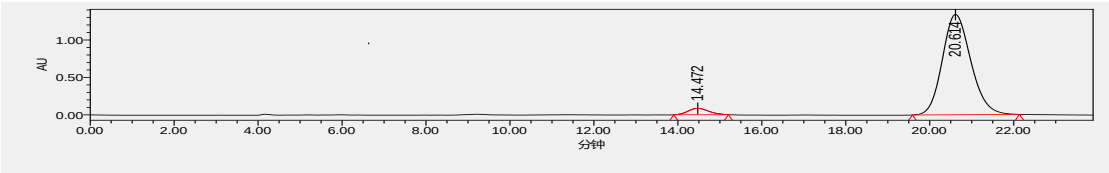
Retention Time	Area	% Area
9.210	1016873	5.71
15.150	16779688	94.29



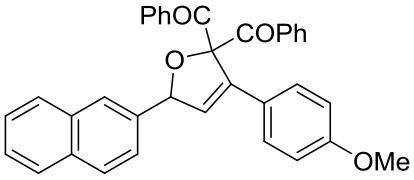
(3-(4-methoxyphenyl)-5-(naphthalen-1-yl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3m: [2] White solid; M.P.: 132-134 ºC; 95% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{11}_{D}$ =

+49.57 (c 0.70, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 10/90, 1.0 mL/min, 254 nm), t_r (minor) = 14.47 min, t_r (major) = 20.61 min, 91% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.09 (m, 2H), 8.07 – 8.02 (m, 1H), 7.90 – 7.84 (m, 2H), 7.81 – 7.75 (m, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.62 - 7.55 (m, 1H), 7.52 – 7.42 (m, 7H), 7.38 – 7.30 (m, 2H), 7.19 (t, *J* = 7.8 Hz, 2H), 6.87 (s, 1H), 6.85 – 6.79 (m, 3H), 3.74 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 197.84, 196.97, 159.77, 142.63, 135.65, 134.89, 134.20, 133.72, 133.14, 132.76, 130.97, 130.00, 129.74, 129.66, 128.93, 128.78, 128.52, 128.01, 127.20, 126.40, 125.78, 125.30, 124.58, 124.11, 123.36, 113.74, 101.26, 85.07, 55.25 ppm; EI-HRMS: Calcd for $C_{35}H_{26}O_4$ $[M+Na]^+$ 533.1723, Found 533.1733.

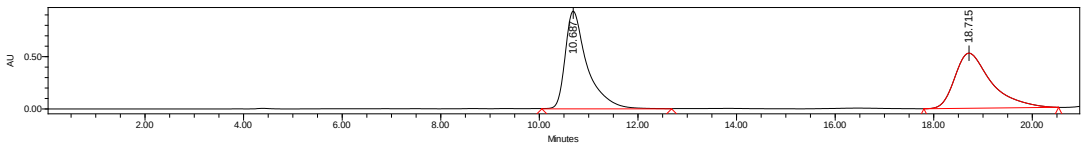


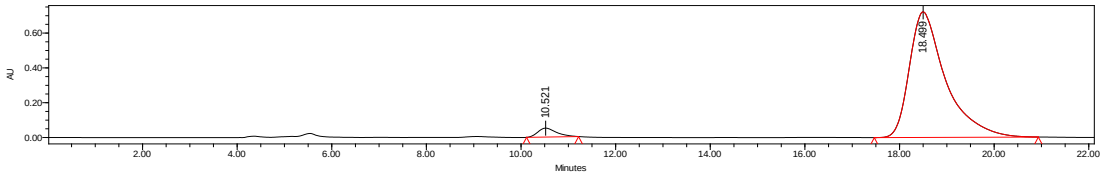


Retention Time	Area	% Area
14.472	2910205	4.41
20.614	63121765	95.59

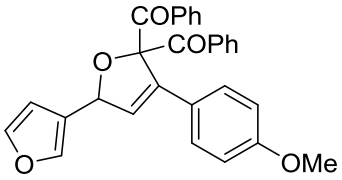


(3-(4-methoxyphenyl)-5-(naphthalen-2-yl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3n:[2] White solid; 43-45 ^{o}C; 94% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{18}_D$ = +297.66 (c 0.94, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 10.52 min, t_r (major) = 18.52 min, 93% ee; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 – 8.04 (m, 2H), 7.96 – 7.91 (m, 2H), 7.80 – 7.72 (m, 2H), 7.72 – 7.66 (m, 2H), 7.63 – 7.57 (m, 1H), 7.53 – 7.42 (m, 7H), 7.36 (dd, *J* = 8.8, 1.8 Hz, 1H), 7.32 – 7.27 (m, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 6.62 (d, *J* = 2 Hz, 1H), 6.34 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.57, 196.69, 159.82, 141.66, 136.27, 135.99, 134.82, 133.24, 133.18, 133.11, 132.75, 130.10, 129.86, 129.32, 128.59, 128.53, 128.16, 128.14, 128.09, 127.67, 126.35, 126.29, 126.26, 124.79, 124.41, 113.74, 101.69, 89.13, 55.26 ppm; EI-HRMS: Calcd for $C_{35}H_{26}O_4$ $[M+K]^+$ 549.1463, Found 549.1472.

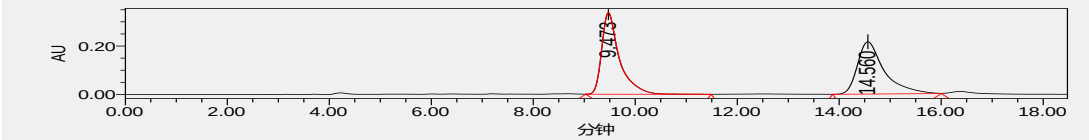


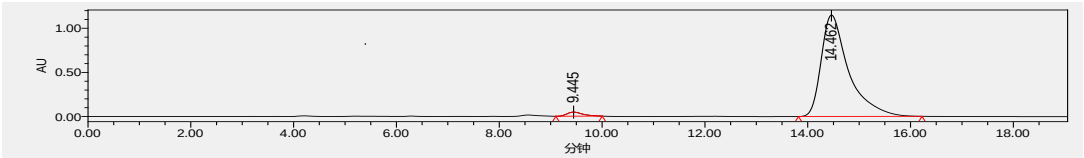


Retention Time	Area	% Area
10.521	1405811	3.54
18.499	38291172	96.46

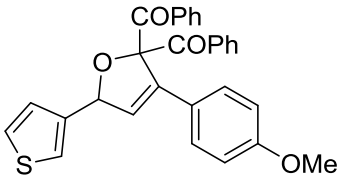


(4-(4-methoxyphenyl)-2,5-dihydro-[2,3'-bifuran]-5,5-diyl)bis(phenylmethanone) 3o: [2] Viscous oil in 81% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{15}_{D}$ = +234.43 (c 0.73, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.45 min, t_r (major) = 14.46 min, 95% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.96 (m, 2H), 7.94 – 7.89 (m, 2H), 7.60 – 7.54 (m, 1H), 7.49 – 7.42 (m, 5H), 7.36 – 7.31 (m, 3H), 7.28 (t, *J* = 1.6 Hz, 1H), 6.82 (d, *J* = 9.2 Hz, 2H), 6.47 (d, *J* = 2 Hz, 1H), 6.25 (d, *J* = 1.2 Hz, 1H), 6.15 (d, *J* = 2 Hz, 1H), 3.78 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.17, 196.53, 159.80, 143.63, 141.93, 140.51, 135.71, 134.64, 133.19, 132.76, 130.03, 129.79, 129.33, 128.51, 128.14, 127.20, 124.28, 124.16, 113.71, 109.38, 101.07, 80.99, 55.24 ppm; EI-HRMS: Calcd for $C_{29}H_{22}O_5$ $[M+Na]^+$ 473.1359, Found 473.1367.



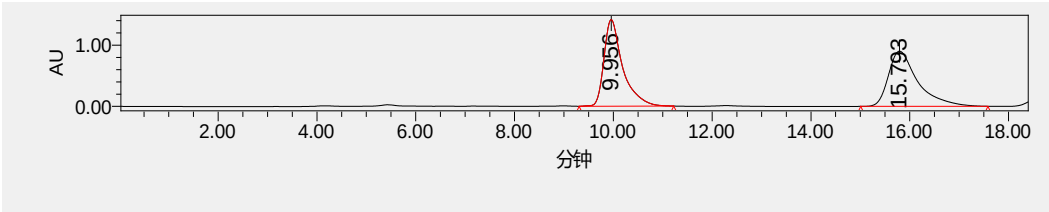


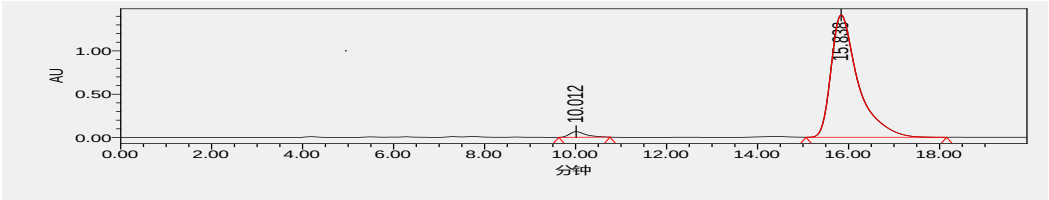
Retention Time	Area	% Area
9.445	1000405	2.30
14.462	42567568	97.70



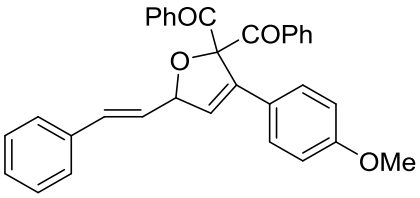
(3-(4-methoxyphenyl)-5-(thiophen-3-yl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3p: [2] White solid; M.P.: 40-42 °C; 90% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{15}_{D}$ = +265.55 (c 0.84, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 10.01 min, t_r (major) = 15.84 min, 95% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 2H), 7.94 – 7.88 (m, 2H), 7.60 – 7.54 (m, 1H), 7.49 – 7.43 (m, 5H), 7.34 – 7.29 (m, 2H), 7.22 – 7.19 (m, 1H), 7.16 – 7.12 (m, 1H), 6.95 (dd, *J* = 5.2, 1.2 Hz, 1H) 6.83 (d, *J* = 8.8 Hz, 2H), 6.65 (d, *J* = 1.6 Hz, 1H), 6.25 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.30, 196.56, 159.78,

141.60, 140.28, 135.76, 134.67, 133.19, 132.77, 130.05, 129.81, 129.34, 128.54, 128.15, 127.64, 126.56, 126.48, 124.31, 123.28, 113.70, 101.26, 84.40, 55.25 ppm; EI-HRMS: Calcd for $C_{29}H_{22}O_4S$ $[M+Na]^+$ 489.1131, Found 489.1126.

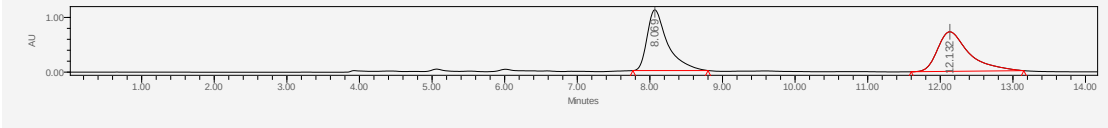


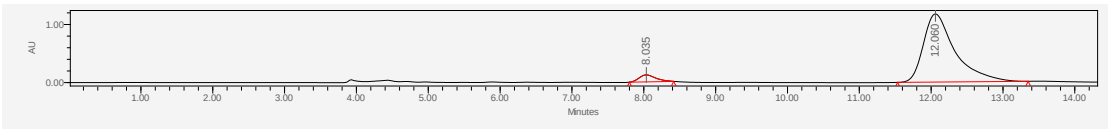


Retention Time	Area	% Area
10.012	1614225	2.70
15.838	58188529	97.30

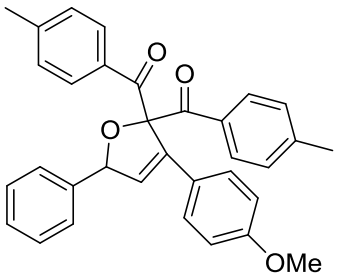


(E)-(3-(4-methoxyphenyl)-5-styryl-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3q: Yellow solid; M.P.: 38-40 ºC; 97% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{11}_D$ = +214.83 (c 0.94, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.04 min, t_r (major) = 12.06 min, 90% ee; ^{1}H NMR (400 MHz, DMSO) δ 7.97 – 7.85 (m, 4H), 7.71 – 7.66 (m, 1H), 7.65 – 7.61 (m, 1H), 7.58 – 7.49 (m, 4H), 7.46 – 7.41 (m, 2H), 7.33 – 7.21 (m, 5H), 6.93 – 6.83 (m, 3H), 6.63 – 6.53 (m, 1H), 6.24 - 6.11 (m, 1H), 5.88 (d, *J* = 6.8 Hz, 1H), 3.73 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO) δ = 197.91, 197.06, 159.75, 141.67, 135.98, 135.52, 134.96, 133.15, 132.96, 132.86, 130.03, 129.73, 129.51, 128.59, 128.47, 128.23, 128.16, 127.39, 126.74, 126.51, 124.39, 113.70, 101.07, 88.05, 55.24 ppm; EI-HRMS: Calcd for $C_{33}H_{26}O_4$ $[M+Na]^+$ 509.1723, Found 509.1730.

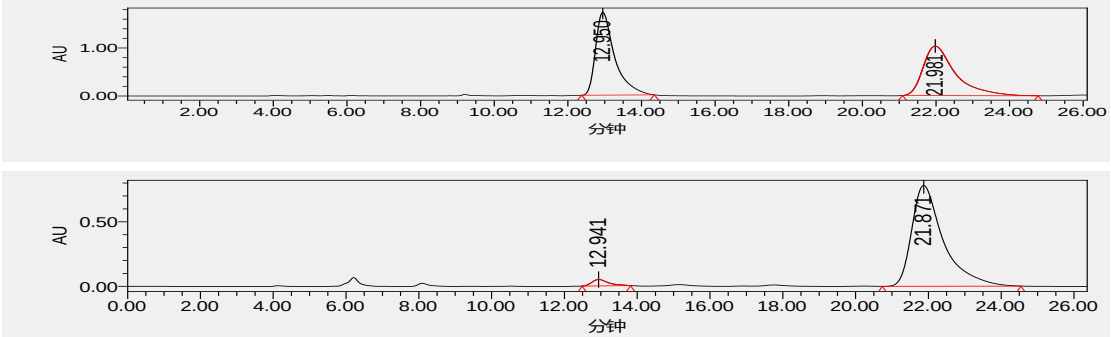




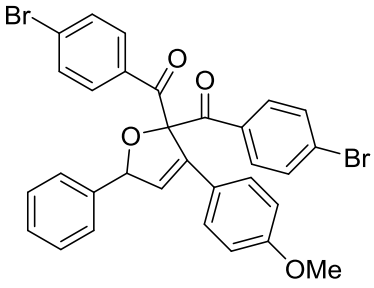
Retention Time	Area	% Area
8.169	3280247	4.98
12.343	62558130	95.02



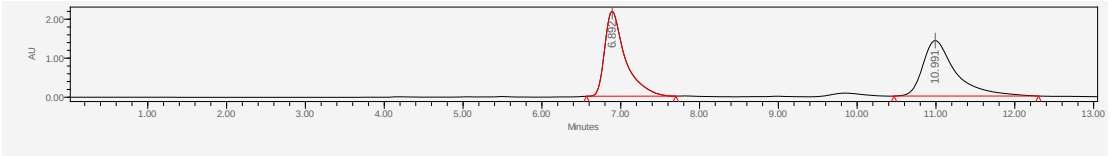
(3-(4-methoxyphenyl)-5-phenyl-2,5-dihydrofuran-2,2-diyl)bis(p-tolylmethanone) 3r: White solid; M.P.: 52-54 °C; 82% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{16}_{D}$ = +293.61 (c 0.80, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 8.04 min, t_r (major) = 12.06 min, 94% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.29 – 7.25 (m, 5H), 7.25 – 7.23 (m, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* =2.0 Hz, 1H), 6.17 (d, *J* = 2.0 Hz, 1H), 3.75 (s, 3H), 2.41 (s, 3H), 2.32 (s, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.16, 196.14, 159.65, 144.00, 143.50, 141.72, 139.03, 133.29, 132.12, 130.26, 129.85, 129.43, 129.23, 128.84, 128.58, 128.33, 128.19, 127.17, 124.55, 113.61, 101.64, 88.83, 55.23, 21.78, 21.67 ppm; EI-HRMS: Calcd for $C_{33}H_{28}O_4$ $[M+Na]^+$ 511.1880, Found 511.1887.

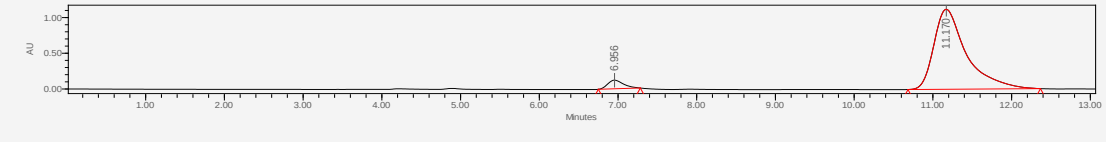


Retention Time	Area	% Area
12.941	1578987	3.24
21.871	47194512	96.76

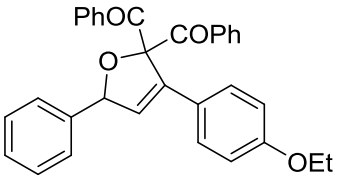


(3-(4-methoxyphenyl)-5-phenyl-2,5-dihydrofuran-2,2-diyl)bis((4-bromophenyl)methanone) 3s: White solid; M.P.: 62-64 °C; 98% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{11}_{D}$ = +235.41 (c 1.21, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 6.96 min, t_r (major) = 11.17 min, 90% ee; ^{1}H NMR (400 MHz, DMSO) δ 7.93 – 7.86 (m, 2H), 7.82 – 7.77 (m, 2H), 7.76 – 7.71 (m, 2H), 7.68 – 7.62 (m, 2H), 7.43 (d, *J* = 8.8 Hz, 2H), 7.33 – 7.25 (m, 3H), 7.25 – 7.20 (m, 2H), 7.05 – 6.99 (m, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.38 (s, 1H), 3.73 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO) δ = 197.96, 195.07, 159.27, 138.98, 138.80, 133.66, 132.79, 132.19, 131.72, 131.35, 130.78, 129.39, 129.36, 128.56, 128.41, 128.21, 127.45, 126.87, 123.71, 113.61, 100.59, 88.81, 55.08 ppm; EI-HRMS: Calcd for $C_{31}H_{22}{}^{78.9183}Br^{80.9163}BrO_4$ $[M+Na]^+$ 640.9764, Found 640.9757; Calcd for $C_{31}H_{22}{}^{78.9183}Br_2O_4$ $[M+Na]^+$ 638.9777, Found 638.9781.

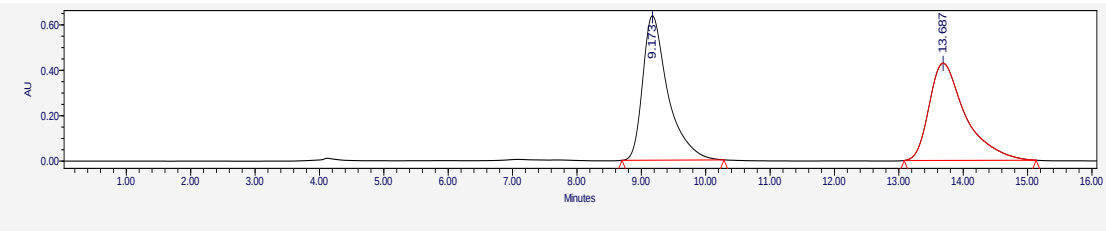


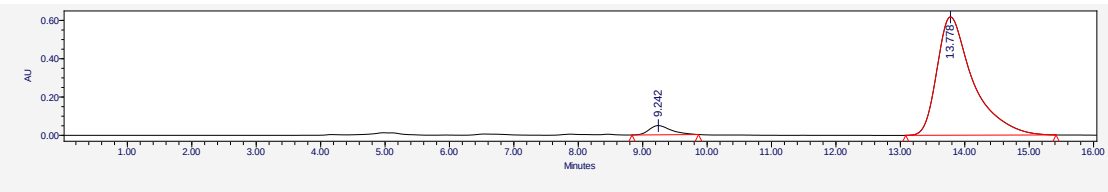


Retention Time	Area	% Area
6.842	3602505	5.24
10.847	65205560	94.76

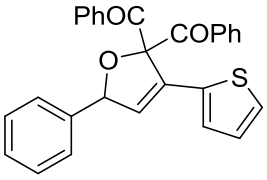


(3-(4-ethoxyphenyl)-5-phenyl-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3t: White solid; M.P.: 36-38 ^{o}C; 93% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{20}_{D}$ = +300.46 (c 0.88, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 9.24 min, t_r (major) = 13.78 min, 91% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.97 (m, 2H), 7.95 – 7.88 (m, 2H), 7.60 – 7.52 (m, 1H), 7.48 – 7.41 (m, 5H), 7.32 – 7.27 (m, 2H), 7.27 – 7.22 (m, 5H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 1.6 Hz, 1H), 6.17 (d, *J* = 2.0 Hz, 1H), 3.98 (q, *J* = 7.0 Hz, 2H), 1.37 (t, *J* = 7.0 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.63, 196.58, 159.17, 141.48, 138.92, 135.99, 134.78, 133.14, 132.74, 130.09, 129.82, 129.28, 128.61, 128.56, 128.42, 128.25, 128.15, 127.19, 124.17, 114.20, 101.61, 88.99, 63.39, 14.82 ppm; EI-HRMS: Calcd for $C_{32}H_{26}O_4$ $[M+Na]^+$ 497.1723, Found 497.1734.



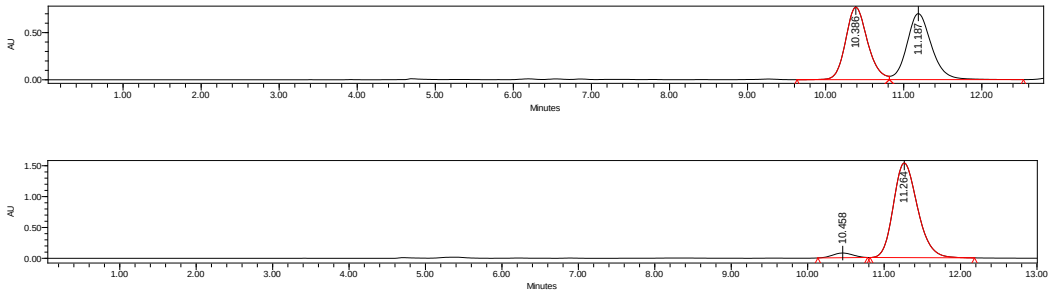


Retention Time	Area	% Area
9.242	1146017	4.58
13.778	23896275	95.42

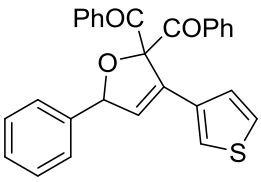


(5-phenyl-3-(thiophen-2-yl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3u: White solid; M.P.: 35-37 ^{o}C; 90% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{16}_{D}$ = +386.07 (c 0.79, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 10.46 min, t_r (major) = 11.26 min, 92% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.98 (m, 2H), 7.97 – 7.89 (m, 2H), 7.63 – 7.55 (m, 1H), 7.52 – 7.42 (m, 3H), 7.32 (t, *J* = 7.8, 2H), 7.27 – 7.20 (m, 6H), 7.16 (d, *J* = 3.6 Hz, 1H), 6.99 – 6.92 (m, 1H), 6.55 (d, *J* = 2.0 Hz, 1H), 6.19 (d, *J* = 2.0 Hz, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.40, 195.79, 138.58, 135.87, 135.27, 134.37,

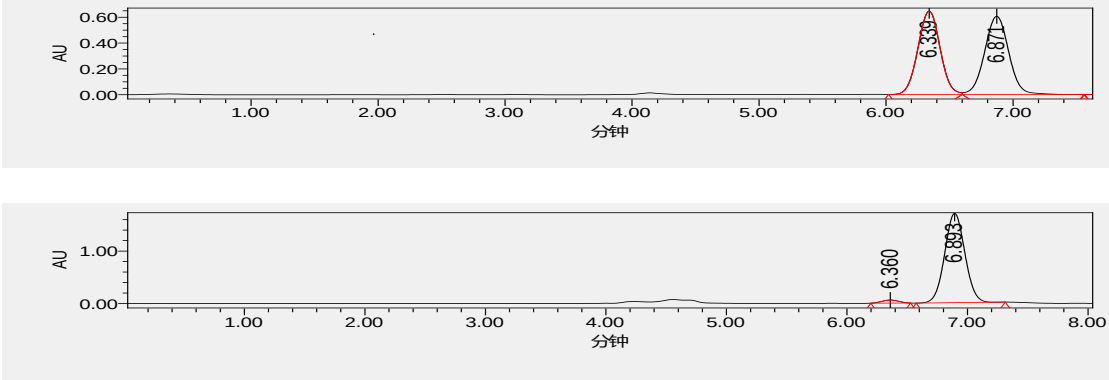
134.08, 133.26, 132.87, 130.11, 129.21, 128.67, 128.63, 128.55, 128.22, 127.94, 127.12, 125.94, 101.52, 89.21 ppm; EI-HRMS: Calcd for $C_{28}H_{20}O_3S$ $[M+Na]^+$ 459.1025, Found 459.1036.



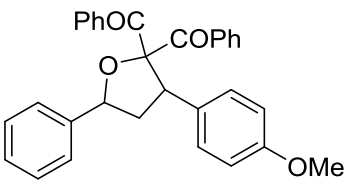
Retention Time	Area	% Area
10.458	1397310	3.99
11.264	33620711	96.01



(5-phenyl-3-(thiophen-3-yl)-2,5-dihydrofuran-2,2-diyl)bis(phenylmethanone) 3v: White solid; M.P.: 48-50 °C; 85% yield (petroleum ether : EtOAc = 5: 1); $[\alpha]^{16}_D$ = +377.53 (c 0.67, CH_2Cl_2); the ee was determined by HPLC analysis using a chiral IC column (*i*PrOH/hexane = 20/80, 1.0 mL/min, 254 nm), t_r (minor) = 6.36 min, t_r (major) = 6.89 min, 95% ee; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.97 (m, 2H), 7.96 – 7.88 (m, 2H), 7.61 – 7.56 (m, 1H), 7.49 – 7.42 (m, 4H), 7.33 – 7.26 (m, 3H), 7.25 – 7.18 (m, 6H), 6.55 (d, *J* = 2.0 Hz, 1H), 6.20 (d, *J* = 1.6 Hz, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.90, 196.09, 138.79, 136.39, 135.96, 134.47, 133.24, 132.82, 132.34, 130.11, 129.21, 128.65, 128.61, 128.60, 128.50, 128.21, 127.35, 127.14, 125.42, 125.11, 101.45, 89.15 ppm; EI-HRMS: Calcd for $C_{28}H_{20}O_3S$ $[M+Na]^+$ 459.1025, Found 459.1028.

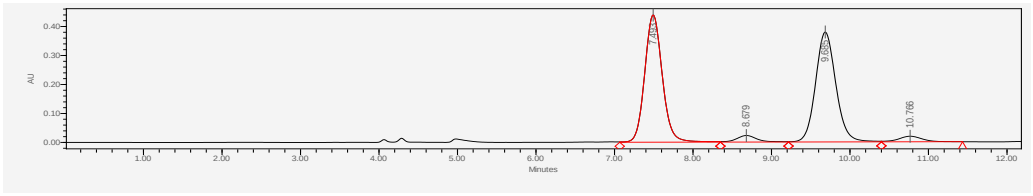


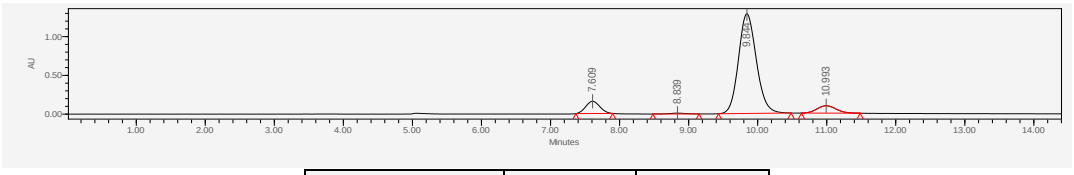
Retention Time	Area	% Area
6.360	564813	2.77
6.893	19857633	97.23



(3-(4-methoxyphenyl)-5-phenyltetrahydrofuran-2,2-diyl)bis(phenylmethanone) 4a: White soild, M.P.: 33-35 °C; 96% yield (petroleum ether : EtOAc = 10: 1); $[\alpha]^{20}_D$ = -165.84 (c 0.85 CH_2Cl_2); The ee was 82% ee, dr = 93:7; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 8.06 (m, 2H), 7.70 – 7.65 (m, 2H), 7.63 – 7.57 (m, 2H), 7.47 – 7.35

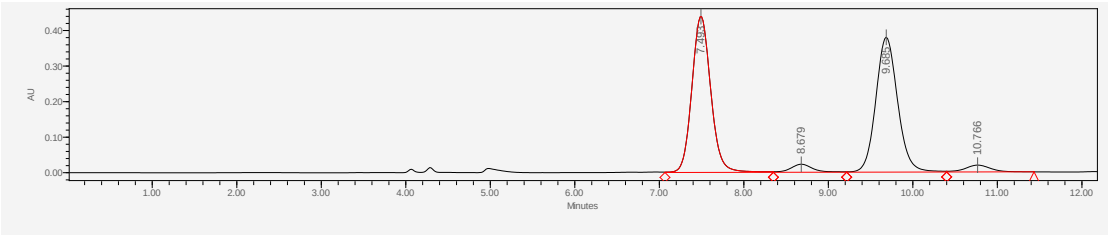
(m, 4H), 7.33 – 7.27 (m, 3H), 7.17 – 7.11 (m, 4H), 6.62 – 6.55 (m, 2H), 5.11 – 4.97 (m, 2H),, 3.65 (s, 3H), 2.93 – 2.81 (m, 1H), 2.55 – 2.42 (m, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 197.35, 195.40, 158.53, 140.41, 136.14, 134.84, 133.18, 132.57, 130.52, 130.24, 130.09, 129.21, 128.54, 128.35, 127.90, 127.81, 126.46, 113.51, 97.86, 81.16, 55.16, 49.46, 42.11 ppm; EI-HRMS: Calcd for $C_{31}H_{26}O_4$ $[M+Na]^+$ 485.1723, Found 485.1728.

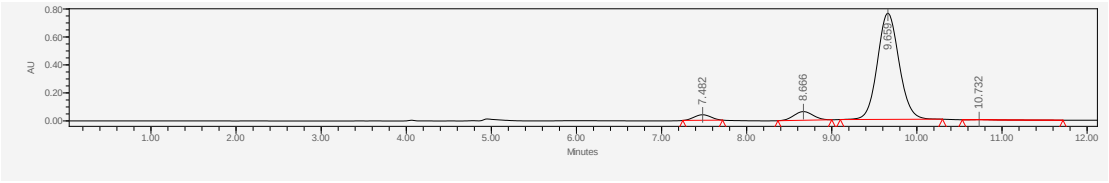




Retention Time	Area	% Area
7.609	2232874	8.16
8.839	171999	0.63
9.844	23129122	84.50
10.993	1837936	6.71

The hydrogeneration of **3a**:



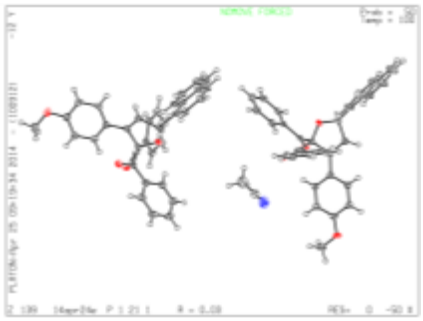


Retention Time	Area	% Area
7.482	546670	3.66
8.666	966862	6.48
9.659	13369508	89.60
10.732	38852	0.26

6. Reference

1) W. L. Chen, L. L. Lin, Y. F. Cai, Y. Xia, W. D. Cao, X. H. Liu, X. M. Feng, *Chem. Commun.* 2014, **50**, 2161.
2) Some ^{1}H NMR datas of products were based on the ^{1}H NMR spectra in low concentration.
3) R. Liu, M. Zhang, J. Zhang, *Chem. Commun.* 2011, **47**, 12870

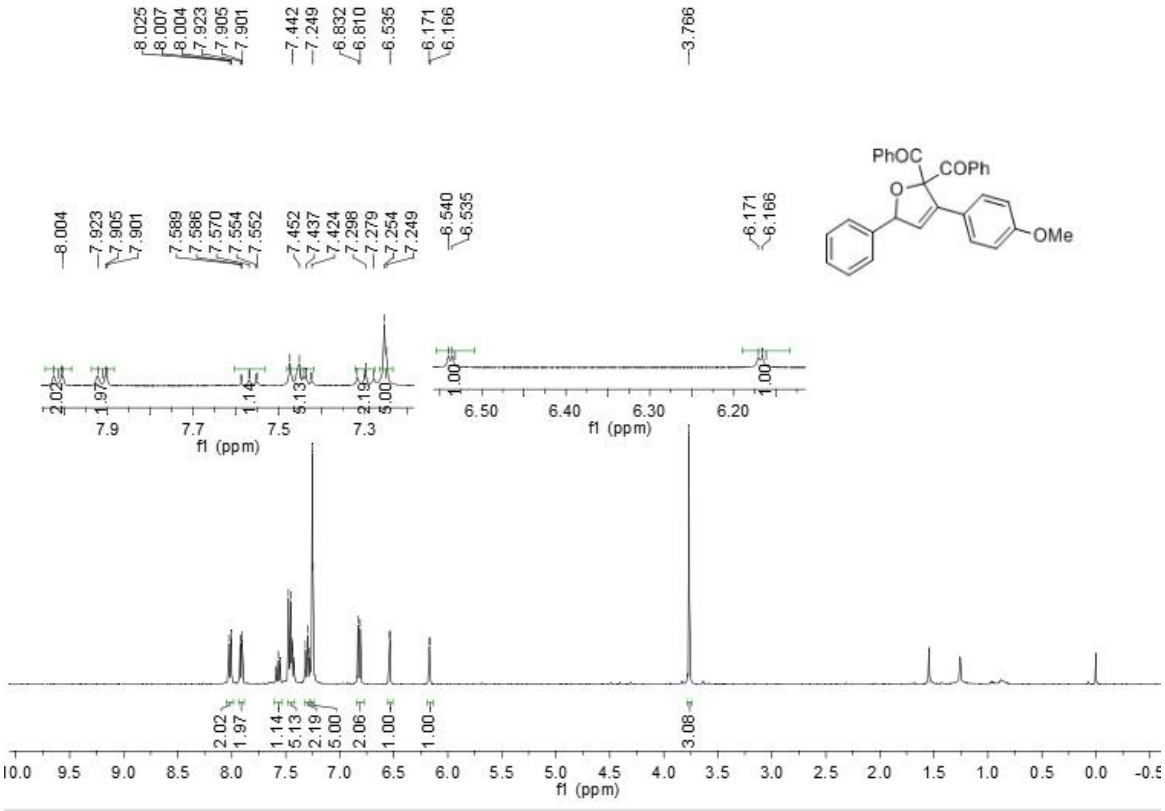
7. X-ray structure of 3m

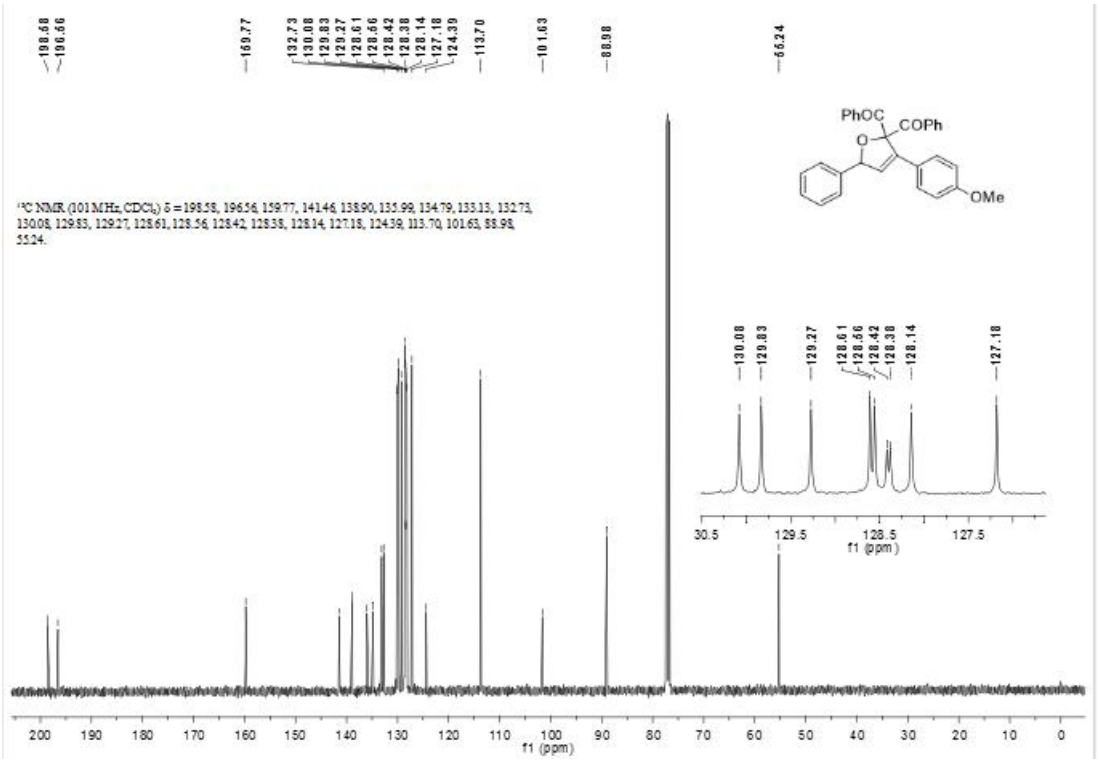


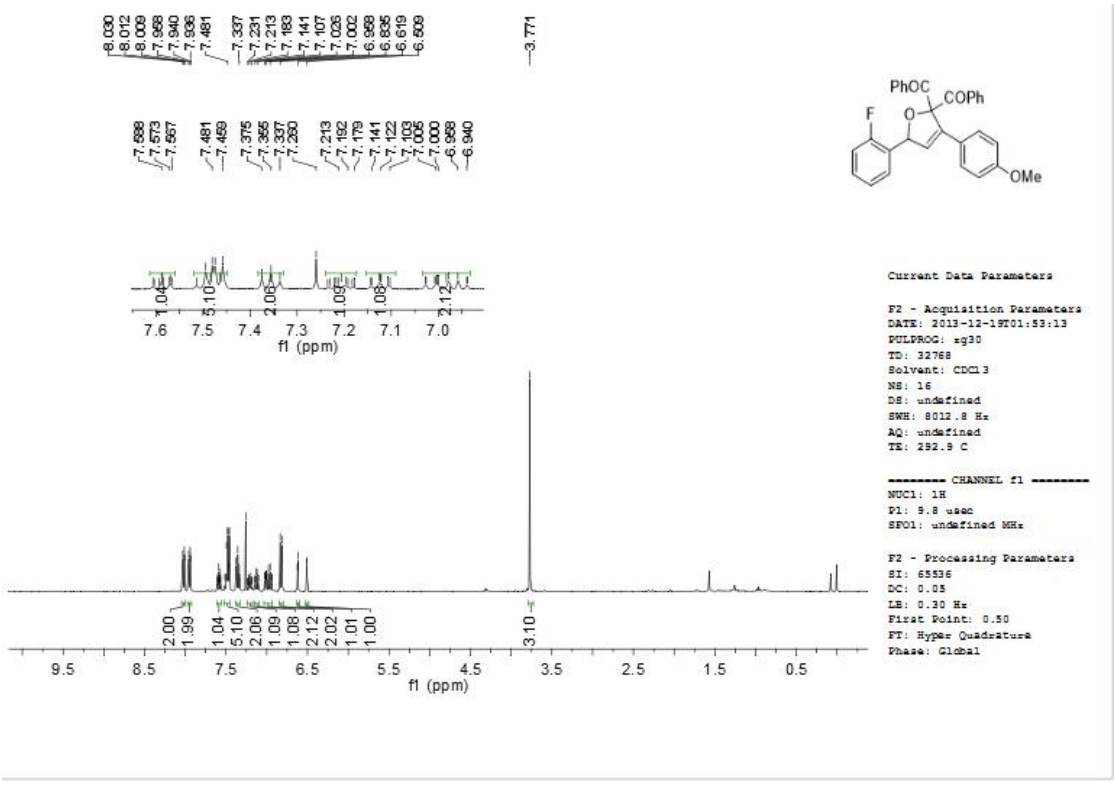
Single crystal of **3m** [$C_{35}H_{26}O_4$] was obtained from the solvent of CH_3CN. The absolute configuration of **3m** is R. CCDC 1003922 contains the supplementary crystallographic data which can be obtained free of charge from The Cambrige Crystallographic Data Centere via www.ccdc.cam.ac.uk/data_request/cif.

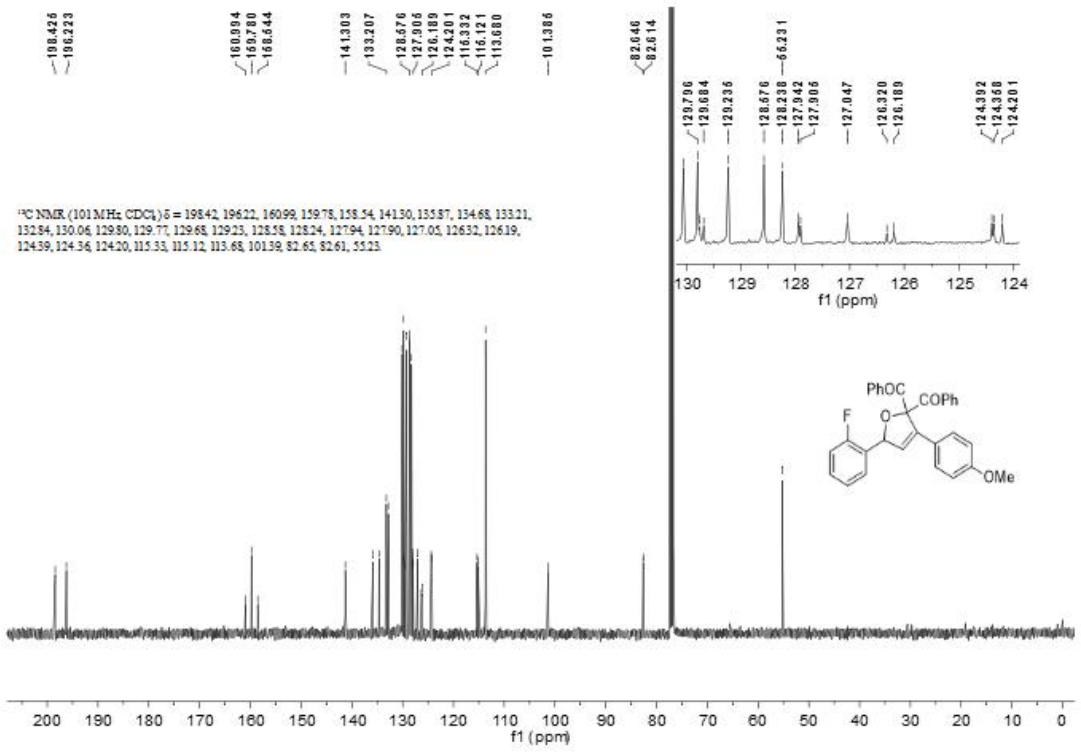
Crystal data. $C_{72}H_{55}NO_8$, M = 1062.17, monoclinic, a = 11.77380(9), b = 12.39402(7), c = 19.49664(13) Å, U = 2721.92(3) $Å^3$, T = 100.0(2) K, space group P1211, Z = 2.

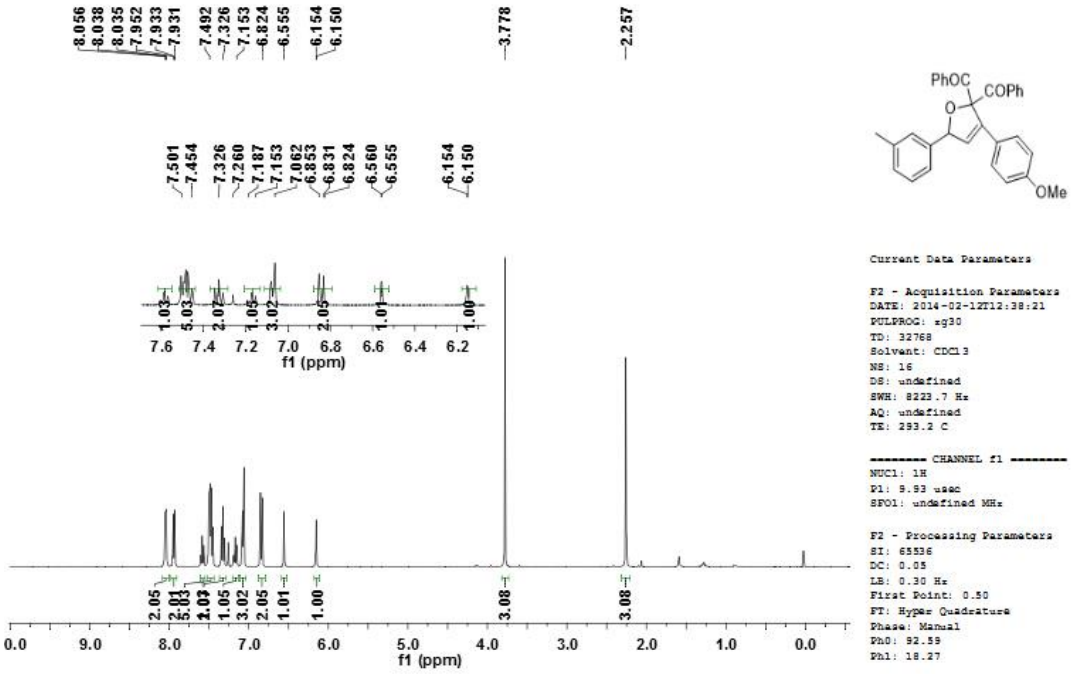
8. Copy of ^{1}H NMR and ^{13}C NMR spectra for products

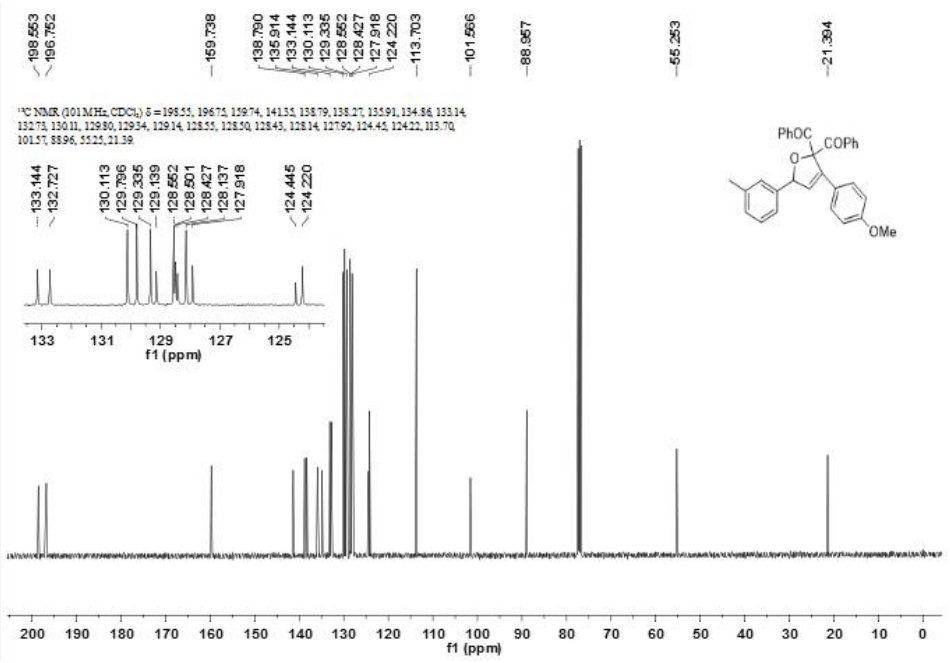


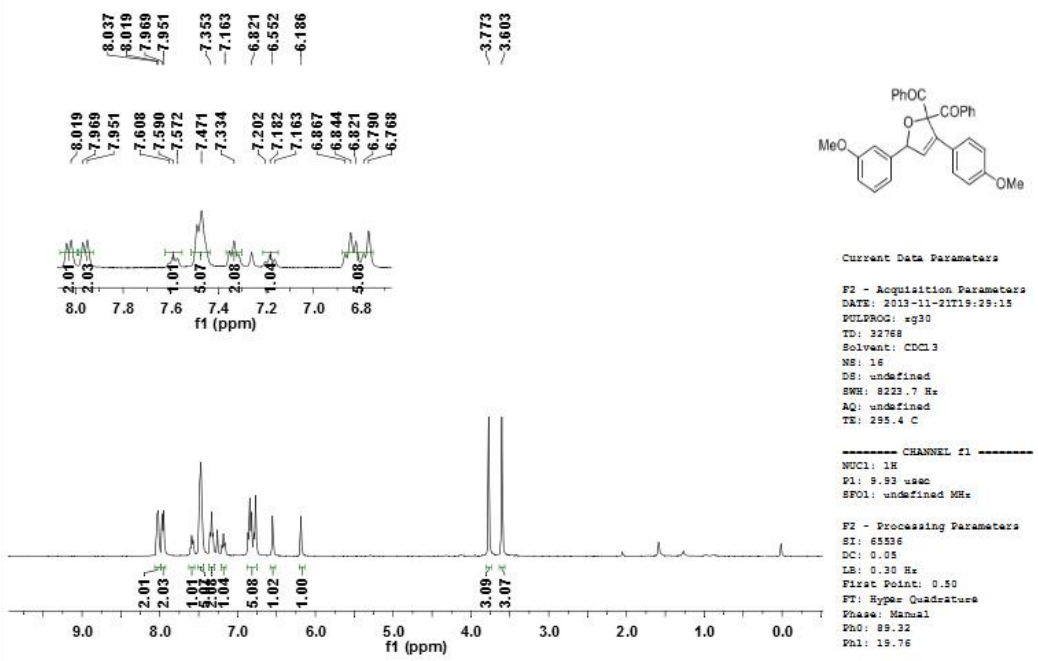

198.58
196.56
159.77
132.73
130.08
129.83
129.27
128.61
128.56
128.42
128.38
128.14
127.18
124.39
113.70
101.63
88.98
55.24
PhOC
COPh
O
OMe
13C NMR (101 MHz, CDCl3) δ = 198.58, 196.56, 159.77, 141.46, 138.90, 135.99, 134.79, 133.13, 132.73, 130.08, 129.83, 129.27, 128.61, 128.56, 128.42, 128.38, 128.14, 127.18, 124.39, 113.70, 101.63, 88.98, 55.24.
130.08
129.83
129.27
128.61
128.56
128.42
128.38
128.14
127.18
30.5
129.5
128.5
127.5
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

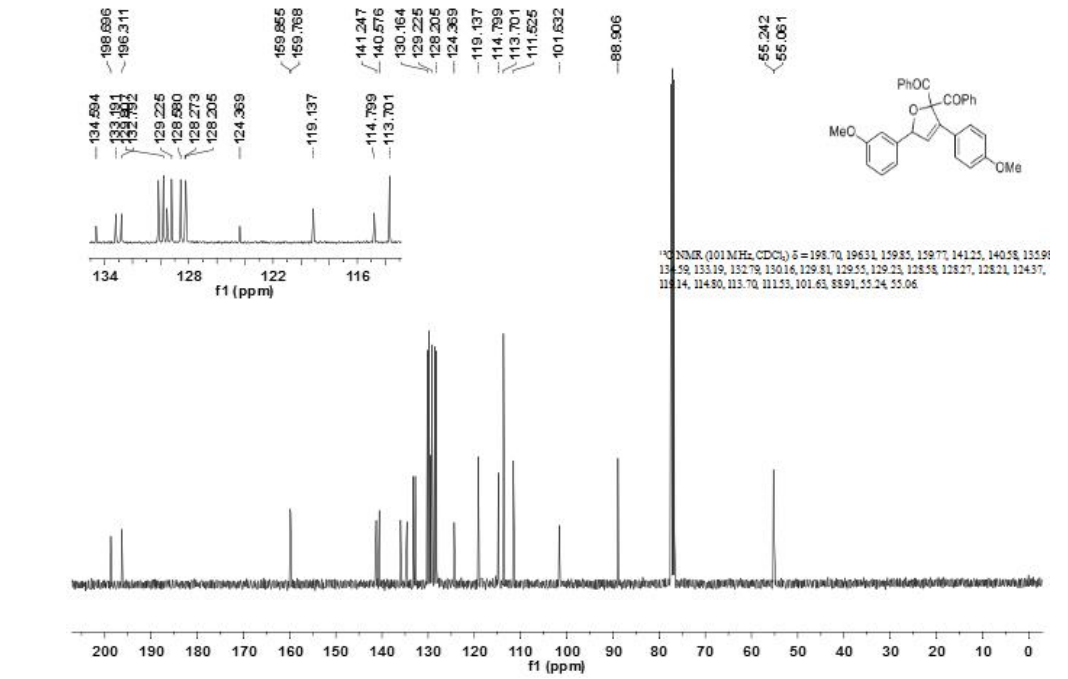

8.030
8.012
8.009
7.968
7.940
7.936
7.481
7.337
7.231
7.213
7.183
7.141
7.107
7.026
7.002
6.968
6.835
6.619
6.609
3.771
7.598
7.573
7.567
7.481
7.469
7.375
7.355
7.337
7.250
7.213
7.192
7.179
7.141
7.122
7.103
7.026
7.000
6.968
6.940
PhOC
COPh
F
O
OMe
1.04
5.10
2.06
1.09
1.08
2.12
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-12-19T01:53:13
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 292.9 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.8 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
2.00
1.99
1.04
5.10
2.06
1.09
1.08
2.12
2.02
1.01
1.00
3.10
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

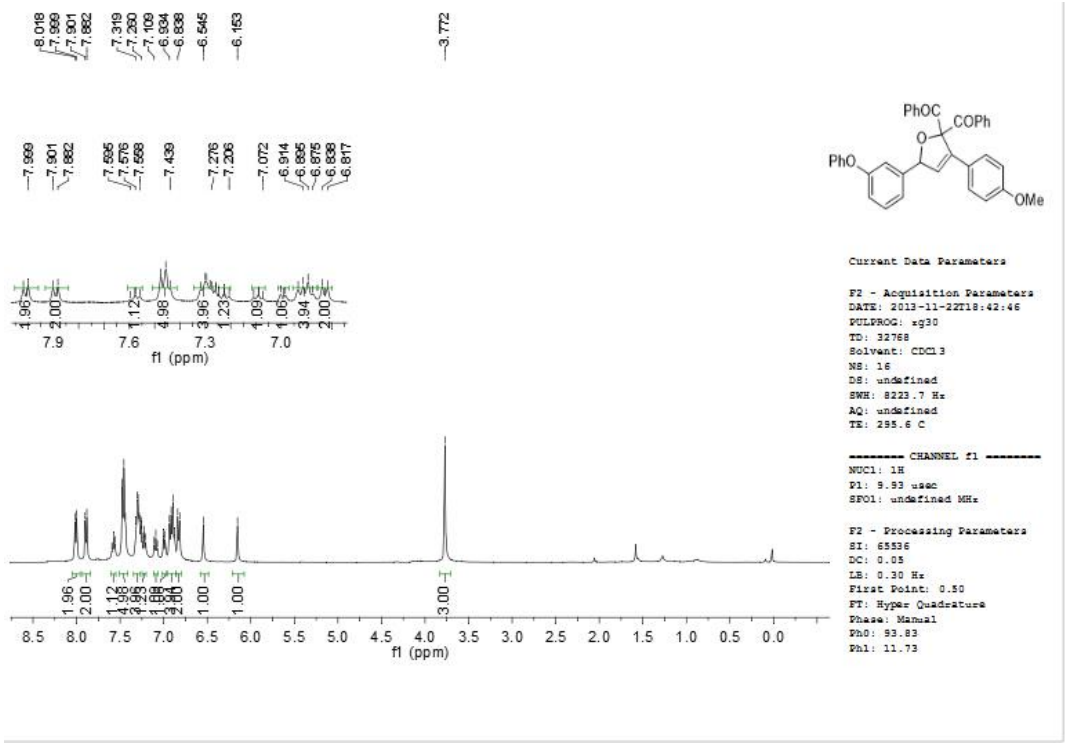

198.426
196.223
160.994
159.780
158.644
141.303
133.207
128.676
127.906
126.189
124.201
116.332
116.121
113.680
101.386
82.646
82.614
129.796
129.684
129.236
128.676
128.238
127.942
127.906
127.047
126.320
126.189
124.392
124.358
124.201
66.231
13C NMR (101 MHz, CDCl3) δ = 198.42, 196.22, 160.99, 159.78, 158.54, 141.30, 135.87, 134.68, 133.21, 132.84, 130.06, 129.80, 129.77, 129.68, 129.23, 128.58, 128.24, 127.94, 127.90, 127.05, 126.32, 126.19, 124.39, 124.36, 124.20, 115.33, 115.12, 113.68, 101.39, 82.65, 82.61, 55.23.
PhOC
COPh
F
O
OMe
f1 (ppm)

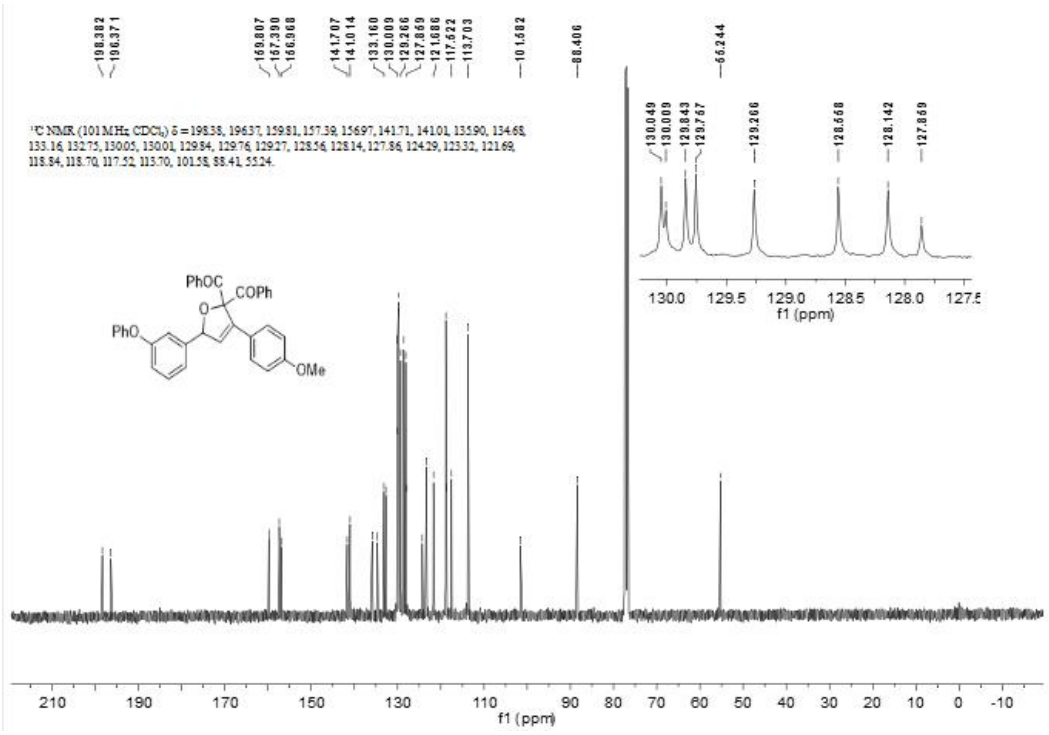

8.056
8.038
8.035
7.952
7.933
7.931
7.492
7.326
7.153
6.824
6.555
6.154
6.150
3.778
2.257
7.501
7.454
7.326
7.260
7.187
7.153
7.062
6.853
6.831
6.824
6.560
6.555
6.154
6.150
PhOC
COPh
O
OMe
1.03
5.03
2.07
1.05
3.02
2.05
1.01
1.00
f1 (ppm)
2.05
2.01
5.03
2.03
1.05
3.02
2.05
1.01
1.00
3.08
3.08
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2014-02-12T12:38:21
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 293.2 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.59
Ph1: 18.27

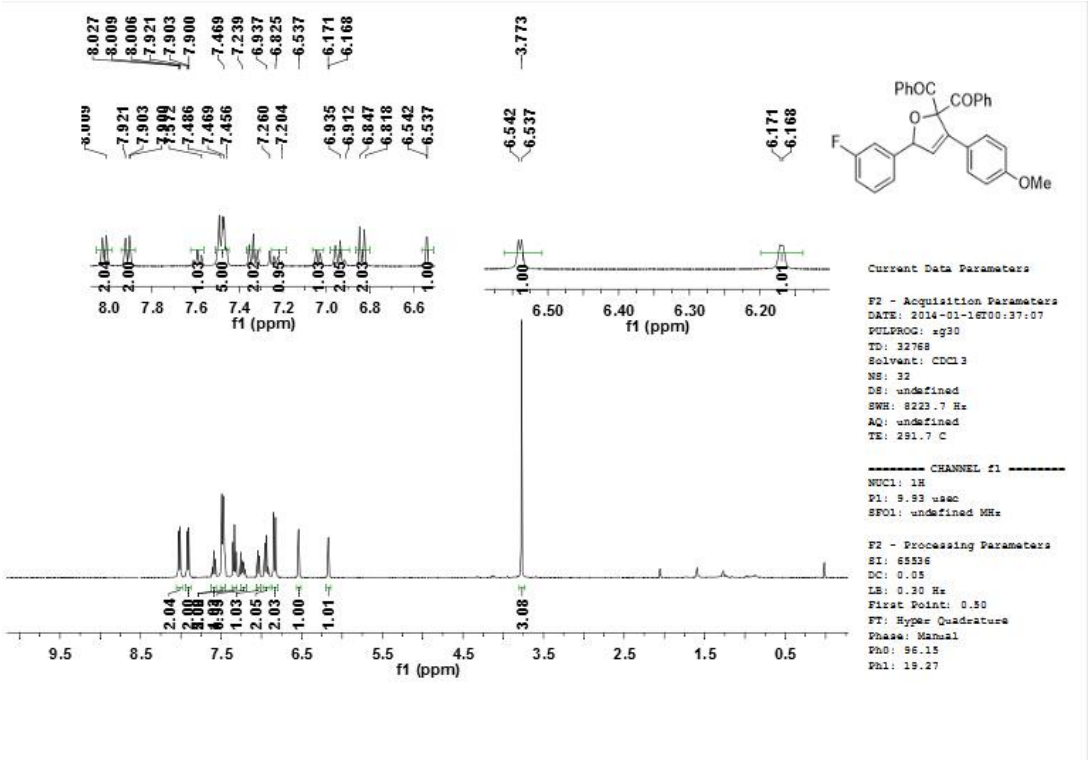

198.553
196.752
159.738
138.790
135.914
133.144
130.113
129.336
128.852
128.427
127.918
124.220
113.703
101.566
88.957
55.253
21.394
133.144
132.727
130.113
129.796
129.335
129.139
128.852
128.801
128.427
128.137
127.918
124.445
124.220
f1 (ppm)
PhOC
COPh
OMe

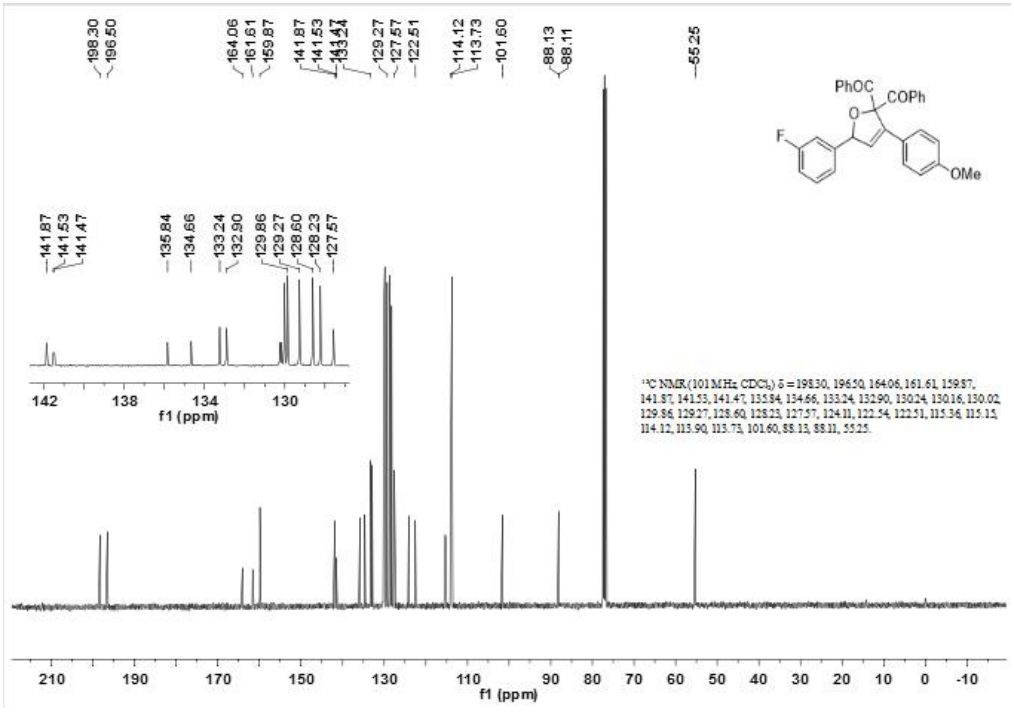

8.037
8.019
7.969
7.951
7.353
7.163
6.821
6.552
6.186
3.773
3.603
8.019
7.969
7.951
7.608
7.590
7.572
7.471
7.334
7.202
7.182
7.163
6.867
6.844
6.821
6.790
6.768
PhOC
COPh
MeO
OMe
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-21T19:29:15
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.4 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 89.32
Ph1: 19.76


198.696
196.311
159.865
159.768
141.247
140.576
130.164
129.225
128.205
124.369
119.137
114.799
113.701
111.525
101.632
88.906
55.242
55.061
PhOC
COPh
MeO
OMe
f1 (ppm)

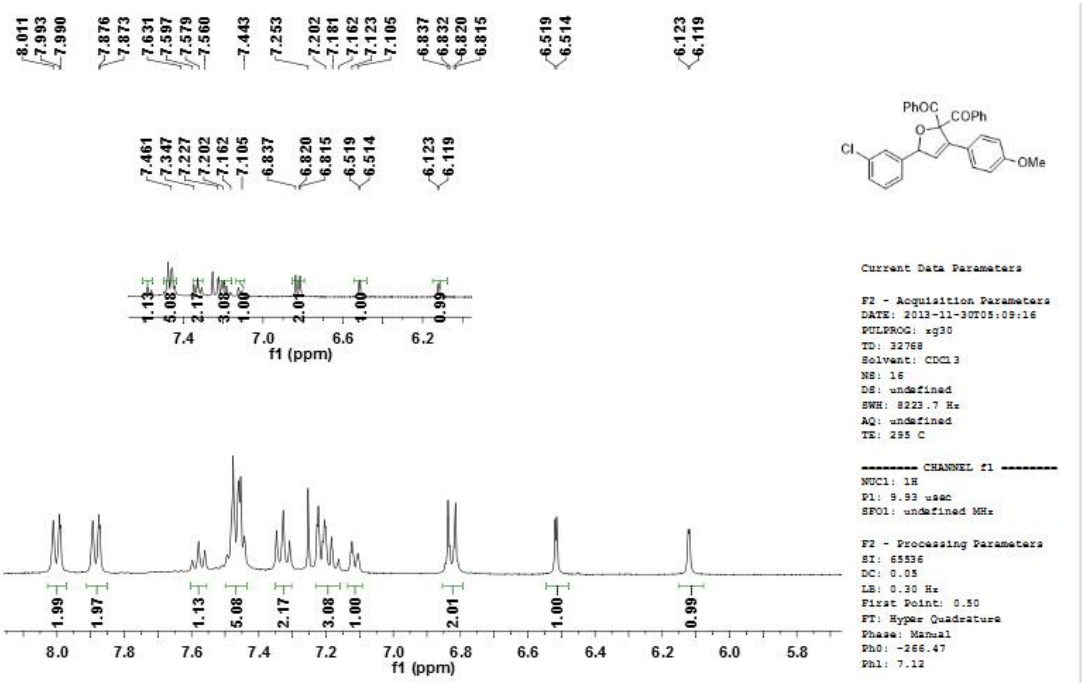

8.018
7.999
7.901
7.882
7.319
7.290
7.109
6.934
6.838
6.545
6.153
3.772
PhOC
COPh
PhO
OMe
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-22T18:42:46
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.6 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.83
Ph1: 11.73
f1 (ppm)


198.382
196.371
159.807
157.390
156.968
141.707
141.014
133.160
130.009
129.266
127.869
121.686
117.622
113.703
101.582
88.406
55.244
130.049
130.009
129.843
129.767
129.266
128.568
128.142
127.869
PhOC
COPh
PhO
OMe
130.0
129.5
129.0
128.5
128.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

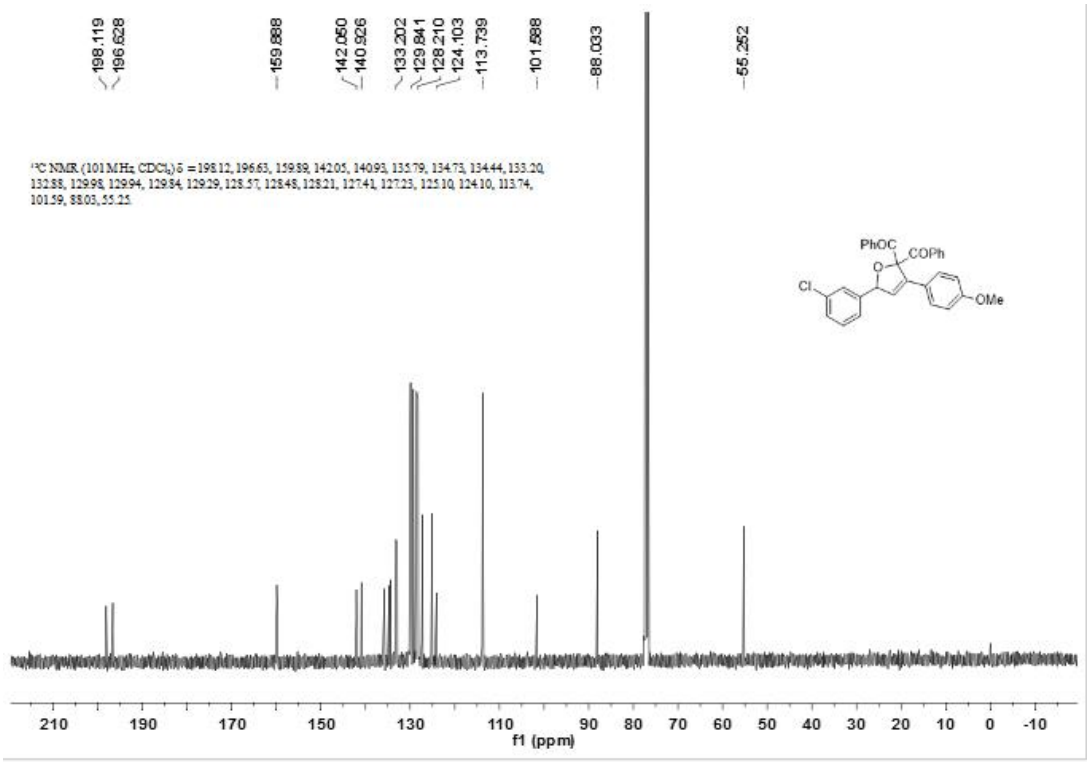

8.027
8.009
8.006
7.921
7.903
7.900
7.469
7.239
6.937
6.825
6.537
6.171
6.168
3.773
7.921
7.903
7.486
7.469
7.456
7.260
7.204
6.935
6.912
6.847
6.818
6.542
6.537
6.542
6.537
6.171
6.168
PhOC
COPh
F
OMe
2.04
2.00
1.03
5.00
2.02
0.95
1.03
2.05
2.03
1.00
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
f1 (ppm)
1.00
1.01
6.50
6.40
6.30
6.20
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2014-01-16T00:37:07
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 291.7 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 96.15
Ph1: 19.27
2.04
2.00
1.03
2.05
2.03
1.00
1.01
3.08
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)



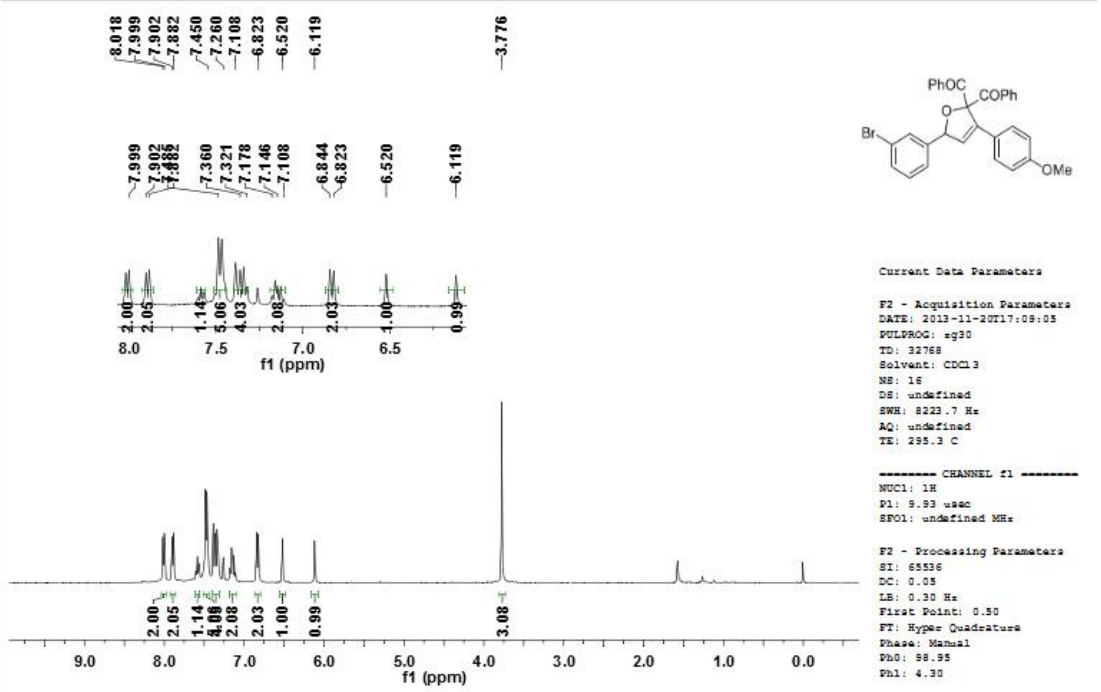
^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.30, 196.50, 164.06, 161.61, 159.87, 141.87, 141.53, 141.47, 135.84, 134.66, 133.24, 132.90, 130.24, 130.16, 130.02, 129.86, 129.27, 128.60, 128.23, 127.57, 124.11, 122.54, 122.51, 115.36, 115.15, 114.12, 113.90, 113.73, 101.60, 88.13, 88.11, 55.25.

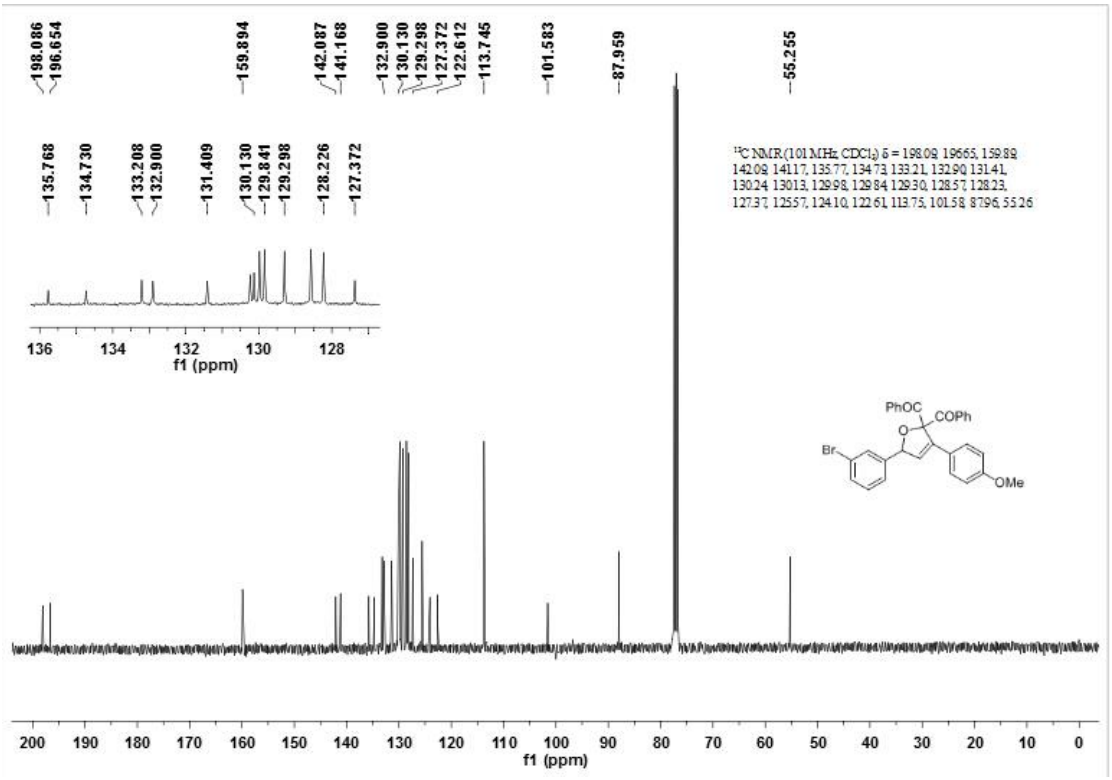


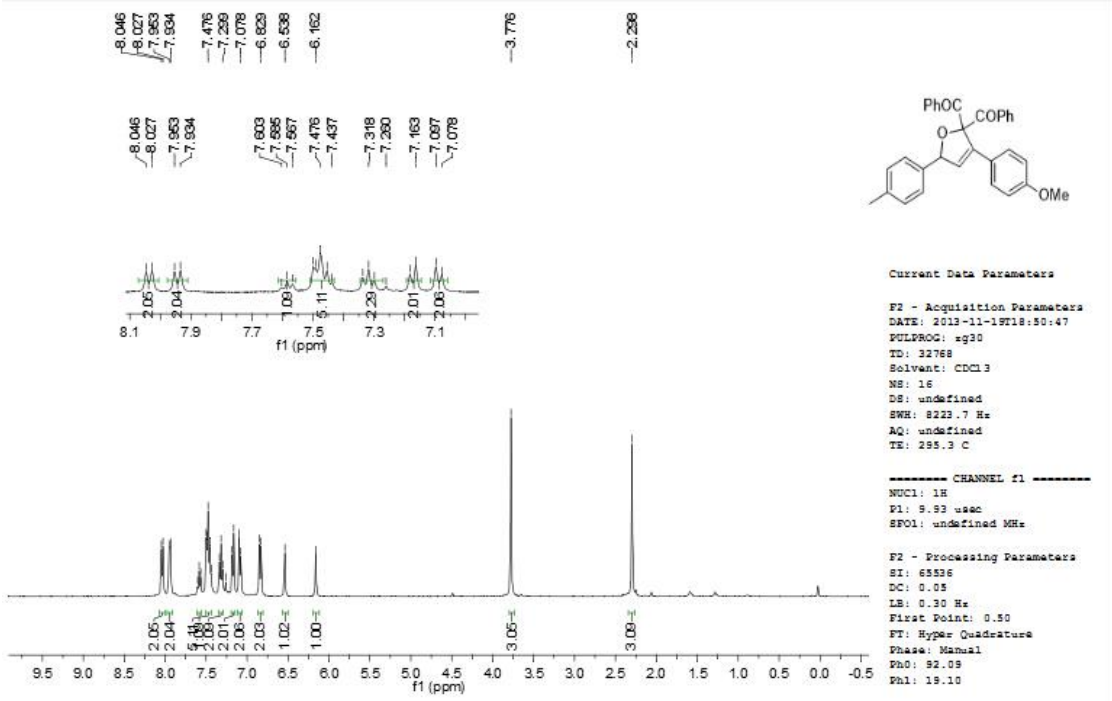
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.97 (m, 2H), 7.91 – 7.85 (m, 2H), 7.60 - 7.55 (m, 1H), 7.50 - 7.44 (m, 5H), 7.35 - 7.30 (m, 2H), 7.23 - 7.16 (m, 3H), 7.14–7.09 (m, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 2.0 Hz, 1H), 6.12 (d, *J* = 1.6 Hz, 1H), 3.77 (s, 3H)

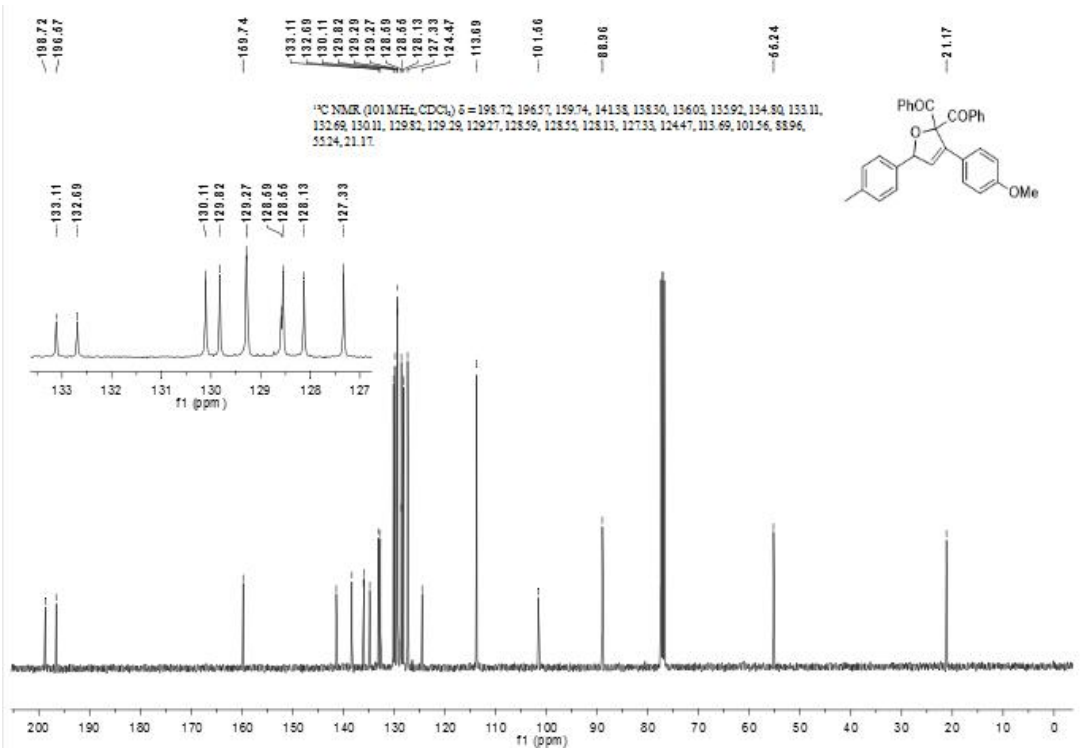


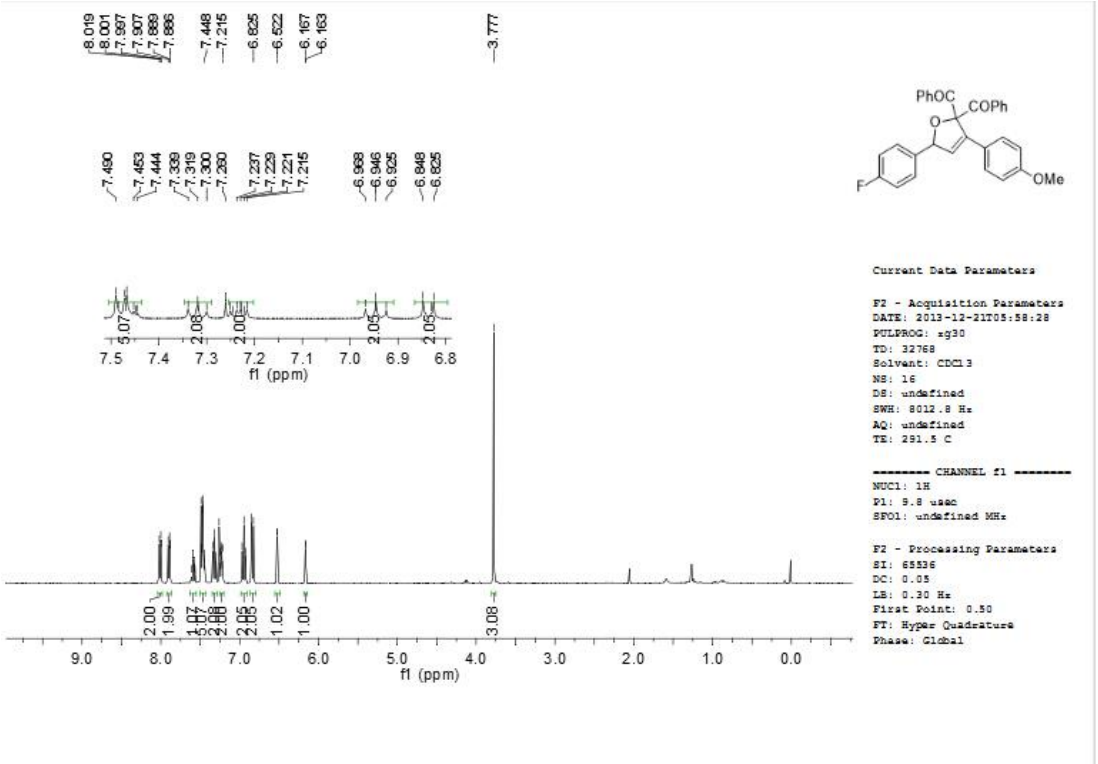
^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.12, 196.63, 159.89, 142.05, 140.93, 135.79, 134.73, 134.44, 133.20, 132.88, 129.98, 129.94, 129.84, 129.29, 128.57, 128.48, 128.21, 127.41, 127.23, 125.10, 124.10, 113.74, 101.59, 88.03, 55.25.

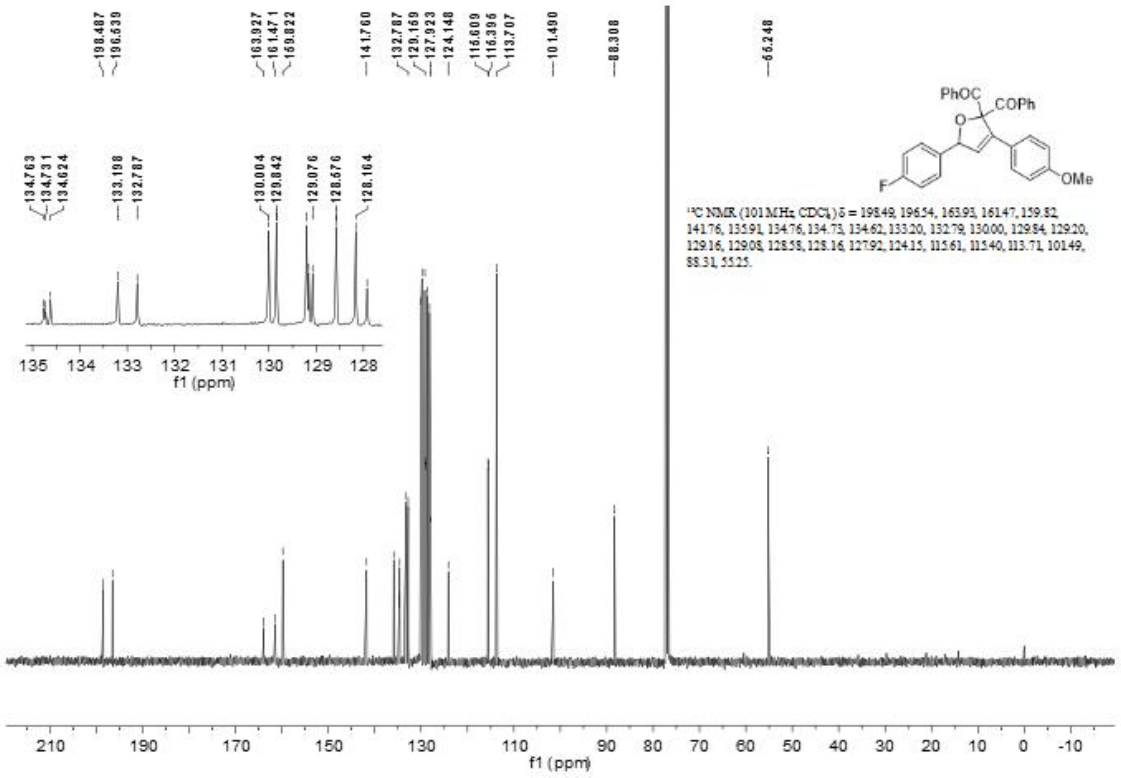


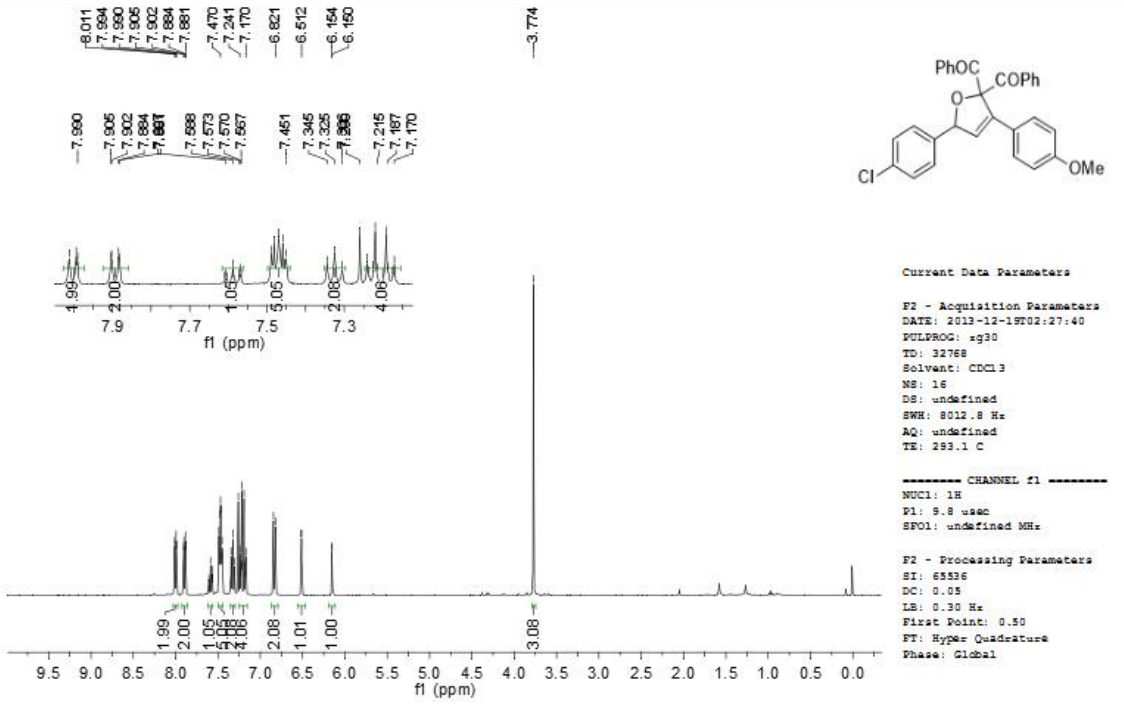

198.086
196.654
159.894
142.087
141.168
132.900
130.130
129.298
127.372
122.612
113.745
101.583
87.959
55.255
135.768
134.730
133.208
132.900
131.409
130.130
129.841
129.298
128.226
127.372
136
134
132
130
128
f1 (ppm)
13C NMR (101 MHz, CDCl3) δ = 198.09, 196.65, 159.89, 142.09, 141.17, 135.77, 134.73, 133.21, 132.90, 131.41, 130.24, 130.13, 129.98, 129.84, 129.30, 128.57, 128.23, 127.37, 125.57, 124.10, 122.61, 113.75, 101.58, 87.96, 55.26
PhOC
COPh
O
Br
OMe
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

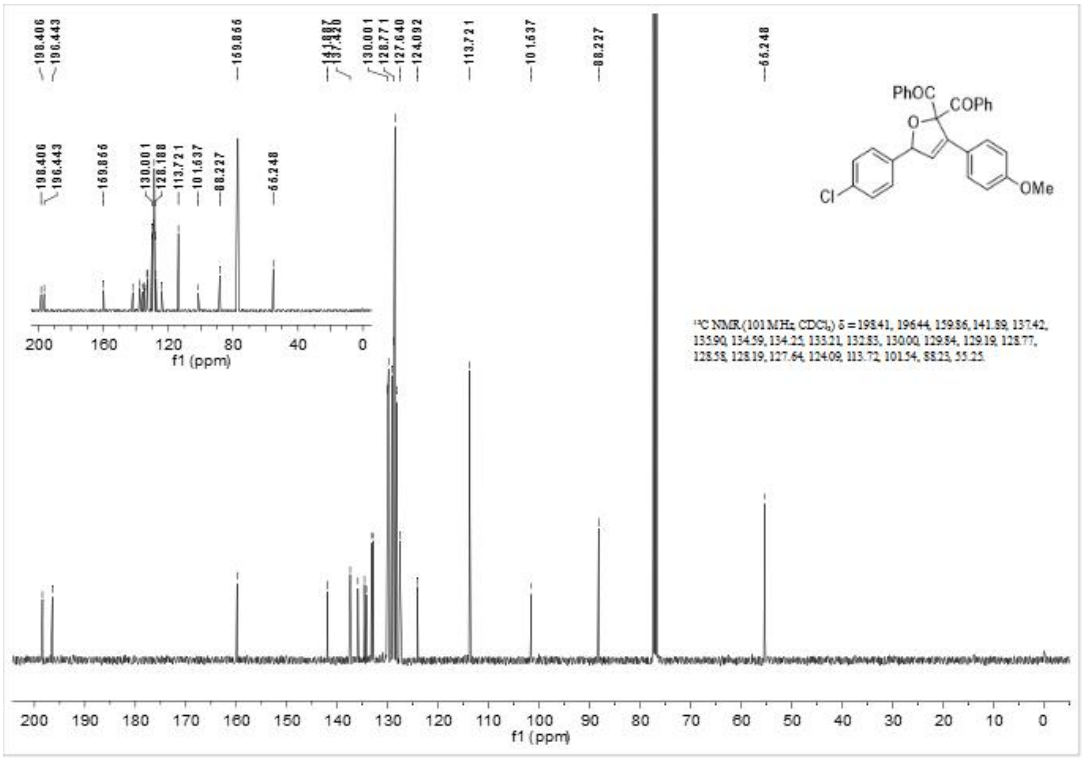

8.046
8.027
7.953
7.934
7.476
7.299
7.078
6.829
6.538
6.162
3.776
2.298
7.603
7.595
7.567
7.437
7.318
7.290
7.163
7.097
2.05
2.04
1.09
5.11
2.29
2.01
2.06
8.1
7.9
7.7
7.5
7.3
7.1
f1 (ppm)
PhOC
COPh
O
OMe
2.05
2.04
5.11
2.08
2.01
2.03
1.02
1.00
3.05
3.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-19T18:50:47
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.3 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.09
Ph1: 19.10

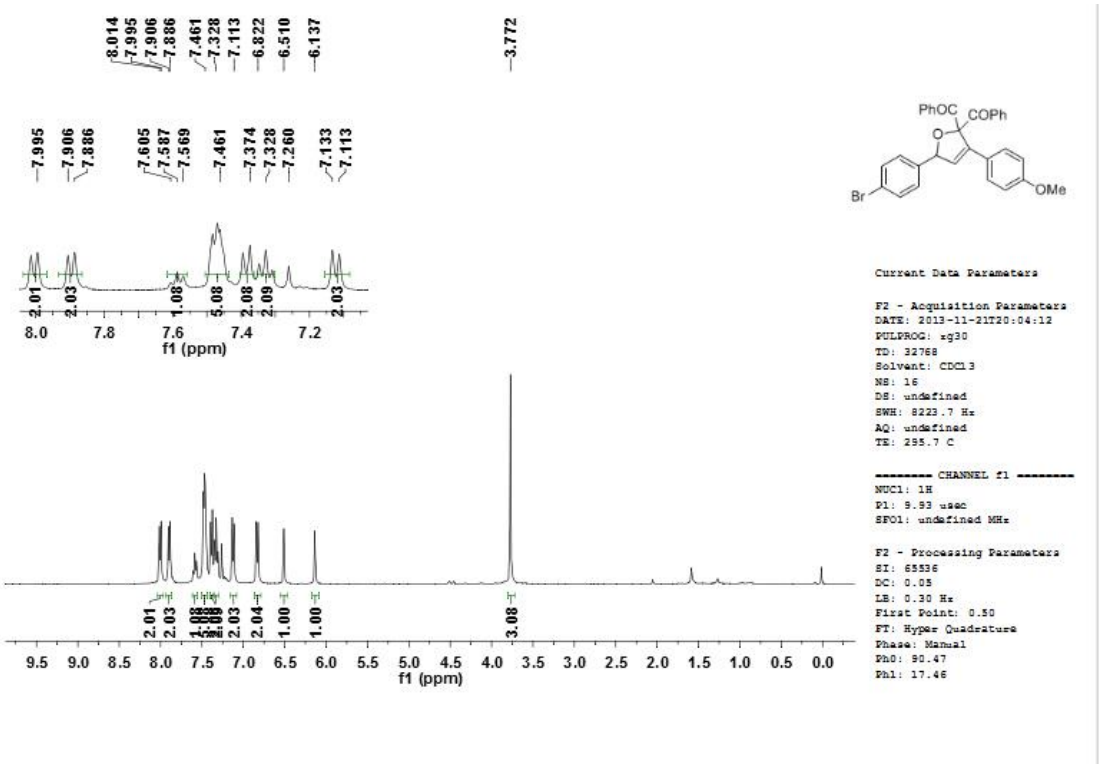

198.72
196.57
169.74
133.11
132.69
130.11
129.82
129.29
129.27
128.59
128.55
128.13
127.33
124.47
113.69
101.56
88.96
55.24
21.17
^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.72, 196.57, 159.74, 141.38, 138.30, 136.03, 135.92, 134.80, 133.11, 132.69, 130.11, 129.82, 129.29, 129.27, 128.59, 128.55, 128.13, 127.33, 124.47, 113.69, 101.56, 88.96, 55.24, 21.17.
PhOC
COPh
O
OMe
f1 (ppm)

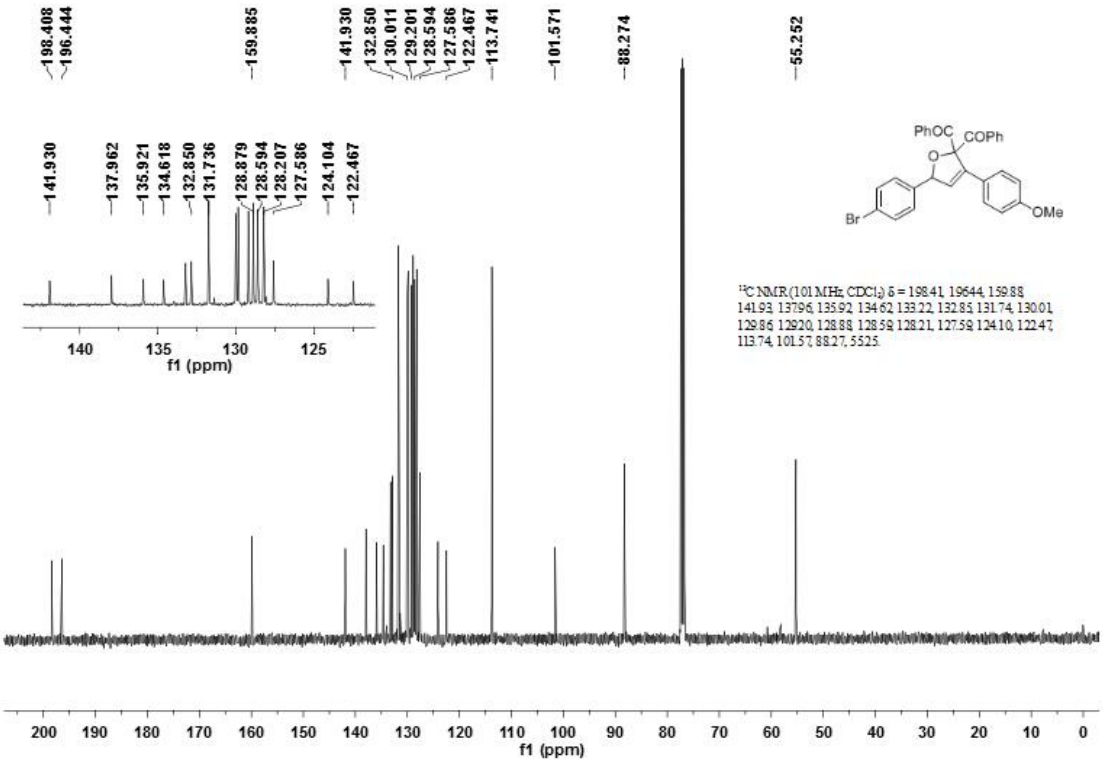

8.019
8.001
7.997
7.907
7.889
7.886
7.448
7.215
6.825
6.522
6.167
6.163
3.777
7.490
7.453
7.444
7.339
7.319
7.300
7.280
7.237
7.229
7.221
7.215
6.988
6.946
6.925
6.848
6.825
PhOC
COPh
O
F
OMe
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-12-21T05:58:28
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 291.5 C
CHANNEL f1
NUC1: 1H
P1: 9.8 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global


PhOC
COPh
O
F
OMe
^{13}C NMR (101 MHz, CDCl$_3$) δ = 198.49, 196.54, 163.93, 161.47, 159.82, 141.76, 135.91, 134.76, 134.73, 134.62, 133.20, 132.79, 130.00, 129.84, 129.20, 129.16, 129.08, 128.58, 128.16, 127.92, 124.15, 115.61, 115.40, 113.71, 101.49, 88.31, 55.25.
f1 (ppm)

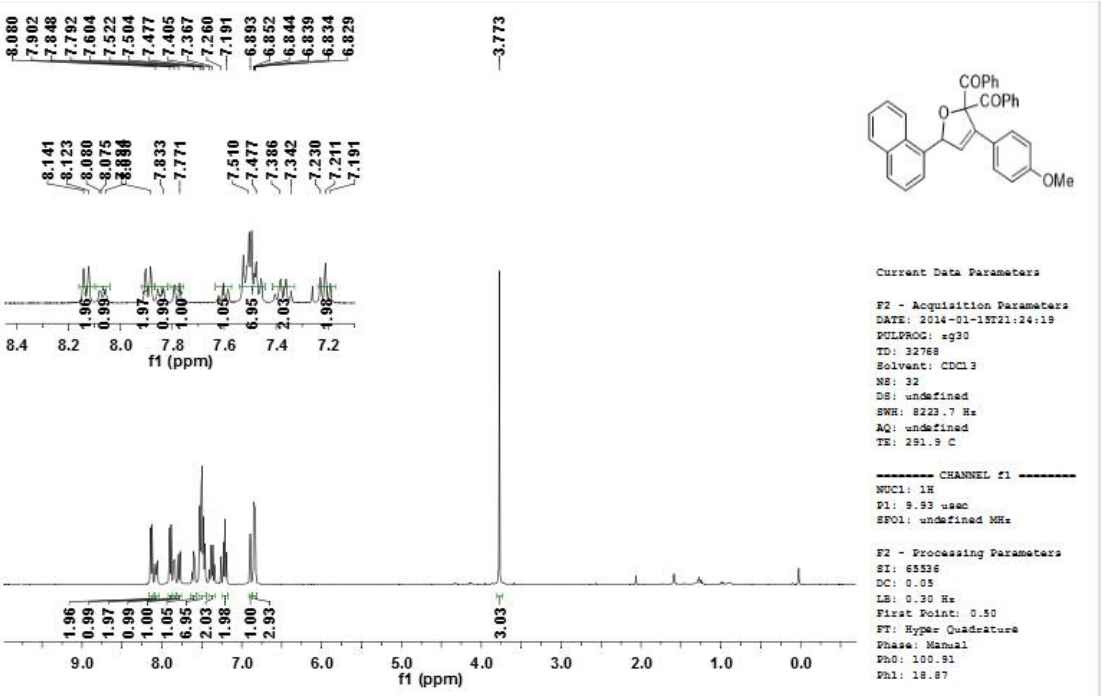

PhOC
COPh
O
Cl
OMe
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-12-19T02:27:40
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 293.1 C
CHANNEL f1
NUC1: 1H
P1: 9.8 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)


198.406
196.443
159.866
141.987
137.420
130.001
128.771
127.640
124.092
113.721
101.637
88.227
55.248
PhOC
COPh
O
Cl
OMe
f1 (ppm)
13C NMR (101 MHz, CDCl3) δ = 198.41, 196.44, 159.86, 141.99, 137.42, 135.90, 134.59, 134.23, 133.21, 132.83, 130.00, 129.84, 129.19, 128.77, 128.58, 128.19, 127.64, 124.09, 113.72, 101.54, 88.23, 55.25.


8.014
7.995
7.906
7.886
7.461
7.328
7.113
6.822
6.510
6.137
3.772
PhOC
COPh
O
Br
OMe
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-21T20:04:12
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.7 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
PH0: 90.47
PH1: 17.46



^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.41, 196.44, 159.88, 141.93, 137.96, 135.92, 134.62, 133.22, 132.85, 131.74, 130.01, 129.86, 129.20, 128.88, 128.59, 128.21, 127.59, 124.10, 122.47, 113.74, 101.57, 88.27, 55.25.



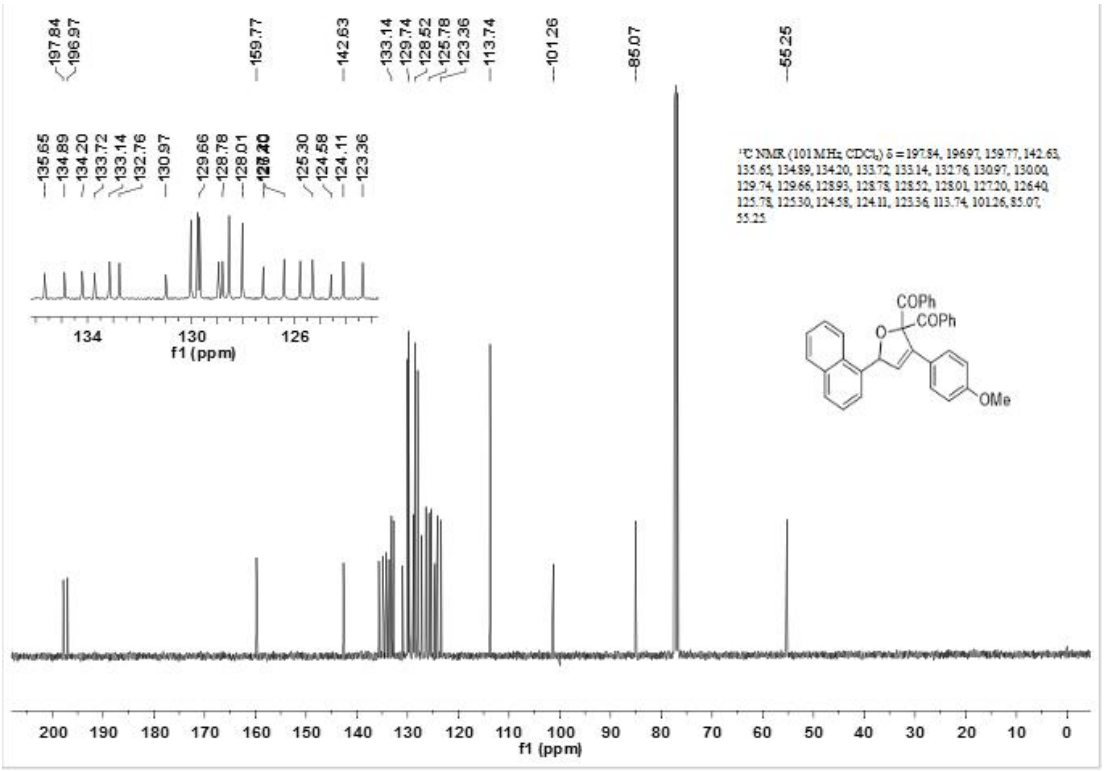
Current Data Parameters

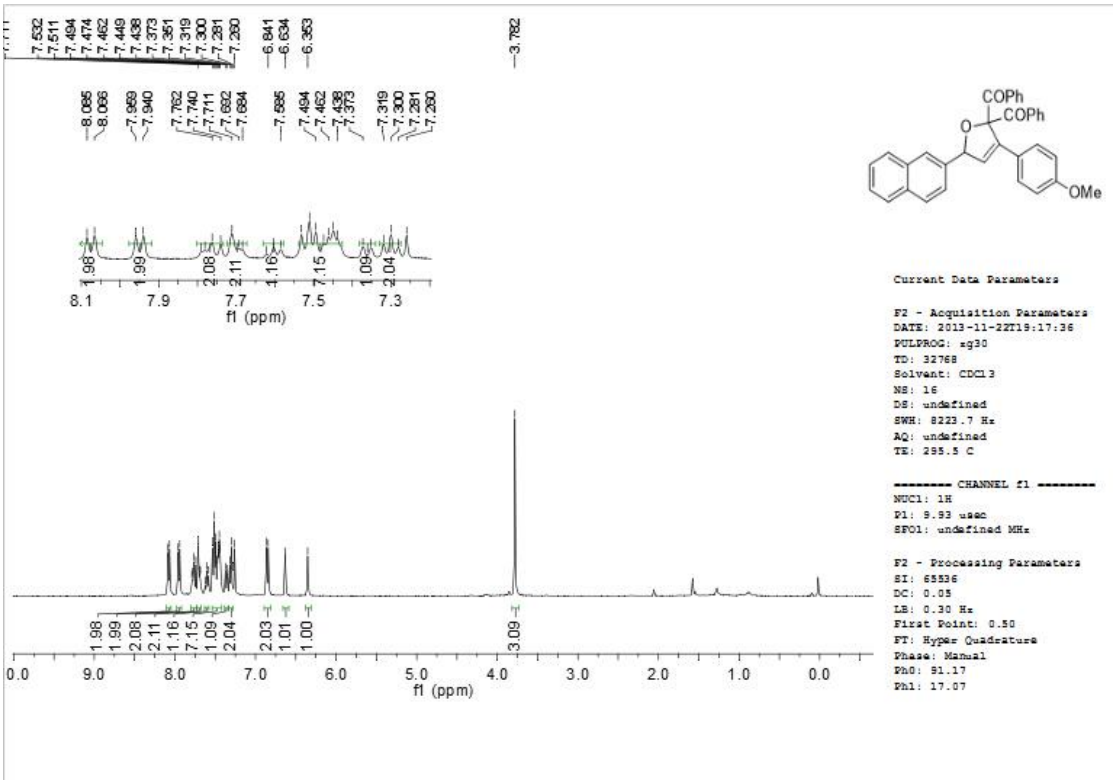
F2 - Acquisition Parameters
DATE: 2014-01-15T21:24:19
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 291.9 C

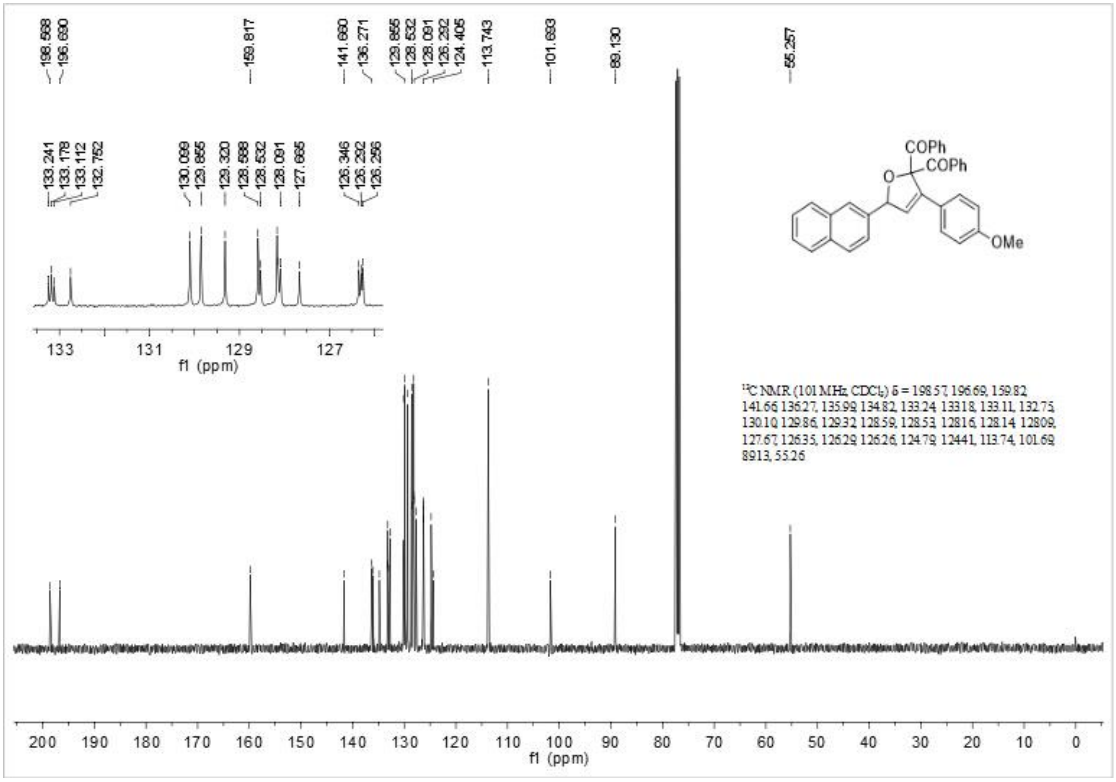
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

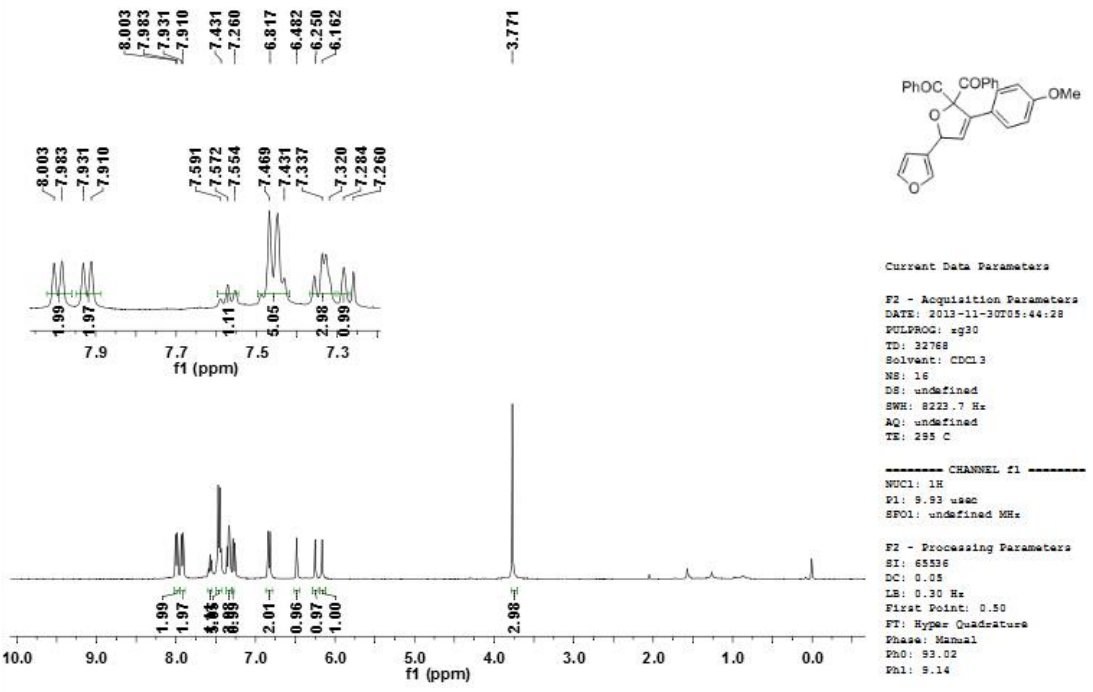
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 100.91
Ph1: 18.87

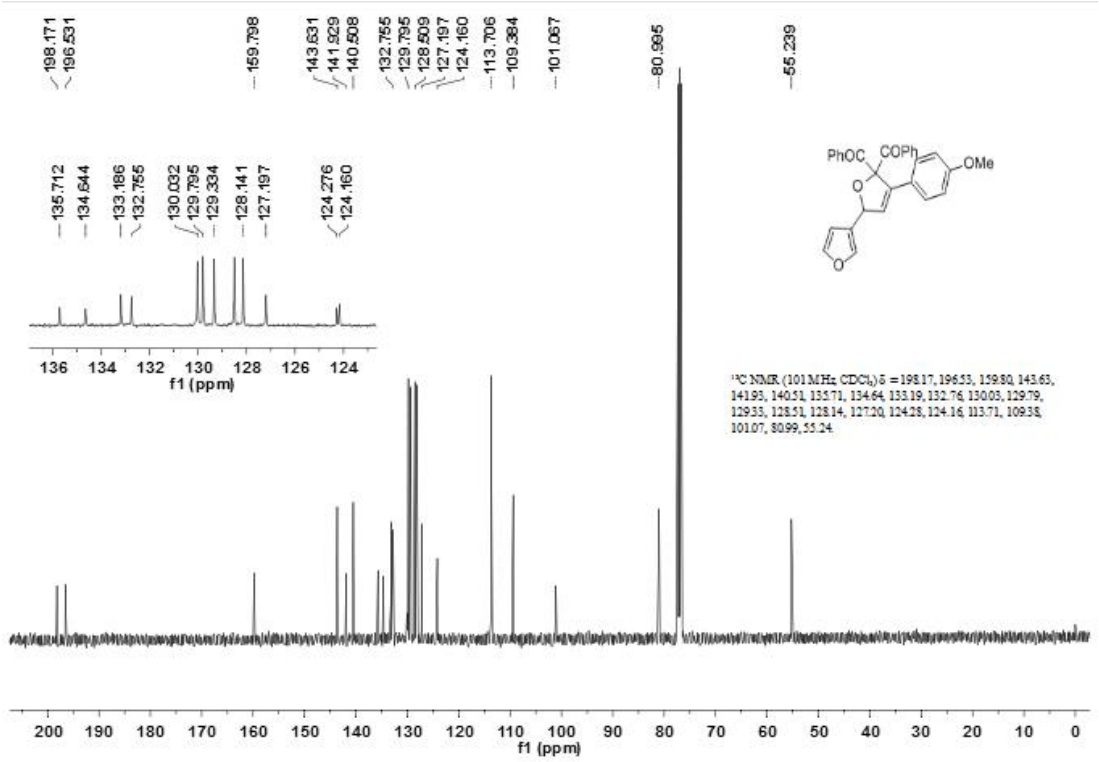
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 - 8.10 (m, 2H), 8.09 – 8.04 (m, 1H), 7.86 - 7.82 (m, 2H), 7.81 - 7.75 (m, 1H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.63 - 7.57 (m, 1H), 7.54 – 7.44 (m, 7H), 7.41 - 7.33 (m, 2H), 7.24 - 7.17 (m, 2H), 6.89 (s, 1H), 6.87 – 6.81 (m, 3H), 3.77 (s, 3H).


197.84
196.97
159.77
142.63
133.14
129.74
128.52
125.78
123.36
113.74
101.26
85.07
55.25
135.65
134.89
134.20
133.72
133.14
132.76
130.97
129.66
128.78
128.01
125.30
124.58
124.11
123.36
134
130
126
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
COPh
COPh
O
OMe

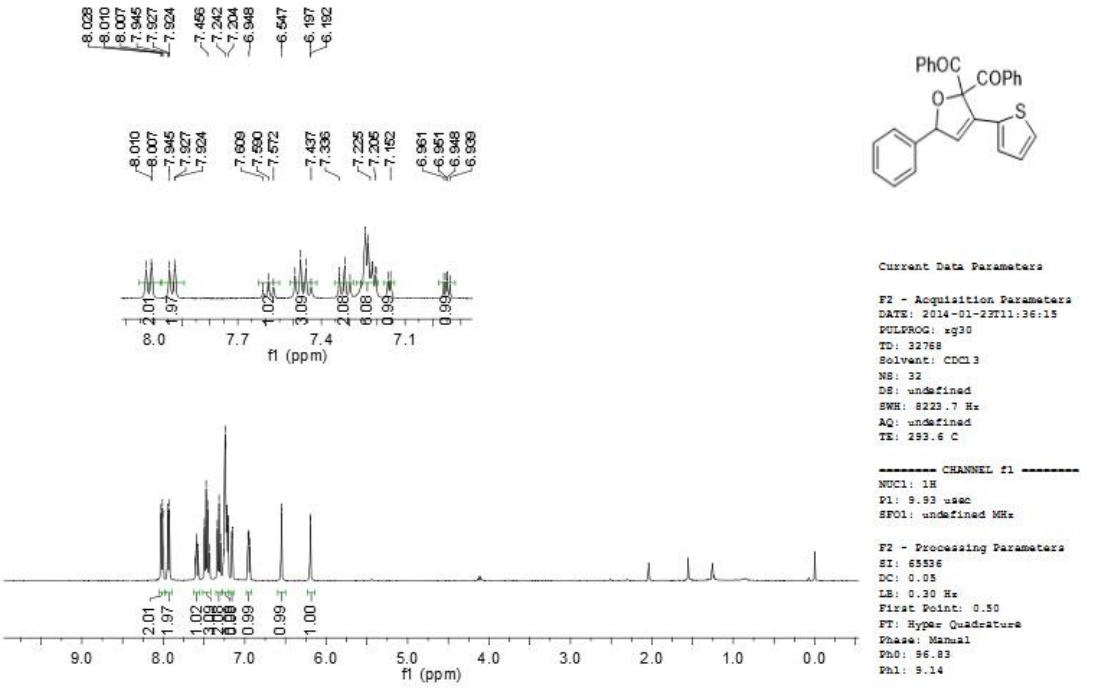

7.532
7.511
7.494
7.474
7.462
7.449
7.438
7.373
7.351
7.319
7.300
7.281
7.260
6.841
6.634
6.353
3.782
8.086
8.066
7.969
7.940
7.762
7.740
7.711
7.692
7.684
7.595
7.494
7.462
7.438
7.373
7.319
7.300
7.281
7.260
1.98
1.99
2.08
2.11
1.16
7.15
1.09
2.04
8.1
7.9
7.7
7.5
7.3
f1 (ppm)
COPh
COPh
O
OMe
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-22T19:17:36
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.5 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.17
Ph1: 17.07
1.98
1.99
2.08
2.11
1.16
7.15
1.09
2.04
2.03
1.01
1.00
3.09
0.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


198.568
196.690
159.817
141.660
136.271
129.855
128.532
128.091
126.292
124.405
113.743
101.693
89.130
55.257
133.241
133.178
133.112
132.762
130.099
129.855
129.320
128.688
128.532
128.091
127.665
126.346
126.292
126.256
COPh
COPh
O
OMe
133
131
129
127
f1 (ppm)
^{13}C NMR (101 MHz, CDCl$_3$) δ = 198.57, 196.69, 159.82, 141.66, 136.27, 135.99, 134.82, 133.24, 133.18, 133.11, 132.75, 130.10, 129.86, 129.32, 128.59, 128.53, 128.16, 128.14, 128.09, 127.67, 126.35, 126.29, 126.26, 124.79, 124.41, 113.74, 101.69, 89.13, 55.26
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

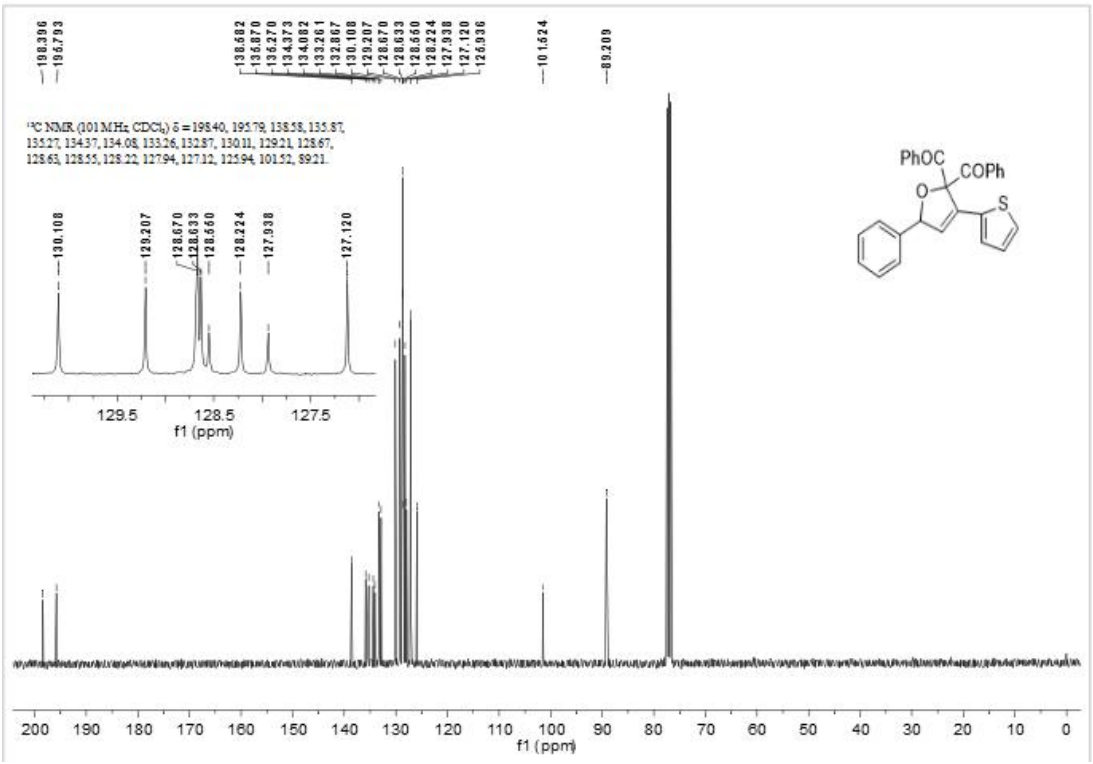

8.003
7.983
7.931
7.910
7.431
7.260
6.817
6.482
6.250
6.162
3.771
PhOC
COPh
OMe
O
1.99
1.97
1.11
5.05
2.98
0.99
7.9
7.7
7.5
7.3
f1 (ppm)
1.99
1.97
2.01
0.96
0.97
1.00
2.98
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-30T05:44:28
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.02
Ph1: 9.14

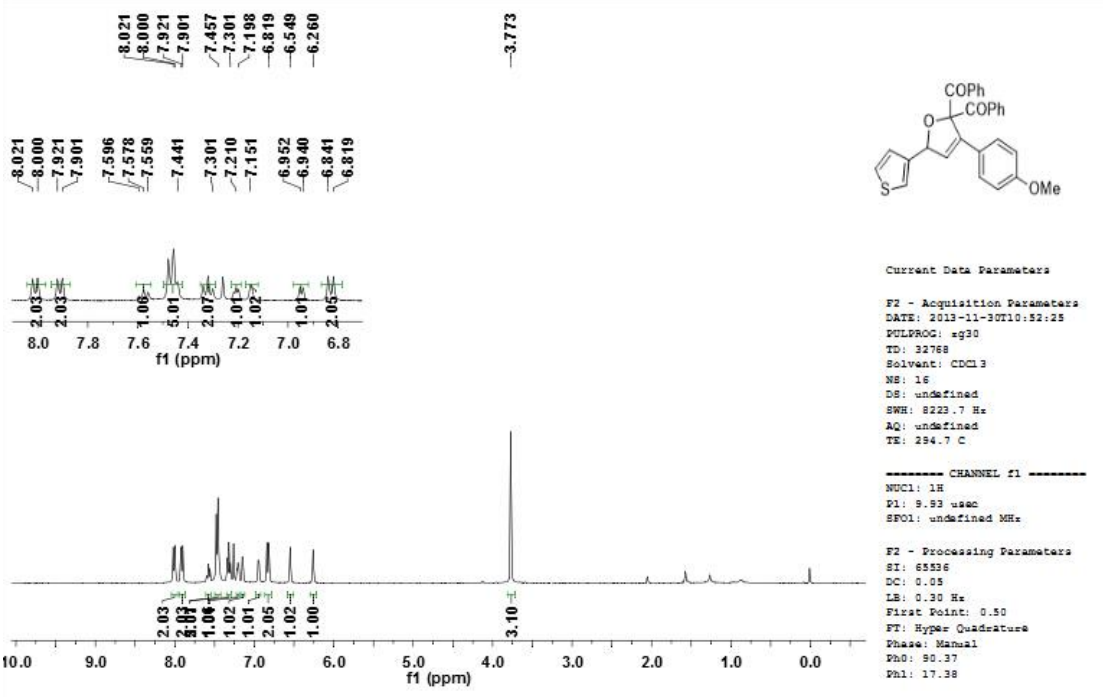


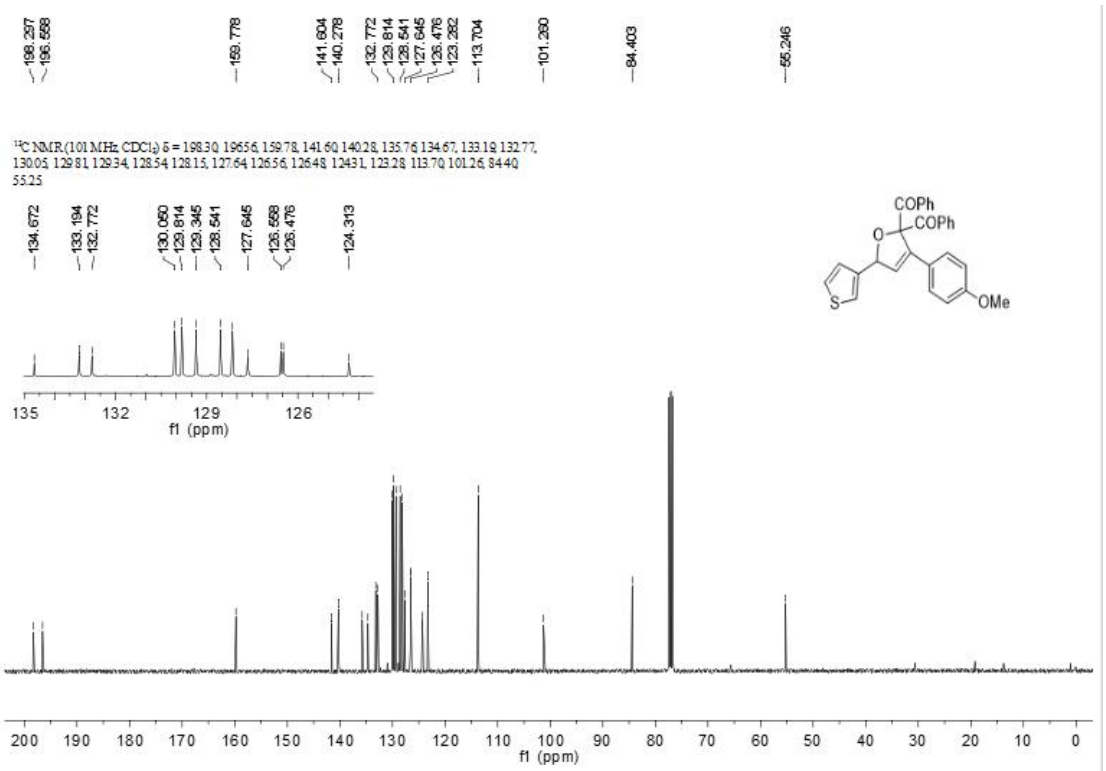
^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.17, 196.53, 159.80, 143.63, 141.93, 140.51, 135.71, 134.64, 133.19, 132.76, 130.03, 129.79, 129.33, 128.53, 128.14, 127.20, 124.28, 124.16, 113.71, 109.38, 101.07, 80.99, 55.24.



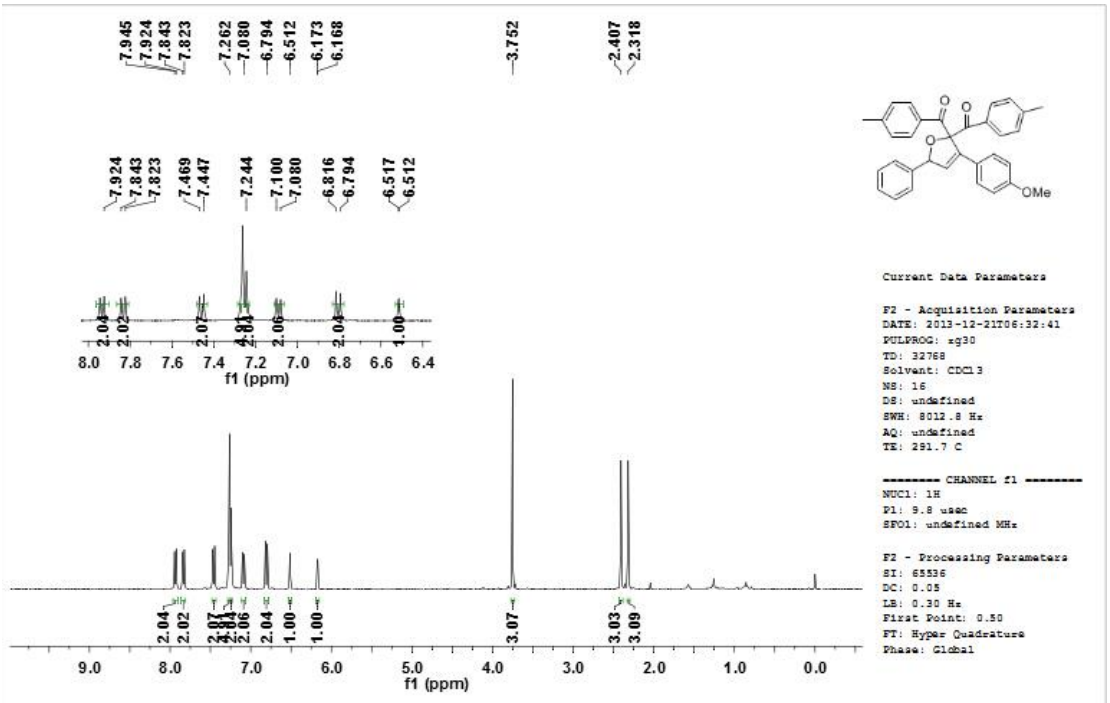

```
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2014-01-23T11:36:15
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 293.6 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 96.83
Ph1: 9.14
```


198.396
195.793
138.682
135.870
135.270
134.373
134.082
133.261
132.867
130.108
129.207
128.670
128.633
128.560
128.224
127.938
127.120
126.936
101.624
89.209
¹³C NMR (101 MHz CDCl₃) δ = 195.40, 195.79, 138.58, 135.87, 135.27, 134.37, 134.08, 133.26, 132.87, 130.11, 129.21, 128.67, 128.63, 128.55, 128.22, 127.94, 127.12, 125.94, 101.52, 89.21.
PhOC
COPh
O
S
129.5
128.5
127.5
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

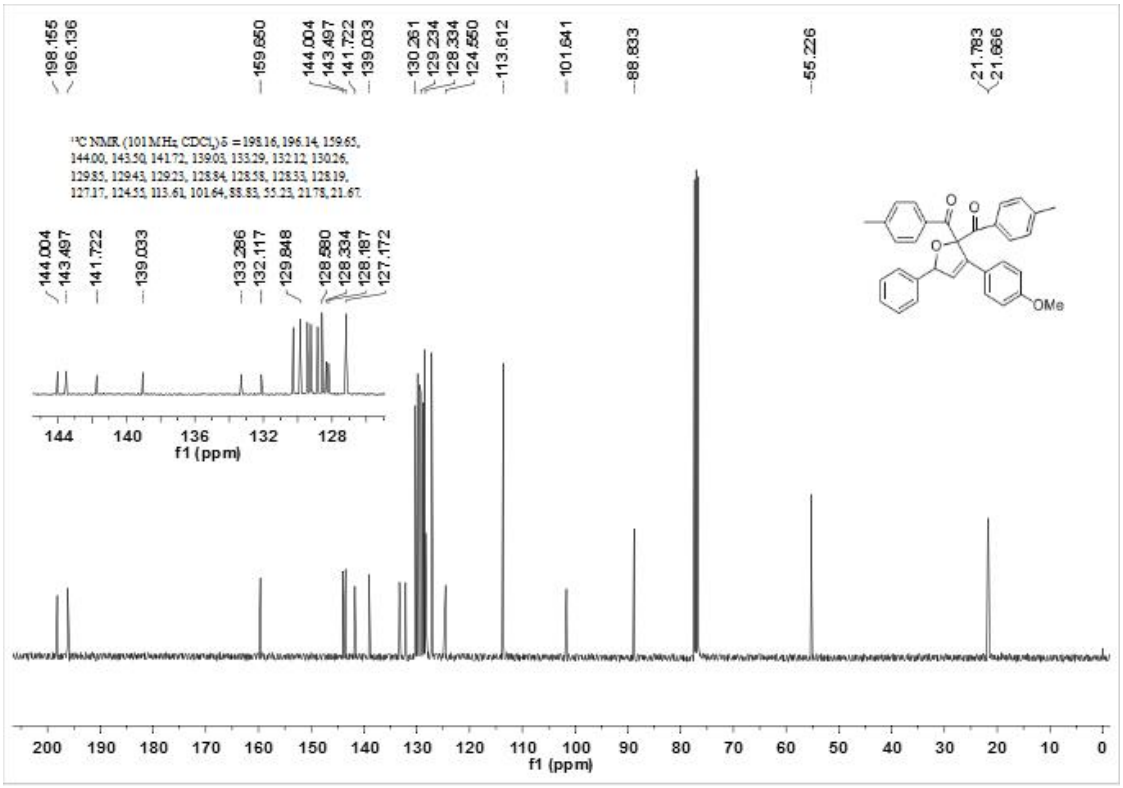

8.021
8.000
7.921
7.901
7.457
7.301
7.198
6.819
6.549
6.260
3.773
COPh
COPh
O
S
OMe
8.0 7.8 7.6 7.4 7.2 7.0 6.8
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2013-11-30T10:52:25
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 294.7 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 90.37
Ph1: 17.38
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

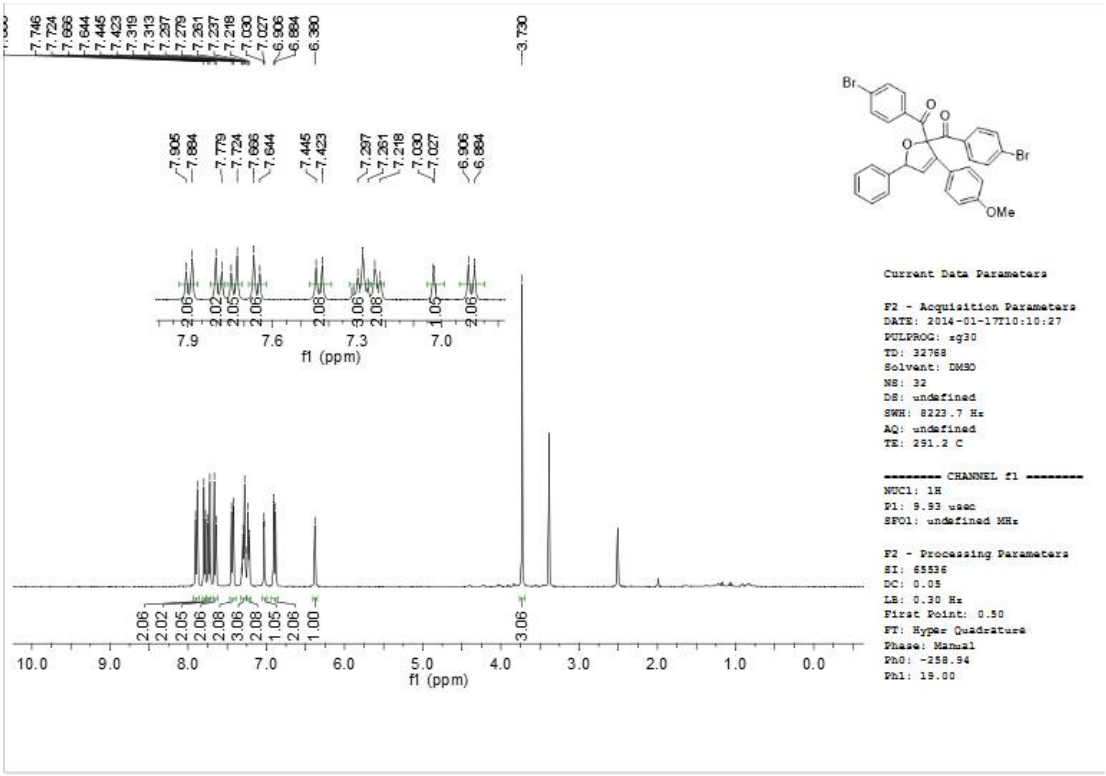


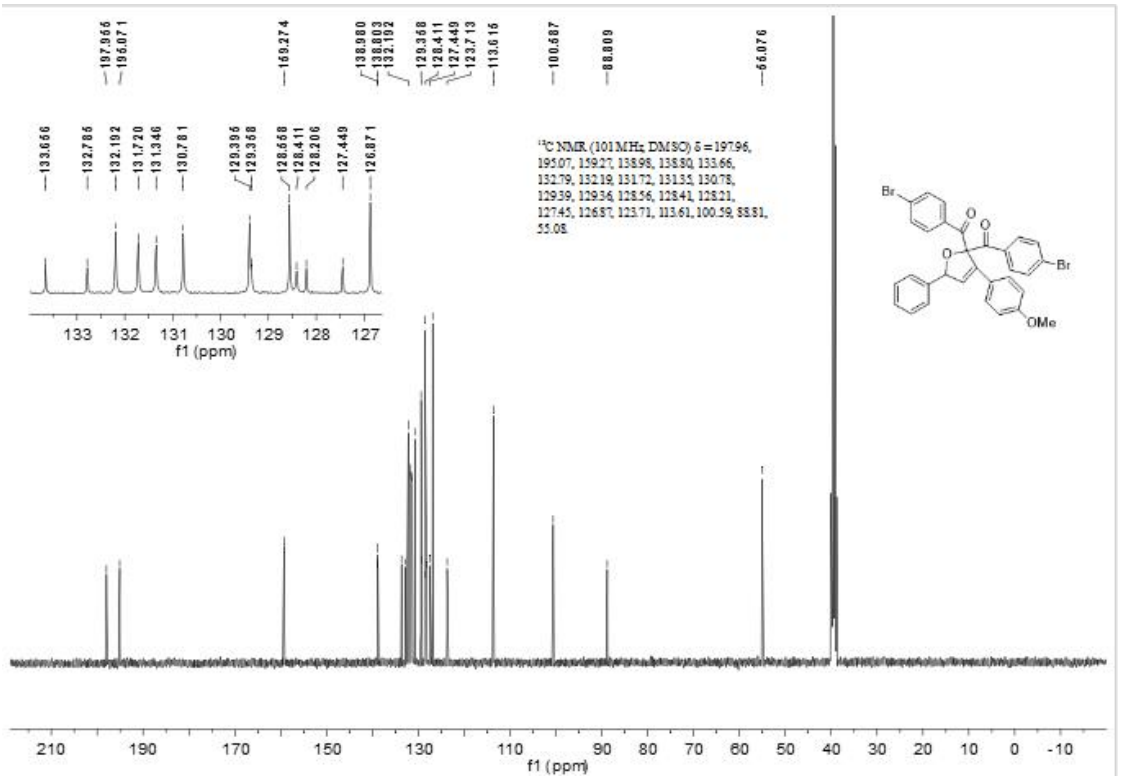
^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.30, 196.56, 159.78, 141.60, 140.28, 135.76, 134.67, 133.19, 132.77, 130.05, 129.81, 129.34, 128.54, 128.15, 127.64, 126.56, 126.48, 124.31, 123.28, 113.70, 101.26, 84.40, 55.25

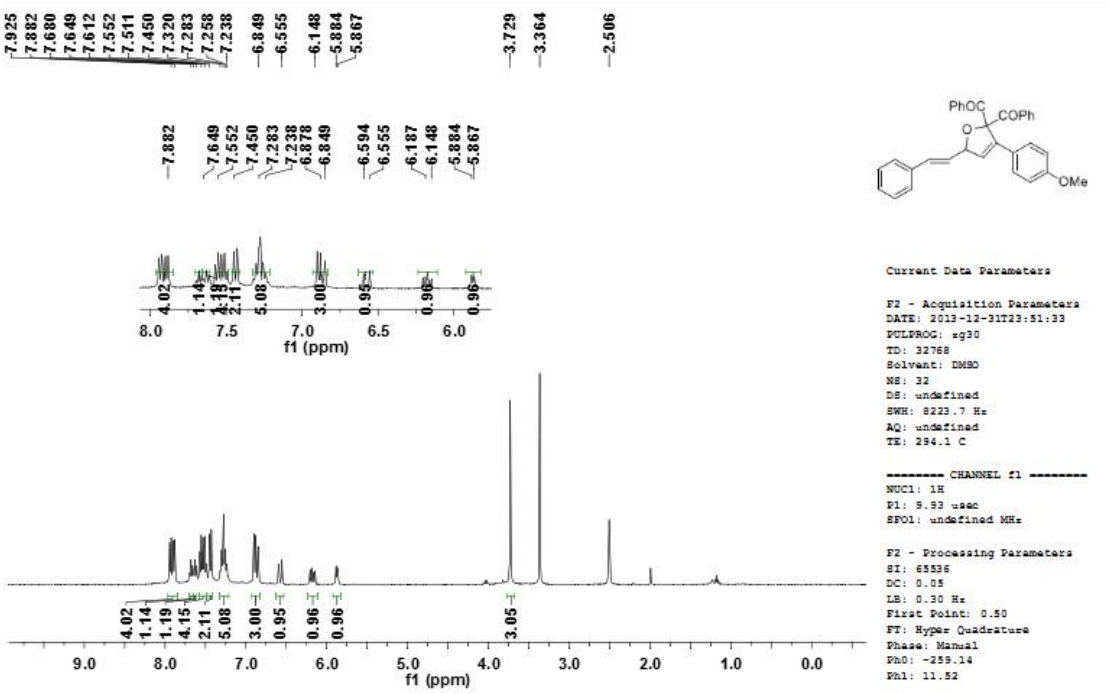


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.29 - 7.25 (m, 5H), 7.25 - 7.23 (m, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* = 2.0 Hz, 1H), 6.17 (d, *J* = 2.0 Hz, 1H), 3.75 (s, 3H), 2.41 (s, 3H), 2.32 (s, 3H).

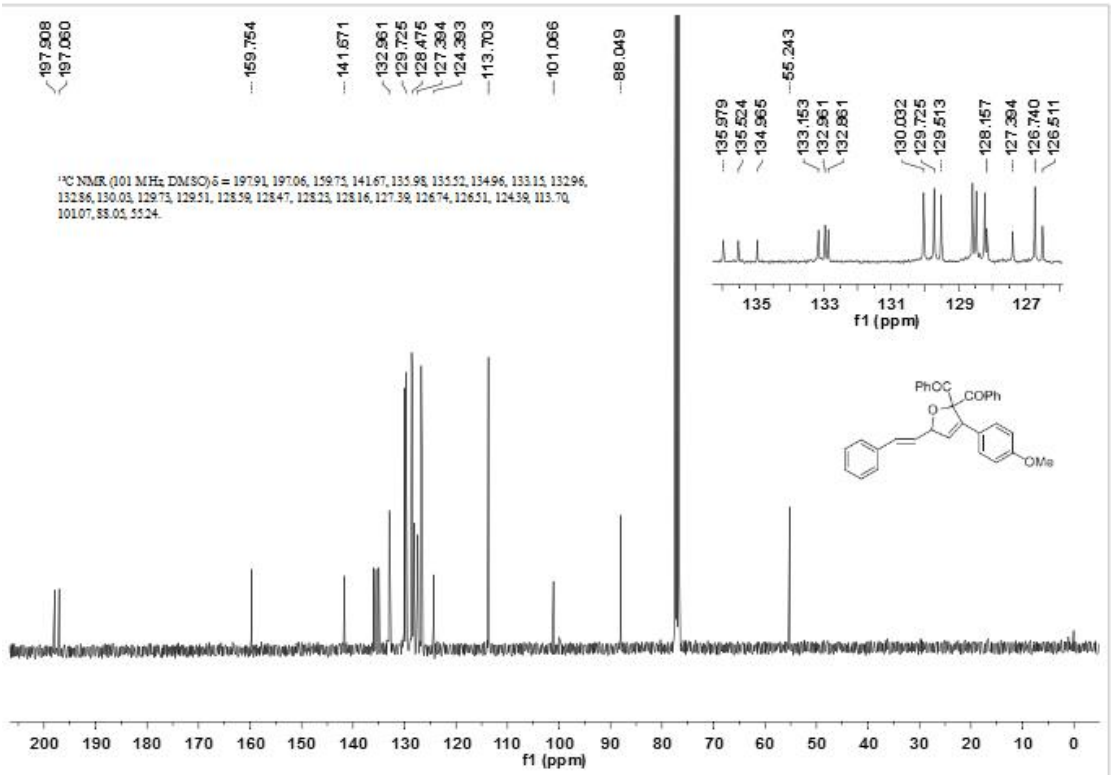

198.155
196.136
159.650
144.004
143.497
141.722
139.033
130.261
129.234
128.334
124.550
113.612
101.641
88.833
55.226
21.783
21.666
^{13}C NMR (101 MHz, CDCl$_3$) δ = 198.16, 196.14, 159.65, 144.00, 143.50, 141.72, 139.03, 133.29, 132.12, 130.26, 129.85, 129.43, 129.23, 128.84, 128.58, 128.33, 128.19, 127.17, 124.55, 113.61, 101.64, 88.83, 55.23, 21.78, 21.67.
f1 (ppm)
OMe

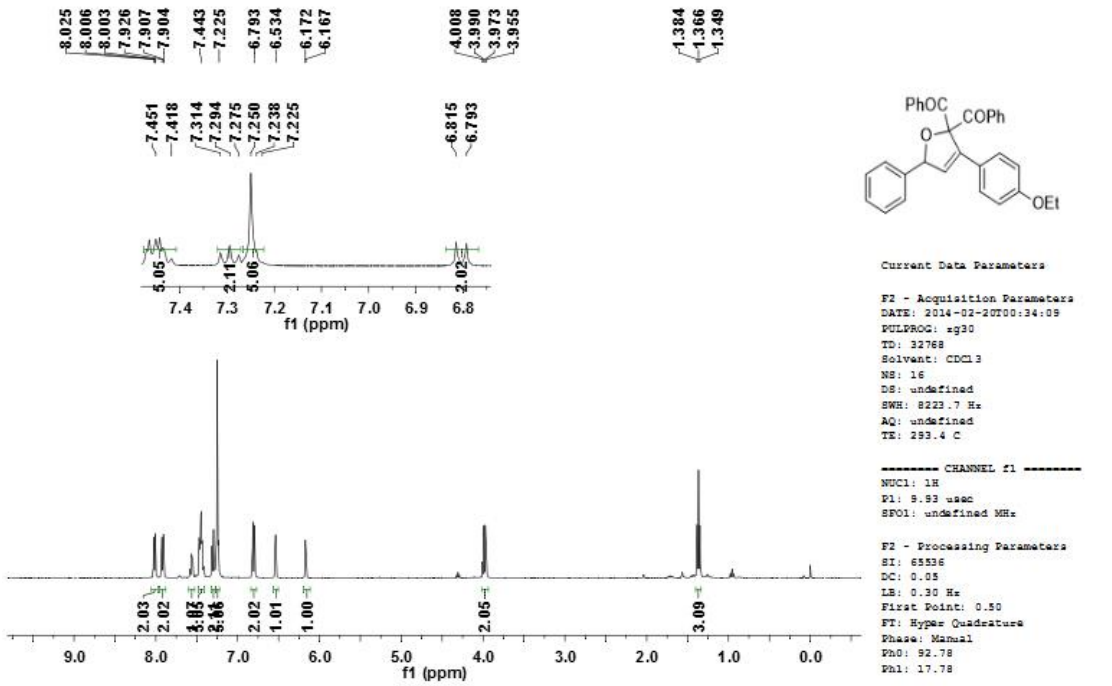

7.746
7.724
7.666
7.644
7.445
7.423
7.319
7.313
7.297
7.279
7.261
7.237
7.218
7.030
7.027
6.906
6.884
6.380
3.730
f1 (ppm)
Br
OMe
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2014-01-17T10:10:27
PULPROG: zg30
TD: 32768
Solvent: DMSO
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 291.2 C
CHANNEL f1
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -258.94
Ph1: 19.00



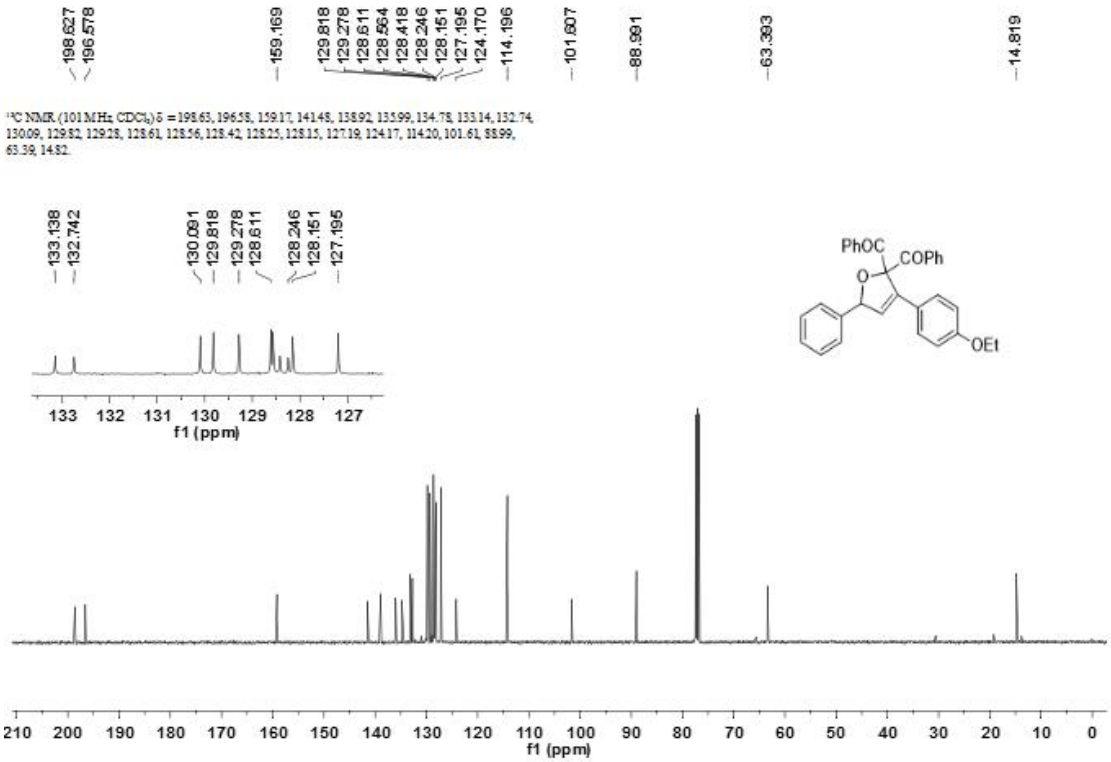


^{1}H NMR (400 MHz, DMSO) δ 7.97 - 7.85 (m, 4H), 7.71 - 7.66 (m, 1H), 7.65 - 7.61 (m, 1H), 7.58 - 7.49 (m, 4H), 7.46 - 7.41 (m, 2H), 7.33 – 7.21 (m, 5H), 6.93 - 6.83 (m, 3H), 6.63 - 6.53 (m, 1H), 6.24 - 6.11 (m, 1H), 5.88 (d, *J* = 6.8 Hz, 1H), 3.73 (s, 3H).

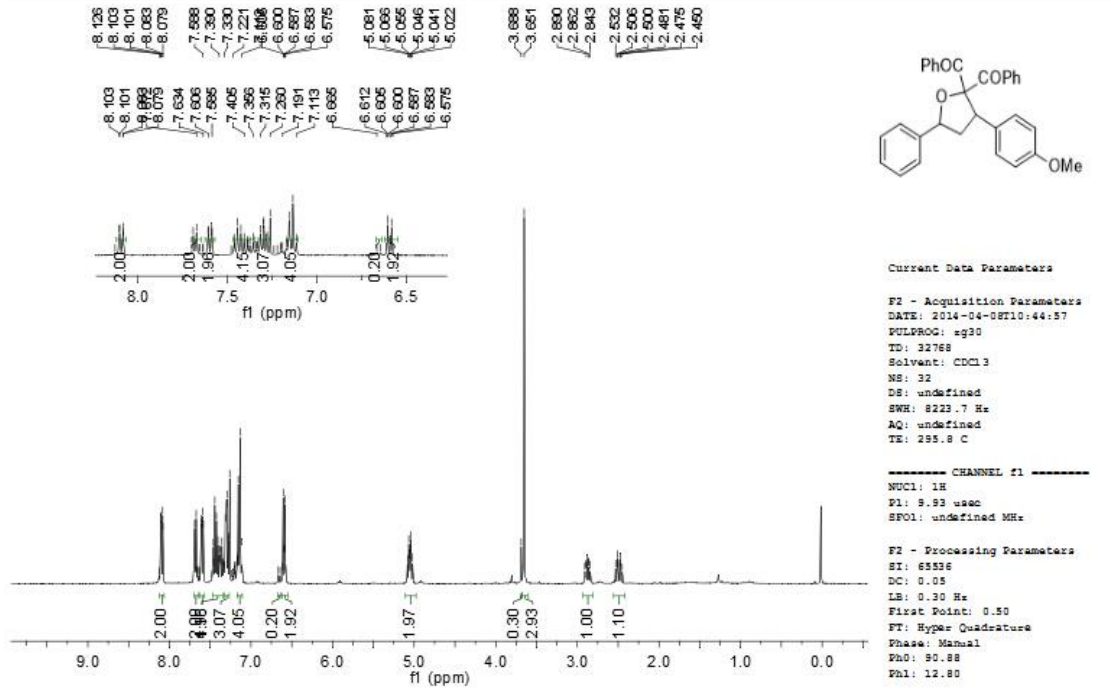




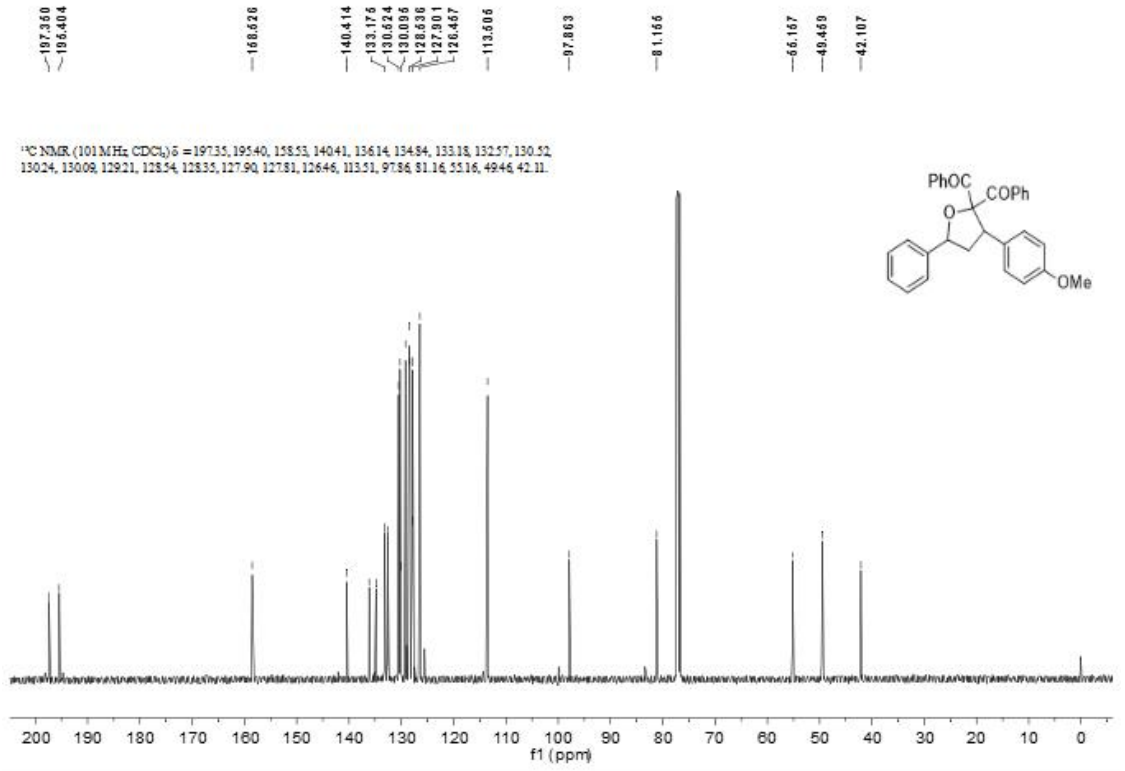

```
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2014-02-20T00:34:09
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 293.4 C

-------- CHANNEL f1 --------
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.78
Ph1: 17.78
```

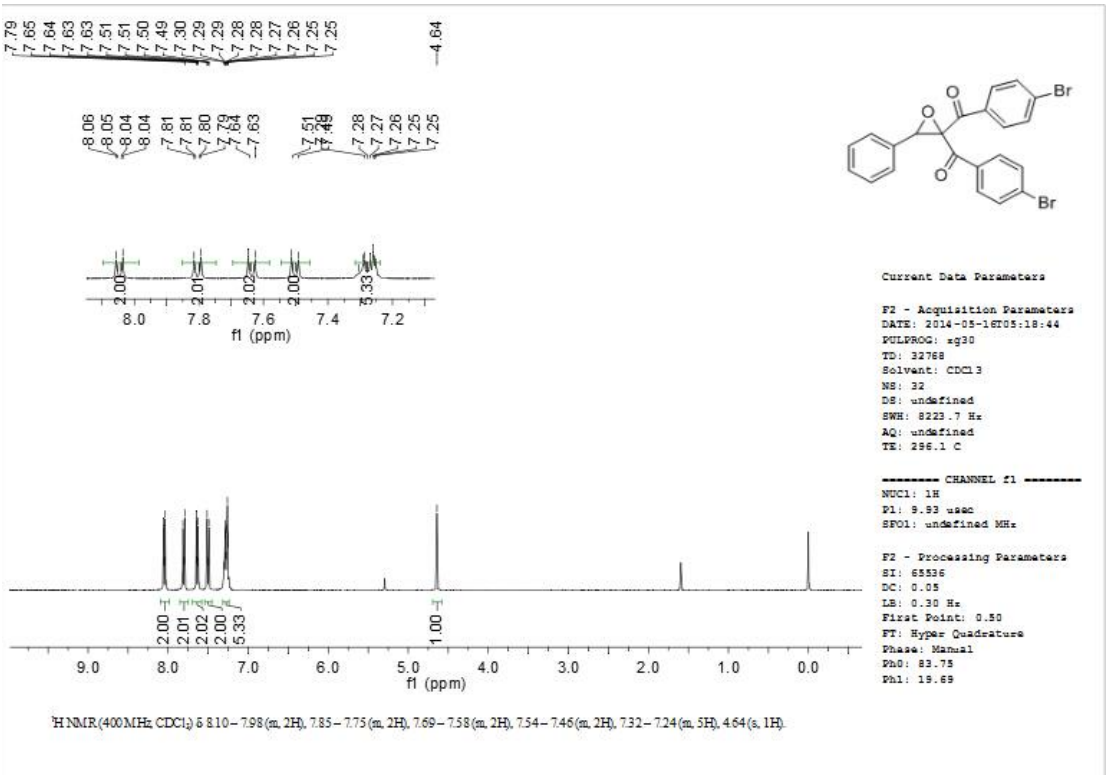


^{13}C NMR (101 MHz, $CDCl_3$) δ = 198.63, 196.58, 159.17, 141.48, 138.92, 135.99, 134.78, 133.14, 132.74, 130.09, 129.82, 129.28, 128.61, 128.56, 128.42, 128.25, 128.15, 127.19, 124.17, 114.20, 101.61, 88.99, 63.39, 14.82.

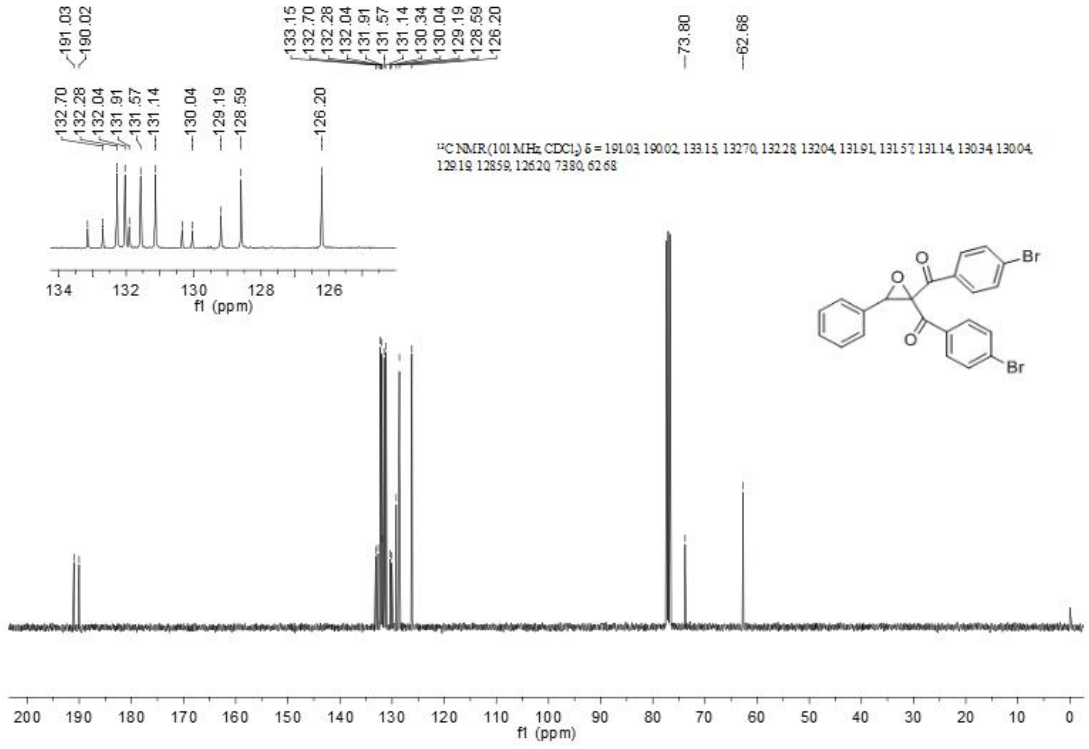


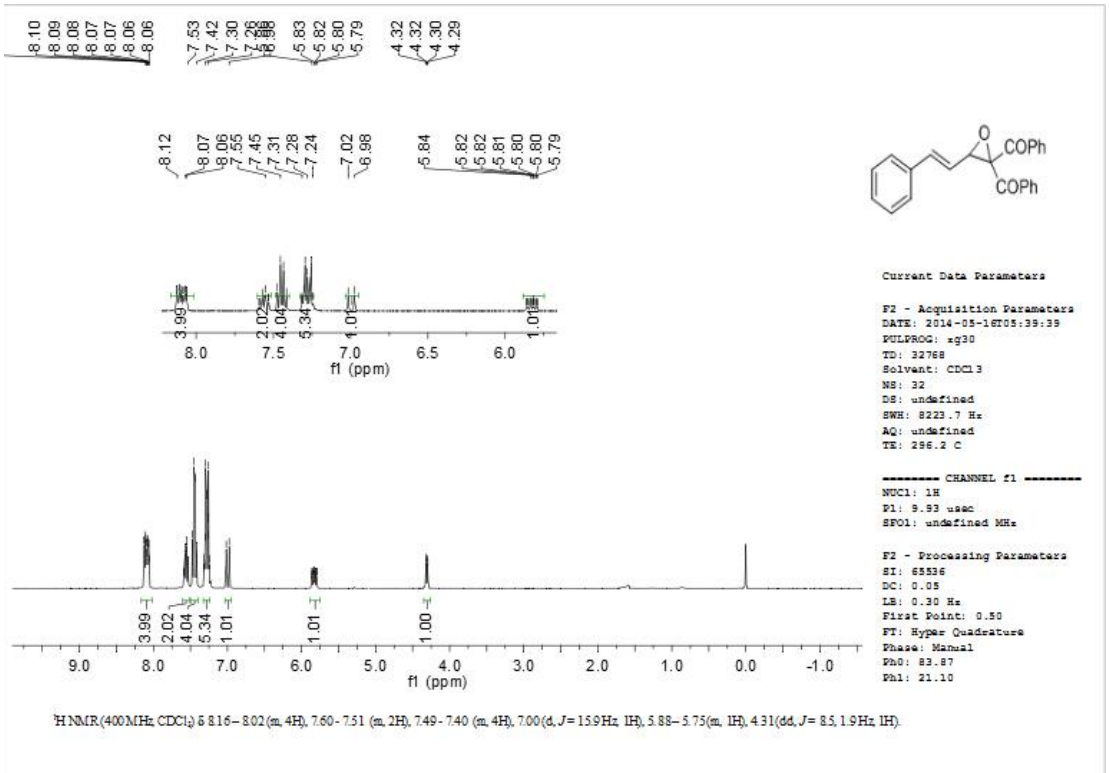
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 - 8.06 (m, 2H), 7.70 - 7.65 (m, 2H), 7.63 - 7.57 (m, 2H), 7.47 – 7.35 (m, 4H), 7.33 – 7.27 (m, 3H), 7.17 – 7.11 (m, 4H), 6.62 – 6.55 (m, 2H), 5.11 - 4.97 (m, 2H), 3.65 (s, 3H), 2.93 - 2.81 (m, 1H), 2.55 - 2.42 (m, 1H).



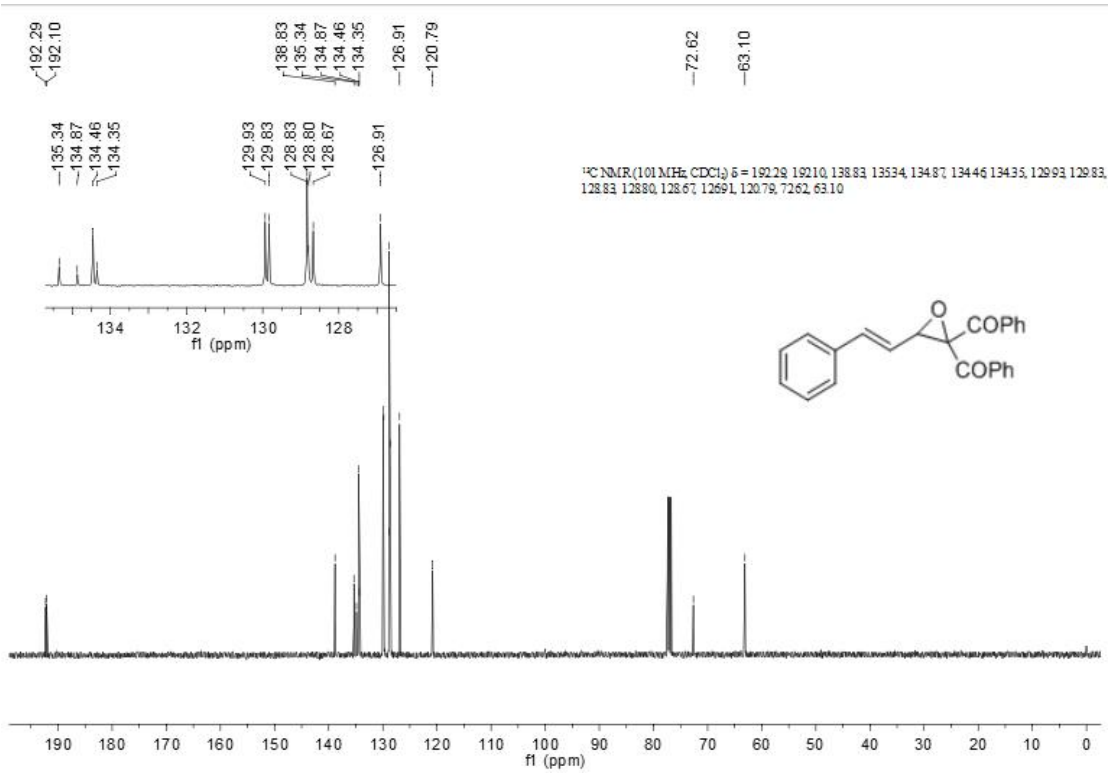


1H NMR (400 MHz, CDCl3) δ 8.10 – 7.98 (m, 2H), 7.85 – 7.75 (m, 2H), 7.69 – 7.58 (m, 2H), 7.54 – 7.46 (m, 2H), 7.32 – 7.24 (m, 5H), 4.64 (s, 1H).

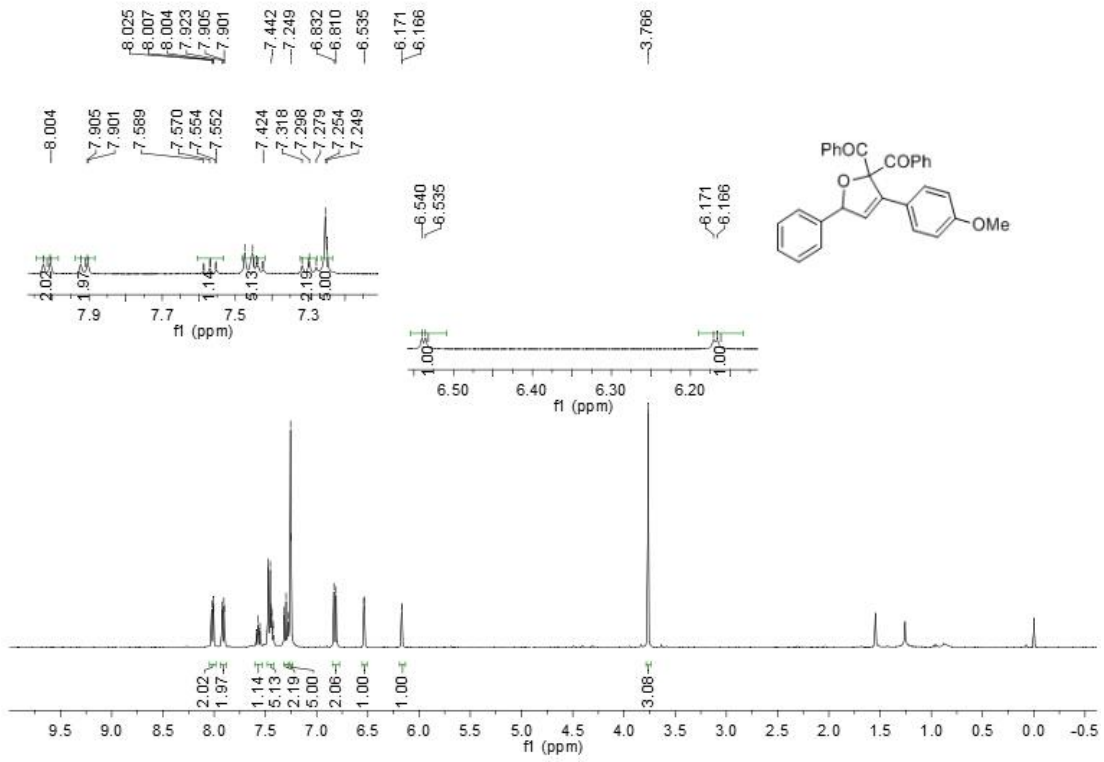




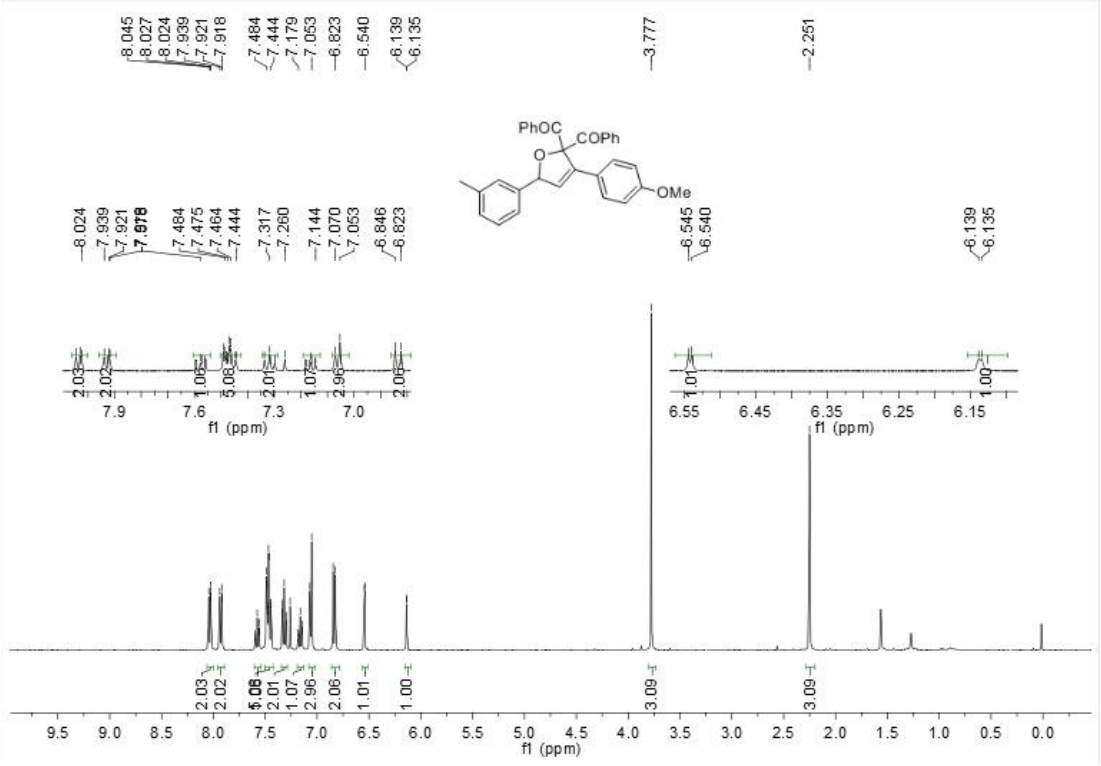
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.16 – 8.02 (m, 4H), 7.60 - 7.51 (m, 2H), 7.49 - 7.40 (m, 4H), 7.00 (d, *J* = 15.9 Hz, 1H), 5.88 – 5.75 (m, 1H), 4.31 (dd, *J* = 8.5, 1.9 Hz, 1H).



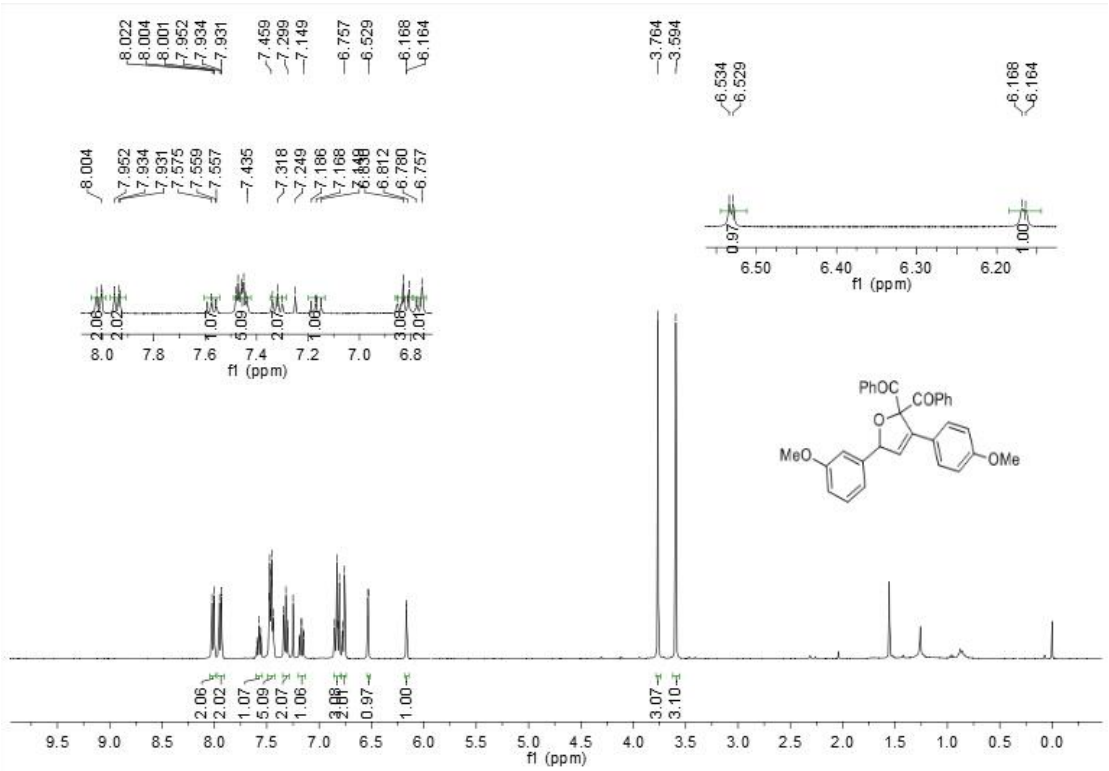
10. ^{1}H NMR spectra for some products in low concentration.



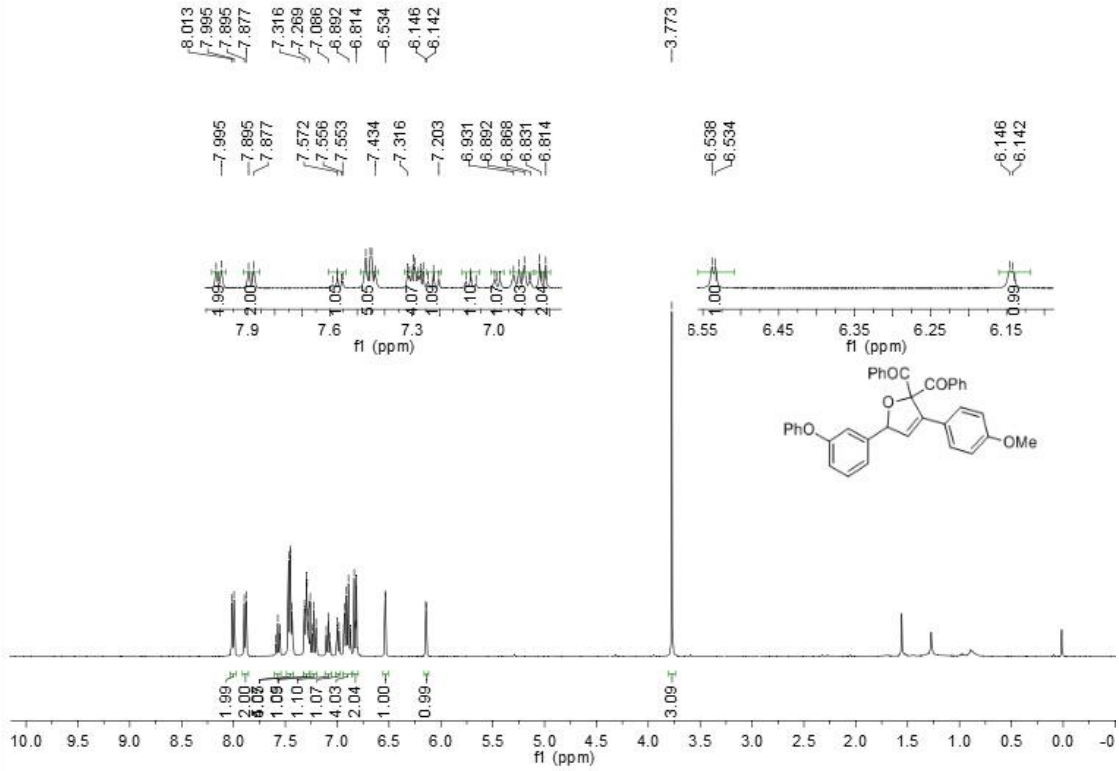
3a: ^{1}H NMR (400 MHz, $CDCl_3$): 8.05–7.98 (m, 2H), 7.94–7.88 (m, 2H), 7.61–7.53 (m, 1H), 7.49–7.42 (m, 5H), 7.33–7.27 (m, 2H), 7.27–7.23 (m, 5H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 2 Hz, 1H), 6.17 (d, *J* = 2 Hz, 1H), 3.77 (s, 3H) ppm.



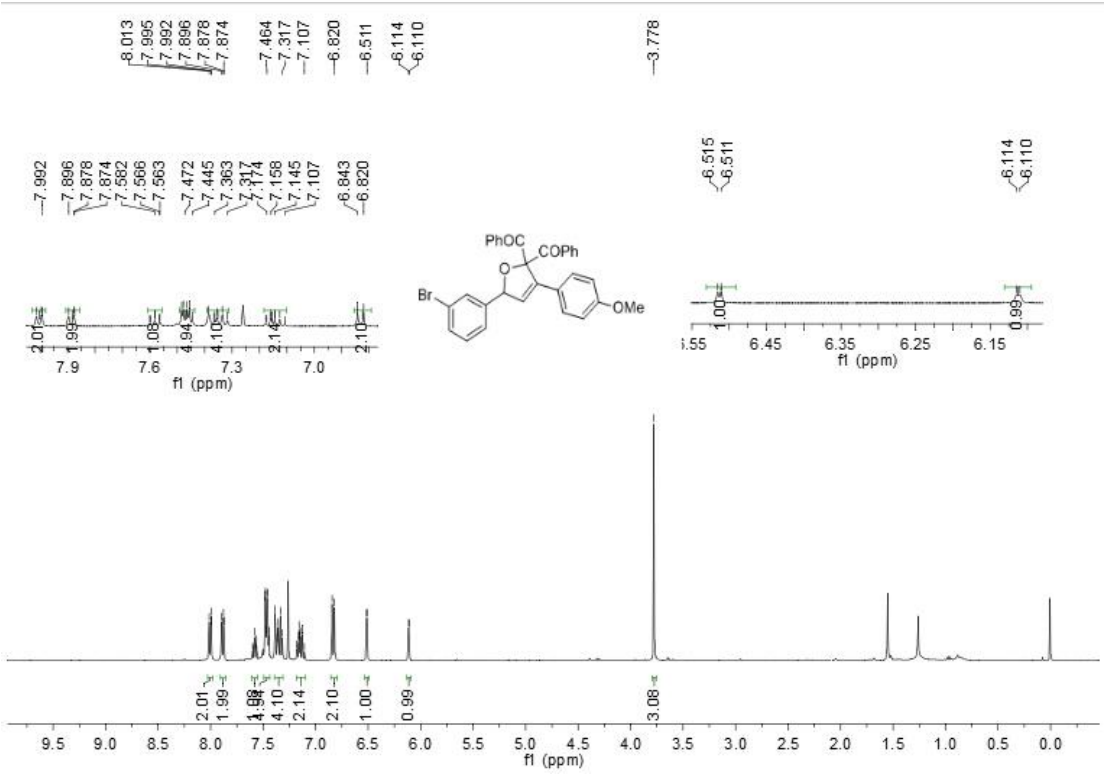
3e: ^{1}H NMR (400 MHz, $CDCl_3$): 8.06–8.00 (m, 2H), 7.96–7.89 (m, 2H), 7.61–7.54 (m, 1H), 7.50–7.42 (m, 5H), 7.34–7.29 (m, 2H), 7.19–7.13 (m, 1H), 7.08–7.02 (m, 3H), 6.83 (d, *J* = 9.2 Hz, 2H), 6.54 (d, *J* = 2 Hz, 1H), 6.13 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H), 3.09 (s, 3H) ppm.



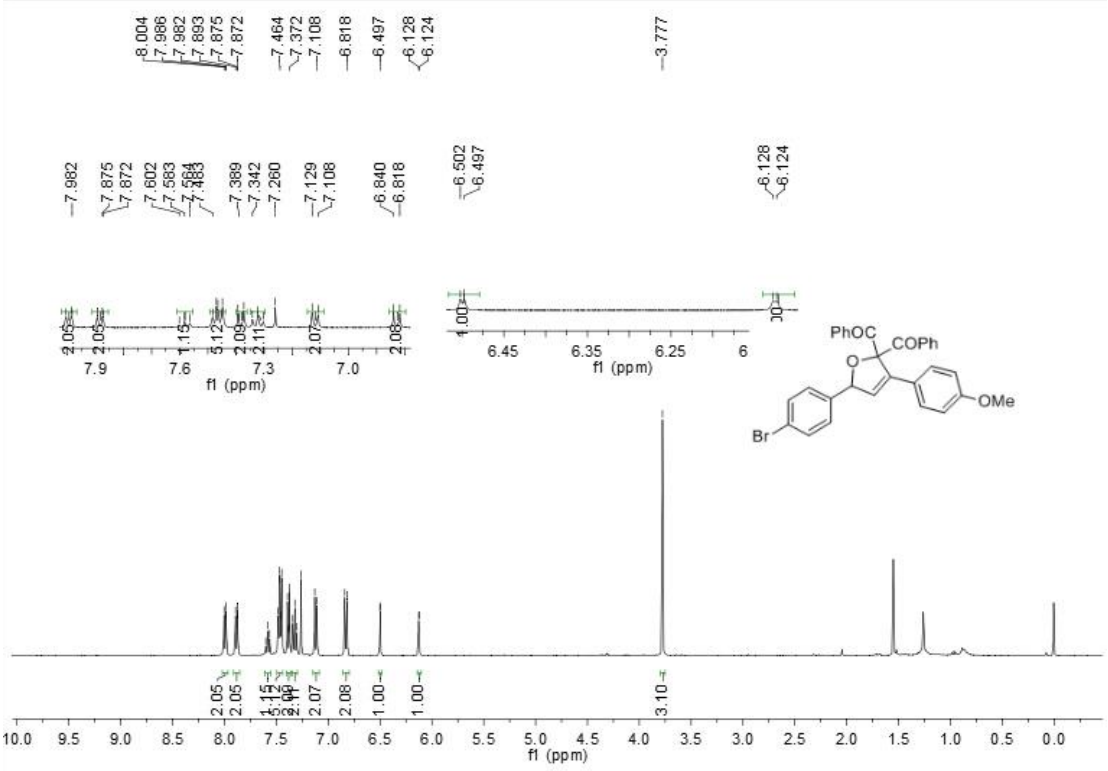
3c: ^{1}H NMR (400 MHz, $CDCl_3$): 8.05–7.99 (m, 2H), 7.97–7.91 (m, 2H), 7.61–7.54 (m, 1H), 7.49–7.42 (m, 5H), 7.35–7.29 (m, 2H), 7.20–7.13 (m, 1H), 6.86–6.80 (m, 3H), 6.79–6.74(m, 2H), 6.53 (d, *J* = 2 Hz, 1H), 6.17 (d, *J* = 1.6 Hz, 1H), 3.76 (s, 3H), 3.59 (s, 3H) ppm.



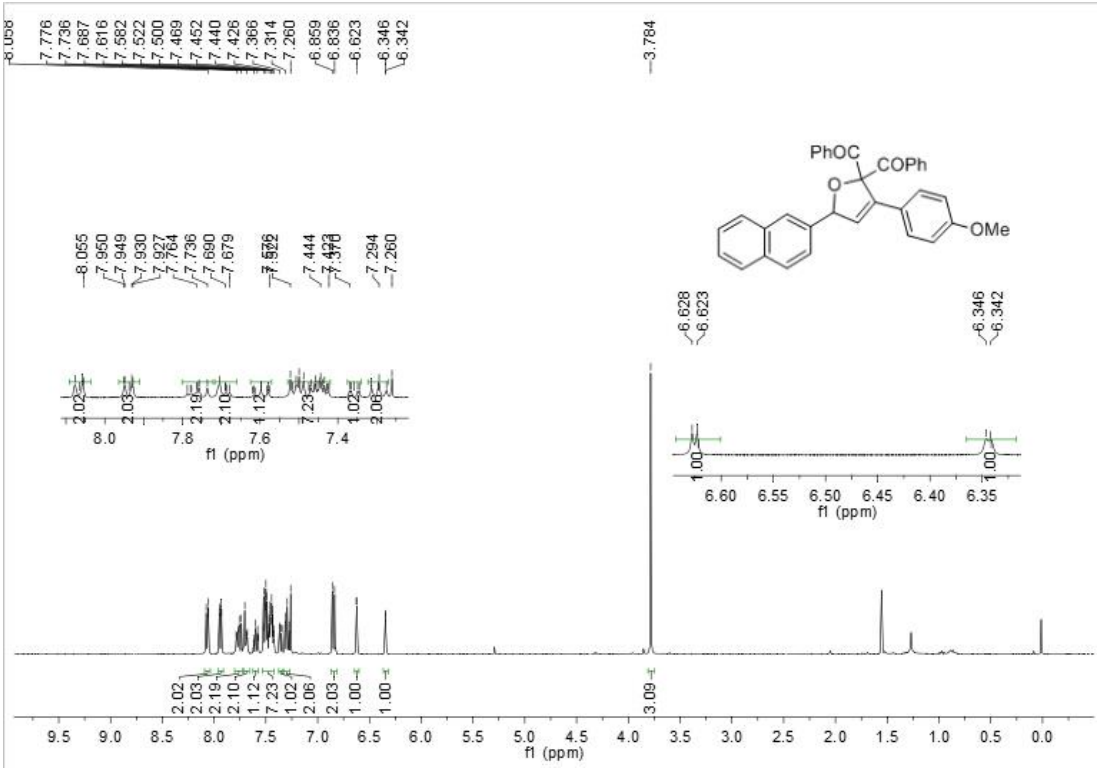
3d: ^{1}H NMR (400 MHz, $CDCl_3$): 8.03–7.98 (m, 2H), 7.91–7.85 (m, 2H), 7.61–7.54 (m, 1H), 7.49–7.42 (m, 5H), 7.33–7.27 (m, 2H), 7.25–7.20 (m, 1H), 7.09 (t, *J* = 7.8 Hz, 1H); 6.99 (d, *J* = 8.0 Hz, 1H), 6.95–6.86 (m, 4H), 6.82 (d, *J* = 8.8 Hz,, 2H), 6.54 (d, *J* = 1.6 Hz, 1H), 6.14 (d, *J* = 1.6 Hz, 1H), 3.77 (s, 3H) ppm.



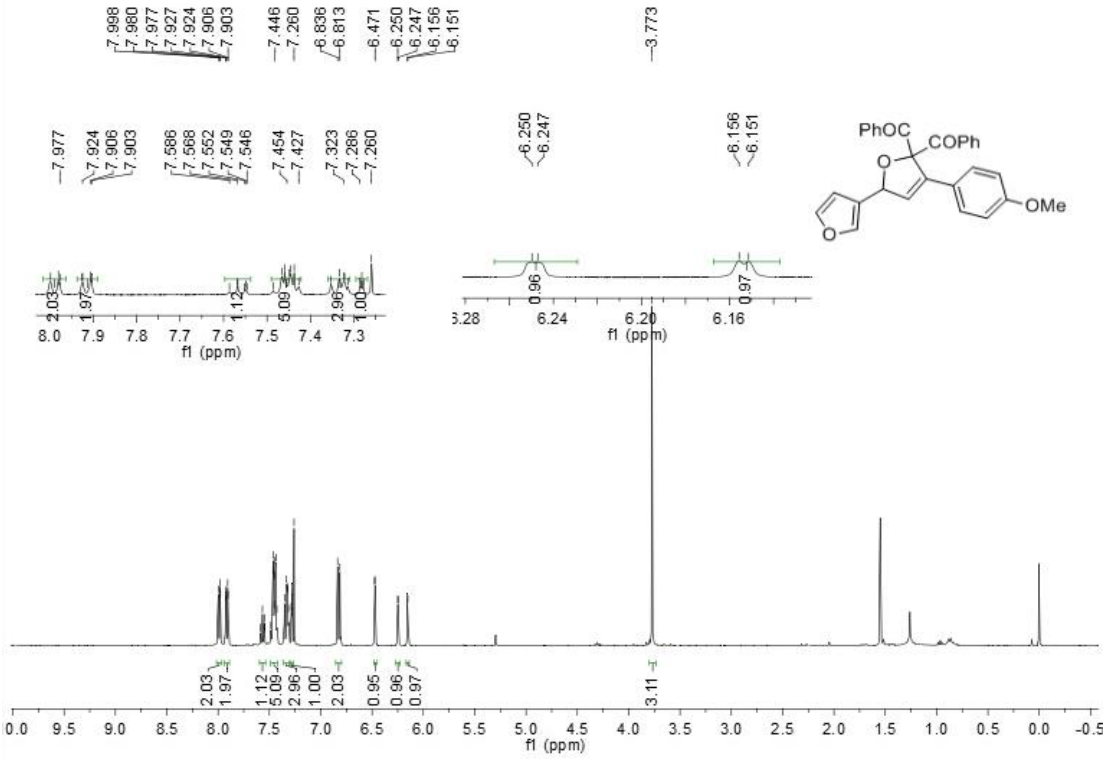
3h: ^{1}H NMR (400 MHz, $CDCl_3$): 8.01–7.98 (m, 2H), 7.91–7.85 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49–7.44 (m, 5H), 7.40–7.31 (m, 4H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* = 1.6 Hz, 1H), 6.13 (d, *J* = 1.6 Hz, 1H), 3.08 (s, 3H) ppm.



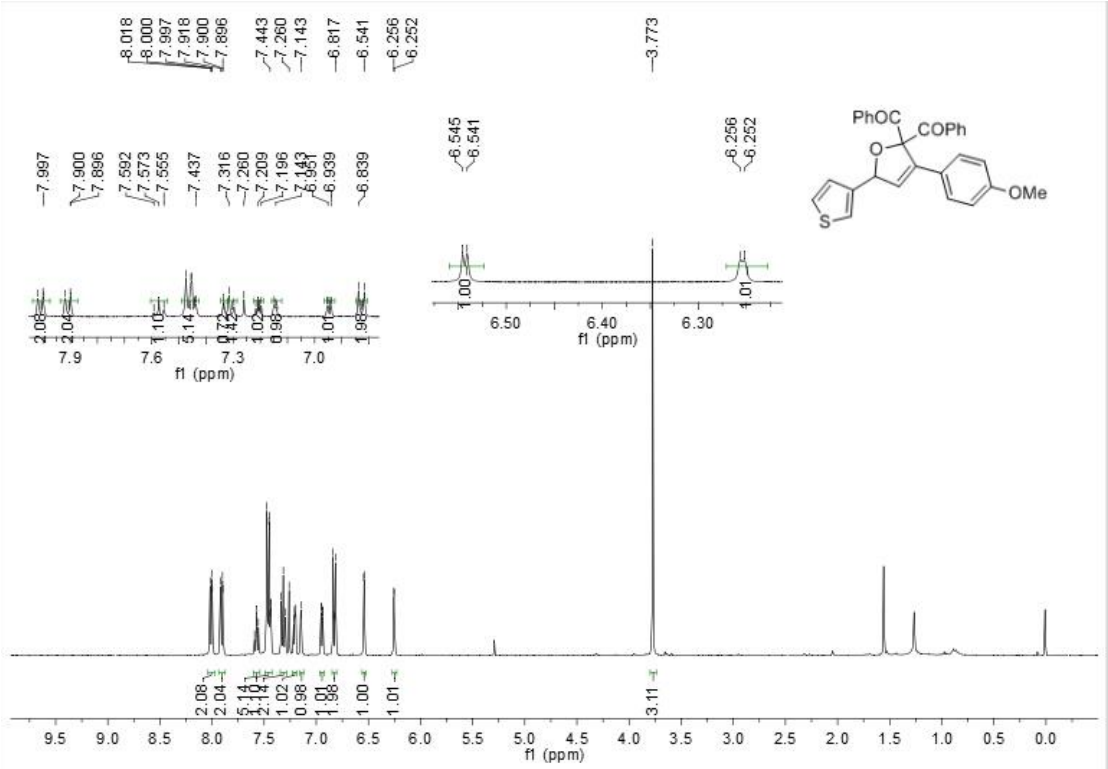
3l: ^{1}H NMR (400 MHz, $CDCl_3$): 8.02–7.97 (m, 2H), 7.91–7.85 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49–7.43 (m, 5H), 7.40–7.36 (m, 2H), 7.35–7.29 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.50 (d, *J* = 2 Hz, 1H), 6.12 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm.



3n: ^{1}H NMR (400 MHz, $CDCl_3$): 8.09–8.04 (m, 2H), 7.96–7.91 (m, 2H), 7.80–7.72 (m, 2H), 7.72–7.66 (m, 2H), 7.63–7.57 (m, 1H), 7.53–7.42 (m, 7H), 7.36 (dd, *J* = 8.8, 1.8 Hz, 1H), 7.32–7.27 (m, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 6.62 (d, *J* = 2 Hz, 1H), 6.34 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm.



3o: ^{1}H NMR (400 MHz, $CDCl_3$): 8.02–7.96 (m, 2H), 7.94–7.89 (m, 2H), 7.60–7.54 (m, 1H), 7.49–7.42 (m, 5H), 7.36–7.31 (m, 3H), 7.28 (t, *J* = 1.6 Hz, 1H), 6.82 (d, *J* = 9.2 Hz, 2H), 6.47 (d, *J* = 2 Hz, 1H), 6.25 (d, *J* = 1.2 Hz, 1H), 6.15 (d, *J* = 2 Hz, 1H), 3.78 (s, 3H) ppm.



3p: ^{1}H NMR (400 MHz, $CDCl_3$): 8.02–7.97 (m, 2H), 7.94–7.88 (m, 2H), 7.60–7.54 (m, 1H), 7.49–7.43 (m, 5H), 7.34–7.29 (m, 2H), 7.22–7.19 (m, 1H), 7.16–7.12 (m, 1H), 6.95 (dd, *J* = 5.2, 1.2 Hz, 1H) 6.83 (d, *J* = 8.8 Hz, 2H), 6.65 (d, *J* = 1.6 Hz, 1H), 6.25 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H) ppm.