Electronic Supplementary Material

7-*Endo* selenocyclization reactions on chiral 3-prenyl and 3-cinnamyl-2-hydroxymethylperhydro-1,3-benzoxazine derivatives. A way to enantiopure 1,4-oxazepanes

Javier Nieto,* Celia Andrés* and Alfonso Pérez-Encabo

Instituto CINQUIMA and Departamento de Química Orgánica, Facultad de Ciencias, Universidad de Valladolid, Paseo de Belén, 7, 47011 Valladolid, Spain.

General procedure for the preparation of starting materials and characterization data.

[(2*S*,4a*S*,7*R*,8a*R*)-3-Cinnamyl-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanone (4). A mixture of benzoxazine **3** (2.25 g, 7.0 mmol), potassium carbonate (1.10 g, 8.0 mmol) and cinnamyl bromide (1.55 g, 7.8 mmol) in acetonitrile (5 mL) was heated in an oil bath at 80-90 ºC until the reaction was complete (TLC, 20 h). The reaction mixture was diluted with ethyl acetate, and the solid was separated by filtration and washed with hot ethyl acetate (3 x 20 mL). The solvent were evaporated under vacuum and the residue was purified by flash chromatography on silica gel using hexanes/EtOAc 15:1 as eluent. Yield (2.54 g, 90%). Colorless solid. Mp: 69-70 ºC (from ethanol). $[\alpha]_D^{25}$ = -43.1 (c = 1.2, $CHCl_3$). ^{1}H NMR (δ): 0.95-1.04 (m, 2H); 0.97 (d, 3H, J = 6.5 Hz); 1.25 (s, 3H); 1.27 (m, 1H); 1.45 (s, 3H); 1.46-1.77 (m, 4H); 2.08 (m, 1H); 3.40 (dd, 1H, J_1 = 17.4 Hz, J_2 = 5.7 Hz); 3.58 (ddd, 1H, J_1 = 17.4 Hz, J_2 = 6.1 Hz, J_3 = 1.0 Hz); 3.65 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.1 Hz); 5.79 (ddd, 1H, J_1 = 16.0 Hz, J_2 = 6.1 Hz, J_3 = 5.7 Hz,); 5.82 (s, 1H); 6.12 (dd, 1H, J_1 = 16.0 Hz, J_2 = 1.0 Hz); 6.96-6.99 (m, 2H,); 7.09-7.19 (m, 3H); 7.27-7.40 (m, 2H); 7.50 (m, 1H); 8.13-8.19 (m, 2H). ^{13}C NMR (δ): 20.4 (CH_3); 22.2 (CH_3); 25.0 (CH_2); 26.8(CH_3); 31.4 (CH); 34.9 (CH_2); 41.3 (CH_2); 46.2 (CH); 46.5 (CH_2); 57.9 (C); 76.4 (CH); 88.1 (CH); 125.9 (2 CH); 126.8 (CH); 128.0 (2 CH); 128.1 (2 CH); 129.3 (2 CH); 130.0 (CH); 130.7 (CH); 133.0 (CH); 135.3 (C); 137.3 (C); 195.0 (C). IR (Nujol dispersion): 3060; 1690; 1600; 750; 680 cm^{-1}. HRMS calcd for $C_{27}H_{34}NO_2$ $[M + H]^+$ 404.2584, found 404.2587.

[(2*S*,4a*S*,7*R*,8a*R*)-3-((*E*)-2-Methyl-3-phenylallyl)-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanone (6). A mixture of alcohol **5** (1.20 g, 4.0 mmol), phenyglyoxal (0,68 g, 5.0 mmol) and toluene (40 mL) was

heated at reflux with a Dean-Stark trap for 38 hours. The solvent was evaporated under vacuum, and the residue was chromatographed on silica gel using hexanes/EtOAc 15/1 as eluent. Yield: 1.27 g, 76%. Colorless solid. Mp: 116-117 ºC (from ethanol). $[\alpha]_D^{25}$ = -56.8 (c = 0.8, $CHCl_3$). ^{1}H NMR (δ): 0.87-1.06 (m, 2H); 0.95 (d, 3H, *J* = 6.5 Hz); 1.21 (s, 3H); 1.28 (m, 1H); 1.44 (s, 3H); 1.45-1.81 (m, 4H); 1.47 (s, 3H); 2.05 (m, 1H); 3.28 (d, 1H, *J* = 17.3 Hz); 3.48 (d, 1H, *J* = 17.3 Hz); 3.64 (td, 1H, J_1 = 10.4 Hz, J_2 = 4.0 Hz); 5.90 (s, 1H); 6.16 (s, 1H); 6.66-6.72 (m, 2H); 7.0-7.19 (m, 3H); 7.40-7.52 (m, 3H); 8.06-8.12 (m, 2H). ^{13}C NMR (δ): 15.8 (CH_3); 20.4 (CH_3); 22.1 (CH_3); 24.9 (CH_2); 26.4 (CH_3); 31.3 (CH); 34.8 (CH_2); 41.1 (CH_2); 45.8 (CH); 51.1 (CH_2); 58.0 (C); 76.4 (CH); 87.5 (CH); 125.4 (CH); 126.7 (CH); 127.5 (2 CH); 127.8 (2 CH); 128.3 (2 CH); 129.2 (2 CH); 132.8 (CH); 135.1 (C); 136.6 (C); 138.1 (C); 194.7 (C). IR (Nujol dispersion): 3080, 1700, 1600, 760, 730, 690 cm^{-1}. HRMS calcd for $C_{28}H_{36}NO_2$ $[M + H]^+$ 418.2741, found 418.2743.

(2*S*,4a*S*,7*R*,8a*R*)-Ethyl 3-cinnamyl-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazine-2-carboxylate (8). This compound was synthesized as benzoxazine **4**, starting from **7** (1.80 g, 7.0 mmol) and purified by flash chromatography on silica gel with hexanes/ethyl acetate 20:1 as eluent. Yield: 2.35 g, 90%). Colorless oil. $[\alpha]_D^{25}$ = -53.8 (c = 1.4, CH_2Cl_2). ^{1}H NMR (δ): 0.84-1.01 (m, 2H); 0.93 (d, 3H, *J* = 6.5 Hz); 1.17 (t, 3H, *J* = 7.1 Hz); 1.19 (m, 1H); 1.21 (s, 3H); 1.25 (s, 3H); 1.42-1.59 (m, 2H); 1.62 (m, 1H); 1.70 (m, 1H); 2.01 (m, 1H); 3.39-3.61 (m, 3H); 4.02 (dq, 1H, J_1 = 10.8 Hz, J_2 = 7.1 Hz); 4.14 (dq, 1H, J_1 = 10.8 Hz, J_2 = 7.1 Hz); 5.13 (s, 1H); 6.21 (dt, 1H, J_1 = 15.9 Hz, J_2 = 5.7 Hz) 6.43 (d, 1H, *J* = 15.9 Hz); 7.14-7.36 (m, 5H). ^{13}C NMR (δ): 13.9 (CH_3); 20.0 (CH_3); 22.0 (CH_3); 24.8 (CH_2); 26.4 (CH_3); 31.1 (CH); 34.7 (CH_2); 40.9 (CH_2); 45.8 (CH); 46.3 (CH_2); 57.1 (C); 61.1 (CH_2); 76.0 (CH); 85.2 (CH); 125.9 (2 CH); 126.8 (CH); 128.2 (2 CH); 129.4 (CH); 130.5 (CH); 137.1 (C); 168.7 (C). IR (Film): 3060, 3040, 3020, 1750,

1600, 730, 700 cm^{-1}. HRMS calcd for $C_{23}H_{34}NO_3$ $[M + H]^+$ 372.2533, found 372.2541.

General procedure for the reduction of ketones 1, 4 and 6 with $NaBH_4$.

To a solution of the corresponding ketone **1**, **4** or **6** (5.0 mmol) in ethanol (30 mL) cooled at -10 ºC, $NaBH_4$ (113 mg, 3.0 mmol) was added with magnetic stirring. After 2 h stirring, additional $NaBH_4$ (75 mg) was added and the mixture was further stirred for 3 h at 0 ºC. Then 75 mL of H_2O was added, and the mixture was stirred for 1 h at room temperature. The ethanol was removed under vacuum and the remaining water was extracted with ethyl acetate (4 x 35 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, concentrated at reduced pressure and the resulting residue was purified by flash chromatography on silica gel using hexanes-ethyl acetate 45:1 as eluent and/or recrystallization.

(*S*)-1-[(2*S*,4a*S*,7*R*,8a*R*)-3-(3-Methylbut-2-enyl)-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl)-2-methylpropan-1-ol (10b). This compound was obtained from **1** (1.10 g). Yield: 1.07 g, 97%. Colorless oil. $[\alpha]_D^{25}$ = -35.5 (c = 0.9, CH_2Cl_2). ^{1}H NMR (δ): 0.87-1.16 (m, 3H); 0.90 (d, 3H, *J* = 6.8 Hz); 0.92 (d, 3H, *J* = 6.5 Hz); 1.01 (d, 3H, *J* = 7.0 Hz); 1.14 (s, 3H); 1.20 (s, 3H); 1.39-1.50 (m, 2H); 1.57-1.72 (m, 2H); 1.61 (s, 3H); 1.68 (s, 3H); 1.85-1.95 (m, 2H); 2.51 (s broad 1H); 3.20 (dd, 1H J_1 = 12.8 Hz, J_2 = 5.3 Hz); 3.35-3.51 (m, 3H); 4.47 (d, 1H, *J* = 8.5 Hz); 5.18 (t, 1H, *J* = 5.3 Hz). ^{13}C NMR (δ): 15.5 (CH_3); 17.7 (CH_3); 19.9 (CH_3); 22.1 (CH_3); 22.6 (CH_3); 24.9 (CH_2); 25.6 (CH_3); 26.9 (CH_3); 29.1 (CH); 31.3 (CH); 35.0 (CH_2); 40.1 (CH_2); 41.2 (CH_2); 45.0 (CH); 57.1 (C); 72.6 (CH); 77.0 (CH); 87.5 (CH); 127.1 (CH); 130.1 (C). IR (Film): 3500 (broad), 750, 700 cm^{-1}. HRMS calcd for $C_{20}H_{38}NO_2$ $[M + H]^+$ 324.22897, found 324.2892.

(*S*)-[(2*S*,4a*S*,7*R*,8a*R*)-3-cinnamyl-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanol (10h). This compound was

obtained from **4** (2.00 g). Yield: 1.90 g, 94%. Colorless solid. Mp: 92-93 ºC (from ethanol). $[\alpha]_D^{25}$ = -109,3 (c = 0.8, CH_2Cl_2). ^{1}H NMR (δ): 0.82-0.89 (m, 2H); 0.85 (d, 3H, *J* = 6.5 Hz); 0.98 (m, 1H); 1.22 (s, 3H); 1.24 (s, 3H); 1.34 (m, 1H); 1.45-1.64 (m, 2H); 1.69 (m, 1H); 1.75 (m, 1H); 3.21 (s, 1H); 3.23 (td, 1H, J_1 = 10.6 Hz, J_2 = 3.9 Hz); 3.54 (dd, 1H, J_1 = 17.8 Hz, J_2 = 5.9 Hz); 3.80 (dd, 1H, J_1 = 17.8 Hz, J_2 = 5.9 Hz); 4.56 (d, 1H, *J* = 8.3 Hz); 4.62 (d, 1H, *J* = 8.3 Hz); 6.37 (dt, 1H, J_1 = 16.0 Hz, J_2 = 5.9 Hz); 6.56 (d, 1H, *J* = 16.0 Hz); 7.15-7.37 (m, 8H); 7.56-7.66 (m, 2H). ^{13}C NMR (δ): 22.2 (CH_3); 22.6 (CH_3); 25.0 (CH_2); 27.1 (CH_3); 31.3 (CH); 35.1 (CH_2); 41.1 (CH_2); 44.3 (CH_2); 45.3 (CH); 57.8 (C); 71.7 (CH); 77.2 (CH); 90.6 (CH); 126.3 (2 CH); 127.2 (3 CH); 127.5 (CH); 127.9 (2 CH); 128.5 (2 CH); 129.9 (CH); 132.0 (CH); 137.2 (C); 141.2 (C). IR (Nujol dispersion): 3460 (broad), 1590, 720, 705, 680 cm^{-1}. HRMS calcd for $C_{28}H_{36}NO_2$ $[M + H]^+$ 406.2741, found 406.2721.

(*S*)-[(2*S*,4a*S*,7*R*,8a*R*)-3-((*E*)-2-Methyl-3-phenylallyl)-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanol (10i). This compound was obtained from **6** (1.05 g). Yield: 0.98 g, 95%. Colorless solid. Mp: 108-109 ºC (from ethanol). $[\alpha]_D^{25}$ = -36.0 (c = 0.9, $CHCl_3$). ^{1}H NMR (δ): 0.83-108 (m, 3H); 0.87 (d, 3H, *J* = 6.5 Hz); 1.21 (s, 3H); 1.32 (s, 3H); 1.33 (m, 1H); 1.48 (m, 1H); 1.57 (m, 1H); 1.64-1.79 (m, 2H); 1.89 (s, 3H); 3.08 (s, 1H); 3.29 (td, 1H, J_1 = 10.4 Hz, J_2 = 3.9 Hz); 3.38 (d, 1H, *J* = 19.1 Hz); 3.73 (d, 1H, *J* = 19.1 Hz); 4.53 (d, 1H, *J* = 8.4 Hz); 4.65 (d, 1H, *J* = 8.4 Hz); 6.90 (s, 1H); 7.09-7.37 (m, 8H); 7.42-7.50 (m, 2H). ^{13}C NMR (δ): 16.8 (CH_3); 22.1 (CH_3); 22.5 (CH_3); 24.9 (CH_2); 26.1 (CH_3); 31.2 (CH); 35.0 (CH_2); 41.0 (CH_2); 45.3 (CH); 48.8 (CH_2); 57.4 (C); 72.6 (CH); 76.9 (CH); 90.6 (CH); 124.2 (CH); 126.0 (CH); 127.1 (2 CH); 127.3 (CH); 127.7 (2 CH); 128.0 (2 CH); 128.8 (2 CH); 137.9 (C); 140.4 (C); 141.0 (C). IR (Nujol dispersion): 3525 (broad), 3020, 1585, 750, 700 cm^{-1}. HRMS calcd for $C_{28}H_{37}NO_2$ $[M + H]^+$ 420.2897, found 420.2880.

(*S*)-[(2*S*,4a*S*,7*R*,8a*R*)-3-((*E*)-3-Phenylbut-2-enyl)-4,4,7-trimethyl-octahydro-2*H*- benzo[e][1,3]oxazin-2-yl)methanol (10g). A mixture of alcohol **2** (1.50 g,

5.0 mmol), glycolaldehyde dimer (0.30 g, 2,5 mmol), toluene (25 mL) and CH_2Cl_2 (10 mL) was heated at reflux. Additional portions of glycolaldehyde dimer (60 mg, 0.5 mmol) were added each day and the heating was continued for 6 days. The solvent was evaporated under vacuum, and the residue was chromatographed on silica gel using hexanes-ethyl acetate 8/1 as eluent. Yield: 1.03 g, 60%. Colorless oil. $[\alpha]_D^{25}$ = -37.9 (c = 1.1, CH_2Cl_2). ^{1}H NMR (δ): 0.86-1.14 (m, 3H); 0.93 (d, 3H, *J* = 6.3 Hz); 1.15 (s, 3H); 1.18 (s, 3H); 1.39-1.50 (m, 2H); 1.55 (m, 1H); 1.67 (m, 1H); 1.94 (m, 1H); 2.02 (s, 3H); 3.05 (s broad, 1H); 3.27 (dd, 1H, J_1 = 16.8 Hz, J_2 = 5.8 Hz); 3.42-3.60 (m, 2H); 3.61 (d, 2H, *J* = 5.4 Hz); 4.62 (t, 1H, *J* = 5.4 Hz); 5.86 (t, 1H, *J* = 5.8 Hz); 7.12-7.39 (m, 5H). ^{13}C NMR (δ): 15.5 (CH_3); 18.7 (CH_3); 21.9 (CH_3); 24.7 (CH_2); 26.5 (CH_3); 30.8 (CH); 34.5 (CH_2); 40.8 (CH_2); 41.7 (CH_2); 46.9 (CH); 56.5 (C); 63.4 (CH_2); 75.4 (CH); 87.3 (CH); 125.2 (2 CH); 126.2 (CH); 127.7 (2 CH); 130.2 (CH); 132.1 (C); 142.8 (C). IR (Film): 3425 (broad), 3060, 3040, 1600, 730, 700, 650 cm^{-1}. HRMS calcd for $C_{22}H_{34}NO_2$ $[M + H]^+$ 344.2584, found 344.2582.

(*S*)-2-[(2*S*,4a*S*,7*R*,8a*R*)-3-cinnamyl-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl)propan-2-ol (10j). To a stirred solution of benzoxazine **8** (1.50 g, 4.0 mmol) in anhydrous ethyl ether (20 mL) cooled to 0 ºC, an ethereal solution of methylmagnesium iodide (3.0 mL of 3.0 M solution, Aldrich) was added dropwise under a nitrogen atmosphere. Stirring was continued until disappearance of the starting ester (TLC, 20 min.). The mixture was quenched with saturated aqueous NH_4Cl (50 mL), the layers were separated and the aqueous was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine, dried over $MgSO_4$ and the solvent was evaporated under vacuum. The residue was purified by flash chromatography on silica gel, using hexanes-ethyl acetate 40:1 as eluent. Yield: 1.25 g, 86%. Colorless solid. Mp: 64-65 ºC (from ethanol). $[\alpha]_D^{25}$ = -24.6 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.83-1.01 (m, 2H); 0.93 (d, 3H, *J* = 6.5 Hz); 1.11 (m, 1H);

1.12 (s, 3H); 1.22 (s, 3H); 1.25 (s, 3H); 1.27 (s, 3H); 1.41-1.63 (m, 3H); 1.70 (m, 1H); 1.92 (m, 1H); 2.51 (s, 1H); 3.41-3.59 (m, 2H); 4.23 (dd, 1H, J_1 = 18.5 Hz, J_2 = 5.0 Hz); 4.36 (s, 1H); 6.33 (dt, 1H, J_1 = 16.0 Hz, J_2 = 5.0 Hz); 6.47 (d, 1H, J = 16.0 Hz); 7.12-7.40 (m, 5H). ^{13}C NMR (δ): 22.3 (CH_3); 23.1 (CH_3); 24.8 (CH_3); 25.0 (CH_2); 26.8 (CH_3); 30.2 (CH_3); 31.5 (CH); 35.2 (CH_2); 41.5 (CH_2); 44.5 (CH); 45.1 (CH_2); 57.8 (C); 72.5 (C); 77.4 (CH); 90.4 (CH); 126.0 (2 CH); 126.6 (CH); 128.4 (3 CH); 134.1 (CH); 137.9 (C). IR (Nujol dispersion): 3560 (broad), 3080, 3060, 3030, 1600, 770, 740, 725, 625 cm^{-1}. HRMS calcd for $C_{23}H_{36}NO_2$ $[M + H]^+$ 358.2741, found 358.2733.

(*R*)-[(2*S*,4a*S*,7*R*,8a*R*)-3-cinnamyl-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanol (10k). To a stirred solution of aldehyde **9** (1.50 g, 4.6 mmol) in anhydrous ethyl ether (20 mL) cooled to -10 ºC, an ethereal solution of freshly prepared phenylmagnesium bromide (6.5 mL, 1.0 M solution) was added dropwise under a nitrogen atmosphere. Stirring was continued for 20 min and the mixture was quenched with saturated aqueous NH_4Cl (50 mL). The layers were separated and the aqueous was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine, dried over $MgSO_4$ and the solvent was evaporated under vacuum. The residue was purified by flash chromatography on silica gel, using hexanes-ethyl acetate 50:1 as eluent. Yield: 1.68 g, 91%. Colorless solid. Mp: 120-121 ^{o}C (from ethanol). $[\alpha]_D^{25}$ = -9.3 (c = 0.5 CH_2Cl_2). ^{1}H NMR (δ): 0.83-1.00 (m, 2H); 0.93 (d, 3H, J = 6.5 Hz); 1.10 (s, 3H); 1.11 (m, 1H); 1.13 (s, 3H); 1.40-1.55 (m, 2H); 1.60 (m, 1H); 1.70 (m, 1H); 1.95 (m, 1H); 3.24 (s broad, 1H); 3.42 (dd, 1H, J_1 = 18.2 Hz, J_2 = 5.3 Hz); 3.53 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.0 Hz); 3.72 (dd, 1H, J_1 = 18.2 Hz, J_2 = 5.3 Hz); 4.73 (d, 1H, J = 5.7 Hz,); 4.77 (d, 1H, J = 5.7 Hz); 6.14 (dt, 1H, J_1 = 16.0 Hz, J_2 = 5.3 Hz); 6.30 (d, 1H, J = 16.0 Hz); 7.18-7.38 (m, 10H). ^{13}C NMR (δ): 20.1 (CH_3); 22.2 (CH_3); 25.0 (CH_2); 26.6 (CH_3); 31.3 (CH); 35.0 (CH_2); 41.3 (CH_2); 45.7 (CH_2); 46.5 (CH); 57.3 (C); 73.6 (CH); 76.3 (CH); 90.2 (CH);

126.1 (2 CH); 126.9 (CH); 127.6 (CH); 127.7 (2 CH); 128.0 (2 CH); 128.4 (2 CH); 129.1 (CH); 132.1 (CH); 137.4 (C); 140.6 (C). IR (Nujol dispersion): 3490 (broad), 3060, 3050, 3030, 1600, 740, 695 cm^{-1}. HRMS calcd for $C_{27}H_{36}NO_2$ $[M + H]^+$ 406.2741, found 406.2738

[(2*S*,4a*S*,7*R*,8a*R*)-3-(But-3-enyl)-4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanone (25). This benzoxazine was synthesized as described for oxazine **6** starting from aminoalcohol **24** (1.0 g), and purified by flash chromatography on silica gel with hexanes/ethyl acetate 16:1 as eluent**.** Yield: 0.69 g, 46%. Colorless oil. $[\alpha]_D^{25}$ = -31.3 (c = 0.6, CH_2Cl_2). ^{1}H NMR (δ): 0.84-1.03 (m, 2H); 0.93 (d, 3H, *J* = 6.5 Hz); 1.21 (m, 1H); 1.22 (s, 3H); 1.39 (s, 3H); 1.44-1.57 (m, 2H); 1.62-1.76 (m, 3H); 1.90 (m, 1H); 2.02 (m, 1H); 2.59 (ddd 1H, J_1 = 15.3 Hz, J_2 = 10.6 Hz, J_3 = 5.4 Hz); 2.77 (ddd, 1H, J_1 = 15.3 Hz, J_2 = 10.8 Hz, J_3 = 5.6 Hz); 3,58 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.1 Hz); 4.71-4.80 (m, 2H); 5.48 (m, 1H); 5.73 (s, 1H); 7.40-7.49 (m, 2H); 7.54 (m,1H); 8.20-8.23 (m, 2H). ^{13}C NMR (δ): 20.2 (CH_3); 22.2 (CH_3); 25.0 (CH_2); 26.4 (CH_3); 31.3 (CH); 34.9 (CH_2); 37.0 (CH_2); 41.2 (CH_2); 44.0 (CH_2); 45.9 (CH); 57.6 (C); 76.1 (CH); 88.0 (CH); 115.3 (CH_2); 128.1 (2 CH); 129.3 (2 CH); 133.1 (CH); 135.3 (C); 136.1 (CH); 195.0 (C). IR (Film): 3080, 1690, 1610, 1485, 1350, 975, 725 cm^{-1}. HRMS calcd for $C_{22}H_{32}NO_2$ $[M + H]^+$ 342.2428, found 342.2428.

(*S*)-[(2*S*,4a*S*,7*R*,8a*R*)-3-(But-3-enyl)--4,4,7-trimethyl-octahydro-2*H*-benzo[e][1,3]oxazin-2-yl](phenyl)methanol (26). This alcohol was synthesized by reduction of ketone **25** (0.55 g) with $NaBH_4$ as described for alcohols **10b**, **10h** and **10i**, and purified by flash chromatography on silica gel with hexanes/ethyl acetate 50:1 as eluent. Yield: 0.53 g, 96%. Colorless solid. Mp: 93-94 ºC (from ethanol). $[\alpha]_D^{25}$ = -71.5 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.83-0.94 (m, 2H); 0.85 (d, 3H, *J* = 6,5 Hz); 0.97 (m, 1H); 1.21 (s, 3H); 1,22 (s, 3H); 1.35 (m, 1H); 1.47 (m, 1H); 1.58 (m, 1H); 1.62-1.71 (m, 2H); 2.23-2.46 (m, 2H); 2.70 (ddd, 1H, J_1 = 15.8 Hz, J_2 = 10.0 Hz, J_3 = 5.7 Hz); 3.06 (ddd, 1H, J_1 = 15.8 Hz, J_2 = 10.5

Hz, J_3 = 5.6 Hz); 3,22 (td, 1H, J_1 = 10.6 Hz, J_2 = 3.8 Hz); 3.37 (s broad, 1H); 4.47 (d, 1H, J = 8.6 Hz); 4,53 (d, 1H, J = 8.6 Hz); 5.02-5.12 (m, 2H); 5.82 (m, 1H); 7.24-7.35 (m, 3H); 7.44-7.47 (m, 2H). ^{13}C NMR (δ): 22.1 (CH_3); 22.8 (CH_3); 25.0 (CH_2); 26.8 (CH_3); 31.3 (CH); 35.1 (CH_2); 38.0 (CH_2); 40.9 (CH_2); 41.9 (CH_2); 44.8 (CH); 57.6 (C); 71.4 (CH); 77.2 (CH); 90.6 (CH); 116.1 (CH_2); 127.2 (2 CH); 127.4 (CH); 127.8 (2 CH); 136.3 (CH); 141.1 (C). IR (Nujol dispersion): 3030, 1590, 750, 720, 690 cm^{-1}. HRMS calcd for $C_{22}H_{34}NO_2$ $[M + H]^+$ 344.2584, found 344.2567.

Characterization data for selenocyclization products

(1*S*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1-Isopropyl-4-phenyselenenyl-3,3,7,7,10-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11b). Colorless solid. Mp: 101-102 ºC (from hexane). $[\alpha]_D^{25}$ = +68.3 (c = 1.3, CH_2Cl_2). ^{1}H NMR (δ): 0.60 (s, 3H); 0.78-1.07 (m, 3H); 0.89 (d, 6H, J = 6.6 Hz); 0.93 (d, 3H, J = 6.8 Hz); 1.09 (s, 3H); 1.15 (m, 1H); 1.21 (s, 3H); 1.37-1.49 (m, 2H); 1.56 (s, 3H); 1.61 (m, 1H); 1.75-1.85 (m, 2H); 2.92 (d, 1H, J = 12.4 Hz); 3.22-3.40 (m, 4H); 4.57 (d 1H, J = 7.1 Hz); 7.11-7.32 (m, 3H); 7.58-7.64 (m, 2H). ^{13}C NMR (δ): 16.8 (CH_3); 20.2 (CH_3); 21.1 (CH_3); 21.2 (CH_3); 22.2 (CH_3); 24.9 (CH_2); 26.9 (CH_3); 30.7 (CH_3); 30.9 (CH); 31.2 (CH); 34.9 (CH_2); 41.5 (CH_2); 44.3 (CH_2); 46.9 (CH); 56.8 (C); 59.4 (CH); 75.0 (CH); 77.1 (CH and C); 86.8 (CH); 127.4 (CH); 128.9 (2 CH); 129.9 (C); 135.0 (2 CH). IR (Nujol dispersion): 3040, 3020, 1580, 750, 695, 670, 640 cm^{-1}. HRMS calcd for $C_{26}H_{42}NO_2Se$ $[M + H]^+$ 480.2377, found 480.2380.

(1*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-1-Isopropyl-4-phenyselenenyl-3,3,7,7,10-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (12b). Colorless oil. $[\alpha]_D^{25}$ = +4.2 (c = 0.2, CH_2Cl_2). ^{1}H NMR (δ): 0.72 (s, 3H); 0.75-1.15 (m, 3H); 0.81 (d, 3H, J = 6.9 Hz); 0.86 (s, 3H); 0.88 (d, 3H, J = 6.5 Hz); 0.89 (d, 3H, J = 6.9 Hz); 1.20-1.35 (m, 2H); 1.23 (s, 3H); 1.42 (s, 3H); 1.50-1.68 (m, 2H); 1.88 (m, 1H); 2.20 (m, 1H); 2.89 (dd, 1H, J_1 = 13.5 Hz, J_2 = 8.9 Hz); 3.08 (d, 1H, J =

13.5 Hz); 3.22 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.3 Hz); 3.35 (dd, 1H, J_1 = 7.7 Hz, J_2 = 2.5 Hz); 3.57 (d, 1H, J = 8.9 Hz); 3.83 (d, 1H, J = 7.7 Hz); 7.20-7.26 (m, 3H); 7.52-7.72 (m, 2H). ^{13}C NMR (δ): 12.6 (CH_3); 15.0 (CH_3); 20.4 (CH_3); 22.1 (CH_3); 25.1 (CH_3); 25.2 (CH_2); 25.8 (CH_3); 26.2 (CH_3); 28.3 (CH); 31.0 (CH); 34.7 (CH_2); 41.1 (CH_2); 47.0 (CH_2); 49.8 (CH); 55.6 (CH); 57.8 (C); 75.1 (CH); 75.8 (C); 76.5 (CH); 87.3 (CH); 127.3 (CH); 129.0 (2 CH); 130.0 (C); 134.3 (2 CH). IR (Film): 3045, 3020, 1580, 745, 700, 670 cm^{-1}. HRMS calcd for $C_{26}H_{42}NO_2Se$ $[M + H]^+$ 480.2377, found 480.2385 .

(1S,3S,6aS,9R,10aR,11aS)-1-Isopropyl-3-(1-methyl-1-phenylselenenylethy)-6,6,9-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazino[3,4-*b*][1,3]benzoxazine (14b). Colorless solid. Mp: 121-122 ºC (from ethanol). $[\alpha]_D^{25}$ = -0.2 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.83-1.14 (m, 3H); 0.93 (d, 3H, J = 7.0 Hz); 0.95 (d, 3H, J = 6.6 Hz); 0.97 (s, 3H); 1.02 (d, 3H, J = 7.0 Hz); 1.18 (s, 3H); 1.22-1.51 (m, 2H); 1.38 (s, 3H); 1.40 (s, 3H); 1.68-1.77 (m, 2H); 1.94 (m, 1H); 2.12 (m, 1H); 2.38 (dd, 1H, J_1 = 11.2 Hz, J_2 = 10.3 Hz); 3.10 (dd, 1H, J_1 = 7.9 Hz, J_2 = 2.3 Hz); 3.21 (dd, 1H, J_1 = 12.2 Hz, J_2 = 1.8 Hz); 3.35 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.3 Hz); 3.48 (dd, 1H, J_1 = 10.3 Hz, J_2 = 1.8 Hz); 3.82 (d, 1H, J = 7.9 Hz); 7.27-7.41 (m, 3H); 7.65-7.70 (m, 2H). ^{13}C NMR (δ): 11.4 (CH_3); 15.8 (CH_3); 20.4 (CH_3); 22.1 (CH_3); 25.0 (CH_2); 25.7 (CH_3); 26.0 (CH_3); 26.9 (CH_3); 27.8 (CH); 31.1 (CH); 34.6 (CH_2); 41.2 (CH_2); 43.6 (CH_2); 49.0 (C); 50.0 (CH); 55.6 (C); 74.3 (CH); 81.6 (CH); 82.2 (CH); 83.4 (CH); 127.4 (C); 128.4 (CH); 128.5 (2 CH); 138.4 (2 CH). IR (Nujol dispersion): 3040, 3020, 1585, 740, 690 cm^{-1}. HRMS calcd for $C_{26}H_{42}NO_2Se$ $[M + H]^+$ 480.2377, found 480.2380

(3*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-4-phenyselenenyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (12f). Colorless solid. Mp: 164-165 ºC (from hexane). $[\alpha]_D^{25}$ = +54.3 (c = 0.5 CH_2Cl_2). ^{1}H NMR (δ): 0.82-0.97 (m, 2H); 0.83 (s, 3H); 0.89 (s, 3H); 0.91 (d, 3H, J = 6.5 Hz); 1.10-1.43 (m, 3H); 1.27 (s, 3H); 1.55-1.68 (m, 2H); 1.90 (m, 1H); 2.82 (d, 1H, J = 14.8 Hz); 3.08 (d, 1H, J = 14.8 Hz); 3.33 (dt, 1H, J_1 = 10.6 Hz, J_2 = 4.1 Hz); 3.80 (dd, 1H, J_1 = 4.0

Hz, J_2 = 14.3 Hz); 4.18 (d, 1H, J = 14.3 Hz); 4.29 (d, 1H, J = 4.0 Hz); 4.50 (s, 1H); 7.20-7.39 (m, 6H); 7.41-7.45 (m, 2H); 7.50-7.54 (m, 2H). ^{13}C NMR (δ): 14.5 (CH_3); 20.5 (CH_3); 22.1 (CH_3); 25.1 (CH_2); 26.8 (CH_3); 31.2 (CH); 34.7 (CH_2); 41.2 (CH_2); 49.2 (CH); 53.4 (CH_2); 54.7 (C); 57.2 (C); 76.3 (CH); 77.4 (CH_2); 87.0 (CH); 92.3 (CH); 126.6 (C); 127.2 (2 CH); 127.5 (CH); 128.4 (2 CH); 128.6 (3 CH); 138.5 (2 CH); 139.3 (C). IR (Nujol dispersion): 3060, 3020, 1580, 745, 715, 700, 650 cm^{-1}. HRMS calcd for $C_{28}H_{38}NO_2Se$ $[M + H]^+$ 500.2062, found 500.2039.

(3S,6aS,9R,10aR,11aS)-3-[(S)-Chloro(phenyl)methyl]-3,6,6,9-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazino[3,4-*b*][1,3]benzoxazine (15f). Colorless solid. Mp: 166-167 ºC (from hexane/ethyl acetate). $[\alpha]_D^{25}$= +21.1 (c= 1.3, CH_2Cl_2). ^{1}H NMR (δ): 0.89-1.14 (m, 3H); 0.92 (d, 3H, J = 6.6 Hz); 0.94 (s, 3H); 1.18 (s, 3H); 1.29 (s, 3H); 1.34-1.50 (m, 2H); 1.57 (m, 1H); 1.65 (m, 1H); 1.93 (m, 1H); 2.35 (d, 1H, J = 11.5 Hz); 2.79 (d, 1H, J = 11.5 Hz); 3.46 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.1 Hz); 3.75 (dd, 1H, J_1 = 12.6 Hz, J_2 = 2.9 Hz); 3.96 (dd, 1H, J_1 = 12.6 Hz, J_2 = 2.9 Hz); 4.50 (t, 1H, J = 2.9 Hz); 5.65 (s, 1H); 7.32-7.47 (m, 3H); 7.48-7.51 (m, 2H). ^{13}C NMR (δ): 19.2 (CH_3); 19.4 (CH_3); 22.2 (CH_3); 24.9 (CH_2); 25.5 (CH_3); 31.3 (CH); 34.8 (CH_2); 41.2 (CH_2); 45.0 (CH); 46.1 (CH_2); 55.7 (C); 64.0 (CH); 64.2 (CH_2); 75.8 (CH); 75.9 (C); 80.2 (CH); 127.8 (2 CH); 128.2 (CH); 129.0 (2 CH); 138.5 (C). IR (Nujol dispersion): 3040, 3020, 750, 730, 700, 670 cm^{-1}. HRMS calcd for $C_{22}H_{33}ClNO_2$ $[M + H]^+$ 378.2194, found 378.2182.

(3R,6aS,9R,10aR,11aS)-3-[(R)-Chloro(phenyl)methyl]-3,6,6,9-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazino[3,4-*b*][1,3]benzoxazine (16f). Colorless oil. $[\alpha]_D^{25}$ = -63.1 (c = 0.6, CH_2Cl_2). ^{1}H NMR (δ): 0.84-1.05 (m, 2H); 0.90 (s, 3H); 0.95 (d, 3H, J = 6.6 Hz); 1.08 (m, 1H); 1.00 (s, 3H); 1.25 (s, 3H); 1.41-1.51 (m, 2H); 1.64-1.73 (m, 2H); 1.95 (m, 1H); 2.14 (d, 1H, J = 12.0 Hz); 2.68 (d, 1H, J = 12.0 Hz); 3.41 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.3 Hz); 3.68 (dd, 1H, J_1 = 11.8 Hz, J_2 = 7.8 Hz); 3.77 (dd, 1H, J_1 = 11.8 Hz, J_2 = 4.1 Hz); 4.21 (dd, 1H, J_1 = 7.8 Hz, J_2 = 4.1 Hz); 5.90 (s, 1H); 7.27-7.32 (m, 3H); 7.51-7.56 (m, 2H). ^{13}C NMR (δ): 12.5 (CH_3); 19.5

(CH_3); 22.1 (CH_3); 24.9 (CH_2); 25.5 (CH_3); 31.1 (CH); 34.6 (CH_2); 41.1 (CH_2); 48.1 (CH_2); 49.5 (CH); 55.7 (C); 63.7 (CH_2 and CH); 74.6 (CH); 76.2 (C); 81.5 (CH); 127.8 (2 CH); 128.2 (CH); 129.1 (2 CH); 138.3 (C). IR (Film): 3060, 3040, 1595, 750, 730, 700, 680, 640 cm^{-1}. HRMS calcd for $C_{22}H_{33}ClNO_2$ $[M + H]^+$ 378.2194, found 378.2182.

(3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-4-phenyselenenyl-3,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11g). Colorless oil. $[\alpha]_D^{25}$ = +39.0 (c = 0.9, $CHCl_3$). ^{1}H NMR (δ): 0.82-0.98 (m, 2H); 0.85 (s, 3H); 0.94 (d, 3H, *J* = 6.5 Hz); 0.99 (s, 3H); 1.14 (m, 1H); 1.29-1.60 (m, 3H); 1.55 (s, 3H); 1.68 (m, 1H); 1.96 (m, 1H); 2.98 (d, 1H, *J* = 13.0 Hz); 3.45 (td, 1H, J_1 = 10.4 Hz, J_2 = 4.2 Hz); 3.47 (d, 1H, *J* = 13.4 Hz); 3.63 (dd, 1H, J_1 = 13.0 Hz, J_2 = 8.3 Hz); 3.90 (dd, 1H, J_1 = 13.4 Hz, J_2 = 4.5 Hz); 4.10 (d, 1H, *J* = 8.3 Hz); 4.45 (d, 1H, *J* = 4.5 Hz); 7.18-730 (m, 6H); 7.55-7.70 (m, 4H). ^{13}C NMR (333 K) (δ): 18.1 (CH_3); 22.1 (CH_3); 25.2 (CH_2); 26.8 (CH_3); 27.6 (CH_3); 31.3 (CH); 35.8 (CH_2); 41.5 (CH_2); 44.6 (CH_2); 47.5 (CH); 57.0 (C); 57.6 (CH); 66.8 (CH_2); 75.6 (CH); 80.7 (C); 84.0 (CH); 126.1 (2 CH); 126.8 (CH); 127.2 (CH); 128.0 (2 CH); 129.1 (2 CH); 132.1 (C); 133.9 (2 CH); 147.2 (C). IR (Film): 3040, 3020, 1580, 760, 700, 670 cm^{-1}. HRMS calcd for $C_{28}H_{38}NO_2Se$ $[M + H]^+$ 500.2062, found 500.2048.

(3*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-4-phenyselenenyl-3,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (12g). Colorless oil. $[\alpha]_D^{25}$ = -0.3 (c = 1.1, CH_2Cl_2). ^{1}H NMR (δ): 0.65 (s, 3H); 0.80-1.00 (m, 3H); 0.87 (d, 3H, *J* = 6.5 Hz); 0.90 (s, 3H); 1.09 (m, 1H); 1.38 (m, 1H); 1.52 (s, 3H); 1.53-1.66 (m, 2H); 1.85 (m, 1H); 3.11 (dd, 1H, J_1 = 13.8 Hz, J_2 = 8.6 Hz); 3.20-3.28 (m, 2H); 3.32 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.2 Hz); 3.58 (dd, 1H, J_1 = 13.0 Hz, J_2 = 2.8 Hz); 4.15-4.22 (m, 2H); 7.20-7.38 (m, 6H); 7.68-7.74 (m, 2H); 7.80-7.85 (m, 2H). ^{13}C NMR (δ): 12.8 (CH_3); 22.0 (CH_3); 25.0 (CH_2); 25.6 (CH_3); 28.2 (CH_3); 31.0 (CH); 34.6 (CH_2); 40.9 (CH_2); 46.5 (CH_2); 49.6 (CH); 54.0 (CH); 57.2 (C); 67.7 (CH_2); 75.1 (CH); 80.4 (C); 84.6 (CH); 126.3 (2 CH); 127.2 (CH); 127.6 (CH); 128.0 (2 CH);

129.1 (2 CH); 130.1 (C); 134.1 (2 CH); 145.7 (C). IR (Film): 3040, 1580, 760, 730, 700, 660 cm^{-1}. HRMS calcd for $C_{28}H_{38}NO_2Se$ $[M + H]^+$ 500.2062, found 500.2075.

(1*S*,3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4-phenyselenenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11h). Colorless solid. Mp: 204-205 ºC, (from ethanol). $[\alpha]_D^{25}$= -59.5 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.78-0.98 (m, 2H); 0.90 (d, 3H, *J* = 6.4 Hz); 0.94 (s, 3H); 1.05 (m, 1H); 1.11 (s, 3H); 1.21 (m, 1H); 1.34 (m, 1H); 1.48 (m, 1H); 1.63 (m, 1H);1.82 (m, 1H); 3.27 (dd, 1H, J_1 = 12.6 Hz, J_2 = 1.0 Hz); 3.35 (td, 1H, J_1 = 10.3 Hz, J_2 = 3.5 Hz); 3.48-3.62 (m, 2H); 4.51-4.58 (m, 2H); 4.67 (d, 1H, *J* = 7.1 Hz); 7.12-7.42 (m, 15H). ^{13}C NMR (333 K) (δ): 19.7 (CH_3); 22.1 (CH_3); 25.1 (CH_2); 27.5 (CH_3); 31.2 (CH); 35.0 (CH_2); 41.4 (CH_2); 46.4 (CH_2); 47.8 (CH); 53.9 (CH); 57.1 (C); 75.2 (CH); 83.8 (CH); 89.1 (CH); 89.7 (CH); 126.6 (2 CH); 126.9 (CH); 127.3 (2 CH); 127.4 (CH); 127.7 (3 CH); 127.9 (2 CH); 128.8 (2 CH); 129.1 (C); 135.1 (2 CH); 141.9 (C); 142.0 (C). IR (Nujol dispersion): 3060, 1600, 1580, 740, 720, 690 cm^{-1}. HRMS calcd for $C_{33}H_{40}NO_2Se$ $[M + H]^+$ 562.2219, found 562.2241.

(1*S*,3*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4-phenyselenenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (12h). Colorless oil. $[\alpha]_D^{25}$ = +17,2 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.77-0.96 (m, 3H); 0.78 (d, 3H, *J* = 6.5 Hz); 0.99 (s, 6H); 1.11-1.62 (m, 5H); 3.11 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.0 Hz); 3.23 (dd, 1H, J_1 = 13.8 Hz, J_2 = 8.3 Hz); 3.51 (dd, 1H, J_1 = 13.8 Hz, J_2 = 1.0 Hz); 4.08 (ddd, 1H, J_1 = 9.0 Hz, J_2 = 8.3 Hz, J_3 = 1.0 Hz); 4.29 (d, 1H, *J* = 7.6 Hz); 4.32 (d, 1H, *J* = 7.6 Hz); 4.85 (d, 1H, *J* = 9.0 Hz); 7.13-7.27 (m, 11H); 7.39-7.44 (m, 2H); 7.59-7.62 (m, 2H). ^{13}C NMR (δ): 14.5 (CH_3); 22.0 (CH_3); 25.2 (CH_2); 26.7 (CH_3); 31.0 (CH); 34.7 (CH_2); 40.8 (CH_2); 47.0 (CH_2); 48.1 (CH); 49.3 (CH); 58.0 (C); 75.2 (CH); 77.9 (CH); 80.4 (CH); 88.4 (CH); 126.9 (CH); 127.5 (2 CH); 127.6 (2 CH); 127.7 (CH); 127.9 (CH); 128.0 (2 CH); 128.1 (2 CH); 129.1 (2 CH); 129.2 (C); 134.7 (2 CH); 139.1 (C); 140.9 (C). IR (Film): 3020, 3010, 1595, 1580, 750, 720, 690 cm^{-1}. HRMS calcd for $C_{33}H_{40}NO_2Se$ $[M + H]^+$ 562.2219, found 562.2234.

(1*S*,3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4-phenyselenenyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11i). Colorless oil. $[\alpha]_D^{25}$ = -67.2 (c = 1.0, CH_2Cl_2). ^{1}H NMR (333 K) (δ): 0.78-0.94 (m, 2H); 0.82 (s, 3H); 0.90 (d, 3H, *J* = 6.5 Hz); 1.02-1.20 (m, 2H); 1.09 (s, 3H); 1.32 (s, 3H); 1.35 (m, 1H); 1.48 (m, 1H); 1.63 (m, 1H); 1.87 (m, 1H); 3.04 (d, 1H, *J* = 14.8 Hz); 3.35 (td, 1H, J_1 = 10.4 Hz, J_2 = 4.0 Hz); 3.70 (d, 1H, *J* = 14.8 Hz); 4.45 (d, 1H, *J* = 7.5 Hz); 4.74 (d, 1H, *J* = 7.5 Hz); 4.76 (s, 1H); 7.10-7.30 (m, 9H); 7.40-7.59 (m, 6H). ^{13}C NMR (333 K) (δ): 20.2 (CH_3); 20.9 (CH_3); 22.2 (CH_3); 25.2 (CH_2); 27.5 (CH_3); 31.3 (CH); 35.2 (CH_2); 41.6 (CH_2); 47.8 (CH) 52.5 (CH_2); 54.7 (C); 56.9 (C); 75.5 (CH); 85.2 (CH); 90.2 (CH); 92.7 (CH); 126.2 (2 CH); 126.9 (CH); 127.1 (2 CH); 127.2 (CH); 127.8 (2 CH); 128.5 (2 CH); 128.6 (3 CH); 128.7 (C); 138.6 (2 CH); 139.8 (C); 142.5 (C). IR (Film): 3060, 3030, 1605, 1580, 7.40, 700 cm^{-1}. HRMS calcd for $C_{34}H_{42}NO_2Se$ $[M + H]^+$ 576.2375, found 576.2382.

(3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-4-phenyselenenyl-1,1,7,7,10-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11j). Colorless solid. Mp: 133-134 ºC (from ethanol). $[\alpha]_D^{25}$= -21.5 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.83-1.08 (m, 3H); 0.92 (d, 3H, *J* = 6.5 Hz); 0.95 (s, 3H); 1.17 (s, 3H); 1.24 (s, 3H); 1.29 (m, 1H); 1.41 (m, 1H); 1.43 (s, 3H); 1.60-1.72 (m, 2H); 1.89 (m, 1H); 3.08 (dd, 1H, J_1 = 14.4 Hz, J_2 = 2.6 Hz); 3.24 (dd, 1H, J_1 = 14.4 Hz, J_2 = 4.9 Hz); 3.35 (td, 1H, J_1 = 10.4 Hz, J_2 = 4.2 Hz); 3.63 (m, 1H); 3.97 (s, 1H); 4.96 (d, 1H, *J* = 7.2 Hz); 7.10-7.26 (m, 8H); 7.32-7.39 (m, 2H). ^{13}C NMR (δ): 13.9 (CH_3); 20.2 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 27.7 (CH_3); 29.4 (CH_3); 30.9 (CH); 34.7 (CH_2); 41.3 (CH_2); 47.0 (CH_2); 49.0 (CH); 54.6 (CH); 57.7 (C); 73.3 (CH); 78.2 (CH); 79.0 (C); 92.9 (CH); 126.3 (2 CH); 127.0 (2 CH); 128.0 (2 CH); 128.8 (2 CH); 130.4 (C); 134.2 (2 CH); 144.0 (C). IR (Nujol dispersion): 3060, 1600, 755, 735, 705, 695 cm^{-1}. HRMS calcd for $C_{29}H_{40}NO_2Se$ $[M + H]^+$ 514.2219, found 514.2204.

(3R,6aS,9R,10aR,11aS)-3-[(S)-Phenyl(phenylselenenyl)methyl]-1,1,6,6,9-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazino[3,4-*b*][1,3]benzoxazine (13j).

Colorless solid. Mp: 130-131 ºC (from ethanol). $[\alpha]_D^{25}$ = +84.1 (c = 1.0, CH_2Cl_2). ^{1}H-RMN (δ): 0.8-1.15 (m, 3H); 0.89 (d, 3H, *J* = 6.5 Hz); 1.03 (s, 3H); 1.08 (s, 3H); 1.13 (s, 3H); 1.23 (s, 3H); 1.27-1.49 (m, 3H); 1.63 (m, 1H); 1.37 (m, 1H); 2.74 (dd, 1H, J_1 = 10.5 Hz, J_2 = 10. 0 Hz); 2.95 (dd, 1H, J_1 = 10.0 Hz, J_2 = 1.1 Hz); 3.34 (td, 1H, J_1 = 10.5 Hz, J_2 = 3.9 Hz); 4.08 (s, 1H); 4.24-4.39 (m, 2H); 7.07-7.26 (m, 8H); 7.32-7.39 (m, 2H). ^{13}C-RMN (δ): 21.8 (CH_3); 22.2 (CH_3); 22.8 (CH_3); 25.1 (CH_2); 25.5 (CH_3); 26.7 (CH_3); 31.4 (CH); 35.0 (CH_2); 41.1 (CH_2); 43.5 (CH); 43.6 (CH_2); 51.7 (CH); 55.7 (C); 72.4 (CH); 73.9 (C); 76.6 (CH); 84.8 (CH); 126.4 (CH); 127.6 (CH); 127.7 (2 CH); 128.5 (2 CH); 128.6 (2 CH); 129.5 (C); 135.5 (2 CH); 140.6 (C). IR (Nujol dispersion): 3040, 1595, 1580, 770, 740, 700 cm^{-1}. HRMS calcd for $C_{29}H_{40}NO_2Se$ $[M + H]^+$ 514.2219, found 514.2208.

(1*R*,3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4-phenyselenenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (11k). Colorless oil. $[\alpha]_D^{25}$ = -44.8 (c = 0.5, $CHCl_3$). ^{1}H NMR (δ): 0.73-0.91 (m, 2H); 0.79 (d, 3H, *J* = 6.4 Hz); 0.82 (s, 3H); 1.07-1.47 (m, 4H); 1.08 (s, 3H); 1.55 (m, 1H); 1.72 (m, 1H); 3.22 (td, 1H, J_1 = 10.5 Hz, J_2 = 3.9 Hz); 3.26 (dd, 1H, J_1 = 12.9 Hz, J_2 = 1.0 Hz); 3.79 (dd, 1H, J_1 = 12.9 Hz, J_2 = 9.6 Hz); 3.93 (ddd, 1H, J_1 = 10.6 Hz, J_2 = 9.6 Hz, J_3 = 1.0 Hz); 4.50 (s, 1H); 4.73 (s, 1H); 4.82 (d, 1H, *J* = 10.6 Hz); 7.03-7.32 (m, 11H); 7.42-7.49 (m, 2H); 7.52-7.60 (m, 2H). ^{13}C NMR (δ): 22.2 (CH_3); 22.5 (CH_3); 25.1 (CH_2); 27.9 (CH_3); 31.3 (CH); 34.9 (CH_2); 41.1 (CH_2); 45.7 (CH); 46.7 (CH_2); 47.3 (CH); 57.8 (C); 76.0 (CH); 76.6 (CH); 80.5 (CH); 86.0 (CH); 126.8 (3 CH); 127.6 (2 CH); 127.9 (CH); 128.0 (3 CH); 128.2 (2 CH); 128.7 (C); 129.2 (2 CH); 135.1 (2 CH); 139.1 (C); 140.6 (C). IR (Film): 3030, 1600, 750, 700 cm^{-1}. HRMS calcd for $C_{33}H_{40}NO_2Se$ $[M + H]^+$ 562.2219, found 562,2241.

(1*R*,3*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4-phenyselenenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (12k). Colorless solid. Mp: 222-223 ºC (from ethanol). $[\alpha]_D^{25}$ = +7.4 (c = 0.7, CH_2Cl_2). ^{1}H NMR (δ): 0.81-0.95 (m, 2H); 0.86 (d, 3H, *J* = 6.5 Hz); 1.08 (m, 1H); 1.20 (s, 3H);

1.22 (s, 3H); 1.23-1.42 (m, 2H); 1.57 (m, 1H); 1.65 (m, 1H); 1.74 (m, 1H); 3.12 (dd, 1H, J_1 = 13.3 Hz, J_2 = 7.3 Hz); 3.28 (td, 1H, J_1 = 10.5 Hz, J_2 = 3.9 Hz); 3.76 (ddd, 1H, J_1 = 7.3 Hz, J_2 = 4.0 Hz, J_3 = 1.2 Hz); 3.92 (dd, 1H, J_1 = 13.3 Hz, J_2 = 1.2 Hz); 4.87 (s, 1H); 5.01 (d, 1H, J = 4.0 Hz); 5.36 (s, 1H); 7.04-7.09 (m, 2H); 7.12-7.37 (m, 9H); 7.40-7.48 (m, 2H); 7.55-7.62 (m, 2H). ^{13}C NMR (δ): 20.3 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 28.0 (CH_3); 31.3 (CH); 34.9 (CH_2); 40.9 (CH_2); 41.4 (CH_2); 47.0 (CH); 56.2 (CH); 57.6 (C); 76.8 (CH); 81.4 (CH); 86.0 (CH); 86.8 (CH); 125.9 (2 CH); 126.7 (3 CH); 127.1 (CH); 127.6 (3 CH); 128.0 (2 CH); 129.0 (2 CH); 130.2 (C); 135.5 (2 CH); 140.5 (C); 143.2 (C). IR (Nujol dispersion): 3060,3025, 1600, 760, 730, 710, 705 cm^{-1}. HRMS calcd for $C_{33}H_{40}NO_2Se$ $[M + H]^+$ 562.2219, found 562,2238.

(1*S*,3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1-Phenyl-3-phenyselenenylmethyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (27). Yield: 0.38 g, 58%. Colorless oil. $[\alpha]_D^{25}$ = +7.2 (c = 3.5, CH_2Cl_2). ^{1}H NMR (δ): 0.83 (m, 2H); 0.92 (d, 3H, J = 6.5 Hz); 1.07 (m, 1H); 1.16 (s, 3H); 1.20 (s, 3H); 1.26-1.47 (m, 2H); 1.60 (m, 1H); 1.63-1.82 (m, 2H); 1.88 (m, 1H); 1.99 (m, 1H); 2.97 (m, 1H); 3.01 (dd, 1H, J_1 = 12.2 Hz, J_2 = 5.6 Hz); 3.24 (m, 1H); 3.28 (dd, 1H, J_1 = 12.2 Hz, J_2 = 7.4 Hz); 3.36 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.1 Hz); 3.84 (m, 1H); 4.33 (d, 1H, J = 7.4 Hz); 4.70 (d, 1H, J = 7.4 Hz); 7.17-7.37 (m, 6H); 7.43-7.50 (m,2H); 7.55-7.59 (m, 2H). ^{13}C NMR (δ): 19.7 (CH_3); 22.1 (CH_3); 24.8 (CH_2); 27.6 (CH_3); 31.0 (CH); 33.6 (CH_2); 34.8 (CH_2); 38.7 (CH_2); 38.8 (CH_2); 41.1 (CH_2); 47.0 (CH); 56.6 (C); 74.8 (CH); 83.0 (CH); 83.8 (CH); 90.2 (CH); 126.3 (2 CH); 126.5 (CH); 126.7 (CH); 127.6 (2 CH); 128.7 (2 CH); 130.5 (C); 132.3 (2 CH); 141.8 (C). IR (Film): 3020, 1580, 5 755, 735, 690 cm-1. HRMS calcd for $C_{28}H_{38}NO_2Se$ $[M + H]^+$ 500.2062, found 500.2042.

(1*S*,3*R*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1-Phenyl-3-phenyselenenylmethyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (28). Yield: 91 mg, 14%. Colorless oil. Purity 90%. ^{1}H NMR (δ): 0.82-1.07 (m, 3H); 0.83

(d, 3H, *J* = 6.5 Hz); 0.99 (s, 3H); 1.19-1.31 (m, 2H); 1.21 (s, 3H); 1.54-1.67 (m, 4H); 1.85 (m, 1H); 2.29 (m, 1H); 2.63 (dd, 1H, J_1 = 12.9 Hz, J_2 = 8.7 Hz); 2.93 (dd, 1H, J_1 = 12.1 Hz, J_2 = 8.2 Hz); 3.13 (dd, 1H, J1 = 12.1 Hz, J2 = 6.0 Hz), 3.16 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.0 Hz); 4.06 (m, 1H); 4.20 (d, 1H, *J* = 7.4 Hz); 4.59 (d, 1H, *J* = 7.4 Hz); 7.17-7.43 (m, 10H). ^{13}C NMR (δ): 14.2 (CH_3); 22.1 (CH_3); 25.3 (CH_2); 27.0 (CH_3); 31.1 (CH); 31.9 (CH_2); 34.8 (CH_2); 35.1 (CH_2); 39.0 (CH_2); 40.9 (CH_2); 49.2 (CH); 57.5 (C); 75.5 (2 CH); 76.1 (CH); 89.4 (CH); 126.9 (CH); 127.1 (CH); 127.6 (4 CH); 129.0 (2 CH); 130.2 (C); 132.7 (2 CH); 141.2 (C). IR (Film): 3020, 1580, 750, 735, 695 cm-1. HRMS calcd for $C_{28}H_{38}NO_2Se$ $[M + H]^+$ 500.2062, found 500.2070.

Characterization data for deselenylation products.

(1*S*,7a*S*,10*R*,11a*R*,12a*S*)-1-Isopropyl-3,3,7,7,10-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17b). This compound was obtained from **11b** (0.52 g). Yield: 0.33 g, 92%. Colorless oil. $[\alpha]_D^{25}$ = -25.8 (c = 0.3, CH_2Cl_2). ^{1}H NMR (δ): 0.81-0.95 (m, 2H); 0.82 (d, 3H, *J* = 6.7 Hz); 0.88 (d, 3H, *J* = 6.5 Hz); 0.92 (d, 3H, *J* = 6.3 Hz); 0.97 (s, 3H); 1.04 (m, 1H); 1.11 (s, 3H); 1.13 (s, 3H); 1.18 (s, 3H); 1.30 (m, 1H); 1.43 (m, 1H); 1.63-1.73 (m, 3H); 1.81-1.94 (m, 2H); 2.18 (m, 1H); 2.44 (dd, 1H, J_1 = 13.2 Hz, J_2 = 9.0 Hz); 2.82 (dd, 1H, J_1 = 13.2 Hz, J_2 = 8.3 Hz); 3.26-3.73 (m, 2H), 3.94 (d, 1H, *J* = 7.6 Hz). ^{13}C-NMR (δ): 13.5 (CH_3); 15.3 (CH_3); 20.3 (CH_3); 22.1 (CH_3); 25.3 (CH_2); 26.4 (CH_3); 26.9 (CH_3); 28.6 (CH); 29.0 (CH_3); 31.1 (CH); 34.8 (CH_2); 37.8 (CH_2); 41.3 (CH_2); 41.7 (CH_2); 49.4 (CH); 57.1 (C); 73.3 (C); 75.2 (CH); 76.5 (CH); 88.2 (CH). HRMS calcd for $C_{20}H_{38}NO_2$ $[M + H]^+$ 324.2897, found 324.2900.

(3*S*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17e). This compound was obtained from a mixture 30/70 of **12e**/**13e** (2.85 g). Yield: 0.51 g, 26%. Colorless oil. [α]

$_D^{25}$ = -14.7 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.82-1.10 (m, 3H); 0.87 (d, 3H, *J* = 6.5 Hz); 1.05 (s, 3H); 1.14 (s, 3H); 1.28 (m, 1H); 1.39 (m, 1H); 1.57-1.68 (m, 2H); 1.88 (m, 1H); 1.95 (m, 1H); 2.19 (m, 1H); 2.73 (dd, 1H, J_1 = 13.7 Hz, J_2 = 8.0 Hz); 3.01 (dd, 1H, J_1 = 13.7 Hz, J_2 = 9.0 Hz); 3.40 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.1 Hz); 3.56 (dd, 1H, J_1 = 13.2 Hz, J_2 = 7.8 Hz); 4.02 (dd, 1H, J_1 = 13.2 Hz, J_2 = 3.8 Hz); 4.58-4.65 (m, 2H); 7.11-7.28 (m, 5H). ^{13}C NMR (δ): 16.6 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 27.0 (CH_3); 31.1 (CH); 34.8 (CH_2); 38.2 (CH_2); 38.5 (CH_2); 41.2 (CH_2); 48.3 (CH); 57.0 (C); 71.4 (CH_2); 75.2 (CH); 81.4 (CH); 85.4 (CH); 125.8 (2 CH); 127.0 (CH); 128.2 (2 CH); 143.6 (C). IR (Film): 3020, 1600, 700 cm^{-1}. HRMS calcd for $C_{21}H_{32}NO_2$ [M + H]$^+$ 330.2428, found 330.2411.

(3*R*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (18e). This compound was obtained from a mixture 30/70 of **12e**/**13e** (2.85 g). Yield: 1.28g, 66%. Colorless oil. [α] $_D^{25}$ = +40,5 (c = 0.7, CH_2Cl_2) ^{1}H NMR (δ): 0.83-0.98 (m, 2H); 0.92 (d, 3H, *J* = 6.5 Hz); 0.99 (s, 3H); 1.10 (m, 1H); 1.19 (s, 3H); 1.21-1.48 (m, 2H); 1.63-1.71 (m, 2H); 1.93 (m, 1H); 2.12 (m, 1H); 2.23 (m, 1H); 2.57 (dd, 1H, J_1 = 13.6 Hz, J_2 = 9.3 Hz); 3.12 (m, 1H); 3.39 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.0 Hz); 3.75 (dd, 1H, J_1 = 13.6 Hz, J_2 = 2.9 Hz); 3.98 (dd, 1H, J_1 = 13.6 Hz, J_2 = 4.5 Hz); 4.28 (dd, 1H, J_1 = 2.9 Hz, J_2 = 4.5 Hz); 4.59 (dd, 1H, J_1 = 10.7 Hz, J_2 = 5.2 Hz); 7.17-7.38 (m, 5H). ^{13}C NMR (δ): 13.7 (CH_3); 22.1 (CH_3); 25.1 (CH_2); 26.5 (CH_3); 31.2 (CH); 34.7 (CH_2); 38.1 (CH_2); 39.9 (CH_2); 41.1 (CH_2); 48.9 (CH); 57.0 (C); 71.8 (CH_2); 75.8 (CH); 81.5 (CH); 87.1 (CH); 125.8 (2 CH); 127.0 (CH); 128.1 (2 CH); 143.1 (C). IR (Film): 3040, 3020, 1595, 740, 730, 695 cm^{-1}. HRMS calcd for $C_{21}H_{32}NO_2$ [M + H]$^+$ 330.2428, found 330.2425.

(3*S*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-3,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17g). This compound was obtained from **11g** (0.85 g). Yield: 0.54 g, 90%. Colorless oil. $[\alpha]_D^{25}$ = -12.2 (c = 0.3, CH_2Cl_2). ^{1}H NMR (δ): 0.83-1.02 (m, 2H); 0.92 (d, 3H, *J* = 6.5 Hz); 0.97 (s, 3H);

1.04-1.46 (m, 3H); 1.15 (s, 3H); 1.43 (s, 3H); 1.60-1.68 (m, 2H); 1.96 (m, 1H); 2.22-2.48 (m, 3H); 2.96 (dd, 1H, J_1 = 12.2 Hz, J_2 = 8.2 Hz); 3.45 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.2 Hz); 3.60 (dd, 1H, J_1 = 13.5 Hz, J_2 = 1.9 Hz); 3.81 (dd, 1H, J_1 = 13.5 Hz, J_2 = 6.3 Hz); 4.36 (dd, 1H, J_1 = 6.3 Hz, J_2 = 1.9 Hz); 7.21 (m, 1H); 7.28-7.34 (m, 2H); 7.40-7.45 (m, 2H). ^{13}C NMR (δ): 16.0 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 27.0 (CH_3); 29.4 (CH_3); 31.2 (CH); 34.8 (CH_2); 36.9 (CH_2); 41.3 (CH_2); 42.2 (CH_2); 48.0 (CH); 56.5 (C); 66.5 (CH_2); 75.7 (CH); 78.7 (C); 85.6 (CH); 125.3 (2 CH); 126.3 (CH); 128.0 (2 CH); 148.0 (C). IR (Film): 3050, 3025, 1600, 760, 700 cm^{-1}. HRMS calcd for $C_{22}H_{34}NO_2$ $[M + H]^+$ 344.2584, found 344.2569.

(3*R*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-3,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (18g). This compound was obtained from **12g** (1.65 g). Yield: 0.98 g, 89%. Colorless oil. $[\alpha]_D^{25}$ = -17.6 (c = 0.8, CH_2Cl_2). ^{1}H NMR (δ): 0.85-1.10 (m, 3H); 0.89 (d, 3H, J = 6.5 Hz); 0.98 (s, 3H); 1.18 (s, 3H); 1.22 (m, 1H); 1.35 (s, 3H); 1.43 (m, 1H); 1.61-1.70 (m, 2H); 1.87 (m, 1H); 2.31 (dd, 1H, J_1 = 14.4 Hz, J_2 = 8.7 Hz); 2.41 (dd, 1H, J_1 = 14.4 Hz, J_2 = 7.6 Hz); 2.57 (dd, 1H, J_1 = 13.6 Hz, J_2 = 8.7 Hz); 2.97 (dd, 1H, J_1 = 13.6 Hz, J_2 = 7.6 Hz); 3.35 (td, 1H, J_1 = 10.6 Hz, J_2 = 4.0 Hz); 3.36 (dd, 1H, J_1 = 13.0 Hz, J_2 = 8.6 Hz); 3.67 (dd, 1H, J_1 = 13.0 Hz, J_2 = 2.6 Hz); 4.22 (dd, 1H, J_1 = 8.6 Hz, J_2 = 2.6 Hz); 7.17-7.37 (m, 5H). ^{13}C NMR (δ): 12.6 (CH_3); 22.1 (CH_3); 25.1 (CH_2); 26.4 (CH_3); 31.1 (CH_3); 31.4 (CH); 34.7 (CH_2); 37.1 (CH_2); 41.2 (2 CH_2); 49.7 (CH); 56.6 (C); 68.2 (CH_2); 75.2 (CH); 78.7 (C); 86.0 (CH); 125.0 (2 CH); 126.3 (CH); 128.1 (2 CH); 148.2 (C). IR (Film): 3040, 1590, 770, 730, 700, 695 cm^{-1}. HRMS calcd for $C_{22}H_{34}NO_2$ $[M + H]^+$ 344.2584, found 344.2581.

(*1S*,3*S*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17h). This compound was obtained from **11h** (0.95 g). Yield: 0.66 g, 96%. Colorless oil. $[\alpha]_D^{25}$ = -89.2 (c = 1.0, CH_2Cl_2). ^{1}H NMR (δ): 0.82-0.93 (m, 2H); 0.90 (d, 3H, J = 6.5 Hz); 1.06 (m, 1H); 1.12 (s, 3H); 1.19 (s, 3H); 1.24-1.42 (m, 2H); 1.55 (m, 1H); 1.64 (m, 1H); 1.85 (m,

1H); 2.01 (m, 1H); 2.13 (m, 1H); 2.97 (m, 1H); 3.26-3.38 (m, 2H); 4.49 (d, 1H, *J* = 7.5 Hz); 4.68 (d, 1H, *J* = 7.5 Hz); 4.71 (dd, 1H, J_1 = 10.5 Hz, J_2 = 4.2 Hz); 7.20-7.34 (m, 6H); 7.37-7.40 (m, 2H); 7.51-7.55 (m, 2H). ^{13}C NMR (δ): 19,4 (CH_3); 22,2 (CH_3); 25,0 (CH_2); 27,7 (CH_3); 31,2 (CH); 35,0 (CH_2); 39,2 (CH_2); 40,6 (CH_2); 41,3 (CH_2); 47,6 (CH); 56,8 (C); 75,0 (CH); 84,4 (CH); 84,5 (CH); 90,6 (CH); 125,7 (2 CH); 126,6 (2 CH); 126,8 (CH); 126,9 (CH); 127,7 (2 CH); 128,0 (2 CH); 142,4 (C); 143,6 (C). IR (Film): 3060, 3030, 1600, 755, 740, 700 cm^{-1}. HRMS calcd for $C_{27}H_{36}NO_2$ $[M + H]^+$ 406.2741, found 406.2731.

(*1S*,3*R*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (18h). This compound was obtained from **12h** (0.28 g). Yield: 0.19 g, 94% Colorless oil. $[\alpha]_D^{25}$ = -11.5 (c = 0.2, CH_2Cl_2). ^{1}H NMR (δ): 0.73-0.95 (m, 3H); 0.79 (d, 3H, *J* = 6.5 Hz); 1.04 (s, 3H); 1.10-1.33 (m, 2H); 1.25 (s, 3H); 1.50-1.62 (m, 3H); 2.36 (m, 1H); 2.45 (m, 1H); 2.84 (m, 1H); 3.14 (td, 1H, J_1 = 10.5 Hz, J_2 = 4.1 Hz); 3.22 (dd, 1H, J_1 = 12.2 Hz, J_2 = 8.7 Hz); 4.31 (d, 1H, *J* = 7.6 Hz); 4.50 (d, 1H, *J* = 7.6 Hz); 5.04 (dd, 1H, J_1 = 10.0 Hz, J_2 = 6.8 Hz); 7.10-7.34 (m, 10H). ^{13}C NMR (δ): 14.6 (CH_3); 22.1 (CH_3); 25.3 (CH_2); 27.1 (CH_3); 31.1 (CH); 34.8 (CH_2); 35.3 (CH_2); 38.9 (CH_2); 40.9 (CH_2); 49.0 (CH); 57.5 (C); 75.4 (CH); 76.9 (CH); 77.2 (CH); 89.7 (CH); 126.3 (2 CH); 126.9 (2 CH); 127.6 (2 CH); 128.1 (2 CH); 130.5 (2 CH); 141.5 (C); 142.5 (C). IR (Film): 3050, 3020, 1600, 750, 690, 635 cm^{-1}. HRMS calcd for $C_{27}H_{36}NO_2$ $[M + H]^+$ 406.2741, found 406.2725.

(*1S*,3*S*,4*R*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17i). This compound was obtained from **11i** (0.96 g). Yield: 0.46 g, 65%. Colorless oil. $[\alpha]_D^{25}$ = -85.5 (c = 1.0, CH_2Cl_2). ^{1}H NMR (333 K) (δ): 0.76 (d, 3H, *J* = 6.8 Hz); 0.88-1.00 (m, 2H); 0.91 (d, 3H, *J* = 6.5 Hz); 1.08 (m, 1H); 1.13 (s, 3H); 1.20 (s, 3H); 1.27-1.46 (m, 2H); 1.60 (m, 1H); 1.67 (m, 1H); 1.87 (m, 1H); 2.07 (m, 1H); 2.92 (dd, 1H, J_1 = 14.6 Hz, J_2 = 3.5 Hz); 3.11 (dd, 1H, J_1 = 14.6 Hz, J_2 = 8.0 Hz); 3.36 (td, 1H, J_1 = 10.5 Hz,

J_2 = 4.1 Hz); 4.10 (d, 1H, *J* = 10.1 Hz); 4.42 (d, 1H, *J* = 7.5 Hz); 4.62 (d, 1H, *J* = 7.5 Hz); 7.11-7.30 (m, 6H); 7.32-7.39 (m, 2H); 7.46-7.50 (m, 2H). ^{13}C NMR (333 K) (δ): 15.7 (CH_3); 19.2 (CH_3); 22.2 (CH_3); 25.3 (CH_2); 27.8 (CH_3); 31.4 (CH); 35.2 (CH_2); 41.6 (CH_2); 43.7 (CH); 47.3 (CH_2); 48.0 (CH); 56.7 (C); 75.0 (CH); 85.1 (CH); 90.6 (CH); 92.0 (CH); 126.7 (2 CH); 126.8 (CH); 127.2 (CH); 127.4 (2 CH); 127.6 (2 CH); 128.0 (2 CH); 142.7 (C); 143.1 (C). IR (Film): 3050, 3030, 1600, 1495, 750, 700 cm^{-1}. HRMS calcd for $C_{28}H_{38}NO_2$ $[M + H]^+$ 420.97, found 420.2891.

(*1S*,3*S*,4*S*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-4,7,7,10-tetramethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (*epi*-17i). This compound was obtained from **11i** (0.96 g). Yield: 0.18 g, 26%. Colorless oil. $[\alpha]_D^{25}$ = -84.0 (c = 0.8, CH_2Cl_2). ^{1}H NMR (δ): 0.67 (d, 3H, *J* = 6.9 Hz); 0.81-1.00 (m, 2H); 0.91 (d, 3H, *J* = 6.6 Hz); 1.03-1.46 (m, 3H); 1.08 (s, 3H); 1.18 (s, 3H); 1.59-1.74 (m, 2H); 1.86 (m, 1H); 2.11 (m, 1H); 2.71 (dd, 1H, J_1 = 14.3 Hz, J_2 = 7.3 Hz); 3.29-3.39 (m, 2H); 4.41 (d, 1H, *J* = 7.3 Hz); 4.52 (d, 1H, *J* = 7.3 Hz); 4.99 (d, 1H, *J* = 4.1 Hz); 7.11-7.37 (m, 8H); 7.50-7.56 (m, 2H). ^{13}C NMR (δ): 12.5 (CH_3); 18.1 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 27.5 (CH_3); 31.2 (CH); 35.0 (CH_2); 39.6 (CH); 41.4 (CH_2); 46.5 (CH_2); 48.2 (CH); 56.7 (C); 74.6 (CH); 85.8 (CH); 86.6 (CH); 91.2 (CH); 125.5 (2 CH); 126.1 (CH); 126.6 (2 CH); 126.9 (CH); 127.7 (4 CH); 142.3 (C); 142.6 (C). IR (Film): 3060, 3030, 1600, 1495, 740, 700 cm^{-1}. HRMS calcd for $C_{28}H_{38}NO_2$ $[M + H]^+$ 420.97, found 420.2877.

(3*S*,7a*S*,10*R*,11a*R*,12a*S*)-3-Phenyl-1,1,7,7,10-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (17j). This compound was obtained from **11j** (1.45 g). Yield: 0.98 g, 98%. Colorless solid. Mp: 98-99 ºC (from ethanol). $[\alpha]_D^{25}$ = -42.6 (c = 0.7, CH_2Cl_2). ^{1}H NMR (δ): 0.87-1.04 (m, 3H); 0.92 (d, 3H, *J* = 6.5 Hz); 0.95 (s, 3H); 1.13 (s, 3H); 1.23-1.50 (m, 2H); 1.30 (s, 3H); 1.35 (s, 3H); 1.62-1.70 (m, 2H); 1.88-1.99 (m, 2H); 2.21 (m, 1H); 2.55 (ddd, 1H, J_1 = 13.7 Hz, J_2 = 10.5 Hz, J_3 = 2.2 Hz); 2.86 (ddd, 1H, J_1 = 13.7 Hz, J_2 = 6.1 Hz, J_3 = 2.8 Hz); 3.33 (td, 1H, J_1 = 10.4 Hz, J_2 = 4.3 Hz); 3.92 (s, 1H); 4.94 (t, 1H, *J* = 6.7 Hz); 7.18

(m, 1H); 7.24-7.30 (m, 2H); 7.32-7.37 (m, 2H). ^{13}C NMR (δ): 13.9 (CH_3); 20.4 (CH_3); 22.2 (CH_3); 25.1 (CH_2); 27.0 (CH_3); 29.3 (CH_3); 31.0 (CH); 34.8 (CH_2); 39.1 (CH_2); 40.0 (CH_2); 41.5 (CH_2); 49.1 (CH); 57.2 (C); 71.2 (CH); 73.6 (CH); 78.3 (C); 93.1 (CH); 125.6 (2 CH); 126.4 (CH); 128.1 (2 CH); 146.0 (C). IR (Nujol dispersion): 3060, 3030, 1605, 745, 725, 700, 660 cm^{-1}. HRMS calcd for $C_{23}H_{36}NO_2$ $[M + H]^+$ 358.2741, found 358.2720.

(3S,6a*S*,9*R*,10a*R*,11a*S*)-3-Benzyl-1,1,6,6,9-pentamethyl-decahydro-3*H*,7*H*-[1,4]oxazino[3,4-*b*][1,3]benzoxazine (19j). This compound was obtained from **13j** (0.16 g). Yield: 0.10 g, 89%. Colorless oil. $[\alpha]_D^{25}$ = -38.3 (c = 0.5, CH_2Cl_2). ^{1}H NMR (δ): 0.79-0.93 (m, 2H); 0.85 (d, 3H, *J* = 6.5 Hz); 0.90 (s, 3H); 1.03 (s, 3H); 1.09 (m, 1H); 1.15 (s, 3H); 1.20 (s, 3H); 1.30-1.48 (m, 3H); 1.64 (m, 1H); 1.84 (m, 1H); 2.41 (dd, 1H, J_1 = 10.6 Hz, J_2 = 2.7 Hz); 2.56 (dd, 1H, J_1 = 13.6 Hz, J_2 = 7.6 Hz); 2.73 (t, 1H, *J* = 10.6 Hz); 2.86 (dd, 1H, J_1 = 13.6 Hz, J_2 = 5.9 Hz); 3.32 (td, 1H, J_1 = 10.4 Hz, J_2 = 3.9 Hz); 3.90 (m, 1H); 4.06 (s, 1H); 7.07-7.20 (m, 5H). ^{13}C NMR (δ): 21.7 (CH_3); 22.3 (CH_3); 23.2 (CH_3); 25.2 (CH_2); 25.9 (CH_3); 26.5 (CH_3); 31.5 (CH); 35.1 (CH_2); 40.9 (CH_2); 41.3 (CH_2); 43.5 (CH); 44.0 (CH_2); 55.6 (C); 71.1 (CH); 73.6 (C); 76.8 (CH); 85.2 (CH); 126.0 (CH); 128.2 (2 CH); 129.2 (2 CH); 138.8 (C). IR (Film): 3060, 3035, 1605, 740, 725, 710, cm^{-1}. HRMS calcd for $C_{23}H_{36}NO_2$ $[M + H]^+$ 538.2741, found 358.2720.

(*1R*,3*R*,7a*S*,10*R*,11a*R*,12a*S*)-1,3-Diphenyl-7,7,10-trimethyl-decahydro-3*H*,7*H*-[1,4]oxazepino[3,4-b][1,3]benzoxazine (18k). This compound was obtained from **12k** (1.10 g). Yield: 0.75 g, 94%. Colorless oil. $[\alpha]_D^{25}$ = +8.5 (c = 0.9, CH_2Cl_2). ^{1}H NMR (δ): 0.79-1.06 (m, 2H); 0.85 (d, 3H, *J* = 6.5 Hz); 1.06-1.48 (m, 3H); 1.13 (s, 3H); 1.16 (s, 3H); 1.55 (m, 1H); 1.63 (m, 1H); 1.71 (m, 1H); 2.12 (m, 1H); 2.39 (m, 1H); 2.77 (dd, 1H, J_1 = 13.6 Hz, J_2 = 9.0 Hz); 3.17-3.32 (m, 2H); 4.72 (s, 1H); 4.83 (t, 1H, *J* = 5.9 Hz); 4.84 (s, 1H); 7.16-7.36 (m, 6H); 7.43- 7.54 (m, 4H). ^{13}C NMR (δ): 18.8 (CH_3); 22.1 (CH_3); 25.2 (CH_2); 27.6 (CH_3); 31.2 (CH); 34.9 (CH_2); 37.8 (CH_2); 39.4 (CH_2); 40.9 (CH_2); 46.8 (CH); 57.2 (C); 76.4 (CH); 80.8 (CH); 81.9

(CH); 88.5 (CH); 126.1 (2 CH); 126.7 (2 CH); 126.9 (2 CH); 127.6 (2 CH); 127.9 (2 CH); 140.9 (C); 144.0 (C). IR (Film): 3060, 3030, 1600, 750, 720, 700 cm^{-1}. HRMS calcd for $C_{27}H_{36}NO_2$ $[M + H]^+$ 406.2741, found 406.2723.

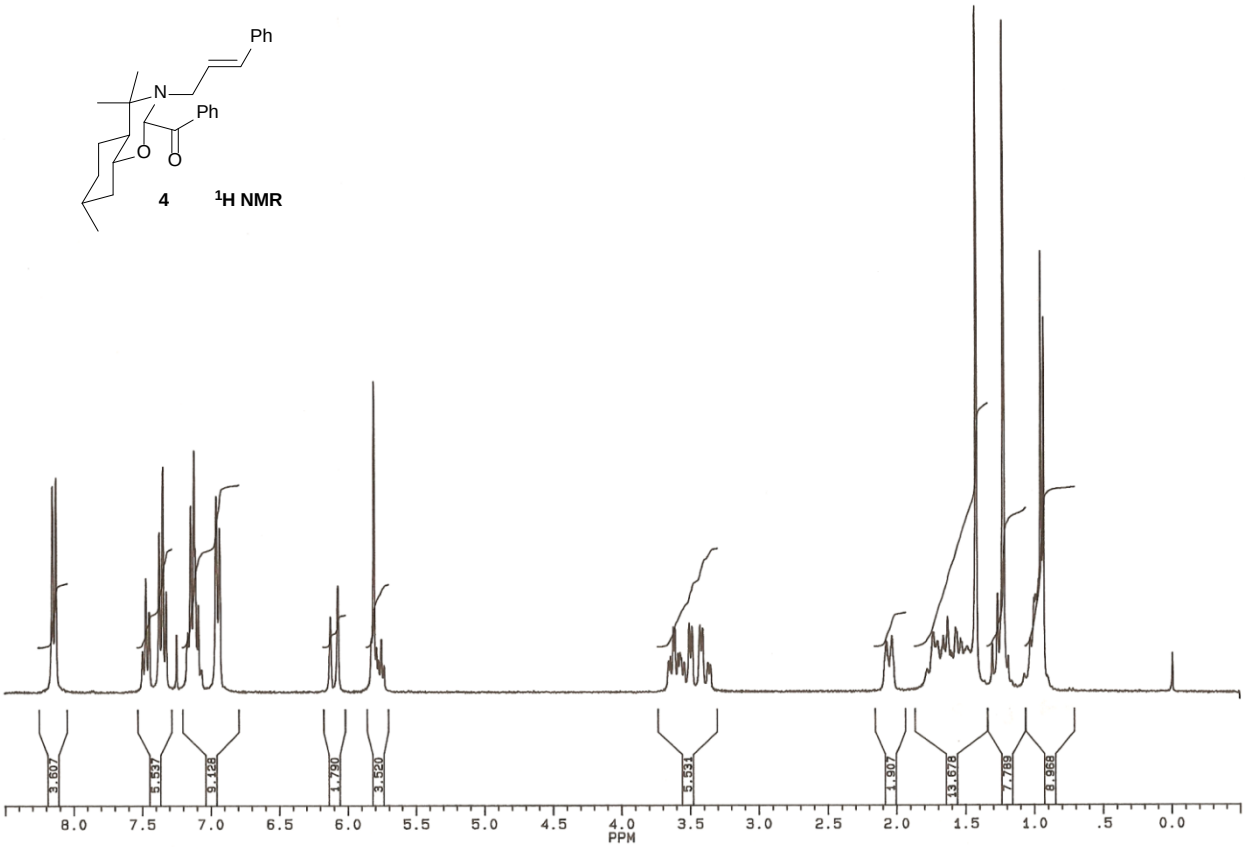
General procedure for preparation of amino alcohols 20 and 21 and characterization data.

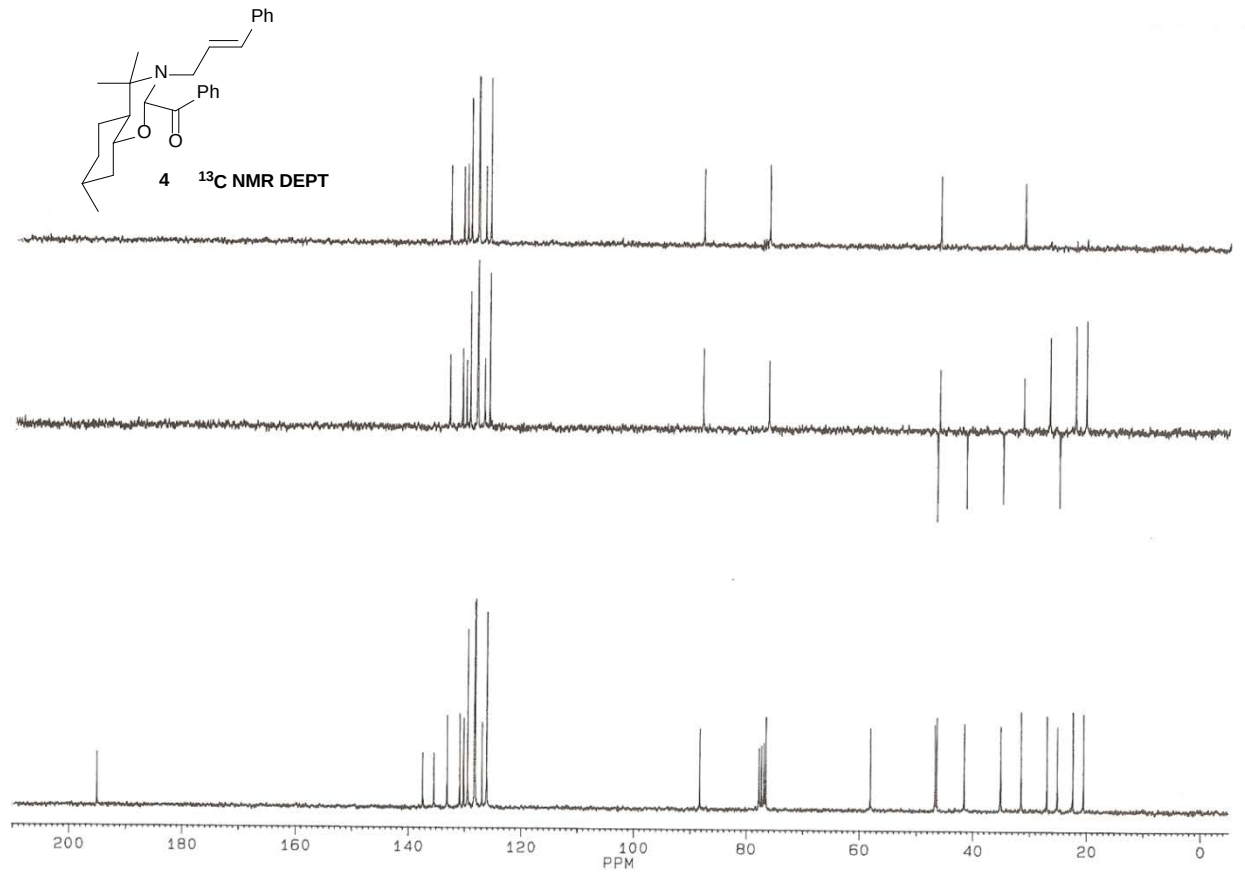
To a suspension of $LiAlH_4$ (0.57 g, 15.0 mmol) in anhydrous THF (25 mL) cooled to -10 ºC and under nitrogen atmosphere was added, in portions, dry $AlCl_3$ (0.67 g, 5.0 mmol). The mixture was stirred for 15 min at -10ºC and a solution of the corresponding benzoxazine **11j** or **18g** (2.0 mmol) in dry THF (15 mL) was slowly added. The reaction mixture was stirred at 0 ºC until disappearance of the starting product (TLC) (additional reflux for 25 min was necessary for reduction of oxazine **11j**), and then it was quenched by addition of 10 % aqueous solution of NaOH (1.5 mL). The resulting mixture was filtered, the solid was washed with hot EtOAc, and the organic layer was dried over anhydrous $MgSO_4$. The solvent was eliminated under reduced pressure, and the residue was chromatographed on silica gel using hexanes/AcOEt 4:1 as eluent.

.**(6*S*,7*R*)-2,2-Dimethyl-4-(8-mentholyl)-7-phenyl-6-phenylselenenyl-1,4-oxazepane (20).** This compound was obtained from **11j** (0.30 g). Yield: 0.25 g, 82%. Colorless solid. Mp: 152-153 ºC (from hexane-EtOAc). $[\alpha]_D^{25}$ = -15.6 (c = 1.3, CH_2Cl_2). ^{1}H NMR (333 K) (δ): 0.83 (s, 3H); 0.84-0.93 (m, 2H); 0.91 (d, 3H, *J* = 6.5 Hz); 1.02 (s, 3H); 1.10 (m, 1H); 1.21 (s, 3H); 1.34 (s, 3H); 1.38 (m, 1H); 1.56 (m, 1H); 1.60-1.70 (m, 2H); 1.98 (m, 1H); 2.50 (d, 1H, *J* = 14.0 Hz); 2.78 (m, 1H); 2.90 (d, 1H, *J* = 14.0 Hz); 3.46 (dd, 1H, J_1 = 13.4 Hz, J_2 = 4.6 Hz); 3.51-3.63 (m, 2H); 4.54 (d, 1H, *J* = 10.1 Hz); 7.04-7.32 (m, 11H). ^{13}C NMR (333 K) (δ): 19.2 (CH_3); 21.9 (CH_3); 22.2 (CH_3); 24.8 (CH_3); 26.1 (CH_2); 29.1 (CH_3);31.0 (CH); 35.2 (CH_2); 44.6 (CH_2); 48.1 (CH); 50.7 (CH); 59.0 (CH_2); 62.0 (C); 62.5 (CH_2); 73.1

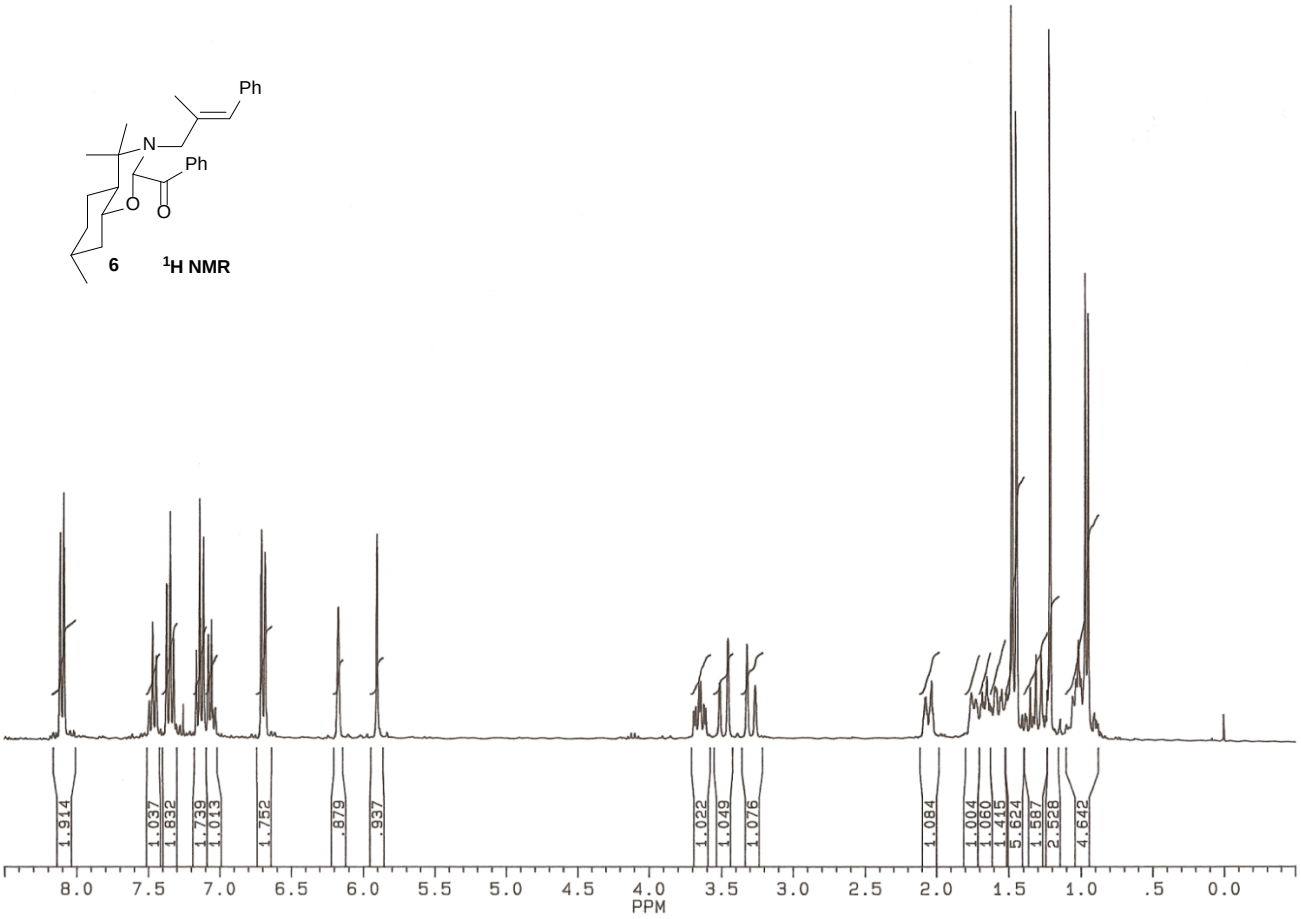
(CH); 74.5 (C); 77.7 (CH); 127.2 (3 CH); 127.5 (CH); 127.8 (2 CH); 128.6 (2 CH); 129.6 (C); 134.7 (2 CH); 142.0 (C). IR (Nujol dispersion): 3100 (broad), 3060, 3030, 1575, 750, 720, 695, 670 cm^{-1}. HRMS calcd for $C_{29}H_{42}NO_2Se$ $[M + H]^+$ 516.2375, found 516.2365.

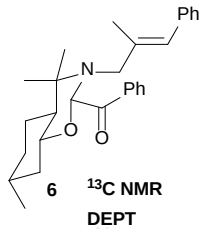
(7*R*)-4-(8-Mentholyl)-7-methyl-7-phenyl-1,4-oxazepane (21). This compound was obtained from **18g** (0.70 g). Yield: 0.64 g, 91%. Colorless solid. Mp: 142-143 ºC (from hexane). $[\alpha]_D^{25}$ = -46.5 (c = 0.9, CH_2Cl_2). ^{1}H NMR (333 K) (δ): 0.88-1.08 (m, 3H); 0.91 (d, 3H, *J* = 6.5 Hz); 0.95 (s, 3H); 1.14 (s, 3H); 1.37 (s, 3H); 1.47 (m, 1H); 1.56-1.74 (m, 3H); 1.93 (m, 1H); 2.26-2.48 (m, 2H); 2.68 (m, 1H); 2.72-2.93 (m, 2H); 3.08 (m, 1H); 3.52 (m, 1H); 3.58 (td, 1H, J_1 = 10.1 Hz, J_2 = 4.0 Hz); 3.77 (m, 1H); 7.18 (m, 1H); 7.22-7.35 (m, 4H); 8.08 (s broad, 1H). ^{13}C NMR (δ): 18.1 (CH_3); 21.4 (CH_3); 21.9 (CH_3); 26.0 (CH_2); 30.9 (CH); 31.2 (CH_3); 35.2 (CH_2); 40.5 (CH_2); 42.6 (CH_2); 44.8 (CH_2); 47.2 (CH); 50.3 (CH_2); 61.1 (C); 64.5 (CH_2); 72.3 (CH); 79.2 (C); 124.9 (2 CH); 126.3 (CH); 128.1 (2 CH); 148.2 (C). IR (Nujol dispersion): 3380 (broad), 3030, 1595, 760, 725, 695 cm^{-1}. HRMS calcd for $C_{22}H_{36}NO_2$ $[M + H]^+$ 346.2741, found 346.2748.


Ph
N
Ph
O
O
4 ^{1}H NMR
3.607
5.537
9.128
1.790
3.520
5.531
1.907
13.678
7.769
8.968
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
.5
0.0
PPM


Ph
N
Ph
O
O
4 ^{13}C NMR DEPT
200
180
160
140
120
100
80
60
40
20
0
PPM

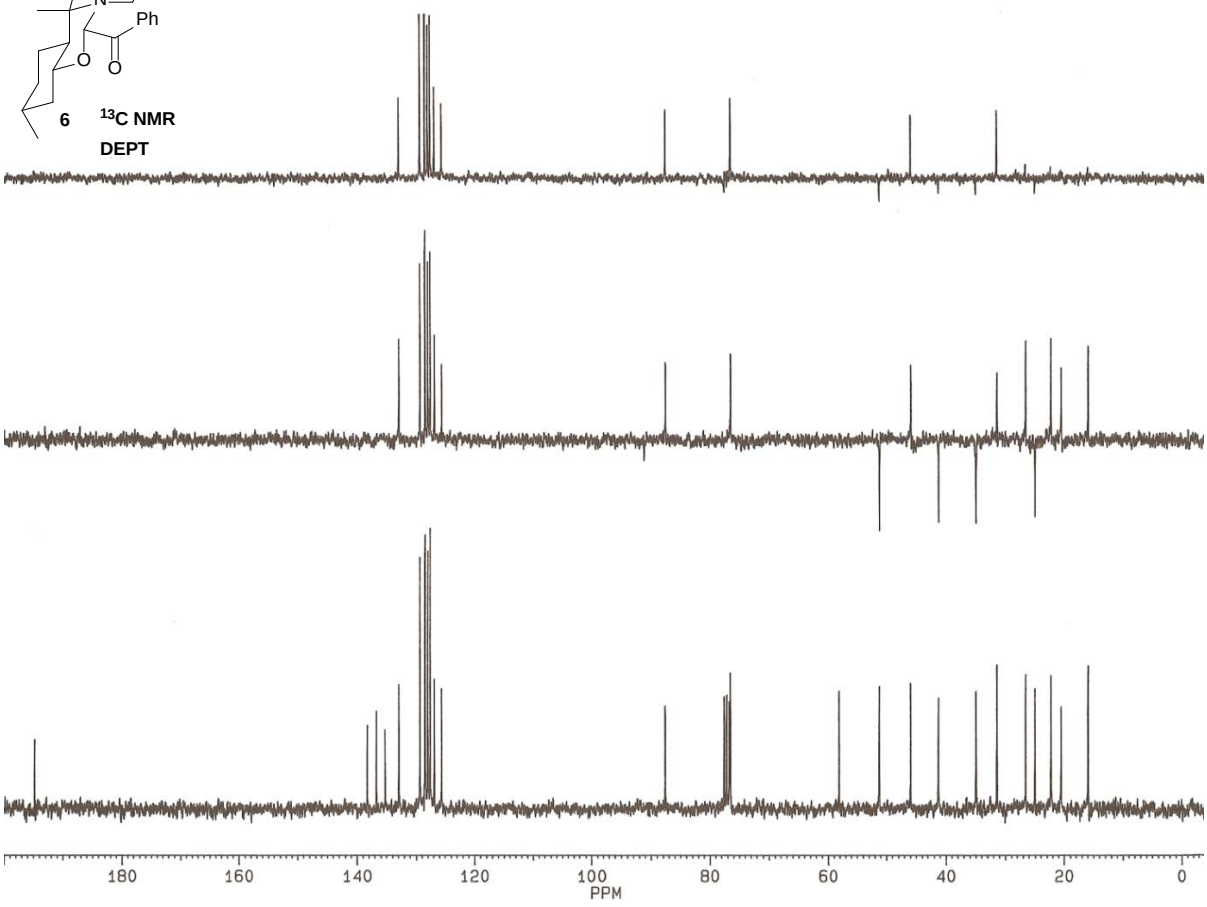
6 **^{1}H NMR**

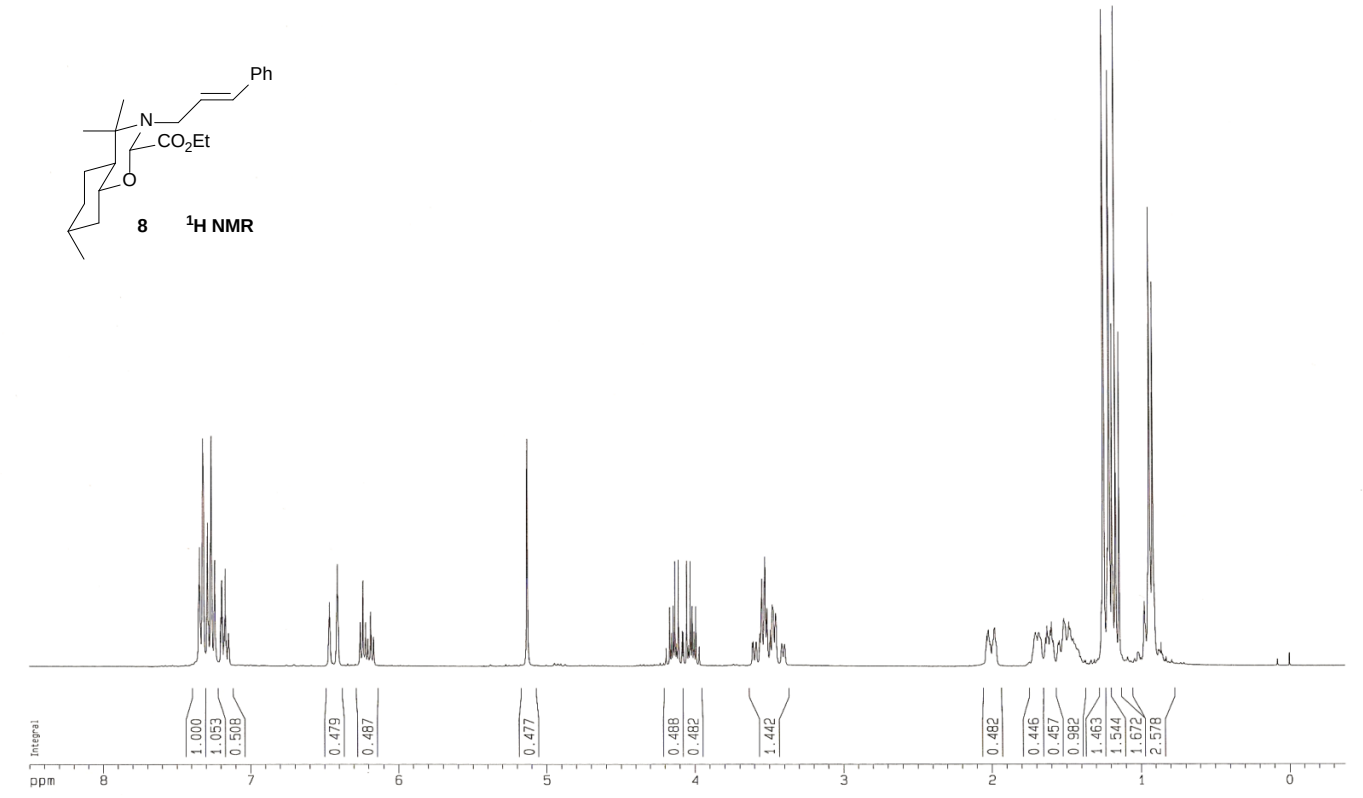


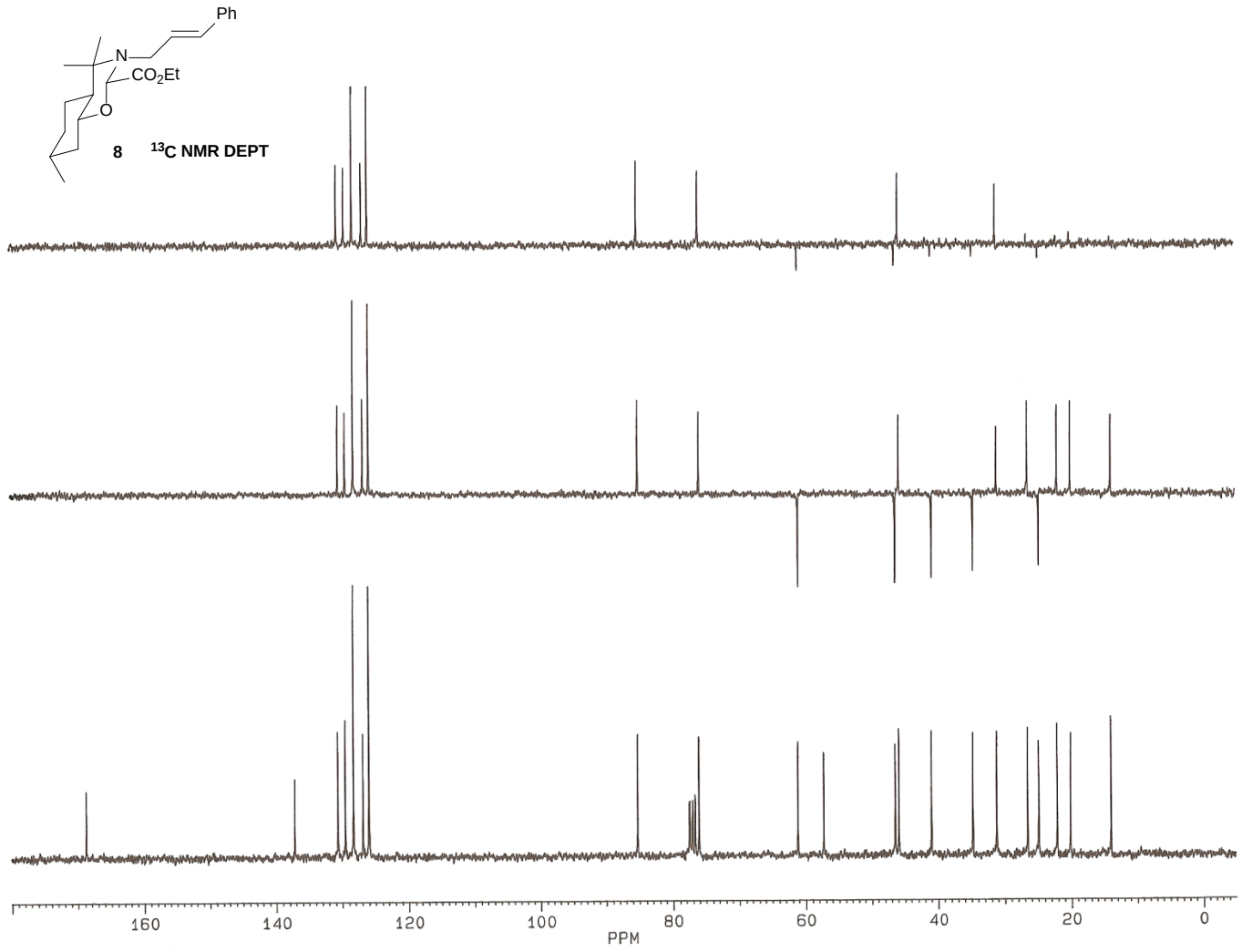


6 **^{13}C NMR**

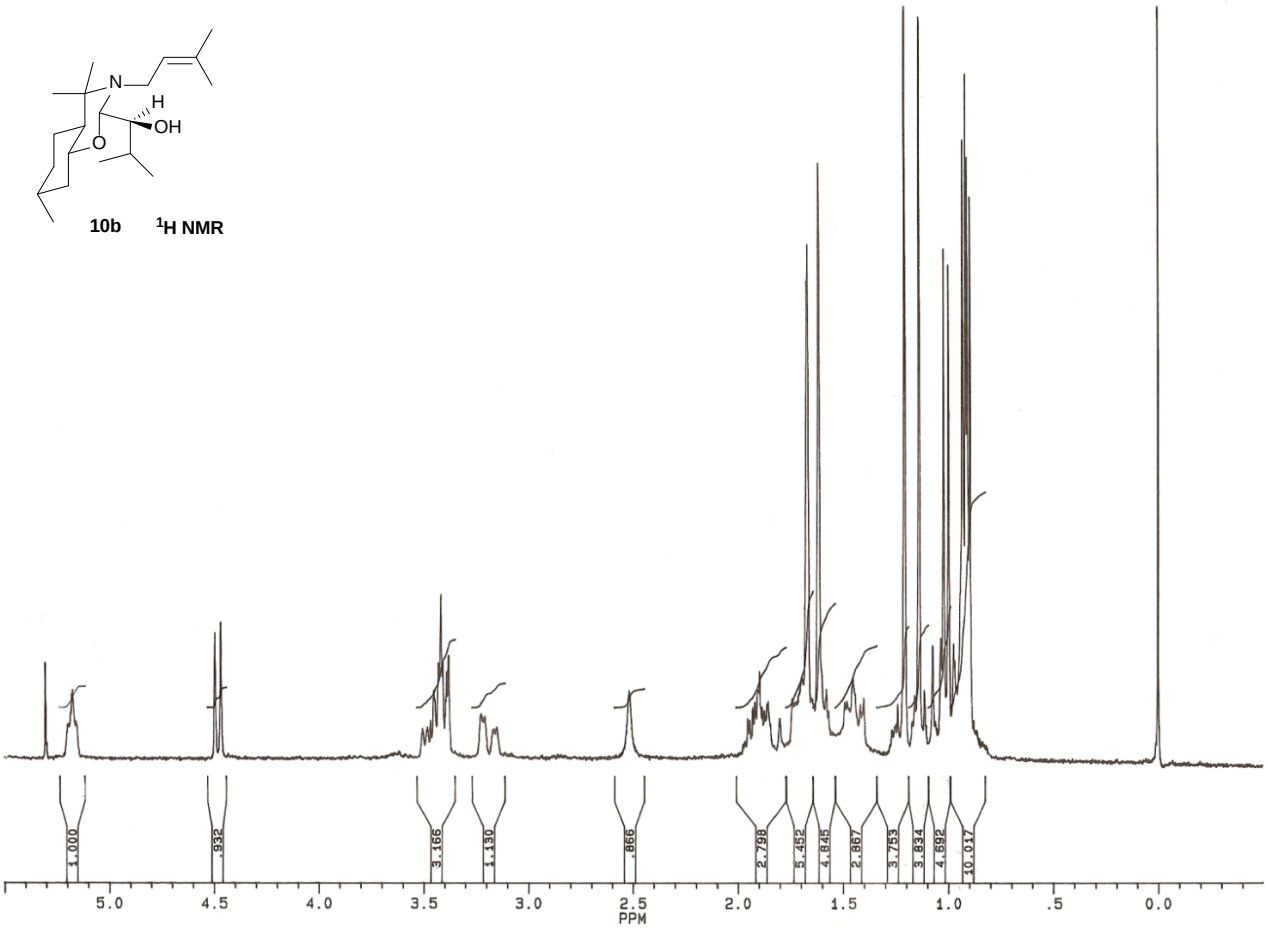
DEPT

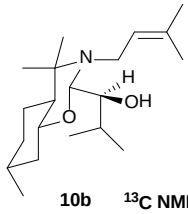



Ph
N
CO2Et
O
8 1H NMR
Integral
1.000
1.053
0.508
0.479
0.487
0.477
0.488
0.482
1.442
0.482
0.446
0.457
0.982
1.463
1.544
1.672
2.578
ppm
8
7
6
5
4
3
2
1
0

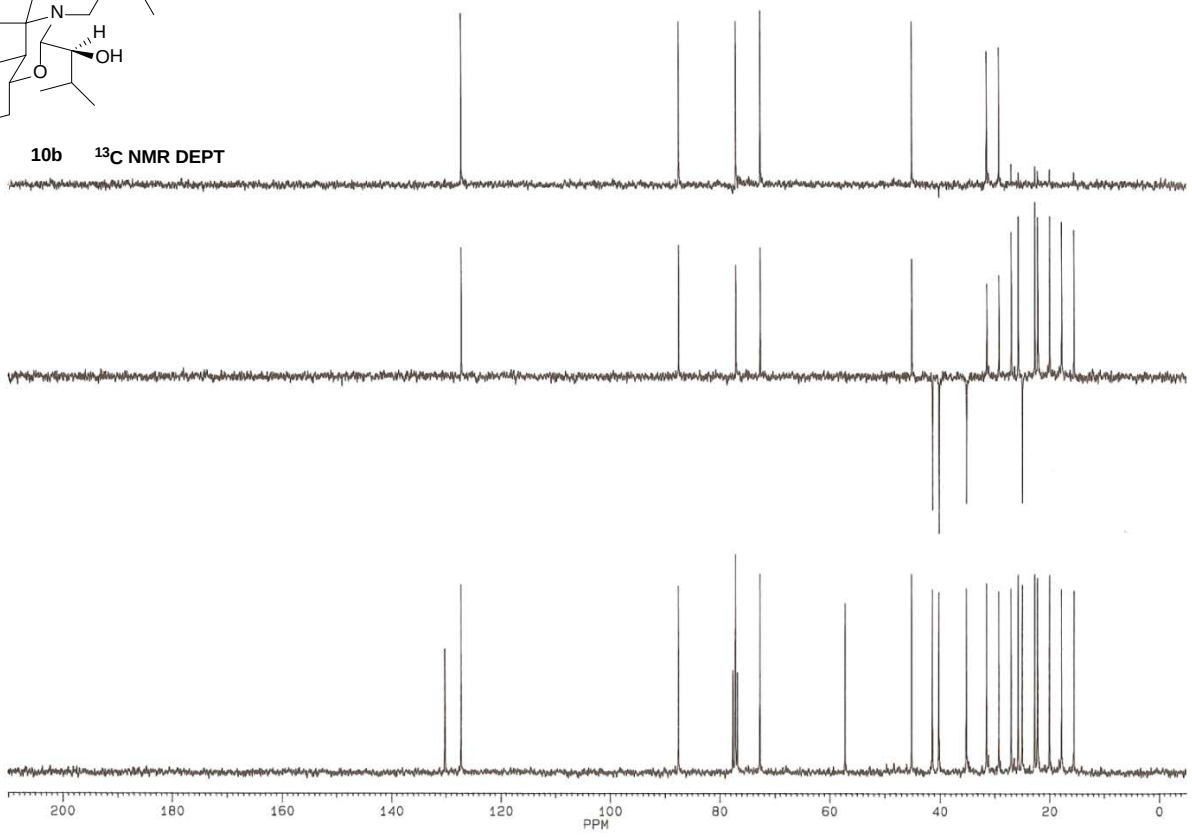

Ph
N
CO2Et
O
8 13C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM

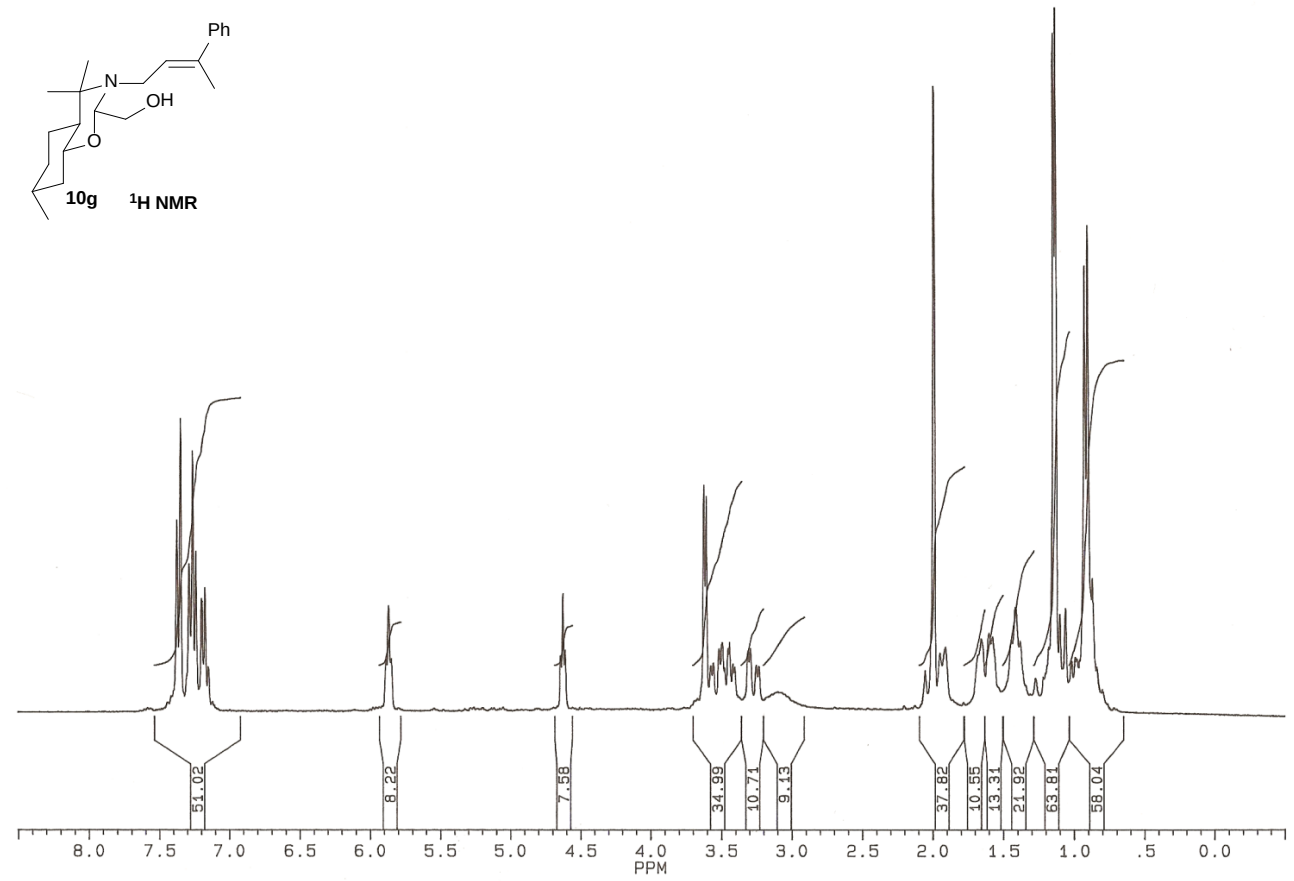
10b ^{1}**H NMR**

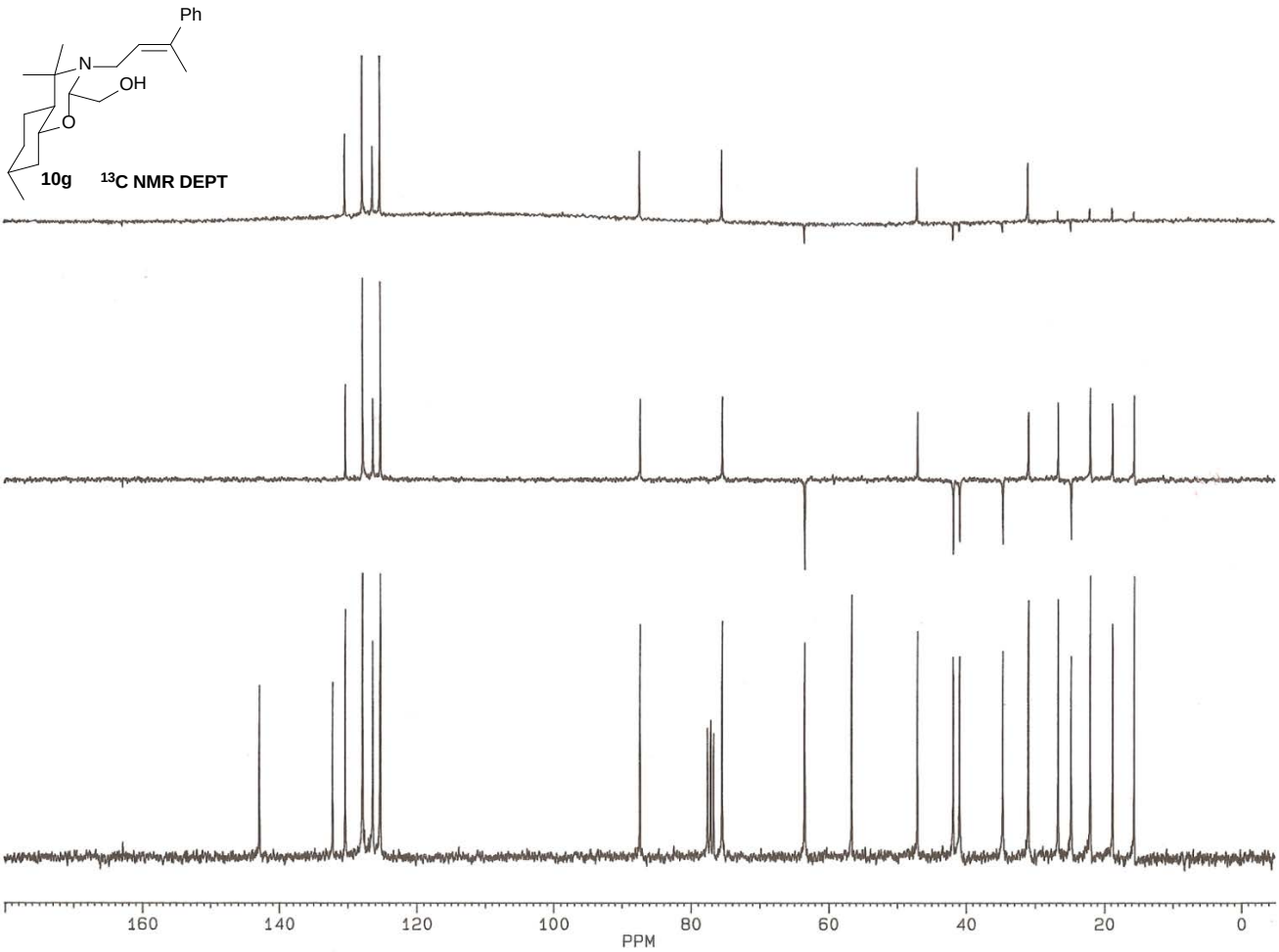


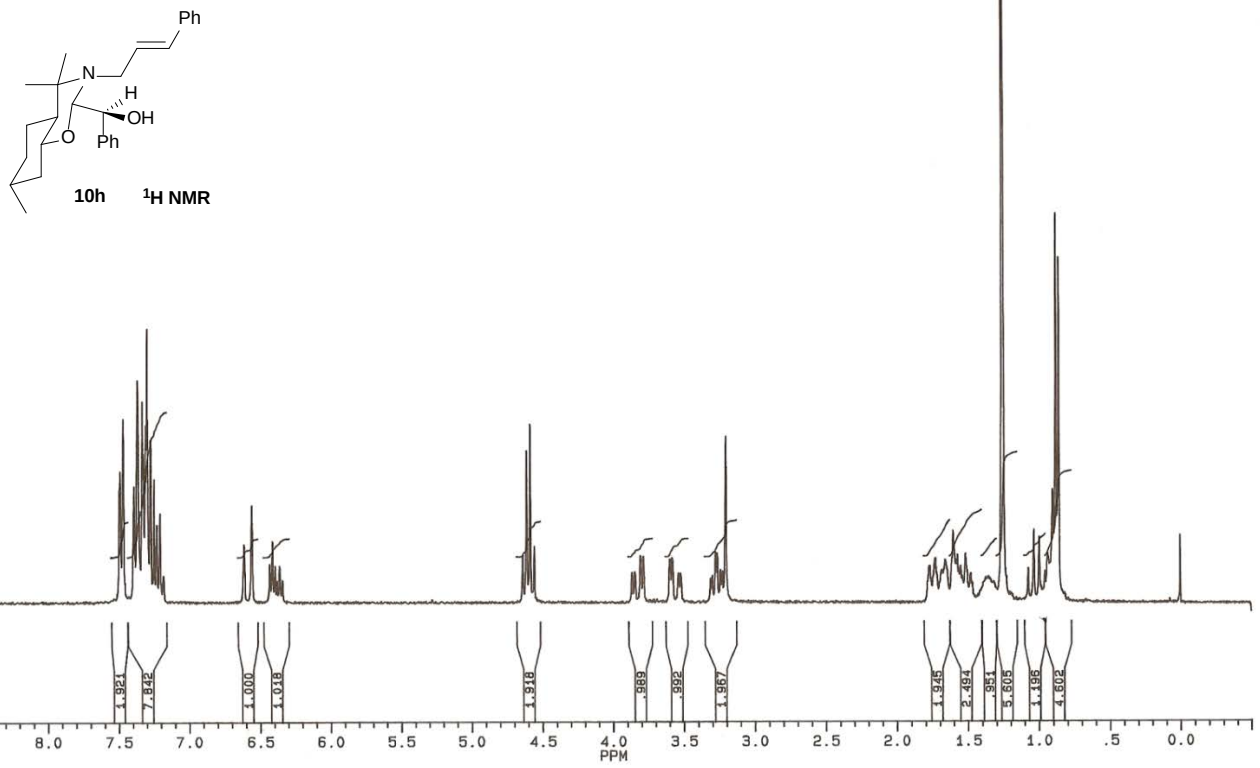


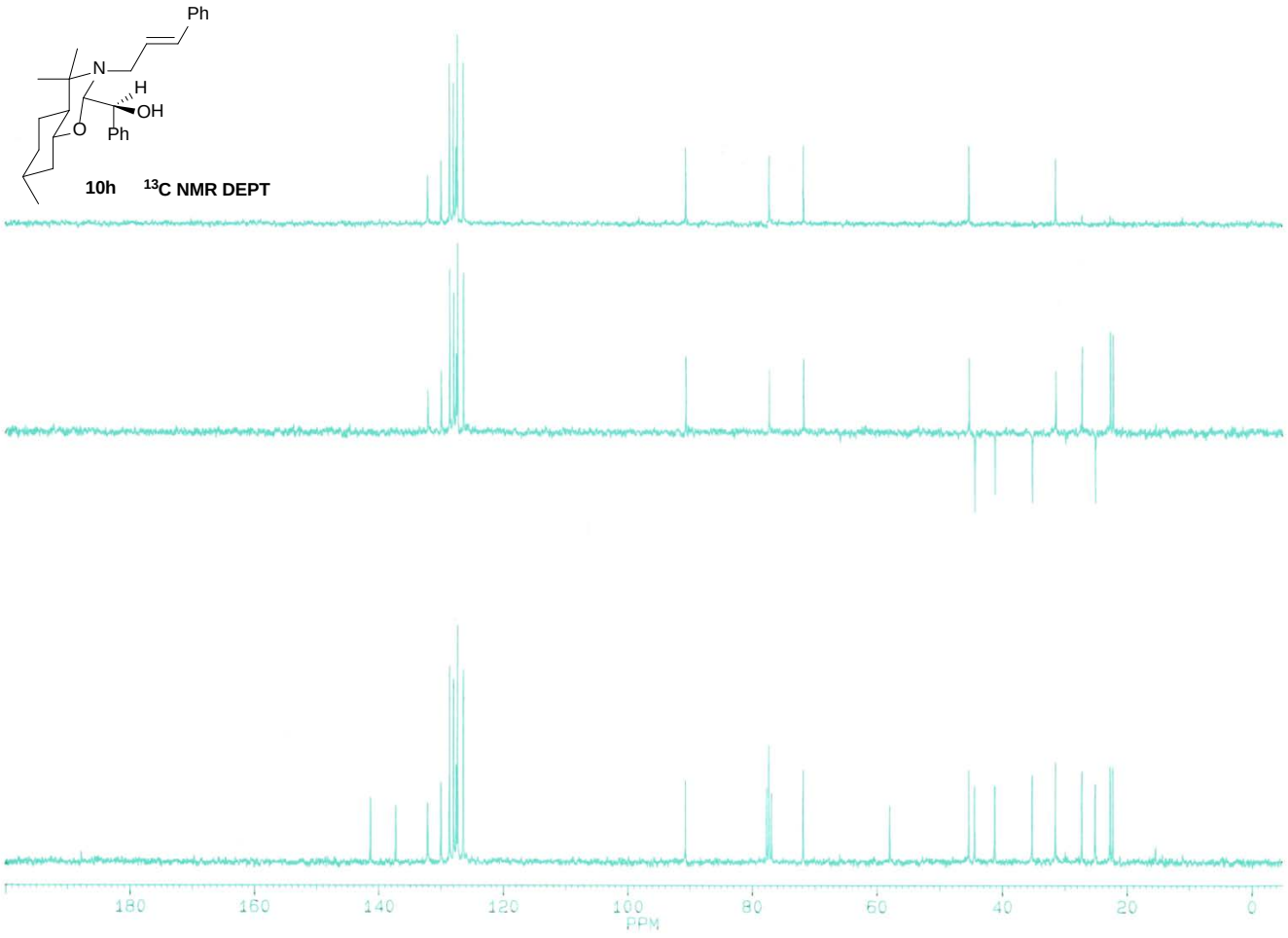
10b ^{13}**C NMR DEPT**

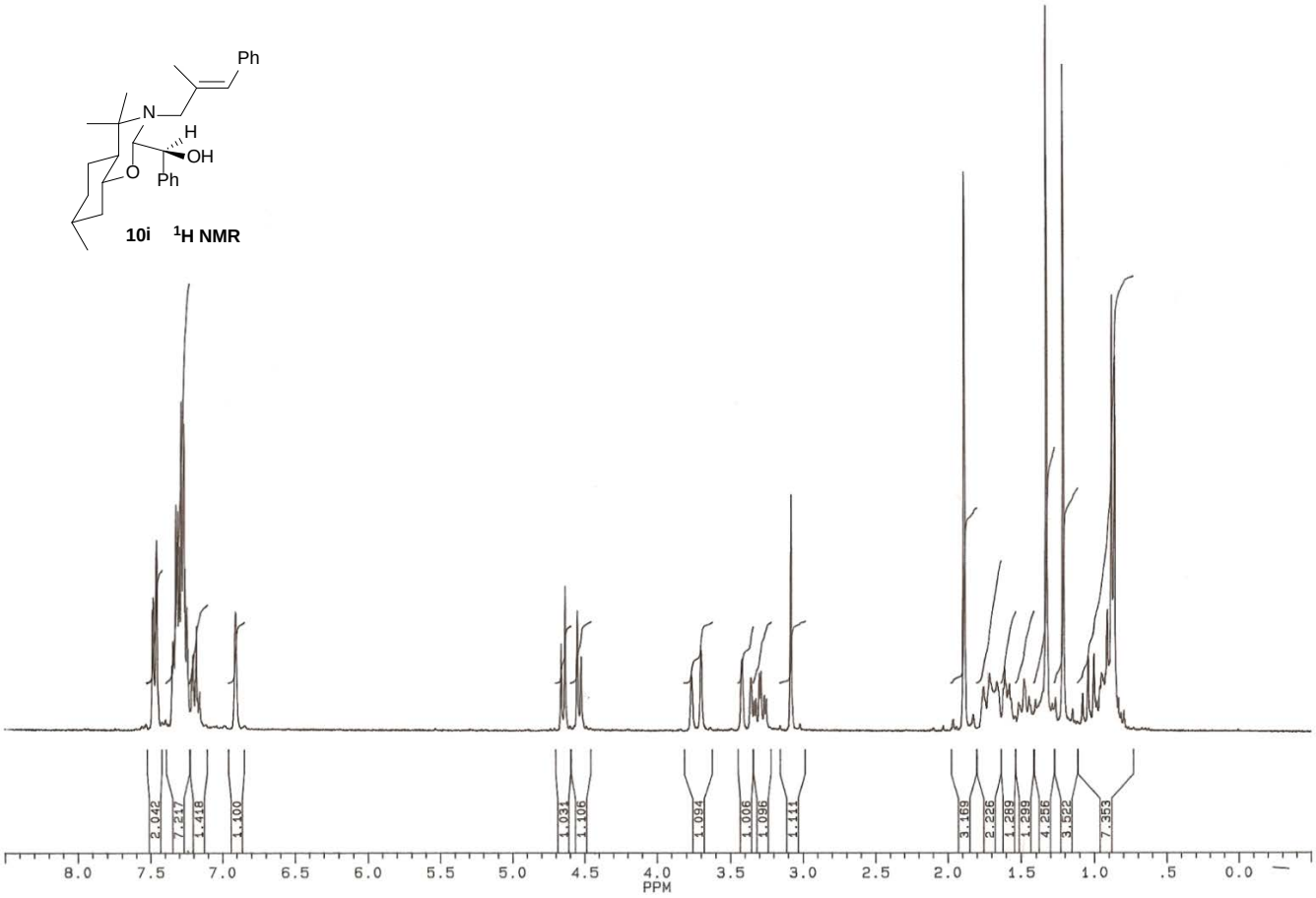


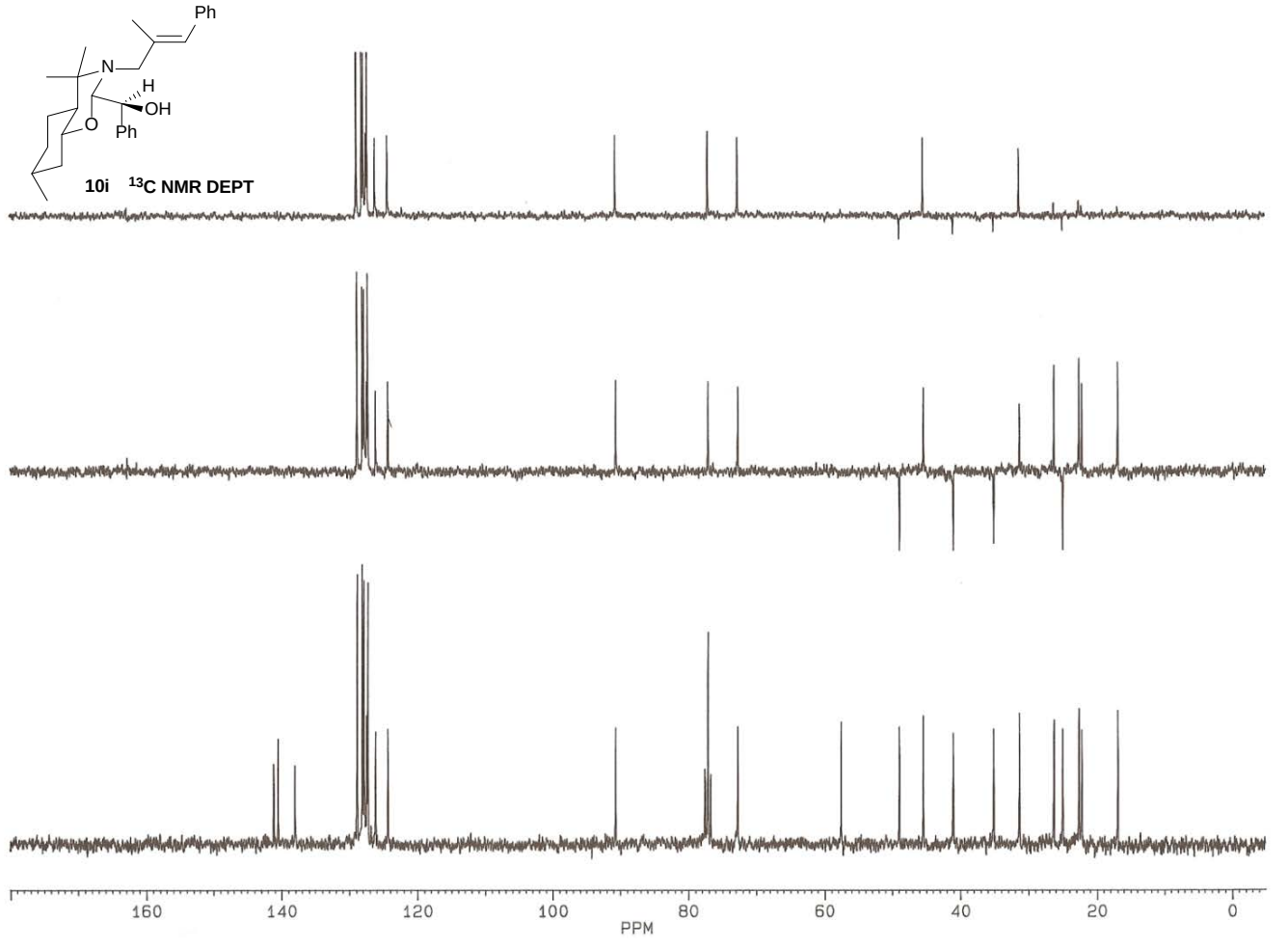

Ph
N
OH
O
10g
1H NMR
51.02
8.22
7.58
34.99
10.71
9.13
37.82
10.55
13.31
21.92
63.81
58.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM


Ph
N
OH
O
10g
13C NMR DEPT
160 140 120 100 80 60 40 20 0
PPM

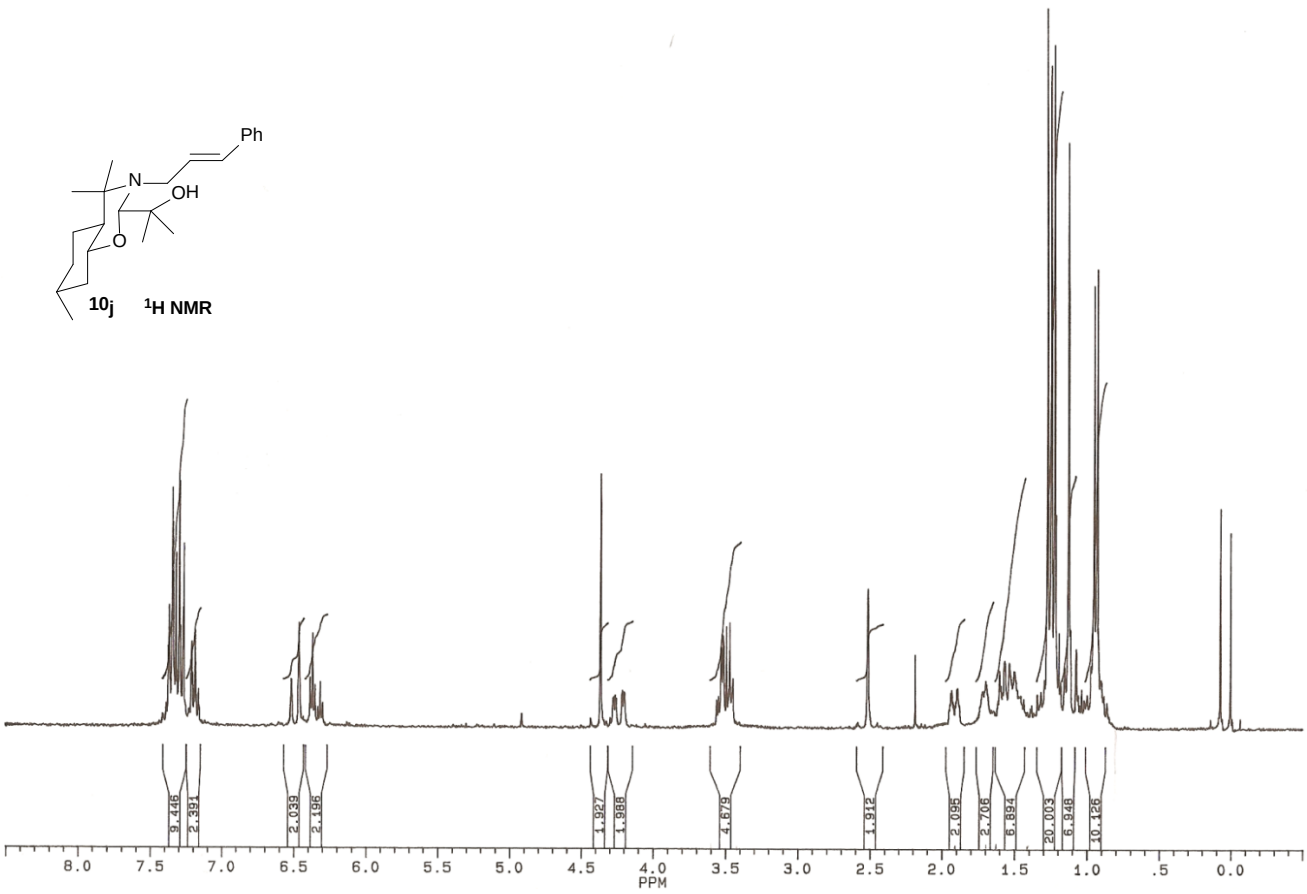

Ph
N
H
OH
O
Ph
10h
1H NMR
1.921
7.842
1.000
1.018
1.818
.989
.992
1.987
1.945
2.494
.951
5.805
1.196
4.602
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
0.0
PPM

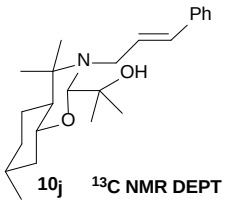

Ph
N
H
OH
O
Ph
10h
13C NMR DEPT
180
160
140
120
100
80
60
40
20
0
PPM


Ph
N
H
OH
O
Ph
10i ¹H NMR
2.042
7.217
1.418
1.100
1.031
1.106
1.094
1.006
1.096
1.111
3.169
2.226
1.289
1.299
4.256
3.522
7.353
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
.5
0.0
PPM

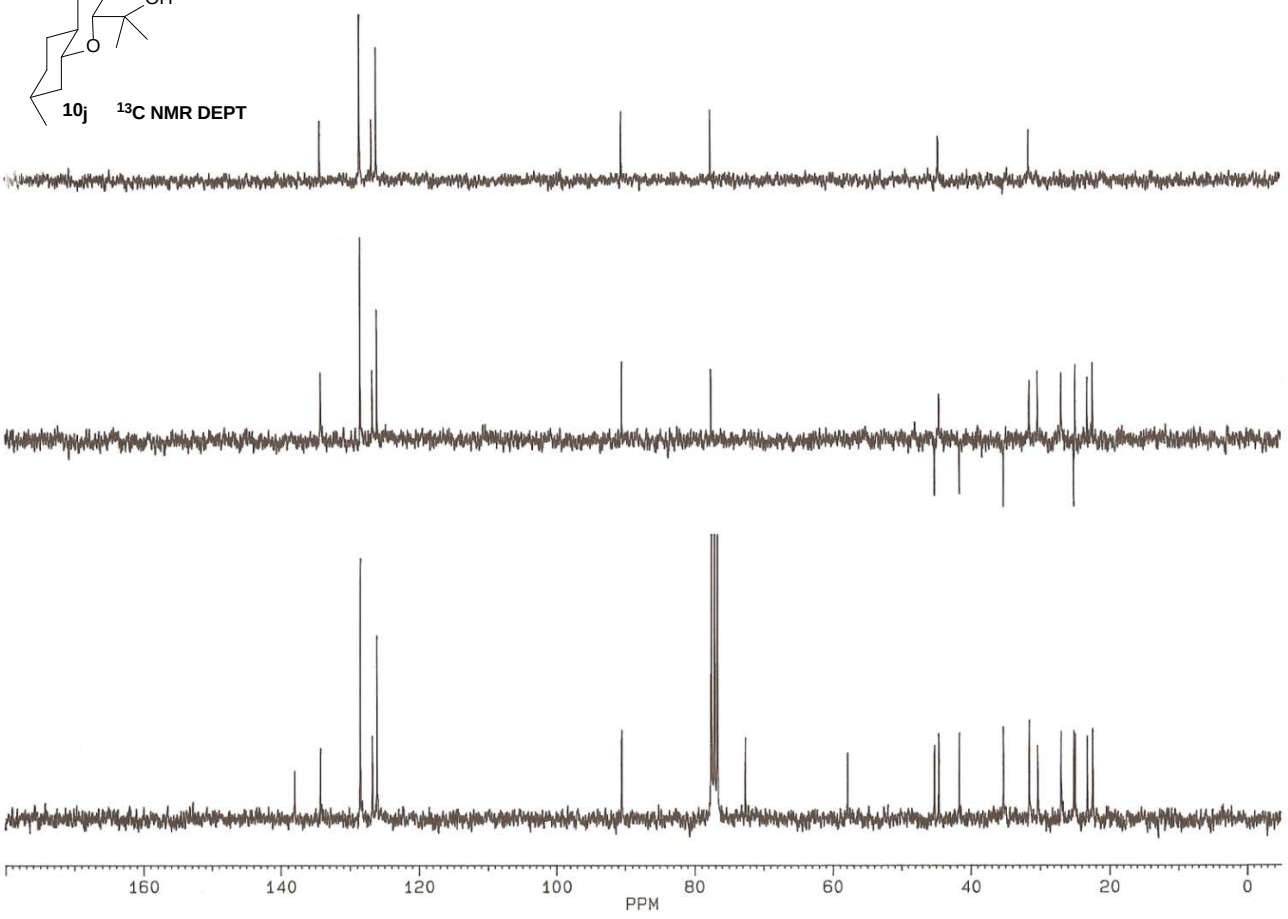

Ph
N
H
OH
O
Ph
10i ¹³C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM

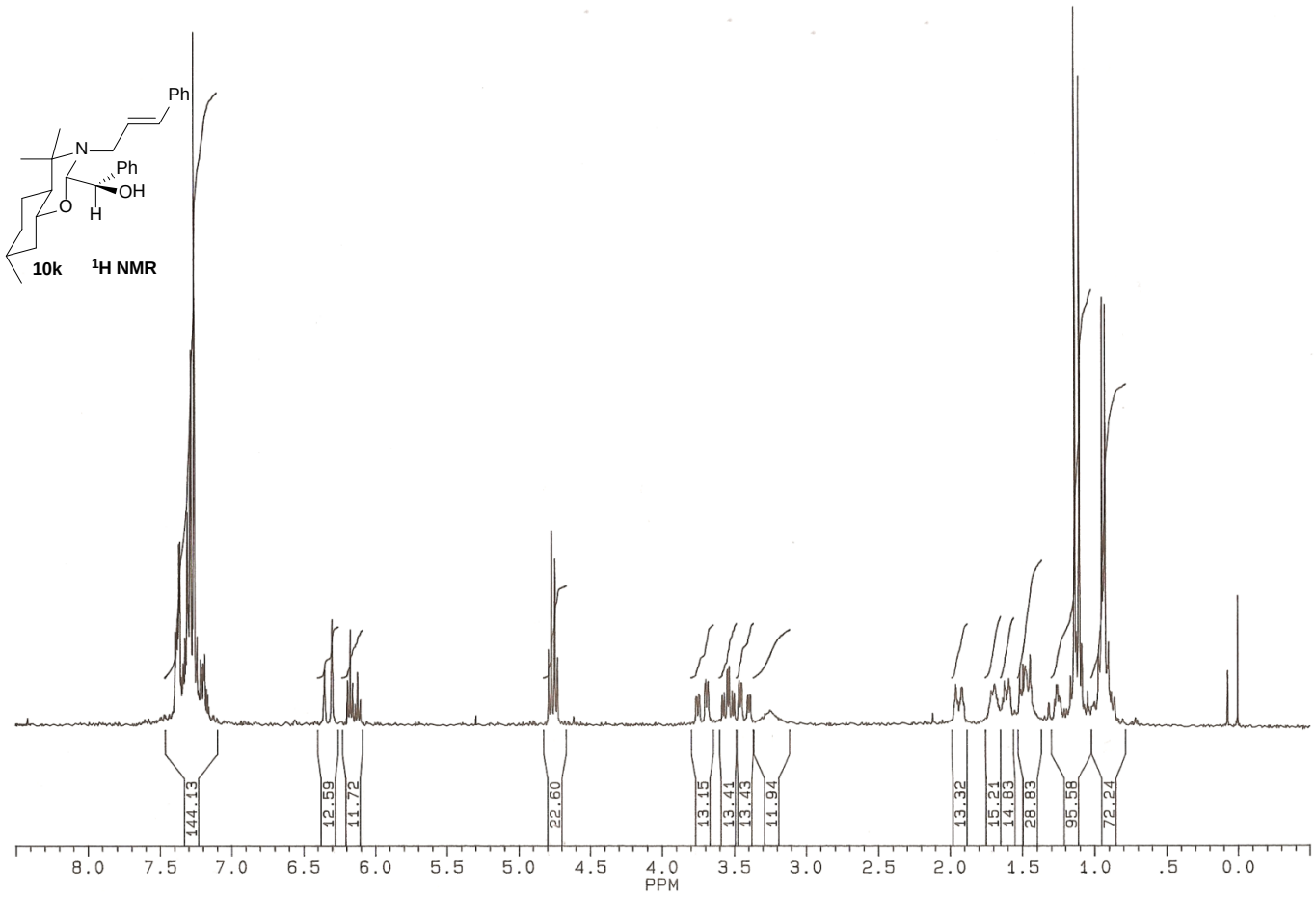
10j **^{1}H NMR**



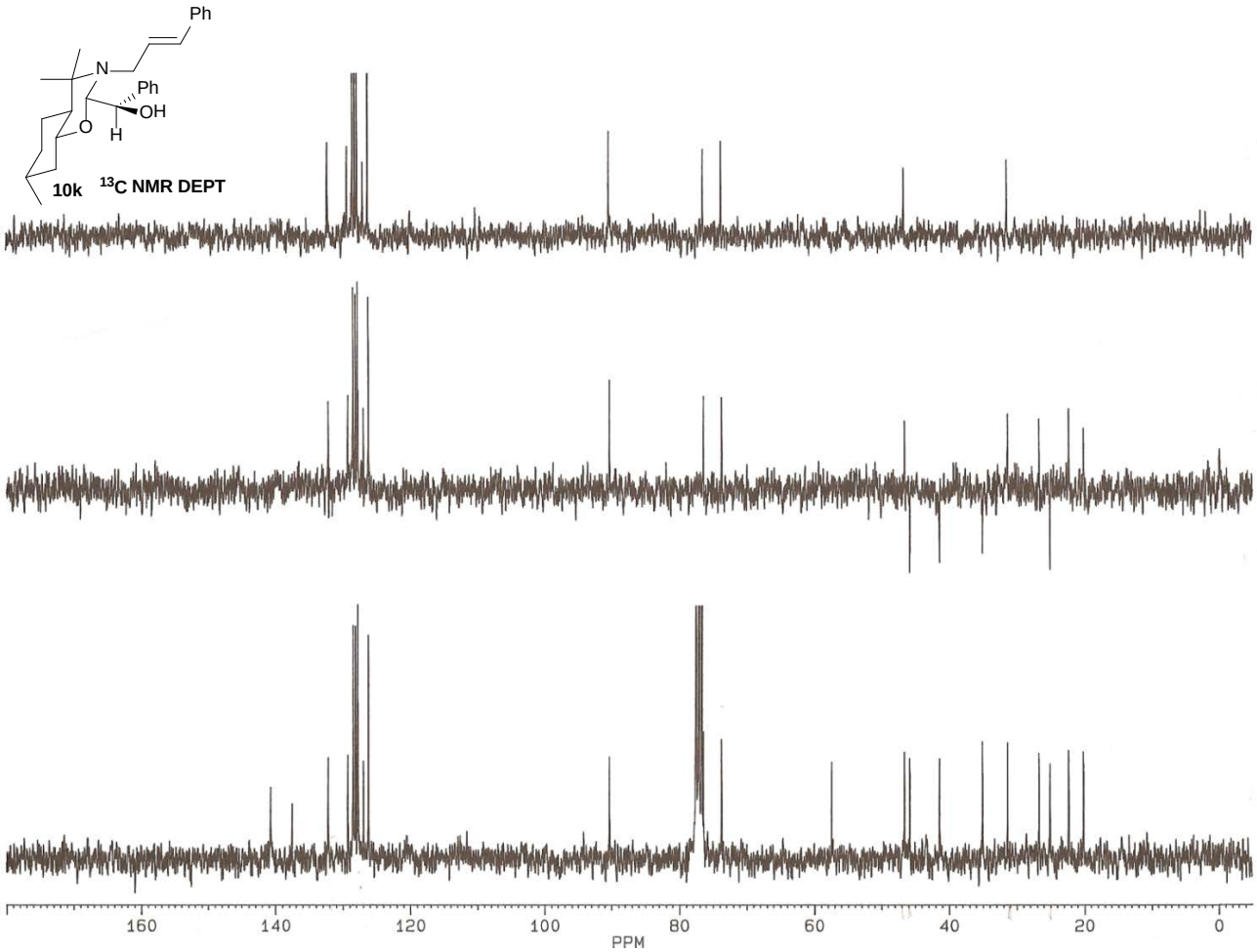


10j **^{13}C NMR DEPT**

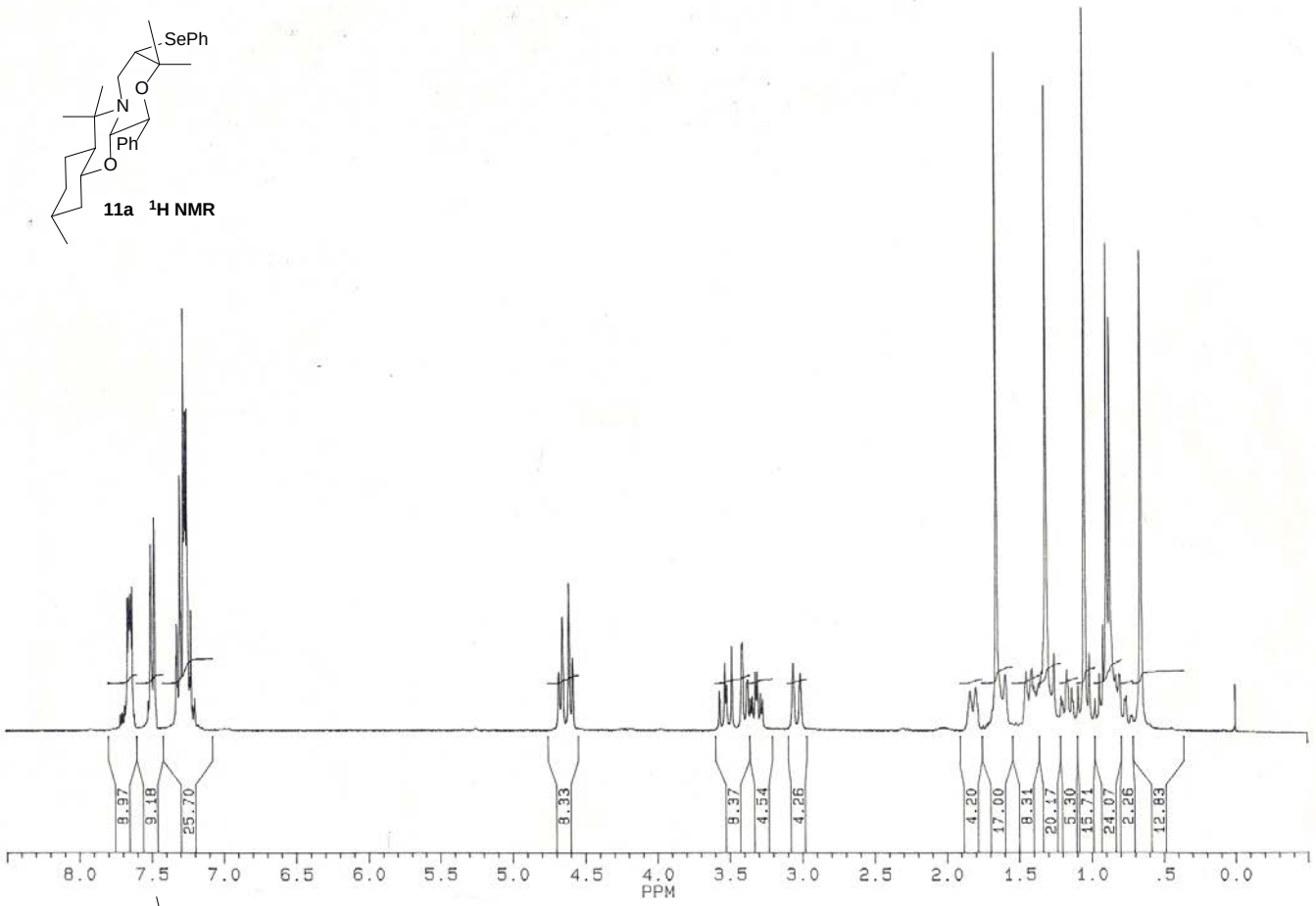


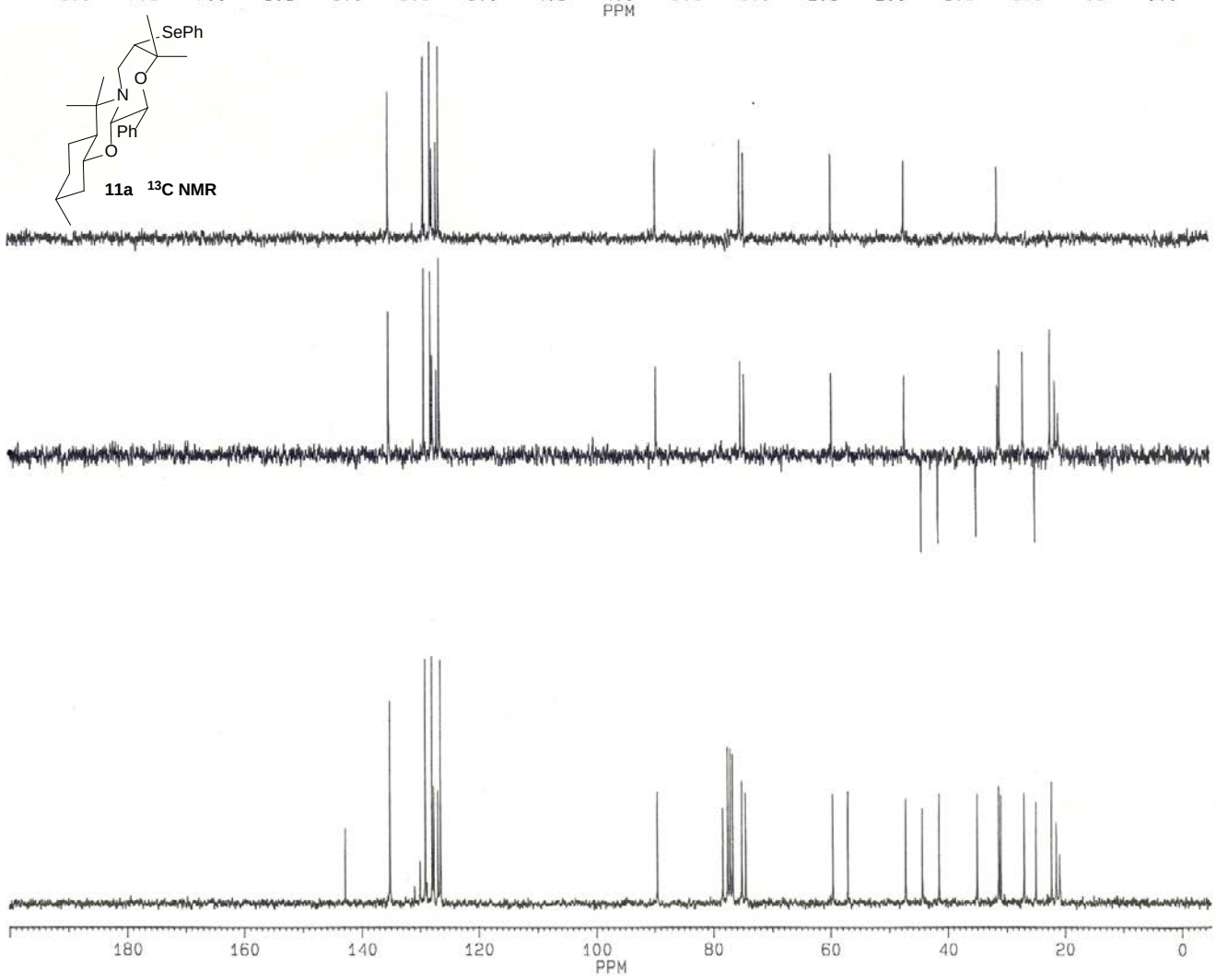


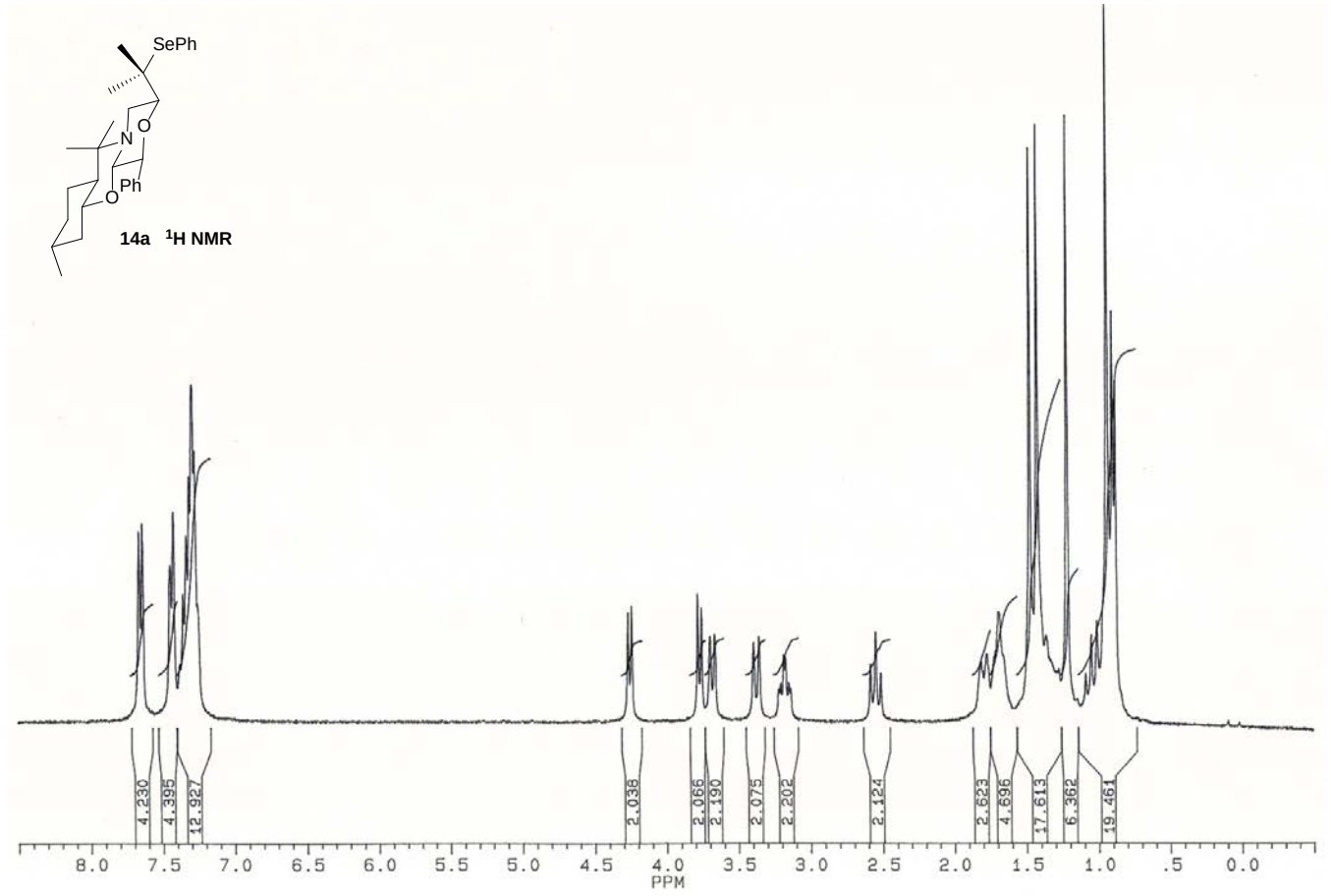
10k **^{1}H NMR**

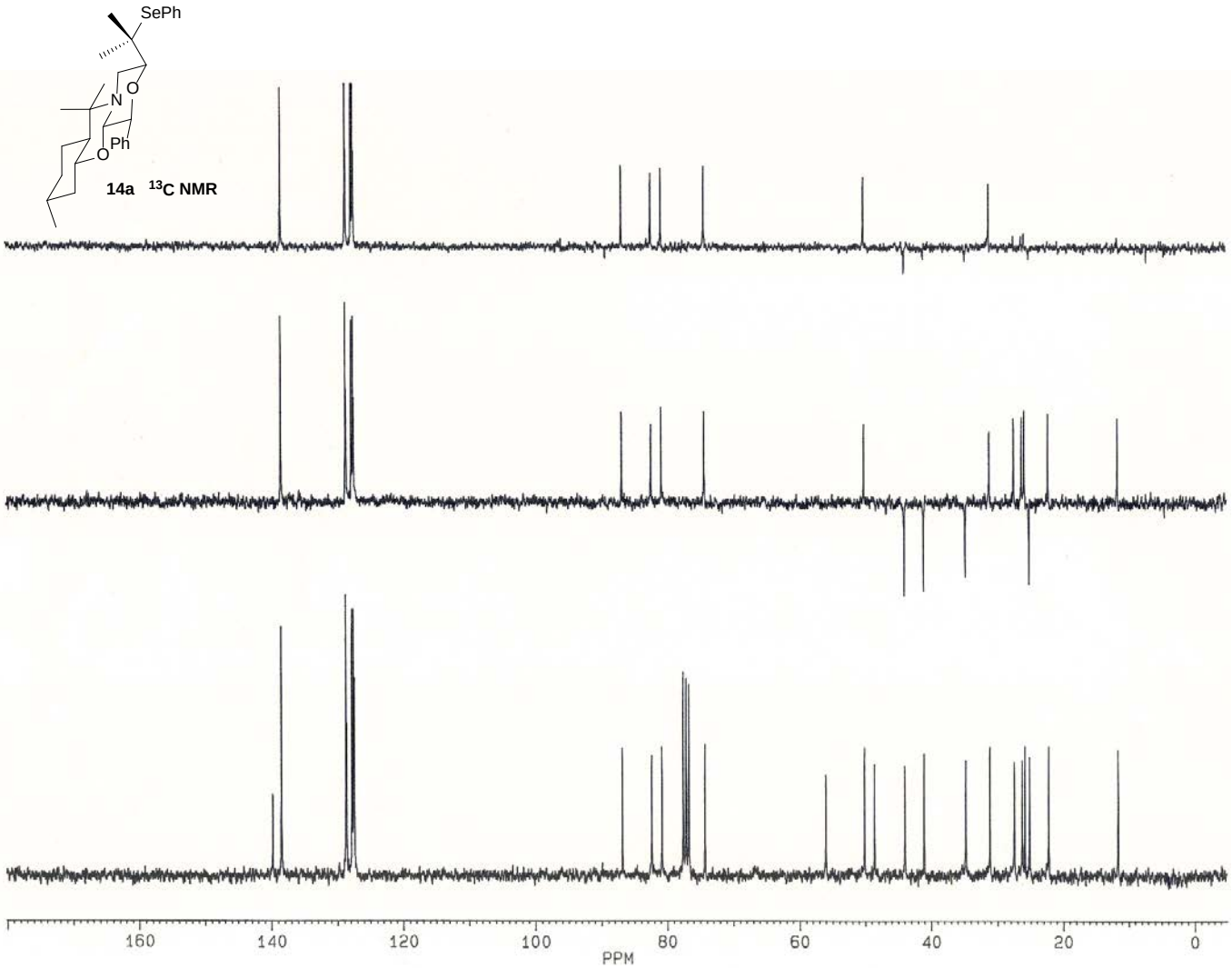


10k **^{13}C NMR DEPT**

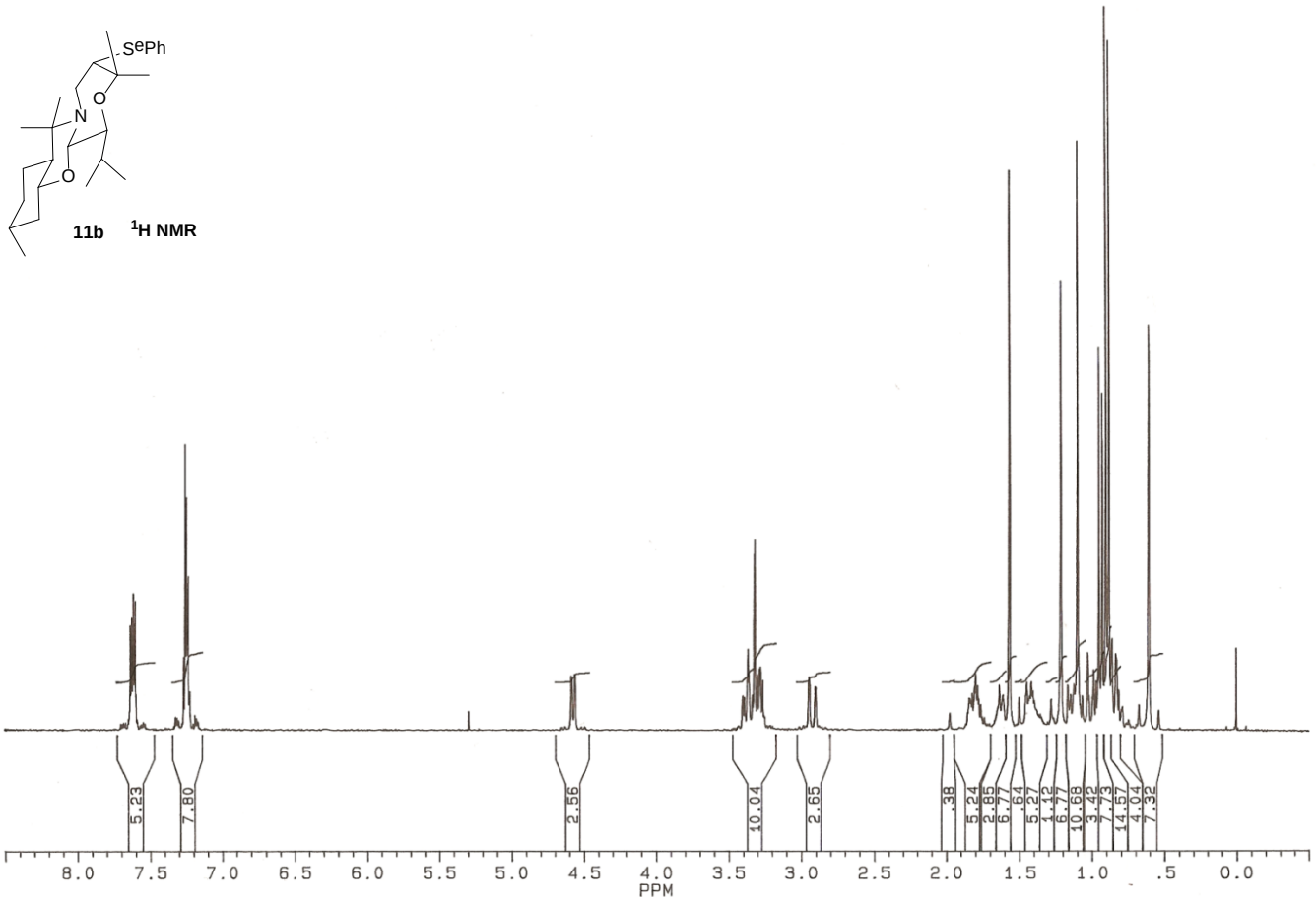

SePh
O
N
Ph
O
11a ^{1}H NMR
8.97
9.18
25.70
8.33
9.37
4.54
4.26
4.20
17.00
8.31
20.17
5.30
15.71
24.07
2.26
12.83
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM

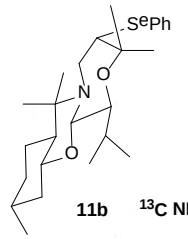

SePh
O
N
Ph
O
11a ^{13}C NMR
180 160 140 120 100 80 60 40 20 0
PPM


SePh
N
O
Ph
O
14a ^{1}H NMR
4.230
4.395
12.927
2.038
2.066
2.190
2.075
2.202
2.124
2.623
4.696
17.613
6.362
19.461
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
.5
0.0
PPM

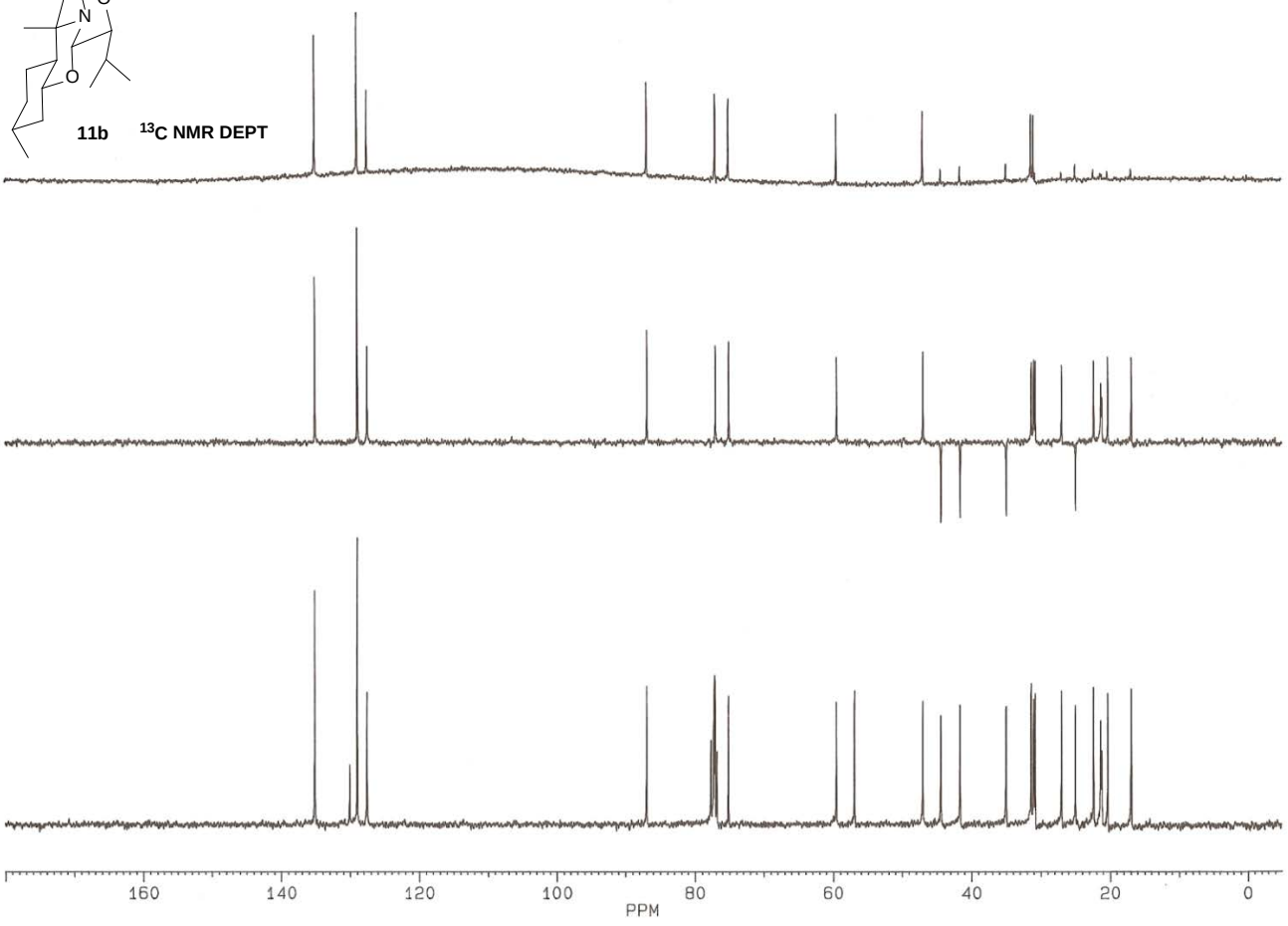

SePh
N
O
Ph
O
14a ^{13}C NMR
160
140
120
100
80
60
40
20
0
PPM

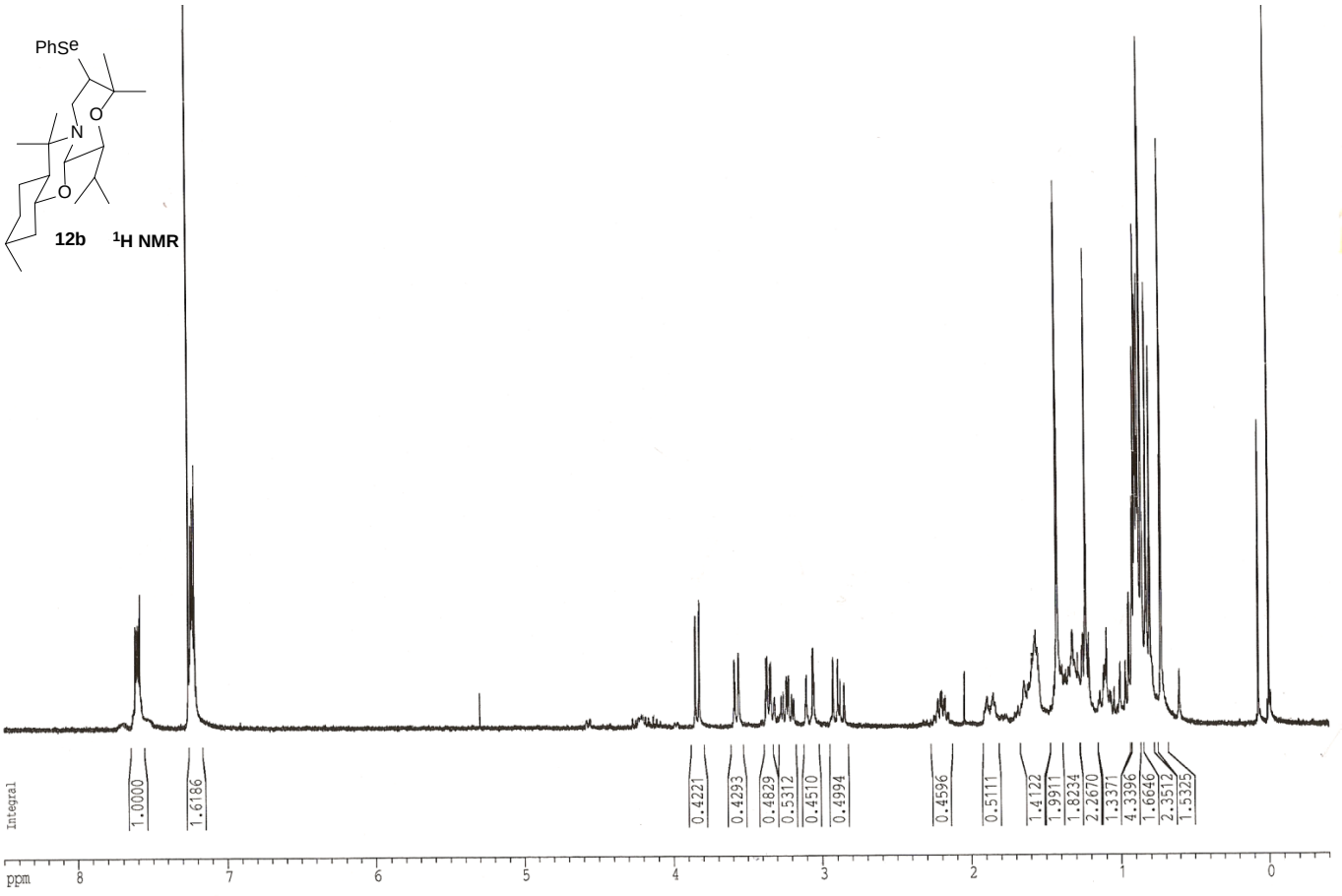
11b **^{1}H NMR**

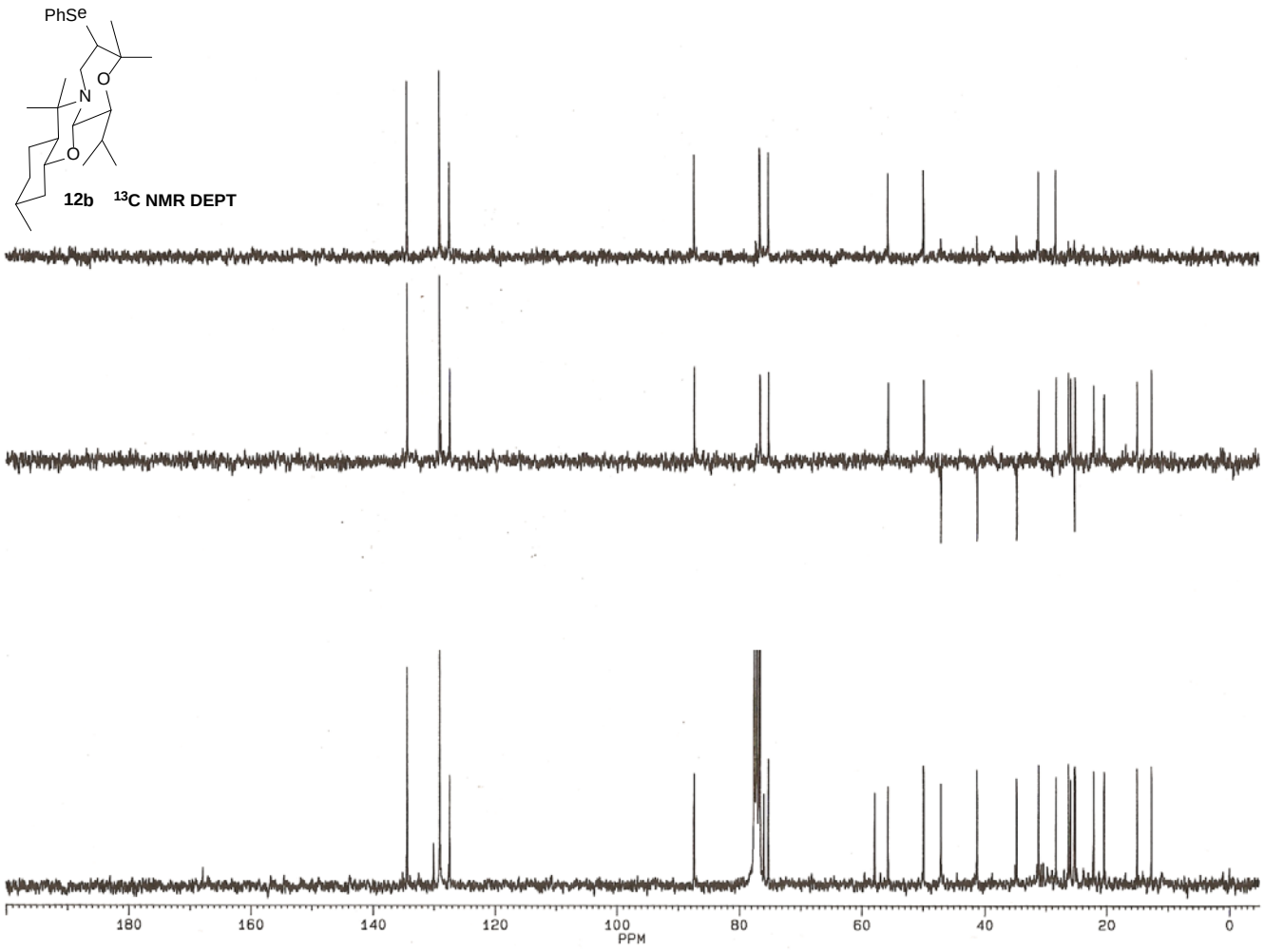


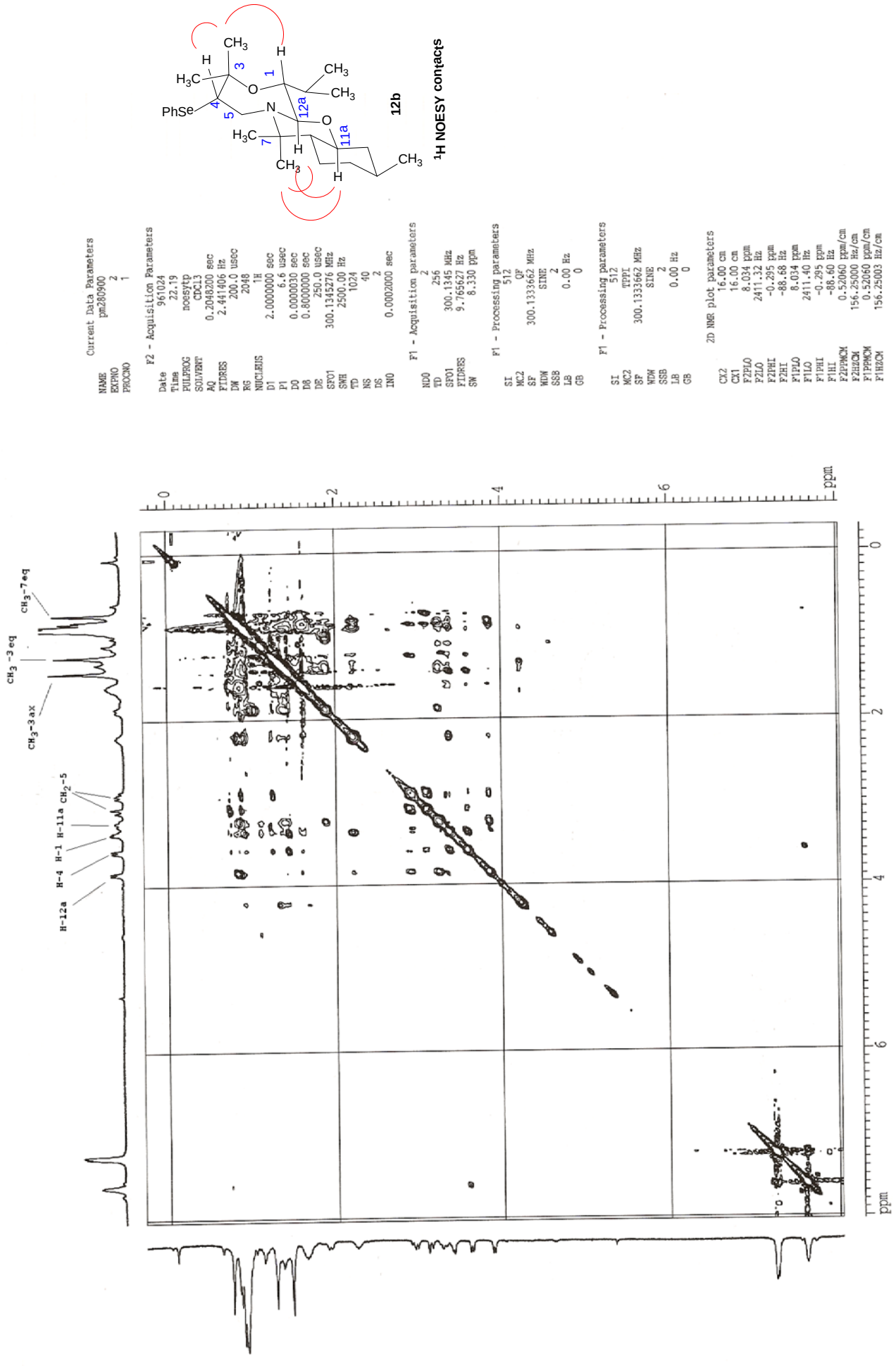


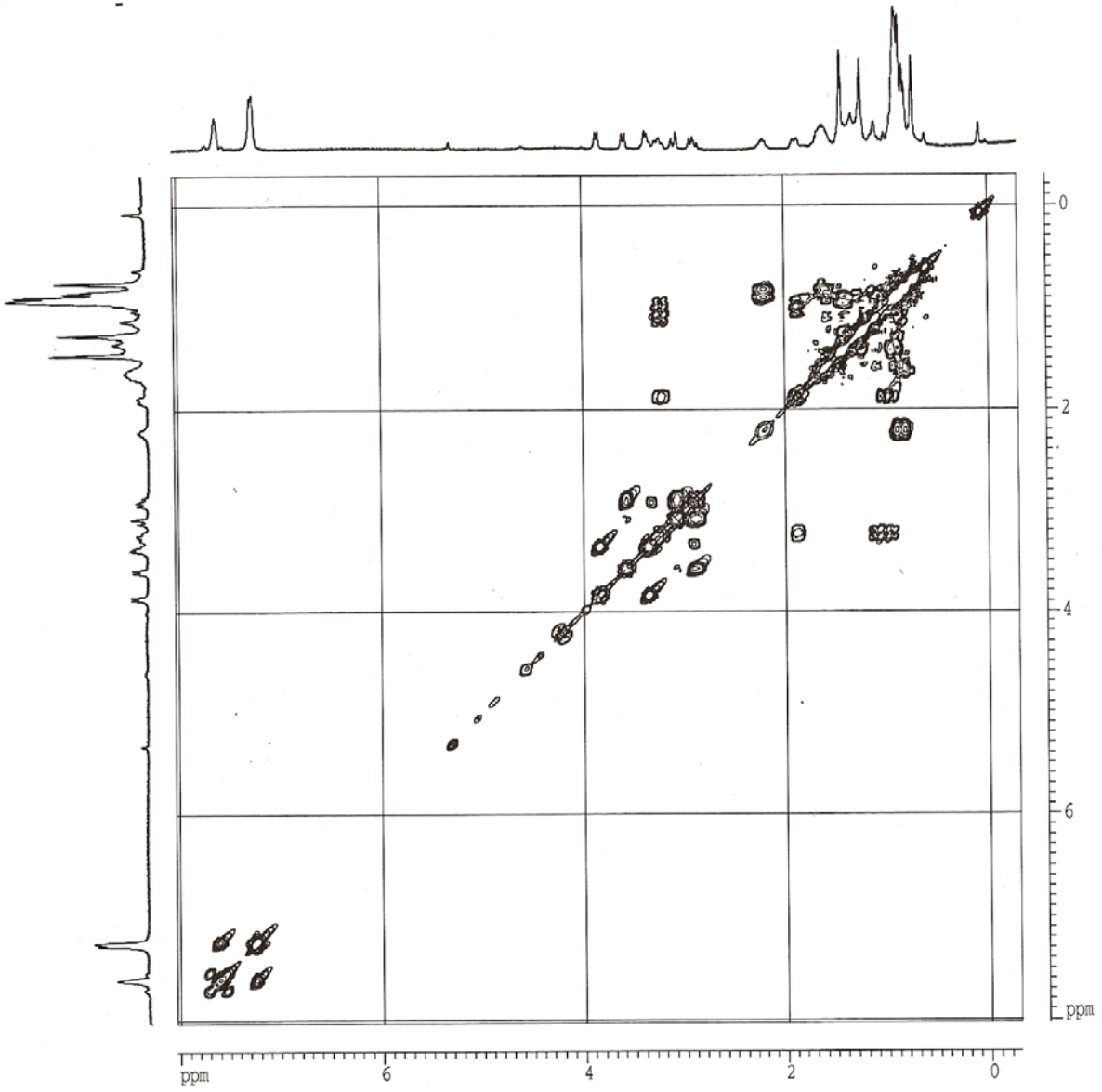
11b **^{13}C NMR DEPT**




PhSe
O
N
O
12b ¹H NMR
Integral
1.0000
1.6186
0.4221
0.4293
0.4829
0.5312
0.4510
0.4994
0.4596
0.5111
1.4122
1.9911
1.8234
2.2670
1.3371
4.3396
1.6646
2.3512
1.5325
ppm
8
7
6
5
4
3
2
1
0


PhSe
O
N
O
12b ¹³C NMR DEPT
180
160
140
120
100
80
60
40
20
0
PPM


12b
^{1}H NOESY contacts
Current Data Parameters
NAME pm280900
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date 961024
Time 22.19
PULPROG noesytp
SOLVENT CDCl3
AQ 0.2048200 sec
FIDRES 2.441406 Hz
DW 200.0 usec
RG 2048
NUCLEUS 1H
D1 2.0000000 sec
P1 6.6 usec
DO 0.0000030 sec
D8 0.8000000 sec
D8 250.0 usec
SFO1 300.1345276 MHz
SWH 2500.00 Hz
TD 1024
NS 40
DS 2
IN0 0.0002000 sec
F1 - Acquisition parameters
NDO 2
TD 256
SFO1 300.1345 MHz
FIDRES 9.765627 Hz
SW 8.330 ppm
F1 - Processing parameters
SI 512
WC2 QF
SF 300.1333662 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0
F1 - Processing parameters
SI 512
WC2 TPPI
SF 300.1333662 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 16.00 cm
CX1 16.00 cm
F2PLO 8.034 ppm
F2LO 2411.32 Hz
F2PHI -0.295 ppm
F2HI -88.68 Hz
F1PLO 8.034 ppm
F1LO 2411.40 Hz
F1PHI -0.295 ppm
F1HI -88.60 Hz
F2PPMCM 0.52060 ppm/cm
F2HZCM 156.25000 Hz/cm
F1PPMCM 0.52060 ppm/cm
F1HZCM 156.25003 Hz/cm
CH3-7eq
CH3-3eq
CH3-3ax
H-12a H-4 H-1 H-11a CH2-5
ppm
ppm



Current Data Parameters

NAME	pm280900
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date	961025
Time	6.56
PULPROG	cosy90
SOLVENT	CDCl3
AQ	0.1024200 sec
FIDRES	4.882813 Hz
DW	200.0 usec
RG	2048
NUCLEUS	1H
D1	1.0000000 sec
P1	6.6 usec
D0	0.0000030 sec
DE	250.0 usec
SFO1	300.1345276 MHz
SWH	2500.00 Hz
TD	512
NS	40
DS	2
IN0	0.0004000 sec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.1345 MHz
FIDRES	9.765627 Hz
SW	8.330 ppm

F1 - Processing parameters

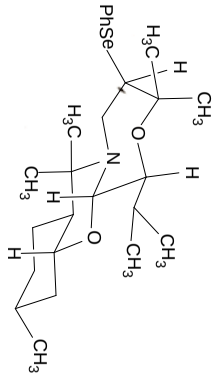
SI	512
MC2	QF
SF	300.1333662 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

F1 - Processing parameters

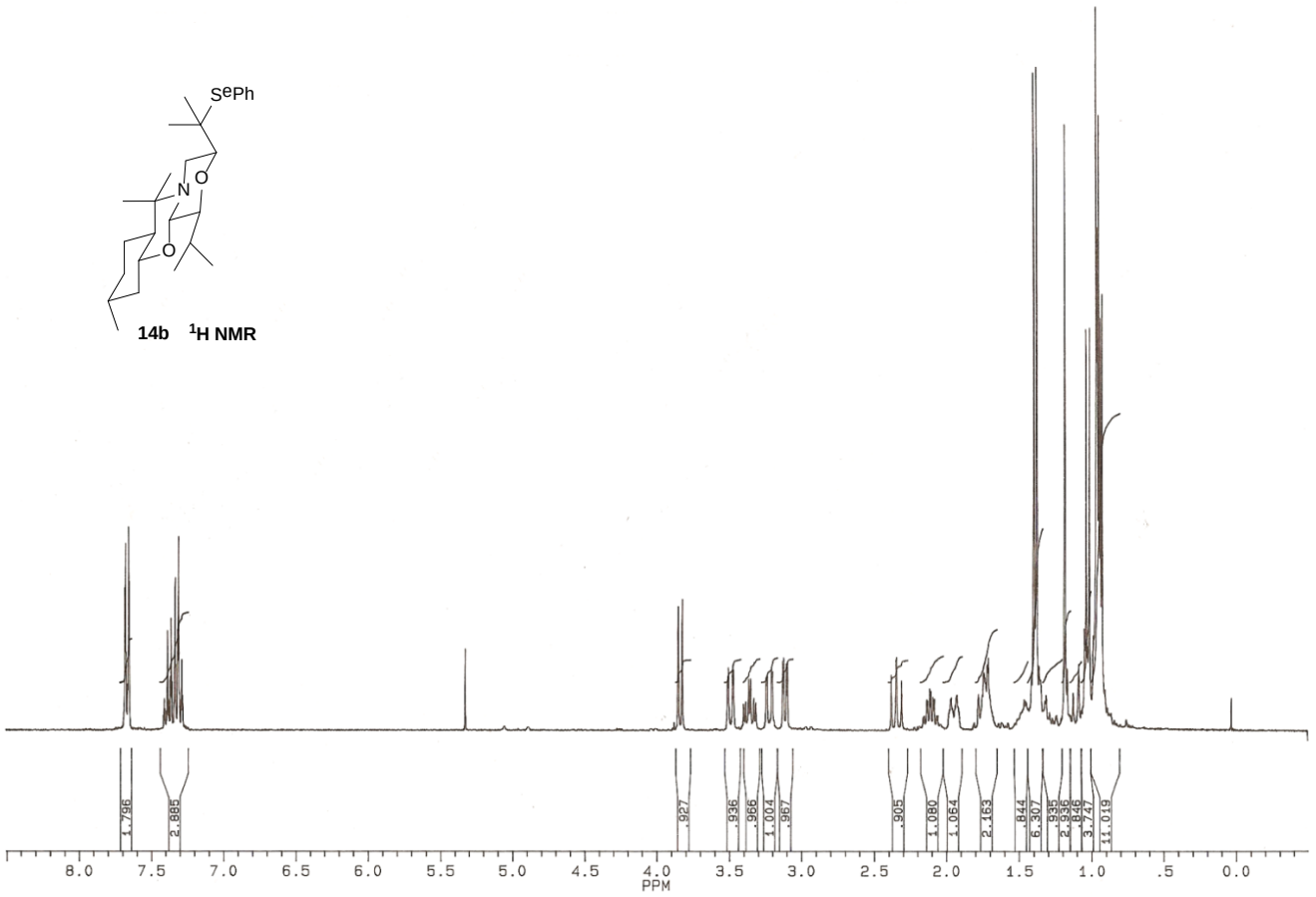
SI	512
MC2	QF
SF	300.1333662 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

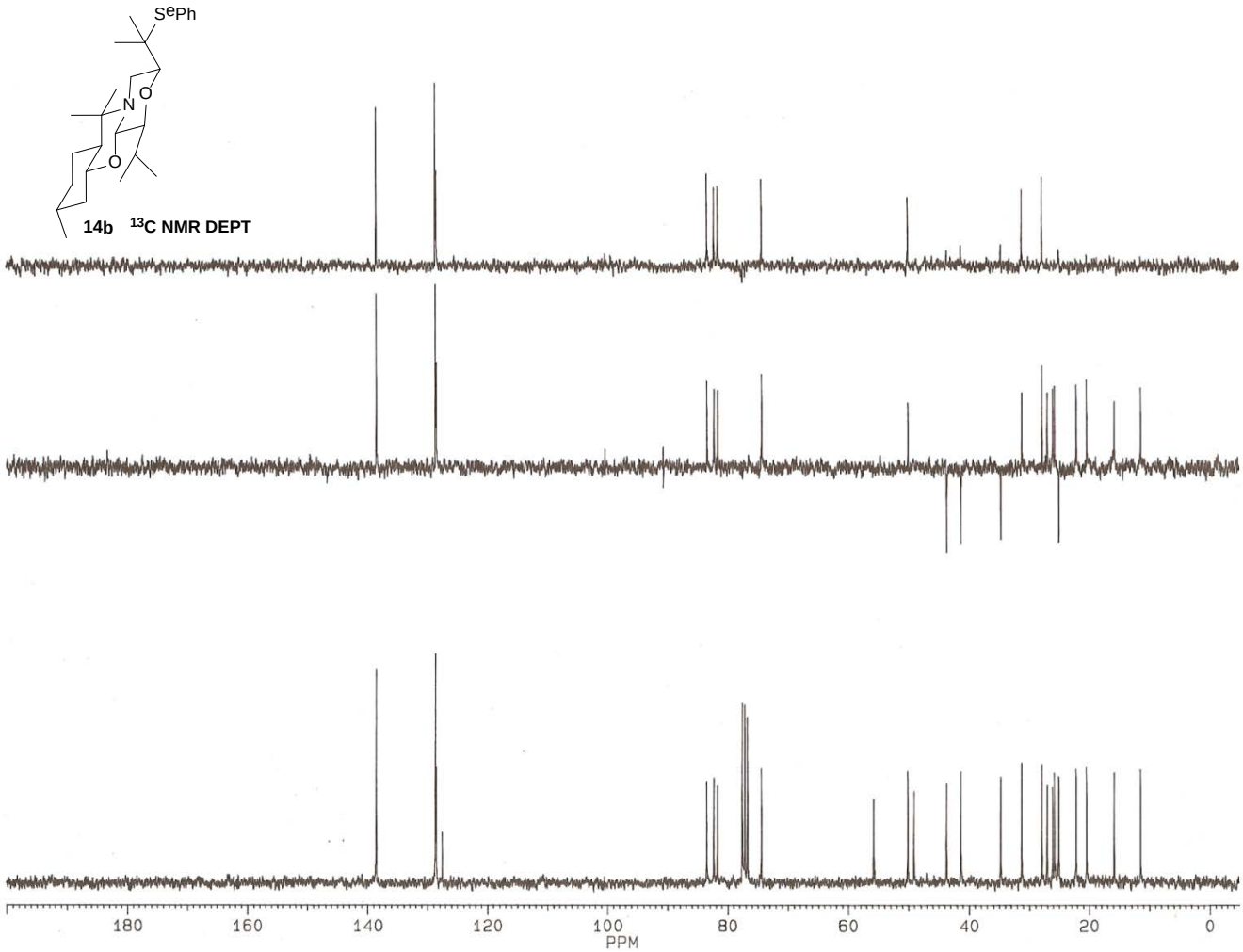
2D NMR plot parameters

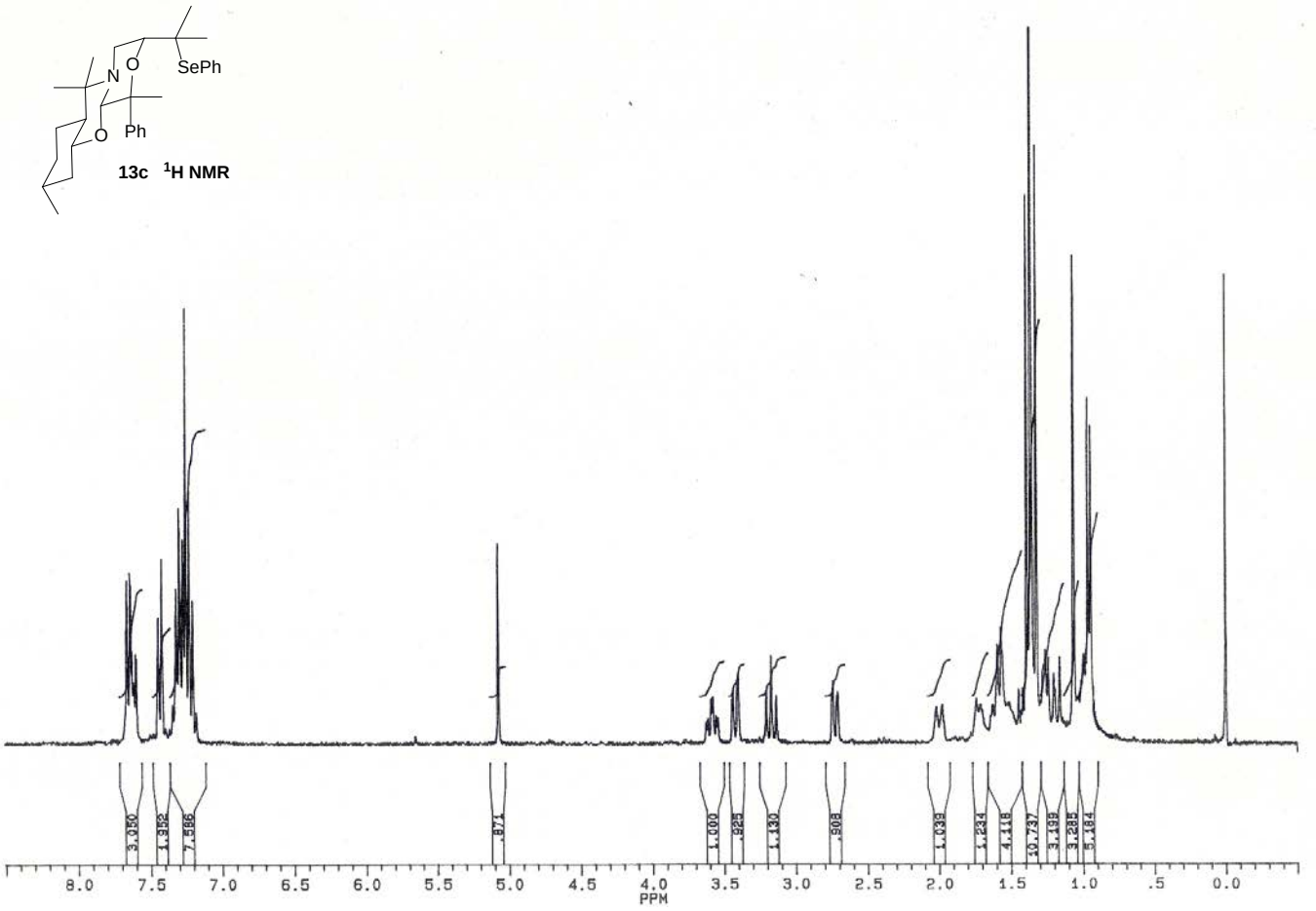
CX2	15.00 cm
CX1	15.00 cm
F2PLO	8.034 ppm
F2LO	2411.32 Hz
F2PHI	-0.295 ppm
F2HI	-88.68 Hz
F1PLO	8.034 ppm
F1LO	2411.40 Hz
F1PHI	-0.295 ppm
F1HI	-88.60 Hz
F2PPMCM	0.55531 ppm/cm
F2HZCM	166.66666 Hz/cm
F1PPMCM	0.55531 ppm/cm
F1HZCM	166.66669 Hz/cm

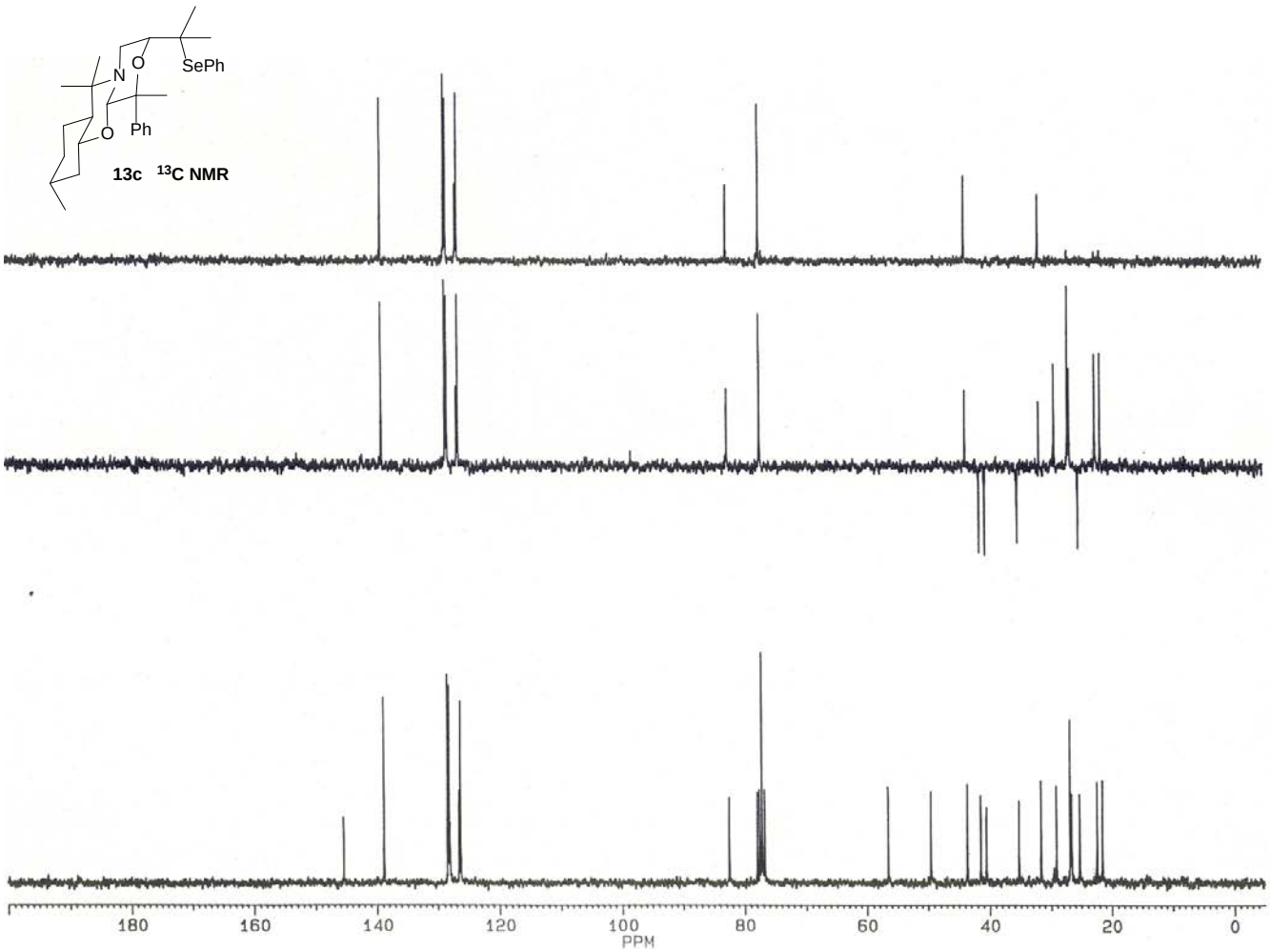


12b COSY


SePh
N
O
O
14b ¹H NMR
1.796
2.885
.927
.936
.966
1.004
.967
.905
1.080
1.064
2.163
.844
6.307
.935
2.936
.846
3.747
11.019
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
.5
0.0
PPM

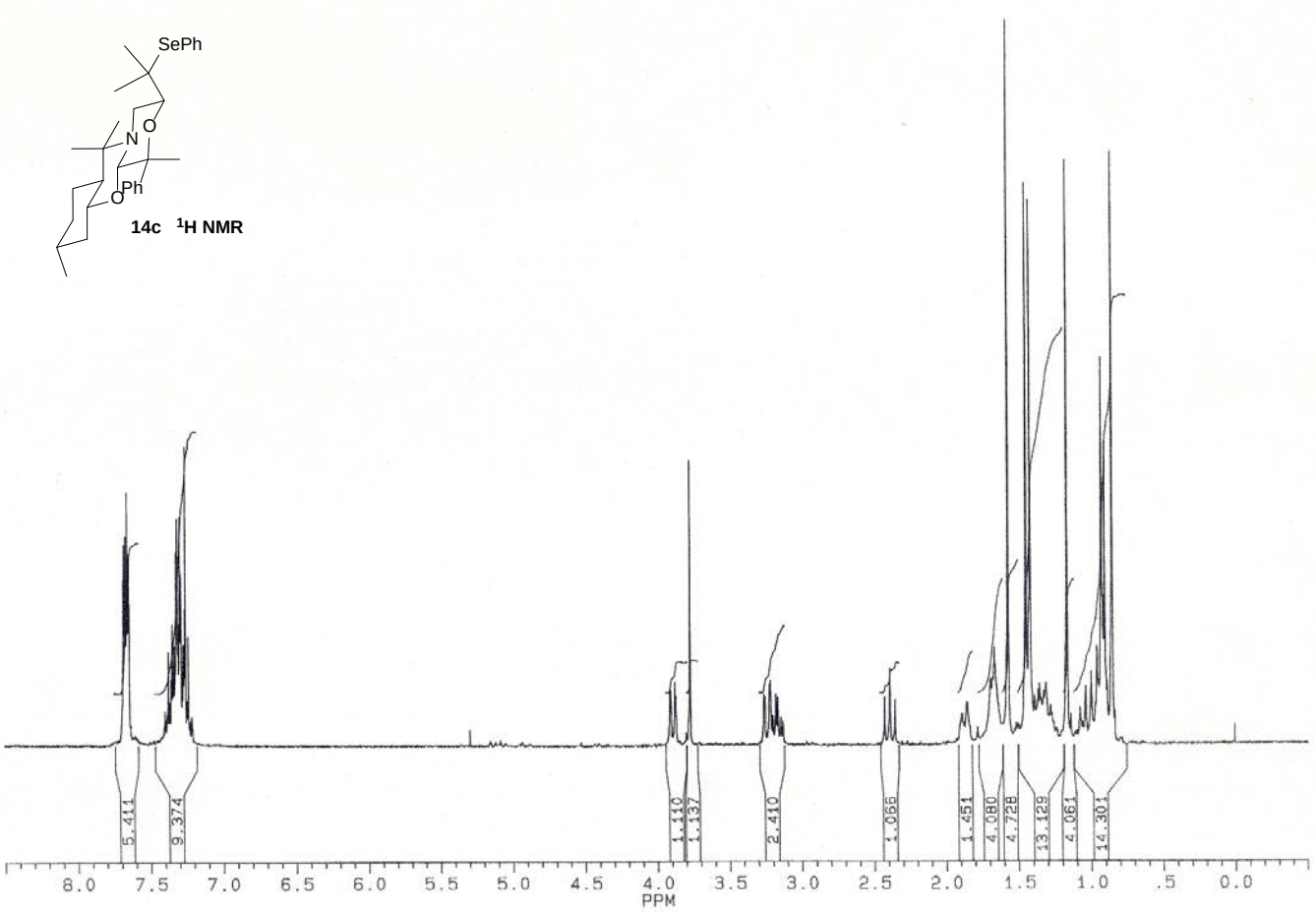

SePh
N
O
O
14b ¹³C NMR DEPT
180
160
140
120
100
80
60
40
20
0
PPM

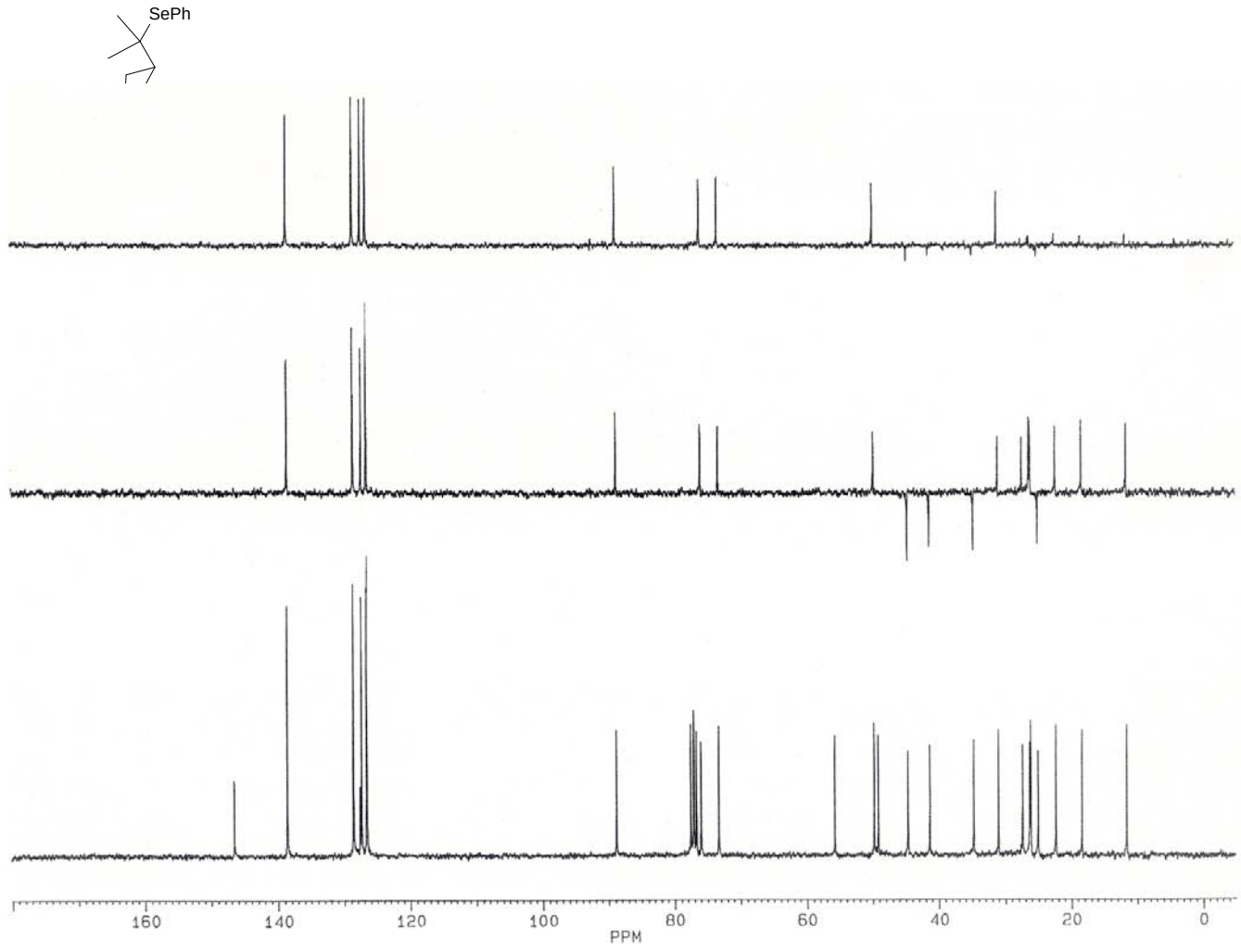

N
O
SePh
O
Ph
13c ^{1}H NMR
3.050
1.352
7.586
.871
1.000
.325
1.130
.908
1.039
1.234
4.118
10.737
3.199
3.285
5.184
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
0.0
PPM

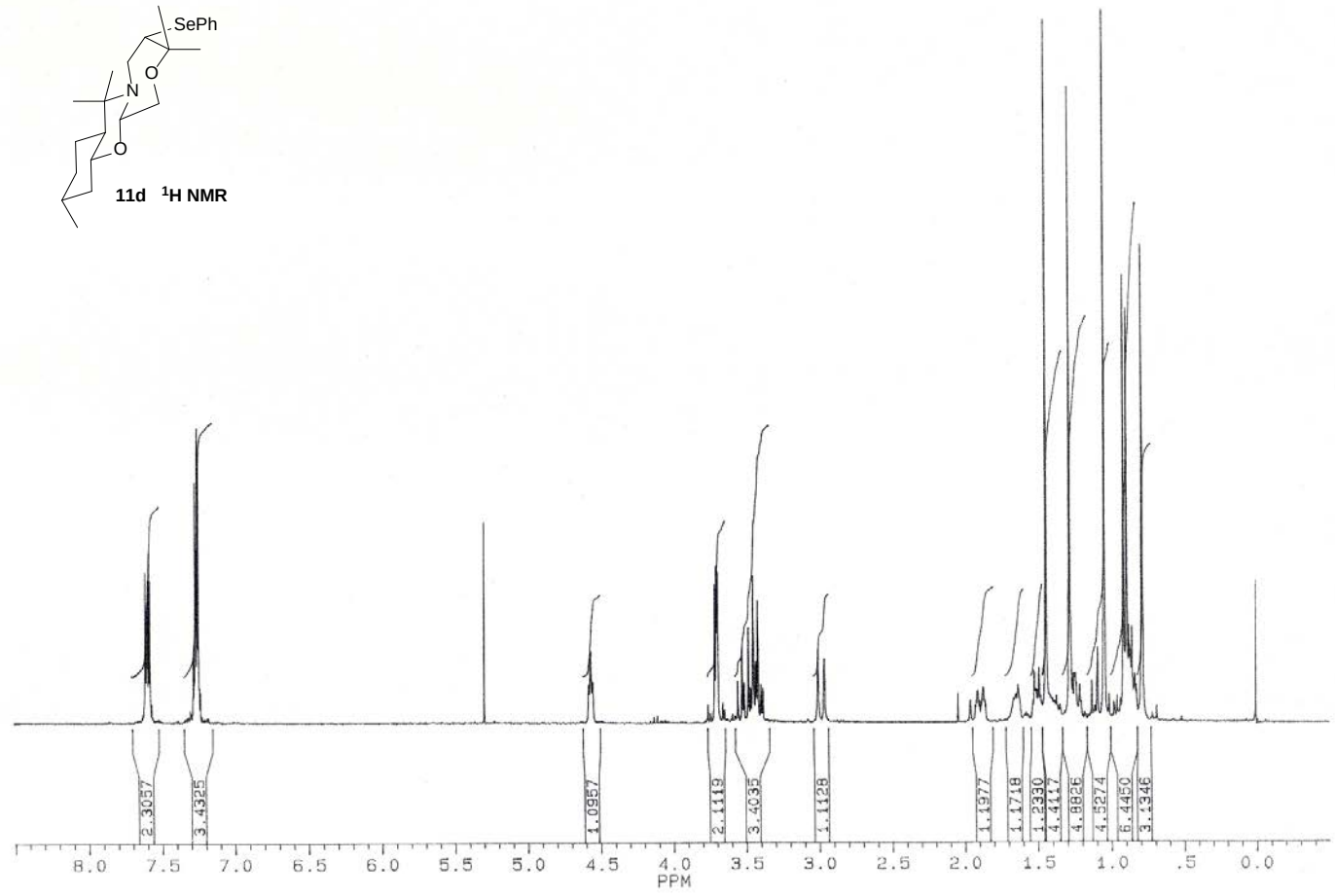

N
O
SePh
O
Ph
13c ^{13}C NMR
180
160
140
120
100
80
60
40
20
0
PPM

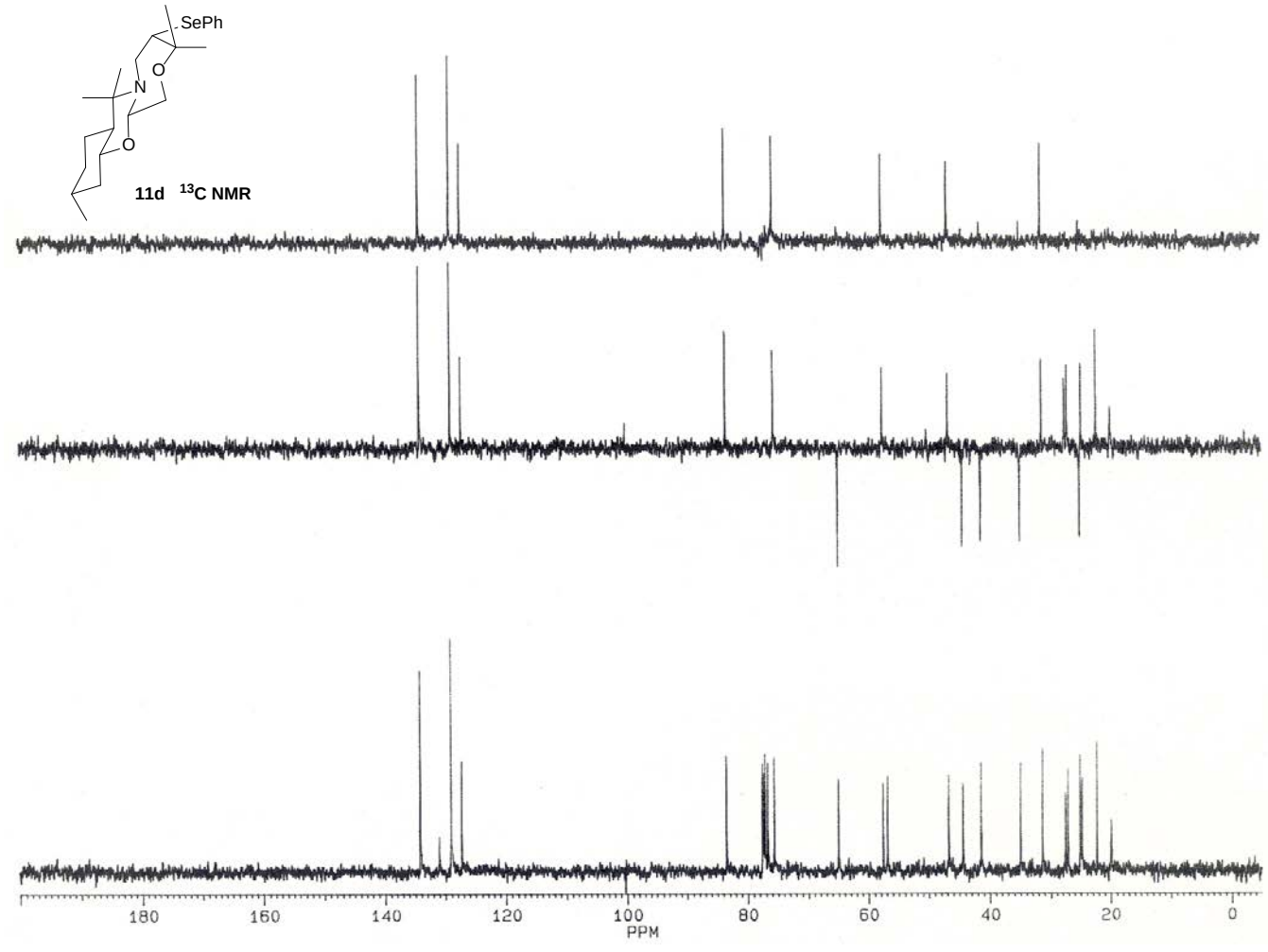
SePh
N
O
O
Ph

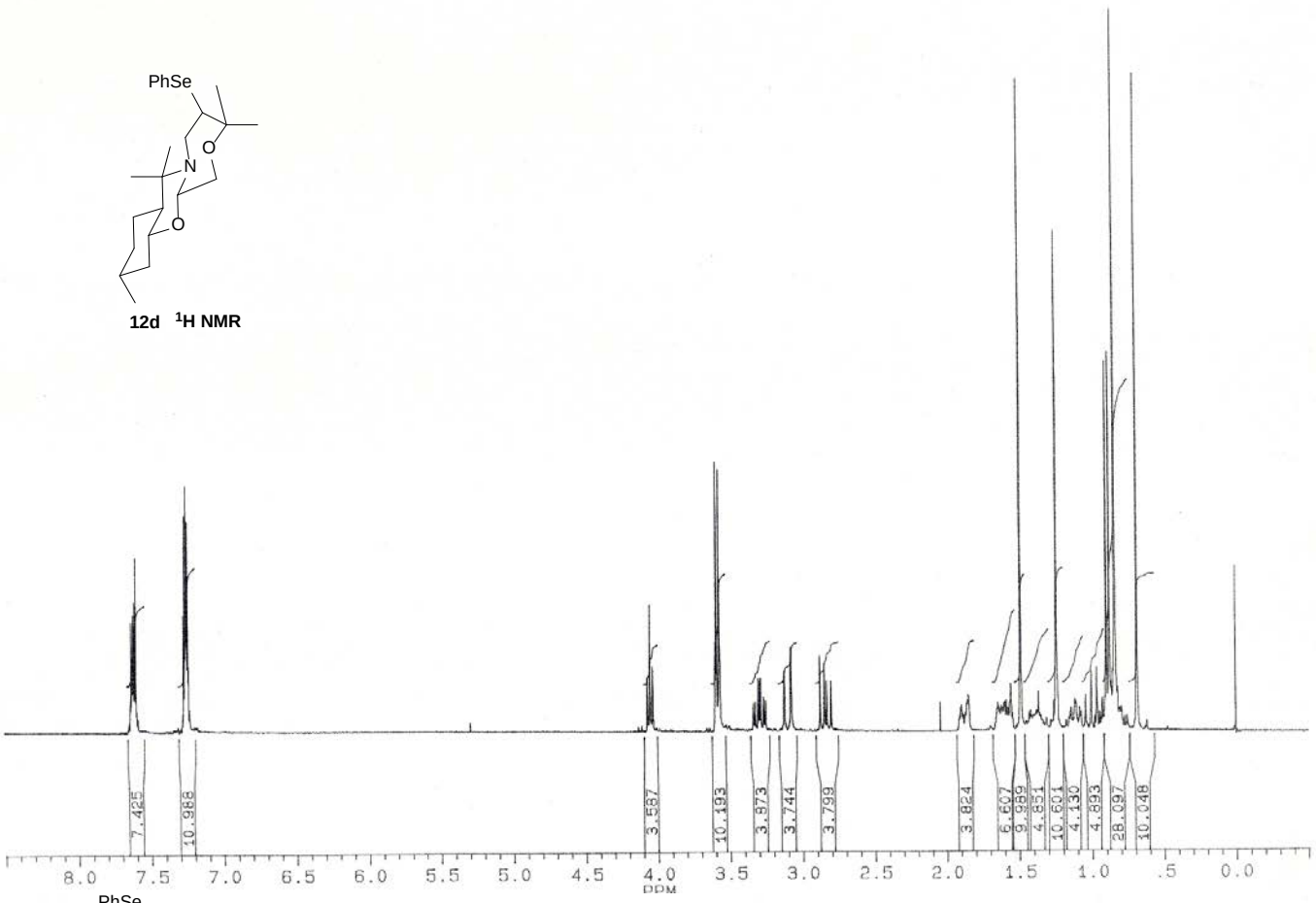
14c ^{1}H NMR


5.411
9.374
1.110
1.137
2.410
1.066
1.451
4.080
4.728
13.129
4.061
14.301
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
.5
0.0
PPM

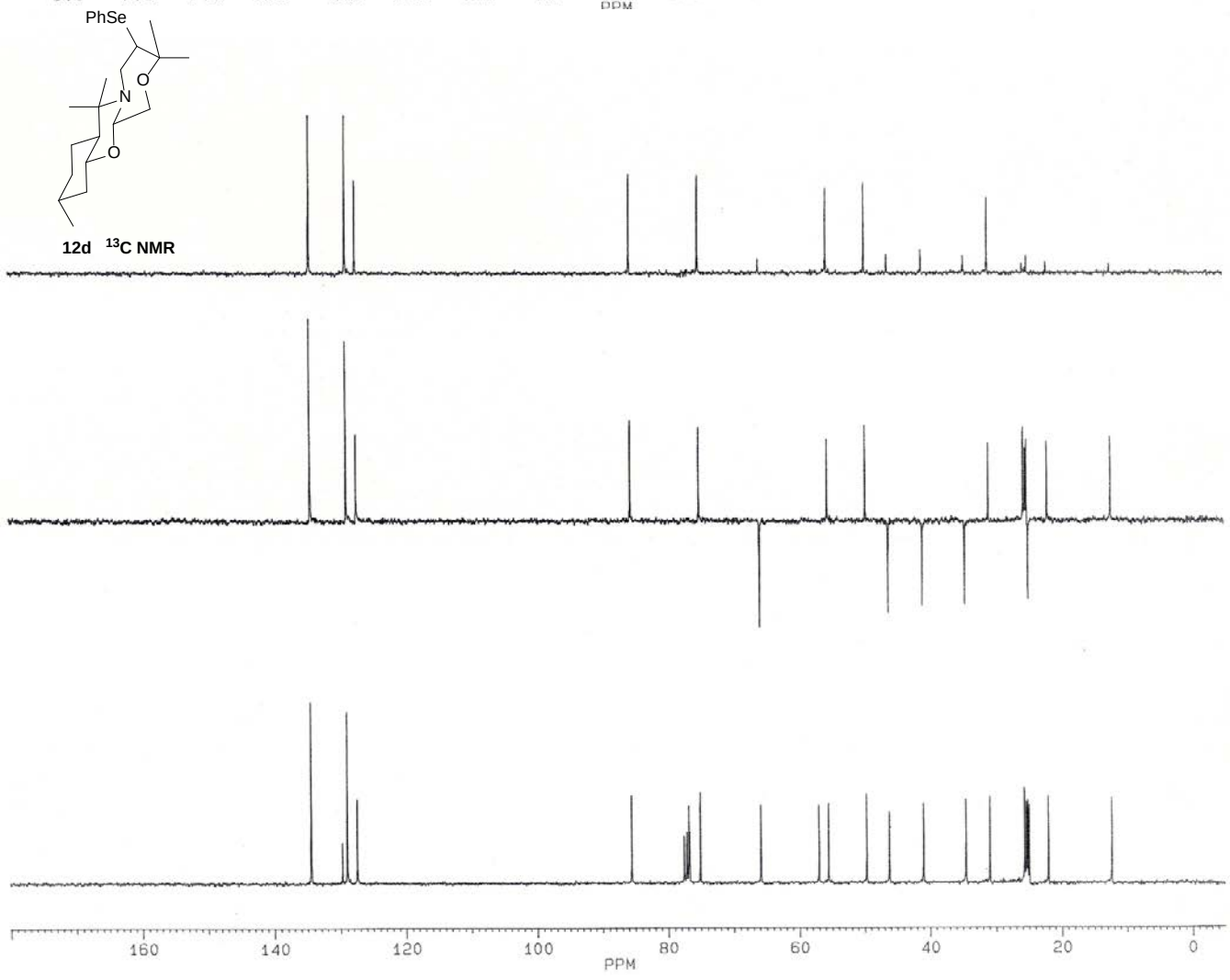

SePh
160
140
120
100
80
60
40
20
0
PPM


SePh
N
O
O
11d ^{1}H NMR
2.3057
3.4325
1.0957
2.1119
3.4035
1.1128
1.1977
1.1718
1.2330
4.4117
4.8826
4.3274
6.4450
3.1346
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 .5 0.0
PPM


SePh
N
O
O
11d ^{13}C NMR
180 160 140 120 100 80 60 40 20 0
PPM

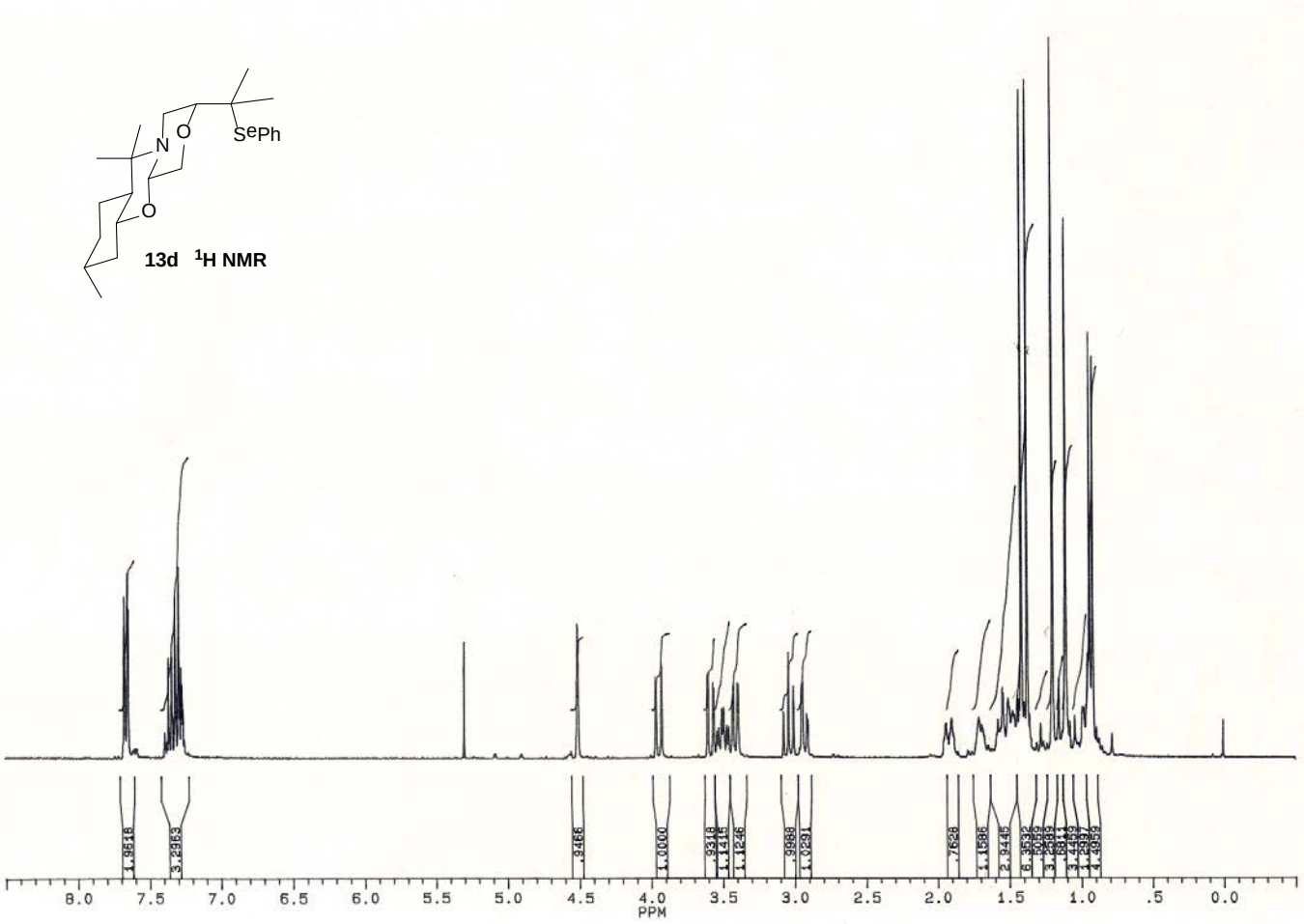


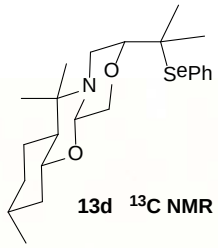
12d ^{1}H NMR



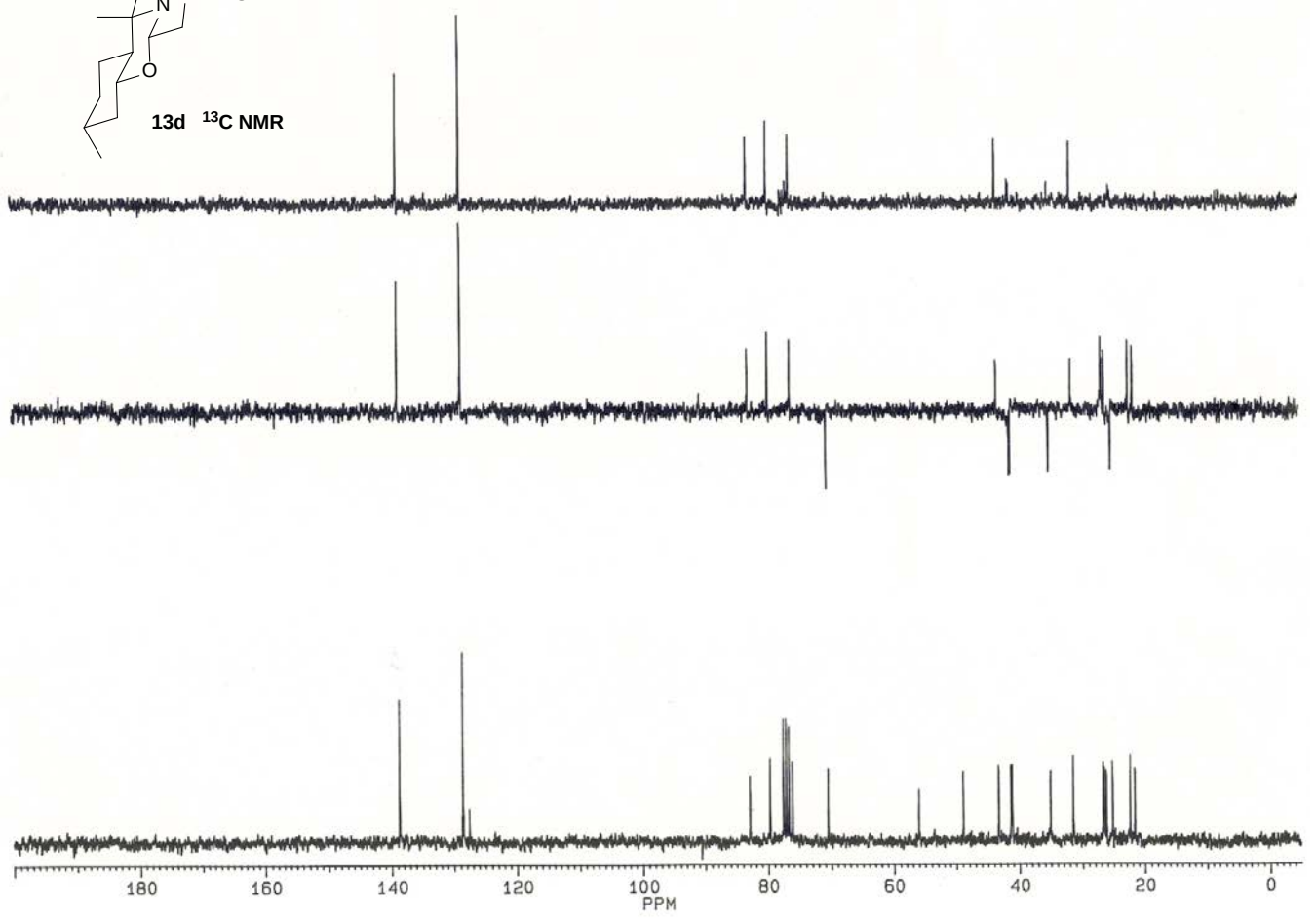
12d ^{13}C NMR

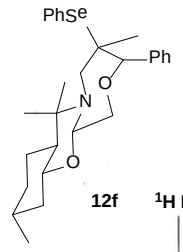
13d ^{1}H NMR



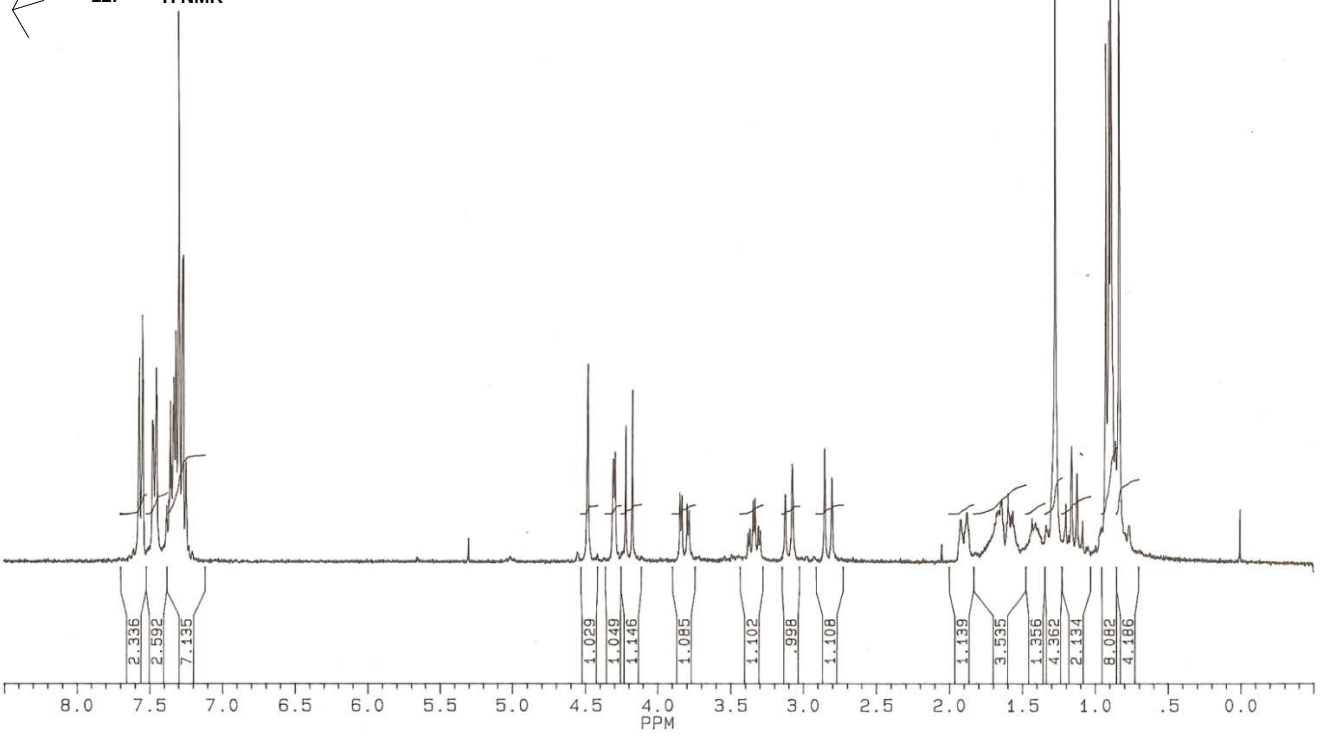


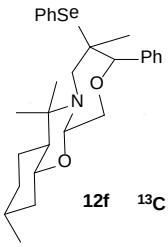
13d ^{13}C NMR



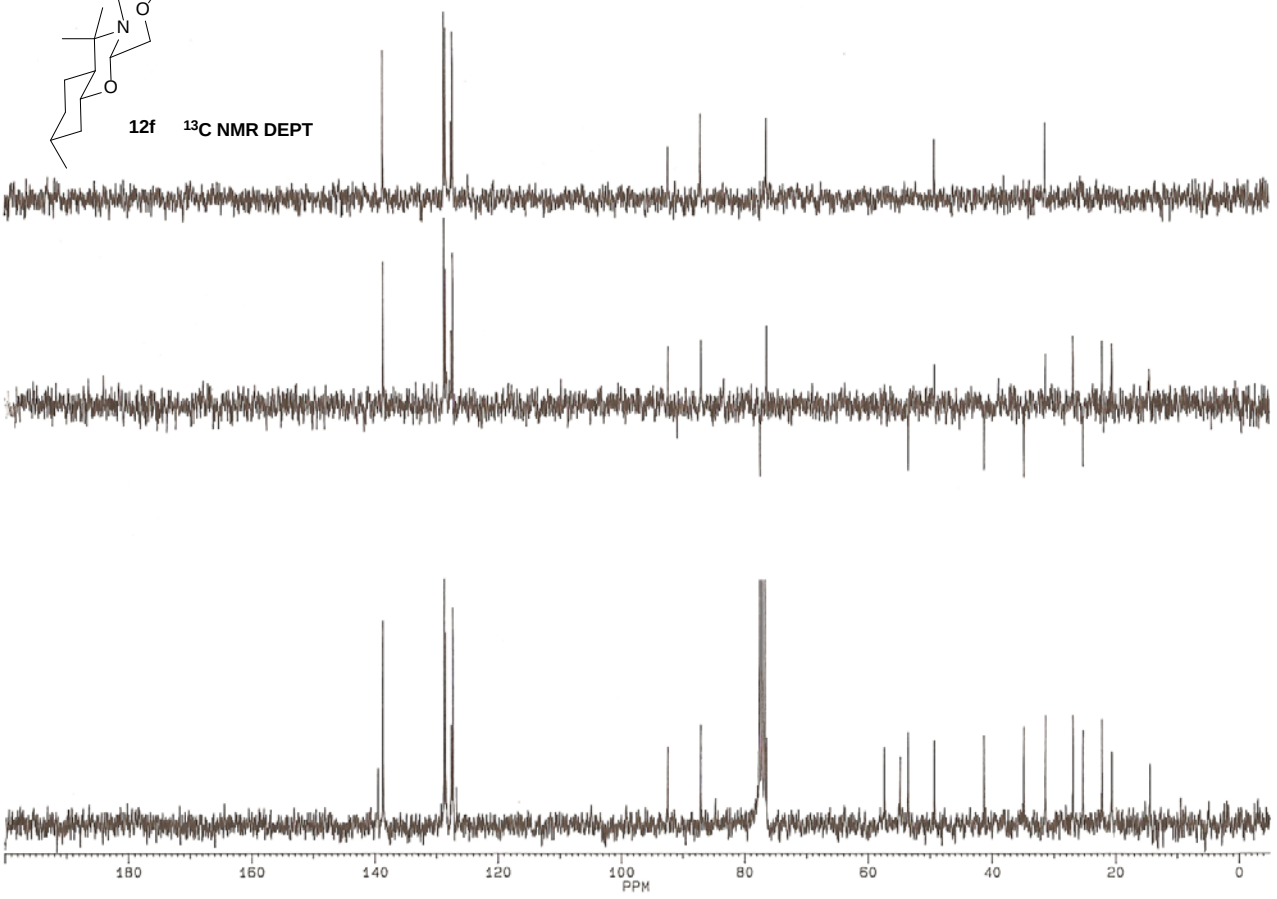


12f **^{1}H NMR**

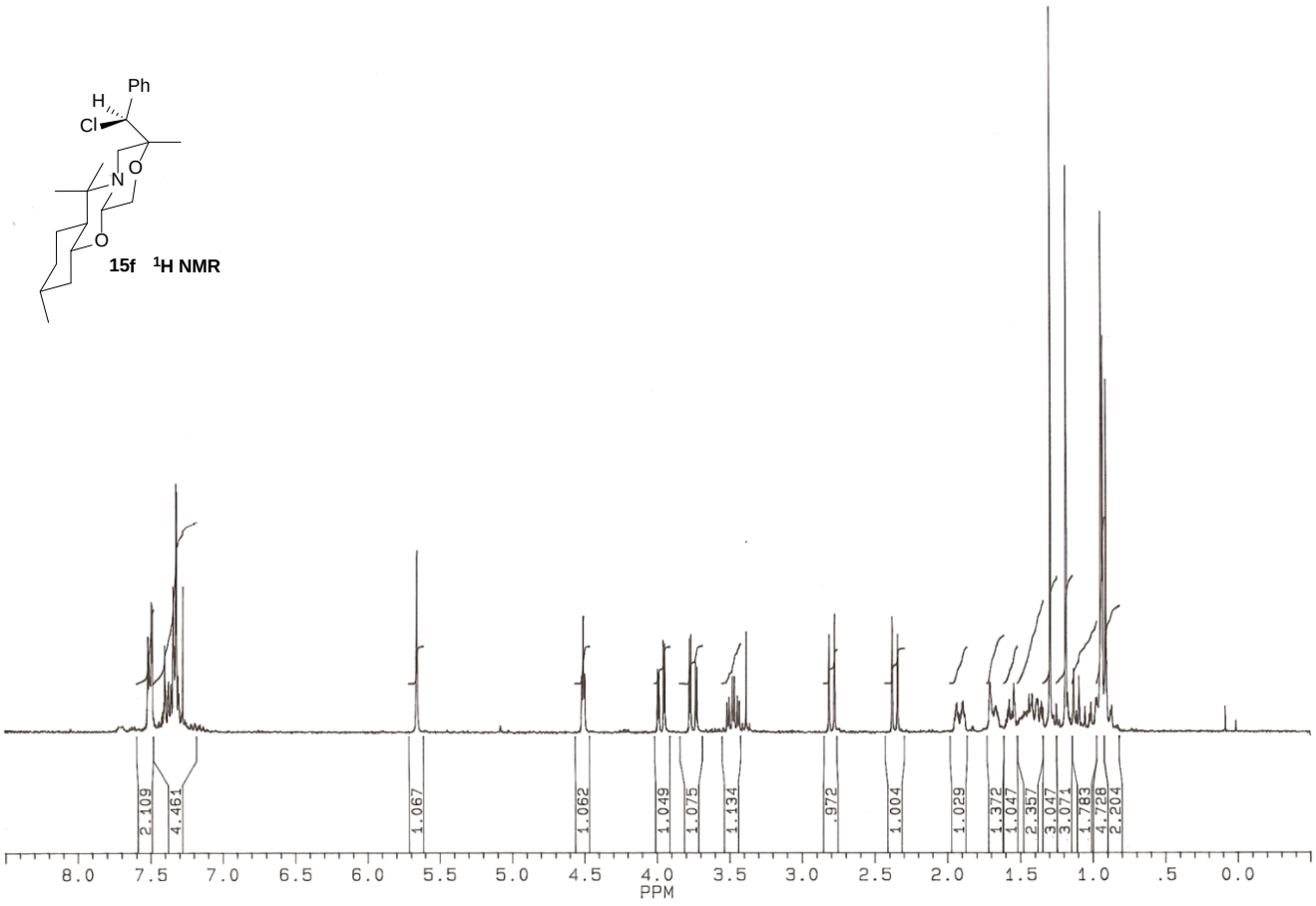


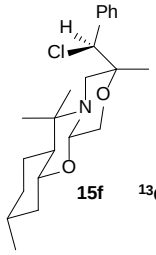


12f **^{13}C NMR DEPT**

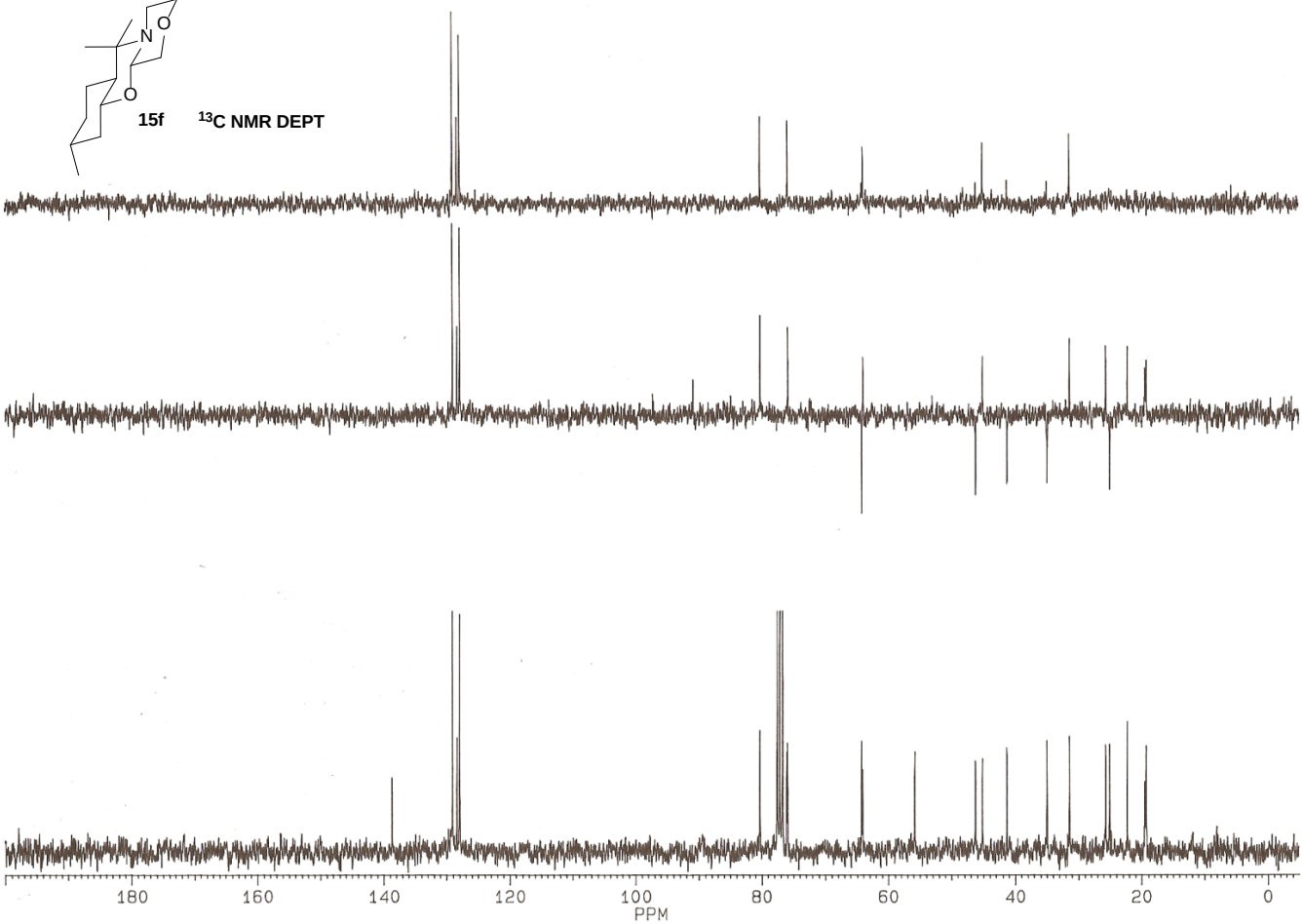


15f **^{1}H NMR**

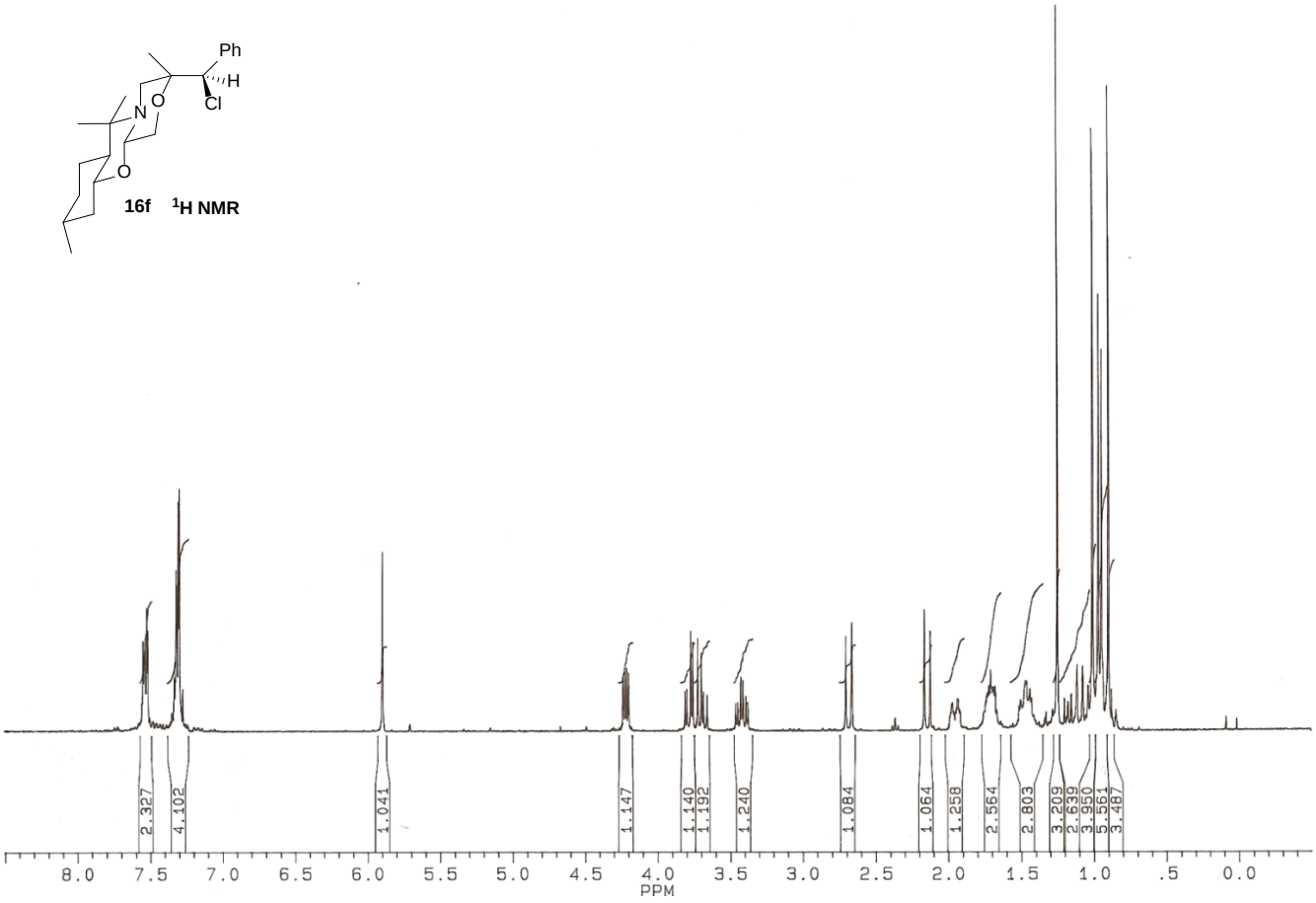


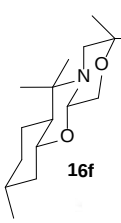


15f **^{13}C NMR DEPT**

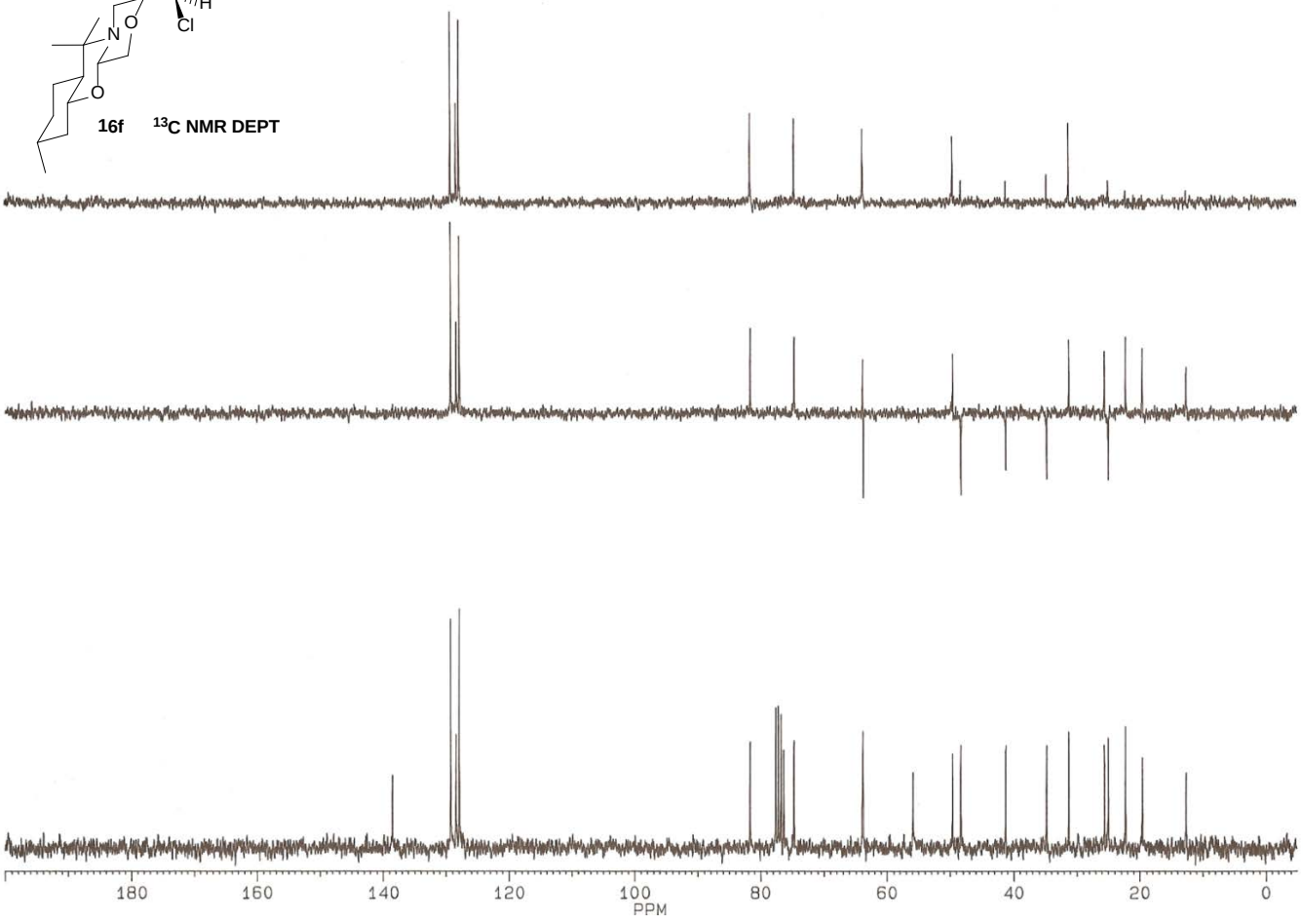


16f **^{1}H NMR**

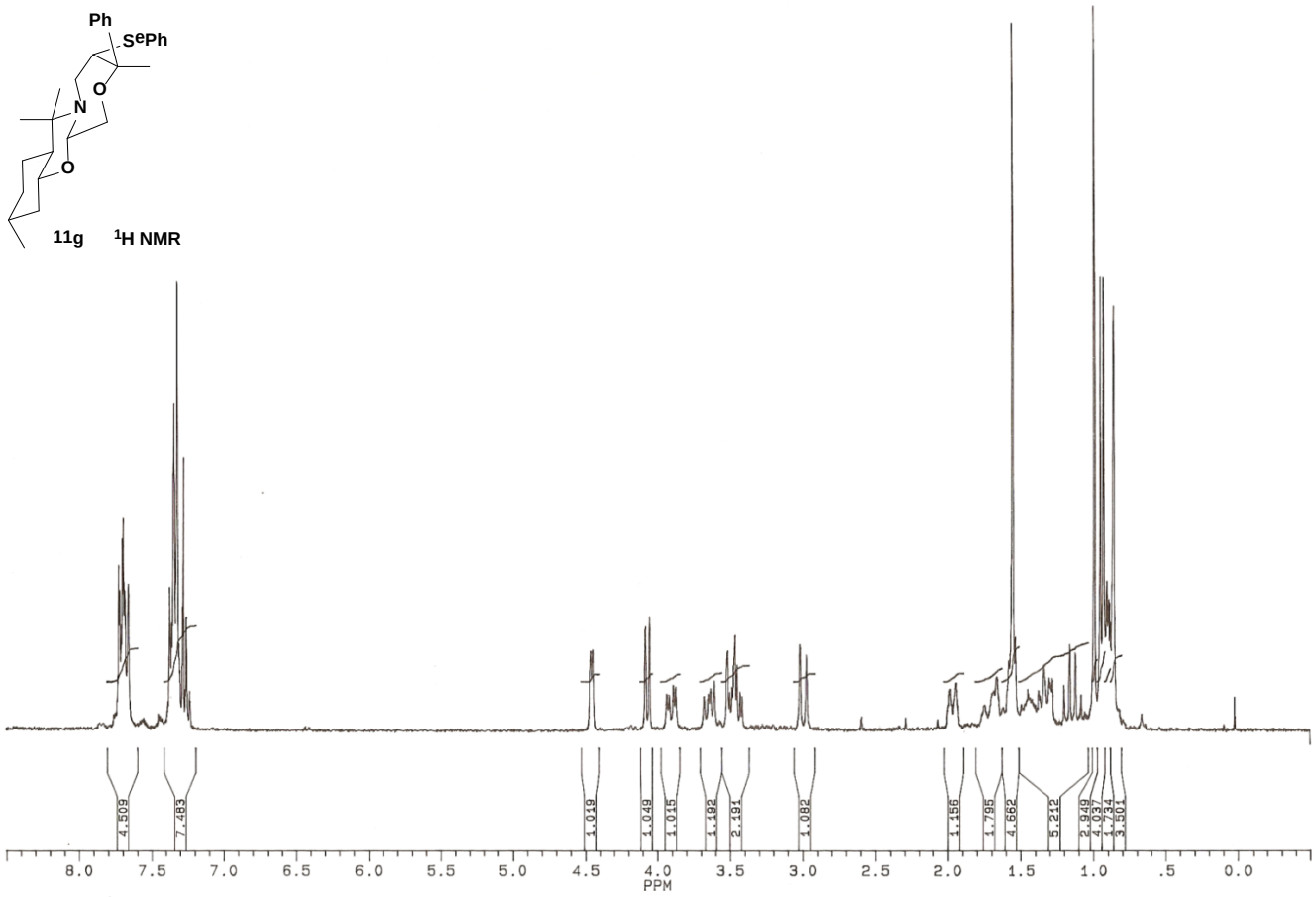


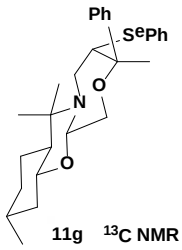


16f **^{13}C NMR DEPT**

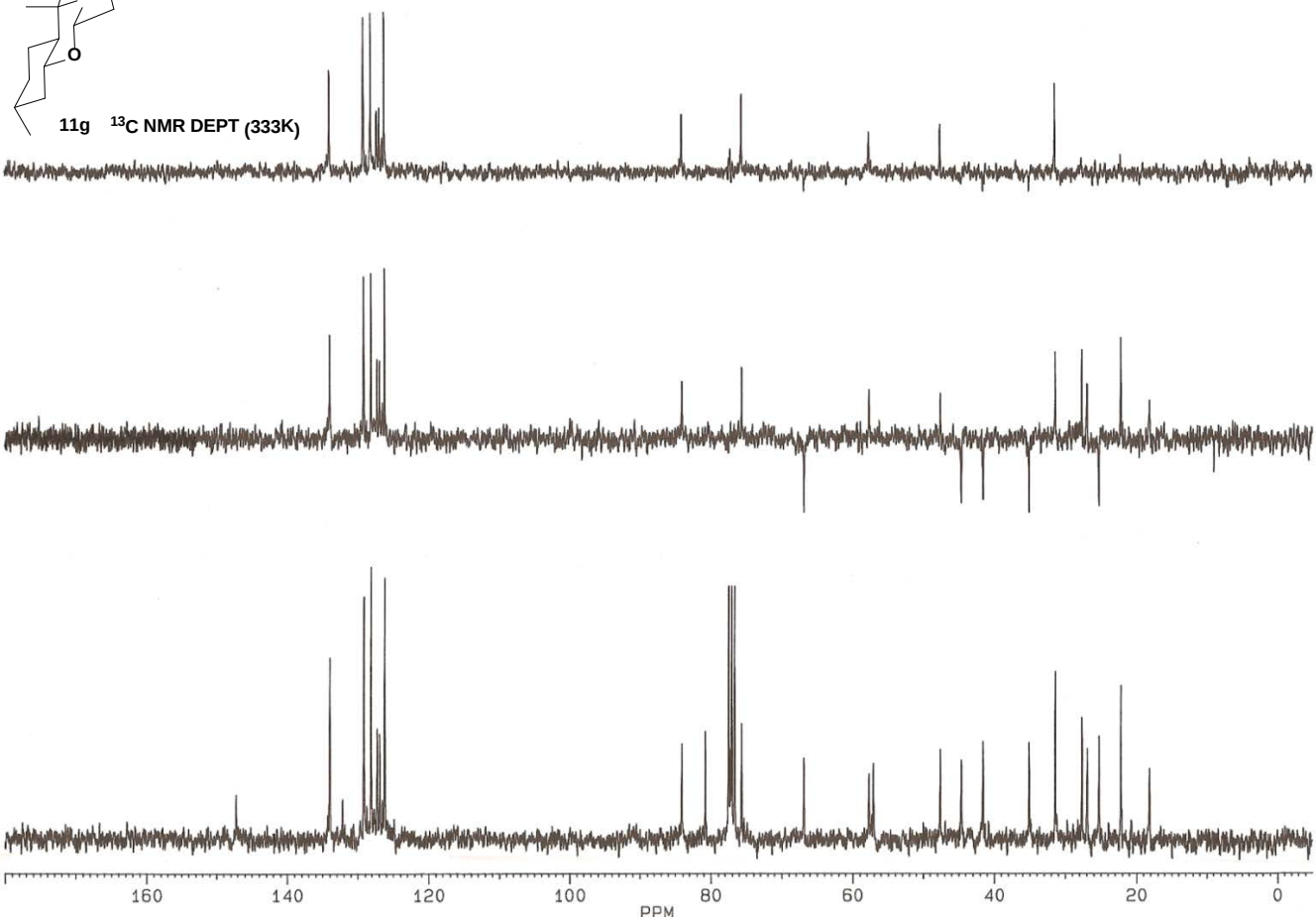


11g ^{1}**H NMR**

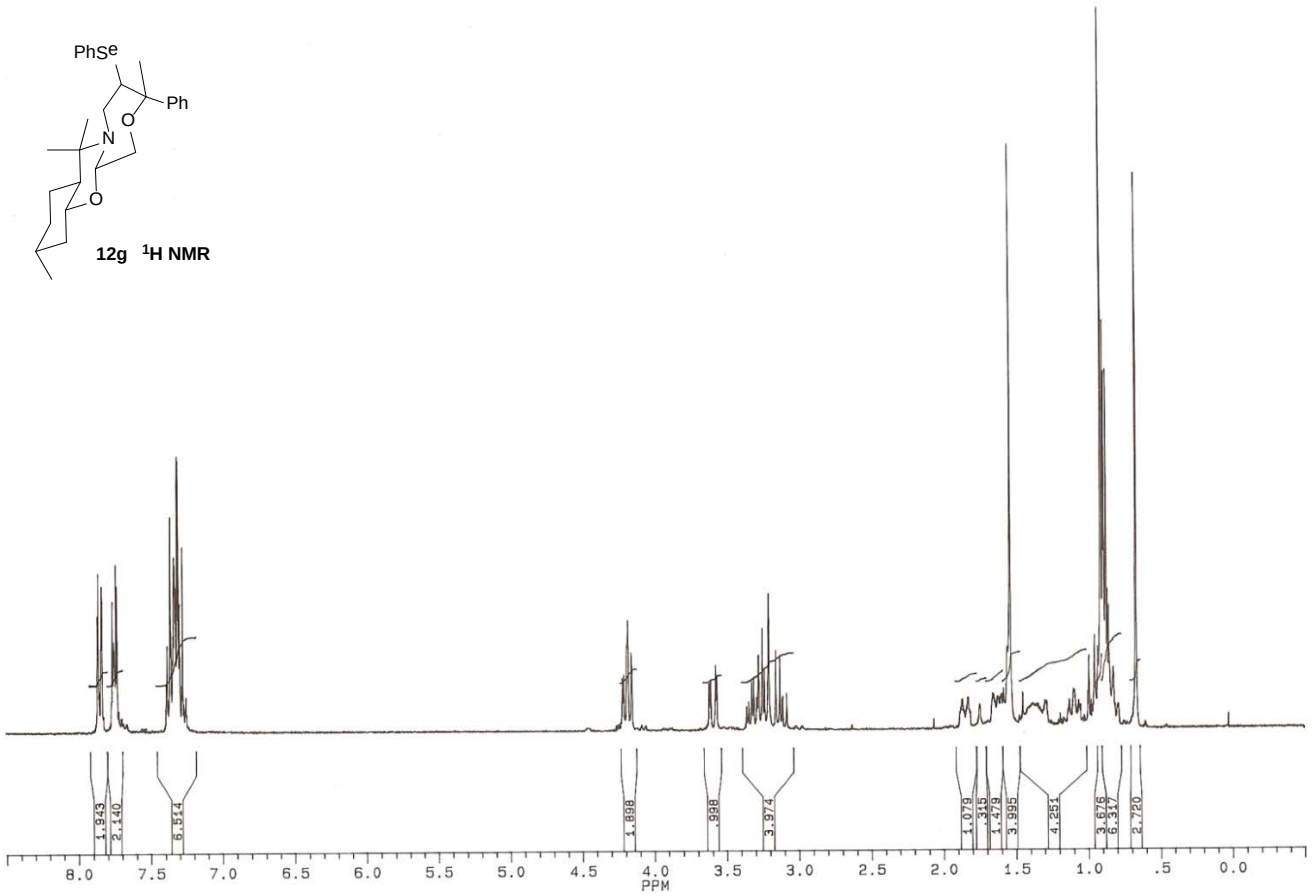


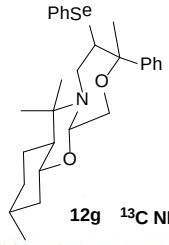


11g ^{13}**C NMR DEPT (333K)**

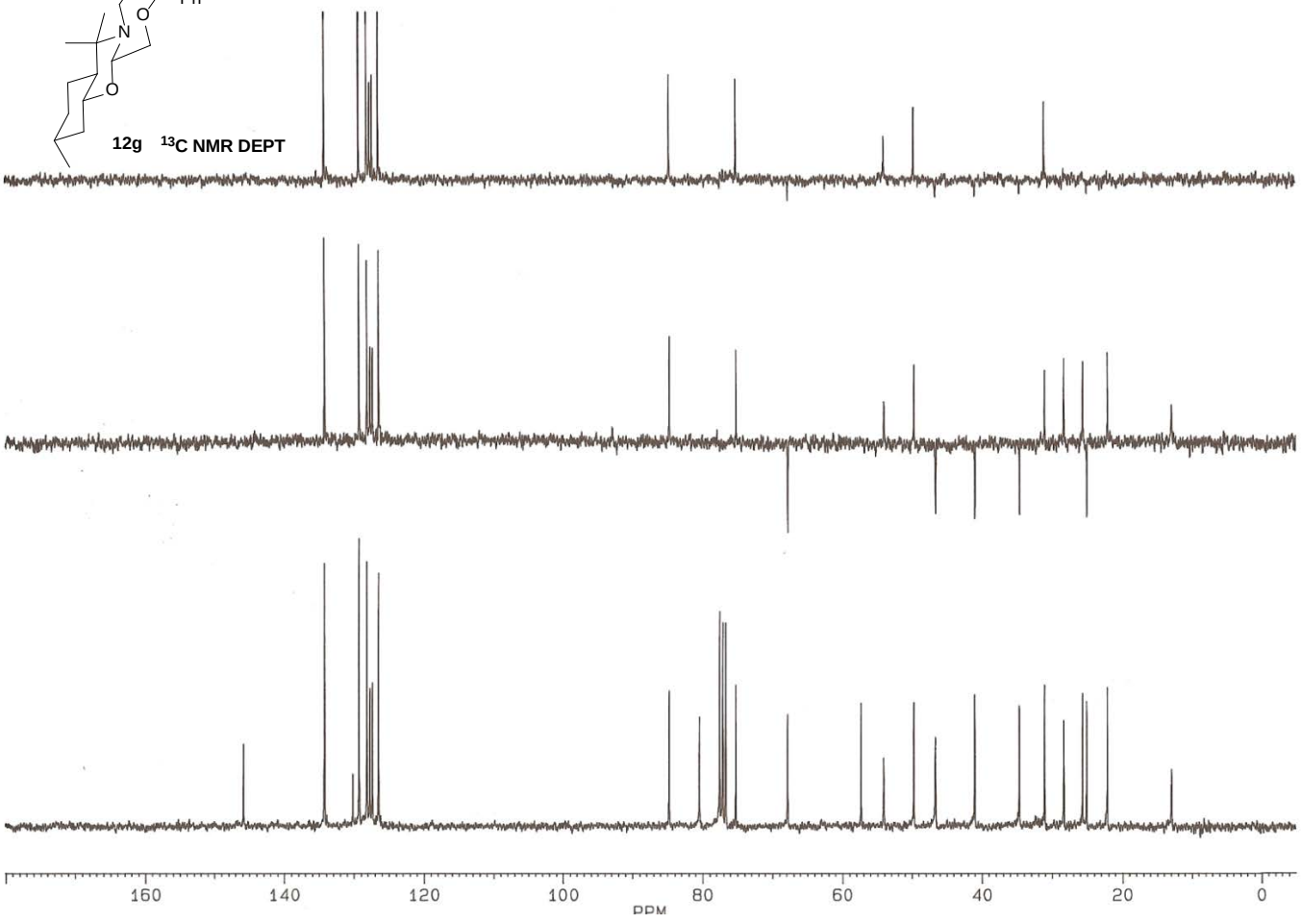


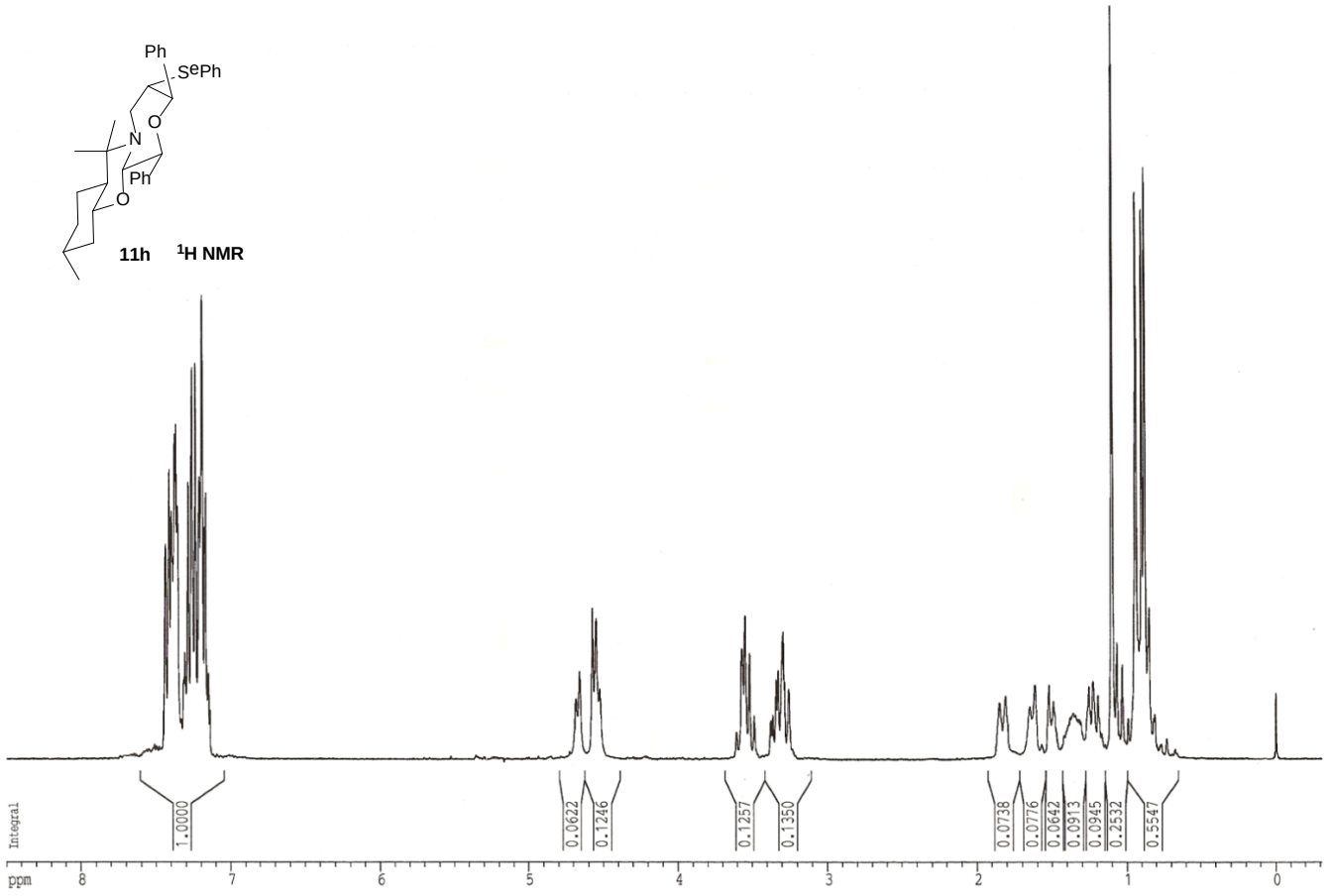
12g ^{1}H NMR

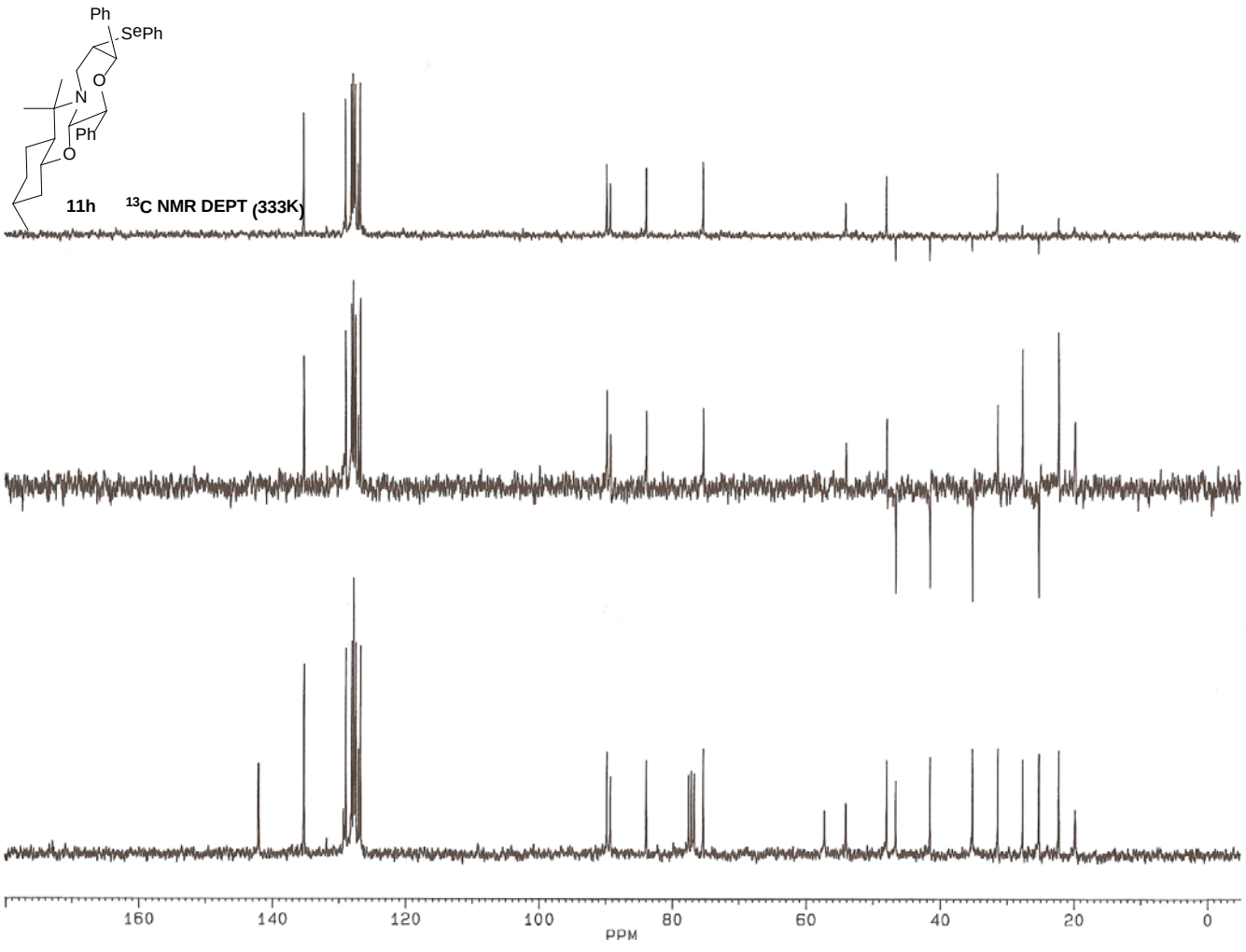


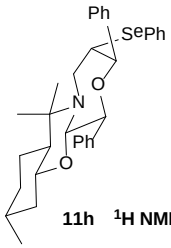


12g ^{13}C NMR DEPT

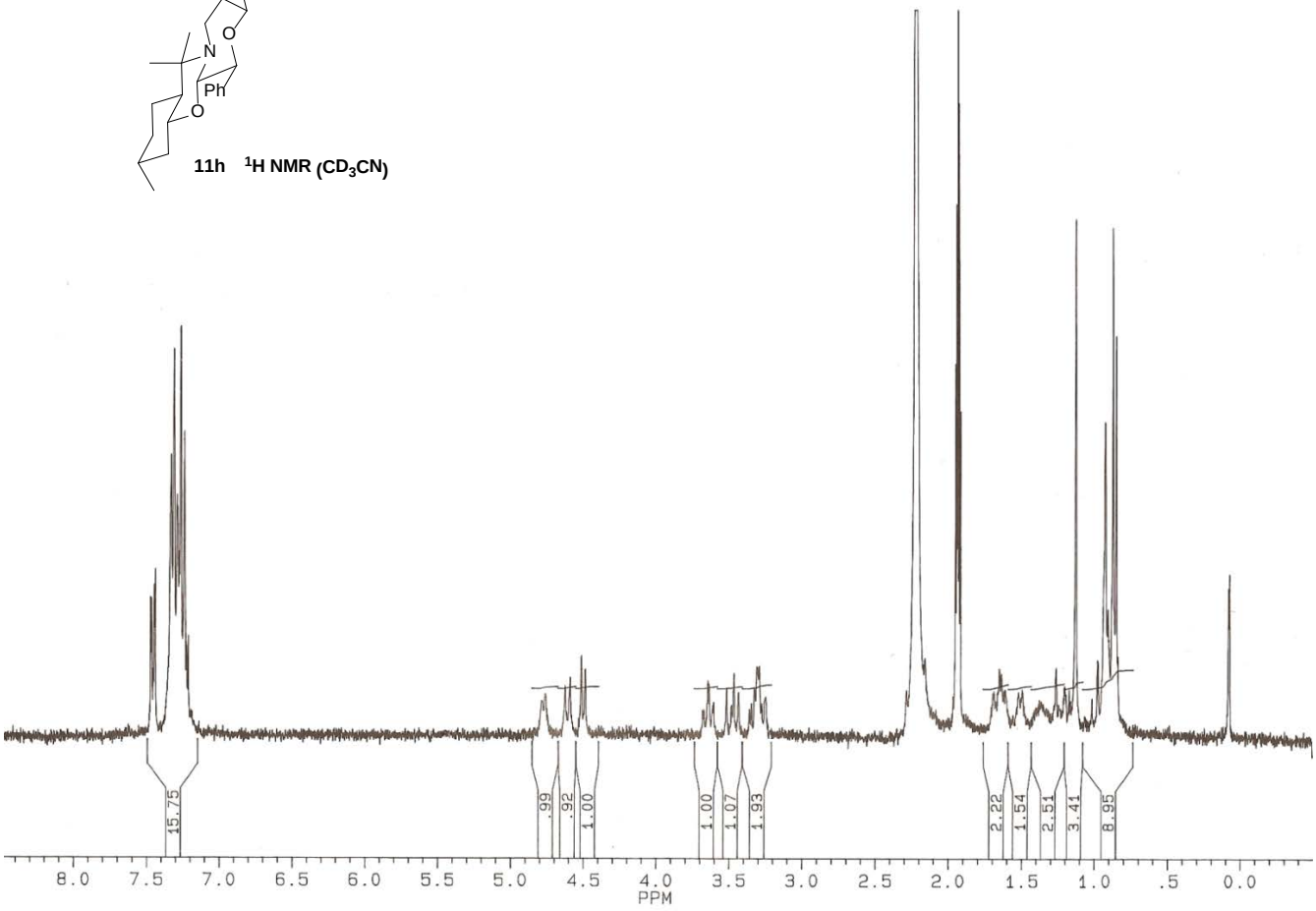


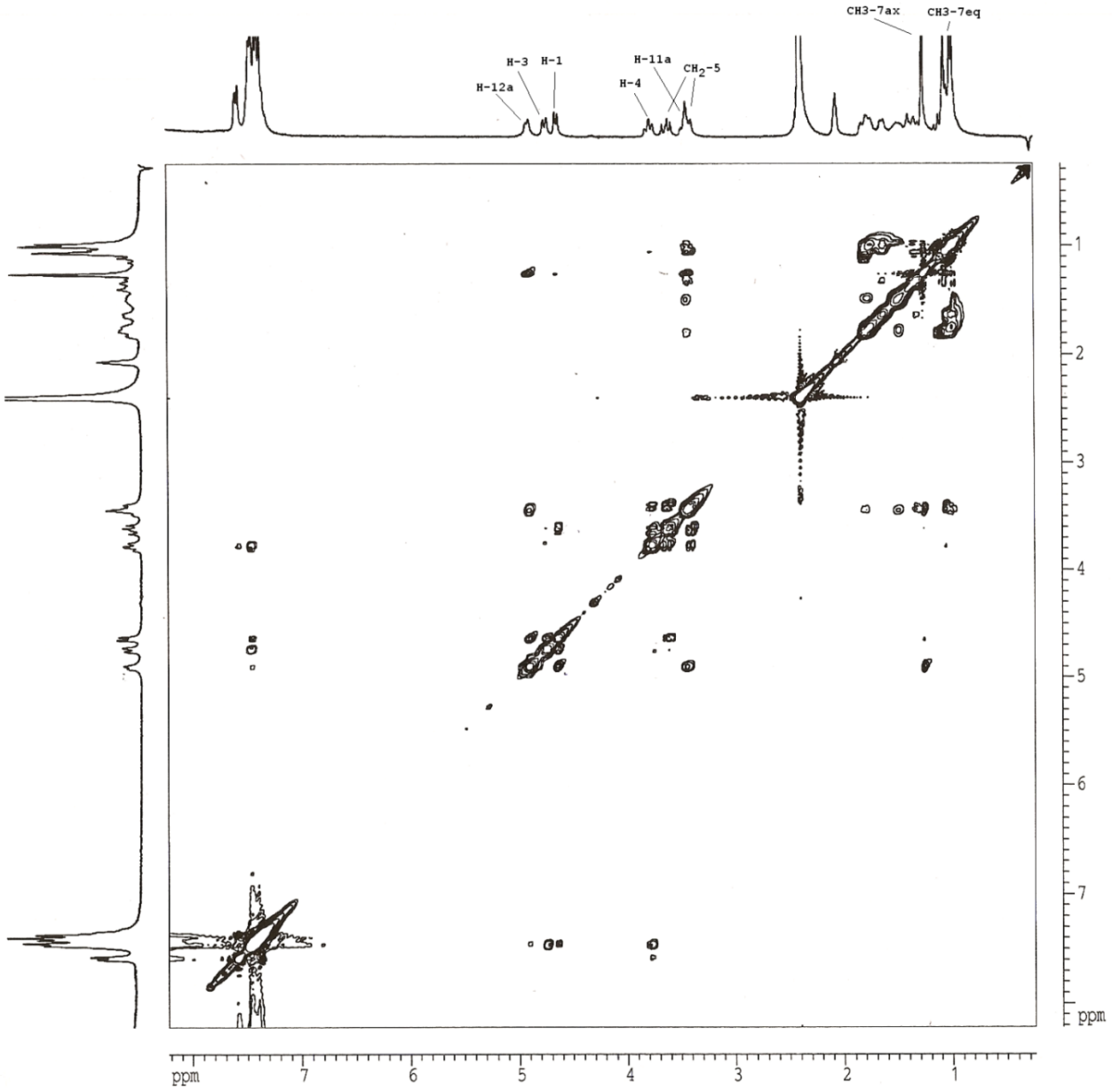

Ph
SePh
O
N
Ph
O
11h ¹H NMR
Integral
1.0000
0.0622
0.1246
0.1257
0.1350
0.0738
0.0776
0.0642
0.0913
0.0945
0.2532
0.5547
ppm
8
7
6
5
4
3
2
1
0


Ph
SePh
O
N
Ph
O
11h ¹³C NMR DEPT (333K)
160
140
120
100
80
60
40
20
0
PPM



11h ^{1}**H NMR (**CD_3CN**)**





Current Data Parameters

NAME	pm080699
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	990526
Time	3.57
PULPROG	noesytp
SOLVENT	~~CHCl3~~ CD_3CN
AQ	0.2140360 sec
FIDRES	2.336274 Hz
DW	209.0 usec
RG	256
NUCLEUS	1H
D1	2.0000000 sec
P1	7.3 usec
D0	0.0000030 sec
D8	0.8000000 sec
DE	261.3 usec
SFO1	300.1361955 MHz
SWH	2392.34 Hz
TD	1024
NS	40
DS	2
IN0	0.0002090 sec

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	300.1362 MHz
FIDRES	9.345140 Hz
SW	7.971 ppm

F1 - Processing parameters

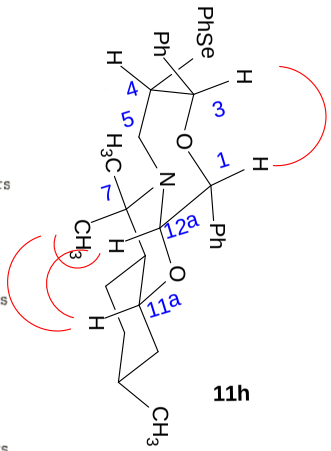
SI	512
MC2	QF
SF	300.1349246 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	TPPI
SF	300.1349246 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

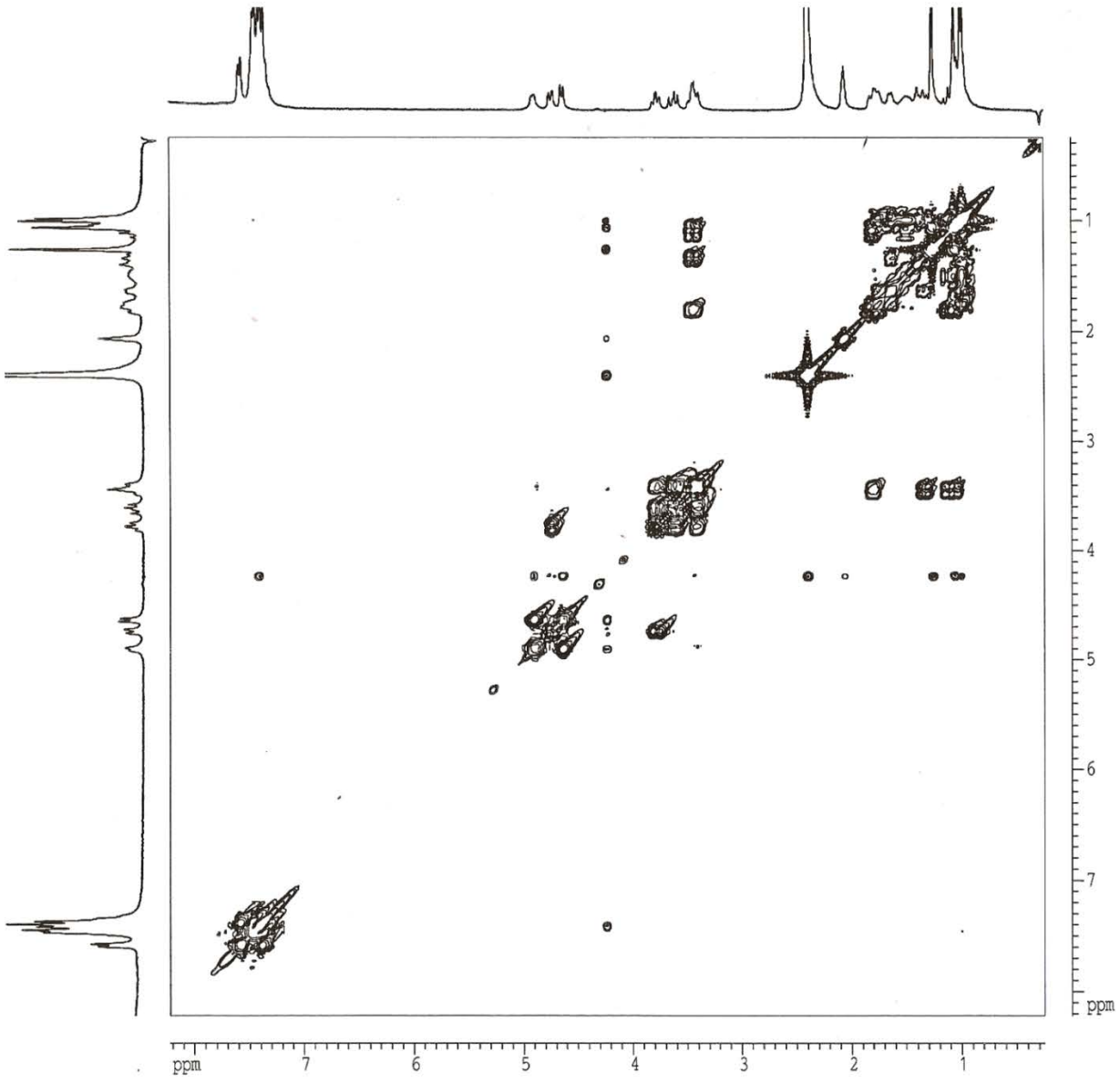
2D NMR plot parameters

CX2	16.00 cm
CX1	16.00 cm
F2PLO	8.220 ppm
F2LO	2467.08 Hz
F2PHI	0.249 ppm
F2HI	74.73 Hz
F1PLO	8.220 ppm
F1LO	2467.08 Hz
F1PHI	0.249 ppm
F1HI	74.72 Hz
F2PPMCM	0.49818 ppm/cm
F2HzCM	149.52153 Hz/cm
F1PPMCM	0.49818 ppm/cm
F1HzCM	149.52223 Hz/cm



11h

^{1}H NOESY contacts (CD_3CN)



Current Data Parameters

NAME	pm080699
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date	990526
Time	18.10
PULPROG	cosy90
SOLVENT	~~CDCl3~~ CD_3CN
AQ	0.1070280 sec
FIDRES	4.672548 Hz
DW	209.0 usec
RG	256
NUCLEUS	1H
D1	1.0000000 sec
P1	7.3 usec
D0	0.0000030 sec
DE	261.3 usec
SFO1	300.1361955 MHz
SWH	2392.34 Hz
TD	512
NS	8
DS	2
IN0	0.0004180 sec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.1362 MHz
FIDRES	9.345140 Hz
SW	7.971 ppm

F1 - Processing parameters

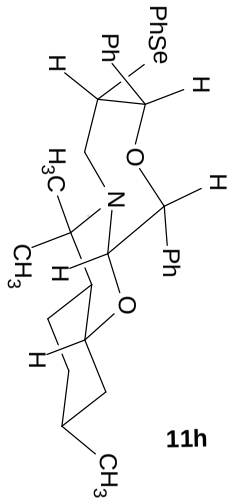
SI	512
MC2	QF
SF	300.1349246 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	QF
SF	300.1349246 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	16.00 cm
CX1	16.00 cm
F2PLO	8.220 ppm
F2LO	2467.08 Hz
F2PHI	0.249 ppm
F2HI	74.73 Hz
F1PLO	8.220 ppm
F1LO	2467.08 Hz
F1PHI	0.249 ppm
F1HI	74.72 Hz
F2PPMCM	0.49818 ppm/cm
F2HZCM	149.52153 Hz/cm
F1PPMCM	0.49818 ppm/cm
F1HZCM	149.52223 Hz/cm

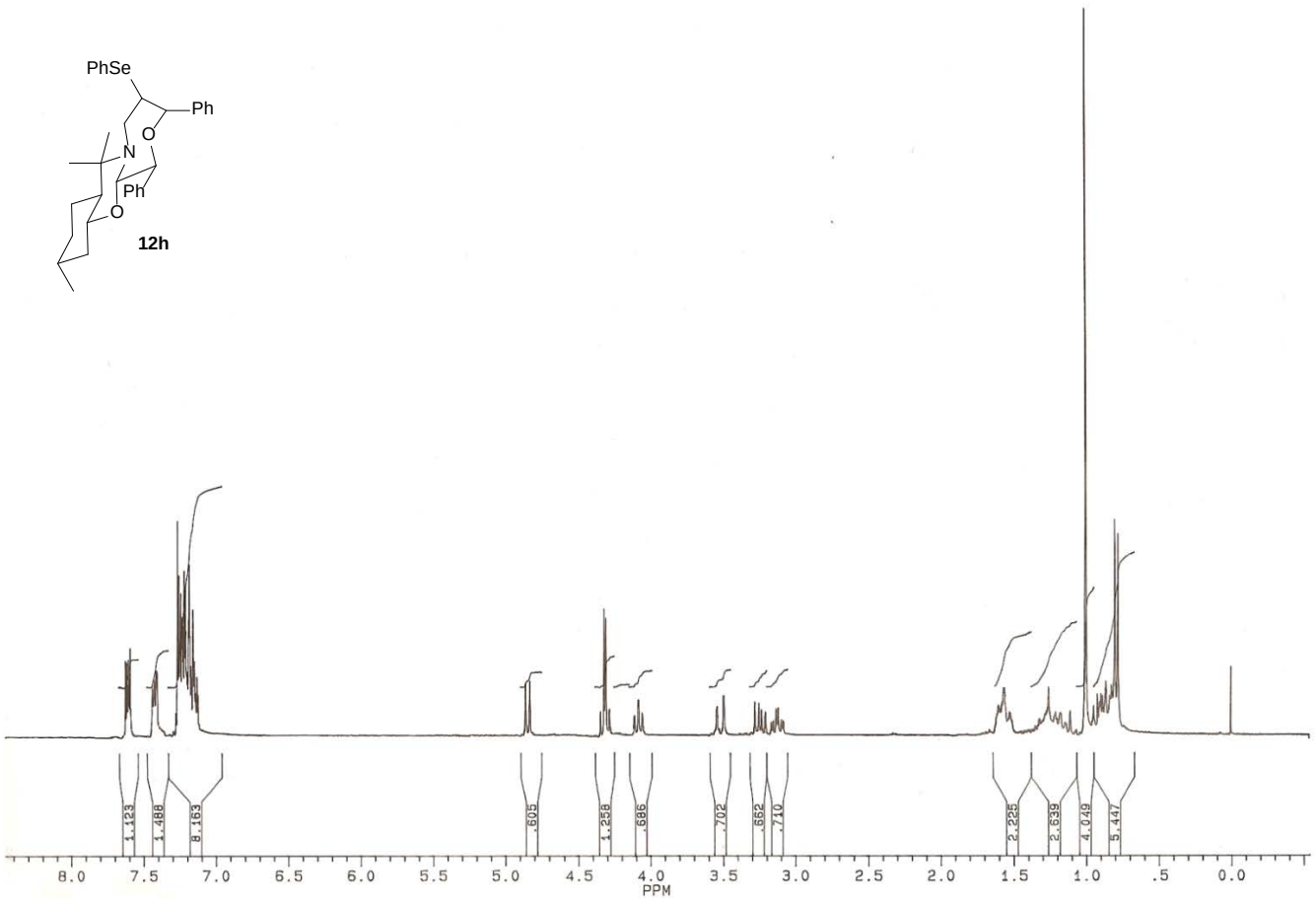


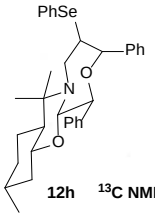
11h

COSY

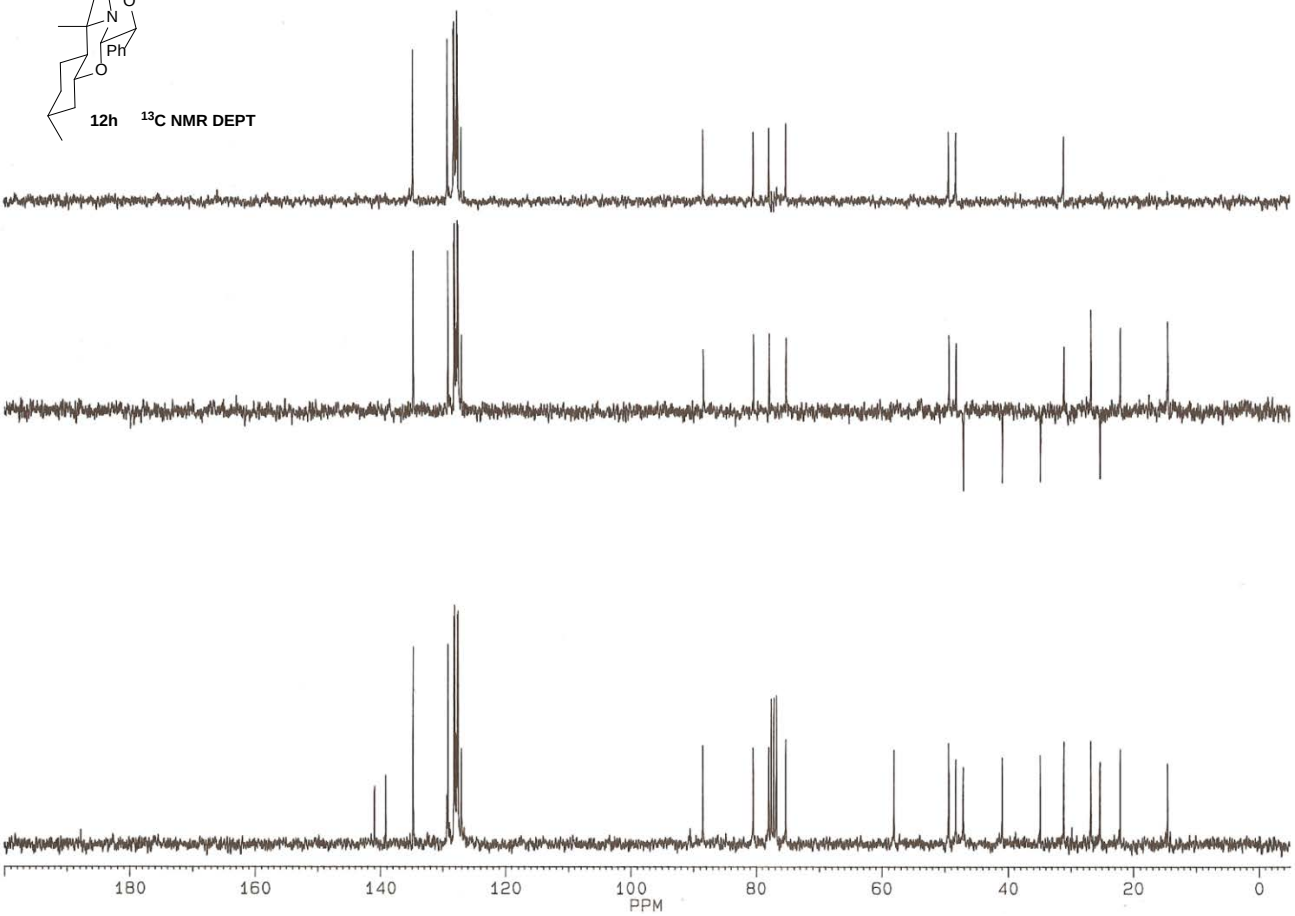
(CD_3CN)

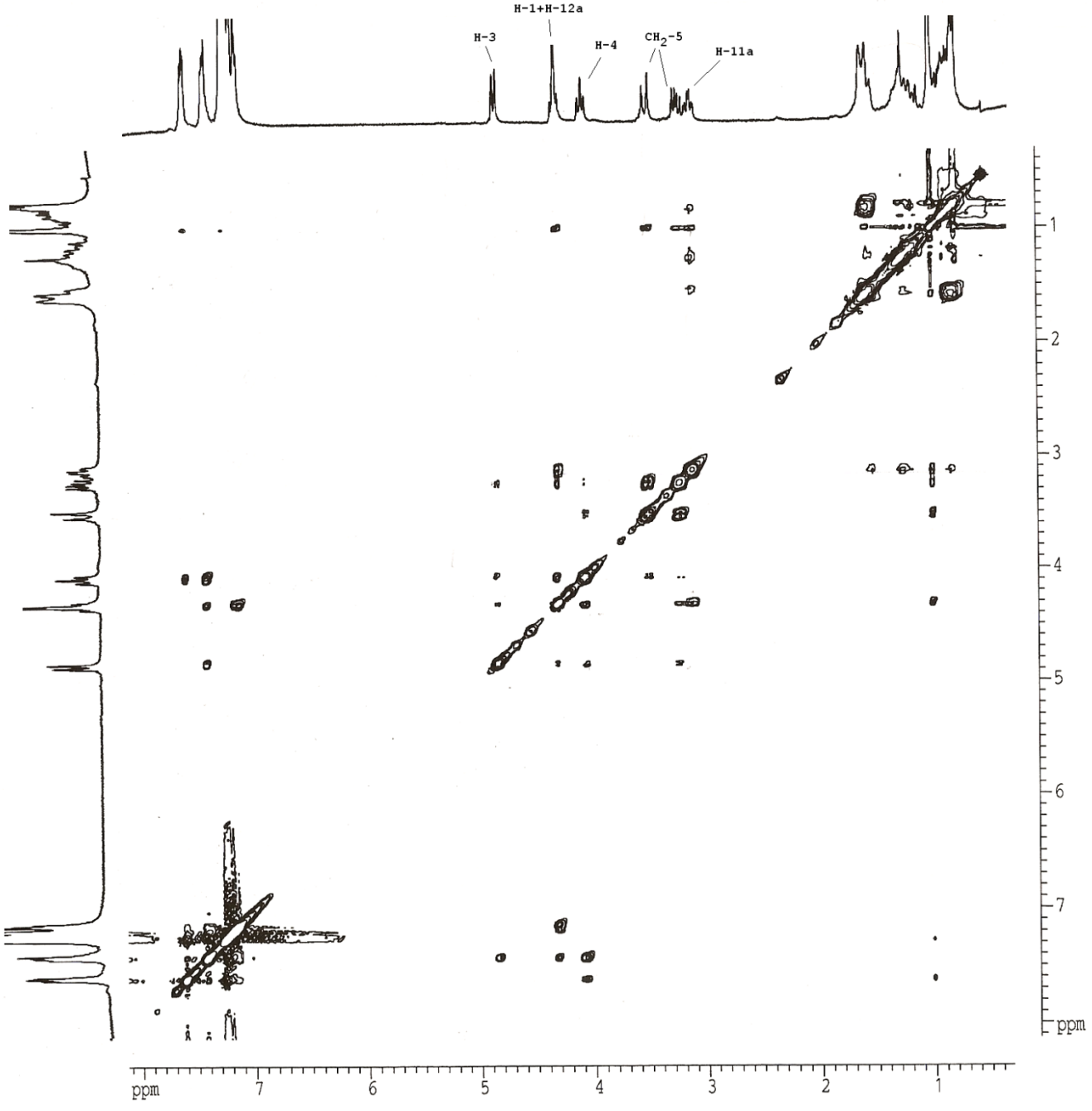
12h





12h **^{13}C NMR DEPT**





Current Data Parameters

NAME	pm110399
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	990226
Time	16.19
PULPROG	noesytp
SOLVENT	CDCl3
AQ	0.2181320 sec
FIDRES	2.292400 Hz
DW	213.0 usec
RG	715
NUCLEUS	1H
D1	2.0000000 sec
P1	7.1 usec
D0	0.0000030 sec
D8	0.8000000 sec
DE	266.3 usec
SFO1	300.1346352 MHz
SWH	2347.42 Hz
TD	1024
NS	40
DS	2
IN0	0.0002130 sec

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	300.1346 MHz
FIDRES	9.169582 Hz
SW	7.821 ppm

F1 - Processing parameters

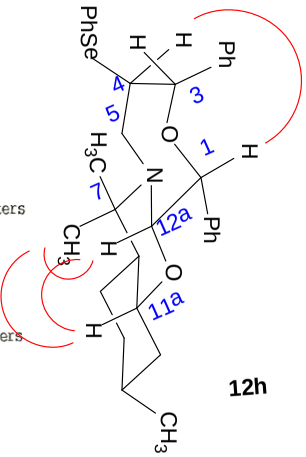
SI	512
MC2	QF
SF	300.1333680 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	TPPI
SF	300.1333680 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

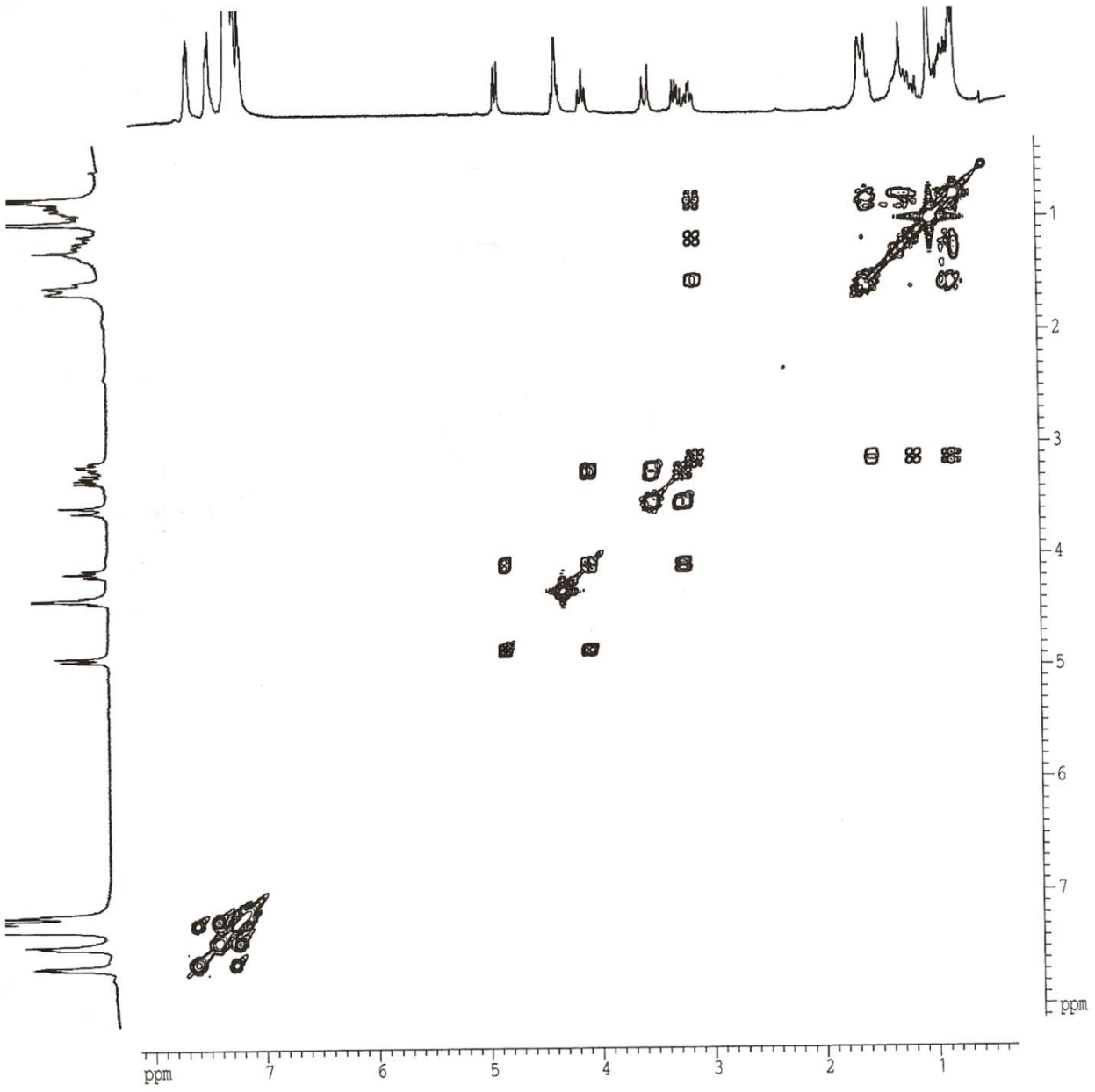
2D NMR plot parameters

CX2	16.00 cm
CX1	16.00 cm
F2PLO	8.133 ppm
F2LO	2440.87 Hz
F2PHI	0.311 ppm
F2HI	93.45 Hz
F1PLO	8.133 ppm
F1LO	2440.91 Hz
F1PHI	0.312 ppm
F1HI	93.49 Hz
F2PPMCM	0.48883 ppm/cm
F2HZCM	146.71361 Hz/cm
F1PPMCM	0.48883 ppm/cm
F1HZCM	146.71332 Hz/cm



12h

^{1}H NOESY contacts



Current Data Parameters

E	pm110399
NO	3
CNO	1

F2 - Acquisition Parameters

Date	990227
Time	0.58
PULPROG	cosy90
SOLVENT	CDCl3
AQ	0.1090760 sec
FIDRES	4.584800 Hz
DW	213.0 usec
RG	715
NUCLEUS	1H
D1	1.0000000 sec
P1	7.1 usec
D0	0.0000030 sec
DE	266.3 usec
SFO1	300.1346352 MHz
SWH	2347.42 Hz
TD	512
NS	8
DS	2
IN0	0.0004260 sec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.1346 MHz
FIDRES	9.169582 Hz
SW	7.821 ppm

F1 - Processing parameters

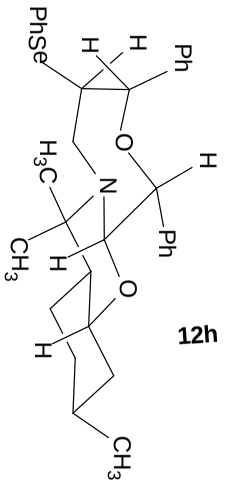
SI	512
MC2	QF
SF	300.1333680 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	QF
SF	300.1333680 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

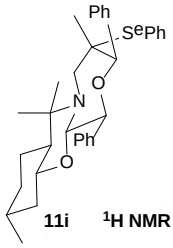
2D NMR plot parameters

CX2	16.00 cm
CX1	16.00 cm
F2PLO	8.133 ppm
F2LO	2440.87 Hz
F2PHI	0.311 ppm
F2HI	93.45 Hz
F1PLO	8.133 ppm
F1LO	2440.91 Hz
F1PHI	0.312 ppm
F1HI	93.49 Hz
F2PPMCM	0.48883 ppm/cm
F2HZCM	146.71361 Hz/cm
F1PPMCM	0.48883 ppm/cm
F1HZCM	146.71332 Hz/cm

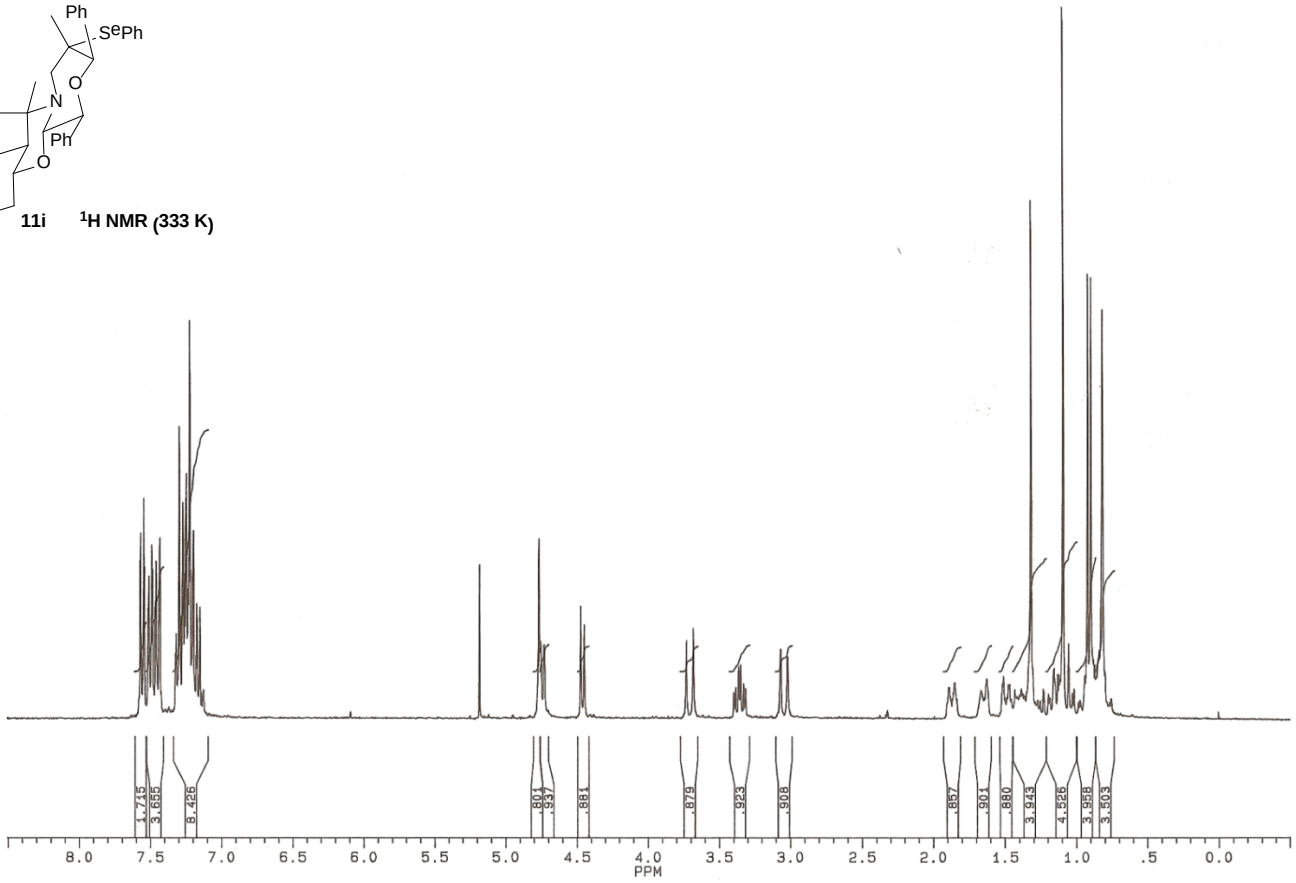


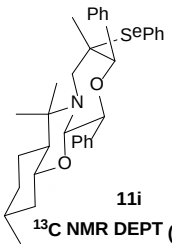
12h

COSY



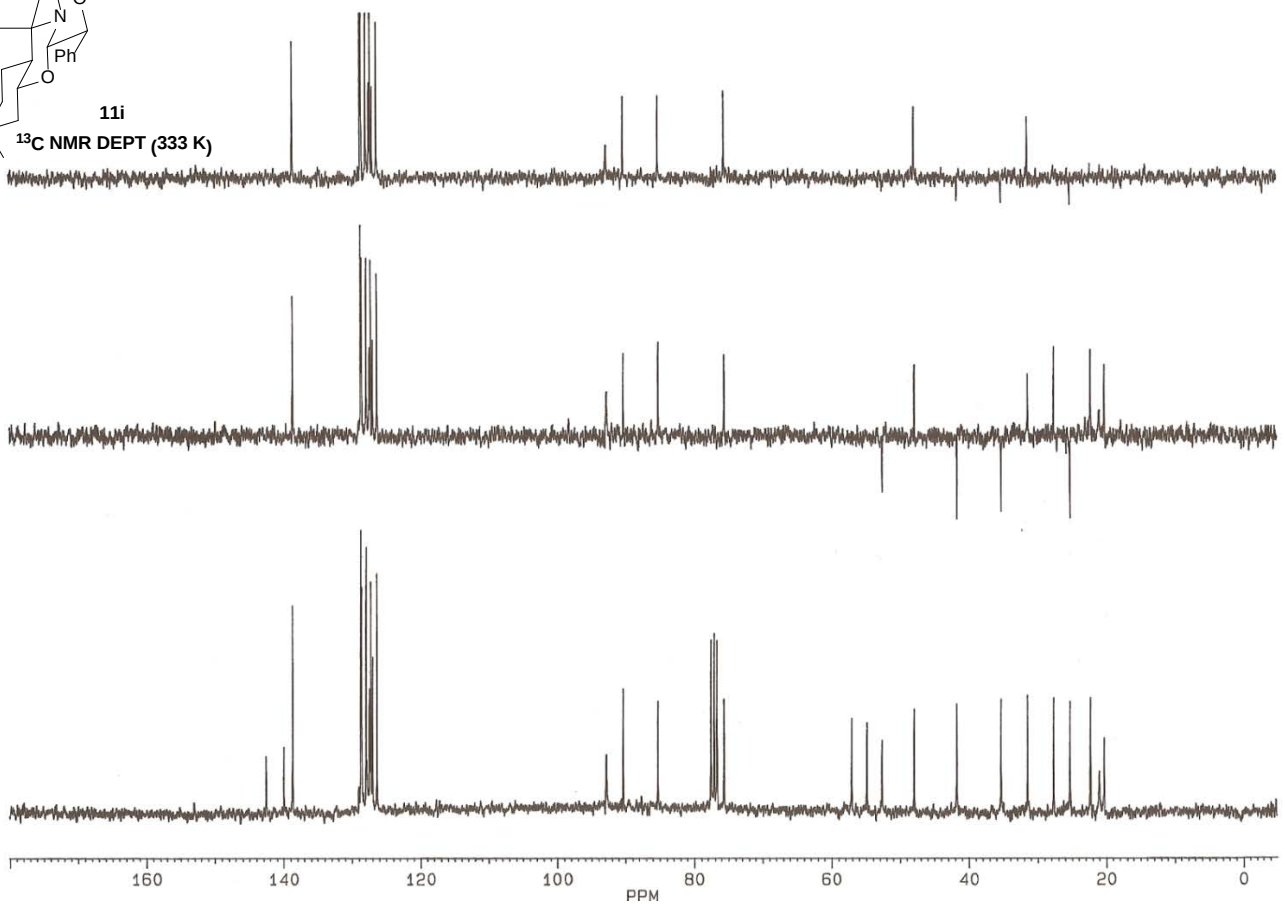
11i **^{1}H NMR (333 K)**

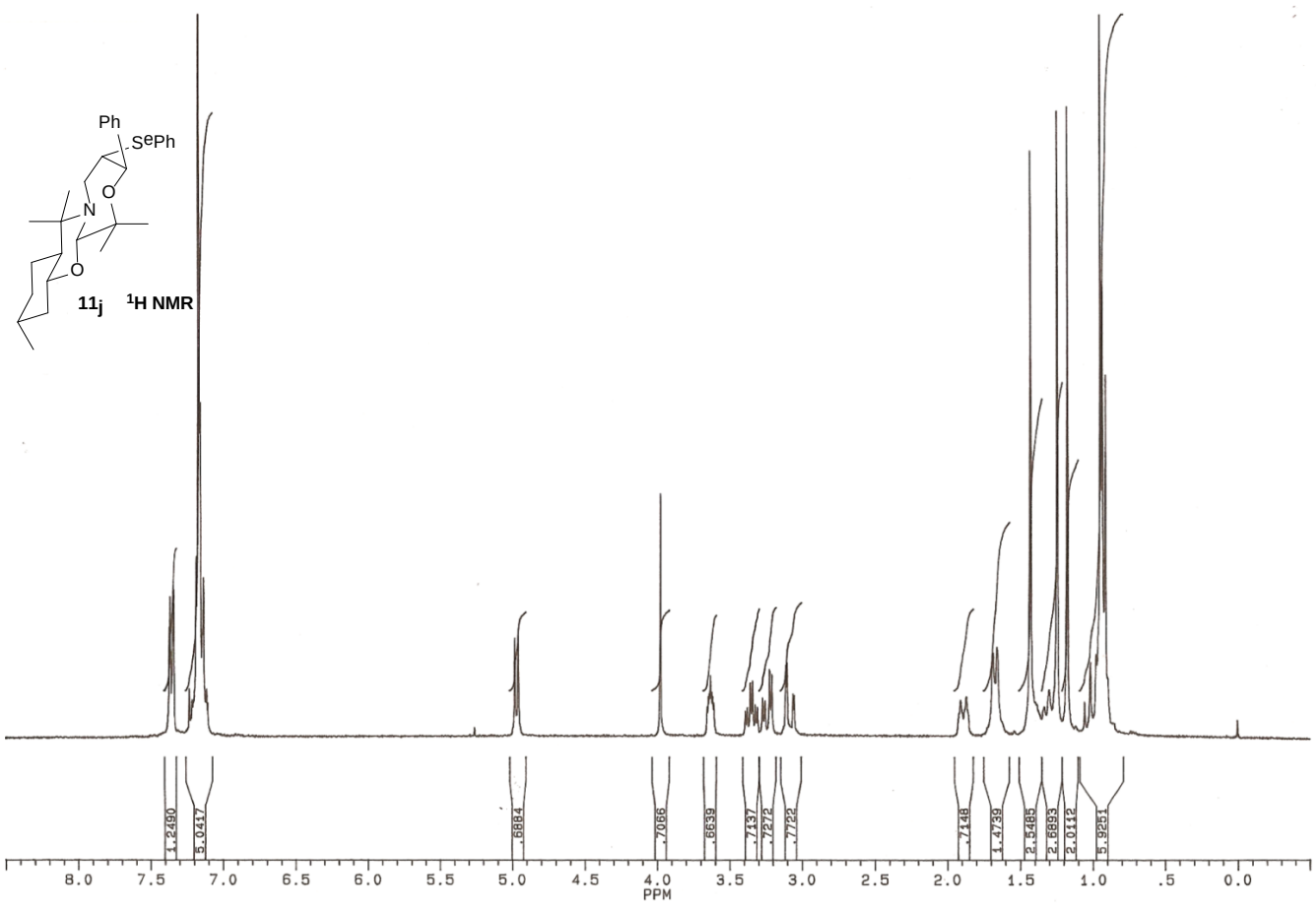


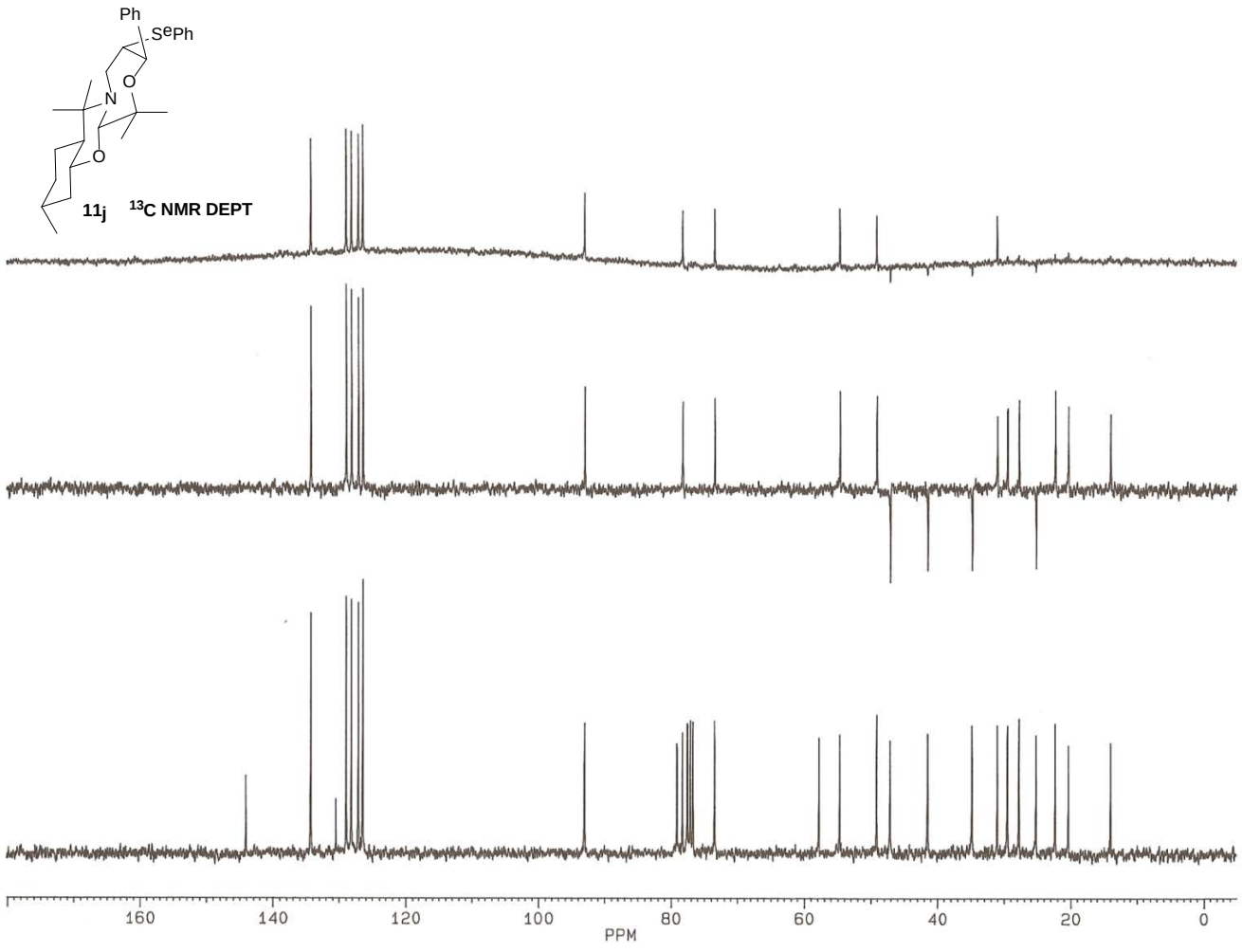


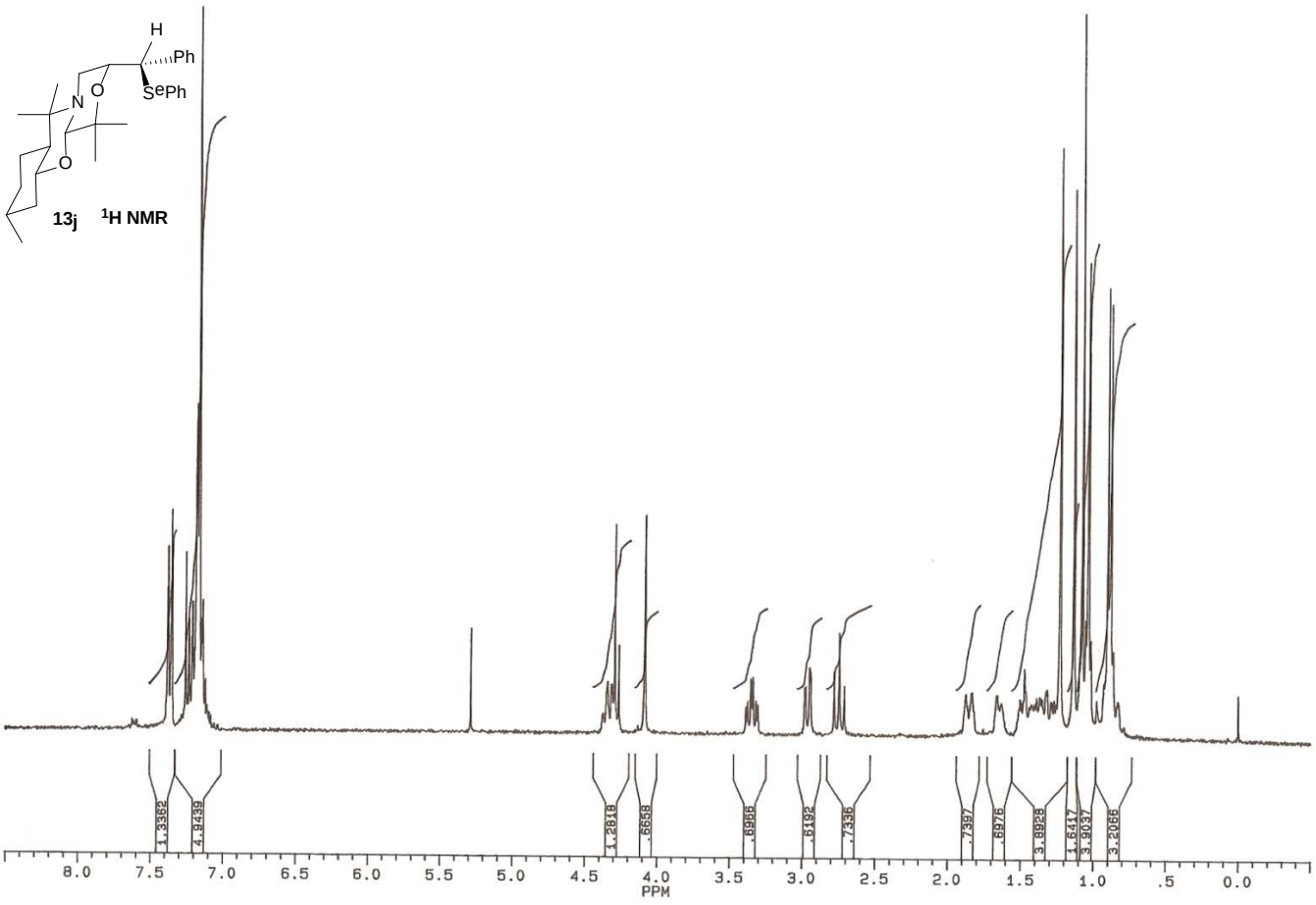
11i

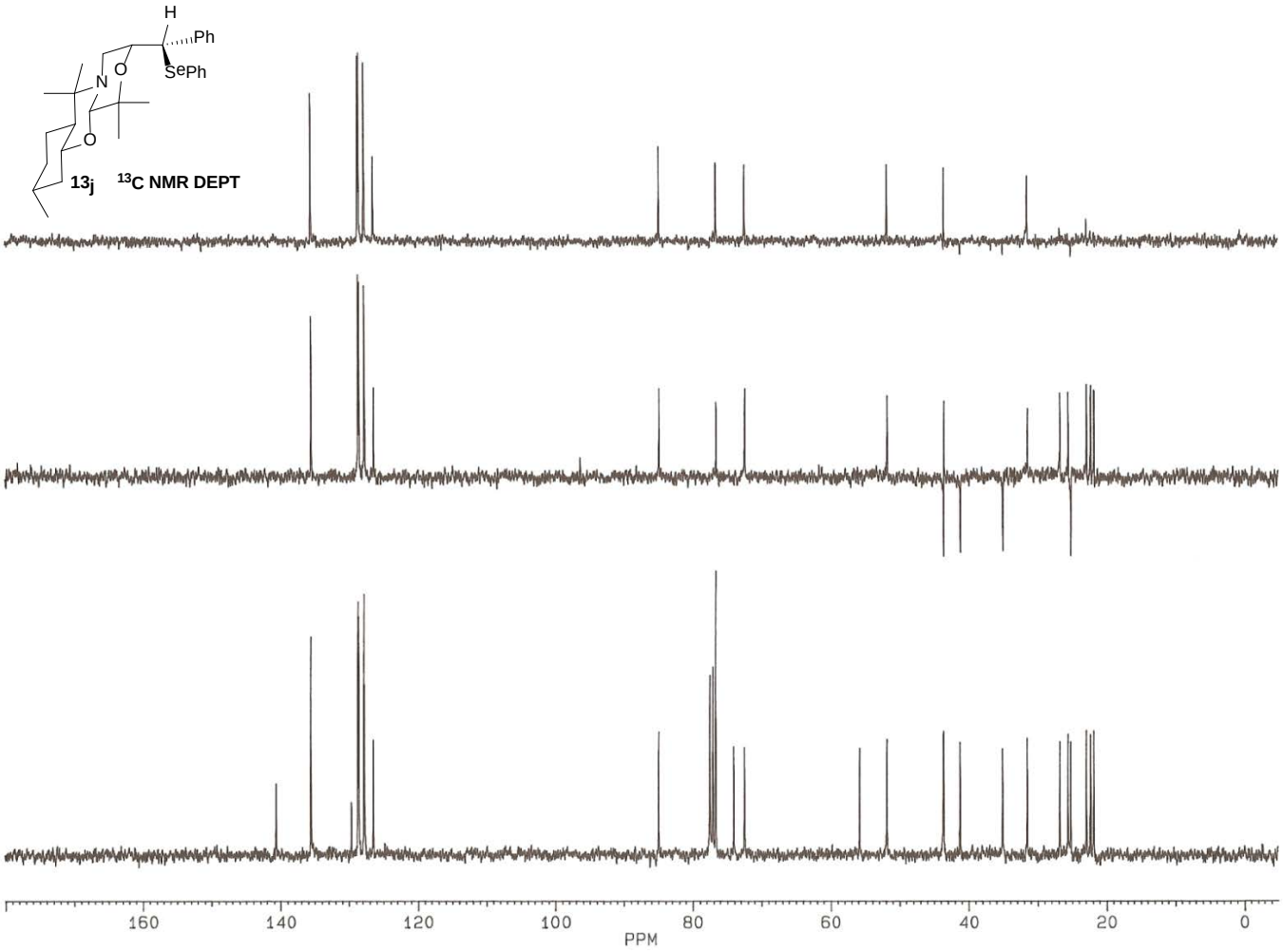
^{13}C NMR DEPT (333 K)

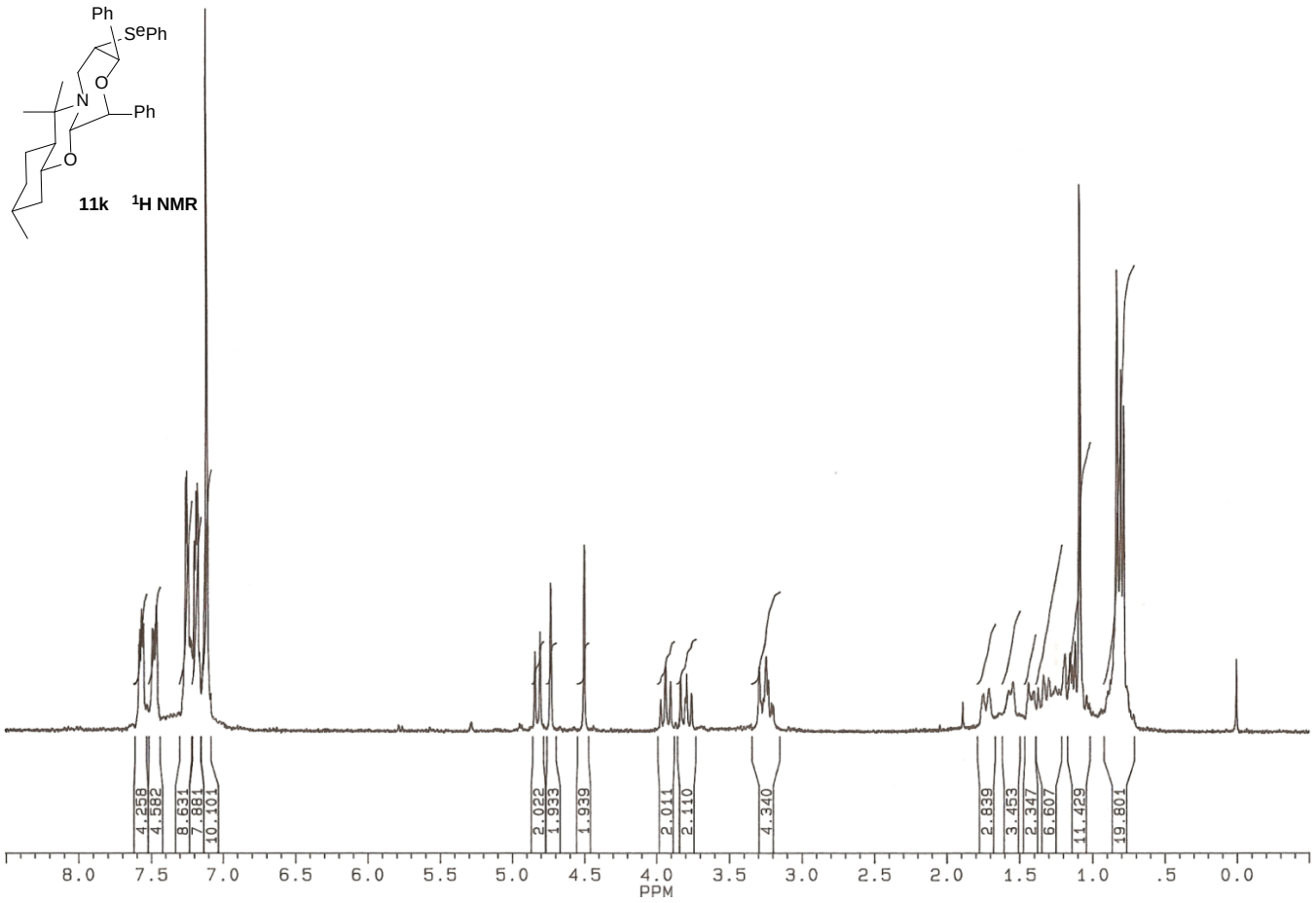


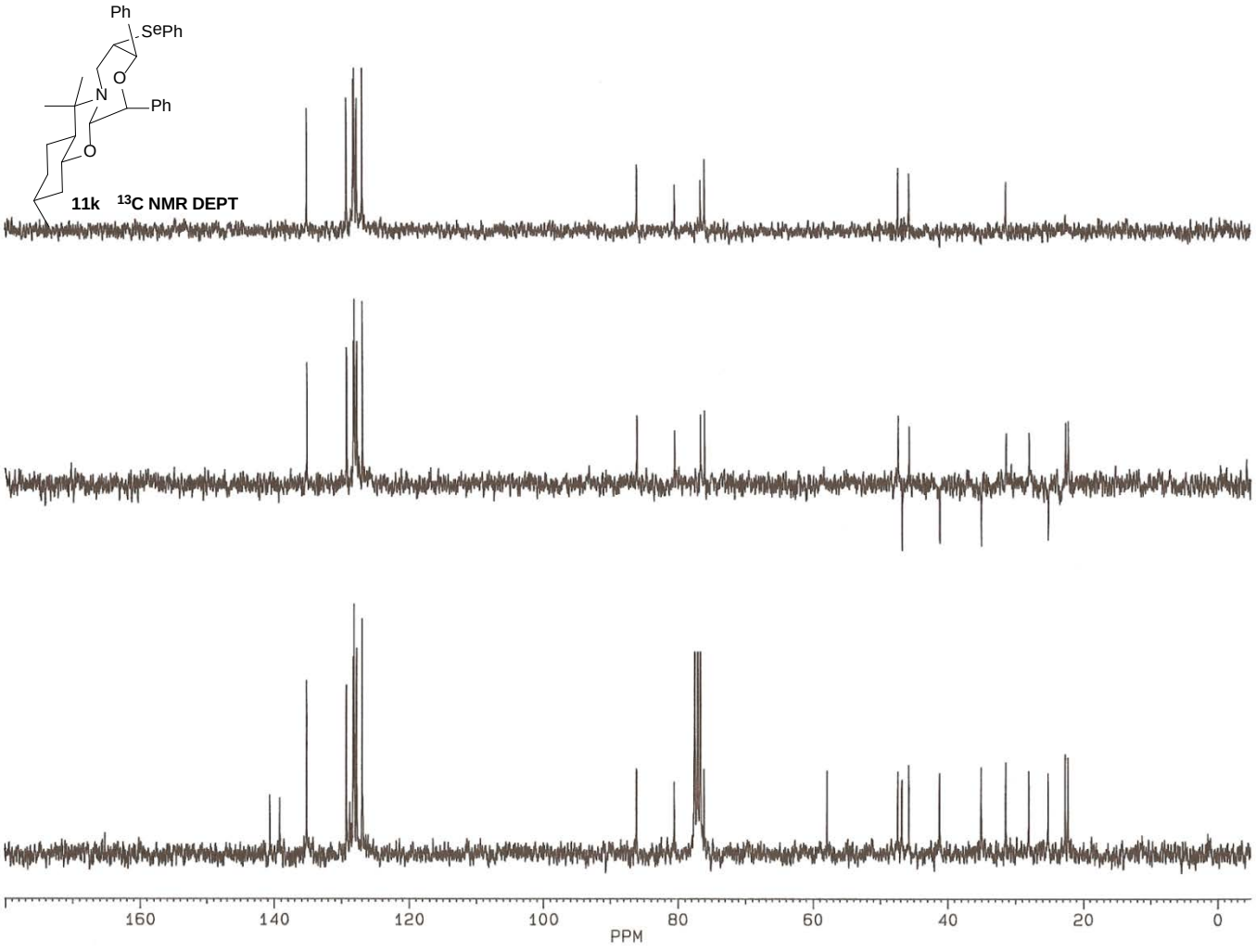

Ph
SePh
N
O
O
11j 1H NMR
1.2490
5.0417
.6884
.7066
.6639
.7137
.7272
.7782
.7148
1.4739
2.5485
2.8893
2.0112
5.9251
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM

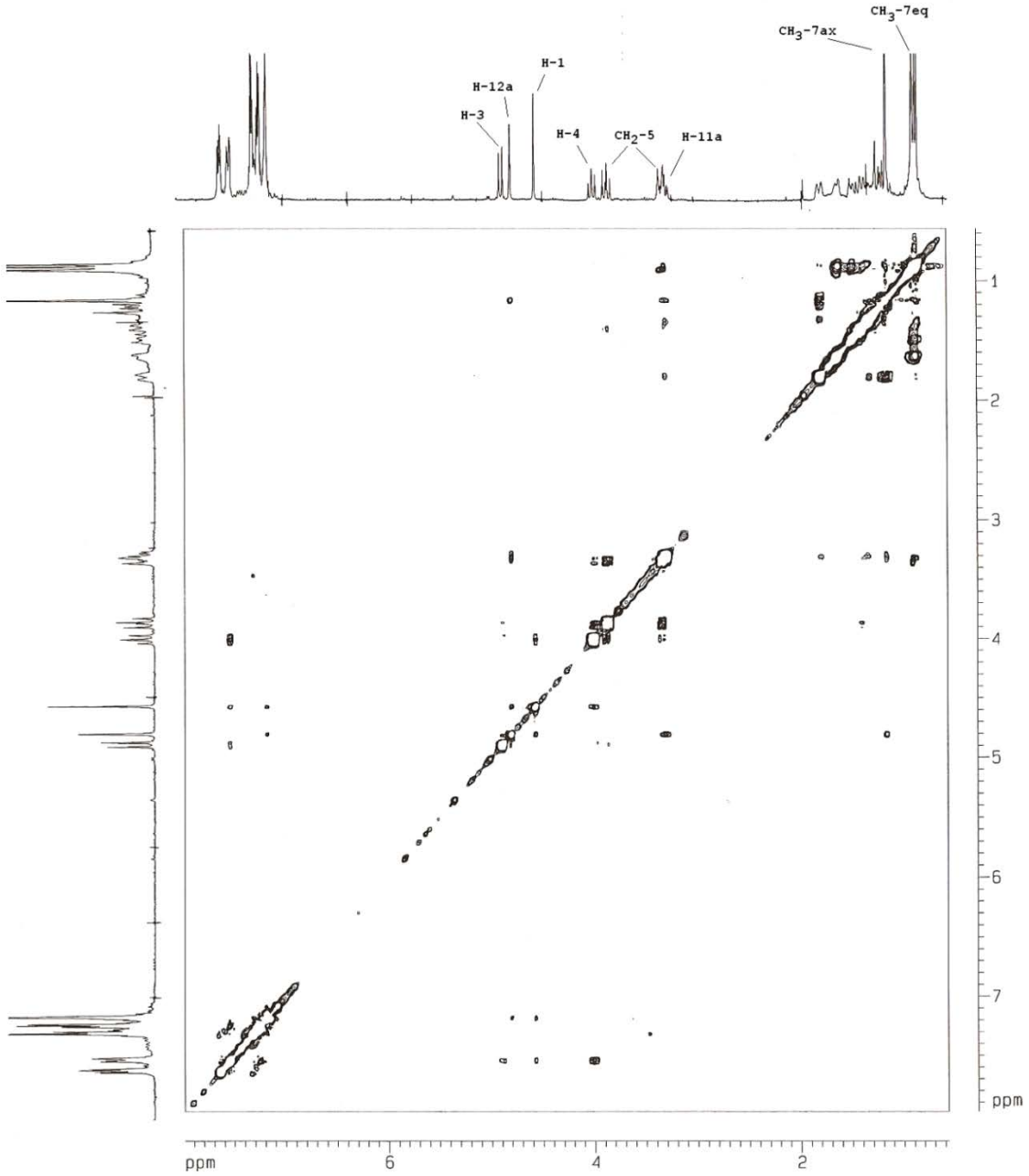

Ph
SePh
N
O
O
11j 13C NMR DEPT
160 140 120 100 80 60 40 20 0
PPM


H
Ph
SePh
N
O
O
13j
1H NMR
1.3362
4.9439
1.2818
.6858
.6966
.6192
.7336
.7397
.6976
3.8928
1.8417
3.9037
3.2066
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
.5
0.0
PPM

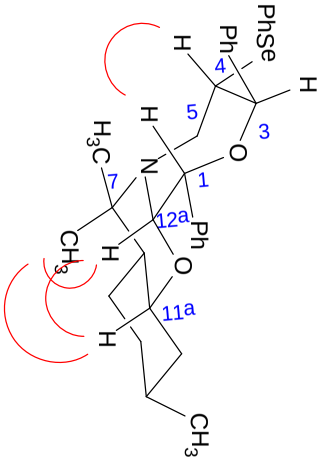

H
Ph
SePh
N
O
O
13j
13C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM


Ph
SePh
O
N
Ph
O
11k ¹H NMR
4.258
4.582
8.631
7.881
10.101
2.022
1.933
1.939
2.011
2.110
4.340
2.839
3.453
2.347
6.607
11.429
19.801
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
.5
0.0
PPM


Ph
SePh
O
N
Ph
O
11k ¹³C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM

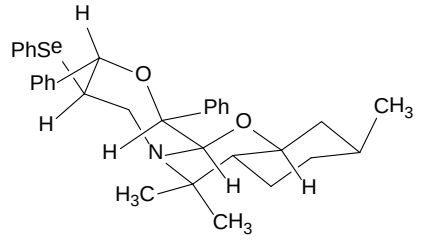



```
Current Data Parameters
NAME            JN-190106
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_         20060121
Time              1.16
INSTRUM           spect
PROBHD     5 mm TBI 1H-
PULPROG         noesygs
TD                1024
SOLVENT           CDCl3
NS                   4
DS                   4
SWH           3496.503 Hz
FIDRES        3.414554 Hz
AQ            0.146820 sec
RG                1024
DW             143.000 usec
DE              204.29 usec
TE              300.0 K
P15            1000.00 usec
L21                100
DB          0.50000000 sec
D15         0.00010000 sec
D1          1.00000000 sec
P1             9.00 usec
D0          0.0000030 sec
D20         0.2489000 sec
D27         0.0000080 sec
P2            18.00 usec
SF01       300.1349872 MHz
NUCLEUS           1H
IN0         0.00014300 sec

F1 - Acquisition parameters
ND0                   2
TD                  256
SF01          300.135 MHz
FIDRES      13.658253 Hz
SW             11.650 ppm

F2 - Processing parameters
SI                 1024
SF         300.1333678 MHz
WDW              QSINE
SSB                   2
LB             0.00 Hz
GB                    0
PC             1.00

F1 - Processing parameters
SI                 1024
MC2                TPPI
SF         300.1333678 MHz
WDW              QSINE
SSB                   2
LB             0.00 Hz
GB                    0

2D NMR plot parameters
CX2              13.00 cm
CX1              15.00 cm
F2PLO             7.990 ppm
F2LO           2397.92 Hz
F2PHI             0.572 ppm
F2HI            171.63 Hz
F1PLO             7.978 ppm
F1LO           2394.50 Hz
F1PHI             0.560 ppm
F1HI            168.21 Hz
F2PPMCM       0.57059 ppm/cm
F2HZCM      171.25302 Hz/cm
F1PPMCM       0.49451 ppm/cm
F1HZCM      148.41968 Hz/cm
```

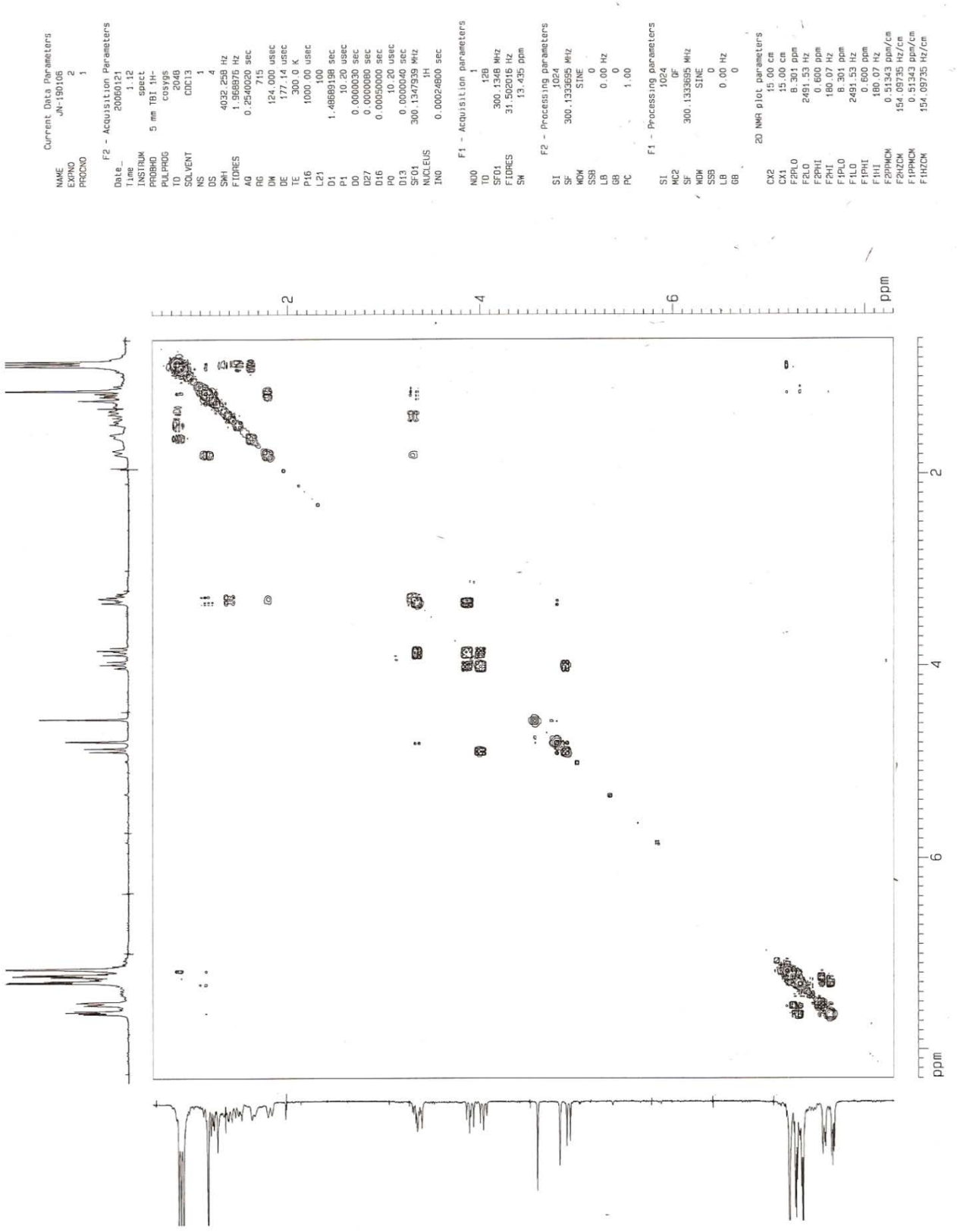


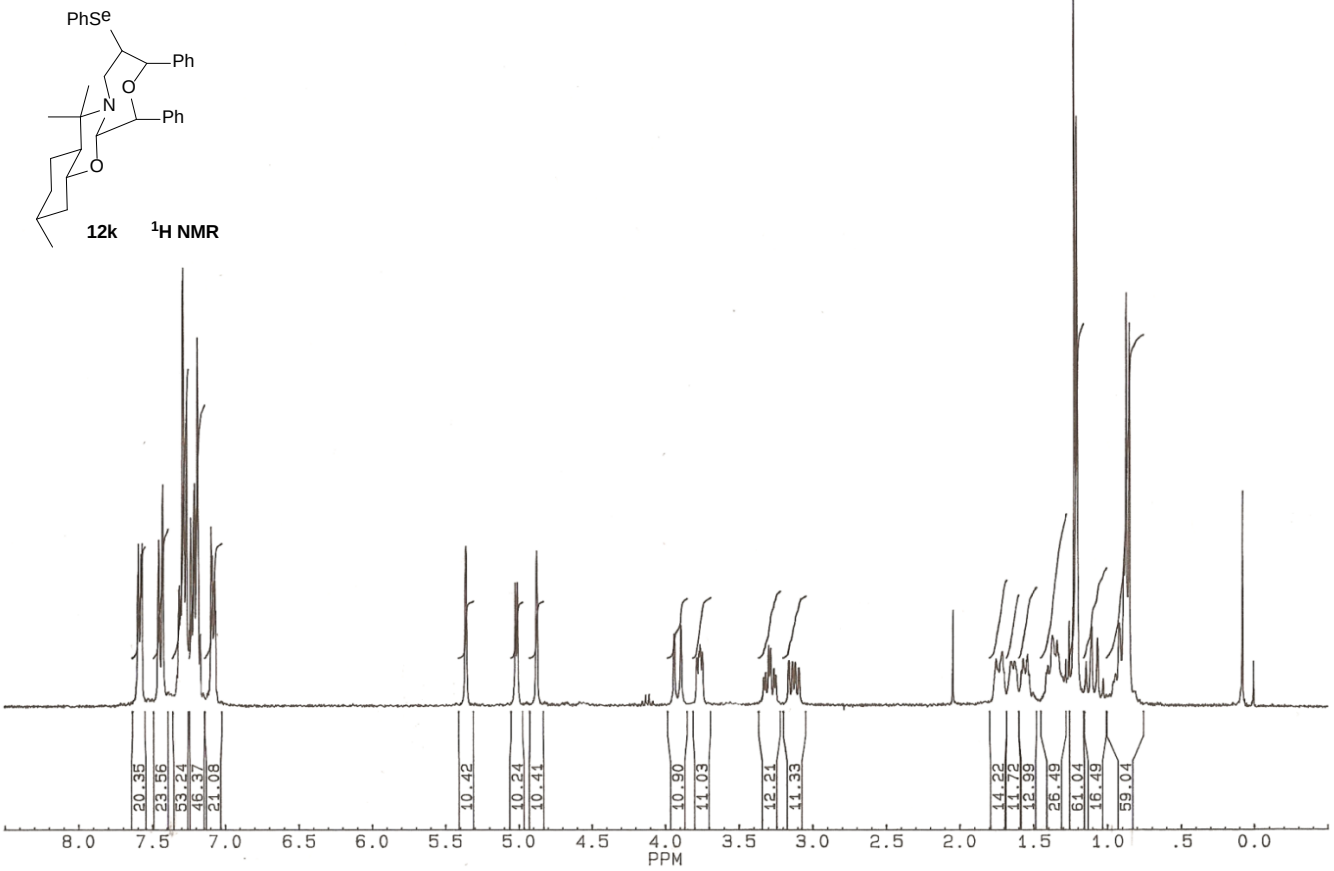
11k

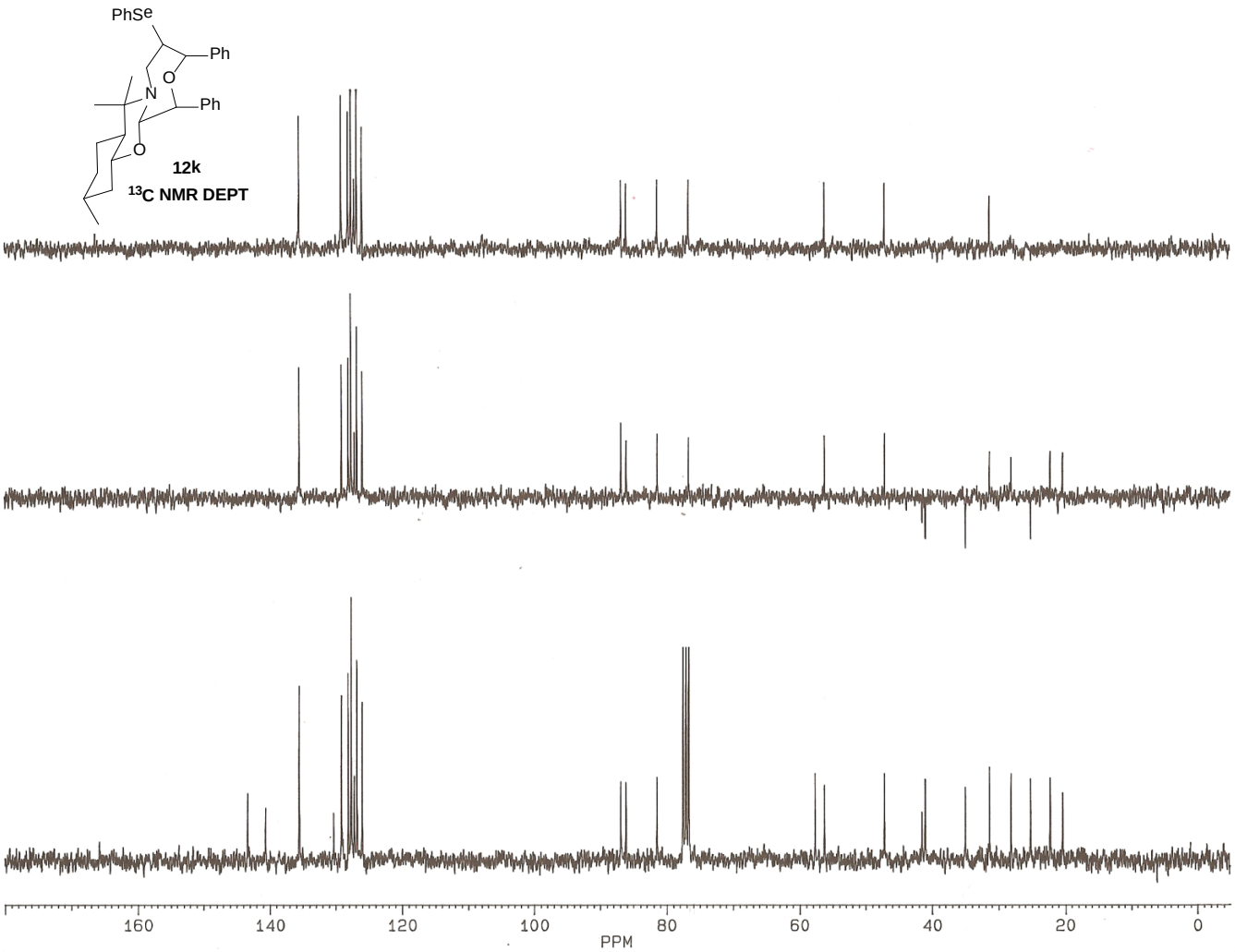
^{1}H NOESY contacts

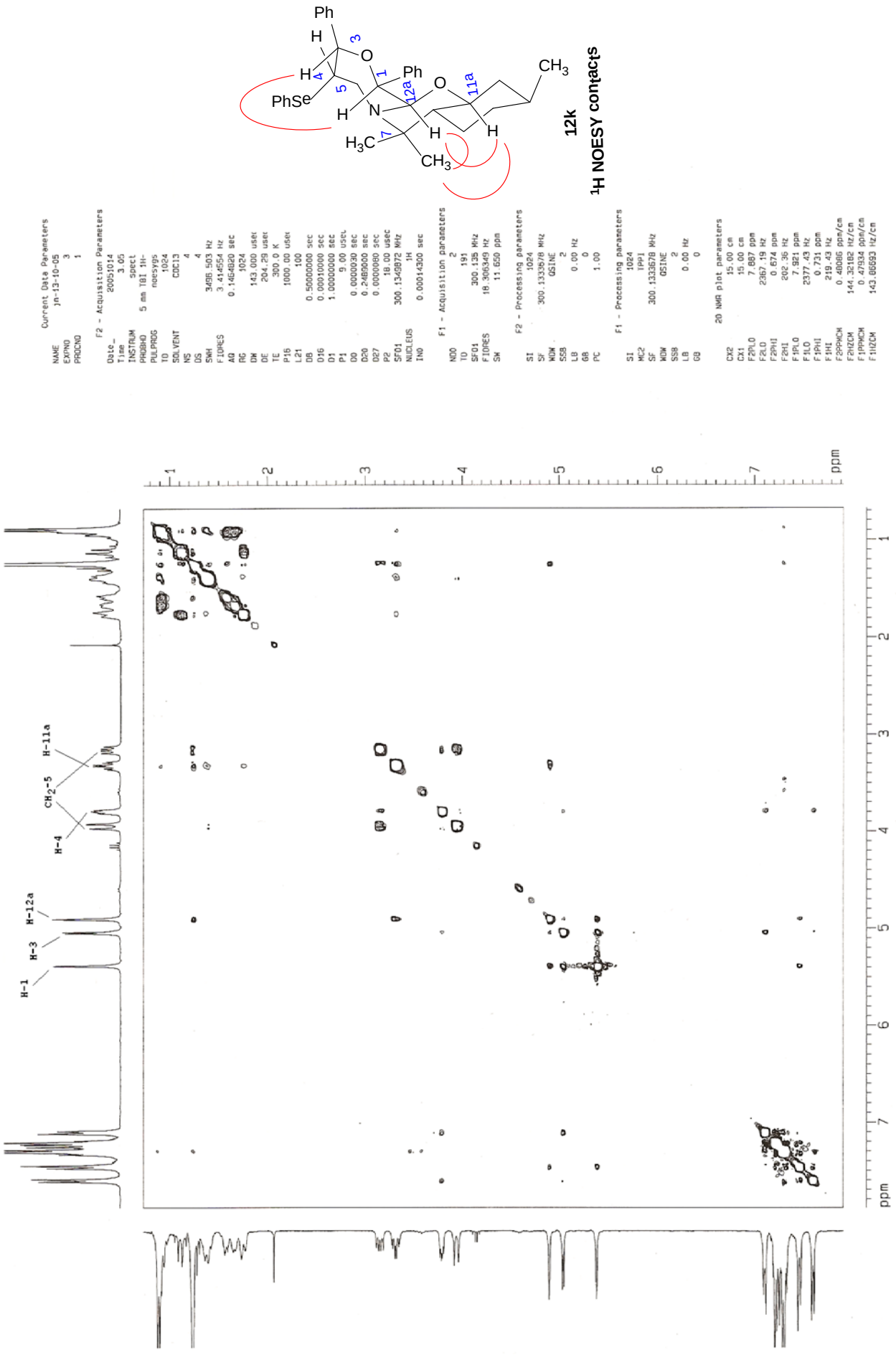

H
PhSe
Ph
O
H
Ph
O
CH3
H
N
H
H
H3C
CH3

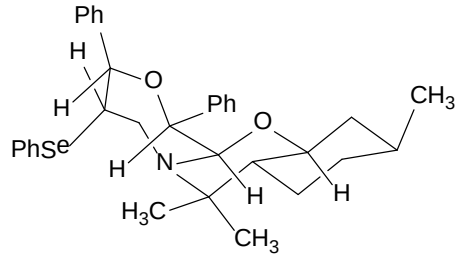
11k COSY


Current Data Parameters
NAME JN-190106
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060121
Time 1.12
INSTRUM spect
PROBHD 5 mm TBI 1H-
PULPROG cosygs
TD 2048
SOLVENT CDCl3
NS 1
DS 4
SWH 4032.258 Hz
FIDRES 1.968875 Hz
AQ 0.2540020 sec
RG 715
DW 124.000 usec
DE 177.14 usec
TE 300.0 K
P16 1000.00 usec
L21 100
D1 1.48689198 sec
P1 10.20 usec
D0 0.0000030 sec
D27 0.0000080 sec
D16 0.00050000 sec
PQ 10.20 usec
D13 0.0000040 sec
SF01 300.1347939 MHz
NUCLEUS 1H
IN0 0.00024800 sec
F1 - Acquisition parameters
NUO 1
TD 128
SF01 300.1348 MHz
FIDRES 31.502016 Hz
SW 13.435 ppm
F2 - Processing parameters
SI 1024
SF 300.1333695 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1333695 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 8.301 ppm
F2LO 2491.53 Hz
F2PHI 0.600 ppm
F2HI 180.07 Hz
F1PLO 8.301 ppm
F1LO 2491.53 Hz
F1PHI 0.600 ppm
F1HI 180.07 Hz
F2PPMCM 0.51343 ppm/cm
F2HZCM 154.09735 Hz/cm
F1PPMCM 0.51343 ppm/cm
F1HZCM 154.09735 Hz/cm
2
4
6
ppm
2
4
6
ppm


PhSe
Ph
O
N
Ph
O
12k
1H NMR
20.35
23.56
53.24
46.37
21.08
10.42
10.24
10.41
10.90
11.03
12.21
11.33
14.22
11.72
12.99
26.49
61.04
16.49
59.04
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
.5
0.0
PPM

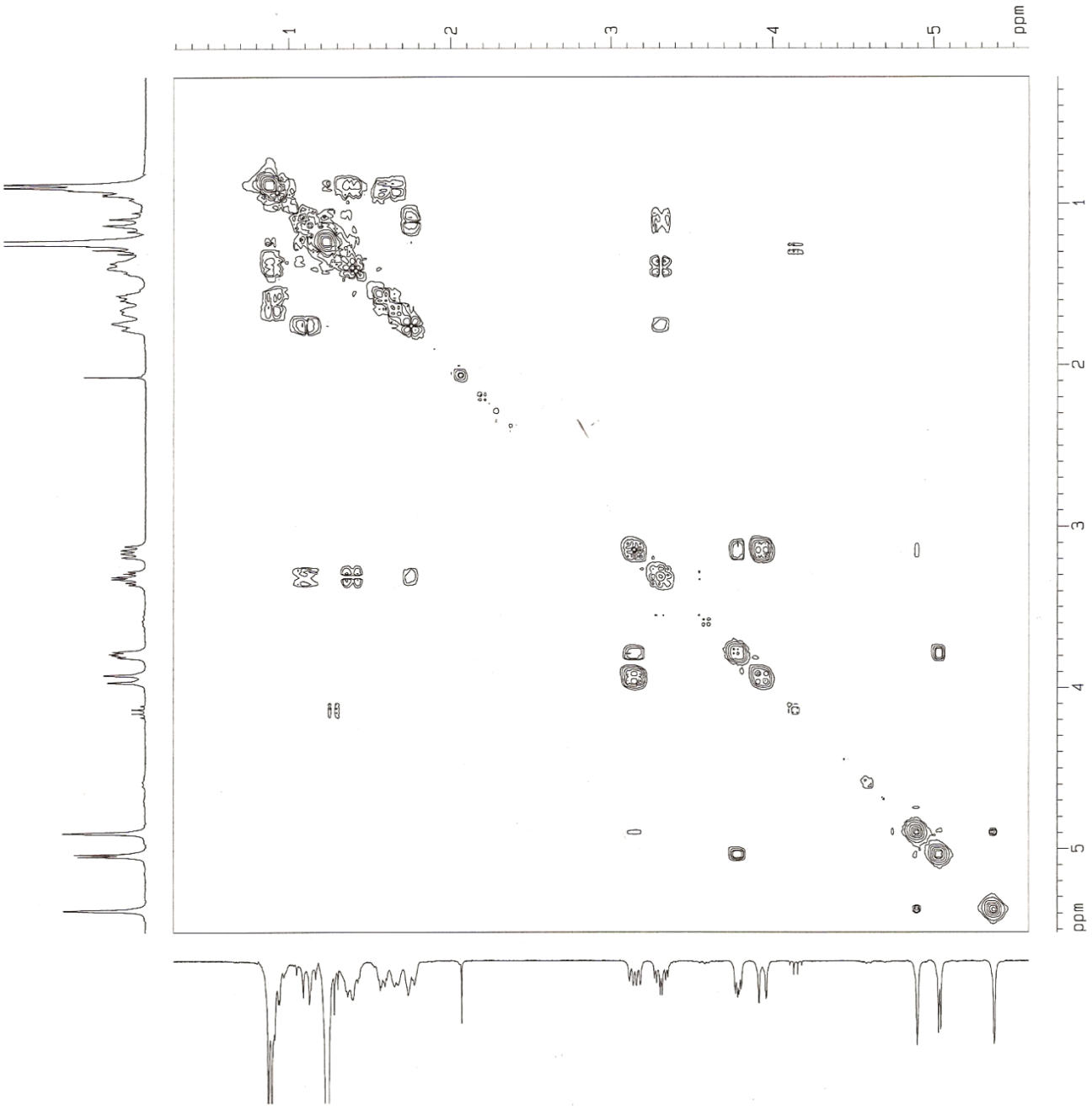

PhSe
Ph
O
N
Ph
O
12k
13C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM


Ph
H
3
O
H
4
1
Ph
5
12a
O
11a
CH3
PhSe
H
N
H
H
H3C
7
CH3
12k
1H NOESY conjacts
H-1
H-3
H-12a
H-4
CH2-5
H-11a
ppm
ppm

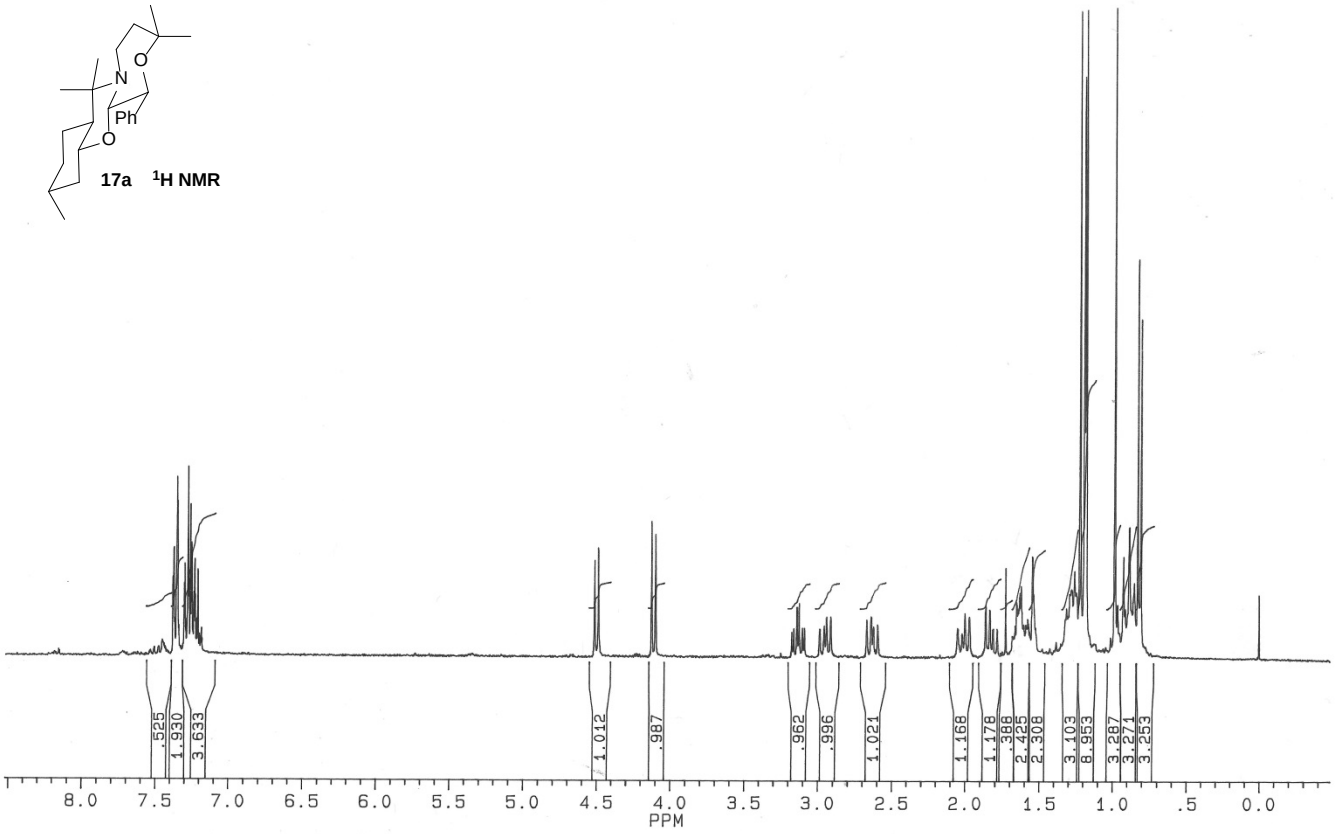


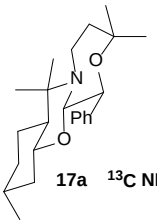
12k COSY

```
Current Data Parameters
NAME          jn-13-10-05
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20051014
Time                3.27
INSTRUM             spect
PROBHD   5 mm TBI 1H-
PULPROG           cosygs
TD                  2048
SOLVENT            CDCl3
NS                     1
DS                     4
SWH             4032.258 Hz
FIDRES          1.968876 Hz
AQ              0.2540020 sec
RG                   715
DW                124.000 usec
DE                177.14 usec
TE                300.0 K
P16              1000.00 usec
L21                  100
D1              1.48689198 sec
P1                10.20 usec
D0              0.0000030 sec
D27             0.0000080 sec
D16             0.00050000 sec
PO                10.20 usec
D13             0.0000040 sec
SF01            300.1347939 MHz
NUCLEUS               1H
INO             0.00024800 sec

F1 - Acquisition parameters
NDO                    1
TD                   128
SF01          300.1348 MHz
FIDRES         31.502016 Hz
SW             13.435 ppm

F2 - Processing parameters
SI                  1024
SF            300.1333695 MHz
WDW                 SINE
SSB                    0
LB              0.00 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF            300.1333695 MHz
WDW                 SINE
SSB                    0
LB              0.00 Hz
GB                     0

2D NMR plot parameters
CX2                15.00 cm
CX1                15.00 cm
F2PLO           5.520 ppm
F2LO            1656.73 Hz
F2PHI           0.219 ppm
F2HI            65.87 Hz
F1PLO           5.599 ppm
F1LO            1680.35 Hz
F1PHI           0.285 ppm
F1HI            85.56 Hz
F2PPMCM         0.35337 ppm/cm
F2HZCM          106.05679 Hz/cm
F1PPMCM         0.35424 ppm/cm
F1HZCM          106.31931 Hz/cm
```

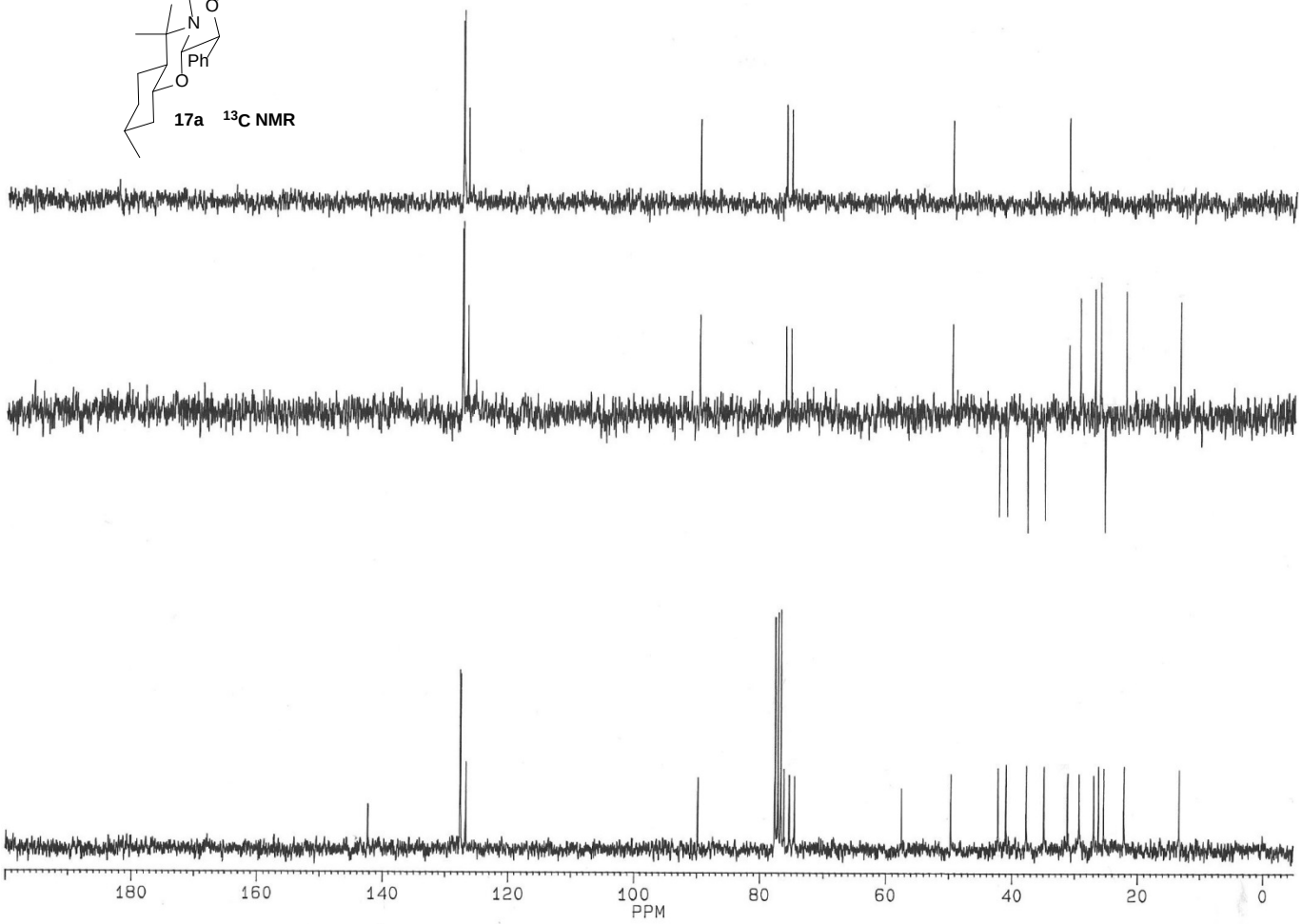


17a ^{1}**H NMR**

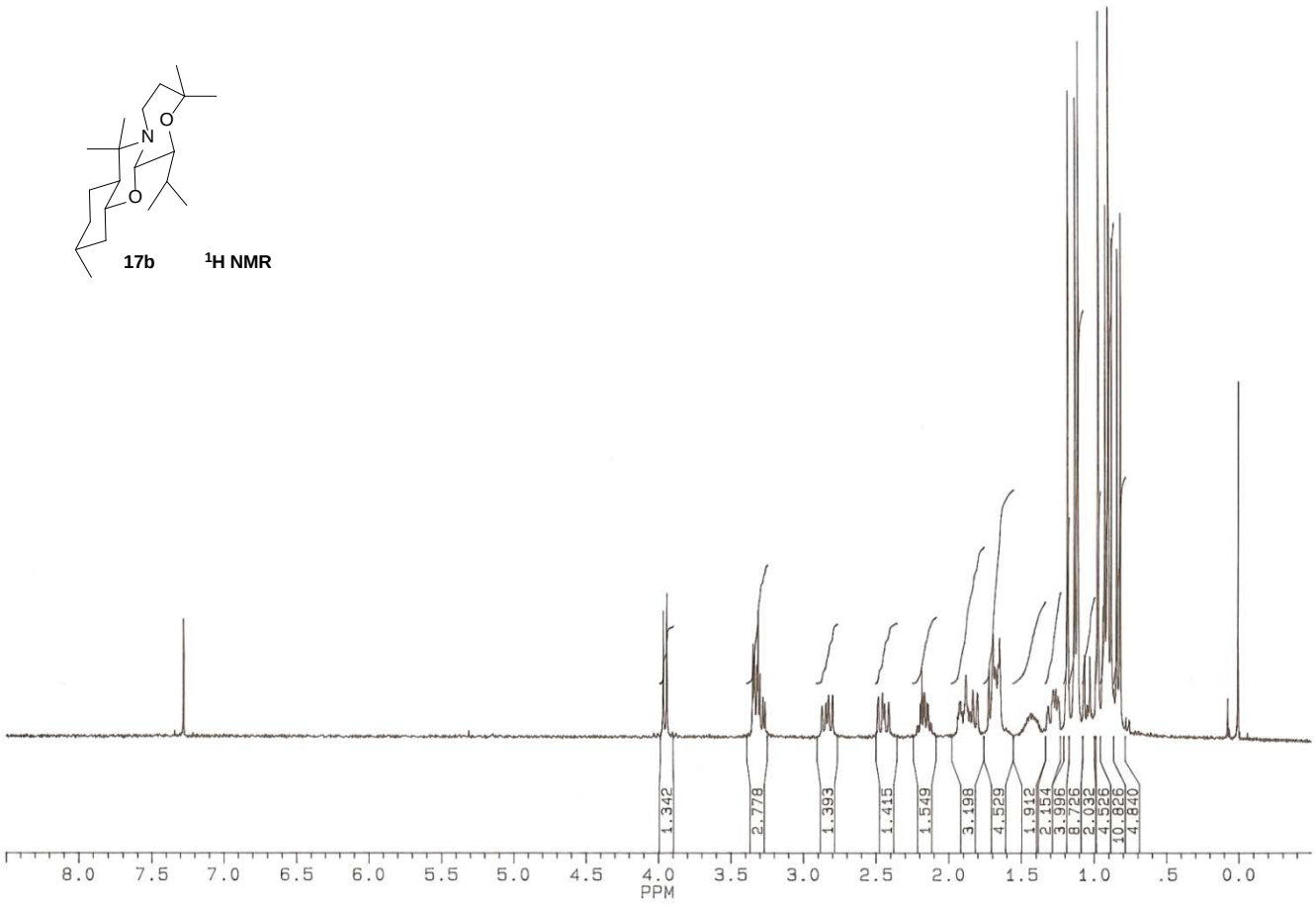


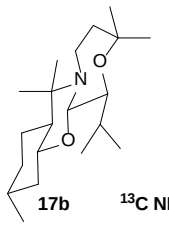


17a ^{13}**C NMR**

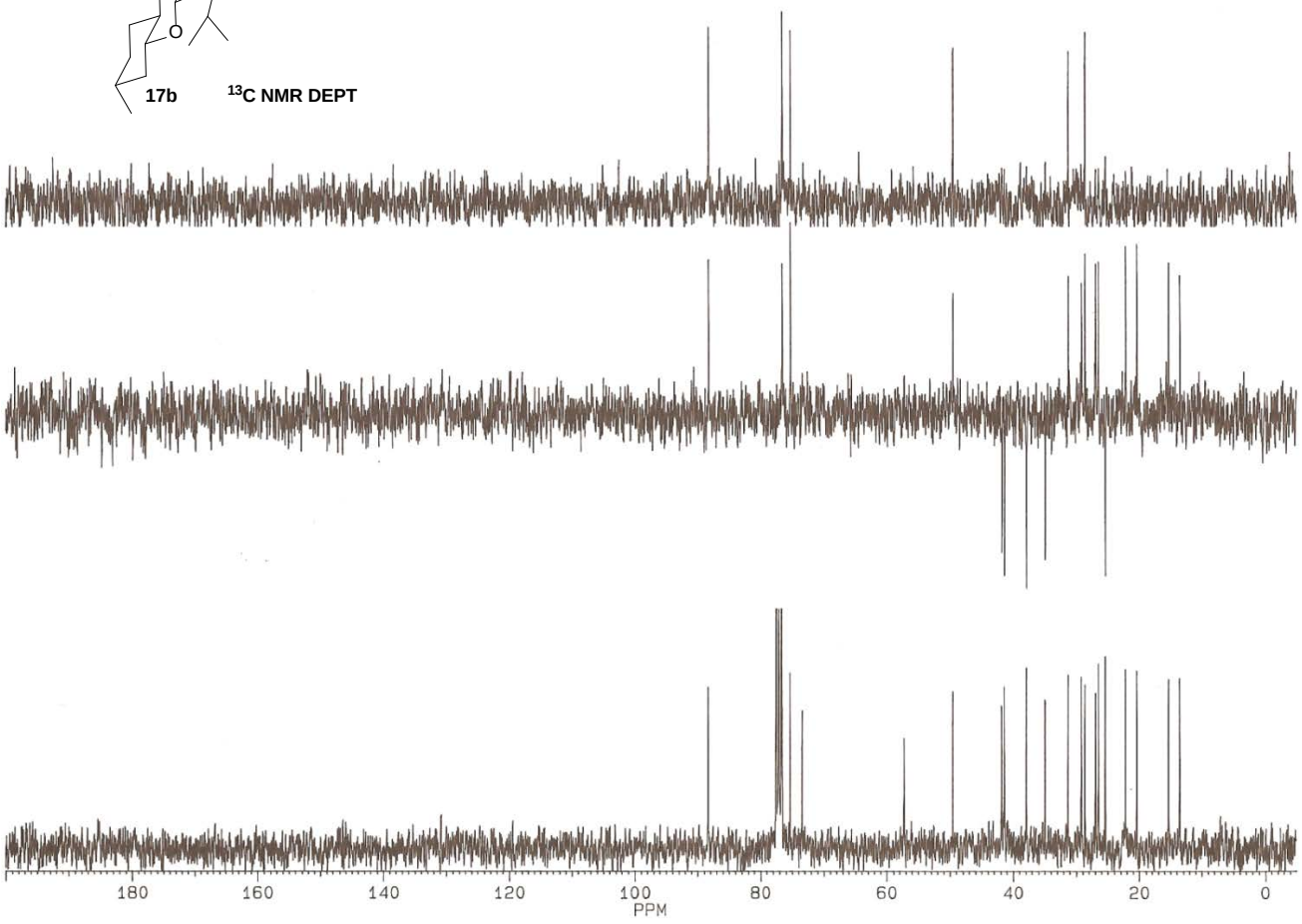


17b **^{1}H NMR**

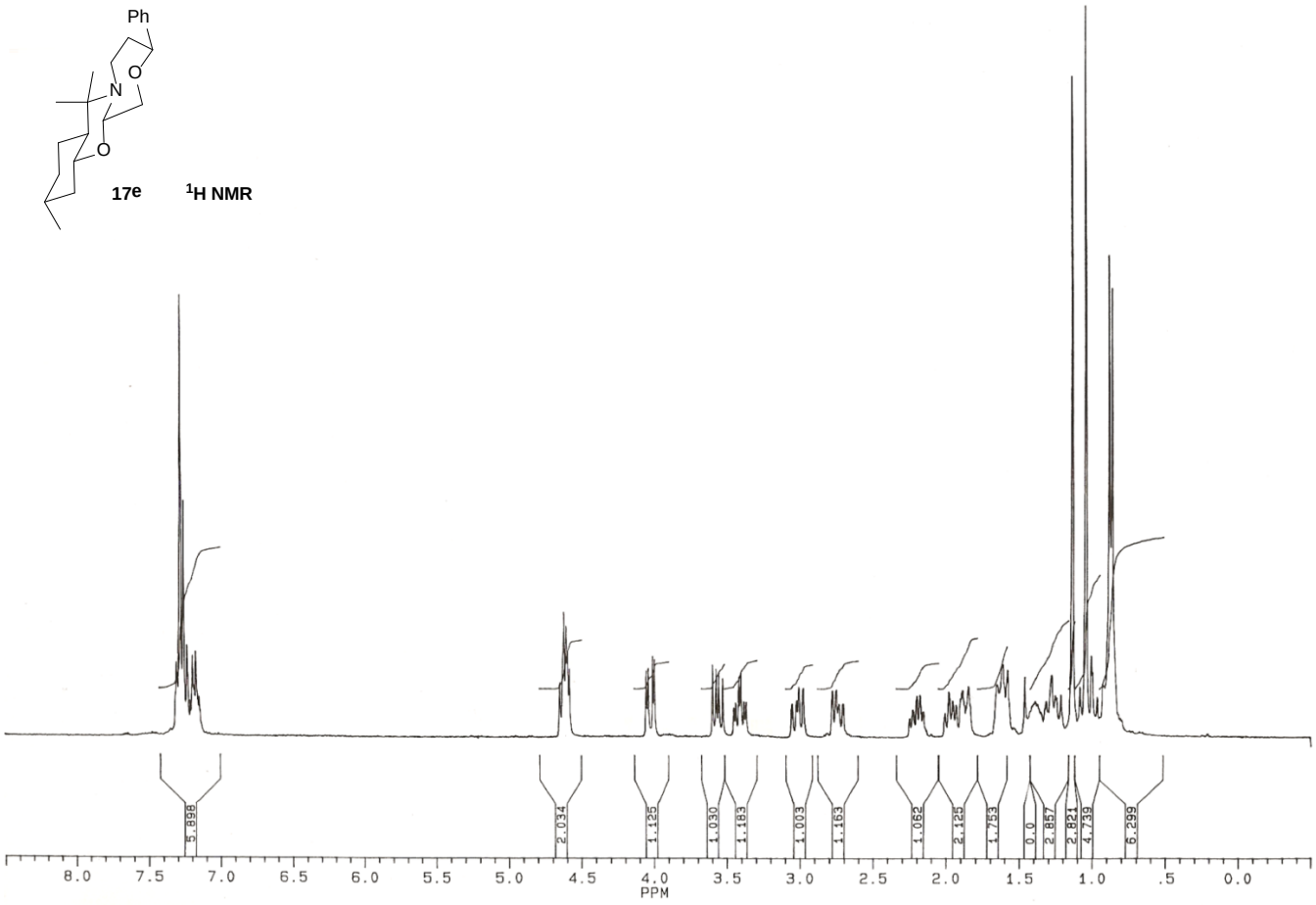


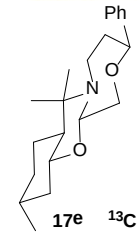


17b **^{13}C NMR DEPT**

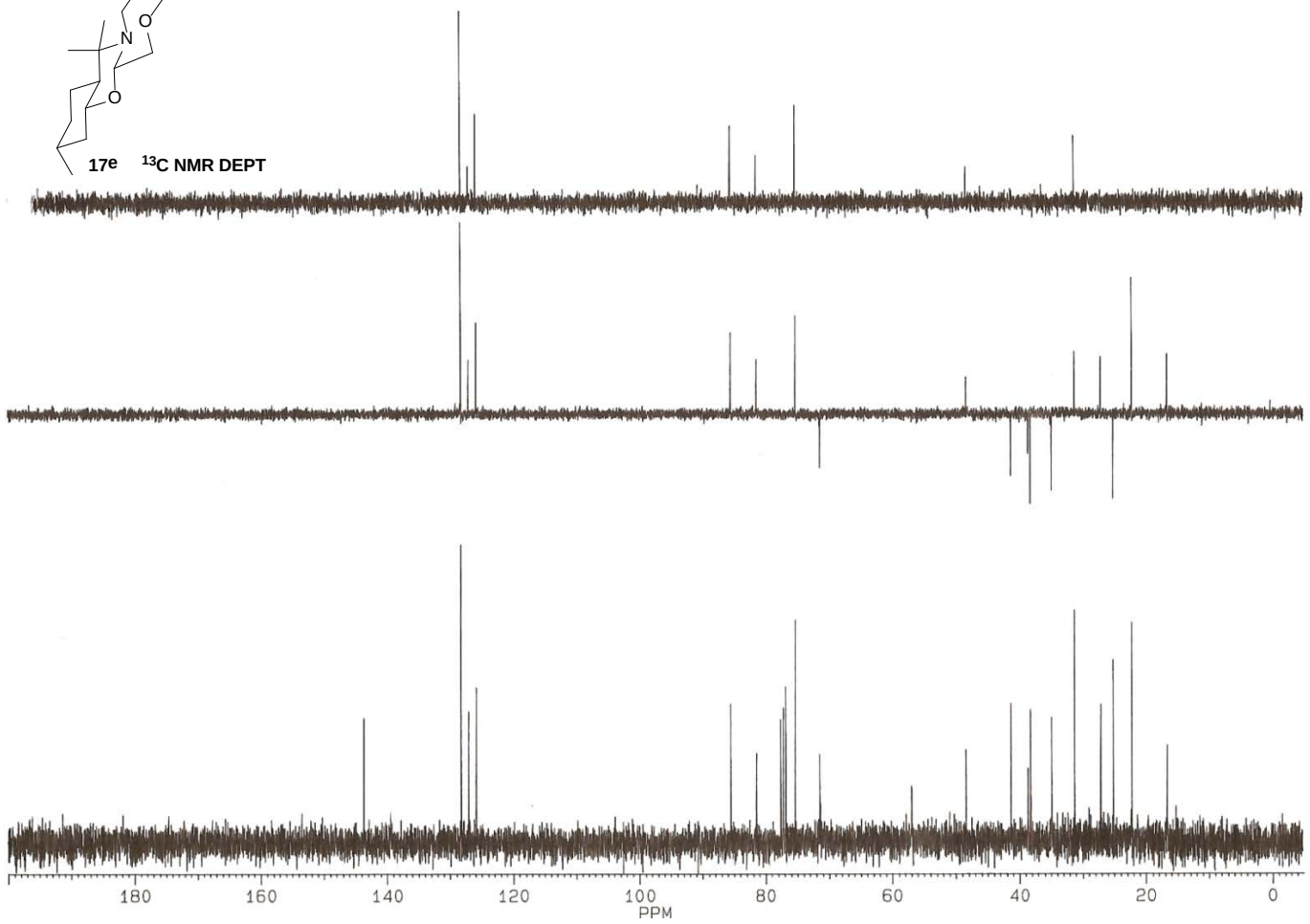


17e **^{1}H NMR**

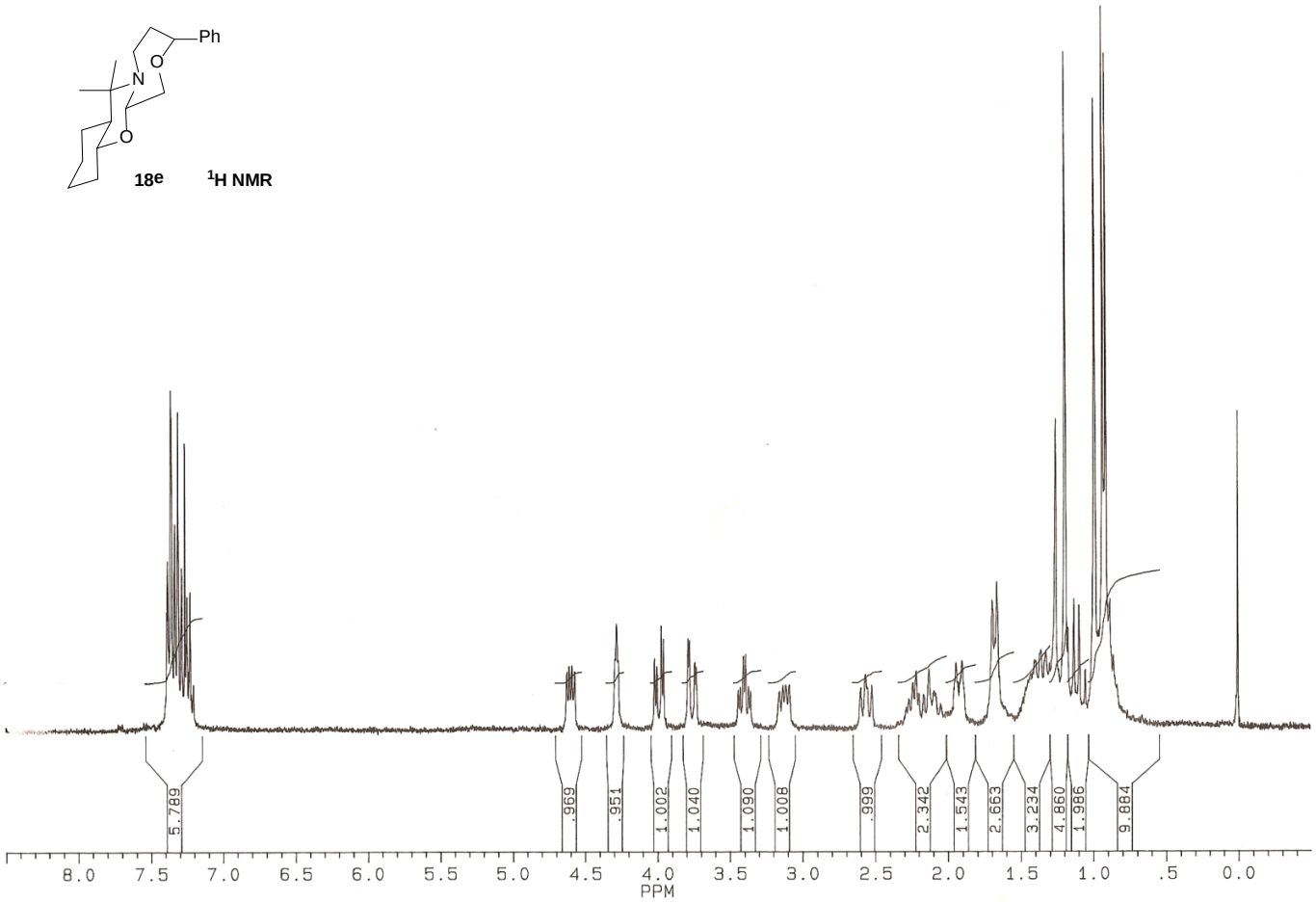


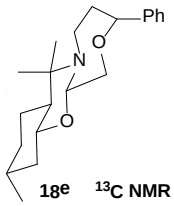


17e **^{13}C NMR DEPT**

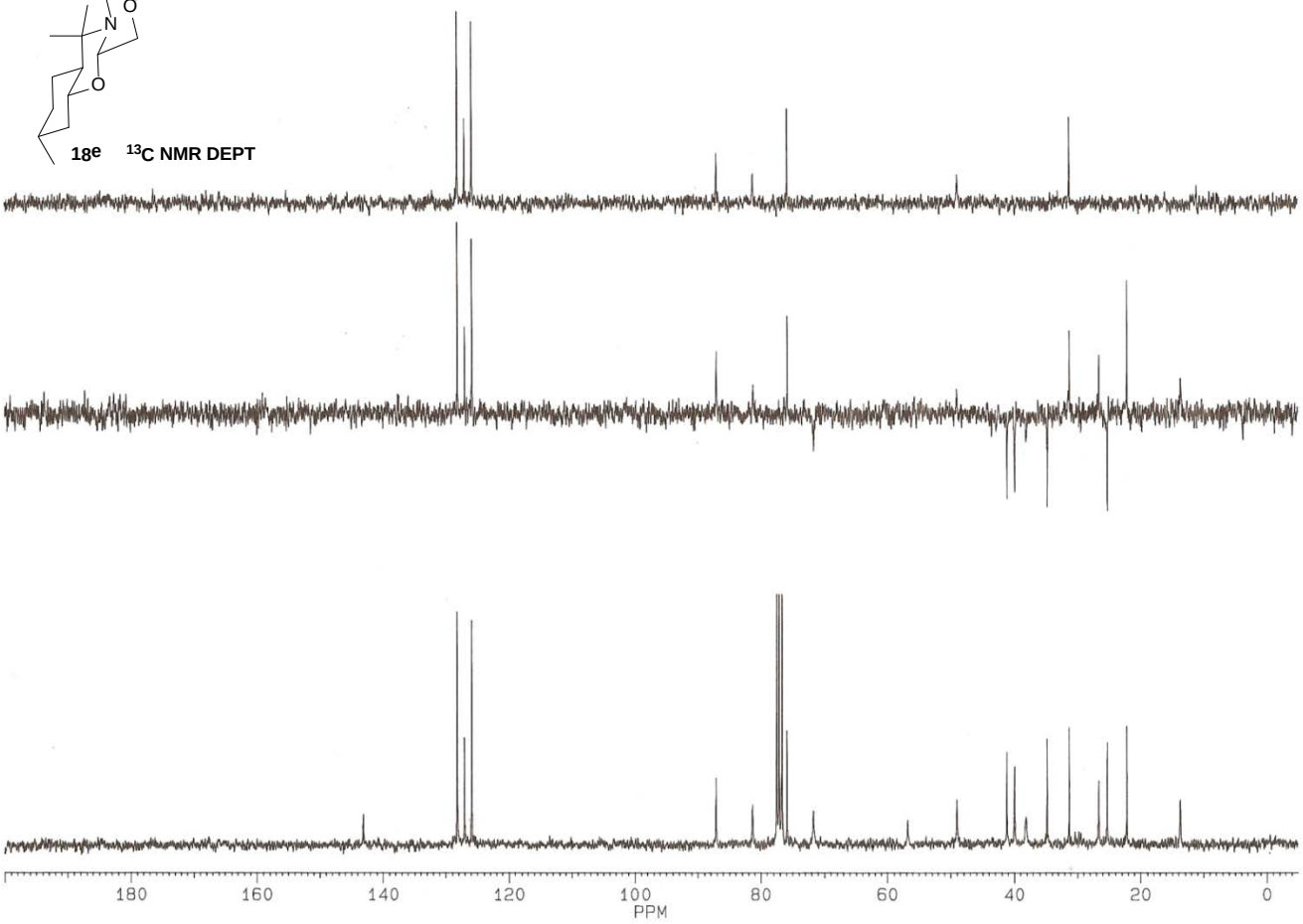


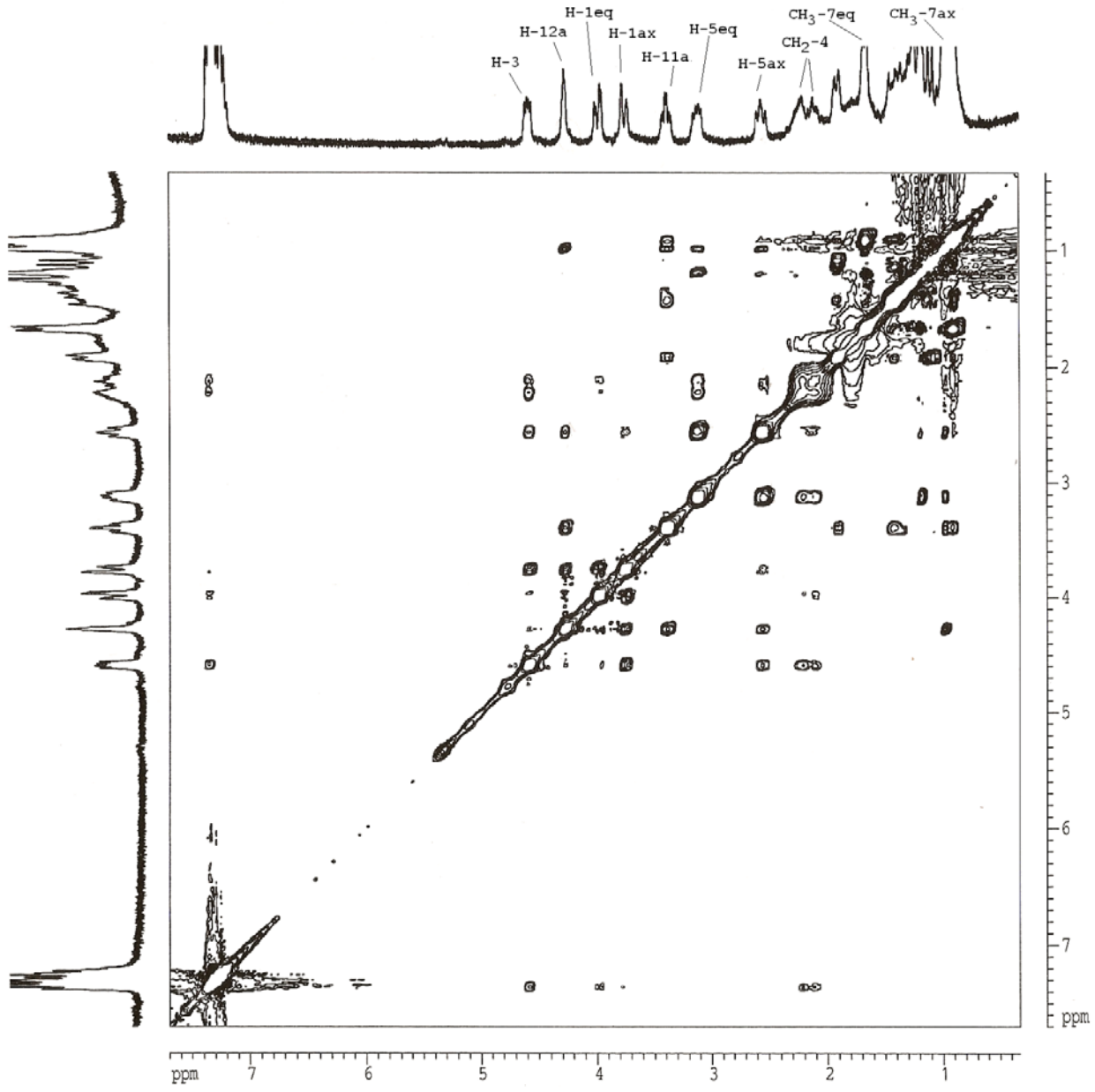
18e **^{1}H NMR**





18e **^{13}C NMR DEPT**





Current Data Parameters

NAME	pm20699
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	990520
Time	3.18
PULPROG	noesytp
SOLVENT	CDCl3
AQ	0.2314440 sec
FIDRES	2.160537 Hz
DW	226.0 usec
RG	1430
NUCLEUS	1H
D1	2.0000000 sec
P1	6.9 usec
D0	0.0000030 sec
D8	0.8000000 sec
DE	282.5 usec
SFO1	300.1345733 MHz
SWH	2212.39 Hz
TD	1024
NS	32
DS	2
INO	0.0002260 sec

F1 - Acquisition parameters

NDO	2
TD	256
SFO1	300.1346 MHz
FIDRES	8.642118 Hz
SW	7.371 ppm

F1 - Processing parameters

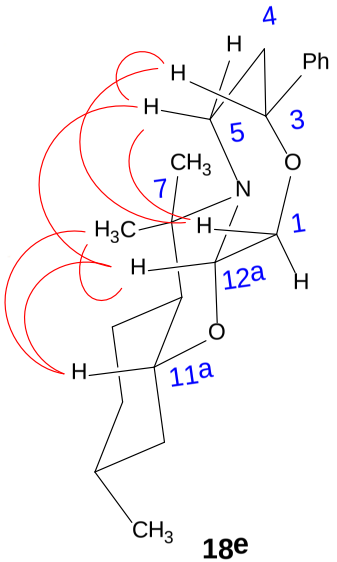
SI	512
MC2	QF
SF	300.1333680 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	TPPI
SF	300.1333680 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

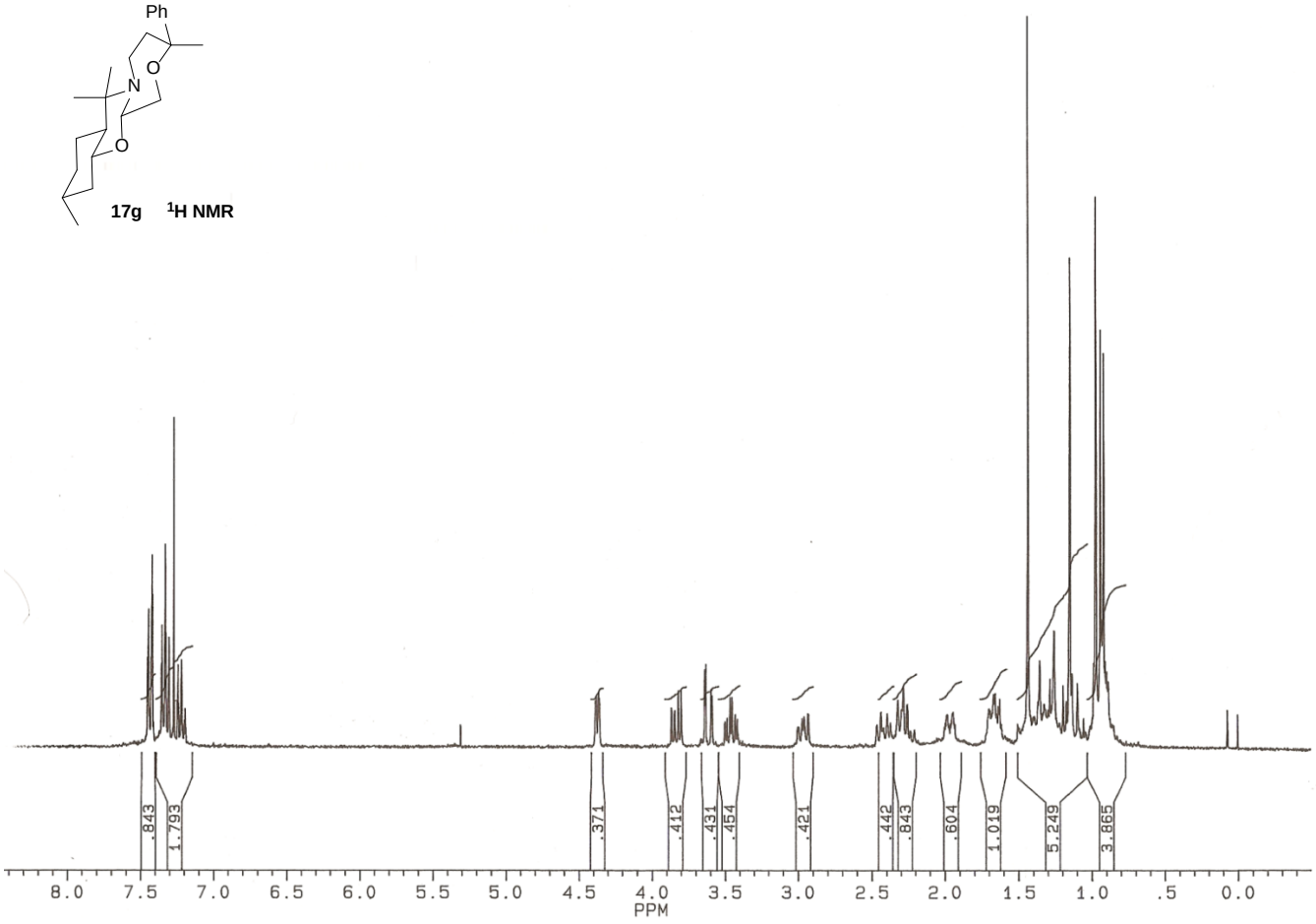
CX2	16.00 cm
CX1	16.00 cm
F2PLO	7.702 ppm
F2LO	2311.54 Hz
F2PHI	0.330 ppm
F2HI	99.15 Hz
F1PLO	7.702 ppm
F1LO	2311.49 Hz
F1PHI	0.330 ppm
F1HI	99.11 Hz
F2PPMCM	0.46071 ppm/cm
F2HZCM	138.27434 Hz/cm
F1PPMCM	0.46071 ppm/cm
F1HZCM	138.27386 Hz/cm

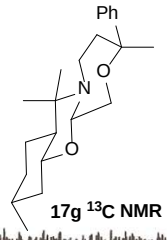


18e

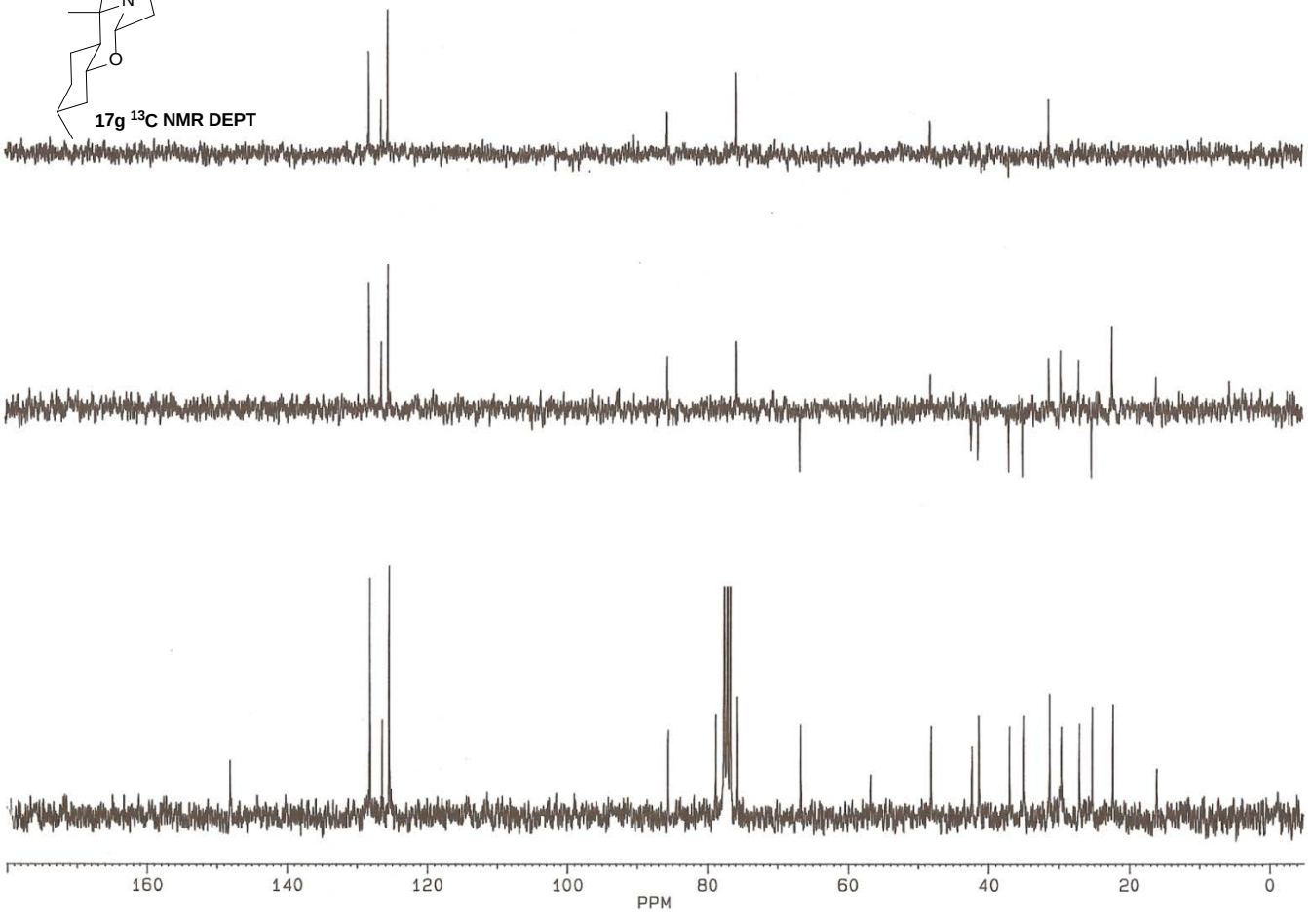
1H NOESY contacts

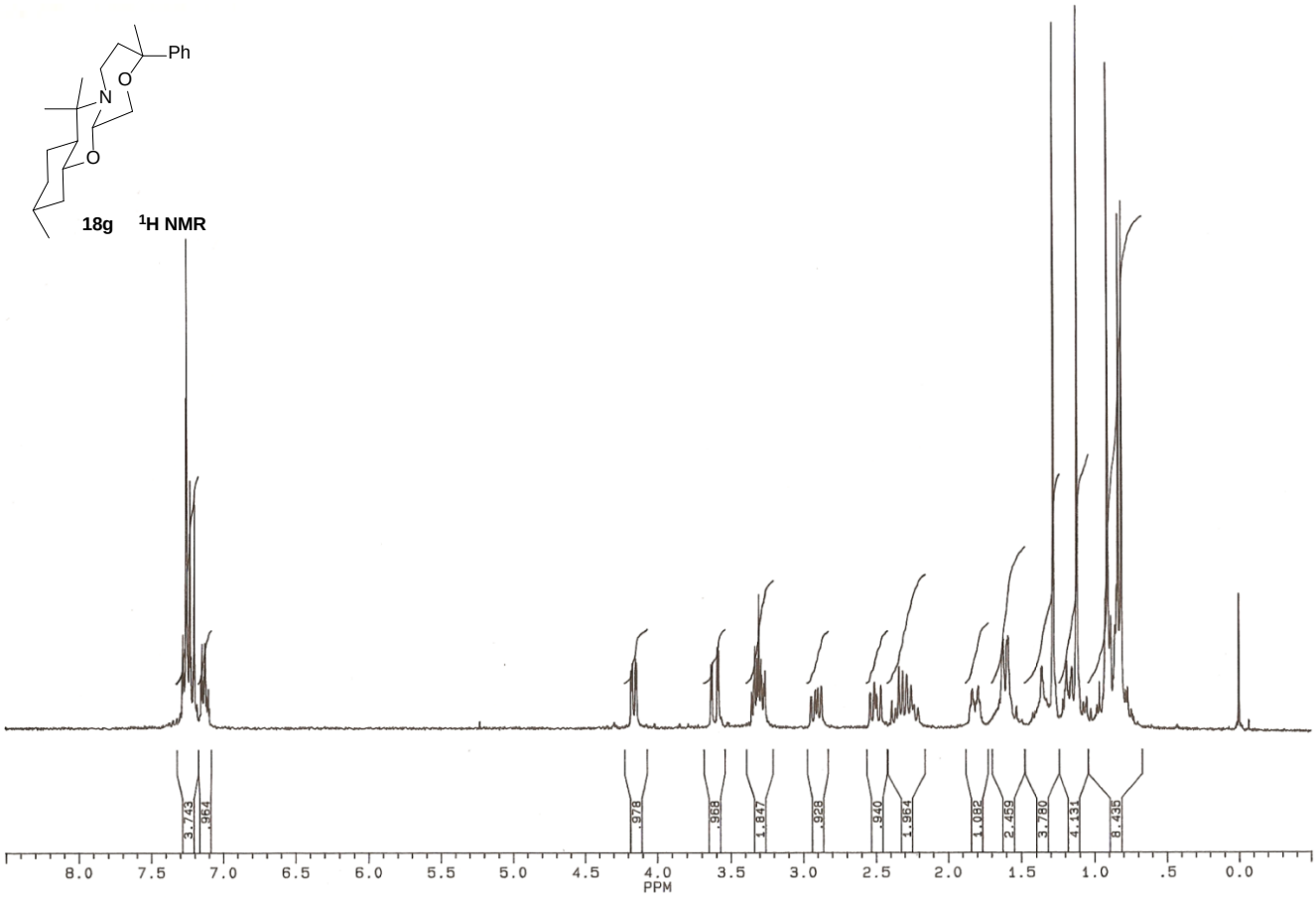
17g ^{1}H NMR



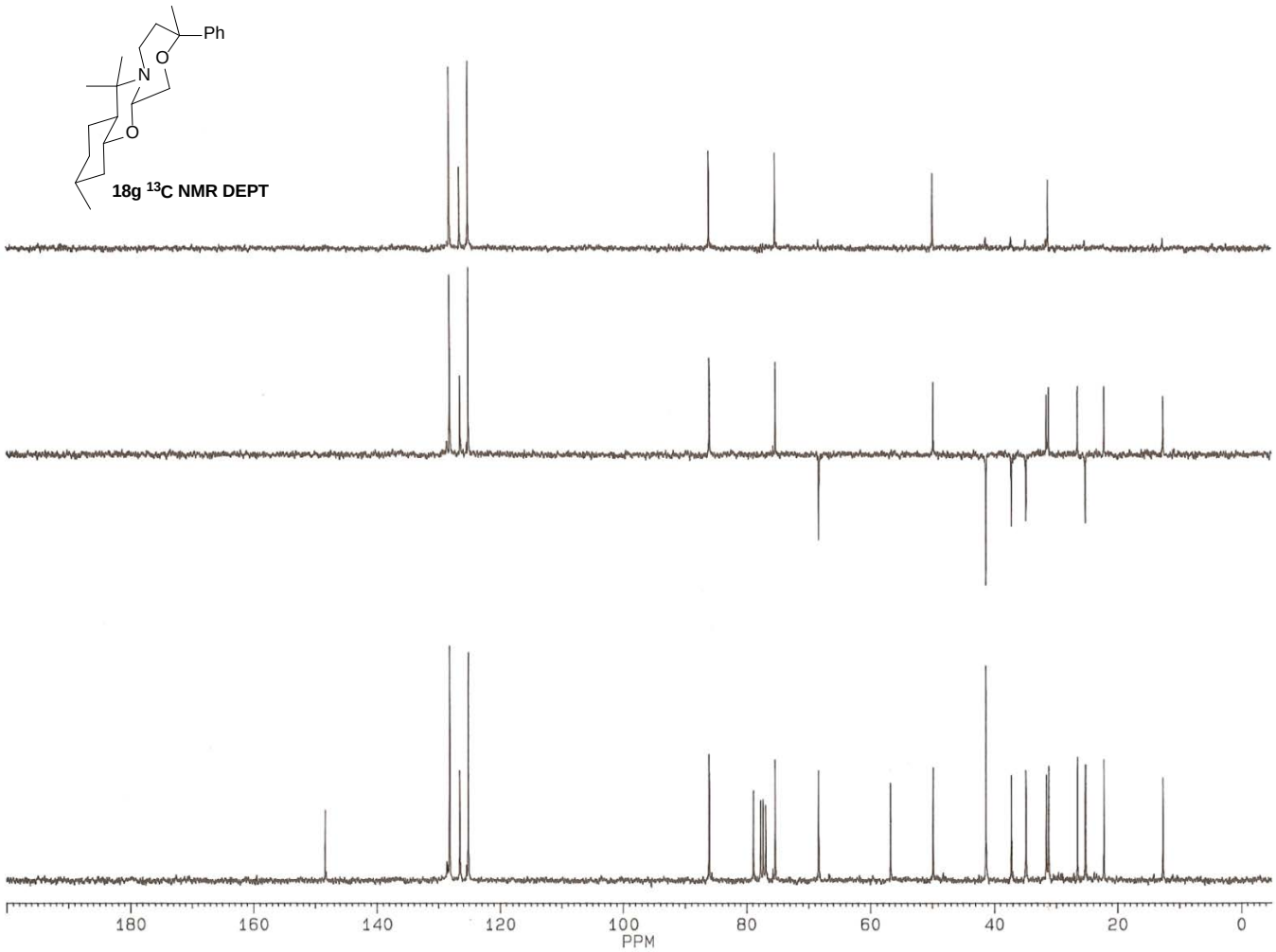


17g ^{13}C NMR DEPT



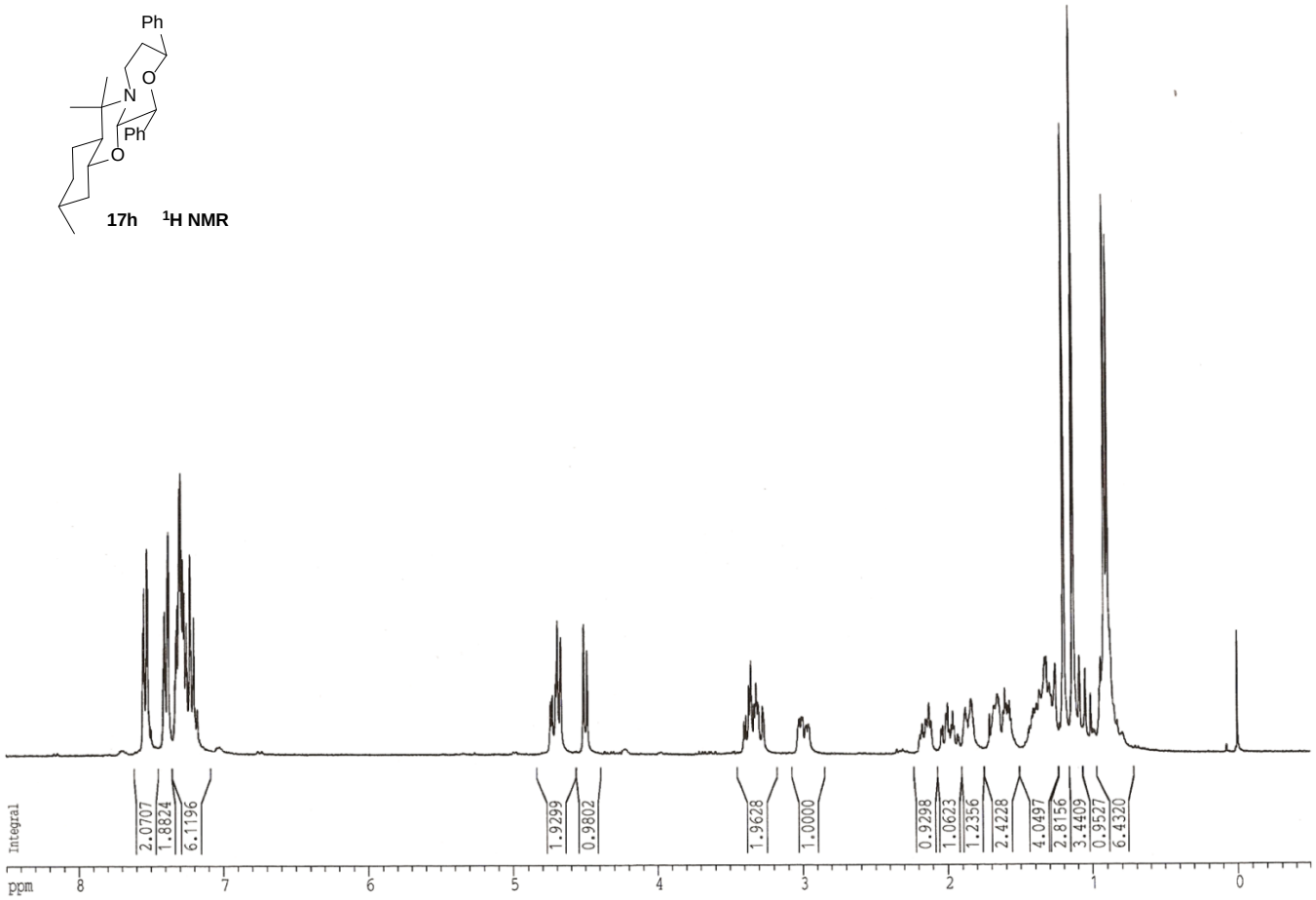


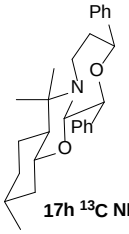
18g ^{1}H NMR



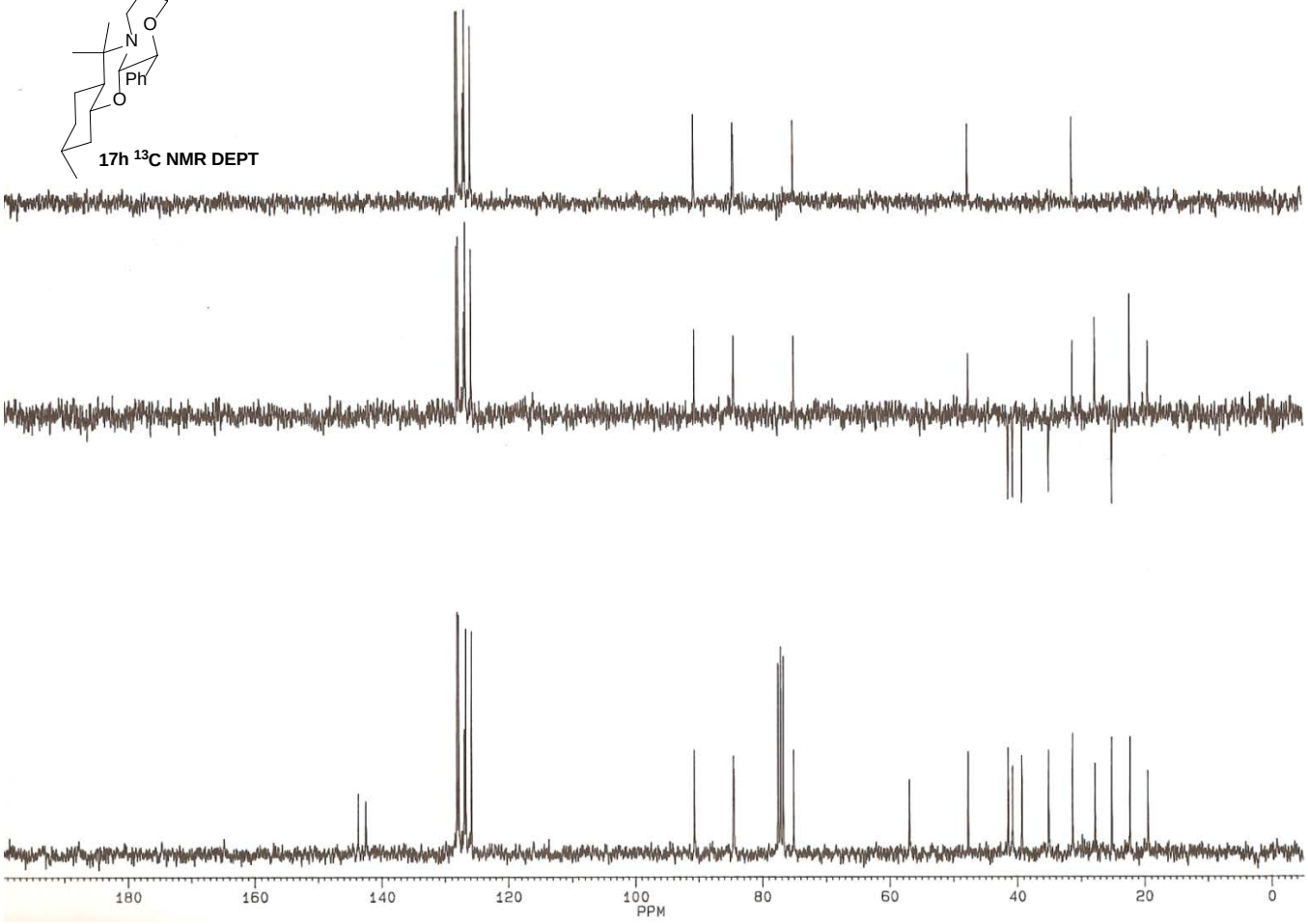
18g ^{13}C NMR DEPT

17h ^{1}H NMR

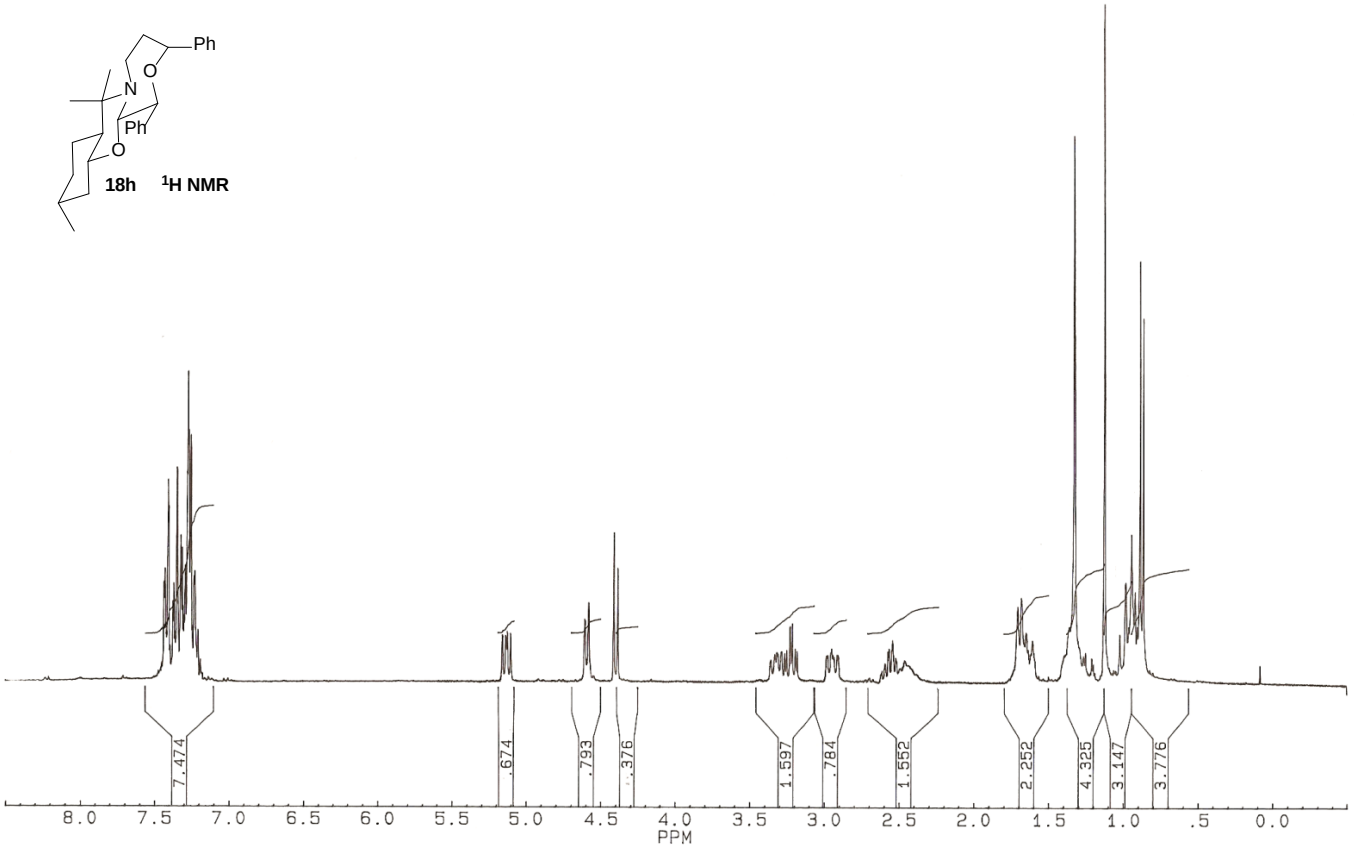


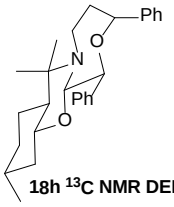


17h ^{13}C NMR DEPT

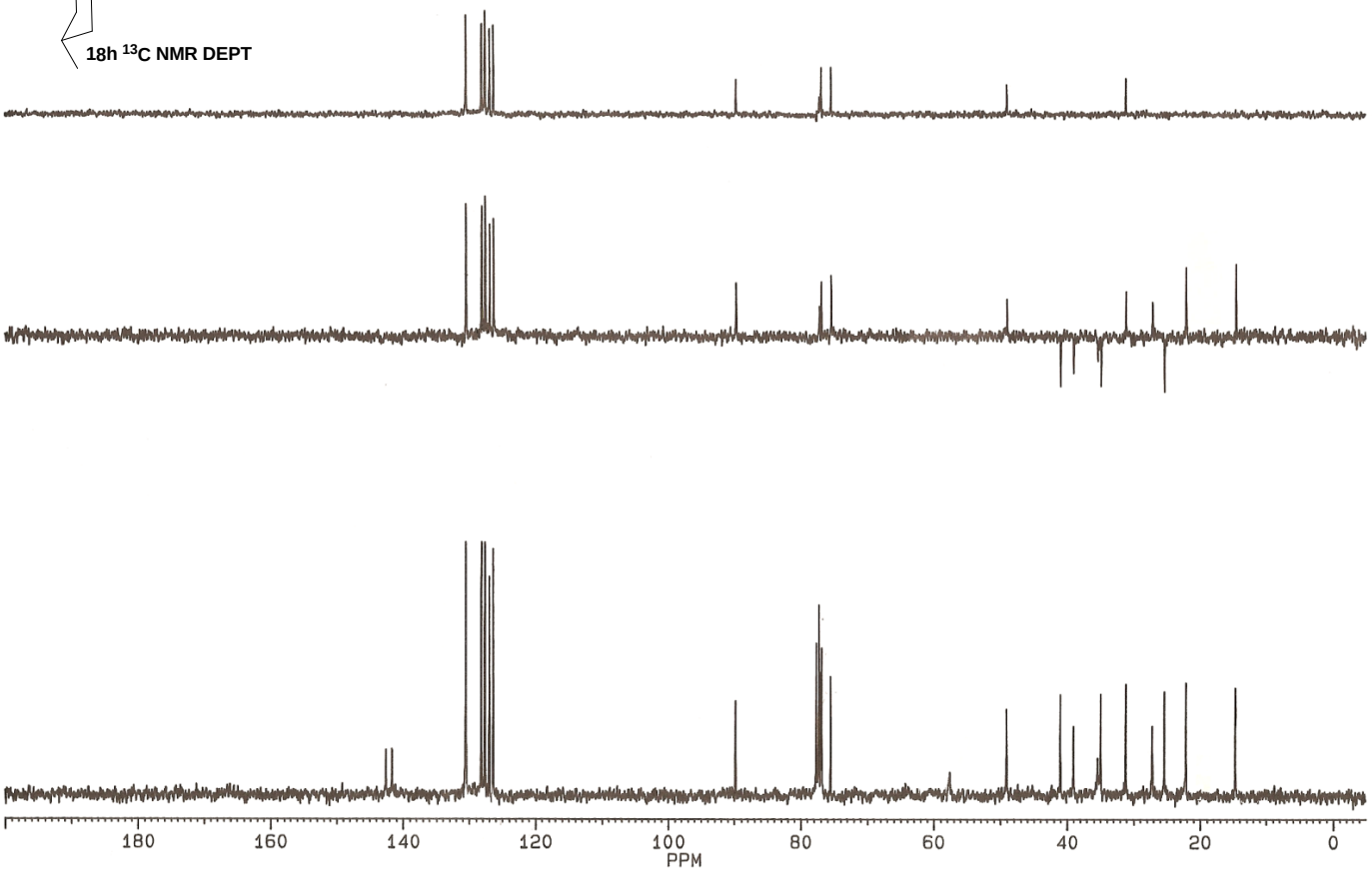


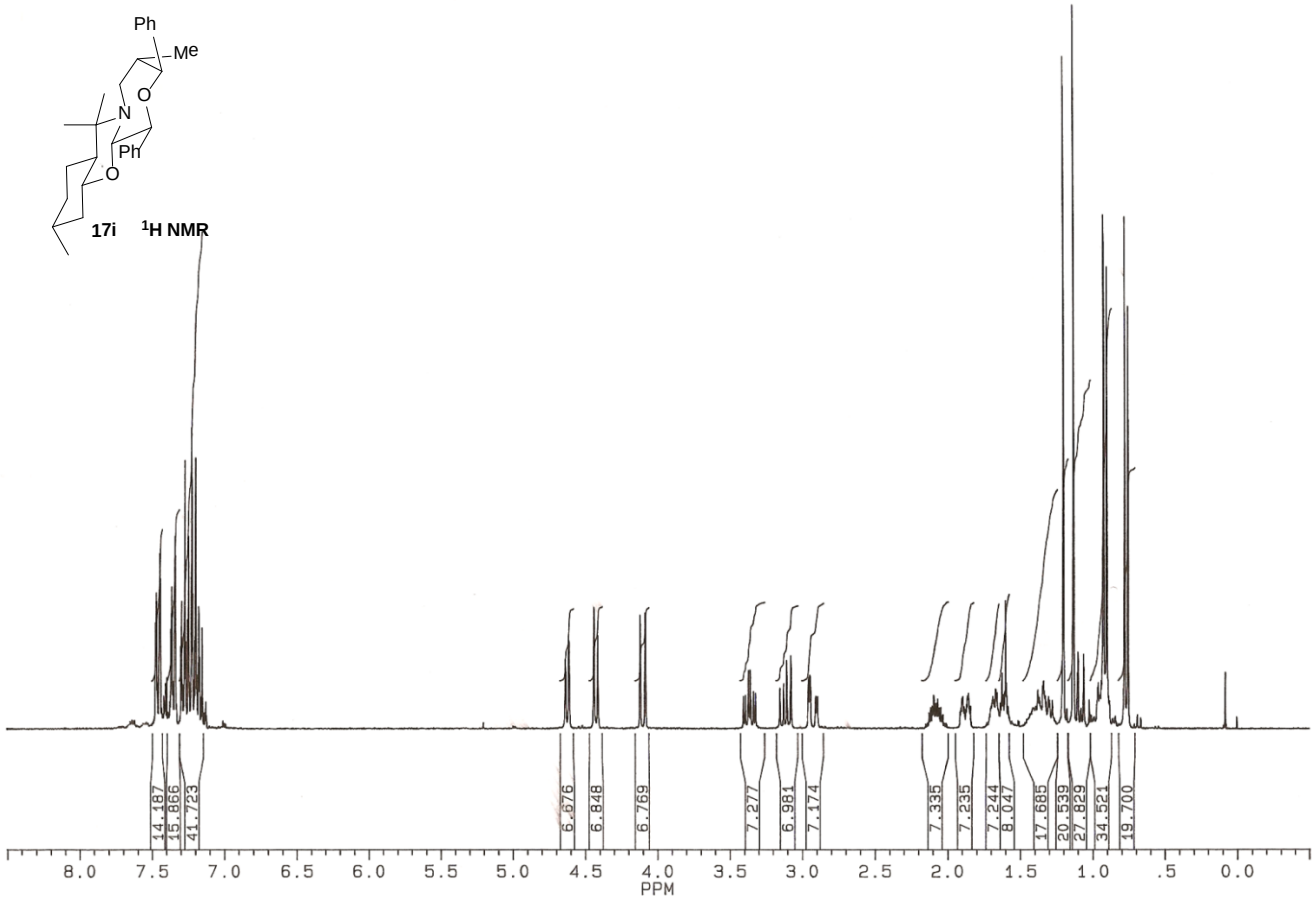
18h ^{1}H NMR

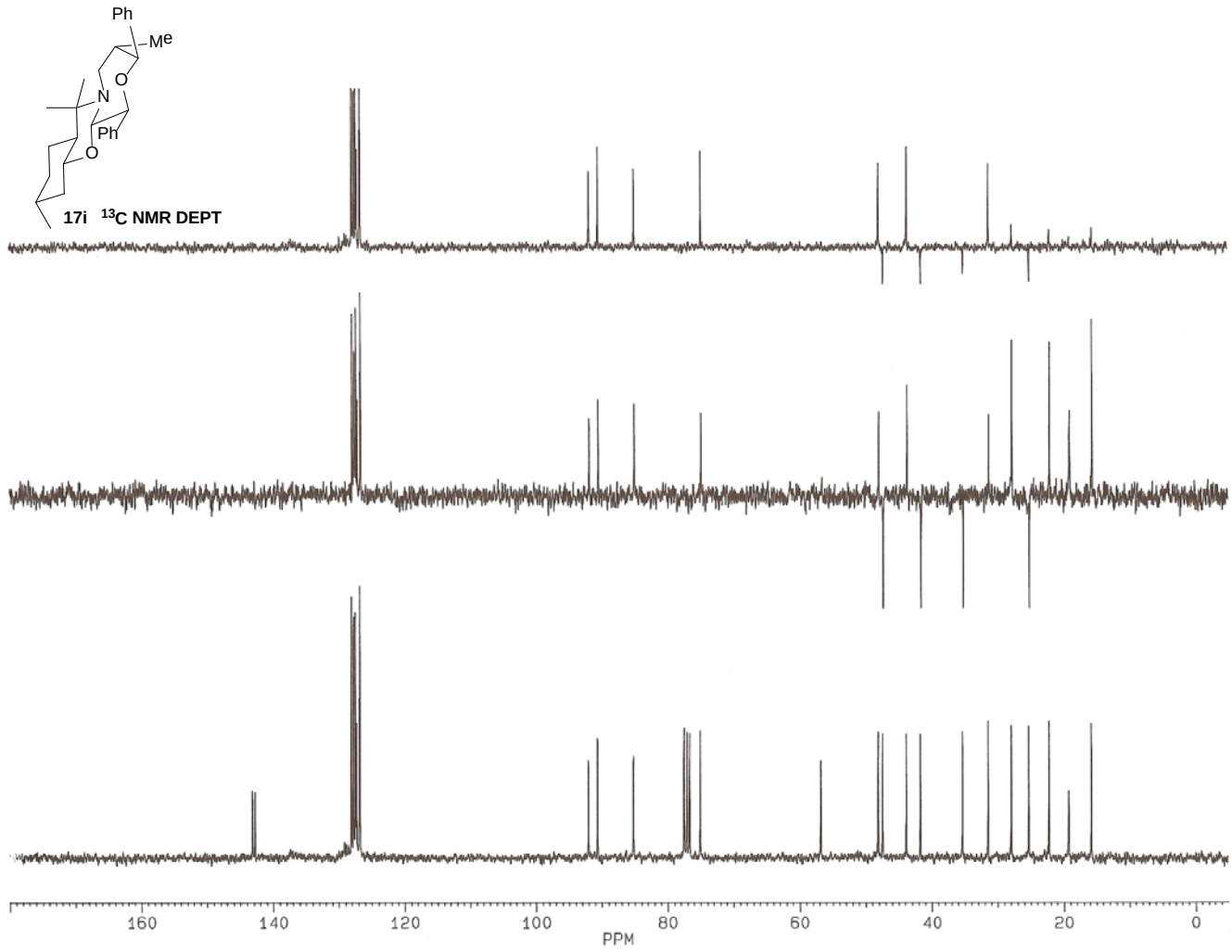


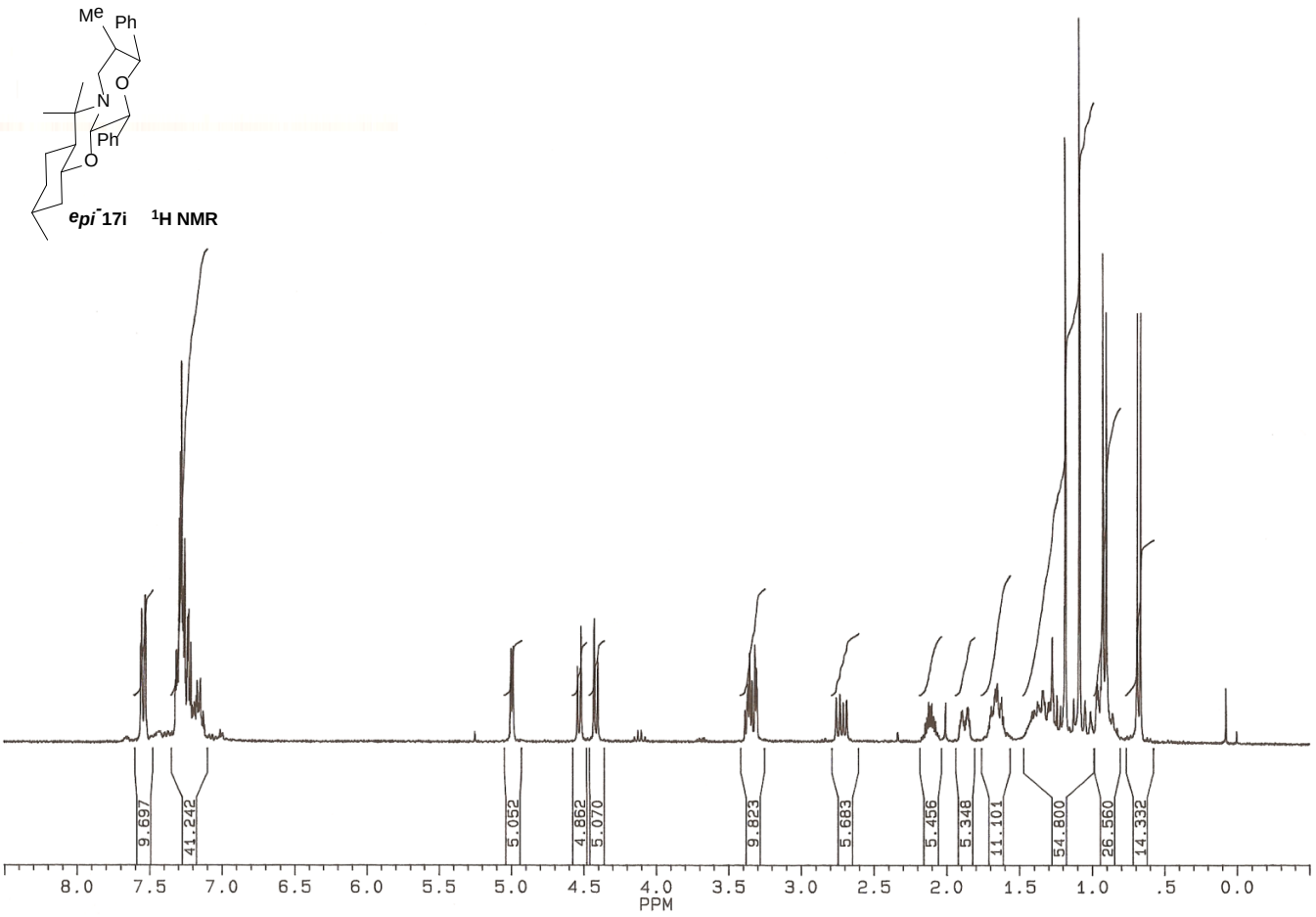


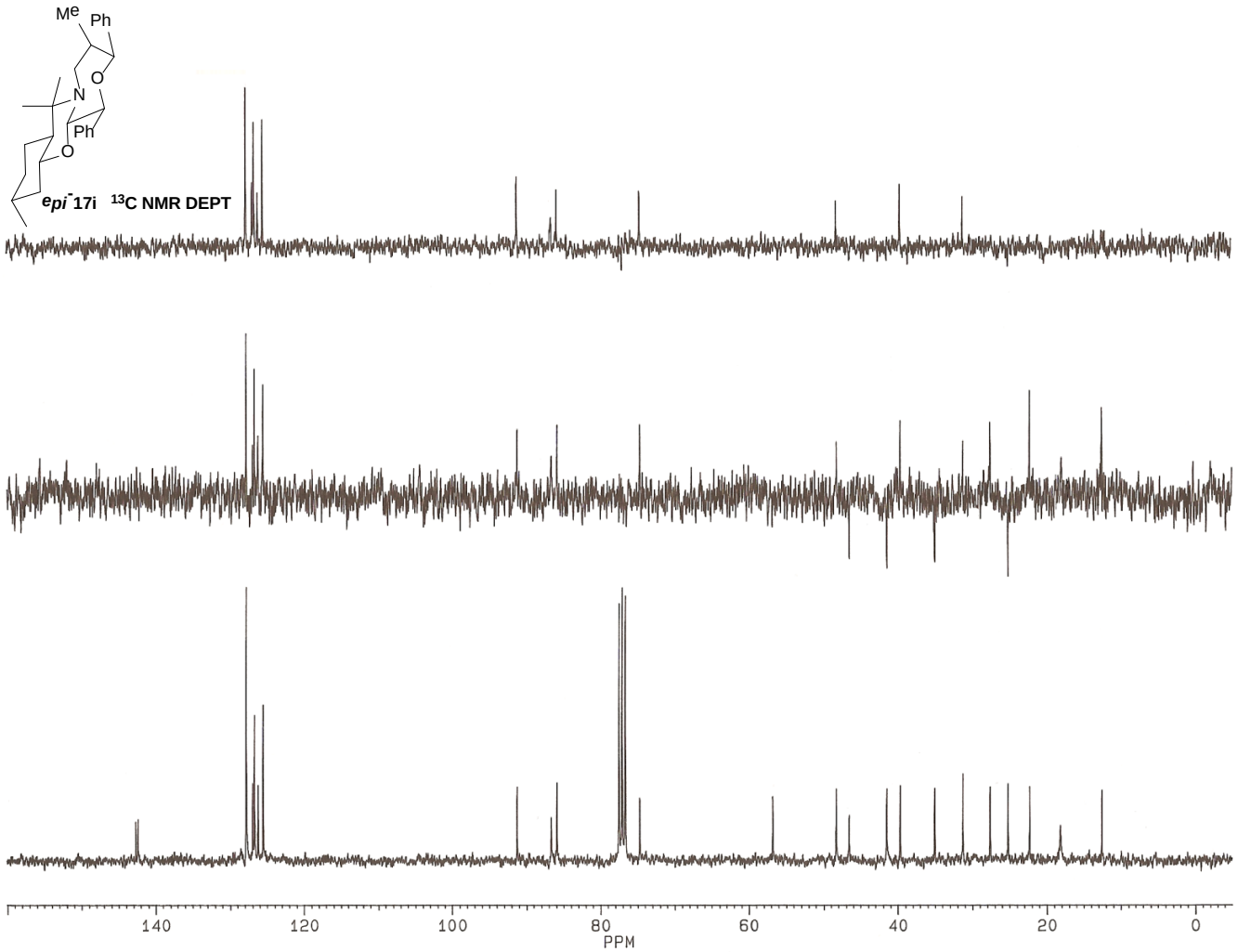
18h ^{13}C NMR DEPT

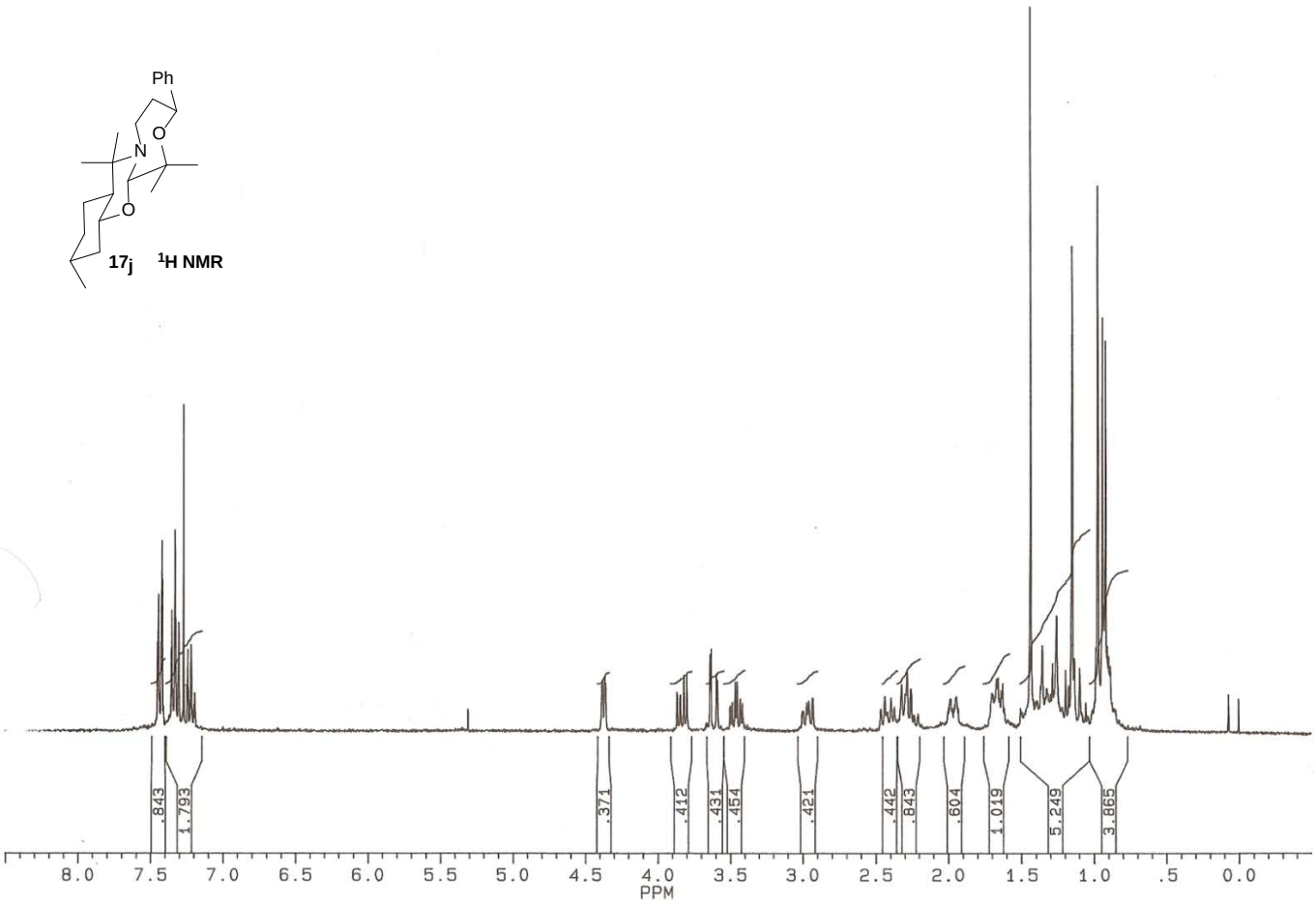


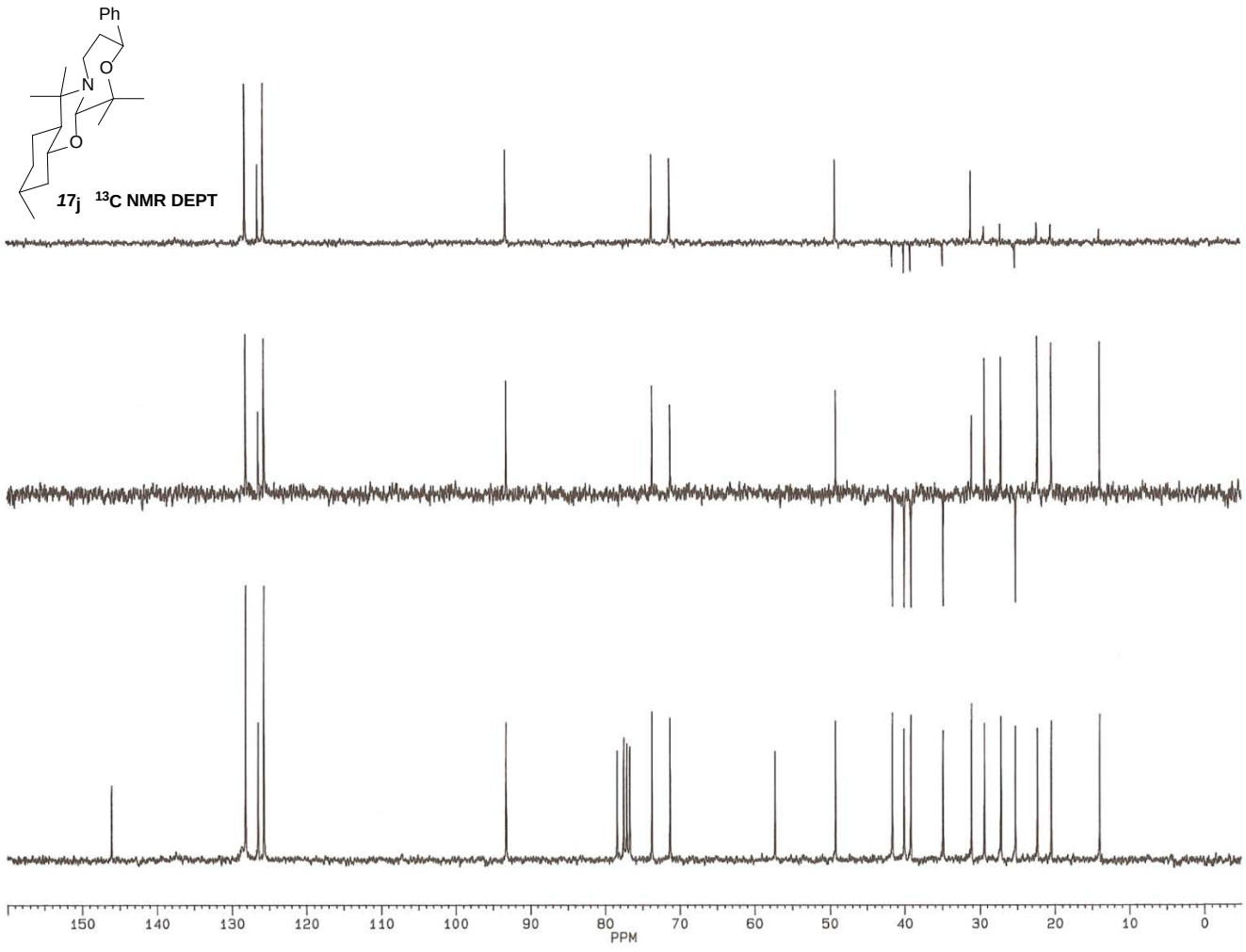

Ph
Me
N
O
Ph
O
17i ¹H NMR
14.187
15.866
41.723
6.676
6.848
6.759
7.277
6.981
7.174
7.335
7.235
7.244
8.047
17.685
20.539
27.829
34.521
19.700
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
0.0
PPM

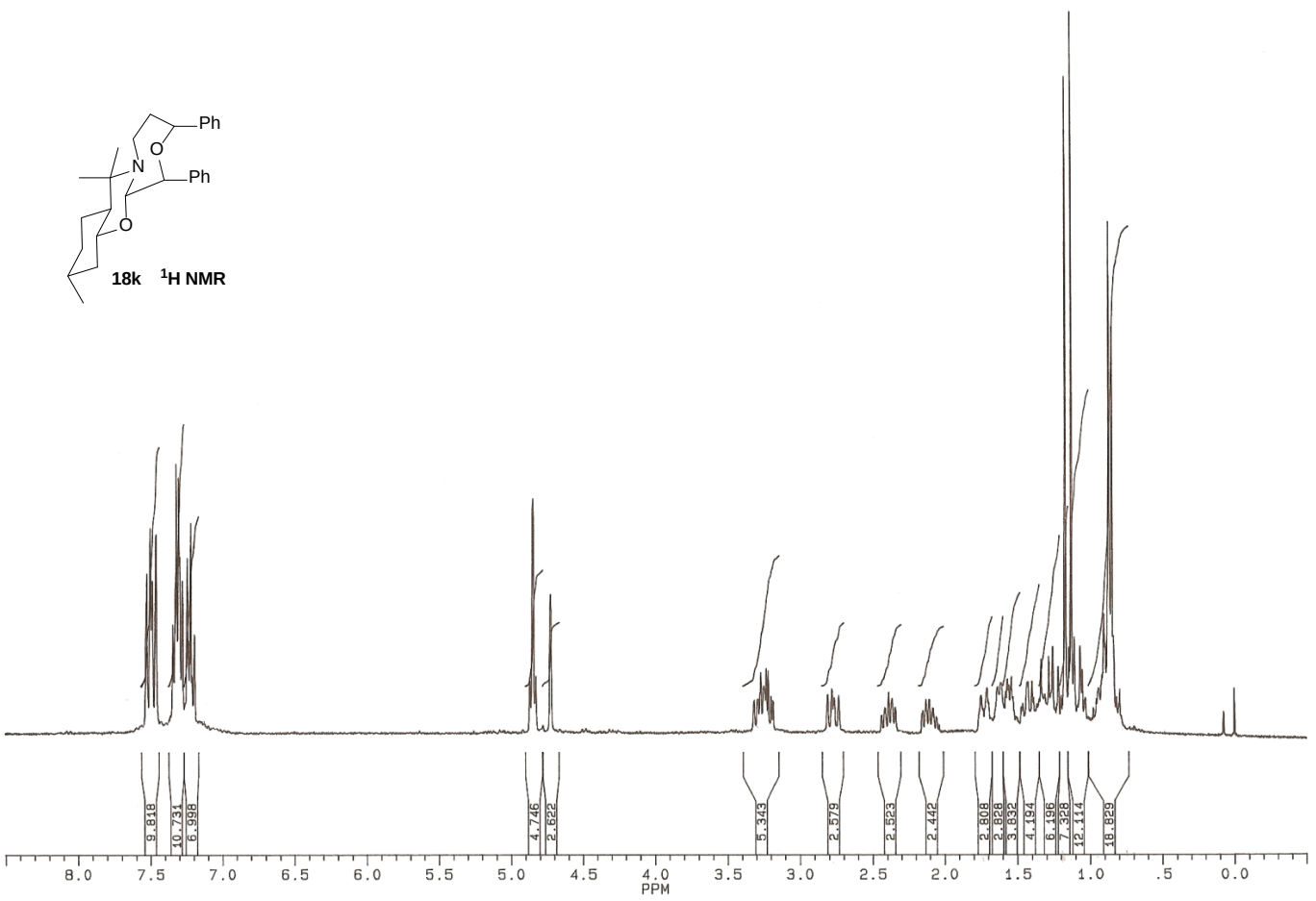

Ph
Me
N
O
Ph
O
17i ¹³C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM

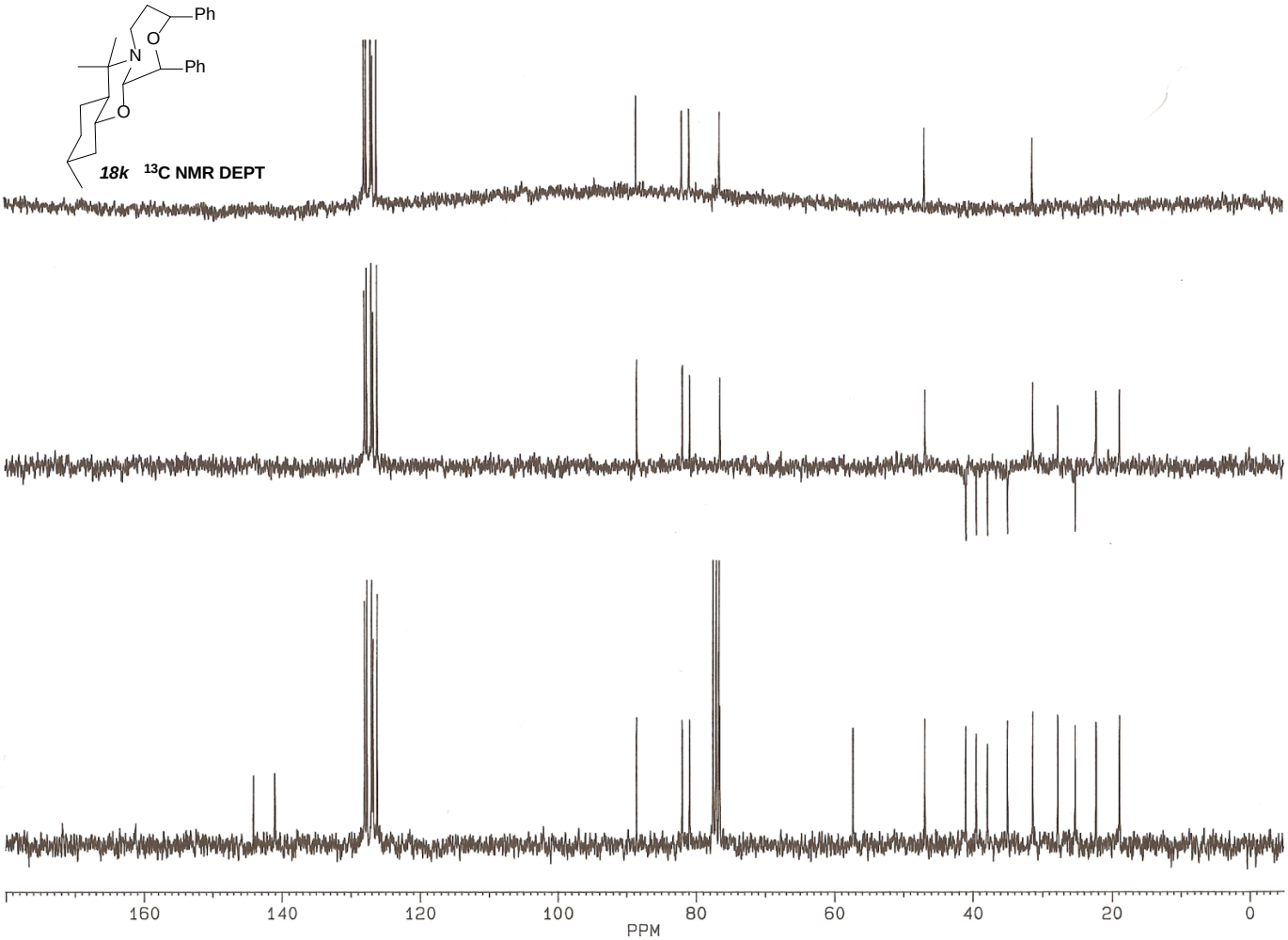

Me
Ph
O
N
Ph
O
epi-17i 1H NMR
9.697
41.242
5.052
4.862
5.070
9.823
5.683
5.456
5.348
11.101
54.800
26.560
14.332
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM

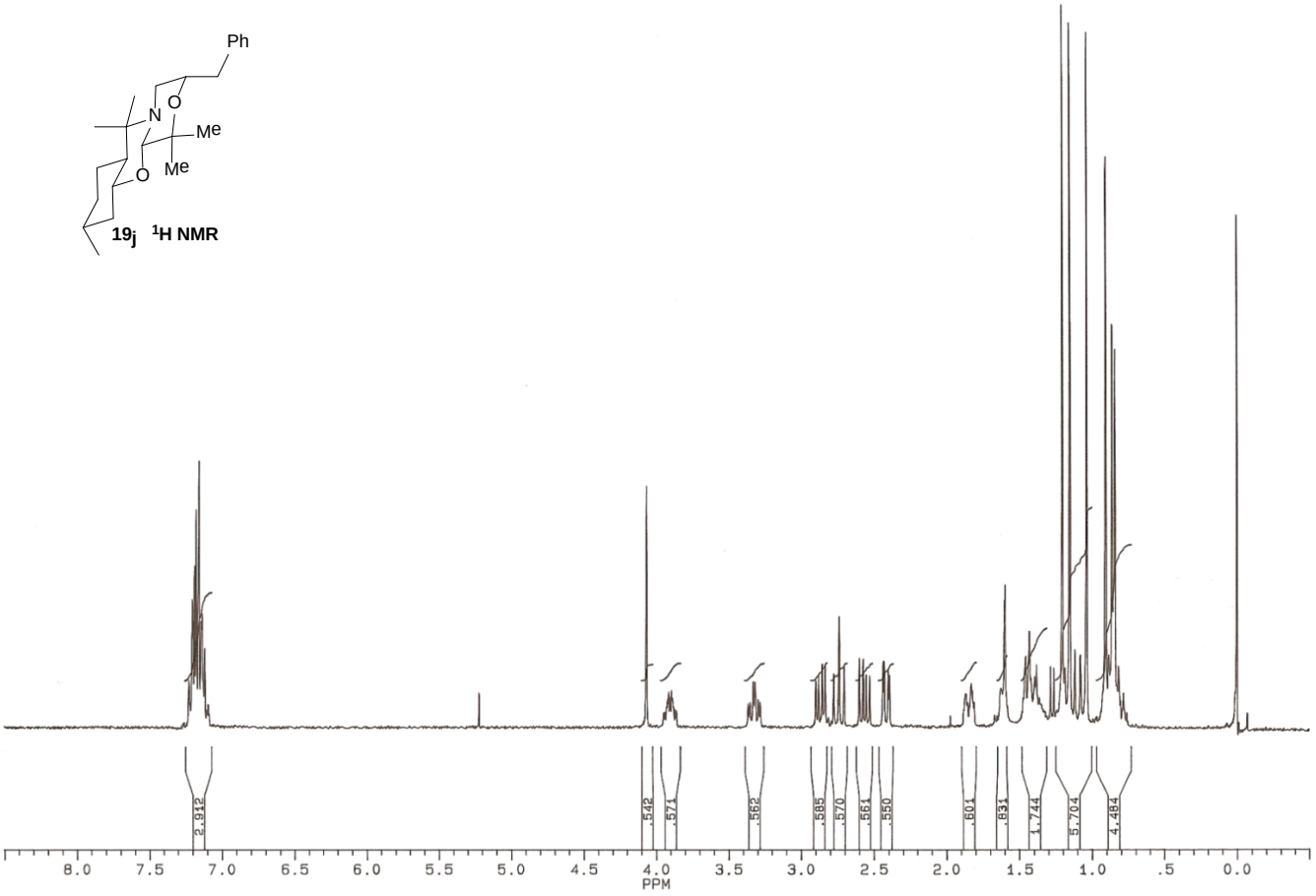

Me
Ph
O
N
Ph
O
epi-17i 13C NMR DEPT
140 120 100 80 60 40 20 0
PPM

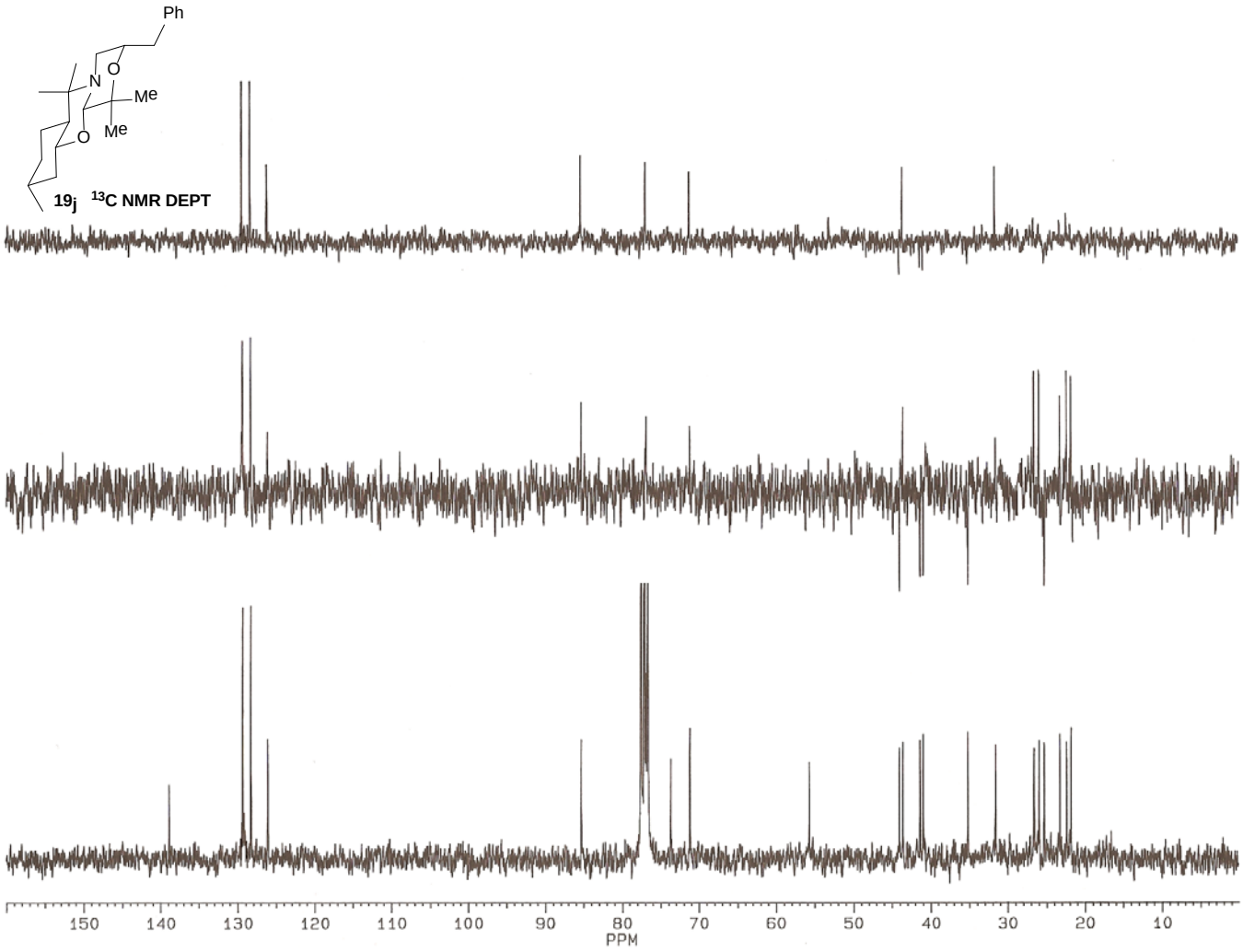

Ph
O
N
O
17j 1H NMR
.843
1.793
.371
.412
.431
.454
.421
.442
.843
.604
1.019
5.249
3.865
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM

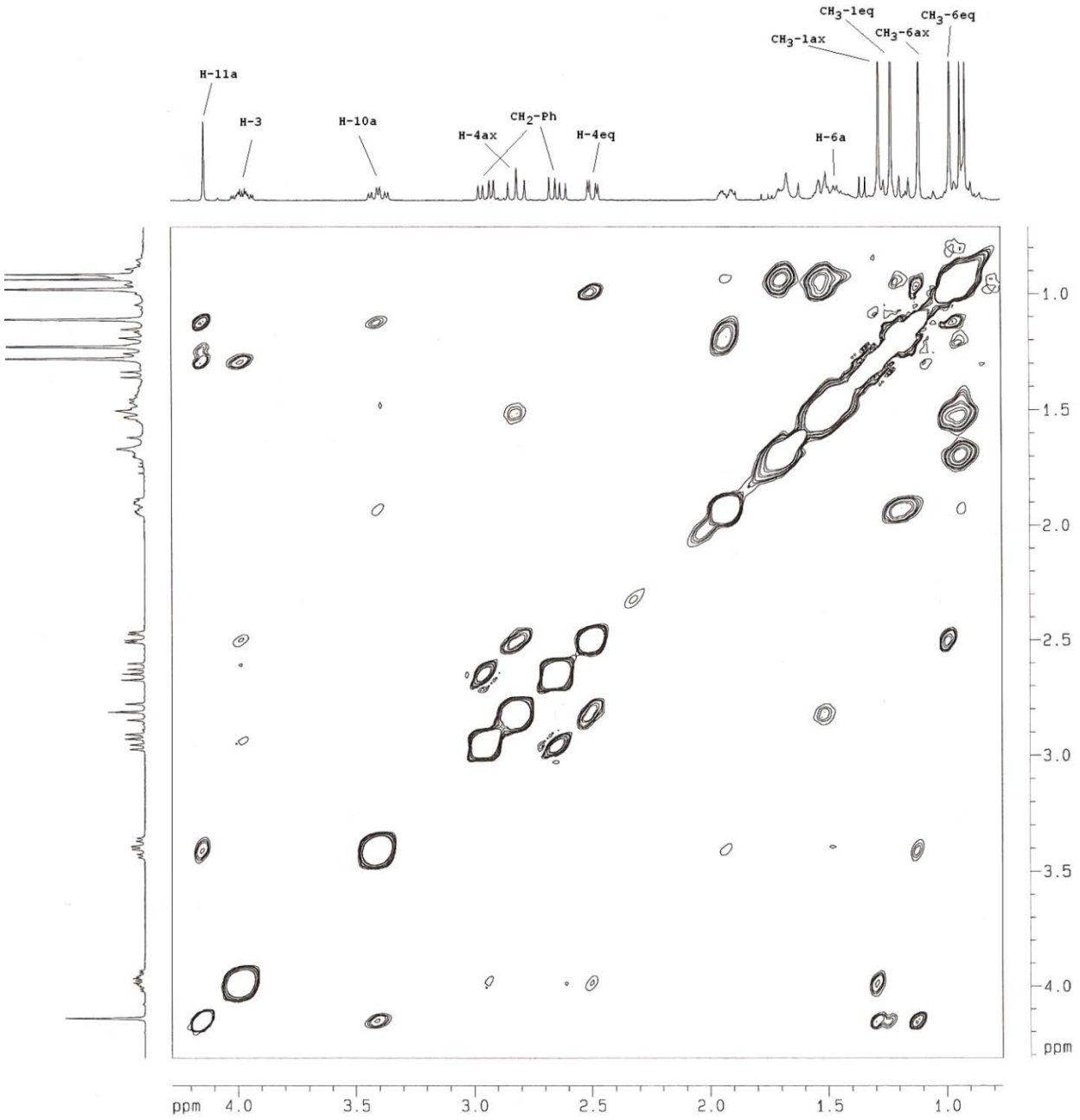

Ph
O
N
O
17j 13C NMR DEPT
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
PPM


Ph
O
N
Ph
O
18k 1H NMR
9.818
10.731
6.998
4.746
2.622
5.343
2.579
2.523
2.442
2.808
2.828
3.832
4.194
6.196
7.328
12.114
18.829
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
.5
0.0
PPM

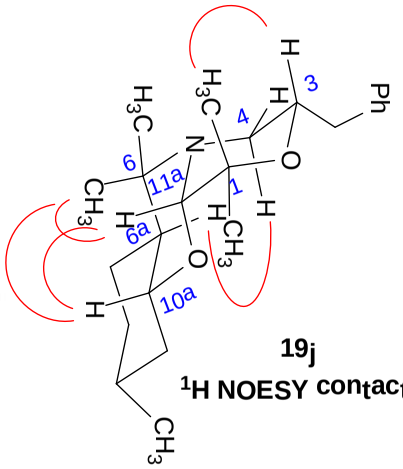

Ph
O
N
Ph
O
18k 13C NMR DEPT
160
140
120
100
80
60
40
20
0
PPM


Ph
N
O
Me
Me
O
19j 1H NMR
2.912
.542
.571
.562
.585
.570
.561
.550
.601
.831
1.744
5.704
4.484
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
1.0
.5
0.0
PPM


Ph
N
O
Me
Me
O
19j 13C NMR DEPT
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
PPM

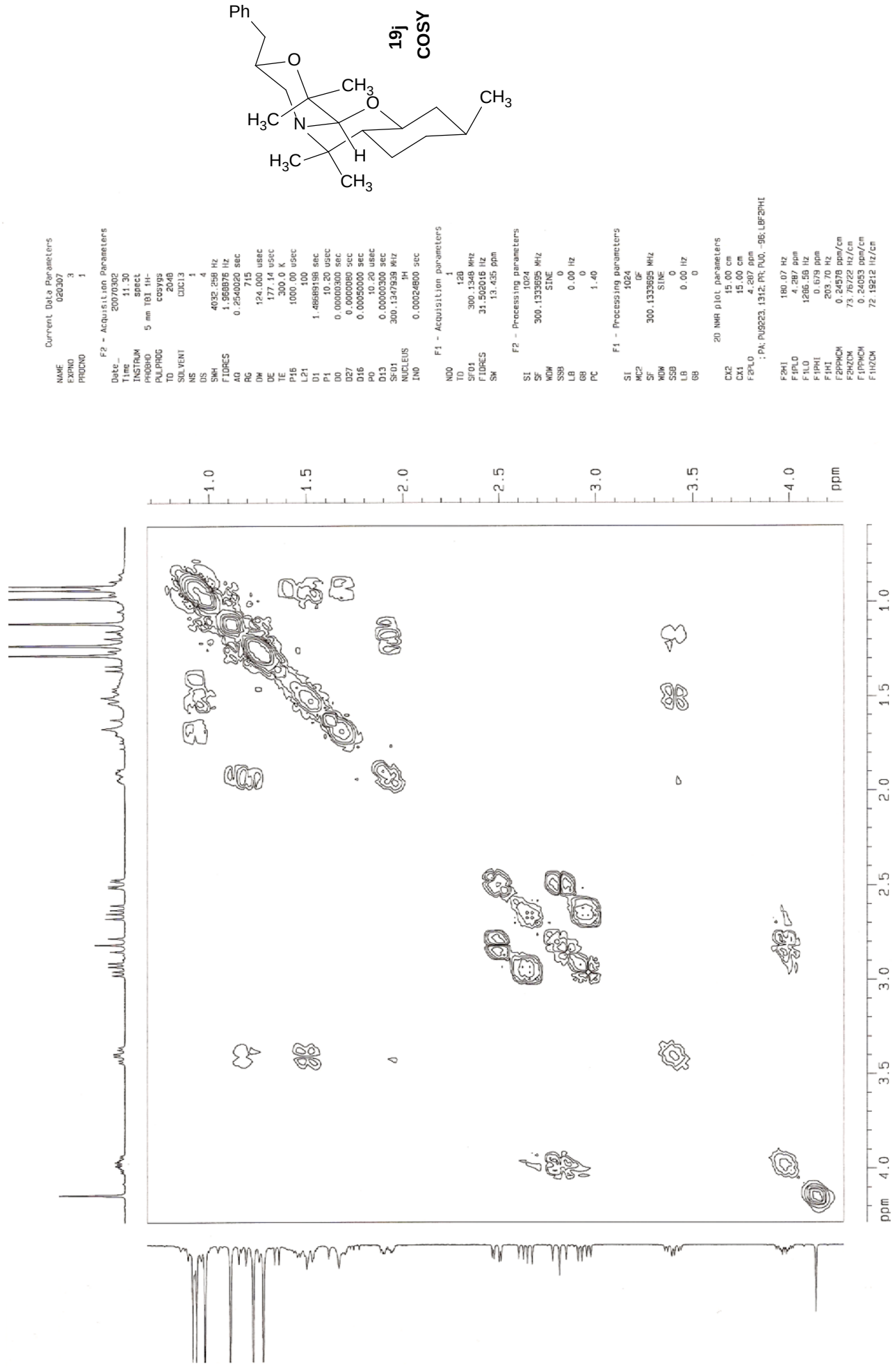



```
Current Data Parameters
NAME                 020307
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20070302
Time                 11.01
INSTRUM              spect
PROBHD     5 mm TBI 1H-
PULPROG              noesygs
TD                     1024
SOLVENT              CDCl3
NS                        4
DS                        4
SWH              3496.503 Hz
FIDRES           3.414554 Hz
AQ               0.1464820 sec
RG                     1024
DW                143.000 usec
DE                 204.29 usec
TE                 300.0 K
P16               1000.00 usec
L21                    100
DB             0.50000000 sec
D16            0.00010000 sec
D1             1.00000000 sec
P1                   9.00 usec
D0             0.0000030 sec
D20            0.2489000 sec
D27            0.0000080 sec
P2                  18.00 usec
SFO1          300.1349872 MHz
NUCLEUS              1H
IND            0.00014300 sec

F1 - Acquisition parameters
NDO                       2
TD                      256
SFO1            300.135 MHz
FIDRES        13.658253 Hz
SW              11.650 ppm

F2 - Processing parameters
SI                     1024
SF            300.1333678 MHz
WDW                  QSINE
SSB                       2
LB                 0.00 Hz
GB                        0
PC                 1.00

F1 - Processing parameters
SI                     1024
MC2                    TPPI
SF            300.1333678 MHz
WDW                  QSINE
SSB                       2
LB                 0.00 Hz
GB                        0

2D NMR plot parameters
CX2                15.00 cm
CX1                15.00 cm
F2PLO              4.281 ppm
F2LO             1284.77 Hz
F2PHI              0.765 ppm
F2HI              229.68 Hz
F1PLO              4.315 ppm
F1LO             1295.02 Hz
F1PHI              0.708 ppm
F1HI              212.60 Hz
F2PPMCM         0.23436 ppm/cm
F2HZCM         70.33981 Hz/cm
F1PPMCM         0.24043 ppm/cm
F1HZCM         72.16110 Hz/cm
```

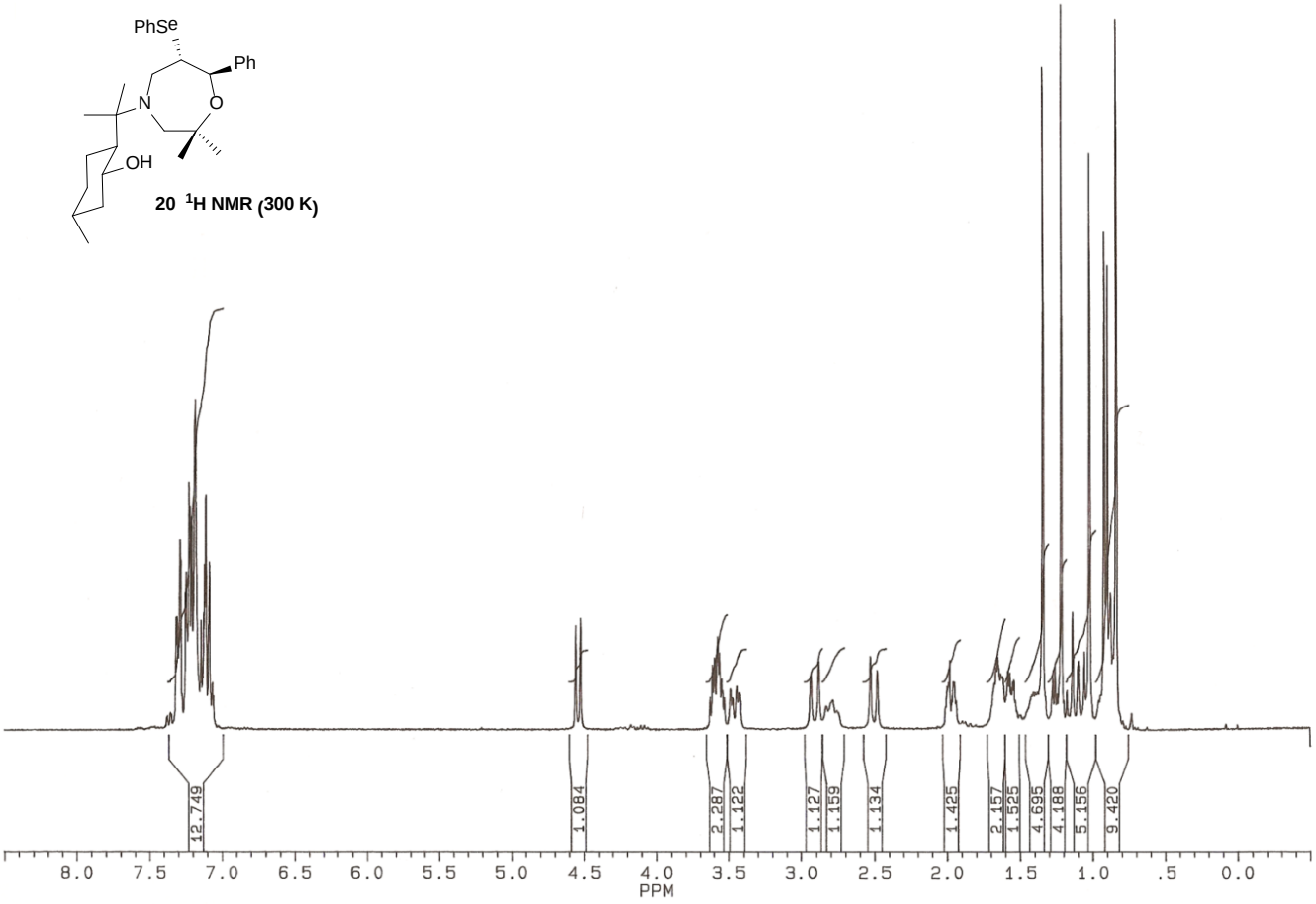


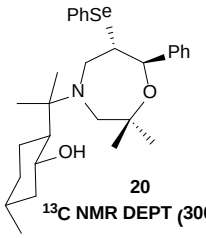
19j

^{1}H NOESY contacts


Ph
O
CH3
H3C
N
O
CH3
H3C
H
CH3
19j
COSY
Current Data Parameters
NAME 020307
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070302
Time 11.30
INSTRUM spect
PROBHD 5 mm TBI 1H-
PULPROG cosyqs
TD 2048
SOLVENT CDCl3
NS 1
DS 4
SWH 4032.258 Hz
FIDRES 1.968876 Hz
AQ 0.2540020 sec
RG 715
DW 124.000 usec
DE 177.14 usec
TE 300.0 K
P16 1000.00 usec
L21 100
D1 1.48689198 sec
P1 10.20 usec
D0 0.00000300 sec
D27 0.00000080 sec
D16 0.00050000 sec
P0 10.20 usec
D13 0.00000300 sec
SFO1 300.1347939 MHz
NUCLEUS 1H
IN0 0.00024800 sec
F1 - Acquisition parameters
NDO 1
TD 128
SFO1 300.1348 MHz
FIDRES 31.502016 Hz
SW 13.435 ppm
F2 - Processing parameters
SI 1024
SF 300.1333695 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1333695 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 4.287 ppm
; PA: PU9223.1312: PR: PU0.-95:LBF2PHI
F2HI 180.07 Hz
F1PLO 4.287 ppm
F1LO 1286.56 Hz
F1PHI 0.679 ppm
F1HI 203.70 Hz
F2PPMCM 0.24578 ppm/cm
F2HZCM 73.76722 Hz/cm
F1PPMCM 0.24053 ppm/cm
F1HZCM 72.19212 Hz/cm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
ppm

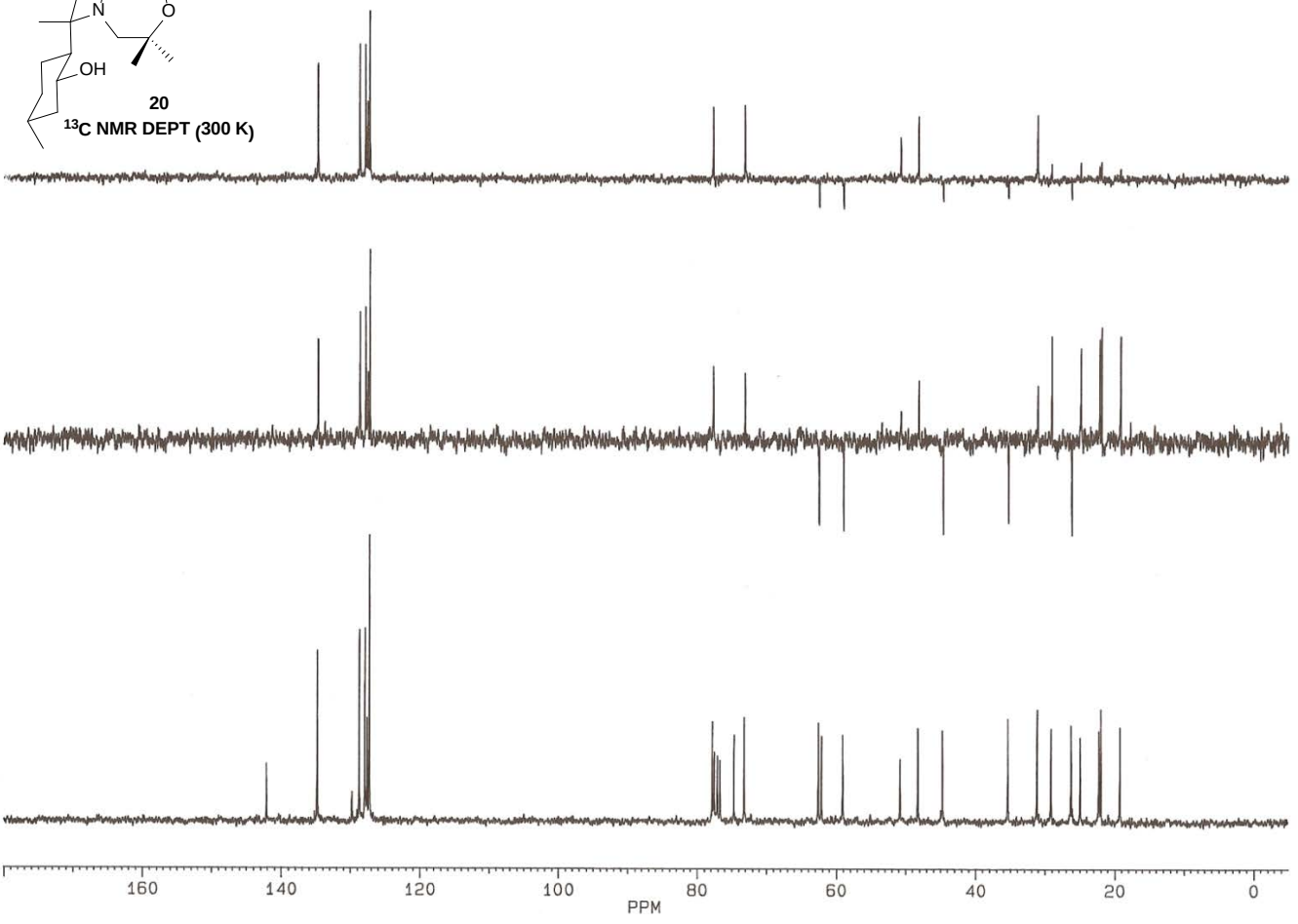
20 ^{1}H NMR (300 K)

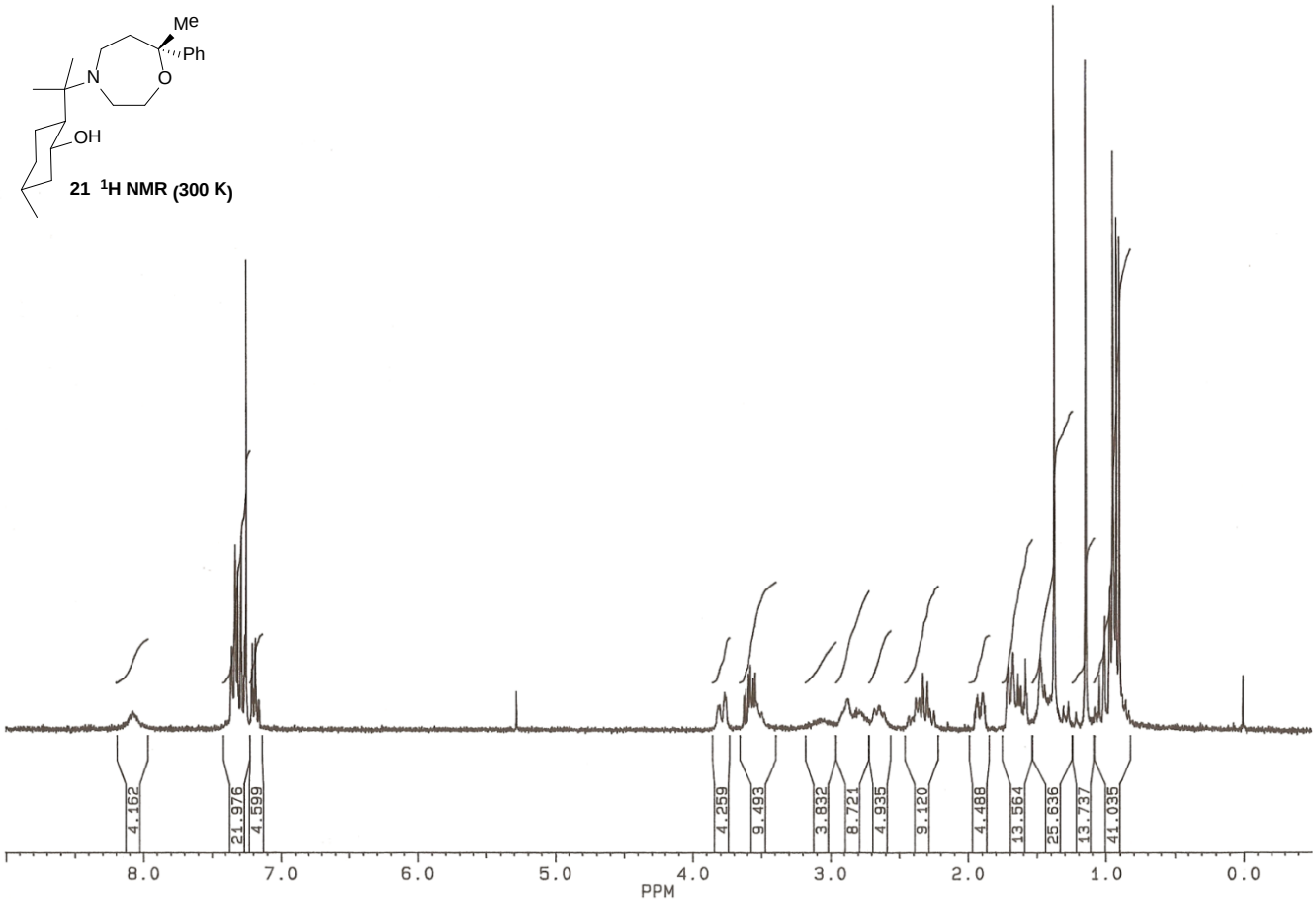




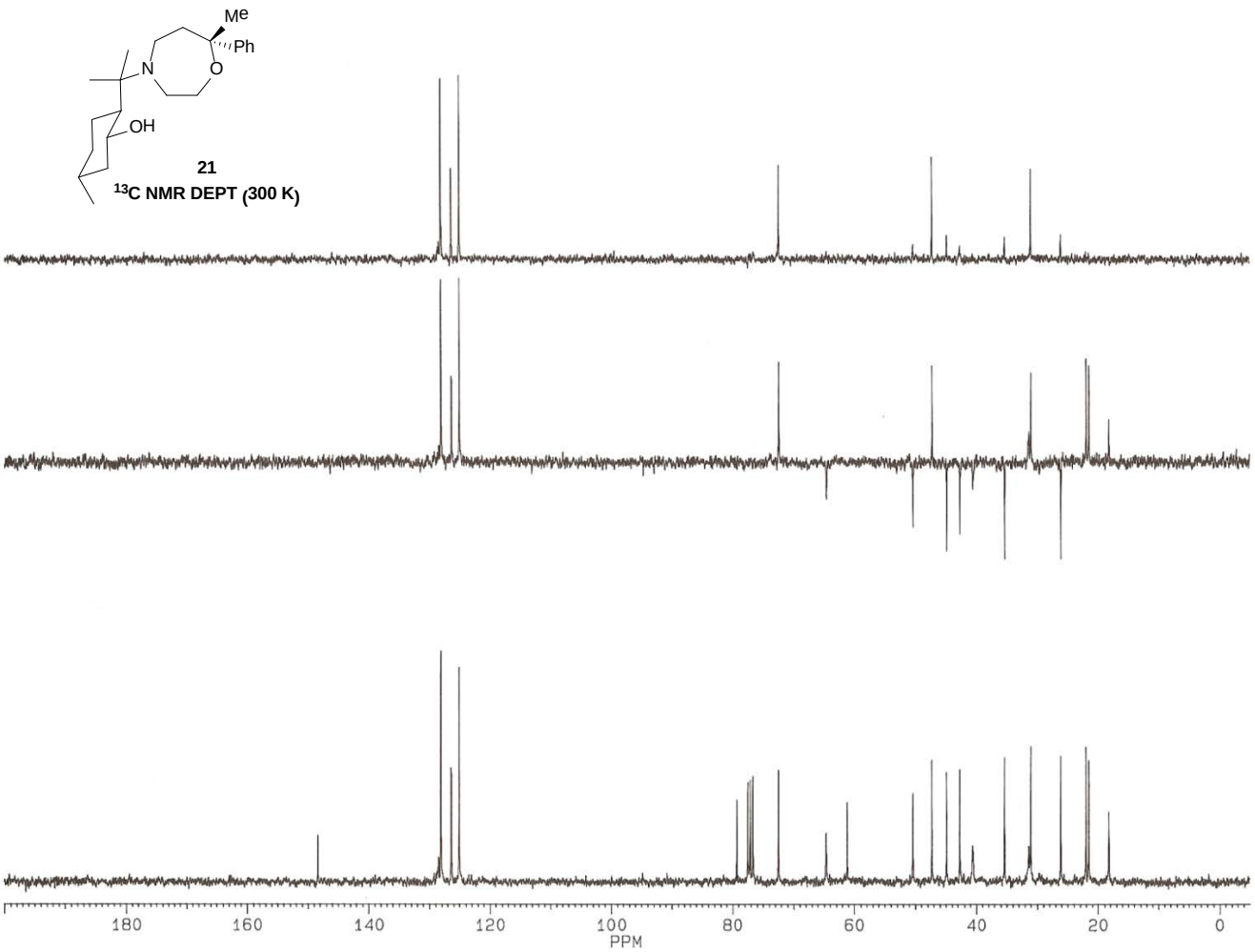
20

^{13}C NMR DEPT (300 K)





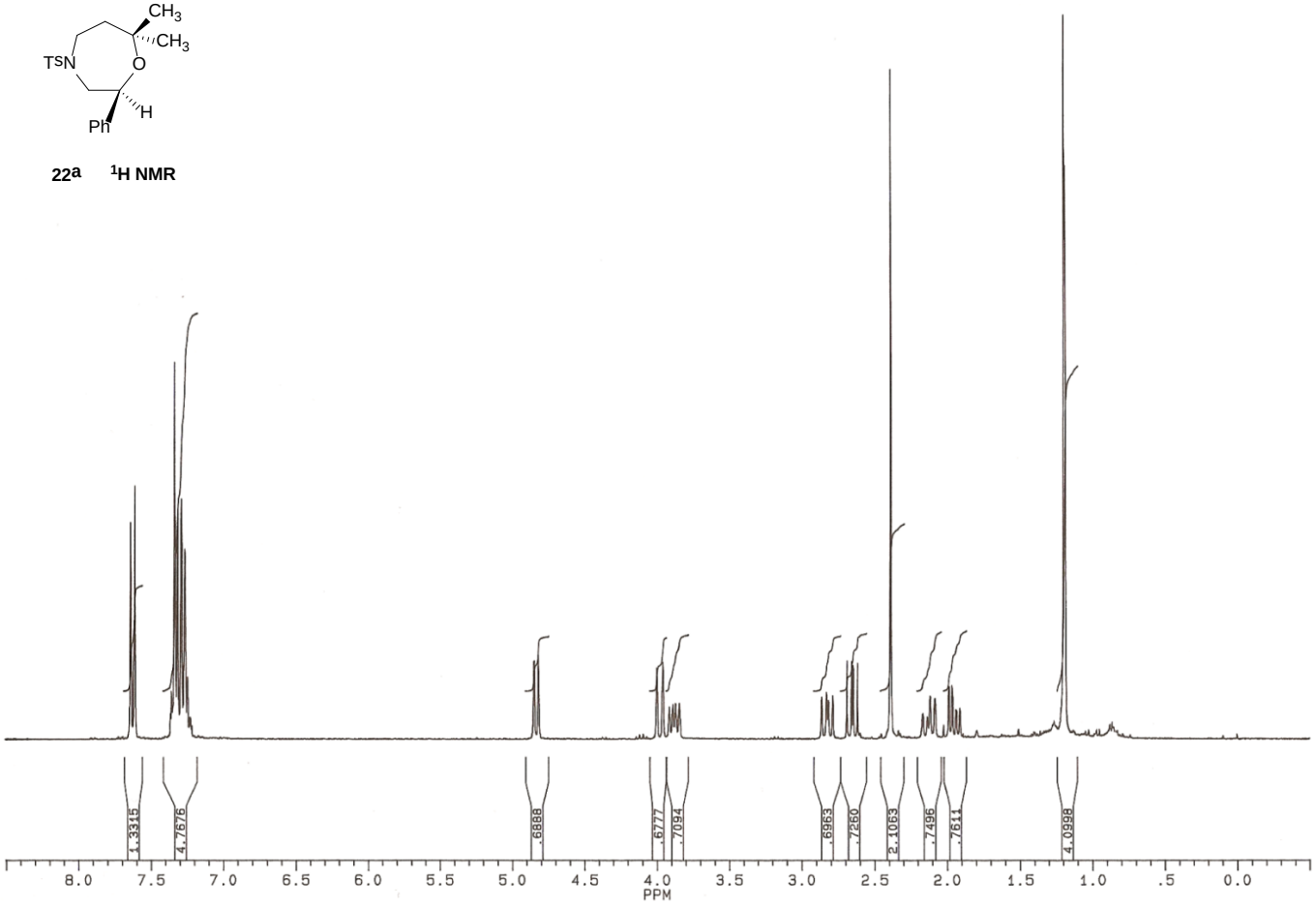
21 ^{1}H NMR (300 K)



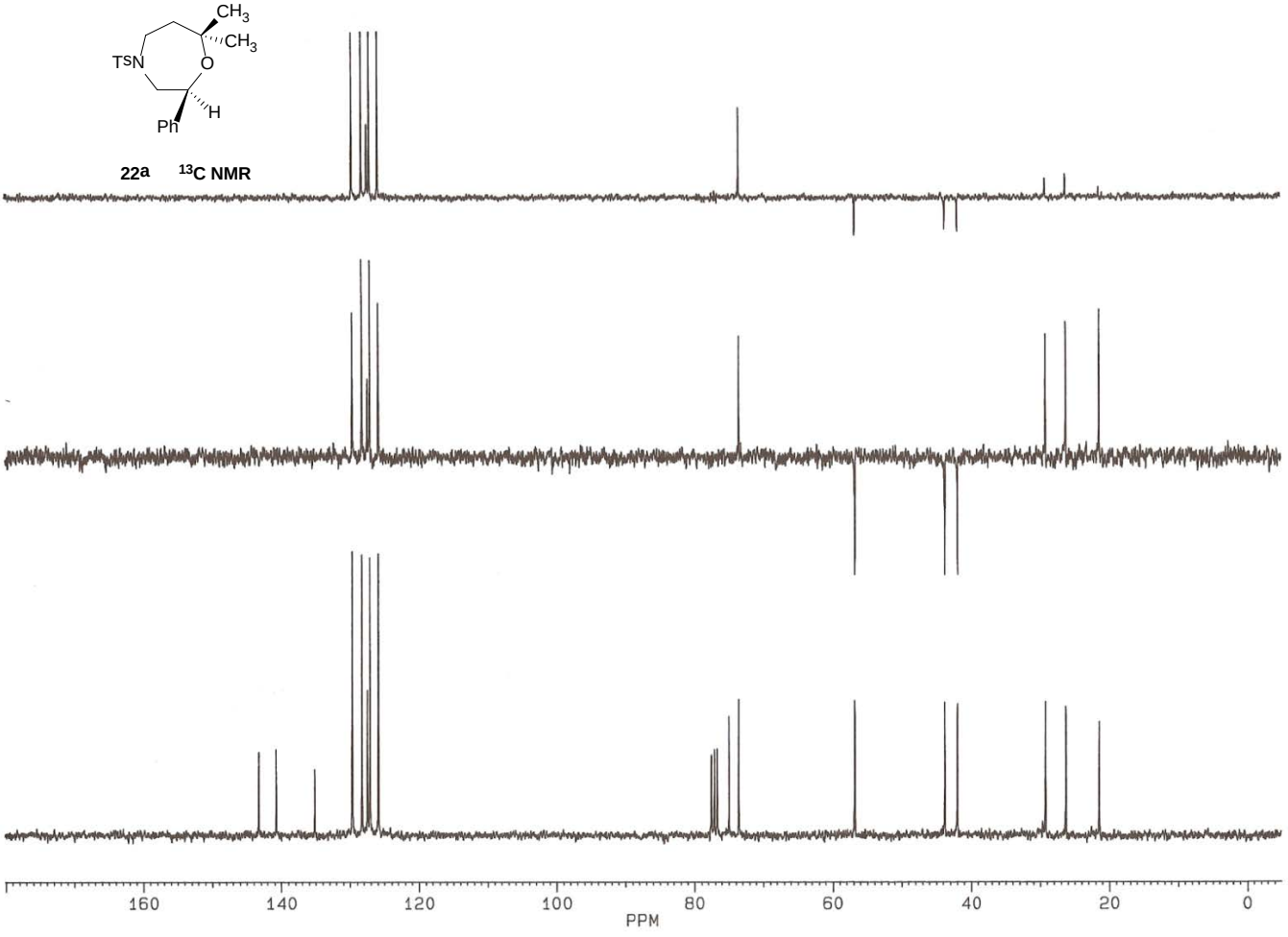
21

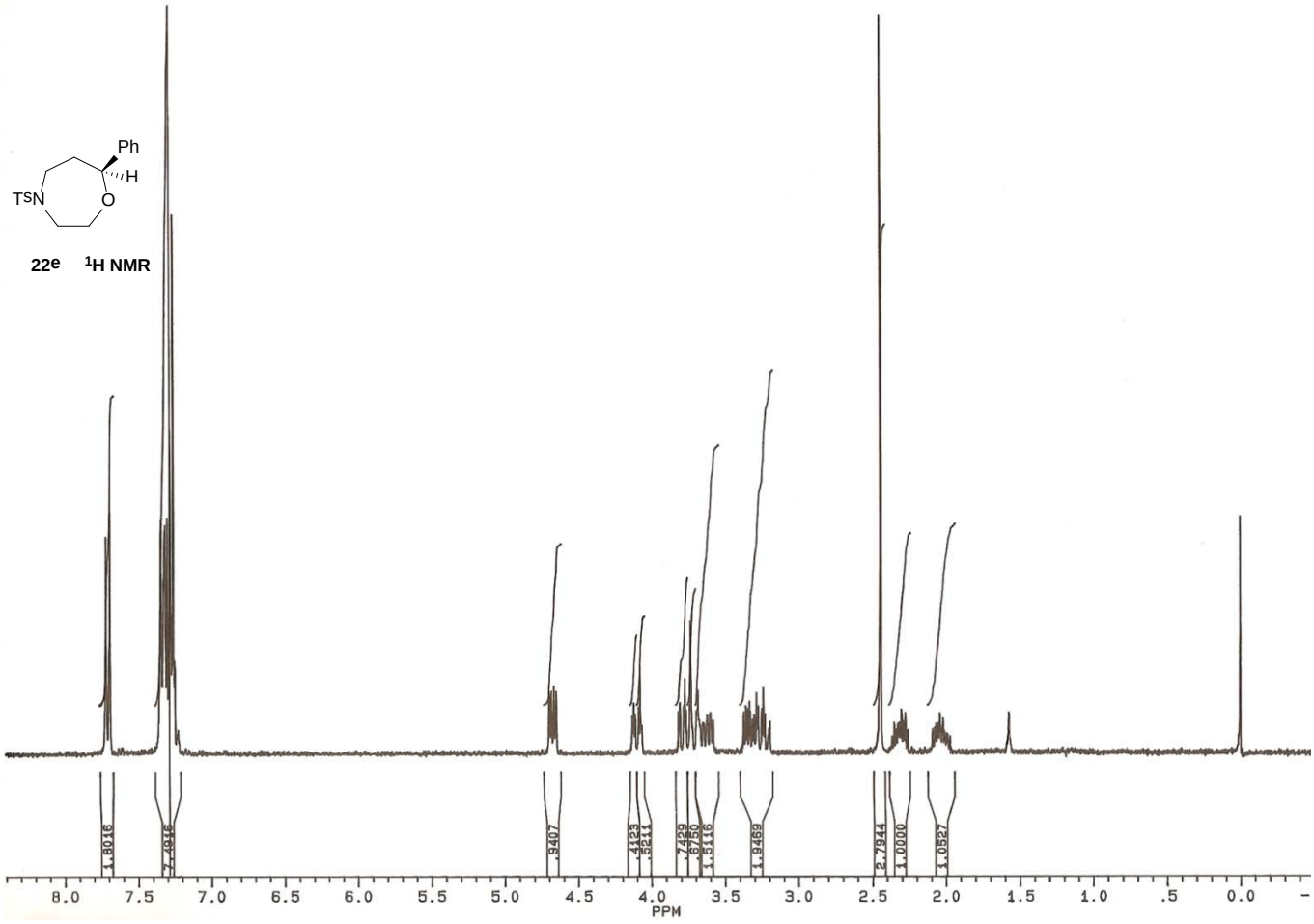
^{13}C NMR DEPT (300 K)

22a **^{1}H NMR**

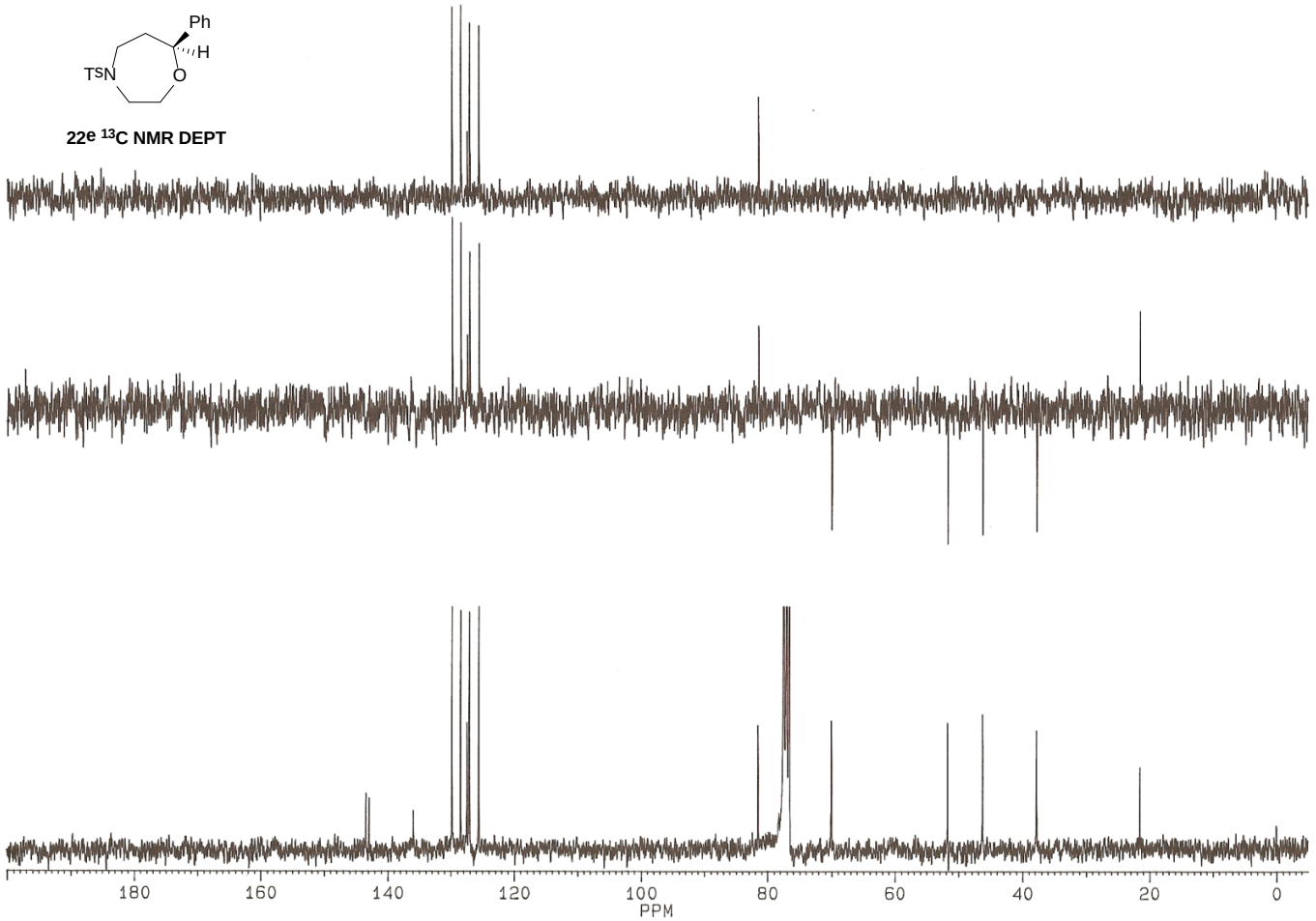


22a **^{13}C NMR**



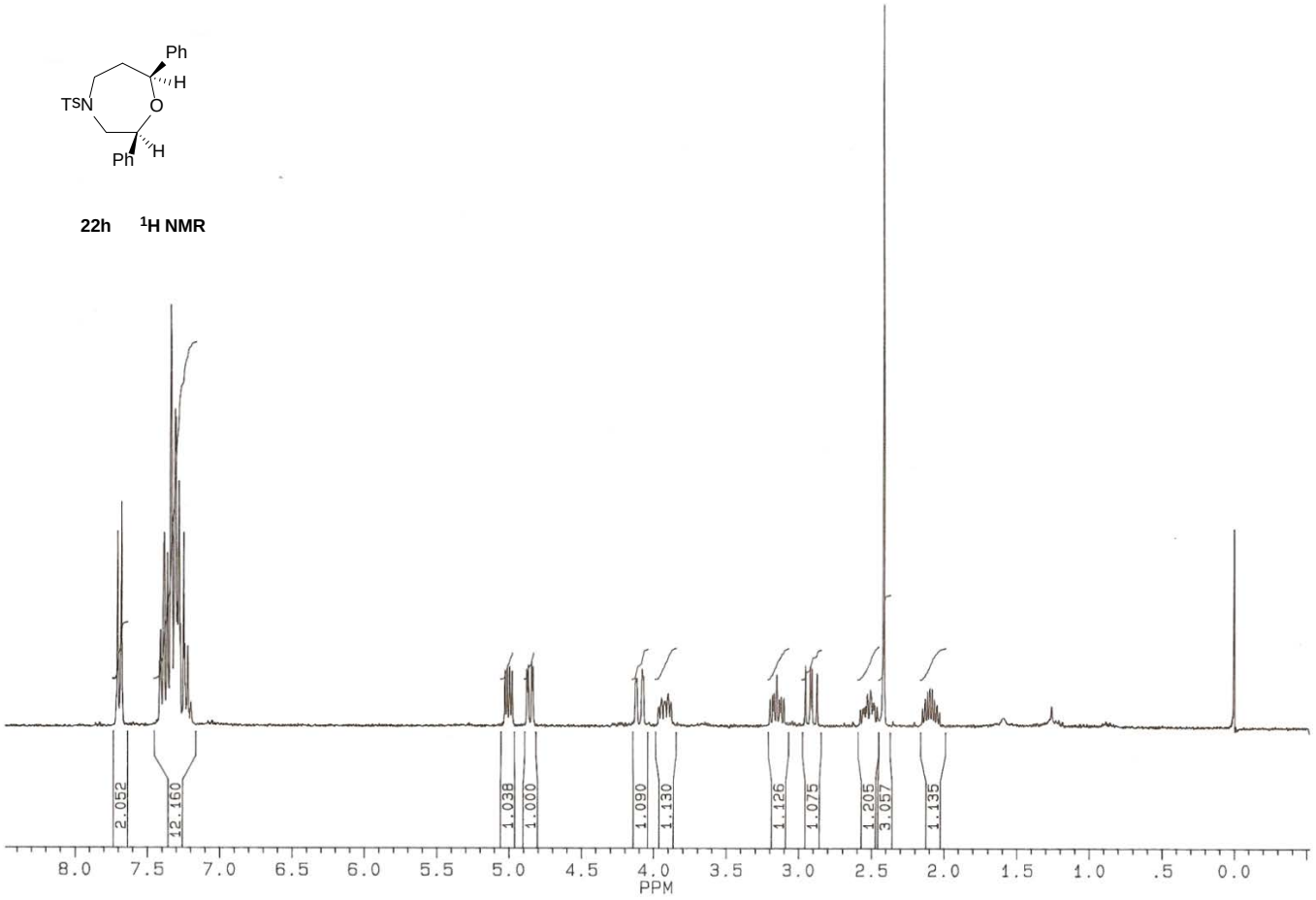


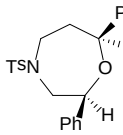
22e ^{1}H NMR



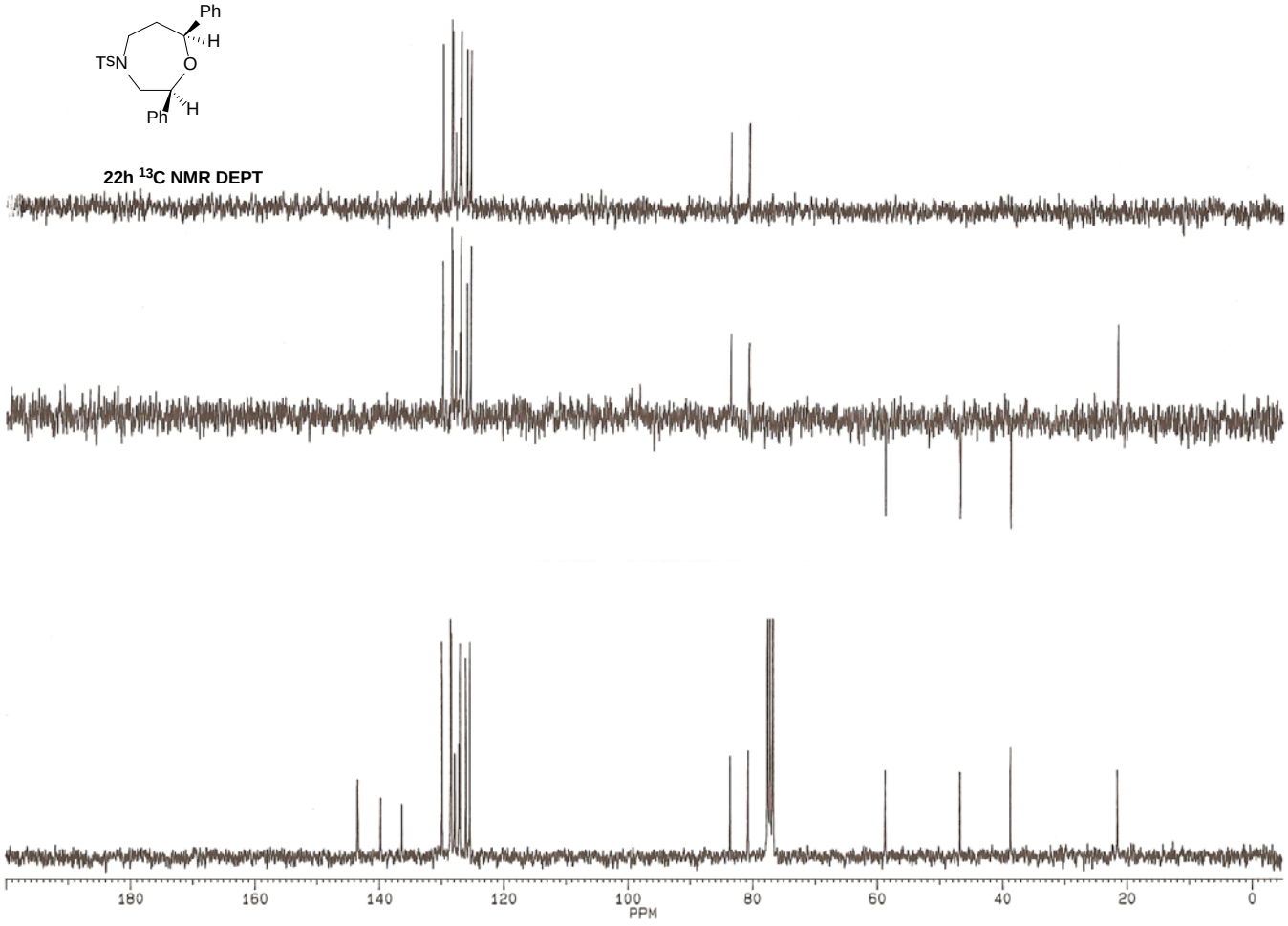
22e ^{13}C NMR DEPT

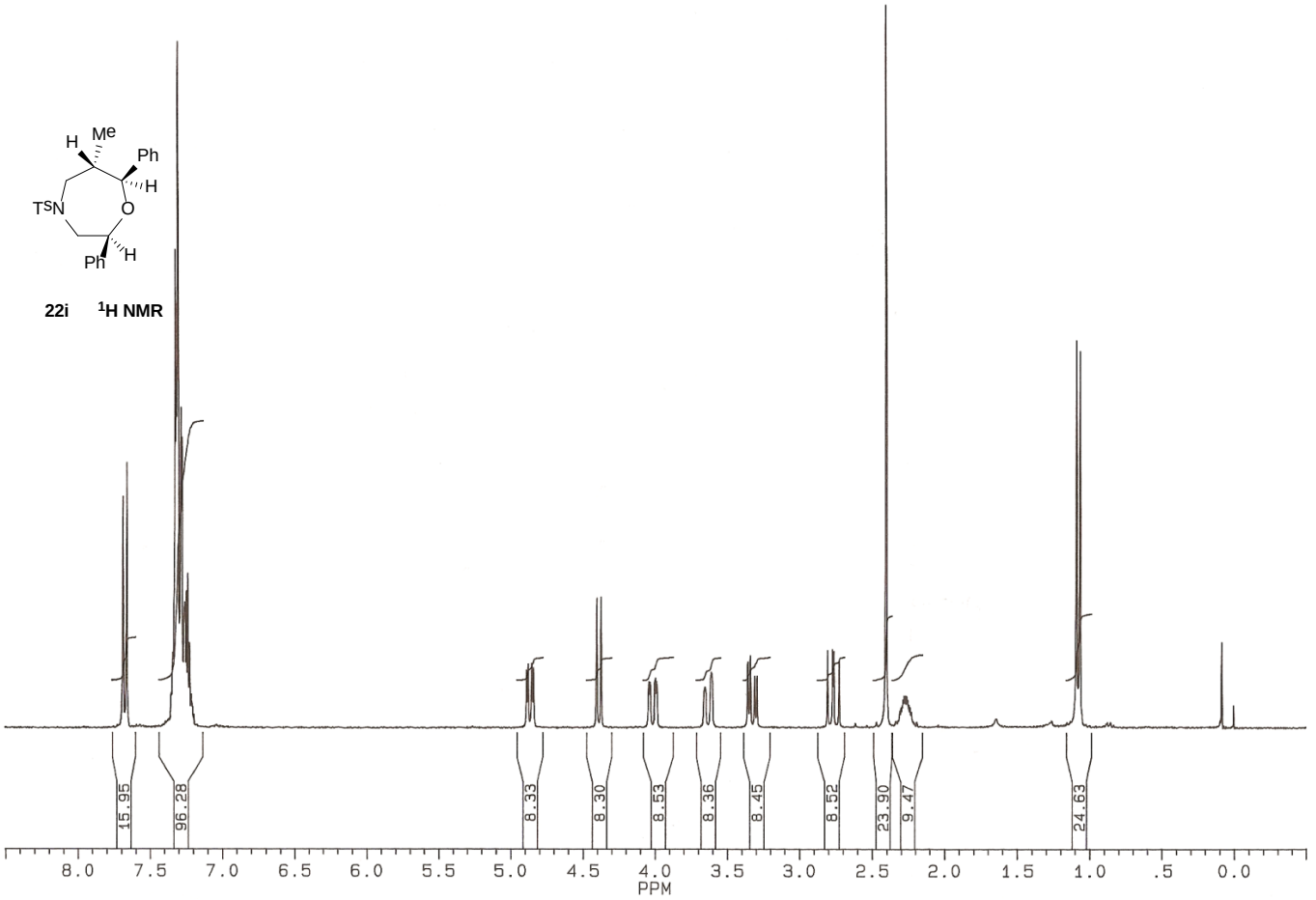
22h ^{1}H NMR



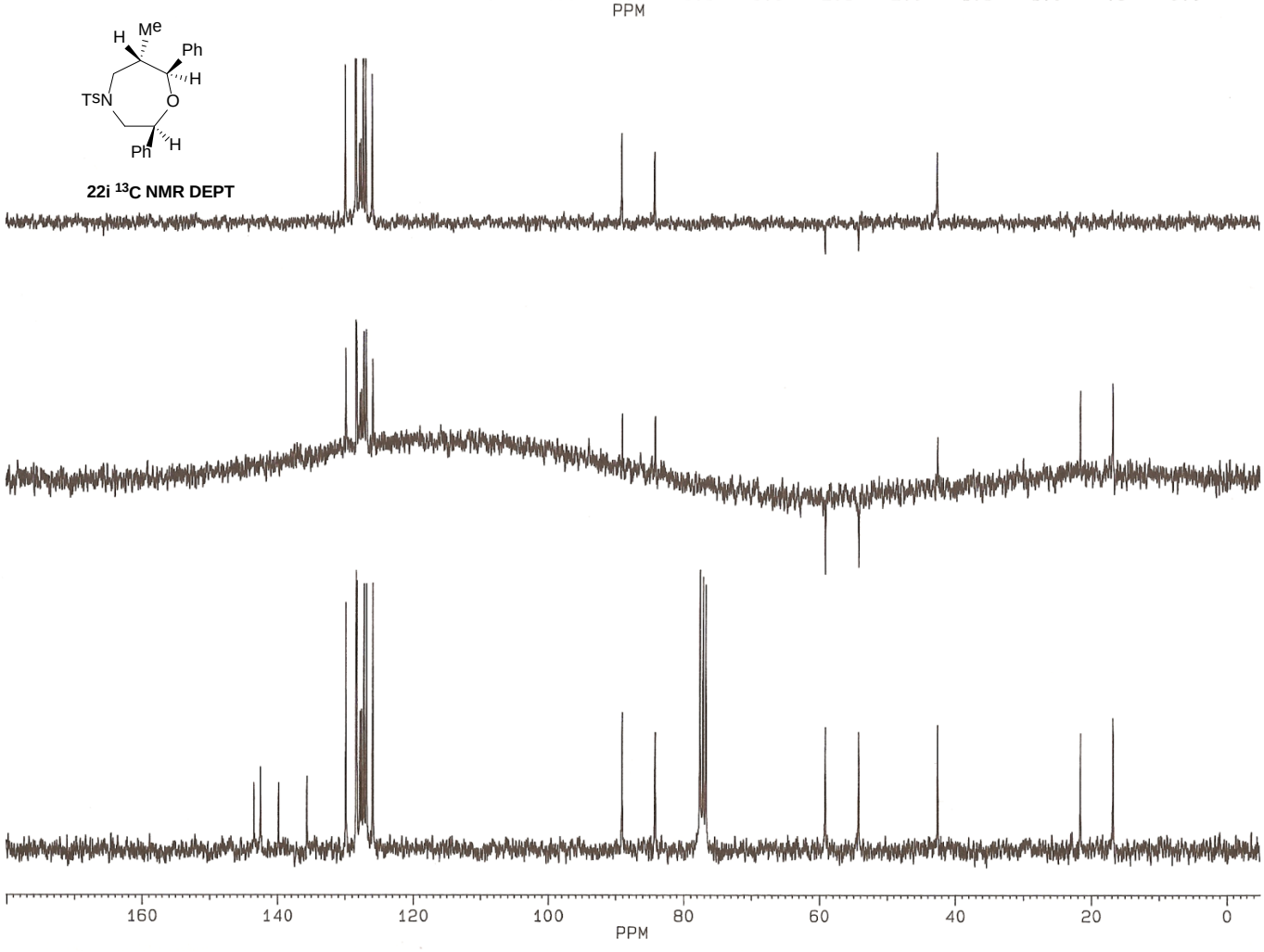


22h ^{13}C NMR DEPT

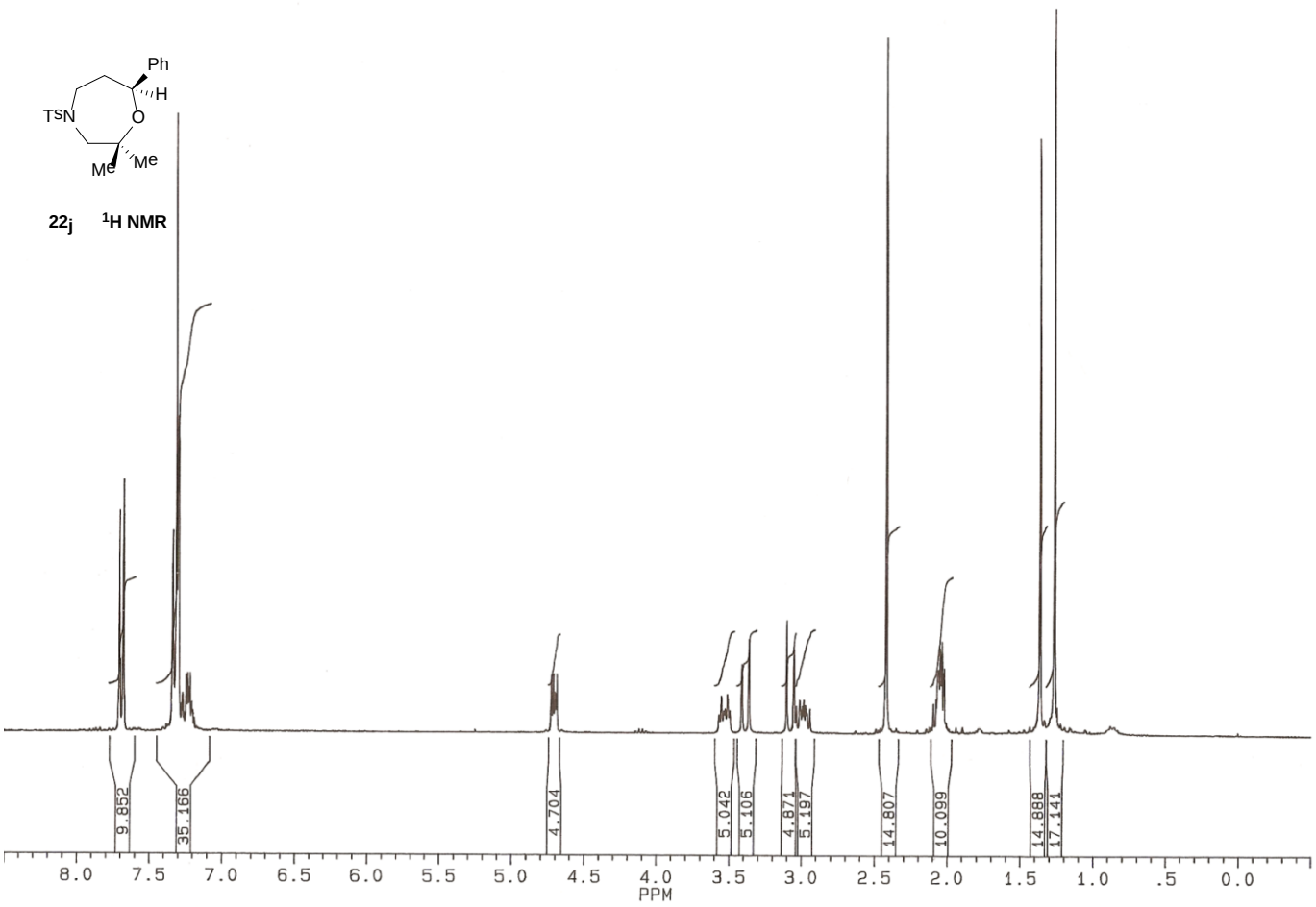


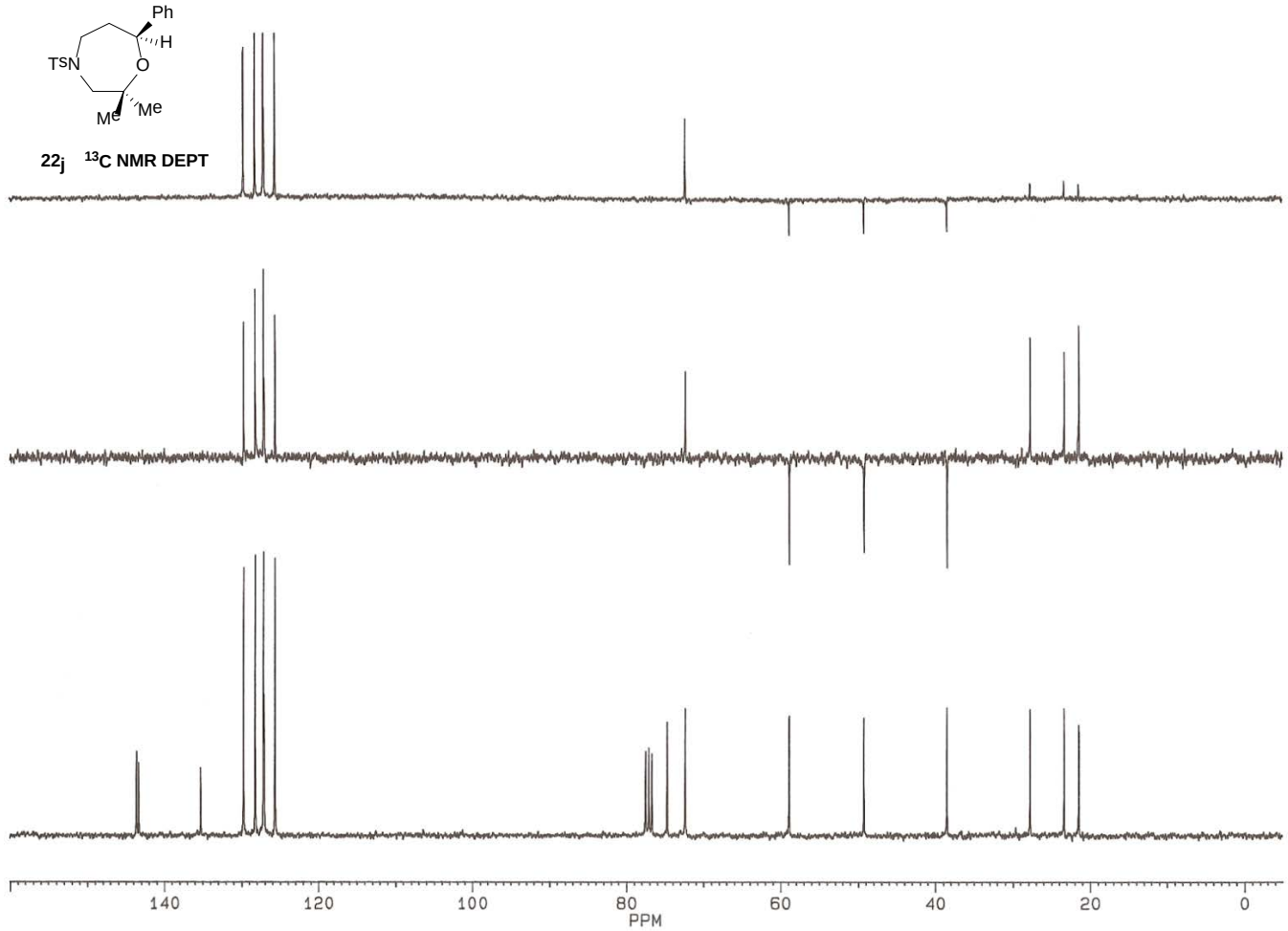


22i **^{1}H NMR**

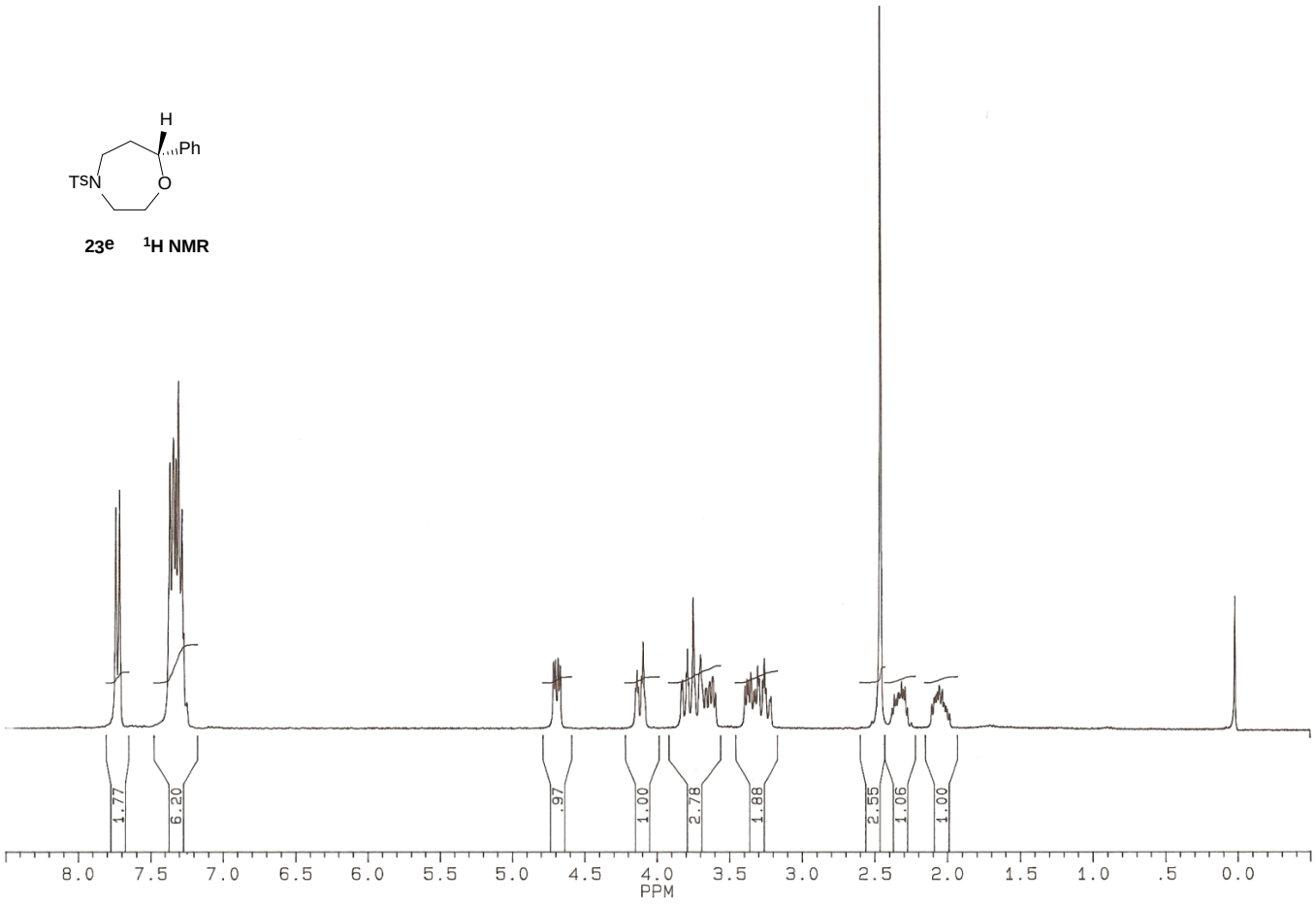


22i ^{13}C NMR DEPT

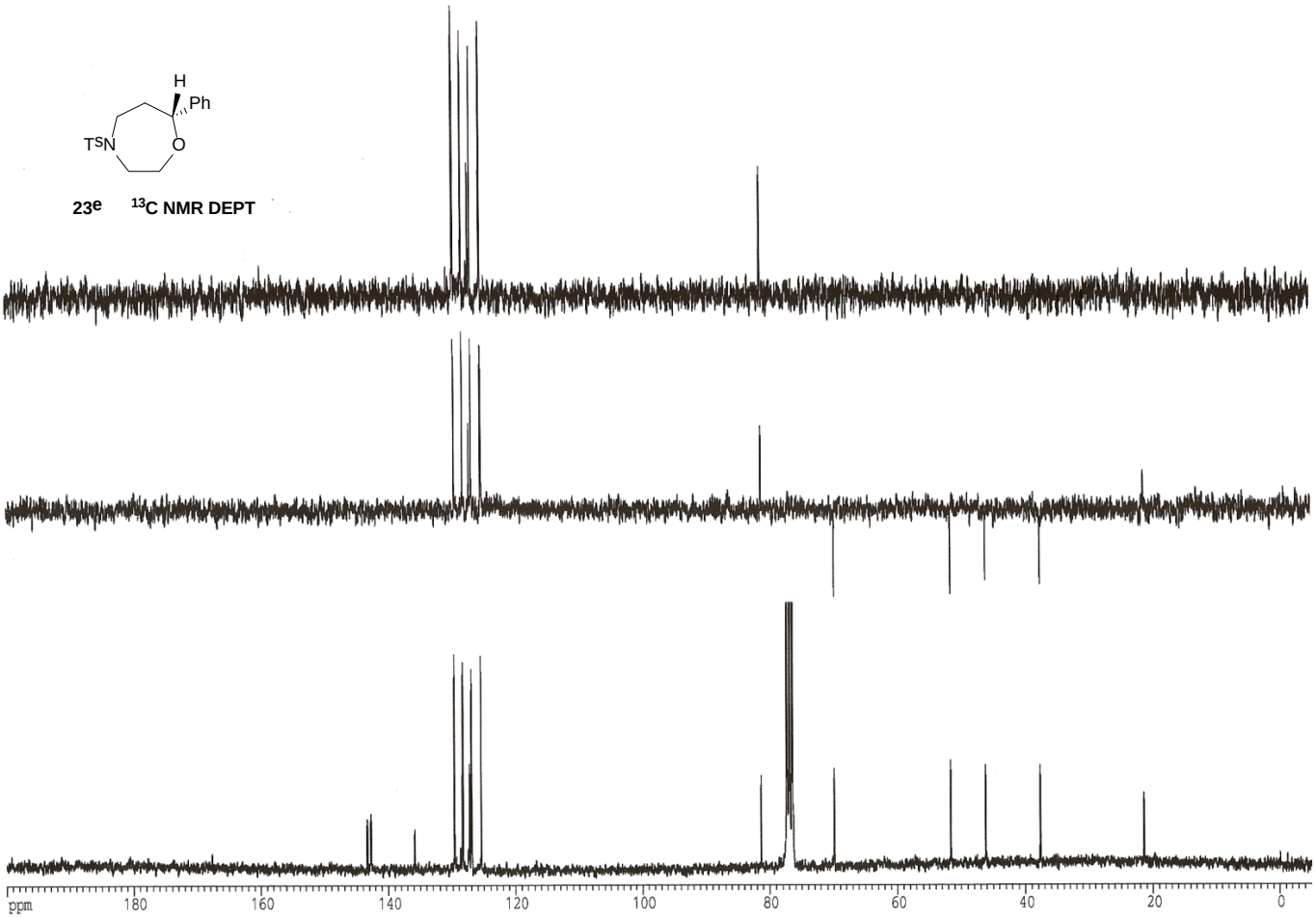

Ph
H
TsN
O
Me
Me
22j 1H NMR
9.852
35.166
4.704
5.042
5.106
4.874
5.197
14.807
10.099
14.888
17.141
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 .5 0.0
PPM

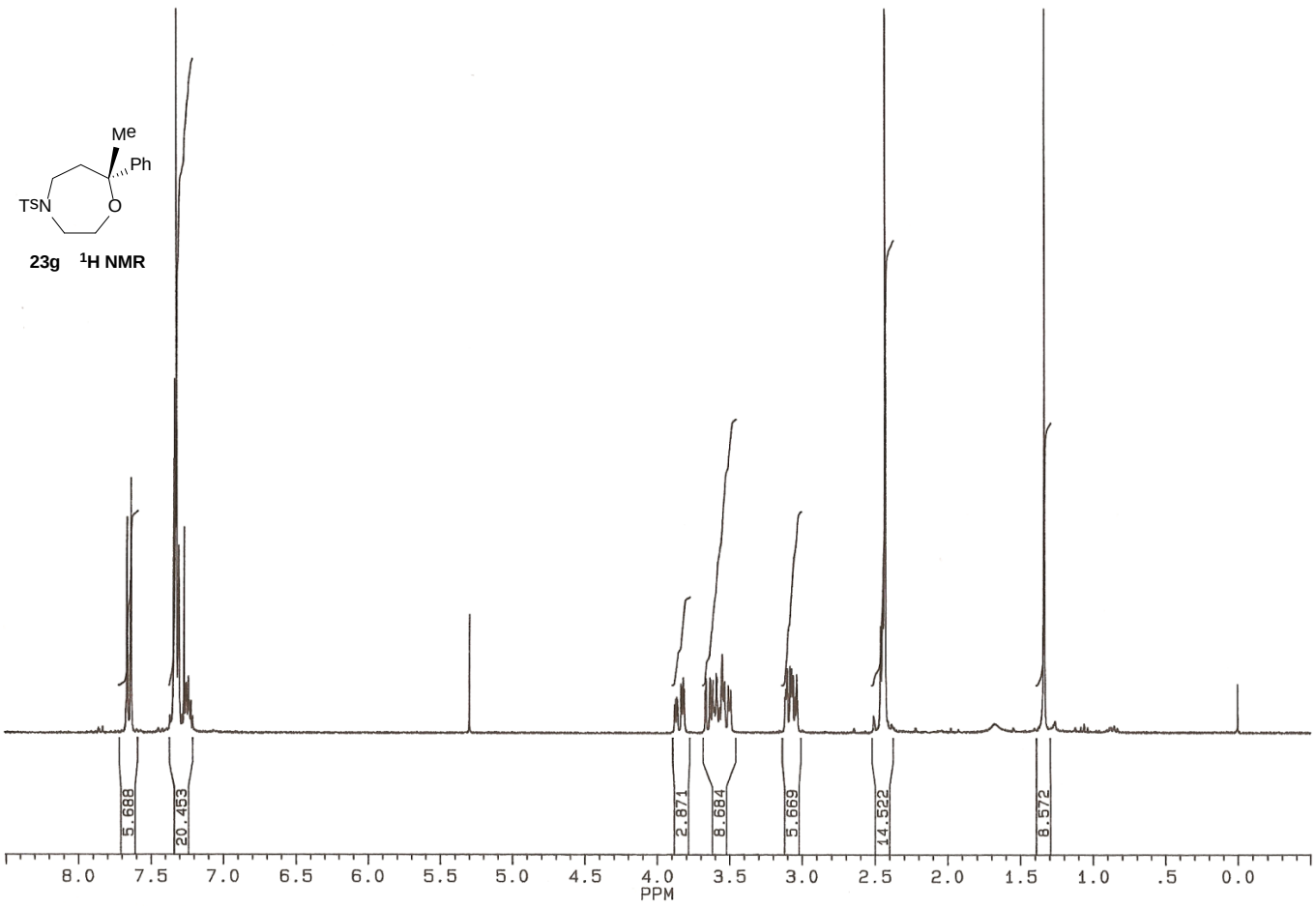

Ph
H
TsN
O
Me
Me
22j 13C NMR DEPT
140 120 100 80 60 40 20 0
PPM

23e **^{1}H NMR**

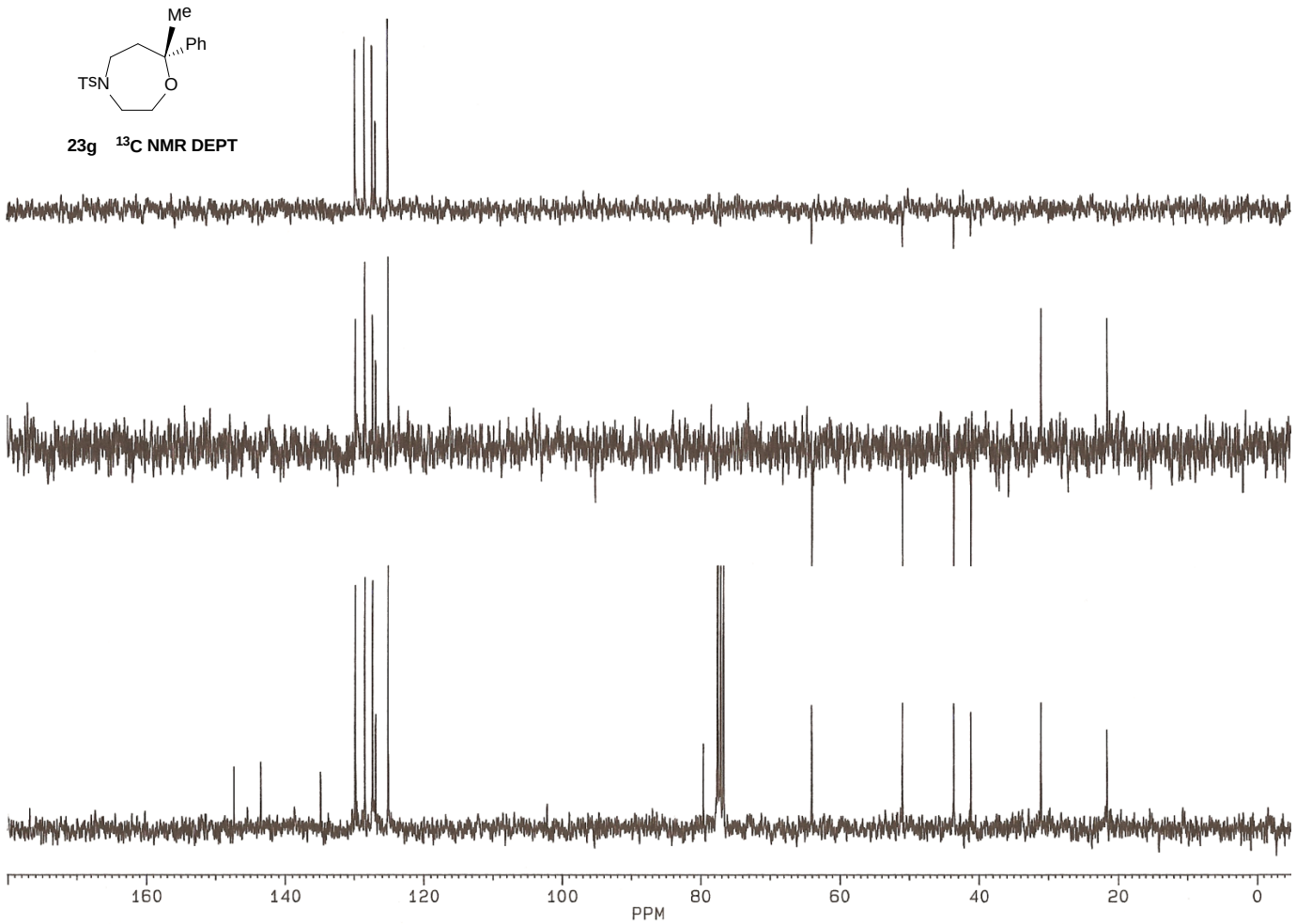


23e **^{13}C NMR DEPT**



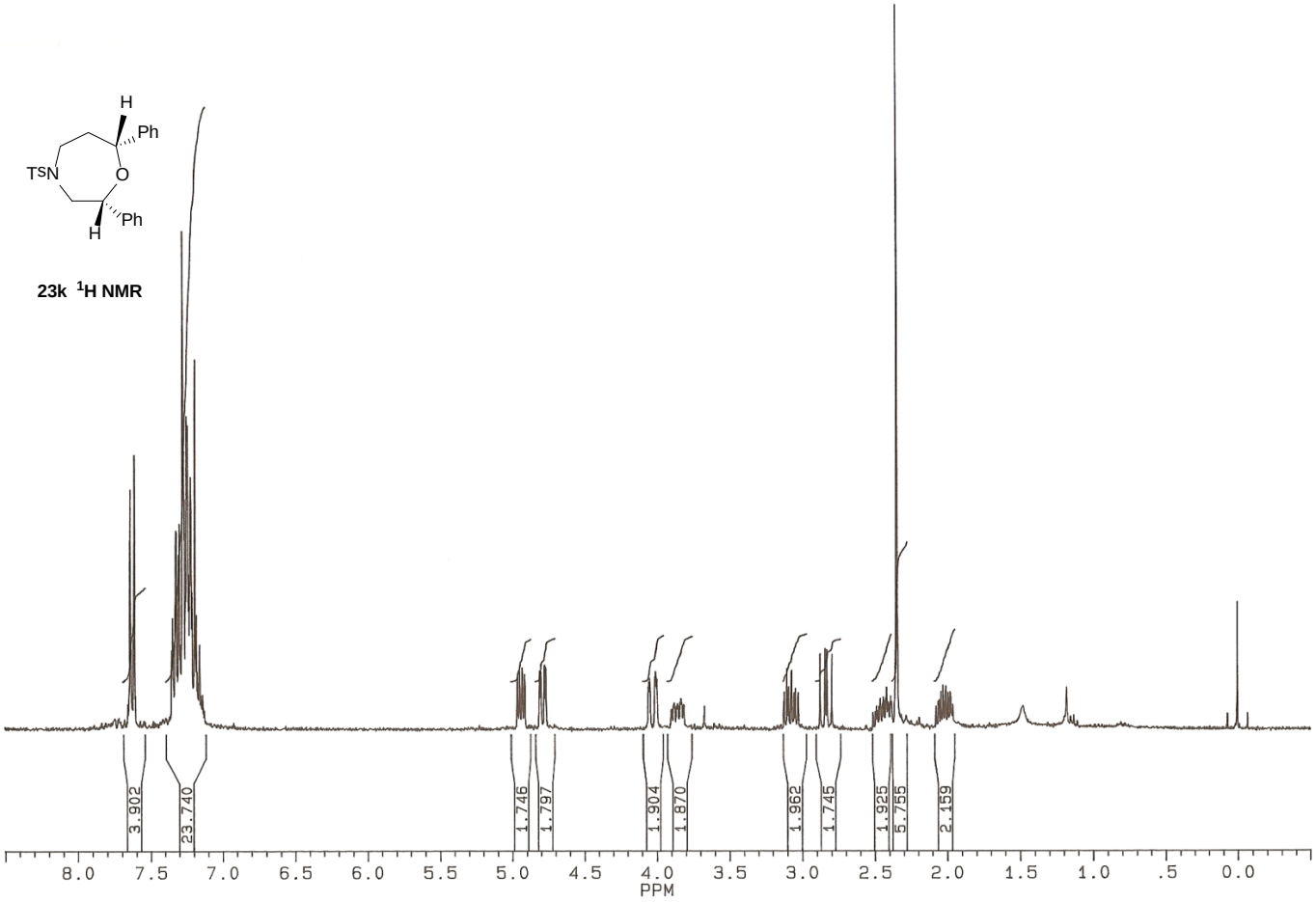


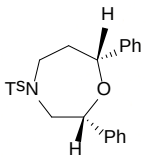
23g ^{1}**H NMR**



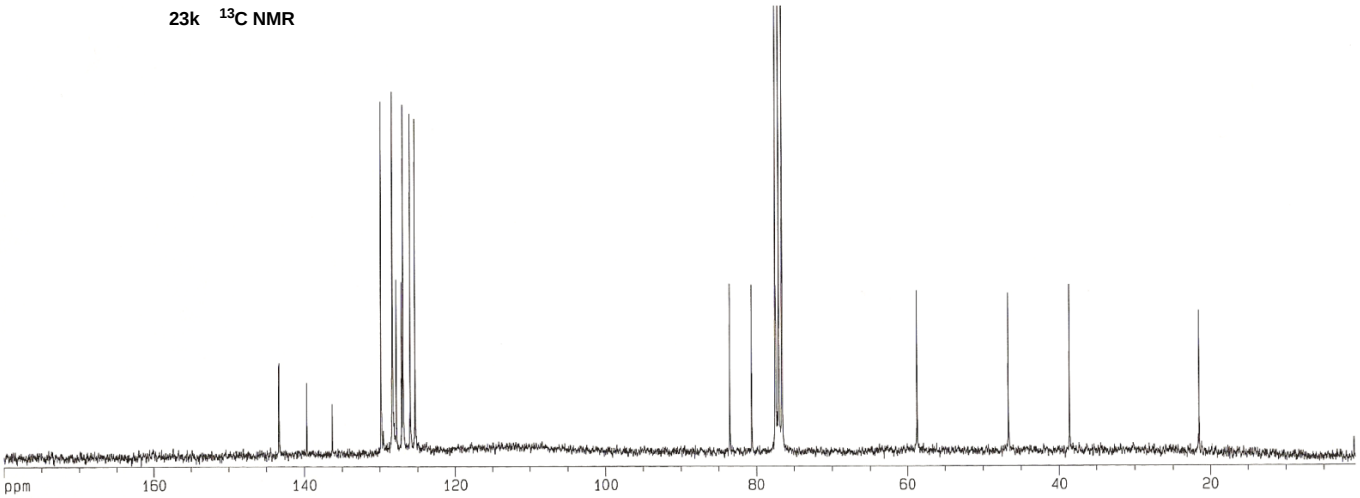
23g ^{13}**C NMR DEPT**

23k ^{1}H NMR

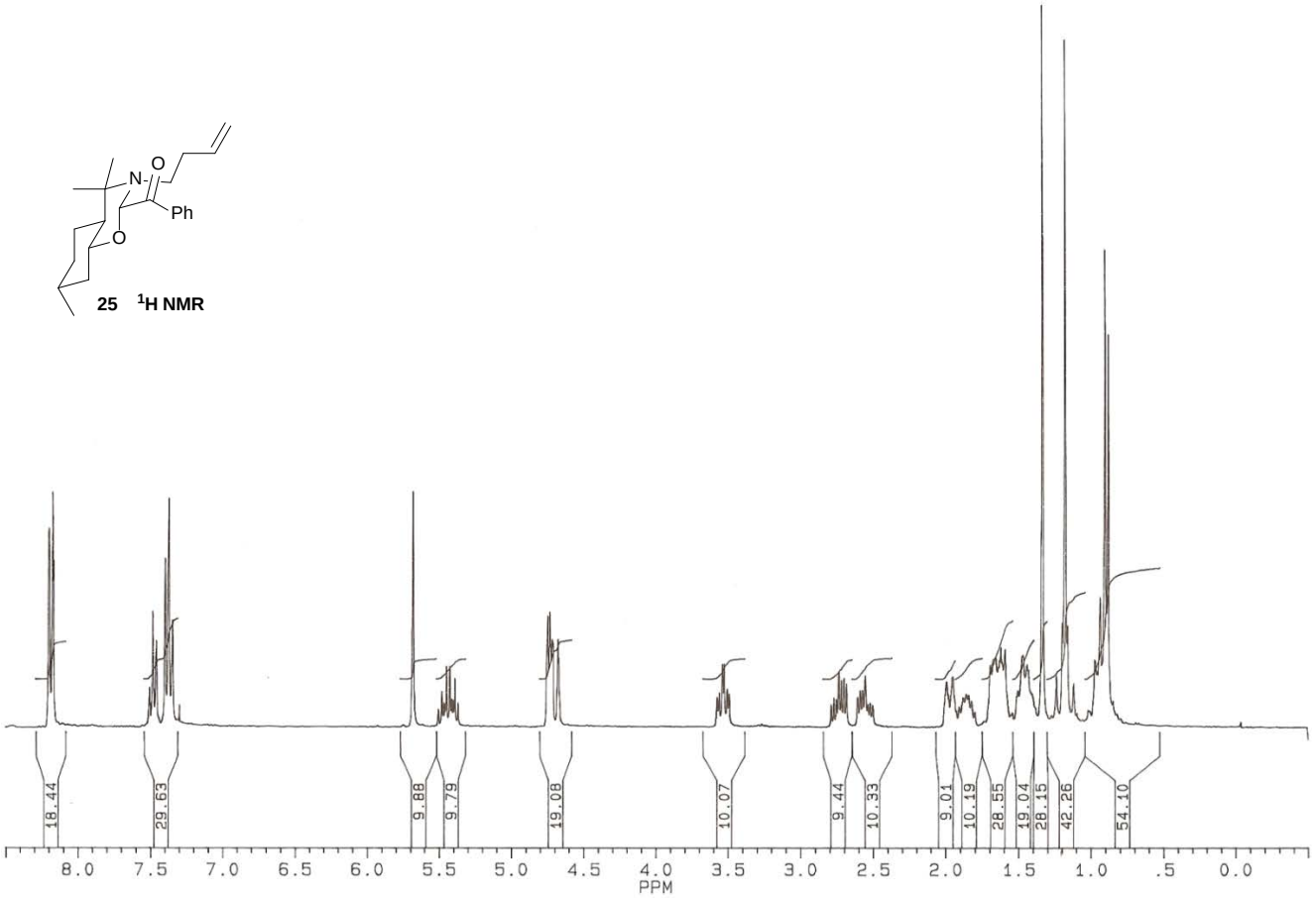




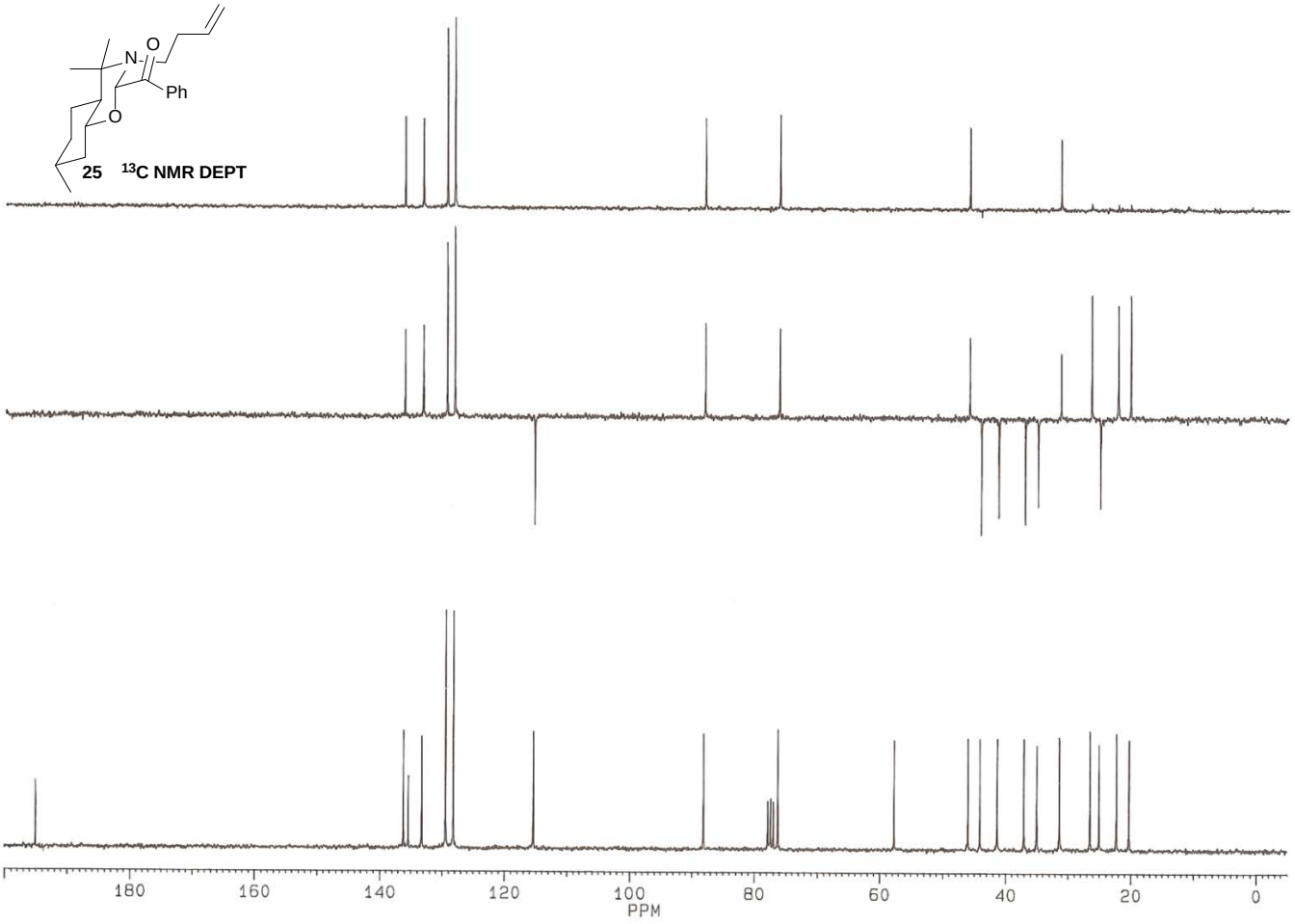
23k ^{13}C NMR



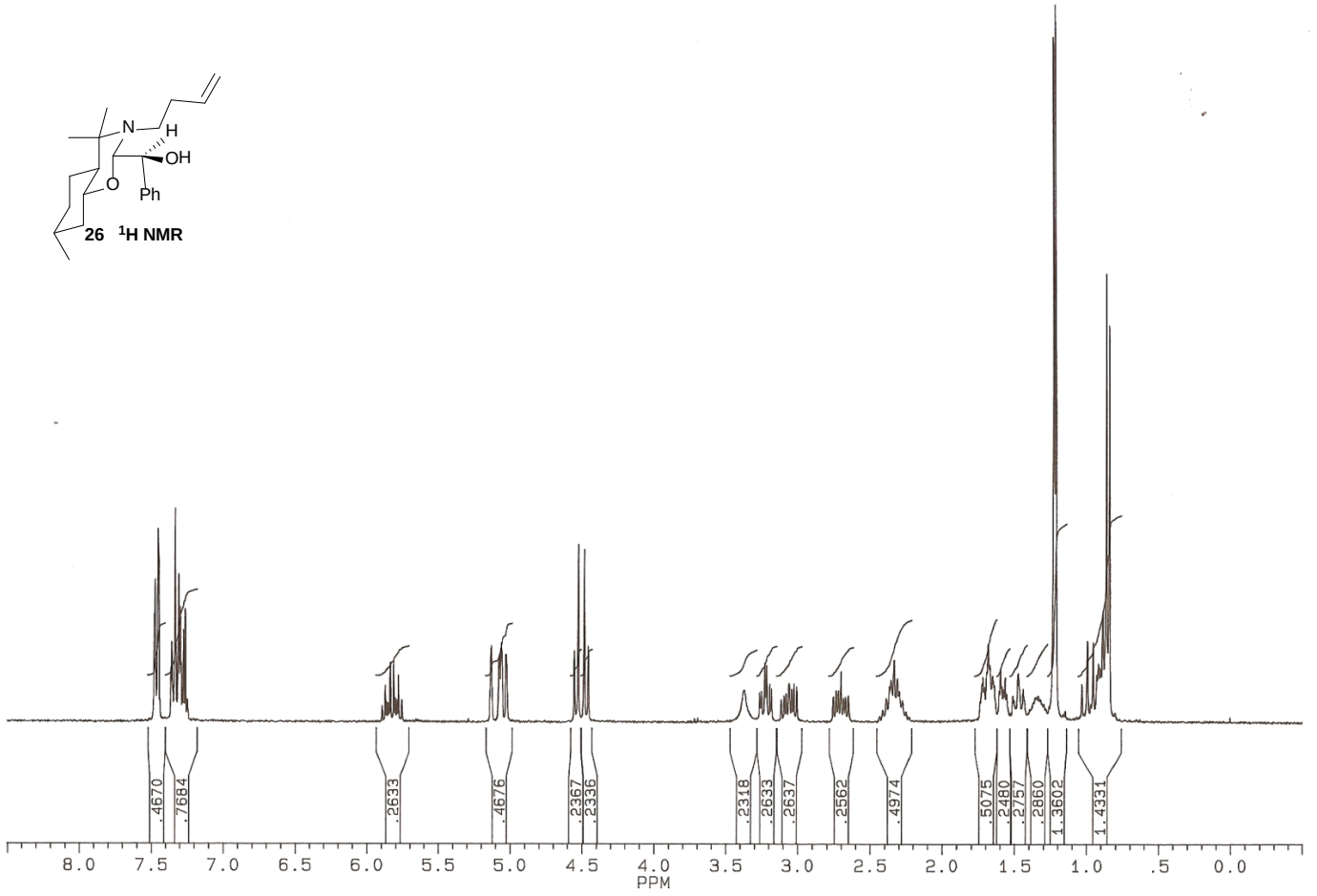
25 ^{1}H NMR



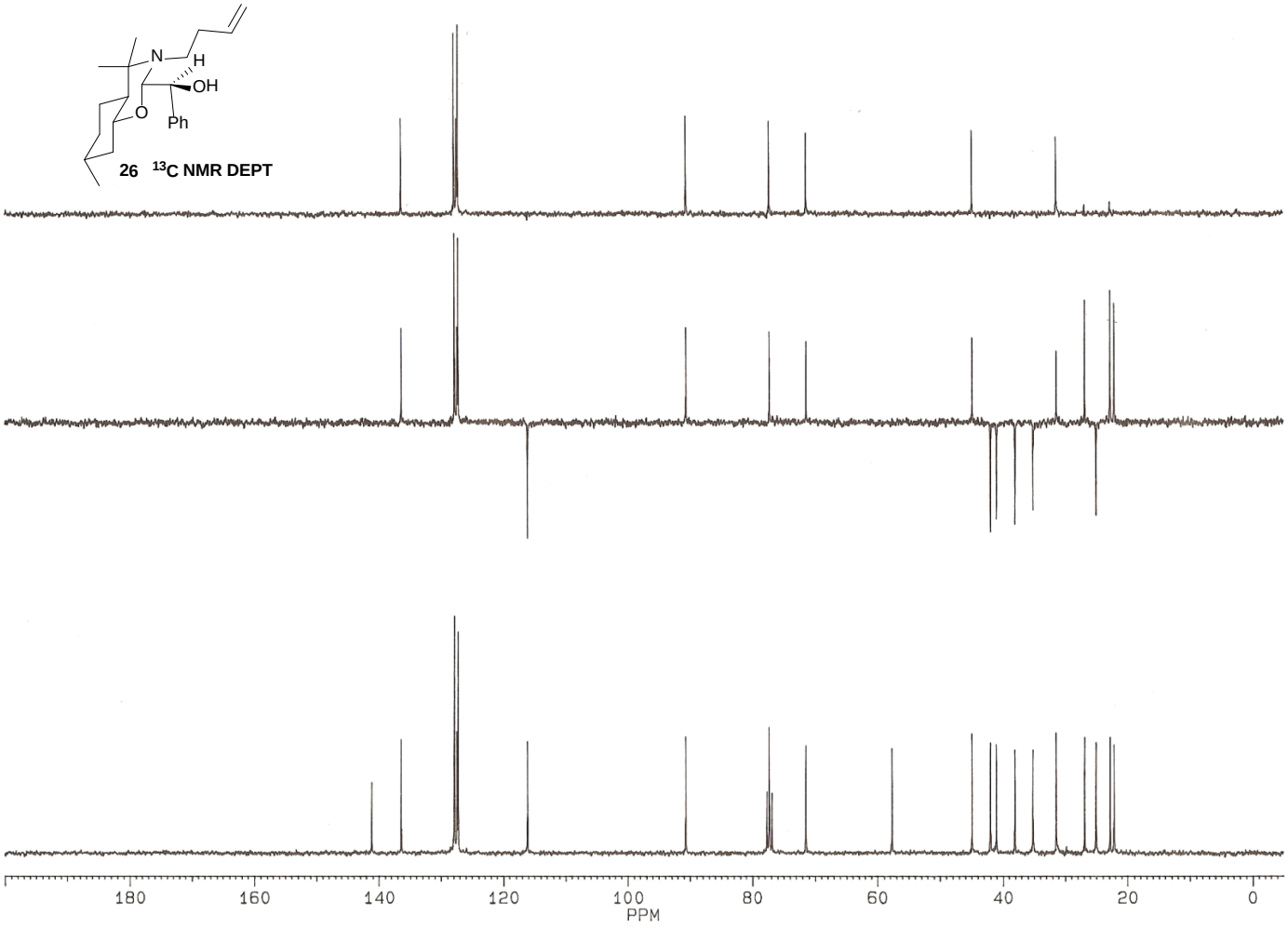
25 ^{13}C NMR DEPT

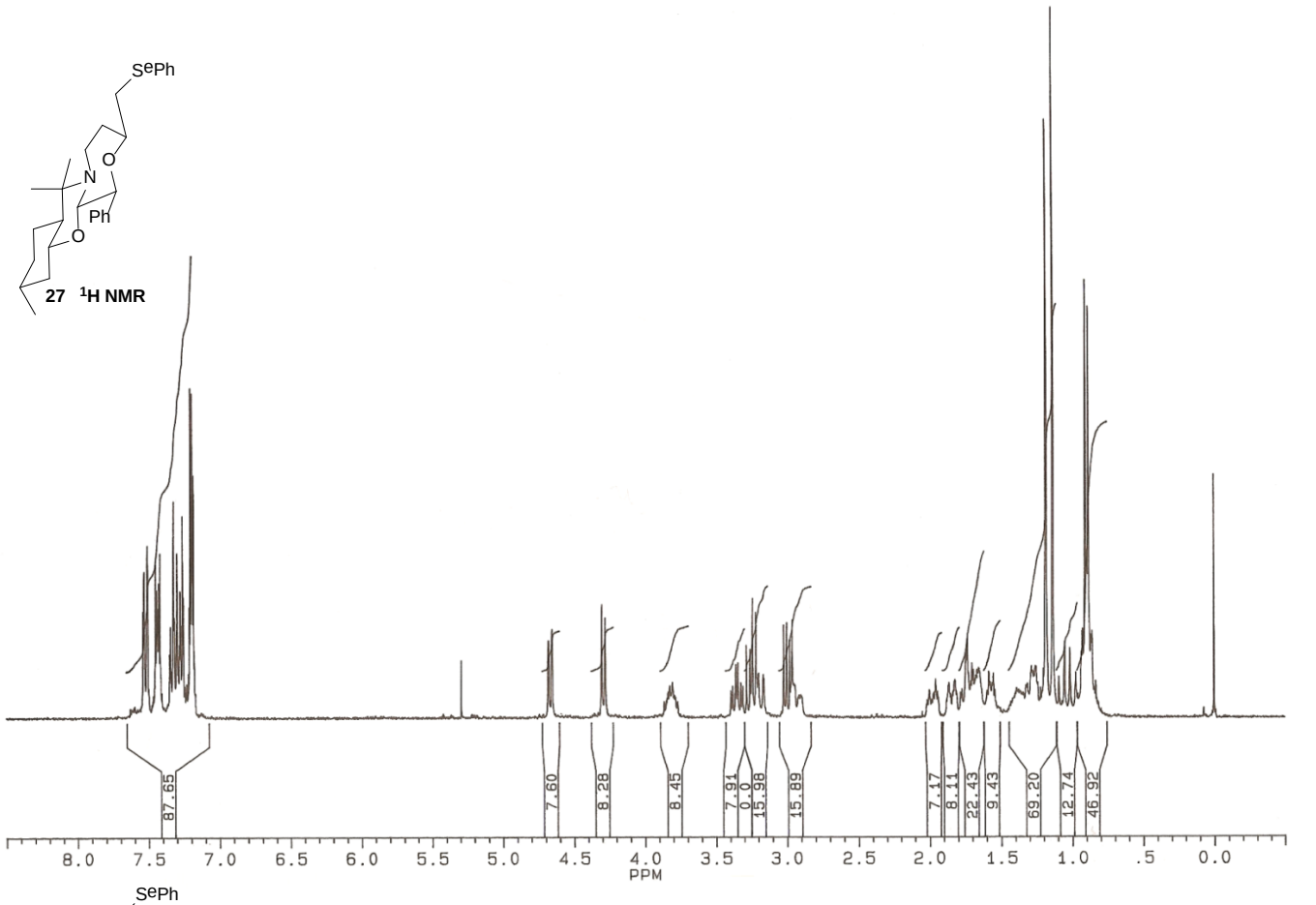


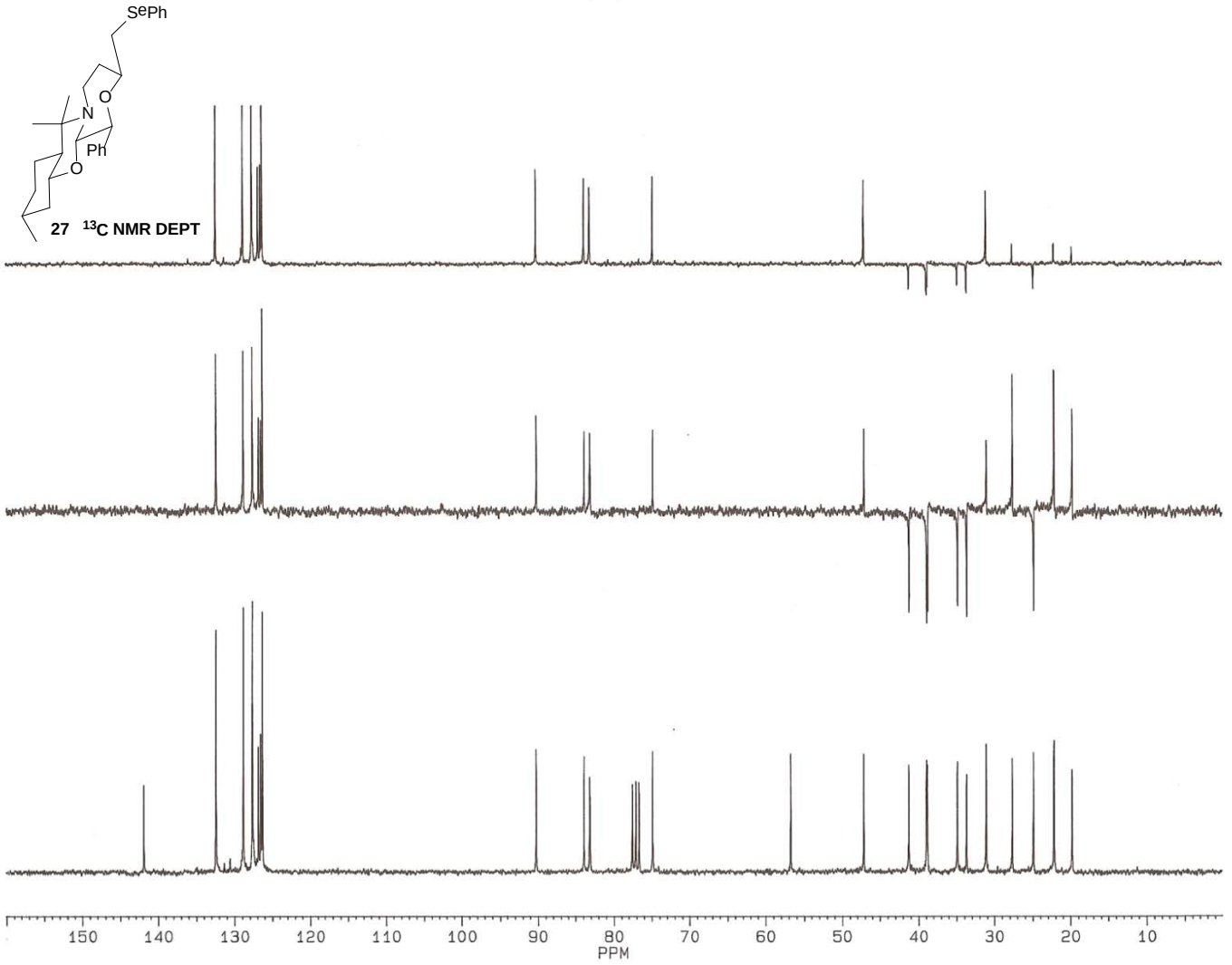
26 ^{1}H NMR

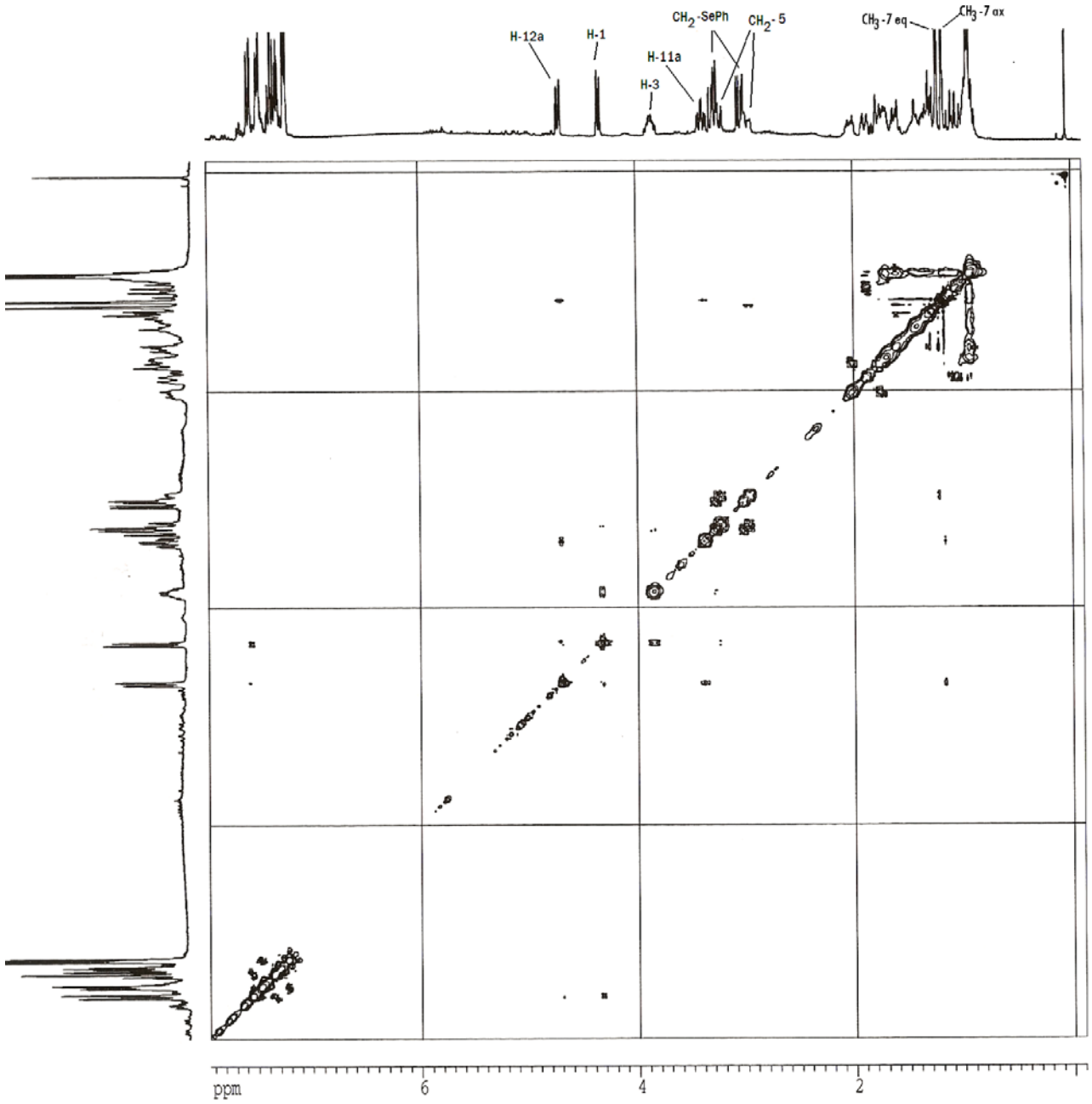


26 ^{13}C NMR DEPT




SePh
O
N
Ph
O
27 1H NMR
87.65
7.60
8.28
8.45
7.91
0.00
15.98
15.89
7.17
8.11
22.43
9.43
69.20
12.74
46.92
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 .5 0.0
PPM


SePh
O
N
Ph
O
27 13C NMR DEPT
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
PPM



Current Data Parameters

NAME	pm131002
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	980913
Time	11.07
PULPROG	noesytp
SOLVENT	CDCl3
AQ	0.2119880 sec
FIDRES	2.358847 Hz
DW	207.0 usec
RG	1024
NUCLEUS	1H
D1	2.0000000 sec
P1	6.6 usec
D0	0.0000030 sec
D8	0.8000000 sec
DE	258.8 usec
SFO1	300.1345329 MHz
SWH	2415.46 Hz
TD	1024
NS	40
DS	2
IN0	0.0002070 sec

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	300.1345 MHz
FIDRES	9.435362 Hz
SW	8.048 ppm

F1 - Processing parameters

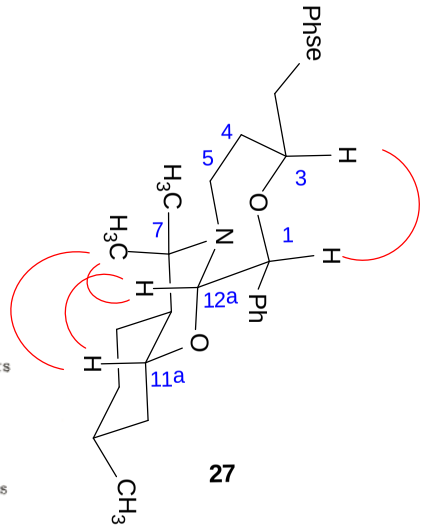
SI	512
MC2	QF
SF	300.1333554 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

F1 - Processing parameters

SI	512
MC2	TPPI
SF	300.1333554 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

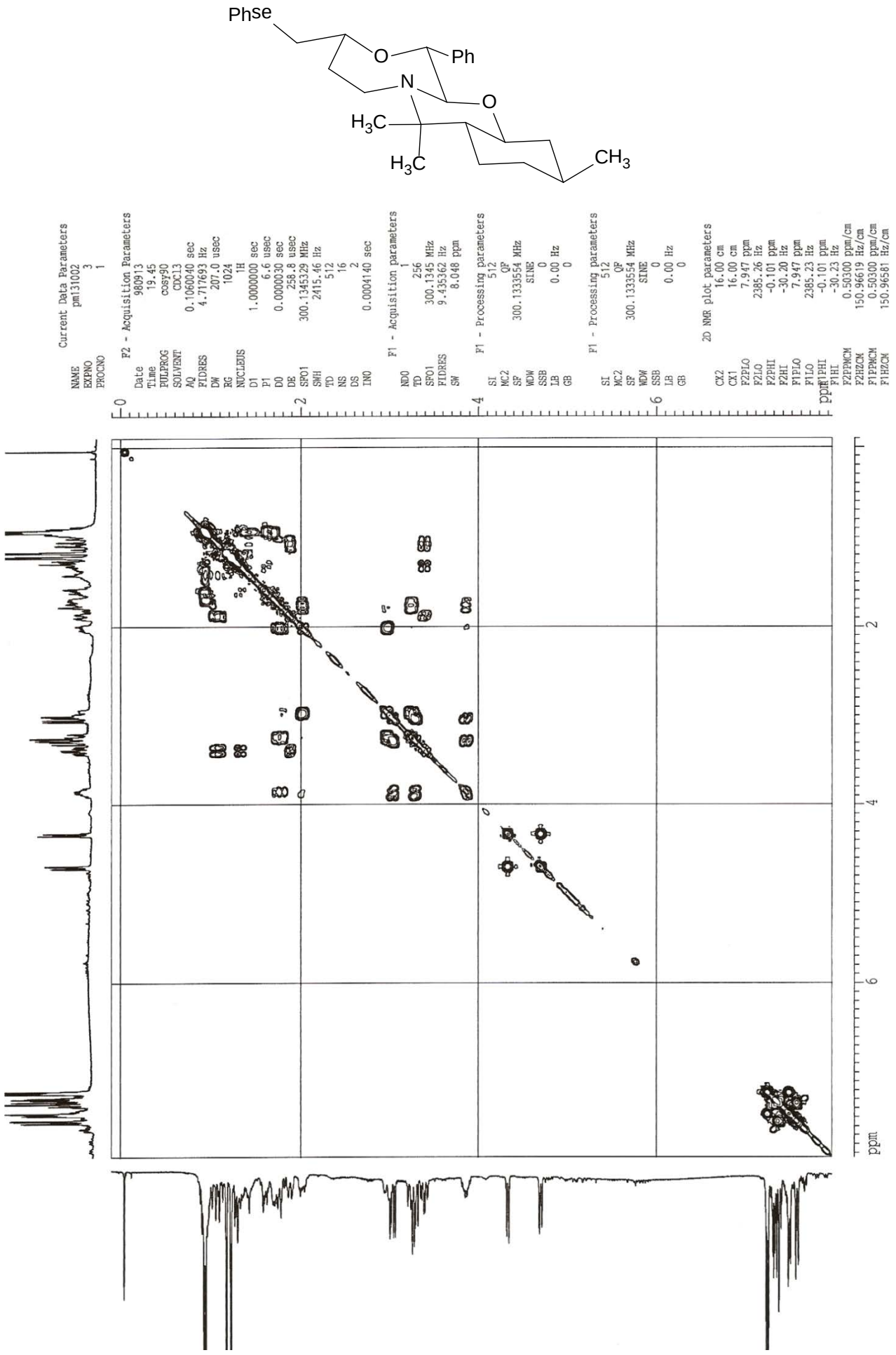
2D NMR plot parameters

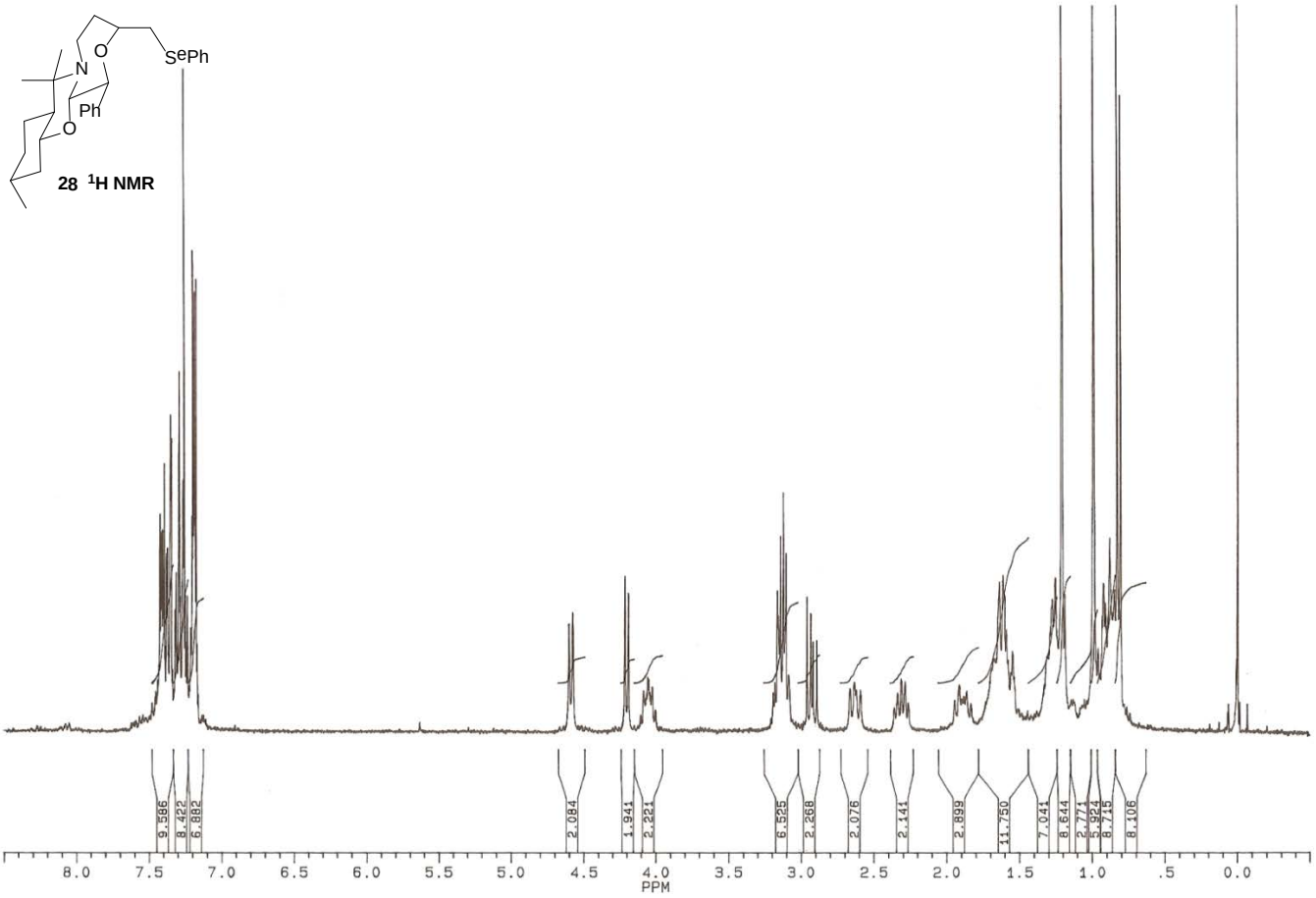
CX2	16.00 cm
CX1	16.00 cm
F2PLO	7.947 ppm
F2LO	2385.26 Hz
F2PHI	-0.101 ppm
F2HI	-30.20 Hz
F1PLO	7.947 ppm
F1LO	2385.23 Hz
F1PHI	-0.101 ppm
F1HI	-30.23 Hz
F2PPMCM	0.50300 ppm/cm
F2HZCM	150.96619 Hz/cm
F1PPMCM	0.50300 ppm/cm
F1HZCM	150.96581 Hz/cm

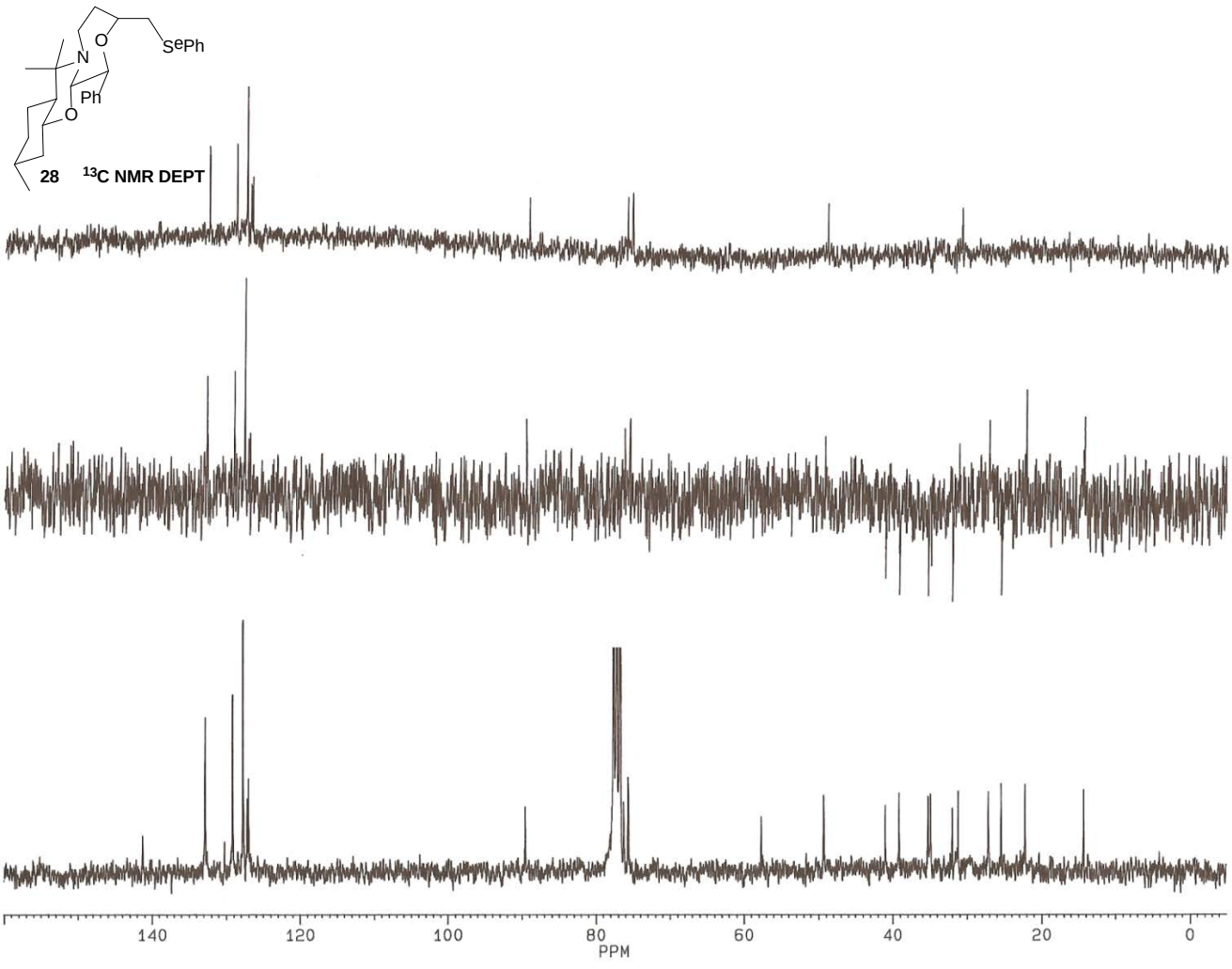


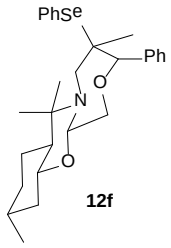
27

^{1}H NOESY contacts

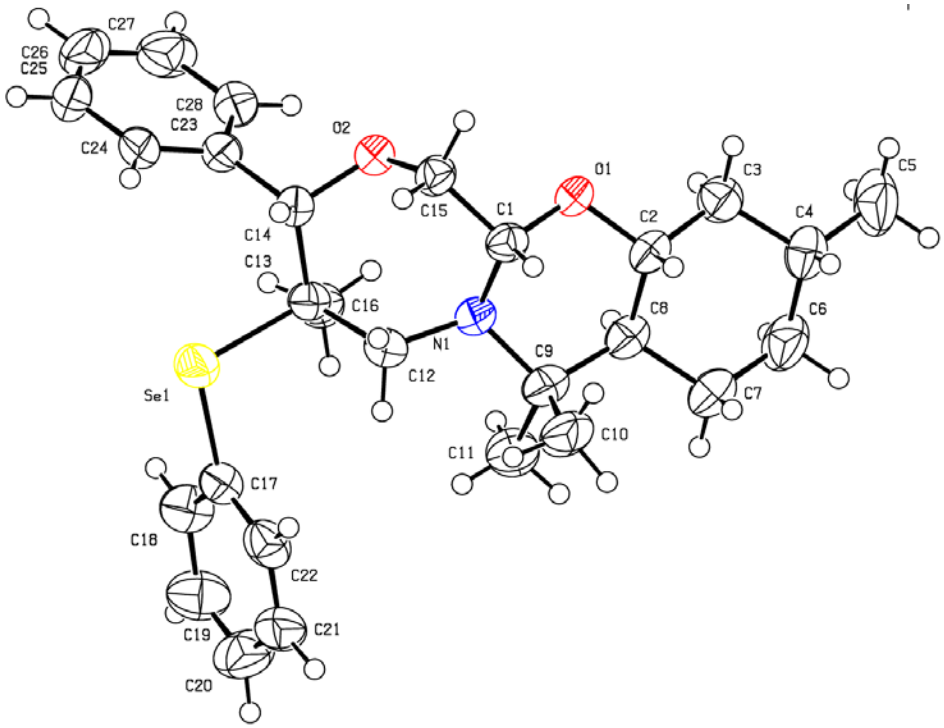

PhSe
O
Ph
N
O
H3C
H3C
CH3
Current Data Parameters
NAME pm131002
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date 980913
Time 19.45
PULPROG cosy90
SOLVENT CDCl3
AQ 0.1060040 sec
FIDRES 4.717693 Hz
DW 207.0 usec
RG 1024
NUCLEUS 1H
D1 1.0000000 sec
P1 6.6 usec
D0 0.0000030 sec
DE 258.8 usec
SFO1 300.1345329 MHz
SWH 2415.46 Hz
TD 512
NS 16
DS 2
INO 0.0004140 sec
F1 - Acquisition parameters
NDO 1
TD 256
SFO1 300.1345 MHz
FIDRES 9.435362 Hz
SW 8.048 ppm
F1 - Processing parameters
SI 512
MC2 QF
SF 300.1333554 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
F1 - Processing parameters
SI 512
MC2 QF
SF 300.1333554 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 16.00 cm
CX1 16.00 cm
F2PLO 7.947 ppm
F2LO 2385.26 Hz
F2PHI -0.101 ppm
F2HI -30.20 Hz
F1PLO 7.947 ppm
F1LO 2385.23 Hz
F1PHI -0.101 ppm
F1HI -30.23 Hz
F2PPMCM 0.50300 ppm/cm
F2HZCM 150.96619 Hz/cm
F1PPMCM 0.50300 ppm/cm
F1HZCM 150.96581 Hz/cm
ppm

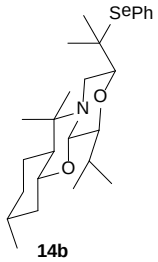

SePh
N
O
Ph
O
28 ^{1}H NMR
9.586
8.422
6.882
2.084
1.941
2.221
6.525
2.268
2.076
2.141
2.899
11.750
7.041
8.644
2.771
2.924
8.715
8.106
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
0.0
PPM


SePh
N
O
Ph
O
28 ^{13}C NMR DEPT
140
120
100
80
60
40
20
0
PPM

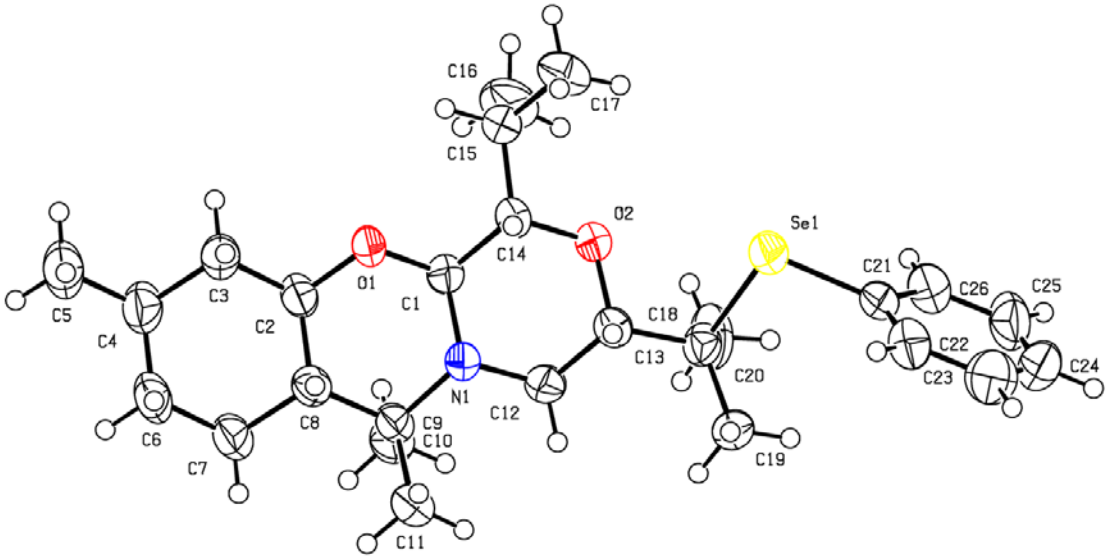

PhSe
Ph
O
N
O

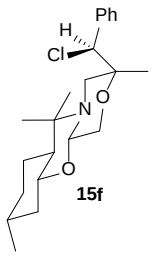
12f


C27
C26
C25
C28
C23
C24
O2
C15
C1
O1
C3
C5
C2
C4
C14
C13
C16
N1
C12
C9
C8
C6
C7
Se1
C11
C10
C17
C18
C22
C19
C21
C20

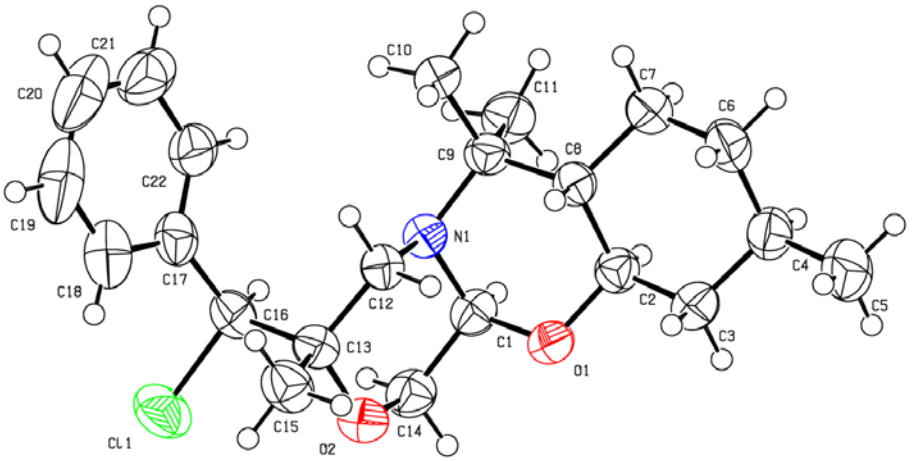

SePh
N
O
O

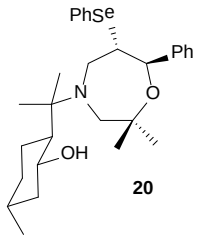
14b


C16
C17
C15
O2
C14
Se1
C21
C26
C25
O1
C1
C3
C5
C2
C4
C18
C13
C22
C23
C24
C20
N1
C12
C8
C9
C10
C6
C7
C19
C11

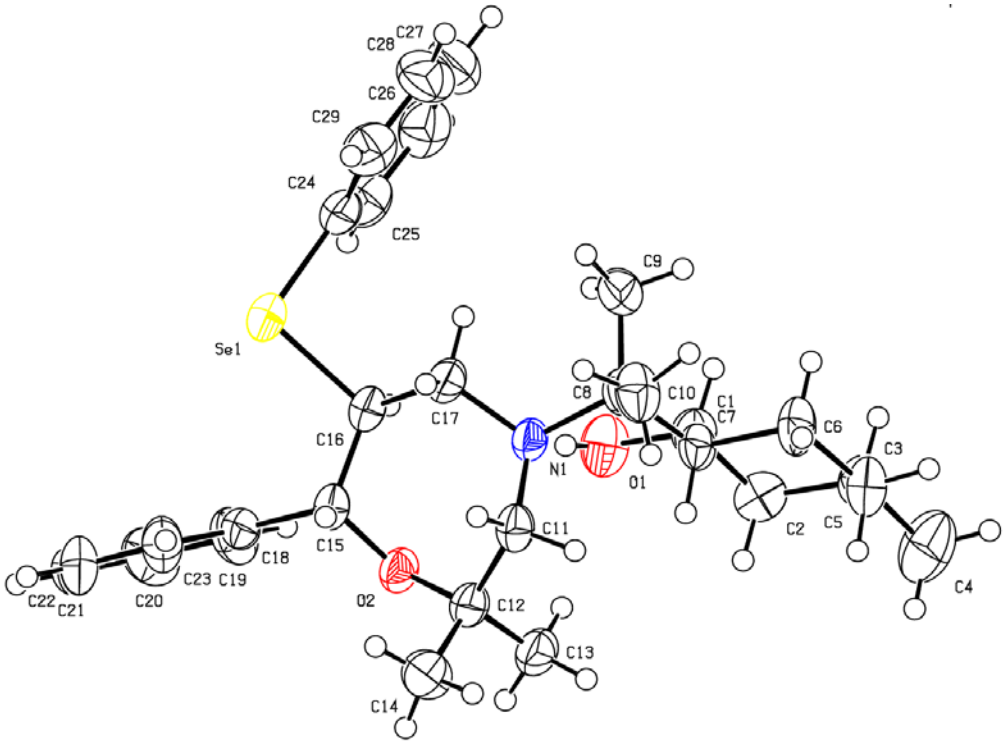

Ph
H
Cl
N
O
O

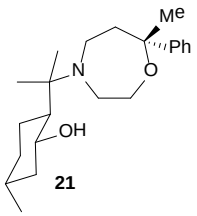
15f


C21
C20
C22
C19
C18
C17
C16
C15
C14
C13
C12
C10
C11
C9
C8
C7
C6
N1
C1
O1
C2
C3
C4
C5
CL1
O2

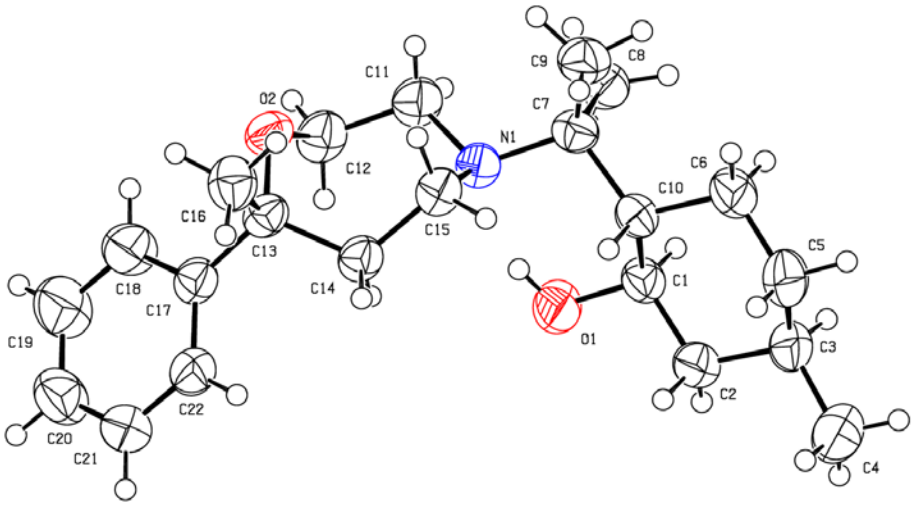

PhSe
Ph
N
O
OH

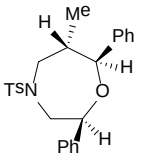
20


C27
C28
C26
C29
C24
C25
Se1
C9
C8
C10
C1
C7
C6
C3
C17
C16
N1
O1
C2
C5
C4
C11
C18
C15
C23
C19
C22
C21
C20
O2
C12
C13
C14

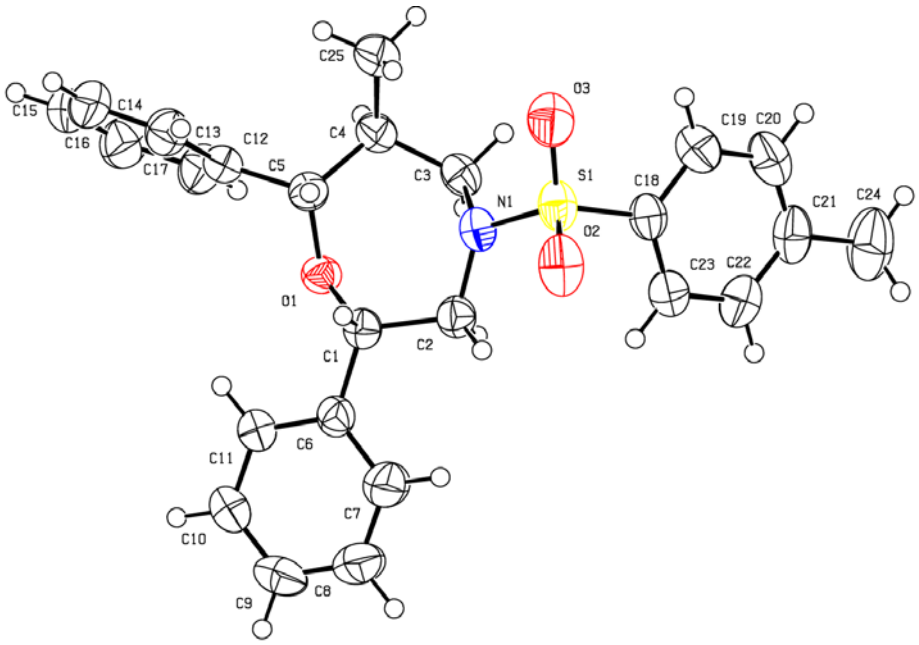

Me
Ph
N
O
OH

21


C11
C9
C8
O2
C7
N1
C12
C6
C10
C16
C13
C15
C5
C18
C14
C1
C17
O1
C3
C19
C2
C22
C20
C21
C4


H
Me
Ph
H
TsN
O
Ph
H

22i **^{1}H NMR**


C25
O3
C15
C14
C19
C20
C16
C13
C12
C4
S1
C18
C21
C24
C17
C5
C3
N1
O2
C23
C22
O1
C2
C1
C11
C6
C7
C10
C9
C8