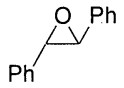
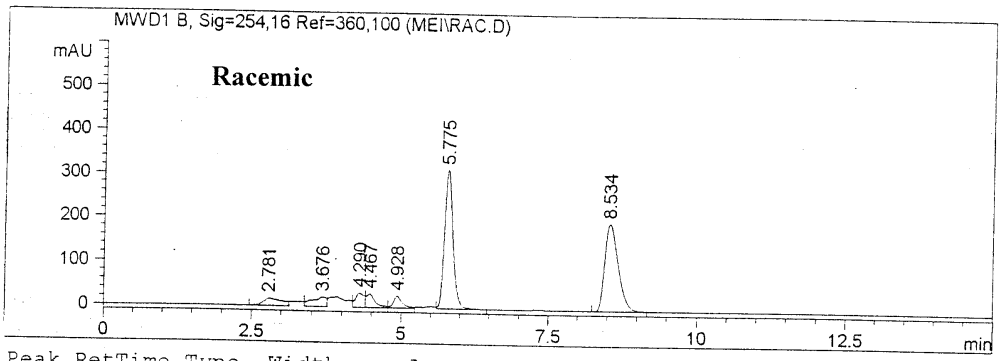
Determination of enantiomeric excess of epoxide of 12 by HPLC

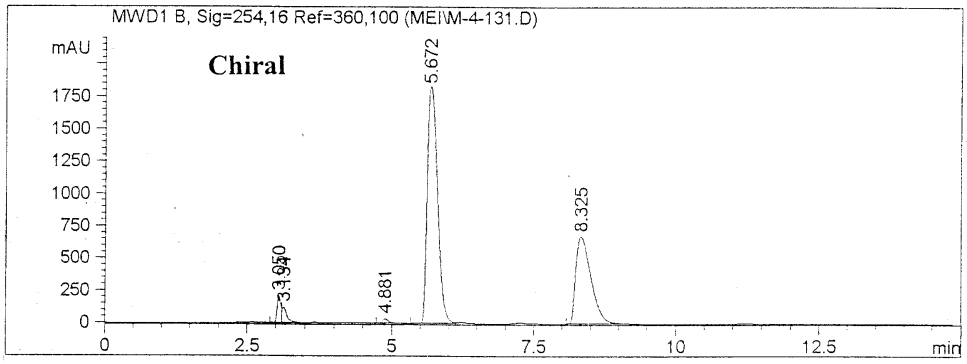
Table 1, entry 4



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-AH045)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



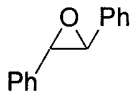
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.781	VV	0.2769	380.88989	17.03100	4.7548
2	3.676	VV	0.2193	373.82172	22.56270	4.6665
3	4.290	VV	0.1433	334.00885	32.12846	4.1696
4	4.467	VV	0.1499	329.70874	30.56289	4.1159
5	4.928	VV	0.1511	283.40952	26.86290	3.5379
6	5.775	VV	0.1504	3155.93799	316.34717	39.3967 ◄
7	8.534	VV	0.2478	3152.89087	198.00575	39.3587 ◄
Totals :				8010.66757	643.50087	



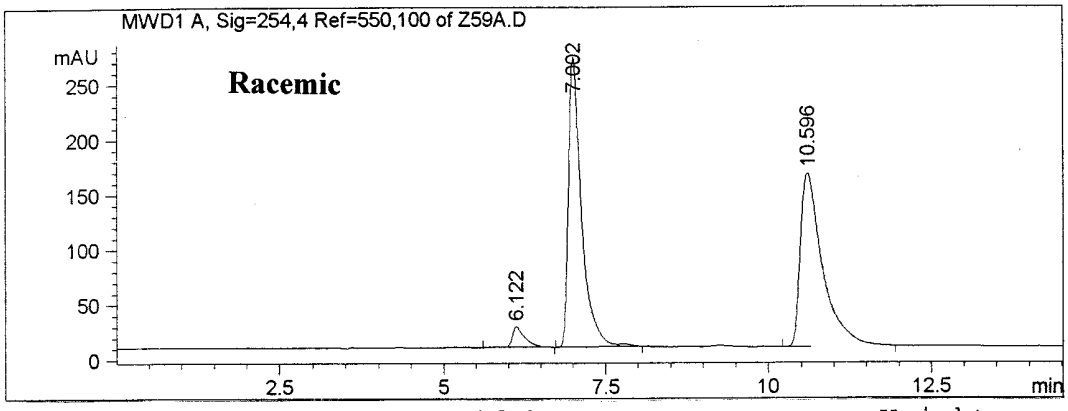
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.050	VV	0.0708	986.33673	210.03909	2.6299
2	3.134	VV	0.0876	730.85956	119.01546	1.9487
3	4.881	VB	0.1274	272.77127	31.99989	0.7273
4	5.672	BV	0.1974	2.28587e4	1834.47424	60.9498 ◄
5	8.325	VV	0.2890	1.26555e4	674.05310	33.7442 ◄
Totals :				3.75042e4	2869.58178	

Determination of enantiomeric excess of epoxide of 12 by HPLC

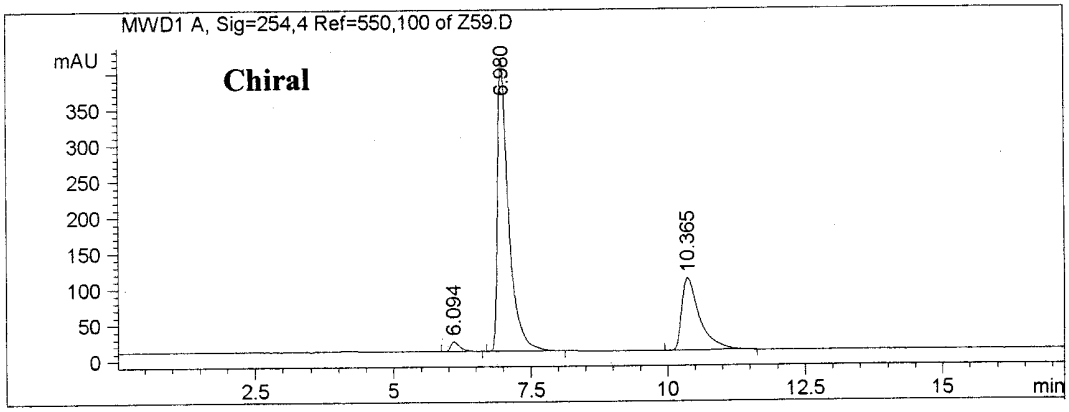
Table 1, entry 5



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JH192)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



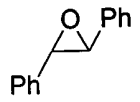
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.122	BB	0.201	264.31732	18.77988	3.4491
2	7.002	BB	0.210	3700.78589	262.00616	48.2922 ←
3	10.596	BB	0.347	3698.22266	157.02022	48.2587 ←
Totals :				7663.32568	437.80627	



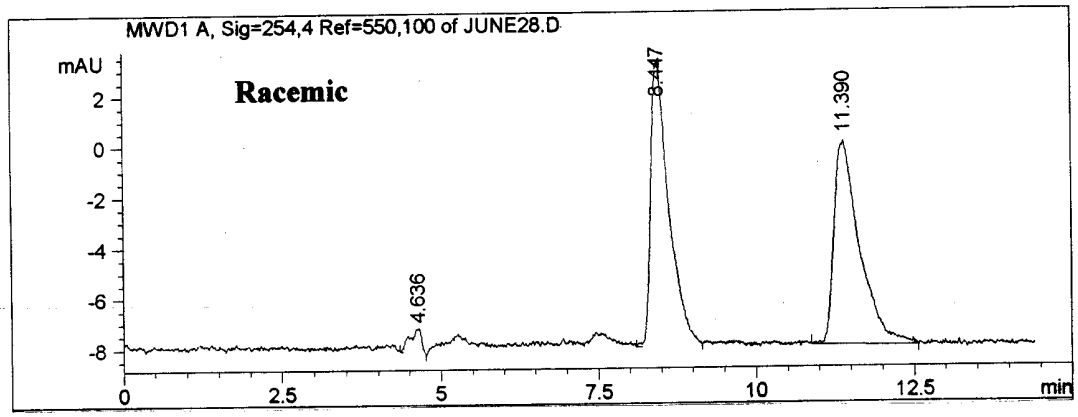
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.094	BB	0.174	161.13721	13.68364	1.9796
2	6.980	BB	0.209	5685.39258	404.00726	69.8475 ←
3	10.365	BB	0.338	2293.18726	100.34383	28.1728 ←
Totals :				8139.71680	518.03473	

Determination of enantiomeric excess of epoxide of 12 by HPLC

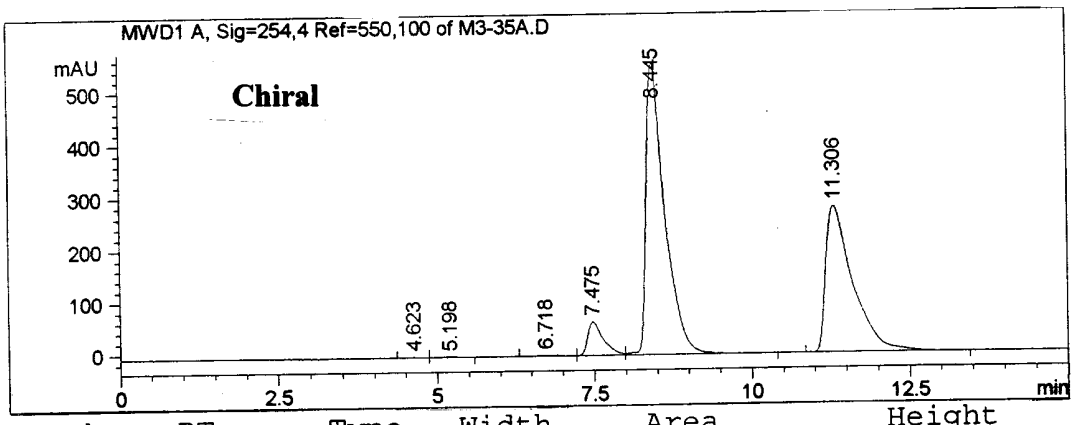
Table 2, entry 1



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



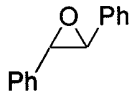
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.636	VV	0.193	13.55257	9.80176e-1	2.9453
2	8.447	BV	0.294	222.53619	11.26474	48.3625 ←
3	11.390	BV	0.337	224.05283	8.11261	48.6921 ←
Totals :				460.14160	20.35753	



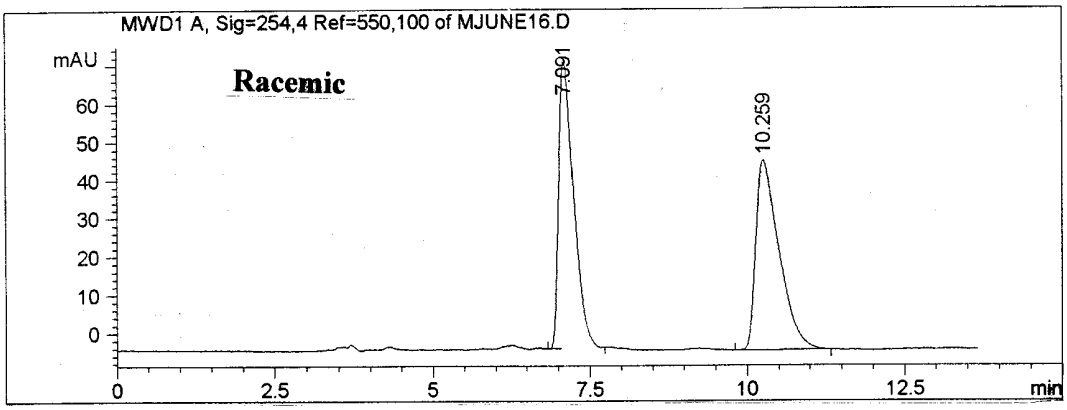
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.623	VV	0.252	16.50654	8.69882e-1	0.0790
2	5.198	VV	0.266	23.31474	1.12430	0.1116
3	6.718	VV	0.294	36.00965	1.58422	0.1723
4	7.475	VV	0.256	1110.93359	64.35889	5.3153
5	8.445	VV	0.303	11395.34766	556.81451	54.5218 ←
6	11.306	BV	0.436	8318.43164	280.66083	39.8001 ←
Totals :				20900.54297	905.41266	

Determination of enantiomeric excess of epoxide of 12 by HPLC

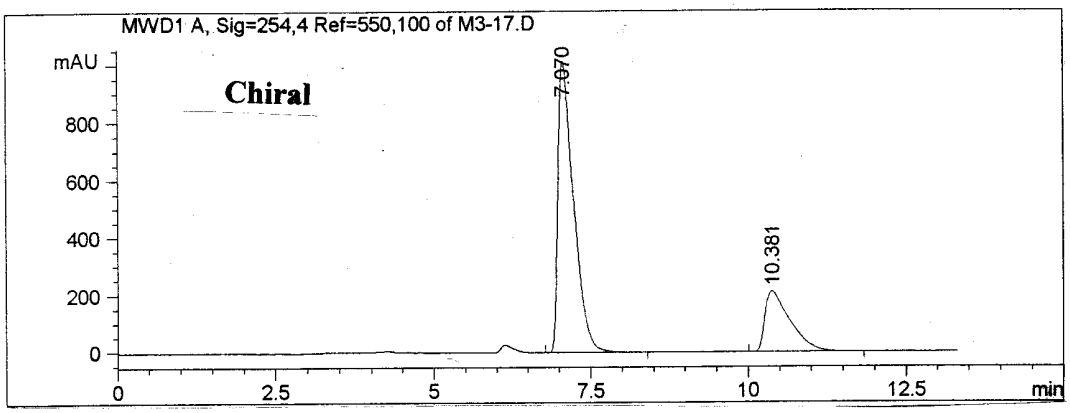
Table 2, entry 6



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



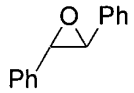
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.091	BB	0.242	1241.73389	75.41746	49.4376
2	10.259	BB	0.369	1269.98535	49.54792	50.5624
Totals :				2511.71924	124.96538	



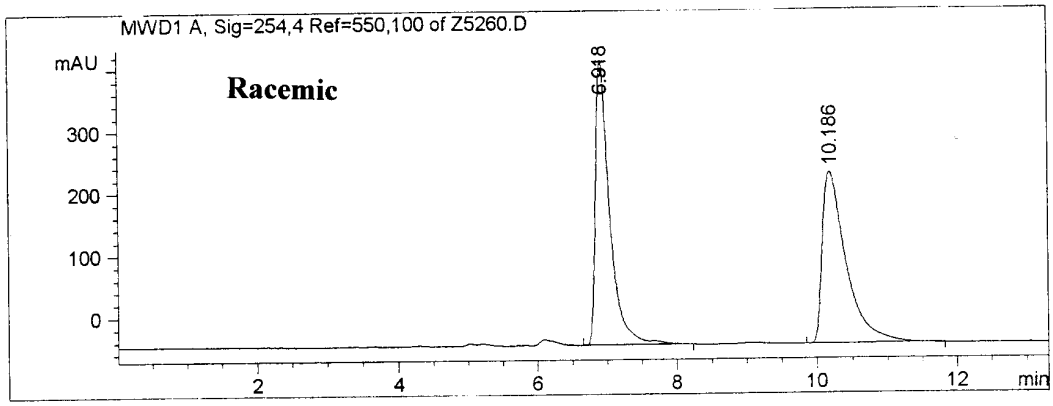
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.070	BB	0.254	17591.93359	1012.28772	74.8865
2	10.381	BB	0.400	5899.53467	212.92455	25.1135
Totals :				23491.46875	1225.21228	

Determination of enantiomeric excess of epoxide of 12 by HPLC

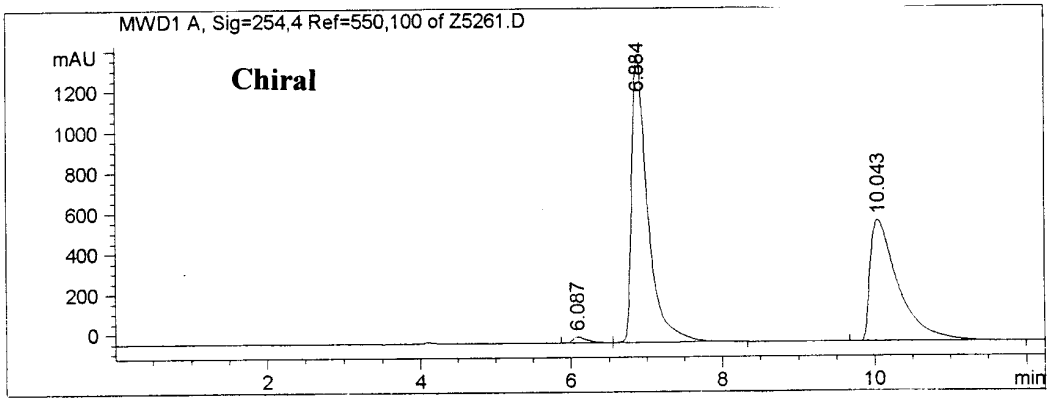
Table 2, entry 7



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



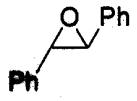
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.918	BB	0.212	6522.30908	458.83734	50.1898
2	10.186	BB	0.347	6472.97510	276.22931	49.8102
Totals :				12995.28418	735.06665	



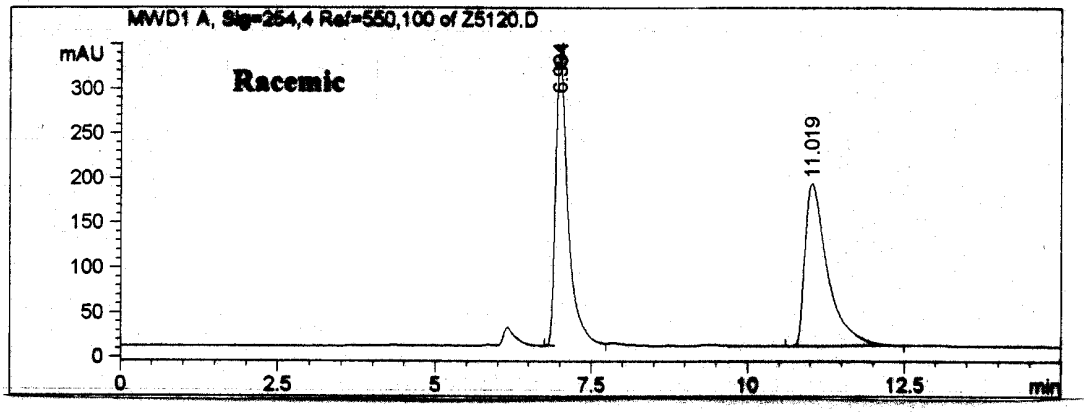
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.087	BV	0.188	396.82706	30.87566	1.0814
2	6.884	VB	0.221	20869.51367	1405.19897	56.8720 ←
3	10.043	BB	0.376	15429.21387	600.37433	42.0465 ←
Totals :				36695.55469	2036.44897	

Determination of enantiomeric excess of epoxide of 12 by HPLC

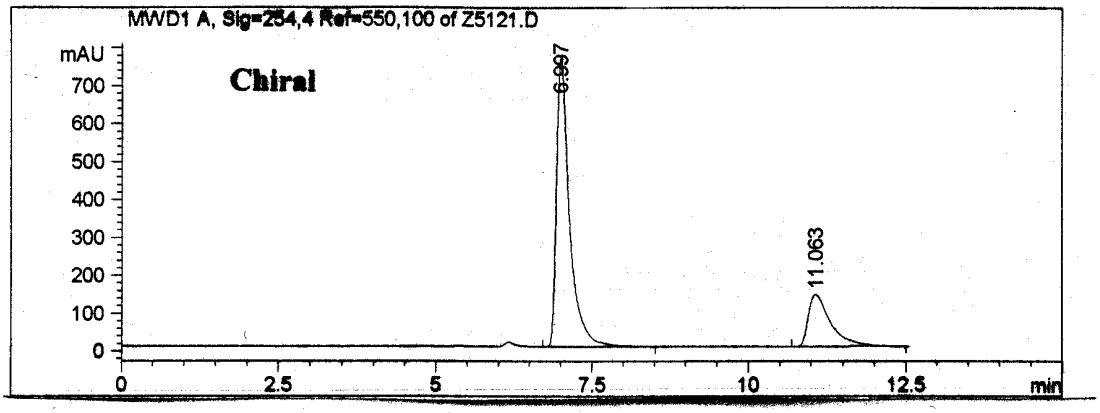
Table 2, entry 9



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



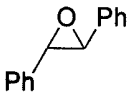
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.994	PV	0.209	4483.89746	320.99002	49.5609
2	11.019	BB	0.373	4563.34521	181.19394	50.4391
Totals				9047.24219	502.18396	



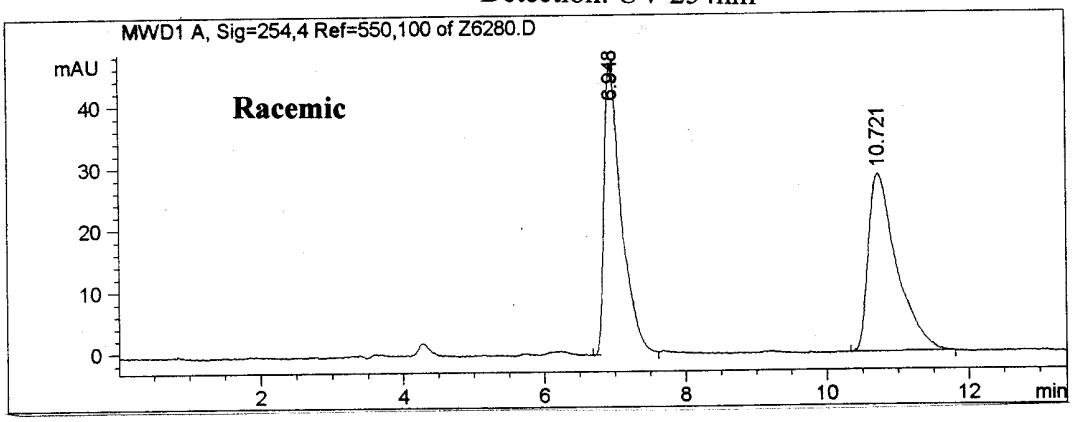
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.997	BB	0.217	11119.41504	762.03595	76.5876
2	11.063	BBA	0.366	3399.14697	137.55124	23.4124
Totals :				14518.56250	899.58716	

Determination of enantiomeric excess of epoxide of 12 by HPLC

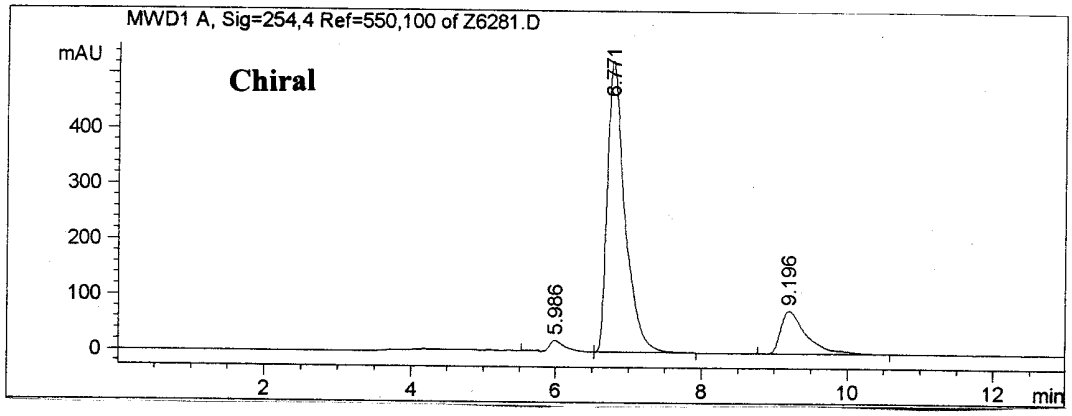
Table 2, entry 10



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



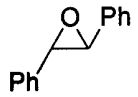
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.948	BB	0.252	787.11218	46.66928	49.6830
2	10.721	BB	0.420	797.15613	28.71158	50.3170
Totals :				1584.26831	75.38086	



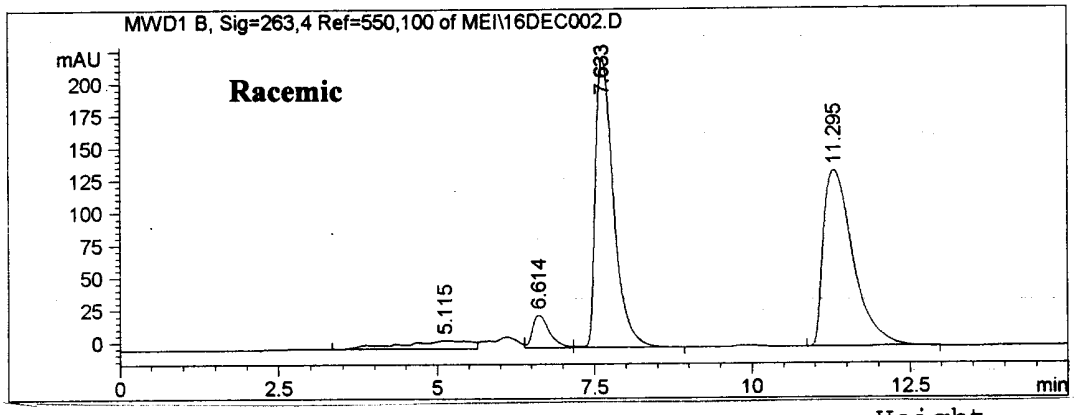
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.986	BV	0.208	272.55548	19.64817	2.4658
2	6.771	PB	0.250	8835.91406	526.42877	79.9380 ←
3	9.196	BB	0.368	1944.98523	77.78242	17.5962 ←
Totals :				11053.45508	623.85937	

Determination of enantiomeric excess of epoxide of 12 by HPLC

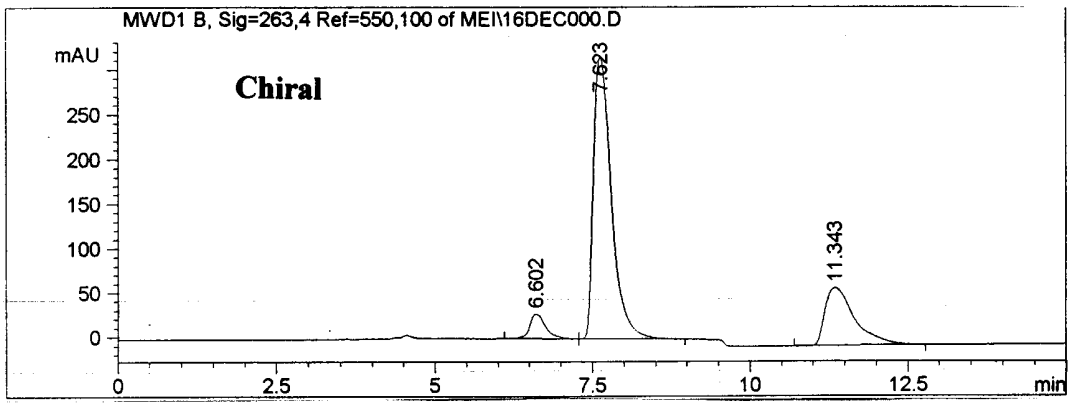
Table 3, entry 1



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 263nm



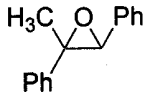
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.115	BV	1.058	499.33588	6.49580	5.0345
2	6.614	VV	0.294	487.11835	25.41404	4.9113
3	7.633	VB	0.315	4492.08838	222.45544	45.2909 ◄
4	11.295	BB	0.510	4439.75146	135.33473	44.7633 ◄
Totals :				9918.29395	389.70001	



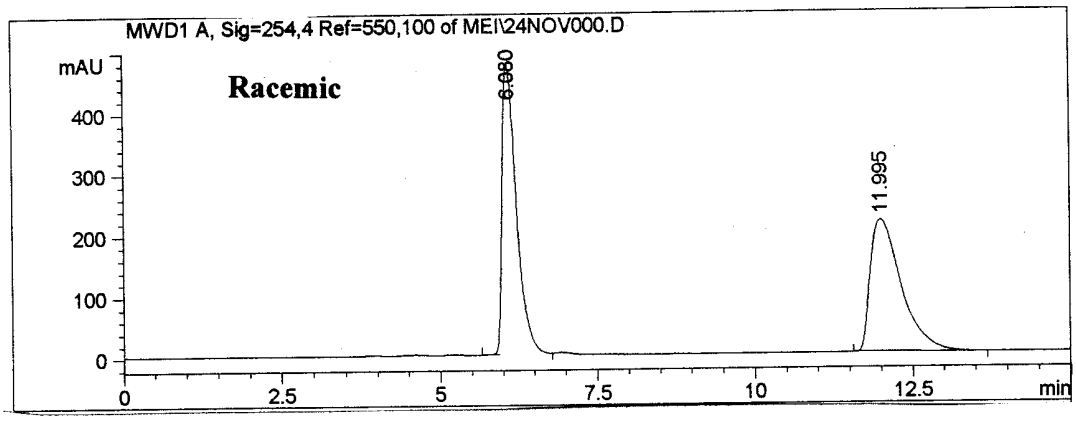
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.602	BV	0.274	498.92612	27.80923	5.5811
2	7.623	PB	0.312	6313.84180	316.19345	70.6284 ◄
3	11.343	BB	0.501	2126.75977	65.19128	23.7905 ◄
Totals :				8939.52734	409.19397	

Determination of enantiomeric excess of epoxide of 13 by HPLC

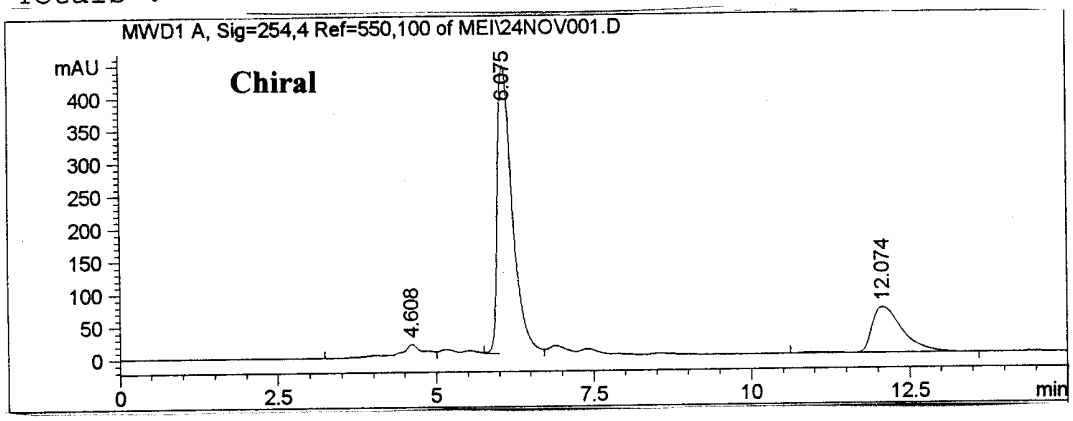
Table 3, entry 2



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



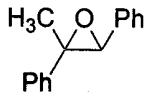
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.080	BV	0.246	7548.02686	474.27679	50.5884
2	11.995	BB	0.530	7372.42969	215.45961	49.4116
Totals :				14920.45703	689.73639	



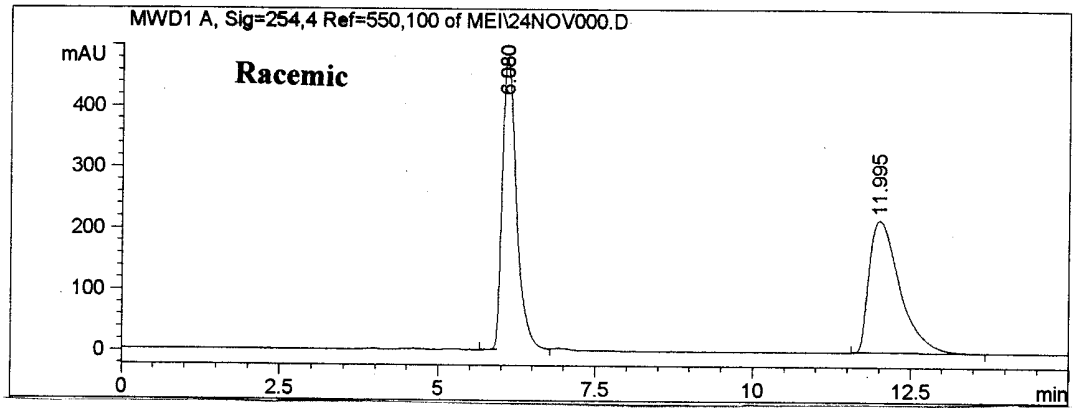
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.608	BV	0.314	416.40900	17.89818	4.1099
2	6.075	VV	0.255	7311.88867	441.48743	72.1670 ←
3	12.074	BB	0.523	2403.60547	70.30433	23.7231 ←
Totals :				10131.90332	529.68994	

Determination of enantiomeric excess of epoxide of 13 by HPLC

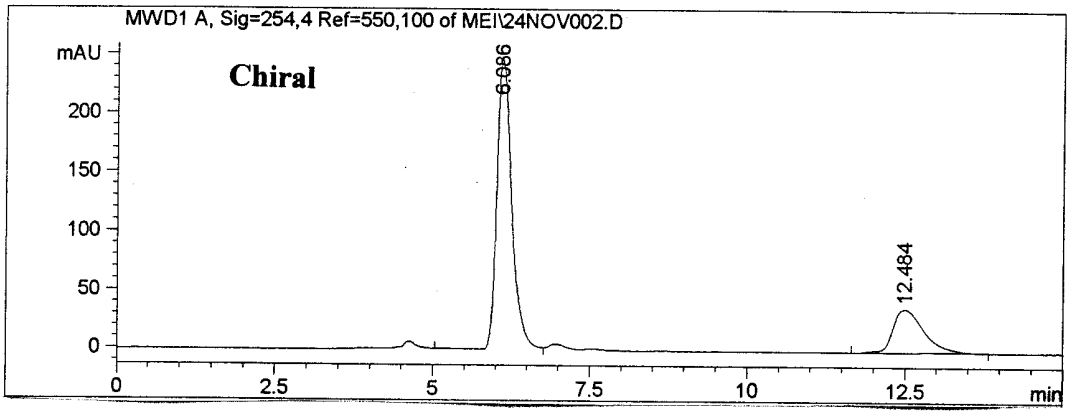
Table 3, entry 3



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



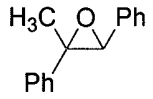
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.080	BV	0.246	7548.02686	474.27679	50.5884
2	11.995	BB	0.530	7372.42969	215.45961	49.4116
Totals :				14920.45703	689.73639	



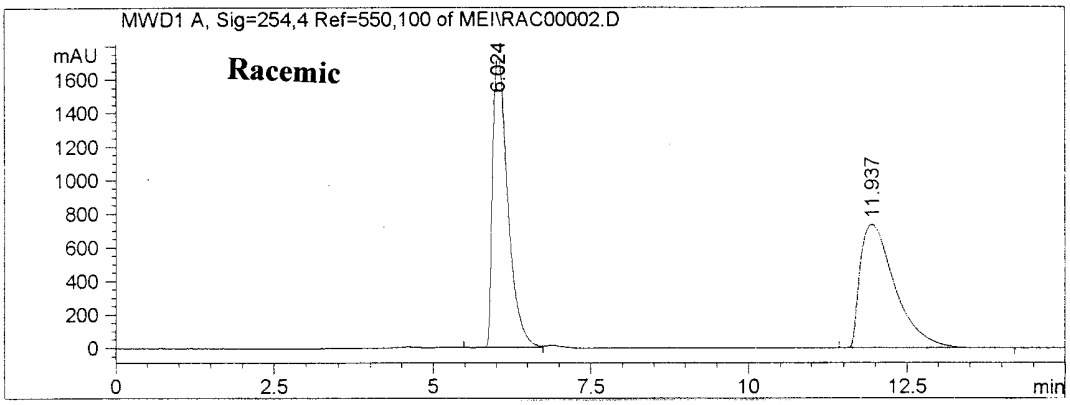
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.086	PV	0.254	4024.51294	245.19695	75.6771
2	12.484	BB	0.529	1293.49536	37.16623	24.3229
Totals :				5318.00830	282.36316	

Determination of enantiomeric excess of epoxide of 13 by HPLC

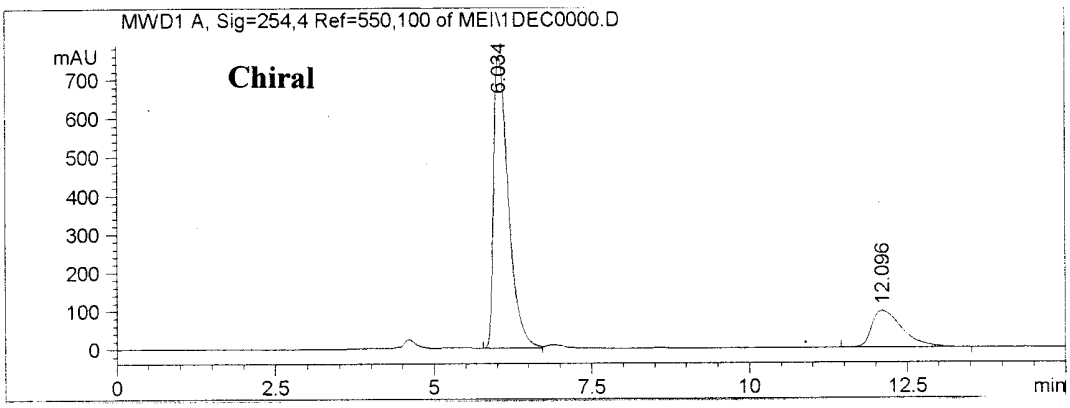
Table 3, entry 4



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



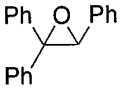
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.024	BV	0.262	29230.96680	1727.85425	50.8136
2	11.937	BB	0.593	28294.88867	735.85498	49.1864
Totals :				57525.85547	2463.70923	



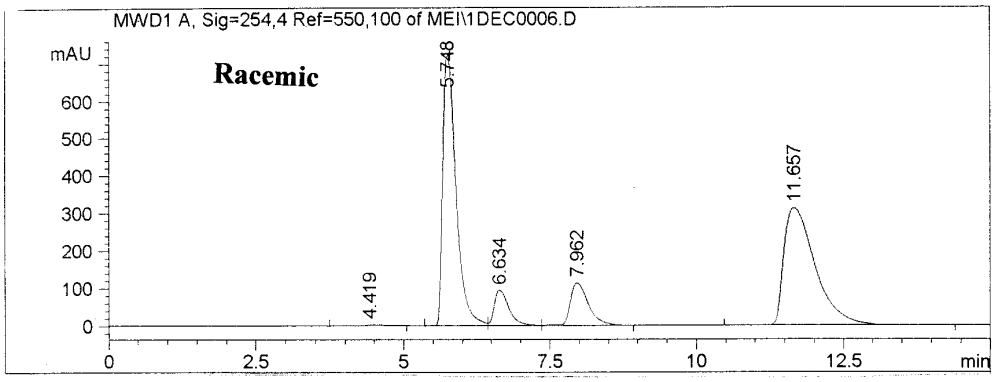
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.034	BV	0.256	12407.69531	751.58984	79.4774
2	12.096	BB	0.524	3203.90967	95.28791	20.5226
Totals :				15611.60547	846.87775	

Determination of enantiomeric excess of epoxide of 14 by HPLC

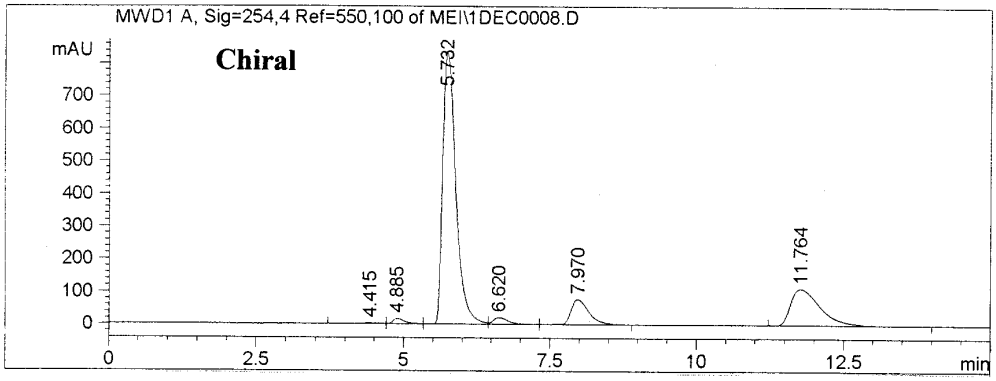
Table 3, entry 5



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



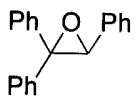
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.419	PV	0.388	61.13882	2.10409	0.2265
2	5.748	BV	0.245	11372.08691	720.48462	42.1303 ←
3	6.634	VV	0.272	1628.90295	93.72545	6.0346
4	7.962	VV	0.334	2428.44556	112.79986	8.9967
5	11.657	BB	0.576	11502.07129	310.93521	42.6119 ←
Totals :				26992.64648	1240.04919	



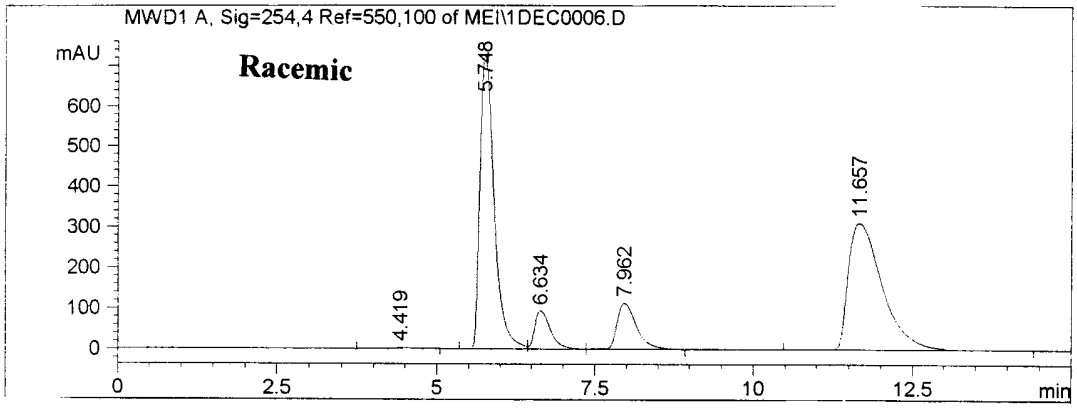
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.415	PV	0.379	68.86162	2.45502	0.3545
2	4.885	VV	0.218	239.64899	16.61662	1.2336
3	5.732	VV	0.246	13084.69824	829.44830	67.3518 ←
4	6.620	VV	0.286	390.24741	20.90193	2.0087
5	7.970	VV	0.339	1689.47485	77.48895	8.6964
6	11.764	BB	0.551	3954.44751	111.66781	20.3550 ←
Totals :				19427.37891	1058.57861	

Determination of enantiomeric excess of epoxide of 14 by HPLC

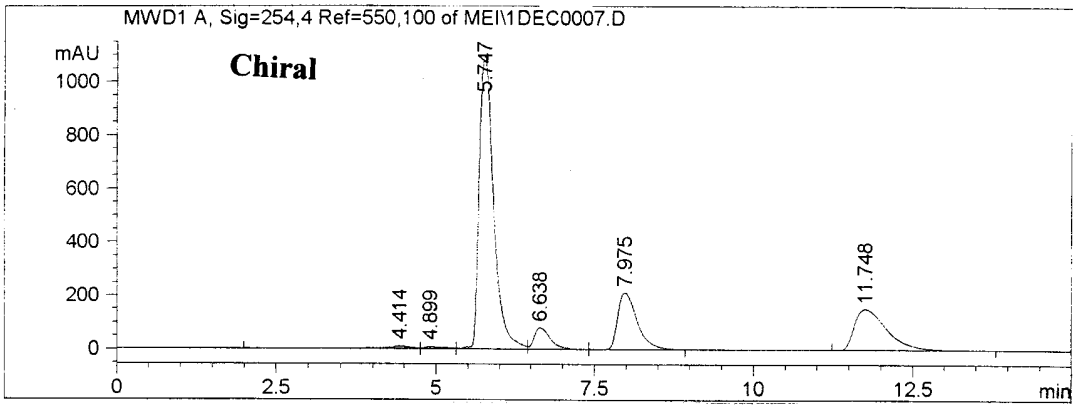
Table 3, entry 6



HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-JK036)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.419	PV	0.388	61.13882	2.10409	0.2265
2	5.748	BV	0.245	11372.08691	720.48462	42.1303 ←
3	6.634	VV	0.272	1628.90295	93.72545	6.0346
4	7.962	VV	0.334	2428.44556	112.79986	8.9967
5	11.657	BB	0.576	11502.07129	310.93521	42.6119 ←
Totals :				26992.64648	1240.04919	

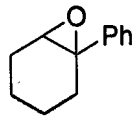


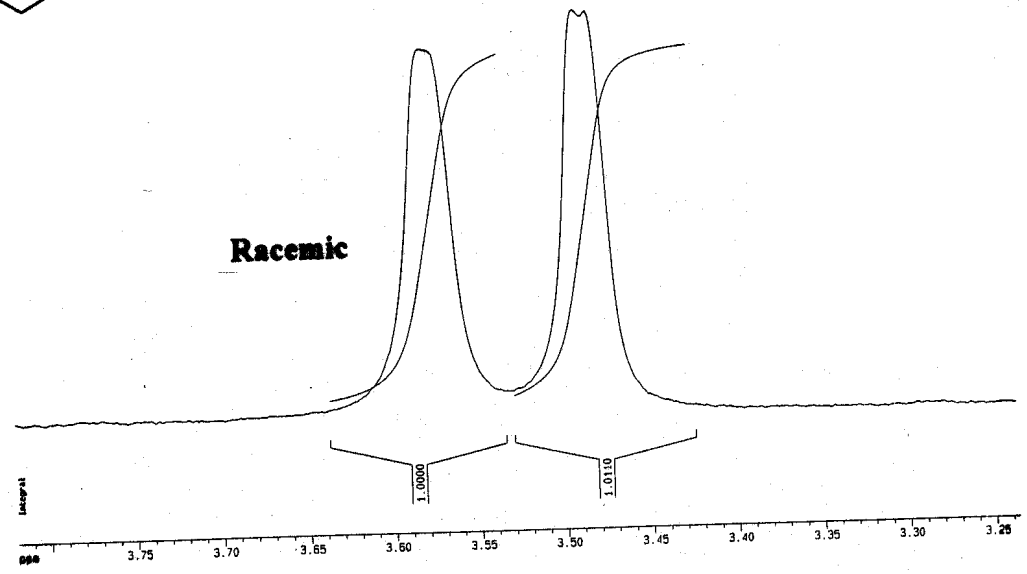
Peak #	RT [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.414	BV	0.410	319.07700	10.41580	1.0876
2	4.899	VV	0.282	170.72032	8.49095	0.5819
3	5.747	VV	0.248	17430.52930	1093.16675	59.4130 ←
4	6.638	VV	0.277	1421.04541	79.80736	4.8437
5	7.975	VV	0.334	4549.32129	212.83951	15.5066
6	11.748	BB	0.559	5447.22949	152.21739	18.5672 ←
Totals :				29337.92187	1556.93774	

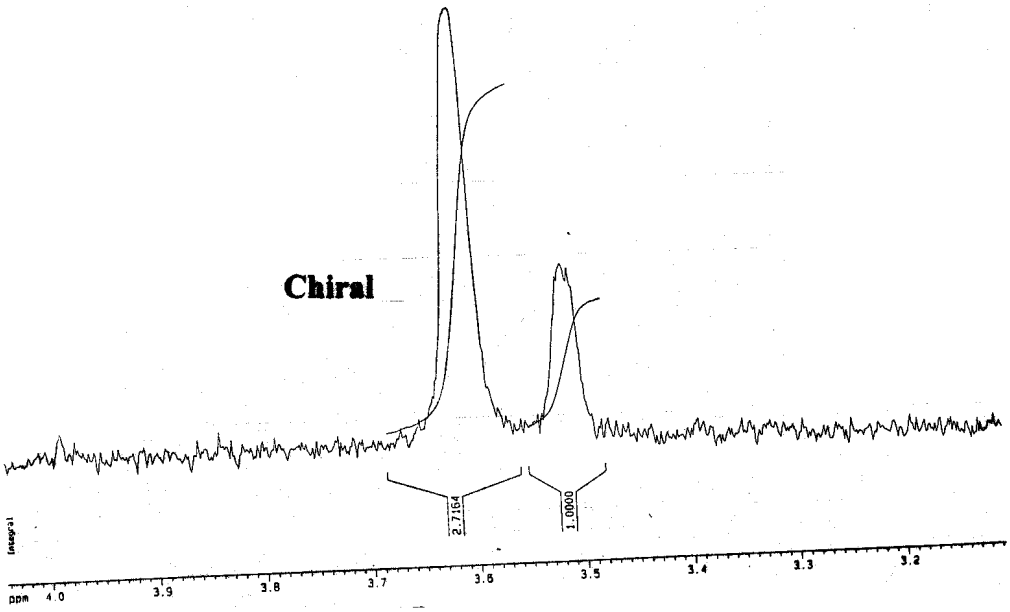
Determination of enantiomeric excess of epoxide of 15 by NMR

Table 3, entry 7

NMR Conditions
Chiral shift reagent: $(hfc)_3Eu(III)$

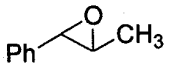




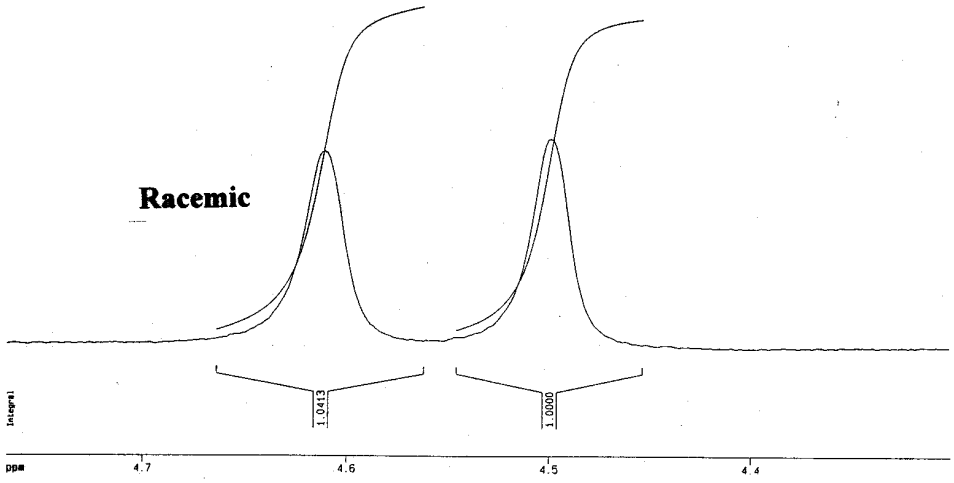


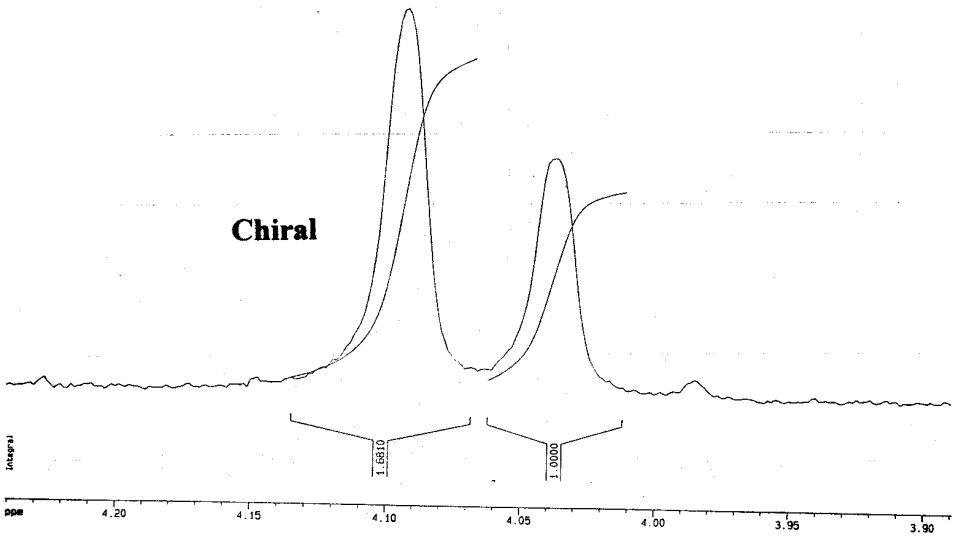
Determination of enantiomeric excess of epoxide of 16 by NMR

Table 3, entry 8



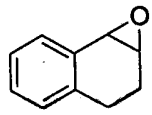
NMR Conditions
Chiral shift reagent: $(hfc)_3Eu(III)$



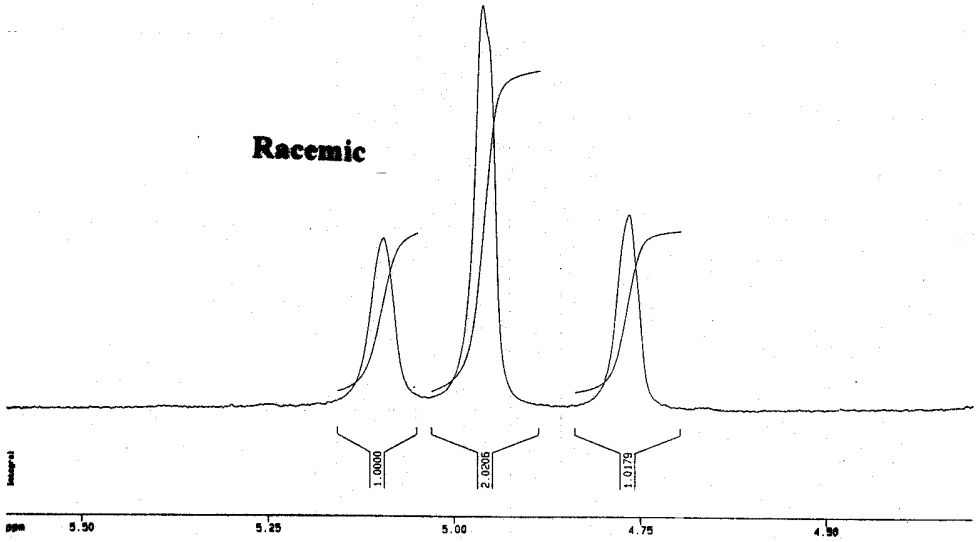


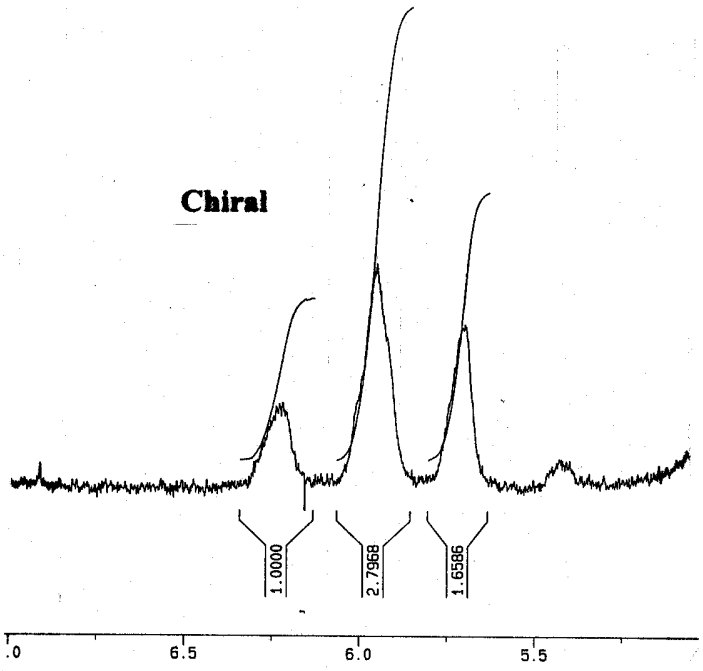
Determination of enantiomeric excess of epoxide of 17 by NMR

Table 3, entry 9



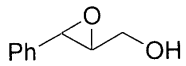
NMR Conditions
Chiral shift reagent: $(hfc)_3Eu(III)$



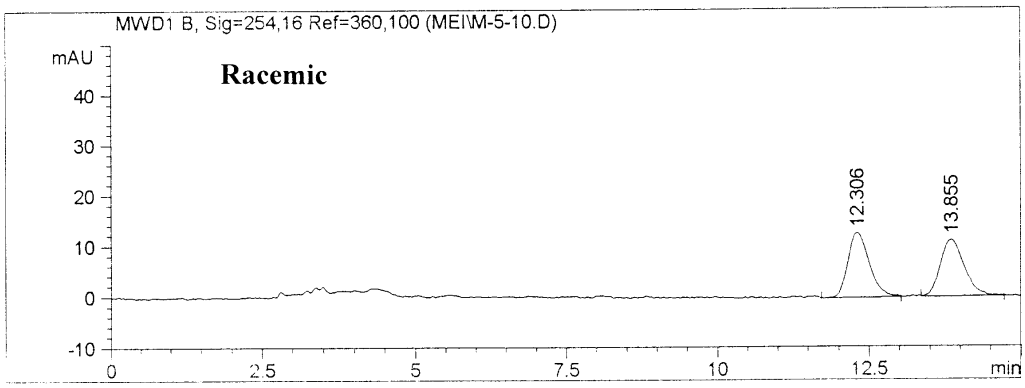


Determination of enantiomeric excess of epoxide of 18 by HPLC

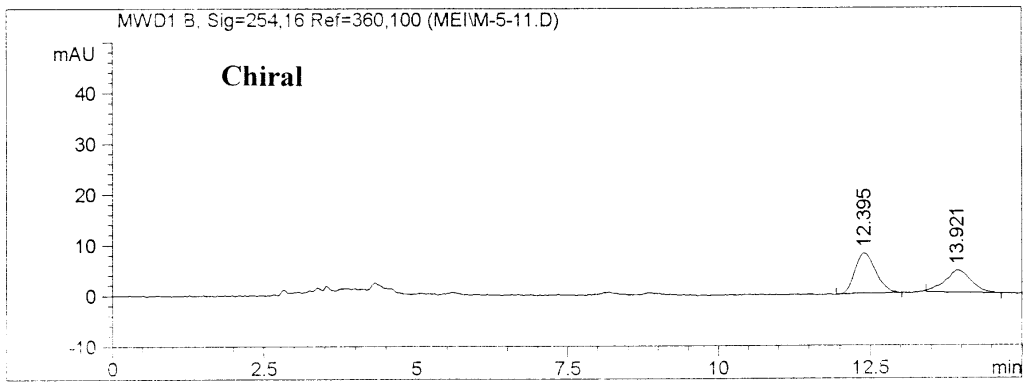
Table 3, entry 10



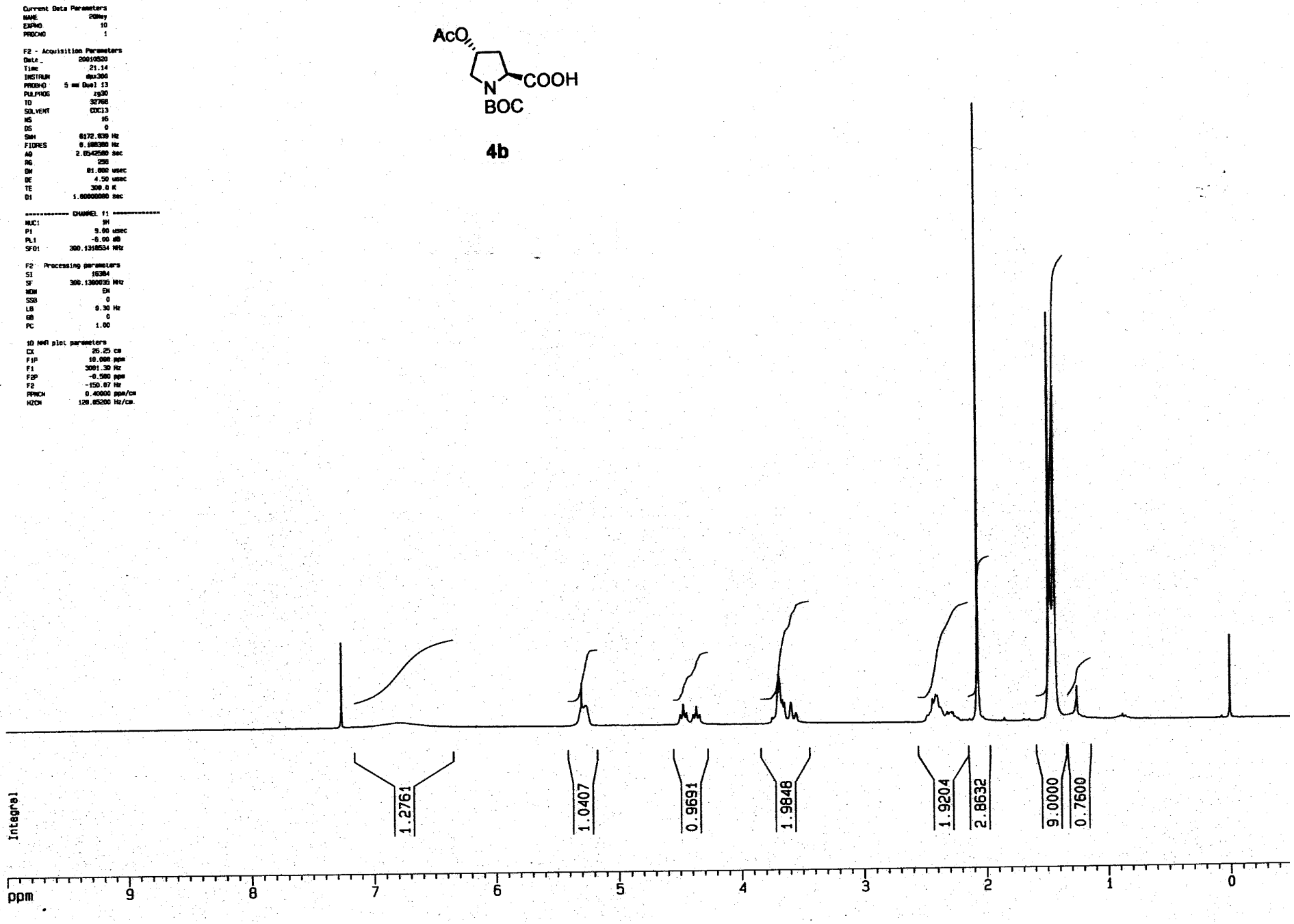
HPLC Conditions
Column: Chiralcel OD (Lot No. OD00CE-AH045)
Solvents: n-hexane/IPA (90.0/10.0)
Flow rate: 1.0 mL/min
Detection: UV 254nm



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.306	VB	0.3502	314.34644	12.81364	50.5979
2	13.855	VB	0.3981	306.91772	11.18590	49.4021
Totals :				621.26416	23.99954	



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.395	PB	0.3581	192.18394	8.17992	58.7000
2	13.921	BP	0.3655	135.21628	4.46206	41.3000
Totals :				327.40022	12.64199	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PROCNO 1
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SOLVENT CDCl3
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FIDRES 0.188380 Hz
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DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
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PL1 -6.00 dB
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F2 - Processing parameters
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SF 300.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
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HZCM 120.05200 Hz/cm
AcO
COOH
N
BOC
4b
Integral
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8
7
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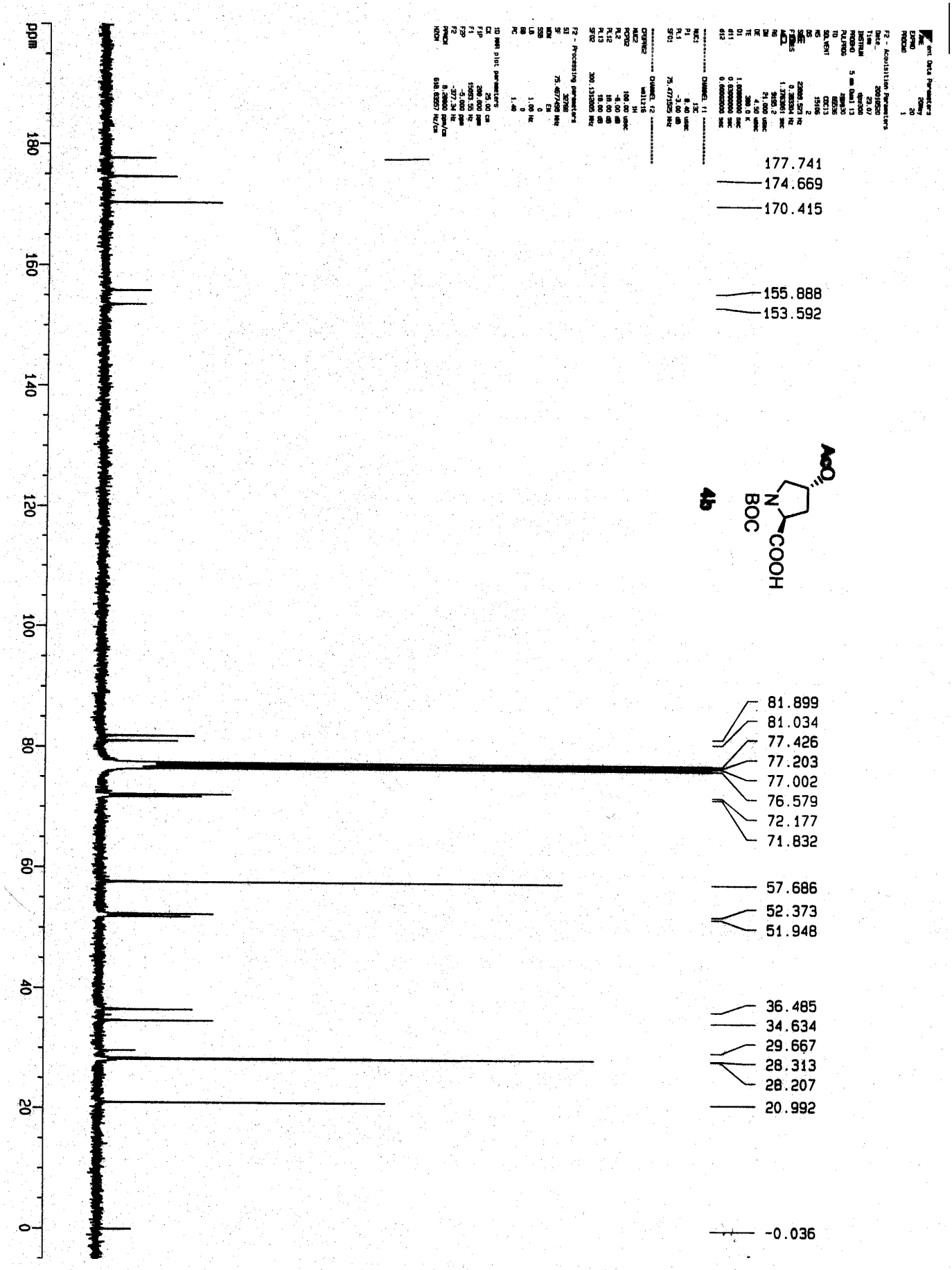
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4b
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60
40
20
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PROCNO                1

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TD                32768
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RG                  181
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TE               300.0 K
D1           1.00000000 sec

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PL1              -6.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
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GB                    0
PC                 1.00

1D NMR plot parameters
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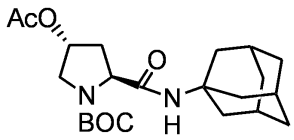

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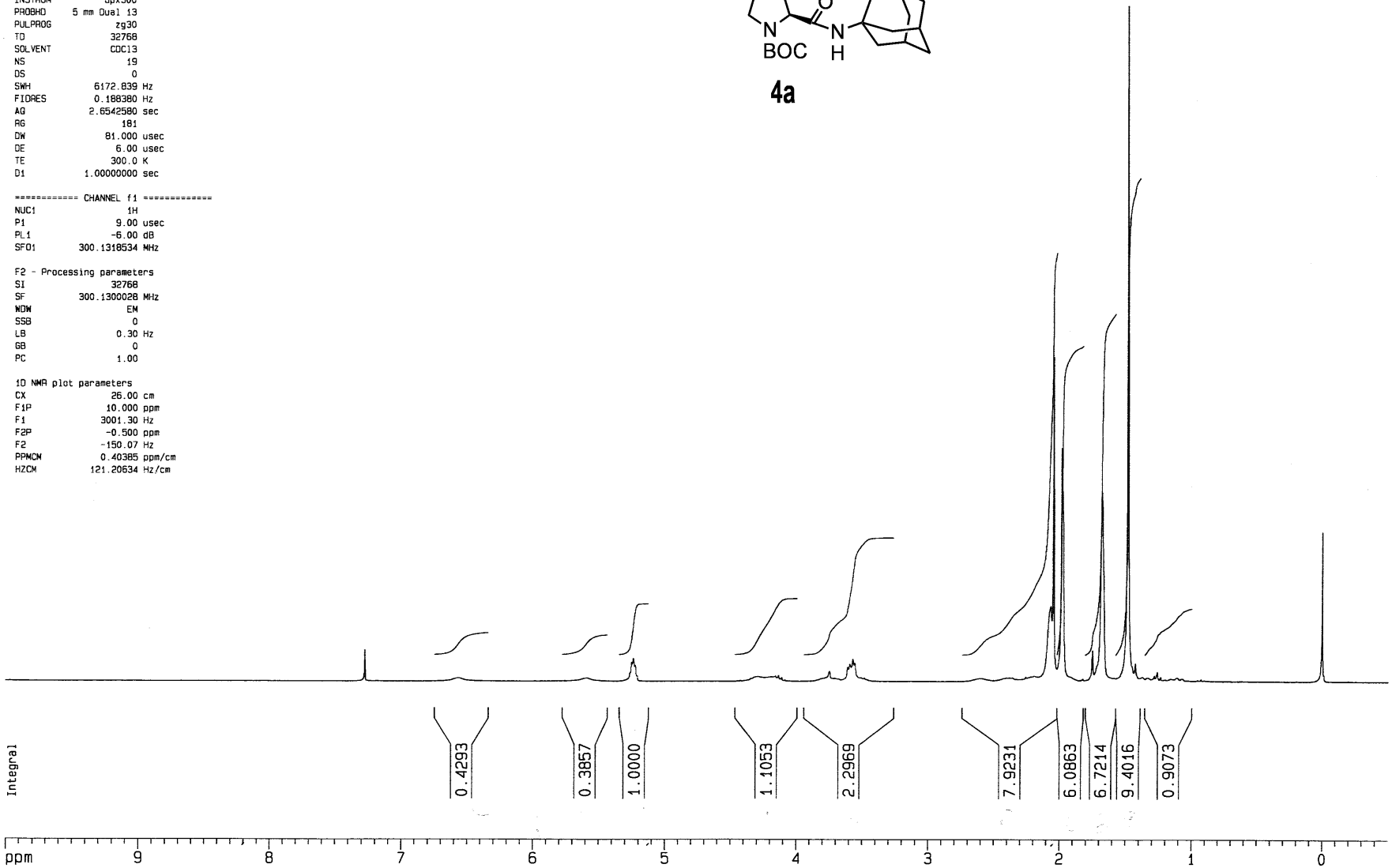
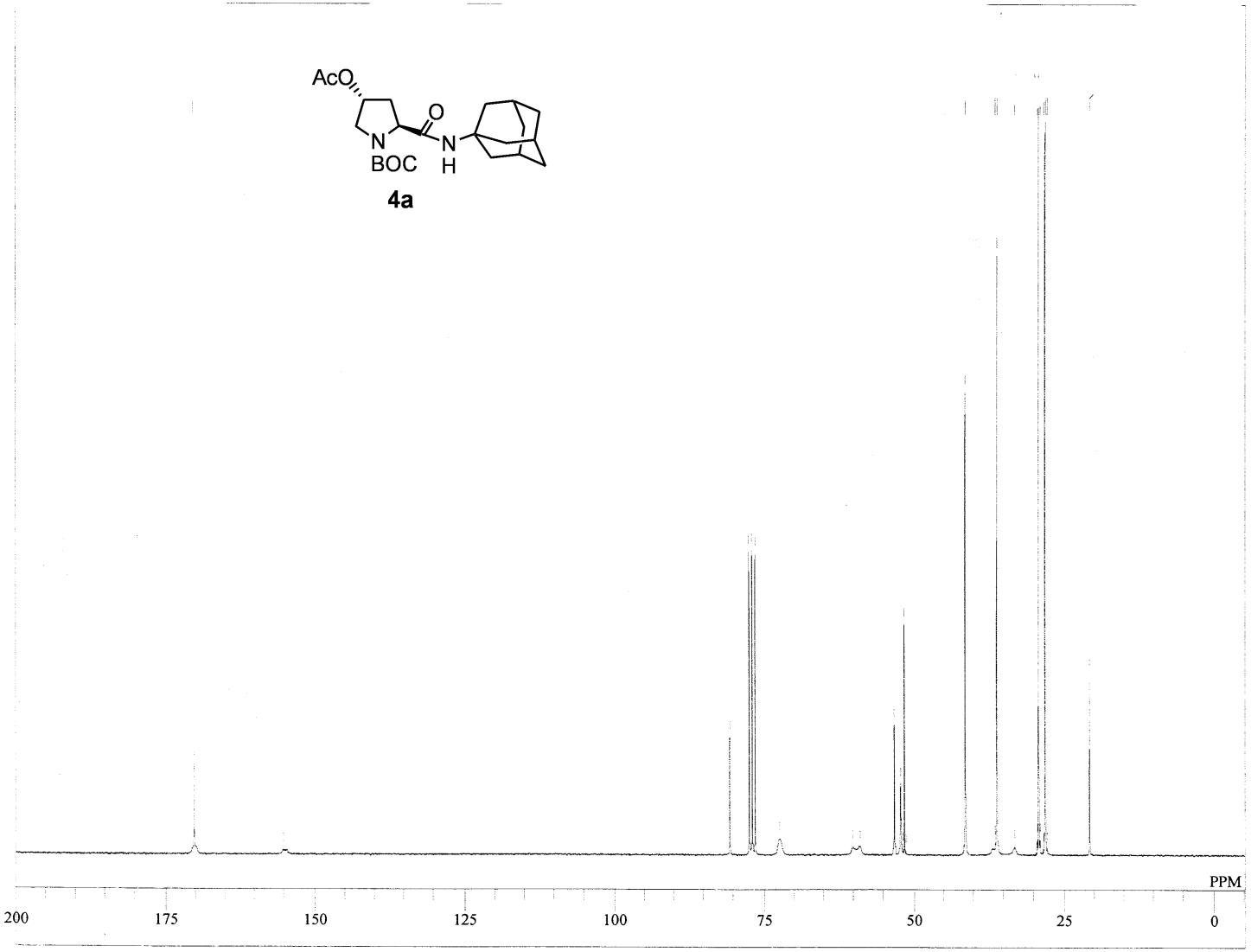


march 9

4a



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DELAY 21.9 us
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PD 3.500 sec
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T3 90.00
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LKPHS 125
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FILDC
FILDF

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FIDRES         0.188380 Hz
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RG                 90.5
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DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec

============ CHANNEL f1 =============
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P1                9.00 usec
PL1              -6.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
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WDW                  EM
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GB                    0
PC                 1.00

1D NMR plot parameters
CX                26.00 cm
F1P               10.000 ppm
F1               3001.30 Hz
F2P               -0.500 ppm
F2               -150.07 Hz
PPMCM            0.40385 ppm/cm
HZCM           121.20634 Hz/cm
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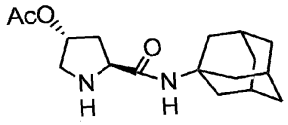

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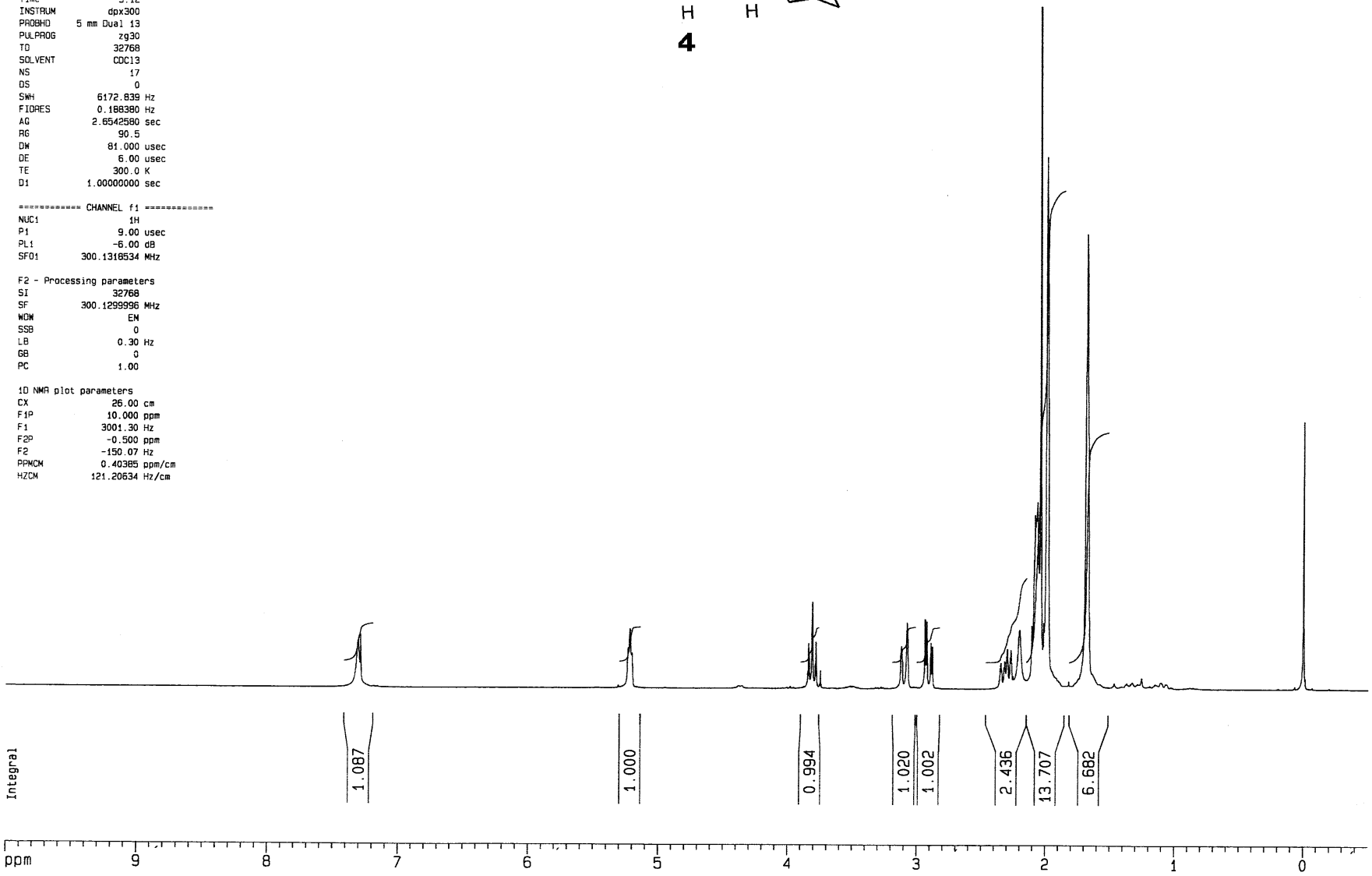

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PROCNO               1

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TD                65536
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d12          0.00002000 sec

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1D NMR plot parameters
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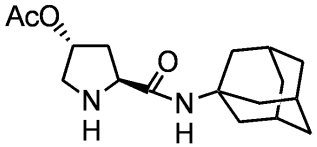
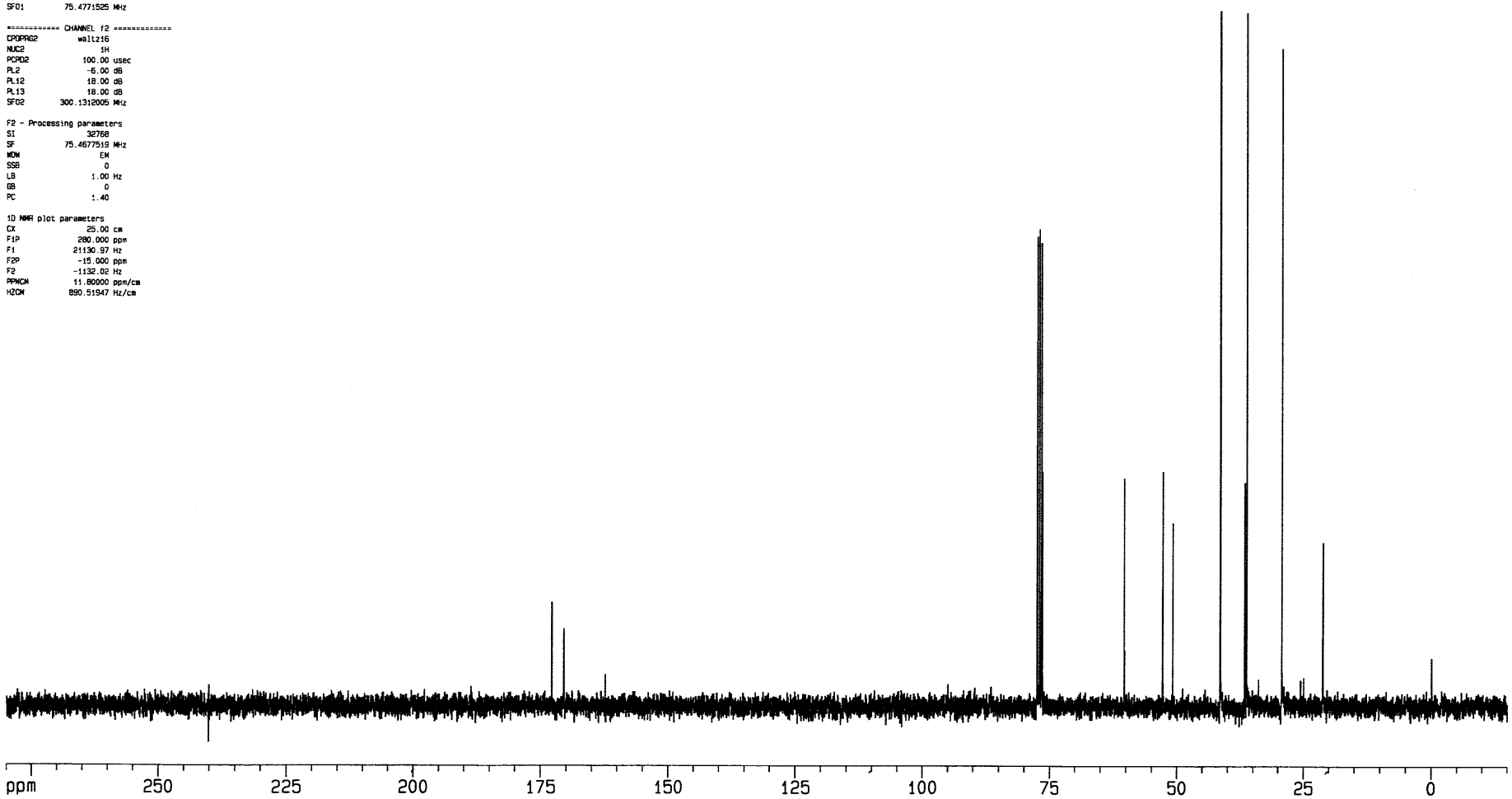
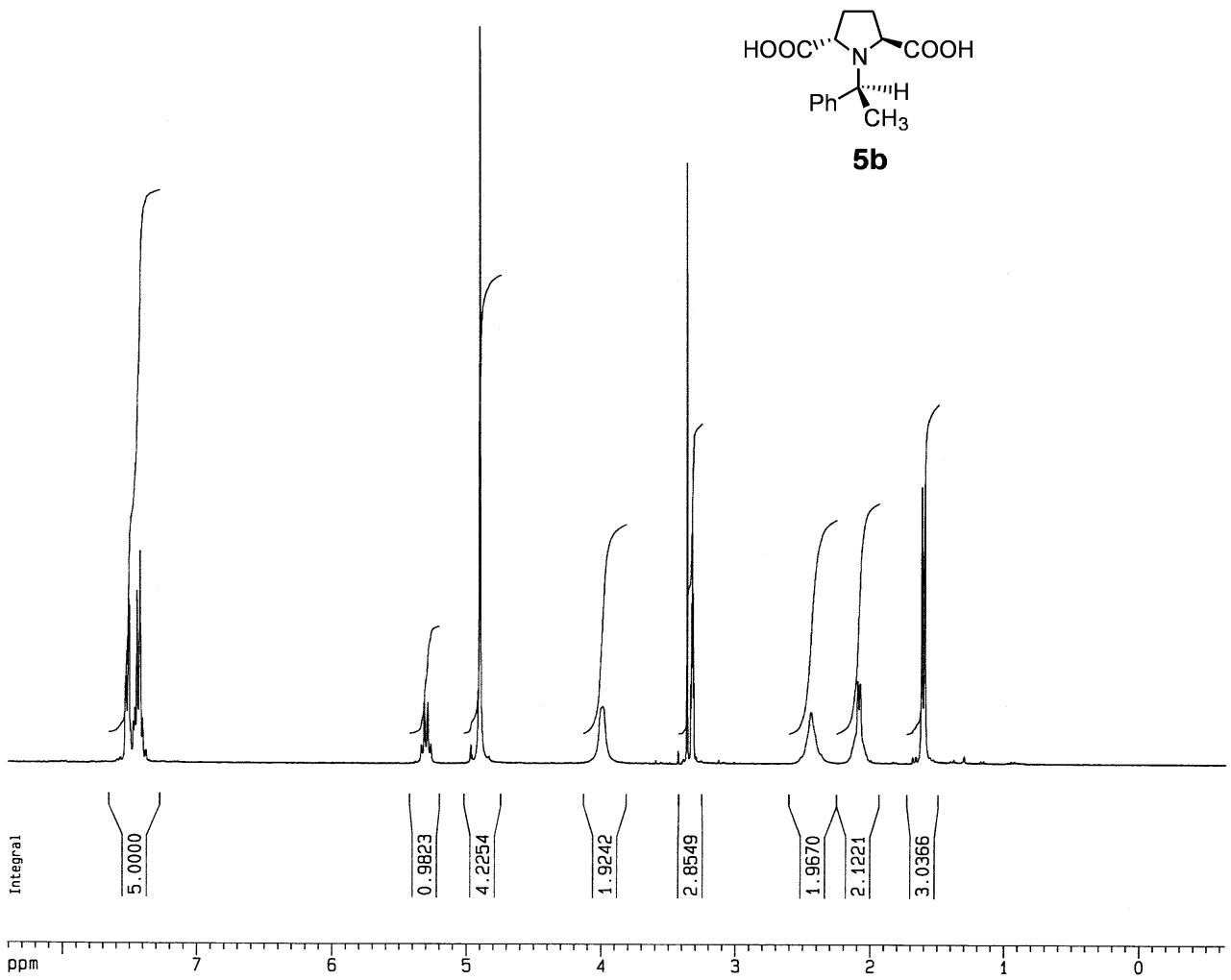


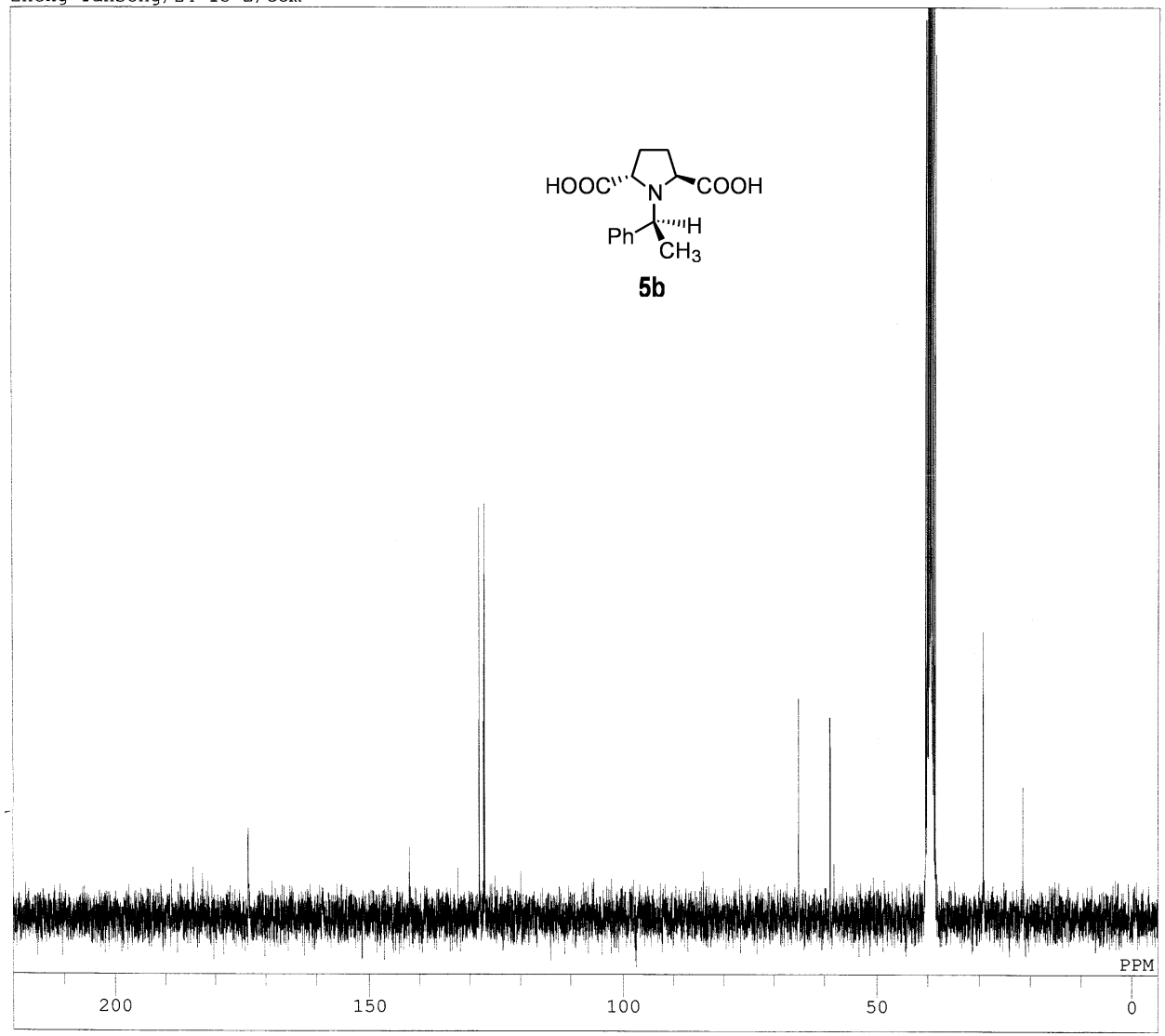
4



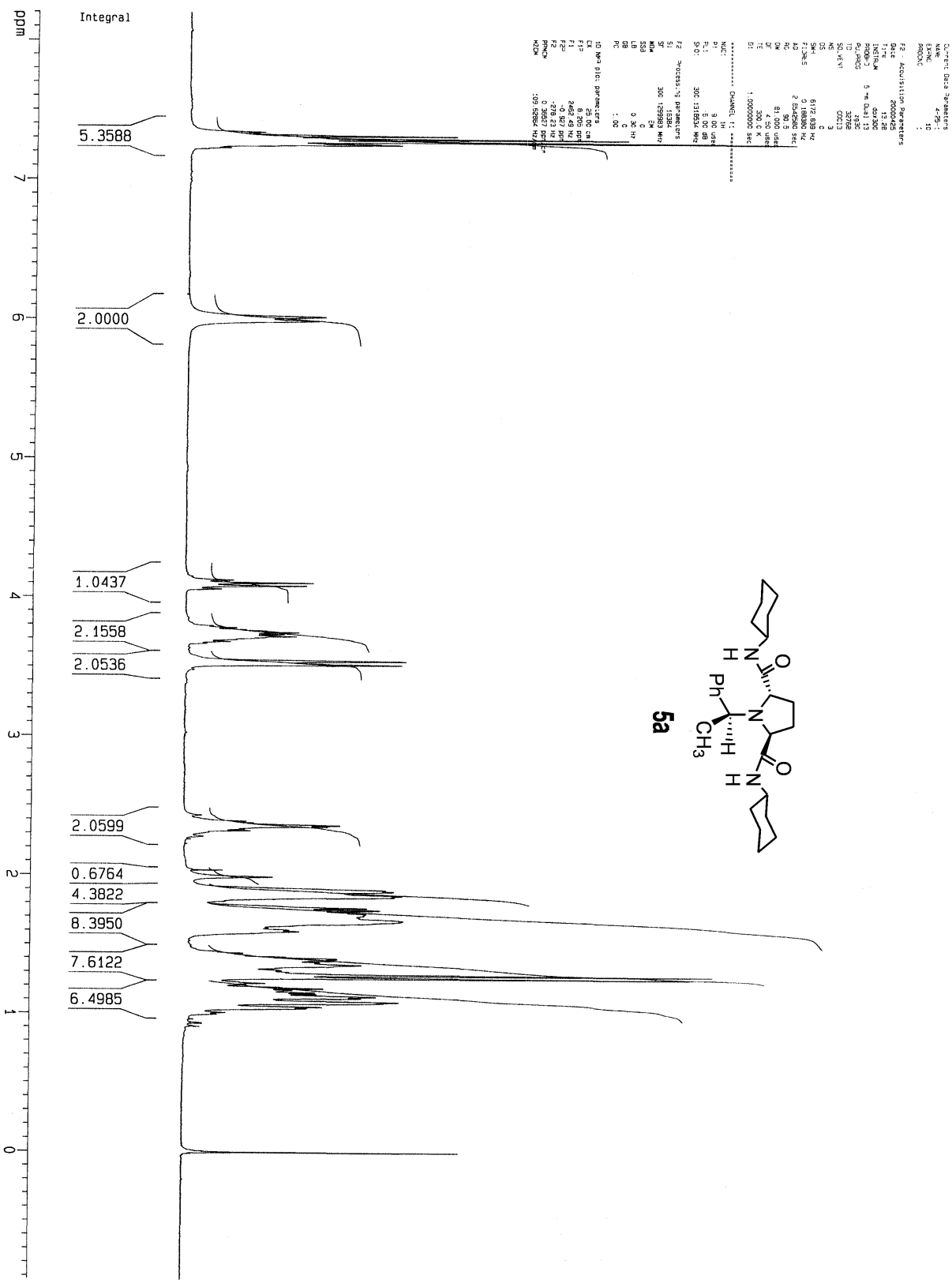


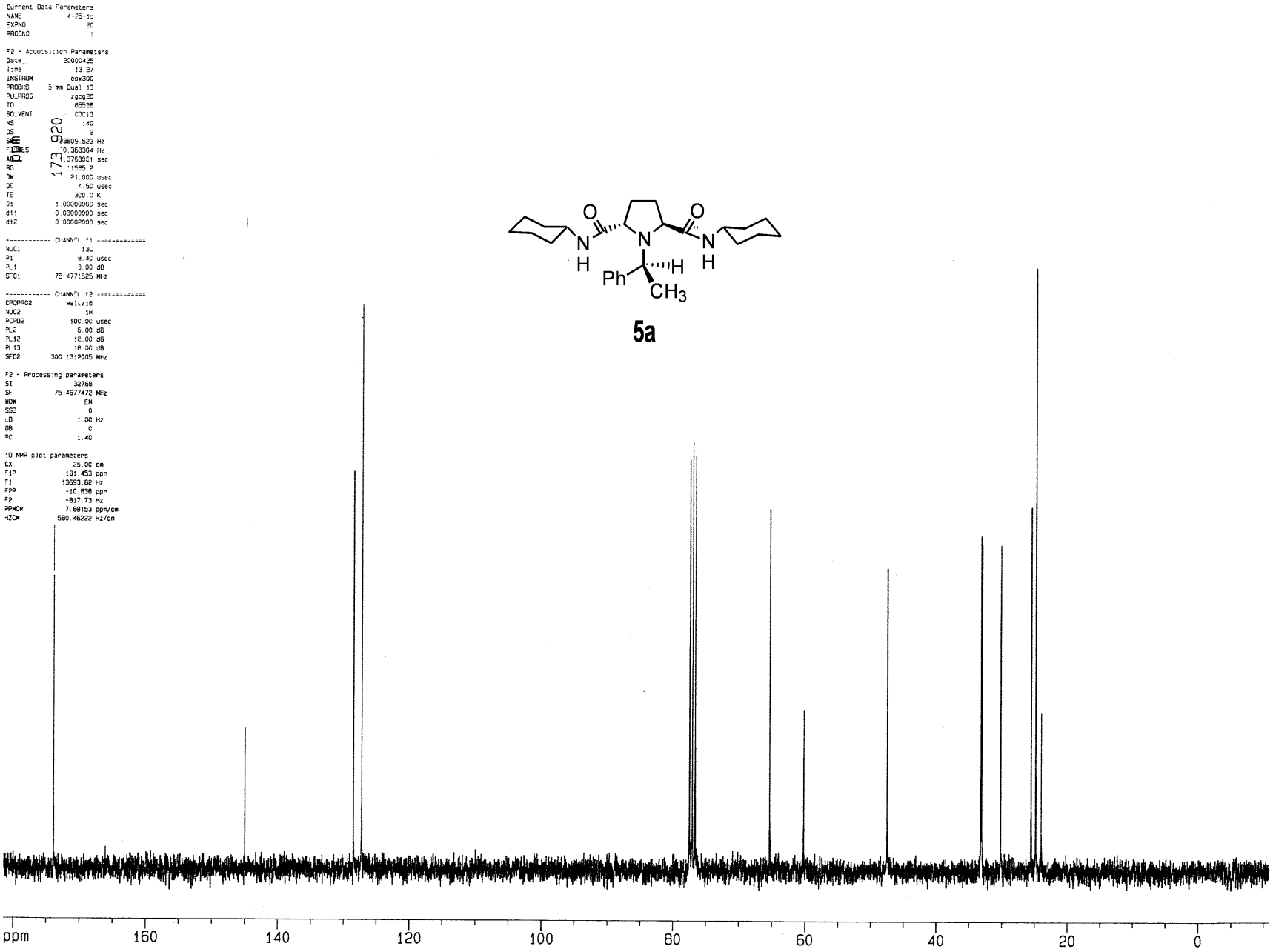

```
Current Data Parameters
NAME              7-11-1
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20000711
Time               15.22
INSTRUM          dpx300
PROBHD     5 mm Dual 13
PULPROG            zg30
TD                32768
SOLVENT            MeOH
NS                    16
DS                     0
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ           2.6542580 sec
RG                   362
DW               81.000 usec
DE                 6.00 usec
TE                300.0 K
D1           1.00000000 sec

============ CHANNEL f1 =============
NUC1                  1H
P1                 9.00 usec
PL1               -6.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300011 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               8.406 ppm
F1              2523.02 Hz
F2P              -0.641 ppm
F2              -192.25 Hz
PPMCM           0.45235 ppm/cm
HZCM          135.76340 Hz/cm
```

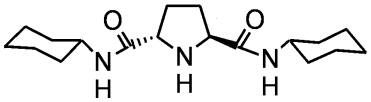
Zheng Yansong/z4-13-2/Com



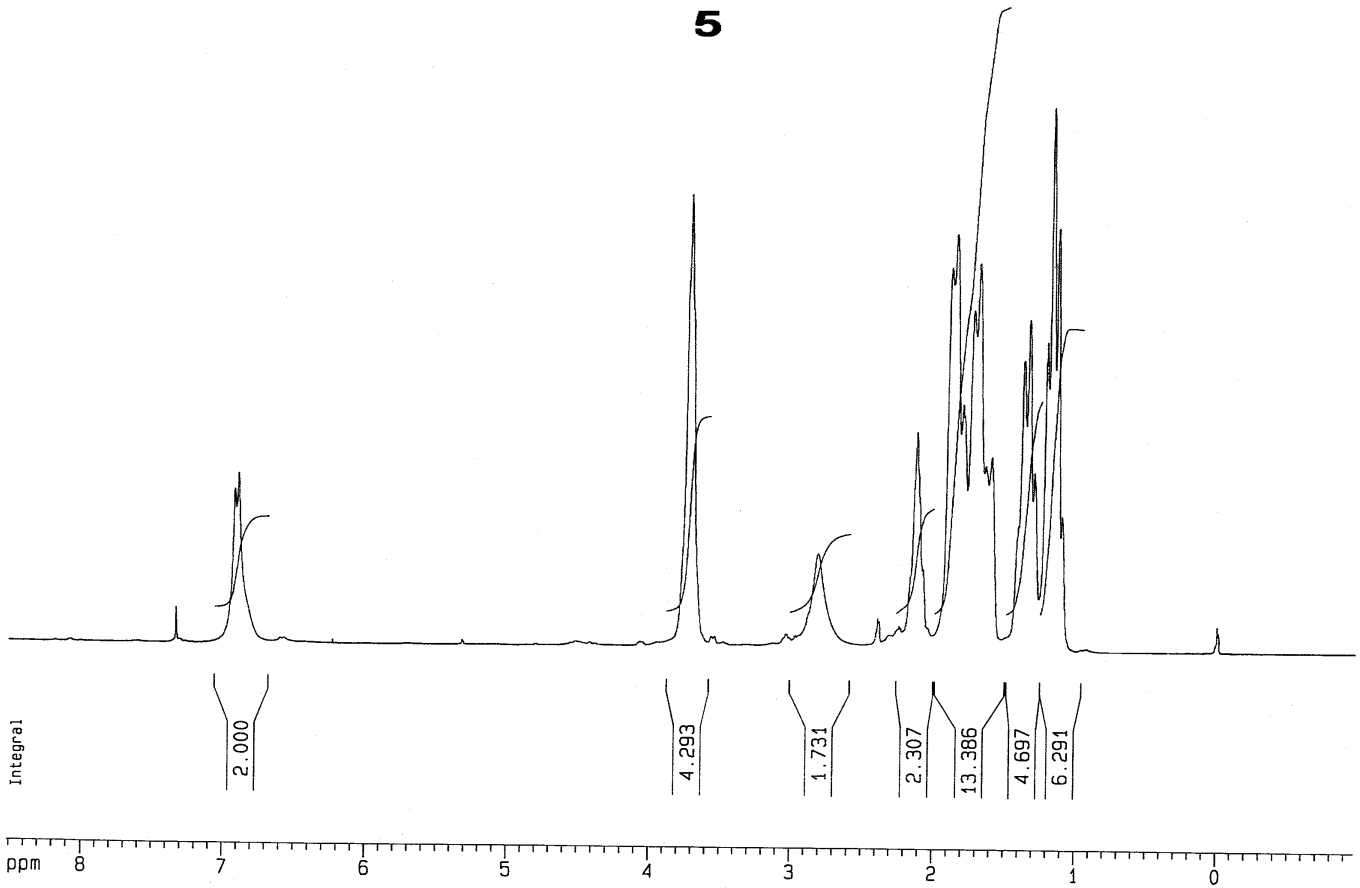

```
MENUF   SGBCM
OBNUC   13C
OFR          67.80 MHz
OBSET       135.00 KHz
OBFIN       5200.0 Hz
PW1            4.2 us
DEADT         30.7 us
PREDL       0.2000 ms
IWT            1.0 sec
POINT        32768
SPO          32768
TIMES        50000
DUMMY            1
FREQU      18315.0 Hz
FLT           9150 Hz
DELAY         21.9 us
ACQTM        1.789 sec
PD           3.000 sec
ADBIT        16.00
RGAIN           26
BF            1.00 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD   SGBCM
EXPCM   "B"SGBCMA:Single.pulse.Complet
IRNUC   1H
IFR         270.05 MHz
IRSET       112.00 KHz
IRFIN       5800.0 Hz
IRRPW           45 us
IRATN           20
DFILE   C:\WINNMR95\COMMON\_DEFAULT.AI
SF      TH5
LKSET        64.40 KHz
LKFIN         76.0 Hz
LKLEV          170
LGAIN           19
LKPHS          110
LKSIG          381
CSPED           11 Hz
FILDC
FILDF
```


Current Data Parameters
NAME
EXPNO 4-25-
PROCNO 10
Acquisition Parameters
Date 20000425
Time 13:28
INSTRUM spect
PROBHD 5 mm QNP 1H/19F
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3
DS 3
SWH 6172.839 Hz
FIDRES 0.188386 Hz
AQ 2.6542560 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 6.00 dB
SFO1 300.1318524 MHz
F2 - Processing parameters
SI 131072
SF 300.1299883 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.00 cm
F1P 8.200 ppm
F1 2459.49 Hz
F2P -0.927 ppm
F2 -278.23 Hz
PPMCM 0.30527 ppm/cm
HZCM 169.92954 Hz/cm
Integral
ppm
5.3588
2.0000
1.0437
2.1558
2.0536
2.0599
0.6764
4.3822
8.3950
7.6122
6.4985
5a

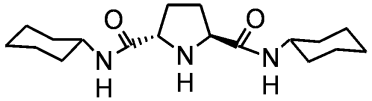

Current Data Parameters
NAME 4-25-1c
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20000425
Time 13.37
INSTRUM cox300
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763081 sec
RG 11585.2
DW 21.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
173.920
========== CHANNEL f1 ==========
NUC1 13C
P1 8.40 usec
PL1 -3.00 dB
SFO1 75.4771525 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 6.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677472 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 25.00 cm
F1P 181.453 ppm
F1 13693.82 Hz
F2P -10.836 ppm
F2 -817.73 Hz
PPMCM 7.69153 ppm/cm
HZCM 580.46222 Hz/cm
O
N
H
O
N
H
N
Ph
H
CH3
5a
ppm
160
140
120
100
80
60
40
20
0



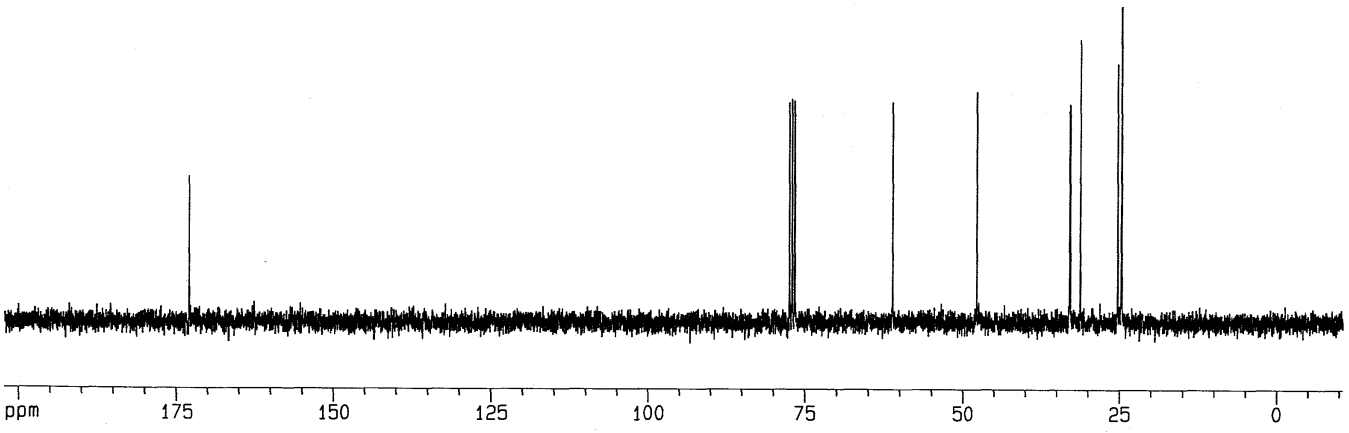
5




```
Current Data Parameters
NAME            11-25-1
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20001126
Time               5.15
INSTRUM          dpx300
PROBHD       5 mm BBO

PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   13
DS                    0
SWH           6172.839 Hz
FIDRES        0.188380 Hz
AQ           2.6542580 sec
RG                45.3
DW              81.000 usec
DE               6.00 usec
TE              300.0 K
D1          1.00000000 sec

============ CHANNEL f1 =============
NUC1                1H
P1               9.00 usec
PL1             -6.00 dB
SFO1       300.1318534 MHz

F2 - Processing parameters
SI                32768
SF         300.1299834 MHz
WDW                 EM
SSB                  0
LB               0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
F1P               8.509 ppm
F1             2553.75 Hz
F2P              -0.972 ppm
F2             -291.75 Hz
PPMCM          0.47404 ppm/cm
HZCM         142.27483 Hz/cm
```



5




```
Current Data Parameters
NAME            11-25-1c
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20001126
Time                5.17
INSTRUM           dpx300
PROBHD        5 mm BBO

PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    15
DS                     0
SWH            18832.393 Hz
FIDRES          0.574719 Hz
AQ             0.8700404 sec
RG                 13004
DW                26.550 usec
DE                  6.00 usec
TE                 300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                 8.40 usec
PL1               -3.00 dB
SFO1          75.4760200 MHz

============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677584 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               202.129 ppm
F1              15254.20 Hz
F2P              -10.561 ppm
F2               -797.03 Hz
PPMCM           10.63450 ppm/cm
HZCM           802.56177 Hz/cm
```