

## Supporting Information for

### **3,3'- Br<sub>2</sub>-BINOL-Zn Complex: A Highly Efficient Catalyst for Enantioselective Hetro-Diels-Alder Reaction**

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#### **General Considerations**

<sup>1</sup>H NMR, <sup>13</sup>C NMR spectra were measured on a Bruker AM300 NMR spectrometer (300 MHz) with CDCl<sub>3</sub> as solvent and recorded in ppm relative to internal tetramethylsilane standard. Coupling constants, *J*, are listed in hertz. Mass spectra (EI, 70 ev) were taken on a HP5989A spectrometer. HRMS data were determined on a Kratos Concept instrument. Elemental analysis was preformed with an Elemental VARIO EL apparatus. Optical rotations were measured on a Perkin-Elmer 341 automatic polarimeter. HPLC analyses were carried out on a JASCO 1580 liquid chromatograph with a JASCO CD-1595 detector and AS-1555 autosampler. Hexane, tetrahydrofuran, toluene and diethyl ether were distilled from sodium benzophenone ketyl under argon and degassed before use. Dichloromethane was distilled from CaH<sub>2</sub> before use. All reactions were performed under argon. All the known chiral diol ligands were purchased from ACROS or prepared according to the procedure reported in the literatures<sup>1-9</sup>.

#### **General Procedure for the Screening of the Chiral Diols Ligands**

To a 1.5-mL polypropylene microtube were added 0.025 M toluene solution of **L** (0.01 mmol, 0.4mL) and 1 M solution of Et<sub>2</sub>Zn in hexane (0.012 mmol, 12 μL). The mixture was kept at room temperature for 0.5 h and then freshly distilled benzaldehyde (10.6 mg, 0.10 mmol) was added. Danishefsky's diene (17.2 mg, 0.1 mmol) was charged after the reaction mixture was kept at 0 °C for 30 min. The reaction was quenched by introducing 5 drops of trifluoroacetic acid after 24h. Internal standard biphenyl (10 mg) in toluene and saturated sodium bicarbonate aqueous solution (0.5 mL) were added to the quenched mixture. The organic layer

was separated and submitted to HPLC analysis for the determination of yields and enantiomeric excesses (*ee*). The yields were determined with a JASCO HPLC1500 with autosampler on Intersil CN-3 column: eluent Hexane/2-propanol (97:3); flow rate 0.5 mL/min; UV detection at  $\lambda = 254$  nm;  $t_R$  of biphenyl, 7.6 min (factor 1.000);  $t_R$  of benzaldehyde, 11.4 min (factor 1.208);  $t_R$  of 2-phenyl-2, 3-dihydro-4*H*-pyran-4-one, 23.0 min (factor 1.742). The enantiomeric excesses were determined by using the same HPLC analytical system on Chiralcel OD column: eluent Hexane/2-propanol (90:10); flow rate 1.0 mL/min; UV detection at  $\lambda = 254$  nm; retention time = 13.0 min (S enantiomer), 15.2 min (R enantiomer). The results were shown in Table 1(see the text).

### Investigation of Nonlinear Effect Using L6 Modified Catalyst

The examination of NLE was carried out following the similar procedure mentioned above at 0 °C with 10 mol% of nonenantio pure **L6**. The enantiomeric excesses of the **L6** employed for the reaction were measured by HPLC on Chiralcel AD column before they were submitted to the reactions. The yields were determined with a JASCO HPLC1500 with autosampler on Intersil CN-3 column: eluent Hexane/2-propanol (97:3); flow rate 0.5 mL/min; UV detection at  $\lambda = 254$  nm;  $t_R$  of biphenyl, 7.6 min (factor 1.000);  $t_R$  of benzaldehyde, 11.4 min (factor 1.208);  $t_R$  of 2-phenyl-2, 3-dihydro-4*H*-pyran-4-one, 23.0 min (factor 1.742). The enantiomeric excesses were determined by using the same HPLC analytical system on Chiralcel OD column: eluent hexane/2-propanol (90:10); flow rate 1.0 mL/min; UV detection at  $\lambda = 254$  nm; retention time = 13.0 min (S enantiomer), 15.2 min (R enantiomer). The results were shown in Table 1(see the text).

Table 1. Search for NLE in the catalytic system.

entry	Ligand ee (%) <sup>a</sup>	Yield (%)	Ee (%)	Configuration
1	0	17	0	-
2	9.8	20	11	S
3	19.7	39	12	S
4	39.1	47	1	S
5	49.3	41	41	R
6	60.0	52	54	R
7	80.0	82	84	R
8	>99	>99	93	R

<sup>a</sup> The ee values of ligand **L6** were determined by using HPLC on Chiralcel OD

column: eluent hexane/2-propanol (60:40); flow rate 1.0 mL/min; UV detection at = 254 nm; retention time = 13.9 min (R enantiomer), 19.5 min (S enantiomer).

## Investigation of Solvent Effect Using L6 Modified Catalyst

Table 2. Solvent effect on the **L6**-Zn catalyzed HDA reaction of **4** with **5a**.

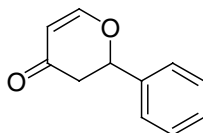
	THF	Et <sub>2</sub> O	CH <sub>2</sub> Cl <sub>2</sub>	Hexane
Ee (%)	51 (R)	28 (S)	46 (R)	7 (S)
Yield (%)	7	89	78	46

## The Procedure for Solvent-free Asymmetric Hetero-Diels- Alder Reaction

To a 1.5-mL polypropylene microtube was added 0.001 mmole of catalyst **L6/Zn** prepared by mixing **L6** and ZnEt<sub>2</sub> in 1:1.2 molar ratio in toluene (40 L, 0.25 M). Freshly distilled benzaldehyde (10.6 mg, 0.10 mmol) was added and Danishefsky's diene (17.2 mg, 0.1 mmol) was charged after the reaction mixture was kept at 0 °C for 30 min. The reaction was quenched by introducing 5 drops of trifluoroacetic acid after 24h. Internal standard biphenyl (10 mg) in toluene (0.1 mL) and saturated sodium bicarbonate aqueous solution (0.5 mL) were added to the quenched mixture. The organic layer was separated and submitted to HPLC analysis for the determination of yields and enantiomeric excesses (*ee*). (see Table 1 in the text).

## General Procedure for Catalytic Asymmetric Hetero-Diels-Alder Reaction Using L6-Zn Catalyst.

(*R*)-2-Phenyl-2,3-dihydro-4H-pyran-4-one **3a**



To a 1.5-mL polypropylene microtube were added 0.025 M toluene solution of **L6** (0.02 mmol, 0.8 mL) and 1 M solution of Et<sub>2</sub>Zn in hexane (0.024 mmol, 24 L). The mixture was kept at room temperature for 0.5 h and then freshly distilled benzaldehyde (21.7 mg, 0.20 mmol) was added. Danishefsky's diene (34.4 mg, 0.2 mmol) was charged after the reaction mixture was kept at -25 °C for 30 min. The reaction was quenched by introducing 10 drops of trifluoroacetic acid after 24 h. Saturated sodium bicarbonate aqueous solution (0.8 mL) was added to the quenched

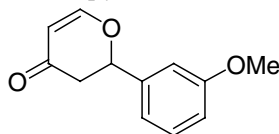
mixture. The aqueous layer was extracted with diethyl ether (3x15 mL), and the combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude material was purified by flash chromatography on silical gel with hexanes/ethyl acetate (4:1) as eluent to afford 34.8 mg (>99% yield) of 2-phenyl-2,3-dihydro-4*H*-pyran-4-one **3a** as colorless liquid with 97.5% ee (determined by HPLC on Chiralcel OD column, hexane : isopropanol = 90 : 10, flow rate = 1.0 mL / min, *t*<sub>R1</sub> = 11.8 min (*S*), *t*<sub>R2</sub> = 13.8 min (*R*). The absolute configuration was determined to be *R* by comparison of retention time with that reported in literature.<sup>10</sup>

IR (liquid film)  $\nu_{\text{max}}$  3064, 1676, 1596, 1402, 1272, 1228, 1210, 1040, 990, 934, 864,

826, 796, 760, 732, 720, 640, 612. <sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.46 (d, *J* = 6.6 Hz, 1H), 7.42-7.36 (m, 5H), 5.51 (dd, *J* = 5.7, 1.2 Hz, 1H), 5.41 (dd, *J* = 14.18, 3.4 Hz, 1H), 2.95-2.84 (m, 1H), 2.68-2.60 (m, 1H).

Following the same procedure mentioned above, the following 2-substituted-2,3-dihydro-4*H*-pyran-4-ones were prepared.

*2-(3-Methoxyphenyl)-2,3-dihydro-4H-pyran-4-one 3b*

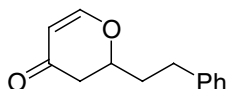


>99% yield, 98.2% ee.

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.49 (d, *J* = 5.97 Hz, 1H), 7.34 (t, *J* = 7.81 Hz, 1H), 6.99-6.90 (m, 3H), 5.54 (dd, *J* = 6.10, 1.15 Hz, 1H), 5.42 (dd, *J* = 14.36, 3.46 Hz, 1H), 3.83 (s, 3H), 2.95-2.85 (m, 1H), 2.70-2.62 (m, 1H). <sup>13</sup>C NMR (300MHz, CDCl<sub>3</sub>) 192.2, 163.3, 160.0, 139.6, 130.1, 118.4, 114.3, 111.9, 107.5, 81.1, 55.4, 43.5. EIMS *m/z* (relative intensity): 204 (*M*<sup>+</sup>, 20.33), 134 (100.00). HRMS (EI) calcd for C<sub>12</sub>H<sub>12</sub>O<sub>3</sub> (*M*<sup>+</sup>): 204.0786, found: 204.0753.

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol = 90:10, flow rate = 1.0 mL/min, *t*<sub>R1</sub> = 18.4 min (minor), *t*<sub>R2</sub> = 24.4 min (major).

*(S)-2-Phenylethyl-2,3-dihydro-4H-pyran-4-one 3c*



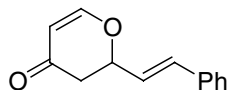
40.0% yield, 58.3% ee. The absolute configuration was determined to be *S* by comparison of retention time with that reported in literature.<sup>10</sup>

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.38 (d, *J* = 6.3 Hz, 1H), 7.32-7.15 (m, 5H), 5.42-5.40

(dd,  $J = 6.0, 0.9$  Hz, 1H), 4.40-4.39 (m, 1H), 2.84-2.76 (m, 2H), 2.61-2.41 (m, 2H), 2.18-2.13 (m, 1H), 2.00-1.88 (m, 1H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol = 90:10, flow rate = 1.0 mL/min,  $t_{R1} = 18.4$  min (minor),  $t_{R2} = 32.4$  min (major).

*(R)*-2-(*E*-Styryl)-2,3-dihydro-4H-pyran-4-one **3d**

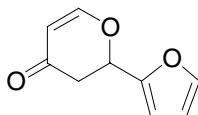


33.7% yield, 86.7 *ee*. The absolute configuration was determined to be *R* by comparison of retention time with that reported in literature.<sup>10</sup>

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.42-7.27 (m, 6H), 6.72 (d,  $J = 15.9$  Hz, 1H), 6.31 (dd,  $J = 15.9, 6.6$  Hz, 1H), 5.47 (d,  $J = 6.3$  Hz, 1H), 5.10-5.03 (m, 1H), 2.80-2.58 (m, 2H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol = 90:10, flow rate = 1.0 mL/min,  $t_{R1} = 20.8$  min (minor),  $t_{R2} = 42.3$  min (major).

*(R)*-2-(2-Furyl)-2,3-dihydro-4H-pyran-4-one **3e**

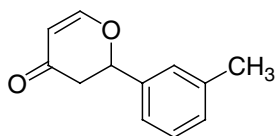


>99% yield, 96.2% *ee*. The absolute configuration was determined to be *R* by comparison of retention time with that reported in literature.<sup>10</sup>

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.49-7.47 (m, 1H), 7.39-7.36 (m, 1H), 6.47-6.40 (m, 2H), 5.52-5.45 (m, 2H), 3.15-3.04 (m, 1H), 2.77-2.70 (m, 1H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol = 95:5, flow rate = 0.5 mL/min,  $t_{R1} = 30.9$  min (major),  $t_{R2} = 33.7$  min (minor).

2-(3-Tolyl)-2,3-dihydro-4H-pyran-4-one **3f**<sup>10</sup>

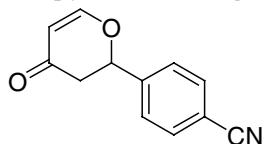


>99% yield, 96.4% *ee*.

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.47 (d,  $J = 5.7$  Hz, 1H), 7.30-7.16 (m, 4H), 5.52 (dd,  $J = 6.0, 0.9$  Hz, 1H), 5.39 (dd,  $J = 14.40, 3.60$  Hz, 1H), 2.97-2.86 (m, 1H), 2.68-2.61 (m, 1H), 2.39 (s, 3H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol = 90:10, flow rate = 1.0 mL/min,  $t_{R1}$  = 10.9 min (minor),  $t_{R2}$  = 12.9 min (major).

**2-(4-Cyanophenyl)-2,3-dihydro-4H-pyran-4-one **3g**<sup>10</sup>**

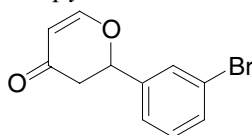


>99% yield, 96.8% *ee*.

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.76 (d, J=10 Hz, 2H), 7.56-7.50 (m, 3H), 5.59 (dd, J=6.1, 1.2 Hz, 1H), 5.48 (dd, J=13.7, 3.9 Hz, 1H), 2.89-2.66 (m, 2H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol 90:10, flow rate = 1.0 mL/min,  $t_{R1}$  = 41.1 min (minor),  $t_{R2}$  = 49.9 min (major).

**2-(3-Bromophenyl)-2,3-dihydro-4H-pyran-4-one **3h****

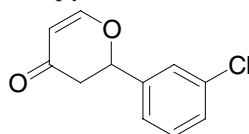


>99% yield, 95.7% *ee*.

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.58 (s, 1H), 7.54-7.47 (m, 2H), 7.33-7.27 (m, 2H), 5.55 (dd, J=6.0, 1.2 Hz, 1H), 5.42 (dd, J=14.2, 3.6 Hz, 1H), 2.91-2.80 (m, 1H), 2.69-2.62 (m, 1H). <sup>13</sup>C NMR (300MHz, CDCl<sub>3</sub>) 191.6, 163.0, 140.3, 132.1, 130.6, 129.3, 124.7, 123.0, 107.7, 80.2, 43.5. EIMS *m/z* (relative intensity): 254 ([M+2]<sup>+</sup>, 10.30), 252 (M<sup>+</sup>, 10.64), 184 (93.87), 182 (100.00). HRMS (EI) calcd for C<sub>11</sub>H<sub>9</sub>BrO<sub>2</sub> (M<sup>+</sup>): 251.9786, found: 251.9757. Anal. calcd for C<sub>11</sub>H<sub>9</sub>BrO<sub>2</sub>: C 52.20%, H 3.58%. Found: C 52.36%, H 3.93%.

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol 90:10, flow rate = 1.0 mL/min,  $t_{R1}$  = 12.9 min (minor),  $t_{R2}$  = 16.5 min (major).

**2-(3-Chlorophenyl)-2,3-dihydro-4H-pyran-4-one **3k**<sup>11</sup>**

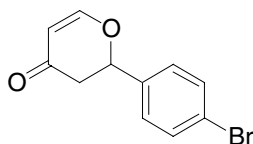


[ $\alpha$ ]<sub>D</sub><sup>25</sup> = -83.5 ° (C = 1.600, CHCl<sub>3</sub>), 98.1% *ee*.

<sup>1</sup>H NMR (300MHz, CDCl<sub>3</sub>) 7.49 (d, J=6.0 Hz, 1H), 7.43 (t, J=0.6 Hz, 1H), 7.40-7.30 (m, 2H), 7.28-7.26 (m, 2H), 5.54 (dd, J=6.6 Hz, 1.2 Hz, 1H), 5.41 (dd, J=14.4, 3.6 Hz, 1H), 2.91-2.81 (m, 1H), 2.70-2.63 (m, 1H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol 90:10, flow rate = 1.0 mL/min,  $t_{R1}$  = 13.1 min (minor),  $t_{R2}$  = 17.0 min (major).

**2-(4-Bromophenyl)-2,3-dihydro-4H-pyran-4-one **3j****

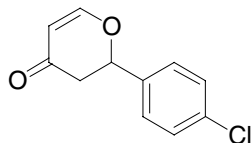


>99% yield, 94.5% *ee*.

$^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ) 7.57 (d,  $J$  = 9.3 Hz, 2H), 7.46 (d,  $J$  = 6.0 Hz, 1H), 7.29 (d,  $J$  = 9.3, 2H), 5.53 (dd,  $J$  = 6.1, 1.2 Hz, 1H), 5.43 (dd,  $J$  = 14.2, 3.4 Hz, 1H), 2.91-2.81 (m, 1H), 2.68-2.61 (m, 1H).  $^{13}\text{C}$  NMR (300MHz,  $\text{CDCl}_3$ ) 191.7, 163.1, 137.1, 132.1, 127.9, 123.0, 107.6, 80.4, 43.5. EIMS  $m/z$  (relative intensity): 254 ( $[\text{M}+2]^+$ , 5.13), 252 ( $\text{M}^+$ , 5.42), 184 (96.52), 182 (100.00). HRMS (EI) calcd for  $\text{C}_{11}\text{H}_9\text{BrO}_2$  ( $\text{M}^+$ ): 251.9786, found: 251.9780. Anal. calcd for  $\text{C}_{11}\text{H}_9\text{BrO}_2$ : C 52.20%, H 3.58%. Found: C 52.47%, H 3.82%.

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol 90:10, flow rate = 1.0 mL/min,  $t_{\text{R}1}$  = 15.4 min (minor),  $t_{\text{R}2}$  = 19.8 min (major).

**2-(4-Chlorophenyl)-2,3-dihydro-4H-pyran-4-one  $3k^{10}$**

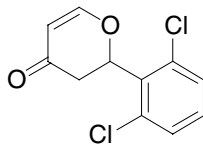


>99% yield, 95.1% *ee*.

$^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ) 7.58 (d,  $J$  = 6.0 Hz, 1H), 7.52-7.44 (m, 4H), 5.65 (dd,  $J$  = 6.1, 1.0 Hz, 1H), 5.55 (dd,  $J$  = 14.2, 3.6 Hz, 1H), 3.02-2.92 (m, 1H), 2.79-2.72 (m, 1H).

The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane:isopropanol 90:10, flow rate = 1.0 mL/min,  $t_{\text{R}1}$  = 14.1 min (minor),  $t_{\text{R}2}$  = 17.2 min (major).

**2-(2,6-dichlorophenyl)-2,3-dihydro-4H-pyran-4-one  $3l$**



$[\alpha]_{\text{D}}^{25}$  = +12.8° ( $C$ =1.6,  $\text{CHCl}_3$ ), 82.4% yield, 89.7% *ee*.

$^1\text{H}$  NMR (300MHz,  $\text{CDCl}_3$ ) 7.49 (dd,  $J$  = 6.6 Hz, 1H), 7.39-7.37 (m, 2H), 7.26 (t,  $J$  = 9.0 Hz), 6.23 (dd,  $J$  = 15.6, 4.2 Hz, 1H), 5.54 (dd,  $J$  = 6.3, 1.2 Hz, 1H), 3.54 (dd,  $J$  = 17.1, 15.6 Hz, 1H), 2.52-2.45 (m, 1H).  $^{13}\text{C}$  NMR (300MHz,  $\text{CDCl}_3$ ) 191.5, 163.1, 135.3, 132.0, 130.7, 129.8, 107.3, 77.6, 38.8. EIMS  $m/z$  (relative intensity): 242 ( $\text{M}^+$ , 3.82), 174 (63.02), 172 (100.00). HRMS (EI) calcd for  $\text{C}_{11}\text{H}_8\text{Cl}_2\text{O}_2$  ( $\text{M}^+$ ): 241.9901, found: 241.9916.

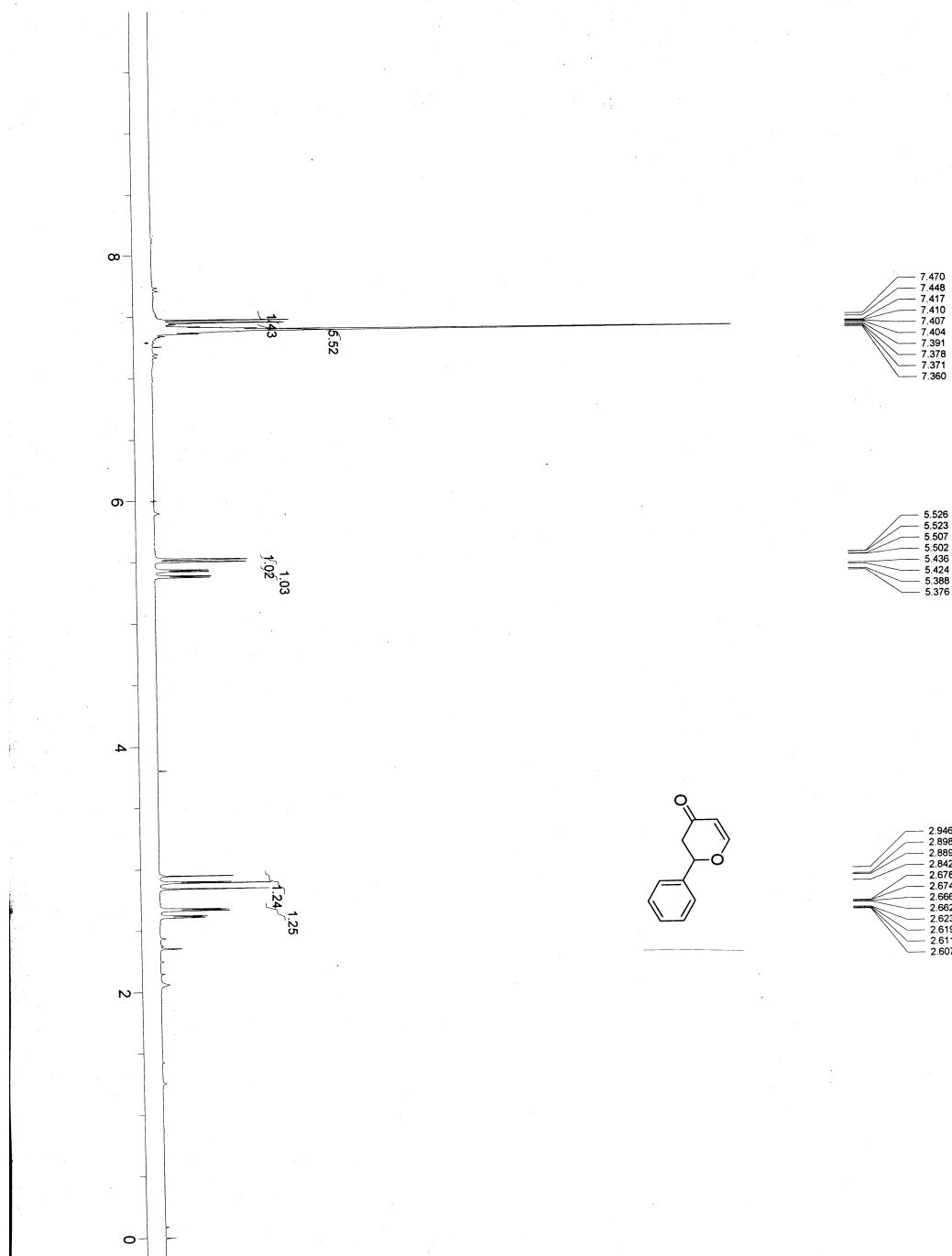
The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane:isopropanol 99.5:0.5, flow rate = 1.0 mL/min,  $t_{\text{R}1}$  = 21.04 min (major),  $t_{\text{R}2}$  =

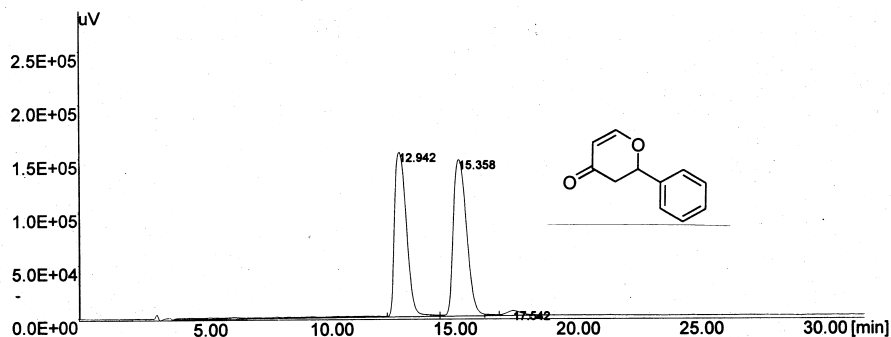
22.47 min (minor).

## References

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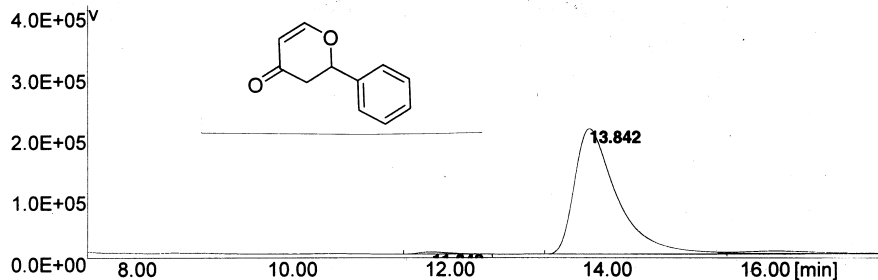
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Total Area of Peak = 10679376.648 [uV.Sec]



File name : DHF501-1-001.CH2

Info :

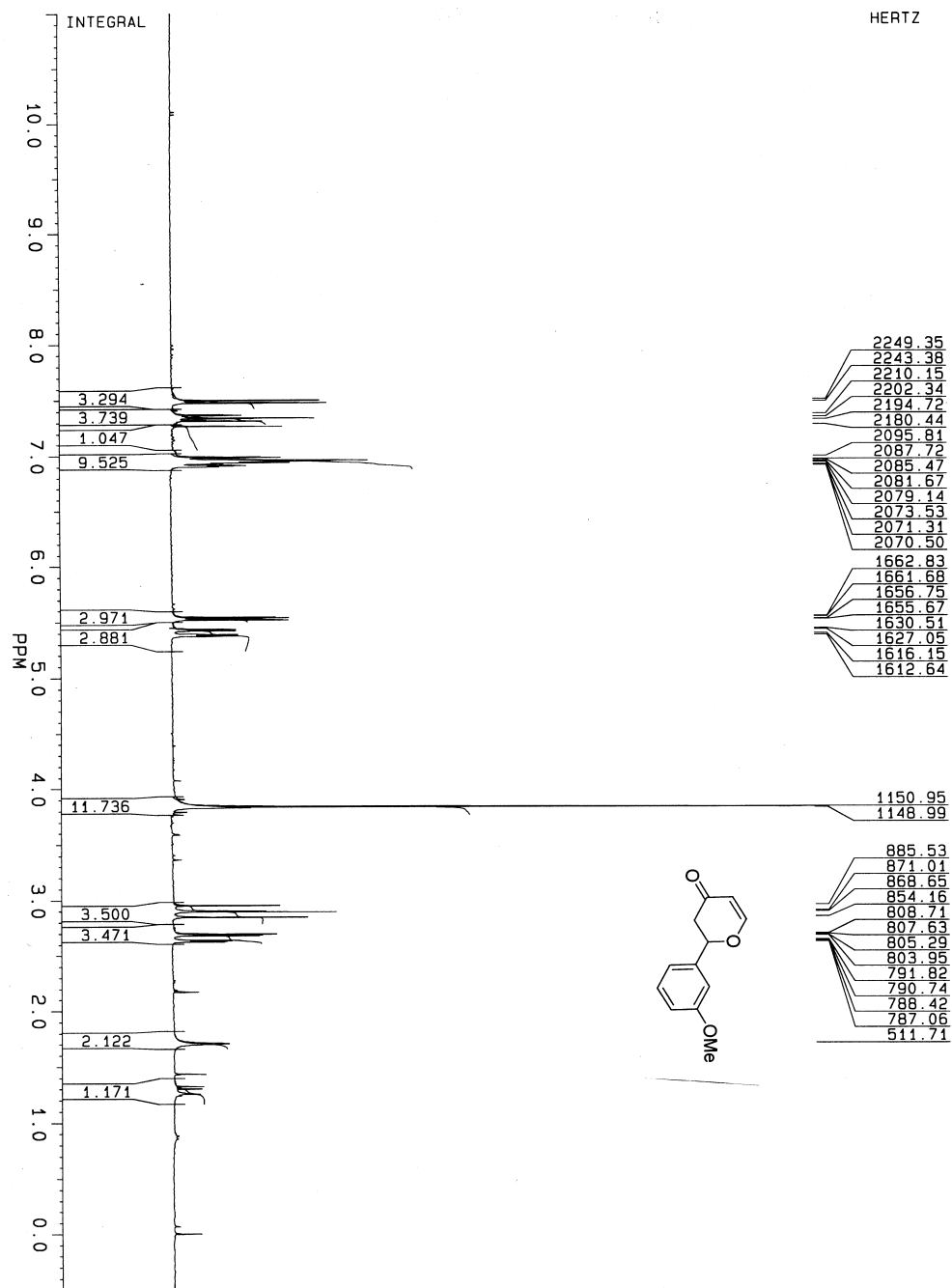
Vial # = 1 Rack # = 1  
 Injection Date : 24-Sep-2006 20:42:36  
 Curr. Date : 22-Oct-2002 10:18:38  
 User : DEFAULT  
 Group : DATA  
 Control Method : COMBIN EE

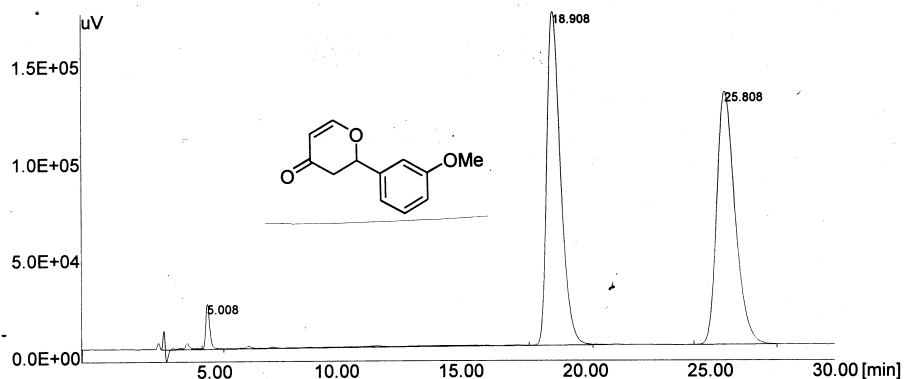
GA entry 1 in Table 2

ee: 97.5%

#	Name	RT	Area[v.Sec]	Quantity
1		11.842	97493.000	0.000
2		13.842	7654938.161	0.000

Total Area of Peak = 7752431.161 [v.Sec]





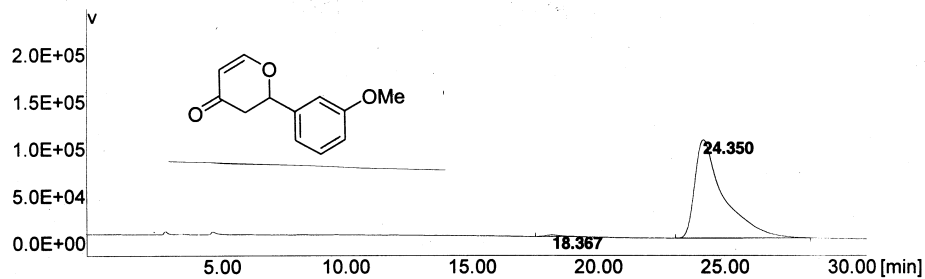
File name : 1j-rac-3-OMe-pyrone002.CH2

Info :  
Chiralcel OD Column; 1.0mL/min; 90:10

Vial # = 1 Rack # = 1  
Injection Date : 10-Apr-2001 10:09:10  
Curr. Date : 10-Apr-2001 10:42:06  
User : DEFAULT  
Group : DATA  
Control Method : M-OME-DIHYDROPYRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		5.008	254526.834	0.000
2		18.908	6537639.608	0.000
3		25.808	6580419.500	0.000

Total Area of Peak = 13372585.942 [uV.Sec]



File name : dhfm-methoxyl-003.CH2

Info :

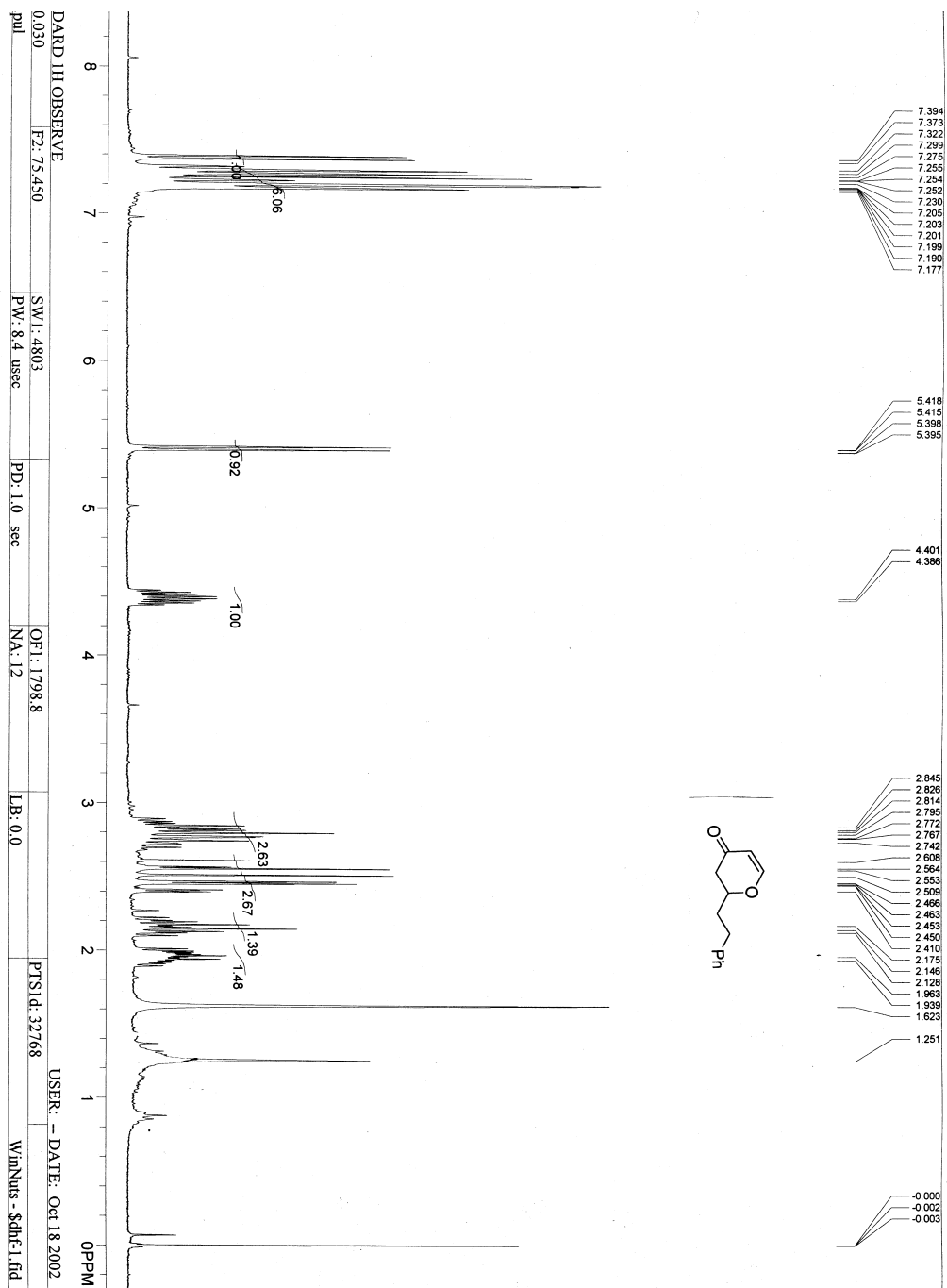
Vial # = 1 Rack # = 1  
Injection Date : 21-Oct-2002 20:31:52  
Curr. Date : 22-Oct-2002 10:04:44  
User : DEFAULT  
Group : DATA  
Control Method : COMBIN EE

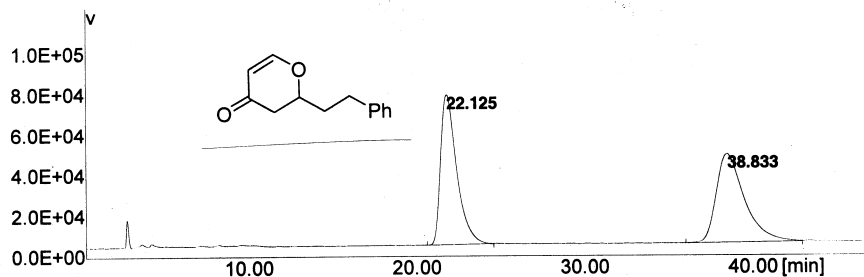
#	Name	RT	Area[v.Sec]	Quantity
1		18.367	72873.965	0.000
2		24.350	8208101.500	0.000

Total Area of Peak = 8280975.465 [v.Sec]

6b, entry 2 in Table 2

ee: 48.2%





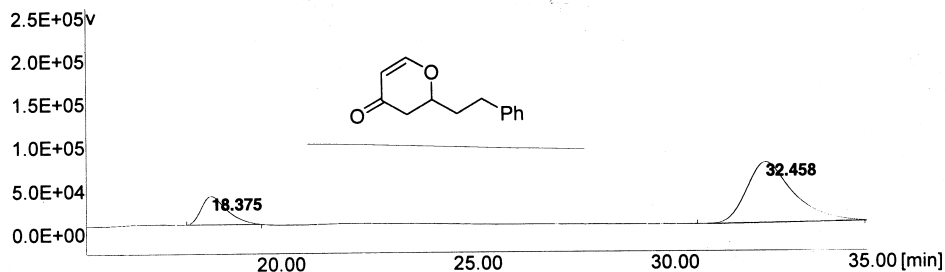
File name : dhfrac88-010.CH2

Info :

Vial # = 6 Rack # = 1  
 Injection Date : 22-Oct-2002 11:07:04  
 Curr. Date : 22-Oct-2002 12:11:54  
 User : DEFAULT  
 Group : DATA  
 Control Method : COMBIN EE

#	Name	RT	Area[v.Sec]	Quantity
1		22.125	4889994.500	0.000
2		38.833	5026113.435	0.000

Total Area of Peak = 9916107.935 [v.Sec]



File name : LS6A4-2-003.CH2

Info :

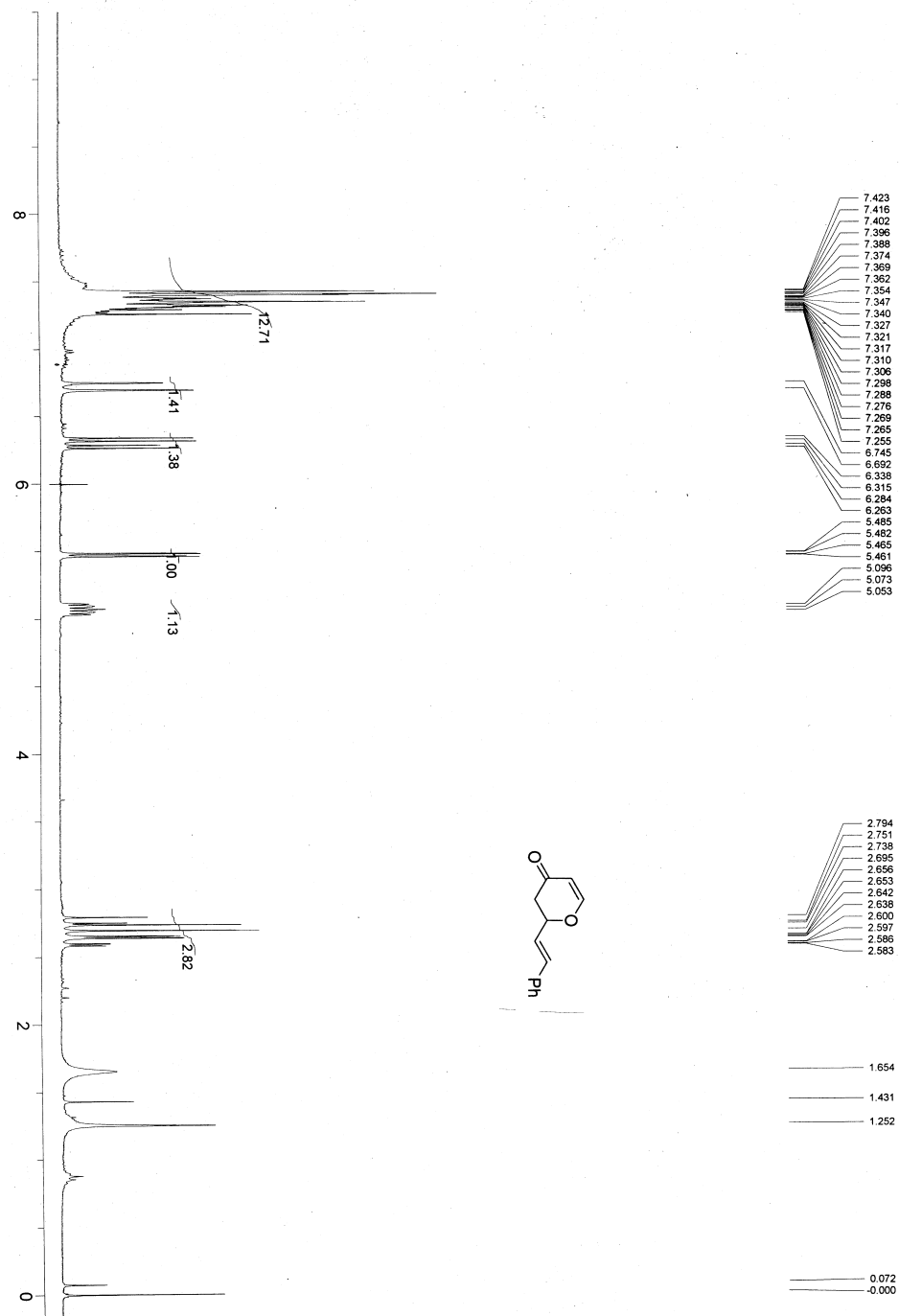
Vial # = 3 Rack # = 1  
 Injection Date : 26-Jun-2006 13:31:50  
 Curr. Date : 22-Oct-2002 10:20:22  
 User : DEFAULT  
 Group : DATA  
 Control Method : COMBIN EE

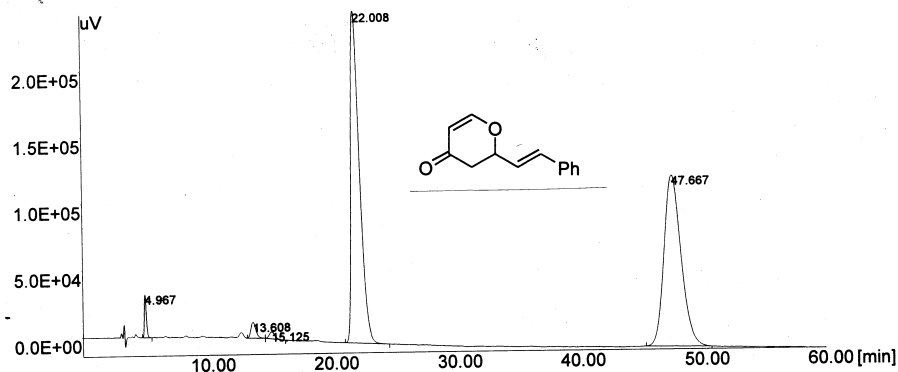
6C, entry 3 in Table 2

ee: 58.3%

#	Name	RT	Area[v.Sec]	Quantity
1		18.375	1494926.500	0.000
2		32.458	5683998.220	0.000

Total Area of Peak = 7178924.720 [v.Sec]





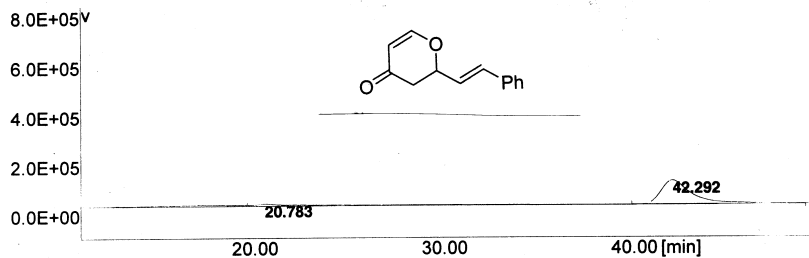
File name : lj-rac-p-Cinna-pyrone005.CH2

Info :  
Chiralcel OD Column; 1.0mL/min; 90:10

Vial # = 1 Rack # = 1  
Injection Date : 10-Apr-2001 11:18:44  
Curr. Date : 10-Apr-2001 12:30:32  
User : DEFAULT  
Group : DATA  
Control Method : P-CINNA-DIHYDROPYRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		4.967	328227.000	0.000
2		13.608	330860.000	0.000
3		15.125	160713.667	0.000
4		22.008	11970240.722	0.000
5		47.667	12178566.500	0.000

Total Area of Peak = 24968607.889 [uV.Sec]



File name : LS3A5-2-002.CH2

Info :

Vial # = 2 Rack # = 1  
Injection Date : 26-Jun-2006 20:17:38  
Curr. Date : 22-Oct-2002 10:22:30  
User : DEFAULT  
Group : DATA  
Control Method : COMBIN EE

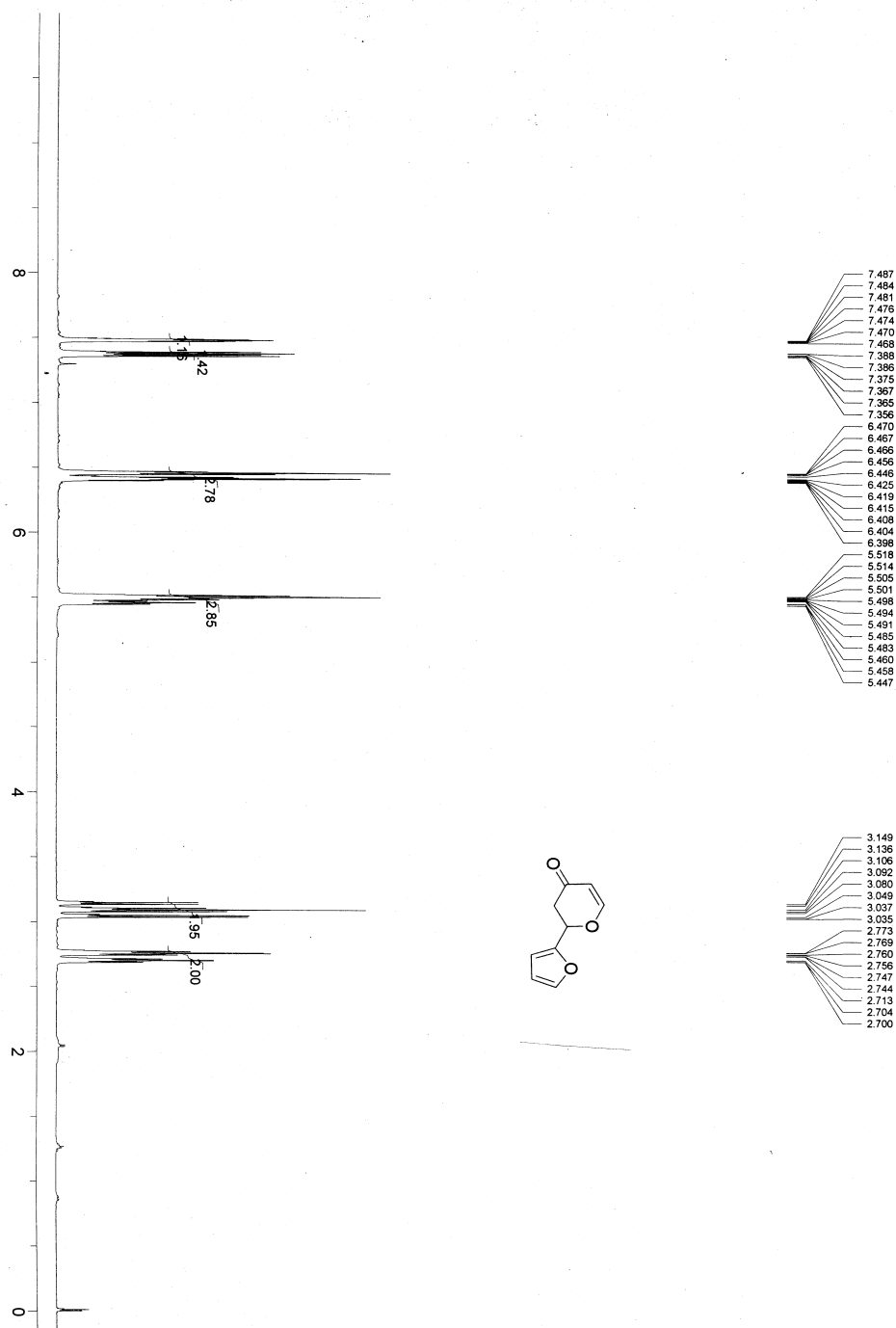
*6d, entry 4 in Table 2*

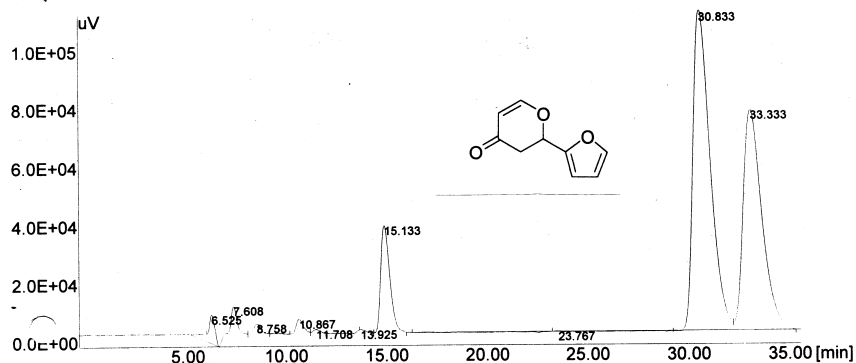
*ee: 86.7%*

#	Name	RT	Area[v.Sec]	Quantity
1		20.783	797826.334	0.000
2		42.292	1117718.856	0.000

Total Area of Peak = 11915545.190 [v.Sec]







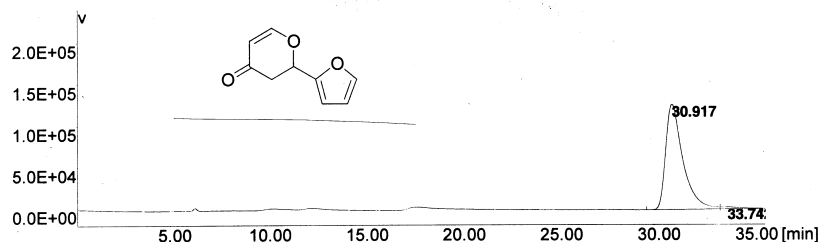
File name : lj-rac-furyl818.CH2

Info :

Vial # = 1 Rack # = 1  
 Injection Date : 19-Jul-2001 15:53:40  
 Curr. Date : 19-Jul-2001 16:30:02  
 User : DEFAULT  
 Group : DATA  
 Control Method : FURYL-DIHYPRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		6.525	182944.864	0.000
2		7.608	0.000	0.000
3		8.758	74437.000	0.000
4		10.867	113772.500	0.000
5		11.708	26853.500	0.000
6		13.925	19933.000	0.000
7		15.133	1126605.500	0.000
8		23.767	1395.842	0.000
9		30.833	6433773.200	0.000
10		33.333	4823108.732	0.000

Total Area of Peak = 12802824.137 [uV.Sec]



File name : FURY-001.CH2

Info :

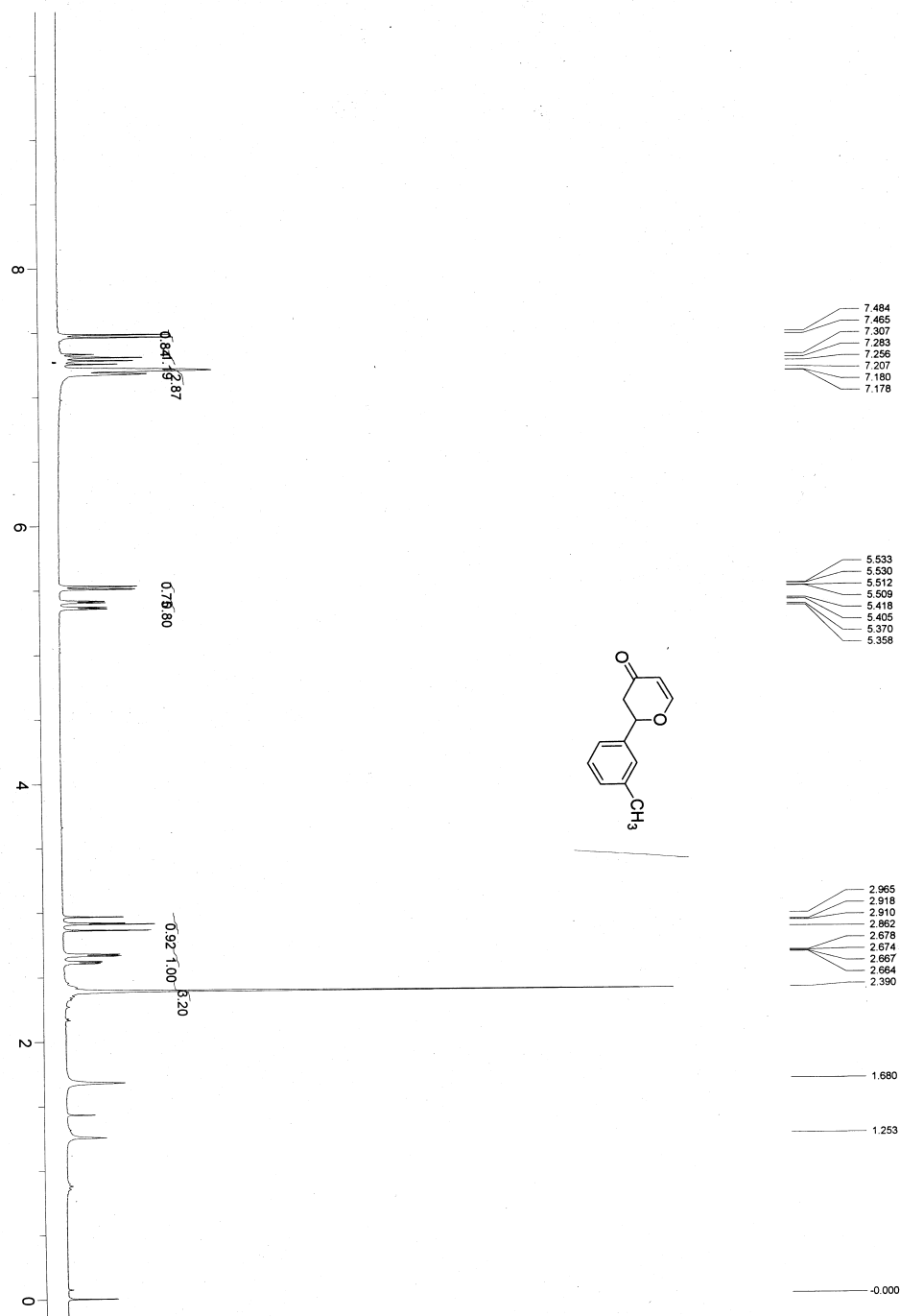
Vial # = 1 Rack # = 1  
 Injection Date : 18-Oct-2006 8:51:12  
 Curr. Date : 22-Oct-2002 10:17:04  
 User : DEFAULT  
 Group : DATA  
 Control Method : FURYL

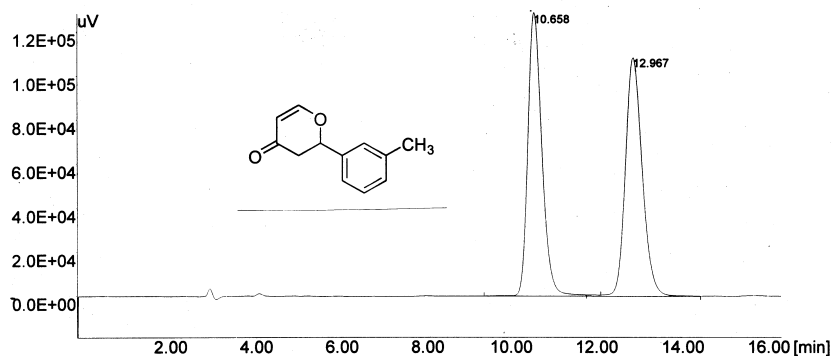
*6e, Entry 5 in Table 2*

*cc: 96.2%*

#	Name	RT	Area[v.Sec]	Quantity
1		30.917	7712558.487	0.000
2		33.742	155166.958	0.000

Total Area of Peak = 7867725.445 [v.Sec]





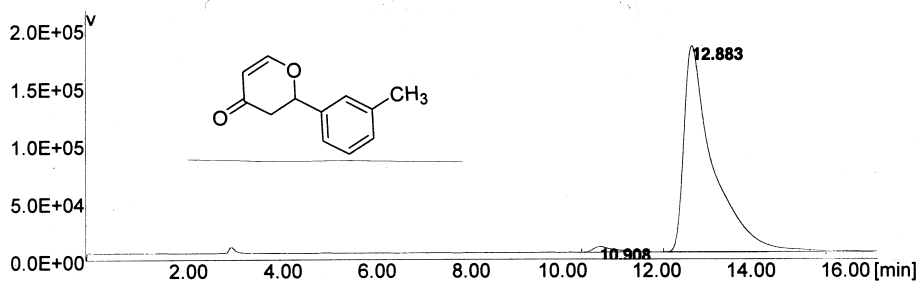
File name : m-me-dihyprone091.CH2

Info :  
chiralcel OD 90:10 1ml/min

Vial # = 1 Rack # = 1  
Injection Date : 18-Apr-2001 11:12:02  
Curr. Date : 18-Apr-2001 11:28:48  
User : DEFAULT  
Group : DATA  
Control Method : M-ME-DIHIPRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		10.658	2937875.862	0.000
2		12.967	2940475.423	0.000

Total Area of Peak = 5878351.285 [uV.Sec]



File name : dhfm-methxyl-004.CH2

Info :

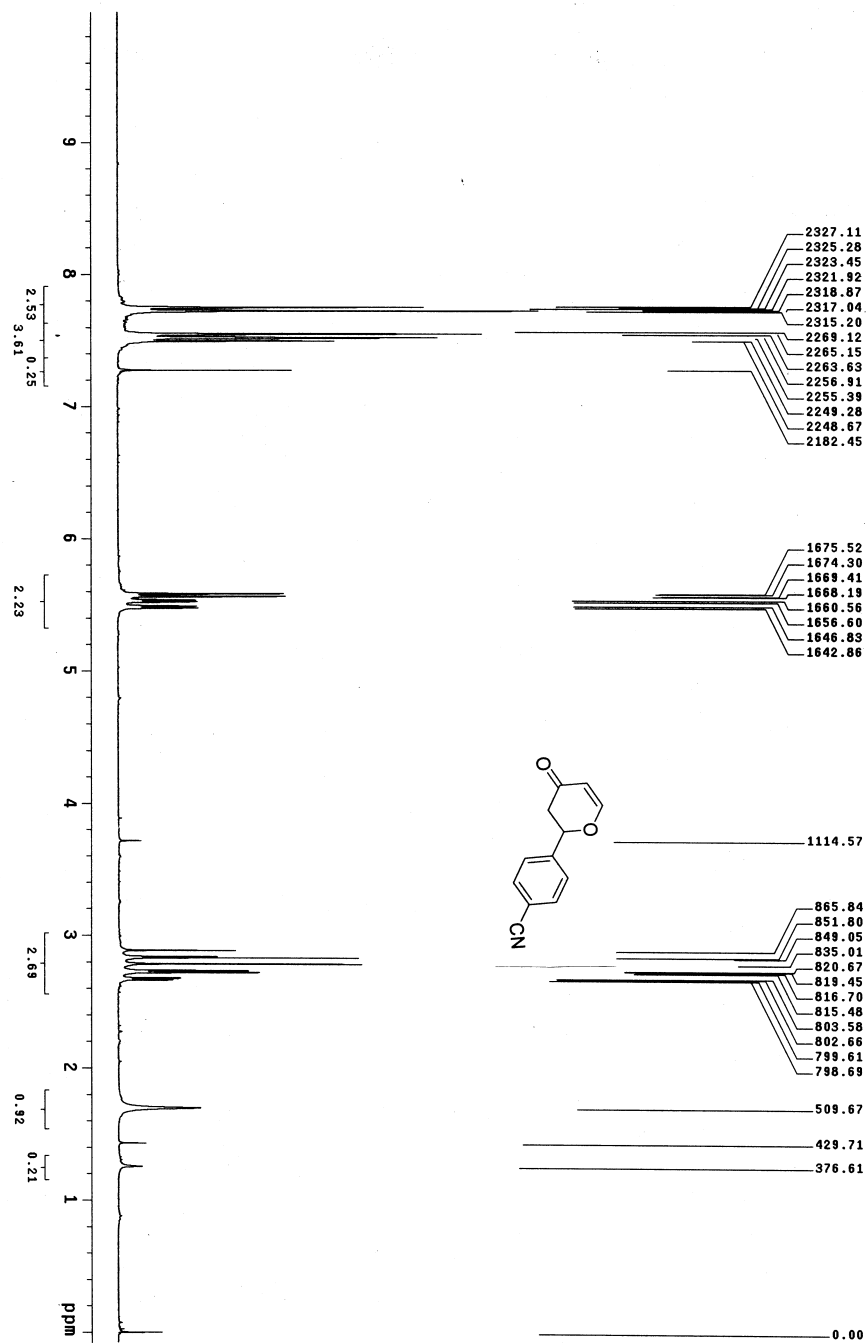
Vial # = 2 Rack # = 1  
Injection Date : 21-Oct-2002 21:04:44  
Curr. Date : 22-Oct-2002 10:06:42  
User : DEFAULT  
Group : DATA  
Control Method : COMBIN EE

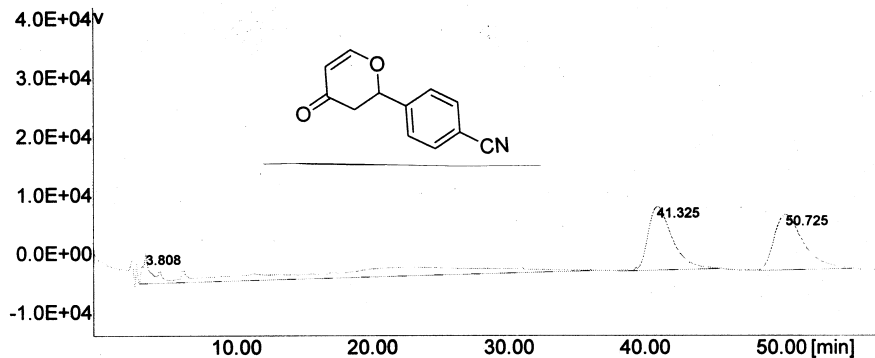
#	Name	RT	Area[v.Sec]	Quantity
1		10.908	142317.237	0.000
2		12.883	7687504.770	0.000

Total Area of Peak = 7829822.007 [v.Sec]

*6f. entry 6 in Table 2*

*ee: 96.4%*





File name : du-77-rac-006.CH2

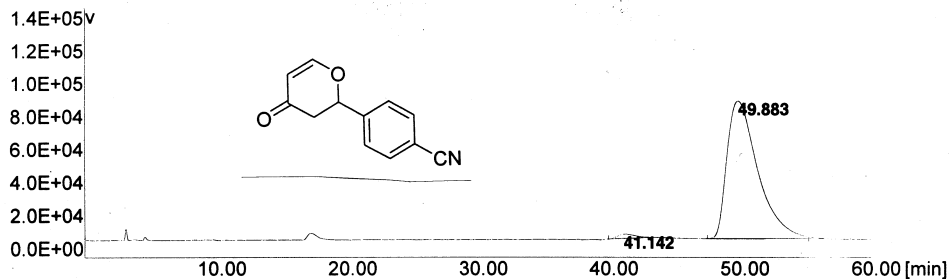
Info :

Vial # = 1 Rack # = 1  
 Injection Date : 24-Apr-2002 13:07:12  
 Curr. Date : 24-Apr-2002 14:05:50  
 User : DU  
 Group : DATA  
 Control Method : COMBINEE

*rac*

#	Name	RT	Area[v.Sec]	Quantity
1		3.808	130124.183	0.000
2		41.325	1259920.692	0.000
3		50.725	1378390.542	0.000

Total Area of Peak = 2768435.417 [v.Sec]



File name : dhf78-009.CH2

Info :

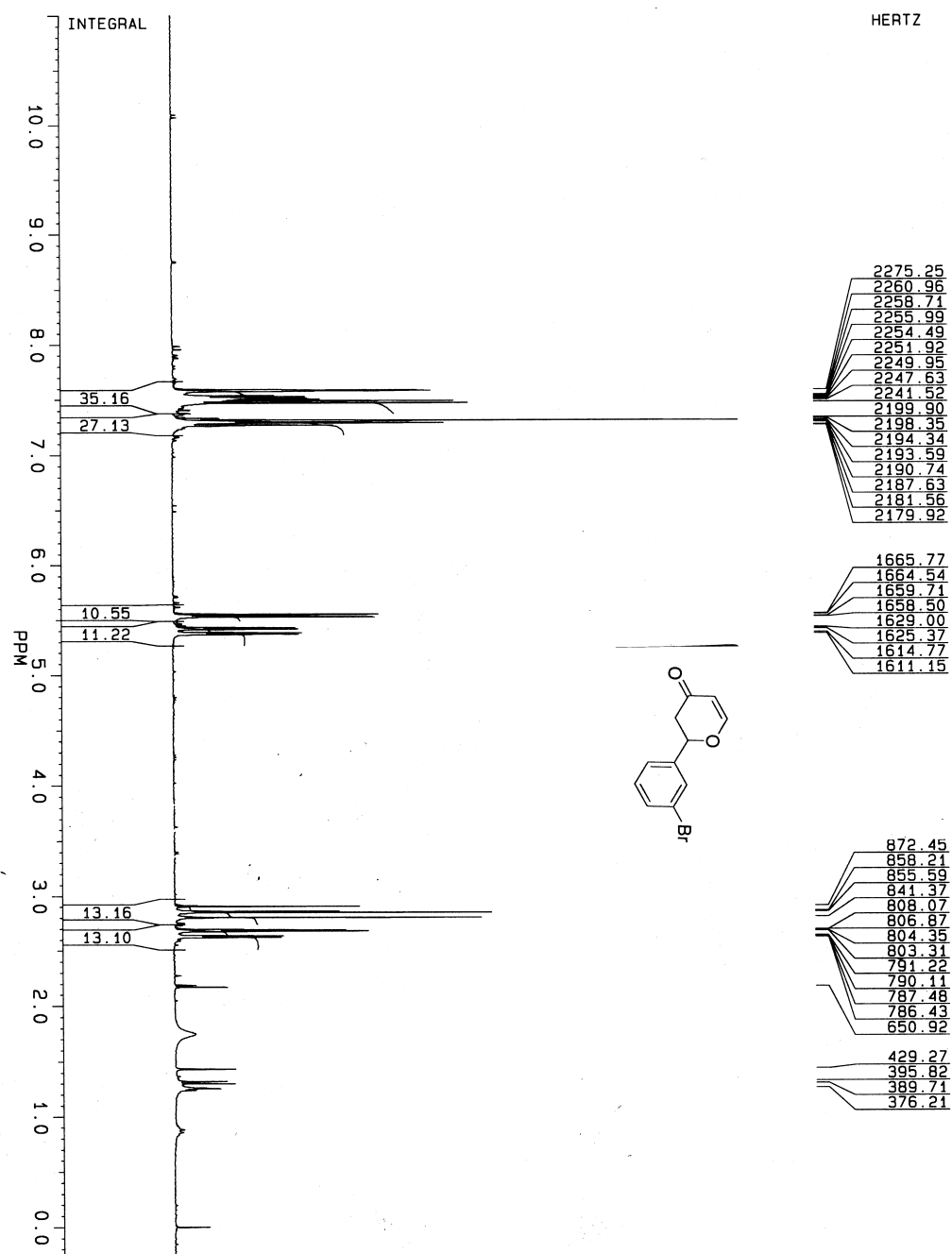
Vial # = 5 Rack # = 1  
 Injection Date : 22-Oct-2002 9:54:32  
 Curr. Date : 22-Oct-2002 11:00:08  
 User : DEFAULT  
 Group : DATA  
 Control Method : COMBIN EE

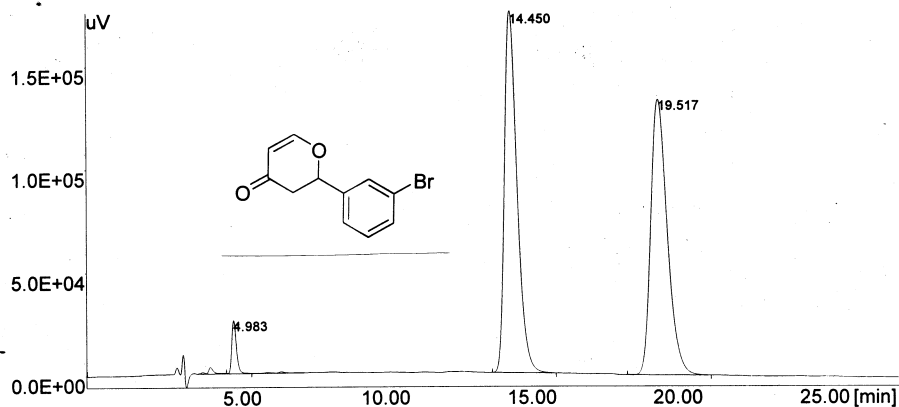
*6g, entry 7 in Table 2*

*ee: 96.8%*

#	Name	RT	Area[v.Sec]	Quantity
1		41.142	224174.556	0.000
2		49.883	1338835.790	0.000

Total Area of Peak = 13613010.346 [v.Sec]





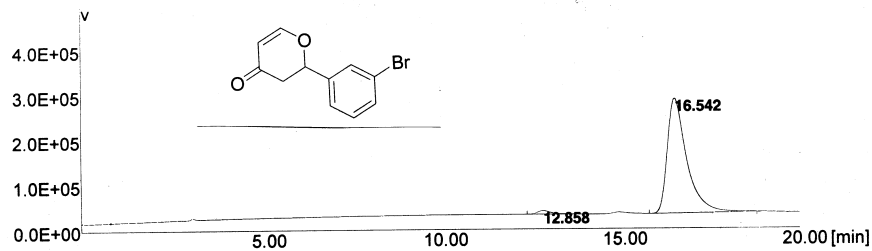
File name : lj-rac-3-Br-pyrone004.CH2

Info :  
Chiralcel OD Column; 1.0mL/min; 90:10

Vial # = 1 Rack # = 1  
Injection Date : 10-Apr-2001 10:43:24  
Curr. Date : 10-Apr-2001 11:14:14  
User : DEFAULT  
Group : DATA  
Control Method : M-BR-DIHYDROPYRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		4.983	272501.751	0.000
2		14.450	5141736.475	0.000
3		19.517	5176939.000	0.000

Total Area of Peak = 10591177.226 [uV.Sec]



File name : O-BR001.CH2

Info :

Vial # = 2 Rack # = 1  
Injection Date : 18-Oct-2006 9:53:04  
Curr. Date : 22-Oct-2002 10:12:14  
User : DEFAULT  
Group : DATA  
Control Method : COMBIN EE

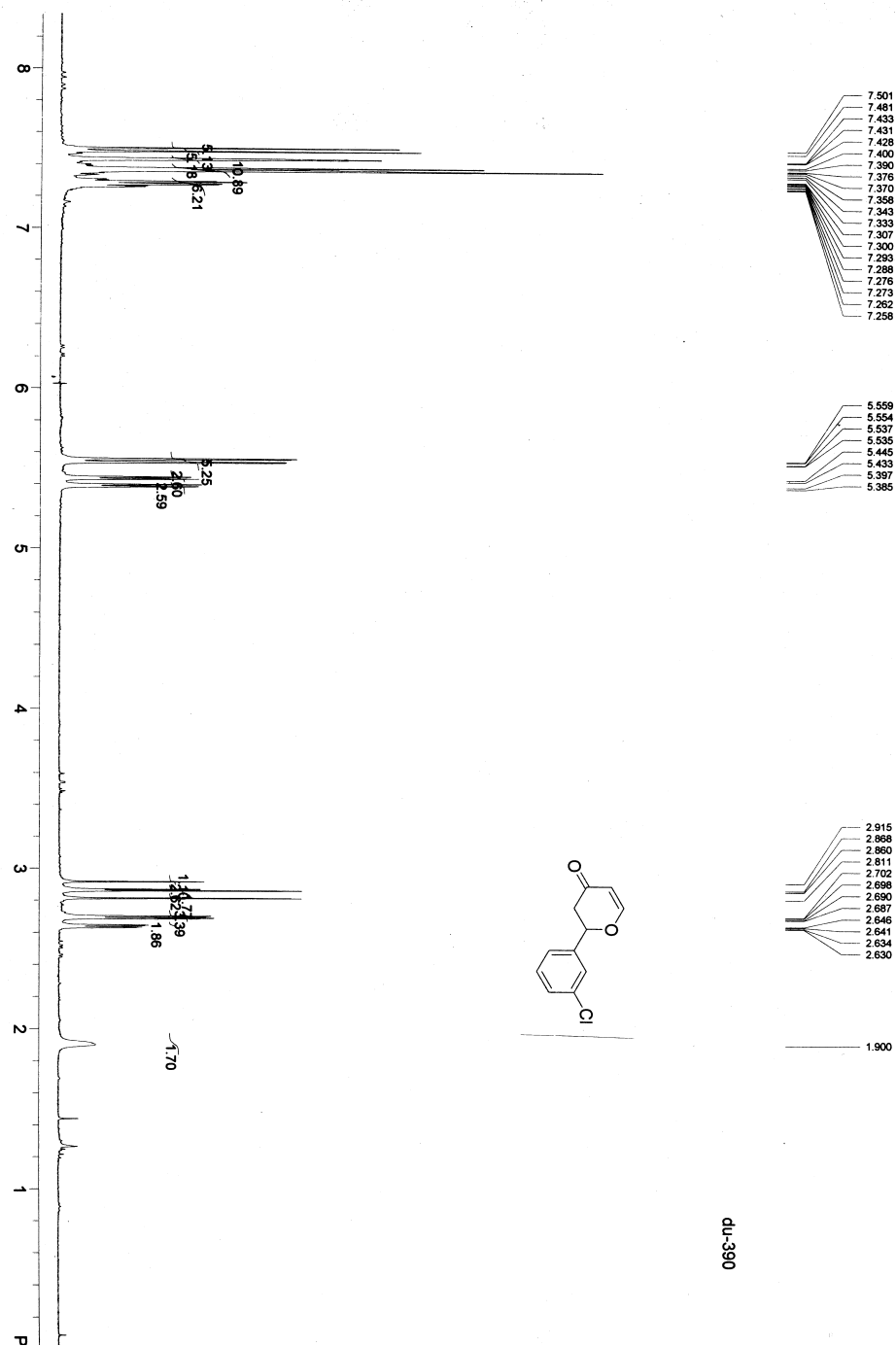
#	Name	RT	Area[v.Sec]	Quantity
1		12.858	211379.582	0.000
2		16.542	9505774.923	0.000

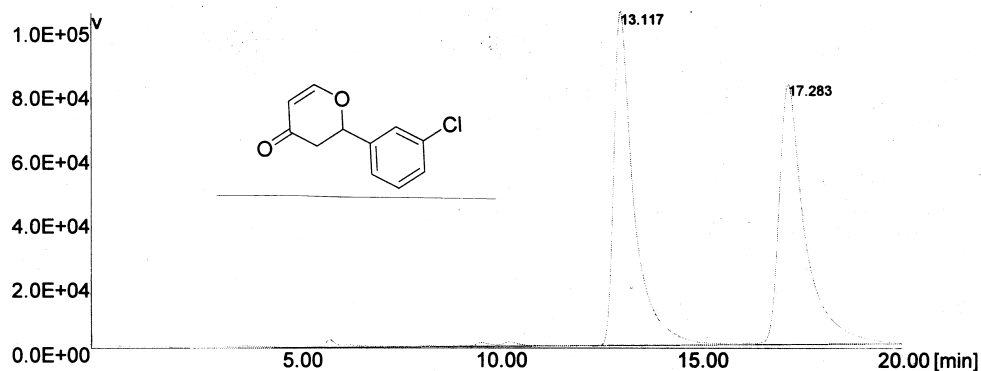
Total Area of Peak = 9717154.505 [v.Sec]

6h, entry 8 in Table 2

ee: 95.7%







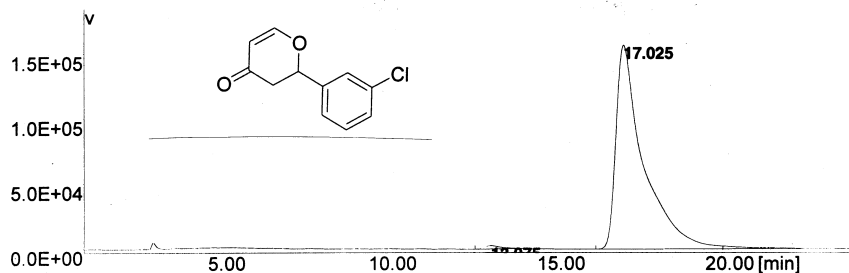
File name : du-88-rac-028.CH2

Info :

Vial # = 1 Rack # = 1  
 Injection Date : 28-Apr-2002 15:08:52  
 Curr. Date : 28-Apr-2002 15:29:22  
 User : DU  
 Group : DATA  
 Control Method : COMBINEE

#	Name	RT	Area[v.Sec]	Quantity
1		13.117	3595050.984	0.000
2		17.283	3590182.177	0.000

Total Area of Peak = 7185233.162 [v.Sec]



File name : dhfm-chloro-005.CH2

Info :

Vial # = 3 Rack # = 1  
 Injection Date : 21-Oct-2002 21:23:42  
 Curr. Date : 22-Oct-2002 10:08:56  
 User : DEFAULT  
 Group : DATA  
 Control Method : COMBIN EE

#	Name	RT	Area[v.Sec]	Quantity
1		13.075	85984.500	0.000
2		17.025	8890047.621	0.000

Total Area of Peak = 8976032.121 [v.Sec]

6i, entry 9 in Table 2

ee: 98.1%

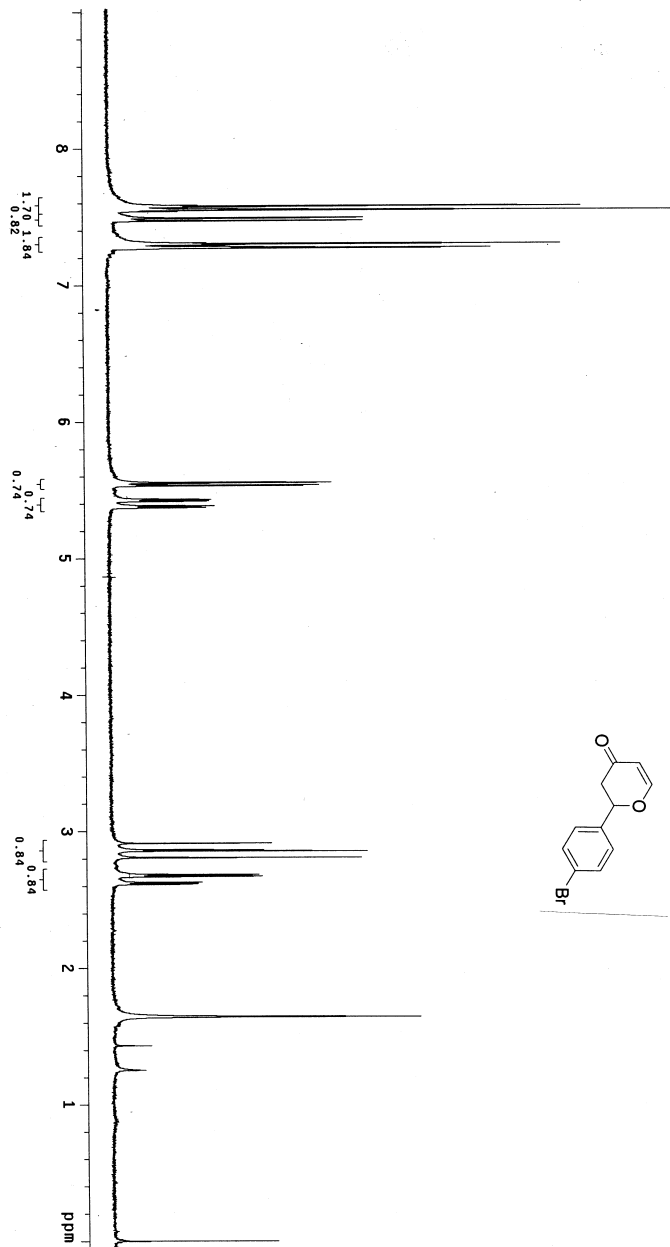
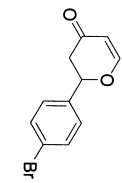
H1  
 VY-34  
 CAC13  
 20010224  
 Pulse Sequence: szpu1  
 Solvent: cdcl3  
 Ambient temperature  
 Mercury-3000  
 Relax. delay: 7.500 sec  
 Pulse: 22.5 deg  
 Acq. time: 1.400 sec  
 F2: 300.136 MHz  
 16 repetitions  
 OBSERVE H1, 300.136 MHz  
 DATA PROCESSING  
 7.443333 sec  
 Total time 0 min, 53 sec

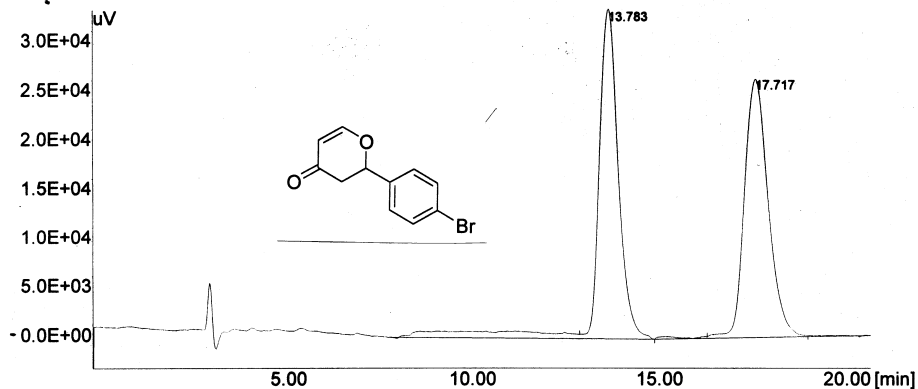
1665.81  
 1664.59  
 1659.70  
 1656.48  
 1628.68  
 1625.26  
 1614.51  
 1610.85

873.45  
 859.28  
 856.60  
 842.43  
 805.55  
 804.08  
 801.88  
 800.66  
 788.69  
 787.47  
 785.03  
 783.81

493.39

0.00





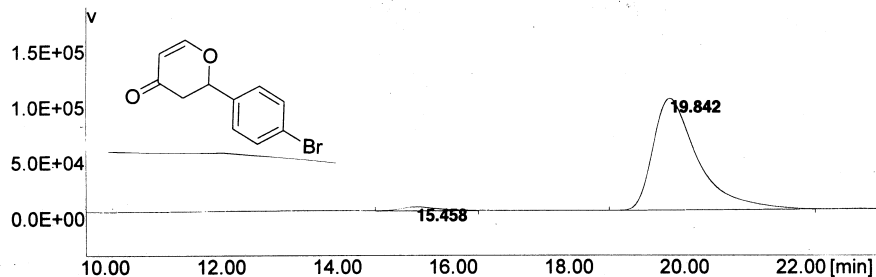
File name : p-Br-dihydroprone001.CH2

Info :  
Chiracel OD; Hex:iPr=90:10; 1.0mL/min; .

Vial # = 1 Rack # = 1  
Injection Date : 23-May-2001 17:57:56  
Curr. Date : 23-May-2001 18:41:18  
User : DEFAULT  
Group : DATA  
Control Method : P-BR-DIHYDROPYRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		13.783	1163437.404	0.000
2		17.717	1157950.627	0.000

Total Area of Peak = 2321388.031 [uV.Sec]



File name : du-75-001.CH2

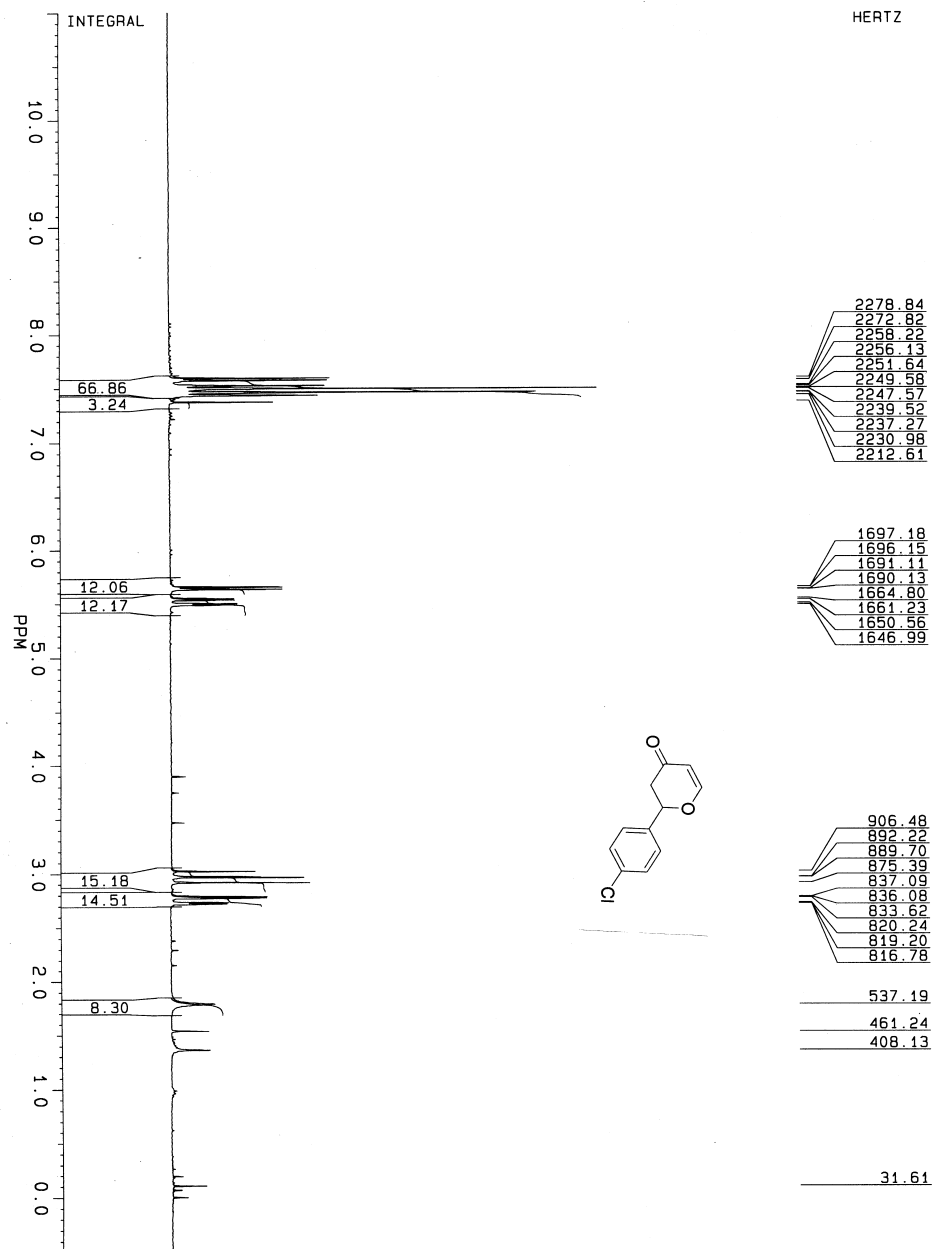
Info :

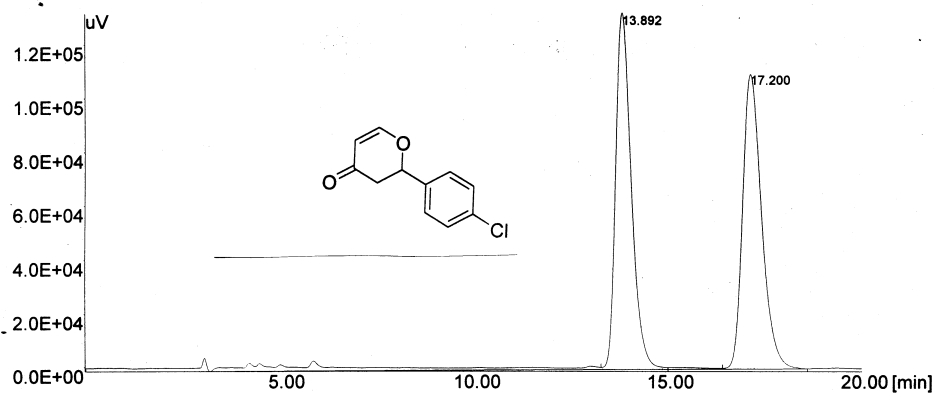
Vial # = 1 Rack # = 1  
Injection Date : 24-Apr-2002 8:55:24  
Curr. Date : 22-Oct-2002 10:10:16  
User : DU  
Group : DATA  
Control Method : COMBINEE

6j, entry 10 in Table 2  
ee: 94.5%

#	Name	RT	Area[v.Sec]	Quantity
1		15.458	148679.250	0.000
2		19.842	5372160.074	0.000

Total Area of Peak = 5520839.324 [v.Sec]





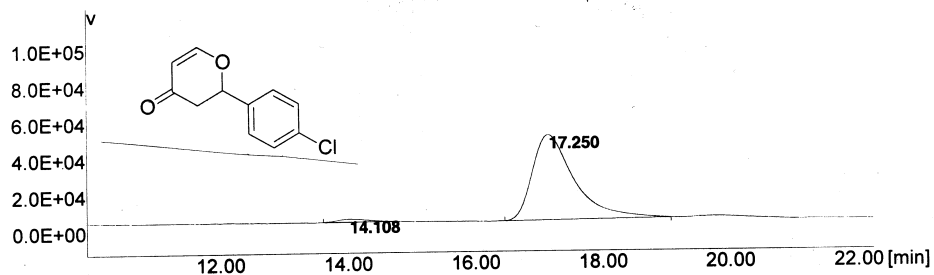
File name : p-cl-dihyprone014.CH2

Info :  
Chiralpak OD Column; 1.0mL/min; 90:10

Vial # = 1 Rack # = 1  
Injection Date : 10-Apr-2001 21:20:00  
Curr. Date : 10-Apr-2001 21:40:30  
User : DEFAULT  
Group : DATA  
Control Method : P-CL-DIHYPRONE

#	Name	RT	Area[uV.Sec]	Quantity
1		13.892	3736704.369	0.000
2		17.200	3727117.780	0.000

Total Area of Peak = 7463822.149 [uV.Sec]



File name : du-76-002.CH2

Info :

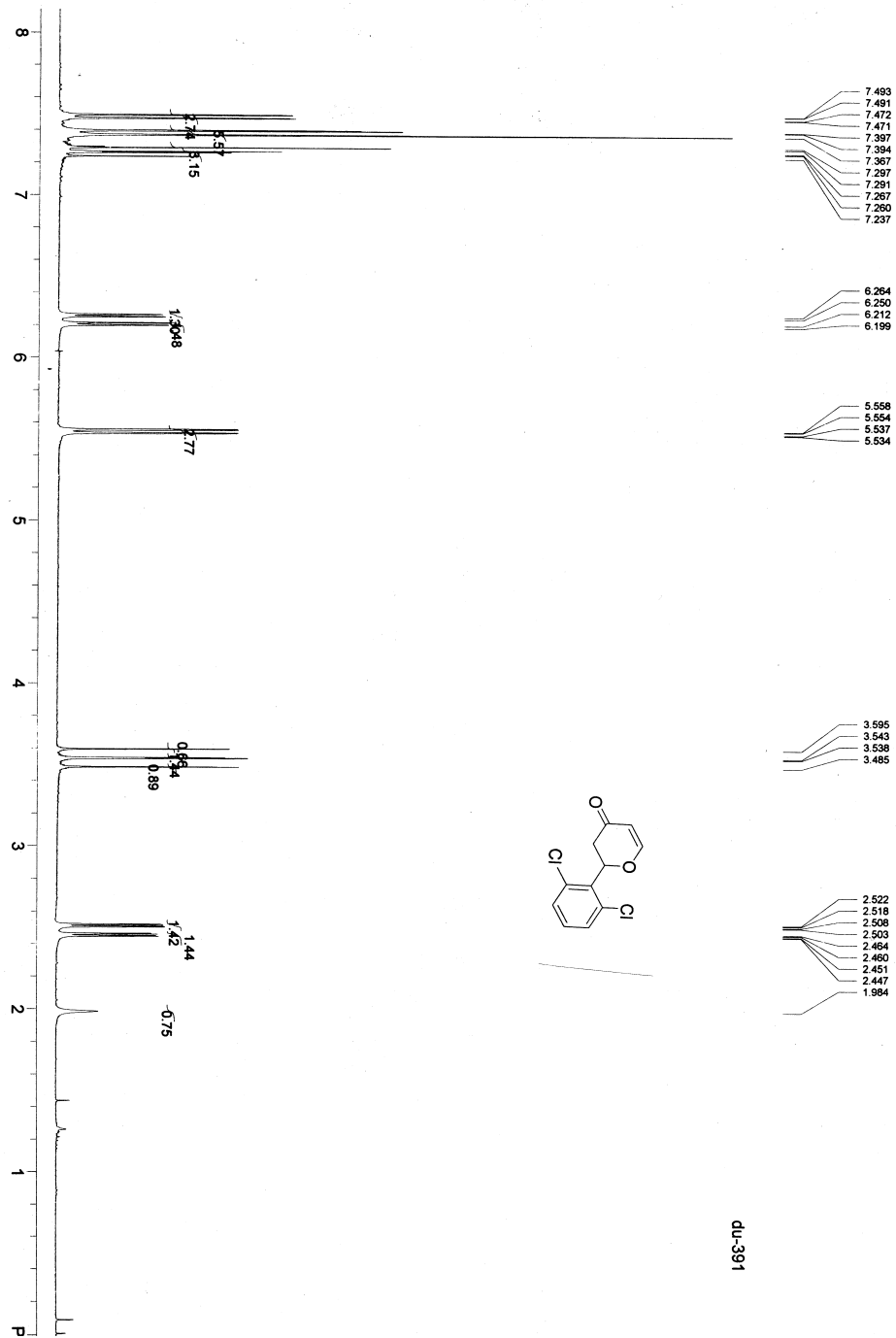
Vial # = 1 Rack # = 1  
Injection Date : 24-Apr-2002 9:21:00  
Curr. Date : 22-Oct-2002 10:11:30  
User : DU  
Group : DATA  
Control Method : COMBINEE

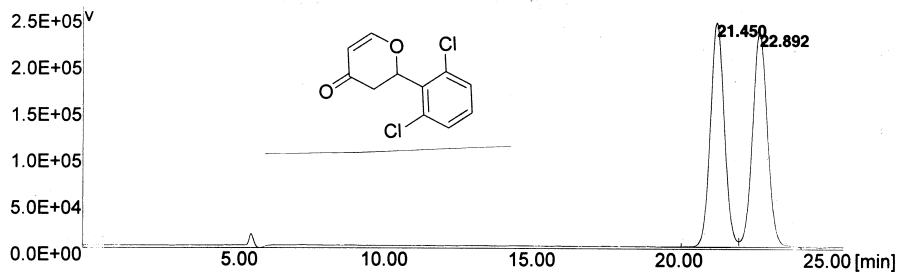
6K, entry 11 in Table 2

ee: 95.1%

#	Name	RT	Area[v.Sec]	Quantity
1		14.108	53363.250	0.000
2		17.250	2124338.000	0.000

Total Area of Peak = 2177701.250 [v.Sec]





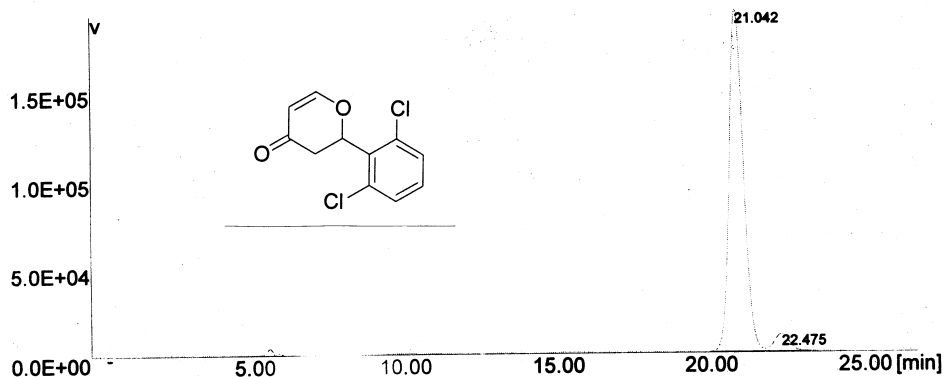
File name : dhfrac-dichloro-007.CH2

Info :

Vial # = 4 Rack # = 1  
 Injection Date : 21-Oct-2002 22:44:30  
 Curr. Date : 22-Oct-2002 10:03:16  
 User : DEFAULT  
 Group : DATA  
 Control Method : DICHLORO

#	Name	RT	Area[v.Sec]	Quantity
1		21.450	7882290.852	0.000
2		22.892	8015447.422	0.000

Total Area of Peak = 15897738.274 [v.Sec]



File name : du-86-027.CH2

Info :

Vial # = 1 Rack # = 1  
 Injection Date : 28-Apr-2002 14:28:36  
 Curr. Date : 28-Apr-2002 14:58:24  
 User : DU  
 Group : DATA  
 Control Method : DU-87-

#	Name	RT	Area[v.Sec]	Quantity
1		21.042	6104702.411	0.000
2		22.475	332542.089	0.000

Total Area of Peak = 6437244.500 [v.Sec]

*6L, entry 12 in Table 2*

*ee: 89.7%*