Selective Conversion of Alcohols into Alkyl Iodides using a Thioiminium Salt

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Supporting Information

Contents

General experimental procedures

All reactions were carried out under argon with dry solvents. Dry THF, toluene and diethyl ether were obtained by passage through activated alumina columns under nitrogen. Reagents were used as obtained from commercial sources.

Alcohol substrates were obtained from commercial sources, with the exception of the following:

4-Benzyloxybutane-1,3-diol (entry 16, table 3) was prepared from butane-1,2,4-triol by selective benzylation;[1] 1-benzyloxy-4-(*tert*-butyldimethylsilanyloxy)butan-2-ol (entry 12, table 3) was prepared from this diol by selective silylation (TBSCl, imidazole, CH_2Cl_2).

1,4-Di(*tert*-butyldimethylsilanyloxy)butan-2-ol (entry 13, table 3) was prepared from butane-1,2,4-triol by selective disilylation (TBSCl, imidazole, CH_2Cl_2).

(*R*,*E*)-4,5-di(*tert*-butyldimethylsilanyloxy)pent-2-en-1-ol (entry 17, table 3) and (*R*,*Z*)-3-(2,2-dimethyl-1,3-dioxolan-4-yl)-prop-2-en-1-ol (entry 18, table 3) were prepared from 1,2:5,6-di-*O*-isopropylidene-D-mannitol by published procedures.[2]

Flash column chromatography was carried out on silica gel (Kieselgel 600) or neutral alumina (50-200 μm).

[1] Boons, G.J.; Castle, G.H.; Clase, J.A.; Grice, P.; Ley, S.V.; Pinel, C. *Synlett* **1993**, 913-914.

[2] Ellwood, A.R.; Mortimer, A.J.P.; Tocher, D.A.; Porter, M.J. *Synlett* **2008**, 2199-2203; Koenig, S.G.; Lowe, R.S.; Austin, D.J. *Synth. Commun.* **2002**, *32*, 1379-1383; Marshall, J.A.; Trometer, J.D.; Cleary, D.G. *Tetrahedron* **1989**, *45*, 391-402.

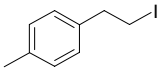
_N_,_N_-Dimethyl-_N_-(methylsulfanylmethylene)ammonium iodide (6)[3]

To a stirred solution of *N*,*N*-dimethylthioformamide (3.00 g, 33.7 mmol) in diethyl ether (70 mL) was added iodomethane (5.26 g, 37.1 mmol). The mixture was stirred at room temperature for 18 hours, and the solid product collected by filtration under argon. The solid was washed with cold diethyl ether (2 × 25 mL) and dried under vacuum to afford salt **6** (7.47 g, 96%) as a white solid which was used without further purification. mp 131-132 ºC; ν_{max}/cm^{-1} (neat) 2983, 1639, 1619, 1442, 1408; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 3.16 (3H, s), 3.40 (3H, s), 3.90 (3H, s), 11.14 (1H, s); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 16.6, 42.7, 48.9, 183.5; *m*/*z* (CI^+) 104 (M-I, 5%), 90 (100); HRMS found 104.0537, $C_4H_{10}NS$ requires 104.0534.

General Procedure for Conversion of Alcohols to Iodides

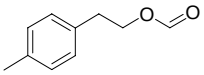
A 0.20-0.25 M solution of alcohol in toluene or THF was heated to 85 ºC (toluene) or reflux (THF). *N*,*N*-Dimethyl-*N*-(methylsulfanylmethylene)ammonium iodide (**6**, 1.5 equiv.) and imidazole (0.5 equiv.) were added and the mixture stirred at the specified temperature until TLC indicated complete conversion to iodide. After cooling to room temperature, the reaction mixture was concentrated *in vacuo* to afford the crude product. Final purification was carried out by flash column chromatography on silica (or, where indicated, neutral alumina) to give pure iodide.
Variations in concentration, temperature and quantities of reagents were as specified in table 3.

1-Iodo-2-(4-methylphenyl)ethane (11; entry 1, table 3)



Colorless oil; ν_{max}/cm^{-1} ($CHCl_3$ cast) 3019, 2920, 1513; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.35 (3H, s), 3.16 (2H, t, *J* 8.0 Hz), 3.36 (2H, t, *J* 8.0 Hz), 7.11 (2H, d, *J* 8.0 Hz), 7.16 (2H, d, *J* 8.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 6.0, 21.2, 40.1, 128.3, 129.4, 136.6, 137.7; *m*/*z* (EI) 246 (M^+, 23%), 149 (28), 128 (47), 127 (33), 120 (69), 119 (100); HRMS found 245.9897, $C_9H_{11}I$ requires 245.9900.

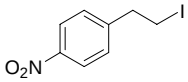
2-(4-Methylphenyl)ethyl formate (12)



Colorless oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 2924, 1719, 1516; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.37 (3H, s), 2.98 (2H, t, *J* 7.1 Hz), 4.41 (2H, t, *J* 7.1), 7.16 (4H, s), 8.07 (1H, s); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.1, 34.6, 64.6, 128.8, 129.4, 134.4, 136.4, 161.1; *m*/*z* (EI) 118 ($[M–HCO_2H]^+$, 100%); HRMS found 118.0786, C_9H_{10} requires 118.0783.

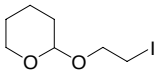
[3] Singh, H.; Batra, M.S.; Singh, P. *Ind. J. Chem. B* **1984**, *23*, 1176-1180; Berrée, F.; Malvaut, Y.; Marchand, E.; Morel, G. *J. Org. Chem.* **1993**, *58*, 6022-6029.

1-Iodo-2-(4-nitrophenyl)ethane (entry 2, table 3)[4]



White solid; mp 98-101 °C; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 3053, 1601, 1520, 1346; ^1H NMR ($CDCl_3$, 600 MHz) δ 3.31 (2H, t, *J* 7.3 Hz), 3.41 (2H, t, *J* 7.3 Hz), 7.39 (2H, m), 8.21 (2H, m); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 3.7, 39.5, 124.0, 129.4, 147.1, 147.7; *m*/*z* (EI) 277 (M^+, 59%), 150 (100); HRMS found 276.9592, $C_8H_8O_2NI^+$ requires 276.9594.

1-Iodo-2-(tetrahydropyran-2-yloxy)ethane (entry 3, table 3)[5]

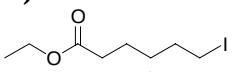


Yellow oil; ν_{max}/cm^{-1} (neat) 2940, 2870; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.51-1.67 (4H, m), 1.73 (1H, m), 1.84 (1H, m), 3.30 (2H, m), 3.52 (1H, m), 3.73 (1H, dt, *J* 11.1, 6.8 Hz), 3.91 (1H, m), 3.95 (1H, ddd, *J* 11.1, 7.2, 6.5 Hz) 4.68 (1H, t, *J* 3.4 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 3.6, 19.3, 25.3, 30.5, 62.3, 68.3, 98.8; *m*/*z* (EI) 256 (M^+, 43%), 155 (100); HRMS found 255.9966, $C_7H_{13}O_2I$ requires 255.9955.

1-*tert*-Butyldimethylsilanyloxy-5-iodopentane (entry 4, table 3)[6]

Colorless oil; ν_{max}/cm^{-1} (neat) 2928, 2856; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.05 (6H, s), 0.91 (9H, s), 1.41-1.50 (2H, m), 1.50-1.59 (2H, m), 1.86 (2H, tt, *J* 7.3, 7.0 Hz), 3.21 (2H, t, *J* 7.0 Hz), 3.62 (2H, t, *J* 6.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ –5.3, 7.1, 18.4, 26.0, 27.0, 31.8, 33.4, 62.9; *m*/*z* (CI^+) 329 (MH^+, 5%), 271 (27), 201 (49), 197 (60), 177 (30), 146 (100); HRMS found 329.0793, $C_{11}H_{26}OSiI$ requires 329.0798.

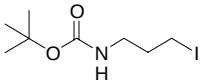
Ethyl 6-iodohexanoate (entry 5, table 3)[7]



Colorless oil; ν_{max}/cm^{-1} (neat) 2935, 2864, 1730; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.27 (3H, t, *J* 7.1 Hz), 1.45 (2H, m), 1.66 (2H, m), 1.85 (2H, quin, *J* 7.3 Hz), 2.32 (2H, t, *J* 7.6 Hz), 3.20 (2H, t, *J* 7.0 Hz), 4.14 (2H, q, *J* 7.1 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 6.6, 14.3, 23.9, 30.0, 33.2, 34.1, 60.4, 173.5; *m*/*z* (CI^+) 271 (MH^+, 37%), 243 (22), 143 (73), 131 (100); HRMS found 271.0197, $C_8H_{16}O_2I$ requires 271.0195.

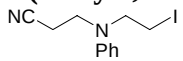
[4] Keeffe, J.R.; Jencks, W.P. *J. Am. Chem. Soc.* **1983**, *105*, 265-279.
[5] Anantanarayan, A.; Dutton, P.J.; Fyles, T.M.; Pitre, M.J. *J. Org. Chem.* **1986**, *51*, 752-755.
[6] Mash, E.A.; Hemperly, S.B.; Nelson, K.A.; Heidt, P.C.; van Deusen, S. *J. Org. Chem.* **1990**, *55*, 2045-2055.
[7] Toshima, H.; Nara, S.; Aramaki, H.; Ichihara, A.; Koda, Y.; Kikuta, Y. *Biosci. Biotech. Biochem.* **1997**, *61*, 1724-1728.

***N*-*tert*-Butoxycarbonyl-3-iodopropylamine (entry 6, table 3)**[8]



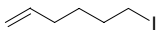
Yellow oil; ν_{max}/cm^{-1} ($CHCl_3$ cast) 3341 br, 2976, 2931, 1683, 1512; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.42 (9H, s), 1.99 (2H, quin, *J* 6.7 Hz), 3.16-3.21 (4H, m), 4.74 (1H, br s); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 3.2, 28.5, 33.5, 41.1, 79.4, 156.0; *m*/*z* (FAB^+) 308 (MNa^+, 49%), 286 (50), 230 (100); HRMS found 308.0118, $C_8H_{16}O_2NINa$ requires 308.0123.

***N*-(2-Cyanoethyl)-*N*-(2-iodoethyl)aniline (entry 7, table 3)**



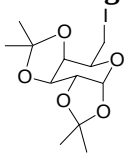
Pale yellow solid; mp 59-62 °C; ν_{max}/cm^{-1} ($CHCl_3$ cast) 3025, 2955, 2247, 1596, 1502; ^1H NMR ($CDCl_3$, 400 MHz) δ 2.64 (2H, t, *J* 6.8 Hz), 3.27 (2H, t, *J* 8.1 Hz), 3.76 (2H, t, *J* 6.8 Hz), 3.80 (2H, t, *J* 8.1 Hz), 6.70 (2H, d, *J* 8.5 Hz), 6.85 (1H, m), 7.30 (2H, m); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 1.7, 16.4, 47.5, 54.6, 112.7, 118.2, 118.6, 130.0, 145.2; *m*/*z* (EI) 300 (M^+, 62%), 260 (86), 173 (23), 159 (100); HRMS found 300.0122, $C_{11}H_{13}N_2I$ requires 300.0118.

6-Iodohex-1-ene (entry 8, table 3)[9]



Colorless oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 3076, 2930, 2855, 1641; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.51 (2H, m), 1.83 (2H, m), 2.09 (2H, m), 3.21 (2H, t, *J* 7.1 Hz), 4.96-5.06 (2H, m), 5.80 (1H, ddt, *J* 17.1, 10.3, 6.7 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 6.9, 29.8, 32.7, 32.9, 115.1, 138.2; *m*/*z* (EI) 210 (M^+, 13%), 178 (100); HRMS found 209.9894, $C_6H_{11}I$ requires 209.9900.

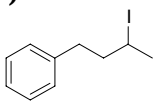
6-Deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (entry 9, table 3)



Off-white solid; mp 59-60 ºC; $[\alpha]_D^{20}$ –53.2 (*c* 0.9, $CHCl_3$); ν_{max}/cm^{-1} (CH_2Cl_2 cast) 2988, 2935, 1067; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.34 (3H, s), 1.36 (3H, s), 1.45 (3H, s) 1.55 (3H, s), 3.20 (1H, dd, *J* 10.1, 7.2 Hz), 3.32 (1H, dd, *J* 10.1, 6.9 Hz), 3.95 (1H, td, *J* 7.0, 1.8 Hz), 4.30 (1H, dd, *J* 5.1, 2.5 Hz), 4.41 (1H, dd, *J* 7.9, 1.8 Hz), 4.61 (1H, dd, *J* 7.9, 2.5 Hz), 5.55 (1H, d, *J* 5.1 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 2.4, 24.5, 25.0, 26.0, 26.1, 69.0, 70.6, 71.2, 71.7, 96.8, 108.9, 109.6; *m*/*z* (CI^+) 371 (MH^+, 74%), 355 (96), 313 (100); HRMS found 371.0352, $C_{12}H_{20}IO_5$ requires 371.0355.

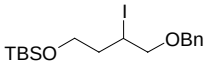
[8] Ensch, C.; Hesse, M. *Helv. Chim. Acta* **2002**, *85*, 1659-1673.
[9] Bailey, W.F.; Gagnier, R.P.; Patricia, J.J. *J. Org. Chem.* **1984**, *49*, 2098-2107.

3-Iodo-1-phenylbutane (entry 11, table 3)[10]



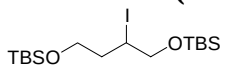
Pale yellow oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 3026, 2915, 1602, 1495, 1452; ^1H NMR ($CDCl_3$, 600 MHz) δ 1.86-1.94 (1H, dddd, *J* 14.5, 9.0, 7.0, 4.5 Hz), 1.97 (3H, d, *J* 6.8 Hz), 2.18 (1H, dtd, *J* 14.5, 9.0, 5.1 Hz), 2.72 (1H, ddd, *J* 13.7, 9.0, 7.1 Hz), 2.88 (1H, ddd, *J* 13.7, 9.0, 5.1 Hz), 4.14 (1H, dqd, *J* 9.0, 6.8, 4.5 Hz), 7.21-7.26 (3H, m), 7.29-7.34 (2H, m); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 29.1, 29.7, 35.9, 44.5, 126.2, 128.6, 128.6, 140.8; *m*/*z* (EI) 260 (MH^+, 15%), 133 (50), 91 (100); HRMS found 260.0060, $C_{10}H_{13}I$ requires 260.0057.

1-Benzyloxy-4-(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 12, table 3)



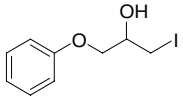
Colorless oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 2953, 2928, 2856; ^1H NMR ($CDCl_3$, 600 MHz) δ 0.06 (3H, s), 0.09 (3H, s), 0.91 (9H, s), 1.95 (1H, ddt, *J* 14.6, 9.9, 4.5 Hz), 2.07 (1H, dddd, *J* 14.6, 8.6, 5.8, 3.9 Hz), 3.72 (1H, ddd, *J* 10.3, 8.6, 4.7 Hz), 3.73 (1H, dd, *J* 10.5, 6.5 Hz), 3.78 (1H, dd, *J* 10.5, 6.0 Hz), 3.81 (1H, ddd, *J* 10.3, 5.8, 4.3 Hz), 4.41 (1H, dtd, *J* 9.9, 6.2, 3.9 Hz), 4.61 (2H, s), 7.30-7.39 (5H, m); ^{13}C NMR ($CDCl_3$, 150 MHz) δ –5.2, 18.4, 26.0, 30.5, 39.4, 62.2, 72.9, 75.9, 127.7, 127.8, 128.5, 137.9; *m*/*z* (CI^+) 421 (MH^+, 6%), 313 (24), 187 (19), 145 (100); HRMS found 421.1066, $C_{17}H_{30}IO_2Si$ requires 421.1060.

1,4-Di(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 13, table 3)



Purified on alumina; colorless oil; ν_{max}/cm^{-1} ($CDCl_3$ cast) 2954, 2929, 2857; ^1H NMR ($CDCl_3$, 600 MHz) δ 0.08 (3H, s), 0.09 (6H, s), 0.10 (3H, s), 0.91 (9H, s), 0.92 (9H, s), 1.87 (1H, dddd, *J* 14.6, 10.2, 4.9, 4.1 Hz), 2.10 (1H, dddd, *J* 14.6, 8.6, 6.0, 3.6 Hz), 3.71 (1H, ddd, *J* 10.3, 8.6, 4.9 Hz), 3.80 (1H, dd, *J* 10.8, 7.3 Hz), 3.82 (1H, ddd, *J* 10.3, 6.0, 4.1 Hz), 3.91 (1H, dd, *J* 10.8, 5.4 Hz), 4.26 (1H, dddd, *J* 10.2, 7.3, 5.4, 3.6 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ –5.3, –5.3, –5.2, 18.3, 18.3, 25.9, 25.9, 34.5, 39.0, 62.4, 69.2; *m*/*z* (CI^+) 445 (MH^+, 100%), 313 (20); HRMS found 445.1452, $C_{16}H_{38}IO_2Si_2$ requires 445.1450.

1-Iodo-3-phenoxypropan-2-ol (entry 15, table 3)[11]



Colorless oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 3390 br, 3038, 2926, 1598, 1597, 1493; ^1H NMR ($CDCl_3$, 400 MHz) δ 2.48 (1H, br s), 3.41 (1H, dd, *J* 10.3, 5.7 Hz), 3.49 (1H, dd, *J* 10.3, 5.3 Hz), 4.01 (1H, qd, *J* 5.6, 4.5 Hz), 4.06 (1H, dd, *J* 9.4, 4.5 Hz), 4.11 (1H, dd, *J* 9.4, 5.7 Hz), 6.93 (2H, m), 6.99 (1H, m), 7.32 (2H, m); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 9.3, 69.7, 70.4, 114.7, 121.6, 129.7, 158.2; *m*/*z* (EI) 278 (M^+, 98%), 133 (34), 107 (99), 94 (100); HRMS found 277.9788, $C_9H_{11}IO_2$ requires 277.9798.

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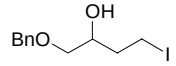
[10] Keinan, E.; Perez, D.; Sahai, M.; Shvily, R. *J. Org. Chem.* **1990**, *55*, 2927-2938; Onishi, Y.; Ogawa, D.; Yasuda, M.; Baba, A. *J. Am. Chem. Soc.* **2002**, *124*, 13690-13691.

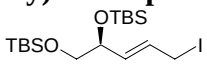
[11] Sharghi, H.; Massah, A. R.; Eshghi, H.; Niknam, K. *J. Org. Chem.* **1998**, *63*, 1455-1461.

1-Benzyloxy-4-iodobutan-2-ol (entry 16, table 3)



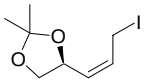
Colorless oil; ν_{max}/cm^{-1} (CH_2Cl_2 cast) 3425 br, 3030, 2859, 1496, 1453; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.88-2.04 (2H, m), 2.40 (1H, br s), 3.30-3.35 (2H, m), 3.39 (1H, dd, *J* 9.4, 7.2 Hz), 3.53 (1H, dd, *J* 9.4, 3.2 Hz), 3.95 (1H, m), 4.57 (2H, s), 7.30-7.41 (5H, m); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 2.4, 37.0, 70.4, 73.5, 73.8, 127.9, 128.0, 128.6, 137.8; *m*/*z* (EI) 306 (M^+, 17%), 91 (100); HRMS found 306.0120, $C_{11}H_{15}O_2I$ requires 306.0111.

(*R*,*E*)-4,5-Di(*tert*-butyldimethylsilanyloxy)-1-iodopent-2-ene (entry 17, table 3)



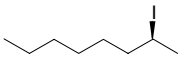
Colorless oil; $[\alpha]_D^{21}$ = –17.0 (*c* 0.70, $CHCl_3$); ν_{max}/cm^{-1} (CH_2Cl_2 cast) 2954, 2928, 2857; ^1H NMR ($CDCl_3$, 600 MHz) δ 0.06 (6H, s), 0.08 (6H, s), 0.90 (9H, s), 0.91 (9H, s), 3.43 (1H, dd, *J* 10.0, 6.2 Hz), 3.54 (1H, dd, *J* 10.0, 6.3 Hz), 3.89 (1H, dddd, *J* 10.6, 8.2, 1.0, 0.6 Hz), 3.92 (1H, dddd, *J* 10.6, 8.0, 1.0, 0.6 Hz), 4.16 (1H, m), 5.74 (1H, ddt, *J* 15.1, 5.4, 1.0 Hz), 5.96 (1H, dtd, *J* 15.1, 8.1, 1.4 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ –5.3, –5.2, –4.6, –4.5, 5.5, 18.3, 18.5, 26.0, 26.0, 67.7, 73.1, 128.4, 134.7; *m*/*z* (EI) 399 (M–^{t}Bu, 30%), 184 (24), 147 (100); HRMS found 399.0671, $C_{13}H_{28}O_2Si_2I$ requires 399.0667.

(*R*,*Z*)-1-(2,2-Dimethyl-1,3-dioxolan-4-yl)-3-iodoprop-1-ene (entry 18, table 3)



Colorless oil; $[\alpha]_D^{20}$ = –163.2 (*c* 0.50, $CHCl_3$); ν_{max}/cm^{-1} (film) 2985, 2932, 2868; ^1H NMR ($CDCl_3$, 600 MHz) δ 1.43 (3H, s), 1.45 (3H, s), 3.60 (1H, t, *J* 7.9 Hz), 3.88 (1H, dd, *J* 9.8, 8.1 Hz), 4.04 (1H, td, *J* 9.8, 0.9 Hz), 4.21 (1H, dd, *J* 8.3, 6.2 Hz), 4.91 (1H, m), 5.47 (1H, dd, *J* 10.6, 8.4 Hz), 5.97 (1H, dddd, *J* 10.8, 9.6, 8.2, 1.3 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ –1.7, 25.9, 26.7, 68.5, 71.0, 109.6, 130.7, 130.9; *m*/*z* (CI^+) 286 (MNH_4^+, 15%), 269 (MH^+, 52), 228 (100), 209 (32); HRMS found 269.0033, $C_8H_{14}O_2I$ requires 269.0033.

(*S*)-2-Iodooctane (14)

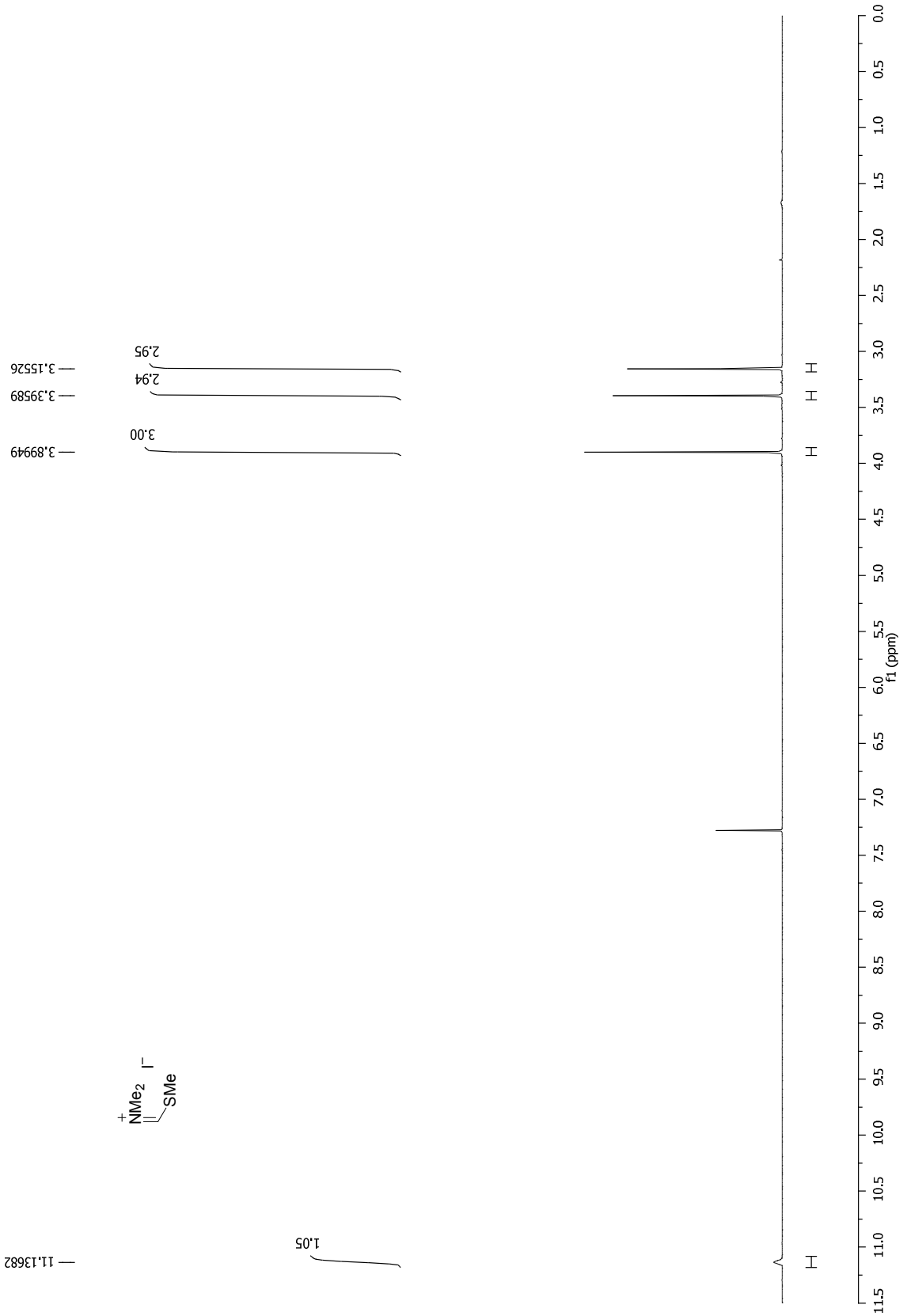


Colorless oil; $[\alpha]_D^{24}$ = +4.8 (*c* 3.7, $CHCl_3$) for a sample with 11% ee (by chiral GC) [lit.[12] $[\alpha]_D^{20}$ = +50.6 (*c* 6.4, $CHCl_3$)]; ν_{max}/cm^{-1} ($CHCl_3$ cast) 2956, 2924, 2854; ^1H NMR ($CDCl_3$, 600 MHz) δ 0.90 (3H, t, *J* 7.1 Hz), 1.26-1.53 (8H, m), 1.62 (1H, dddd, *J* 14.3, 10.4, 9.9, 5.1 Hz), 1.86 (1H, dddd, *J* 14.3, 9.9, 8.4, 4.7 Hz), 1.94 (3H, d, *J* 6.8 Hz), 4.20 (1H, dqd, *J* 8.4, 6.8, 5.1 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 14.1, 22.6, 28.4, 29.0, 29.7, 31.0, 31.7, 42.9; *m*/*z* (EI) 240 (M^+, 15%), 219 (37), 169 (28), 155 (45), 128 (100); HRMS found 240.0376, $C_8H_{17}I$ (M^+) requires 240.0370. Chiral GC analysis was carried out using a Supelco Beta-Dex™ 120 column (30 m × 0.25 mm i.d.); carrier gas He, flow rate 100 mL min^{-1}, oven temperature 80 ºC (20 mins) then increasing at 2 ºC per minute; retention times 39.4 min (*S*-isomer), 39.7 min (*R*-isomer).

[12] Hebert, E.; Maigrot, N.; Welvart, Z. *Tetrahedron Lett.* **1983**, *24*, 4683-4686.

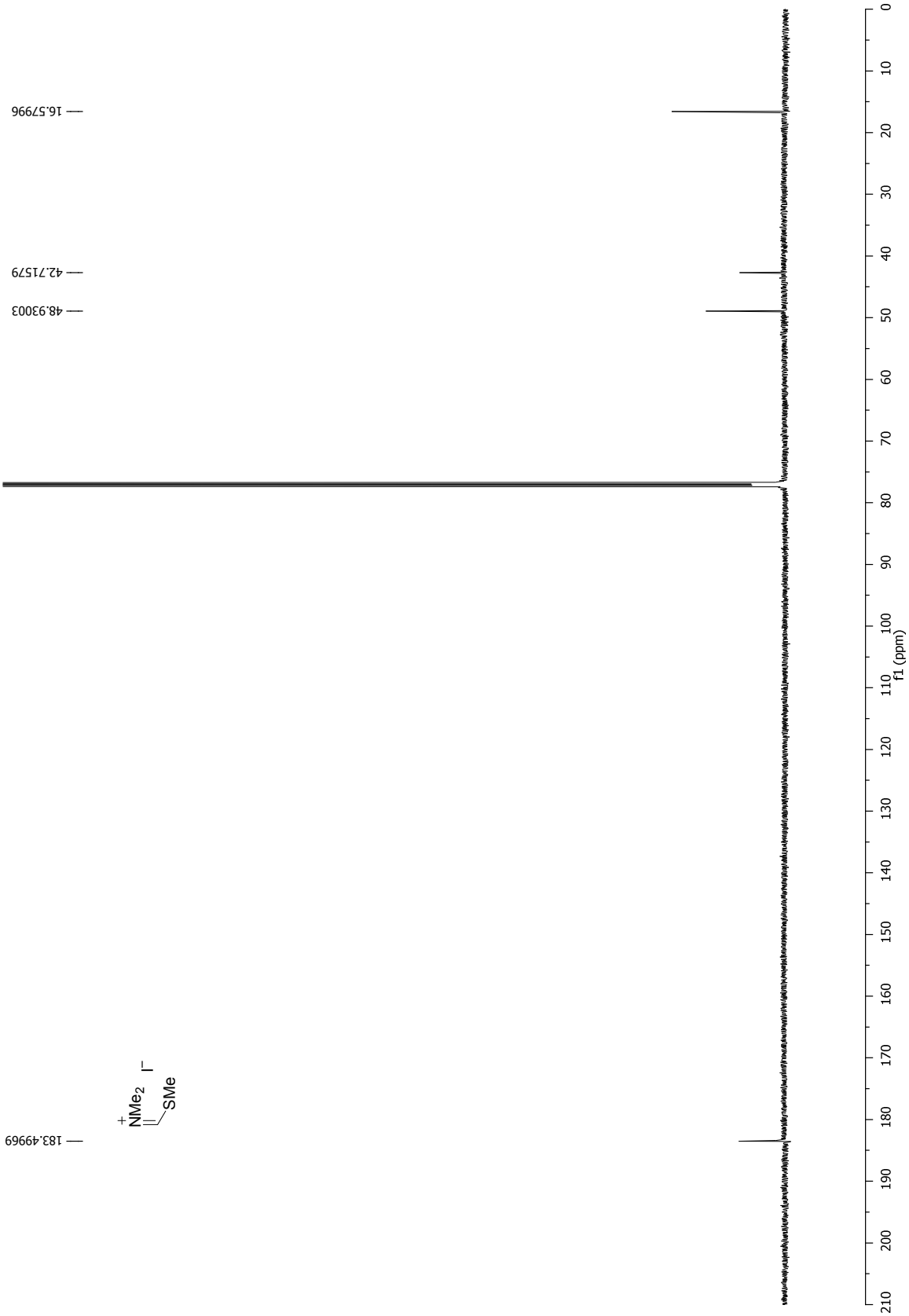
_N_,_N_-Dimethyl-_N_-(methylsulfanylmethylene)ammonium iodide (6)

^{1}H NMR, 600 MHz, $CDCl_3$



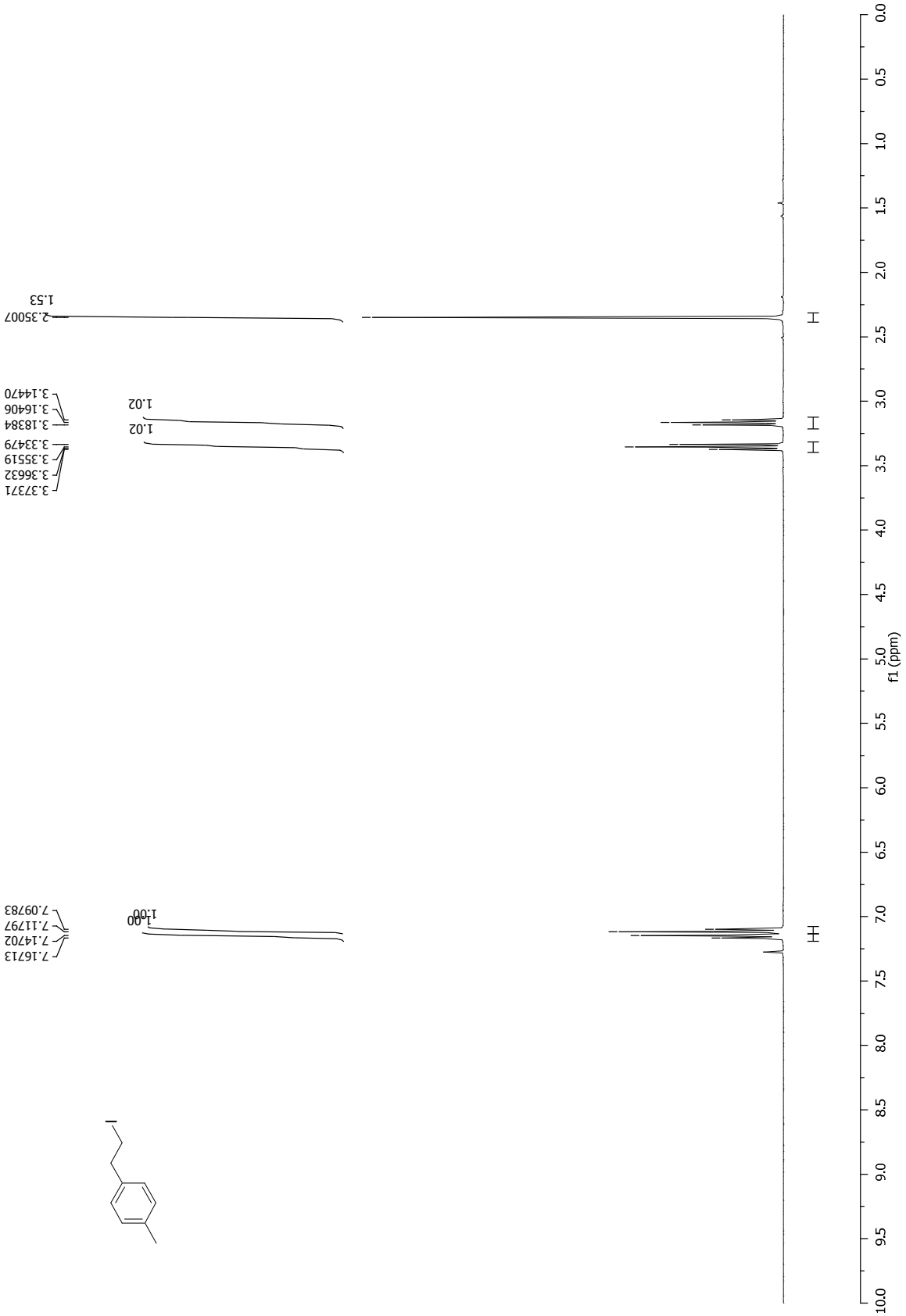
_N_,_N_-Dimethyl-_N_-(methylsulfanylmethylene)ammonium iodide (6)

^{13}C NMR, 150 MHz, $CDCl_3$



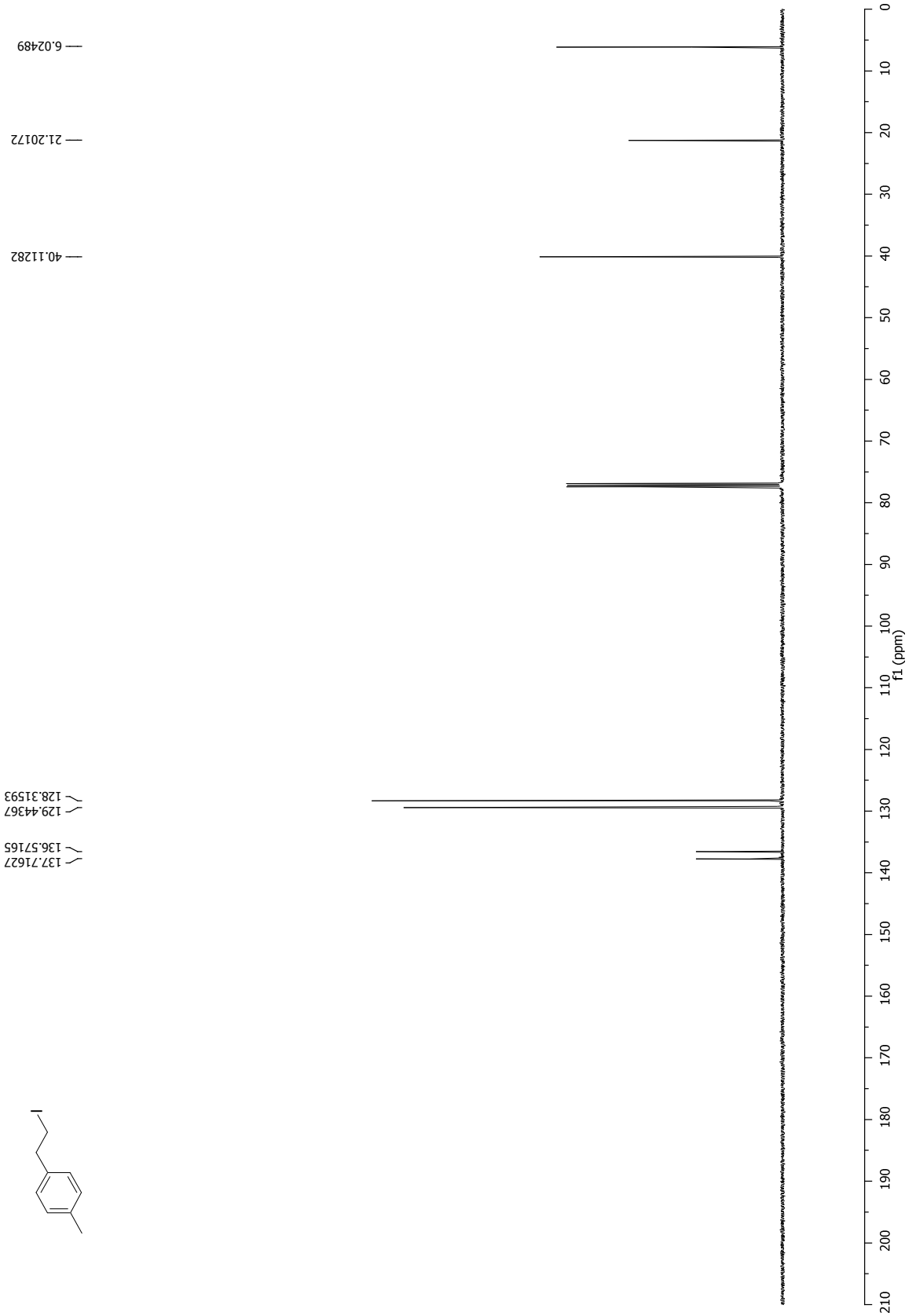
1-Iodo-2-(4-methylphenyl)ethane (11; entry 1, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



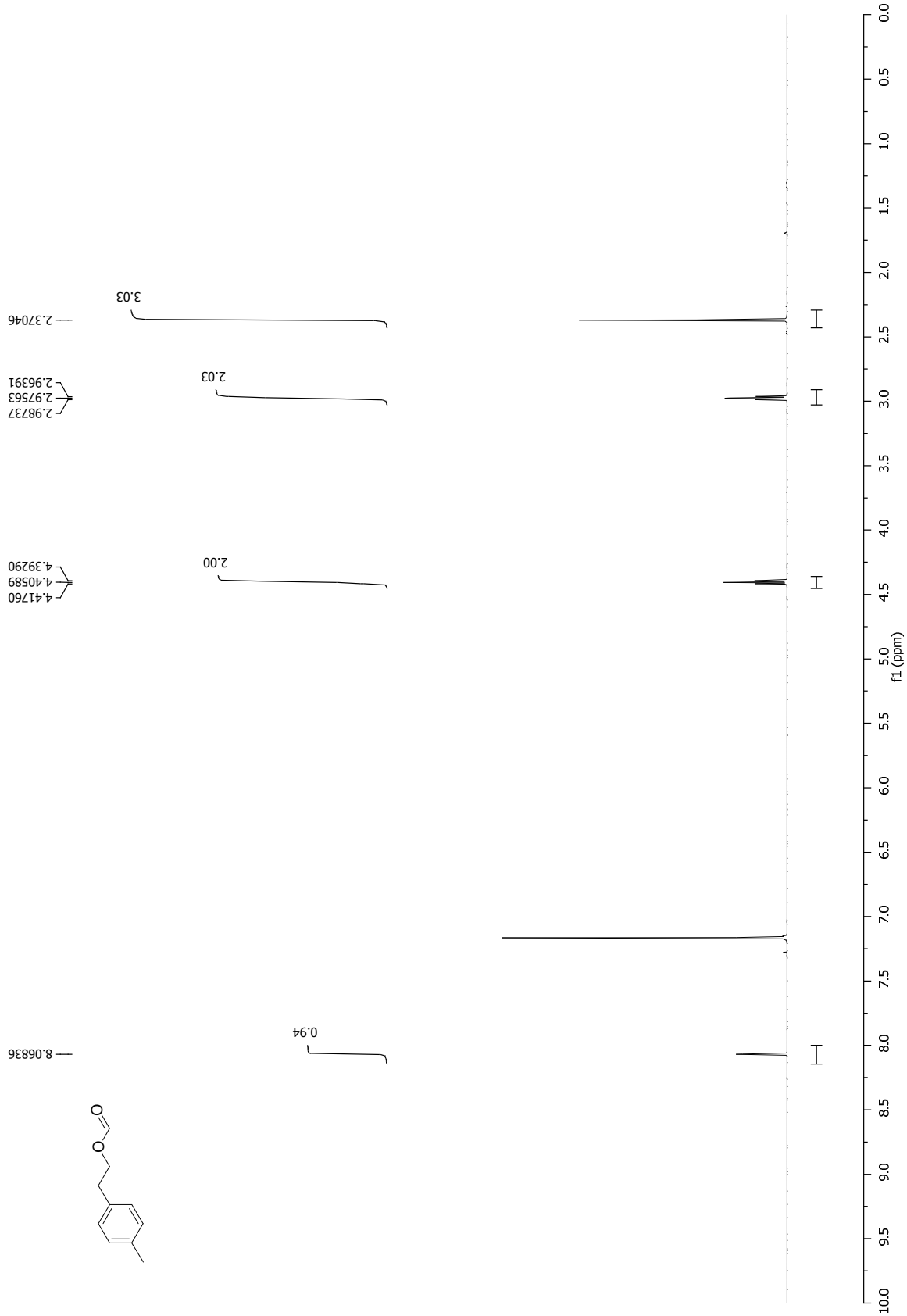
1-Iodo-2-(4-methylphenyl)ethane (11; entry 1, table 3)

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



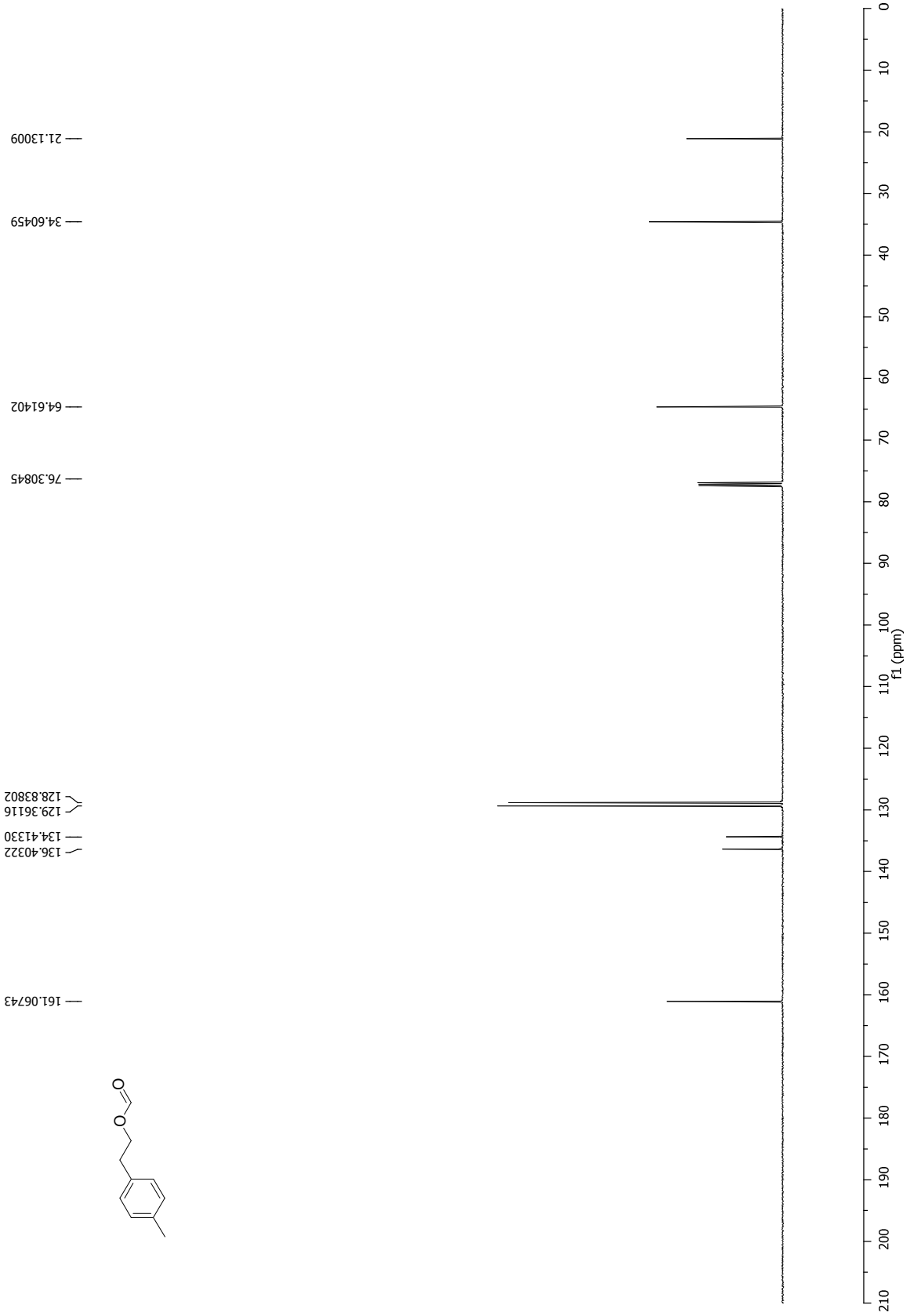
2-(4-Methylphenyl)ethyl formate (12)

^{1}H NMR, 400 MHz, $CDCl_3$



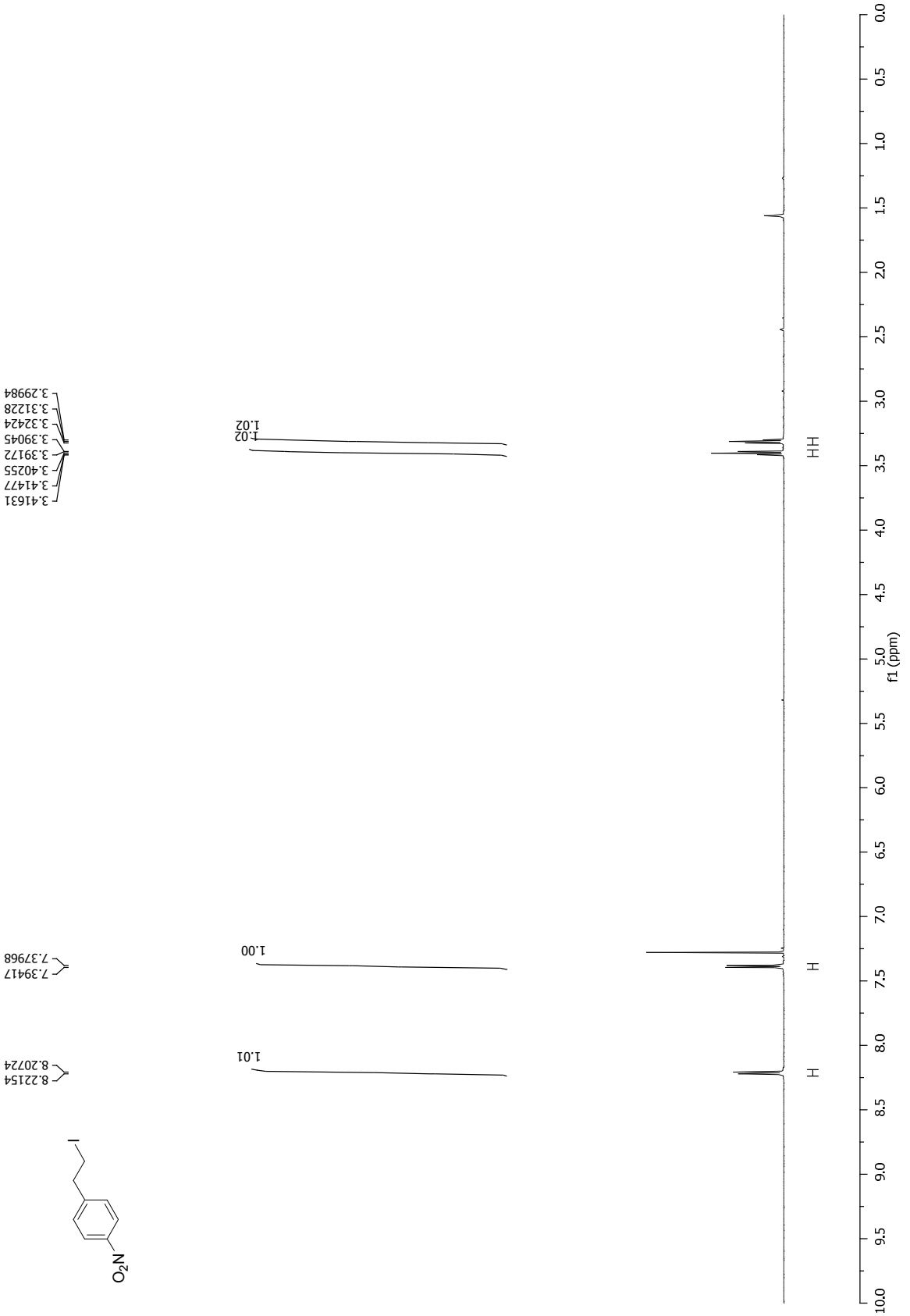
2-(4-Methylphenyl)ethyl formate (12)

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



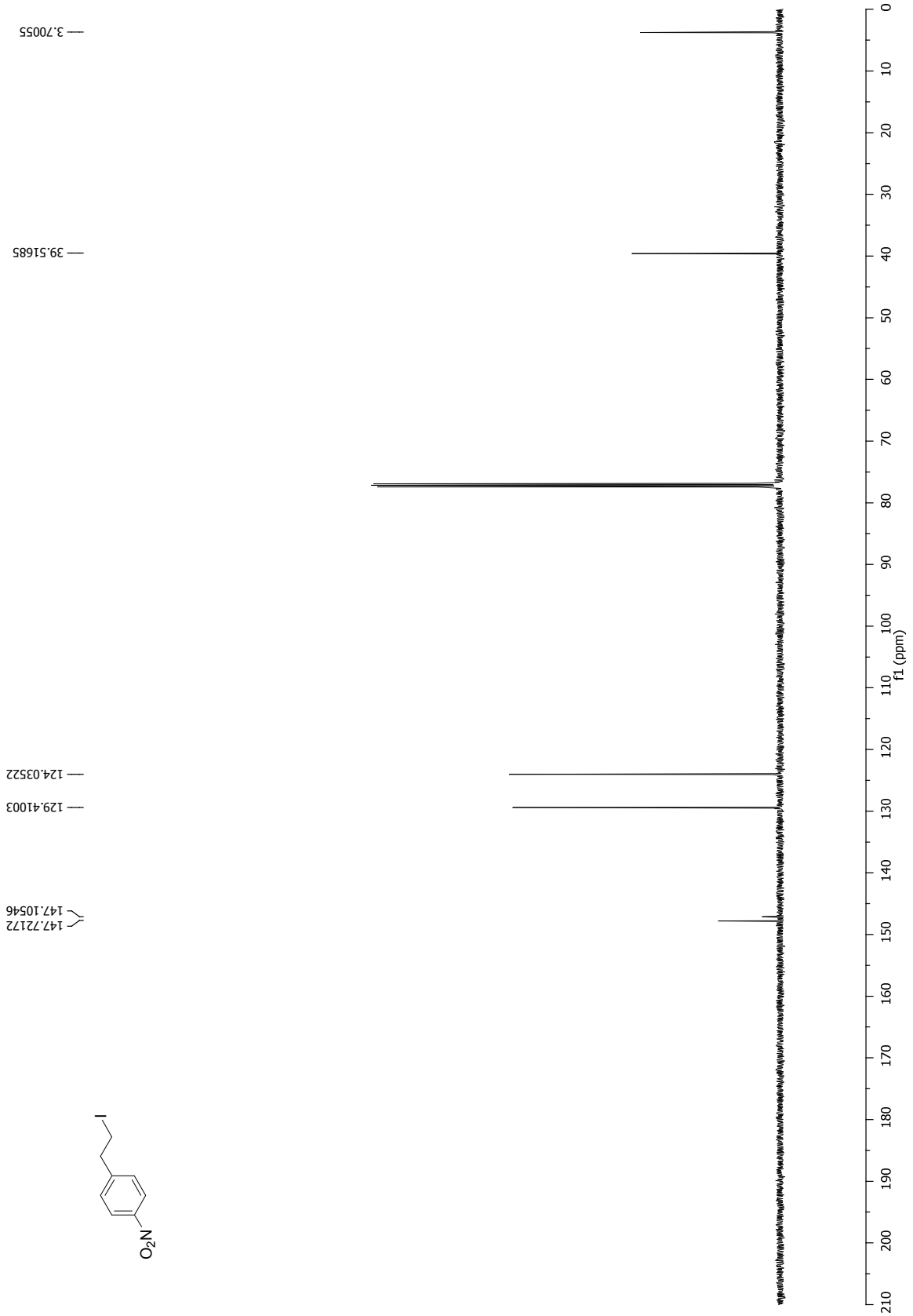
1-Iodo-2-(4-nitrophenyl)ethane (entry 2, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



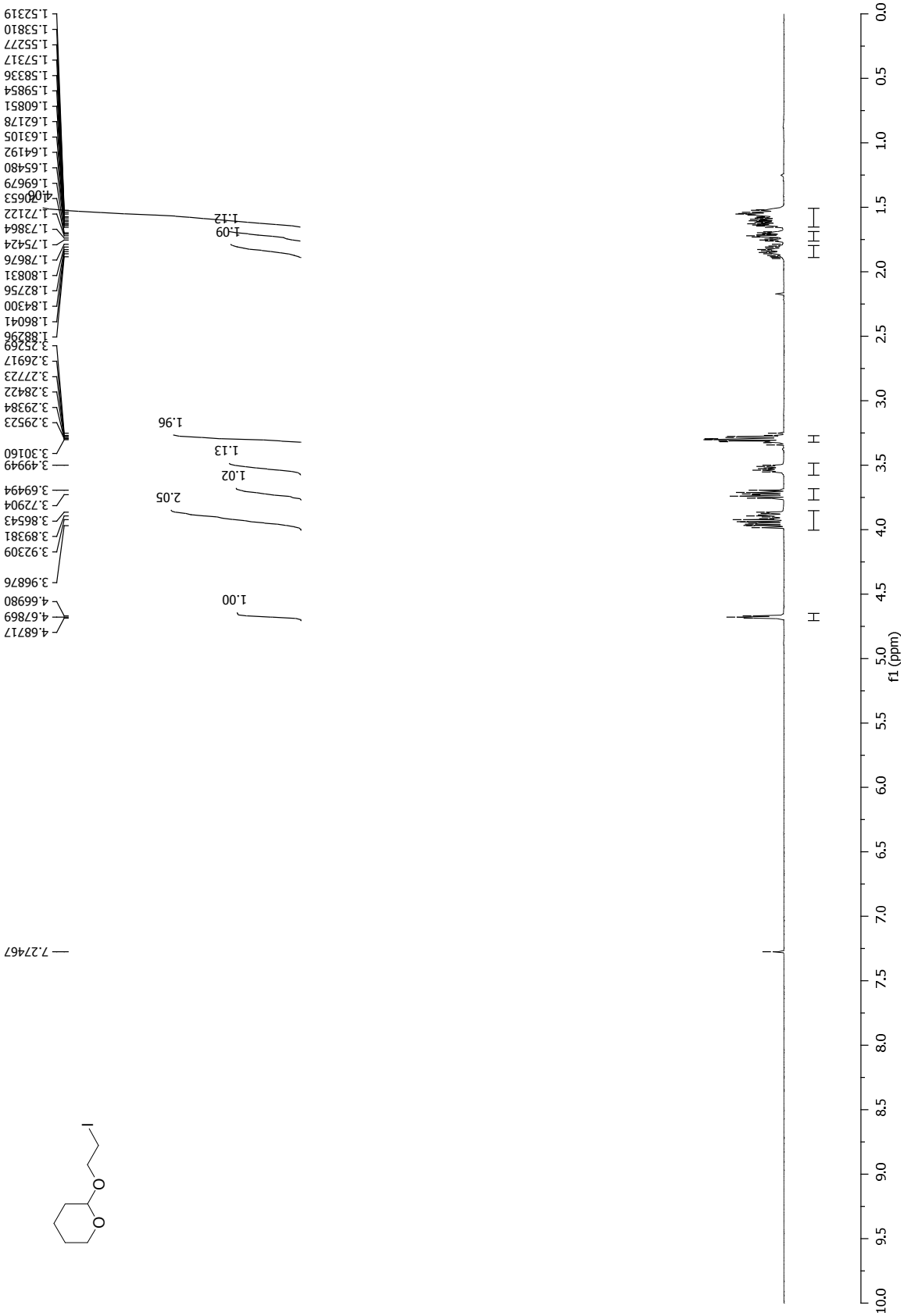
1-Iodo-2-(4-nitrophenyl)ethane (entry 2, table 3)

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



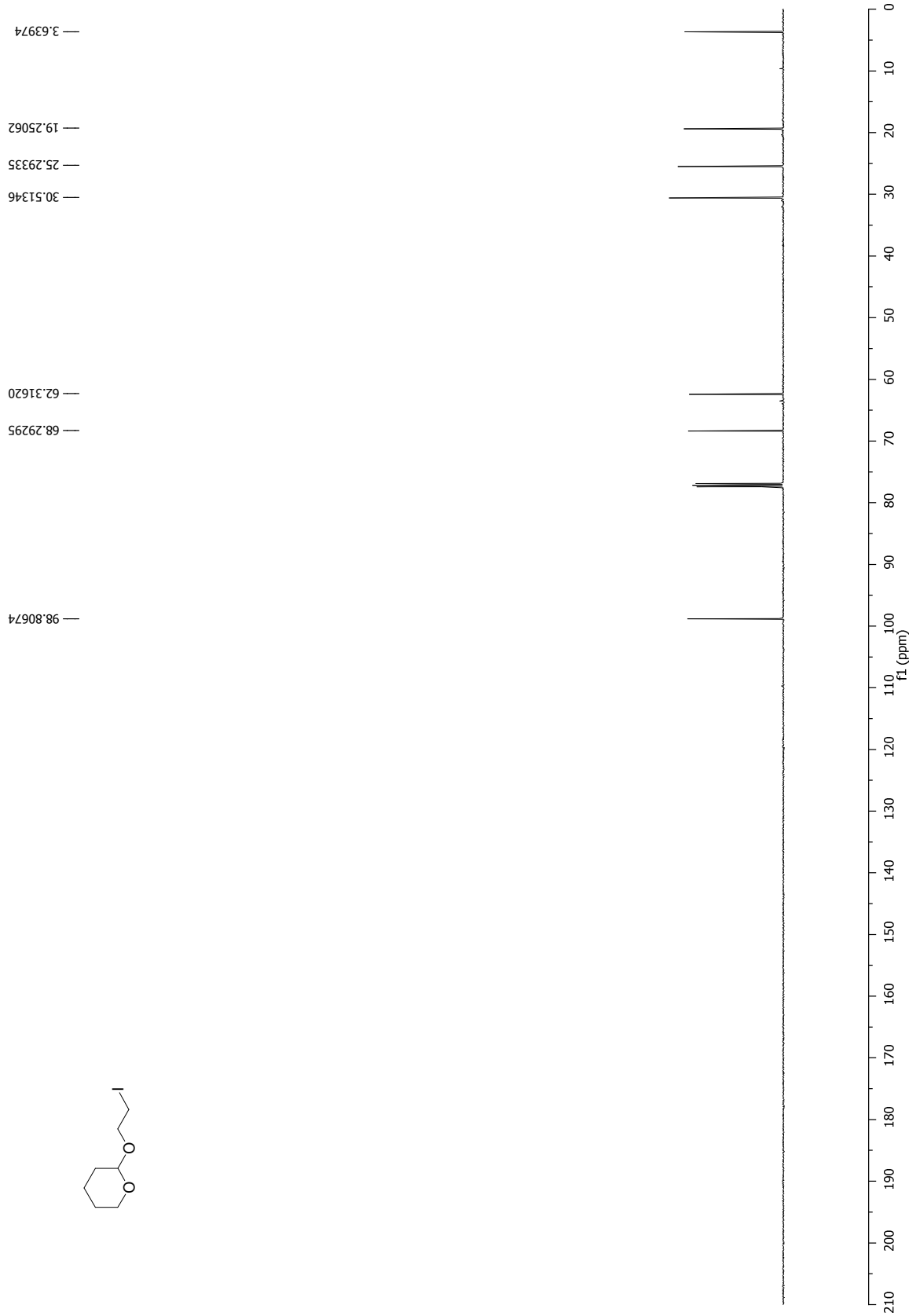
1-Iodo-2-(tetrahydropyran-2-yloxy)ethane (entry 3, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



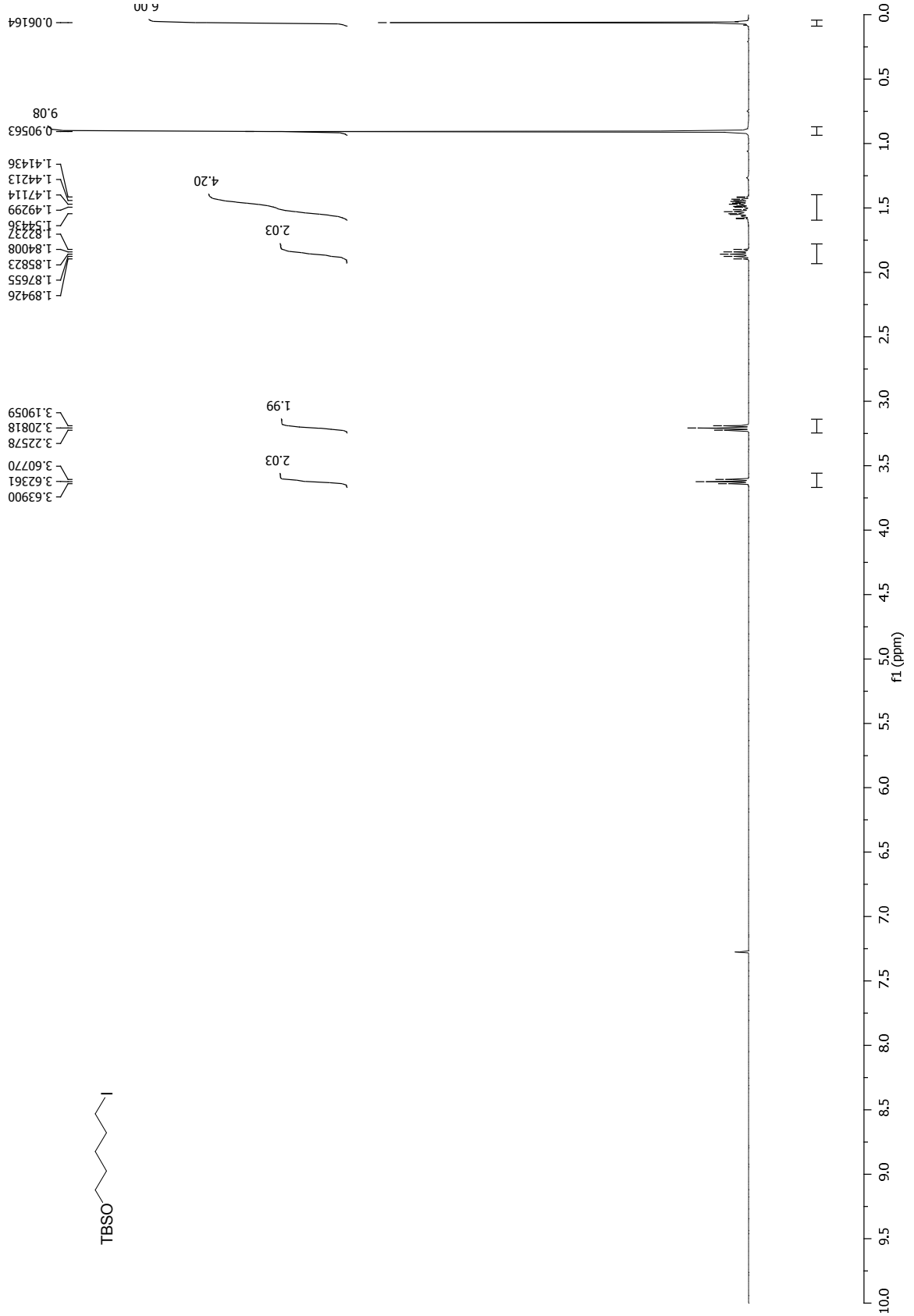
1-Iodo-2-(tetrahydropyran-2-yloxy)ethane (entry 3, table 3)

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



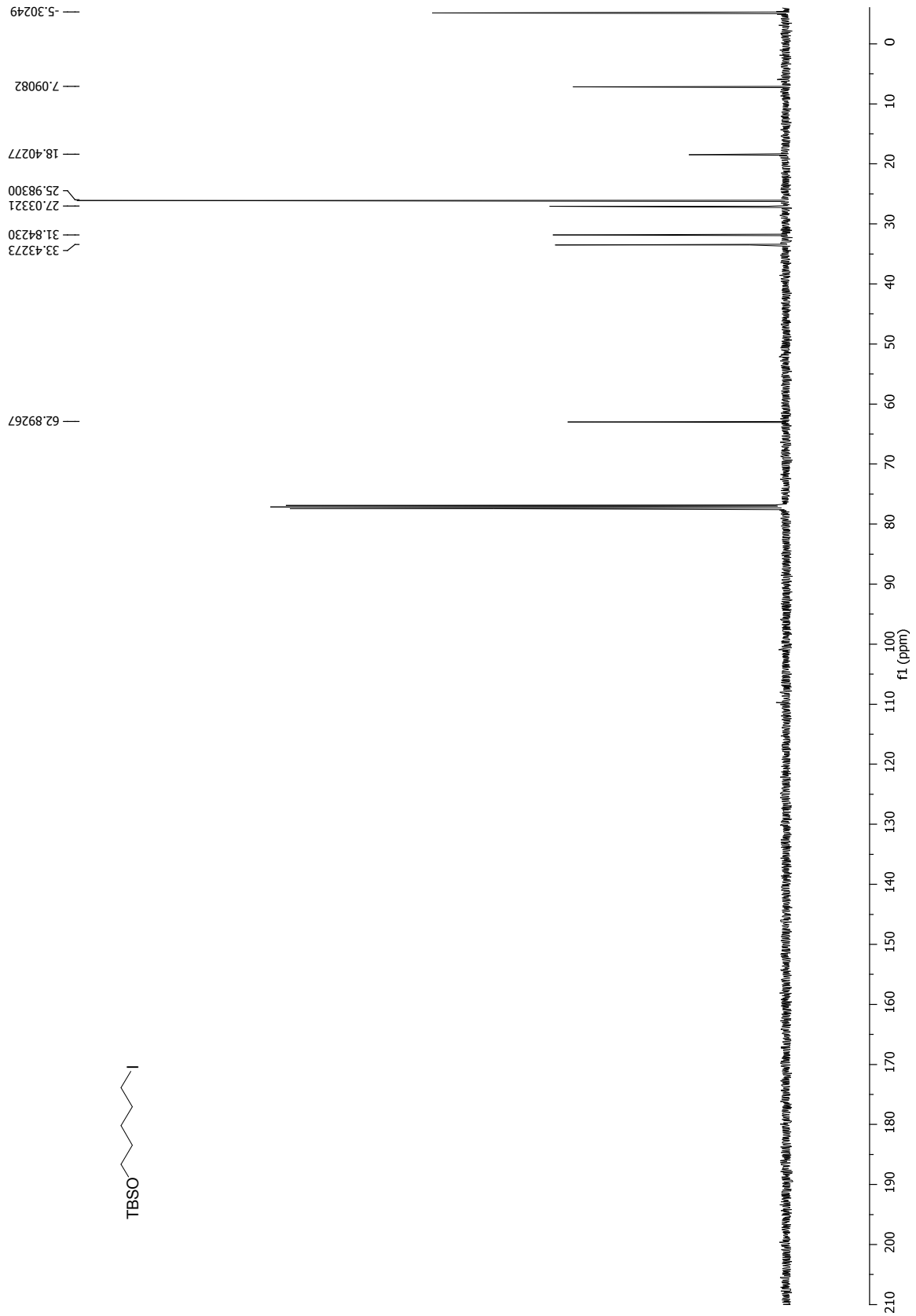
1-*tert*-Butyldimethylsilanyloxy-5-iodopentane (entry 4, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



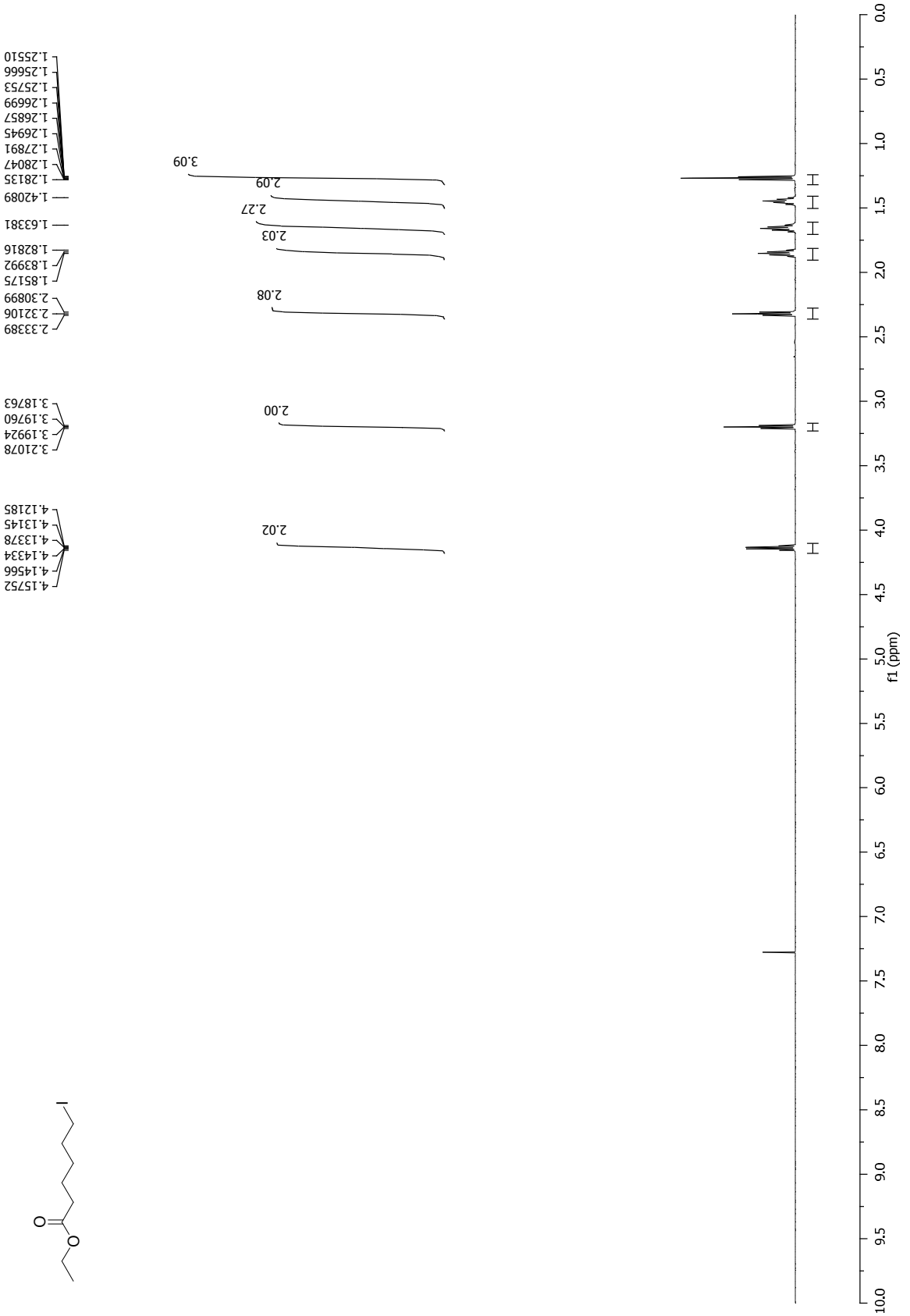
1-*tert*-Butyldimethylsilanyloxy-5-iodopentane (entry 4, table 3)

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



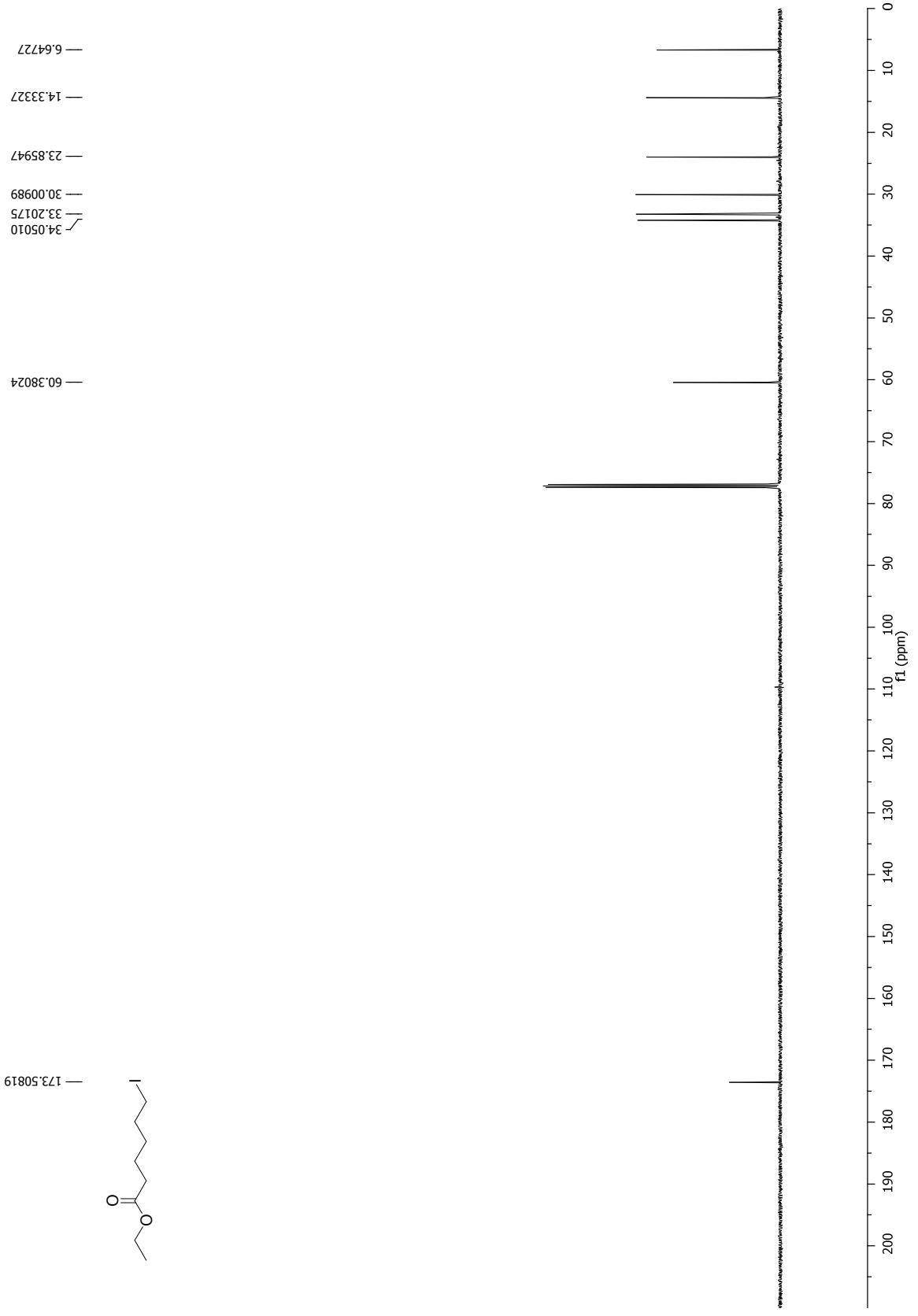
Ethyl 6-iodohexanoate (entry 5, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



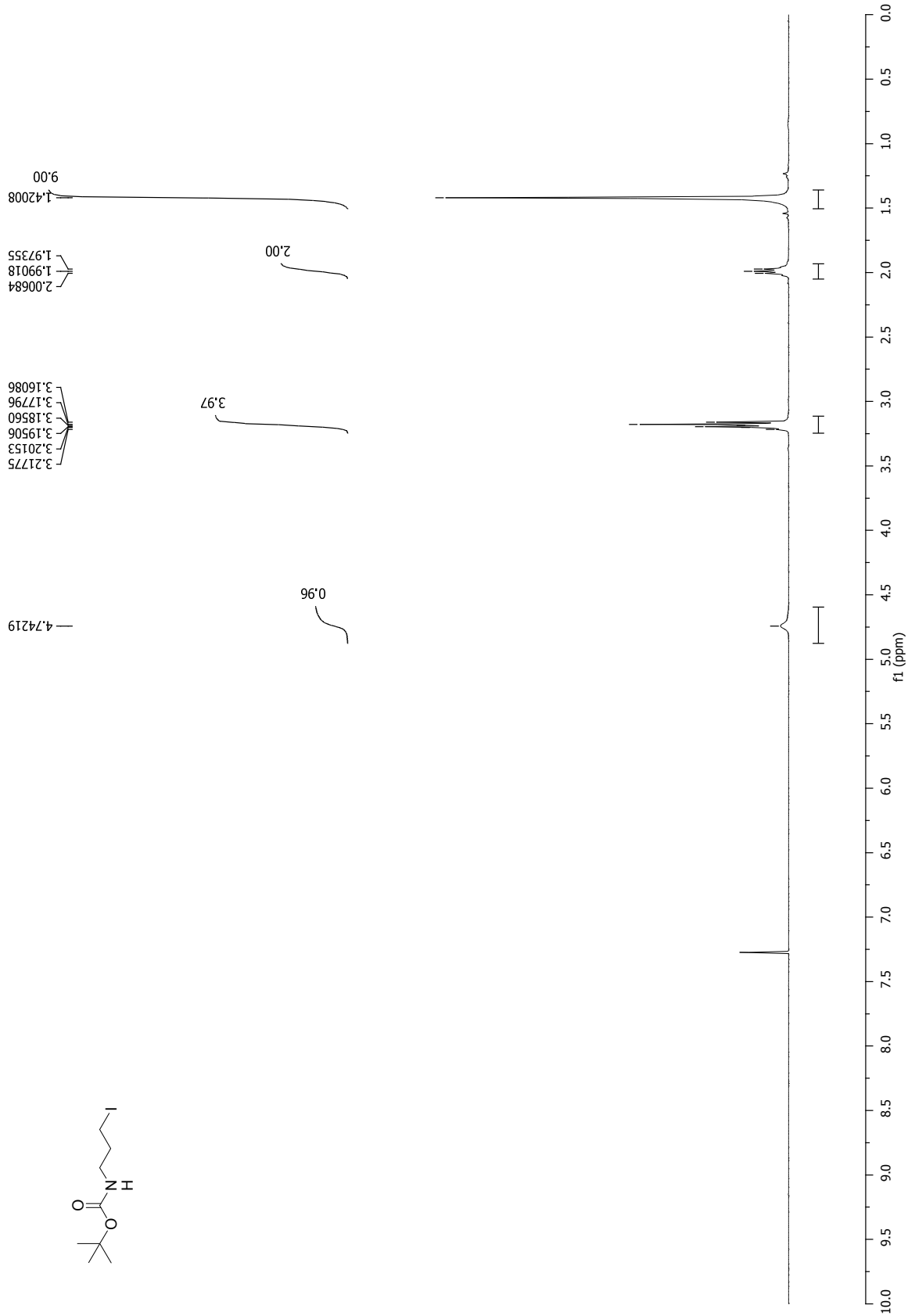
Ethyl 6-iodohexanoate (entry 5, table 3)

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



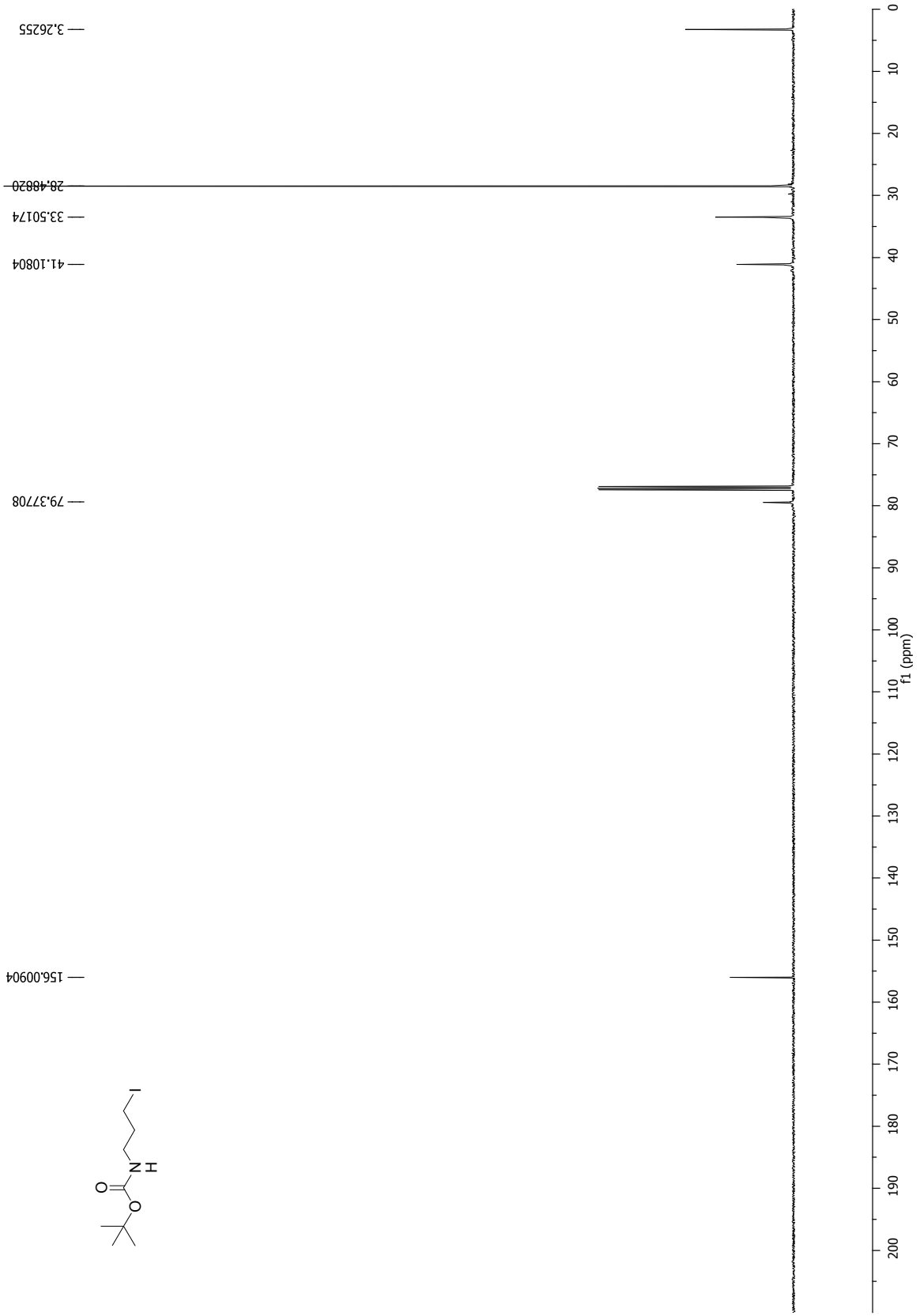
N-tert-Butoxycarbonyl-3-iodopropylamine (entry 6, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



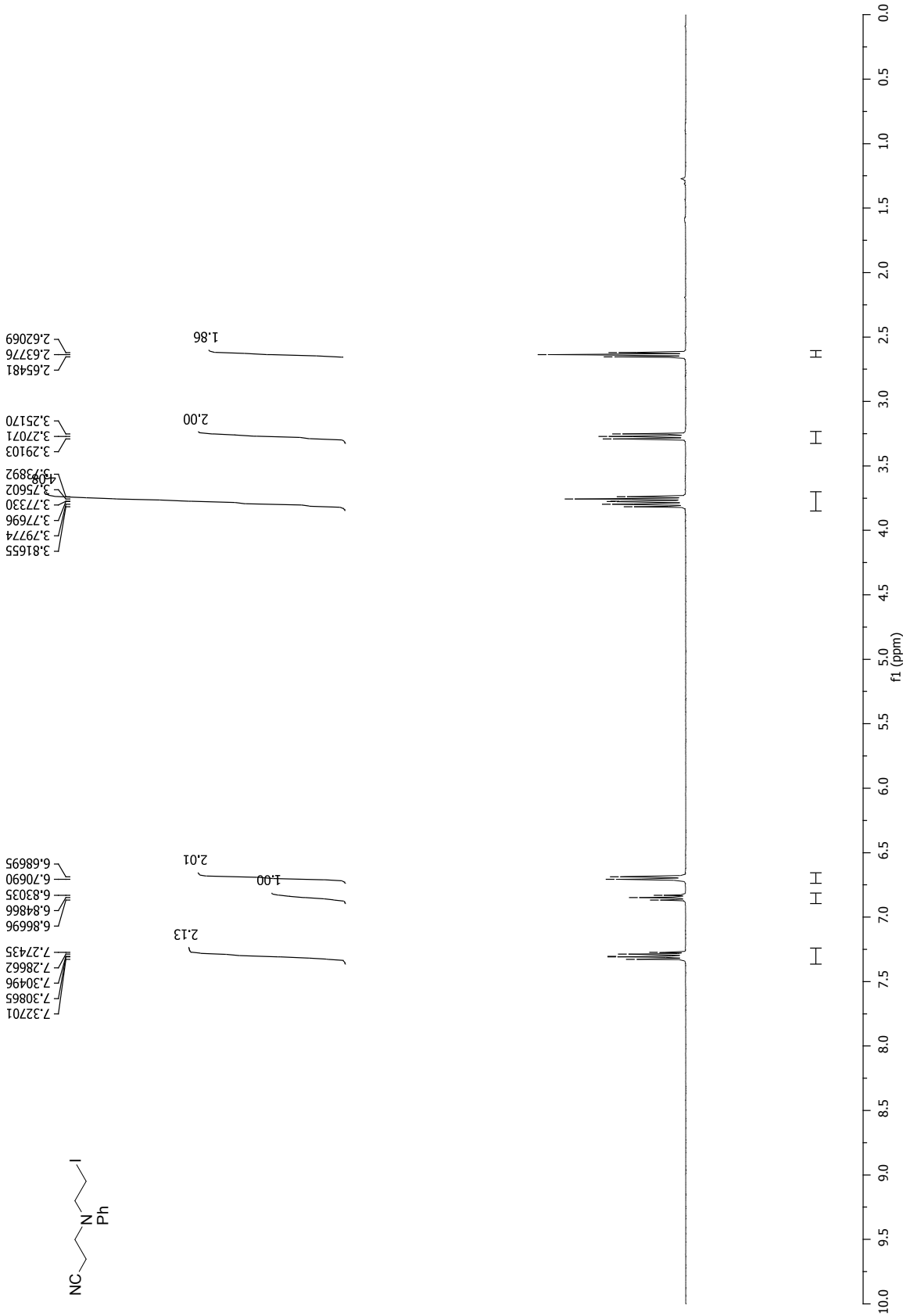
N-tert-Butoxycarbonyl-3-iodopropylamine (entry 6, table 3)

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



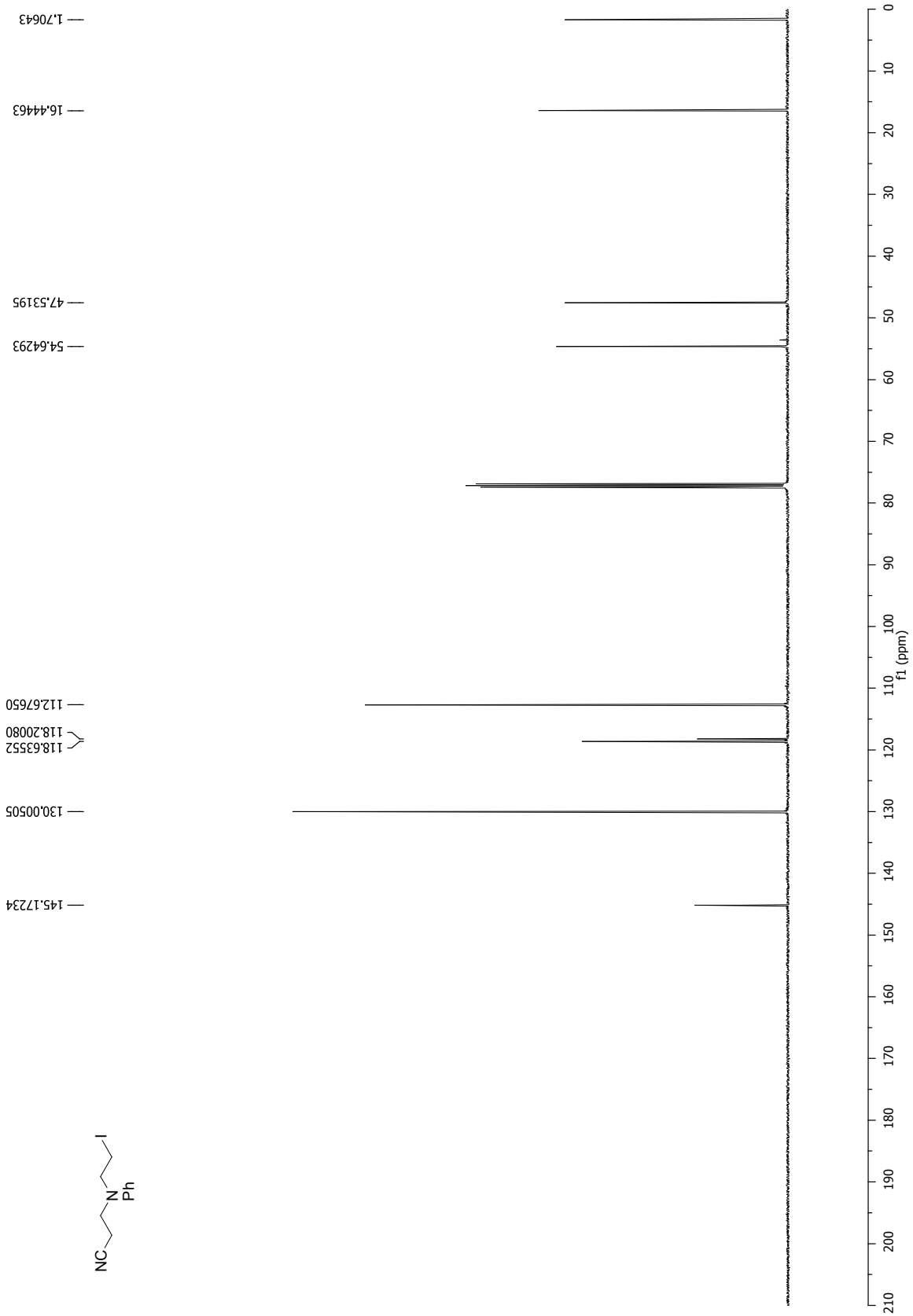
_N_-(2-Cyanoethyl)-_N_-(2-iodoethyl)aniline (entry 7, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



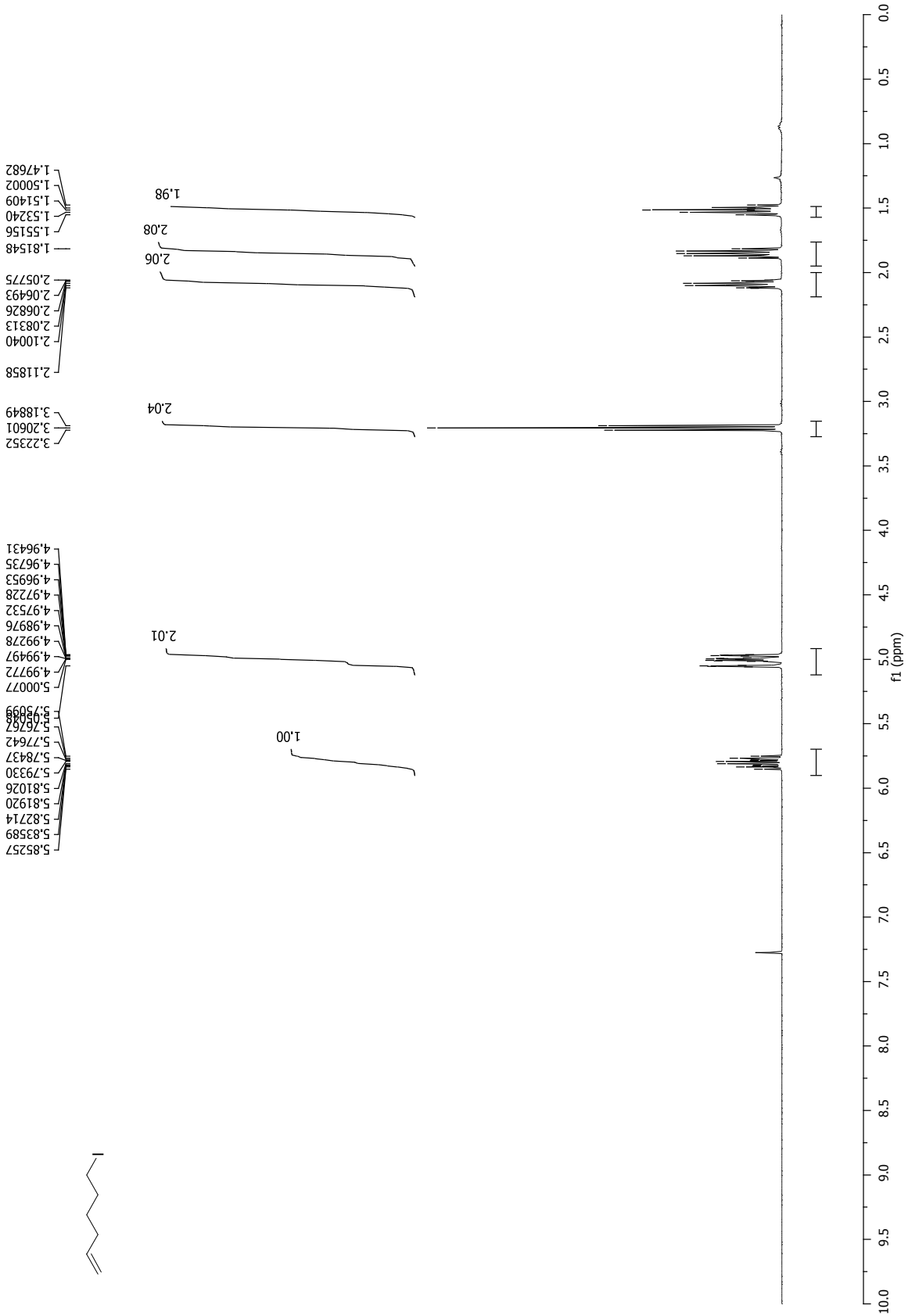
_N_-(2-Cyanoethyl)-_N_-(2-iodoethyl)aniline (entry 7, table 3)

^{13}C NMR, 150 MHz, $CDCl_3$



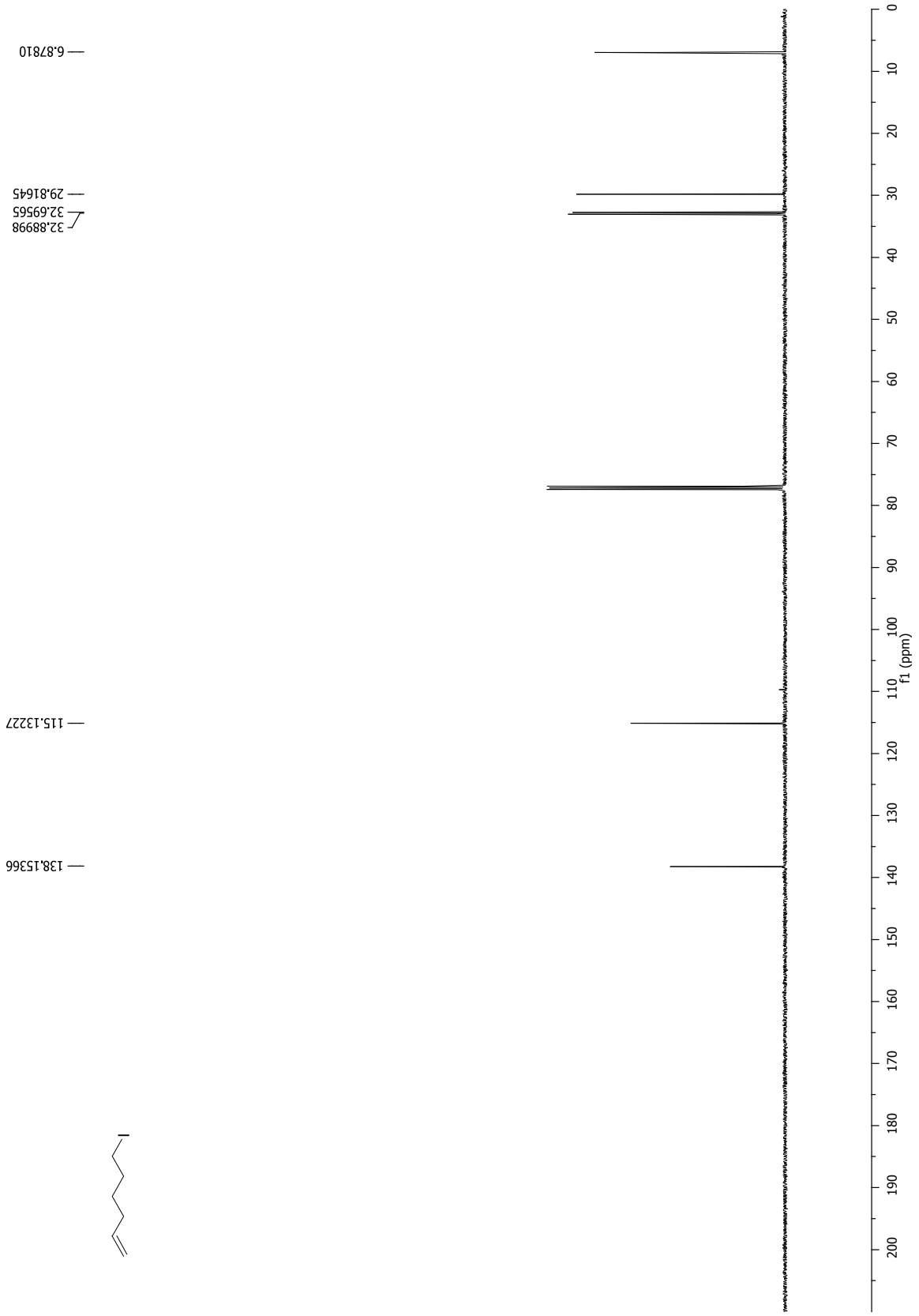
6-Iodohex-1-ene (entry 8, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



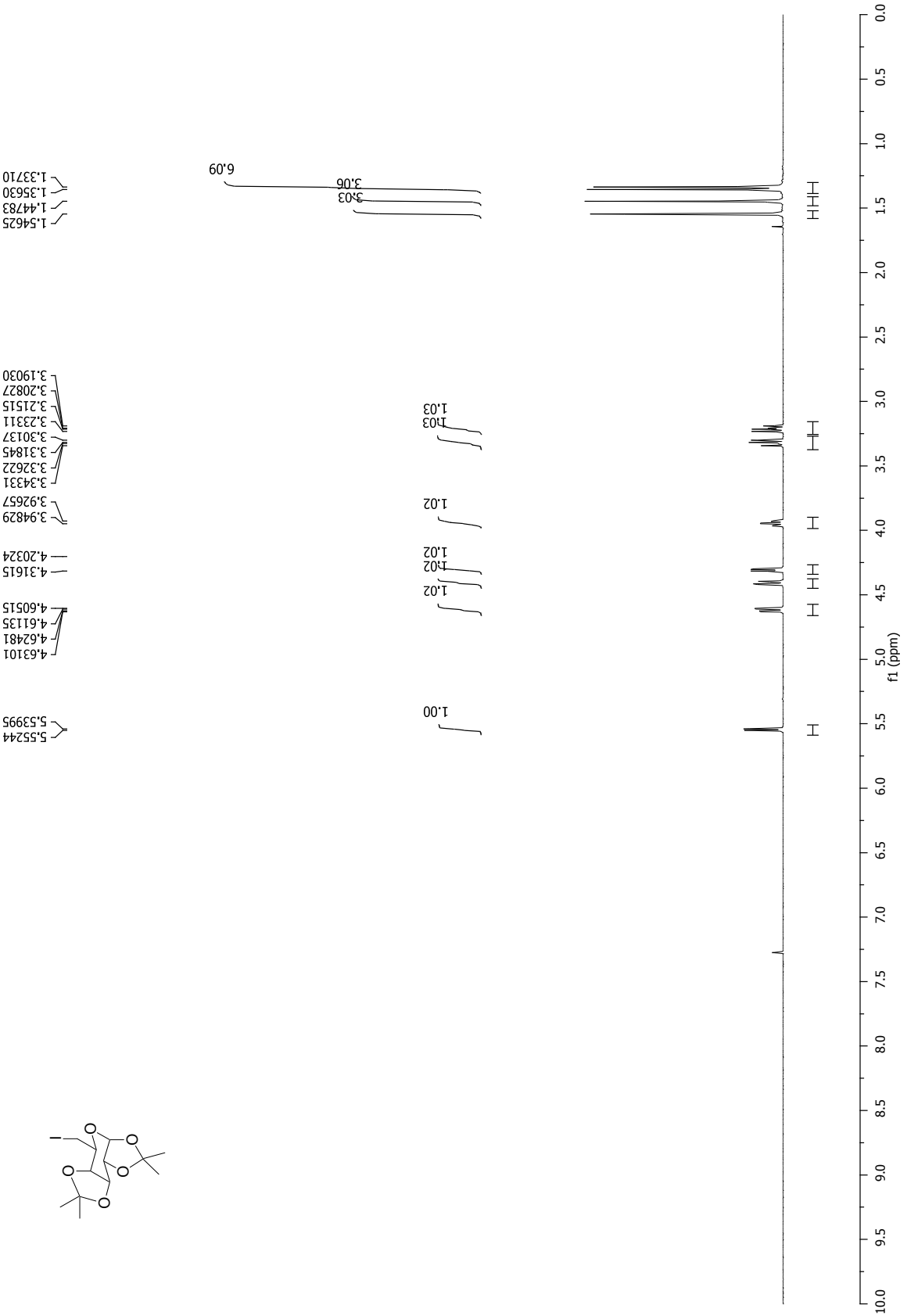
6-Iodohex-1-ene (entry 8, table 3)

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



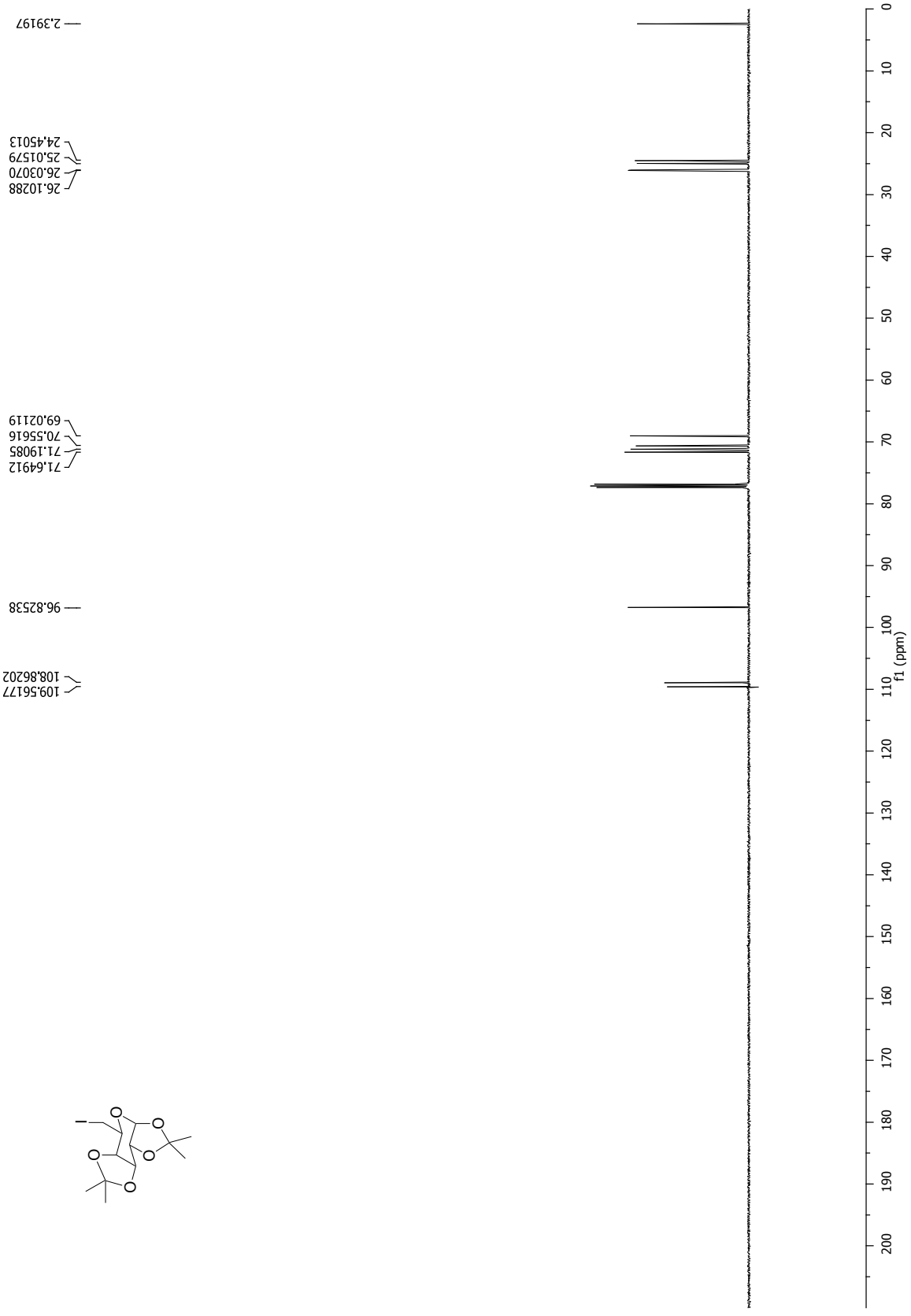
6-Deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (entry 9, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



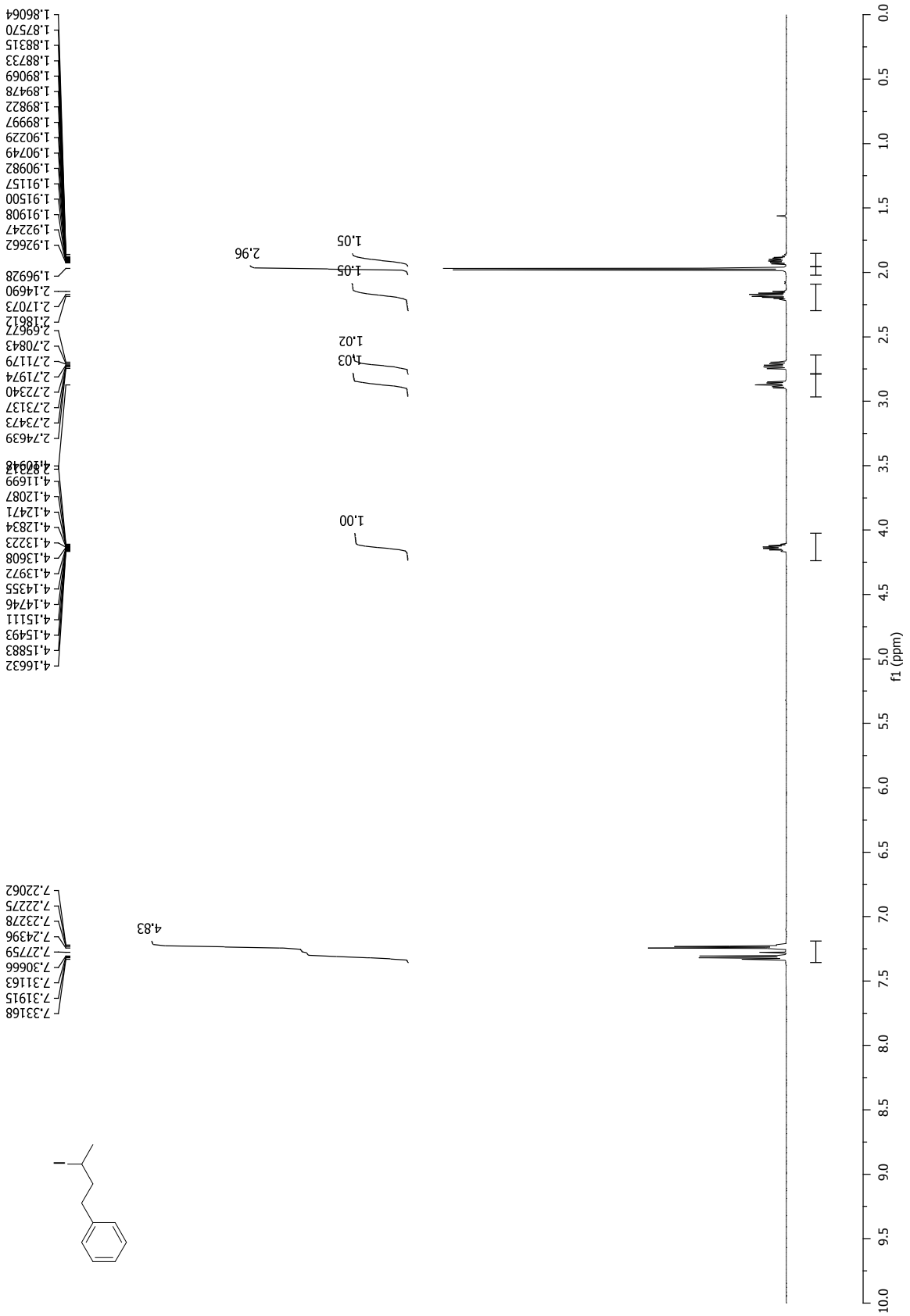
6-Deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (entry 9, table 3)

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



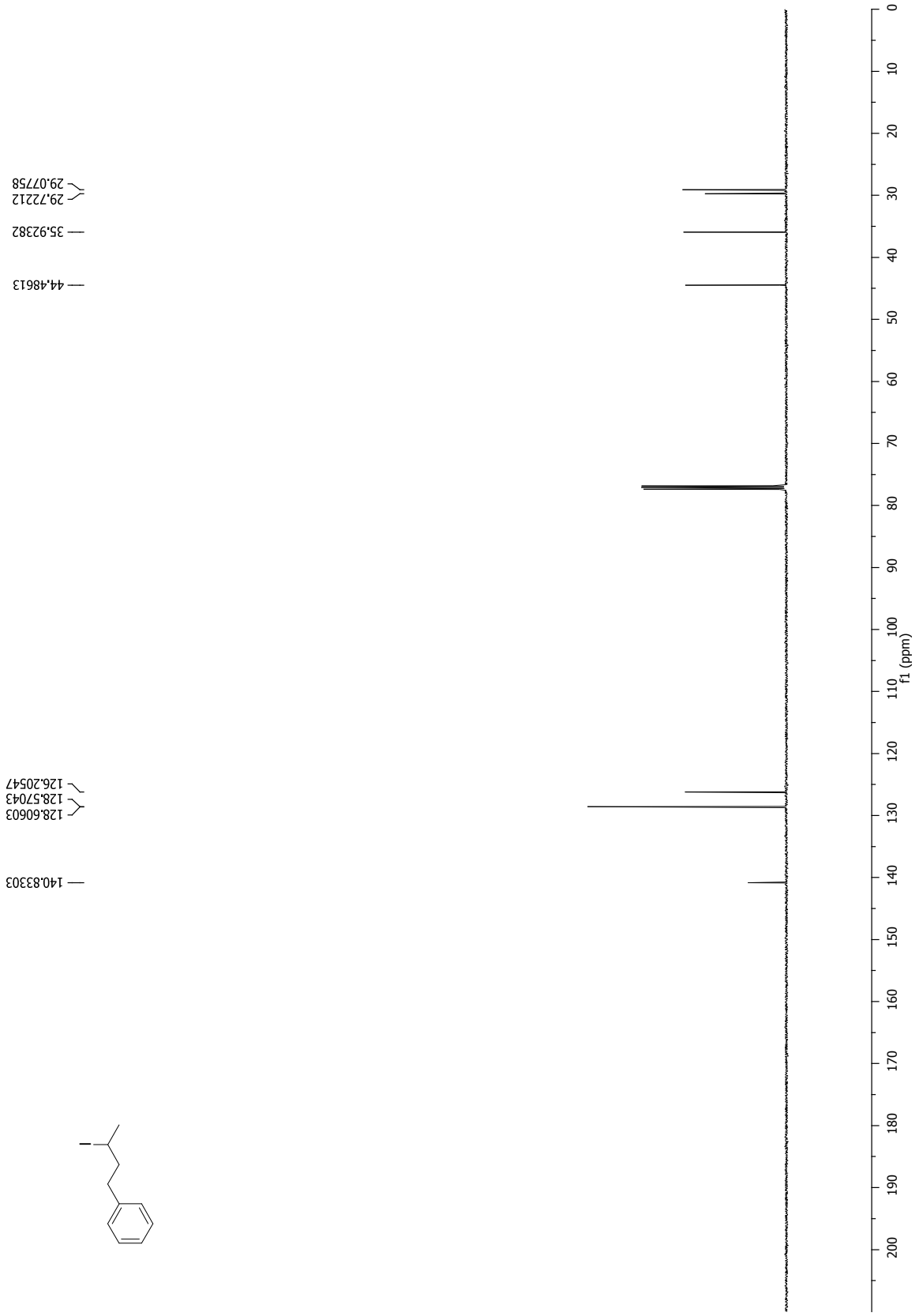
3-Iodo-1-phenylbutane (entry 11, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



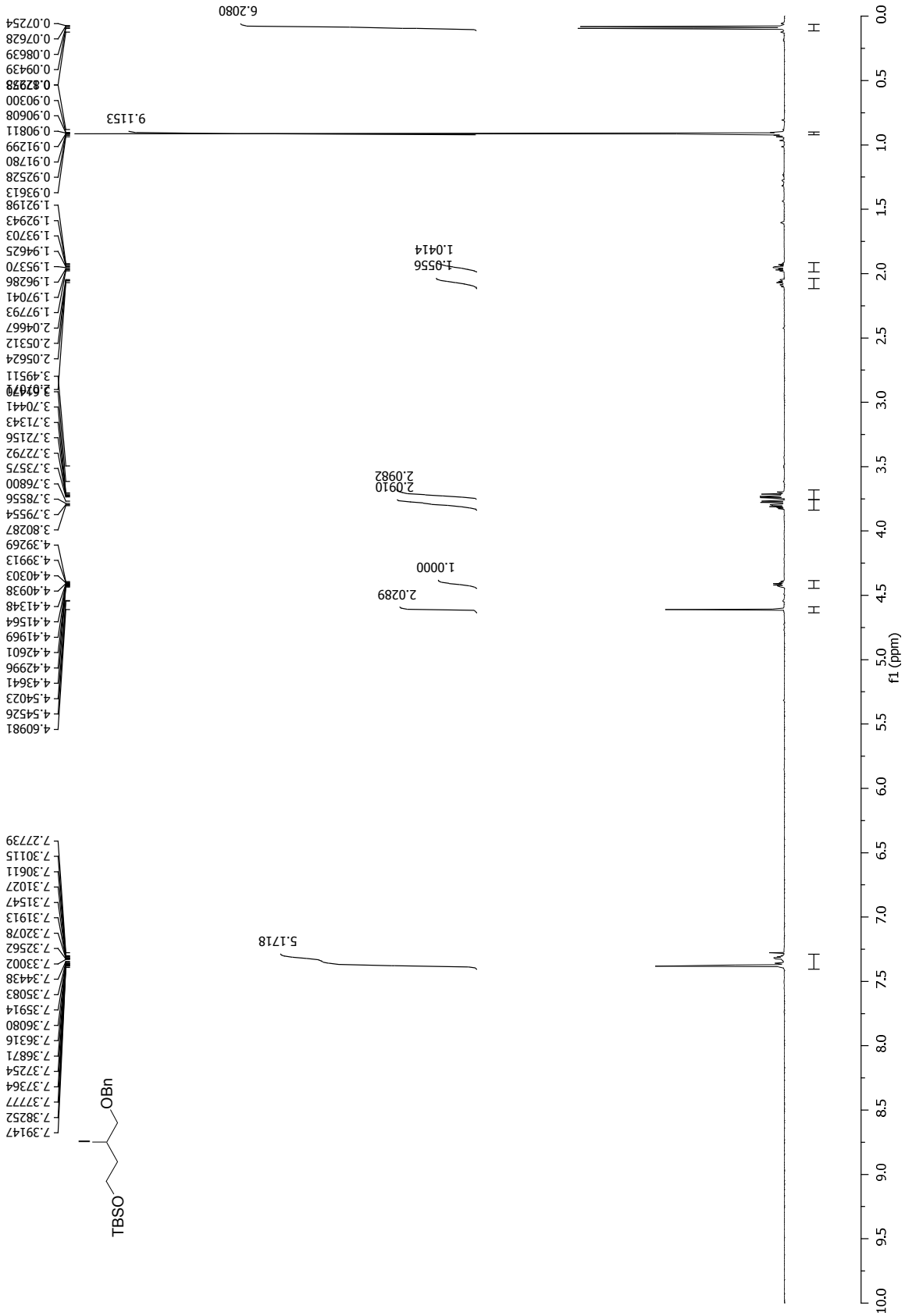
3-Iodo-1-phenylbutane (entry 11, table 3)

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



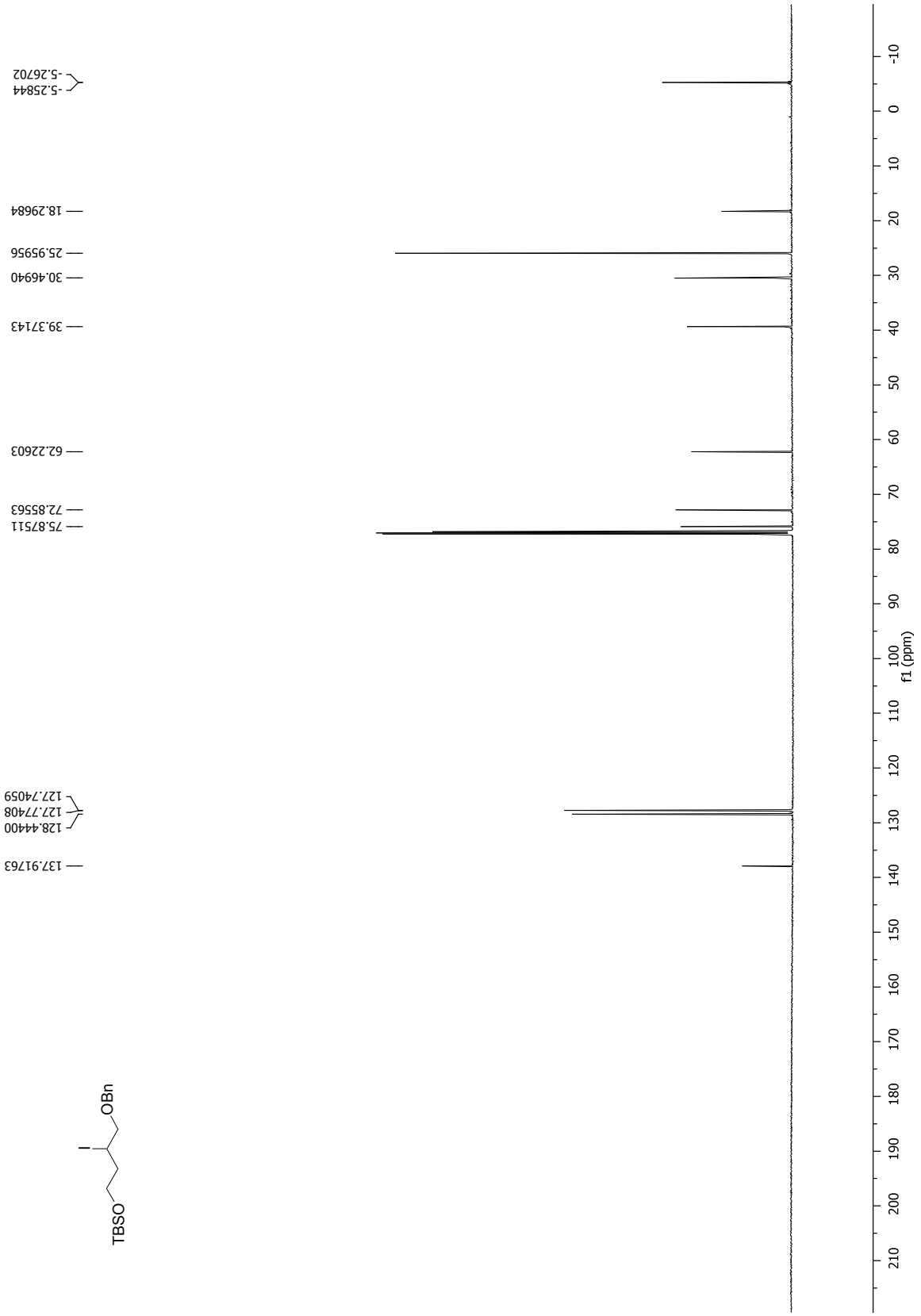
1-Benzyloxy-4-(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 12, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



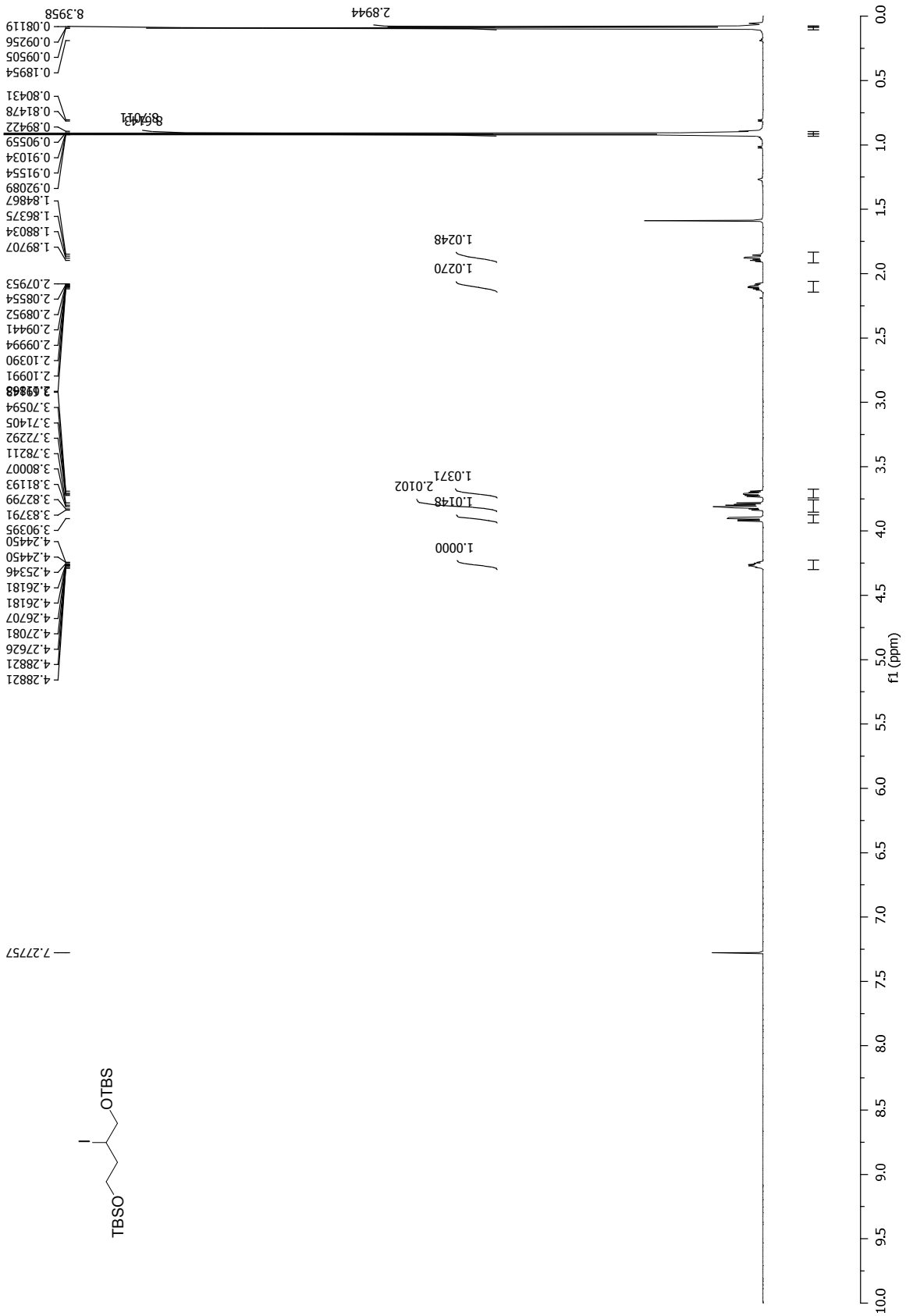
1-Benzyloxy-4-(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 12, table 3)

^{13}C NMR, 150 MHz, $CDCl_3$



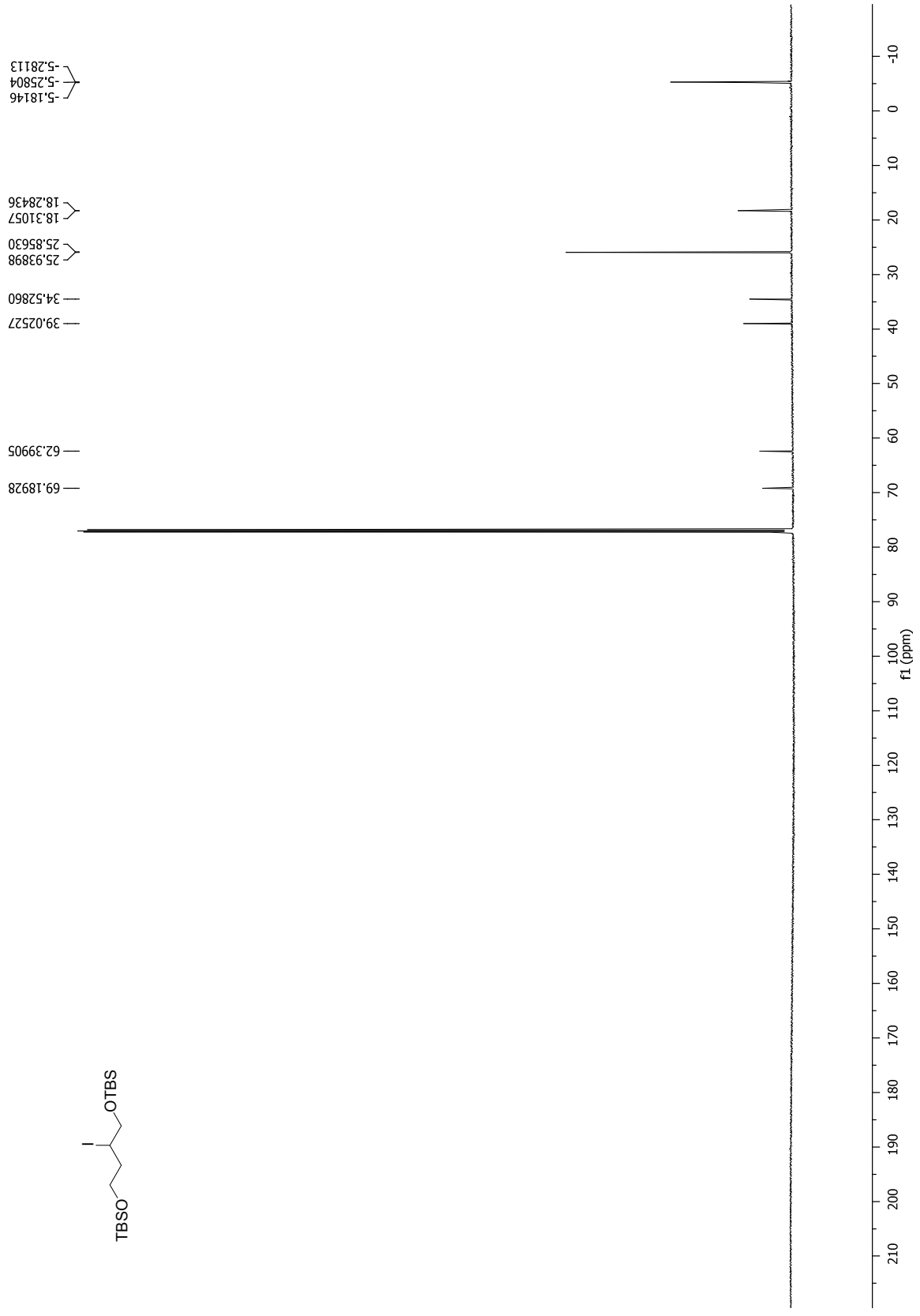
1,4-Di(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 13, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



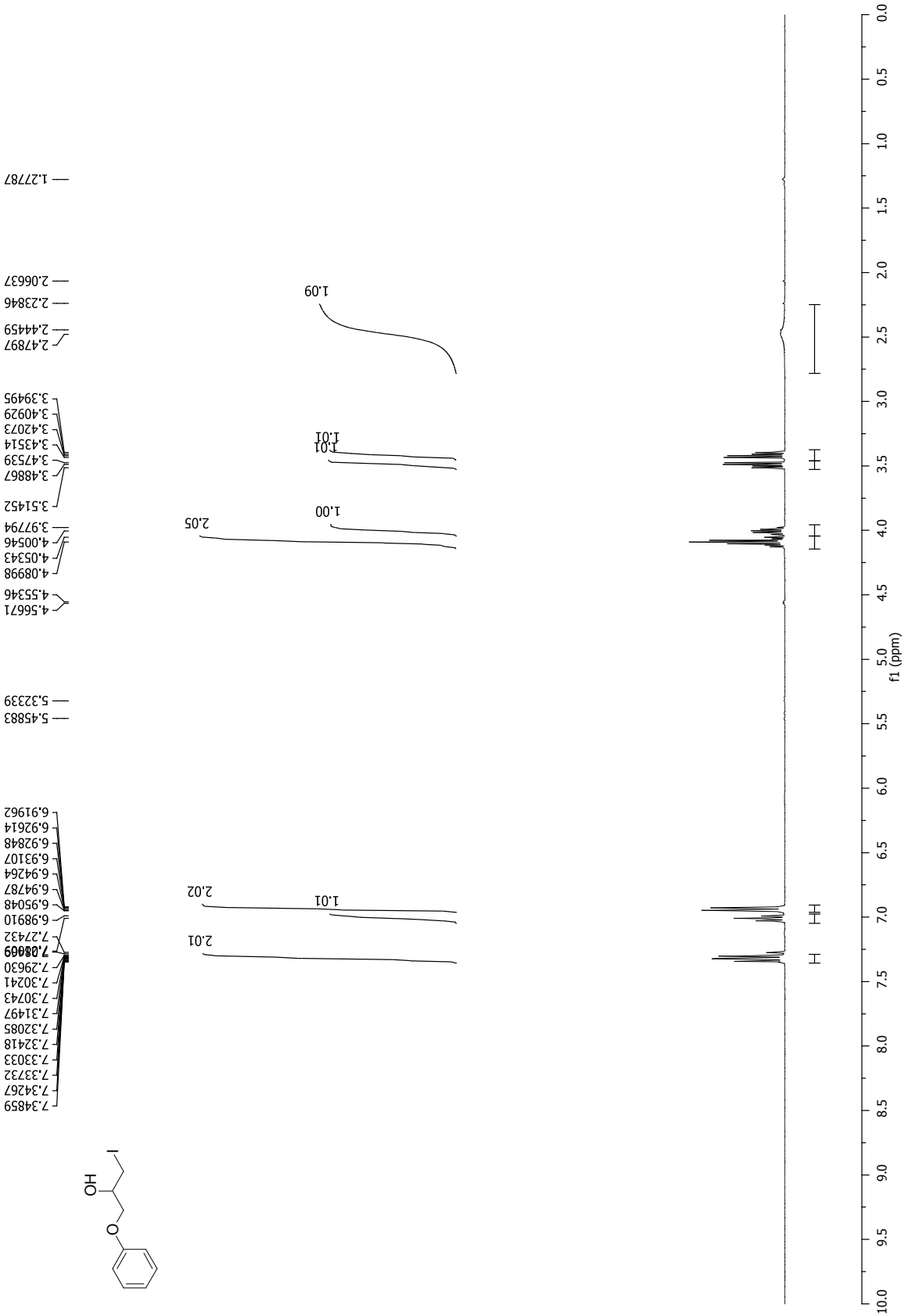
1,4-Di(*tert*-butyldimethylsilanyloxy)-2-iodobutane (entry 13, table 3)

^{13}C NMR, 150 MHz, $CDCl_3$



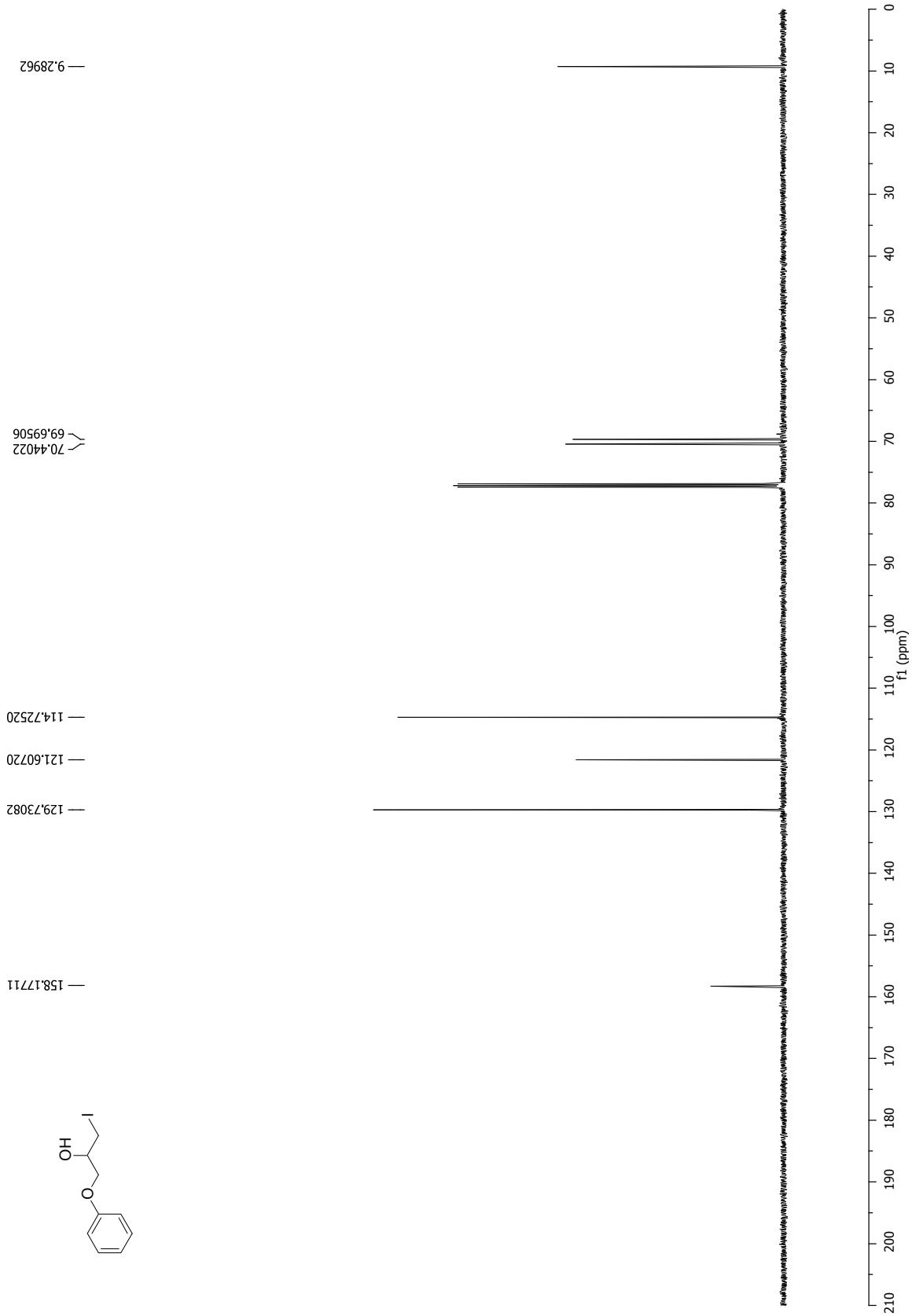
1-Iodo-3-phenoxypropan-2-ol (entry 15, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



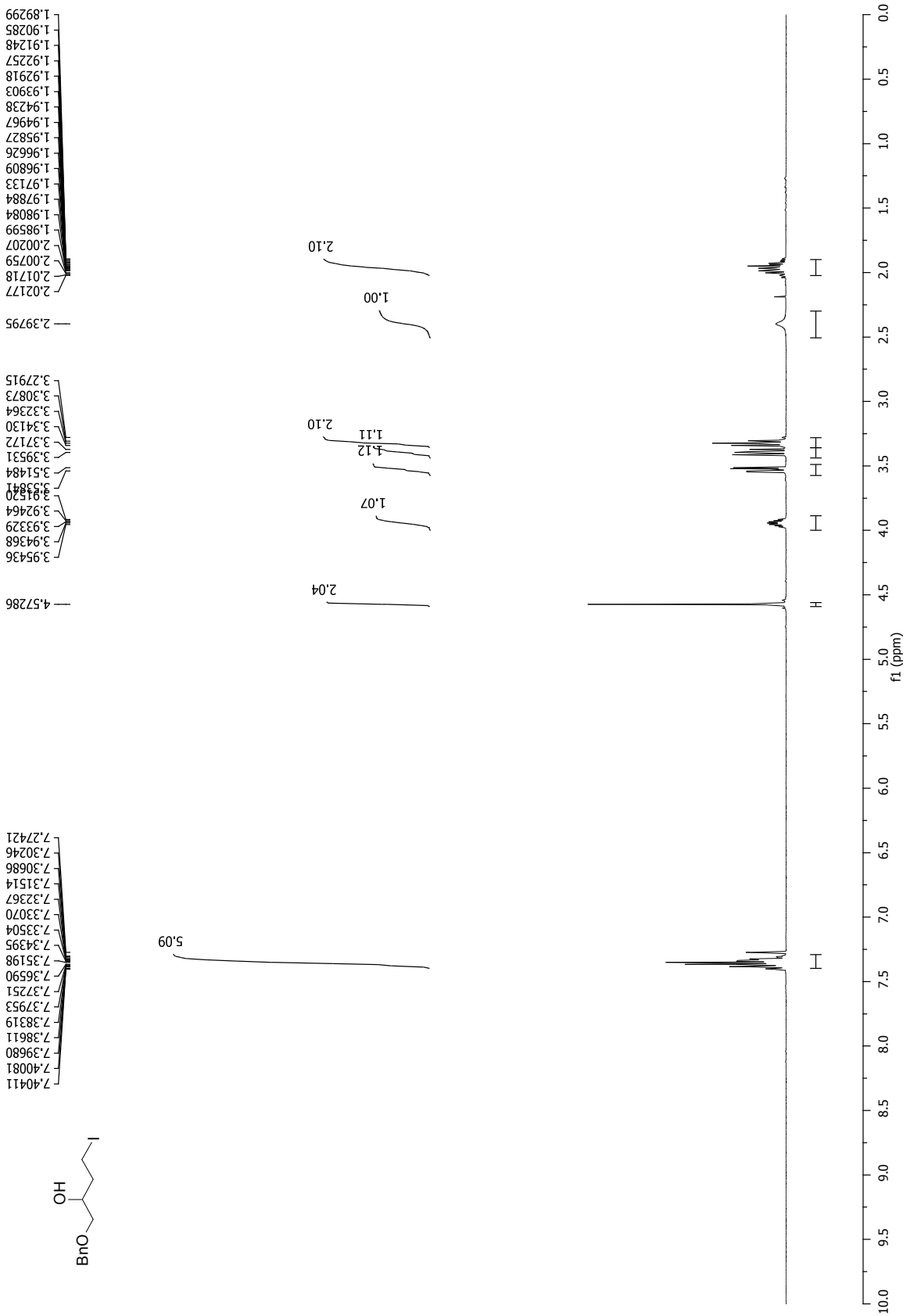
1-Iodo-3-phenoxypropan-2-ol (entry 15, table 3)

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



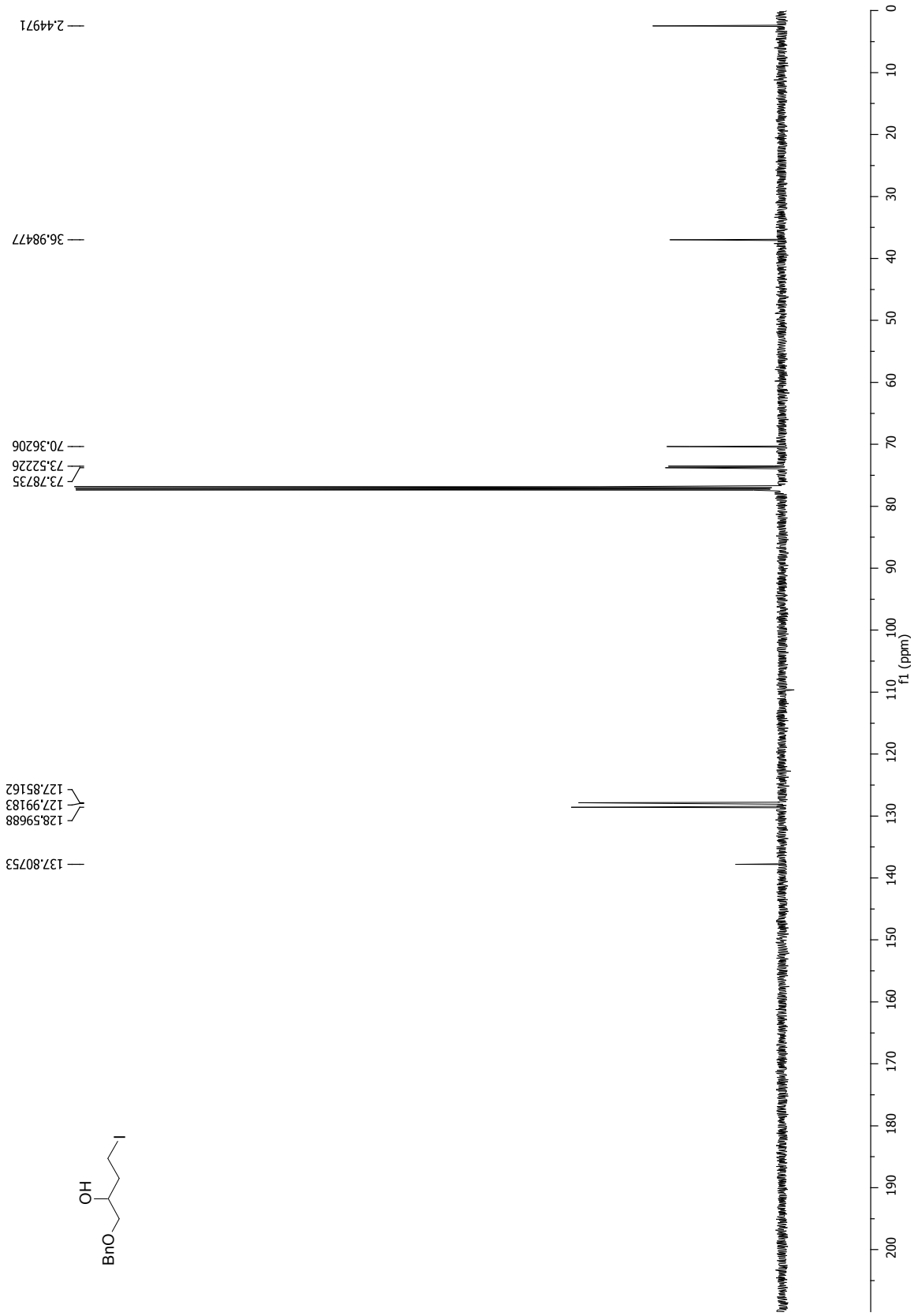
1-Benzyloxy-4-iodobutan-2-ol (entry 16, table 3)

^{1}H NMR, 400 MHz, $CDCl_3$



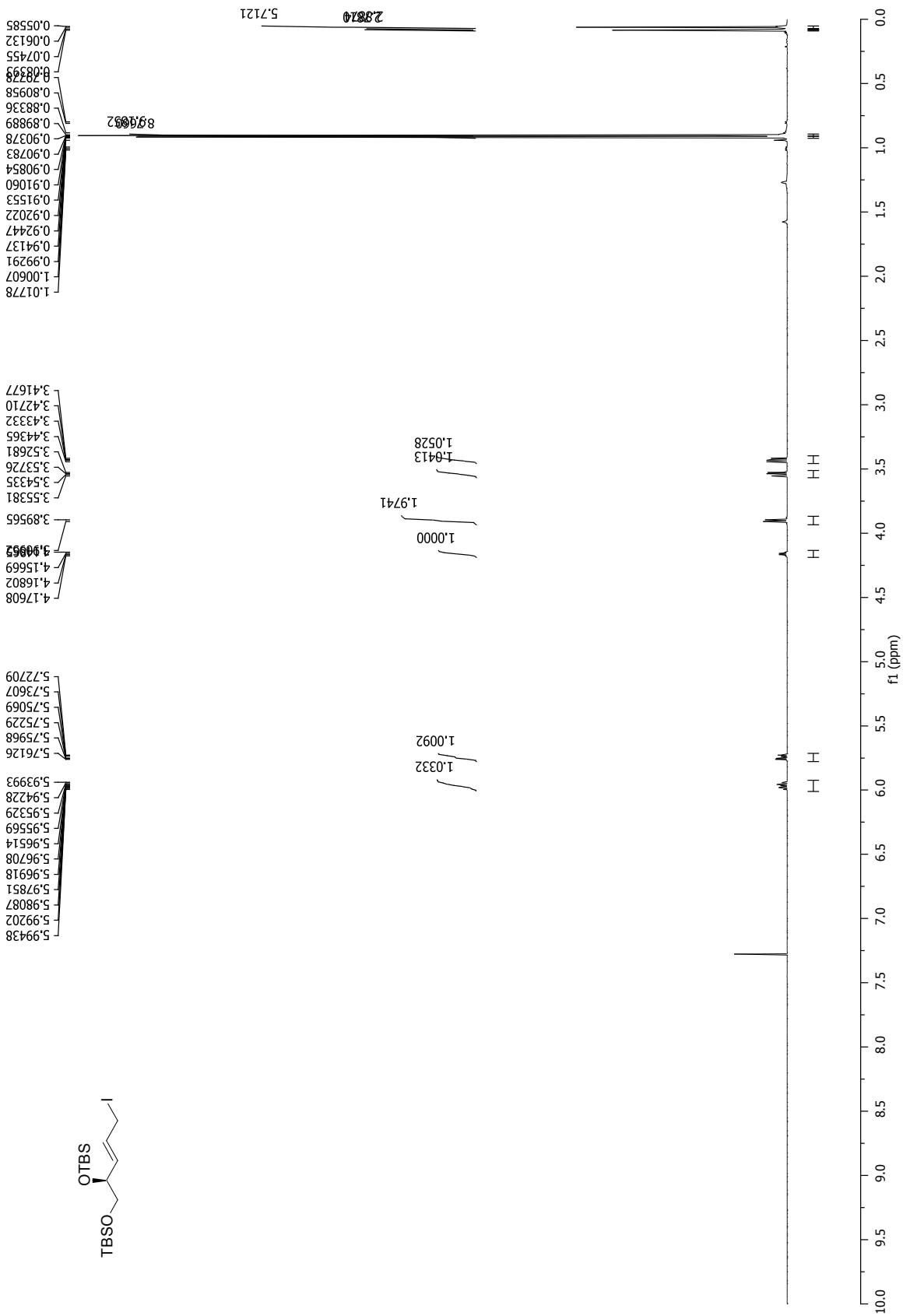
1-Benzyloxy-4-iodobutan-2-ol (entry 16, table 3)

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



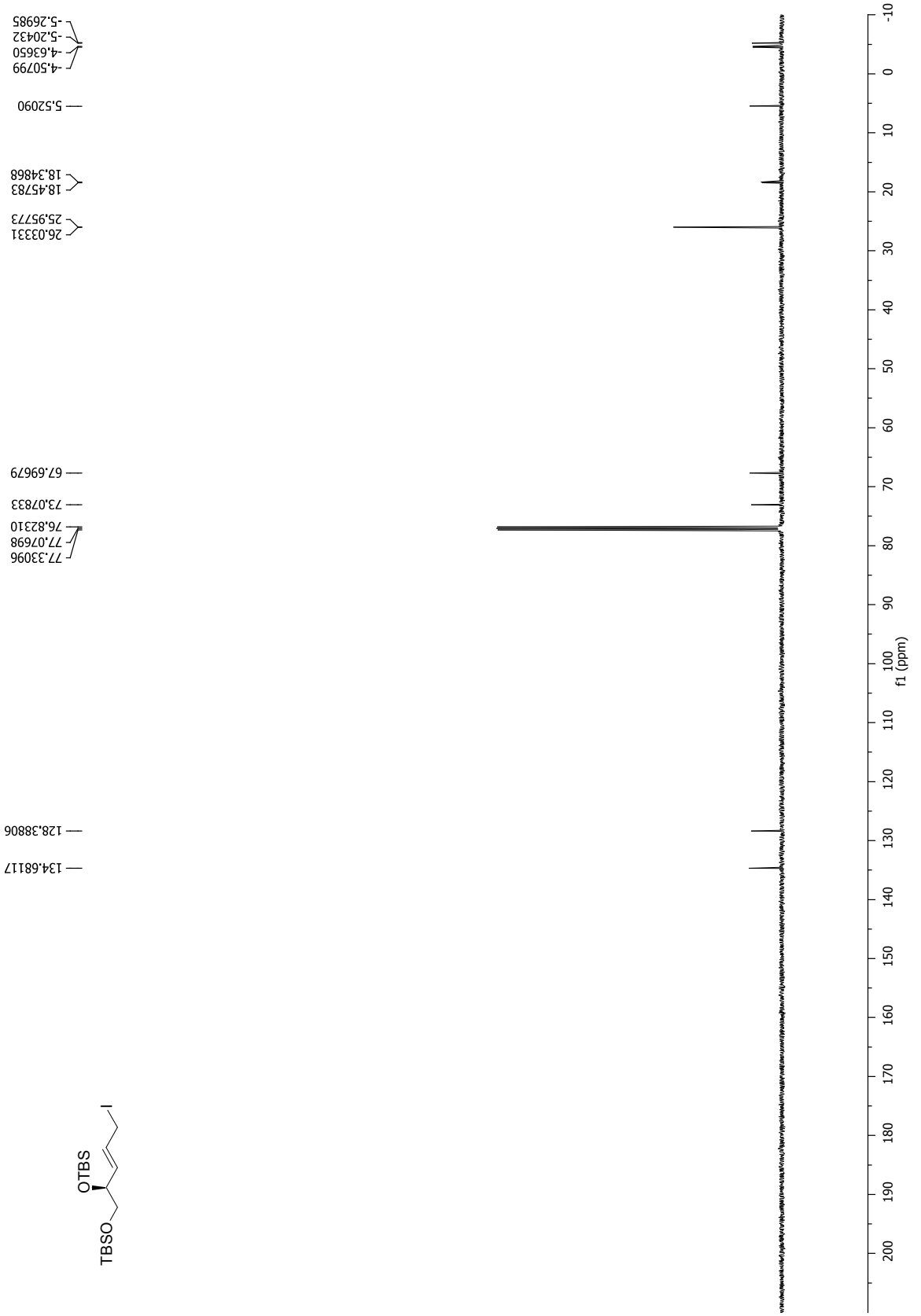
(*R*,*E*)-4,5-Di(*tert*-butyldimethylsilanyloxy)-1-iodopent-2-ene (entry 17, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



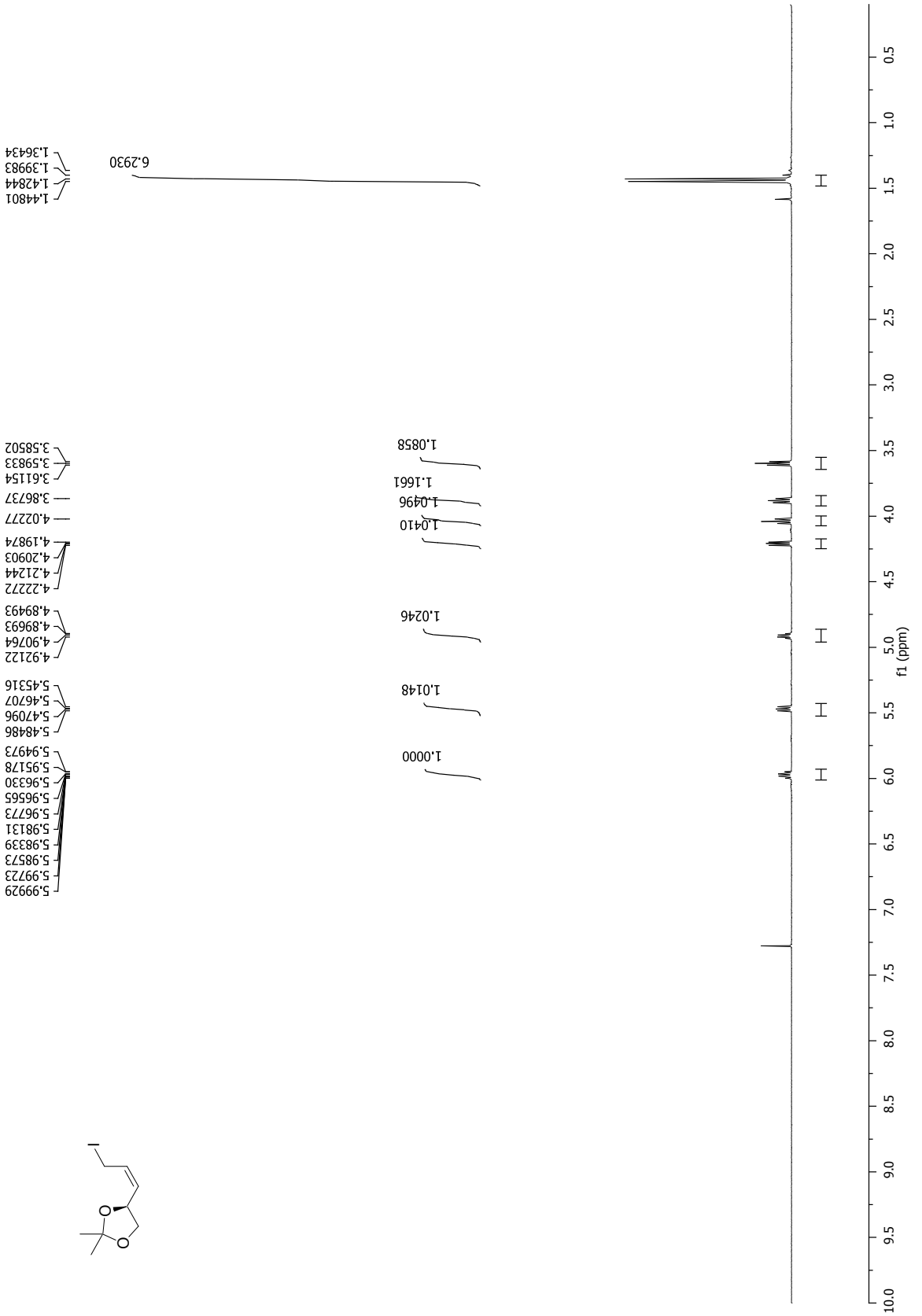
(*R*,*E*)-4,5-Di(*tert*-butyldimethylsilanyloxy)-1-iodopent-2-ene (entry 17, table 3)

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



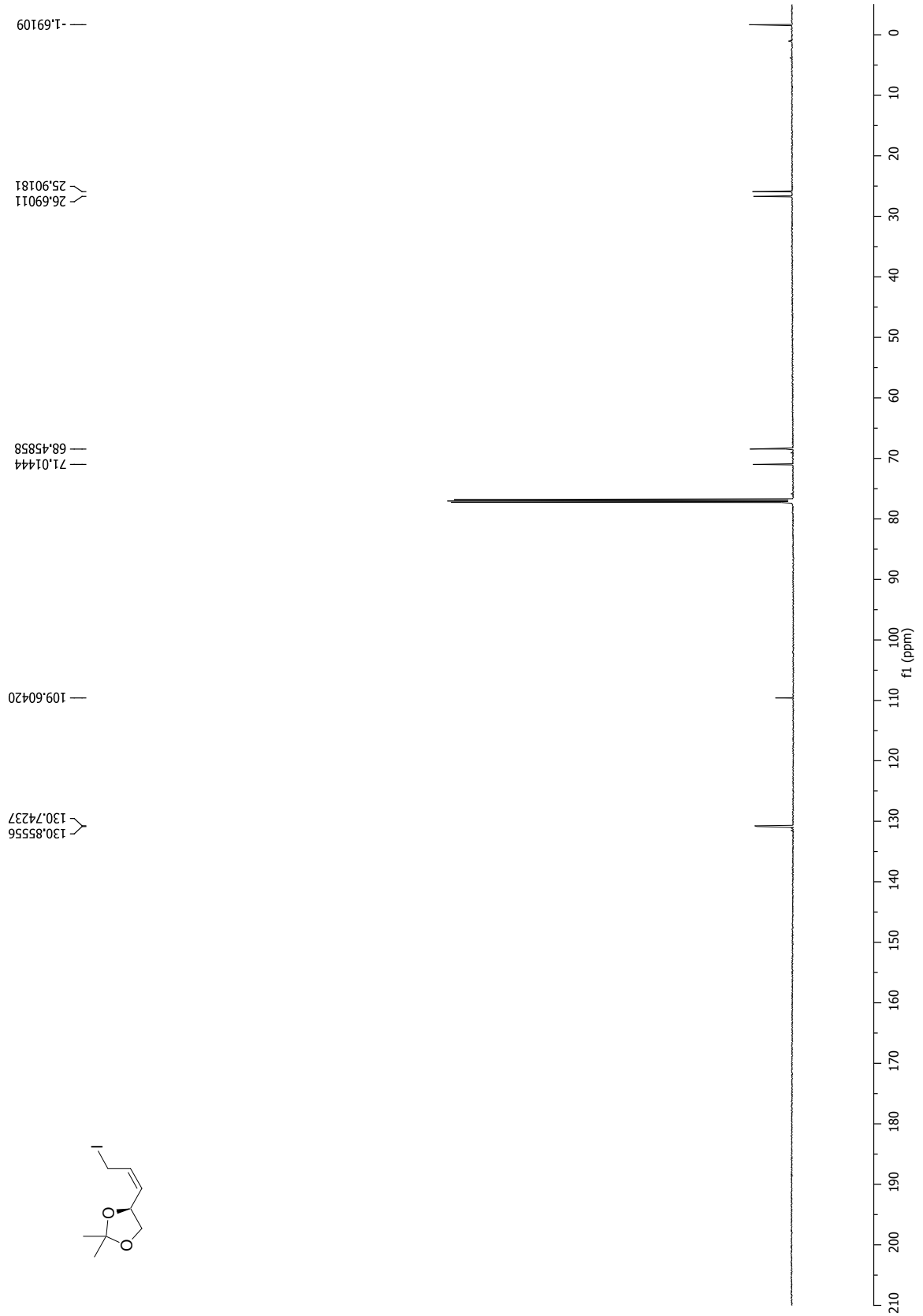
(*R*,*Z*)-1-(2,2-Dimethyl-1,3-dioxolan-4-yl)-3-iodoprop-1-ene (entry 18, table 3)

^{1}H NMR, 600 MHz, $CDCl_3$



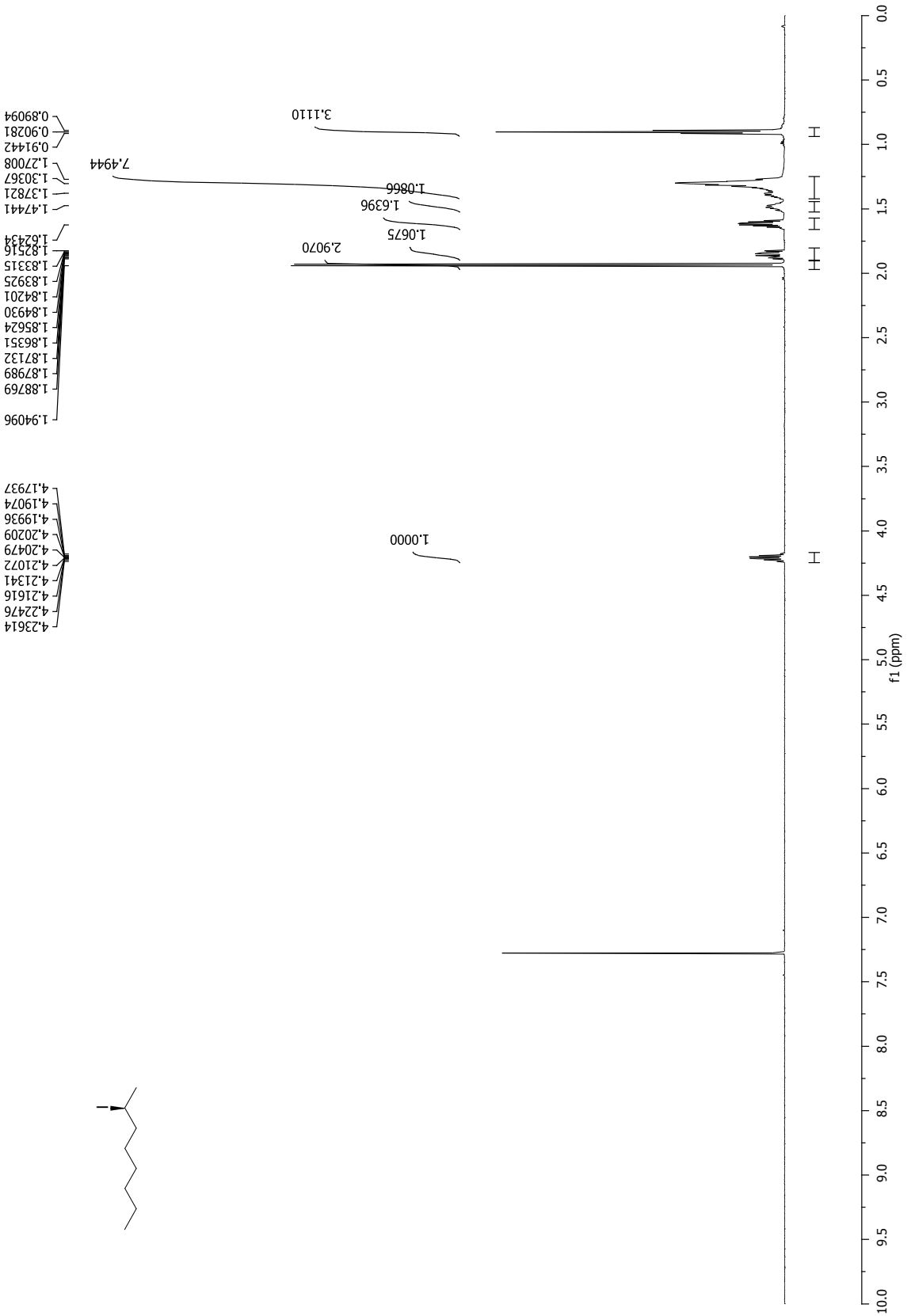
(*R*,*Z*)-1-(2,2-Dimethyl-1,3-dioxolan-4-yl)-3-iodoprop-1-ene (entry 18, table 3)

^{13}C NMR, 150 MHz, $CDCl_3$



(*S*)-2-Iodooctane (14)

^{1}H NMR, 600 MHz, $CDCl_3$



(*S*)-2-Iodooctane (14)

^{13}C NMR, 150 MHz, $CDCl_3$