# Supporting Information

## Efficient Direct Asymmetric Vinylogous Michael Addition Reactions of γ-Butenolides to Chalcones Catalyzed by Vicinal Primary-diamine Salts

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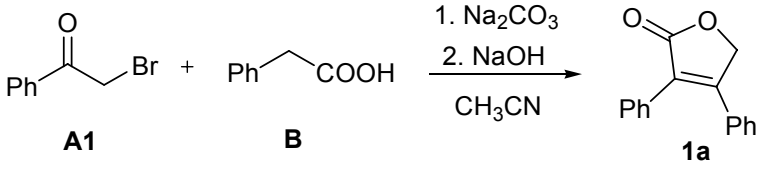
### General Information

Unless otherwise stated, all reagents were purchased from commercial suppliers and were used without further purification. 2(*5H*)-Furanones **1** that were not commercially available were prepared in our lab from substituted phenacyl bromide[1]. Reactions were monitored by thin layer chromatography (TLC) on $GF_{254}$ silica gel plates. $^{1}H$ NMR spectra and $^{13}C$ NMR spectra were recorded on a JEOL JNM-AL300 (300 MHz) spectrometer in needful D-reagents with tetramethylsilane (TMS) as an internal reference. Data for $^{1}H$ NMR are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, dd = double of doublet, br = broad, m = multiplet), coupling constants (Hz) and integration; Data for $^{13}C$ NMR are reported as ppm. Melting points were measured on an $X_4$-type micro-melting point apparatus and were uncorrected. Elemental analyses were performed on a Vario EL Ⅲ instrument. High-resolution mass spectra (HRMS) were obtained on a Bruker APEX IV FT_MS (7.0) spectrometer for electrospray ionization (ESI). HPLC analyses were performed using a Daicel ChiralPak AD column purchased. Crystal structure determination of the Michael product **3p** was carried out on a Bruker Smart Apex-II CCD diffractometer. Optical rotations were measured on a AA-10R automatic polarimeter and are reported as follows: $[\alpha]_D^{25}$ (*c* in g per 100 mL of solvent).

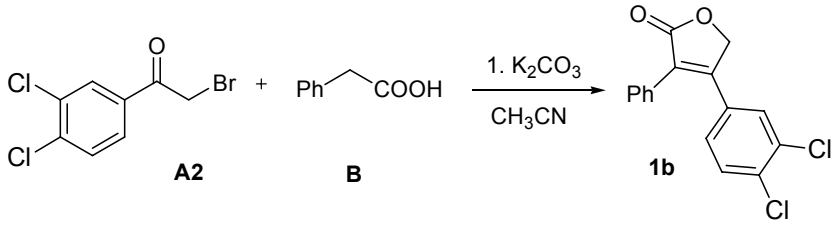
**Representative procedure for the synthesis of 2(*5H*)-Furanones 1:**

**Syntheis of 1a, 1c and 1d (Synthesis of 1a as the example):**




Phenacyl bromide **A1** (20.0 mmol) was added to a solution of phenylacetic acid **B** (20.0 mmol) in acetonitrile (30-50ml) containing sodium bicarbonate (24.0 mmol) at room temperature with stirring. The mixture was refluxed for the required time untill the disappearance of **A** monitored by TLC (silica gel, pet ether: ethyl acetate, 4:1). The mixture was added NaOH 2.0-4.0 mmol and refluxed until the completion of the reaction by TLC (silica gel, pet ether: ethyl acetate, 4:1). The resulting mixture was filtered off through a short column of silica gel and washed with ethyl acetate and pet ether (pet ether: ethyl acetate, 2:1). The solvent from the filtrate was removed under reduced pressure and the solid residue was purified by recrystallisation (pet ether: ethyl acetate, 4:1) to afford the pure product **1a**.

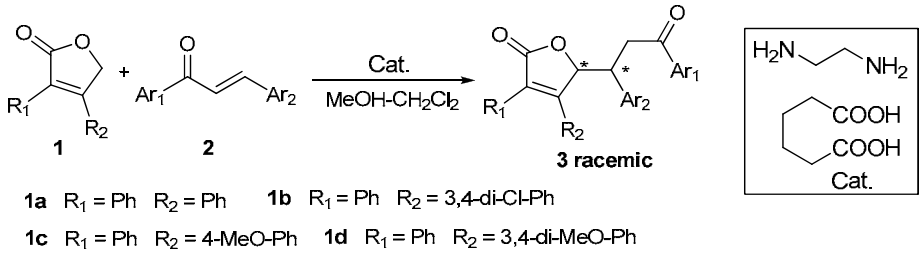
**Syntheis of 1b:**




The substituted phenacyl bromide **A2** (20.0 mmol) was added to a solution of phenylacetic acid **B** (20.0 mmol) in acetonitrile (30-50ml) containing potassium carbonate (30.0 mmol) at room temperature with stirring. The mixture was refluxed for the required time monitored by TLC (silica gel, pet ether: ethyl acetate, 4:1). The resulting mixture was filtered off through a short column of silica gel and washed with ethyl acetate and pet ether (pet ether: ethyl acetate, 2:1). The solvent from the filtrate was removed under reduced pressure and the solid residue was purified by

recrystallisation (pet ether: ethyl acetate, 4:1) to afford the pure product **1b**.

**Preparation for the racemic vinylogous Michael products:**

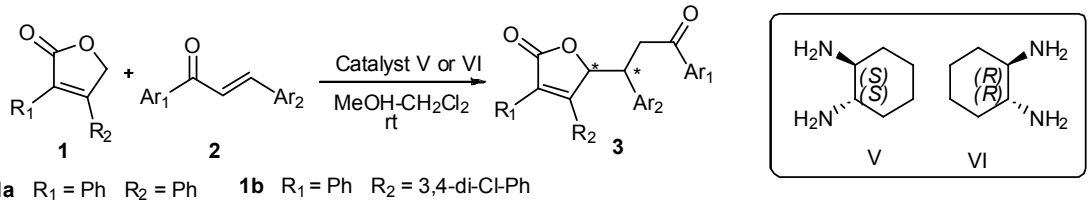



**1a** $R_1$ = Ph $R_2$ = Ph **1b** $R_1$ = Ph $R_2$ = 3,4-di-Cl-Ph

**1c** $R_1$ = Ph $R_2$ = 4-MeO-Ph **1d** $R_1$ = Ph $R_2$ = 3,4-di-MeO-Ph

To a mixture of **1a**, **1b, 1c or 1d** (0.5 mmol), a chalcone (0.6 mmol) and ethanediamine (0.1 mmol) in 2.0 ml of $CH_2Cl_2$ was added the acid additive (0.1 mmol of hexanedioic acid prepared in 1.0 ml of anhydrous $CH_3OH$). The mixture was refluxed for the required time monitored by TLC (silica gel, pet ether: ethyl acetate, 5:1) and then directly purified by filtration and/or preparative TLC chromatography to afford the pure products.

**General procedure for vinylogous Michael reaction:**



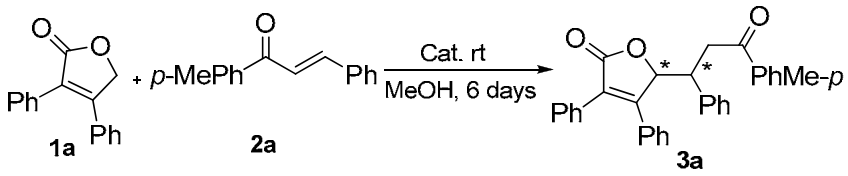

**1a** $R_1$ = Ph $R_2$ = Ph **1b** $R_1$ = Ph $R_2$ = 3,4-di-Cl-Ph

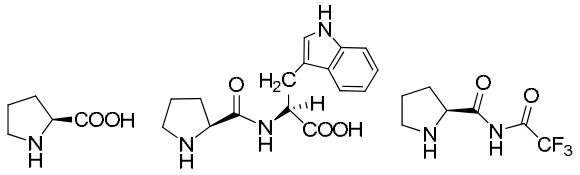
**1c** $R_1$ = Ph $R_2$ = 4-MeO-Ph **1d** $R_1$ = Ph $R_2$ = 3,4-di-MeO-Ph

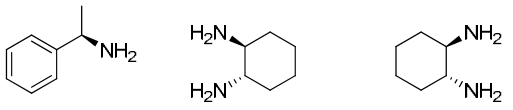
To a mixture of **1a**, **1b, 1c or 1d** (0.5 mmol), a chalcone (0.6 mmol) and chiral catalyst 1,2-diaminocyclohexane **V** or **VI** (0.1 mmol) in 0.8 ml of $CH_2Cl_2$ was added the acid additive (0.1 mmol of hexanedioic acid prepared in 0.4 ml of anhydrous $CH_3OH$). The resulting mixture was stirred under room temperature in dark for 6 days and then directly purified by filtration and/or preparative TLC chromatography to afford the pure products.

## Optimization of Reaction Conditions:

## Catalyst Screening



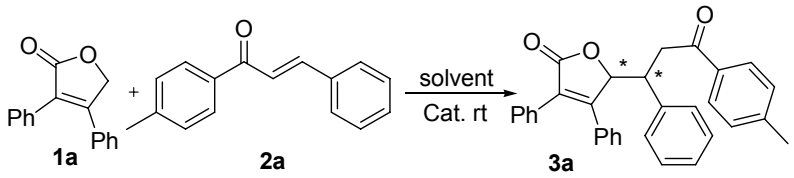




| Entry | Cat.[a] | Additive[a] | Yield (%)[c] | dr[d] | ee (%)[d] |
|---|---|---|---|---|---|
| 1 | I | - | N.R | - | - |
| 2 | III | - | Trace | - | - |
| 3 | I | TEA | - | - | - |
| 4 | II | TEA | Trace | - | - |
| 5 | IV | AcOH | 8 | - | 0 |
| 6 | V | AcOH[b] | **35** | 98:2 | **87** |
| 7 | V | Hexanedioic acid | **40** | 98:2 | **90** |
| 8 | V | TfOH | trace | - | - |
| 9 | V | - | trace | - | - |
| 10[e] | V | Hexanedioic acid | **69** | **>99:1** | **96** |

[a]The catalyst and additive loading was 20 mol%. [b]The additive loading of HOAc was 40 mol%. [c]Isolated yield of the corresponding product. [d]Determined by chiral-phase HPLC and/or $^1$H NMR. [e](1S, 2S)-1,2-diaminocyclohexane. [f]The reaction was conducted at room temperature in dark for 6 days; solvent: $CH_2Cl_2$:MeOH = 2:1.

## Acid Additive and Solvent Screening

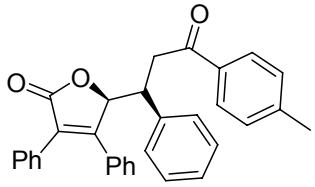



| Entry | Catalyst | Additive | Solvent | Time | Yield(%)[a] | dr (%)[b] | ee (%)[b] |
|---|---|---|---|---|---|---|---|
| 1 | V[c] | AcOH | MeOH | 6days | 35 | >98:2 | 87 |
| 2 | V | $CF_3COOH$ | MeOH | 6days | Trace | - | - |
| 3 | V | $HO_2CCH_2CO_2H$ | MeOH | 6days | 37 | >96:4 | 84 |
| 4 | V | $HO_2C(CH_2)_4CO_2H$ | MeOH | 6days | 40 | **>98:2** | **90** |
| 5 | V | $HO_2C(CH_2)_4CO_2H$ | EtOH | 6days | 28 | 90:10 | 86 |
| 6 | V | $HO_2C(CH_2)_4CO_2H$ | $CH_2Cl_2$ | 6days | - | - | - |
| 7 | V | $HO_2C(CH_2)_4CO_2H$ | $CHCl_3$ | 6days | - | - | - |
| 8 | V | $HO_2C(CH_2)_4CO_2H$ | THF | 6days | - | - | - |
| 9 | V | $HO_2C(CH_2)_4CO_2H$ | DMF | 6days | - | - | - |
| 10 | V | $HO_2C(CH_2)_4CO_2H$ | DMSO | 6days | - | - | - |
| 11 | V | $HO_2C(CH_2)_4CO_2H$ | $PhCH_3$ | 6days | - | - | - |
| 12 | V | $HO_2C(CH_2)_4CO_2H$ | MeOH $CH_2Cl_2$[d] | 7days | **69** | **>99:1** | **96** |

[a]Isolated yield of the corresponding product. [b]Determined by chiral-phase HPLC and/or H NMR. [c](1S, 2S)-1,2-diaminocyclohexane. [d]The reaction was conducted at room temperature in dark for 6 days; solvent: $CH_2Cl_2$:MeOH = 2:1.

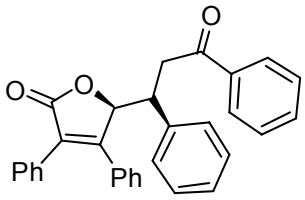
## Scope of the vinylogous Michael addition reaction

**5-(3-Oxo-1-phenyl-3-*p*-tolylpropyl)-3,4-diphenylfuran-2(5*H*)-one 3a:** obtained in 69% yield; white powder; m.p. 210-211℃; $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.95 (d, $J$ = 7.8 Hz, 2H), 7.40 (m, 3H), 7.30-7.18 (m, 10H), 6.96-6.88 (d, 4H), 6.01 (d, $J$ = 2.4 Hz, 1H), 4.09 (dd, $J$ = 10.0 Hz, 18.0 Hz, 1H), 3.88-3.84 (m, 1H), 3.41 (dd, $J$ = 4.0 Hz, 18.4 Hz, 1H), 2.43 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 198.2, 172.7, 158..9, 144.4, 136.5, 134.3, 130.7, 130.3, 130.0, 129.4, 128.9, 128.8, 128.8, 128.4, 128.4, 128.1, 128.0, 127.6, 127.5, 81.8, 42.5, 40.5, 21.6; Anal. Calcd for

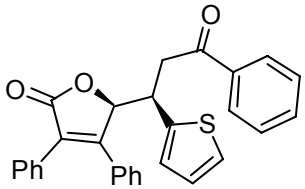


$C_{32}H_{26}O_3$: C 83.82, H 5.72; found: C 83.68, H 5.68; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.47 min (major enantiomer), 12.87 min (minor enantiomer); dr >99:1; ee 96%; $[\alpha]_D^{25}$ = +39 (*c* = 1.0, $CHCl_3$).

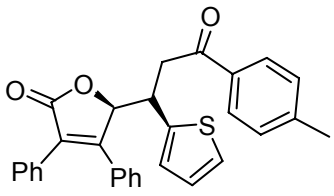
**5-(3-Oxo-1,3-diphenylpropyl)-3,4-diphenylfuran-2(5*H*)-one 3b:** Obtained in 65% yield; white powder; 210-211℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.04 (d, *J* = 7.8 Hz, 2H), 7.61 (tri, *J* = 6.6 Hz, 1H), 7.51 (tri, *J* = 7.2 Hz, 2H), 7.41 (d, *J* = 6.9 Hz, 3H), 7.30-7.19 (m, 8H), 6.96-6.88 (m, 4H), 6.01 (s, 1H), 4.11 (dd, *J* = 6.9 Hz, 18.0 Hz, 1H), 3.88-3.85 (m, 1H), 3.44 (dd, *J* = 3.9 Hz, 18.0 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 198.5, 172.6, 158.8, 136.6, 136.3, 133.5, 130.6, 130.3, 129.9, 128.8, 128.7, 128.4, 128.0, 127.5, 81.7, 42.4, 40.6; Anal. Calcd for $C_{31}H_{24}O_3$: C 83.76, H 5.44; found: C 83.32, H 5.33; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 7.19 min (major enantiomer), 11.26 min (minor enantiomer); dr >93:7; ee 91%; $[\alpha]_D^{25}$ = +33 (*c* = 1.3, $CHCl_3$).




**5-[3-oxo-3-phenyl-1-(thiophen-2-yl)propyl]-3,4-diphenylfuran-2(5*H*)-one 3c1:** Obtained in 62% yield; white powder; 206-207℃; $^1$H NMR (125 MHz, $CDCl_3$): $\delta$ 7.95 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.41 (d, *J* = 4.0 Hz, 1H), 7.32-7.22 (m, 8H), 7.16 (dd, *J* = 3.6 Hz, 8.4 Hz, 1H), 6.93 (d, *J* = 6.4 Hz, 4H), 5.97 (d, *J* = 2.0 Hz, 1H), 4.09 (dd, *J* = 4.4 Hz, 18.4 Hz, 1H), 3.82 (m, 1H), 3.42 (dd, *J* = 3.6 Hz, 18.4 Hz, 1H), 2.45 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 198.0, 171.9, 156.1, 144.5, 136.2, 134.6, 134.1, 133.4, 131.0, 130.6, 130.4, 129.4, 129.2, 129.1, 128.9, 128.8, 128.7, 128.2, 128.2, 127.9, 127.8, 81.5, 42.4, 40.4, 21.7; Anal. Calcd for $C_{29}H_{22}O_3S$: C 77.31, H 4.92; found: C 77.34, H 5.02; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 7.32 min (major enantiomer), 12.61 min (minor enantiomer); dr >99:1; ee 89%; $[\alpha]_D^{25}$ = +28 (*c* = 1.1, $CHCl_3$); **3c2:** HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 7.29 min (minor enantiomer), 12.73 min (major enantiomer); dr >95:5; ee -91%; $[\alpha]_D^{25}$ = -30 (*c* = 1.3, $CHCl_3$).

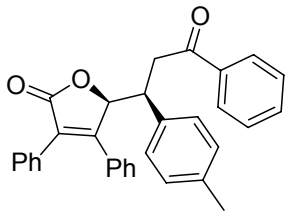


**5-[3-oxo-1-(thiophen-2-yl)-3-*p*-tolylpropyl]-3,4-diphenylfuran-2(*5H*)-one 3d:**

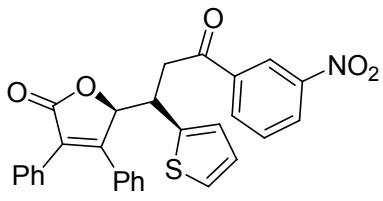


Obtained in 70% yield; white powder; 181-182℃; $^1$H NMR (125 MHz, $CDCl_3$): $\delta$ 7.95 (d, $J$ = 8.0 Hz, 2H), 7.44-7.38 (m, 3H), 7.36-7.34 (m, 2H), 7.32-7.26 (m, 2H), 7.16 (m, 1H), 7.11-7.08 (m, 2H), 6.87 (m, 1H), 6.56 (d, $J$ = 3.2 Hz, 1H), 5.98 (d, $J$ = 2.4 Hz, 1H), 4.24 (m, 1H), 4.00 (dd, $J$ = 10.4 Hz, 18.0 Hz, 1H), 3.44 (dd, $J$ = 4.0 Hz, 18.4 Hz, 1H), 2.44 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 197.7, 172.7, 158.7, 144.6, 138.4, 134.1, 129.5, 129.0, 128.9, 128.8, 128.5, 128.2, 127.9, 126.5, 126.3, 124.6, 81.2, 42.2, 38.2, 21.7; Anal. Calcd for $C_{30}H_{24}O_3S$: C 77.56, H 5.21; found: C 77.52, H 5.22; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.67 min (major enantiomer), 14.23 min (minor enantiomer); dr >99:1; ee 91%; $[\alpha]_D^{25}$ = +29 ($c$ = 0.9, $CHCl_3$).

**5-(3-Oxo-3-phenyl-1-*p*-tolylpropyl)-3,4-diphenylfuran-2(5*H*)-one 3e:** obtained in



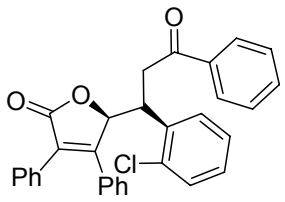
61% yield; white powder; m.p. 192-193℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.04 (d, J = 7.8 Hz, 2H), 7.60 (tri, $J$ = 6.9 Hz, 1H), 7.51 (tri, $J$ = 7.2 Hz, 2H), 7.41 (d, $J$ = 6.9 Hz, 3H), 7.31-6.98 (m, 5H), 7.01-6.98 (m, 4H), 6.78 (d, $J$ = 7.8 Hz, 2H), 5.99 (s, 1H), 4.09 (dd, $J$ = 9.9 Hz, 18.3 Hz, 1H), 3.85-3.81 (m, 1H), 3.42 (dd, $J$ = 3.3 Hz, 18.3 Hz, 1H), 2.29 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 198.6, 172.7, 159.0, 137.1, 136.7, 133.5, 133.3, 130.7, 130.3, 130.0, 128.9, 128.7, 128.4, 128.0, 127.5, 81.8, 42.0, 40.8, 21.0; Anal. Calcd for $C_{32}H_{26}O_3$: C 83.82, H 5.72; found: C 83.62, H 5.66; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 7.46 min (major enantiomer), 12.55 min (minor enantiomer); dr 96:4; ee 94%; $[\alpha]_D^{25}$ = +37 ($c$ = 1.2, $CHCl_3$).



**5-[3-(3-Nitrophenyl)-3-oxo-1-(thiophen-2-yl)propyl ]-3,4-diphenylfuran-2(*5H*)-one 3f:** obtained in 36% yield; white powder; m.p. 206-207℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.85 (s, 1H), 8.47 (d, $J$ = 6.9 Hz, 1H), 8.35 (d, $J$ = 6.6 Hz, 1H), 7.74 (tri, $J$ = 7.5 Hz, 1H), 7.42 (d, $J$ = 5.7 Hz, 2H), 7.32-7.26

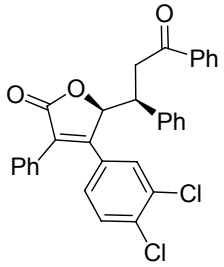
(m, 3H), 7.17 (d, *J* = 4.8 Hz, 2H), 7.09 (m, 2H), 6.86 (m, 1H), 6.54 (s, 1H), 5.96 (s, 1H), 4.21 (m, 1H), 4.06 (dd, *J* = 9.6 Hz, 18.3 Hz, 1H), 3.55 (dd, *J* = 4.2 Hz, 18.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 195.8, 172.4, 158.3, 148.4, 137.5, 133.5, 130.4, 130.2, 130.1, 129.7, 128.8, 128.6, 128.5, 128.4, 127.8, 126.6, 126.4, 124.8, 122.9, 80.9, 42.6, 37.9; Anal. Calcd for $C_{29}H_{21}NO_5S$: C 70.29, H 4.27, N 2.83; found: C 70.24, H 4.29, N 2.85; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 11.29 min (major enantiomer), 32.51 min (minor enantiomer); dr 93:6; ee 96%; $[\alpha]_D^{25}$ = +62 (*c* = 0.9, $CHCl_3$).

**5-(1-(2-Chlorophenyl)-3-oxo-3-phenylpropyl)-3,4-diphenylfuran-2(*5H*)-one 3g:**



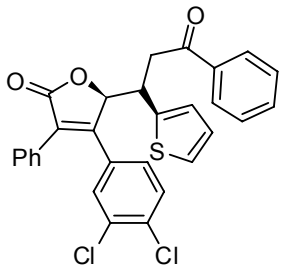

obtained in 63% yield; white powder; m.p. 179-180℃; $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.03 (d, *J* = 7.2 Hz, 2H), 7.62-7.47 (m, 4H), 7.40-7.30 (m, 3H), 7.28-7.22 (m, 6H), 7.14-7.12 (m, 2H), 7.08-7.05 (m, 2H), 6.17 (d, *J* = 3.2 Hz, 1H), 4.65-4.61 (m, 1H), 4.04 (dd, *J* = 10.0 Hz, 18.4 Hz, 1H), 3.33 (dd, *J* = 4.4 Hz, 18.4 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 197.9, 172.7, 159.2, 136.5, 135.1, 134.3, 133.5, 130.7, 130.3, 129.9, 129.6, 129.3, 128.9, 128.8, 128.7, 128.6, 128.6, 128.4, 128.4, 128.0, 127.3, 126.7, 81.7, 41.2, 37.3; Anal. Calcd for $C_{31}H_{23}ClO_3$: C 77.74, H 4.84; found: C 77.61, H 4.79; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 6.83 min (major enantiomer), 9.02 min (minor enantiomer); dr >99:1; ee 80%; $[\alpha]_D^{25}$ = +33 (*c* = 1.0, $CHCl_3$).

**4-[3,4-Dichlorophenyl)-5-(3-oxo-1,3-diphenylpropyl]-3-phenylfuran-2(*5H*)-one**

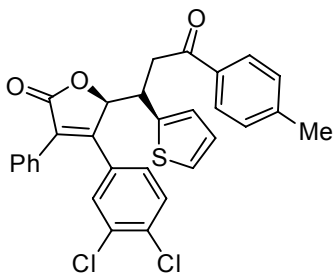



**3h:** obtained in 66% yield; white powder; m.p. 209-210℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.04 (d, *J* = 8.4 Hz, 2H), 7.62 (tri, *J* = 7.5 Hz, 1H), 7.51 (tri, *J* = 9.0 Hz, 3H), 7.39 (d, *J* = 1.5 Hz, 1H), 7.28-7.22 (m, 6H), 7.14 (dd, *J* = 1.5 Hz, 8.4 Hz, 1H), 6.92 (d, *J* = 6.0 Hz, 4H), 5.96 (d, *J* = 1.8 Hz, 1H), 4.11 (dd, *J* = 10.2 Hz, 18.6 Hz, 1H), 3.84-3.81 (m, 1H), 3.44 (dd, *J* = 3.3 Hz, 18.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 198.4, 171.9, 156.0, 136.5, 136.1, 134.6, 133.6, 133.4, 131.0, 130.6, 130.4,

130.3, 129.1, 128.9, 128.7, 128.7, 128.2, 128.0, 127.9, 81.4, 42.4, 40.5; Anal. Calcd for $C_{31}H_{22}Cl_2O_3$: C 72.52, H 4.32; found: C 72.33, H 4.25; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 9.07 min (major enantiomer), 15.13 min (minor enantiomer); dr 98:2; ee 95%; $[\alpha]_D^{25}$ = +40 (*c* = 1.1, $CHCl_3$).

**4-(3,4-dichlorophenyl)-5-[3-oxo-3-phenyl-1-(thiophen-2-yl)propyl]-3-phenylfuran-2(*5H*)-one 3i:** 

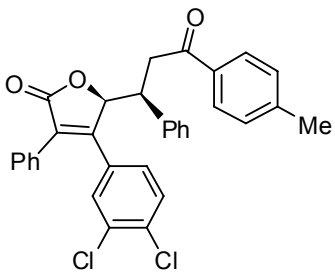
Obtained in 71% yield; white powder; 194-195℃; $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.04 (d, *J* = 7.6 Hz, 2H), 7.63 (tri, *J* = 8.0 Hz, 1H), 7.54-7.46 (m, 4H), 7.33-7.27 (m, 3H), 7.21-7.18 (m, 2H), 7.08-7.05 (m, 2H), 6.90 (m, 1H), 6.62 (d, *J* = 3.2 Hz, 1H), 5.93 (d, *J* = 2.0 Hz, 1H), 4.19 (dd, *J* = 2.8 Hz, 7.6 Hz, 1H), 4.06 (dd, *J* = 10.0 Hz, 18.0 Hz, 1H), 3.47 (dd, *J* = 4.4 Hz, 18.4 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 197.9, 171.9, 155.7, 137.9, 134.7, 133.8, 133.4, 131.0, 130.4, 129.4, 129.2, 129.1, 128.8, 128.8, 128.1, 128.0, 126.6, 126.6, 124.8, 80.9, 42.2, 38.0; Anal. Calcd for $C_{29}H_{20}Cl_2O_3S{\cdot}0.3H_2O$: C 66.36, H 3.96; found: C 66.52, H 3.92; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 8.63 min (major enantiomer), 17.48 min (minor enantiomer); dr = 97:3; ee 95%; $[\alpha]_D^{25}$ = +37 (*c* = 1.3, $CHCl_3$).

**4-(3,4-dichlorophenyl)-5-[3-oxo-1-(thiophen-2-yl)-3-*p*-tolylpropyl]-3-phenylfuran-2(*5H*)-one 3j:** 

Obtained in 74% yield; white powder; 221-223℃; $^1$H NMR (125 MHz, $CDCl_3$): $\delta$ 7.94 (d, *J* = 8.0 Hz, 2H), 7.48 (tri, *J* = 8.0 Hz, 1H), 7.32-7.29 (m, 5H), 7.21-7.18 (m, 2H), 7.07-7.05 (m, 2H), 6.90 (m, 1H), 6.62 (d, *J* = 2.8 Hz, 1H), 5.92 (d, *J* = 2.0 Hz, 1H), 4.19 (m, 1H), 4.03 (dd, *J* = 10.0 Hz, 18.0 Hz, 1H), 3.43 (dd, *J* = 3.6 Hz, 18.4 Hz, 1H), 2.45 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 197.6, 171.9, 155.7, 144.7, 137.9, 129.5, 129.4, 129.2, 129.0, 128.8, 128.7, 128.2, 128.0, 126.6, 126.5, 124.8, 80.9, 42.0, 38.0, 21.7, 1.03; Anal. Calcd for $C_{30}H_{22}Cl_2O_3S{\cdot}0.3H_2O$: C 66.87, H 4.23; found: C 66.86, H 4.18; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 15.64 min (major enantiomer),

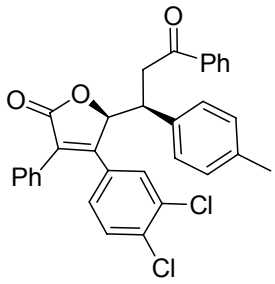
18.63 min (minor enantiomer); dr >99:1; ee 91%; $[\alpha]_D^{25}$ = +35 (*c* = 1.0, $CHCl_3$).

**4-(3,4-dichlorophenyl)-5-[3-oxo-1-phenyl-3-*p*-tolylpropyl]-3-phenylfuran-2(*5H*)-o**

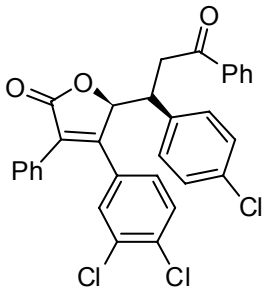



**ne 3k1:** Obtained in 75% yield; white powder; 205-206℃; $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.95 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.41 (m, 1H), 7.32-7.21 (m, 8H), 6.93 (dd, *J* = 1.6 Hz, 6.4 Hz, 1H), 5.96 (d, *J* = 2.4 Hz, 1H), 4.09 (dd, *J* = 10.4 Hz, 18.4 Hz, 1H), 3.81 (m, 1H), 3.42 (dd, *J* = 3.6 Hz, 18.0 Hz, 1H), 2.45 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 198.0, 172.0, 156.1, 144.5, 131.0, 130.4, 129.5, 129.2, 129.2, 128.9, 128.8, 128.7, 128.3, 128.2, 128.0, 127.9, 81.5, 42.5, 40.4, 21.7, 1.04; Anal. Calcd for $C_{32}H_{24}Cl_2O_3 \cdot 0.3H_2O$: C 72.13, H 4.65; found: C 72.11, H 4.61; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.93 min (major enantiomer), 12.23 min (minor enantiomer); dr >99:1; ee 96%; $[\alpha]_D^{25}$ = +35 (*c* = 1.0, $CHCl_3$); **3k2:** HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.06 min (minor enantiomer), 12.25 min (major enantiomer); dr >99:1; ee -95%; $[\alpha]_D^{25}$ = -33 (*c* = 1.2, $CHCl_3$).

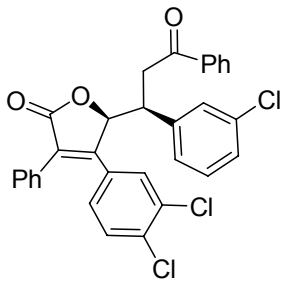
**4-(3,4-Dichlorophenyl)-5-(3-oxo-3-phenyl-1-*p*-tolylpropyl)-3-phenylfuran-2(*5H*)-**



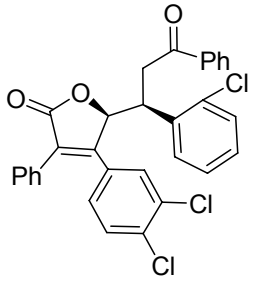

**one 3l:** obtained in 69% yield; white powder; m.p. 203-204℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.04 (d, *J* = 7.5 Hz, 2H), 7.61 (tri, *J* = 7.5 Hz, 1H), 7.51 (tri, *J* = 8.1 Hz, 3H), 7.41 (s, 1H), 7.27 (d, *J* = 6.6 Hz, 3H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.03 (d, *J* = 7.5 Hz, 2H), 6.95 (d, *J* = 6.9 Hz, 2H), 6.81 (d, *J* = 7.5 Hz, 2H), 5.93 (s, 1H), 4.07 (dd, *J* = 9.9 Hz, 18.3 Hz, 1H), 3.79 (d, *J* = 9.9 Hz, 1H), 3.41 (d, *J* = 18.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 198.5, 172.0, 156.1, 137.5, 136.6, 134.6, 133.6, 133.4, 133.0, 131.0, 130.7, 130.4, 129.2, 129.0, 128.9, 128.7, 128.6, 128.0, 127.9, 81.6, 42.0, 40.7, 21.0; Anal. Calcd for $C_{32}H_{24}Cl_2O_3$: C 72.87, H 4.59; found: C 72.65, H 4.70; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.00 min (major enantiomer), 17.44 min (minor enantiomer); dr >99:1; ee 96%; $[\alpha]_D^{25}$ = +43 (*c* = 1.0, $CHCl_3$).

**5-(1-(4-Chlorophenyl)-3-oxo-3-phenylpropyl)-4-(3,4-dichlorophenyl)-3-phenylfuran-2(*5H*)-one 3m:** 

Obtained in 78% yield; white powder; 180-182℃; $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.04 (d, $J$ = 7.2 Hz, 2H), 7.51 (tri, $J$ = 6.6 Hz, 1H), 7.63 (m, 1H), 7.54-7.49 (m, 3H), 7.44 (m, 1H), 7.31-7.27 (m, 3H), 7.22 (m, 2H), 7.14 (m, 1H), 6.98 (m, 2H), 6.88 (d, $J$ = 8.4 Hz, 2H), 5.93 (d, $J$ = 2.0 Hz, 1H), 4.05 (dd, $J$ = 10.0 Hz, 15.6 Hz, 1H), 3.82 (m, 1H), 3.42 (dd, $J$ = 4.0 Hz, 15.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 198.1, 171.7, 155.8, 133.8, 131.1, 130.5, 130.3, 130.1, 129.3, 129.1, 129.0, 128.8, 128.8, 128.7, 128.5, 128.1, 127.9, 81.3, 41.8, 40.6; Anal. Calcd for $C_{31}H_{21}Cl_3O_3 \cdot 0.3H_2O$: C 67.30, H 3.94; found: C 67.53, H 3.91; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.21 min (major enantiomer), 21.53 min (minor enantiomer); dr = 91:9; ee 92%; $[\alpha]_D^{25}$ = +31 ($c$ = 1.4, $CHCl_3$).

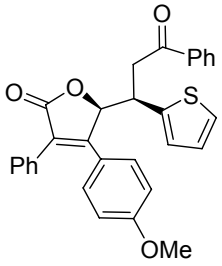
**5-(1-(3-Chlorophenyl)-3-oxo-3-phenylpropyl)-4-(3,4-dichlorophenyl)-3-phenylfuran-2(*5H*)-one 3n:** obtained in 78% yield; white powder; m.p. 181-182℃; $^1$H NMR 

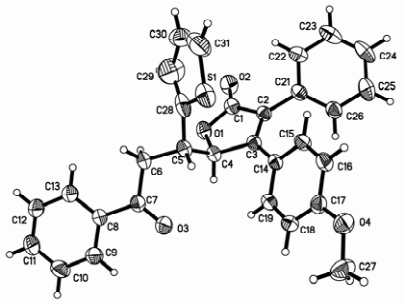
(300 MHz, $CDCl_3$): $\delta$ 8.04 (d, $J$ = 7.8 Hz, 2H), 7.63 (tri, $J$ = 7.5 Hz, 1H), 7.52 (tri, $J$ = 7.5 Hz, 3H), 7.40 (s, 1H), 7.29-7.21 (m, 5H), 7.17 (d, $J$ = 7.8 Hz, 1H), 6.98 (d, $J$ = 7.2 Hz, 2H), 6.90 (d, $J$ = 7.2 Hz, 1H), 6.83 (s, 1H), 5.92 (s, 1H), 4.06 (dd, $J$ = 7.2 Hz, 18.3 Hz, 1H), 3.81-3.78 (m, 1H), 3.45 (dd, $J$ = 3.9 Hz, 18.3 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 197.9, 171.6, 155.8, 138.1, 136.3, 134.8, 134.0, 133.7, 133.6, 131.1, 130.5, 130.3, 130.2, 129.5, 129.2, 129.1, 129.0, 128.8, 128.7, 128.7, 128.0, 127.9, 127.8, 126.6, 81.1, 42.0, 40.2; Anal. Calcd for $C_{31}H_{22}Cl_3O_3$: C 67.96, H 3.86; found: C 67.77, H 3.80; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 8.36 min (major enantiomer), 16.70 min (minor enantiomer); dr >95:5; ee 91%; $[\alpha]_D^{25}$ = +32 ($c$ = 1.2, $CHCl_3$).

**5-(1-(2-Chlorophenyl)-3-oxo-3-phenylpropyl)-4-(3,4-dichlorophenyl)-3-phenylfuran-2(*5H*)-one 3o:** 

obtained in 73% yield; white powder; m.p. 184-185℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 7.80 (d, $J$ = 7.5 Hz, 2H), 7.56 (tri, $J$ = 7.2 Hz, 1H), overlapping with 7.50 (d, $J$ = 7.5 Hz, 1H), 7.45-7.33 (m, 8H), 7.26-7.18 (m, 4H), 7.10 (dd, $J$ = 1.5 Hz, 8.1 Hz, 1H), 5.67 (d, $J$ = 2.7 Hz, 1H), 4.43 (m, 1H), 3.46 (dd, $J$ = 8.4 Hz, 15.4 Hz, 1H), 3.24 (dd, $J$ = 5.4 Hz, 15.4 Hz, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 196.1, 171.4, 155.9, 137.6, 136.1, 134.7, 133.5, 133.4, 133.3, 130.8, 130.1, 130.0, 130.0, 129.8, 129.3, 129.2, 129.1, 129.0, 128.7, 127.9, 127.6, 127.3, 82.7, 38.9, 35.9; Anal. Calcd for $C_{31}H_{21}Cl_3O_3$: C 67.96, H 3.86; found: C 67.78, H 3.76; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 9.66 min (major enantiomer), 13.18 min (minor enantiomer); dr >98:2; ee 83%; $[\alpha]_D^{25}$ = +44 ($c$ = 1.3, $CHCl_3$).

**4-(4-Methoxyphenyl)-5-(3-oxo-3-phenyl-1-(thiophen-2-yl)propyl)-3-phenylfuran-2(*5H*)-one 3p:**

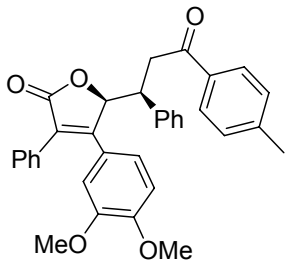


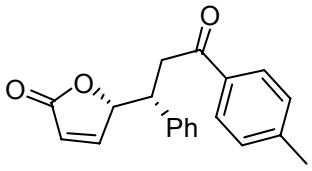

Obtained in 37% yield; white powder; m.p. 178-180℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 8.05 (d, $J$ = 7.2 Hz, 2H), 7.62 (tri, $J$ = 7.2 Hz, 1H), 7.51 (tri, $J$ = 7.5 Hz, 2H), 7.35-7.26 (m, 5H), 7.16 (d, $J$ = 3.9 Hz, 1H), 7.08-7.04 (m, 2H), 6.94-6.86 (m, 3H), 6.57 (d, $J$ = 3.3 Hz, 1H), 5.93 (d, $J$ = 2.4 Hz, 1H), 4.26-4.23 (m, 1H), 4.10 (dd, $J$ = 10.2 Hz, 18.3 Hz, 1H), 3.86 (s, 3H), 3.46 (dd, $J$ = 3.6 Hz, 18.3 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 198.3, 172.9, 161.2, 158.3, 138.2, 136.4, 133.7, 130.4, 130.3, 128.9, 128.8, 128.5, 128.3, 128.1, 126.4, 126.3, 125.9, 124.5, 122.7, 114.3, 80.9, 55.3, 42.2, 38.3; HRMS-ESI (*m/z*): [(M+Na)$^+$] calcd. for $C_{17}H_{22}O_7Na$ 503.1293, found 503.1298; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 10.79 min (major

enantiomer), 19.54 min (minor enantiomer); dr >99:1; ee 92%; $[\alpha]_D^{25}$ = +12.7 (*c* = 0.6, $CHCl_3$); HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 11.53 min (major enantiomer), 22.07 min (minor enantiomer); dr >99:1; ee 92%; $[\alpha]_D^{25}$ = -12.3 (*c* = 0.7, $CHCl_3$).

**4-(3,4-Fimethoxyphenyl)-5-(3-oxo-1-phenyl-3-*p*-tolylpropyl)-3-phenylfuran-2(*5H*)-one 3q:** obtained in 46% yield; white powder; m.p. 200-201℃; $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 7.96 (d, *J* = 7.8 Hz, 2H), 7.32-7.15 (m, 9H), 6.96-6.88 (m, 5H), 6.69 (s, 1H), 5.96 (s, 1H), 4.16 (dd, *J* = 10.8 Hz, 18.3 Hz, 1H), 3.99-3.95 (m, 1H) overlapping with 3.95 (s, 3H), 3.59 (s, 3H), 3.37 (dd, *J* = 3.0 Hz, 18.3 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 198.4, 172.9, 158.7, 150.8, 148.8, 144.4, 136.6, 134.3, 130.8, 129.4, 129.0, 129.0, 128.5, 128.2, 128.2, 128.0, 128.6, 126.0, 123.2, 122.1, 111.8, 111.1, 81.5, 55.9, 55.5, 42.9, 40.3, 21.7; Anal. Calcd for $C_{34}H_{35}O_5$: C 78.74, H 5.83; found: C 78.53, H 5.85; HPLC (AD, hexane:*i*-PrOH 50:50, 1.0 mL/min): $t_R$ 9.16 min (major enantiomer), 10.78 min (minor enantiomer); dr 98:2; ee 95%; $[\alpha]_D^{25}$ = +43 (*c* = 0.9, $CHCl_3$).




**5-(3-oxo-1-phenyl-3-p-tolylpropyl)furan-2(*5H*)-one 3r**: obtained in 67% yield; colorless oil; $^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 7.88 (d, *J* = 8.0 Hz, 2.52H), 7.78 (d, *J* = 8.0 Hz, 4.85H); 7.36-7.31 (m, 11.2H), 7.29-7.19 (m, 18.5H), 6.07 (dd, *J* = 2.0 Hz, 5.5 Hz, 2.01H), 5.85 (dd, *J* = 2.0 Hz, 5.5 Hz, 1.00H), 5.45 (p, *J* = 1.5 Hz, 1.14H), 3.27-2.26 (m, 2.38H); 3.97-3.94 (m, 1.28H), 3.78 (dd, *J* = 8.0 Hz, 17.5 Hz, 1.43H), 3.73-3.68 (m, 2.60H); 3.56-3.51 (m, 2.79H); 3.46-3.40 (m, 3.95H); 2.41 (s, 3.91H); 7.36 (s, 7.36); $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 197.4, 196.9, 172.7, 155.6, 155.3, 144.4, 144.2, 139.8, 137.3, 134.1, 129.4, 129.3, 128.9, 128.6, 128.4, 128.2, 128.1, 128.1, 127.6, 122.1, 121.9, 85.8, 84.4, 44.3, 43.0, 40.0, 39.8, 29.7, 21.7, 21.6.

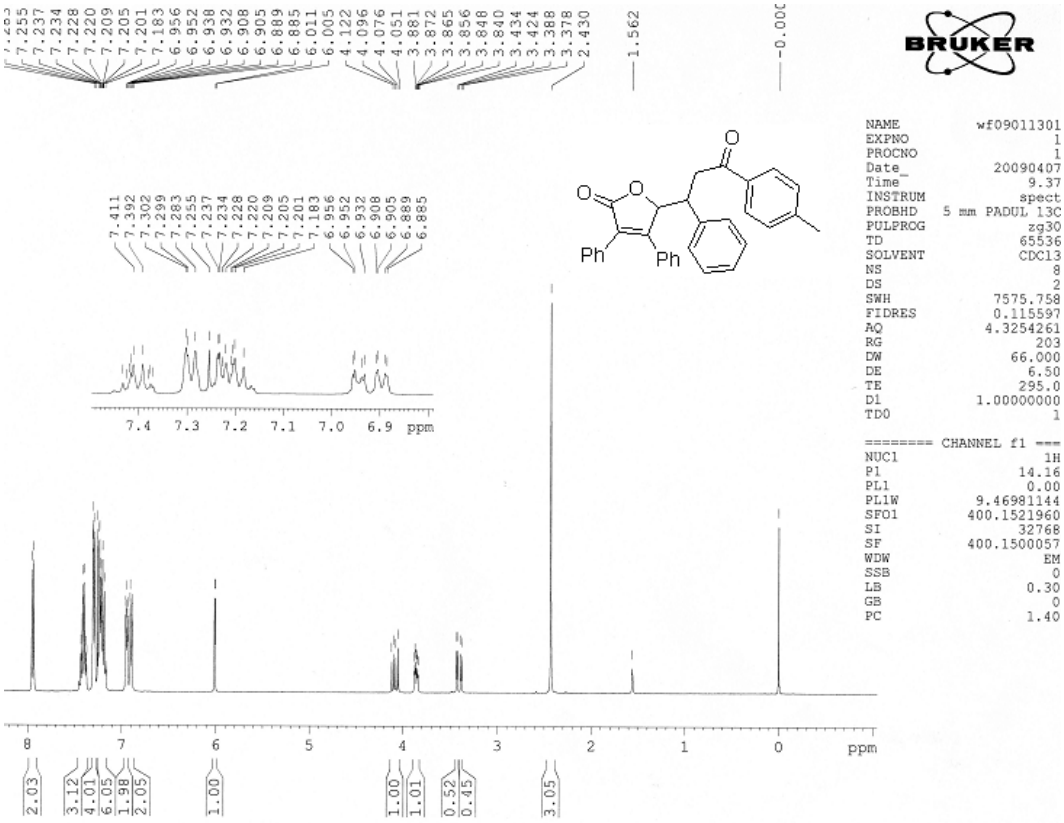


## Reference:

1. A. Zarghi et al. *Bioorg. Med. Chem*. **2007**, *15*, 1056.

# NMR spectra for Michael product 3

## 3a

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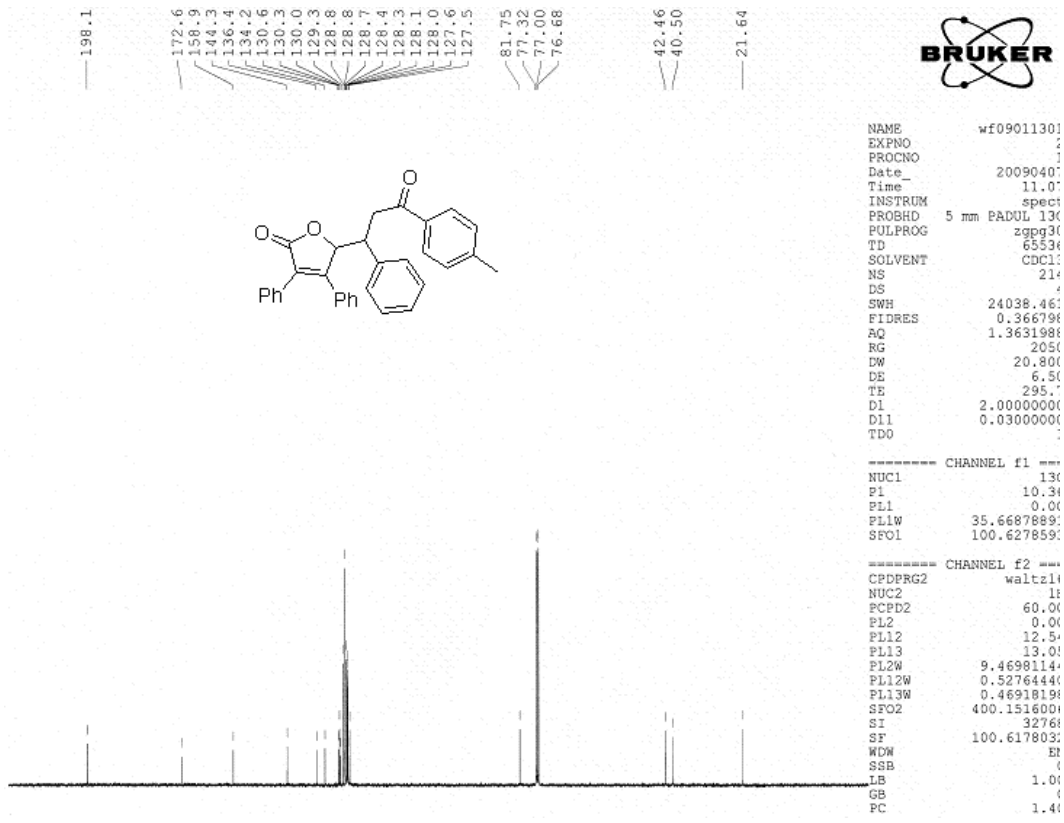


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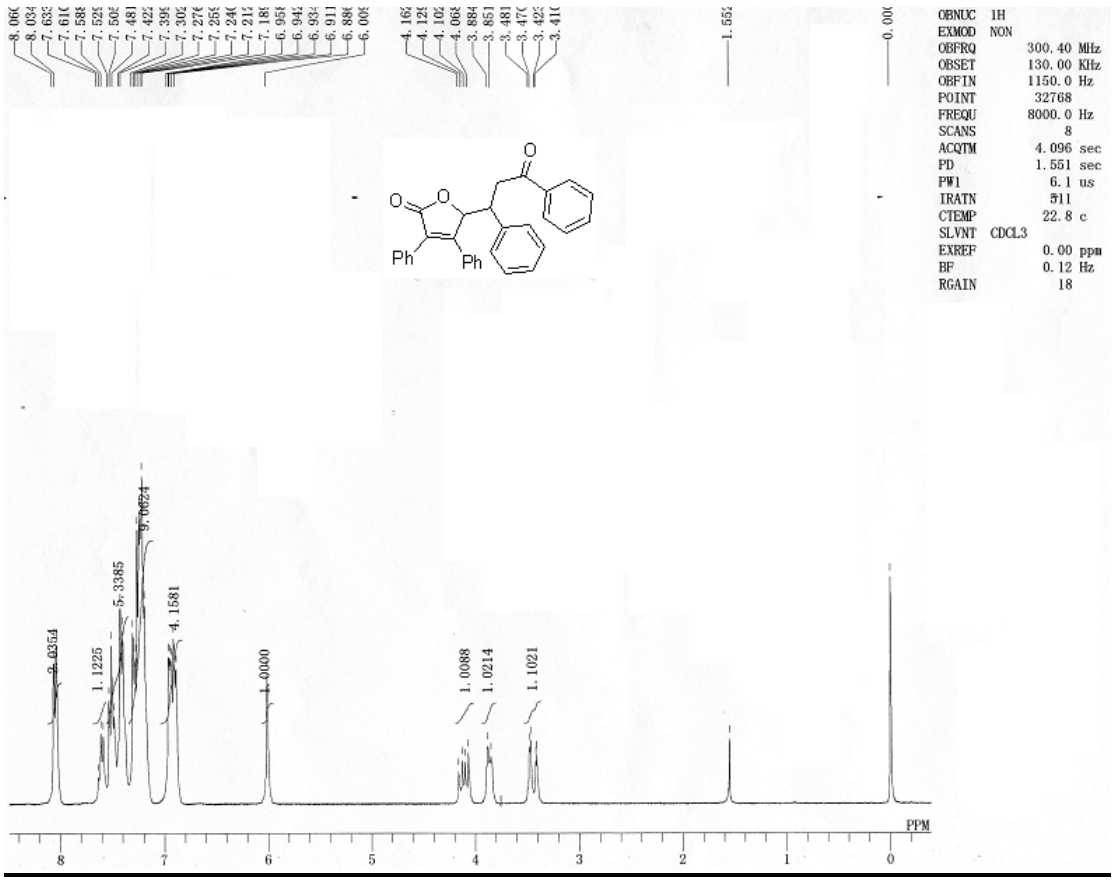
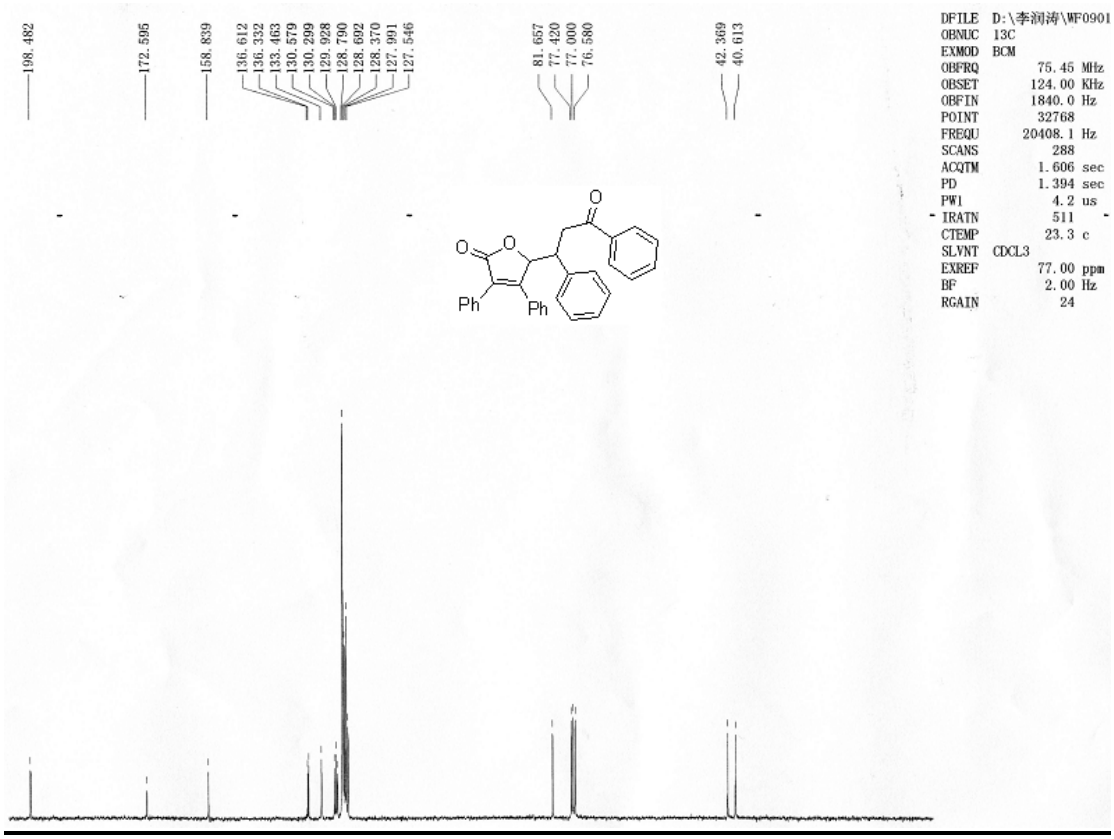
NUC1                 13
P1                10.3
PL1                0.0
PL1W         35.668788
SFO1         100.62785

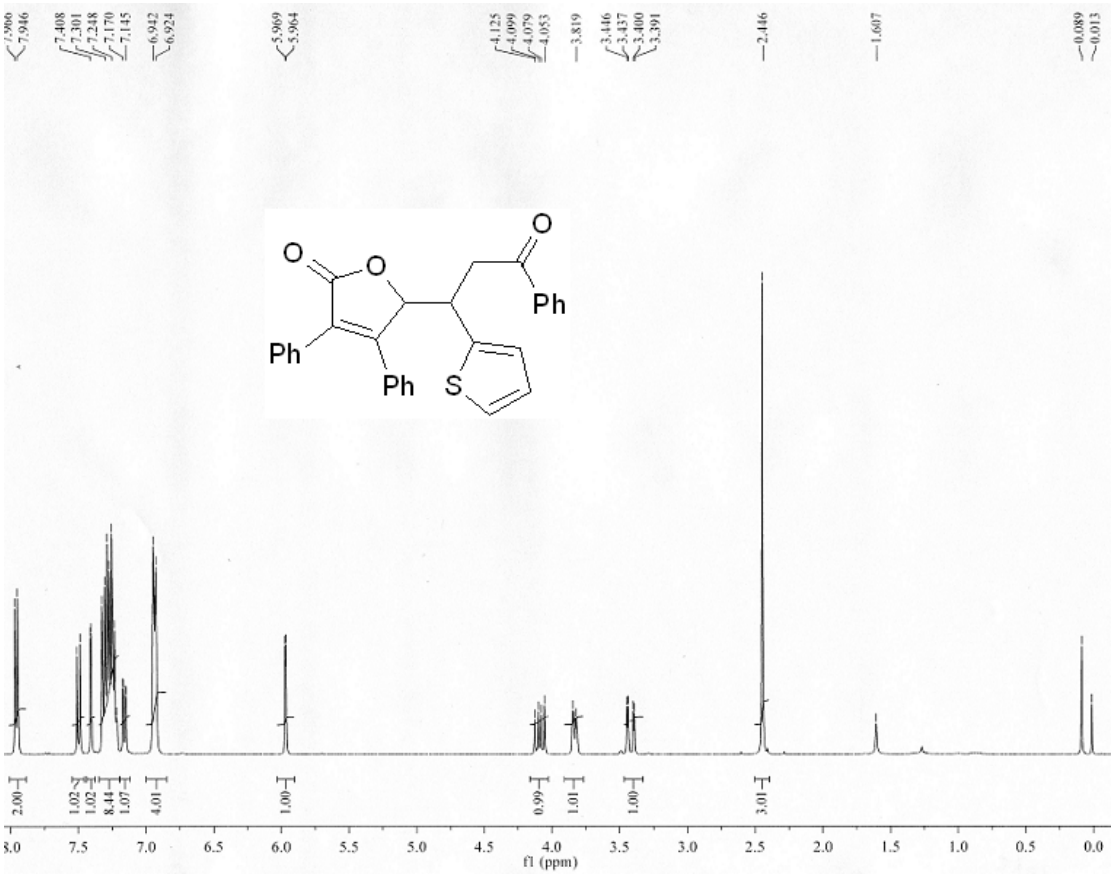
======== CHANNEL f2 ===
CPDPRG2          waltz1
NUC2                 1
PCPD2             60.0
PL2                0.0
PL12              12.5
PL13              13.0
PL2W          9.469811
PL12W         0.527644
PL13W         0.469181
SFO2         400.15160
SI                3276
SF           100.61780
WDW                  E
SSB                  0
LB                1.0
GB                   0
PC                1.4
```

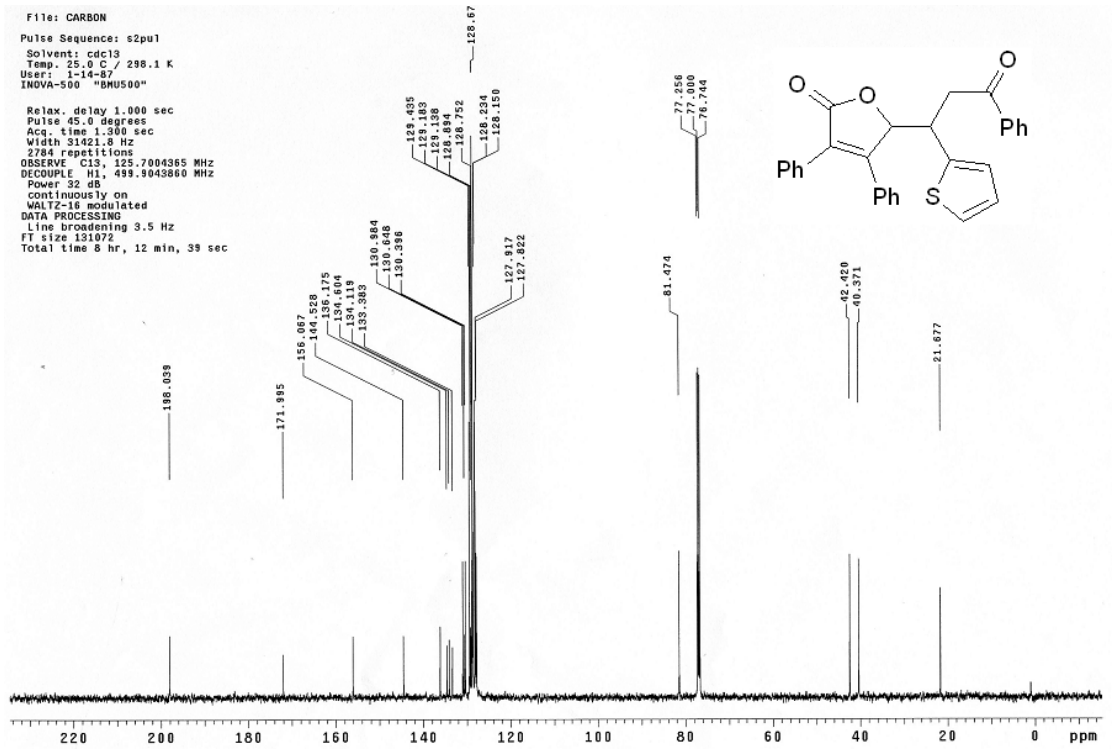
**3b**


OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18
Ph
Ph
2.0354
1.1225
5.3385
9.0624
4.1581
1.0000
1.0088
1.0214
1.1021
PPM

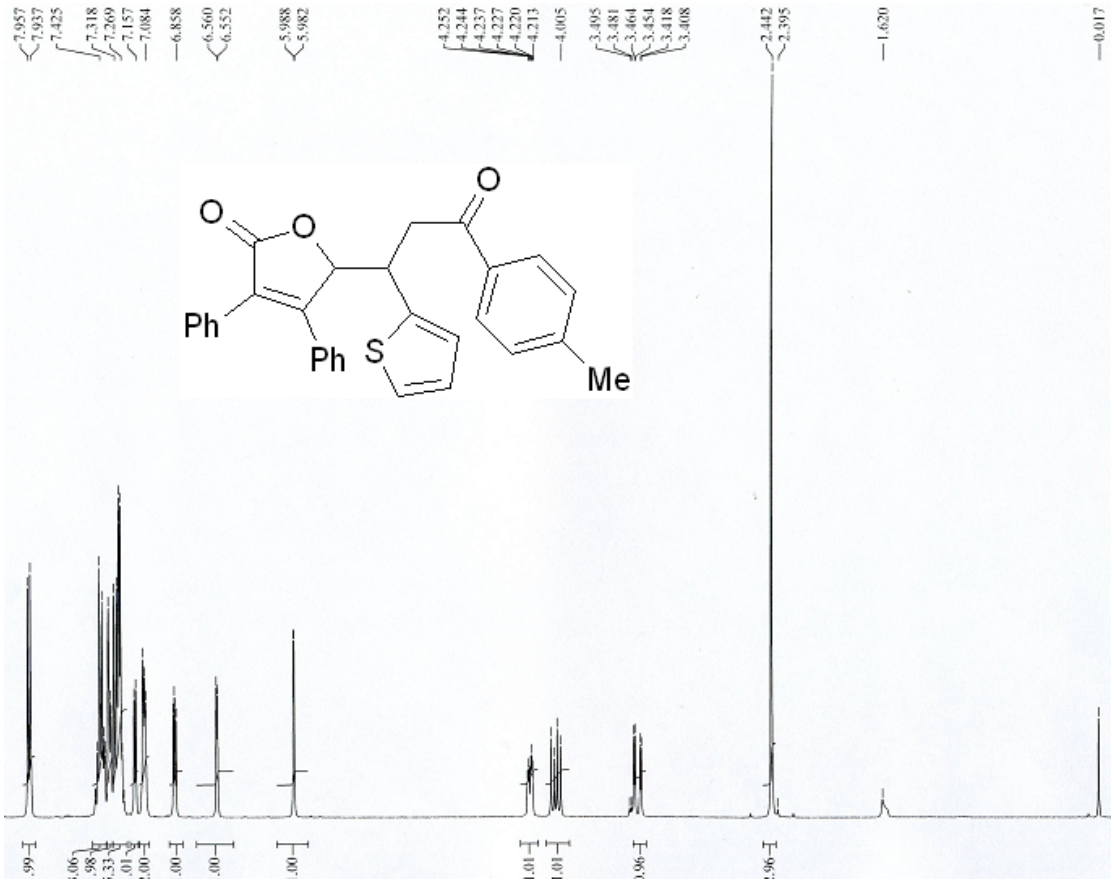

198.482
172.595
158.839
136.612
136.332
133.463
130.579
130.299
129.928
128.790
128.692
128.370
127.991
127.546
81.657
77.420
77.000
76.580
42.369
40.613
DFILE D:\李润涛\WF0901
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 288
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
Ph
Ph

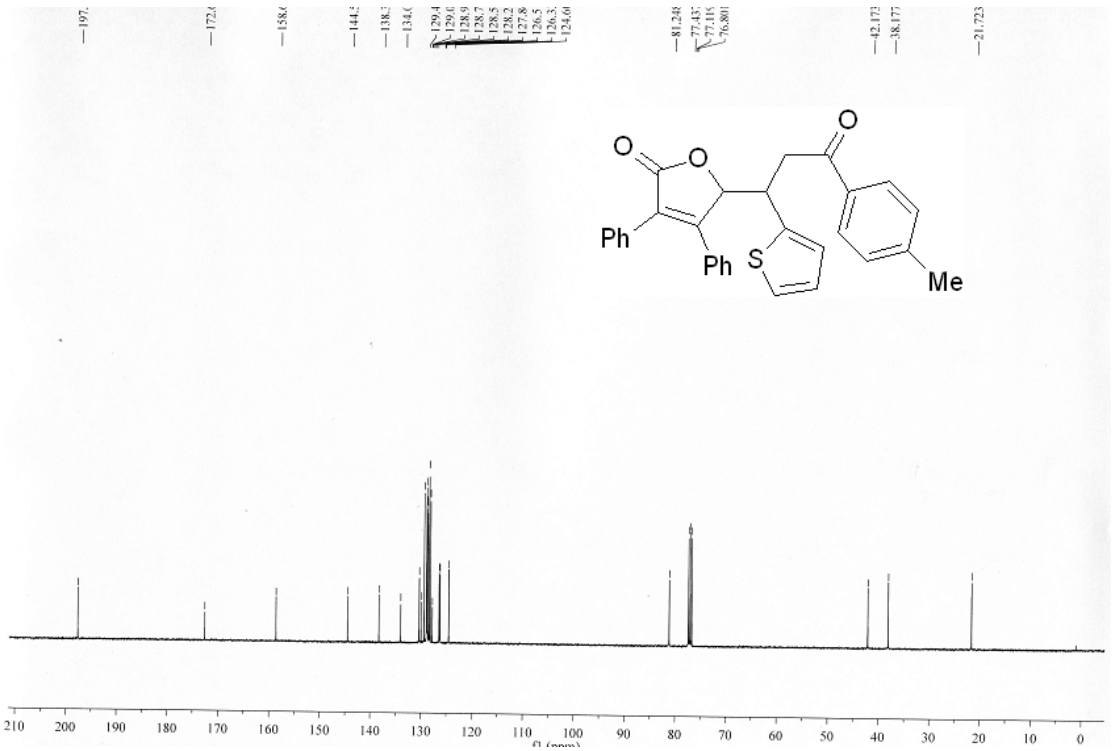
**3c**


7.966
7.946
7.408
7.301
7.248
7.170
7.145
6.942
6.924
5.969
5.964
4.125
4.099
4.079
4.053
3.819
3.446
3.437
3.400
3.391
2.446
1.607
0.089
0.013
O
O
O
Ph
Ph
Ph
S
2.00
1.02
1.02
8.44
1.07
4.01
1.00
0.99
1.01
1.00
3.01
f1 (ppm)

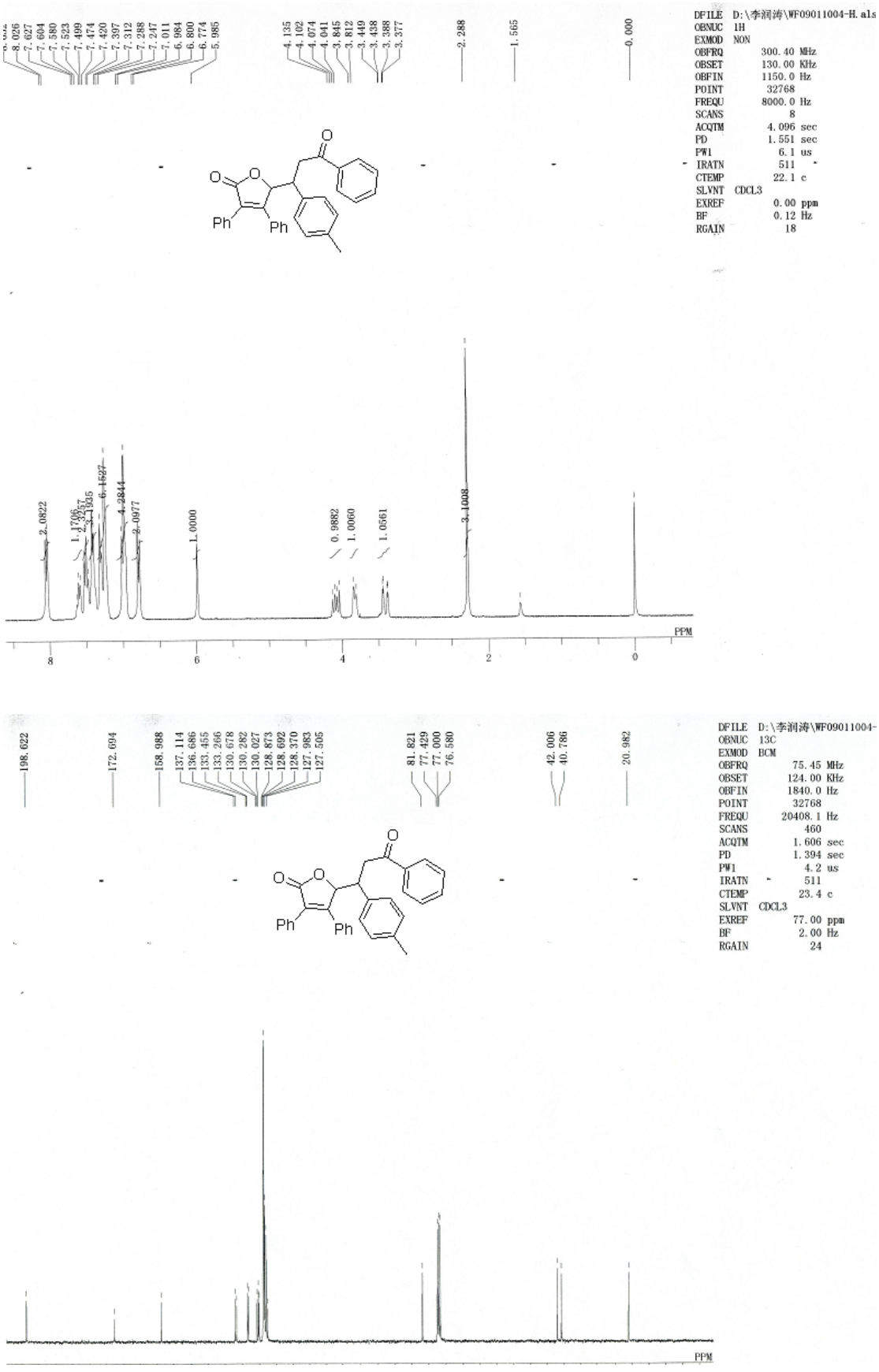

File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
2784 repetitions
OBSERVE C13, 125.7004365 MHz
DECOUPLE H1, 499.9043860 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
Total time 8 hr, 12 min, 39 sec
128.67
129.435
129.183
128.930
128.894
128.752
128.234
128.150
77.256
77.000
76.744
130.984
130.648
130.396
127.917
127.822
156.067
144.528
136.175
134.604
134.119
133.383
180.039
171.995
61.474
42.420
40.371
21.677
O
O
O
Ph
Ph
Ph
S
220
200
180
160
140
120
100
80
60
40
20
0
ppm

**3d**

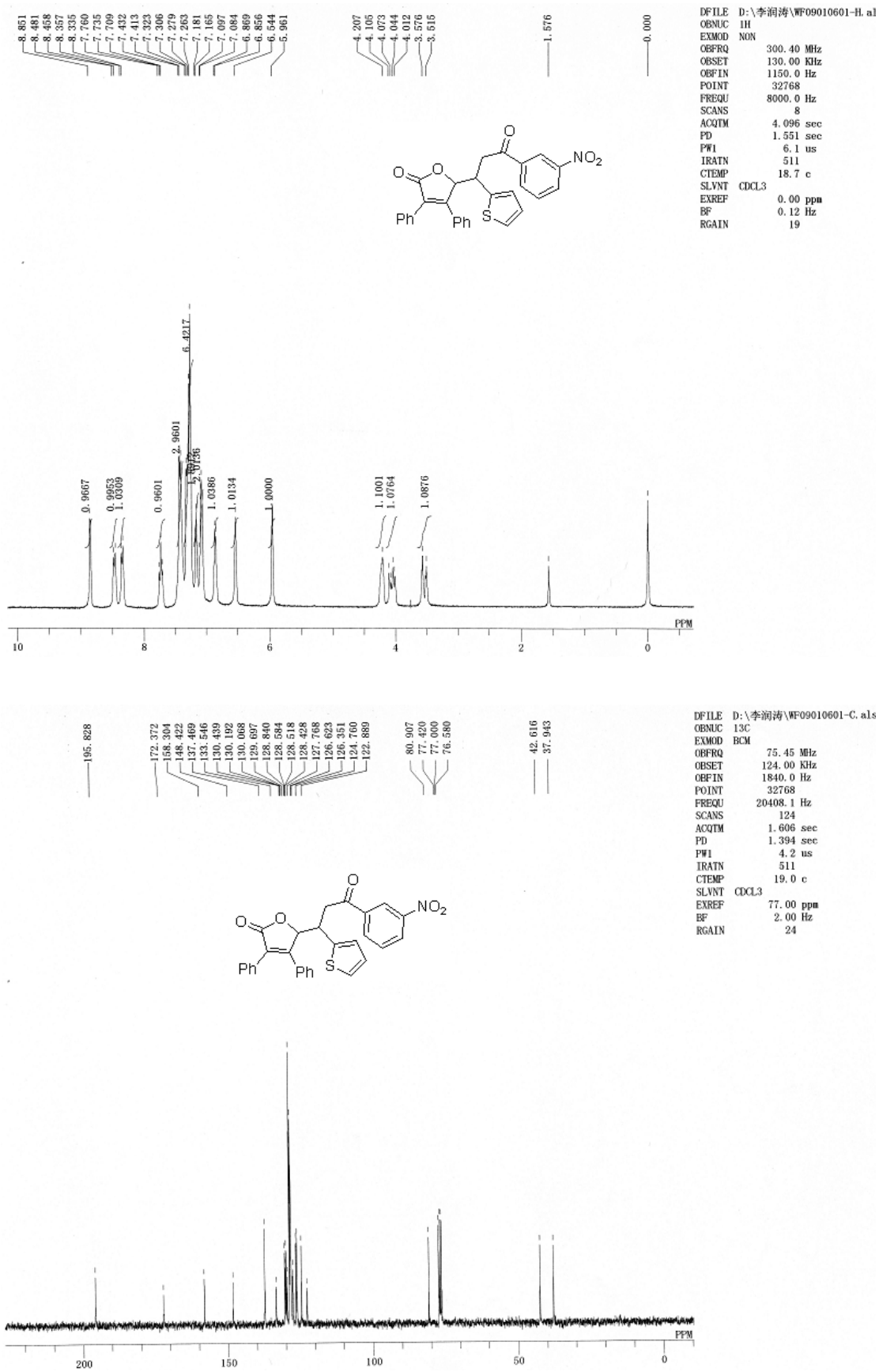

7.957
7.937
7.425
7.318
7.269
7.157
7.084
6.858
6.560
6.552
5.988
5.982
4.252
4.244
4.237
4.227
4.220
4.213
4.005
3.495
3.481
3.464
3.454
3.418
3.408
2.442
2.395
1.620
0.017
O
O
O
Ph
Ph
S
Me


O
O
O
Ph
Ph
S
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

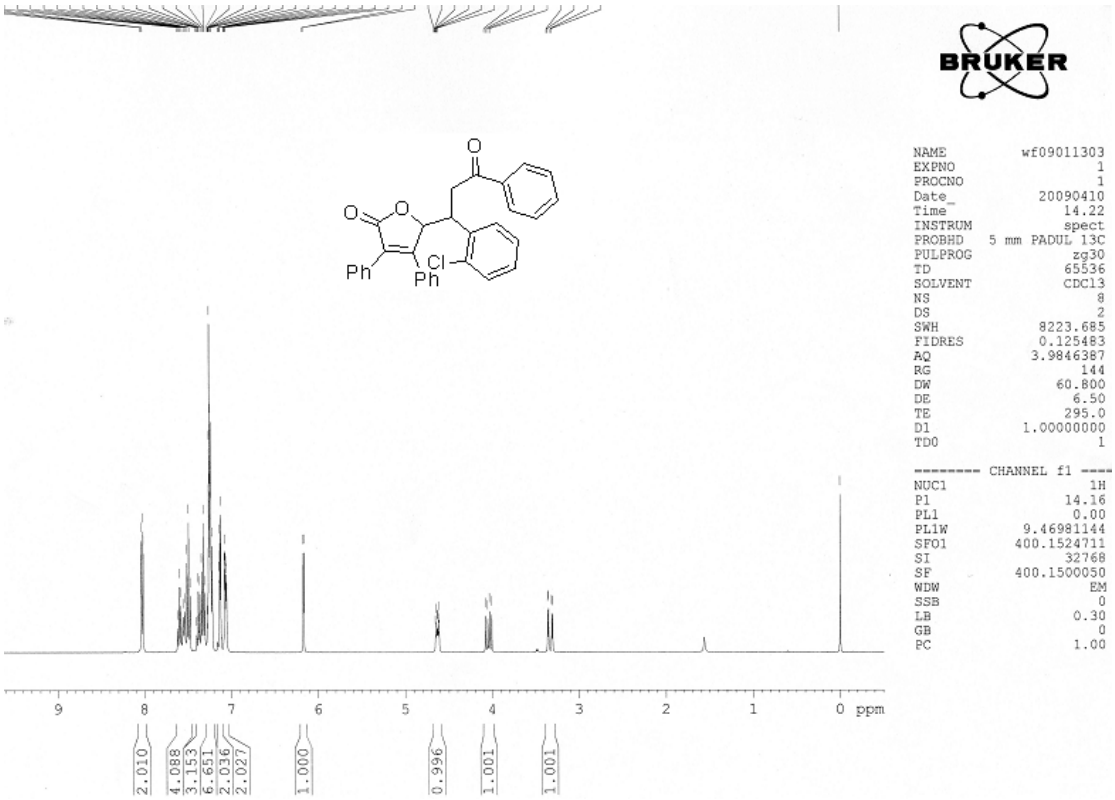
**3e**

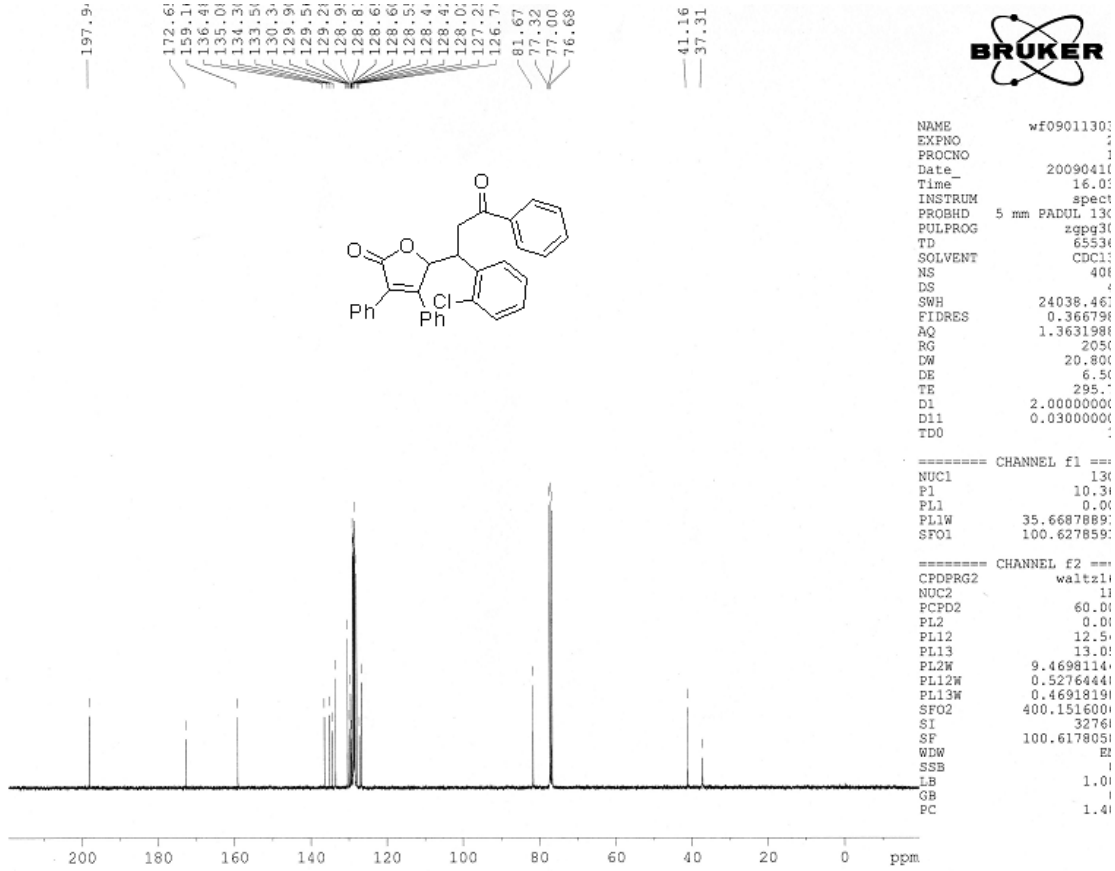

8.026
7.627
7.604
7.580
7.523
7.499
7.474
7.420
7.397
7.312
7.288
7.247
7.011
6.984
6.800
6.774
5.985
4.135
4.102
4.074
4.041
3.845
3.812
3.449
3.438
3.388
3.377
2.288
1.565
0.000
DFILE D:\李润涛\WF09011004-H.als
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18
O
O
O
Ph
Ph
2.0822
1.1706
2.3235
2.1935
6.1627
4.2844
2.0977
1.0000
0.9882
1.0060
1.0561
3.1008
PPM
8
6
4
2
0
198.622
172.694
158.988
137.114
136.686
133.455
133.266
130.678
130.282
130.027
128.873
128.692
128.370
127.983
127.505
81.821
77.429
77.000
76.580
42.006
40.786
20.982
DFILE D:\李润涛\WF09011004-
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 460
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
O
O
O
Ph
Ph
PPM

**3f**

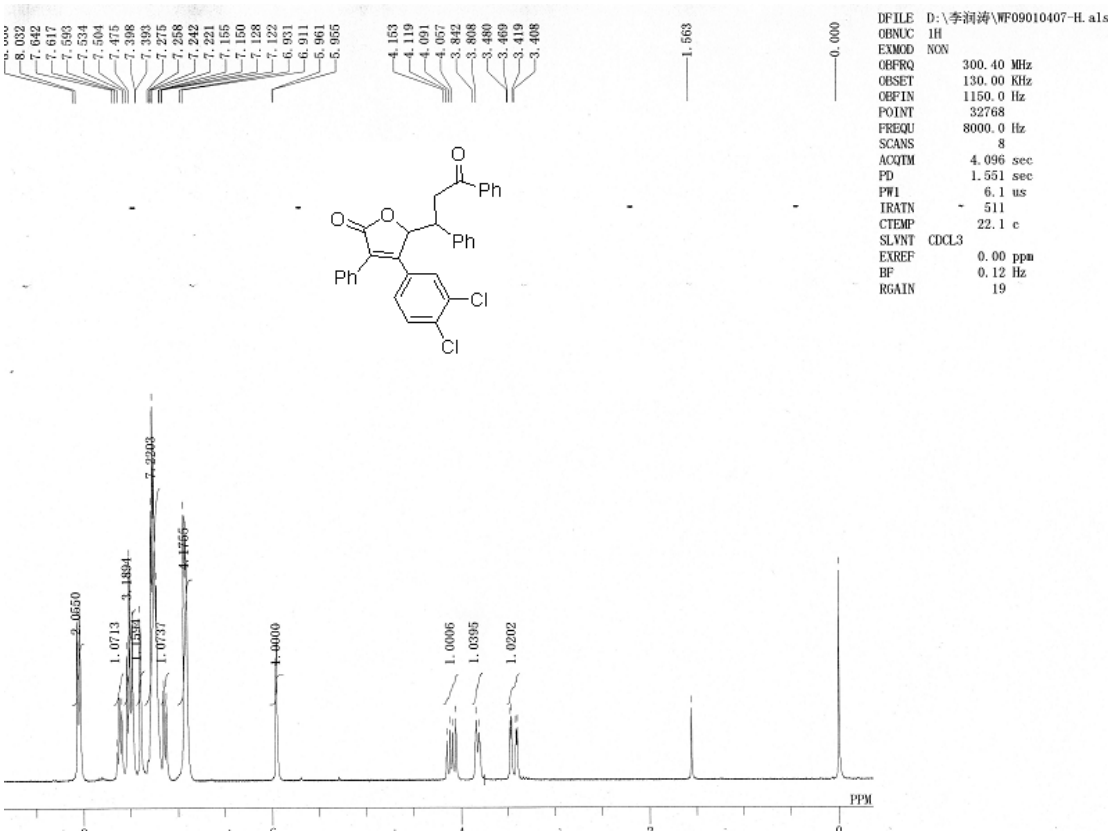

8.851 8.481 8.458 8.357 8.335 7.760 7.735 7.709 7.432 7.413 7.323 7.306 7.279 7.263 7.181 7.165 7.097 7.084 6.869 6.856 6.544 5.961
4.207 4.105 4.073 4.044 4.012 3.576 3.515
1.576
0.000
DFILE D:\李润涛\WF09010601-H.al
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 18.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
O
O
O
NO2
Ph
Ph
S
0.9667 0.9953 1.0309 0.9601 2.9601 6.4217 1.0386 1.0134 1.0000 1.1001 1.0764 1.0876
PPM
10 8 6 4 2 0
195.828
172.372 158.304 148.422 137.469 133.546 130.439 130.192 130.068 129.697 128.840 128.584 128.518 128.428 127.768 126.623 126.351 124.760 122.889
80.907 77.420 77.000 76.580
42.616 37.943
DFILE D:\李润涛\WF09010601-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 124
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
O
O
O
NO2
Ph
Ph
S
PPM
200 150 100 50 0

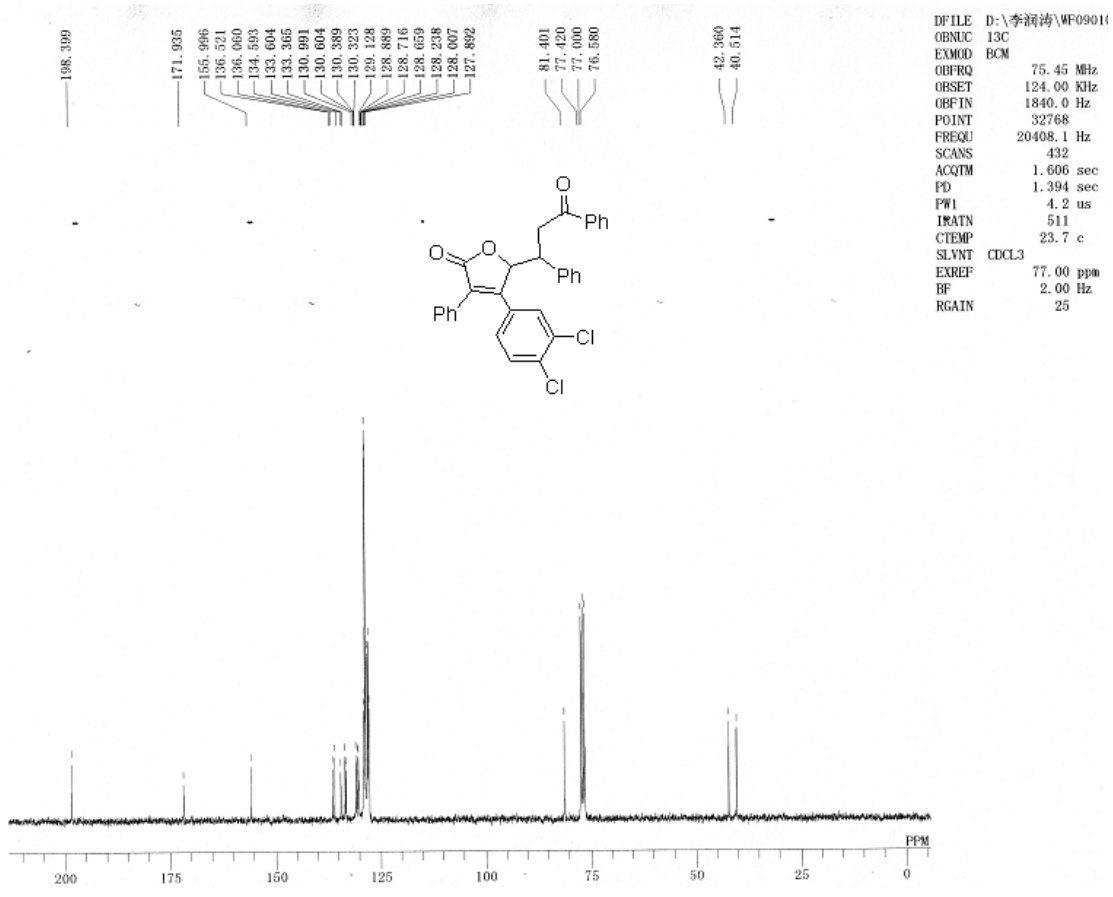
**3g**


BRUKER
NAME wf09011303
EXPNO 1
PROCNO 1
Date_ 20090410
Time 14.22
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685
FIDRES 0.125483
AQ 3.9846387
RG 144
DW 60.800
DE 6.50
TE 295.0
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 14.16
PL1 0.00
PL1W 9.46981144
SFO1 400.1524711
SI 32768
SF 400.1500050
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
O
O
O
Ph
Cl
Ph
9 8 7 6 5 4 3 2 1 0 ppm
2.010
4.088
3.153
6.651
2.036
2.027
1.000
0.996
1.001
1.001

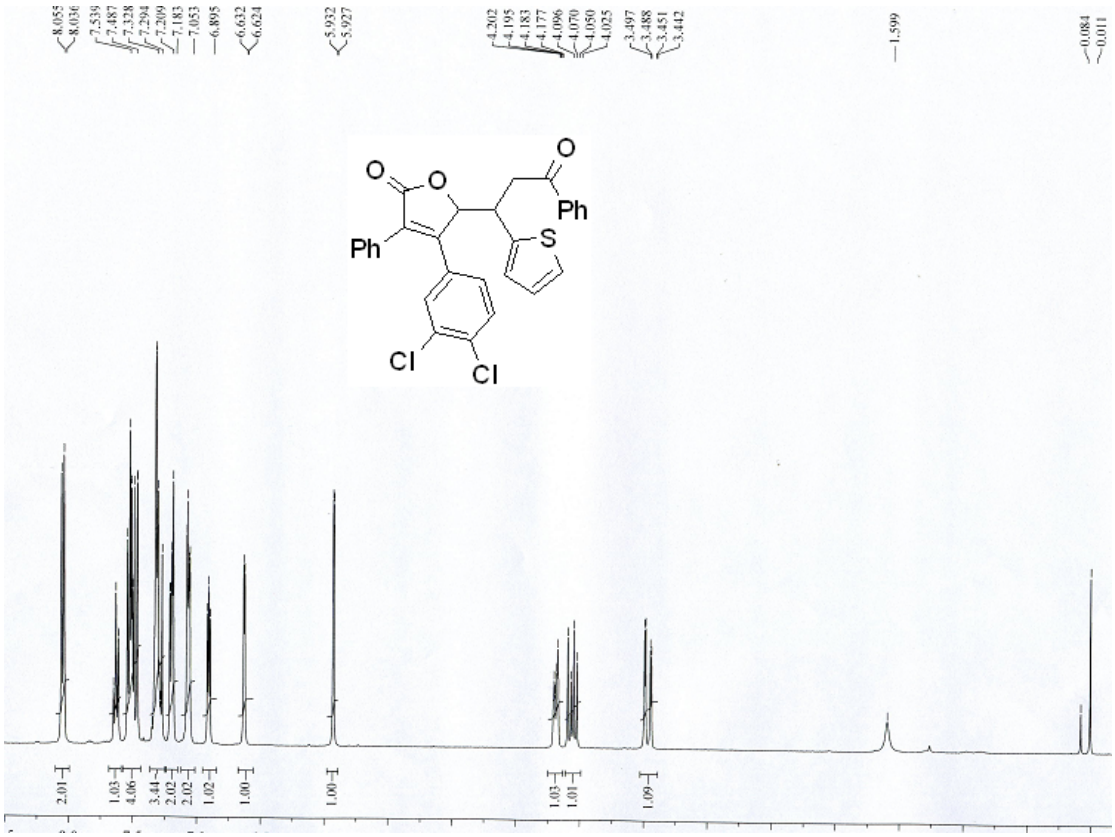

197.9
172.6
159.1
136.4
135.0
134.3
133.5
130.3
129.9
129.5
129.2
128.9
128.8
128.6
128.6
128.5
128.4
128.4
128.0
127.2
126.7
81.67
77.32
77.00
76.68
41.16
37.31
BRUKER
NAME wf09011303
EXPNO
PROCNO
Date_ 2009041
Time 16.0
INSTRUM spect
PROBHD 5 mm PADUL 13
PULPROG zgpg3
TD 6553
SOLVENT CDCl
NS 40
DS
SWH 24038.46
FIDRES 0.36679
AQ 1.3631988
RG 205
DW 20.80
DE 6.5
TE 295.
D1 2.0000000
D11 0.0300000
TD0
======== CHANNEL f1 ===
NUC1 13
P1 10.3
PL1 0.0
PL1W 35.6687889
SFO1 100.6278593
======== CHANNEL f2 ===
CPDPRG2 waltz1
NUC2 1
PCPD2 60.0
PL2 0.0
PL12 12.5
PL13 13.0
PL2W 9.4698114
PL12W 0.5276444
PL13W 0.4691819
SFO2 400.151600
SI 3276
SF 100.617805
WDW E
SSB
LB 1.0
GB
PC 1.4
O
O
O
Ph
Cl
Ph
200 180 160 140 120 100 80 60 40 20 0 ppm

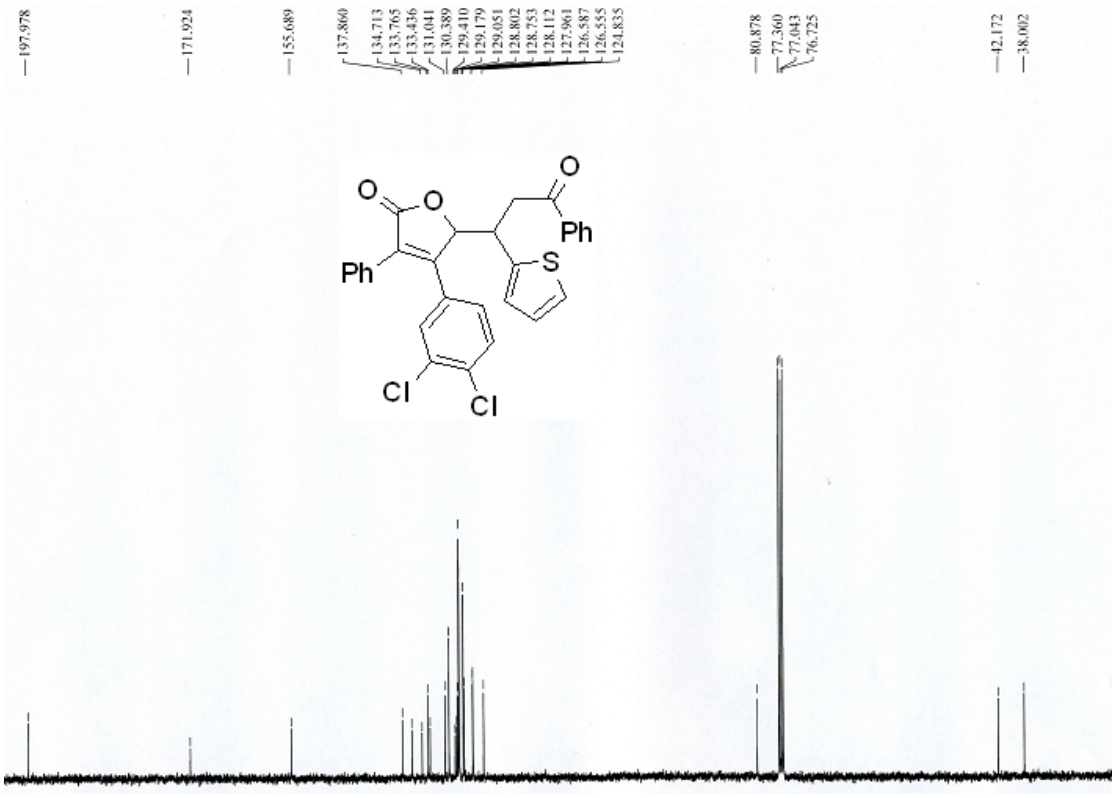
**3h**


8.032
7.642
7.617
7.593
7.534
7.504
7.475
7.398
7.393
7.275
7.258
7.242
7.221
7.155
7.150
7.128
7.122
6.931
6.911
6.961
5.955
4.153
4.119
4.091
4.057
3.842
3.808
3.480
3.469
3.419
3.408
1.563
0.000
DFILE D:\李润涛\WF09010407-H.als
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
O
Ph
O
O
Ph
Ph
Cl
Cl
2.0650
1.0713
3.1894
1.5541
1.0737
7.2303
4.1565
1.0000
1.0006
1.0395
1.0202
PPM


198.399
171.935
155.996
136.521
136.060
134.593
133.604
133.365
130.991
130.604
130.389
130.323
129.128
128.889
128.716
128.659
128.238
128.007
127.892
81.401
77.420
77.000
76.580
42.360
40.514
DFILE D:\李润涛\WF0901
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 432
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
O
Ph
O
O
Ph
Ph
Cl
Cl
PPM
200
175
150
125
100
75
50
25
0

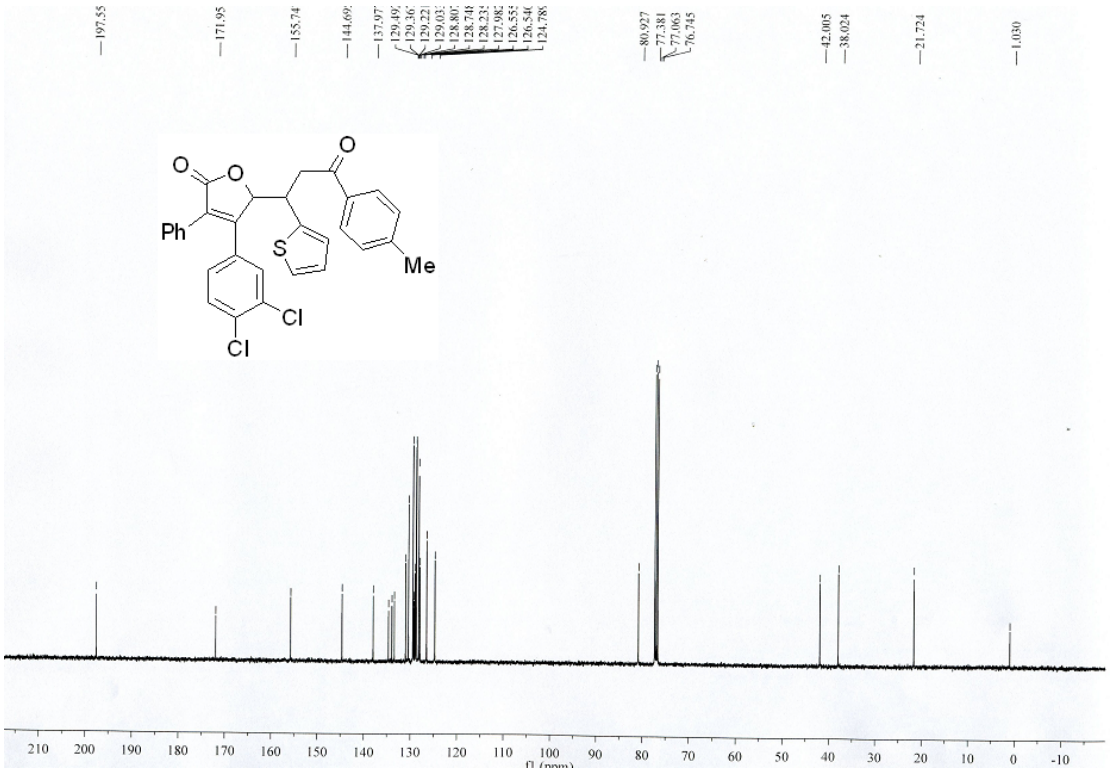
**3i**


8.055
8.036
7.539
7.487
7.328
7.294
7.209
7.183
7.053
6.895
6.632
6.624
5.932
5.927
4.202
4.195
4.183
4.177
4.096
4.070
4.050
4.025
3.497
3.488
3.451
3.442
1.599
0.084
0.011
O
O
O
Ph
Ph
S
Cl
Cl
2.01
1.03
4.06
3.44
2.02
2.02
1.02
1.00
1.00
1.03
1.01
1.09

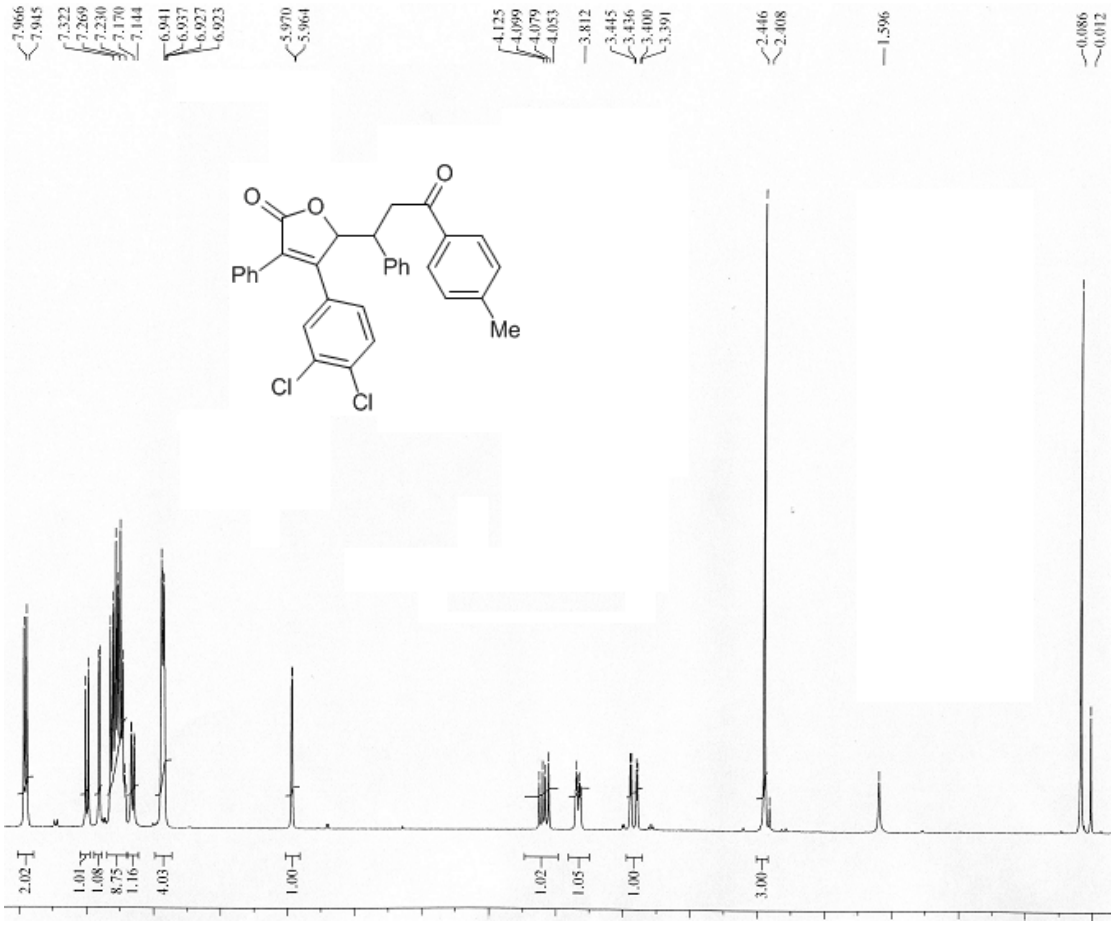

197.978
171.924
155.689
137.860
134.713
133.765
133.436
131.041
130.389
129.410
129.179
129.051
128.802
128.753
128.112
127.961
126.587
126.555
124.835
80.878
77.360
77.043
76.725
42.172
38.002
O
O
O
Ph
Ph
S
Cl
Cl

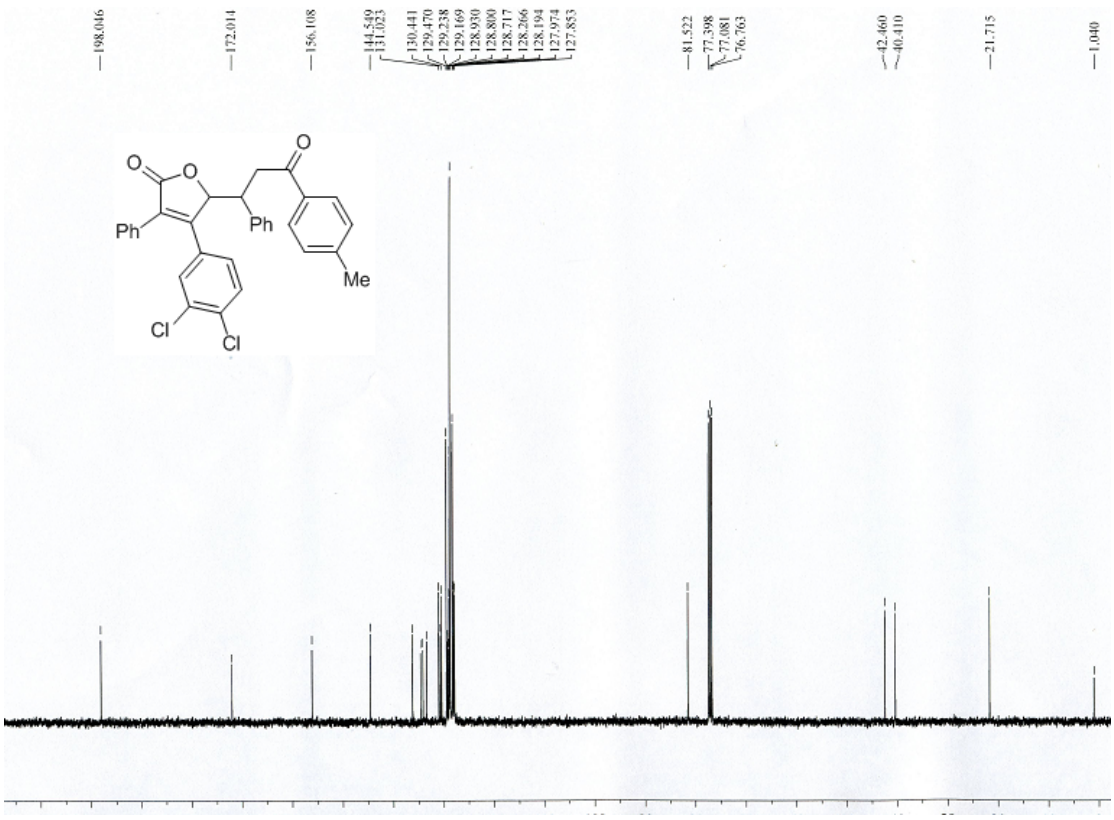
**3j**


7.952
7.932
7.484
7.461
7.456
7.277
7.189
7.051
6.892
6.629
6.622
5.929
5.924
4.202
4.195
4.189
4.177
4.170
4.069
4.044
4.024
3.998
3.459
3.450
3.413
3.404
2.446
1.582
0.012
O
O
O
Ph
S
Me
Cl
Cl
2.00
2.04
5.49
2.00
2.00
1.02
0.99
1.00
1.01
1.01
1.05
3.00

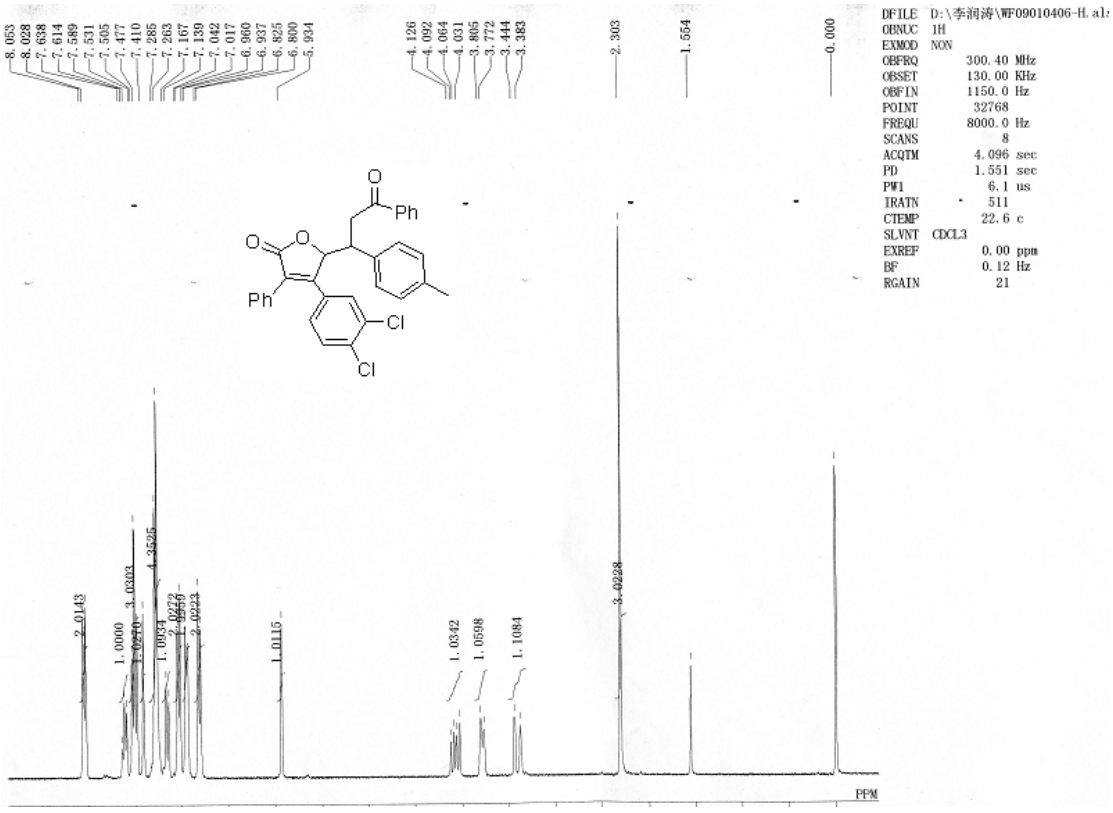

197.55
171.95
155.74
144.69
137.97
80.927
77.381
77.063
76.745
42.005
38.024
21.724
14.030
O
O
O
Ph
S
Me
Cl
Cl
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

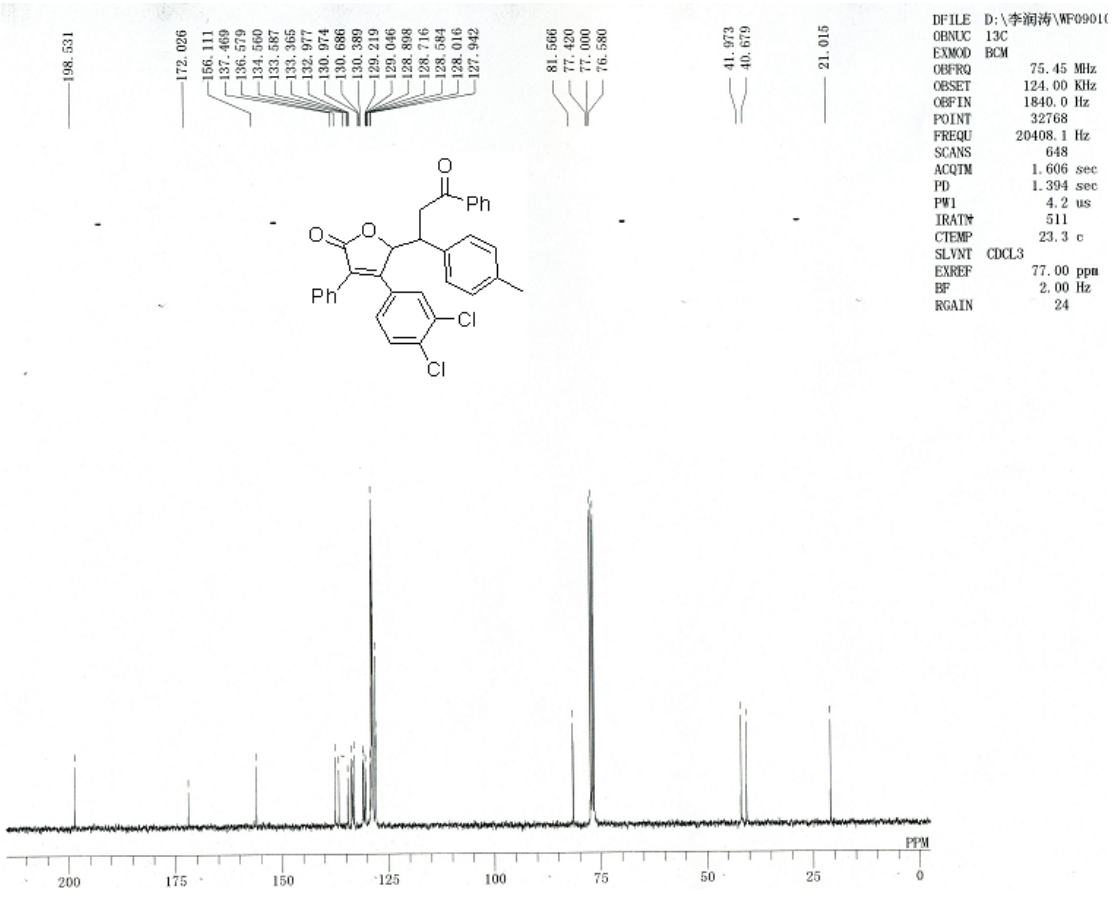
**3k**


7.966
7.945
7.322
7.269
7.230
7.170
7.144
6.941
6.937
6.927
6.923
5.970
5.964
4.125
4.099
4.079
4.053
3.812
3.445
3.436
3.400
3.391
2.446
2.408
1.596
0.086
0.012
O
O
Ph
Ph
O
Me
Cl
Cl
2.02
1.01
1.08
8.75
1.16
4.03
1.00
1.02
1.05
1.00
3.00

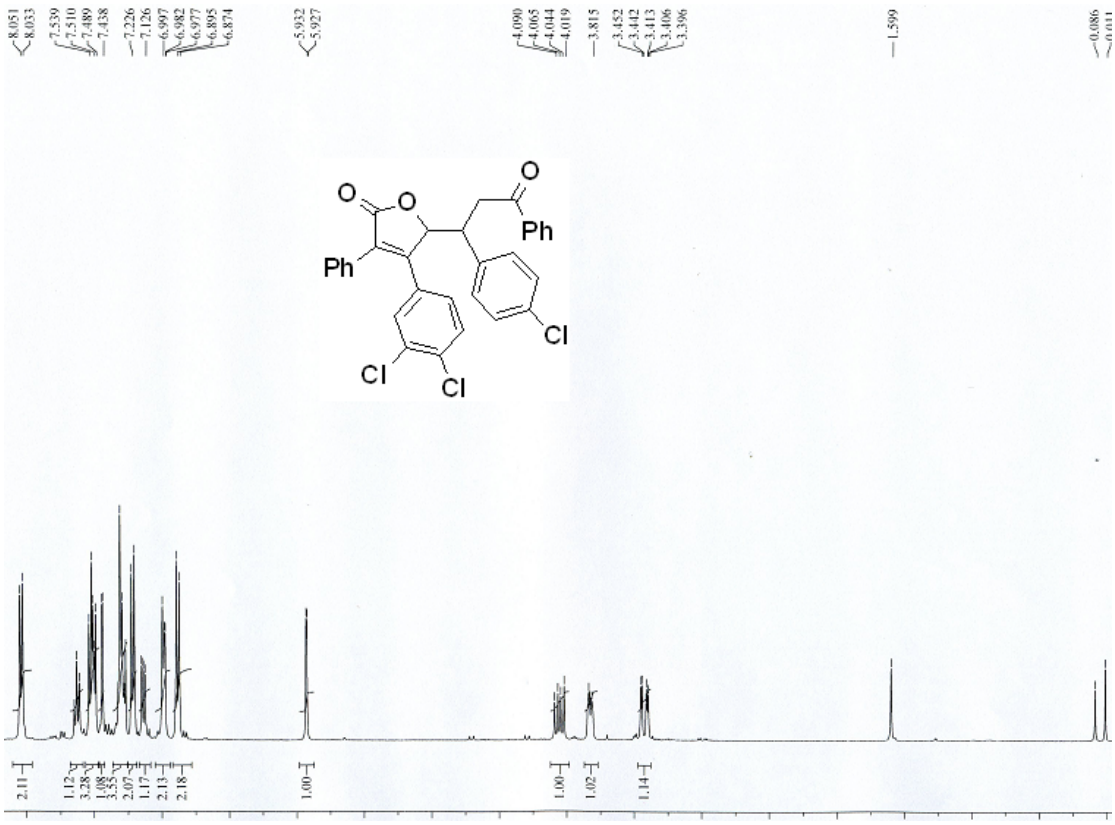

198.046
172.014
156.108
144.549
131.023
130.441
129.470
129.238
129.169
128.930
128.800
128.717
128.266
128.194
127.974
127.853
81.522
77.398
77.081
76.763
42.460
40.410
21.715
1.040
O
O
Ph
Ph
O
Me
Cl
Cl

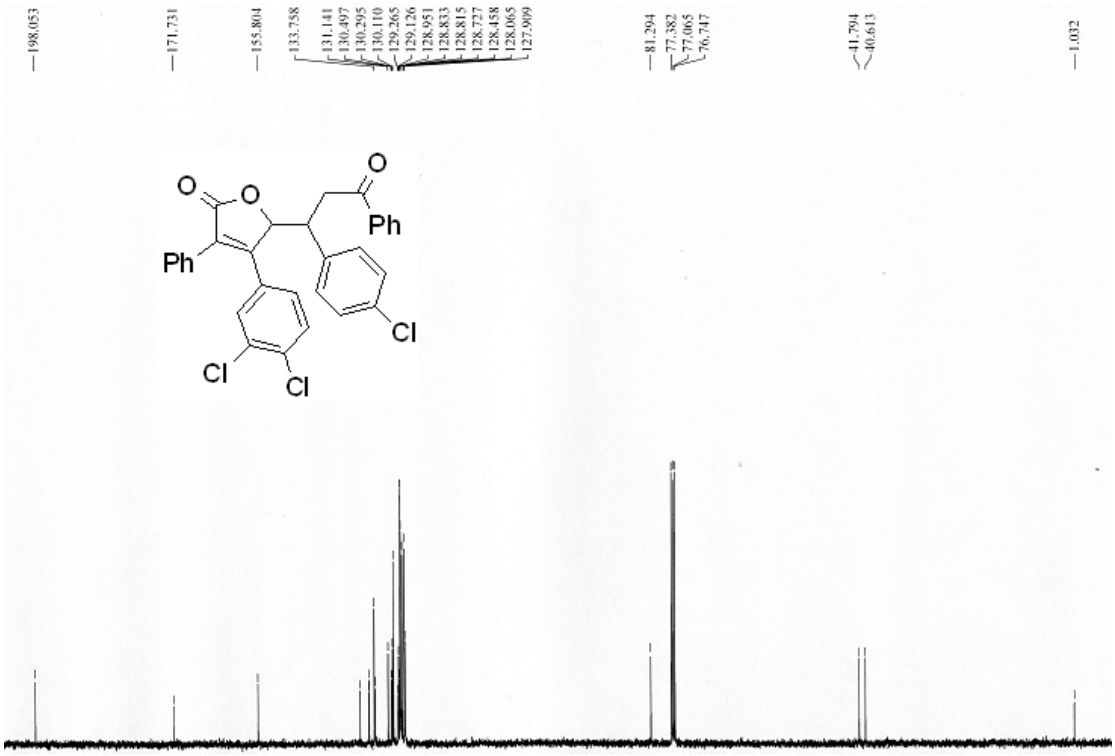
**3l**


8.053
8.028
7.638
7.614
7.589
7.531
7.505
7.477
7.410
7.285
7.263
7.167
7.139
7.042
7.017
6.960
6.937
6.825
6.800
5.934
4.126
4.092
4.064
4.031
3.805
3.772
3.444
3.383
2.303
1.554
0.000
DFILE D:\李润涛\WF09010406-H.al
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 21
Ph
O
O
Ph
Cl
Cl
2.0143
1.0000
3.0303
1.0250
4.3525
1.0934
2.0572
2.0559
2.0223
1.0115
1.0342
1.0598
1.1084
3.0228
PPM

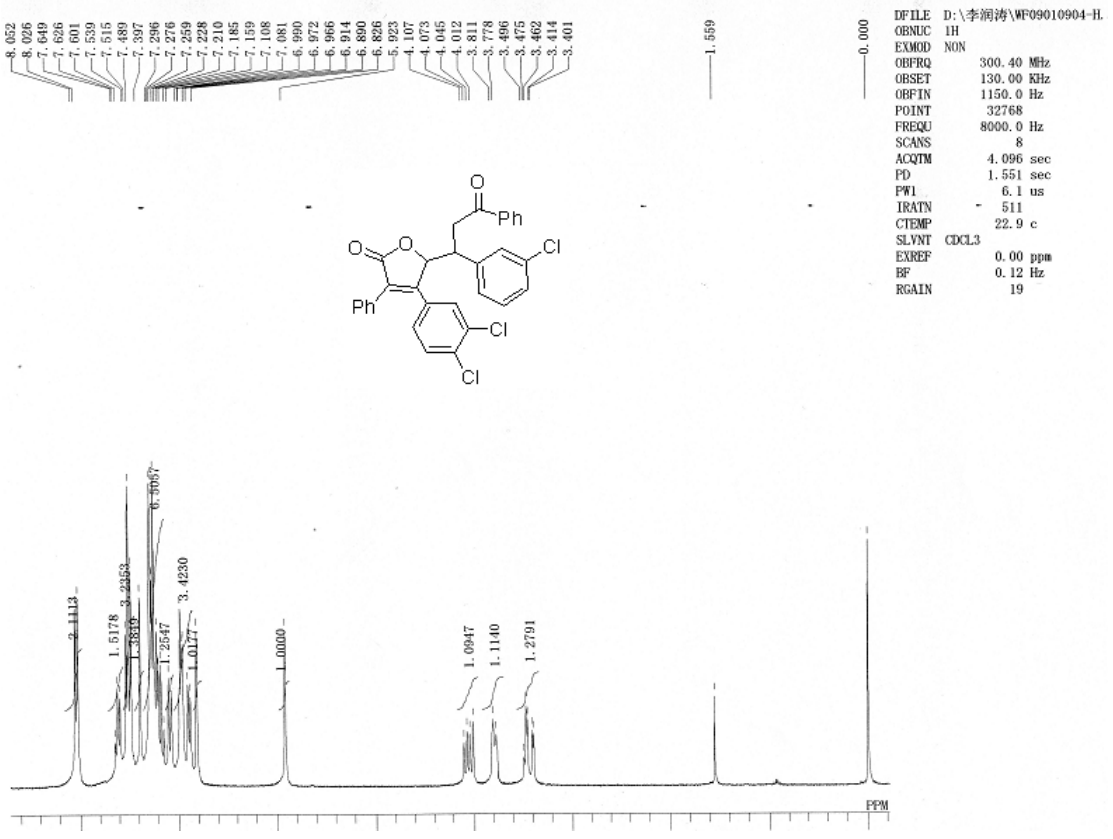

198.531
172.026
156.111
137.469
136.579
134.560
133.587
133.365
132.977
130.974
130.686
130.389
129.219
129.046
128.898
128.716
128.584
128.016
127.942
81.566
77.420
77.000
76.580
41.973
40.679
21.015
DFILE D:\李润涛\WF0901(
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 648
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
O
Ph
O
O
Ph
Cl
Cl
PPM
200
175
150
125
100
75
50
25
0

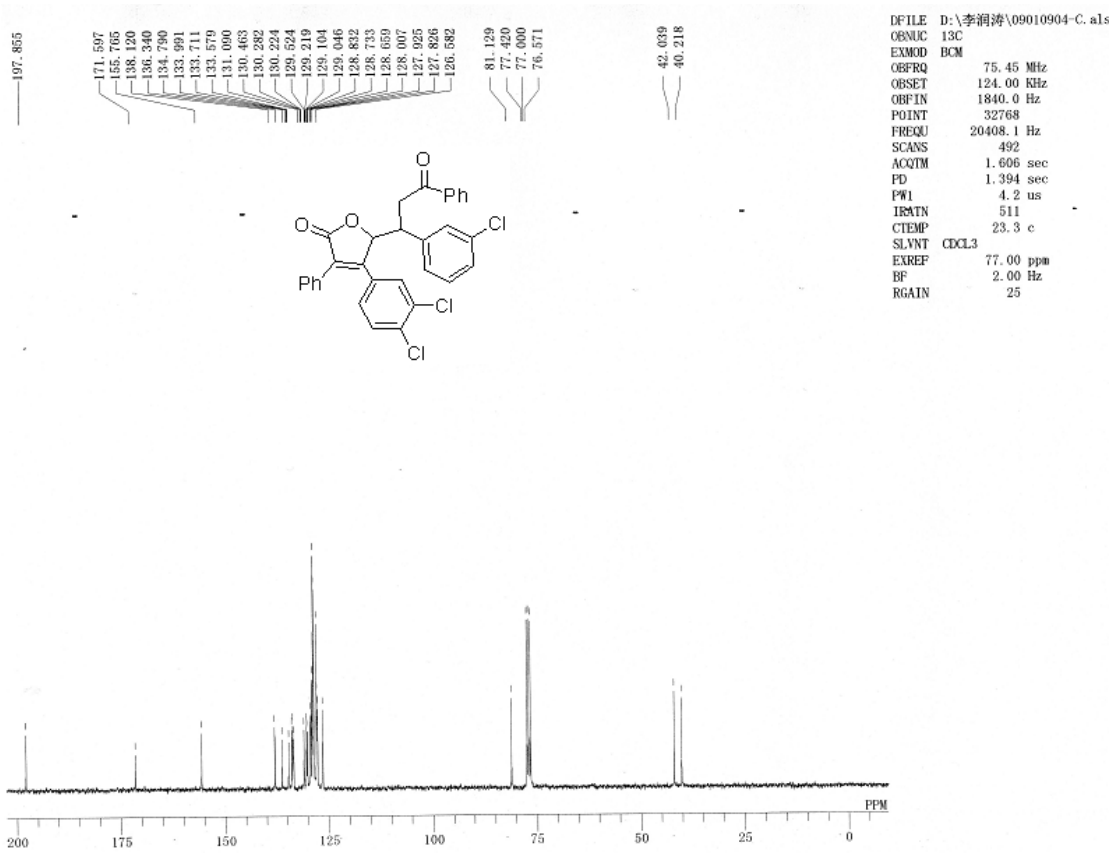
**3m**


8.051
8.033
7.539
7.510
7.489
7.438
7.226
7.126
6.997
6.982
6.977
6.895
6.874
5.932
5.927
4.090
4.065
4.044
4.019
3.815
3.452
3.442
3.413
3.406
3.396
1.599
0.086
0.011
O
O
O
Ph
Ph
Cl
Cl
Cl
2.11
1.12
3.28
1.08
1.55
2.07
1.17
2.13
2.18
1.00
1.00
1.02
1.14

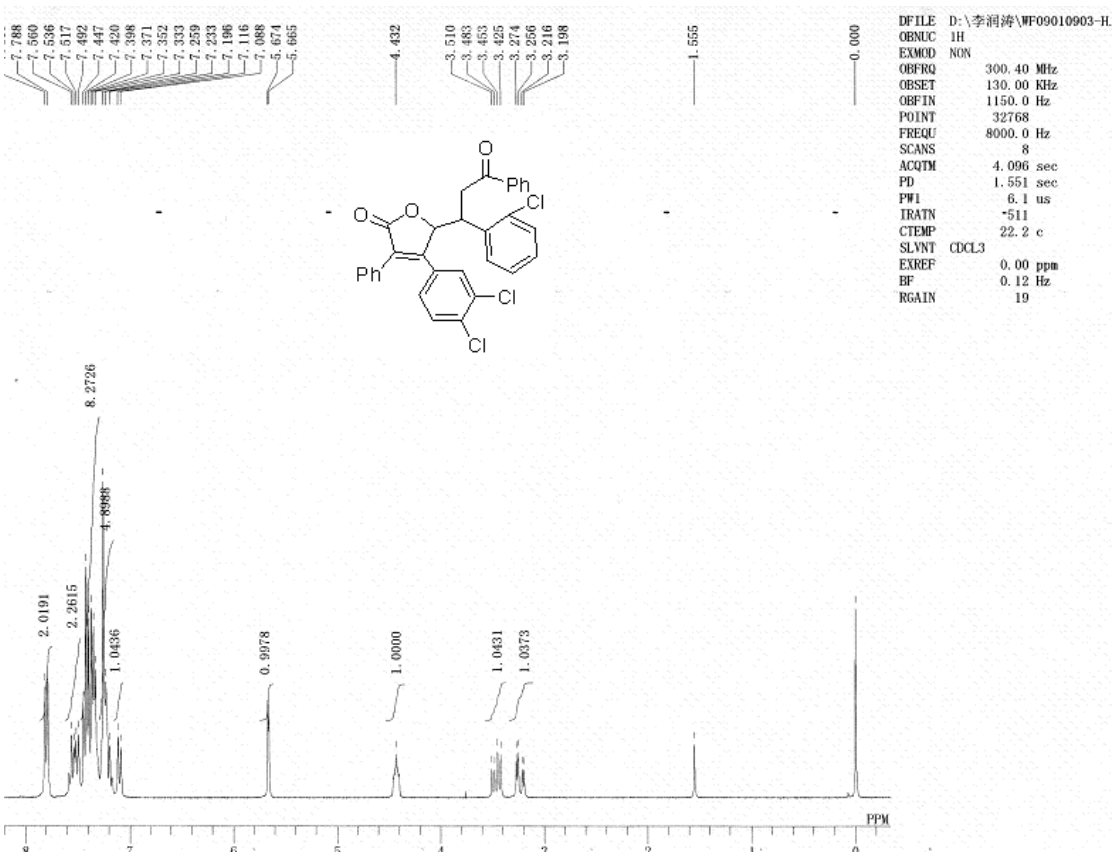

198.053
171.731
155.804
133.758
131.141
130.497
130.295
130.110
129.265
129.126
128.951
128.833
128.815
128.727
128.458
128.065
127.909
81.294
77.382
77.065
76.747
41.794
40.613
1.032
O
O
O
Ph
Ph
Cl
Cl
Cl

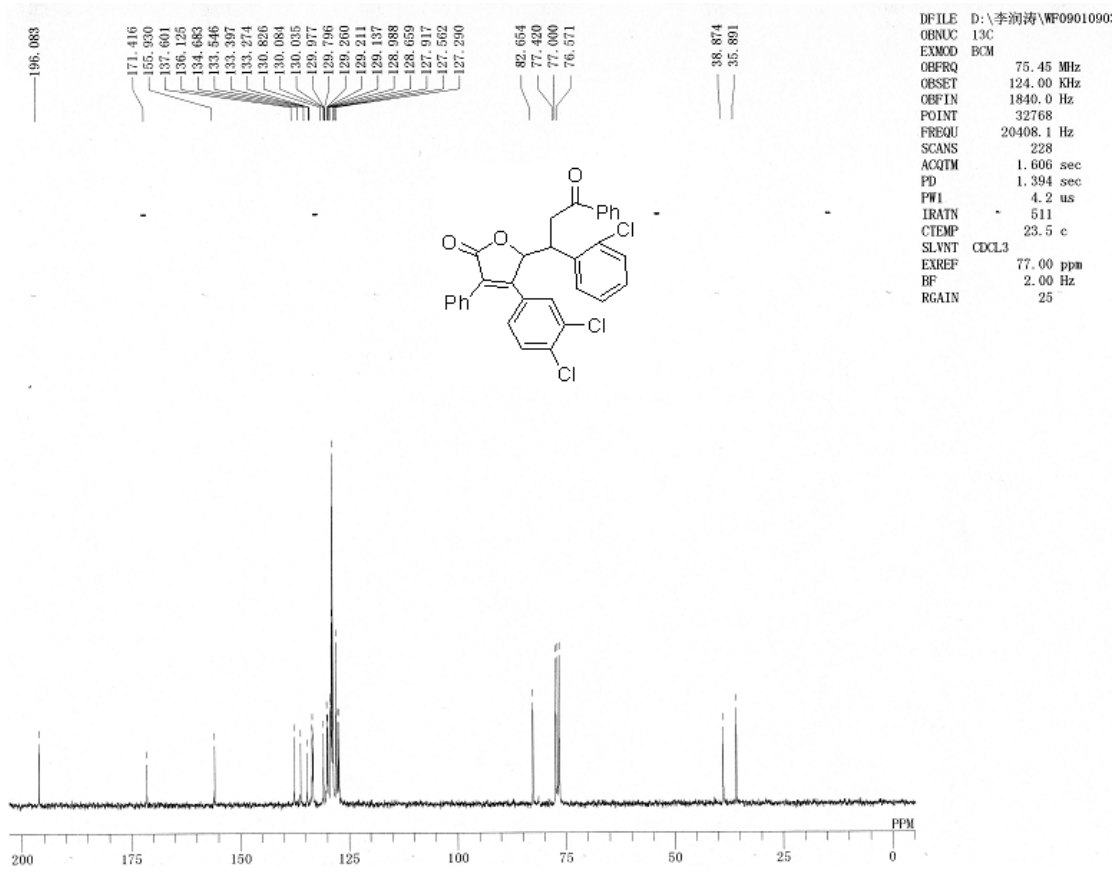
**3n**


8.052
8.026
7.649
7.626
7.601
7.539
7.515
7.489
7.397
7.296
7.276
7.259
7.228
7.210
7.185
7.159
7.108
7.081
6.990
6.972
6.966
6.914
6.880
6.826
5.923
4.107
4.073
4.045
4.012
3.811
3.778
3.496
3.475
3.462
3.414
3.401
1.559
0.000
DFILE D:\李润涛\WF09010904-H.
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
O
Ph
O
O
Cl
Ph
Cl
Cl
2.1113
1.5178
3.2353
1.3819
6.5067
1.2547
3.4230
1.0177
1.0000
1.0947
1.1140
1.2791
PPM

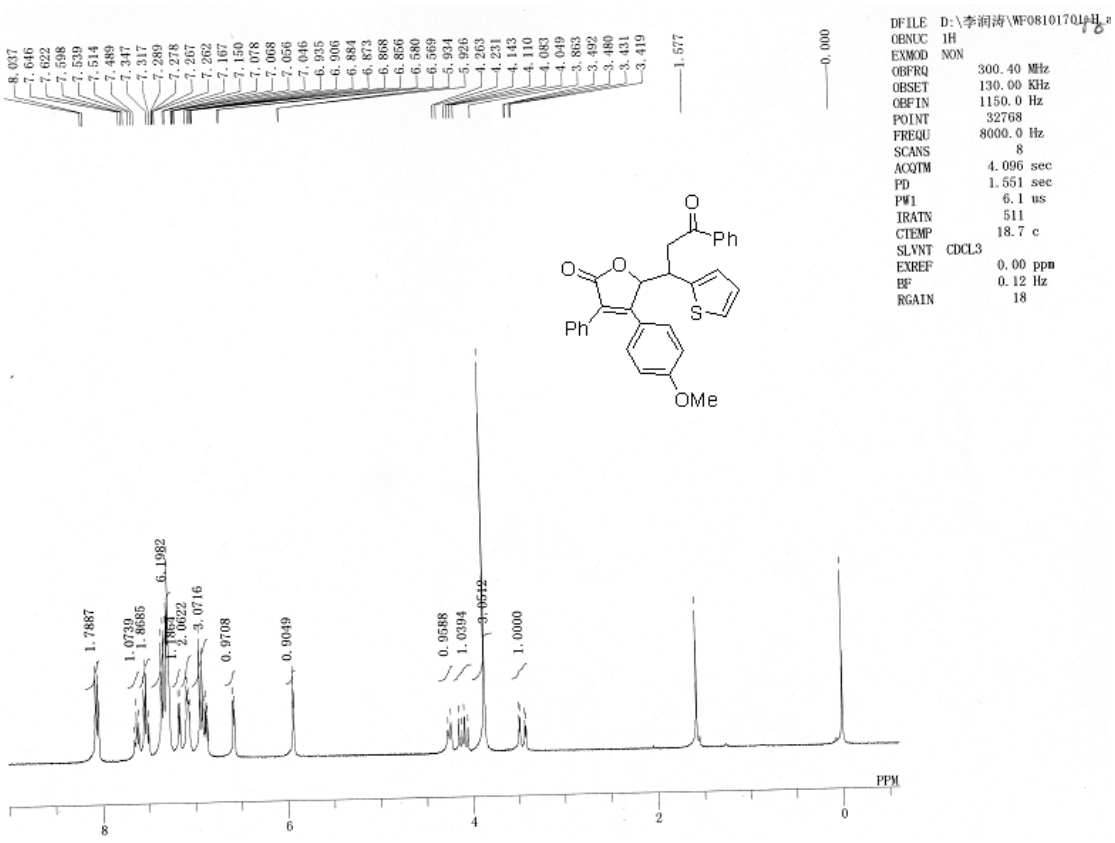

197.855
171.597
155.765
138.120
136.340
134.790
133.991
133.711
133.579
131.090
130.463
130.282
130.224
129.524
129.219
129.104
129.046
128.832
128.733
128.659
128.007
127.925
127.826
126.582
81.129
77.420
77.000
76.571
42.039
40.218
DFILE D:\李润涛\09010904-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 492
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
O
Ph
O
O
Cl
Ph
Cl
Cl
PPM
200
175
150
125
100
75
50
25
0

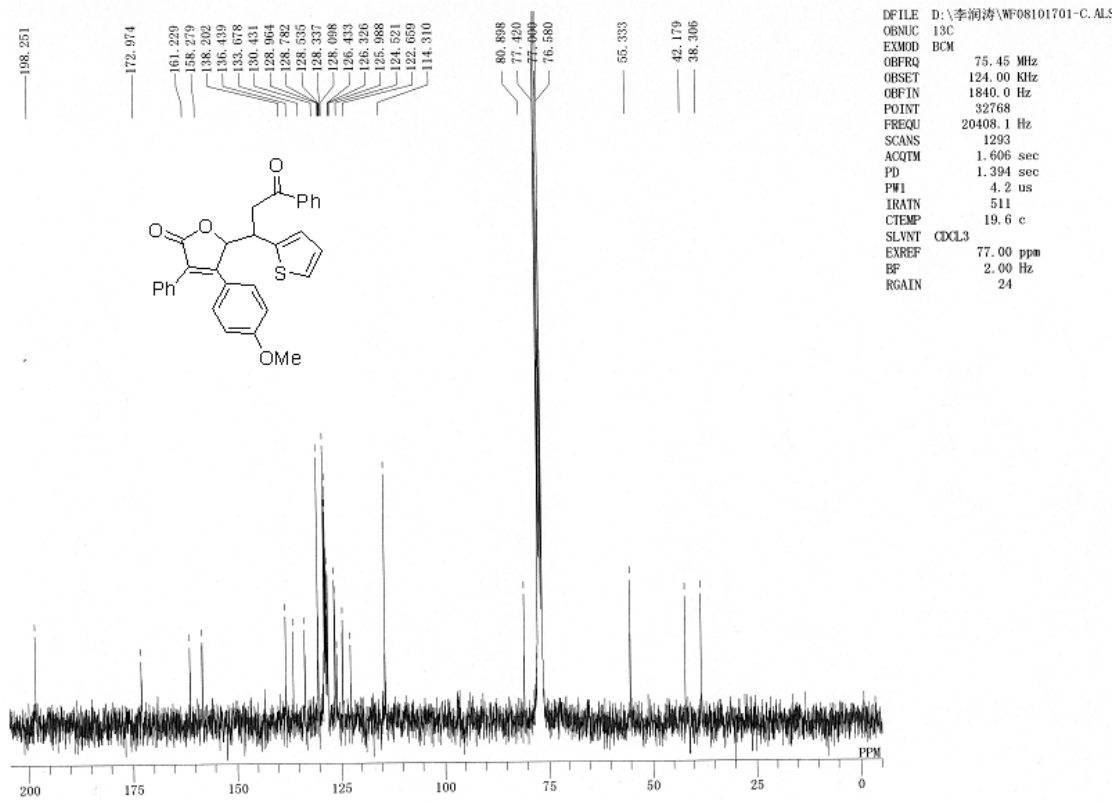
## 3o


7.788
7.560
7.536
7.517
7.492
7.447
7.420
7.398
7.371
7.352
7.333
7.259
7.233
7.196
7.116
7.088
5.674
5.665
4.432
3.510
3.483
3.453
3.425
3.274
3.256
3.216
3.198
1.555
0.000
DFILE D:\李润涛\WF09010903-H.
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
O
Ph
Cl
O
O
Ph
Cl
Cl
8.2726
4.8988
2.0191
2.2615
1.0436
0.9978
1.0000
1.0431
1.0373
PPM

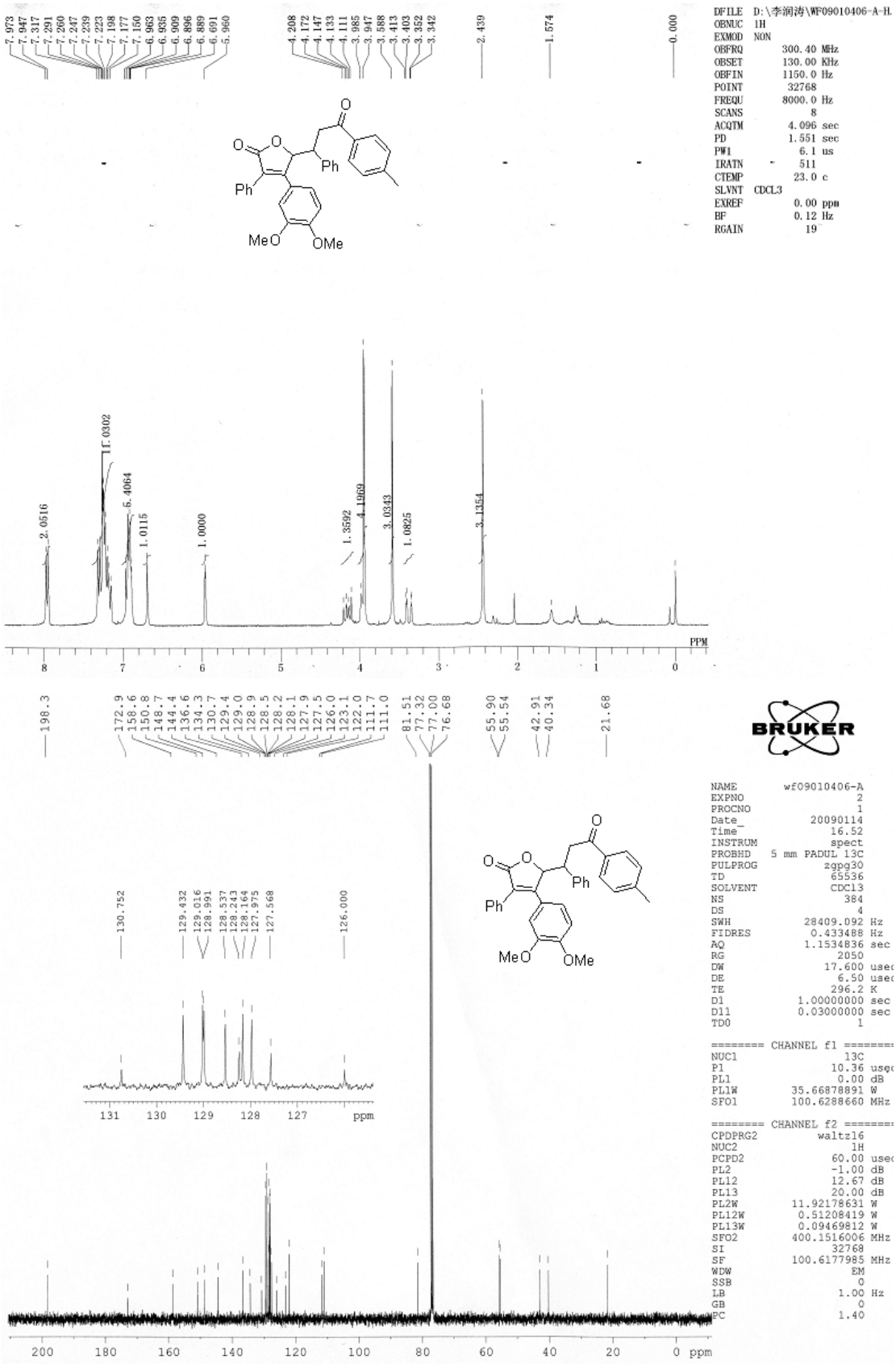

196.083
171.416
155.930
137.601
136.125
134.683
133.946
133.397
133.274
130.826
130.084
130.035
129.977
129.796
129.260
129.211
129.137
128.988
128.659
127.917
127.562
127.290
82.654
77.420
77.000
76.571
38.874
35.891
DFILE D:\李润涛\WF0901090
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 228
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
O
Ph
Cl
O
O
Ph
Cl
Cl
PPM
200
175
150
125
100
75
50
25
0

**3p**

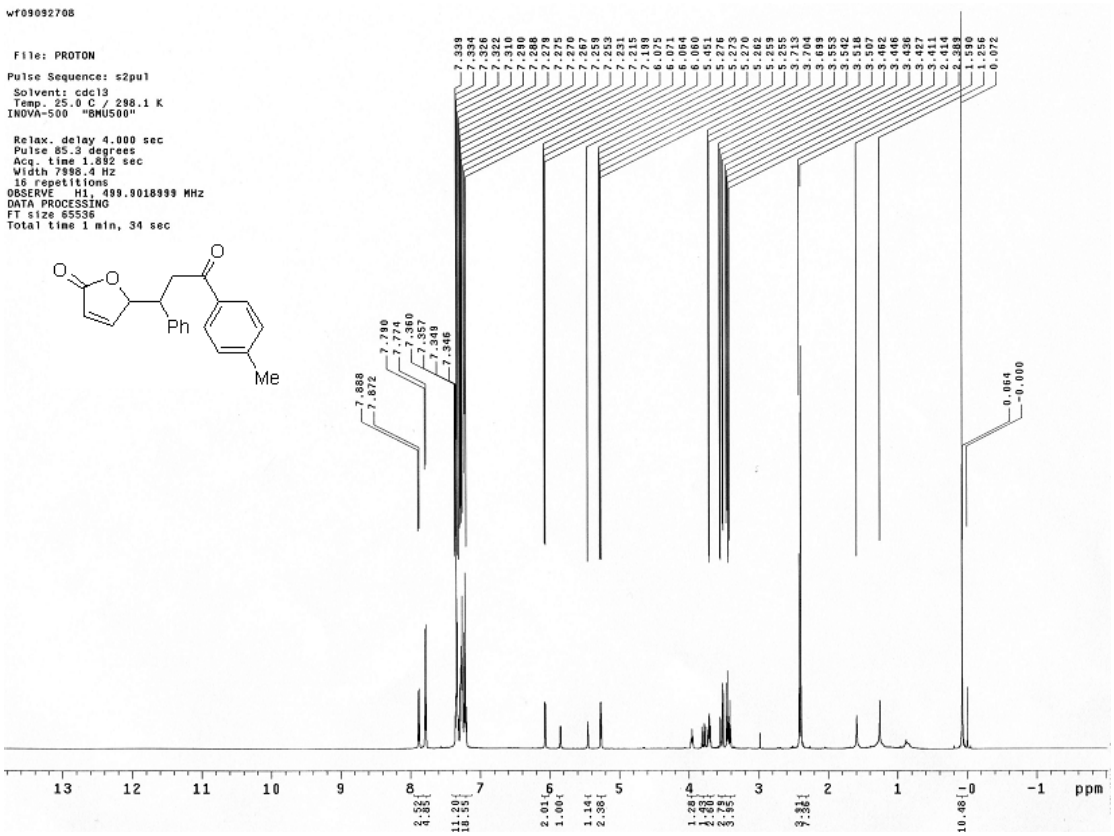

8.037
7.646
7.622
7.598
7.539
7.514
7.489
7.347
7.317
7.289
7.278
7.267
7.262
7.167
7.150
7.078
7.068
7.056
7.046
6.935
6.906
6.884
6.873
6.868
6.856
6.580
6.569
5.934
5.926
4.263
4.231
4.143
4.110
4.083
4.049
3.863
3.492
3.480
3.431
3.419
1.577
0.000
DFILE D:\李润涛\WF08101701-H.a
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 18.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18
Ph
O
O
Ph
S
OMe
1.7887
1.0739
1.8685
6.1982
1.0622
3.0716
0.9708
0.9049
0.9588
1.0394
3.0512
1.0000
PPM
8
6
4
2
0

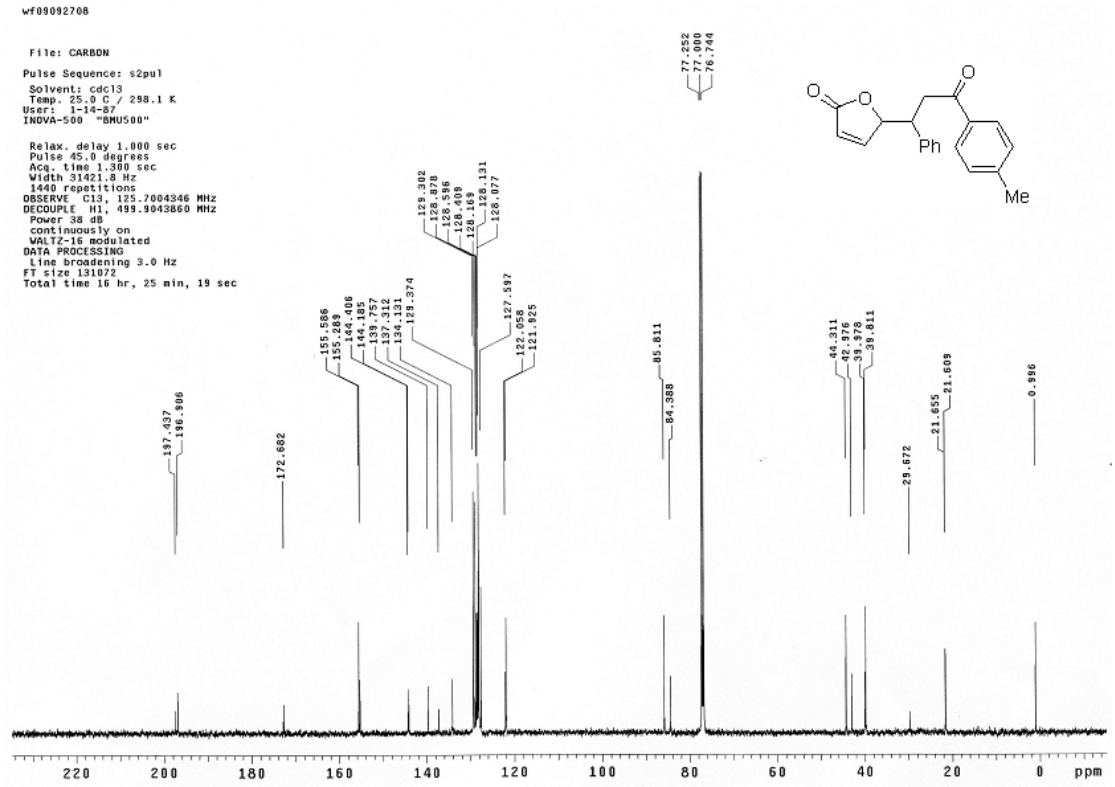

198.251
172.974
161.229
158.279
138.202
136.439
133.678
130.431
128.964
128.782
128.535
128.337
128.098
126.433
126.326
125.988
124.521
122.659
114.310
80.898
77.420
77.000
76.580
55.333
42.179
38.306
DFILE D:\李润涛\WF08101701-C.AL
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 1293
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
O
Ph
O
O
Ph
S
OMe
PPM
200
175
150
125
100
75
50
25
0

**3q**


7.973
7.947
7.317
7.291
7.260
7.247
7.239
7.223
7.198
7.177
7.160
6.963
6.935
6.909
6.896
6.889
6.691
5.960
4.208
4.172
4.147
4.133
4.111
3.985
3.947
3.588
3.413
3.403
3.352
3.342
2.439
1.574
0.000
DFILE D:\李润涛\WF09010406-A-H.
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
Ph
Ph
MeO
OMe
2.0516
17.0302
6.4064
1.0115
1.0000
1.3592
4.1969
3.0343
1.0825
3.1354
PPM
198.3
172.9
158.6
150.8
148.7
144.4
136.6
134.3
130.7
129.4
129.0
128.9
128.5
128.2
128.1
127.9
127.5
126.0
123.1
122.0
111.7
111.0
81.51
77.32
77.00
76.68
55.90
55.54
42.91
40.34
21.68
BRUKER
NAME wf09010406-A
EXPNO 2
PROCNO 1
Date_ 20090114
Time_ 16.52
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 28409.092 Hz
FIDRES 0.433488 Hz
AQ 1.1534836 sec
RG 2050
DW 17.600 use
DE 6.50 use
TE 296.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.36 use
PL1 0.00 dB
PL1W 35.66878891 W
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 use
PL2 -1.00 dB
PL12 12.67 dB
PL13 20.00 dB
PL2W 11.92178631 W
PL12W 0.51208419 W
PL13W 0.09469812 W
SFO2 400.1516006 MHz
SI 32768
SF 100.6177985 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
130.752
129.432
129.016
128.991
128.537
128.243
128.164
127.975
127.568
126.000
ppm
Ph
Ph
MeO
OMe

**3r**

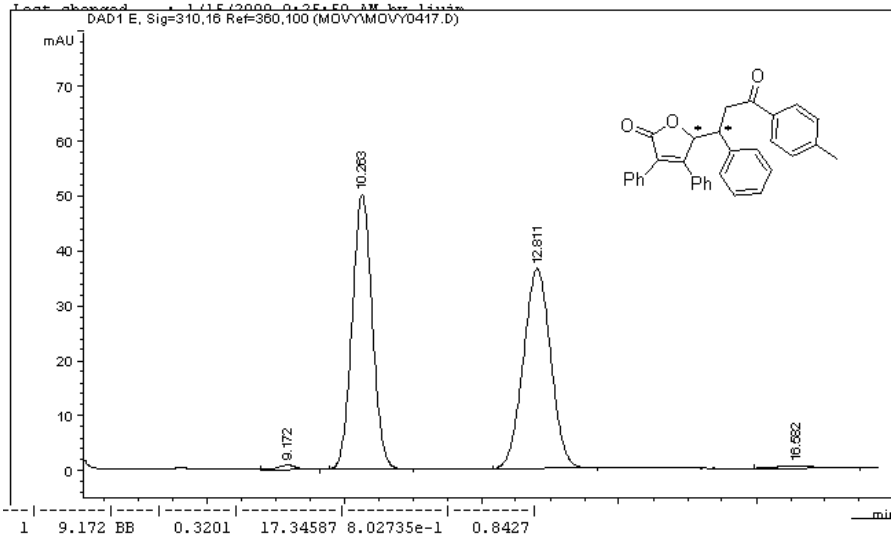

wf09092708
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.882 sec
Width 7988.4 Hz
16 repetitions
OBSERVE H1, 499.9018999 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
Ph
Me
ppm


wf09092708
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1440 repetitions
OBSERVE C13, 125.7004346 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 16 hr, 25 min, 19 sec
Ph
Me
ppm

# HPLC Analyses for Michael product 3

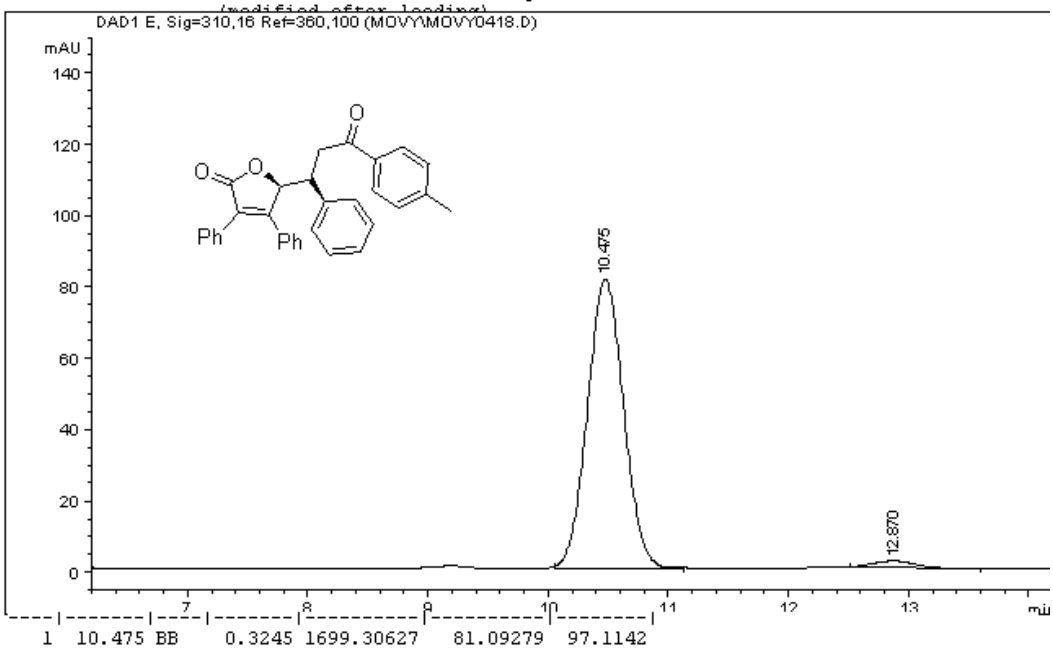
## 3a

```
Injection Date  : 1/15/2009 9:35:08 AM
Sample Name     : wf09011301re
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
```



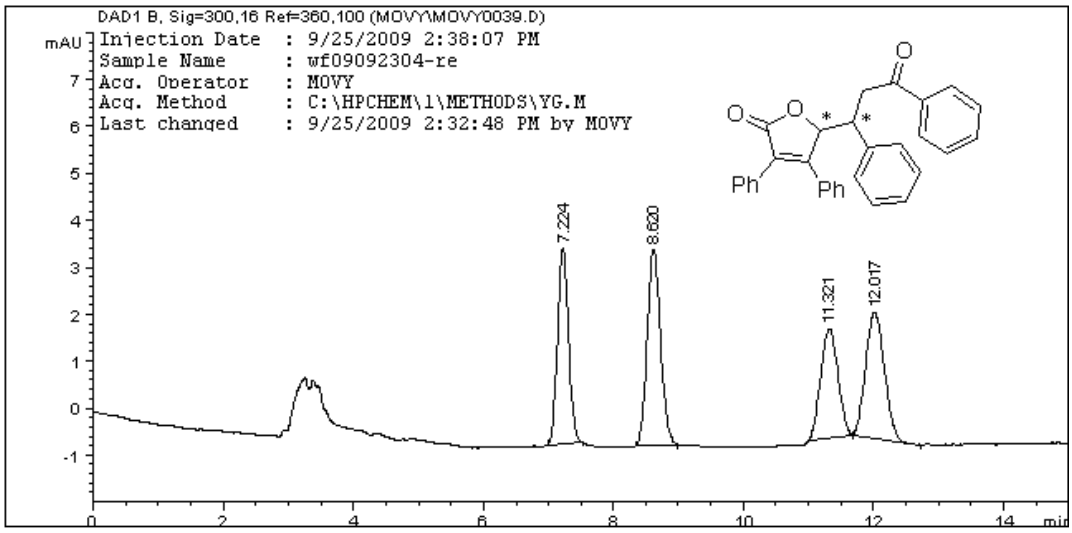

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | 9.172 | BB | 0.3201 | 17.34587 | 8.02735e-1 | 0.8427 | |
| 2 | 10.263 | BB | 0.3178 | 1019.39410 | 50.01398 | 49.5215 | |
| 3 | 12.811 | BB | 0.4319 | 1008.49298 | 36.32982 | 48.9920 | |
| 4 | 16.582 | BB | 0.4562 | 13.25402 | 3.68015e-1 | 0.6439 | |

```
Injection Date  : 1/15/2009 9:55:07 AM
Sample Name     : wf09011301
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/15/2009 9:25:50 AM by liujm
                  (modified after loading)
```

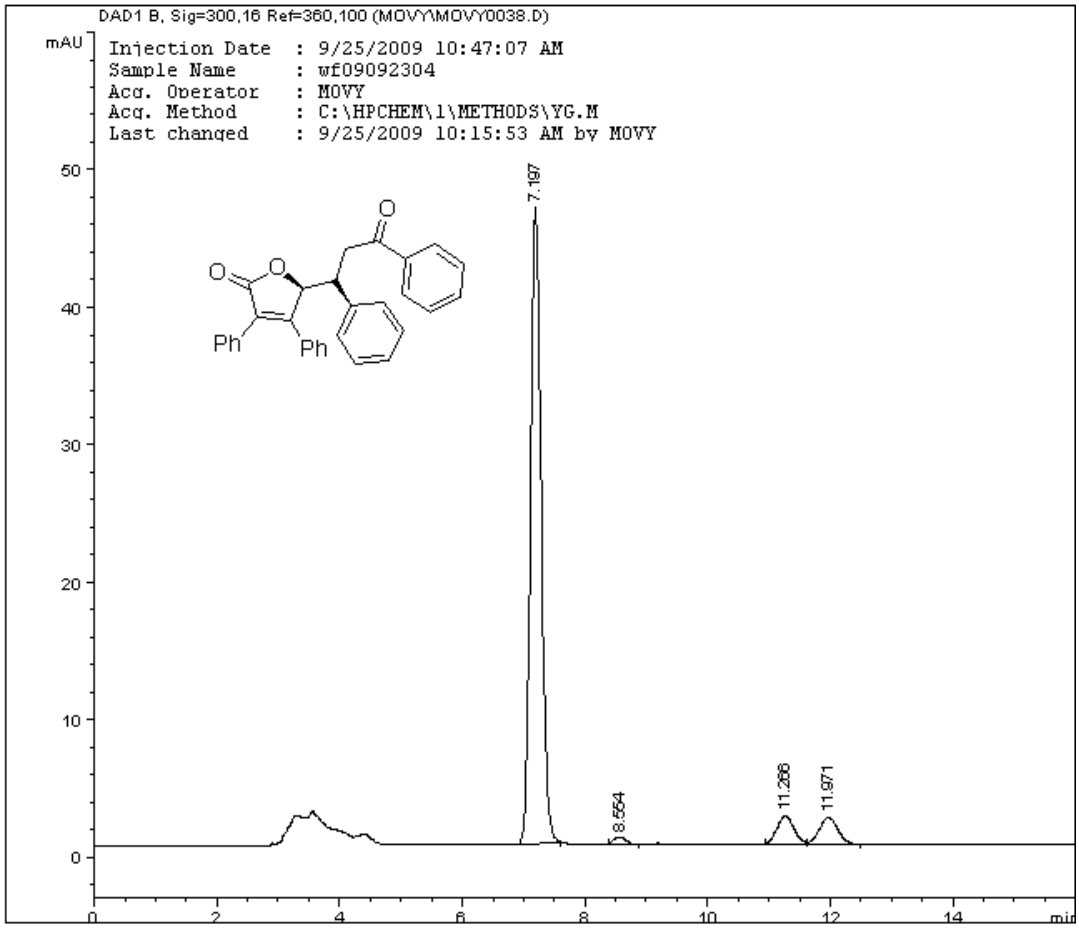



| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | 10.475 | BB | 0.3245 | 1699.30627 | 81.09279 | 97.1142 | |
| 2 | 12.870 | BP | 0.4171 | 50.49509 | 1.90619 | 2.8858 | |

**3b**

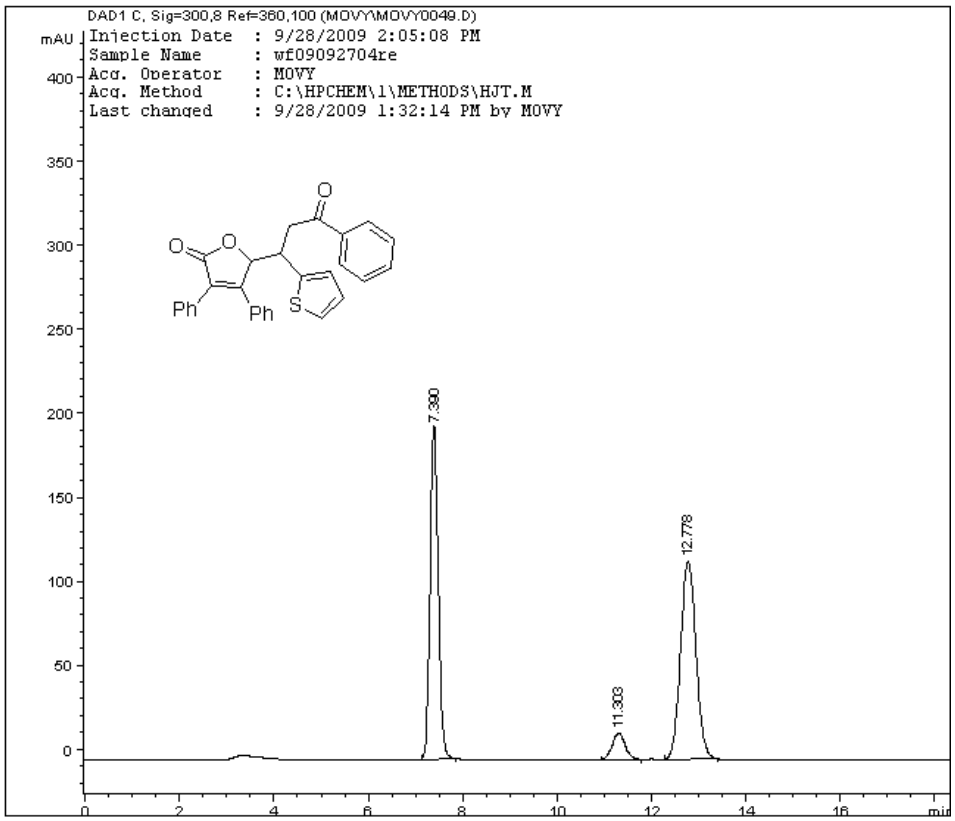



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.224 | BBA | 0.1808 | 48.94807 | 4.17576 | 23.9320 |
| 2 | 8.620 | BBA | 0.2229 | 59.42868 | 4.15327 | 29.0563 |
| 3 | 11.321 | BB | 0.2774 | 42.54493 | 2.32572 | 20.8014 |
| 4 | 12.017 | BBA | 0.3117 | 53.60776 | 2.67635 | 26.2103 |



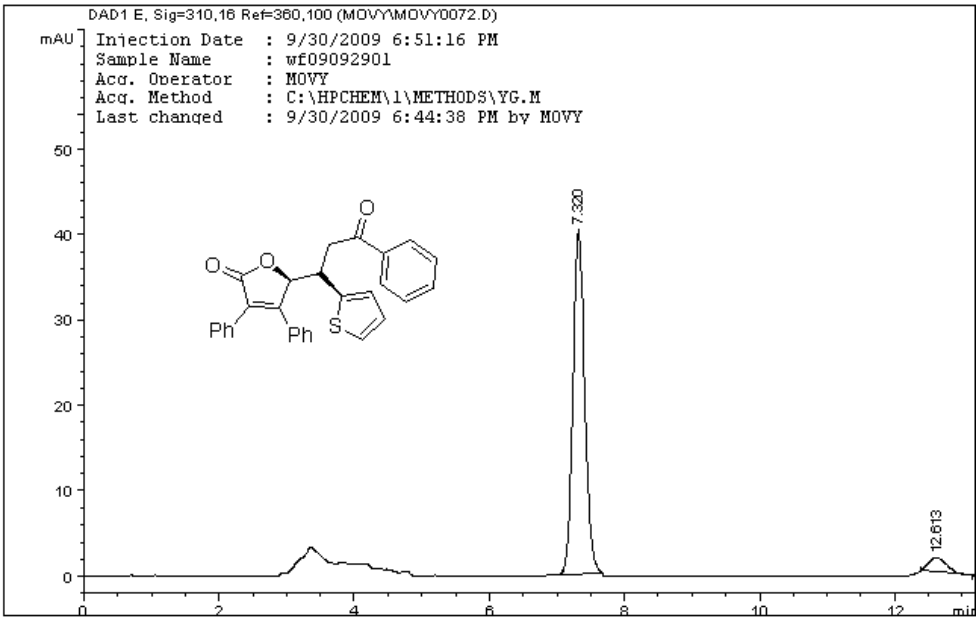

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.197 | BB | 0.1833 | 552.01685 | 46.24564 | 86.9135 |
| 2 | 8.554 | BP | 0.1911 | 6.22492 | 4.87091e-1 | 0.9801 |
| 3 | 11.266 | BV | 0.2949 | 37.29797 | 1.97023 | 5.8725 |
| 4 | 11.971 | VB | 0.3062 | 39.59422 | 1.92351 | 6.2340 |

## 3c



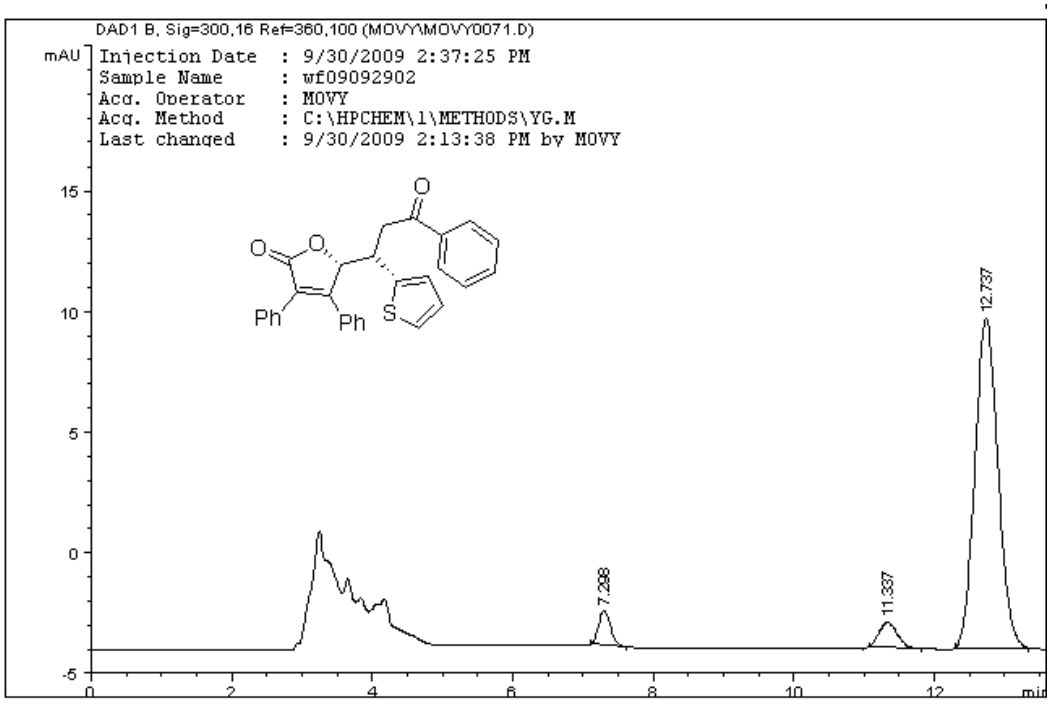

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.390 | BB | 0.1841 | 2376.09521 | 197.84175 | 44.6463 |
| 2 | 11.303 | BB | 0.2927 | 292.27112 | 15.59276 | 5.4917 |
| 3 | 12.778 | BB | 0.3487 | 2653.67773 | 117.87108 | 49.8620 |

## 3c1



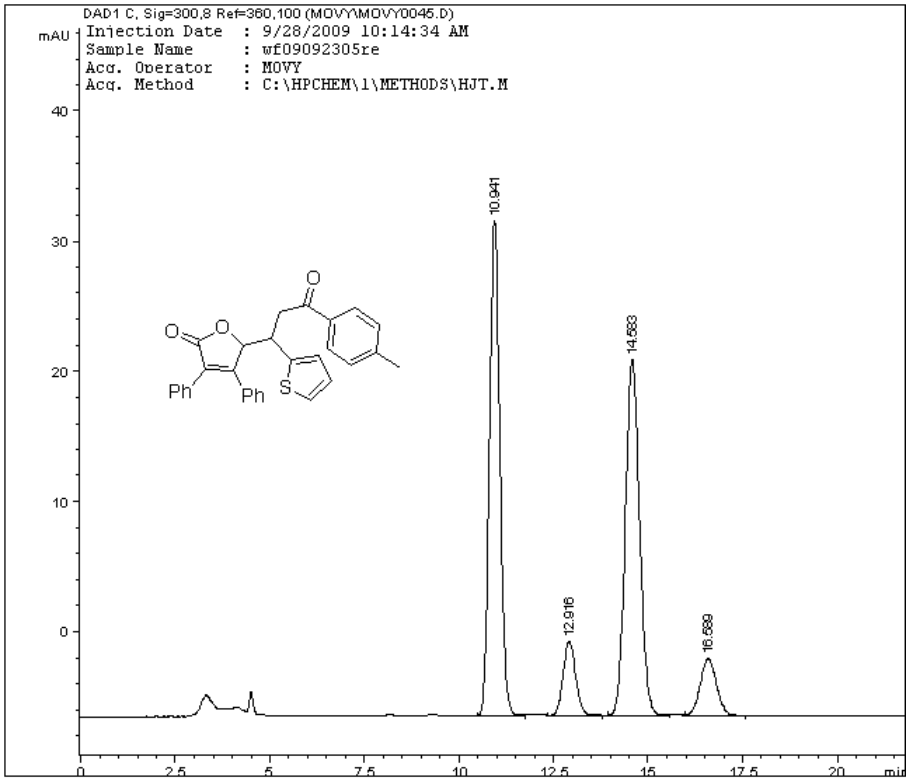

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.320 | BB | 0.1882 | 491.57642 | 40.33688 | 94.0119 |
| 2 | 12.613 | BP | 0.3066 | 31.31087 | 1.64130 | 5.9881 |

**3c2**

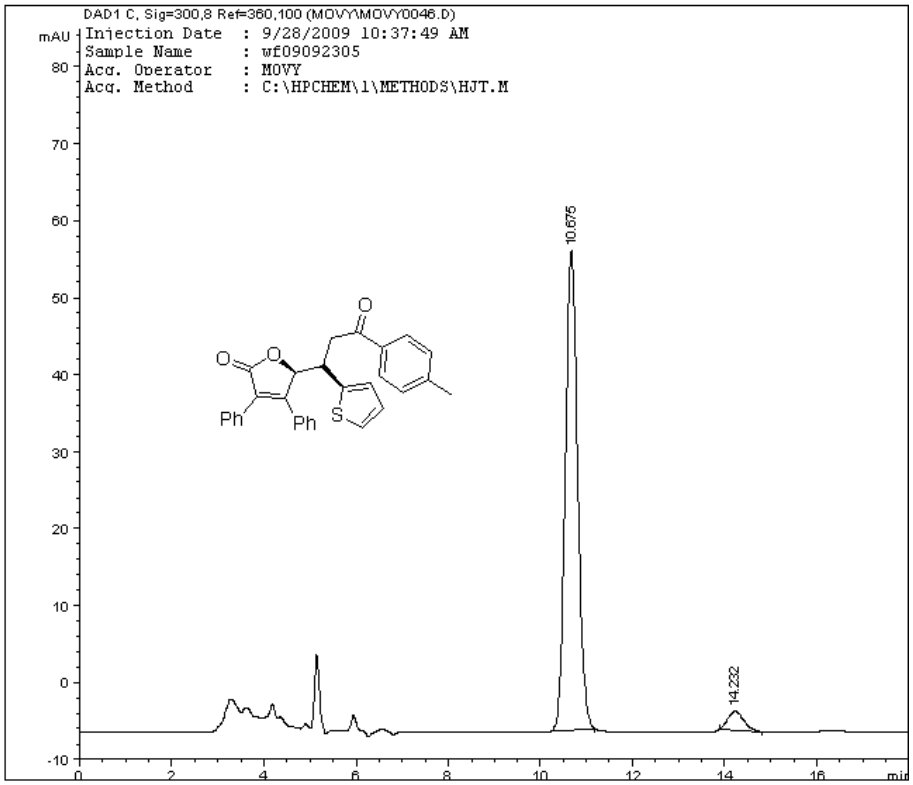



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.298 | BB | 0.1820 | 16.79106 | 1.41914 | 4.8320 |
| 2 | 11.337 | BB | 0.2646 | 18.58061 | 1.02949 | 5.3470 |
| 3 | 12.737 | BB | 0.3539 | 312.12695 | 13.69407 | 89.8211 |

## 3d



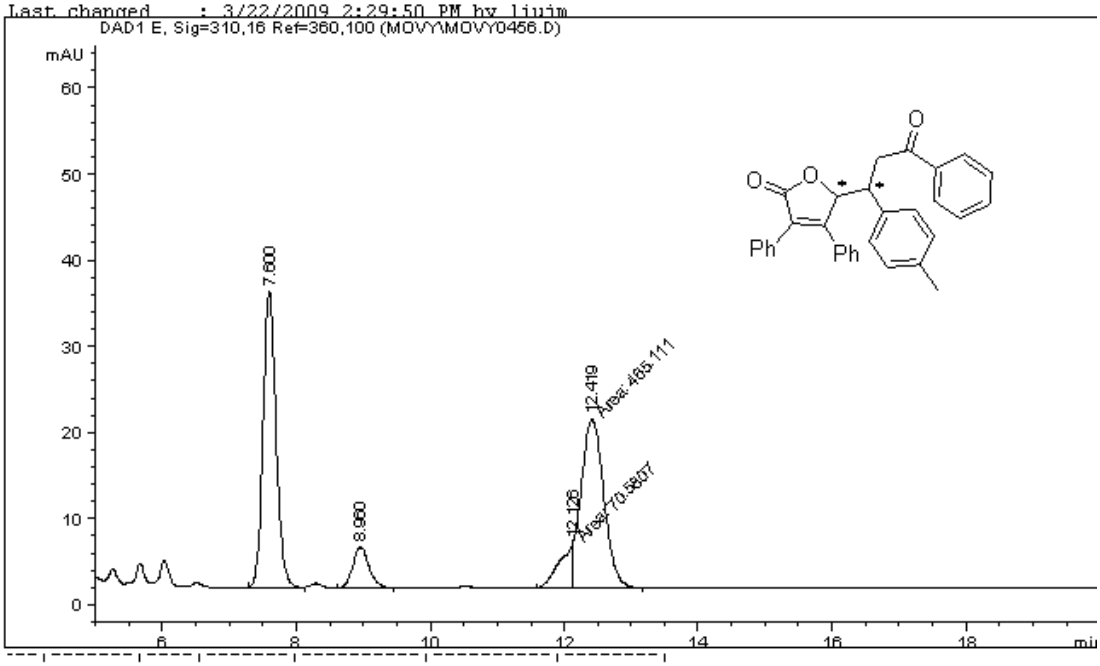

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.941 | BB | 0.2987 | 725.84155 | 38.02822 | 42.4352 |
| 2 | 12.916 | VB | 0.3606 | 133.87436 | 5.72961 | 7.8268 |
| 3 | 14.583 | BB | 0.4106 | 718.97174 | 27.35441 | 42.0335 |
| 4 | 16.589 | BB | 0.4692 | 131.78377 | 4.40238 | 7.7045 |




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.675 | BB | 0.2898 | 1164.45569 | 62.38278 | 95.4144 |
| 2 | 14.232 | BP | 0.3574 | 55.96334 | 2.38771 | 4.5856 |

## 3e

```
==================================================
Injection Date  : 3/22/2009 2:35:54 PM
Sample Name     : wf09010909re                    I
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 3/22/2009 2:29:50 PM by liuim
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.600 | BV | 0.2080 | 464.87949 | 34.32478 | 43.1298 |
| 2 | 8.960 | VP | 0.2539 | 77.29051 | 4.69904 | 7.1707 |
| 3 | 12.126 | MF | 0.2323 | 70.58074 | 5.06338 | 6.5482 |
| 4 | 12.419 | FM | 0.3957 | 465.11078 | 19.58941 | 43.1513 |

```
Injection Date  : 1/12/2009 12:10:41 PM
Sample Name     : wf09010909
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 9:20:28 AM by MOVY
                  (modified after loading)
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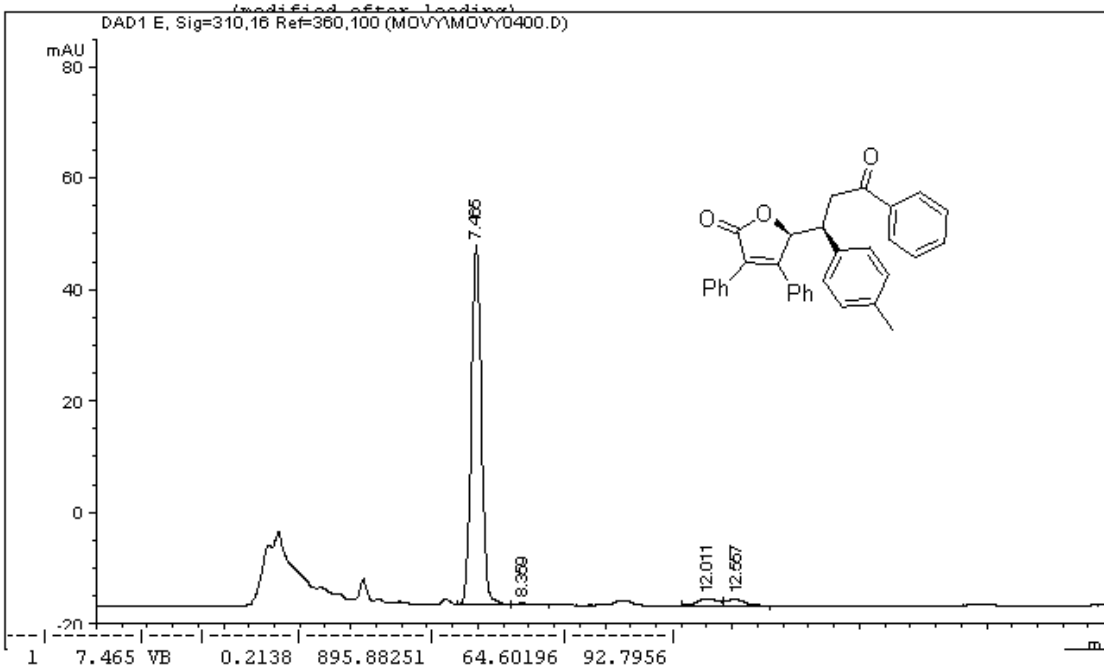


| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.465 | VB | 0.2138 | 895.88251 | 64.60196 | 92.7956 |
| 2 | 8.359 | BB | 0.2302 | 5.07192 | 3.64754e-1 | 0.5253 |
| 3 | 12.011 | BV | 0.4216 | 35.46931 | 1.28688 | 3.6739 |
| 4 | 12.557 | VB | 0.4089 | 29.01265 | 1.09577 | 3.0051 |

==========

## 3f

```
Injection Date  : 1/7/2009 9:58:23 AM
Sample Name     : 09010501                          L
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/7/2009 9:20:41 AM by liujm
                  (modified after loading)
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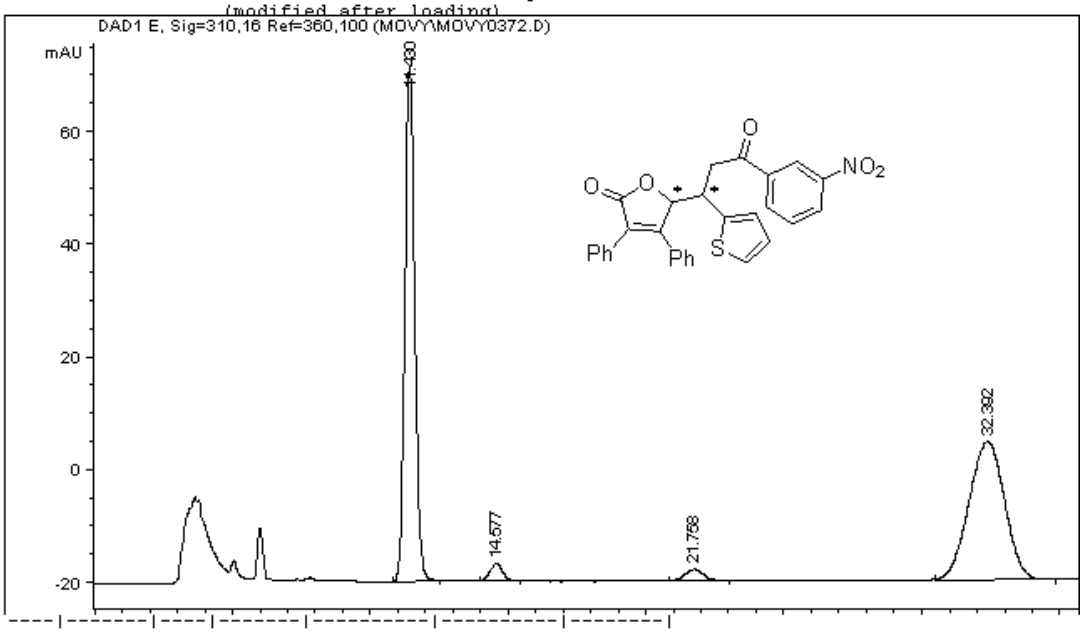
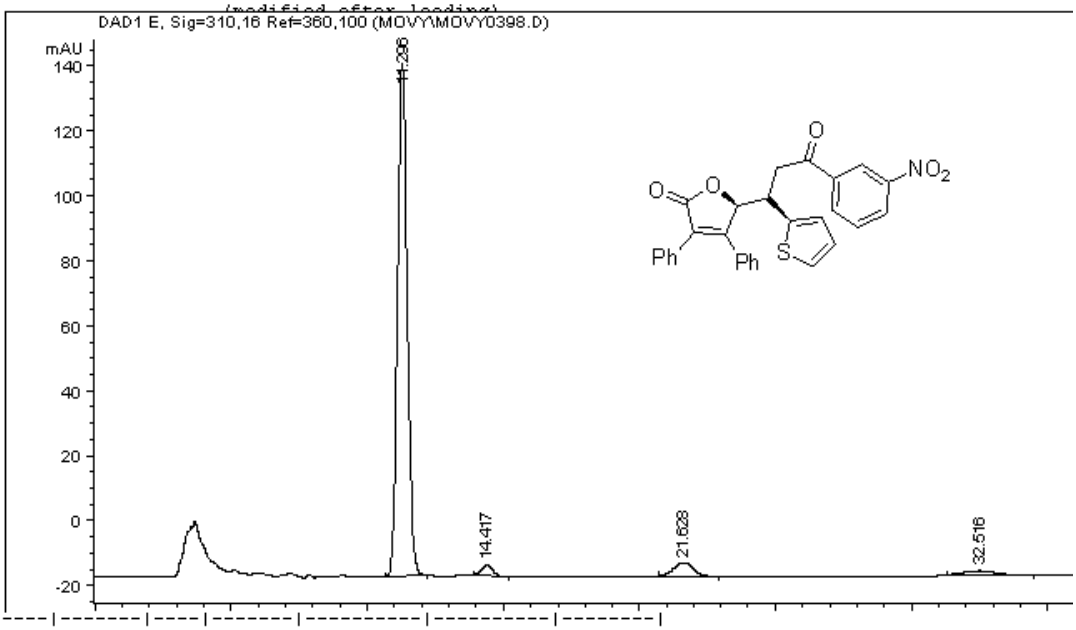



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 11.430 | BB | 0.3775 | 2232.20898 | 91.21676 | 48.1078 |
| 2 | 14.577 | BB | 0.4887 | 95.85977 | 3.06674 | 2.0659 |
| 3 | 21.758 | PP | 0.6818 | 93.30087 | 1.97973 | 2.0108 |
| 4 | 32.392 | BB | 1.4224 | 2218.64941 | 24.54834 | 47.8155 |

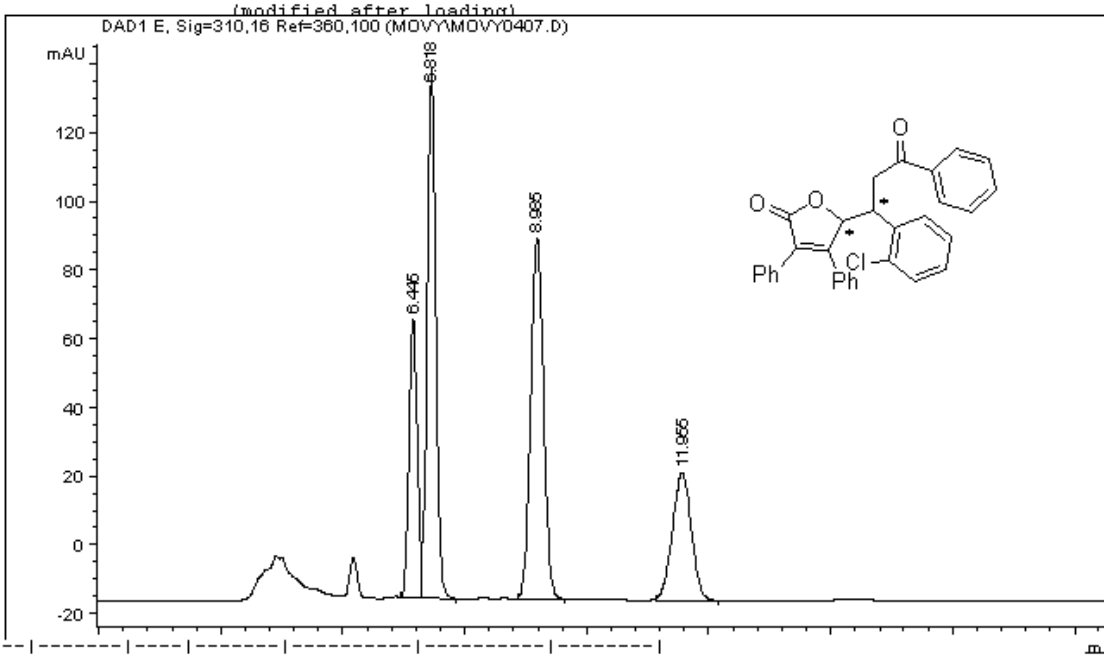
```
Injection Date  : 1/12/2009 10:54:49 AM
Sample Name     : wf09010601
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 9:20:28 AM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 11.296 | BB | 0.3881 | 3960.13452 | 158.14342 | 90.8196 |
| 2 | 14.417 | BP | 0.4019 | 88.72993 | 3.11916 | 2.0349 |
| 3 | 21.628 | BP | 0.6046 | 207.40509 | 4.25556 | 4.7565 |
| 4 | 32.516 | BB | 0.9345 | 104.17231 | 1.31740 | 2.3890 |

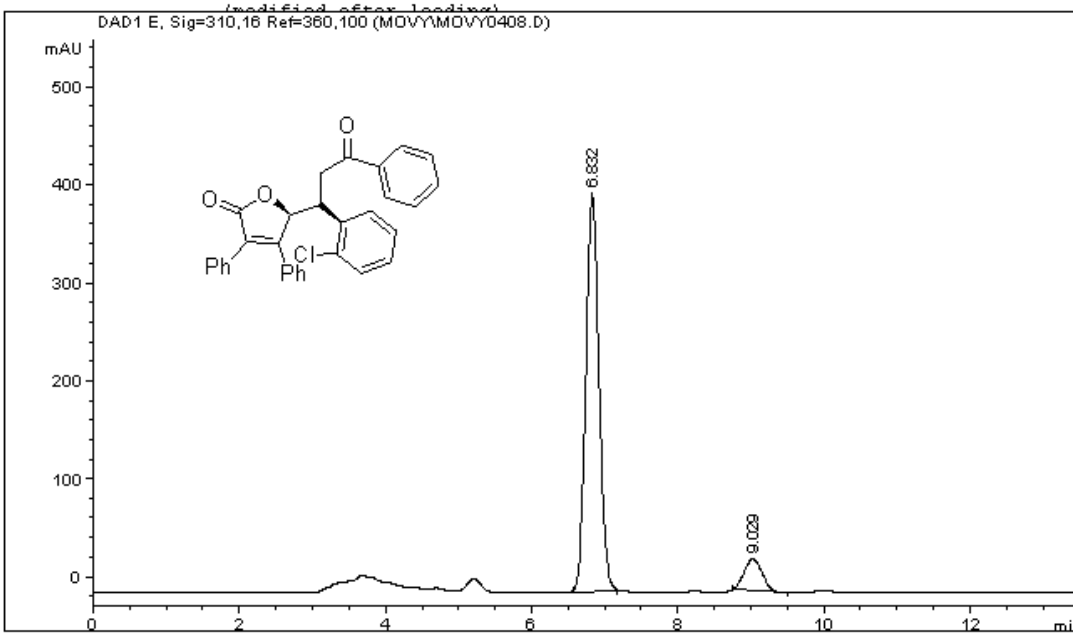
**3g**

```
Injection Date  : 1/12/2009 4:56:12 PM
Sample Name     : wf09010908re
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 6.445 | BV | 0.1887 | 979.50385 | 81.22973 | 16.8738 |
| 2 | 6.818 | VP | 0.1932 | 1920.23083 | 154.29214 | 33.0795 |
| 3 | 8.985 | BP | 0.2839 | 1902.84985 | 104.77612 | 32.7801 |
| 4 | 11.955 | BB | 0.4247 | 1002.30908 | 36.92279 | 17.2666 |

```
Injection Date  : 1/12/2009 5:19:24 PM
Sample Name     : wf09010908
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 6.832 | BB | 0.1932 | 5053.09033 | 406.12128 | 90.0809 |
| 2 | 9.029 | BP | 0.2717 | 556.41461 | 32.49842 | 9.9191 |

## 3h

```
Injection Date  : 1/8/2009 4:37:51 PM
Sample Name     : wf09010407                        I
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/8/2009 4:01:34 PM by MOVY
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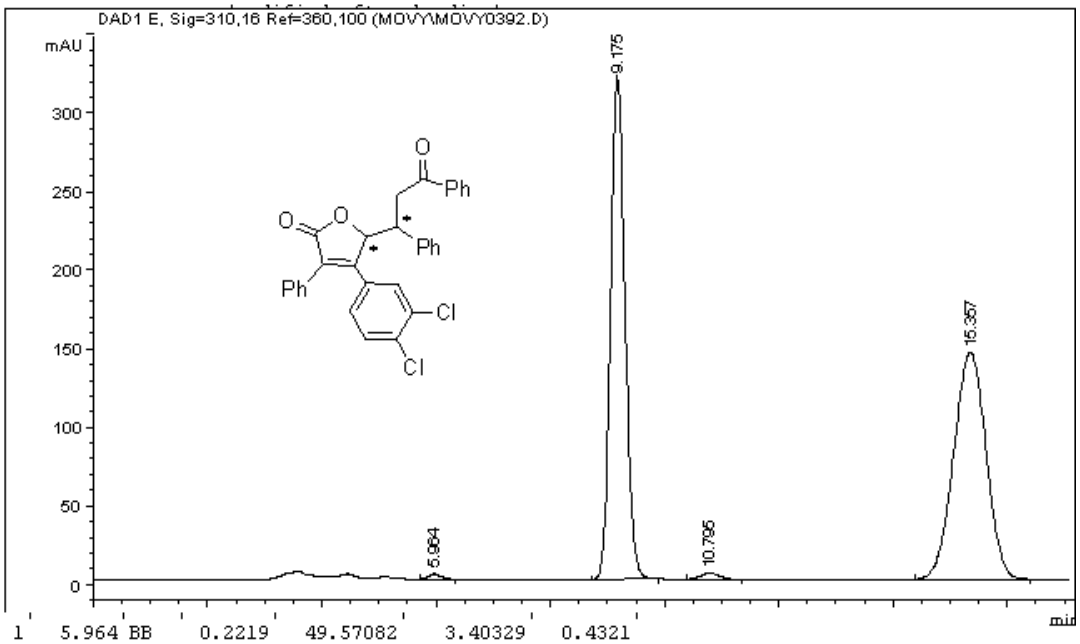
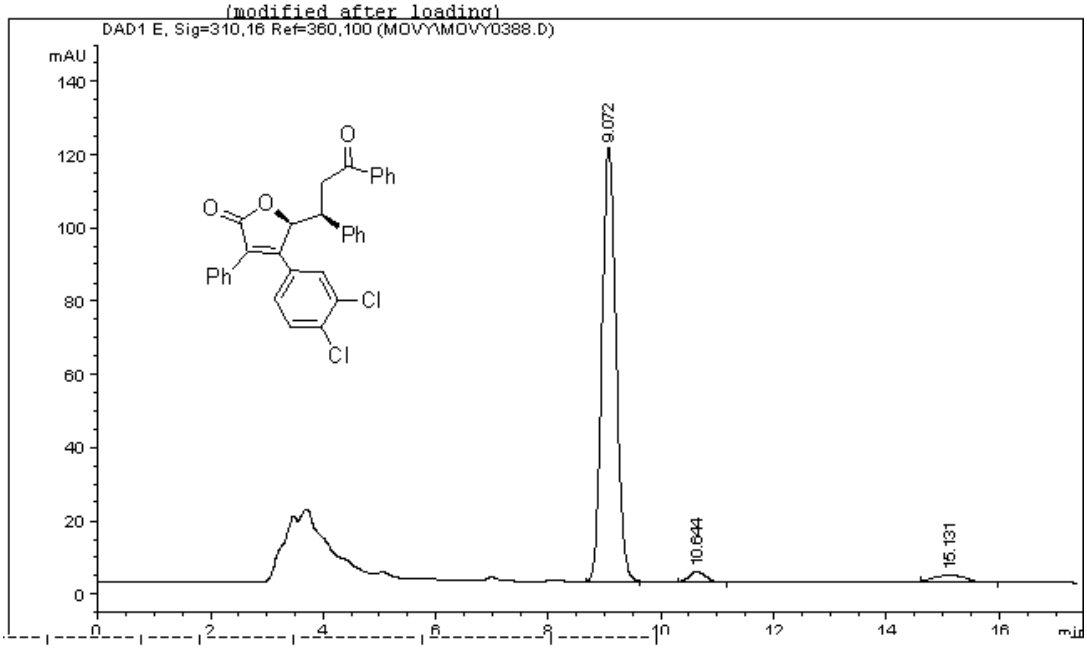


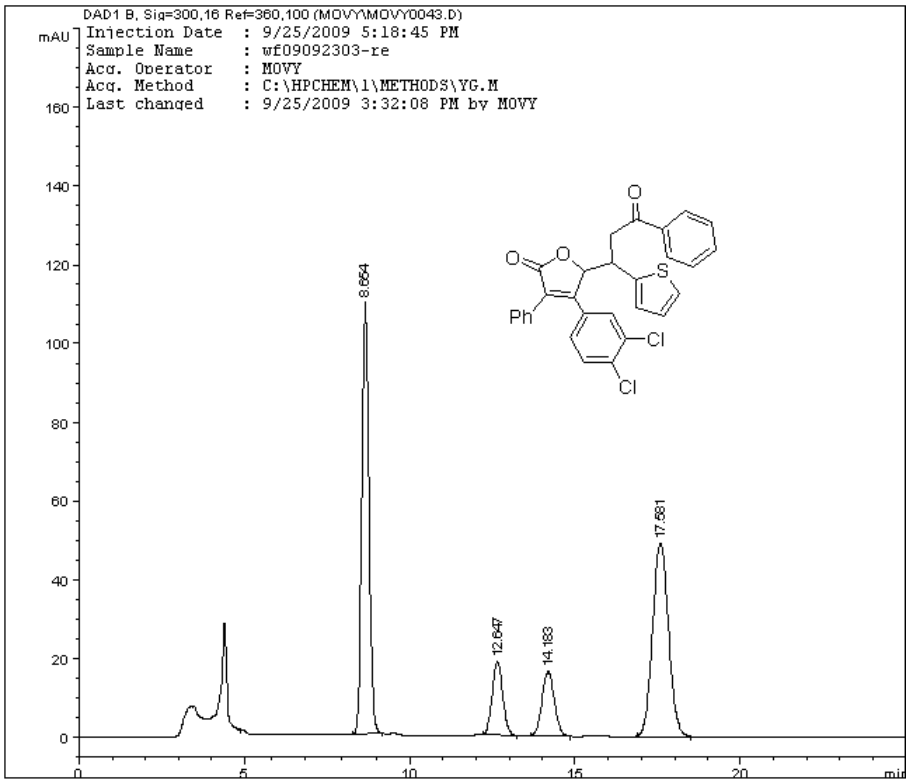

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 5.964 | BB | 0.2219 | 49.57082 | 3.40329 | 0.4321 |
| 2 | 9.175 | BB | 0.2760 | 5652.32422 | 320.09763 | 49.2714 |
| 3 | 10.795 | BB | 0.3183 | 83.82024 | 4.00325 | 0.7307 |
| 4 | 15.357 | BB | 0.5927 | 5686.09326 | 144.61058 | 49.5658 |

```
Injection Date  : 1/8/2009 2:58:44 PM
Sample Name     : wf09010602                        I
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/8/2009 2:22:19 PM by Linshi
                  (modified after loading)
```

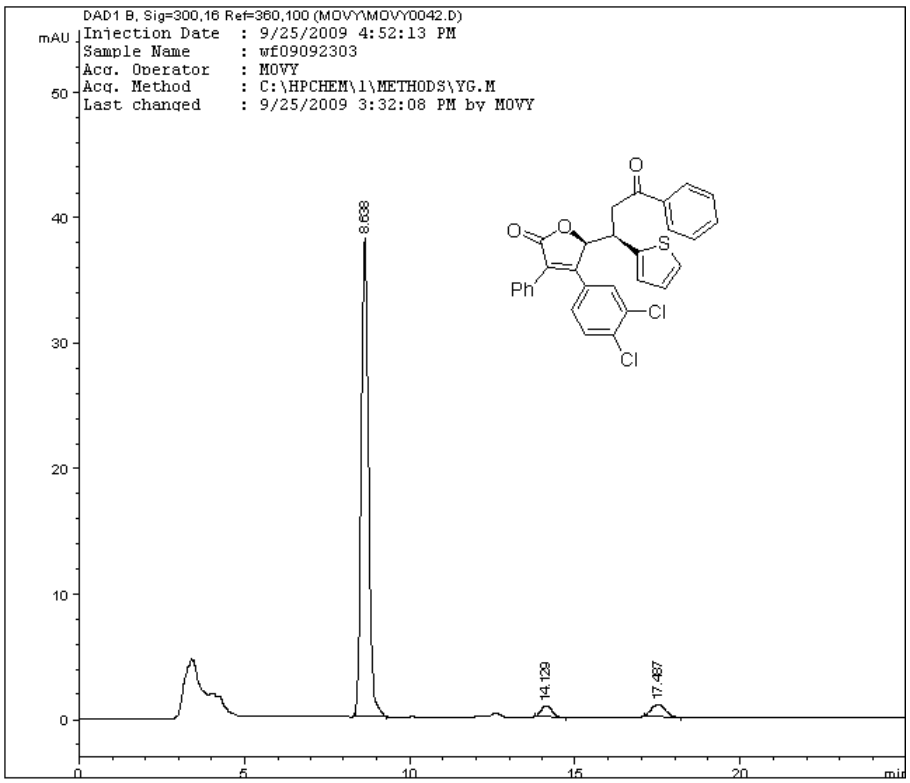



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 9.072 | BB | 0.2683 | 2051.10522 | 118.31512 | 94.5836 |
| 2 | 10.644 | BP | 0.3053 | 53.39702 | 2.64784 | 2.4623 |
| 3 | 15.131 | BP | 0.4116 | 64.06063 | 1.88927 | 2.9541 |

**3i**

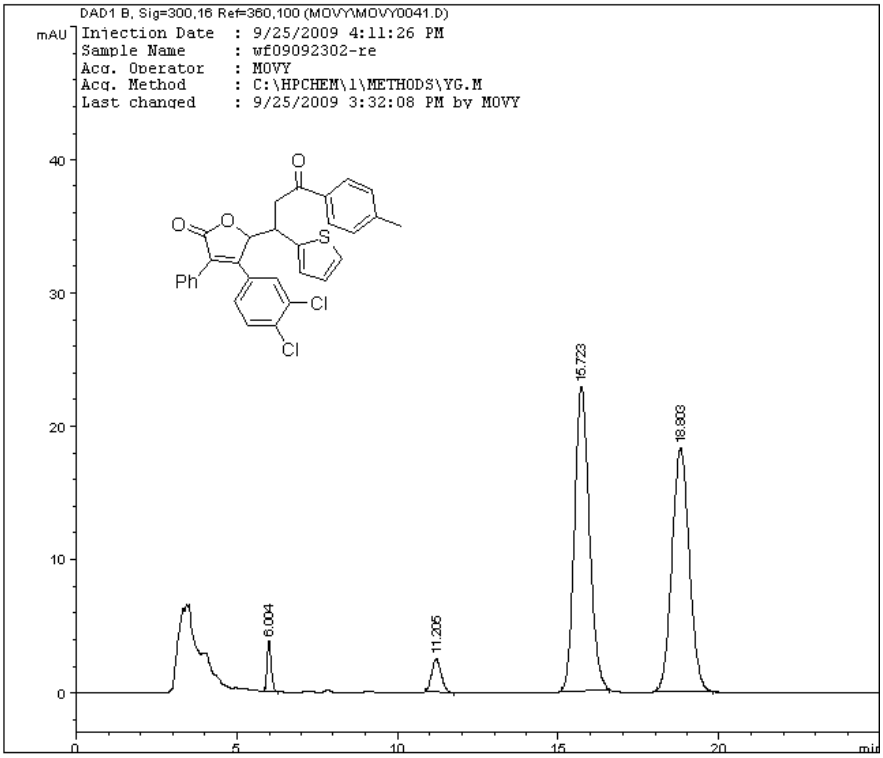



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 8.654 | VB | 0.2329 | 1640.74670 | 109.47485 | 39.7772 |
| 2 | 12.647 | BB | 0.3473 | 417.25119 | 18.76987 | 10.1156 |
| 3 | 14.183 | BB | 0.4035 | 426.89191 | 16.41112 | 10.3493 |
| 4 | 17.581 | BB | 0.5227 | 1639.95032 | 49.22349 | 39.7579 |

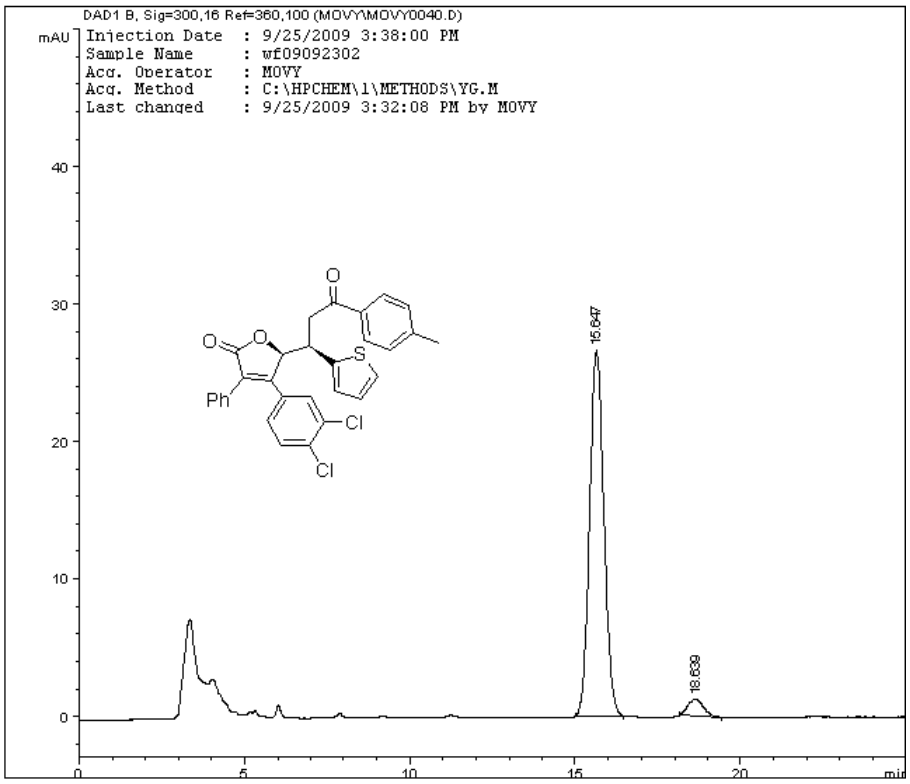



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 8.638 | BB | 0.2358 | 580.98608 | 38.12855 | 92.0706 |
| 2 | 14.129 | BB | 0.2971 | 20.95009 | 9.08199e-1 | 3.3200 |
| 3 | 17.487 | BP | 0.3659 | 29.08608 | 9.64409e-1 | 4.6094 |

**3i**

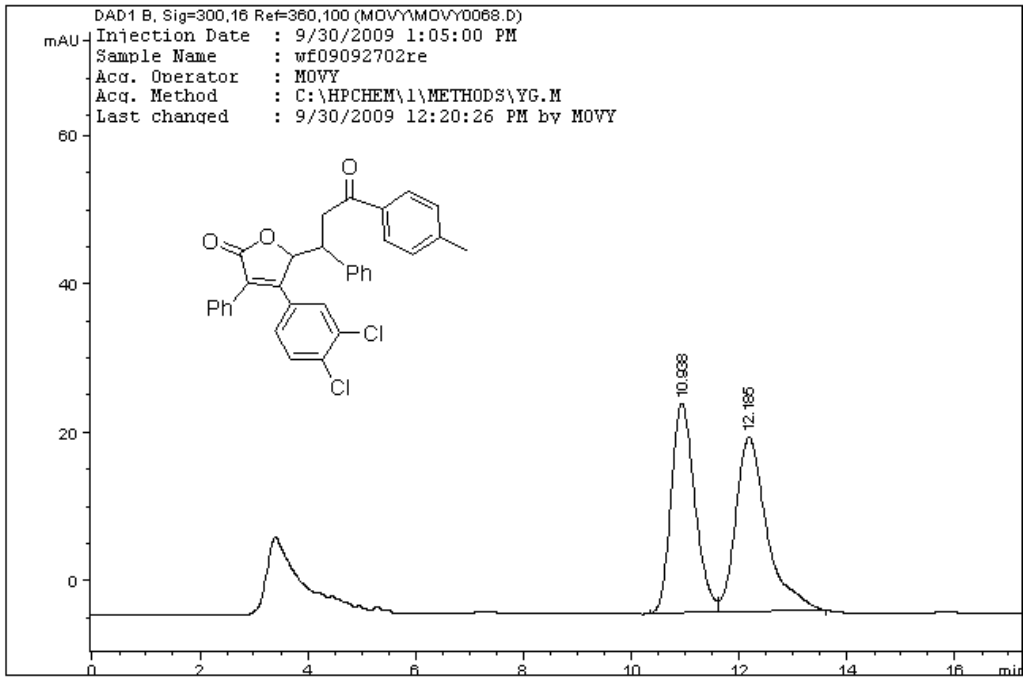



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 6.004 | BB | 0.1349 | 33.45866 | 3.79409 | 2.2335 |
| 2 | 11.205 | BP | 0.3083 | 50.22109 | 2.47992 | 3.3524 |
| 3 | 15.723 | BB | 0.4957 | 734.32947 | 22.80445 | 49.0190 |
| 4 | 18.803 | BB | 0.5733 | 680.04047 | 18.22723 | 45.3951 |



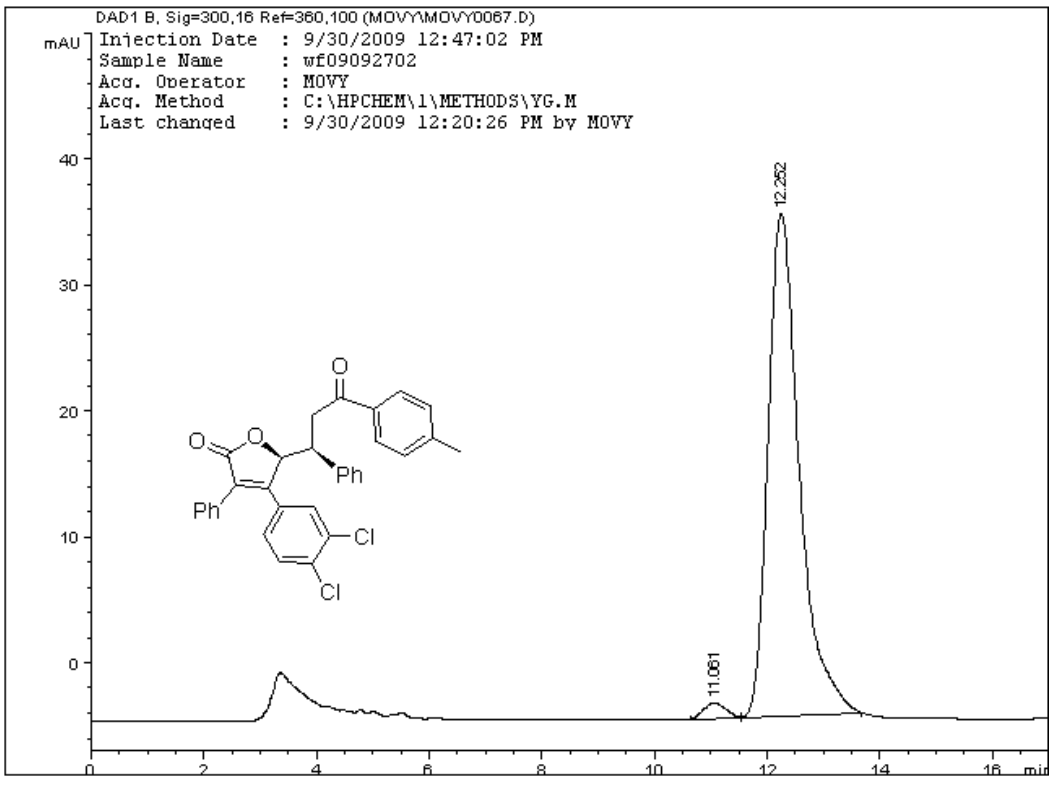

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 15.647 | BB | 0.4654 | 802.83997 | 26.65538 | 95.4830 |
| 2 | 18.639 | BB | 0.3930 | 37.98014 | 1.16304 | 4.5170 |

## 3k



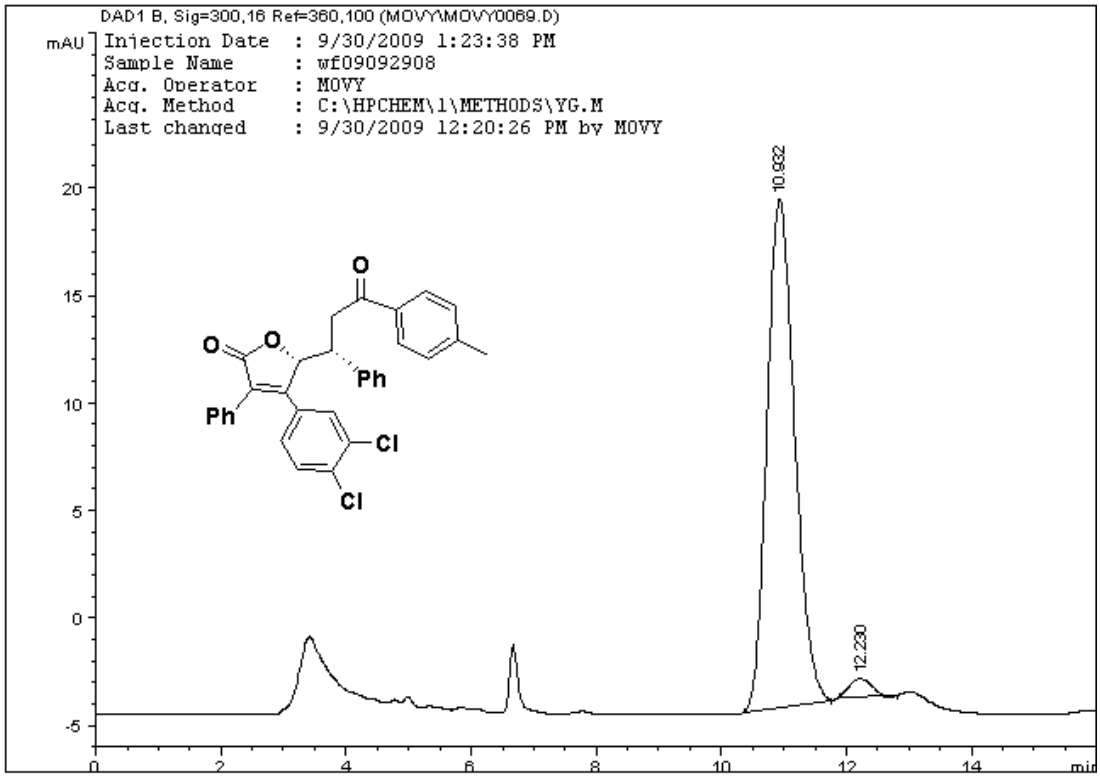

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.938 | BV | 0.4775 | 868.35950 | 28.17914 | 47.6844 |
| 2 | 12.185 | VB | 0.6045 | 952.69519 | 23.52096 | 52.3156 |

## 3k1




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 11.061 | BV | 0.3475 | 33.19333 | 1.20446 | 2.1072 |
| 2 | 12.252 | VB | 0.5837 | 1542.00647 | 39.82727 | 97.8928 |

**3k2**




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.932 | BB | 0.4741 | 724.20502 | 23.59231 | 97.4304 |
| 2 | 12.230 | BP | 0.2867 | 19.09976 | 8.11312e-1 | 2.5696 |

**3l**

```
Injection Date  : 1/9/2009 10:43:37 AM
Sample Name     : wf09010603re
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Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/9/2009 9:56:07 AM by MOVY
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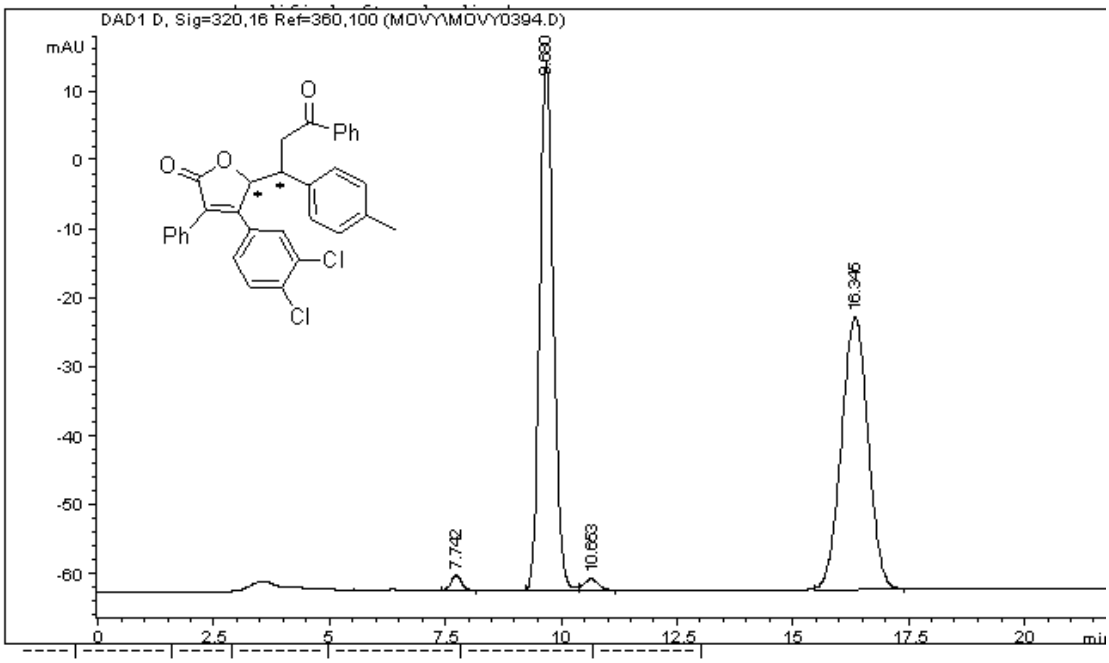
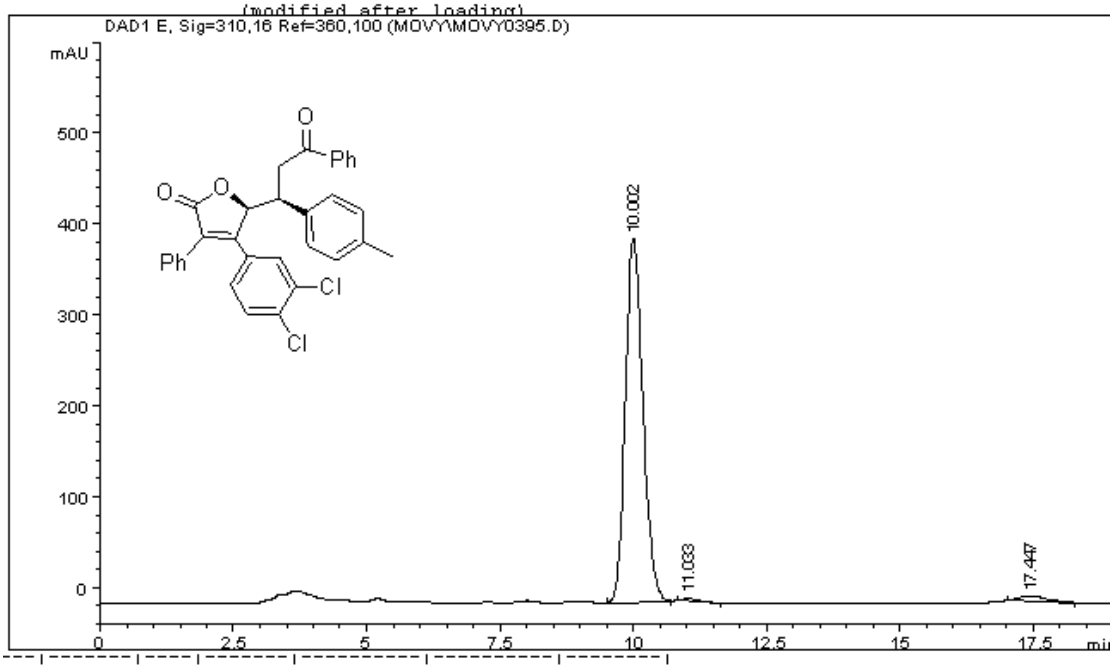


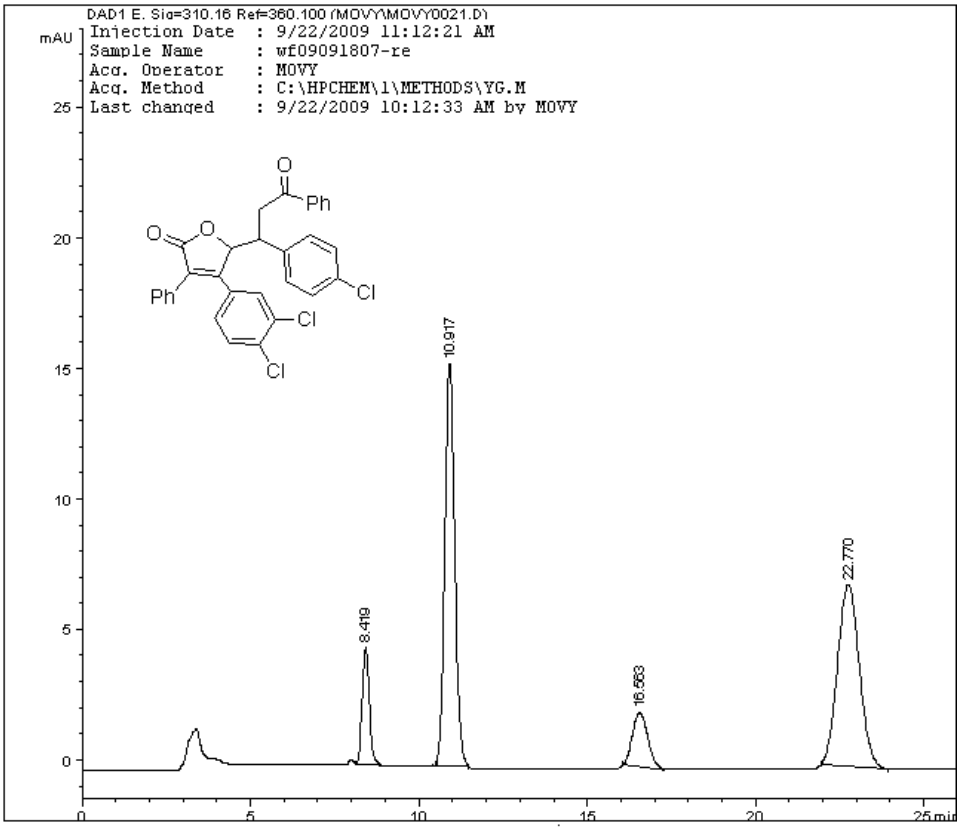

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | 7.742 | BB | 0.2455 | 35.48849 | 2.25631 | 1.0969 |
| 2 | 9.680 | BB | 0.3209 | 1583.55957 | 76.67655 | 48.9451 |
| 3 | 10.653 | BB | 0.3295 | 35.58815 | 1.63767 | 1.1000 |
| 4 | 16.345 | BB | 0.6268 | 1580.73987 | 39.50880 | 48.8580 |

```
Injection Date  : 1/12/2009 9:46:34 AM
Sample Name     : wf09010603                          L
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 9:20:28 AM by MOVY
                  (modified after loading)
```

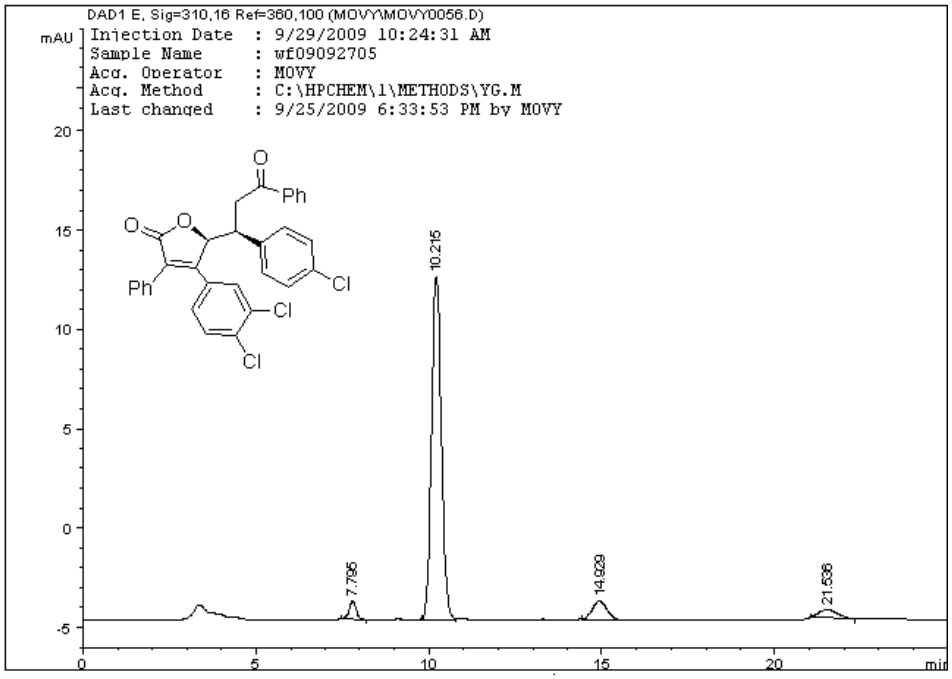



| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.002 | BB | 0.3552 | 9147.00977 | 399.37811 | 97.4499 |
| 2 | 11.033 | BP | 0.2820 | 51.38319 | 3.12197 | 0.5474 |
| 3 | 17.447 | BP | 0.4779 | 187.97829 | 5.24761 | 2.0027 |

## 3m



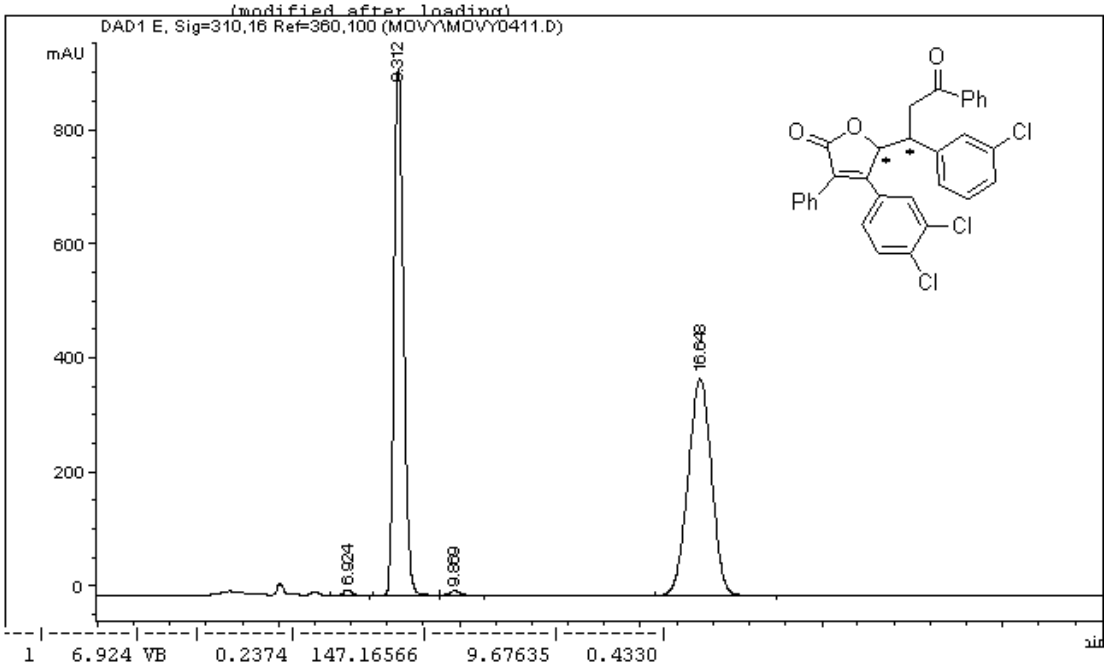

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 8.419 | BB | 0.2253 | 64.13092 | 4.47160 | 8.4401 |
| 2 | 10.917 | BB | 0.3203 | 317.56873 | 15.41871 | 41.7945 |
| 3 | 16.562 | BB | 0.4521 | 65.36770 | 2.06077 | 8.6029 |
| 4 | 22.770 | BB | 0.6758 | 312.76727 | 6.96647 | 41.1625 |




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.795 | BP | 0.2175 | 13.79115 | 9.49371e-1 | 3.5167 |
| 2 | 10.215 | BB | 0.3017 | 337.17154 | 17.27672 | 85.9783 |
| 3 | 14.929 | BP | 0.3468 | 26.49560 | 9.34338e-1 | 6.7563 |
| 4 | 21.536 | BB | 0.4361 | 14.70080 | 4.02555e-1 | 3.7487 |

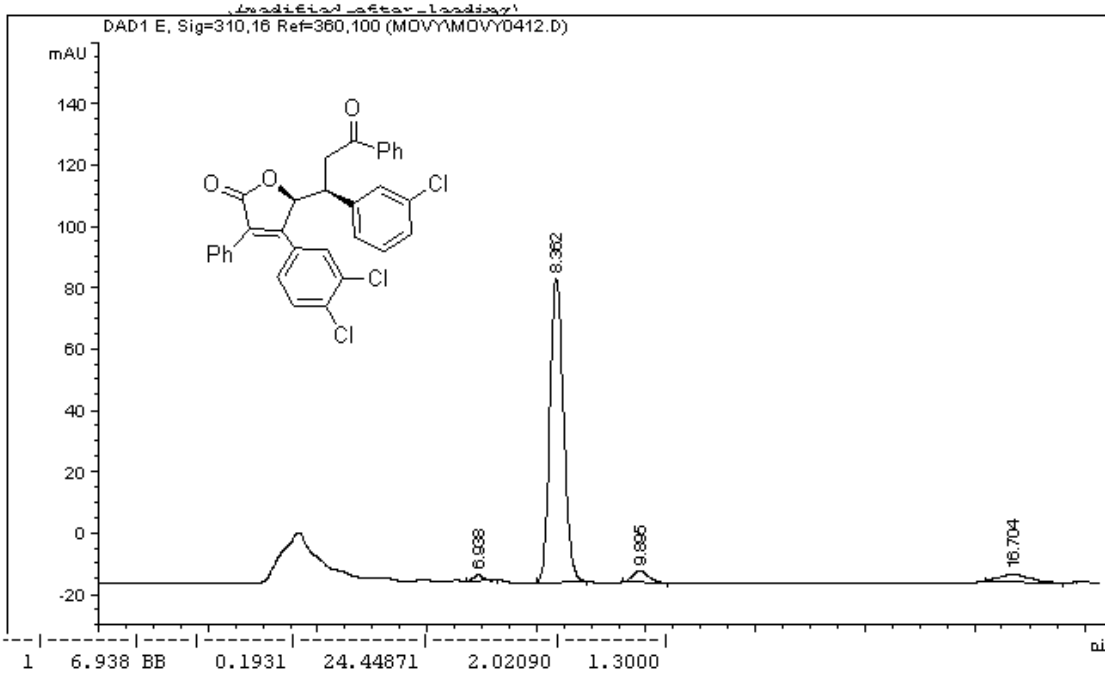
## 3n

```
Injection Date  : 1/12/2009 6:06:45 PM
Sample Name     : wf09010904re
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 6.924 | VB | 0.2374 | 147.16566 | 9.67635 | 0.4330 |
| 2 | 8.312 | BV | 0.2824 | 1.66534e4 | 923.53345 | 48.9983 |
| 3 | 9.869 | VB | 0.3287 | 148.26791 | 6.84395 | 0.4362 |
| 4 | 16.648 | BB | 0.7083 | 1.70389e4 | 379.18185 | 50.1325 |

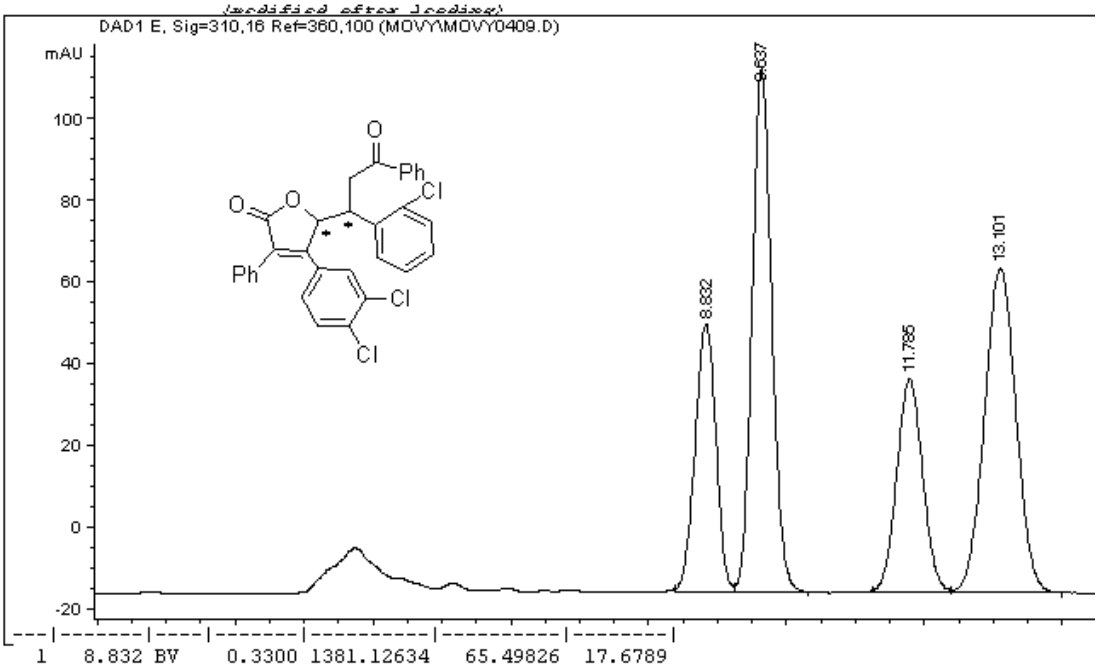
```
Injection Date  : 1/12/2009 6:36:44 PM
Sample Name     : wf09010904                    I
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 6.938 | BB | 0.1931 | 24.44871 | 2.02090 | 1.3000 |
| 2 | 8.362 | BB | 0.2672 | 1704.81372 | 98.86922 | 90.6506 |
| 3 | 9.895 | BB | 0.2970 | 71.44710 | 3.67127 | 3.7991 |
| 4 | 16.704 | BB | 0.4446 | 79.93303 | 2.18626 | 4.2503 |

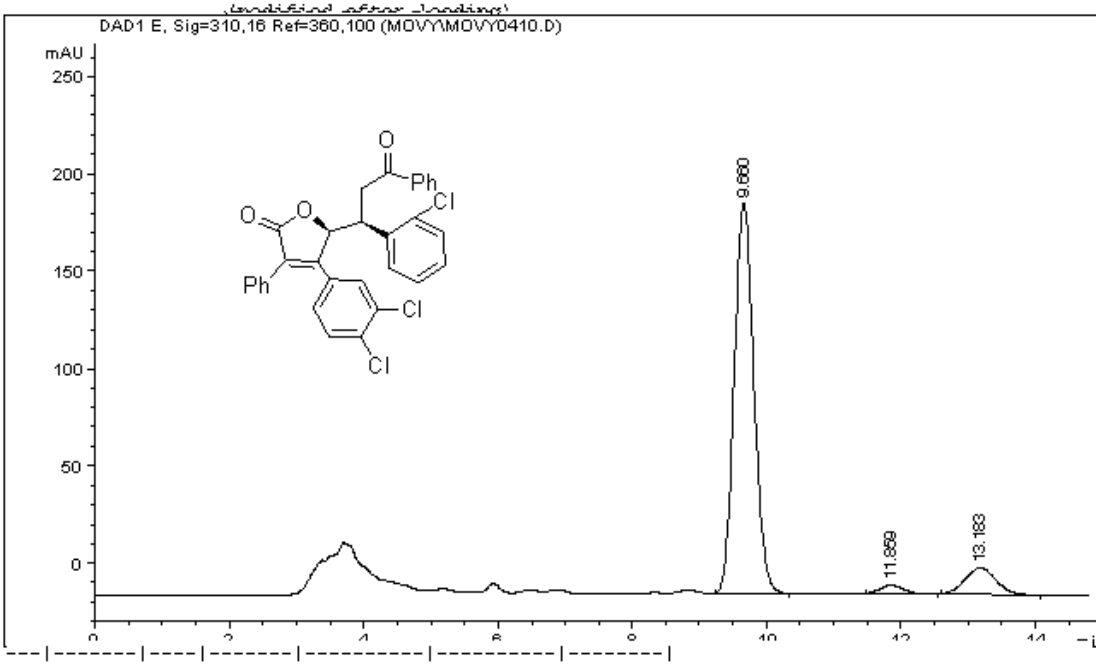
## 3o

```
Injection Date  : 1/12/2009 5:36:00 PM
Sample Name     : wf09010903re                    L
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 | 8.832 | BV | 0.3300 | 1381.12634 | 65.49826 | 17.6789 |
| | 2 | 9.637 | VB | 0.3068 | 2535.49829 | 128.18059 | 32.4552 |
| = | 3 | 11.785 | BV | 0.4102 | 1368.47949 | 52.14366 | 17.5170 |
| | 4 | 13.101 | VB | 0.4999 | 2527.19360 | 79.27847 | 32.3489 |

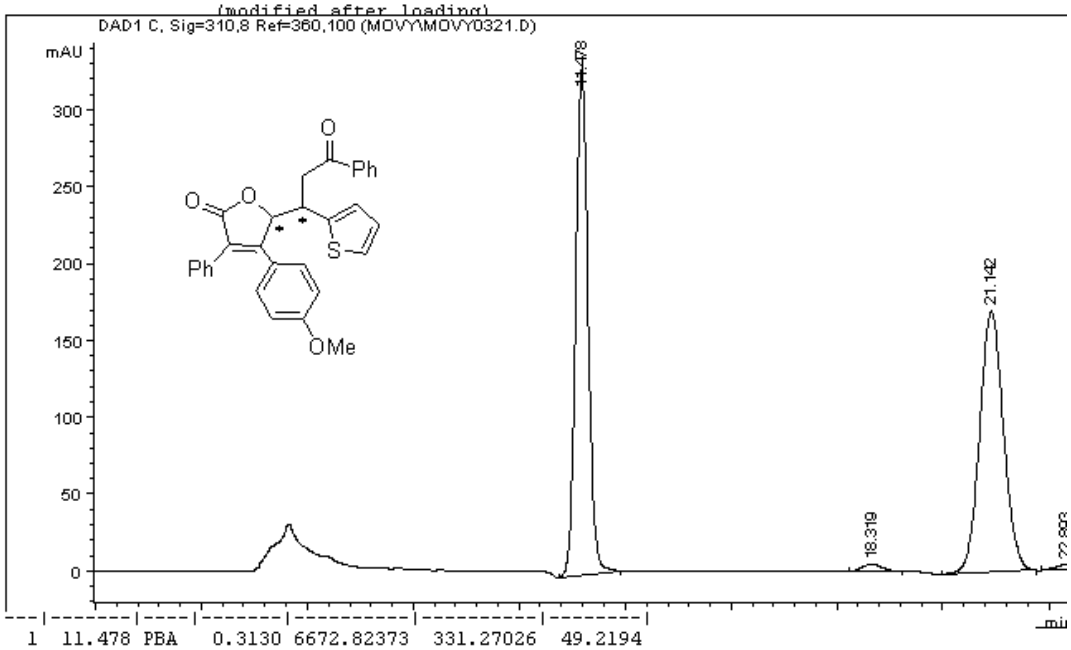
```
Injection Date  : 1/12/2009 5:51:07 PM
Sample Name     : wf09010903
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/12/2009 1:22:16 PM by MOVY
                  (modified after loading)
```




| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 | 9.660 | PP | 0.3200 | 4063.18848 | 200.82997 | 88.5222 |
| | 2 | 11.859 | BP | 0.3852 | 100.28519 | 4.16080 | 2.1849 |
| | 3 | 13.183 | BP | 0.5040 | 426.55075 | 13.37875 | 9.2930 |

## 3p

```
Injection Date  : 12/22/2008 6:38:49 PM
Sample Name     : wf08121610re
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 12/22/2008 3:28:33 PM by MOVY
                  (modified after loading)
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 11.478 | PBA | 0.3130 | 6672.82373 | 331.27026 | 49.2194 |
| 2 | 18.319 | BBA | 0.4252 | 145.45155 | 4.69835 | 1.0729 |
| 3 | 21.142 | BBA | 0.6132 | 6653.91260 | 169.77853 | 49.0799 |
| 4 | 22.893 | BBA | 0.3165 | 85.10750 | 3.67081 | 0.6278 |

```
Injection Date  : 12/22/2008 4:45:43 PM
Sample Name     : wf08121609                    I
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 12/22/2008 3:28:33 PM by MOVY
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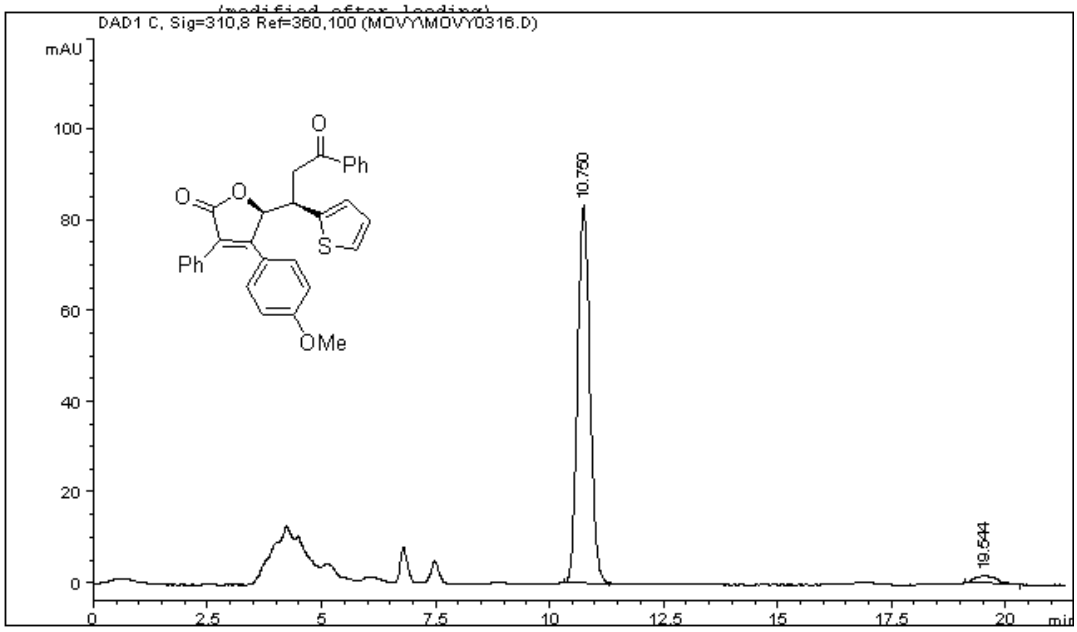


| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 10.750 | BB | 0.2881 | 1554.94800 | 83.19254 | 96.4303 |
| 2 | 19.544 | BB | 0.4094 | 57.56238 | 1.72449 | 3.5697 |

## 3q

```
Injection Date  : 1/8/2009 2:27:58 PM
Sample Name     : wf09010801
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/8/2009 2:22:19 PM by Linshi
                  (modified after loading)
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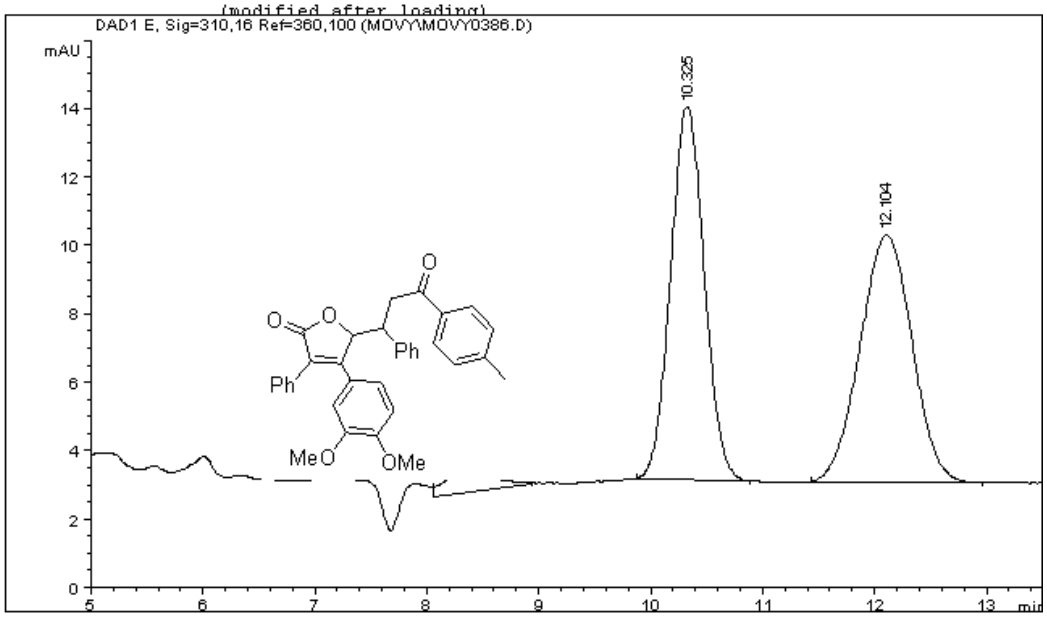


| | | | | | | |
|---|---|---|---|---|---|---|
| 5 | 10.325 | BB | 0.3251 | 229.22478 | 10.91076 | 22.1477 |
| 6 | 12.104 | BP | 0.4924 | 232.61876 | 7.21030 | 22.4756 |

```
Injection Date  : 1/8/2009 4:05:09 PM
Sample Name     : wf09010406
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 1/8/2009 4:01:34 PM by MOVY
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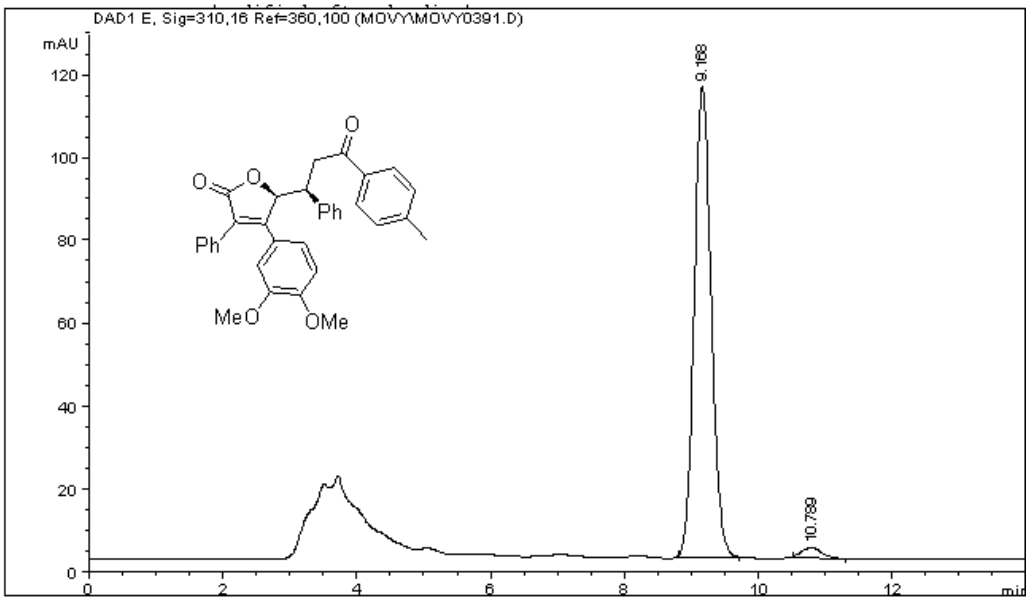


| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 9.168 | BBA | 0.2760 | 2002.28528 | 113.37322 | 97.4822 |
| 2 | 10.789 | BBA | 0.3136 | 51.71552 | 2.53897 | 2.5178 |

## 3r

```
Injection Date  : 9/22/2009 10:16:22 AM
Sample Name     : wf09091804-re
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 9/22/2009 10:12:33 AM by MOVY
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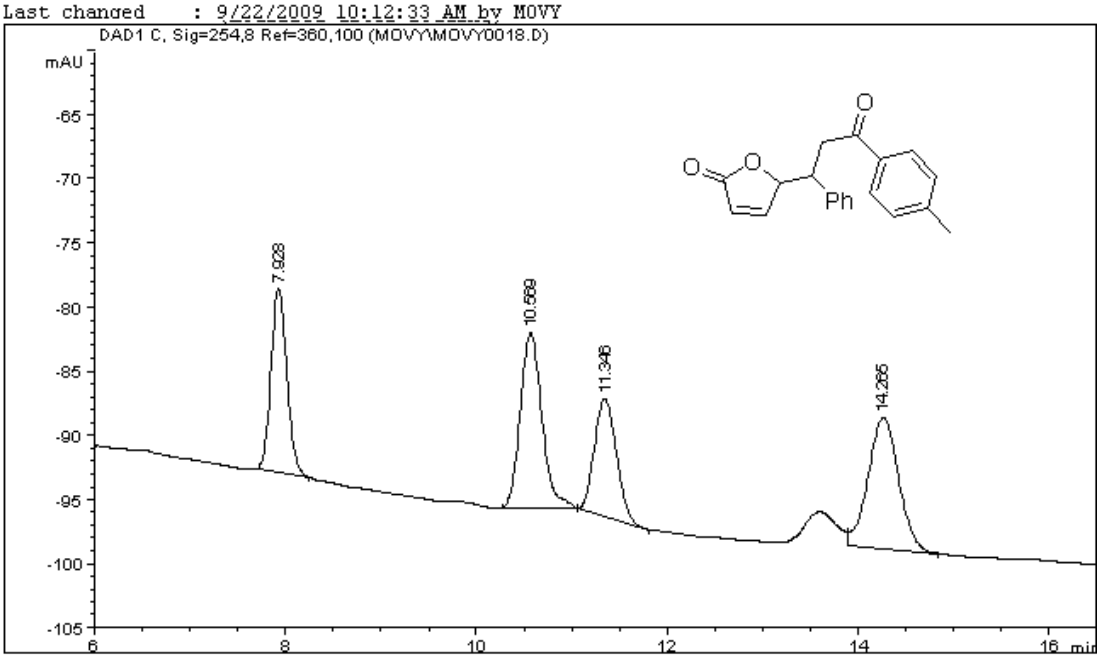
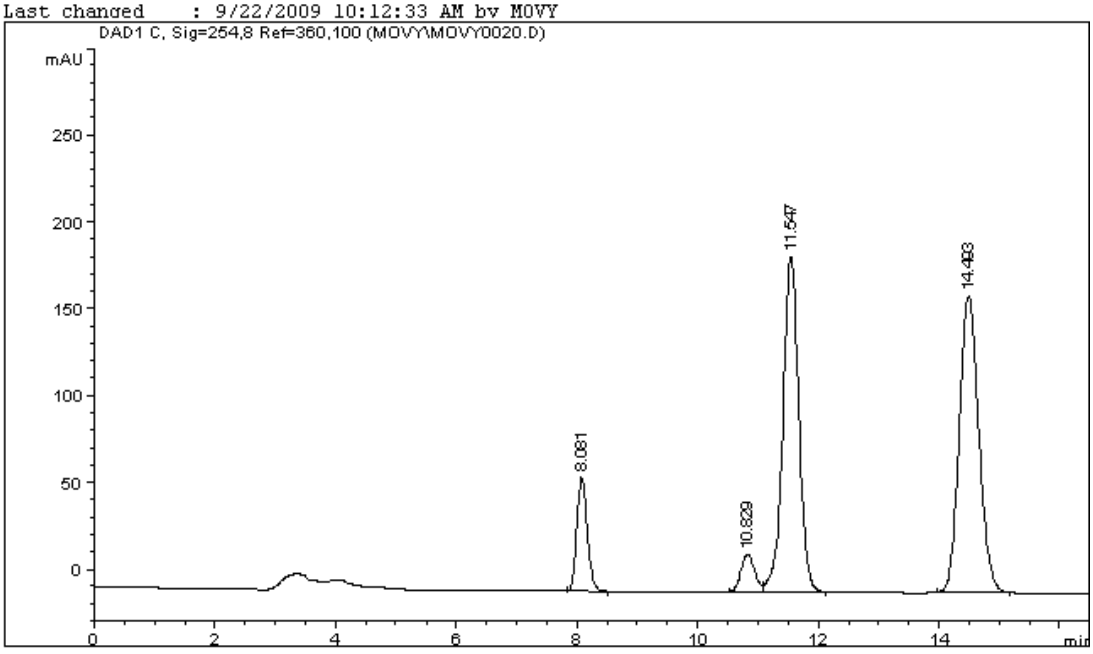


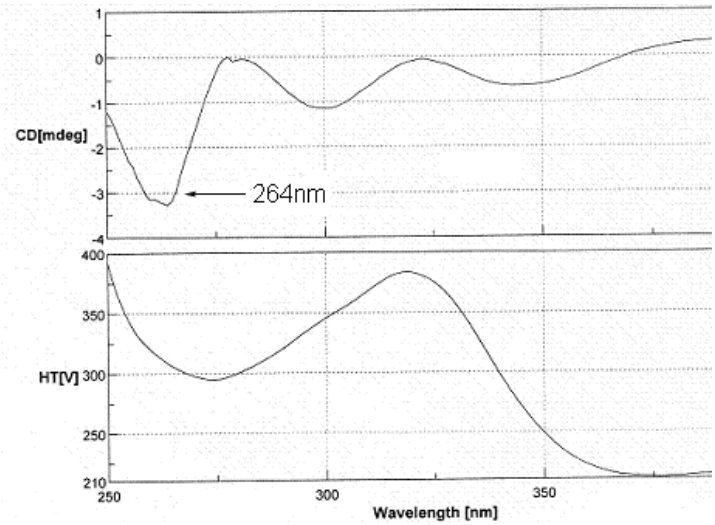

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 7.928 | BB | 0.1790 | 166.44727 | 14.38752 | 21.9400 |
| 2 | 10.569 | BB | 0.2449 | 215.88104 | 13.62358 | 28.4561 |
| 3 | 11.346 | BP | 0.2557 | 148.13232 | 9.20885 | 19.5259 |
| 4 | 14.265 | VB | 0.3453 | 228.18553 | 10.26635 | 30.0780 |

```
Injection Date  : 9/22/2009 10:55:02 AM
Sample Name     : wf09091608
Acq. Operator   : MOVY
Acq. Method     : C:\HPCHEM\1\METHODS\YG.M
Last changed    : 9/22/2009 10:12:33 AM by MOVY
```




| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 8.081 | BB | 0.1869 | 792.14832 | 65.60139 | 9.2822 |
| 2 | 10.829 | BV | 0.2499 | 352.02310 | 21.85523 | 4.1249 |
| 3 | 11.547 | VB | 0.2801 | 3549.58887 | 193.45633 | 41.5931 |
| 4 | 14.493 | BB | 0.3469 | 3840.31592 | 170.41284 | 44.9998 |

CD Spectra of **3p**




| | |
|---|---|
| Date | 2010-1-21 9:58 |
| File name | Memory#2 |
| Model | J-810 |
| Serial No. | B038360750 |
| Band width | 2 nm |
| Response | 1 sec |
| Sensitivity | Standard |
| Measurement range | 400 - 250 nm |
| Data pitch | 1nm |
| Scanning speed | 200 nm/min |
| Accumulation | 5 |
| Cell Length | 1 cm |
| Solvent | CDCl3 |
| Temperature | Room Temperature |
| | |
| Sample name | 3p |
| Operator | jfwang |
| Comment | |