Supporting Information for:

Stereoselective synthesis of Norephedrine and Norpseudoephedrine by Using Asymmetric Transfer Hydrogenation Accompanied by Dynamic Kinetic Resolution

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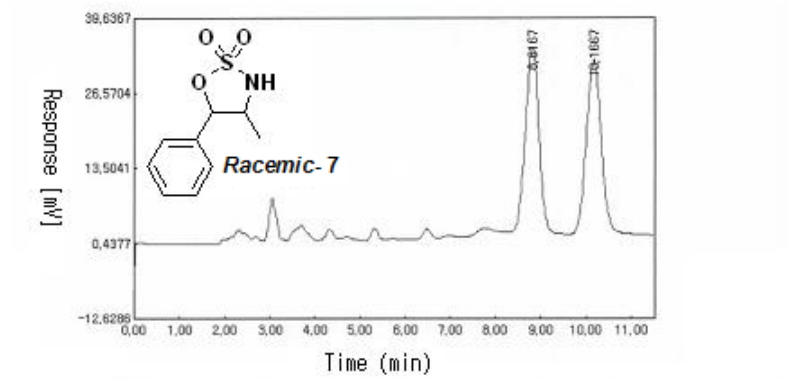
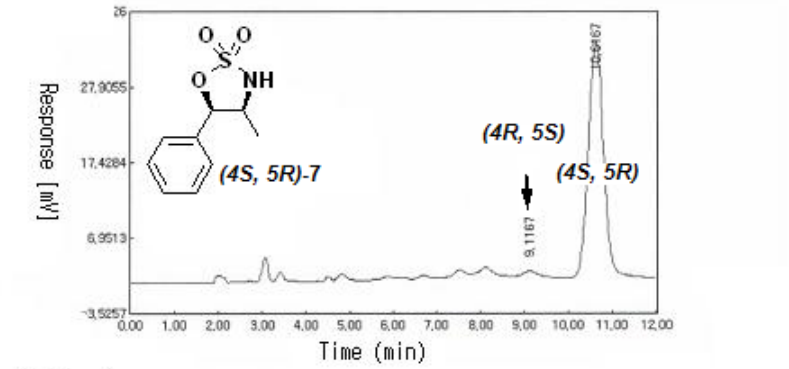
1. Chiral HPLC Chromatogram

1-1. (*4S,5R*)-4-Methyl-5-phenyl-[1,2,3]oxathiazolidine 2,2-dioxide, (*4S,5R*)-7

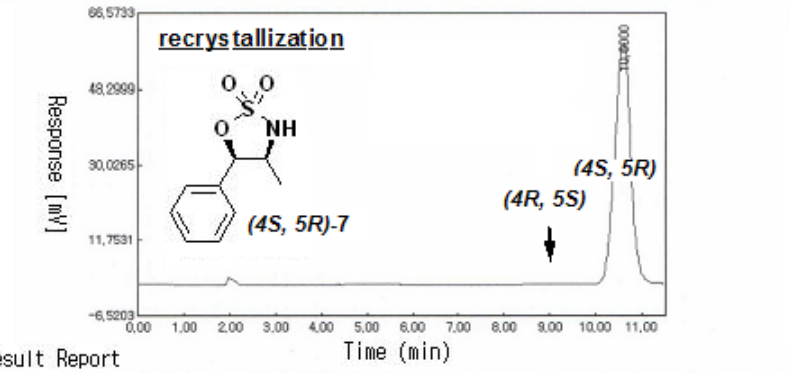
Chiralcel AD-H, 10% isopropanol/hexanes, 1.5 mL/min, 254 nm, tr(minor) = 9.1 min, tr(major) = 10.6 min.





Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	9.1167	16.3031	FF	50.0000	1.9564
2	10.6167	817.0167	FF	122.0000	98.0436
Total		833.3198			

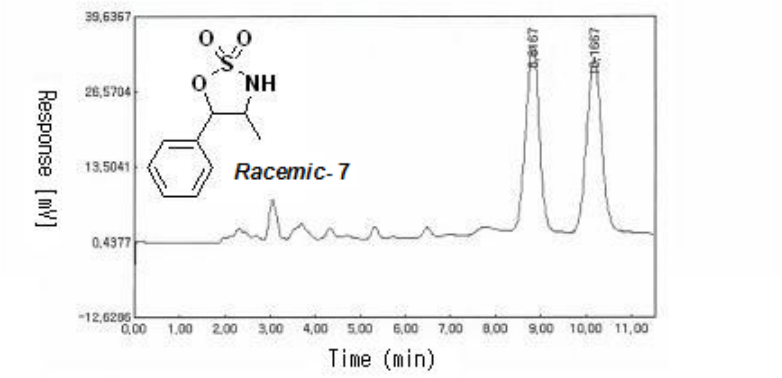


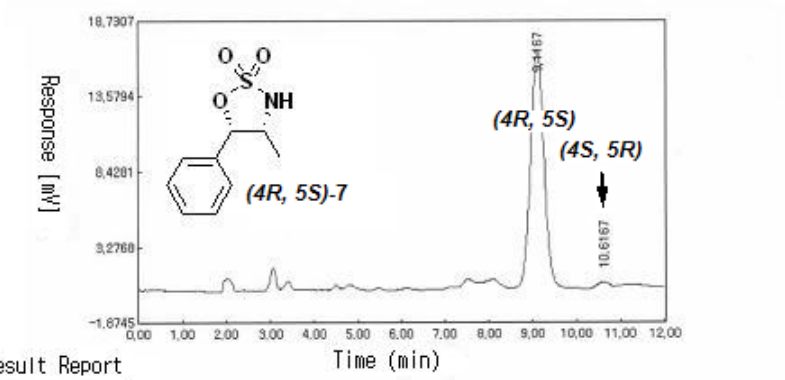
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	10.6000	1432.5622	BB	68.0000	100.0000
Total		1432.5623			

1-2. (*4R,5S*)-4-Methyl-5-phenyl-[1,2,3]oxathiazolidine 2,2-dioxide, (*4R,5S*)-7

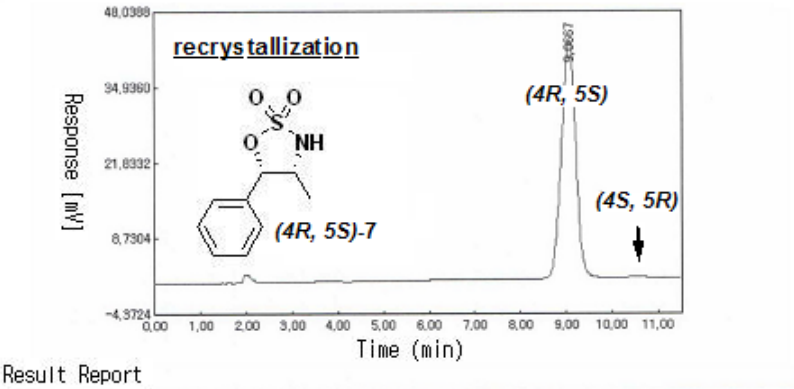
Chiralcel AD-H, 10% isopropanol/hexanes, 1.5 mL/min, 254 nm, tr(major) = 9.1 min, tr(minor) = 10.6 min.





Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	9.1167	361.7222	FF	85.0000	98.2133
2	10.6167	6.5804	FF	38.0000	1.7867
Total		368.3026			

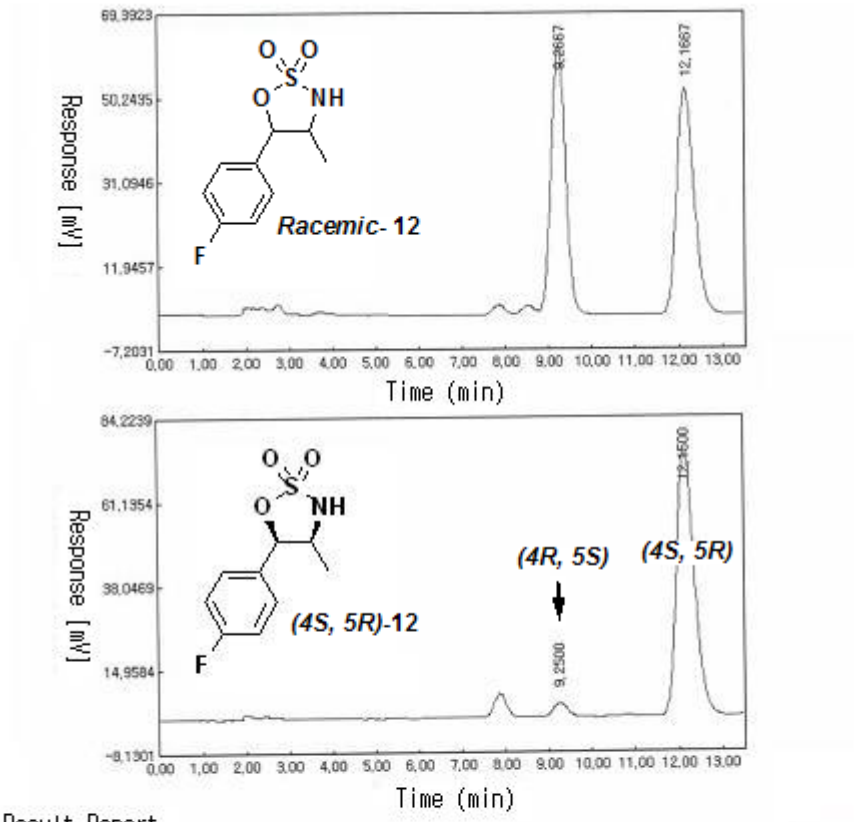


Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	9.0667	910.1313	BB	59.0000	100.0000
Total		910.1313			

1-3. (*4S,5R*)-5-(4-Fluoro-phenyl)-4-methyl-[1,2,3]oxathiazolidine 2,2-dioxide, (*4S,5R*)-12

Chiralcel AD-H, 10% isopropanol/hexanes, 1.5 mL/min, 254 nm, tr(minor) = 9.3 min, tr(major) = 12.1 min.

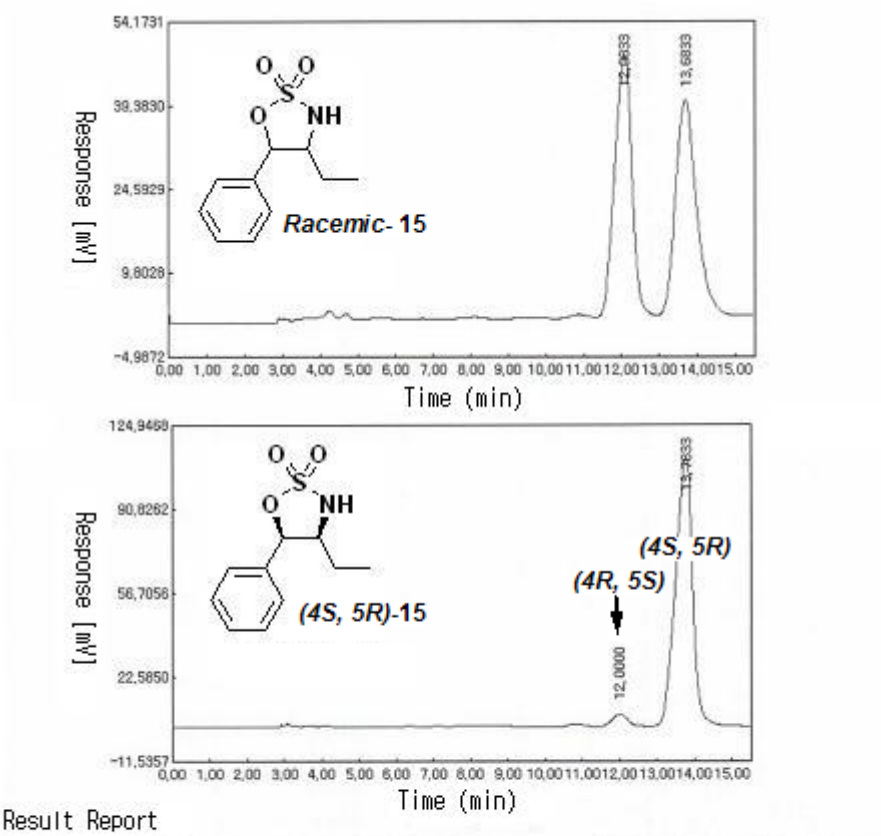


Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	9.2500	78.7450	BB	42.0000	3.6071
2	12.1500	2104.3198	BB	78.0000	96.3929
Total		2183.0647			

1-4. (*4S,5R*)-4-Ethyl-5-phenyl-[1,2,3]oxathiazolidine 2,2-dioxide, (*4S,5R*)-15

Chiralcel AD-H, 10% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(minor) = 12.0 min, tr(major) = 13.8 min.

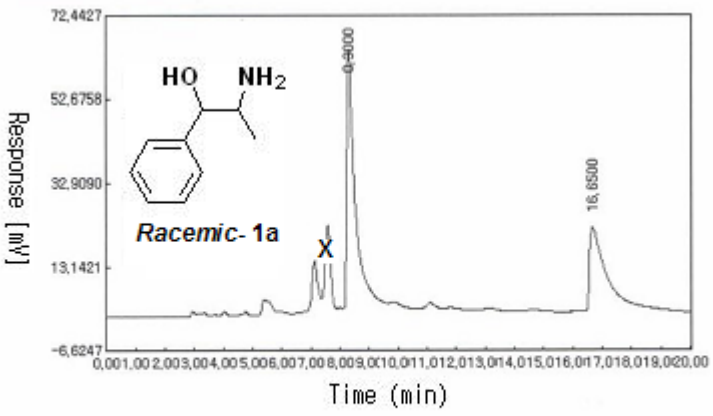


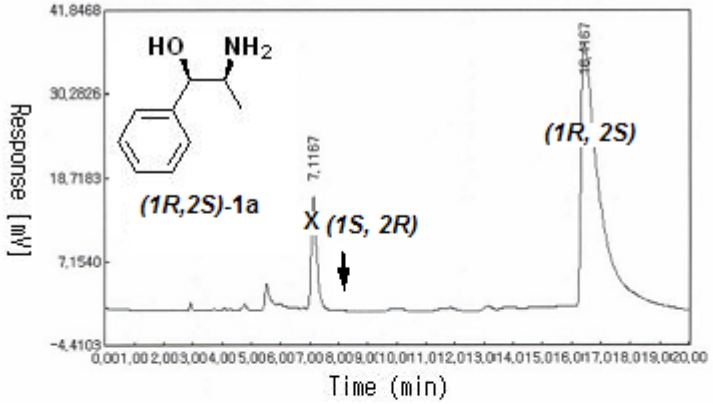
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	12.0000	119.7793	BB	51.0000	3.3040
2	13.7833	3505.4774	BB	94.0000	96.6960
Total		3625.2568			

2-1. (*1R,2S*)-2-Amino-1-phenyl-propan-1-ol, (*1R,2S*)-1a, [(*1R,2S*)-(-)-Norephedrine]

Chiralcel AD-H, 10% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(minor) = 8.3 min, tr(major) = 16.4 min.



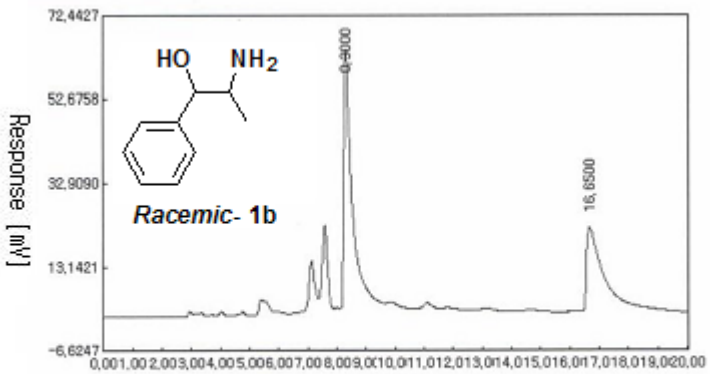


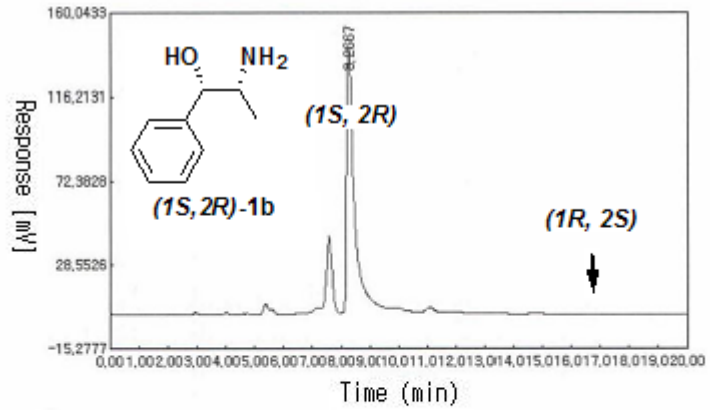
Result Report

Peak #	Time (min)	Area[mV*s]	BL	wide(sec)	Area (%)
1	7.1167	208.1875	BB	39.0000	13.3438
2	16.4167	1351.9909	BB	96.0000	86.6562
Total		1560.1783			

2-2. (*1S,2R*)-2-Amino-1-phenyl-propan-1-ol, (*1S,2R*)-1b, [(*1S,2R*)-(+)-Norephedrine]

Chiralcel AD-H, 10% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(major) = 8.3 min, tr(minor) = 16.4 min.



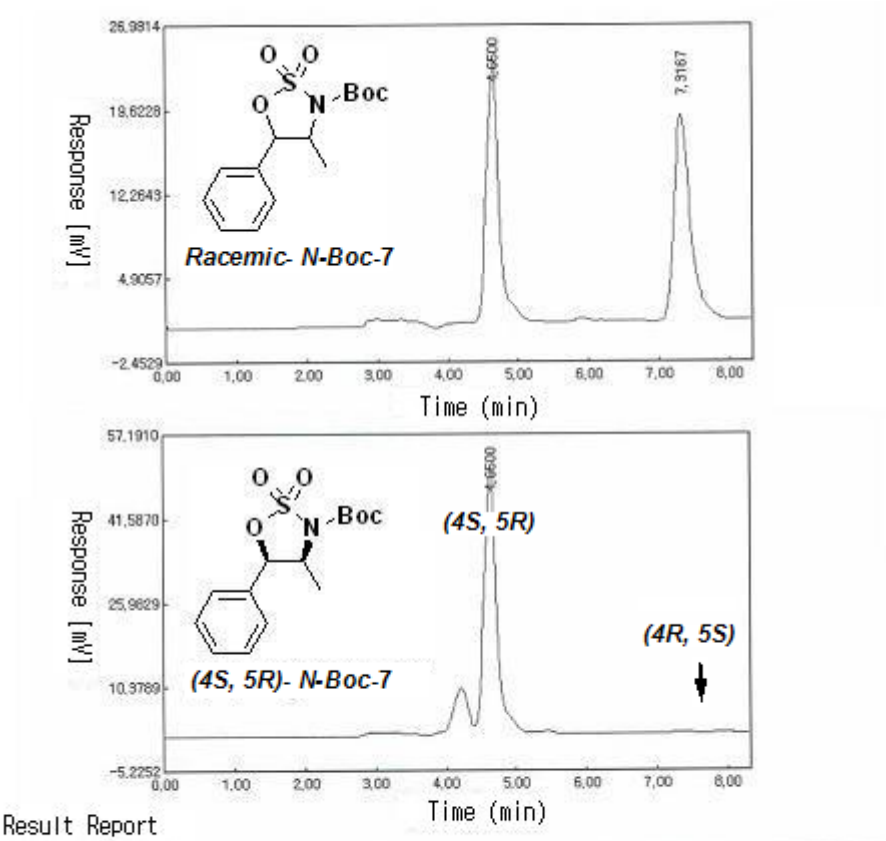


Result Report

Peak #	Time (min)	Area[mV*s]	BL	wide(sec)	Area (%)
1	8.2667	2517.0946	BB	83.0000	100.0000
Total		2517.0947			

3-1. (*4S,5R*)-4-Methyl-2,2-dioxo-5-phenyl-2λ^6-[1,2,3]oxathiazolidine-3-carboxylic acid tert-butyl ester, *N-Boc*-(*4S,5R*)-7

Chiralcel AD-H, 30% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(major) = 4.6 min, tr(minor) = 7.3 min.

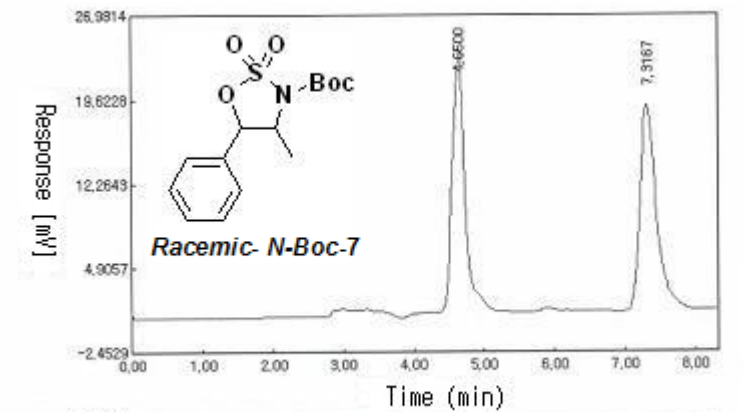


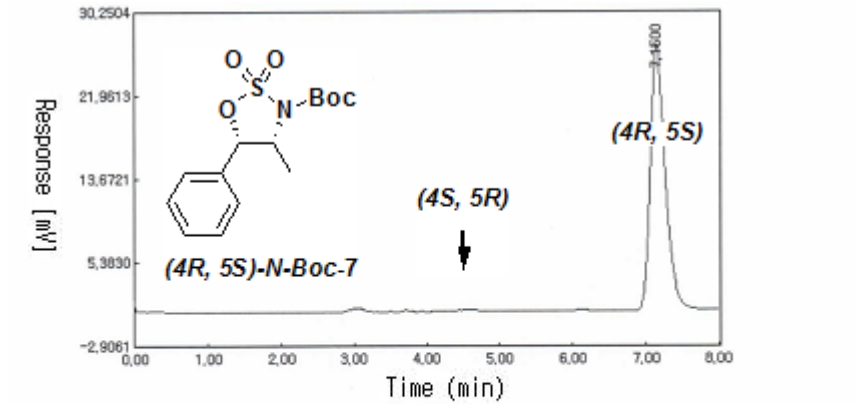
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	4.6500	602.9331	BB	45.0000	100.0000
Total		602.9331			

3-2. (*4R,5S*)-4-Methyl-2,2-dioxo-5-phenyl-2λ^6-[1,2,3]oxathiazolidine-3-carboxylic acid tert-butyl ester, *N-Boc*-(*4R,5S*)-7

Chiralcel AD-H, 30% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(minor) = 4.6 min, tr(major) = 7.3 min.

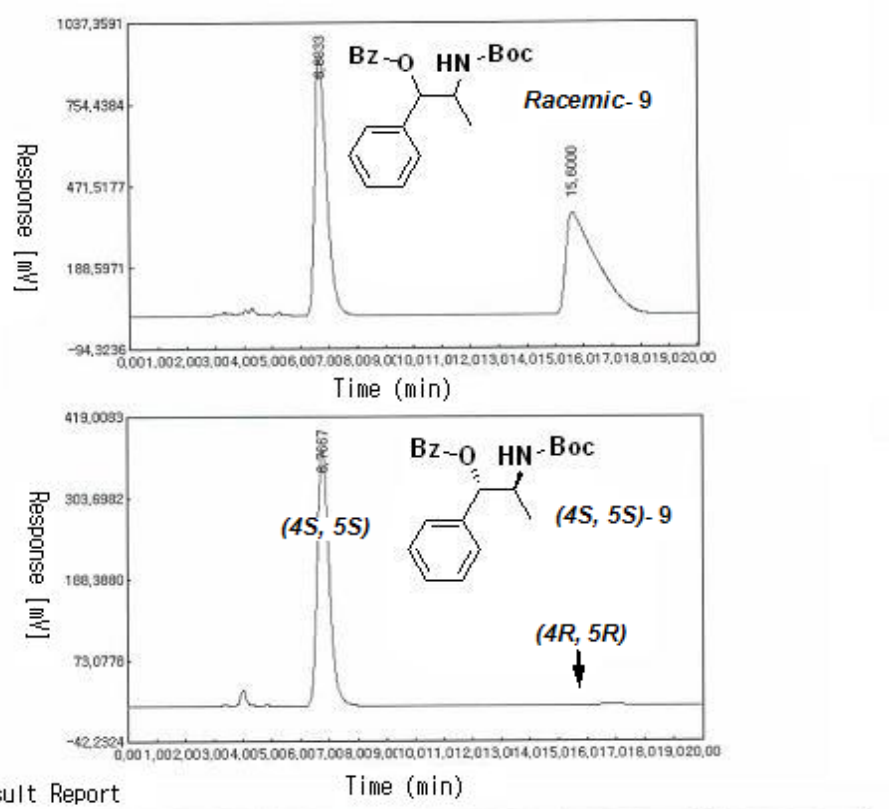




Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	7.1500	375.0975	BB	43.0000	100.0000
Total		375.0975			

3-3. (*4S,5S*)-2-(*tert*-Butoxycarbonyl-amino)-1-(benzoyl-oxy)-1-phenyl-propane, (*4S,5S*)-9

Chiralcel AD-H, 40% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(major) = 6.8 min, tr(minor) = 15.6 min.

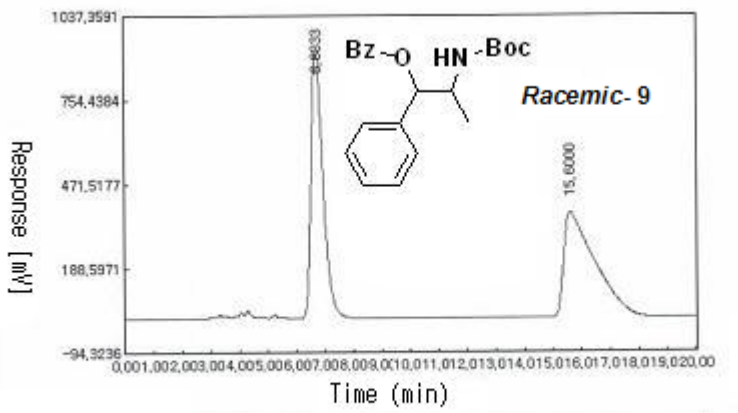


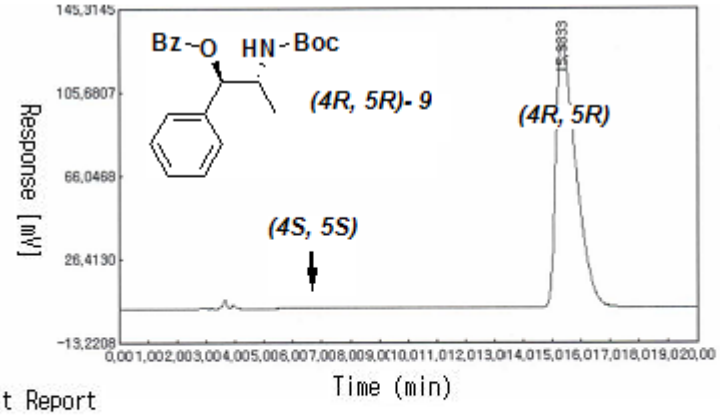
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	6.7667	10875.9801	BB	107.0000	100.0000
Total		10875.9805			

3-4. (*4R,5R*)-2-(*tert*-Butoxycarbonyl-amino)-1-(benzoyl-oxy)-1-phenyl-propane, (*4R,5R*)-9

Chiralcel AD-H, 40% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(minor) = 6.8 min, tr(major) = 15.6 min.



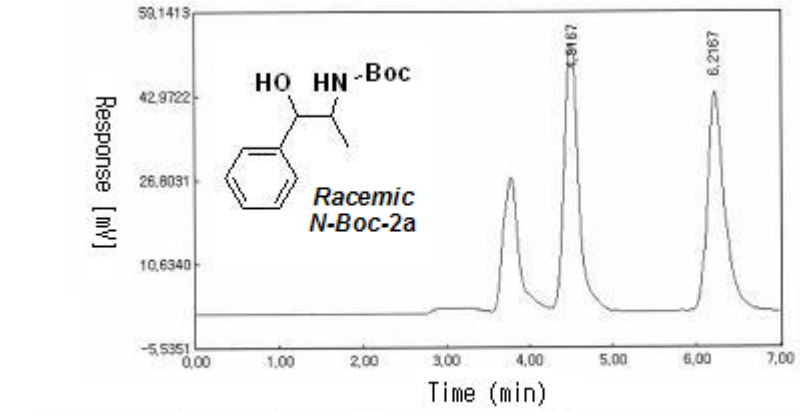


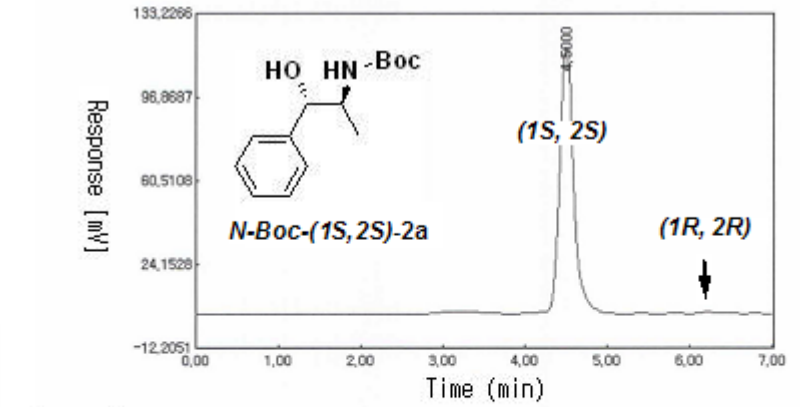
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	15.3333	6226.1009	BB	132.0000	100.0000
Total		6226.1011			

3-5. (*1S,2S*)-2-(*tert*-Butoxycarbonyl-amino)-1-phenyl-propan-1-ol, *N-Boc-(1S,2S)*-2a

Chiralcel AD-H, 30% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(major) = 4.5 min, tr(minor) = 6.2 min.



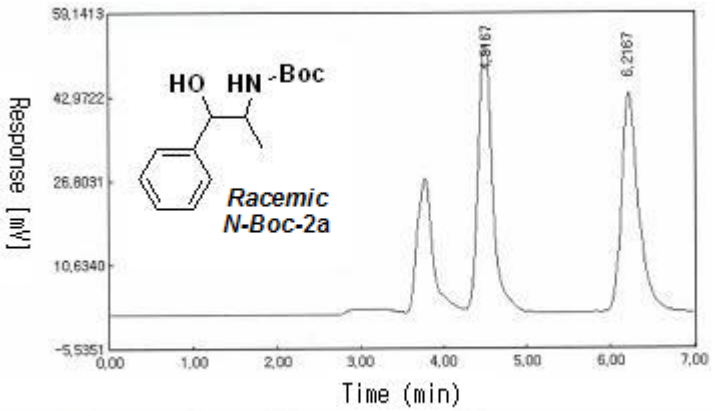


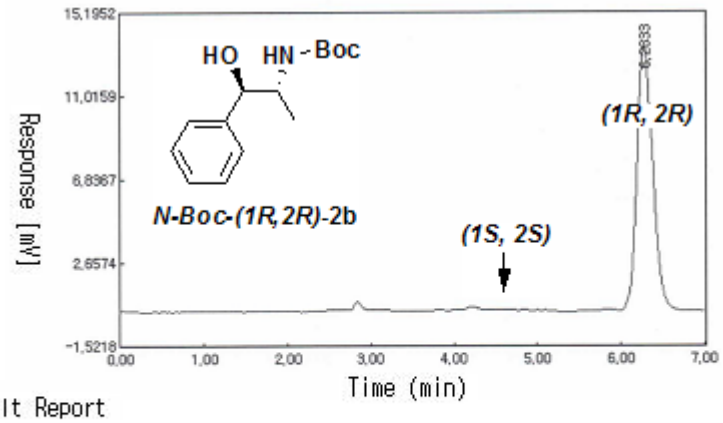
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	4.5000	1374.6741	BB	65.0000	100.0000
Total		1374.6741			

3-6. (*1R,2R*)-2-(*tert*-Butoxycarbonyl-amino)-1-phenyl-propan-1-ol, *N-Boc-(1R,2R)*-2b

Chiralcel AD-H, 30% isopropanol/hexanes, 1.0 mL/min, 254 nm, tr(minor) = 4.5 min, tr(major) = 6.2 min.



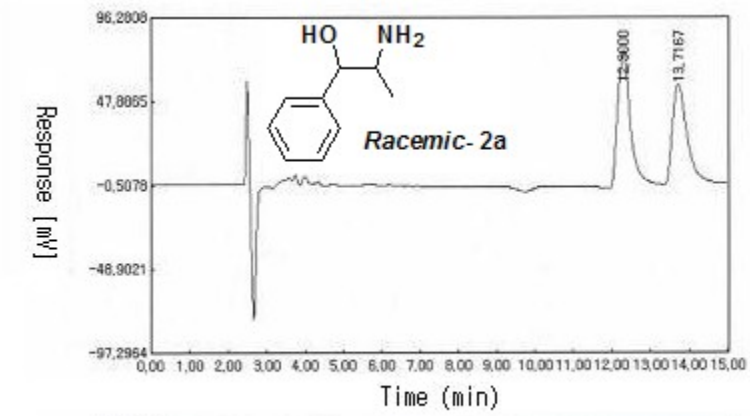


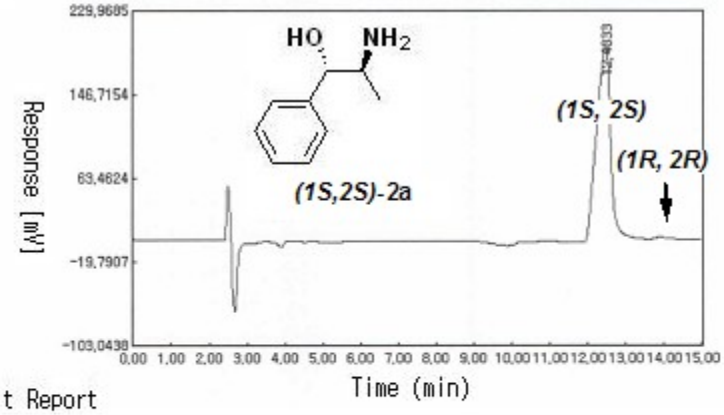
Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	6.2833	174.7468	BB	34.0000	100.0000
Total		174.7468			

3-7. (*1S,2S*)-2-Amino-1-phenyl-propan-1-ol, (*1S,2S*)-2a, [(*1S,2S*)-(+)-norpseudoephedrine]

Chiralcel AD-H, 0.1/5/95 Et_3N/isopropanol/hexanes (v/v/v), 1.2 mL/min, 254 nm, tr(major) = 12.5 min, tr(minor) = 13.9 min.



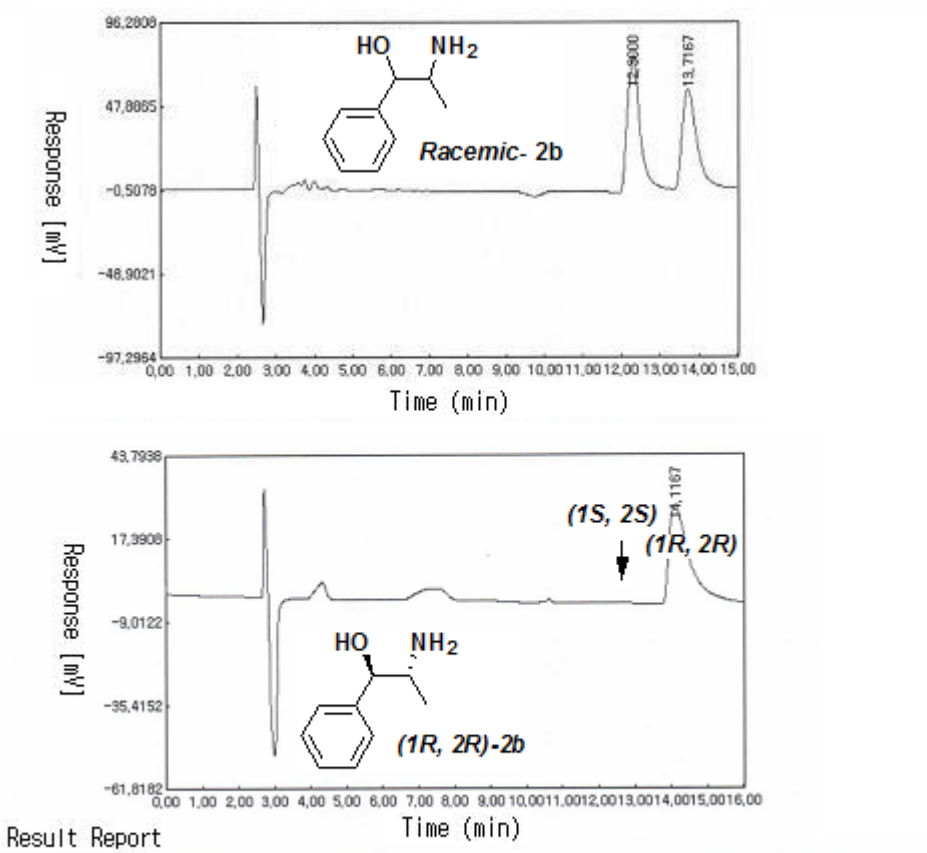


Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	12.4833	5181.6801	BB	86.0000	100.0000
Total		5181.6802			

3-8. (*1R,2R*)-2-Amino-1-phenyl-propan-1-ol, (*1R,2R*)-2b, [(*1R,2R*)-(-)-norpseudoephedrine]

Chiralcel AD-H, 0.1/5/95 Et_3N/isopropanol/hexanes (v/v/v), 1.2 mL/min, 254 nm, tr(minor) = 12.5 min, tr(major) = 13.9 min.

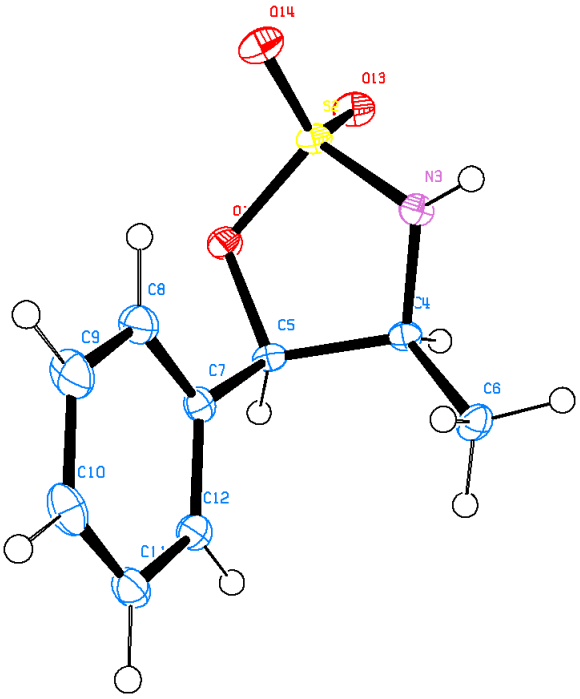


Result Report

Peak #	Time (min)	Area [mV*s]	BL	wide(sec)	Area (%)
1	14.1167	1106.7888	BB	88.0000	100.0000
Total		1106.7888			

2. X-ray crystallography analysis data of (4*S*,5*R*)-7

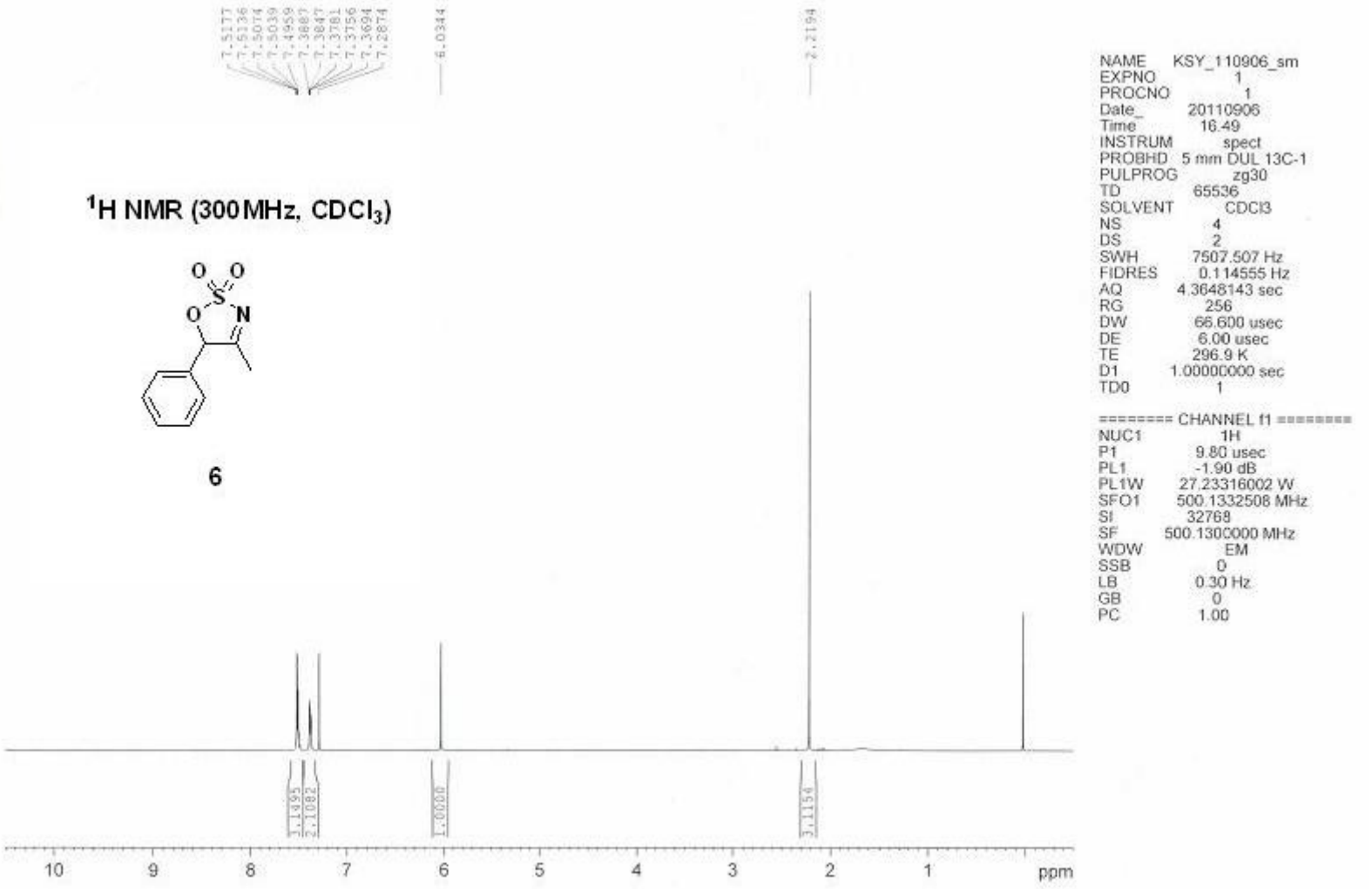
CCDC 872756 contains the supplementary crystallographic data for (4*S*,5*R*)-**7**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.



Crystal data for (4*S*,5*R*)-7

Identification code	20120117lt2_0m	
Empirical formula	C9 H11 N O3 S	
Formula weight	213.25	
Temperature	100(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 6.6397(1) Å	= 90°
	b = 9.9763(2) Å	= 92.753(1)°
	c = 7.5180(2) Å	= 90°
Volume	497.415(18) $Å^3$	
Z	2	
Density (calculated)	1.424 Mg/m^3	

Absorption coefficient	0.305 mm^{-1}
F(000)	224
Crystal size	0.22 x 0.18 x 0.08 mm^3
Theta range for data collection	2.71 to 28.57°
Index ranges	-8<=h<=8, -13<=k<=13, -9<=l<=10
Reflections collected	4518
Independent reflections	2000 [R(int) = 0.0172]
Completeness to theta = 28.57°	97.8 %
Absorption correction	Multi-scan
Max. and min. transmission	0.9760 and 0.9359
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2000 / 1 / 127
Goodness-of-fit on F^2	1.029
Final R indices [I>2sigma(I)]	R1 = 0.0289, wR2 = 0.0754
R indices (all data)	R1 = 0.0307, wR2 = 0.0770
Absolute structure parameter	0.07(7)
Largest diff. peak and hole	0.412 and -0.451 $e.Å^{-3}$



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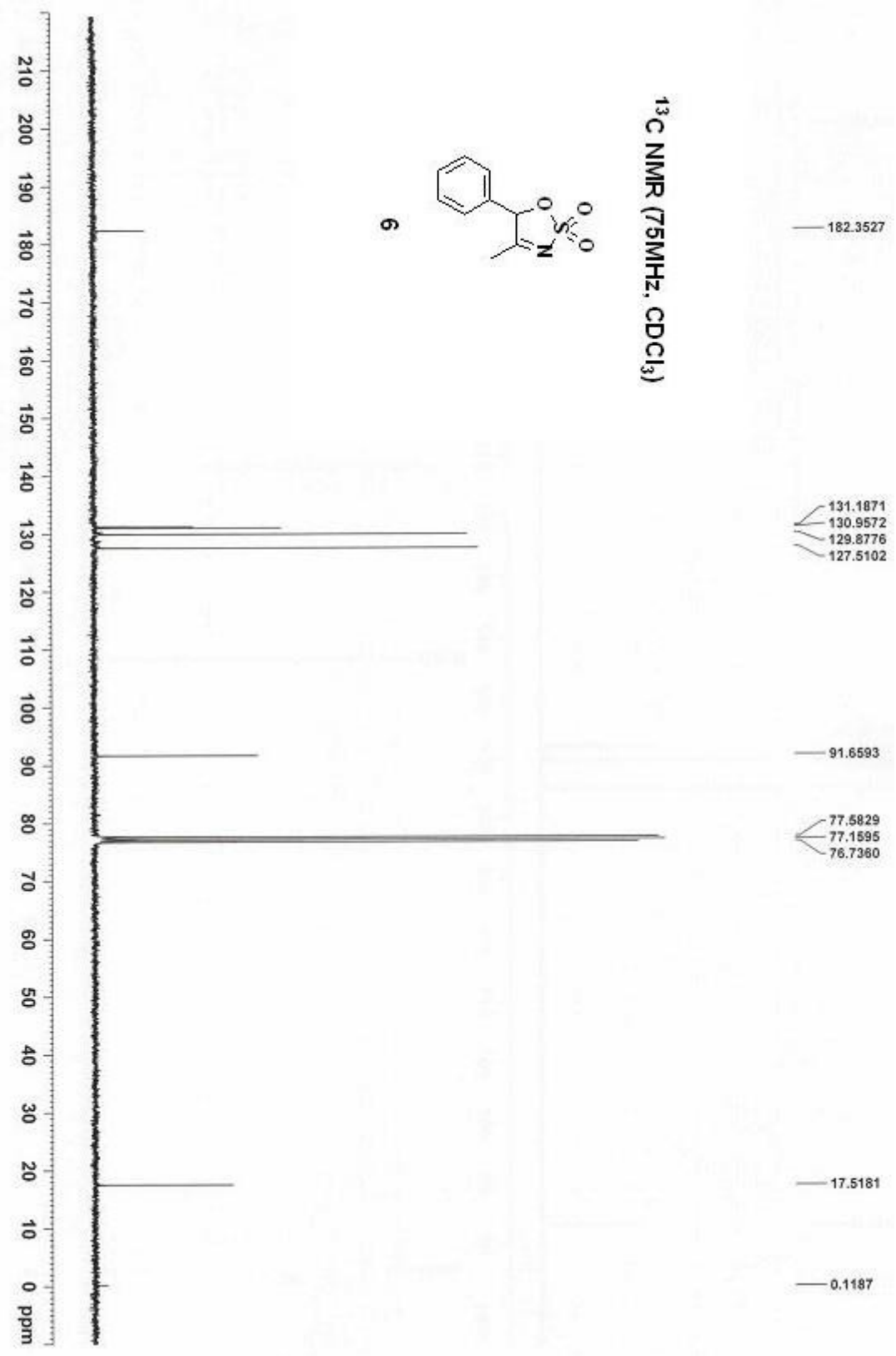
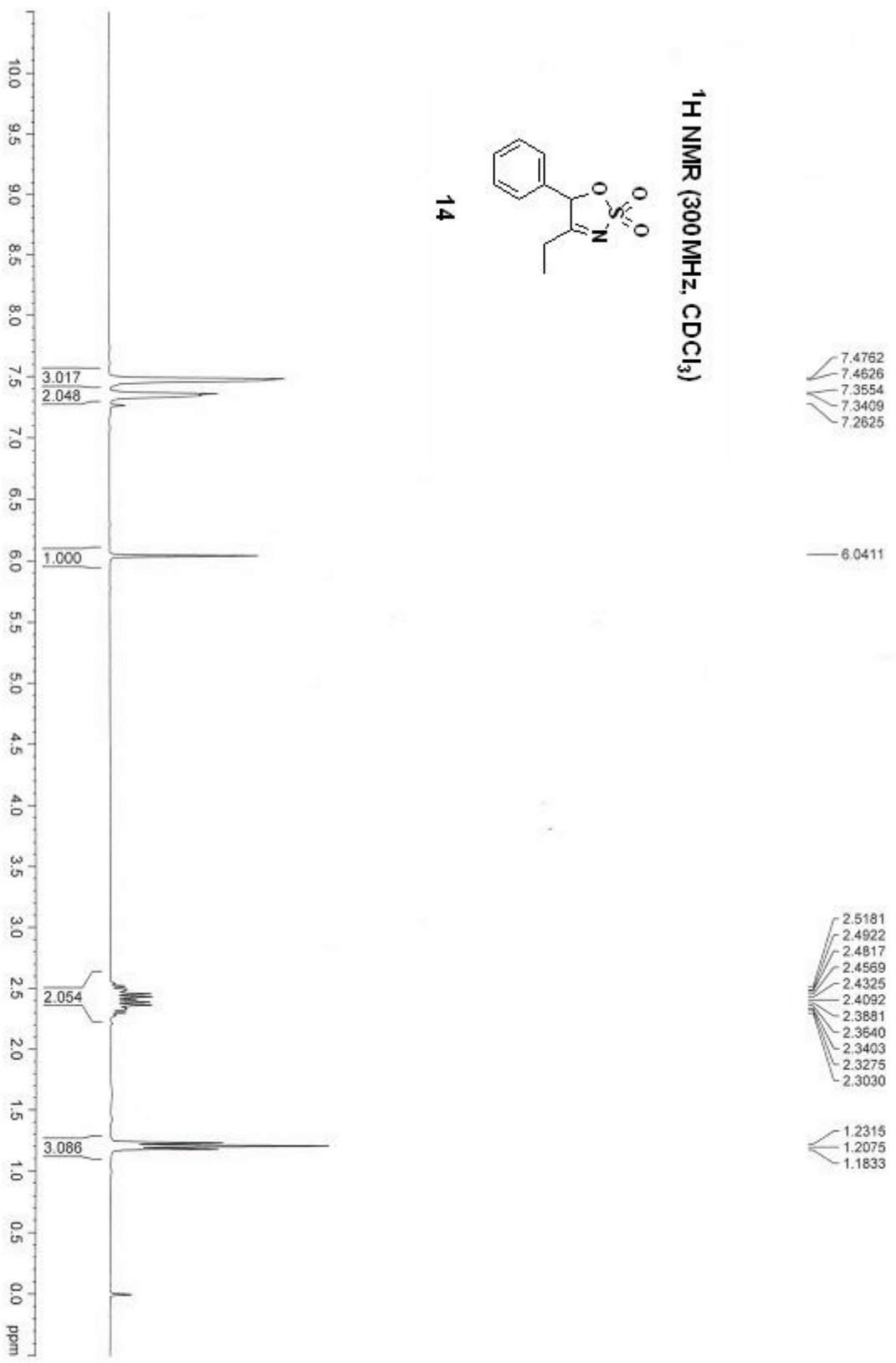
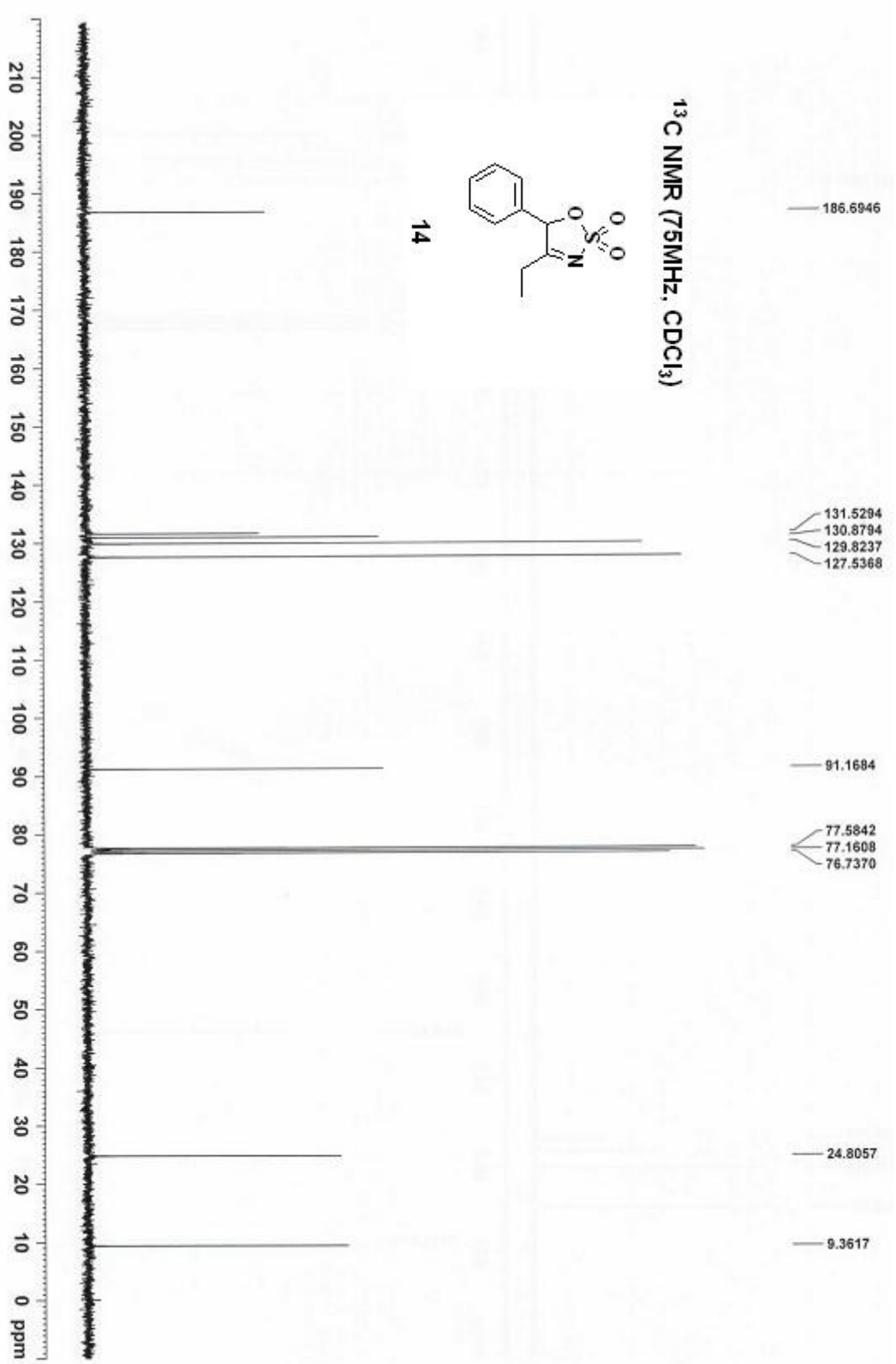
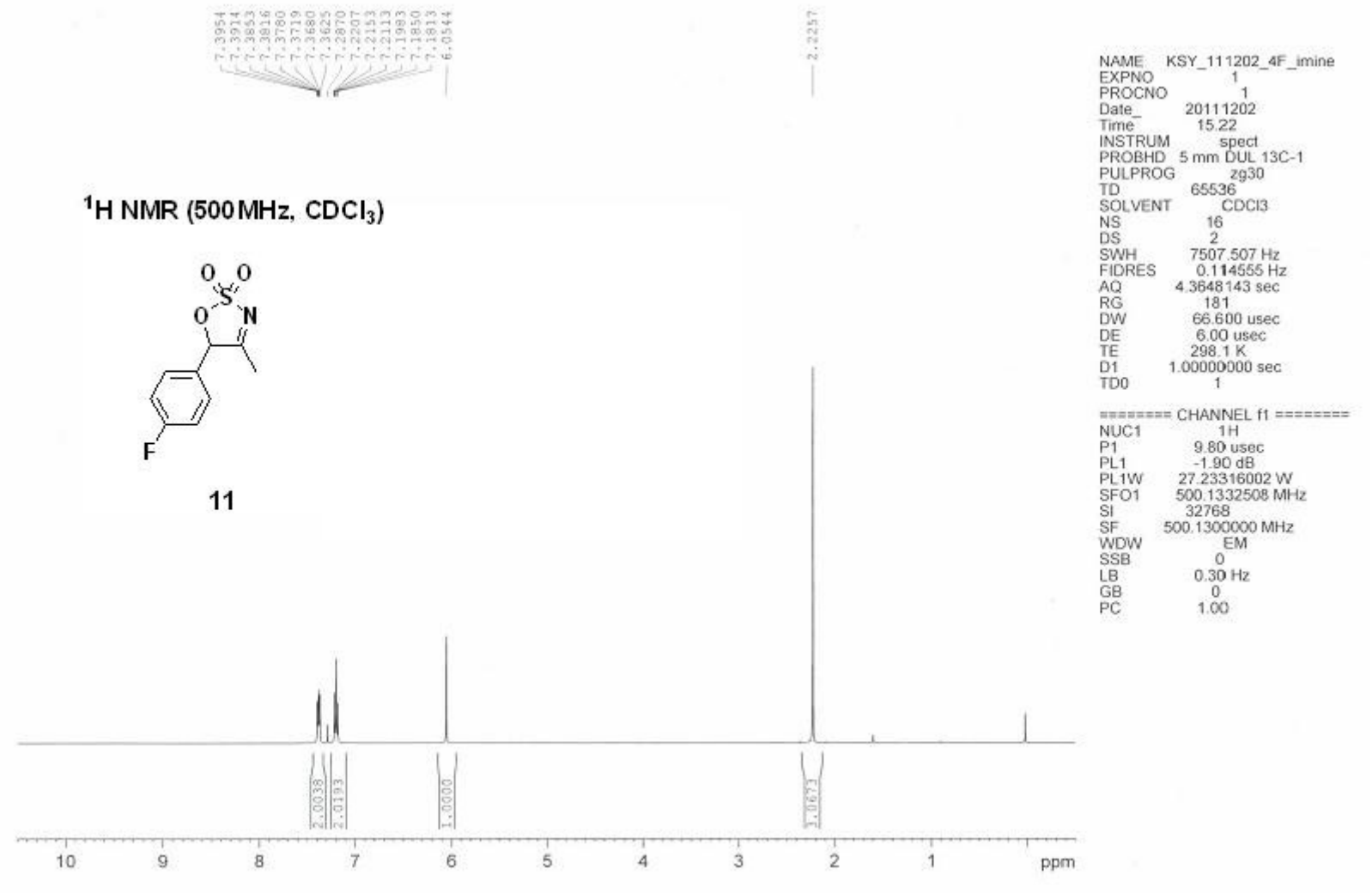
EXPNO 1
PROCNO 1
Date_ 20110906
Time 16.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 256
DW 66.600 usec
DE 6.00 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1

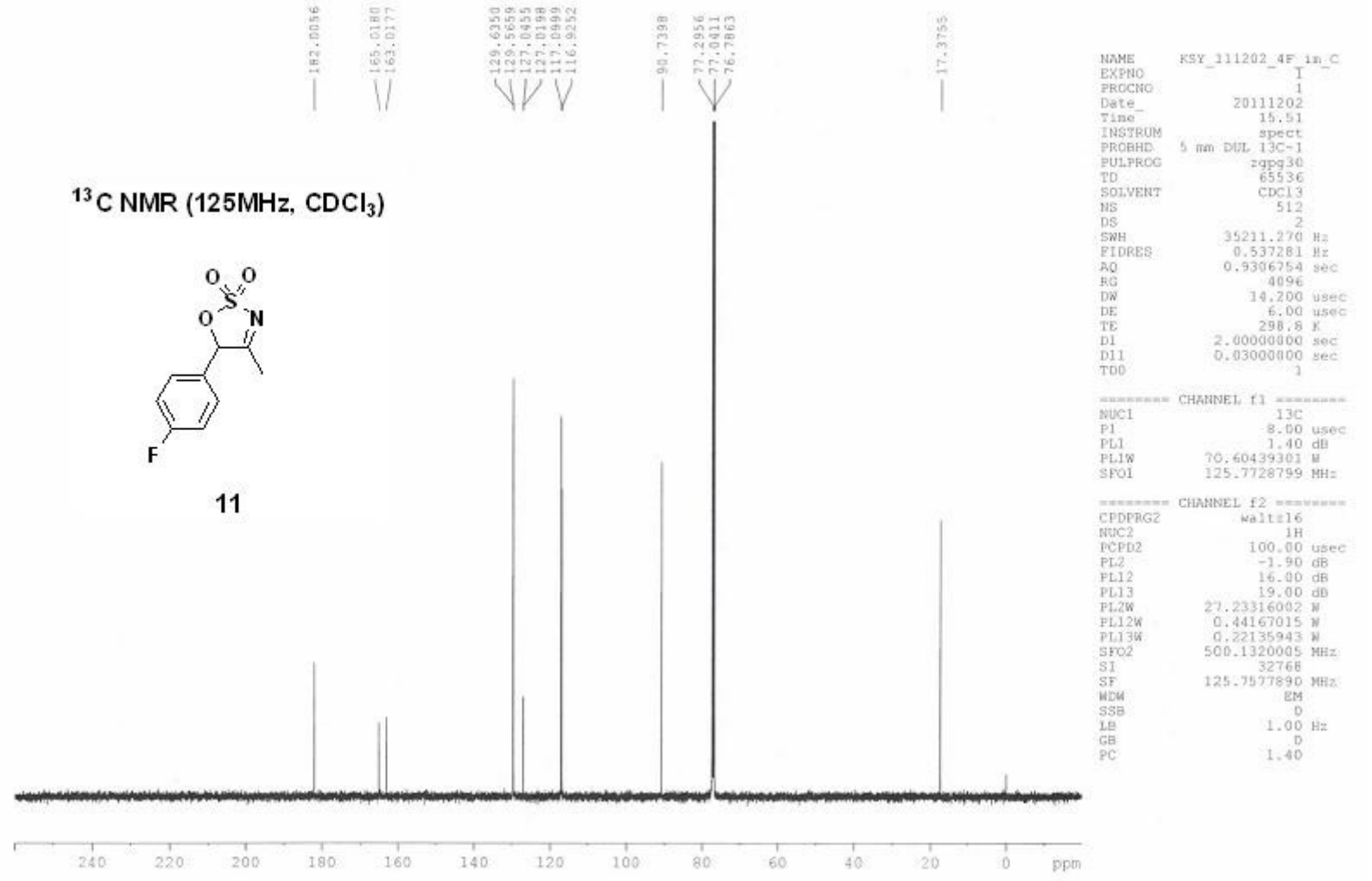
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


^{13}C NMR (75MHz, $CDCl_3$)
6
182.3527
131.1871
130.9572
129.8776
127.5102
91.6593
77.5829
77.1595
76.7360
17.5181
0.1187
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

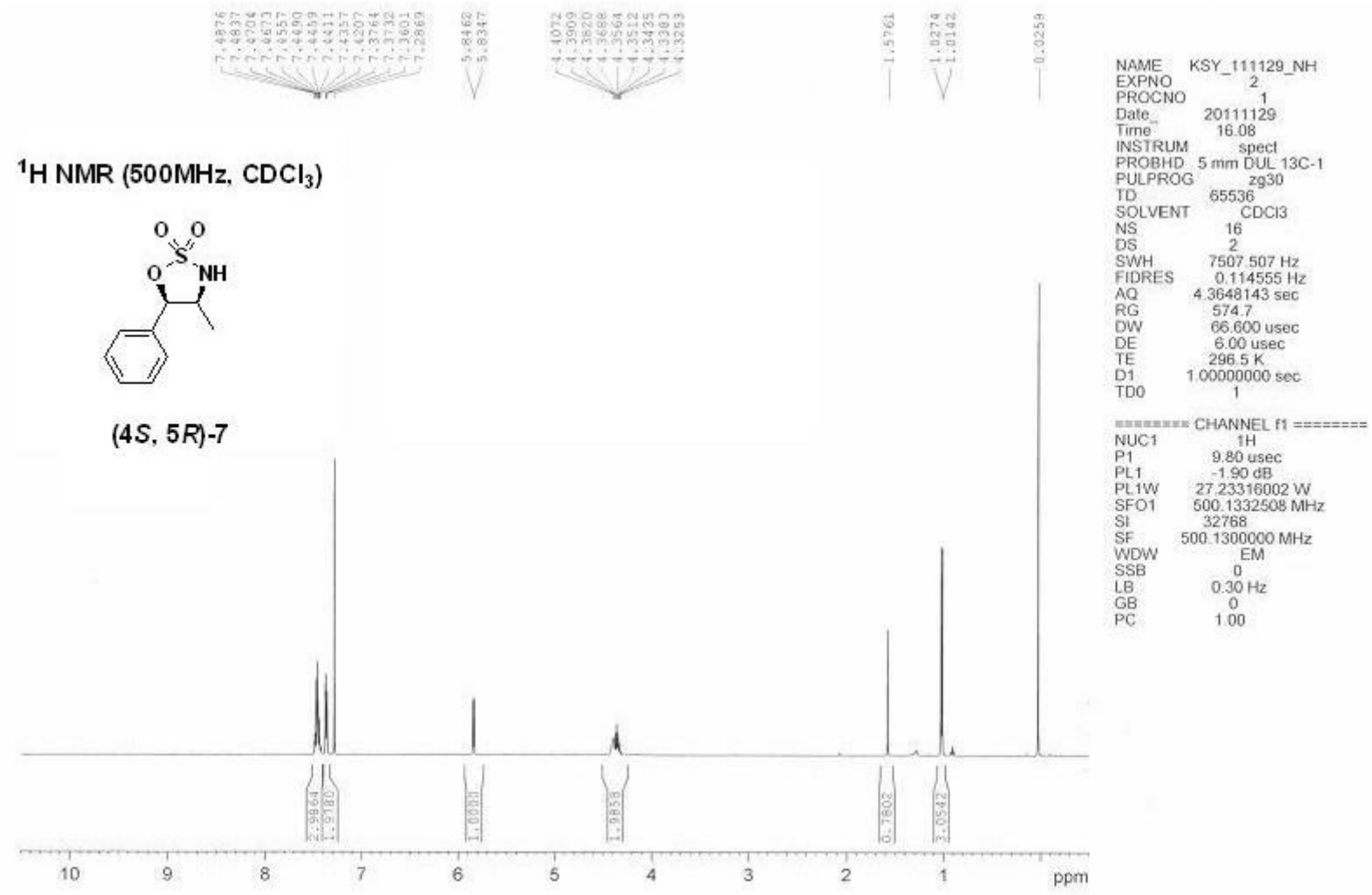

14
^{1}H NMR (300 MHz, $CDCl_3$)
7.4762
7.4626
7.3554
7.3409
7.2625
6.0411
2.5181
2.4922
2.4817
2.4569
2.4325
2.4092
2.3881
2.3640
2.3403
2.3275
2.3030
1.2315
1.2075
1.1833
3.017
2.048
1.000
2.054
3.086
ppm


^{13}C NMR (75MHz, $CDCl_3$)
14
186.6946
131.5294
130.8794
129.8237
127.5368
91.1684
77.5842
77.1608
76.7370
24.8057
9.3617
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3954
7.3914
7.3853
7.3816
7.3780
7.3719
7.3680
7.3625
7.2870
7.2207
7.2153
7.2113
7.1983
7.1850
7.1813
6.0544
2.2257
^{1}H NMR (500 MHz, $CDCl_3$)
11
NAME KSY_111202_4F_imine
EXPNO 1
PROCNO 1
Date_ 20111202
Time 15.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 181
DW 66.600 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.0038
2.0193
1.0000
3.0673
10
9
8
7
6
5
4
3
2
1
ppm



NAME	KSY_111202_4F in_C
EXPNO	1
PROCNO	1
Date_	20111202
Time	15.51
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	2
SWH	35211.270 Hz
FIDRES	0.537281 Hz
AQ	0.9306754 sec
RG	4096
DW	14.200 usec
DE	6.00 usec
TE	298.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7728799 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

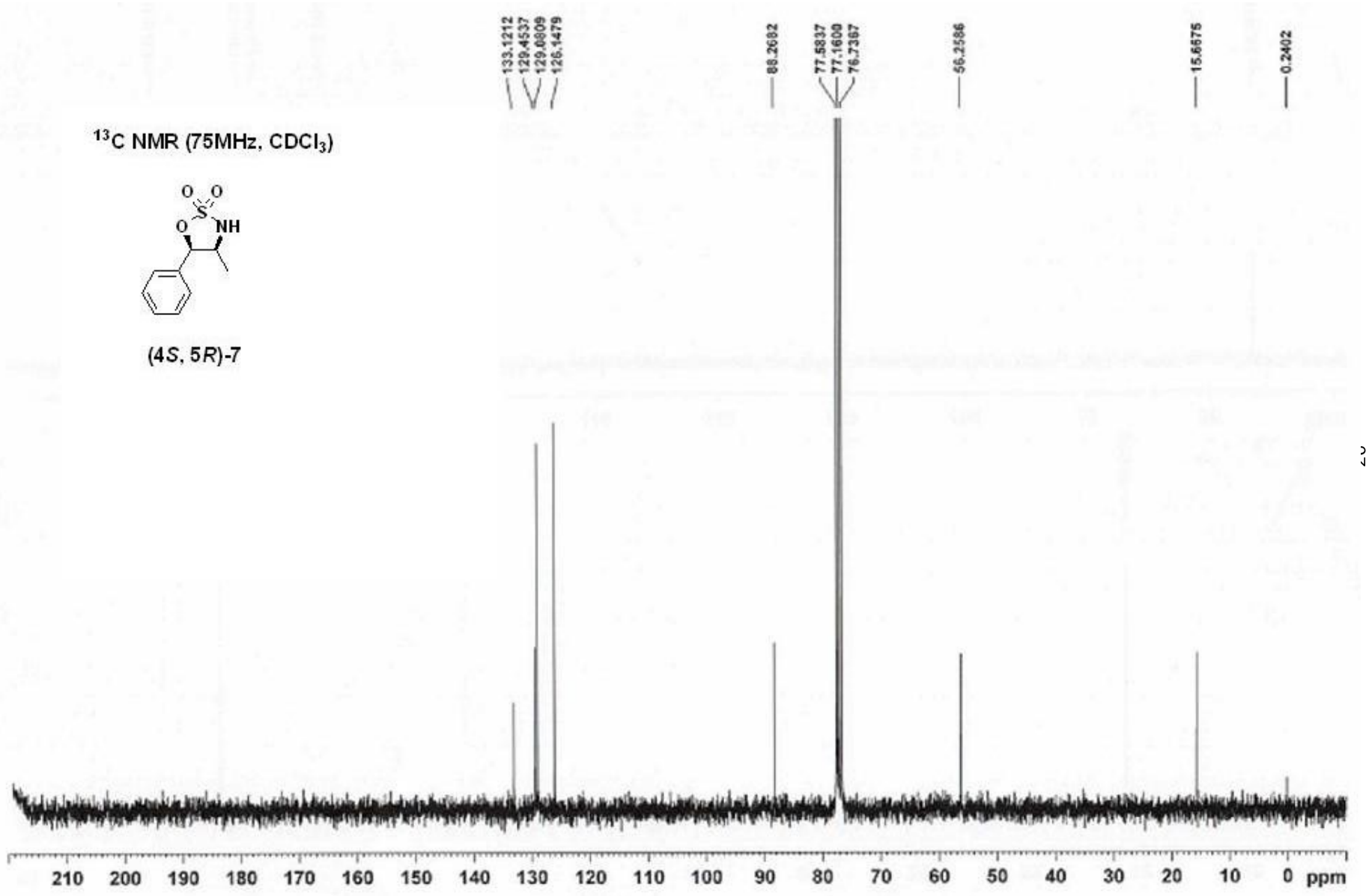


^{1}H NMR (500MHz, $CDCl_3$)

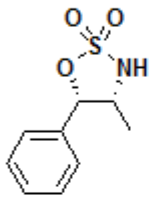
(4*S*, 5*R*)-7

NAME KSY_111129_NH
EXPNO 2
PROCNO 1
Date_ 20111129
Time 16.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 574.7
DW 66.600 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


133.1212
129.4537
129.0809
126.1479
88.2682
77.5837
77.1600
76.7367
56.3586
15.6575
0.2402
^{13}C NMR (75MHz, $CDCl_3$)
O
O
S
O
NH
(4S, 5R)-7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

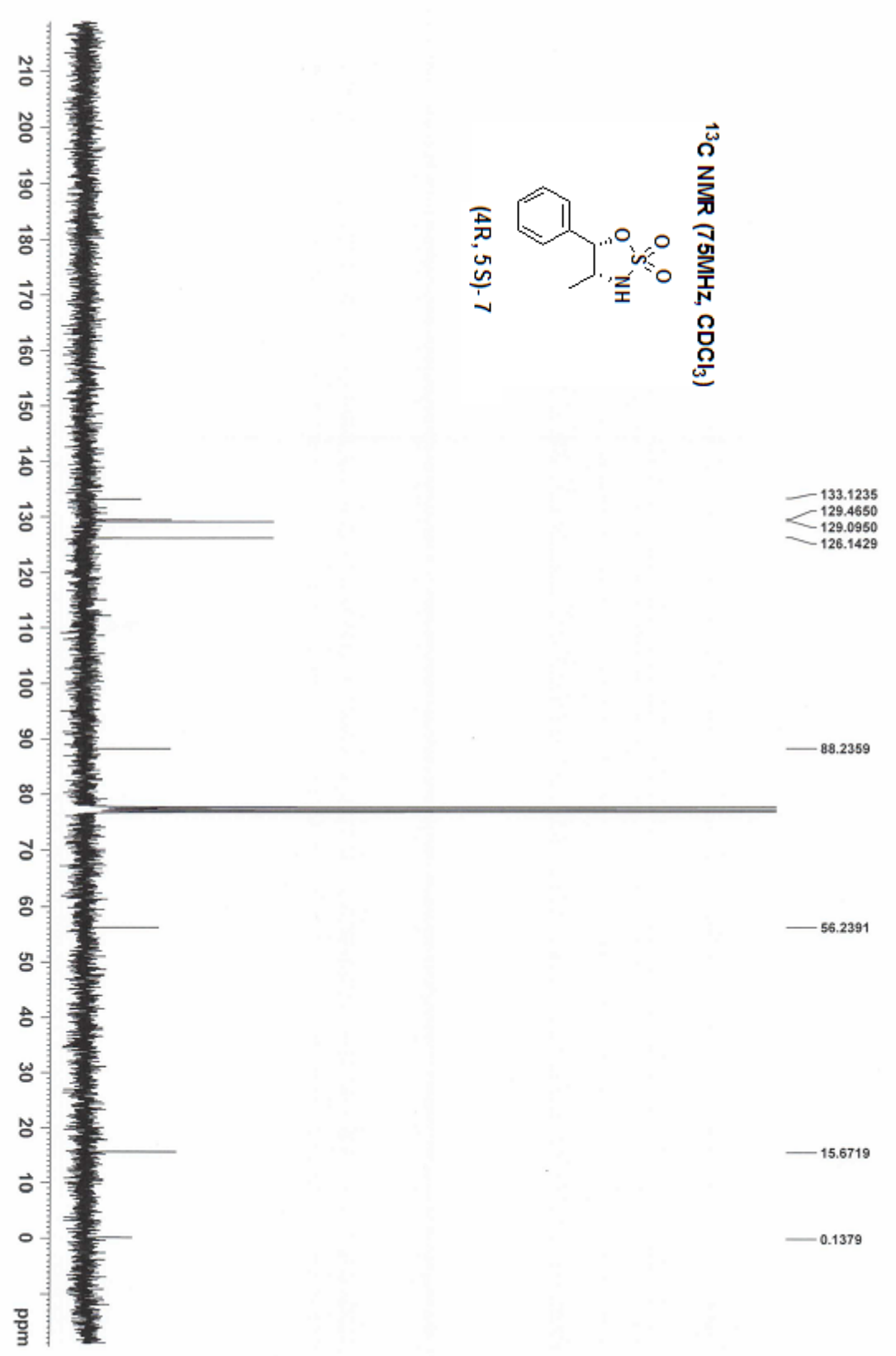
^{1}H NMR (500MHz, $CDCl_3$)

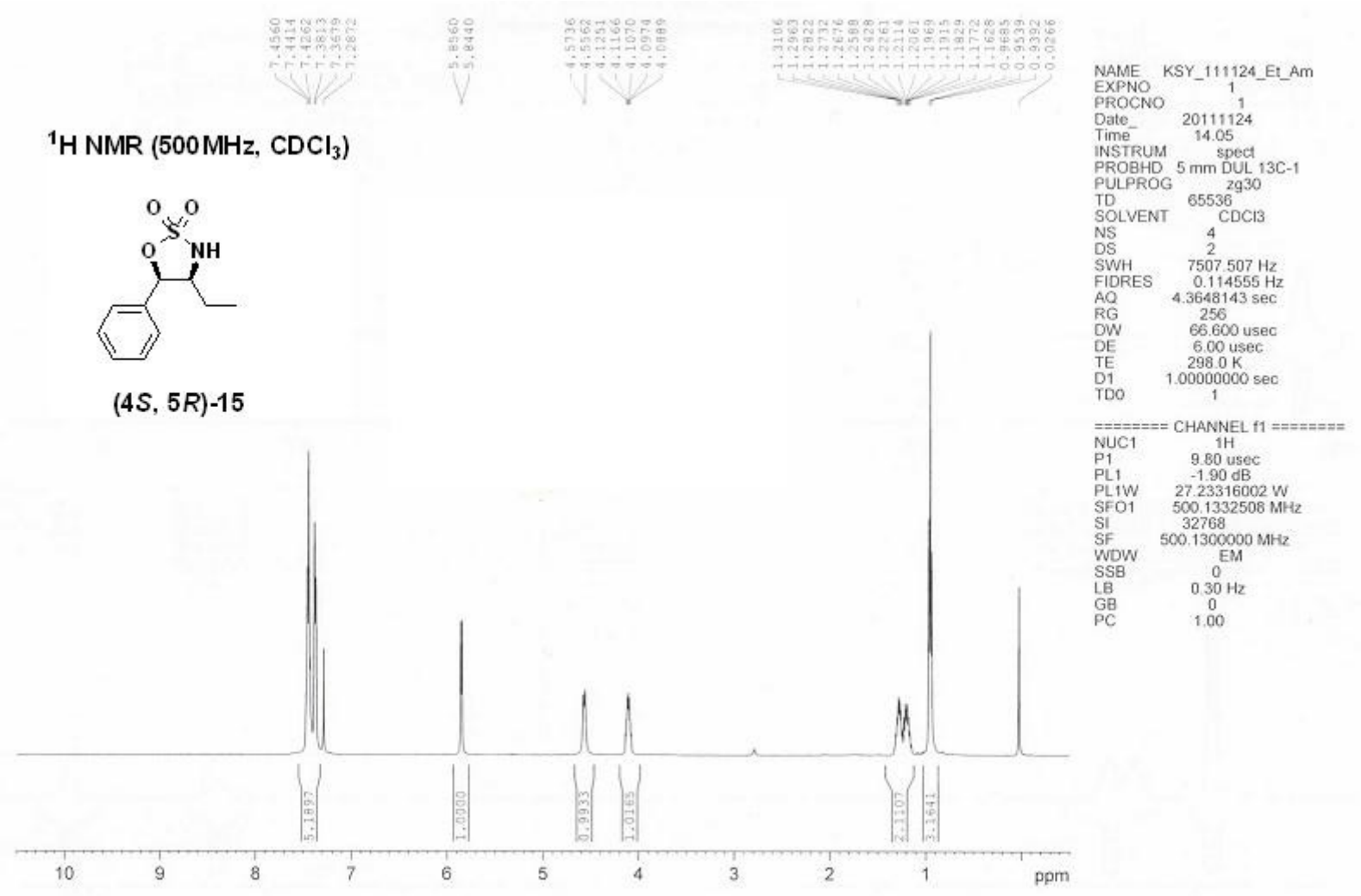


(4R, 5S)- 7

NAME KSY_111004_NH
EXPNO 1
PROCNO 1
Date_ 20111004
Time 16.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 574.7
DW 66.600 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


^{13}C NMR (75MHz, CDCl$_3$)
(4R, 5S)- 7
133.1235
129.4650
129.0950
126.1429
88.2359
56.2391
15.6719
0.1379
ppm

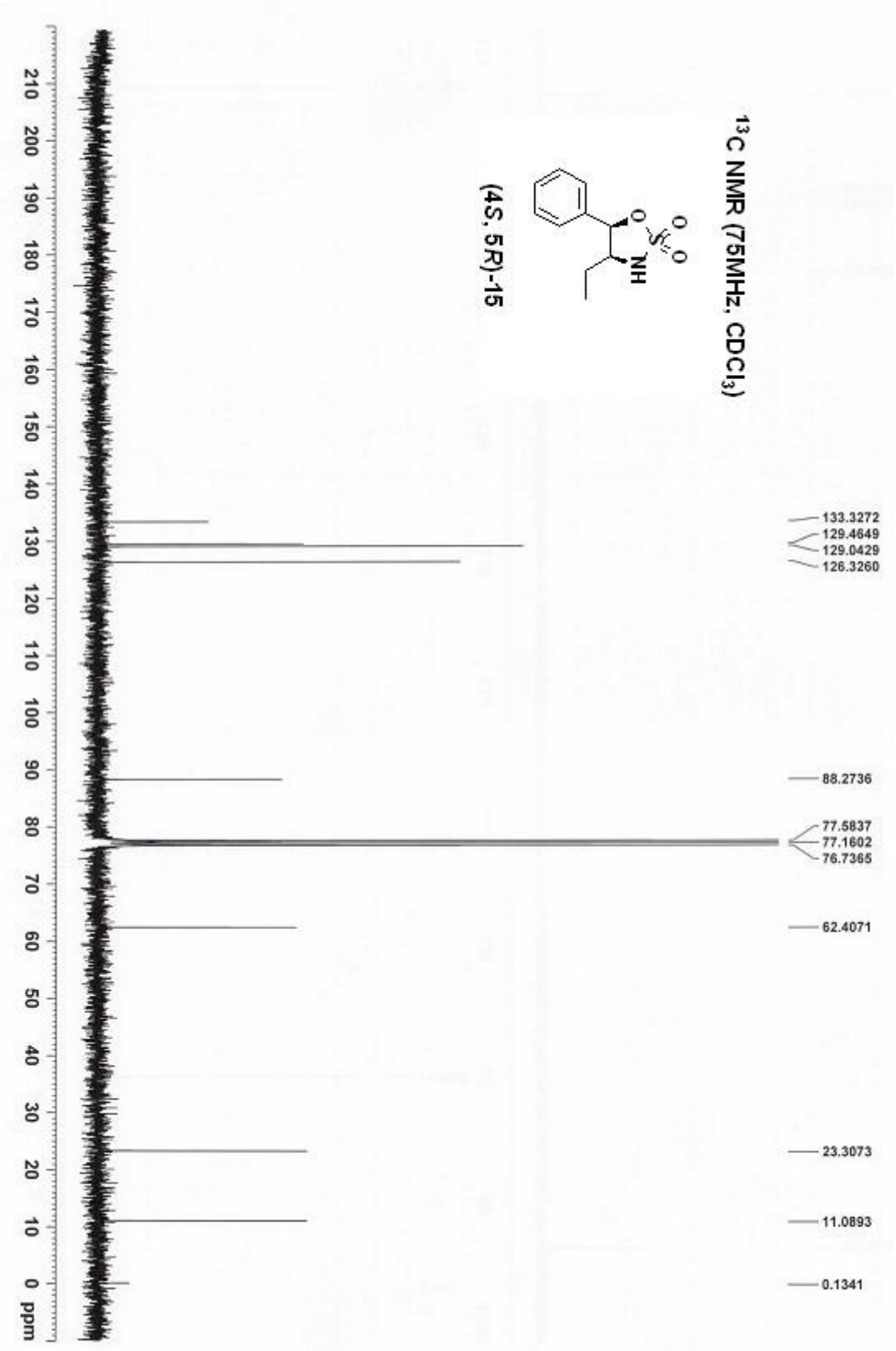


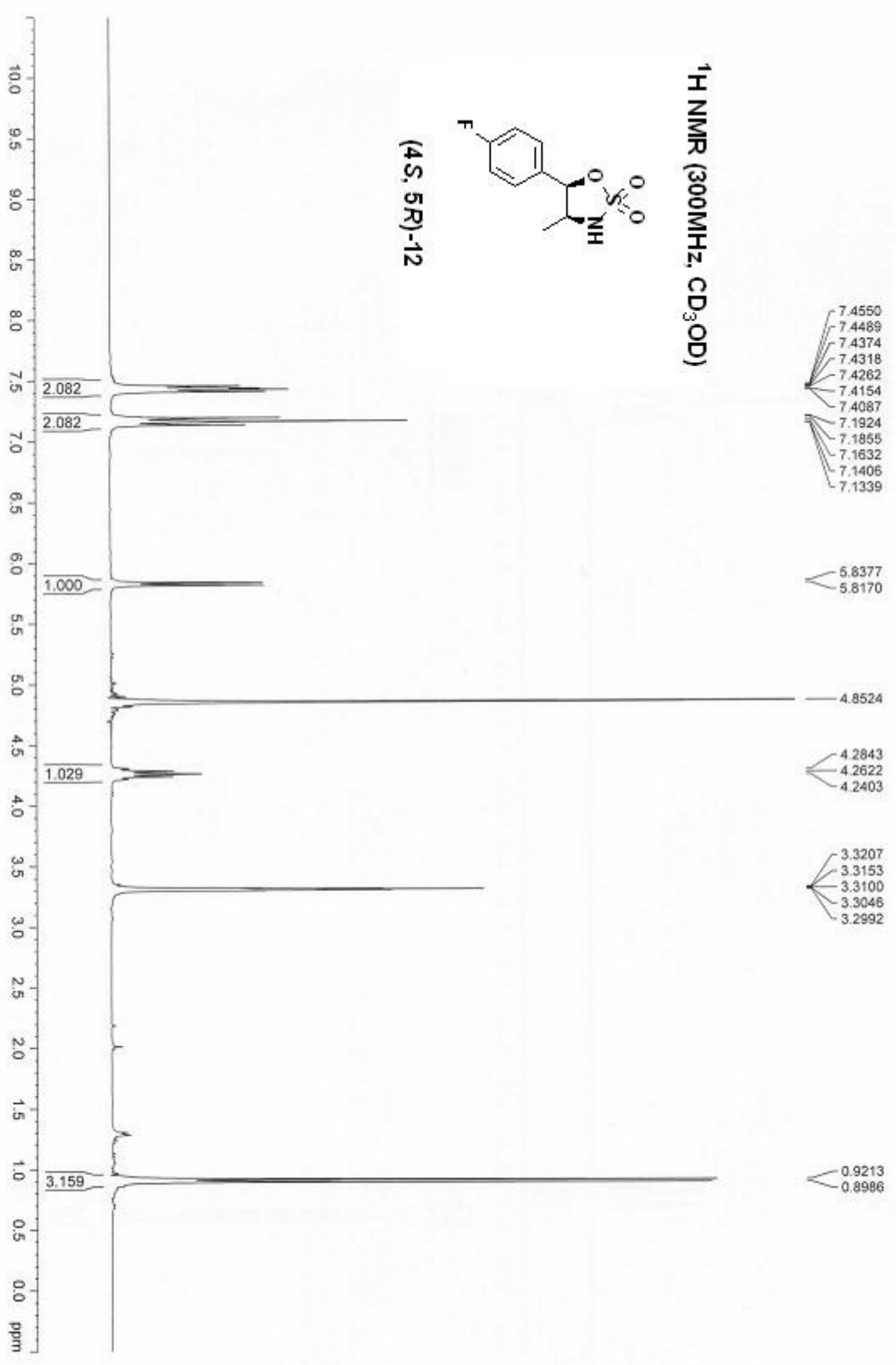
^{1}H NMR (500 MHz, $CDCl_3$)

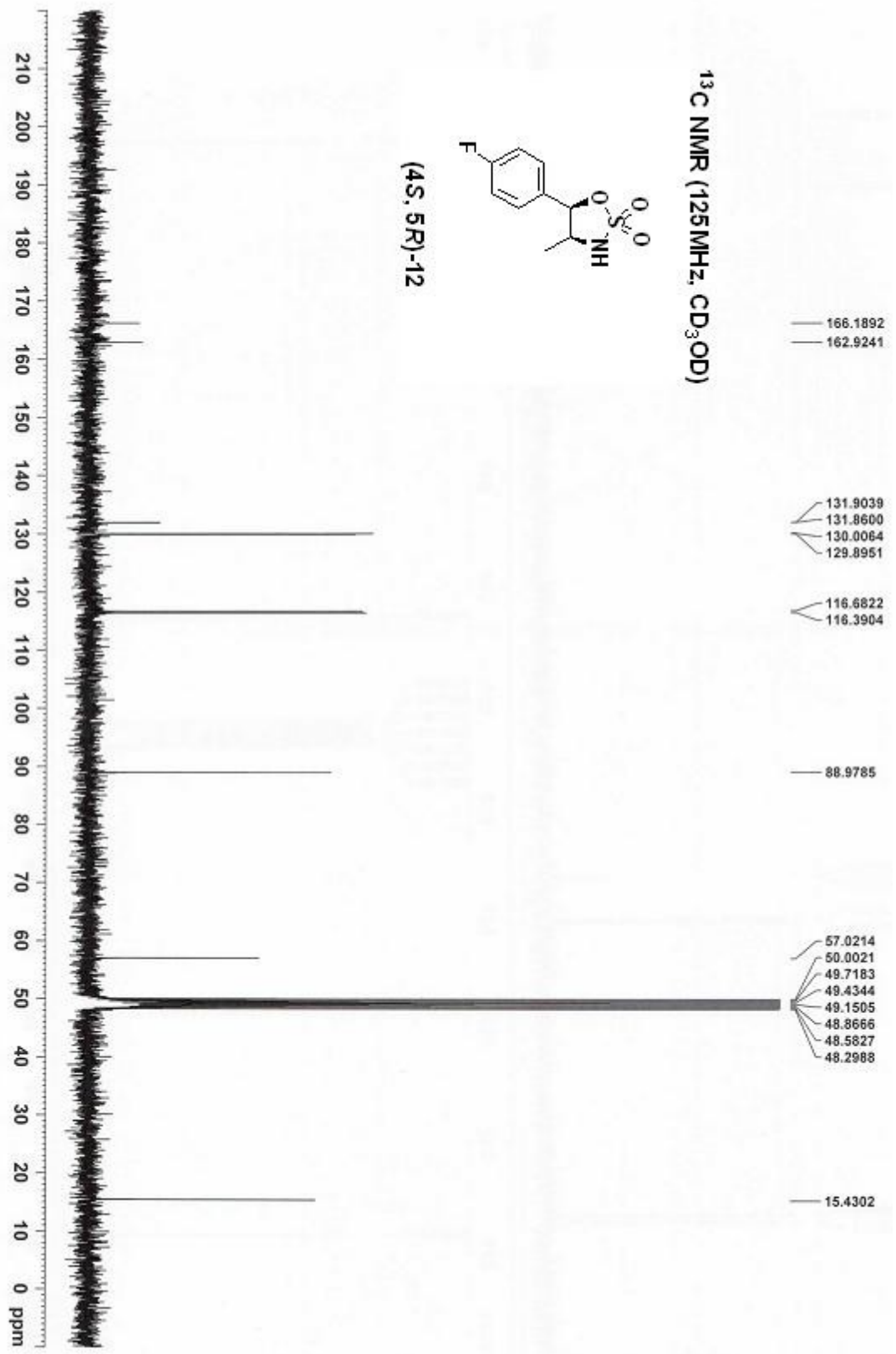
(4*S*, 5*R*)-15

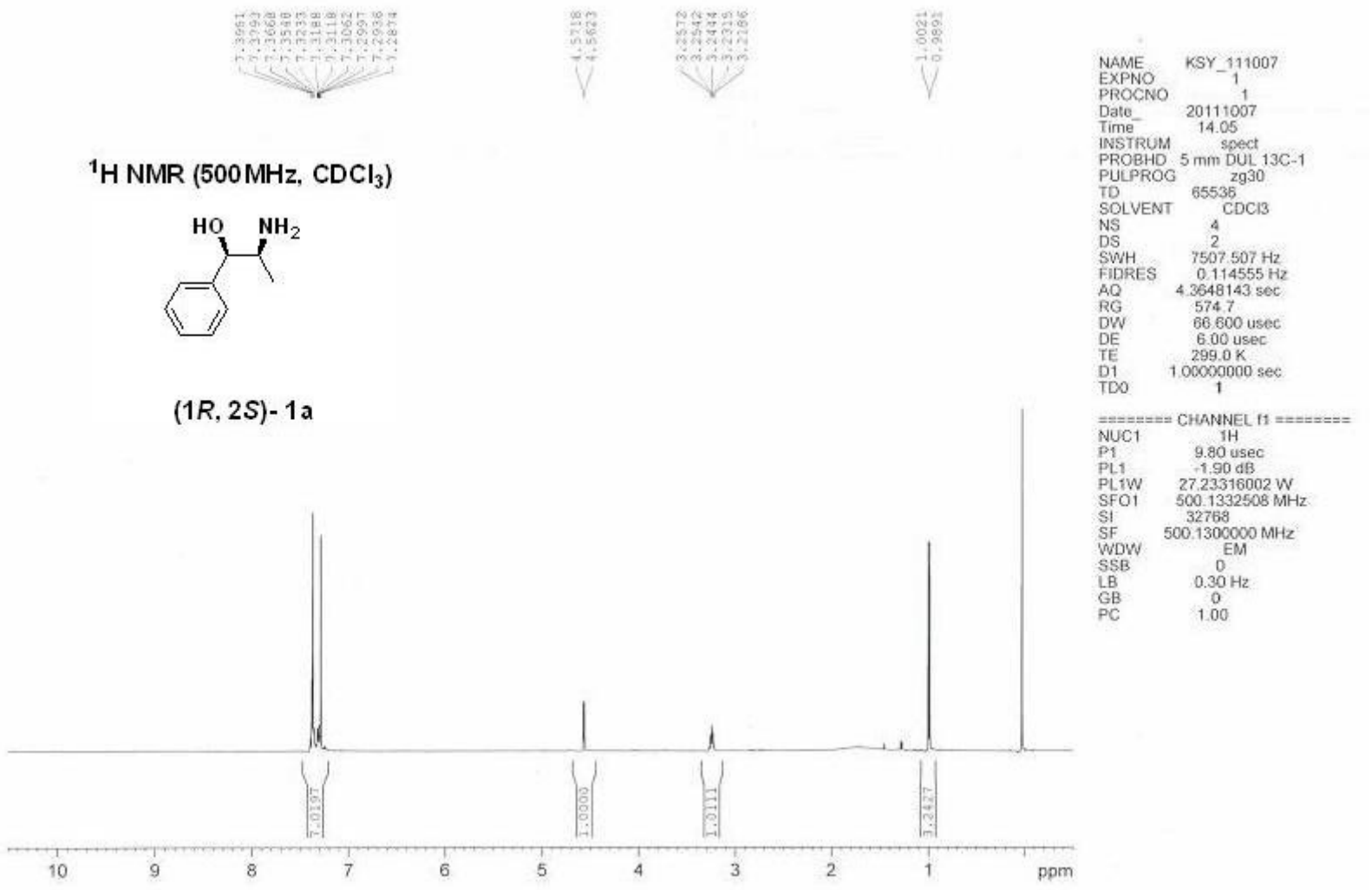
NAME KSY_111124_Et_Am
EXPNO 1
PROCNO 1
Date_ 20111124
Time 14.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 256
DW 66.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

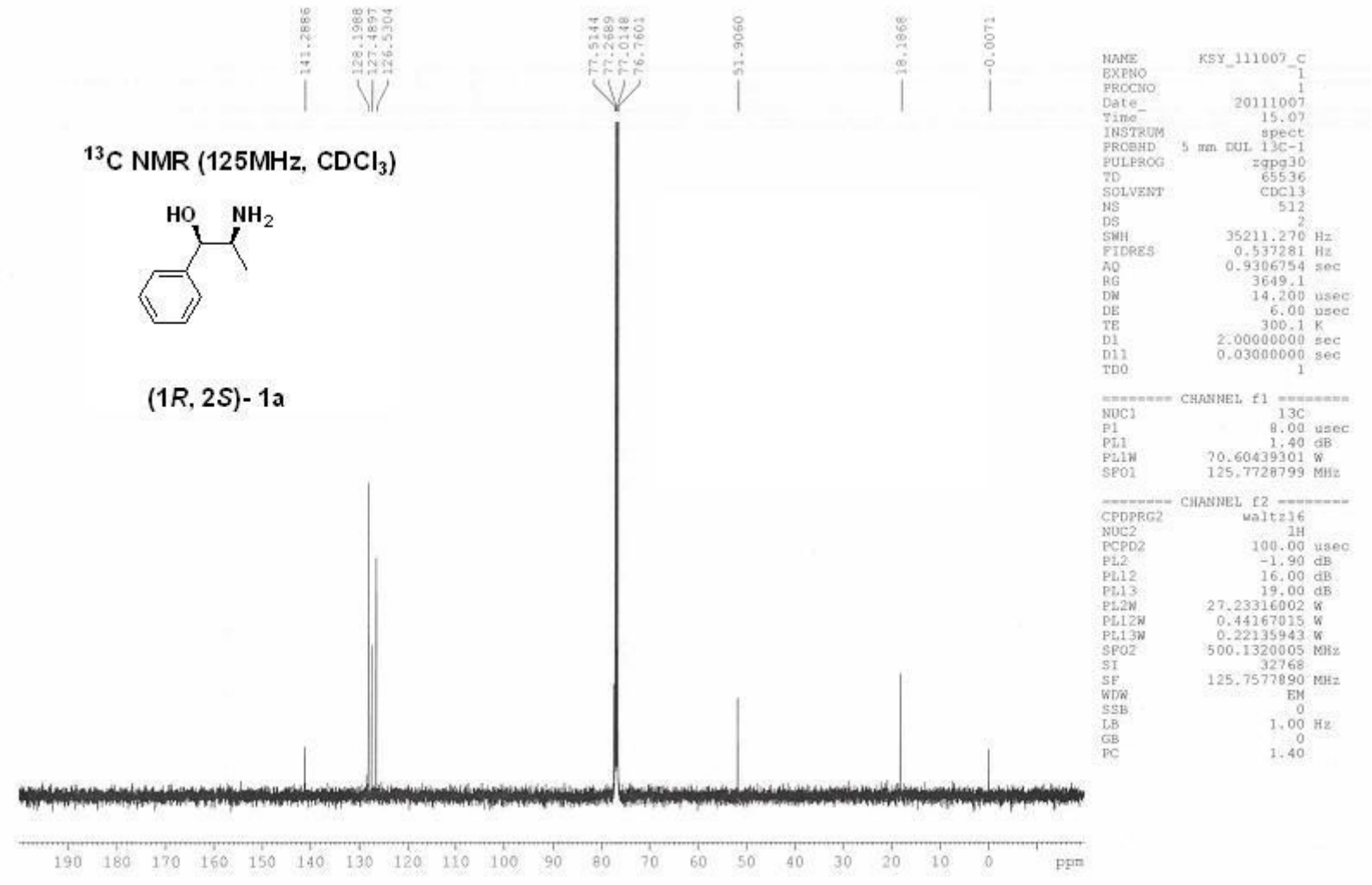
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

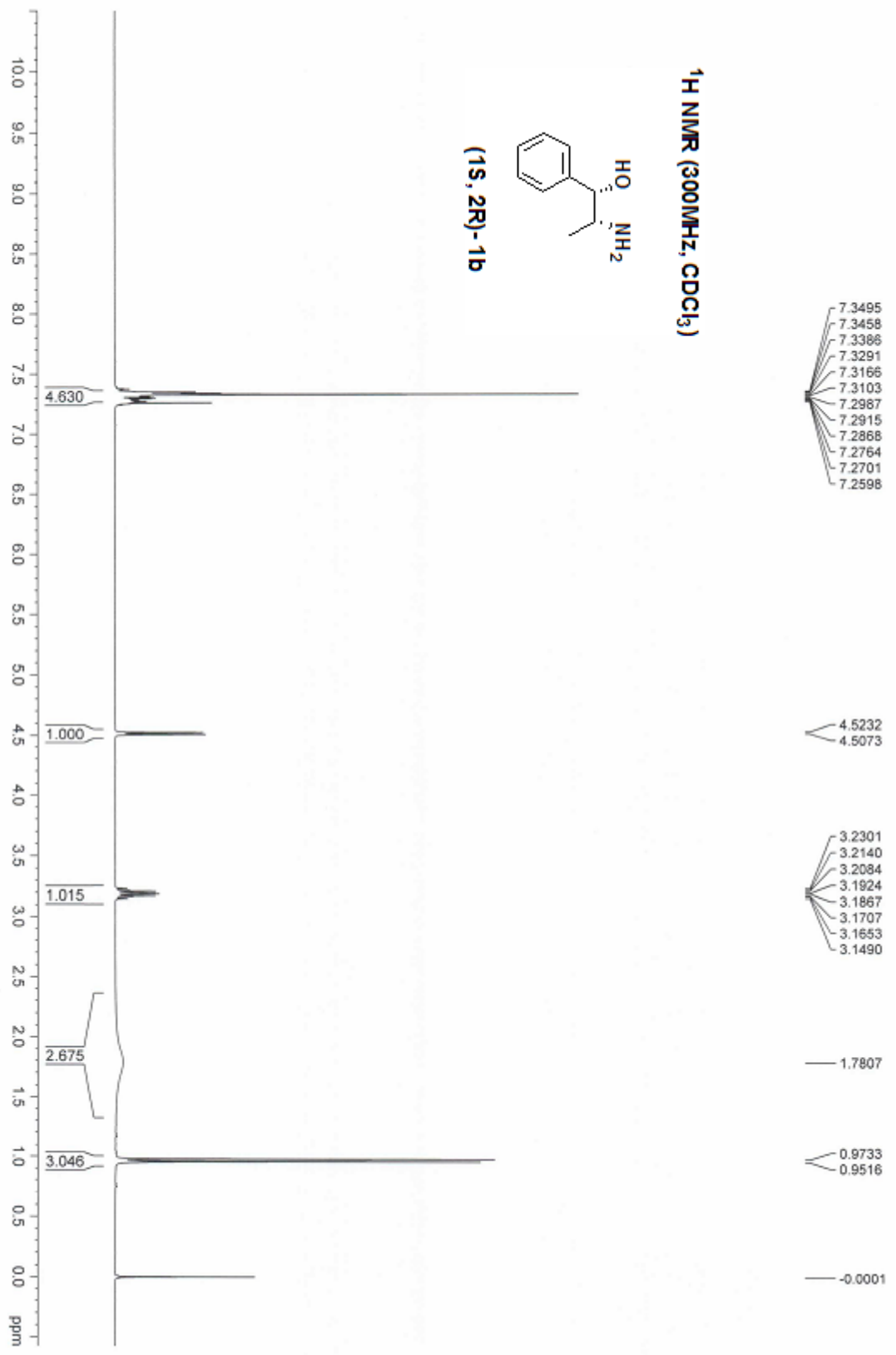

^{13}C NMR (75MHz, $CDCl_3$)
(4S, 5R)-15
NH
133.3272
129.4649
129.0429
126.3260
88.2736
77.5837
77.1602
76.7365
62.4071
23.3073
11.0893
0.1341
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

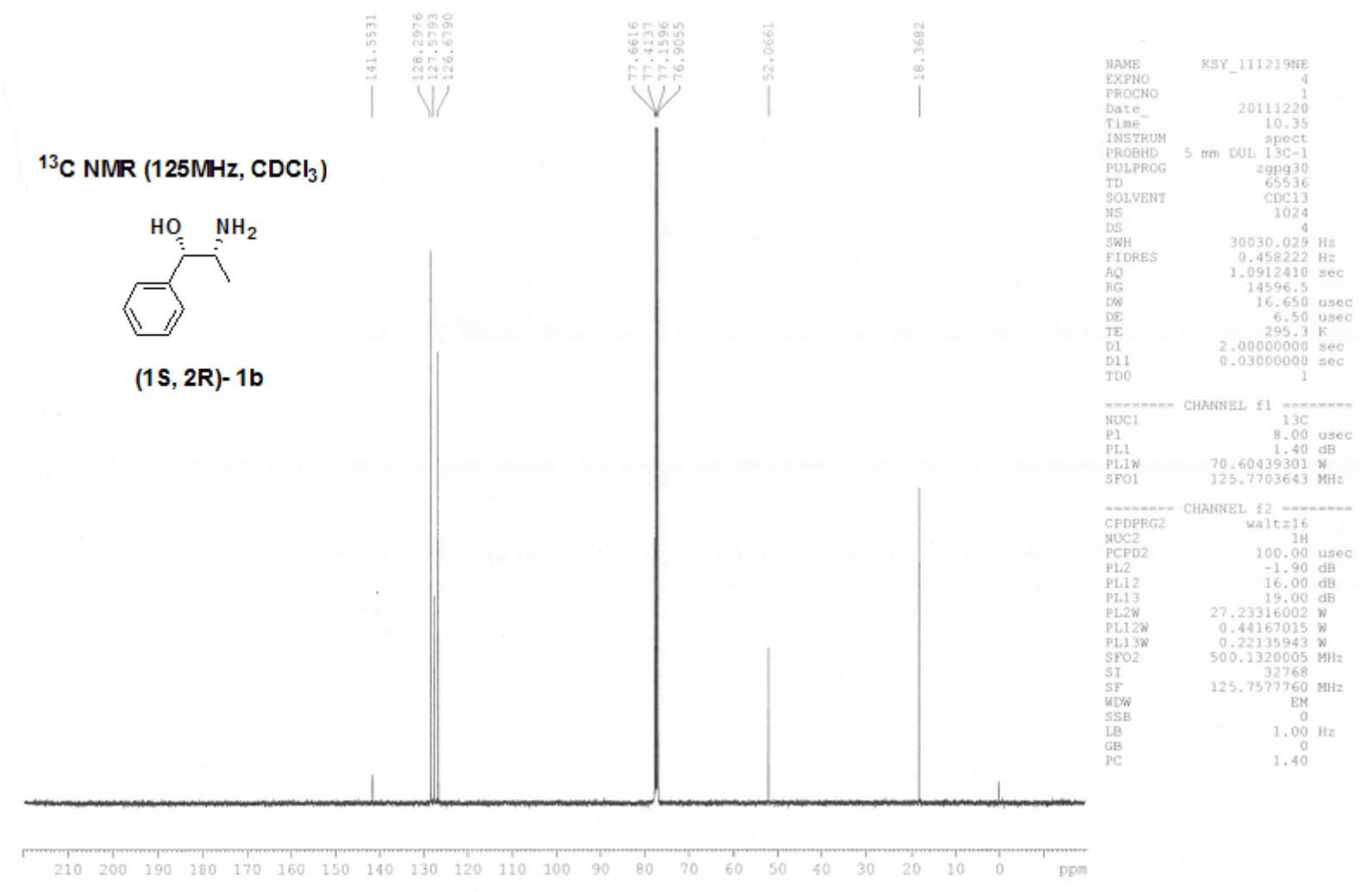

^{1}H NMR (300MHz, CD_3OD)
(4S, 5R)-12
F
O
O
S
O
NH
7.4550
7.4489
7.4374
7.4318
7.4262
7.4154
7.4087
7.1924
7.1855
7.1632
7.1406
7.1339
5.8377
5.8170
4.8524
4.2843
4.2622
4.2403
3.3207
3.3153
3.3100
3.3046
3.2992
0.9213
0.8986
2.082
2.082
1.000
1.029
3.159
ppm


13C NMR (125MHz, CD3OD)
(4S, 5R)-12
F
O
O
S
O
NH
166.1892
162.9241
131.9039
131.8600
130.0064
129.8951
116.6822
116.3904
88.9785
57.0214
50.0021
49.7183
49.4344
49.1505
48.8666
48.5827
48.2988
15.4302
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

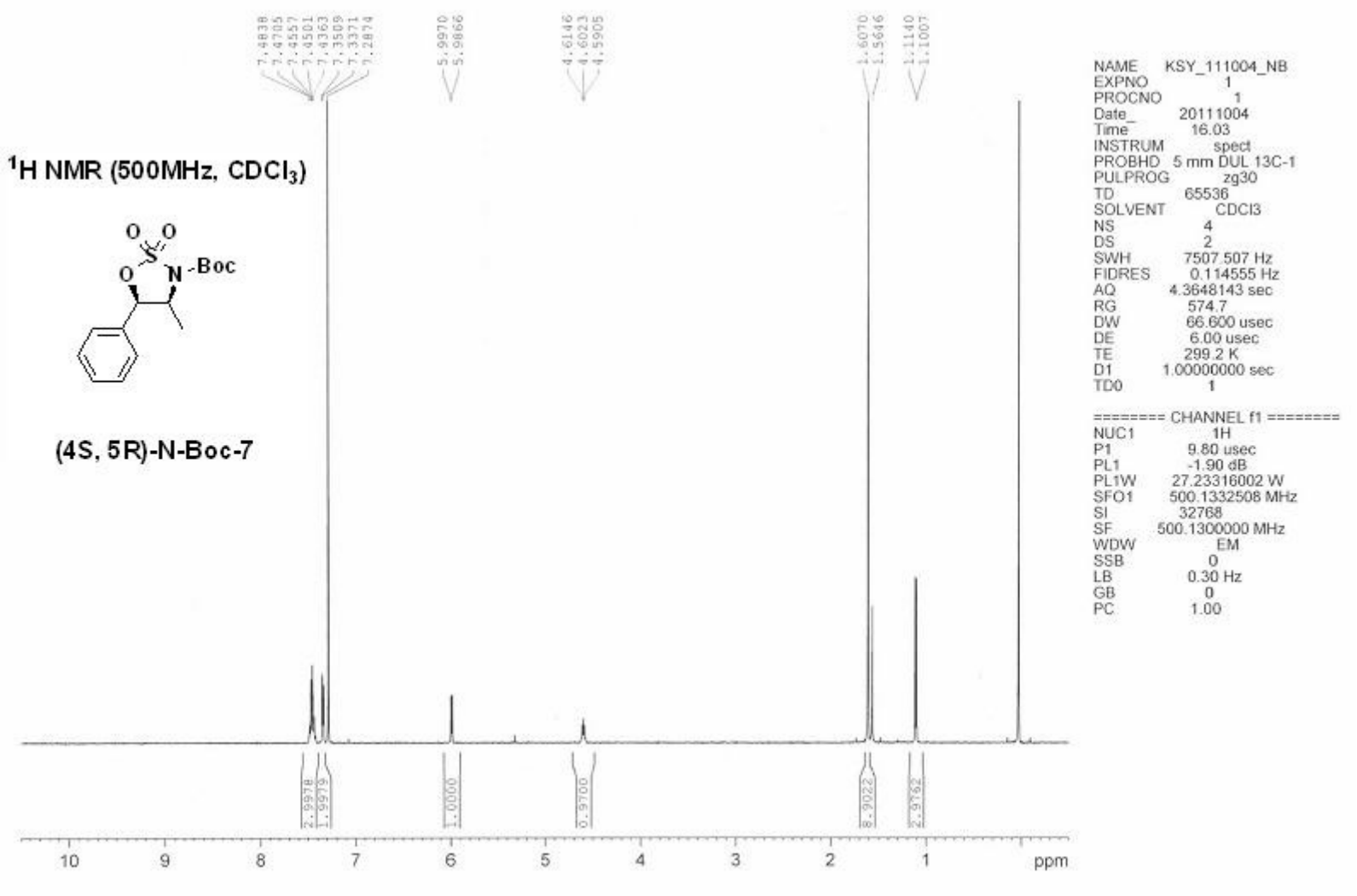

7.3961
7.3793
7.3668
7.3590
7.3233
7.3188
7.3118
7.3062
7.2997
7.2936
7.2874
4.5718
4.5623
3.2572
3.2542
3.2444
3.2315
3.2186
1.0021
0.9891
^{1}H NMR (500 MHz, $CDCl_3$)
HO
NH_2
(1*R*, 2*S*)- 1a
NAME KSY_111007
EXPNO 1
PROCNO 1
Date_ 20111007
Time 14.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 574.7
DW 66.600 usec
DE 6.00 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.0197
1.0000
1.0111
3.2427
10
9
8
7
6
5
4
3
2
1
ppm

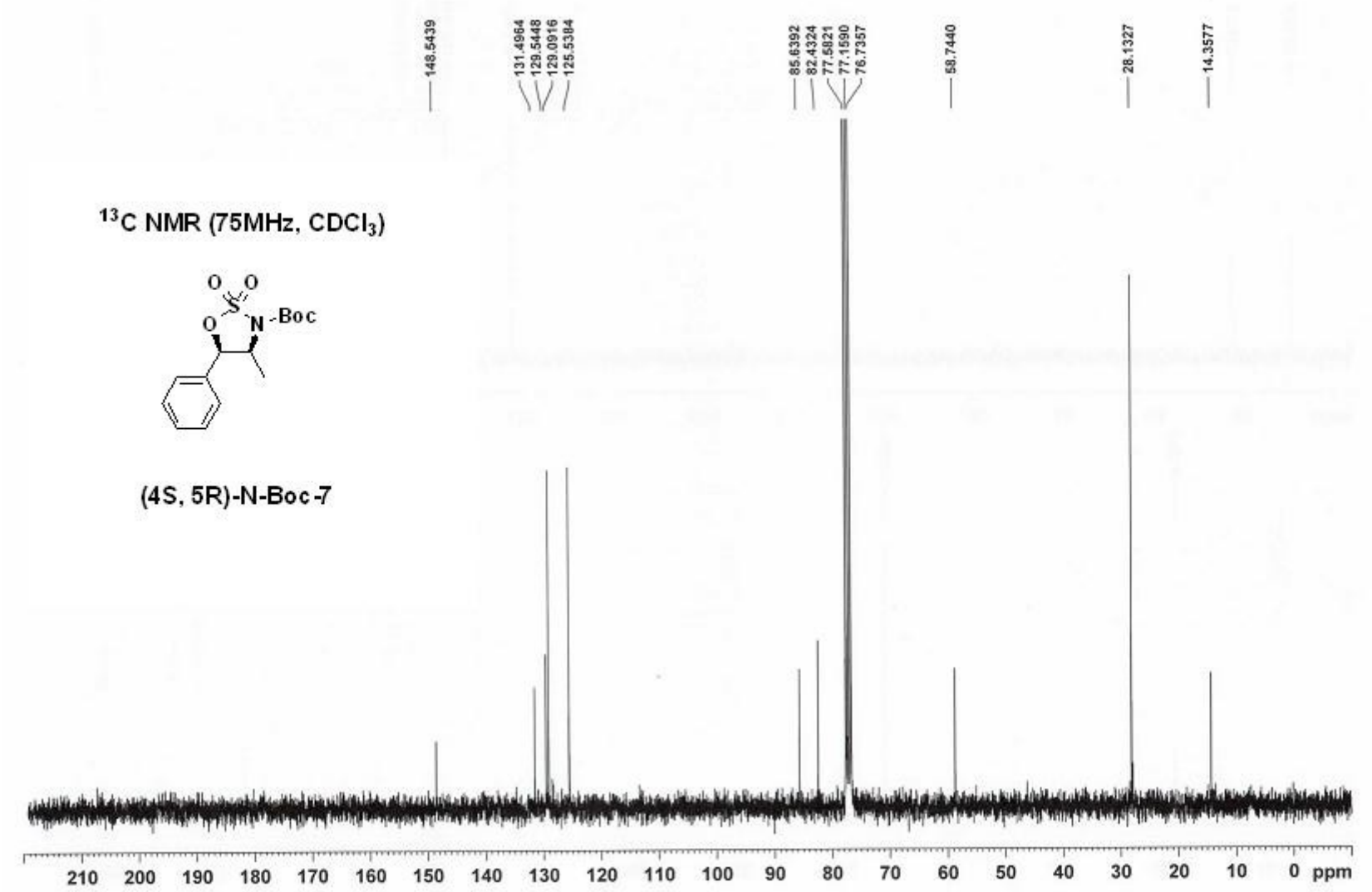

141.2886
128.1968
127.4897
126.5304
77.5344
77.2689
77.0148
76.7601
51.9060
18.1868
-0.0071
^{13}C NMR (125MHz, $CDCl_3$)
HO
NH_2
(1*R*, 2*S*)- 1a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
NAME KSY_111007_C
EXPNO 1
PROCNO 1
Date_ 20111007
Time 15.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306754 sec
RG 3649.1
DW 14.200 usec
DE 6.00 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 1.40 dB
PL1W 70.60439301 W
SFO1 125.7728799 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.90 dB
PL12 16.00 dB
PL13 19.00 dB
PL2W 27.23316002 W
PL12W 0.44167015 W
PL13W 0.22135943 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

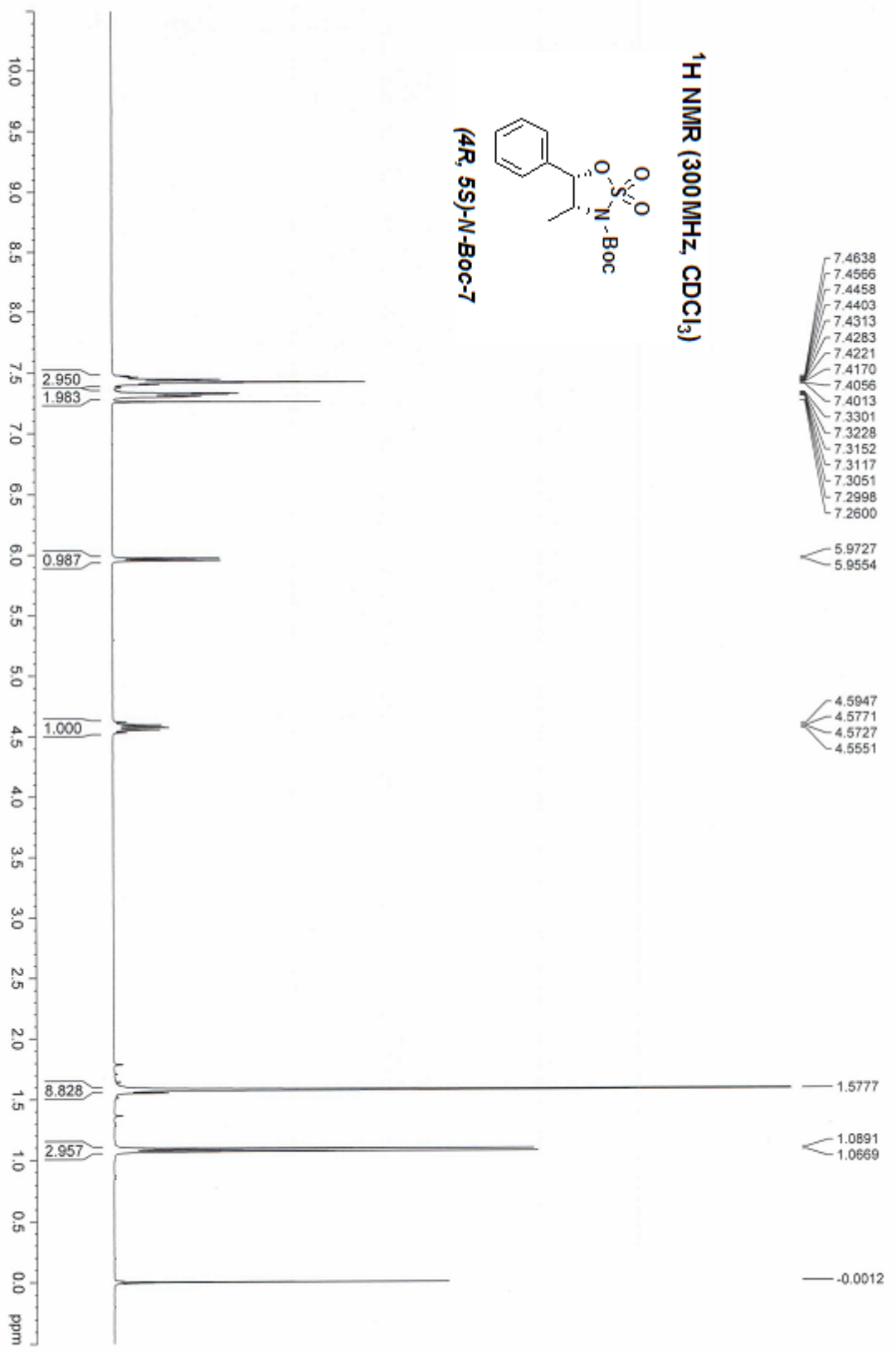

1H NMR (300MHz, CDCl3)
(1S, 2R)- 1b
HO
NH2
7.3495
7.3458
7.3386
7.3291
7.3166
7.3103
7.2987
7.2915
7.2868
7.2764
7.2701
7.2598
4.5232
4.5073
3.2301
3.2140
3.2084
3.1924
3.1867
3.1707
3.1653
3.1490
1.7807
0.9733
0.9516
-0.0001
4.630
1.000
1.015
2.675
3.046
ppm

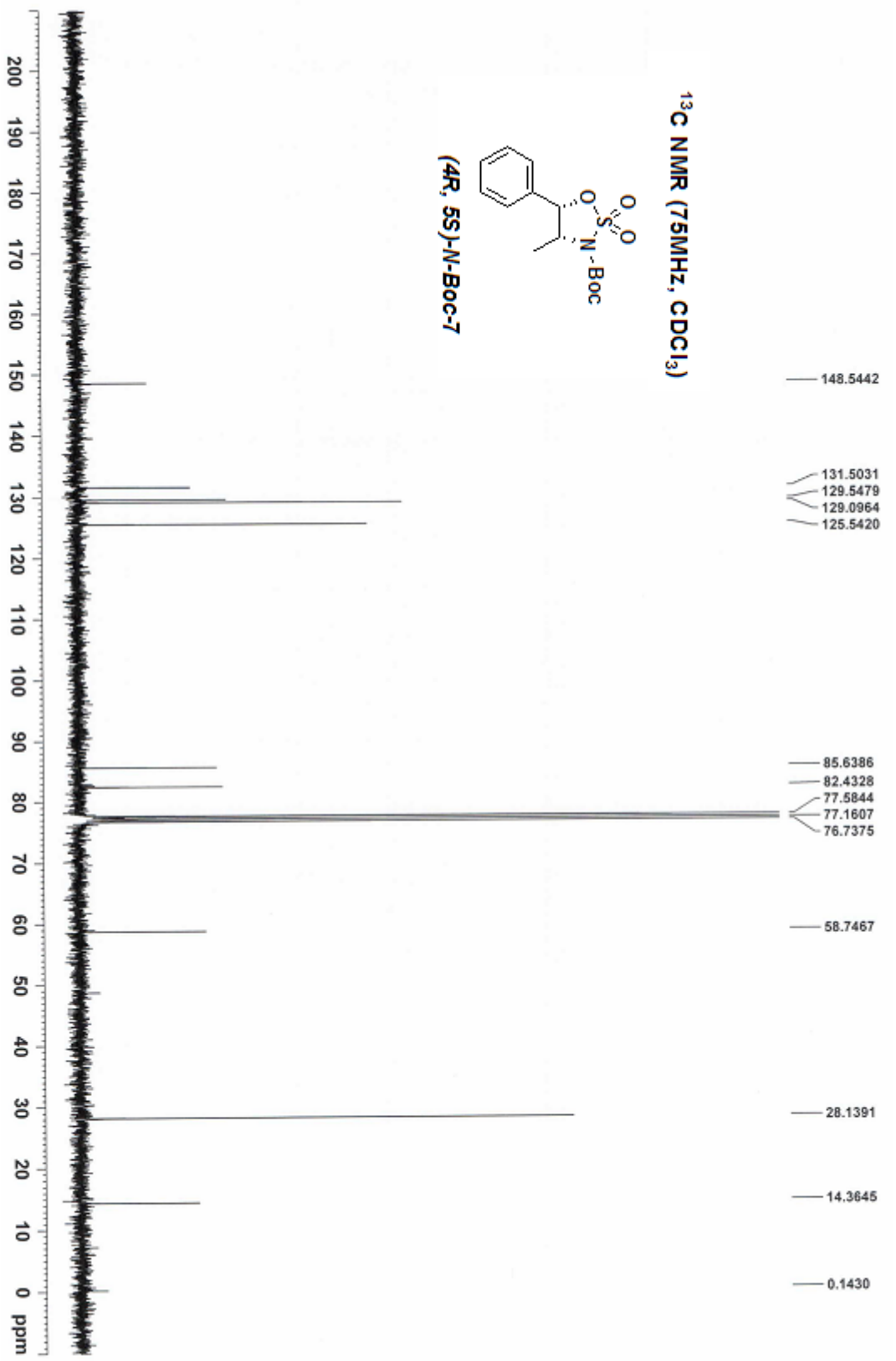


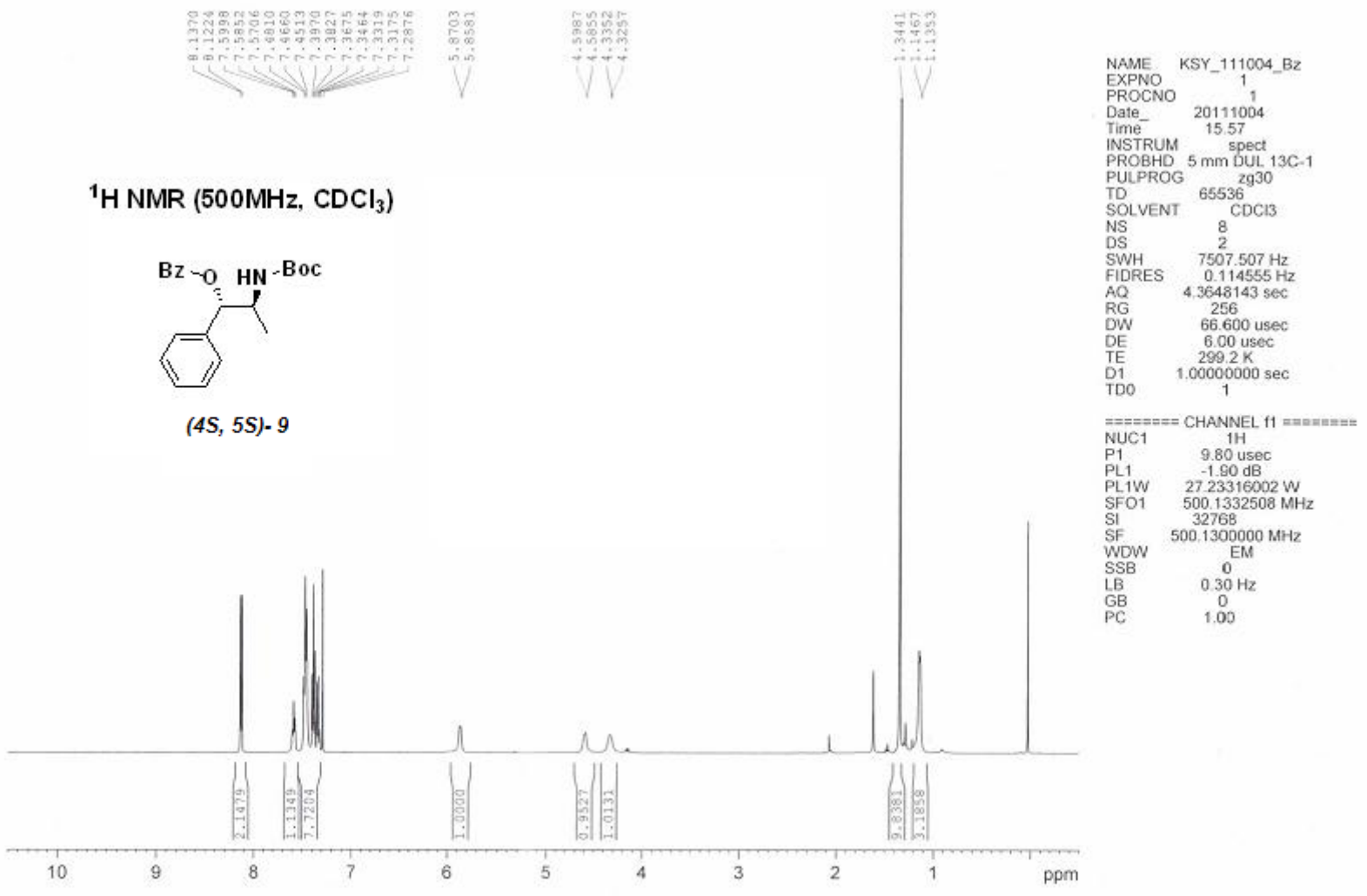
NAME	KSY_111219NE
EXPNO	4
PROCNO	1
Date_	20111220
Time	10.35
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	14596.5
DW	16.650 usec
DE	6.50 usec
TE	295.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577760 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


^{1}H NMR (500MHz, $CDCl_3$)
(4S, 5R)-N-Boc-7
7.4838
7.4705
7.4557
7.4501
7.4363
7.3509
7.3371
7.2874
5.9970
5.9866
4.6146
4.6023
4.5905
1.6070
1.5646
1.1140
1.1007
2.9978
1.9979
1.0000
0.9700
8.9022
2.9762
10
9
8
7
6
5
4
3
2
1
ppm
NAME KSY_111004_NB
EXPNO 1
PROCNO 1
Date_ 20111004
Time 16.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 574.7
DW 66.600 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


148.5439
131.4964
129.5448
129.0916
125.5384
85.6392
82.4324
77.5821
77.1590
76.7357
58.7440
28.1327
14.3577
^{13}C NMR (75MHz, CDCl$_3$)
O
O
S
O
N
-Boc
(4S, 5R)-N-Boc-7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


^{1}H NMR (300MHz, $CDCl_3$)
(4R, 5S)-N-Boc-7
Boc
7.4638
7.4566
7.4458
7.4403
7.4313
7.4283
7.4221
7.4170
7.4056
7.4013
7.3301
7.3228
7.3152
7.3117
7.3051
7.2998
7.2600
5.9727
5.9554
4.5947
4.5771
4.5727
4.5551
1.5777
1.0891
1.0669
-0.0012
2.950
1.983
0.987
1.000
8.828
2.957
ppm


^{13}C NMR (75MHz, $CDCl_3$)
(4R, 5S)-N-Boc-7
Boc
148.5442
131.5031
129.5479
129.0964
125.5420
85.6386
82.4328
77.5844
77.1607
76.7375
58.7467
28.1391
14.3645
0.1430
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



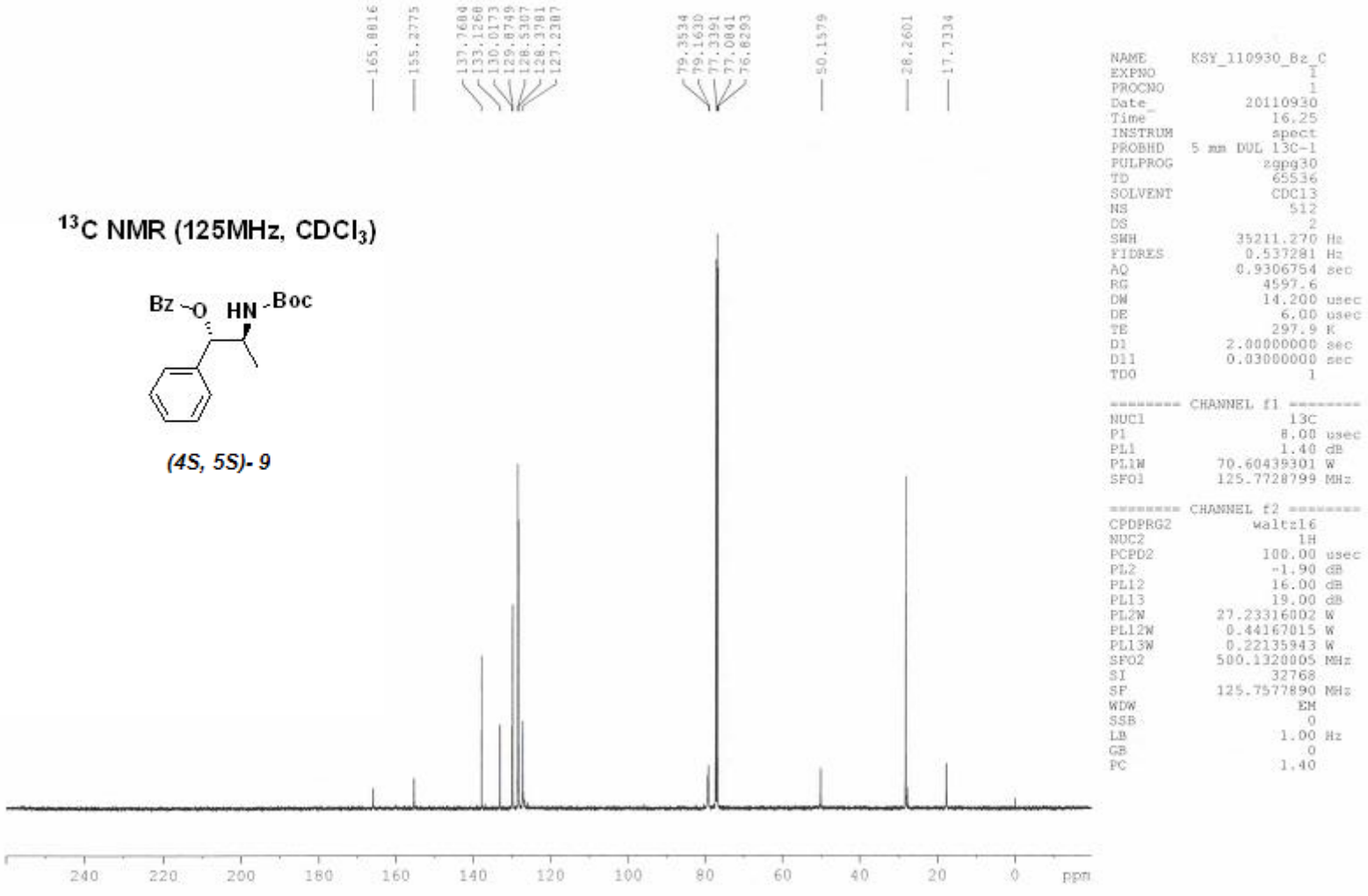
^{1}H NMR (500MHz, $CDCl_3$)

(4S, 5S)- 9

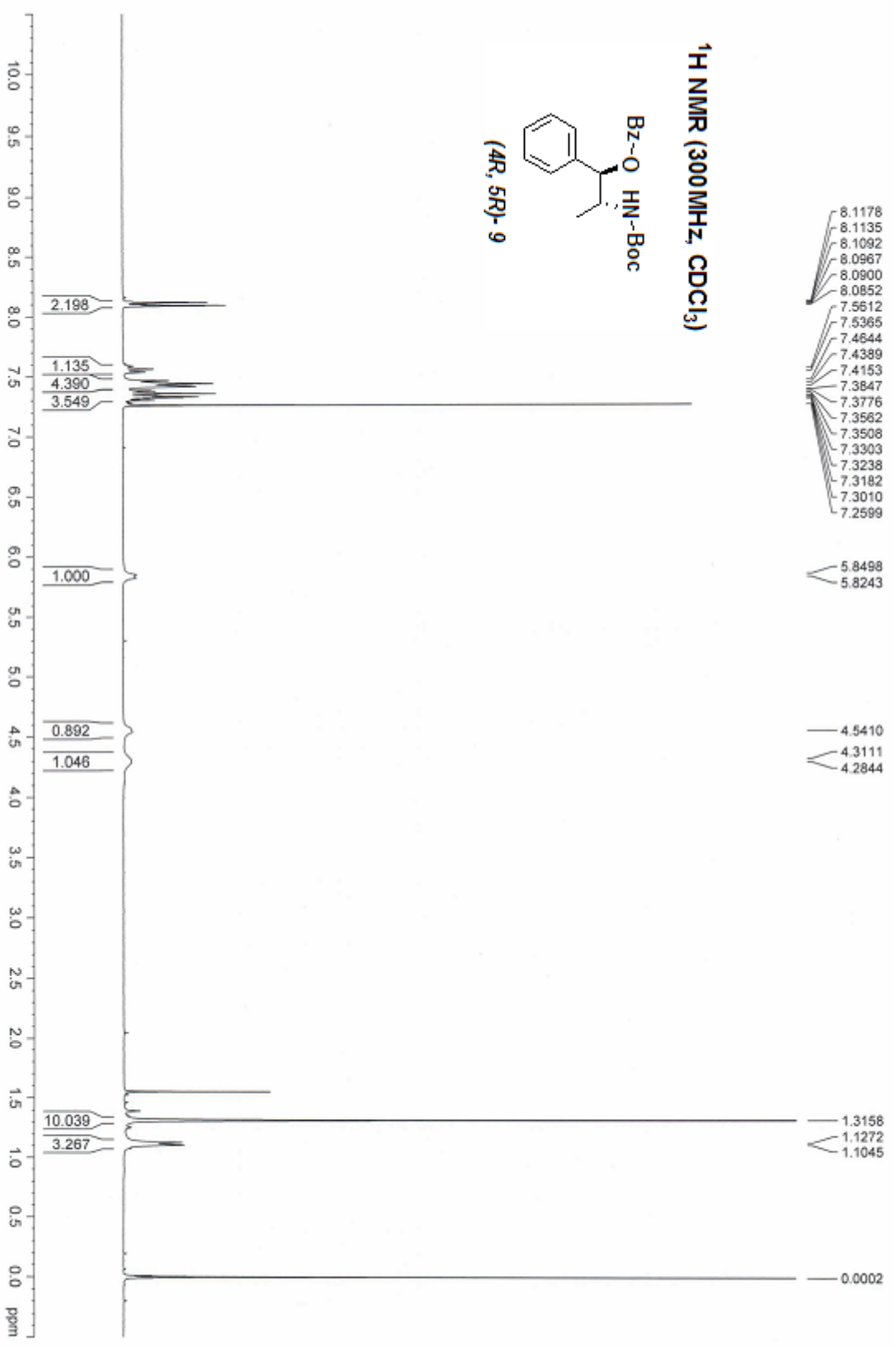
NAME	KSY_111004_Bz
EXPNO	1
PROCNO	1
Date_	20111004
Time	15.57
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	7507.507 Hz
FIDRES	0.114555 Hz
AQ	4.3648143 sec
RG	256
DW	66.600 usec
DE	6.00 usec
TE	299.2 K
D1	1.00000000 sec
TD0	1

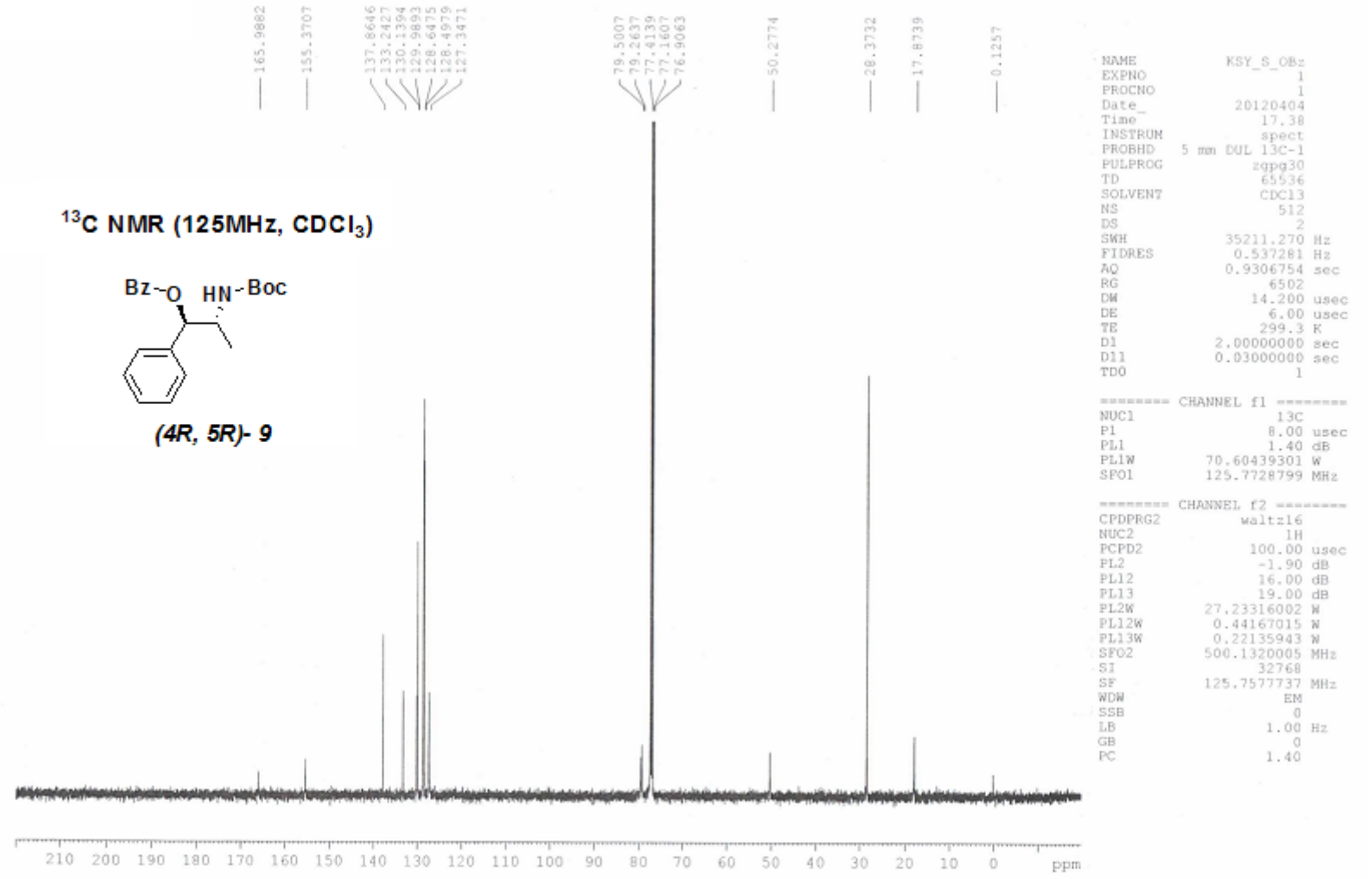
======== CHANNEL f1 ========

NUC1	1H
P1	9.80 usec
PL1	-1.90 dB
PL1W	27.23316002 W
SFO1	500.1332508 MHz
SI	32768
SF	500.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

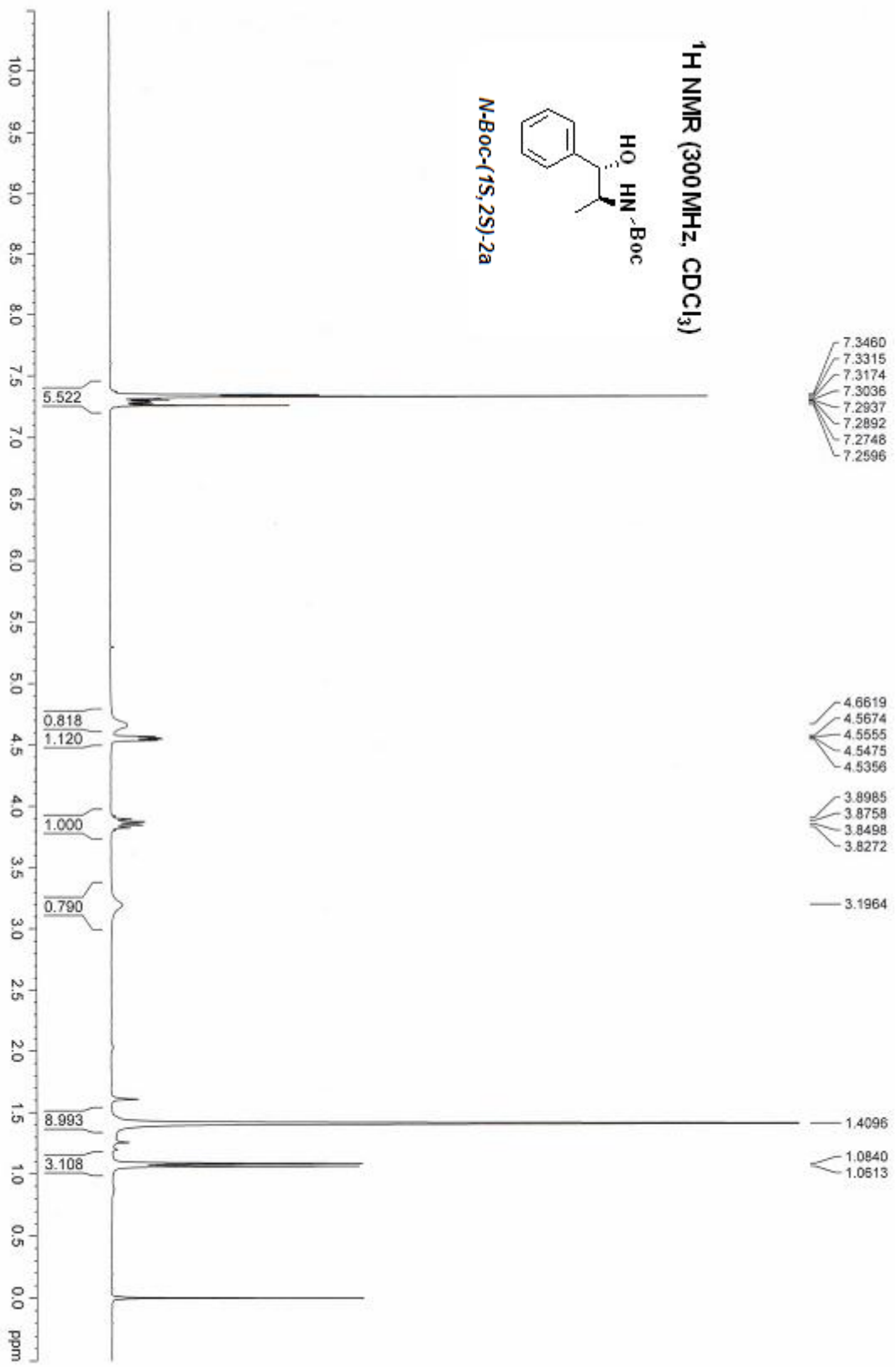


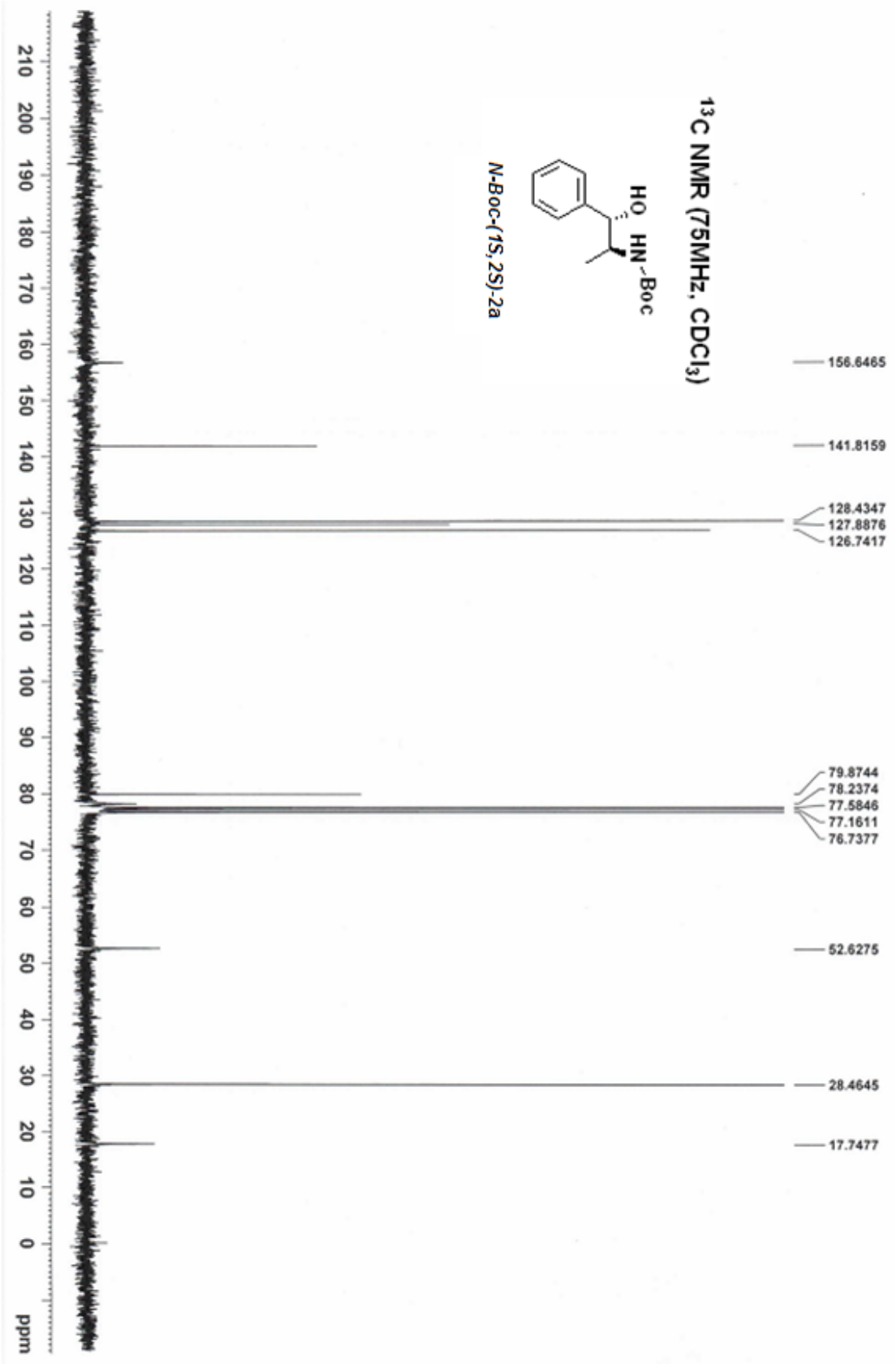
NAME	KSY_110930_Bz_C
EXPNO	1
PROCNO	1
Date_	20110930
Time	16.25
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	2
SWH	35211.270 Hz
FIDRES	0.537281 Hz
AQ	0.9306754 sec
RG	4597.6
DW	14.200 usec
DE	6.00 usec
TE	297.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7728799 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

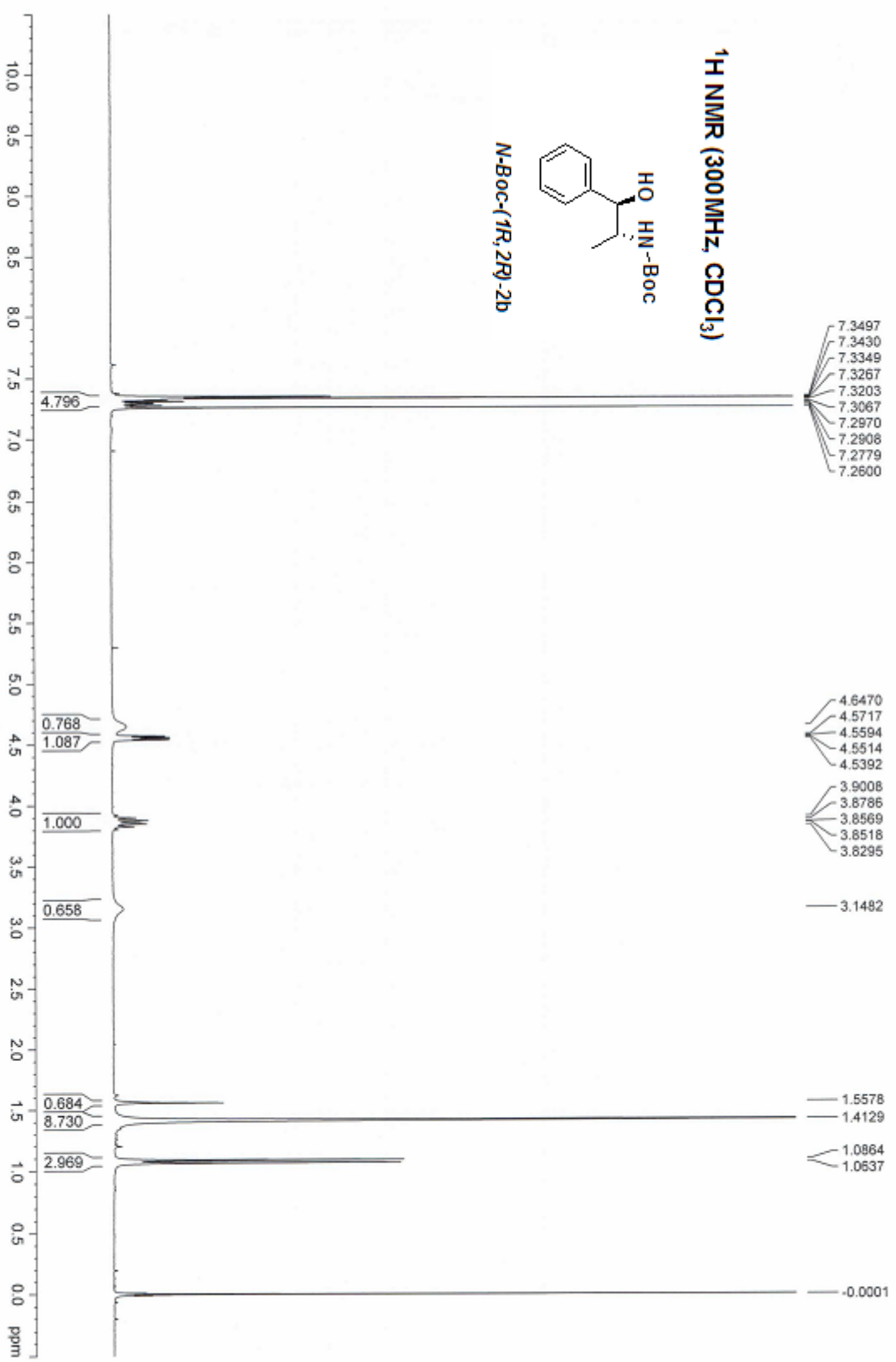

^{1}H NMR (300MHz, $CDCl_3$)
Bz-O HN-Boc
(4R, 5R)- 9
8.1178
8.1135
8.1092
8.0967
8.0900
8.0852
7.5612
7.5365
7.4644
7.4389
7.4153
7.3847
7.3776
7.3562
7.3508
7.3303
7.3238
7.3182
7.3010
7.2599
5.8498
5.8243
4.5410
4.3111
4.2844
1.3158
1.1272
1.1045
0.0002
2.198
1.135
4.390
3.549
1.000
0.892
1.046
10.039
3.267
ppm

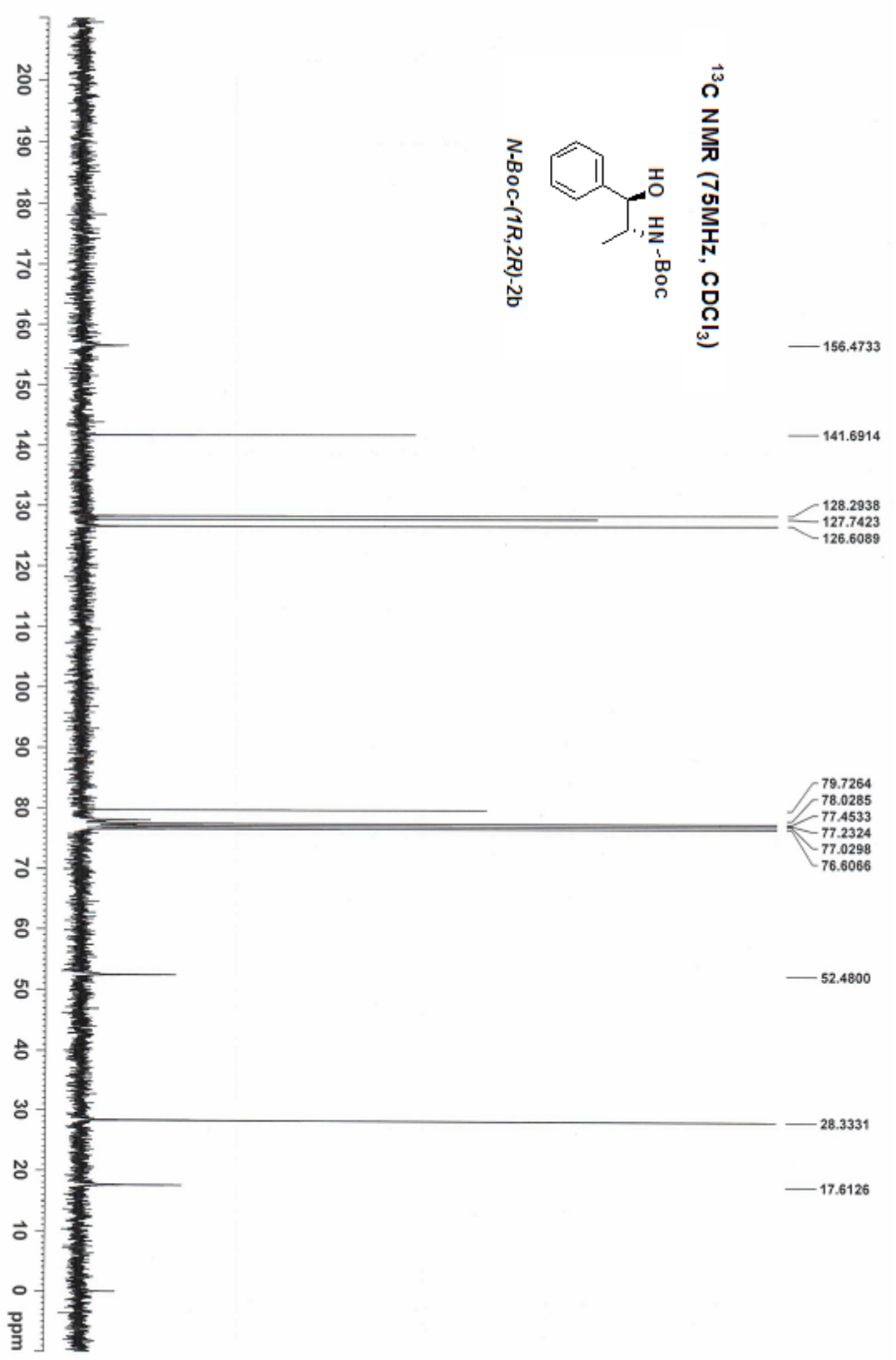


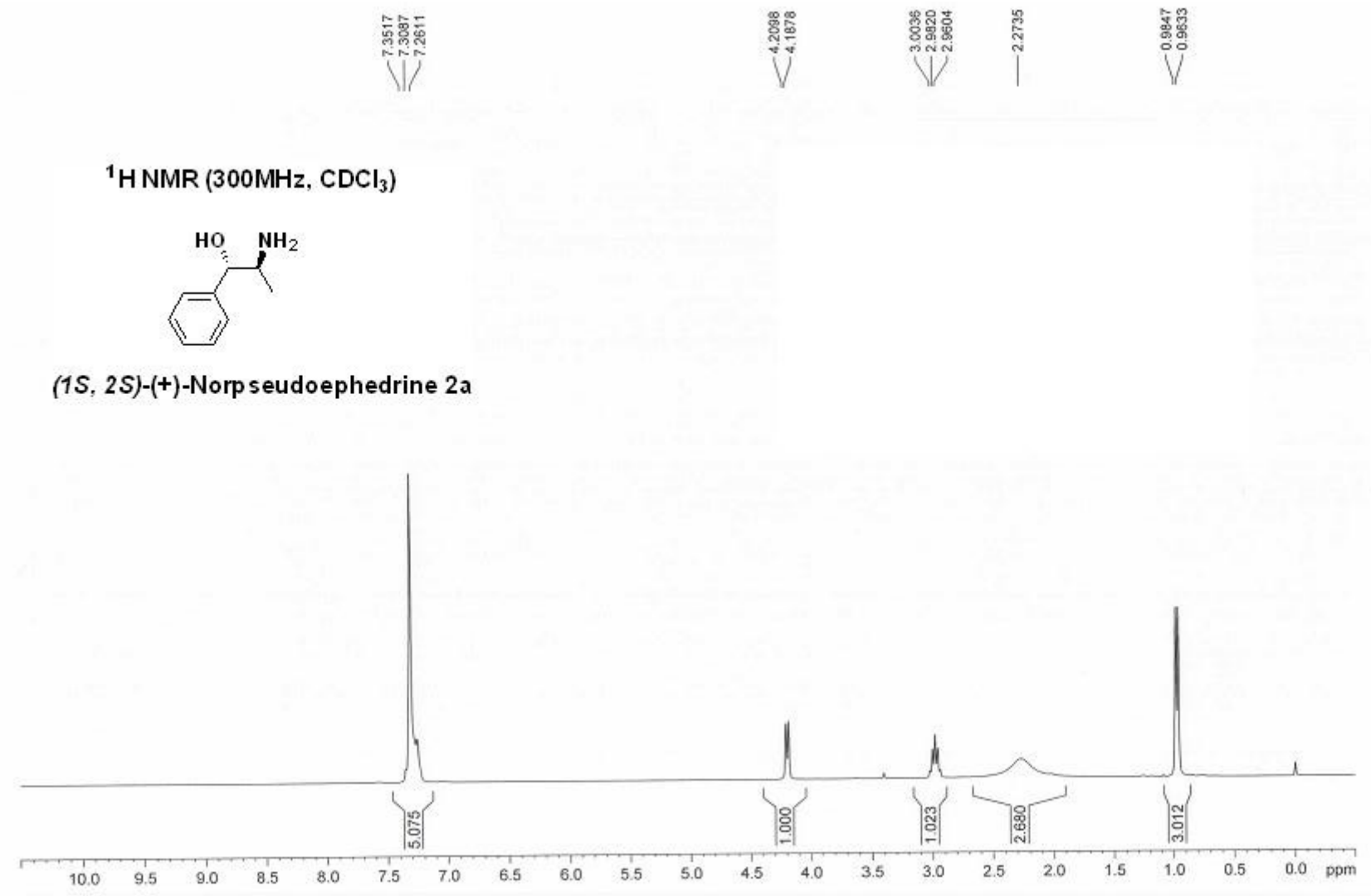
NAME	KSY_S_OBz
EXPNO	1
PROCNO	1
Date_	20120404
Time	17.38
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	2
SWH	35211.270 Hz
FIDRES	0.537281 Hz
AQ	0.9306754 sec
RG	6502
DW	14.200 usec
DE	6.00 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7728799 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577737 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


^{1}H NMR (300MHz, CDCl$_3$)
N-Boc-(1S,2S)-2a
HO
HN
Boc
7.3460
7.3315
7.3174
7.3036
7.2937
7.2892
7.2748
7.2596
4.6619
4.5674
4.5555
4.5475
4.5356
3.8985
3.8758
3.8498
3.8272
3.1964
1.4096
1.0840
1.0613
5.522
0.818
1.120
1.000
0.790
8.993
3.108
ppm

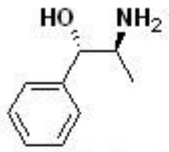

13C NMR (75MHz, CDCl3)
HO
HN
Boc
N-Boc-(1S,2S)-2a
156.6465
141.8159
128.4347
127.8876
126.7417
79.8744
78.2374
77.5846
77.1611
76.7377
52.6275
28.4645
17.7477
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


^{1}H NMR (300MHz, CDCl$_3$)
N-Boc-(1R, 2R)-2b
HO
HN-Boc
7.3497
7.3430
7.3349
7.3267
7.3203
7.3067
7.2970
7.2908
7.2779
7.2600
4.6470
4.5717
4.5594
4.5514
4.5392
3.9008
3.8786
3.8569
3.8518
3.8295
3.1482
1.5578
1.4129
1.0864
1.0637
-0.0001
4.796
0.768
1.087
1.000
0.658
0.684
8.730
2.969
ppm


^{13}C NMR (75MHz, CDCl$_3$)
HO HN -Boc
N-Boc-(1R,2R)-2b
156.4733
141.6914
128.2938
127.7423
126.6089
79.7264
78.0285
77.4533
77.2324
77.0298
76.6066
52.4800
28.3331
17.6126
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

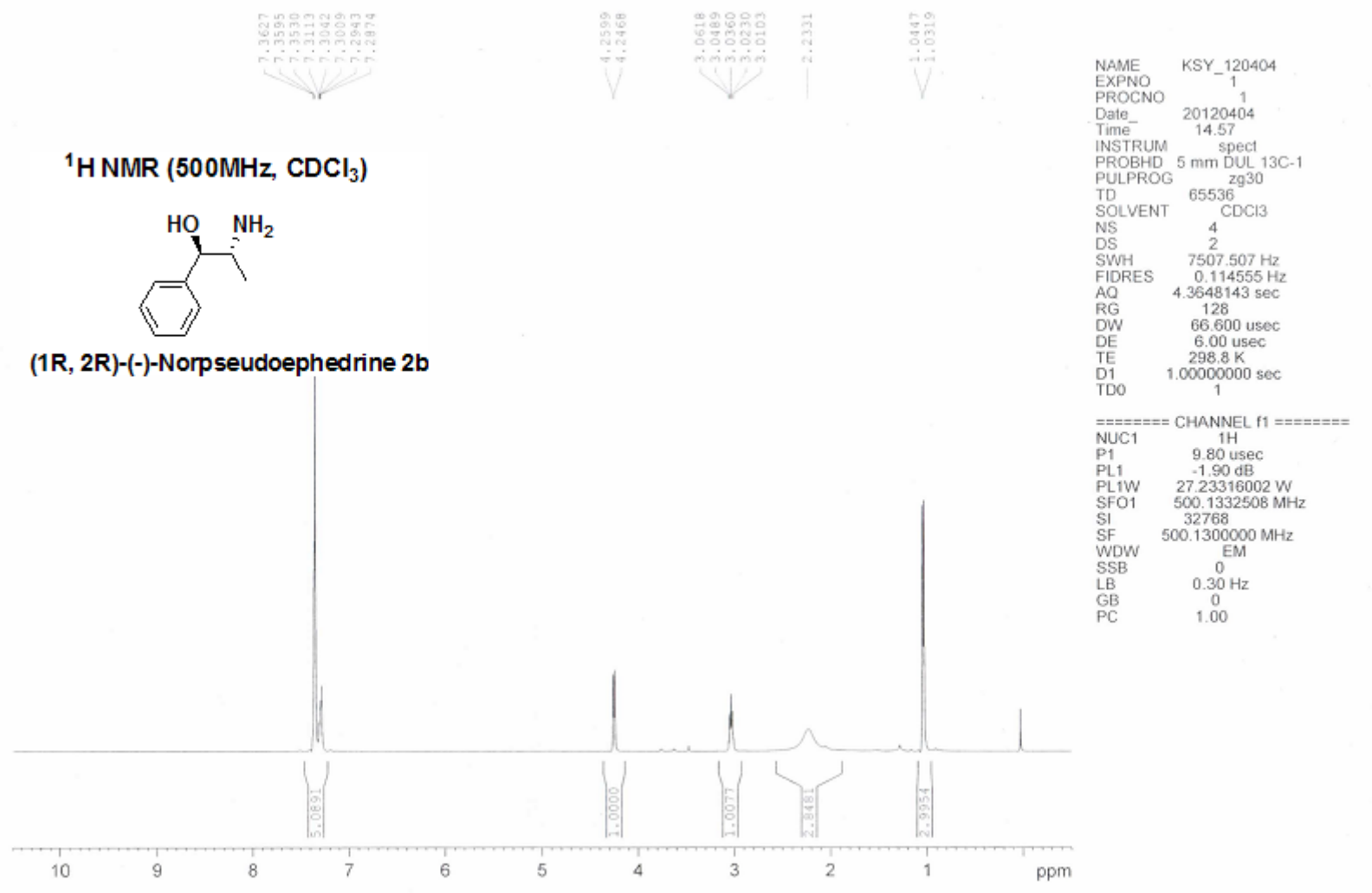

7.3517
7.3087
7.2611
4.2098
4.1878
3.0036
2.9820
2.9604
2.2735
0.9847
0.9633
^{1}H NMR (300MHz, $CDCl_3$)
HO
NH_2
(1S, 2S)-(+)-Norpseudoephedrine 2a
5.075
1.000
1.023
2.680
3.012
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

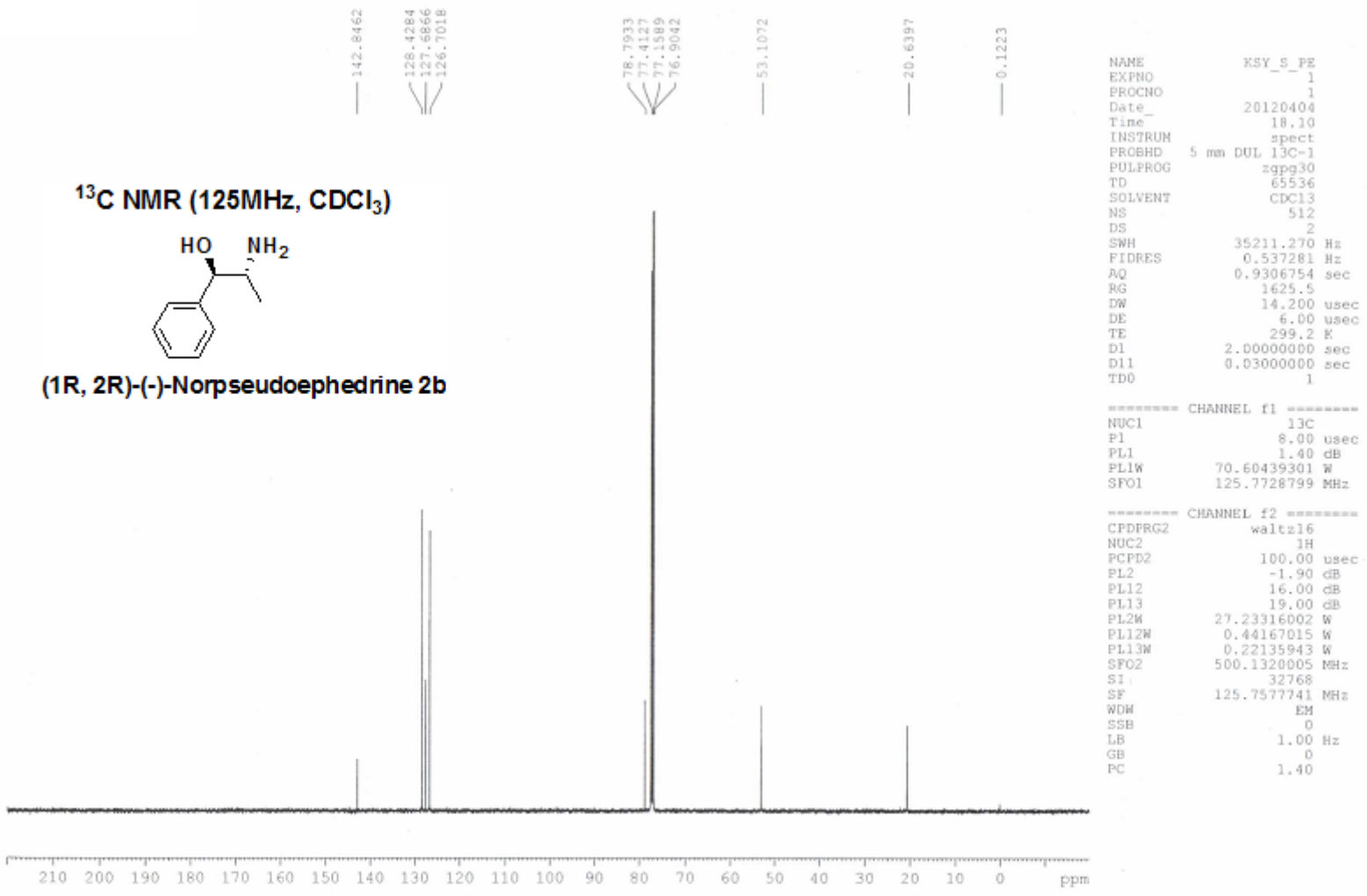
^{13}C NMR (125MHz, $CDCl_3$)



(1S, 2S)-(+)-Norpseudoephedrine 2a

NAME	KSY_111216_PE
EXPNO	1
PROCNO	1
Date_	20111216
Time	17.34
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	7298.2
DW	16.650 usec
DE	6.50 usec
TE	296.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577795 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


7.3627
7.3595
7.3530
7.3113
7.3042
7.3009
7.2943
7.2874
4.2599
4.2468
3.0618
3.0489
3.0360
3.0230
3.0103
2.2331
1.0447
1.0319
^{1}H NMR (500MHz, CDCl$_3$)
HO
NH$_2$
(1R, 2R)-(-)-Norpseudoephedrine 2b
NAME KSY_120404
EXPNO 1
PROCNO 1
Date_ 20120404
Time 14.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 128
DW 66.600 usec
DE 6.00 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -1.90 dB
PL1W 27.23316002 W
SFO1 500.1332508 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.0891
1.0000
1.0077
2.8481
2.9954
10
9
8
7
6
5
4
3
2
1
ppm



NAME	KSY_S_PE
EXPNO	1
PROCNO	1
Date_	20120404
Time	18.10
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	512
DS	2
SWH	35211.270 Hz
FIDRES	0.537281 Hz
AQ	0.9306754 sec
RG	1625.5
DW	14.200 usec
DE	6.00 usec
TE	299.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	1.40 dB
PL1W	70.60439301 W
SFO1	125.7728799 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.90 dB
PL12	16.00 dB
PL13	19.00 dB
PL2W	27.23316002 W
PL12W	0.44167015 W
PL13W	0.22135943 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577741 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40