Electronic Supplementary Information

Direct Reductive Coupling of Secondary Amides: Chemoselective Formation of Vicinal Diamines and Vicinal Amino Alcohols

Department of Chemistry and Fujian Provincial Key Laboratory for Chemical Biology, Collaborative Innovation Centre of Chemistry for Energy Materials, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen, Fujian 361005, P.R. China.

Fax: 86-592-2186400; pqhuang@xmu.edu.cn

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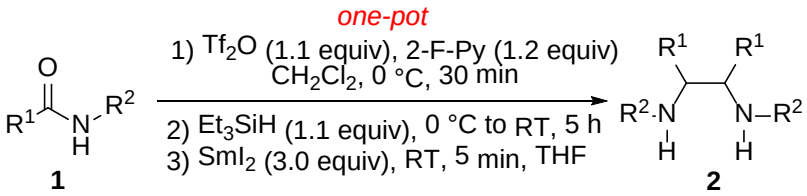
General: Melting points were uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a spectrometer at 400 and 100 MHz, respectively. Chemical shifts (δ) are reported in ppm and respectively referenced to internal standard Me_4Si and solvent signals (Me_4Si, 0 ppm for ^{1}H NMR and $CDCl_3$, 77.0 ppm for ^{13}C NMR). HRFABMS spectra were recorded on a 7.0T FT-MS. Silica gel (300-400 mesh) was used for flash column chromatography (FC), eluting (unless otherwise stated) with ethyl acetate/ hexane mixture. Trifluoromethanesulfonic anhydride (Tf_2O) was distilled over phosphorous pentoxide and was stored for no more than a week before re-distillation. Dry dichloromethane was distilled over calcium hydride under Argon. All reactions were carried out under an Argon atmosphere.

Table 1. Effects of NiI_2 on the reductive homocoupling reactions of *sec*-amides.



entry	R^1	R^2	% yield[a] (ratio)[b]	
			without NiI_2	with NiI_2[c]
1	Ph	*c*-hex	**2a**: 86 (54 : 46)	88 (53 : 47)
2	4-MeC_6H_4	*c*-hex	**2b**: 84 (60 : 40)	90 (55: 45)
3	4-BrC_6H_4	*c*-hex	**2f**: 71 (66 : 34)	79 (54 : 46)
4	Ph	*i*-Pr	**2n**: 91 (54 : 46)	93 (58 : 42)
5	4-MeC_6H_4	*i*-Pr	**2o**: 92 (66 : 34)	94 (59 : 41)

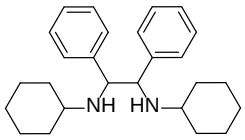
[a] Isolated yield; [b] Determined by ^{1}H NMR analysis; [c] Taken from Table 2 in the main text.

General procedure A: One-pot Preparation of Vicinal Diamines from Secondary Amides by Direct Reductive Homocoupling

Into a dry 10-mL round-bottom flask equipped with a stirring bar were added successively an amide (1.0 mmol, 1.0 equiv), 4 mL of anhydrous dichloromethane and 2-fluoropyridine (116.5 mg, 103 μL, 1.2 mmol, 1.2 equiv.). After being cooled to 0 °C, trifluoromethanesulfonic anhydride (Tf_2O) (310 mg, 185 μL, 1.1 mmol, 1.1 equiv) was added dropwise *via* a syringe at 0 °C and the reaction was stirred for 30 min. To the resulting mixture, triethylsilane (Et_3SiH) (128 mg, 176 μL, 1.1 mmol, 1.1 equiv) was added dropwise at 0 °C and the reaction was stirred for 30 min. The

mixture was allowed to warm-up to room temperature and stirred for 5 h. The resulting mixture was added to a solution of NiI_2-containing SmI_2 in THF, prepared by the addition of a THF solution of SmI_2 (3.0 mmol, 3.0 equiv, 30 mL) to a solution of anhydrous NiI_2 (3.1 mg, 0.01 mmol, 0.01 equiv) in THF (2 mL). The reaction mixture was added dropwise over 5 min at room temperature and stirred for another 5 min, then quenched with HCl (0.1 M) and stirred for 30 min, then extracted with diethyl ether (2×50 mL), The pH of the aqueous phase was adjusted to neutrality by the addition of a saturated aqueous $NaHCO_3$. The aqueous phase was extracted with diethyl ether and the combined organic extracts were washed successively with saturated aqueous sodium thiosulfate ($Na_2S_2O_3$) and brine, dried over Na_2SO_4 and filtered. The solvents were removed under reduced pressure and the residue was purified by flash chromatography on silica gel to give the corresponding vicinal diamines as *meso*/ *dl* mixture.

***N,N'*-Dicyclohexyl-1,2-diphenyl-1,2-ethanediamine (2a)**



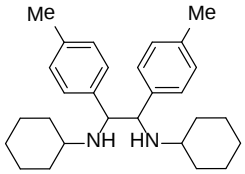
Following the general procedure A, the reduction homocoupling of amide **1a** (203 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the known diamine **2a**[1] (166 mg, 88%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2a** was obtained by recrystallization (EtOAc/Hexane), and pure *dl*-**2a** was isolated by repeated FC.

meso-**2a**: white solid, mp: 138-139 °C; (lit[1c]: mp: 135-139 °C). IR (film) ν_{max}: 3303, 3021, 2918, 2848, 1452, 1127, 1073, 889, 758, 728, 701 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.71-0.82 (m, 2H), 0.86-1.13 (m, 9H), 1.38-1.58 (m, 9H), 1.69-1.79 (m, 2H), 2.06-2.14 (m, 2H), 3.90 (s, 2H), 7.19-7.31 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.5, 24.9, 26.1, 32.3, 34.6, 53.0, 65.1, 127.1, 128.0, 128.3, 141.9 ppm.

dl-**2a**: white solid, mp: 123-124 °C; (lit[1d]: mp: 126-128 °C). IR (film) ν_{max}: 3303, 3025, 2925, 2851, 1450, 1115, 1027, 889, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.94-1.20 (m, 10H), 1.45-1.53 (m, 2H), 1.54-1.68 (m, 6H), 1.81-2.00 (m, 4H), 2.14-2.26 (m, 2H), 3.73 (s, 2H), 7.00-7.16 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6, 25.0, 26.2, 32.6, 34.9, 53.8, 66.3, 126.5, 127.7, 127.8, 142.6 ppm.

HRMS-ESI calcd for $C_{26}H_{36}N_2$: $(M+Na)^+$ 399.2776; found: 399.2778.

***N,N'*-Dicyclohexyl-1,2-di(4'-methylphenyl)-1,2-ethanediamine (2b)**



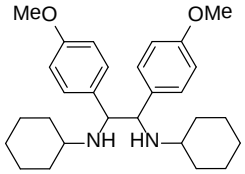
Following the general procedure A, the reduction homocoupling of amide **1b** (217 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2b** (182 mg, 90%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2b**: white solid, mp: 175-176 °C; IR (film) ν_{max}: 3411, 3017, 2919, 2848, 1510, 1455, 1124, 1105, 1017, 888, 841, 760, 722 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.67-0.79 (m, 2H), 0.84-1.12 (m, 9H), 1.38-1.58 (m, 9H), 1.71-1.80 (m, 2H), 2.03-2.13 (m, 2H), 2.33 (s, 6H), 3.84 (s, 2H), 7.06-7.15 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.1, 24.5, 25.0, 26.1, 32.3, 34.6, 52.8, 64.7, 128.2, 128.7, 136.5, 138.8 ppm.

dl-**2b**: white solid, mp: 105-106 °C; IR (film) ν_{max}: 3291, 3017, 2925, 2852, 1511, 1449, 1366, 1254, 1110, 1017, 889, 821, 719 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.91-1.17 (m, 10H), 1.44-1.52 (m, 2H), 1.54-1.65 (m, 6H), 1.79-1.92 (m, 4H), 2.14-2.21 (m, 2H), 2.25 (s, 6H), 3.70 (s, 2H), 6.90-6.97 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.0, 24.6, 25.0, 26.2, 32.6, 34.9, 53.7, 65.7, 127.7, 128.4, 135.8, 139.6 ppm.

HRMS-ESI calcd for $C_{28}H_{40}N_2$: $(M+H)^+$ 405.3270; found: 405.3271.

***N,N'*-Dicyclohexyl-1,2-di(4'-methoxyphenyl)-1,2-ethanediamine (2c)**



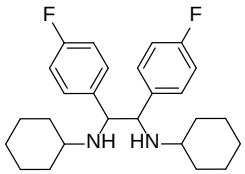
Following the general procedure A, the reduction homocoupling of amide **1c** (233 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/15/0.05 to 1/8/0.05), the desired diamine **2c** (175 mg, 80%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2c**: white solid, mp: 163-164 °C; IR (film) ν_{max}: 3299, 3008, 2924, 2848, 1608, 1583, 1507, 1455, 1301, 1241, 1168, 1121, 1103, 1036, 888, 844, 819, 804, 766, 728 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.65-0.78 (m, 2H), 0.84-0.94 (m, 2H), 0.96-1.11 (m, 6H), 1.28-1.56 (m, 10H), 1.71-1.81 (m, 2H), 2.02-2.11 (m, 2H), 3.81 (s, 2H), 3.82 (s, 6H), 6.81-6.87 (m, 4H), 7.15-7.20 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.6, 25.0, 26.1, 32.3, 34.7, 52.8, 55.2, 64.4, 113.4, 129.3, 133.9, 158.7 ppm.

dl-**2c**: IR (film) ν_{max}: 3299, 3021, 2925, 2851, 1615, 1585, 1510, 1450, 1301, 1245, 1177, 1105, 1037, 889, 829, 805, 736 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.93-1.19 (m, 10H), 1.44-1.67 (m, 8H), 1.81-1.96 (m, 4H), 2.13-2.24 (m, 2H), 3.66 (s, 2H), 3.74 (s, 6H), 6.64-6.71 (m, 4H), 6.89-6.96 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.7, 25.0, 26.2, 32.6, 34.9, 53.6, 55.1, 65.5, 113.1, 128.8, 134.6, 158.1 ppm.

HRMS-ESI calcd for $C_{28}H_{40}N_2O_2$: $(M+Na)^+$ 459.2988; found: 459.2994.

***N,N'*-Dicyclohexyl-1,2-di(4'-fluorophenyl)-1,2-ethanediamine (2d)**



Following the general procedure A, the reduction homocoupling of amide **1d** (221 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O =

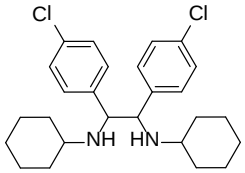
1/40/0.05 to 1/20/0.05), the desired diamine **2d** (177 mg, 86%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2d** was obtained by recrystallization (EtOAc/Hexane), a pure sample of *dl*-**2d** was isolated by repeated FC.

meso-**2d**: white solid, mp: 170-171 °C; IR (film) ν_{max}: 3303, 3029, 2917, 2849, 1600, 1504, 1461, 1415, 1127, 1151, 1122, 1090, 891, 824, 812, 731 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.71-0.83 (m, 2H), 0.88-0.97 (m, 2H), 0.99-1.12 (m, 6H), 1.39-1.60 (m, 10H), 1.69-1.79 (m, 2H), 2.03-2.13 (m, 2H), 3.85 (s, 2H), 6.93-7.01 (m, 4H), 7.12-7.19 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6, 25.0, 26.0, 32.5, 34.6, 53.0, 64.4, 114.9 (d, J_{F-C} = 21.0 Hz), 129.6 (d, J_{F-C} = 7.9 Hz), 137.4, 162.0 (d, J_{F-C} = 244.6 Hz) ppm.

dl-**2d**: white solid, mp: 125-127 °C; IR (film) ν_{max}: 3303, 3033, 2925, 2852, 1604, 1507, 1449, 1261, 1222, 1091, 1014, 840 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.94-1.28 (m, 10H), 1.48-1.90 (m, 12H), 2.12-2.22 (m, 2H), 3.65 (s, 2H), 6.79-6.86 (m, 4H), 6.92-6.99 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6, 25.0, 26.1, 32.6, 34.9, 53.8, 65.8, 114.6 (d, J_{F-C} = 21.1 Hz), 129.1 (d, J_{F-C} = 7.9 Hz), 138.2, 161.6 (d, J_{F-C} = 244.6 Hz) ppm.

HRMS-ESI calcd for $C_{26}H_{34}F_2N_2$: $(M+H)^+$ 413.2768; found: 413.2773.

1,2-Di(4'-chlorophenyl)-*N,N'*-dicyclohexyl-1,2-ethanediamine (2e)



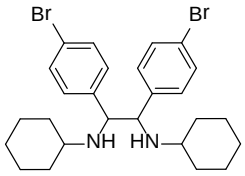
Following the general procedure A, the reduction homocoupling of amide **1e** (238 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2e** (196 mg, 88%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2e** was obtained after recrystallization (EtOAc/Hexane), a pure sample of *dl*-**2e** was isolated by repeated FC.

meso-**2e**: white solid, mp: 188-189 °C; IR (film) ν_{max}: 3398, 3021, 2920, 2848, 1485, 1456, 1406, 1123, 1088, 1009, 889, 811, 744, 721 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.72-0.84 (m, 2H), 0.87-1.14 (m, 8H), 1.19(br s, 2H), 1.42-1.60 (m, 8H), 1.70-1.80 (m, 2H), 2.02-2.14 (m, 2H), 3.86 (s, 2H), 7.07-7.12 (m, 4H), 7.22-7.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.6, 25.0, 26.0, 32.5, 34.6, 53.1, 64.2, 128.2, 129.6, 132.8, 140.1 ppm.

dl-**2e**: IR (film) ν_{max}: 3291, 3018, 2925, 2851, 1489, 1449, 1403, 1091, 1013, 821, 597 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.95-1.18 (m, 10H), 1.46-1.68 (m, 8H), 1.77-1.90 (m, 4H), 2.10-2.20 (m, 2H), 3.64 (s, 2H), 6.94 (d, J = 8.2 Hz, 4H), 7.11 (d, J = 8.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6, 25.0, 26.1, 32.6, 34.9, 53.8, 65.7, 128.0, 129.1, 132.3, 141.1 ppm.

HRMS-ESI calcd for $C_{26}H_{34}Cl_2N_2$: $(M+H)^+$ 445.2177; found: 445.2177.

1,2-Di(4'-bromophenyl)-*N,N'*-dicyclohexyl-1,2-ethanediamine (2f)



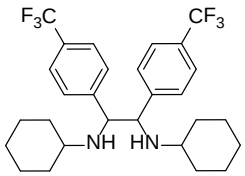
Following the general procedure A, the reduction homocoupling of amide **1f** (282 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2f** (211 mg, 79%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2f** was obtained after recrystallization (EtOAc/Hexane), a pure sample of *dl*-**2f** was isolated by repeated FC.

meso-**2f**: white solid, mp: 186-187 °C; IR (film) ν_{max}: 3287, 3017, 2919, 2845, 1586, 1463, 1400, 1255, 1119, 1095, 1068, 1006, 888, 812, 748, 718 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.72-0.84 (m, 2H), 0.86-1.24 (m, 9H), 1.42-1.69 (m, 9H), 1.72-1.80 (m, 2H), 2.02-2.13 (m, 2H), 3.85 (s, 2H), 7.01-7.07 (m, 4H), 7.39-7.42 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.6, 25.0, 26.0, 32.5, 34.6, 53.1, 64.2, 120.9, 130.0, 131.2, 140.6 ppm.

dl-**2f**: IR (film) ν_{max}: 3301, 3017, 2925, 2851, 1666, 1485, 1449, 1261, 1118, 1071, 1010, 820, 740, 722 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.93-1.19 (m, 10H), 1.45-1.70 (m, 8H), 1.80-1.88 (m, 2H), 1.94 (br s, 2H), 2.10-2.21 (m, 2H), 3.64 (s, 2H), 6.86-6.92 (m, 4H), 7.25-7.29 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.6, 24.9, 26.1, 32.6, 34.9, 53.8, 65.6, 120.4, 129.5, 131.0, 141.5 ppm.

HRMS-ESI calcd for $C_{26}H_{34}Br_2N_2$: $(M+H)^+$ 535.1147; found: 535.1138.

***N,N'*-Dicyclohexyl-1,2-di(4'-(trifluoromethyl)phenyl)-1,2-ethanediamine (2g)**



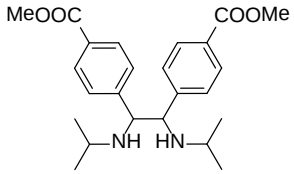
Following the general procedure A, the reduction homocoupling of amide **1g** (271 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2g** (207 mg, 81%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2g** was obtained after recrystallization (EtOAc/Hexane), a pure sample of *dl*-**2g** was isolated by repeated FC

meso-**2g**: white solid, mp: 179-180 °C; IR (film) ν_{max}: 3411, 3042, 2929, 2850, 1619, 1455, 1417, 1325, 1161, 1122, 1101, 1068, 1012, 861, 821 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.77-0.88 (m, 2H), 0.90-1.15 (m, 8H), 1.41-1.65 (m, 10H), 1.72-1.81 (m, 2H), 2.05-2.14 (m, 2H), 4.00 (s, 2H), 7.24 (d, J = 8.0 Hz, 4H), 7.52 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.5, 24.9, 26.0, 32.5, 34.6, 53.2, 64.4, 124.2 (d, J_{F-C} = 272.3 Hz), 124.9 (q, J_{F-C} = 3.4 Hz), 128.5, 129.5 (q, J_{F-C} = 32.5 Hz), 145.6 ppm.

dl-**2g**: IR (film) ν_{max}: 3299, 3037, 2928, 2854, 1618, 1451, 1418, 1325, 1164, 1125, 1068, 1017, 849 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.96-1.20 (m, 10H), 1.46-1.70 (m, 8H), 1.80-1.88 (m, 2H), 1.89 (br s, 2H), 2.11-2.21 (m, 2H), 3.76 (s, 2H), 7.13 (d, J = 8.0 Hz, 4H), 7.39 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.5, 24.9, 26.1, 32.6, 34.9, 53.9, 66.0, 124.2 (d, J_{F-C} = 272.0 Hz), 124.8 (q, J_{F-C} = 3.8 Hz), 128.0, 129.1 (q, J_{F-C} = 32.3 Hz), 146.6 ppm.

HRMS-ESI calcd for $C_{28}H_{34}F_6N_2$: $(M+H)^+$ 513.2704; found: 513.2694.

Dimethyl 4,4'-(1,2-bis(isopropylamino)ethane-1,2-diyl)dibenzoate (2h)



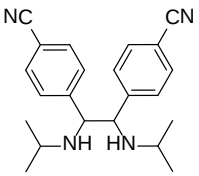
Following the general procedure A, the reduction homocoupling of amide **1h** (221 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.05 to 1/4/0.05), the desired diamine **2h** (85 mg, 41%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2h** was obtained after recrystallization (EtOAc/Hexane), a pure sample of *dl*-**2h** was isolated by repeated FC.

meso-**2h**: white solid, mp: 188-189 °C; IR (film) ν_{max}: 3307, 3029, 2961, 2918, 2848, 1719, 1607, 1469, 1440, 1414, 1276, 1196, 1168, 1116, 1104, 1015, 878, 854, 769, 732, 710 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.87 (d, J = 6.2 Hz, 6H), 0.90 (d, J =6.3 Hz, 6H), 1.44 (br s, 2H), 2.48 (septet, J = 6.2 Hz, 2H), 3.91 (s, 6H), 3.99 (s, 2H), 7.13-7.18 (m, 4H), 7.91-7.95 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.9, 24.1, 45.6, 52.0, 65.3, 128.2, 129.2, 129.3, 146.6, 167.0 ppm.

dl-**2h**: white solid, mp: 113-114 °C; IR (film) ν_{max}: 3305, 2960, 2921, 2847, 1724, 1610, 1435, 1382, 1279, 1180, 1113, 1018, 861, 771, 711 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.92 (d, J = 6.2 Hz, 6H), 0.99 (d, J = 6.2 Hz, 6H), 2.46-2.57 (m, 2H), 1.92 (br s, 2H), 3.74 (s, 2H), 3.87 (s, 6H), 7.09 (d, J = 8.1 Hz, 4H), 7.80 (d, J = 8.1 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 24.4, 46.3, 51.9, 66.5, 127.8, 128.8, 129.2, 147.5, 166.9 ppm.

HRMS-ESI calcd for $C_{24}H_{32}N_2O_4$: $(M+H)^+$ 413.2440; found: 413.2439.

4,4'-(1,2-Bis(isopropylamino)ethane-1,2-diyl)dibenzonitrile (2i)



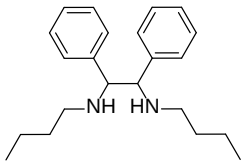
Following the general procedure A, the reduction homocoupling of amide **1i** (188 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.05 to 1/4/0.05), the desired diamine **2i** (101 mg, 58%) as a *meso*/ *dl* mixture, which could be separated by repeated FC. A pure sample of *meso*-**2i** was obtained after recrystallization (EtOAc/Hexane).

meso-**2i**: white solid, mp: 151-152 °C; IR (film) ν_{max}: 3299, 2967, 2917, 2842, 2224, 1606, 1478, 1465, 1440, 1407, 1357, 1270, 1150, 1125, 1013, 880, 851, 830, 768, 739 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.90 (d, J = 6.2 Hz, 6H), 0.93 (d, J =6.2 Hz, 6H), 1.31 (br s, 2H), 2.48 (septet, J = 6.2 Hz, 2H), 3.97 (s, 2H), 7.14-7.19 (m, 4H), 7.50-7.60 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 24.0, 45.8, 64.9, 111.3, 118.7, 128.9, 131.8, 146.6 ppm.

dl-**2i**: (data read from the spectra of a diastereomeric mixture) ^1H NMR (400 MHz, $CDCl_3$): δ 0.93 (d, J =6.2 Hz, 6H), 0.97 (d, J =6.2 Hz, 6H), 1.80 (br s, 2H), 2.43-2.54 (m, 2H), 3.71 (s, 2H),

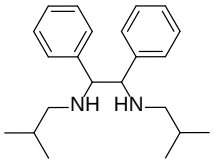
7.10-7.17 (m, 4H), 7.42-7.47 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 24.0, 45.8, 64.9, 111.3, 118.7, 128.9, 131.8, 146.6 ppm.
HRMS-ESI calcd for $C_{22}H_{26}N_4$: $(M+Na)^+$ 369.2055; found: 369.2057.

***N,N'*-Dibutyl-1,2-diphenyl-1,2-ethanediamine (2j)**



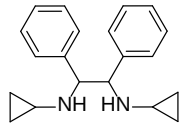
Following the general procedure A, the reduction homocoupling of amide **1j** (177 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the known diamine **2j**[2](116 mg, 71%) as a *meso*/ *dl* mixture, which were separated by repeated FC.
meso-**2j**: white solid, mp: 32-33 °C; IR (film) ν_{max}: 3320, 3027, 2956, 2926, 2857, 1454, 1378, 1275, 1200, 1139, 1028, 914, 759, 701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.75 (t, J = 7.3 Hz, 6H), 1.04-1.14 (m, 4H), 1.20-1.28 (m, 4H), 1.42 (br s, 2H), 2.17-2.32 (m, 4H), 3.72 (s, 2H), 7.25-7.35 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): 13.8, 20.1, 31.9, 47.1, 68.6, 127.4, 128.2, 128.3, 141.4 ppm.
dl-**2j**: colorless oil, 0.85 (t, J = 7.3 Hz, 6H), 1.24-1.31 (m, 4H), 1.37-1.46 (m, 4H), 1.98 (br s, 2H), 2.32-2.47 (m, 4H), 3.61 (s, 2H), 7.00-7.05 (m, 4H), 7.07-7.17 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 20.4, 32.3, 47.4, 69.4, 126.7, 127.8 (2C), 141.6 ppm.
HRMS-ESI calcd for $C_{22}H_{32}N_2$: $(M+H)^+$ 325.2644; found: 325.2642.

***N,N'*-Diisobutyl-1,2-diphenyl-1,2-ethanediamine (2k)**



Following the general procedure A, the reduction homocoupling of amide **1k** (177 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2k** (135 mg, 83%) as a *meso*/ *dl* mixture, which were separated by repeated FC.
meso-**2k**: white solid, mp: 59-60 °C; IR (film) ν_{max}: 3344, 3062, 3027, 2954, 2869, 2804, 1602, 1468, 1453, 1386, 1367, 1247, 1200, 1121, 1071, 1029, 918, 808, 758, 701, 602 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.65 (d, J = 6.4 Hz, 6H), 0.67 (d, J =6.4 Hz, 6H), 1.34-1.62 (m, 4H), 2.02-2.14 (m, 4H), 3.68 (s, 2H), 7.23-7.36 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.2, 20.4, 27.8, 55.4, 68.6, 127.4, 128.2, 128.3, 141.5 ppm.
dl-**2k**: IR (film) ν_{max}: 3320, 3063, 3026, 2954, 2870, 1492, 1453, 1386, 1362, 1263, 1242, 1109, 1027, 764, 699, 599 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.84 (d, J = 6.6 Hz, 6H), 0.87 (d, J = 6.6 Hz, 6H), 1.62-1.74 (m, 2H), 2.05 (br s, 2H), 2.17 (dd, J = 6.6, 11.4 Hz, 2H), 2.25 (dd, J = 6.6, 11.4 Hz, 2H), 3.57 (s, 2H), 6.99-7.05 (m, 4H), 7.06-7.16 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.6, 20.7, 28.5, 55.7, 69.6, 126.6, 127.8 (2C), 141.8 ppm.
HRMS-ESI calcd for $C_{22}H_{32}N_2$: $(M+Na)^+$ 347.2463; found: 347.2466.

***N,N'*-Dicyclopropyl-1,2-diphenyl-1,2-ethanediamine (2l)**



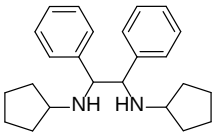
Following the general procedure A, the reduction homocoupling of amide **1l** (161 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2l** (96 mg, 66%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2l**: white solid, mp: 82-83 °C; IR (film) ν_{max}: 3324, 3085, 3061, 3026, 3005, 2921, 2850, 1493, 1452, 1369, 1343, 1217, 1103, 1067, 1015, 806, 759, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.13-0.28 (m, 8H), 1.81-1.88 (m, 2H), 1.93 (br s, 2H), 3.92 (s, 2H), 7.08-7.13 (m, 4H), 7.22-7.29 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 5.9, 6.9, 28.8, 67.8, 127.1, 127.9, 128.3, 141.1 ppm.

dl-**2l**: white solid, mp: 73-75 °C; IR (film) ν_{max}: 3261, 3062, 3021, 3005, 2918, 2847, 1444, 1365, 1260, 1096, 1005, 868, 799, 753, 697 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.21-0.33 (m, 8H), 1.84-2.08 (m, 4H), 3.69 (s, 2H), 7.01-7.16 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 5.9, 7.0, 29.0, 68.7, 126.6, 127.7, 128.1, 141.8 ppm.

HRMS-ESI calcd for $C_{20}H_{24}N_2$: $(M+Na)^+$ 315.1837; found: 315.1841.

***N,N'*-Dicyclopentyl-1,2-diphenyl-1,2-ethanediamine (2m)**



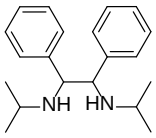
Following the general procedure A, the reduction homocoupling of amide **1m** (189 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2m** (153 mg, 88%) as a *meso*/ *dl* mixture, which were separated by repeated FC. A pure sample of *meso*-**2m** was obtained by recrystallization (EtOAc/Hexane); pure *dl*-**2m** was isolated by repeated FC.

meso-**2m**: white solid, mp: 98-99 °C; IR (film) ν_{max}: 3324, 3062, 3027, 2950, 2855, 1492, 1452, 1348, 1176, 1099, 1069, 1027, 892, 757, 734, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.97-1.08 (m, 4H), 1.25-1.49 (m, 10H), 1.54-1.66 (m, 4H), 2.67 (quint, J = 7.0 Hz, 2H), 3.80 (s, 2H), 7.26-7.35 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.4, 23.4, 32.0, 33.6, 56.9, 67.0, 127.4, 128.2, 128.4, 141.6 ppm.

dl-**2m**: white solid, mp: 67-68 °C; IR (film) ν_{max}: 3299, 3061, 3025, 2954, 2866, 1492, 1453, 1356, 1182, 1118, 1072, 1027, 872, 758, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.15-1.26 (m, 2H), 1.28-1.48 (m, 6H), 1.56-1.74 (m, 8H), 1.96 (br s, 2H), 2.77-2.85 (m, 2H), 3.64 (s, 2H), 7.02-7.16 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.6, 23.7, 32.3, 34.1, 57.2, 67.8, 126.6, 127.7, 128.0, 142.1 ppm.

HRMS-ESI calcd for $C_{24}H_{32}N_2$: $(M+Na)^+$ 371.2463; found: 371.2463.

***N,N'*-Diisopropyl-1,2-diphenyl-1,2-ethanediamine (2n)**



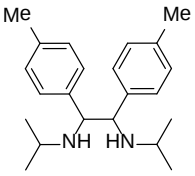
Following the general procedure A, the reduction homocoupling of amide **1n** (163 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/10/0.05 to 1/6/0.05), the desired diamine **2n** (138 mg, 93%) as a *meso*/ *dl* mixture, which were separated by repeated FC. Pure *meso*-**2n** was obtained by recrystallization (EtOAc/Hexane).

meso-**2n**: white solid, mp: 113-114 °C; IR (film) ν_{max}: 3303, 3026, 2958, 2923, 2850, 1469, 1453, 1361, 1272, 1166, 1123, 1066, 1026, 923, 866, 798, 761, 730 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.84 (d, J = 6.2 Hz, 6H), 0.87 (d, J = 6.2 Hz, 6H), 1.35 (br s, 2H), 2.47 (septet, J = 6.2 Hz, 2H), 3.90 (s, 2H), 7.12-7.18 (m, 4H), 7.20-7.30 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.8, 24.2, 45.4, 65.5, 127.1, 128.0, 128.3, 141.4 ppm.

dl-**2n:**(data read from the spectra of a diastereomeric mixture) ^1H NMR (400 MHz, $CDCl_3$): δ 0.94 (d, J = 6.2 Hz, 6H), 0.98 (d, J = 6.2 Hz, 6H), 1.77 (br s, 2H) 2.48-2.58 (m, 2H), 3.71 (s, 2H), 6.98-7.04 (m, 4H), 7.06-7.16 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 24.5, 46.0, 66.7, 126.6, 127.7, 127.9, 142.2 ppm.

HRMS-ESI calcd for $C_{20}H_{28}N_2$: $(M+H)^+$ 297.2331; found: 297.2326.

***N,N'*-Diisopropyl-1,2-di-p-tolyl-1,2-ethanediamine (2o)**



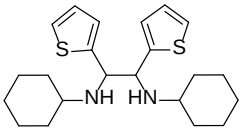
Following the general procedure A, the reduction homocoupling of amide **1o** (177 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/10/0.05 to 1/6/0.05), the desired diamine **2o** (153 mg, 94%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2o**: white solid, mp: 136-137 °C; IR (film) ν_{max}: 3386, 3017, 2960, 2917, 2849, 1469, 1440, 1382, 1360, 1165, 1121, 1106, 1080, 868, 822, 757, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.82 (d, J = 6.2 Hz, 6H), 0.86 (d, J = 6.2 Hz, 6H), 1.20 (br s, 2H), 2.33 (s, 6H), 2.39-2.50 (m, 2H), 3.84 (s, 2H), 7.05-7.11 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.1, 21.7, 24.2, 45.2, 65.2, 128.2, 128.7, 136.5, 138.3 ppm.

dl-**2o**: IR (film) ν_{max}: 3307, 3013, 2960, 2924, 2865, 1512, 1467, 1379, 1364, 1173, 1124, 1077, 822, 723, 609 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.92 (d, J = 6.2 Hz, 6H), 0.96 (d, J = 6.2 Hz, 6H), 1.91 (br s, 2H), 2.25 (s, 6H), 2.52 (septet, J = 6.2 Hz, 2H), 3.70 (s, 2H), 6.87-6.97 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.0, 21.9, 24.4, 45.8, 66.0, 127.8, 128.4, 135.9, 138.9 ppm.

HRMS-ESI calcd for $C_{22}H_{32}N_2$: $(M+Na)^+$ 347.2463; found: 347.2467.

***N,N'*-Dicyclohexyl-1,2-di(thiophen-2-yl)-1,2-ethanediamine (2r)**



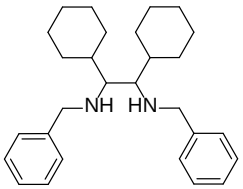
Following the general procedure A, the reduction homocoupling of amide **1r** (209 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/40/0.05 to 1/20/0.05), the desired diamine **2r** (126 mg, 65%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2r**: white solid, mp: 136-137 °C; IR (film) ν_{max}: 3301, 3108, 3071, 2920, 2848, 1466, 1446, 1363, 1294, 1134, 1120, 1040, 963, 889, 851, 815, 761, 738, 706 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.67-0.78 (m, 2H), 0.92-1.18 (m, 8H), 1.40-1.58 (m, 8H), 1.60-1.68 (m, 2H), 1.75-1.84 (m, 2H), 2.21-2.29 (m, 2H), 4.14 (s, 2H), 6.95 (dd, *J* = 3.4, 5.0 Hz, 2H), 7.02 (dd, *J* = 1.1, 3.4 Hz, 2H), 6.95 (dd, *J* = 1.1, 5.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.4, 25.0, 26.0, 32.0, 34.4, 52.9, 61.8, 125.1, 126.1 (2C), 147.1 ppm.

dl-**2r**: IR (film) ν_{max}: 3295, 3065, 2925, 2851, 1449, 1369, 1260, 1143, 1111, 1039, 890, 832, 694 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.96-1.21 (m, 10H), 1.47-1.55 (m, 2H), 1.61-1.71 (m, 6H), 1.85-1.94 (m, 4H), 2.31-2.40 (m, 2H), 4.04 (s, 2H), 5.58-6.62 (m, 2H), 6.81 (dd, *J* = 3.4, 5.0 Hz, 2H), 7.12 (dd, *J* = 0.7, 5.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 24.6, 25.0, 26.1, 32.5, 34.7, 53.9, 62.4, 123.7, 124.7, 126.2, 147.8 ppm.

HRMS-ESI calcd for $C_{22}H_{32}N_2S_2$: $(M+Na)^+$ 411.1905; found: 411.1902.

***N,N'*-Dibenzyl-1,2-diphenyl-1,2- ethanediamine (2s)**



Following the general procedure A, the reduction homocoupling of amide **1s** (217 mg, 1.0 mmol) gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/20/0.05 to 1/10/0.05), the desired diamine **2s**[1a, 3](109 mg, 54%) as a *meso*/ *dl* mixture, which were separated by repeated FC.

meso-**2s**: white solid, mp: 105-106 °C; IR (film) ν_{max}: 3352, 3062, 3021, 2219, 2849, 1579, 1537, 1493, 1451, 1384, 1095, 1028, 696 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.08-1.26 (m, 10H), 1.45-1.68 (m, 8H), 1.69-1.78 (m, 4H), 1.86-1.95 (m, 2H), 2.42-2.46 (m, 2H), 3.70 (d, *J* = 12.9 Hz, 2H), 3.74 (d, *J* = 12.9 Hz, 2H), 7.19-7.25 (m, 2H), 7.27-7.33 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.6 (2C), 26.9, 29.2, 32.0, 40.2, 54.9, 63.7, 126.7, 128.2 (2C), 141.5 ppm.

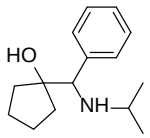
dl-**2s:** ^1H NMR (400 MHz, $CDCl_3$): δ 0.98-1.28 (m, 11H), 1.42-1.52 (m, 2H), 1.63-1.81 (m, 11H), 2.39 (d, *J* = 3.1 Hz, 2H), 3.73 (d, *J* = 12.6 Hz, 2H), 3.87 (d, *J* = 12.6 Hz, 2H), 7.20-7.35 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 26.7, 26.8 (2C), 29.6, 30.0, 41.8, 54.1, 62.4, 126.9, 128.3, 128.4, 141.1 ppm.

MS-ESI calcd for $C_{28}H_{40}N_2$: $(M+H)^+$ 405; found: 405.

General procedure B: One-pot Preparation of Vicinal Amino Alcohols from Secondary Amides by Direct Reductive Cross-coupling with Ketones

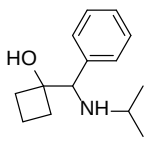
Into a dry 10-mL round-bottom flask equipped with a stirring bar were added successively an amide (1.0 mmol, 1.0 equiv), 4 mL of anhydrous dichloromethane and 2-fluoropyridine (116.5 mg, 103 μL, 1.2 mmol, 1.2 equiv). After being cooled to 0 °C, trifluoromethanesulfonic anhydride (Tf_2O) (310 mg, 185 μL, 1.1 mmol, 1.1 equiv) was added dropwise *via* a syringe at 0 °C and the reaction was stirred for 30 min. To the resulting mixture, triethylsilane (Et_3SiH) (128 mg, 176 μL, 1.1 mmol, 1.1 equiv) was added dropwise at 0 °C and the reaction was stirred for 30 min. The mixture was allowed to warm-up to room temperature and stirred for 5 h. The solution was then cooled to 0 °C and triethylamine (Et_3N) (152 mg, 209 μL 1.5 mmol, 1.5 equiv) was added dropwise and the resulting mixture was stirred for 30 min. Ketone (3 mmol, 3 equiv) was added to a solution of NiI_2-containing SmI_2 in THF, prepared by the addition of a THF solution of SmI_2 (2.5 mmol, 2.5 equiv, 25 mL) to a solution of anhydrous NiI_2 (3.1 mg, 0.01 mmol, 0.01 equiv) in THF (2 mL) at 0 °C. The above reaction mixture was added dropwise to the solution of ketone and SmI_2 in THF in about 13 min at 0 °C and stirred another 2 min, then the reaction was quenched with HCl (0.1 M) and stirred for another 30 min at room temperature, then extracted with ether (2×50 mL), The pH of the aqueous phase was adjusted to neutrality by the addition of saturated aqueous $NaHCO_3$. The aqueous phase was extracted with ether and the combined organic extracts were washed with saturated aqueous sodium thiosulfate ($Na_2S_2O_3$) and brine, dried over Na_2SO_4. The solvents were removed under reduced pressure and the residue was purified by flash chromatography on silica gel to give the corresponding vicinal amino alcohols.

1-[(Isopropylamino)(phenyl)methyl]cyclopentanol (3a)



Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3a** (177 mg, 76%) as a white solid, mp: 53-54 °C; IR (film) ν_{max}: 3446, 3025, 2961, 2870, 1469, 1452, 1380, 1339, 1174, 1125, 1069, 1029, 1002, 870, 704 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.24-1. 31 (m, 1H), 1.41 -1.51 (m, 3H), 1.56-1.63 (m, 1H), 1.68-1.82 (m, 3H), 2.61 (septet, J = 6.2 Hz, 1H), 3.34 (br s, 1H), 3.73 (s, 1H), 7.22-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 22.0, 23.3, 23.4, 24.3, 35.7, 37.7, 45.6, 67.3, 83.5, 127.1, 128.0, 128.3, 140.8 ppm. HRMS-ESI calcd for $C_{15}H_{23}NO$: $(M+H)^+$ 234.1858; found: 234.1850.

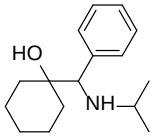
1-[(Isopropylamino)(phenyl)methyl]cyclobutanol (3b)



Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol)

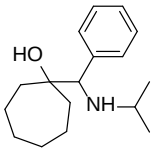
with *c*-butanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3b** (158 mg, 72%) as a pale yellow oil; IR (film) ν_{max}: 3420, 3026, 2963, 2868, 1467, 1452, 1380, 1339, 1242, 1170, 1126, 1096, 873, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.00 (d, J = 6.3 Hz, 3H), 1.04 (d, J = 6.3 Hz, 3H), 1.06-1.14 (m, 1H), 1.61-1.72 (m, 1H), 1.80-1.89 (m, 1H), 1.97-2.05 (m, 1H), 2.12-2.18 (m, 2H), 2.64 (septet, J = 6.3 Hz, 1H), 3.76 (s, 1H), 7.27-7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 12.2, 22.1, 24.1, 32.2, 34.1, 45.4, 65.9, 76.2, 127.3, 128.1(2C), 139.6 ppm. HRMS-ESI calcd for $C_{14}H_{21}NO$: $(M+H)^+$ 220.1701; found: 220.1694.

1-[(Isopropylamino)(phenyl)methyl]cyclohexanol (3c)



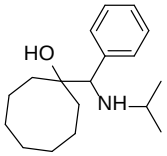
Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with *c*-hexanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3c** (186 mg, 75%) as a white solid, mp: 44-45 °C; IR (film) ν_{max}: 3428, 3025, 2925, 2850, 1468, 1450, 1383, 1249, 1124, 1085, 1030, 973, 872, 720 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 0.99 (d, J = 6.2 Hz, 3H), 1.06-1.16 (m, 1H), 1.19-1.29 (m, 2H), 1.31-1.38 (m, 1H), 1.42-1.52 (m, 2H), 1.53-1.70 (m, 4H), 2.58 (septet, J = 6.2 Hz, 1H), 3.50 (s, 1H), 7.19-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 21.5, 22.0 (2C), 24.3, 25.8, 32.1, 35.6, 46.1, 69.3, 72.1, 127.1, 128.0, 128.4, 140.3 ppm. HRMS-ESI calcd for $C_{16}H_{25}NO$: $(M+H)^+$ 248.2014; found: 248.2010.

1-[(Isopropylamino)(phenyl)methyl]cycloheptanol (3d)



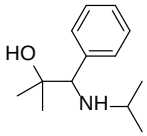
Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with *c*-heptanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.0 to 1/5/0.05), the desired β-amino alcohol **3d** (183 mg, 71%) as a pale yellow oil; IR (film) ν_{max}: 3439, 3026, 2923, 2856, 1462, 1380, 1341, 1174, 1125, 1072, 1044, 872, 704 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.97 (d, J = 6.2 Hz, 3H), 0.99 (d, J = 6.2 Hz, 3H), 1.07-1.17 (m, 1H), 1.21-1.32 (m, 3H), 1.34-1.44 (m, 2H), 1.45-1.52 (m, 2H), 1.52-1.64 (m, 3H), 1.67-1.79 (m, 2H), 2.58 (septet, J = 6.2 Hz, 1H), 3.50 (s, 1H), 7.23-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 22.1 (2C), 22.5, 24.5, 29.5 (2C), 36.0, 39.4, 46.0, 69.6, 75.9, 127.0, 128.0, 128.4, 140.8 ppm. HRMS-ESI calcd for $C_{17}H_{27}NO$: $(M+H)^+$ 262.2171; found: 262.2165.

1-[(Isopropylamino)(phenyl)methyl]cyclooctanol (3e)



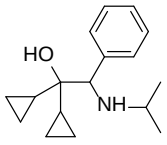
Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with *c*-octanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.0 to 1/6/0.05), the desired β-amino alcohol **3e** (165 mg, 60%) as a pale yellow oil; IR (film) ν_{max}: 3444, 3025, 2925, 2851, 1450, 1383, 1289, 1200, 1099, 1002, 889, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.97 (d, J = 6.3 Hz, 3H), 0.98 (d, J = 6.3 Hz, 3H), 1.32-1.46 (m, 8H), 1.48 -1.57 (m, 2H), 1.58-1.69 (m, 2H), 1.75-1.88 (m, 2H), 2.56 (septet, J = 6.3 Hz, 1H), 3.51 (s, 1H), 7.22-7.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 21.6, 22.2 (2C), 24.3, 24.7, 27.8, 28.6, 33.0, 34.5, 46.0, 68.1, 75.7, 127.0, 128.4 (2C), 141.2 ppm. HRMS-ESI calcd for $C_{18}H_{29}NO$: $(M+H)^+$ 276.2327; found: 276.2325.

1-(Isopropylamino)-2-methyl-1-phenylpropan-2-ol (3f)



Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with acetone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/4/0.0 to 1/2/0.05), the desired β-amino alcohol **3f** (155 mg, 75%) as a white solid, mp: 81-82 °C; IR (film) ν_{max}: 3360, 3303, 2962, 2918, 2866, 1622, 1491, 1452, 1375, 1306, 1152, 1068, 1029, 975, 887, 795, 776, 738, 703 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.02 (d, J = 6.3 Hz, 3H), 1.03 (d, J = 6.3 Hz, 3H), 1.04 (s, 3H), 1.15 (s, 3H), 2.61 (septet, J = 6.3 Hz, 1H), 3.00 (br s, 2H), 3.56 (s, 1H), 7.21-7.35 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 22.0, 24.0, 24.4, 27.3, 46.1, 69.3, 71.5, 127.2, 128.0 (2C), 140.4 ppm. HRMS-ESI calcd for $C_{13}H_{21}NO$: $(M+H)^+$ 208.1701; found: 208.1698.

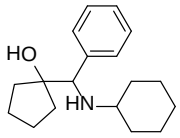
1,1-Dicyclopropyl-2-(isopropylamino)-2-phenylethanol (3g)



Following the general procedure B, the reductive cross-coupling of amide **1n** (163 mg, 1.0 mmol) with dicyclopropylketone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/10/0.0 to 1/8/0.05), the desired β-amino alcohol **3g** (197 mg, 76%) as a white solid, mp: 52-53 °C; IR (film) ν_{max}: 3390, 3086, 3007, 2966, 2926, 2868, 1495, 1467, 1402, 1323, 1192, 1175, 1126, 1090, 1021, 1001, 903, 874, 766, 706 cm^{-1}; H NMR (400 MHz, $CDCl_3$): δ 0.10-0.19 (m, 1H), 0.22-0.36 (m, 4H), 0.40-0.61 (m, 4H), 0.63-0.72 (m ,1H), 0.95 (d, J = 6.2 Hz, 3H), 0.99 (d, J = 6.2 Hz, 3H), 1.90 (br s, 1H), 2.61 (septet, J = 6.2 Hz, 1H), 3.75 (s, 1H), 3.92 (br s, 1H), , 7.23-7.40 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): -1.7, -1.1, -0.9, 1.5, 14.3, 18.4,

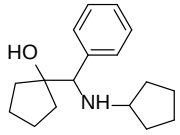
21.8, 24.6, 45.4, 69.3, 69.8, 127.0, 127.8 , 128.3, 140.1 ppm. HRMS-ESI calcd for $C_{17}H_{25}NO$: $(M+H)^+$ 260.2014; found: 260.2017.

1-[(Cyclohexylamino)(phenyl)methyl]cyclopentanol (3h)



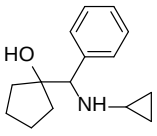
Following the general procedure B, the reductive cross-coupling of amide **1a** (203 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.0 to 1/5/0.05), the desired β-amino alcohol **3h** (186 mg, 68%) as a white solid, mp: 70-71 °C; IR (film) ν_{max}: 3444, 3025, 2925, 2851, 1450, 1383, 1289, 1200, 1099, 1002, 889, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.96-1.20 (m, 5H), 1.24-1.31 (m, 1H), 1.38-1.83 (m, 12H), 1.86-1.96 (m, 1H), 2.21-2.31 (m, 1H), 3.46 (br s, 1H), 3.78 (s, 1H), 7.22-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 23.3, 23.4, 24.4, 24.9, 26.1, 32.8, 34.8, 35.5, 37.6, 53.4, 66.6, 83.5, 127.0, 127.9, 128.3, 140.9 ppm. HRMS-ESI calcd for $C_{18}H_{27}NO$: $(M+H)^+$ 274.2171; found: 274.2171.

1-[(Cyclopentylamino)(phenyl)methyl]cyclopentanol (3i)



Following the general procedure B, the reductive cross-coupling of amide **1m** (189 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.0 to 1/5/0.05), the desired β-amino alcohol **3i** (179 mg, 69%) as a white solid, mp: 85-86 °C; IR (film) ν_{max}: 3437, 3025, 2954, 2869, 1493, 1451, 1384, 1195, 1122, 1075, 995, 875, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.82 (m, 17H), 2.83-2.92 (m, 1H), 3.37 (br s, 1H), 3.67 (s, 1H), 7.23-7.36 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 23.3, 23.4, 23.7 (2 C), 32.4, 34.1, 35.5, 37.6, 56.9, 68.5, 83.5, 127.1, 128.0, 128.5, 140.6 ppm. HRMS-ESI calcd for $C_{17}H_{25}NO$: $(M+H)^+$ 260.2014; found: 260.2010.

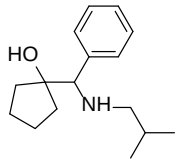
1-[(Cyclopropylamino)(phenyl)methyl]cyclopentanol (3j)



Following the general procedure B, the reductive cross-coupling of amide **1l** (161 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/8/0.0 to 1/5/0.05), the desired β-amino alcohol **3j** (125 mg, 54%) as a pale yellow oil; IR (film) ν_{max}: 3440, 3085, 2957, 2870, 1494, 1452, 1384, 1368, 1215, 1101, 1014, 704 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.24-0.46 (m, 4H), 1.23-1.32 (m, 1H), 1.37-1.53

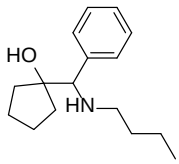
(m, 3H), 1.54-1.63 (m, 1H), 1.65-1.81 (m, 3H), 1.91-1.97 (m, 1H), 2.51(br s, 2H), 3.73 (s, 1H), 7.24-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 6.6, 7.0, 23.3, 23.4, 28.8, 35.6, 37.4, 70.3, 83.4, 127.1, 128.0, 128.4, 140.8 ppm. HRMS-ESI calcd for $C_{15}H_{21}NO$: $(M+Na)^+$ 254.1521; found: 254.1518.

1-[(Isobutylamino)(phenyl)methyl]cyclopentanol (3k)



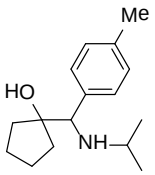
Following the general procedure B, the reductive cross-coupling of amide **1k** (177 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3k** (151 mg, 61%) as a pale yellow oil; IR (film) ν_{max}: 3444, 3025, 2953, 2870, 1495, 1452, 1385, 1196, 1099, 895, 852, 704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (d, J = 5.8 Hz, 3H), 0.88 (d, J = 5.8 Hz, 3H), 1.24-1.33 (m, 1H), 1.43-1.53 (m, 3H), 1.56-1.84 (m, 5H), 2.20-2.33 (m, 2H), 3.61 (s, 1H), 7.22-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 20.4, 20.6, 23.2, 23.4, 28.5, 35.6, 37.9, 55.8, 70.5, 83.7, 127.1, 127.9, 128.3, 140.5 ppm. HRMS-ESI calcd for $C_{16}H_{25}NO$: $(M+H)^+$ 248.2014; found: 248.2011.

1-[(Butylamino)(phenyl)methyl]cyclopentanol (3l)



Following the general procedure B, the reductive cross-coupling of amide **1j** (177 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3l** (111 mg, 45%) as a pale yellow oil; IR (film) ν_{max}: 3439, 3026, 2957, 2929, 2871, 1490, 1453, 1378, 1200, 1100, 997, 877, 704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.86 (t, J = 7.3 Hz, 3H), 1.22-1.34 (m, 3H), 1.38-1.54 (m, 5H), 1.55-1.65 (m, 1H), 1.68-1.85 (m, 3H), 2.36-2.52 (m, 2H), 3.62 (s, 1H), 7.22-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 13.9, 20.3, 23.3, 23.4, 32.3, 35.8, 37.9, 47.5, 70.4, 83.6, 127.1, 128.0, 128.3, 140.5 ppm. HRMS-ESI calcd for $C_{16}H_{25}NO$: $(M+H)^+$ 248.2014; found: 248.2013.

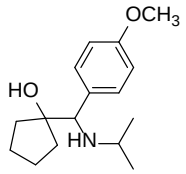
1-[(Isopropylamino)(p-tolyl)methyl]cyclopentanol (3m)



Following the general procedure B, the reductive cross-coupling of amide **1o** (177 mg, 1.0 mmol)

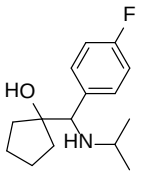
with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3m** (151 mg, 61%) as a pale yellow oil; IR (film) ν_{max}: 3445, 2961, 2869, 1512, 1469, 1438, 1380, 1339, 1175, 1113, 1083, 1003, 873, 822, 794, 731 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.24-1.32 (m, 1H), 1.38 -1.51 (m, 3H), 1.53-1.62 (m, 1H), 1.66-1.84 (m, 3H), 2.33 (s, 3H), 2.60 (septet, J = 6.2 Hz, 1H), 3.34 (br s, 1H), 3.70 (s, 1H), 7.08-7.18 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 21.0, 22.0, 23.3, 23.4, 24.4, 35.5, 37.6, 45.6, 67.0, 83.5, 128.2, 128.7, 136.6, 137.6 ppm. HRMS-ESI calcd for $C_{16}H_{25}NO$: $(M+H)^+$ 248.2014; found: 248.2012.

1-[(Isopropylamino)(4-methoxyphenyl)methyl]cyclopentanol (3n)



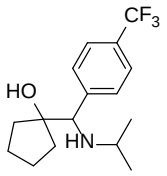
Following the general procedure B, the reductive cross-coupling of amide **1t** (193 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3n** (208 mg, 79% yield) as a pale yellow oil; IR (film) ν_{max}: 3440, 2960, 2870, 1610, 1583, 1511, 1465, 1441, 1380, 1302, 1246, 1177, 1036, 874, 833 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.25-1.31 (m, 1H), 1.41-1.51 (m, 3H), 1.55-1.61 (m, 1H), 1.66-1.82 (m, 3H), 2.61 (septet, J = 6.2 Hz, 1H), 3.69 (s, 1H), 3.80 (s, 3H), 6.83-6.88 (m, 2H), 7.17-7.22 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 22.0, 23.4, 23.5, 24.3, 35.7, 37.8, 45.6, 55.2, 66.7, 83.7, 113.4, 129.3, 132.7, 158.7 ppm. HRMS-ESI calcd for $C_{16}H_{25}NO_2$: $(M+H)^+$ 264.1964; found: 264.1961.

1-[(4-Fluorophenyl)(isopropylamino)methyl]cyclopentanol (3o)



Following the general procedure B, the reductive cross-coupling of amide **1u** (181 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3o** (146 mg, 58% yield) as a white solid, mp: 39-40 °C; IR (film) ν_{max}: 3444, 2926, 2871, 1603, 1508, 1470, 1439, 1381, 1223, 1097, 1003, 873, 836 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.20-1.28 (m, 1H), 1.39-1.52 (m, 3H), 1.53-1.61 (m, 1H), 1.68-1.82 (m, 3H), 2.57 (septet, J = 6.2 Hz, 1H), 3.71 (s, 1H), 6.97-7.04 (m, 2H), 7.23-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 22.0, 23.4, 23.5, 24.3, 35.9, 38.1, 45.6, 66.7, 83.6, 114.8 (d, J_{F-C} = 20.9 Hz), 129.8 (d, J_{F-C} = 7.8 Hz), 136.5 (d, J_{F-C} = 3.1 Hz), 162.0 (d, J_{F-C} = 245.2 Hz) ppm. HRMS-ESI calcd for $C_{15}H_{22}FNO$: $(M+H)^+$ 252.1764; found: 252.1764.

1-[(Isopropylamino)(4-(trifluoromethyl)phenyl)methyl]cyclopentanol (3p)



Following the general procedure B, the reductive cross-coupling of amide **1v** (231 mg, 1.0 mmol) with *c*-pentanone gave, after flash column chromatography on silica gel (eluent: EtOAc/Hexane/NH_3•H_2O = 1/6/0.0 to 1/4/0.05), the desired β-amino alcohol **3p** (160 mg, 53% yield) as a pale yellow oil; IR (film) ν_{max}: 3434, 2964, 2873, 1619, 1471, 1383, 1326, 1164, 1124, 1068, 1017, 838 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, J = 6.2 Hz, 3H), 1.01 (d, J = 6.2 Hz, 3H), 1.19-1.27 (m, 1H), 1.38-1.45 (m, 1H), 1.46-1.56 (m, 2H), 1.57-1.64 (m, 1H), 1.71-1.85 (m, 3H), 2.56 (septet, J = 6.2 Hz, 1H), 3.78 (s, 1H), 7.43 (d, J = 8.1 Hz, 2H), 7.58 (d, J = 8.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 21.8, 23.3, 23.4, 24.2, 36.2, 38.3, 45.8, 67.2, 83.5, 124.2 (d, J_{F-C} = 270.3 Hz), 124.9 (q, J_{F-C} = 3.9 Hz), 128.8, 129.4 (q, J_{F-C} = 32.3 Hz), 144.9 ppm. HRMS-ESI calcd for $C_{16}H_{22}F_3NO$: $(M+H)^+$ 302.1732; found: 302.1731.

References

1. (a) E. J. Enholm, D. C. Forbes, D. P. Holub, *Synth. Commun.*, 1990, **20**, 981; (b) M. Periasamy, G. Srinivas, G. V. Karunakar, P. Bharathi, *Tetrahedron Lett.*, 1999, **40**, 7577; (c) I. Ugi, U. Fetzer, *Chem. Ber.*, 1961, **94**, 2239; (d) J. Roland, *Helv. Chim. Acta.*, 1956, **39**, 111.
2. M. Kim, B. W. Knettle, A. Dahlén, G. Hilmersson, R. A Flowers II, *Tetrahedron*, 2003, **59**, 10397.
3. R. Annunziata, M. Benaglia, M. Caporale, L. Raimondi, *Tetrahedron: Asymmetry*, 2002, **13**, 2727.

^{1}H NMR spectra of *meso*/*dl* mixture, *meso*-, and *dl*-diastereomers of compounds **2b** and **2f.**

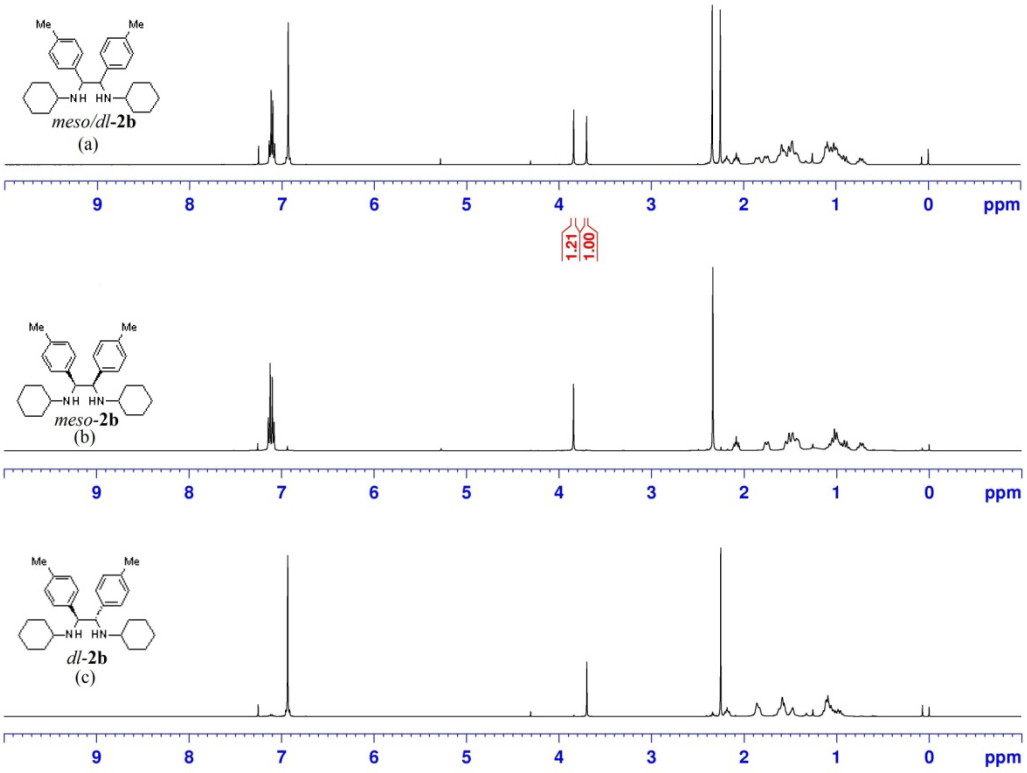


Figure 1: ^{1}H NMR spectra of a diastereomeric mixture of **2b**, *meso*-**2b**, and *dl*-**2b**.

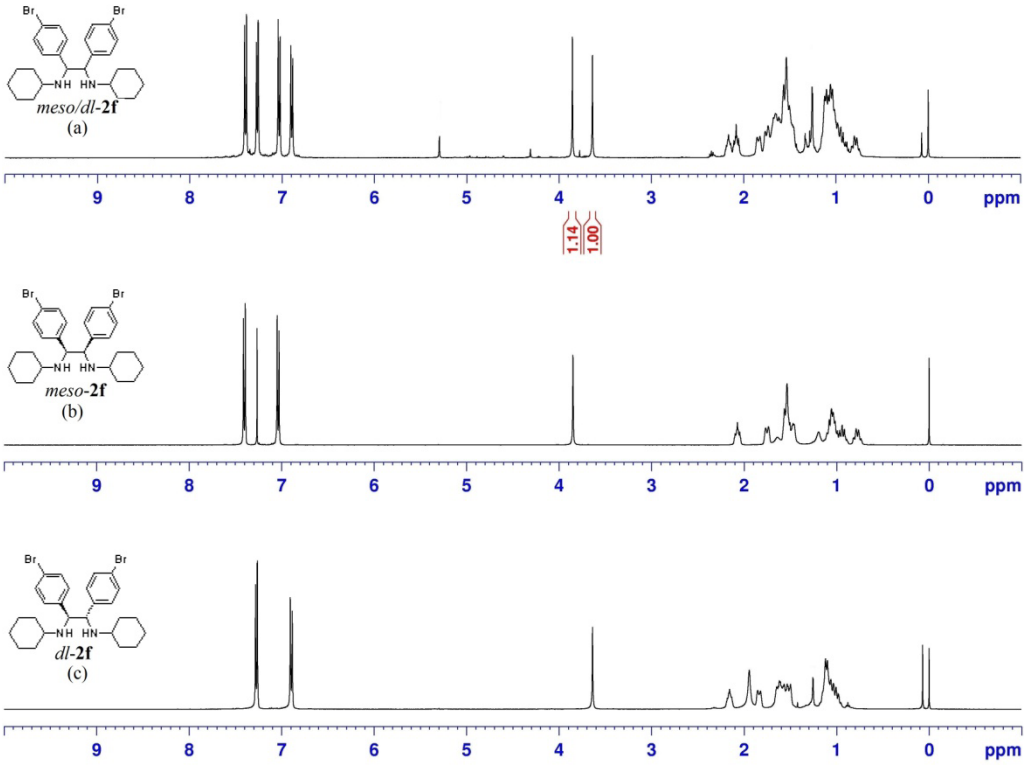
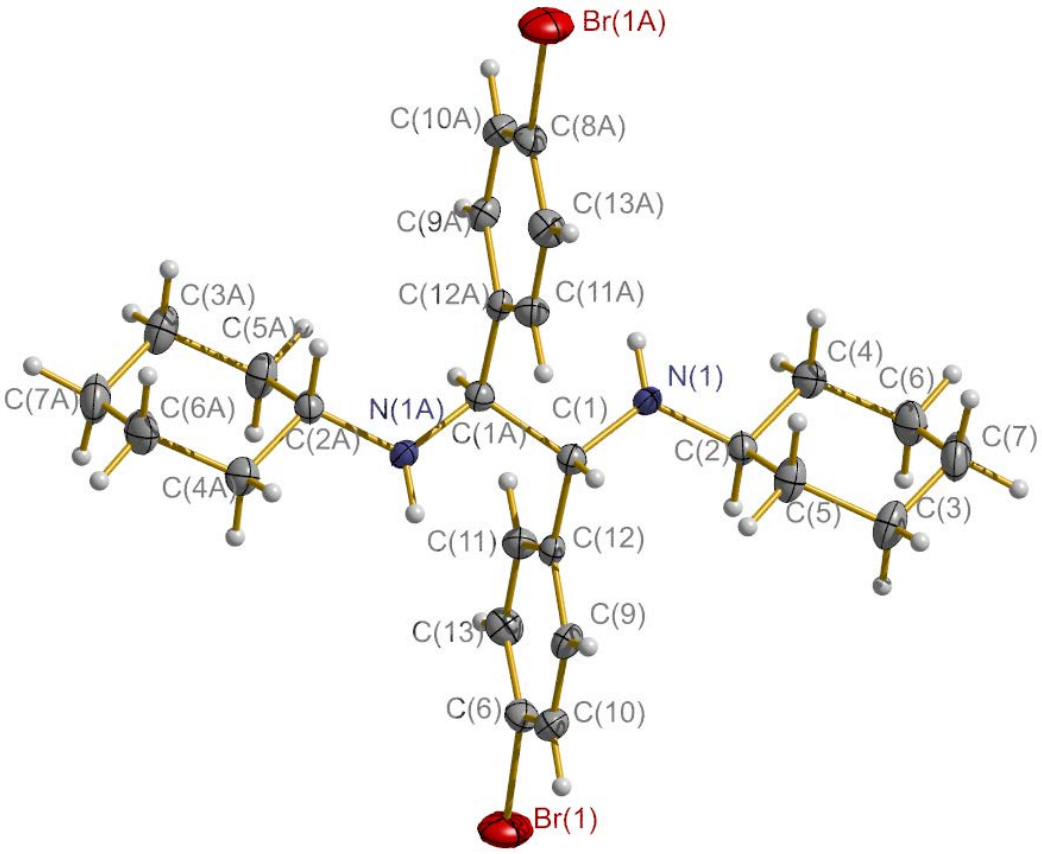


Figure 2: ^{1}H NMR spectra of a diastereomeric mixture of **2f**, *meso*-**2f**, and *dl*-**2f**.

X-Ray structure of *meso*-2f:

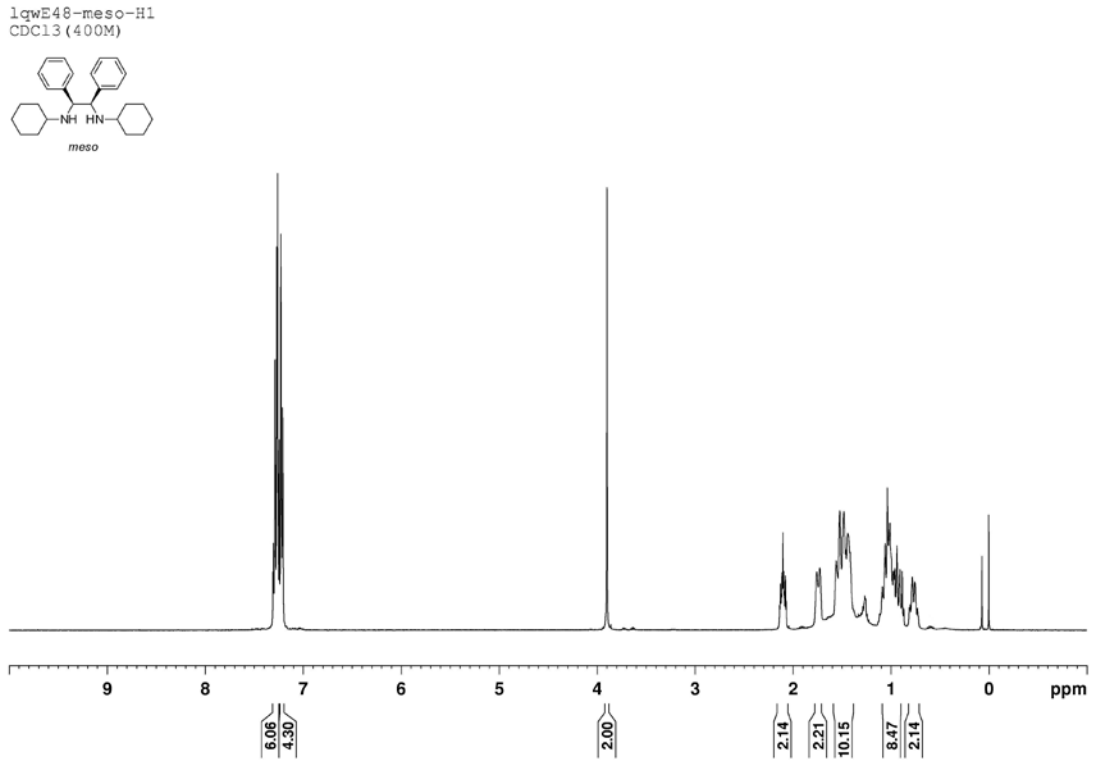


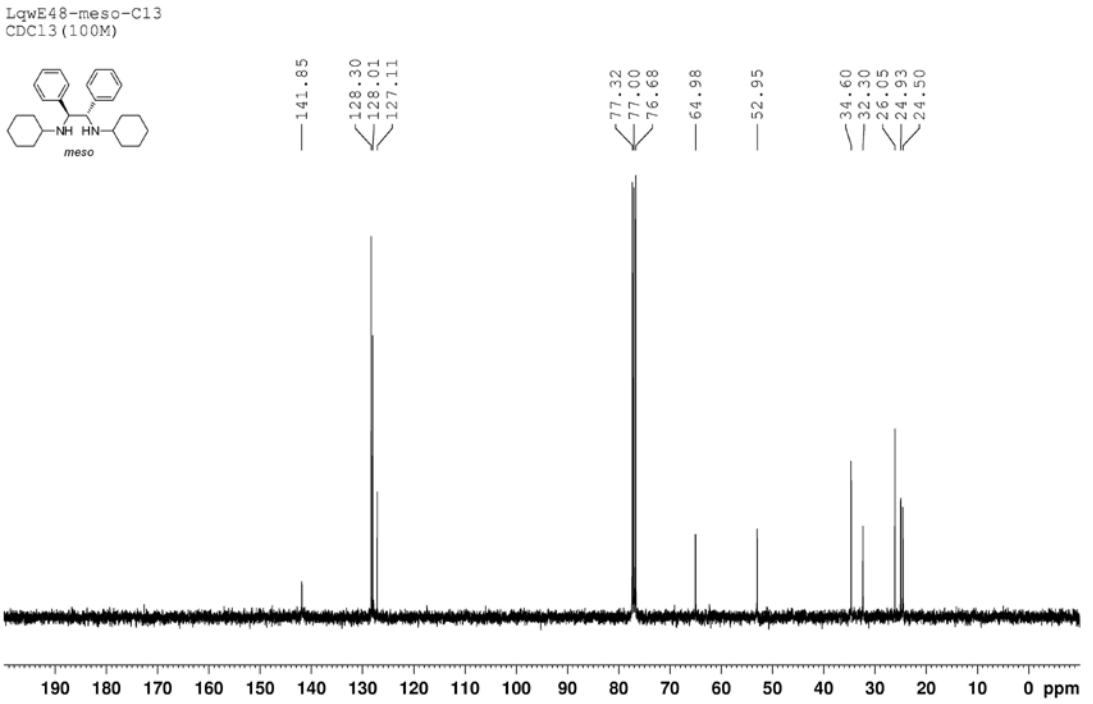
Summary of Data for *meso*-**2f**

Compound Name: *meso*-1,2-bis(4-bromophenyl)-N1,N2-dicyclohexylethane-1,2-diamine
Formula: C26 H34 Br2 N2
Unit Cell Parameters: a 5.9858(5) b 10.0355(12) c 10.1711(12) P-1
CCDC number 1019290.

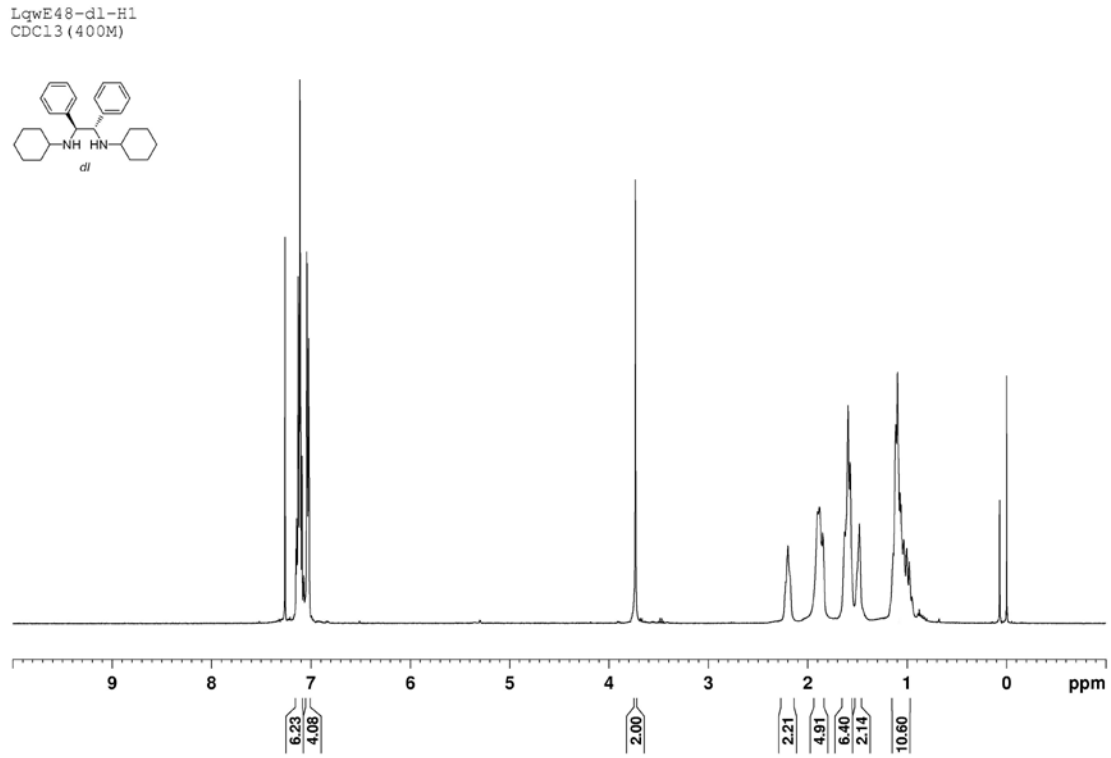
Identification code	m1
Empirical formula	C26 H34 Br2 N2
Formula weight	534.37
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 5.9858(5) A alpha = 93.862(10) deg.
	b = 10.0355(12) A beta = 91.455(9) deg.
	c = 10.1711(12) A gamma = 96.575(9) deg.

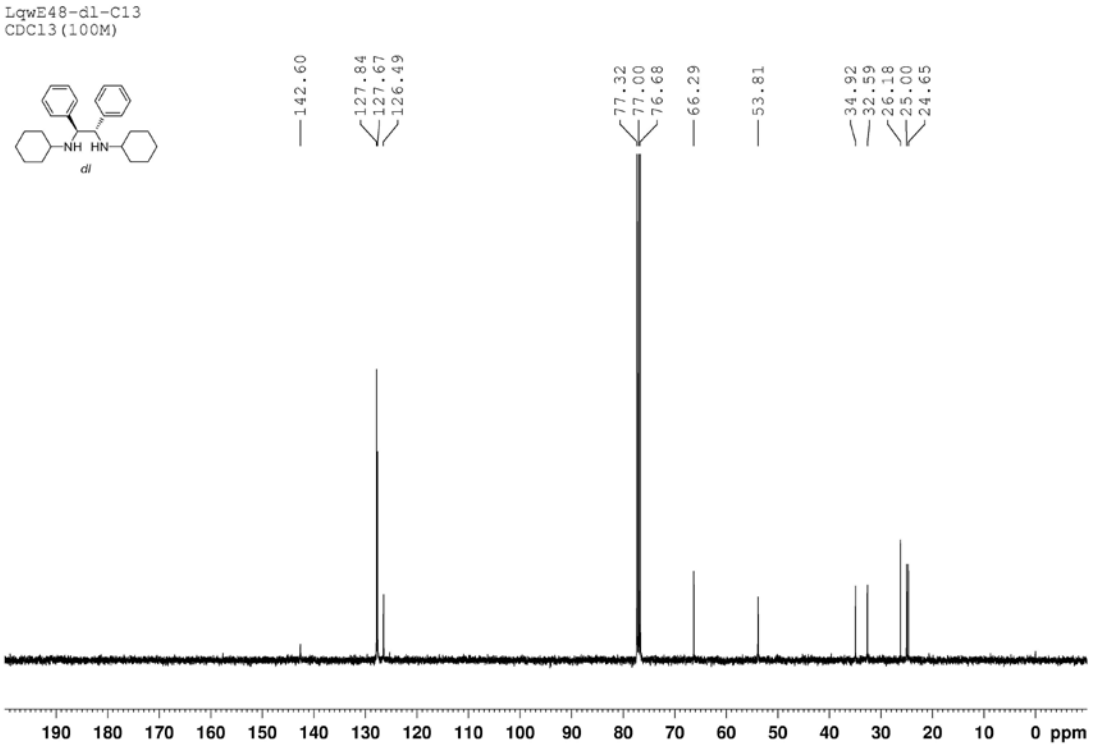
^{1}H and ^{13}C NMR spectra of *meso*-**2a**:



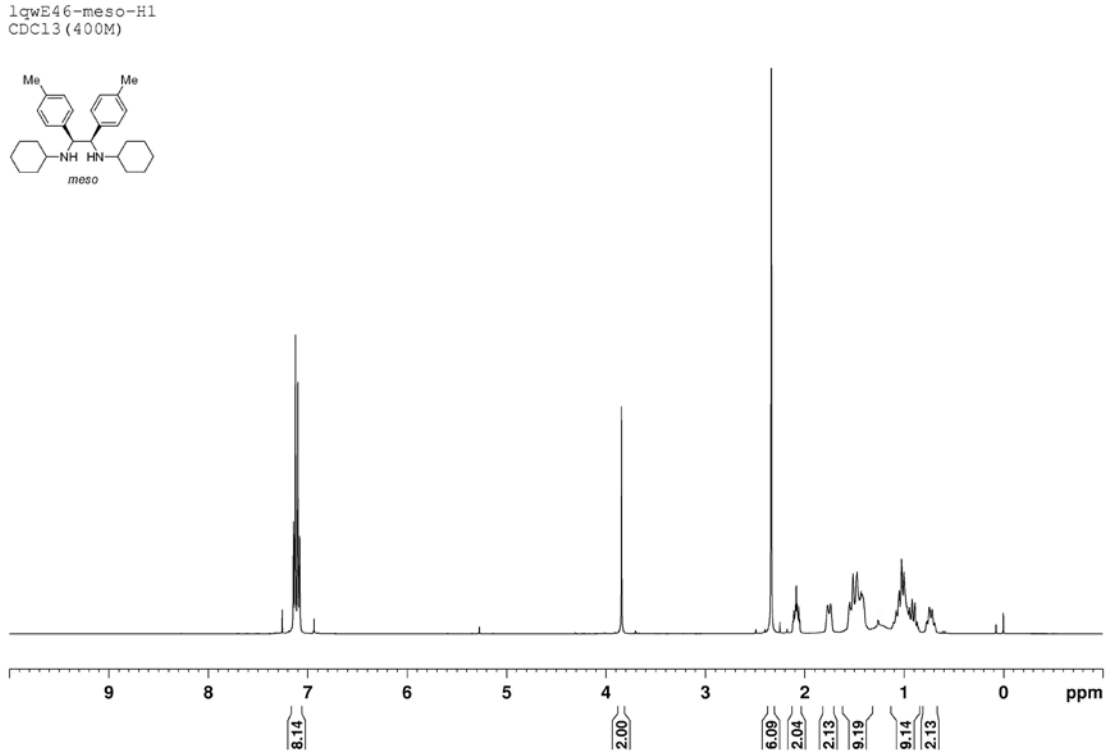


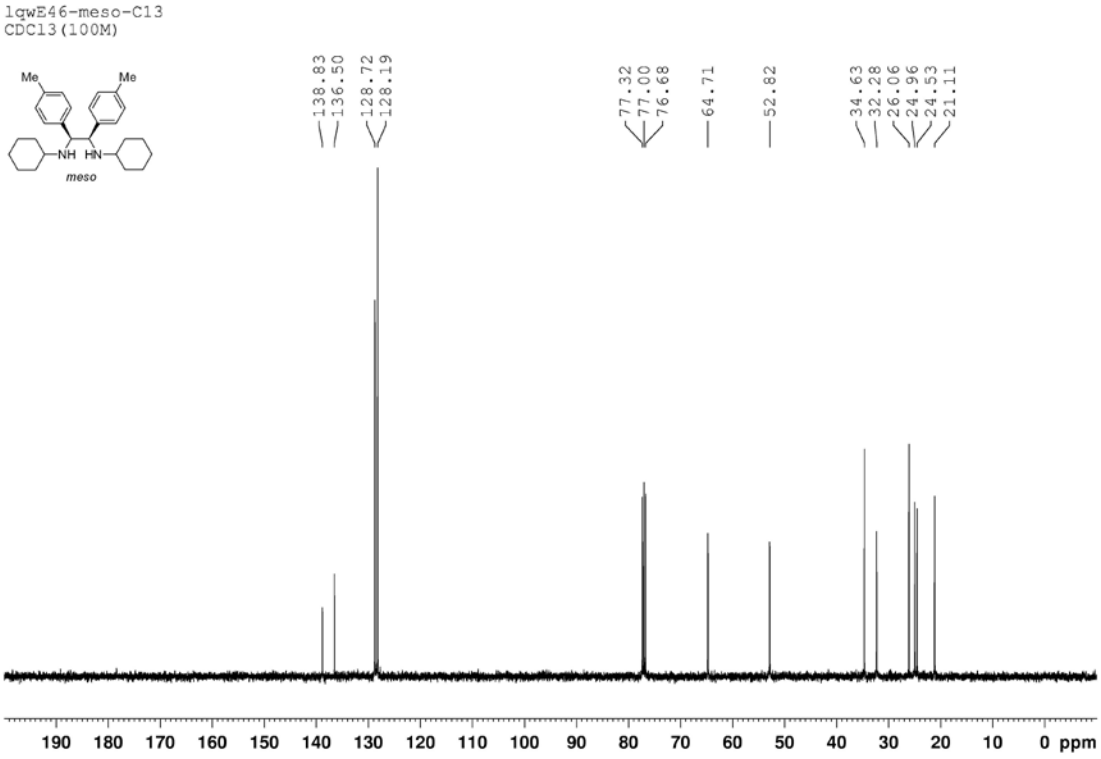
^{1}H and ^{13}C NMR spectra of *dl*-**2a**:



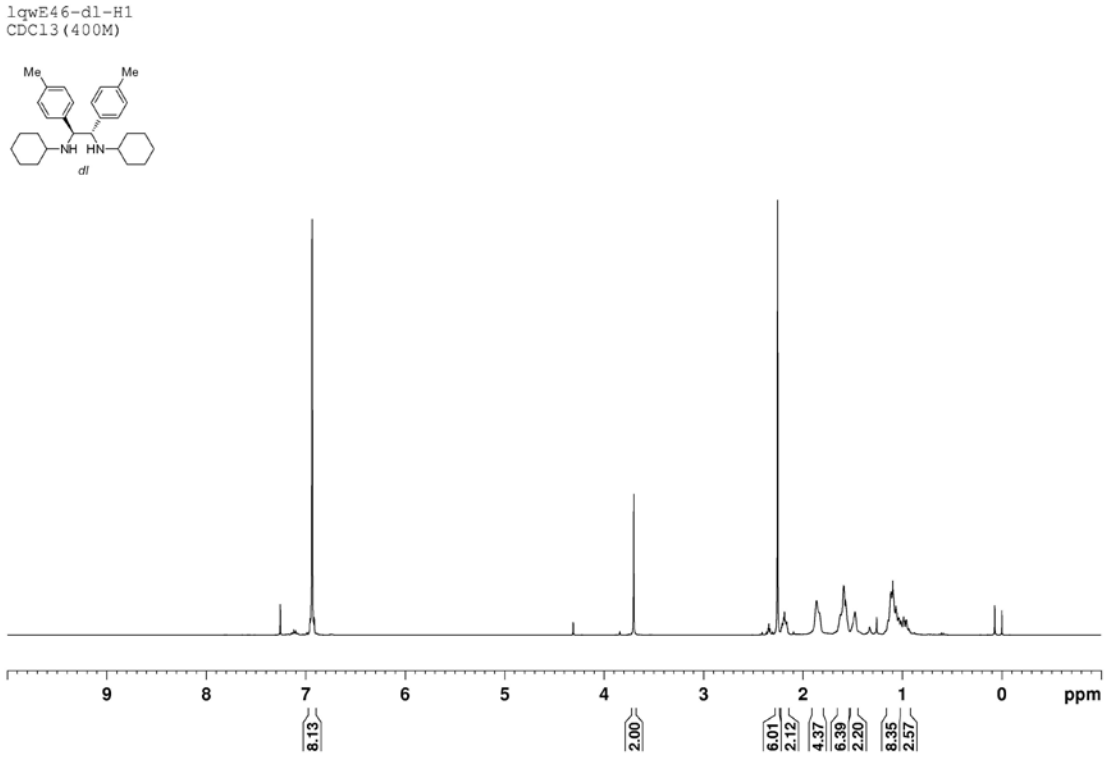


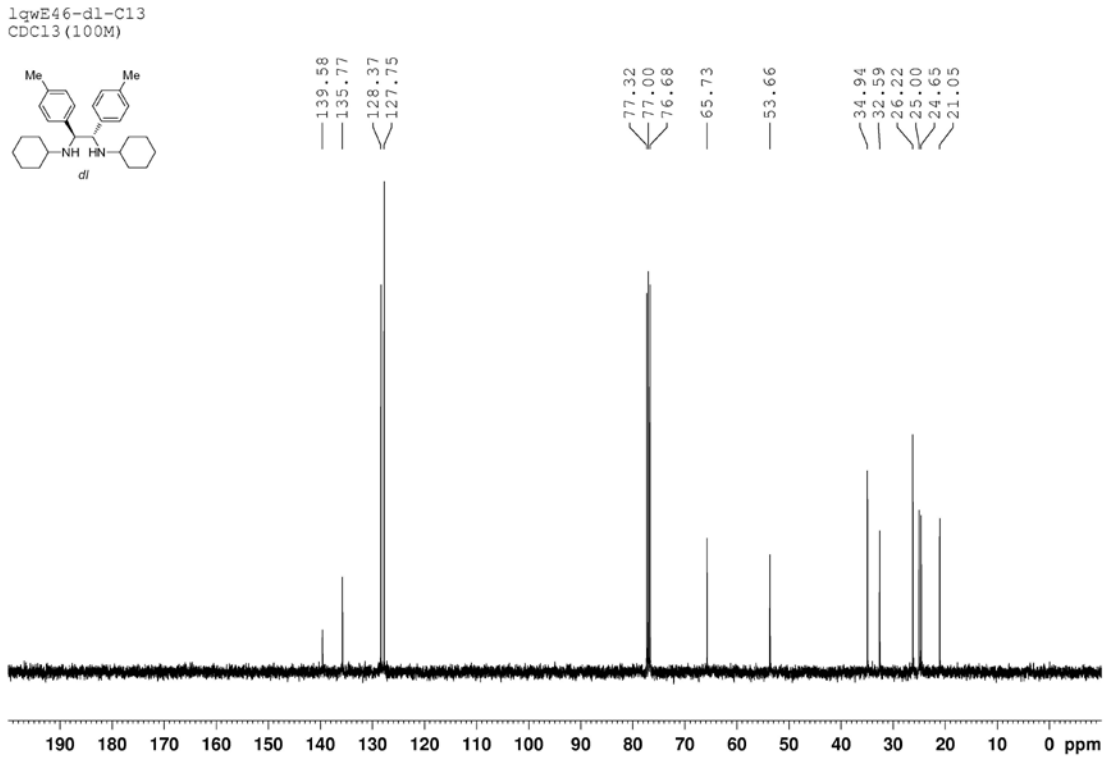
^{1}H and ^{13}C NMR spectra of *meso*-**2b**:



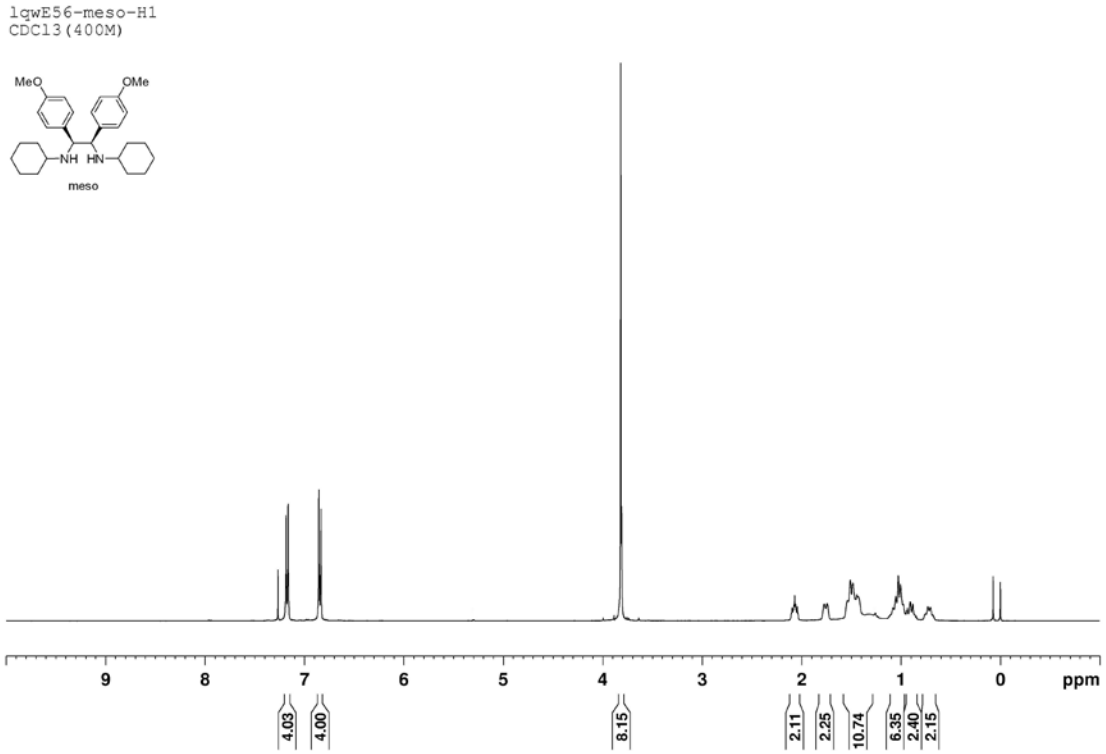


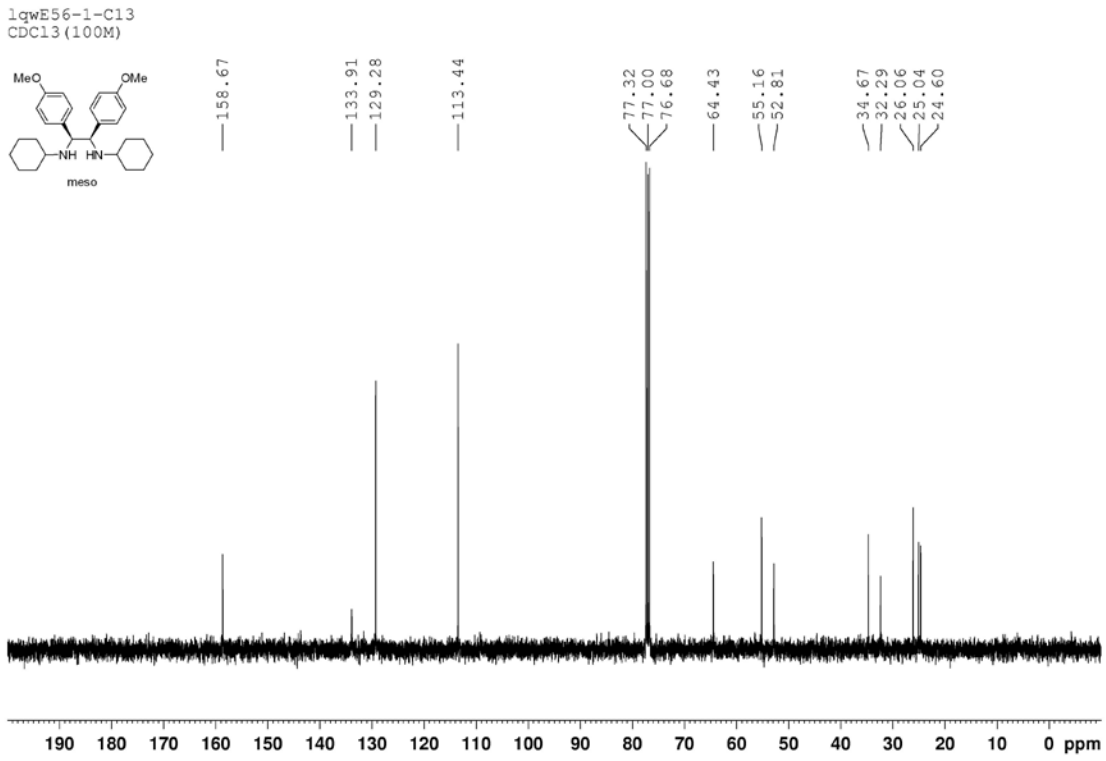
^{1}H and ^{13}C NMR spectra of *dl*-**2b**:



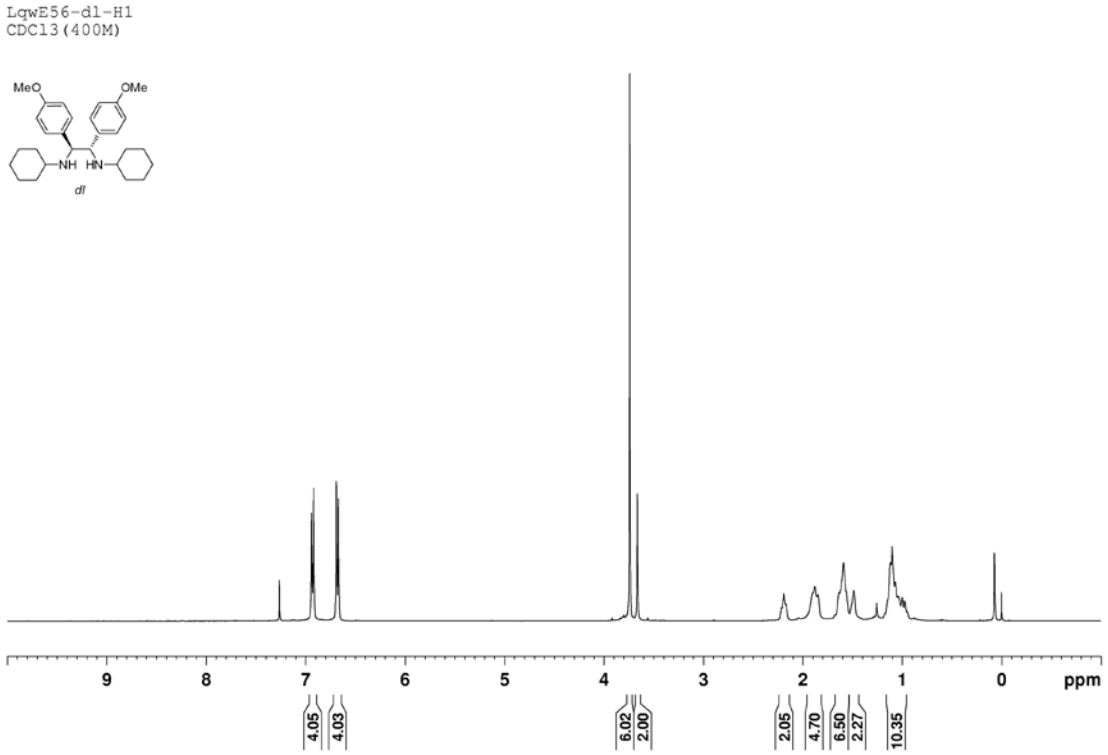


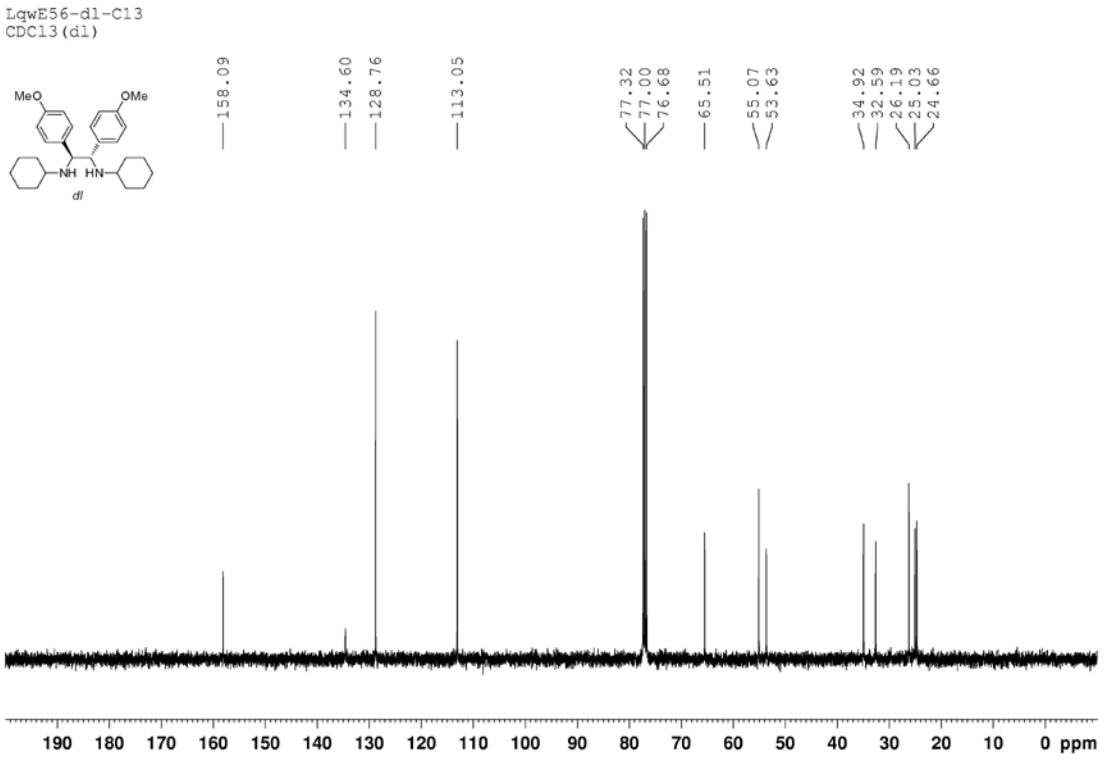
^{1}H and ^{13}C NMR spectra of *meso*-**2c**:



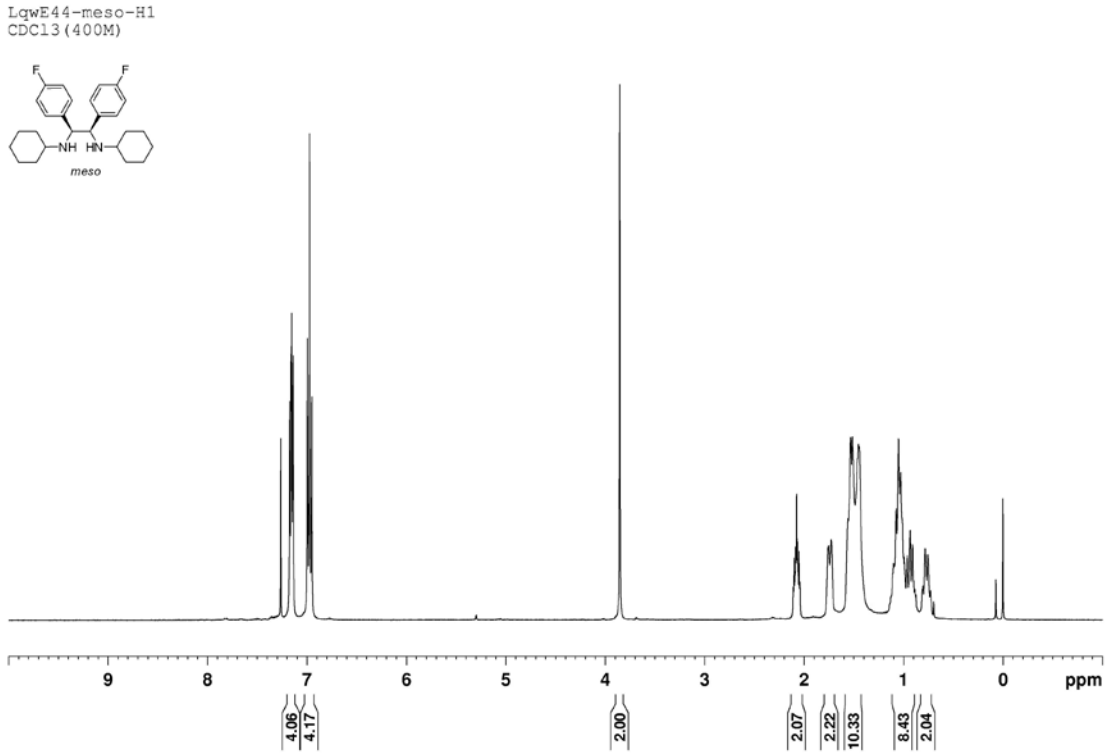


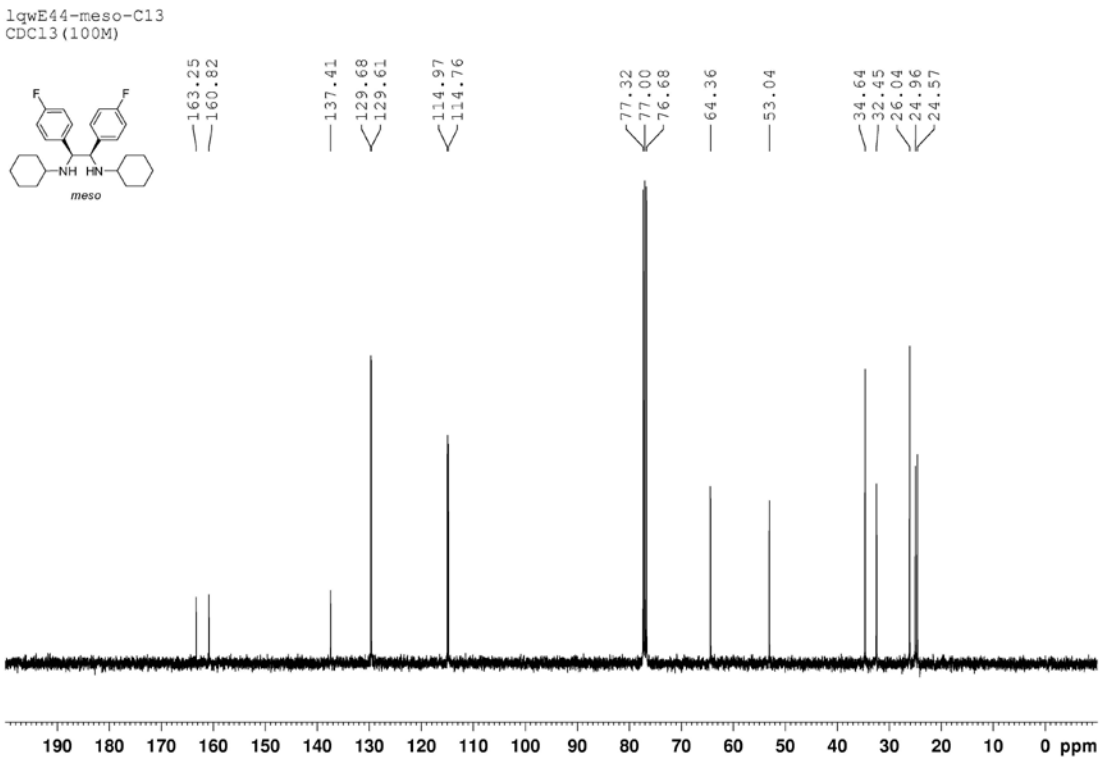
^{1}H and ^{13}C NMR spectra of *dl*-**2c**:



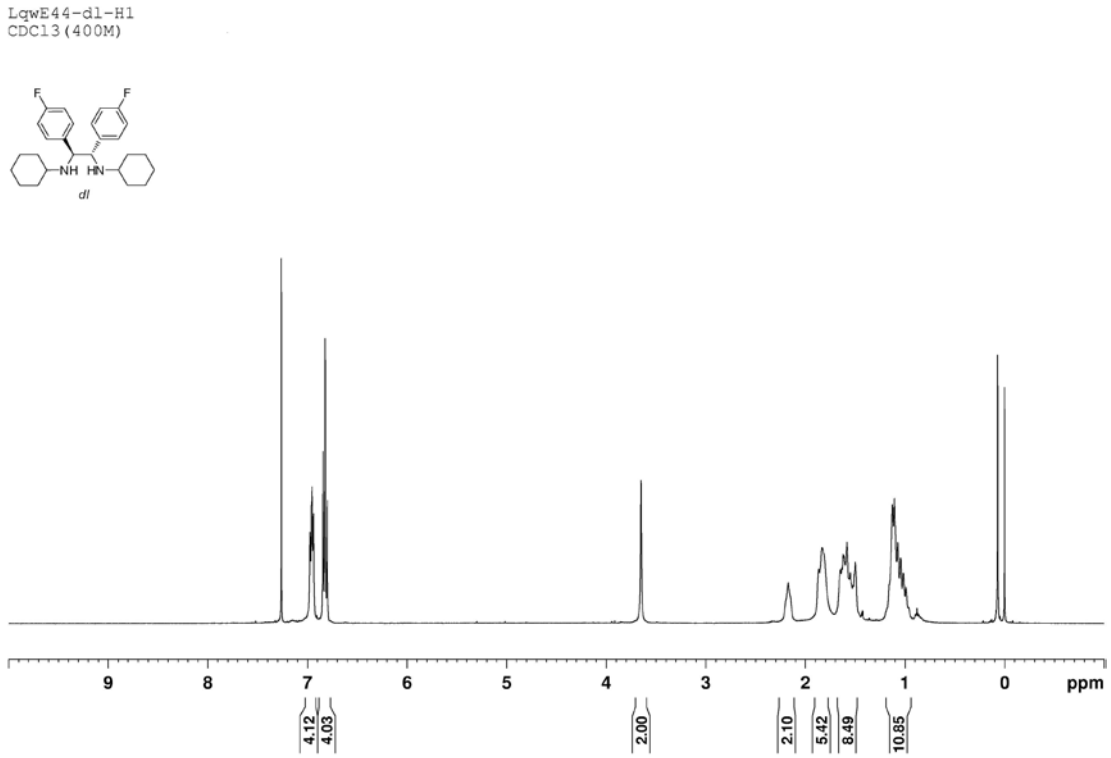


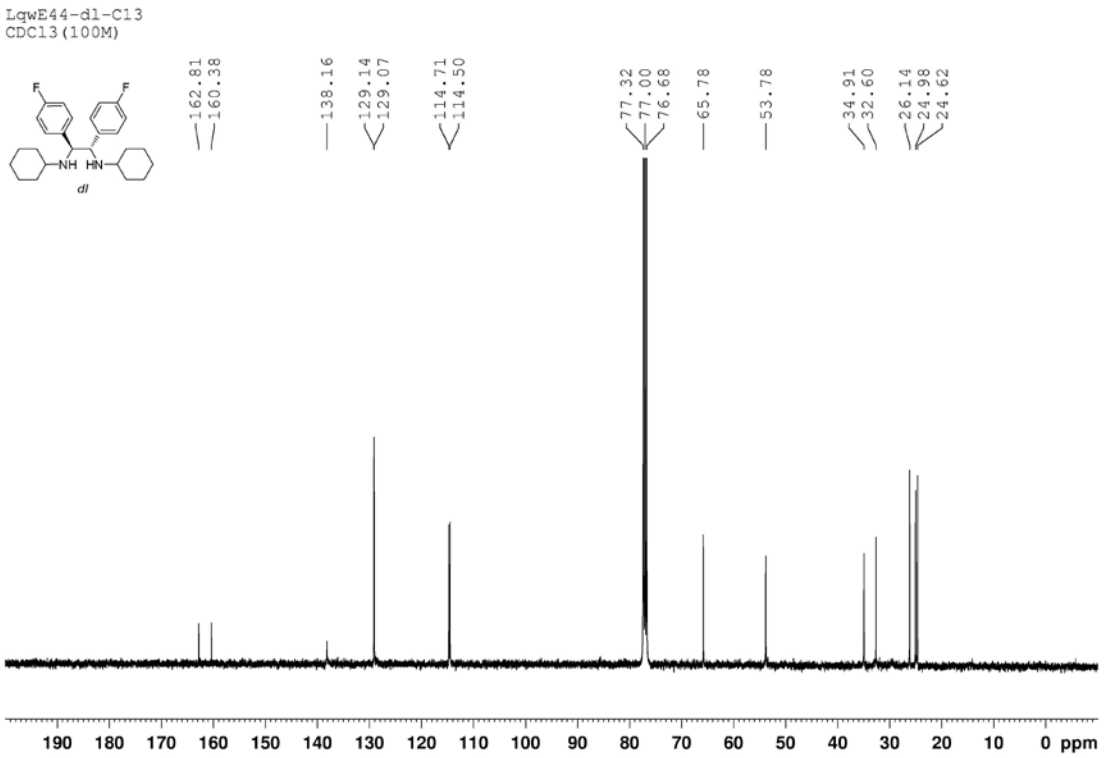
^{1}H and ^{13}C NMR spectra of *meso*-**2d**:



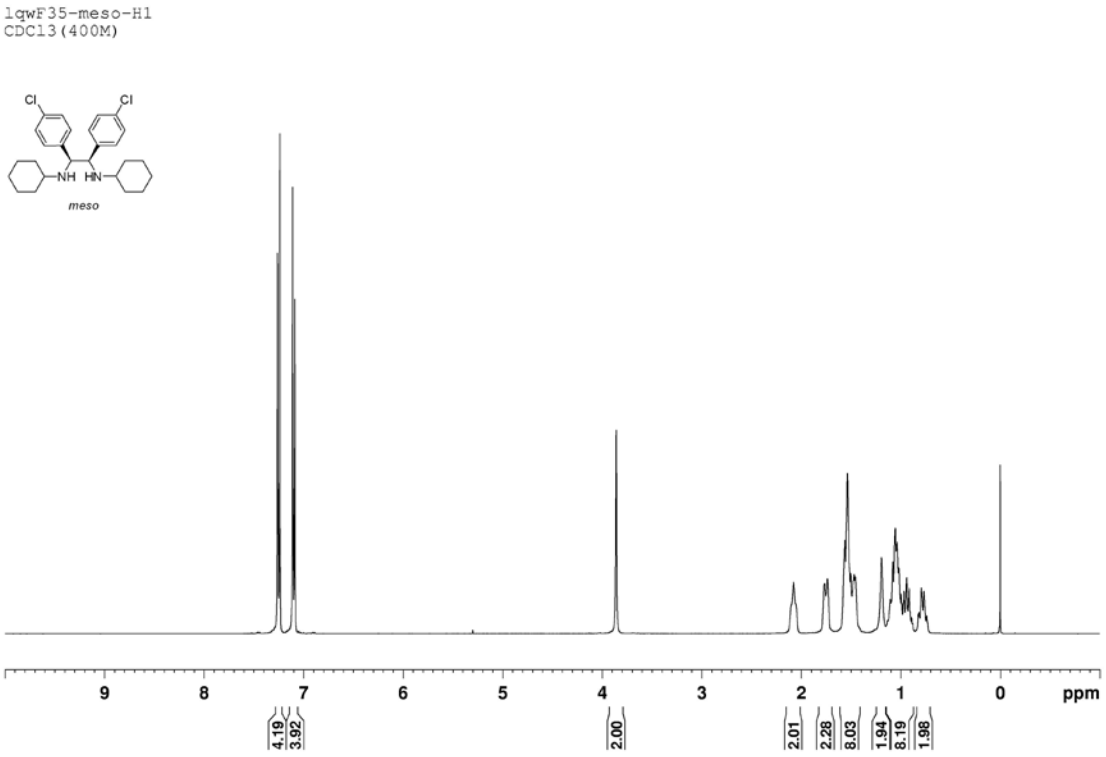


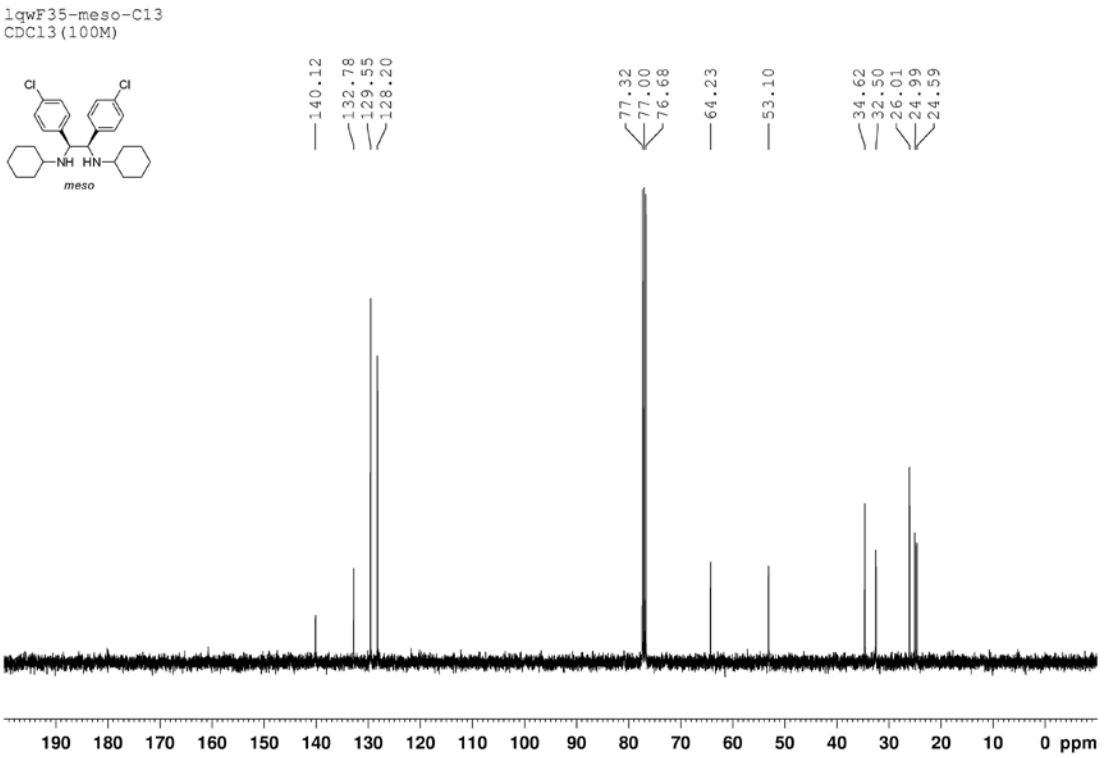
^{1}H and ^{13}C NMR spectra of *dl*-**2d**:



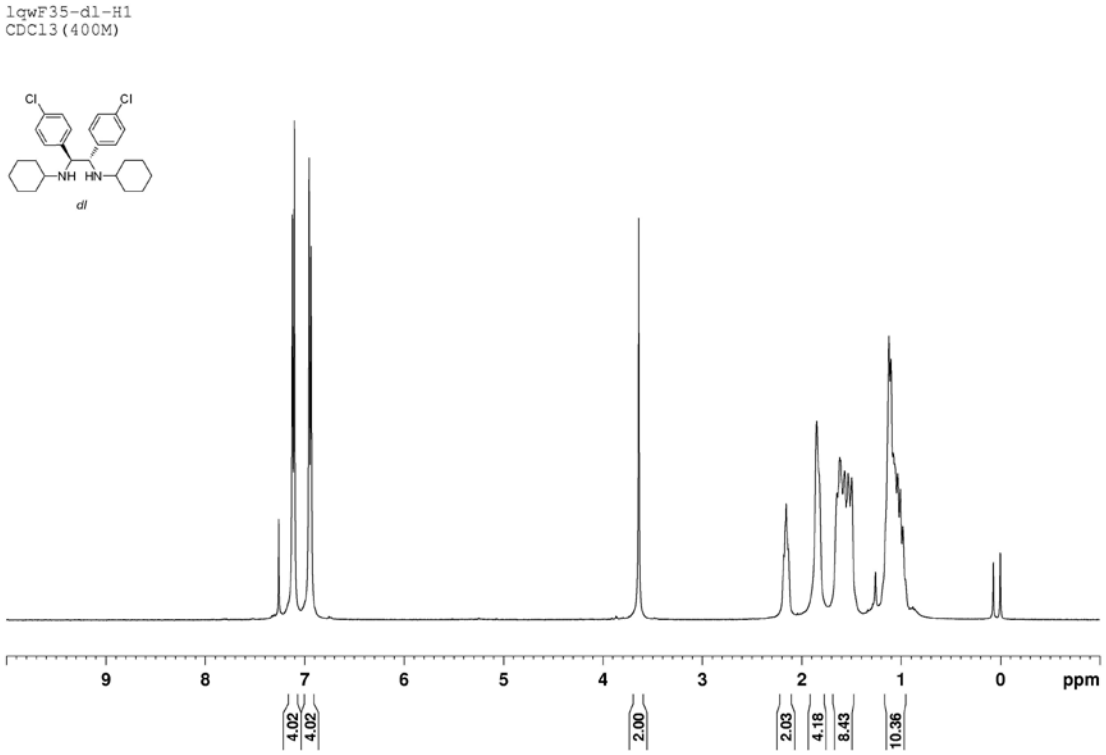


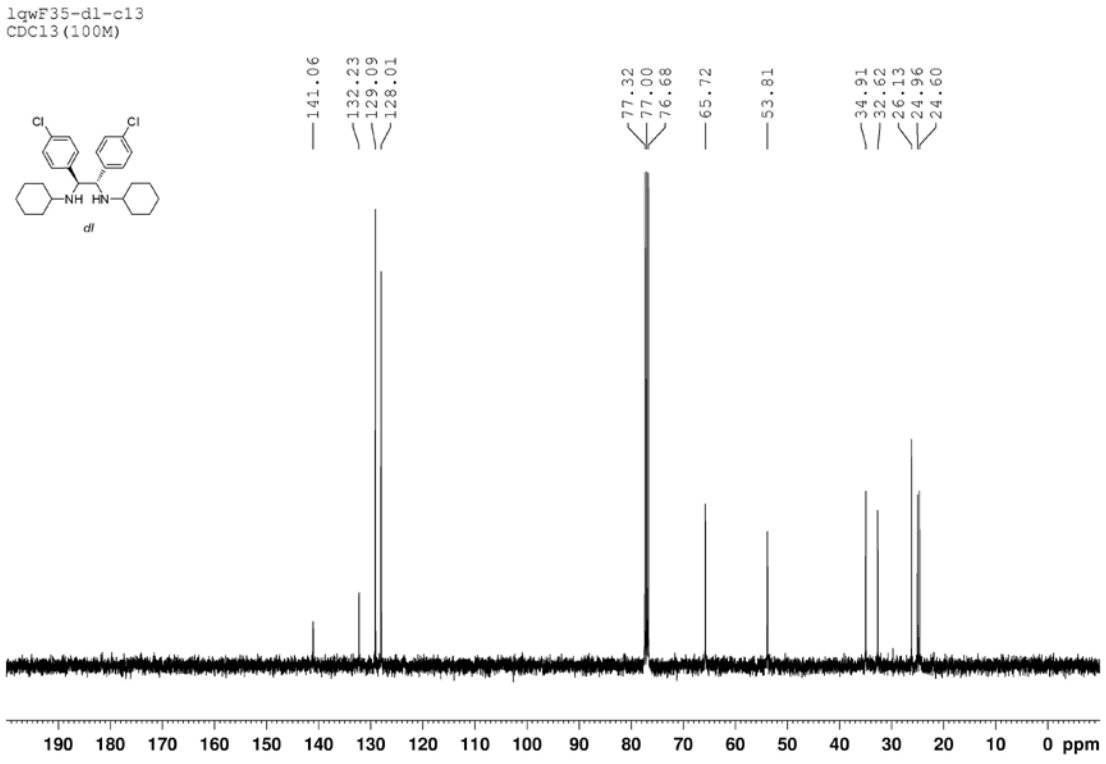
^{1}H and ^{13}C NMR spectra of *meso*-**2e**:



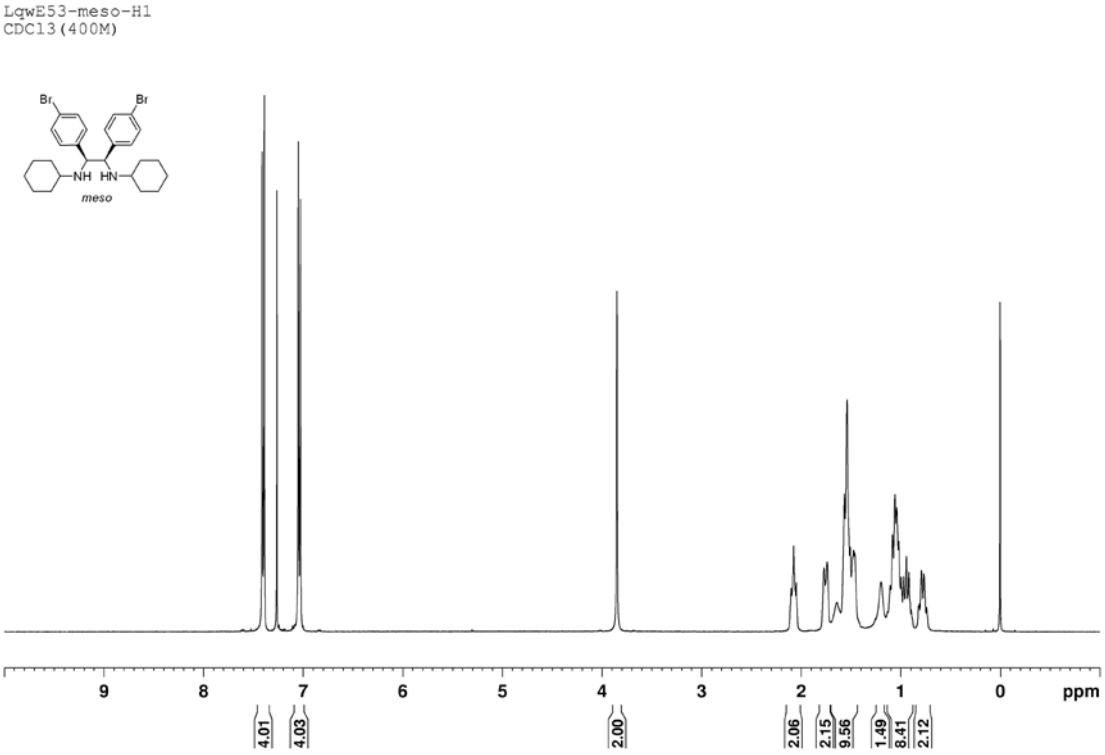


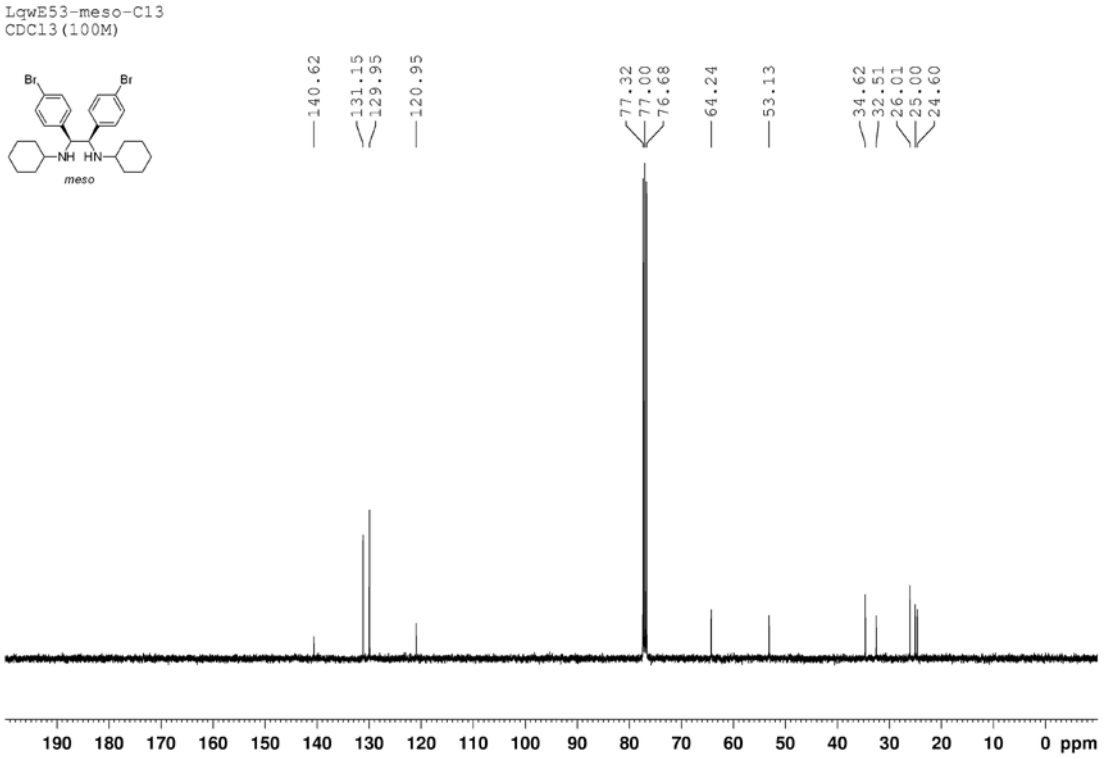
^{1}H and ^{13}C NMR spectra of *dl*-**2e**:



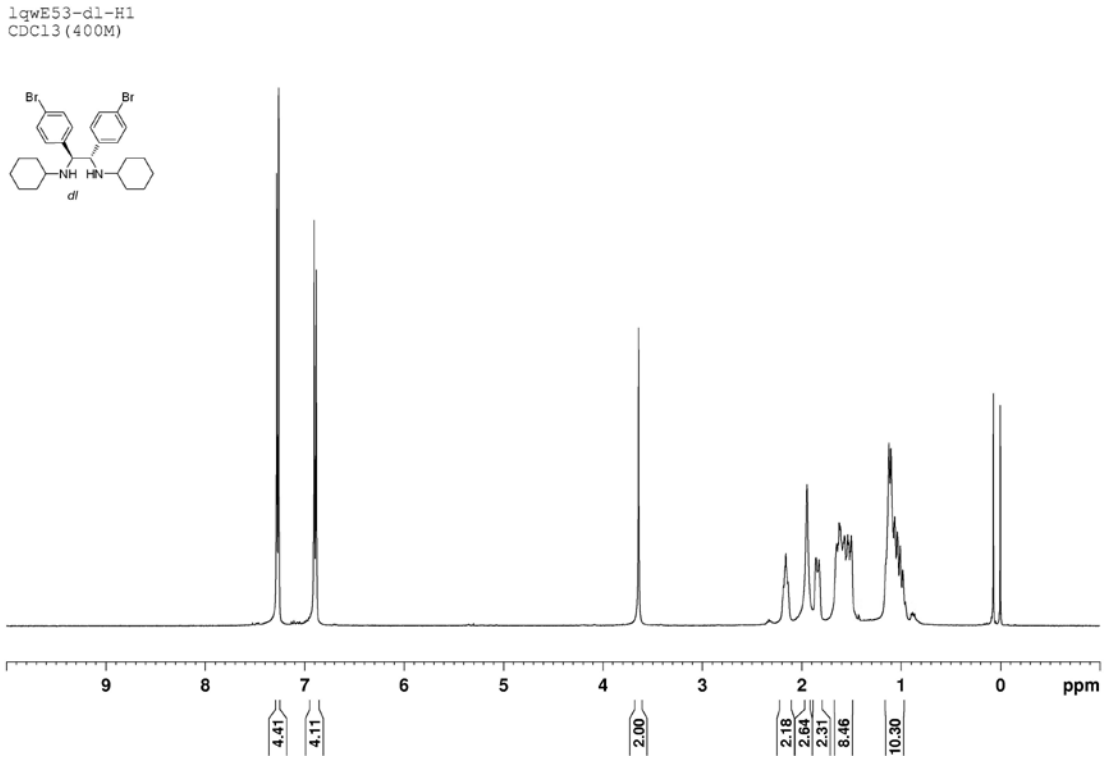


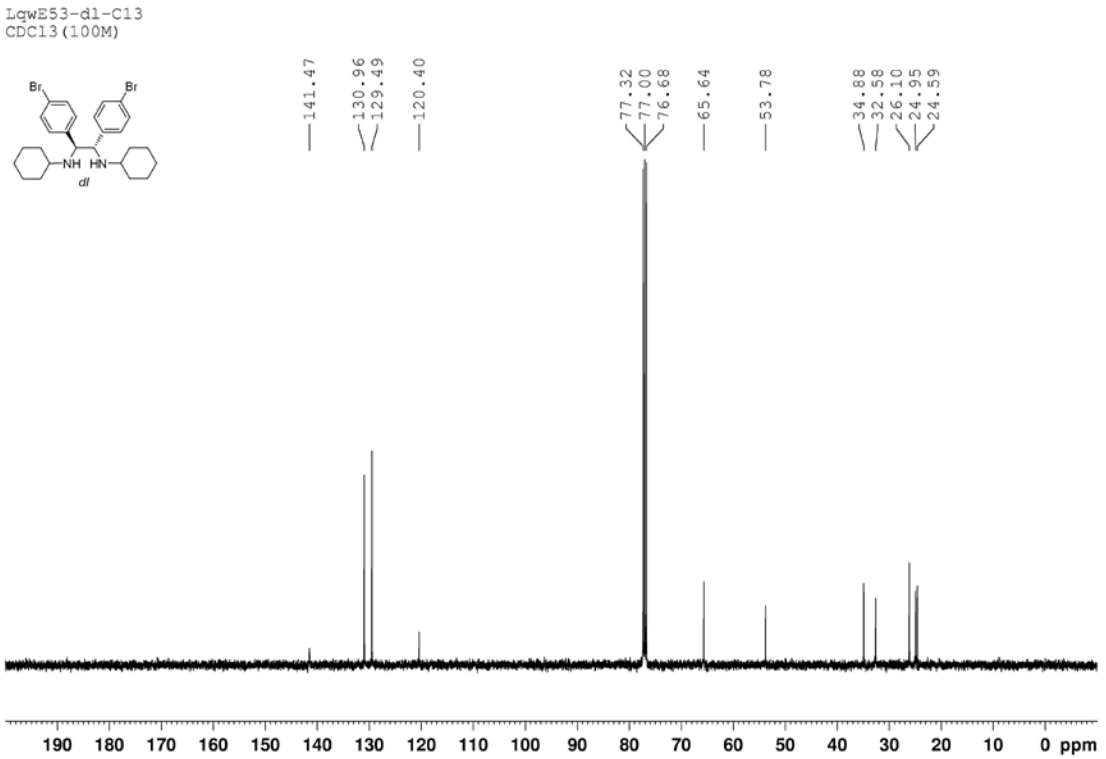
^{1}H and ^{13}C NMR spectra of *meso*-**2f**:



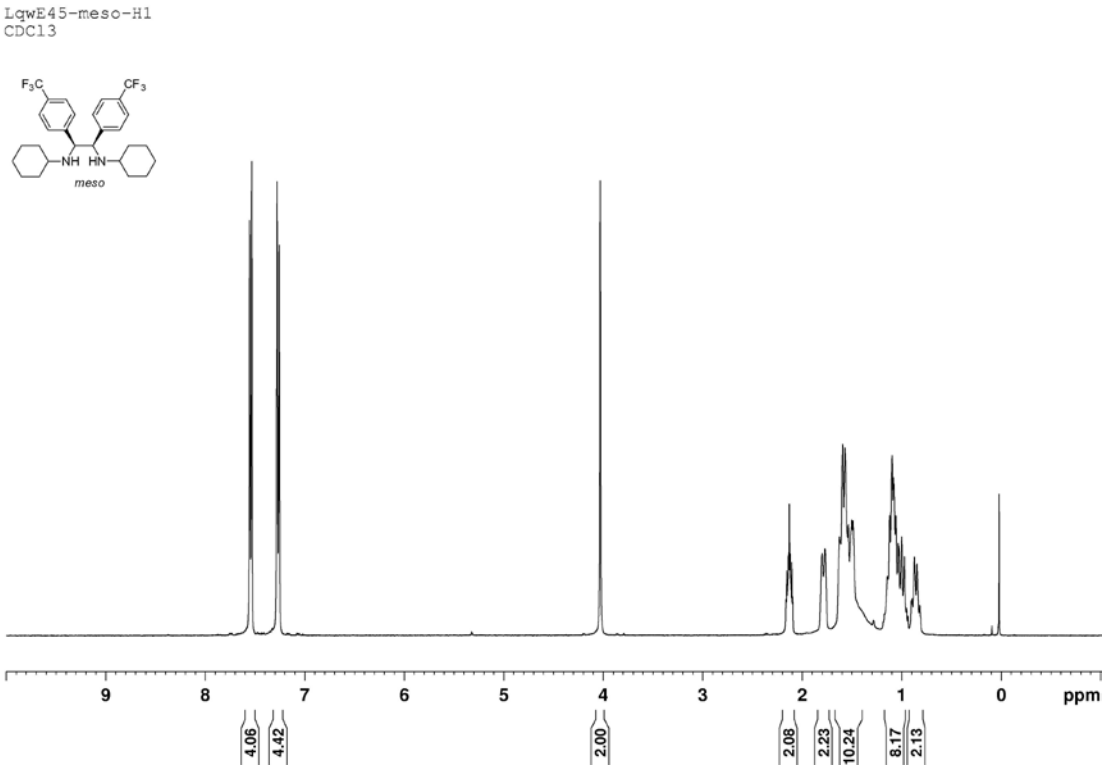


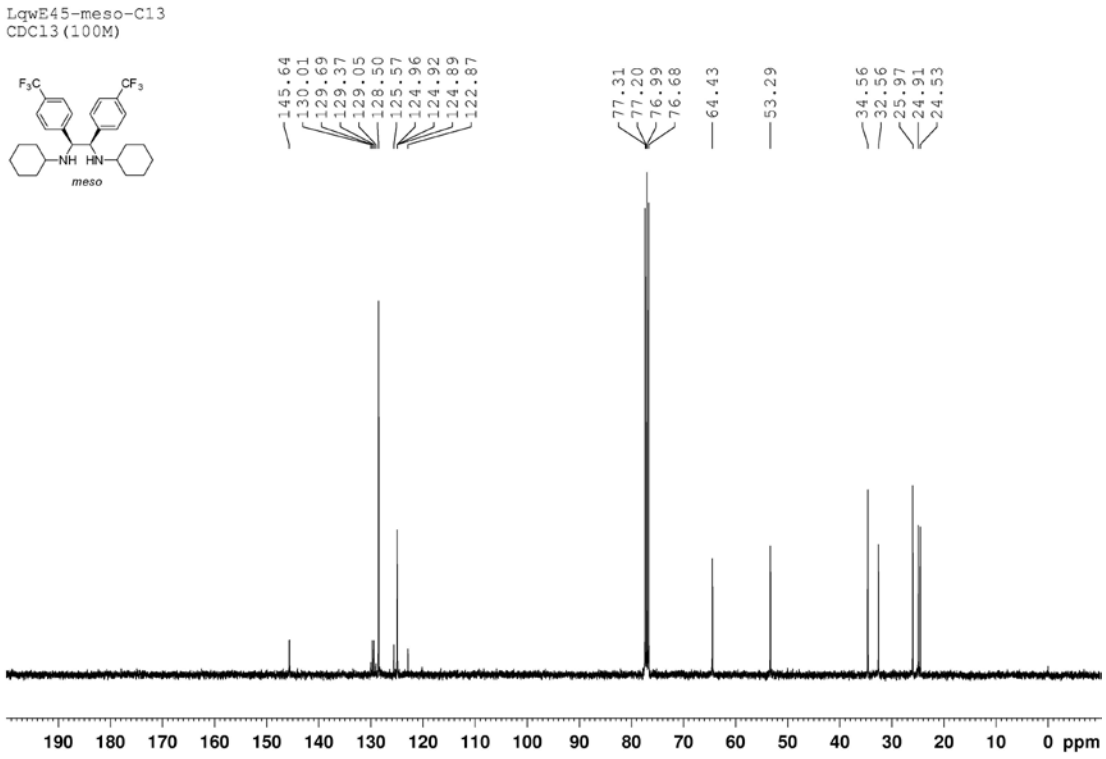
^{1}H and ^{13}C NMR spectra of *dl*-**2f**:



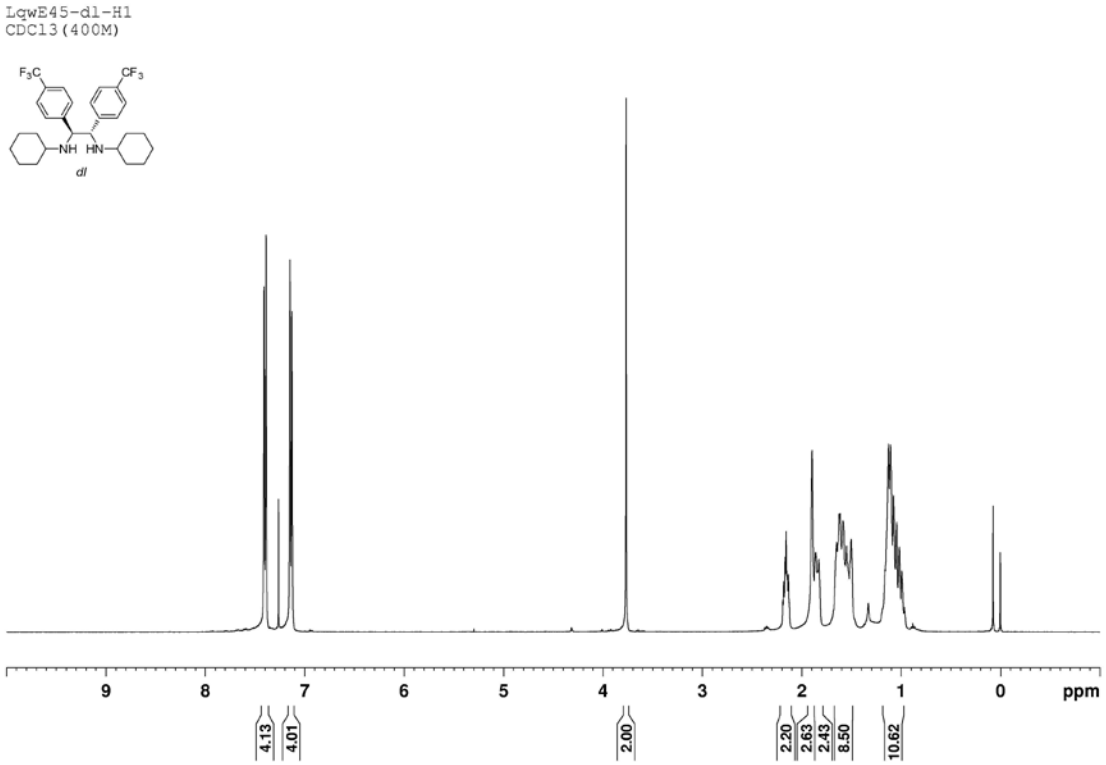


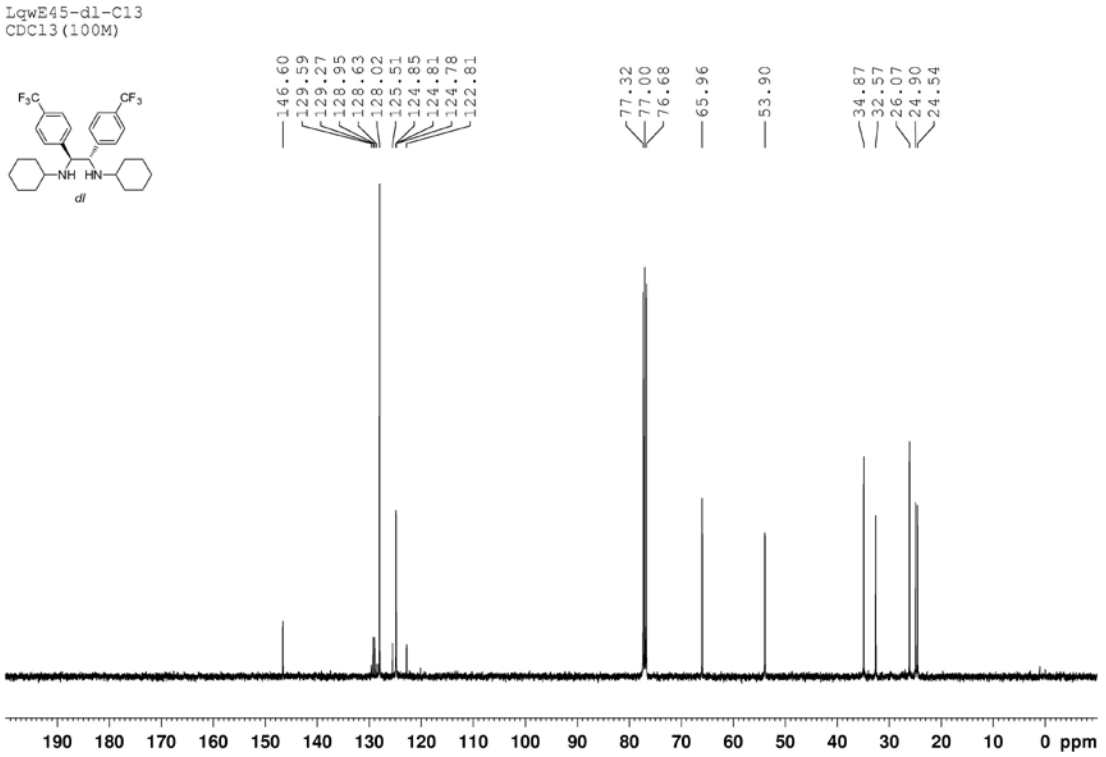
^{1}H and ^{13}C NMR spectra of *meso*-**2g**:



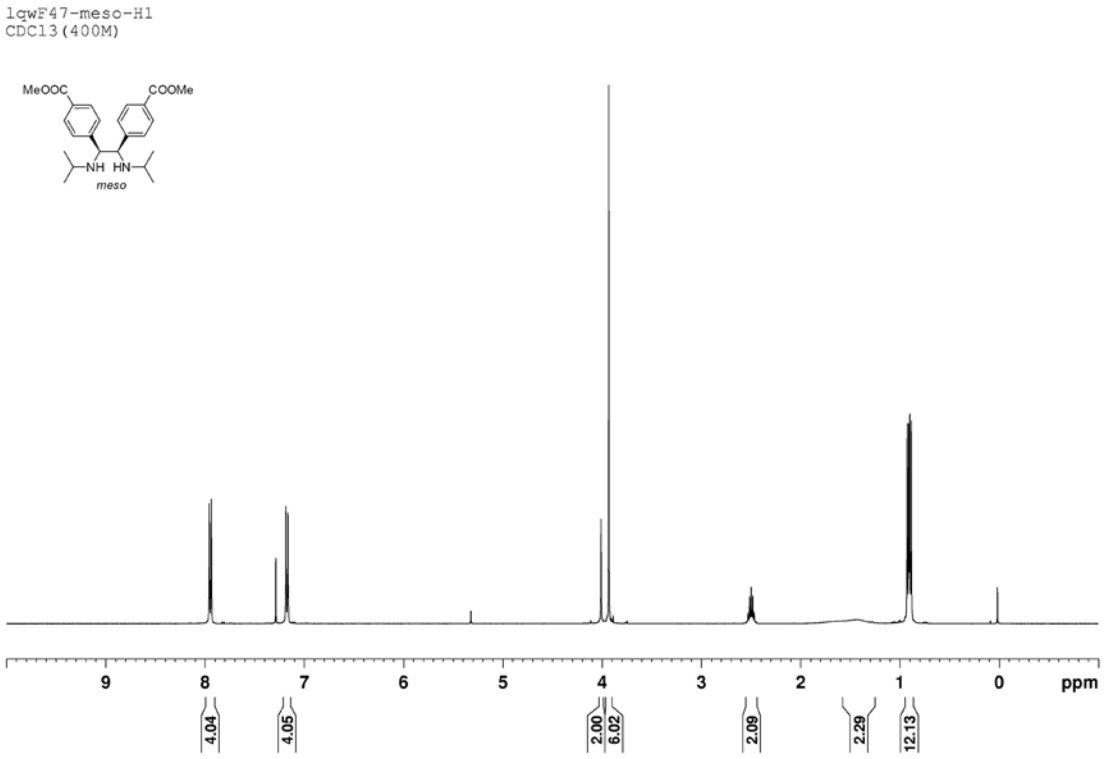


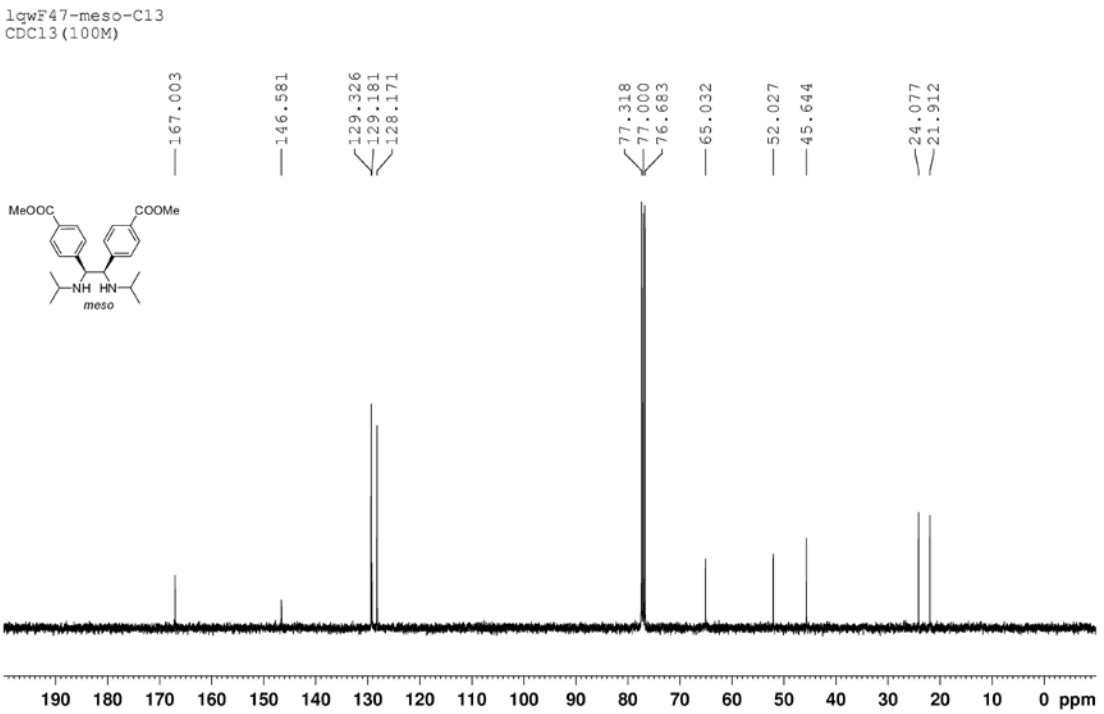
^{1}H and ^{13}C NMR spectra of *dl*-**2g**:



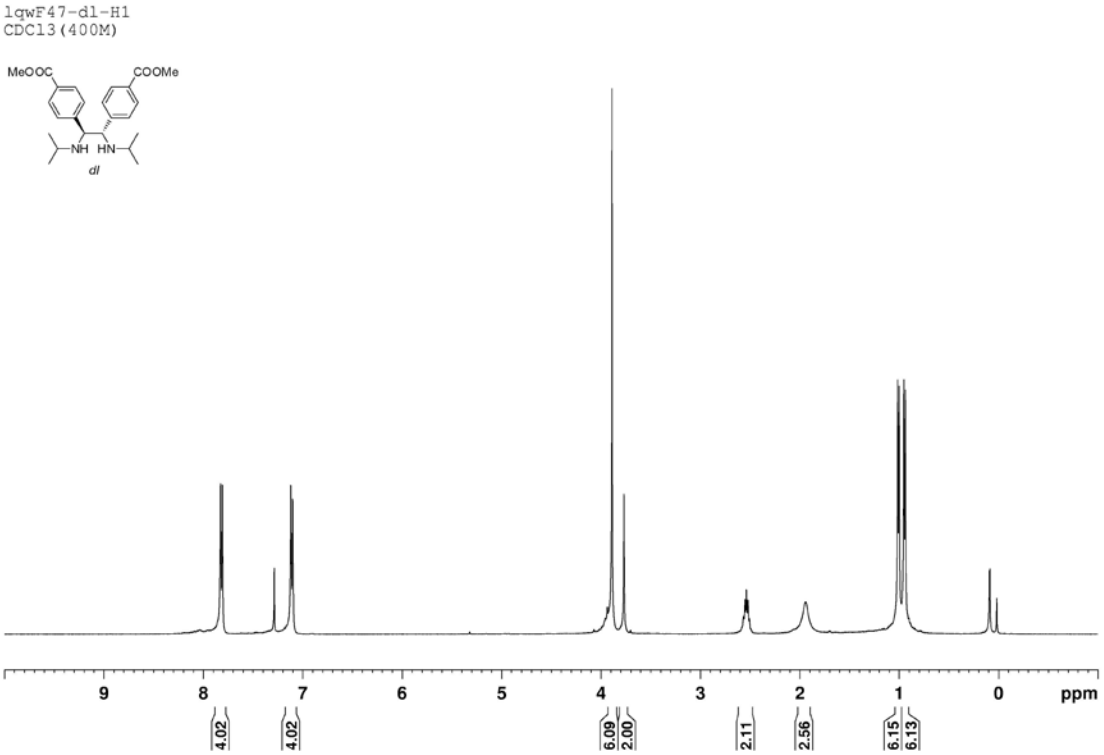


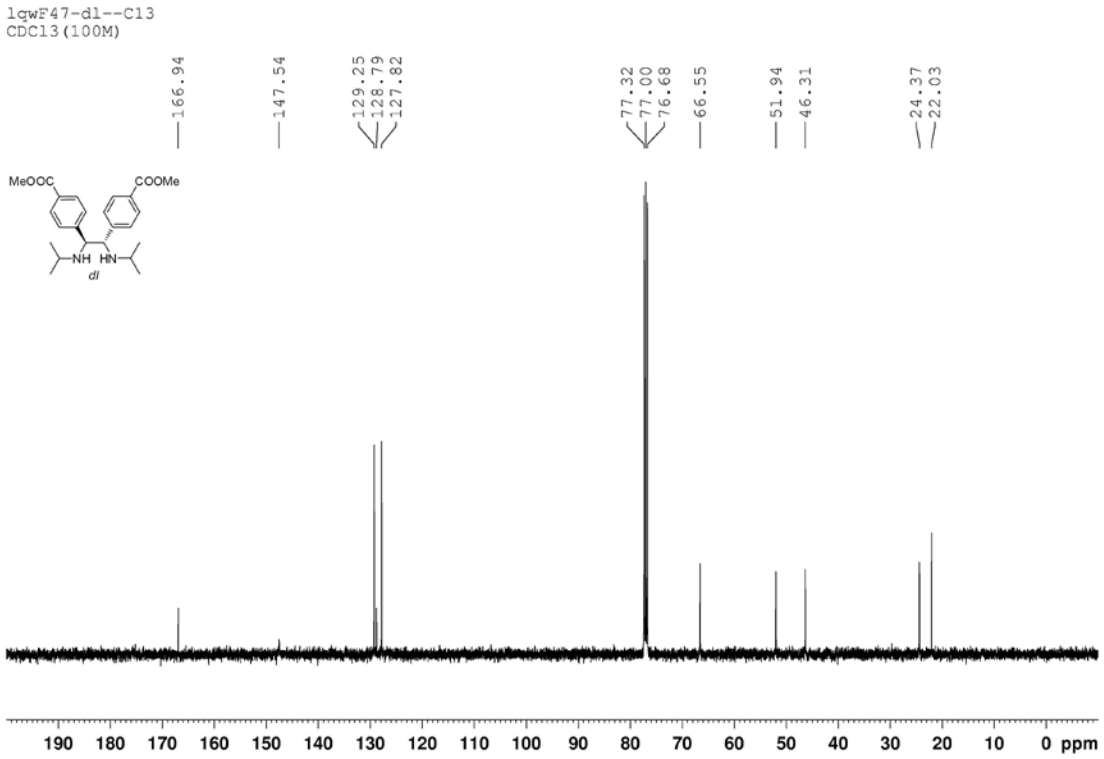
^{1}H and ^{13}C NMR spectra of *meso*-**2h**:



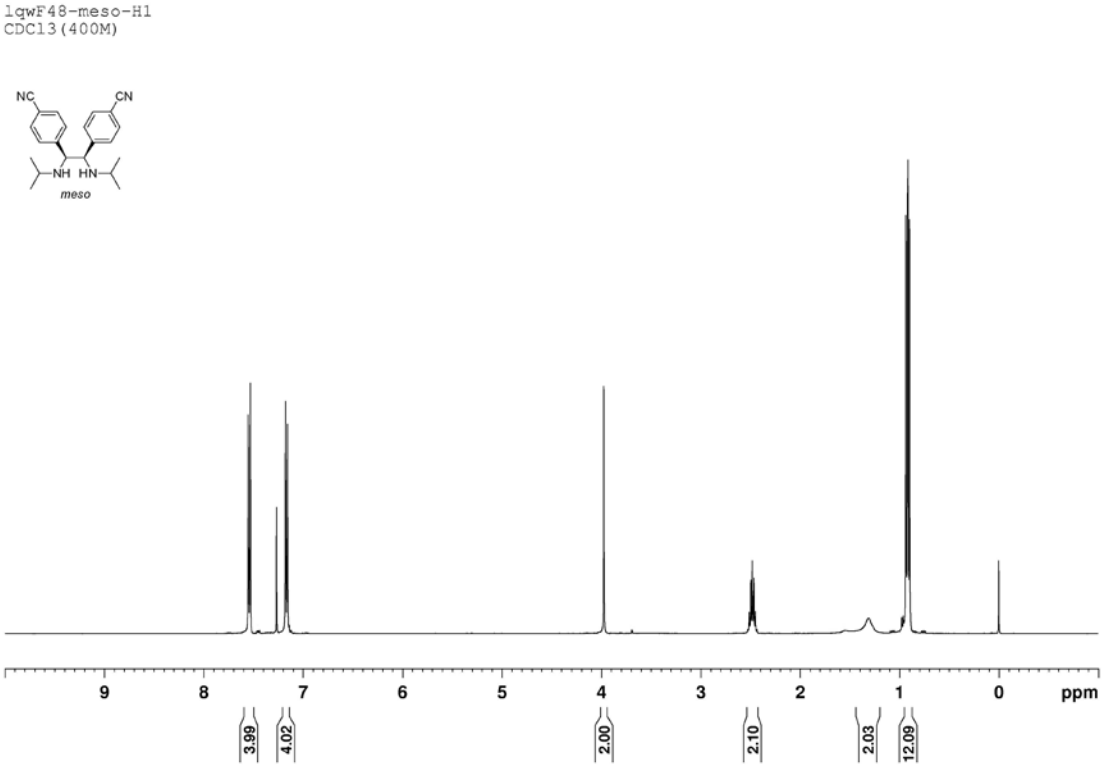


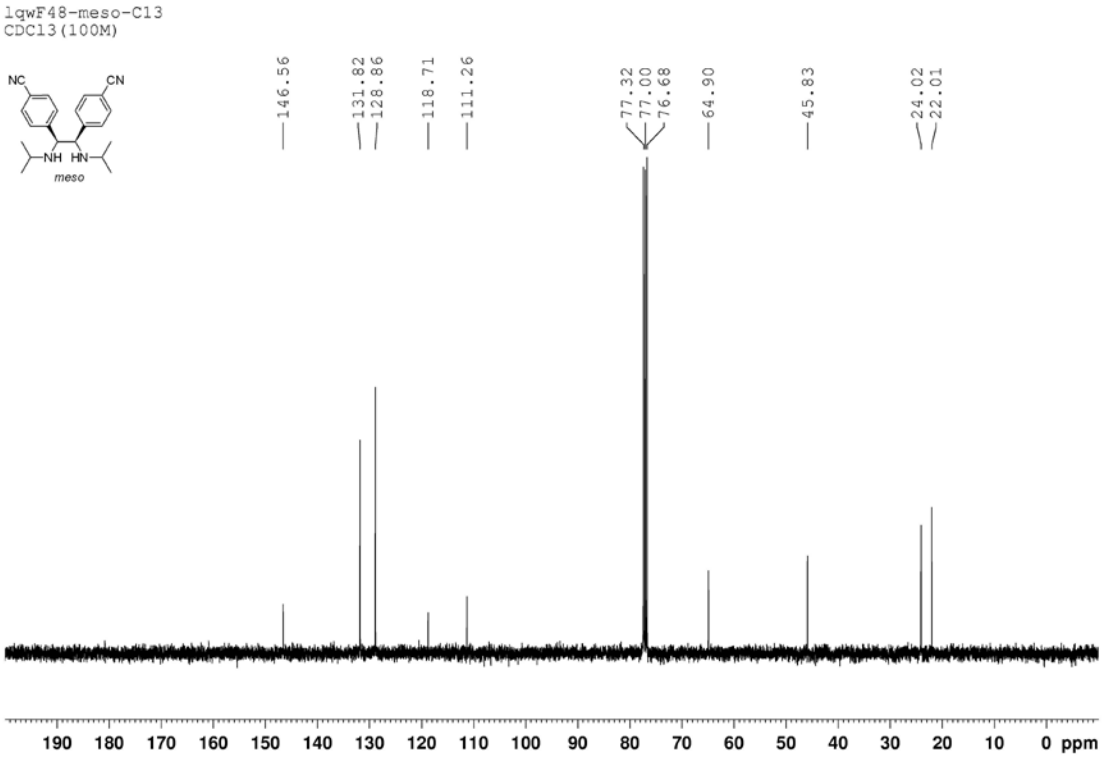
^{1}H and ^{13}C NMR spectra of *dl*-**2h**:



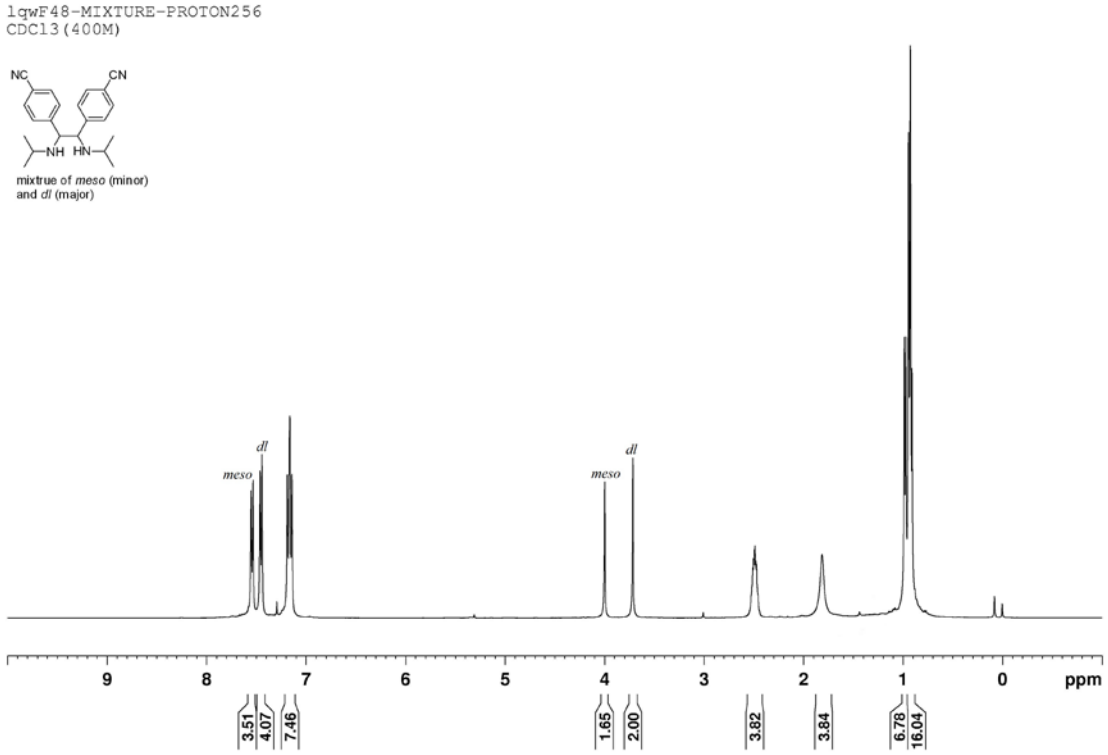


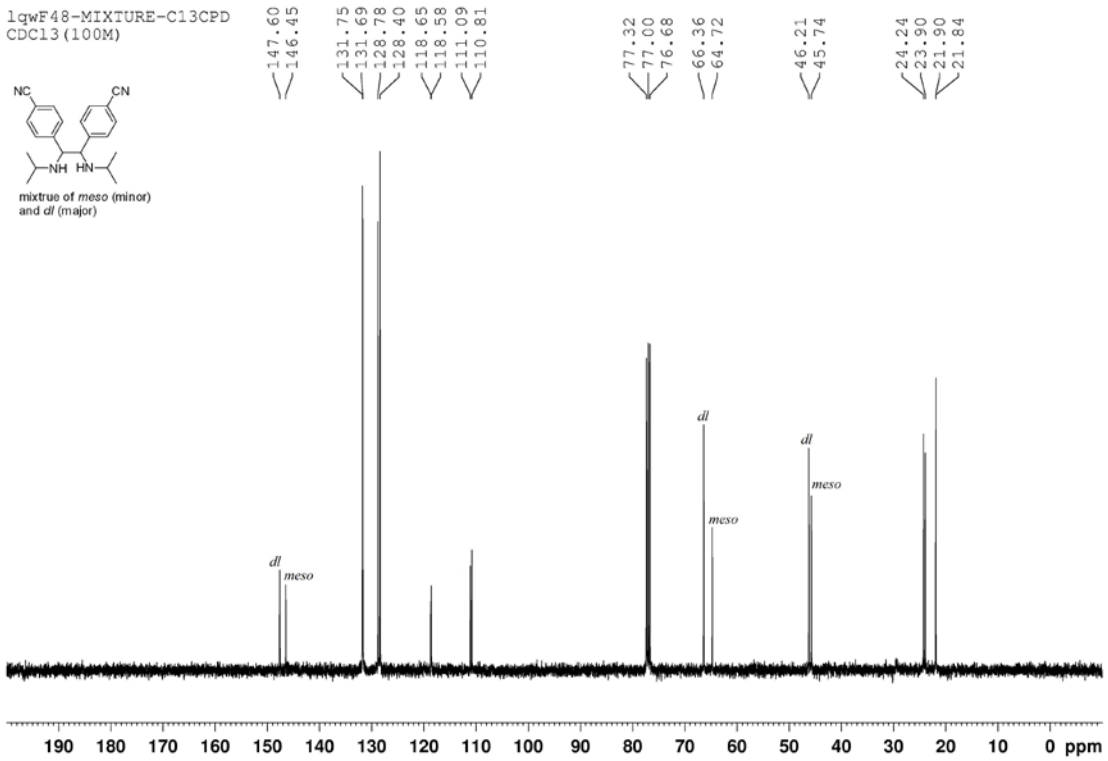
^{1}H and ^{13}C NMR spectra of *meso*-**2i**:



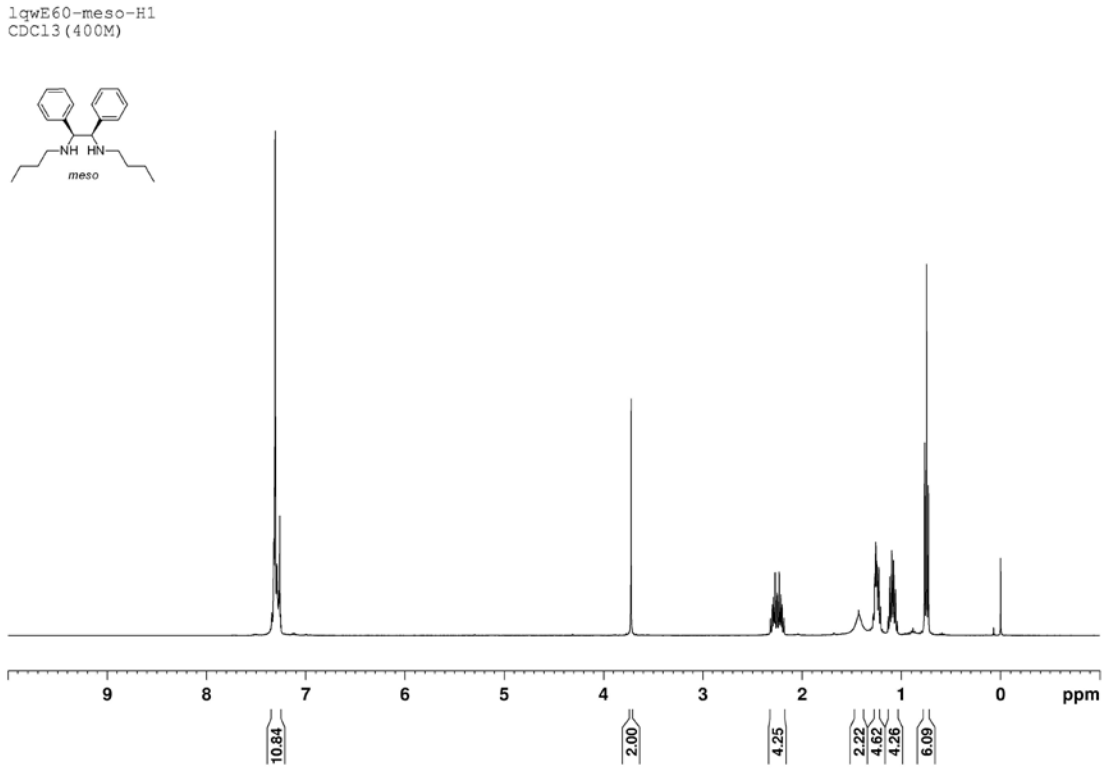


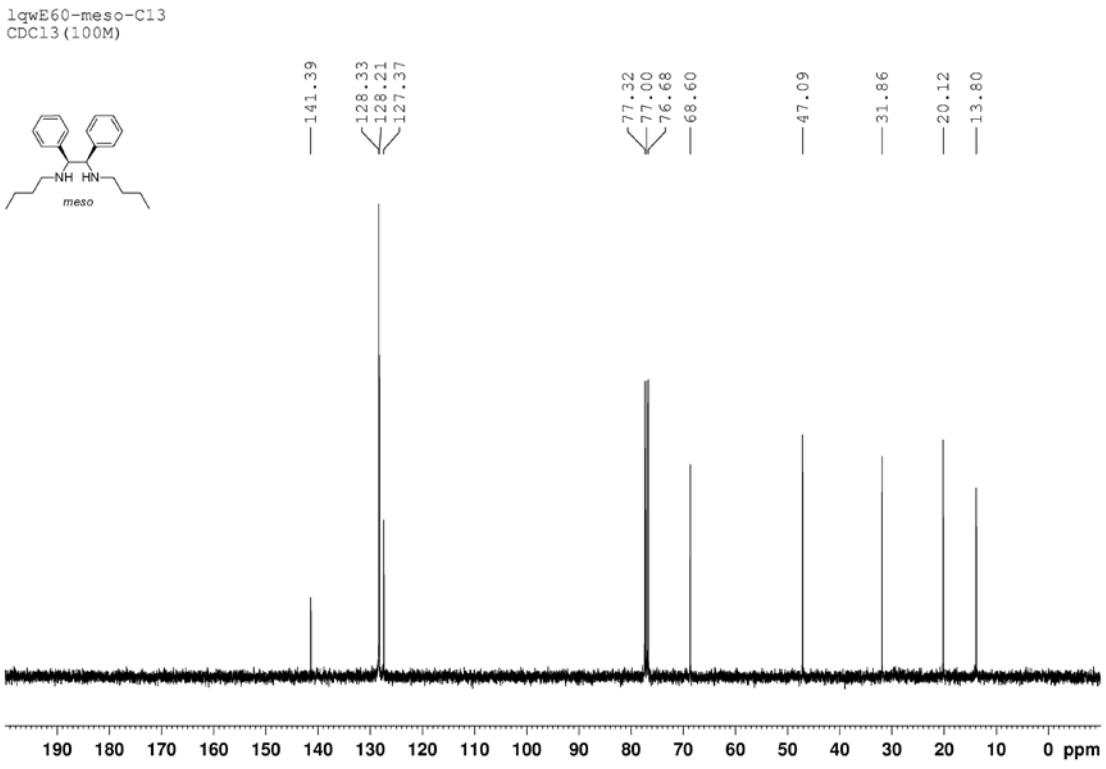
^{1}H and ^{13}C NMR spectra of a fraction of *meso/dl* mixture of compound 2i



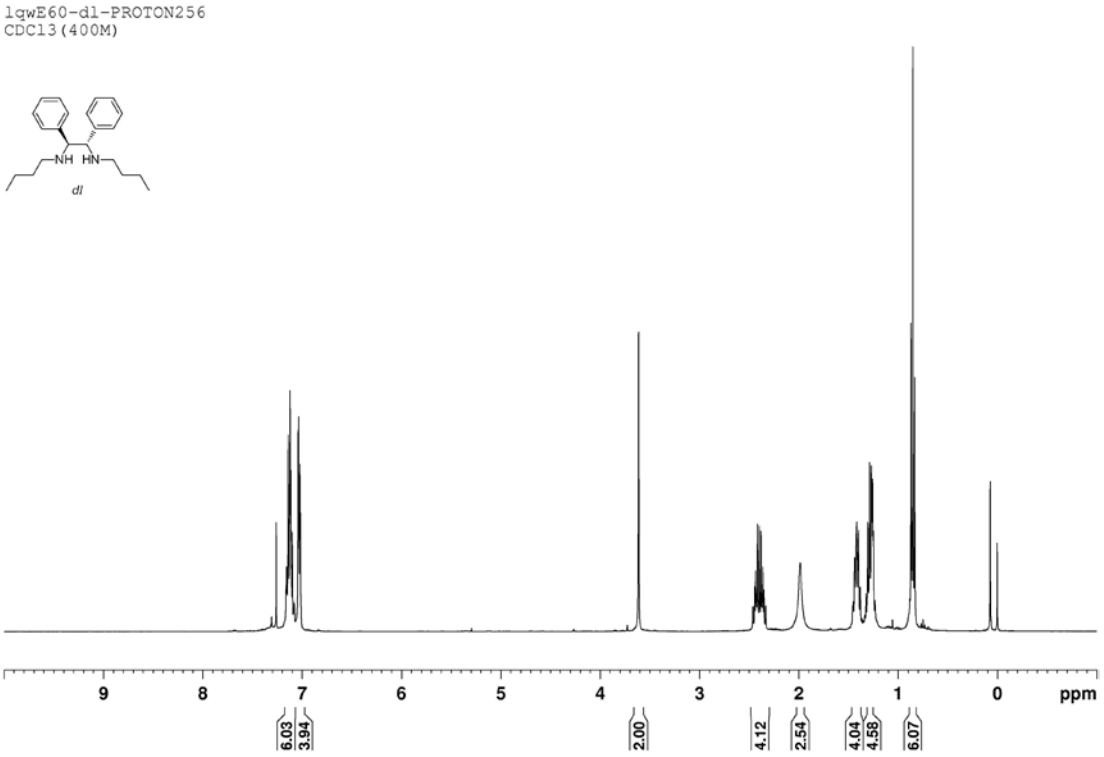


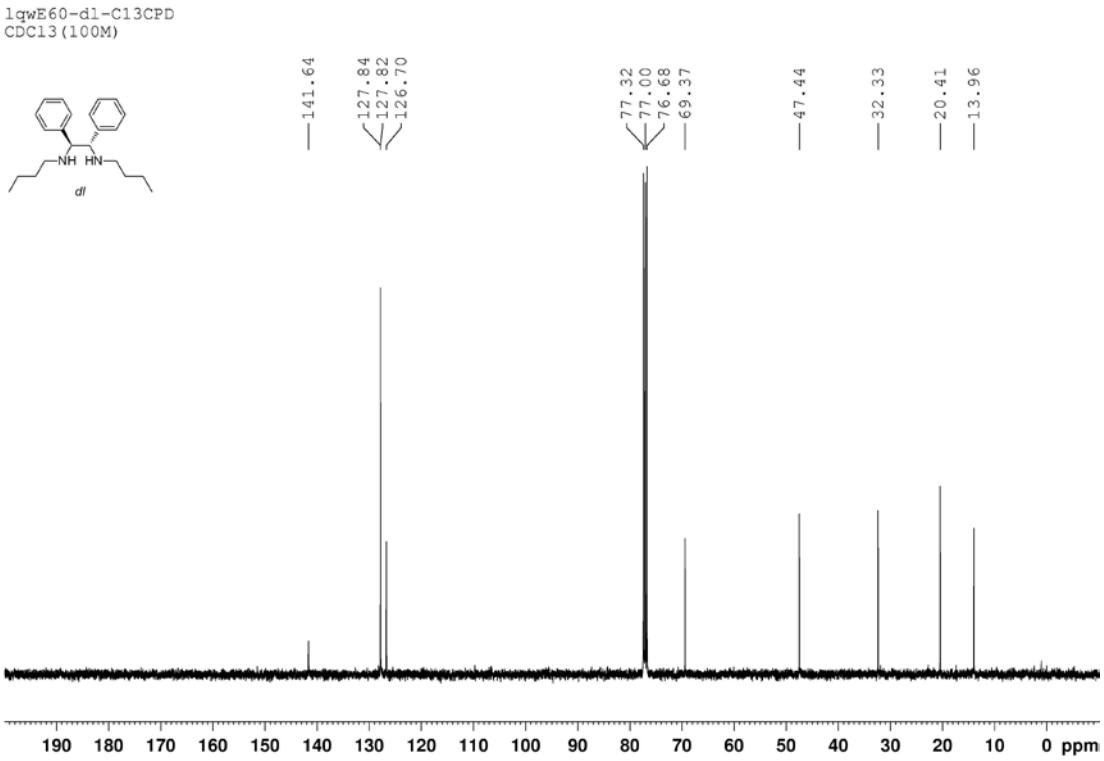
^{1}H and ^{13}C NMR spectra of *meso*-**2j**:



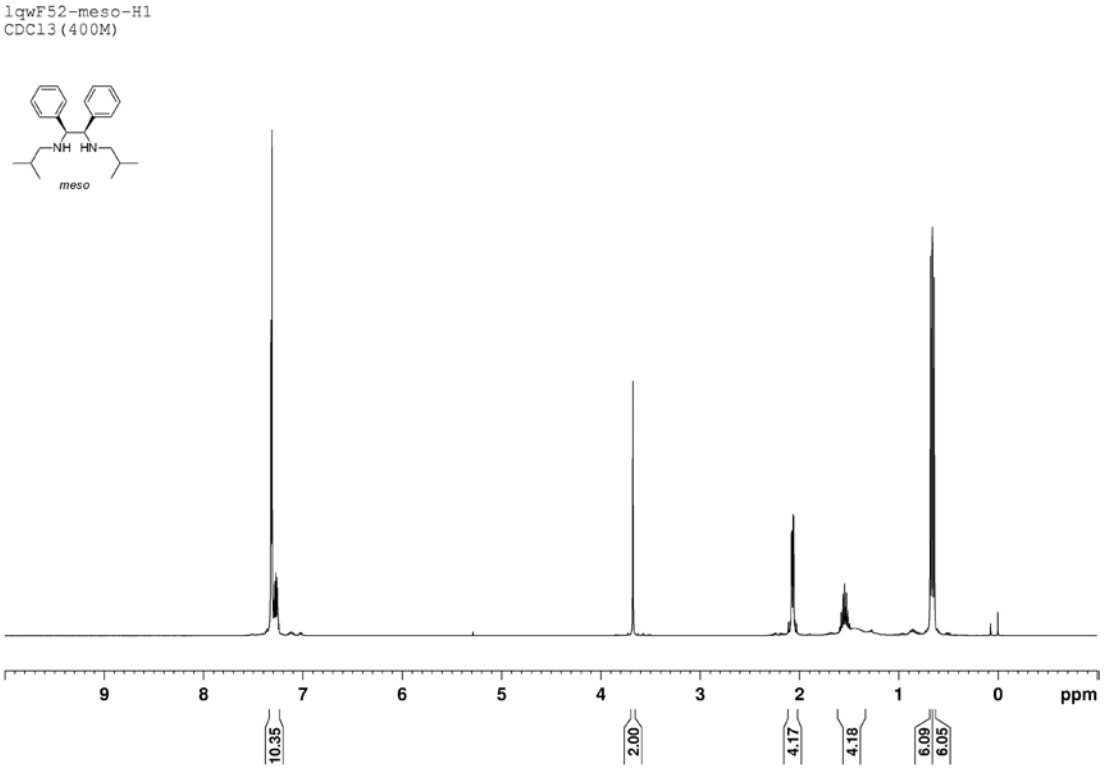


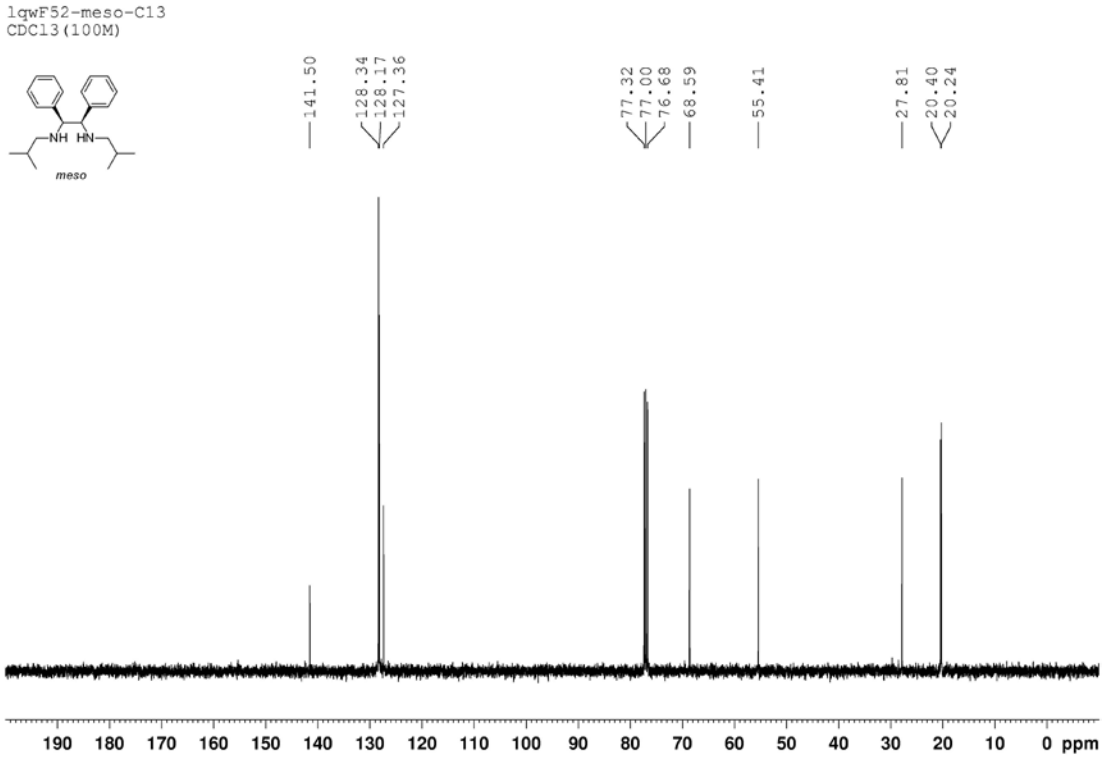
^{1}H and ^{13}C NMR spectra of *dl* -**2j**:



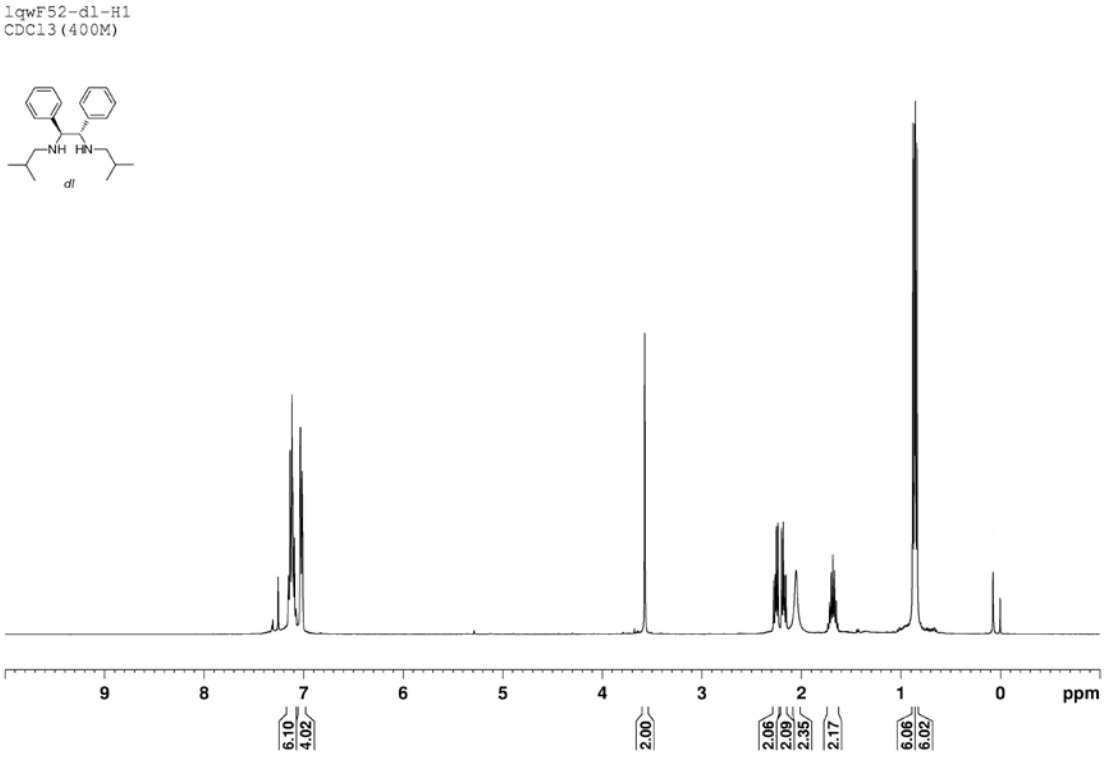


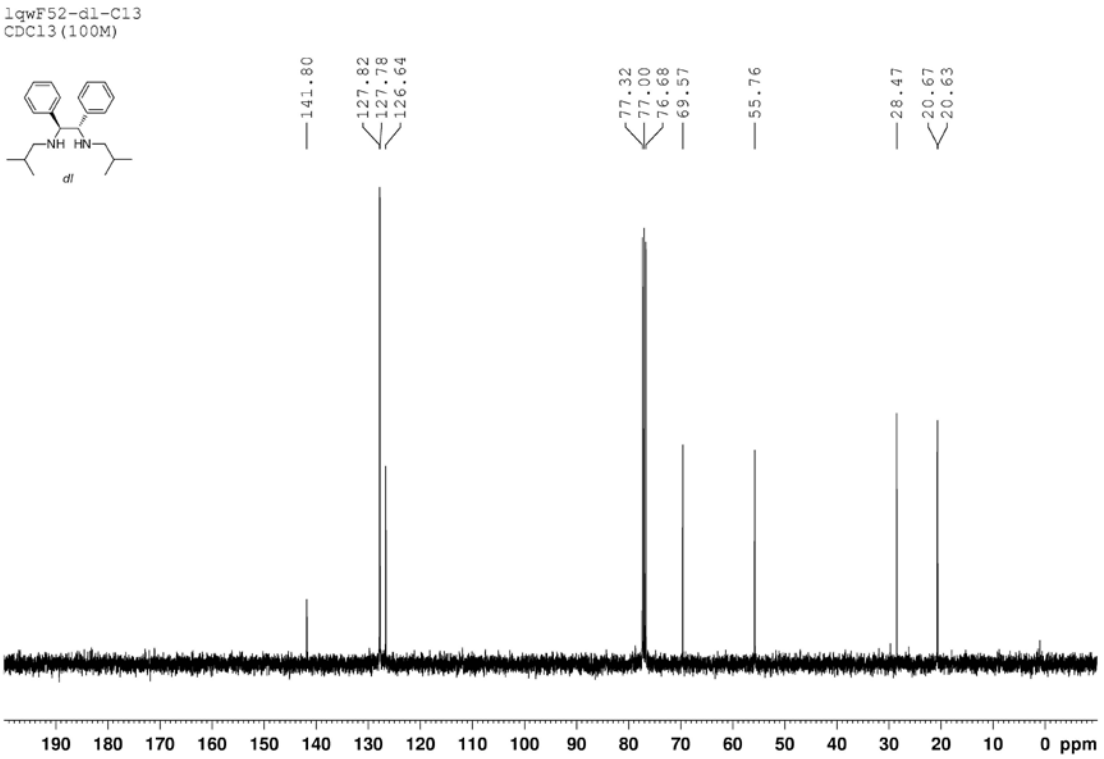
^{1}H and ^{13}C NMR spectra of *meso*-**2k**:



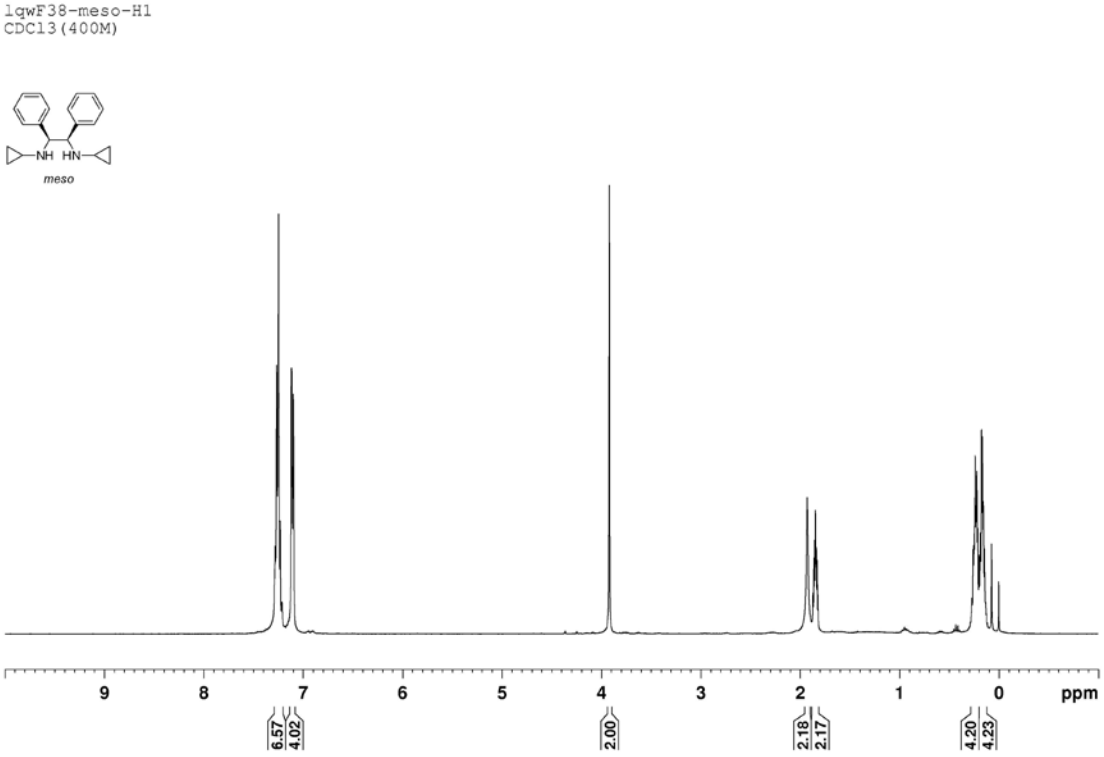


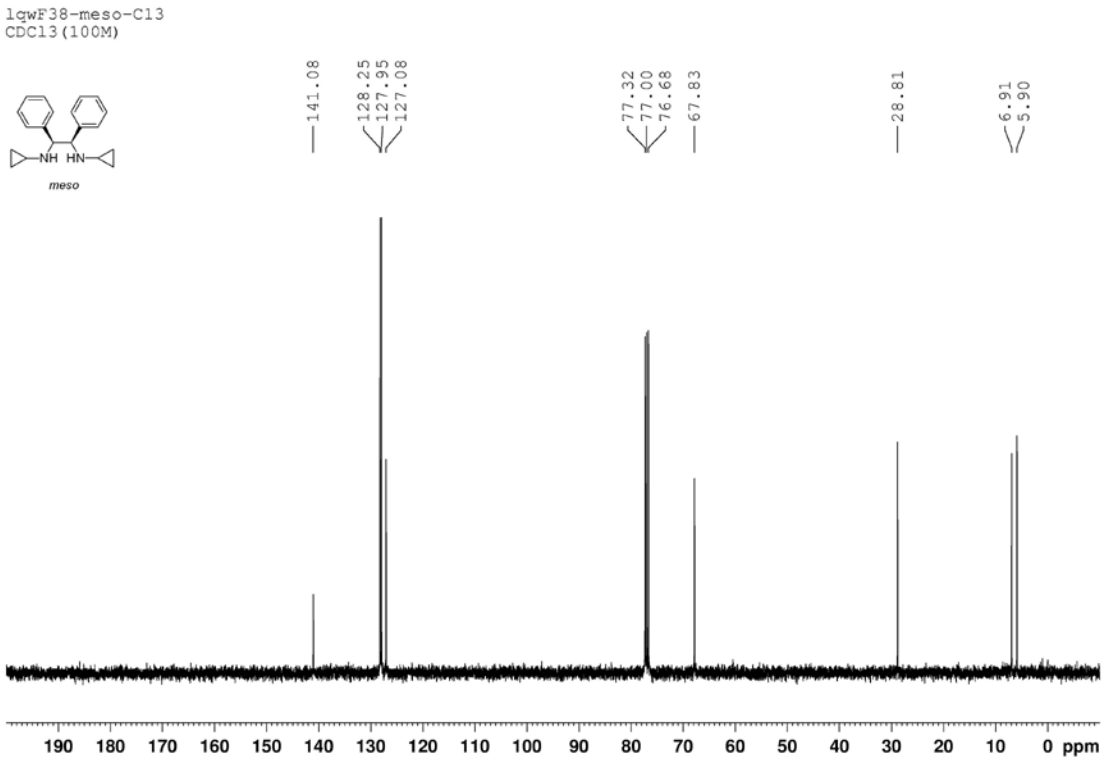
^{1}H and ^{13}C NMR spectra of *dl*-**2k**:



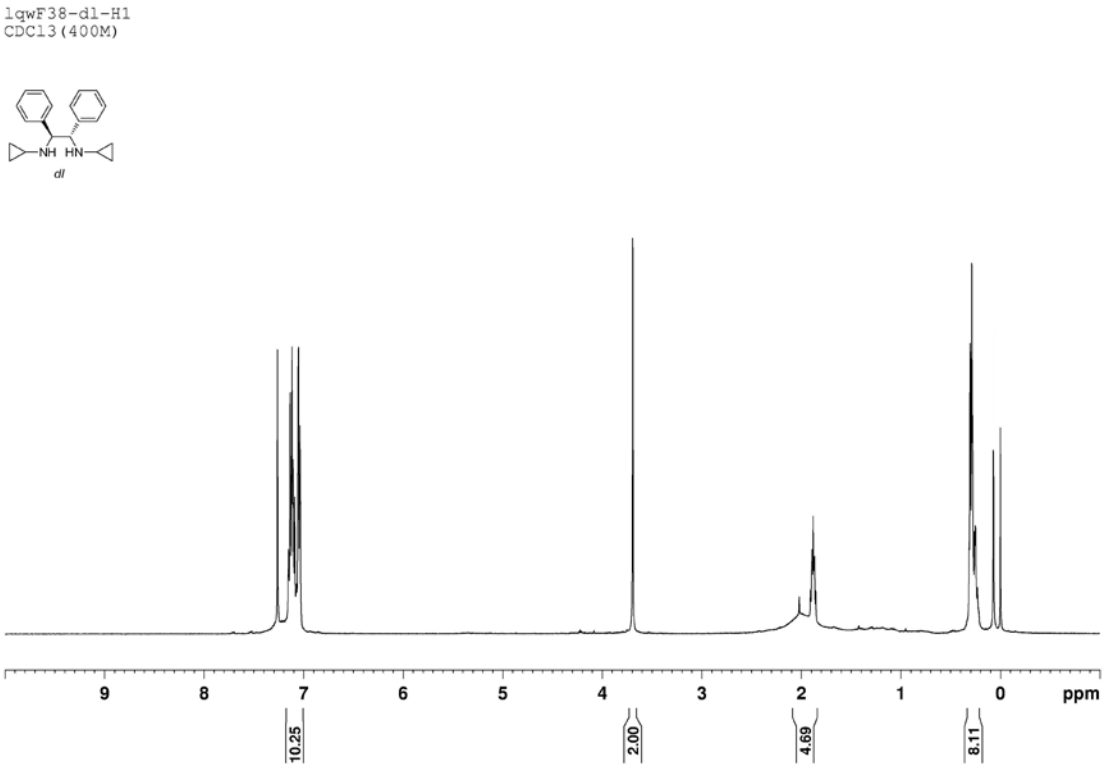


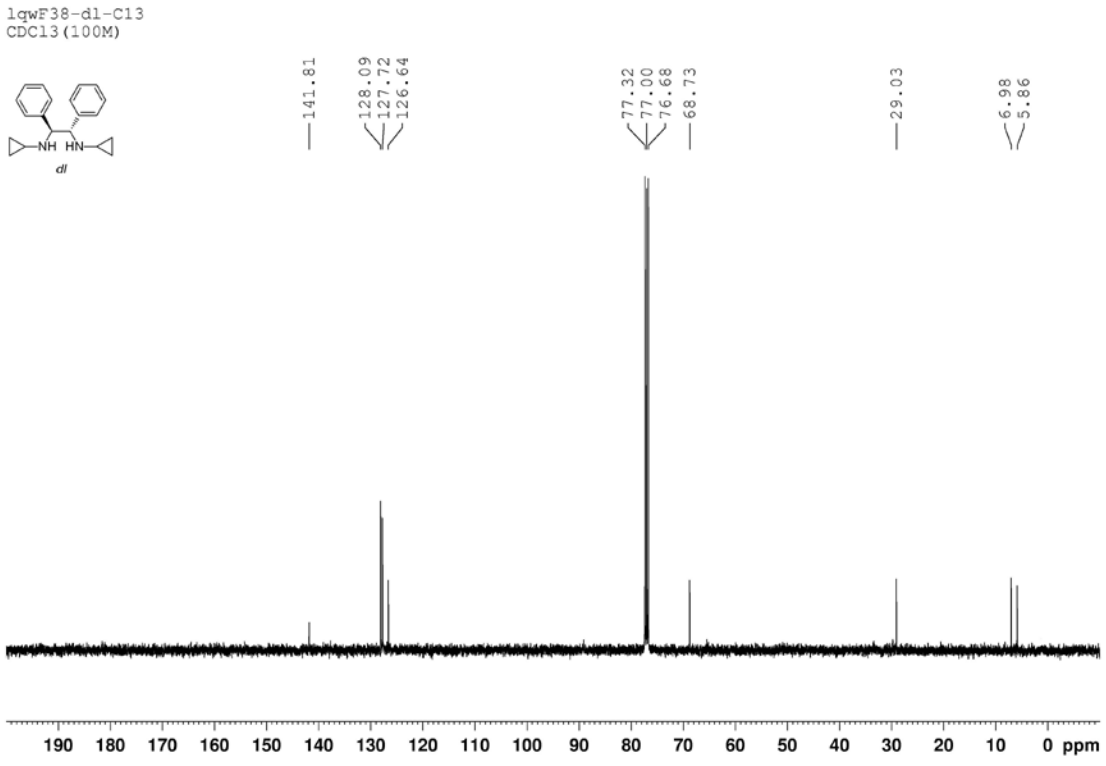
^{1}H and ^{13}C NMR spectra of *meso*-**2l**:



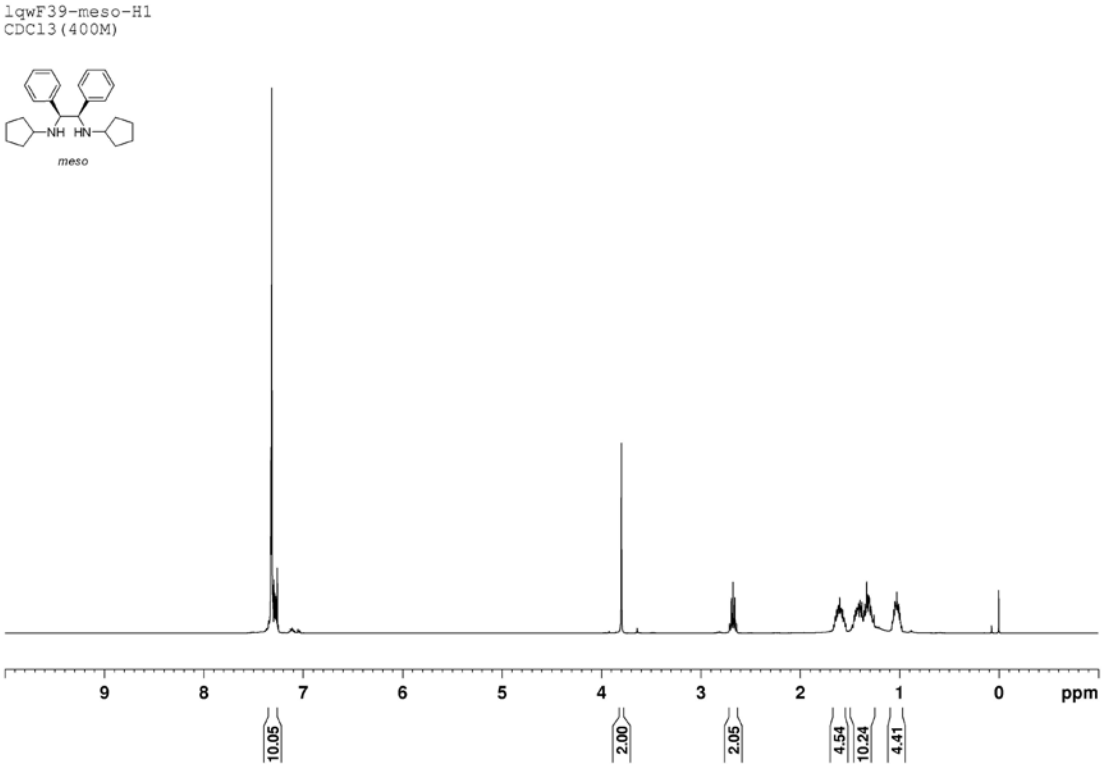


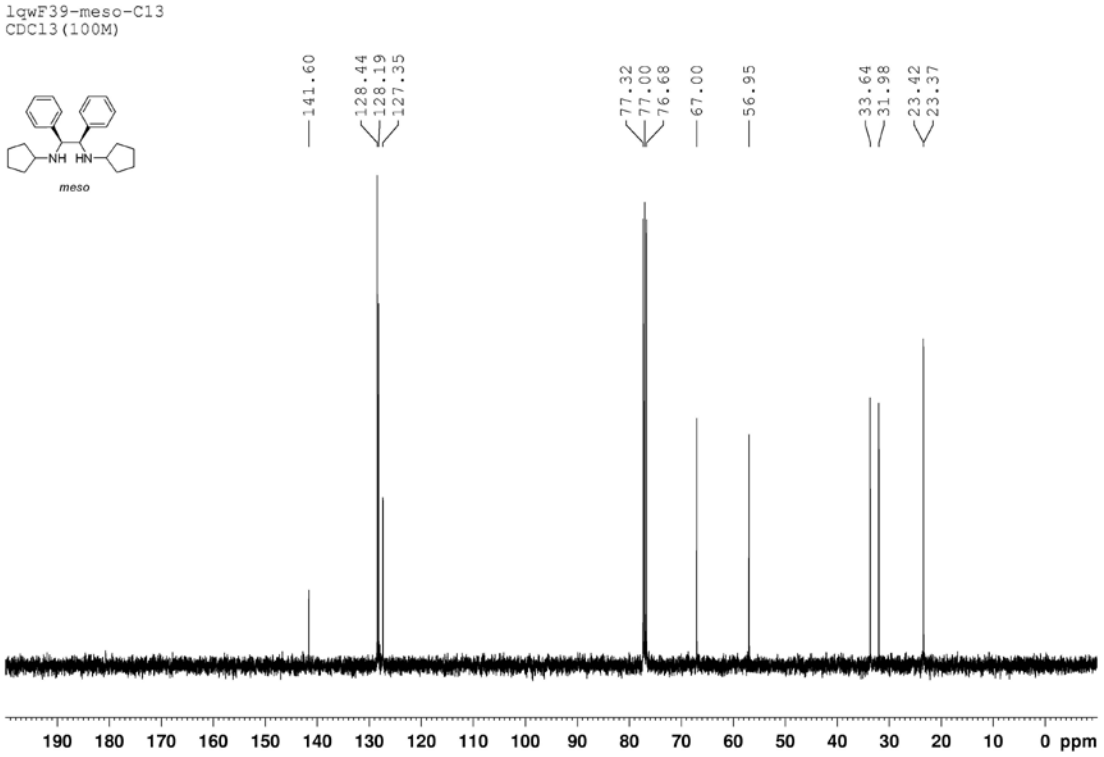
^{1}H and ^{13}C NMR spectra of *dl*-**2l**:



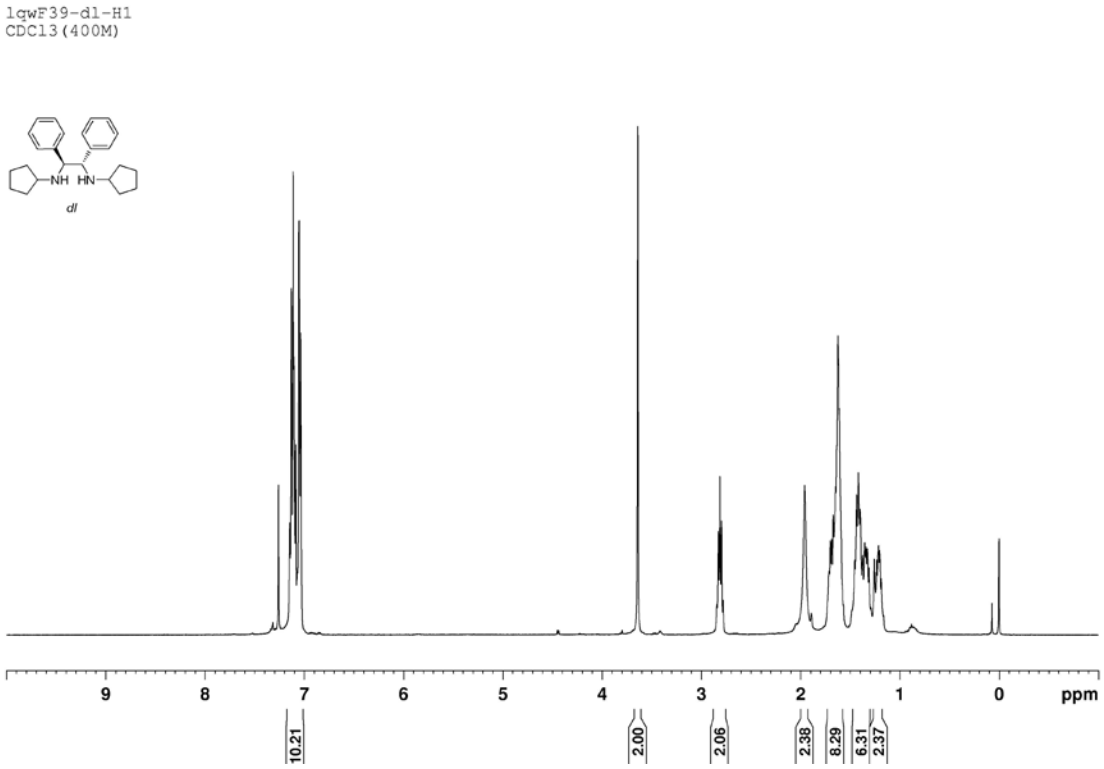


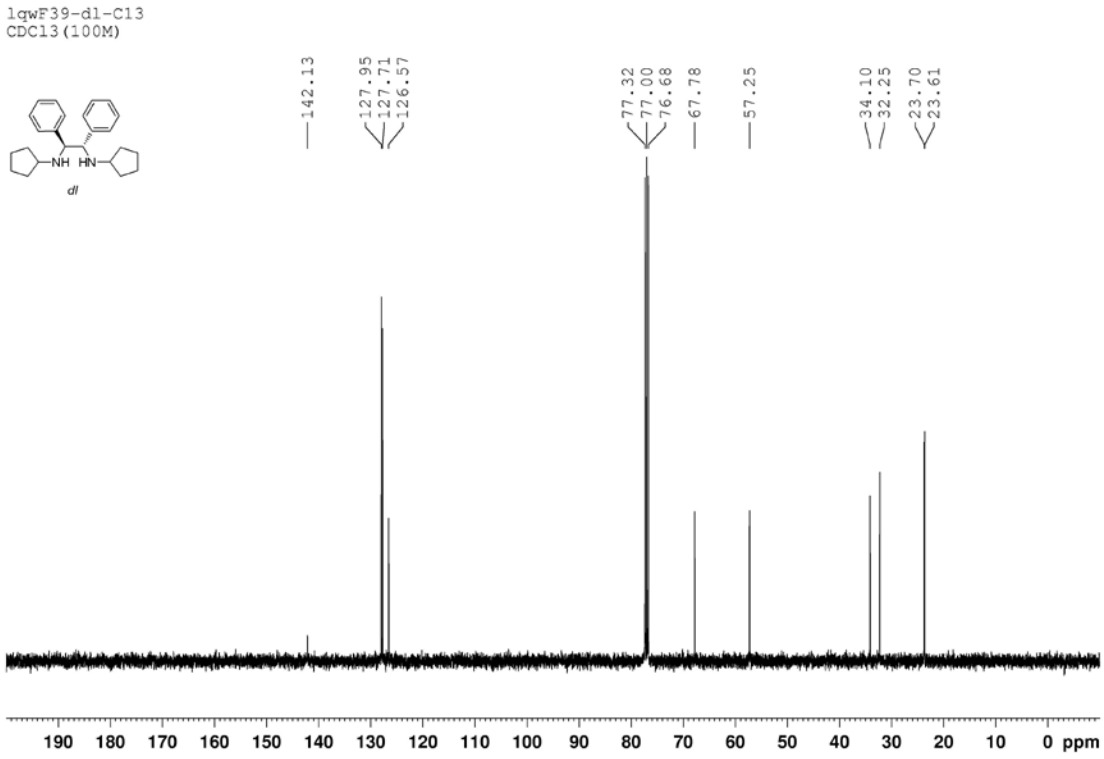
^{1}H and ^{13}C NMR spectra of *meso*-**2m**:



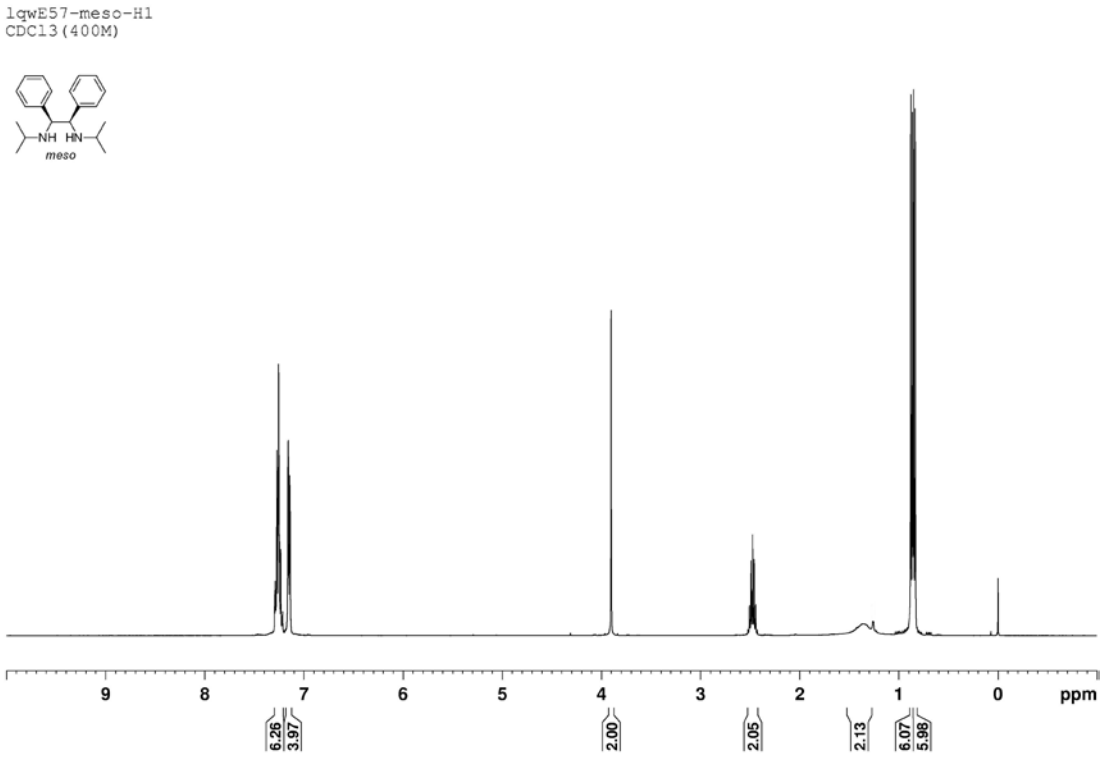


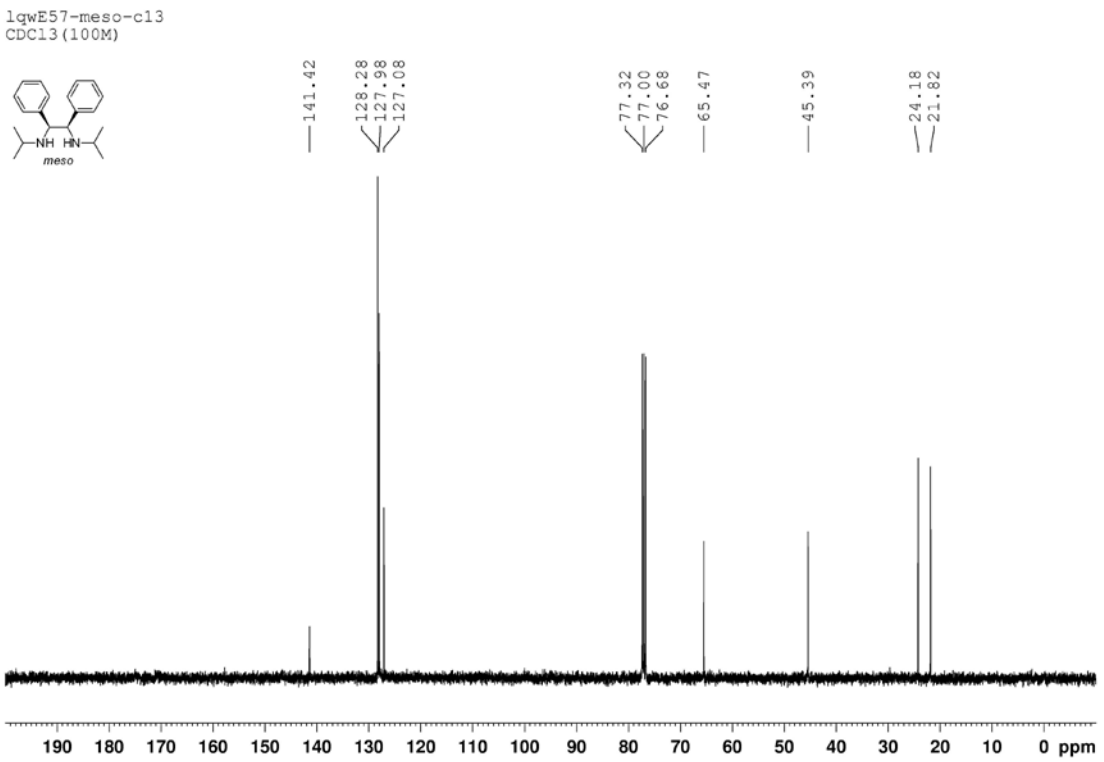
^{1}H and ^{13}C NMR spectra of *dl*-**2m**:



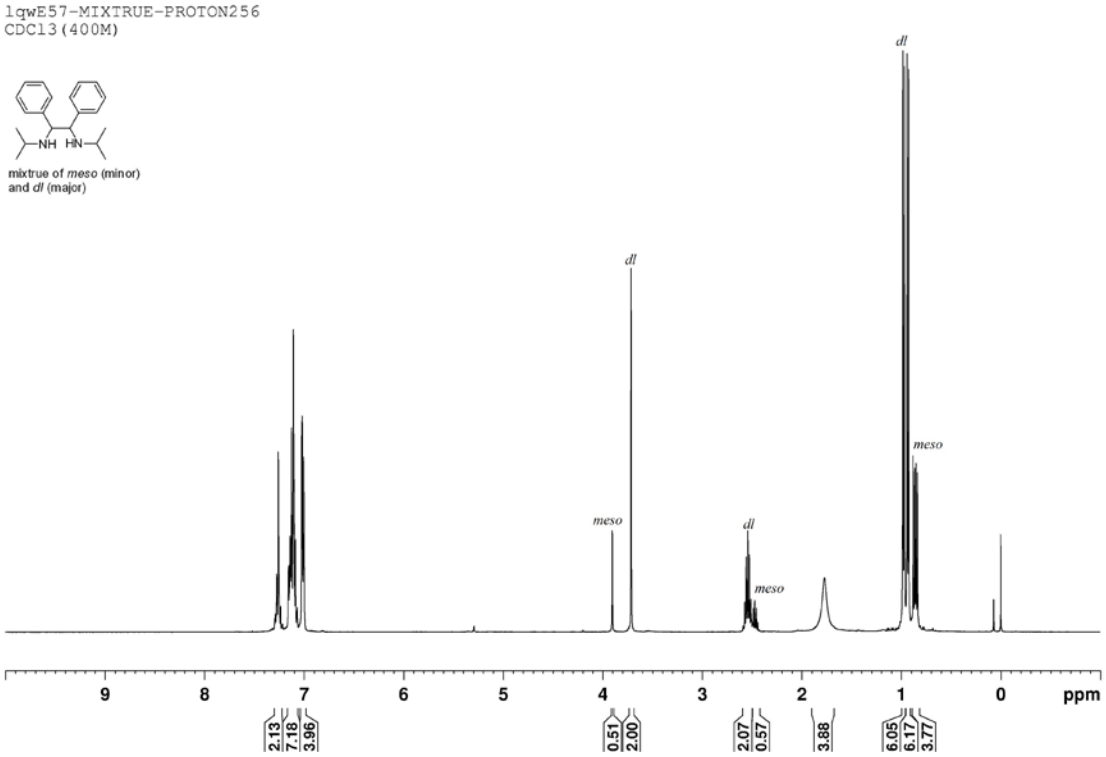


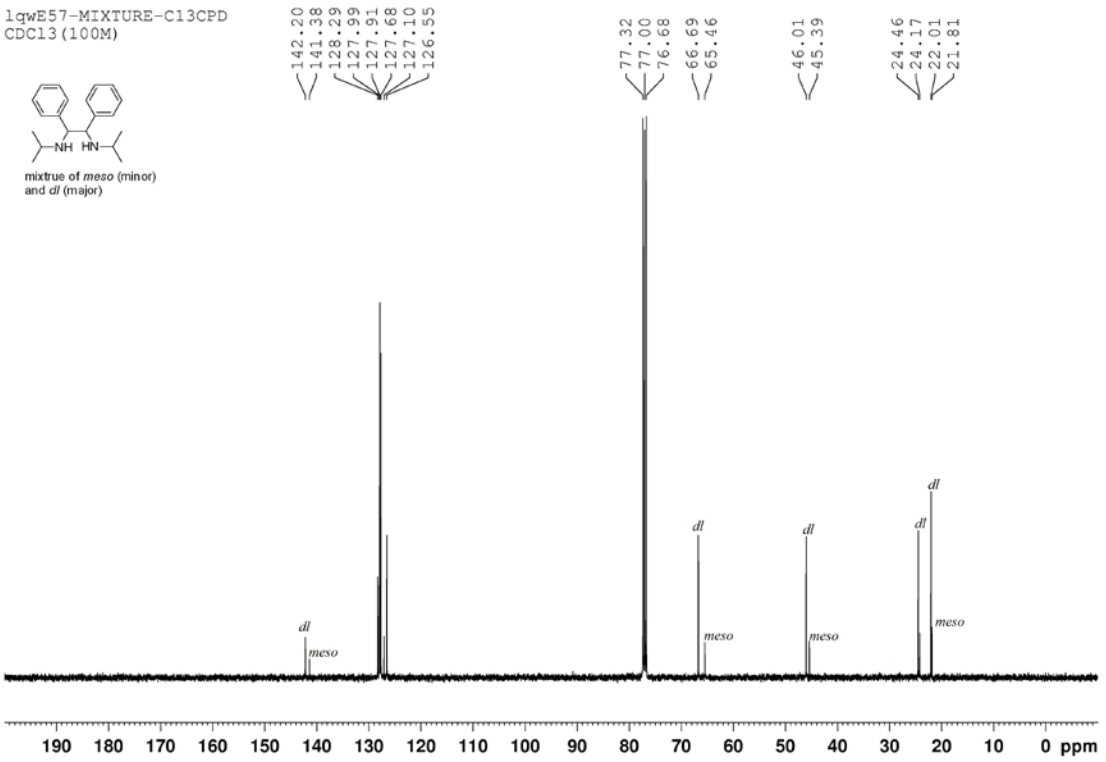
^{1}H and ^{13}C NMR spectra of *meso*-**2n**:



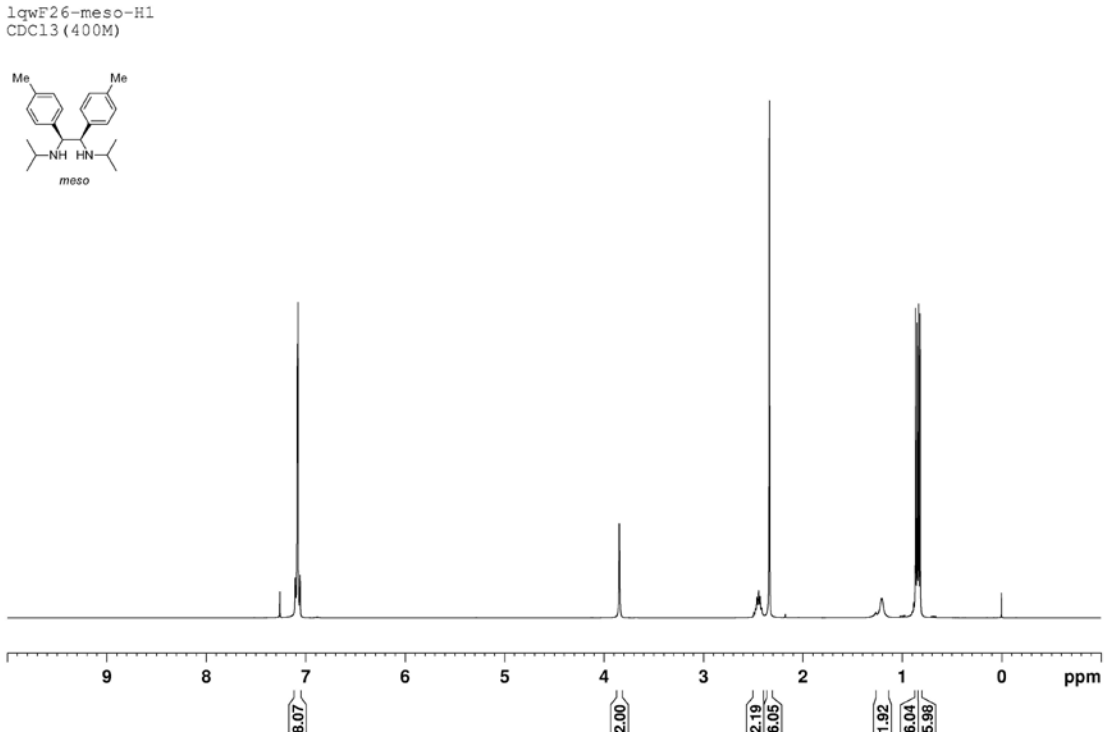


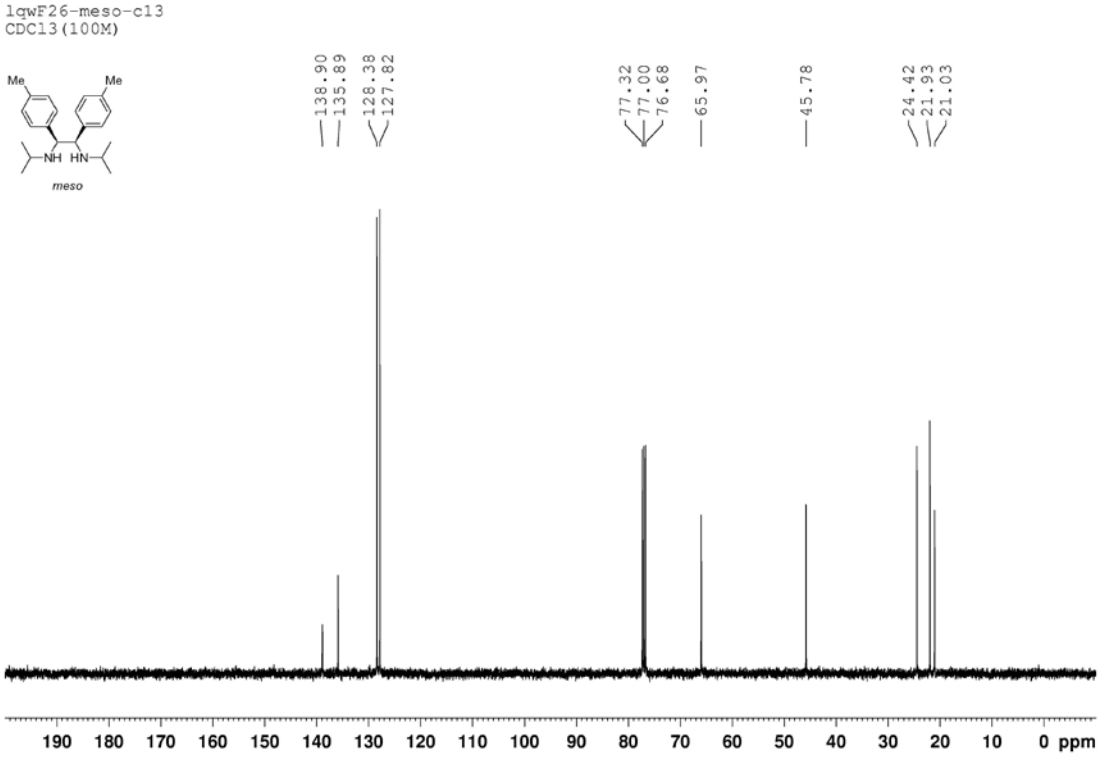
1H and 13C NMR spectra of a fraction of meso/dl mixture of **2n**:



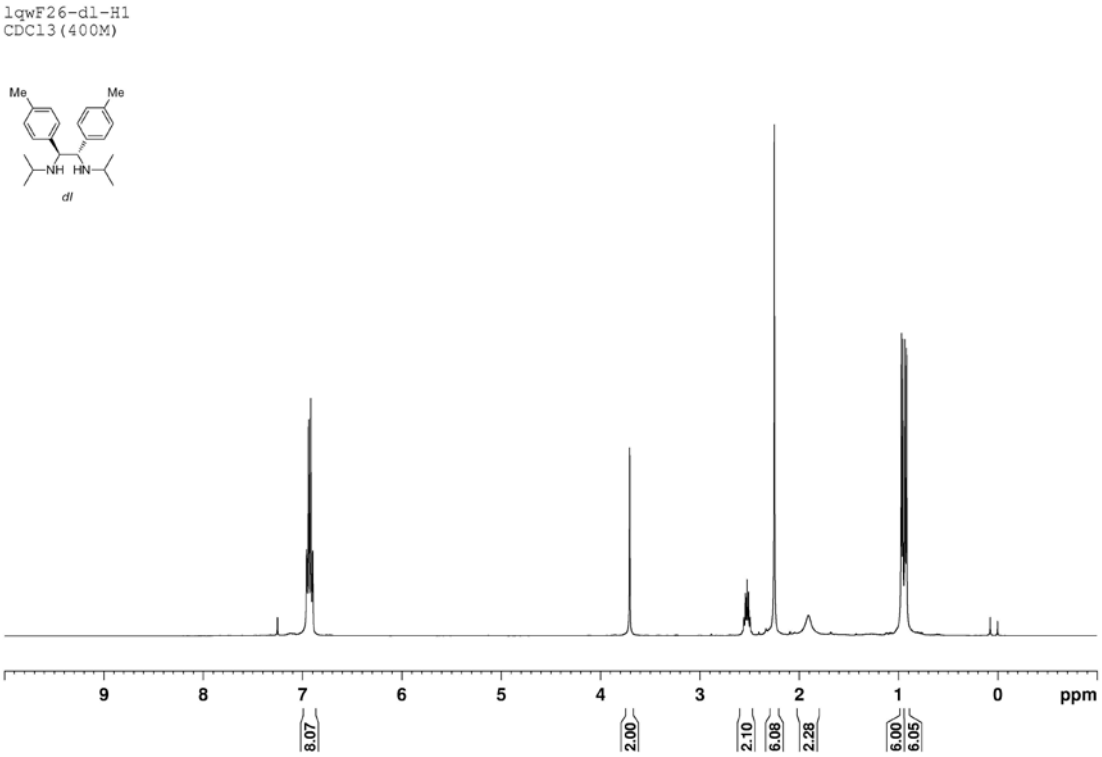


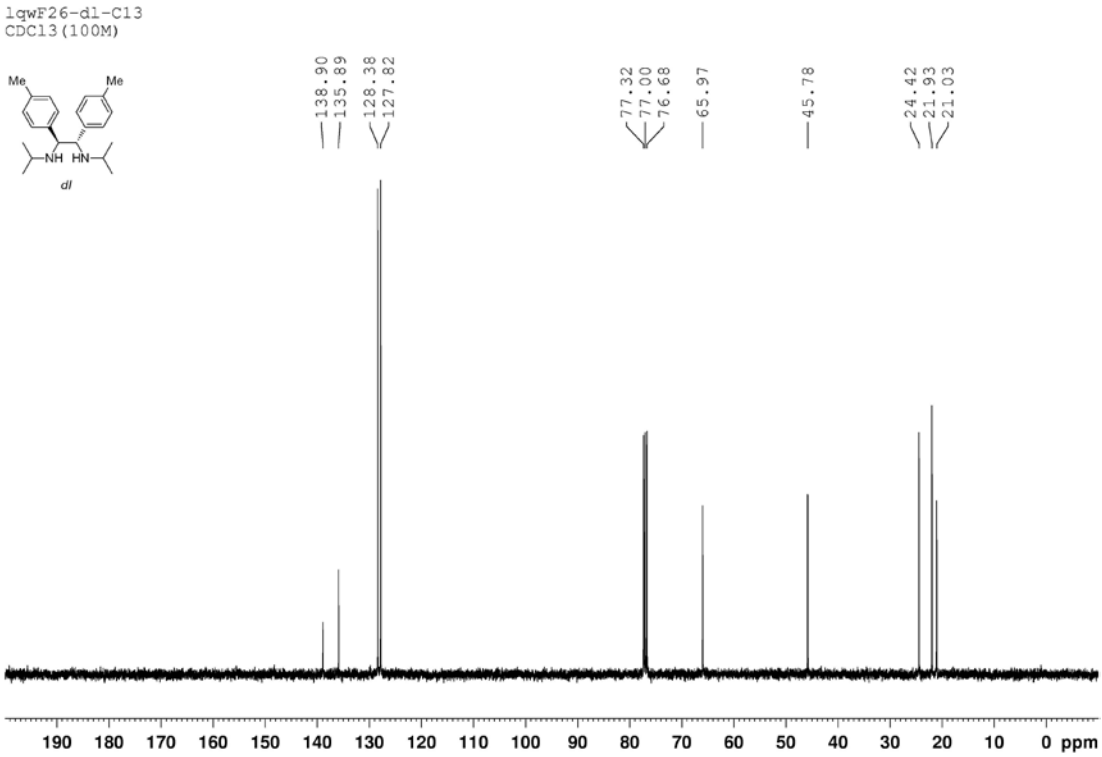
^{1}H and ^{13}C NMR spectra of *meso*-**2o**:



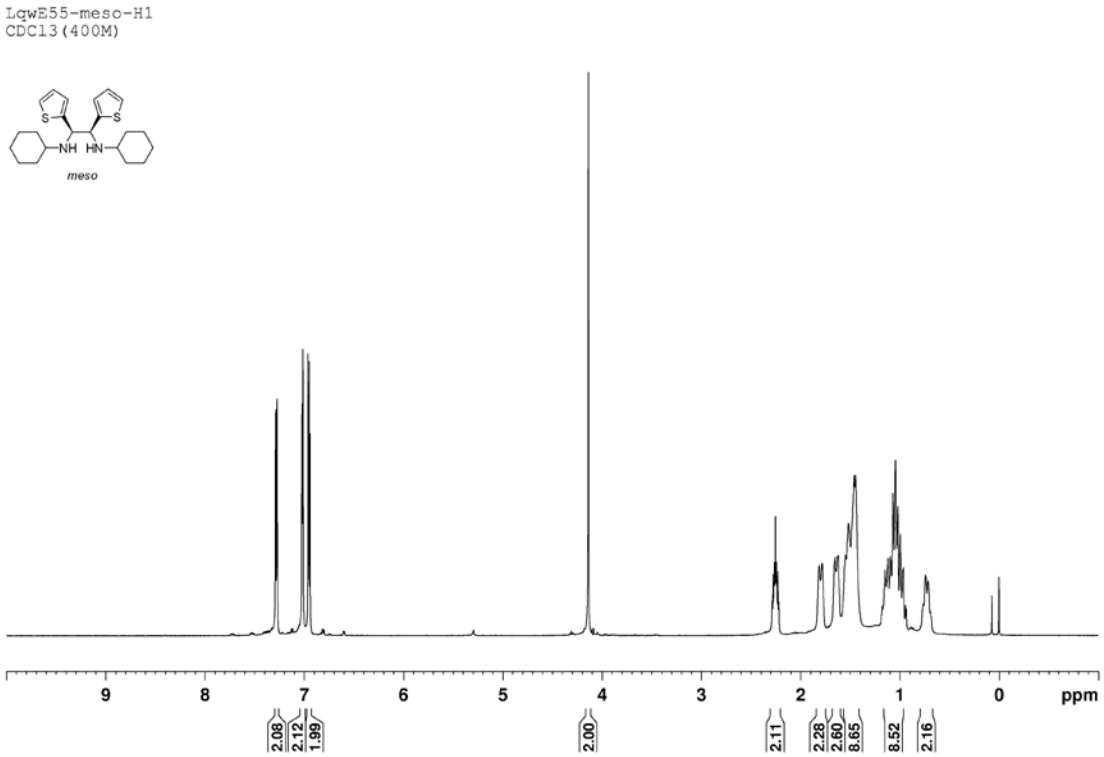


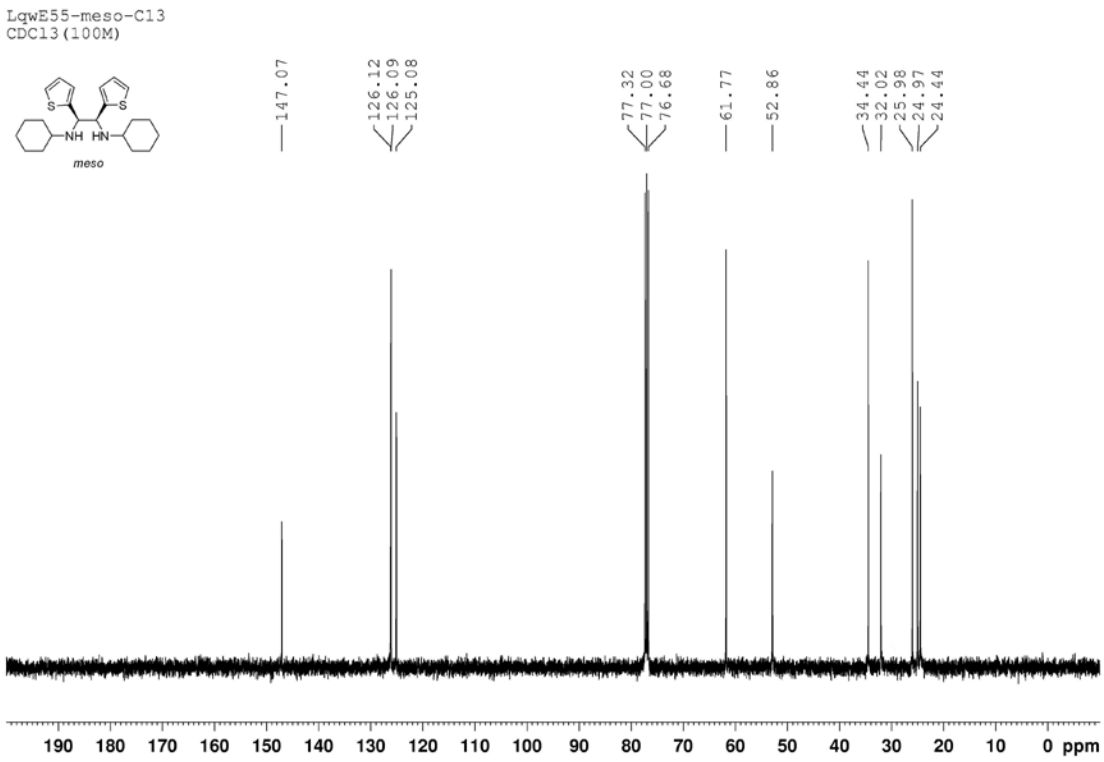
^{1}H and ^{13}C NMR spectra of *dl*-**2o**:



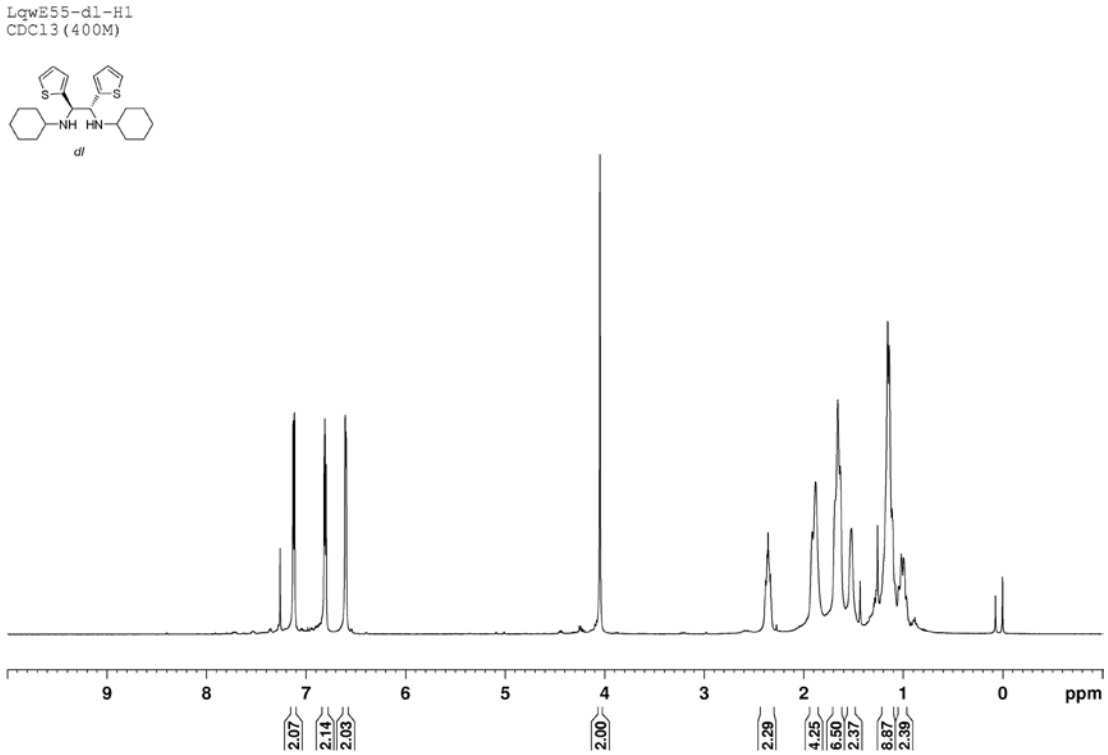


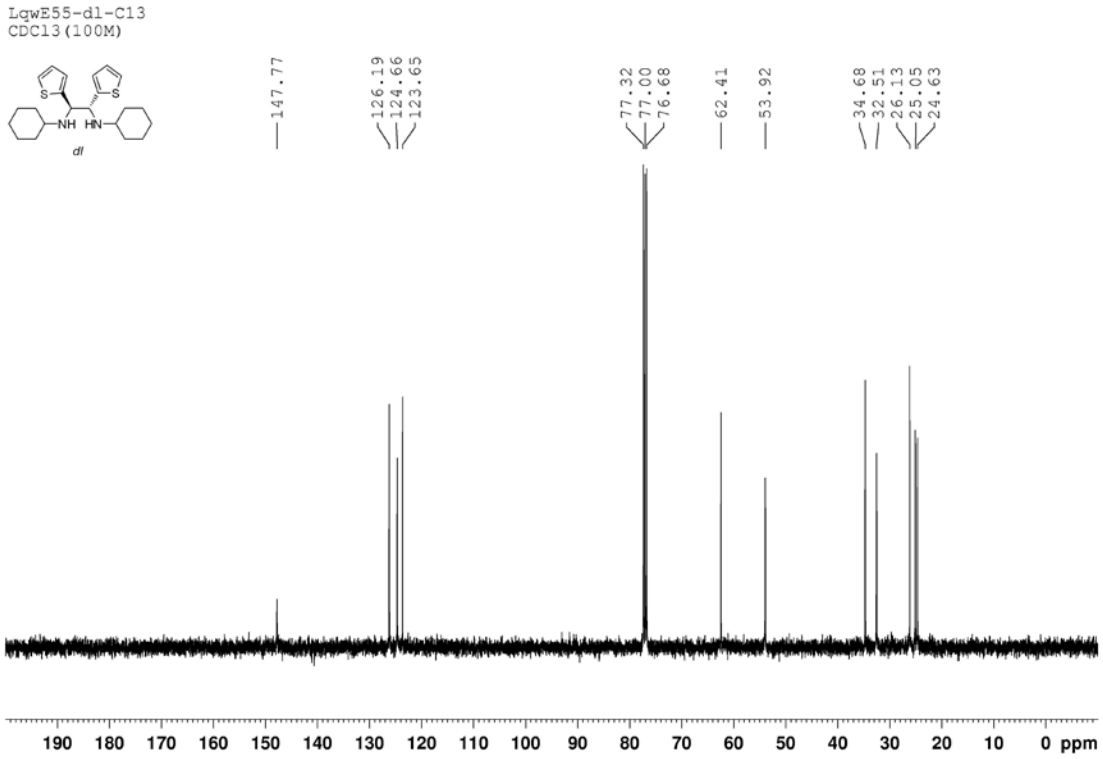
^{1}H and ^{13}C NMR spectra of *meso*-**2r**:



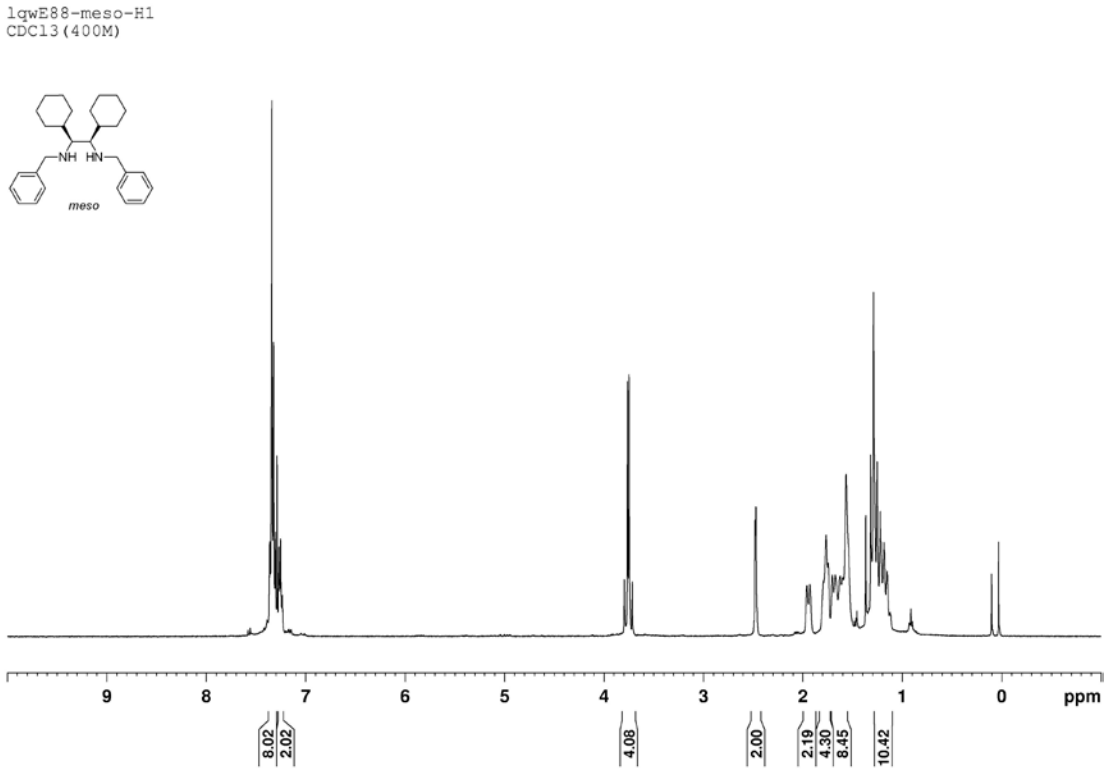


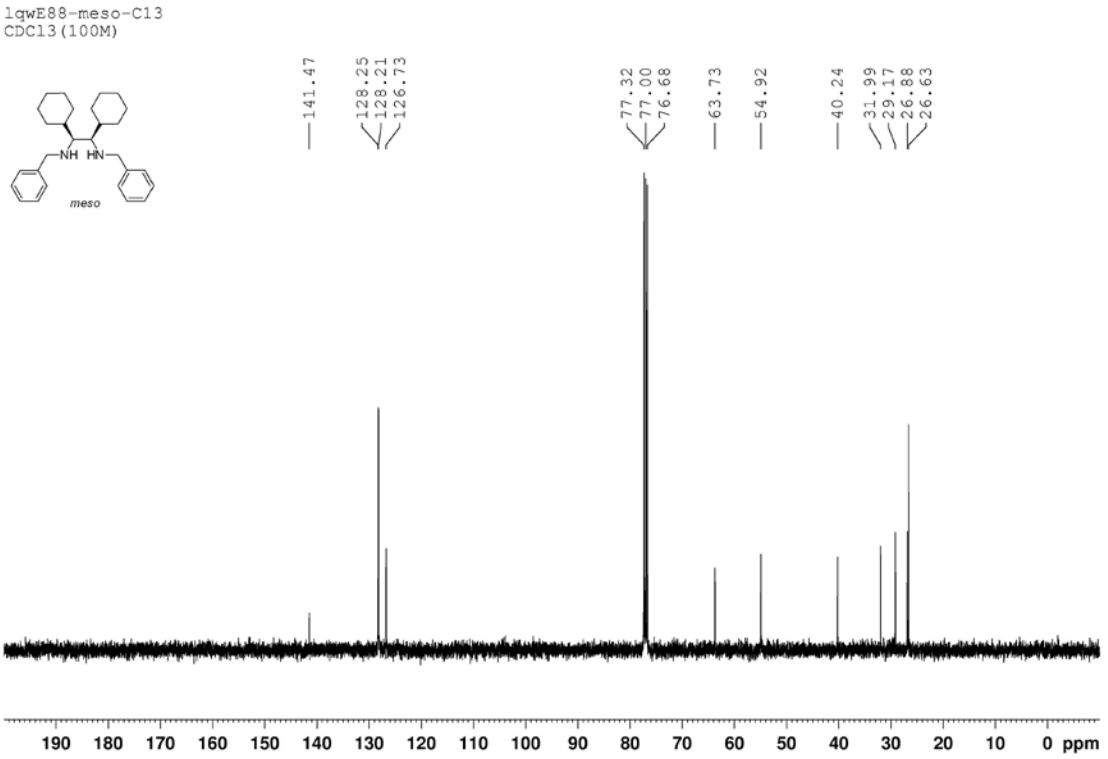
^{1}H and ^{13}C NMR spectra of *dl*-**2r**:



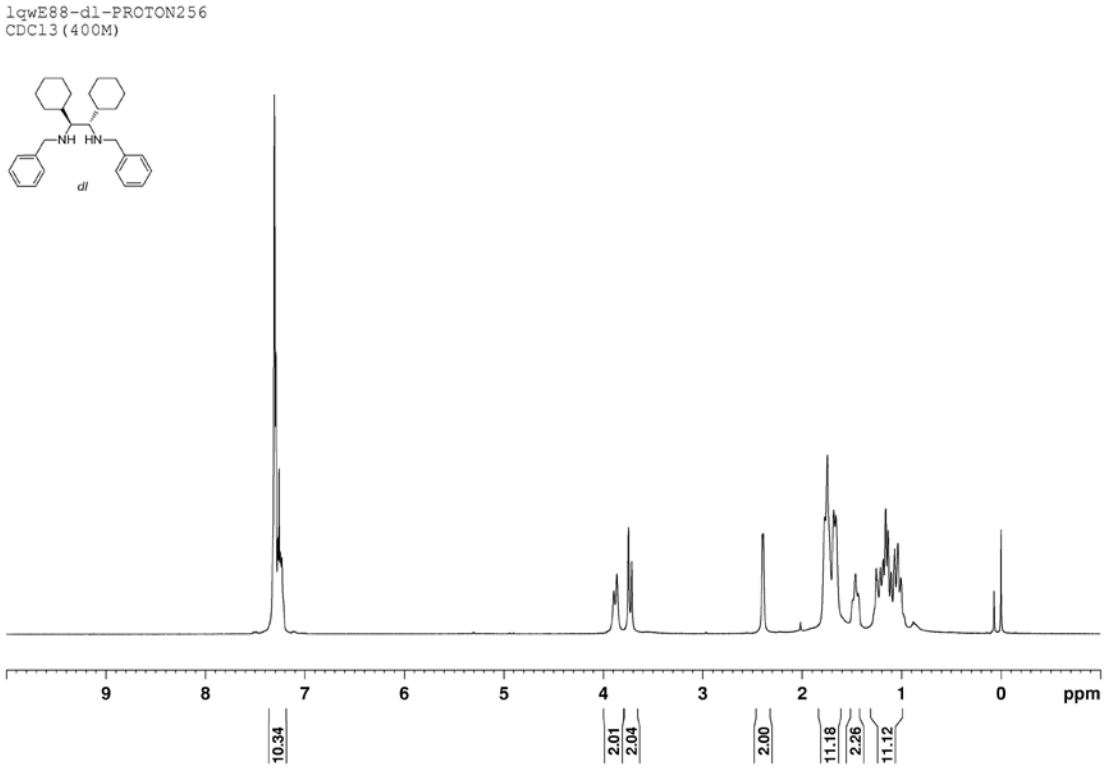


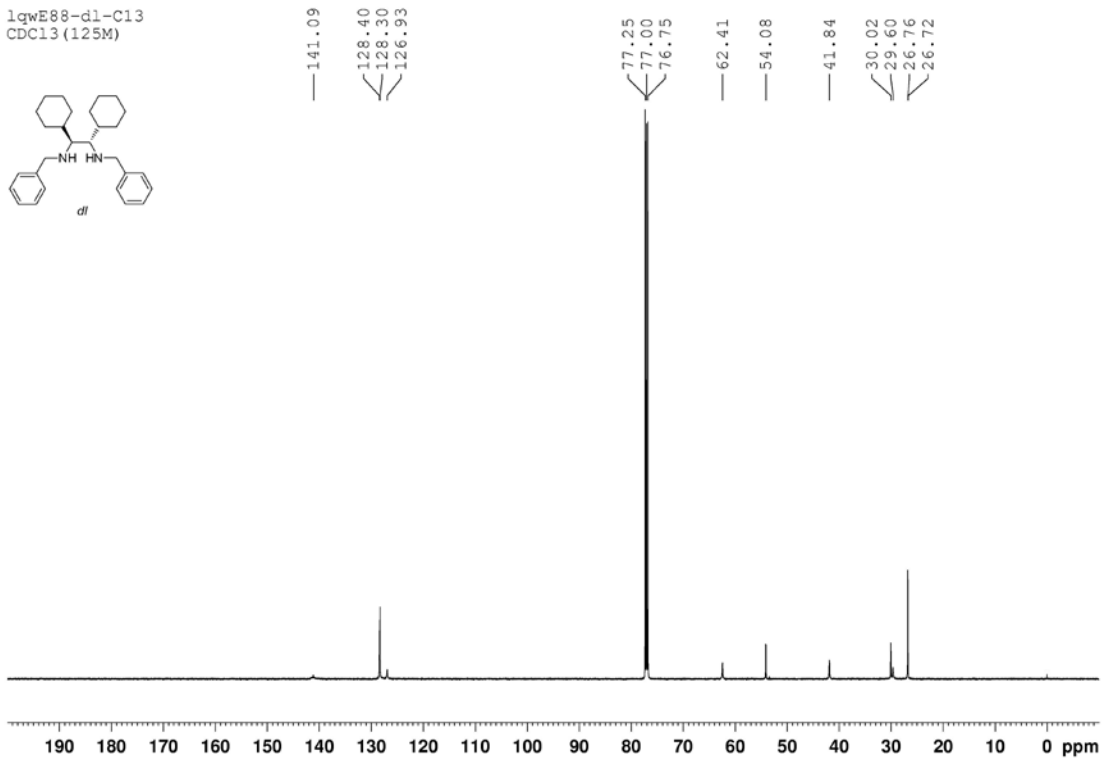
^{1}H and ^{13}C NMR spectra of *meso*-**2s**:



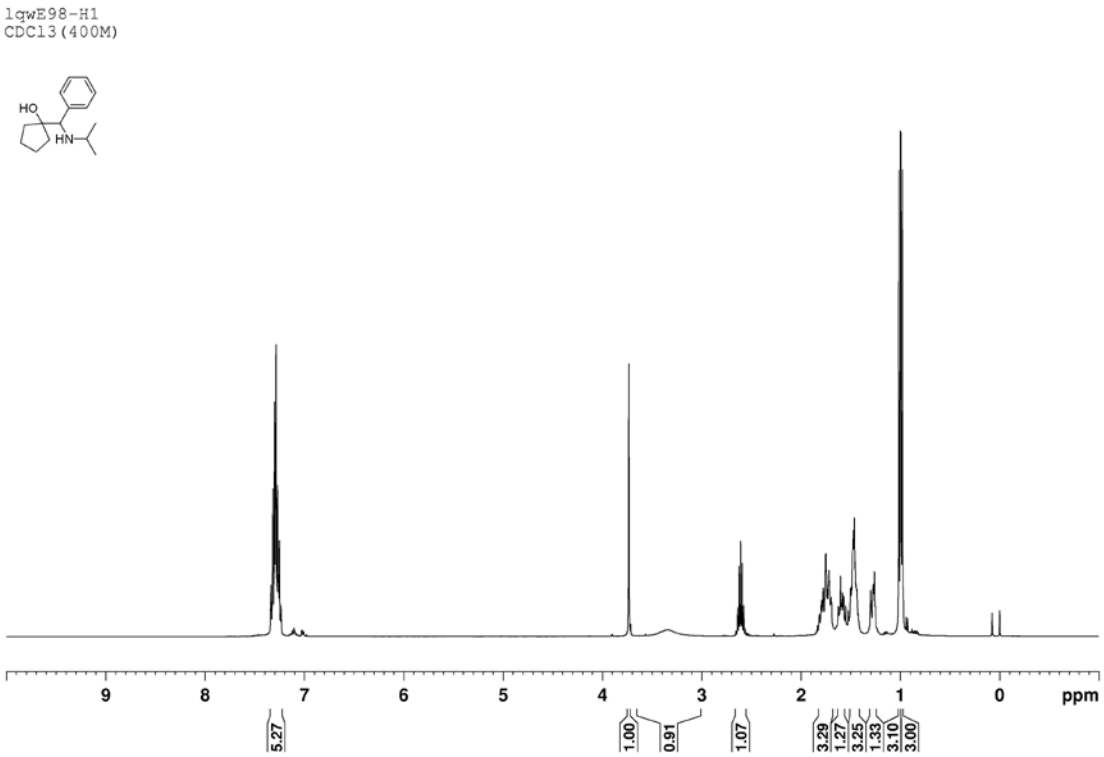


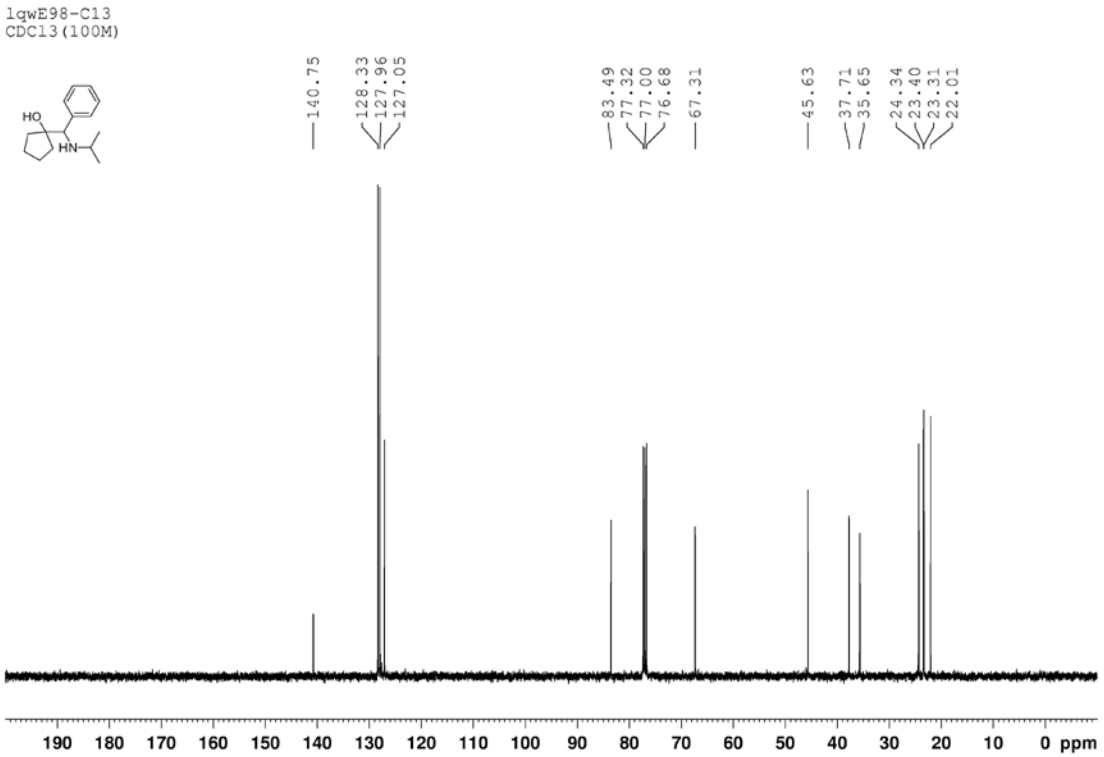
^{1}H and ^{13}C NMR spectra of *dl*-**2s**:



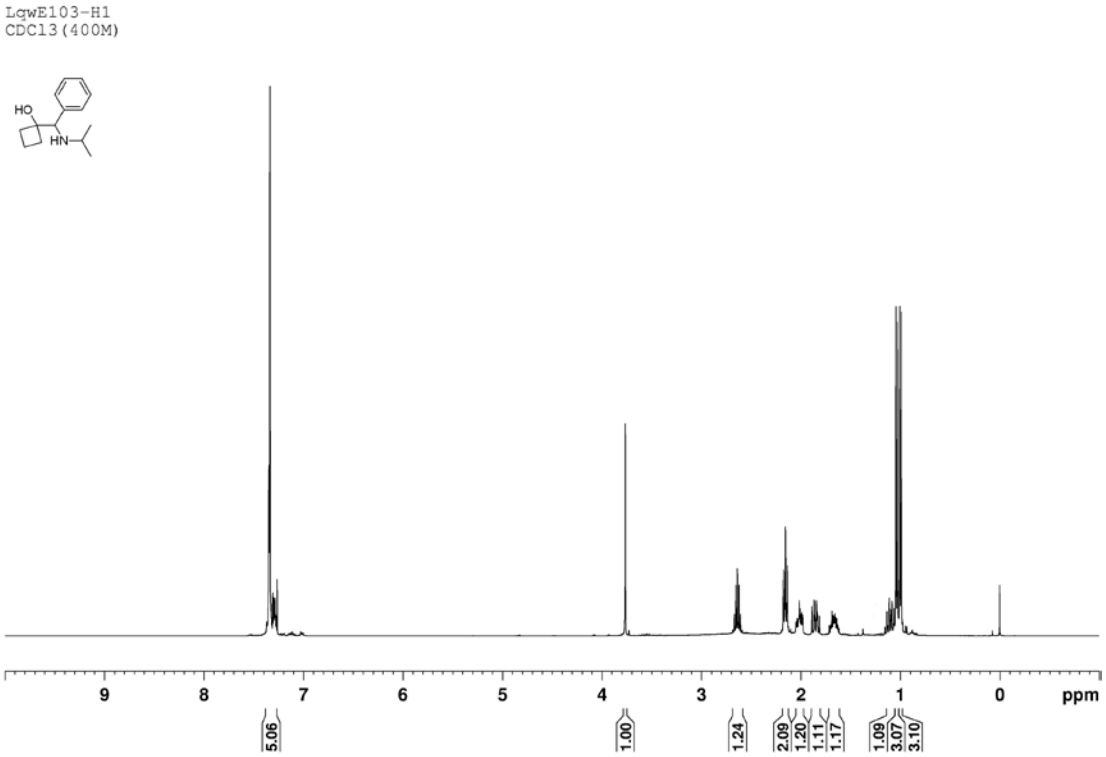


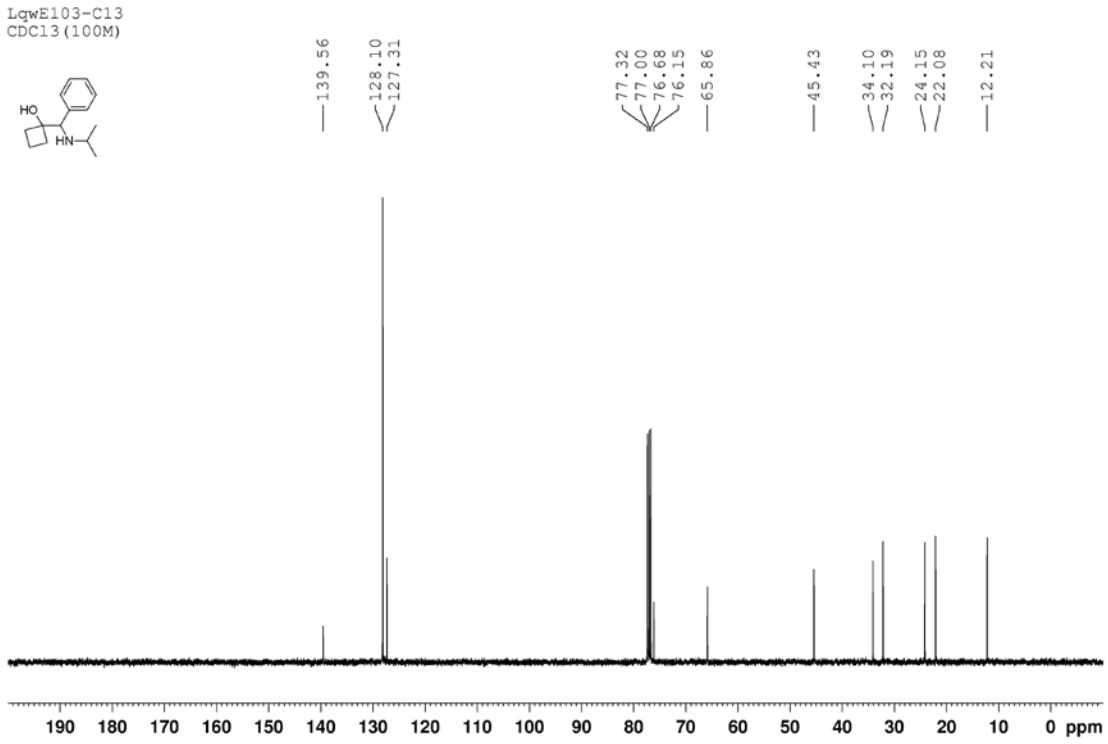
^{1}H and ^{13}C NMR spectra of **3a**:



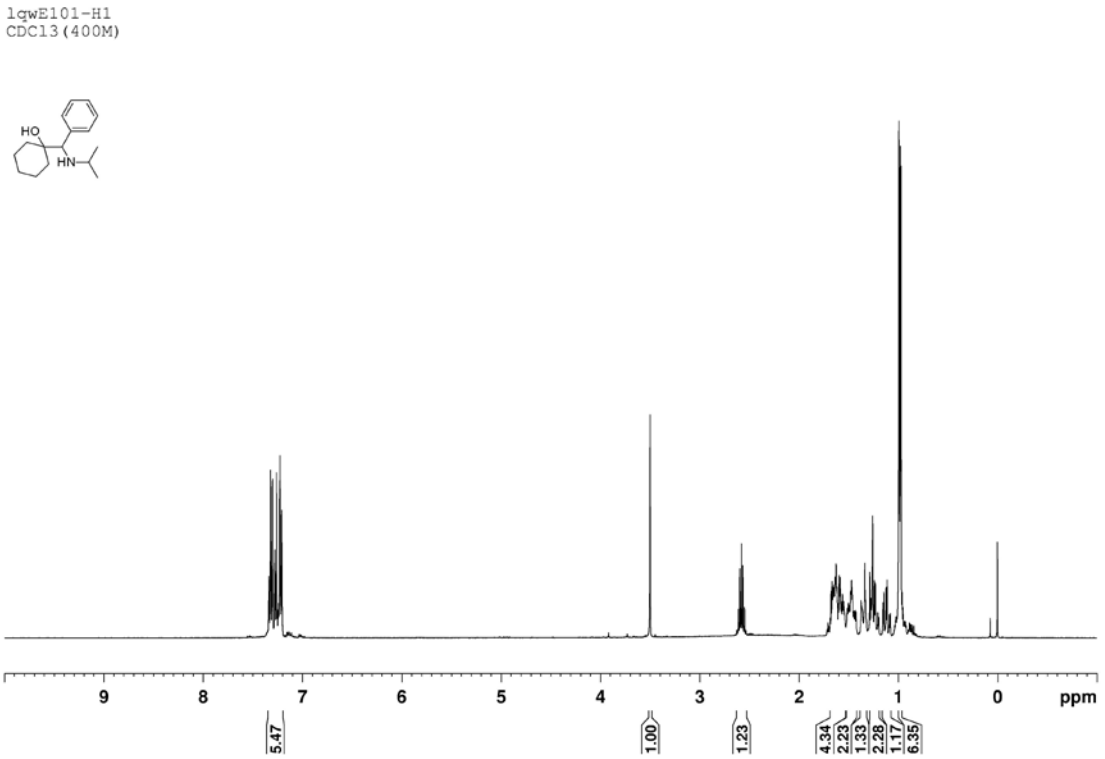


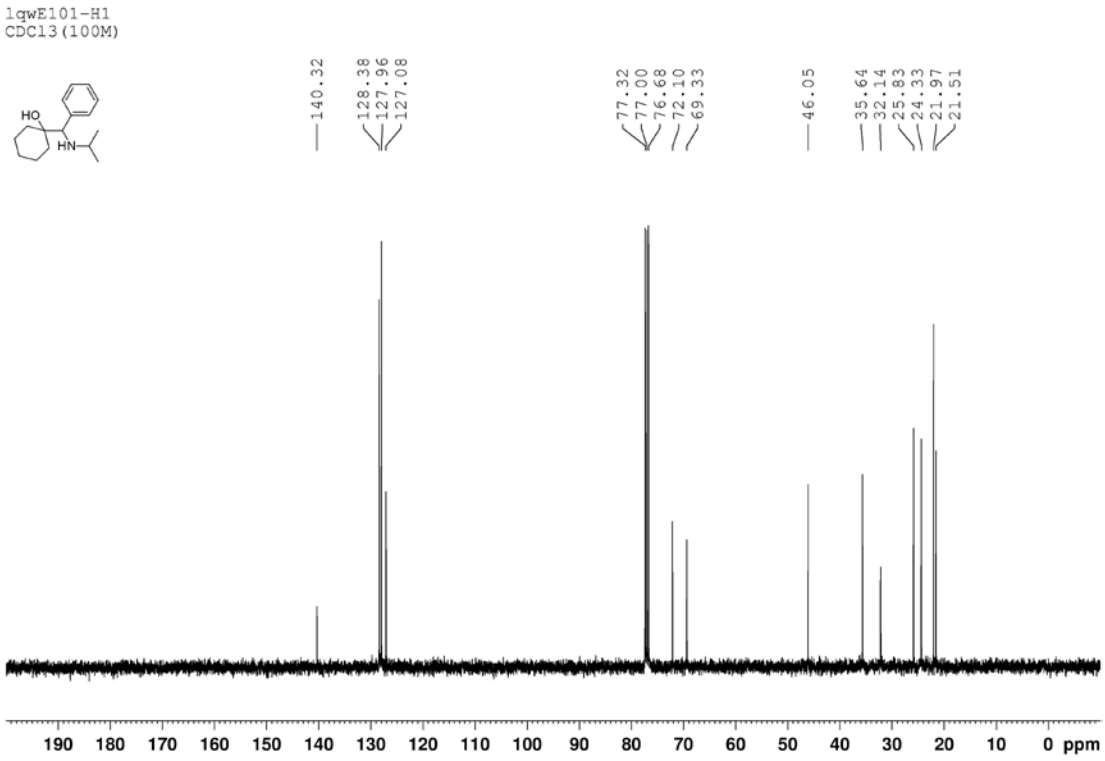
^{1}H and ^{13}C NMR spectra of **3b**:



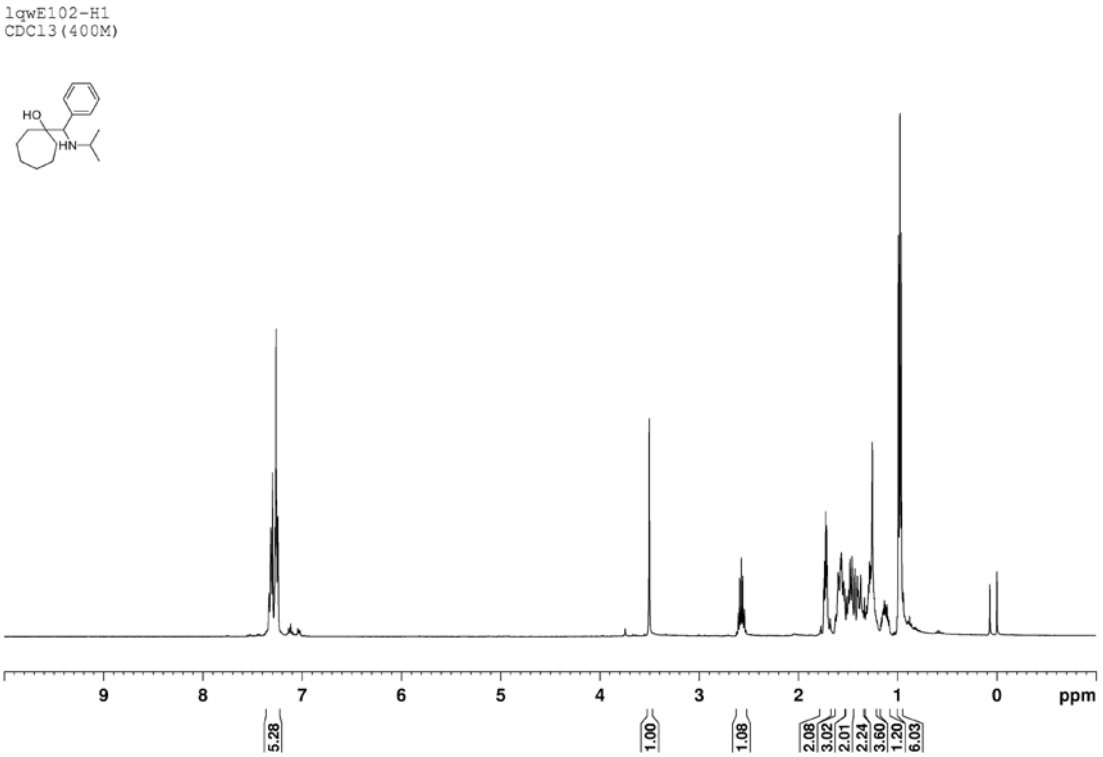


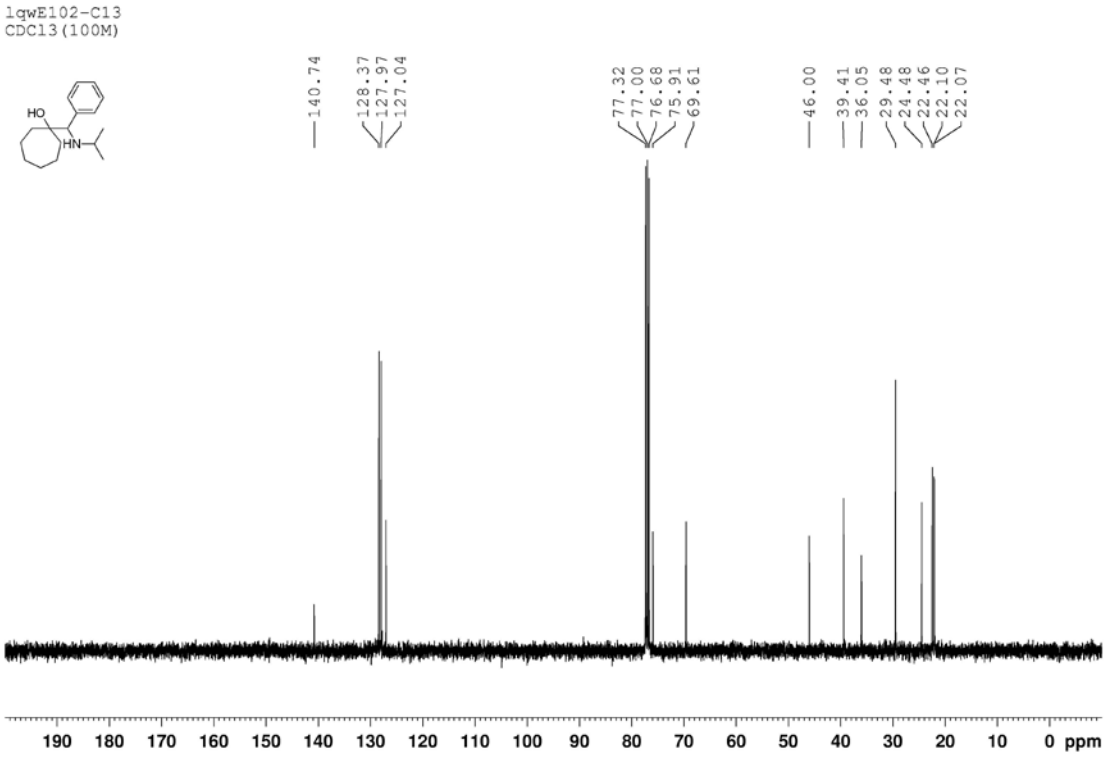
^{1}H and ^{13}C NMR spectra of **3c**:



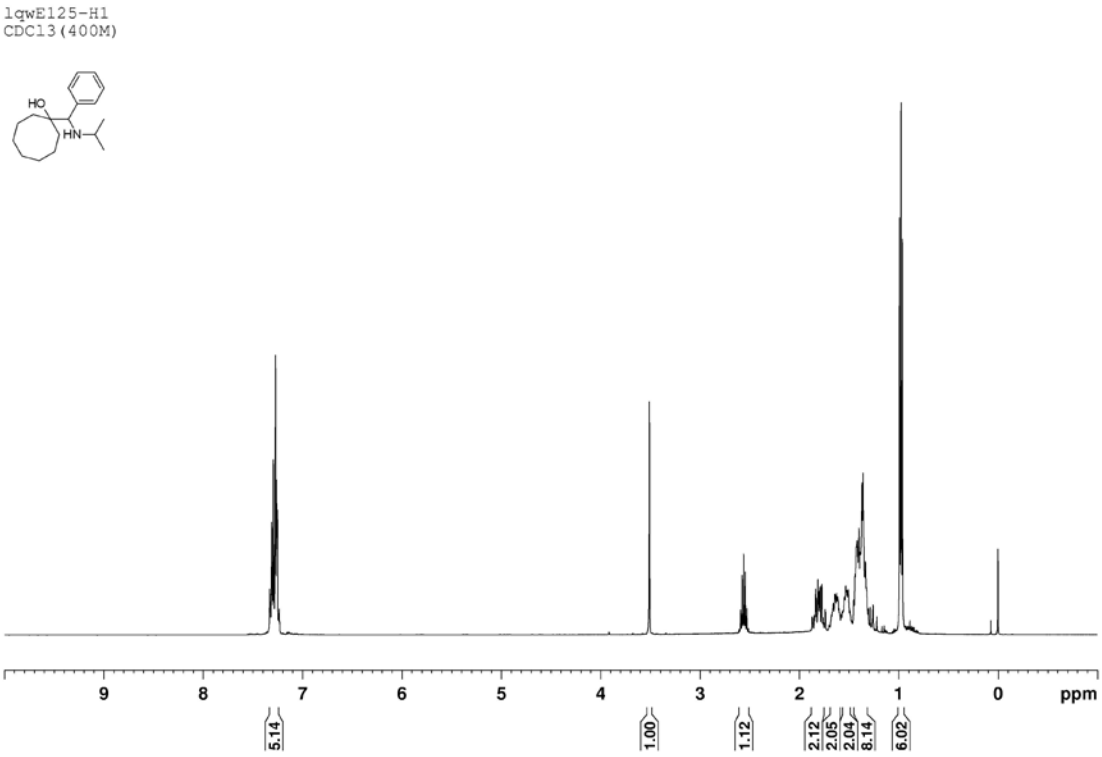


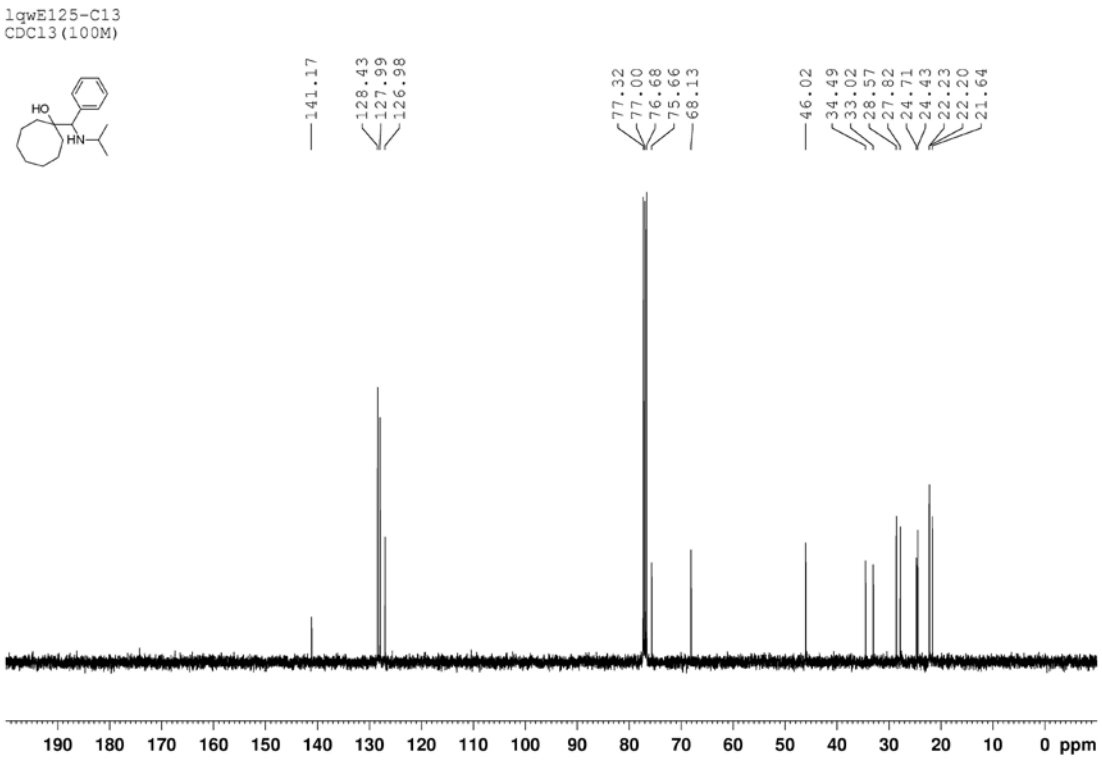
^{1}H and ^{13}C NMR spectra of **3d**:



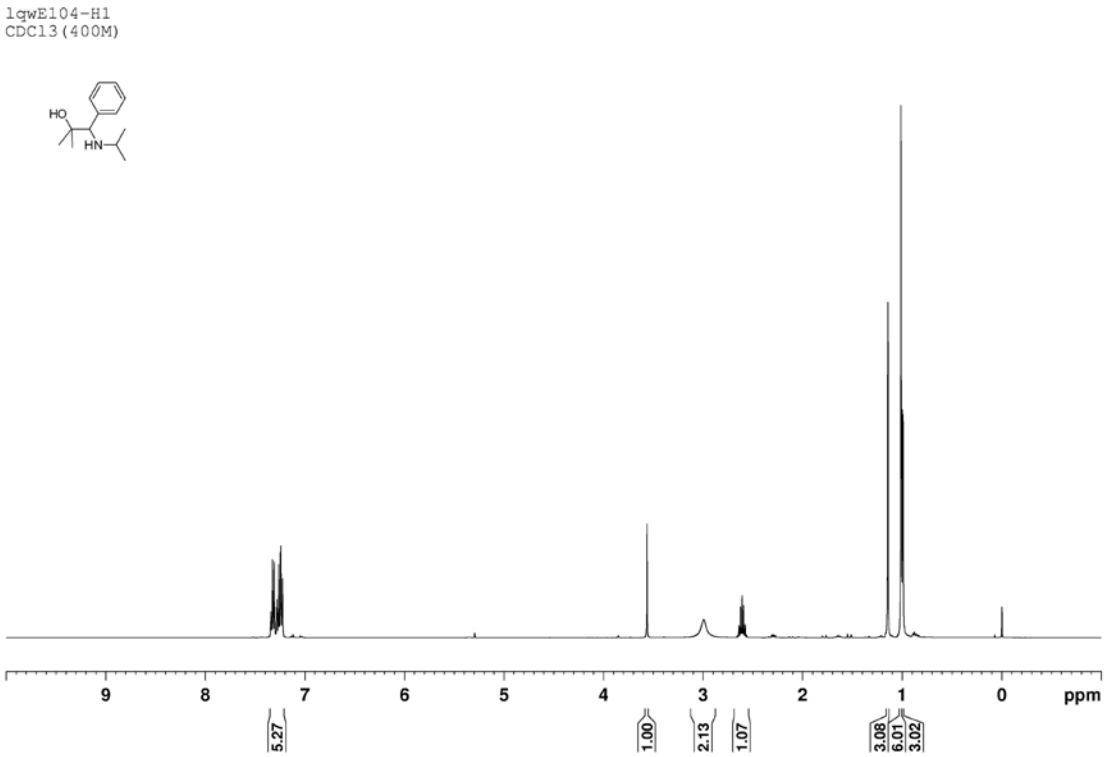


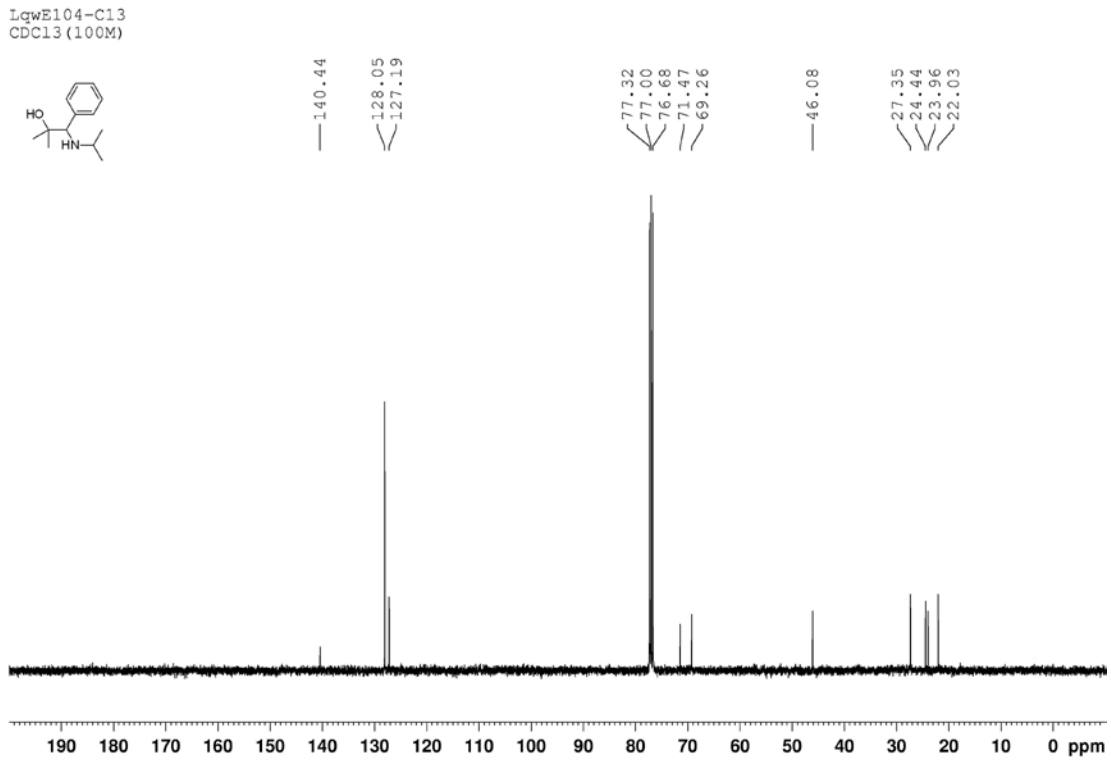
^{1}H and ^{13}C NMR spectra of **3e**:



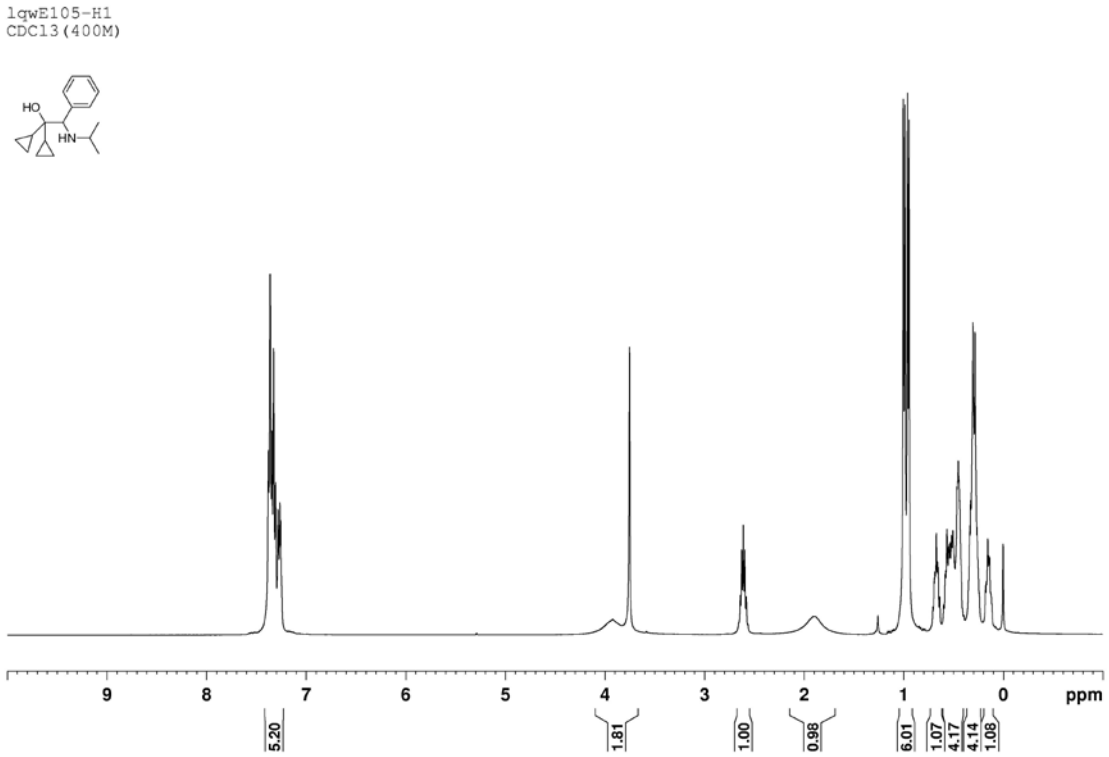


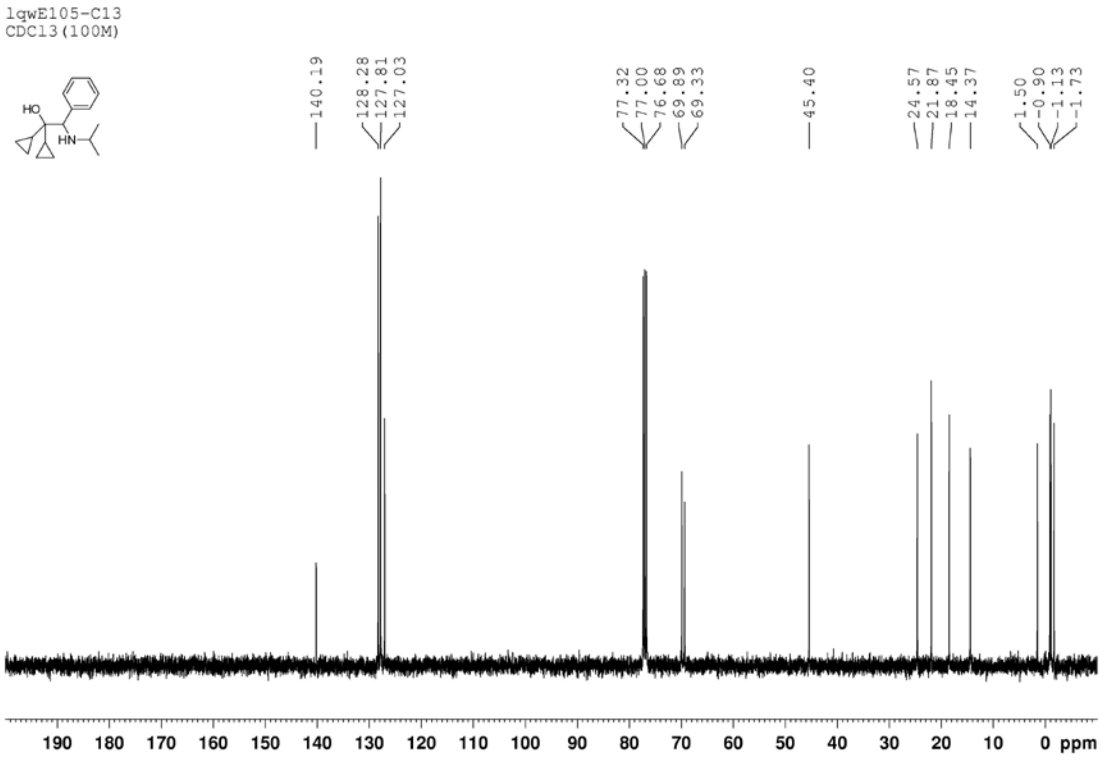
^{1}H and ^{13}C NMR spectra of **3f**:



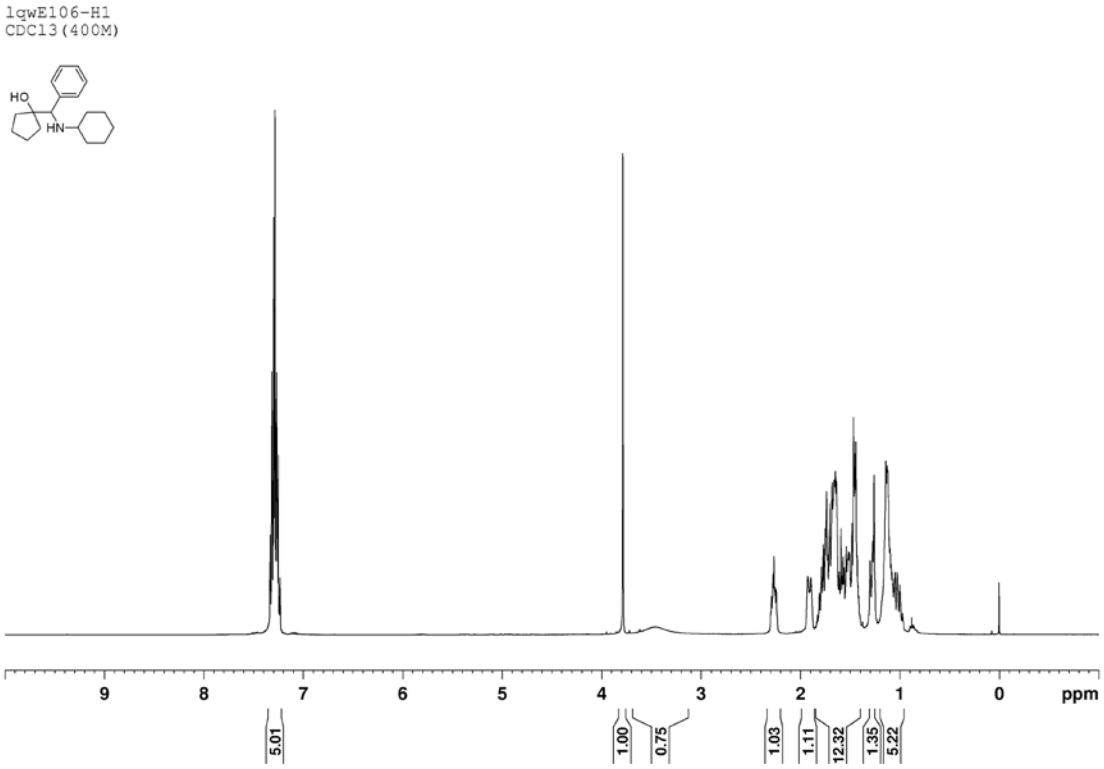


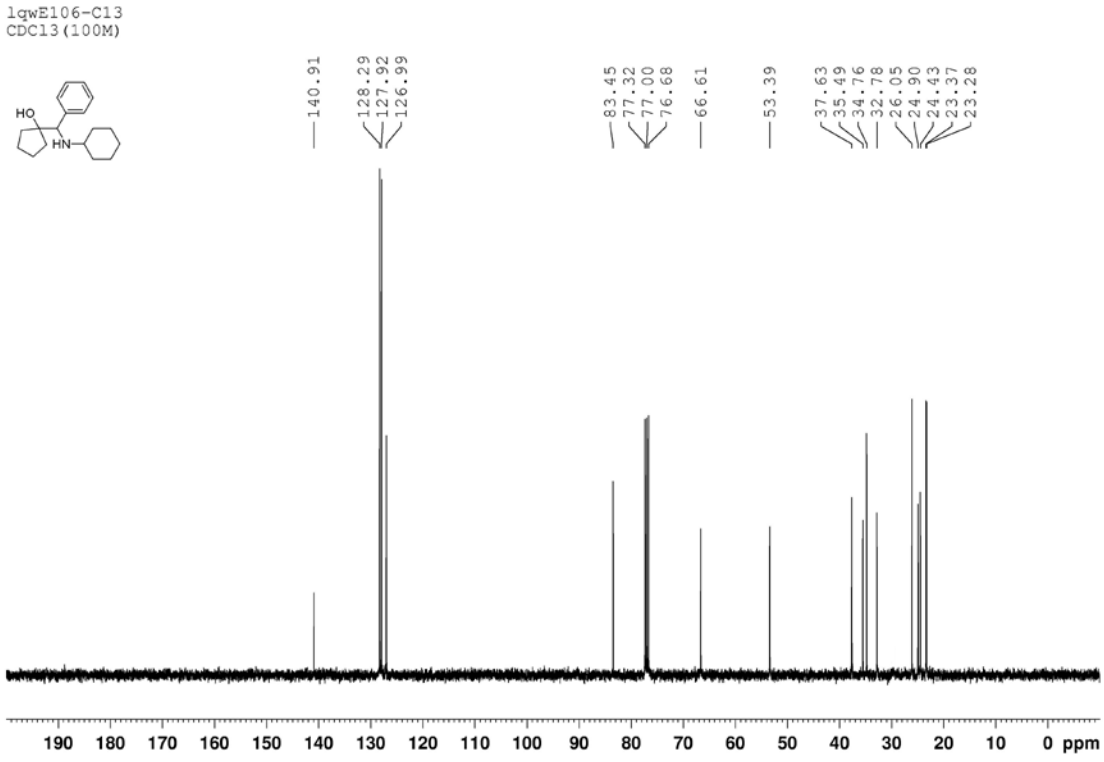
^{1}H and ^{13}C NMR spectra of **3g**:



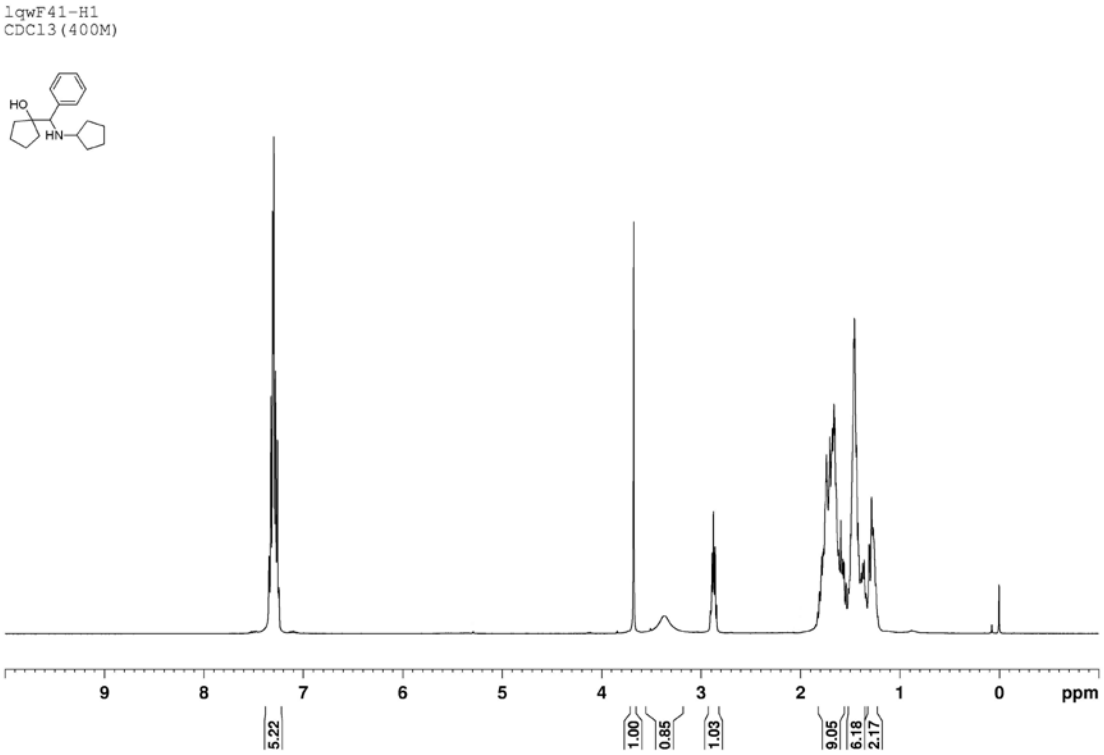


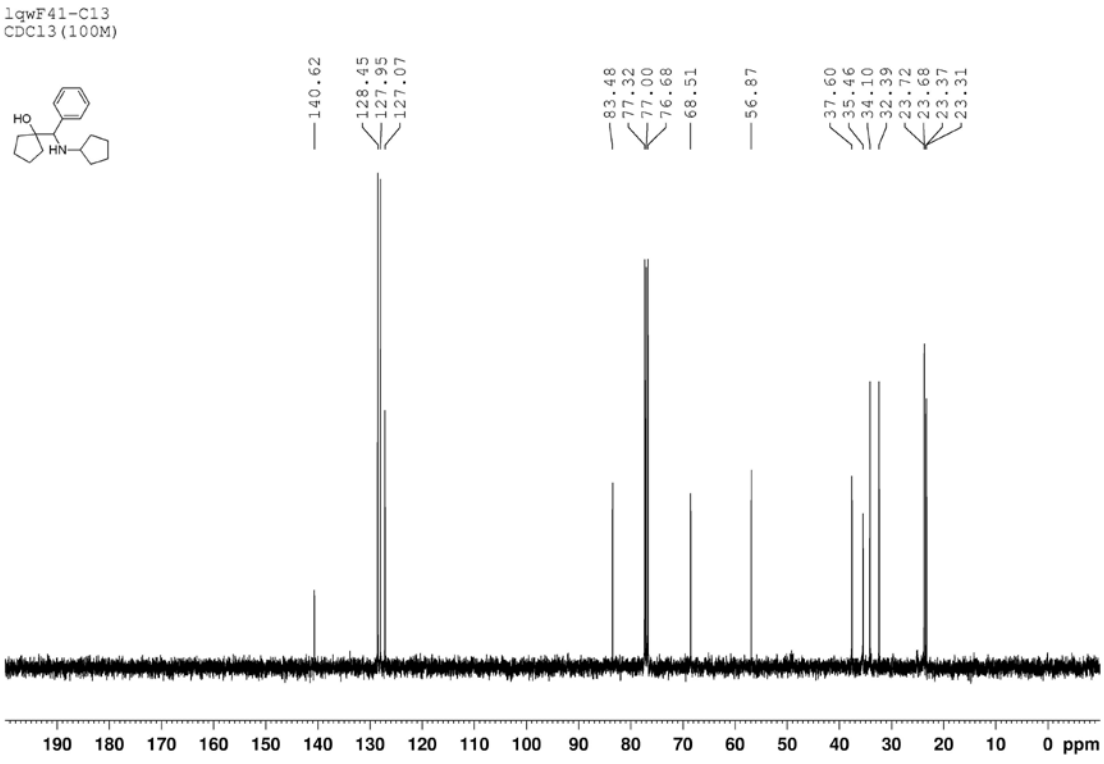
^{1}H and ^{13}C NMR spectra of **3h**:



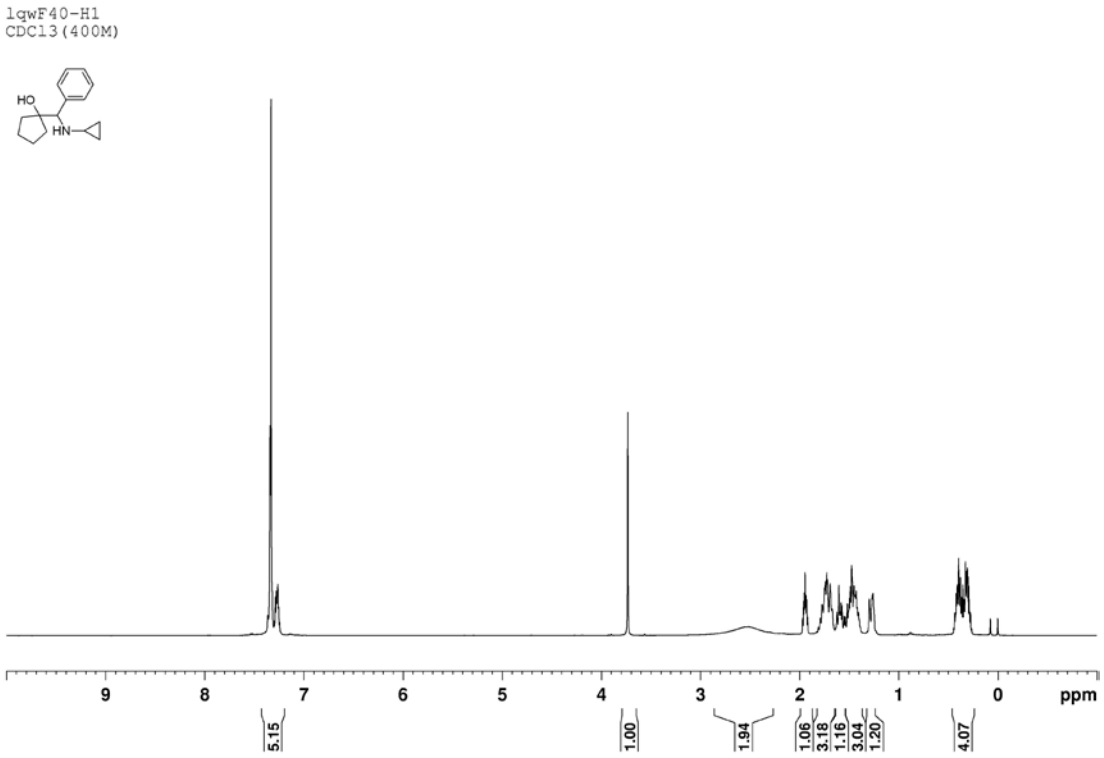


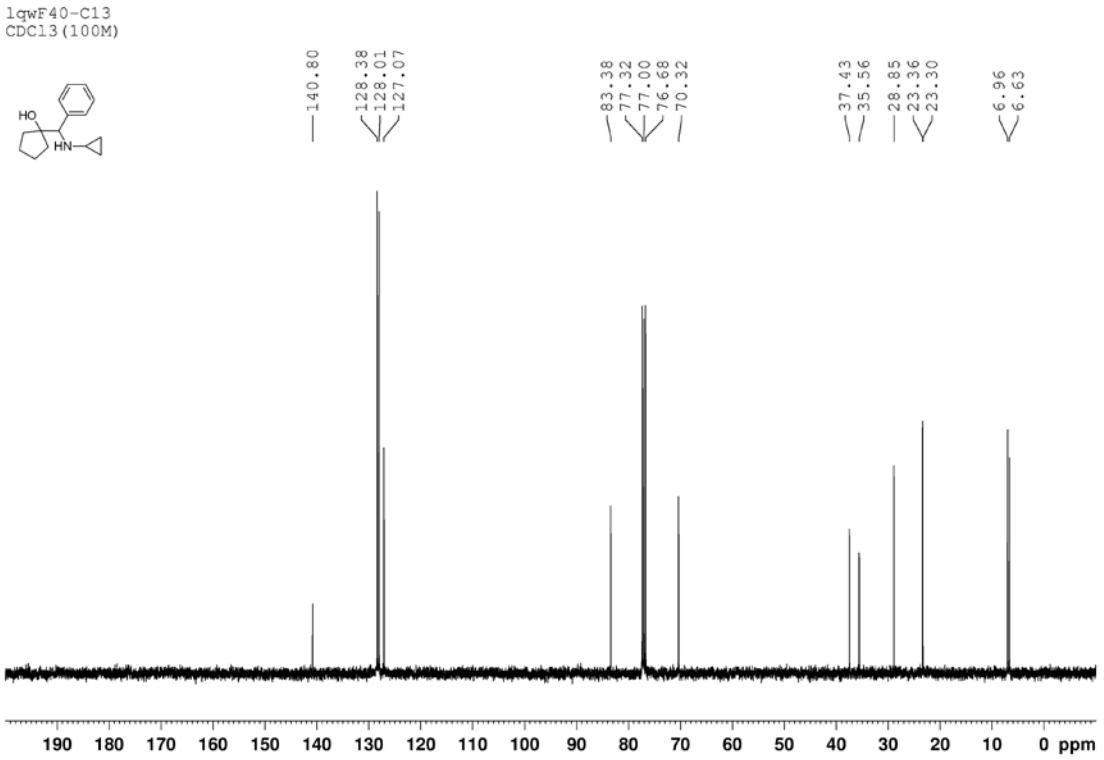
^{1}H and ^{13}C NMR spectra of **3i**:



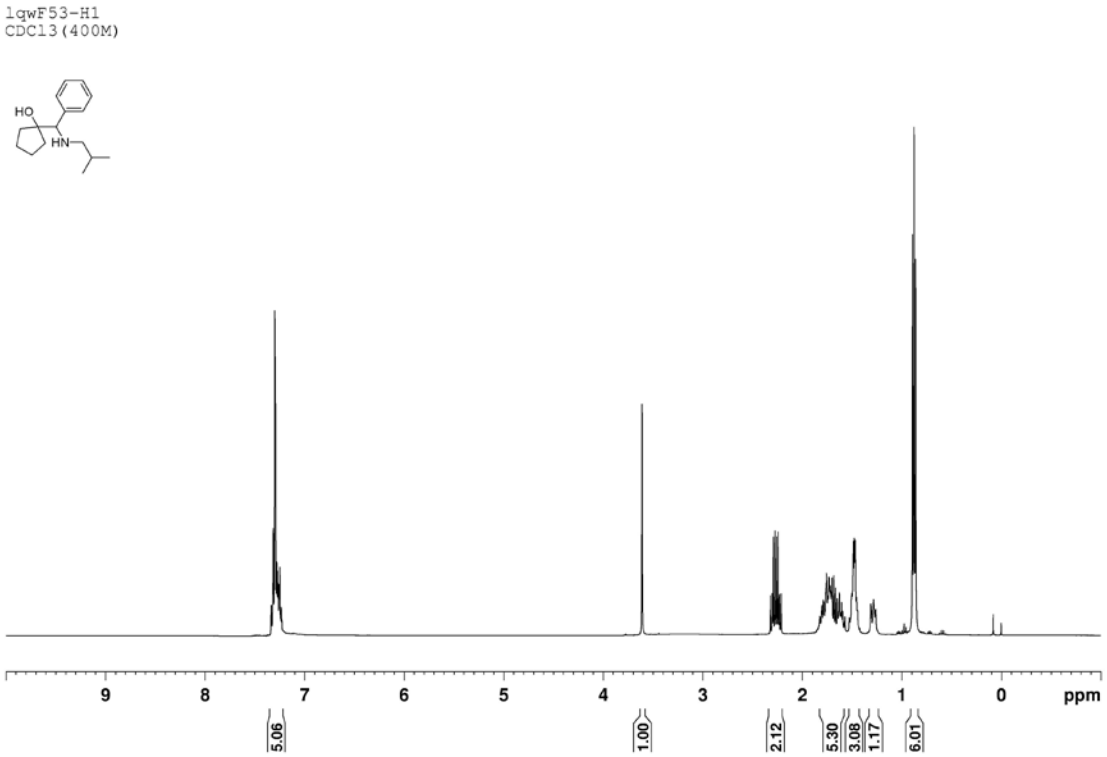


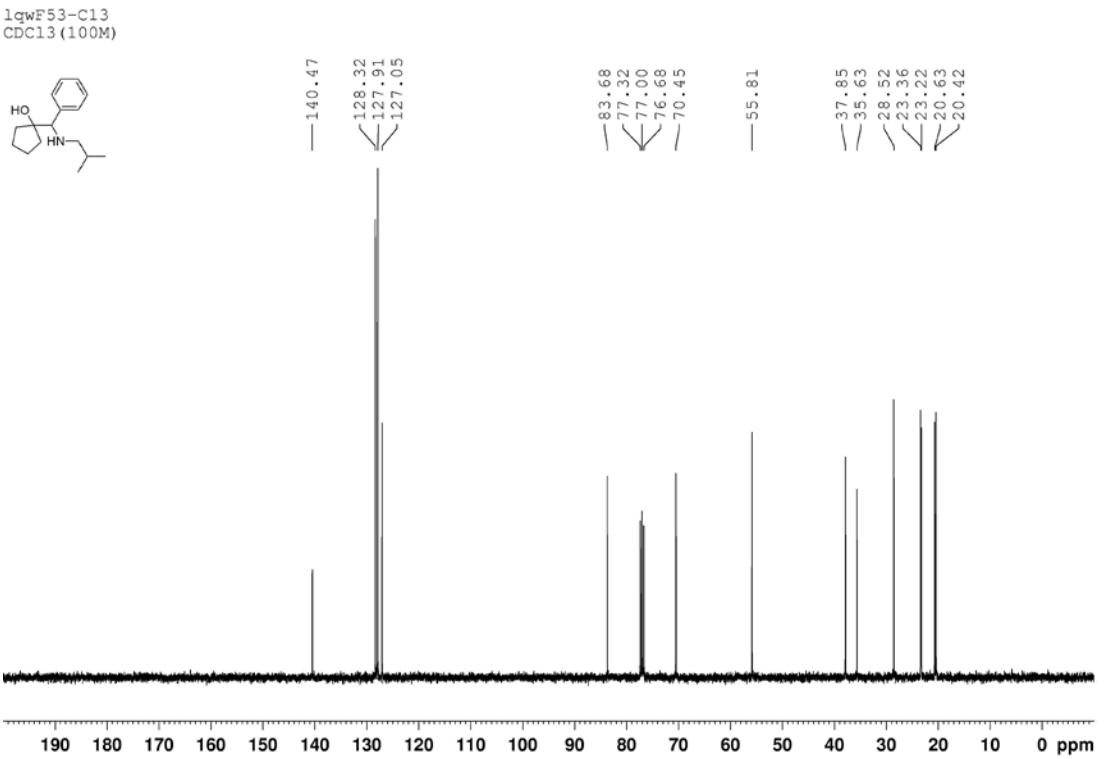
^{1}H and ^{13}C NMR spectra of **3j**:



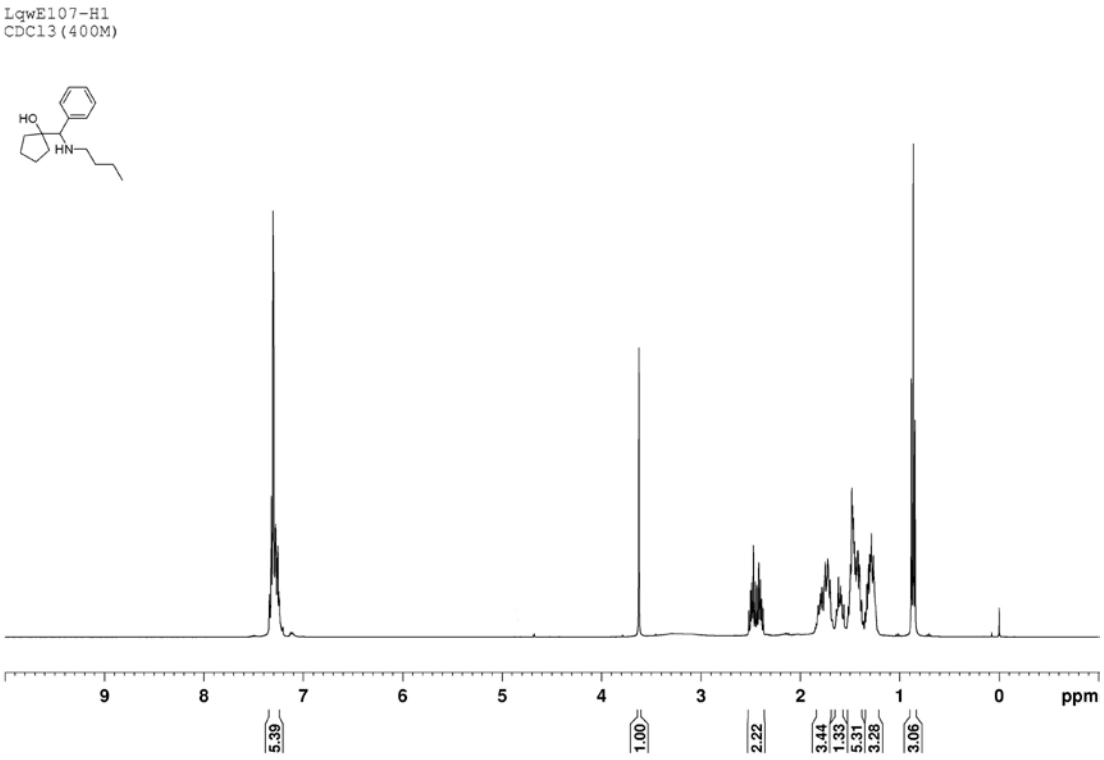


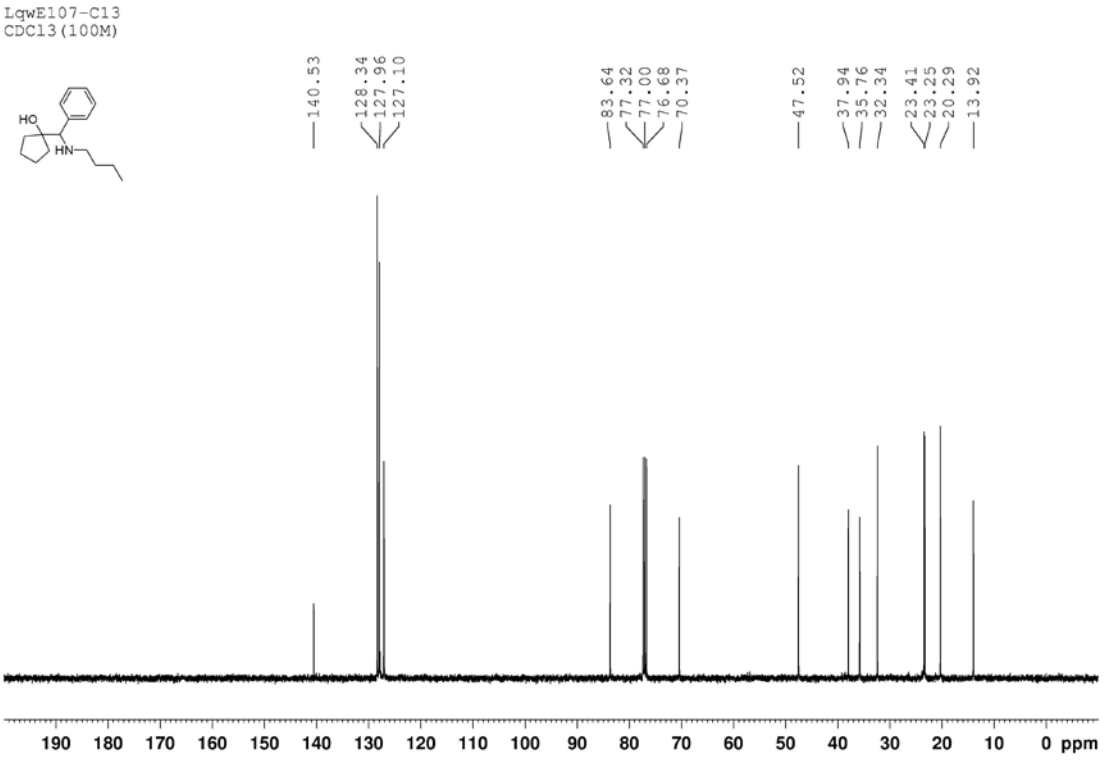
^{1}H and ^{13}C NMR spectra of **3k**:



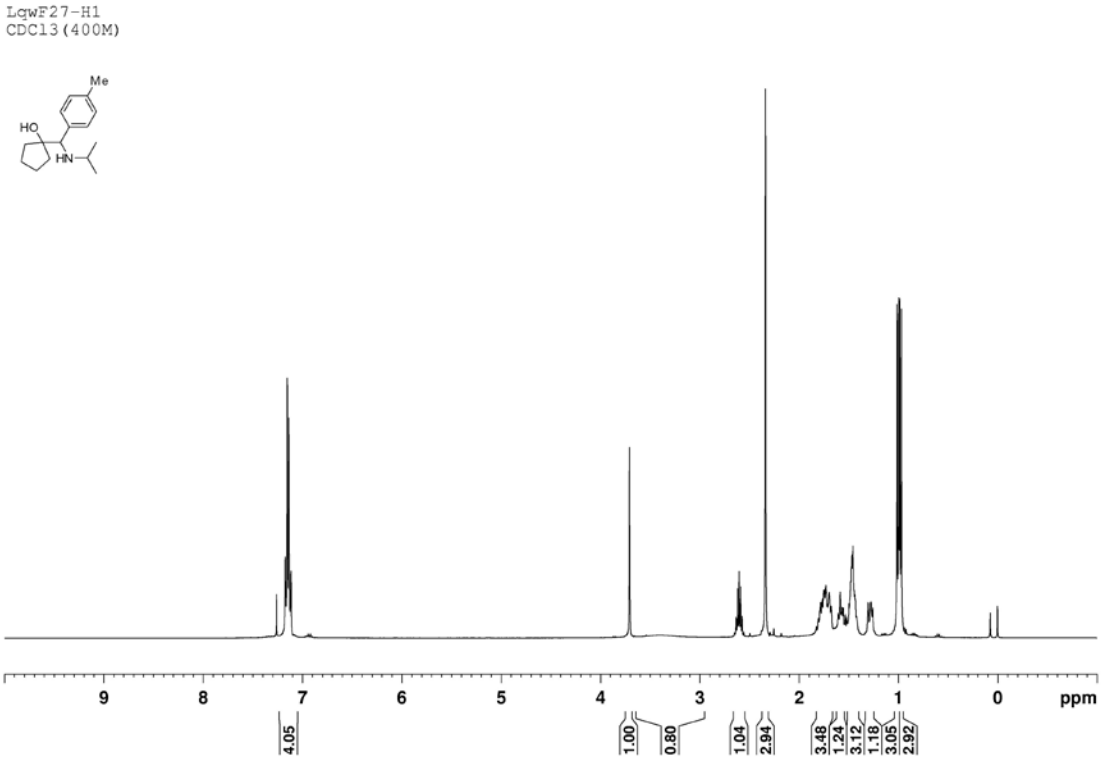


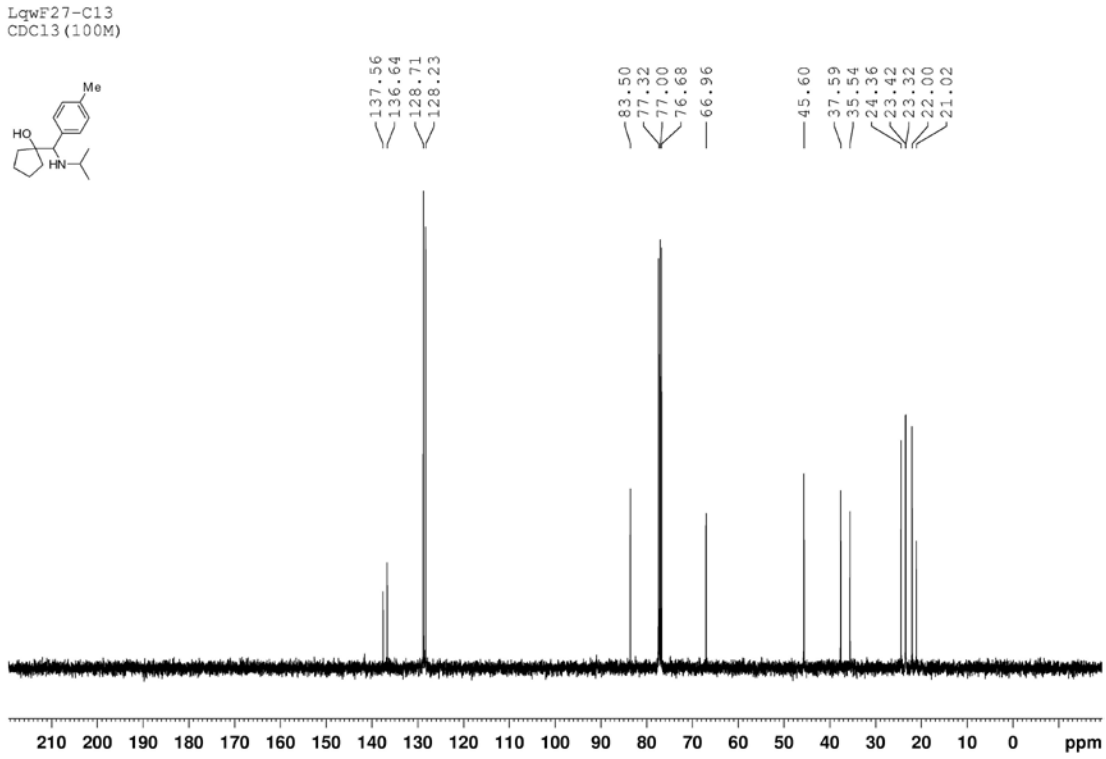
^{1}H and ^{13}C NMR spectra of **3l**:



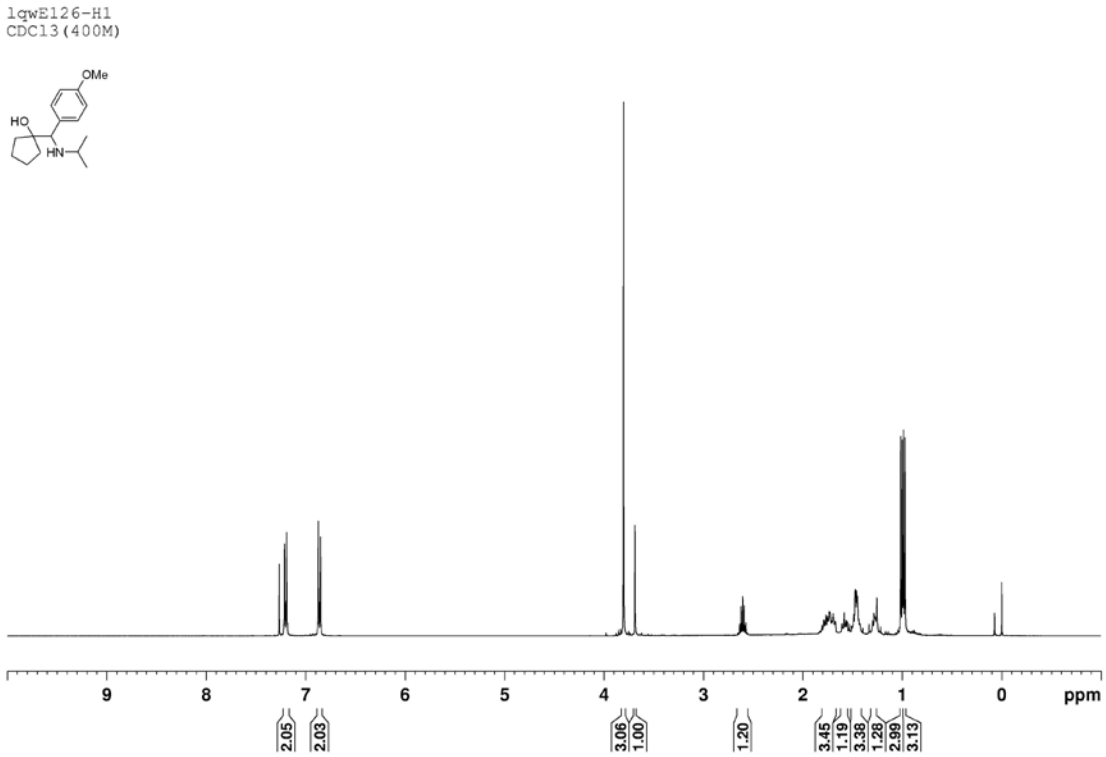


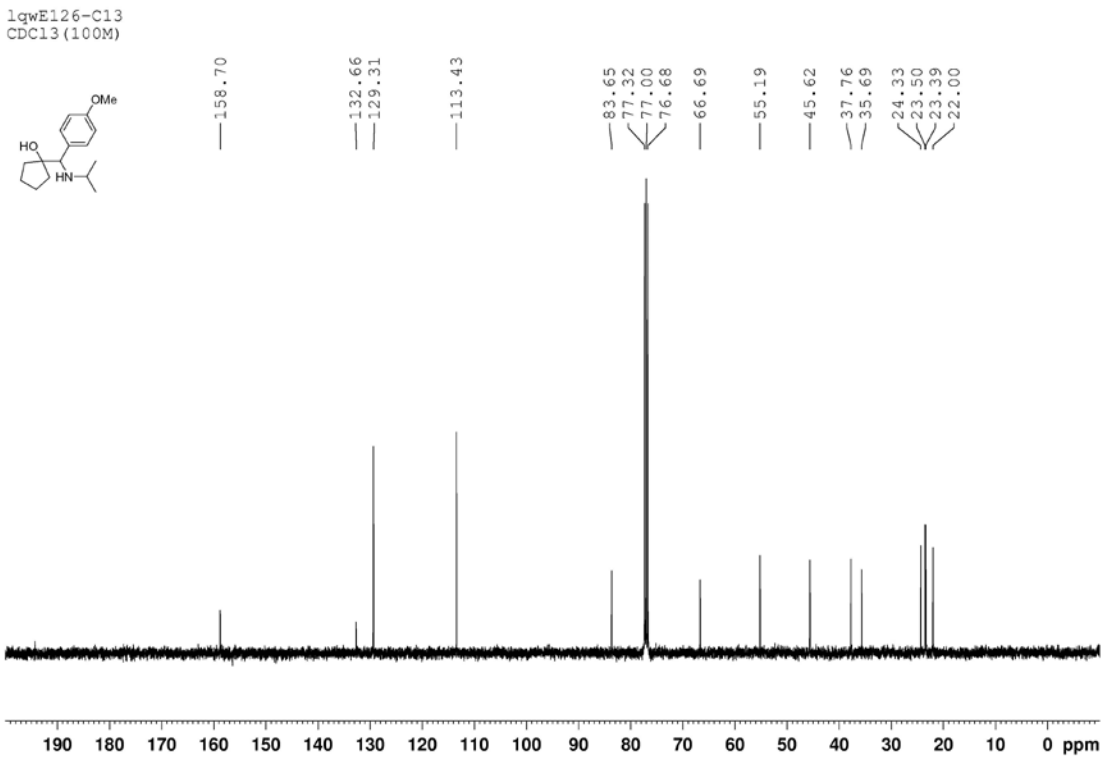
^{1}H and ^{13}C NMR spectra of **3m**:



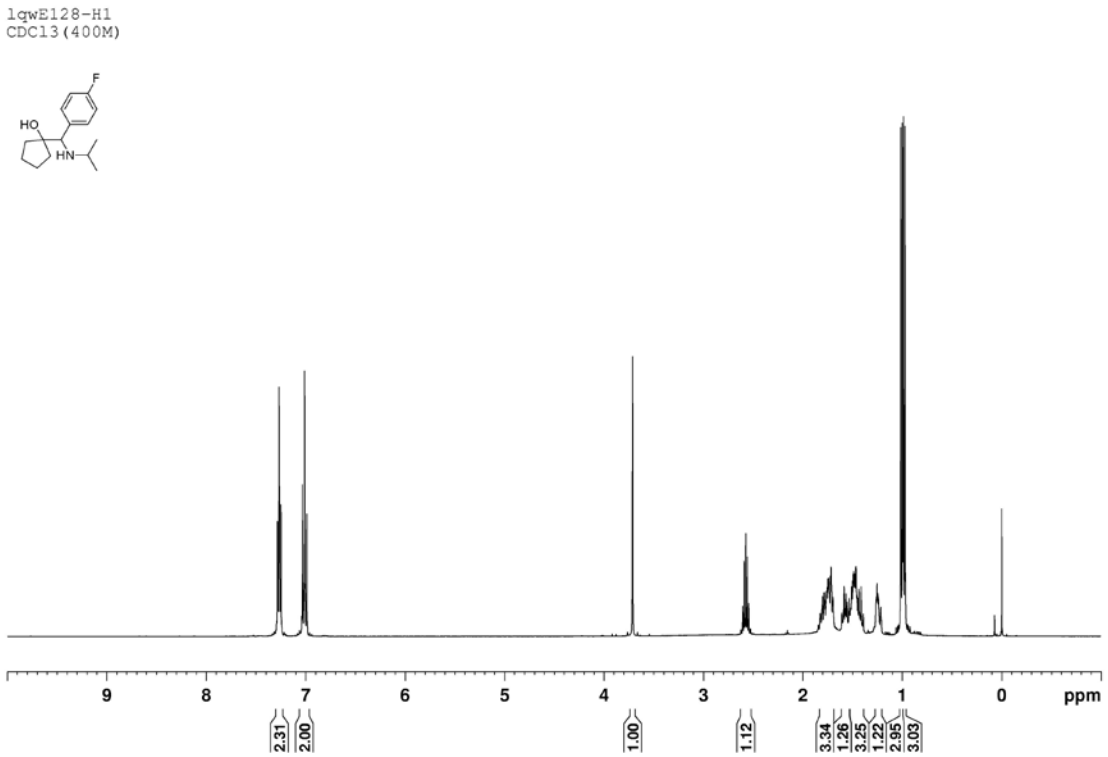


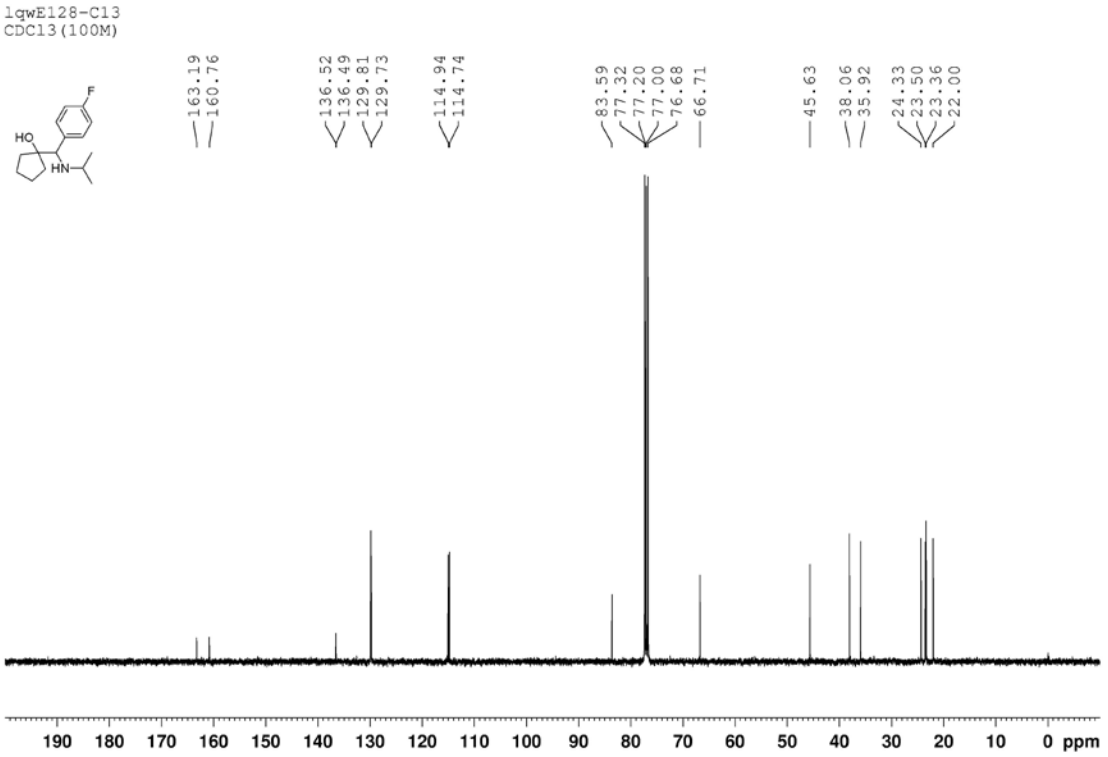
^{1}H and ^{13}C NMR spectra of **3n**:





^{1}H and ^{13}C NMR spectra of **3o**:





^{1}H and ^{13}C NMR spectra of **3p**: