

Ms No. JA981869

Supporting Information

Revised 9-2-98

Materials and methods.

All reagents were used as supplied commercially unless otherwise noted. THF and diethyl ether were distilled from sodium under N_2 before use. CH_2Cl_2 was distilled under N_2 from P_2O_5 . CH_3CN was dried with P_2O_5 and distilled from K_2CO_3 . 2,2'-dimethoxy-1,1'-binaphthyl-3,3'-dicarboxylic acid was prepared according to literature method.[†] 1H NMR spectra were performed on a Varian XL-400 or XL-500 Spectrometer in the solvent specified, referenced to the residual proton signals. Routine mass spectra were obtained from the University of California, San Francisco Mass Spectrometry Facility. UV-visible spectra were recorded on a Hewlett-Packard 8452 Diode Array Spectrometer. Epoxide formation in olefin epoxidations was analyzed on a Hewlett-Packard 5890 Gas Chromatograph equipped with a DB-5 capillary column (0.32 mm \times 25 M, i.d., 0.25 μ m, J&W Scientific). Enantiomeric excesses were determined by chiral GLC with a Cyclodex-B-PH chiral capillary column (0.26mm \times 30 M, i.d., 0.25 μ m, J&W Scientific).

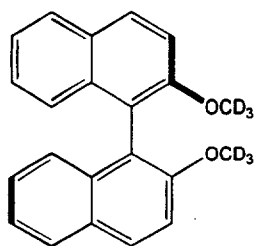
General Procedure for Asymmetric Olefin Epoxidation.

Catalyst **1** (1 μ mol), dodecane (25 μ L, as a GC standard) and an olefin (1.0 mmol) were dissolved in freshly distilled CH_2Cl_2 (2 mL) in a Shlenk tube. The mixture was deaerated by evacuation and refilling N_2 for several times. With stirring and under a N_2 flow, $PhIO$ (0.10 mmol) was added in one portion. The reaction mixture was stirred at rt and monitored by GC periodically. Each aliquot (ca. 10 μ L) was passed through a short pad of silica gel (0.5 cm) and eluted with CH_2Cl_2 (200 μ L). The elute was collected and injected on GC for analysis. At the end of the reaction, CH_2Cl_2 was removed under reduced pressure and the reaction mixture was loaded on a short silica gel column (1 \times 5 cm). PhI and unreacted olefins were eluted with pentane. Epoxides were eluted with 20% ether/pentane. The ee's of the epoxides were determined by GC on a Cyclodex-B chiral stationary phase column. The GC conditions are as follows: (epoxide, oven temperature,

[†] Naruta, Y.; Tani, F.; Ishibara, N.; Maruyama, K. *J. Am. Chem. Soc.* **1991**, *113*, 6865.

retention time with the absolute configuration): (styrene oxide, 95°C, 14.3 min (R), 14.7 min (S); pentafluorostyrene oxide, 70°C, 32.3 min (R), 32.9 min (S); *m*-chlorostyrene oxide, 95°C, 44.2 min (R), 44.7 min (S); *o*-nitrostyrene oxide, 125°C, 41.4 min (R), 41.9 min (S); *m*-nitrostyrene oxide, 120°C, 107.7 min (R), 109.0 min (S); 1,2-dihydronaphthalene oxide, 95°C, 31.3 min (1S, 2R), 32.7 min (1R, 2S); *cis*- β -methylstyrene oxide, 105°C, 13.6 min (1R, 2S), 14.1 min (1S, 2R); *t*-butyloxirane, 25°C, 15.6 min, 15.9 min; trimethylsilyloxirane, 35°C, 12.2 min, 12.6 min.

Preparation of (R)-binaphthyl di-CD₃ ether



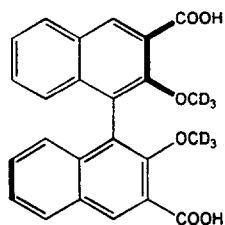
(R)-(+)-2,2'-Binaphthol (572 mg, 2.0 mmol) and CD₃I (870 mg, 6.0 mmol) were dissolved in anhydrous MeCN (20 mL, dried and distilled from K₂CO₃). To the solution was added in one portion dry Cs₂CO₃ (1.96 g, 6.0 mmol). The mixture was stirred at room temperature under an atmosphere of N₂ for 24h. Ether (100 mL) was added and the mixture was washed with saturated NaCl solution (20 mL \times 3). The ether layer was separated and dried over MgSO₄. After evaporation of the solvent, the crude product was purified by passing through a short silica gel column (eluent: CH₂Cl₂/hexane = 1/1). Yield 627 mg (98%).

¹H NMR (400 MHz/CDCl₃) 7.97(d, J = 8.9 Hz, 2H), 7.86(d, J = 8.1 Hz, 2H), 7.45(d, J = 8.9 Hz, 2H), 7.31(dd, J = 8.1, 7.0 Hz, 2H), 7.20(dd, J = 8.3, 7.0 Hz, 2H), 7.10(d, J = 8.3 Hz, 2H).

¹³C NMR (400 MHz/CDCl₃) 154.93, 133.97, 129.38, 129.16, 127.91, 126.30, 125.22, 123.49, 119.52, 114.18.

MS: m/e = 323.2 (M+H)⁺ for C₂₂H₁₂D₆O₂ (LSIMS⁺).

Preparation of CD₃O-substituted binaphthyl diacid



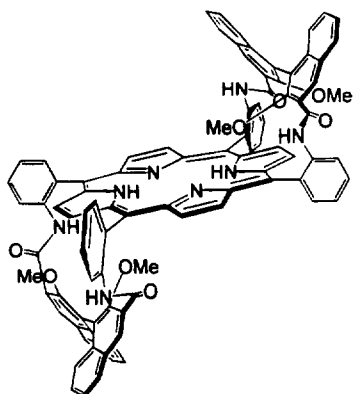
A 200 mL three-necked flask was fitted with a reflux condenser, a N₂ inlet and gas outlet (on top of the condenser) connected to a bubbler. The system was flushed with dry N₂ and the walls of the glassware were dried with a small flame. Under a N₂ flow, the flask was charged with di-CD₃ ether (500 mg, 1.56 mmol), TMEDA (N, N, N', N'-tetramethylethylenediamine, 1.0 mL) and diethyl ether freshly distilled from sodium (100 mL). The mixture was cooled to 0°C by an ice-water bath. With stirring, n-BuLi (2.5M in hexane, 2.5 mL, ca. 6.2 mmol) was slowly added via a syringe. After the addition was complete, the mixture was warmed to room temperature and refluxed for 24h. This mixture was cooled to 0°C again and CO₂ (dried by passing through concentrated H₂SO₄) was bubbled through the solution for 1h. After stirring for another hour, ether was removed by rotary evaporation, water (100 mL) was added and the resulting mixture was washed with benzene (30 mL × 2) (The benzene layer may contain unreacted starting material if the metalation is not complete). The aqueous layer was then acidified with 5M HCl solution and extracted with CHCl₃ (50 mL × 3). The CHCl₃ extractions were combined and dried with Na₂SO₄. Removal of the solvent gave the crude product which was recrystallized from hot benzene. Yield 414 mg (65%).

¹H NMR (400 MHz/CDCl₃) 8.95(s, 2H), 8.07(d, J = 7.5 Hz, 2H), 7.54(t, J = 7.5 Hz, 2H), 7.45(t, J = 7.5 Hz, 2H), 7.15(d, J = 7.5 Hz, 2H).

¹³C NMR (400 MHz/CDCl₃) 166.52, 153.89, 143.80, 136.38, 136.14, 130.04, 129.95, 126.57, 125.31, 124.42, 121.36.

MS (m/e): 409.1 (M+H)⁺ for C₂₄H₁₂D₆O₆ (LSIMS⁺).

Preparation of ααββ bis-binaphthyl-strapped free base porphyrin



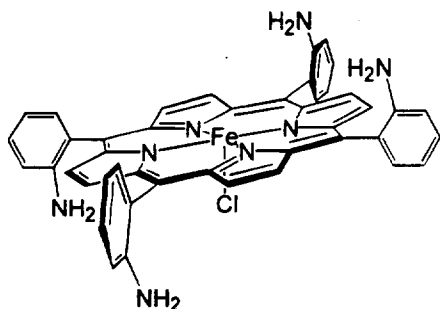
Binaphthyl diacid chloride was freshly prepared by refluxing 2,2'-dimethoxy-1,1'-binaphthyl-3,3'-dicarboxylic acid (45 mg, 0.11 mmol) with SOCl_2 for 8h and dried *in vacuo*. $\alpha\alpha\beta\beta$ -TAPP (tetrakis(*o*-aminophenyl)porphine) (34 mg, 0.05 mmol) and *N,N*-diethylaniline (45 mg, 0.30 mmol) were dissolved in anhydrous THF (25 mL, freshly distilled from sodium). Under a flow of dry N_2 , the solution was cooled to 0°C with an ice-water bath and a solution of the diacid chloride in THF (10 mL) was added by syringe pump over 1h. The mixture was allowed to stir at room temperature overnight. THF was removed by rotary evaporation and the residue was taken up in CH_2Cl_2 (100 mL) and washed with water (20 mL). After dried with Na_2SO_4 , the solution was reduced to dryness and the residue was subject to column chromatography on silica gel (eluent: $\text{CH}_3\text{OH}/\text{CH}_2\text{Cl}_2 = 1/99$) to give bis-binaphthyl-strapped porphyrin (42 mg, 60%).

^1H NMR (400 MHz/ CDCl_3) 11.85(s, 2H), 9.11(d, $J = 8.0$ Hz, 2H), 8.86-8.85(m, 6H), 8.69(s, 4H), 8.40(d, $J = 8.0$ Hz, 2H), 8.33(d, $J = 6.7$ Hz, 2H), 7.99(s, 2H), 7.95-7.85(m, 4H), 7.82(t, $J = 6.7$ Hz, 2H), 7.69(t, $J = 6.7$ Hz, 2H), 7.64(s, 2H), 7.34-7.20(m, 8H), 7.06(t, $J = 6.6$ Hz, 2H), 6.93(t, $J = 6.6$ Hz, 2H), 6.74(t, $J = 6.7$ Hz, 2H), 6.51(d, $J = 6.6$ Hz, 2H), 6.37(d, $J = 6.7$ Hz, 2H), 2.96(s, 6H), -0.65(s, 6H), -2.93(s, 2H).

MS: $m/e = 1407.5$ ($\text{M}+\text{H}$) $^+$ for $\text{C}_{92}\text{H}_{62}\text{N}_8\text{O}_8$ (LSIMS $^+$).

UV-vis (CH_2Cl_2): 418(soret), 510 nm.

Preparation of $\text{Fe}(\alpha\alpha\beta\beta\text{-TAPP})\text{Cl}$

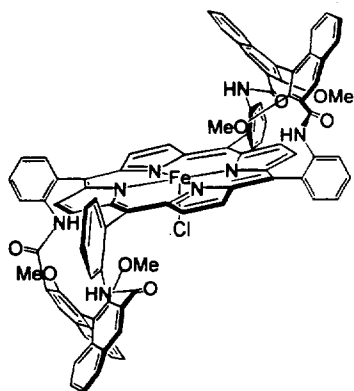


A mixture $\alpha\alpha\beta\beta$ -TAPP (67.4 mg, 0.10 mmol), FeBr_2 (200 mg, 0.93 mmol), 2,6-lutidine (0.20 mL) in benzene-THF solution (1:1, 50 mL) was stirred for 24h at room temperature in a dry box. The metalation was complete as indicated by UV-visible spectroscopy. This mixture was brought out and stirred under air for 3h. The solvents were removed under vacuum and the residue was dissolved in CH_2Cl_2 (200 mL) and filtered to remove iron salt. The CH_2Cl_2 layer was washed with aqueous NH_4Cl solution, dried over MgSO_4 and concentrated to give $\text{Fe}(\alpha\alpha\beta\beta\text{-TAPP})\text{Cl}$. The yield is quantitative.

MS: $m/e = 728.2 \text{ (M-Cl)}^+$ for $\text{C}_{44}\text{H}_{32}\text{ClFeN}_8$ (LSIMS⁺).

UV-vis (CH_2Cl_2): 370, 416, 510, 580 nm.

Preparation of $\alpha\alpha\beta\beta$ bis-binaphthyl-strapped Fe porphyrin (1)



Binaphthyl diacid chloride was freshly prepared by refluxing the diacid (45 mg, 0.11 mmol) with SOCl_2 for 8h and dried *in vacuo*. $\text{Fe}(\alpha\alpha\beta\beta\text{-TAPP})\text{Cl}$ (35 mg, 0.05 mmol) and N, N-diethylaniline (45 mg, 0.30 mmol) were dissolved in anhydrous THF (25 mL, freshly distilled from sodium). Under a flow of dry N_2 , the solution was cooled to 0°C with an ice-water bath and a solution of the diacid chloride in THF (10 mL) was added by syringe pump over 1h. The mixture was allowed to stir at room temperature overnight.

THF was removed by rotary evaporation and the residue was taken up in CH_2Cl_2 (100 mL) and washed with water (20 mL). After dried with Na_2SO_4 , the solution was reduced to dryness and the residue was subject to column chromatography on silica gel (eluent: $\text{CH}_3\text{OH}/\text{CH}_2\text{Cl}_2 = 5/95$). The fraction was washed with 10% aqueous HCl and dried to give bis-binaphthyl-strapped Fe porphyrin **1** (40 mg, 53%).

MS $m/e = 1461.4$ (M-Cl)⁺ for $\text{C}_{92}\text{H}_{60}\text{ClFeN}_8\text{O}_8$ (LSIMS⁺).

UV-vis (CH_2Cl_2): 346, 420, 504, 566 nm.

Catalyst **1a** was synthesized following the same procedure using CD_3O -substituted binaphthyl diacid.

MS $m/e = 1473.4$ (M-Cl)⁺ for $\text{C}_{92}\text{H}_{48}\text{ClD}_{12}\text{FeN}_8\text{O}_8$ (LSIMS⁺).

UV-vis (CH_2Cl_2): 345, 420, 504, 564 nm.

Demetalation of 1. A mixture of **1** (10 mg) and NaBH_4 (38 mg) in CH_3OH (5 mL) was stirred at rt under N_2 for 1h. To the mixture was added deaerated HCl solution (6 N, 2 mL) with cooling. The demetalated porphyrin was taken up with CH_2Cl_2 (10 mL), washed with water (5 mL \times 2) and dried with Na_2SO_4 . Evaporation of the solvents gave the corresponding metal free porphyrin. The ^1H NMR and mass spectra are identical with the porphrin prepared from $\alpha\alpha\beta\beta$ -TAPP and **4**.

Experiment procedure for determining the ee's at different turnover numbers.

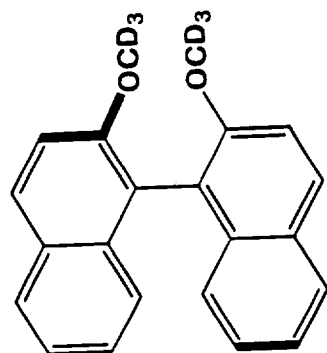
Catalyst **1** or **1a** (1 μmol), dodecane (25 μL , as a GC standard) and styrene (2.0 mL, ca. 17.5 mmol) and CH_2Cl_2 (2 mL) were mixed in a Shlenk tube. The mixture was deaerated by evacuation and refilling N_2 several times. With stirring and under a N_2 flow, PhIO was added in 10 μmol , 10 μmol , 100 μmol , 200 μmol , 400 μmol , 1000 μmol , 2000 μmol , 2000 μmol portions over time intervals during which the previous batch of oxidant was mostly consumed as indicated by the disappearance of solid PhIO. Small aliquots of the reaction mixture were taken periodically and subject to GC analysis as described in **General Procedure**. Ee's were plotted against the corresponding turnover numbers.

Styrene epoxidation catalyzed by 1 with different axial ligands.

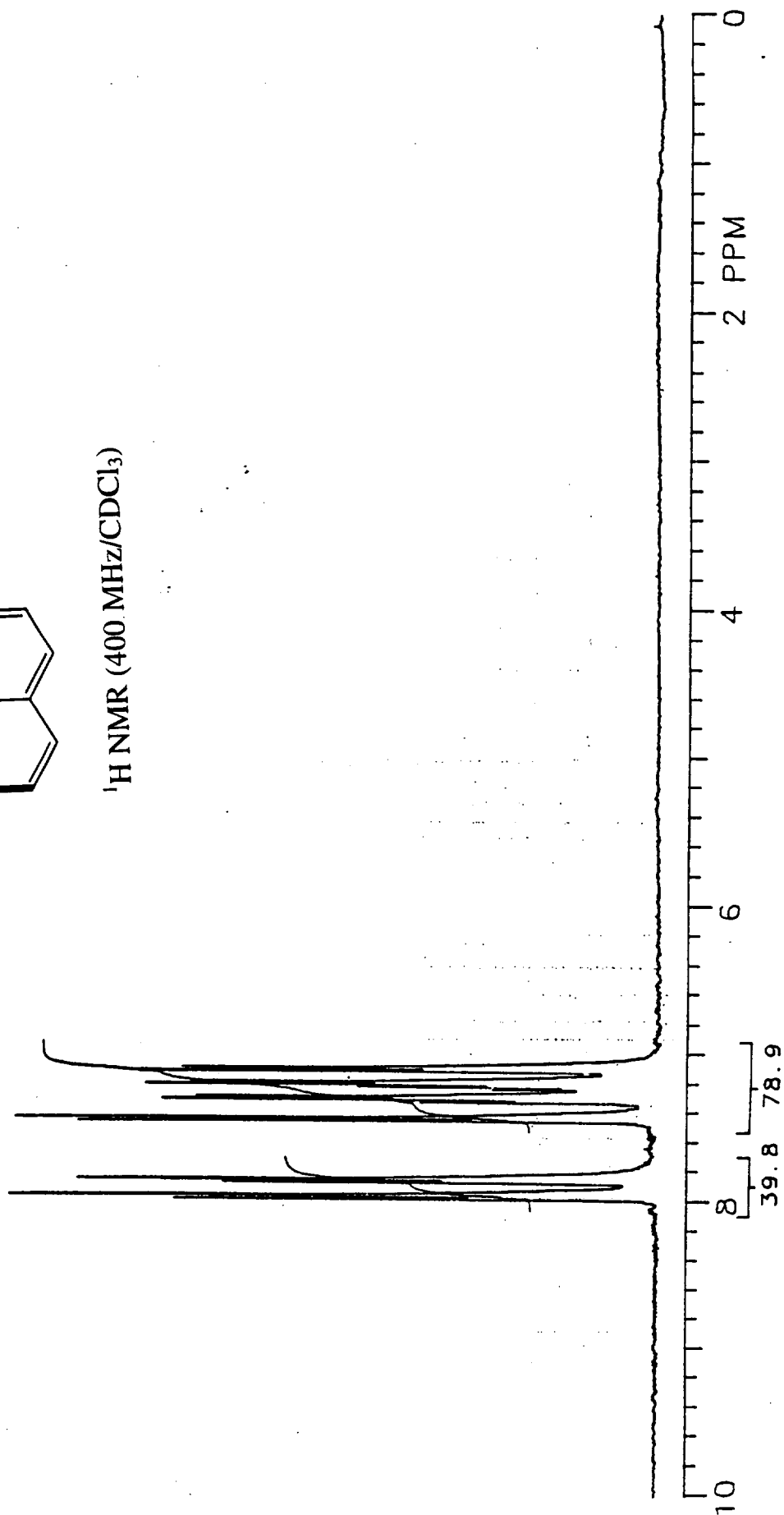
Catalyst 1 (1 μmol), dodecane (25 μL , as a GC standard), styrene (1.0 mmol) and an ligand (30 μmol) were dissolved in freshly distilled CH_2Cl_2 (2 mL) in a Shlenk tube. When weak field ligands such as DMSO and 2,6-difluoropyridine are used, AgBF_4 (5 μmol) was added and the mixture was stirred for 1h to remove choride ligand from the iron (aluminum foil was applied to protect the tube from light). The mixture was deaerated by evacuation and refilling N_2 several times. With stirring and under a N_2 flow, PhIO (0.10 mmol) was added. The reaction mixture was stirred at rt. The epoxidation products were analyzed by GC as described in **General Procedure**.

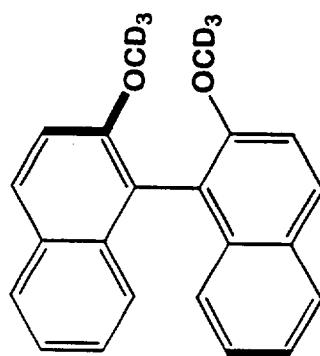
Epoxidation Reaction Using Olefin as the Limiting Reagent.

Catalyst 1 (1 μmol) and an olefin (1.0 mmol) were dissolved in CH_2Cl_2 (2 mL) in a Shlenk tube. PhIO (264 mg, 1.2 mmol) was added to the solution in 10 portions at room temperature with stirring. Each subsequent portion was added when the previous batch of PhIO had been consumed as indicated by the disappearance of the solid oxidant. The reaction was monitored with GC until the substrate was completely consumed. The reaction mixture was concentrated and the residue was subject to column chromatography on silica gel to give the epoxide. The ee of the product was determined by GC as described in **General Procedure**.

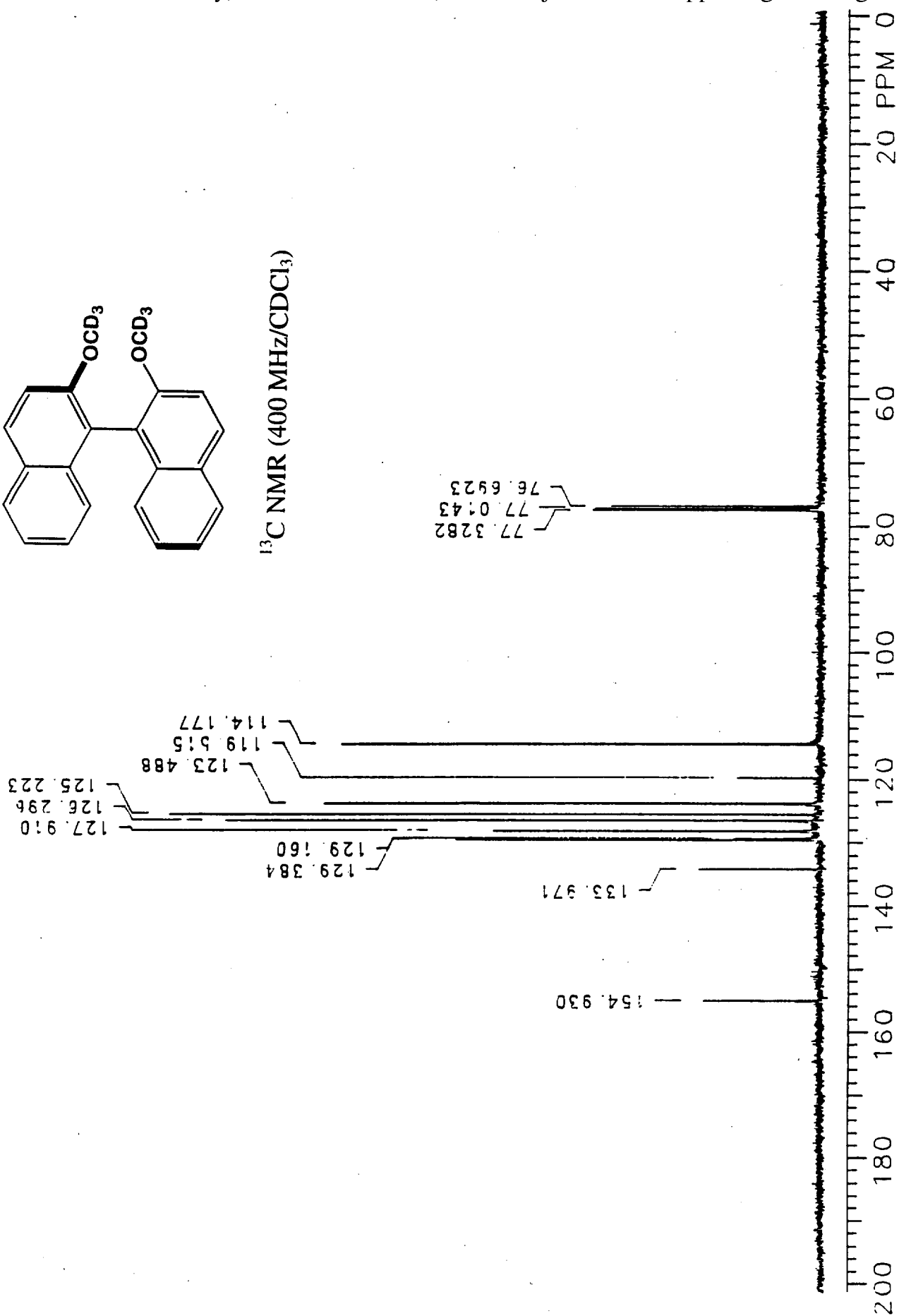


¹H NMR (400 MHz/CDCl₃)



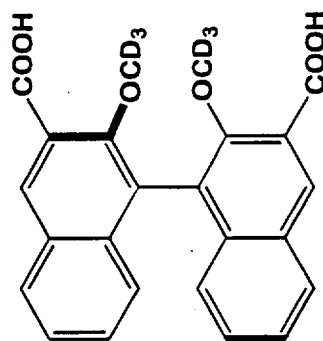


^{13}C NMR (400 MHz/ CDCl_3)

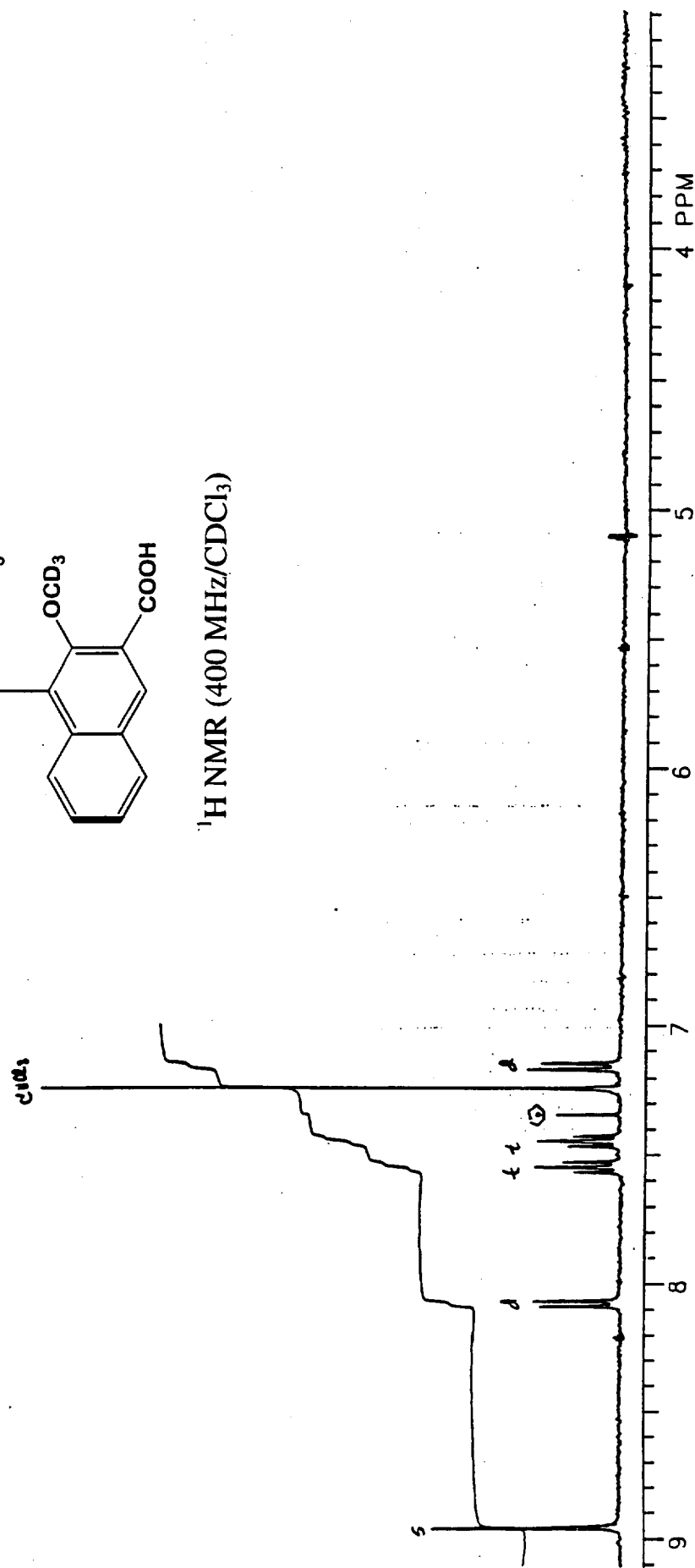


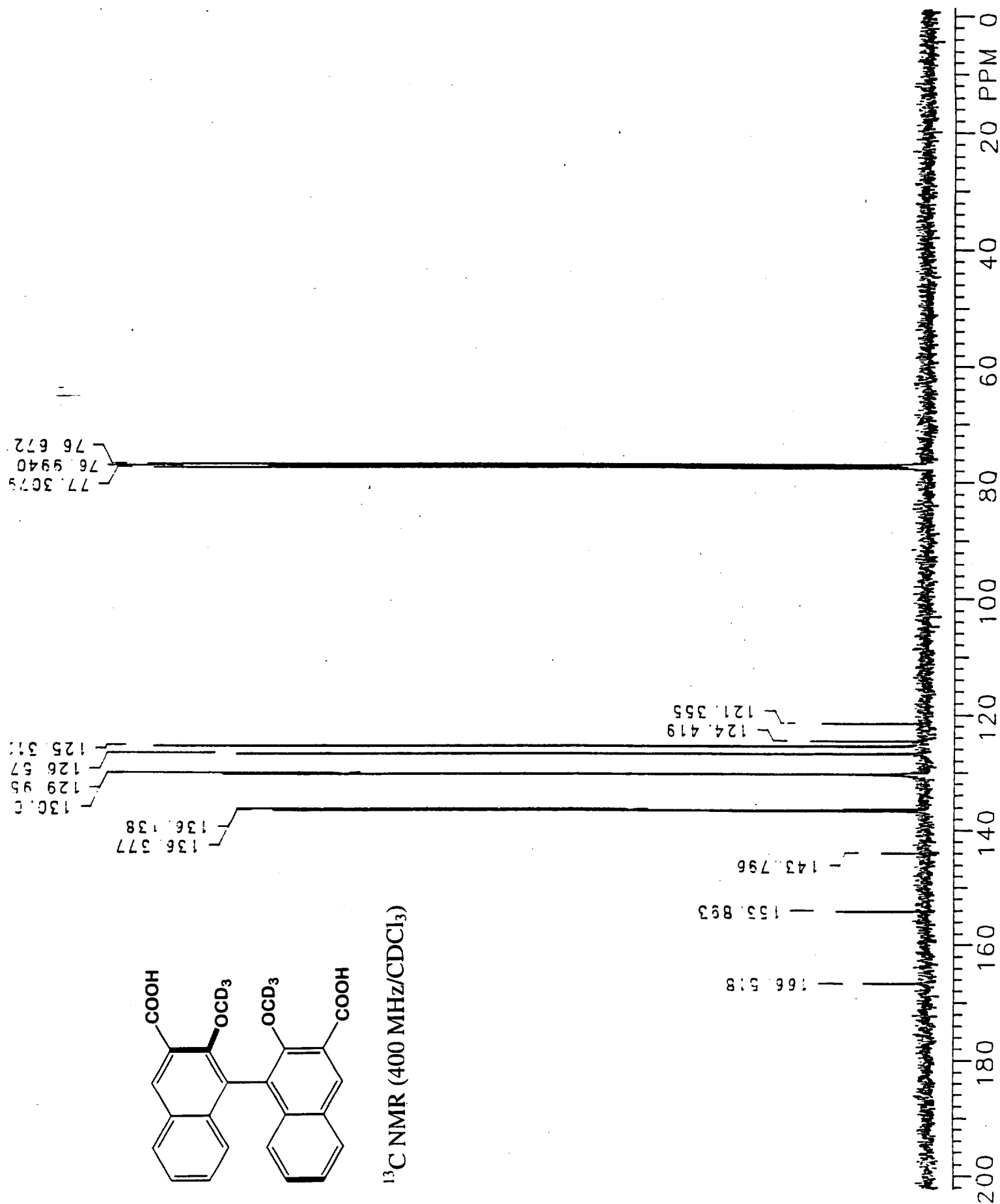
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DATE 07-19-96
SOLVENT CDCL3
FILE CDCL3

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RELAXATION DELAY 1.0 MSEC
PULSE WIDTH 41 DEGREES
AMBIENT TEMPERATURE
NO. REPETITIONS 16
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
LINE BROADENING 0.2 HZ
FT SIZE 32K



¹H NMR (400 MHz/CDCl₃)





aabb TAPP + binap dladid c1, frac 1

Solvent: CDCl₃
Ambient temperature
INNOVA-500 "01500"

PULSE SEQUENCE

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Width 8999.9 Hz

10000 repetitions

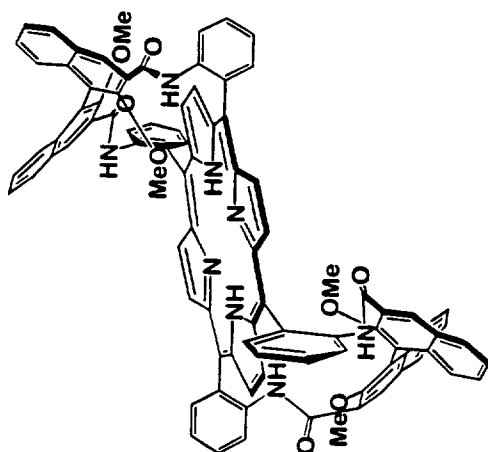
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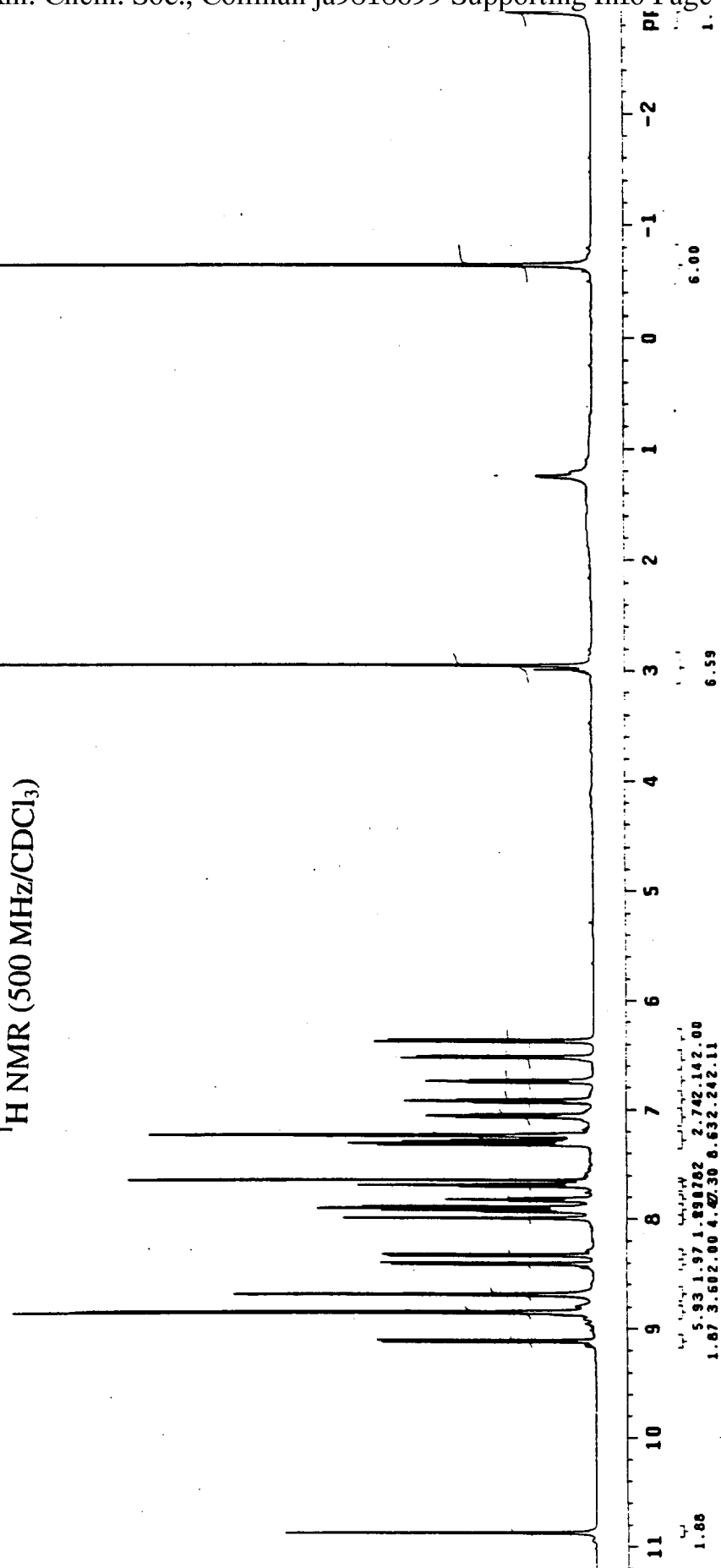
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Total time 5.6 hours



¹H NMR (500 MHz/CDCl₃)

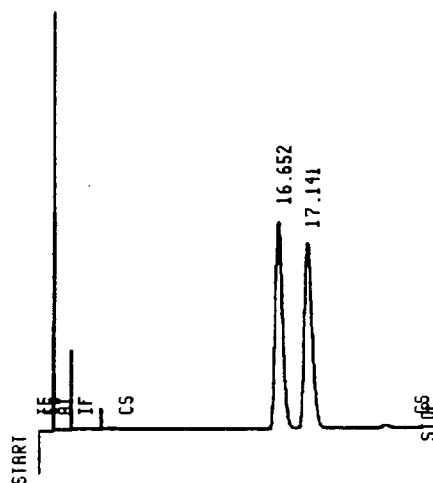


Cyclodex-B-PH
J & W Scientific
FID

Column:
Detection:

GC Conditions:

Racemate
Oven temperature: 90°C



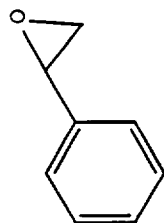
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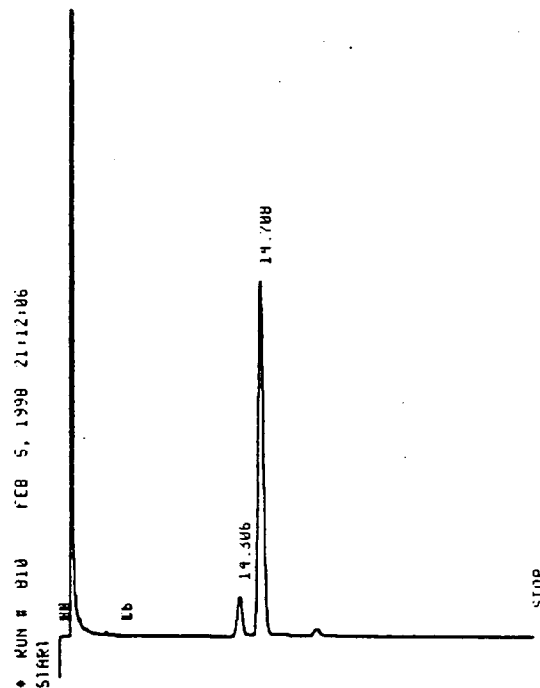
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Reaction Product
Oven temperature: 95°C



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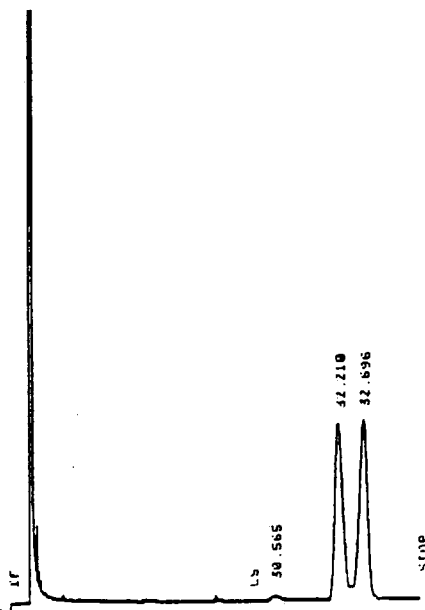
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GC Conditions: Column: Cyclodex-B-PH
J & W Scientific
Oven temperature: 70°C
Detection: FID

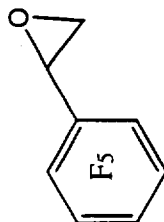
Racemate

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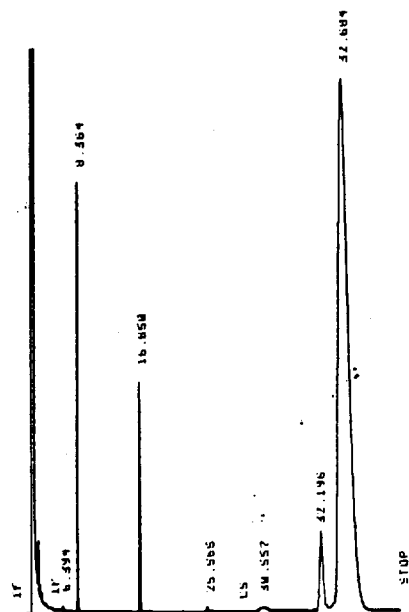


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Reaction Product



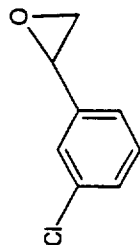
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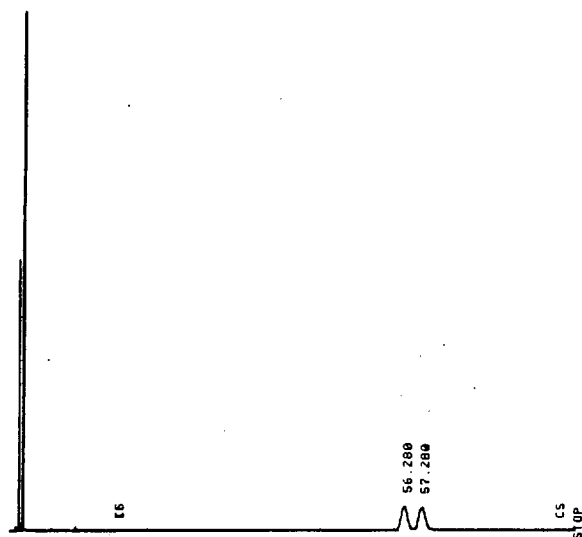
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GC conditions: Column: Cyclodex-B-PH
J & W Scientific
Oven temperature: 90°C
Detection: FID

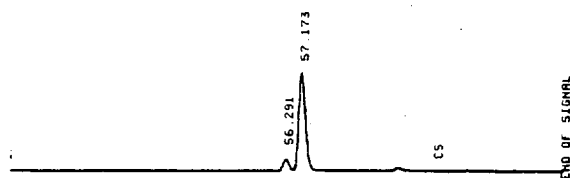
Racemate



Reaction Mixture



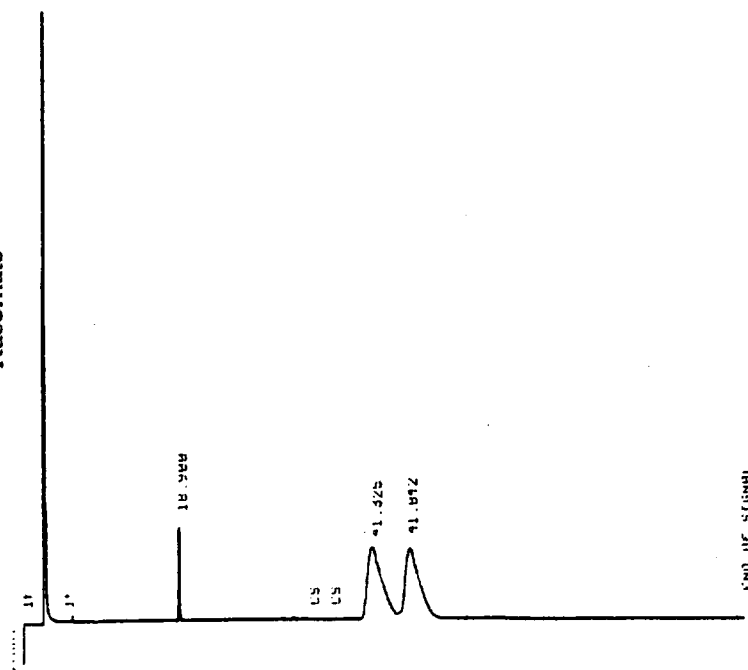
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J & W Scientific
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Detection: FID

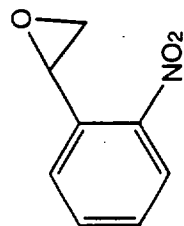
Racemate



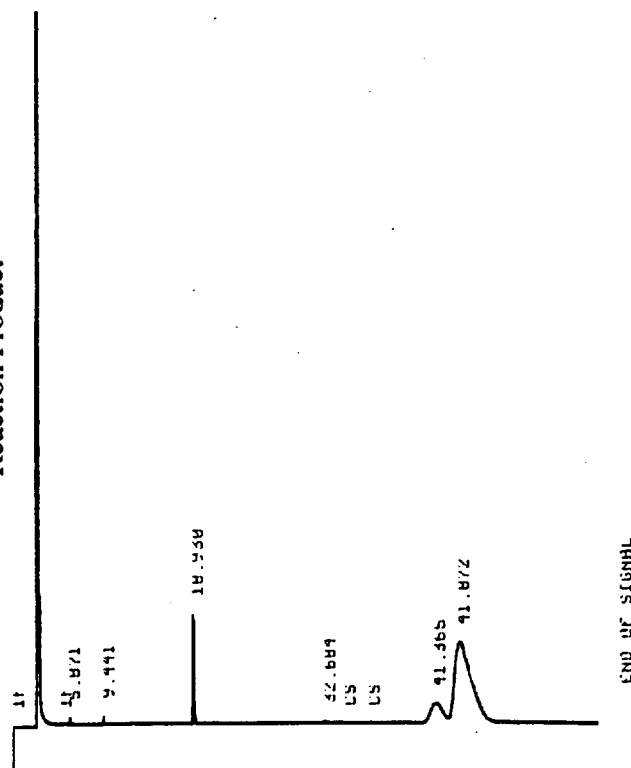
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Reaction Product



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42.684	2244	PB	.281	1.43337
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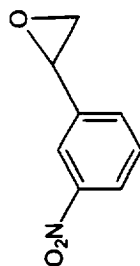
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J & W Scientific
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Detection: FID

Racemate



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Reaction Mixture



END OF SIGNAL

Closing signal file M:SIGNAL .BNA
RUN# 1747 SEP 1, 1998 19:22:22
SIGNAL FILE: M:SIGNAL .BNA
AREA# RT AREA TYPE WIDTH AREA#
152.854 12467 PU 1.167 13.88523
154.352 77319 UP 1.247 86.11476
TOTAL AREA= 89786
MUL FACTOR=1.0000E+00

GC Conditions:

Column:

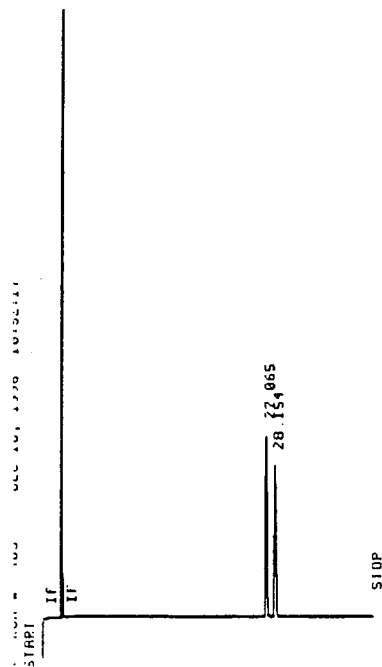
Cyclodex-B-PH
J & W Scientific

Detection:

FID

Racemate

Oven temperature: 100°C



Closing signal file M:SIGNAL.BNC

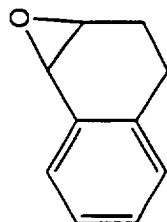
RUN# 403 DEC 19, 1996 10:52:17

SIGNAL FILE: M:SIGNAL.BNC

RT	AREA	TYPE	WIDTH	APRAX
28.134	59576	PB	.209	93.86774
28.154	59892	PB	.225	58.13225

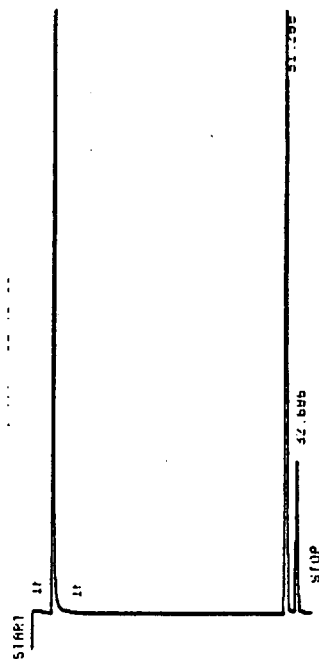
TOTAL AREA= 119468

MUL FACTOR=1.0086E+00



Reaction Product

Oven temperature: 95°C



Closing signal file M:SIGNAL.BNC

RUN# 202 OCT 24, 1997 12:40:38

SIGNAL FILE: M:SIGNAL.BNC

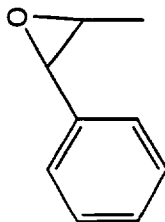
RT	AREA	TYPE	WIDTH	APRAX
31.289	117500	PB	.278	77.49434
32.686	34153	PB	.278	22.50565

TOTAL AREA= 151653

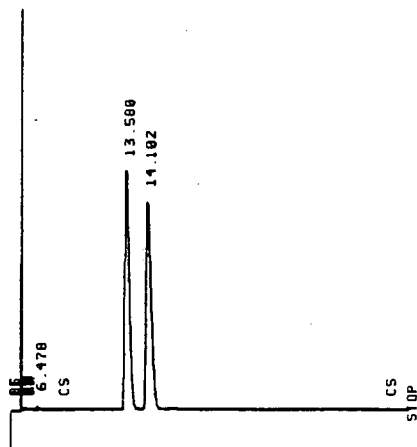
MUL FACTOR=1.0086E+00

Cyclodex-B-PH
J & W Scientific
95°C
FID

GC Conditions:
Column:
Oven temperature:
Detection:

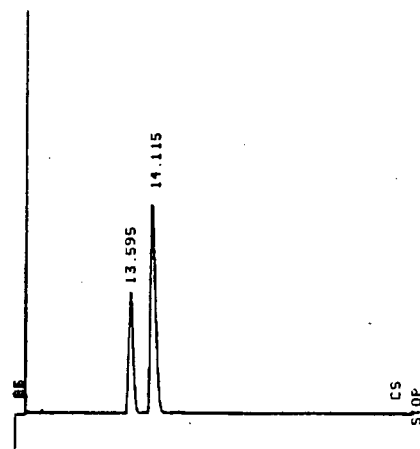


Racemate

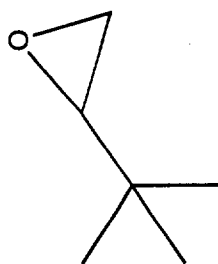


Closing signal file M:SIGNAL .BNC
RUN# 871 AUG 11, 1997 21:50:20
SIGNAL FILE: M:SIGNAL.BNC
AREAX RT AREA TYPE WIDTH AREAX
6.478 1244 BB .059 .66484
{ 13.580 92884 PB .121 49.58070
14.102 93211 BB .139 49.75526
TOTAL AREA= 107339
MUL FACTOR=1.0000E+00

Reaction Product

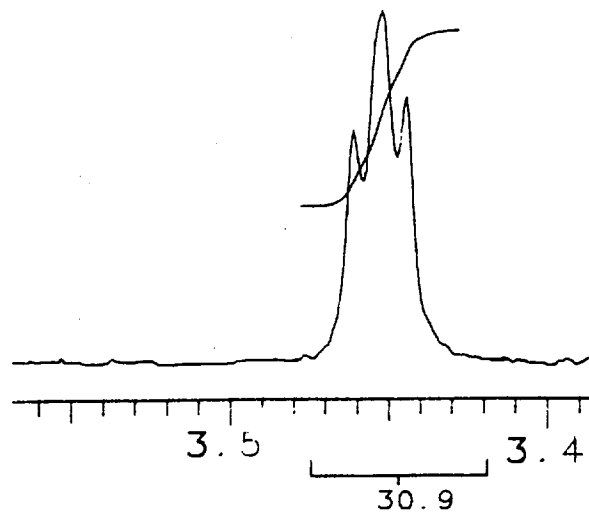


Closing signal file M:SIGNAL .BNC
RUN# 871 AUG 11, 1997 21:50:20
SIGNAL FILE: M:SIGNAL.BNC
AREAX RT AREA TYPE WIDTH AREAX
13.595 32826 PB .121 25.49983
14.115 93567 BB .139 74.58017
TOTAL AREA= 125593
MUL FACTOR=1.0000E+00

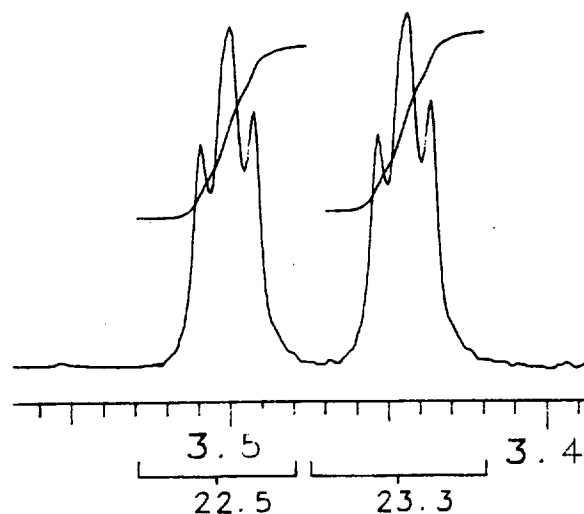


¹H NMR (400 MHz/CDCl₃)

Reaction Product + Eu(hfc)₃



Racemate + Eu(hfc)₃



There is no peak corresponding to the minor isomer. Assuming a 5% detection limit for NMR spectrometer, the minor isomer should be $\leq 5\%$, and the ee $\geq 90\%$.

GC Conditions:

Column:

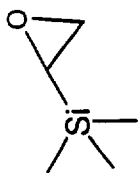
Cyclodex-B-PH
J & W Scientific

Oven temperature:

35°C

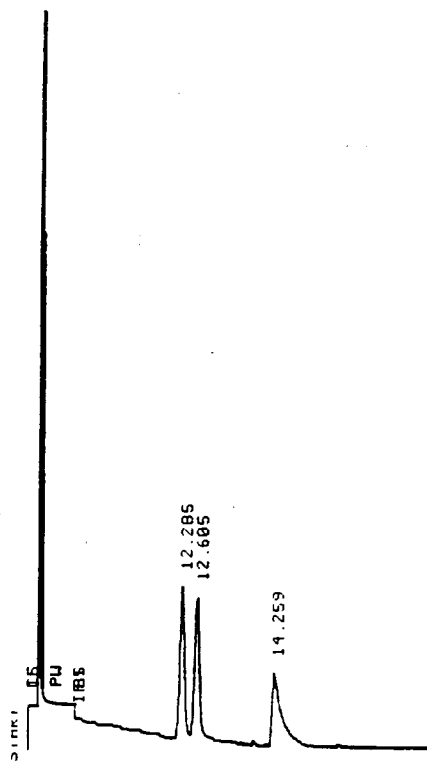
Detection:

FID



Racemate

Reaction Product



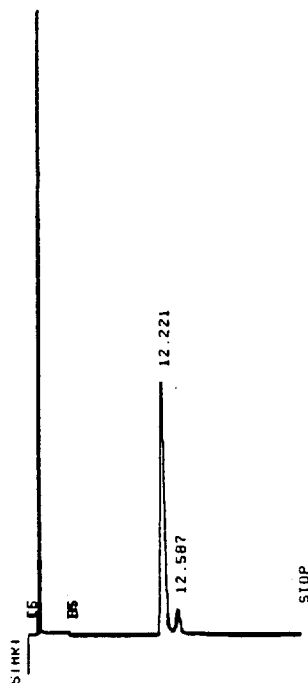
END OF SIGNAL

Closing signal file M:SIGNAL .BNA

RUN# 276 MAR 22, 1998 17:20:13

SIGNAL FILE: M:SIGNAL.BNA

AREA#	RT	AREA	TYPE	WIDTH	AREA#
1	12.285	1501	PU	.113	28.80446
2	12.605	1493	UB	.118	28.55894
3	14.259	1792	PB	.260	34.38880
4	23.165	425	PP	.288	8.15582

TOTAL AREA= 5211
MUL FACTOR=1.0000E+00

Closing signal file M:SIGNAL .BNC

RUN# 280 MAR 22, 1998 22:33:21

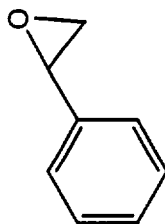
SIGNAL FILE: M:SIGNAL.BNC

AREA#	RT	AREA	TYPE	WIDTH	AREA#
1	12.221	11277	UU	.116	98.96554
2	12.587	1120	UP	.113	9.83441

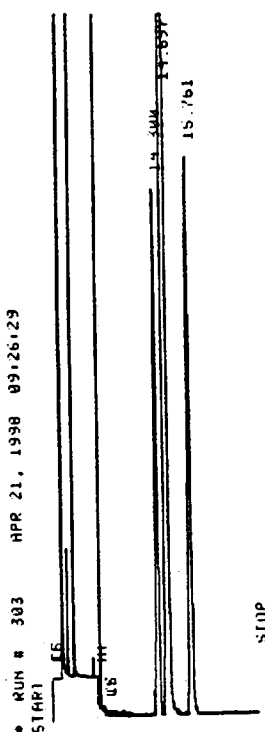
TOTAL AREA= 12397

MUL FACTOR=1.0000E+00

GC Conditions: Column: Cyclodex-B-PH
 J & W Scientific
 Oven temperature: 95°C
 Detection: FID

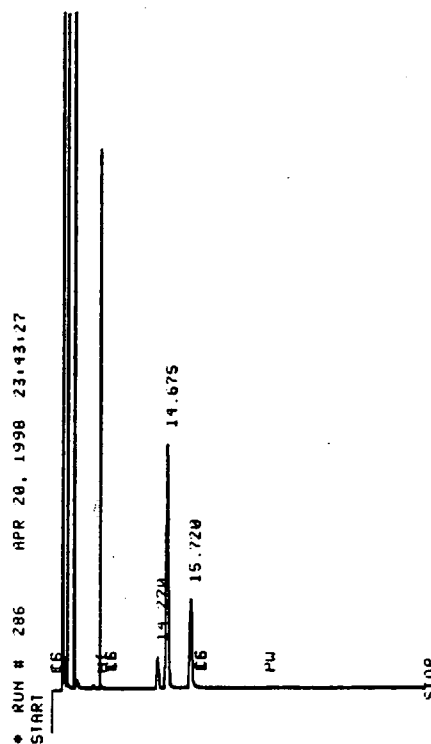


Reaction Product using pyridine as the ligand



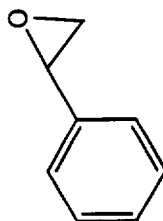
Closing signal file M:SIGNAL .BNC
 RUN# 303 APR 21, 1998 09:26:29
 SIGNAL FILE: M:SIGNAL .BNC
 AREAX RT AREA TYPE WIDTH AREAX
 { 14.300 2626 BP .110 10.60582
 14.697 18986 PB .119 76.35782
 15.761 3228 BB .127 13.83716
 TOTAL AREA= 24760
 MUL FACTOR=1.0000E+00

Reaction Product using 2,6-difluoropyridine as the ligand



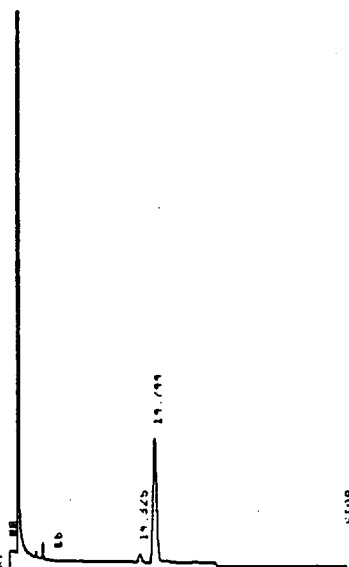
Closing signal file M:SIGNAL .BNC
 RUN# 286 APR 20, 1998 23:43:27
 SIGNAL FILE: M:SIGNAL .BNC
 AREAX RT AREA TYPE WIDTH AREAX
 { 14.270 1290 PP .114 8.43192
 14.675 10031 PB .116 65.56637
 15.720 3978 PB .124 26.00171
 TOTAL AREA= 15299
 MUL FACTOR=1.0000E+00

GC Conditions: Column: Cyclodex-B-PH
J & W Scientific
Oven temperature: 95°C
Detection: FID



Reaction Product using DMSO as the ligand

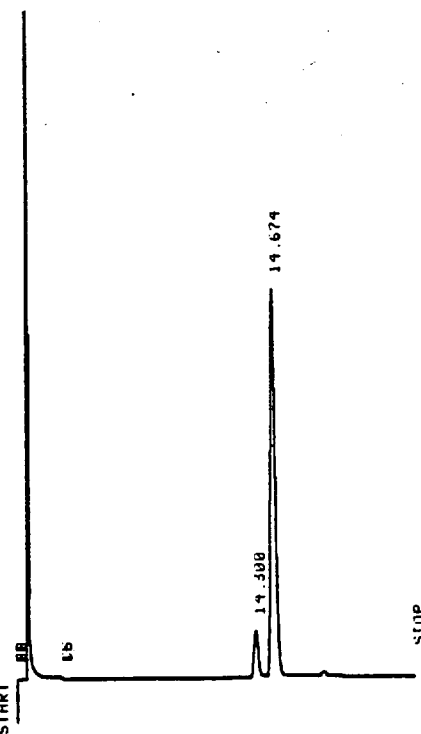
• RUN # 809 FEB 5, 1998 20:50:09
START



Closing signal file M:SIGNAL.BNC
RUN# 809 FEB 5, 1998 20:50:09
SIGNAL FILE: M:SIGNAL.BNC
AREA# RT AREA TYPE WIDTH AREA#
14.325 502 BP .118 6.19983
14.744 7595 PB .114 93.00017
TOTAL AREA# 8097
MUL FACTOR=1.0000E+00

Reaction Product using Br⁻ as the ligand

• RUN # 773 FEB 3, 1998 21:10:57
START



Closing signal file M:SIGNAL.BNC
RUN# 773 FEB 3, 1998 21:10:57
SIGNAL FILE: M:SIGNAL.BNC
AREA# RT AREA TYPE WIDTH AREA#
14.308 3656 BP .108 9.07334
14.674 33373 PB .119 90.12666
TOTAL AREA# 37029
MUL FACTOR=1.0000E+00