



Supporting Information

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Supporting Information for

Chiral Brønsted Acid Catalyzed Asymmetric Baeyer-Villiger Reaction of 3-Substituted Cyclobutanones Using Aqueous H_2O_2

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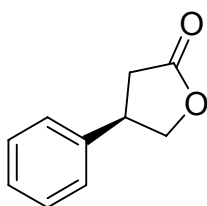
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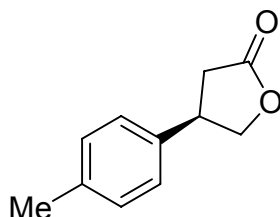
General Methods. Melting point was measured with XT-4 micro melting point apparatus and uncorrected. NMR spectra were recorded on a Varian Mercury 300 (^1H 300 MHz; ^{19}F 282 MHz) spectrometer in CDCl_3 . Chemical shifts are expressed in ppm with TMS as an internal standard ($\delta = 0$ ppm) for ^1H NMR. Coupling constants, J , are listed in hertz. Data for ^{19}F NMR were reported in terms of chemical shift (δ , ppm). HPLC analysis was carried out on a JASCO PU 2089 liquid chromatograph with a UV 2075 detector. Specific optical rotations and CD spectra were obtained on PE-341 and JASCO 810 spectrometers, respectively. Unless stated otherwise, all solvents were purified and dried according to standard methods prior to use.

General Procedure for Asymmetric Baeyer-Villiger Oxidation

To a 5-mL Schlenk tube was charged 7.6 mg of (*R*)-**1r** (10 mol %, 0.01 mmol), 0.1 mmol of 3-substituted cyclobutanone **2** and 1 mL of CHCl_3 . The mixture was stirred and cooled to -40°C , and then 1.5 equiv. of aqueous H_2O_2 (30%) was added at this temperature. The reaction mixture was stirred for 18-36 h at -40°C . After completion of the reaction, H_2O_2 was decomposed with aqueous Na_2SO_3 and the product **3** was isolated by column chromatography on silica gel with petroleum ether / ethyl acetate = 6/1 as the eluent. The enantiomeric excess was determined by chiral HPLC analysis.

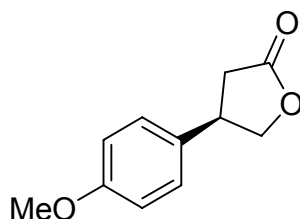


(-)-(R)-4-phenyldihydrofuran-2(3H)-one (3a)^[1]: white solid, 99% yield, m.p. $60-62^\circ\text{C}$ (lit^[1] $59-60^\circ\text{C}$); $[\alpha]_{\text{D}}^{20} = -46.7$ (c 0.48, CHCl_3), 88.0% ee; [Lit.^[1]: $[\alpha]_{\text{D}}^{20} = -67.2$ (c 2.0, CHCl_3) for *R*-isomer with 99% ee]. ^1H NMR (300 MHz, CDCl_3) δ = 7.22-7.41 (m, 5 H), 4.68 (dd, $J = 9.0, 8.6$ Hz, 1 H), 4.28 (dd, $J = 8.4, 8.4$ Hz, 1 H), 3.74-3.86 (m, 1 H), 2.94 (dd, $J = 17.4, 9.0$ Hz, 1 H), 2.68 (dd, $J = 17.4, 9.0$ Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AS-H column (hexane/2-PrOH = 95/5, flow rate = 1.0 mL/min, $\lambda = 220$ nm), $t_{\text{R}} = 40.4$ min (major); 46.9 min (minor).

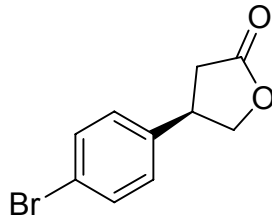


(-)-(R)-4-(4-tolyl)dihydrofuran-2(3H)-one (3b)^[2]: yellow solid; 99% yield, m.p. $66-68^\circ\text{C}$ (lit^[2] $63.5-65^\circ\text{C}$); $[\alpha]_{\text{D}}^{20} = -31.5$ (c 0.67, CHCl_3), 93% ee; [Lit.^[2]: $[\alpha]_{\text{D}}^{21} =$

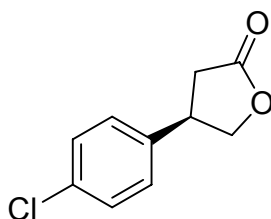
+50 ($c = 1.25$, CHCl_3) for *S*-isomer]. ^1H NMR (300 MHz, CDCl_3) $\delta = 7.10$ - 7.20 (m, 4 H), 4.65 (dd, $J = 8.4$, 8.4 Hz, 1 H), 4.24 (dd, $J = 8.5$, 8.4 Hz, 1 H), 3.72-3.79 (m, 1 H), 2.90 (dd, $J = 17.1$, 8.7 Hz, 1 H), 2.65 (dd, $J = 17.1$, 8.7 Hz, 1 H), 2.35 (s, 3 H) ppm; The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 214$ nm), $t_R = 17.9$ min (minor), 18.9 min (major).



(-)-(R)-4-(4-methoxyphenyl)dihydrofuran-2(3H)-one (3c)^[3]: yellow solid; 99% yield, m.p. 92-94°C; $[\alpha]_D^{20} = -28.3$ (c 0.90, CHCl_3) for *R*-isomer with 85% ee. ^1H NMR (300 MHz, CDCl_3) $\delta = 7.15$ (d, $J = 7.3$, 2.1 Hz, 2 H), 6.90 (dd, $J = 7.2$, 2.4 Hz, 2 H), 4.64 (dd, $J = 8.7$, 8.1 Hz, 1 H), 4.22 (dd, $J = 8.7$, 8.4 Hz, 1 H), 3.81 (s, 3 H), 3.71-3.83 (m, 1 H), 2.90 (dd, $J = 17.4$, 8.7 Hz, 1 H), 2.63 (dd, $J = 17.4$, 9.3 Hz, 1 H) ppm; The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 214$ nm), $t_R = 31.8$ min (minor), 33.5 min (major).

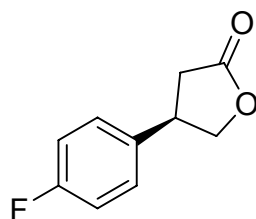


(-)-(R)-4-(4-bromophenyl)dihydrofuran-2(3H)-one (3d)^[3]: yellow solid; 99% yield, m.p. 84-86°C; $[\alpha]_D^{20} = -30.7$ (c 1.18, CHCl_3) for *R*-isomer with 83% ee. ^1H NMR (300 MHz, CDCl_3) δ 7.50 (d, $J = 8.7$ Hz, 1 H), 7.12 (d, $J = 8.4$ Hz, 1 H), 4.66 (dd, $J = 8.7$, 7.8 Hz, 1 H), 4.23 (dd, $J = 8.4$, 7.5 Hz, 1 H), 3.73-3.79 (m, 1 H), 2.94 (dd, $J = 17.7$, 8.7 Hz, 1 H), 2.63 (dd, $J = 17.4$, 8.4 Hz, 1 H); The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 230$ nm), $t_R = 31.9$ min (minor), 33.8 min (major).

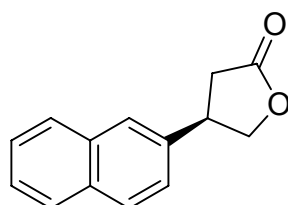


(-)-(R)-4-(4-chlorophenyl)dihydrofuran-2(3H)-one (3e)^[4]: yellow solid; 99%

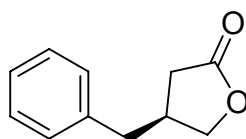
yield, m.p. 72-74°C (lit.^[4] 73-75°C); $[\alpha]_D^{20} = -47.2$ (c 0.90, CHCl₃) for *R*-isomer with 81.7% ee [Lit.^[4]: $[\alpha]_D^{20} = -48.5$ (c 1.38, CHCl₃) for *R*-isomer]. ¹H NMR (300 MHz, CDCl₃) δ = 7.35 (d, *J* = 8.4 Hz, 2 H), 7.17 (d, *J* = 7.8 Hz, 2 H), 4.66 (dd, *J* = 8.4, 8.4 Hz, 1 H), 4.24 (dd, *J* = 9.0, 8.2 Hz, 1 H), 3.74-3.81 (m, 1 H), 2.94 (dd, *J* = 17.7, 8.7 Hz, 1 H), 2.63 (dd, *J* = 17.7, 8.7 Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, λ = 230 nm), *t*_R = 28.9 min (minor), 30.5 min (major).



(-)-(R)-4-(4-fluorophenyl)dihydrofuran-2(3H)-one (3f)^[3]: yellow solid; 99% yield, m.p. 58-60°C; $[\alpha]_D^{20} = -40.2$ (c 0.85, CHCl₃) for *R*-isomer with 84% ee. ¹H NMR (300 MHz, CDCl₃) δ = 7.19-7.24 (m, 2 H), 7.03-7.10 (m, 2 H), 4.66 (dd, *J* = 9.3, 8.1 Hz, 1 H), 4.24 (dd, *J* = 9.0, 7.8 Hz, 1 H), 3.75-3.82 (m, 1 H), 2.94 (dd, *J* = 17.1, 8.4 Hz, 1 H), 2.64 (dd, *J* = 17.1, 8.7 Hz, 1 H) ppm; ¹⁹F NMR (282 MHz, CDCl₃) δ = -114.88 ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, λ = 214 nm), *t*_R = 27.2 min (minor), 28.4 min (major).

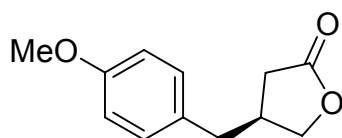


(-)-(R)-4-(naphthalen-2-yl)dihydrofuran-2(3H)-one (3g)^[5]: white powder, 91% yield, m.p. 116-117°C; $[\alpha]_D^{20} = -57.9$ (c 0.90, CHCl₃) for *R*-isomer with 86% ee. ¹H NMR (300 MHz, CDCl₃) δ = 7.80-7.89 (m, 3 H), 7.68 (s, 1 H), 7.48-7.53 (m, 2 H), 7.33-7.37 (m, 1 H), 4.75 (dd, *J* = 9.0, 8.1 Hz, 1 H), 4.39 (dd, *J* = 9.0, 8.1 Hz, 1 H), 3.94-4.00 (m, 1 H), 3.02 (dd, *J* = 17.4, 8.7 Hz, 1 H), 2.79 (dd, *J* = 17.4, 9.0 Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, λ = 254 nm), *t*_R = 29.1 min (minor), 31.9 min (major).

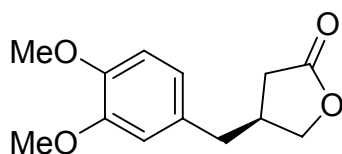


(-)-(S)-4-benzylidihydrofuran-2(3H)-one (3h)^[6]: yellow oil; 99% yield, $[\alpha]_D^{20} =$

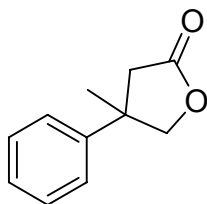
-3.8 (c 0.80, CHCl₃) for S-isomer with 58% ee [Lit.^[6]: $[\alpha]_D^{30} = -8.64$ (c 0.71, CHCl₃) for S-isomer]. ¹H NMR (300 MHz, CDCl₃) $\delta = 7.14-7.35$ (m, 5 H), 4.34 (dd, $J = 8.7, 6.6$ Hz, 1 H), 4.04 (dd, $J = 8.7, 6.6$ Hz, 1 H), 2.76-2.89 (m, 3 H), 2.61 (dd, $J = 17.5, 7.5$ Hz, 1 H), 2.29 (dd, $J = 17.5, 6.9$ Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 214$ nm), $t_R = 23.2$ min (minor), 25.0 min (major).



(-)-(S)-4-(4-methoxybenzyl)dihydrofuran-2(3H)-one (3i)^[7]: white solid; 99% yield, m.p. 58-60°C (lit.^[7] 56.5-59°C); $[\alpha]_D^{25} = -4.2$ (c 1.0, CHCl₃) for S-isomer with 57% ee; [Lit.^[5]: $[\alpha]_D^{20} = -5.8$ (c 2.18, CHCl₃) for S-isomer]. ¹H NMR (300 MHz, CDCl₃) $\delta = 7.07$ (d, $J = 8.7$ Hz, 2 H), 6.84 (d, $J = 8.1$ Hz, 2 H), 4.33 (dd, $J = 9.3, 6.9$ Hz, 1 H), 4.03 (dd, $J = 9.3, 6.0$ Hz, 1 H), 3.80 (s, 3 H), 2.70-2.85 (m, 3 H), 2.59 (dd, $J = 17.4, 8.1$ Hz, 1 H), 2.27 (dd, $J = 17.1, 7.2$ Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 214$ nm), $t_R = 32.7$ min (minor), 34.7 min (major).



(-)-(S)-4-(3,4-dimethoxybenzyl)dihydrofuran-2(3H)-one (3j)^[8]: colorless oil, 99% yield, $[\alpha]_D^{20} = -3.9$ (c 1.1, CHCl₃) for S-isomer with 55% ee; [Lit.^[8]: $[\alpha]_D^{23} = -7.54$ (c 1.9, CHCl₃) for S-isomer]. ¹H NMR (300 MHz, CDCl₃) $\delta = 6.82$ (d, $J = 8.1$ Hz, 1 H), 6.66-6.72 (m, 2 H), 4.33 (dd, $J = 9.3, 6.6$ Hz, 1 H), 4.05 (dd, $J = 9.3, 6.3$ Hz, 1 H), 3.88 (s, 3 H), 3.87 (s, 3 H), 2.70-2.87 (m, 3 H), 2.61 (dd, $J = 17.4, 8.1$ Hz, 1 H), 2.33 (dd, $J = 18.0, 9.3$ Hz, 1 H) ppm. The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 95/5, flow rate = 0.7 mL/min, $\lambda = 214$ nm), $t_R = 57.5$ min (minor), 59.0 min (major).



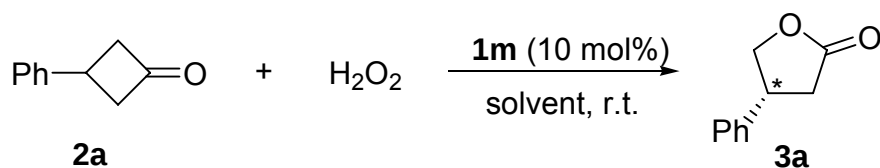
(+)-4-methyl-4-phenyldihydrofuran-2(3H)-one (3k)^[9]: yellow oil, 99% yield, $[\alpha]_D^{20} = +7.3$ (c 0.73, CHCl₃) for the isomer with 61% ee. ¹H NMR (300 MHz,

CDCl₃) δ 7.17-7.41 (m, 5 H), 4.42 (s, 2 H), 2.92 (d, J = 16.8 Hz, 1 H), 2.67 (d, J = 16.8 Hz, 1 H), 1.52 (s, 3 H). The enantiomeric excess was determined by chiral HPLC analysis on a Chiralpak AD-H column (hexane/2-PrOH = 90/10, flow rate = 0.7 mL/min, λ = 214 nm), t_R = 13.4 min (minor), 16.8 min (major).

References:

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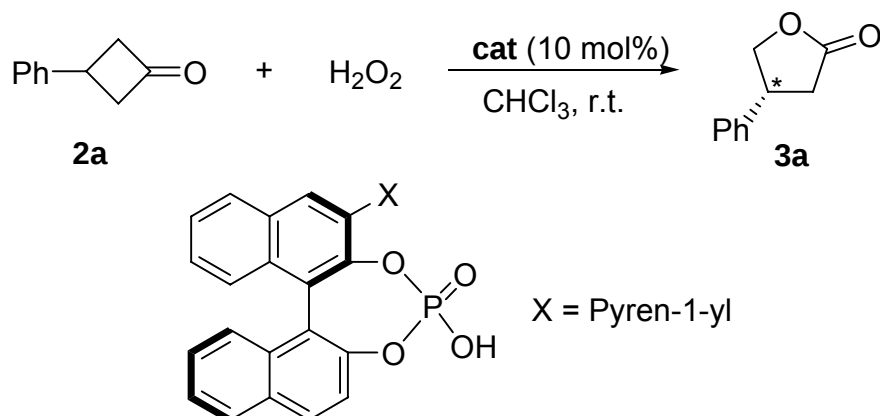
Table S1. Solvent effect on the BV oxidation of **2a** using 1.5 equiv. of aqueous H₂O₂ (30%) in the presence of **1m** as the catalyst.^[a]



Entry	Sovent	Yield [%] ^[b]	<i>Ee</i> [%] ^[c]
1	CH ₂ Cl ₂	68	45
2	CHCl ₃	72	54
3	CCl ₄	69	28
4	ClCH ₂ CH ₂ Cl	80	44
5	Toluene	93	33
6	Hexane	48	2
7	Et ₂ O	21	-6
8	MeOH	no reaction	n.d.
9	EtOH	no reaction	n.d.
10	EtOAc	no reaction	n.d.
11	THF	no reaction	n.d.
12	CH ₃ CN	no reaction	n.d.

[a] The reaction was carried out at room temperature with [**2a**] = 0.1 M to give **3a**. [b] The yield of isolated product. [c] The enantiomeric excess of **3a** was determined by HPLC analysis on a chiral column (Chiralpak AS-H).

Table S2. Relationship between the ee of the product and the ee of the catalyst.^[a]



ee of cat. [%]	ee of 3a [%] ^[b]
0	0
20	8
40	18
80	41
100	51

[a] The reaction was carried out at room temperature with [**2a**] = 0.1 M in CHCl₃. [b] The enantiomeric excess of **3a** was determined by HPLC analysis on a chiral column (Chiralpak AS-H).

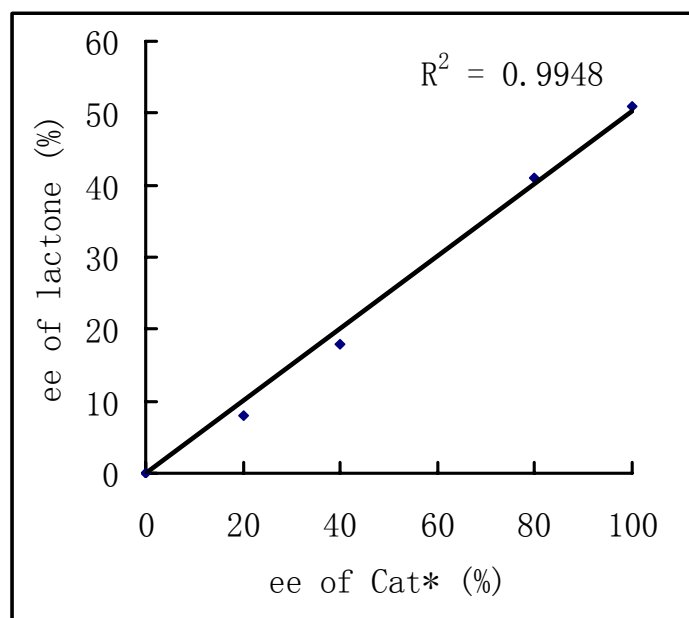
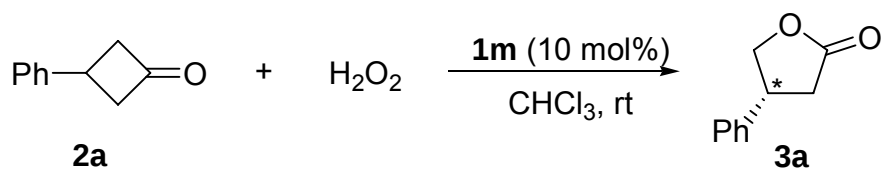


Figure S1. NLE effect in the catalysis of BV oxidation of **3a** using a chiral phosphoric acid as the catalyst in CHCl₃ at room temperature.

Table S3. Concentration effect on the enantioselectivity of BV oxidation of **2a** in the presence of **1m**



Entry	Time(h)	[1m] (M)	Yield(%) ^[a]	E.e.(%) ^[b]
1	24	0.02	84	51
2	24	0.01	90	53
3	24	0.0067	83	55
4	24	0.005	79	55
5	24	0.004	83	55
6	24	0.0033	83	55
7	24	0.0028	83	55

[a] The yield of isolated product. [b] The enantiomeric excess of **3a** was determined by HPLC analysis on a chiral column (Chiralpak AS-H).

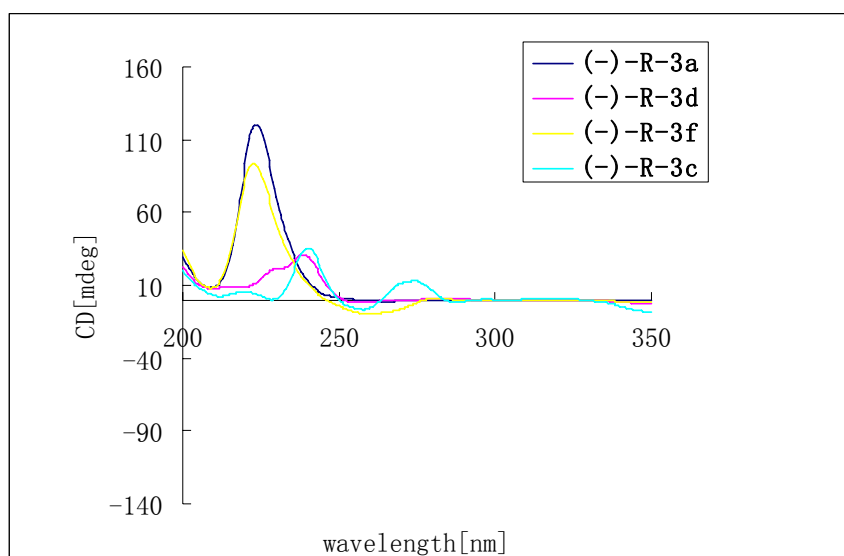


Figure S2. CD spectra for **3a**, **3c**, **3d** and **3f** in methanol, (*R*)-**3a** is an authentic sample.

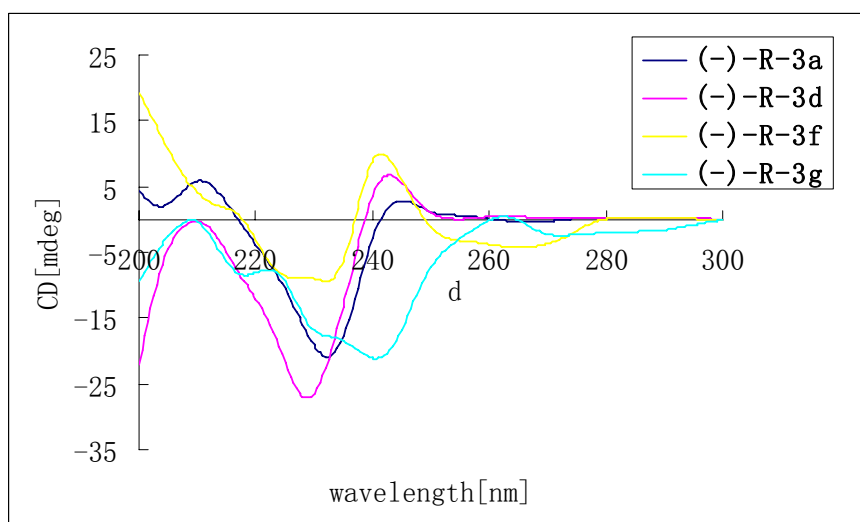
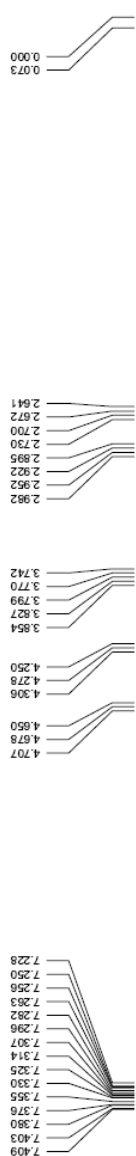
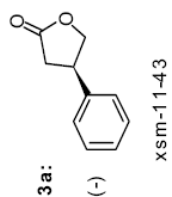
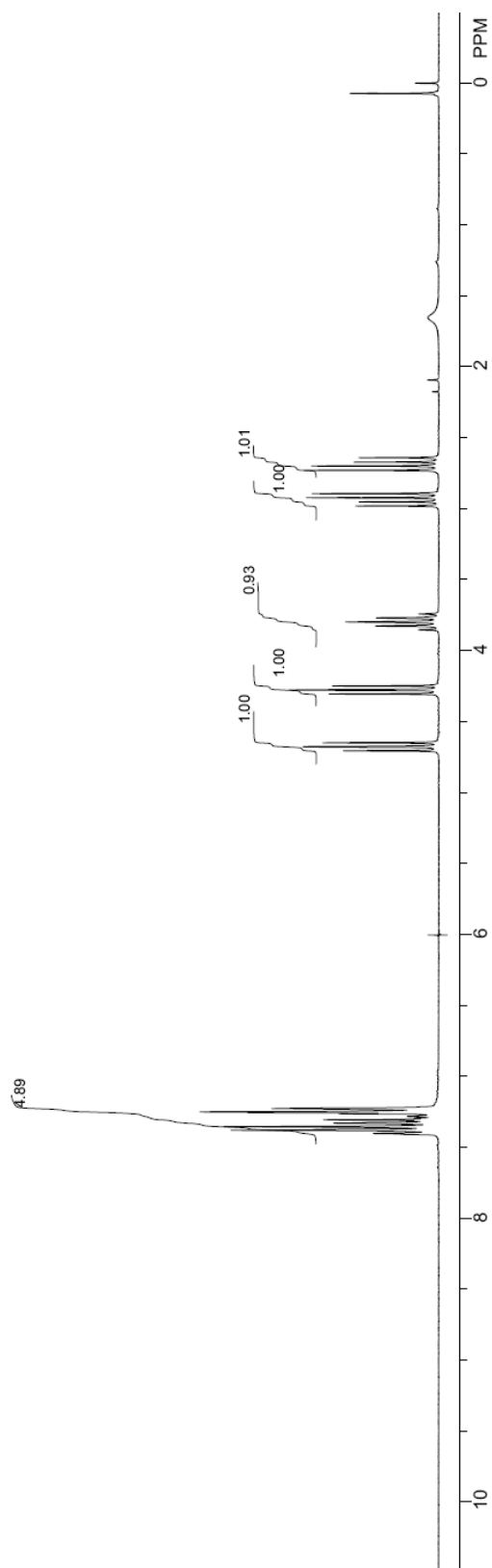
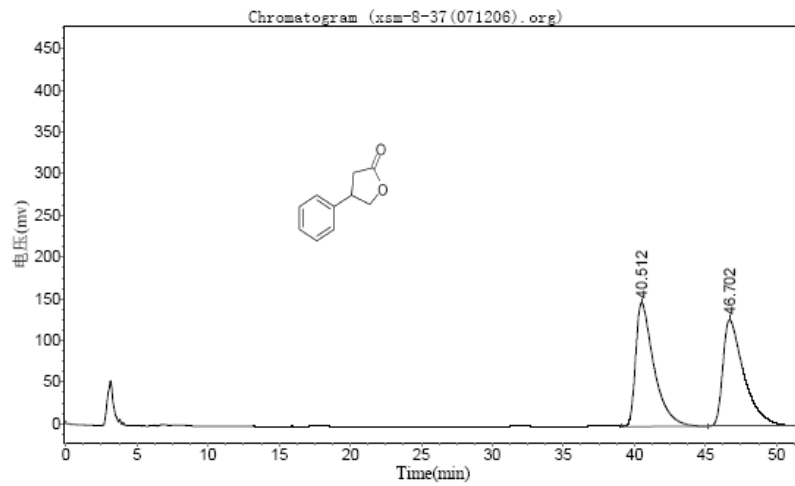


Figure S3. CD spectra of **3a**, **3d**, **3f** and **3g** in chloroform, (*R*)-**3a** is an authentic sample.

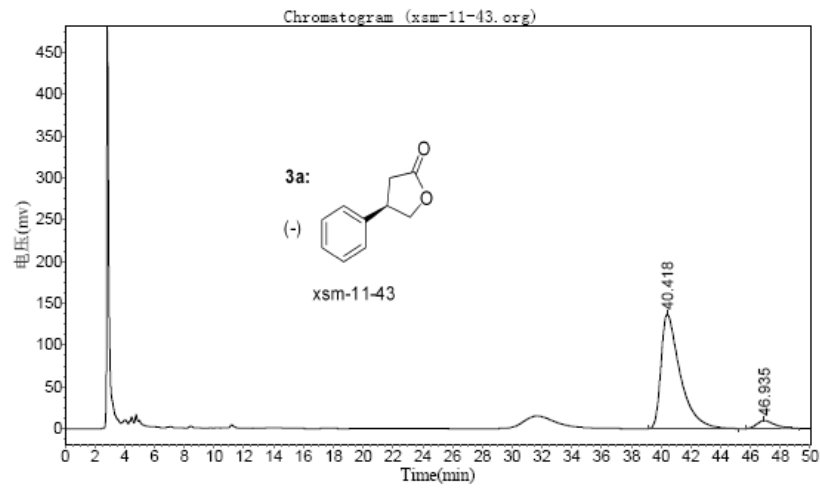
From Figure S2 and S3, it can be concluded that **3c**, **3d**, **3f** and **3g** should have the same absolute configuration as that of **3a**.





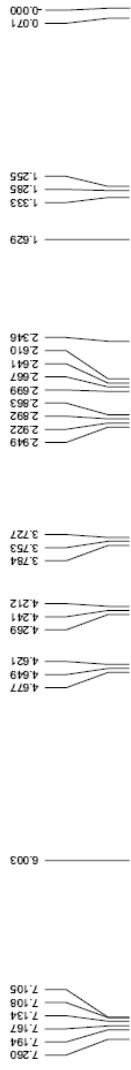
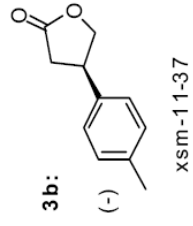
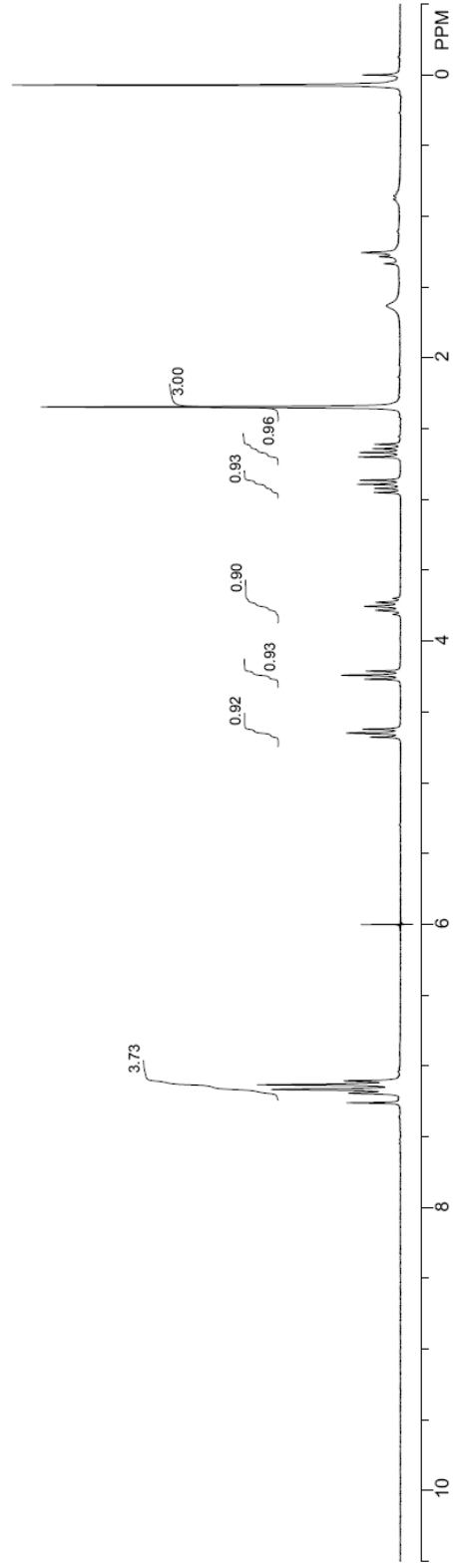
Results

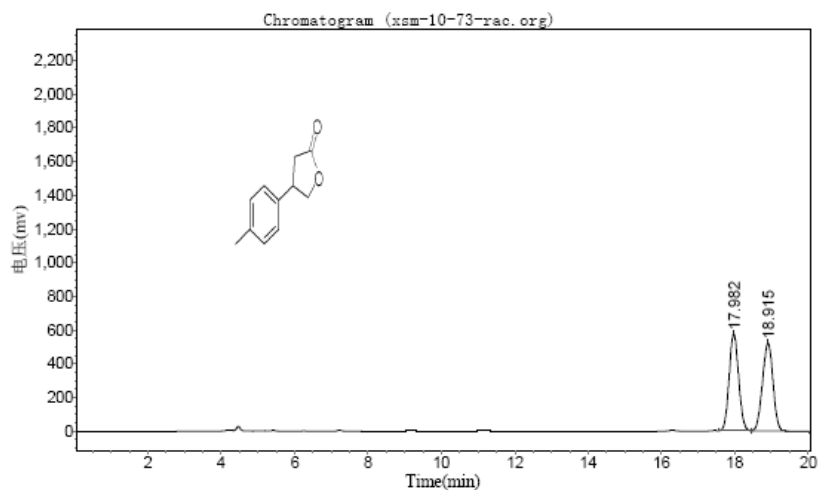
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		40.512	148461.172	13324734.000	50.0986
2		46.702	127987.039	13272300.000	49.9014
Total			276448.211	26597034.000	100.0000



Results

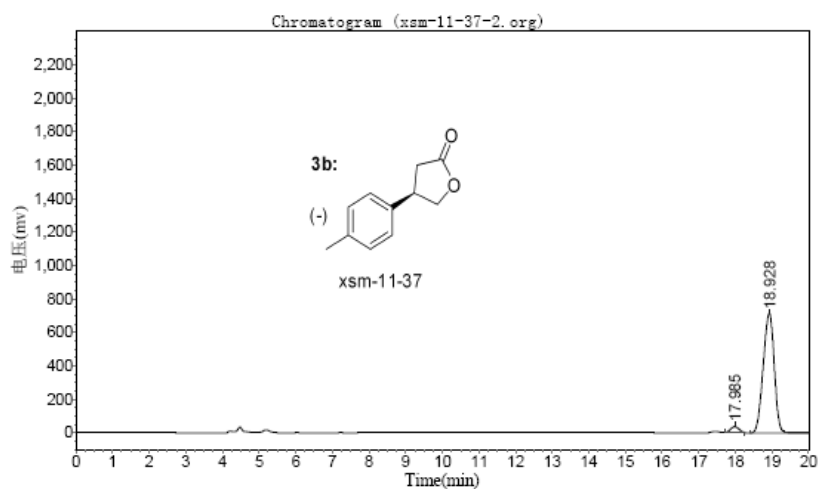
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		40.418	136511.922	12186496.000	94.0317
2		46.935	9336.074	773484.875	5.9683
Total			145847.996	12959980.875	100.0000





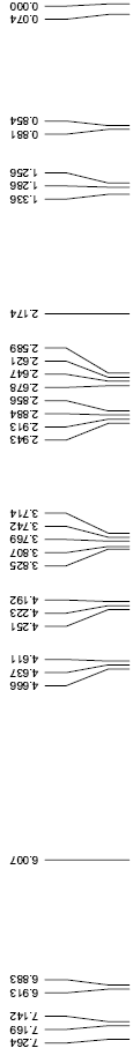
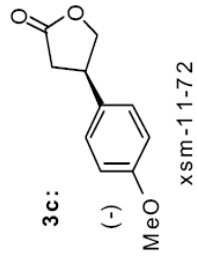
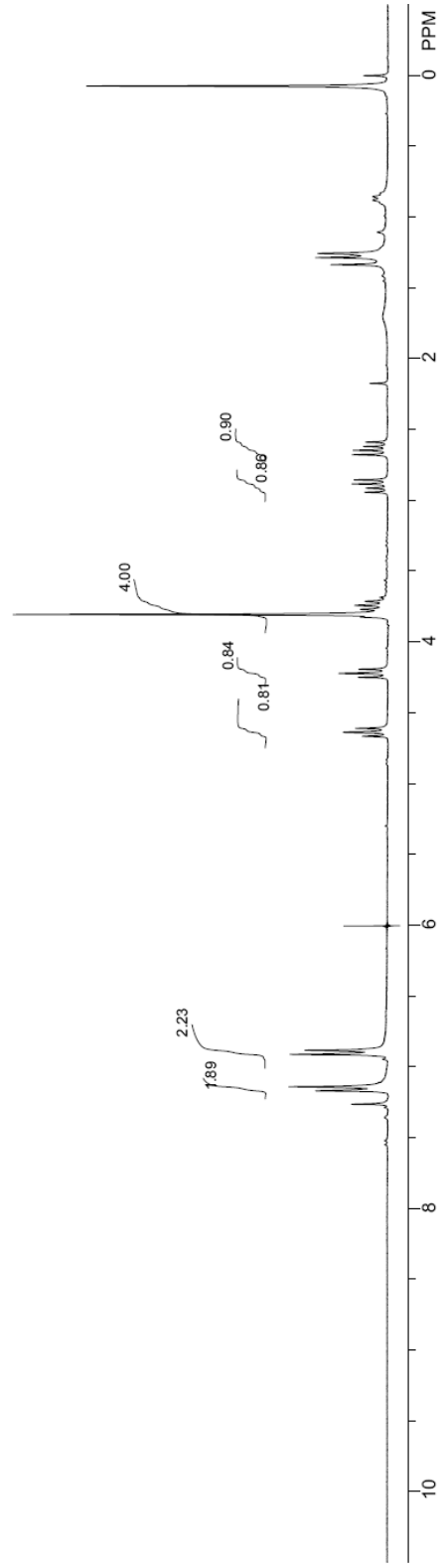
Results

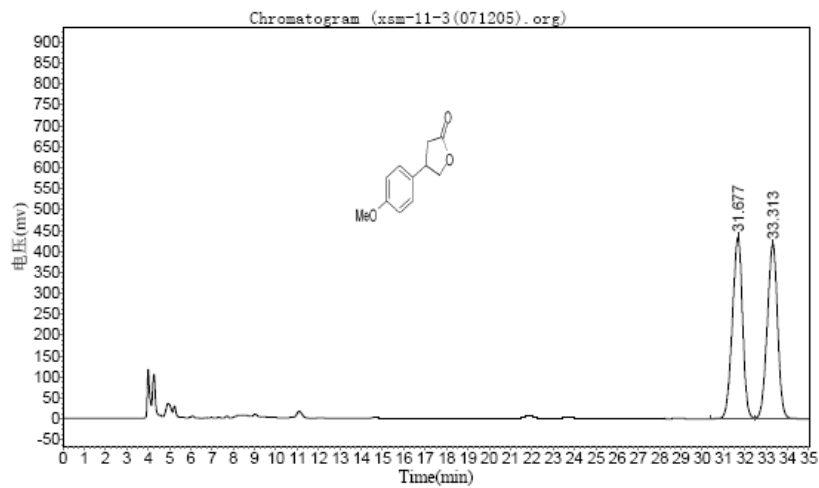
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.982	577510.188	10543849.000	49.9838
2		18.915	522548.313	10550693.000	50.0162
Total			1100058.500	21094542.000	100.0000



Results

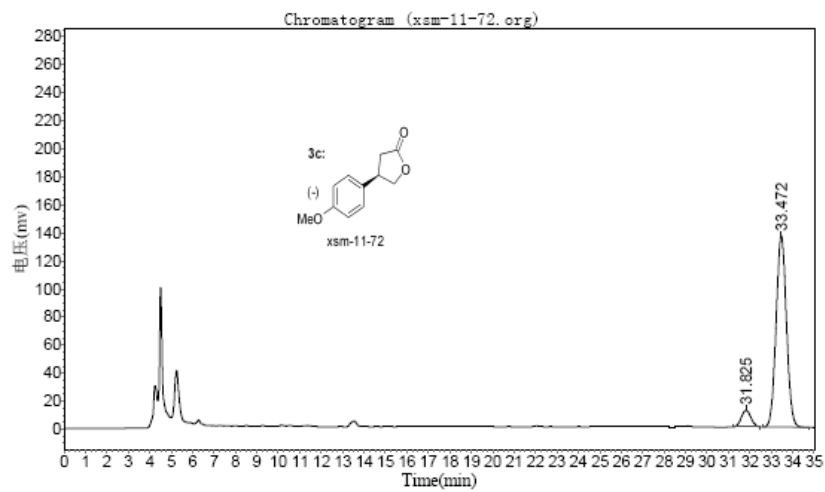
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.985	34570.641	543436.813	3.4990
2		18.928	713920.688	14987666.000	96.5010
Total			748491.328	15531102.813	100.0000





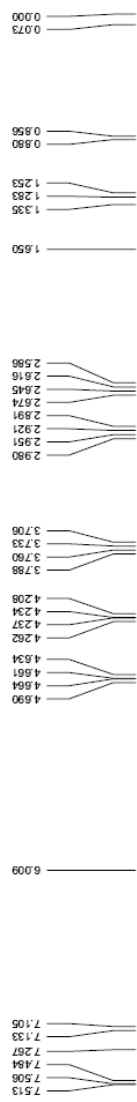
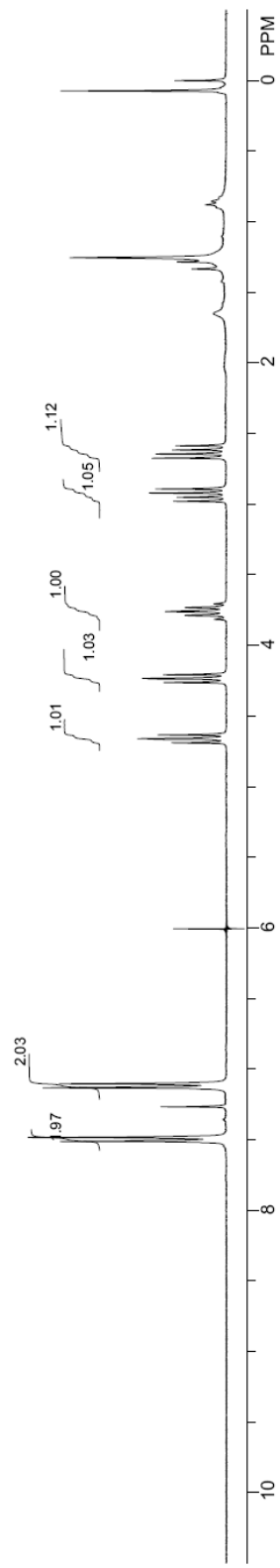
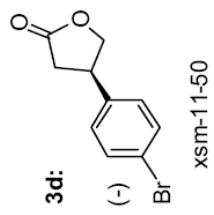
Results

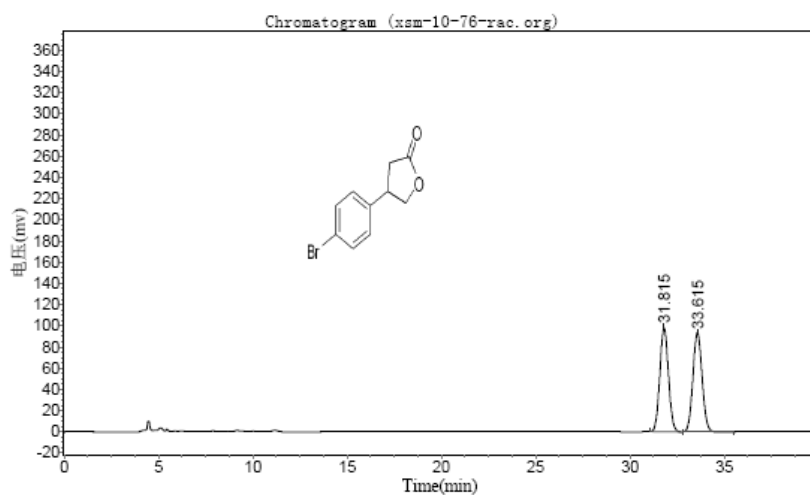
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		31.677	433089.281	14371134.000	50.4848
2		33.313	416969.563	14095121.000	49.5152
Total			850058.844	28466255.000	100.0000



Results

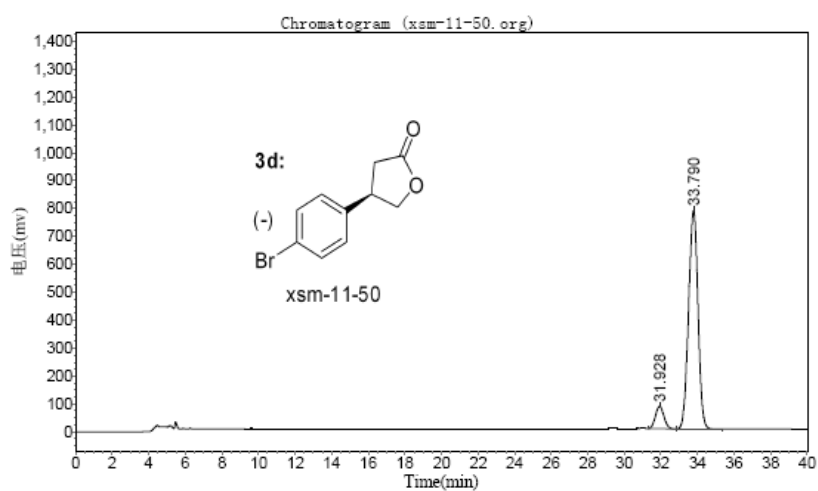
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		31.825	11903.113	368892.094	7.4513
2		33.472	136356.969	4581846.000	92.5487
Total			148260.082	4950738.094	100.0000





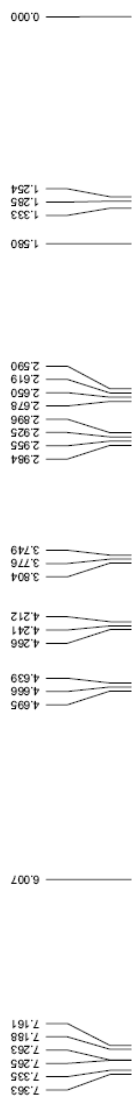
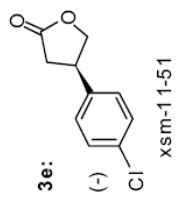
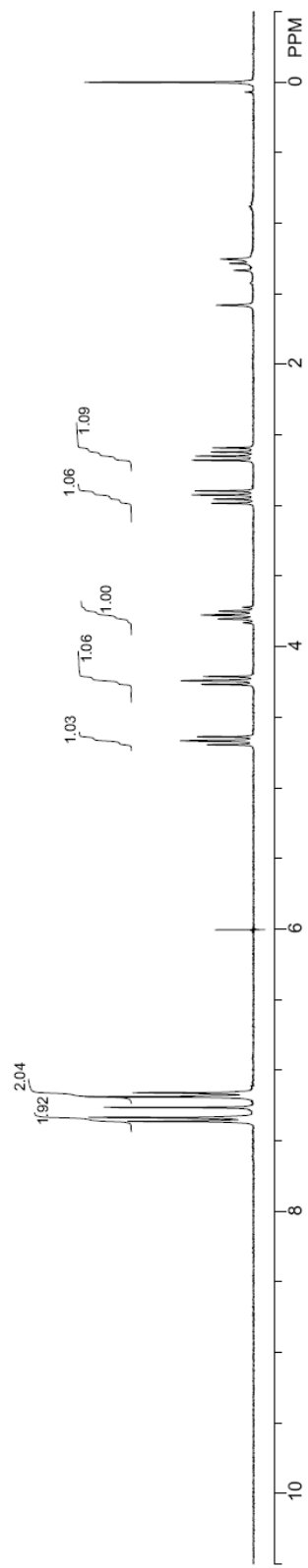
Results

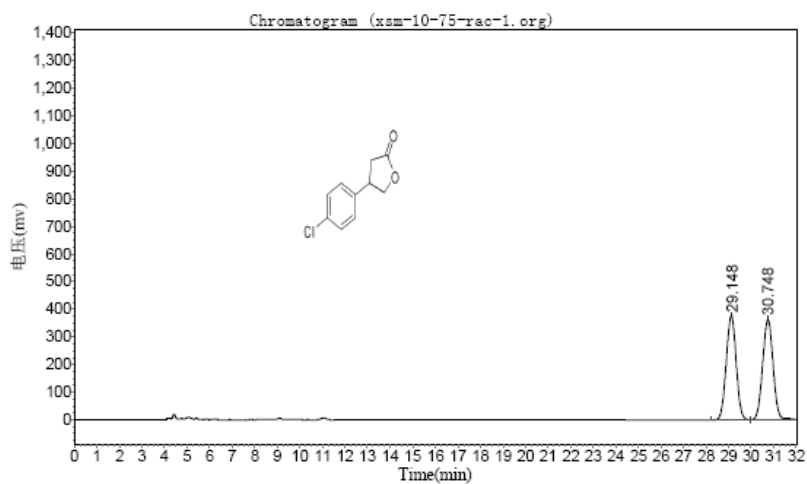
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		31.815	97797.234	3170604.000	49.7894
2		33.615	93569.594	3197429.250	50.2106
Total			191366.828	6368033.250	100.0000



Results

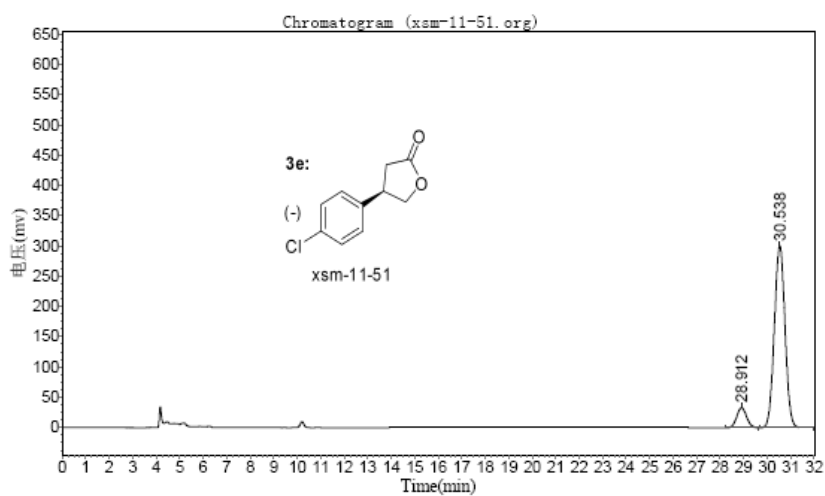
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		31.928	80181.266	2594646.000	8.5116
2		33.790	782434.813	27888898.000	91.4884
Total			862616.078	30483544.000	100.0000





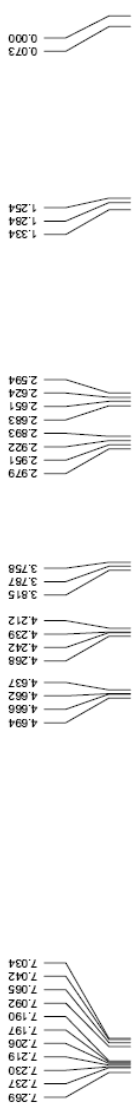
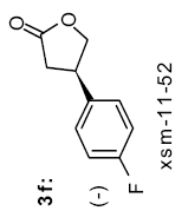
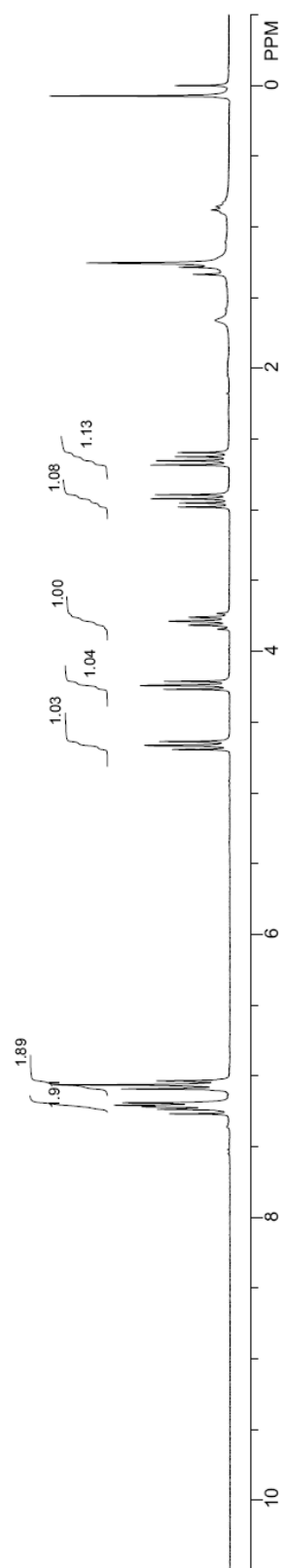
Results

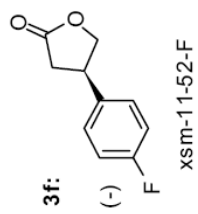
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		29.148	375561.094	11562442.000	49.5773
2		30.748	361990.281	11759593.000	50.4227
Total			737551.375	23322035.000	100.0000



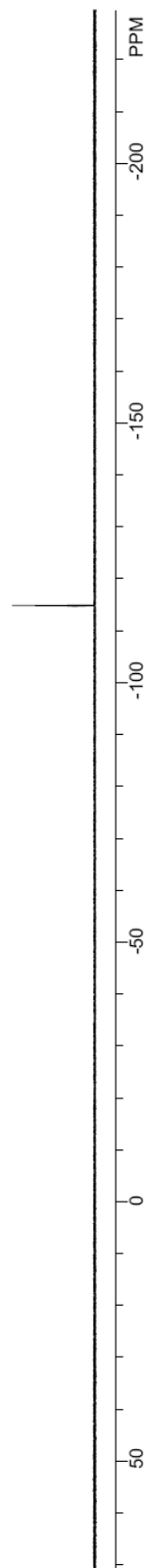
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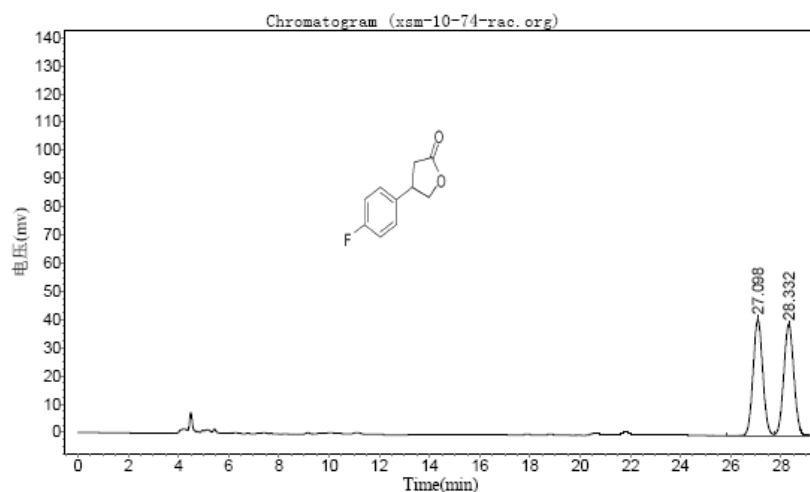
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		28.912	32965.449	956492.313	9.1461
2		30.538	300592.750	9501417.000	90.8539
Total			333558.199	10457909.313	100.0000





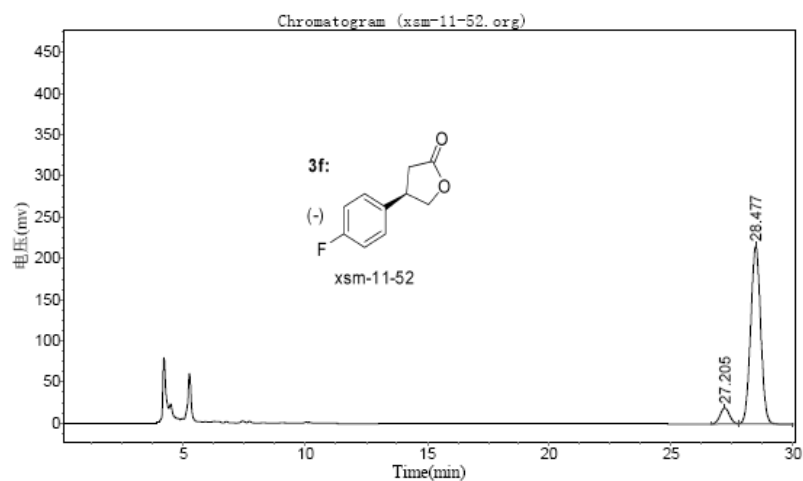
114.864





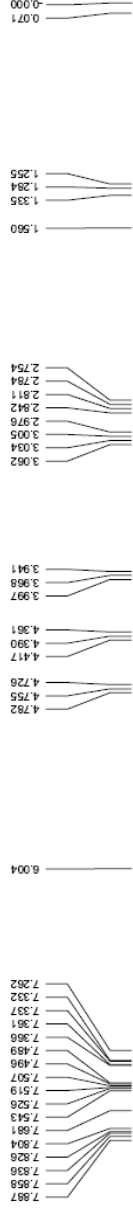
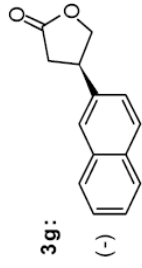
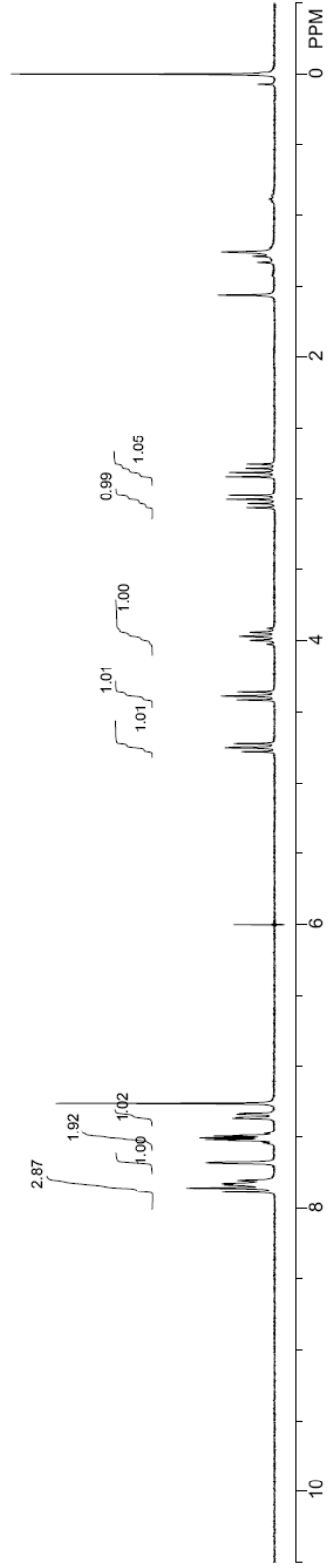
Results

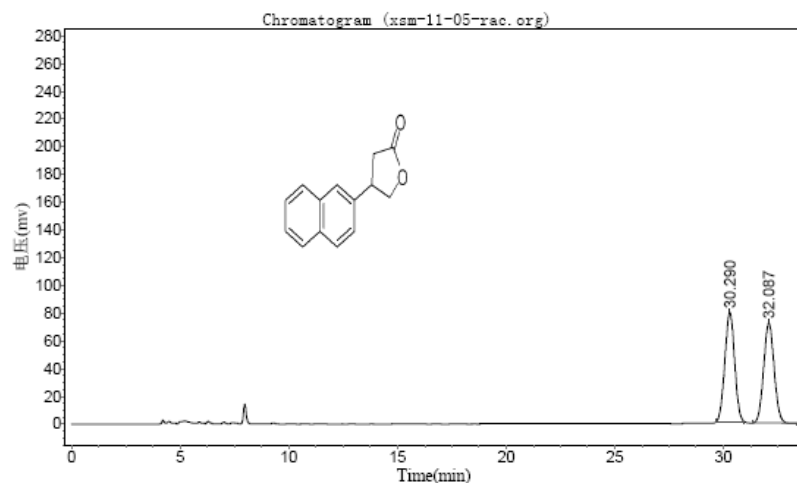
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		27.098	41054.000	1107401.250	50.0175
2		28.332	39423.000	1106625.750	49.9825
Total			80477.000	2214027.000	100.0000



Results

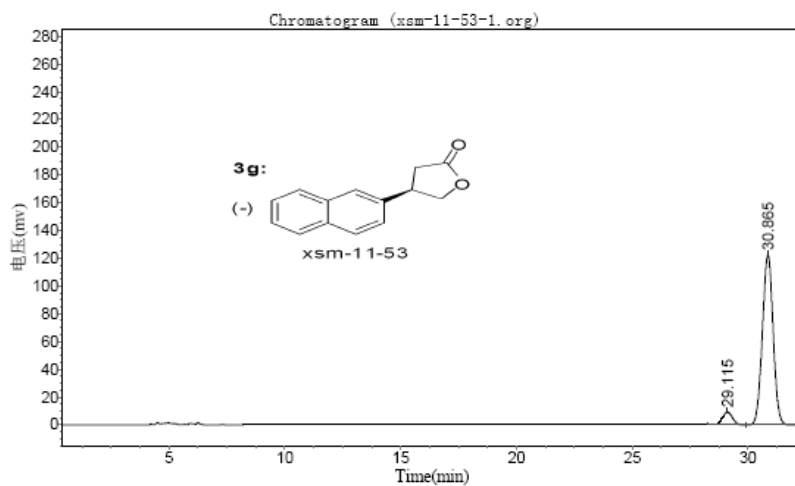
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		27.205	19225.258	510590.094	7.6431
2		28.477	215268.156	6169790.500	92.3569
Total			234493.414	6680380.594	100.0000





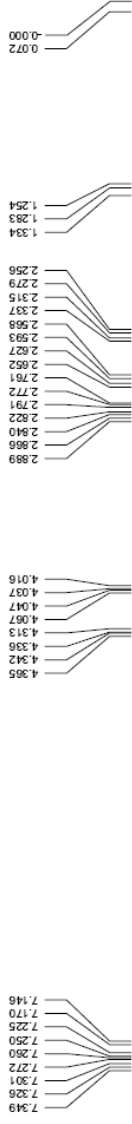
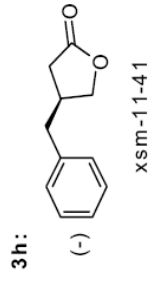
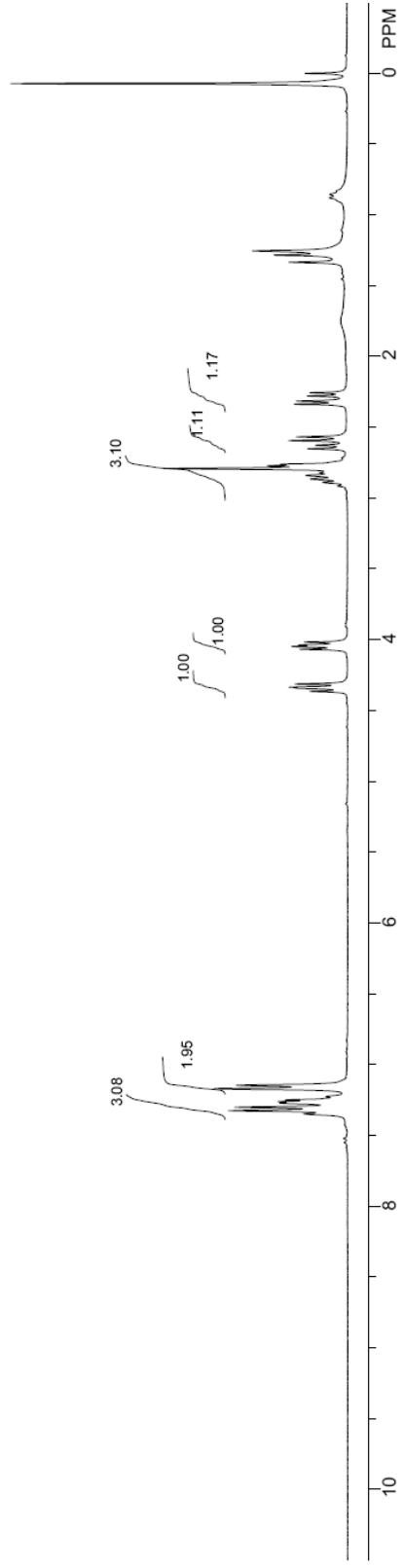
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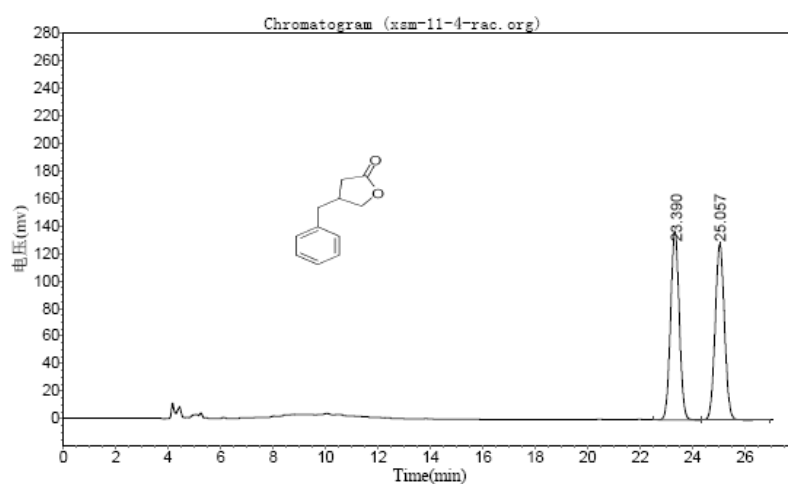
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		30.290	78845.227	2470052.000	50.5412
2		32.087	71845.039	2417152.750	49.4588
Total			150690.266	4887204.750	100.0000



Results

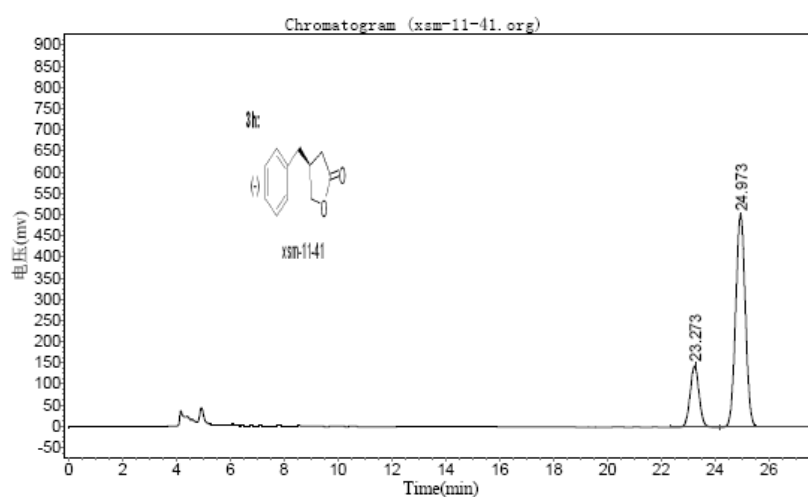
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		29.115	9389.758	283649.000	6.6930
2		30.865	122228.852	3954341.500	93.3070
Total			131618.609	4237990.500	100.0000





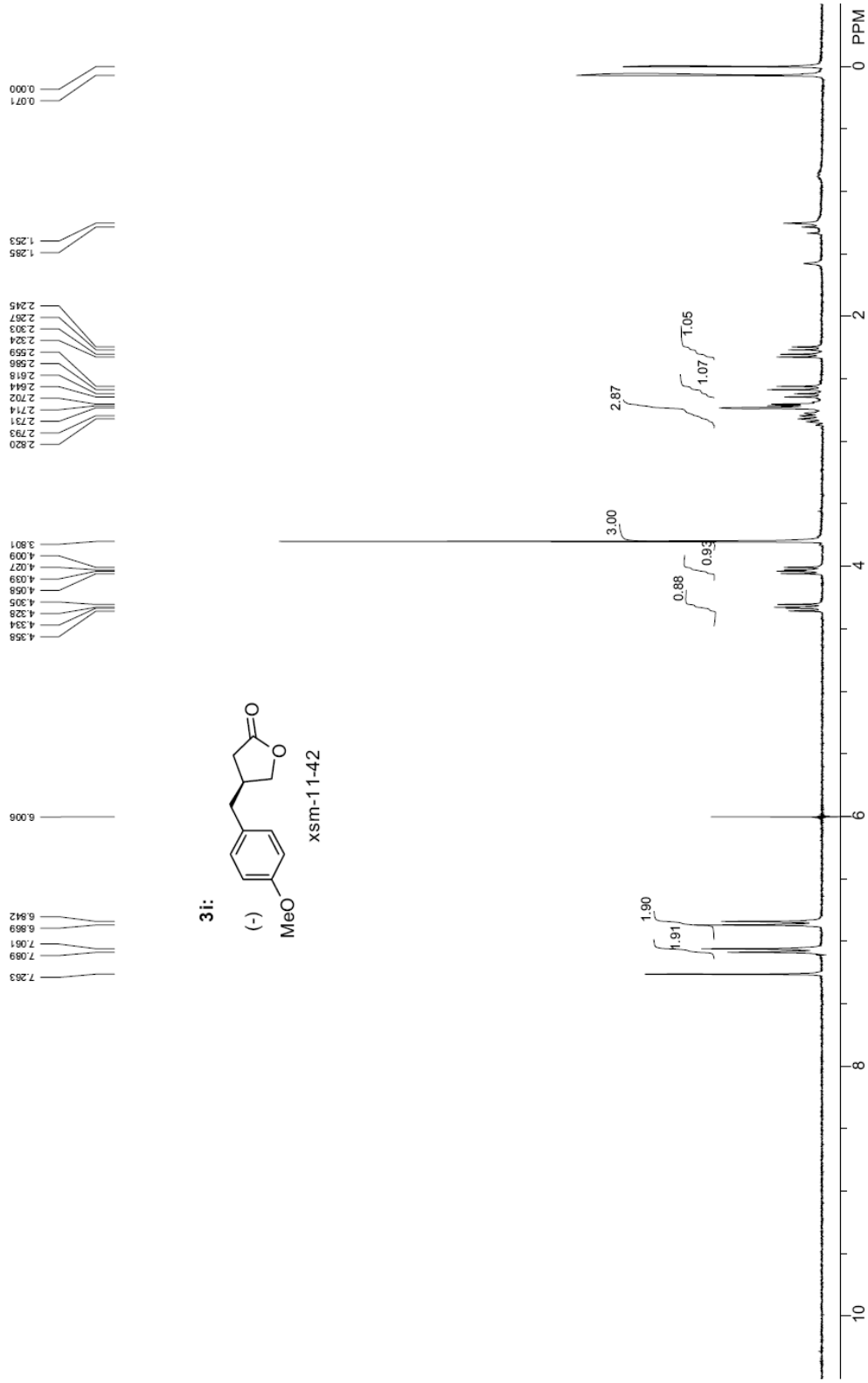
Results

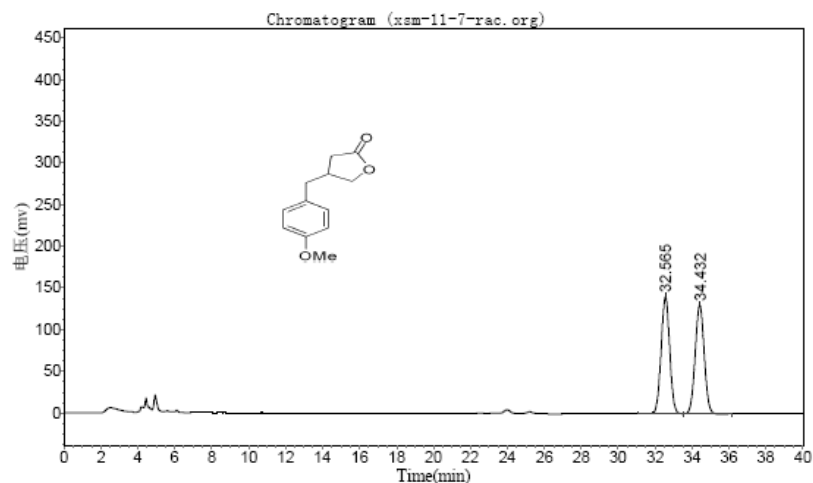
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		23.390	133838.203	3170580.250	50.0038
2		25.057	126396.320	3170098.000	49.9962
Total			260234.523	6340678.250	100.0000



Results

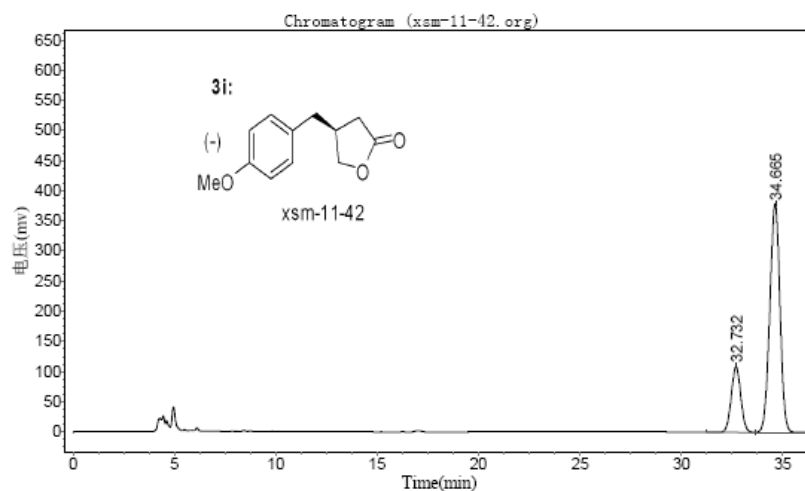
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		23.273	143389.531	3325873.000	20.9169
2		24.973	501076.125	12574528.000	79.0831
Total			644465.656	15900401.000	100.0000





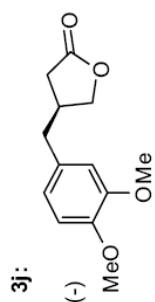
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		32.565	139715.344	4566143.000	50.3963
2		34.432	130618.414	4494321.000	49.6037
Total			270333.758	9060464.000	100.0000

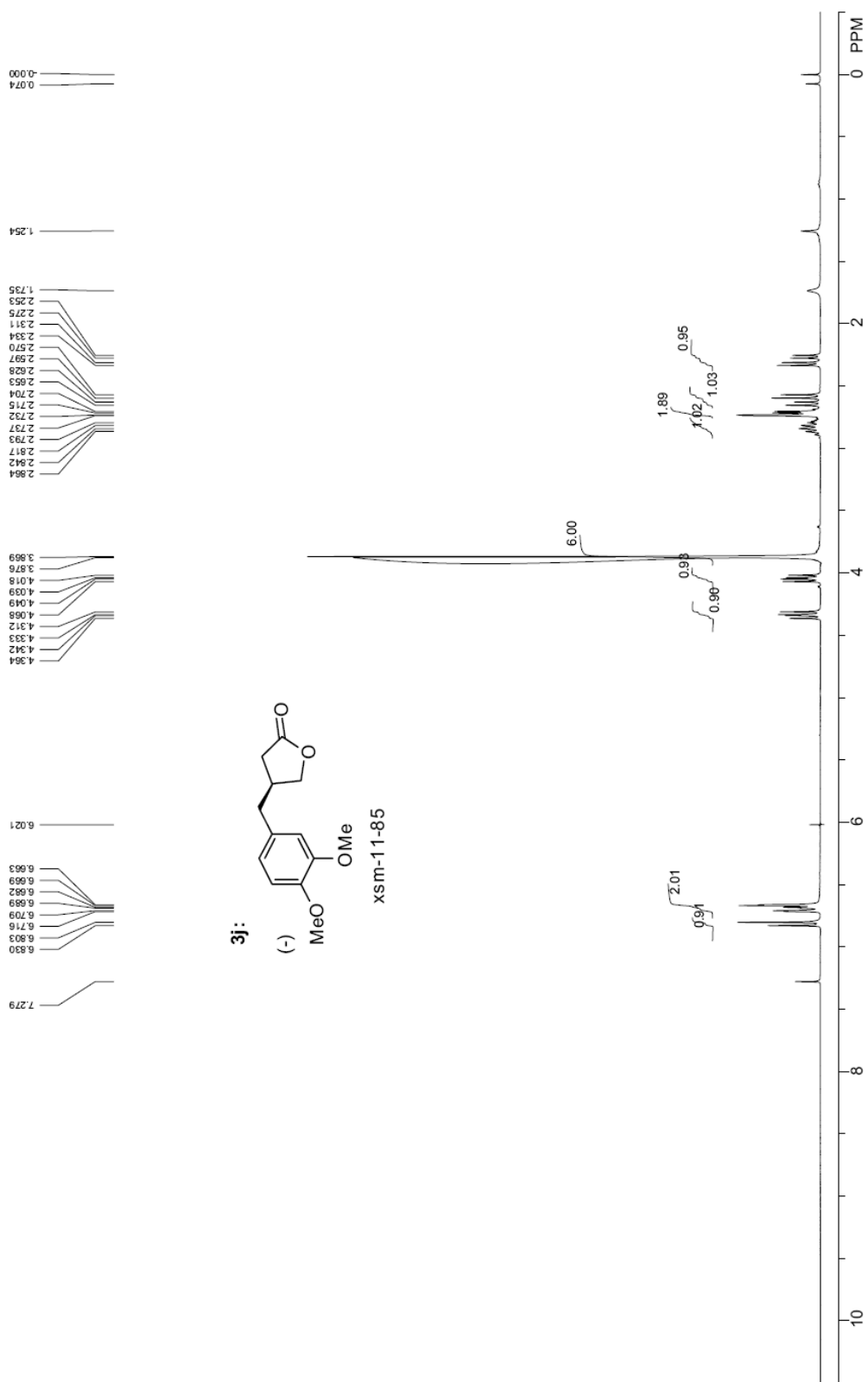


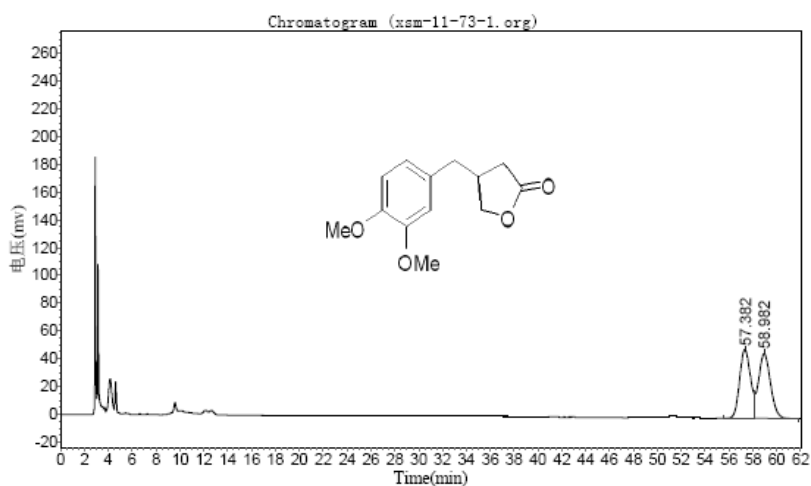
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		32.732	108294.406	3581633.500	21.2789
2		34.665	378413.344	13250196.000	78.7211
Total			486707.750	16831829.500	100.0000



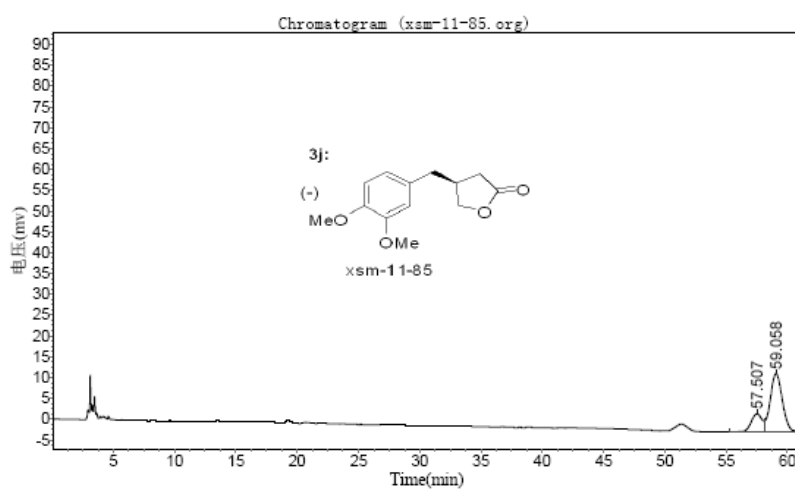
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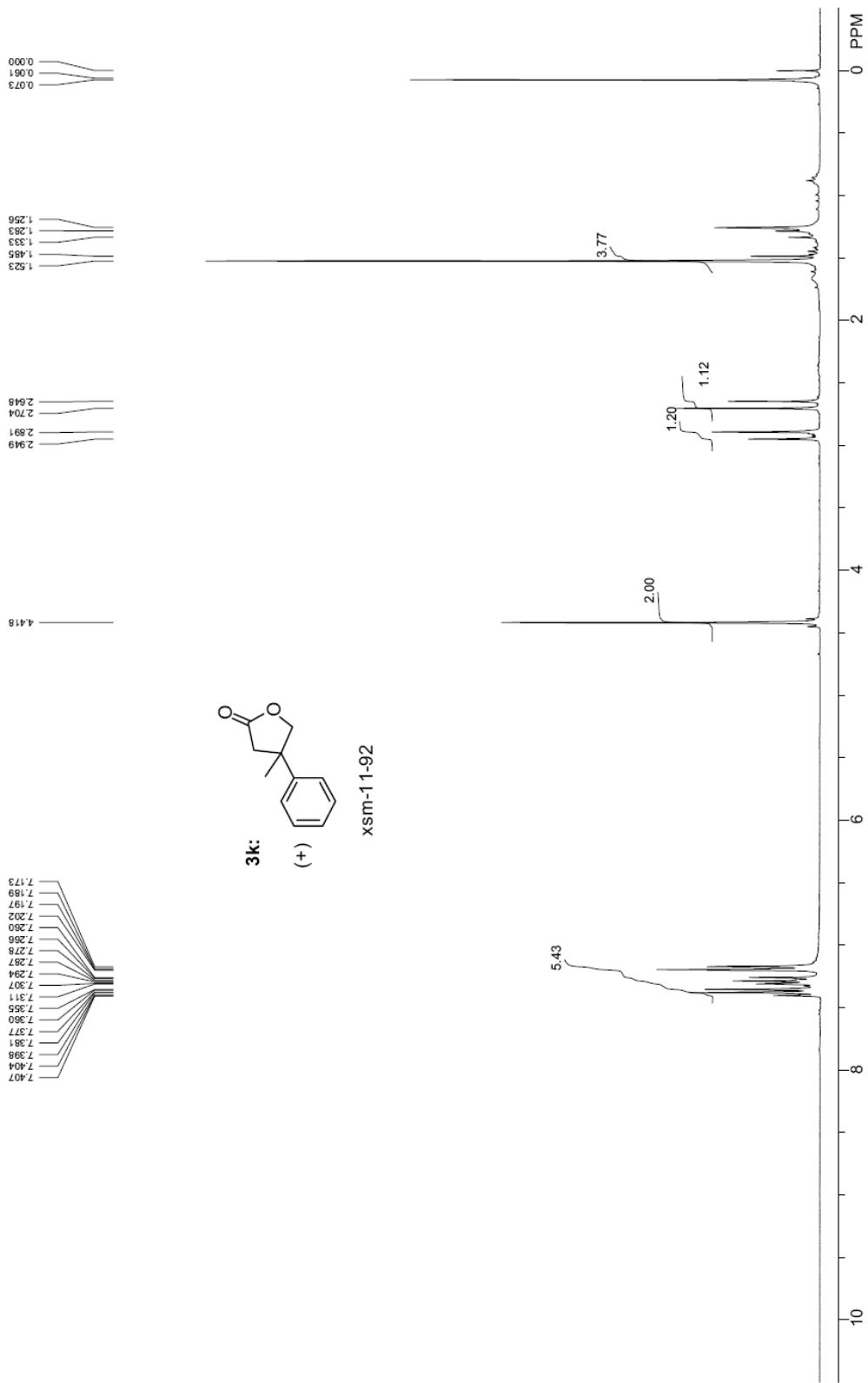
Results

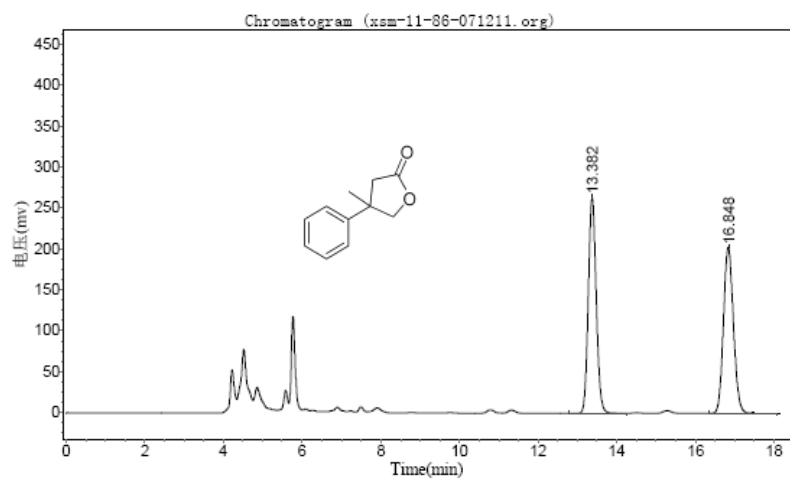
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		57.382	49456.828	3184712.500	49.5002
2		58.982	46582.254	3249021.750	50.4998
Total			96039.082	6433734.250	100.0000



Results

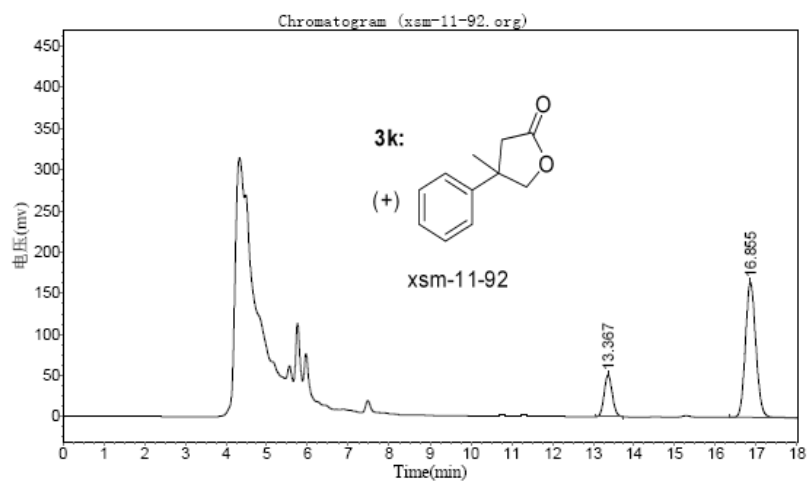
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		57.507	4370.330	272590.719	22.1782
2		59.058	14005.797	956500.875	77.8218
Total			18376.126	1229091.594	100.0000





Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.382	262657.563	3689598.750	50.6919
2		16.848	202464.406	3588880.000	49.3081
Total			465121.969	7278478.750	100.0000



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.367	50854.379	704733.688	19.4390
2		16.855	163899.297	2920622.500	80.5610
Total			214753.676	3625356.188	100.0000