

Cinchona alkaloid ester derivatives as ligands in the asymmetric dihydroxylation and aminohydroxylation of alkenes

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Chiral HPLC analysis of diols:

(1) **1,2-Diphenyl-1,2-ethanediol.** Daicel Chiralcel OJ-H. hexane/ⁱPrOH=90/10. flow rate=0.5 ml/min. $t_R(\text{min})=28.1$ (major), 31.3 (minor);

(2) **Ethyl-2,3-Dihydroxy-3-phenylpropionate.** Daicel Chiralcel OJ-H. hexane/ⁱPrOH =90/10. flow rate=1.0ml/min. $t_R(\text{min})=15.1$ (major), 19.8 (minor);

(3) **Methyl-2,3-Dihydroxy-3-phenylpropionate.** Daicel Chiralcel OJ-H. hexane/ⁱPrOH =90/10. flow rate=1.0ml/min. $t_R(\text{min})=23.7$ (major), 31.1 (minor);

(4) **1-phenyl-1,2-propanediol.** Daicel Chiralcel OD-H. hexane/ⁱPrOH=97/3. flow rate=0.8 ml/min. $t_R(\text{min})=31.4$ (minor), 33.8(major);

(5) **2-phenyl-1,2-propanediol.** Daicel Chiralcel OJ-H. hexane/ⁱPrOH=90/10. flow rate=1.0 ml/min. $t_R(\text{min})=10.5$ (major), 13.6(minor);

(6) **1-phenyl-1,2-ethanediol.** Daicel Chiralcel OB-H. hexane/ⁱPrOH=90/10. flow rate=0.5 ml/min. $t_R(\text{min})=15.3$ (minor), 18.4 (major);

(7) **3-(α -naphthoxy)-1,2-propanediol.** Daicel Chiralcel OD-H. hexane/ⁱPrOH =90/10. flow rate=1.0ml/min. $t_R(\text{min})=24.9$ (major), 30.7 (minor).

Chiral HPLC analysis of amino alcohols (table 2) :

(1) **The AA reaction product of styrene:** Daicel Chiralcel AD. hexane/ⁱPrOH =90/10 , flow rate=0.7 mL/min, $t_R(\text{min})=18.1$ (major), 25.3(minor);

(2) **The AA reaction product of 1-chloro-4-vinylbenzene:** Dalcel Chiralcel AD. hexane/ⁱPrOH =85/15 . flow rate=0.7 mL/min, $t_R(\text{min})= 16.6$ (major), 26.3(minor);

(3) **The AA reaction product of 2-vinylnaphthalene:** Daicel Chiralcel AD, hexane/ⁱPrOH =70/30 , flow rate=0.7 mL/min , $t_R(\text{min})= 10.5$ (major), 16.1(minor);

(4) **The AA reaction product of ethyl cinnamate:** Dalcel Chiralcel OD-H, hexane/ⁱPrOH =85/15 , flow rate=0.6 mL/min , $t_R(\text{min})= 15.9$ (major);

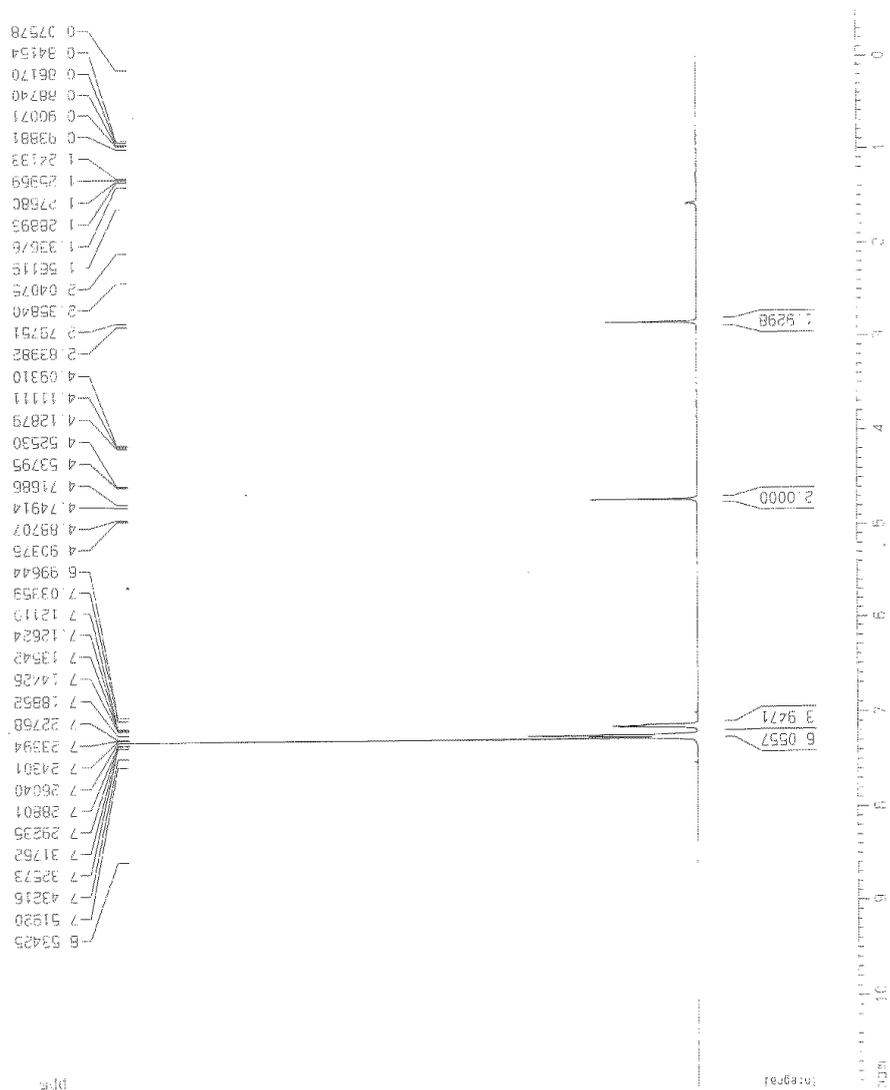
(5) **The AA reaction product of propyl cinnamate:** Daicel Chiralcel OD-H, hexane/ⁱPrOH =82/18 . flow rate=0.4 mL/min , $t_R(\text{min})= 20.5$ (minor), 22.8(major).

Current Data Parameters
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 EXPRN: 10
 PROCNO: 1

F2 - Acquisition Parameters
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 PROBNM: 5 mm BBI 1H-600
 PULPROG: zgpg30
 TO: 32768
 SOLVENT: CDCl3
 NS: 16
 DS: 0
 SWH: 8228.146 Hz
 FIDRES: 0.252629 Hz
 AQ: 1.9753372 sec
 RG: 161.3
 DW: 66.400 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 1.0000000 sec
 NUC1: 1H
 P1: 5.40 usec
 PL1: 4.00 dB
 SFO1: 400.1324710 MHz

F2 - Processing parameters
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 SF: 400.1300051 MHz
 MDW: EH
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR Plot parameters
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 CY: 10.67 cm
 F1P: 11.000 ppm
 F1: 4.80143 Hz
 F2P: 0.500 ppm
 F2: -200.07 Hz
 AFMCM: 0.87500 ppm/cm
 HZCM: 238.07475 Hz/cm



(1) 1,2-Diphenyl-1,2-ethanediol ¹H NMR
 (Table 1, entry 1)

```

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PROCNO       1

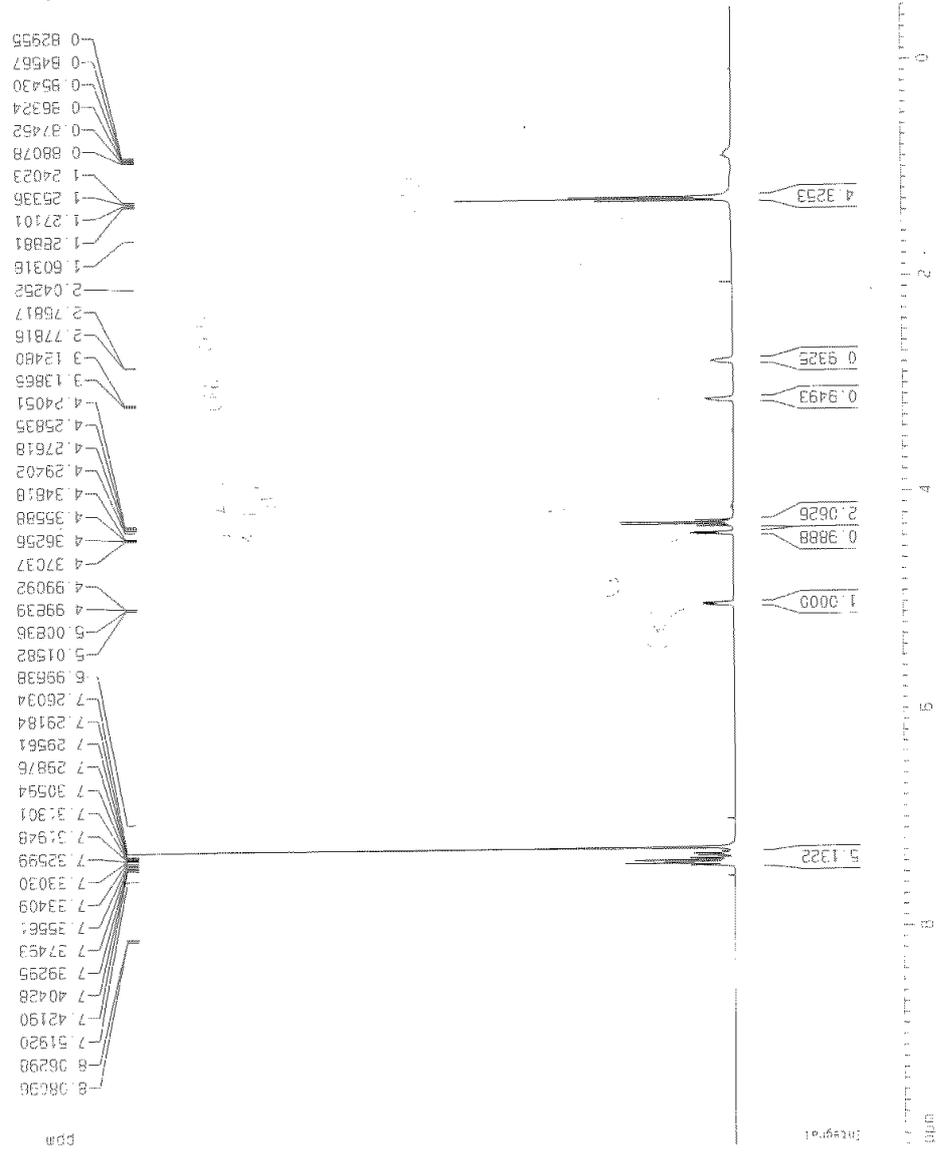
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SOLVENT     CDCl3
NS           16
DS           0
SWH          8278.146 Hz
FIDRES       0.252629 Hz
AQ           1.9792372 sec
RG           128
DW           60.400 usec
DE           6.90 usec
TE           300.0 K
D1           1.00000000 sec

***** CHANNEL f1 *****
NUC1         1H
P1           5.40 usec
PL1          -4.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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SF           400.1300091 MHz
WDW          EM
SSB          0
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GB           0
PC           1.00

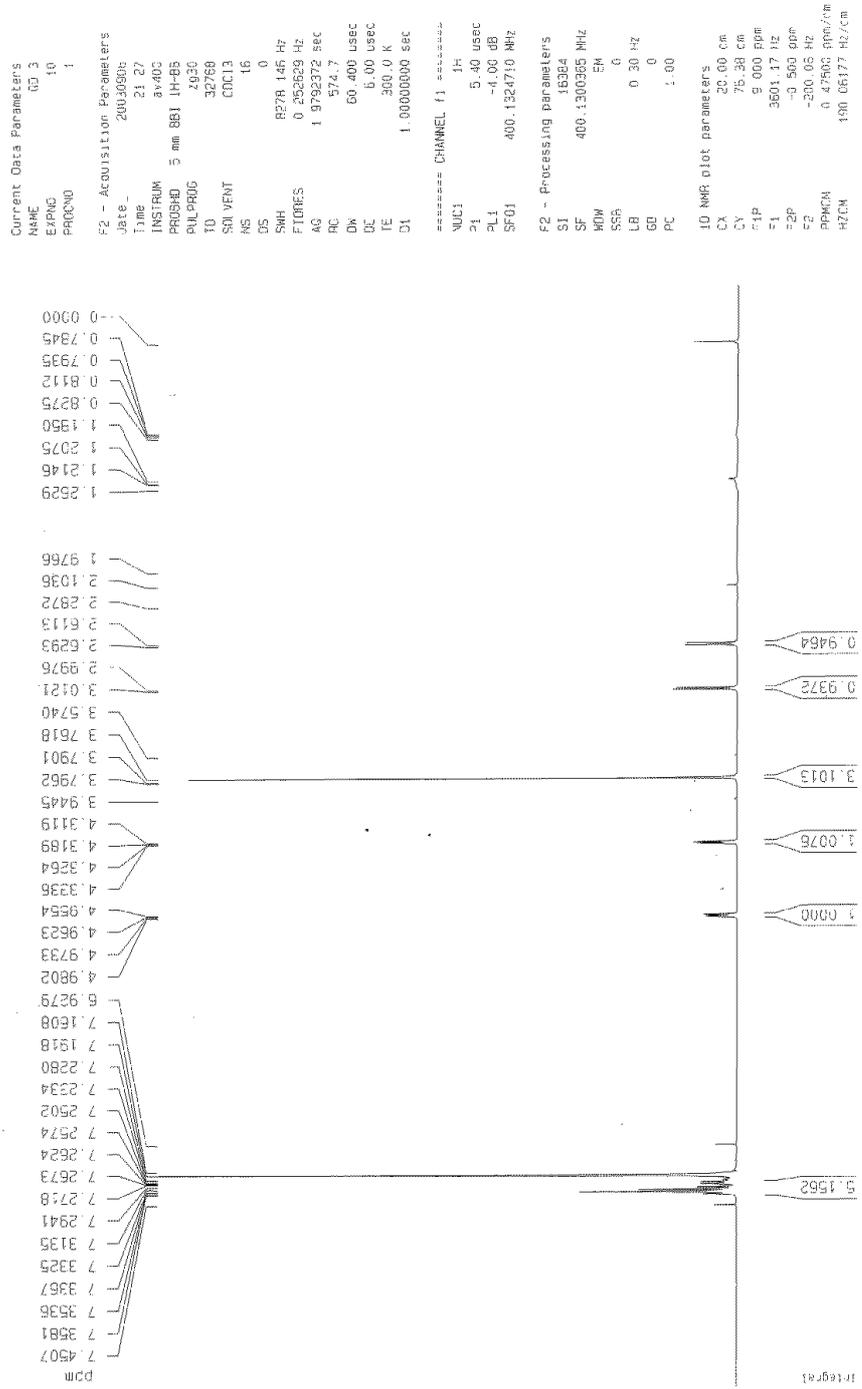
1D NMR plot parameters
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CY           23.11 cm
F1P          10.000 ppm
F2           4001.300 Hz
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ZD           -200.00 Hz
P0           0.52500 ppm/cm
SFO          210.06825 Hz/cm

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(2) Ethyl-2,3-Dihydroxy-3-phenylpropionate. ¹H NMR
(Table1, entry 2)

(3) Methyl-2,3-Dihydroxy-3-phenylpropionate ¹H NMR
 (Table1, entry 3)



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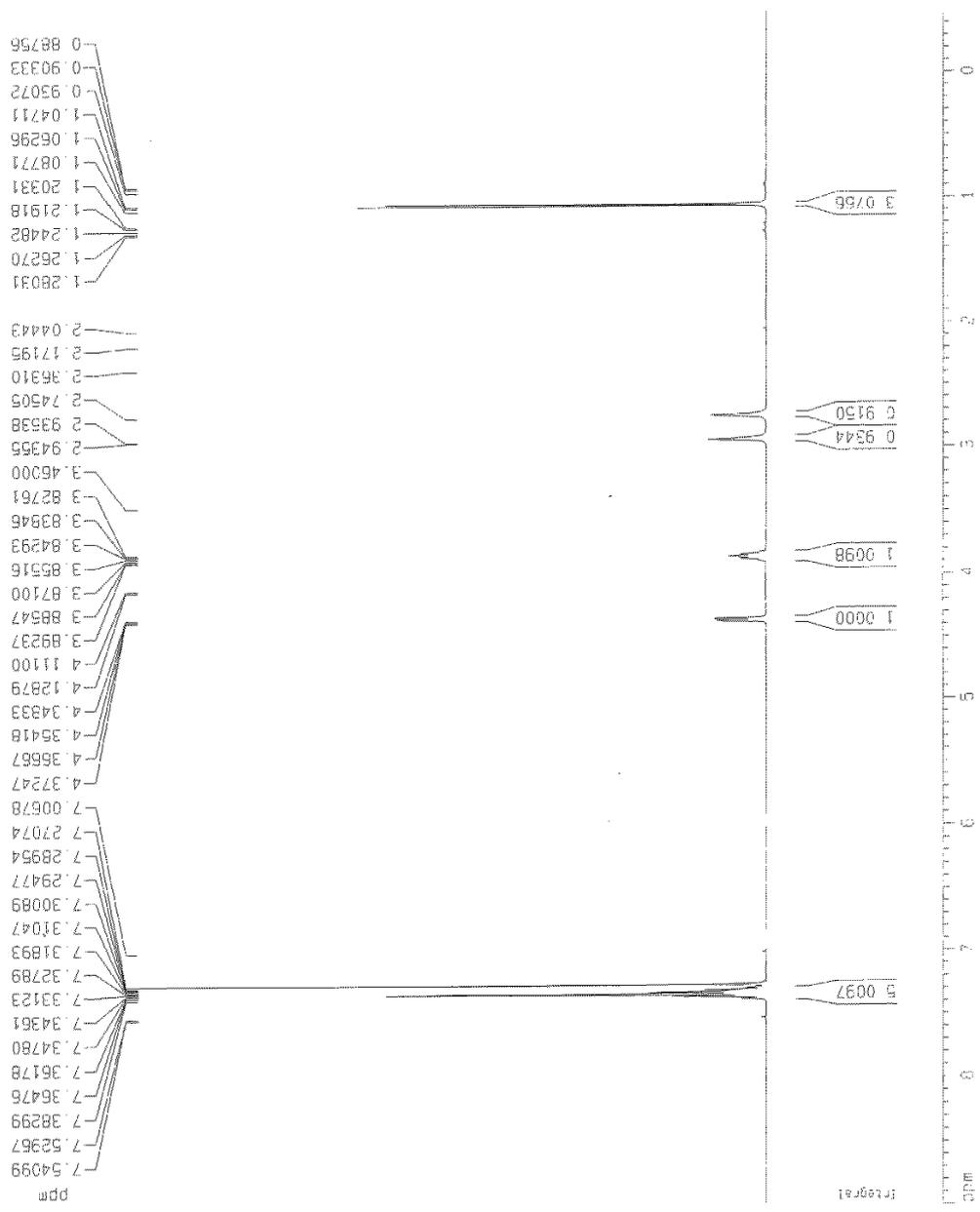
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PULPROG  zgpg
TO       32765
SOLVENT  CDCl3
NS       16
DS       0
SWH      8278.146 Hz
FIDRES   0.252629 Hz
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RG       203.2
AQ       60.400 usec
DC       5.00 usec
TE       300.0 K
C1       1.00000000 sec

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GB       0
PC       1.00

1D NMR plot parameters
CX       20.00 cm
CY       12.50 cm
F1P      9.000 ppm
F1       360.17 Hz
F2P      -0.500 ppm
F2       -203.05 Hz
PPMCH   0.47500 ppm/cm
HZCM    190.06175 Hz/cm

```



(4) 1-phenyl-1,2-propanediol ¹H NMR
(Table1, entry4)

```

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PROCNO   1

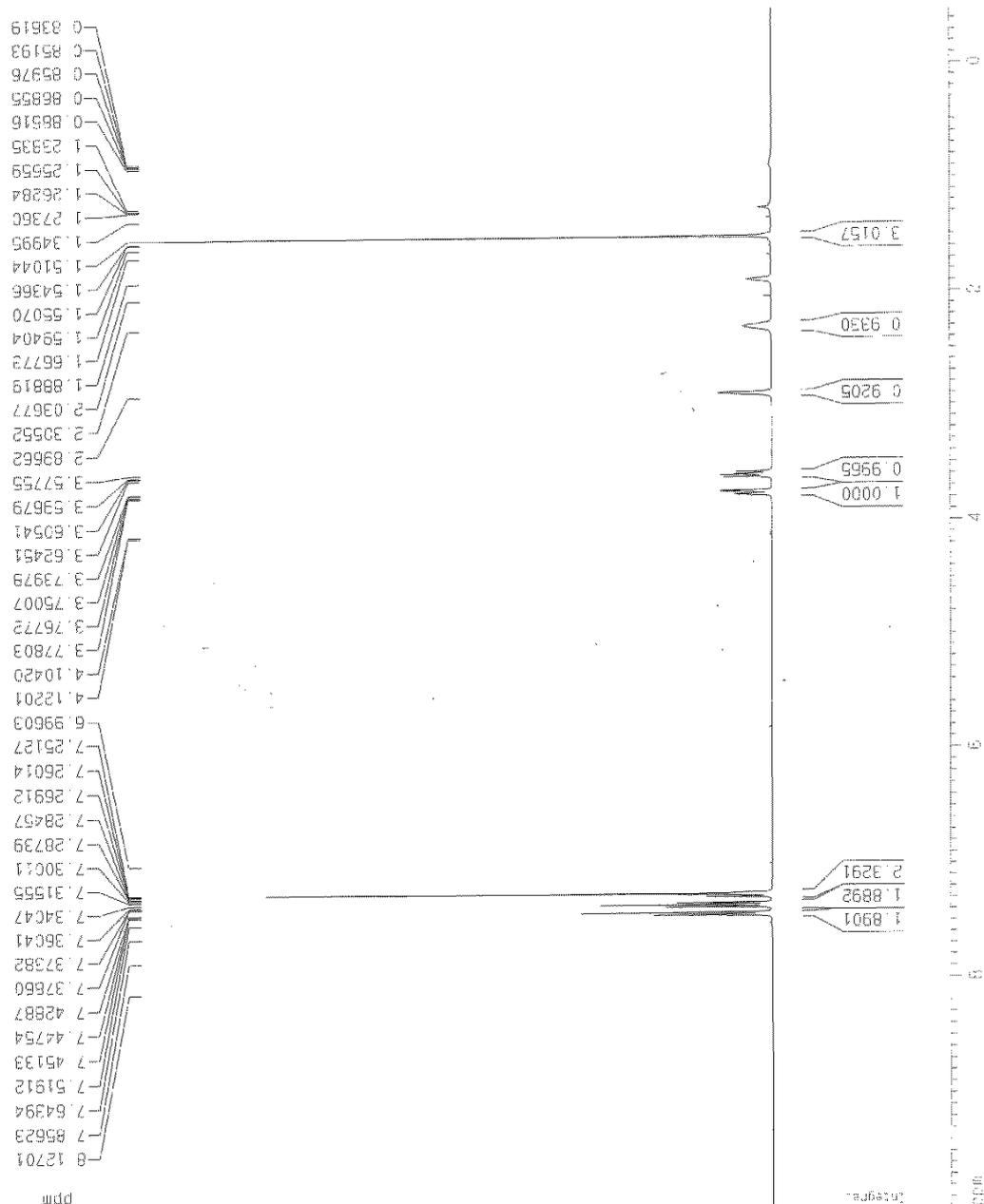
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PULPROG  zg30
TD       32768
SOLVENT  CDCl3
NS       16
DS       6
SWH      6278.146 Hz
FIDRES   0.252639 Hz
AQ       1.9792372 sec
RG       50.8
DM       60.400 user
DE       6.00 user
TE       300.0 K
D1       1.0000000 sec

===== CHANNEL f1 =====
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PL1     -4.00 dB
SFO1    400.1324710 MHz

F2 - Processing parameters
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SF       400.1300991 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00

1D NMR plot parameters
CX       20.00 cm
CY       11.81 cm
F1P      10.000 cm
F1       4001.30 Hz
F2P      -0.500 ppm
F2       -200.90 Hz
PQACHI   0.52500 ppm/cm
HZCM     210.06295 Hz/cm

```



(5) 2-phenyl-1,2-propanediol ¹H NMR
(Table1, entry 5)


```

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PROCNO       1

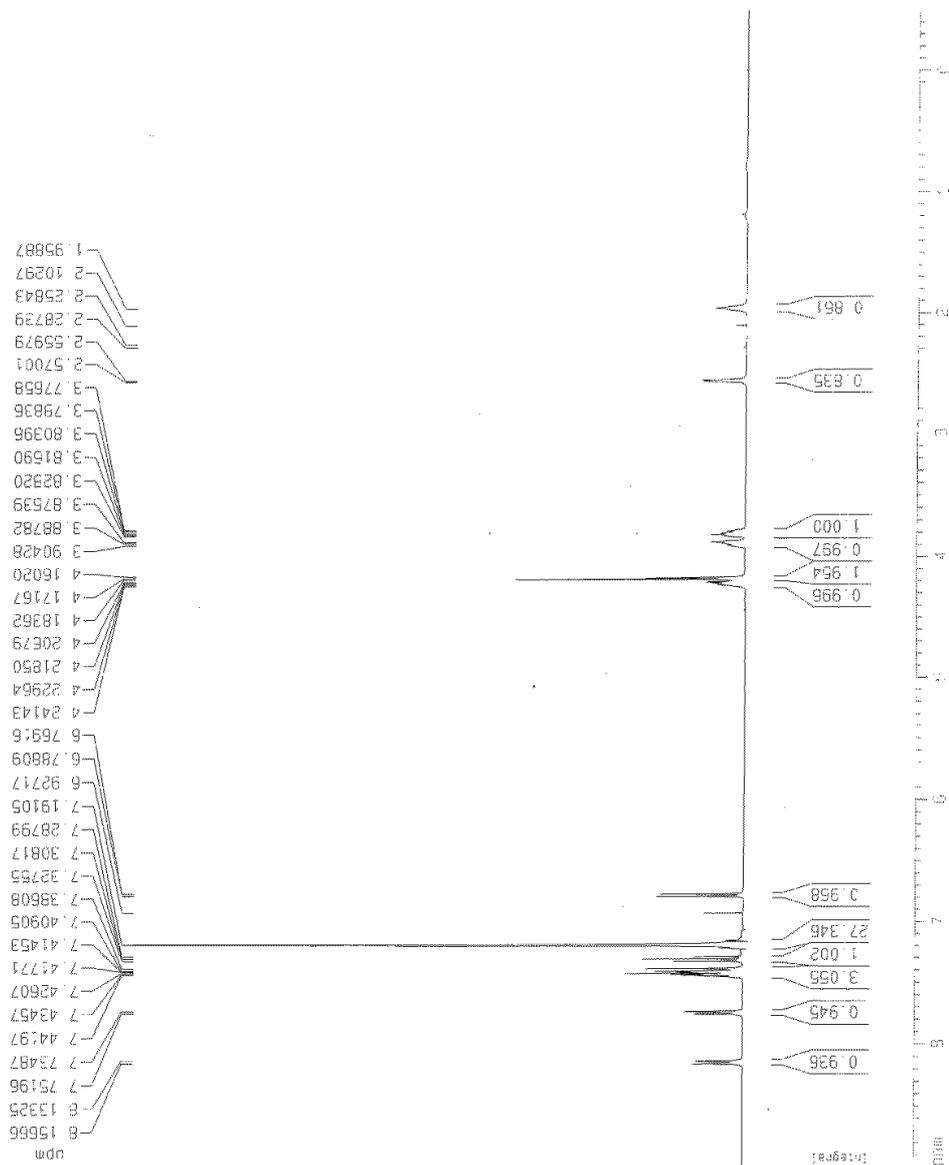
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TD            32768
SOLVENT      CDCl3
NS            16
DS            0
SWH           8278.146 Hz
FIDRES       0.256524 Hz
AQ           1.9792372 sec
RG           912.3
DE           60.400 usec
TE           300.2 K
D1           1.00000000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           5.40 usec
PL1         -4.00 dB
SF01        400.1324710 MHz

F2 - Processing parameters:
SI           16384
SF           400.1300365 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00

1D NMR plot parameters
CX           20.00 CH
CY           134.65 CH
F1P          9.000 DDM
F2P          -0.500 DDM
F2           -200.00 Hz
MPCOM       0.47500 DDM/CH
HZCM        190.06177 Hz/CM

```



(7) 3-(α -naphthoxy)-1,2-propanediol ^1H NMR
(Table1, entry 7)

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PROCNO       1

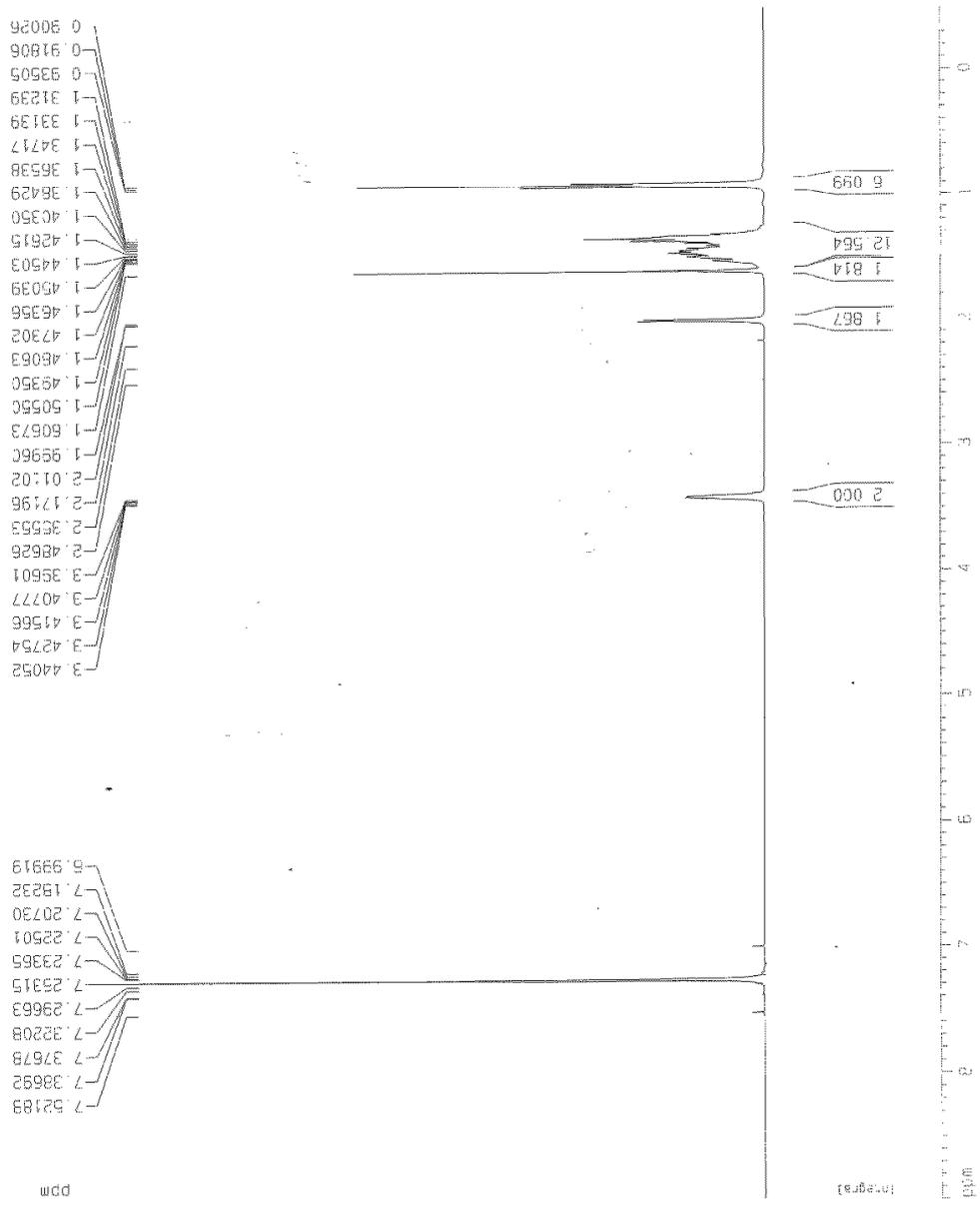
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PULPROG      zg30
TD           32768
SOLVENT      CDCl3
NS           16
DS           0
SWH          8278.146 Hz
FIDRES       0.262629 Hz
AQ           1.9792372 sec
RG           128
DM           60.400 usec
DE           6.00 usec
TE           300.0 K
SI           1
SF           1000000000.000 sec

===== CHANNEL f1 =====
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PL1         -4.00 dB
SFO1        400.1324710 MHz

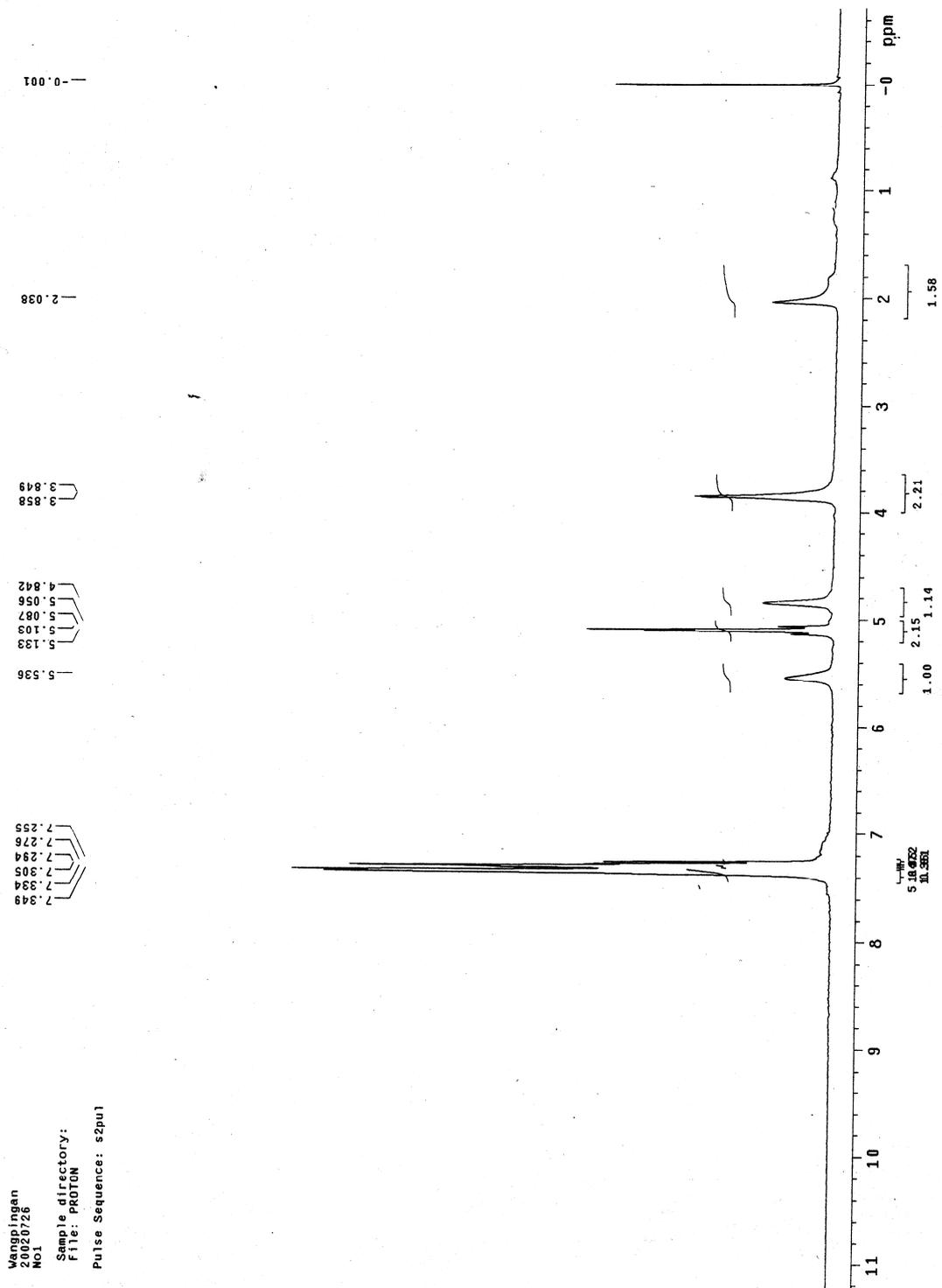
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SSB         0
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GB         0
PC          1.00

1D NMR plot parameters
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CY          37.83 cm
F1P         9.000 ppm
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F2P         -0.500 ppm
F2          -200.06 Hz
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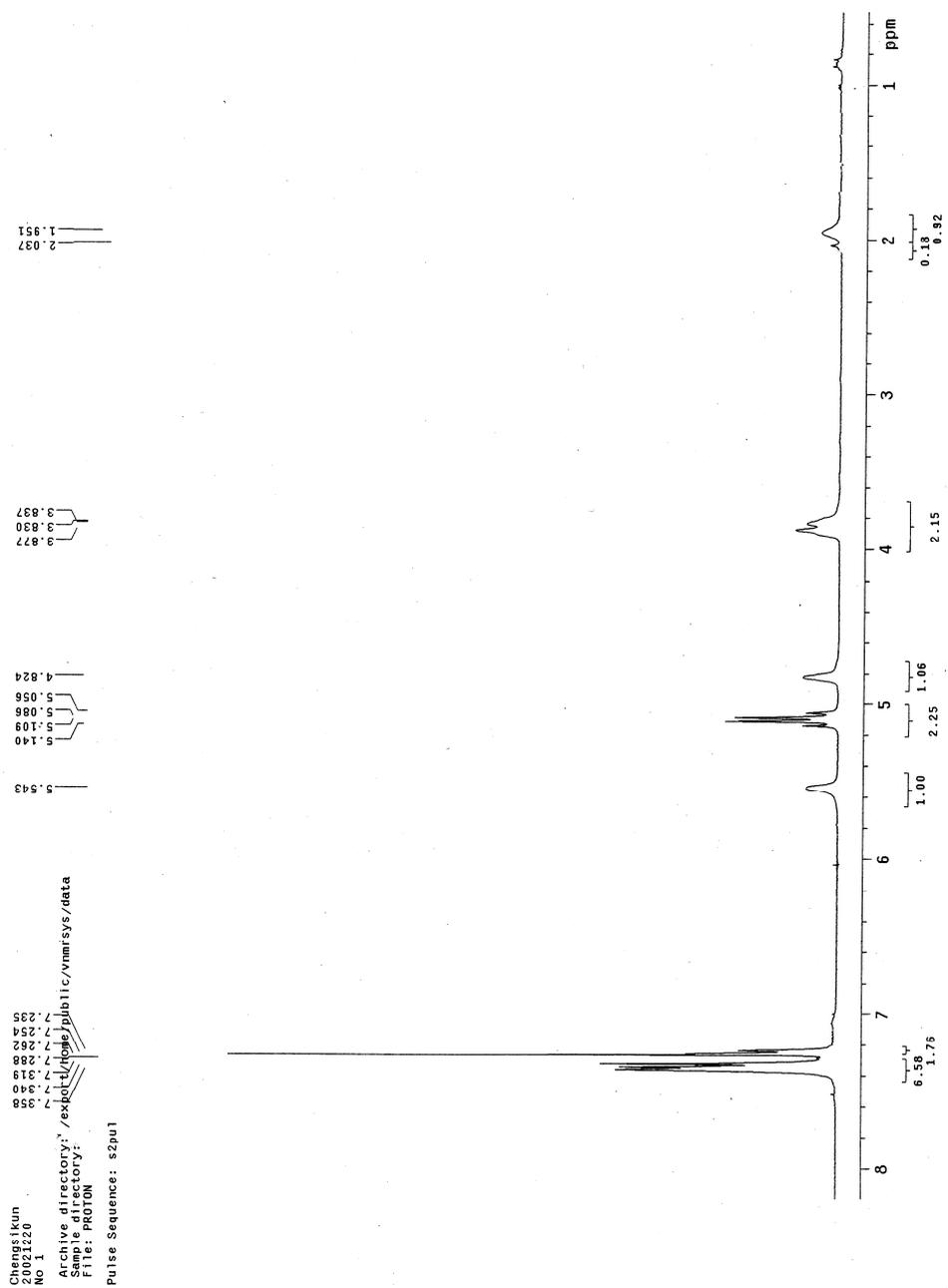
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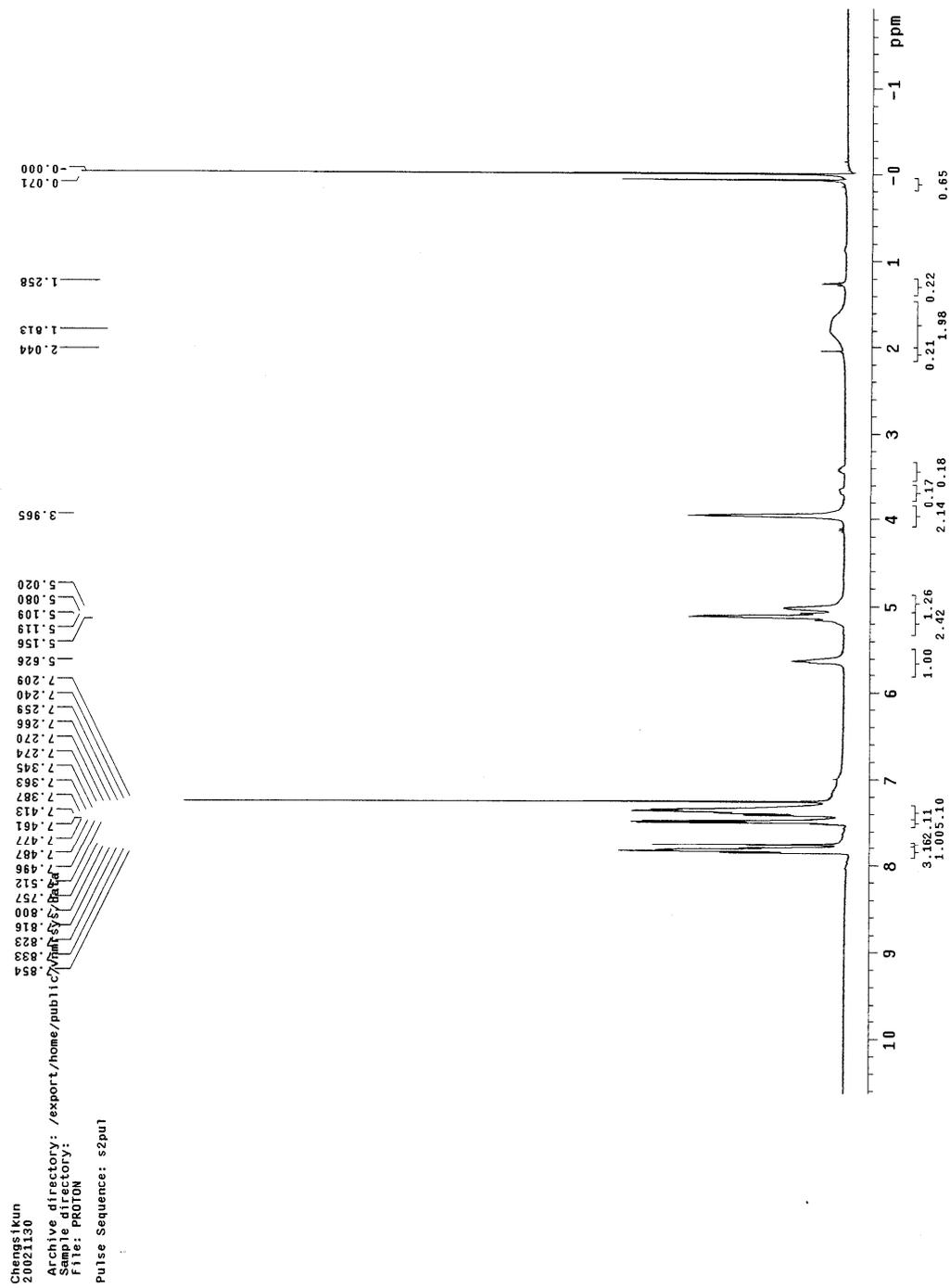
(8) decane-5,6-diol ¹H NMR
(Table1, entry 8)



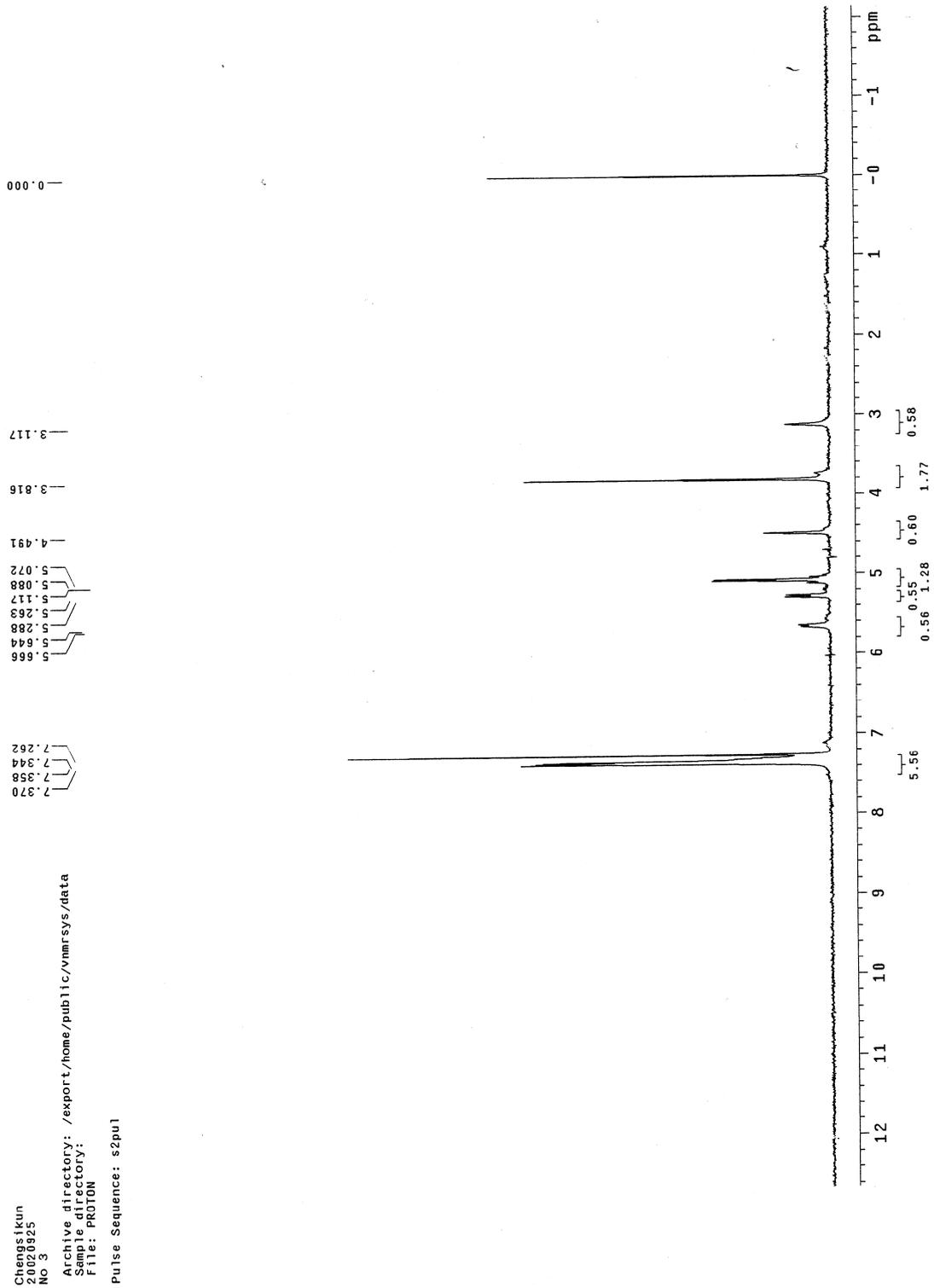
(9) The AA reaction product of styrene ^1H NMR
 (Table2, entry 1)



(10) The AA reaction product of 1-chloro-4-vinylbenzene ^1H NMR
 (Table2, entry 2)



(11) The AA reaction product of 2-vinylnaphthalene ¹H NMR
 (Table2, entry 3)



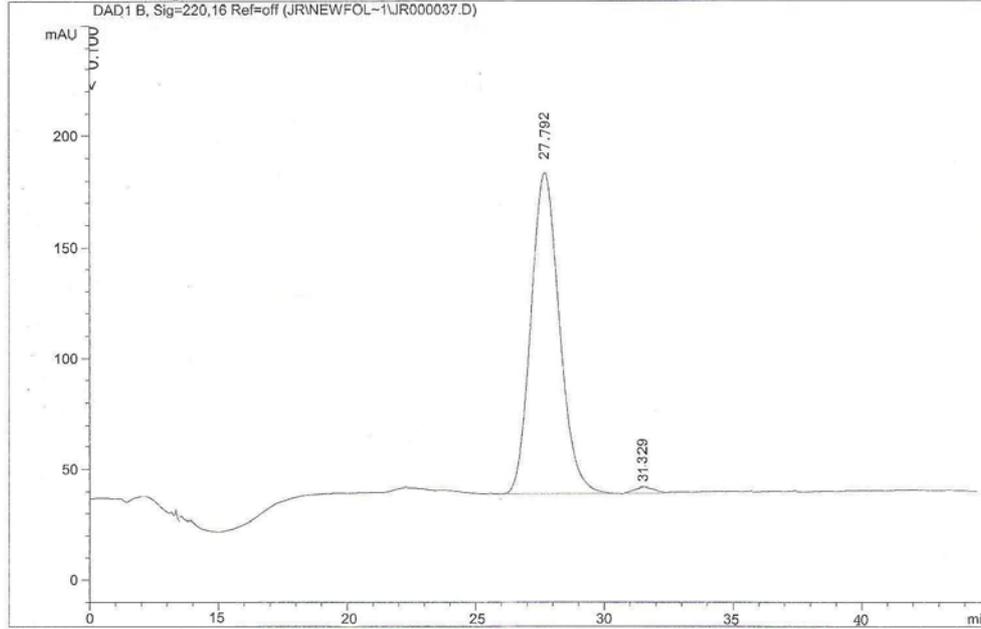
(12) The AA reaction product of ethyl cinnamate ¹H NMR
Table2, entry

OJ colum hex:iPROH=90:10flow: 0.5ml/min

```

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Acq. Instrument: Instrument 1
Acq. Method    : C:\HPCHEM\1\JR-AD.M
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                (modified after loading)
Analysis Method: D:\HPCHEM\1\METHODS\JR.M
Last changed   : 9/15/2009 4:03:03 PM by ly
                (modified after loading)
    
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method for analysis



Area Percent Report

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Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
    
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Signal 1: DAD1 B, Sig=220,16 Ref=off

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1	27.792	BB	1.1756	1.11281e4	144.31137	98.5754
2	31.329	BP	0.7011	160.81828	2.91759	1.4246

Totals : 1.12889e4 147.22896

Results obtained with enhanced integrator!

Summed Peaks Report

Signal 1: DAD1 B, Sig=220,16 Ref=off

HPLC (1,2-Diphenyl-1,2-ethanediol)

OJ colum hex:iPrOH=90:10 flow: 1.0ml/min

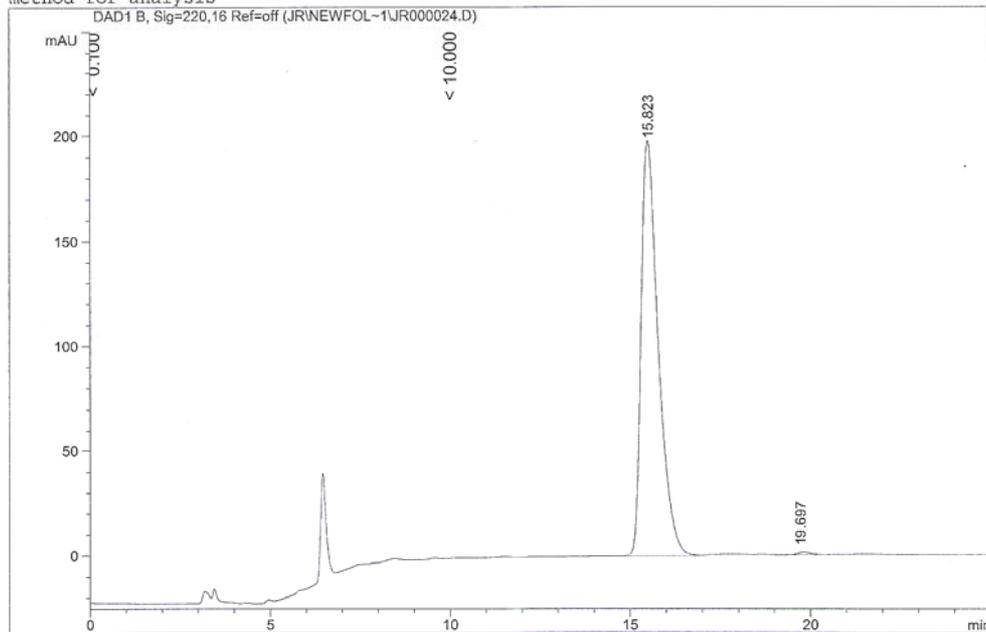
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Acq. Instrument : Instrument 1
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Last changed   : 9/15/2009 4:05:32 PM by ly
                (modified after loading)

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method for analysis

DAD1 B, Sig=220,16 Ref=off (JRNEWFOL-1JR000024.D)



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Area Percent Report
=====

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Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000

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Signal 1: DAD1 B, Sig=220,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.823	BB	0.5070	6591.54541	197.66968	99.4995
2	19.697	BB	0.3560	33.15410	1.29786	0.5005

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Totals :                6624.69951  198.96754
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Results obtained with enhanced integrator!

```

=====
Summed Peaks Report
=====

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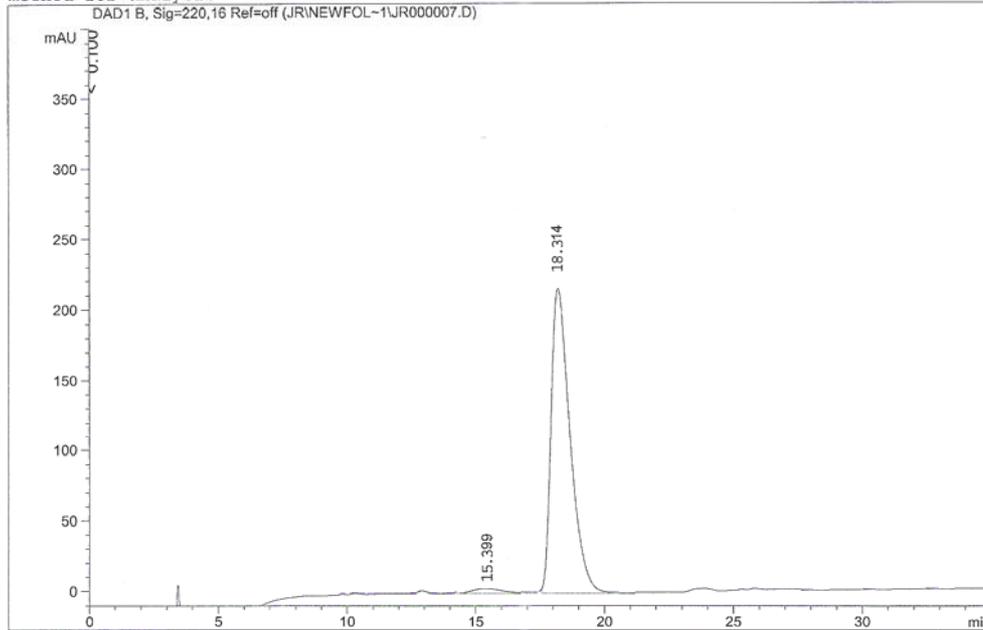
Signal 1: DAD1 B, Sig=220,16 Ref=off

HPLC (Ethyl-2,3-Dihydroxy-3-phenylpropionate)

OB colum hex:iPROH=90:10 flow: 0.5 ml/min

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Acq. Instrument : Instrument 1
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Last changed : 9/13/2009 11:41:10 AM by ly
(modified after loading)

method for analysis



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Area Percent Report
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Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=220,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.399	BB	1.2783	250.11389	3.09659	2.2331
2	18.314	BBA	0.7839	1.09504e4	215.83185	97.7669

Totals : 1.12005e4 218.92844

Results obtained with enhanced integrator!

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Summed Peaks Report
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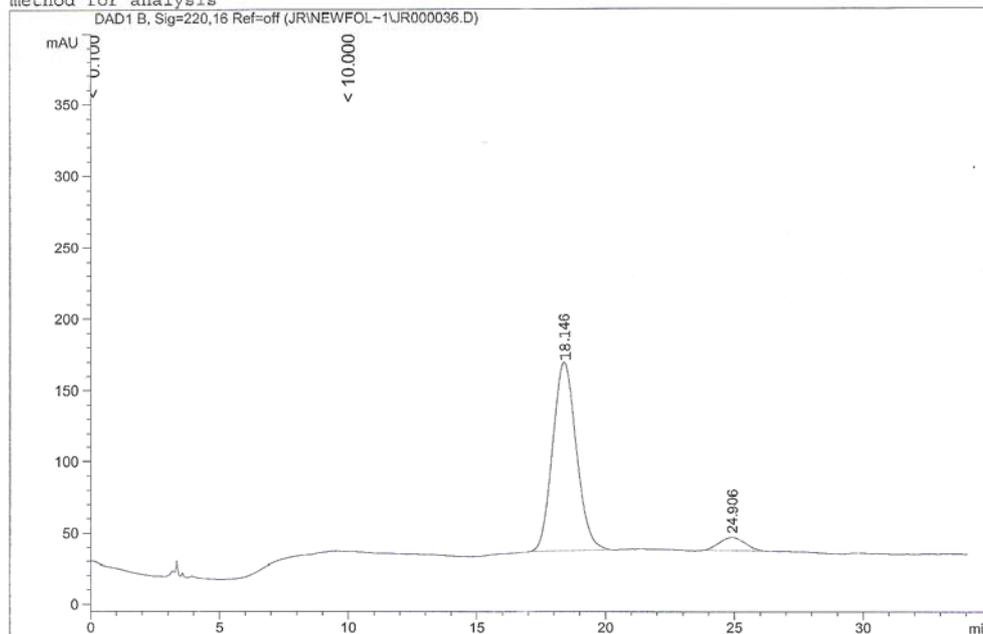
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HPLC (1-phenyl-1,2-ethanediol. Daicel)

AD column hex:iPrOH=90:10flow: 0.7ml/min

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Last changed   : 9/13/2009 11:10:28 AM by ly  
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method for analysis

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Area Percent Report
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Dilution       :      1.0000
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Signal 1: DAD1 B, Sig=220,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
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2	24.906	BB	0.9602	646.06561	9.37227	7.1198

Totals : 9074.14862 141.75049

Results obtained with enhanced integrator!

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Summed Peaks Report
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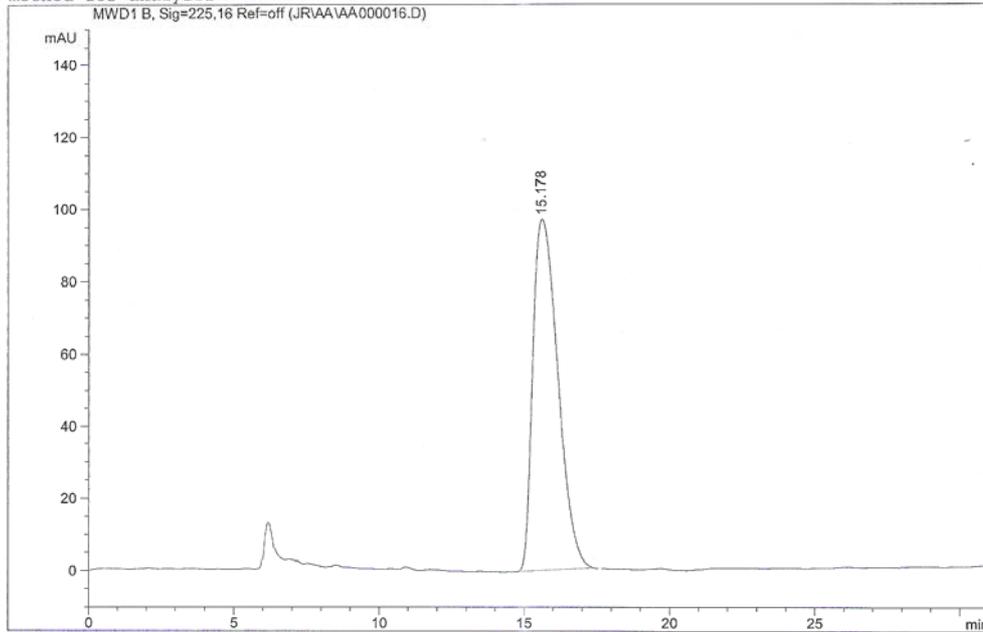
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HPLC(the AA reaction product of styrene)

Hex:iPr-OH=85:15, flow=0.6ml/min, CHIRALCEL OD-H

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Analysis Method : D:\HPCHEM\1\METHODS\JR.M
Last changed : 8/14/2009 11:33:01 AM by LY
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method for analysis



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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MWD1 B, Sig=225,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.178	BB	0.9546	5798.08691	96.82544	100.0000

Totals : 5798.08691 96.82544

Results obtained with enhanced integrator!

=====
Summed Peaks Report
=====

Signal 1: MWD1 B, Sig=225,16 Ref=off

HPLC(the AA reaction product of ethyl cinnamate)