ANODIC COUPLING REACTIONS: PROBING THE STEREOCHEMISTRY OF TETRAHYDROFURAN FORMATION. A SHORT, CONVENIENT SYNTHESIS OF LINALOOL OXIDE

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Supporting Information

Example experimental procedure for electrolysis reaction; spectroscopic and analytical data for **5, 7, 8, 17a-f, 18a-f, 20** and ^{1}H NMR NOE data for **5, 18a, 18b, 18e, 20**. (43pages).

A. Example experimental procedure for electrolysis reaction.

A solution of 356 mg (1.18 mmol) tetraethyl ammonium p-toluene sulfonate in 40 mL of 30% MeOH/THF containing 220 mg (1.18 mmol) of enol ether **(17a)** and 0.82 mL (7.08 mmol) 2,6-lutidine was placed in a three necked round bottom flask equipped with a retriculated vitreous carbon anode (suspended from a carbon rod), a platinum wire cathode, and a nitrogen inlet. The reaction was degassed via sonnication (10 min), and electrolyzed at a constant current of 8.0 mA until 227.7 C (2.0 Faradays per mole) of charge had been passed. When complete, the solvent was removed in vacuo and the residue was chromatographed through 100 mL of silica gel packed with 20% ethyl ether in hexane. Elution with the same solvent led to the isolation of 214 mg (84%) of **18a** as a 5:1 *trans*/*cis* mixture**.**

B. Characterization Data for Electrolysis Substrates

1-methoxy-2, 6, 6-trimethyl-(E,Z)-1-hepten-5-ol (17a) (TLC (Et_2O/Hexane (1:4)) R_f=0.2; The spectrum of the major compound was reported here. ^1H NMR ($CDCl_3$/300MHz) δ 5.84(s, 1H,), 3.55 (s, 3H), 3.17(d, 1H, J=10.5Hz), 2.13(ddd, 1H, J=5.1, 8.7, 13.5Hz), 1.93(ddd, 1H, J=7.2, 7.5, 14.1Hz), 1.68-1.57(m, 1H), 1.60(s, 3H), 1.53(s, 1H), 1.37-1.26(m, 1H), 0.89(s, 9H); ^{13}C NMR($CDCl_3$/75MHz) δ 142.1, 113.5, 79.2, 59.2, 34.8, 31.2, 29.5, 25.7, 12.6; IR (neat/NaCl) 3428, 2852, 1686, 1204, 1129, 1069cm^{-1}; LRMS (EI) *m/z* 186(M^+, 60), 155(M-OCH_3, 65), 137(M-tBu, 60), 129(70), 111(90), 85(100) ; HRMS (EI) $C_{11}H_{22}O_2(M^+)$ calcd 186.1620, found 186.1615.

2, 6-dimethyl-1-methoxy-(E,Z)-1-hepten-5-ol (17b): (TLC (Et_2O/Hexane (1:2)) R_f=0.30; ^1H NMR ($CDCl_3$/300MHz) δ 5.81(q, 1H, J=1.2Hz), 3.53(s, 3H), 3.32(ddd, 1H, J=3.0, 5.1, 8.7Hz), 2.09-1.86(m, 2H), 1.68-1.28(m, 4H), 1.58(d, 3H, J=1.2Hz), 0.90(d, 3H, J=6.6Hz), 0.89(d, 3H, J=6.6Hz) ; ^{13}C NMR($CDCl_3$/75MHz) δ 142.0, 113.6, 76.2, 59.2, 45.9, 33.6, 32.2, 30.5, 18.7, 17.2; IR (neat/NaCl) 3440, 2958, 1721, 1108, 1052cm^{-1}; LRMS (EI) *m/z* 172(M^+, 30), 141(M-OCH_3, 25), 111(85), 85(100); HRMS (EI) $C_{10}H_{20}O_2(M^+)$ calcd 172.1463, found 172.1454.

1-methoxy-2-methyl-(E,Z)-1-heptene-5-ol (17c): (TLC (Et_2O/Hexane (1:1)) R_f=0.25; ^1H NMR ($CDCl_3$/300MHz) δ 5.82(s, 1H), 3.79(m, 1H), 3.55(s, 3H), 2.67(s, 1H), 2.42(ddd, J=7.5, 9.0, 14.4Hz), 1.92(dt, 1H, Jt=13.5Hz, Jd=6.0Hz), 1.52(s, 3H), 1.54-1.45(m, 2H), 1.17(d, 3H, J=6.3Hz); ^{13}C NMR($CDCl_3$/75MHz) δ 141.9, 113.5, 67.8, 59.2, 37.4, 30.2, 23.5, 12.7; IR (neat/NaCl) 2970, 2933, 1724, 1455, 1384, 1099, 1053cm^{-1}; LRMS (EI) *m/z* 144(M^+, 70), 113(M-OCH_3, 75), 85(100); HRMS (EI) $C_8H_{16}O_2(M^+)$ calcd 144.1150, found 144.1153.

1-methoxy-2-methyl- (E, Z)-1-hepten-6-ol (17d): (TLC (Et_2O/Hexane (1:4)) R_f=0.10; ^1H NMR ($CDCl_3$/300MHz) δ 5.77(s, 0.6H), 5.76(s, 0.4H), 3.80(h, 1H, J=6.0Hz), 3.54(s, 1.8H), 3.51(s, 1.2H), 2.13-1.77(m, 3H), 1.58(s, 1.8H), 1.52(s, 1.2H), 1.41-1.38(m, 4H), 1.18(d, 6H, J=6.0Hz) ; ^{13}C NMR($CDCl_3$/75MHz) δ 141.8, 141.6, 114.0, 113.6, 67.9, 59.1, 59.0, 38.6, 37.7, 29.1, 28.4, 24.0, 23.4, 17.0, 12.5; IR (neat/NaCl) 3369, 2932, 1685, 1460, 1204, 1136cm^{-1}; LRMS (EI) *m/z* 158(M^+, 75), 126(M-$HOCH_3$, 60), 108(100), 98(100); HRMS (EI) $C_9H_{18}O_2(M^+)$ calcd 158.1307, found 158.1306.

7, 7-dimethyl-1-methoxy-(E, Z)-1-octen-6-ol (17e): (TLC (Et_2O/Hexane (1:4)) R_f=0.18; ^1H NMR ($CDCl_3$/300MHz) δ 6.29(d, 0.86H, J=12.6Hz), 5.88(dt, 0.14H, J=6.3,

1.5Hz), 4.73(dt, 0.86H, J=12.6Hz, 7.2Hz), 4.35(dt, 0.14H, J=6.3, 7.2Hz), 3.57(s, 0.42H), 3.50(s, 2.58H), 3.18(dd, 1H, J=10.2, 2.4Hz), 2.26-1.89(m, 2H), 1.78-1.18(m, 5H), 0.90(s, 1.26H), 0.89(s, 7.74H); ^{13}C NMR($CDCl_3$/75MHz) δ 147.1, 147.0, 102.9, 102.8, 79.8, 55.9, 55.8, 34.9, 30.8, 28.2, 27.6, 25.7, 25.6; IR (neat/NaCl) 3440, 3056, 2950, 1656, 1463, 1209, 1133cm^{-1}; LRMS (EI) m/z 186(M^+, 16), 155(M-OCH_3, 25), 137(M-OCH_3-H_2O, 75), 129(M-^{t}Bu, 85), 97(100) ; HRMS (EI) $C_{11}H_{22}O_2$(M^+) calcd 186.1620, found 186.1615.

2, 7-dimethyl-1-methoxy-(E,Z)-1-octen-6-ol (17f): (TLC (Et_2O/Hexane (1:4)) R_f=0.2; ^{1}H NMR ($CDCl_3$/300MHz) δ 6.29(dt, 0.75H, J=12.6, 1.2Hz), 5.88(dt, 0.25H, J=1.5, 6.0Hz), 4.73(dt, 0.75H, J=12.6, 7.2Hz), 4.34(dt, 0.25H, J=7.2, 6.0Hz), 3.58(s, 0.75H), 3.50(s, 2.25H), 3.38(m, 1H), 2.17-1.92(m, 2H), 1.70-1.19(m, 6H), 0.92(d, 0.75H, J=6.9Hz), 0.91(d, 2.25H, J=6.9Hz), 0.90(d, 2.25H, J=6.9Hz), 0.87(d, 0.75H, J=6.9Hz); ^{13}C NMR($CDCl_3$/75MHz) δ 147.1, 146.2, 106.6, 102.8, 76.5, 76.5, 59.4, 55.8, 33.5, 33.4, 30.0, 29.0, 27.7, 27.1, 22.6, 18.8, 17.1, 14.1; IR (neat/NaCl) 3391, 2956, 1656, 1462, 1209, 1130cm^{-1}; LRMS (EI) *m/z* 172(M^+, 34), 140(70), 129(100); HRMS (EI) $C_{10}H_{20}NO_2$(M^+) calcd 172.1463, found 172.1466.

C. Characterization Data for Electrolysis Products:

2-dimethoxymethyl-2-methyl-5-*tert*-butyltetrahydrofuran (18a): (TLC (Et_2O/Hexane (1:1)) R_f=0.64; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.31(s, 0.83H), 4.28(s, 0.17H), 3.92(t, 0.17H, J=7.0Hz), 3.85(dd, 0.83H, J=9.0, 5.0Hz), 3.79(s, 0.51H), 3.77(s, 2.49H), 3.74(s, 0.51H), 3.72(s, 2.49H), 2.22-2.16(m, 0.83H), 2.05-2.00(m, 0.17H), 1.94-1.88(m, 0.17H), 1.88-1.83(m, 0.83H), 1.79-1.63(m, 2H), 1.24(s, 0.51H), 1.22(s, 2.49H), 0.95(s, 1.53H), 0.94(s, 7.47H); ^{13}C NMR($CDCl_3$/75MHz) δ 110.8, 88.3, 86.4, 84.3, 57.8, 57.5, 57.0, 33.8, 33.6, 33.0, 32.6, 26.5, 26.4, 25.9, 25.8, 23.0, 20.5; IR (neat/NaCl) 2955, 2869, 1464, 1188, 1108, 1085, 734cm^{-1}; LRMS (EI) *m/z* 185(M^+-OCH_3, 35), 159(M-^{t}Bu, 75), 141(M-$CH(OCH_3)_2$,100); HRMS (EI) $C_{11}H_{21}O_2$(M^+-OCH_3) calcd 185.1542, found 185.1545.

2-dimethoxymethyl-2-methyl-5-isopropyltetrahydrofuran (18b): (TLC (Et_2O/Hexane (1:4)) R_f=0.54; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.02(s, 0.8H), 3.98(s, 0.2H), 3.68-3.61(m, 1H), 3.52(s, 0.6H), 3.52(s, 2.4H), 3.50(s, 0.6H), 3.48(s, 2.4H), 2.07-1.98(m, 1H), 1.91-

1.82(m, 1H), 1.71-1.65(m, 1H), 1.63-1.51(m, 2H), 1.17(s, 0.6H), 1.16(s, 2.4H), 0.96(d, 0.6H, J=5.5Hz), 0.86(d, 3H, J=5.5Hz); ^{13}C NMR($CDCl_3$/75MHz) δ 110.6, 85.6, 84.6, 84.3, 57.7, 57.6, 57.5, 56.9, 33.5, 33.0, 32.7, 32.6, 28.9, 28.5, 23.2, 21.4, 19.4, 19.3, 18.2, 18.0; IR (neat/NaCl) 2959, 2868, 1467, 1112, 1085, 733cm^{-1}; LRMS (EI) *m/z* 171(M^+-OCH_3, 46), 159(M-*i*Pr, 36), 139(M-$CH(OCH_3)_2$, 35), 127(100), 109(100), 75(100); HRMS (EI) $C_{10}H_{19}O_2$(M^+-OCH_3) calcd 171.1385, found 171.1379.

2-dimethoxymethyl-2, 5-dimethyltetrahydropyran (18c): (TLC (Et_2O/Hexane (1:1)) R_f=0.50; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.17-4.00(m, 1H), 4.04(s, 0.6H), 3.99(s, 0.4H), 3.53(s, 3.0H), 3.51(s, 1.2H), 3.49(s, 1.8H), 2.15-1.92(m, 2H), 1.66-1.42(m, 2H), 1.24(d, 1.8H, J=6.3Hz), 1.23(d, 1.2H, J=6.0Hz), 1.20(s, 1.8H), 1.19(s, 1.2H) ; ^{13}C NMR($CDCl_3$/75MHz) δ 110.7, 85.0, 84.6, 76.2, 74.9, 58.2, 57.9, 57.5, 57.2, 33.8, 33.7, 33.6, 33.0, 23.7, 22.1, 21.5, 21.1; IR (neat/NaCl) 2968, 2830, 2630, 1451, 1190, 1112, 1080cm^{-1}; LRMS (EI) *m/z* 143(M^+-OCH_3, 30), 99 (M-$CH(OCH_3)_2$, 85), 75(100); HRMS (EI) $C_8H_{15}O_2$(M^+-OCH_3) calcd 143.1072, found 143.1067.

2-dimethoxymethyl-2,6-dimethyltetrahydropyran(18d): (TLC (Et_2O/Hexane (1:4)) R_f=0.36; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.59(s, 0.4H), 3.91(s, 0.6H), 3.82(pd, 0.4H, J=2.4, 6.0Hz), 3.69(pd, 0.6H, J=6.3, 2.1Hz), 3.58(s, 1.2H), 3.53(s, 1.8H), 3.50(s, 1.2H), 3.49(s, 1.8H), 1.92-1.83(m, 0.4H), 1.68-1.47(m, 3.2H), 1.44-1.35(m, 1.6H), 1.35-1.20(m, 0.8H), 1,18(s, 1.8H), 1.15(d, 1.2H, J=6.0Hz), 1.14(s, 1.2H), 1.11(d, 1.8H, J=6.0Hz) ; ^{13}C NMR($CDCl_3$/75MHz) δ 111.8, 105.5, 77.0, 76.0, 67.1, 65.9, 58.2, 57.9, 57.7, 57.6, 33.5, 32.8, 31.7, 29.6, 22.9, 22.6, 22.5, 19.6, 19.2, 15.1; IR (neat/NaCl) 2966, 2932, 1085cm^{-1}; LRMS (EI) *m/z* 157(M^+-OCH_3, 15), 113(M-$CH(OCH_3)_2$, 100), 75(100); HRMS (EI) $C_9H_{17}O_2$(M^+-OCH_3) calcd 157.1229, found 157.1230.

2-dimethoxymethyl-6-*tert*-butyltetrahydropyran (18e): (TLC (Et_2O/Hexane (1:1)) R_f=0.48; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.75(d, 0.28H, J=6.5Hz), 4.14(d, 0.72H, J=4.5Hz), 3.85-3.82(m, 0.28H), 3.39(s, 2.16H), 3.38(s, 2.16H), 3.37(s, 0.84H), 3.36(s, 0.84H), 3.38-3.34(m, 0.72H), 3.15(d, 0.28H, J=9.5Hz), 2.82(d, 0.72H, J=9.5Hz), 1.88-1.86(m, 0.72H), 1.70-1.15(m, 5.28H), 0.87(s, 6.48H), 0.86(2.52H); ^{13}C NMR($CDCl_3$/75MHz) δ 106.3, 100.4, 85.6, 78.9, 78.1, 71.2, 55.5, 54.8, 53.6, 50.7, 34.1, 26.4, 26.0, 25.8, 25.2, 25.1, 24.5, 23.3, 19.6; IR (neat/NaCl) 2953, 2863, 1466, 1363, 1093, 1052, 914cm^{-1}; LRMS

(EI) *m/z* 185(M^+-OCH_3, 10), 159(M-^{t}Bu, 20), 141(M-$CH(OCH_3)_2$, 30), 127(100); HRMS (EI) $C_{11}H_{21}O_2$(M^+-OCH_3) calcd 185.1542, found 185.1543.

2-dimethoxymethyl-6-isopropyltetrahydropyran (18f): (TLC (Et_2O/Hexane (1:4)) R_f=0.39; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.46(d, 0.36H, J=6.6Hz), 4.18(d, 0.64H, J=5.7Hz), 4.22(dd, 0.36H, J=5.7, 3.6Hz), 3.41(s, 1.08H), 3.40(s, 1.92H), 3.40(s, 1.92H), 3.39(s, 1.08H), 3.38-3.31(m, 0.64H), 3.29-3.26(m, 0.36H), 2.95(ddd, 0.64H, J=11.1, 6.9, 2.1Hz), 1.90-1.14(m, 7H), 0.97(d, 1.08H, J=7.2Hz), 0.95(d, 1.92H, J=6.9Hz), 0.87(d, 1.92H, J=6.9Hz),).85(d, 1.08H, J=7.2Hz); ^{13}C NMR($CDCl_3$/75MHz) δ 105.9, 103.3, 83.5, 83.2, 77.9, 77.7, 55.2, 54.9, 53.6, 52.7, 33.1, 33.0, 28.8, 28.0, 26.9, 26.5, 23.4, 23.1, 19.0, 18.9, 18.7, 18.4; IR (neat/NaCl) 2950, 2939, 1469, 1103, 910cm^{-1}; LRMS (EI) *m/z* 171(M^+-OCH_3, 5), 122(35), 104(77), 71(100); HRMS (EI) $C_{10}H_{19}O_2$(M^+-OCH_3) calcd 171.1385 found 171.1386.

D. Characterization Data for the Synthesis of Linalool Oxide

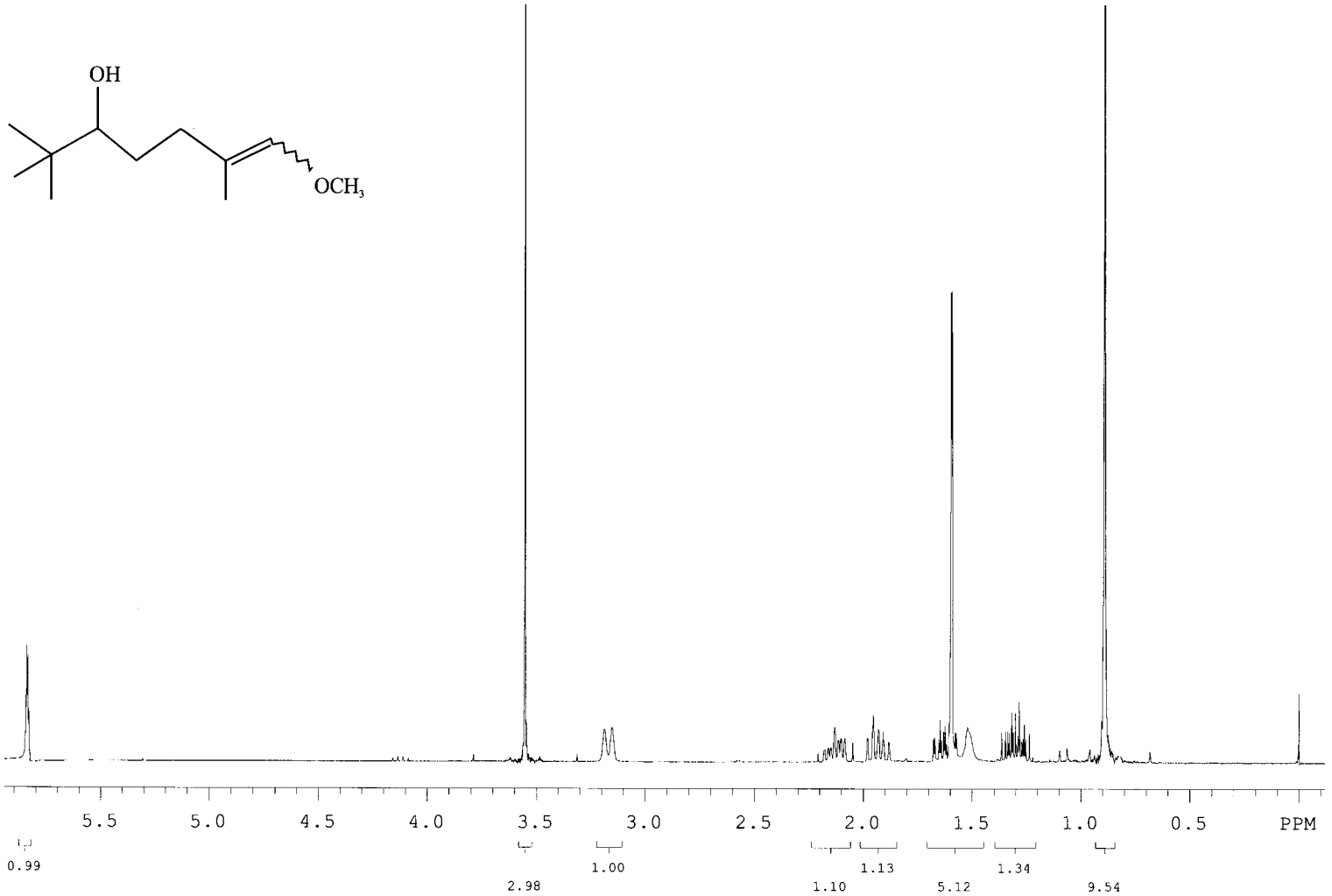
(5s)-2, 6-dimethyl-1-methoxy-(E, Z)-1-hepten-5, 6-diol (8): (TLC (EtOAc/Hexane (1:1)) R_f=0.16, 0.22; The spectral data of major isomer was reported here. ^{1}H NMR ($CDCl_3$/300MHz) δ 5.83 (s, 1H), 3.56 (s, 3H), 3.33 (dd, 1H, J=5.7, 2.1Hz), 3.26 (bs, 1H), 2.71 (bs, 1H), 2.50 (ddd, 1H, J=13.8, 10.8, 6.3Hz), 1.95 (dt, 1H, J=13.8, 5.4Hz), 1.60-1.36 (m, 1H), 1.53 (s, 3H), 1.18 (s, 3H), 1.15 (s, 3H) ; ^{13}C NMR($CDCl_3$/75MHz) δ 141.7, 112.7, 76.2, 72.6, 58.8, 30.6, 29.4, 26.1, 22.8, 12.3; IR (neat/NaCl) 3407, 2930, 1682, 1205cm^{-1}; LRMS (EI) *m/z* 188(M^+, 40), 171(M-OH, 40), 157(M-OCH_3, 35), 98(90), 85(100); HRMS (EI) $C_{10}H_{20}O_3$(M^+) calcd 188.1412, found 188.1414.

(2S(R), 5S)-2-dimethoxymethyl-5-(1-hydroxy-1-methylethyl)-2-methyltetraahydrofuran (20): TLC (EtOAc/Hexane (1:1)) R_f=0.36; ^{1}H NMR ($CDCl_3$/300MHz) δ 4.16(s, 0.12H), 4.04(s, 0.88H), 3.87(t, 0.12H, J=6.0Hz), 3.77(dd, 0.88H, J=7.5, 4.5Hz), 3.52(s, 3H), 3.51(s, 0.36H), 3.48(s, 2.64H), 2.18(bs, 1H), 2.13-2.05(m, 1H), 1.84-1.74(m, 1.12H), 1.63-1.59(m, 0.88H), 1.24(s, 0.36H), 1.21(s, 3H), 1.19(s, 2.64H), 1.13(s, 2.64H), 1.09(s, 0.36H); ^{13}C NMR($CDCl_3$/75MHz) δ 110.5, 109.4, 86.8, 85.9, 85.0, 71.3, 70.3, 57.7, 56.9, 56.3, 33.1, 32.5, 27.8, 27.4, 26.2, 25.8, 24.8, 24.0, 23.2, 22.9; IR (neat/NaCl) 3473, 2975, 1464, 1372, 1108, 1082cm^{-1}; LRMS (EI) *m/z*

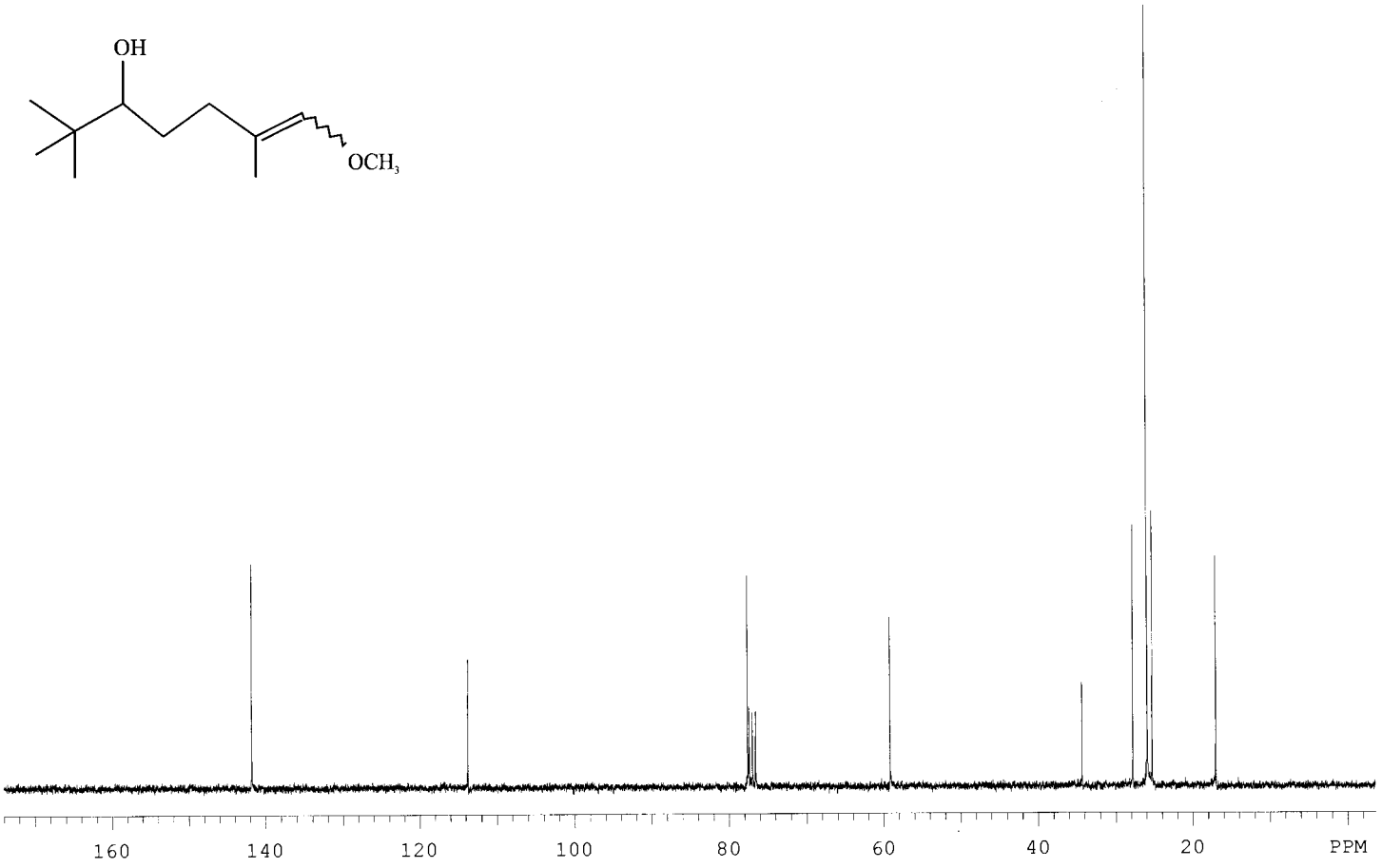
201(M^+-OH, 85), 187(M-OCH_3, 100), 143 (M-$CH(OCH_3)_2$,100); HRMS (EI) $C_{11}H_{21}O_3$(M^+-OH) calcd 201.1491, found 201.1502.

(2S, 5S)-2-hydrocarbonyl-5-(1-hydroxyl-1-methylethyl)-2-methyltetrahydrofuran (7): TLC (Et_2O/Hexane (1:4)) R_f=0.10; ^{1}H NMR ($CDCl_3$/300MHz) δ 9.57(s, 1H), 3.86(dd, 1H, J=8.4, 6.6Hz), 2.15(dt, 1H, J=12.3, 8.1Hz), 1.97-1.82(m, 2H), 1.75-1.67(m, 1H), 1.31(s, 3H), 1.27(s, 3H), 1.16(s, 3H) ; ^{13}C NMR($CDCl_3$/75MHz) δ 202.7, 87.5, 86.5, 70.7, 32.7, 27.3, 25.8, 24.3, 21.0; IR (neat/NaCl) 3452, 2976, 1734, 1033cm^{-1}; LRMS (EI) *m/z* 155(M^+-OH, 80), 143(M-CHO, 86), 125(40), 85(70), 71(100); HRMS (EI) $C_8H_{15}O_2$(M^+-CHO) calcd 143.1072, found 143.1073.

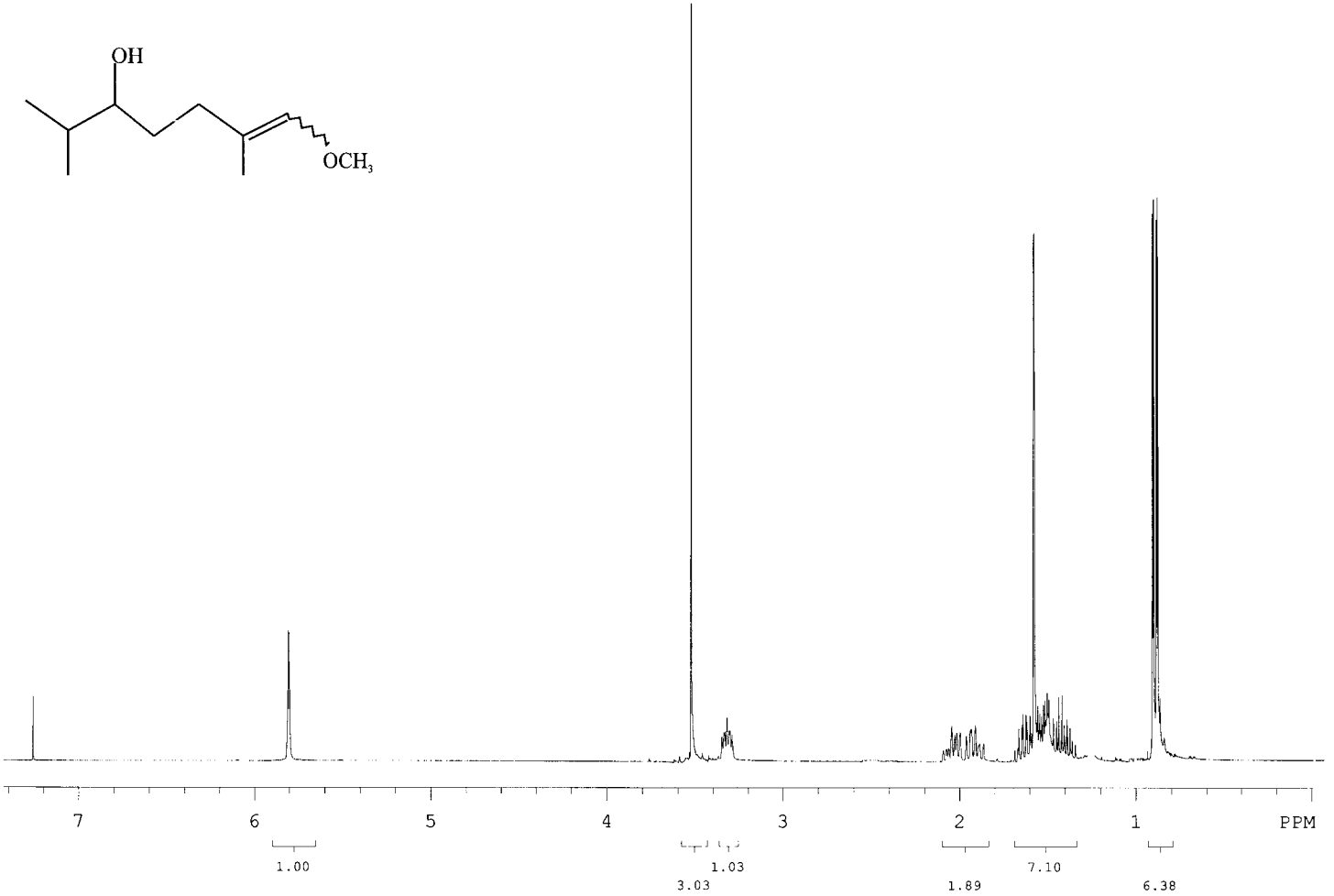
(2S, 5S)-5-(1-hydroxyl-1-methylethyl)-2-methyl-2-vinyltetrahydrofuran (5): $[\alpha]_D^{20}$=+8.33 (C=0.6, $CHCl_3$); TLC (EtOAc/Hexane (1:4)) R_f=0.25; ^{1}H NMR ($CDCl_3$/500MHz) δ 5.88(dd, 1H, J=14.5, 8.5Hz), 5.19(dd, 1H, J=14.5, 1.5Hz), 5.00(dd, 1H, J=8.5, 1.5Hz), 3.80(t, 1H, J=6.0Hz), 2.21(bs, 1H), 1.93-1.88(m, 1H), 1.84(dd, 2H, J=12, 6.0Hz), 1.75-1.70(m, 1H), .1.32(s, 3H), 1.23(s, 3H), 1.14(s, 3H); ^{13}C NMR($CDCl_3$/75MHz) δ 143.7, 111.3, 85.5, 83.0, 71.1, 37.4, 27.2, 26.8, 26.3, 24.1; IR (neat/NaCl) 3458, 3088, 2975, 1642, 1054cm^{-1}; LRMS (EI) *m/z* 155(M^+-CH_3, 12), 137(M-CH_3-H_2O, 12), 111(M-$C(OH)(CH_3)_2$, 50), 94(70), 59(100); HRMS (EI) $C_9H_{15}O_2$(M^+-CH_3) calcd 155.1072, found 155.1075.



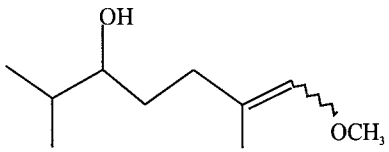
1-methoxy-2,6,6-trimethyl-(E, Z)-1-hepten-5-ol **17a** (in CDCl3)

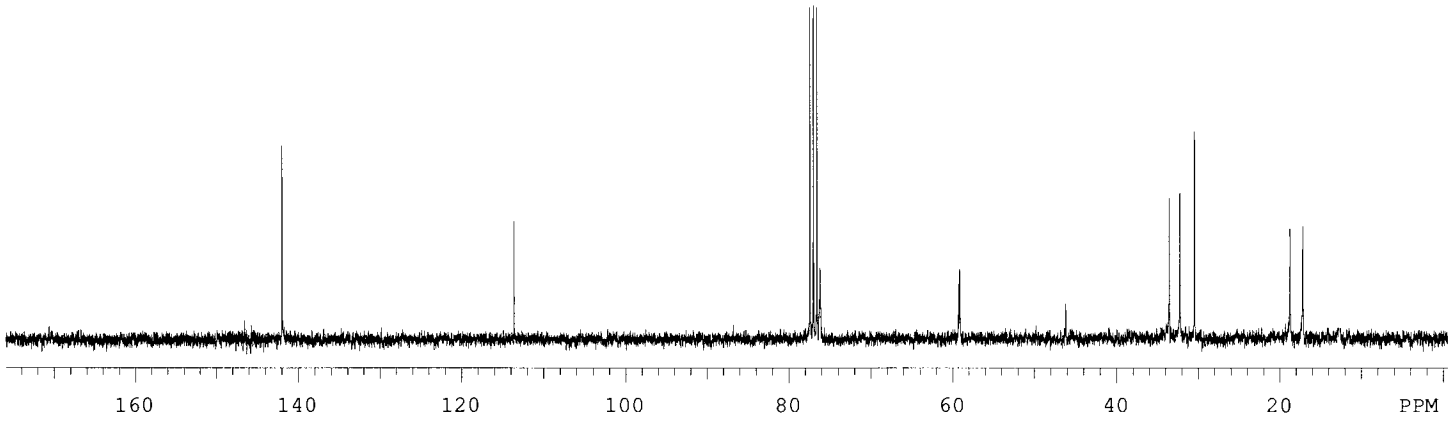


1-methoxy-2,6,6-trimethyl-(E, Z)-1-hepten-5-ol **17a** (in CDCl3)

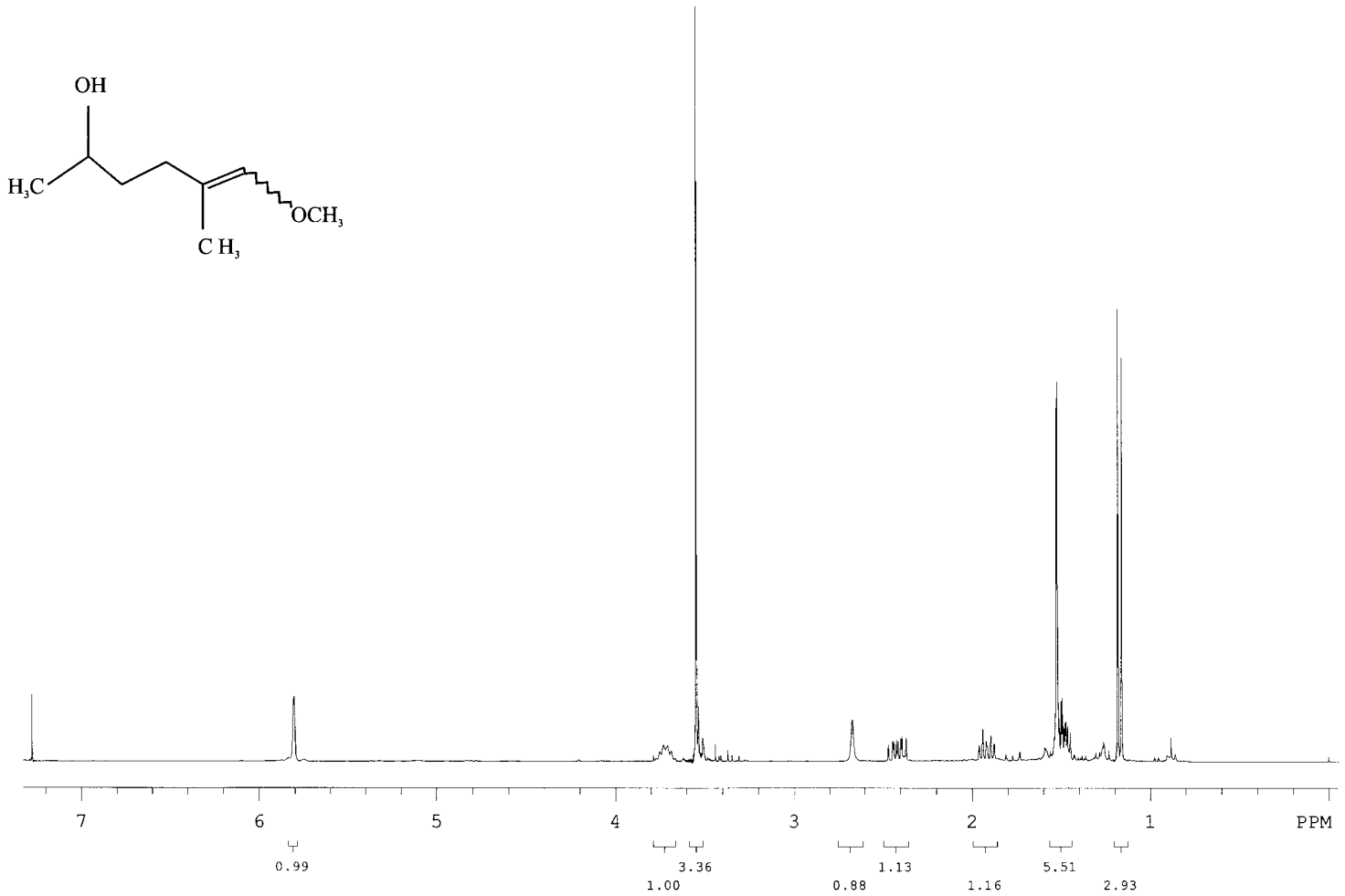


2,6-dimethyl-1-methoxy-(E, Z)-1-hepten-5-ol **17b** (in CDCl3)

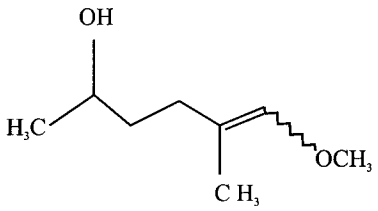


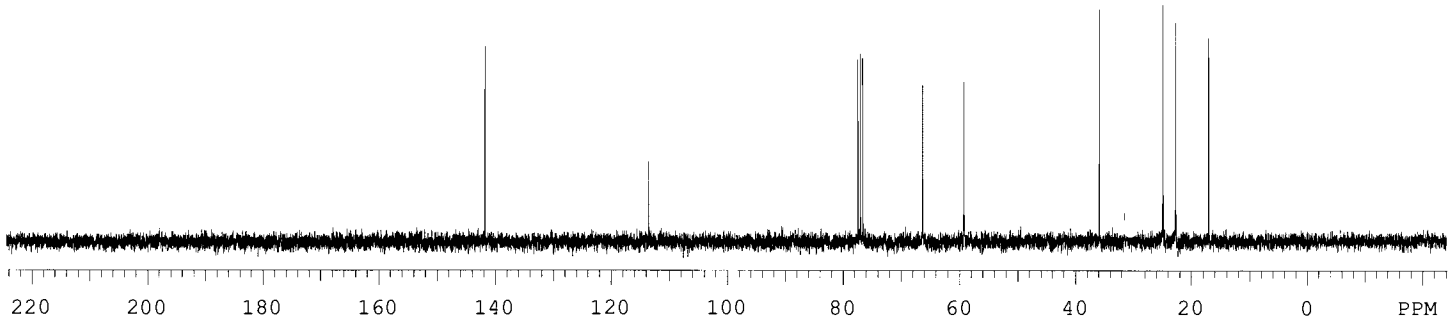


2,6-dimethyl-1-methoxy-(E, Z)-1-hepten-5-ol **17b** (in CDCl3)

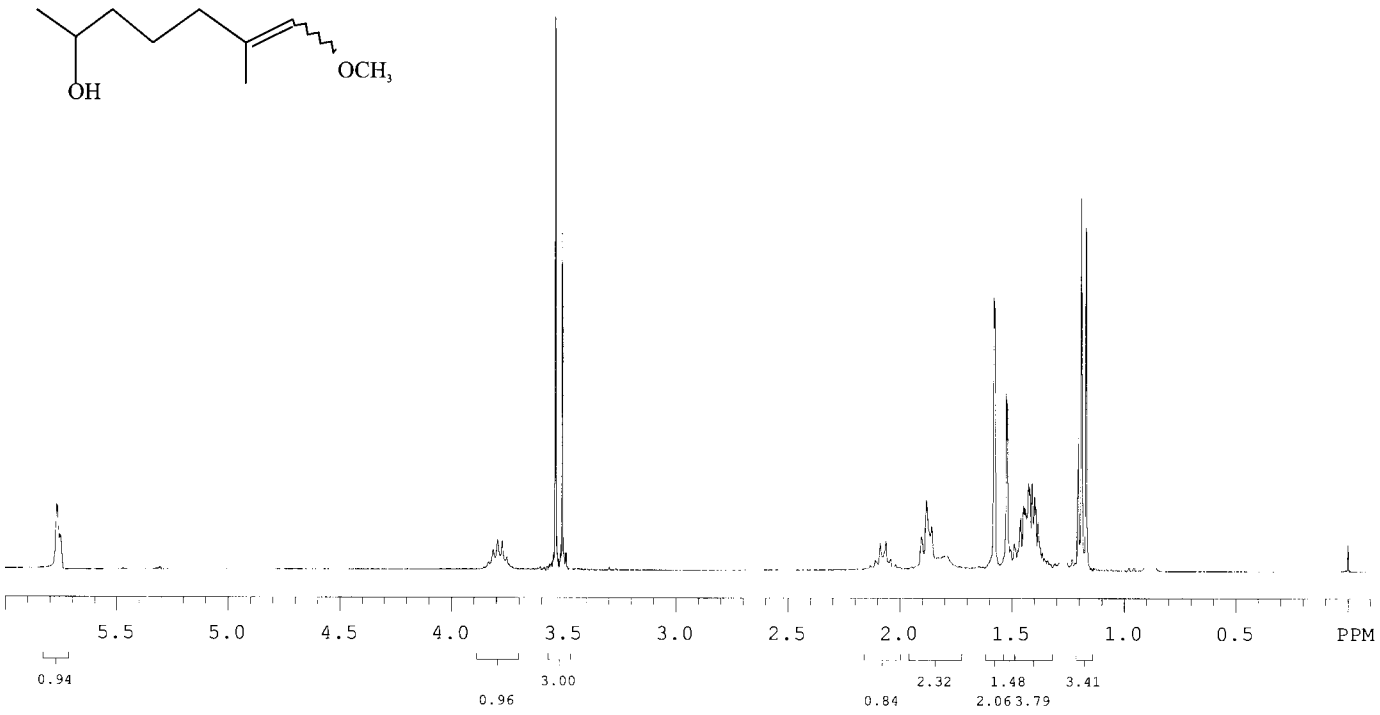


1-methoxy-2-methyl- (E, Z)-1-hepten-5-ol **17c** (in CDCl3)

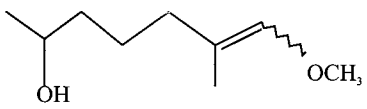


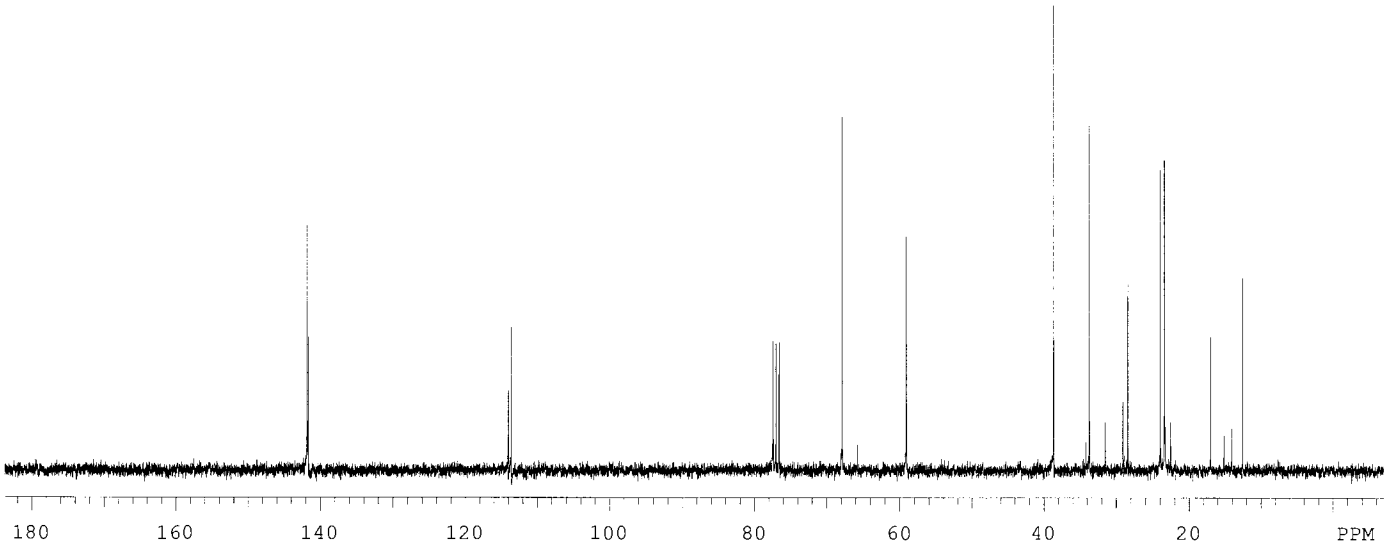


1-methoxy-2-methyl- (E, Z)-1-hepten-5-ol **17c** (in CDCl3)

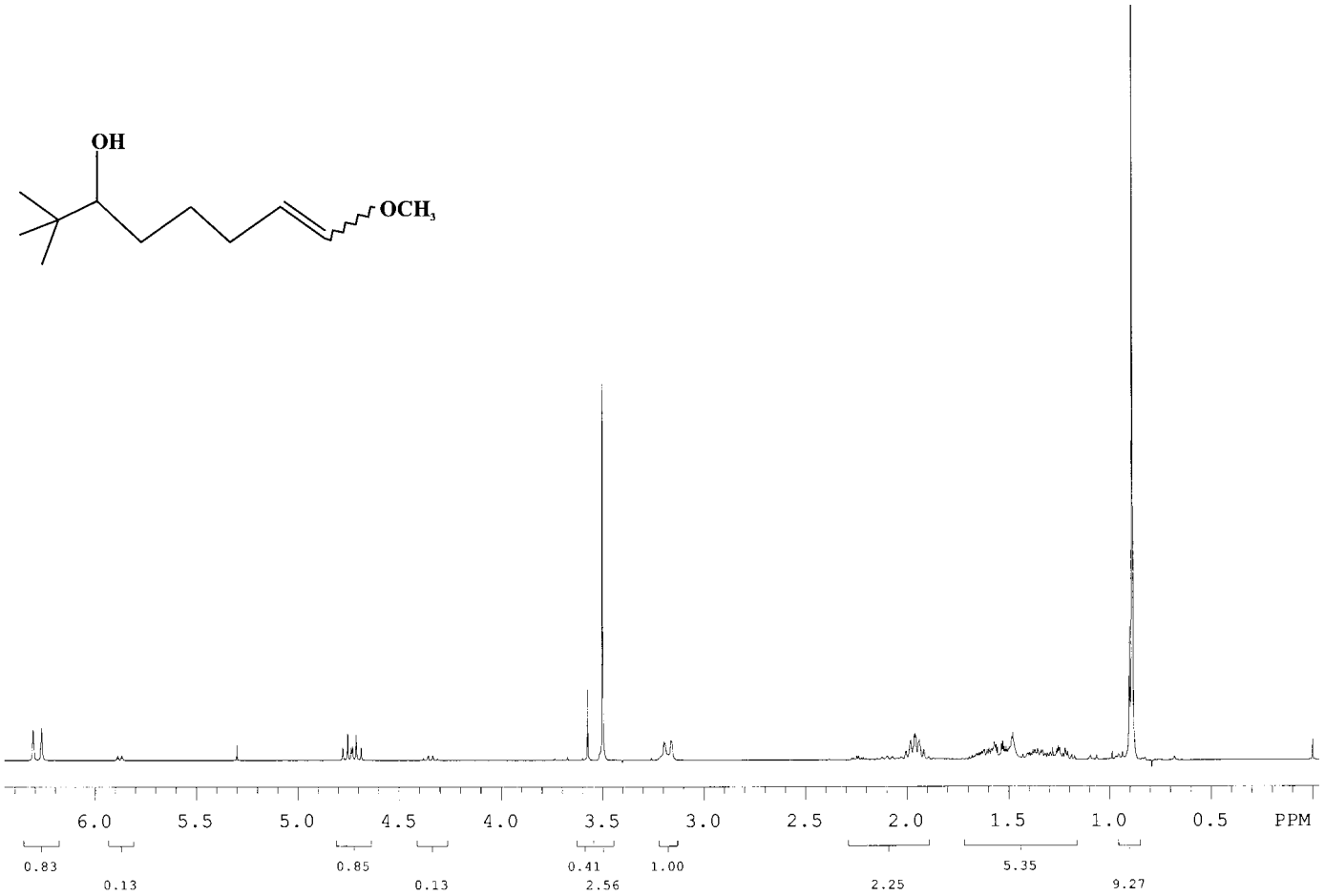


1-methoxy-2-methyl-(E, Z)-1-hepten-6-ol **17d** (in CDCl3)

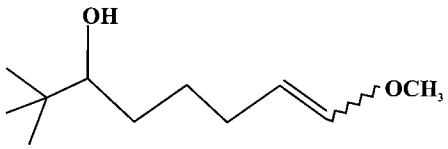


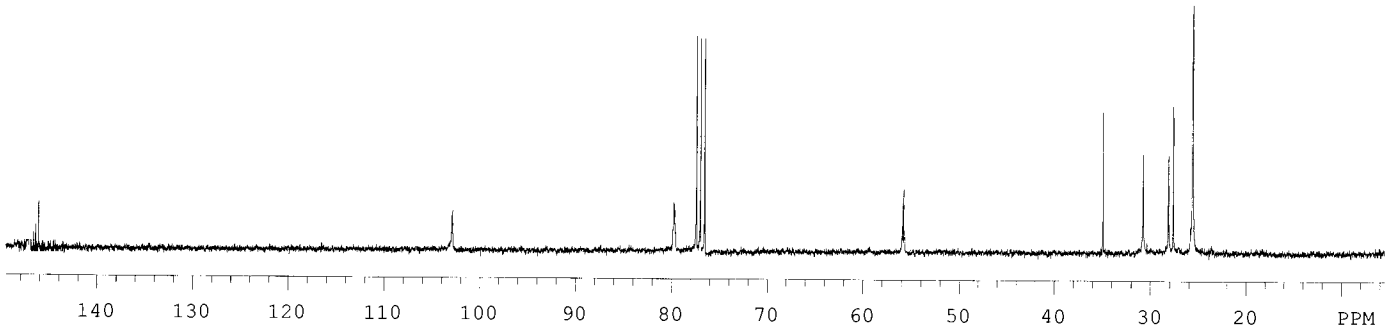


1-methoxy-2-methyl-(E, Z)-1-hepten-6-ol **17d** (in CDCl3)

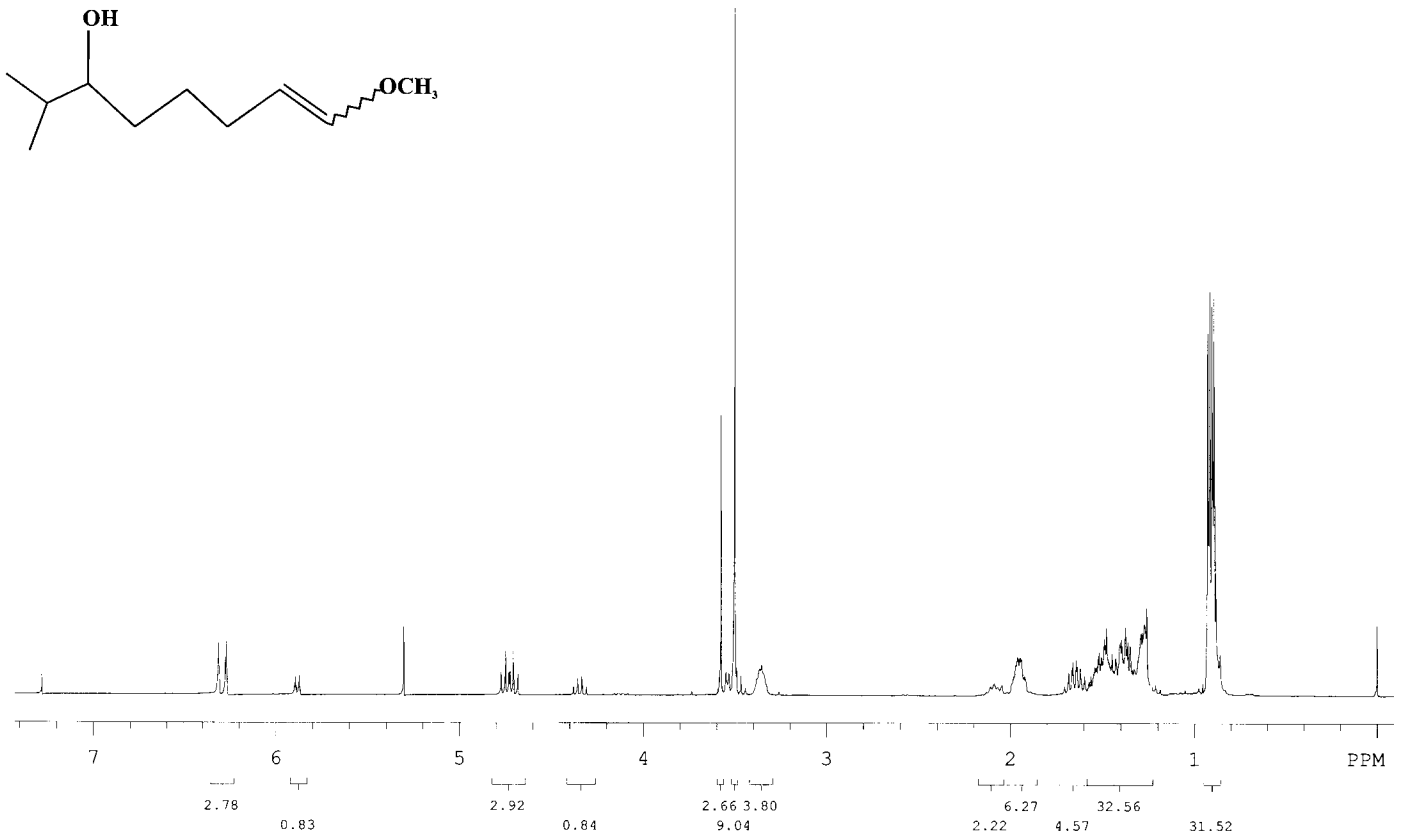


7, 7-dimethyl-1-methoxy-(E, Z)-1-octen-6-ol **17e** (in CDCl3)

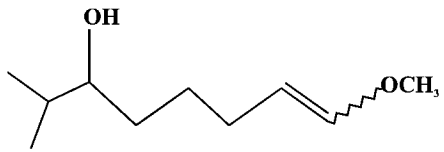


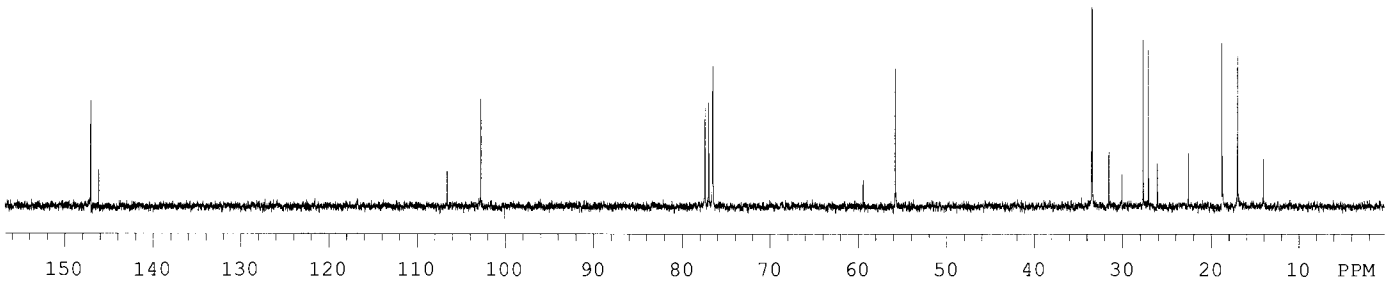


7, 7-dimethyl-1-methoxy-(E, Z)-1-octen-6-ol **17e** (in CDCl3)

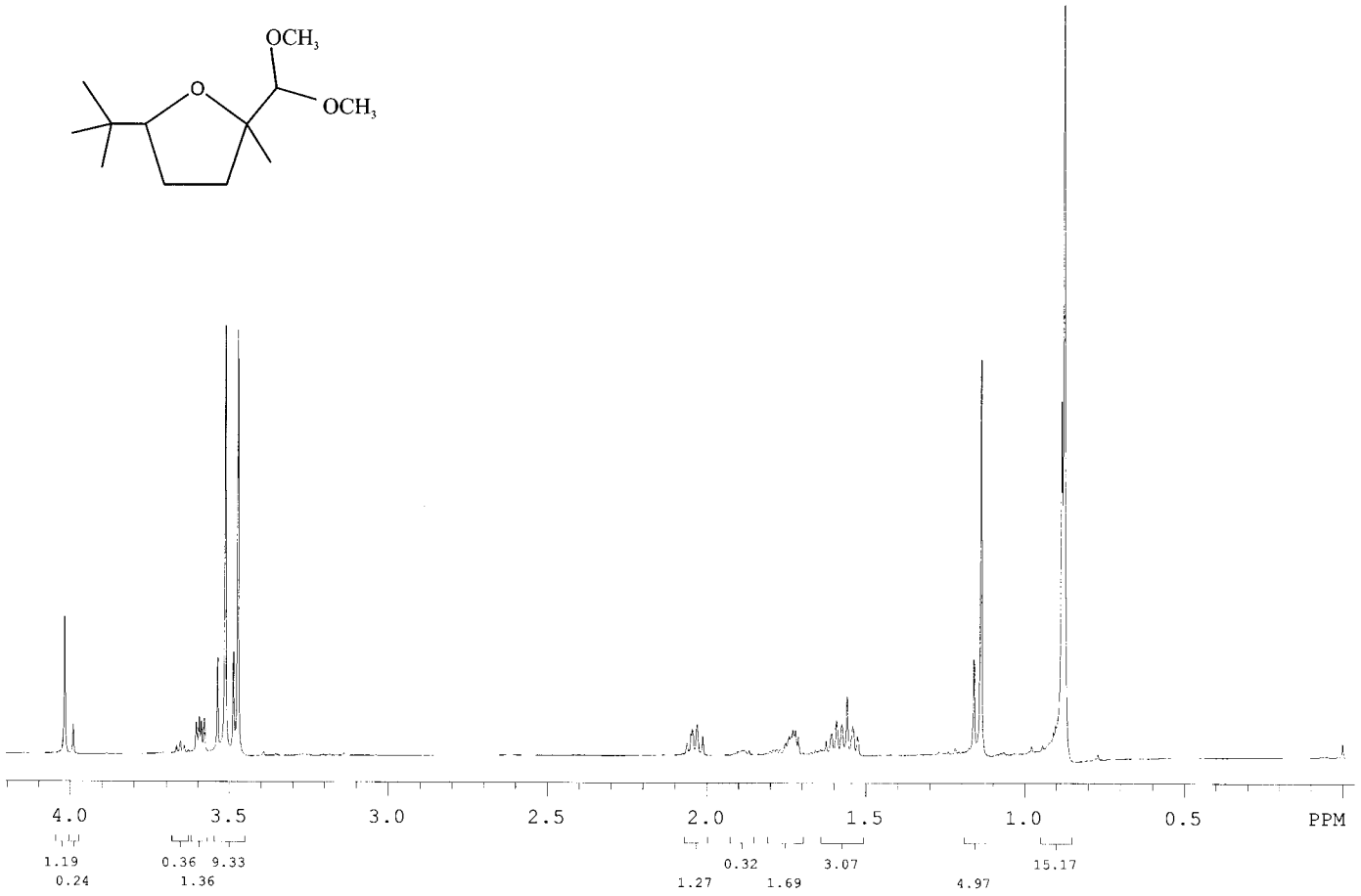


2, 7-dimethyl-1-methoxy- (E, Z)-1-octen-6-ol **17f** (in CDCl3)

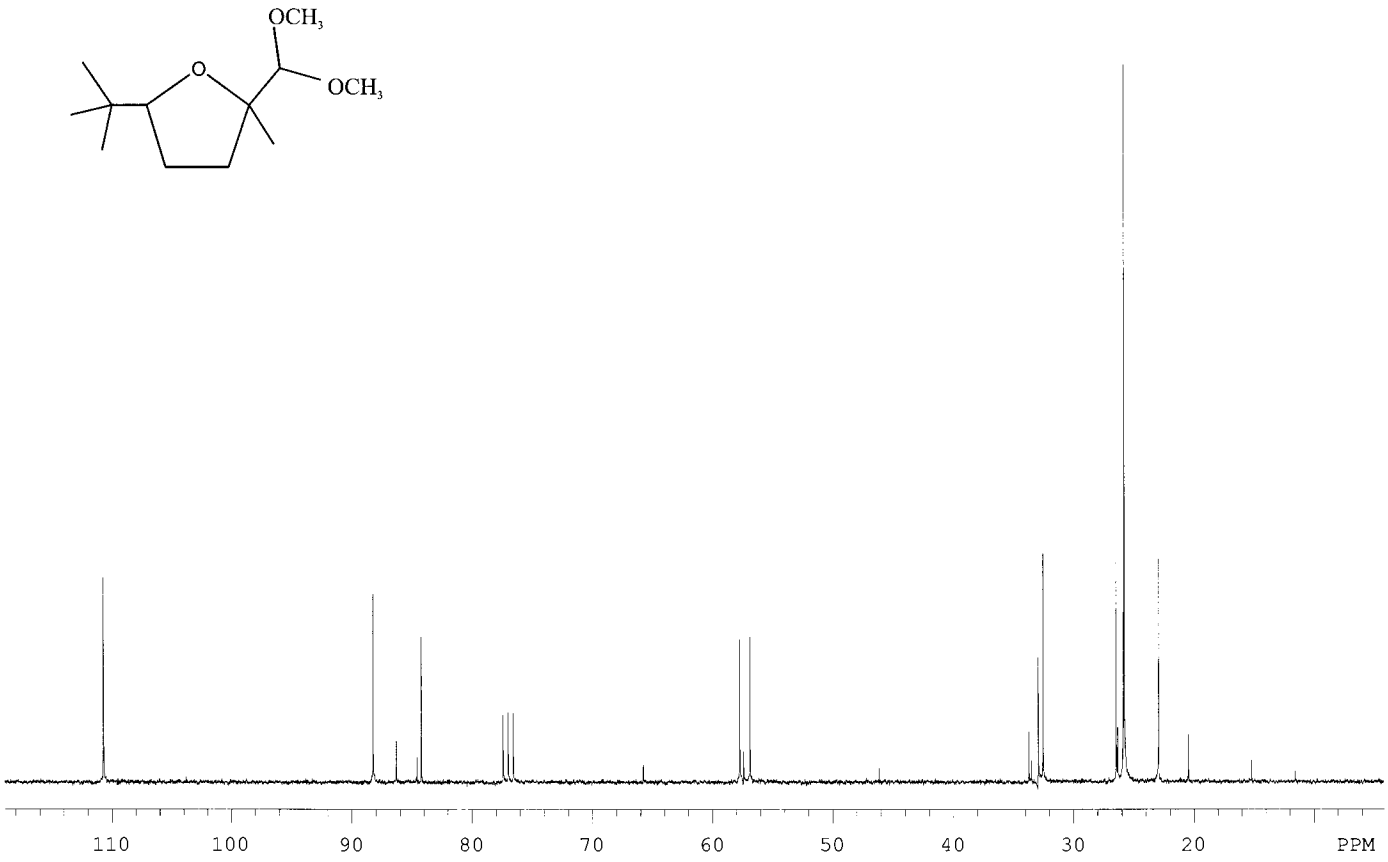




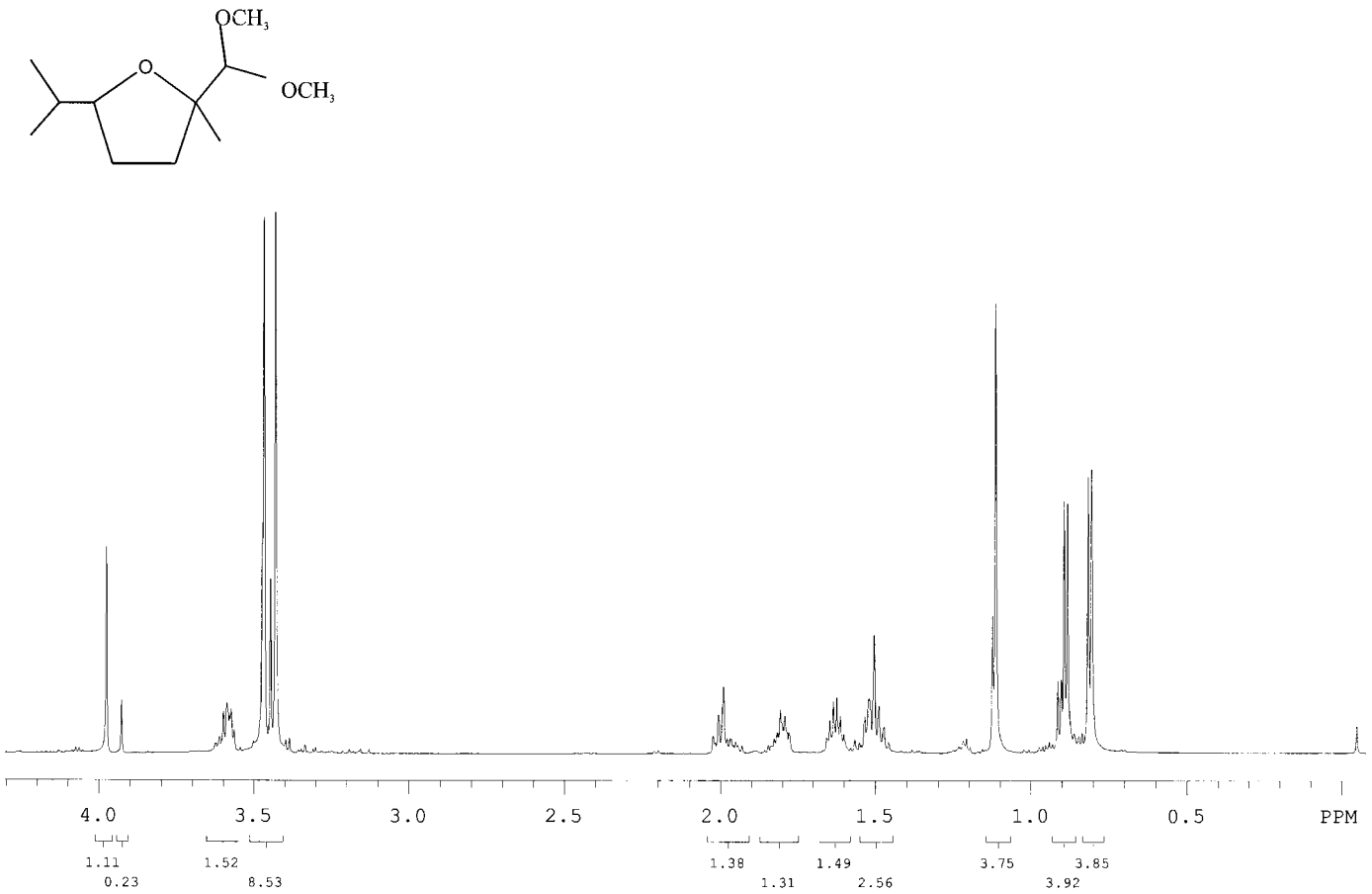
2, 7-dimethyl-1-methoxy- (E, Z)-1-octen-6-ol **17f** (in CDCl3)



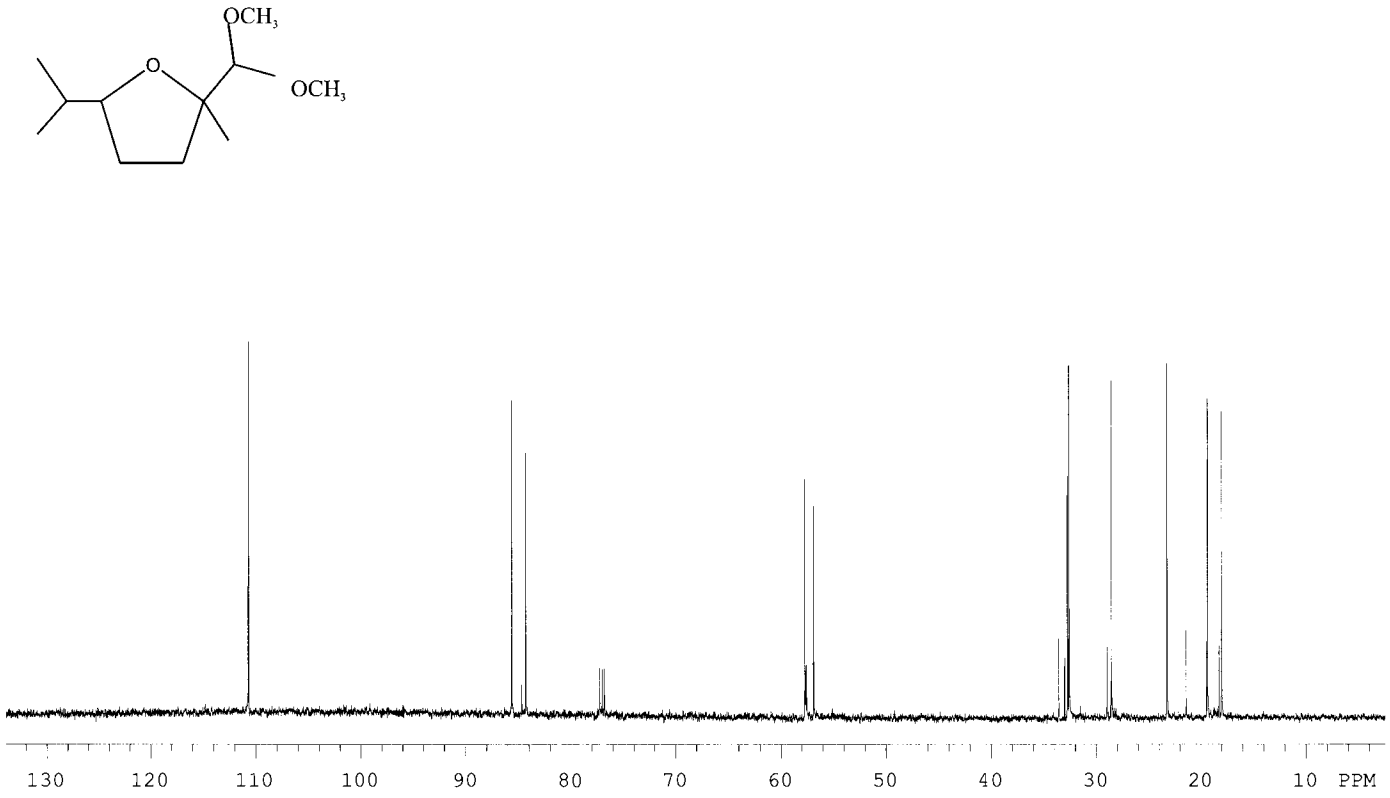
2-dimethoxymethyl-2-methyl-5-*tert*-butyl tetrahydrofuran **18a** (in CDCl3)



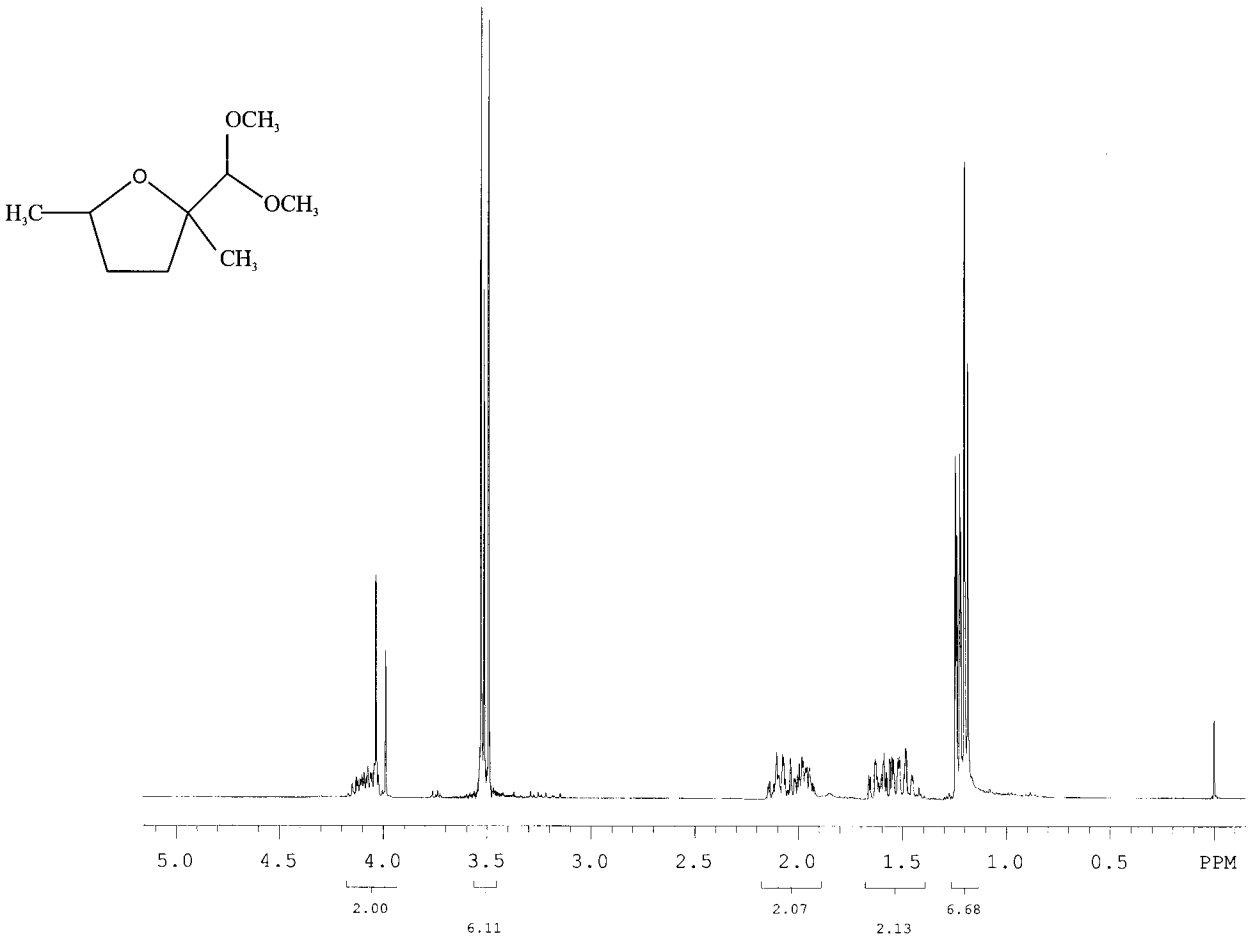
2-dimethoxymethyl-2-methyl-5-*tert*-butyl tetrahydrofuran **18a** (in CDCl3)



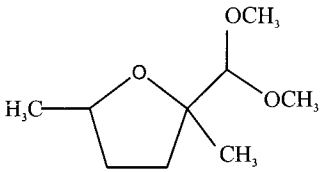
2-dimethoxymethyl-2-methyl-5-isopropyl tetrahydrofuran **18b** (in CDCl3)

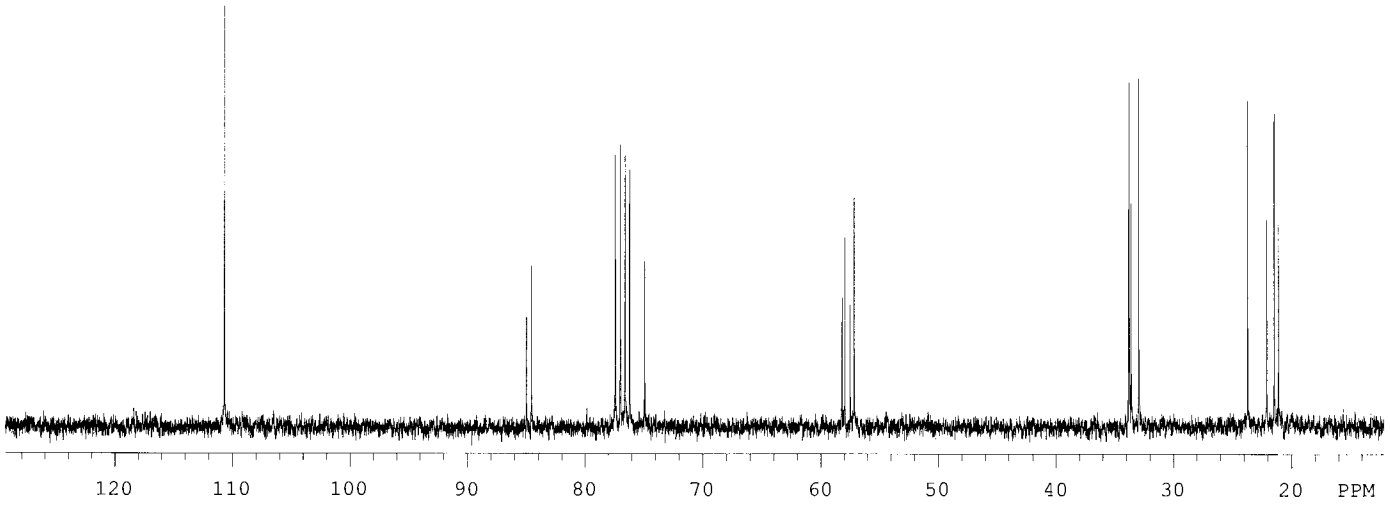


2-dimethoxymethyl-2-methyl-5-isopropyl tetrahydrofuran **18b** (in CDCl3)

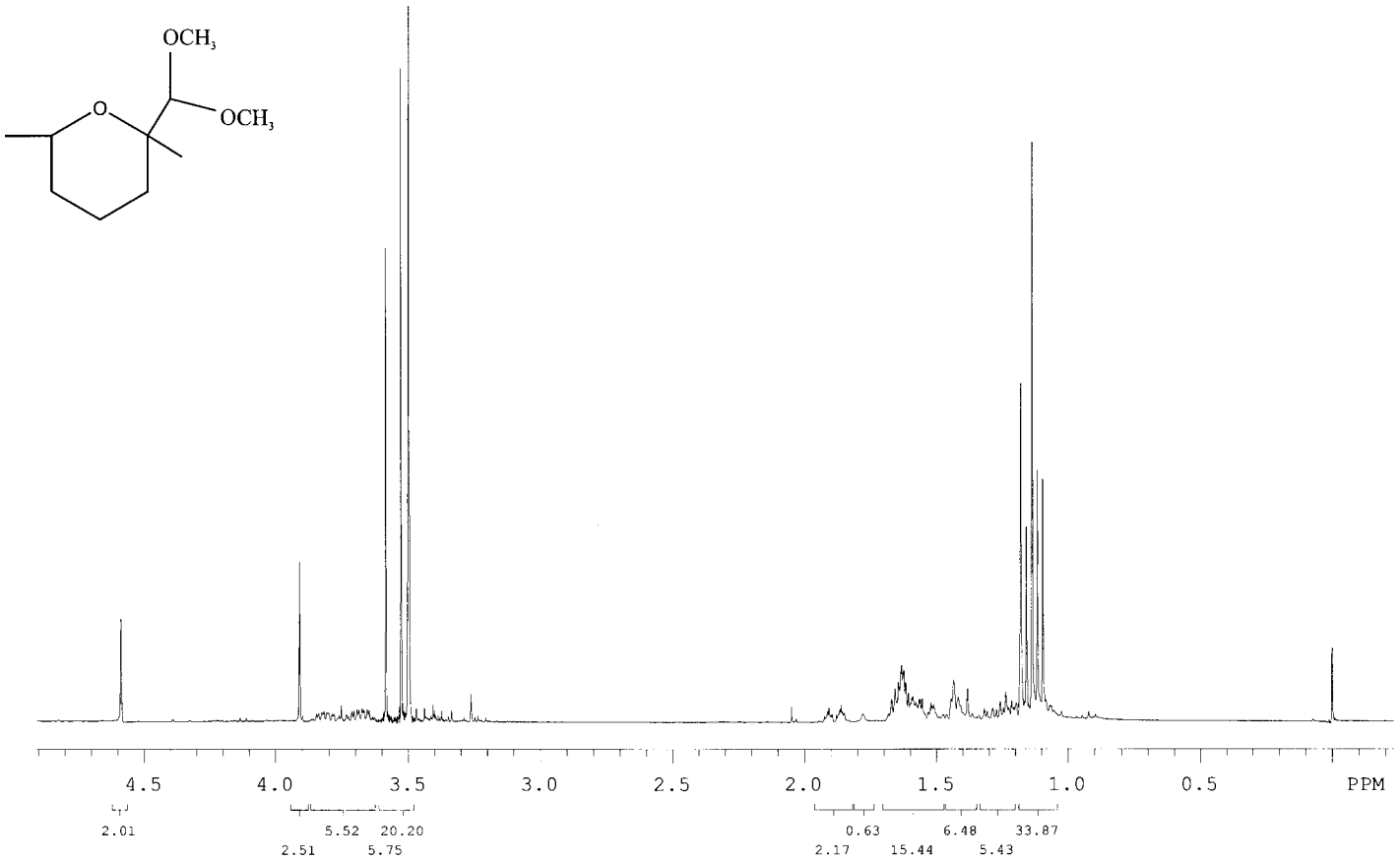


2-dimethoxymethyl-2, 5-dimethyl tetrahydrofuran **18c** (in $CDCl_3$)

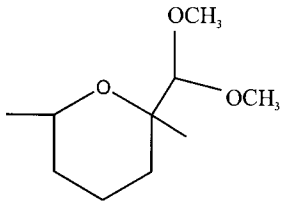


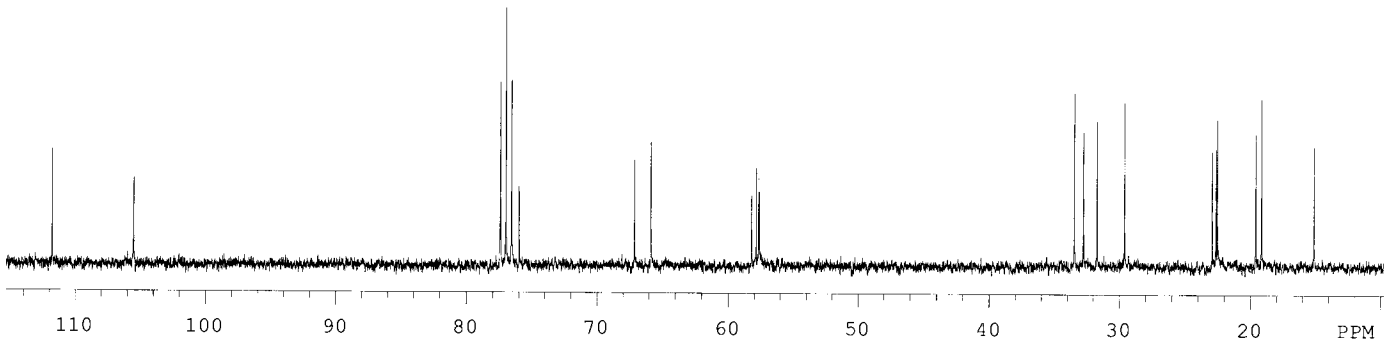


2-dimethoxymethyl-2, 5-dimethyl tetrahydrofuran **18c** (in $CDCl_3$)

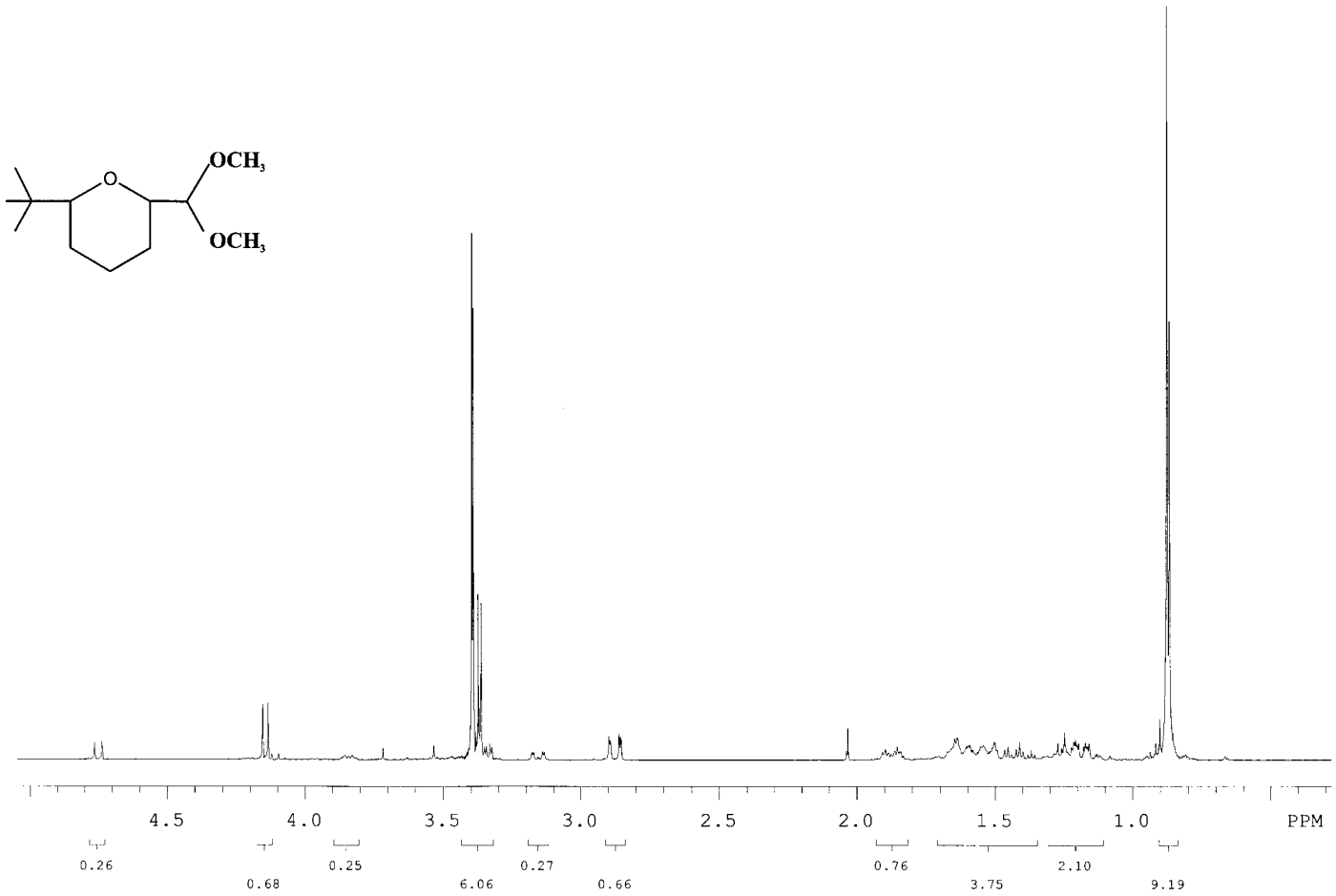


2-dimethoxymethyl-2,6-dimethyl tetrahydropyran **18d** (in CDCl3)

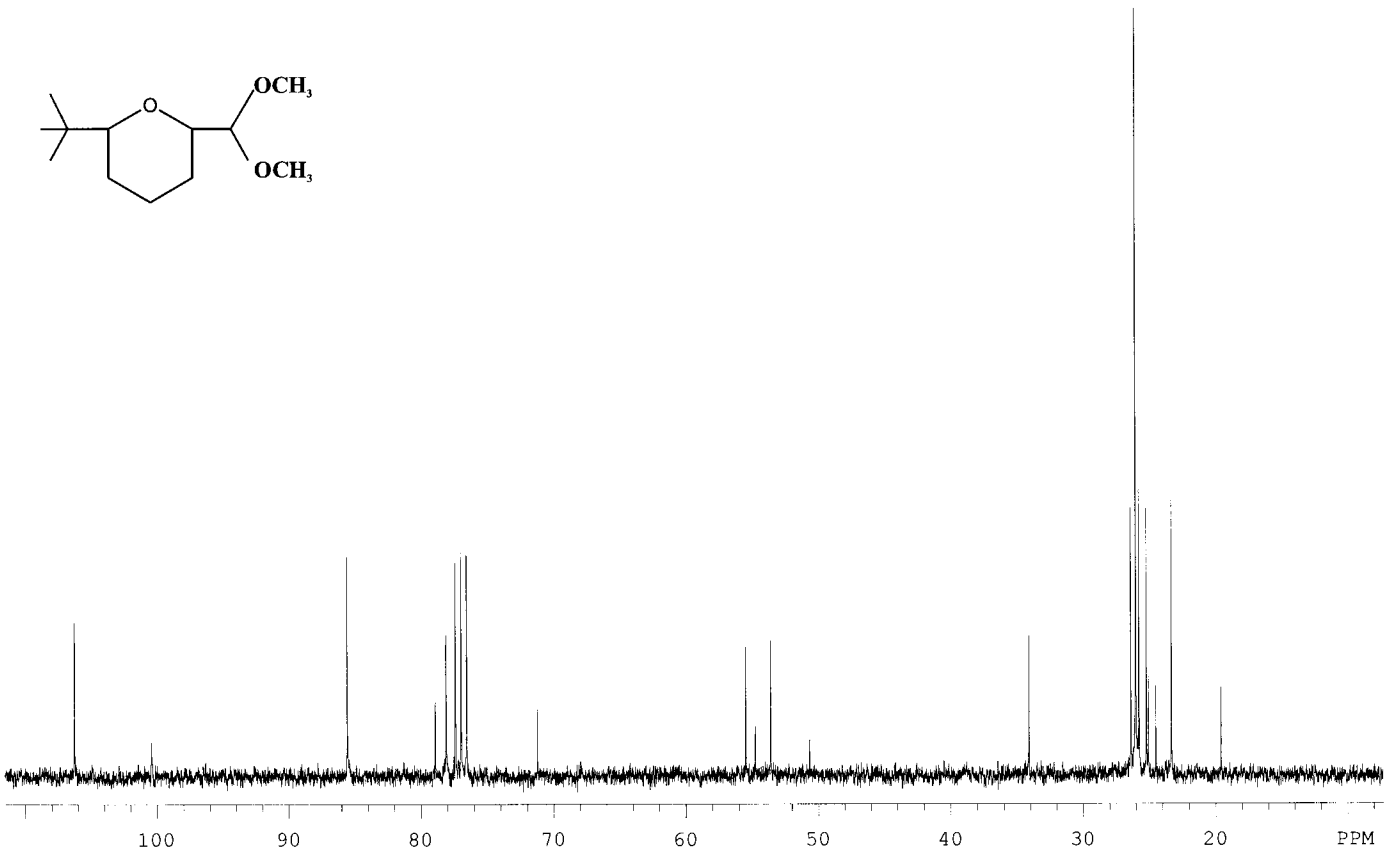




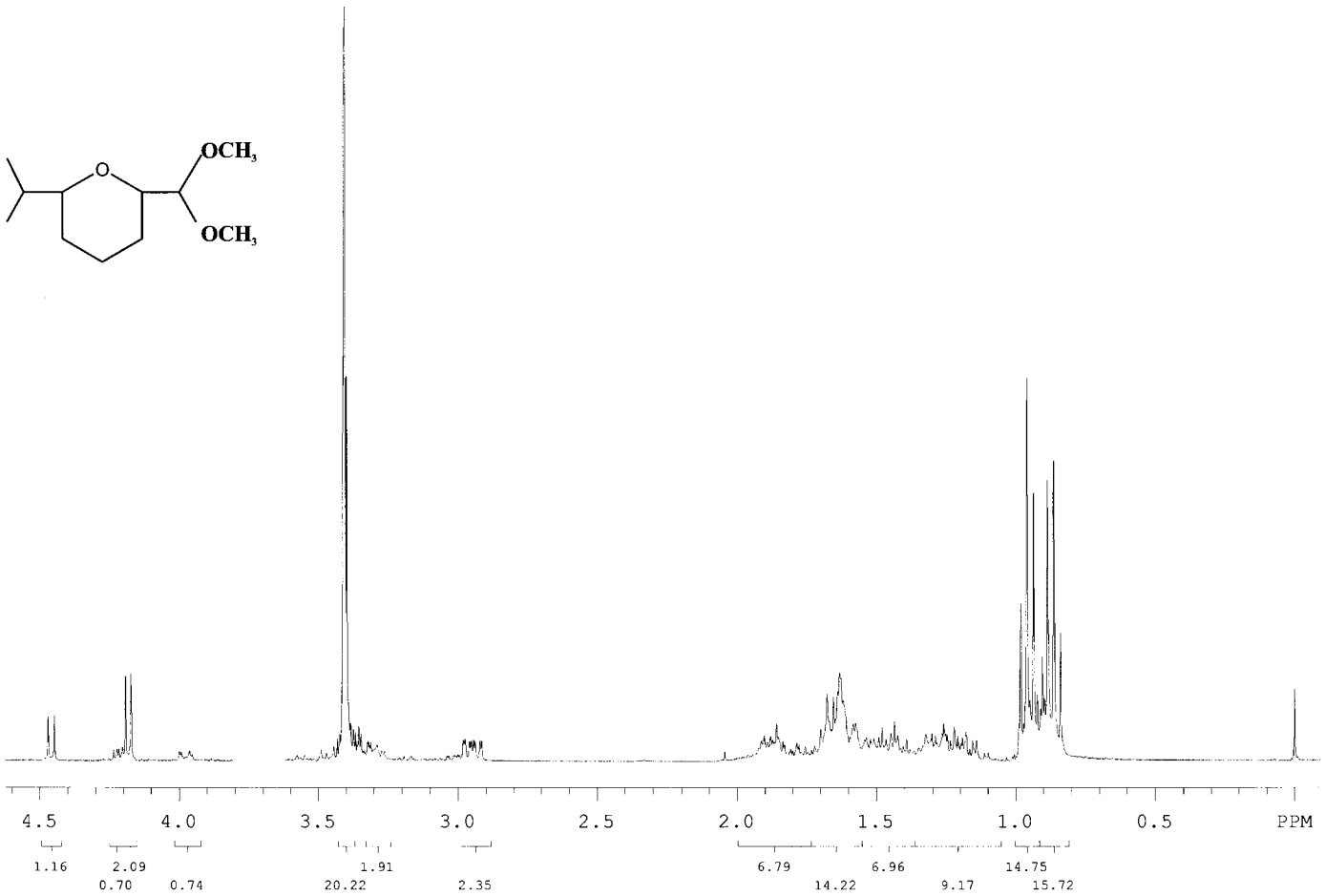
2-dimethoxymethyl-2,6-dimethyl tetrahydropyran **18d** (in CDCl3)



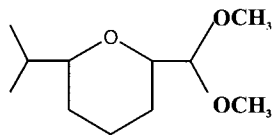
2-dimethoxymethyl-6-*tert*-butyl tetrahydropyran **18e** (in CDCl3)

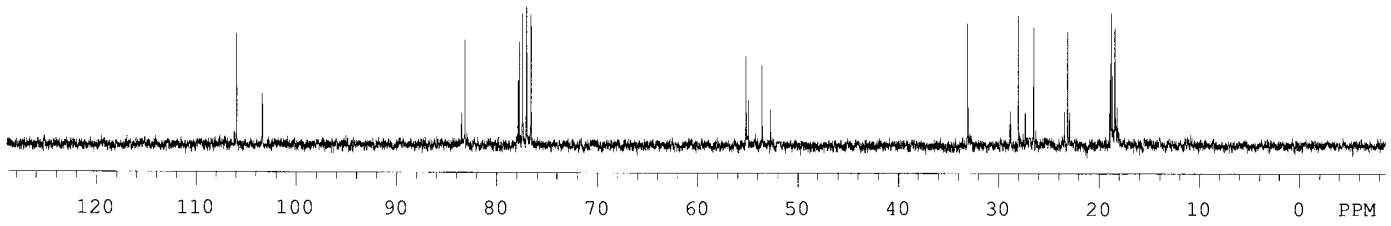


2-dimethoxymethyl-6-*tert*-butyl tetrahydropyran **18e** (in CDCl3)

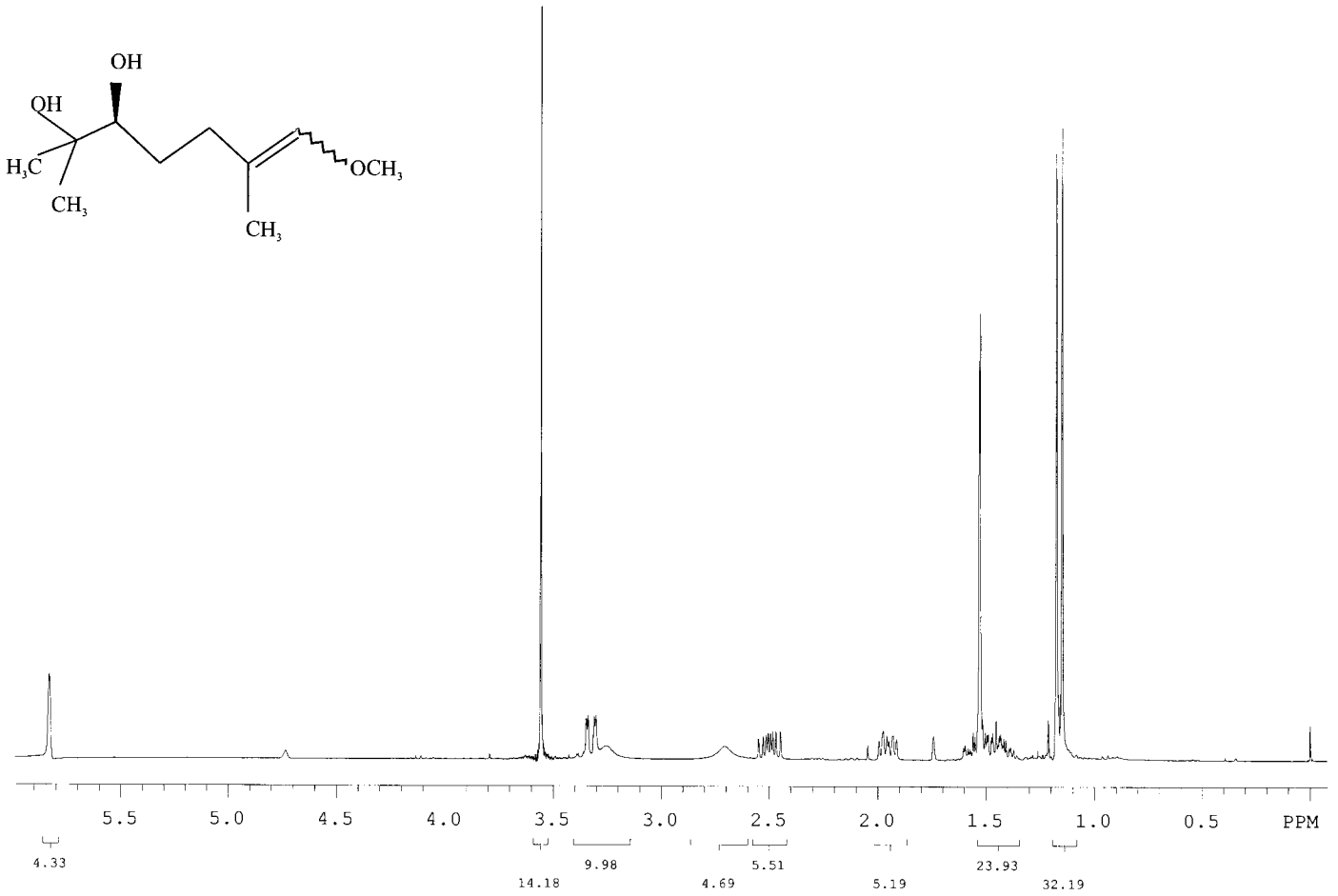


2-dimethoxymethyl-6-isopropyl tetrahydropyran **18f** (in CDCl3)

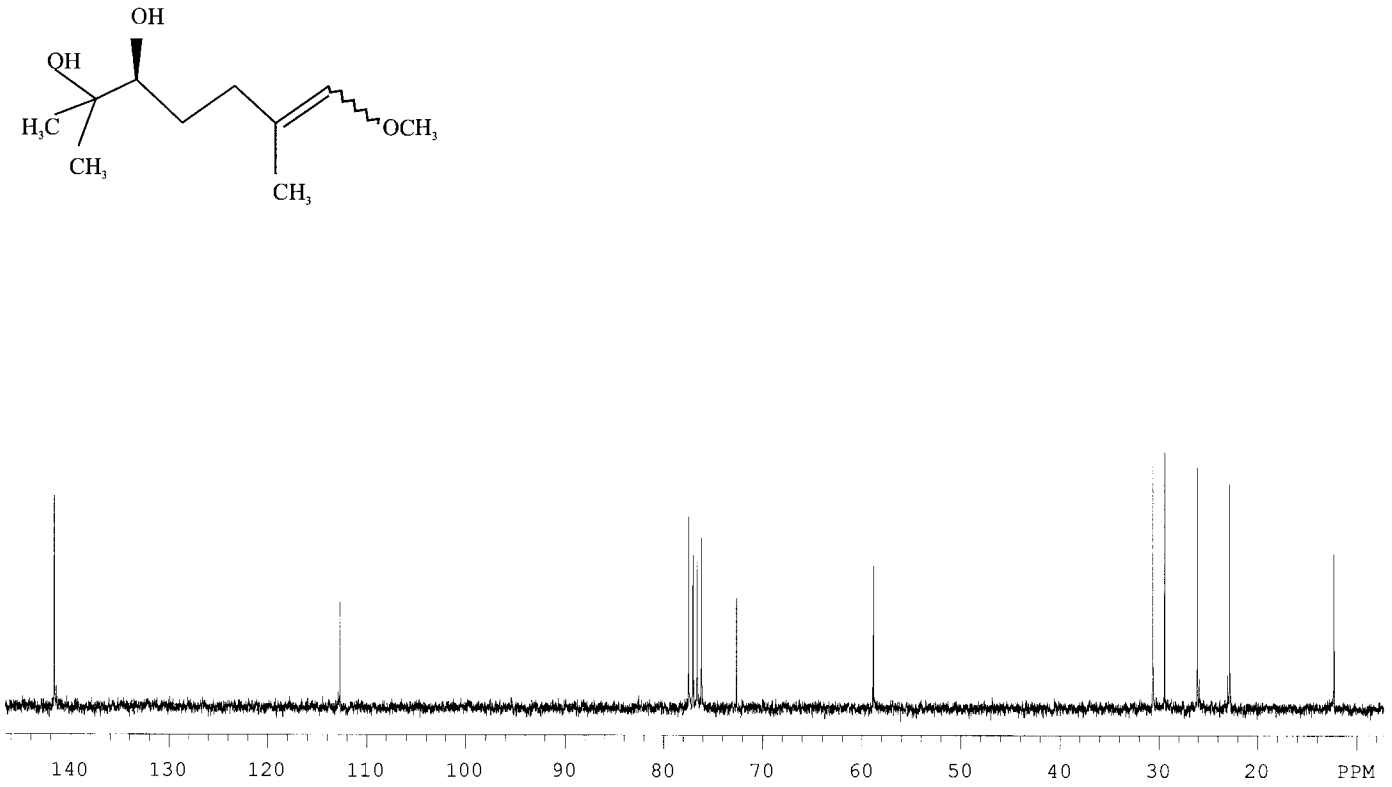




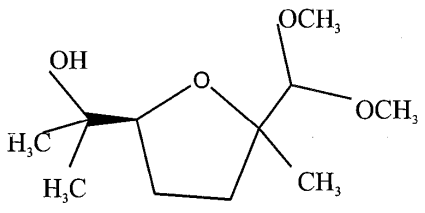
2-dimethoxymethyl-6-isopropyl tetrahydropyran **18f** (in CDCl3)

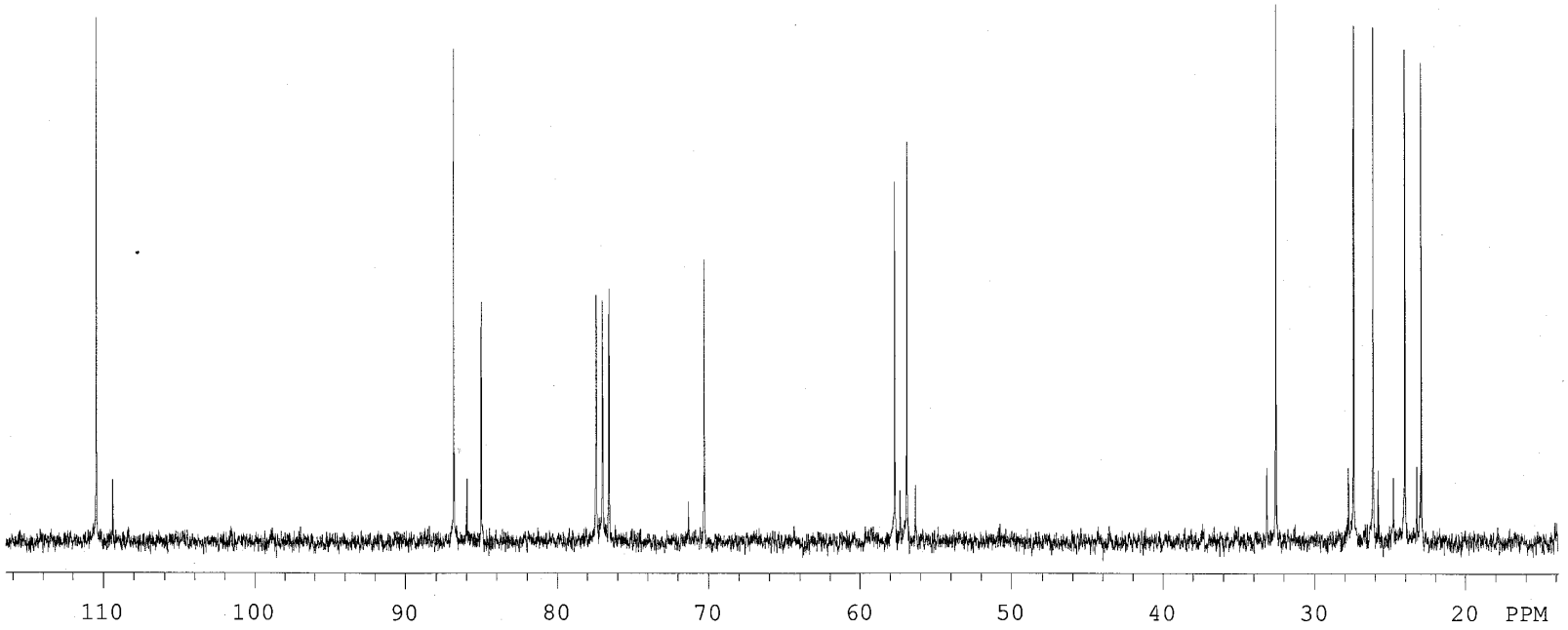


(5S)-2,6-dimethyl-1-methoxy-(E, Z)-1-hepten-5,6-diol **8** (in CDCl3)

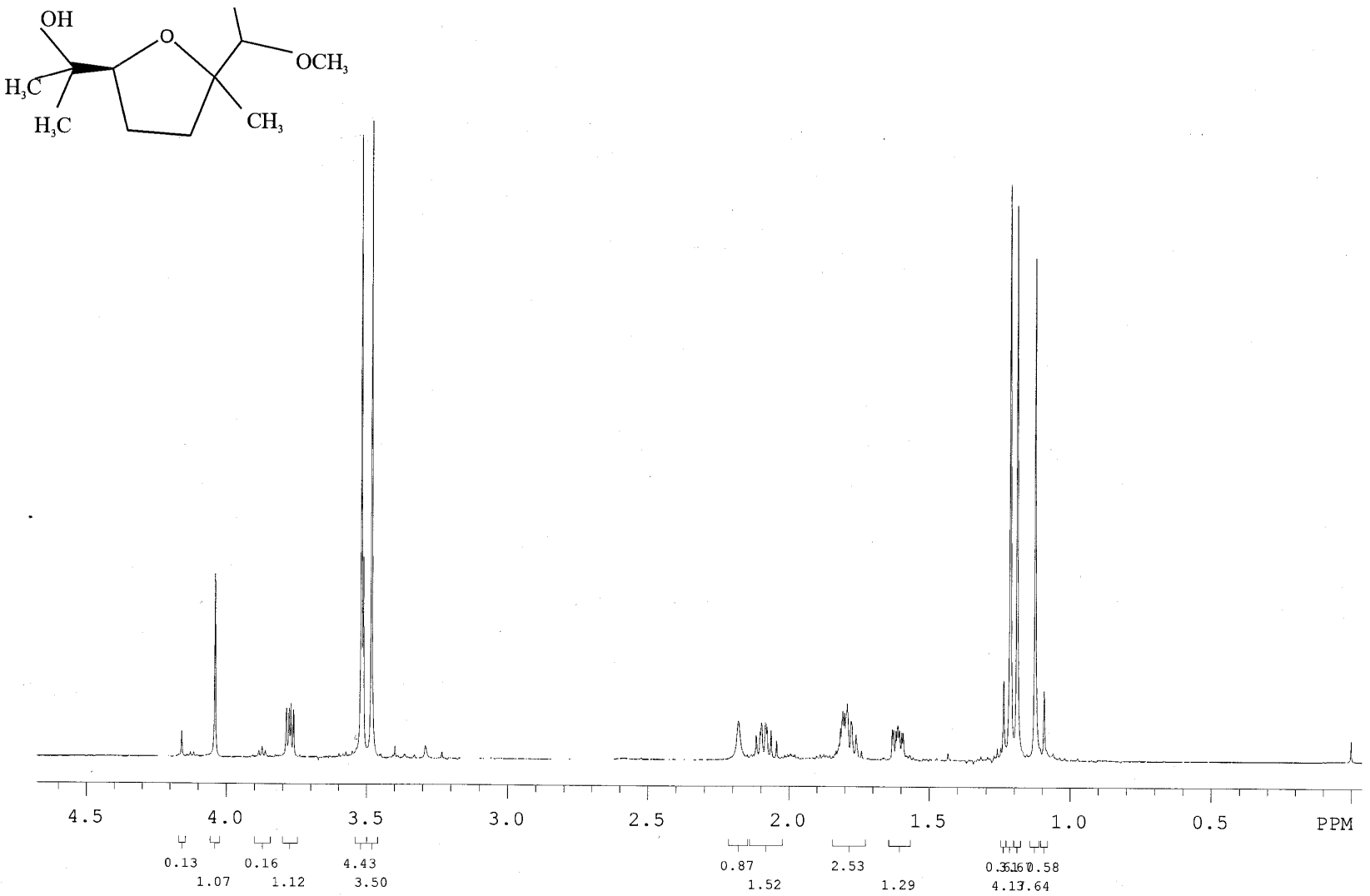


(5S)-2,6-dimethyl-1-methoxy-(E, Z)-1-hepten-5,6-diol **8** (in CDCl3)

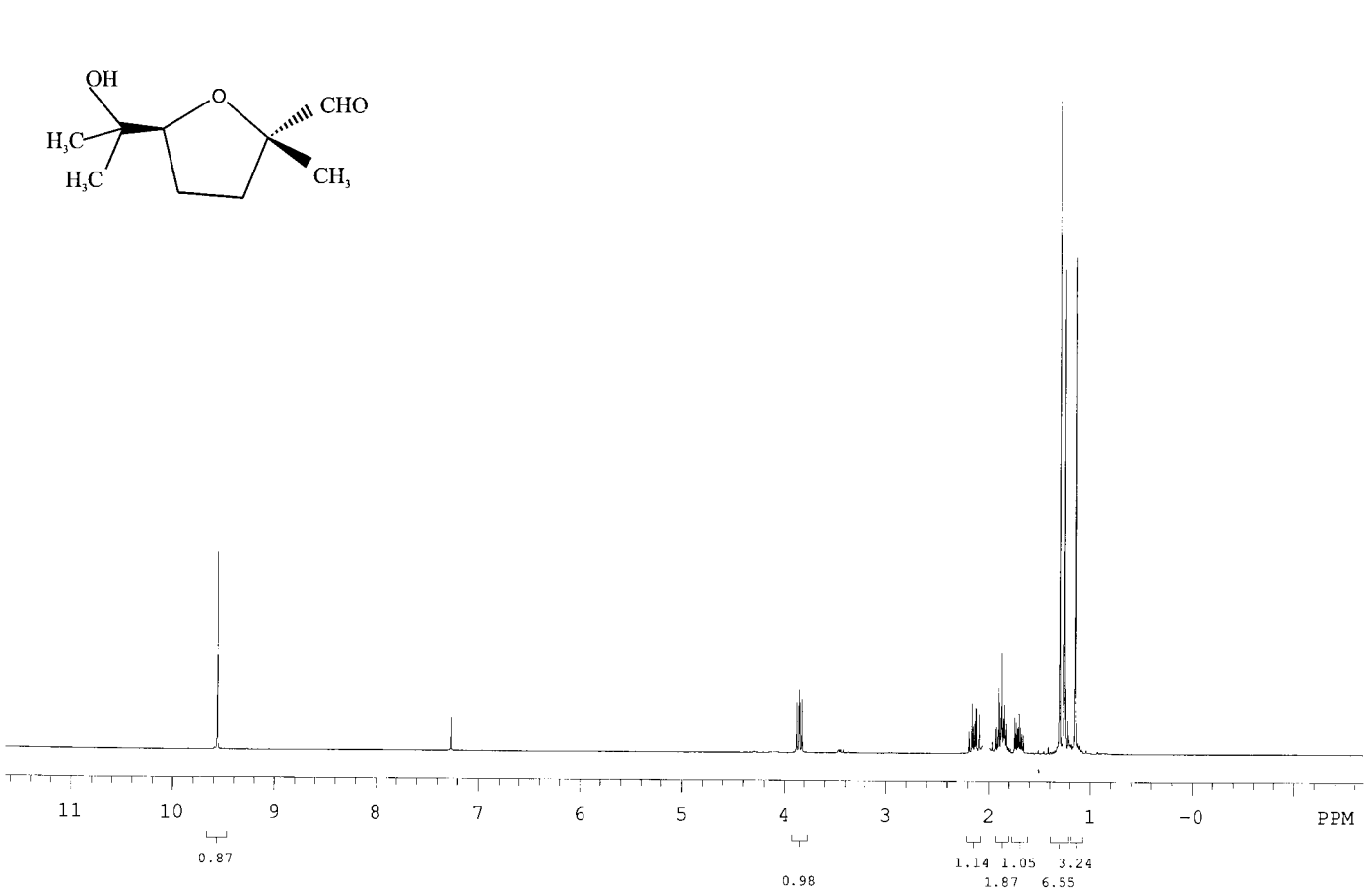




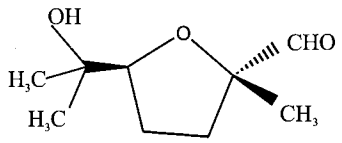
(5S)-2-dimethoxymethyl-5-(1-hydroxy-1-methylethyl)-2-methyl tetrahydrofuran **20** (in CDCl3)

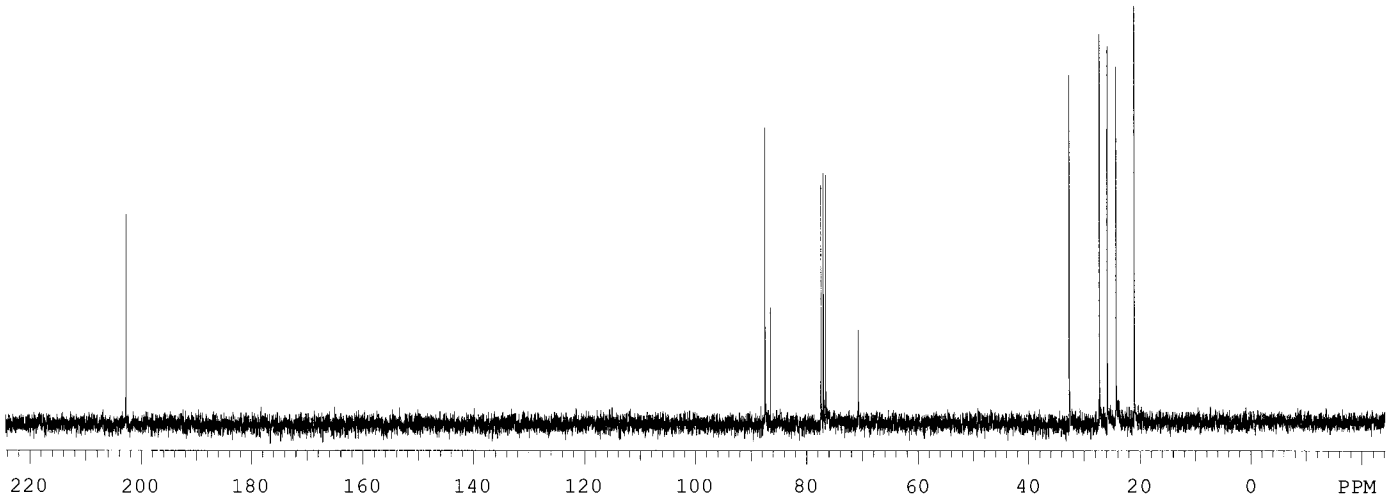


(5S)-2-dimethoxymethyl-5-(1-hydroxy-1-methylethyl)-2-methyl tetrahydrofuran **20** (in CDCl3)

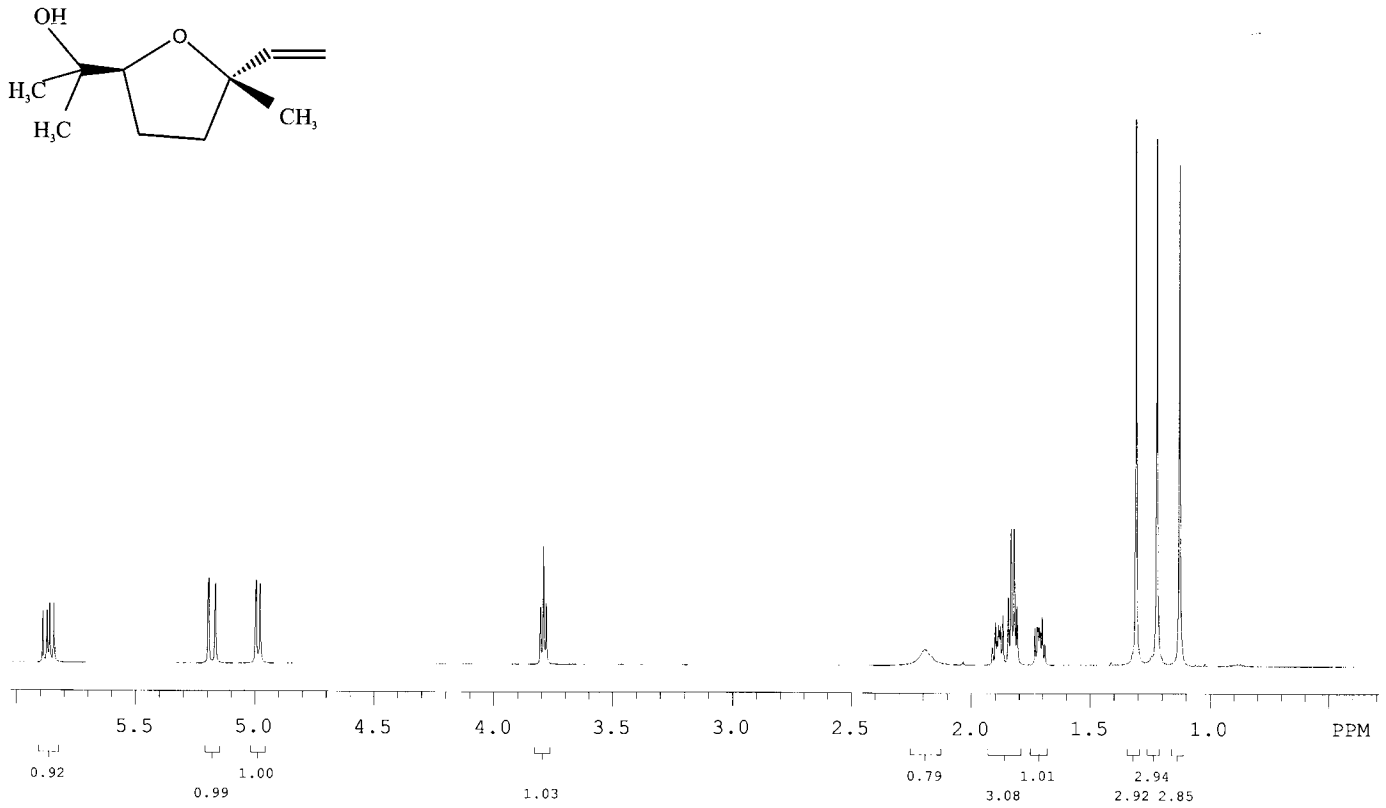


{2S,5S)-5-(1-hydroxy-1-methylethyl)-2-methyl-2-hydrocarbonyl tetrahydrofuran **7** (in CDCl3)

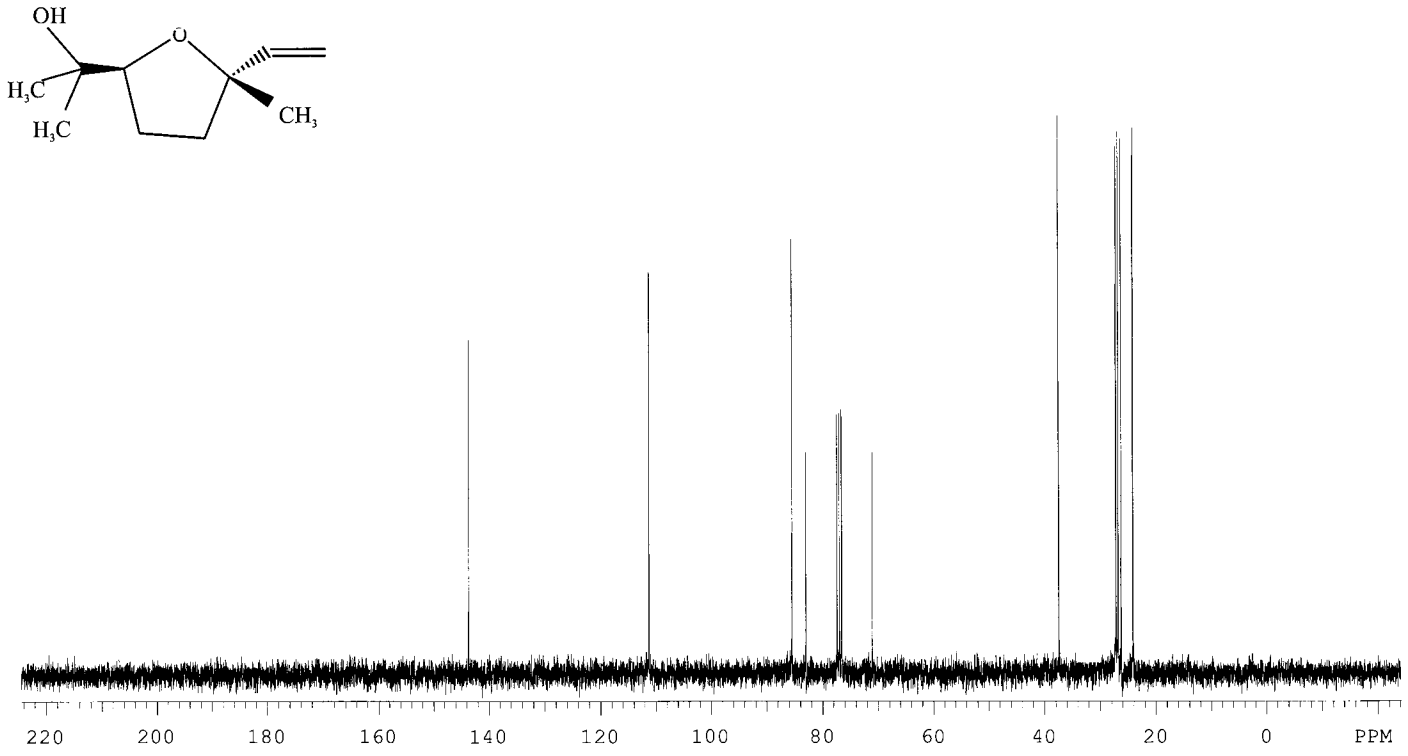




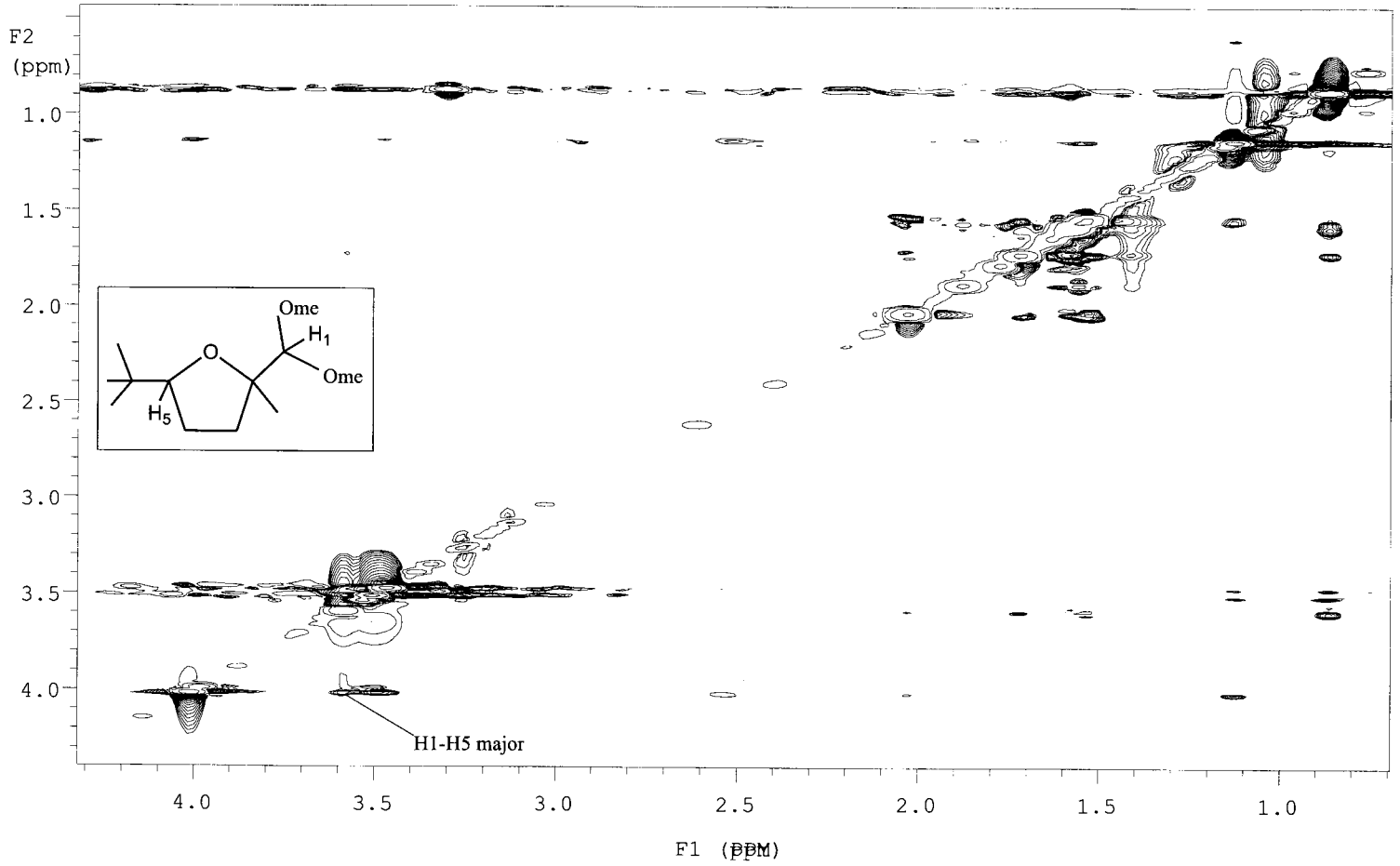
{2S,5S)-5-(1-hydroxy-1-methylethyl)-2-methyl-2-hydrocarbonyl tetrahydrofuran **7** (in CDCl3)



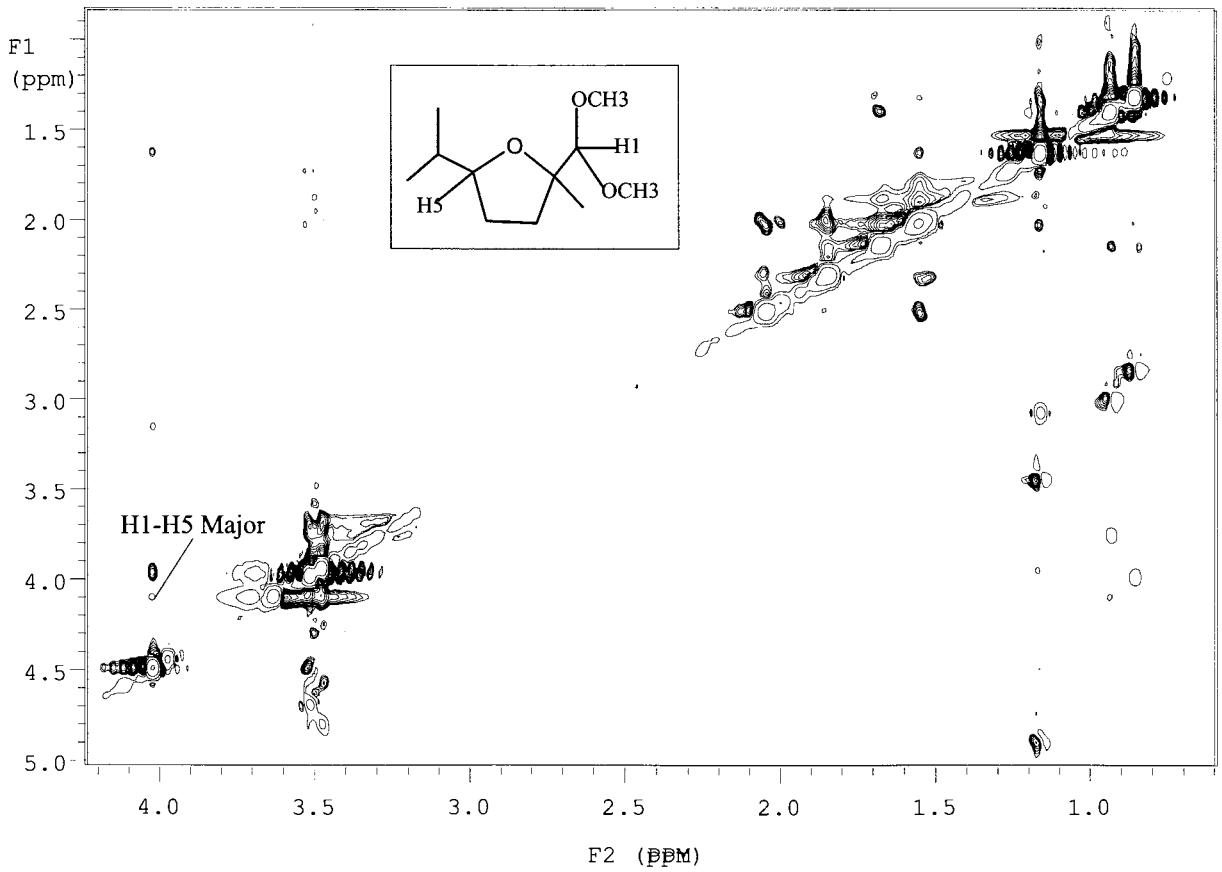
(2S, 5S)-5-(1-hydroxy-1-methylethyl)-2-methyl-2-vinyl tetrahydrofuran **5** (in CDCl3)



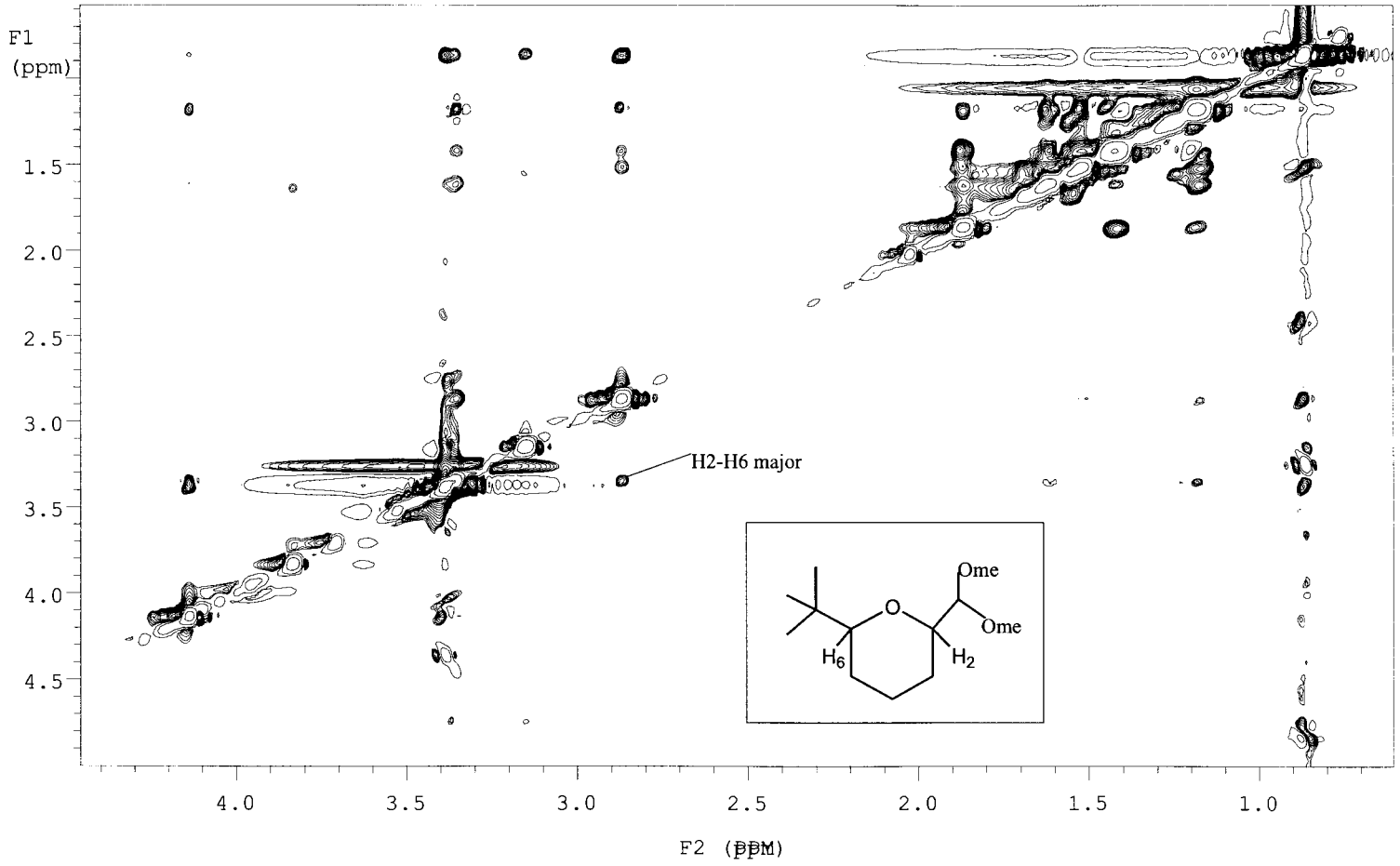
(2S, 5S)-5-(1-hydroxy-1-methylethyl)-2-methyl-2-vinyl tetrahydrofuran **5** (in CDCl3)



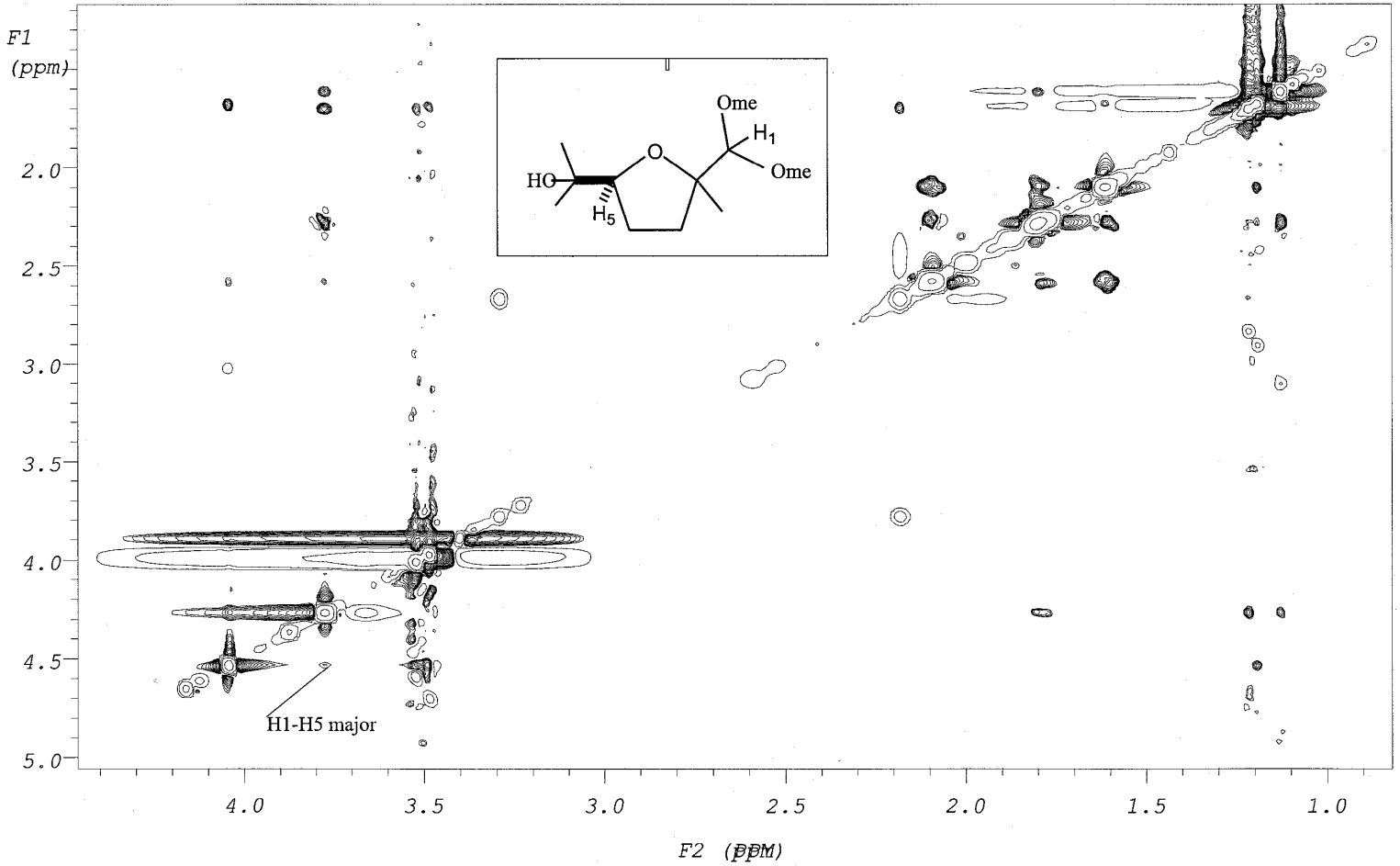
NOE Spectra of **18a**



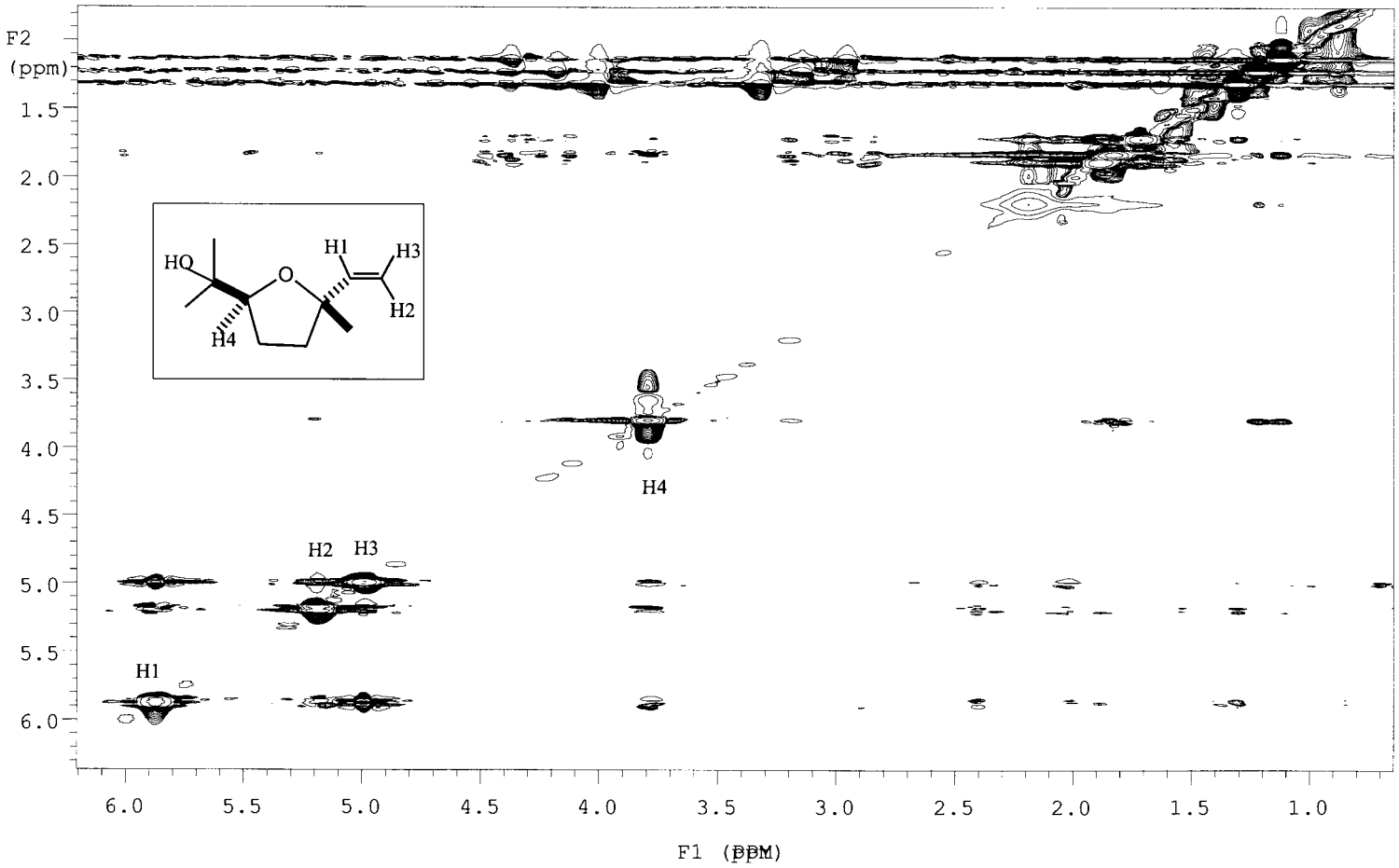
NOE spectra of **18b**



Noe Spectra of **18e**



NOE Spectra of 20



NOE spectra of **5**