Supporting Information for:

γ-Selective Directed Catalytic Asymmetric Hydroboration of 1,1-Disubstituted Alkenes.

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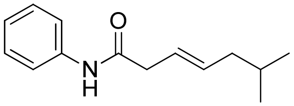
Department of Chemistry and Physics, Purdue University Calumet, Hammond, IN 46323-2094



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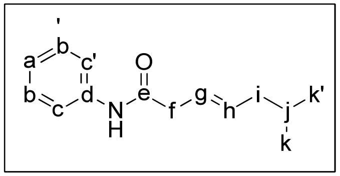
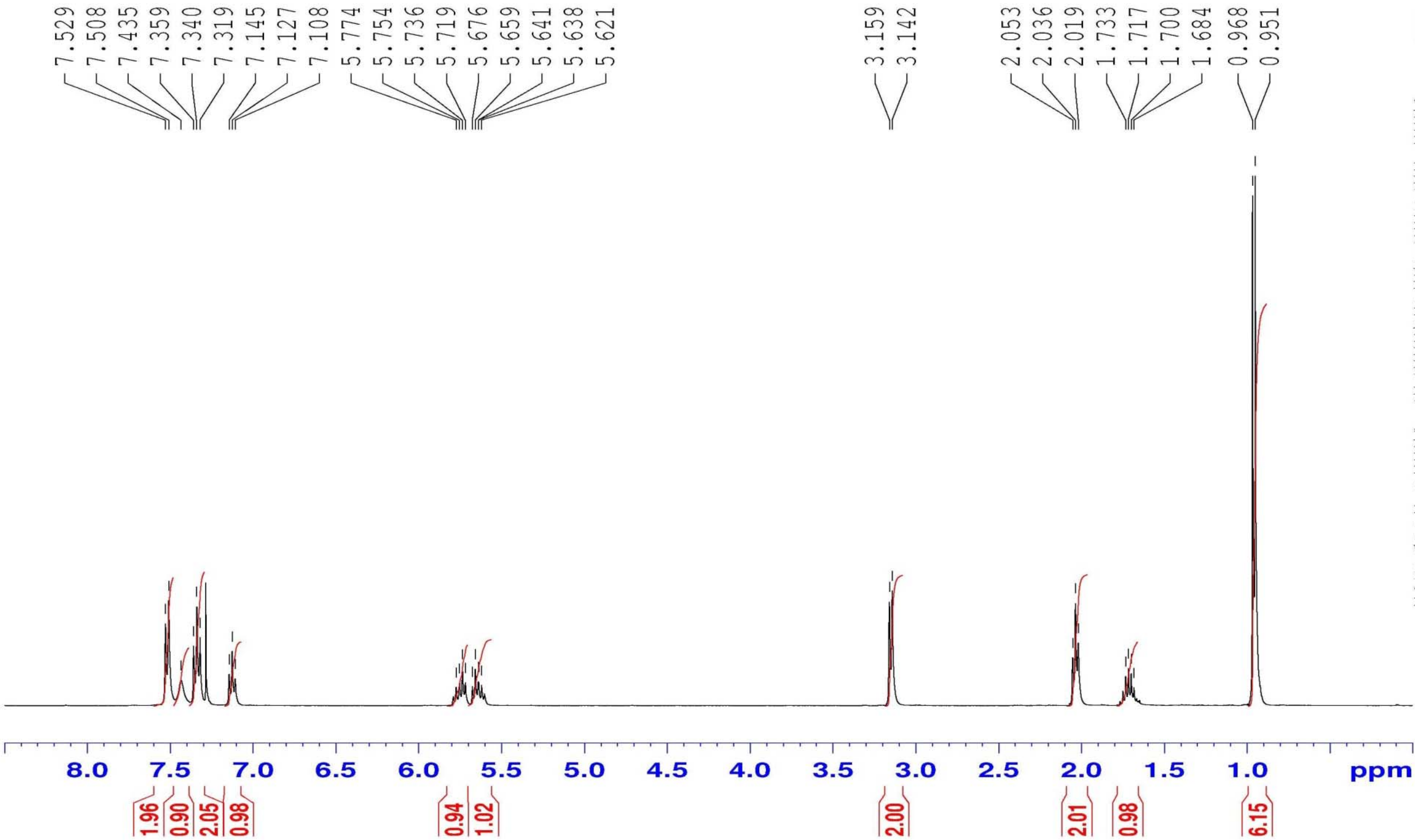


Following the amidation procedure with EDCI affords, after flash chromatography on silica gel (90:10 hexanes:ethyl acetate), the title compound (70%) as a light brown solid.

m.p.	87–88.5 °C
TLC analysis	R_f 0.5 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.52 (2H, d, J = 8.3 Hz, c,c’) 7.44 (1H, br s, NH), 7.34 (2H, t, J = 7.7 Hz, b,b’), 7.13 (1H, t, J = 7.4 Hz, a), 5.80-5.55 (2H, m, g,h), 3.15 (2H, d, J = 6.9 Hz, f), 2.04 (2H, t, J = 6.8 Hz, i), 1.80-1.60 (1H, m, j), 0.96 (6H, d, J = 6.6 Hz, k,k’).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 169.39 (e), 137.79 (d), 136.53 (g), 129.02 (b,b’), 124.31 (a), 123.41 (h), 119.61 (c,c’), 41.93 (f), 41.73 (i), 28.25 (j), 22.35 (k,k’).
IR (neat)	3244 (N-H stretch), 2952, 1654 (C=O stretch), 1595, 1544 (N-H bend), 1443, 1247 (C-N stretch), 1187, 967, 843, 756 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{14}H_{20}NO$ (M+H): 218.1545, found 218.1541 *m/z*.

^{1}H NMR of 1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1.700
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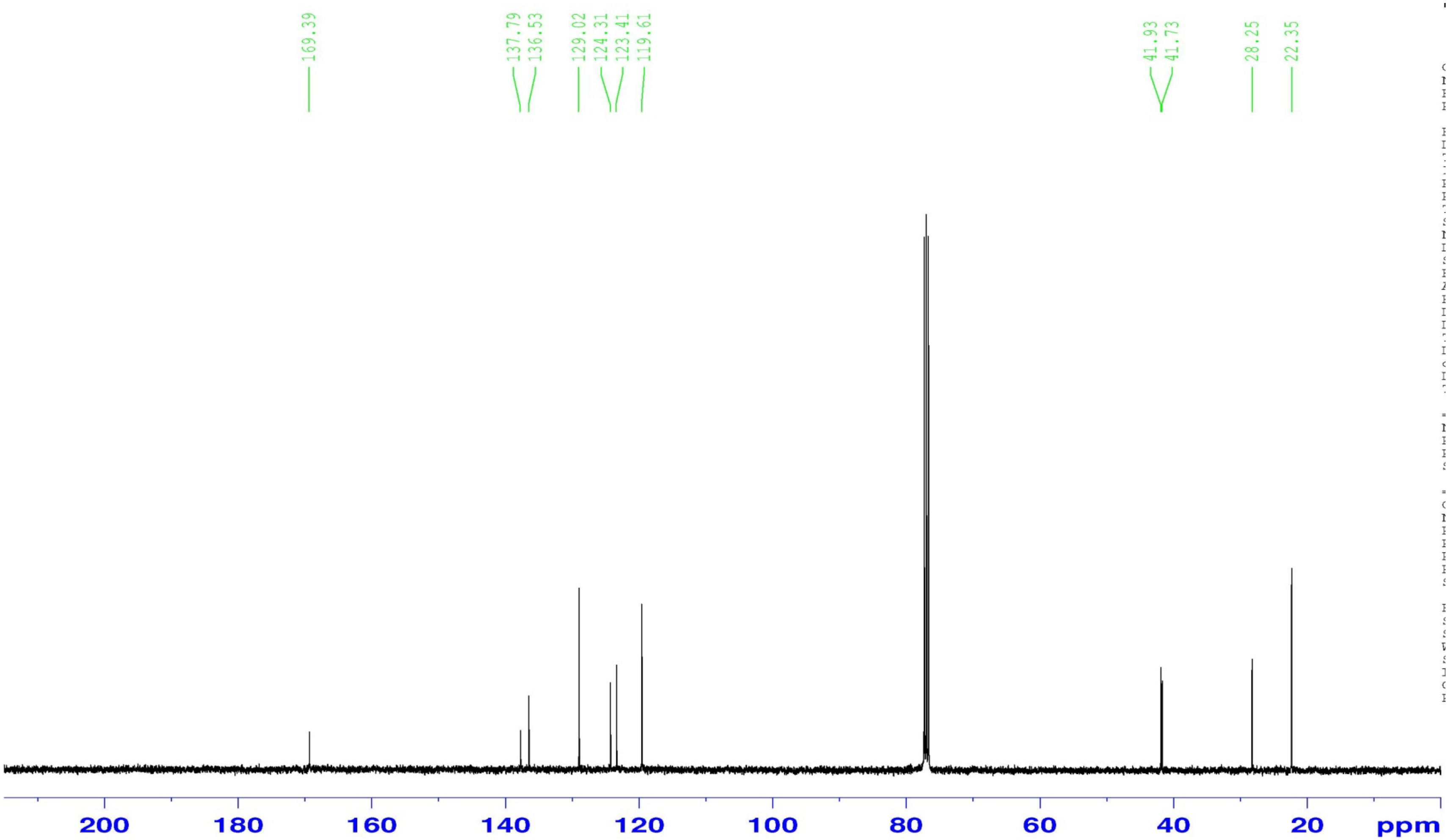
^{13}C NMR of 1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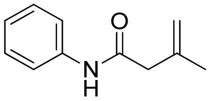
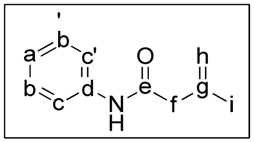


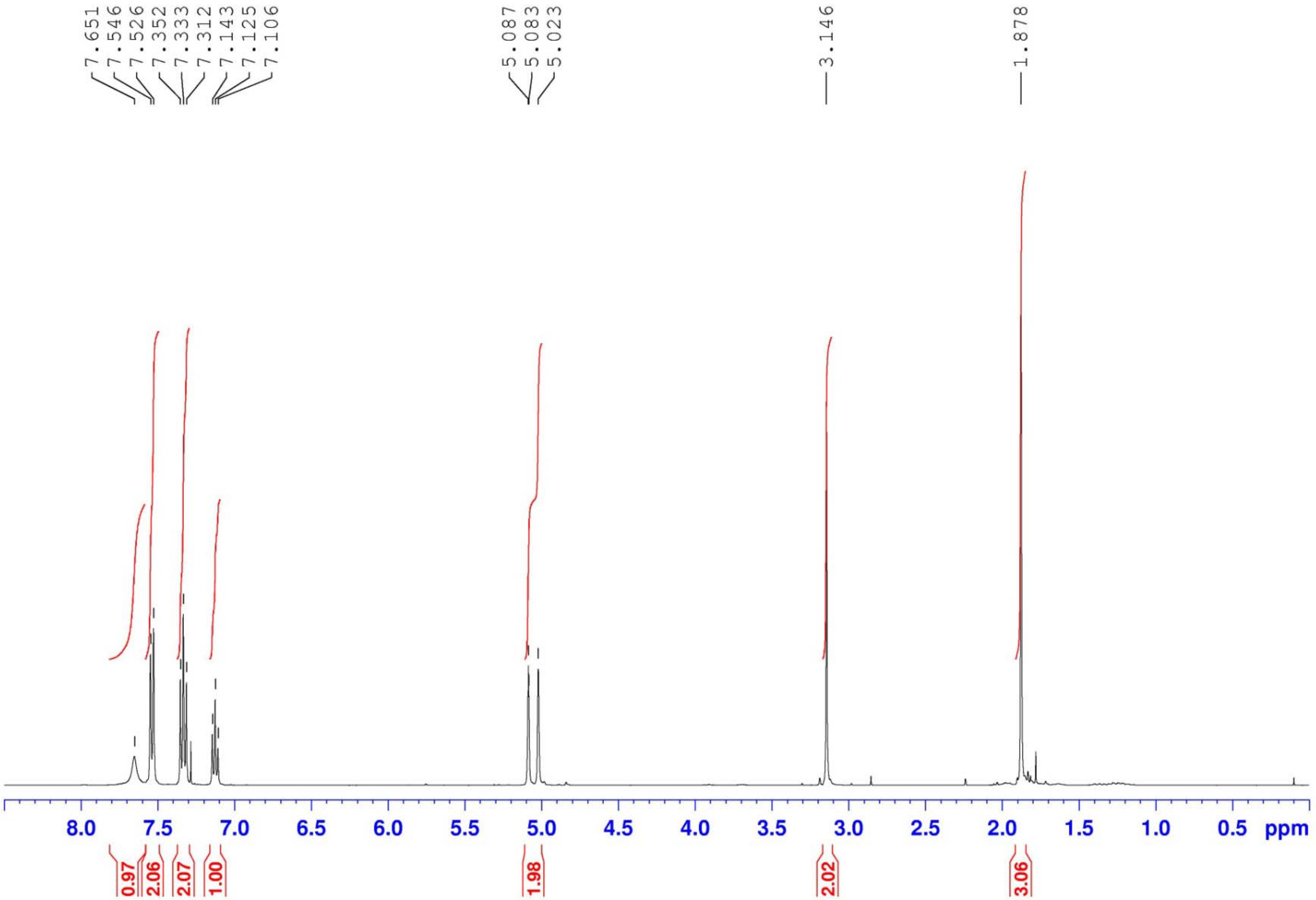
7a



Following the general amidation procedure with DCC affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (65%) as a white solid.

m.p.	97.5–99.5 °C
TLC analysis	R_f 0.3 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.65 (1H, br s, NH), 7.53 (2H, d, J = 8.0 Hz, c,c’), 7.33 (2H, t, J = 7.6 Hz, b,b’), 7.13 (1H, t, J = 7.2 Hz, a), 5.09 and 5.02 (2H, s’s, h), 3.15 (2H, s, f), 1.88 (3H, s, i).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 168.58 (e), 140.35 (d), 137.80 (g), 128.98 (b,b’), 124.36 (a), 119.79 (c,c’), 116.09 (h), 47.41 (f), 22.46 (i).
IR (neat)	3291 (N-H stretch), 3060, 2953, 2921, 2865, 1657 (C=O stretch), 1638, 1595, 1525 (N-H bend), 1440, 1307, 1251 (C-N stretch), 1162, 869, 738, 688, 617 cm^{-1}.

^{1}H NMR of 7a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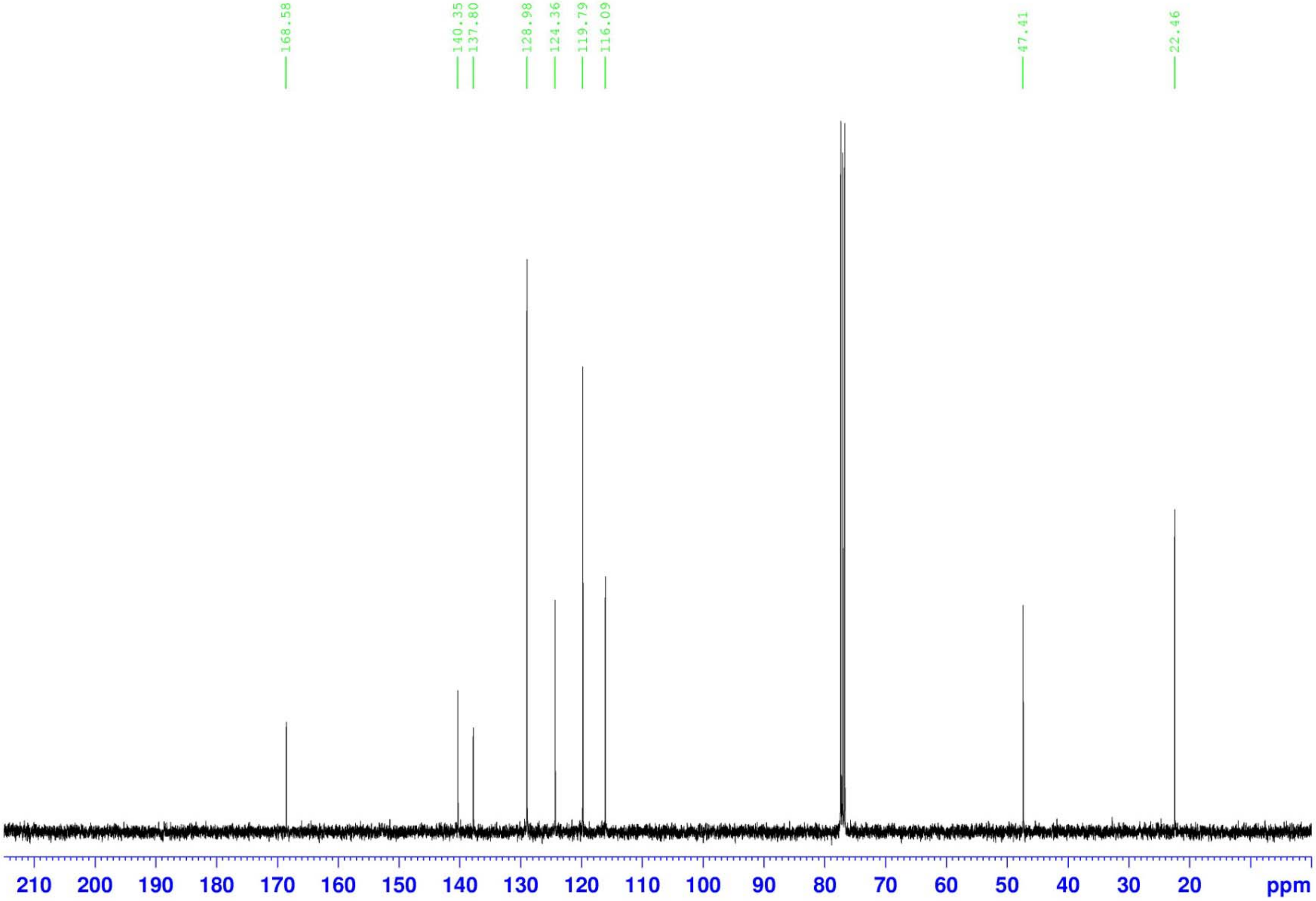
3.06

^{13}C NMR of 7a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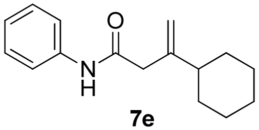

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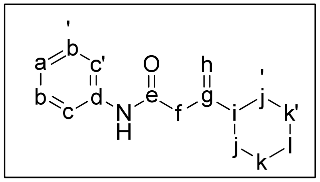
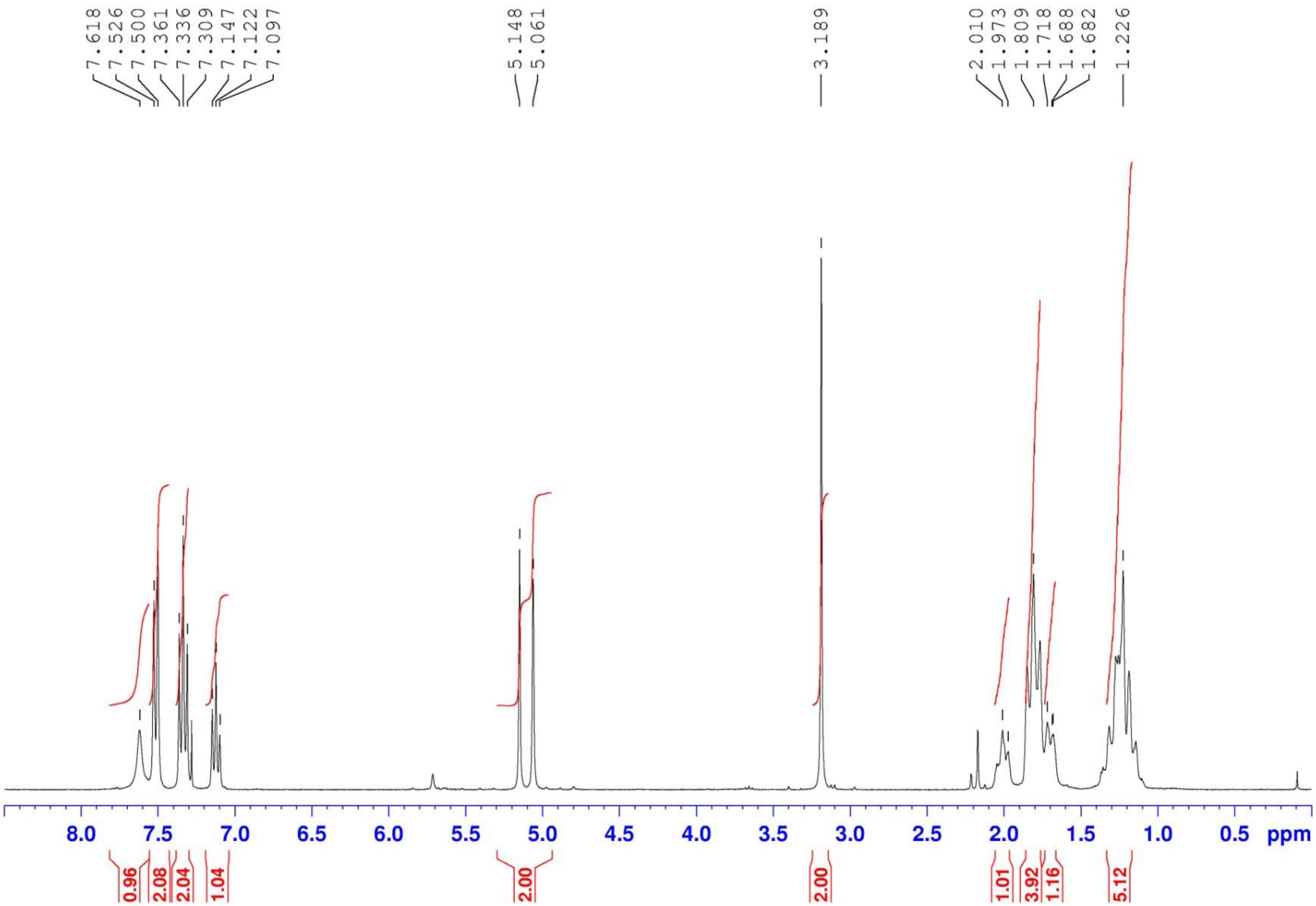


Following the general carbonylation-hydrolysis-amidation procedure affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (39%, 3 steps) as a white solid.

m.p.	81–83 °C
TLC analysis	R_f 0.4 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.62 (1H, br s, NH), 7.51 (2H, d, J = 7.8 Hz, c,c'), 7.34 (2H, t, J = 8.1 Hz, b,b'), 7.12 (1H, t, J = 7.5 Hz, a), 5.15 and 5.06 (2H, s's, h), 3.19 (2H, s, f), 2.05–1.95 (1H, m, i), 1.90–1.75 (4H, m, k,k',l,j), 1.75–1.65 (1H, m, j'), 1.30–1.10 (5H, m, j,j',k,k',l).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 168.85 (e), 150.39 (d), 137.72 (g), 129.00 (b,b'), 124.31 (a), 119.65 (c,c'), 113.79 (h), 44.06 (i), 44.42 (f), 32.18 (j,j'), 26.48 (k,k'), 26.09 (l).
IR (neat)	3330 (N-H stretch), 2921, 2848, 1665 (C=O stretch), 1596, 1514 (N-H bend), 1436, 1346, 1245 (C-N stretch), 1167, 956, 905, 749, 691, 586 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{16}H_{21}NaNO$ (M+Na): 266.1521, found 266.1526 *m/z*..

^{1}H NMR of 7e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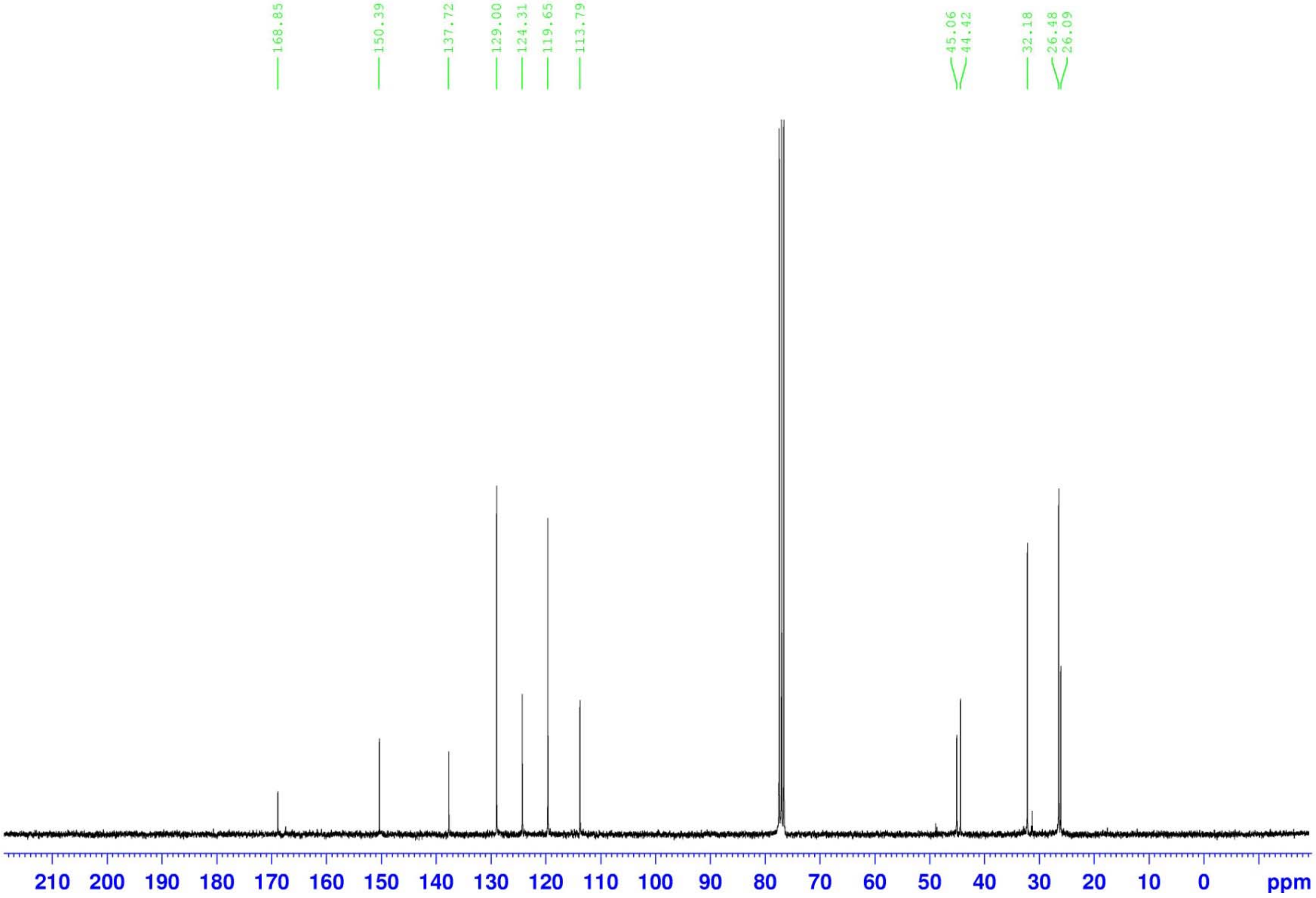
^{13}C NMR of 7e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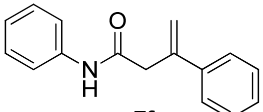
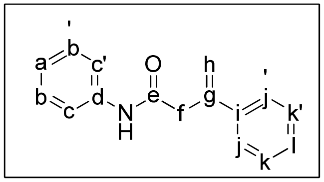


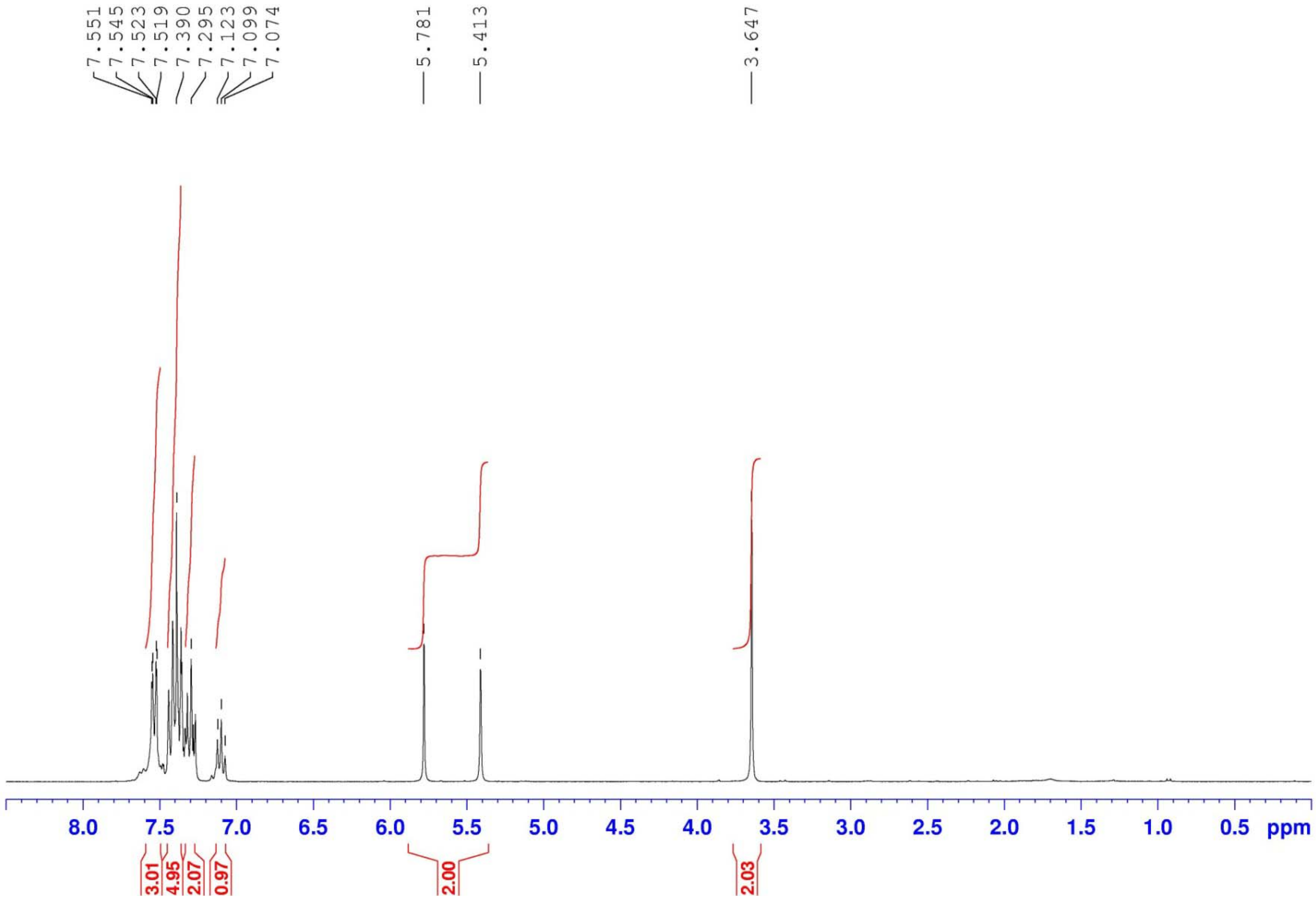
7f



Following the general carbonylation-hydrolysis-amidation procedure affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (18%, 3 steps) as a white solid.

m.p.	90.5–93.5 °C
TLC analysis	R_f 0.4 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.60–7.50 (3H, m, j,j',NH), 7.45–7.35 (5H, m, c,c',k,k',l), 7.35–7.25 (2H, m, b,b'), 7.10 (1H, t, *J* = 7.5 Hz, a), 5.78 and 5.41 (2H, s's, h), 3.65 (2H, s, f).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 168.35 (e), 142.08 (d), 138.76 (i), 137.61 (g), 128.94 (b,b'), 128.82 (j,j'), 128.47 (l), 125.78 (k,k'), 124.43 (a), 119.84 (c,c'), 117.44 (h), 45.14 (f).
IR (neat)	3248 (N-H stretch), 3192, 3135, 3085, 2929, 1804, 1656 (C=O stretch), 1597, 1554 (N-H bend), 1484, 1441, 1338, 1232 (C-N stretch), 1162. 896, 770, 752, 688 cm^{-1}.

^{1}H NMR of 7f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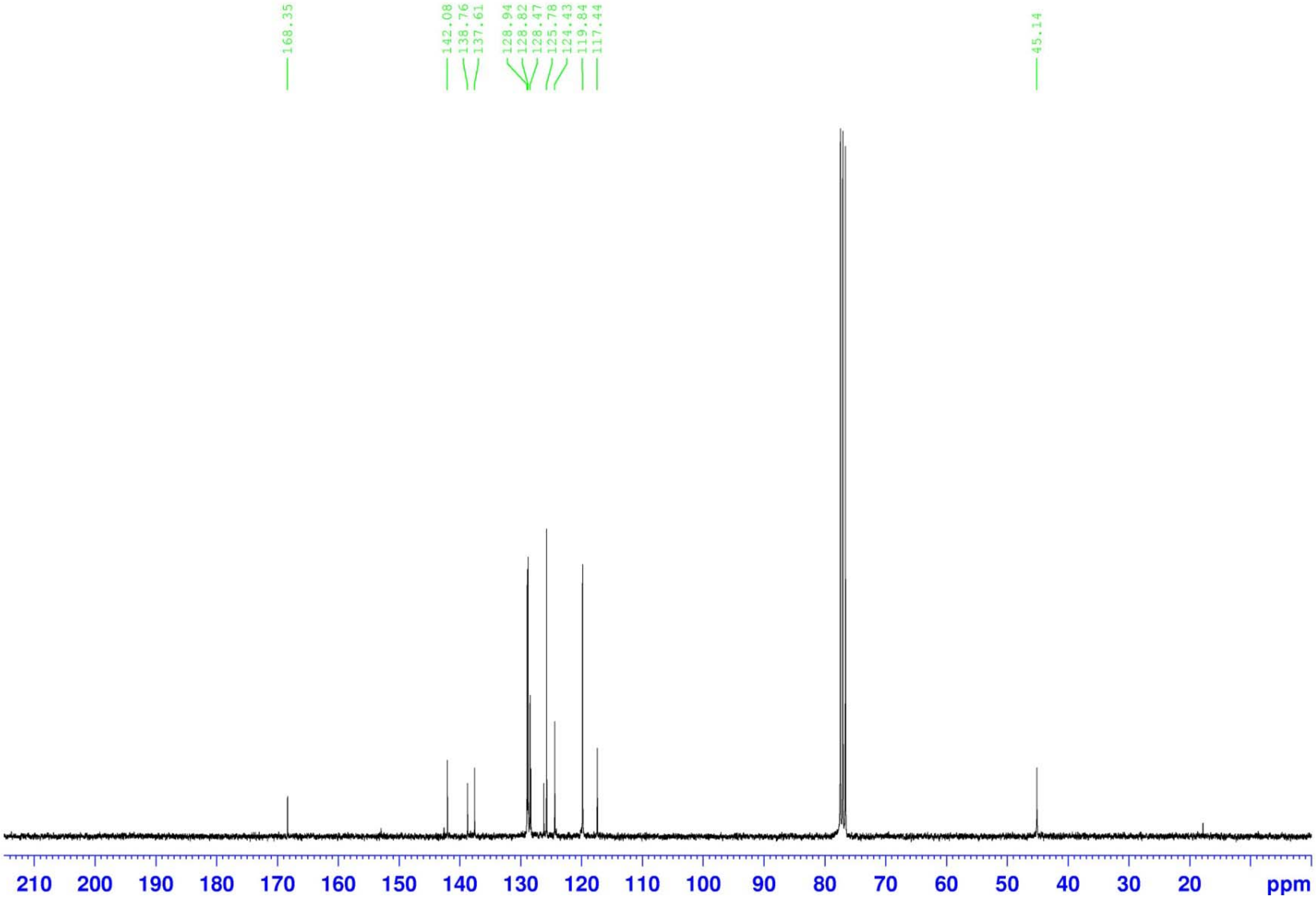
^{13}C NMR of 7f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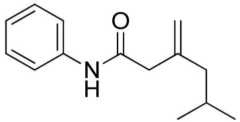

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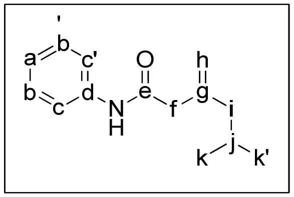
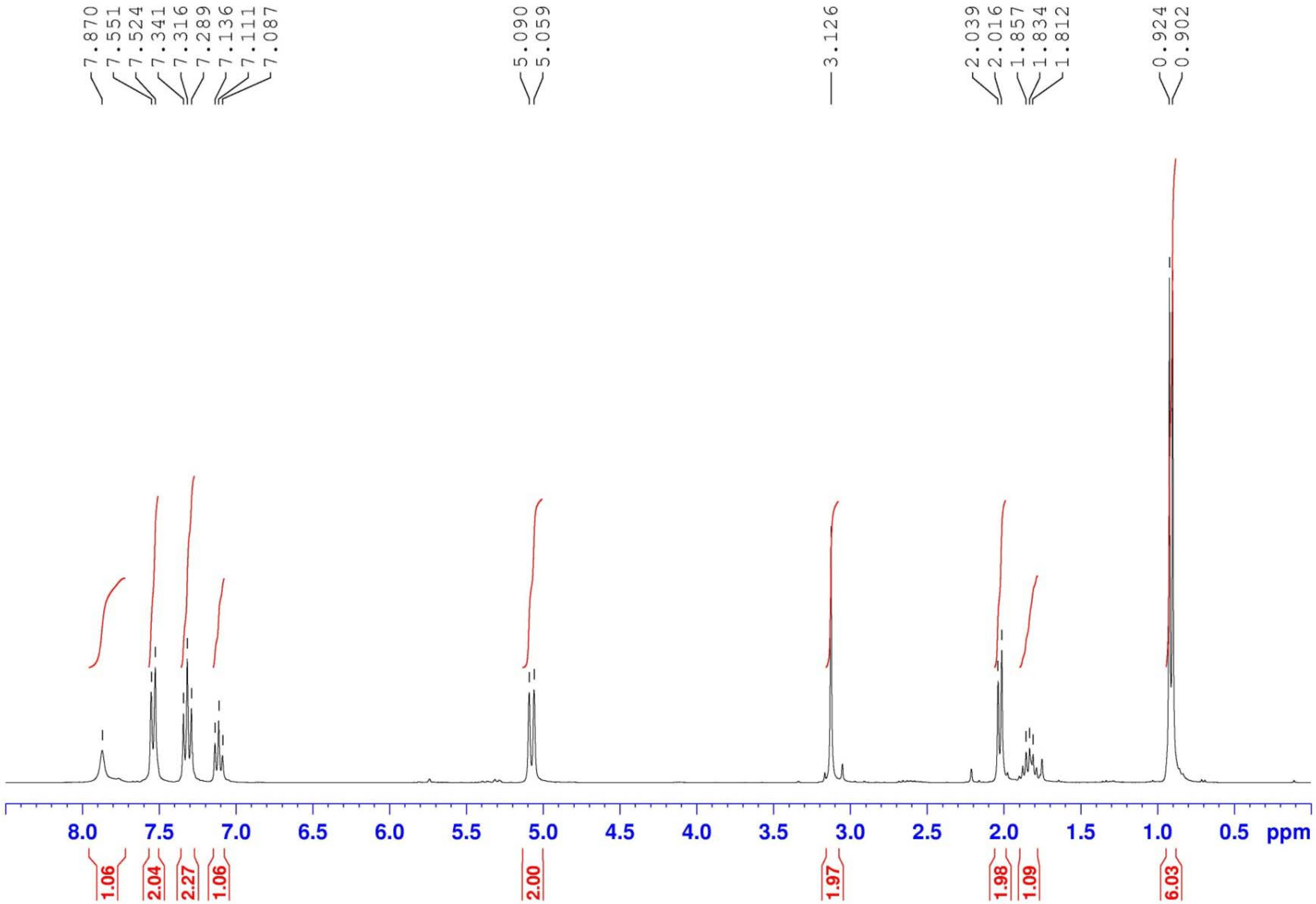


Following the general procedure for hydrolysis of *tert*-butyl esters proceeded by amidation affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (63%, 2 steps) as a white solid.

m.p.	91–92.5 °C
TLC analysis	R_f 0.4 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.87 (1H, br s, NH), 7.53 (2H, d, J = 8.1 Hz, c,c’), 7.32 (2H, t, J = 8.1 Hz, b,b’), 7.11 (1H, t, J = 7.5 Hz, a), 5.09 and 5.06 (2H, s’s, h), 3.13 (2H, s, f), 2.02 (2H, d, J = 6.9 Hz, i), 1.90–1.75 (1H, m, j), 0.91 (6H, d, J = 6.6 Hz, k,k’).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 168.92 (e), 143.29(d), 137.90 (g), 128.94 (b,b’), 124.30 (a), 119.84 (c,c’), 116.17 (h), 45.67 (f), 45.63 (j), 25.96 (i), 22.41 (k,k’).
IR (neat)	3290 (N-H stretch), 2953, 2921, 2865, 1657 (C=O stretch), 1638, 1595, 1530 (N-H bend), 1440, 1393, 1307, 1295, 1251 (C-N stretch), 1223, 1162, 1120, 996, 869, 738, 668, 617 cm^{-1}.
HRMS (CI)	Calcd. for $C_{14}H_{20}NO$ (M+H): 218.1545, found 218.1539 *m/z*.

^{1}H NMR of 3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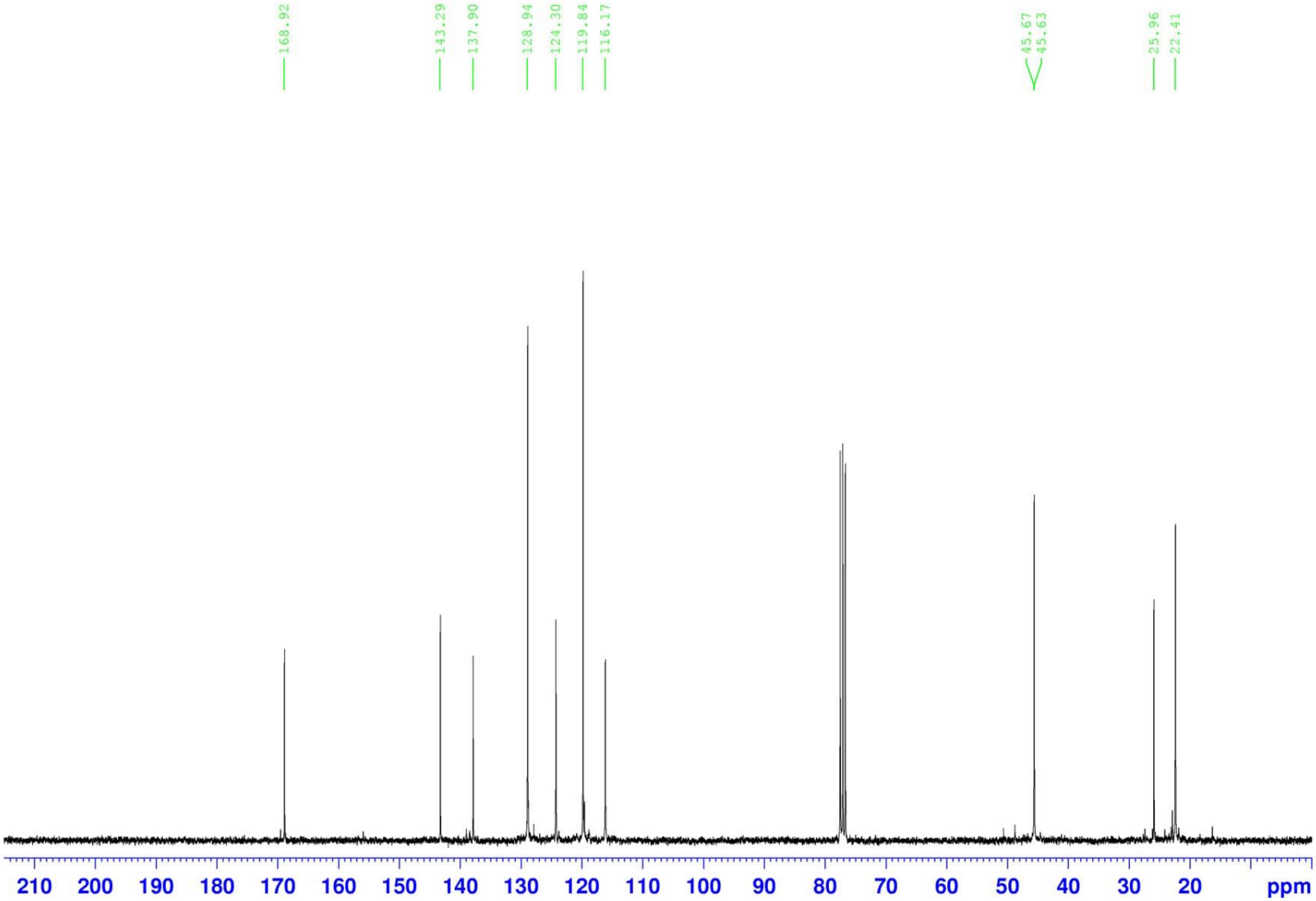
^{13}C NMR of 3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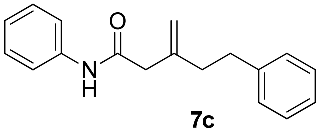
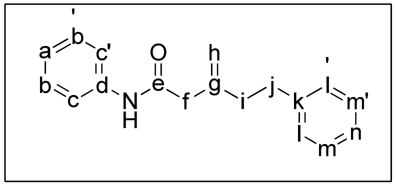


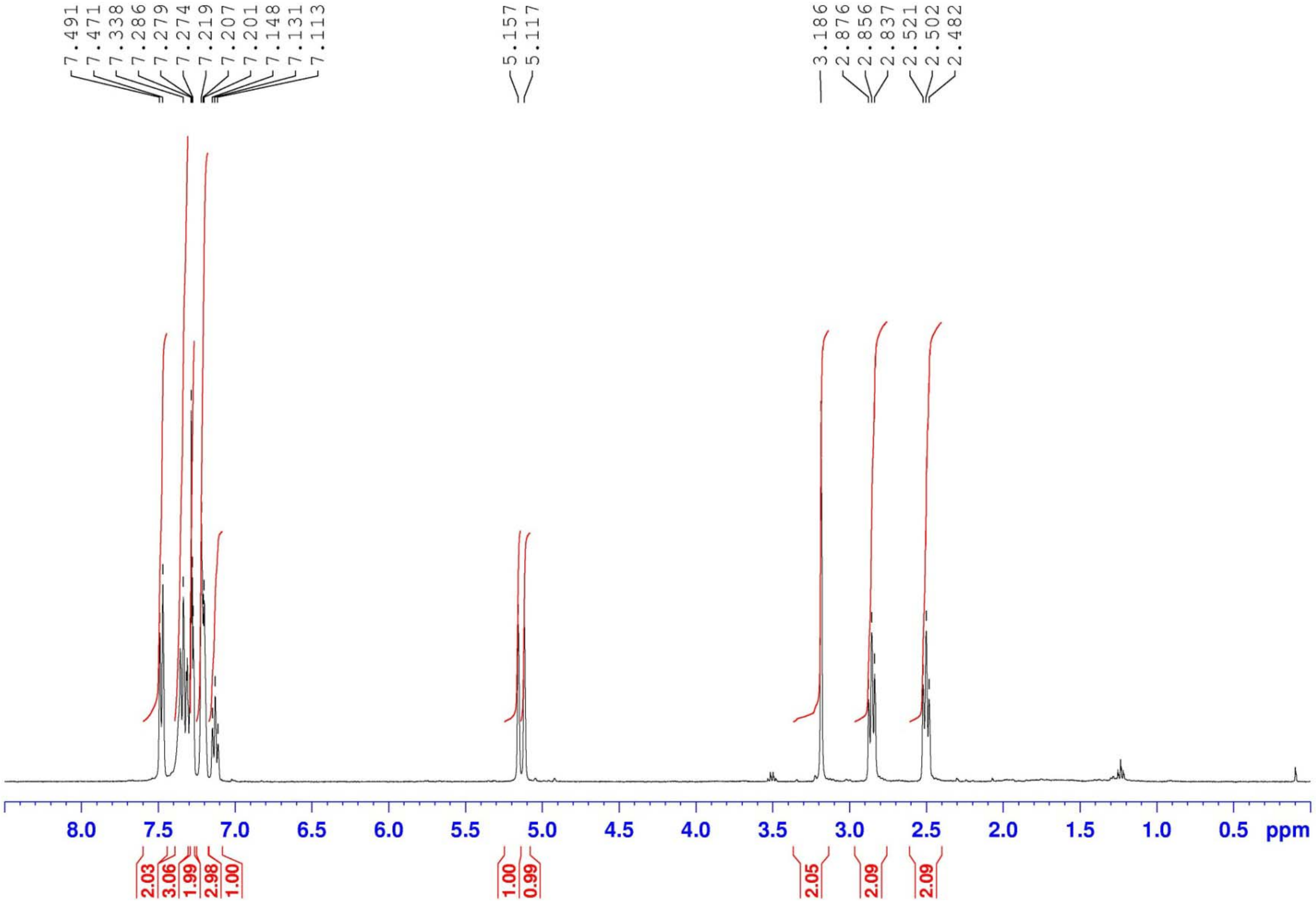
7c



Following the general procedure for hydrolysis of *tert*-butyl esters proceeded by amidation affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (61%, 2 steps) as a white solid.

m.p.	79–80 °C
TLC analysis	R_f 0.4 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.48 (2H, d, *J* = 8.0 Hz, l,l’), 7.40–7.30 (3H, m, c,c’, NH), 7.30–7.25 (2H, m, b,b’), 7.25–7.20 (3H, m, m,m’,n), 7.14 (1H, t, *J* = 6.8 Hz, a), 5.16 and 5.12 (2H, s’s, h), 3.19 (2H, s, f), 2.86 (2H, t, *J* = 8.0 Hz, j), 2.50 (2H, t, *J* = 8.4 Hz, i).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 168.31 (e), 143.69 (d), 141.21 (k), 137.66 (g), 128.99 (b,b’), 128.44 (l,l’), 128.35 (m,m’), 126.06 (n), 124.38 (a), 119.69 (c,c’), 115.75 (h), 46.25 (f), 37.44 (i), 33.91 (j).
IR (neat)	3237 (N-H stretch), 3185, 3061, 3025, 1652 (C=O stretch), 1596, 1541 (N-H bend), 1469, 1443, 1398, 1346, 1247 (C-N stretch), 1193, 961, 897, 747, 694, 616 cm^{-1}.
HRMS (EI)	Calcd. for $C_{19}H_{21}NO$: 279.1623, found 279.1649 *m/z*.

^{1}H NMR of 7c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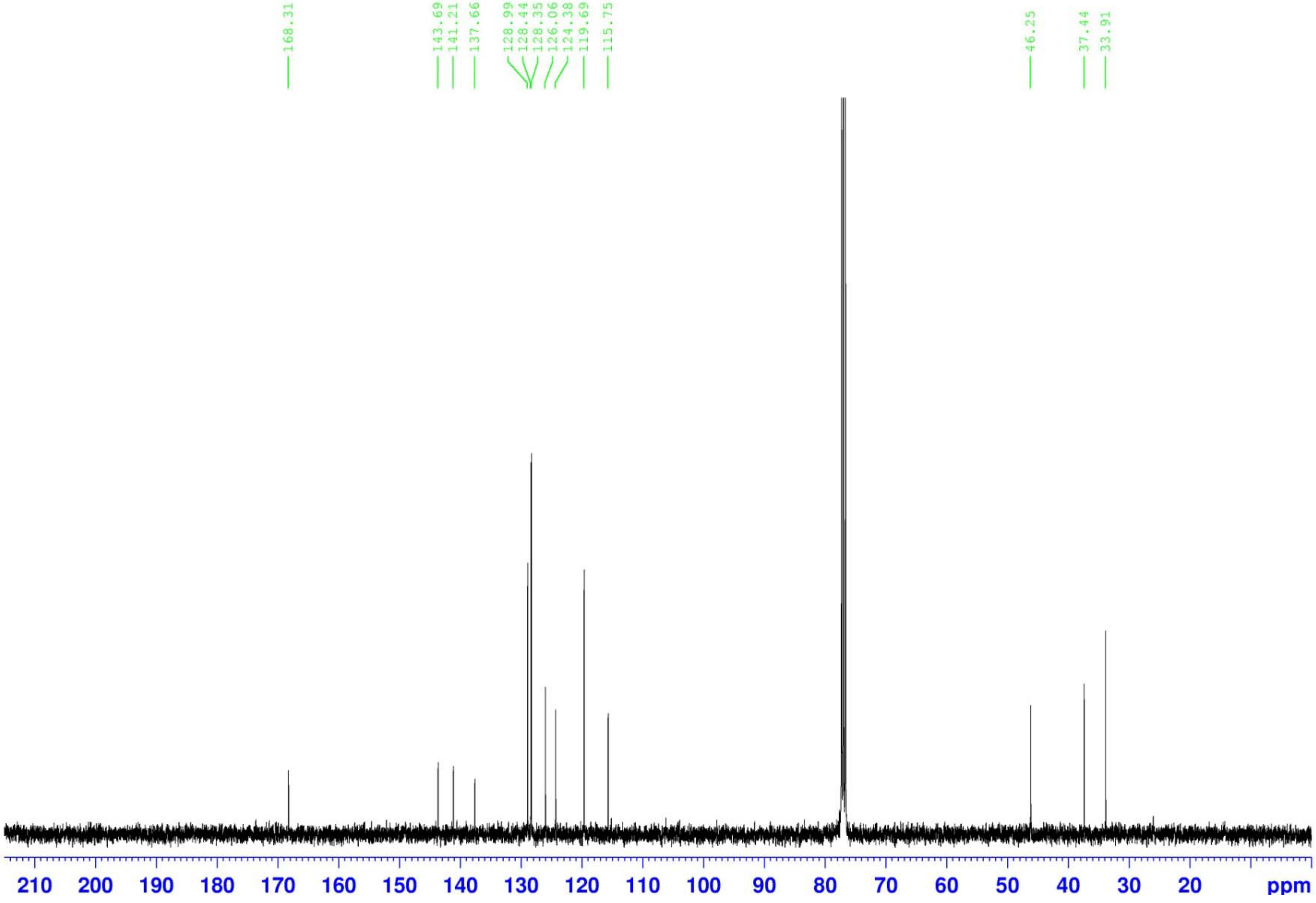
2.09

^{13}C NMR of 7c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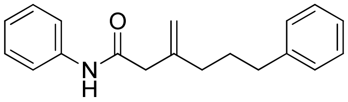

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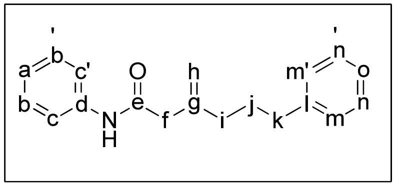
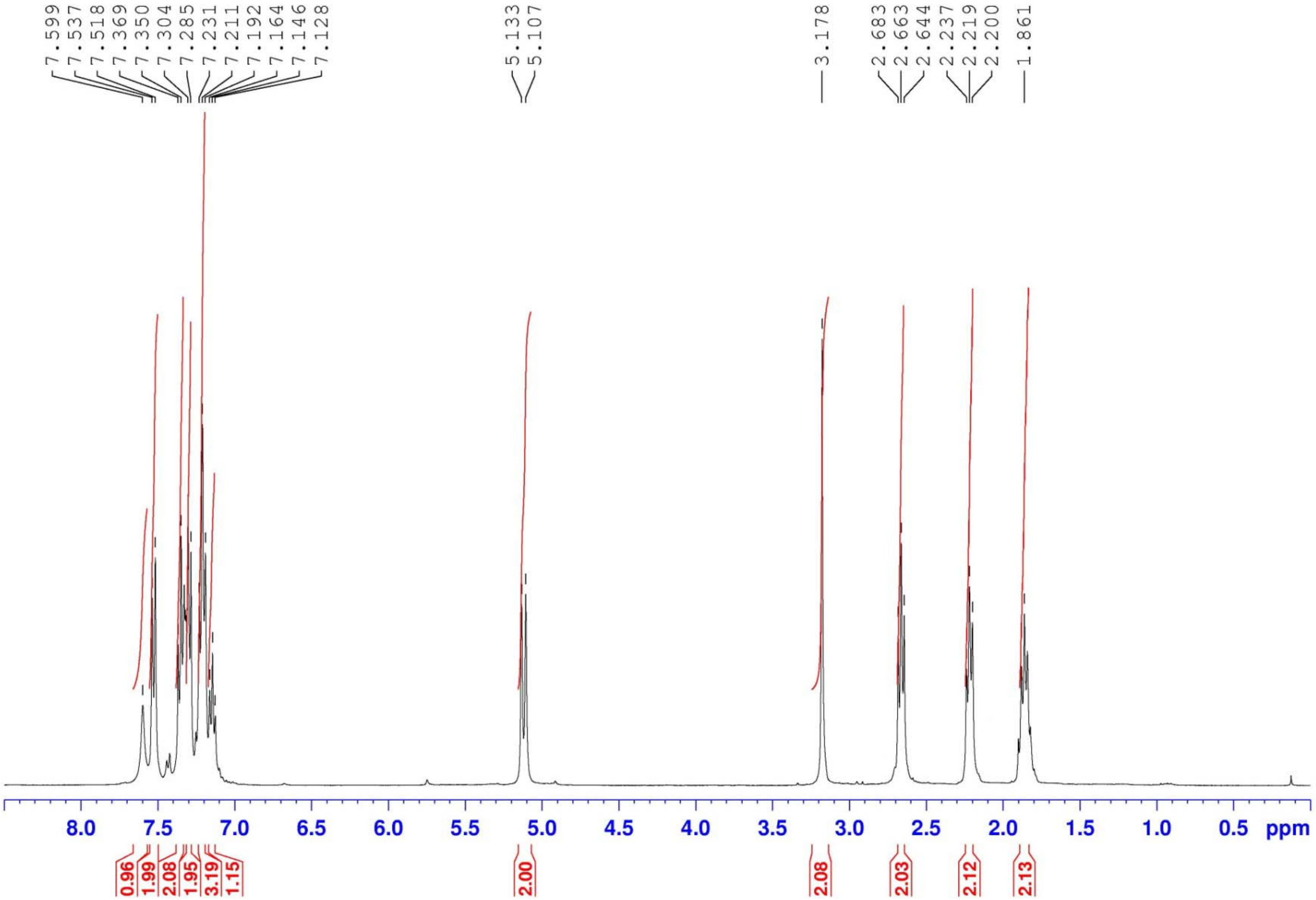


Following the general procedure for hydrolysis of *tert*-butyl esters proceeded by amidation affords, after flash chromatography on silica gel (90–80:10–20 hexanes:ethyl acetate), the title compound (57%, 2 steps) as a white solid.

m.p.	51–52.5 °C
TLC analysis	R_f 0.5 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.60 (1H, br s, NH), 7.52 (2H, d, *J* = 7.9 Hz, m,m’), 7.40–7.35 (2H, m, c,c’), 7.35–7.25 (2H, m, b,b’), 7.25–7.20 (3H, m, n,n’,o), 7.15 (1H, t, *J* = 7.3 Hz, a), 5.13 and 5.11 (2H, s’s, h), 3.18 (2H, s, f), 2.66 (2H, t, *J* = 7.7 Hz, k), 2.22 (2H, t, *J* = 7.4 Hz, i), 1.90–1.80 (2H, m, j).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 168.71 (e), 144.20 (d), 141.98 (l), 137.71 (g), 129.02 (b,b’), 128.44 (m,m’), 128.37 (n,n’), 125.87 (o), 124.43 (a), 119.83 (c,c’), 115.20 (h), 46.06 (f), 35.52 (i), 35.42 (k), 29.24 (j).
IR (neat)	3303 (N-H stretch), 3061, 3028, 2935, 1659 (C=O stretch), 1598, 1543 (N-H bend), 1497, 1441, 1334, 1245 (C-N stretch), 1155, 899, 749, 690 cm^{-1}.
HRMS (EI)	Calcd. for $C_{19}H_{21}NO$: 279.1623, found 279.1618 *m/z*.

^{1}H NMR of 7d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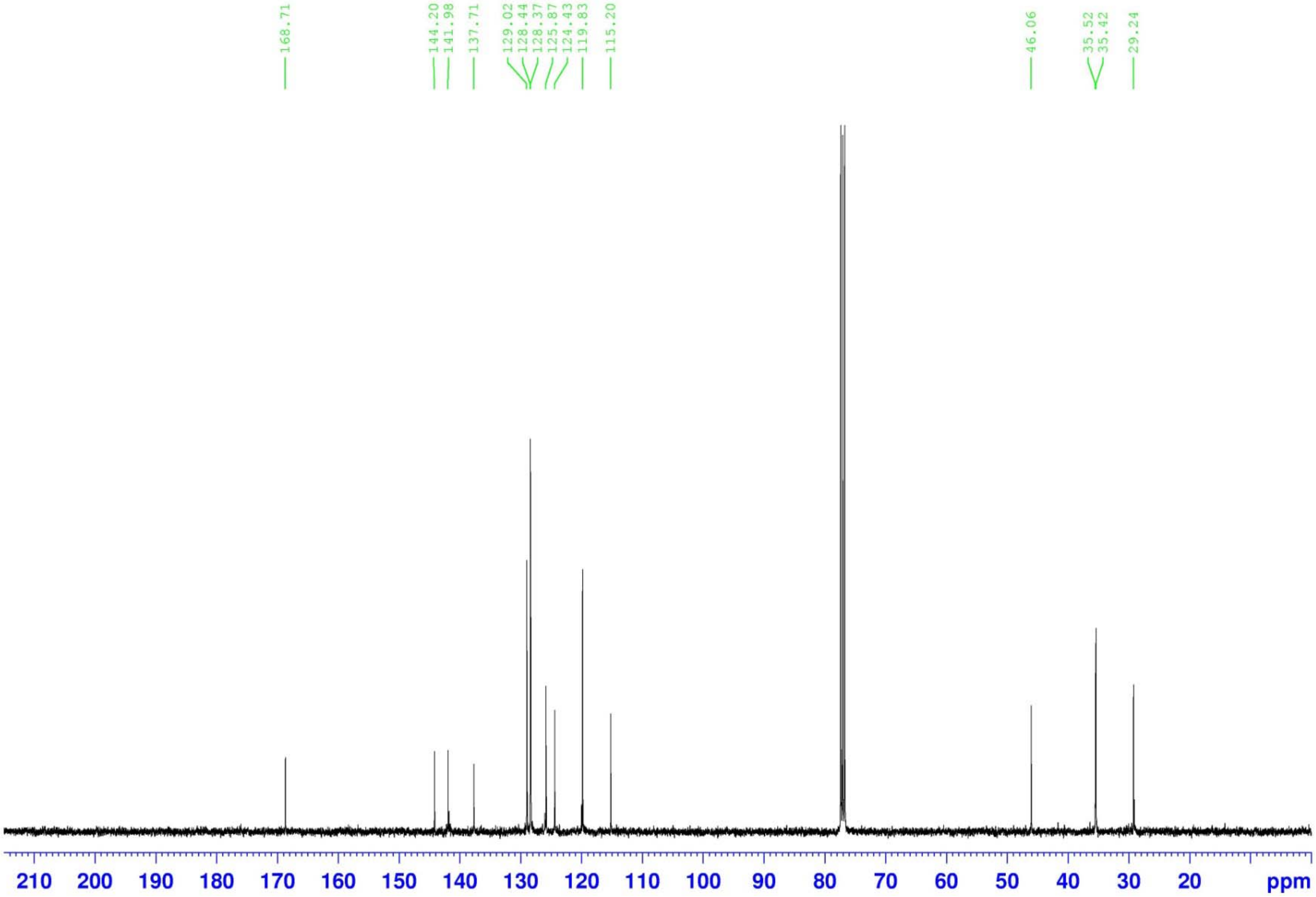
7.518
7.369
7.350
7.304
7.285
7.231
7.211
7.192
7.164
7.146
7.128
5.133
5.107
3.178
2.683
2.663
2.644
2.237
2.219
2.200
1.861
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.96
1.99
2.08
1.95
3.19
1.15
2.00
2.08
2.03
2.12
2.13

^{13}C NMR of 7d


168.71
144.20
141.98
137.71
129.02
128.44
128.37
125.87
124.43
119.83
115.20
46.06
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35.42
29.24
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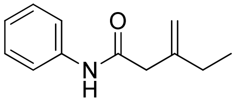
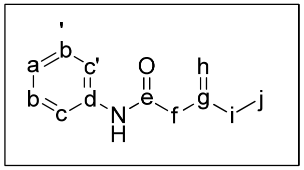
50
40
30
20
ppm



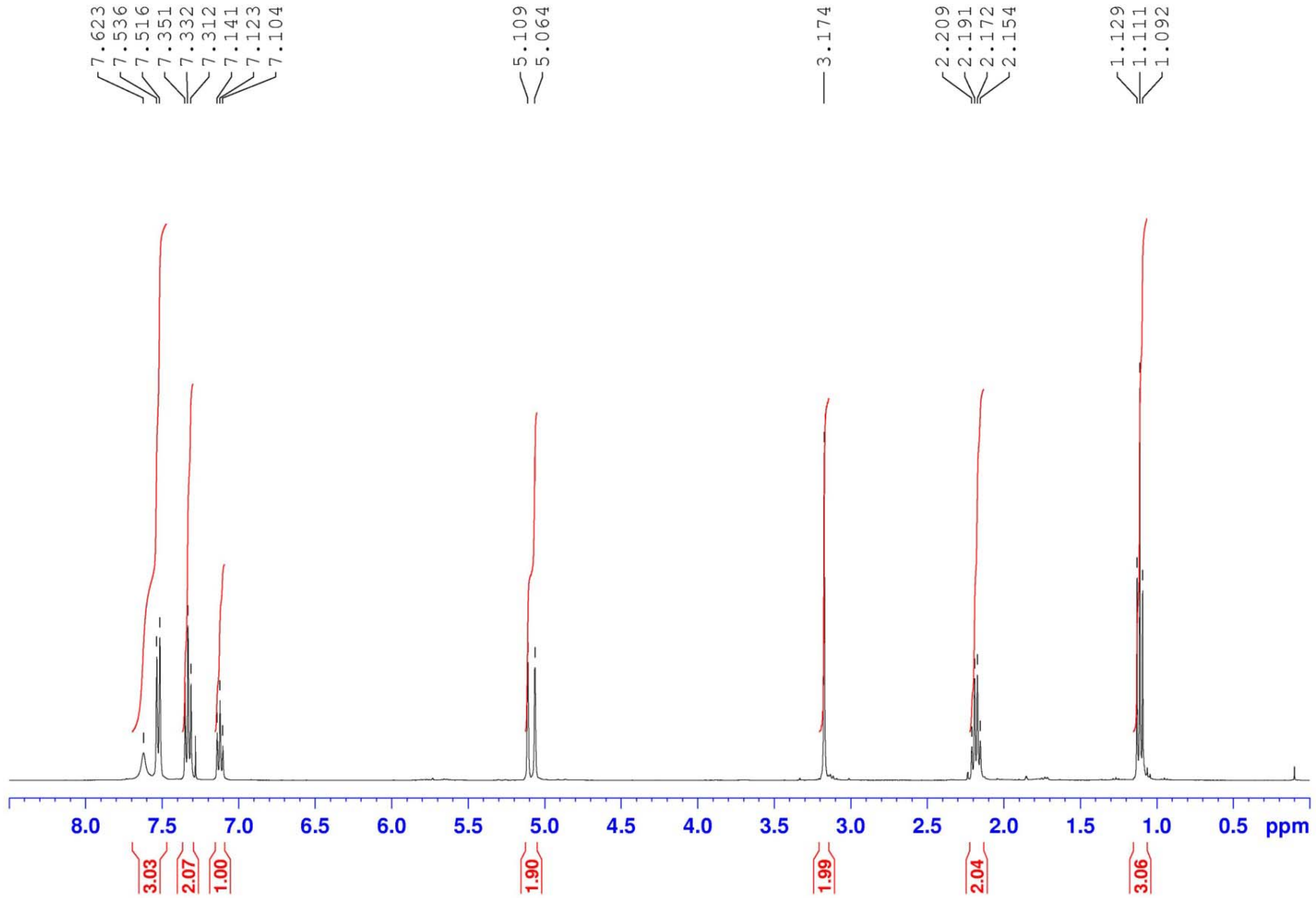
7b



Following the general procedure for hydrolysis of *tert*-butyl esters proceeded by amidation affords, after flash chromatography on silica gel (85–75:15–25 hexanes:ethyl acetate), the title compound (64%, 2 steps) as a white solid.

m.p.	101–102 °C
TLC analysis	R_f 0.3 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.62 (1H, br s, NH), 7.52 (2H, d, *J* = 8.0 Hz, c,c'), 7.33 (2H, t, *J* = 7.6 Hz, b,b'), 7.12 (1H, t, *J* = 7.6 Hz, a), 5.11 and 5.06 (2H, s's, h), 3.17 (2H, s, f), 2.18 (2H, q, *J* = 7.2 Hz, i), 1.11 (3H, t, *J* = 7.2 Hz, j).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 168.72 (e), 146.09(d), 137.79 (g), 128.98 (b,b'), 124.33 (a), 119.74 (c,c'), 113.92 (h), 46.23 (f), 28.89 (i), 12.12 (j).
IR (neat)	3240 (N-H stretch), 3187, 2955, 2839, 1658 (C=O stretch), 1595, 1544 (N-H bend), 1488, 1444, 1400, 1352, 1297, 1252 (C-N stretch), 1187, 969, 759, 693 cm^{-1}.
HRMS (CI)	Calcd. for $C_{12}H_{16}NO$ (M+H): 190.1232, found 190.1237 *m/z*.

^{1}H NMR of 7b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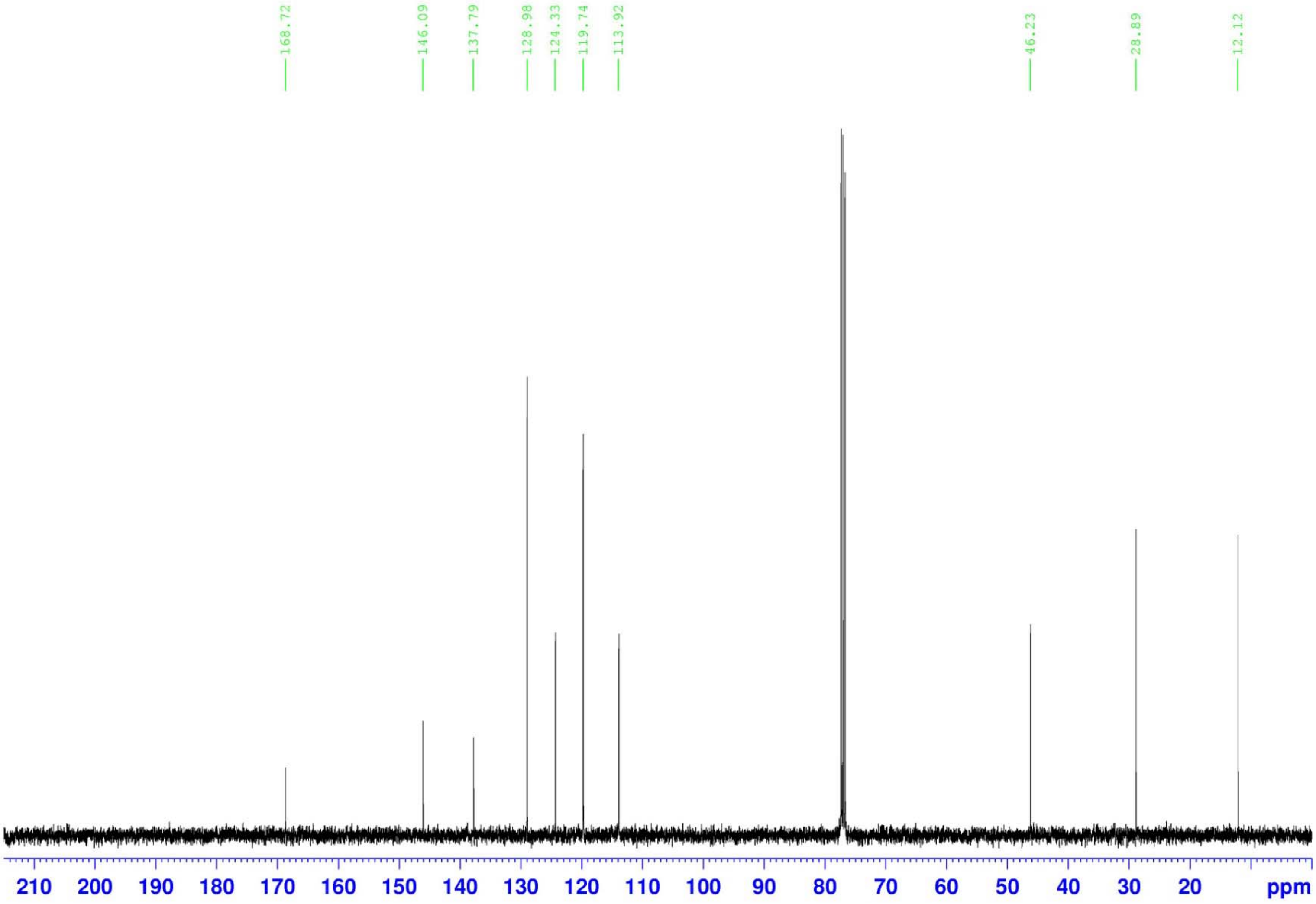
7.516
7.351
7.332
7.312
7.141
7.123
7.104
5.109
5.064
3.174
2.209
2.191
2.172
2.154
1.129
1.111
1.092
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.03
2.07
1.00
1.90
1.99
2.04
3.06

^{13}C NMR of 7b


168.72
146.09
137.79
128.98
124.33
119.74
113.92
46.23
28.89
12.12
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200
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160
150
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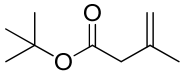
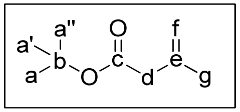
80
70
60
50
40
30
20
ppm



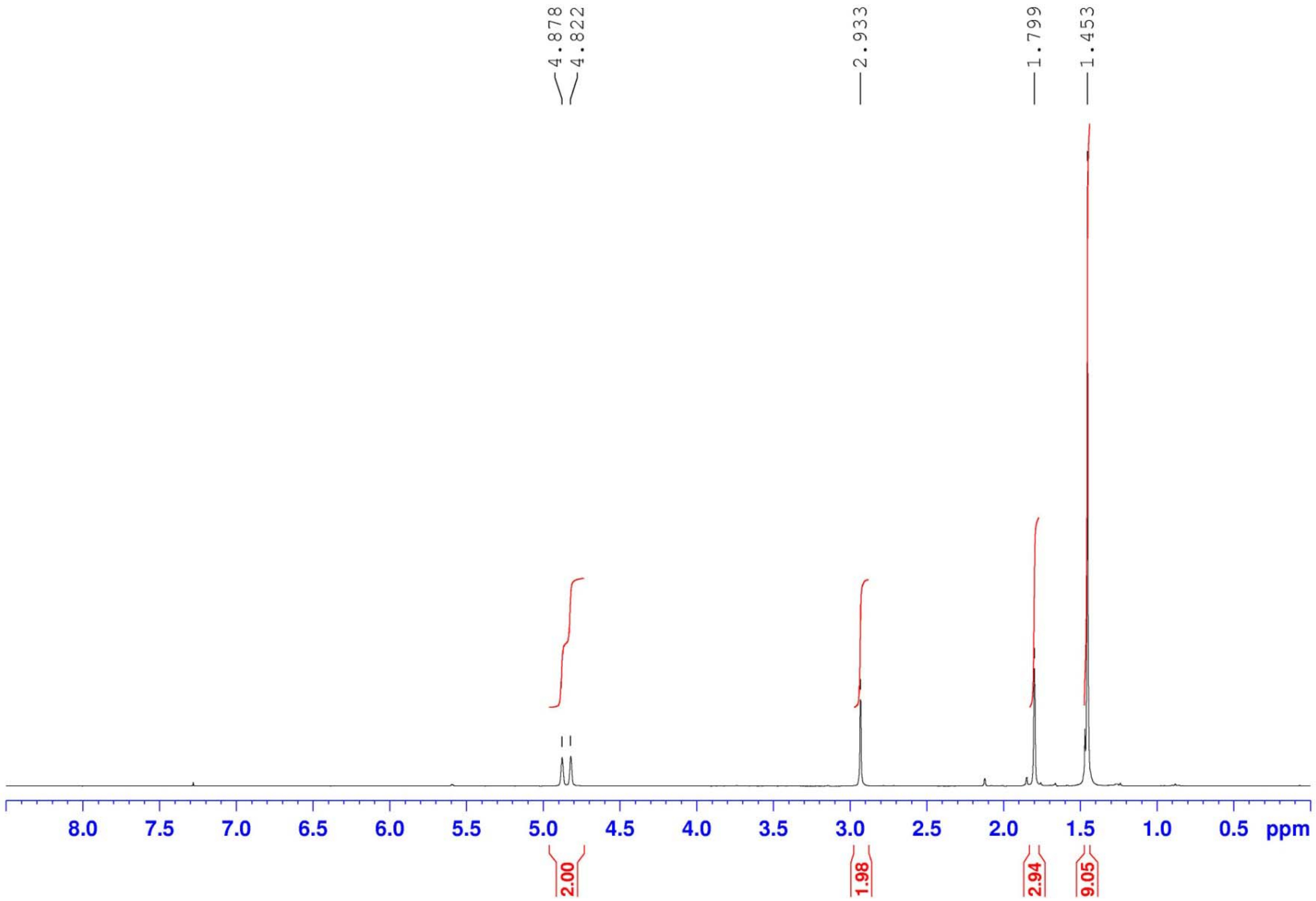
7g



Following the general procedure for the preparation of *tert*-butyl esters via nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (59%) as a light yellow oil.

TLC analysis	R_f 0.5 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 4.88 and 4.82 (2H, s's, f), 2.93 (2H, s, d), 1.80 (3H, s, g), 1.45 (9H, s, a,a',a").
^{13}C NMR (75 MHz, CDCl$_3$)	δ 170.72 (c), 139.13 (e), 114.09 (f), 80.42 (b), 44.79 (d), 27.99 (a,a',a"), 22.38 (g).
IR (neat)	3075, 2976, 2934, 1728 (C=O stretch), 1647, 1455, 1366, 1258 (C-O stretch), 1139, 690, 843 cm^{-1}.

^{1}H NMR of 7g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4.5
4.0
3.5
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1.0
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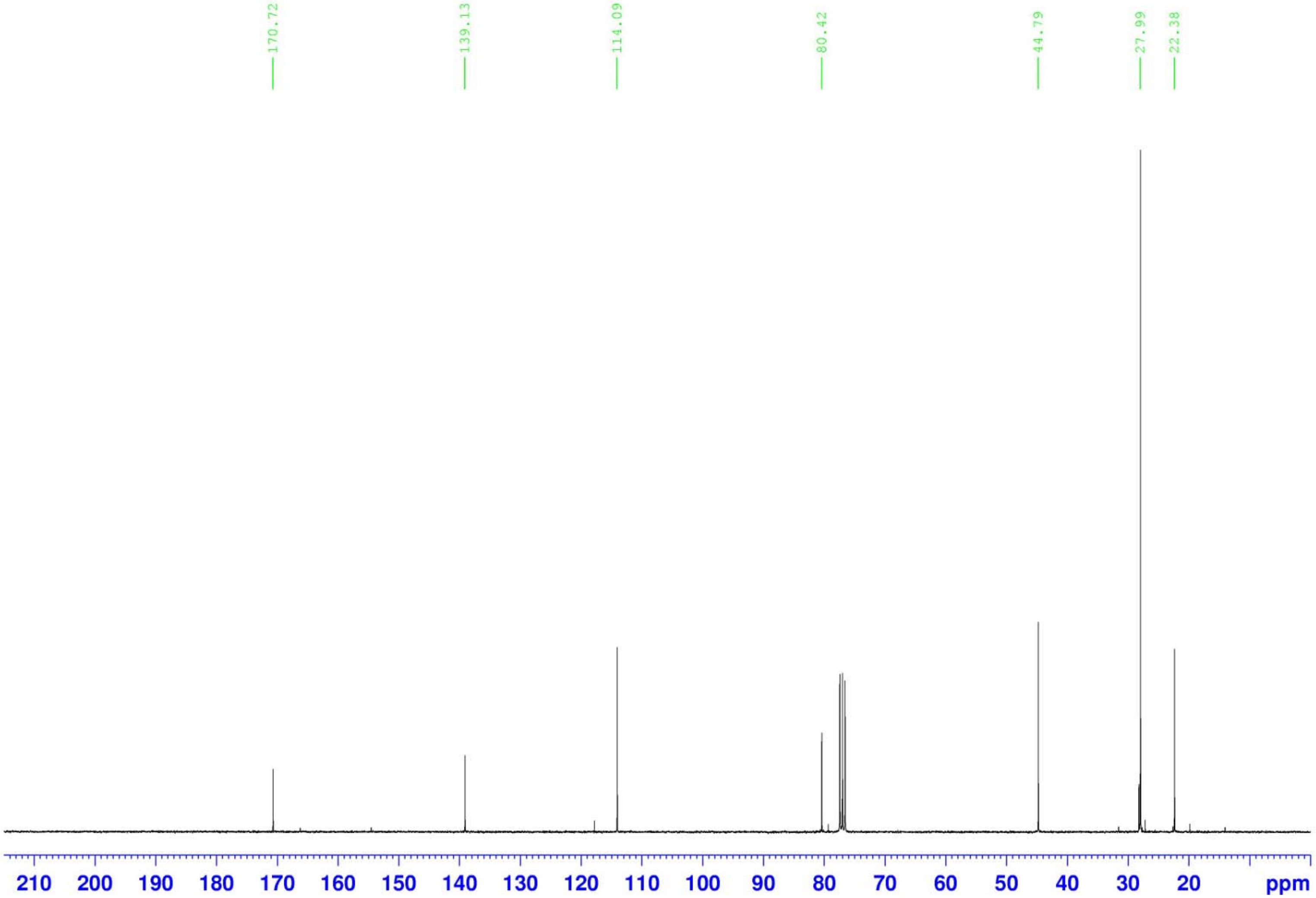
ppm
2.00
1.98
2.94
9.05

^{13}C NMR of 7g


170.72
139.13
114.09
80.42
44.79
27.99
22.38
210
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80
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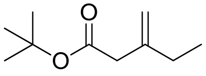
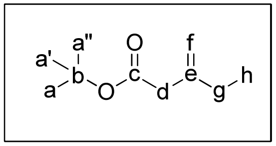
20
ppm



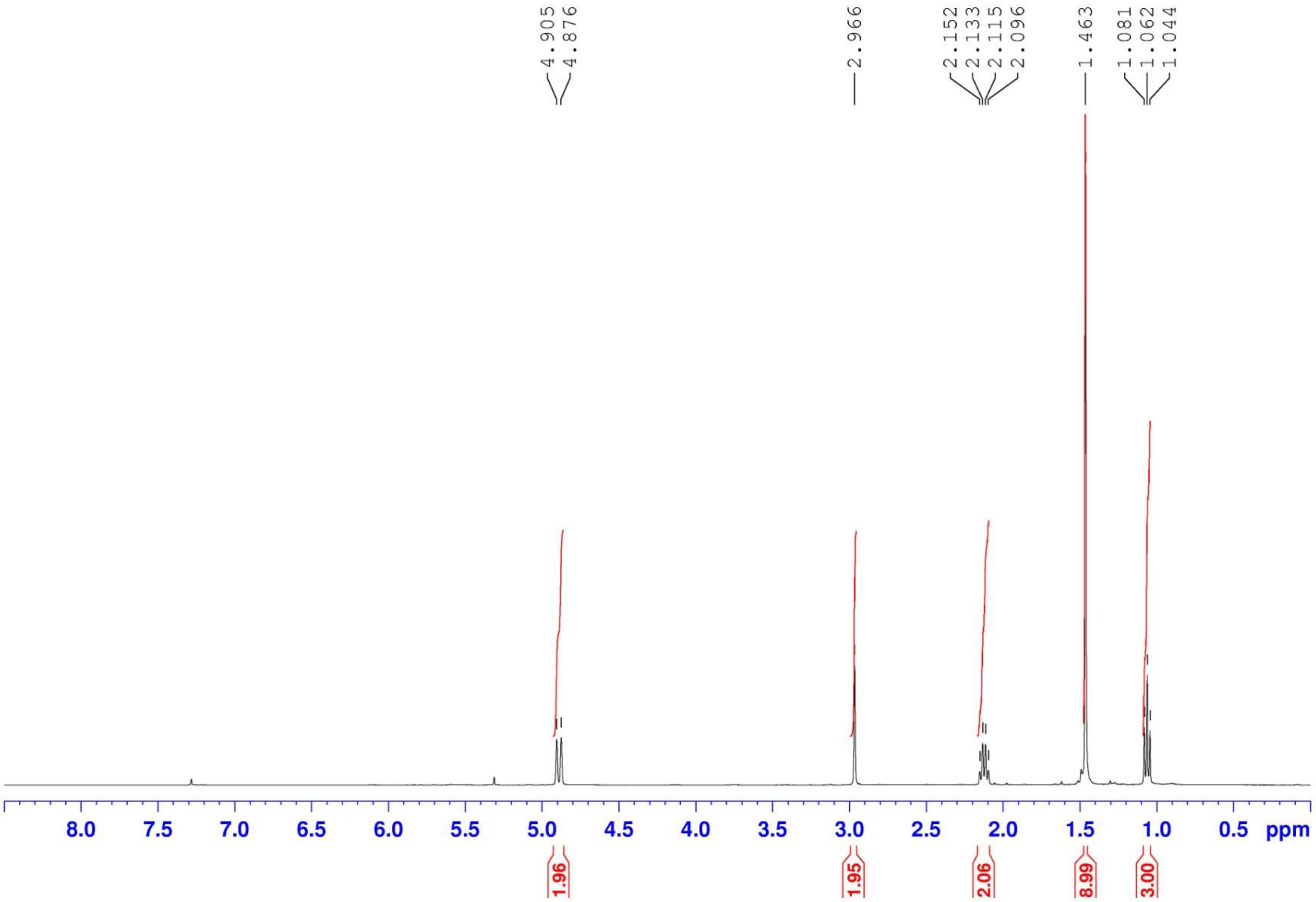
7h



Following the general procedure for the preparation of *tert*-butyl esters via nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (62%) as a light yellow oil.

TLC analysis	R_f 0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 4.91 and 4.88 (2H, s's, f), 2.97 (2H, s, d), 2.12 (2H, q, J = 7.6 Hz, g), 1.46 (9H, s, a,a',a"), 1.06 (3H, t, J = 7.6 Hz, h).
^{13}C NMR (100 MHz, CDCl$_3$)	170.99 (c), 144.65 (e), 111.83 (f), 80.40 (b), 43.47 (d), 28.78 (g), 28.01 (a,a',a"), 12.02 (h).
IR (neat)	2935, 2848, 1731 (C=O stretch), 1653, 1391, 1252 (C-O stretch), 1145, 1122, 1040, 948, 761, 576 cm^{-1}.

^{1}H NMR of 7h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1.081
1.062
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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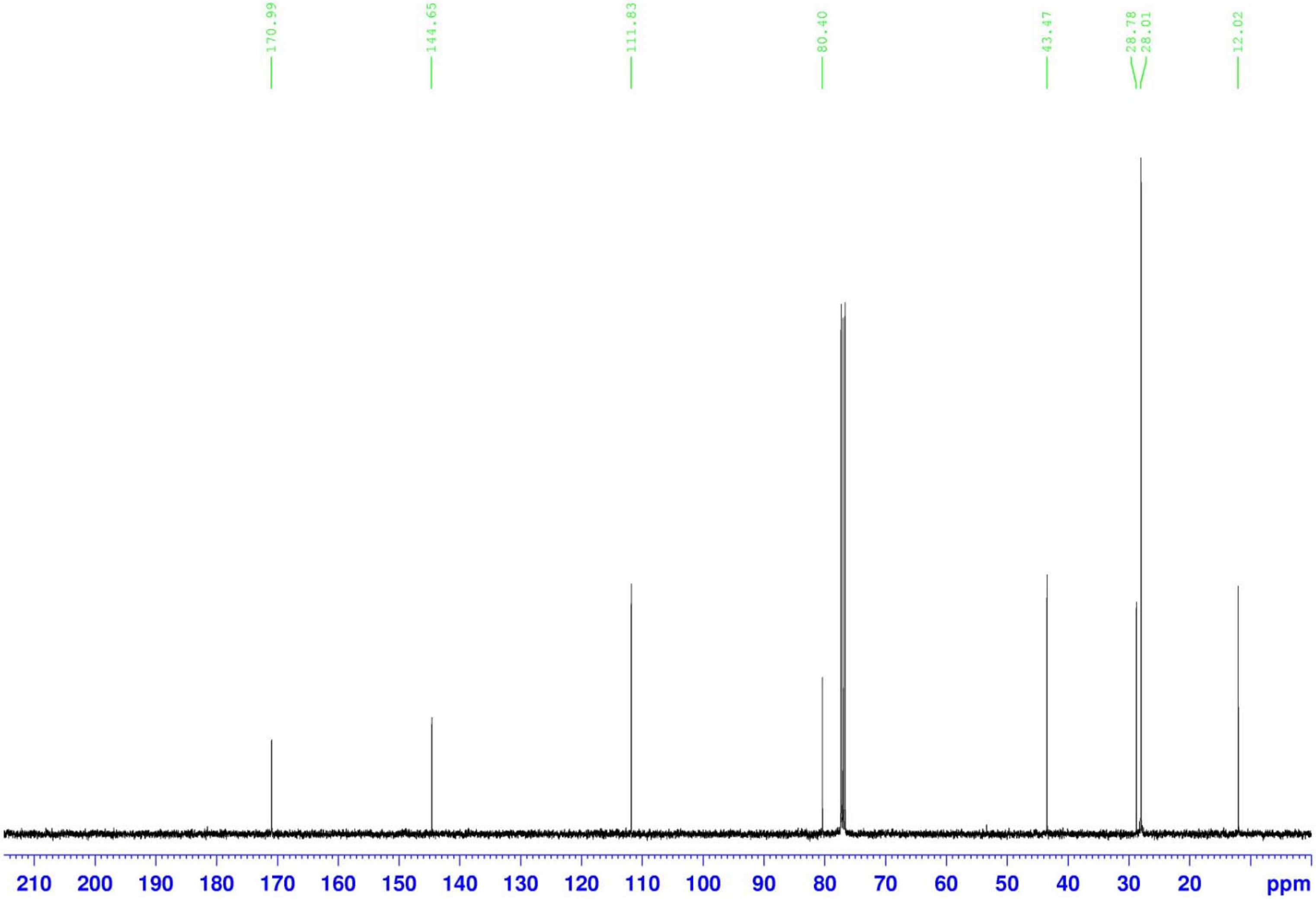
1.0
0.5
ppm
1.96
1.95
2.06
8.99
3.00

^{13}C NMR of 7h


170.99
144.65
111.83
80.40
43.47
28.78
28.01
12.02
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200
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50
40
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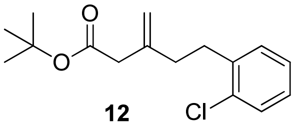
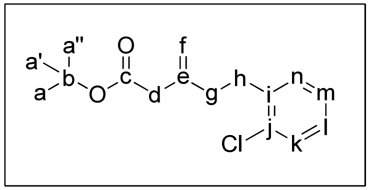
20
ppm



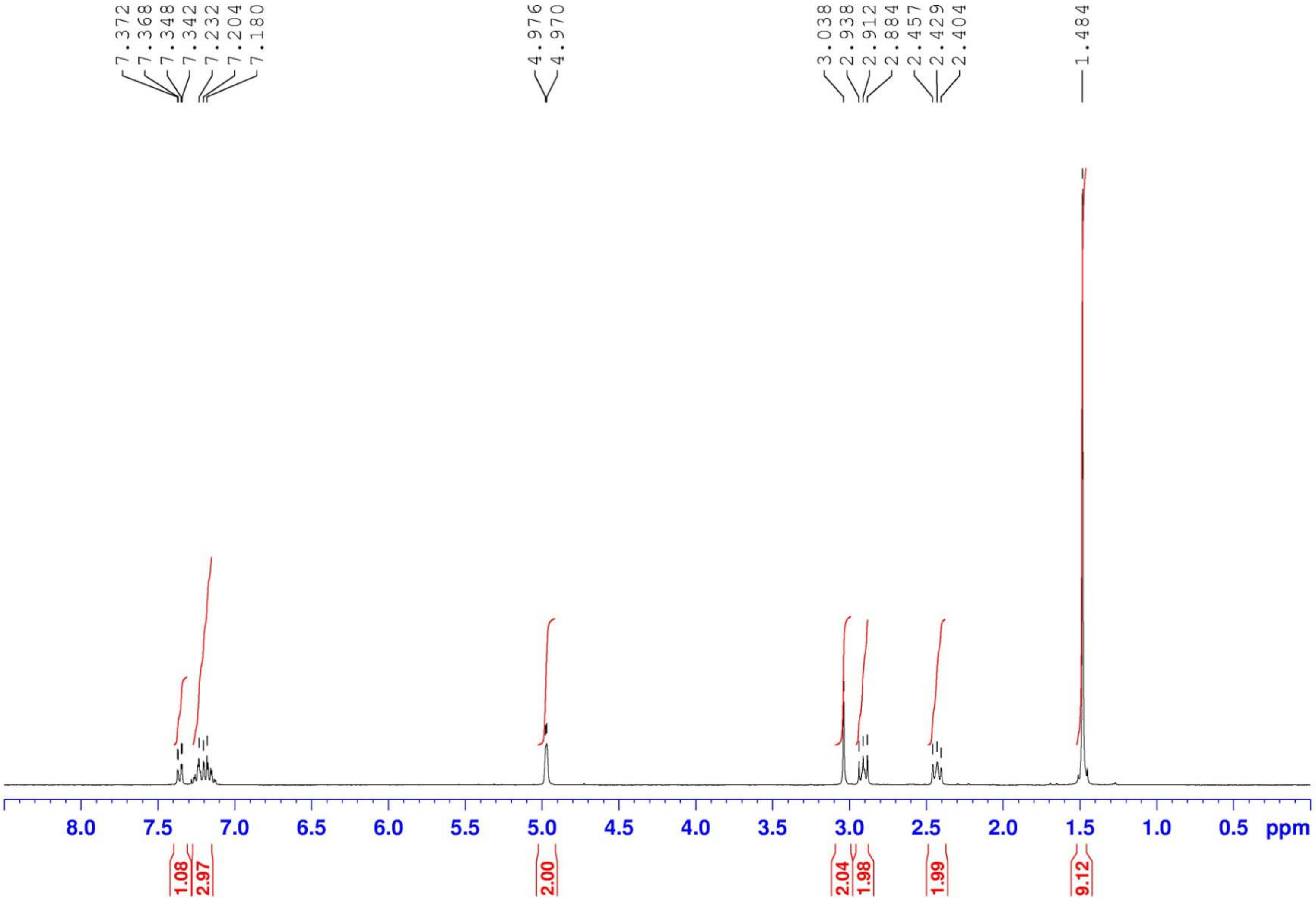
12



Following the general procedure for the preparation of *tert*-butyl esters via nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (41%) as a light yellow oil.

TLC analysis	R_f 0.5 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.40–7.30 (1H, m, k), 7.30–7.10 (3H, m, n,m,l), 4.98 and 4.97 (2H, s's, f), 3.04 (2H, s, d), 2.91 (2H, t, J = 7.8 Hz, h), 2.43 (2H, t, J = 8.4 Hz, g), 1.48 (9H, s, a,a',a").
^{13}C NMR (75 MHz, CDCl$_3$)	δ 170.76 (c), 142.22 (i), 139.34 (e), 133.91 (j), 130.28 (k), 129.45 (l), 127.38 (m), 126.77 (n), 113.79 (f), 80.61 (b), 43.39 (d), 35.89 (g), 31.91 (h), 28.05 (a,a',a").
IR (neat)	2975, 2931, 1722 (C=O stretch), 1647, 1474, 1443, 1366, 1254 (C-O stretch), 1142, 1052, 1038, 954, 900, 824, 749, 736, 671, 575 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{16}H_{21}ClNaO_2$ (M+Na): 303.1128, found 303.1120 *m/z*.

^{1}H NMR of 12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2.457
2.429
2.404
1.484
8.0
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7.0
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6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
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ppm
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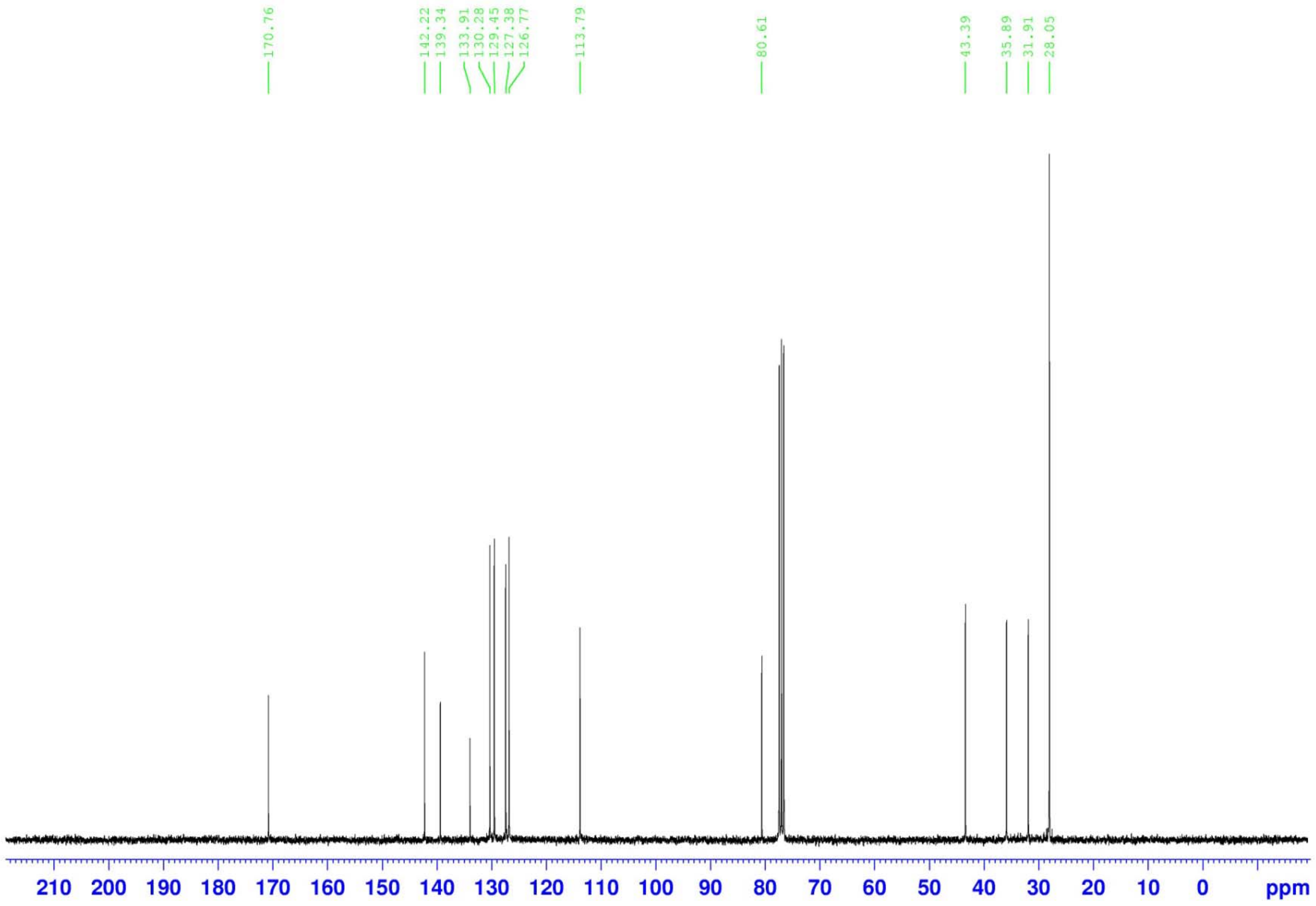
2.04
1.98
1.99
9.12

^{13}C NMR of 12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200
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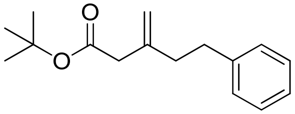
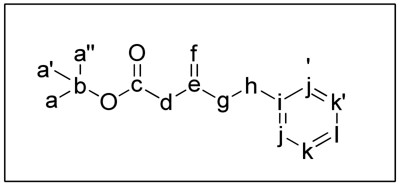
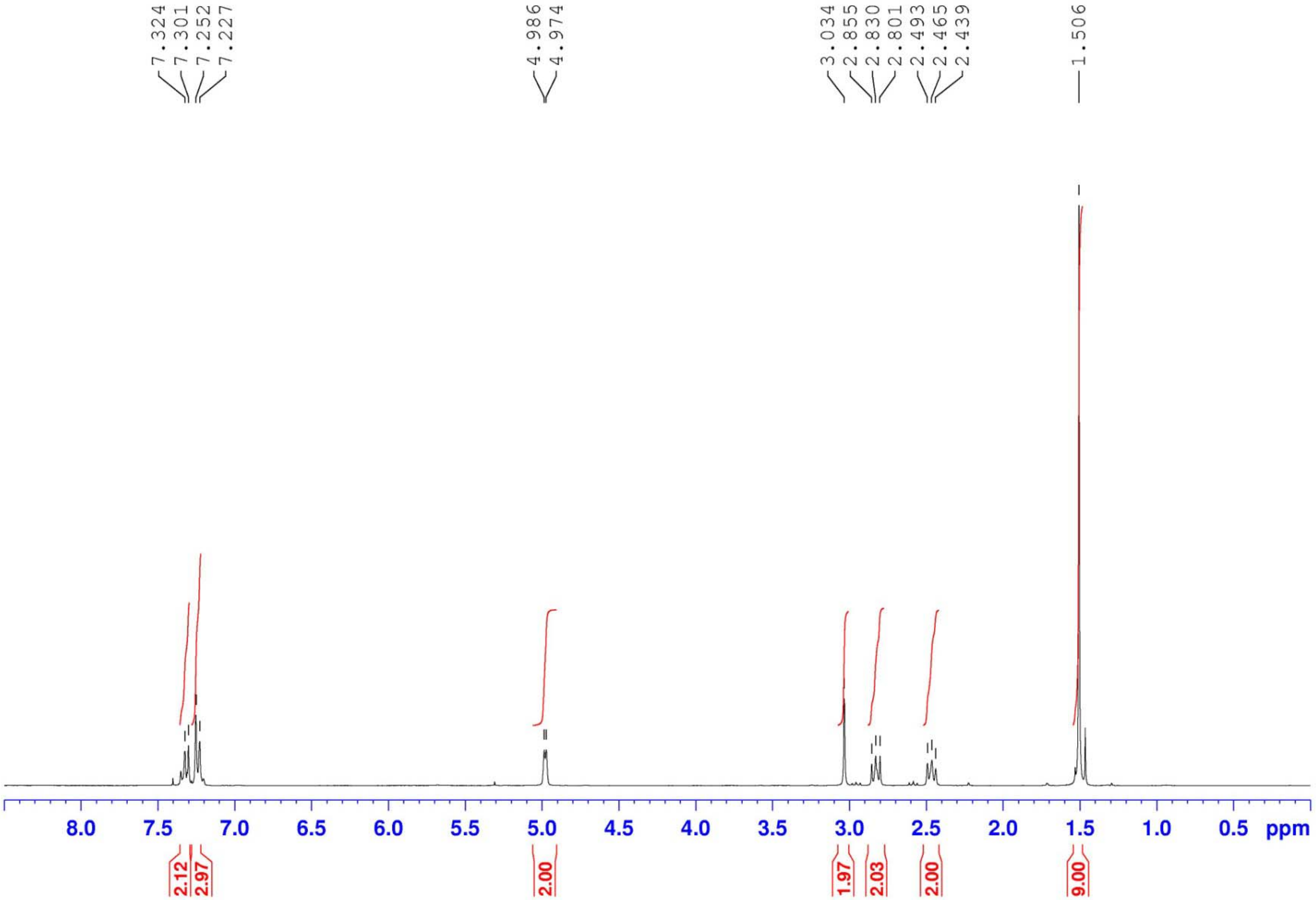




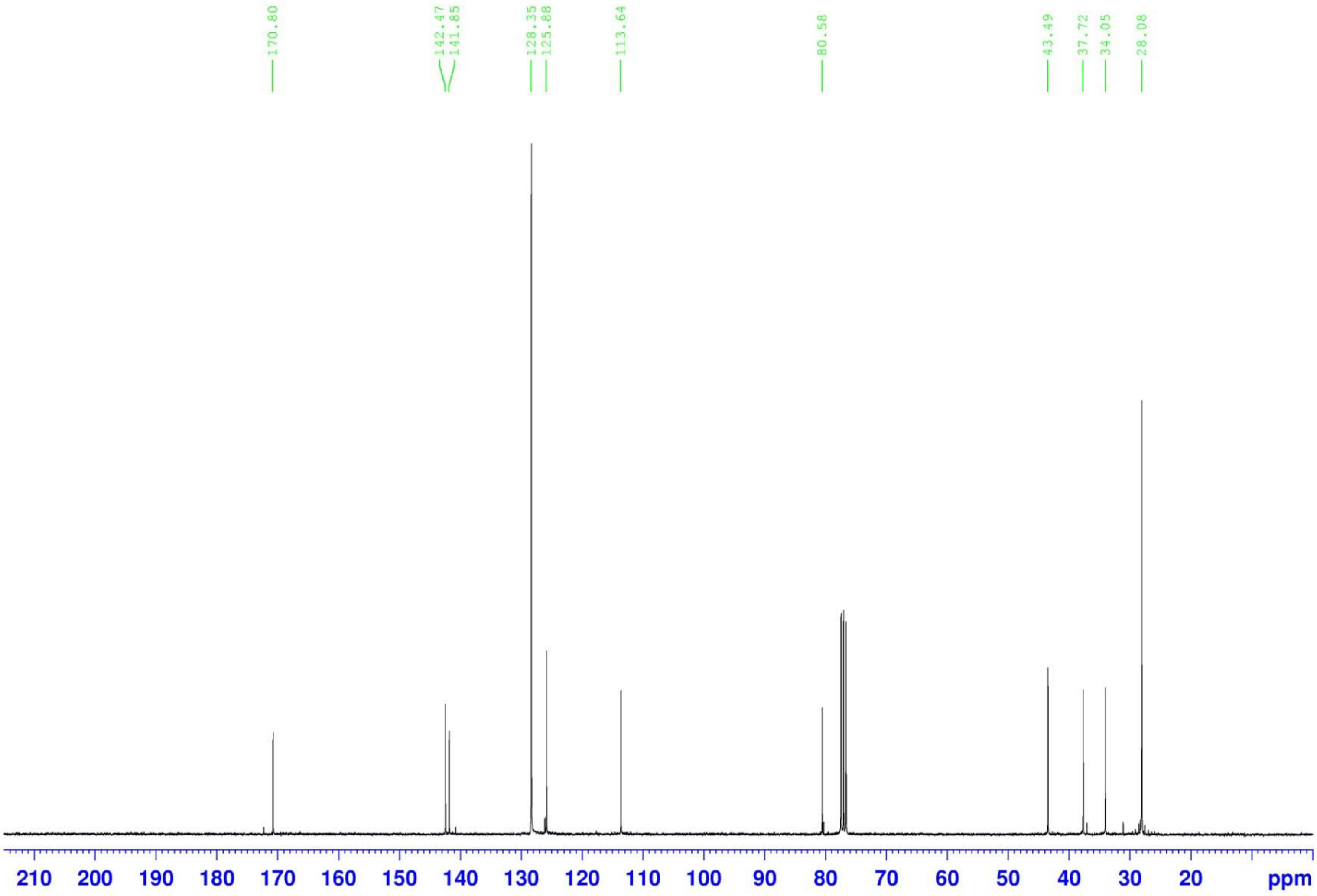
Following the general procedure for the preparation of *tert*-butyl esters via nickel-catalyzed substitution affords, after flash chromatography on silica gel (75–70:25–30 hexanes:dichloromethane), the title compound (53%) as a light yellow oil.

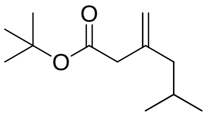
TLC analysis	R_f 0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.40–7.30 (2H, m, k,k'), 7.30–7.20 (3H, m, j,j',l), 4.99 and 4.97 (2H, s's, f), 3.03 (2H, s, d), 2.83 (2H, t, J = 7.5 Hz, h), 2.46 (2H, t, J = 8.4 Hz, g), 1.51 (9H, s, a,a',a").
^{13}C NMR (75 MHz, $CDCl_3$)	δ 170.80 (c), 142.47 (i), 141.85 (e), 128.35 (j,j',k,k'), 125.88 (l), 113.64 (f), 80.58 (b), 43.49 (d), 37.72 (g), 34.05 (h), 28.08 (a,a',a").
IR (neat)	3028, 2978, 2931, 1726 (C=O stretch), 1647, 1496, 1454, 1366, 1255 (C-O stretch), 1139, 1030, 956, 896, 841, 744, 697 cm^{-1}.

^{1}H NMR of 3-methylidene-5-phenylpentanoic acid *tert*-butyl ester

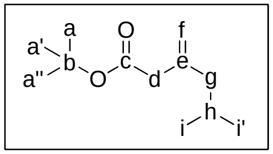


^{13}C NMR of 3-methylidene-5-phenylpentanoic acid *tert*-butyl ester





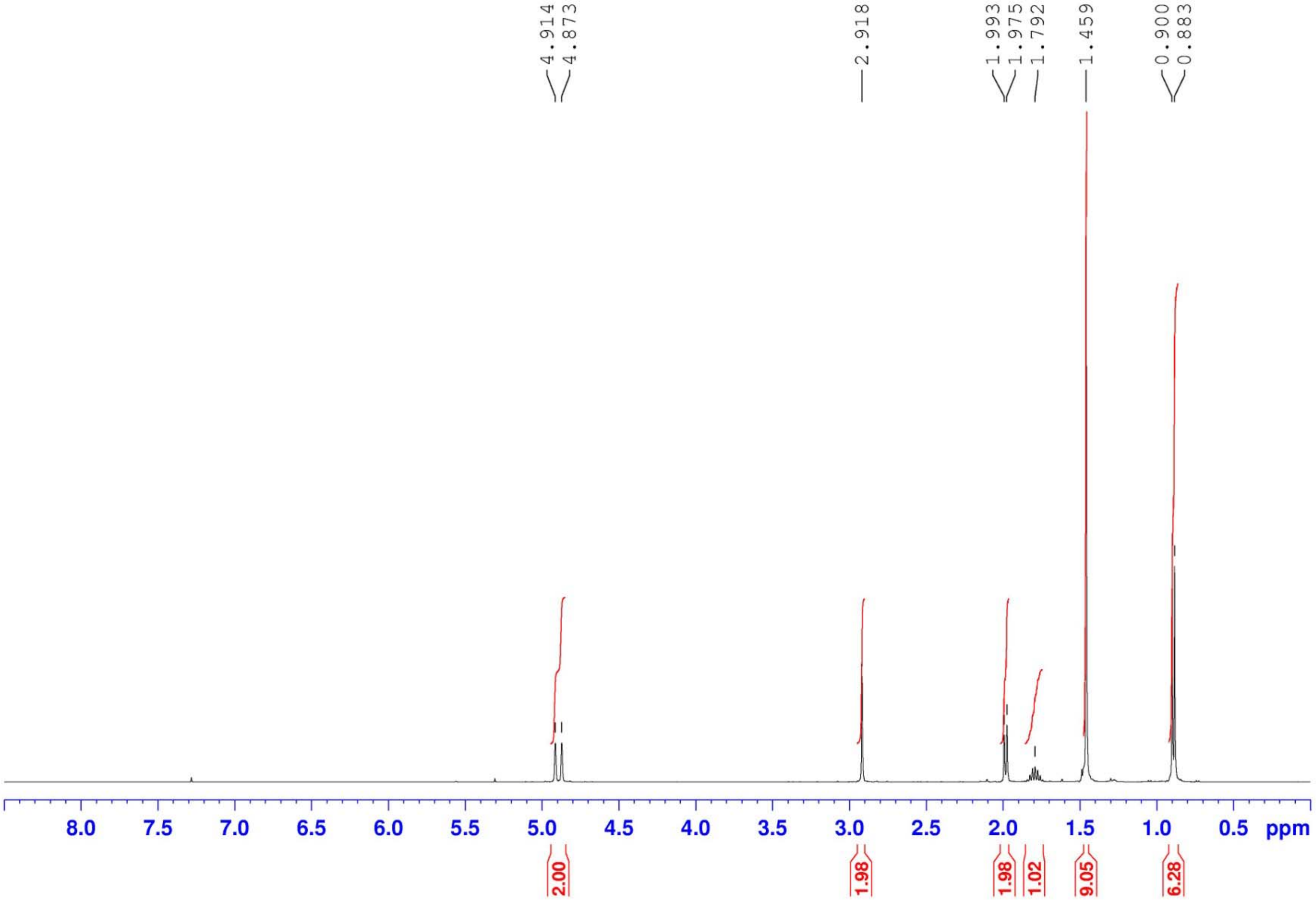
7i



Following the general procedure for the preparation of *tert*-butyl esters through the synthesis of vinyl bromides followed by nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (45%, 2 steps) as a light yellow oil.

TLC analysis	R_f 0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 4.91 and 4.87 (2H, s's, f), 2.92 (2H, s, d), 1.98 (2H, d, J = 7.2 Hz, g), 1.95–1.85 (1H, m, h,), 1.46 (9H, s, a,a',a"), 0.89 (6H, d, J = 6.6 Hz, i,i').
^{13}C NMR (100 MHz, CDCl$_3$)	δ 170.90 (c), 141.96 (e), 114.33 (f), 80.36 (b), 45.79 (d), 43.03 (g), 28.00 (a,a',a"), 25.75 (h), 22.38 (i,i').
IR (neat)	2969, 2912, 1722 (C=O stretch), 1431, 1376, 1177 (C-O stretch), 1117, 884, 826, 740, 521 cm^{-1}.
HRMS (CI)	Calcd. for $C_{12}H_{23}O_2$ (M+H): 199.1698, found 199.1705 *m/z*.

^{1}H NMR of 7i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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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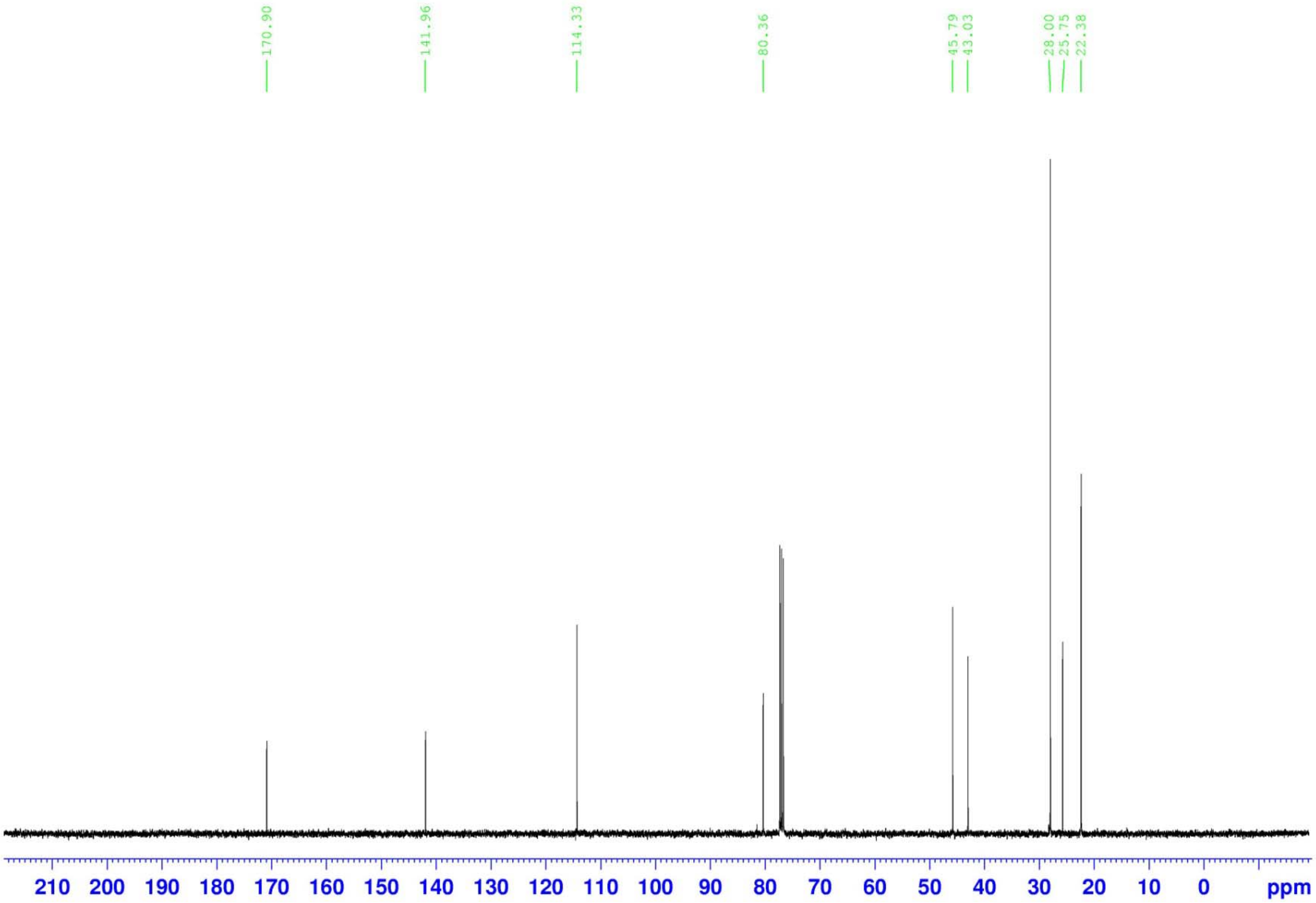
ppm
2.00
1.98
1.98
1.02
9.05
6.28

^{13}C NMR of 7i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114.33
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43.03
28.00
25.75
22.38
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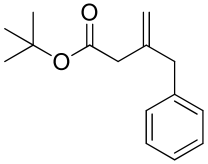
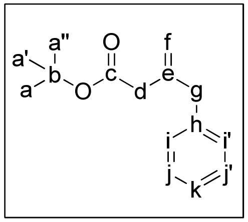
ppm



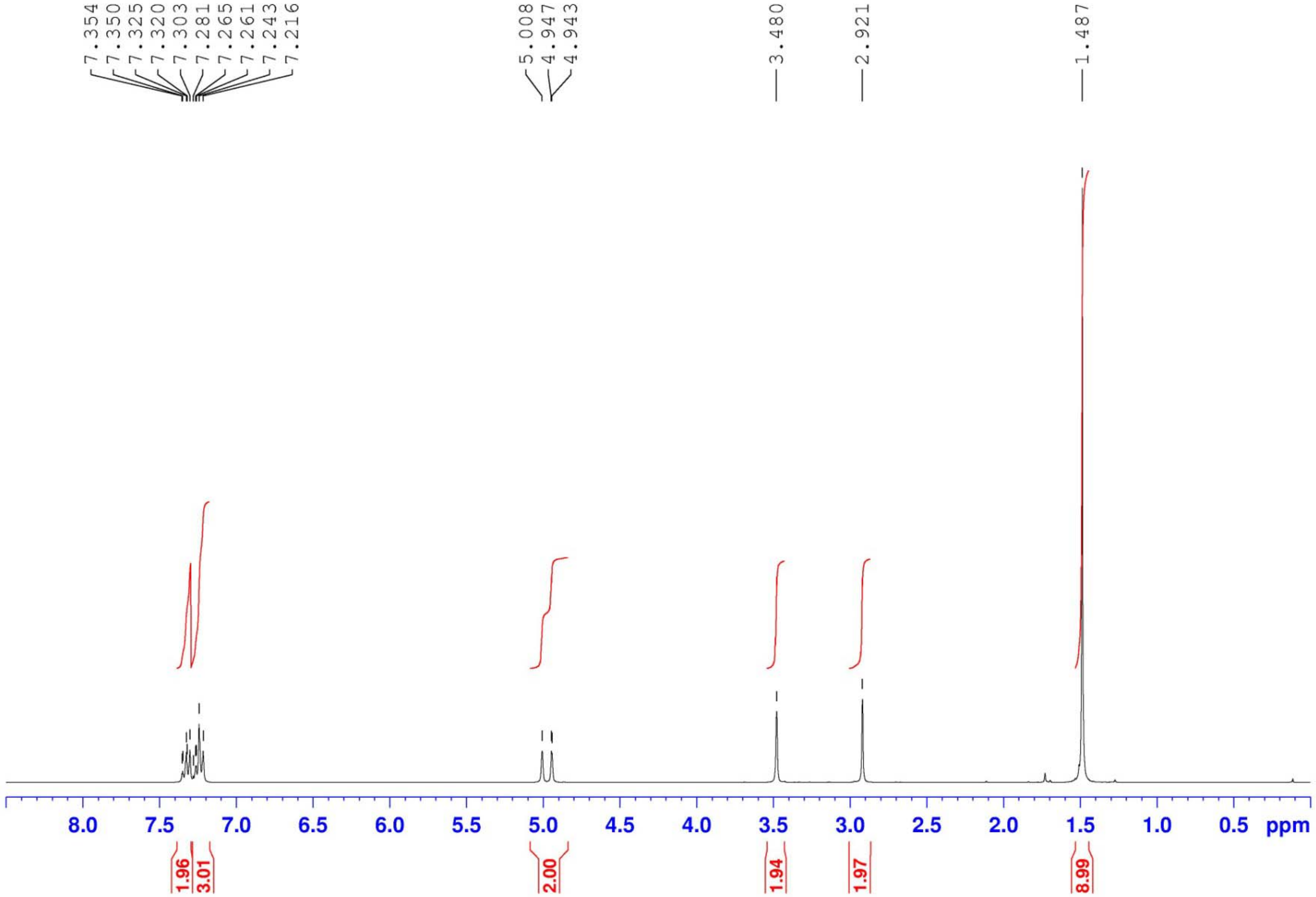
21a



Following the general procedure for the preparation of *tert*-butyl esters by the synthesis of vinyl bromides followed by nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (54%, 2 steps) as a light yellow oil.

TLC analysis	R_f 0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.40–7.30 (2H, m, j,j'), 7.30–7.20 (3H, m, i,i',k), 5.01 and 4.95 (2H, s's, f), 3.48 (2H, s, g), 2.92 (2H, s, d), 1.49 (9H, s, a,a',a").
^{13}C NMR (75 MHz, $CDCl_3$)	δ 170.75 (c), 142.43 (h), 138.97 (e), 129.17 (i,i'), 128.39 (j,j'), 126.30 (k), 115.26 (f), 80.59 (b), 42.68 (g), 42.40 (d), 28.06 (a,a',a").
IR (neat)	2978, 1725 (C=O stretch), 1647, 1494, 1366, 1253 (C-O stretch), 1137, 966, 898, 838, 728, 696, 628 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{15}H_{20}NaO_2$ (M+Na): 255.1361, found 255.1371 *m/z*.

^{1}H NMR of 21a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4.943
3.480
2.921
1.487
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
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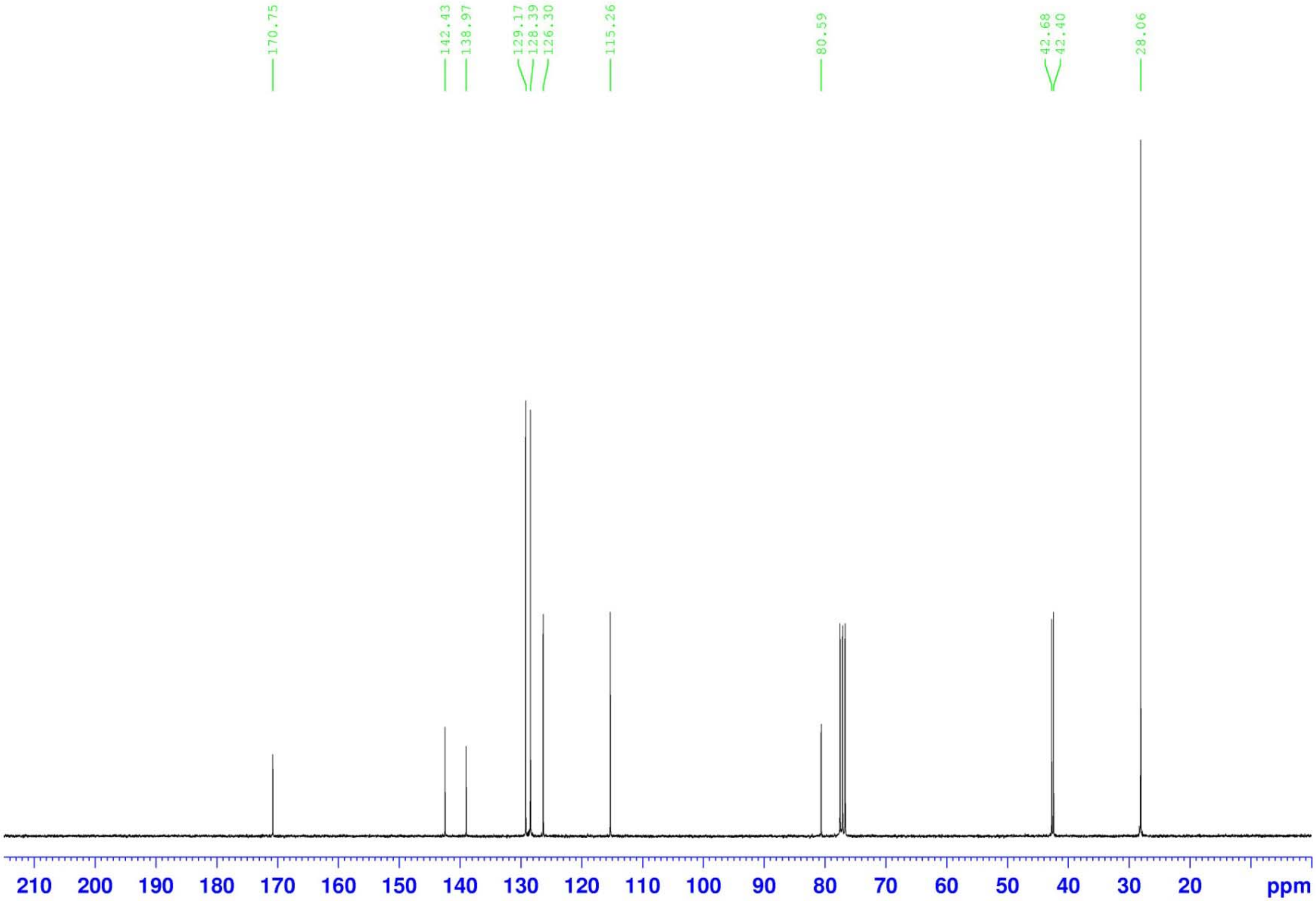
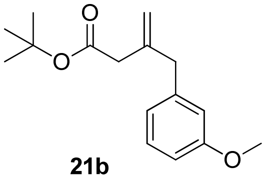
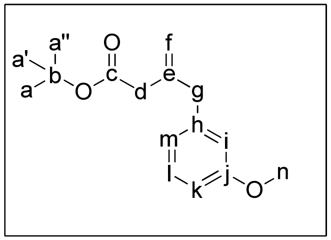
0.5
ppm
1.96
3.01
2.00
1.94
1.97
8.99

^{13}C NMR of 21a





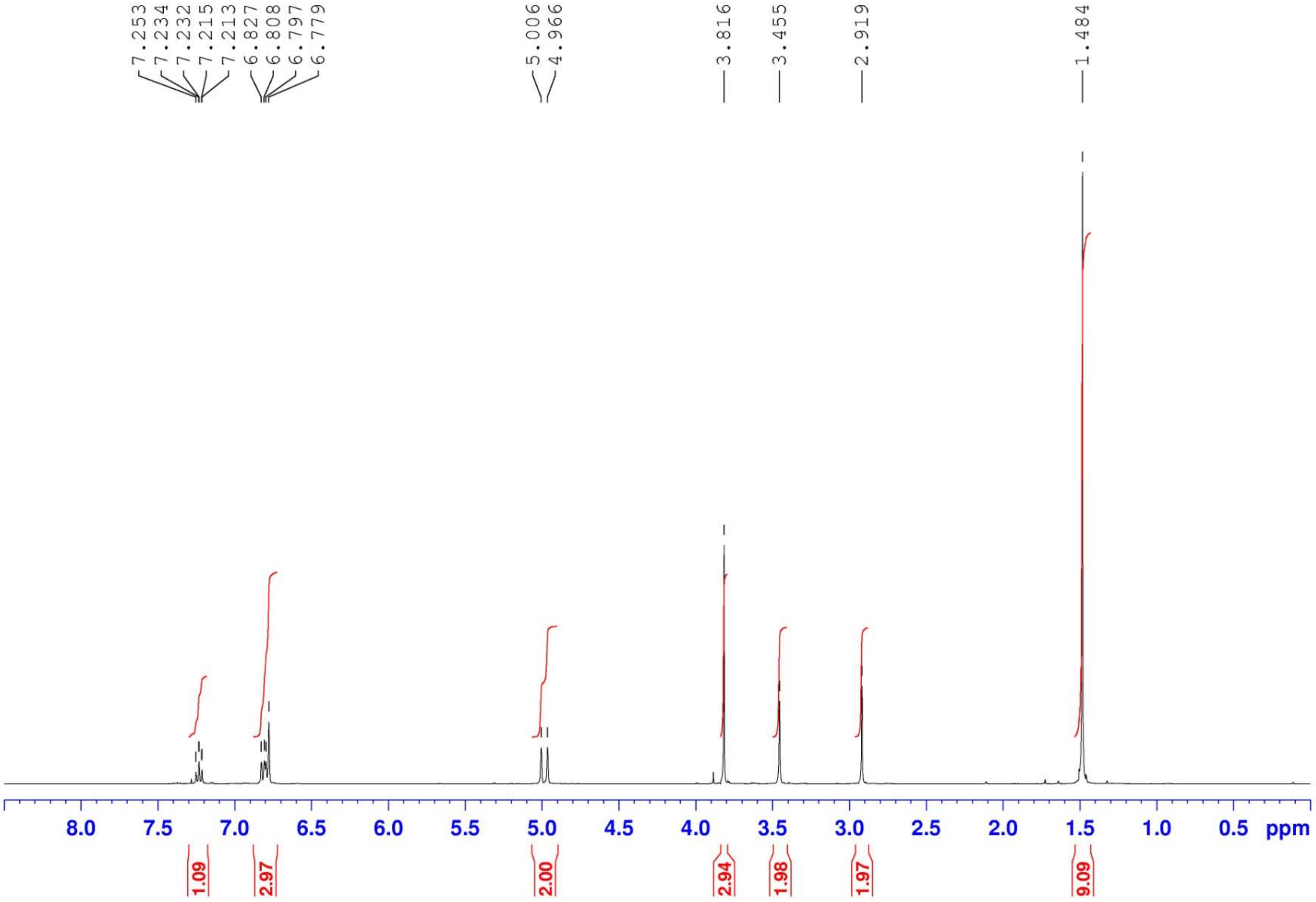
21b



Following the general procedure for the preparation of *tert*-butyl esters through the synthesis of vinyl bromides followed by nickel-catalyzed substitution affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (42%, 2 steps) as a light yellow oil.

TLC analysis	R_f 0.3 (50:50 hexanes:dichloromethane)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.30–7.20 (1H, m, i), 6.85–6.75 (3H, m, m,l,k), 5.01 and 4.97 (2H, s's, f), 3.82 (3H, s, n), 3.45 (2H, s, g), 2.92 (2H, s, d), 1.48 (9H, s, a,a',a").
^{13}C NMR (100 MHz, CDCl$_3$)	δ 170.70 (e), 159.70 (j), 142.22 (h), 140.57 (e), 129.32 (l), 121.59 (m), 115.31 (i), 114.78 (f), 111.71 (k), 80.55 (b), 55.12 (n), 42.73 (d), 42.33 (g), 28.05 (a,a',a").
IR (neat)	2977, 1725 (C=O stretch), 1600, 1584, 1488, 1435, 1366, 1257 (C-O stretch), 1140, 1050, 899, 779, 754, 695, 571 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{16}H_{22}NaO_3$ (M+Na): 285.1467, found 285.1479 *m/z*.

^{1}H NMR of 21b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3.455
2.919
1.484
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
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ppm
1.09
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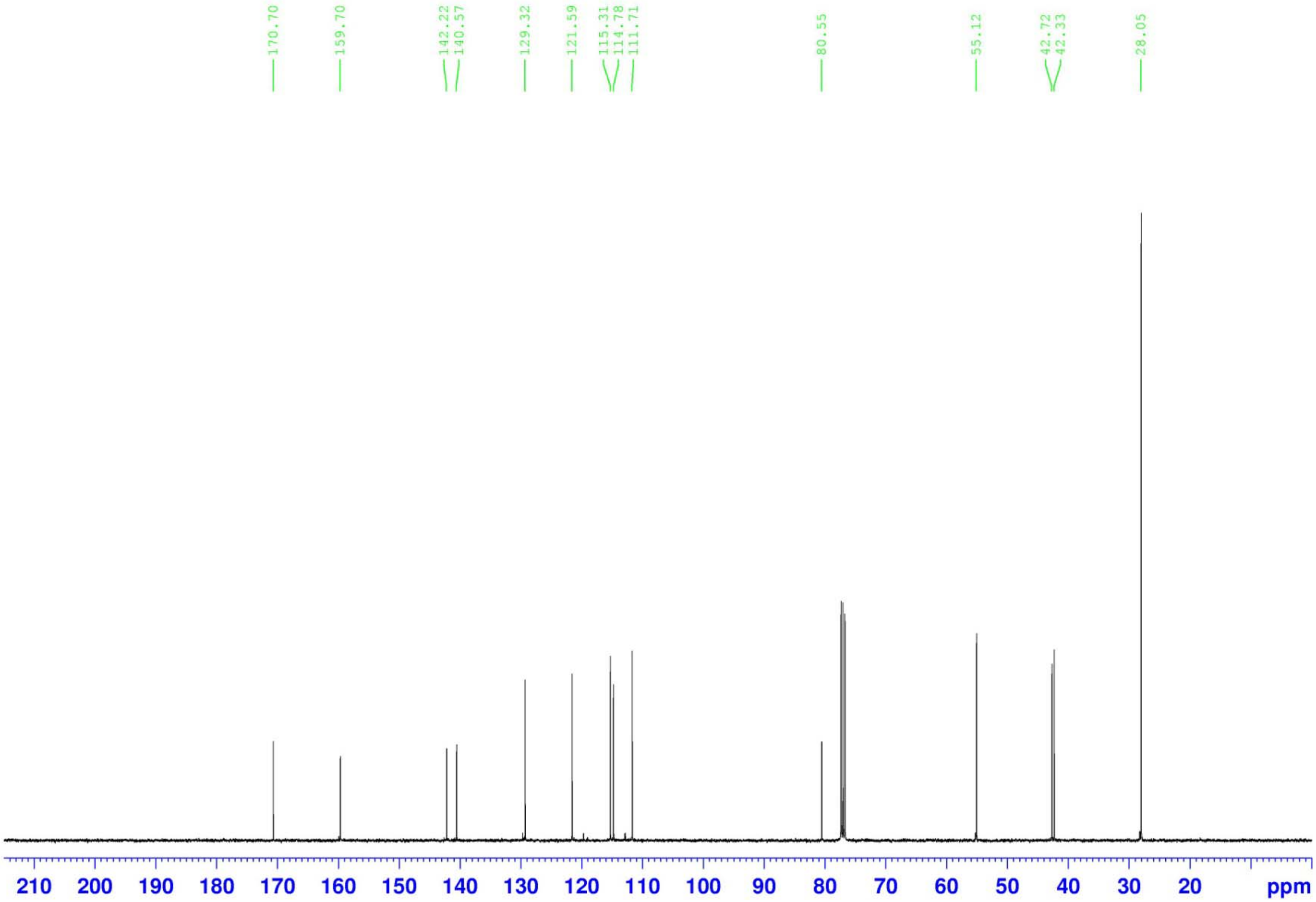
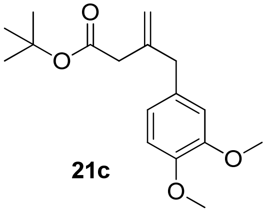
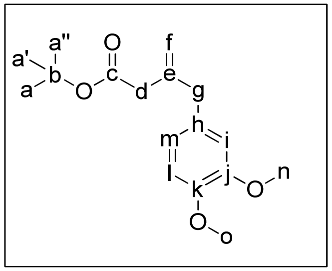
2.00
2.94
1.98
1.97
9.09

^{13}C NMR of 21b





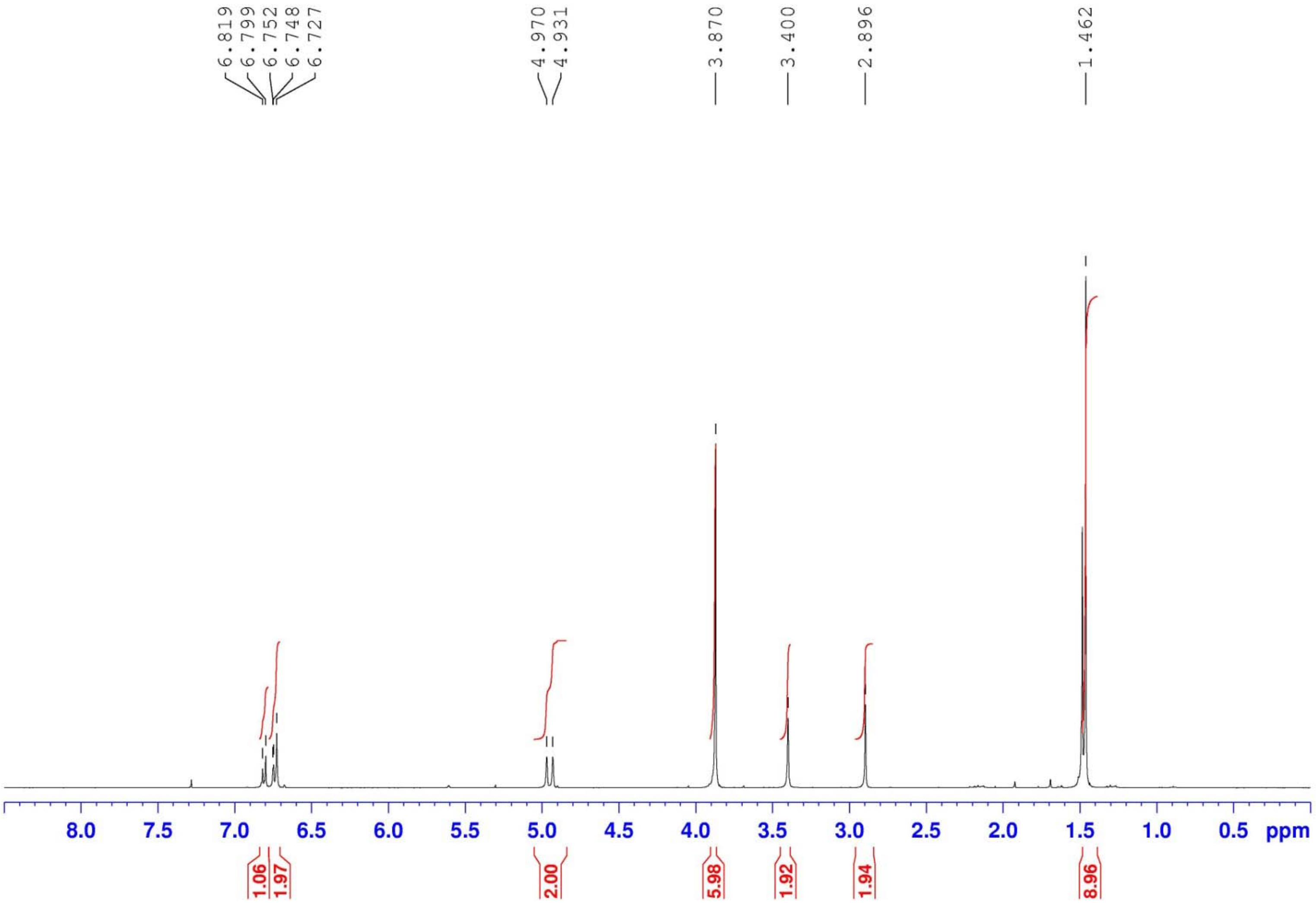
21c



Following the general procedure for the preparation of *tert*-butyl esters through the synthesis of vinyl bromides followed by nickel-catalyzed substitution affords, after flash chromatography on silica gel (75–65:25–35 hexanes:dichloromethane), the title compound (36%, 2 steps) as a light yellow oil.

TLC analysis	R_f 0.3 (50:50 hexanes:dichloromethane)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 6.85–6.75 (1H, m, i), 6.75–6.65 (2H, m, l,m), 4.97 and 4.93 (2H, s's, f), 3.87 (6H, s, n,o), 3.40 (2H, s, g), 2.90 (2H, s, d), 1.46 (9H, s, a,a',a").
^{13}C NMR (100 MHz, CDCl$_3$)	δ 170.74 (c), 148.85 (k), 147.54 (j), 142.66 (e), 131.51 (h), 121.16 (m), 114.95 (i), 112.29 (l), 111.15 (f), 80.53 (b), 55.90 (o), 55.80 (n), 42.29 (g), 42.25 (d), 28.05 (a,a',a").
IR (neat)	2978, 1725 (C=O stretch), 1647, 1599, 1584, 1488, 1454, 1366, 1256 (C-O stretch), 1139, 1050, 899, 779, 738, 694, 572 cm^{-1}.
HRMS (EI)	Calcd. for $C_{17}H_{24}O_4$: 292.1675, found 292.1684 *m/z*.

^{1}H NMR of 21c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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
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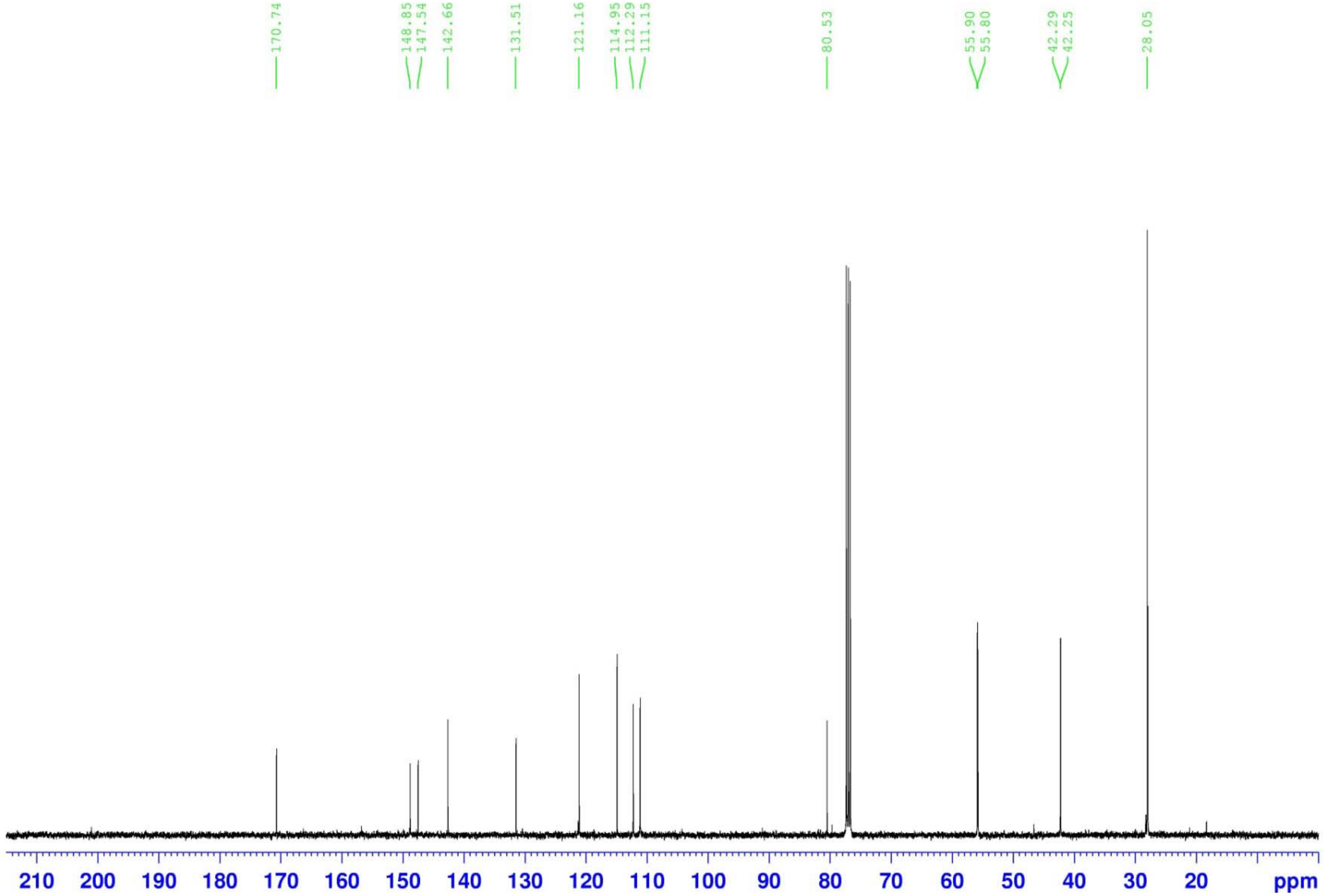
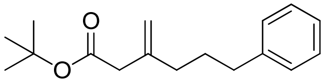
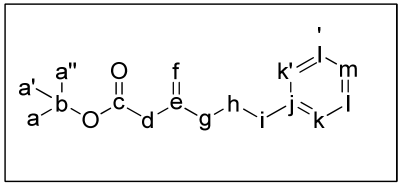
2.00
5.98
1.92
1.94
8.96

^{13}C NMR of 21c



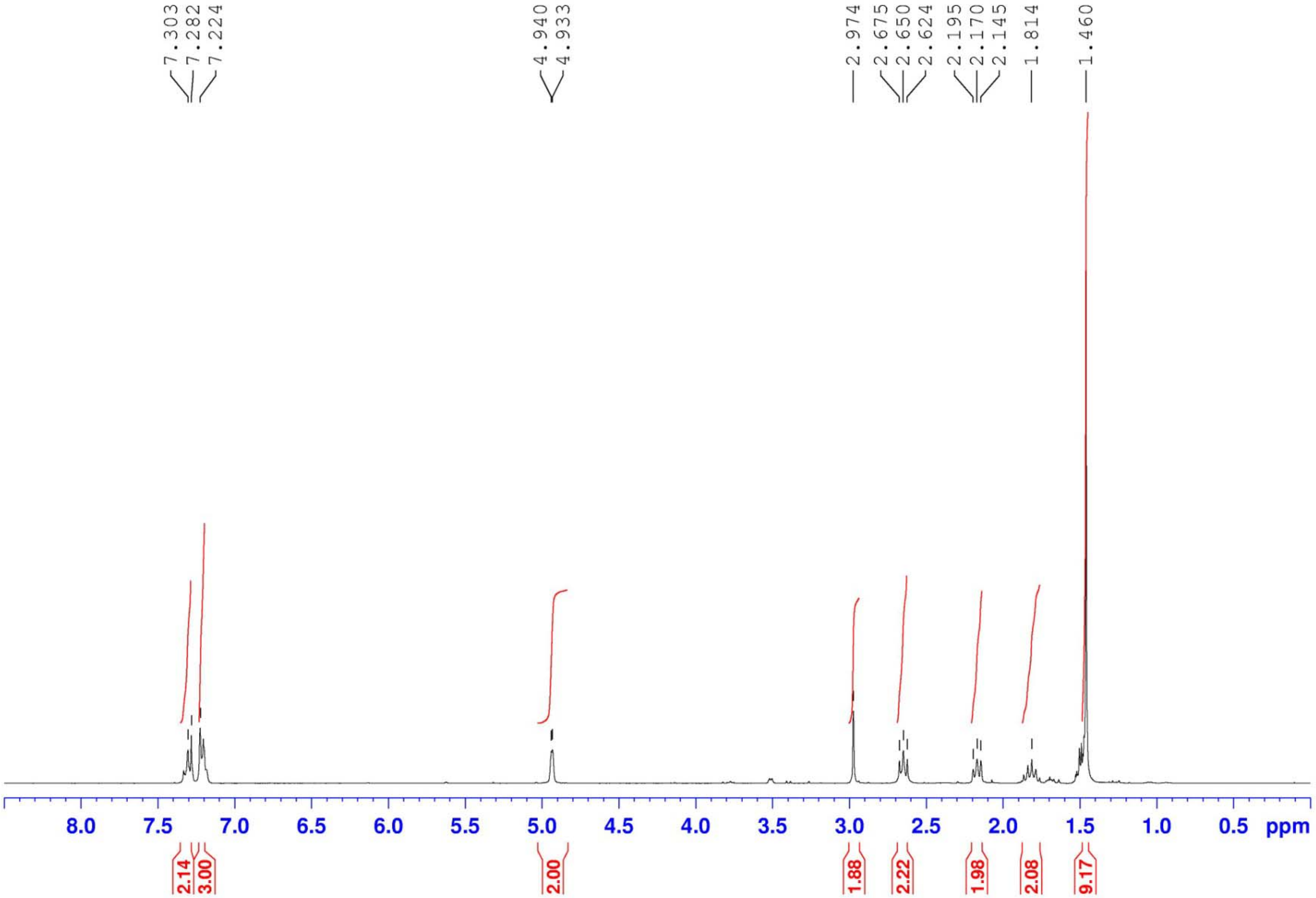




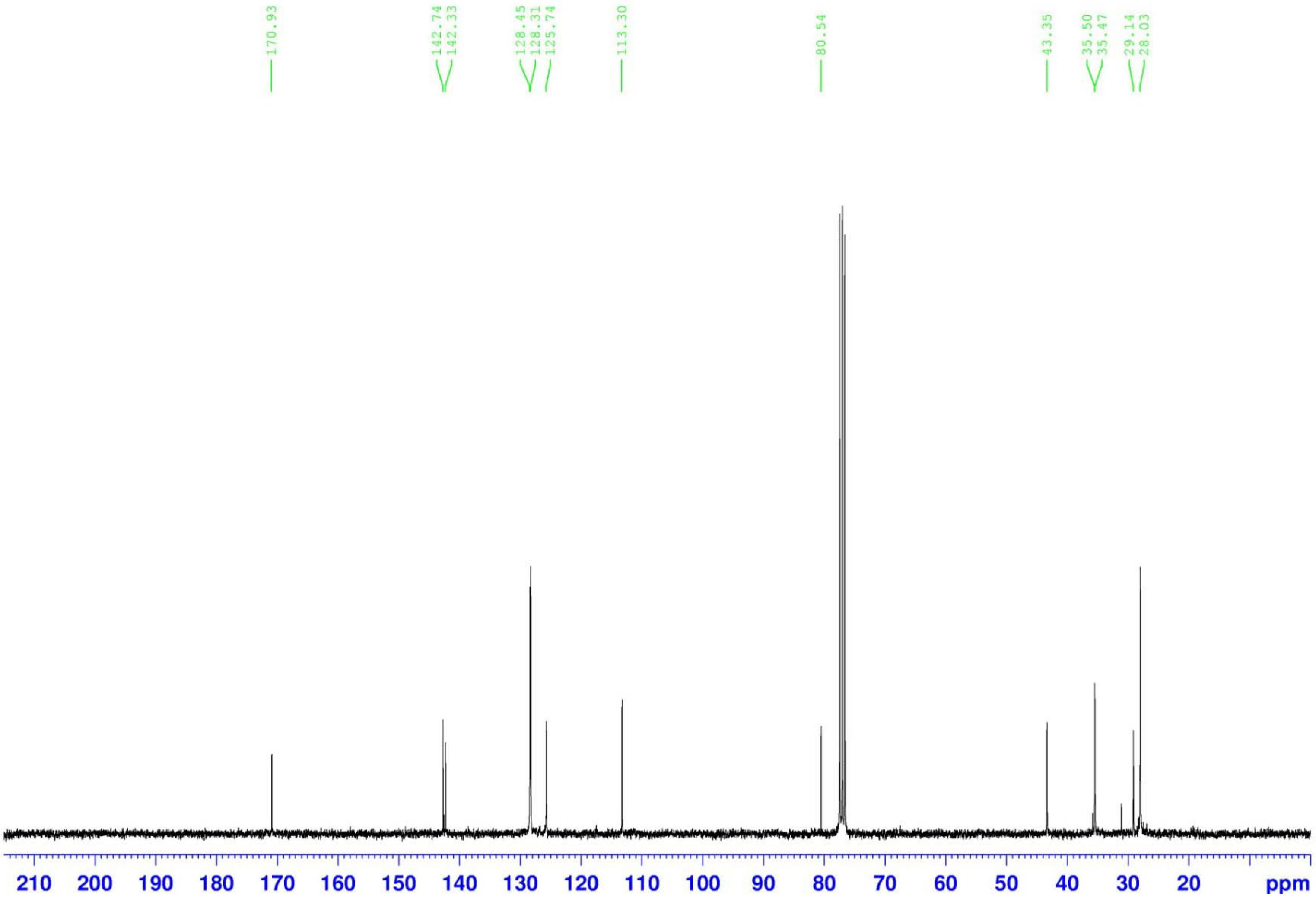
Following the general procedure for the preparation of *tert*-butyl esters through the synthesis of vinyl bromides followed by nickel-catalyzed substitution affords, after flash chromatography on silica gel (75–70:25–30 hexanes:dichloromethane), the title compound (57%, 2 steps) as a light yellow oil.

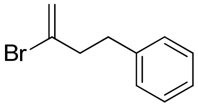
TLC analysis	R_f 0.60 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.35–7.25 (2H, m, l,l’), 7.25–7.15 (3H, m, k,k’,m), 4.94 and 4.93 (2H, s’s, f), 2.97 (2H, s, d), 2.65 (2H, t, J = 7.6 Hz, i), 2.17 (2H, t, J = 7.5 Hz, g), 1.90–1.75 (2H, m, h), 1.46 (9H, s, a,a’,a”).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 170.93 (c), 142.74 (j), 142.33 (e), 128.45 (k,k’), 128.31 (l,l’), 125.74 (m), 113.30 (f), 80.54 (b), 43.35 (d), 35.50 (g), 35.47 (i), 29.14 (h), 28.03 (a,a’,a”).
IR (neat)	3026, 2933, 2863, 1726 (C=O stretch), 1645, 1496, 1366, 1255 (C-O stretch), 1140, 897, 839, 744, 695 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{17}H_{24}NaO_2$ (M+Na): 283.1674, found 283.1682 *m/z*.

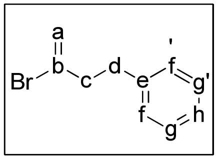
^{1}H NMR of 3-methylidene-6-phenylhexanoic acid *tert*-butyl ester



^{13}C NMR of 3-methylidene-6-phenylhexanoic acid *tert*-butyl ester



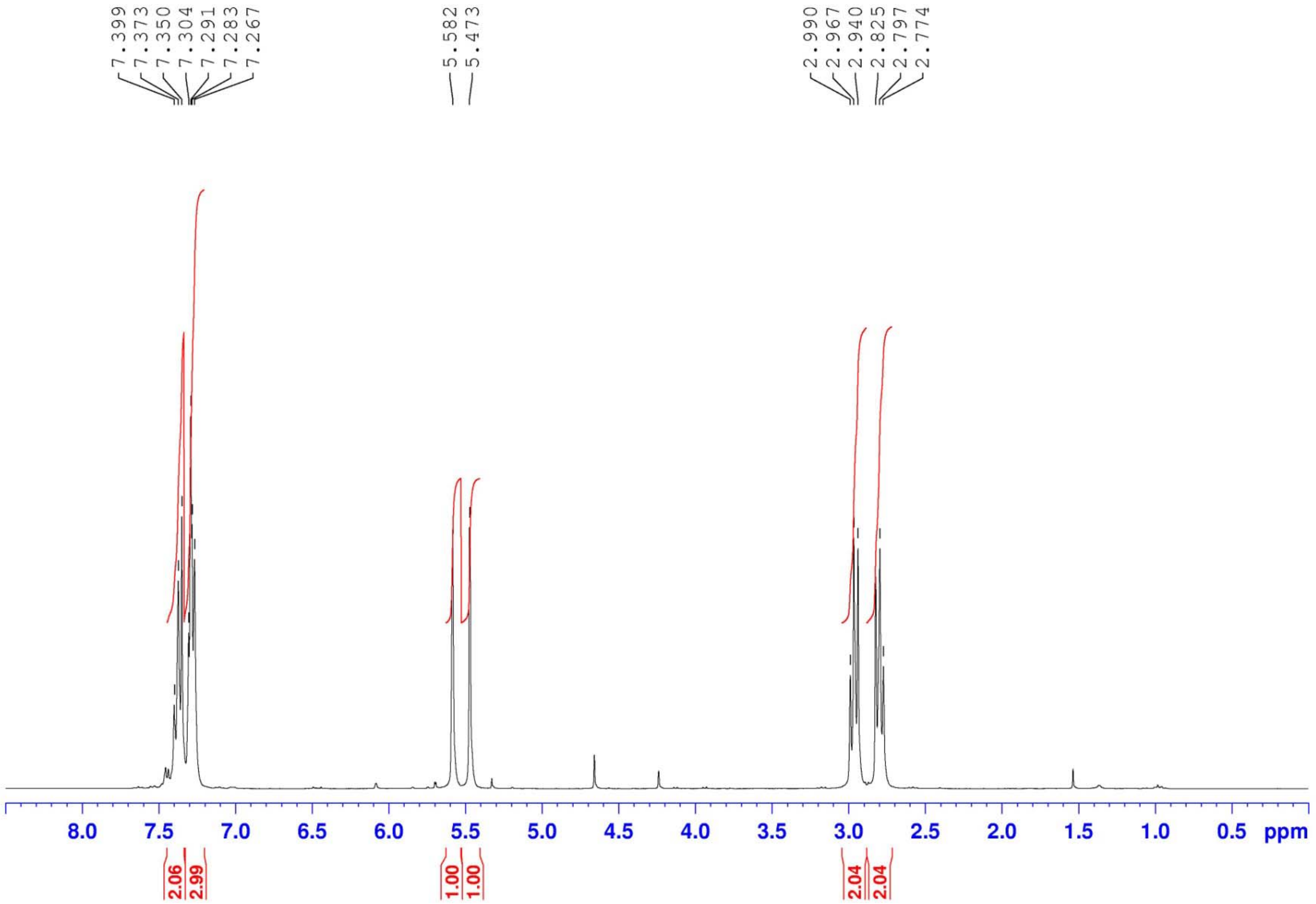




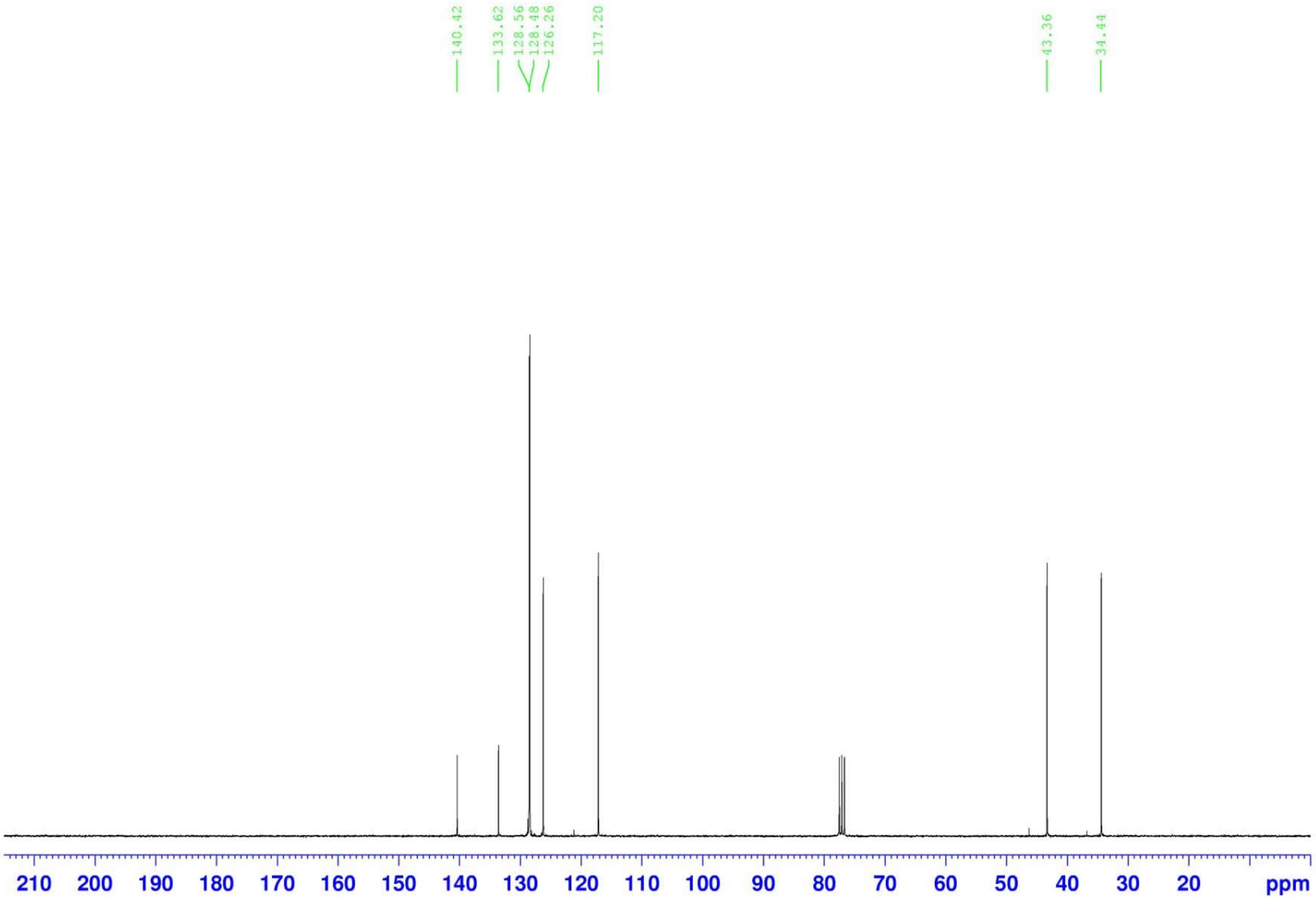
Following the general procedure for the preparation of vinyl bromides by the direct insertion of magnesium into benzylicchlorides affords, after flash chromatography on silica gel (hexanes), the title compound (55%) as a light brown oil.

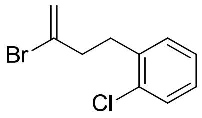
TLC analysis	*R*R_f 0.7 (hexanes)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.37 (2H, t, J = 7.9 Hz, g,g’), 7.30–7.20 (3H, m, f,f’,h), 5.58 (1H, s, a), 5.47 (1H, s, a), 2.97 (2H, t, J = 6.9 Hz, c), 2.80 (2H, t, J = 8.1 Hz, d).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 140.42 (e), 133.62 (b), 128.56 (f,f’), 128.48 (g,g’), 126.26 (h), 117.20 (a), 43.36 (c), 34.44 (d).
IR (neat)	3027, 2949, 2858, 1756, 1628, 1495, 1428, 1233, 1115, 1071 (C-Br stretch), 886, 766, 747, 696, 646, 559 cm^{-1}.

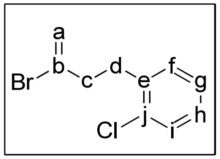
^{1}H NMR of 2-bromo-4-phenylbutene



^{13}C NMR of 2-bromo-4-phenylbutene



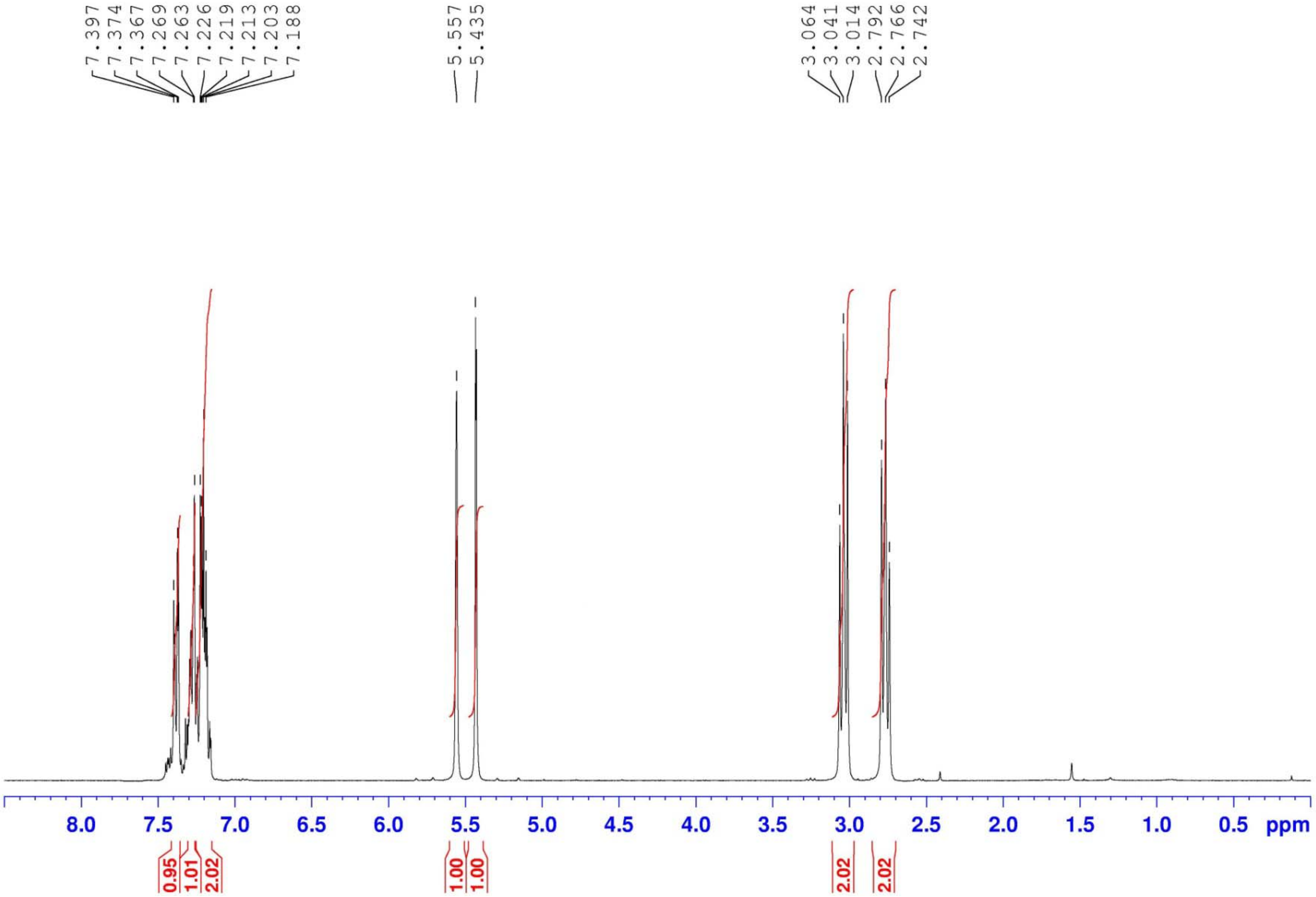




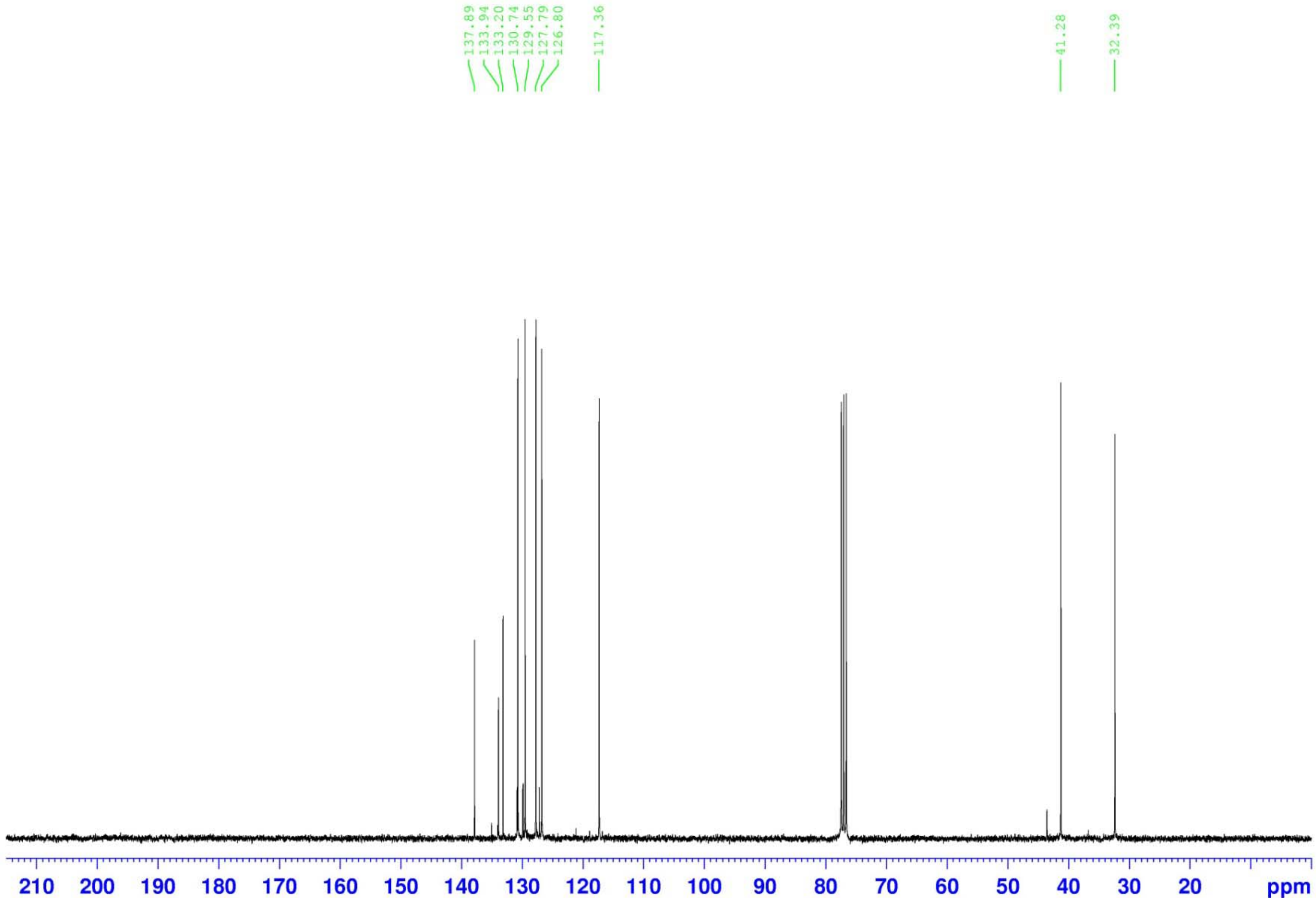
Following the general procedure for the preparation of vinyl bromides by the direct insertion of magnesium into benzylicchlorides affords, after flash chromatography on silica gel (hexanes), the title compound (59%) as a colorless oil.

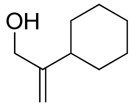
TLC analysis	R_f 0.70 (hexanes)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.40–7.35 (1H, m, i), 7.30–7.25 (1H, m, g), 7.25–7.15 (2H, m, f,h), 5.56 (1H, s, a), 5.44 (1H, s, a), 3.04 (2H, t, J = 7.2 Hz, c), 2.77 (2H, J = 8.0 Hz, d).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 137.89 (e), 133.94 (j), 133.20 (b), 130.74 (i), 129.55 (h), 127.79 (g), 126.80 (f), 117.36 (a), 41.28 (c), 32.39 (d).
IR (neat)	3027, 1628, 1474, 1282, 1189 (C-Cl stretch), 1052 (C-Br stretch), 1038, 890, 824, 751, 734, 684, 669, 550, 506 cm^{-1}.
HRMS (EI)	Calcd. for $C_{10}H_{10}BrCl$: 243.9654, found 243.9660 *m/z*.

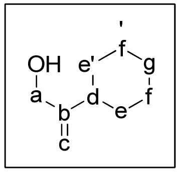
^{1}H NMR of 2-bromo-4-(2chlorophenyl)butene



^{13}C NMR of 2-bromo-4-(2chlorophenyl)butene



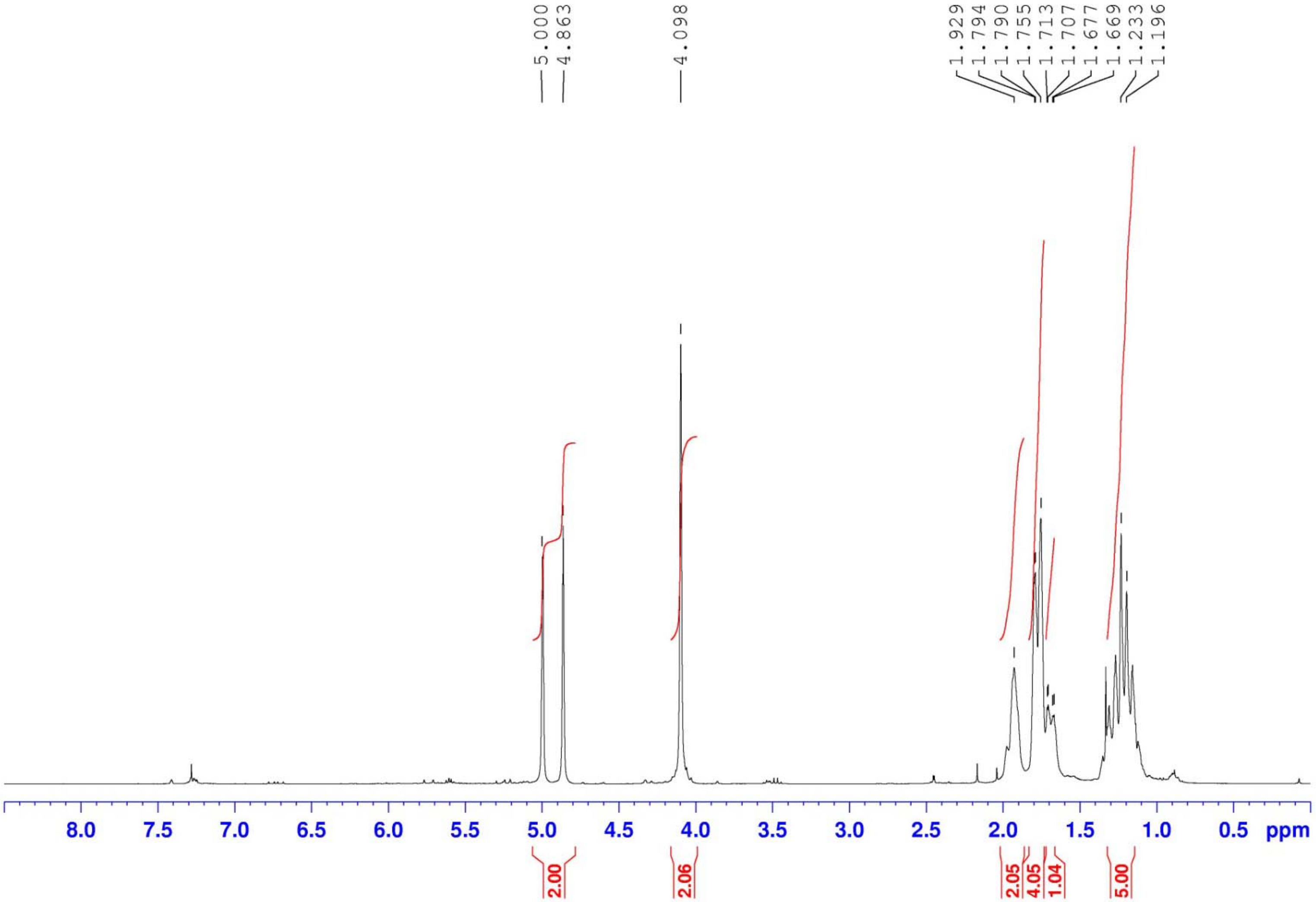




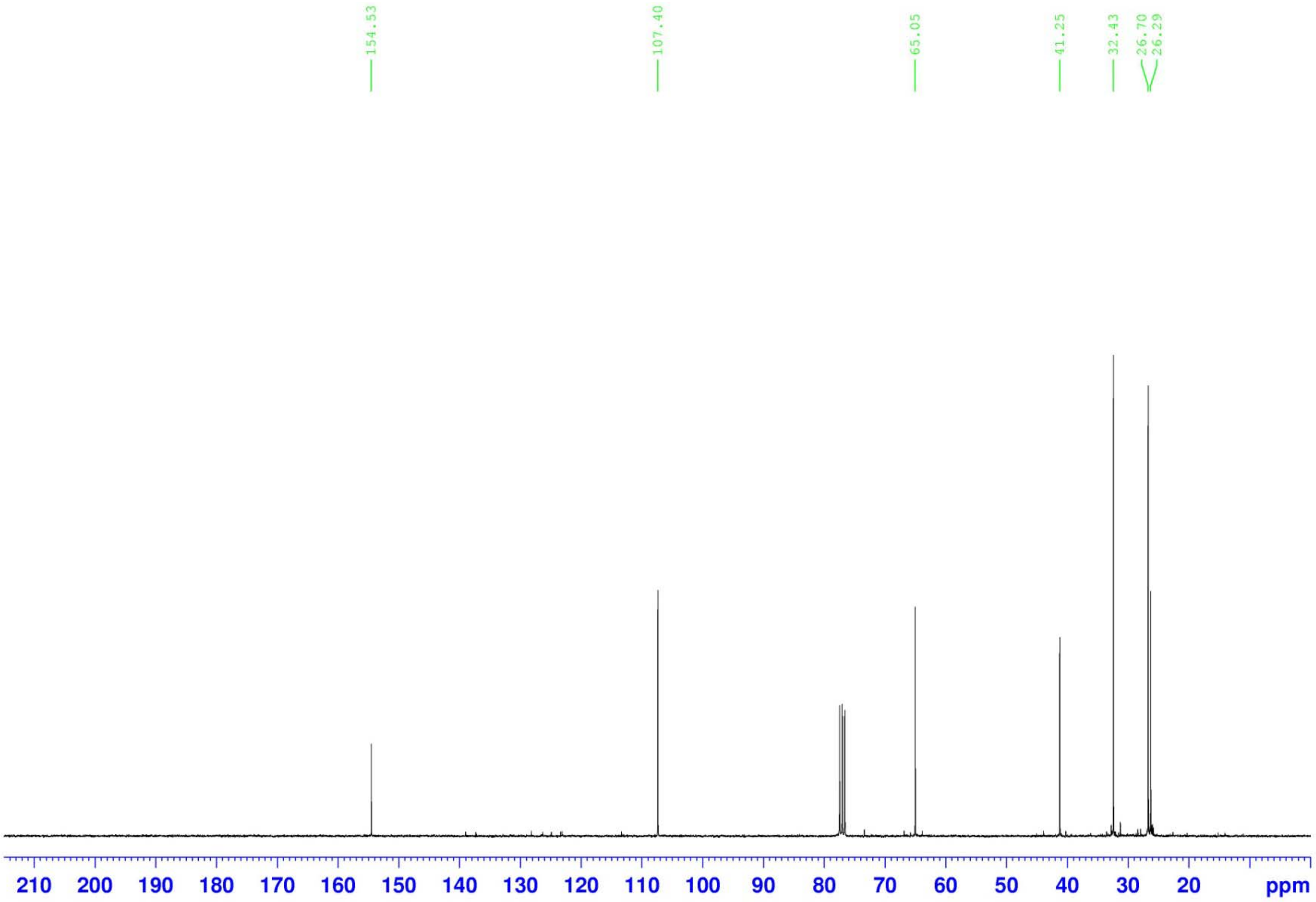
Following the general procedure for the preparation of allylic alcohols affords, after flash chromatography on silica gel (80:20 hexanes:ethyl acetate), the title compound (79%) as a colorless oil.

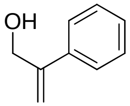
TLC analysis	*R_f* 0.35 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 5.00 (1H, s, c), 4.86 (1H, s, c), 4.10 (2H, s, a), 2.00–1.85 (2H, m, a,OH), 1.85–1.70 (4H, m, e,e',f,f'), 1.70–1.65 (1H, m, g), 1.30–1.10 (5H, m, e,e',f,f',g).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 154.53 (b), 107.40 (c), 65.05 (a), 41.25 (d), 32.43 (e,e'), 26.70 (f,f'), 26.29 (g).
IR (neat)	3306 (O-H stretch), 2850, 1649, 1060, 1019 (C-O stretch), 889, 625 cm^{-1}.
HRMS (EI)	Calcd. for $C_9H_{16}O$: 140.1201, found 140.1204 *m/z*.

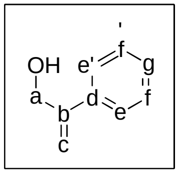
^{1}H NMR of 2-cyclohexyl-2-propenol



^{13}C NMR of 2-cyclohexyl-2-propenol



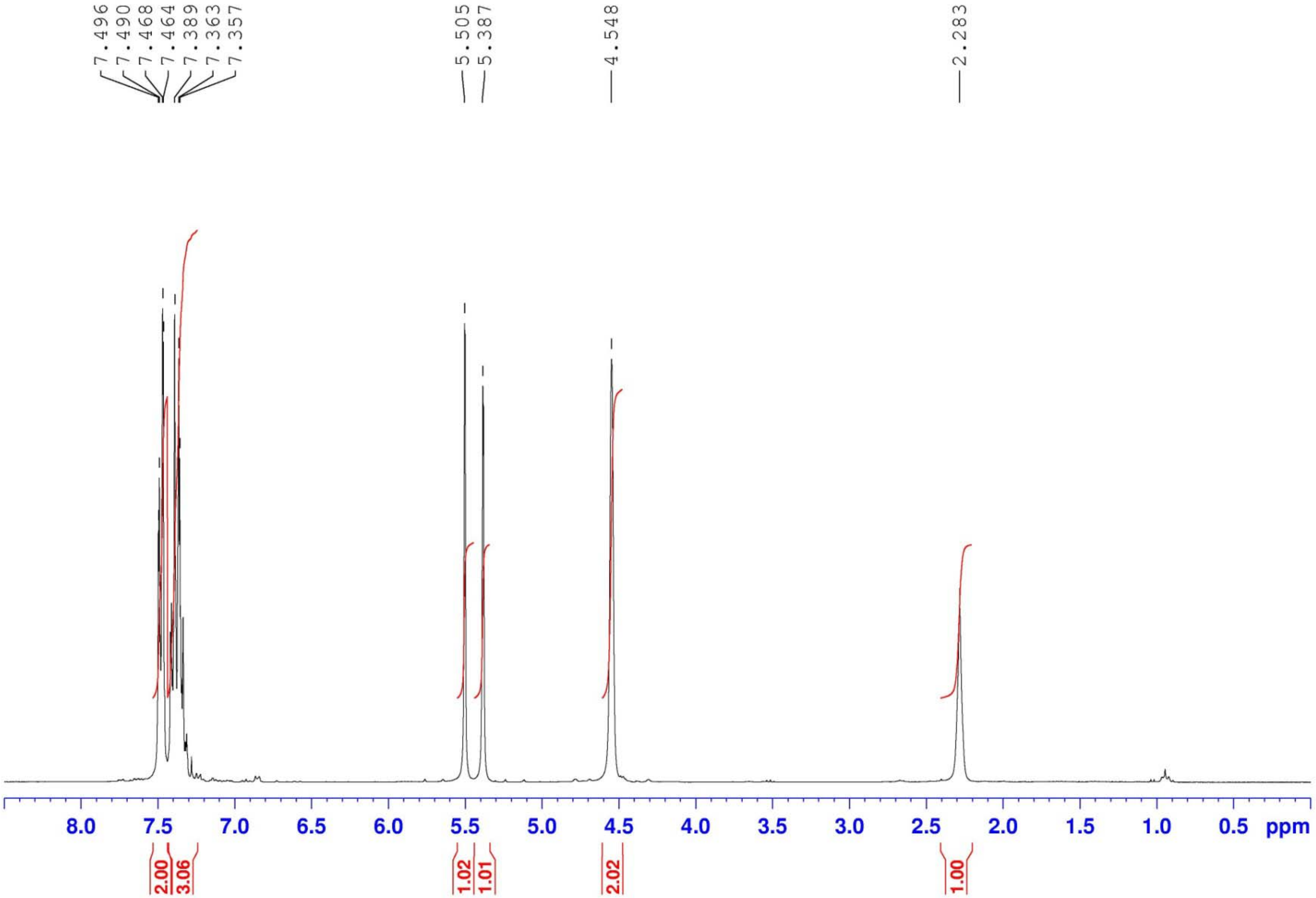




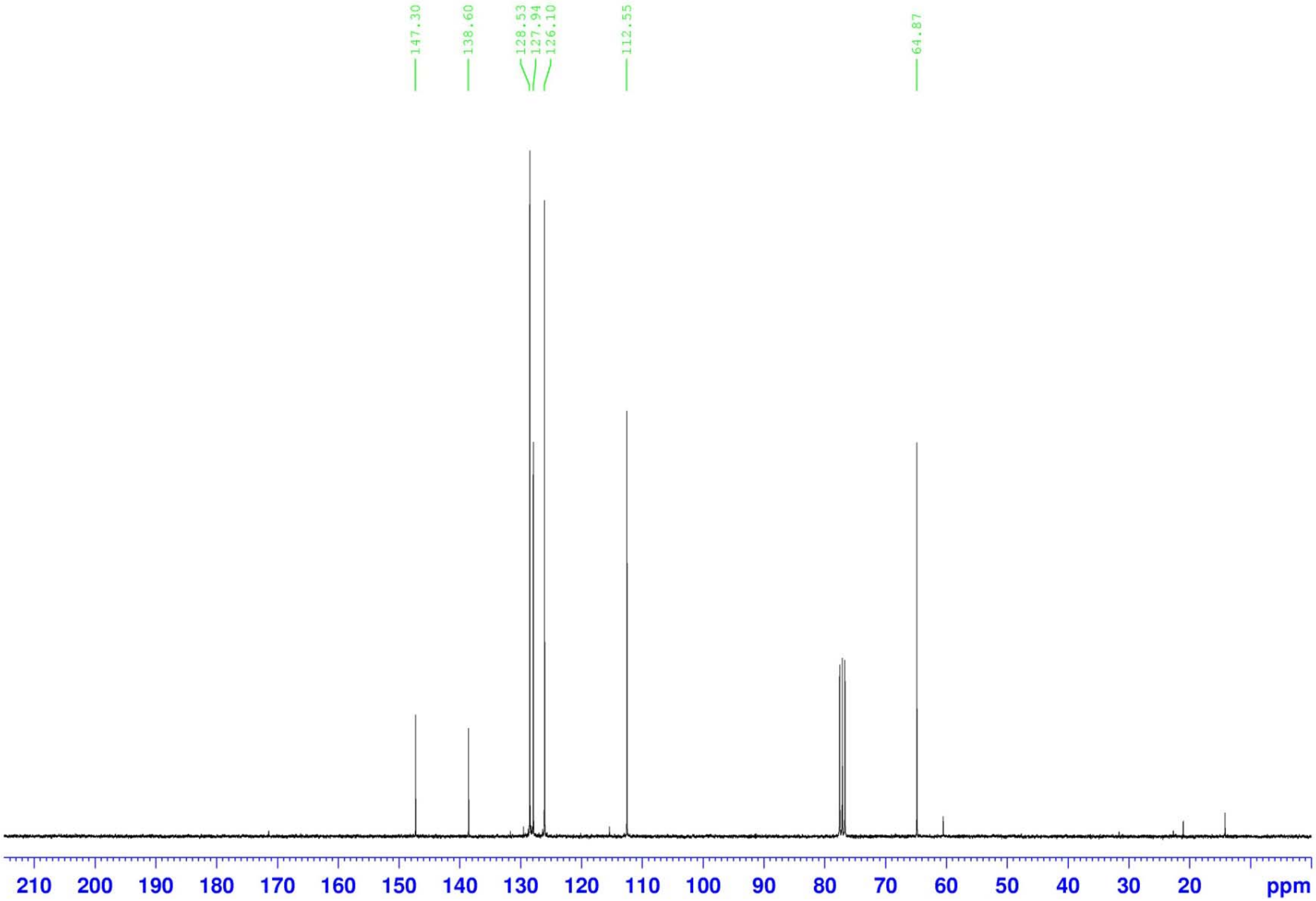
Following the general procedure for the preparation of allylic alcohols affords, after flash chromatography on silica gel (80:20 hexanes:ethyl acetate), the title compound (77%) as a light yellow oil.

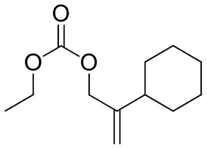
TLC analysis	R_f 0.30 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.55–7.45 (2H, m, e,e'), 7.45–7.30 (3H, m, f,f',g), 5.50 (1H, s, c), 5.39 (1H, s, c), 4.55 (2H, s, a), 2.28 (1H, br s, OH).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 147.30 (b), 138.60 (d), 128.53 (f,f'), 127.94 (g), 126.10 (e,e'), 112.55 (c), 64.87 (a).
IR (neat)	3370 (O-H stretch), 2945, 2883, 1735, 1632, 1495, 1444, 1372, 1239, 1043 (C-O stretch), 1024, 902, 778, 706, 609 cm^{-1}..

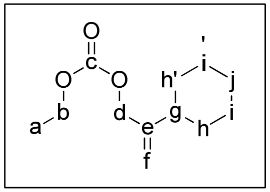
^{1}H NMR of 2-phenyl-2-propenol



^{13}C NMR of 2-phenyl-2-propenol



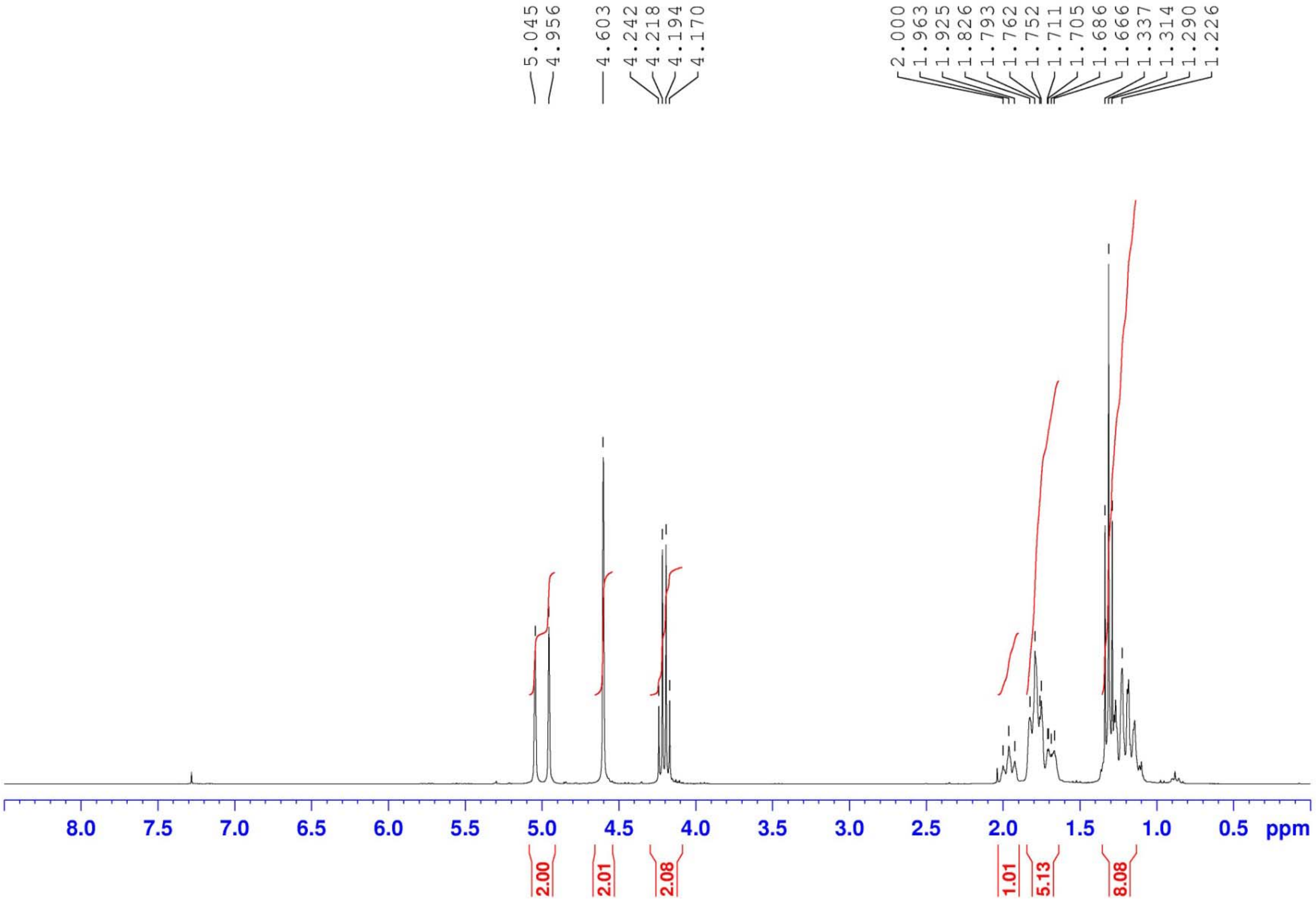




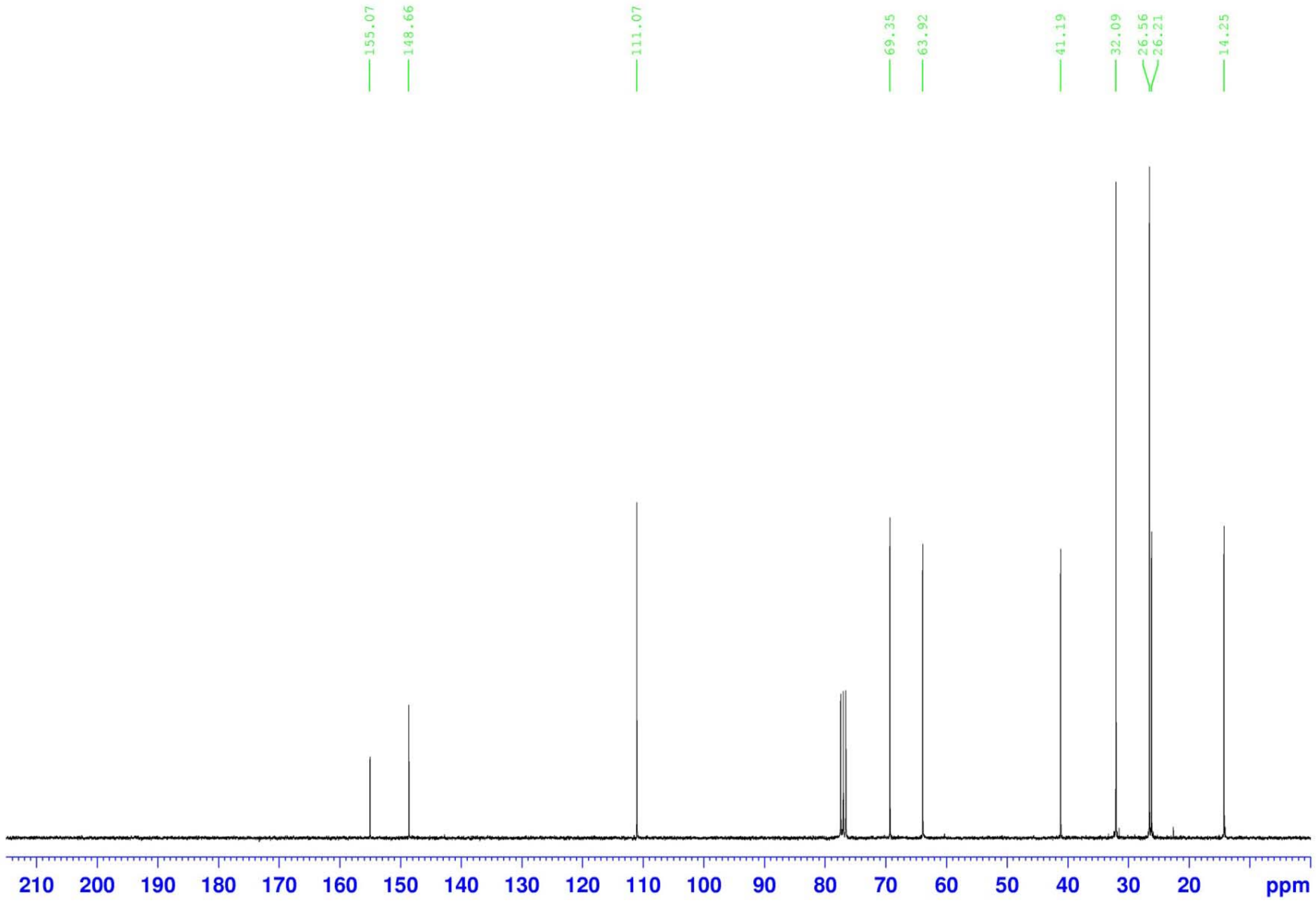
Following the general procedure for the preparation of allylic carbonates affords, after flash chromatography on silica gel (98–95:2–5 hexanes:ethyl acetate), the title compound (87%) as a colorless oil.

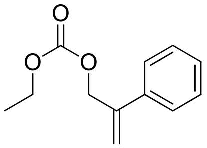
TLC analysis	R_f 0.75 (95:5 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 5.04 (1H, s, f), 4.96 (1H, s, f), 4.60 (2H, s, d), 4.20 (2H, q, J = 7.1 Hz, b), 2.00–1.90 (1H, m, g), 1.90–1.75 (4H, m, h,h’,i,i’), 1.75–1.65 (1H, m, j), 1.31 (3H, t, J = 7.1 Hz, a), 1.30–1.10 (5H, m, h,h’,i,i’,j).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 155.07 (c), 148.66 (e), 111.07 (f), 69.35 (d), 63.92 (b), 41.19 (g), 32.09 (h,h’), 26.56 (i,i’), 26.21 (j), 14.25 (a).
IR (neat)	2926, 2853, 1742 (C=O stretch), 1649, 1448, 1374, 1241 (C-O stretch), 1004, 908, 890, 790, 630 cm^{-1}.
HRMS (CI)	Calcd. for $C_{12}H_{21}O_3$ (M+H): 213.1491, found 213.1493 *m/z*.

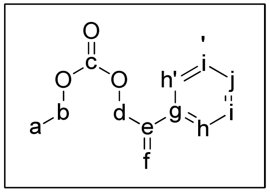
^{1}H NMR of 2-cyclohexylallyl ethyl carbonate



^{13}C NMR of 2-cyclohexylallyl ethyl carbonate



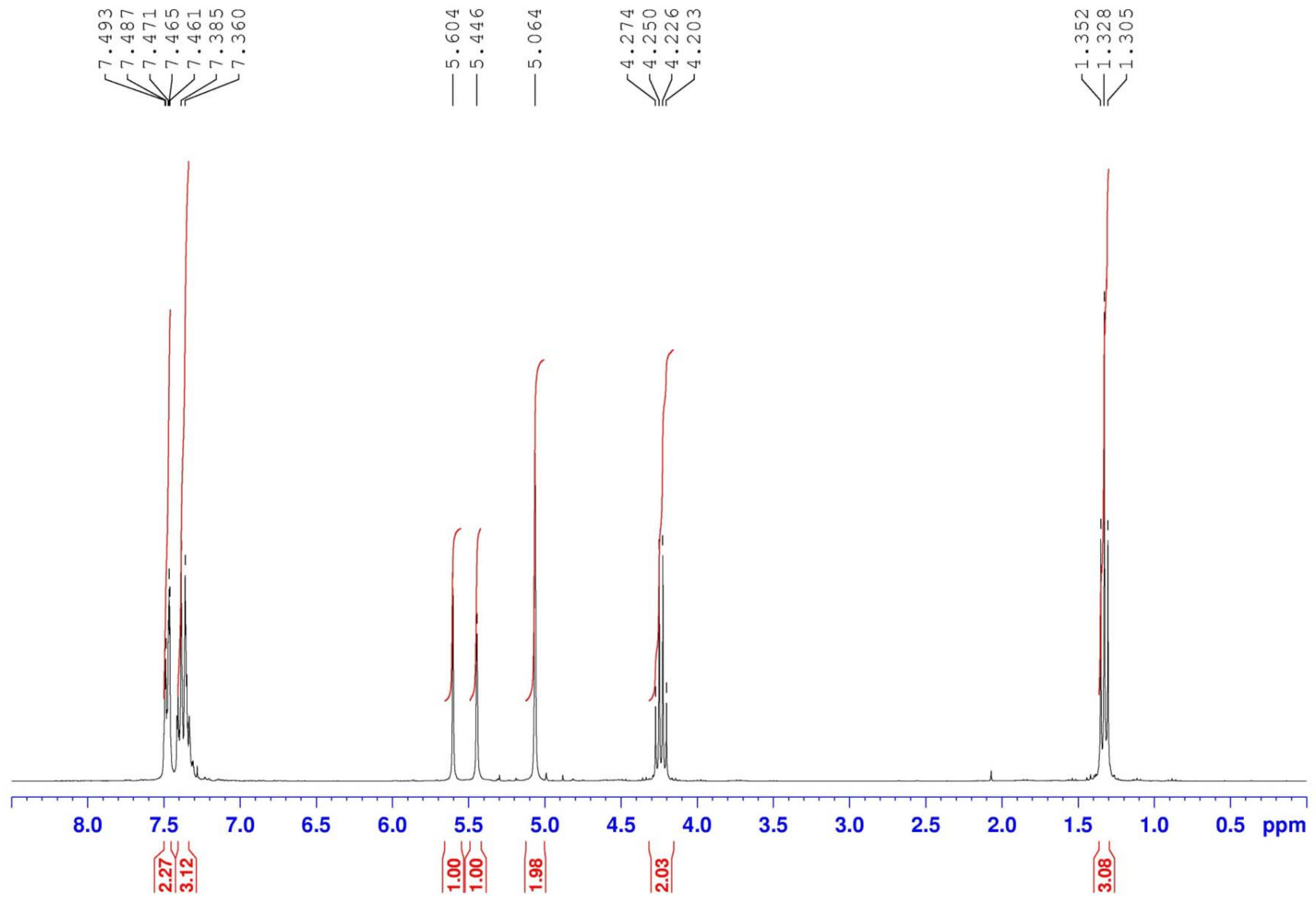




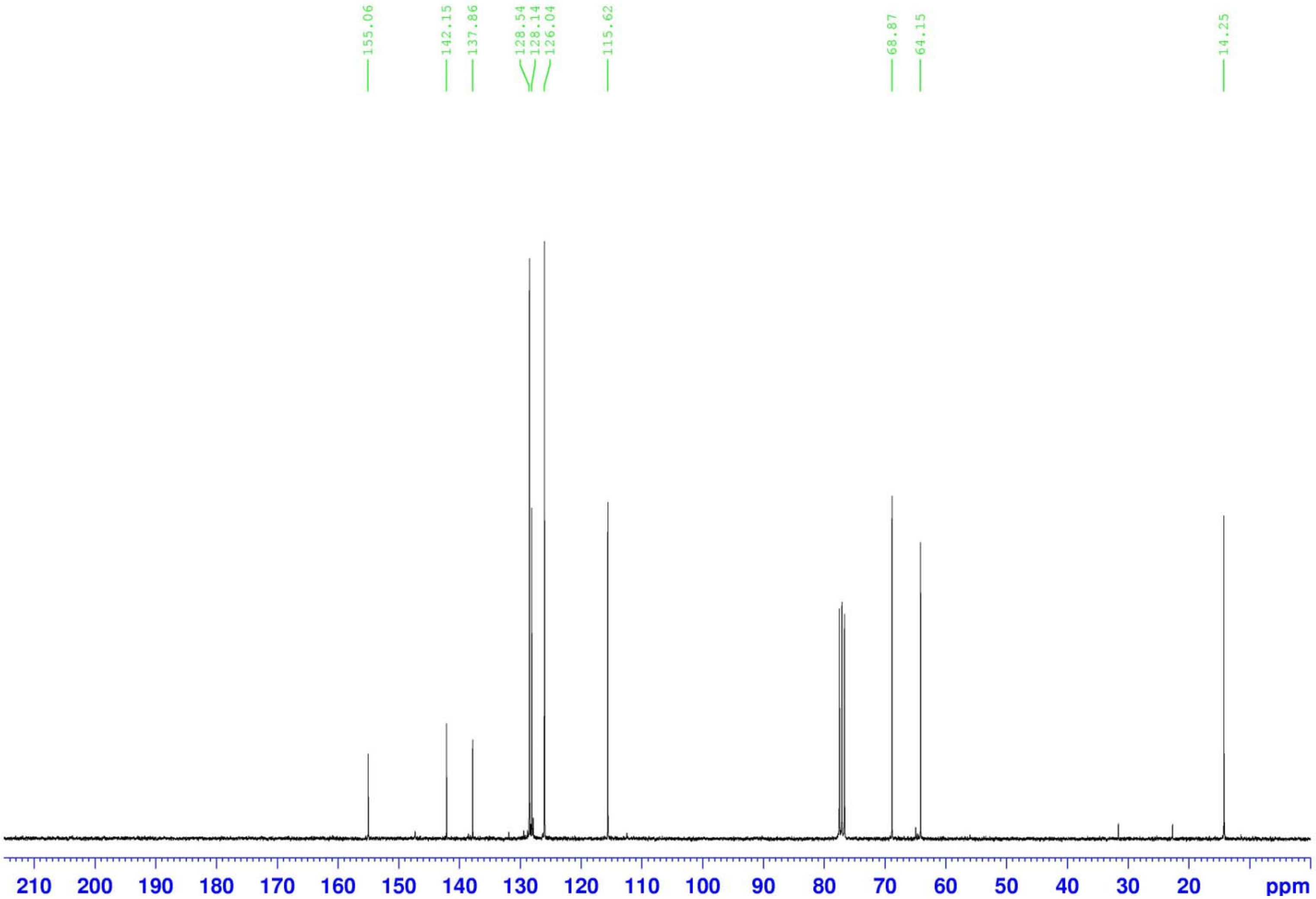
Following the general procedure for the preparation of allylic carbonates affords, after flash chromatography on silica gel ((98–95:2–5 hexanes:ethyl acetate), the title compound (84%) as a colorless oil.

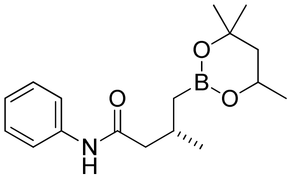
TLC analysis	*R*R_f 0.75 (95:5 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.50–7.45 (2H, m, h,h’), 7.45–7.30 (3H, m, i,i’,j), 5.60 (1H, s, f), 5.45 (1H, s, f), 5.06 (2H, s, d), 4.23 (2H, q, J = 7.1 Hz, b), 1.33 (3H, t, J = 7.1 Hz, a).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 155.06 (c), 142.15 (e), 137.86 (g), 128.54 (i,i’), 128.14 (j), 126.04 (h,h’), 115.62 (f), 68.87 (d), 64.15 (b), 14.25 (a).
IR (neat)	IR (neat) 2984, 1740 (C=O stretch), 1634, 1375, 1242 (C-O stretch), 1006, 910, 872, 789, 705, 547 cm^{-1}.

^{1}H NMR of 2-phenylallyl ethyl carbonate

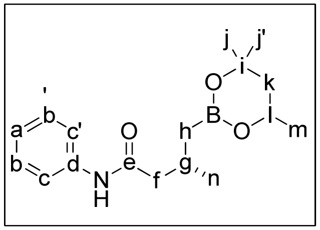


^{13}C NMR of 2-phenylallyl ethyl carbonate





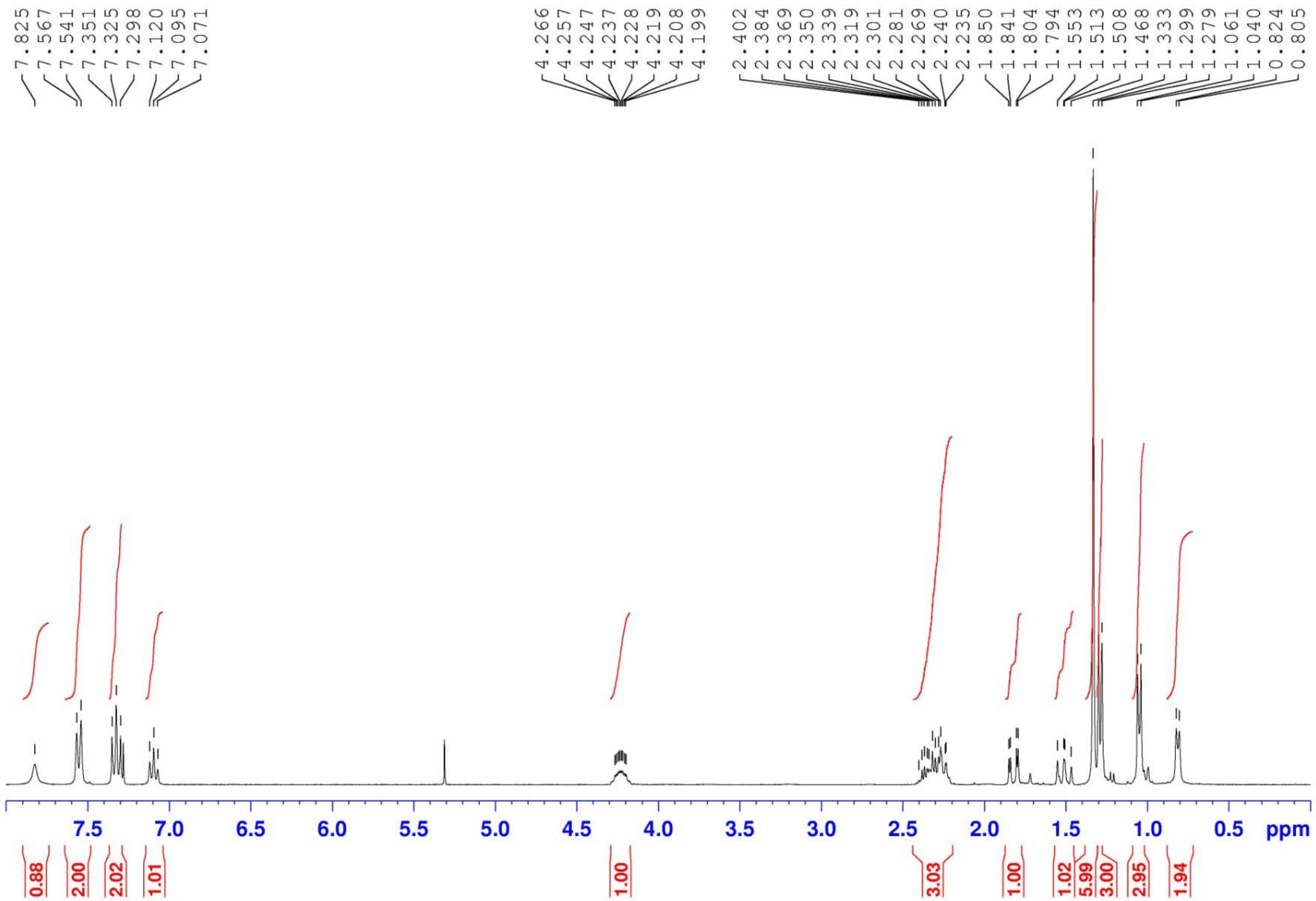
(*S*)-**8a**



After following the general procedure for the directed CAHB of **7a**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (53%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -18.8^{o}$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.82 (1H, br s, NH), 7.56 (2H, d, *J* = 7.9 Hz, c,c'), 7.33 (2H, t, *J* = 7.8 Hz, b,b'), 7.11 (1H, t, *J* = 7.4 Hz, a), 4.30–4.20 (1H, m, l), 2.40–2.20 (3H, m, g,f), 1.82 (1H, dd, *J* = 14.0 Hz, 2.9 Hz, k), 1.51 (1H, dd, *J* = 13.6 Hz, 11.8 Hz, k), 1.33 (6H, s, j,j'), 1.29 (3H, d, *J* = 6.2 Hz, m), 1.05 (3H, d, *J* = 6.2 Hz, n), 0.81 (2H, d, *J* = 5.7 Hz, h).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 171.38 (e), 138.33 (d), 128.92 (b,b'), 123.85 (a), 119.62 (c,c'), 71.02 (i), 64.79 (l), 46.97 (k), 45.89 (f), 31.35 (h), 28.38 (j,j'), 28.20 (m), 23.24 (n), 22.85 (g).
IR (neat)	3301 (N-H stretch), 2972, 1660 (C=O stretch), 1600, 1554 (N-H bend), 1499, 1442, 1390, 1302 (C-O stretch), 1209 (C-N stretch), 1161, 755, 692 cm^{-1}.
HRMS (EI)	Calcd. for $C_{17}H_{26}BNO_3$: 303.2006, found 303.1996 *m/z*.

^{1}H NMR of (*S*)-8a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4.237
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4.219
4.208
4.199
2.402
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2.369
2.350
2.339
2.319
2.301
2.281
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2.240
2.235
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1.794
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1.508
1.468
1.333
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0.805
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7.0
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4.5
4.0
3.5
3.0
2.5
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1.0
0.5
ppm
0.88
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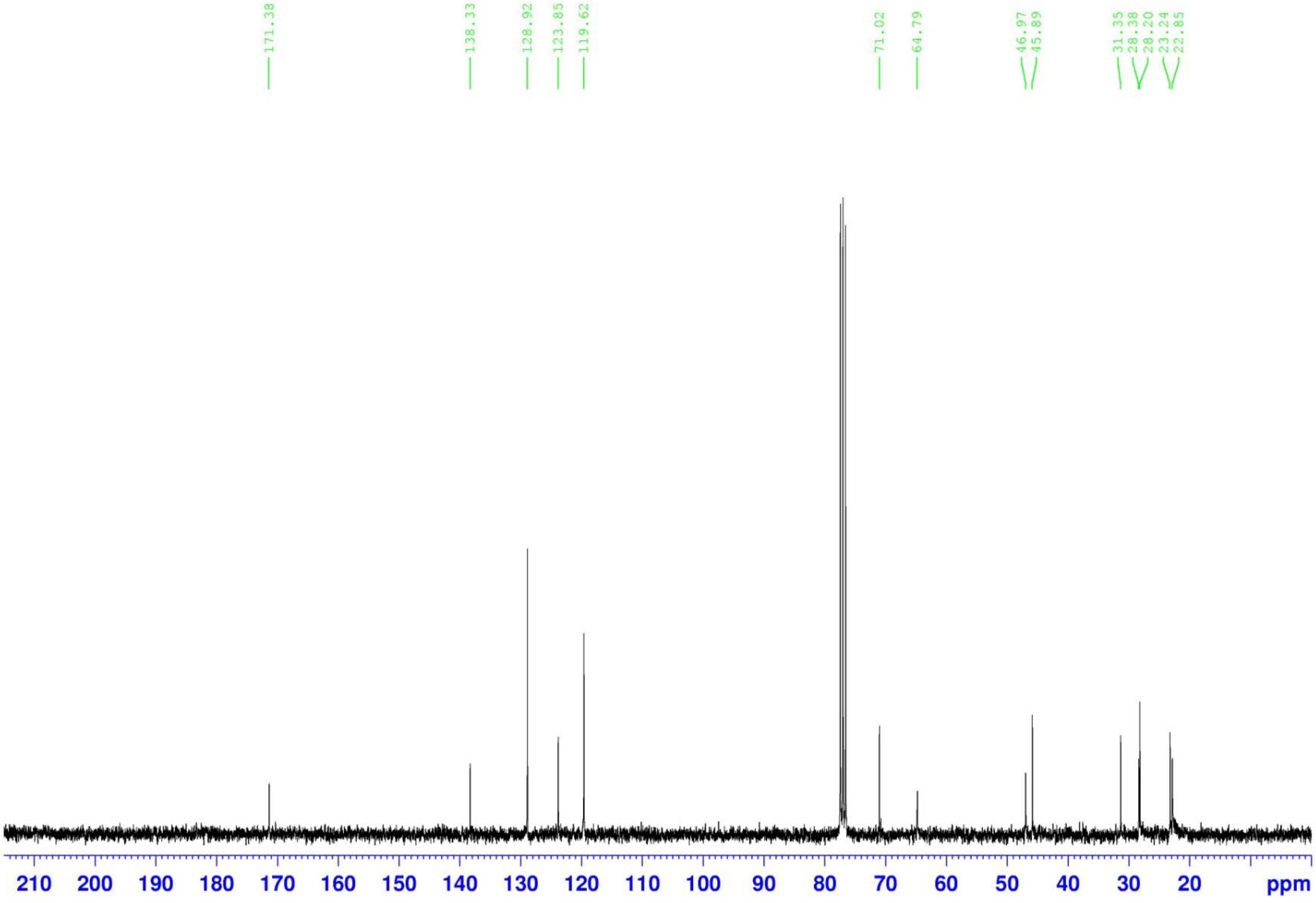
3.00
2.95
1.94

^{13}C NMR of XX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22.85
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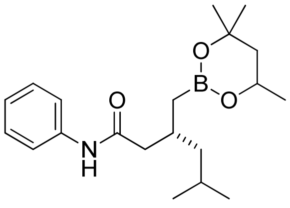
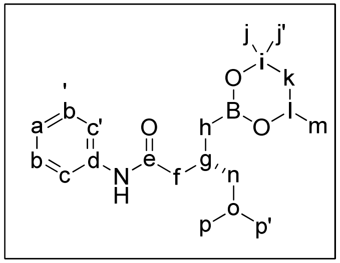


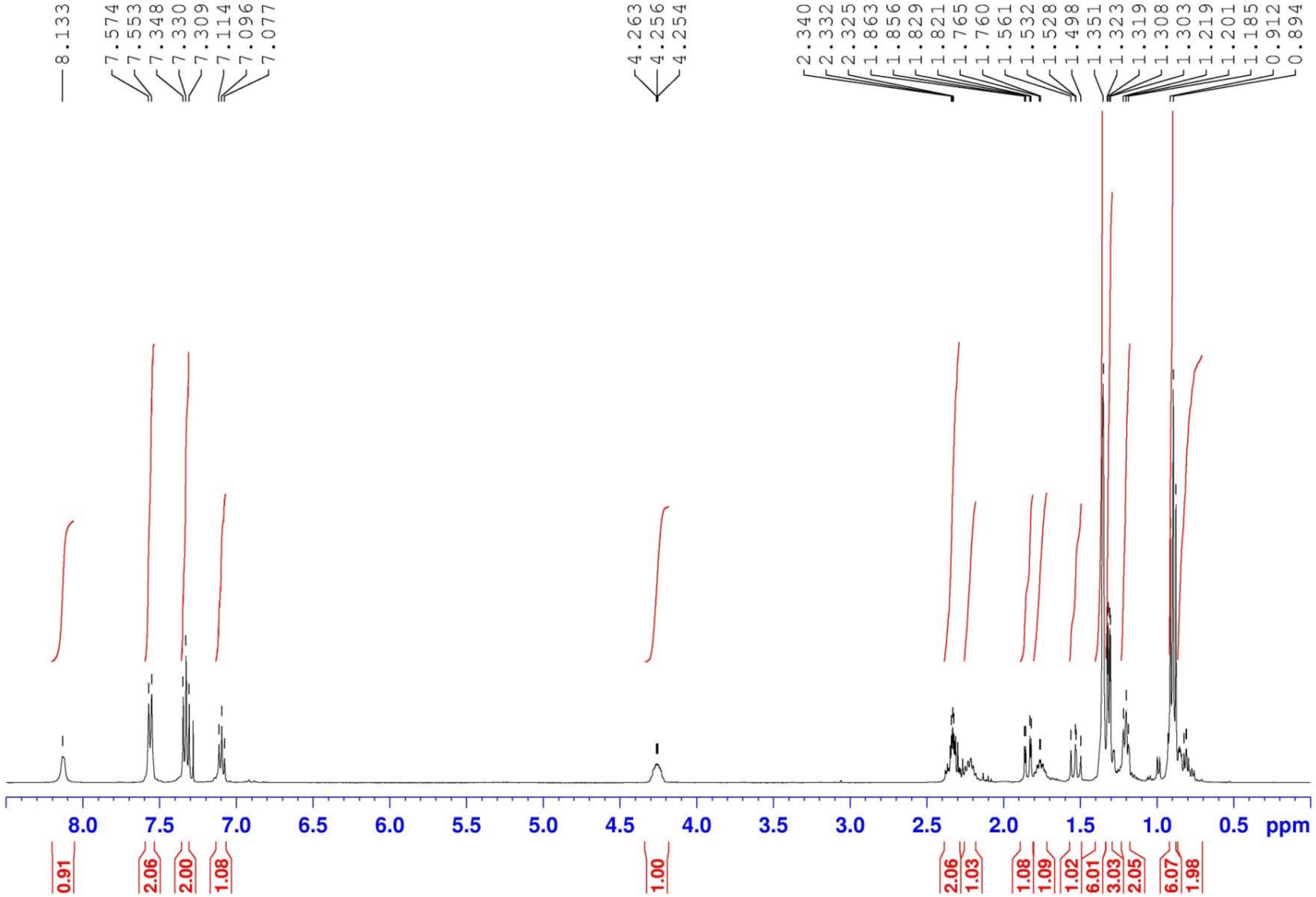
(*R*)-**4**



After following the general procedure for the directed CAHB of **3**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (72%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -17.9^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 8.13 (1H, br s, NH), 7.56 (2H, d, *J* = 8.3 Hz, c,c'), 7.33 (2H, t, *J* = 7.9 Hz, b,b'), 7.10 (1H, t, *J* = 7.4 Hz, a), 4.30–4.20 (1H, m, l), 2.40–2.25 (2H, m, f), 2.25–2.15 (1H, m, g), 1.84 (1H, dd, *J* = 13.9 Hz, 2.9 Hz, k), 1.80–1.70 (1H, m, o), 1.53 (1H, dd, *J* = 13.7 Hz, 11.8 Hz, k), 1.35 (6H, s, j,j'), 1.31 (3H, dd, *J* = 6.2 Hz, 2.0 Hz, m), 1.20 (2H, t, *J* = 6.8 Hz, n), 0.95–0.85 (6H, t, *J* = 7.1 Hz, p), 0.90–0.70 (2H, m, h).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.62 (e), 138.45 (d), 128.91 (b,b'), 123.77 (a), 119.61 (c,c'), 71.21 and 71.16 (i), 64.90 (l), 46.75 (n), 45.88 (k), 44.99 (f), 31.40 (h), 30.96 (g), 28.26 and 28.20 (j,j'), 25.14 and 25.11 (o), 23.30 and 23.28 (m), 23.08, 23.06, and 23.34 (p,p').
IR (neat)	3304 (N-H stretch), 2954, 1659 (C=O stretch), 1600, 1544 (N-H bend), 1499, 1442, 1389, 1302 (C-O stretch), 1208 (C-N stretch), 1161, 755 cm^{-1}.
HRMS (EI)	Calcd. for $C_{20}H_{32}BNO_3$: 345.2475, found 345.2486 *m/z*.

^{1}H NMR of (*R*)-4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2.332
2.325
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1.856
1.829
1.821
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1.760
1.561
1.532
1.528
1.498
1.351
1.323
1.319
1.308
1.303
1.219
1.201
1.185
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6.5
6.0
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4.0
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2.0
1.5
1.0
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ppm
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2.00
1.08
1.00
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6.01
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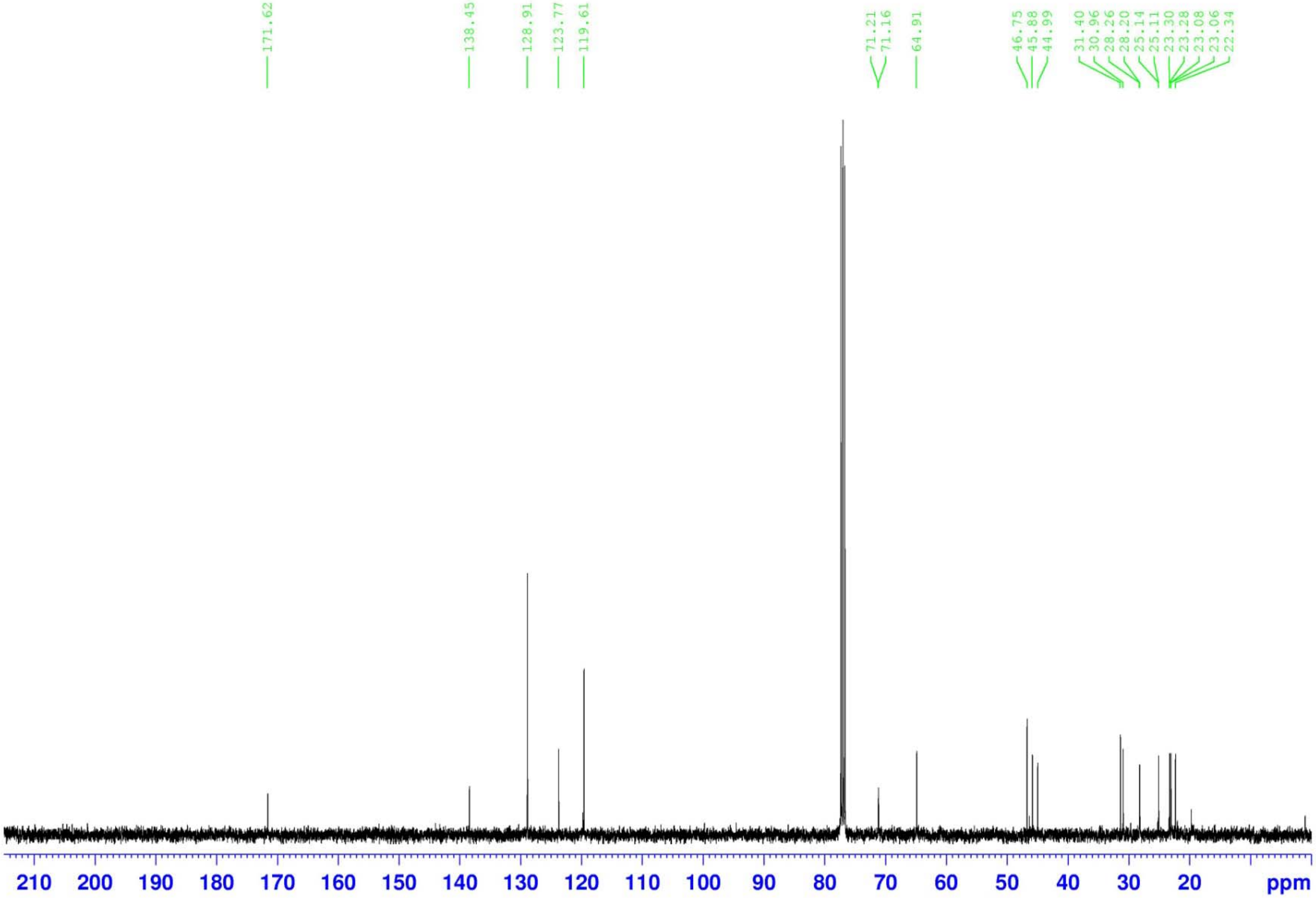
6.07
1.98

^{13}C NMR of (*R*)-4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71.21
71.16
64.91
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45.88
44.99
31.40
30.96
28.26
28.20
25.14
25.11
23.30
23.28
23.08
23.06
22.34
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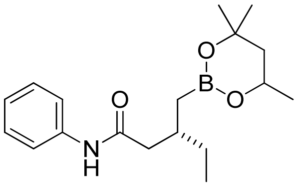
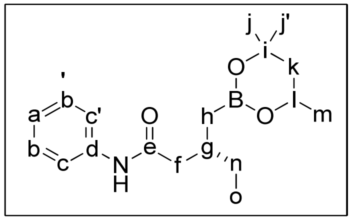


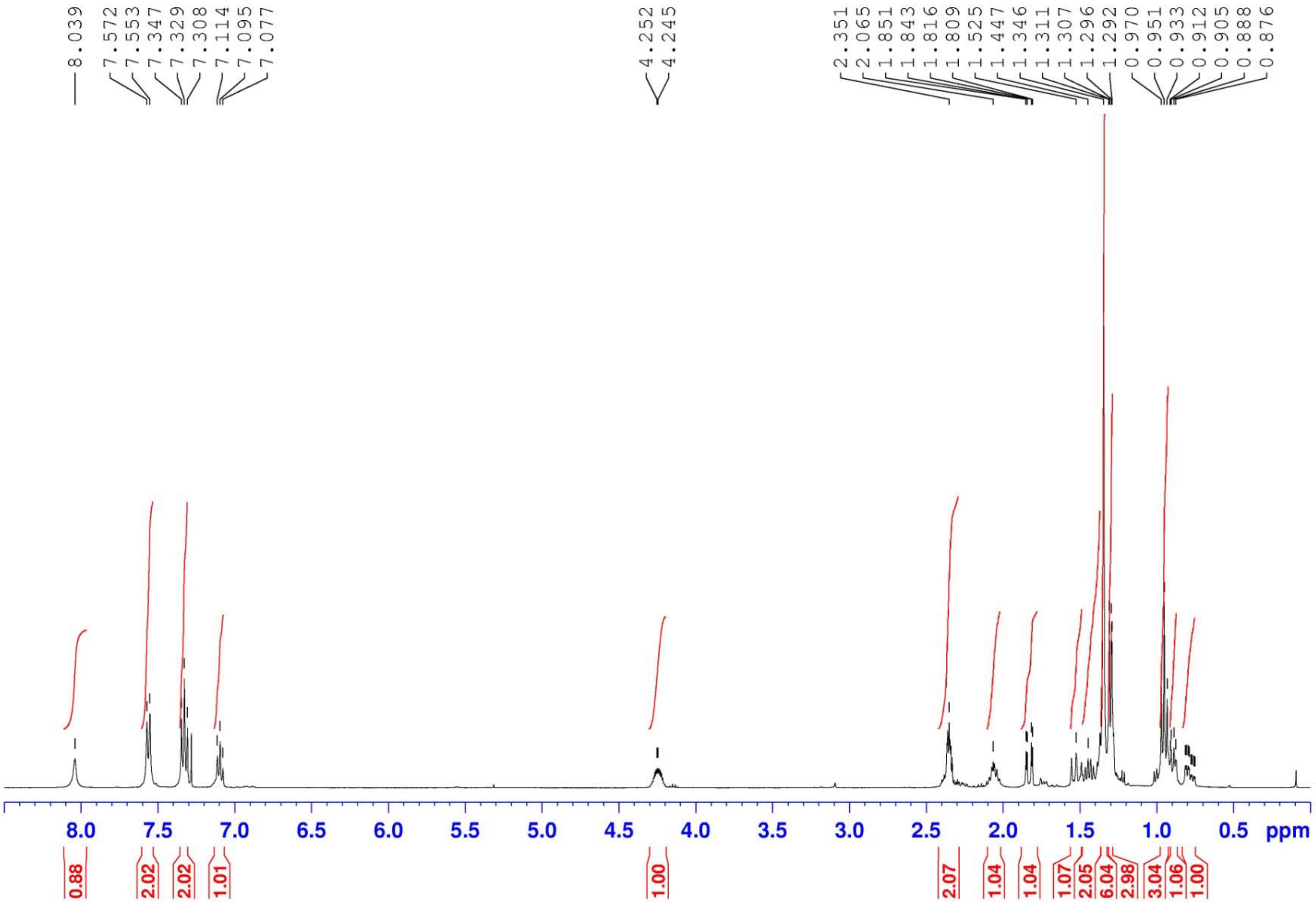
(*R*)-**8b**



After following the general procedure for the directed CAHB of **7b**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (60%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -22.4° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 8.04 (1H, br s, NH), 7.56 (2H, d, *J* = 8.0 Hz, c,c'), 7.33 (2H, t, *J* = 7.9 Hz, b,b'), 7.10 (1H, t, *J* = 7.4 Hz, a), 4.30–4.20 (1H, m, l), 2.40–2.30 (2H, m, f), 2.15–2.00 (1H, m, g), 1.83 (1H, dd, *J* = 14.0 Hz, 2.9 Hz, k), 1.52 (1H, dd, *J* = 13.8 Hz, 11.9 Hz, k), 1.50–1.35 (2H, m, n), 1.35 (6H, s, j,j'), 1.30 (3H, dd, *J* = 6.2 Hz, 1.5 Hz, m), 0.95 (3H, t, *J* = 7.4 Hz, o), 0.95–0.85 (1H, m, h), 0.85–0.75 (1H, m, h).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.73 (e), 138.46 (d), 128.89 (b,b'), 123.77 (a), 119.63 (c,c'), 71.13 and 71.09 (i), 64.86 (l), 45.84 (k), 44.34 (f), 34.88 (g), 31.37 (h), 29.92 (n), 28.22 and 28.18 (j,j'), 23.26 (m), 11.66 (o).
IR (neat)	3296 (N-H stretch), 2971, 2926, 1660 (C=O stretch), 1600, 1543 (N-H bend), 1499, 1441, 1389, 1302 (C-O stretch), 1209 (C-N stretch), 755 cm^{-1}.
HRMS (EI)	Calcd. for $C_{18}H_{28}BNO_3$: 317.2162, found 317.2172 *m/z*.

^{1}H NMR of (*R*)-8b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1.843
1.816
1.809
1.525
1.447
1.346
1.311
1.307
1.296
1.292
0.970
0.951
0.933
0.912
0.905
0.888
0.876
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ppm
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1.00
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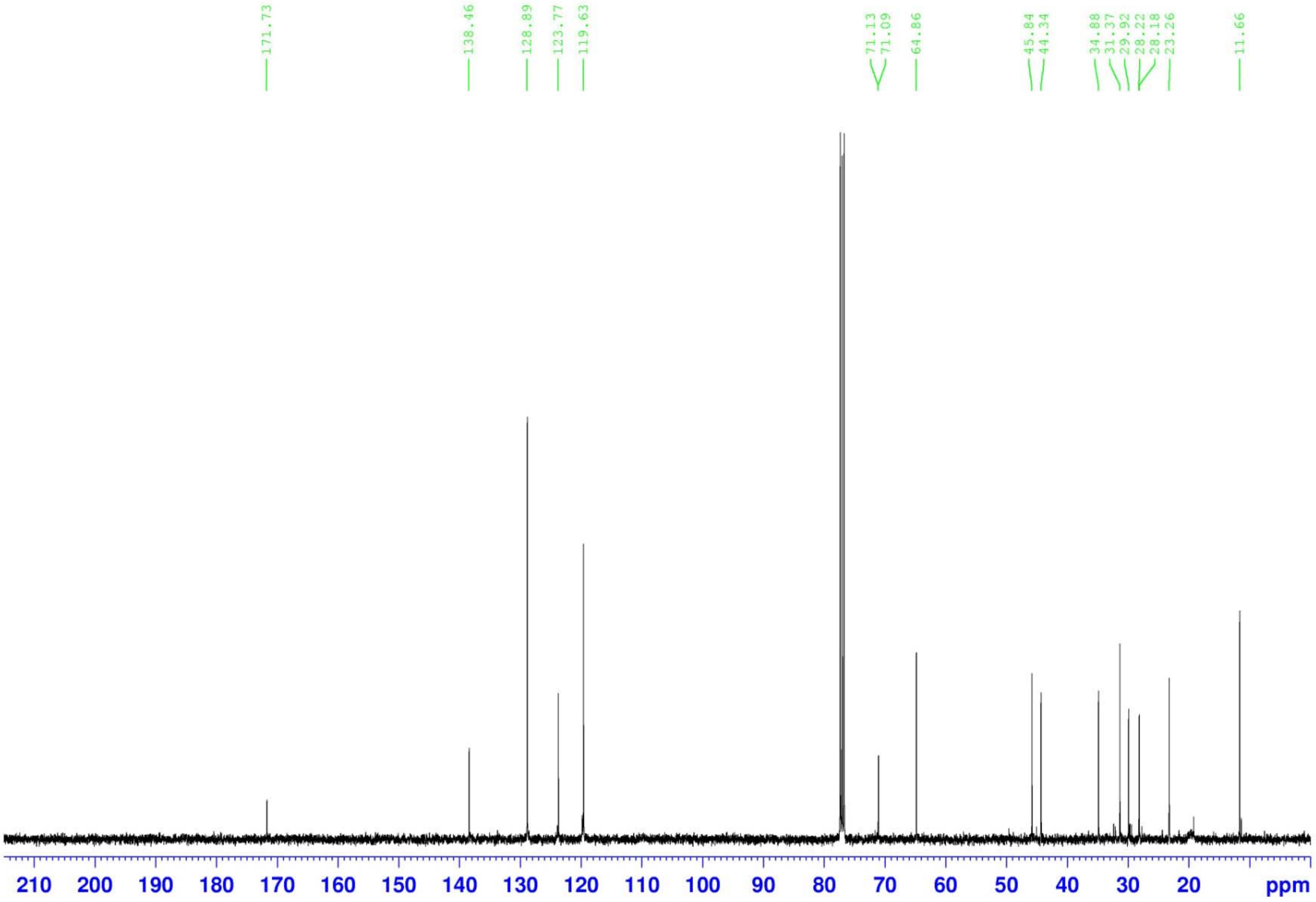
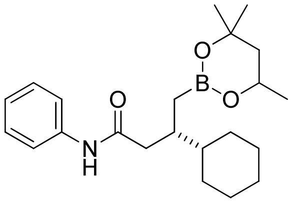
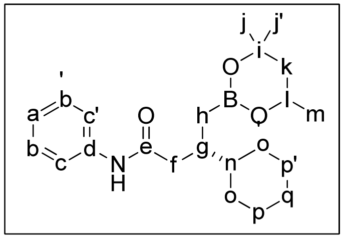
1.06
1.00

^{13}C NMR of (*R*)-8b





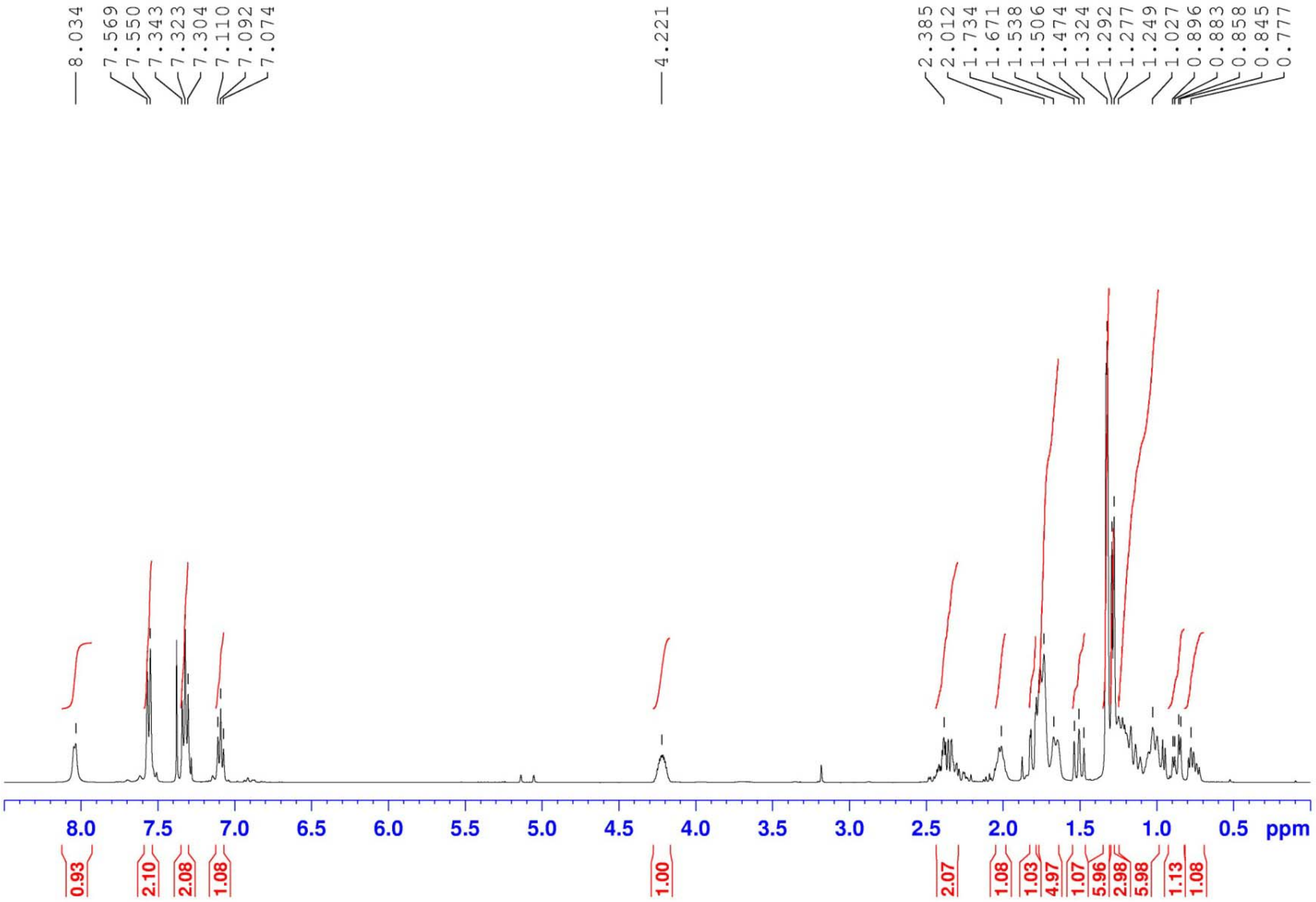
(*S*)-**8e**



After following the general procedure for the directed CAHB of **7e**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (72%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -16.4° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 8.03 (1H, br s, NH), 7.56 (2H, d, *J* = 7.9 Hz, c,c'), 7.32 (2H, t, *J* = 7.9 Hz, b,b'), 7.09 (1H, t, *J* = 7.4 Hz, a), 4.30–4.15 (1H, m, l), 2.50–2.30 (2H, m, f), 2.10–2.00 (1H, m, g), 1.80 (1H, dd, *J* = 14.1 Hz, 2.6 Hz, k), 1.80–1.65 (5H, m, n,p,p'), 1.51 (1H, t, *J* = 12.8 Hz, k), 1.32 (6H, s, j,j'), 1.28 (3H, d, *J* = 6.0 Hz, m), 1.25–0.95 (6H, m, o,o',q), 0.87 (1H, dd, *J* = 15.3 Hz, 5.2 Hz, h), 0.80–0.70 (1H, m, h).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.35 (e), 138.48 (d), 128.90 (b,b'), 123.76 (a), 119.64 (c,c'), 71.09 and 71.00 (i), 64.86 and 64.80 (l), 45.78 (k), 43.29 and 43.26 (n), 42.25 and 42.21 (f), 38.48 (g), 31.36 (h), 30.49 (o,o'), 29.81, 29.70, 28.16, and 28.08 (j,j'), 26.73 (p,p'), 26.68 (q), 23.25 (m).
IR (neat)	3298 (N-H stretch), 3255, 2928, 1656 (C=O stretch), 1595 (N-H bend), 1414, 1318 (C-O stretch), 1246, 1206 (C-N stretch), 1141, 755 cm^{-1}.
HRMS (EI)	Calcd. for $C_{22}H_{34}BNO_3$: 371.2632, found 371.2627 *m/z*.

^{1}H NMR of (*S*)-8e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1.324
1.292
1.277
1.249
1.027
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0.858
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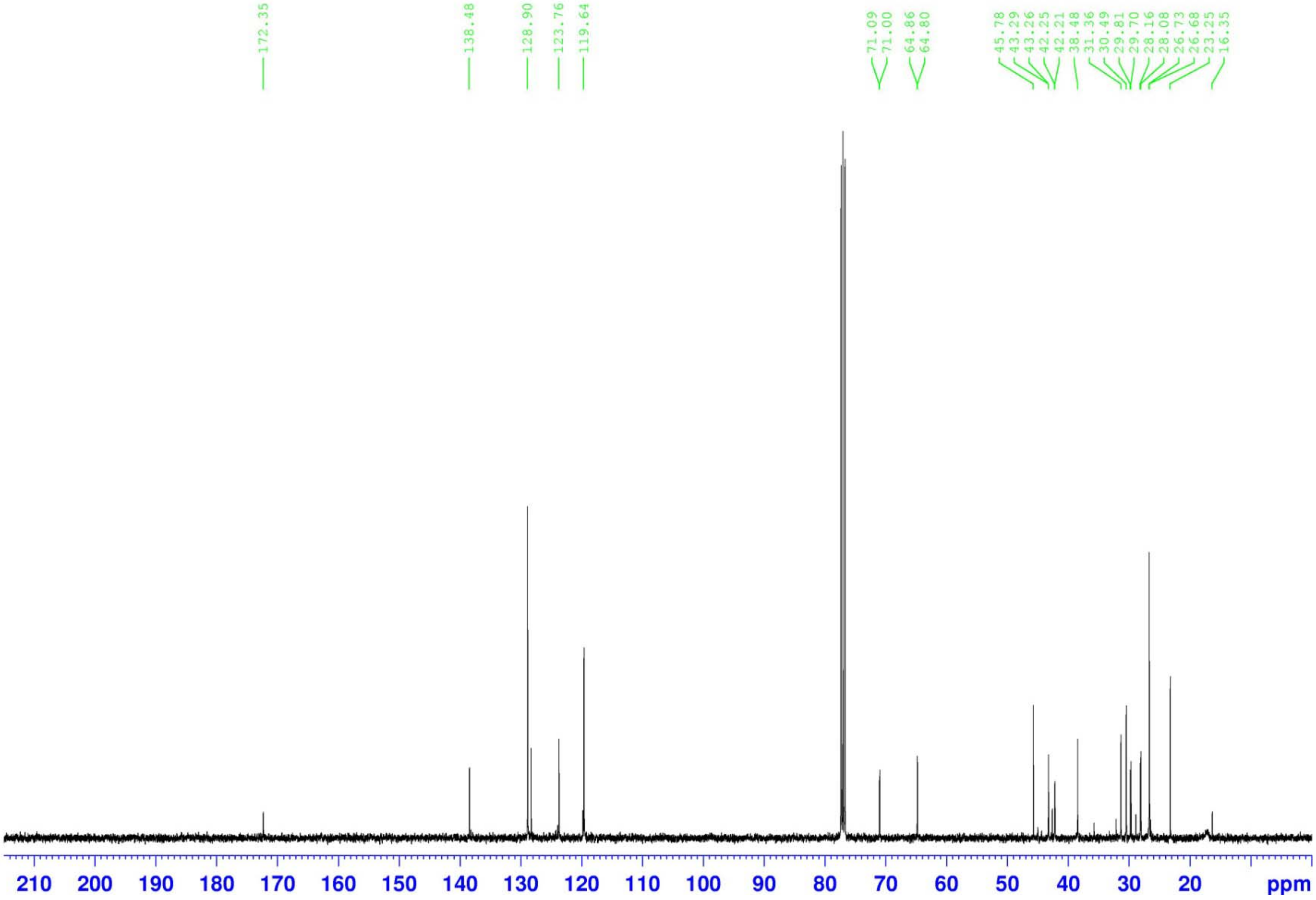
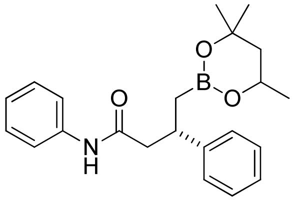
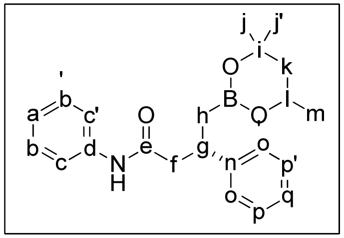
^{13}C NMR of (*S*)-8e





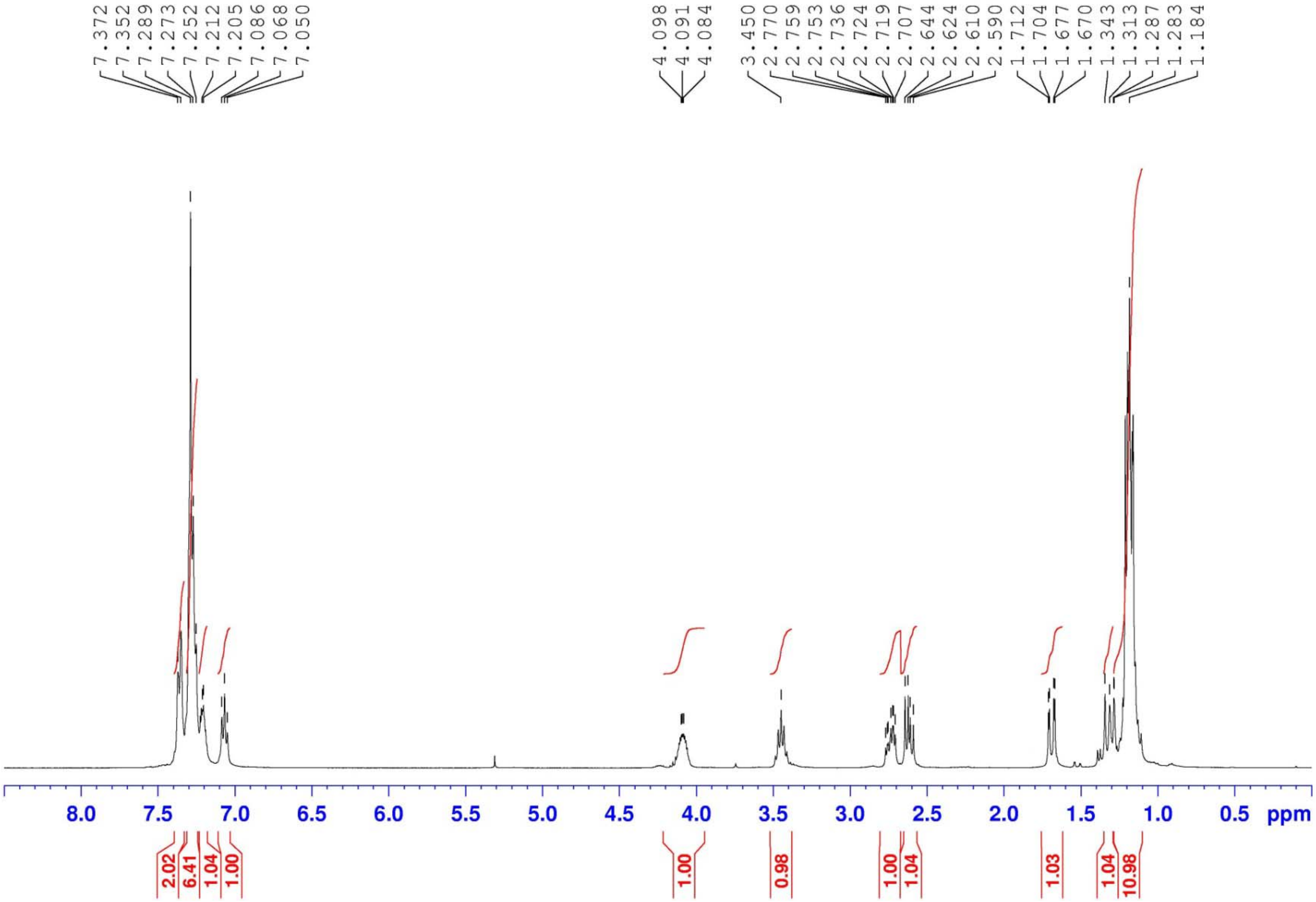
(*S*)-**8f**



After following the general procedure for the directed CAHB of **7f**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (71%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -18.8° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.36 (2H, d, *J* = 7.8 Hz, c,c'), 7.35–7.25 (6H, m, b,b',o,o',p,p'), 7.25–7.15 (1H, m, q), 7.07 (1H, t, *J* = 7.2 Hz, a), 4.15–4.00 (1H, m, l), 3.50–3.40 (1H, m, g), 2.80–2.70 (1H, m, f), 2.62 (1H, dd, *J* = 13.6 Hz, 2.8 Hz, f), 1.69 (1H, dd, *J* = 13.9 Hz, 2.8 Hz, k), 1.35–1.25 (1H, m, k), 1.25–1.05 (11H, m, h,j,j',m).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 170.56 (e), 146.23 (n), 137.95 (d), 128.84 (b,b'), 128.41 (p,p'), 127.32 and 127.30 (o,o'), 126.29 (q), 123.99 (a), 119.80 (c,c'), 70.80 (i), 64.71 and 64.68 (l), 47.15 and 47.11 (f), 45.70 (k), 39.05 (g), 31.15 and 31.11 (h), 27.97 and 27.90 (j,j'), 23.08 and 23.07 (m).
IR (neat)	3299 (N-H stretch), 2973, 1657 (C=O stretching), 1600, 1544 (N-H bend), 1499, 1442, 1392, 1302 (C-O stretch), 1208 (C-N stretch), 1187, 909, 755, 731 cm^{-1}.
HRMS (EI)	Calcd. for $C_{22}H_{28}BNO_3$: 365.2162, found 365.2169 *m/z*.

^{1}H NMR of (*S*)-8f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1.04
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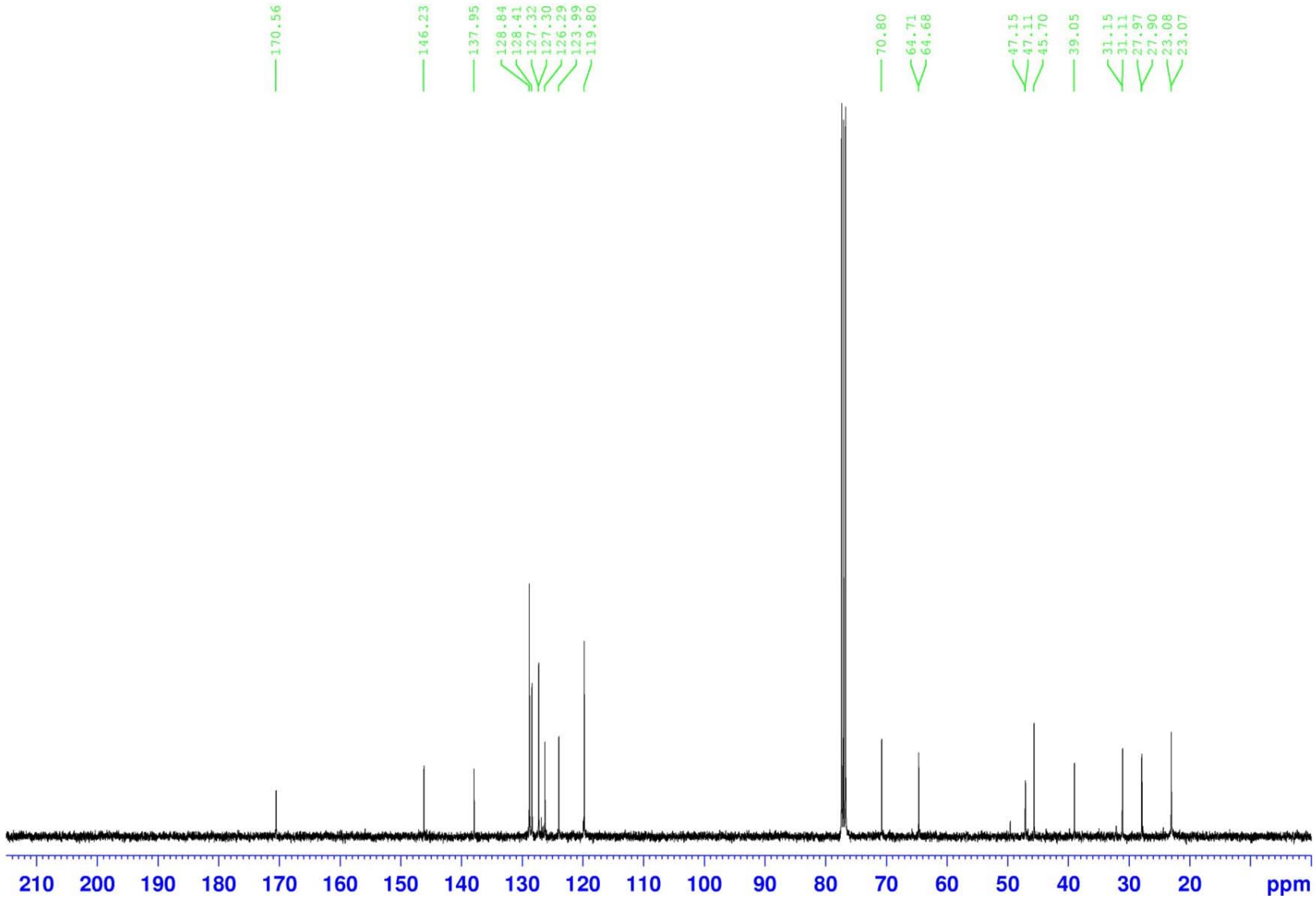
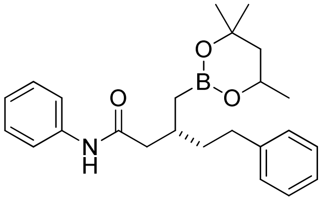
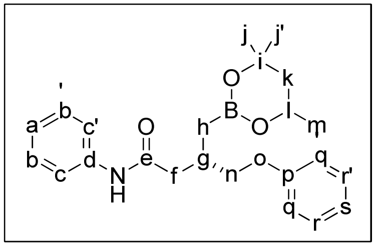
^{13}C NMR of (*S*)-8f





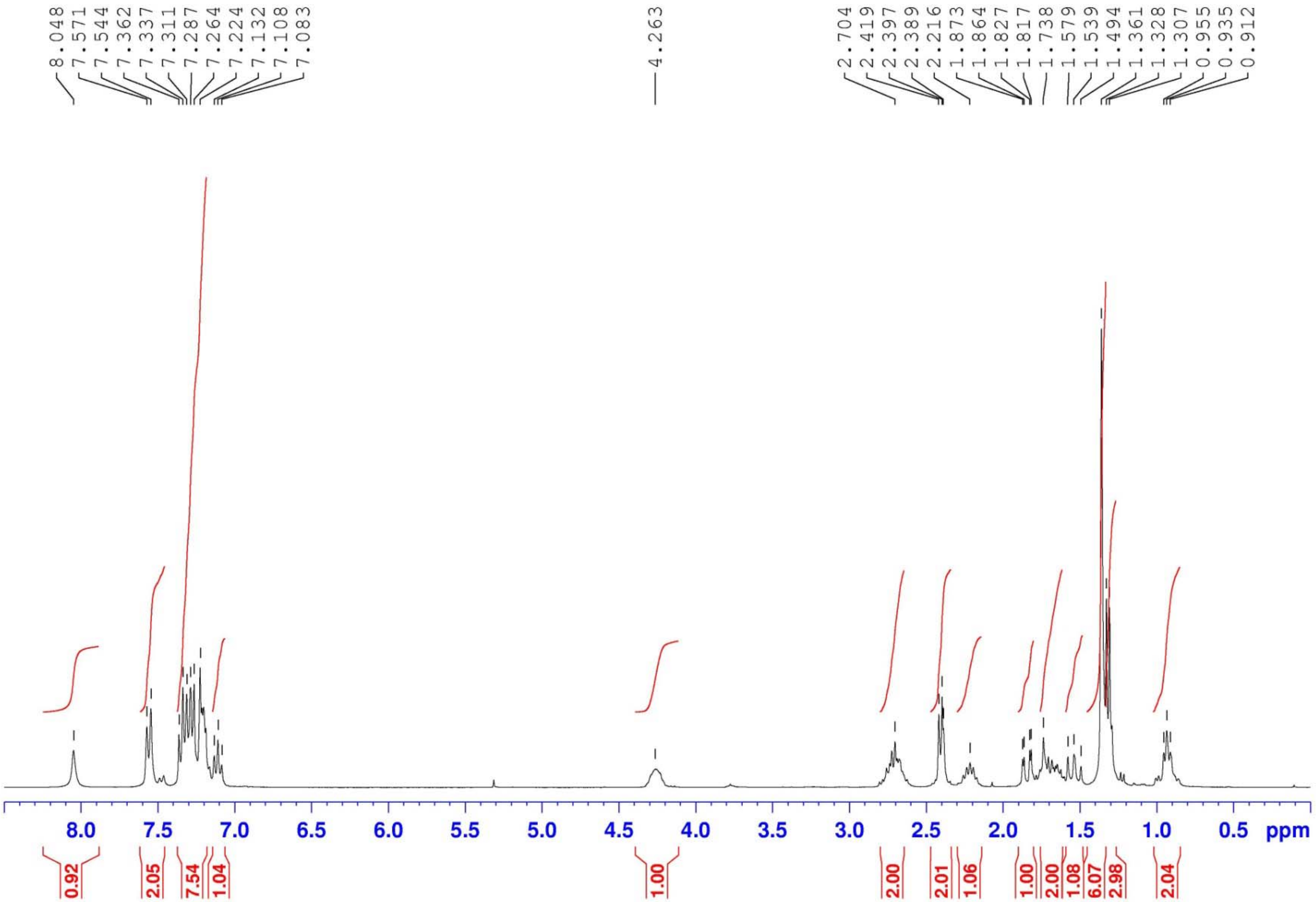
(*R*)-**8c**



After following the general procedure for the directed CAHB of **7c**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (73%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -12.6° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 8.05 (1H, br s, NH), 7.56 (2H, d, *J* = 8.0 Hz, c,c'), 7.40–7.15 (7H, m, b,b',o,o',p,p',q), 7.11 (1H, t, *J* = 7.3 Hz, a), 4.35–4.15 (1H, m, l), 2.85–2.60 (2H, m, o), 2.45–2.35 (2H, m, f), 2.30–2.15 (1H, m, g), 1.85 (1H, dd, *J* = 14.0 Hz, 2.8 Hz, k), 1.80–1.60 (2H, m, n), 1.60–1.45 (1H, m, k), 1.36 (6H, s, j,j'), 1.32 (3H, d, *J* = 6.2 Hz, m), 1.00–0.90 (2H, m, h).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 171.35 (e), 142.74 (p), 138.36 (d), 128.93 (b,b'), 128.43 (r,r'), 128.29 (q,q'), 125.62 (s), 123.87 (a), 119.66 (c,c'), 71.28 and 71.24 (i), 64.95 (l), 45.86 (k), 44.63 (f), 39.24 (n), 33.59 (o), 33.09 (g), 31.40 (h), 28.29 and 28.24 (j,j'), 23.29 and 23.27 (m).
IR (neat)	3305 (N-H stretch), 2972, 2929, 1659 (C=O stretch), 1600, 1541 (N-H bend), 1498, 1442, 1390, 1303 (C-O stretch), 1209 (C-N stretch), 906, 754 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{24}H_{32}BNaNO_3$ (M+Na): 416.2373, found 416.2379 *m/z*.

^{1}H NMR of (*R*)-8c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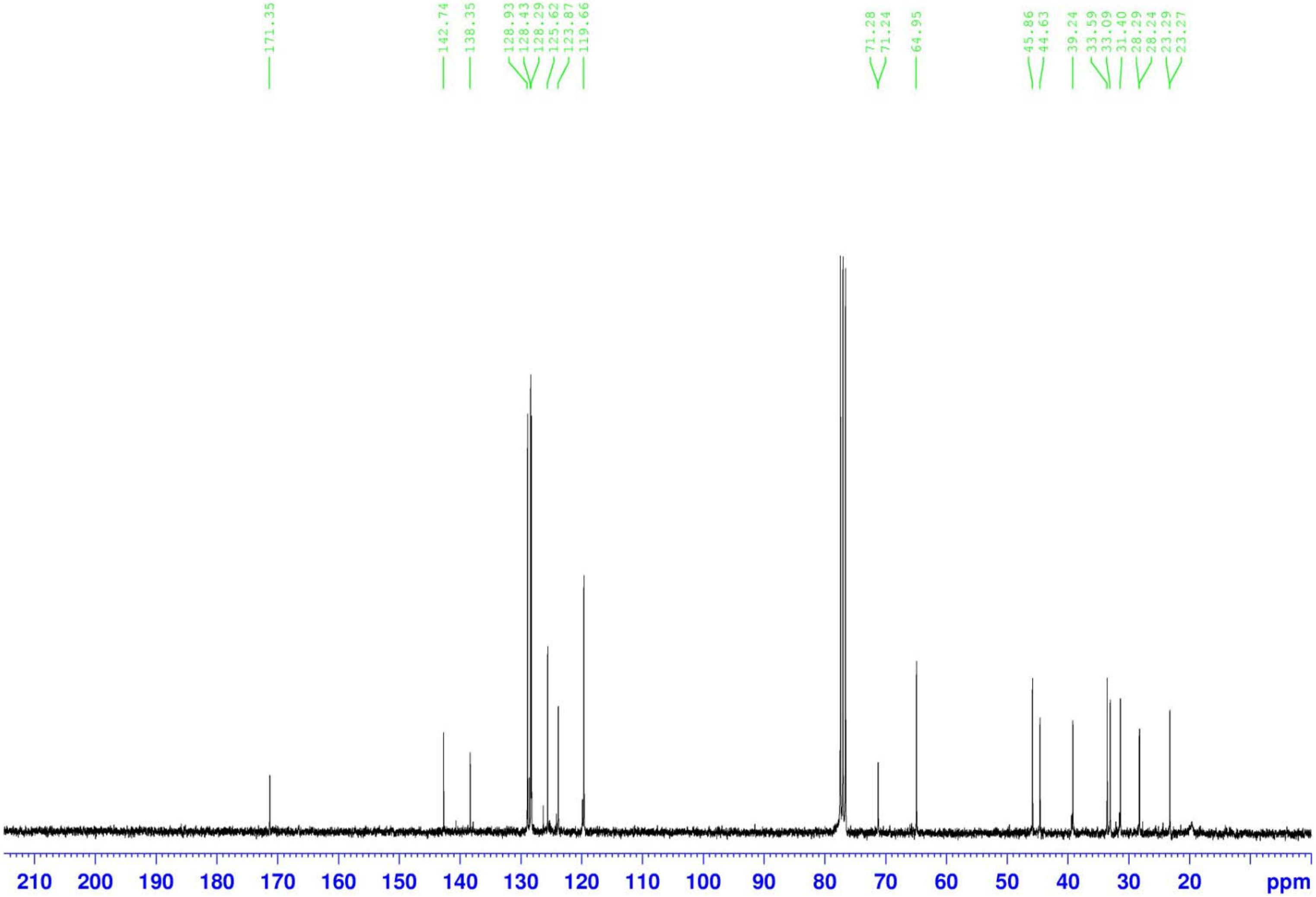
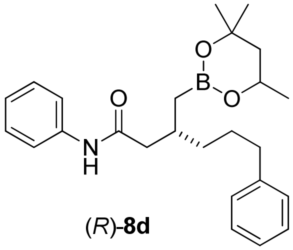
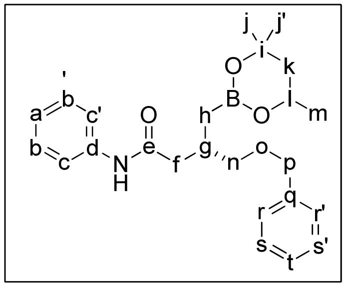
4.263
2.704
2.419
2.397
2.389
2.216
1.873
1.864
1.827
1.817
1.738
1.579
1.539
1.494
1.361
1.328
1.307
0.955
0.935
0.912
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.92
2.05
7.54
1.04
1.00
2.00
2.01
1.06
1.00
2.00
1.08
6.07
2.98
2.04

^{13}C NMR of (*R*)-8c





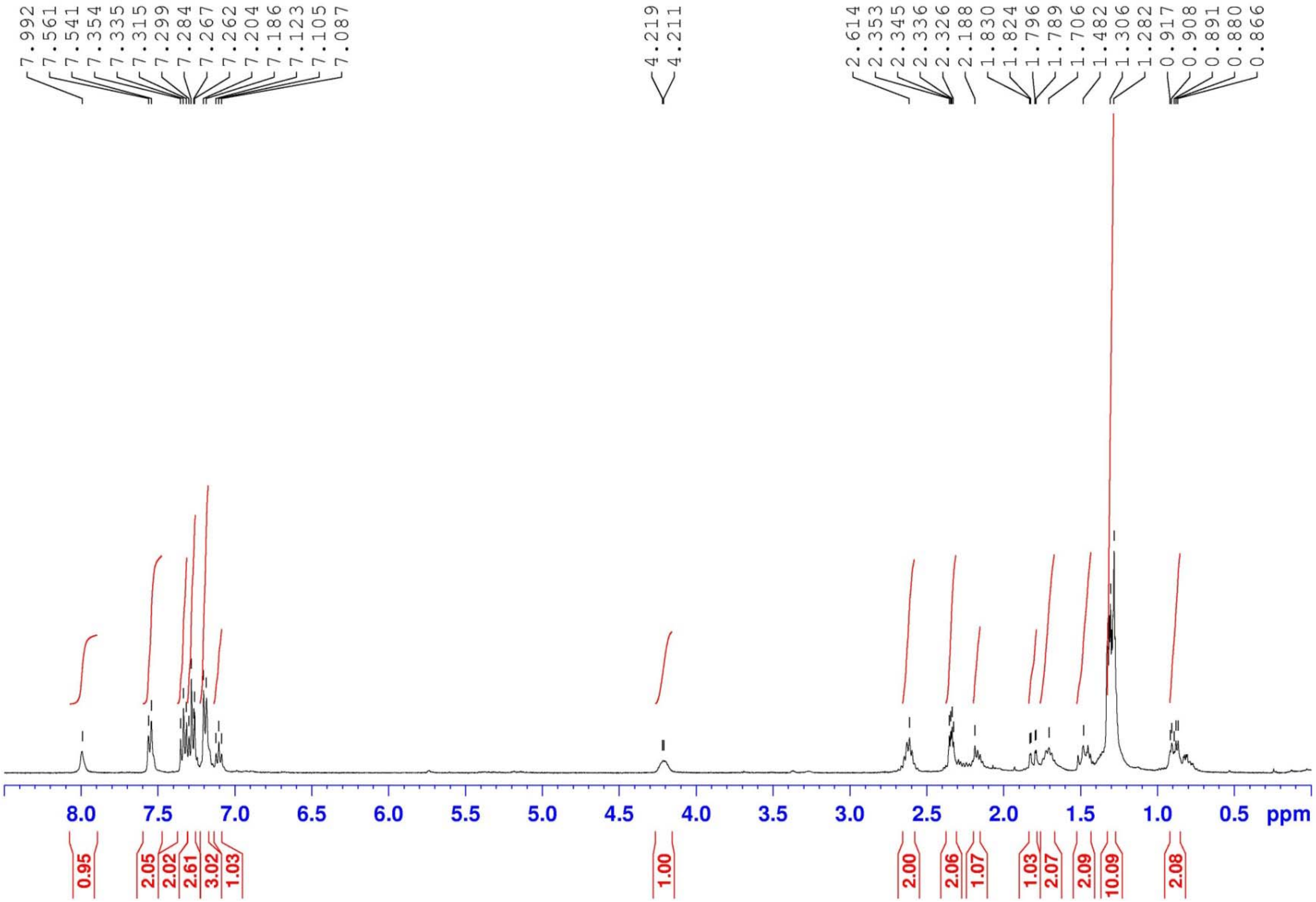
(*R*)-**8d**



After following the general procedure for the directed CAHB of **7d**, flash chromatography on silica gel (80:20 hexanes:ethyl acetate) affords the title compound (70%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -14.5° (*c* 0.5, CHCl$_3$)
TLC analysis	R$_f$ 0.7 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, CDCl$_3$)	δ 7.99 (1H, br s, NH), 7.55 (2H, d, *J* = 7.8 Hz, c,c'), 7.33 (2H, t, *J* = 7.7 Hz, b,b'), 7.30–7.25 (2H, m, s,s'), 7.25–7.15 (3H, m, r,r',t), 7.11 (1H, t, *J* = 7.3 Hz, a), 4.25–4.15 (1H, m, l), 2.70–2.50 (2H, m, p), 2.40–2.30 (2H, m, f), 2.25–2.10 (1H, m, g), 1.81 (1H, dd, *J* = 14.0 Hz, 2.6 Hz, k), 1.80–1.65 (2H, m, o), 1.55–1.40 (2H, m, k,n), 1.35–1.25 (10H, m, n,j,j',m), 0.95–0.85 (2H, m, h).
^{13}C NMR (100 MHz, CDCl$_3$)	δ 171.51 (e), 142.71 (q), 138.36 (d), 128.92 (b,b'), 128.42 (s,s'), 128.20 (r,r'), 125.57 (t), 123.84 (a), 119.63 (c,c'), 71.13 (i), 64.86 (l), 45.81 (k), 44.77 (f), 36.82 (n), 36.04 (p), 33.20 (g), 31.35 (h), 29.71 and 29.04 (j,j'), 28.15 (o), and 23.23 (m).
IR (neat)	2973 (N-H stretch), 2925, 2854, 1649 (C=O stretch), 1595, 1495 (N-H bend), 1386, 1378, 1307, 1239 (C-N stretch), 1143 (C-O stretch), 1119 (C-O stretch), 966, 868, 772, 749, 698 cm^{-1}.
HRMS (CI)	Calcd. for $C_{25}H_{35}BNO_3$ (M+H): 408.2710, found 408.2711 *m/z*.

^{1}H NMR of (*R*)-8d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7.123
7.105
7.087
4.219
4.211
2.614
2.353
2.345
2.336
2.326
2.188
1.830
1.824
1.796
1.789
1.706
1.482
1.306
1.282
0.917
0.908
0.891
0.880
0.866
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
ppm
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2.02
2.61
3.02
1.03
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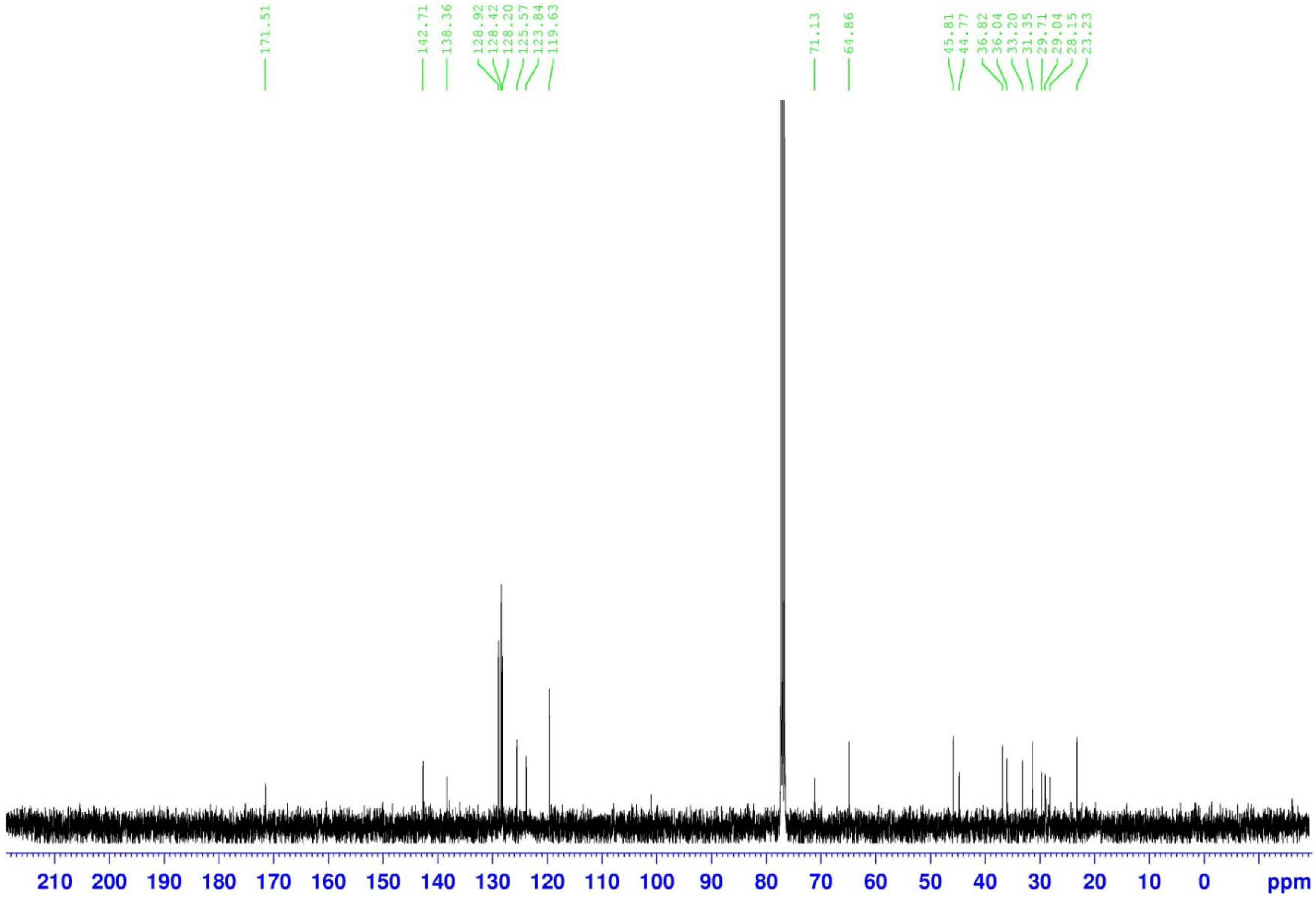
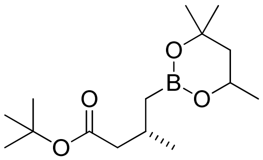
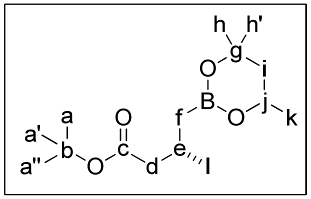
2.07
2.09
10.09
2.08

^{13}C NMR of (*R*)-8d





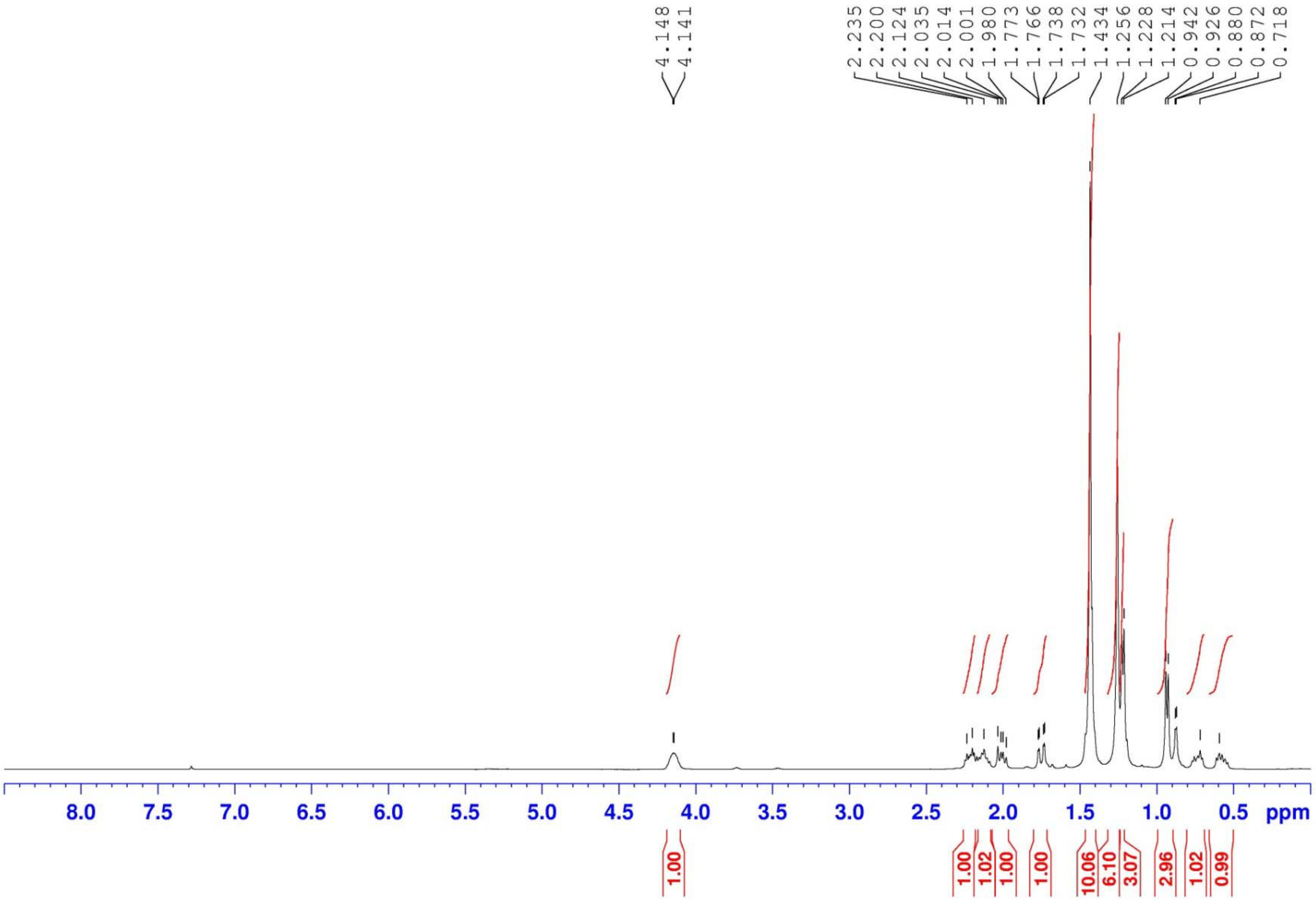
(*S*)-**8g**



After following the general procedure for the directed CAHB of **7g**, flash chromatography on silica gel (95:5 hexanes:ethyl acetate) affords the title compound (62%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -4.0^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.5 (80:20 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 4.20–4.05 (1H, m, j), 2.25–2.15 (1H, m, d), 2.15–2.05 (1H, m, e), 2.05–1.95 (1H, m, d), 1.75 (1H, dd, *J* = 13.8 Hz, 2.7 Hz, i), 1.50–1.40 (1H, m, i), 1.43 (9H, s, a,a',a''), 1.26 (6H, s, h,h'), 1.22 (3H, d, *J* = 5.8 Hz, k), 0.93 (3H, d, *J* = 6.3 Hz, l), 0.80–0.70 (1H, m, f), 0.65–0.50 (1H, m, f).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.96 (c), 79.50 (b), 70.39 (g), 64.44 (j), 45.95 (i), 45.19 (e), 45.15 (d), 31.25 (f), 28.13 (a,a',a''), 27.24 and 27.21 (h,h'), 23.18 (k), 22.01 and 21.98 (l).
IR (neat)	2979, 2930, 1725 (C=O stretch), 1474, 1440, 1366, 1319, 1248, 1141 (C-O stretch), 1051, 968, 917, 846, 750, 733, 681, 667 cm^{-1}.
HRMS (CI)	Calcd. for $C_{15}H_{30}BO_4$ (M+H): 285.2237, found 285.2243 *m/z*.

^{1}H NMR of (*S*)-8g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1.434
1.256
1.228
1.214
0.942
0.926
0.880
0.872
0.718
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
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0.5
ppm
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1.00
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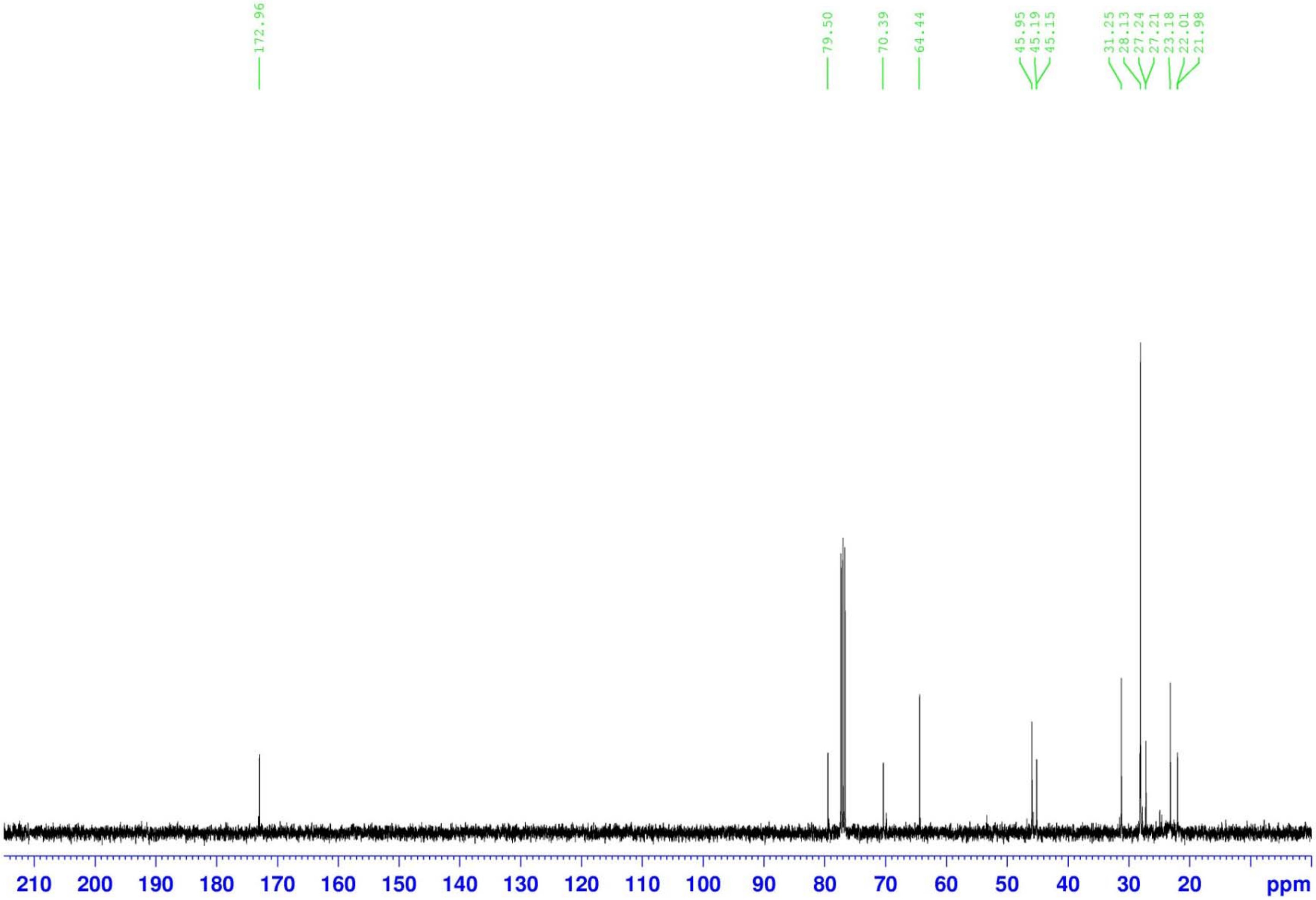
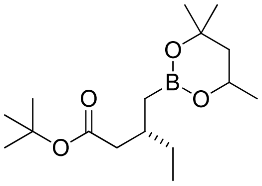
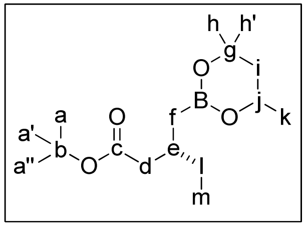
1.02
0.99

^{13}C NMR of (*S*)-8g





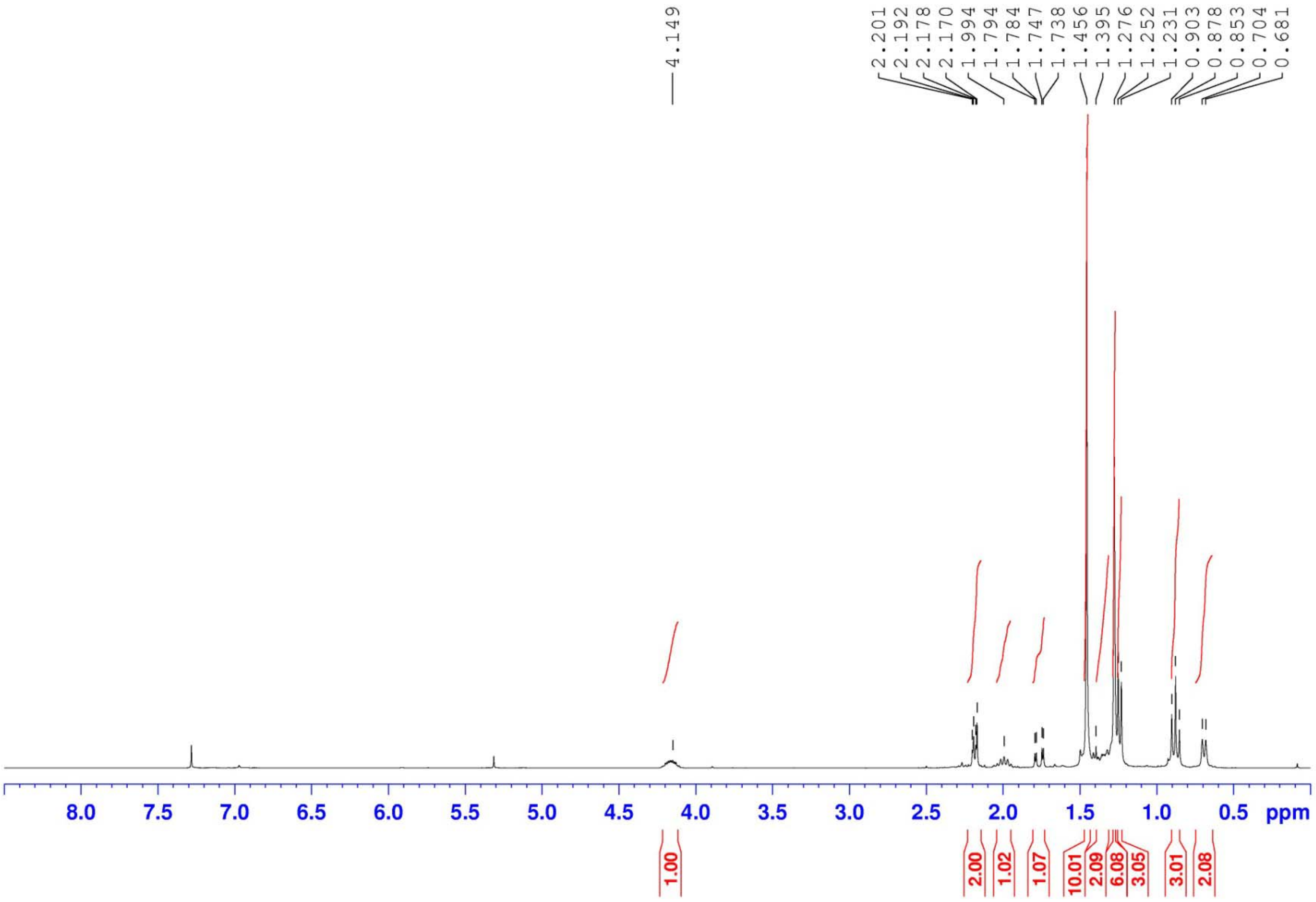
(*R*)-**8h**



After following the general procedure for the directed CAHB of **7h**, flash chromatography on silica gel (95:5 hexanes:ethyl acetate) affords the title compound (65%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -3.8^{\circ}$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.5 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 4.25–4.10 (1H, m, j), 2.19 (2H, dd, *J* = 6.9 Hz, 2.6 Hz, d), 2.05–1.95 (1H, m, e), 1.77 (1H, dd, *J* = 13.8 Hz, 3.0 Hz, i), 1.50–1.40 (1H, m, i),1.46 (9H, s, a,a’,a’’), 1.40–1.30 (2H, m, l), 1.28 (6H, s, h,h’), 1.24 (3H, d, *J* = 6.2 Hz, k), 0.88 (3H, t, *J* = 7.4 Hz, m), 0.69 (2H, d, *J* = 6.8 Hz, f).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.24 (c), 79.51 (b), 70.39 (g), 64.41 (j), 45.96 (i), 42.33 (d), 33.27 (e), 31.28 (f), 28.94 (l), 28.16 (a,a’,a’’), 28.06 (h,h’), 23.21 (k), 11.19 (m).
IR (neat)	2976, 2926, 1726 (C=O stretch), 1456, 1379, 1315, 1246, 1213, 1138 (C-O stretch), 966, 850, 669, 518 cm^{-1}.
HRMS (CI)	calcd. for $C_{16}H_{32}BO_4$ (M+H): 299.2394, found 299.2397 *m/z*.

^{1}H NMR of (*R*)-8h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1.252
1.231
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0.878
0.853
0.704
0.681
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6.5
6.0
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0.5
ppm
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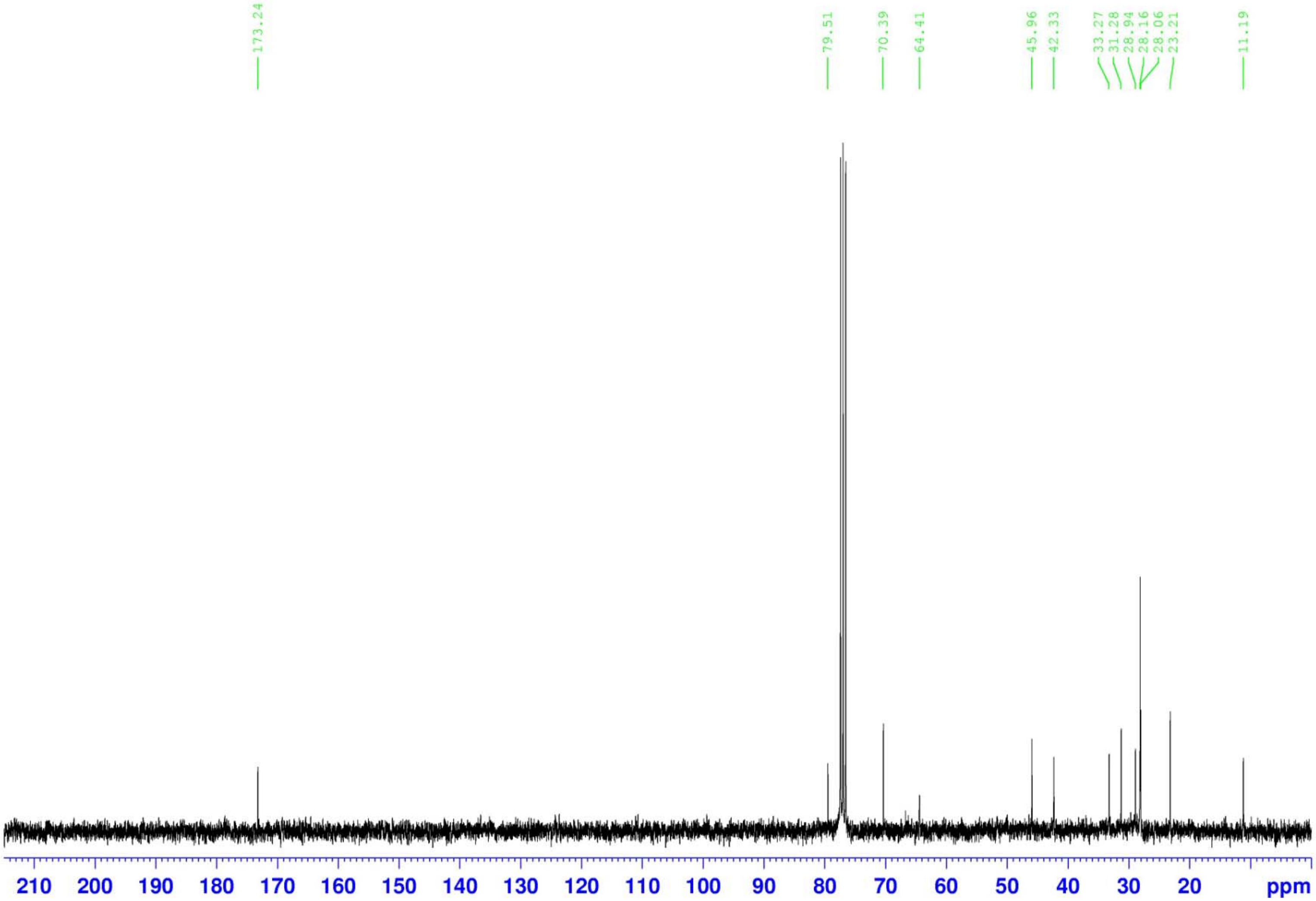
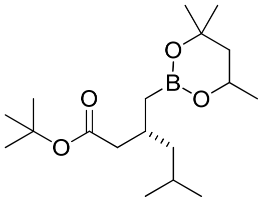
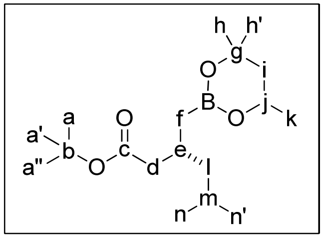
3.01
2.08

^{13}C NMR of (*R*)-8h





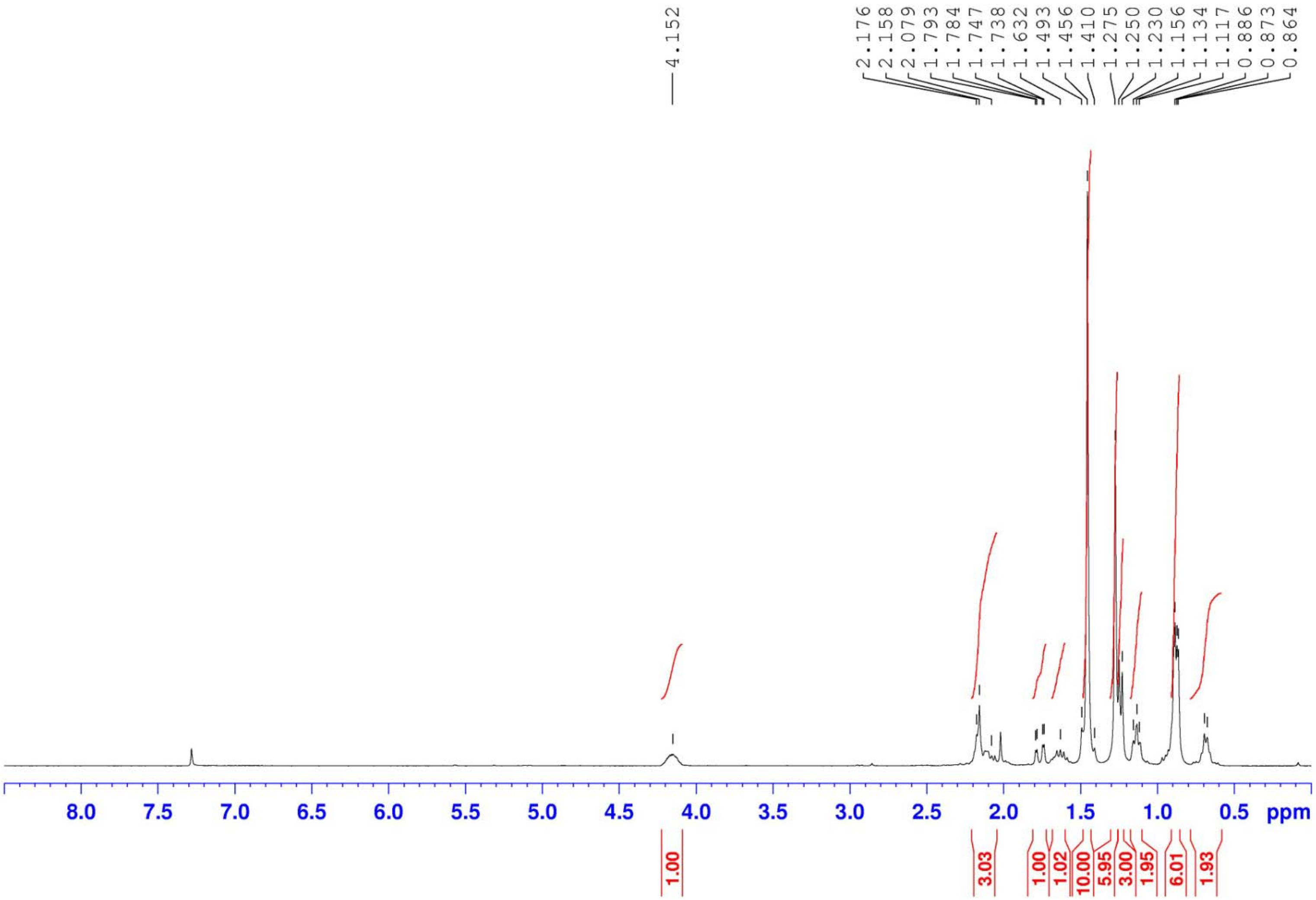
(*R*)-**8i**



After following the general procedure for the directed CAHB of **7i**, flash chromatography on silica gel (95:5 hexanes:ethyl acetate) affords the title compound (78%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -2.7° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.5 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 4.20–4.10 (1H, m, j), 2.20–2.05 (3H, m, d,e), 1.77 (1H, dd, *J* = 13.8 Hz, 2.8 Hz, i), 1.70–1.55 (1H, m, m), 1.50–1.40 (1H, m, i), 1.46 (9H, s, a,a',a''), 1.28 (6H, s, h,h'), 1.24 (3H, d, *J* = 6.2 Hz, k), 1.13 (2H, t, *J* = 5.9 Hz, l), 0.90–0.85 (6H, m, n,n'), 0.69 (2H, d, *J* = 5.9 Hz, f).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.16 (c), 79.51 (b), 70.39 (g), 64.43 (j), 46.21 (l), 45.97 (i), 43.17 (d), 31.30 (f), 29.62 (e), 28.16 (a,a',a''), 28.06 (h,h'), 25.32 (m), 23.21 (k), 23.15, 23.04, 22.83, 22.64 (n,n').
IR (neat)	2971, 2931, 2870, 1725 (C=O stretch), 1456, 1389, 1366, 1300, 1206, 1156 (C-O stretch), 1134 (C-O stretch), 956, 846, 739 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{18}H_{35}NaBO_4$ (M+Na): 349.2526, found 349.2515 *m/z*.

^{1}H NMR of (*R*)-8i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1.230
1.156
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1.117
0.886
0.873
0.864
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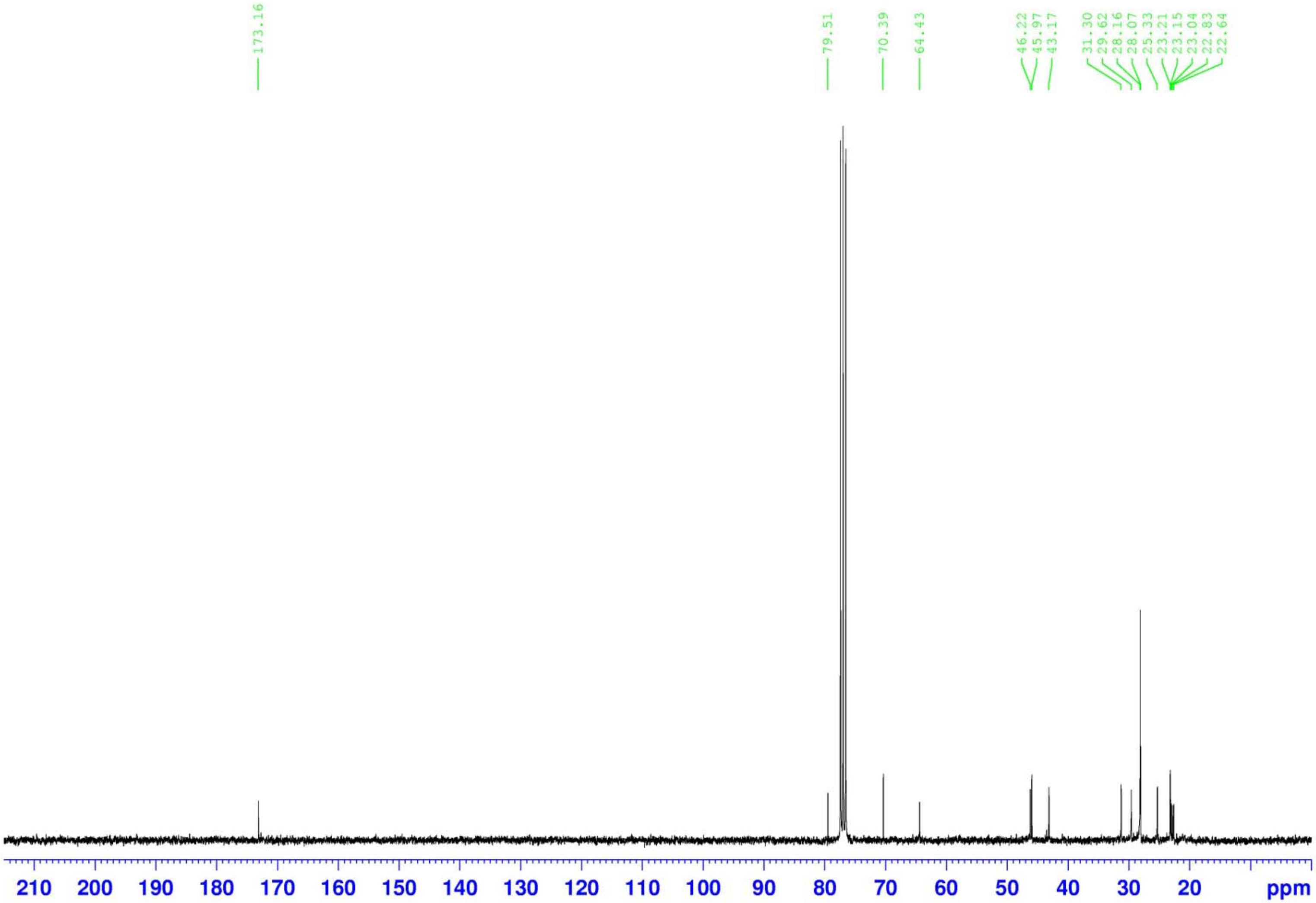
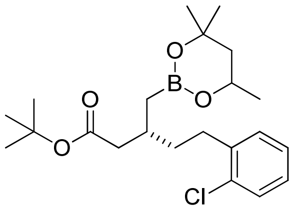
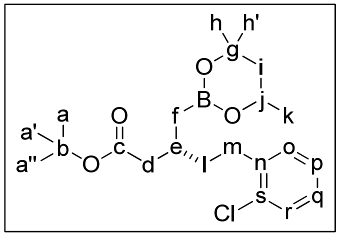
^{13}C NMR of (*R*)-8i





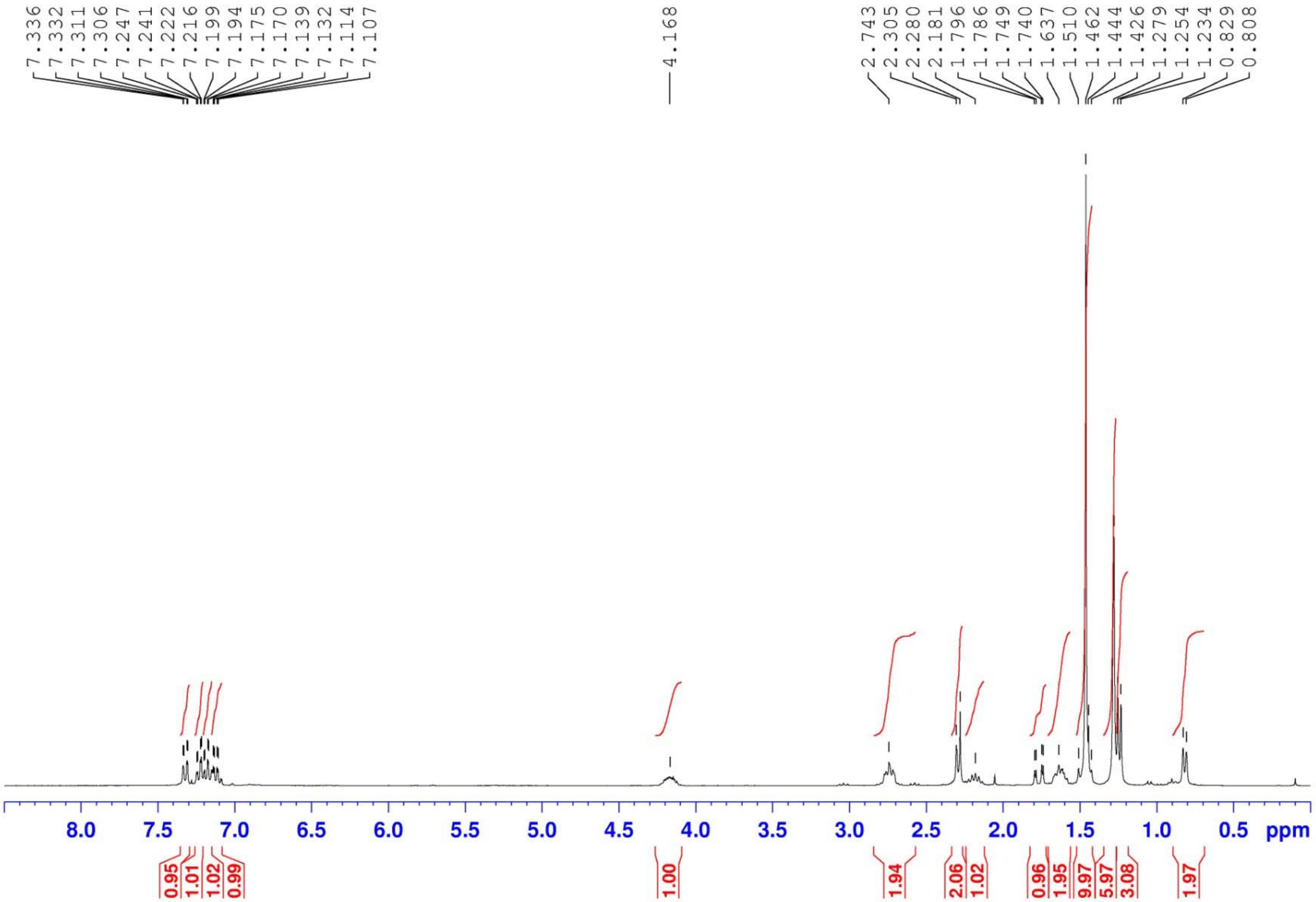
(*R*)-**13**



After following the general procedure for the directed CAHB of **12**, flash chromatography on silica gel (95:5 hexanes:ethyl acetate) affords the title compound (74%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -4.5^\circ$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.5 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.32 (1H, dd, *J* = 7.6 Hz, 1.2 Hz, r), 7.23 (1H, dd, *J* = 7.5 Hz, 1.9 Hz, p), 7.18 (1H, dd, *J* = 7.2 Hz, 1.5 Hz, o), 7.12 (1H, dd, *J* = 7.6 Hz, 2.1 Hz, q), 4.25–4.10 (1H, m, j), 2.80–2.70 (2H, m, m), 2.35–2.25 (2H, m, d), 2.25–2.10 (1H, m, e), 1.77 (1H, dd, *J* = 13.8 Hz, 2.9 Hz, i), 1.70–1.55 (2H, m, l), 1.55–1.40 (1H, m, i), 1.46 (9H, s, a,a',a''), 1.28 (6H, s, h,h'), 1.24 (3H, d, *J* = 6.2 Hz, k), 0.82 (2H, d, *J* = 6.2 Hz, f).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 172.90 (c), 140.65 (n), 133.84 (s), 130.29 (r), 129.33 (q), 126.99 (o), 126.67 (p), 79.71 and 79.70 (b), 70.51 (g), 64.52 (j), 45.94 (i), 42.56 (d), 36.45 (l), 31.82 (m), 31.28 (f), 31.08 (e), 28.16 (a,a',a''), 28.11 (h,h'), 23.20 (k).
IR (neat)	2978, 2930, 1724 (C=O stretch), 1475, 1443, 1366, 1320, 1240, 1142 (C-O stretch), 1050, 968, 917, 846, 750, 732, 680, 647 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{22}H_{34}NaBClO_4$ (M+Na): 431.2136, found 431.2145 *m/z*.

^{1}H NMR of (*R*)-13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7.132
7.114
7.107
4.168
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2.280
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1.786
1.749
1.740
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1.462
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1.426
1.279
1.254
1.234
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1.02
0.99
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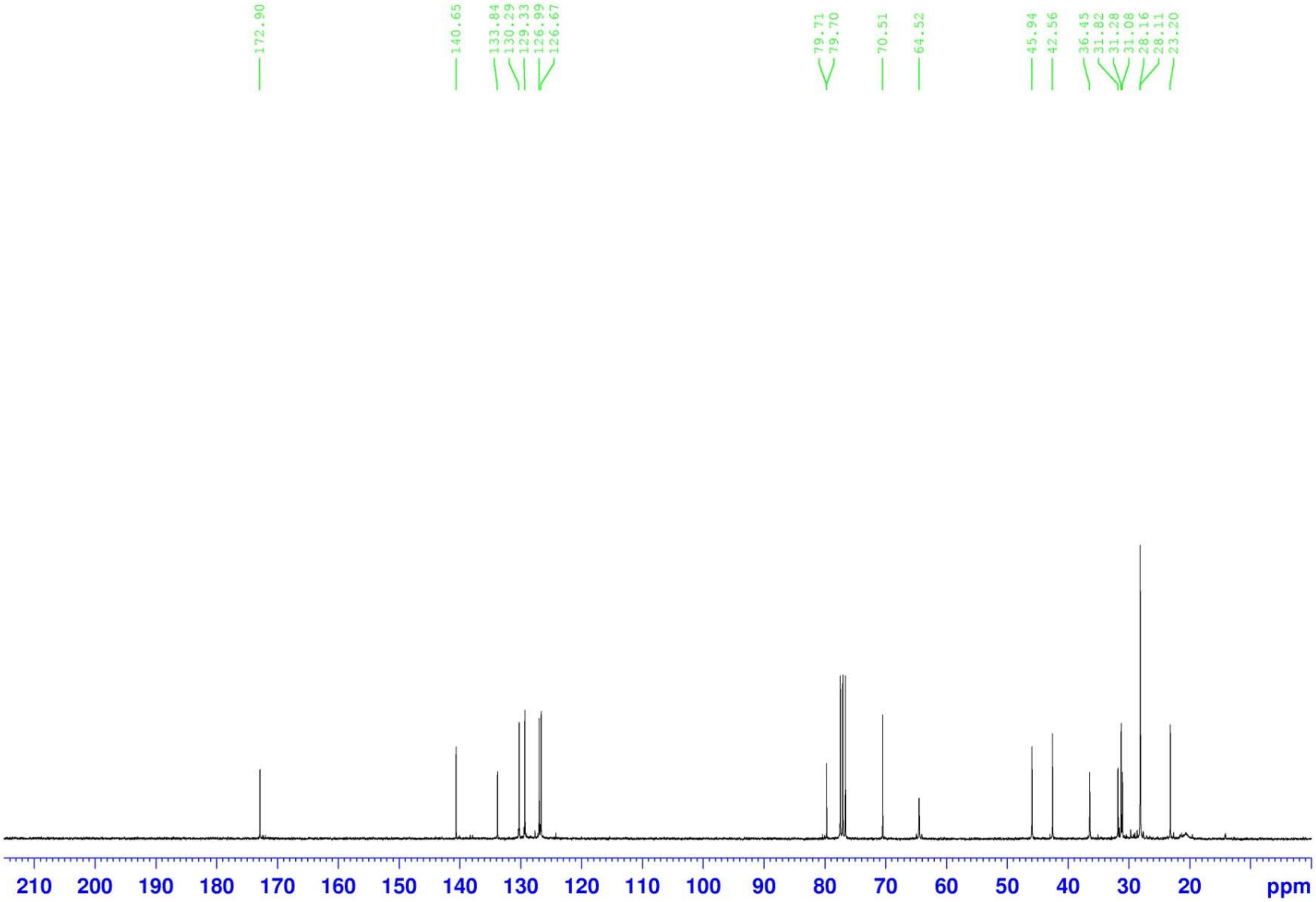
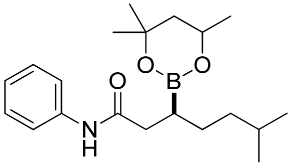
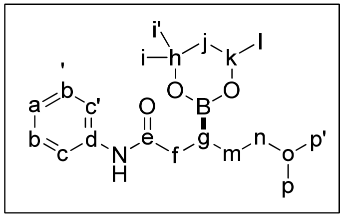
3.08
1.97

^{13}C NMR of (*R*)-13





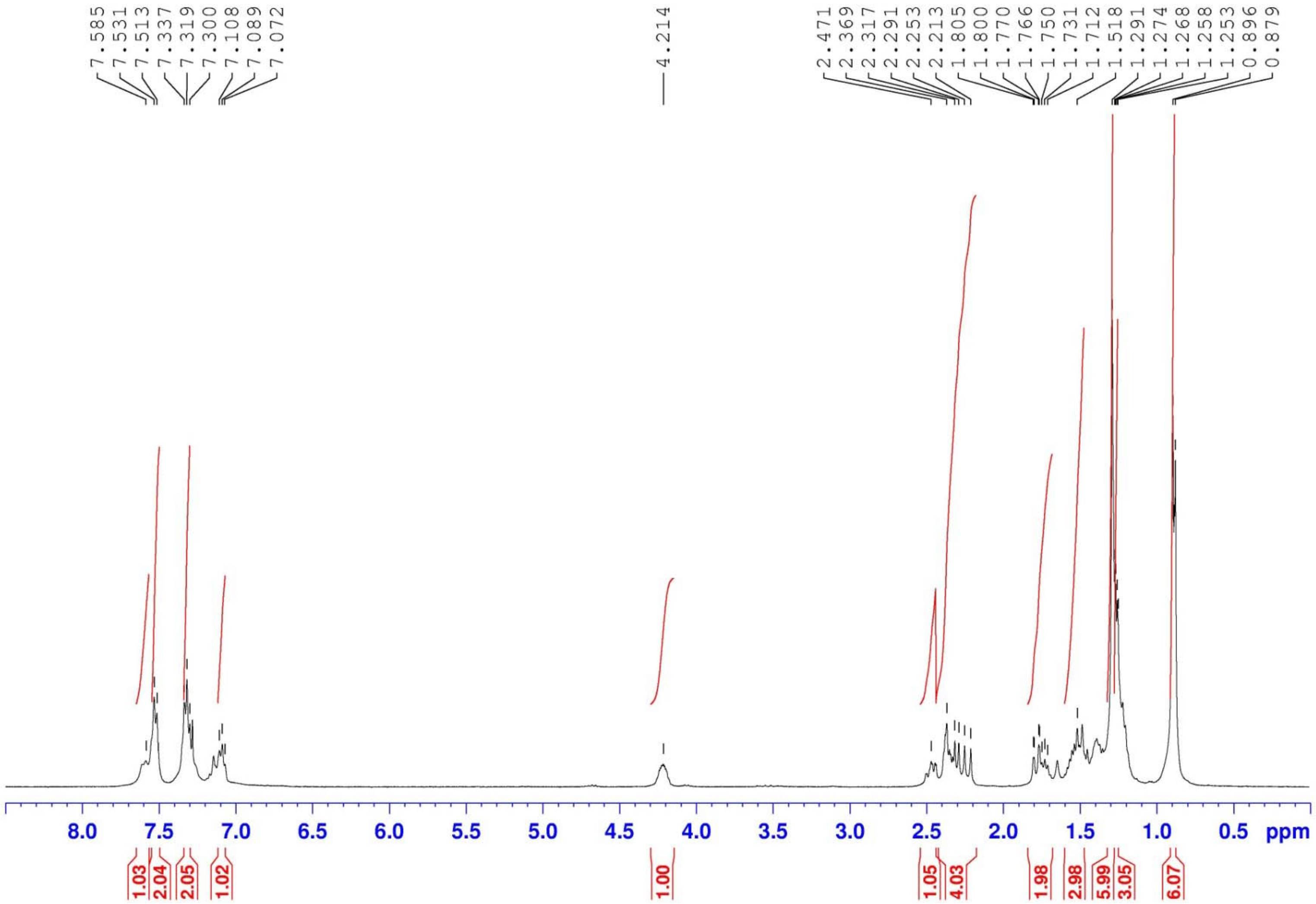
(*S*)-**2**



After following the general procedure for the directed CAHB of **1**, flash chromatography on silica gel (95:5 hexanes:ethyl acetate) affords the title compound (79%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = -7.9^\circ$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (80:20 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.59 (1H, br s, NH), 7.52 (2H, d, *J* = 7.1 Hz, c,c'), 7.32 (2H, t, *J* = 7.4 Hz, b,b'), 7.09 (1H, t, *J* = 7.2 Hz, a), 4.30–4.15 (1H, m, k), 4.30–4.10 (1H, m, k), 2.55–2.40 (1H, m, f), 2.40–2.20 (4H, m, f,g,m), 1.85–1.65 (2H, m, j,o), 1.60–1.45 (3H, m, j,n), 1.29 (6H, s, j,j'), 1.26 (3H, dd, *J* = 6.2 Hz, 2.1 Hz, l), 0.89 (6H, d, *J* = 6.4 Hz, p,p').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.34 (e), 138.39 (d), 128.90 (b,b'), 123.72 (a), 119.60 (c,c'), 70.80 (h), 64.80 and 64.76 (k), 45.93 and 45.89 (j), 39.79 (f), 38.67 (n), 38.11 and 38.05 (g), 31.28 and 27.99 (i,i'), 29.08 (o), 28.20 (m), 23.22 (l), 22.59 (p,p').
IR (neat)	3304 (N-H stretch), 2973, 1660 (C=O stretch), 1600, 1541 (N-H stretch), 1442, 1391, 1369, 1302 (C-O stretch), 1209 (C-N stretch), 1161, 898, 756 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{20}H_{32}NaBNO_3$ (M+Na): 368.2373, found 368.2364 *m/z*.

^{1}H NMR of (*S*)-2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1.766
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2.0
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0.5
ppm
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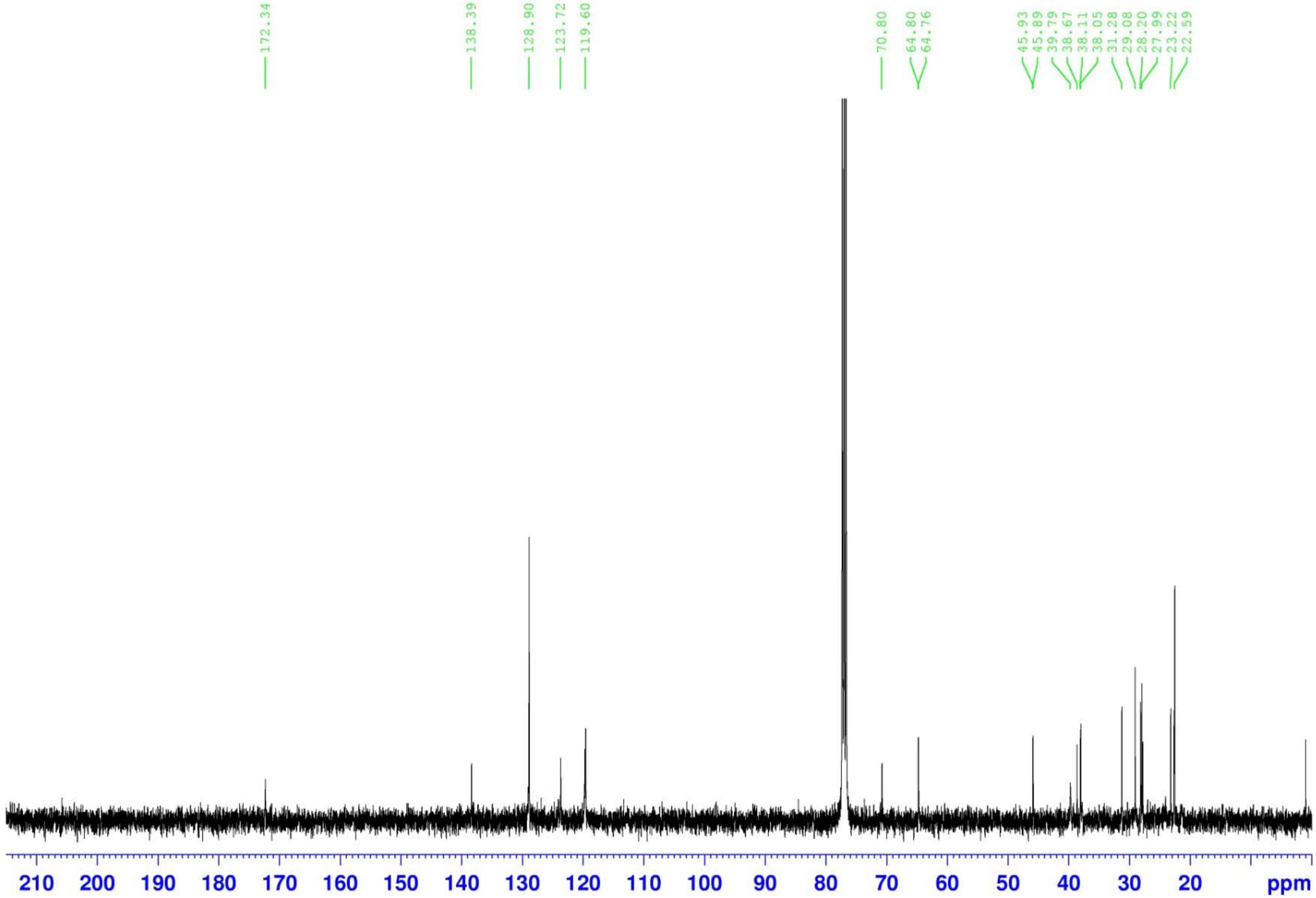
3.05
6.07

^{13}C NMR of (*S*)-2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31.28
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28.20
27.99
23.22
22.59
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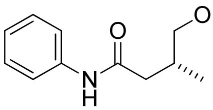
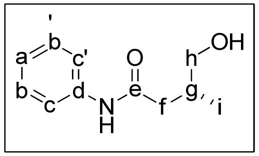
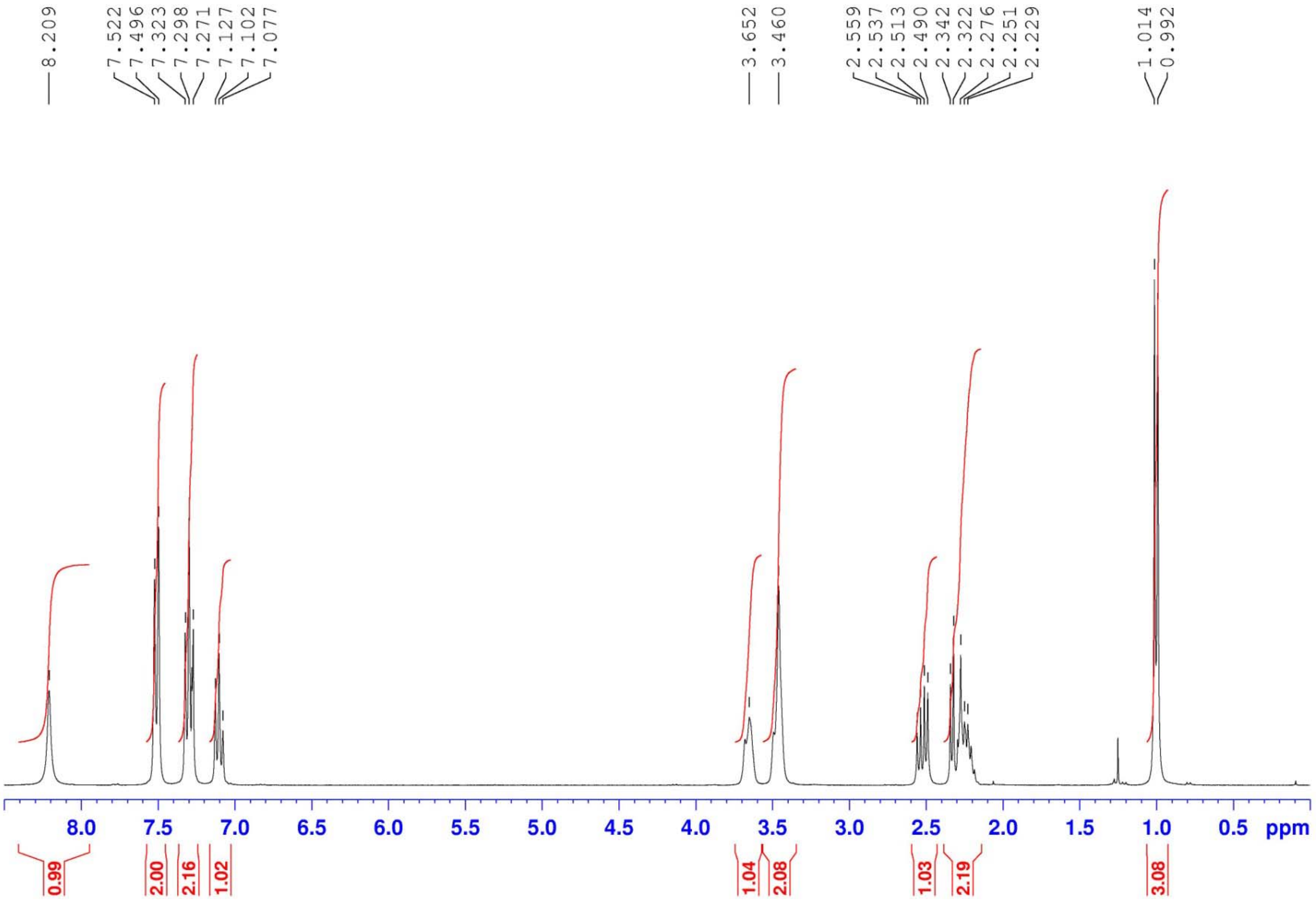




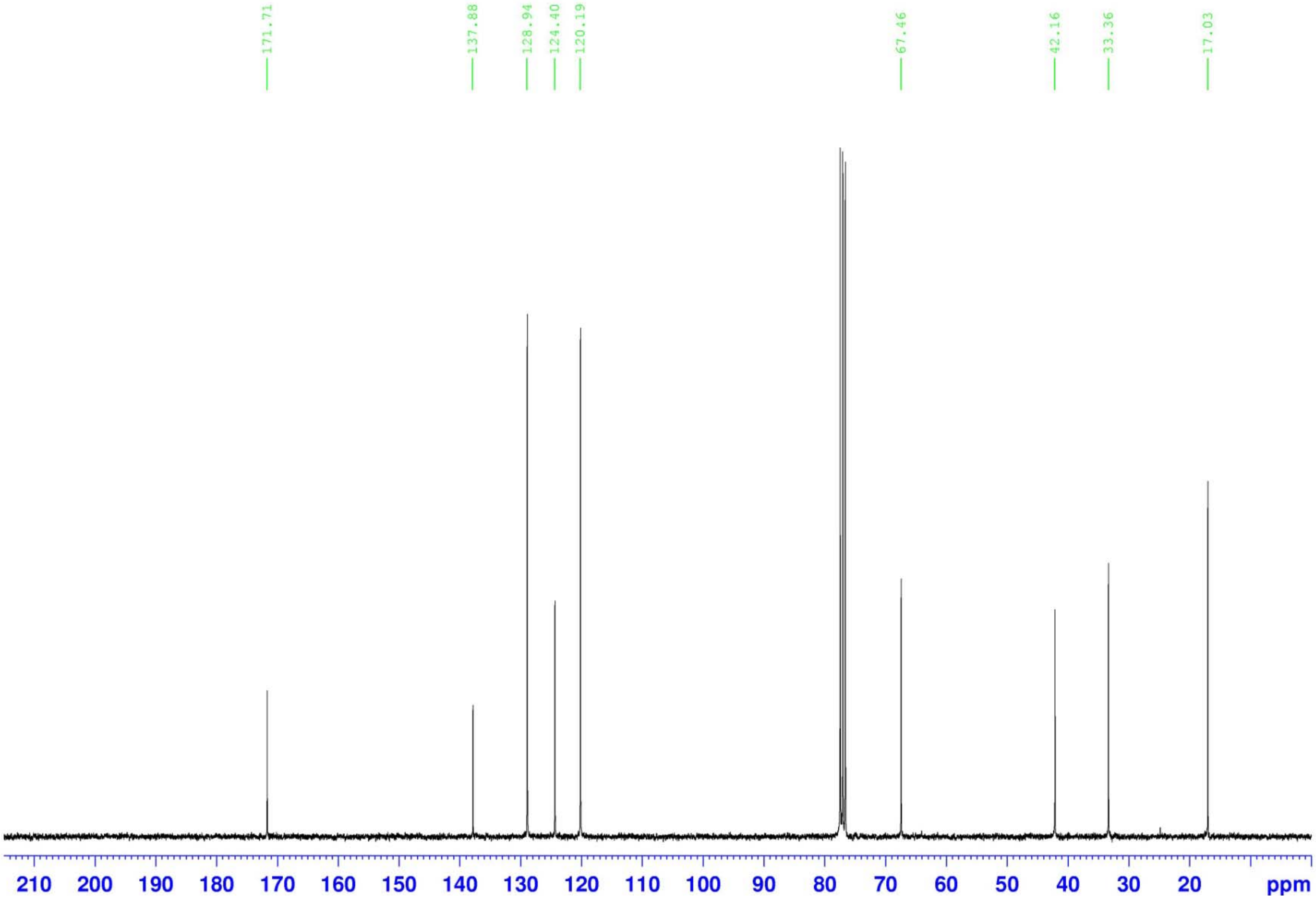
Oxidation of (*S*)-**8a** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (97%) as a white solid.

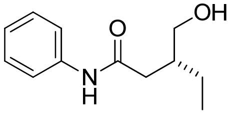
m.p.	115–117 ºC
Optical rotation	$[\alpha]_D^{20} = +2.5^{o}$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 80:20 hexanes:isopropanol) showed peaks at 19 minutes (2.5% (S)) and 36 minutes (97.5% (R))
TLC analysis	R_f 0.3 (30:70 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 8.21 (1H, br s, OH), 7.51 (2H, d, *J* = 7.8 Hz, c,c'), 7.30 (2H, t, *J* = 7.6 Hz, b,b'), 7.10 (1H, t, *J* = 7.4 Hz, a), 3.70–3.55 (1H, m, h), 3.55–3.40 (2H, m, h,OH), 2.52 and 2.29 (2H, overlapping dd's, J_1 = 14.0 Hz, 6.8 Hz, J_2 = 14.0 Hz, 6.00 Hz, f), 2.30–2.20 (1H, m, g), 1.00 (3H, d, *J* = 6.7 Hz, i).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 171.71 (e), 137.88 (d), 128.94 (b,b'), 124.40 (a), 120.19 (c,c'), 67.46 (h), 42.16 (f), 33.36 (g), 17.03 (i).
IR (neat)	3286 (O-H stretch), 3195 (N-H stretch), 3139, 2957, 2928, 1667 (C=O stretch), 1599, 1549 (N-H bend), 1487, 1444, 1376, 1319 (C-O stretch), 1257, 1231 (C-N stretch), 1132, 1047, 754 cm^{-1}.
HRMS (CI)	Calcd. for $C_{11}H_{16}NO_2$ (M+H): 194.1181, found 194.1180 *m/z*.

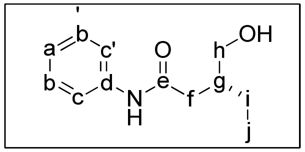
^{1}H NMR of (*R*)-4-hydroxy-3-methylbutanoic acid phenyl amide



^{13}C NMR of (*R*)-4-hydroxy-3-methylbutanoic acid phenyl amide



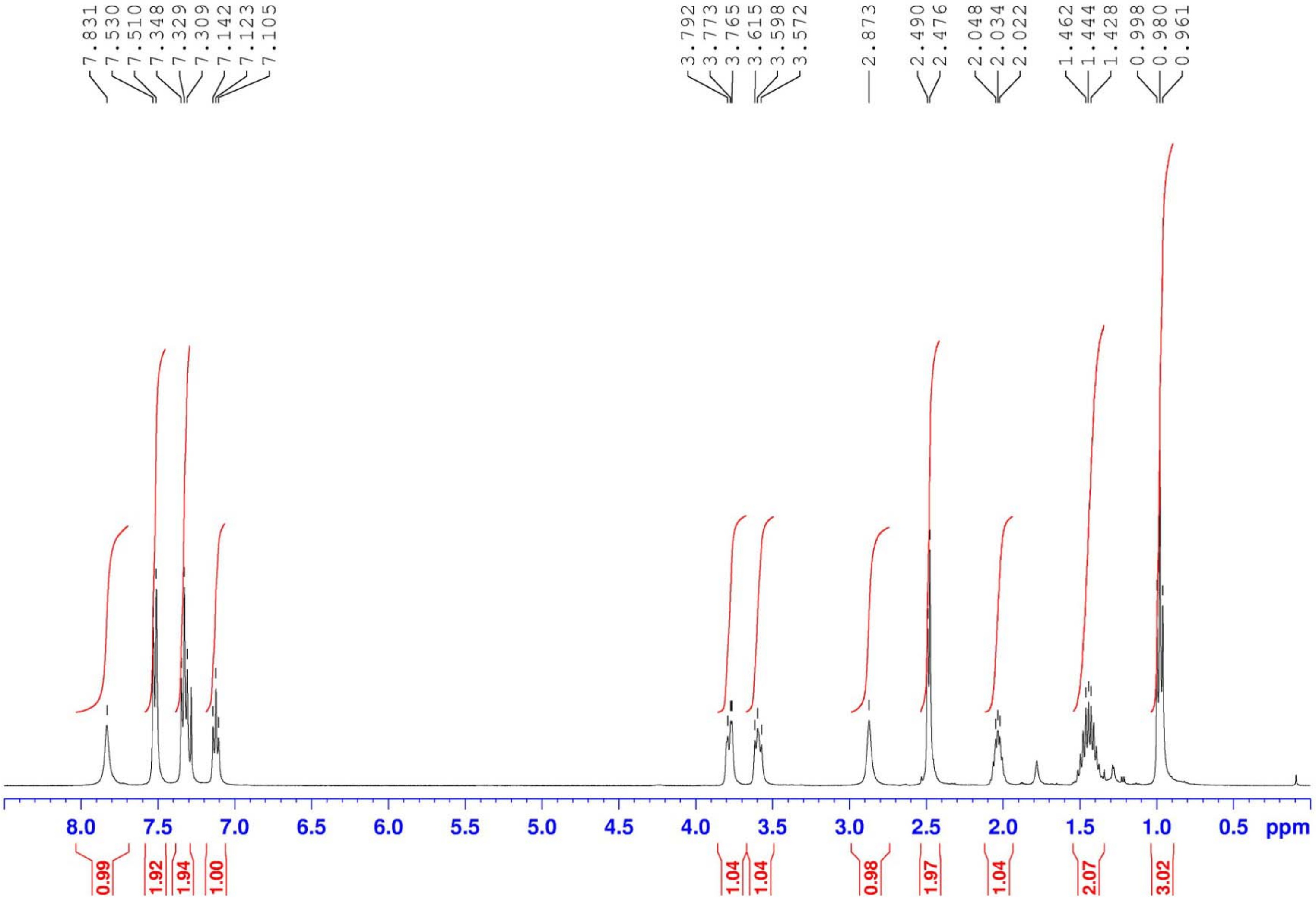




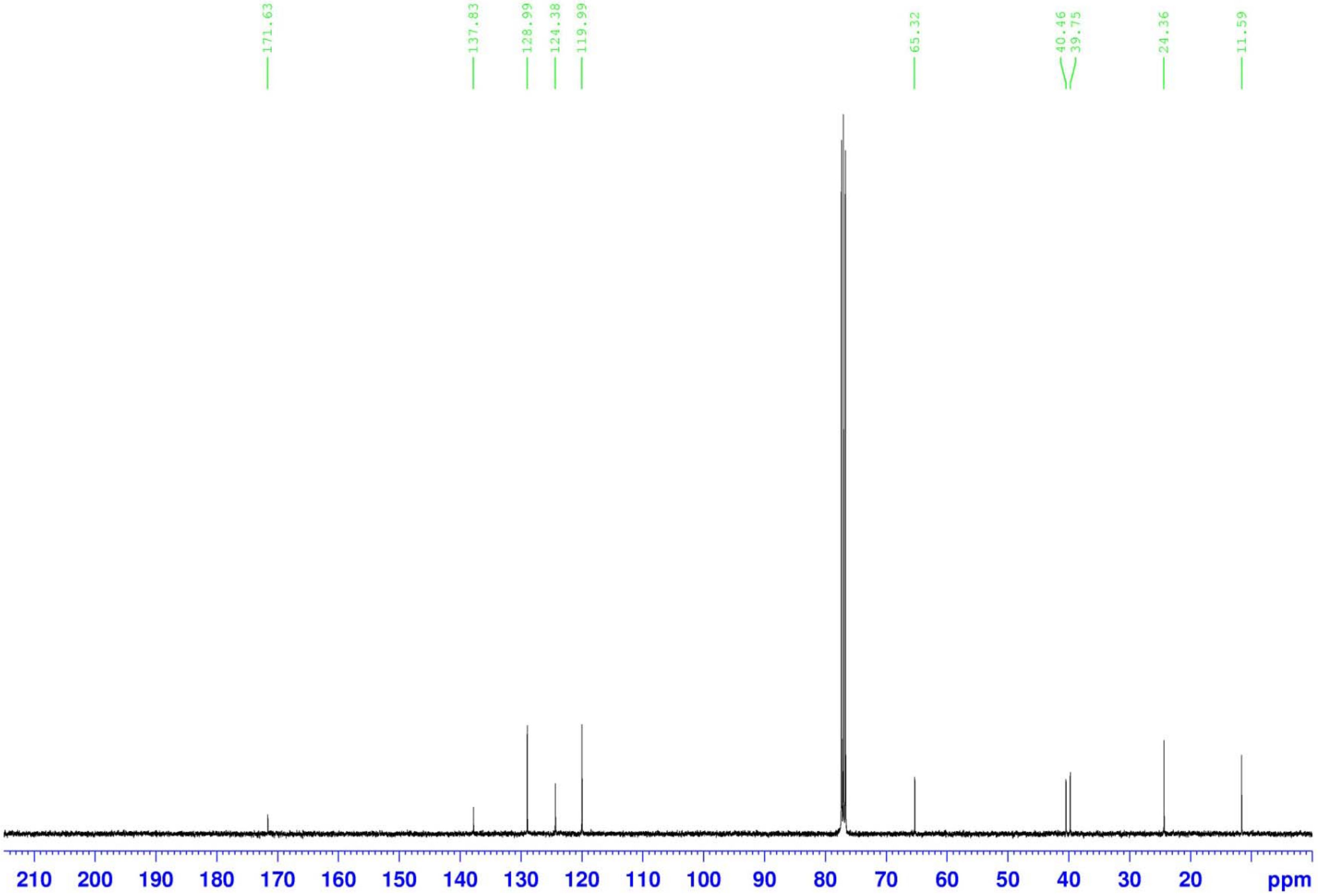
Oxidation of (*R*)-**8b** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (98%) as a white solid.

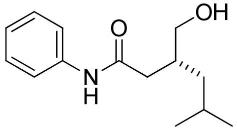
m.p.	118–119.5 ºC
Optical rotation	$[\alpha]_D^{20}$ = +2.1º (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 90:10 hexanes:isopropanol) showed peaks at 40 minutes (4.0% (S)) and 46 minutes (96.0% (R))
TLC analysis	R_f 0.3 (30:70 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.83 (1H, br s, NH), 7.52 (2H, d, *J* = 7.9 Hz, c,c’), 7.33 (2H, t, *J* = 7.8 Hz, b,b’), 7.12 (1H, t, *J* = 7.3 Hz, a), 3.78 (1H, dd, J_1 = 10.5 Hz, J_2 = 3.0 Hz, h), 3.59 (1H, dd, J_1 = 10.5 Hz, J_2 = 6.8 Hz, h), 2.87 (1H, br s, OH), 2.55–2.45 (2H, m, f), 2.10–2.00 (1H, g), 1.50–1.35 (2H, m, i), 0.98 (3H, t, *J* = 7.4 Hz, j).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.63 (e), 137.83 (d), 128.99 (c,c’), 124.38 (a), 119.99 (b,b’), 65.32 (h), 40.46 (f), 39.75 (g), 24.36 (i), 11.59 (j).
IR (neat)	3349 (O-H stretch), 3253 (N-H stretch), 2959, 1670 (C=O stretch), 1599, 1551 (N-H bend), 1443, 1319 (C-O stretch), 1255 (C-N stretch), 1143, 1074, 758 cm^{-1}.
HRMS (CI)	Calcd. for $C_{12}H_{18}NO_2$ (M+H): 208.1338, found 208.1333 *m/z*.

^{1}H NMR of (*R*)-3-(hydroxymethyl)pentanoic acid phenyl amide

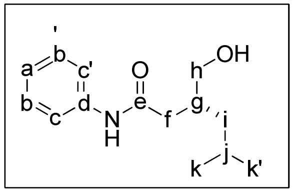


^{13}C NMR of (*R*)-3-(hydroxymethyl)pentanoic acid phenyl amide





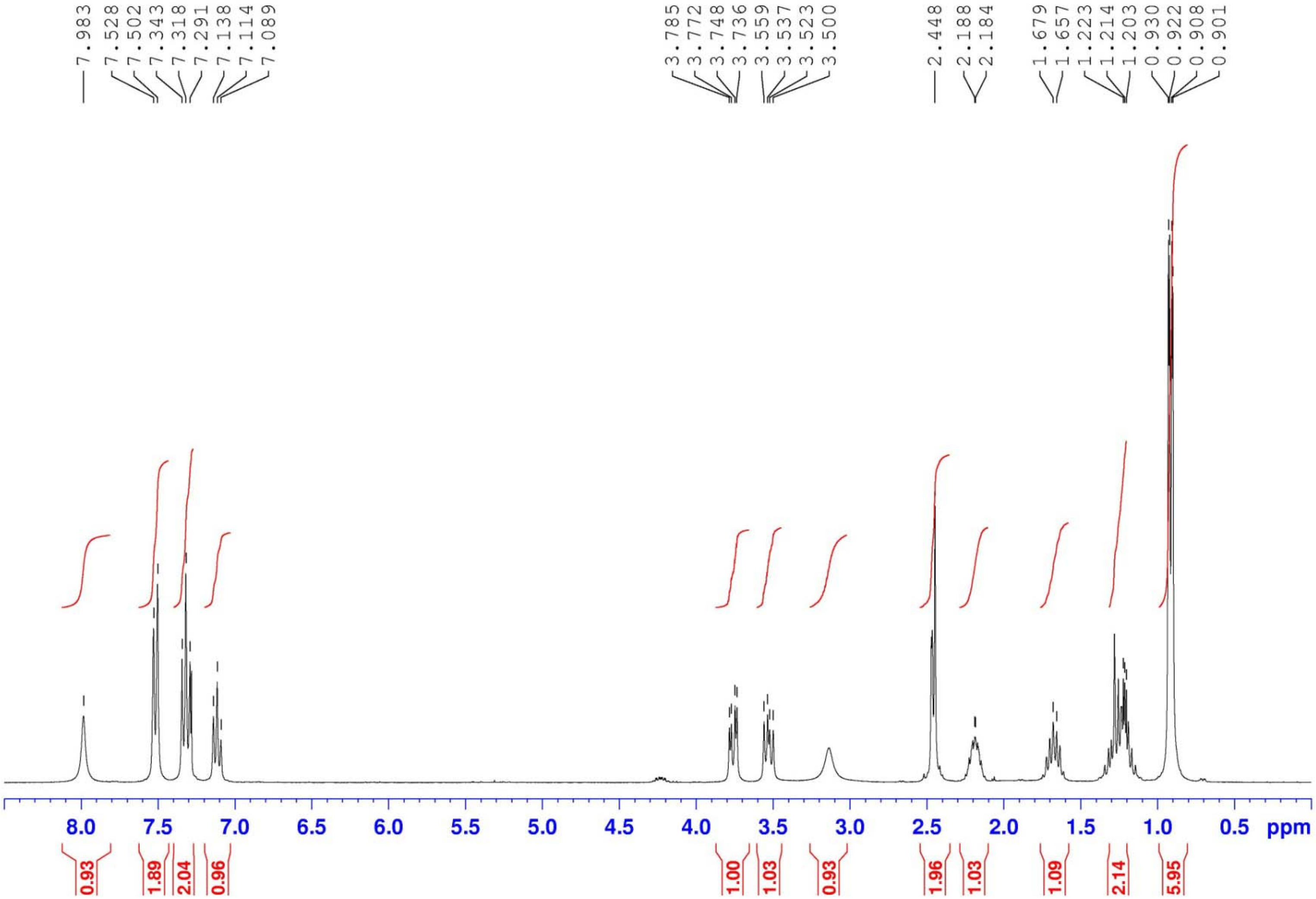
(*R*)-**17**



Using the general procedure for CAHB-oxidation of **3** affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (71%) as a white solid.

m.p.	92.5–94 ºC
Optical rotation	$[\alpha]_D^{20} = +5.1^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 90:10 hexanes:isopropanol) showed peaks at 27 minutes (2.5% (S)) and 30 minutes (97.5% (R))
TLC analysis	R_f 0.4 (30:70 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.98 (1H, br s, NH), 7.51 (2H, d, *J* = 7.8 Hz, c,c'), 7.32 (2H, t, *J* = 7.6 Hz, b,b'), 7.11 (1H, t, *J* = 7.4 Hz, a), 3.75 (1H, dd, *J* = 10.9 and 3.8 Hz, h), 3.52 (1H, dd, *J* = 10.9 and 6.8 Hz, h), 3.14 (1H, br s, OH), 2.45–2.40 (2H, m, f), 2.30–2.15 (1H, m, g), 1.75–1.60 (1H, m, j), 1.30–1.10 (2H, m, i), 0.92 and 0.91 (6H, overlapping d's, *J* = 6.5 Hz, k,k')
^{13}C NMR (75 MHz, $CDCl_3$)	δ 171.71 (e), 137.85 (d), 128.97 (b,b'), 124.36 (a), 120.06 (c,c'), 65.73 (h), 40.89 (i), 40.72 (f), 35.80 (g), 25.27 (j), 22.78 (k), 22.65 (k').
IR (neat)	3307 (O-H stretch), 3150 (N-H stretch), 2951, 2925, 2856, 1641 (C=O stretch), 1547 (N-H bend), 1452, 1355 (C-O stretch), 1205 (C-N stretch), 1143, 1077, 1032, 878, 735, 695 cm^{-1}.
HRMS (CI)	Calcd. for $C_{14}H_{22}NO_2$ (M+H): 236.1651, found 236.1651 *m/z*.

^{1}H NMR of (*R*)-17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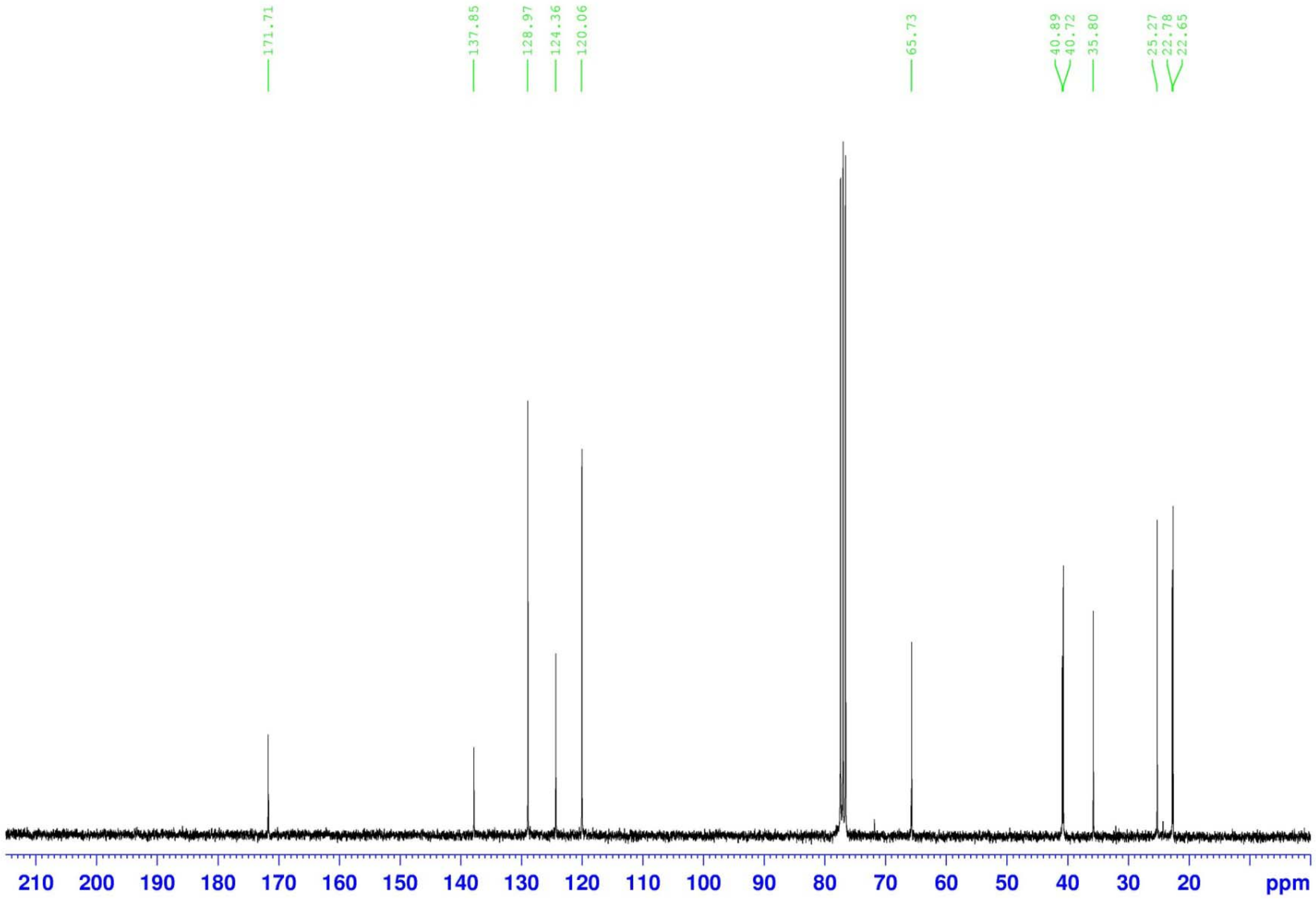
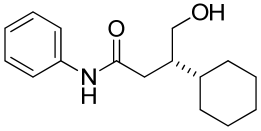
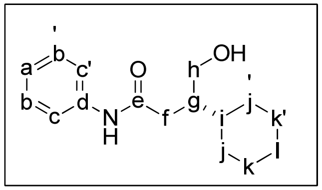
^{13}C NMR of (*R*)-17



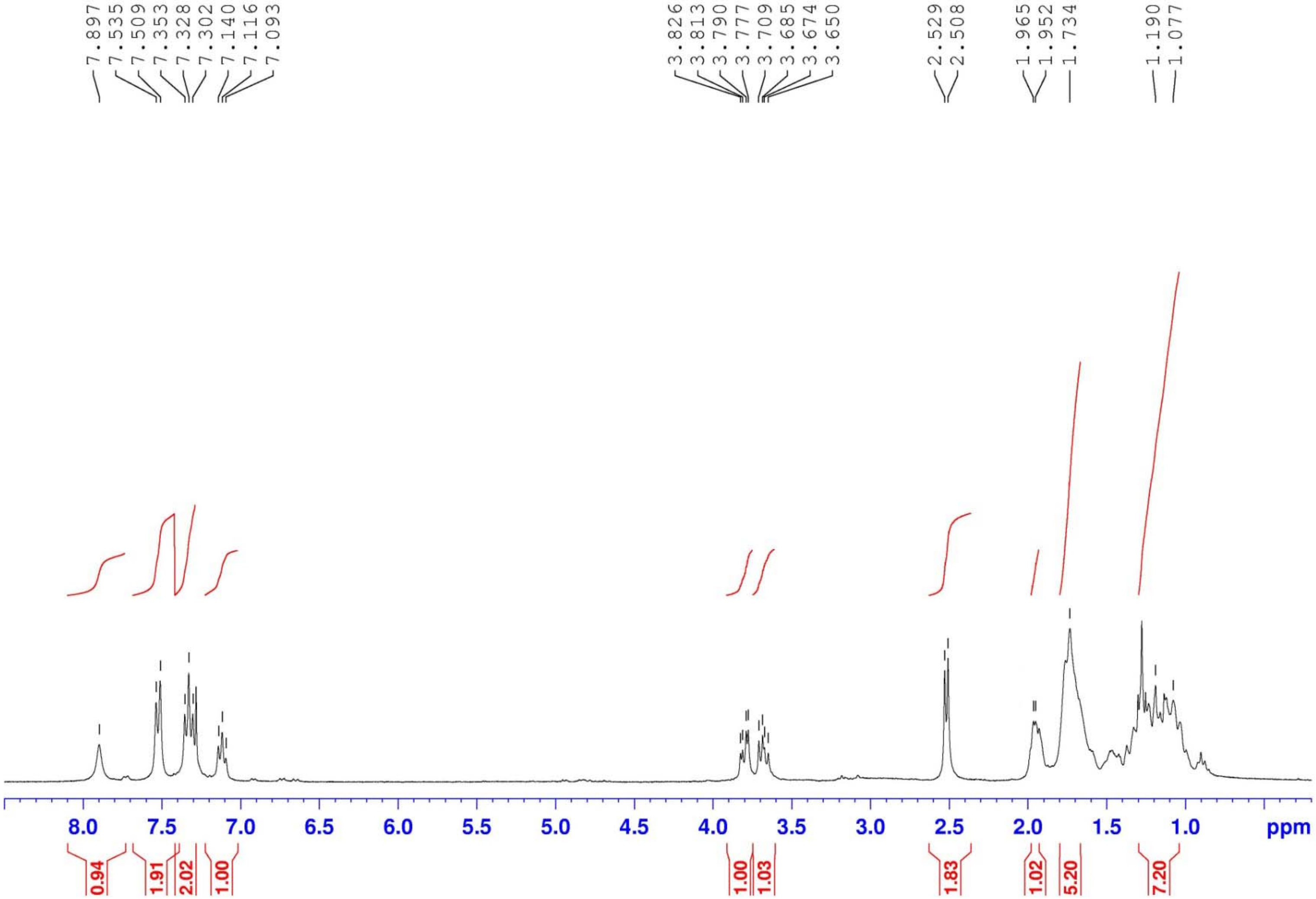




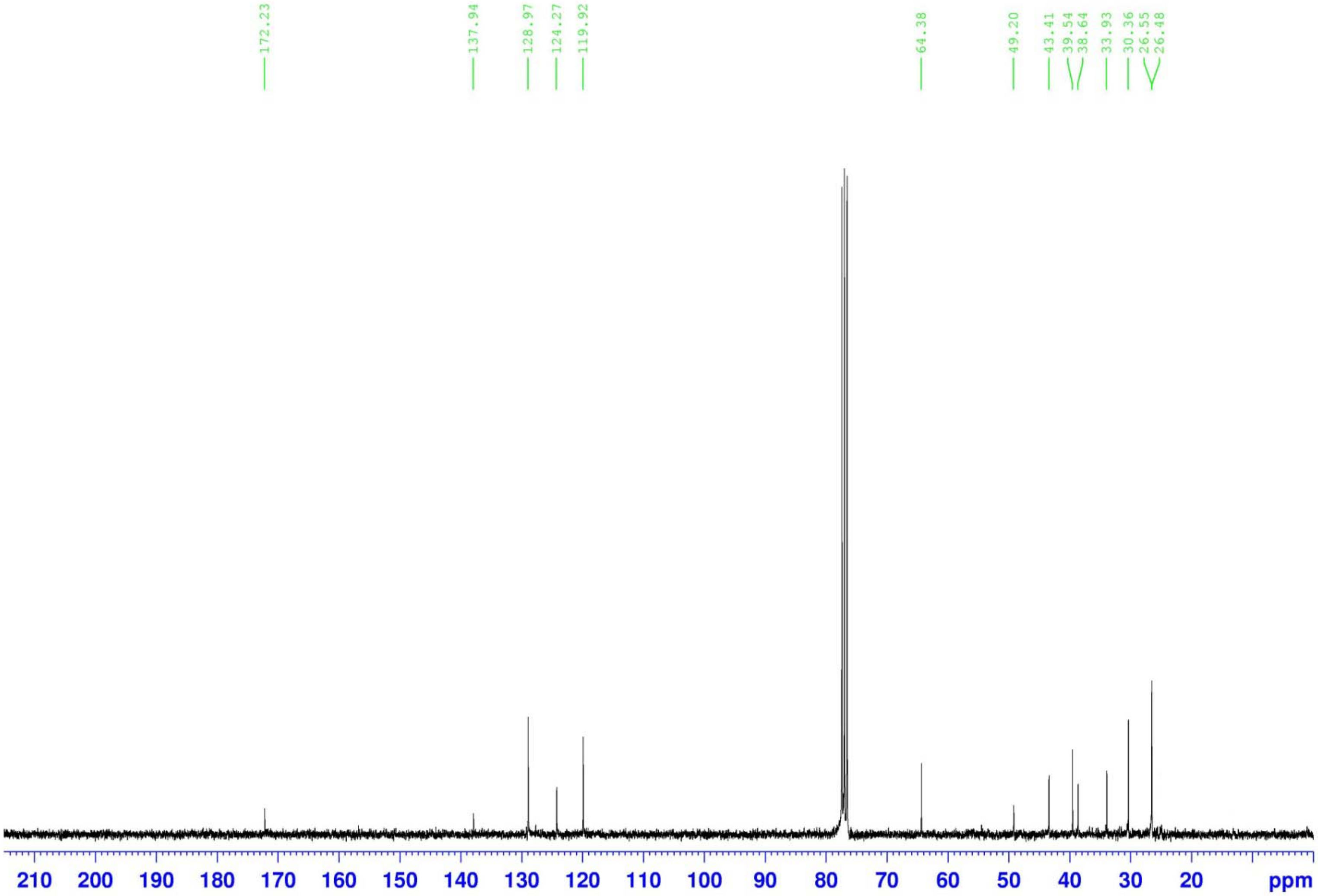
Oxidation of (*S*)-**8e** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (96%) as a white solid.

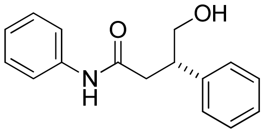
m.p.	88–89 ºC
Optical rotation	$[\alpha]_D^{20} = +3.1^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 90:10 hexanes:isopropanol) showed peaks at 48 minutes (5.0% (S)) and 52 minutes (95.0% (R))
TLC analysis	R_f 0.4 (30:70 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.90 (1H, br s, NH), 7.52 (2H, d, *J* = 7.9 Hz, c,c'), 7.33 (2H, t, *J* = 7.9 Hz, b,b'), 7.12 (1H, t, *J* = 7.2 Hz, a), 3.82 (1H, dd, *J* = 10.8 and 4.0 Hz, h), 3.67 (1H, dd, *J* = 10.8 and 7.1 Hz, h), 2.51 (2H, d, *J* = 6.2 Hz, f), 2.00–1.80 (1H, m, g), 1.80–1.60 (5H, m, j,j',k,k',OH), 1.35–1.00 (7H, m, i,j,j',k,k',l).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 172.23 (e), 137.94 (d), 128.97 (c,c'), 124.27 (a), 119.92 (b,b'), 64.38 (h), 49.20 (g), 43.41 (f), 39.54 (i), 38.64 (j), 33.93 (j'), 30.36 (l), 26.55 (k), 26.48 (k').
IR (neat)	3292 (O-H stretch), 3100 (N-H stretch), 2957, 2893, 1655 (C=O stretch), 1599, 1540 (N-H bend), 1444, 1327 (C-O stretch), 1268 (C-N stretch), 1121, 1053, 881, 753 cm^{-1}.
HRMS (CI)	Calcd. for $C_{16}H_{24}NO_2$ (M+H): 262.1807, found 262.1808 *m/z*.

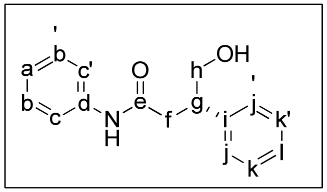
^{1}H NMR of (*S*)-3-cyclohexyl-4-hydroxybutanoic acid phenyl amide



^{13}C NMR of (*S*)-3-cyclohexyl-4-hydroxybutanoic acid phenyl amide



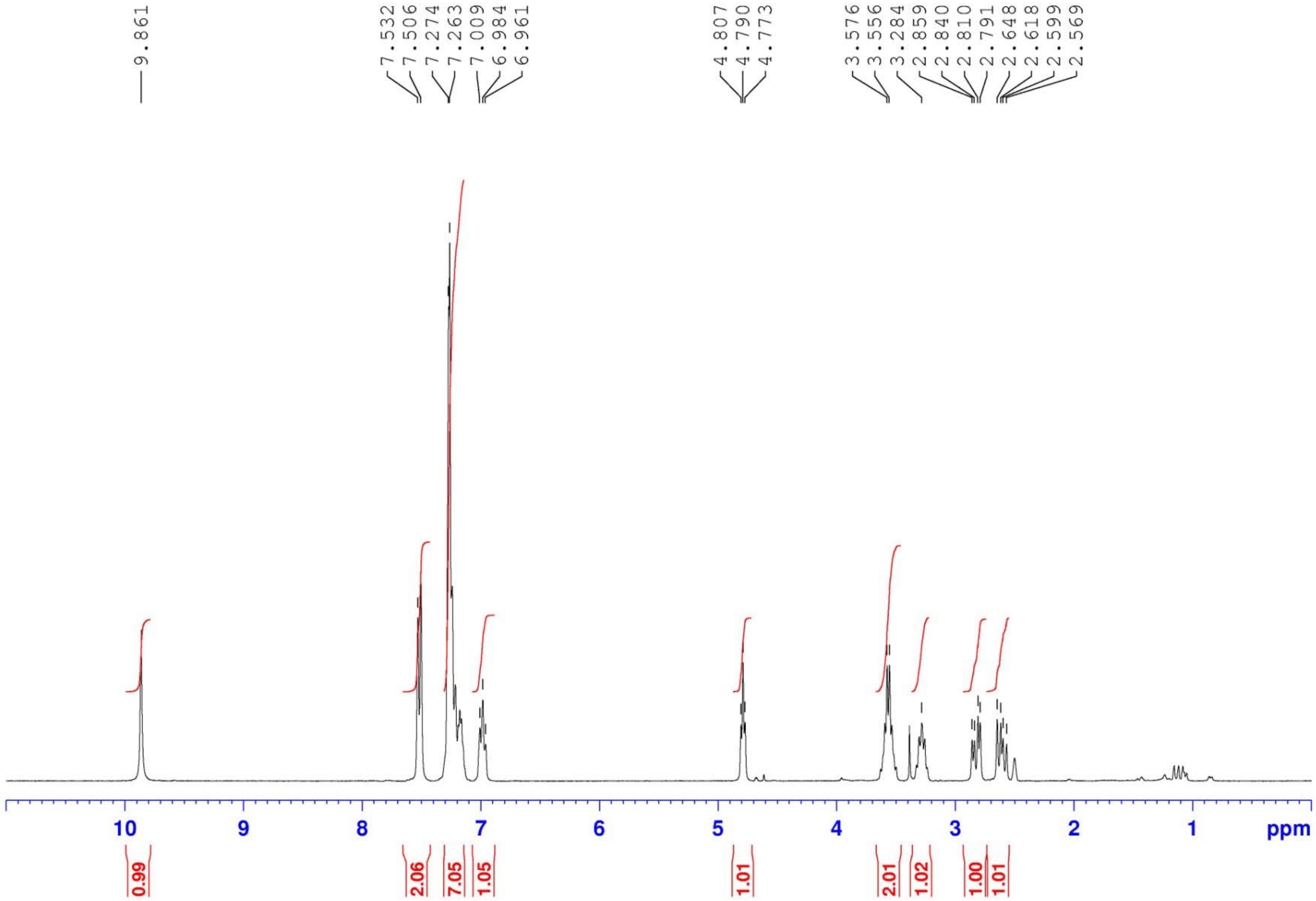




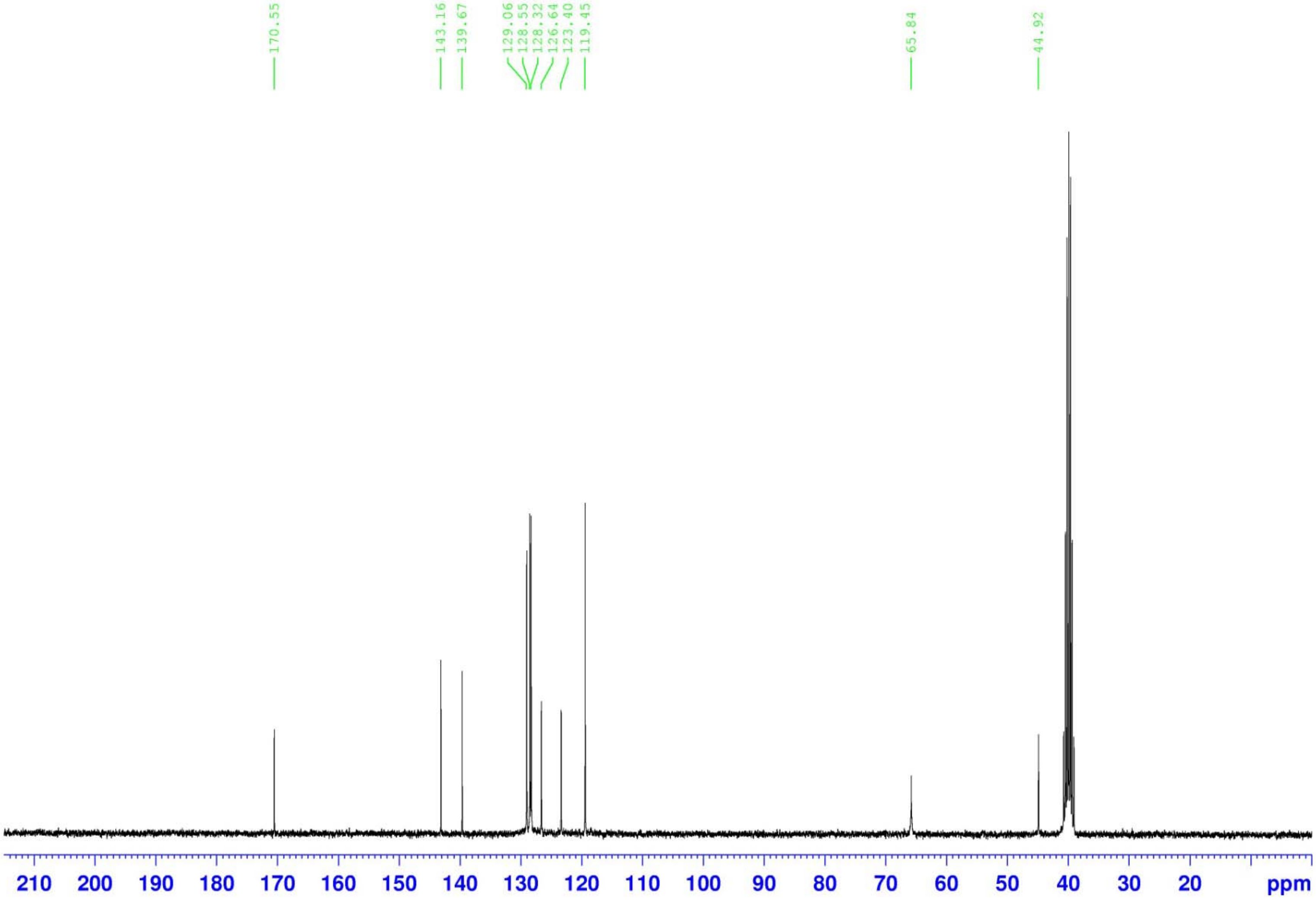
Oxidation of (*S*)-**8f** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (98%) as a white solid.

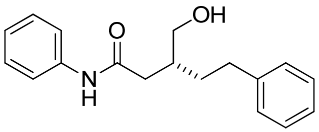
m.p.	95.5–97 ºC
Optical rotation	$[\alpha]_D^{20} = +3.8^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 90:10 hexanes:isopropanol) showed peaks at 56 minutes (3.5% (S)) and 85 minutes (96.5% (R))
TLC analysis	0.4 (30:70 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, DMSO-d$_6$)	δ 9.86 (1H, br s, NH), 7.51 (2H, d, *J* = 8.0 Hz, c,c'), 7.30–7.10 (7H, m, b,b',j,j',k,k',l), 6.98 (1H, t, *J* = 7.3 Hz, a), 4.79 (1H, t, *J* = 5.2 Hz, OH), 3.65–3.50 (2H, m, h), 3.35–3.20 (1H, m, g), 2.82 and 2.60 (2H, overlapping dd's, J_1 = 14.8 Hz, 5.9 Hz, J_2 = 14.8 Hz, 8.9 Hz, f).
^{13}C NMR (75 MHz, DMSO-d$_6$)	δ 170.55 (e), 143.16 (d), 139.67 (i), 129.06 (j,j'), 128.55 (b,b'), 128.32 (k,k'), 126.64 (l), 123.40 (a), 119.45 (c,c'), 65.84 (h), 44.92(f).
IR (neat)	3365 (O-H stretch), 3254, 3191 (N-H stretch), 1667 (C=O stretch), 1607, 1597, 1550 (N-H bend), 1498, 1443, 1316 (C-O stretch), 1254 (C-N stretch), 1189, 1132, 960, 852, 756 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{16}H_{18}NO_2$ (M+H): 256.1338, found 256.1337 *m/z*.

^{1}H NMR of (*S*)-4-hydroxy-3-phenylbutanoic acid phenyl amide

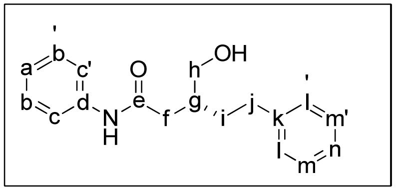


^{13}C NMR of (*S*)-4-hydroxy-3-phenylbutanoic acid phenyl amide





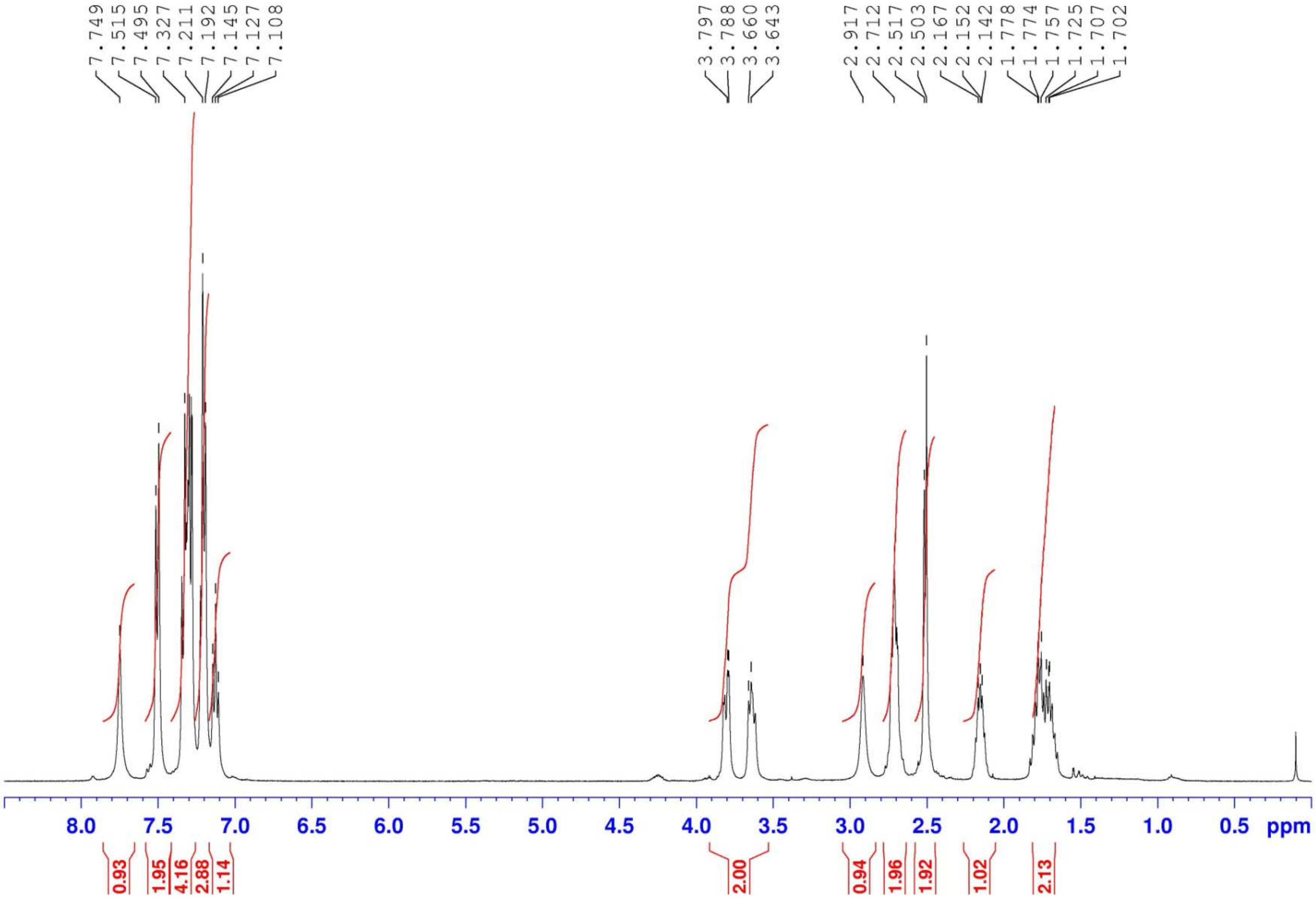
(*R*)-**18**



Using the general procedure for CAHB-oxidation of **7c** affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (71%) as a white solid.

m.p.	92–94 °C
Optical rotation	$[\alpha]_D^{20}$ = +4.3° (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 85:15 hexanes:isopropanol) showed peaks at 23 minutes (3.0% (S)) and 26 minutes (97.0% (R))
TLC analysis	R_f 0.4 (30:70 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.75 (1H, br s, NH), 7.50 (2H, d, *J* = 7.8 Hz, c,c’), 7.35–7.25 (4H, m, b,b’,l,l’), 7.25–7.15 (3H, m, m,m’,n), 7.13 (1H, t, *J* = 7.4 Hz, a), 3.85–3.75 (1H, m, h), 3.70–3.60 (1H, m, h), 2.92 (1H, br s, OH), 2.80–2.65 (2H, m, j), 2.55–2.45 (2H, m, f), 2.20–2.10 (1H, m, g), 1.80–1.65 (2H, m, i).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.32 (e), 141.89 (k), 137.75 (d), 129.01 (l,l’), 128.48 (b,b’), 128.37 (m,m’), 125.98 (n), 124.44 (a), 120.02 (c,c’), 65.36 (h), 40.60 (f), 37.77 (j), 33.41 (g), 33.11 (i).
IR (neat)	3679 (O-H stretch), 3150 (N-H stretch), 2993, 2950, 1678 (C=O stretch), 1598, 1498 (N-H bend), 1443, 1322 (C-O stretch), 1304, 1197 (C-N stretch), 1174, 1095, 957, 762 cm^{-1}.
HRMS (CI)	Calcd. for $C_{18}H_{22}NO_2$ (M+H): 284.1651, found 284.1656 *m/z*.

^{1}H NMR of (*R*)-18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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
0.93
1.95
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2.00
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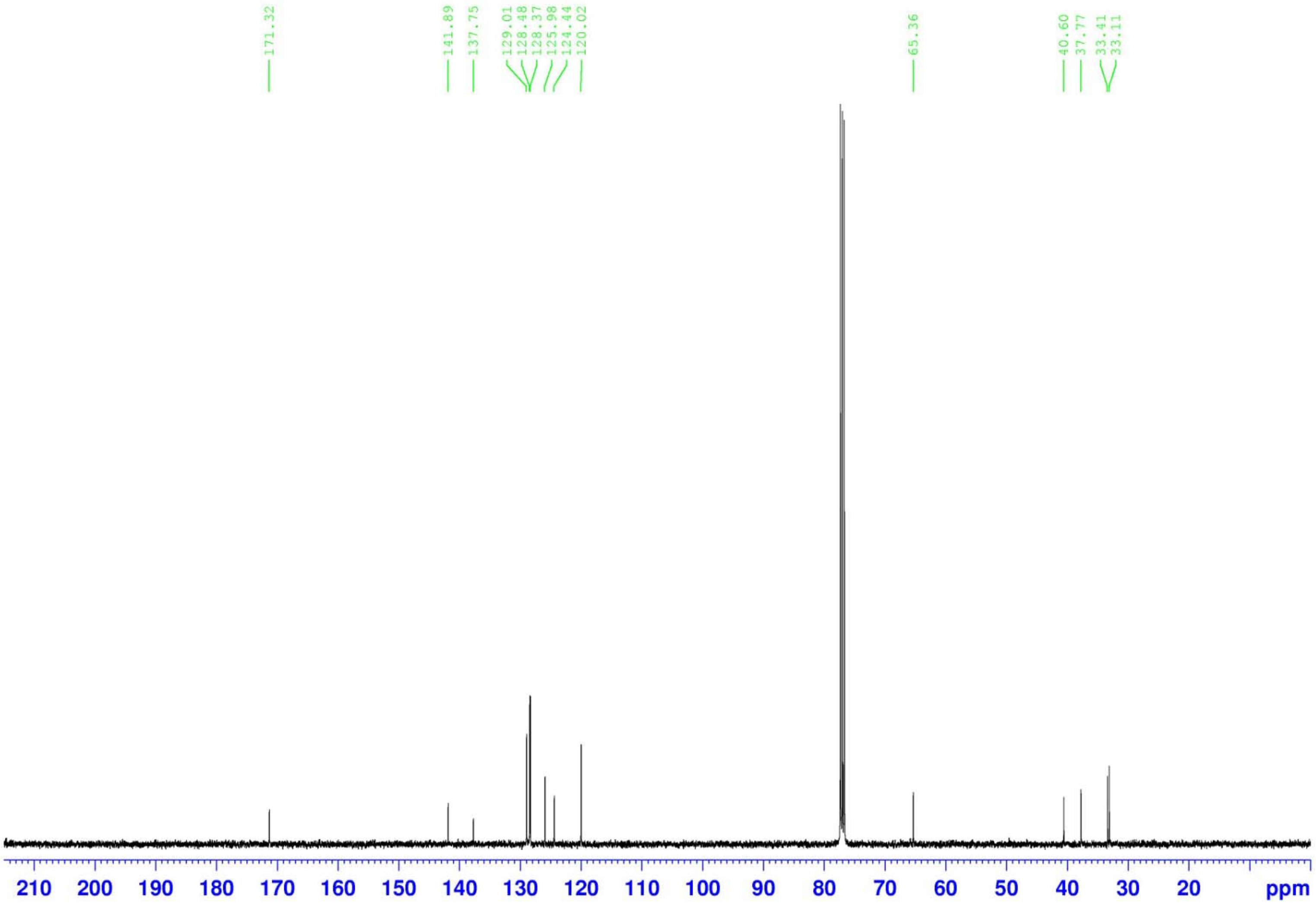
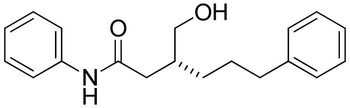
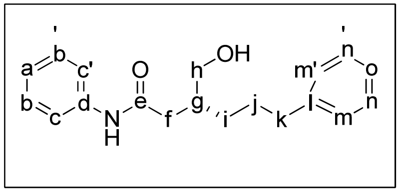
1.92
1.02
2.13

^{13}C NMR of (*R*)-18



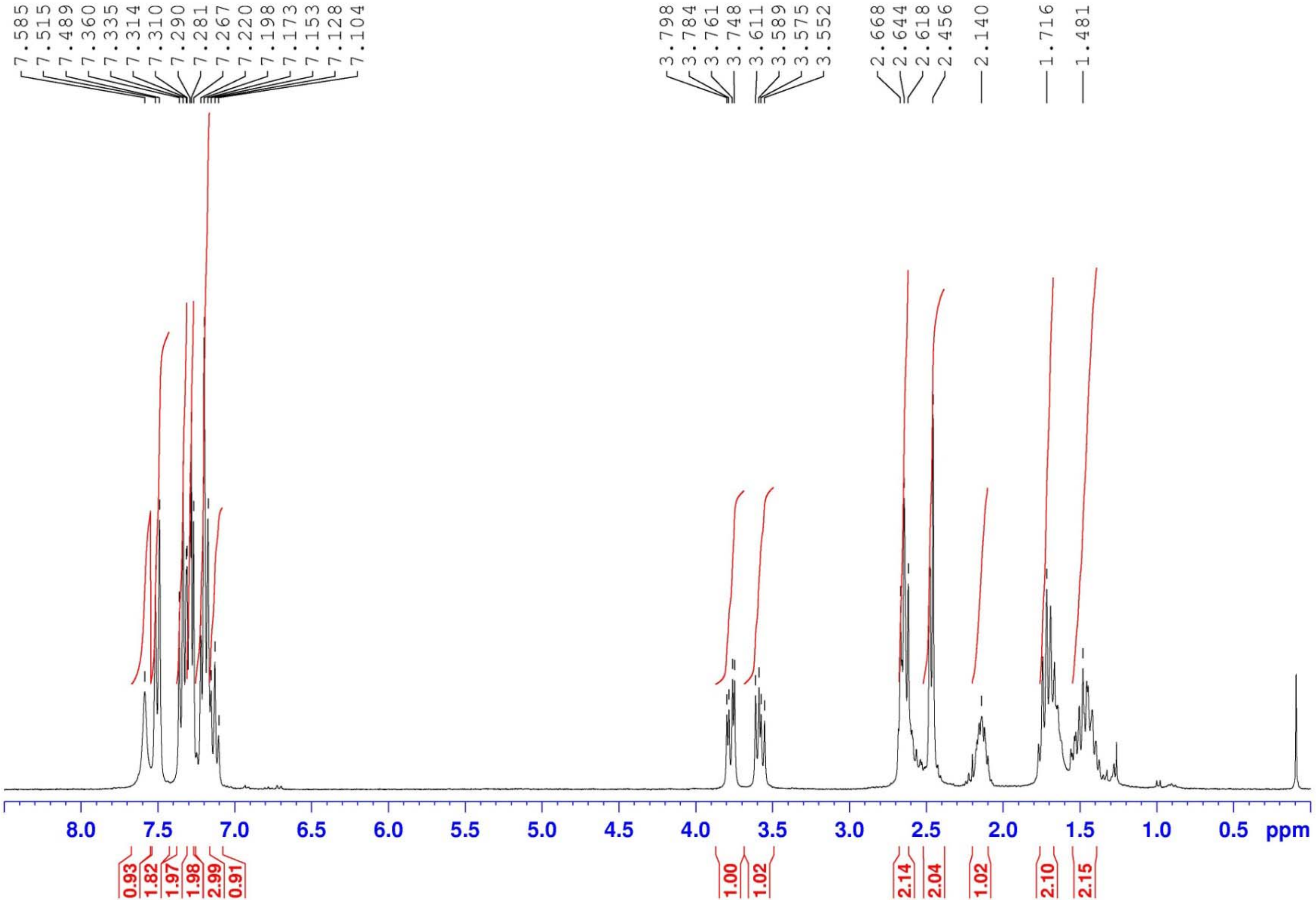




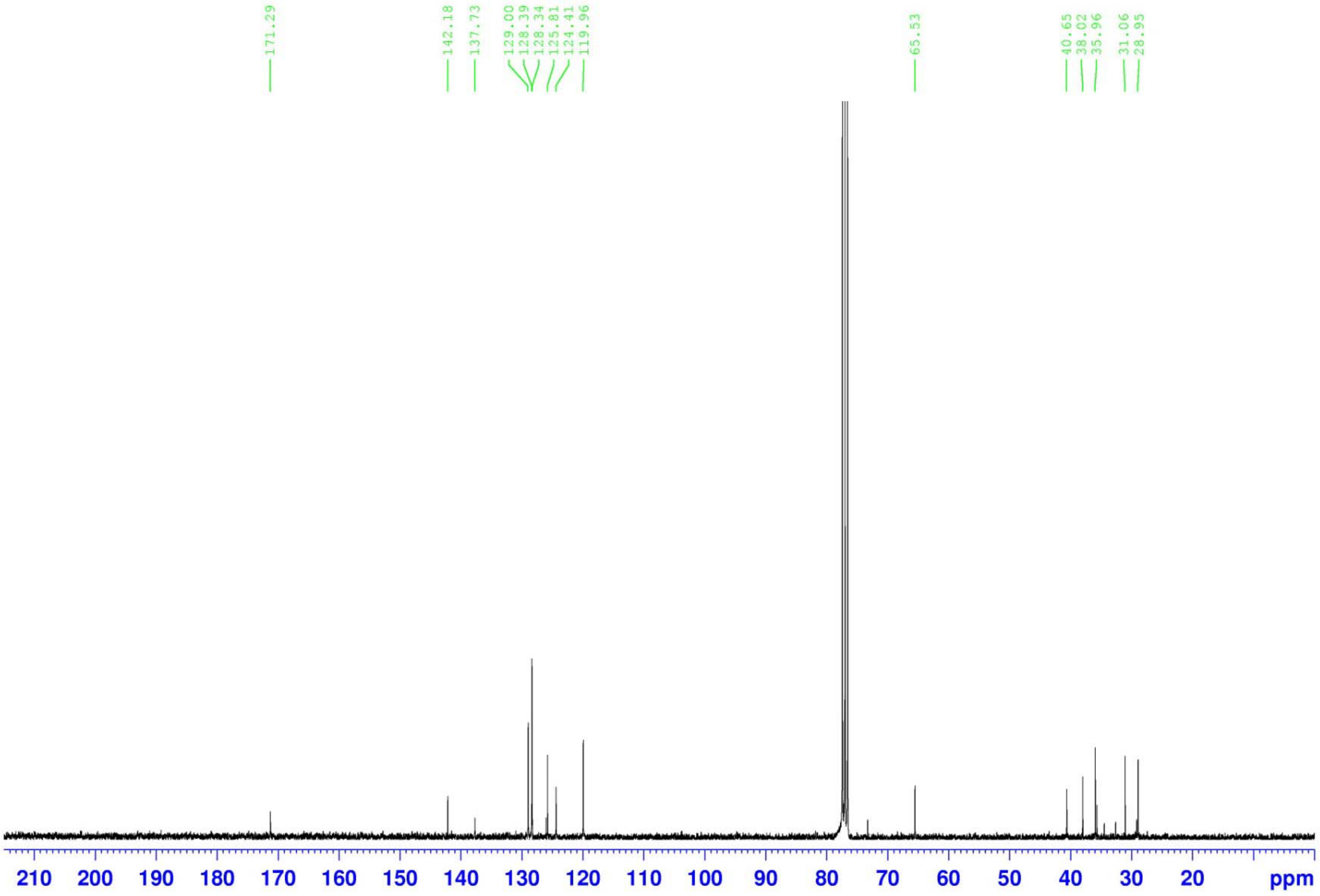
Following Oxidation of (*R*)-**8d** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (98%) as a white solid.

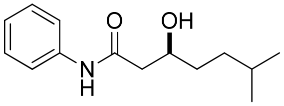
m.p.	78.5–80 ºC
Optical rotation	$[\alpha]_D^{20} = +4.1^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-IC, 90:10 hexanes:isopropanol) showed peaks at 35 minutes (4.0% (S)) and 37 minutes (96.0% (R))
TLC analysis	R_f 0.5 (30:70 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.58 (1H, br s, NH), 7.50 (2H, d, *J* = 7.9 Hz, m,m'), 7.40–7.30 (2H, m, c,c'), 7.30–7.25 (2H, m, b,b'), 7.25–7.15 (3H, m, n,n',o), 7.13 (1H, t, *J* = 7.5 Hz, a), 3.77 (1H, dd, *J* = 10.9 and 4.0 Hz, h), 3.58 (1H, dd, *J* = 10.9 and 6.7 Hz, h), 2.64 (2H, t, *J* = 7.3 Hz, k), 2.50–2.40 (2H, m, f), 2.20–2.10 (1H, m, g), 1.80–1.60 (2H, m, i), 1.60–1.40 (2H, m, j).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 171.29 (e), 142.18 (d), 137.73 (l), 129.00 (b,b'), 128.39 (m,m'), 128.34 (n,n'), 125.81 (o), 124.41 (a), 119.96 (c,c'), 65.53 (h), 40.65 (f), 38.02 (g), 35.96 (k), 31.06 (i), 28.95 (j).
IR (neat)	3714 (O-H stretch), 3040 (N-H stretch), 2890, 1680 (C=O stretch), 1601, 1590, 1545 (N-H bend), 1499, 1451, 1369, 1308 (C-O stretch), 1204 (C-N stretch), 1186, 1100, 1020, 990, 756 cm^{-1}.
HRMS (CI)	Calcd. for $C_{19}H_{24}NO_2$ (M+H): 298.1807, found 298.1800 *m/z*.

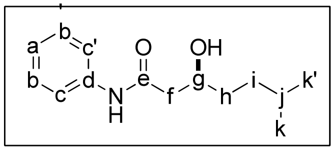
^{1}H NMR of (*R*)-3-(hydroxymethyl)-6-phenylhexanoic acid phenyl amide



^{13}C NMR of (*R*)-3-(hydroxymethyl)-6-phenylhexanoic acid phenyl amide



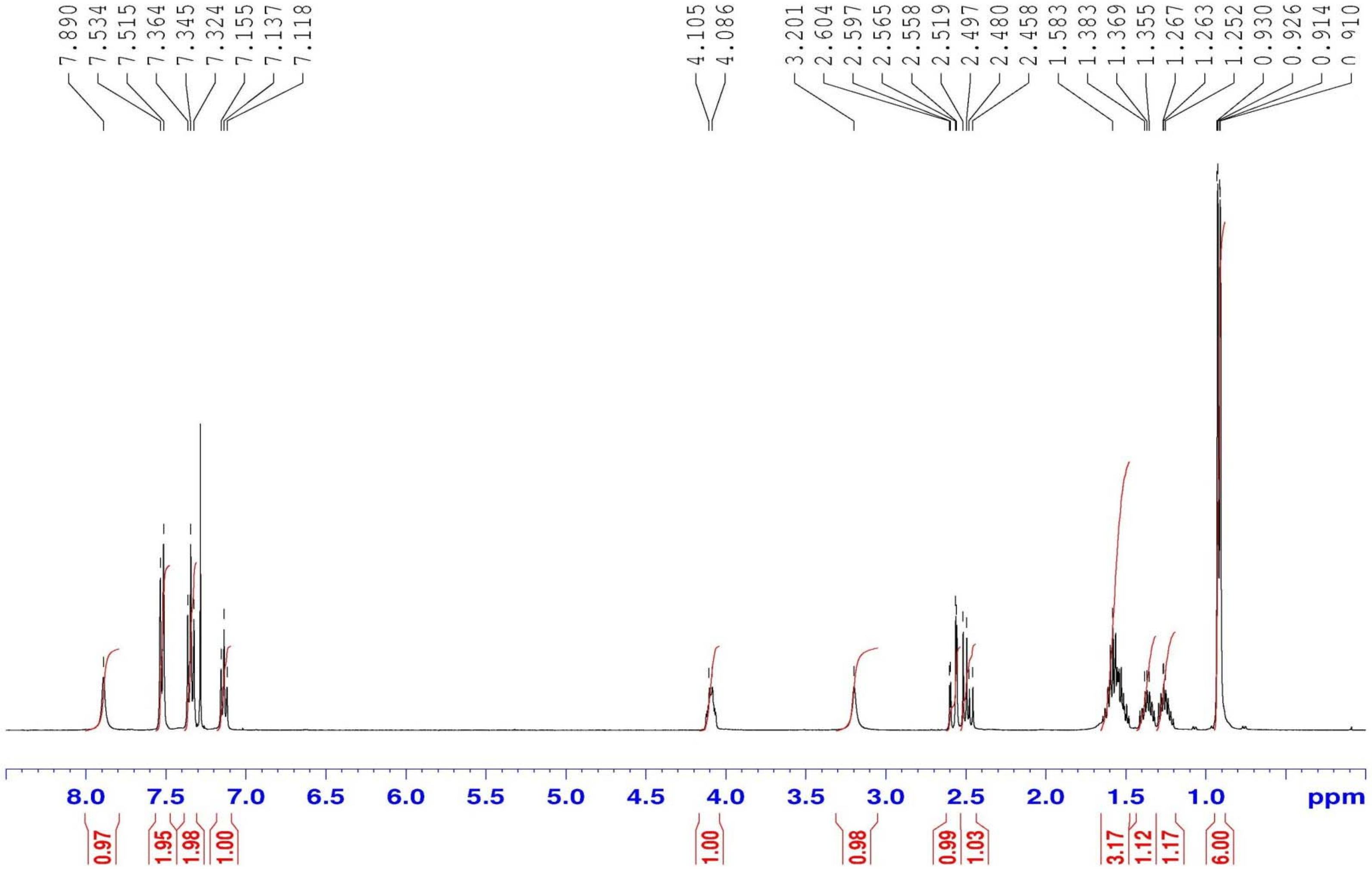




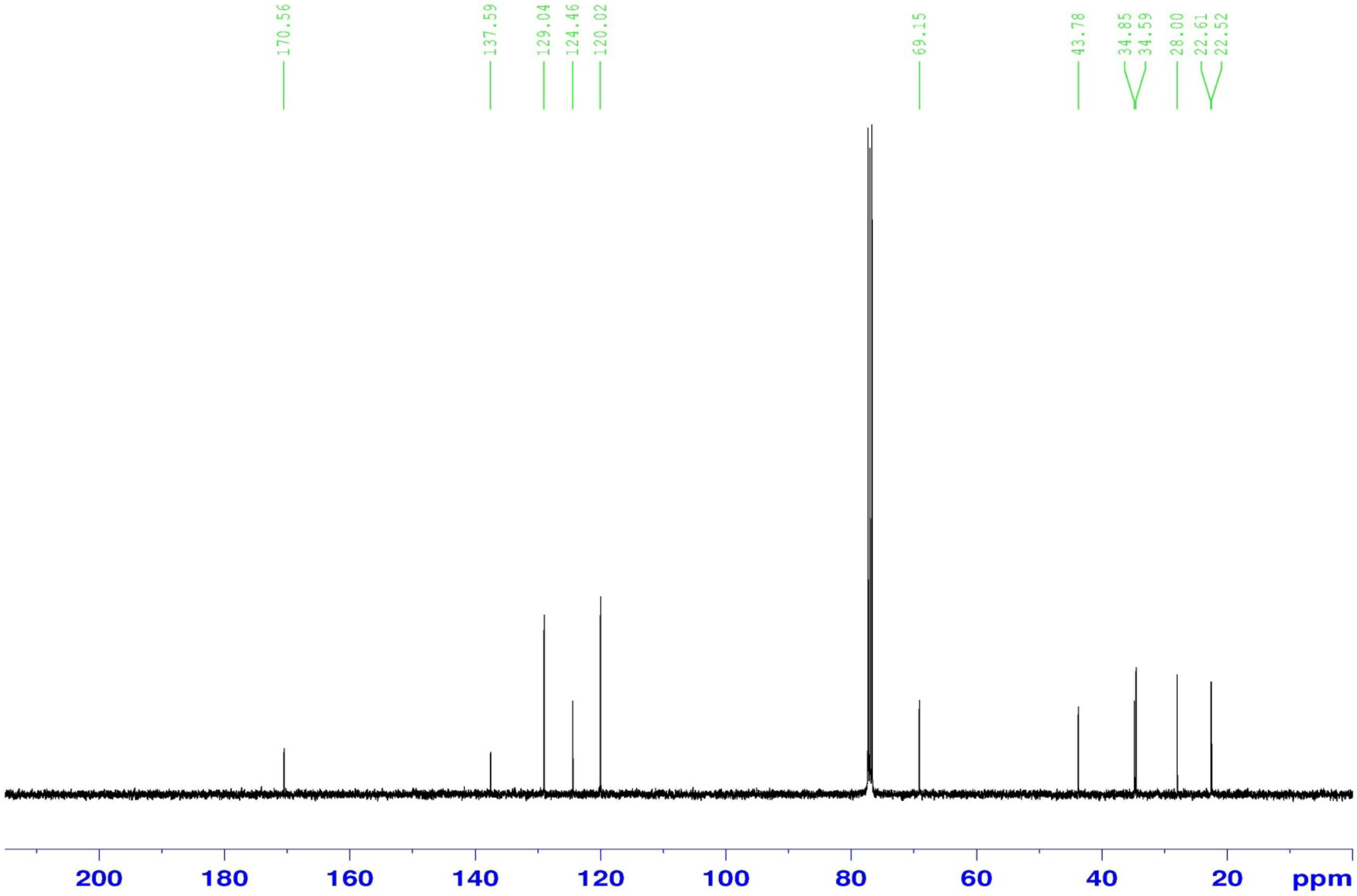
Following Oxidation of (*S*)-**2** using the general procedure for small scale oxidation of purified organoboronates with H_2O_2 affords, after flash chromatography on silica gel (80–40:20–60 hexanes:ethyl acetate), the title compound (98%) as a white solid.

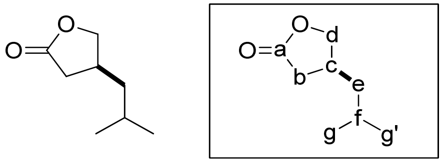
m.p.	130.2-131.7 ^{o}C
Optical rotation	$[\alpha]_D^{20}$ = +6.8^{o} (*c* 0.5, ethanol)
HPLC analysis	(Chiralcel-ASH, 90:10 hexanes:isopropanol) showed peaks at 41.1 minutes (2.5% (R)) and 48.3 minutes (97.5% (S)).
TLC analysis	R_f 0.4 (50:50 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.89 (1H, br s, NH), 7.52 (2H, d, *J* = 7.7 Hz, c,c'), 7.35 (2H, t, *J* = 7.6 Hz, b,b'), 7.14 (1H, t, *J* = 7.4 Hz, a), 4.15-4.00 (1H, m, g), 3.20 (1H, br s, OH), 2.58 and 2.49 (2H, overlapping dd's, J_1 = 15.5 Hz, 2.7 Hz, J_2 = 15.5 Hz, 8.8 Hz, f) 1.70-1.50 (3H, m, h,i), 1.40-1.30 (1H, m, i), 1.30-1.15 (1H, m, j), 0.92 (3H, d, *J* = 6.6 Hz, k), 0.91 (3H, d, *J* = 6.6 Hz, k').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 170.56 (e), 137.59 (d), 129.04 (b,b'), 124.46 (a), 120.02 (c,c'), 69.15 (g), 43.78 (f), 34.85 (h), 34.59 (i), 28.00 (j), 22.61 (k), 22.52 (k').
IR (neat)	3286 (N-H stretch), 2953, 2869, 1663 (C=O stretch), 1598, 1548, 1442, 1314, 1256, 1079, 716, 692 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{14}H_{22}NO_2$ (M+H): 236.1651, found 236.1645 *m/z*.

^{1}H NMR of (*S*)-3-hydroxy-6-methylheptanoic acid phenyl amide



^{13}C NMR of (*S*)-3-hydroxy-6-methylheptanoic acid phenyl amide



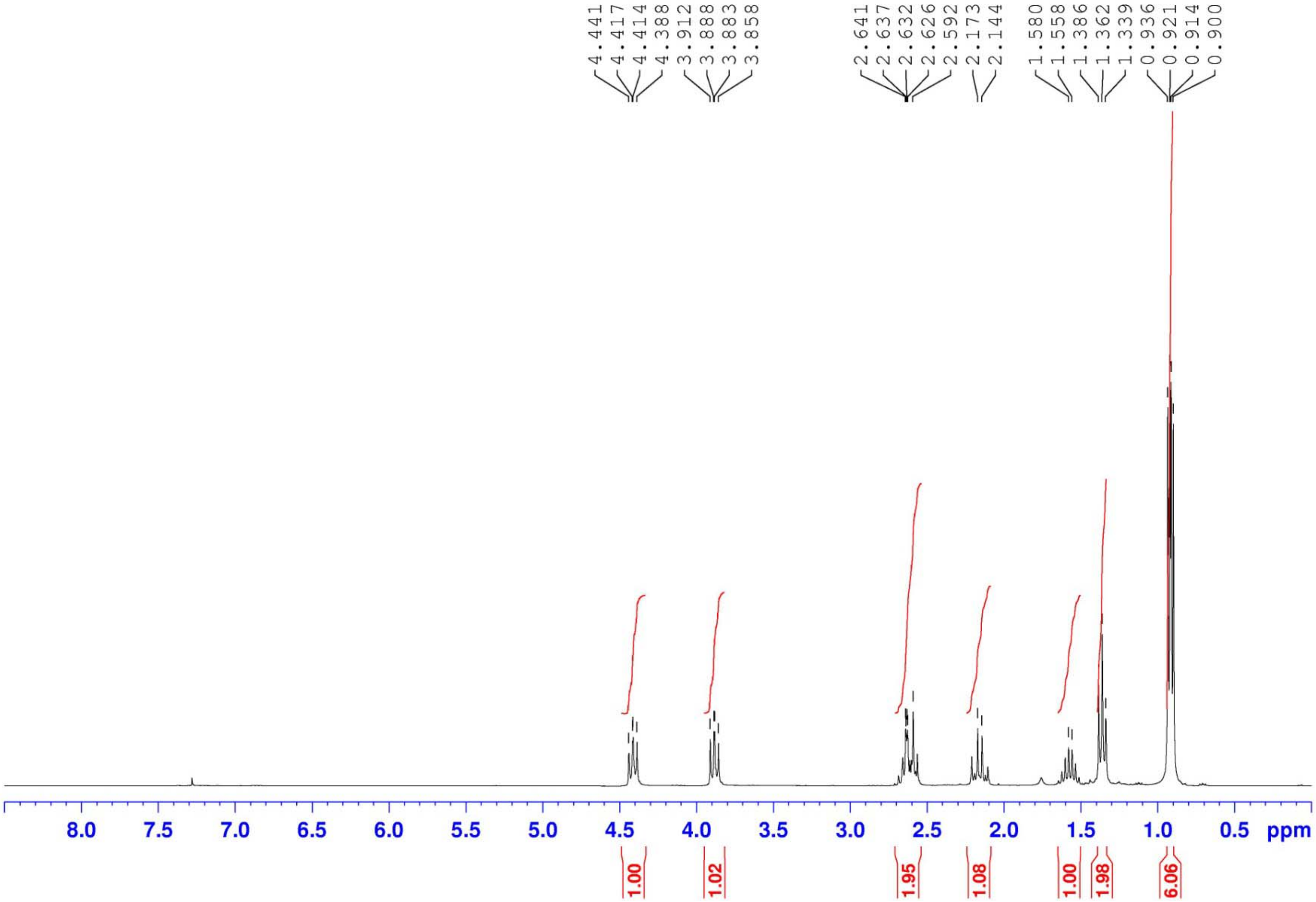


(*S*)-**20**

Following the procedure for lactonization via CAHB-oxidation of **7i** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (78%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20}$ = -1.5° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.5 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 4.41 (1H, dd, J_1 = 8.8 Hz, J_2 = 8.1 Hz, d), 3.88 (1H, dd, J_1 = 8.9 Hz, J_2 = 8.6 Hz, d), 2.70–2.55 (2H, m, b), 2.25–2.10 (1H, m, c), 1.65–1.50 (1H, m, f), 1.36 (2H, t, J = 7.1 Hz, e), 0.93 (3H, t, J = 6.6 Hz, g), 0.90 (3H, t, J = 6.6 Hz, g').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 177.22 (a), 73.56 (d), 42.21 (e), 34.76 (b), 33.83 (c), 26.28 (f), 22.64 (g), 22.40 (g').
IR (neat)	2956, 2903, 1773 (C=O stretch), 1469, 1420, 1367, 1216, 1168 (C-O stretch), 1011, 913, 838, 730, 646, 557 cm^{-1}.

^{1}H NMR of (*S*)-20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2.173
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1.339
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0.900
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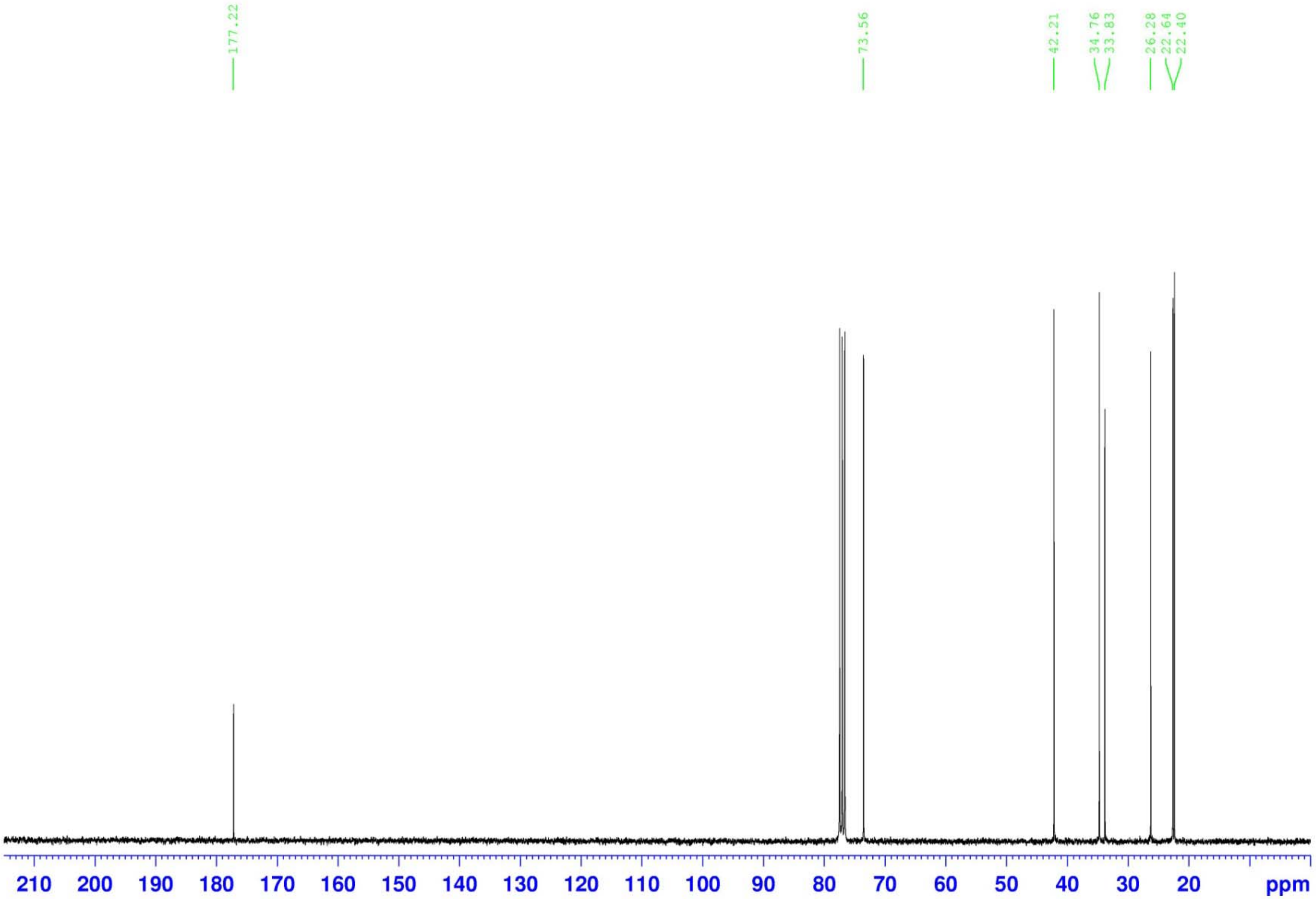
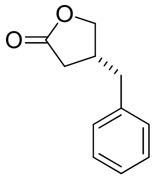
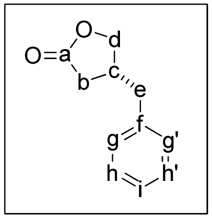
^{13}C NMR of (*S*)-20





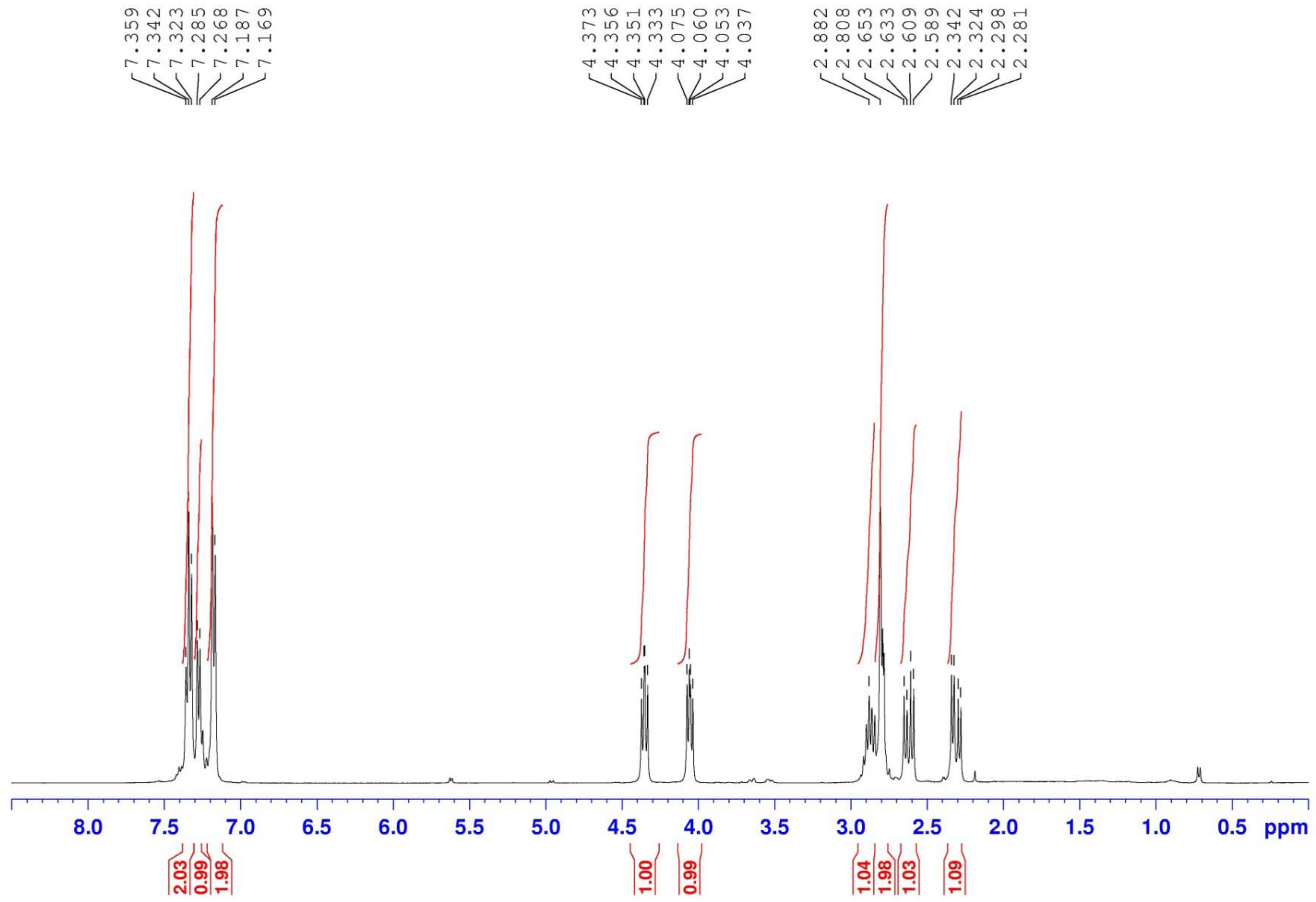
(*R*)-**22a**



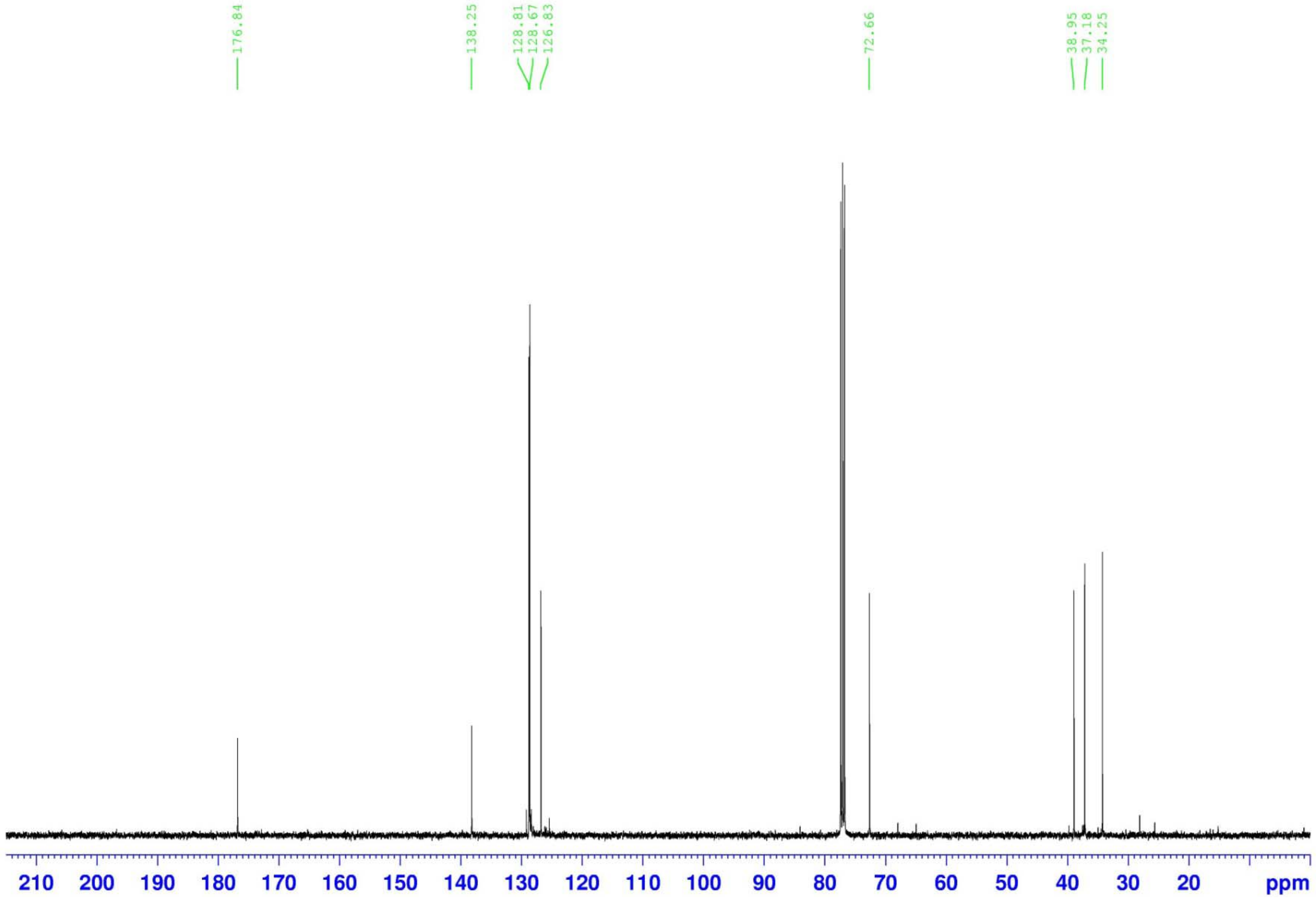
Following the procedure for lactonization via CAHB-oxidation of **21a** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (80%) as a light yellow oil.

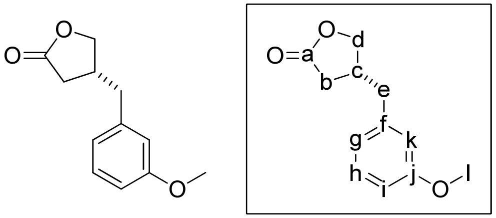
Optical rotation	$[\alpha]_D^{20} = +5.3^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-AD, 95:5 hexanes:isopropanol) shows peaks at 36 minutes (97.5% (R)) and 40 minutes (2.5% (S))
TLC analysis	R_f 0.4 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.34 (2H, t, *J* = 7.2 Hz, h,h'), 7.27 (1H, d, *J* = 6.9 Hz, i), 7.17 (2H, d, *J* = 7.3 Hz, g,g'), 4.35 (1H, dd, J_1 = 8.9 Hz, J_2 = 8.9 Hz, d), 4.05 (1H, dd, J_1 = 6.2 Hz, J_2 = 6.1 Hz, d), 2.95–2.85 (1H, m, c), 2.85–2.75 (2H, m, e), 2.62 (1H, dd, J_1 = 17.4 Hz, J_2 = 7.9 Hz, b), 2.31 (1H, dd, J_1 = 17.4 Hz, J_2 = 6.9 Hz, b).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 176.84 (a), 138.25 (f), 128.81 (g,g'), 128.67 (h,h'), 126.83 (i), 72.66 (d), 38.95 (e), 37.18 (b), 34.25 (c).
IR (neat)	2963, 2909, 1773 (C=O stretch), 1496, 1417, 1257, 1166 (C-O stretch), 1088, 1012, 910, 797, 731, 699, 638, 531 cm^{-1}.

^{1}H NMR of (*R*)-22a



^{13}C NMR of (*R*)-22a



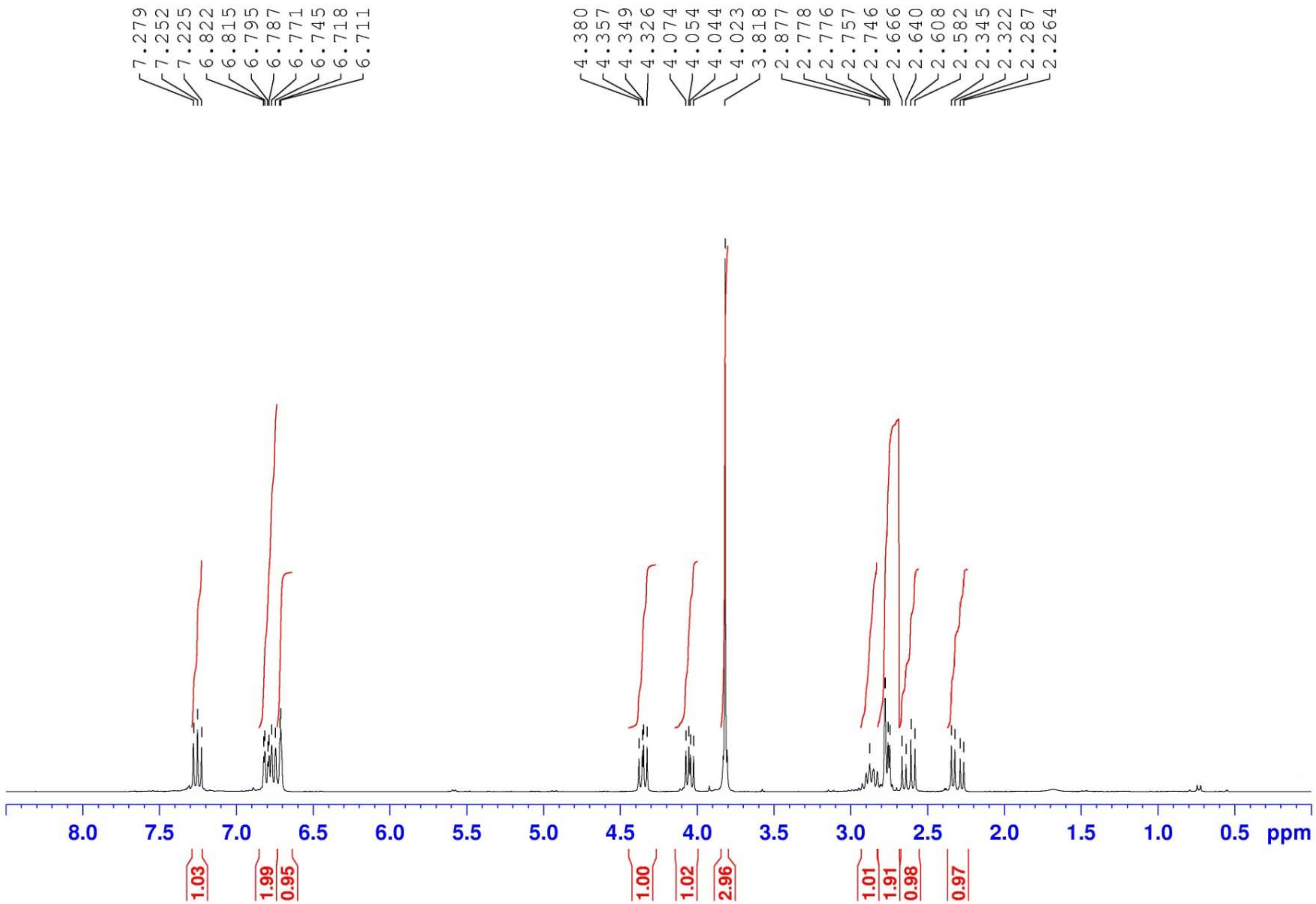


(*R*)-**22b**

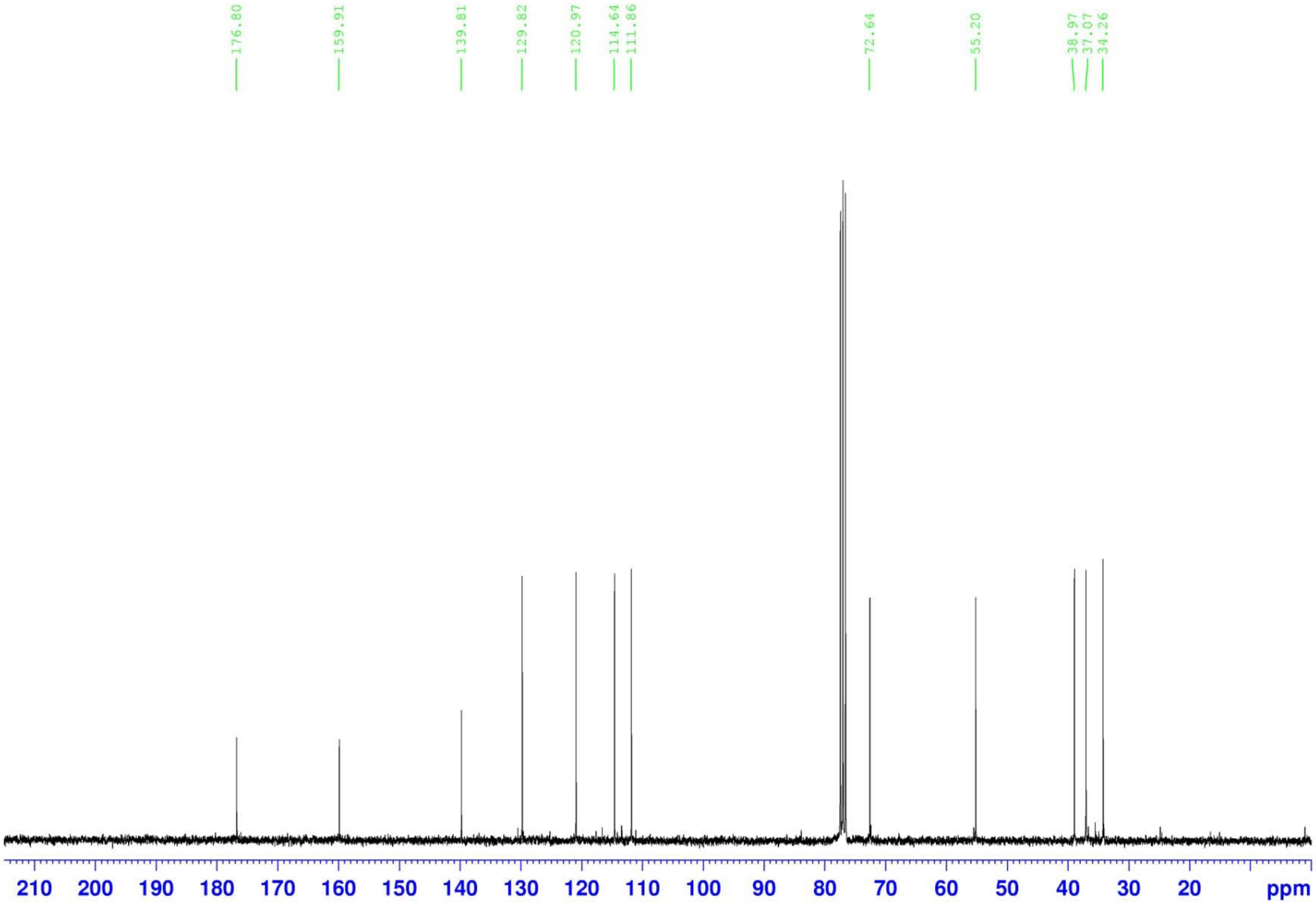
Following the procedure for lactonization via CAHB-oxidation of **21b** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (79%) as a light yellow oil.

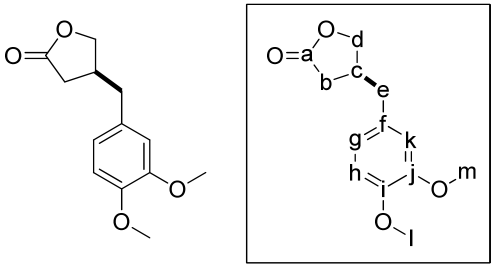
Optical rotation	$[\alpha]_D^{20}$ = +5.5° (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-AD, 90:10 hexanes:isopropanol) showed peaks at 31 minutes (97.5% (R)) and 34 minutes (2.5% (S))
TLC analysis	0.3 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.25 (1H, t, J = 8.1 Hz, h), 6.85–6.80 (1H, m, i), 6.75 (1H, d, J = 7.5 Hz, g), 7.71 (1H, s, k), 4.35 (1H, dd, J_1 = 9.1 Hz, J_2 = 6.9 Hz, d), 4.05 (1H, dd, J_1 = 9.1 Hz, J_2 = 6.0 Hz, d), 3.82 (3H, s, l), 2.95–2.80 (1H, m, c), 2.80–2.75 (2H, m, e), 2.62 (1H, dd, J_1 = 17.4 Hz, J_2 = 7.9 Hz, b), 2.30 (1H, dd, J_1 = 17.4 Hz, J_2 = 6.9 Hz, b).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 176.80 (a), 159.91 (j), 139.81 (f), 129.82 (h), 120.97 (g), 114.64 (k), 111.86 (i), 72.64 (d), 55.20 (l), 38.97 (e), 37.07 (b), 34.26 (c).
IR (neat)	2913, 1774 (C=O stretch), 1601, 1584, 1481, 1454, 1261, 1165 (C-O stretch), 1152 (C-O stretch), 1039, 1013, 909, 784, 727, 698, 647 cm^{-1}.

^{1}H NMR of (*R*)-22b



^{13}C NMR of (*R*)-22b



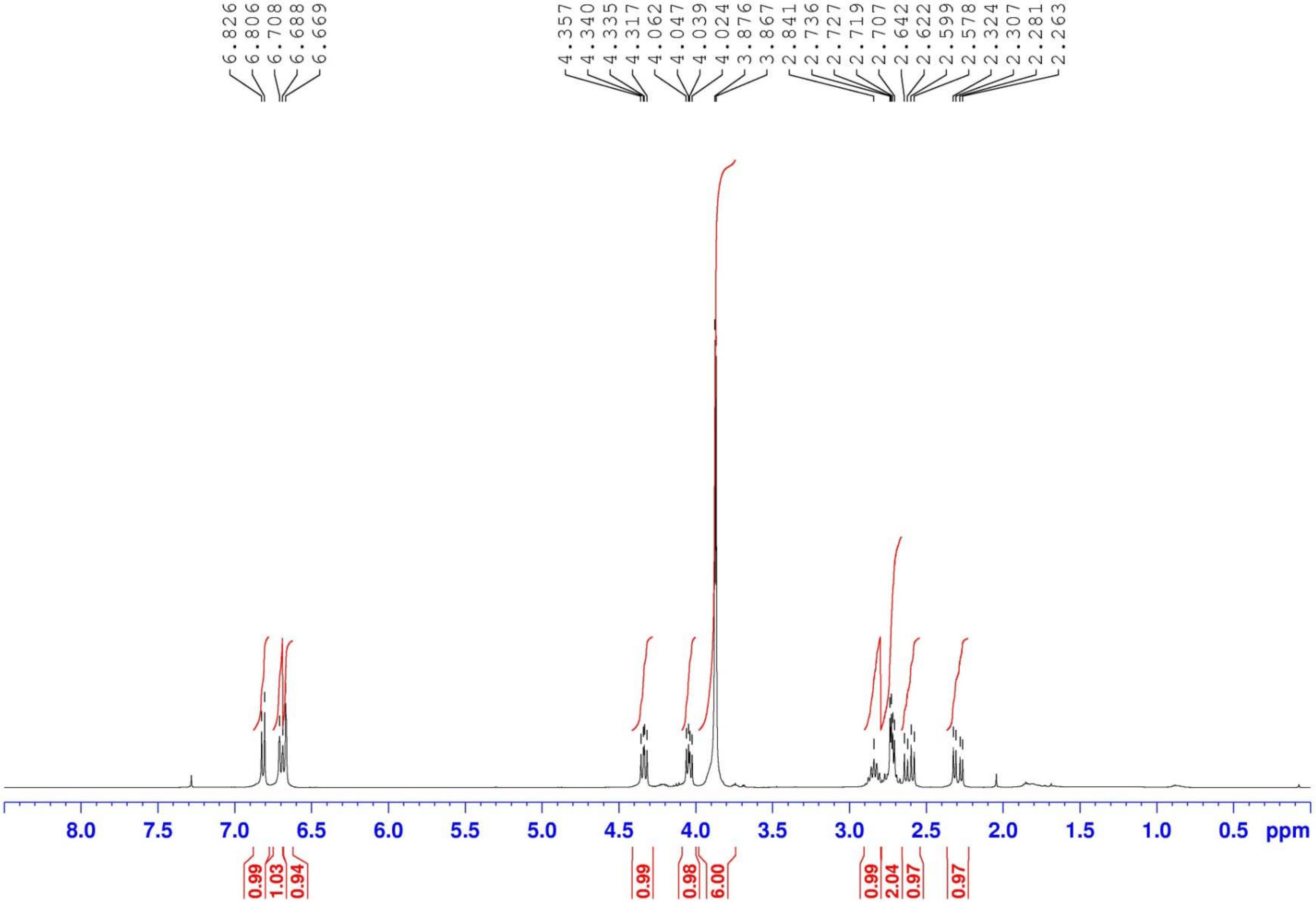


(*S*)-**22c**

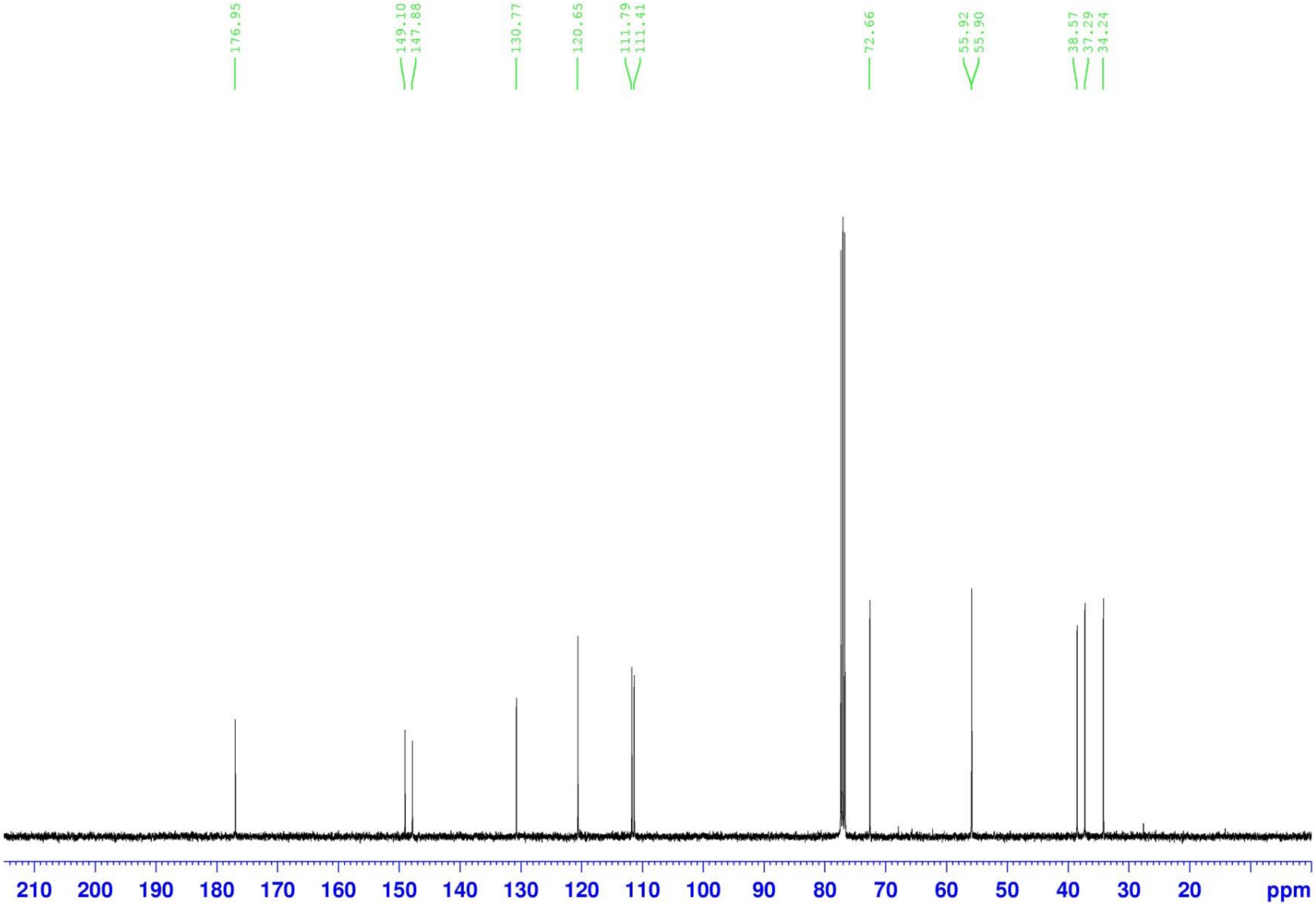
Following the procedure for lactonization via CAHB-oxidation of **21c** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (75%) as a light yellow oil.

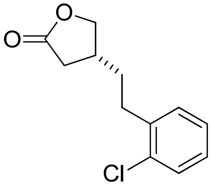
Optical rotation	$[\alpha]_D^{20} = -5.8^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-AD, 90:10 hexanes:isopropanol) shows peaks at 30 minutes (4.0% (R)) and 34 minutes (96.0% (S))
TLC analysis	R_f 0.3 (75:25 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 6.81 (1H, d, J = 8.1 Hz, h), 6.75–6.70 (1H, m, g), 6.67 (1H, s, k), 4.33 (1H, dd, J_1 = 9.0 Hz, J_2 = 7.0 Hz, d), 4.04 (1H, dd, J_1 = 9.0 Hz, J_2 = 6.1 Hz, d), 3.88 (3H, s, m), 3.87 (3H, s, l), 2.90–2.80 (1H, m, c), 2.80–2.65 (2H, m, e), 2.61 (1H, dd, J_1 = 17.5 Hz, J_2 = 8.1 Hz, b), 2.30 (1H, dd, J_1 = 17.5 Hz, J_2 = 6.8 Hz, e).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 176.95 (a), 149.10 (j), 147.88 (i), 130.77 (f), 120.65 (g), 111.79 (k), 111.41 (h), 72.66 (d), 55.92 (l), 55.90 (m), 38.57 (e), 37.29 (b), 34.24 (c).
IR (neat)	2908, 2836, 2253, 1770 (C=O stretch), 1514, 1464, 1418, 1262, 1234, 1158 (C-O stretch), 1139 (C-O stretch, 1014 (C-O stretch), 912, 805, 725, 646 cm^{-1}.

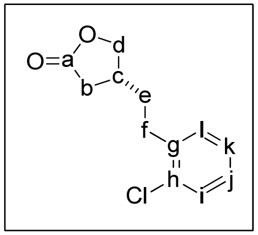
^{1}H NMR of (*S*)-22c



^{13}C NMR of (*S*)-22c



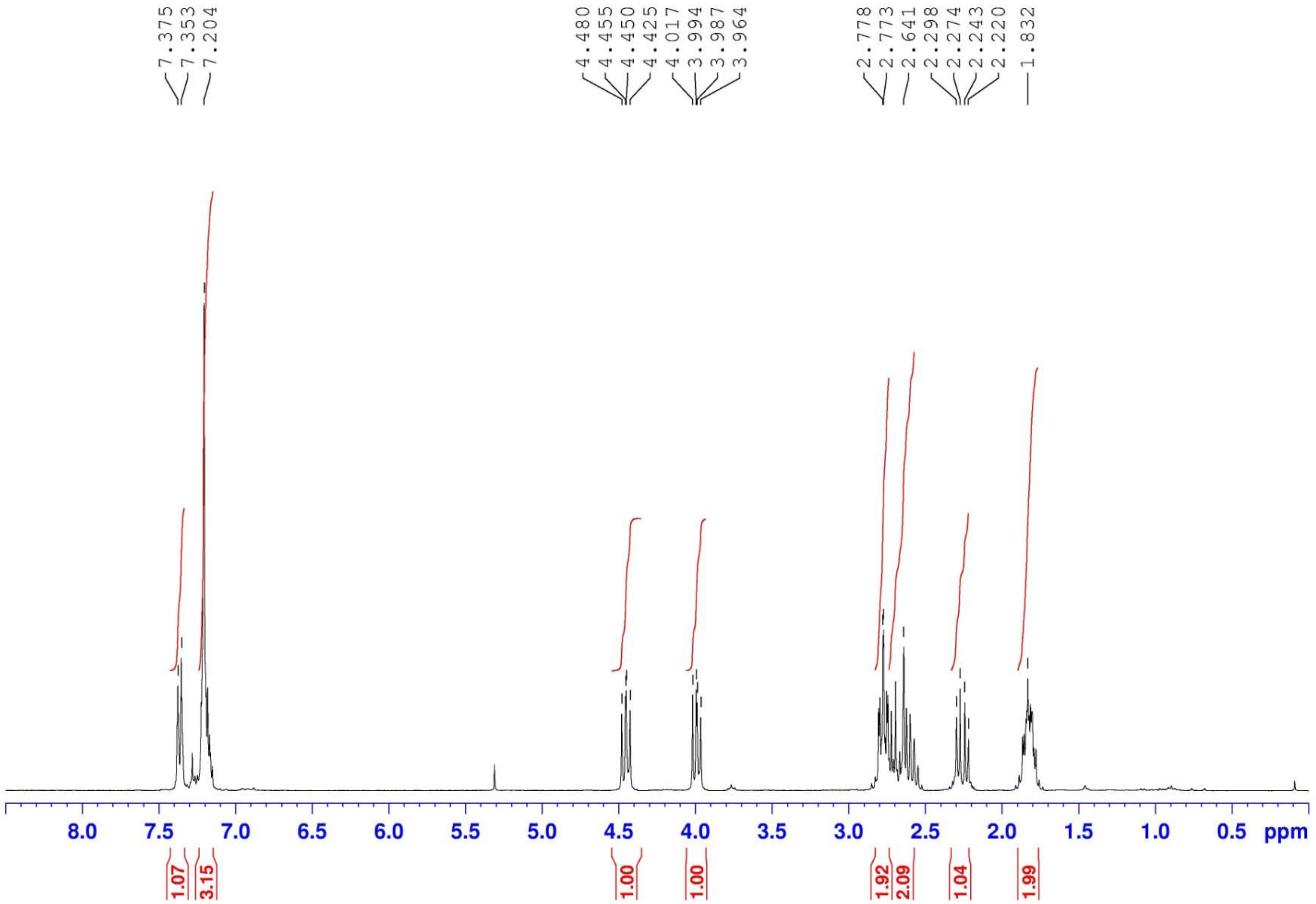




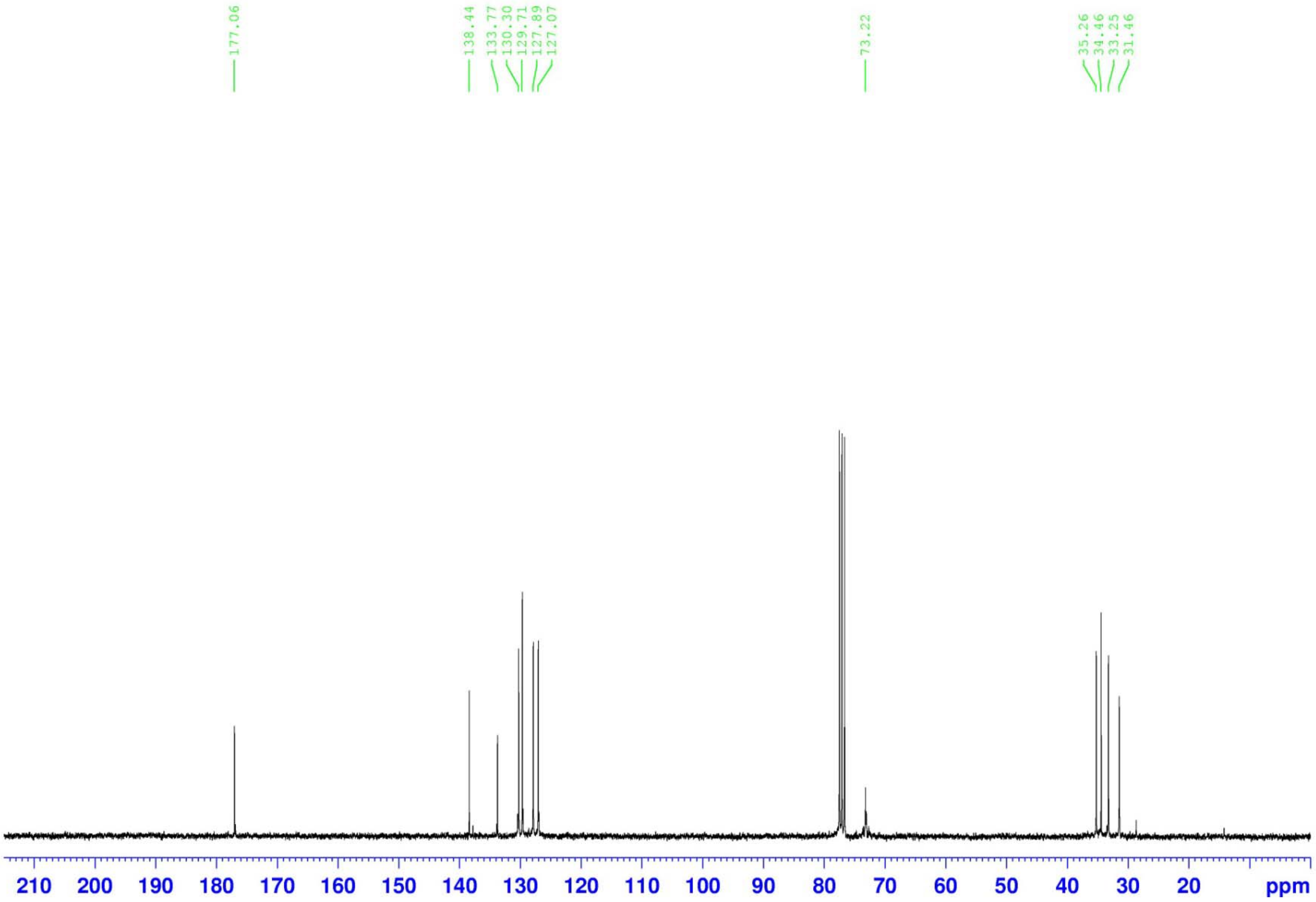
Following the procedure for lactonization via CAHB-oxidation of **12** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (73%) as a light yellow oil.

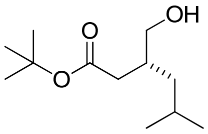
Optical rotation	$[\alpha]_D^{20} = +4.4^o$ (*c* 0.5, $CHCl_3$)
HPLC analysis	(Chiralpak-AD, 90:10 hexanes:isopropanol) shows peaks at 25 minutes (95.0% (R)) and 28 minutes (5.0% (S))
TLC analysis	R_f 0.5 (75:25 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.40 (1H, m, j), 7.25–7.15 (3H, m, i,k,l), 4.45 (1H, dd, J_1 = 9.0 Hz, J_2 = 7.5 Hz, d), 3.99 (1H, dd, J_1 = 9.0 Hz, J_2 = 7.0 Hz, d), 2.85–2.75 (2H, m, f), 2.75–2.50 (2H, m, b), 2.30–2.20 (1H, m, c), 1.90–1.75 (2H, m, e).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 177.06 (a), 138.44 (g), 133.77 (h), 130.30 (i), 129.71 (j), 127.89 (k), 127.07 (l), 73.22 (d), 35.26 (e), 34.46 (b), 33.25 (f), 31.46 (c).
IR (neat)	2923, 1771 (C=O stretch), 1474, 1170 (C-O stretch), 1050, 1020, 991, 908, 840, 752, 727, 680 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{12}H_{13}ClO_2$: 224.0604, found 224.0600 *m/z*.

^{1}H NMR of (*R*)-4-(2-(2-chlorophenyl)ethyl)butyrolactone

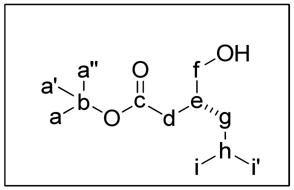


^{13}C NMR of (*R*)-4-(2-(2-chlorophenyl)ethyl)butyrolactone





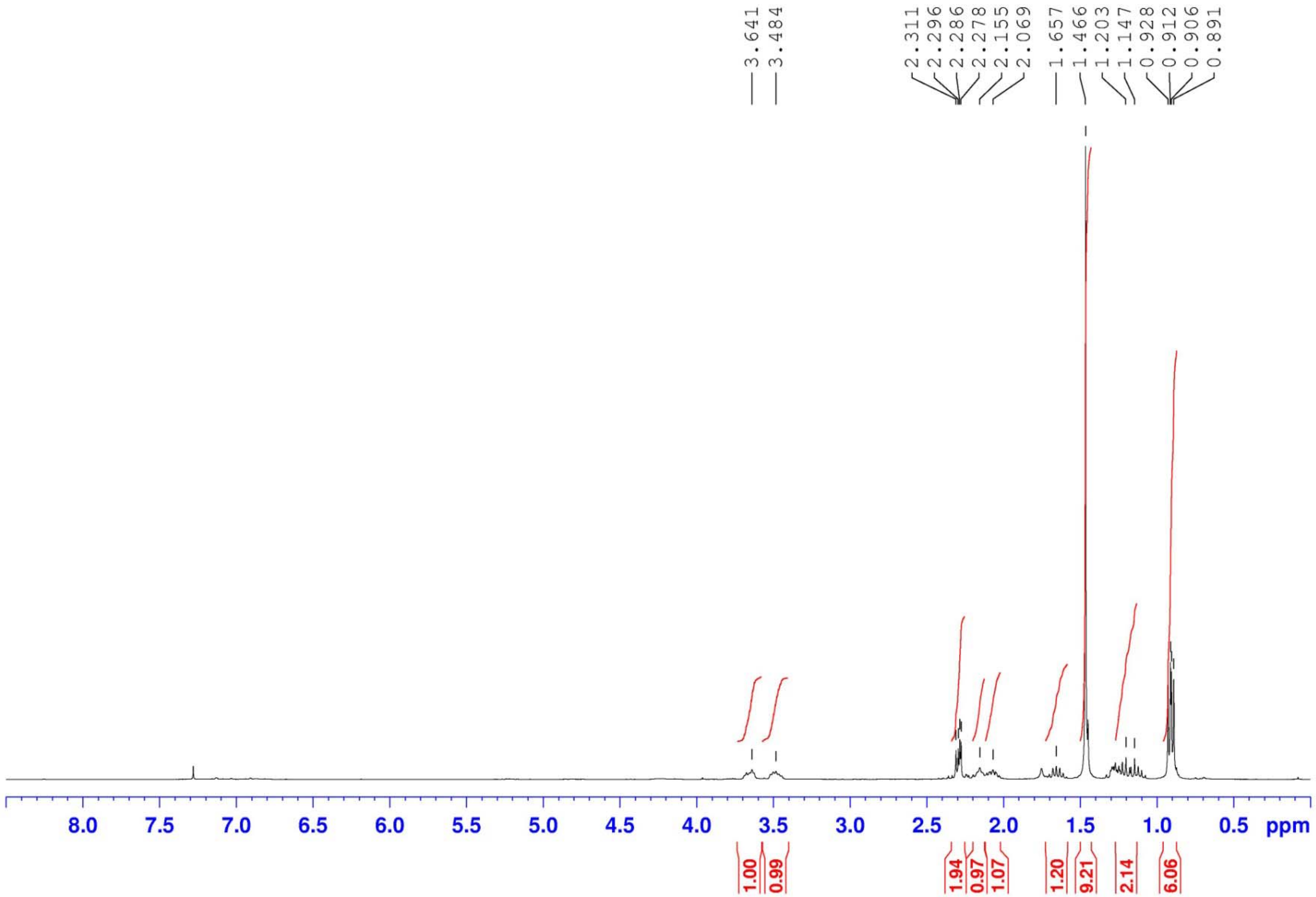
(*R*)-**19**



Following the general procedure for CAHB-oxidation of **7i** with $NaBO_3$ affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (77%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = +2.3^{\circ}$ (*c* 0.5, $CHCl_3$)
TLC analysis	0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 3.70–3.60 (1H, m, f), 3.60–3.40 (1H, m, f), 2.28 (2H, dd, J_1 = 4.4 Hz, J_2 = 2.5 Hz, d), 2.16 (1H, br s, OH), 2.10–2.00 (1H, m, e), 1.70–1.60 (1H, m, h), 1.47 (9H, s, a,a',a''), 1.30–1.20 (2H, m, g), 0.92 (3H, d, *J* = 4.7 Hz, i), 0.90 (3H, d, *J* = 4.7 Hz, i').
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.32 (c), 80.62 (b), 66.07 (f), 40.43 (g), 38.46 (d), 35.71 (e), 28.08 (a,a',a''), 25.19 (h), 22.80 (i), 22.67 (i').
IR (neat)	3404 (O-H stretch), 2956, 2930, 1728 (C=O stretch), 1468, 1367, 1311 (C-O stretch), 1254, 1154, 1062, 959, 733 cm^{-1}.

^{1}H NMR of (*R*)-19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1.466
1.203
1.147
0.928
0.912
0.906
0.891
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
ppm
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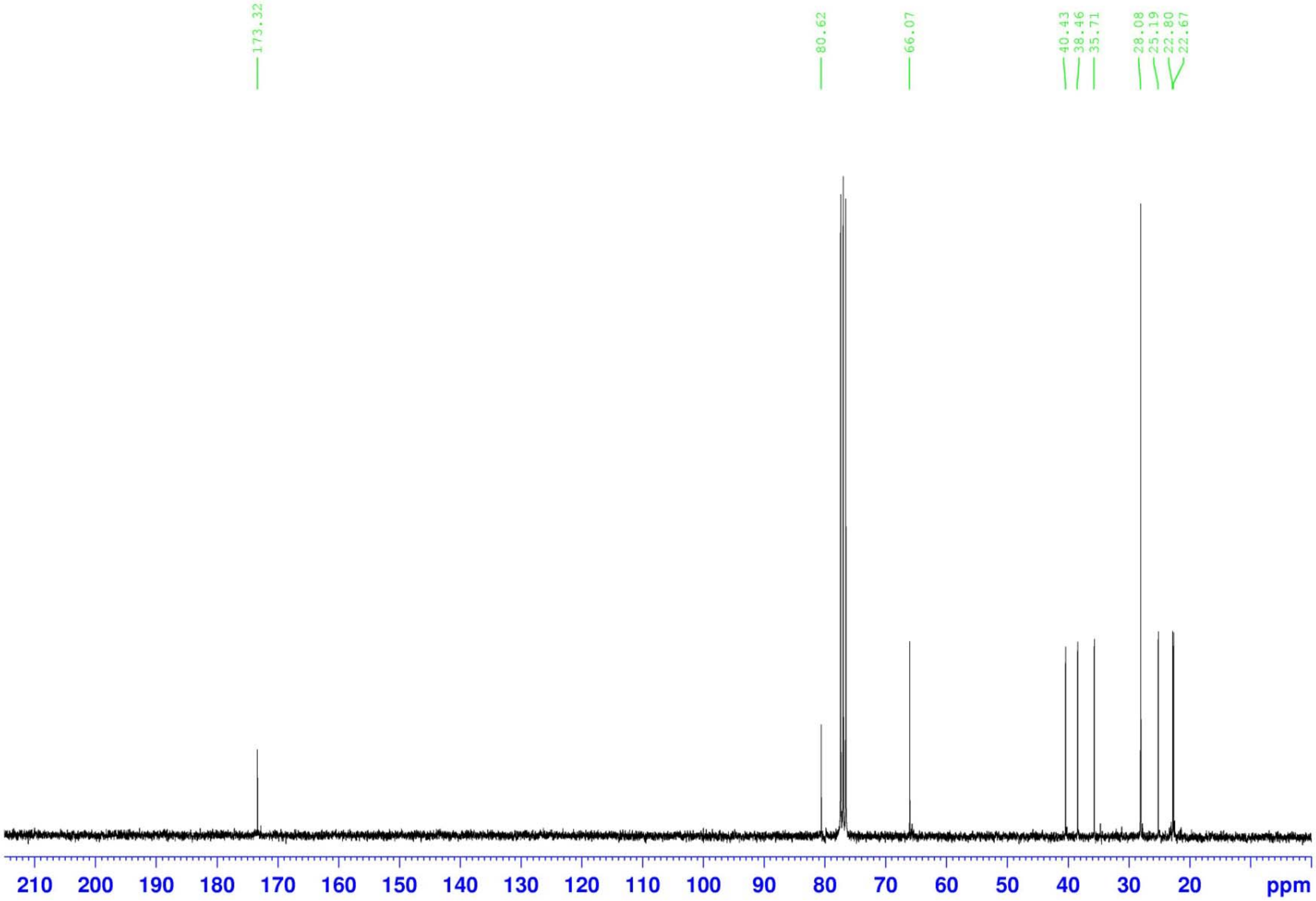
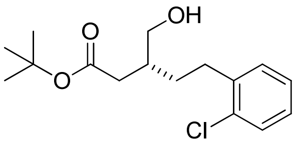
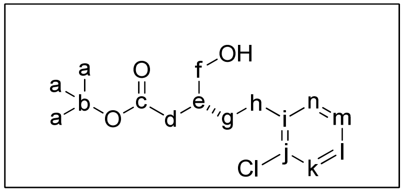
0.99
1.94
0.97
1.07
1.20
9.21
2.14
6.06

^{13}C NMR of (*R*)-19





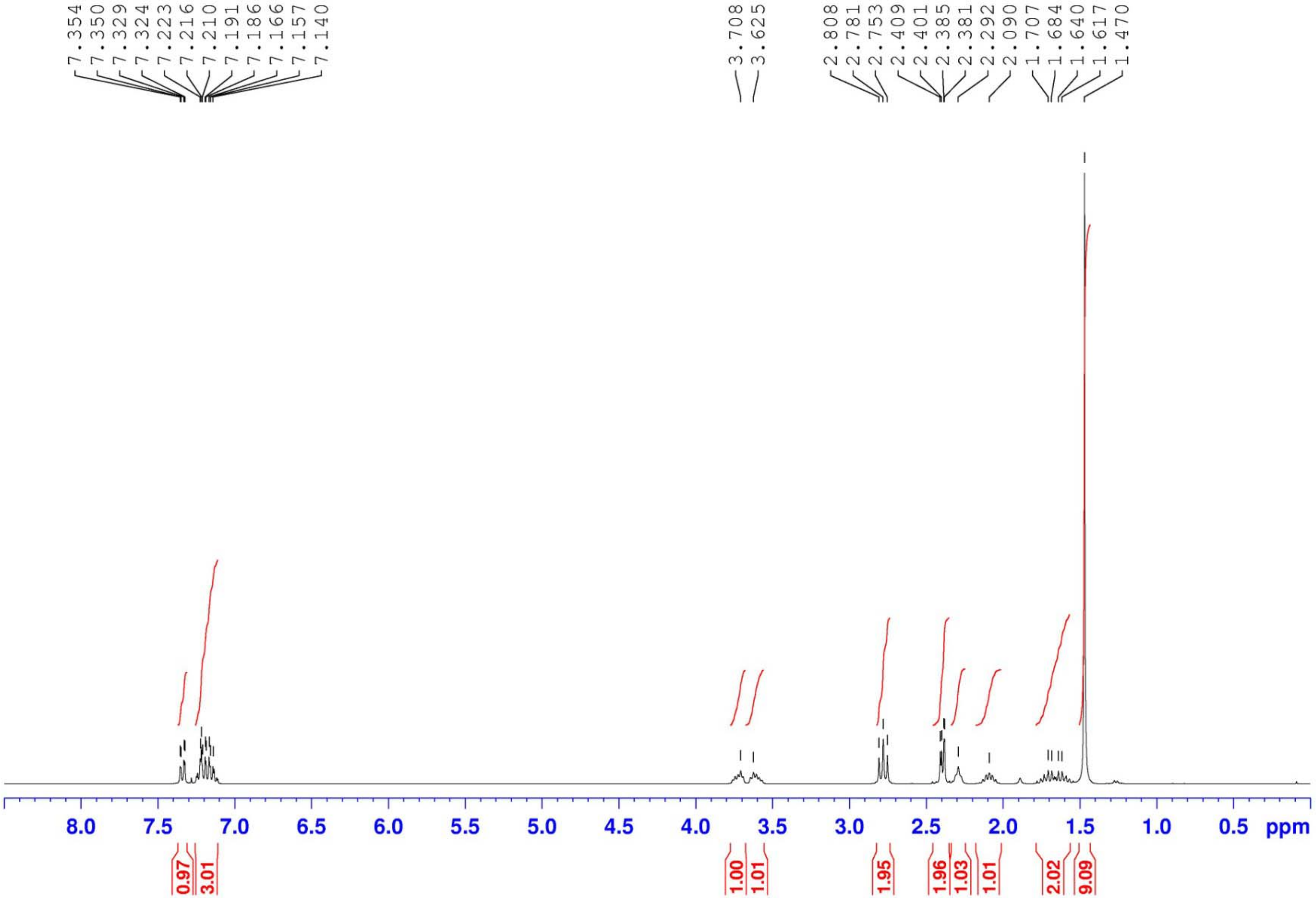
(*R*)-**15**



Following the general procedure for CAHB-oxidation of **12** with $NaBO_3$ affords, after flash chromatography on silica gel (80–70:20–30 hexanes:dichloromethane), the title compound (73%) as a light yellow oil.

Optical rotation	$[\alpha]_D^{20} = +4.5^{\circ}$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (50:50 hexanes:dichloromethane)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.40–7.30 (1H, m, k), 7.25–7.10 (3H, m, l,m,n), 3.80–3.65 (1H, m, f), 3.65–3.55 (1H, m, f), 2.78 (2H, t, *J* = 8.2 Hz, h), 2.45–2.35 (2H, m, d), 2.29 (1H, br s, OH), 2.15–2.00 (1H, m, e), 1.80–1.60 (2H, m, g), 1.47 (9H, s, a,a',a").
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.04 (c), 139.69 (i), 133.79 (j), 130.27 (k), 129.49 (l), 127.38 (m), 126.85 (n), 80.76 (b), 65.49 (f), 38.03 (d), 37.78 (e), 31.30 (h), 31.03 (g), 28.09 (a,a',a").
IR (neat)	3437 (O-H stretch), 2930, 1720 (C=O stretch), 1474, 1456, 1367 (C- O stretch), 1148, 1051, 909, 751, 732 cm^{-1}.
HRMS (EI)	Calcd. for $C_{16}H_{23}ClNaO_3$ (M+Na): 321.1233, found 321.1237 *m/z*.

^{1}H NMR of (*R*)-15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7.191
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7.157
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3.625
2.808
2.781
2.753
2.409
2.401
2.385
2.381
2.292
2.090
1.707
1.684
1.640
1.617
1.470
8.0
7.5
7.0
6.5
6.0
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4.5
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3.5
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2.0
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1.0
0.5
ppm
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1.01
1.95
1.96
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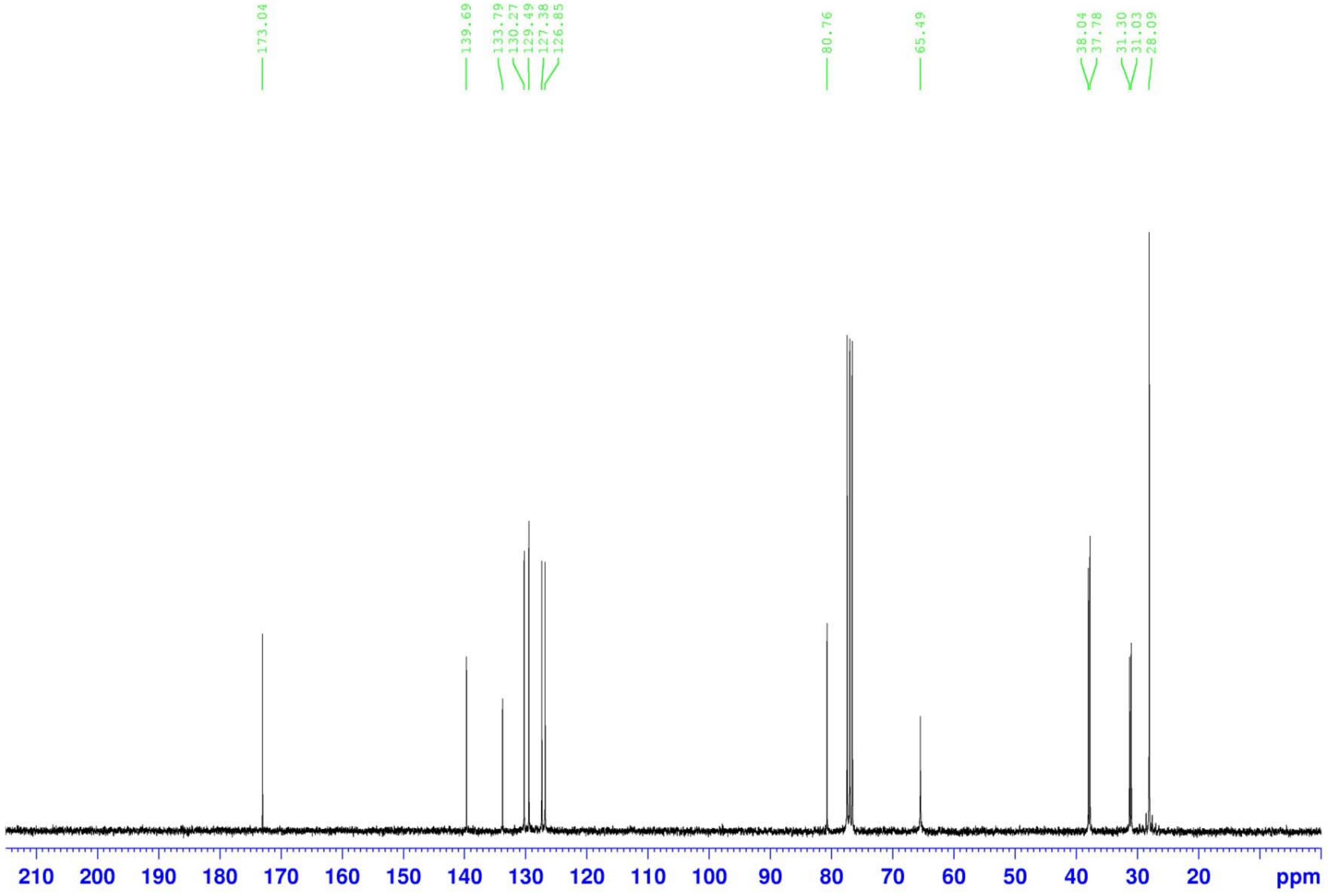
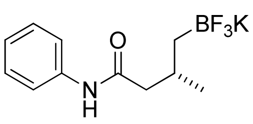
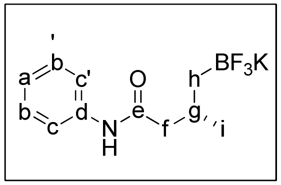
1.01
2.02
9.09

^{13}C NMR of (*R*)-15





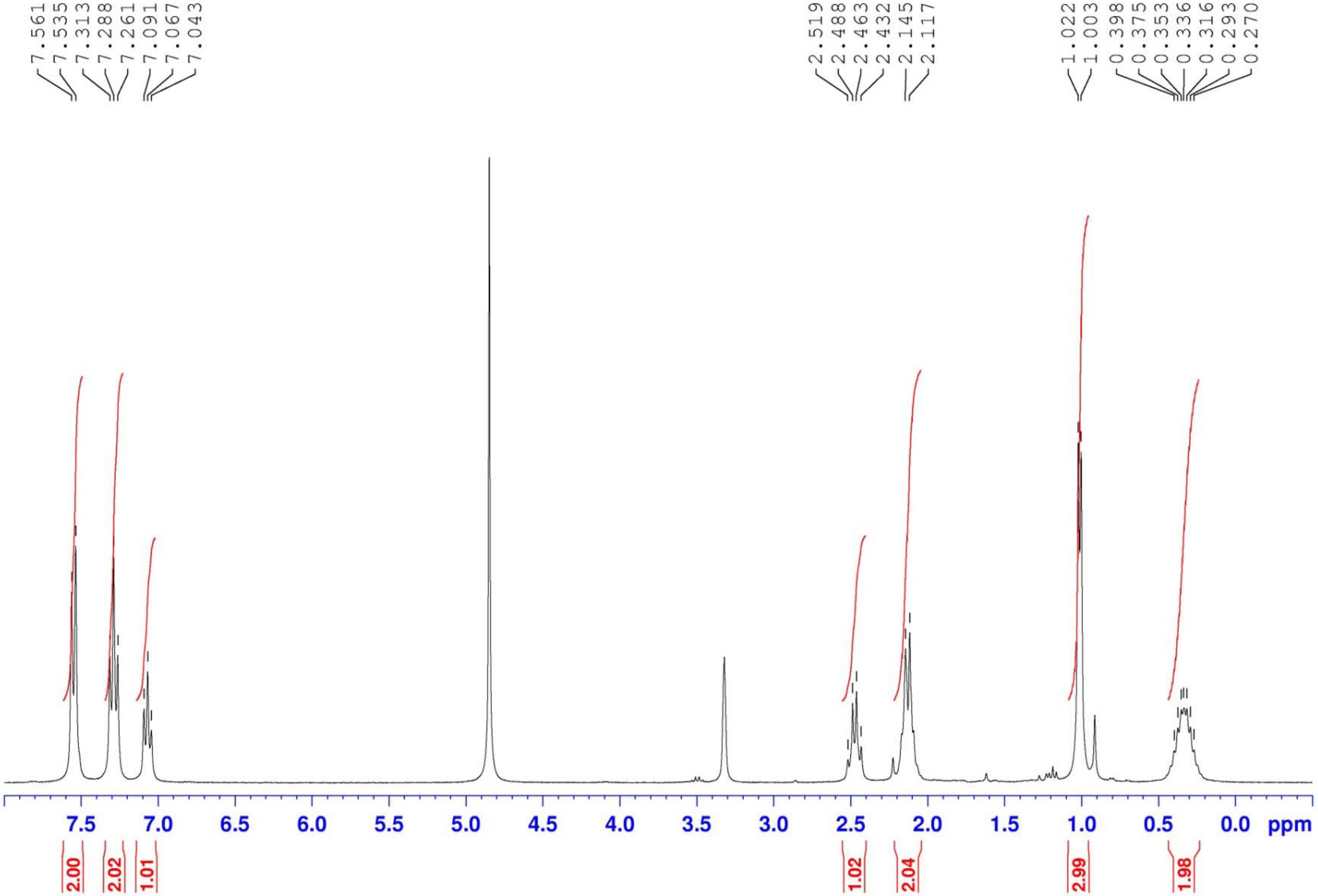
(*S*)-**10a**



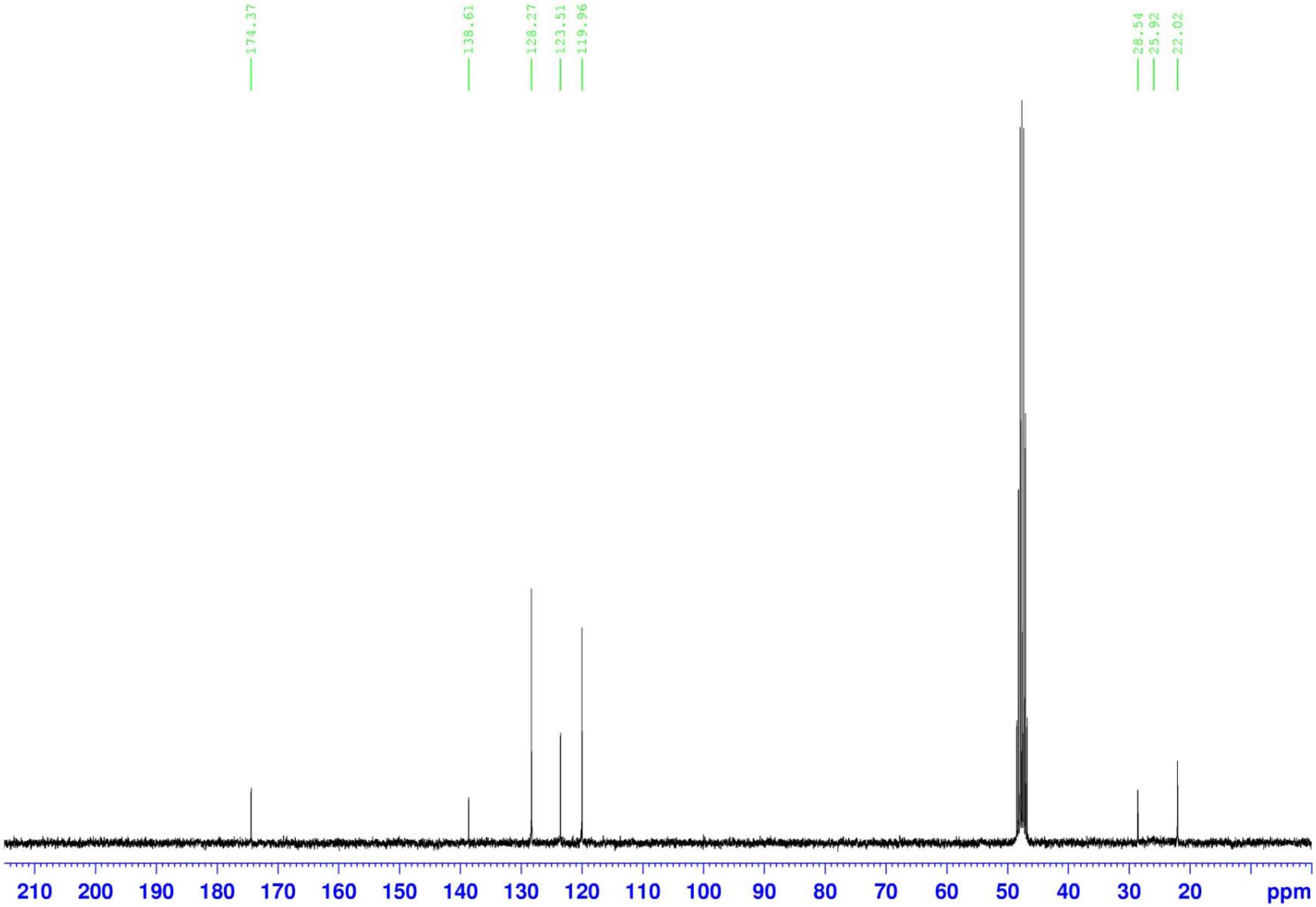
Following the general procedure for the preparation of trifluoroborate salts with (*S*)-**8a** affords the title compound (65%) as a white solid.

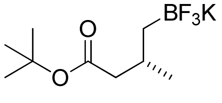
m.p.	176.5–177.5 ºC
Optical rotation	$[\alpha]_D^{20}$ = +5.7º (*c* 0.5, MeOH)
^{1}H NMR (300 MHz, MeOD)	δ 7.55 (2H, d, *J* = 7.8 Hz, c,c'),7.29 (2H, t, *J* = 7.6 Hz, b,b'),7.07 (1H, t, *J* = 7.3 Hz, a), 2.55–2.40 (1H, m, f), 2.20–2.00 (2H, m, f,g), 1.01 (3H, d, *J* = 5.7 Hz, i), 0.45–0.20 (2H, m, h).
^{13}C NMR (75 MHz, MeOD)	δ 174.37 (e), 138.61 (d), 128.27 (b,b'), 123.51 (a), 119.96 (c,c'), 28.54 (g),25.92 (h), and 22.02 (i)
IR (neat)	3129 (N-H bend), 2975, 2898, 1728 (C=O stretch), 1545 (N-H bend), 1390, 1369, 1267 (C-N stretch), 1152, 1086, 960, 777 cm^{-1}.
HRMS (CI)	Calcd. for $C_{11}H_{15}NO$ (M+H-BF_3K): 177.1153, found 177.1148 *m/z*.

^{1}H NMR of (*S*)-10a

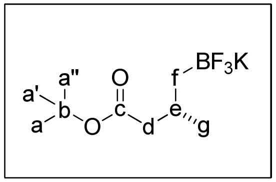


^{13}C NMR of (*S*)-10a





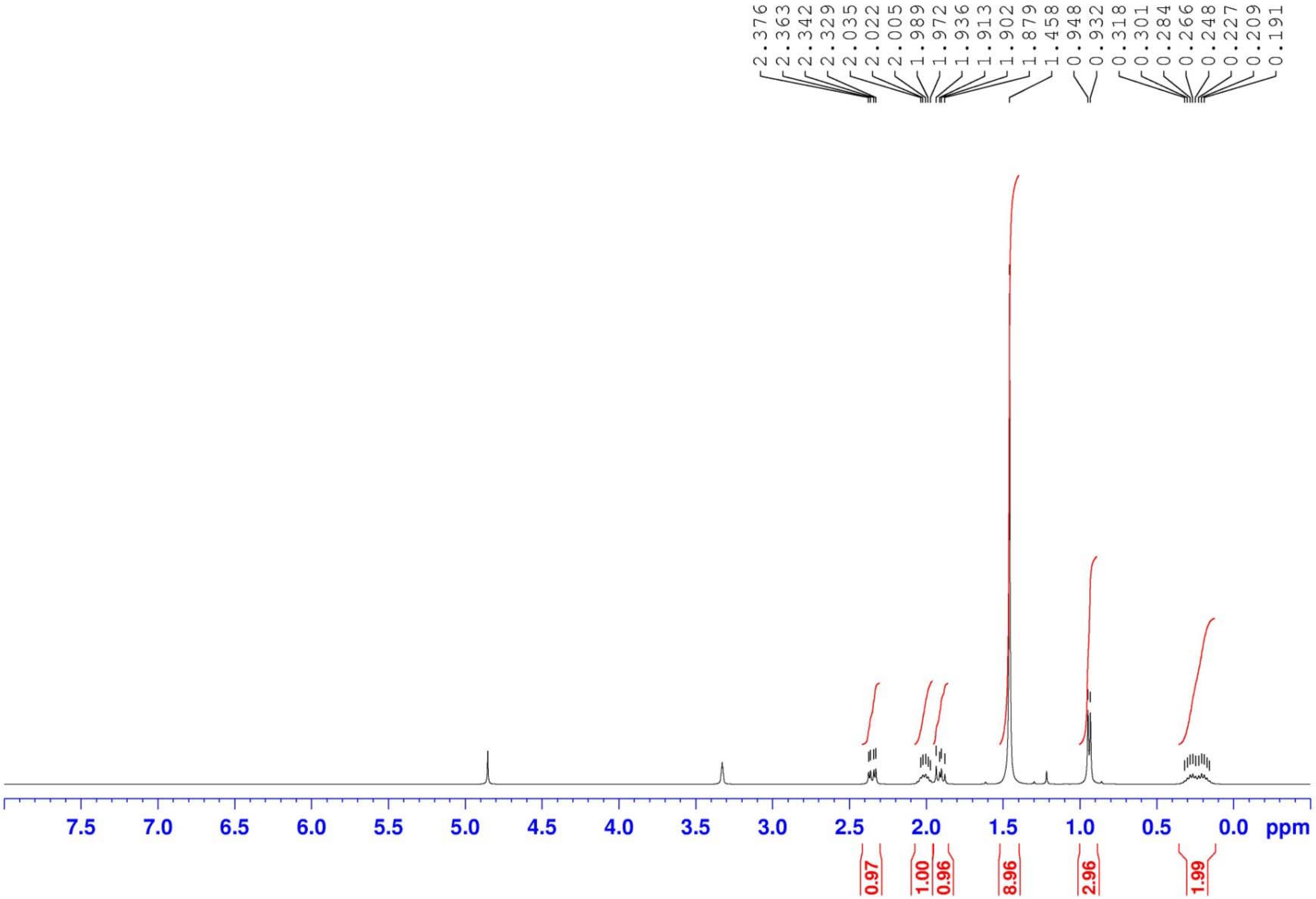
(*S*)-**10g**



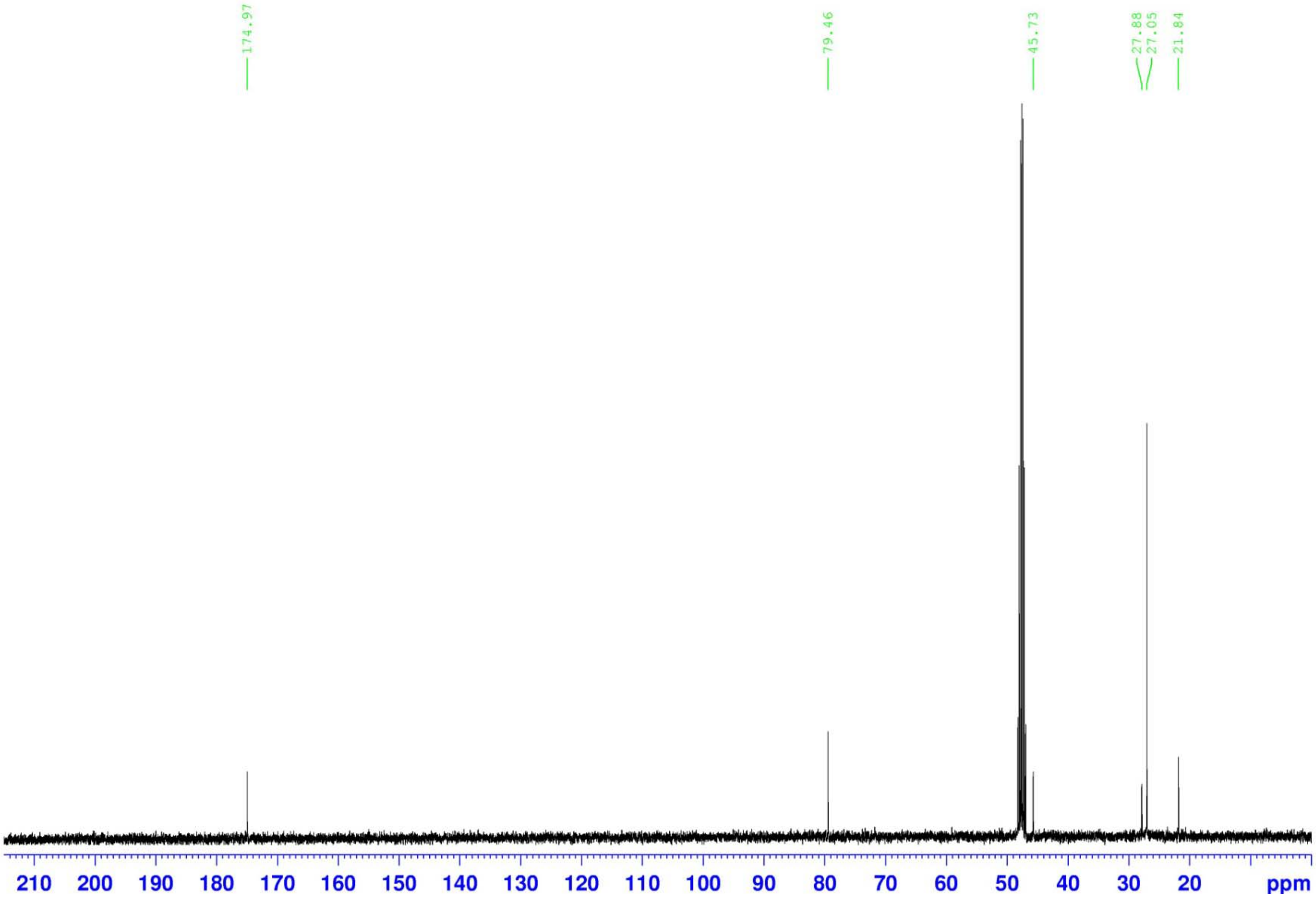
Following the general procedure for the preparation of trifluoroborate salts with (*S*)-**8g** affords the title compound (62%) as a white solid.

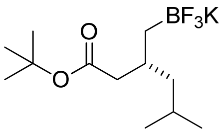
m.p.	176.0–178.0 ºC
Optical rotation	$[\alpha]_D^{20} = +6.4^{o}$ (*c* 0.5, MeOH)
^{1}H NMR (400 MHz, MeOD)	δ 2.35 (1H, dd, *J* = 13.7 Hz, 5.2 Hz, d), 2.05–1.95 (1H, m, e), 1.91 (1H, dd, *J* = 13.7 Hz, 8.9 Hz, d), 1.46 (9H, s, a,a',a''), 0.94 (3H, d, *J* = 6.5 Hz, g), 0.35–0.15 (2H, m, f).
^{13}C NMR (100 MHz, MeOD)	δ 174.97 (c), 79.46 (b), 45.73 (d), 27.88 (e), 27.05 (a,a',a''), 21.84 (g).
IR (neat)	2983, 1727 (C=O stretch), 1457, 1368 (C-O stretch), 1274, 1152, 1099, 956, 918, 842, 769 cm^{-1}.
HRMS (CI)	Calcd. for $C_9H_{17}O_2$ (M-BF$_3$K): 147.1214, found 147.1215 *m/z*.

^{1}H NMR of (*S*)-10g

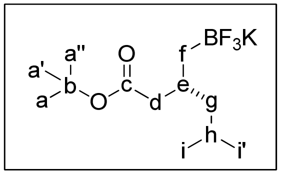


^{13}C NMR of (*S*)-10g





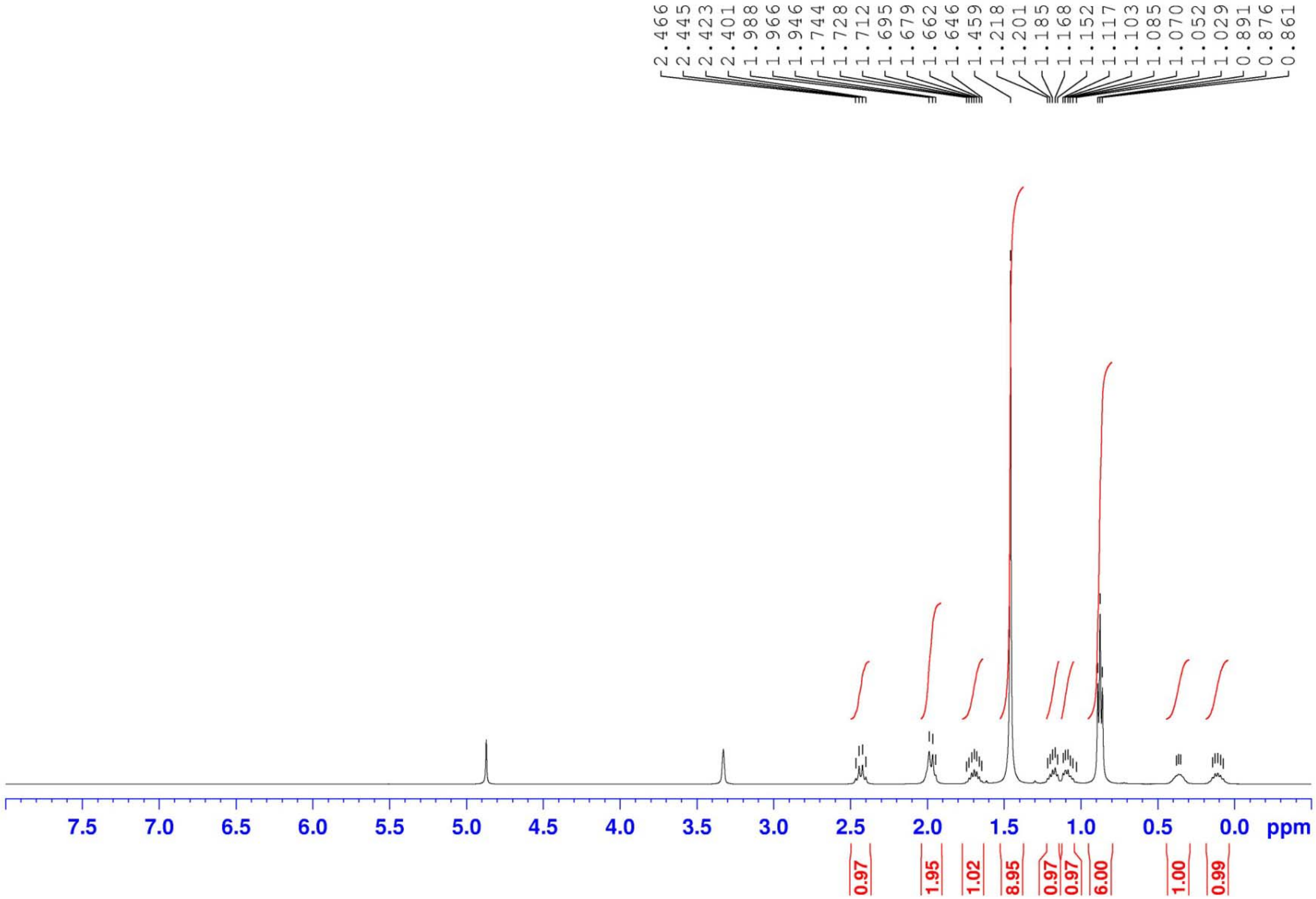
(*R*)-**10i**



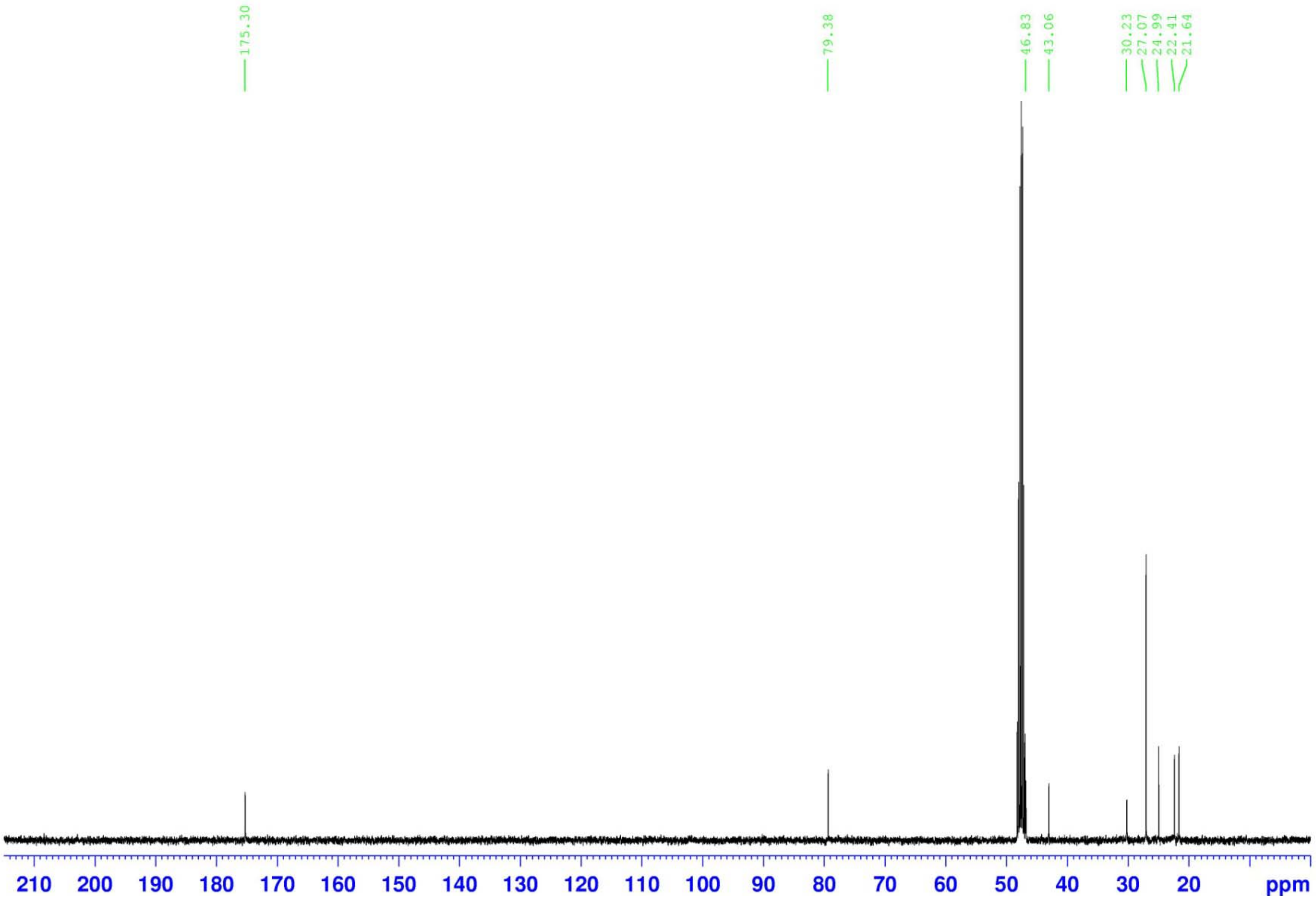
Following the general procedure for the preparation of trifluoroborate salts with (*R*)-**8i** affords the title compound (75%) as a white solid.

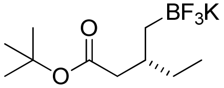
m.p.	179.0–179.5 ºC
Optical rotation	$[\alpha]_D^{20}$ = +9.7º (*c* 0.5, MeOH)
^{1}H NMR (400 MHz, MeOD)	δ 2.50–2.40 (1H, m, d), 2.05–1.95 (2H, m, d,e), 1.75–1.65 (1H, m, h), 1.46 (9H, s, a,a',a''), 1.25–1.15 (1H, m, g), 1.15–1.00 (1H, m, g), 0.88 (6H, t, *J* = 6.1 Hz, i,i'), 0.40–0.35 (1H, m, f), 0.15–0.05 (1H, m, f).
^{13}C NMR (100 MHz, MeOD)	δ 175.30 (c), 79.38 (b), 46.83 (g), 43.06 (d), 30.23 (e), 27.07 (a,a',a''), 24.99 (h), 22.41 and 21.64 (i,i').
IR (neat)	2953, 2906, 1728 (C=O stretch), 1392, 1367 (C-O stretch), 1256, 1125, 1069, 974, 908, 759 cm^{-1}.
HRMS (CI)	Calcd. for $C_{12}H_{23}O_2$ (M-BF_3K): 199.1698, found 199.1510 *m/z*.

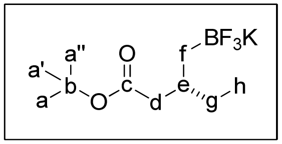
^{1}H NMR of (*R*)-10i



^{13}C NMR of (*R*)-10i





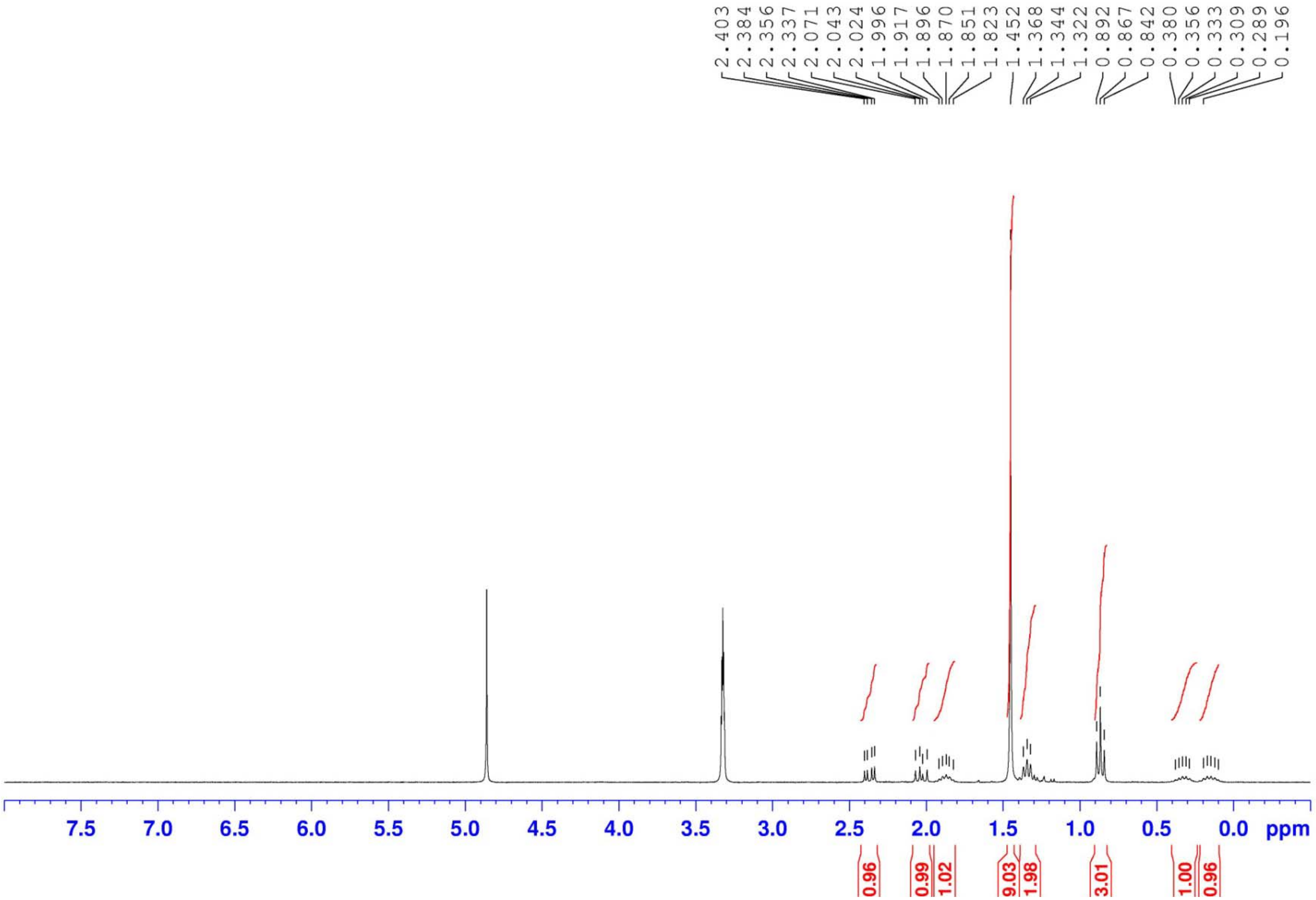


(*R*)-**10h**

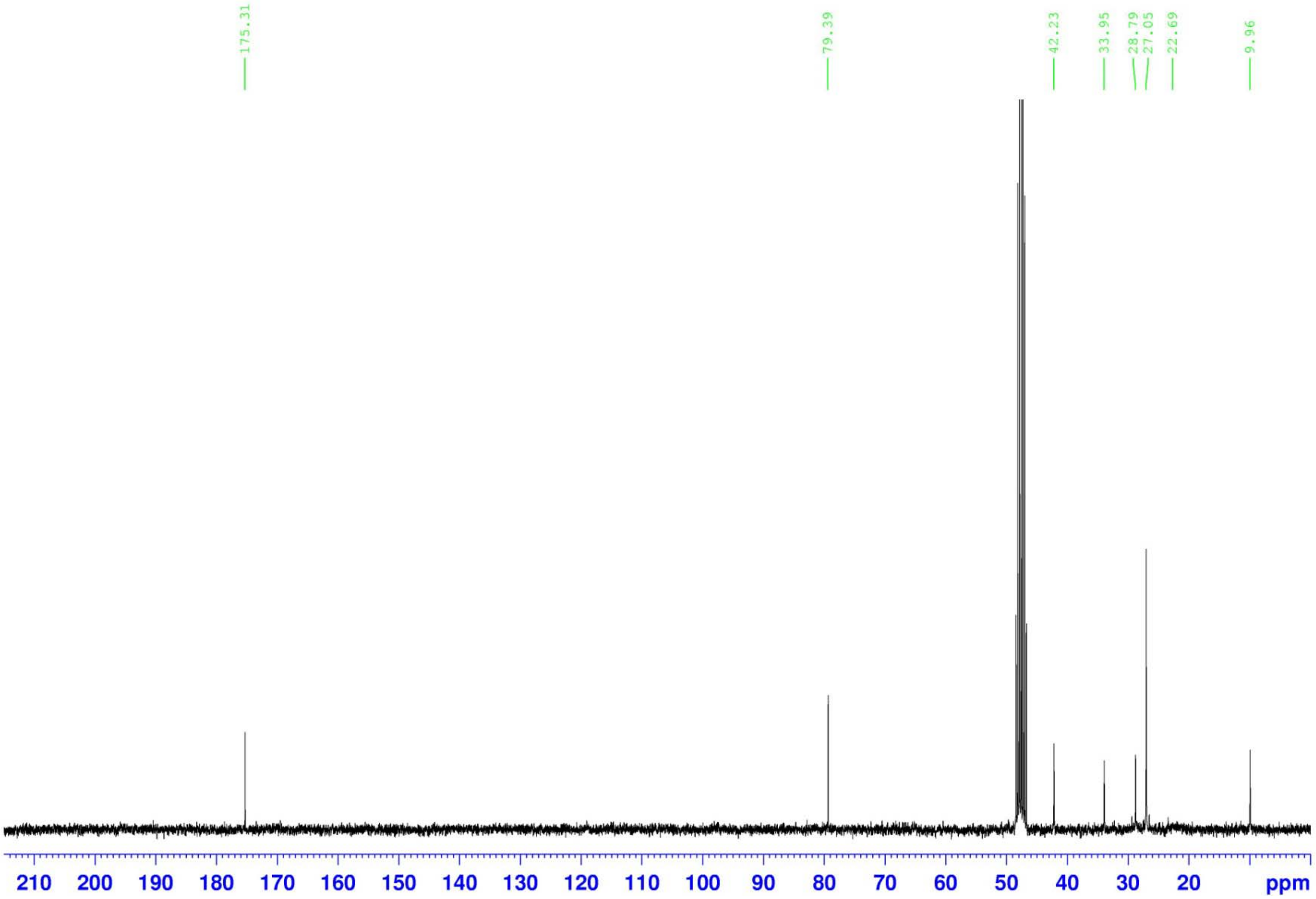
Following the general procedure for the preparation of trifluoroborate salts with (*R*)-**8h** affords the title compound (62%) as a white solid.

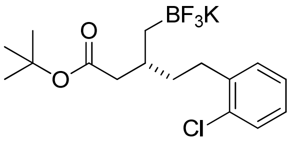
m.p.	174.5–176.0 ºC
Optical rotation	$[\alpha]_D^{20} = +6.3^{\circ}$ (*c* 0.5, MeOH)
^{1}H NMR (300 MHz, MeOD)	δ 2.37 (1H, dd, *J* = 14.2 Hz, 5.6 Hz, d), 2.03 (1H, dd, *J* = 14.2 Hz, 8.6 Hz, d), 1.95–1.80 (1H, m, e), 1.45 (9H, s, a,a’,a’’), 1.34 (2H, t, *J* = 6.9 Hz, g), 0.87 (3H, t, *J* = 7.4 Hz, h), 0.40–0.25 (1H, m, f), 0.20–0.10 (1H, m, f).
^{13}C NMR (75 MHz, MeOD)	δ 175.31 (c), 79.39 (b), 42.23 (d), 33.95 (e), 28.79 (g), 27.05 (a,a’,a’’), 22.69 (f), 9.96 (h).
IR (neat)	2983, 1728 (C=O stretch), 1499, 1329 (C-O stretch), 1080, 1029, 966, 915, 751 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{10}H_{19}O_2$ (M-BF_3K): 171.1386, found 171.1185 *m/z*.

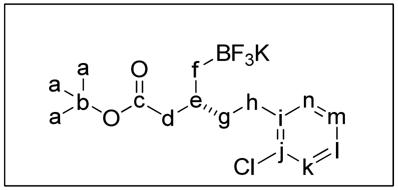
^{1}H NMR of (*R*)-10h



^{13}C NMR of (*R*)-10h



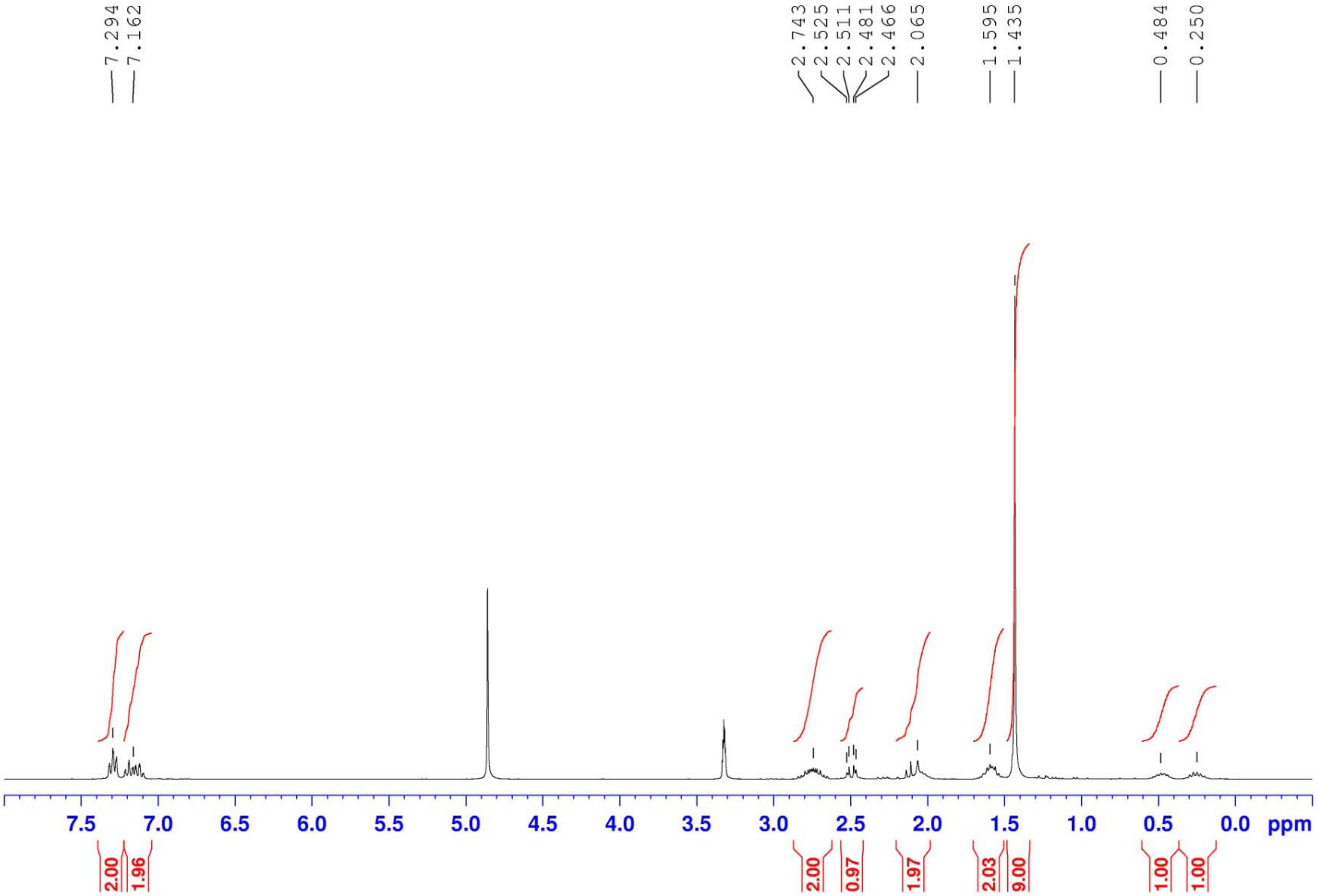




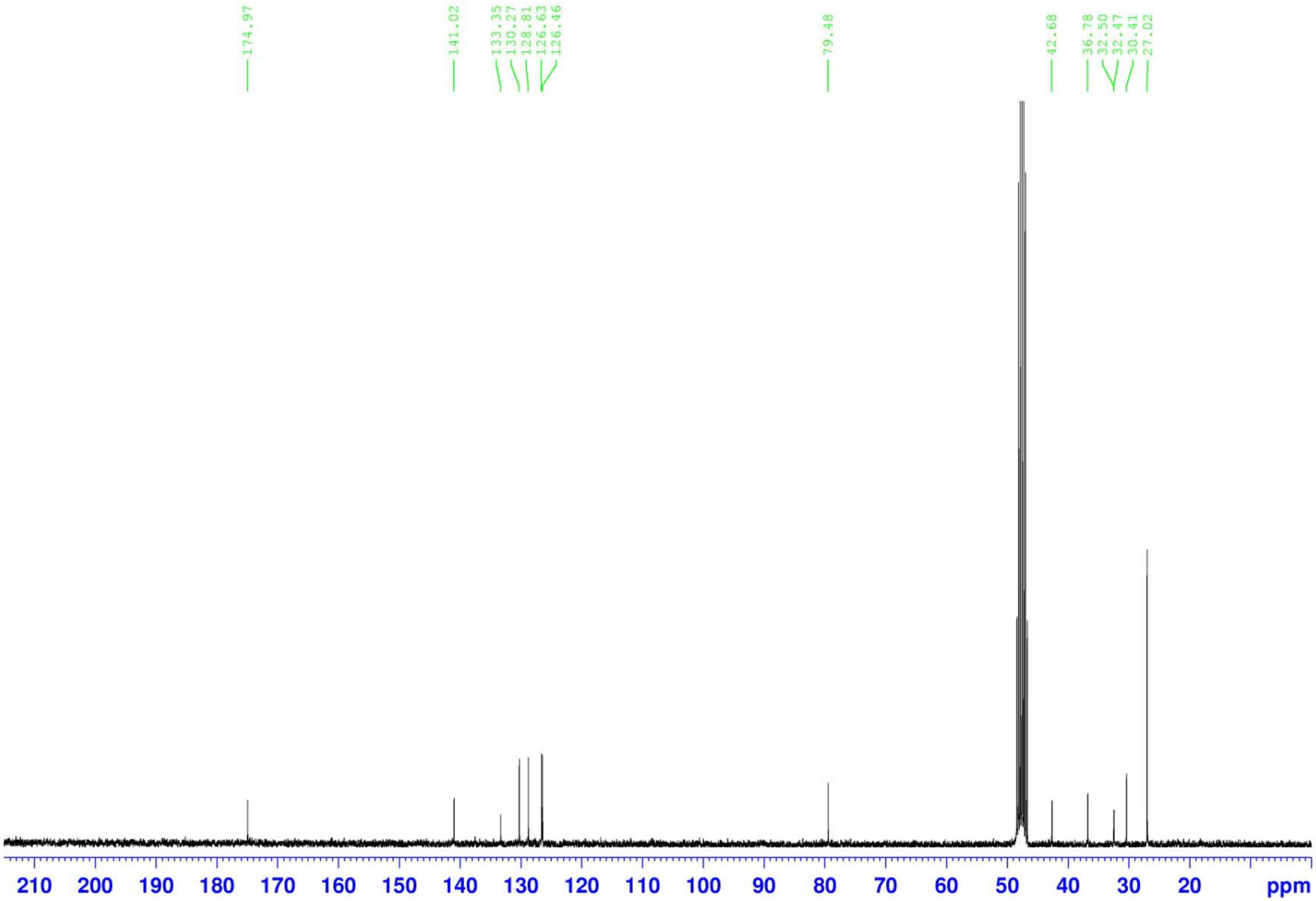
Following the general procedure for the preparation of trifluoroborate salts with (*R*)-**13** affords the title compound (76%) as a white solid.

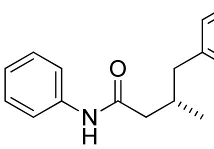
m.p.	174.6–175.6 °C
Optical rotation	$[\alpha]_D^{20}$ = +6.9° (*c* 0.5, MeOH)
^{1}H NMR (300 MHz, MeOD)	δ 7.35–7.25 (2H, m, l,m), 7.25–7.05 (2H, m, j,k), 2.85–2.60 (2H, m, h), 2.50 (1H, dd, *J* = 13.4 Hz, 4,3 Hz, d), 2.20–1.95 (2H, m, d,e), 1.70–1.50 (2H, m, g), 1.43 (9H, s, a,a’,a’’), 0.55–0.40 (1H, m, f), 0.35–0.15 (1H, m, f).
^{13}C NMR (75 MHz, MeOD)	δ 174.97 (c), 141.01 (i), 133.35 (n), 130.27 (m), 128.81 (l), 126.63 (j), 126.46 (k), 79.48 (b), 42.68 (d), 36.78 (g), 32.50 and 32.47 (e), 30.41 (h), 27.02 (a,a’,a’’).
IR (neat)	2978, 2922, 1703 (C=O stretch), 1474, 1391, 1310 (C-O stretch), 1260, 1030, 967, 953, 841, 777 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{16}H_{22}ClO_2$ (M-BF$_3$K): 281.1308, found 281.1312 *m/z*.

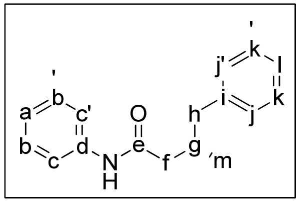
^{1}H NMR of (*R*)-5-(2-chlorophenyl)-3-(trifluoroboratomethyl)pentanoic acid *tert*-butyl ester potassium salt



^{13}C NMR of (*R*)-5-(2-chlorophenyl)-3-(trifluoroboratomethyl)pentanoic acid *tert*-butyl ester potassium salt



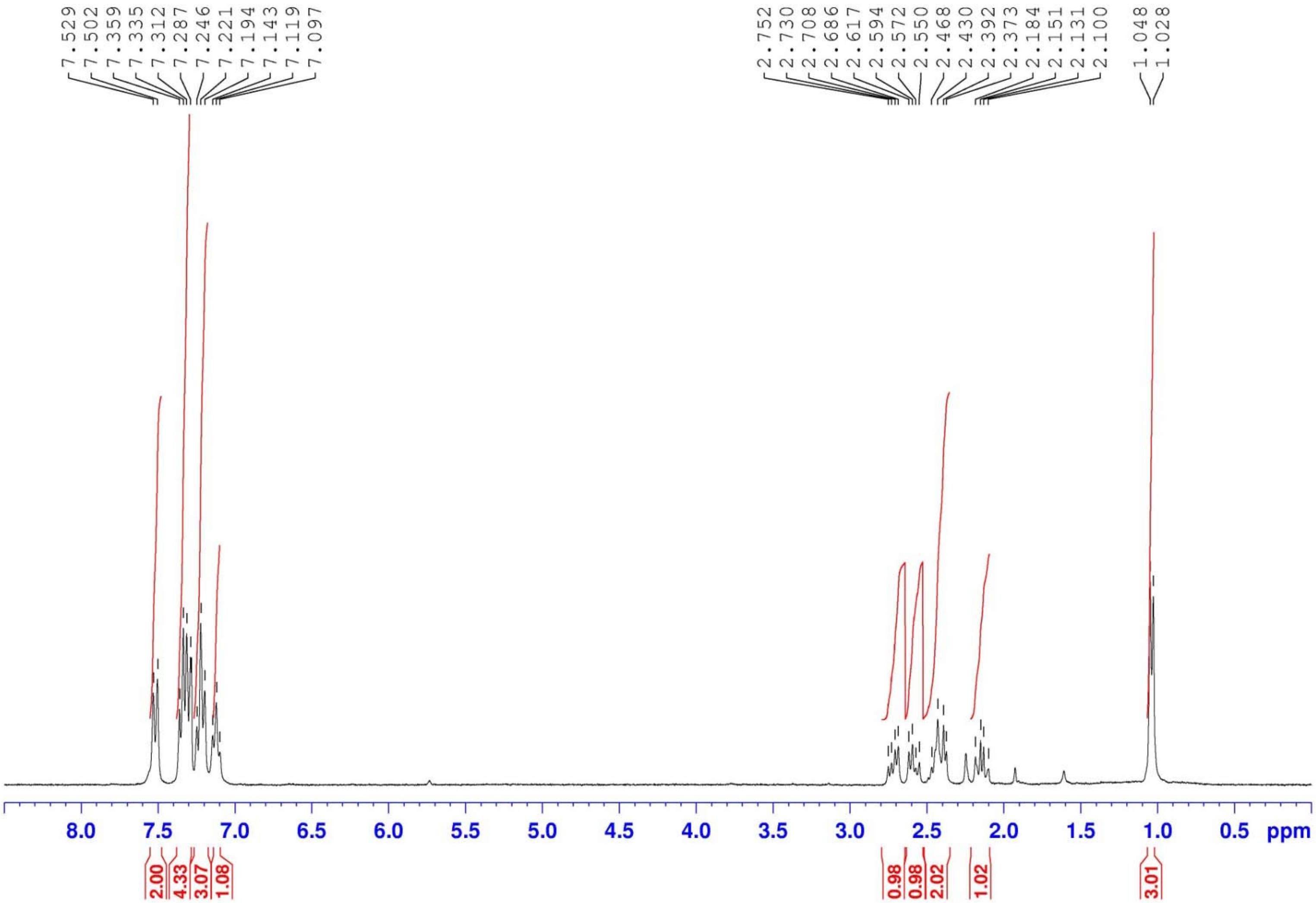




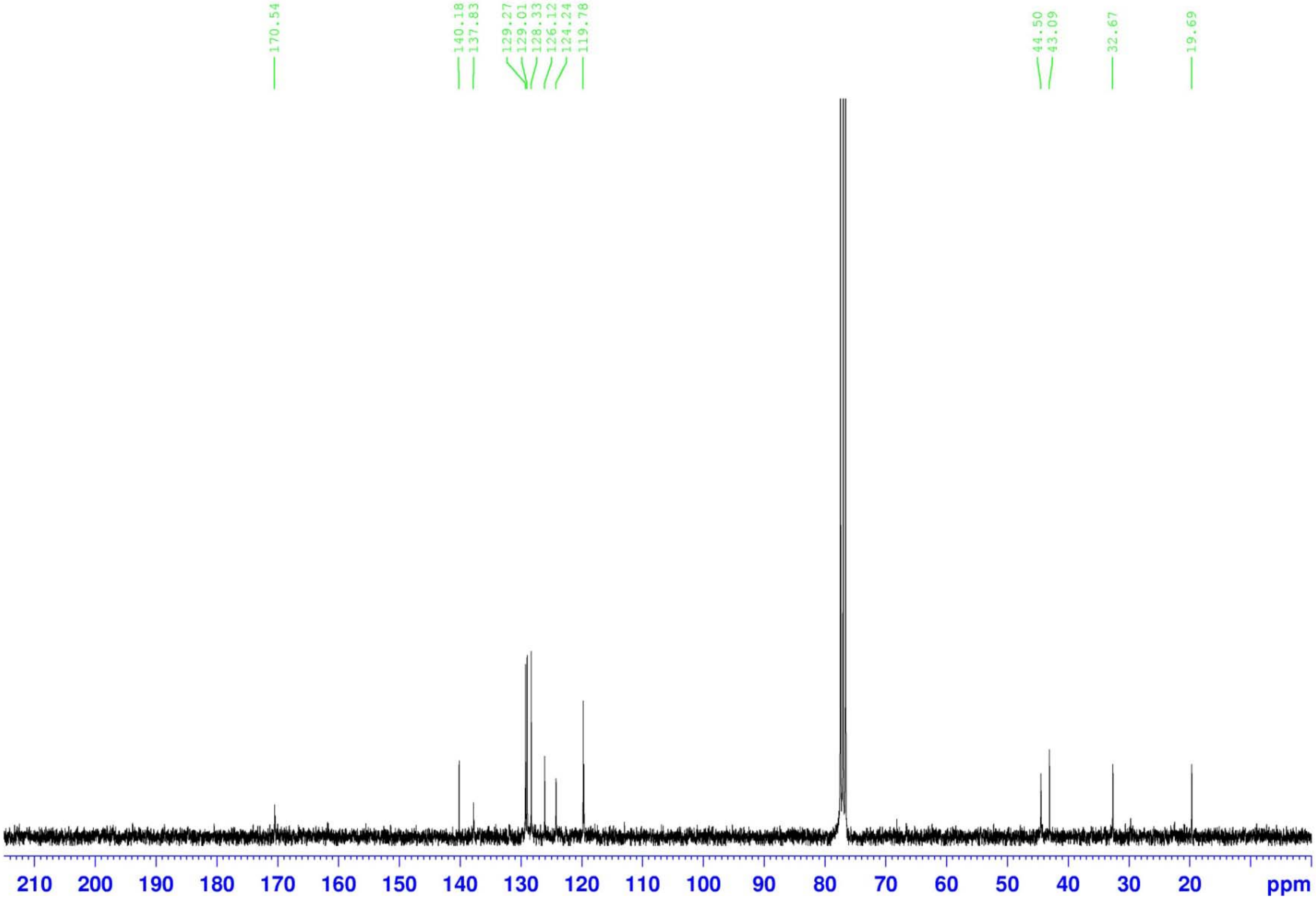
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10a** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (81%) as a white solid.

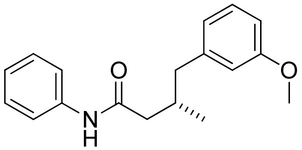
m.p.	59–60.5 ºC
Optical rotation	$[\alpha]_D^{20} = +6.5^\circ$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.4 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.52 (2H, d, *J* = 8.0 Hz, c,c'), 7.40–7.25 (4H, m, j,j',k,k'), 7.25–7.15 (3H, m, b,b',l), 7.12 (1H, t, *J* = 6.9 Hz, a), 2.72 (1H, dd, *J* = 13.2 Hz, 6.4 Hz, h), 2.58 (1H, dd, *J* = 13.2 Hz, 6.8Hz, h), 2.50–2.35 (2H, m, f,g), 2.20–2.05 (1H, m, f), 1.04 (3H, d, *J* = 6.2 Hz, m).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 170.54 (e), 140.18 (i), 137.83 (d), 129.27 (b,b'), 129.01 (j,j'), 128.33 (k,k'), 126.11 (l), 124.24 (a), 119.78 (c,c'), 44.50 (h), 43.09 (f), 32.67 (g), 19.69 (m).
IR (neat)	3292 (N-H stretch), 3062, 2924, 1657 (C=O stretch), 1600, 1544 (N-H bend), 1498, 1442, 1397, 1097 (C-N stretch), 1050, 754 cm^{-1}.
HRMS (EI)	Calcd. for $C_{17}H_{19}NO$: 253.1467, found 253.1474 *m/z*.

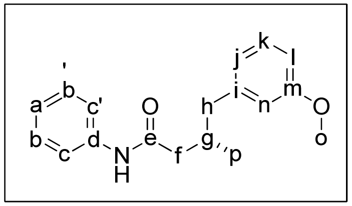
^{1}H NMR of (*S*)-3-methyl-4-phenylbutanoic acid phenyl amide



^{13}C NMR of (*S*)-3-methyl-4-phenylbutanoic acid phenyl amide



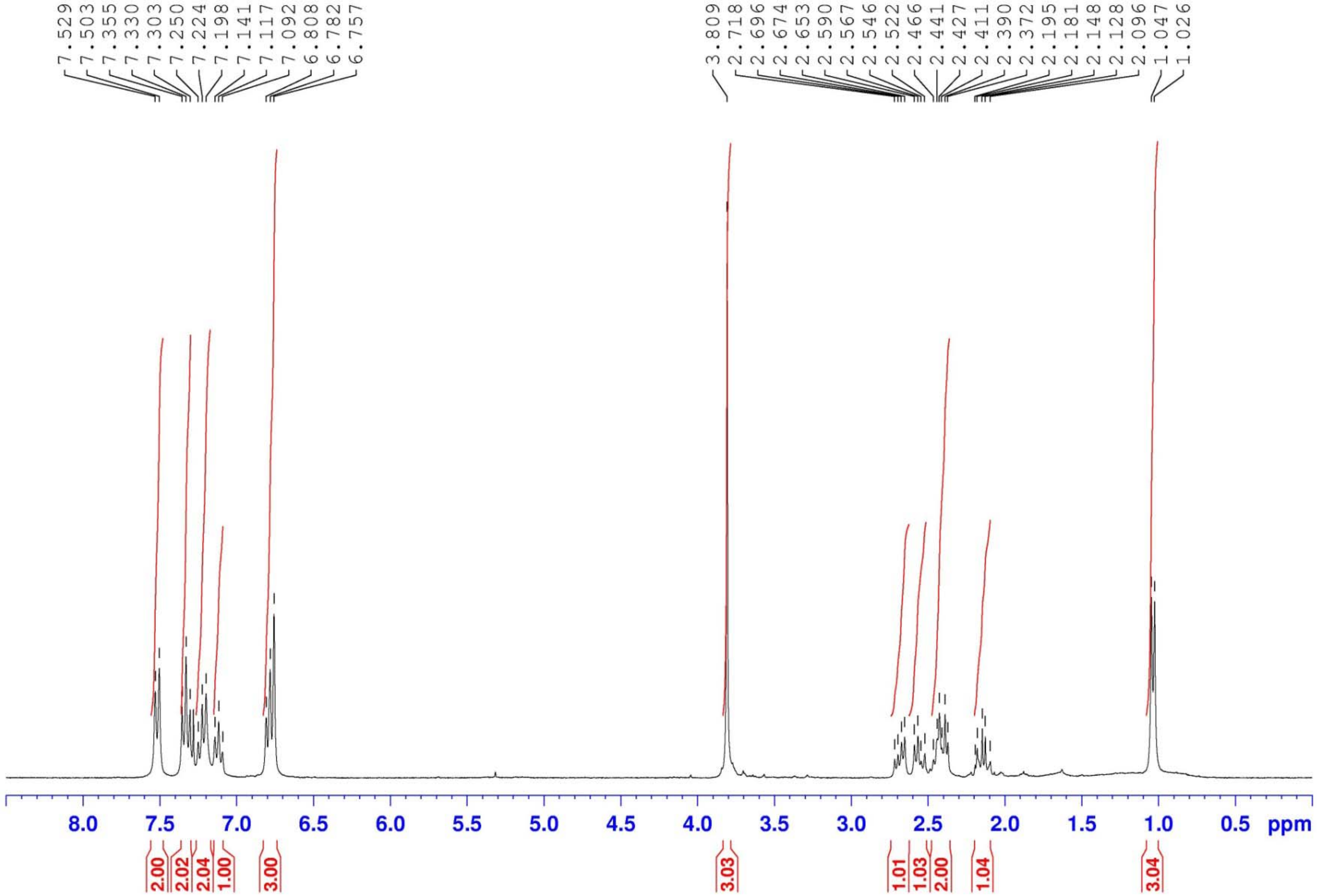




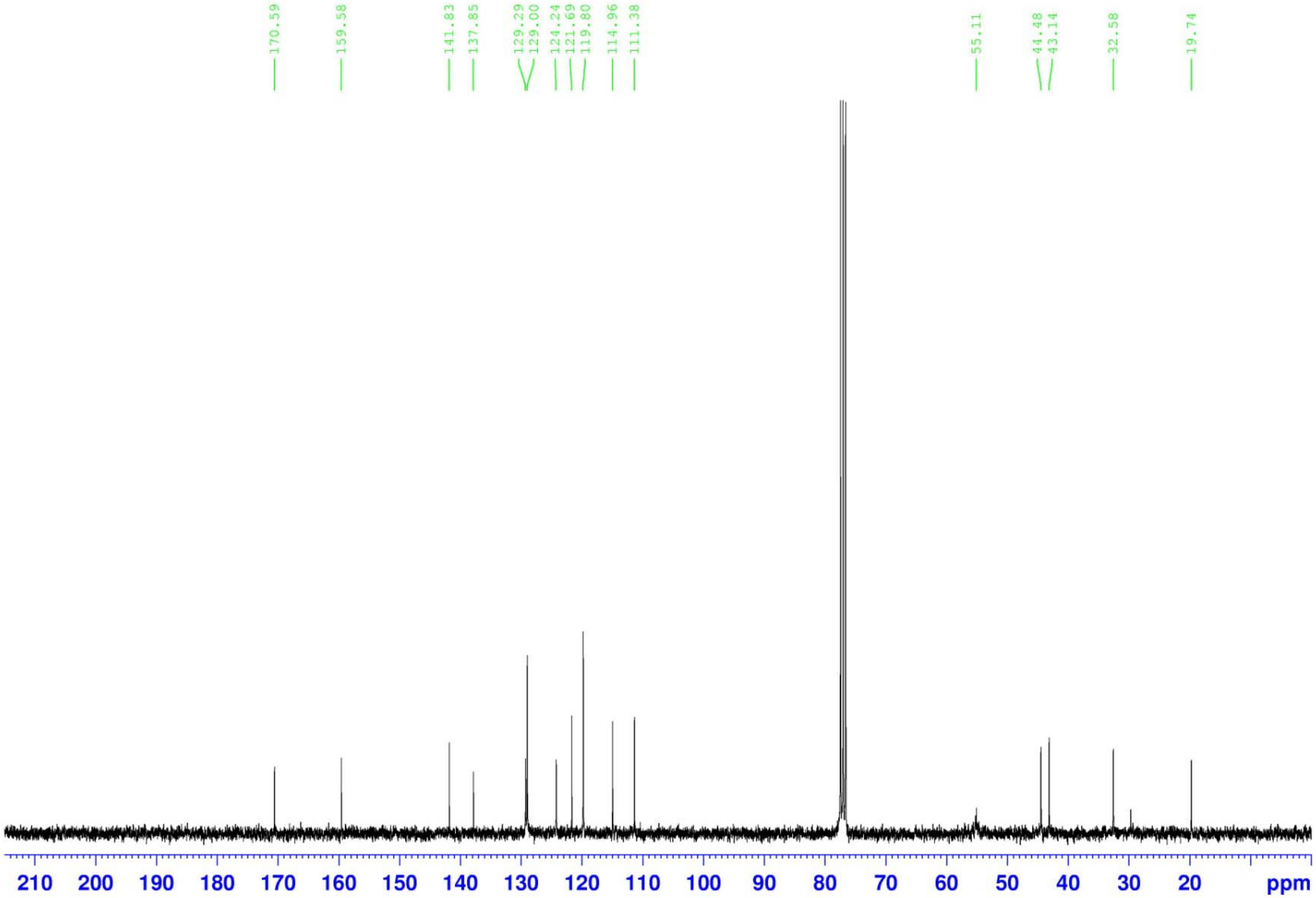
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10a** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (71%) as a white solid.

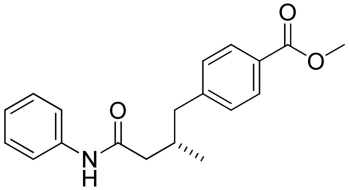
m.p.	65–66 ºC
Optical rotation	$[\alpha]_D^{20}$ = +2.9º (*c* 0.5, $CHCl_3$)
TLC analysis	0.40 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.52 (2H, d, *J* = 7.9 Hz, c,c’), 7.33 (2H, t, *J* = 7.8 Hz, b,b’), 7.30–7.15 (2H, m, NH,k), 7.12 (1H, t, *J* = 7.5 Hz, a), 6.85–6.70 (3H, m, j,l,n), 3.81 (3H, s, o), 2.67 (1H, dd, *J* = 13.2 Hz, 6.5 Hz, h), 2.56 (1H, dd, *J* = 13.2 Hz, 6.9 Hz, h), 2.50–2.35 (2H, m, f,g), 2.20–2.05 (1H, m, f), 1.04 (3H, d, *J* = 6.2 Hz, m).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 170.59 (e), 159.58 (m), 141.83 (i), 137.85 (d), 129.29 (b,b’), 129.00 (k), 124.24 (a), 121.69 (j), 119.80 (c,c’), 114.96 (n), 111.38 (l), 55.11 (o), 44.48 (h), 43.14 (f), 32.58 (g), 19.69 (p).
IR (neat)	3308 (N-H stretch), 2927, 1658 (C=O stretch), 1600, 1544 (N-H bend), 1499, 1441, 1322 (C-O stretch), 1260 (C-N stretch), 1153, 1069, 1043, 755 cm^{-1}.
HRMS (EI)	Calcd. for $C_{18}H_{21}NO_2$: 283.1572, found 283.1573 *m/z*.

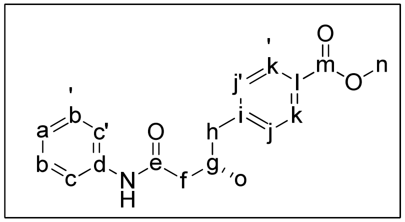
^{1}H NMR of (*S*)-4-(3-methoxyphenyl)-3-methylbutanoic acid phenyl amide



^{13}C NMR of (*S*)-4-(3-methoxyphenyl)-3-methylbutanoic acid phenyl amide



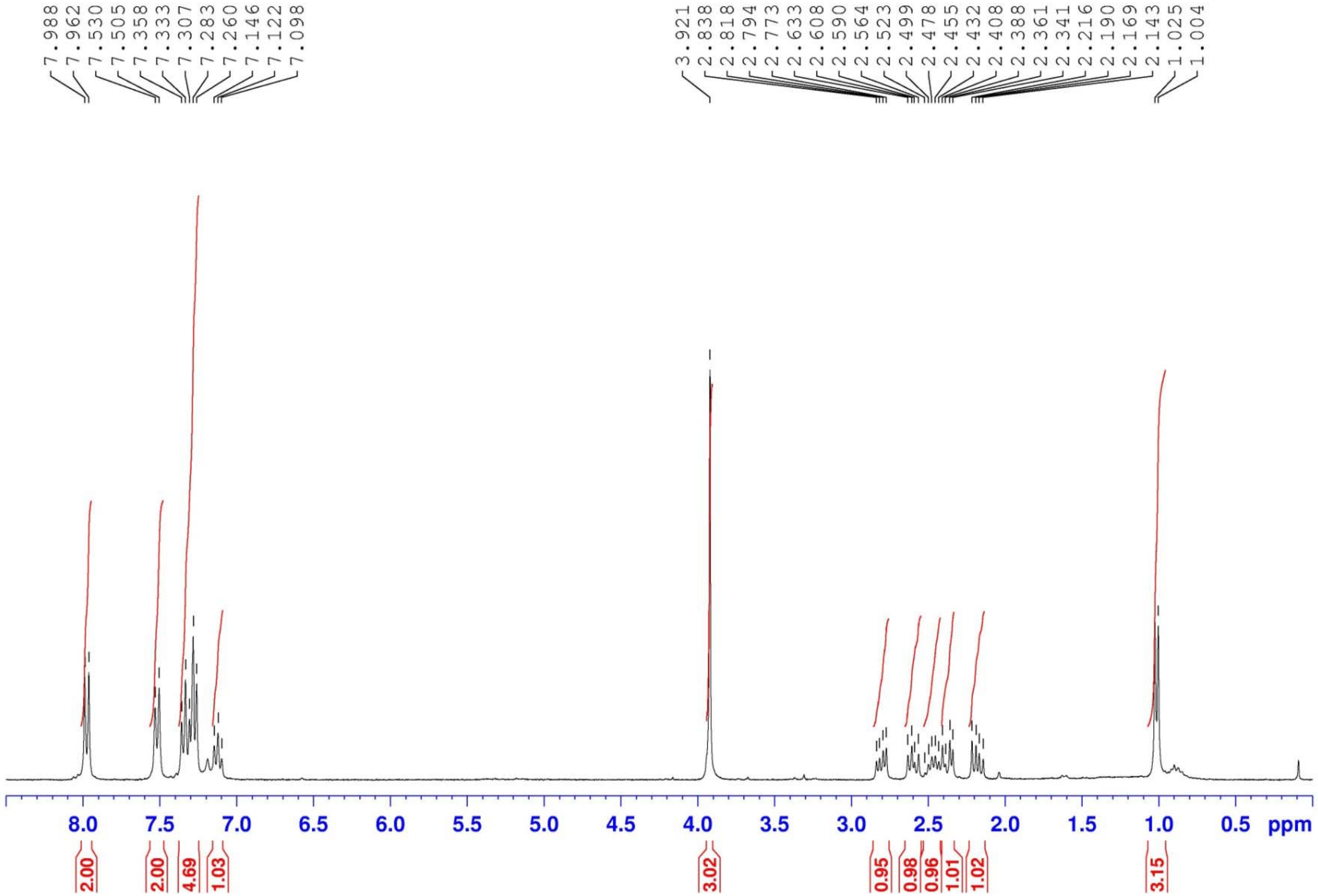




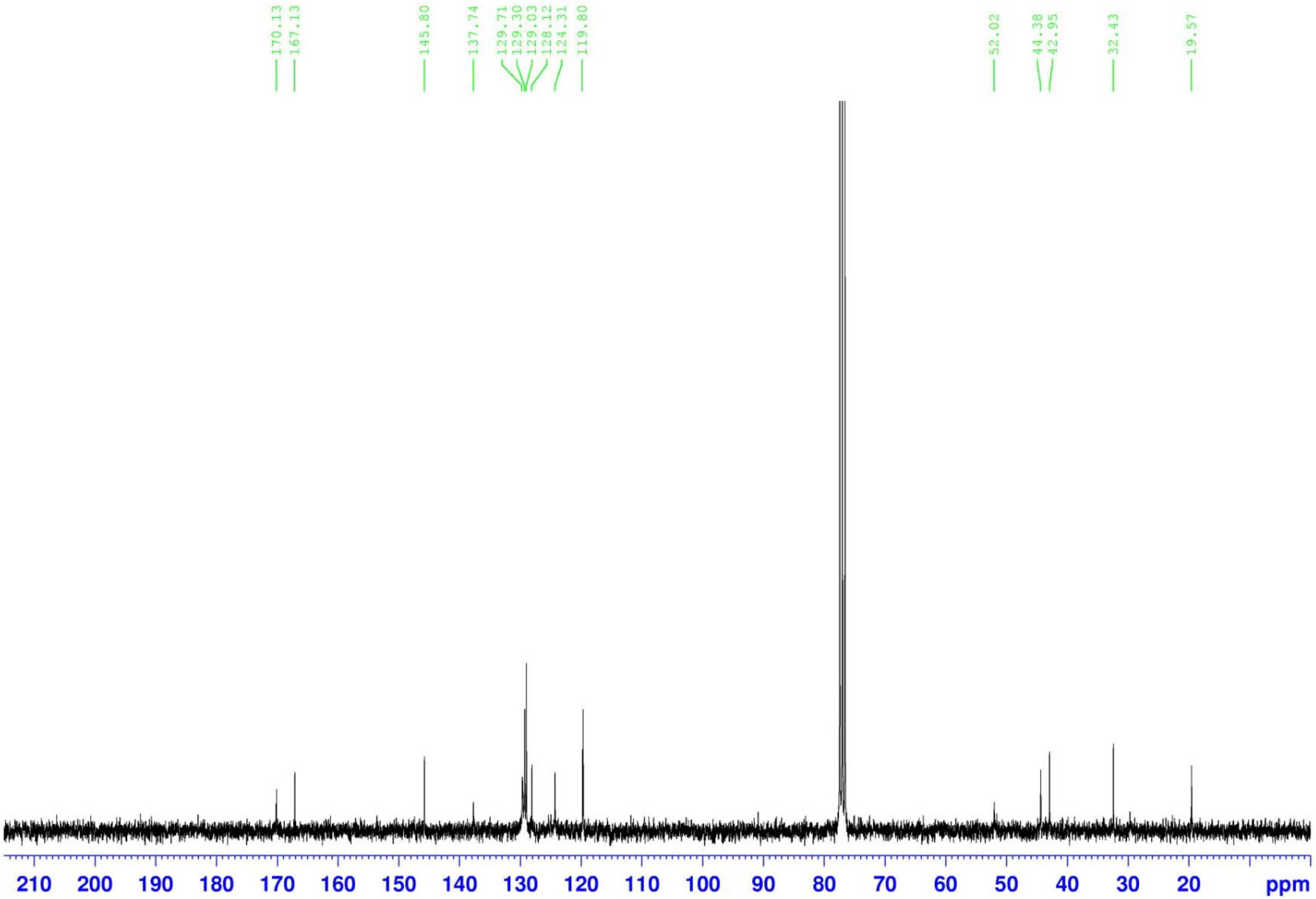
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10a** affords, after flash chromatography on silica gel (75:25 hexanes:ethyl acetate), the title compound (70%) as a white solid.

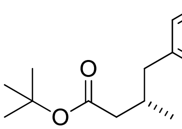
m.p.	68.5–69.5 ºC
Optical rotation	$[\alpha]_D^{20} = +8.0^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.4 (80:20 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.98 (2H, d, *J* = 7.9 Hz, k,k'), 7.52 (2H, d, *J* = 7.8 Hz, c,c'), 7.40–7.25 (4H, m, b,b',j,j'), 7.12 (1H, t, *J* = 7.3 Hz, a), 3.92 (3H, s, n), 2.81 (1H, dd, *J* = 13.1 Hz, 5.9 Hz, h), 2.60 (1H, dd, *J* = 13.1 Hz, 7.6 Hz, h), 2.55–2.40 (1H, m, g), 2.37 (1H, dd, *J* = 14.2 Hz, 5.9 Hz, f), 2.18 (1H, dd, *J* = 14.1 Hz, 7.7 Hz, f), 1.01 (3H, d, *J* = 6.4 Hz, o).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 170.13 (e), 167.13 (m), 145.80 (i), 137.74 (d), 129.70 (k,k'), 129.30 (b,b'), 129.03 (j,j'), 128.12 (l), 124.31 (a), 119.80 (c,c'), 52.02 (n), 44.38 (h), 42.95 (f), 32.43 (g), 19.57 (o).
IR (neat)	3311 (N-H stretch), 2923, 1719 (C=O stretch), 1655, 1600, 1530 (N-H stretch), 1444, 1301 (C-O stretch) 1273, 1175 (C-N stretch), 1020, 868, 767 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{19}H_{21}NaNO_3$ (M+Na): 334.1419, found 334.1426 *m/z*.

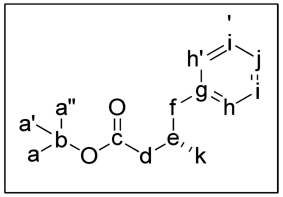
^{1}H NMR of (*S*)-3-methyl-4-(4-methylcarboxyphenyl)butanoic acid phenyl amide



^{13}C NMR of (*S*)-3-methyl-4-(4-methylcarboxyphenyl)butanoic acid phenyl amide



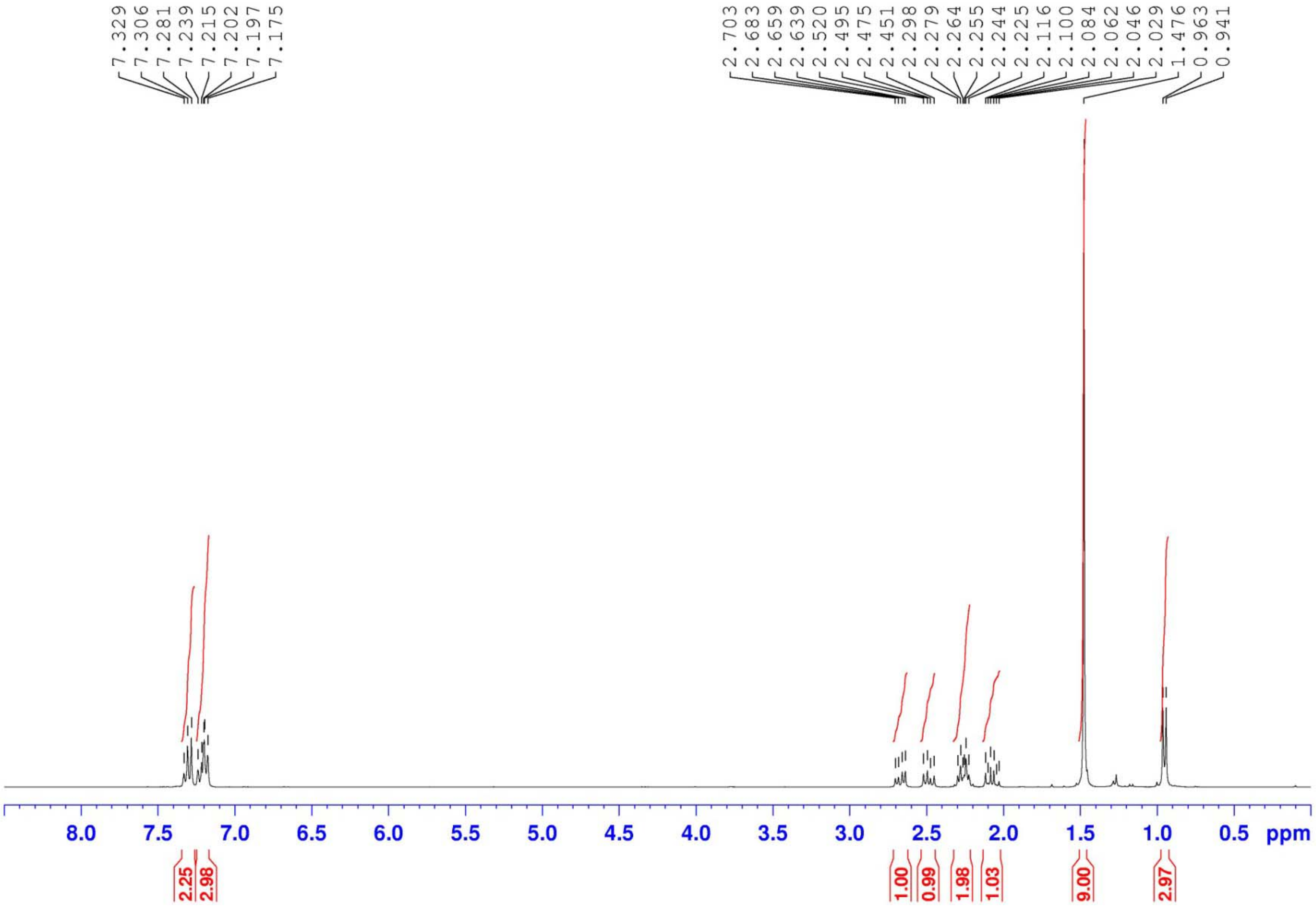




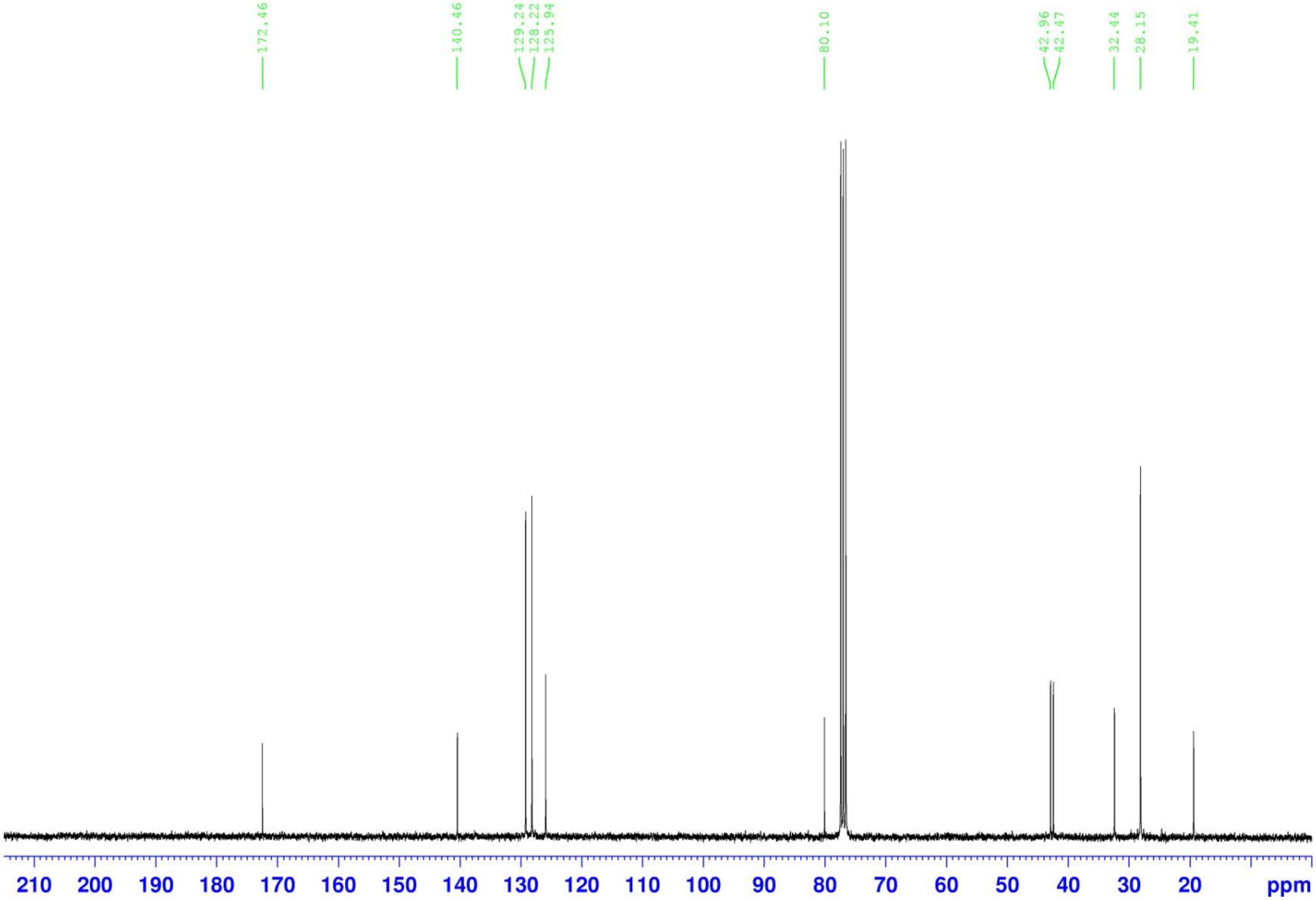
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10g** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (82%) as a colorless oil.

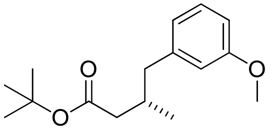
Optical rotation	$[\alpha]_D^{20} = +3.0^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (90:10 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.35–7.25 (2H, m, i,i'), 7.25–7.15 (3H, m, h,h',j), 2.67 (1H, dd, *J* = 13.4 Hz, 6.1 Hz, f), 2.49 (1H, dd, *J* = 13.4 Hz, 7.4 Hz, f), 2.30–2.20 (2H, m, d,e), 2.15–2.00 (1H, m, d), 1.48 (9H, s, a,a',a''), 0.95 (3H, d, *J* = 6.4 Hz, k).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 172.46 (c), 140.46 (g), 129.24 (i,i'), 128.22 (h,h'), 125.94 (j), 80.10 (b), 42.96 (f), 42.47 (d), 32.44 (e), 28.15 (a,a',a''), 19.41 (k).
IR (neat)	2977, 1725 (C=O stretch), 1454, 1392, 1367 (C-O stretch), 1146, 1051, 755 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{15}H_{22}NaO_2$ (M+Na): 257.1518, found 257.1520 *m/z*.

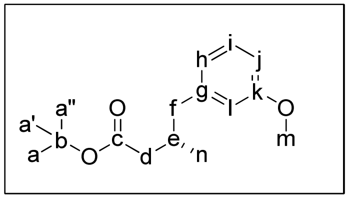
^{1}H NMR of (*S*)-3-methyl-4-phenylbutanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-methyl-4-phenylbutanoic acid *tert*-butyl ester



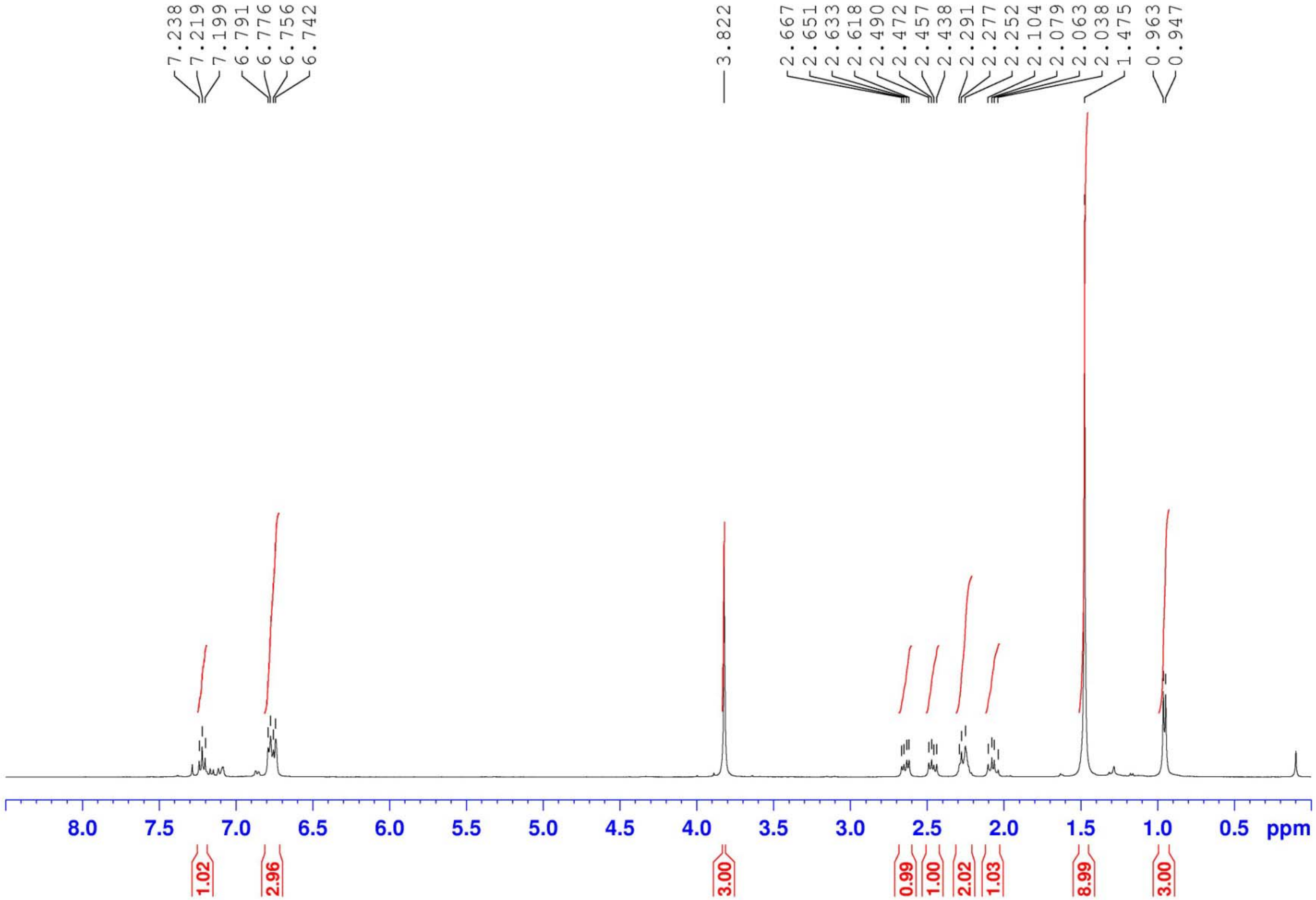




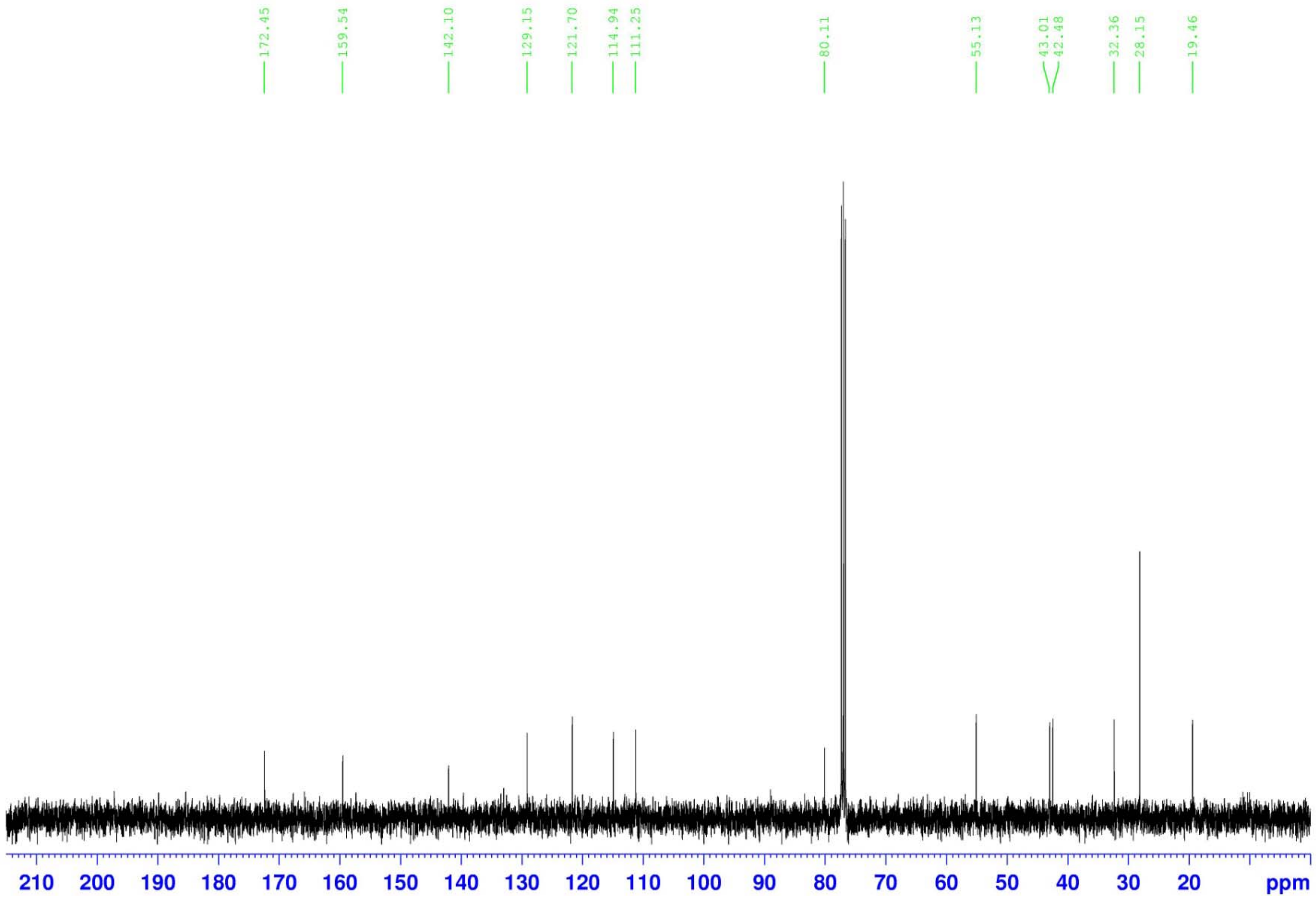
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10g** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (82%) as a colorless oil.

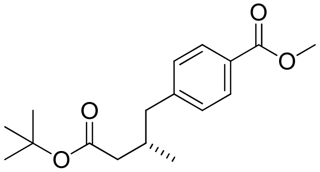
Optical rotation	$[\alpha]_D^{20} = +3.2^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.22 (1H, t, *J* = 7.8 Hz, i), 6.80–6.70 (3H, m, h,j,l), 3.82 (3H, s, m), 2.64 (1H, dd, *J* = 13.3 Hz, 6.1 Hz, f), 2.46 (1H, dd, *J* = 13.3, 7.4 Hz, f), 2.30–2.20 (2H, m, d,e), 2.15–2.00 (1H, m, d), 1.48 (9H, s, a,a',a''), 0.96 (3H, d, *J* = 6.3 Hz, n).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.45 (c), 159.54 (k), 142.10 (g), 129.15 (i), 121.70 (h), 114.94 (l), 111.25 (j), 80.11 (b), 55.13 (m), 43.01 (f), 42.48 (d), 32.36 (e), 28.15 (a,a',a''), 19.46 (n).
IR (neat)	2929, 1725 (C=O stretch), 1601, 1584, 1455, 1366, 1316 (C-O stretch), 1260, 1044, 779 cm^{-1}.
HRMS (EI)	Calcd. for $C_{16}H_{24}O_3$: 264.1725, found 264.1729 *m/z*.

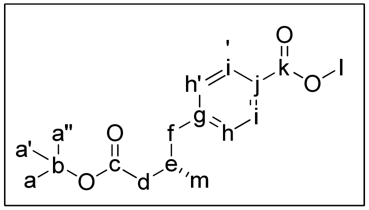
^{1}H NMR of (*S*)-3-methyl-4-(3-methoxyphenyl)butanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-methyl-4-(3-methoxyphenyl)butanoic acid *tert*-butyl ester



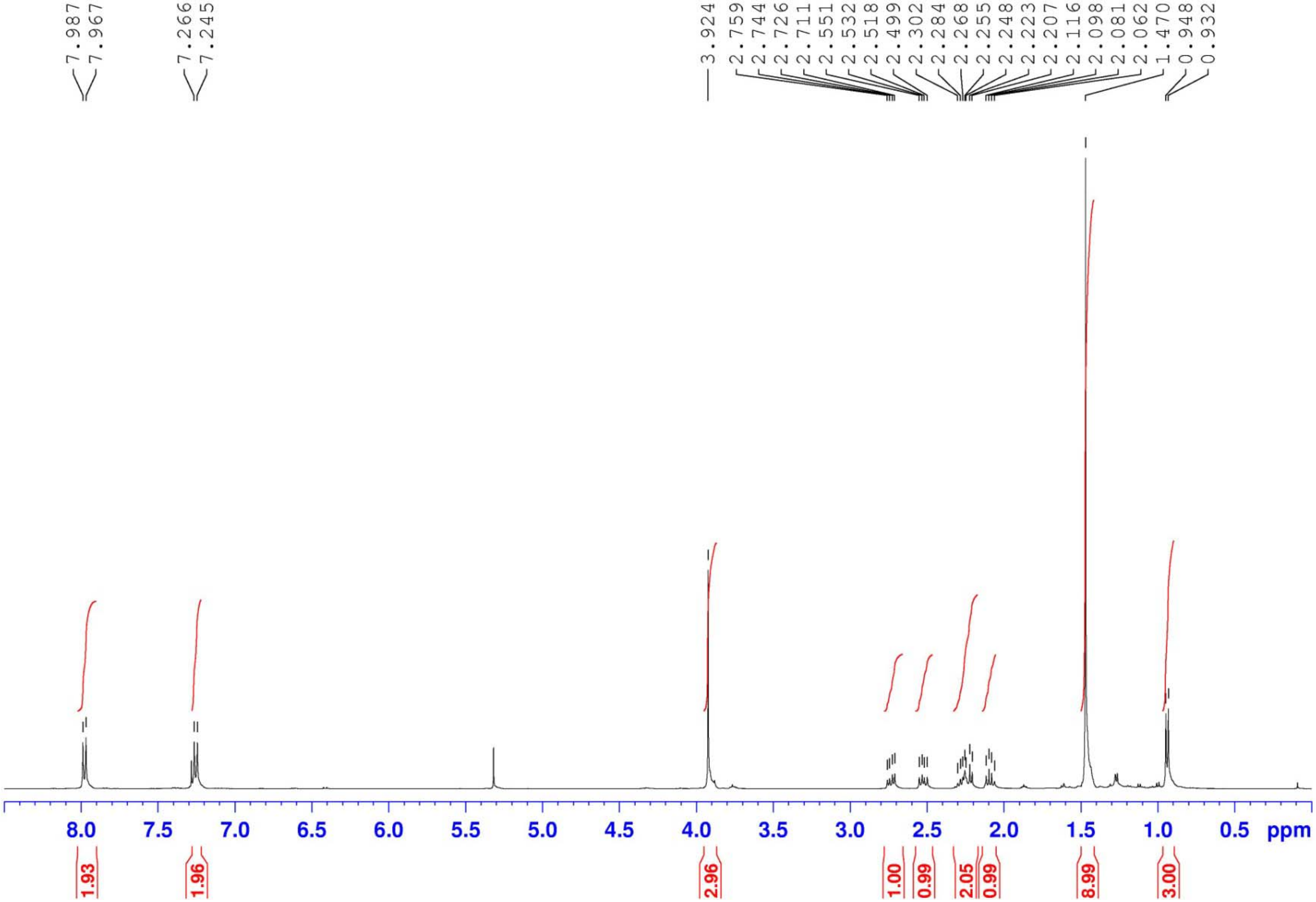




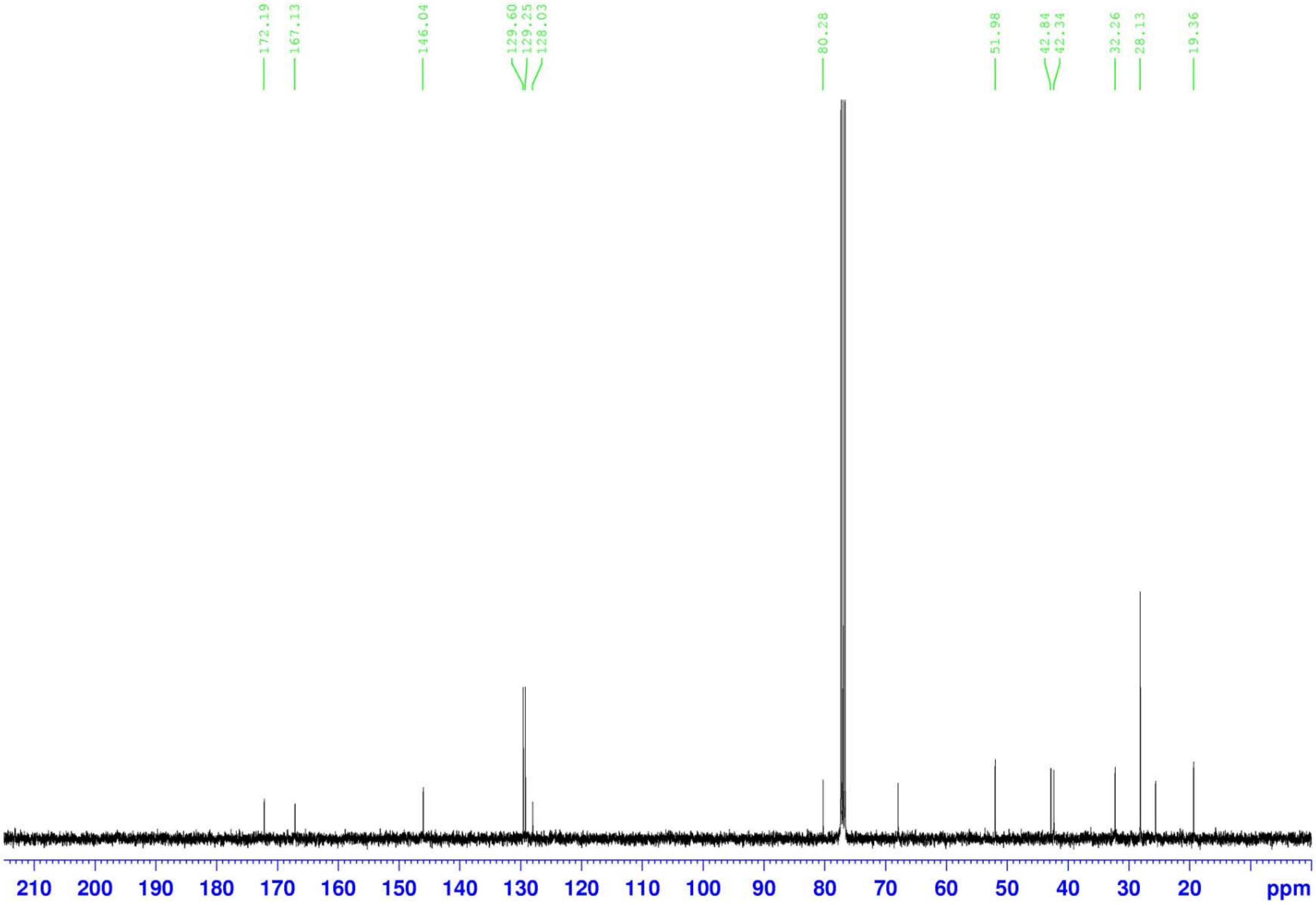
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*S*)-**10g** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (88%) as a white solid.

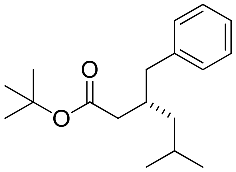
Optical rotation	$[\alpha]_D^{20} = +2.6^o$ (c 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.98 (2H, d, J = 8.2 Hz, i,i'), 7.26 (2H, d, J = 8.2 Hz, h,h'), 3.92 (3H, s, l), 2.73 (1H, dd, J = 13.3 Hz, 6.0 Hz, f), 2.53 (1H, dd, J = 13.3 Hz, 7.7 Hz, f), 2.35–2.20 (2H, m, d,e), 2.15–2.05 (1H, m, d), 1.47 (9H, s, a,a',a''), 0.94 (3H, d, J = 6.6 Hz, m).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.19 (c), 167.13 (k), 146.04 (g), 129.60 (i,i'), 129.25 (h,h'), 128.03 (j), 80.28 (b), 52.00 (l), 42.84 (f), 42.34 (d), 32.26 (e), 28.13 (a,a',a''), 19.36 (m).
IR (neat)	2931, 1722 (C=O stretch), 1610, 1457, 1415, 1367 (C-O stretch), 1278, 1178, 1148, 1108, 667, 759 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{17}H_{24}NaO_4$ (M+Na): 315.1572, found 315.1566 *m/z*.

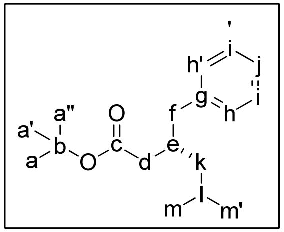
^{1}H NMR of (*S*)-3-methyl-4-(4-methylcarboxyphenyl)butanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-methyl-4-(4-methylcarboxyphenyl)butanoic acid *tert*-butyl ester



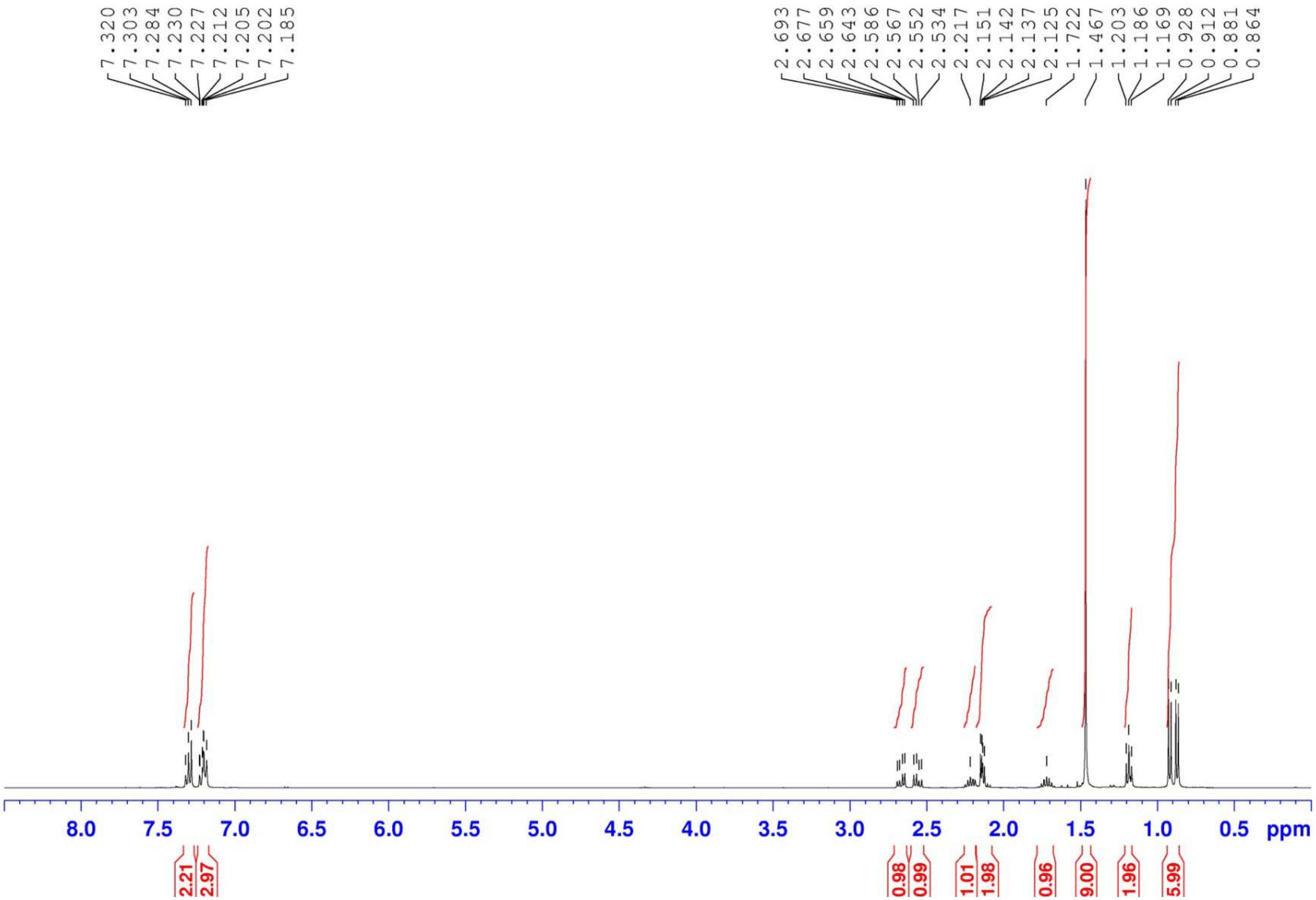




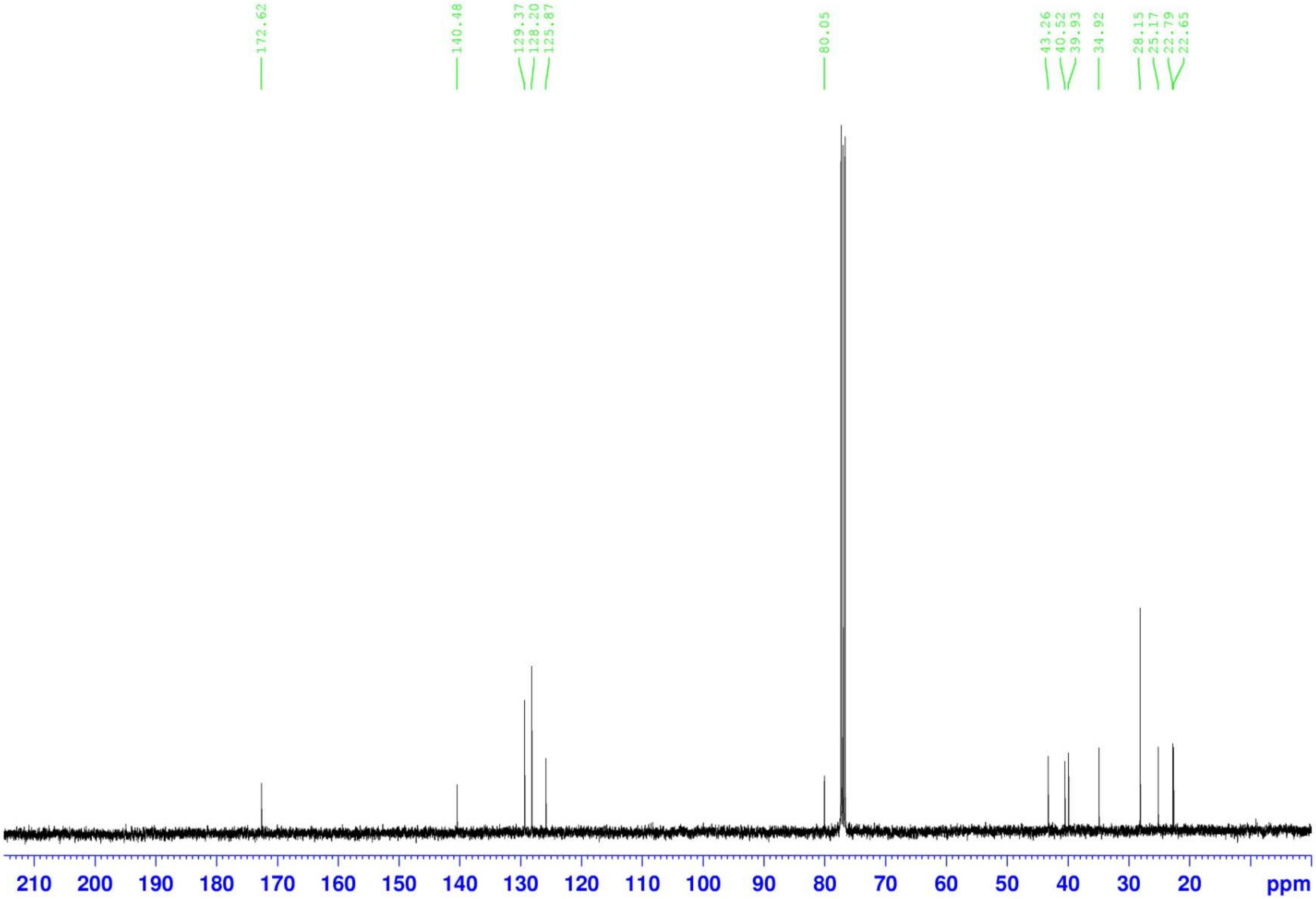
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (80%) as a colorless oil.

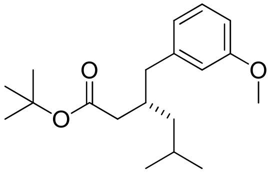
Optical rotation	$[\alpha]_D^{20}$ = +5.6° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.35–7.25 (2H, m, i,i’), 7.25–7.15 (3H, m, h,h’,j), 2.67 (1H, dd, *J* = 13.5 Hz, 6.1 Hz, f), 2.56 (1H, dd, *J* = 13.5 Hz, 7.2 Hz, f), 2.30–2.15 (1H, m, e), 2.15–2.10 (2H, m, d), 1.80–1.65 (1H, m, l), 1.47 (9H, s, a,a’,a’’), 1.86 (2H, t, *J* = 6.9 Hz, k), 0.90 (6H, dd, *J* = 19.1 Hz, 6.6 Hz, m,m’).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.62 (c), 140.46 (g), 129.37 (i,i’), 128.20 (h,h’), 125.87 (j), 80.05 (b), 43.26 (k), 40.52 (f), 39.93 (d), 34.92 (e), 28.15 (a,a’,a’’), 25.17 (l), 22.79 and 22.65 (m,m’).
IR (neat)	2955, 1727 (C=O stretch), 1454, 1391, 1366 (C-O stretch), 1299, 1256, 1146, 747 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{18}H_{28}NaO_2$ (M+Na): 299.1987, found 299.1974 *m/z*.

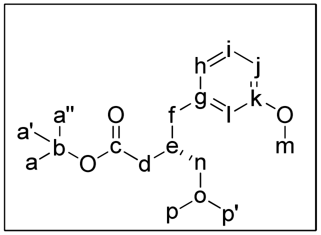
^{1}H NMR of *S*)-5-methyl-3-phenylmethylhexanoic acid *tert*-butyl ester



^{13}C NMR of *S*)-5-methyl-3-phenylmethylhexanoic acid *tert*-butyl ester



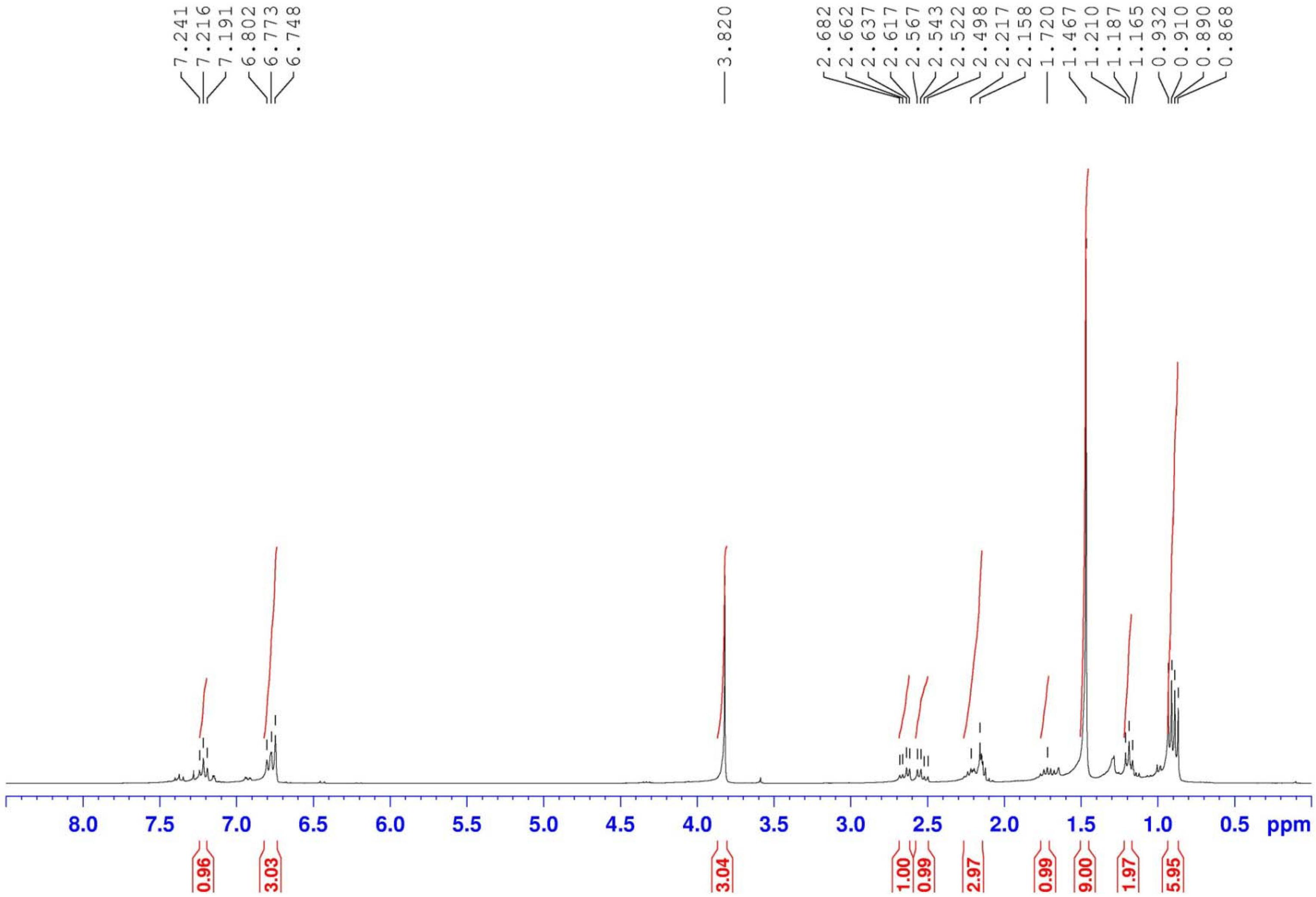




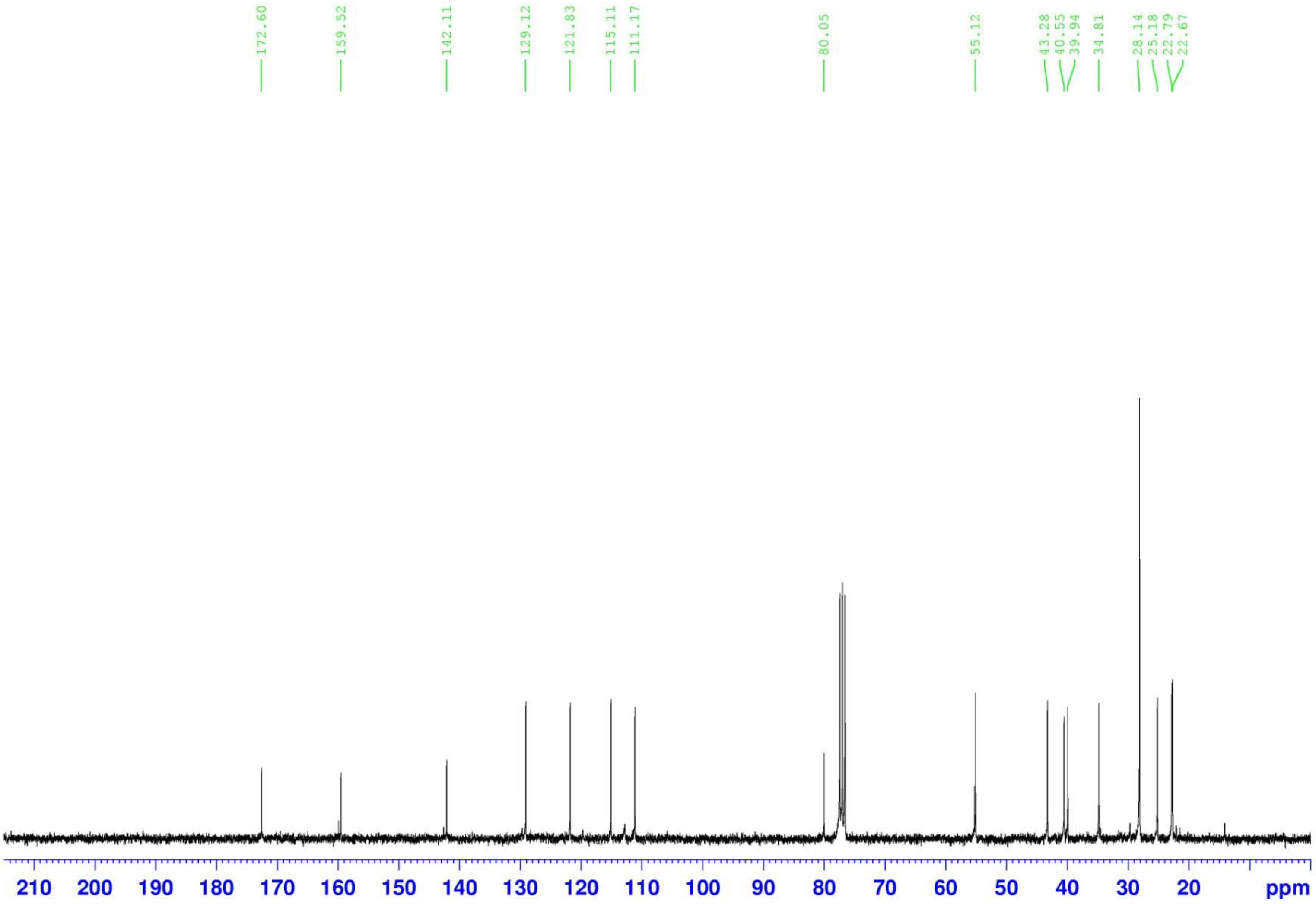
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (92%) as a colorless oil.

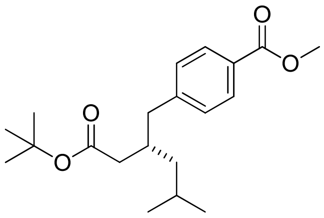
Optical rotation	$[\alpha]_D^{20} = +2.3^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.60 (90:10 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.22 (1H, t, $J = 7.5$ Hz, i), 6.85–6.70 (3H, m, h,j,l), 3.82 (3H, s, m), 2.65 (1H, dd, $J = 13.5$ Hz, 5.8 Hz, f), 2.53 (1H, dd, $J = 13.5$ Hz, 7.0 Hz, f), 2.30–2.05 (3H, m, d,e), 1.80–1.65 (1H, m, o), 1.47 (9H, s, a,a',a''), 1.19 (2H, t, $J = 6.8$ Hz, n), 0.90 (6H, dd, $J = 12.7$ Hz, 6.5 Hz, p,p').
^{13}C NMR (75 MHz, $CDCl_3$)	δ 172.60 (c), 159.52 (k), 142.11 (g), 129.12 (i), 121.83 (h), 115.11 (l), 111.17 (j), 80.05 (b), 55.12 (m), 43.28 (n), 40.55 (f), 39.94 (d), 34.81 (e), 28.14 (a,a',a''), 25.17 (o), 22.79 and 22.67 (p,p').
IR (neat)	2955, 1726 (C=O stretch), 1600, 1488, 1455, 1366 (C-O stretch), 1260, 1150, 1047, 777 cm^{-1}.
HRMS (EI)	Calcd. for $C_{19}H_{30}O_3$: 306.2195, found 306.2207 *m/z*.

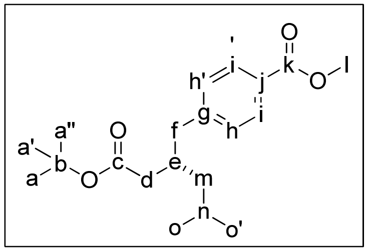
^{1}H NMR of (*S*)-5-methyl-3-(3-methoxyphenyl)methylhexanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-5-methyl-3-(3-methoxyphenyl)methylhexanoic acid *tert*-butyl ester



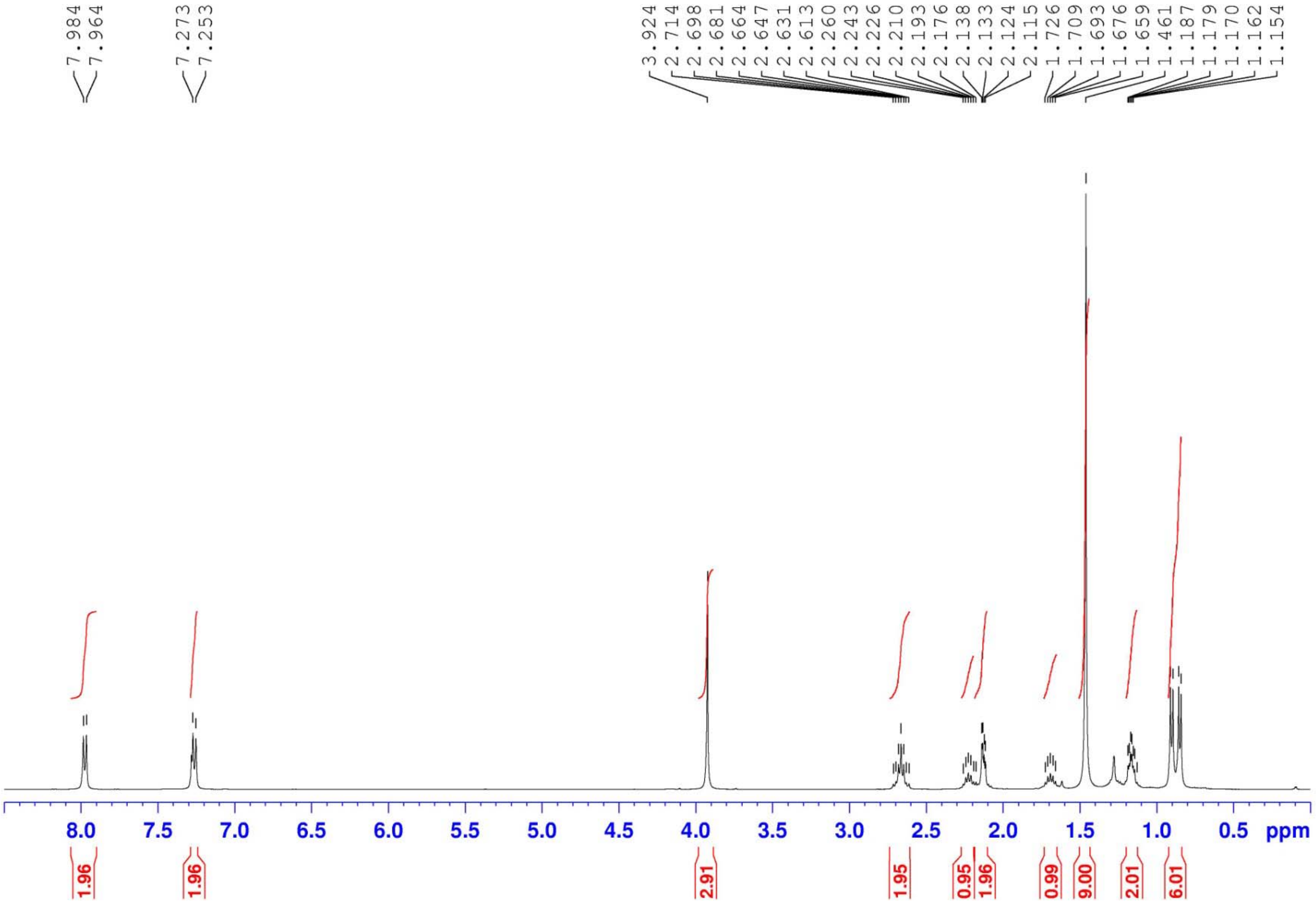




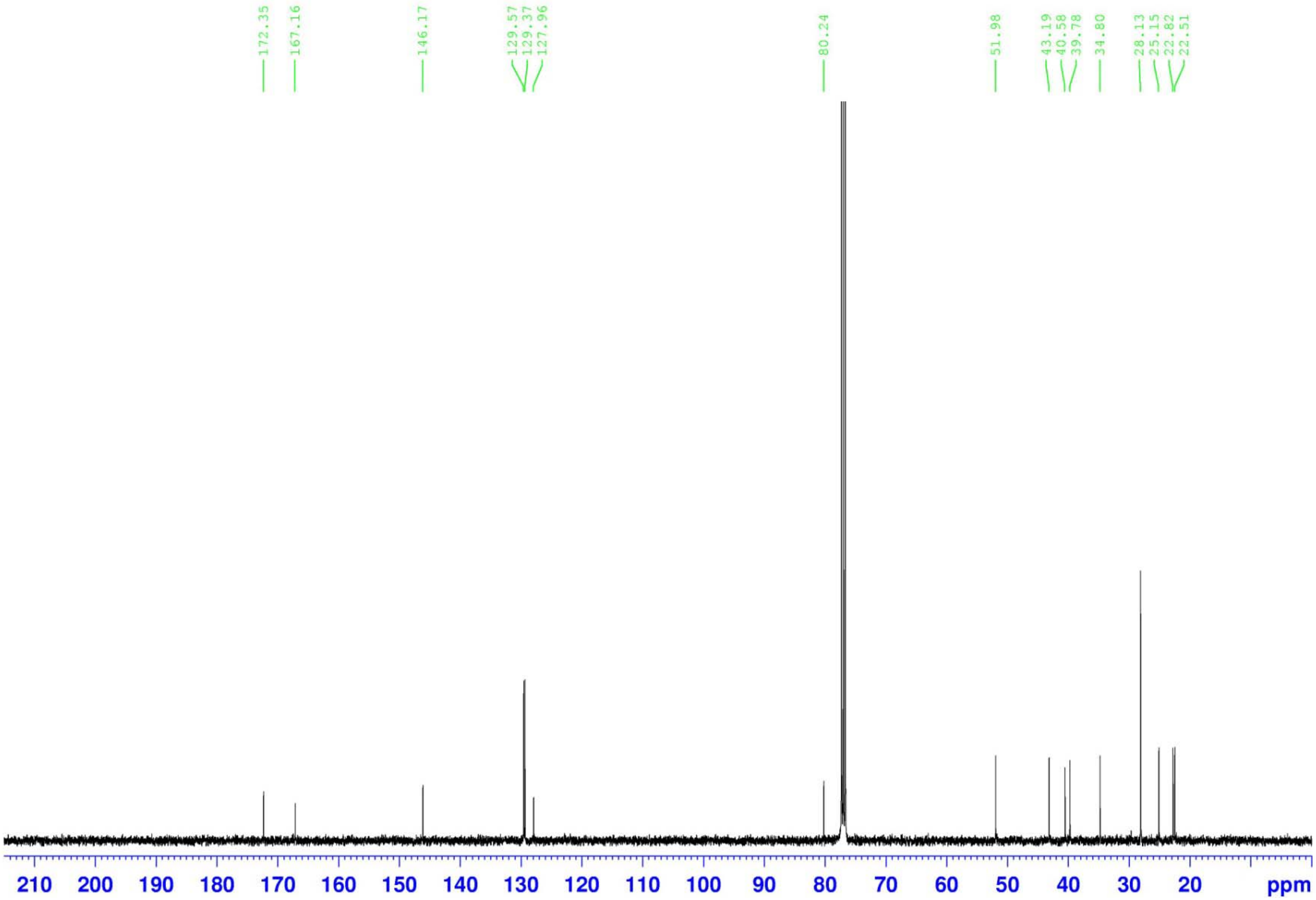
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (94%) as a colorless oil.

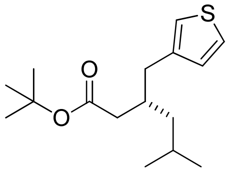
Optical rotation	$[\alpha]_D^{20} = +4.1^{o}$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.7 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.97 (2H, d, *J* = 8.1 Hz, i,i'), 7.26 (2H, d, *J* = 8.1 Hz, h,h'), 3.92 (3H, s, l), 2.75–2.60 (2H, m, f), 2.30–2.15 (1H, m, e), 2.15–2.10 (2H, m, d), 1.75–1.60 (1H, m, n), 1.46 (9H, s, a,a',a''), 1.20–1.10 (2H, m, m), 0.88 (6H, dd, *J* = 21.3 Hz, 6.5 Hz, o,o').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.35 (c), 167.16 (k), 146.17 (g), 129.57 (i,i'), 129.37 (h,h'), 127.96 (j), 80.24 (b), 51.98 (l), 43.19 (m), 40.58 (f), 39.78 (d), 34.80 (e), 28.13 (a,a',a''), 25.15 (n), 22.82 and 22.51 (o,o').
IR (neat)	2955, 1726 (C=O stretch), 1600, 1584, 1488, 1455, 1366 (C-O stretch), 1260, 1150, 1047, 851, 777 cm^{-1}.
HRMS (ESI)	Calcd. for $C_{20}H_{30}NaO_4$ (M+Na): 357.2042, found 357.2028 *m/z*.

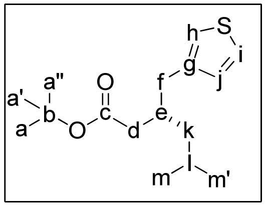
^{1}H NMR of (*S*)-3-(4-methylcarboxyphenyl)methyl-5-methylhexanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-(4-methylcarboxyphenyl)methyl-5-methylhexanoic acid *tert*-butyl ester



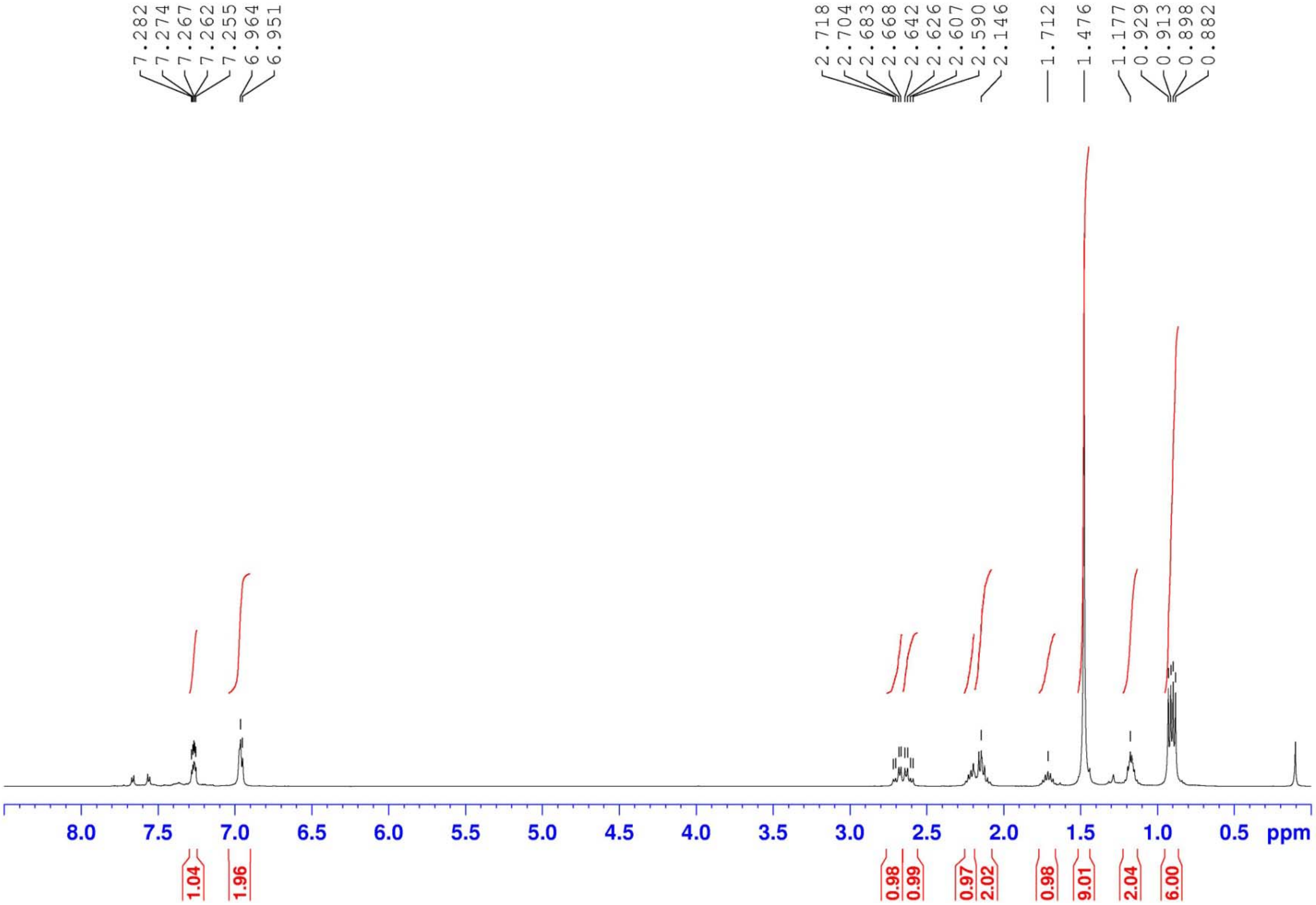




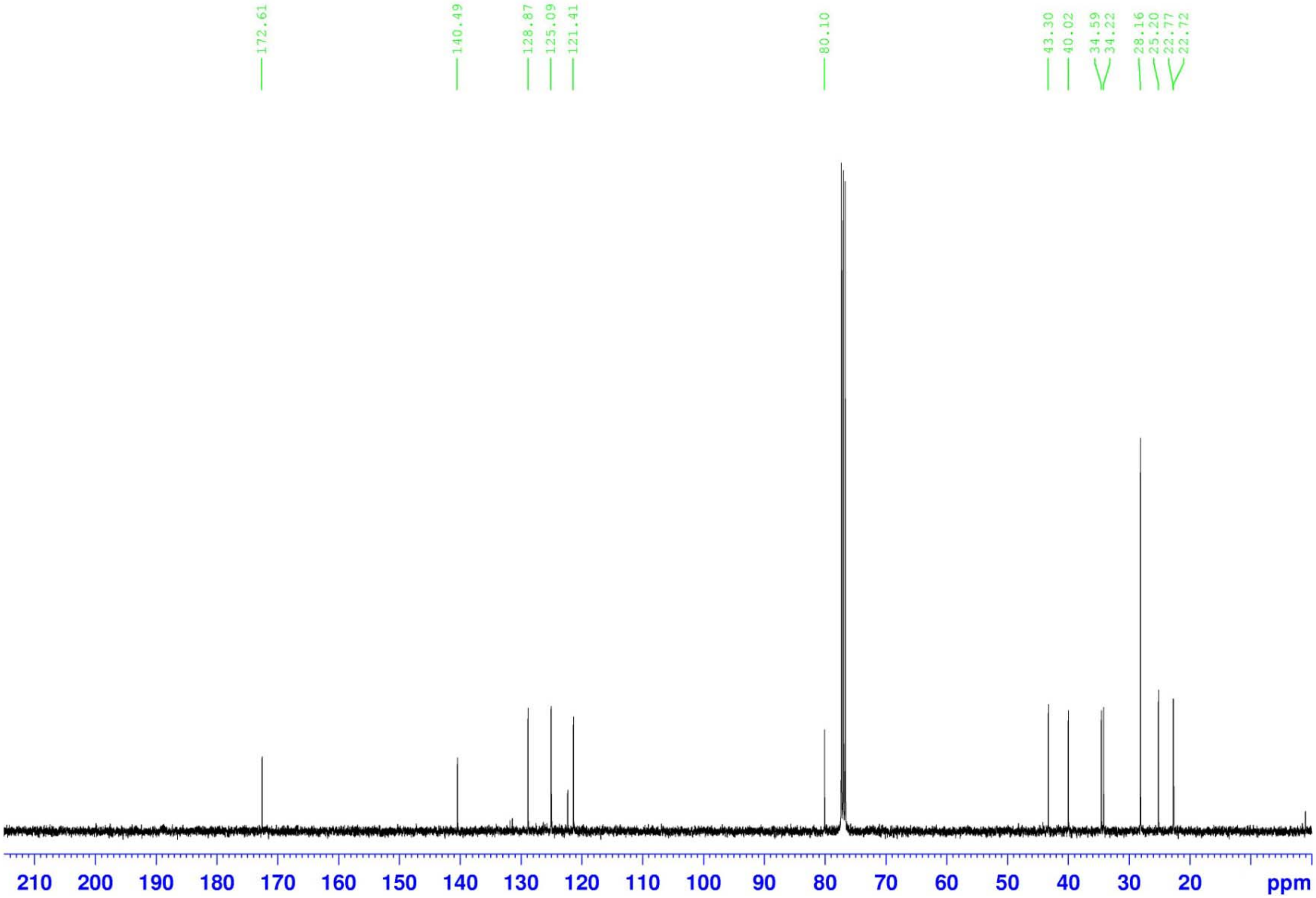
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (98%) as a yellow oil.

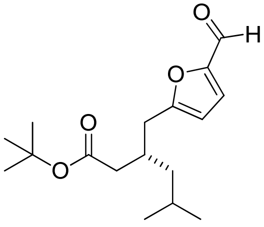
Optical rotation	$[\alpha]_D^{20} = +1.9^o$ (c 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.20 (1H, m, i), 7.00–6.90 (2H, m, h,j), 2.69 (1H, dd, J = 14.1 Hz, 5.6 Hz, f), 2.62 (1H, dd, J = 14.1 Hz, 6.7 Hz, f), 2.25–2.15 (1H, m, e), 2.15–2.05 (2H, m, d), 1.80–1.65 (1H, m, l), 1.48 (9H, s, a,a',a''), 1.25–1.10 (2H, m, k), 0.91 (6H, dd, J = 12.4 Hz, 6.6 Hz, m,m').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.61 (c), 140.49 (g), 128.87 (j), 125.09 (i), 121.41 (h), 80.10 (b), 43.30 (k), 40.02 (d), 34.59 (f), 34.22 (e), 25.20 (a,a',a''), 25.20 (l), 22.77 and 22.72 (m,m').
IR (neat)	2956, 2928, 1725 (C=O stretch), 1455, 1391, 1366 (C-O stretch), 1298, 1255, 1146, 851, 772 cm^{-1}.
HRMS (EI)	Calcd. for $C_{16}H_{26}OS$: 282.1653, found 282.1652 *m/z*.

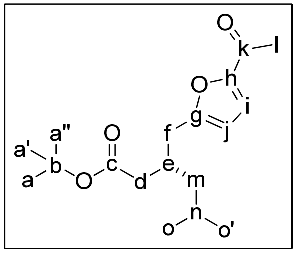
^{1}H NMR of (*S*)-5-methyl-3-(thiophen-3-yl)methylhexanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-5-methyl-3-(thiophen-3-yl)methylhexanoic acid *tert*-butyl ester



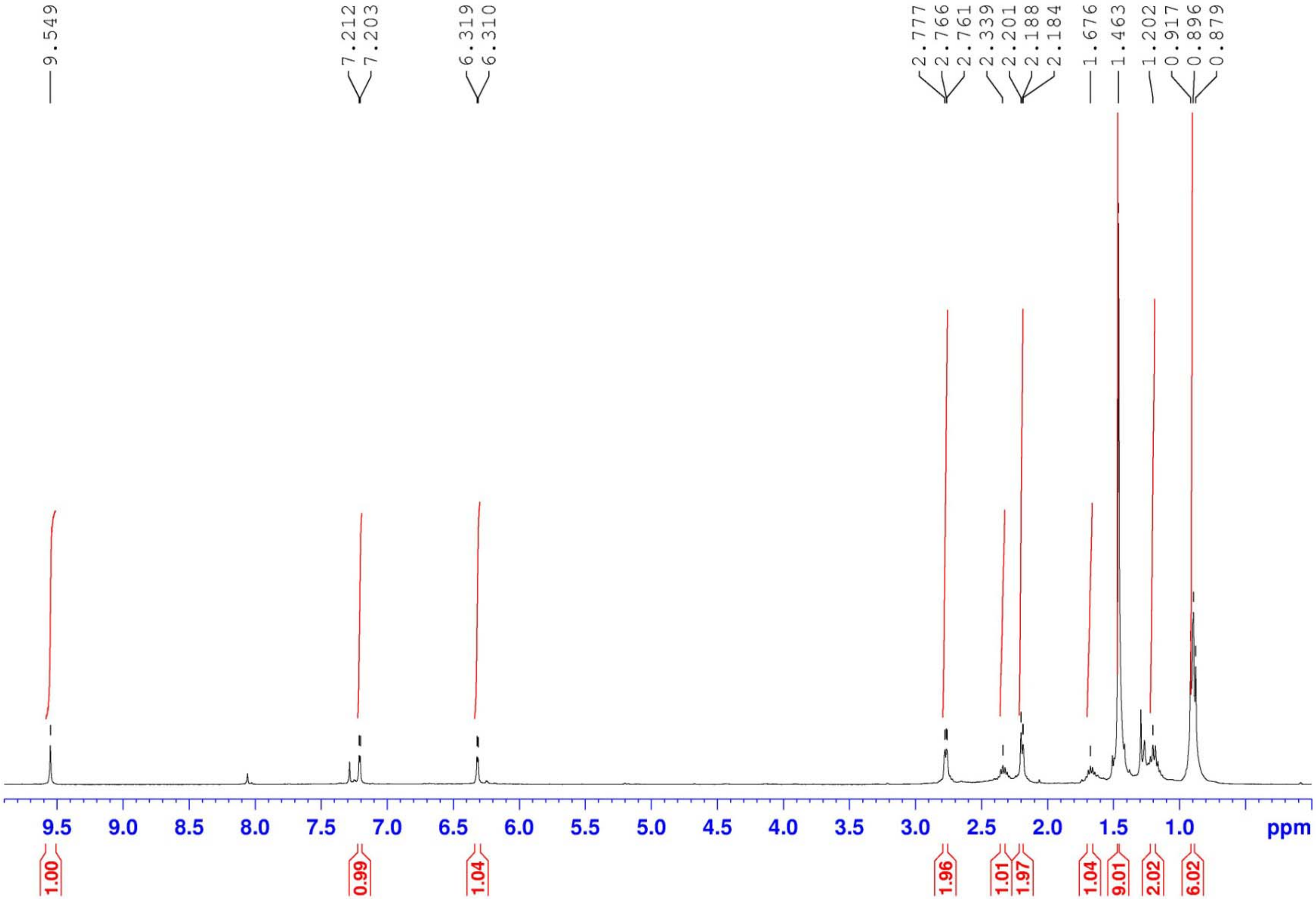




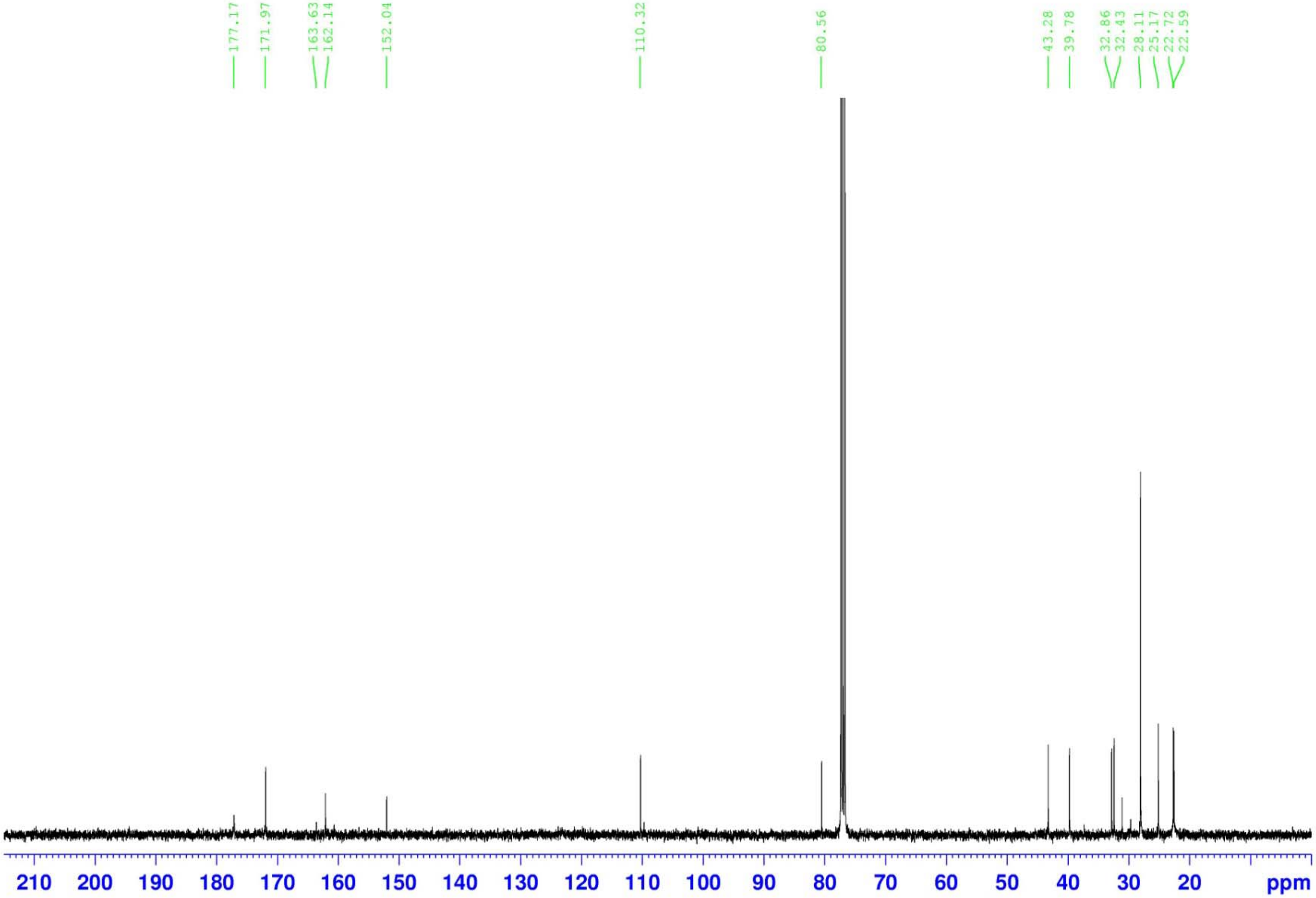
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (96:4 hexanes:ethyl acetate), the title compound (84%) as a yellow oil.

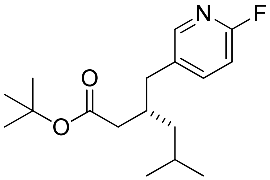
Optical rotation	$[\alpha]_D^{20} = +4.3^{\circ}$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.40 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 9.55 (1H, s, l), 7.21 (1H, d, *J* = 3.3 Hz, i), 6.31 (1H, d, *J* = 3.3 Hz, j), 2.80–2.70 (2H, m, f), 2.40–2.25 (1H, m, e), 2.25–2.15 (2H, m, d), 1.75–1.60 (1H, m, n), 1.46 (9H, s, a,a',a''), 1.25–1.10 (2H, m, m), 0.90 (6H, dd, *J* = 8.3 Hz, 6.9 Hz, o,o').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 177.17 (k), 172.00 (c), 162.14 (g), 152.04 (h), 110.32 (j), 80.56 (b), 43.28 (m), 39.78 (d), 32.86 (f), 32.43 (e), 28.11 (a,a',a''), 25.17 (l), 22.72 and 22.59 (o,o').
IR (neat)	2956, 1723 (C=O stretch), 1680 (C=O stretch), 1516, 1468, 1368 (C-O stretch), 1255, 1152, 1023, 960, 755 cm^{-1}.
HRMS (EI)	Calcd. for $C_{17}H_{26}NaO_4$ (M+Na): 317.1729, found 317.1735 *m/z*.

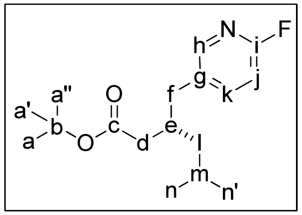
^{1}H NMR of (*S*)-3-(5-acetylfuran-2-yl)methyl-5-methylhexanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-(5-acetylfuran-2-yl)methyl-5-methylhexanoic acid *tert*-butyl ester



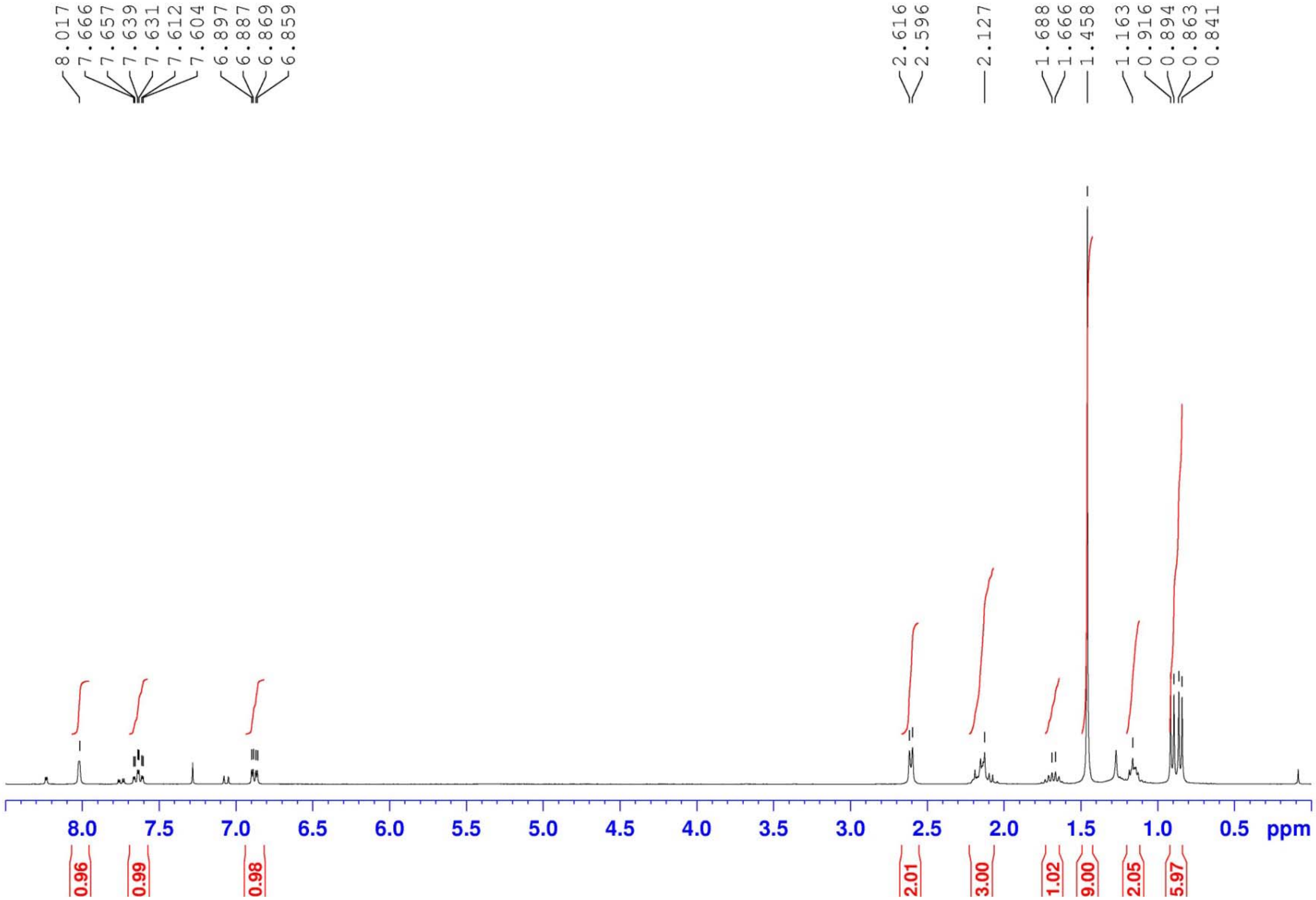




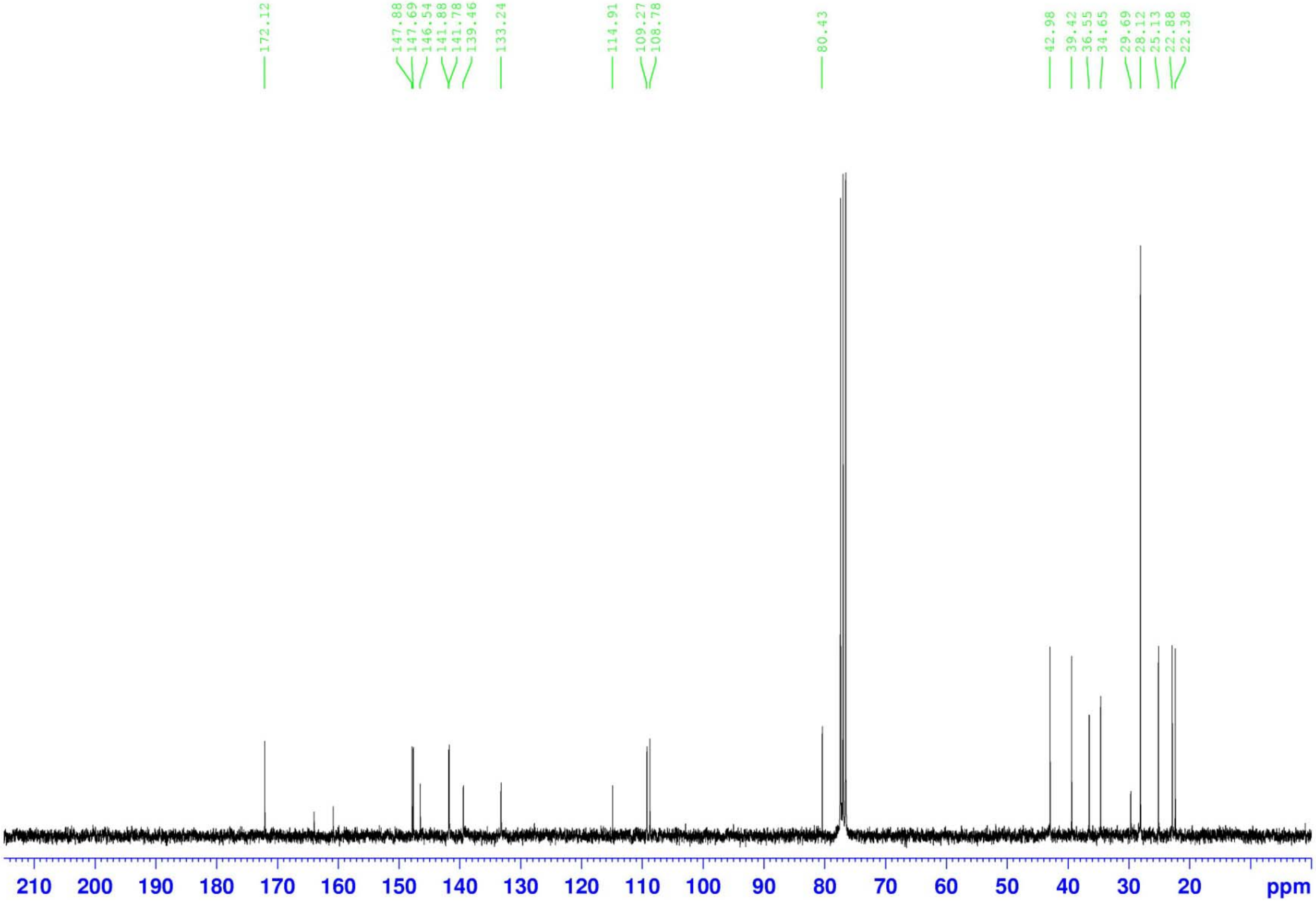
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10i** affords, after flash chromatography on silica gel (92:8 hexanes:ethyl acetate), the title compound (51%) as a colorless oil.

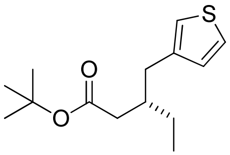
Optical rotation	$[\alpha]_D^{20}$ = +3.8° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 8.02 (1H, s, k), 7.63 (1H, td, *J* = 8.1 Hz, 2.5 Hz, h), 6.88 (1H, dd, *J* = 8.3 Hz, 2.9 Hz, i), 2.61 (2H, d, *J* = 6.0 Hz, f), 2.20–2.05 (3H, m, d,e), 1.80–1.55 (1H, m, m), 1.46 (9H, s, a,a',a''), 1.20–1.10 (2H, m, l), 0.88 (6H, dd, *J* = 15.9 Hz, 6.5 Hz, n,n').
^{13}C NMR (75 MHz, $CDCl_3$)	δ 172.12 (c), 163.99 (j), 147.88 and 147.69 (k), 141.88 and 141.78 (h), 139.46 (g), 109.27 and 108.78 (i), 80.43 (b), 43.98 (l), 39.42 (d), 36.55 (f), 34.65 (e), 28.12 (a,a',a''), 25.13 (m), 22.88 and 22.38 (n,n').
IR (neat)	2958, 2929, 1724 (C=O stretch), 1593, 1484, 1451, 1394, 1367 (C-O stretch), 1249, 1201 (C-N stretch), 1109, 1024, 824, 759 cm^{-1}.
HRMS (CI)	Calcd. for $C_{17}H_{27}FNO_2$ (M+H): 296.2026, found 296.2038 *m/z*.

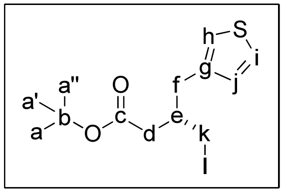
^{1}H NMR of (*S*)-3-(6-fluoropyridin-3-yl)methyl-5-methylhexanoic acid *tert*-butyl ester



^{13}C NMR of (*S*)-3-(6-fluoropyridin-3-yl)methyl-5-methylhexanoic acid *tert*-butyl ester



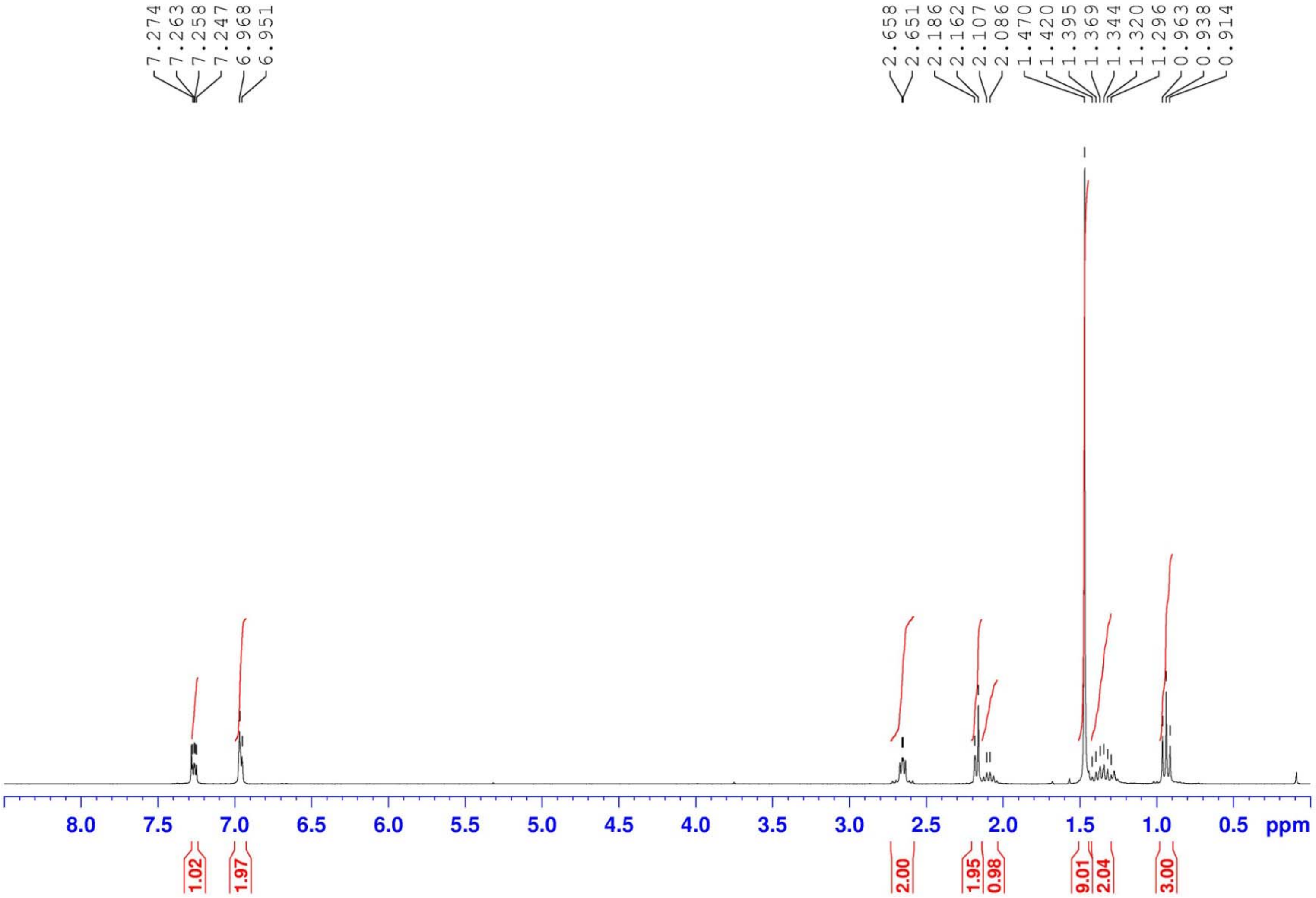




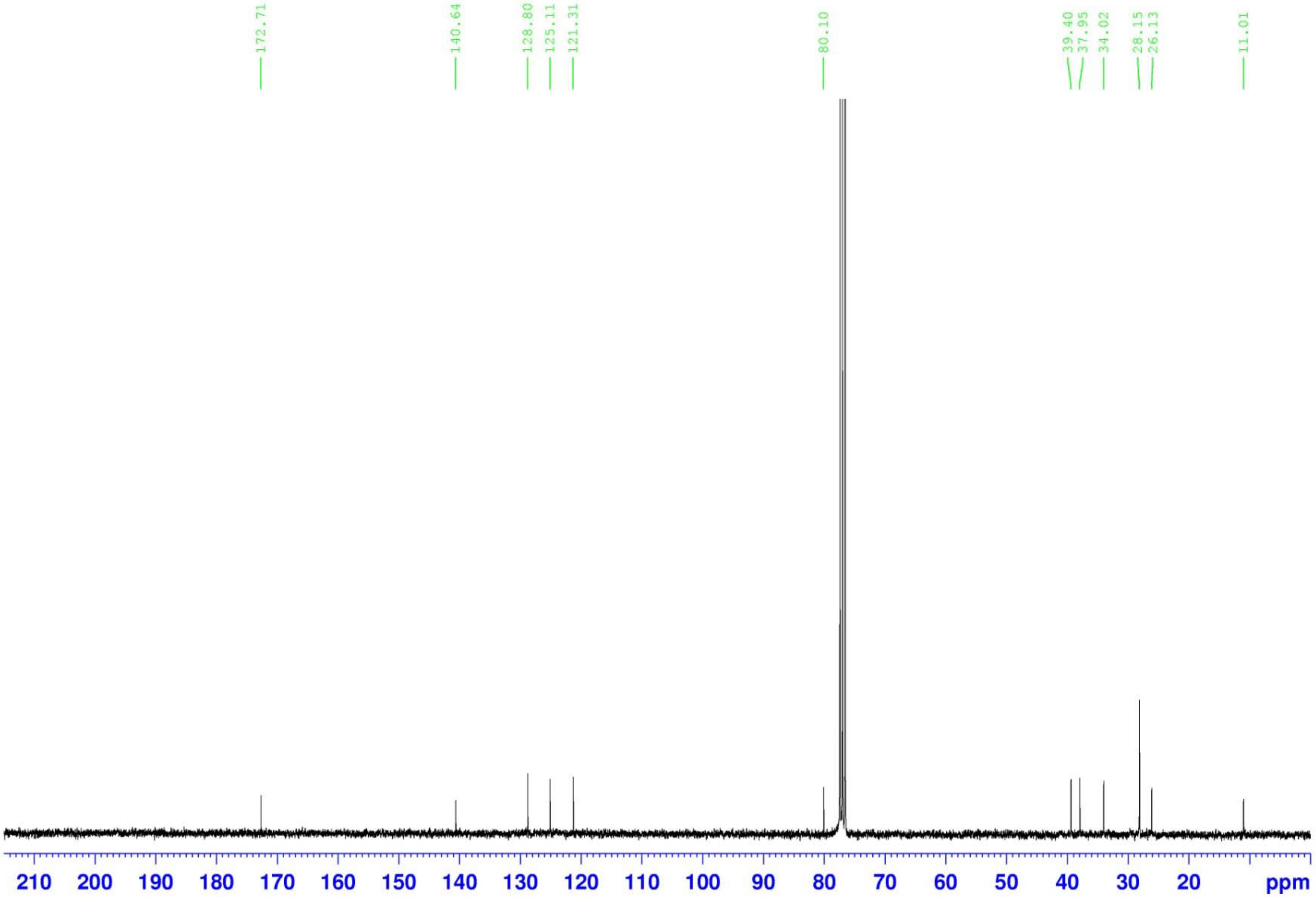
Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-**10h** affords, after flash chromatography on silica gel (98:2 hexanes:ethyl acetate), the title compound (80%) as a colorless oil.

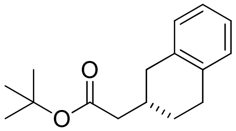
Optical rotation	$[\alpha]_D^{20} = +1.8^o$ (*c* 0.5, CHCl)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, CDCl$_3$)	δ 7.26 (1H, dd, *J* = 4.7 Hz, 3.2 Hz, i), 7.00–6.90 (2H, m, h,j), 2.75–2.55 (2H, m, f), 2.20–2.15 (2H, m, d), 2.15–2.00 (1H, m, e), 1.47 (9H, s, a,a',a''), 1.45–1.25 (2H, m, k), 0.94 (3H, t, *J* = 7.4 Hz, l).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 172.71 (c), 140.64 (g), 128.80 (j), 125.11 (i), 121.31 (h), 80.10 (b), 39.40 (d), 37.95 (e), 34.02 (f), 28.15 (a,a',a''), 26.13 (k), 11.01 (l).
IR (neat)	2964, 2929, 1726 (C=O stretch), 1459, 1366 (C-O stretch), 1255, 1144, 1090, 949, 850, 766, 693 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{14}H_{22}NaSO_2$ (M+Na): 277.1238, found 277.1245 *m/z*.

^{1}H NMR of (*S*)-3-(thiophen-3-yl)methylpentanoic acid *tert*-butyl ester

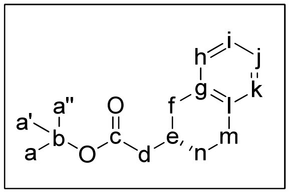


^{13}C NMR of (*S*)-3-(thiophen-3-yl)methylpentanoic acid *tert*-butyl ester





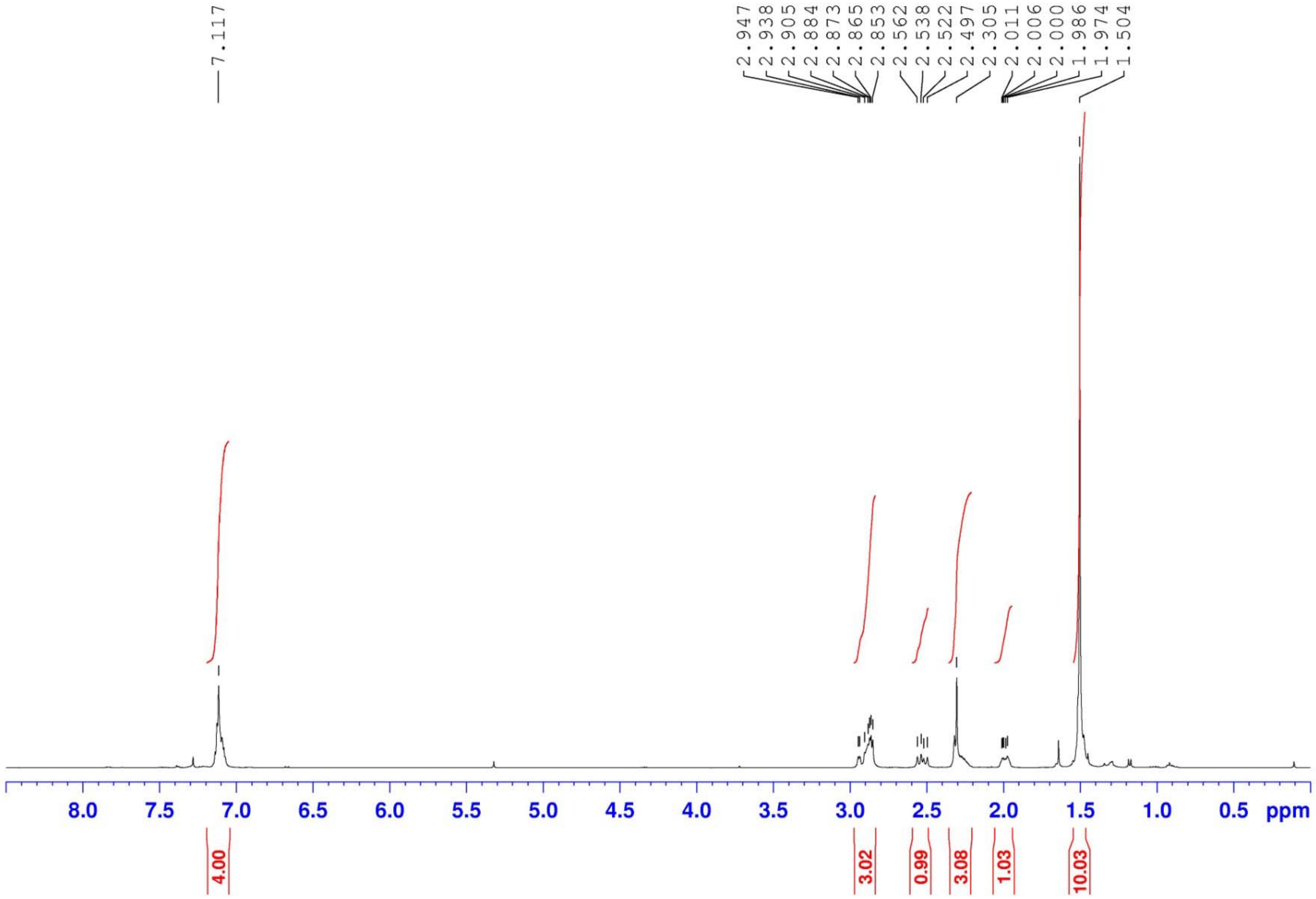
(*S*)-**14**



Following the general procedure for the palladium catalyzed C-C cross-coupling of trifluoroborate salts with (*R*)-5-(2-Chlorophenyl)-3-(trifluoroboratomethyl)pentanoic acid *tert*-butyl ester potassium salt affords, after flash chromatography on silica gel (97:3 hexanes:ethyl acetate), the title compound (91%) as a colorless oil.

Optical rotation	$[\alpha]_D^{20} = +1.7^{\circ}$ (c 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.00 (4H, m, h,i,j,k), 2.95–2.80 (3H, m, f,m), 2.53 (1H, dd, J = 16.4 Hz, 9.9 Hz, f), 2.35–2.20 (3H, m, d,e), 2.05–1.95 (1H, m, n), 1.55–1.45 (1H, m, n), 1.50 (9H, s, a,a',a'').
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.19 (c), 136.34 (l), 135.99 (g), 129.14 (h), 128.88 (k), 125.66 (i), 125.57 (j), 80.26 (b), 42.41 (d), 35.62 (f), 31.60 (e), 29.16 (n), 28.83 (m), 28.18 (a,a',a'').
IR (neat)	2977, 2925, 1728 (C=O stretch), 1495, 1453, 1366 (C-O stretch), 1289, 1255, 1146, 1064, 1039, 744 cm^{-1}.
HRMS (CI)	Calcd. for $C_{16}H_{23}O_2$ (M+H): 247.1698, found 247.1693 *m/z*.

^{1}H NMR of (*S*)-14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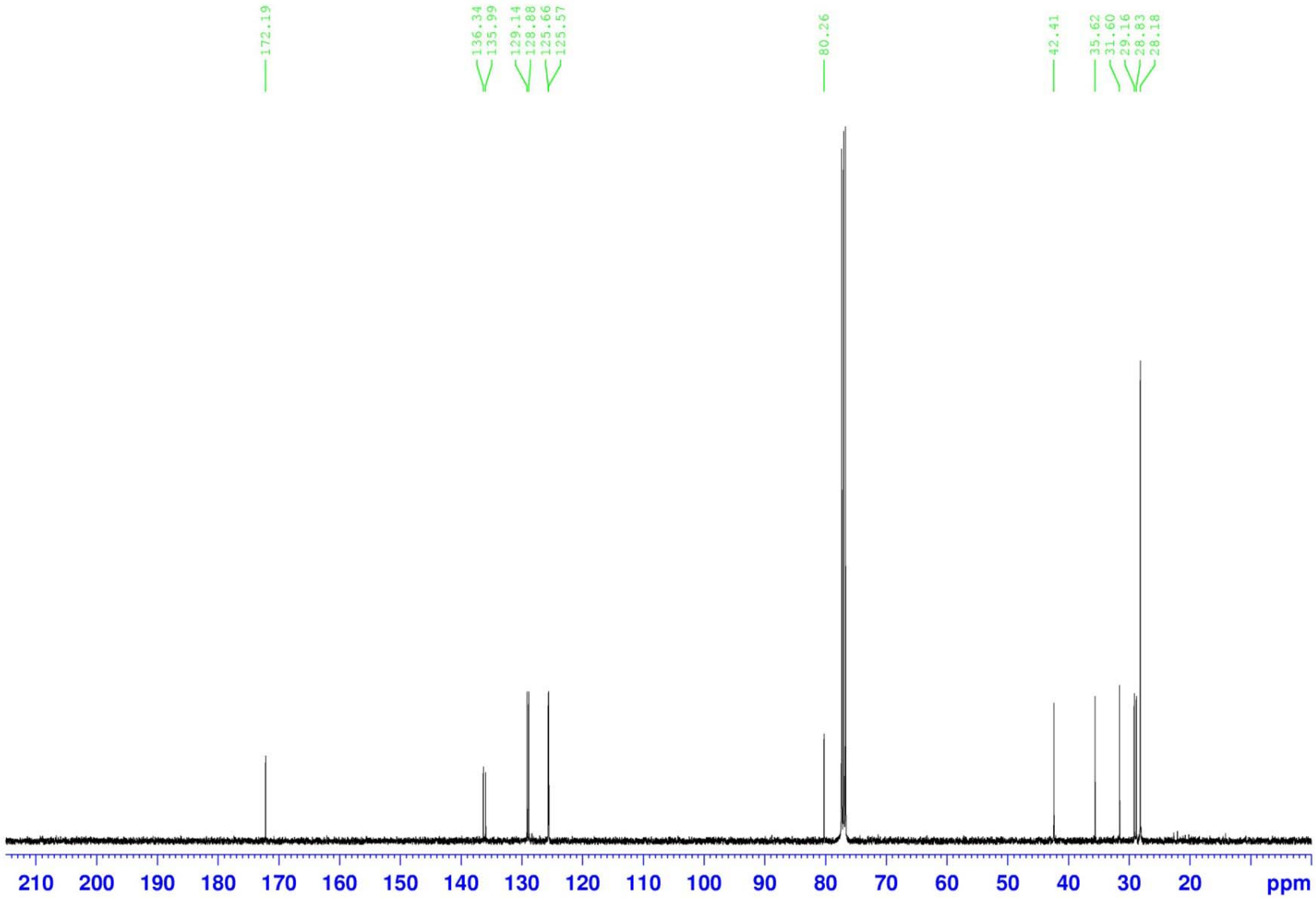
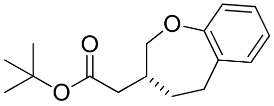
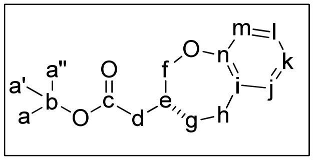
2.884
2.873
2.865
2.853
2.562
2.538
2.522
2.497
2.305
2.011
2.006
2.000
1.986
1.974
1.504
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.00
3.02
0.99
3.08
1.03
10.03

^{13}C NMR of (*S*)-14





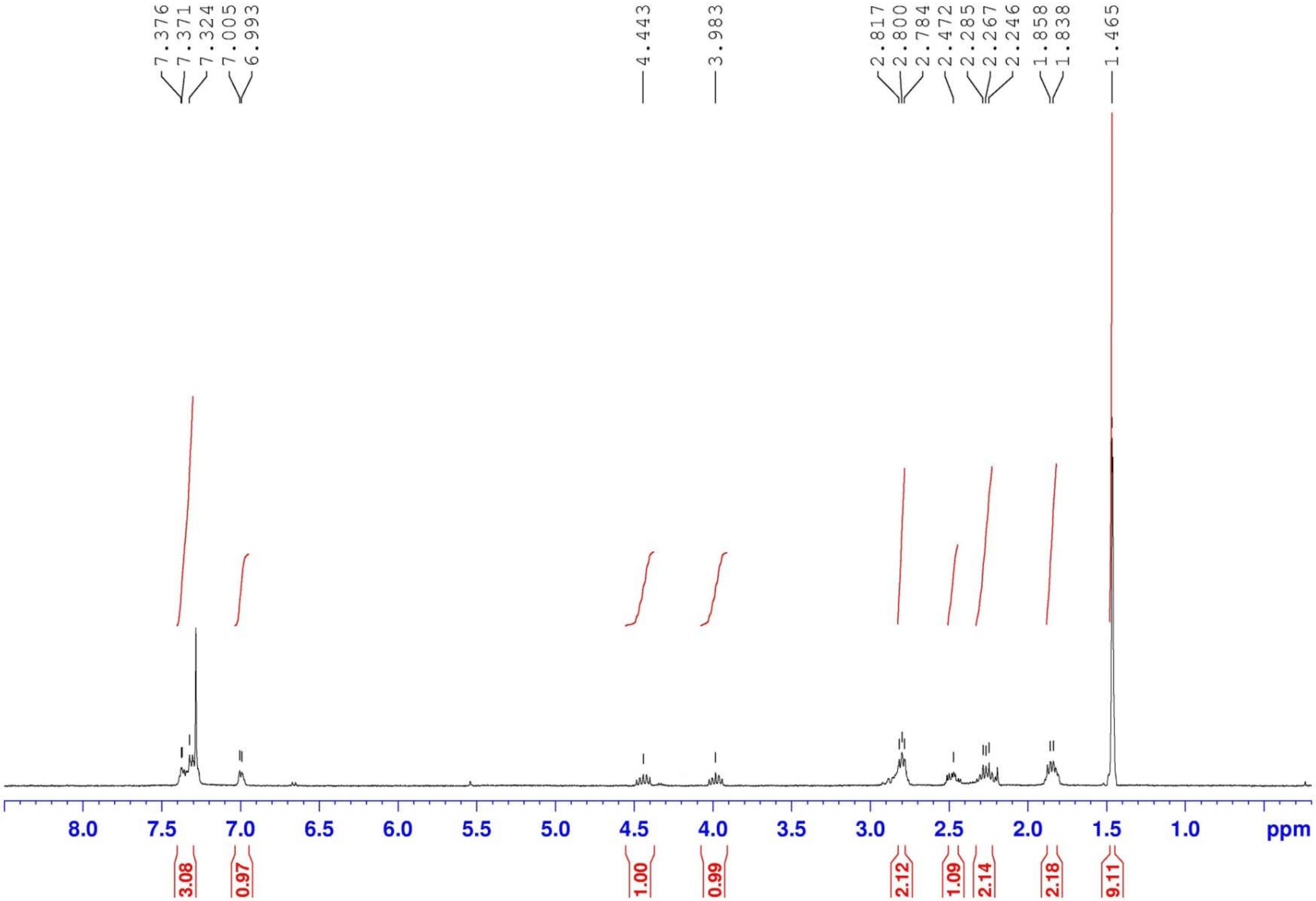
(*R*)-**16**



Following the general procedure for the palladium catalyzed C-O cross-coupling of (*R*)-**15** affords, after flash chromatography on silica gel (95:5 hexane:ethyl acetate), the title compound (75%) as a colorless oil.

Optical rotation	$[\alpha]_D^{20} = +3.3^o$ (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.6 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.40–7.25 (3H, m, m,k,j), 7.05–6.95 (1H, m, l), 4.50–4.40 (1H, m, f), 4.05–3.95 (1H, m, f), 2.85–2.75 (2H, m, h), 2.55–2.45 (1H, m, e), 2.20–2.30 (2H, m, d), 1.90–1.80 (2H, m, g), 1.47 (9H, s, a,a’,a”).
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.04 (c), 139.69 (i), 133.79 (j), 130.27 (k), 129.49 (l), 127.38 (m), 126.85 (n), 80.76 (b), 65.49 (f), 38.03 (d), 37.78 (e), 31.30 (h), 31.03 (g), 28.09 (a,a’,a”).
IR (neat)	2954, 1723 (C=O stretch), 1610, 1415, 1367 (C-O stretch), 1277, 1178, 1148, 1110, 1020, 760 cm^{-1}.
HRMS (CI)	Calcd. for $C_{16}H_{23}O_3$ (M+H): 263.1647, found 263.1652 *m/z*.

^{1}H NMR of (*R*)-16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7.324
7.005
6.993
4.443
3.983
2.817
2.800
2.784
2.472
2.285
2.267
2.246
1.858
1.838
1.465
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
ppm
3.08
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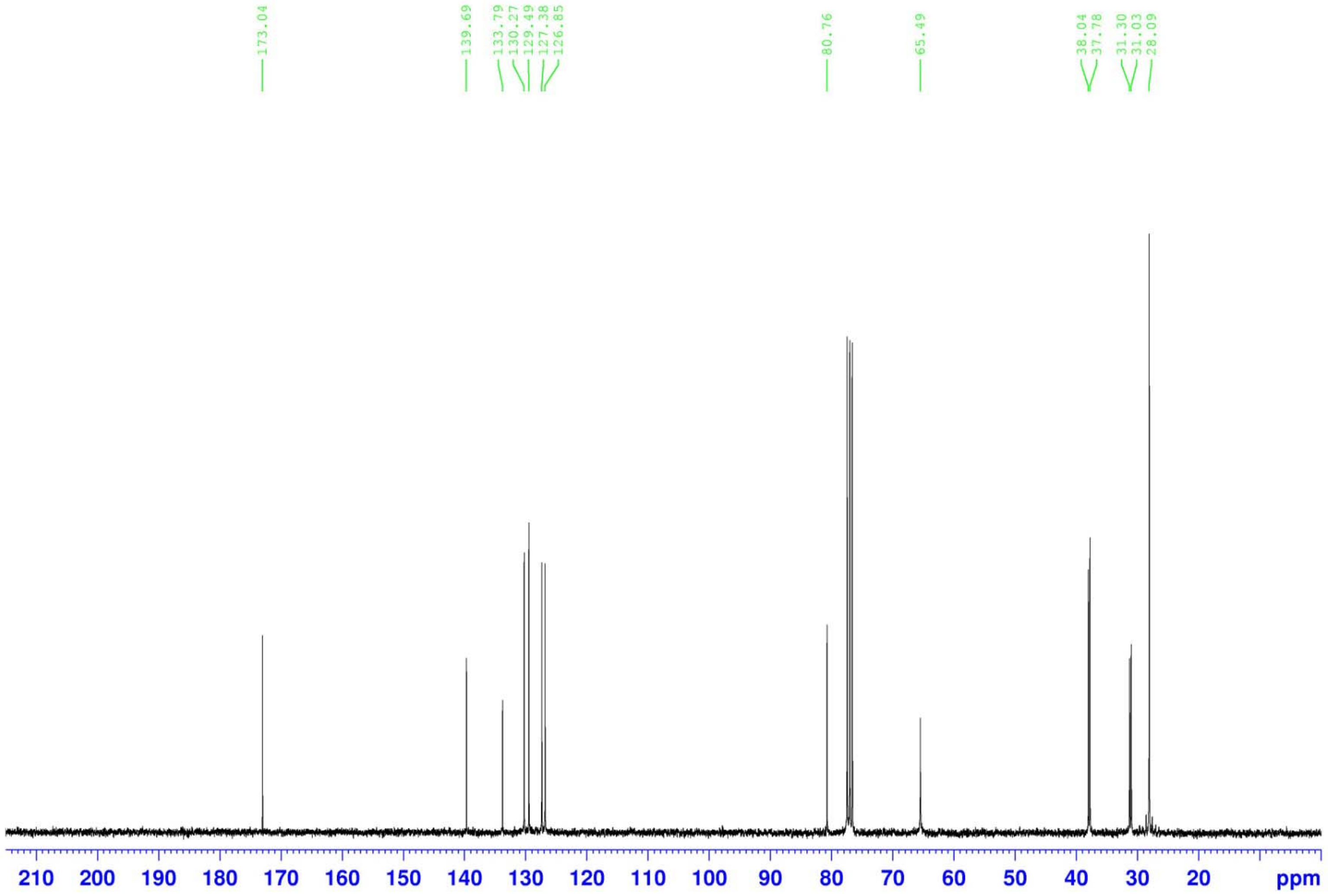
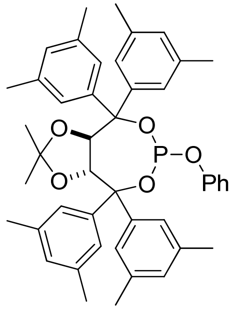
1.00
0.99
2.12
1.09
2.14
2.18
9.11

^{13}C NMR of (*R*)-16



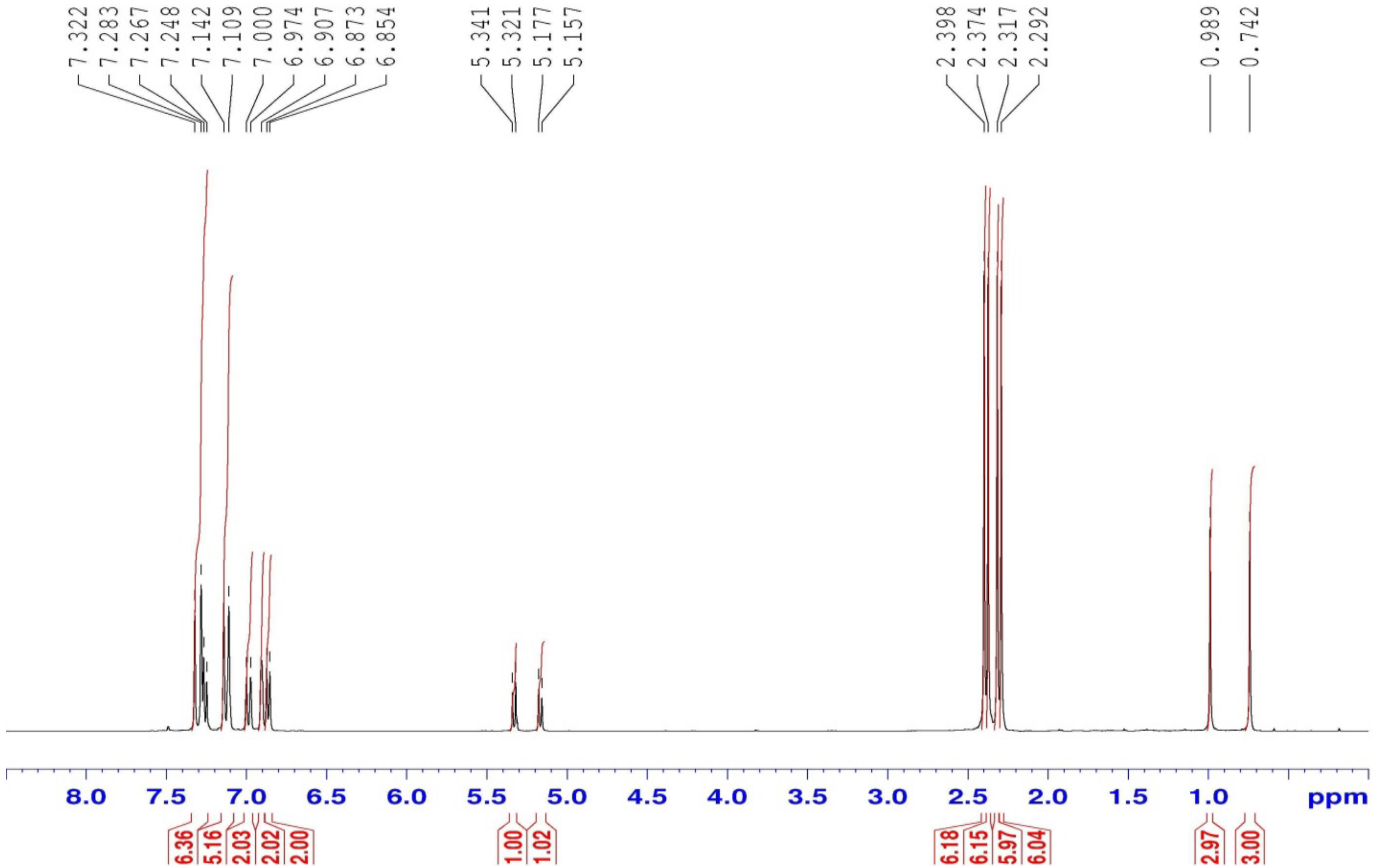


(*R*,*R*)-**6**

Following the procedure for the preparation of TADDOL-derived phosphite affords, after flash chromatography on silica gel (92:8 hexanes:ethyl acetate), the title compound (68%) as a white foamy solid.

m.p.	97–98 °C
Optical rotation	$[\alpha]_D^{20}$ = -120.0° (*c* 0.5, $CHCl_3$)
TLC analysis	R_f 0.80 (95:5 hexanes: ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.35-7.20 (6H, m), 7.15-7.05 (5H, m), 6.99 (2H, d, *J* = 10.5 Hz), 6.90 (2H, s), 6.86 (2H, d, *J* = 7.6 Hz), 5.33 (1H, d, *J* = 8.2 Hz), 5.17 (1H, d, *J* = 8.2 Hz), 2.40 (6H, s), 2.37 (6H, s), 2.32 (6H, s), 2.92 (6H, s), 0.99 (3H, s), 0.74 (3H, s).
^{13}C NMR (100 MHz, $CDCl_3$)	δ 152.16 (J_{CP} = 2.9 Hz), 146.10 (J_{CP} = 2.0 Hz), 145.83, 141.23 (3.0 Hz), 141.02, 137.37, 136.99, 136.50, 136.29, 129.45, 129.07, 128.94, 128.78, 126.89, 126.84, 125.10, 125.08, 123.33, 120.89, 120.81, 112.65, 85.51 (J_{CP} = 8.1 Hz), 84.64 (J_{CP} = 4.2 Hz), 82.34 (J_{CP} = 13.8 Hz), 81.28 (J_{CP} = 4.8 Hz), 26.95, 26.48, 21.69, 21.59, 21.48.
^{31}P NMR (162 MHz), $CDCl_3$)	δ 129.36.
IR (neat)	2916, 2863 (P-O ing), 1595, 1489, 1455, 1370, 1213 (C-O-C), 1159, 1035, 939, 853, 800, 761, 689 cm^{-1}.
HRMS (FAB)	Calcd. for $C_{45}H_{49}O_5P$ (M+H): 701.3396, found 701.3409 *m/z*.

^{1}H NMR of (*R*,*R*)-6


7.322
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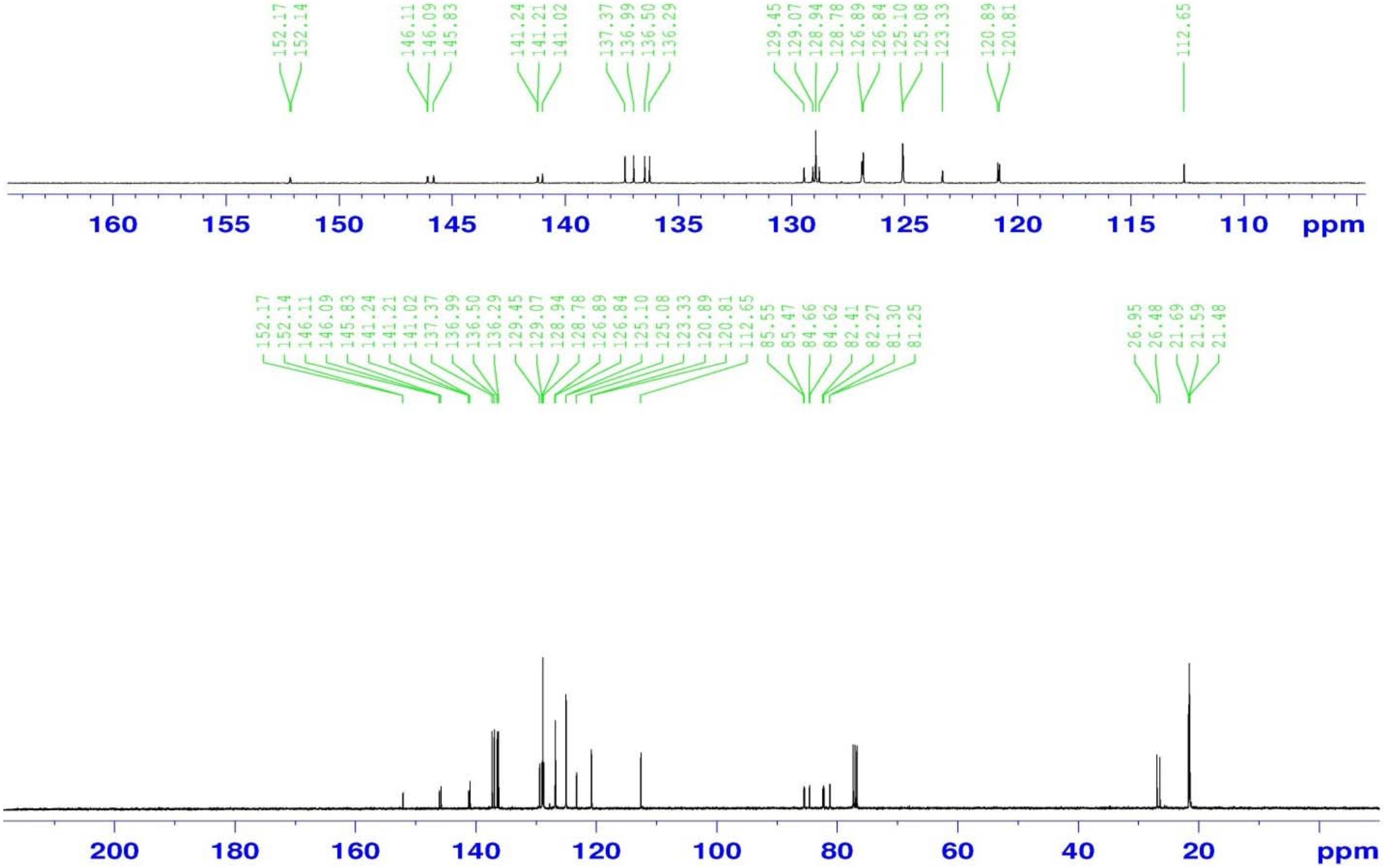
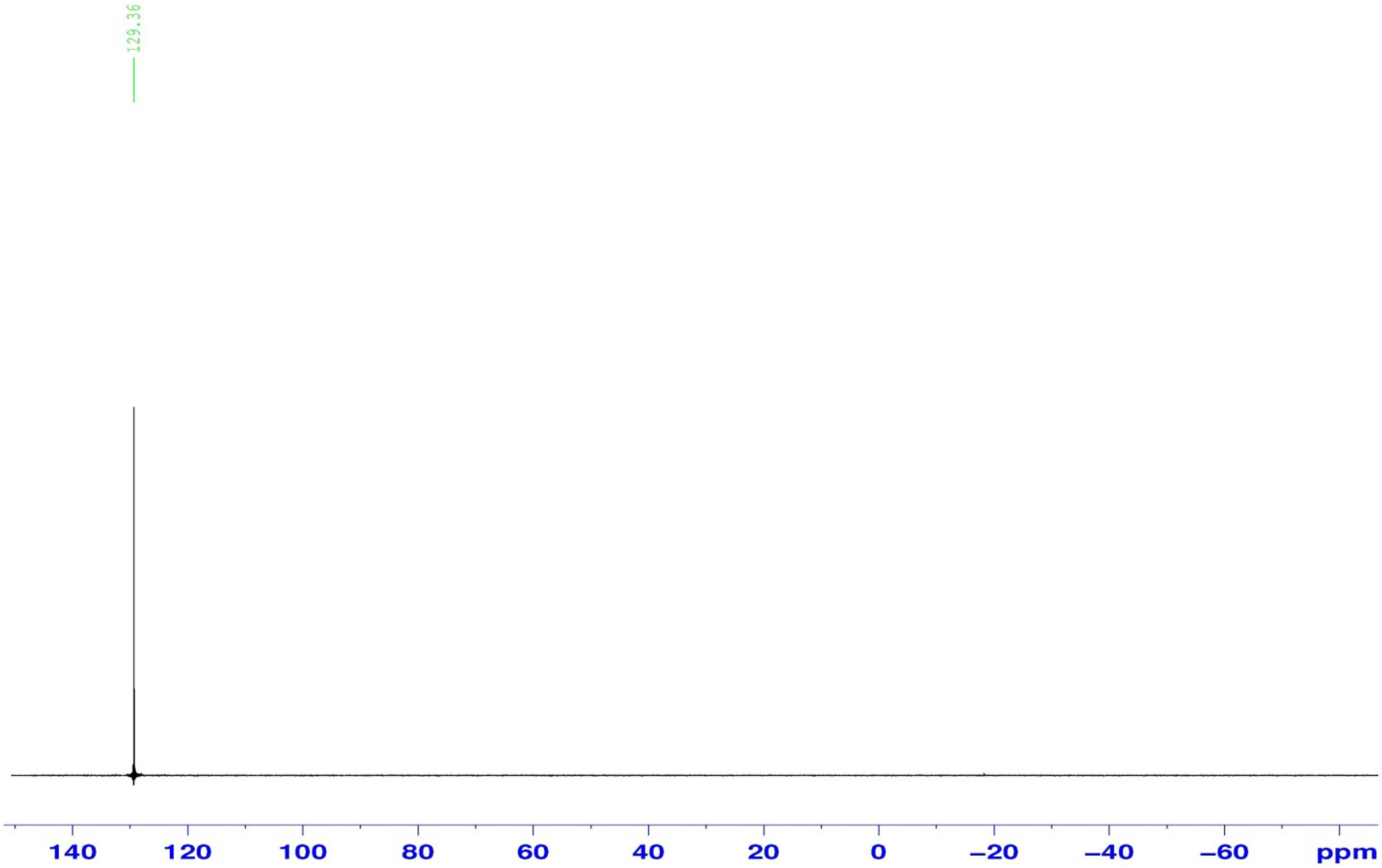
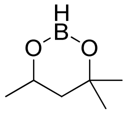
7.267
7.248
7.142
7.109
7.000
6.974
6.907
6.873
6.854
5.341
5.321
5.177
5.157
2.398
2.374
2.317
2.292
0.989
0.742
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
6.36
5.16
2.03
2.02
2.00
1.00
1.02
6.18
6.15
5.97
6.04
2.97
3.00

^{13}C NMR of (*R*,*R*)-6

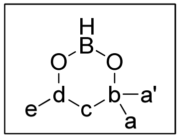


^{31}P NMR of (*R*,*R*)-6





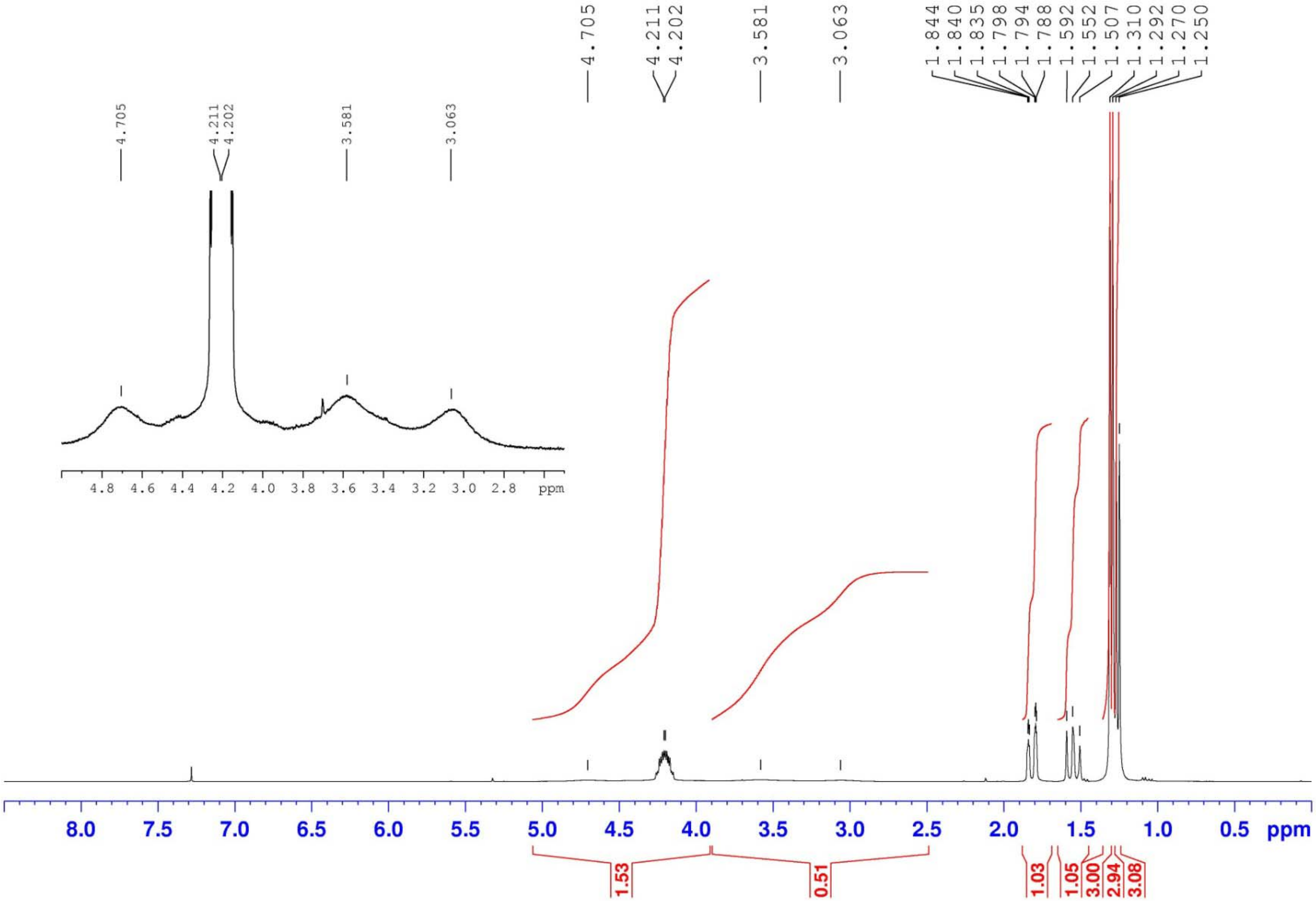
tmdBH (**5**)



Following the procedure for the preparation of tmdBH affords, after bulb-to-bulb distillation, the title compound (75%) as a colorless liquid.

b.p.	160–165 °C
^{1}H NMR (300 MHz, CDCl$_3$)	δ 4.30–4.15 (1H, m, d), 3.84 (1H, q, *J* = 155.6 Hz, BH), 1.90–1.75 (1H, m, c), 1.60–1.45 (1H, m, c), 1.31 (3H, s, a), 1.29 (3H, s, a'), 1.26 (3H, d, *J* = 6.2 Hz, e).
^{13}C NMR (75 MHz, CDCl$_3$)	δ 70.99 (b), 64.73 (d), 46.17 (c), 31.02 (a), 28.14 (a'), 22.93 (e).
^{11}B NMR (193 MHz, THF with residual CDCl$_3$)	δ 24.96 (d, *J* = 169.1 Hz).
IR (neat)	2976 (CH sp^3 stretch), 2879, 2400, 1495, 1427, 1384, 1291, 1156 (C-O stretch), 1094, 1024, 889, 789, 666 cm^{-1}.
HRMS (FAB)	Calcd. for $C_6H_{14}BO_2$ (M+H): 129.1087, found 129.1082 *m/z*.

^{1}H NMR of 5


4.705
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4.202
3.581
3.063
1.844
1.840
1.835
1.798
1.794
1.788
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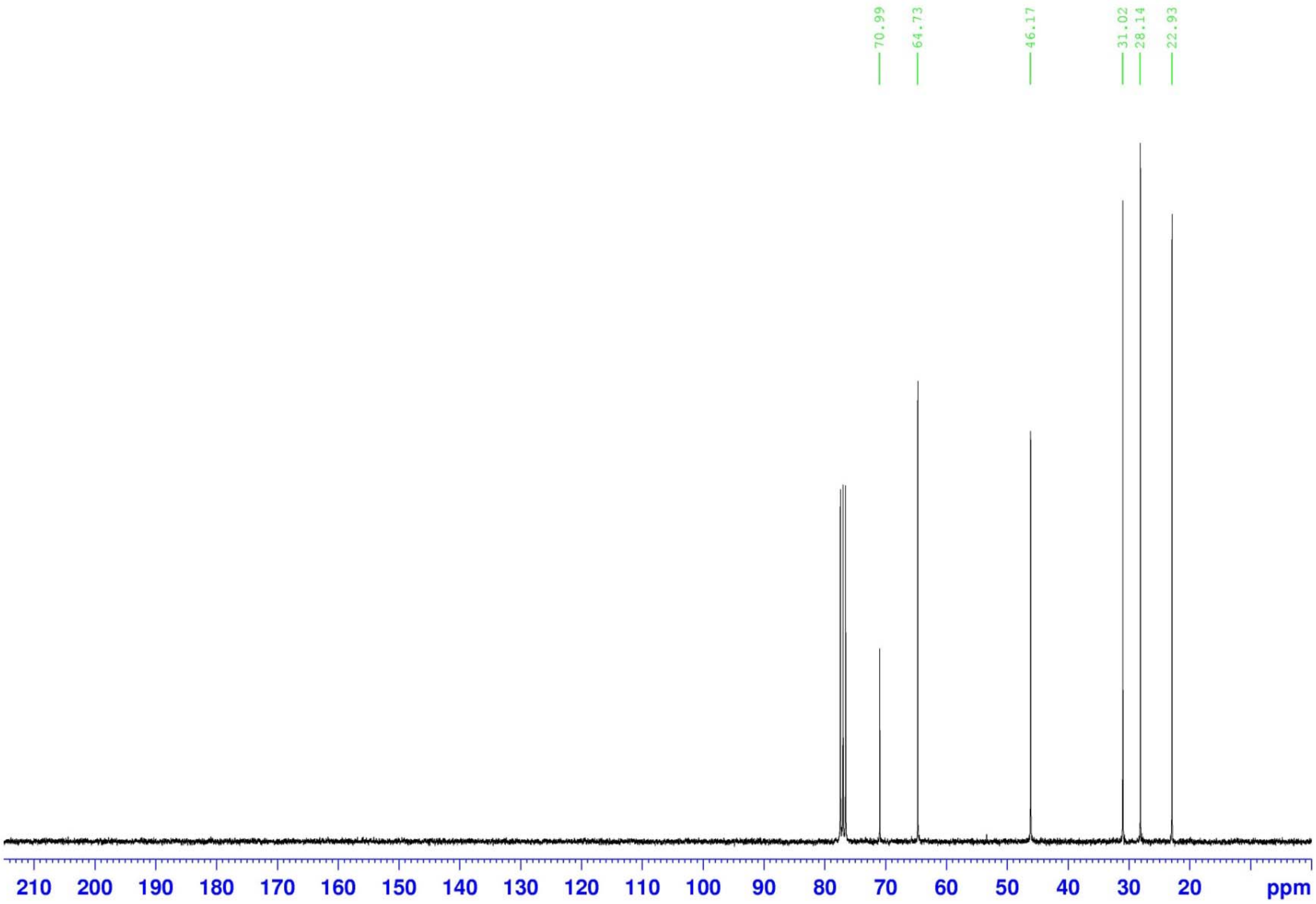
1.552
1.507
1.310
1.292
1.270
1.250
4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 ppm
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.53
0.51
1.03
1.05
3.00
2.94
3.08

^{13}C NMR of 5


70.99
64.73
46.17
31.02
28.14
22.93
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

^{11}B NMR of 5