

Supporting Information

Stereoselective One-pot, Three-Component Synthesis of 4-Aryltetrahydropyran via Prins-Friedel-Crafts reaction

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Supplementary informations: Experimental procedures, ^1H and ^{13}C NMR, IR and elemental analysis of compounds **1b-21b**, **22-30** and ^{19}F NMR spectral data of **4b** and **8b**; ^1H and ^{13}C NMR spectra of **1b-21b**, **22-30** and ^{19}F NMR spectra of **4b** and **8b**; X-ray structure and crystal parameters of compound **2b**.

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General procedure for the synthesis of compounds 1b-21b and 22-25: To a mixture of aldehydes (1.0 equiv.) in arene (3.0 mL) was added boron trifluoride etherate (1.2 equiv.). The mixture was cooled to 0 °C and then homoallyl alcohol (1.0 equiv) in aryl compound (2.0 mL) was added drop by drop over 10 min. The temperature was slowly brought to rt in 1h. The reaction mixture was stirred at rt for specified time. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluents. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel to give the title compounds.

Synthesis of 2,4-diphenyltetrahydropyran (1b, Table 1): To a solution of benzaldehyde **1a** (0.20 mL, 2.0 mmol) in benzene (3.0 mL) was added boron trifluoride etherate (0.30 mL, 2.4 mmol). The mixture was cooled to 0 °C and then buten-1-ol (144 mg, 2.0 mmol) in benzene (2.0 mL) was added drop by drop over 10 min. The temperature was slowly brought to rt in 1h. The reaction mixture was stirred at rt for 6h. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel to give 2,4-diphenyltetrahydropyran **1b** (358 mg, 75%) as a gum.

General procedure for the synthesis of compounds 26 and 27: To a mixture of *m*-nitrobenzaldehyde (1.0 equiv) in CH₂Cl₂ (2.0 mL) was added boron trifluoride etherate (1.2 equiv). The mixture was cooled to 0 °C and then homoallyl alcohol (1.0 equiv) and nucleophile (arene) (5.0 eq.) in CH₂Cl₂ (1.0 mL) was added drop by drop over 10 min. The temperature was slowly brought to rt in 1h. The reaction mixture was stirred at rt for specified time. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluents. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel to give the title compounds.

4-(2-Methoxy-phenyl)-2-(3-nitro-phenyl) tetrahydro-pyran and 4-(4-Methoxy-phenyl)-2-(3-nitro-phenyl) tetrahydro-pyran (26*o/p*, table 2): To a mixture of *m*-nitrobenzaldehyde (302 mg, 2.0 mmol) in CH₂Cl₂ (2.0 mL) was added boron trifluoride etherate (0.30 mL, 2.4 mmol). The reaction mixture was cooled to 0 °C and then a solution of 3-buten-1-ol (144 mg, 2.0 mmol) and anisole (1.0 mL, 9.2 mmol) in CH₂Cl₂ (1.0 mL) was added drop by drop over 10 min. The temperature was then slowly increased to rt in 1h. The reaction mixture was stirred at rt for 6h. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel to give **26*o/p*** as two regioisomers with a *o/p* ratio 2:1 and 53% and 27% yields respectively (501 mg, overall 80%).

General procedure for the three component reaction with ketone 28-30:

To a solution of ketone (1 equiv) and 3-buten-ol (1 equiv) in benzene was added boron trifluoride etherate (1.2 equiv). The reaction mixture was stirred for the specified time at 40 °C. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluents. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel. The product was characterized by spectrometric methods.

Synthesis of 4-Phenyl-1-oxa-spiro[5.5]undecane (28, Table 3): To a solution of cyclohexanone (196 mg, 2.0 mmol) and 3-buten-1-ol (144 mg, 2.0 mmol) in benzene (3.0 mL) was added boron trifluoride etherate (0.30 mL, 2.4 mmol). The reaction mixture was stirred at 40 °C for 8h. After completion of the reaction, the product was extracted with ethyl acetate, washed with brine and water. The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by short column chromatography over silica gel to give 4-Phenyl-1-oxa-spiro[5.5]undecane **28** (258 mg, 56%) as a gum.

2,4-Diphenyltetrahydropyran (1b): ^1H NMR (400 MHz, CDCl_3): Semisolid; δ 1.80 (q, $J = 12.0$ Hz, 1 H), 1.85-1.95 (m, 2 H), 2.08 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.97 (tt, $J = 11.6$ and 4.0 Hz, 1 H), 3.77 (dt, $J = 11.2$ and 3.2 Hz, 1 H), 4.29 (ddd, $J = 11.2$, 4.0 and 2.4 Hz, 1 H), 4.48 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.19-7.40 (m, 10 H); ^{13}C NMR (100 MHz, CDCl_3): δ 33.3, 41.5, 42.0, 68.5, 79.8, 125.7, 126.4, 126.7, 127.4, 128.3, 128.5, 142.9, 145.4. IR: 3061, 3029, 2938, 2845, 1494, 1452, 1374, 1252, 1128, 1088, 1041, 961, 918, 751, 701 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{18}\text{O}$: C, 85.67; H, 7.61. Found: C, 85.55; H, 7.78.

2-(4-Nitro-phenyl)-4-phenyl-tetrahydropyran(2b): Solid, M. P.: 77-78 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.1.64 (q, $J = 12.4$ Hz, 1 H), 1.85-1.95 (m, 2 H), 2.10 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.98 (tt, $J = 12.4$ and 4.0 Hz, 1 H), 3.76 (dt, $J = 11.6$ and 3.2 Hz, 1 H), 4.30 (ddd, $J = 11.6$, 4.0 and 2.0 Hz, 1 H), 4.57 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.19-7.33 (m, 5 H) 7.53 (d, $J = 8.8$ Hz, 2 H), 8.17 (d, $J = 8.8$ Hz, 2 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.2, 41.7, 42.0, 68.7, 78.8, 123.7, 126.5, 126.7, 126.8, 128.8, 145.0, 147.2, 150.3. IR: 3085, 2941, 2848, 1633, 1603, 1520, 1347, 1130, 1106, 1088, 850, 748 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_3$: C, 72.07; H, 6.05; N, 4.94. Found: C, 72.18; H, 6.21; N, 4.88.

2-(3-Nitro-phenyl)-4-phenyl-tetrahydropyran (3b): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.70 (q, $J = 12.4$ Hz, 1 H), 1.86-1.98 (m, 2 H), 2.13 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.99 (tt, $J = 11.6$ and 4.0 Hz, 1 H), 3.78 (dt, $J = 11.2$ and 3.6 Hz, 1 H), 4.33 (dd, $J = 10.0$ and 2.8 Hz, 1 H), 4.58-4.61 (m, 1 H), 7.21-7.36 (m, 5 H), 7.48-7.52 (t, $J = 8.0$ Hz, 1 H), 7.71 (d, $J = 8.0$ Hz, 1 H), 8.12 (d, $J = 8.0$ Hz, 1 H), 8.29 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.1, 41.6, 41.9, 68.7, 78.5, 120.9, 122.3, 126.6, 126.8, 128.7, 129.3, 131.9, 145.0, 145.1, 148.3. IR: 3086, 3062, 3028, 2917, 2848, 1602, 1526, 1349, 1130, 1091, 845, 807, 759 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_3$: C, 72.07; H, 6.05; N, 4.94. Found: C, 72.10; H, 6.18; N, 5.03.

2-(4-Fluoro-phenyl)-4-phenyl-tetrahydro-pyran (4b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.72 (q, $J = 12.0$ Hz, 1 H), 1.80-1.93 (m, 2 H), 2.04 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.95 (tt, $J = 11.6$ and 4.4 Hz, 1 H), 3.75 (dt, $J = 11.6$ and 3.6 Hz, 1 H), 4.27

(ddd, $J = 11.6, 4.4$ and 2.4 Hz, 1 H), 4.45 (dd, $J = 9.2$ and 2.0 Hz, 1 H), 6.99-7.05 (m, 2 H), 7.19-7.25 (m, 3 H), 7.29-7.37 (m, 4 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.2, 41.5, 41.9, 68.4, 79.0, 115.0 (d, $J = 20.6$ Hz), 126.4, 126.7, 127.3 (d, $J = 7.6$ Hz), 128.5, 138.8, 145.3, 162.2 (d, $J = 243$ Hz). ^{19}F NMR (376 MHz, $\text{CDCl}_3/\text{C}_6\text{F}_6$): δ 46.44-46.51 (m, 1 F). IR: 3062, 3029, 2918, 2846, 1604, 1509, 1374, 1252, 1223, 1157, 1128, 1086, 833, 777, 758, 700 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{FO}$: C, 79.66; H, 6.69. Found: C, 79.59; H, 6.72.

2-(4-Chloro-phenyl)-4-phenyl-tetrahydropyran (5b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.70 (q, $J = 11.2$ Hz, 1 H), 1.80-1.93 (m, 2 H), 2.05 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.94 (tt, $J = 12.0$ and 3.6 Hz, 1 H), 3.74 (dt, $J = 11.6$ and 3.6 Hz, 1 H), 4.27 (ddd, $J = 11.2, 4.4$ and 2.0 Hz, 1 H), 4.45 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.18-7.33 (m, 9 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.4, 41.6, 42.1, 68.7, 79.2, 126.6, 126.9, 127.3, 128.6, 128.7, 133.1, 141.5, 145.4. IR: 3060, 3028, 2916, 2845, 1601, 1491, 1374, 1129, 1088, 825, 758, 699 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{ClO}$: C, 74.86; H, 6.28. Found: C, 74.77; H, 6.37.

2-(3-Chloro-phenyl)-4-phenyl-tetrahydropyran (6b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.70 (q, $J = 12.4$ Hz, 1 H), 1.81-1.93 (m, 2 H), 2.06 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.95 (tt, $J = 12.0$ and 4.0 Hz, 1 H), 3.75 (dt, $J = 11.6$ and 3.6 Hz, 1 H), 4.28 (ddd, $J = 12.0, 4.0$ and 2.0 Hz, 1 H), 4.46 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.19-7.33 (m, 8 H), 7.40 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.1, 41.4, 41.8, 68.4, 78.8, 123.8, 125.9, 126.4, 126.7, 127.3, 128.5, 129.5, 134.1, 145.0, 145.1. IR: 3062, 3028, 2917, 2939, 2845, 1600, 1575, 1429, 1355, 1130, 1086, 863, 784, 758, 699 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{ClO}$: C, 74.86; H, 6.28. Found: C, 74.91; H, 6.17.

2-(3-Bromo-phenyl)-4-phenyl-tetrahydropyran (7b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.70 (q, $J = 12.0$ Hz, 1 H), 1.81-1.93 (m, 2 H), 2.05 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.94 (tt, $J = 11.6$ and 3.6 Hz, 1 H), 3.74 (dt, $J = 11.6$ and 4.4 Hz, 1 H), 4.28 (ddd, $J = 11.6, 3.6$ and 2.4 Hz, 1 H), 4.44 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.17-7.40 (m, 8 H), 7.56 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.0, 41.3, 41.7, 68.3, 78.6, 122.3, 124.3, 126.3, 126.6, 128.4, 128.7, 129.8, 130.2, 145.0, 145.2. IR: 3062, 3028, 2939,

2846, 1597, 1568, 1426, 1372, 1129, 1084, 884, 783, 758, 697 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{BrO}$: C, 64.37; H, 5.40. Found: C, 64.45; H, 5.55.

4-Phenyl-2-(4-trifluoromethyl-phenyl)-tetrahydropyran (8b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.70 (q, $J = 12.0$ Hz, 1 H), 1.82-1.94 (m, 2 H), 2.07 (dq, $J = 13.6$ and 2.0 Hz, 1 H), 2.97 (tt, $J = 12.0$ and 4.4 Hz, 1 H), 3.76 (dt, $J = 11.6$ and 4.0 Hz, 1 H), 4.30 (ddd, $J = 11.6$, 4.0 and 1.6 Hz, 1 H), 4.53 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 7.19-7.33 (m, 5 H), 7.48 (d, $J = 8.4$, 2 H), 7.58 (d, $J = 8.4$, 2 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.3, 41.7, 42.1, 68.6, 79.1, 124.4 (q, $J = 270.7$ Hz), 125.2, 126.1, 126.6, 126.8, 128.7, 129.5 (q, $J = 32.1$ Hz), 145.3, 147.1. ^{19}F NMR (376 MHz, $\text{CDCl}_3/\text{C}_6\text{F}_6$): δ 99.36 (s, 3F). IR: 3064, 3030, 2919, 2848, 1622, 1416, 1326, 1164, 1127, 1091, 1067, 838, 760, 700 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{17}\text{F}_3\text{O}$: C, 70.58; H, 5.59. Found: C, 70.64; H, 5.55.

4-(4-Phenyl-tetrahydropyran-2-yl)-benzoic acid methyl ester (9b): Solid; M. P. 61.2-63.4 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.71 (q, $J = 11.2$ Hz, 1 H), 1.82-1.95 (m, 2 H), 2.09 (dq, $J = 13.2$ and 1.6 Hz, 1 H), 2.97 (tt, $J = 11.6$ and 4.4 Hz, 1 H), 3.76 (dt, $J = 11.6$ and 3.2 Hz, 1 H), 3.90 (s, 3 H), 4.30 (ddd, $J = 11.6$, 4.0 and 2.0 Hz, 1 H), 4.54 (dd, $J = 11.2$ and 2.4 Hz, 1 H), 7.19-7.33 (m, 5 H), 7.45 (d, $J = 7.6$ Hz, 2 H), 8.02 (d, $J = 7.6$ Hz, 2 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.3, 41.6, 42.1, 52.1, 68.7, 79.4, 125.7, 126.6, 126.8, 128.7, 129.2, 129.8, 145.3, 148.0, 167.0. IR: 3061, 3029, 2950, 2845, 1722, 1611, 1435, 1279, 1112, 1089, 855, 757, 701 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_3$: C, 77.00; H, 6.80. Found: C, 77.12; H, 6.75.

2-Naphthalen-1-yl-4-phenyl-tetrahydropyran (10b): Semisolid, ^1H NMR (400 MHz, CDCl_3): δ 1.79-1.97 (m, 3 H), 2.07 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 3.05 (tt, $J = 11.6$ and 4.0 Hz, 1 H), 3.86 (dt, $J = 11.6$ and 3.2 Hz, 1 H), 4.38 (ddd, $J = 11.2$, 4.4 and 2.0 Hz, 1 H), 4.68 (dd, $J = 11.2$ and 1.6 Hz, 1 H), 7.18-7.32 (m, 5 H), 7.42-7.50 (m, 3 H), 7.80-7.84 (m, 4 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.6, 41.6, 42.3, 68.9, 80.1, 124.4, 124.5, 125.9, 126.2, 126.6, 127.0, 127.8, 128.2, 128.4, 128.7, 133.1, 133.6, 140.4, 145.6. IR: 3027, 3057, 2938, 2844, 1633, 1602, 1507, 1494, 1452, 1373, 1252, 1127, 1088, 962, 856, 819, 757, 700 cm^{-1} . Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{O}$: C, 87.46; H, 6.99. Found: C, 87.55; H, 7.12.

4-Phenyl-2-*p*-tolyl-tetrahydropyran (11b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.77 (q, $J = 12.0$ Hz, 1 H), 1.83-1.93 (m, 2 H), 2.05 (dq, $J = 13.2$ and 1.6 Hz, 1 H), 2.32 (s, 3 H), 2.95 (tt, $J = 12.0$ and 4.4 Hz, 1 H), 3.75 (dt, $J = 11.6$ and 3.6 Hz, 1 H), 4.27 (ddd, $J = 11.6$, 4.0 and 1.2 Hz, 1 H), 4.45 (dd, $J = 10.8$ and 1.6 Hz, 1 H), 7.13-7.33 (m, 9 H). ^{13}C NMR (100 MHz, CDCl_3): δ 21.1, 33.4, 41.6, 42.1, 68.6, 79.7, 125.8, 126.4, 126.7, 128.5, 129.0, 136.9, 139.9, 145.6. IR: 3059, 3027, 2937, 2844, 1602, 1515, 1453, 1374, 1252, 1129, 1086, 814, 758, 700 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{O}$: C, 85.67; H, 7.99. Found: C, 85.78; H, 8.14.

2-(4-Methoxy-phenyl)-4-phenyl-tetrahydro-pyran (12b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.77 (q, $J = 12.4$ Hz, 1 H), 1.82-1.93 (m, 2 H), 2.04 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.94 (tt, $J = 12.0$ and 4.0 Hz, 1 H), 3.73 (dt, $J = 11.2$ and 3.2 Hz, 1 H), 3.77 (s, 3 H), 4.26 (ddd, $J = 11.6$, 4.4 and 2.4 Hz, 1 H), 4.42 (dd, $J = 10.8$ and 2.0 Hz, 1 H), 6.87 (d, $J = 8.8$ Hz, 2 H), 7.18-7.33 (m, 7 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.5, 41.4, 42.2, 55.3, 68.8, 79.7, 113.8, 126.5, 126.9, 127.3, 128.7, 135.2, 145.7, 159.1. IR: 3060, 3001, 2936, 2838, 1612, 1513, 1453, 1375, 1248, 1176, 1108, 1128, 1084, 1036, 829, 758, 700 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{O}_2$: C, 80.56; H, 7.51. Found: C, 80.67; H, 7.45.

2-Cyclohexyl-4-phenyl-tetrahydro-pyran (13b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 0.95-1.08 (m, 2 H), 1.11-1.28 (m, 3 H), 1.36-1.49 (m, 2 H), 1.64-1.78 (m, 5 H), 1.81-1.86 (m, 1 H), 1.90-1.94 (m, 1 H), 1.98-2.12 (m, 1 H), 2.73 (tt, $J = 12.4$, 4.0 Hz, 1 H), 3.15 (ddd, $J = 11.2$, 6.4 and 2.0 Hz, 1 H), 3.51-3.57 (m, 1 H), 4.12 (dt, $J = 10.8$ and 3.2 Hz, 1 H), 7.19-7.33 (m, 5 H). ^{13}C NMR (100 MHz, CDCl_3): δ 26.4, 26.5, 26.8, 28.9, 29.3, 33.9, 36.6, 42.2, 43.4, 68.5, 82.4, 126.4, 127.0, 128.6, 146.3. IR: 3062, 3028, 2927, 2851, 1603, 1496, 1451, 1254, 1127, 1090, 757, 699 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{24}\text{O}$: C, 83.55; H, 9.90. Found: C, 83.46; H, 9.97.

2-Hexyl-4-phenyl-tetrahydropyran (14b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 0.95 (t, $J = 7.2$ Hz, 3 H), 1.26-1.35 (m, 6 H), 1.38-1.47 (m, 4 H), 1.54-1.61 (m, 1 H), 1.72-1.78 (m, 2 H), 1.84 (dq, $J = 12.8$ and 2.0 Hz, 1 H), 2.76 (tt, $J = 12.0$ and 4.0 Hz, 1 H), 3.36-3.42 (m, 1 H), 3.53-3.59 (m, 1 H), 4.11 (dt, $J = 10.8$ and 3.2 Hz, 1 H), 7.19-7.33 (m,

5 H). ^{13}C NMR (100 MHz, CDCl_3): δ 14.2, 22.8, 25.6, 29.6, 32.0, 33.8, 36.7, 39.7, 42.0, 68.3, 78.0, 126.4, 126.9, 128.6, 146.1. IR: 3063, 3029, 2931, 2857, 1604, 1496, 1454, 1379, 1252, 1135, 1088, 757, 699 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{26}\text{O}$: C, 82.87; H, 10.64. Found: C, 82.79; H, 10.57.

2-Ethyl-4-phenyl-tetrahydropyran (15b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 0.95 (t, $J = 7.6$ Hz, 3 H), 1.37-1.66 (m, 3 H), 1.73-1.79 (m, 2 H), 1.82-1.87 (m, 1 H), 2.76 (tt, $J = 12.4$ and 4.0 Hz, 1 H), 3.29-3.35 (m, 1 H), 3.53-3.60 (m, 1 H), 4.12 (dt, $J = 11.6$ and 3.2 Hz, 1 H), 7.19-7.33 (m, 5 H). ^{13}C NMR (100 MHz, CDCl_3): δ 10.1, 29.5, 33.8, 39.3, 42.0, 68.4, 79.4, 126.5, 127.0, 128.7, 146.2. IR: 3063, 3029, 2936, 2842, 1495, 1454, 1380, 1251, 1138, 1086, 756, 699 cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{O}$: C, 82.06; H, 9.53. Found: C, 82.14; H, 9.64.

2-Furan-2-yl-4-phenyl-tetrahydropyran (16b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 1.79-1.96 (m, 2 H), 2.05 (q, $J = 11.2$ Hz, 1 H), 2.12 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.92 (tt, $J = 12.0$ and 4.0 Hz, 1 H), 3.77 (dt, $J = 11.6$ and 2.8 Hz, 1 H), 4.24 (ddd, $J = 11.6$, 4.4 and 1.6 Hz, 1 H), 4.56 (dd, $J = 11.6$ and 2.0 Hz, 1 H), 6.29 (d, $J = 3.6$ Hz, 1 H), 6.34 (dd, $J = 3.2$ and 2.0 Hz, 1 H), 7.21-7.27 (m, 3 H), 7.31-7.35 (m, 2 H), 7.38 (d, $J = 1.6$ Hz, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.3, 37.3, 41.8, 68.8, 73.3, 106.5, 110.3, 126.7, 127.0, 128.8, 142.3, 145.4, 154.9. IR: 3062, 3029, 2942, 2847, 1627, 1497, 1353, 1252, 1124, 1081, 1013, 807, 739, 700 cm^{-1} . Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2$: C, 78.92; H, 7.06. Found: C, 78.88; H, 7.18.

2-Hex-1-enyl-4-phenyl-tetrahydropyran (17b): Liquid; ^1H NMR (400 MHz, CDCl_3): δ 0.88 (t, $J = 7.2$ Hz, 3 H), 1.26-1.42 (m, 4 H), 1.58 (q, $J = 12.8$ Hz, 1 H), 1.74-1.8 (m, 2 H), 1.83-1.88 (m, 1 H), 2.00-2.10 (m, 2 H), 2.81 (tt, $J = 12.0$ and 3.2 Hz, 1 H), 3.63 (dt, $J = 11.2$ and 3.6 Hz, 1 H), 3.85-3.92 (m, 1 H), 4.15 (ddd, $J = 10.8$, 4.4 and 1.6 Hz, 1 H), 5.49 (dd, $J = 14.4$ and 6.4 Hz, 1 H), 5.71 (dt, $J = 15.6$ and 6.0 Hz, 1 H), 7.19-7.34 (m, 5 H). ^{13}C NMR (100 MHz, CDCl_3): δ 14.0, 22.3, 31.3, 32.1, 33.3, 39.9, 41.8, 68.2, 78.3, 126.4, 126.8, 128.6, 130.8, 132.2, 145.8. IR: 3062, 3028, 2930, 2853, 1603, 1496, 1454, 1377, 1252, 1125, 1084, 1035, 969, 756, 699 cm^{-1} . Anal. Calcd for $\text{C}_{17}\text{H}_{24}\text{O}$: C, 83.55; H, 9.90. Found: C, 83.68; H, 10.06.

2-(4-Nitro-phenyl)-4,6-diphenyl tetrahydropyran (18b): Solid, M. P.: 101-103 °C; ¹H NMR (400 MHz, CDCl₃): δ 1.70 (q, *J* = 12.4 Hz, 1 H), 1.80 (q, *J* = 12.8 Hz, 1 H), 2.15-2.18 (m, 2 H), 3.17 (tt, *J* = 12.0 and 3.6 Hz, 1 H), 4.73 (dd, *J* = 11.2 and 2.0 Hz, 1 H), 4.80 (dd, *J* = 11.2 and 2.0 Hz, 1 H), 7.18-7.39 (m, 8 H), 7.46 (d, *J* = 8.8 Hz, 2 H), 7.59 (d, *J* = 8.4 Hz, 2 H), 8.16 (d, *J* = 8.8 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃): δ 41.0, 41.2, 42.3, 78.9, 80.1, 123.7, 125.9, 126.3, 126.6, 126.9, 127.7, 128.6, 128.8, 142.5, 144.6, 147.2, 150.3. IR: 3062, 3029, 2941, 2849, 1602, 1516, 1495, 1345, 1287, 1102, 1078, 1030, 987, 851, 748 cm⁻¹. Anal. Calcd for C₂₃H₂₁NO₃: C, 76.86; H, 5.89; N, 3.90 Found: C, 76.92; H, 5.78; N, 3.88.

2-Hexyl-6-(4-nitro-phenyl)-4-phenyl tetrahydropyran (19b): Semisolid; ¹H NMR (400 MHz, CDCl₃): δ 0.89 (t, *J* = 7.2 Hz, 3 H), 1.26-1.39 (m, 8 H), 1.41-1.64 (m, 3 H), 1.66-1.75 (m, 1 H), 1.90-1.95 (m, 1 H), 2.08-2.13 (m, 1 H), 2.99 (tt, *J* = 12.4 and 4.0 Hz, 1 H), 3.63-3.69 (m, 1 H), 4.62 (dd, *J* = 11.2 and 2.0 Hz, 1 H), 7.21-7.34 (m, 5 H), 7.56 (d, *J* = 8.4 Hz, 2 H), 8.19 (d, *J* = 8.8 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃): δ 14.2, 22.8, 25.6, 29.5, 31.9, 36.4, 38.7, 41.3, 42.1, 78.2 (2C), 123.6, 126.5, 126.6, 126.8, 128.7, 145.2, 147.1, 150.7. IR: 3063, 3029, 2930, 2856, 1602, 1519, 1454, 1346, 1132, 1083, 1014, 853, 748, 698 cm⁻¹. Anal. Calcd for C₂₃H₂₉NO₃: C, 75.17; H, 7.95; N, 3.81 Found: C, 75.24; H, 7.88; N, 3.90.

2-Cyclohexyl-6-(4-nitro-phenyl)-4-phenyl-tetrahydropyran (20b): Semisolid; ¹H NMR (400 MHz, CDCl₃): δ 1.05-1.32 (m, 5 H), 1.48-1.62 (m, 4 H), 1.67-1.80 (m, 3 H), 1.90-1.94 (m, 1 H), 1.98-2.00 (m, 1 H), 2.10-2.14 (m, 1 H), 2.97 (tt, *J* = 12.0 and 4.0 Hz, 1 H), 3.42 (ddd, *J* = 11.2, 6.4 and 2.0 Hz, 1 H), 4.61 (dd, *J* = 11.2 and 2.0 Hz, 1 H), 7.22-7.26 (m, 3 H), 7.30-7.33 (m, 2 H), 7.56 (d, *J* = 8.4 Hz, 2 H), 8.19 (d, *J* = 8.4 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃): δ 26.3, 26.4, 26.8, 28.9, 29.1, 35.6, 41.3, 42.2, 43.2, 78.2, 82.3, 123.6, 126.4, 126.6, 126.9, 128.7, 145.4, 147.0, 150.9. IR: 2927, 2849, 1600, 1523, 1507, 1449, 1346, 1103, 1079, 852, 747, 696 cm⁻¹. Anal. Calcd for C₂₃H₂₇NO₃: C, 75.59; H, 7.45; N, 3.83 Found: C, 75.66; H, 7.57; N, 3.87.

4-Phenyl-2,6-di(4-nitrophenyl)-tetrahydropyran (21b): Solid, M.P.: 224-225 °C; ¹H NMR (400 MHz, CDCl₃): δ 1.78 (q, *J* = 12.8 Hz, 2 H), 2.23-2.26 (m, 2 H), 3.25 (tt, *J* =

12.4, and 3.6 Hz, 1 H), 4.89 (dd, $J=11.2$ and 1.6 Hz, 2 H), 7.24-7.35 (m, 5 H), 7.65 (d, $J=8.8$ Hz, 4 H), 8.24 (d, $J=8.8$ Hz, 4 H); ^{13}C NMR (100 MHz, CDCl_3): δ 41.0, 42.3, 79.2, 123.9, 126.6, 126.9, 127.2, 129.0, 144.0, 147.5, 149.6; IR: 3085, 2915, 2846, 1600, 1512, 1344, 1103, 1091, 850 cm^{-1} . Anal. Calcd for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_5$: C, 68.31; H, 4.98; N, 6.93. Found: C, 68.39; H, 5.10; N, 6.87.

2-(3-Nitro-phenyl)-4-*o*-tolyl tetrahydropyran and 2-(3-Nitro-phenyl)-4-*p*-tolyl tetrahydropyran (22; two *ortho/para* regioisomers: 3:3.4): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.64-1.96 (m, 3 H), 2.04 (dq, $J=13.6$ and 2.0 Hz, 0.54 H), 2.10 (dq, $J=13.2$ and 2.0 Hz, 0.46 H), 2.32 (s, 1.4 H), 2.41 (s, 1.6 H), 2.95 (tt, $J=12.0$ and 4.0 Hz, 0.54 H), 3.22 (tt, $J=11.6$ and 4.0 Hz, 0.46 H), 3.74-3.85 (m, 1 H), 4.28-4.35 (m, 1 H), 4.56-4.63 (m, 1 H), 7.03-7.24 (m, 4 H), 7.47-7.51 (m, 1 H), 7.69-7.72 (m, 1 H), 8.10-8.13 (m, 1 H), 8.28 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 19.4, 21.0, 32.5, 33.1, 33.2, 37.6, 40.7, 41.5, 41.6, 41.8, 68.7, 68.9, 78.5, 78.7, 120.8, 122.3, 123.8, 125.5, 126.3, 126.5, 126.6, 127.4, 127.5, 128.5, 129.2, 129.3, 130.6, 131.9, 135.1, 136.1, 138.0, 142.8, 145.0, 148.3. IR: 3020, 2919, 2848, 1605, 1529, 1349, 1130, 1087, 1042, 810, 737 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_3$: C, 72.71; H, 6.44; N, 4.71. Found: C, 72.85; H, 6.59; N, 4.66.

4-(2,3-Dimethyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran and 4-(3,4-dimethyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran (23; two regioisomers 1:1.2): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.64-1.74 (m, 1 H), 1.78-1.90 (m, 2 H), 2.02 (dq, $J=13.2$ and 2.0 Hz, 0.54 H), 2.08 (dq, $J=13.2$ and 2.0 Hz, 0.46 H), 2.23 (s, 1.4 H), 2.25 (s, 1.6 H), 2.30 (s, 3 H), 2.93 (tt, $J=12.0$ and 4.0 Hz, 0.54 H), 3.29 (tt, $J=12.4$ and 4.0 Hz, 0.46 H), 3.76 (dt, $J=11.6$ and 3.2 Hz, 0.46 H), 3.82 (dt, $J=12.0$ and 2.8 Hz, 0.54 H), 4.29-4.35 (m, 1 H), 4.57 (dd, $J=11.2$ and 2.0 Hz, 0.46 H), 4.62 (dd, $J=11.2$ and 2.0 Hz, 0.54 H), 6.96-7.10 (m, 3 H), 7.47-7.52 (m, 1 H), 7.70-7.72 (m, 1 H), 8.10-8.13 (m, 1 H), 8.28 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 14.8, 19.4, 19.9, 21.2, 32.8, 33.4, 38.0, 41.1, 41.5, 41.8, 68.7, 68.9, 78.6, 78.8, 120.6, 122.0, 123.0, 123.9, 125.7, 127.9, 129.0, 129.7, 131.7, 133.5, 134.4, 136.6, 136.8, 142.4, 142.5, 145.0, 145.1, 148.1. IR: 2941,

2848, 1633, 1529, 1442, 1350, 1130, 1090, 1041, 807, 737, 693 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.45; H, 6.94; N, 4.56.

4-(2,4-Dimethyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran and 4-(2,6-dimethyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran (24; two regioisomers: 2:1): Solid; Mixed M.P.: 75-82 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.67-1.80 (m, 1 H), 1.82-1.90 (m, 1 H), 1.94 (dq, $J = 13.2$ and 2.0 Hz, 0.46 H), 2.00 (dq, $J = 13.2$ and 2.0 Hz, 0.54 H), 2.14-2.23 (m, 1 H), 2.28 (s, 3 H), 2.37 (s, 1.98 H), 2.45 (s, 1.02 H), 3.18 (tt, $J = 12.0$ and 3.6 Hz, 0.66 H), 3.50 (tt, $J = 12.4$ and 3.6 Hz, 0.34 H), 3.74-3.84 (m, 1 H), 4.30-4.36 (m, 1 H), 4.56-4.62 (m, 1 H), 7.00 (d, $J = 5.2$ Hz, 2 H), 7.10 (d, $J = 7.6$ Hz, 1 H), 7.48-7.52 (m, 1 H), 7.71 (d, $J = 7.6$ Hz, 1 H), 8.10-8.13 (m, 1 H), 8.28 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 19.1, 20.7, 21.2, 21.7, 29.3, 32.5, 33.0, 37.5, 37.7, 38.7, 40.7, 41.5, 41.6, 68.6, 69.2, 78.5, 79.0, 120.5, 120.6, 122.0, 124.5, 125.2, 126.1, 126.9, 129.0, 131.2, 131.6, 131.7, 134.7, 135.4, 136.0, 137.8, 139.7, 139.8, 145.1, 148.1. IR: 2941, 2848, 1633, 1529, 1442, 1130, 1090, 1041, 807, 737, 692 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.35; H, 6.88; N, 4.61.

4-(2,5-Dimethyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran (25): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.73 (q, $J = 12.8$ Hz, 1 H), 1.74-1.81 (m, 1 H), 1.84-1.95 (m, 1 H), 2.01 (dq, $J = 12.4$ and 2.0 Hz, 1 H), 2.29 (s, 3 H), 2.36 (s, 3 H), 3.19 (tt, $J = 12.0$ and 3.6 Hz, 1 H), 3.80 (dt, $J = 12.0$ and 2.4 Hz, 1 H), 4.32 (ddd, $J = 11.6$, 4.4 and 1.6 Hz, 1 H), 4.60 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 6.92-6.94 (m, 1 H), 7.02-7.10 (m, 2 H), 7.50 (t, $J = 8.0$ Hz, 1 H), 7.71 (d, $J = 7.6$ Hz, 1 H), 8.10-8.13 (m, 1 H), 8.30 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 19.0, 21.2, 32.6, 37.6, 40.8, 68.9, 78.8, 120.9, 122.3, 126.3, 127.0, 129.3, 130.5, 131.9, 132.0, 135.8, 142.6, 145.2, 148.3. IR: 3077, 3017, 2945, 2848, 1614, 1528, 1441, 1348, 1131, 1082, 1043, 808, 739, 695 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.55; H, 6.89; N, 4.65.

4-(2-Methoxy-phenyl)-2-(3-nitro-phenyl) tetrahydro-pyran (26o): Solid: M. P. 79-83 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.67 (q, $J = 12.0$ Hz, 1 H), 1.80-1.95 (m, 2 H), 2.10 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 3.45 (tt, $J = 12.0$ and 3.6 Hz, 1 H), 3.77-3.82 (m, 1 H), 3.86 (s, 3 H), 4.31 (ddd, $J = 11.6$, 4.4 and 1.6 Hz, 1 H), 4.63 (dd, $J = 11.2$ and

2.0 Hz, 1 H), 6.86-7.00 (m, 2 H), 7.16-7.23 (m, 2 H), 7.50 (t, $J = 8.0$ Hz, 1 H), 7.71 (d, $J = 7.6$ Hz, 1 H), 8.10 (d, $J = 8.4$ Hz, 1 H), 8.28 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 31.8, 34.8, 40.4, 55.5, 69.1, 78.9, 110.6, 120.9, 121.1, 122.5, 126.7, 127.5, 129.4, 132.2, 133.1, 145.4, 148.4, 156.9. IR: 2939, 2841, 1600, 1528, 1493, 1349, 1240, 1132, 1083, 1030, 846, 810, 753 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_4$: C, 68.99; H, 6.11; N, 4.47. Found: C, 68.95; H, 6.14; N, 4.52.

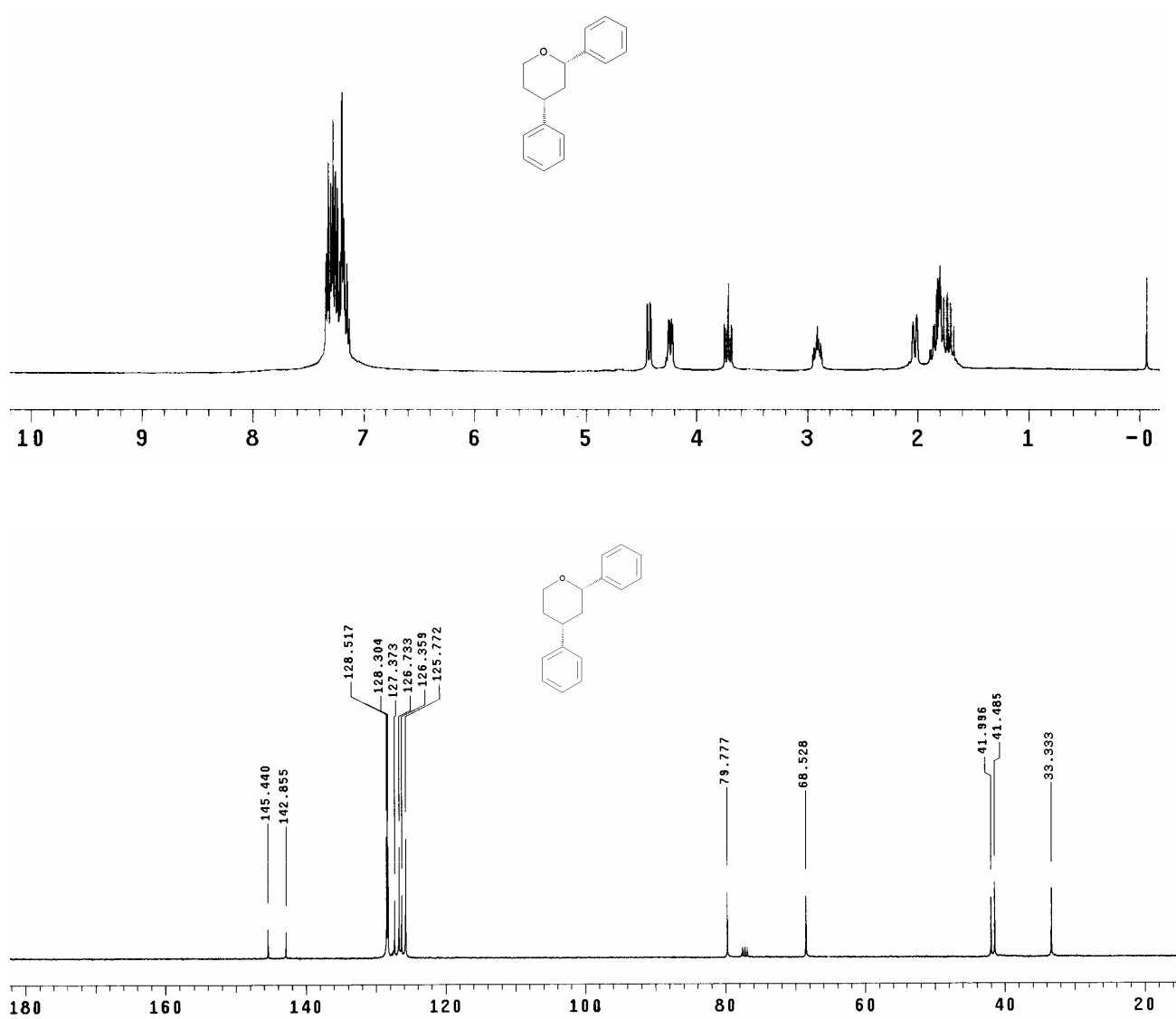
4-(4-Methoxy-phenyl)-2-(3-nitro-phenyl) tetrahydro-pyran (26p): Gum; ^1H NMR (400 MHz, CDCl_3): δ 1.65 (q, $J = 12.0$ Hz, 1 H), 1.82-1.91 (m, 2 H), 2.09 (dq, $J = 13.2$ and 2.0 Hz, 1 H), 2.93 (tt, $J = 12.0$ and 3.6 Hz, 1 H), 3.71-3.80 (m, 1 H), 3.77 (s, 3 H), 4.31 (dt, $J = 10.8$, and 1.2 Hz, 1 H), 4.56 (dd, $J = 10.8$ and 1.6 Hz, 1 H), 6.85 (d, $J = 8.8$ Hz, 2 H), 7.15 (d, $J = 8.8$ Hz, 2 H), 7.48 (t, $J = 8.0$ Hz, 1 H), 7.69 (d, $J = 7.6$ Hz, 1 H), 8.10 (dd, $J = 8.4$ and 1.2 Hz, 1 H), 8.27 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 33.4, 41.1, 41.9, 55.3, 68.7, 78.6, 114.1, 120.9, 122.4, 127.7, 129.4, 132.0, 137.2, 145.2, 148.3, 158.3. IR: 2935, 2847, 1610, 1528, 1463, 1351, 1249, 1180, 1086, 1036, 830, 807, 738 cm^{-1} . Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_4$: C, 68.99; H, 6.11; N, 4.47. Found: C, 69.04; H, 6.08; N, 4.55.

4-(2-Methoxy-5-methyl-phenyl)-2-(3-nitro-phenyl)-tetrahydropyran (27): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.1.69 (q, $J = 11.6$ Hz, 1 H), 1.78-1.98 (m, 2 H), 2.00-2.10 (m, 1 H), 2.33 (s, 3 H), 3.18 (tt, $J = 11.6$ and 4.0 Hz, 1 H), 3.77 (s, 3 H), 3.80-3.86 (m, 1 H), 4.32 (dd, $J = 10.8$ and 3.2 Hz, 1 H), 4.61 (dd, $J = 11.2$ and 2.0 Hz, 1 H), 6.66-6.69 (m, 1 H), 6.76 (d, $J = 2.0$ Hz, 1 H), 7.00 (d, $J = 8.9$ Hz, 1 H), 7.50 (t, $J = 8.0$, 1 H), 7.71 (d, $J = 7.6$ Hz, 1 H), 8.12 (d, $J = 8.0$ Hz, 1 H) 8.28 (s, 1 H). ^{13}C NMR (100 MHz, CDCl_3): δ 18.7, 32.5, 38.0, 40.8, 55.4, 69.0, 78.9, 111.1, 112.1, 121.1, 122.5, 127.2, 129.4, 131.5, 132.0, 144.2, 145.1, 148.5, 158.5. IR: 3086, 2926, 2851, 1610, 1529, 1463, 1251, 1109, 1085, 1041, 806, 738 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_4$: C, 69.71; H, 6.47 N, 4.28. Found: C, 69.85; H, 6.55; N, 4.33.

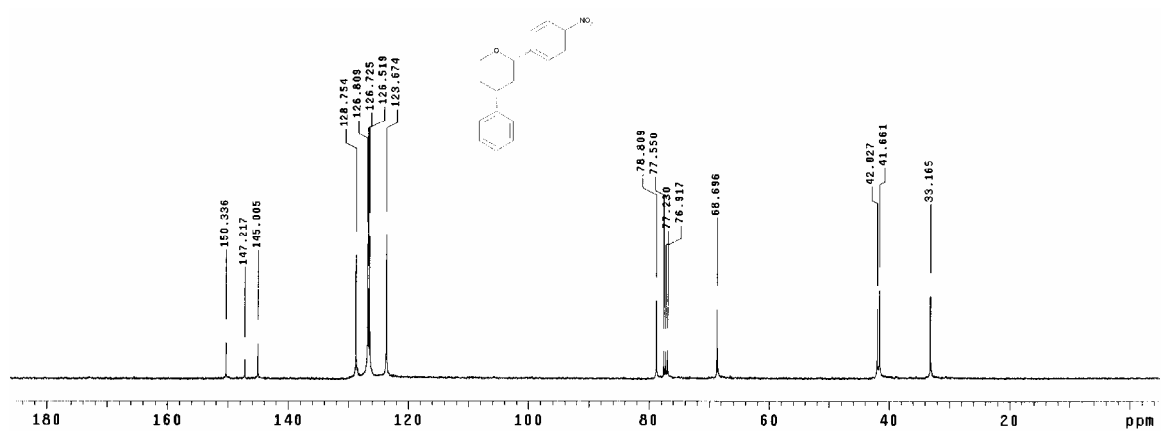
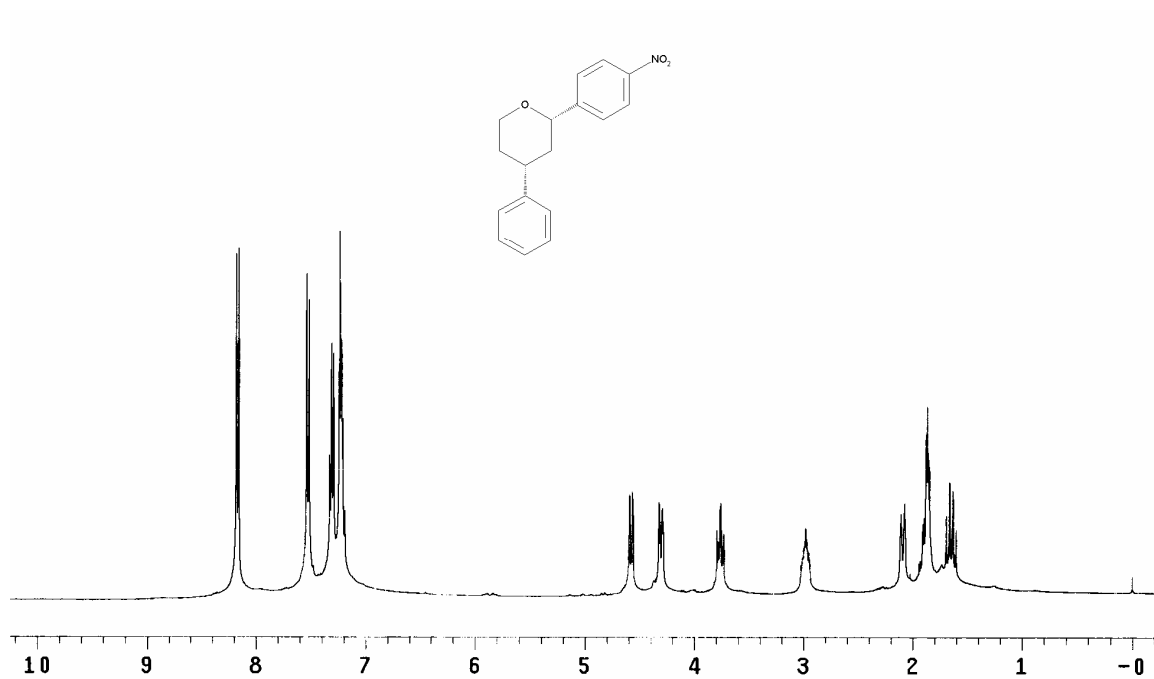
4-Phenyl-1-oxa-spiro[5.5]undecane (28): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.26-1.80 (m, 14 H), 2.10-2.14 (m, 1 H), 2.90-3.00 (m, 1 H), 3.73-3.85 (m, 1 H), 7.18-7.23 (m, 3 H), 7.28-7.33 (m, 2 H). ^{13}C NMR (100 MHz, CDCl_3): δ 21.6, 21.8, 26.3, 29.7, 33.7, 36.7, 40.5, 43.6, 60.7, 72.4, 126.3, 126.8, 128.5, 146. IR: 3027, 2927, 2855, 1603, 1492, 1445, 1375, 1175, 1101, 1079, 974, 910, 759, 698 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{22}\text{O}$: C, 83.43; H, 9.63. Found: C, 83.54; H, 9.54.

4,13-Diphenyl-1,10-dioxadispiro [5.2.5.2] hexadecane (29): Solid; M.P. 195.0-198.0 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.37-1.84 (m, 14 H), 2.23-2.28 (m, 2 H), 2.94-3.04 (m, 2 H), 3.68-3.75 (m, 2 H), 3.81-3.88 (m, 2 H), 7.19-7.23 (m, 6 H), 7.29-7.33 (m, 4 H). ^{13}C NMR (100 MHz, CDCl_3): δ 23.7, 24.1, 33.8, 35.0, 35.4, 36.8, 37.0, 44.7, 61.2, 72.3, 126.4, 127.0, 128.7, 146.3. IR: 3026, 2929, 2853, 1600, 1491, 1452, 1375, 1108, 1084, 996, 757, 698 cm^{-1} . Anal. Calcd for $\text{C}_{26}\text{H}_{32}\text{O}_2$: C, 82.94; H, 8.57. Found: C, 82.87; H, 8.65.

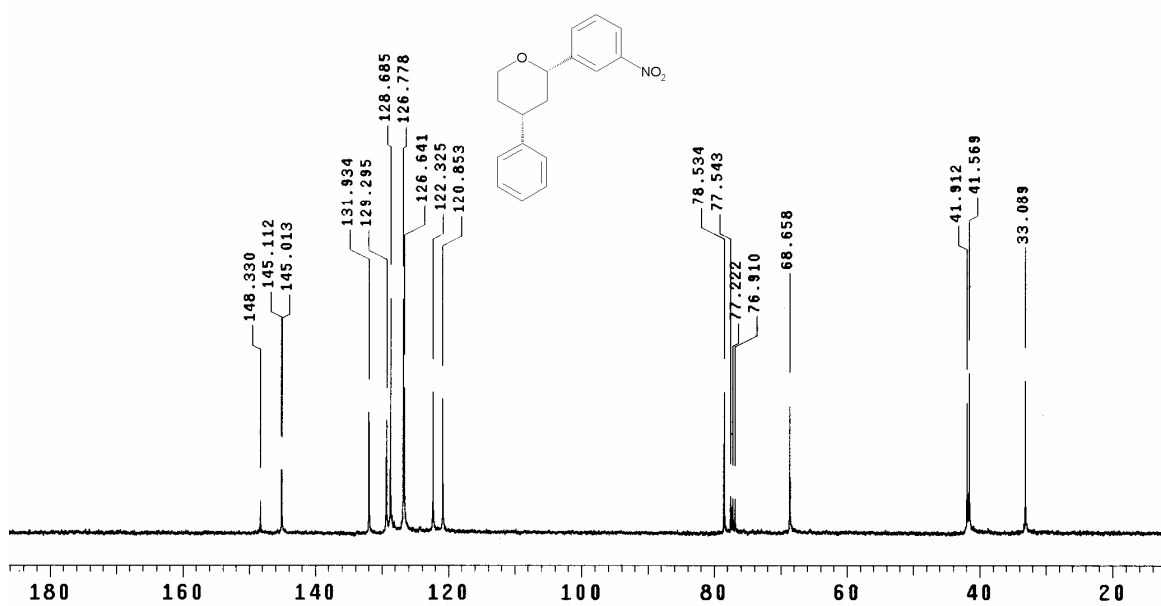
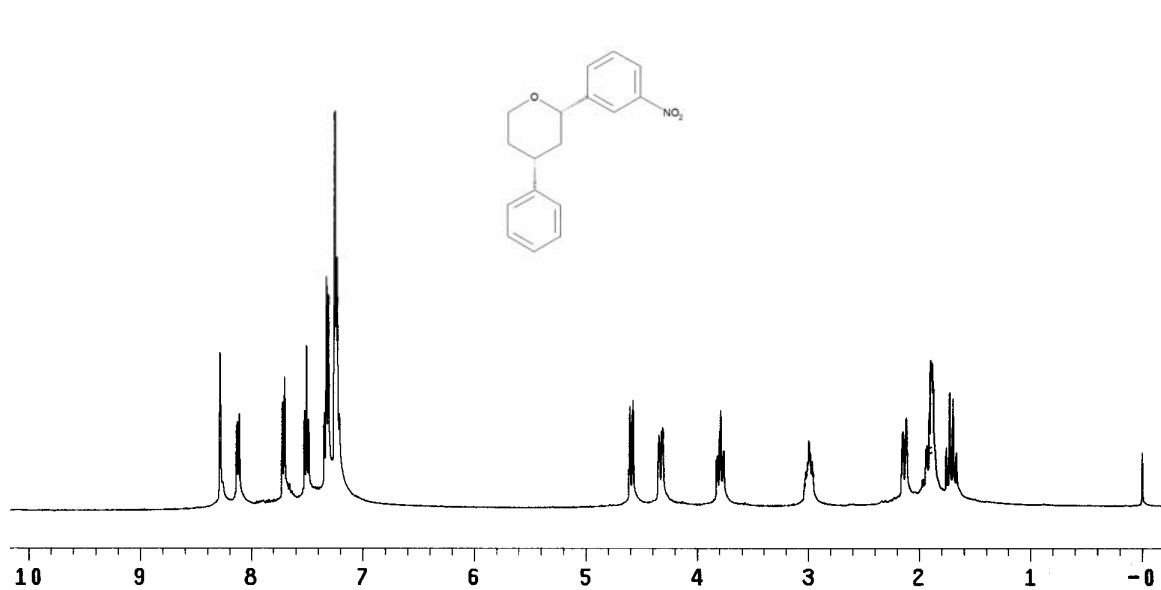
2,2-Bis-chloromethyl-4-phenyl-tetrahydropyran (30): Semisolid; ^1H NMR (400 MHz, CDCl_3): δ 1.62 (t, $J = 13.2$ Hz, 1 H), 1.75-1.82 (m, 2 H), 2.12 (dd, $J = 14.00$ and 3.2 Hz, 1 H), 2.88 (tt, $J = 12.4$ and 4.0 Hz, 1 H), 3.62 (q, $J_{\text{AB}} = 11.2$ Hz, 2 H), 3.60-3.78 (m, 2 H), 3.92 (q, $J_{\text{AB}} = 12.0$ Hz, 2 H), 7.21-7.25 (m, 3 H), 7.31-7.35 (m, 2 H). ^{13}C NMR (100 MHz, CDCl_3): δ 32.5, 36.8, 41.9, 49.8, 62.8, 75.2, 126.8, 126.9, 128.9, 144.5. IR: 2943, 2833, 1602, 1495, 1441, 1257, 1120, 1082, 783, 750, 699 cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{Cl}_2\text{O}$: C, 60.25; H, 6.22. Found: C, 60.32; H, 6.16.



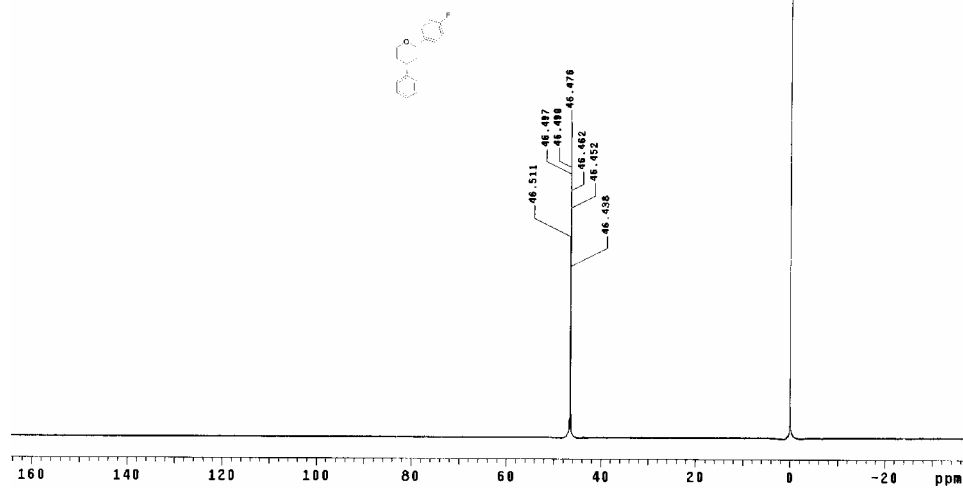
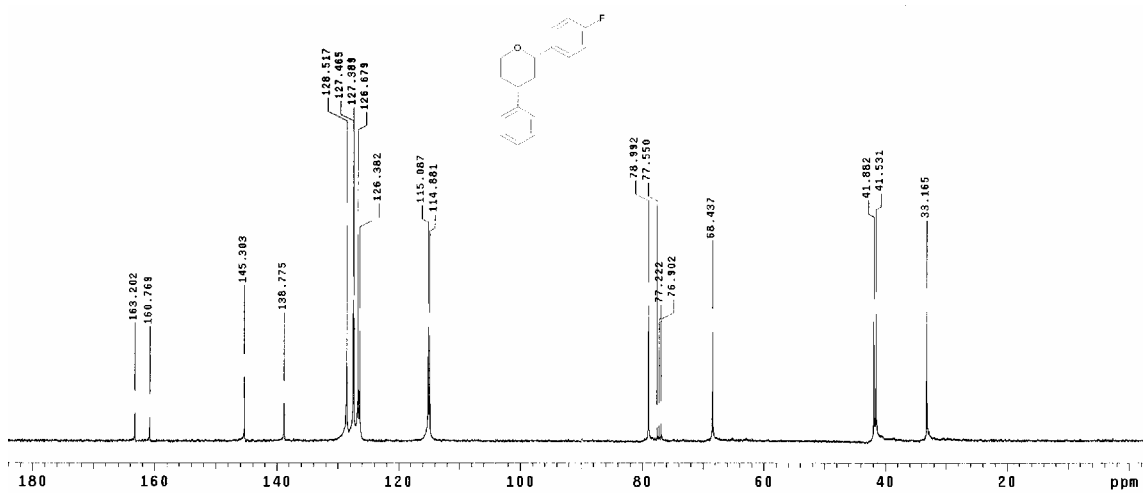
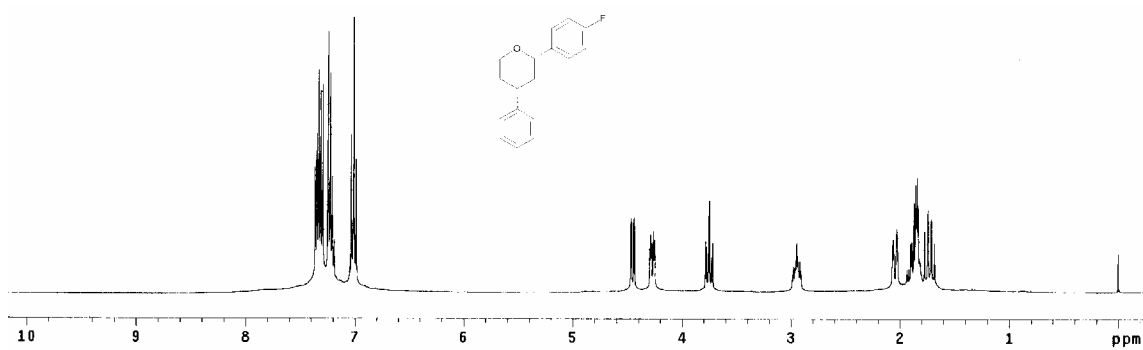
^1H and ^{13}C NMR spectra of compound **1b**



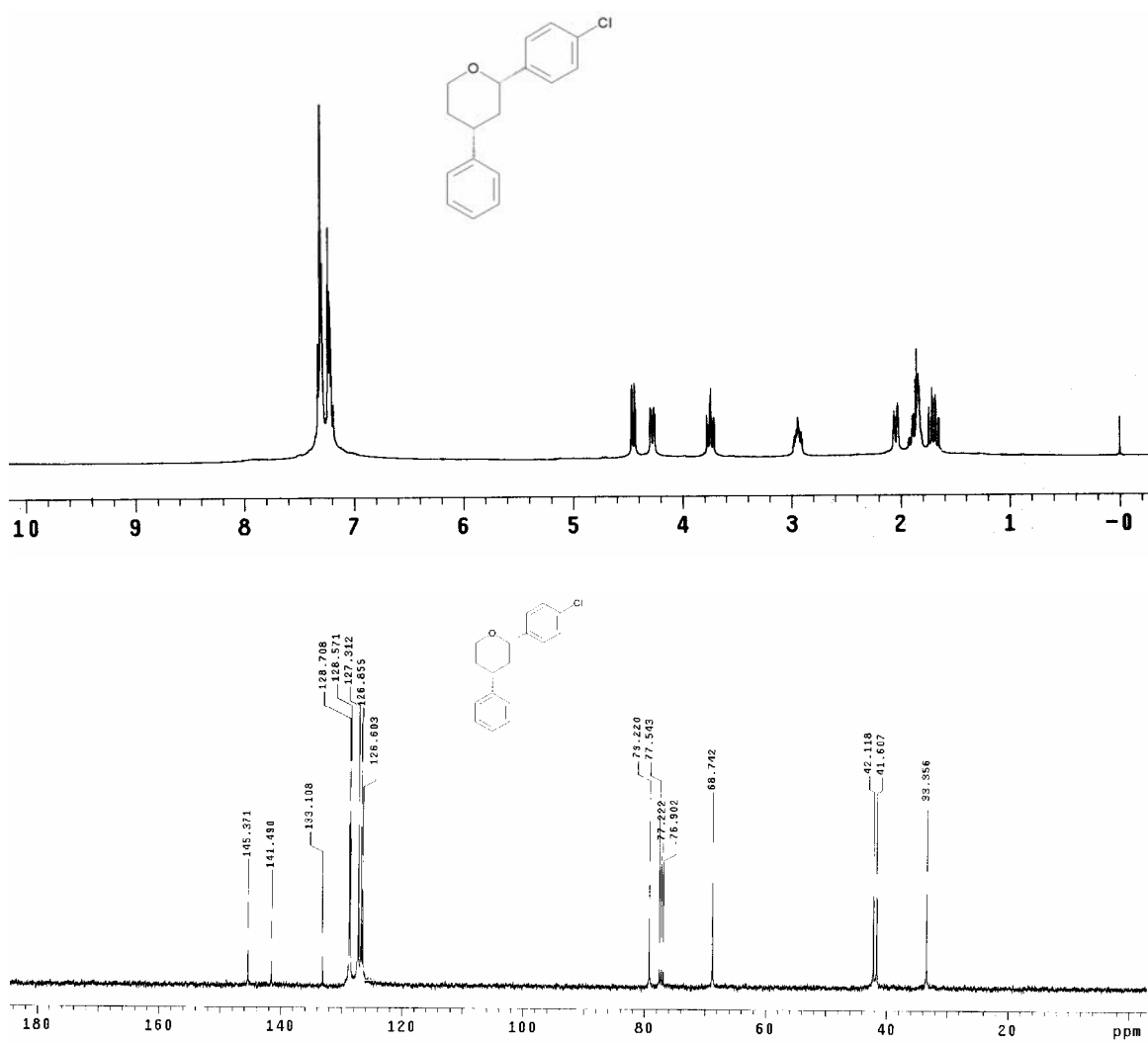
¹H and ¹³C NMR spectra of compound **2b**



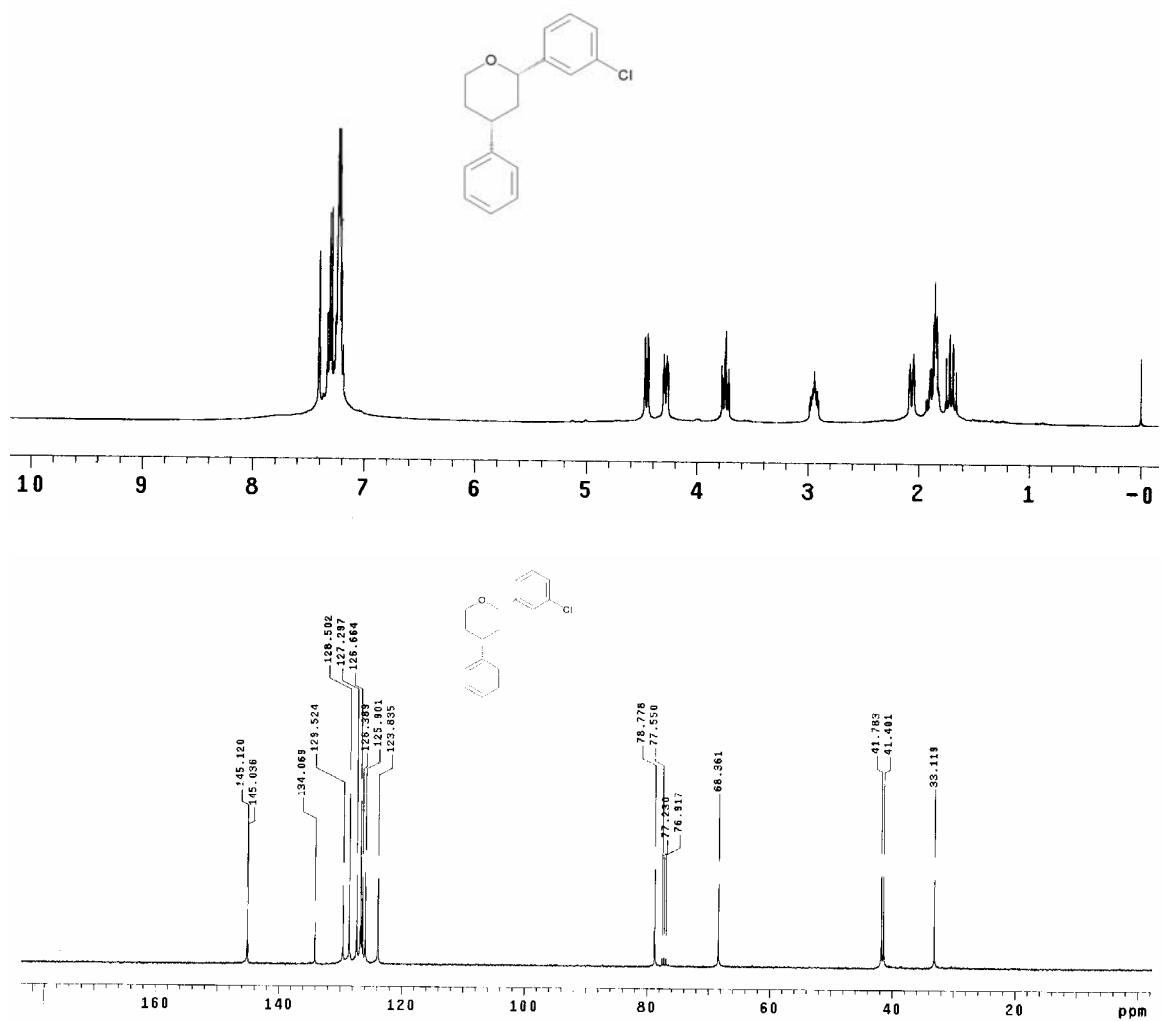
¹H and ¹³C NMR spectra of compound **3b**



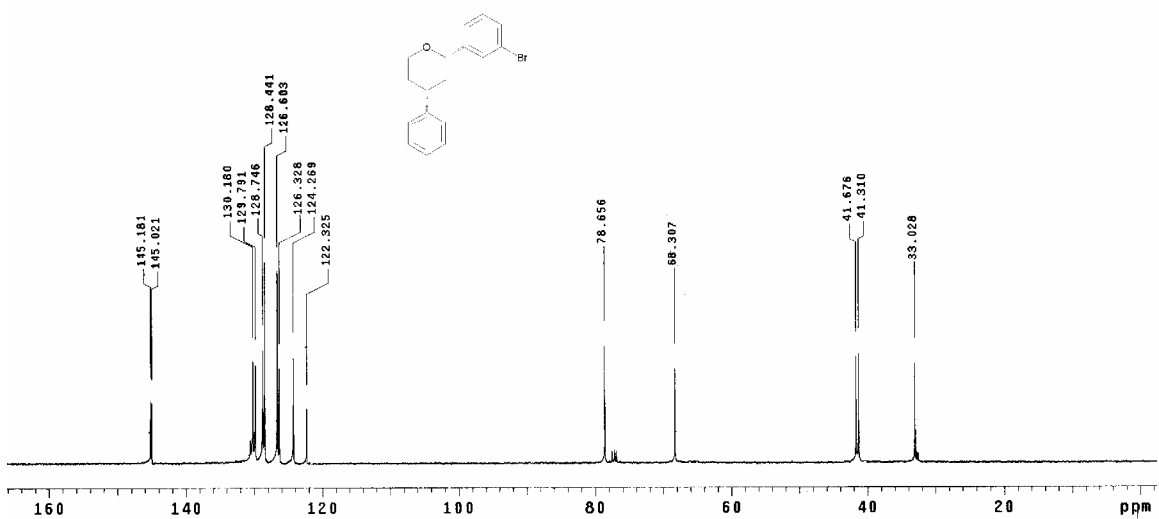
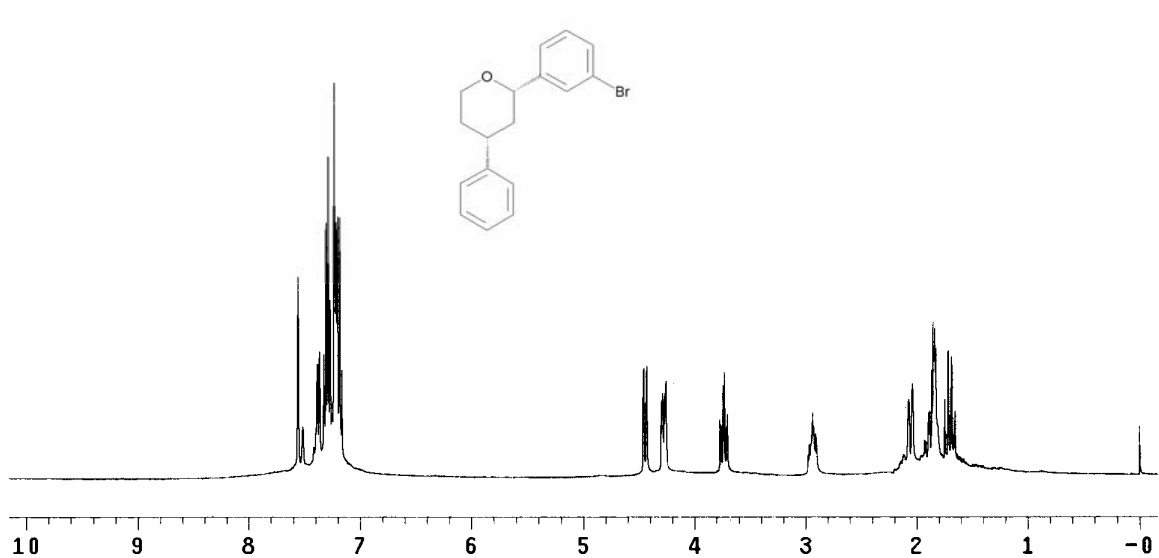
¹H, ¹³C and ¹⁹F NMR spectra of compound **4b**



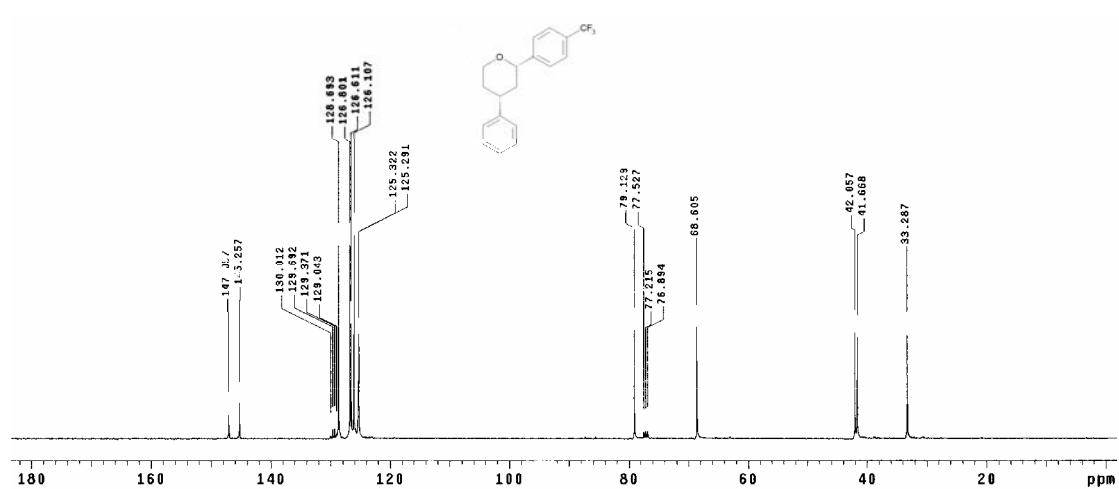
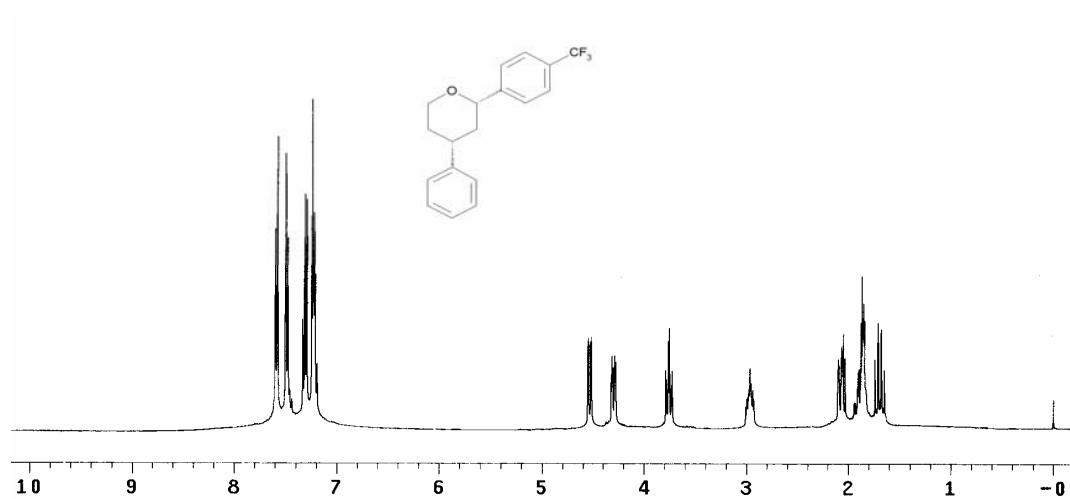
^1H and ^{13}C NMR spectra of compound **5b**

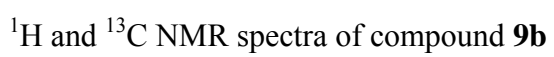


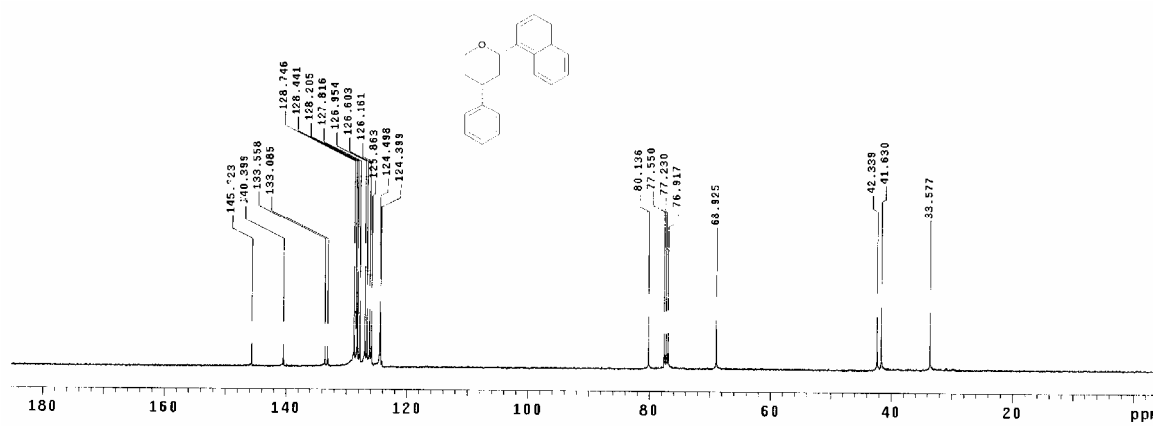
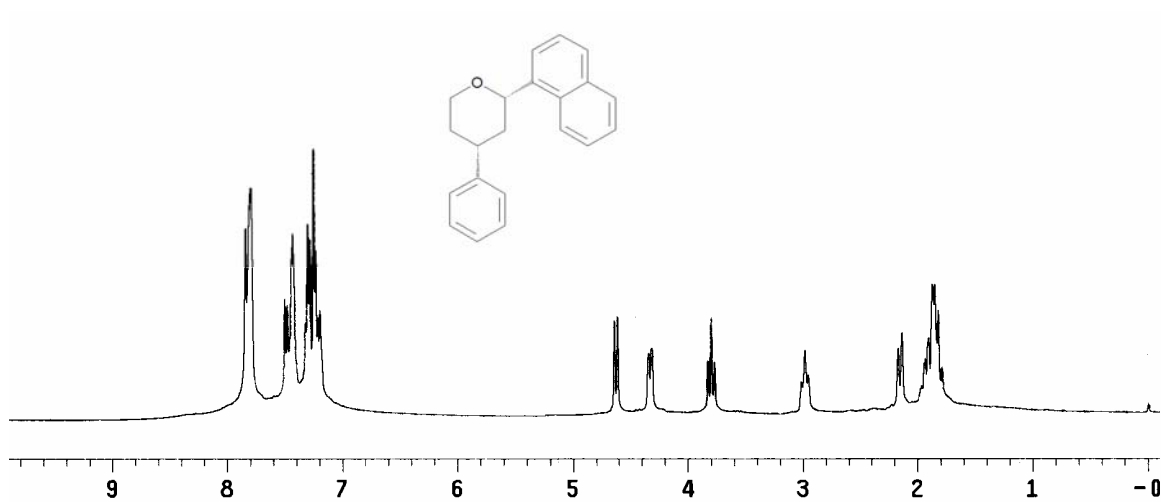
^1H and ^{13}C NMR spectra of compound **6b**



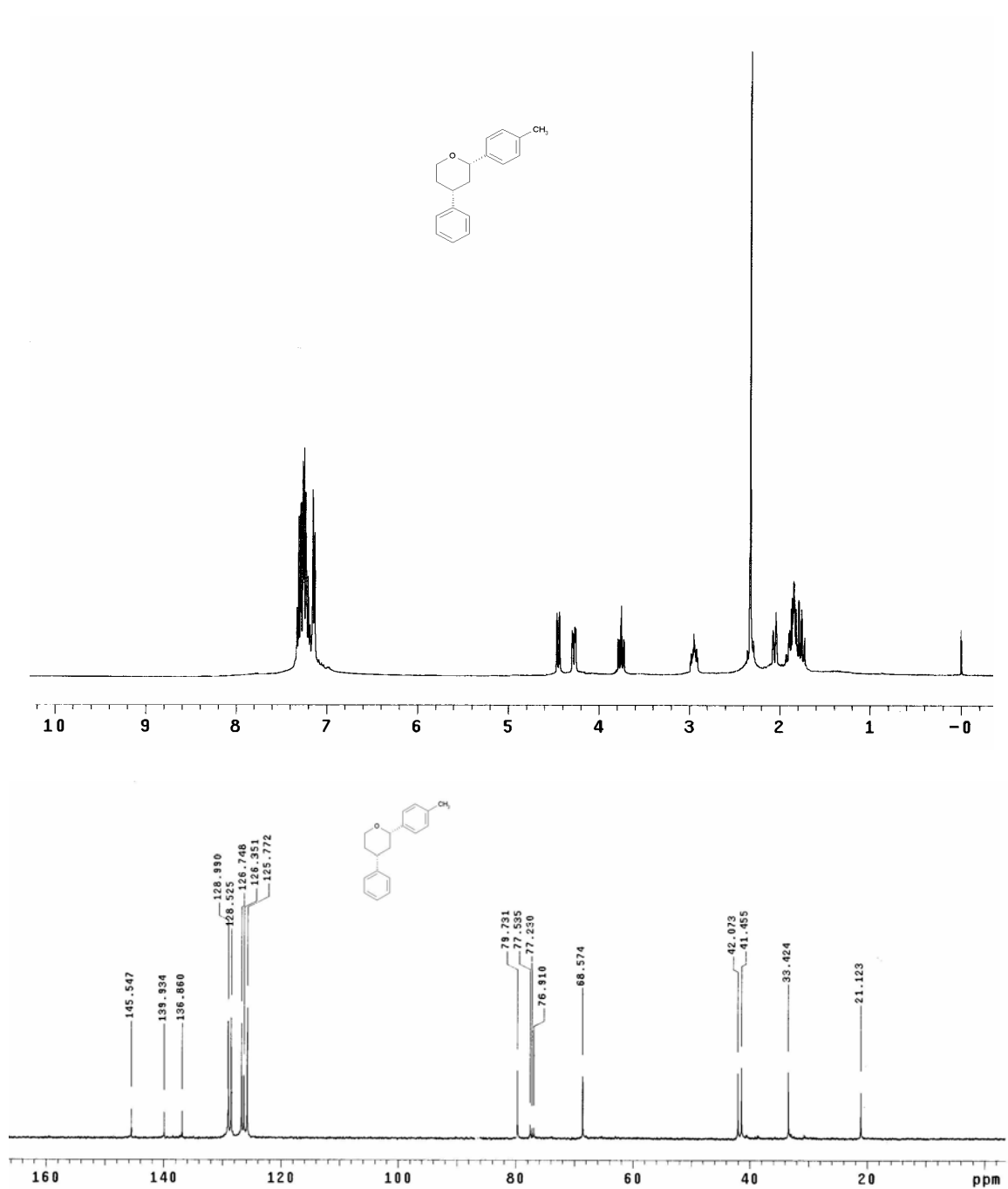
^1H and ^{13}C NMR spectra of compound **7b**



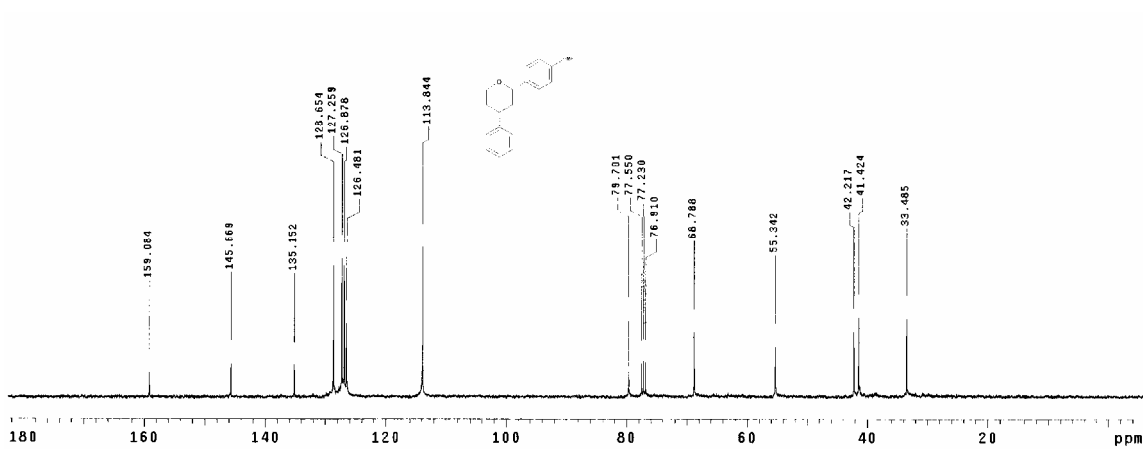
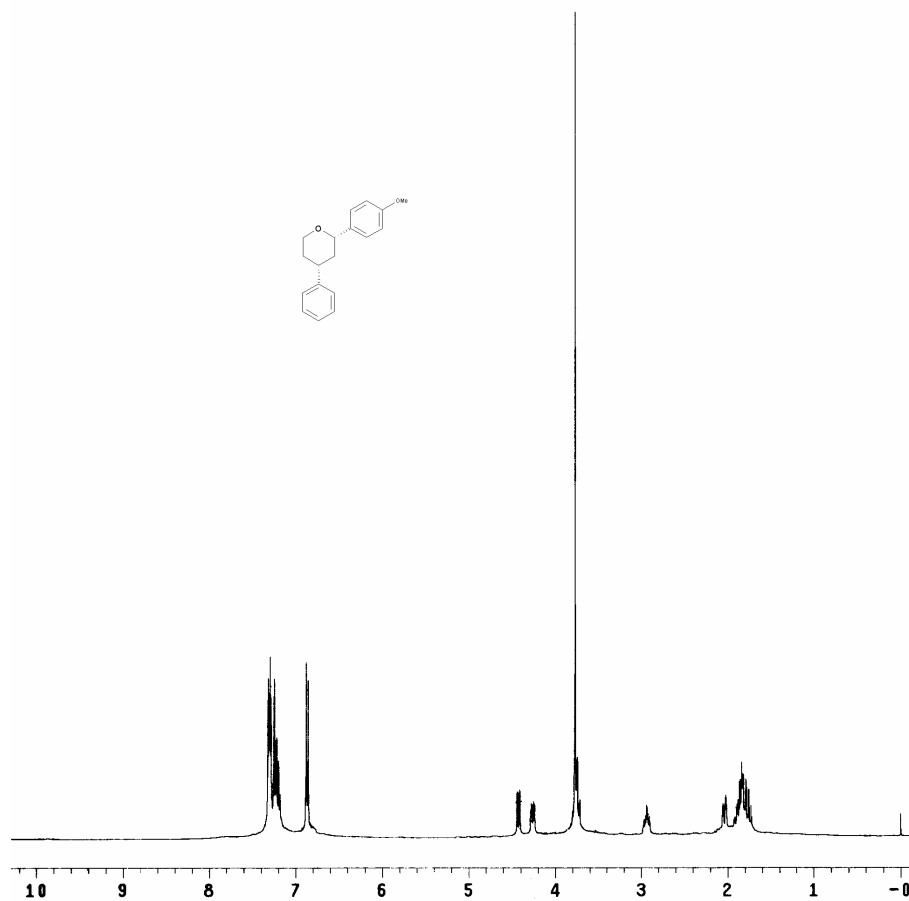




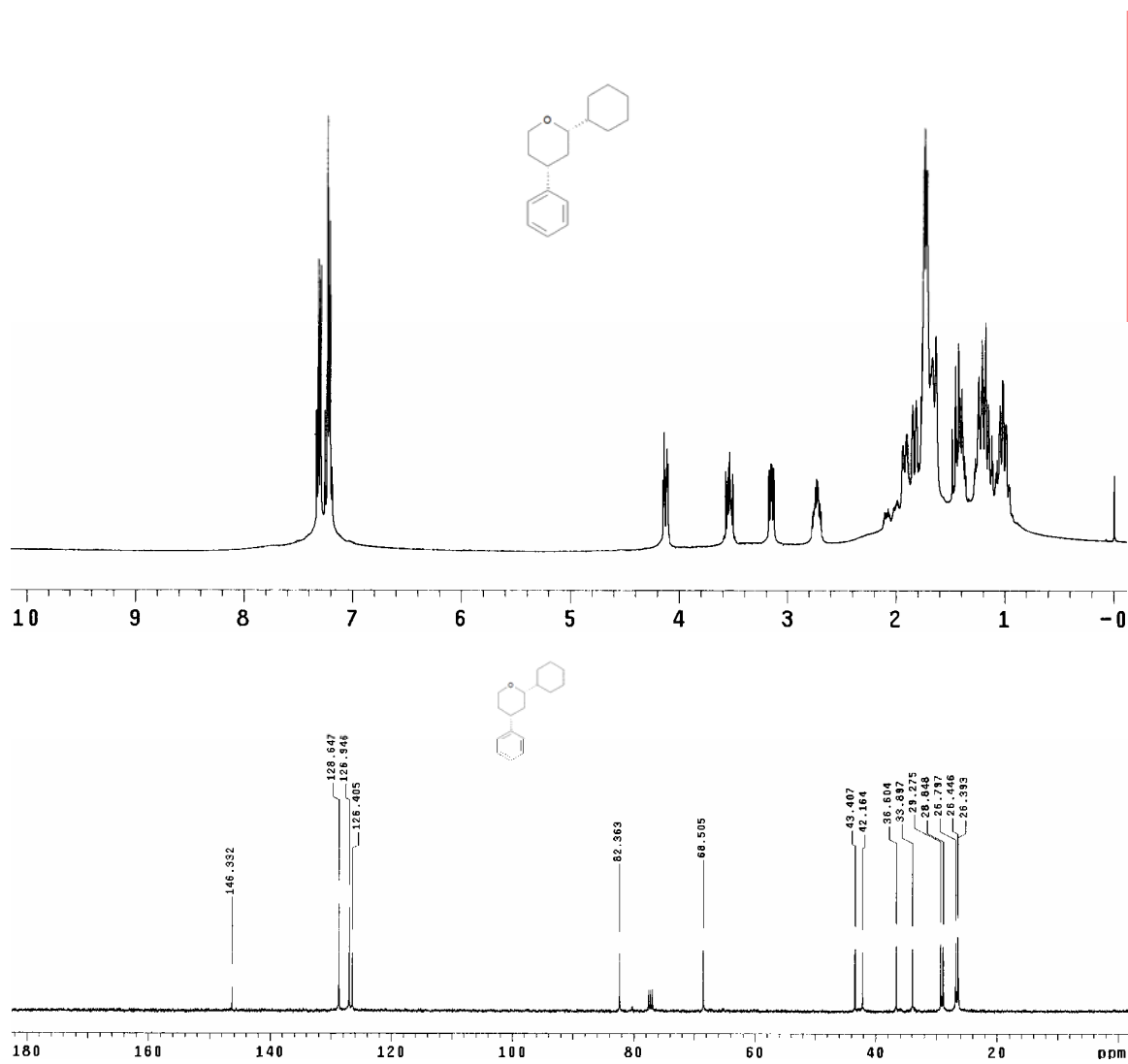
^1H and ^{13}C NMR spectra of compound **10b**



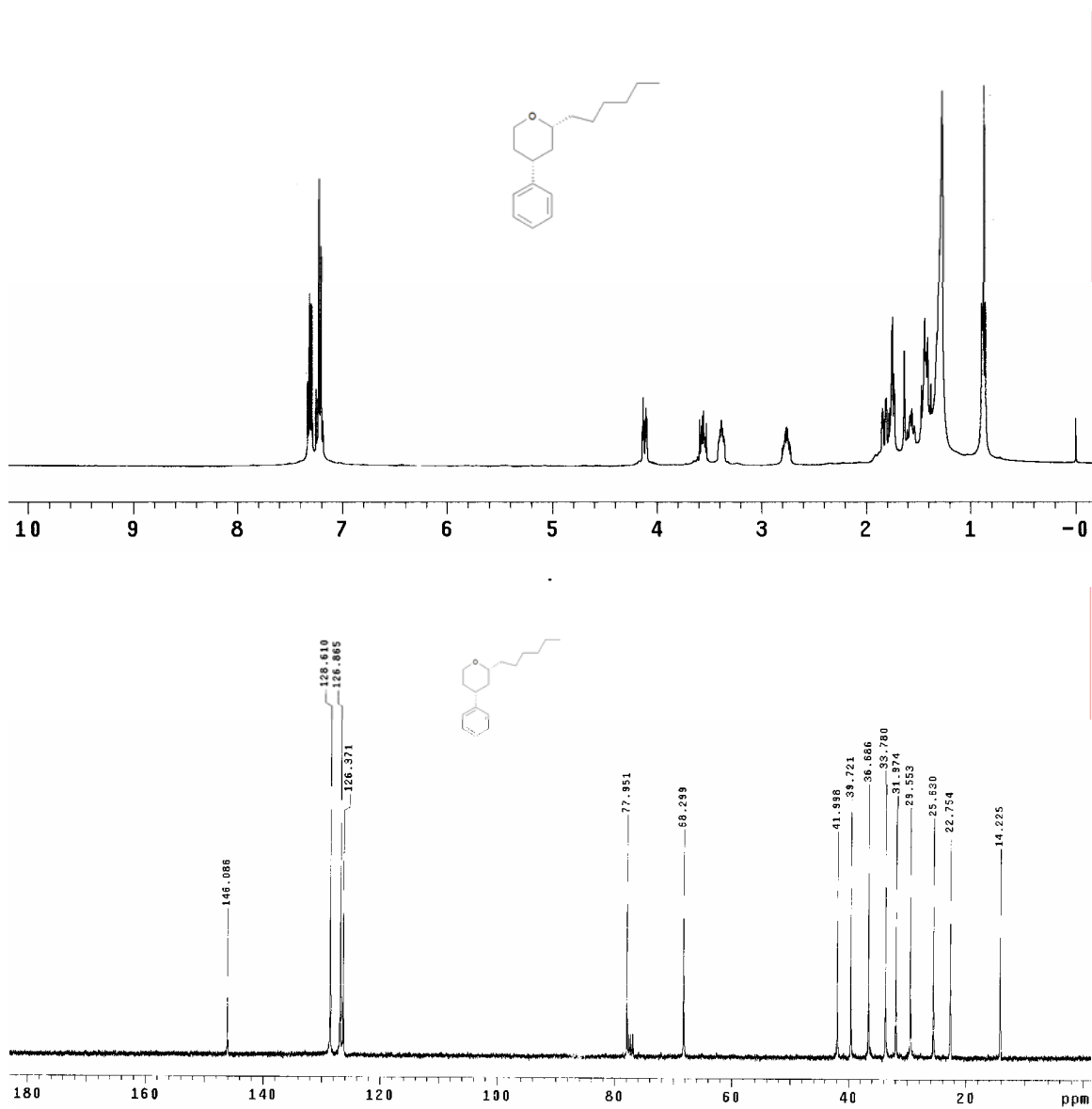
^1H and ^{13}C NMR spectra of compound **11b**



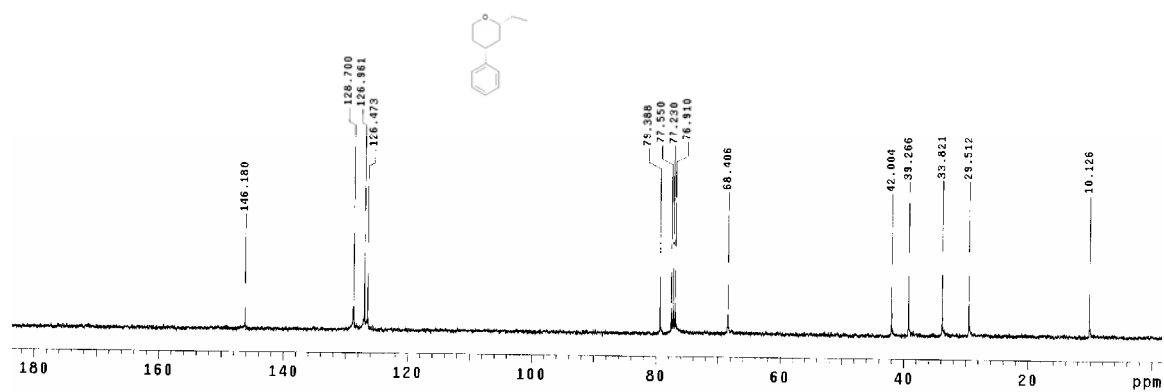
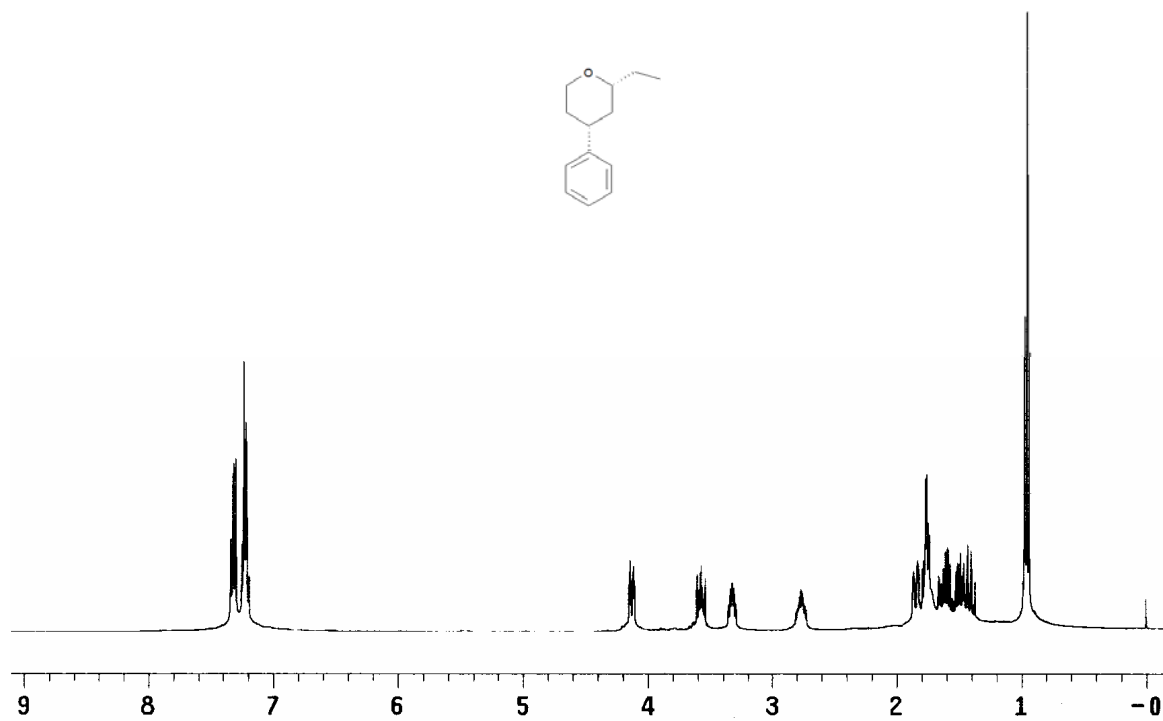
¹H and ¹³C NMR spectra of compound **12b**



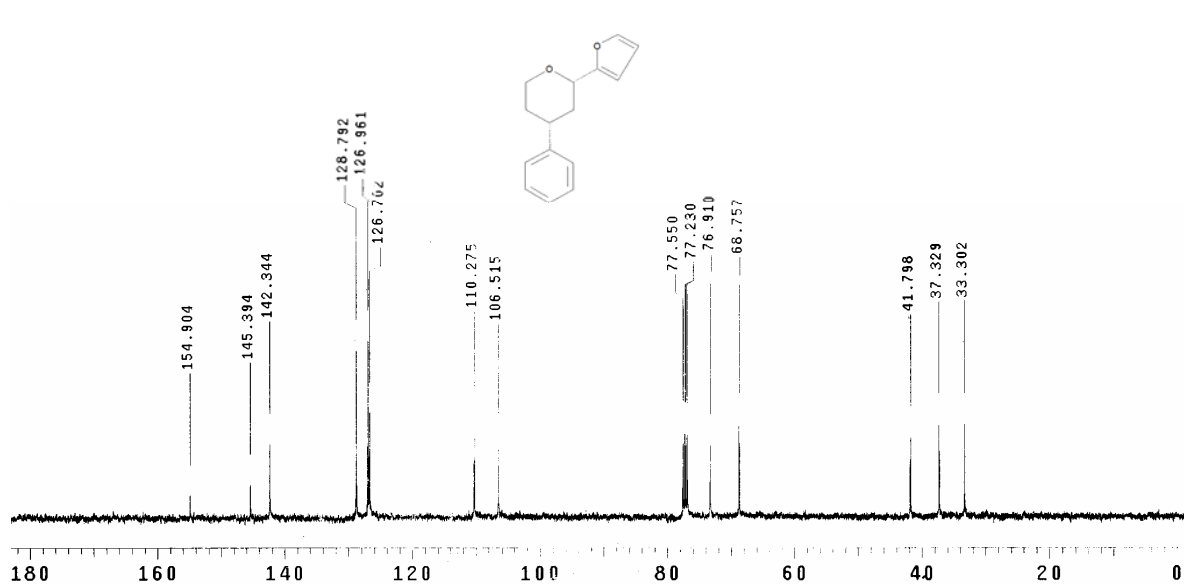
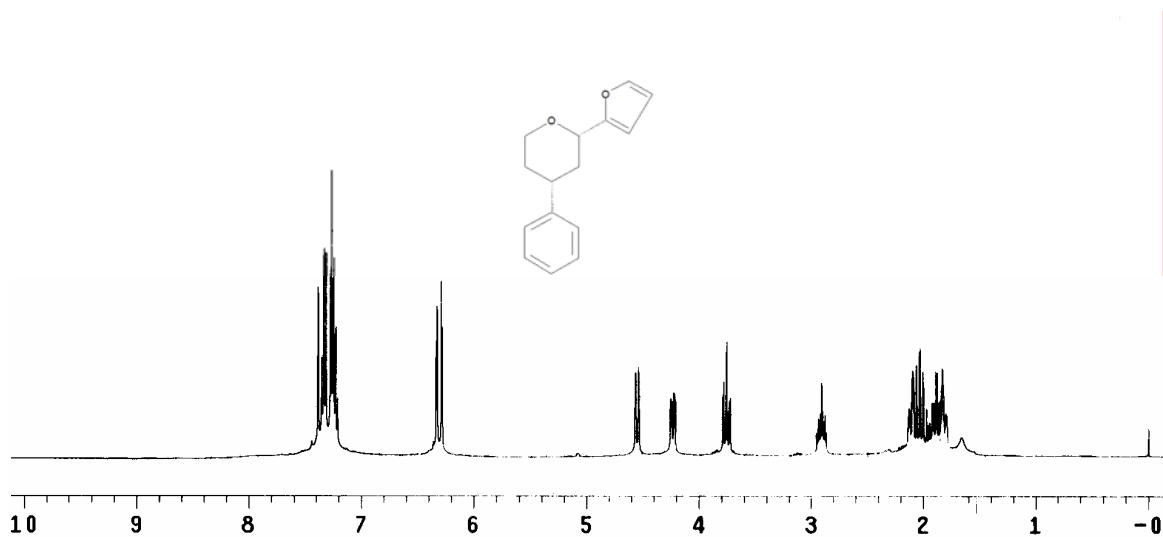
^1H and ^{13}C NMR spectra of compound **13b**



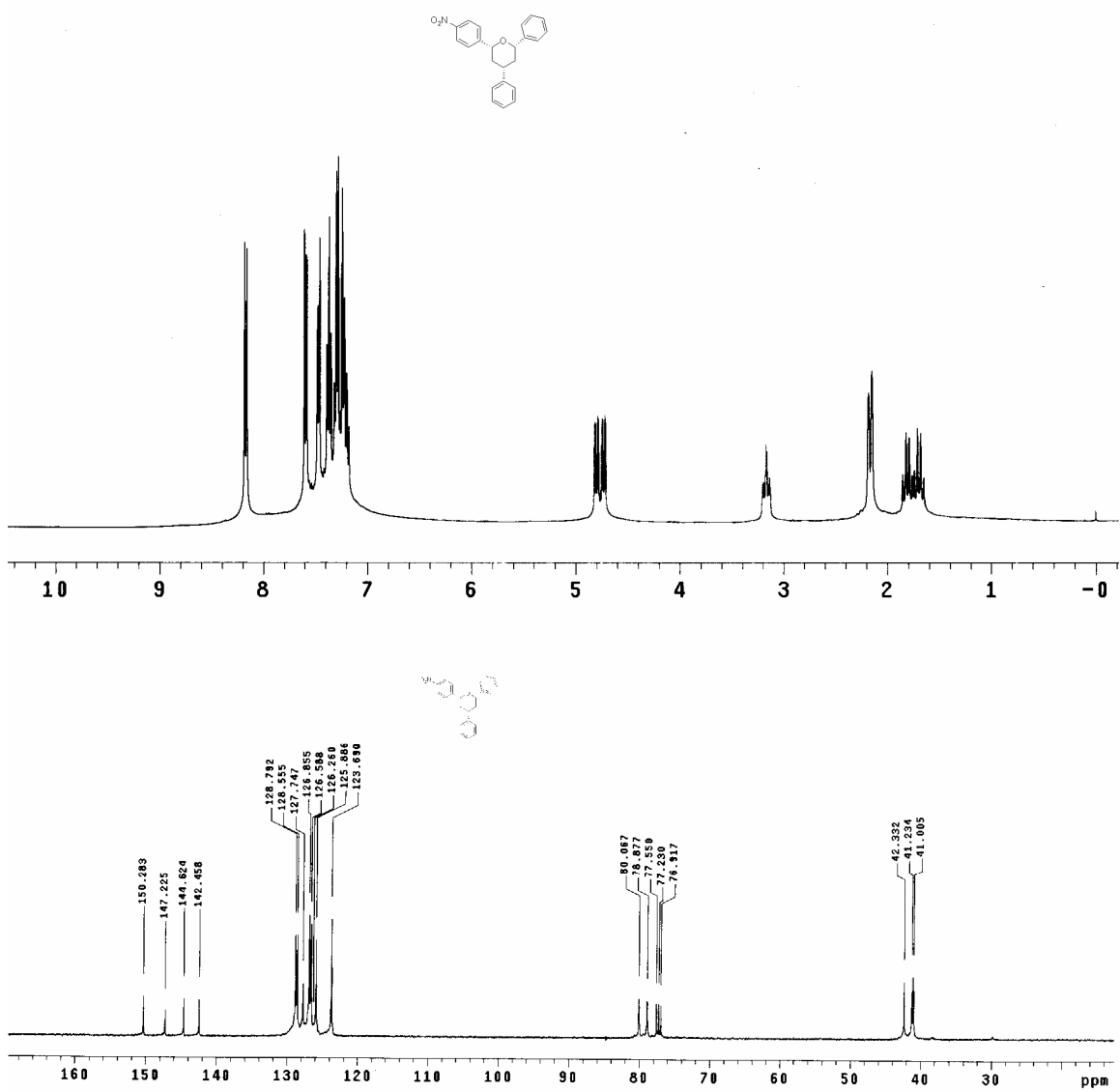
^1H and ^{13}C NMR spectra of compound **14b**



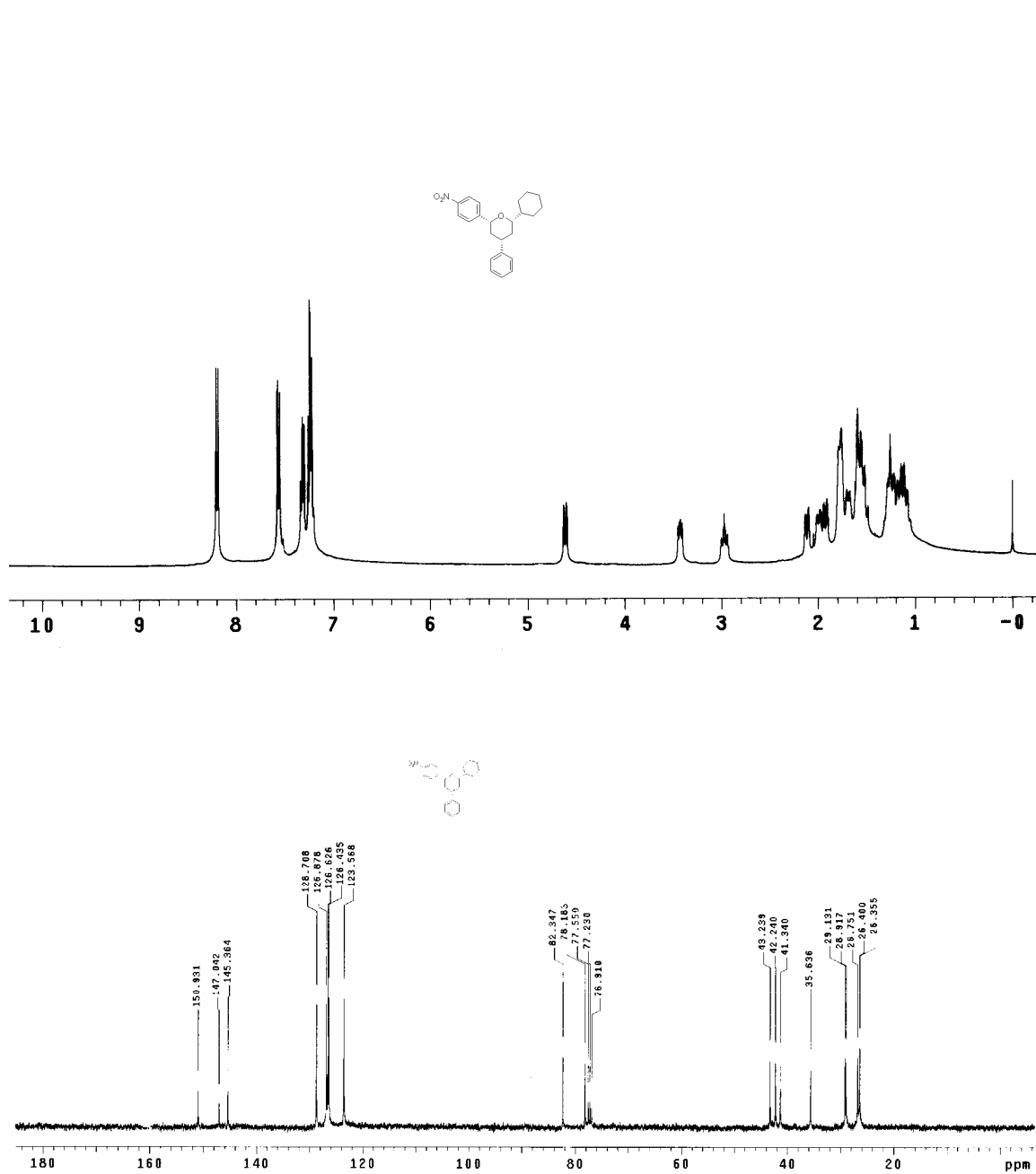
¹H and ¹³C NMR spectra of compound **15b**



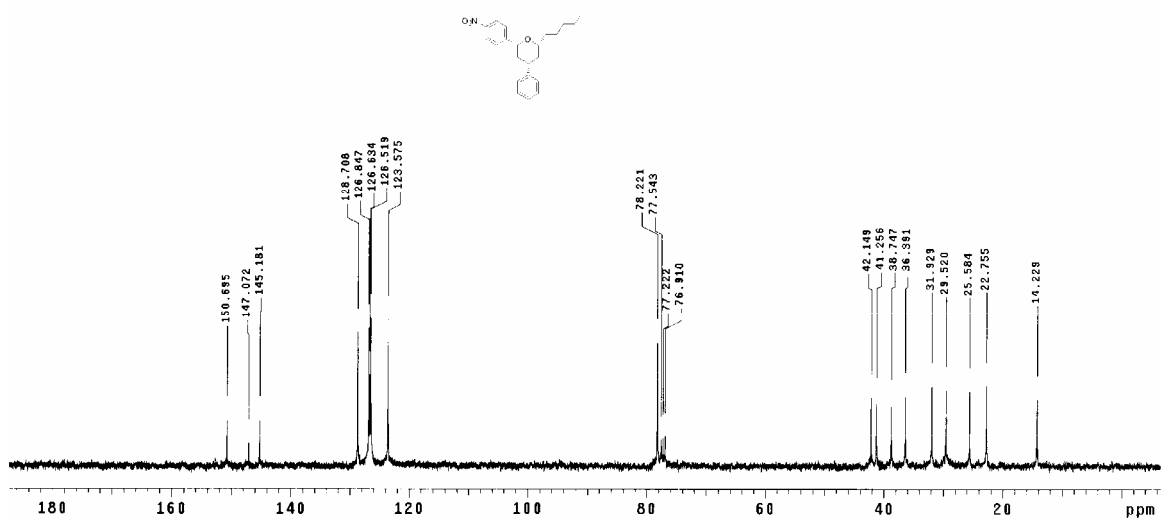
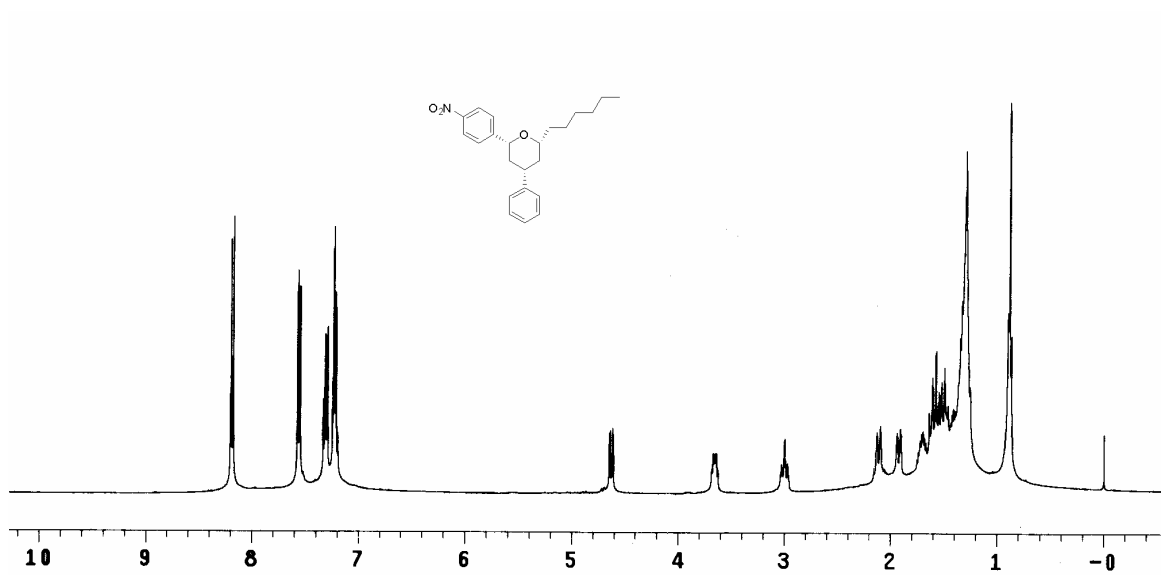
¹H and ¹³C NMR spectra of compound **16b**



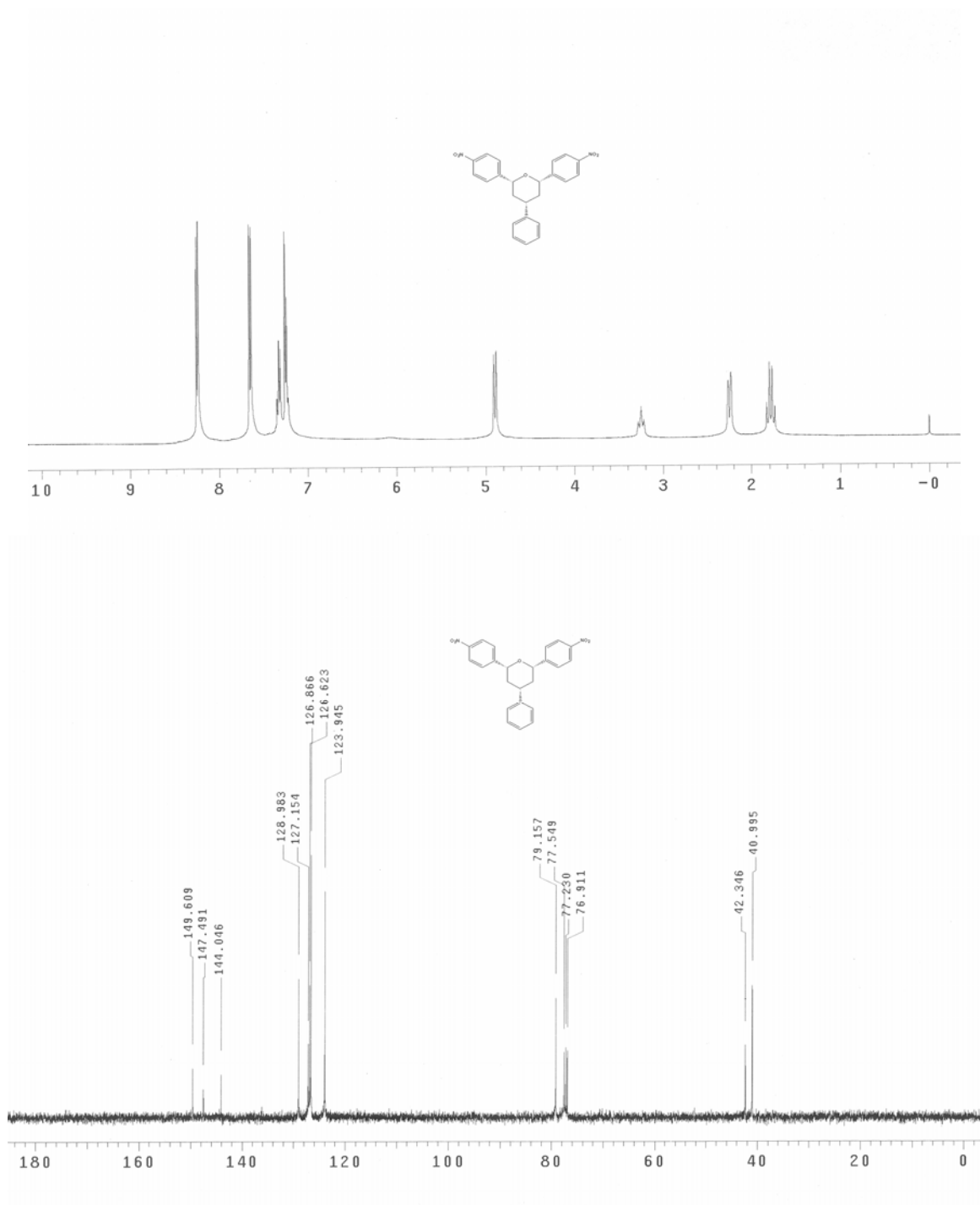
^1H and ^{13}C NMR spectra of compound **18b**



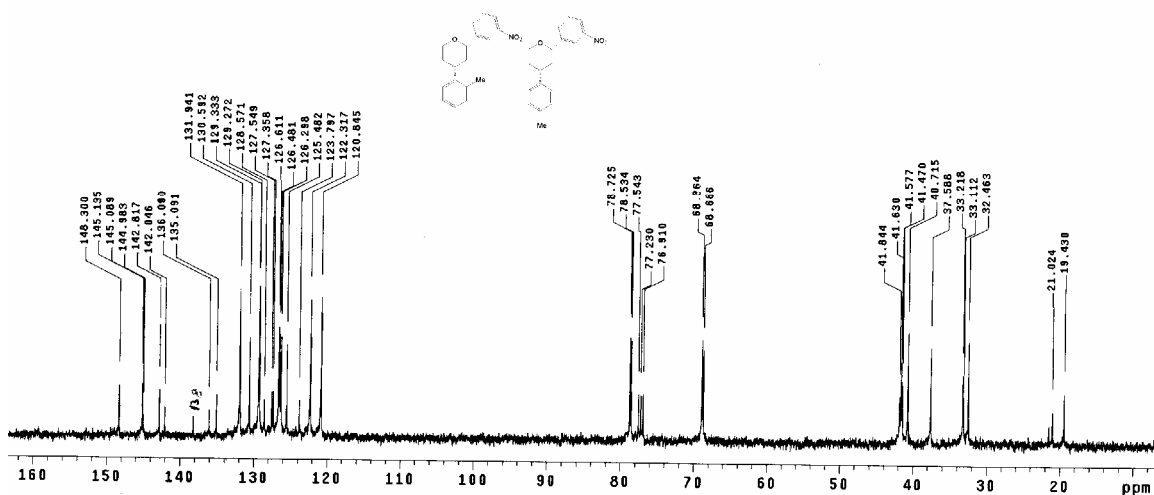
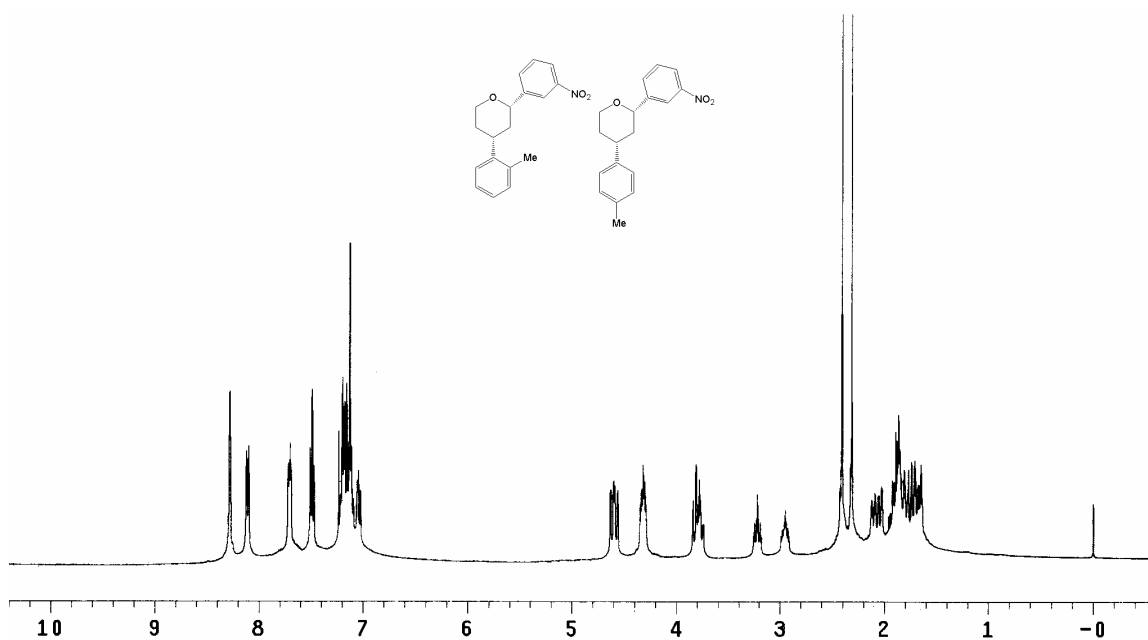
^1H and ^{13}C NMR spectra of compound **19b**



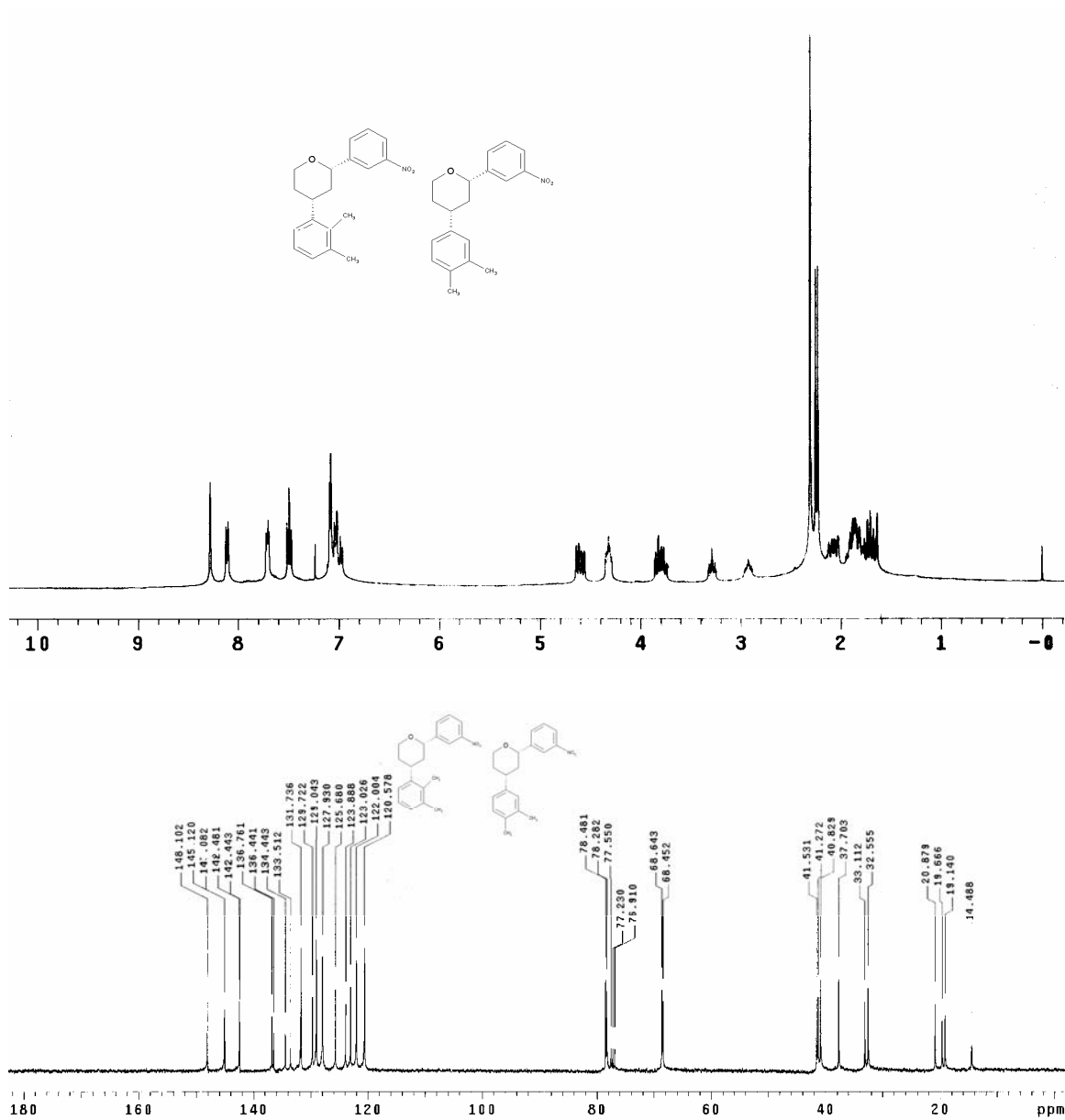
¹H and ¹³C NMR spectra of compound **20b**



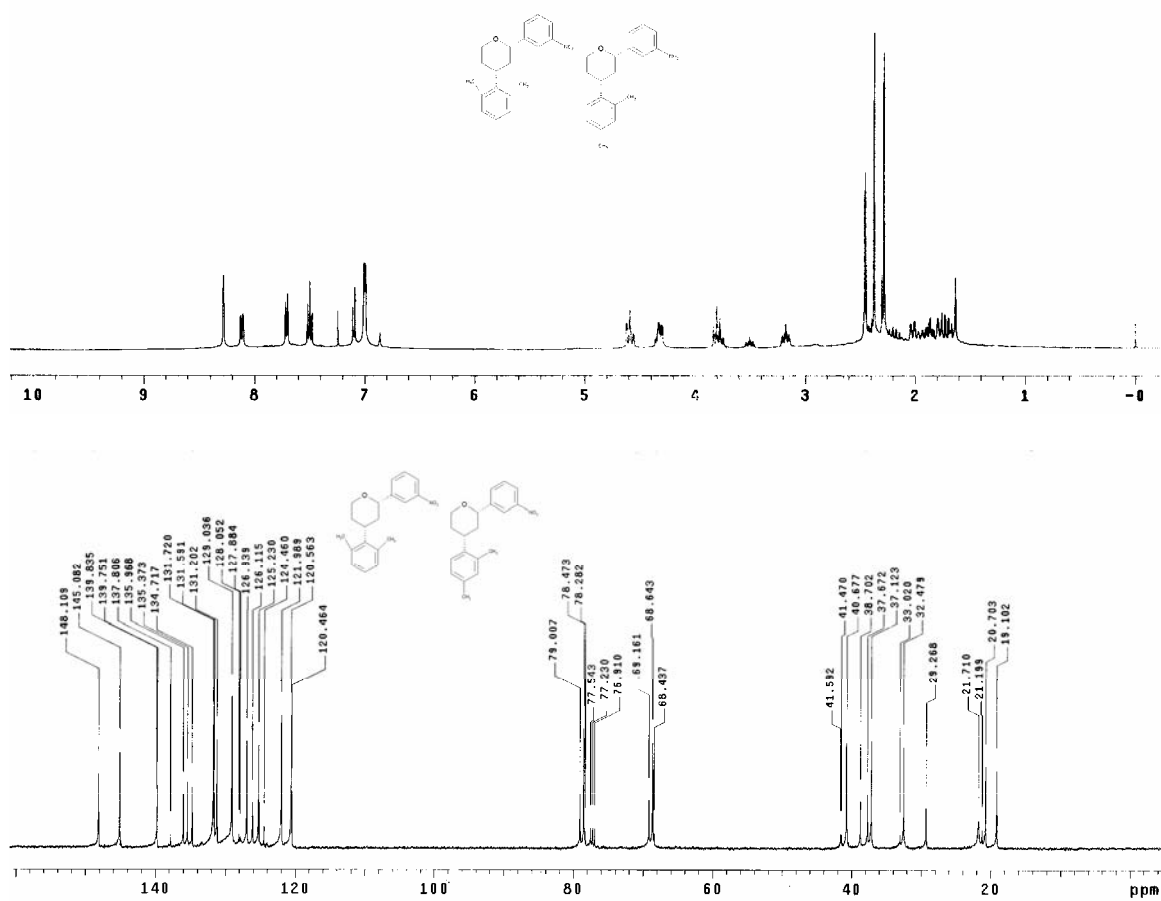
^1H and ^{13}C NMR spectra of compound **21b**



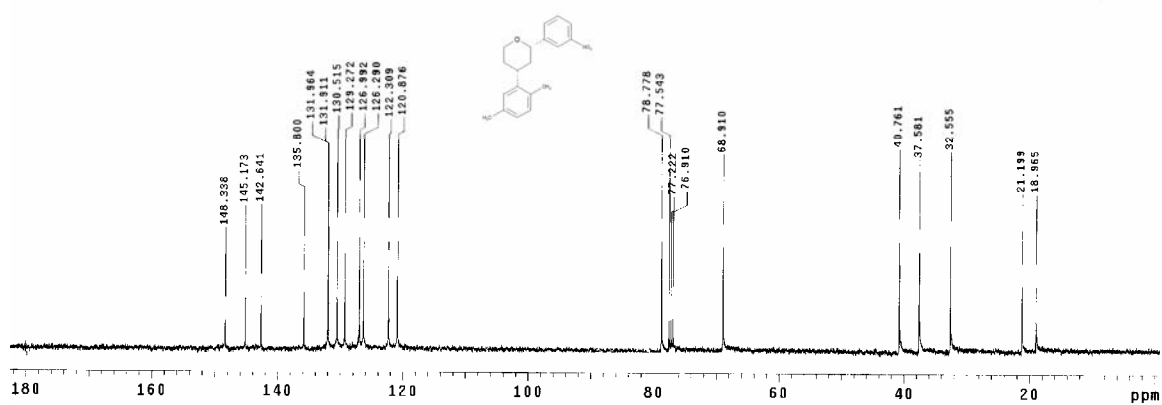
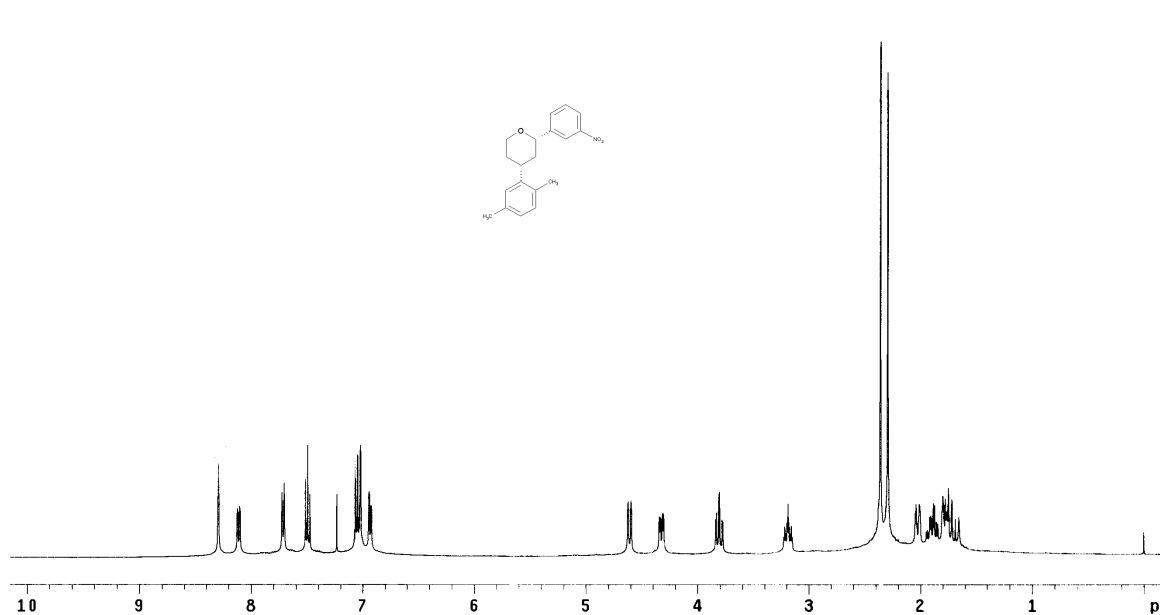
¹H and ¹³C NMR spectra of compound 22



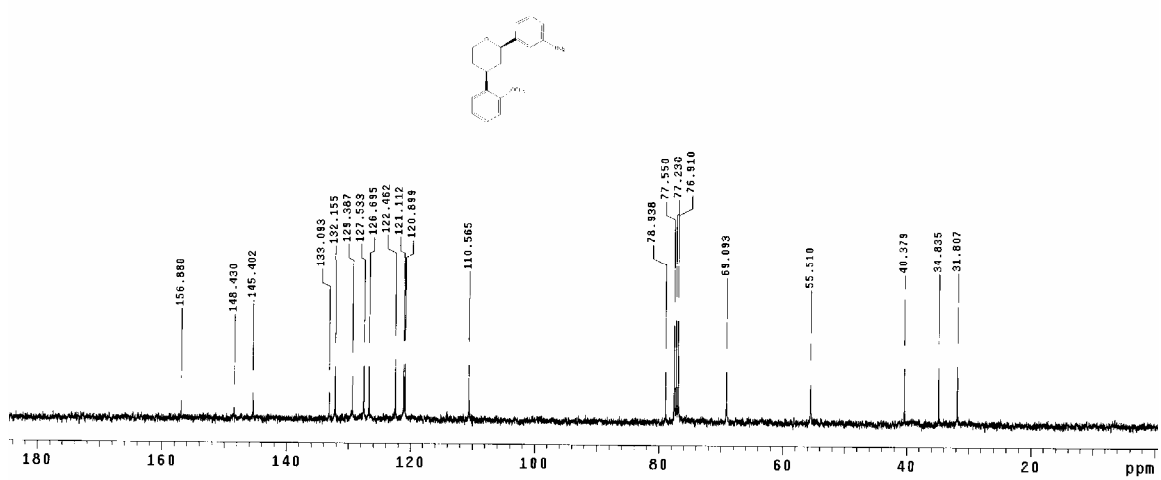
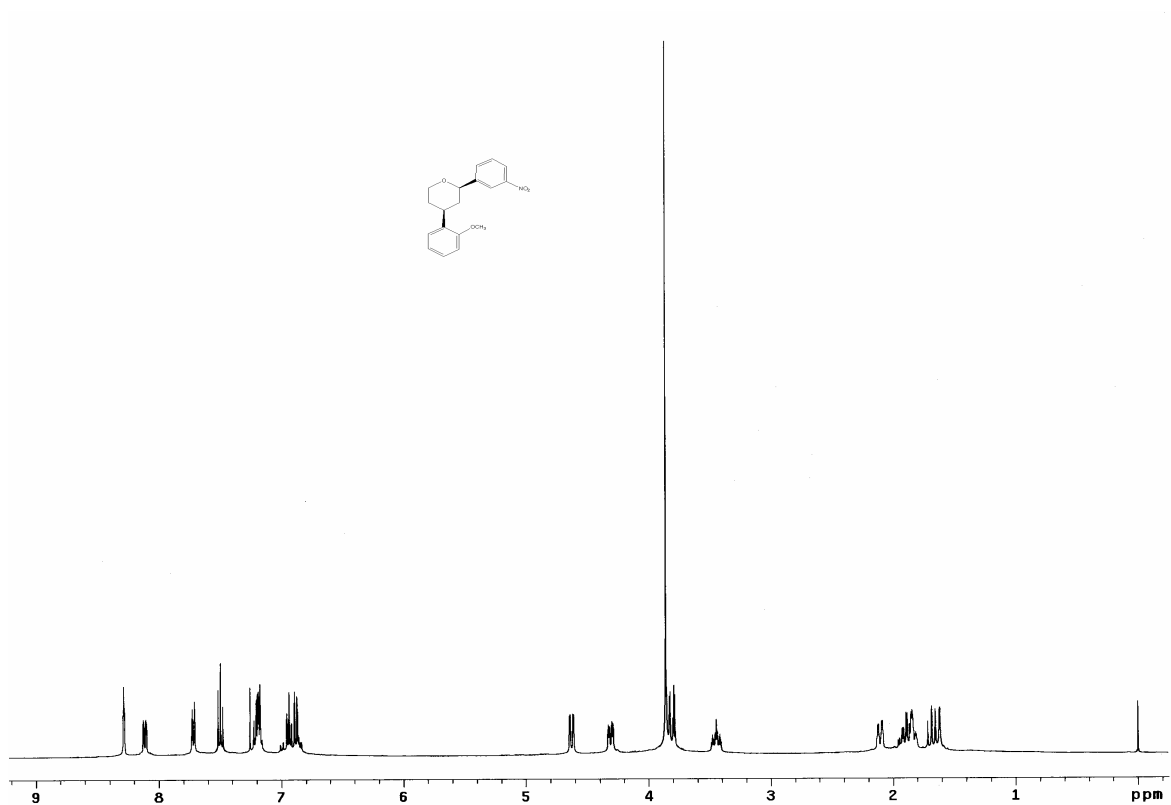
^1H and ^{13}C NMR spectra of compound **23**

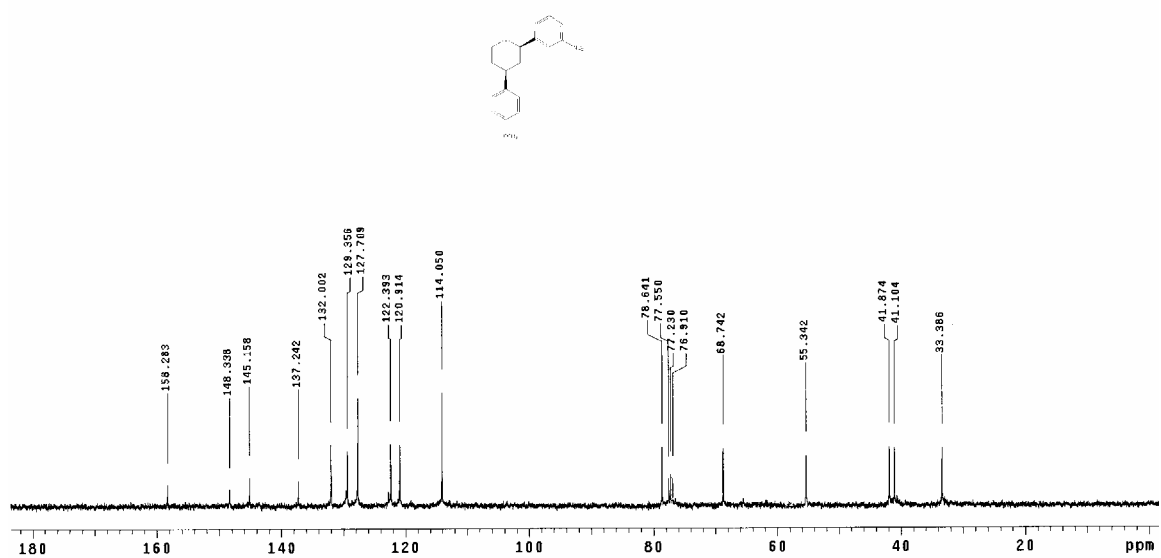
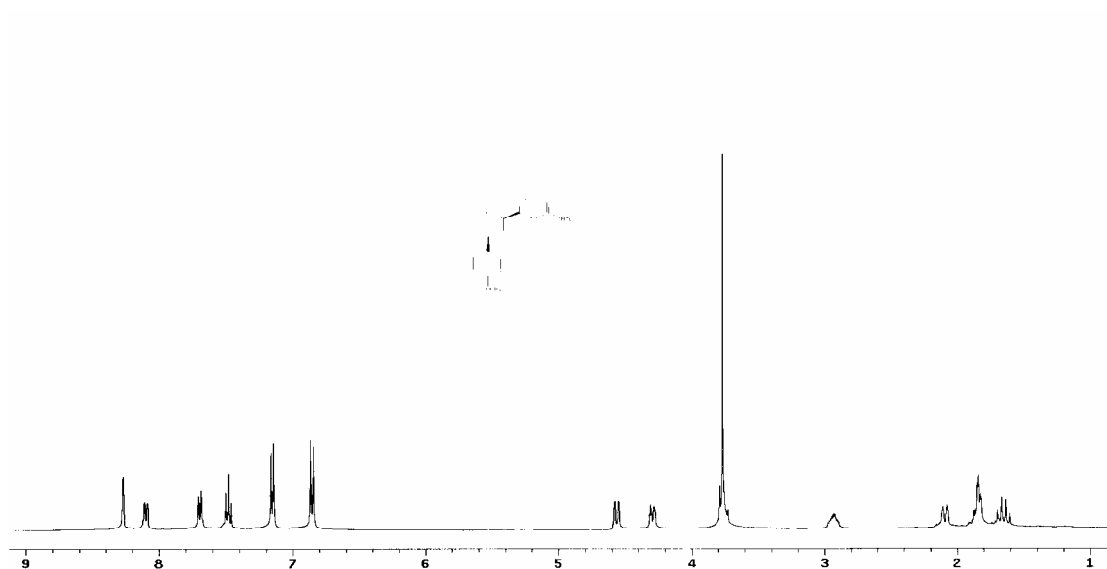


¹H and ¹³C NMR spectra of compound **24**

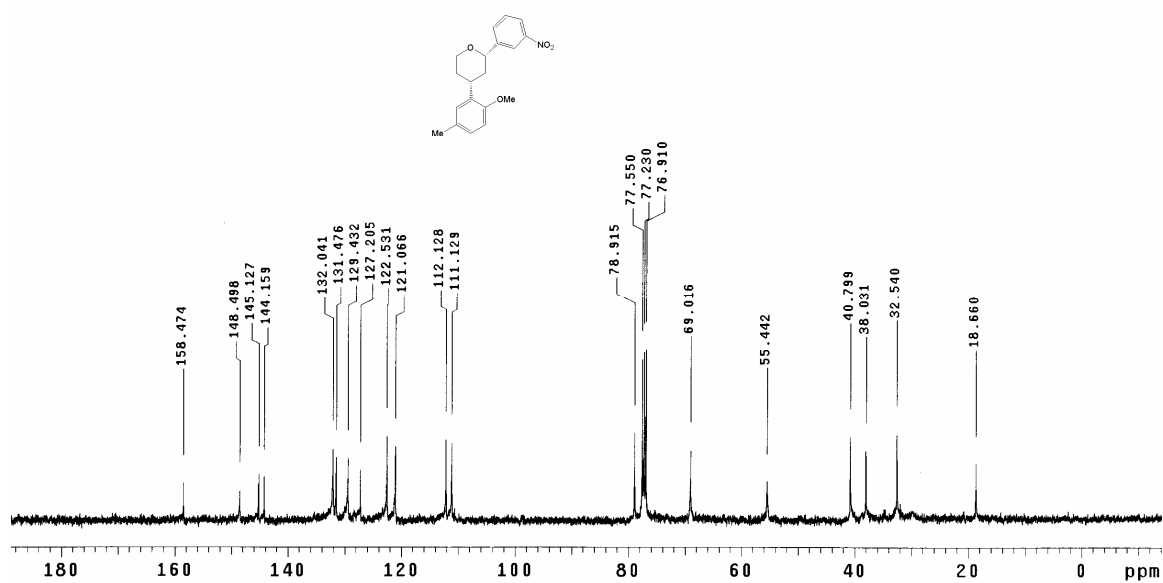
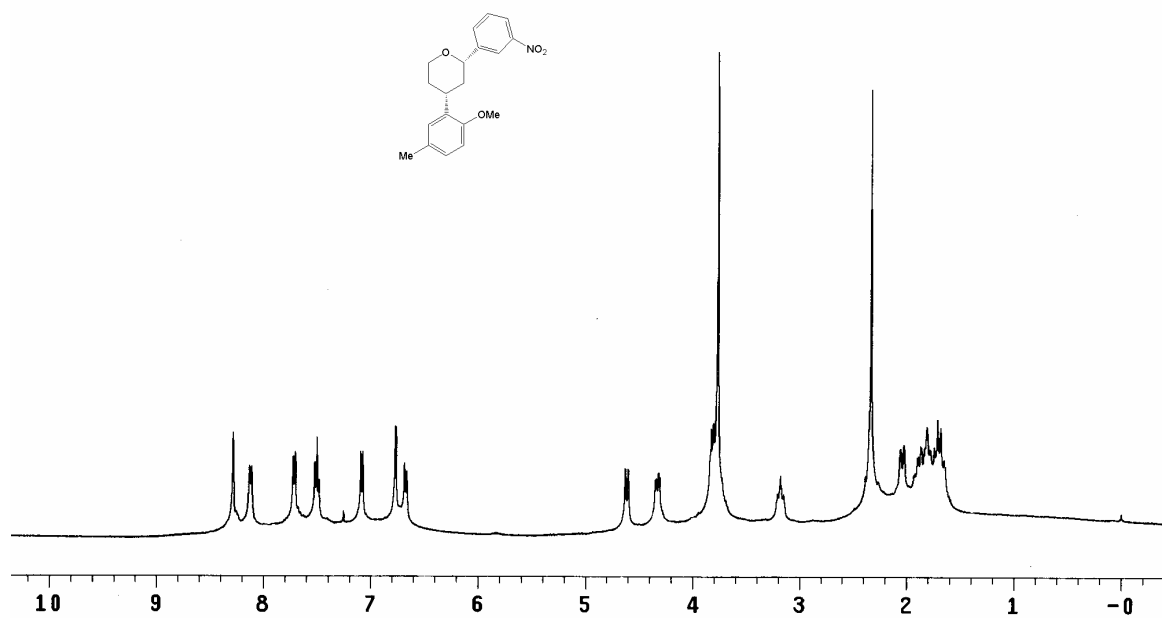


^1H and ^{13}C NMR spectra of compound **25**

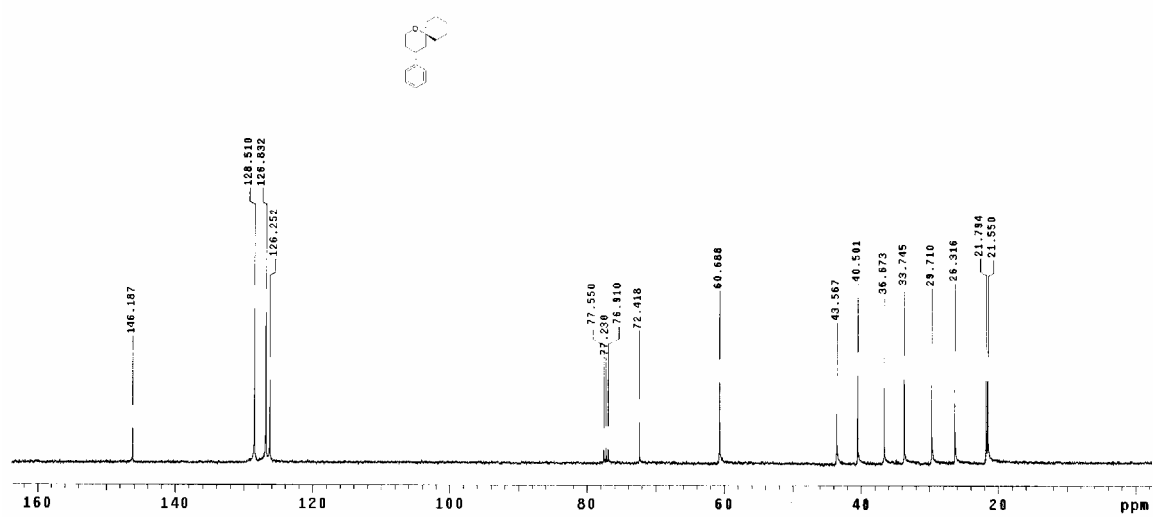
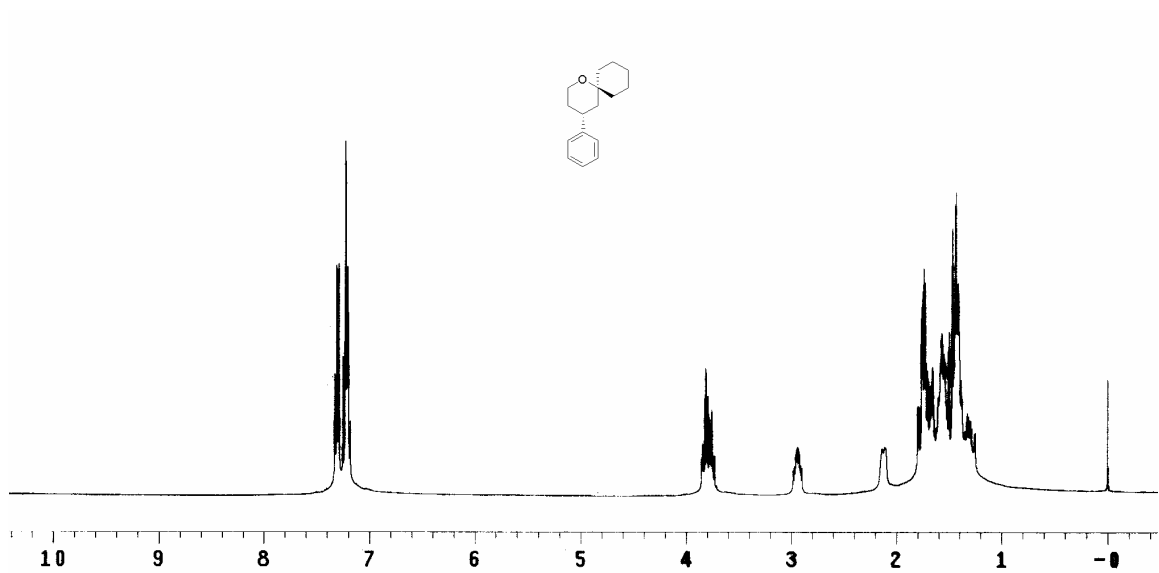
¹H and ¹³C NMR spectra of compound **26o**



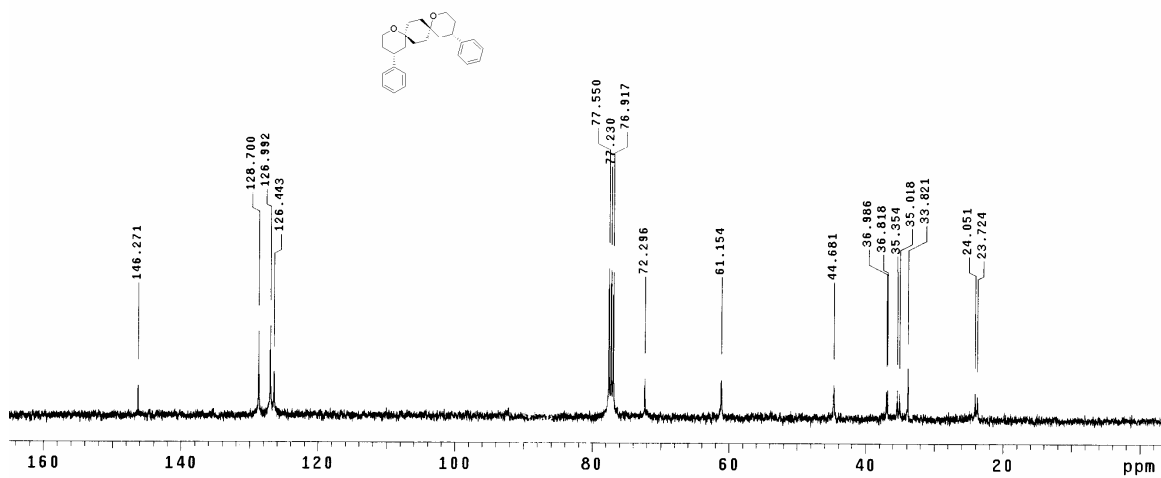
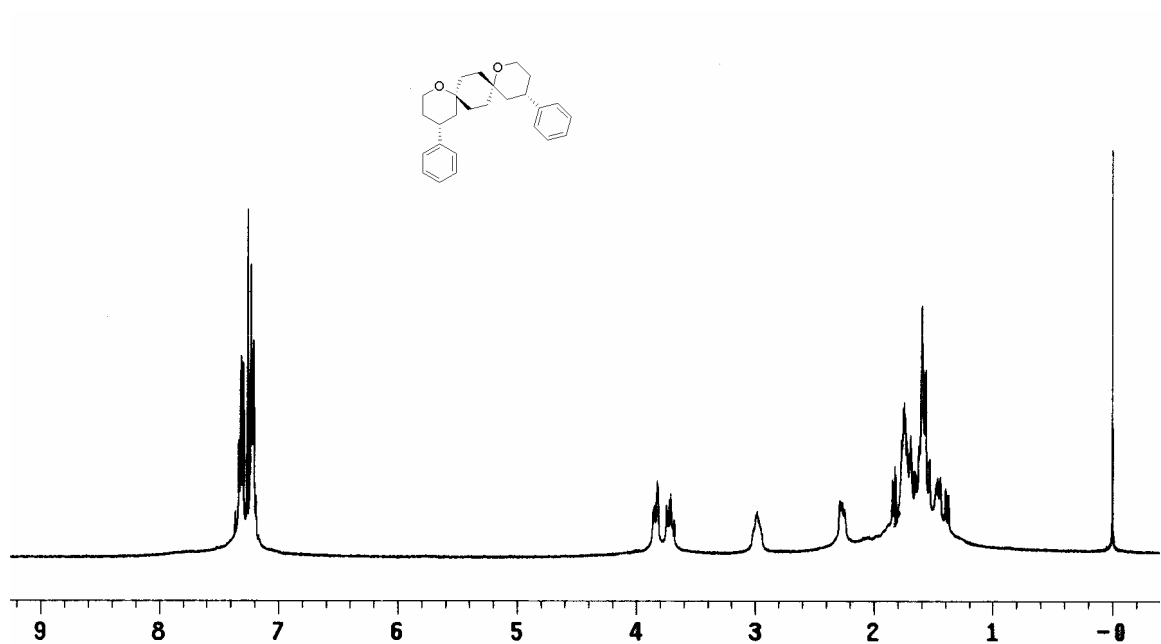
^1H and ^{13}C NMR spectra of compound **26p**



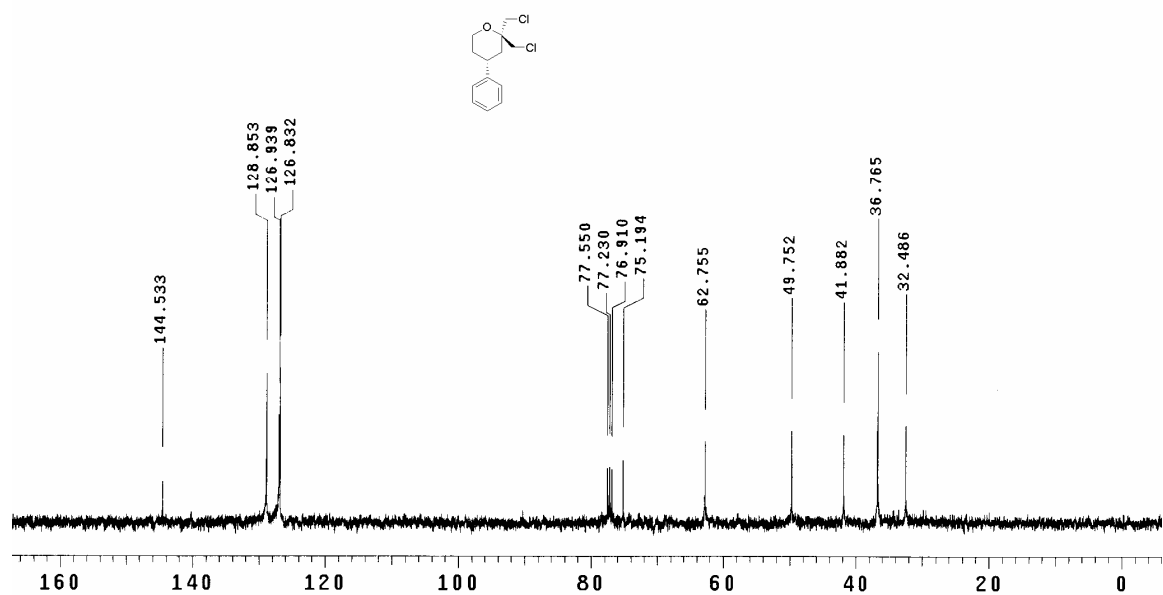
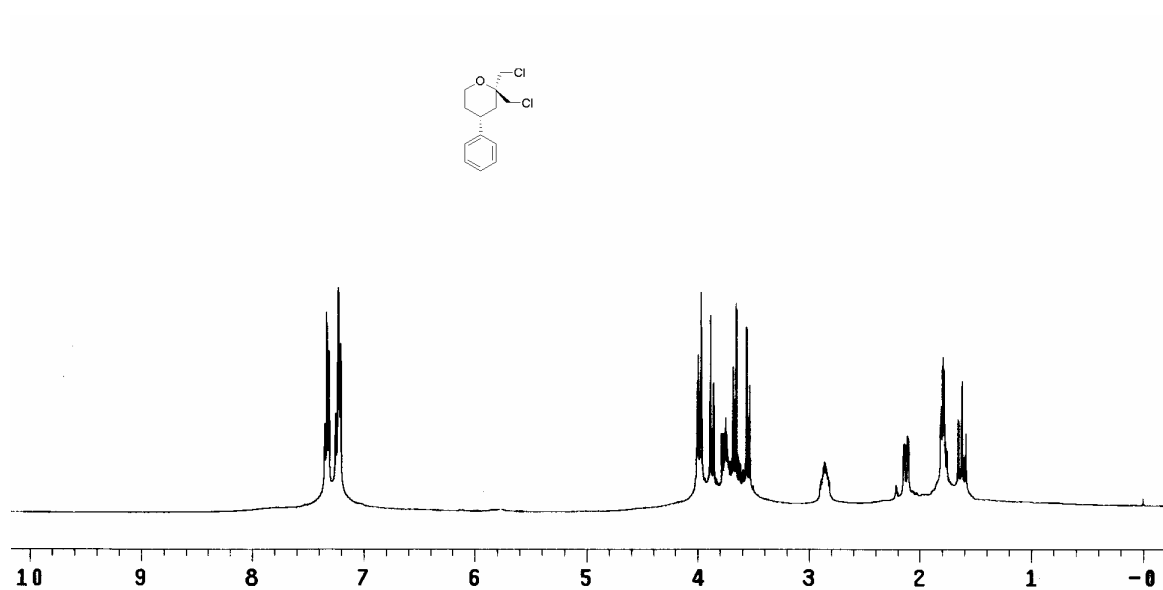
¹H and ¹³C NMR spectra of compound **27**



^1H and ^{13}C NMR spectra of compound **28**

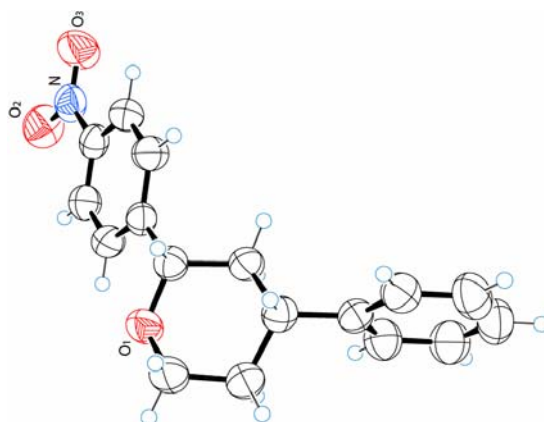


^1H and ^{13}C NMR spectra of compound **29**



^1H and ^{13}C NMR spectra of compound **30**

Figure 1: The ORTEP structure of 2-(4-Nitro-phenyl)-4-phenyl-tetrahydropyran(2b)



The crystal parameters of compound 2b.

| | 1b (CMR-2; CCDC 658766) |
|------------------------------|---|
| Formula | C ₁₇ H ₁₇ NO ₃ |
| Formula weight | 283.32 |
| <i>T</i> /K | 296(2) |
| Crystal system | Monoclinic |
| Space group | P2(1)/c |
| <i>a</i> /Å | 15.0086(18) |
| <i>b</i> /Å | 5.5608(7) |
| <i>c</i> /Å | 18.443(2) |
| α /° | 90.00 |
| β /° | 105.738(3) |
| γ /° | 90.00 |
| <i>V</i> /Å ³ | 1481.6(3) |
| <i>Z</i> | 4 |
| Abs. Coeff./mm ⁻¹ | 0.087 |
| Abs. Correction | None |
| GOF on <i>F</i> ² | 0.968 |
| Final <i>R</i> indices | <i>R</i> 1 = 0.0434 |
| [<i>I</i> > 2σ(<i>I</i>)] | <i>wR</i> 2 = 0.0996 |
| <i>R</i> indices [all data] | <i>R</i> 2 = 0.0984 |
| | <i>wR</i> 2 = 0.1218 |