Isomenthyl-derived 1,3-diol ligands catalyzing an asymmetric aldol reaction identified through parallel synthesis and screening

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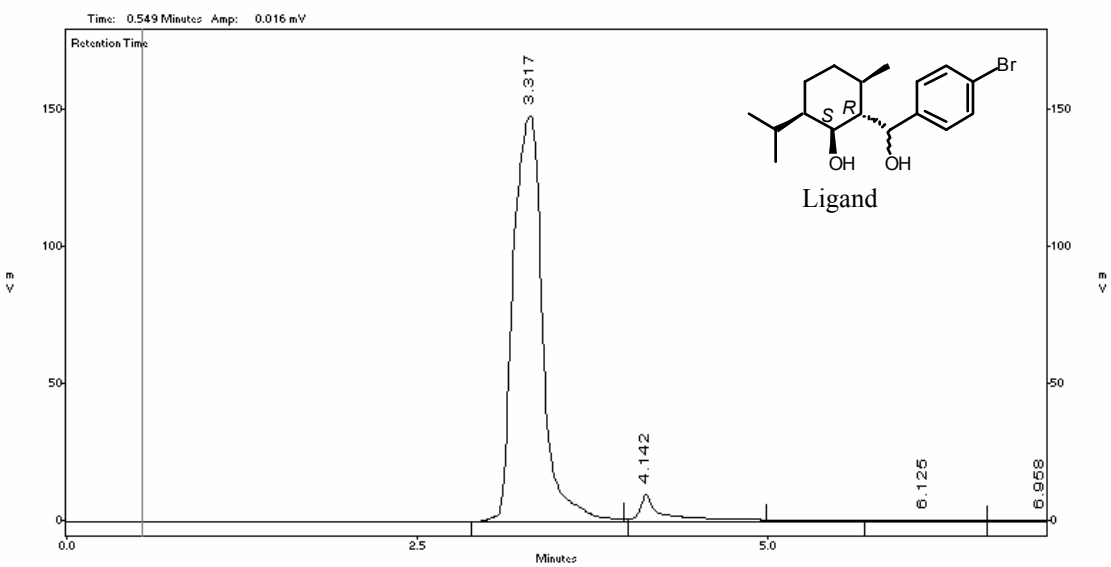
Contents:

Entry 1



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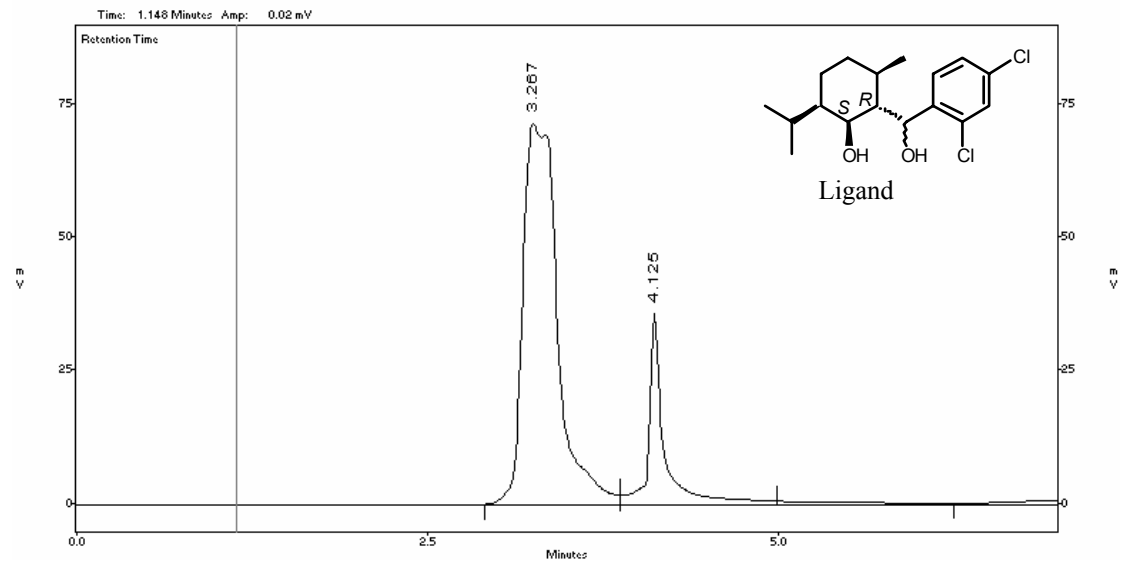

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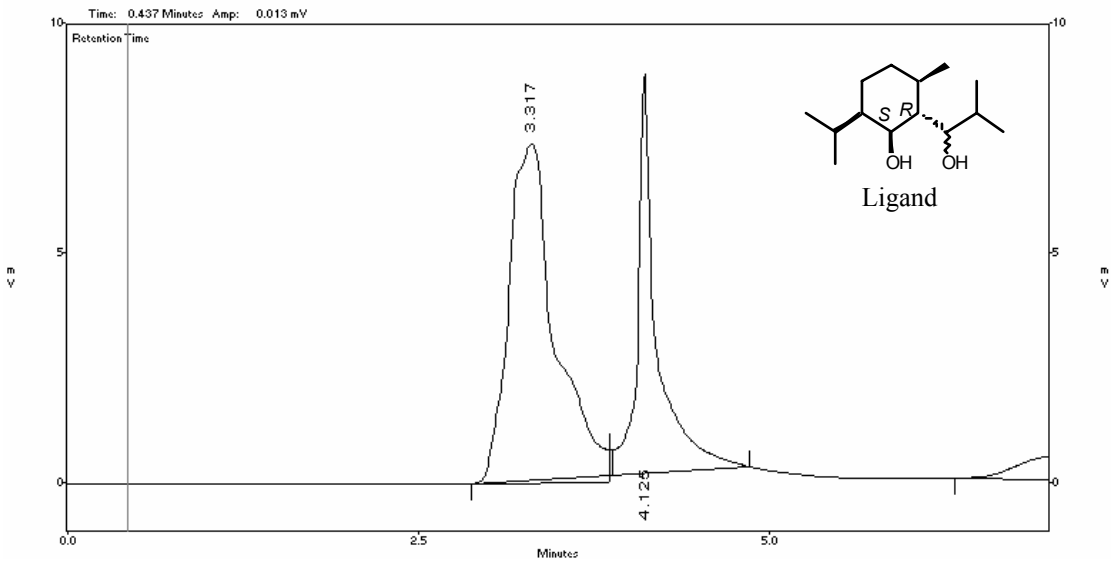

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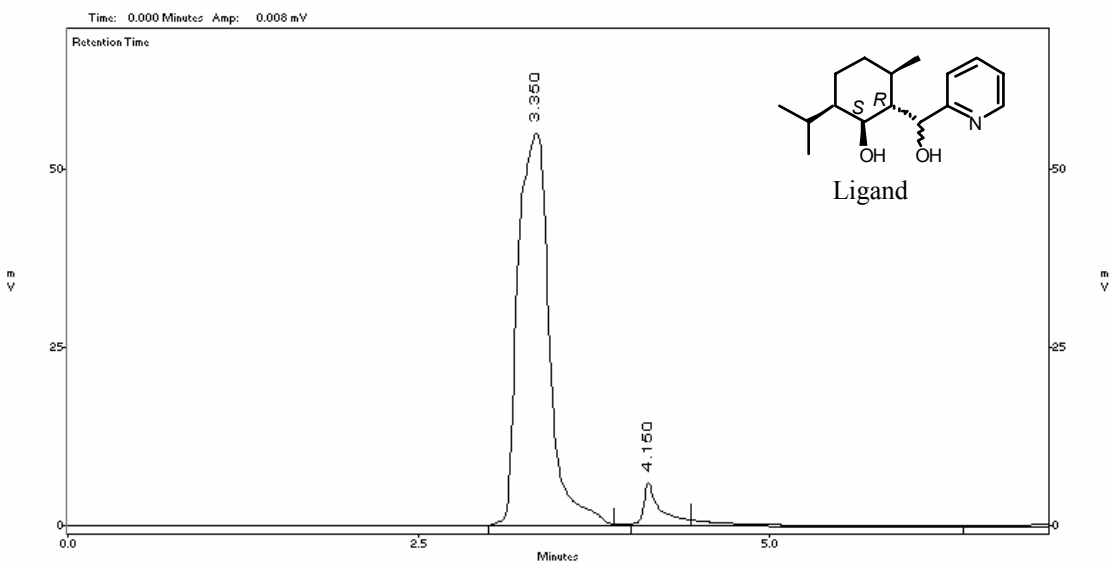

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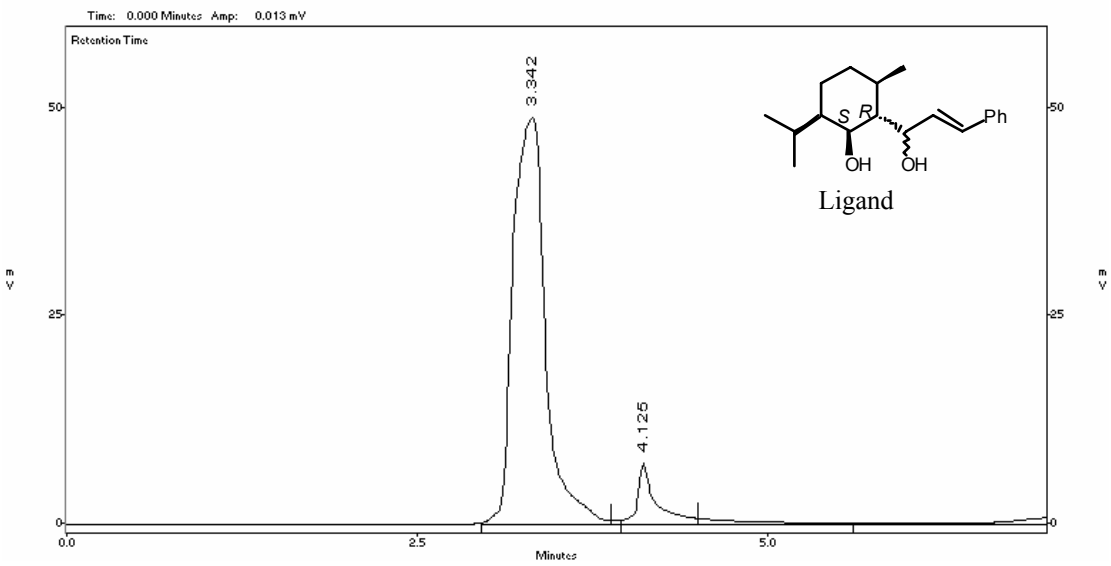

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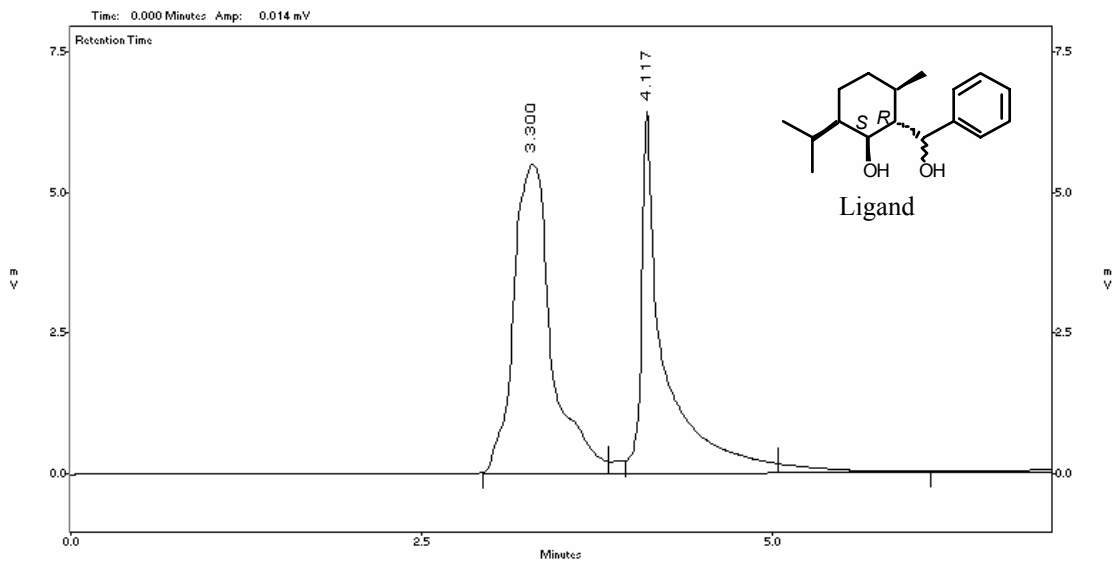

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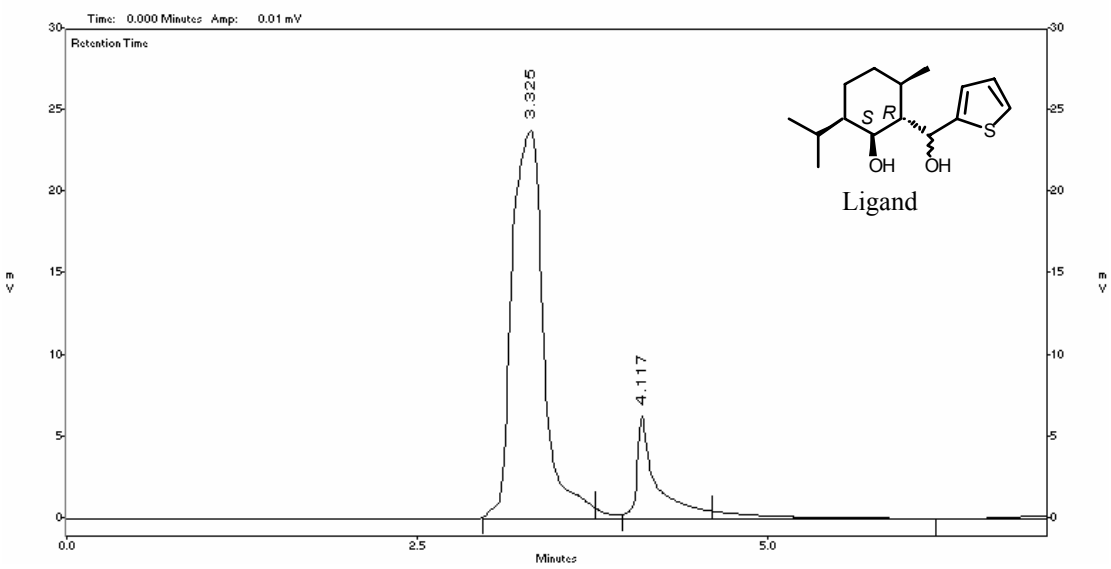

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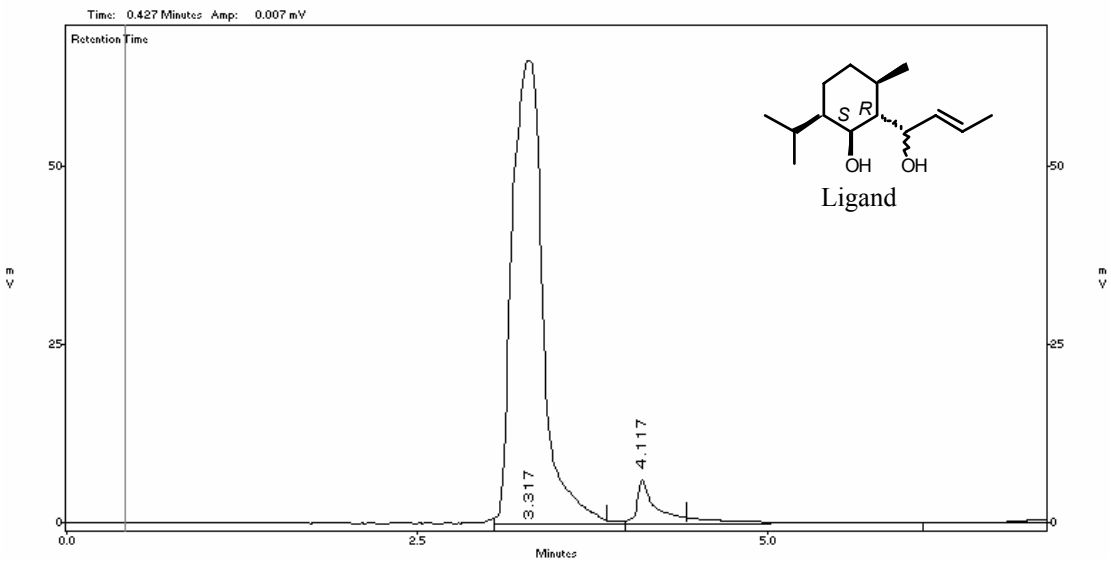

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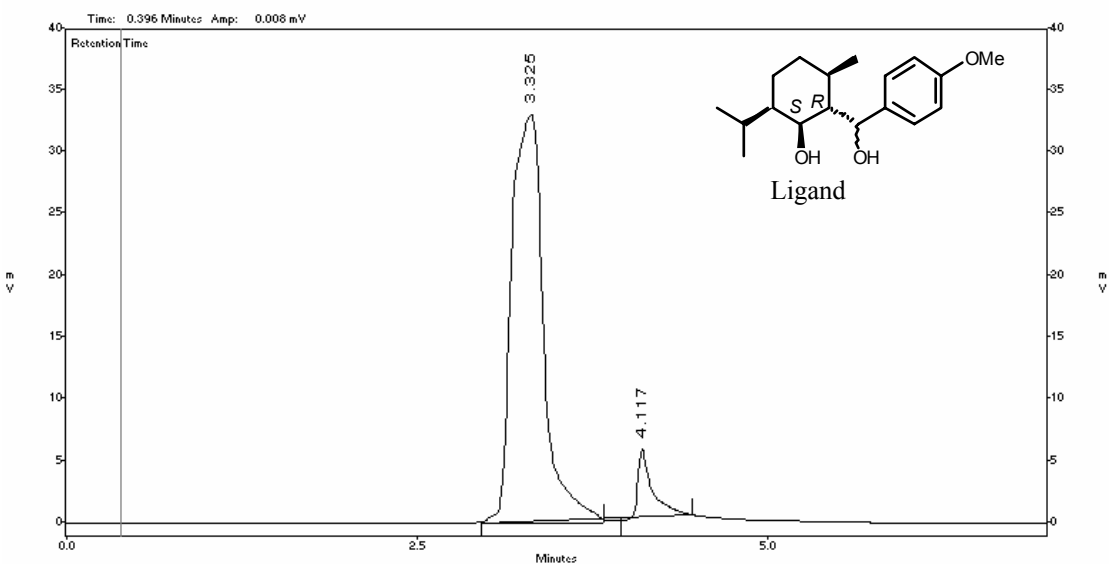

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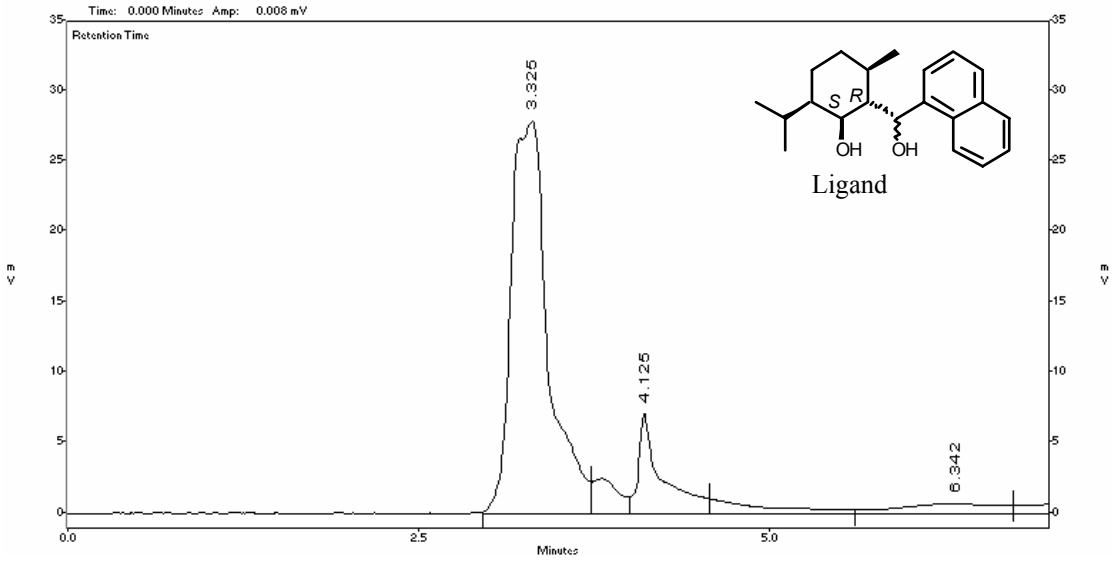

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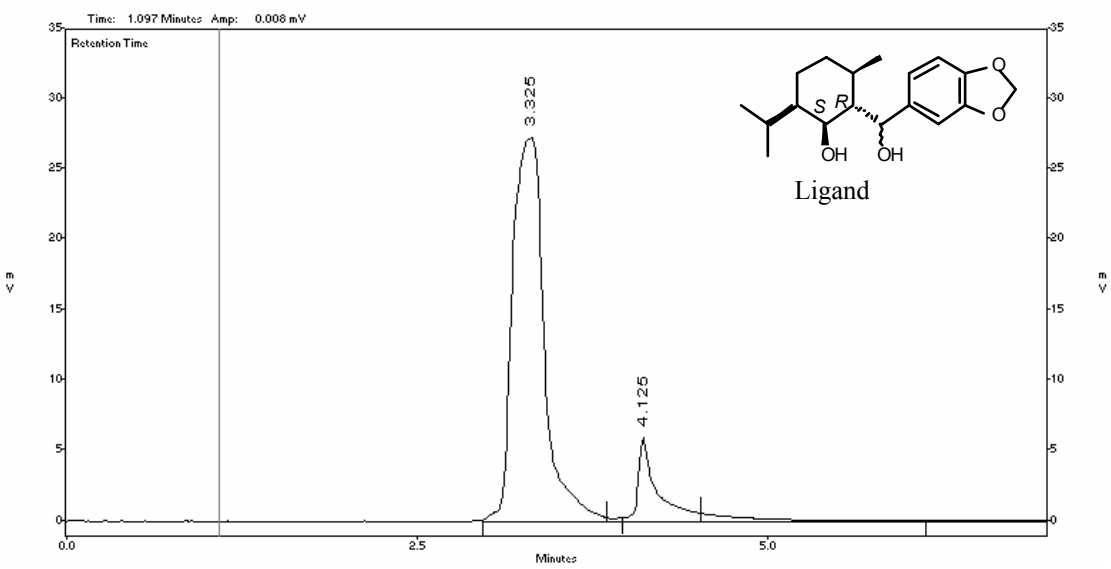
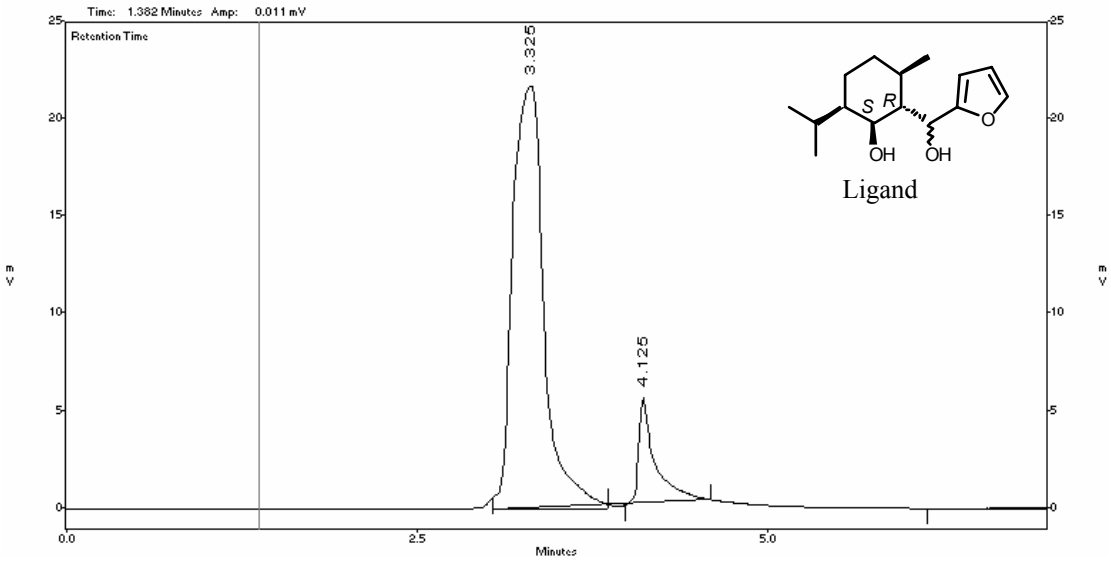


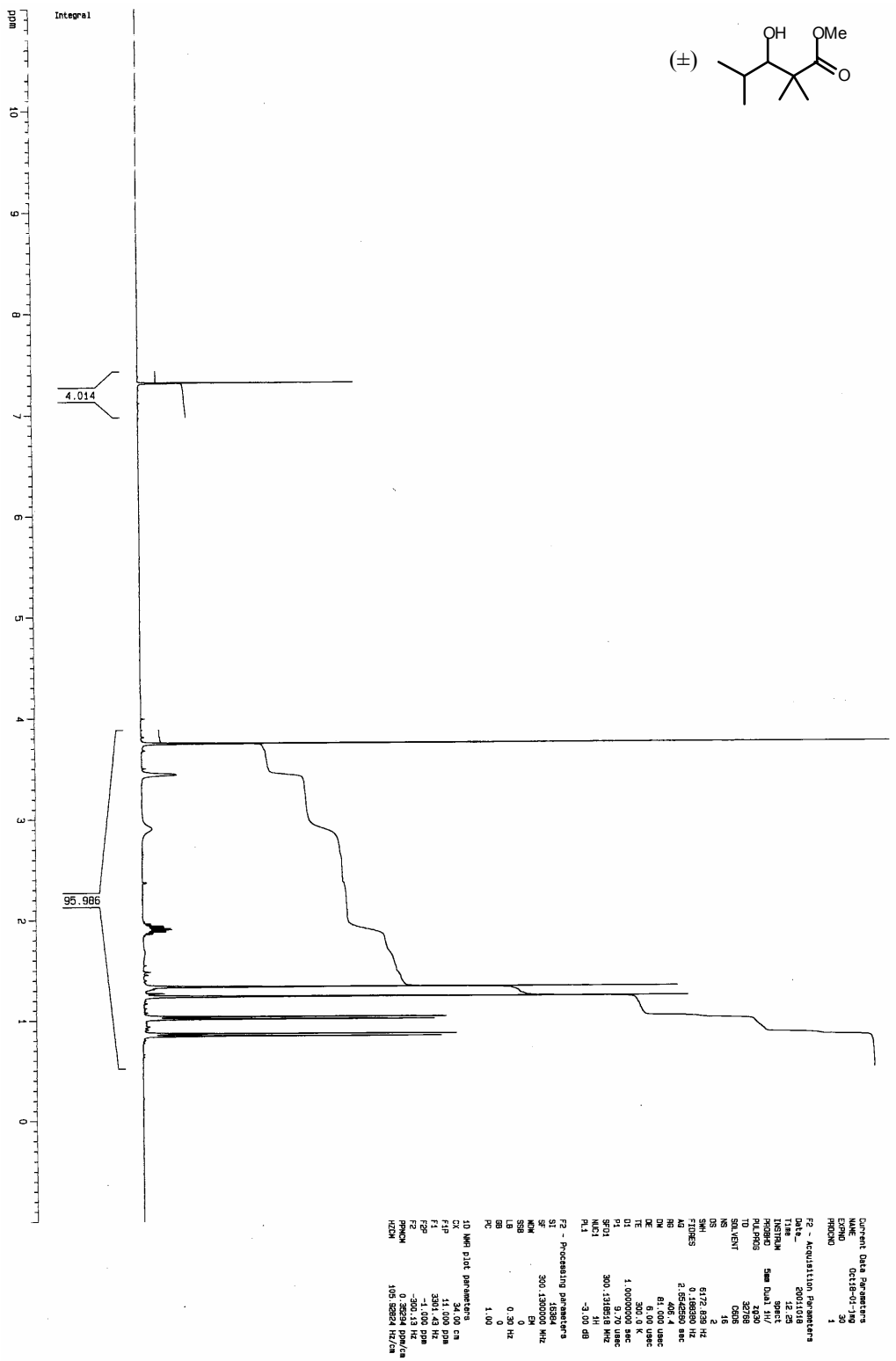
Ligand

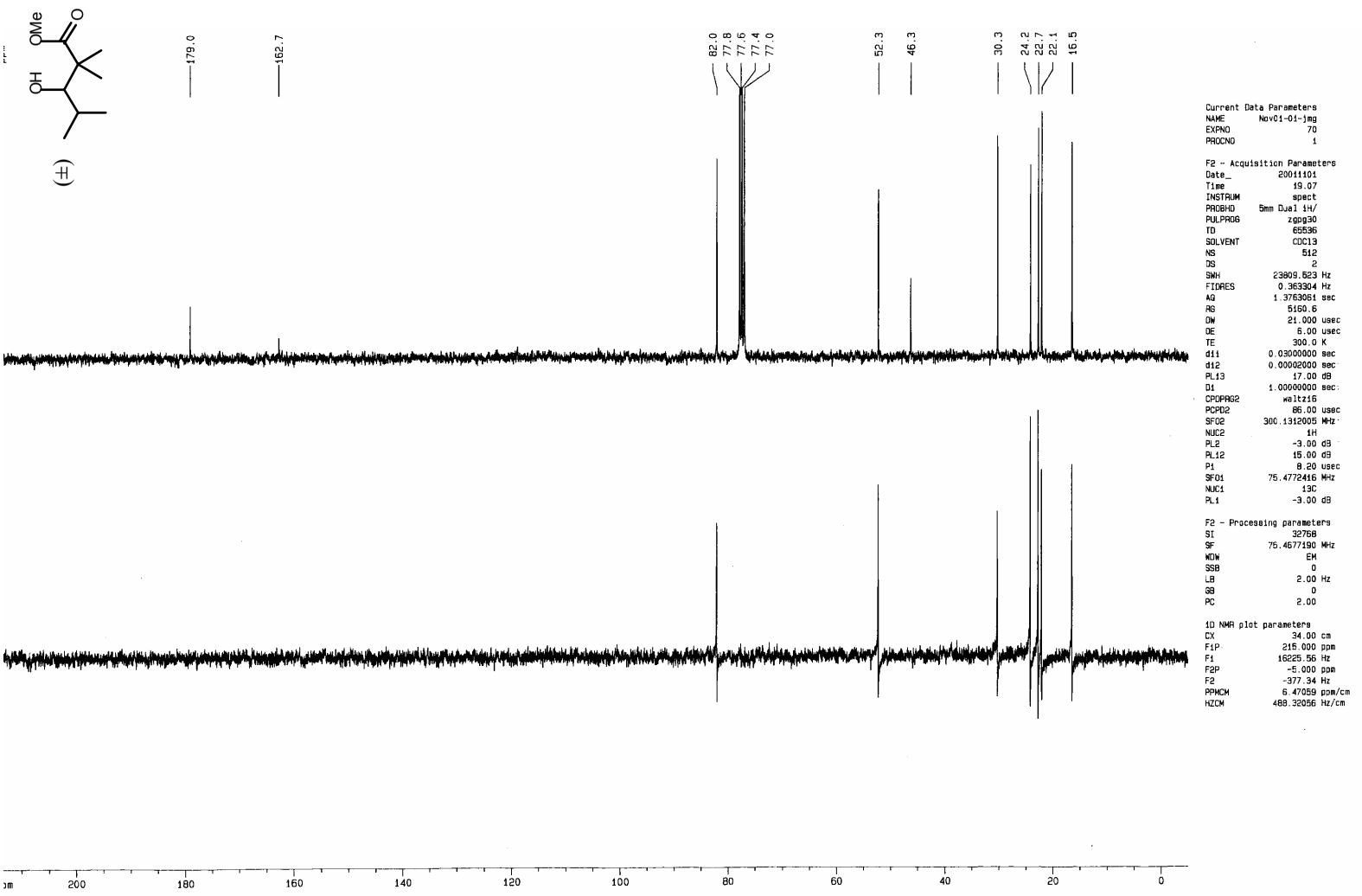
Run12



Ligand

NMR Data for racemic Mukaiyama aldol product



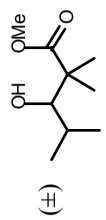


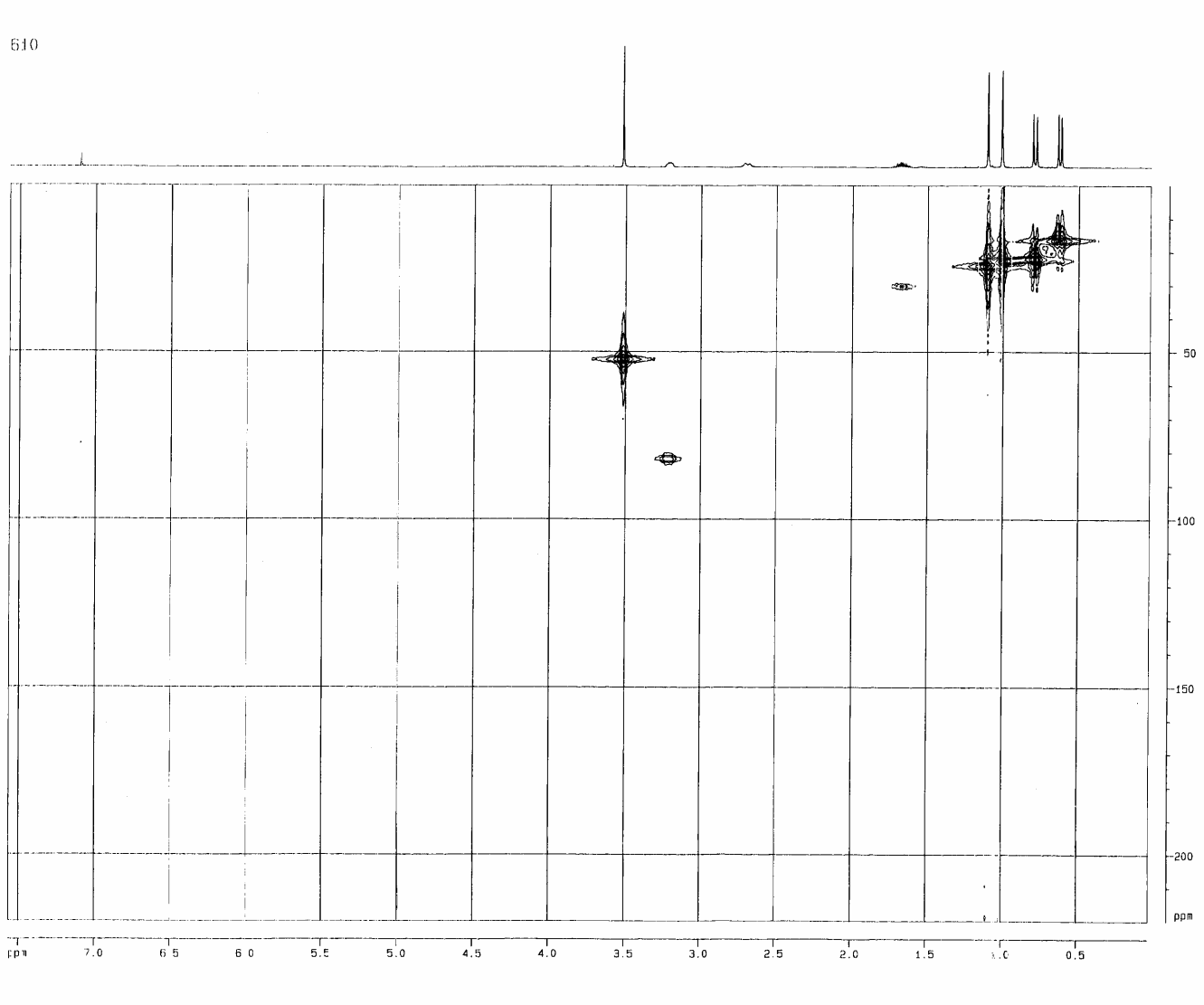
Current Data Parameters
NAME Nov01-01-jmg
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011101
Time 19.07
INSTRUM spect
PROBHD 5mm Dual 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763081 sec
RG 5160.6
DW 21.000 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 86.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 15.00 dB
P1 8.20 usec
SFO1 75.4772416 MHz
NUC1 13C
PL1 -3.00 dB

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 34.00 cm
F1P 215.000 ppm
F1 16225.56 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 6.47059 ppm/cm
HZCM 488.32056 Hz/cm





Current Data Parameters

NAME	Nov02-01-1mg
EXPNO	111
PROCNO	1

F2 - Acquisition Parameters

Date_	20011102
Time	17.15
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	inv4gp
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	2264.493 Hz
FIDRES	1.105709 Hz
AQ	0.4522484 sec
RG	20642.5
DW	220.800 usec
DE	6.00 usec
TE	300.0 K
P1	9.70 usec
p2	19.40 usec
d0	0.00000300 sec
CNST2	145.0000000
d2	0.00344828 sec
d12	0.00002000 sec
d13	0.00000300 sec
P16	1000.00 usec
d26	0.00240528 sec
D1	1.28384697 sec
SFO1	300.1319931 MHz
NUC1	1H
PL1	-3.00 dB
PL2	-3.00 dB
P3	9.00 usec
SFO2	75.4760204 MHz
NUC2	13C
GPX1	0.00 %
GPY1	0.00 %
GPZ1	50.00 %
GPNAM1	sine.100
D16	0.00010000 sec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	30.00 %
GPNAM2	sine.100
GPX3	0.00 %
GPY3	0.00 %
GPZ3	40.00 %
GPNAM3	sine.100
PL12	14.00 dB
CPDPRG2	garp
PCPD2	88.00 usec
IND	0.00003011 sec

F1 - Acquisition parameters

NDO	2
TD	128
SFO1	75.47602 MHz
FIDRES	129.721878 Hz
SW	219.995 ppm

F2 - Processing parameters

SI	2048
SF	300.1300539 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

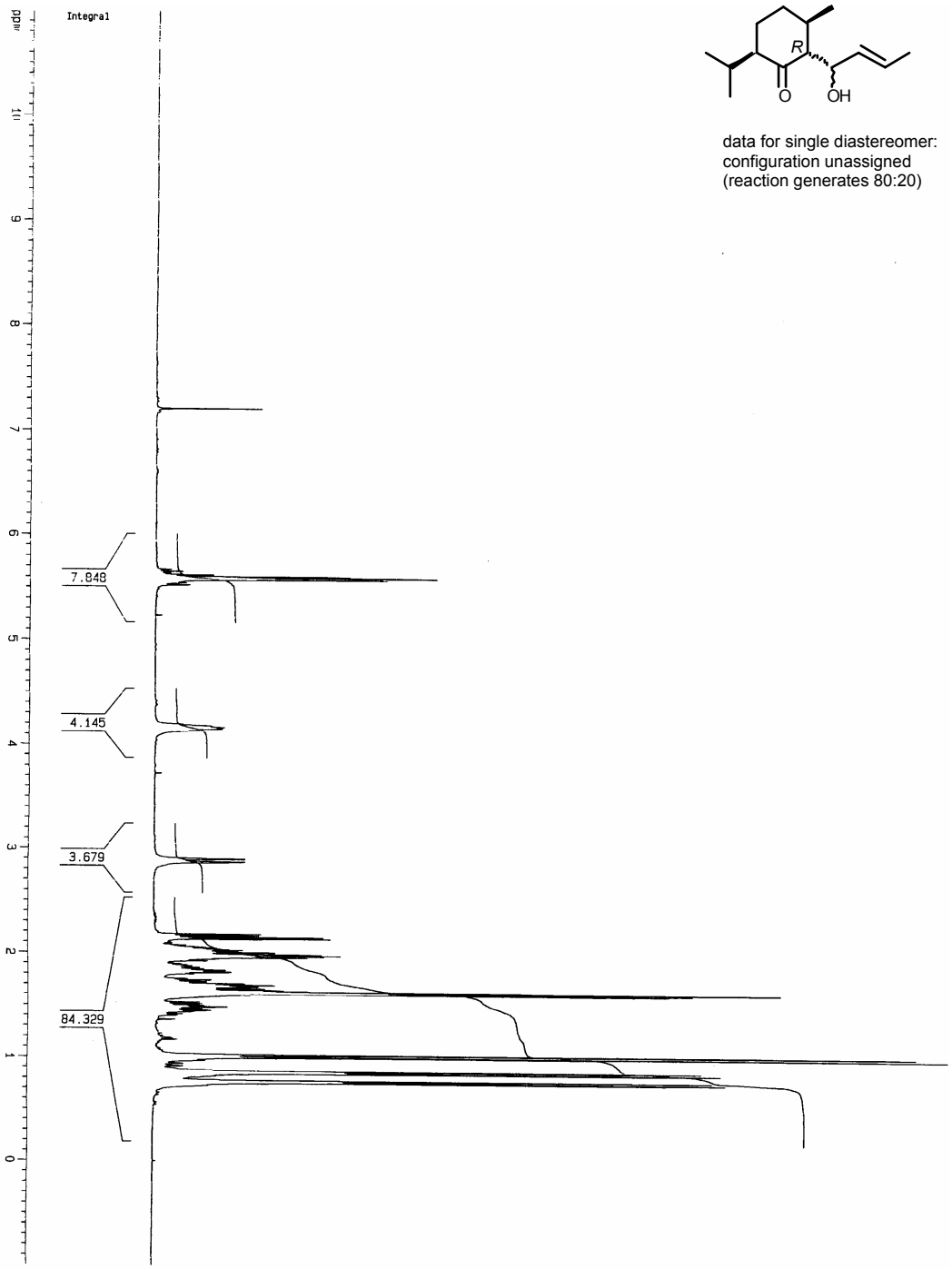
F1 - Processing parameters

SI	512
MC2	QF
SF	75.4677190 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	31.00 cm
CX1	20.00 cm
F2PLO	7.565 ppm
F2LO	2270.45 Hz
F2PHI	0.020 ppm
F2HI	5.87 Hz
F1PLO	219.996 ppm
F1LO	16602.62 Hz
F1PHI	0.004 ppm
F1HI	0.28 Hz
F2PPMCM	0.24338 ppm/cm
F2HZCM	73.04788 Hz/cm
F1PPMCM	10.99963 ppm/cm
F1HZCM	830.11719 Hz/cm

NMR Data for independently prepared isomenthone aldol products and diols



data for single diastereomer:
configuration unassigned
(reaction generates 80:20)

```
Current Data Parameters
NAME                  Mar12-01-jmg
EXPNO                         110
PROCNO                          1

F2 - Acquisition Parameters
Date_               20010312
Time                    21.22
INSTRUM                 spect
PROBHD       5mm Dual 1H/
PULPROG                 zg30
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  6172.839 Hz
FIDRES               0.188380 Hz
AQ                   2.6542580 sec
RG                        128
DW                   81.000 usec
DE                    6.00 usec
TE                    300.0 K
D1                    1.00000000 sec
P1                       9.70 usec
SFO1                 300.1319534 MHz
NUC1                        1H
PL1                   -3.00 dB

F2 - Processing parameters
SI                       16384
SF                   300.1300219 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00

1D NMR plot parameters
CX                    30.00 cm
F1P                   11.000 ppm
F1                    3301.43 Hz
F2P                   -1.000 ppm
F2                    -300.13 Hz
PPMCM                 0.40000 ppm/cm
HZCM                  120.05202 Hz/cm
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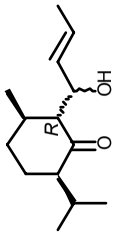
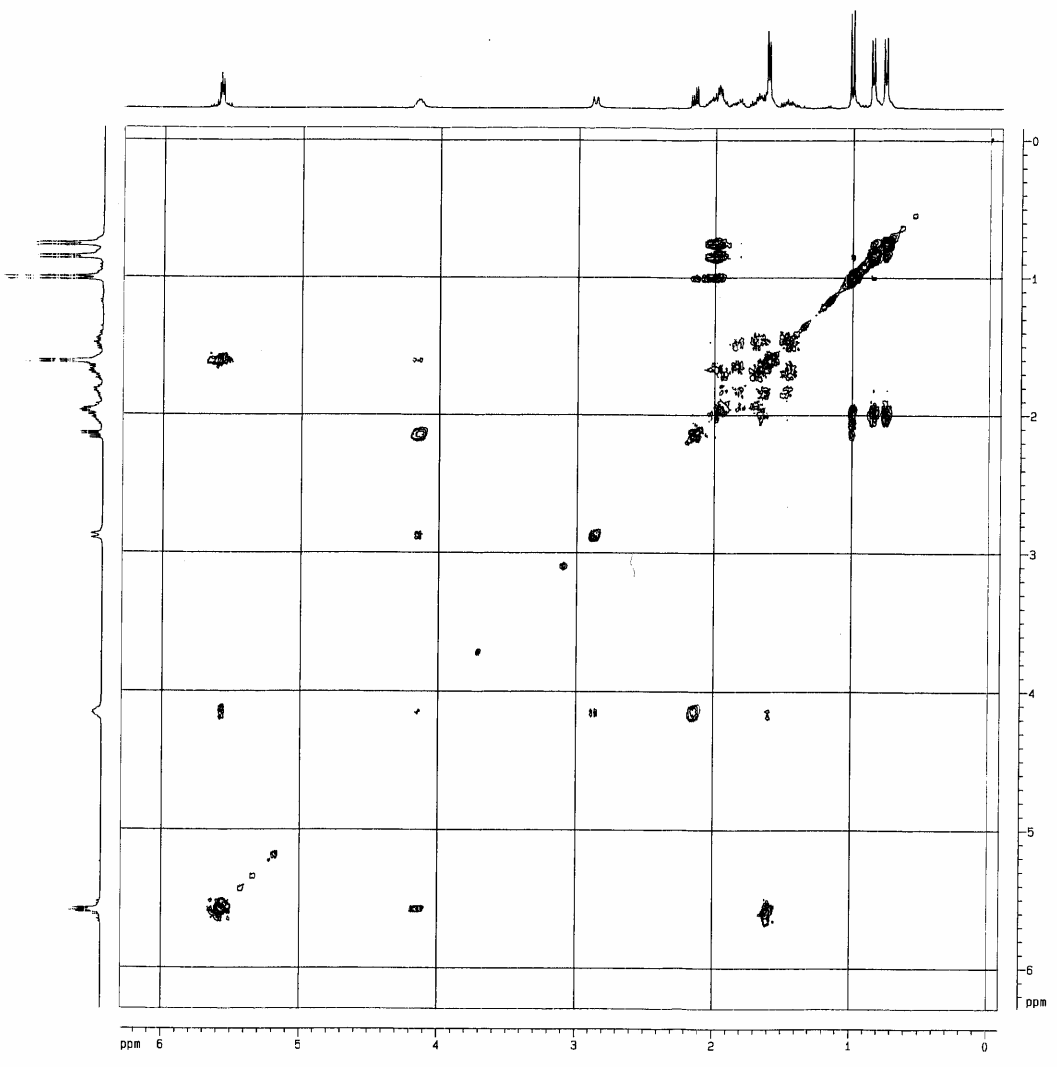


data for single diastereomer:
configuration unassigned
(reaction generates 80:20)



pc455b single diastere

Current Data Parameters

NAME	Mar12-01-jmg
EXPNO	111
PROCNO	1

F2 - Acquisition Parameters

Date_	20010312
Time	21.23
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	cosygp
TD	2048
SOLVENT	CDCl3
NS	1
DS	16
SWH	1917.178 Hz
FIDRES	0.936122 Hz
AQ	0.5341684 sec
RG	101.6
DW	260.800 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
d13	0.00000300 sec
D1	0.87495573 sec
P1	9.70 usec
SFO1	300.1309505 MHz
NUC1	1H
PL1	-3.00 dB
P16	1000.00 usec
GPX1	0.00 %
GPY1	0.00 %
GPZ1	10.00 %
GPNAM1	sine.100
D16	0.00010000 sec
P0	9.70 usec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	10.00 %
GPNAM2	sine.100
IN0	0.00052150 sec

F1 - Acquisition parameters

NDO	1
TD	256
SFO1	300.131 MHz
FIDRES	7.488976 Hz
SW	6.388 ppm

F2 - Processing parameters

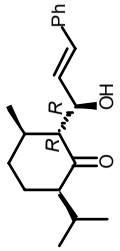
SI	1024
SF	300.1300219 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

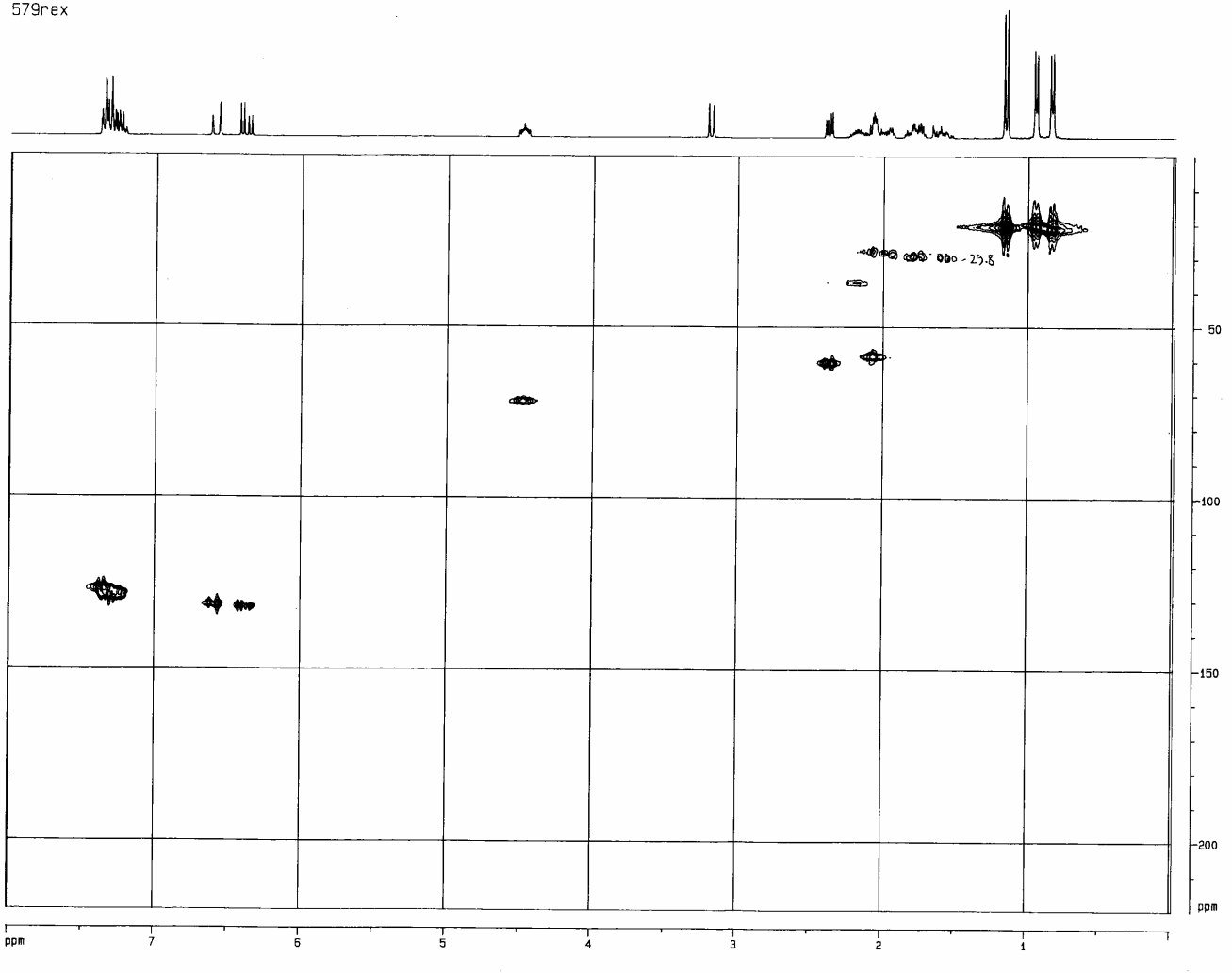
SI	512
MC2	QF
SF	300.1300219 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	22.00 cm
CX1	22.00 cm
F2PLO	6.298 ppm
F2LO	1890.14 Hz
F2PHI	-0.090 ppm
F2HI	-27.03 Hz
F1PLO	6.298 ppm
F1LO	1890.14 Hz
F1PHI	-0.090 ppm
F1HI	-27.03 Hz
F2PPMCM	0.29075 ppm/cm
F2HZCM	87.14413 Hz/cm
F1PPMCM	0.290 ppm/cm
F1HZCM	87.14 Hz/cm



579rex



Current Data Parameters

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EXPNO	74
PROCNO	1

F2 - Acquisition Parameters

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Time	20.00
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	inv4gp
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	2408.478 Hz
FIDRES	1.176015 Hz
AQ	0.425214 sec
RG	23170.5
DW	207.600 usec
DE	6.00 usec
TE	300.0 K
P1	9.70 usec
p2	19.40 usec
d0	0.00000300 sec
CNST2	145.0000000
d2	0.00344828 sec
d12	0.00002000 sec
d13	0.00000300 sec
P16	1000.00 usec
d20	0.00242526 sec
D1	1.31047106 sec
SFO1	300.1312010 MHz
NUC1	1H
PL1	-3.00 dB
PL2	-3.00 dB
P3	9.00 usec
SFO2	75.4760204 MHz
NUC2	13C
GPX1	0.00 %
GPY1	0.00 %
GPZ1	50.00 %
GPNAM1	sine.100
D16	0.00010000 sec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	30.00 %
GPNAM2	sine.100
GPX3	0.00 %
GPY3	0.00 %
GPZ3	40.00 %
GPNAM3	sine.100
PL12	14.00 dB
CPDPRG2	garp
PCPD2	68.00 usec
IND	0.00003011 sec

F1 - Acquisition parameters

ND0	2
TD	128
SFO1	75.47602 MHz
FIDRES	129.721878 Hz
SW	219.996 ppm

F2 - Processing parameters

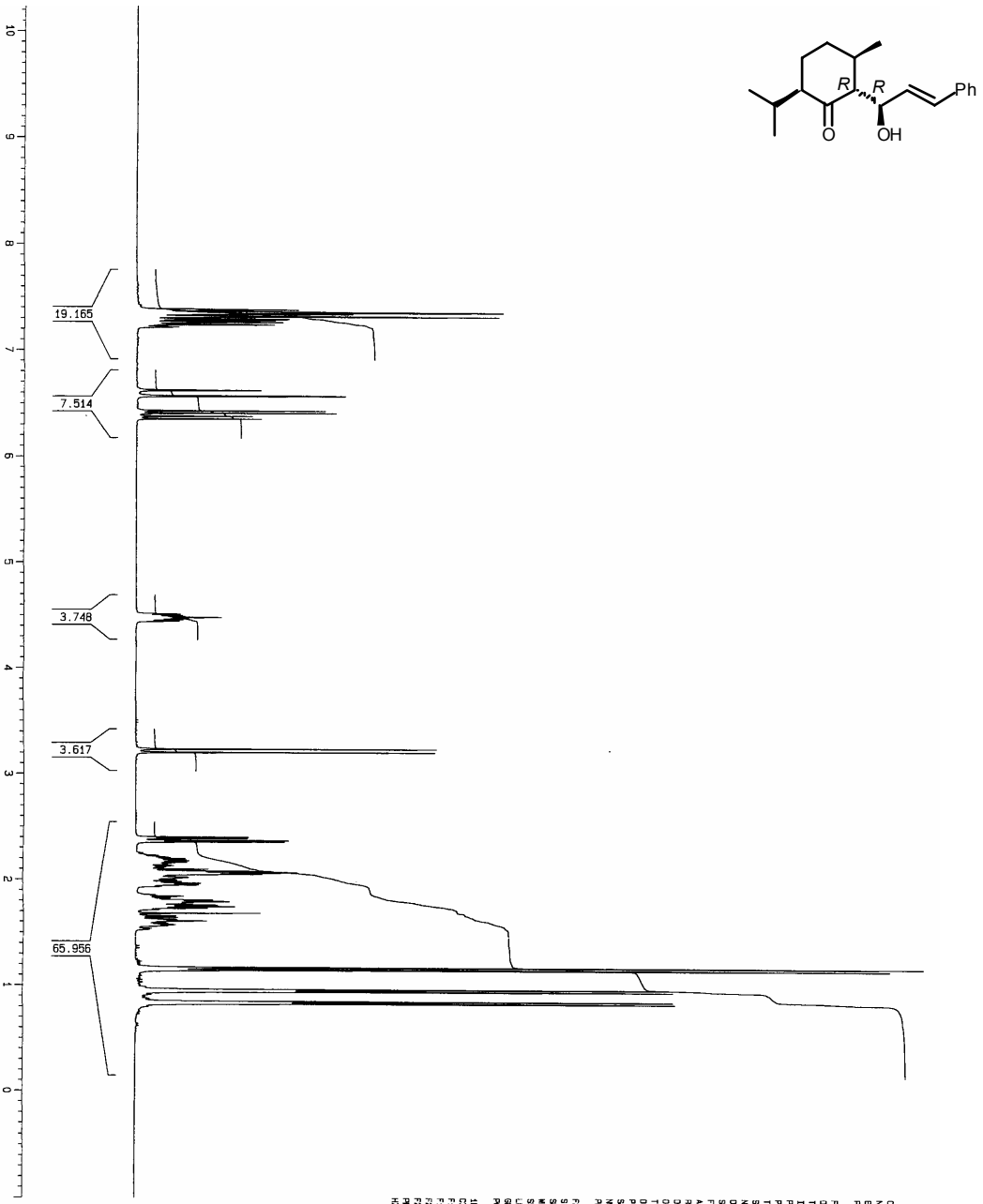
SI	2048
SF	300.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

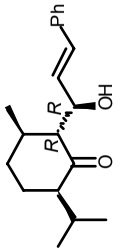
F1 - Processing parameters

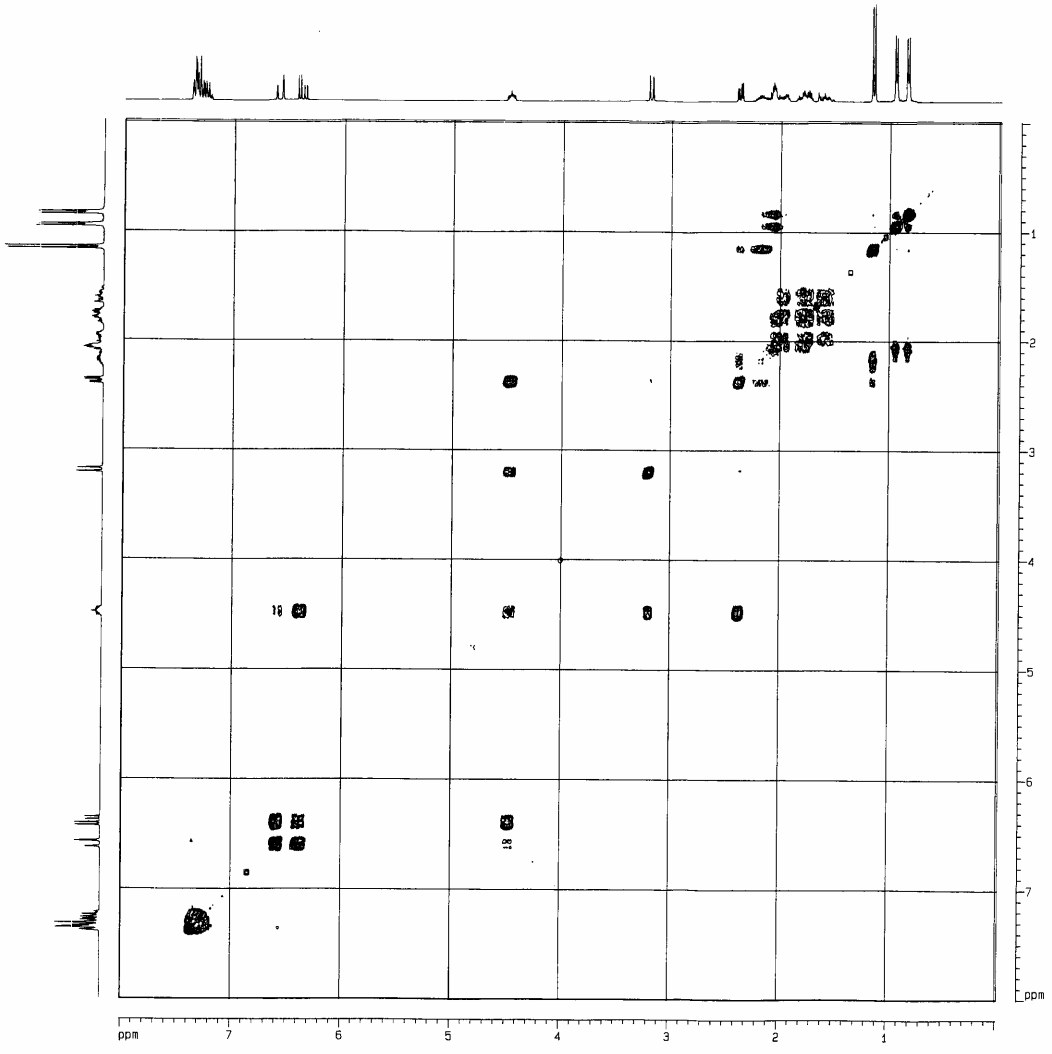
SI	512
MC2	QF
SF	75.4677190 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	31.00 cm
CX1	20.00 cm
F2PLO	8.006 ppm
F2LO	2402.94 Hz
F2PHI	-0.018 ppm
F2HI	-5.53 Hz
F1PLO	219.996 ppm
F1LO	16602.52 Hz
F1PHI	0.004 ppm
F1HI	0.28 Hz
F2PPMCM	0.25886 ppm/cm
F2HZCM	77.69252 Hz/cm
F1PPMCM	10.99963 ppm/cm
F1HZCM	830.11713 Hz/cm


19.165
7.514
3.748
3.617
65.956
R
R
Ph
OH
O





Current Data Parameters

NAME	Aug13-01-jmg
EXPNO	73
PROCNO	1

F2 - Acquisition Parameters

Date_	20010813
Time	19.52
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	cosygp
TD	2048
SOLVENT	CDCl3
NS	1
DS	16
SWH	2408.478 Hz
FIDRES	1.176015 Hz
AQ	0.4252148 sec
RG	114
DW	207.600 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
d13	0.00000300 sec
D1	0.98145258 sec
P1	9.70 usec
SFO1	300.1312010 MHz
NUC1	1H
PL1	-3.00 dB
P16	1000.00 usec
GPX1	0.00 %
GPY1	0.00 %
GPZ1	10.00 %
GPNAM1	sine.100
D16	0.00010000 sec
P0	9.70 usec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	10.00 %
GPNAM2	sine.100
IN0	0.00041520 sec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.1312 MHz
FIDRES	9.408116 Hz
SW	8.025 ppm

F2 - Processing parameters

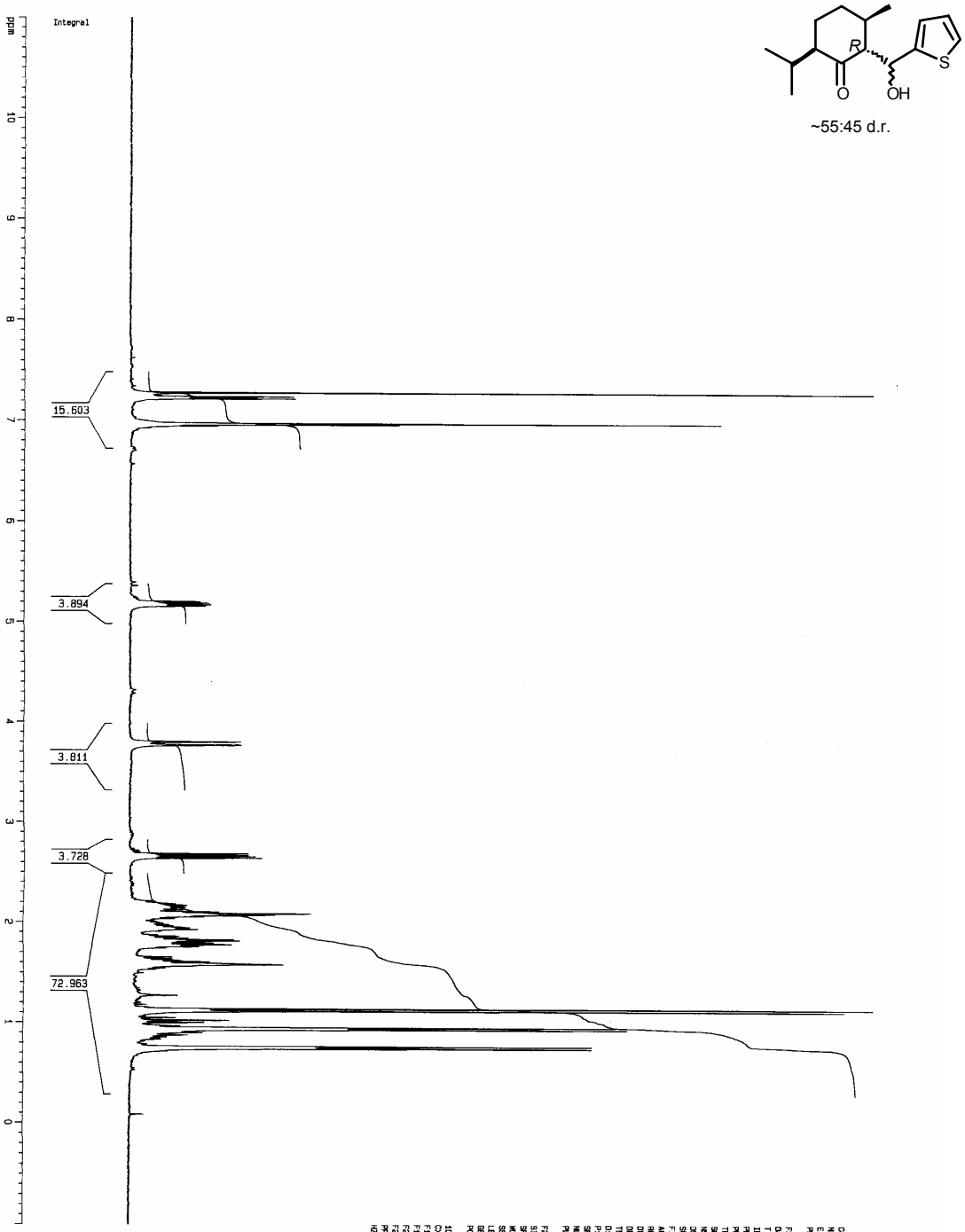
SI	1024
SF	300.1300000 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	512
MC2	QF
SF	300.1300000 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0

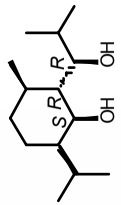
2D NMR plot parameters

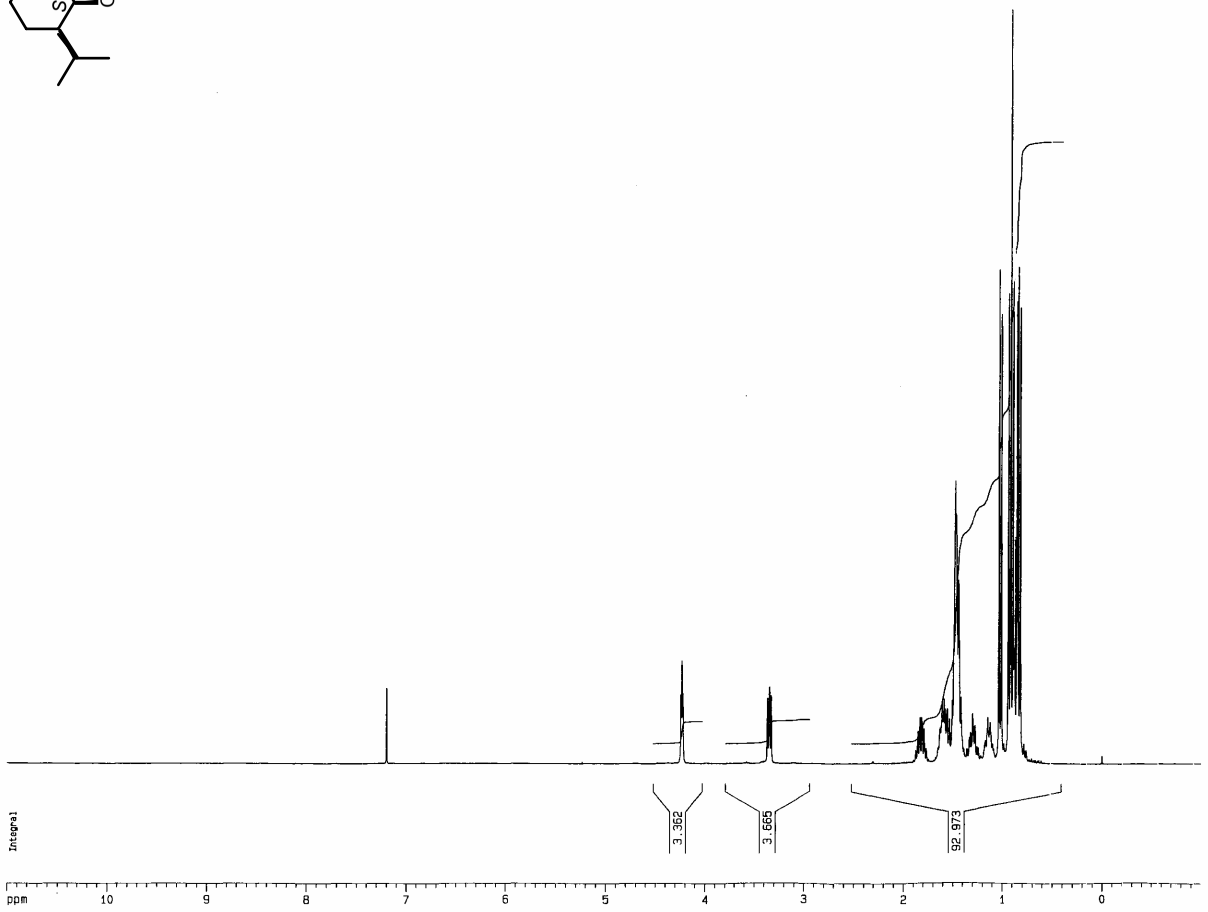
CX2	22.00 cm
CX1	22.00 cm
F2PLO	8.006 ppm
F2LO	2402.94 Hz
F2PHI	-0.018 ppm
F2HI	-5.53 Hz
F1PLO	8.006 ppm
F1LO	2402.94 Hz
F1PHI	-0.018 ppm
F1HI	-5.53 Hz
F2PPMCM	0.36476 ppm/cm
F2HZCM	109.47583 Hz/cm
F1PPMCM	0.36476 ppm/cm
F1HZCM	109.47583 Hz/cm



~55:45 d.r.

455c->111





Current Data Parameters

NAME	Sep03-01-jmg
EXPNO	110
PROCNO	1

F2 - Acquisition Parameters

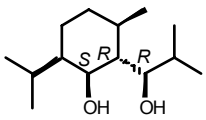
Date_	20010903
Time	19.00
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	161.3
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	9.70 usec
SFO1	300.1318534 MHz
NUC1	1H
PL1	-3.00 dB

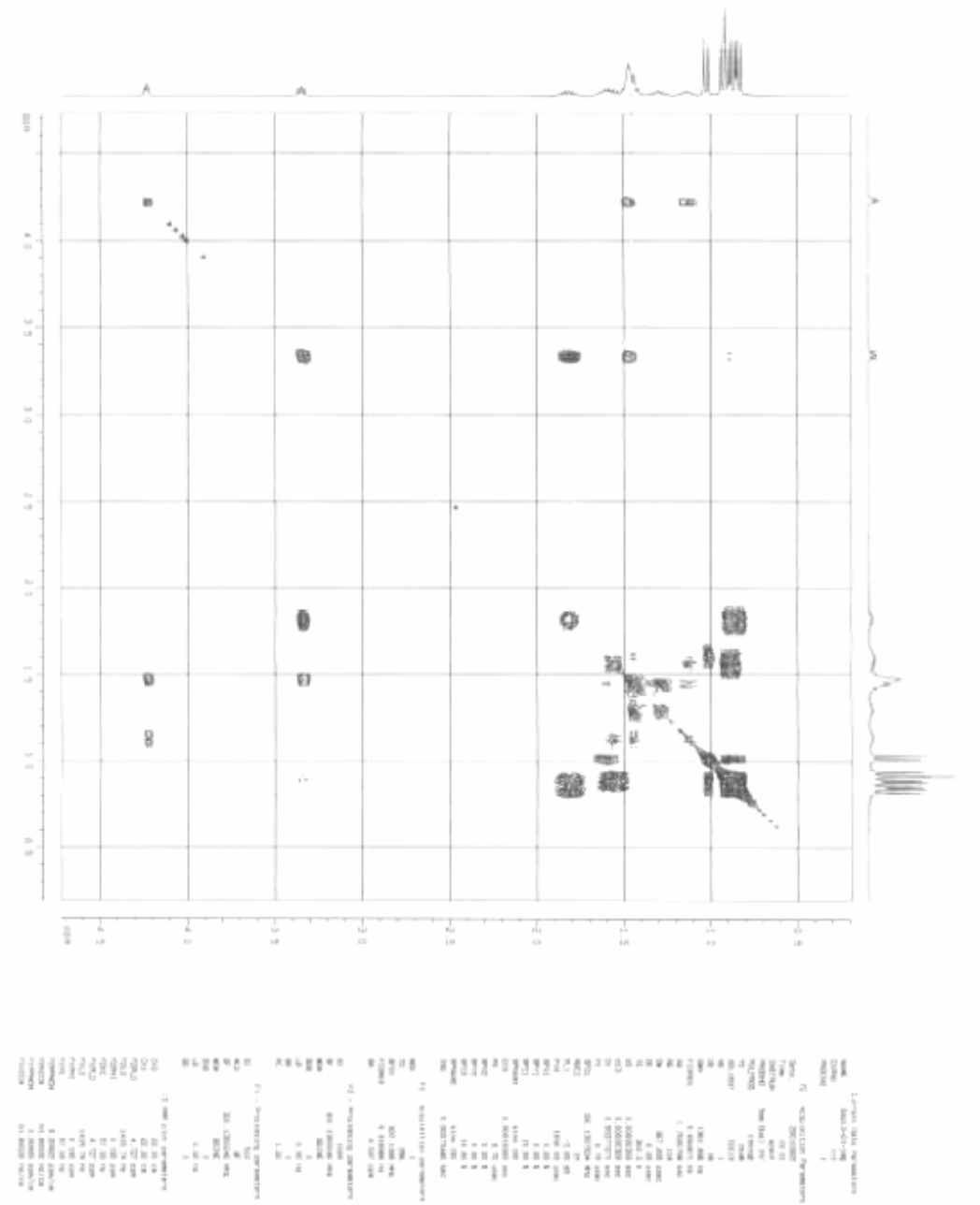
F2 - Processing parameters

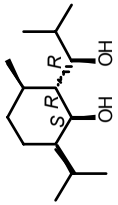
SI	16384
SF	300.1300240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

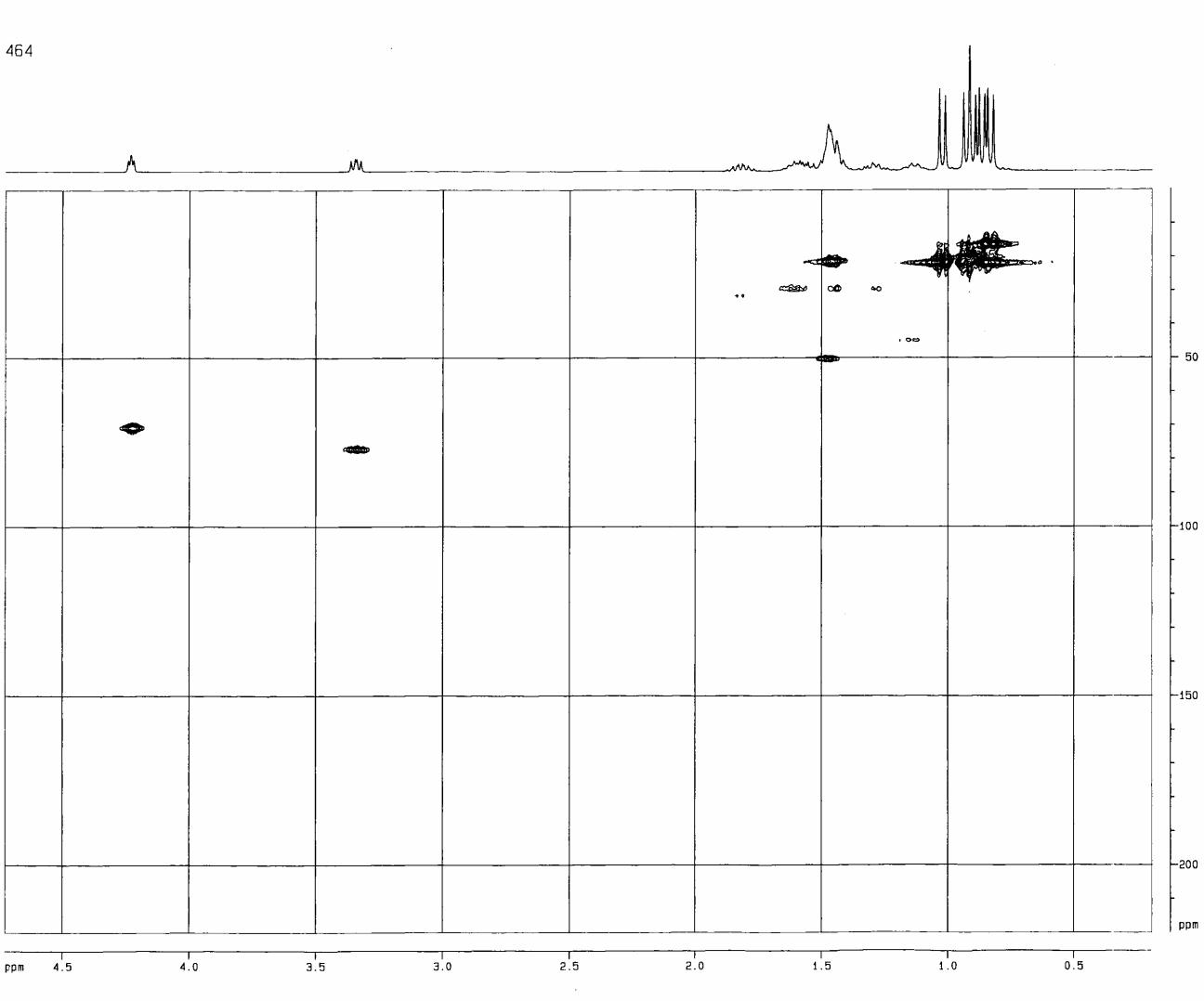
CX	30.00 cm
F1P	11.000 ppm
F1	3301.43 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCM	0.40000 ppm/cm
HZCM	120.05202 Hz/cm


S
R
R
OH
OH





464



Current Data Parameters

NAME	Sep03-01-jmp
EXPNO	112
PROCNO	1

F2 - Acquisition Parameters

Date_	20010903
Time	19.09
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	inv4gp
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	1361.655 Hz
FIDRES	0.664671 Hz
AQ	0.7520756 sec
RG	20642.5
DW	367.200 usec
DE	6.00 usec
TE	300.0 K
P1	9.70 usec
p2	19.40 usec
d0	0.00000300 sec
CNST2	145.0000000
d2	0.00344828 sec
d12	0.00002000 sec
d13	0.00000300 sec
P16	1000.00 usec
d20	0.00242528 sec
D1	0.98279142 sec
SFO1	300.1307634 MHz
NUC1	1H
PL1	-3.00 dB
PL2	-3.00 dB
P3	9.00 usec
SFO2	75.4760264 MHz
NUC2	13C
GPX1	0.00 %
GPY1	0.00 %
GPZ1	50.00 %
GPNAM1	sine.100
D16	0.00010000 sec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	30.00 %
GPNAM2	sine.100
GPX3	0.00 %
GPY3	0.00 %
GPZ3	40.00 %
GPNAM3	sine.100
PL12	14.00 dB
CPDPRG2	garp
PCPD2	68.00 usec
INO	0.00003011 sec

F1 - Acquisition parameters

ND0	2
TD	128
SFO1	75.47602 MHz
FIDRES	129.721878 Hz
SW	219.996 ppm

F2 - Processing parameters

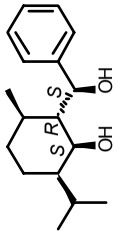
SI	2048
SF	300.1300240 MHz
WDW	GSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

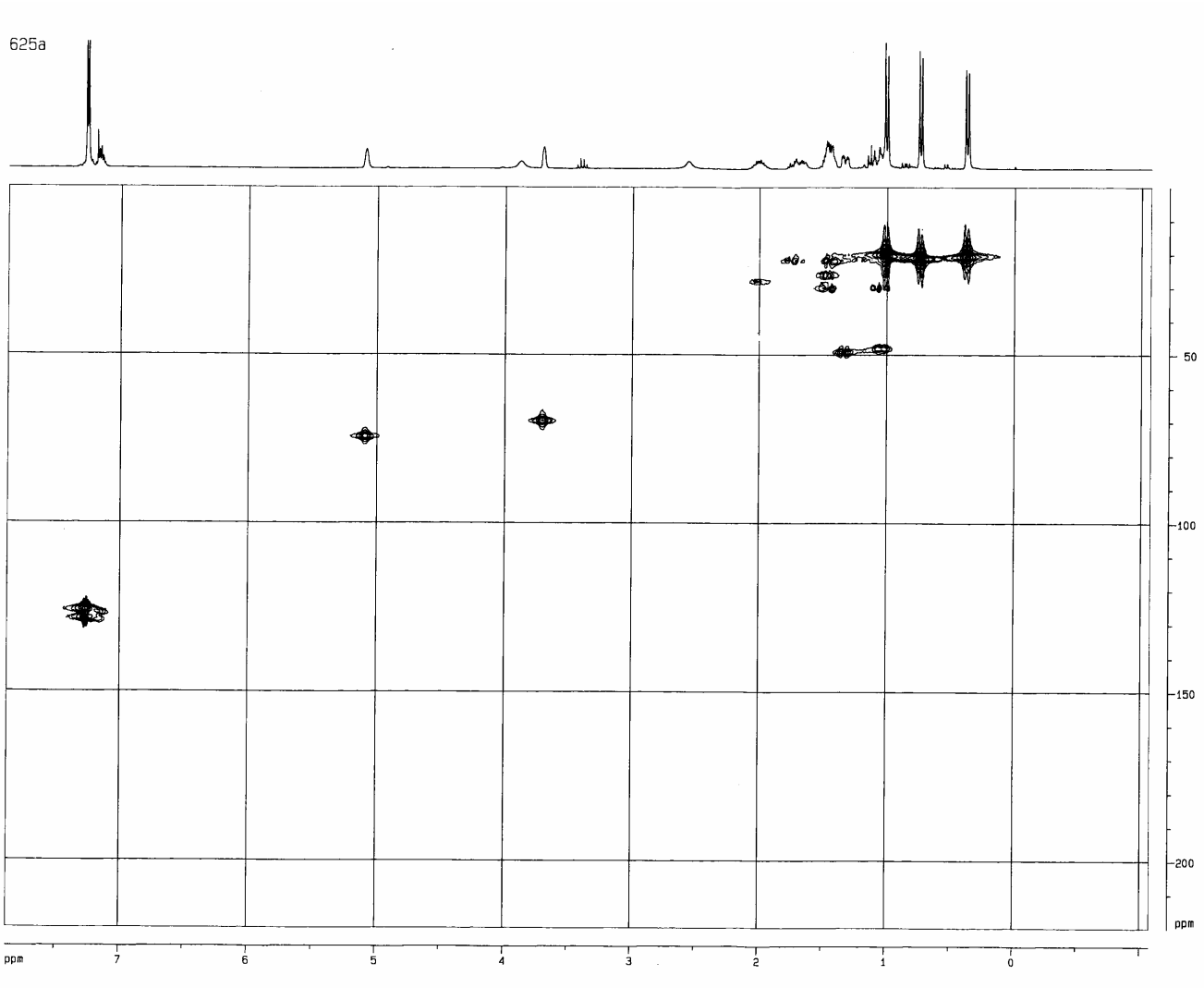
SI	512
MC2	QF
SF	75.4677190 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	31.00 cm
CX1	20.00 cm
F2PLO	4.727 ppm
F2LO	1418.74 Hz
F2PKI	0.190 ppm
F2HI	57.09 Hz
F1PLO	219.996 ppm
F1LO	16602.82 Hz
F1PKI	0.004 ppm
F1HI	0.28 Hz
F2PPMCM	0.14635 ppm/cm
F2HZCM	43.92427 Hz/cm
F1PPMCM	10.99963 ppm/cm
F1HZCM	830.11713 Hz/cm



625a



Current Data Parameters

NAME	Nov30-01-1mg
EXPNO	134
PROCNO	1

F2 - Acquisition Parameters

Date_	20011130
Time	20.47
INSTRUM	spect
PROBHD	5mm Dual 1H/
PULPROG	inv4gp
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	2688.172 Hz
FIDRES	1.312584 Hz
AQ	0.3809780 sec
RG	26008
DW	186.000 usec
DE	6.00 usec
TE	300.0 K
P1	9.70 usec
p2	19.40 usec
d0	0.00000300 sec
CNST2	145.0000000
d2	0.00344828 sec
d12	0.00002000 sec
d13	0.00000300 sec
P16	1000.00 usec
d20	0.00242528 sec
D1	1.35552704 sec
SFO1	300.1310481 MHz
NUC1	1H
PL1	-3.00 dB
PL2	-3.00 dB
P3	9.00 usec
SFO2	75.4760204 MHz
NUC2	13C
GPX1	0.00 %
GPY1	0.00 %
GPZ1	80.00 %
GPNAM1	sine.100
D16	0.00010000 sec
GPX2	0.00 %
GPY2	0.00 %
GPZ2	30.00 %
GPNAM2	sine.100
GPX3	0.00 %
GPY3	0.00 %
GPZ3	40.00 %
GPNAM3	sine.100
PL12	14.00 dB
CPDPRG2	garp
PCPD2	68.00 usec
IN0	0.00003011 sec

F1 - Acquisition parameters

ND0	2
TD	128
SFO1	75.47602 MHz
FIDRES	129.721878 Hz
SW	219.996 ppm

F2 - Processing parameters

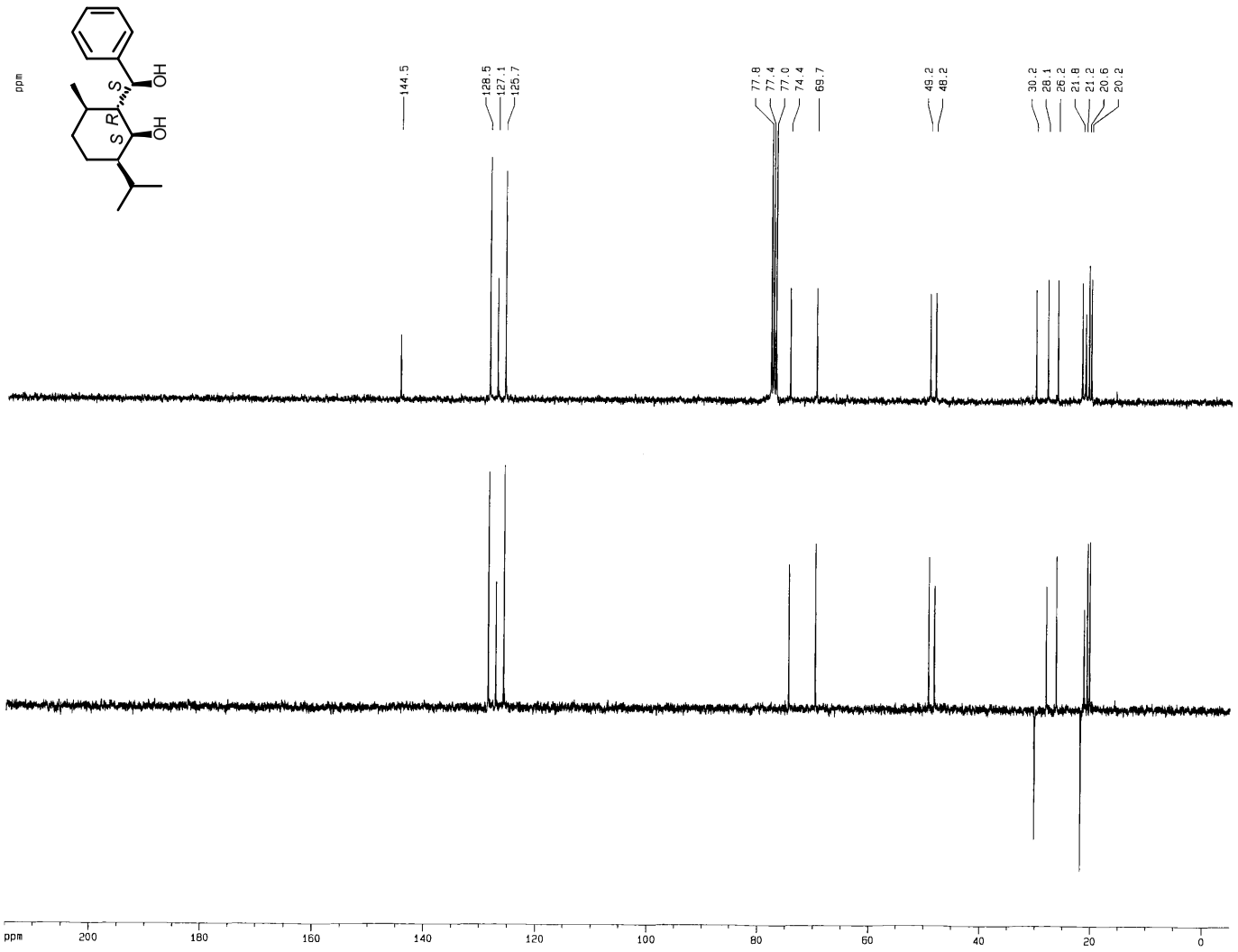
SI	2048
SF	300.1300269 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	512
MC2	QF
SF	75.4677190 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	31.00 cm
CY1	20.00 cm
F2PLO	7.881 ppm
F2LO	2365.23 Hz
F2PHI	-1.076 ppm
F2HI	-322.93 Hz
F1PLO	219.996 ppm
F1LO	16502.62 Hz
F1PHI	0.004 ppm
F1HI	0.28 Hz
F2PPMCM	0.28852 ppm/cm
F2HZCM	86.71494 Hz/cm
F1PPMCM	10.99963 ppm/cm
F1HZCM	830.11713 Hz/cm



Current Data Parameters
NAME Nov30-01-jmg
EXPNO 132
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011130
Time 20.35
INSTRUM spect
PROBHD 5mm Dual 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 16384
DW 21.000 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 86.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 15.00 dB
P1 9.20 usec
SFO1 75.4772416 MHz
NUC1 13C
PL1 -3.00 dB

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 34.00 cm
F1P 215.000 ppm
F1 16225.56 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 6.47059 ppm/cm
HZCM 488.32056 Hz/cm