Eur. J. Org. Chem. **2007** · © WILEY-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2007 · ISSN 1434–193X

SUPPORTING INFORMATION

Title: Efficient One-Pot Synthesis of Enantiomerically Pure 2-(Hydroxymethyl)morpholines
Author(s): Matthias Breuning,* Malte Winnacker, Melanie Steiner
Ref. No.: O200601006

2D NMR Correlations

Until otherwise noted, all NMR spectra were recorded in $CDCl_3$ at 400 MHz (^1H) and 100 MHz (^{13}C). The ^1H and ^{13}C signals were assigned by HMQC correlations.

1-[Benzyl(2-hydroxyethyl)amino]-3-chloropropan-2-ol (*rac*-9)

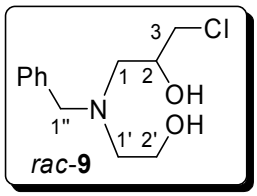


Table S1. ^{13}C and ^1H NMR data of *rac*-**9** and significant COSY correlations.

No.	^{13}C: δ [ppm]	^1H: δ [ppm] (int., m, *J*)	COSY
1		α: 2.67 (1H, dd, 13.4, 8.3 Hz) β: 2.70 (1H, ddd, 13.4, 5.3, 4.4 Hz)	1β, 2 1α, 2
2	68.9	3.88 (1H, m)	1α, 1β, 3α, 3β
3	47.5	α: 3.50 (1H, dd, 11.1, 5.6 Hz) β: 3.54 (1H, dd, 11.1, 4.8 Hz	3β, 2 3α, 2
1'	56.7	α: 2.74 (1H, dd, 13.4, 4.2 Hz) β: 2.79 (1H, ddd, 13.4, 6.8, 4.8 Hz)	2' 2'
2'	57.7	3.64 (2H, m)	1'α, 1'β
1"	57.7	α: 3.67 (1H, d, 13.9 Hz) β: 3.79 (1H, d, 13.6 Hz)	1"β 1"α
Ph	127.6, 128.7 129.1, 138.5	7.20–7.40 ppm (5H, m)	
1-OH		2.27 (1H, br. s), 3.25 (1H, br. s)	

2-[Benzyl(oxiranylmethyl)amino]ethanol (*rac*-10)

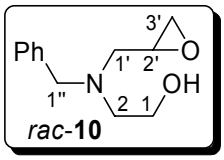
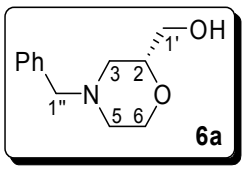


Table S2. ^{13}C and ^1H NMR data of *rac*-**10** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^1H: δ [ppm] (int., m, *J*)	COSY	HMBC
1	59.0	3.62 (2H, m)	2α, 2β	2
2	56.1	α: 2.72 (1H, ddd, 13.1, 5.6, 4.4 Hz) β: 2.85 (1H, ddd, 13.1, 6.6, 4.8 Hz)	1, 2β 1, 2α	1, 1', 1" 1, 1', 1"
1'	56.0	α: 2.51 (1H, dd, 14.0, 6.3 Hz) β: 2.89 (1H, dd, 14.1, 3.3 Hz)	1'β, 2' 1'α, 2'	1", 2, 2', 3' 1", 2, 2', 3'
2'	50.9	3.07 (1H, m)	1'α, 1'β, 3'α, 3'β	1'
3'	45.1	α: 2.48 (1H, dd, 4.8, 2.7 Hz) β: 2.74 (1H, dd, 4.8, 4.1 Hz)	2', 3'β 2', 3'α	1', 2' 1', 2'
1"	59.4	α: 3.68 (1H, d, 13.6 Hz) β: 3.83 (1H, d, 13.5 Hz)	1"β 1"α	1', 2, Ph 1', 2, Ph
Ph	127.5, 128.6 129.1, 138.7	7.20-7.40 (5H, m)		1"
1-OH		2.55-2.80 (1H, br. s)		

(*R*)-*N*-Benzyl-2-(hydroxymethyl)morpholine (6a)



The following NMR spectra of **6a** were recorded in C_6D_6 as the solvent. The ^1H and ^{13}C NMR data of **6a** in $CDCl_3$ as the solvent are given in the article.

Table S3. ^{13}C and ^1H NMR data of **6a** and significant COSY, HMBC, and NOESY correlations.[a]

No.	^{13}C: δ [ppm]	^1H: δ [ppm] (int., m, *J*)	COSY	HMBC	NOESY
2	76.5	3.51 (1H, m)	1', 3α, 3β	1', 3	1', 3α, 3β
3	55.2	α: 1.80 (1H, dd, 10.9, 10.2 Hz)	2, 3β	1', 1", 2, 5	1', 1", 2, 3β
		β: 2.45 (1H, dt, 11.1, 1.9 Hz)	2, 3α	1", 2, 5	1', 1", 2, 3α
5	53.4	α: 1.89 (1H, td, 11.2, 3.4 Hz)	5β, 6α, 6β	1", 3, 6	1", 5β, 6α, 6β
		β: 2.29 (1H, dq, 11.2, 1.9 Hz)	5α, 6α, 6β	1", 3	1", 5α, 6α, 6β
6	66.7	α: 3.49 (1H, td, 11.1, 2.4 Hz)	5α, 5β	2, 5	5α, 5β, 6β
		β: 3.59 (1H, ddd, 11.1, 3.4, 1.8 Hz)	5α, 5β	2	1', 5α, 5β, 6α
1'	64.3	3.36 (2H, m)	2	2, 3	2, 3α, 3β, 6β
1"	63.5	3.22 (2H, s)		3, 5, Ph	3α, 3β, 5α, 5β, Ph
Ph	127.5, 128.6 129.3, 138.6	7.07-7.32 (5H, m)		1"	1"
1'-OH		1.60 (1H, br. s)			

[a] Solvent: C_6D_6.

(*S*)-*N*-Benzyl-1,4-oxazepan-6-ol (11)

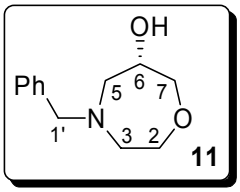


Table S4. ^{13}C and ^1H NMR data of **11** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^1H: δ [ppm] (int., m, *J*)	COSY[a]	HMBC[a]
2	70.2	3.73 (2H, m)	3α, (3β)	(7)
3	57.8	α: 2.51 (1H, ddd, 12.4, 7.3, 5.1 Hz)	(2), (3β)	1', 2, 5
		β: 2.83 (1H, m)	(2), (3α)	(1'), (2), (5)
5	57.2	α: 2.83 (1H, m)	(5β), (6)	(1'), (3), (6), (7)
		β: 2.97 (1H, ddd, 12.6, 5.8, 1.5 Hz)	(5α), 6	1', 3, 6, 7
6	69.1	3.83 (1H, m)	(5α), 5β, 7	
7	76.4	3.79 (2H, m)	6	2, 5, 6
1'	63.5	3.73 (2H, m)		(3), (5), Ph
Ph	127.6, 128.6 129.1, 138.6	7.20-7.40 (5H, m)		1'
6-OH		3.96 (1H, m)		

[a] Values in parentheses refer to cross peaks which are not unequivocally assignable due to signal overlap.

(2*R*,5*S*)-*N*-Benzyl-2-(hydroxymethyl)-5-isopropylmorpholine (6b)

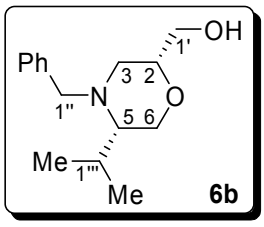


Table S5. ^{13}C and ^1H NMR data of **6b** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^1H: δ [ppm] (int., m, *J*)	COSY	HMBC
2	71.6	3.76 (1H, m)	1'α, 1'β, 3α, 3β	1', 6
3	48.7	α: 2.37 (1H, dd, 13.1, 3.2 Hz)	2, 3β	1', 1", 2, 5
		β: 2.82 (1H, dd, 13.1, 7.8 Hz)	2, 3α	1', 1", 5
5	63.9	2.15 (1H, m)	1'", 6α, 6β	1", 1'", 1'"α-Me 1'"β-Me, 3, 6
6	62.3	α: 3.82 (1H, dd, 11.8, 3.3 Hz)	5, 6β	1'", 2, 5
		β: 3.90 (1H, dd, 11.8, 4.4 Hz)	5, 6α	1'", 2, 5
1'	64.7	α: 3.57 (1H, dd, 11.5, 3.3 Hz)	1'β, 2	2, 3
		β: 3.67 (1H, dd, 11.5, 6.6 Hz)	1'α, 2	2, 3
1"	58.2	α: 3.59 (1H, d, 13.5 Hz)	1"β	3, 5, Ph
		β: 4.02 (1H, d, 13.5 Hz)	1"α	3, 5, Ph
1'"	26.1	2.30 (1H, oct, 6.9 Hz)	1'"α-Me, 1'"β-Me, 5	1'"α-Me, 1'"β-Me 5, 6
1'"α-Me	19.0	0.95 (3H, d, 6.8 Hz)	1'"	1'", 1'"β-Me, 5
1'"β-Me'	20.4	0.99 (3H, d, 6.8 Hz)	1'"	1'", 1'"α-Me, 5
Ar	127.2, 128.5 128.8, 139.4	7.20-7.40 (5H, m)		1"
1'-OH	---	2.45 (1H, br. s)		

(2*R*,5*S*)-*N*-Benzyl-5-*tert*-butyl-2-(hydroxymethyl)morpholine (6c)

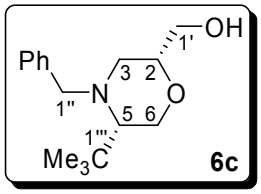


Table S6. ^{13}C and ^{1}H NMR data of **6c** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY	HMBC[a]
2	69.7	3.94 (1H, m)	1', 3α, 3β	1', 3, 6
3	45.2	α: 2.46 (1H, dd, 14.6, 3.9 Hz) β: 2.87 (1H, dd, 14.6, 11.5 Hz)	2, 3β 2, 3α	1", 5 1', 1", 2
5	66.7	2.34 (1H, t, 5.6 Hz)	6α, 6β	1", 1'", 1'"-Me, 3, 6
6	60.6	α: 3.83 (1H, dd, 12.2, 5.6 Hz) β: 3.88 (1H, dd, 12.1, 5.8 Hz)	5 5	(1'"), (2), (5) (1'"), (2), (5)
1'	64.1	3.45 (2H, m)	2	2, 3
1"	61.2	α: 3.85 (1H, d, 13.7 Hz) β: 4.05 (1H, d, 13.7 Hz)	1"β 1"α	3, (5), Ph 3, 5, Ph
1'"	36.1			
1'"-Me	28.1	0.98 (9H, s)		1'", 1'"-Me, 5
Ph	127.2, 128.4 128.8, 140.2	7.25 (1H, m), 7.31 (2H, m), 7.36 (2H, m)		1"
1'-OH	---	1.89 (1H, dd, 7.1, 5.0 Hz)	1'	

[a] Values in parentheses refer to cross peaks which are not unequivocally assignable due to signal overlap.

(2*R*,5*R*)-*N*-Benzyl-2-(hydroxymethyl)-5-phenylmorpholine (6d)

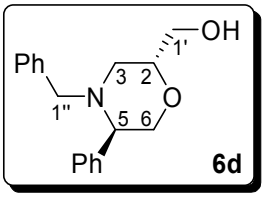


Table S7. ^{13}C and ^{1}H NMR data of **6d** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY	HMBC
2	76.8	3.75 (1H, m)	1'α, 1'β, 3α, 3β	6
3	53.1	α: 2.06 (1H, dd, 11.5, 10.7 Hz) β: 2.78 (1H, dd, 11.5, 2.3 Hz)	2, 3β 2, 3α	1', 1", 2, 5 1", 5
5	67.3	3.38 (1H, dd, 10.4, 3.4 Hz)	6α, 6β	1", 3, 6, Ph
6	73.5	α: 3.56 (1H, dd, 11.4, 10.5 Hz) β: 3.86 (1H, dd, 11.4, 3.5 Hz)	5, 6β 5, 6α	5, Ph 2, 5
1'	64.3	α: 3.54 (1H, ddd, 11.5, 6.3, 5.3 Hz) β: 3.62 (1H, ddd, 11.6, 7.1, 3.4 Hz)	1'α, 2 1'β, 2	2, 3 2
1"	59.4	α: 2.88 (1H, d, 13.3 Hz) β: 3.84 (1H, d, 13.4 Hz)	1"β 1"α	3, 5, Ph 3, 5, Ph
Ph	127.1, 128.1, 128.4 128.87, 128.94 138.5, 139.5	7.20-7.32 (6H, m), 7.37 (2H, t, 8.0 Hz) 7.49 (2H, d, 7.2 Hz)		1", 5
1'-OH	---	1.83 (1H, dd, 7.1, 5.4 Hz)		

(*R*)-*N*-Benzyl-2-(hydroxymethyl)-5,5-dimethylmorpholine (6e)

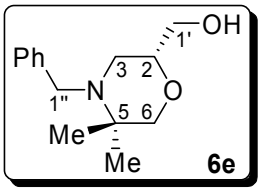


Table S8. ^{13}C and ^{1}H NMR data of **6e** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY	HMBC[a]
2	77.3	3.57 (1H, m)	3α, 3β	1', 3, 6
3	47.5	α: 2.26 (1H, dd, 11.8, 9.9 Hz) β: 2.34 (1H, dd, 11.8, 2.9 Hz)	2, 3β 2, 3α	1', 1", 2, 5 1', 1", 2, 5
5	52.9	---		
6	77.7	α: 3.45 (1H, dd, 10.9, 0.6 Hz) β: 3.52 (1H, d, 10.9 Hz)	6β 6α	2, 5, 5α-Me, 5β-Me 2, 5, 5α-Me, 5β-Me
1'	64.3	3.57 (2H, m)		(2), 3
1"	53.8	α: 3.02 (1H, d, 13.8 Hz) β: 4.01 (1H, d, 13.8 Hz)	1"β 1"α	3, Ph 3, 5, Ph
5α-Me	14.7	1.05 (3H, s)		5, 6, 5β-Me
5β-Me	24.4	1.05 (3H, s)		5, 6, 5α-Me
Ph	126.9, 128.4 128.7, 140.1	7.20-7.40 (5H, m)		1"
1'-OH	---	1.88 (1H, br. s)		

[a] Values in parentheses refer to cross peaks which are not unequivocally assignable due to signal overlap.

(2*R*,5*R*)-2-(Hydroxymethyl)-*N*-methyl-5-phenylmorpholine (6f)

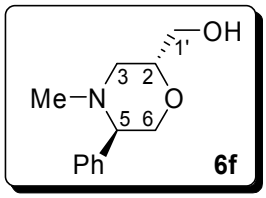


Table S9. ^{13}C and ^{1}H NMR data of **6f** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY	HMBC
2	76.6	3.86 (1H, m)	1'α, 1'β, 3α, 3β	1', 3
3	56.9	α: 2.21 (1H, dd, 11.4, 10.9 Hz) β: 2.87 (1H, dd, 11.5, 2.3 Hz)	2, 3β 2, 3a	1', 2, 5, NMe 2, 5, NMe
5	69.0	3.04 (1H, dd, 10.4, 3.4 Hz)	6α, 6β	3, 6, NMe
6	73.1	α: 3.51 (1H, dd, 11.5, 10.4 Hz) β: 3.80 (1H, dd, 11.5, 3.5 Hz)	5, 6β 5, 6α	2,5 2
1'	64.3	α: 3.63 (1H, br. dd, 11.4, 6.5 Hz) β: 3.72 (1H, br. d, 11.4 Hz)	1'β, 2 1'α, 2	2, 3 2, 3
NMe	43.8	2.07 (3H, s)		
Ph	128.0, 128.6 128.7, 139.0	7.25-7.40 (5H, m)		
1'-OH	---	1.91 (1H, br. s)		

(*R*)-*N*-Benzyl-2-(hydroxymethyl)-3,4-dihydro-2*H*-1,4-benzoxazine (6g)

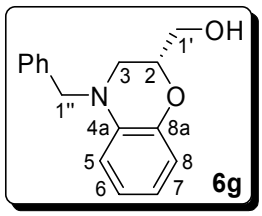


Table S10. ^{13}C and ^{1}H NMR data of **6g** and significant COSY and HMBC correlations.

No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY	HMBC
2	73.9	4.29 (1H, m)	1', 3	1, 1", 3, 8a
3	48.4	3.28 (2H, m)		1', 1", 2, 4a
4a	135.3			
5	116.6	6.87 (1H, dd, 7.9, 1.5 Hz)	6, 7	4a, 6, 7, 8a
6	121.9	6.81 (1H, m)	5, 7, 8	4a, 5, 7, 8, 8a
7	118.2	6.66 (1H, m)	5, 6, 8	5, 8, 8a
8	112.7	6.71 (1H, dd, 8.1, 1.5 Hz)	6, 7	7, 8a
8a	143.6			
1'	63.6	3.82 (2H, m)	2, OH	2, 3
1"	55.1	4.44 (2H, s)		2, 3, 4a, Ph
Ph	127.3, 127.4 128.9, 138.0	7.23-7.34 (5H, m)		
1'-OH	---	1.9 (1H, t, 6.4 Hz)		

(2*S*,5*R*)-*N*-Benzyl-2-(hydroxymethyl)-5-phenylmorpholine (6k)

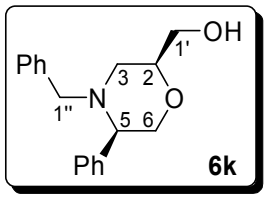
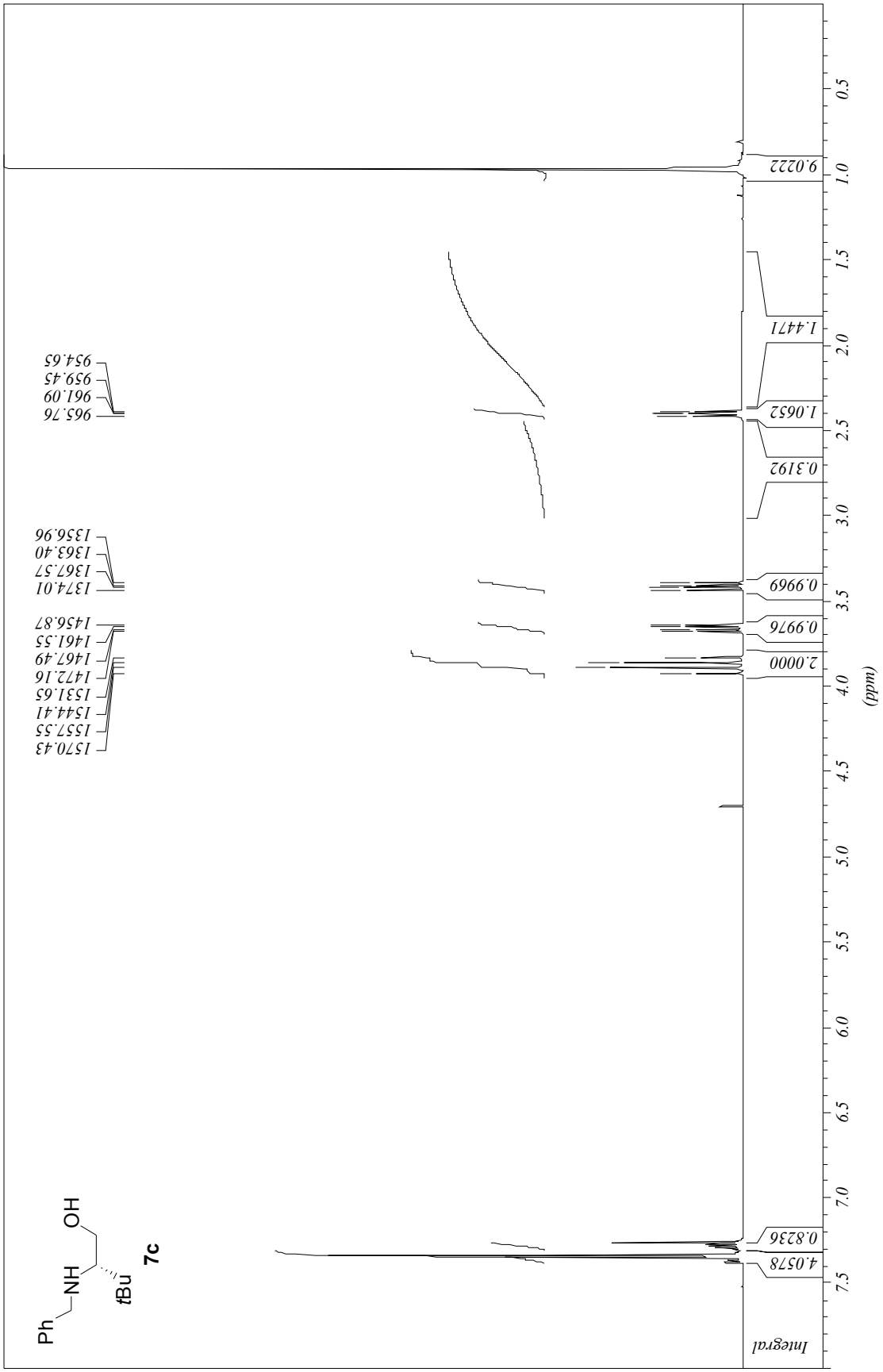


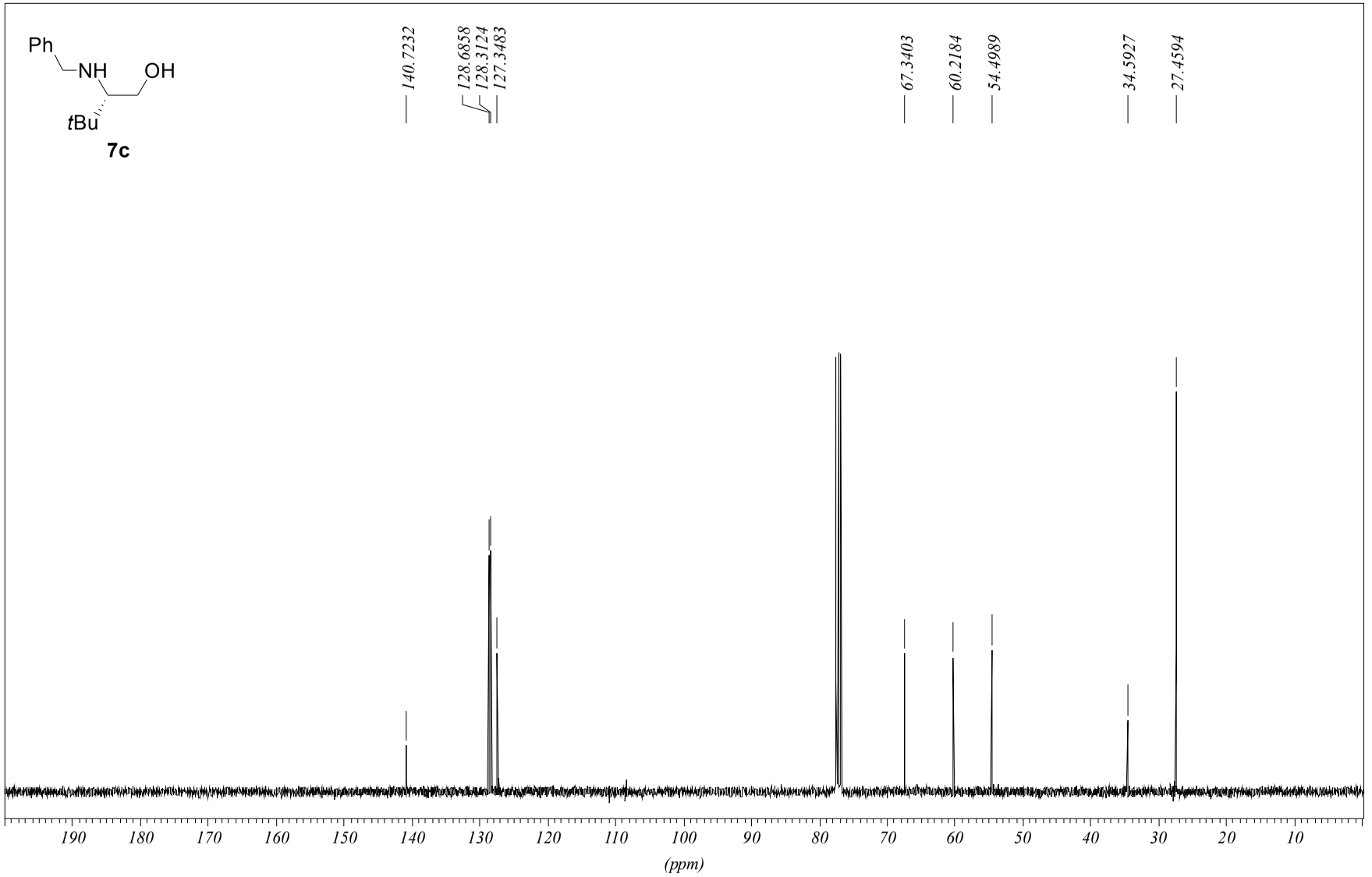
Table S11. ^{13}C and ^{1}H NMR data of **6k** and significant COSY and HMBC correlations.

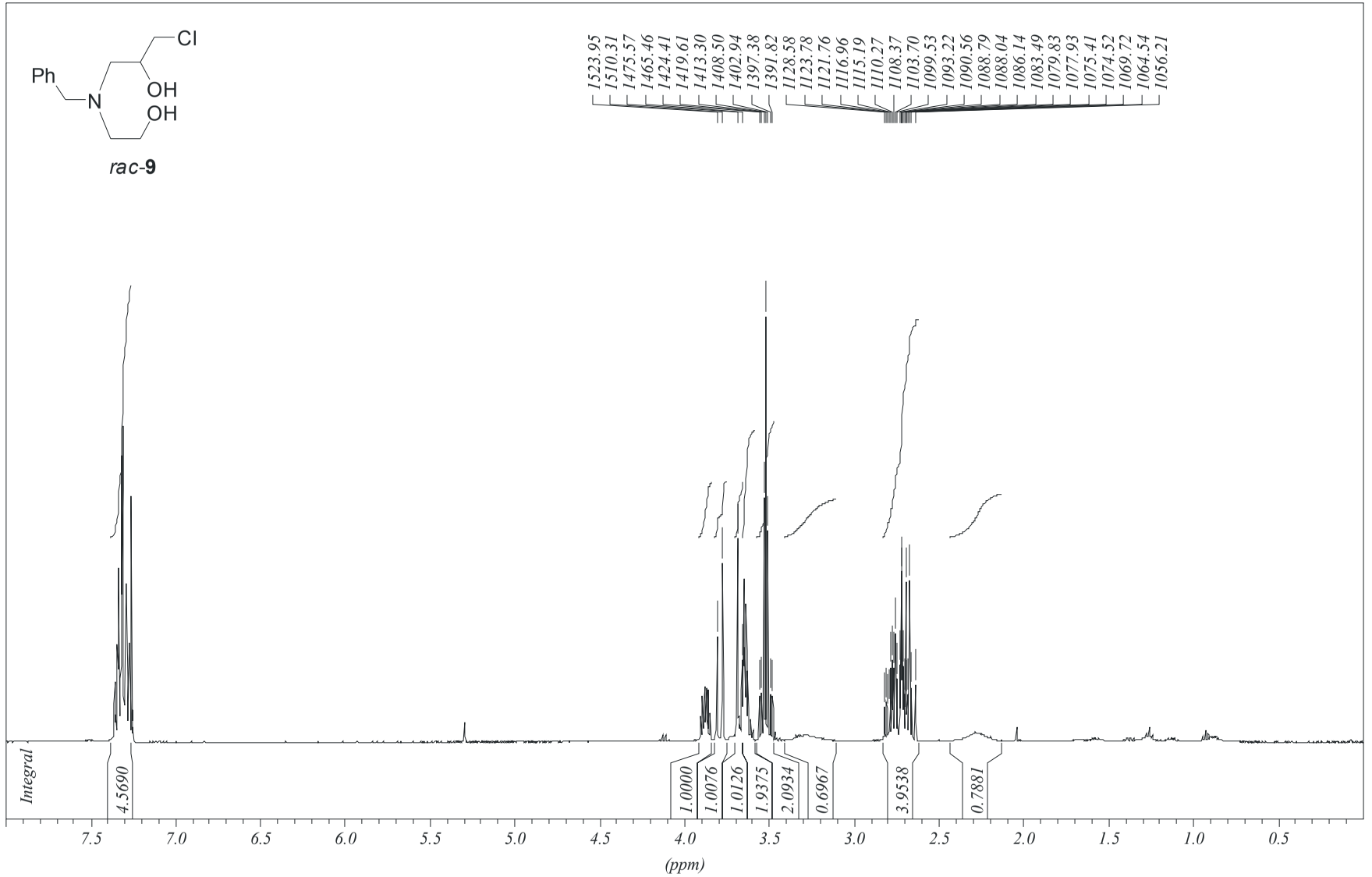
No.	^{13}C: δ [ppm]	^{1}H: δ [ppm] (int., m, *J*)	COSY[a]	HMBC[a]
2	73.4	3.87 (1H, m)	(1'α), 1'β, 3α, 3β	1', 3, 6
3	51.7	α: 2.49 (1H, dd, 12.1, 4.0 Hz) β: 2.83 (1H, dd, 12.1, 2.7 Hz)	2, 3β 2, 3α	1', 1", 2, 5 1', 1", 2, 5
5	66.8	3.51 (1H, dd, 9.3, 3.8 Hz)	(6α), 6β	1", 3, 6, Ph
6	68.3	α: 3.73 (1H, m) β: 4.02 (1H, dd, 11.6, 7.4 Hz)	(5), (6β), (OH) (5), (6α),	(2), (3), (5) 2, 5, Ph
1'	63.7	α: 3.73 (1H, m) β: 4.20 (1H, dd, 11.6, 7.4 Hz)	(1'β), 2 (1'α), 2	(2), (3) 2, 3
1"	59.3	α: 2.91 (1H, d, 13.4 Hz) β: 3.75 (1H, d, 13.4 Hz)	(1"α) (1"β)	3, 5, Ph (3), (5), Ph
Ph	127.3, 128.1 128.52, 128.54 128.76, 128.81 138.1, 139.0	7.20-7.40 (8H, m) 7.46 (2H, m)		1", 5
1'-OH	---	2.99 (1H, br. s)	(6α)	

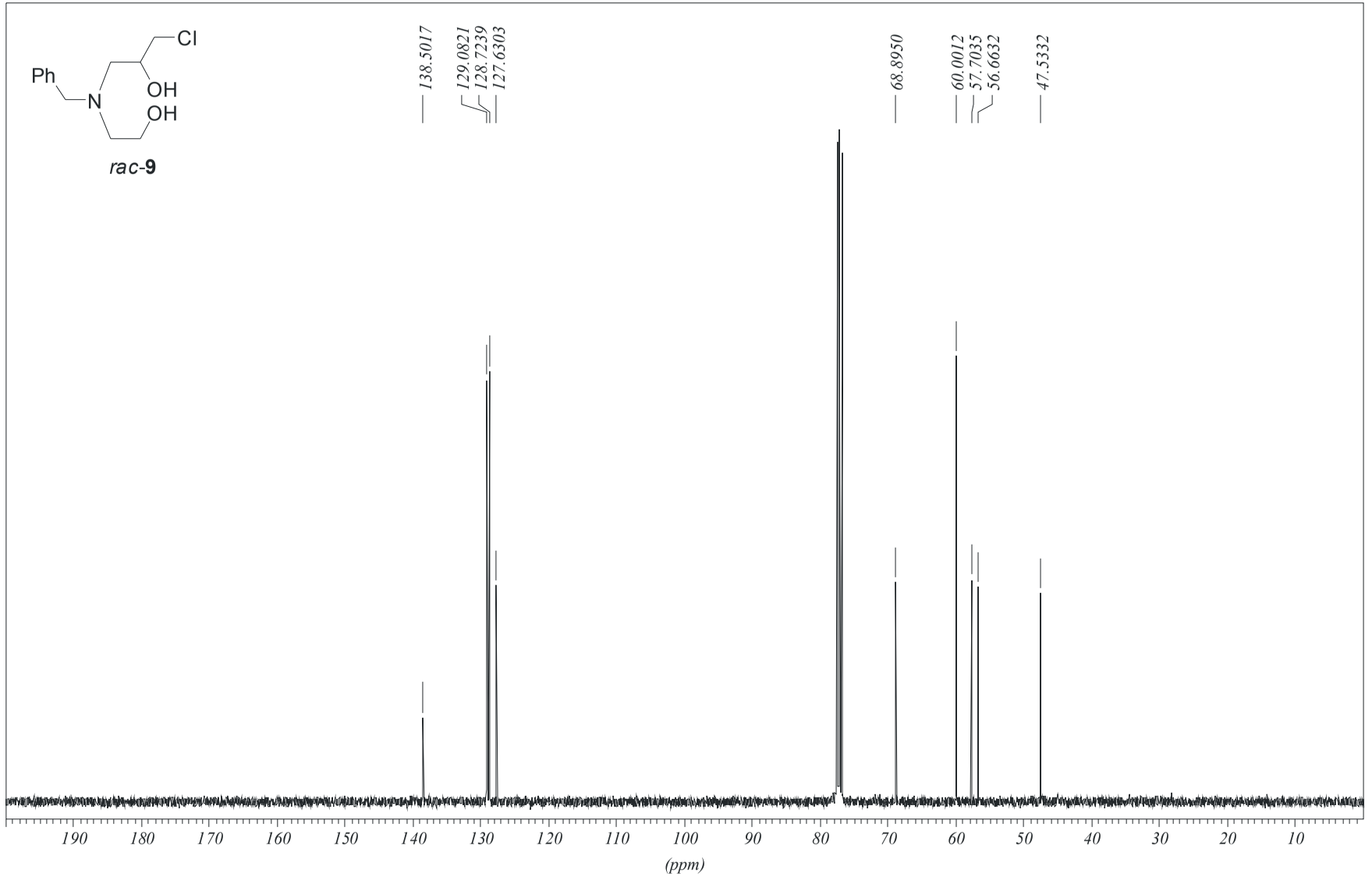
[a] Values in parentheses refer to cross peaks which are not unequivocally assignable due to signal overlap.

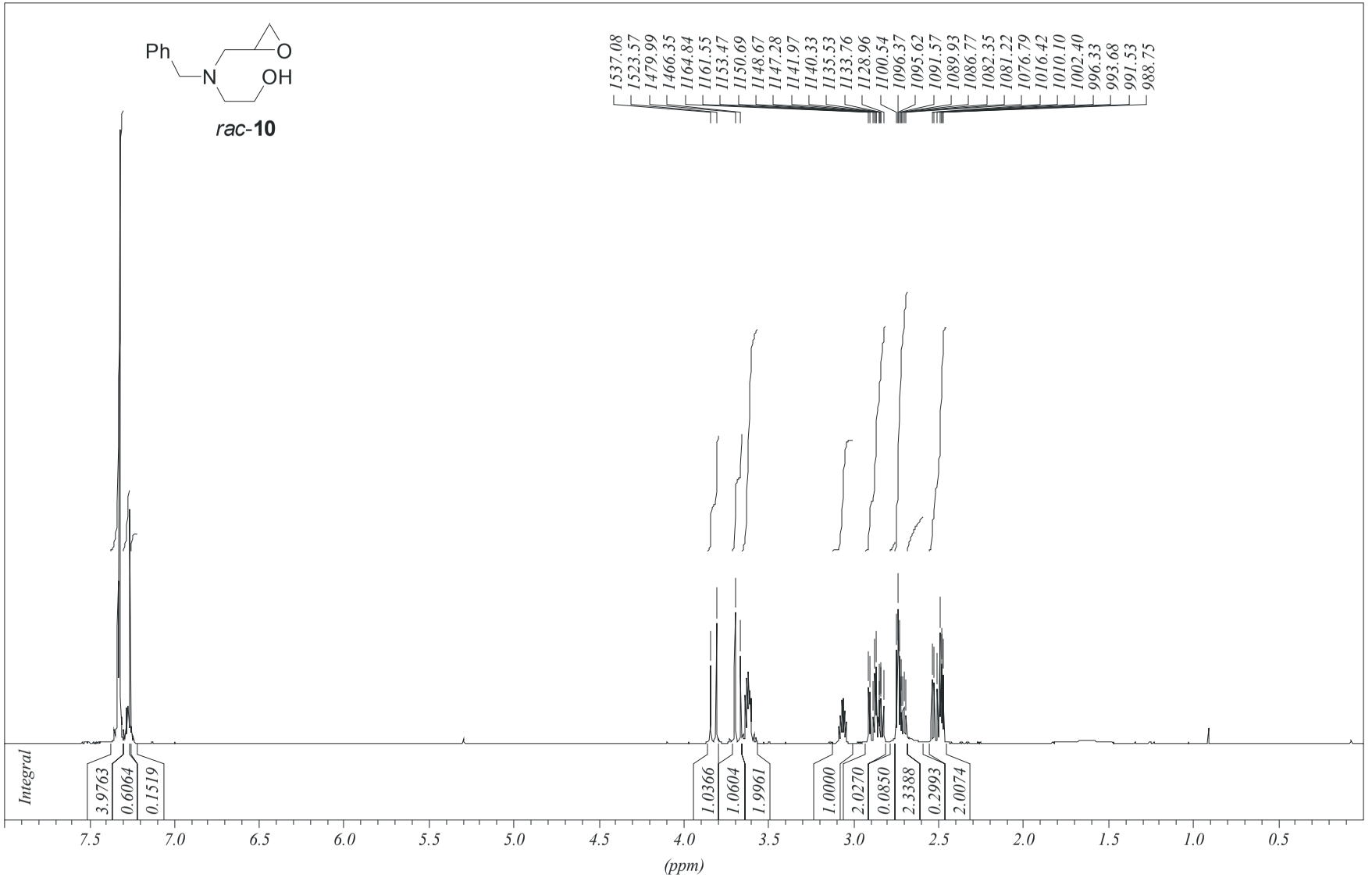
^{1}H and ^{13}C NMR Spectra

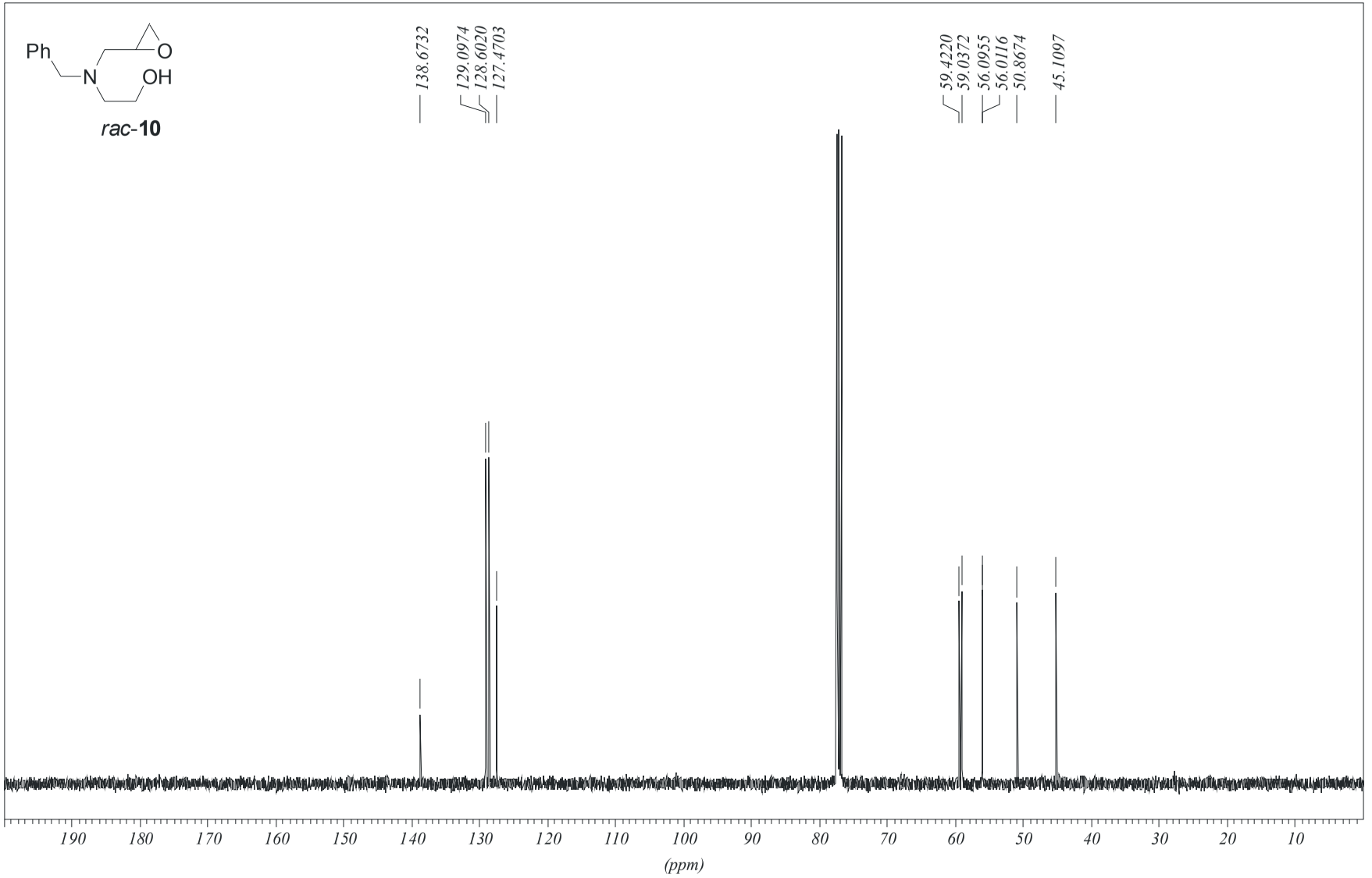


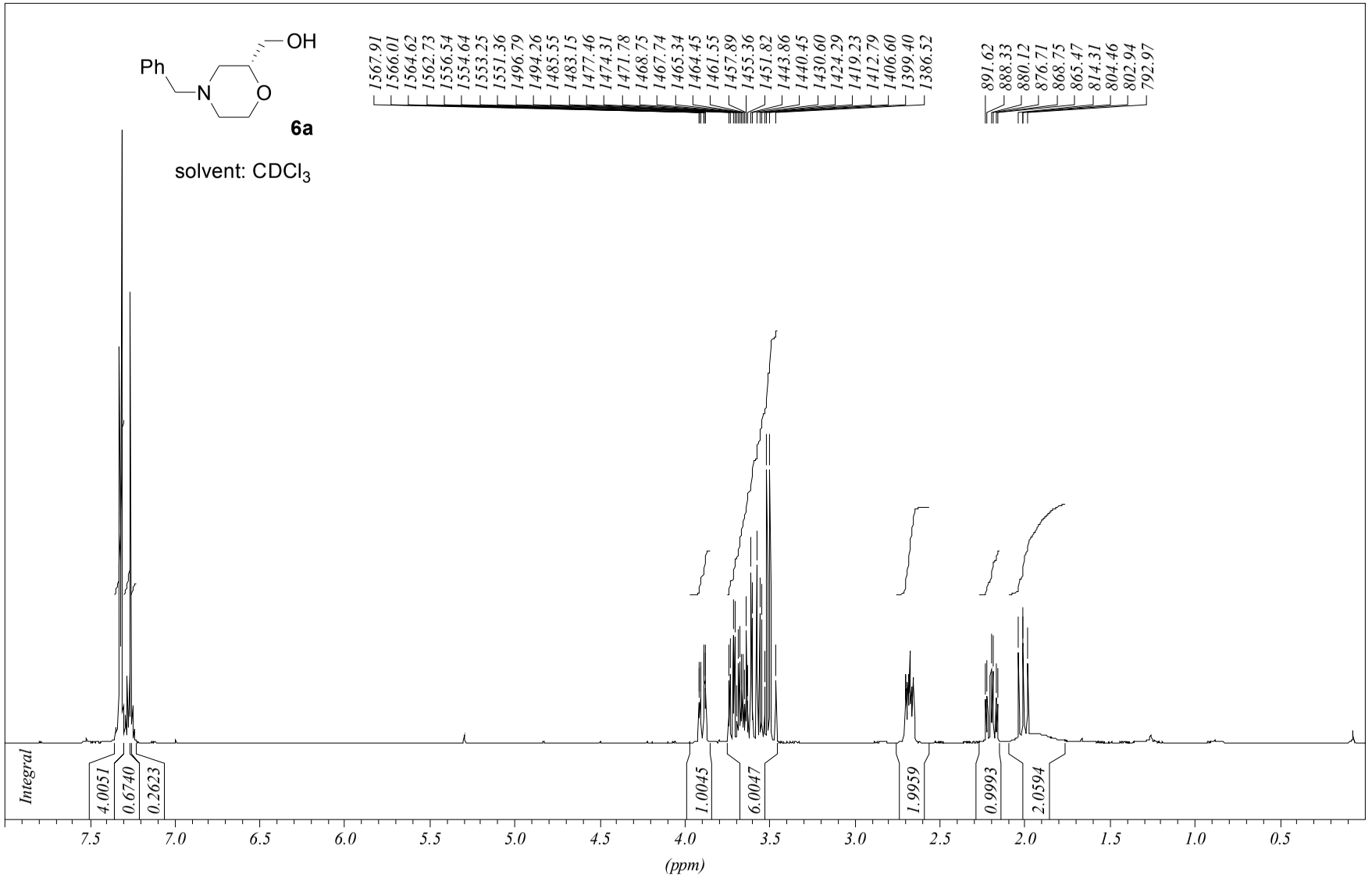

Ph
NH
OH
tBu
7c
140.7232
128.6858
128.3124
127.3483
67.3403
60.2184
54.4989
34.5927
27.4594
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

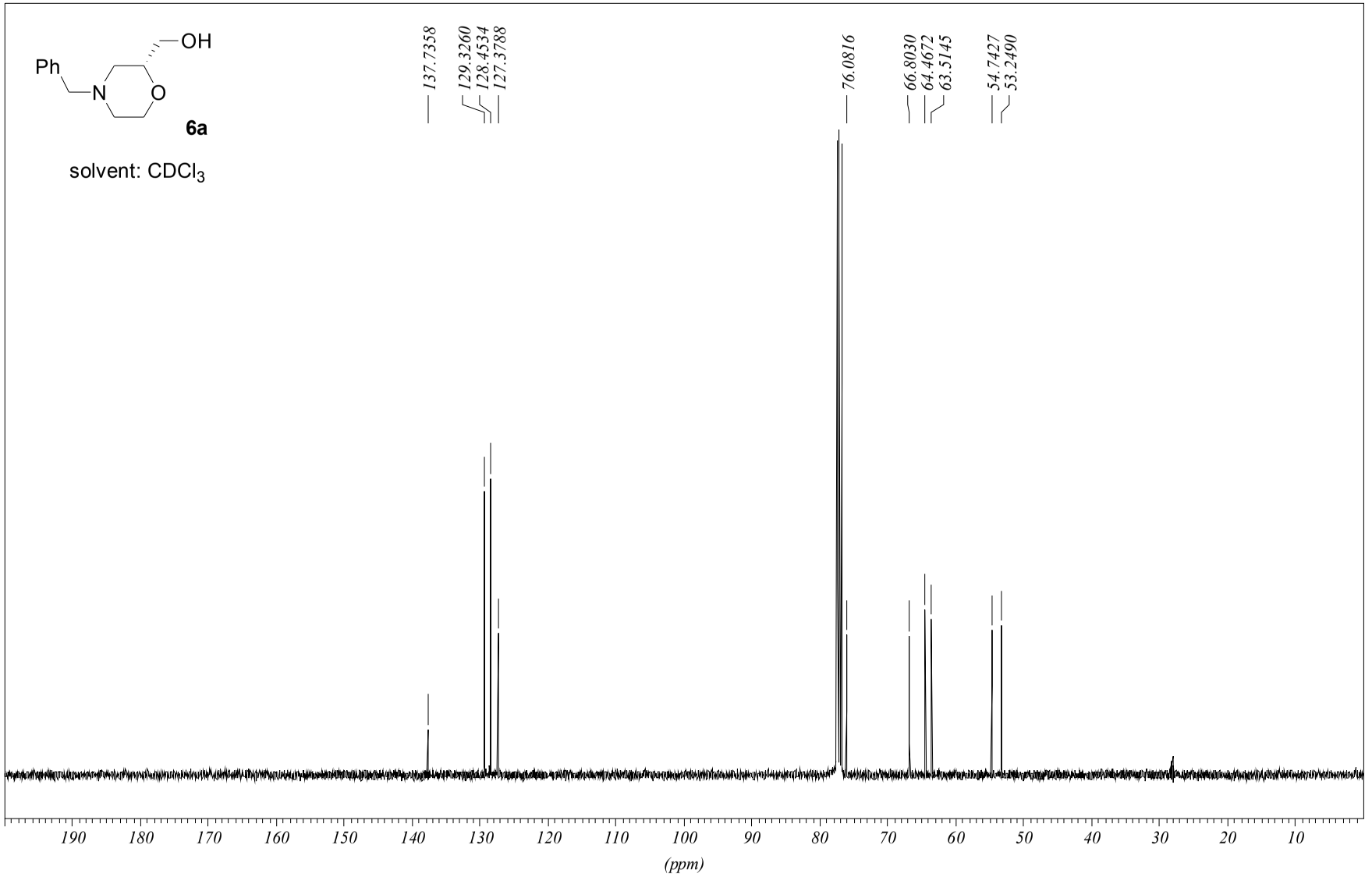

Ph
N
Cl
OH
OH
rac-9
1523.95
1510.31
1475.57
1465.46
1424.41
1419.61
1413.30
1408.50
1402.94
1397.38
1391.82
1128.58
1123.78
1121.76
1116.96
1115.19
1110.27
1108.37
1103.70
1099.53
1093.22
1090.56
1088.79
1088.04
1086.14
1083.49
1079.83
1077.93
1075.41
1074.52
1069.72
1064.54
1056.21
Integral
4.5690
1.0000
1.0076
1.0126
1.9375
2.0934
0.6967
3.9538
0.7881
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
(ppm)

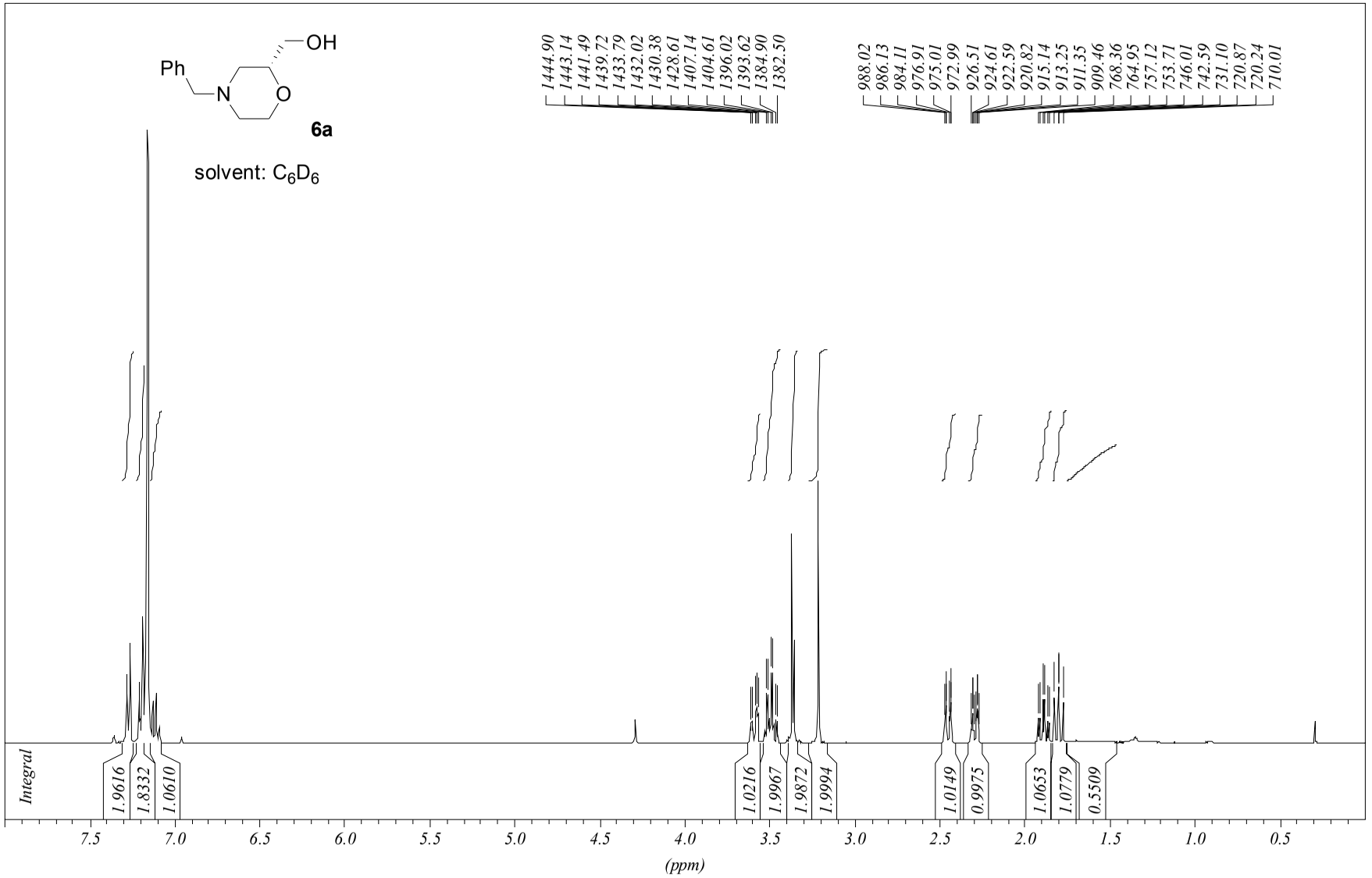

Cl
Ph
N
OH
OH
rac-9
138.5017
129.0821
128.7239
127.6303
68.8950
60.0012
57.7035
56.6632
47.5332
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

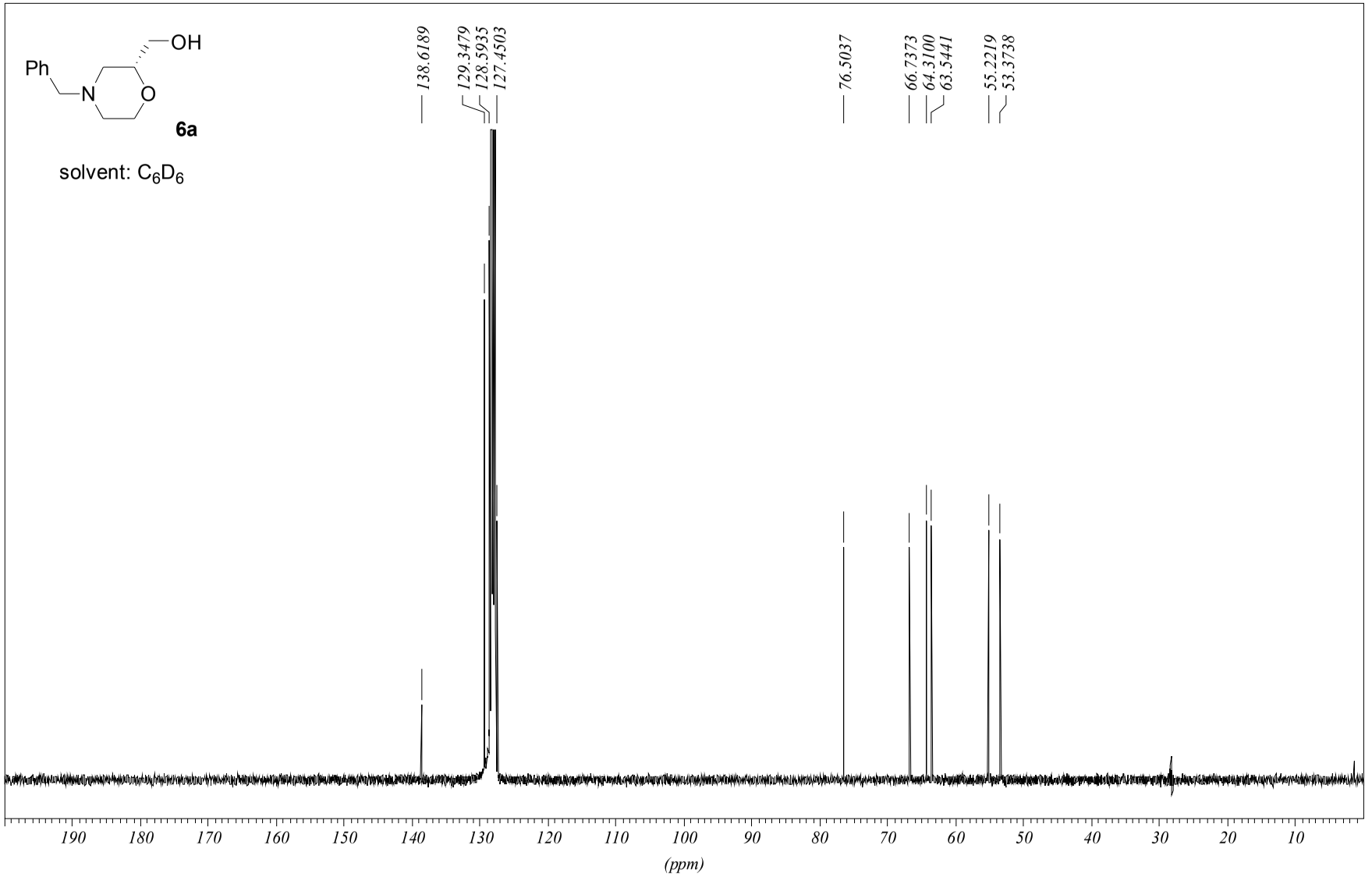

Ph
N
O
OH
rac-10
1537.08
1523.57
1479.99
1466.35
1164.84
1161.55
1153.47
1150.69
1148.67
1147.28
1141.97
1140.33
1133.53
1133.76
1128.96
1100.54
1096.37
1095.62
1091.57
1089.93
1086.77
1082.35
1081.22
1076.79
1016.42
1010.10
1002.40
996.33
993.68
991.53
988.75
Integral
3.9763
0.6064
0.1519
1.0366
1.0604
1.9961
1.0000
2.0270
0.0850
2.3388
0.2993
2.0074
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
(ppm)

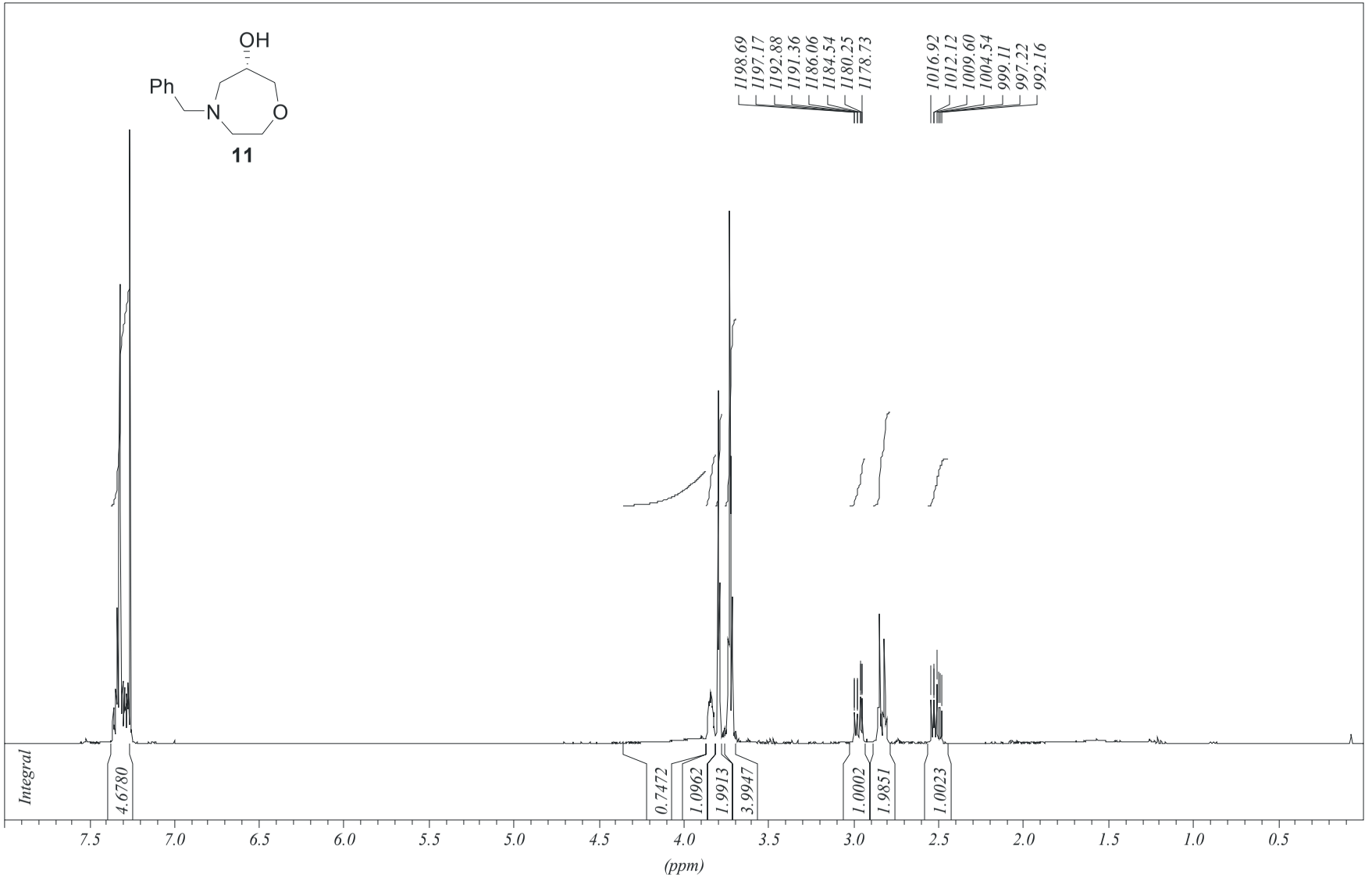

Ph
N
O
OH
rac-10
138.6732
129.0974
128.6020
127.4703
59.4220
59.0372
56.0955
56.0116
50.8674
45.1097
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

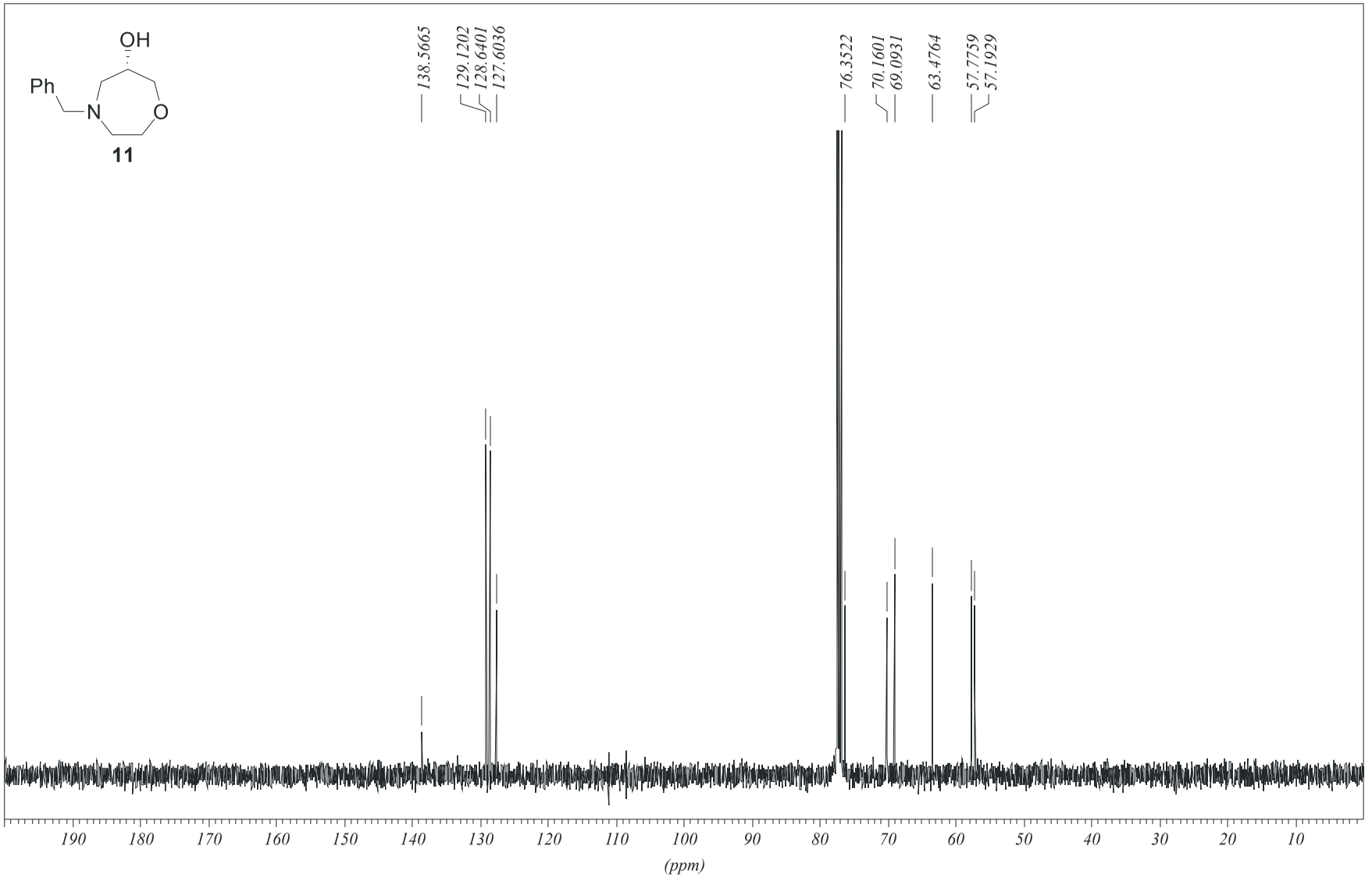

OH
Ph
N
O
6a
solvent: CDCl3
1567.91
1566.01
1564.62
1562.73
1556.54
1554.64
1553.25
1551.36
1496.79
1494.26
1485.55
1483.15
1477.46
1474.31
1471.78
1468.75
1467.74
1465.34
1464.45
1461.55
1457.89
1455.36
1451.82
1443.86
1440.45
1430.60
1424.29
1419.23
1412.79
1406.60
1399.40
1386.52
891.62
888.33
880.12
876.71
868.75
865.47
814.31
804.46
802.94
792.97
Integral
4.0051
0.6740
0.2623
1.0045
6.0047
1.9959
0.9993
2.0594
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
(ppm)


OH
Ph
N
O
6a
solvent: CDCl3
137.7358
129.3260
128.4534
127.3788
76.0816
66.8030
64.4672
63.5145
54.7427
53.2490
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

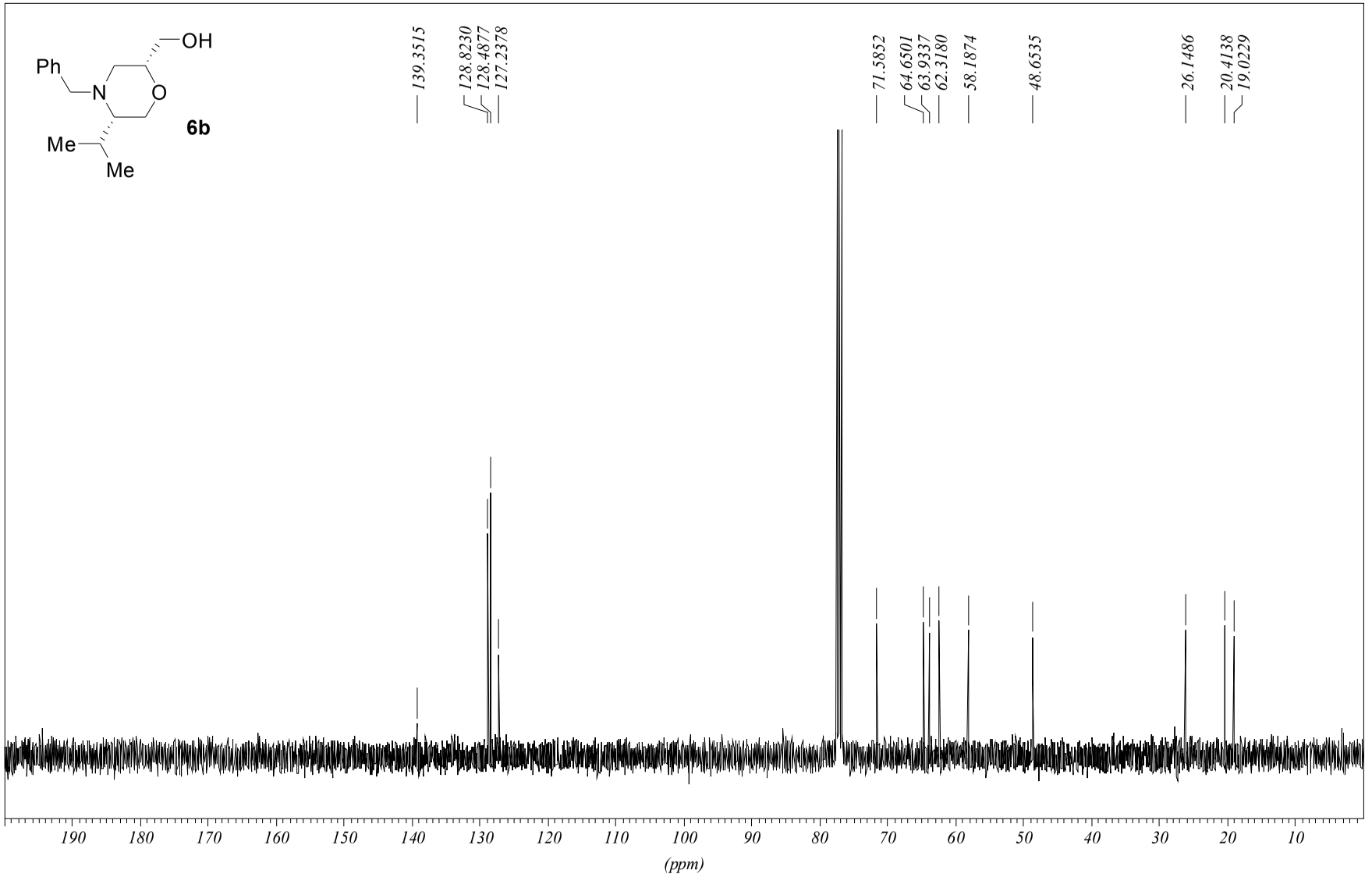

Ph
N
O
OH
6a
solvent: C6D6
1444.90
1443.14
1441.49
1439.72
1433.79
1432.02
1430.38
1428.61
1407.14
1404.61
1396.02
1393.62
1384.90
1382.50
988.02
986.13
984.11
976.91
975.01
972.99
926.51
924.61
922.59
920.82
915.14
913.25
911.35
909.46
768.36
764.95
757.12
753.71
746.01
742.59
731.10
720.87
720.24
710.01
Integral
1.9616
1.8332
1.0610
1.0216
1.9967
1.9872
1.9994
1.0149
0.9975
1.0653
1.0779
0.5509
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
(ppm)

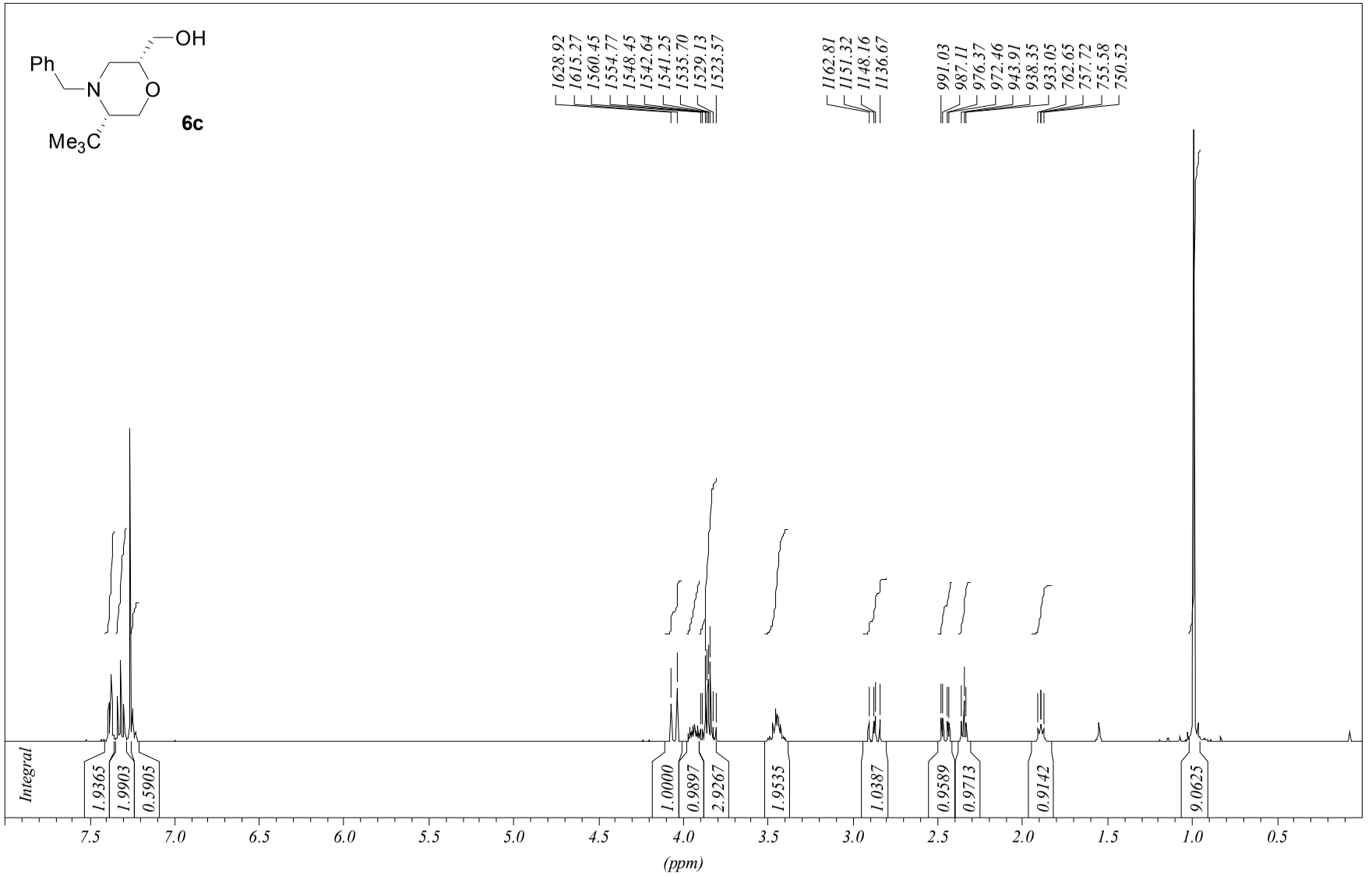

OH
Ph
N
O
6a
solvent: C6D6
138.6189
129.3479
128.5935
127.4503
76.5037
66.7373
64.3100
63.5441
55.2219
53.3738
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

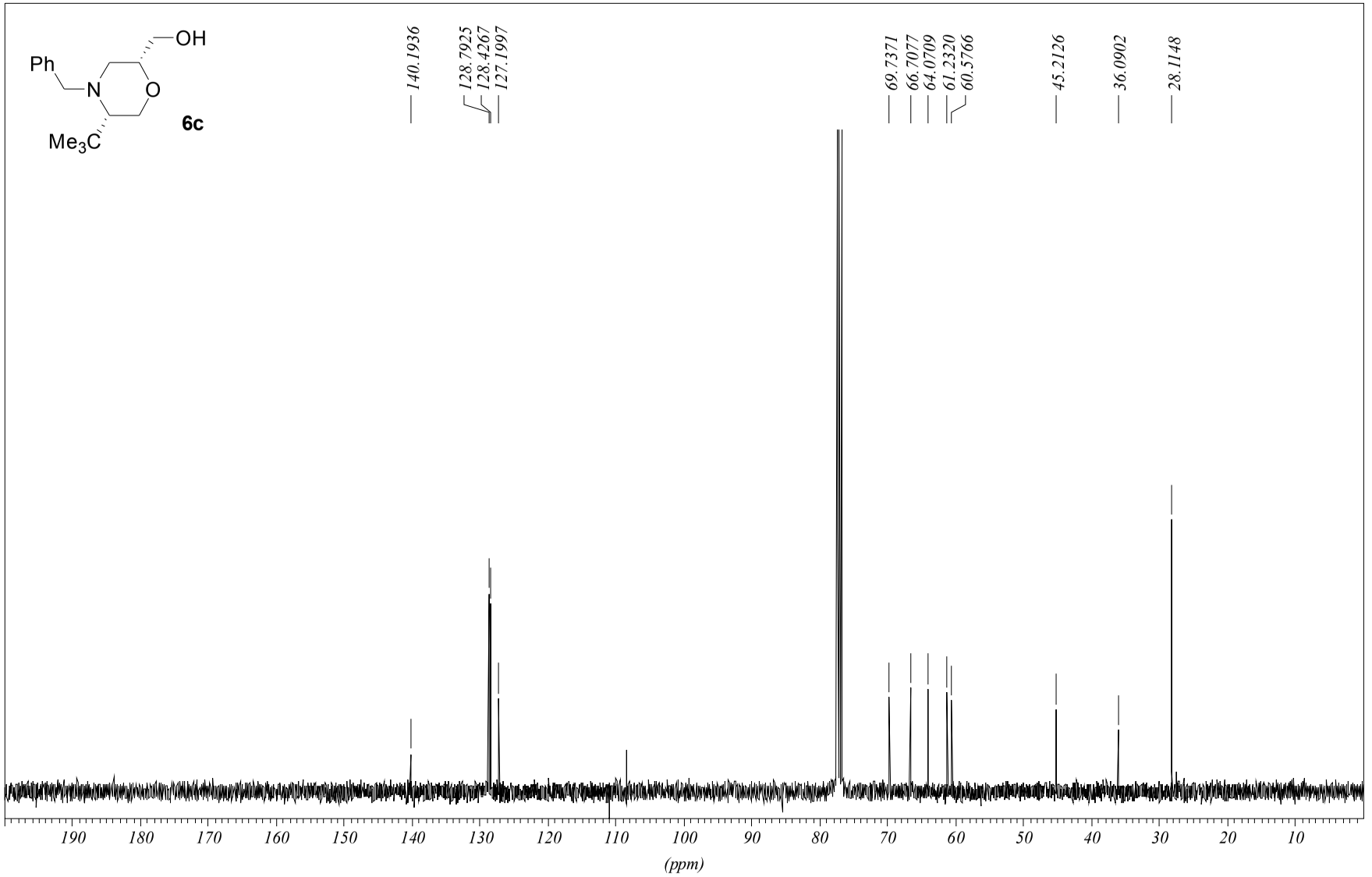

OH
Ph
N
O
11
1198.69
1197.17
1192.88
1191.36
1186.06
1184.54
1180.25
1178.73
1016.92
1012.12
1009.60
1004.54
999.11
997.22
992.16
Integral
4.6780
0.7472
1.0962
1.9913
3.9947
1.0002
1.9851
1.0023
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)


OH
Ph
N
O
11
138.5665
129.1202
128.6401
127.6036
76.3522
70.1601
69.0931
63.4764
57.7759
57.1929
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

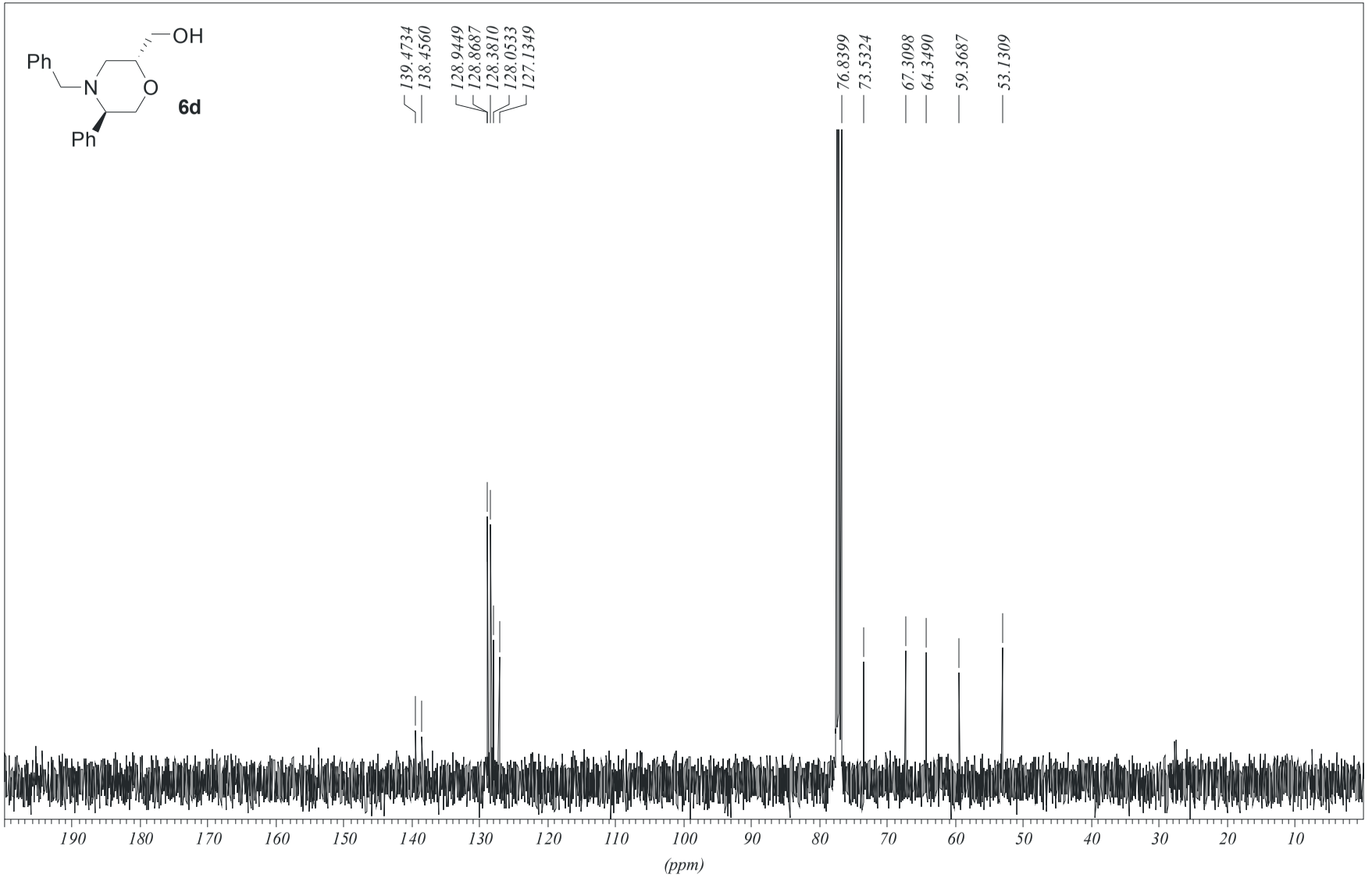
6b
OH
O
N
Ph
Me
Me
Integral
3.9410
0.2437
0.5936
1.0000
0.9684
0.9659
1.0125
1.0372
1.9492
0.9869
0.7267
1.0628
0.9669
0.9962
5.9810
(ppm)
401.64
394.82
387.12
380.30
1614.39
1600.87
1569.67
1565.13
1557.80
1553.38
1537.72
1534.31
1525.84
1522.56
1476.96
1470.26
1465.34
1458.77
1442.98
1438.56
1435.28
1429.46
1427.06
1423.78
1138.69
1130.86
1125.55
1117.72
956.42
953.13
943.15
940.00


OH
Ph
N
O
Me
Me
6b
139.3515
128.8230
128.4877
127.2378
71.5852
64.6501
63.9337
62.3180
58.1874
48.6535
26.1486
20.4138
19.0229
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

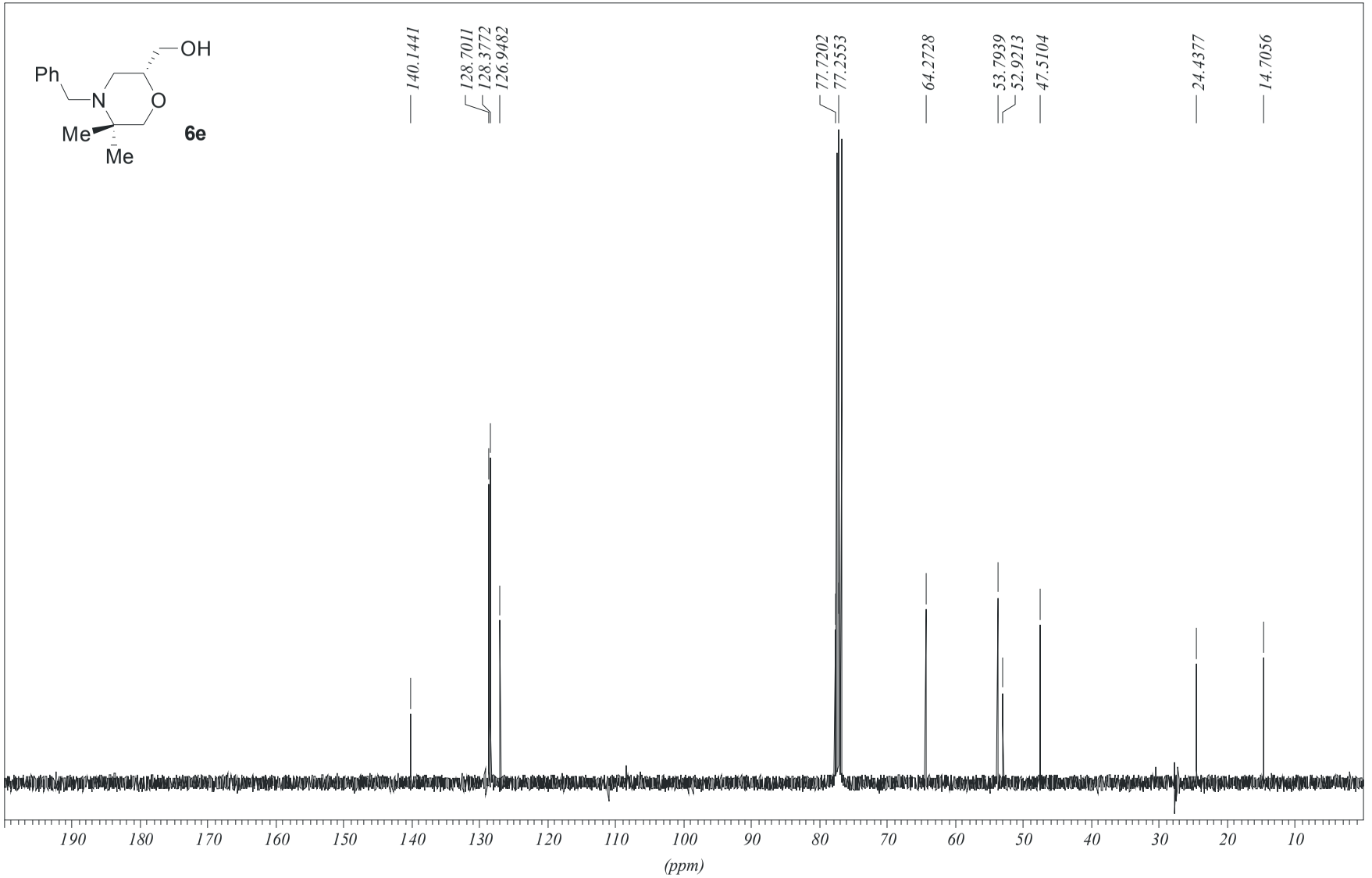

Ph
N
O
OH
Me3C
6c
1628.92
1615.27
1560.45
1554.77
1548.45
1542.64
1541.25
1535.70
1529.13
1523.57
1162.81
1151.32
1148.16
1136.67
991.03
987.11
976.37
972.46
943.91
938.35
933.05
762.65
757.72
755.58
750.52
Integral
1.9365
1.9903
0.5905
1.0000
0.9897
2.9267
1.9535
1.0387
0.9589
0.9713
0.9142
9.0625
7.5
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
(ppm)

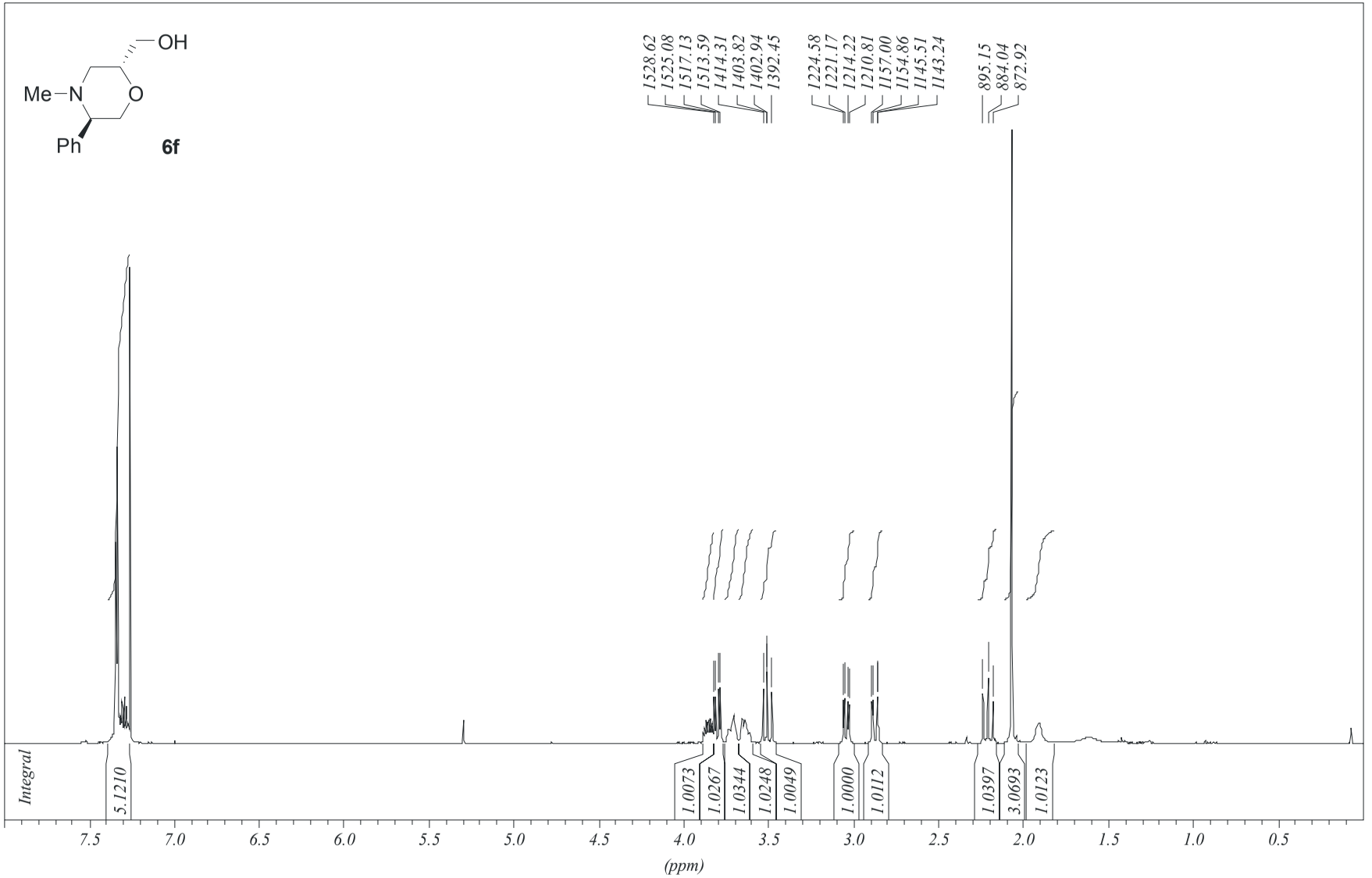

OH
Ph
N
O
Me3C
6c
140.1936
128.7925
128.4267
127.1997
69.7371
66.7077
64.0709
61.2320
60.5766
45.2126
36.0902
28.1148
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

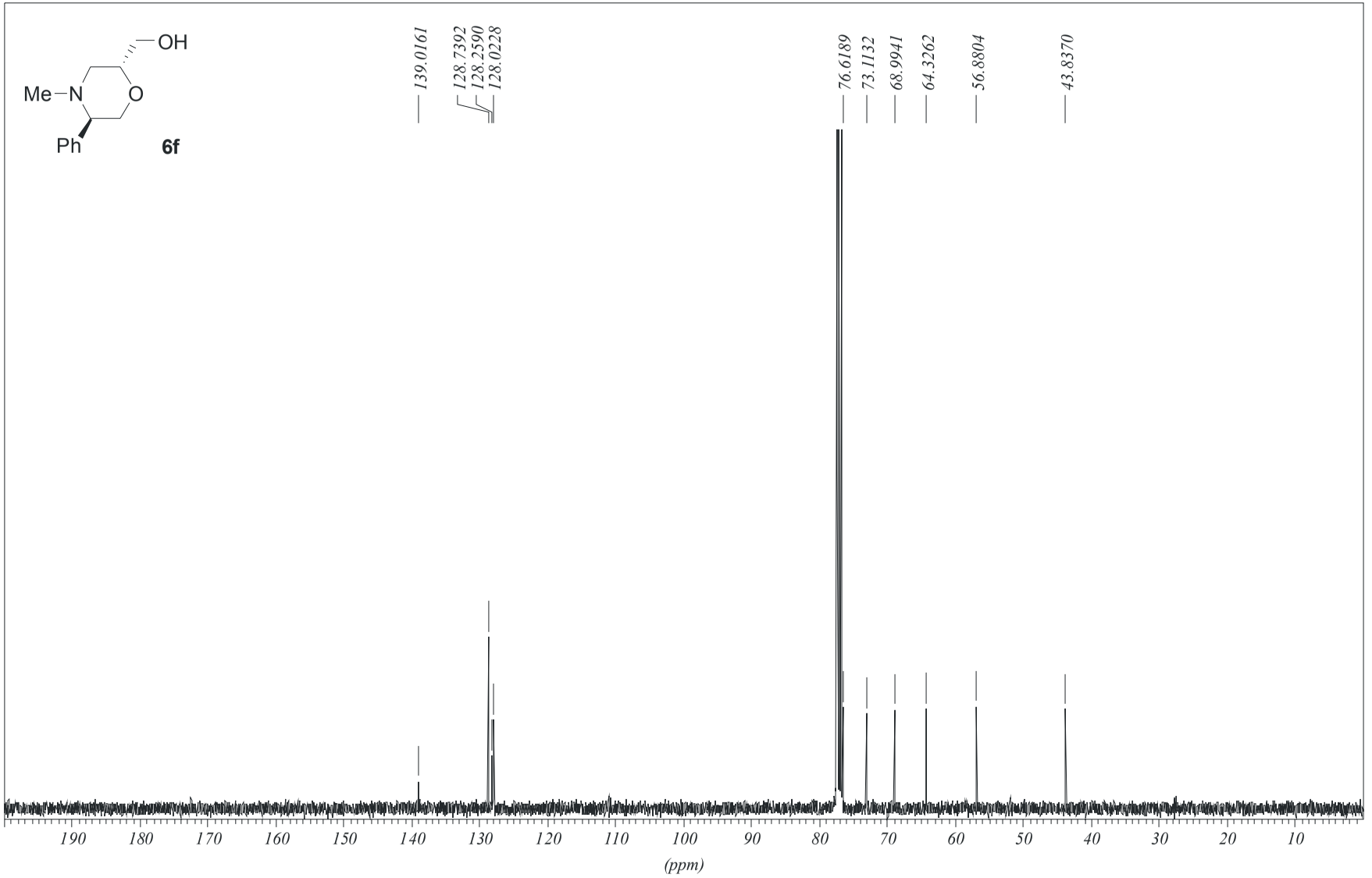
Integral
1.9700
2.0357
4.6349
1.0962
0.9742
0.9923
0.9768
0.9977
1.0001
3.0365
1.0150
2.0108
(ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
6d
OH
O
N
Ph
Ph
1551.48
1548.07
1543.40
1540.12
1536.71
1530.01
1458.64
1455.23
1451.70
1447.15
1443.74
1440.08
1435.78
1427.82
1425.30
1424.41
1422.52
1421.63
1416.20
1413.93
1411.02
1410.01
1404.71
1361.00
1357.59
1350.64
1347.23
1157.51
1144.25
1118.35
1116.08
1106.73
1104.46
836.17
825.43
824.67
813.93
738.52
733.09
731.45
726.02

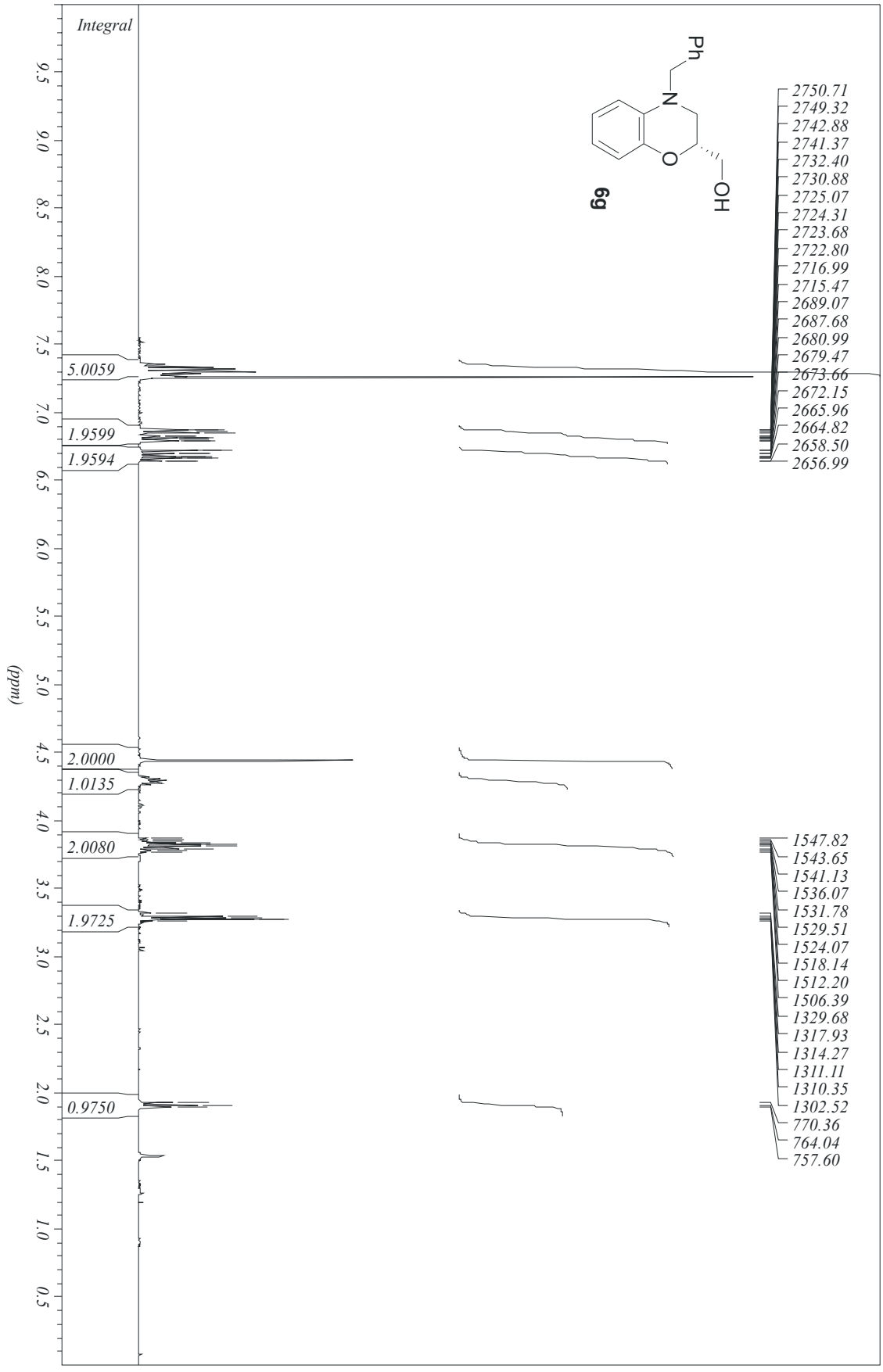

OH
Ph
N
O
Ph
6d
139.4734
138.4560
128.9449
128.8687
128.3810
128.0533
127.1349
76.8399
73.5324
67.3098
64.3490
59.3687
53.1309
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

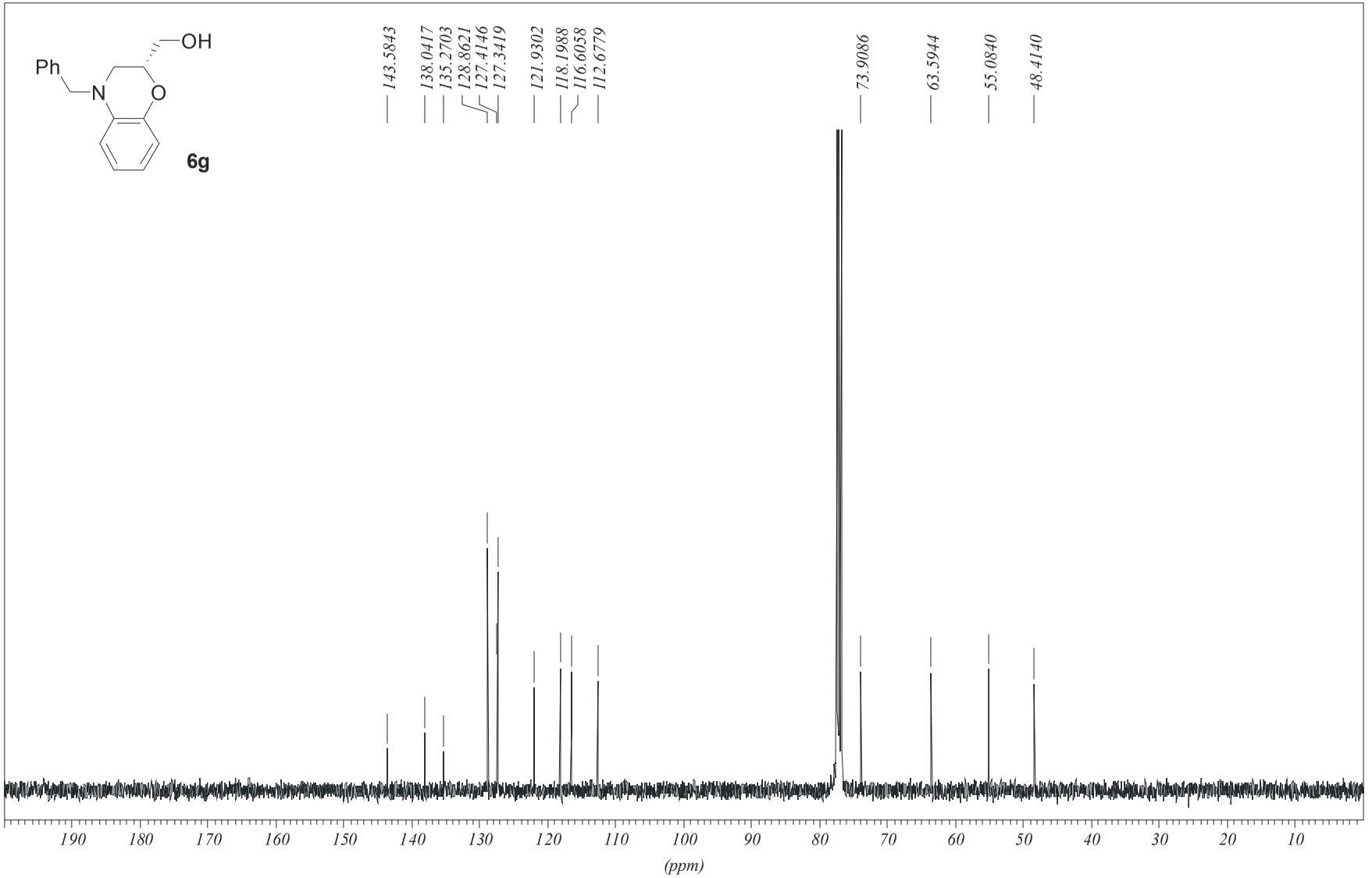
Integral
3.9732
0.9222
1.0002
3.9617
1.0120
0.9810
1.9757
0.8776
5.9864
(ppm)
1610.73
1596.96
1416.20
1405.34
1385.38
1384.75
1374.52
1373.76
1213.97
1200.20
946.19
943.41
934.44
931.41
915.36
905.51
903.74
893.64
OH
O
N
Me
Me
Ph
6e

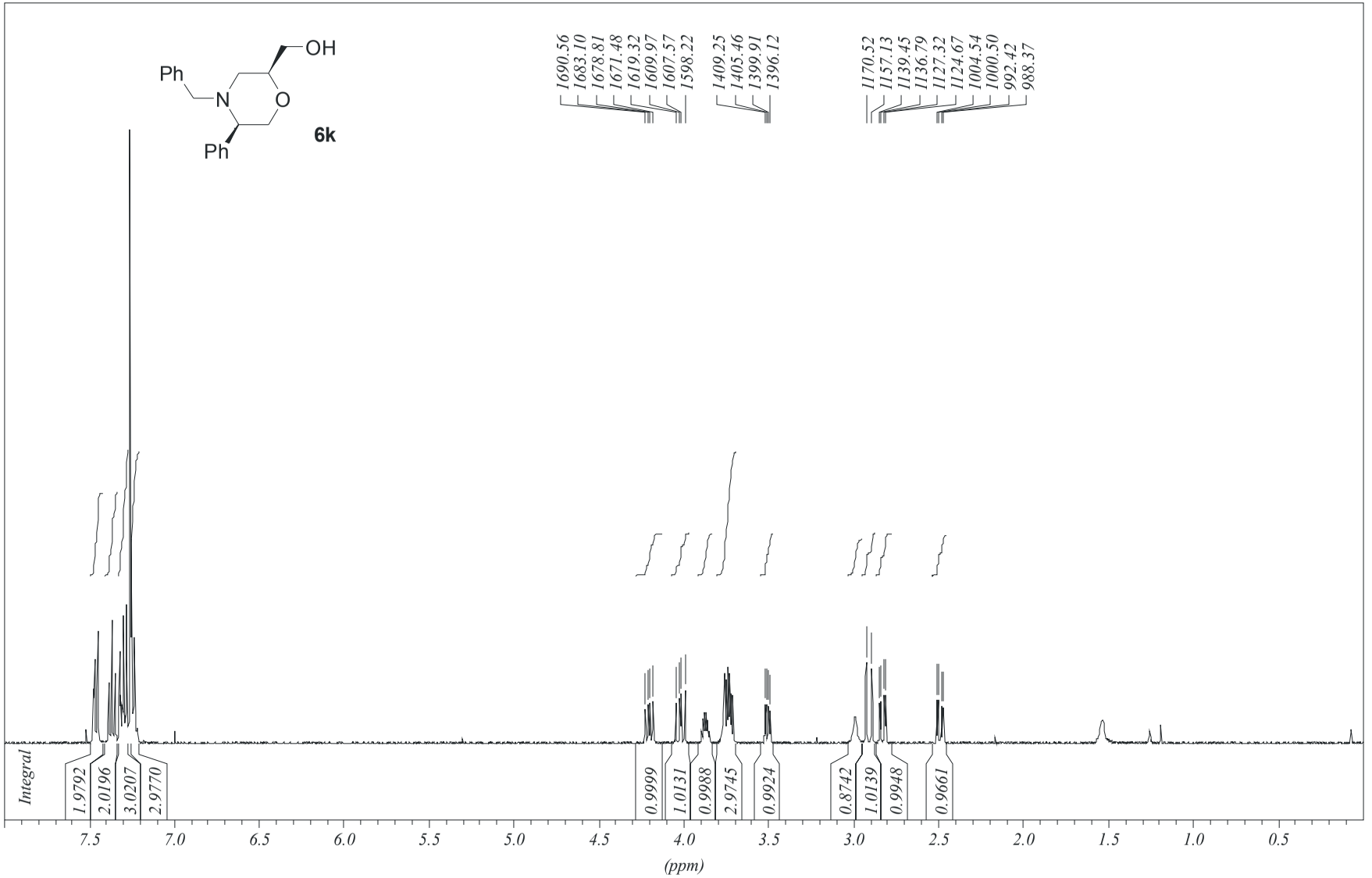

OH
Ph
N
O
Me
Me
6e
140.1441
128.7011
128.3772
126.9482
77.7202
77.2553
64.2728
53.7939
52.9213
47.5104
24.4377
14.7056
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

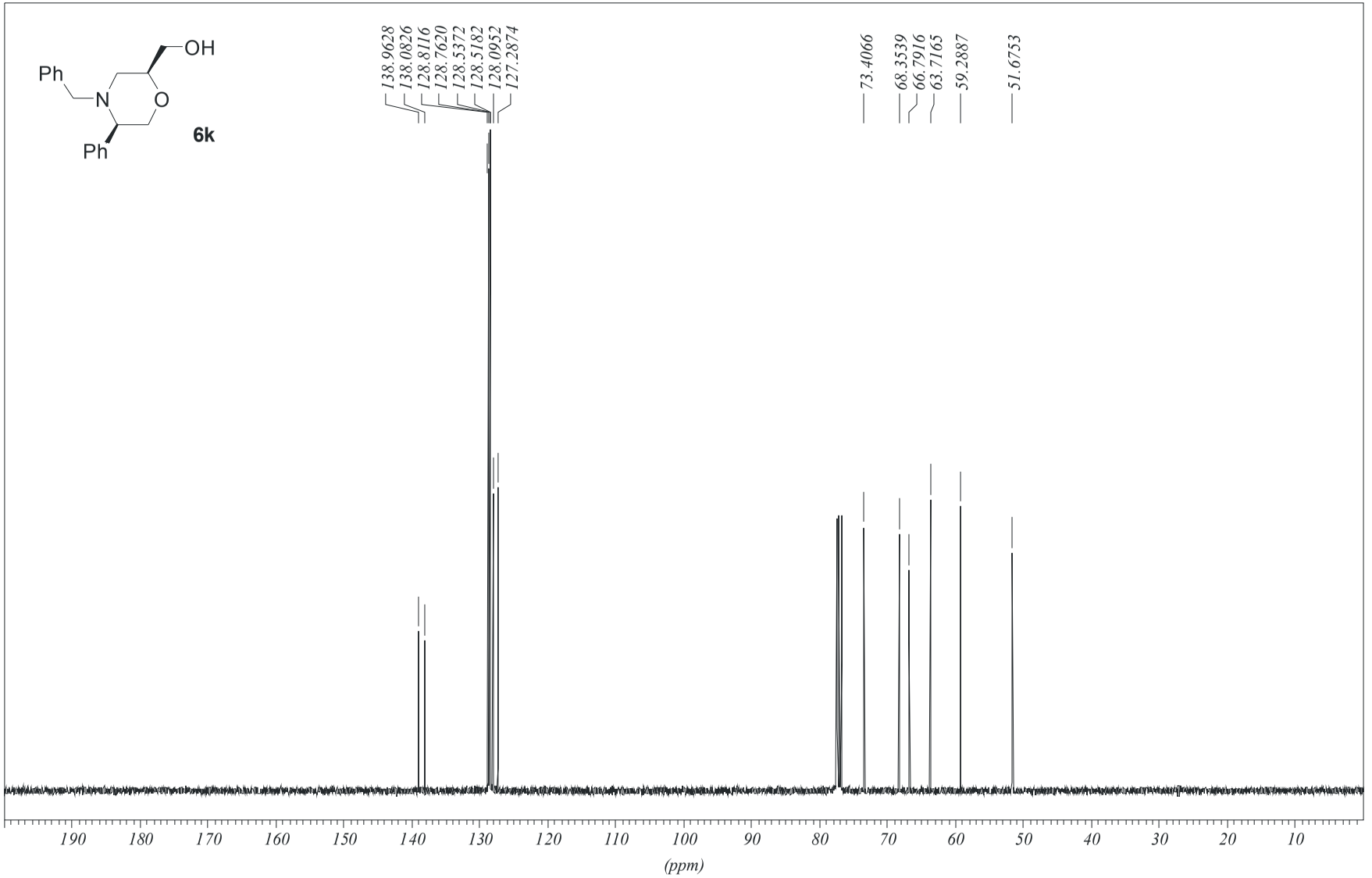

OH
Me–N
O
Ph
6f
1528.62
1525.08
1517.13
1513.59
1414.31
1403.82
1402.94
1392.45
1224.58
1221.17
1214.22
1210.81
1157.00
1154.86
1145.51
1143.24
895.15
884.04
872.92
Integral
5.1210
1.0073
1.0267
1.0344
1.0248
1.0049
1.0000
1.0112
1.0397
3.0693
1.0123
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

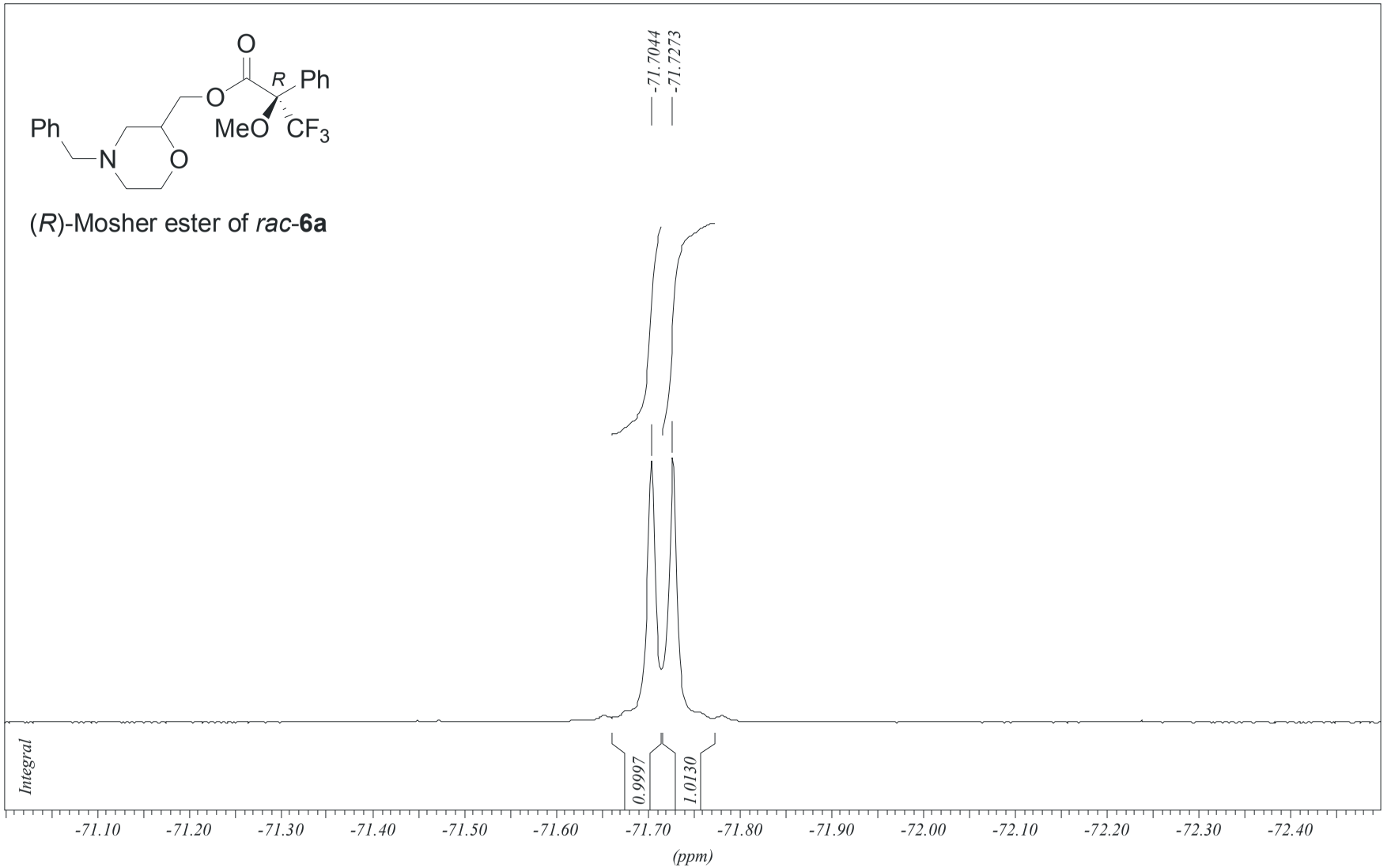

OH
Me–N
O
Ph
6f
139.0161
128.7392
128.2590
128.0228
76.6189
73.1132
68.9941
64.3262
56.8804
43.8370
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)


Integral
5.0059
1.9599
1.9594
2.0000
1.0135
2.0080
1.9725
0.9750
(ppm)
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
Ph
N
O
OH
6g
2750.71
2749.32
2742.88
2741.37
2732.40
2730.88
2725.07
2724.31
2723.68
2722.80
2716.99
2715.47
2689.07
2687.68
2680.99
2679.47
2673.66
2672.15
2665.96
2664.82
2658.50
2656.99
1547.82
1543.65
1541.13
1536.07
1531.78
1529.51
1524.07
1518.14
1512.20
1506.39
1329.68
1317.93
1314.27
1311.11
1310.35
1302.52
770.36
764.04
757.60

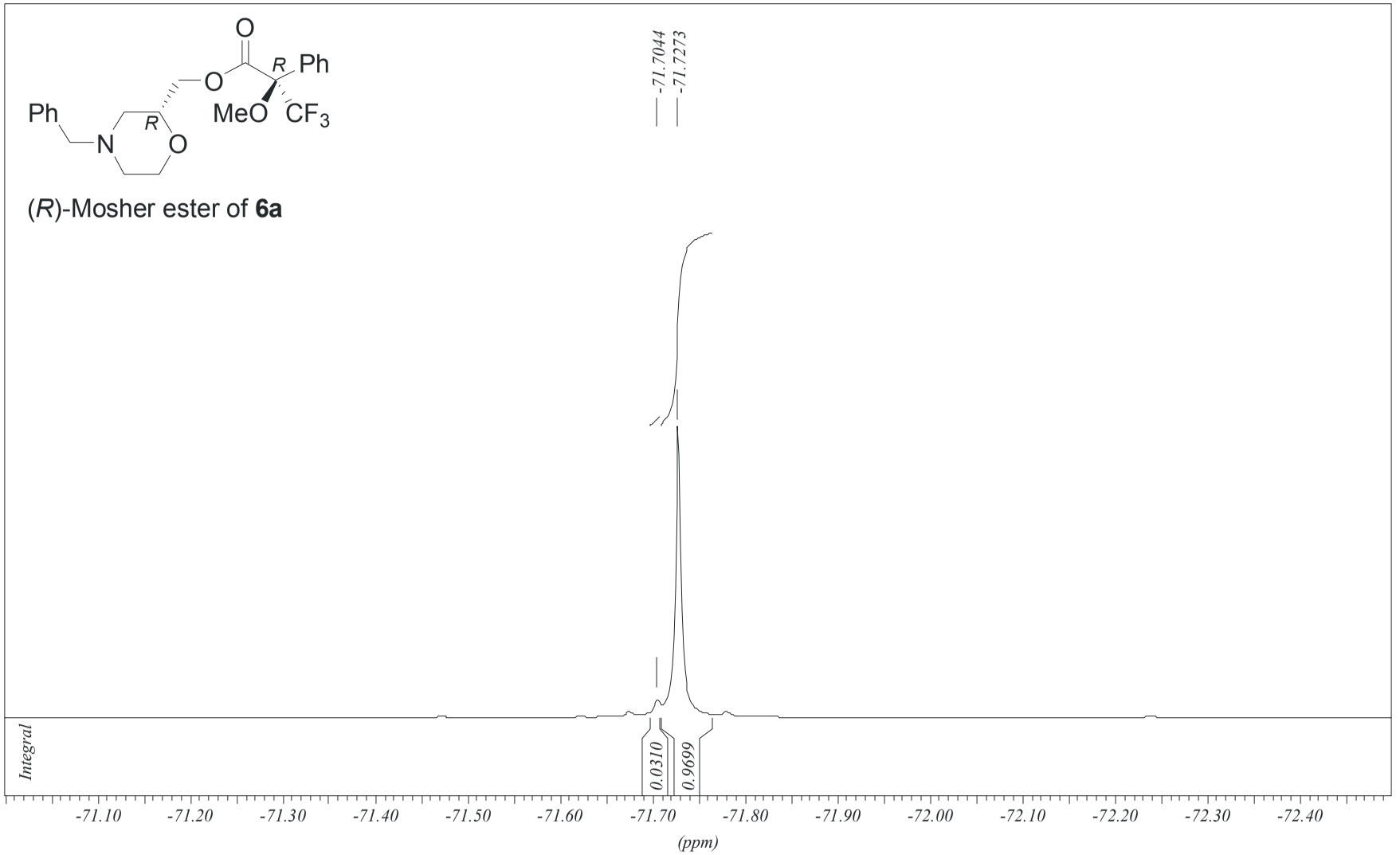

OH
Ph
N
O
6g
143.5843
138.0417
135.2703
128.8621
127.4146
127.3419
121.9302
118.1988
116.6058
112.6779
73.9086
63.5944
55.0840
48.4140
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)


OH
Ph
N
O
Ph
6k
1690.56
1683.10
1678.81
1671.48
1619.32
1609.97
1607.57
1598.22
1409.25
1405.46
1399.91
1396.12
1170.52
1157.13
1139.45
1136.79
1127.32
1124.67
1004.54
1000.50
992.42
988.37
Integral
1.9792
2.0196
3.0207
2.9770
0.9999
1.0131
0.9988
2.9745
0.9924
0.8742
1.0139
0.9948
0.9661
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
(ppm)

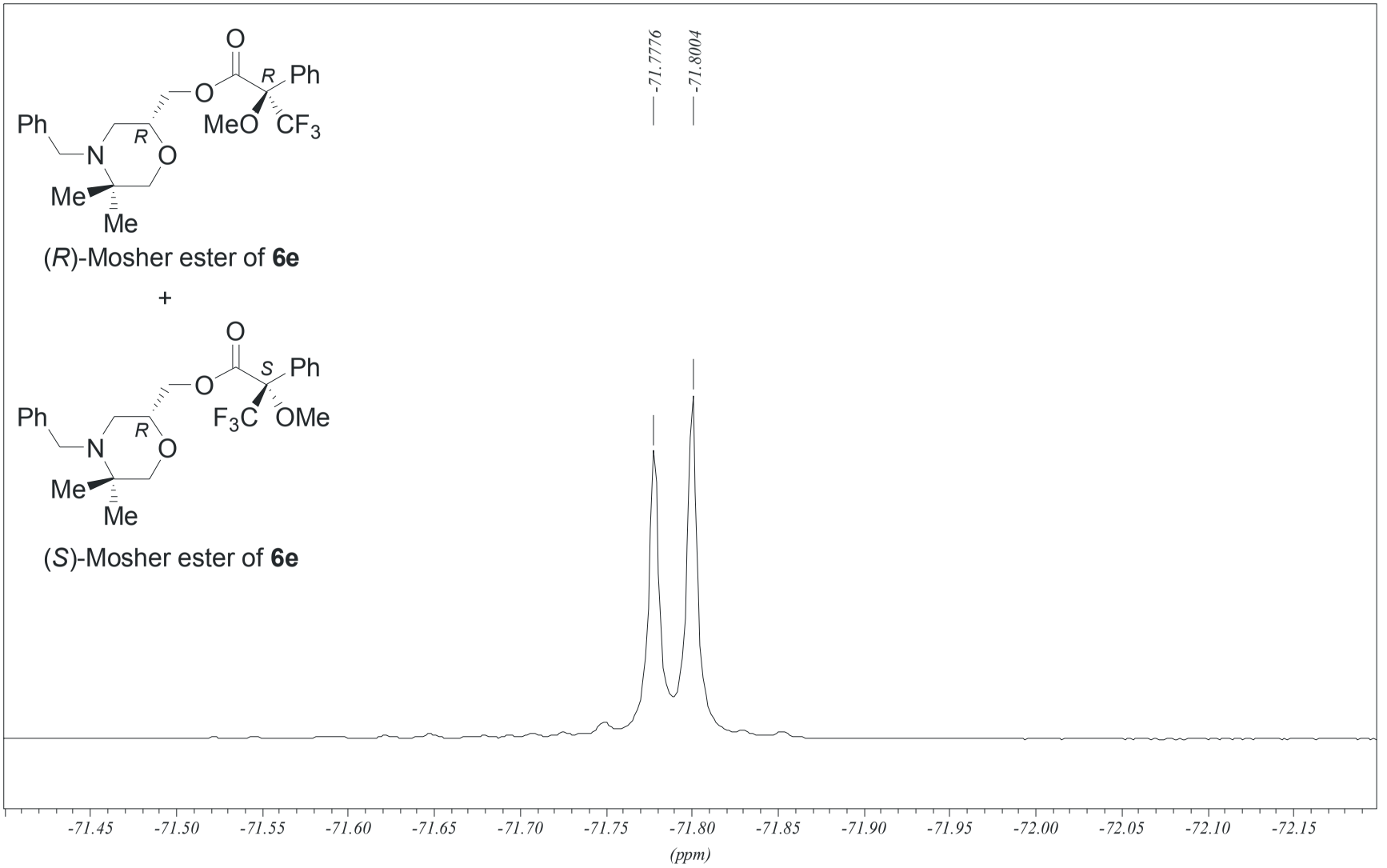

OH
Ph
N
O
Ph
6k
138.9628
138.0826
128.8116
128.7620
128.5372
128.5182
128.0952
127.2874
73.4066
68.3539
66.7916
63.7165
59.2887
51.6753
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

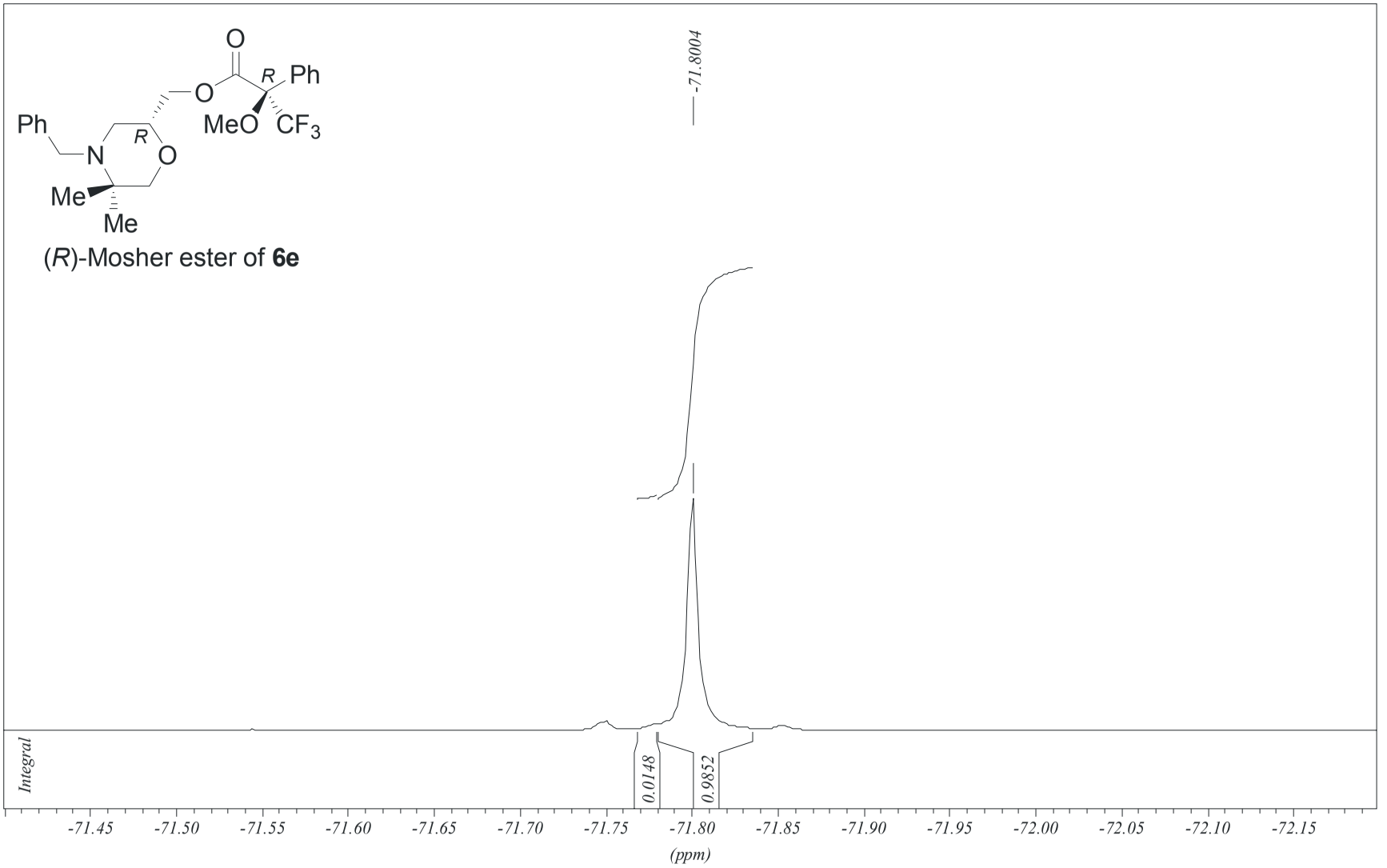


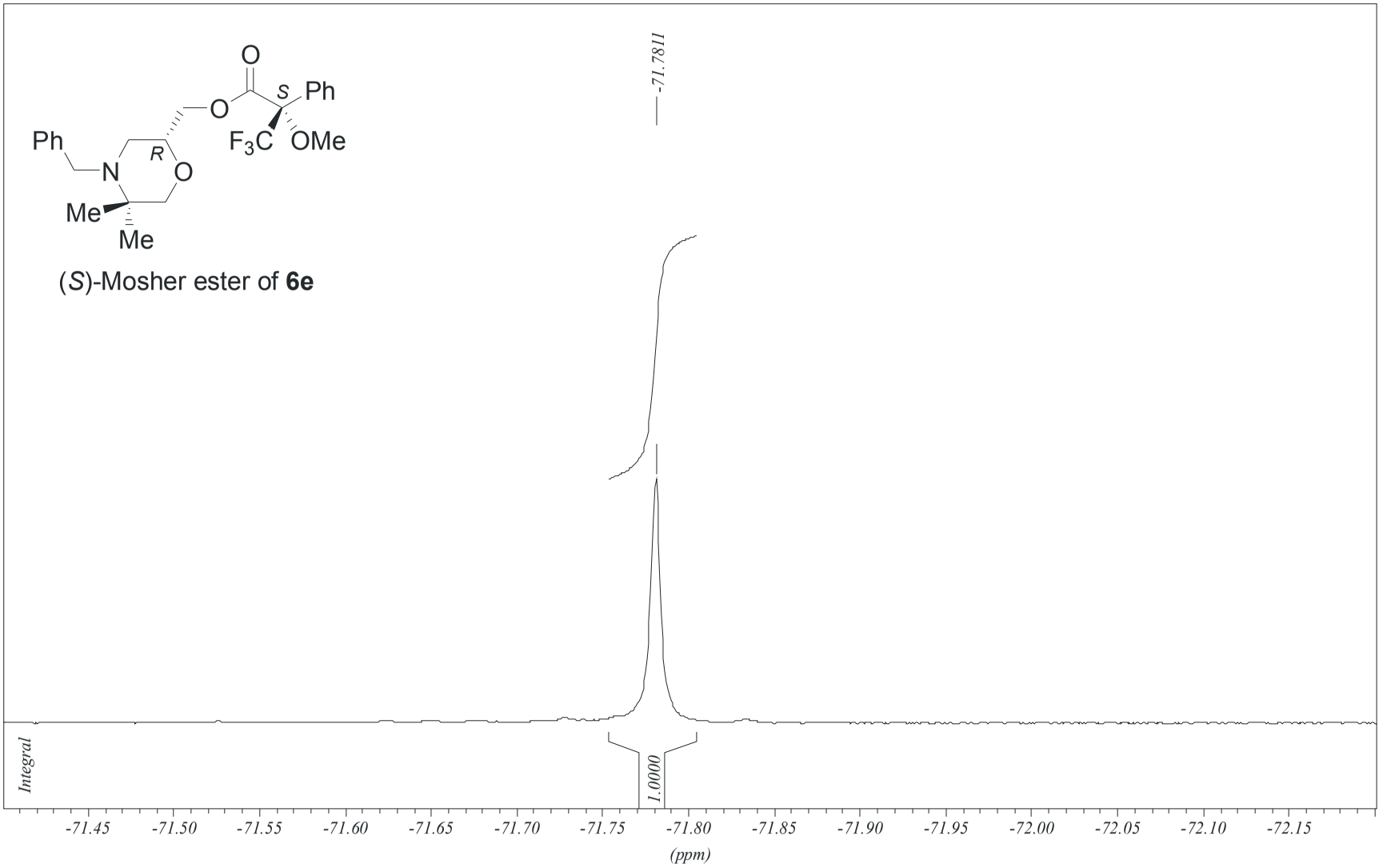
(*R*)-Mosher ester of *rac*-**6a**

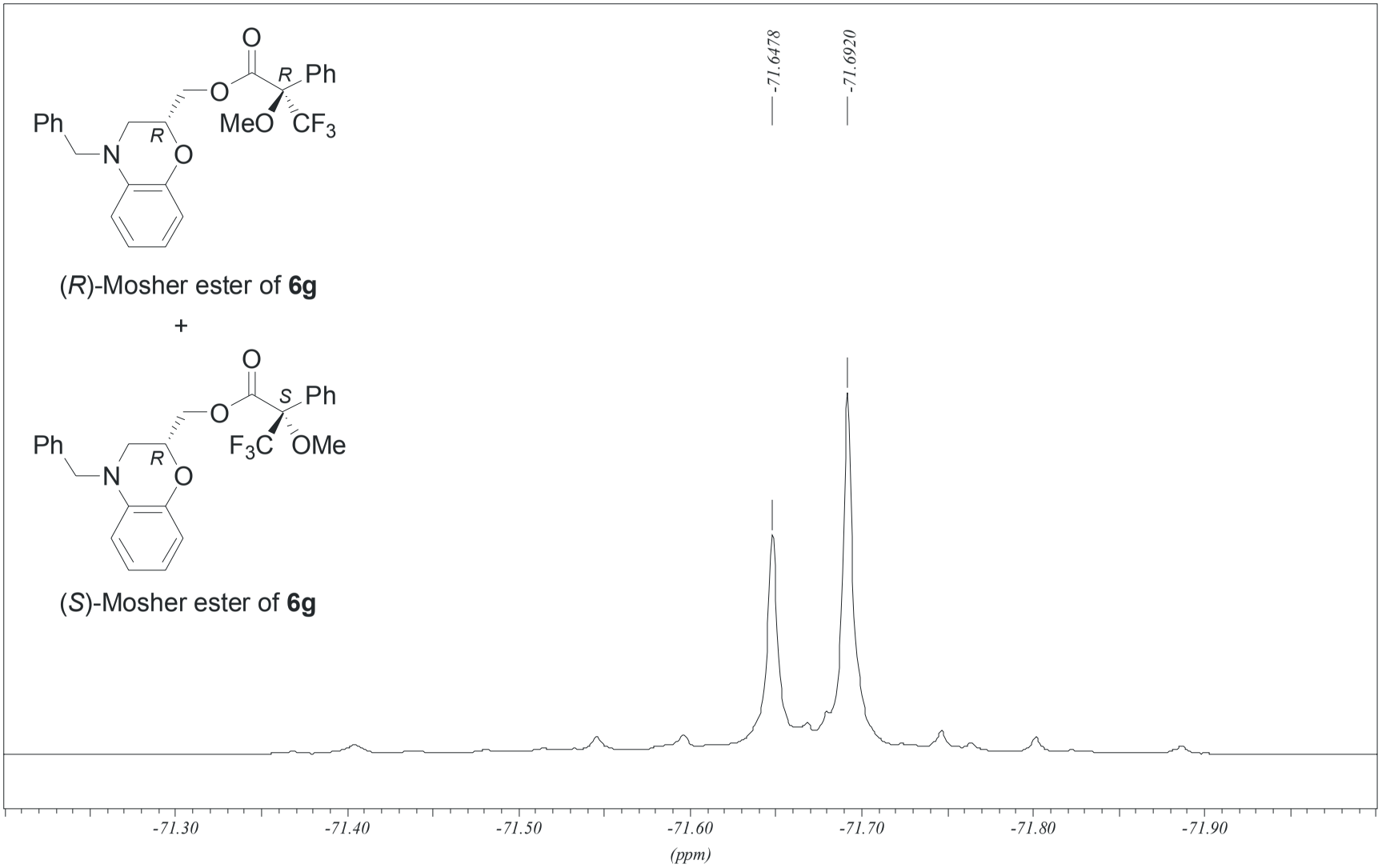


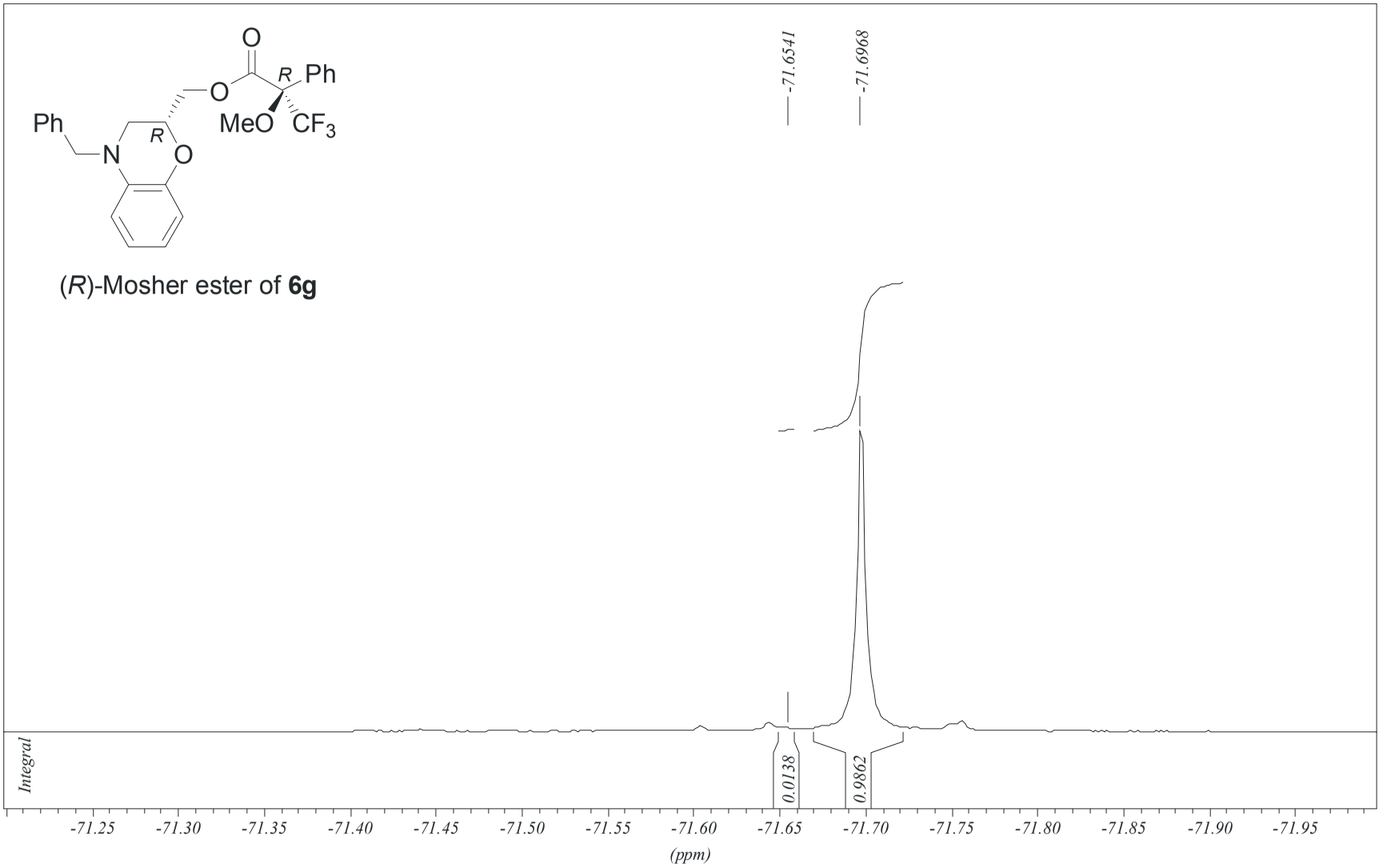
(*R*)-Mosher ester of **6a**

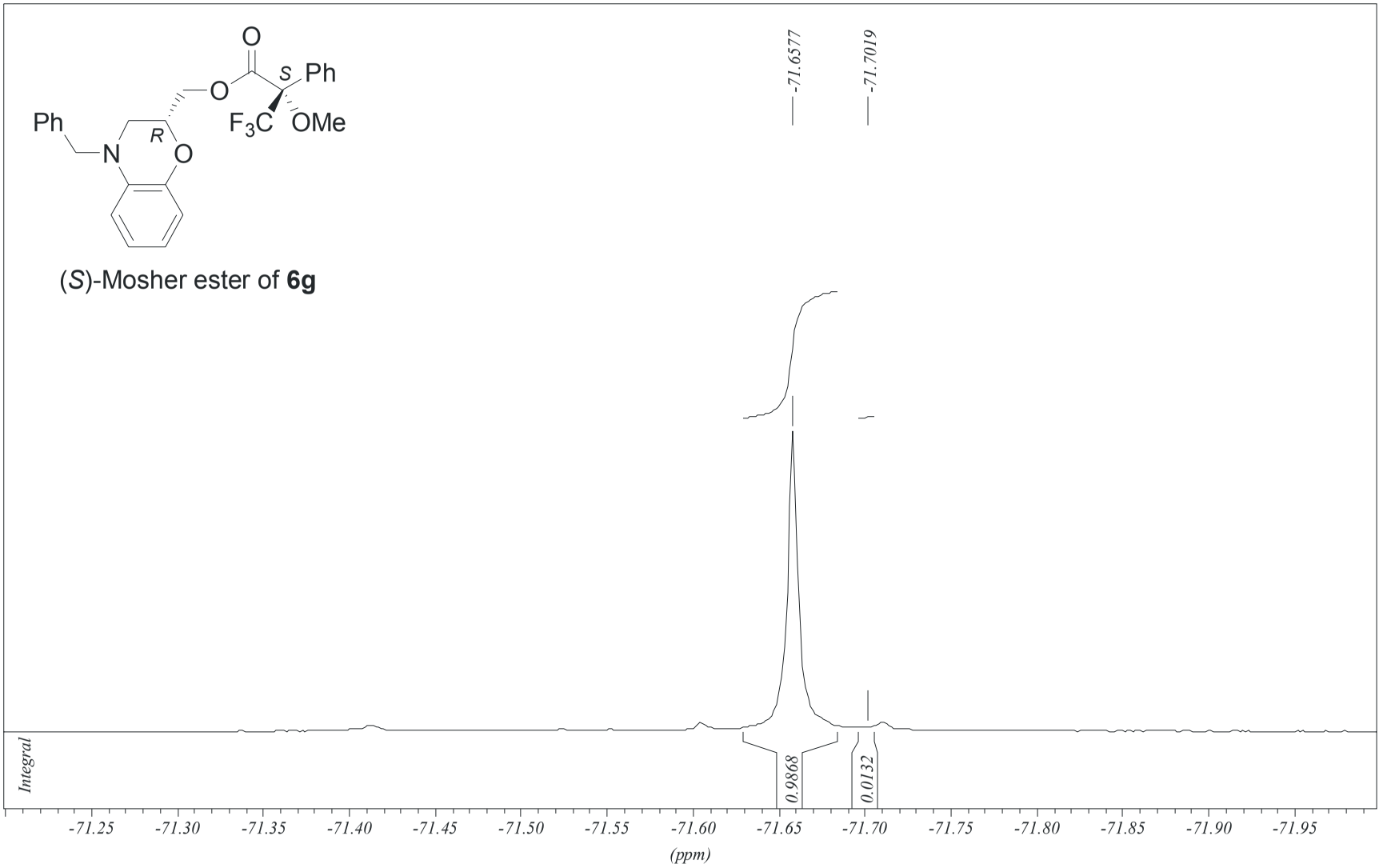

-71.7776
-71.8004
(R)-Mosher ester of 6e
+
(S)-Mosher ester of 6e
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
-71.85
-71.90
-71.95
-72.00
-72.05
-72.10
-72.15
(ppm)


O
R
Ph
O
MeO
CF3
Ph
R
N
O
Me
Me
(R)-Mosher ester of 6e
-71.8004
Integral
0.0148
0.9852
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
-71.85
-71.90
-71.95
-72.00
-72.05
-72.10
-72.15
(ppm)


-71.7811
Ph
R
N
O
Me
Me
O
O
S
Ph
F3C
OMe
(S)-Mosher ester of 6e
Integral
1.0000
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
-71.85
-71.90
-71.95
-72.00
-72.05
-72.10
-72.15
(ppm)


-71.6478
-71.6920
(R)-Mosher ester of 6g
+
(S)-Mosher ester of 6g
-71.30
-71.40
-71.50
-71.60
-71.70
-71.80
-71.90
(ppm)


O
R
Ph
O
MeO
CF3
Ph
R
N
O
(R)-Mosher ester of 6g
-71.6541
-71.6968
Integral
0.0138
0.9862
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
-71.85
-71.90
-71.95
(ppm)


O
S
Ph
O
Ph
R
N
O
F3C
OMe
(S)-Mosher ester of 6g
-71.6577
-71.7019
Integral
0.9868
0.0132
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
-71.85
-71.90
-71.95
(ppm)