

Synthesis of Heterocyclic 1,3-Oxazolines from Aldehydes with Trimethylphenylammonium Tribromide

Shinsei Sayama*

Supporting Information

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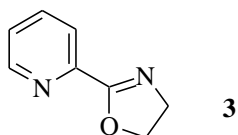
General Information

Melting points were measured on a Buchi Melting Point B-540 apparatus. IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer.

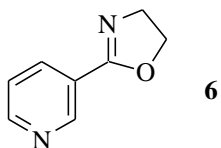
^1H and ^{13}C NMR spectra were recorded with a JEOL JNM-EX270 spectrometer at room temperature and the chemical shifts (δ) were given relative to internal standard SiMe_4 in CDCl_3 . Elemental analyses were performed on a Yanagimoto MT-6.

Typical procedure:

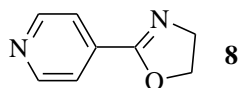
To a solution of 3-quinolinecarbaldehyde **17** (79 mg, 0.5 mmol), 2-aminoethanol (200 μL , 3.2 mmol), and pyridine (80 μL , 1.0 mmol) in MeOH (6 mL), was added PTAB (trimethylphenylammonium tribromide or phenyltrimethylammonium tribromide, 376 mg, 1.0 mmol) at room temperature. After stirring for 22 h at rt, the reaction mixture was treated with 0.5 M aq $\text{Na}_2\text{S}_2\text{O}_3$ (10 mL), 1.0 M NaHCO_3 (15 mL) and extracted with EtOAc (60 mL). The organic layer was washed with 0.5 M $\text{Na}_2\text{S}_2\text{O}_3$ and successively washed with saturated aq NaCl, and dried over MgSO_4 . After removal of solvent in vacuo, the residue was purified by column chromatography on silica gel (Wako C-200) with CCl_4 , CCl_4 - CHCl_3 (2:1 v/v). 3-(Oxazolin-2-yl)quinoline **18** (85 mg) was obtained in 86% yield.



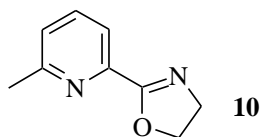
Pale yellow solid; mp 44.5-45.5 °C. IR (neat, cm^{-1}) 3063, 2979, 2909, 2881, 1654, 1585, 1570, 1472, 1441, 1367, 1293, 1269, 1248, 1195, 1151, 1122, 1098, 1044, 1000, 977, 944, 905, 802, 747, 674. ^1H NMR (CDCl_3) δ 4.14 (2H, t, $J = 8.1$ Hz), 4.53 (2H, t, $J = 8.1$ Hz), 7.40 (1H, dd, $J = 8.1, 5.4$ Hz), 7.79 (1H, t, $J = 8.1$ Hz), 8.05 (1H, d, $J = 8.1$ Hz), 8.71 (1H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 55.04, 68.13, 123.73, 125.47, 136.57, 146.66, 149.62, 163.80. *Anal.* Calcd for $\text{C}_8\text{H}_8\text{ON}_2$: C, 64.84; H, 5.44; N, 18.91. Found: C, 65.01; H, 5.47; N, 18.86.



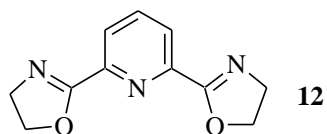
Pale yellow solid; mp 69.0-69.5 °C. IR (KBr, cm^{-1}) 3090, 3045, 2985, 2952, 2935, 2909, 2881, 1652, 1591, 1573, 1484, 1422, 1359, 1263, 1240, 1189, 1116, 1080, 1034, 1022, 979, 933, 896, 819, 745, 703, 686, 622. ^1H NMR (CDCl_3) δ 4.09 (2H, t, $J = 8.1$ Hz), 4.47 (2H, t, $J = 8.1$ Hz), 7.36 (1H, dd, $J = 5.4, 2.7$), 8.22 (1H, d, $J = 5.4$ Hz), 7.71 (1H, dd, $J = 5.4, 2.7$ Hz), 9.16 (1H, brs). ^{13}C NMR (CDCl_3) δ 54.92, 67.73, 123.16, 123.81, 135.46, 149.38, 151.95, 162.65. *Anal.* Calcd for $\text{C}_8\text{H}_8\text{ON}_2$: C, 64.84; H, 5.44; N, 18.91. Found: C, 64.86; H, 5.37; N, 18.84.



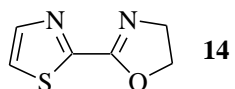
Pale yellow solid; mp 113.5-114.2 °C. IR (neat, cm^{-1}) 3032, 2974, 1650, 1599, 1552, 1497, 1474, 1409, 1365, 1335, 1260, 1215, 1092, 1074, 992, 972, 943, 898, 841, 743, 672. ^1H NMR (CDCl_3) δ 4.11 (2H, t, $J = 8.1$ Hz), 4.48 (2H, t, $J = 8.1$ Hz), 7.78 (2H, d, $J = 5.4$ Hz), 8.71 (2H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 55.06, 67.91, 121.84, 134.99, 150.22, 162.92. *Anal.* Calcd for $\text{C}_8\text{H}_8\text{ON}_2$: C, 64.84; H, 5.44; N, 18.91. Found: C, 64.46; H, 5.29; N, 18.45.



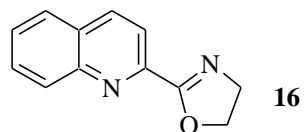
Colorless solid; mp 47.0-48.0 °C. IR (KBr, cm^{-1}) 3054, 2998, 2975, 2926, 2901, 2875, 1656, 1642, 1591, 1572, 1459, 1364, 1329, 1279, 1254, 1230, 1163, 1129, 1109, 1079, 1008, 996, 989, 974, 948, 911, 834, 814, 746, 690, 669. ^1H NMR (CDCl_3) δ 2.64 (3H, s), 4.12 (2H, t, $J = 8.1$ Hz), 4.53 (2H, t, $J = 8.1$ Hz), 7.27 (1H, d, $J = 8.1$ Hz), 7.67 (1H, t, $J = 8.1$ Hz), 7.85 (1H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 24.61, 54.99, 68.19, 120.90, 125.32, 136.76, 146.06, 158.79, 163.93. *Anal.* Calcd for $\text{C}_9\text{H}_{10}\text{ON}_2$: C, 66.64; H, 6.21; N, 17.27. Found: C, 66.47; H, 6.09; N, 17.07.



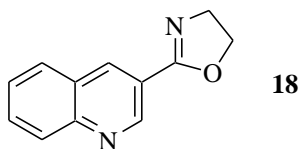
Colorless solid; mp 140-160 (decomp). IR (neat, cm^{-1}) 3049, 2974, 2932, 2874, 1644, 1569, 1476, 1453, 1427, 1357, 1323, 1274, 1239, 1151, 1125, 1096, 1065, 992, 975, 955, 937, 910, 894, 843, 743, 660, 648. ^1H NMR (CDCl_3) δ 4.13 (4H, t, $J = 8.1$ Hz), 4.54 (4H, t, $J = 8.1$ Hz), 7.88 (1H, t, $J = 8.1$ Hz), 8.16 (2H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 55.05, 68.32, 125.49, 137.35, 146.73, 163.44. *Anal.* Calcd for $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2$: C, 60.82; H, 5.10; N, 19.34. Found: C, 61.15; H, 5.28; N, 18.88.



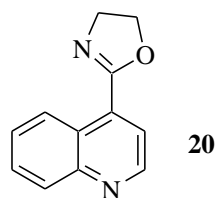
Pale yellow solid; mp 80.1-81.1 °C. IR (neat, cm^{-1}) 3107, 3075, 2984, 2875, 1645, 1493, 1426, 1360, 1314, 1260, 1243, 1198, 1149, 1070, 1040, 992, 972, 925, 875, 765, 695 607. ^1H NMR (CDCl_3) δ 4.13 (2H, t, $J = 8.1$ Hz), 4.55 (2H, t, $J = 8.1$ Hz), 7.51 (1H, d, $J = 5.4$ Hz), 7.96 (1H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 55.04, 68.71, 122.89, 144.34, 155.62, 159.38. *Anal.* Calcd for $\text{C}_6\text{H}_6\text{N}_2\text{OS}$: C, 46.74; H, 3.92; N, 18.17. Found: C, 46.58; H, 4.01; N, 17.75.



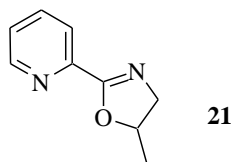
Colorless solid; mp 107.9-108.3 °C. IR (KBr, cm^{-1}) 3055, 3009, 2974, 2932, 2903, 2876, 1641, 1591, 1558, 1503, 1481, 1465, 1460, 1429, 1366, 1341, 1299, 1268, 1242, 1207, 1133, 1120, 1095, 1078, 1004, 975, 956, 936, 911, 882, 844, 793, 760, 625. ^1H NMR (CDCl_3) δ 4.19 (2H, t, $J = 8.1$ Hz), 4.60 (2H, t, $J = 8.1$ Hz), 7.60 (1H, t, $J = 8.1$ Hz), 7.76 (1H, t, $J = 8.1$ Hz), 7.78 (1H, d, $J = 5.4$ Hz), 8.15-8.29 (3H, m). ^{13}C NMR (CDCl_3) δ 55.12, 68.41, 120.57, 127.46, 127.87, 128.85, 130.01, 130.30, 136.72, 146.66, 147.49, 164.09. *Anal.* Calcd for $\text{C}_{12}\text{H}_{10}\text{ON}_2$: C, 72.70; H, 5.08; N, 14.13. Found: C, 72.85; H, 5.07; N, 13.96.



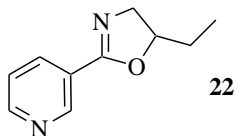
Colorless solid; mp 128.3-128.8 °C. IR (KBr, cm^{-1}) 2979, 2941, 2908, 2882, 1651, 1618, 1572, 1497, 1460, 1426, 1380, 1352, 1314, 1277, 1237, 1221, 1193, 1147, 1129, 1082, 1065, 1019, 978, 968, 933, 918, 906, 867, 785, 705, 631. ^1H NMR (CDCl_3) δ 4.15 (2H, t, $J = 8.1$ Hz), 4.51 (2H, t, $J = 8.1$ Hz), 7.59 (1H, t, $J = 8.1$ Hz), 7.78 (1H, t, $J = 8.1$ Hz), 7.88 (1H, d, $J = 8.1$ Hz), 8.14 (1H, d, $J = 8.1$ Hz), 8.67 (1H, brs), 9.45 (1H, brs). ^{13}C NMR (CDCl_3) δ 55.09, 67.68, 120.84, 126.94, 127.25, 128.62, 129.42, 130.95, 136.01, 149.02, 149.47, 162.78. *Anal.* Calcd for $\text{C}_{12}\text{H}_{10}\text{ON}_2$: C, 72.70; H, 5.08; N, 14.13. Found: C, 72.86; H, 5.09; N, 13.98.



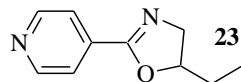
Pale yellow solid; 64.5-65.6 °C. IR (KBr, cm^{-1}) 3079, 2978, 2931, 2908, 2875, 1641, 1577, 1509, 1463, 1353, 1326, 1300, 1266, 1249, 1206, 1188, 1145, 1128, 1073, 999, 973, 943, 902, 879, 866, 813, 798, 768, 659. ^1H NMR (CDCl_3) δ 4.25 (2H, t, $J = 8.1$ Hz), 4.49 (2H, t, $J = 8.1$ Hz), 7.63 (1H, t, $J = 8.1$ Hz), 7.76 (1H, t, $J = 8.1$ Hz), 7.88 (1H, d, $J = 5.4$ Hz), 8.15 (1H, d, $J = 8.1$ Hz), 8.98 (1H, d, $J = 2.7$ Hz), 9.09 (1H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 55.86, 66.89, 121.71, 125.26, 126.37, 127.73, 129.54, 129.90, 132.19, 148.83, 149.72, 162.81. *Anal.* Calcd for $\text{C}_{12}\text{H}_{10}\text{ON}_2$: C, 72.70; H, 5.08; N, 14.13. Found: C, 72.79; H, 4.97; N, 13.94.



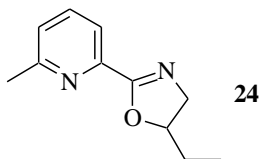
Pale yellow oil. IR (neat, cm^{-1}) 3063, 2968, 2938, 2878, 1651, 1644, 1585, 1569, 1530, 1471, 1441, 1382, 1359, 1291, 1269, 1247, 1201, 1102, 1045, 992, 964, 929, 909, 802, 747, 678. ^1H NMR (CDCl_3) δ 1.03 (3H, t, $J = 8.1$ Hz), 1.73 (2H, m), 3.74 (1H, dd, $J = 13.5, 8.1$ Hz), 4.17 (1H, dd, $J = 13.5, 8.1$ Hz), 4.78 (1H, m), 7.38 (1H, m), 7.77 (1H, t, $J = 8.1$ Hz), 8.03 (1H, d, $J = 8.1$ Hz), 8.71 (1H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 8.87, 27.97, 59.44, 81.84, 123.65, 125.32, 136.49, 146.84, 149.58, 163.24. *Anal.* Calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}$: C, 68.16; H, 6.86; N, 15.90. Found: C, 68.01; H, 6.80; N, 15.73.



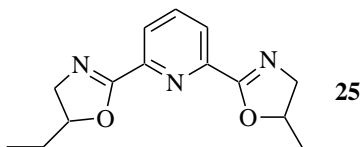
Pale yellow oil. IR (neat, cm^{-1}) 3046, 2968, 2938, 2878, 1651, 1594, 1571, 1484, 1475, 1463, 1430, 1416, 1351, 1297, 1263, 1193, 1085, 1036, 1024, 963, 929, 902, 819, 708. ^1H NMR (CDCl_3) δ 1.03 (3H, t, $J = 8.1$ Hz), 1.73 (2H, m), 3.69 (1H, dd, $J = 13.5, 8.1$ Hz), 4.13 (1H, dd, $J = 13.5, 8.1$ Hz), 4.71 (1H, m), 7.34 (1H, m), 8.22 (1H, d, $J = 8.1$ Hz), 8.70 (1H, d, $J = 5.4$ Hz), 9.15 (1H, brs). ^{13}C NMR (CDCl_3) δ 9.12, 28.24, 59.54, 81.50, 123.17, 124.15, 135.47, 149.35, 151.85, 162.11. *Anal.* Calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}$: C, 68.16; H, 6.86; N, 15.90. Found: C, 68.20; H, 7.05; N, 15.75.



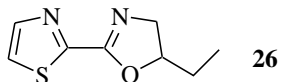
Colorless oil. IR (neat, cm^{-1}) 2969, 2939, 2878, 1651, 1600, 1556, 1497, 1463, 1412, 1383, 1357, 1321, 1296, 1263, 1228, 1094, 1063, 994, 963, 928, 905, 838, 745, 680. ^1H NMR (CDCl_3) δ 1.04 (3H, t, $J = 8.1$ Hz), 1.77 (2H, m), 3.70 (1H, dd, $J = 13.5, 8.1$ Hz), 4.15 (1H, dd, $J = 13.5, 8.1$ Hz), 4.71 (1H, m), 7.79 (2H, d, $J = 5.4$ Hz), 8.70 (2H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 8.89, 28.02, 59.45, 81.51, 121.70, 135.19, 149.92, 162.11. *Anal.* Calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}$: C, 68.16; H, 6.86; N, 15.90. Found: C, 68.32; H, 6.99; N, 15.48.



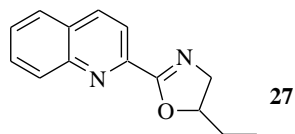
Pale yellow oil. IR (neat, cm^{-1}) 3065, 2966, 2938, 2877, 1639, 1590, 1575, 1463, 1380, 1351, 1275, 1256, 1236, 1166, 1119, 1084, 1016, 966, 929, 837, 810, 747, 695, 670. ^1H NMR (CDCl_3) δ 1.02 (3H, t, $J = 8.1$ Hz), 1.77 (2H, m), 2.64 (3H, s), 3.72 (1H, dd, $J = 13.5, 8.1$ Hz), 4.16 (1H, dd, $J = 13.5, 8.1$ Hz), 4.76 (1H, m), 7.24 (1H, d, $J = 8.1$ Hz), 7.68 (1H, t, $J = 8.1$ Hz), 7.80 (1H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 8.96, 24.56, 28.08, 59.51, 81.81, 120.77, 125.19, 136.65, 146.22, 158.84, 163.34. *Anal.* Calcd for $\text{C}_{11}\text{H}_{14}\text{N}_2\text{O}$: C, 69.45; H, 7.42; N, 14.73. Found: C, 69.69; H, 7.41; N, 14.81.



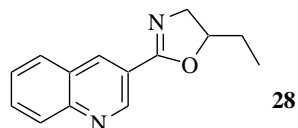
Pale yellow oil. IR (neat, cm^{-1}) 2968, 2938, 2878, 1660, 1575, 1536, 1461, 1434, 1382, 1344, 1273, 1244, 1172, 1138, 1111, 1079, 996, 966, 930, 831, 748, 667, 649. ^1H NMR (CDCl_3) δ 1.03 (6H, t, $J = 8.1$ Hz), 1.76 (4H, m), 3.73 (2H, dd, $J = 16.2, 8.1$ Hz), 4.17 (2H, dd, $J = 16.2, 8.1$ Hz), 4.77 (2H, m), 7.86 (1H, t, $J = 8.1$ Hz), 8.08 (2H, m). ^{13}C NMR (CDCl_3) δ 9.14, 9.17, 28.12, 28.16, 59.55, 59.58, 82.26, 82.30, 125.32, 137.34, 146.92, 162.90. *Anal.* Calcd for $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_2$: C, 65.91; H, 7.01; N, 15.37. Found: C, 65.79; H, 6.69; N, 15.32.



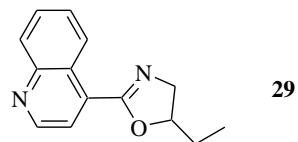
Pale yellow oil. IR (neat, cm^{-1}) 3082, 2967, 2937, 2877, 1645, 1533, 1494, 1462, 1428, 1349, 1314, 1244, 1148, 1043, 957, 924, 875, 761, 610. ^1H NMR (CDCl_3) δ 1.04 (3H, t, $J = 8.1$ Hz), 1.80 (2H, m), 3.72 (1H, dd, $J = 13.5, 8.1$ Hz), 4.16 (1H, dd, $J = 13.5, 8.1$ Hz), 4.81 (1H, m), 7.51 (1H, d, $J = 2.7$ Hz), 7.95 (1H, d, $J = 2.7$ Hz). ^{13}C NMR (CDCl_3) δ 8.94, 27.95, 59.48, 82.73, 122.77, 144.25, 156.00, 158.86. *Anal.* Calcd for $\text{C}_8\text{H}_{10}\text{OSN}_2$: C, 52.72; H, 5.53; N, 15.37. Found: C, 52.25; H, 5.47; N, 14.92.



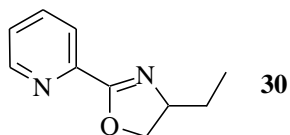
Pale yellow solid; mp 81.4-83.0 °C. IR (neat, cm^{-1}) 3061, 2966, 2937, 2876, 1637, 1595, 1561, 1505, 1464, 1429, 1377, 1364, 1354, 1302, 1268, 1243, 1209, 1125, 1085, 949, 929, 840, 766. ^1H NMR (CDCl_3) δ 1.05 (3H, t, $J = 8.1$ Hz), 1.84 (2H, m), 3.80 (1H, dd, $J = 16.2, 8.1$ Hz), 4.23 (1H, dd, $J = 16.2, 8.1$ Hz), 4.84 (1H, m), 7.59 (1H, t, $J = 8.1$ Hz), 7.75 (1H, t, $J = 8.1$ Hz), 7.83 (1H, d, $J = 8.1$ Hz), 8.16-8.30 (3H, m). ^{13}C NMR (CDCl_3) δ 8.89, 28.03, 59.57, 82.08, 120.51, 127.37, 127.73, 128.59, 129.85, 130.35, 136.60, 146.88, 147.54, 163.56. *Anal.* Calcd for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.32; H, 6.40; N, 12.33.



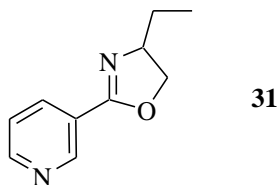
Pale yellow oil. IR (neat, cm^{-1}) 2967, 2938, 2876, 1651, 1620, 1571, 1497, 1462, 1428, 1382, 1315, 1275, 1236, 1196, 1128, 1070, 1007, 968, 927, 865, 844, 787, 756. ^1H NMR (CDCl_3) δ 1.07 (3H, t, $J = 8.1$ Hz), 1.84 (2H, m), 3.75 (1H, dd, $J = 16.2, 8.1$ Hz), 4.19 (1H, dd, $J = 16.2, 8.1$ Hz), 4.76 (1H, m), 7.60 (1H, t, $J = 8.1$ Hz), 7.79 (1H, t, $J = 8.1$ Hz), 7.89 (1H, d, $J = 8.1$ Hz), 8.15 (1H, d, $J = 8.1$ Hz), 8.68 (1H, brs), 9.44 (1H, brs). ^{13}C NMR (CDCl_3) δ 9.13, 28.23, 59.64, 81.46, 121.10, 126.98, 127.24, 128.60, 129.33, 130.92, 135.98, 148.93, 149.43, 162.24. *Anal.* Calcd for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.44; H, 6.39; N, 12.31.



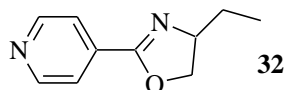
Pale yellow oil. IR (neat, cm^{-1}) 3066, 2966, 2937, 2876, 1644, 1576, 1509, 1463, 1434, 1380, 1337, 1322, 1296, 1268, 1247, 1180, 1144, 1130, 1093, 1073, 1026, 1001, 959, 931, 904, 878, 858, 816, 770, 657, 627. ^1H NMR (CDCl_3) δ 1.05 (3H, t, $J = 8.1$ Hz), 1.79 (2H, m), 3.85 (1H, dd, $J = 16.2, 8.1$ Hz), 4.28 (1H, dd, $J = 16.2, 8.1$ Hz), 4.73 (1H, m), 7.63 (1H, t, $J = 8.1$ Hz), 7.75 (1H, t, $J = 8.1$ Hz), 7.89 (1H, d, $J = 5.4$ Hz), 8.15 (1H, d, $J = 8.1$ Hz), 8.98 (1H, d, $J = 5.4$ Hz), 9.08 (1H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 9.14, 28.23, 60.35, 80.61, 121.66, 125.31, 126.34, 127.67, 129.51, 129.81, 132.59, 148.78, 149.67, 162.31. *Anal.* Calcd for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.28; H, 6.03; N, 12.14.



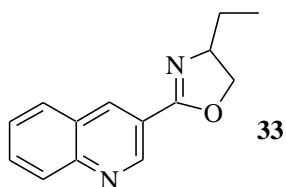
Brown oil. IR (neat, cm^{-1}) 2965, 2934, 2877, 1655, 1585, 1524, 1465, 1435, 1365, 1291, 1246, 1087, 997, 947, 748. ^1H NMR (CDCl_3) δ 1.03 (3H, t, $J = 8.1$ Hz), 1.73 (2H, m), 4.14 (1H, t, $J = 8.1$ Hz), 4.29 (1H, m), 4.58 (1H, t, $J = 8.1$ Hz), 7.39 (1H, t, $J = 8.1$ Hz), 7.78 (1H, t, $J = 8.1$ Hz), 8.04 (1H, d, $J = 5.4$ Hz), 8.71 (1H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 10.01, 28.44, 68.22, 72.65, 123.78, 125.39, 136.52, 146.76, 149.62, 162.55.



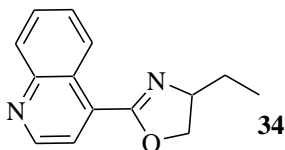
Yellow oil. IR (neat, cm^{-1}) 2965, 2933, 2878, 1659, 1651, 1645, 1593, 1573, 1475, 1462, 1429, 1416, 1361, 1330, 1291, 1247, 1193, 1109, 1079, 1036, 1024, 947, 900, 819, 709. ^1H NMR (CDCl_3) δ 1.01 (3H, t, $J = 8.1$ Hz), 1.70 (2H, m), 4.08 (1H, t, $J = 8.1$ Hz), 4.25 (1H, m), 4.51 (1H, t, $J = 8.1$ Hz), 7.35 (1H, t, $J = 5.4$ Hz), 8.22 (1H, dd, $J = 5.4, 2.7$ Hz), 8.69 (1H, d, $J = 2.7$ Hz), 9.14 (1H, brs). ^{13}C NMR (CDCl_3) δ 9.95, 28.53, 68.05, 72.30, 123.14, 124.01, 135.58, 149.44, 151.89, 161.52.



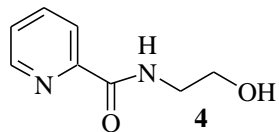
Yellow oil. IR (neat, cm^{-1}) 2966, 2934, 2878, 1651, 1601, 1556, 1462, 1413, 1364, 1291, 1063, 1000, 946, 903, 838, 745, 689. ^1H NMR (CDCl_3) δ 1.01 (3H, t, $J = 8.1$ Hz), 1.70 (2H, m), 4.09 (1H, t, $J = 8.1$ Hz), 4.27 (1H, m), 4.52 (1H, t, $J = 8.1$ Hz), 7.78 (2H, d, $J = 5.4$), 8.70 (2H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 9.87, 28.37, 68.13, 72.40, 121.91, 135.19, 150.09, 161.70.



Pale yellow solid; mp 50.2-51.0 °C. IR (neat, cm^{-1}) 2964, 2932, 2878, 1651, 1620, 1571, 1496, 1462, 1379, 1320, 1234, 1195, 1127, 1062, 981, 945, 787, 756. ^1H NMR (CDCl_3) δ 1.04 (3H, t, $J = 8.1$ Hz), 1.77 (2H, m), 4.13 (1H, t, $J = 8.1$ Hz), 4.33 (1H, m), 4.56 (1H, t, $J = 8.1$ Hz), 7.60 (1H, t, $J = 8.1$ Hz), 7.79 (1H, t, $J = 8.1$ Hz), 7.88 (1H, d, $J = 8.1$ Hz), 8.15 (1H, d, $J = 8.1$ Hz), 8.69 (1H, brs), 9.44 (1H, brs). ^{13}C NMR (CDCl_3) δ 10.00, 28.59, 68.25, 72.27, 121.02, 127.02, 127.26, 128.63, 129.43, 130.95, 136.15, 149.04, 149.54, 161.66.

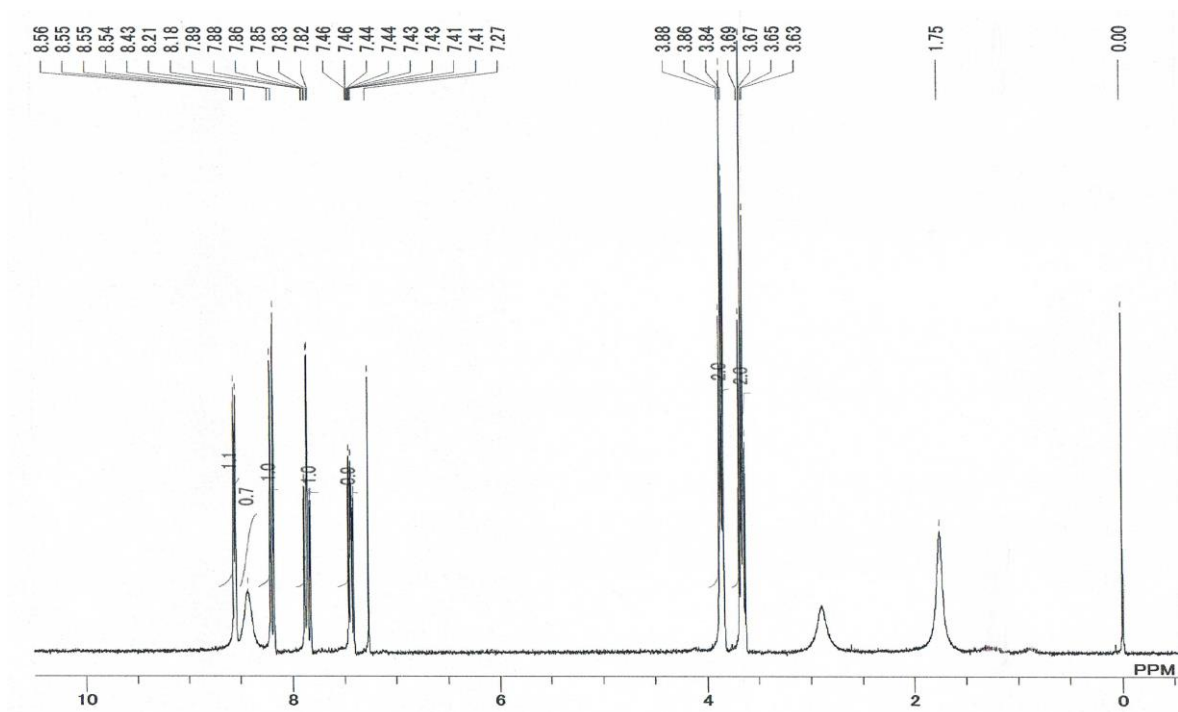
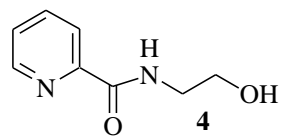


Yellow oil. IR (neat, cm^{-1}) 2964, 2932, 2874, 1645, 1576, 1509, 1462, 1347, 1274, 1248, 1182, 1145, 1072, 996, 947, 857, 770, 657. ^1H NMR (CDCl_3) δ 1.08 (3H, t, $J = 8.1$ Hz), 1.80 (2H, m), 4.12 (1H, t, $J = 8.1$ Hz), 4.44 (1H, m), 4.55 (1H, t, $J = 8.1$ Hz), 7.64 (1H, t, $J = 8.1$ Hz), 7.76 (1H, t, $J = 8.1$ Hz), 7.87 (1H, d, $J = 5.4$ Hz), 8.15 (1H, d, $J = 8.1$ Hz), 8.98 (1H, d, $J = 5.4$ Hz), 9.08 (1H, d, $J = 8.1$ Hz). ^{13}C NMR (CDCl_3) δ 10.05, 28.64, 69.02, 71.51, 121.72, 125.36, 126.43, 127.73, 129.55, 129.87, 132.52, 148.82, 149.72, 161.60.

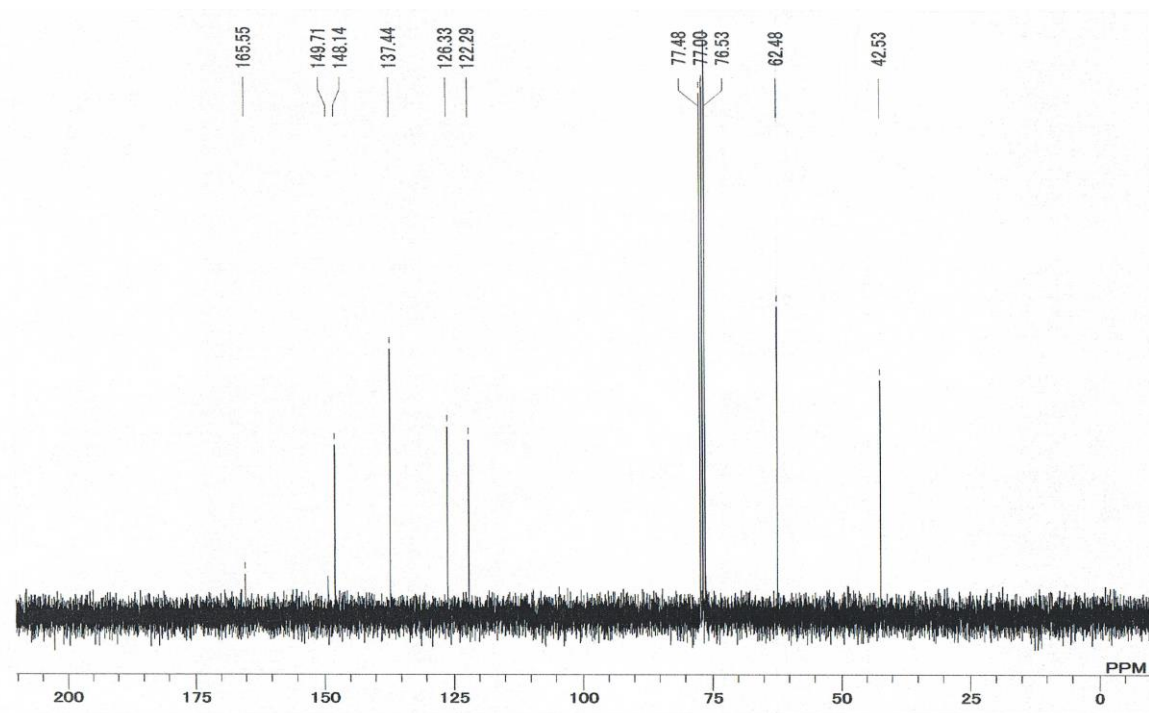
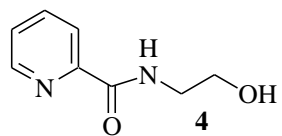


Brown oil. IR (neat, cm^{-1}) 3378, 3059, 2935, 2878, 1661, 1590, 1569, 1537, 1465, 1435, 1361, 1290, 1246, 1220, 1170, 1147, 1065, 997, 913, 820, 750, 693, 621. ^1H (CDCl_3) δ 3.66 (2H, t, $J = 5.4$ Hz), 3.86 (2H, t, $J = 5.4$ Hz), 7.44 (1H, m), 7.86 (1H, t, $J = 8.1$ Hz), 8.20 (1H, d, $J = 8.1$ Hz), 8.55 (1H, d, $J = 5.4$ Hz). ^{13}C NMR (CDCl_3) δ 42.53, 62.48, 122.29, 126.33, 137.44, 148.14, 149.71, 165.55. *Anal.* Calcd for $\text{C}_8\text{H}_{10}\text{O}_2\text{N}_2$: C, 57.82; H, 6.07; N, 16.86. Found: C, 57.95; H, 6.04; N, 16.97.

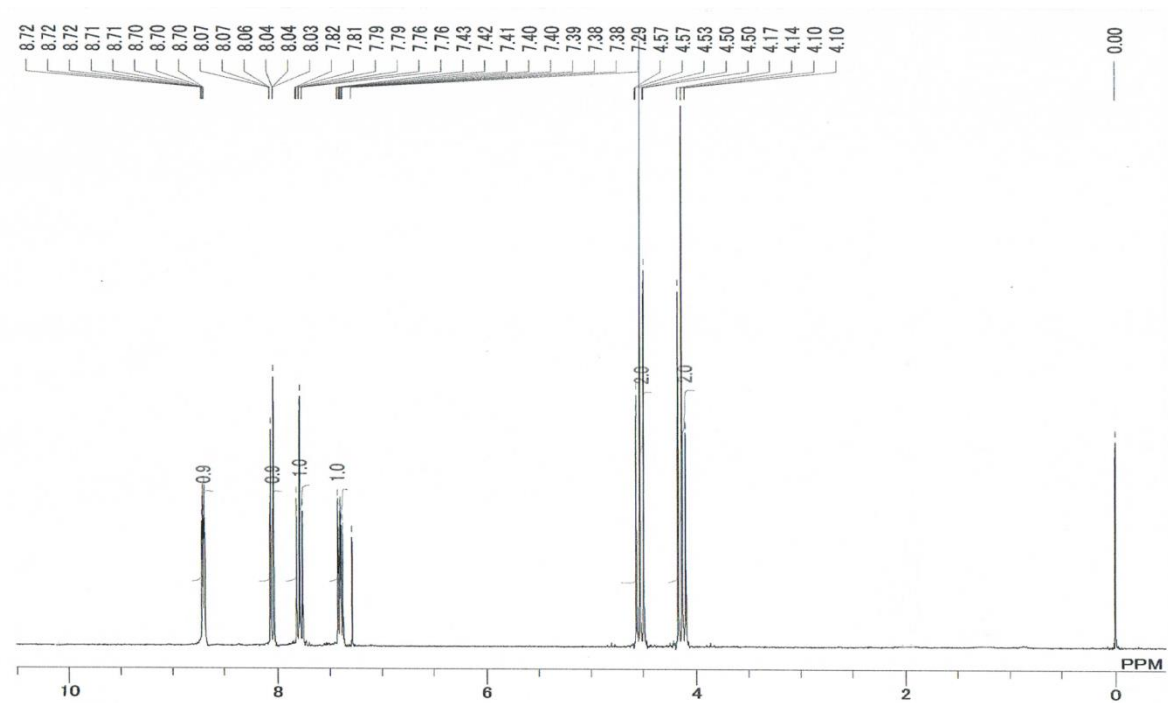
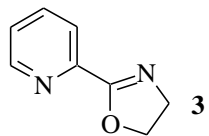
^1H NMR (CDCl_3 , 270.05 MHz)



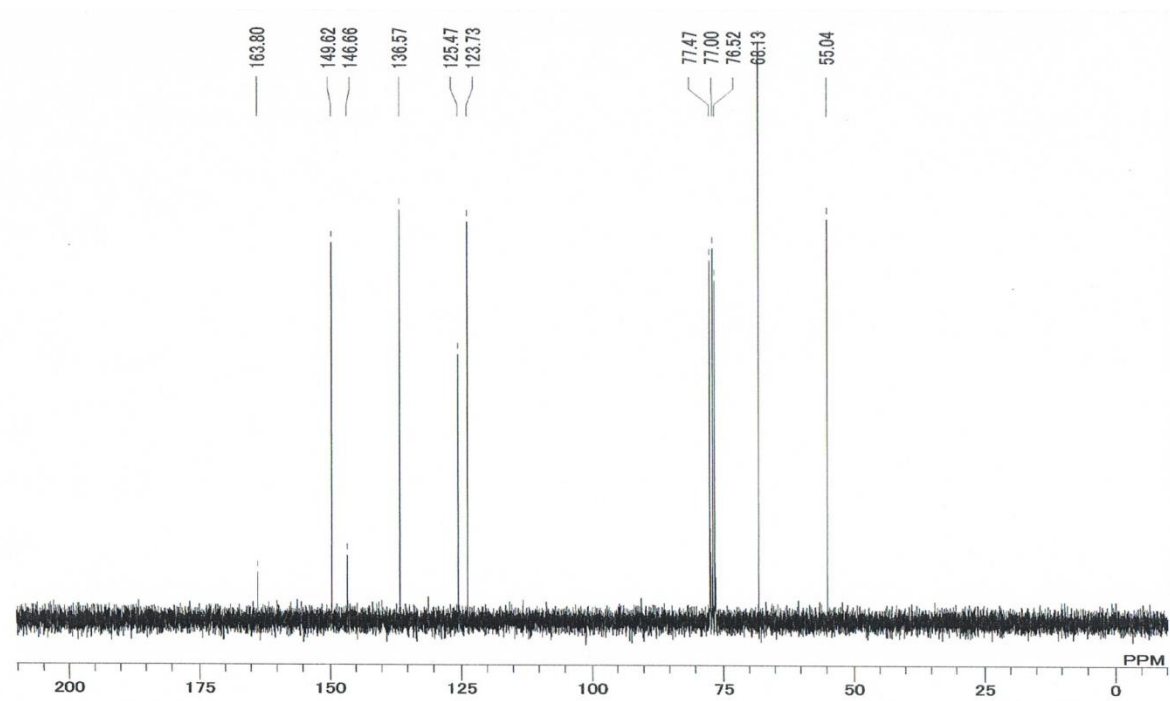
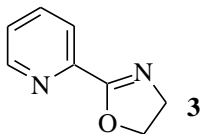
^{13}C NMR (CDCl_3 , 67.80 MHz)



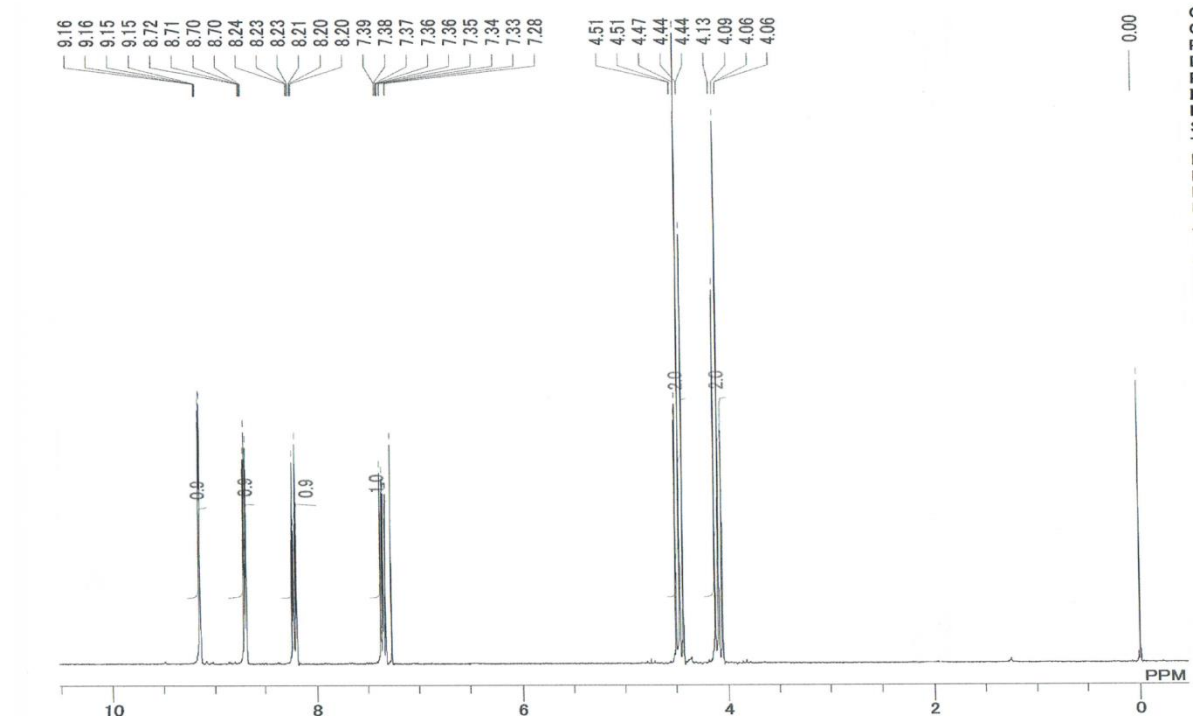
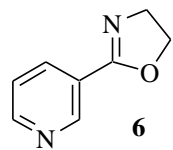
^1H NMR (CDCl_3 , 270.05 MHz)



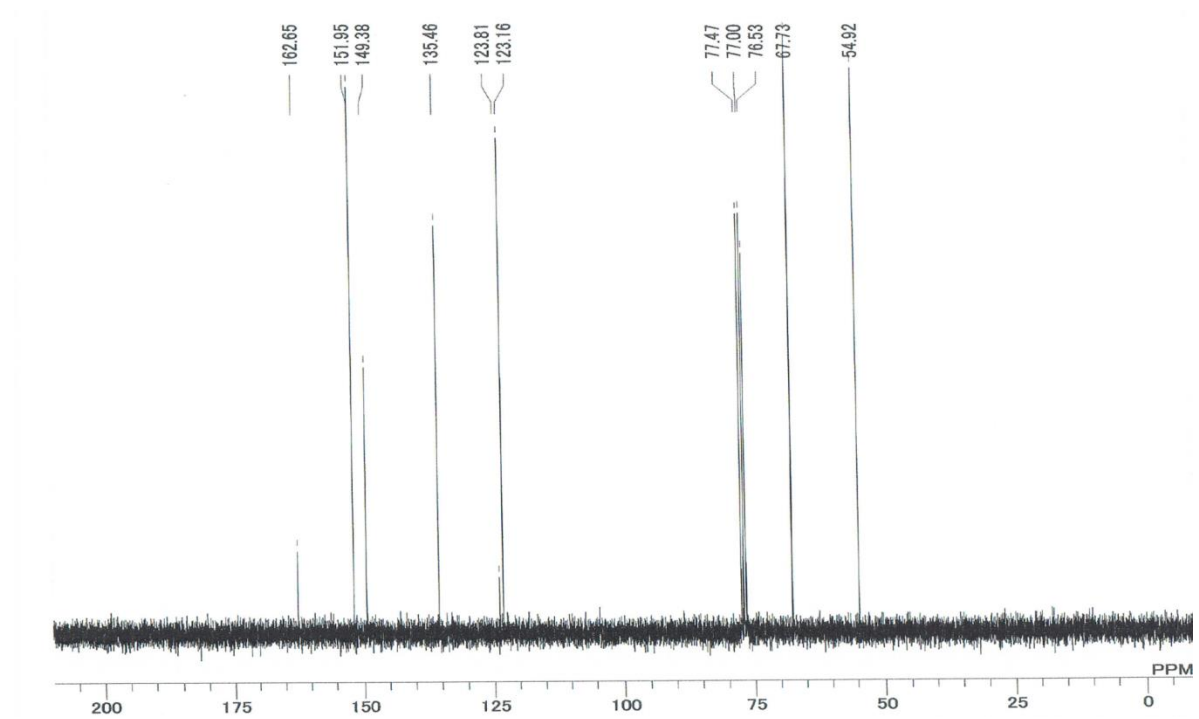
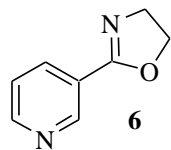
^{13}C NMR (CDCl_3 , 67.80 MHz)



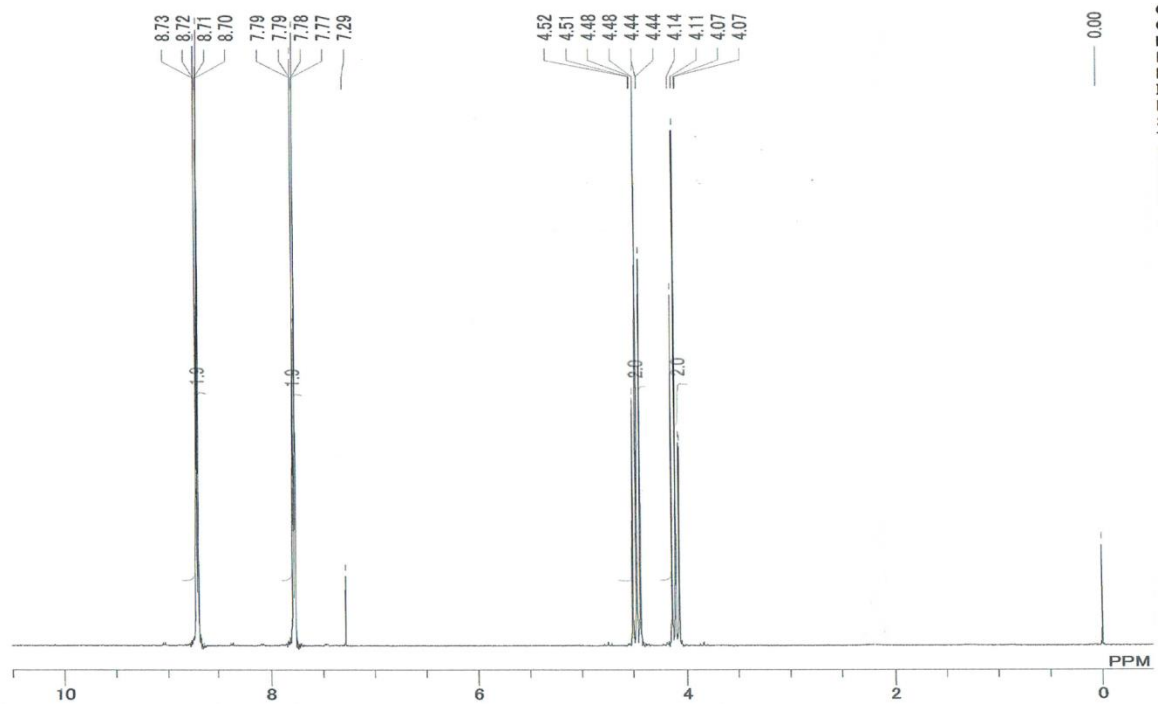
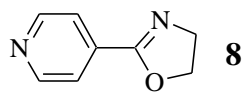
^1H NMR (CDCl_3 , 270.05 MHz)



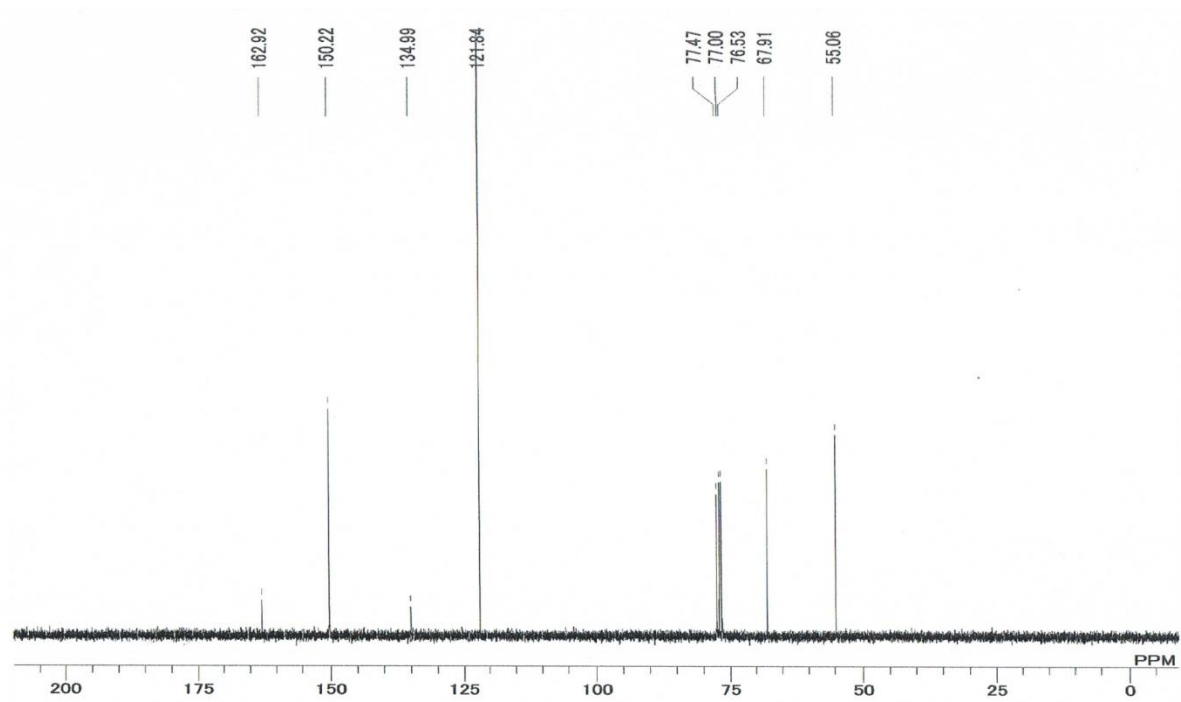
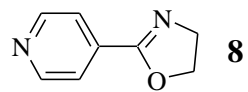
^{13}C NMR (CDCl_3 , 67.80 MHz)



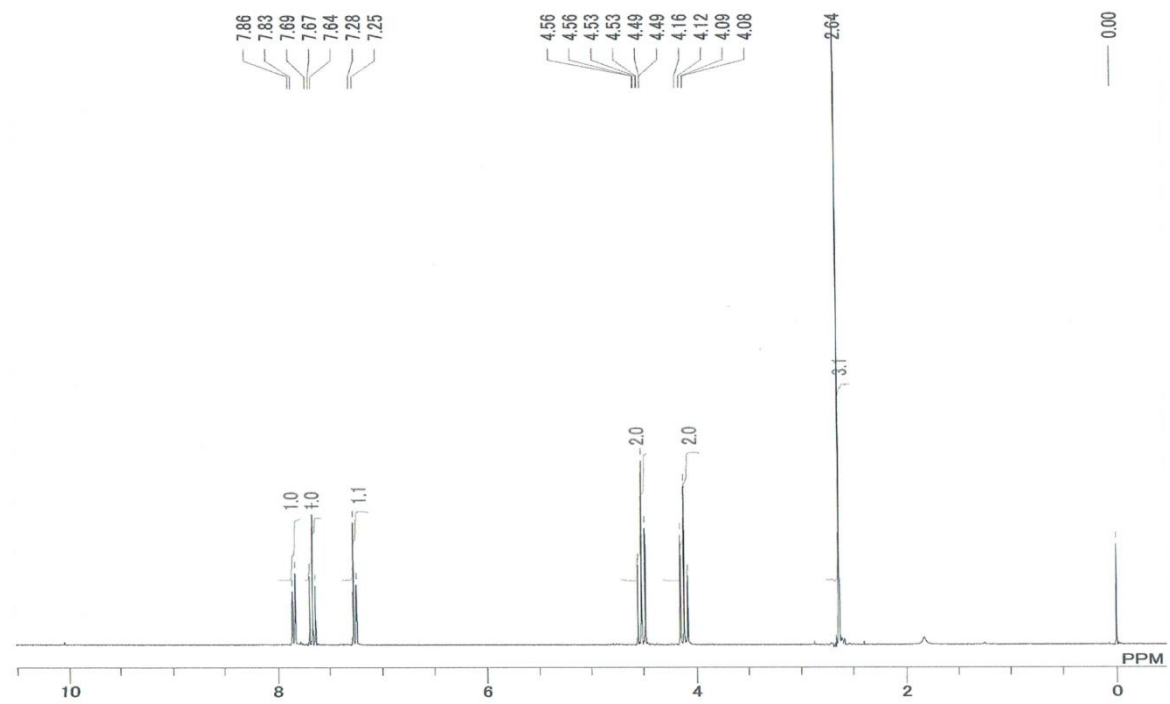
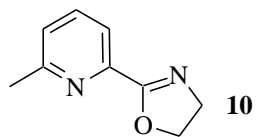
^1H NMR (CDCl_3 , 270.05 MHz)



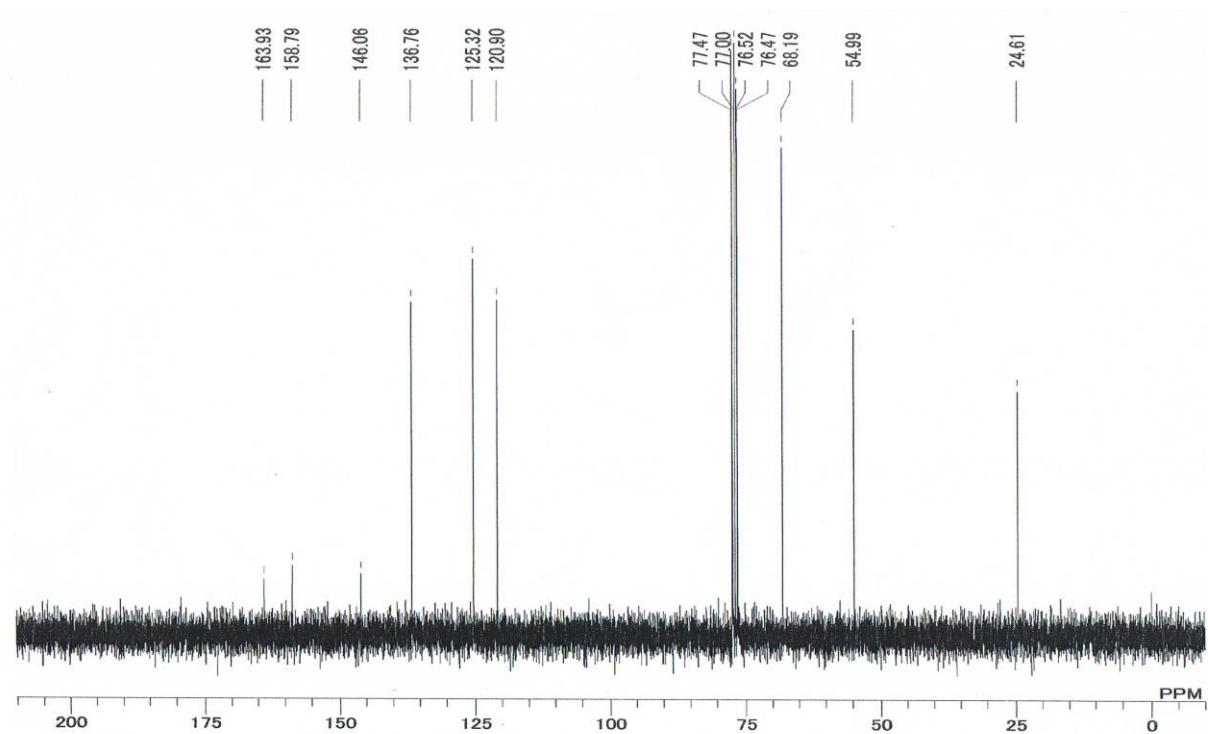
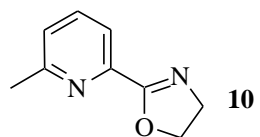
^{13}C NMR (CDCl_3 , 67.80 MHz)



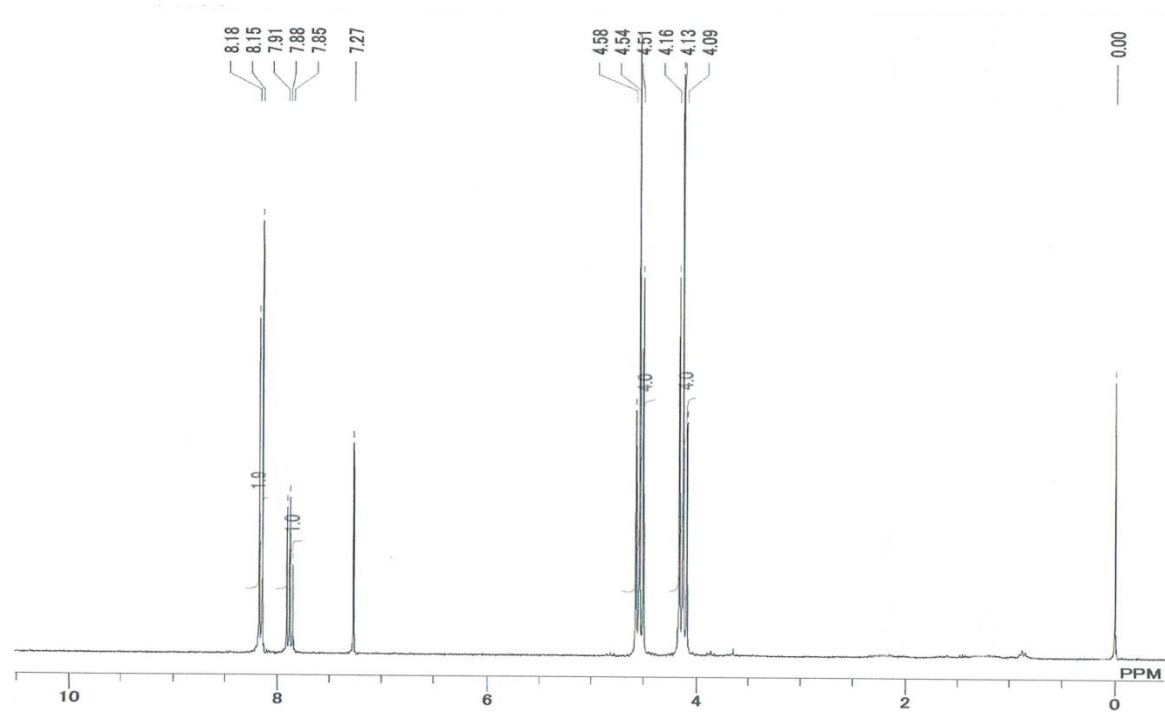
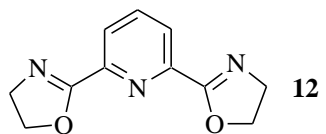
^1H NMR(CDCl_3 , 270.05 MHz)



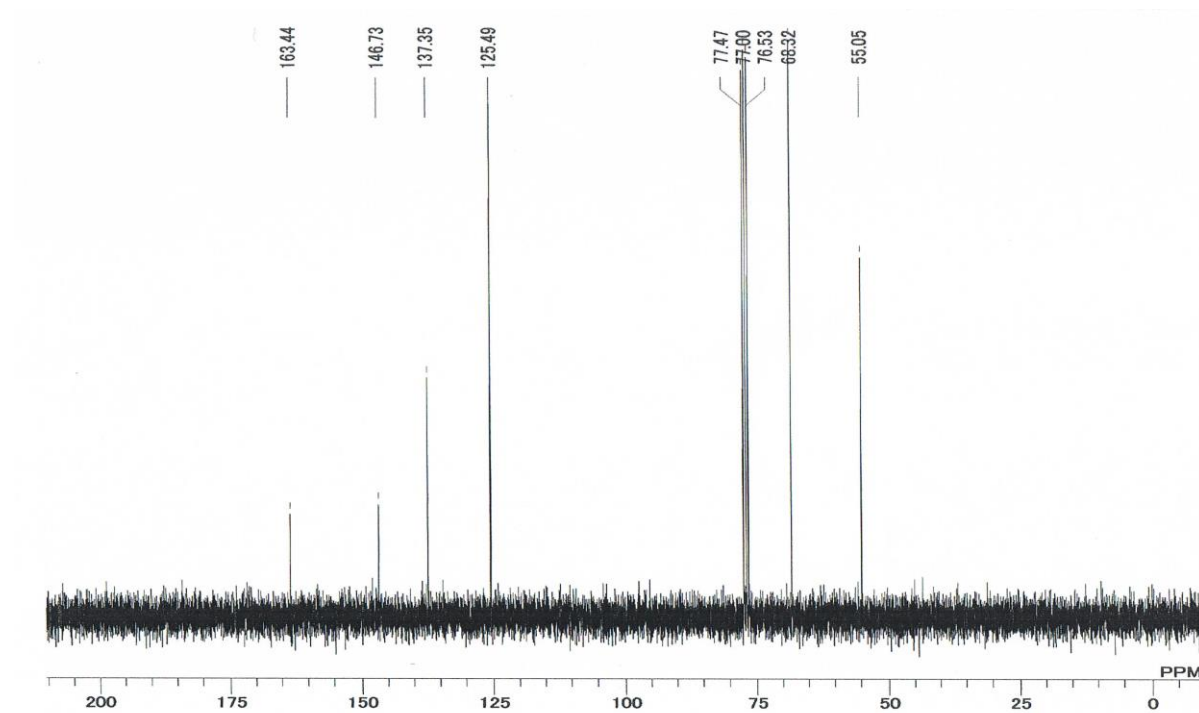
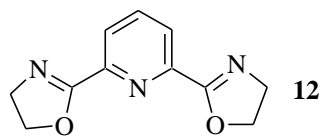
^{13}C NMR(CDCl_3 , 67.80 MHz)



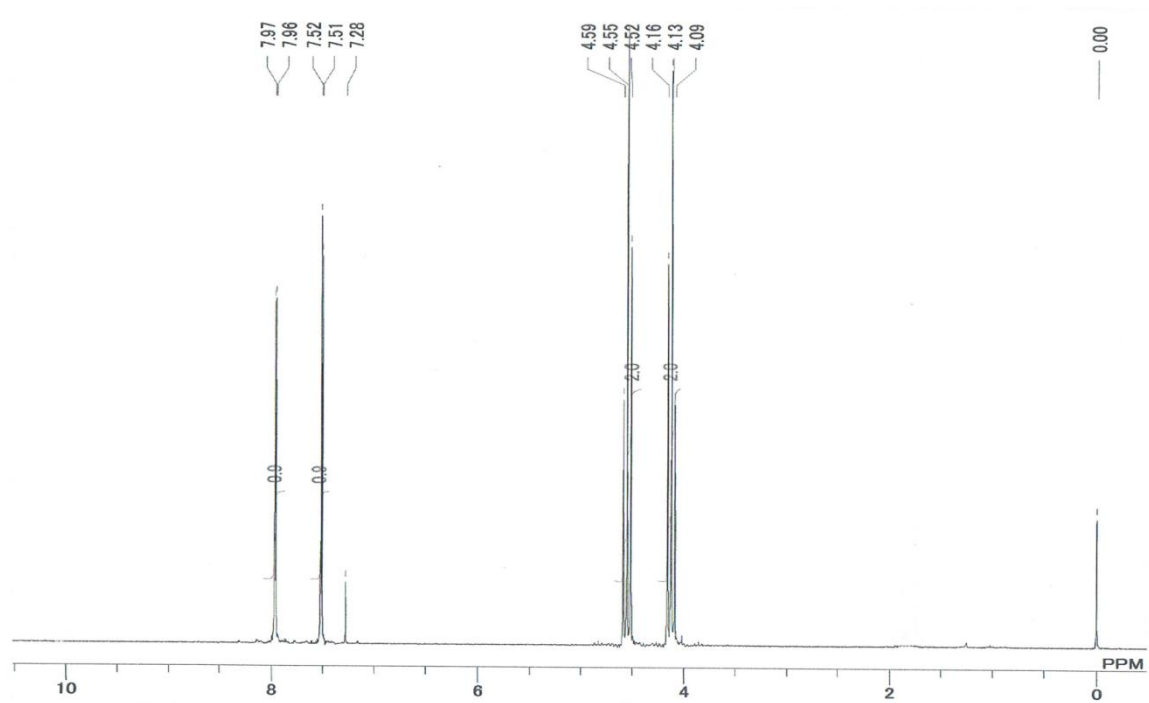
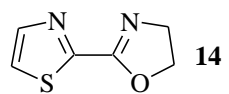
^1H NMR(CDCl_3 , 270.05 MHz)



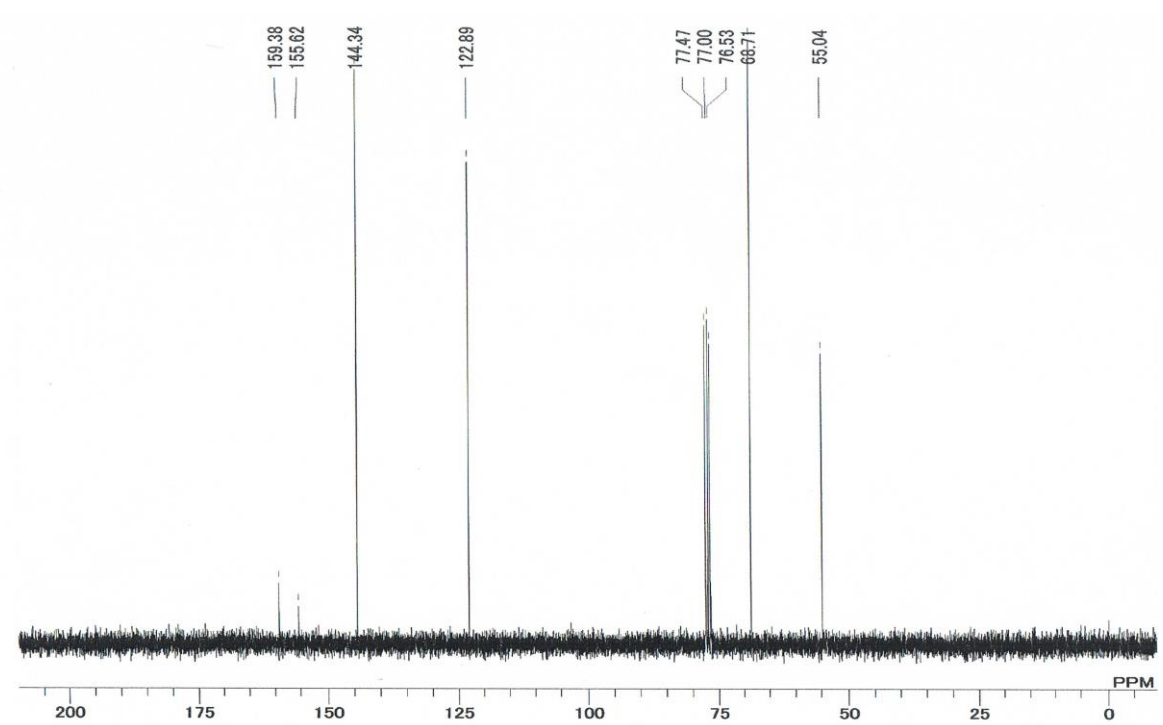
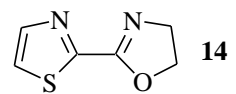
^{13}C NMR(CDCl_3 , 67.80 MHz)



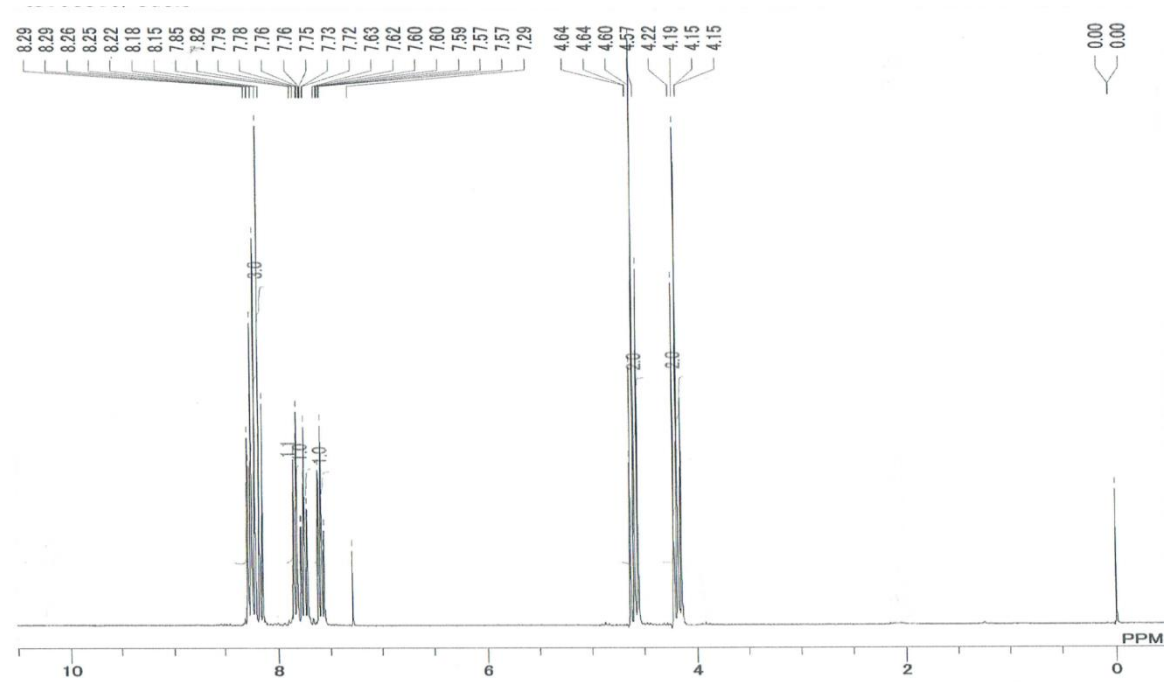
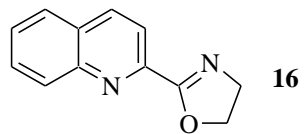
^1H NMR(CDCl_3 , 270.05 MHz)



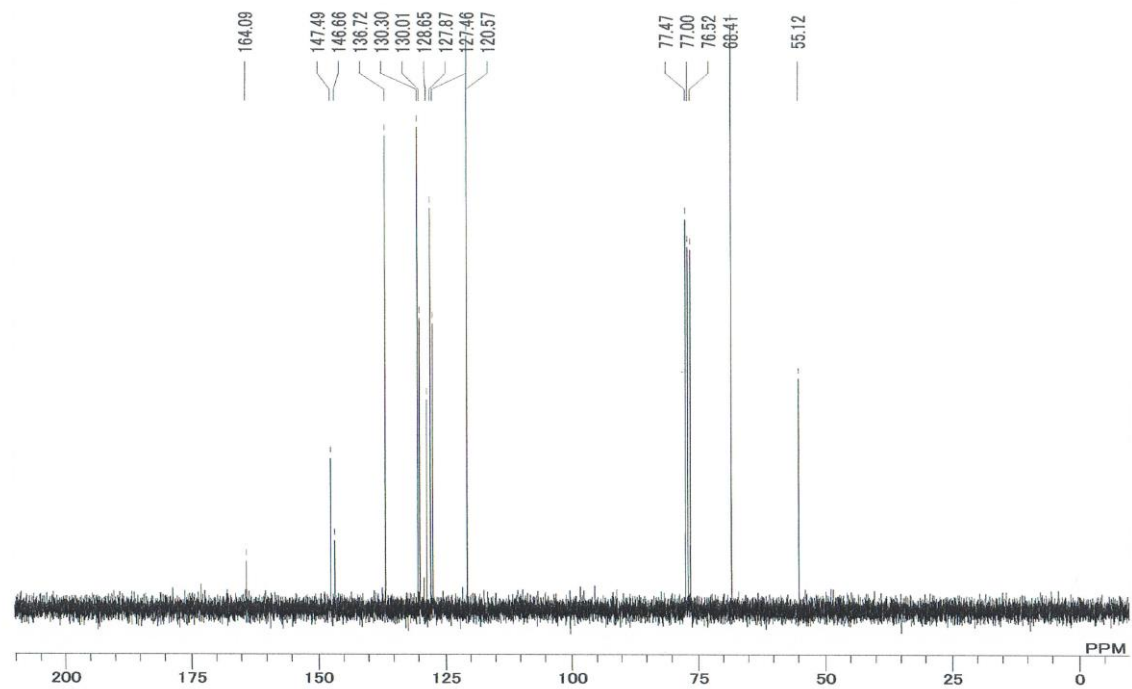
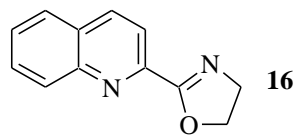
^{13}C NMR(CDCl_3 , 67.80 MHz)



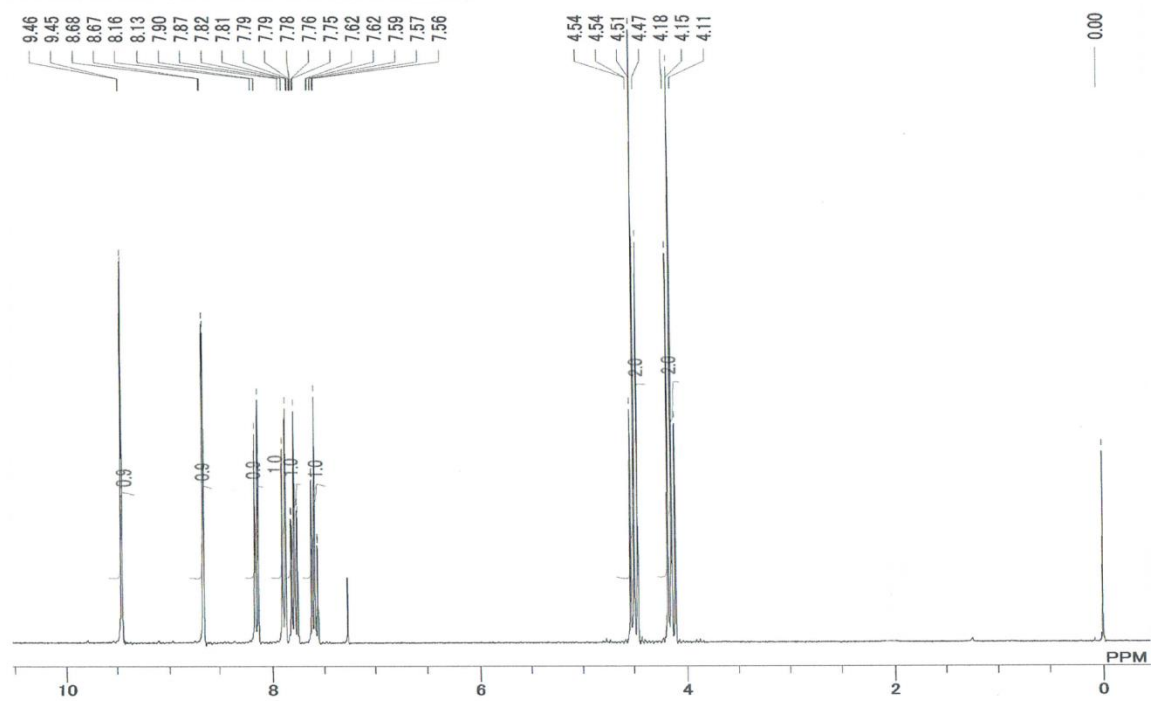
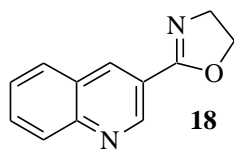
^1H NMR(CDCl_3 , 270.05 MHz)



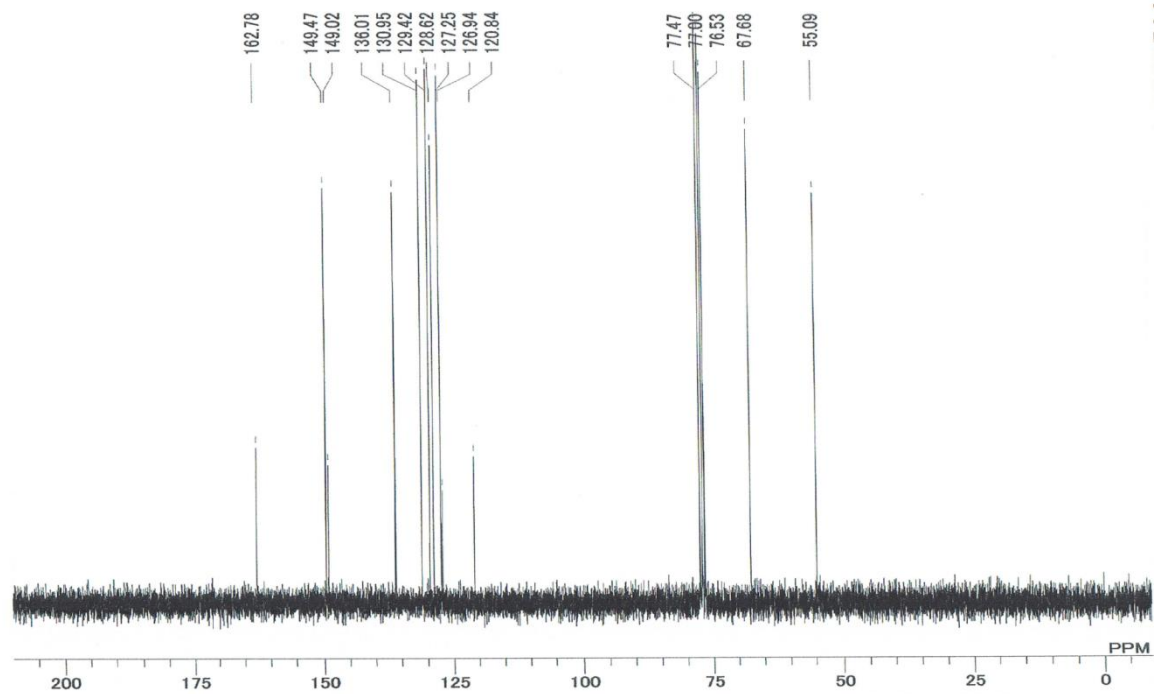
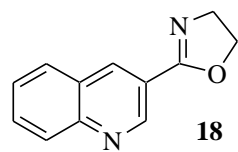
^{13}C NMR(CDCl₃, 67.80 MHz)



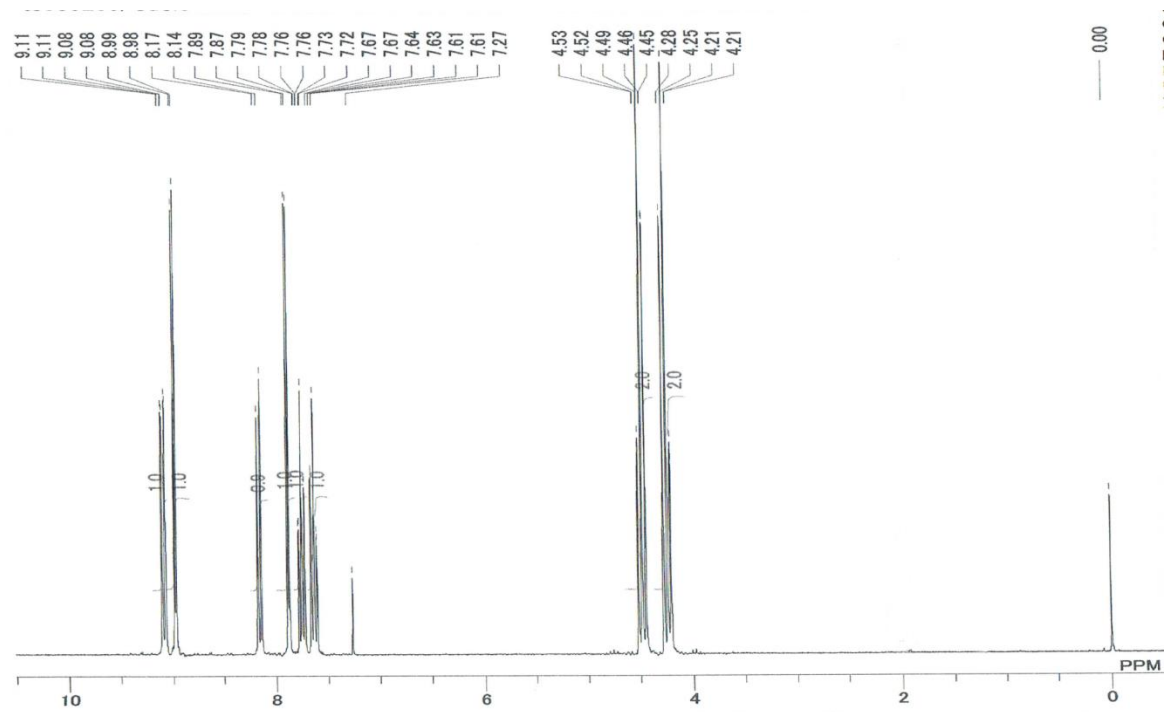
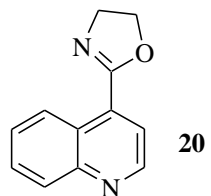
$^1\text{H NMR}(\text{CDCl}_3, 270.05 \text{ MHz})$



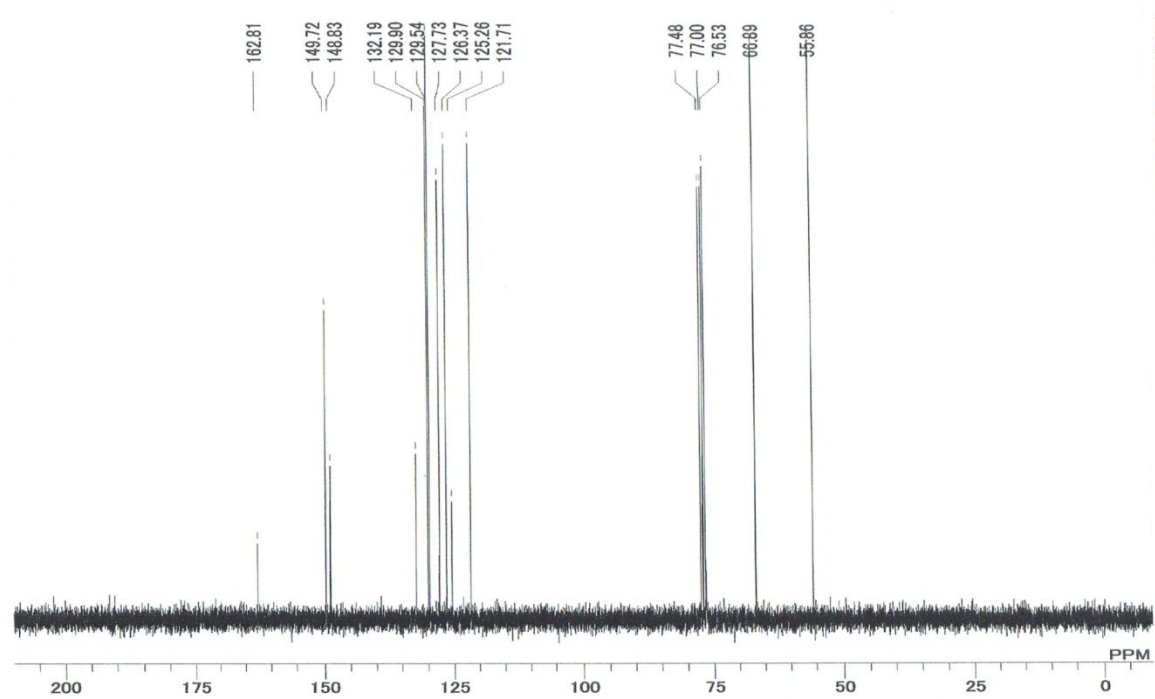
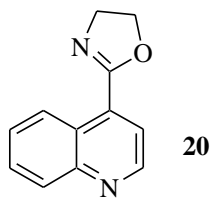
^{13}C NMR(CDCl_3 , 67.80 MHz)



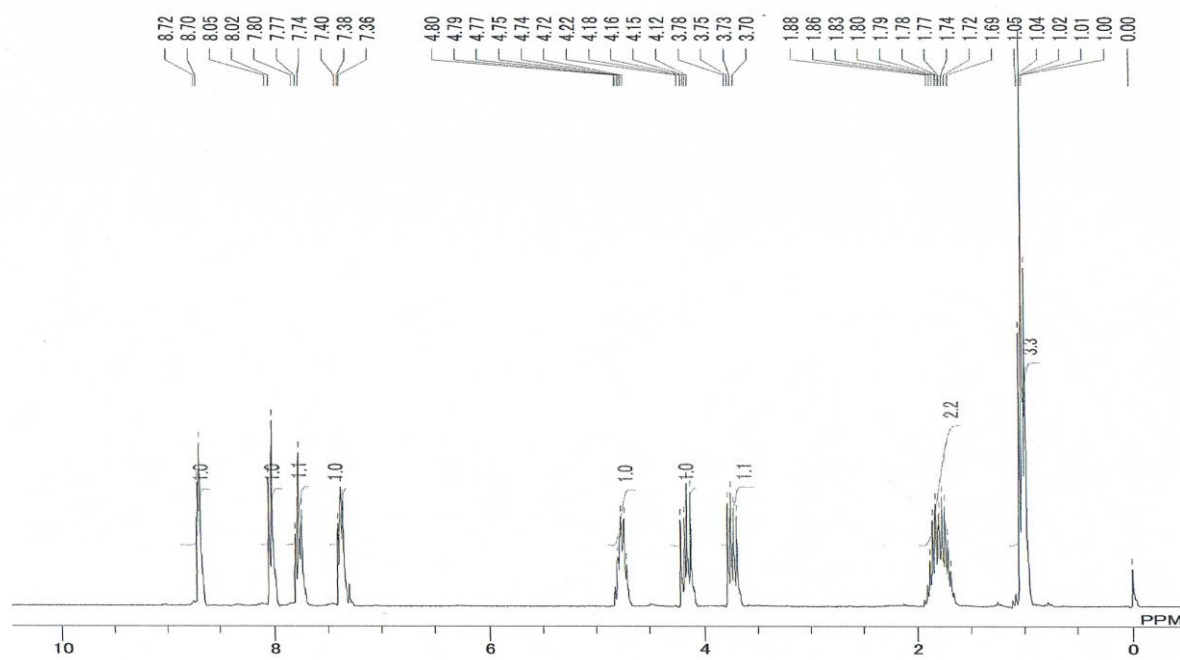
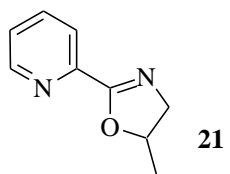
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



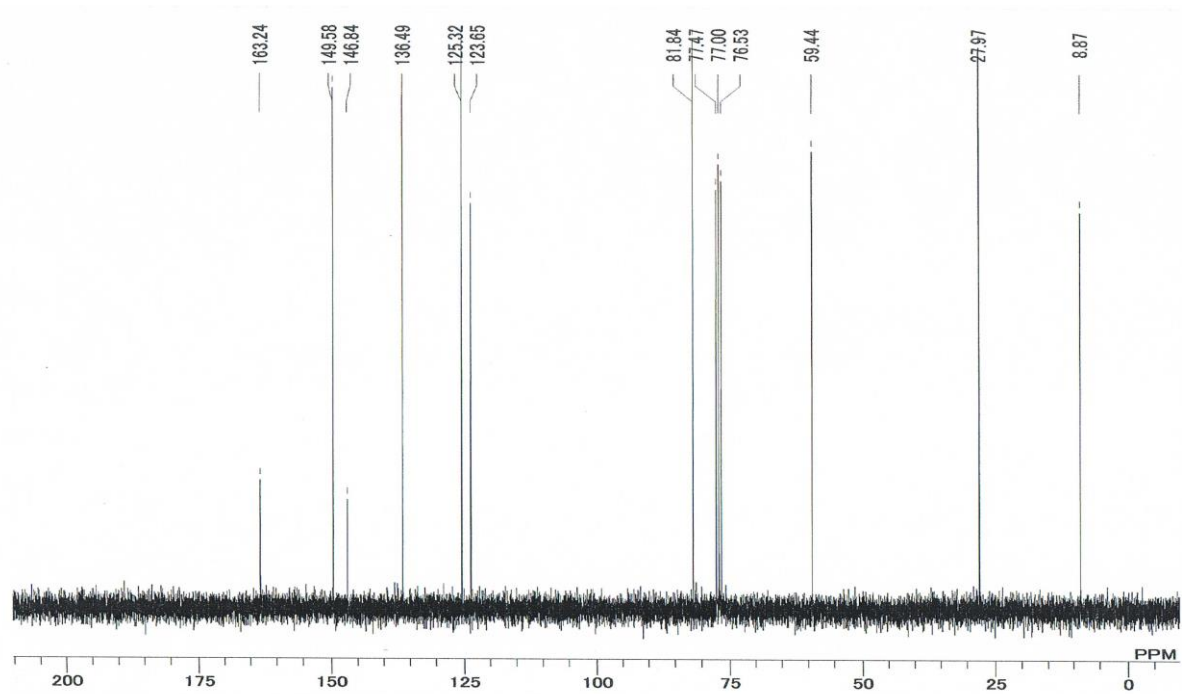
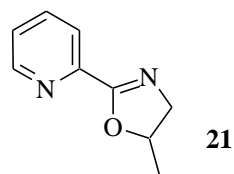
^{13}C NMR(CDCl_3 , 67.80 MHz)



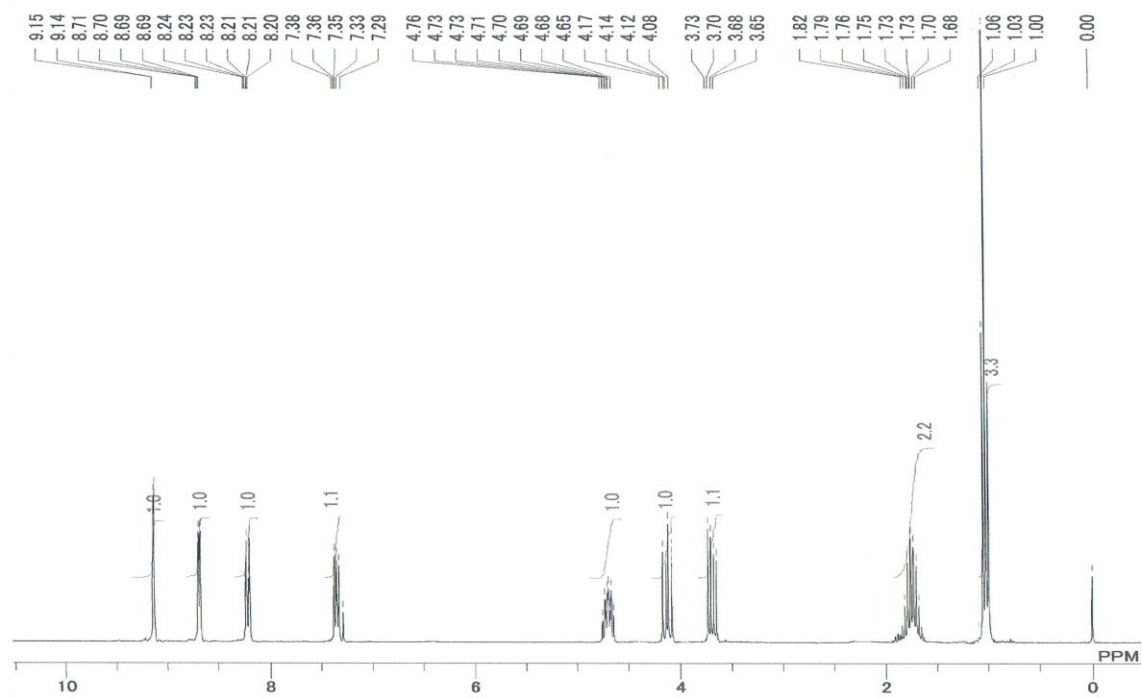
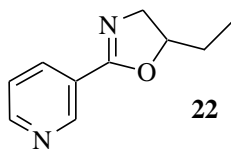
^1H NMR(CDCl_3 , 270.05 MHz)



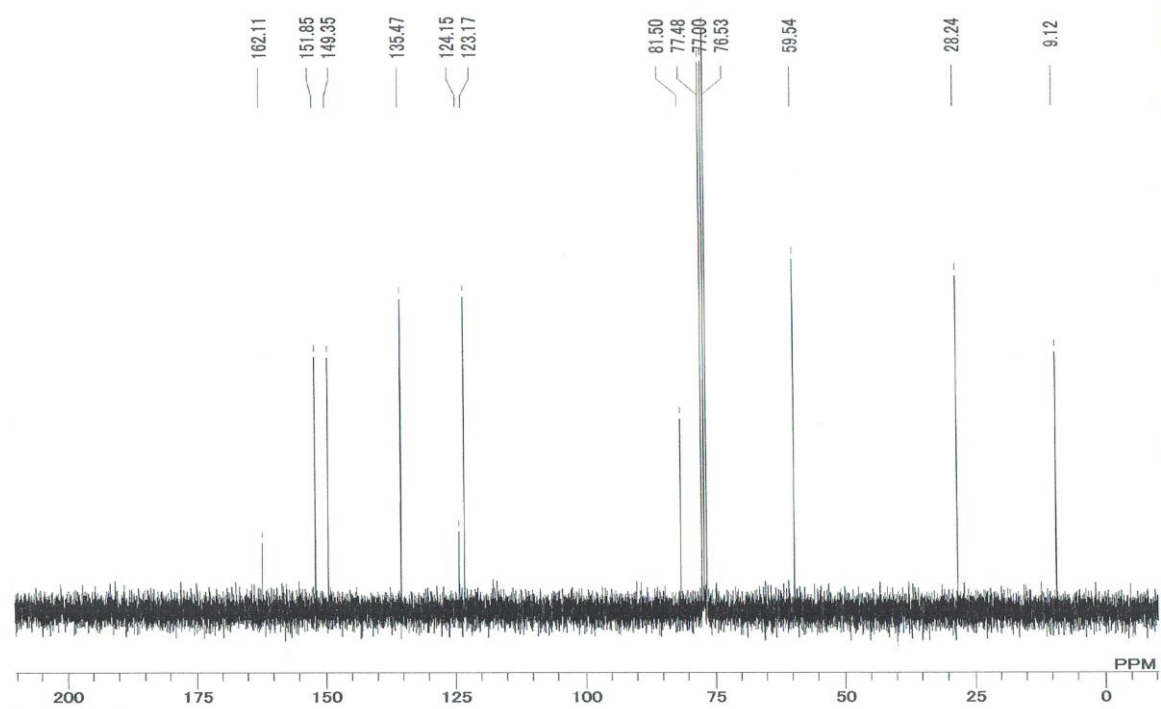
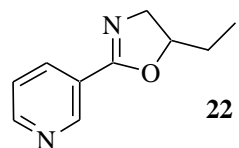
^{13}C NMR(CDCl_3 , 67.80 MHz)



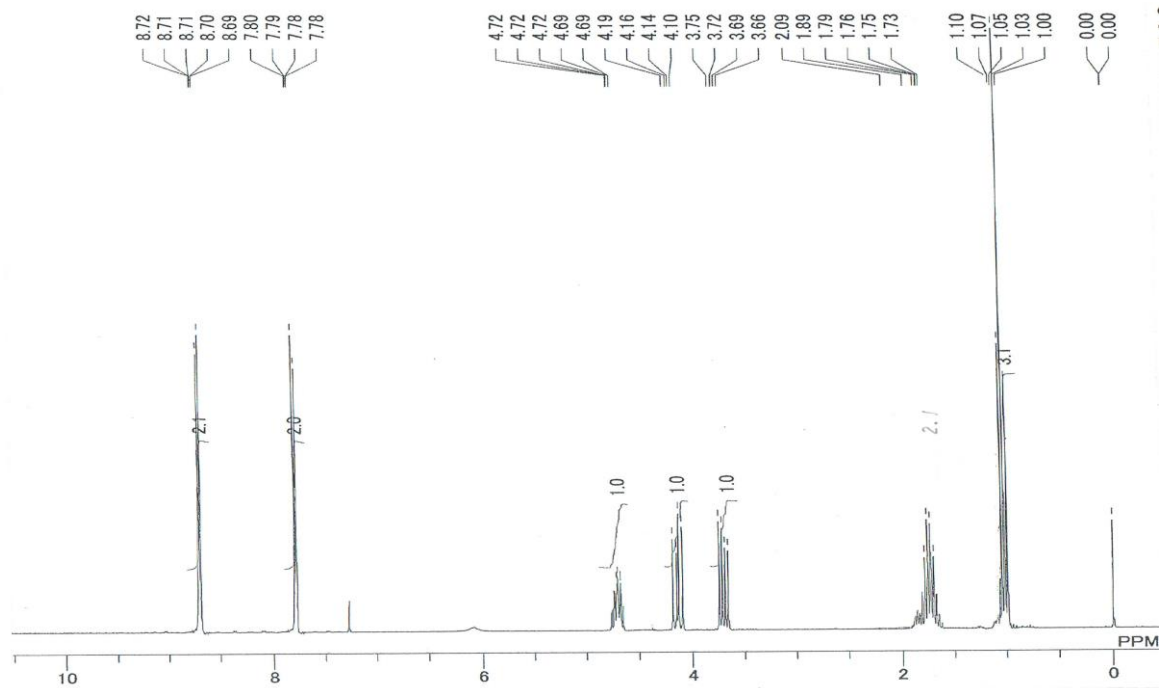
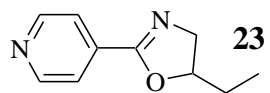
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



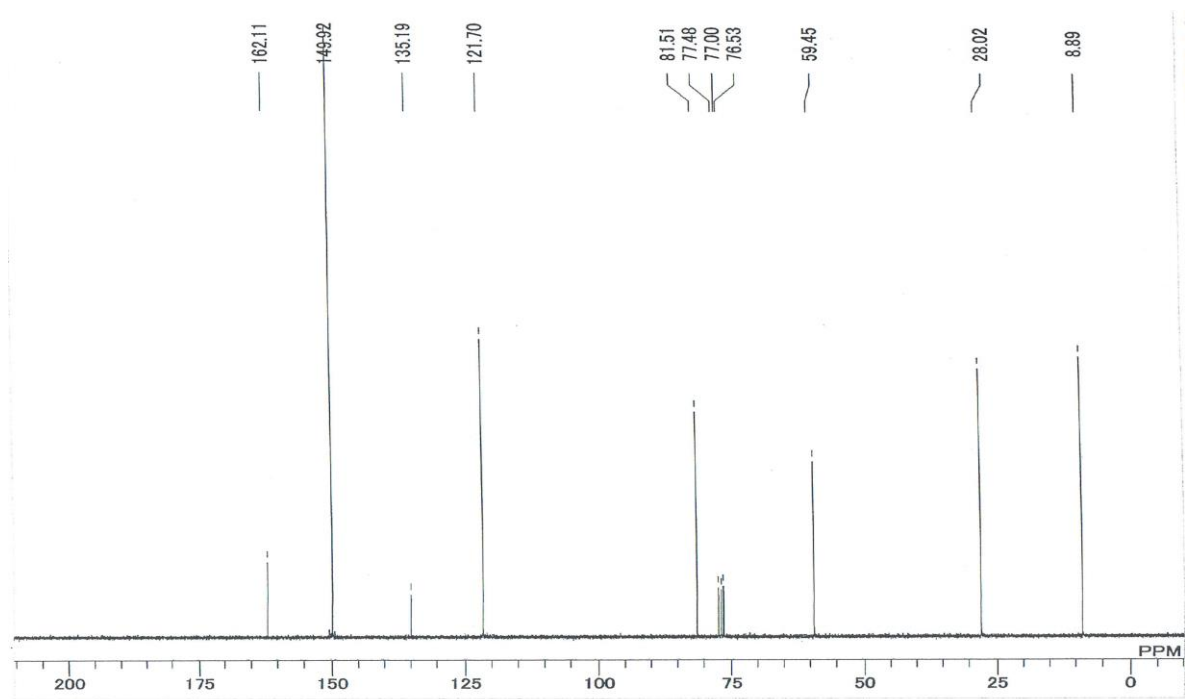
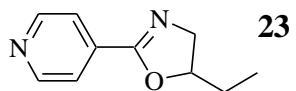
^{13}C NMR(CDCl_3 , 67.80 MHz)



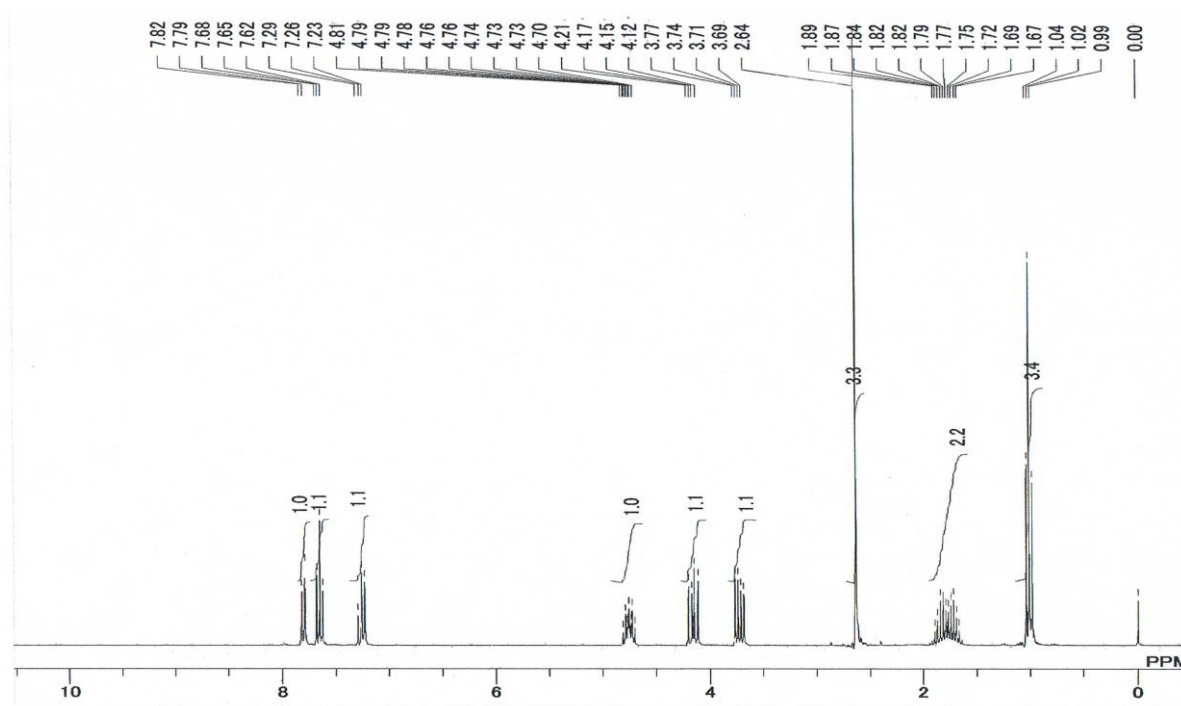
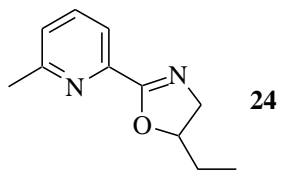
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



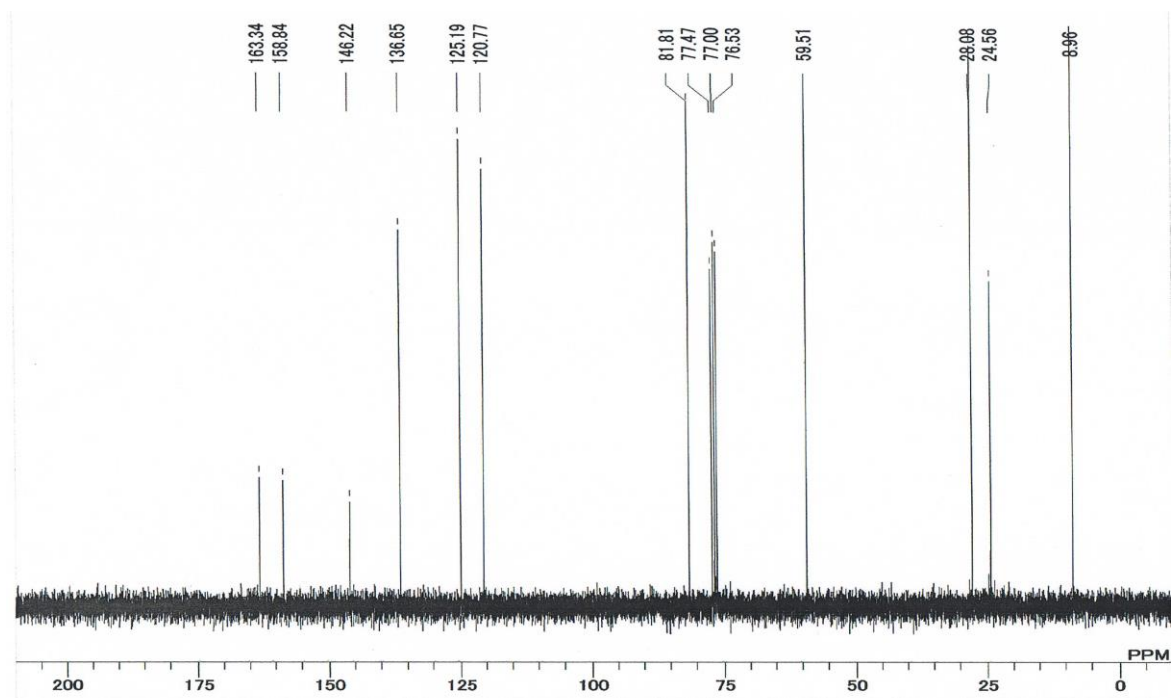
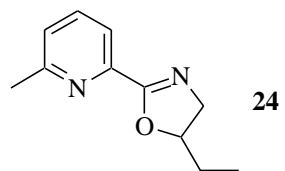
^{13}C NMR(CDCl_3 , 67.80 MHz)



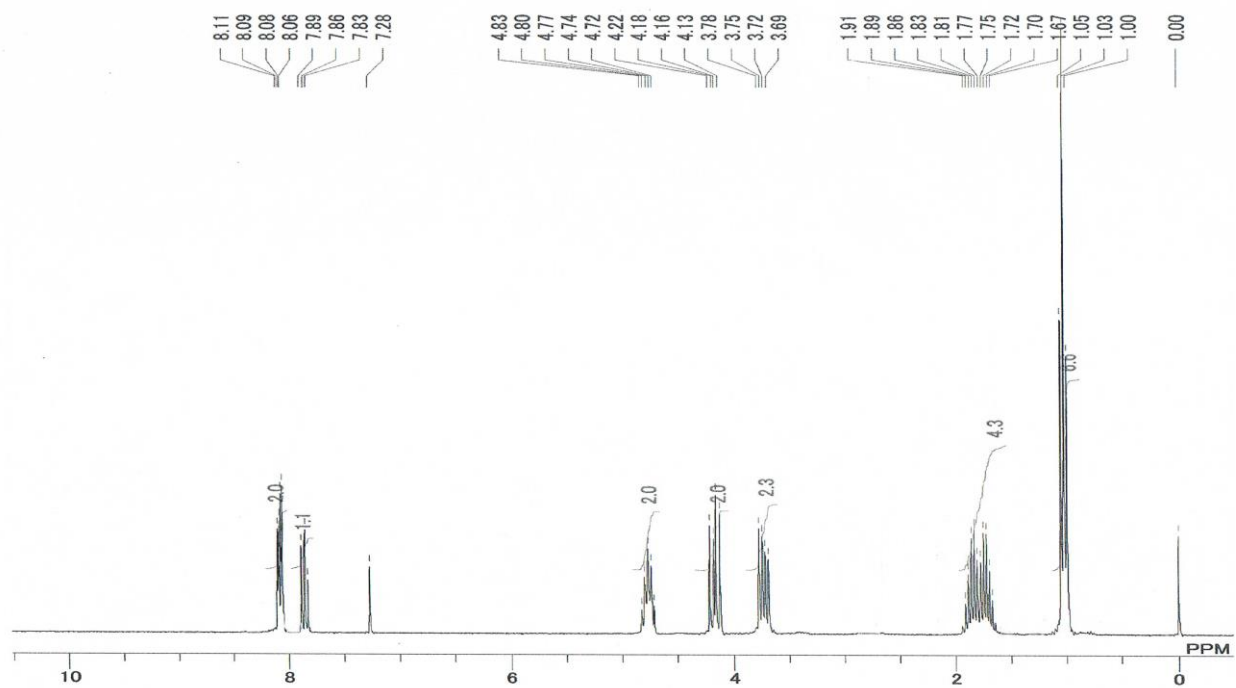
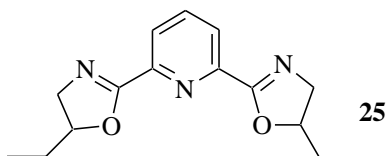
^1H NMR(CDCl_3 , 270.05 MHz)



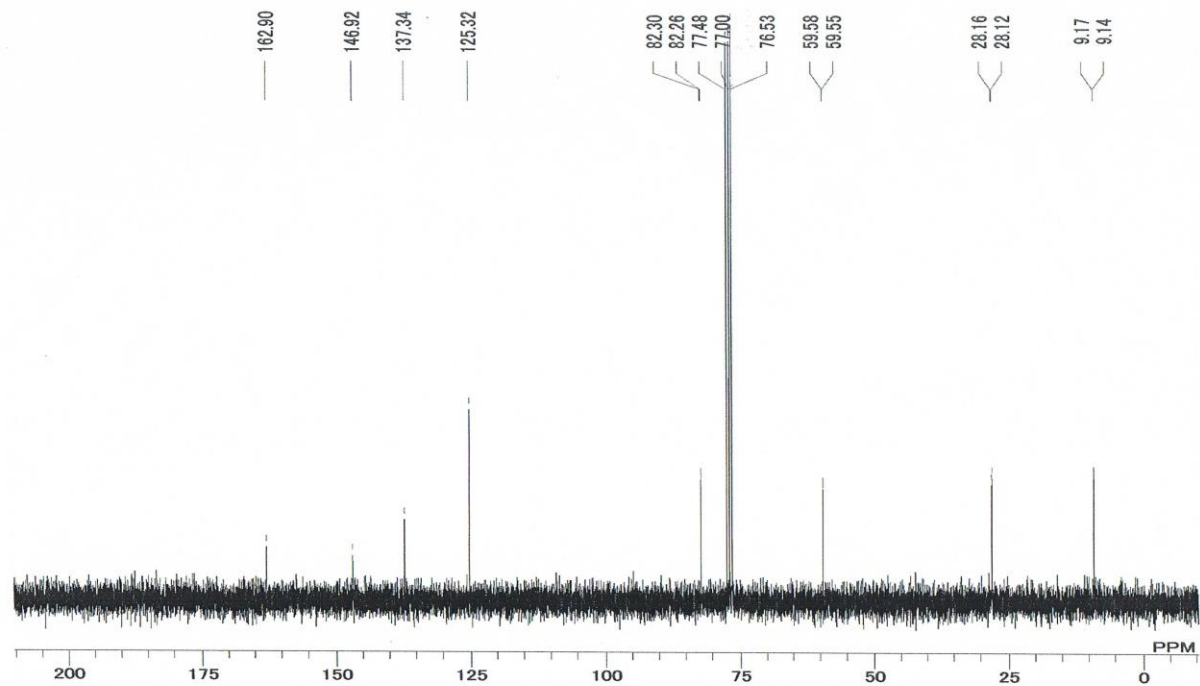
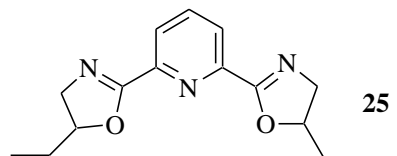
^{13}C NMR(CDCl_3 , 67.80 MHz)



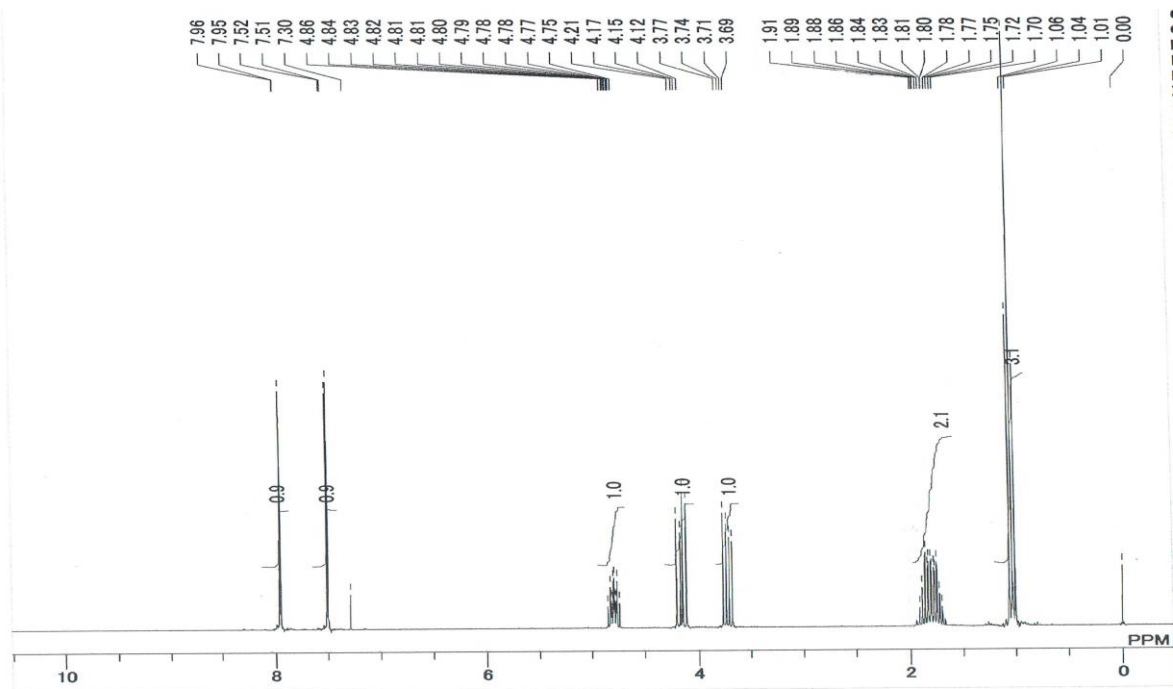
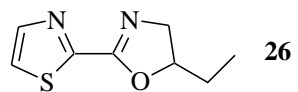
^1H NMR(CDCl_3 , 270.05 MHz)



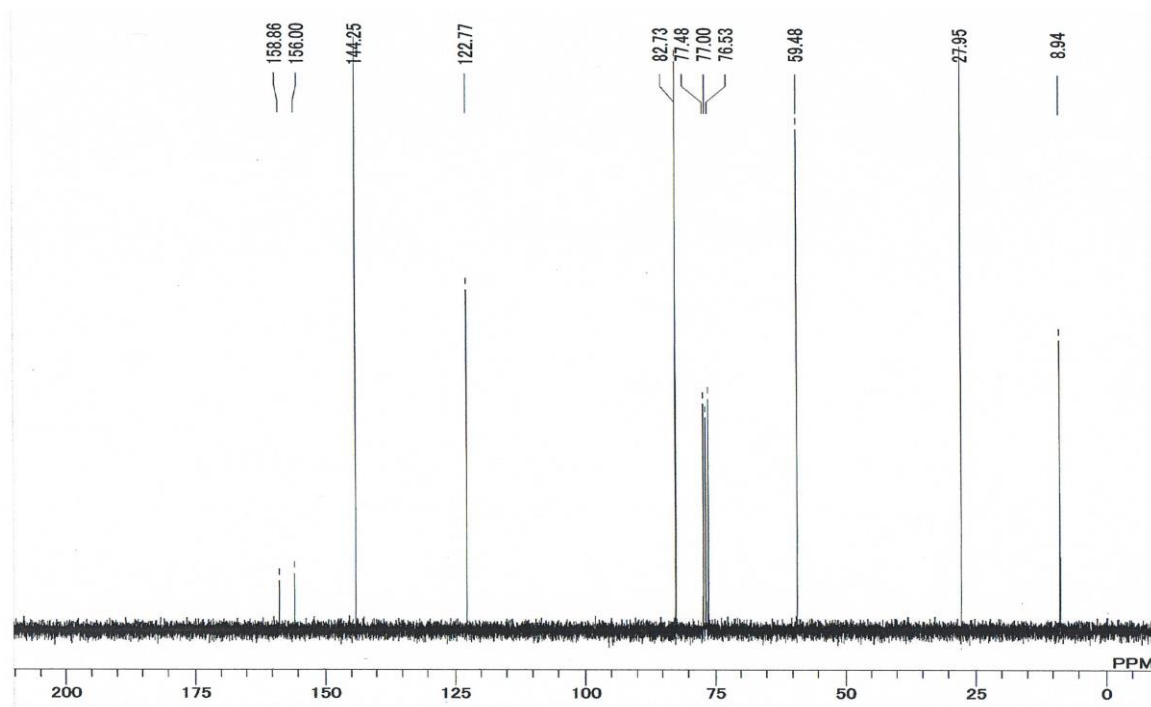
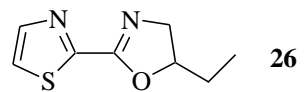
^{13}C NMR(CDCl_3 , 67.80 MHz)



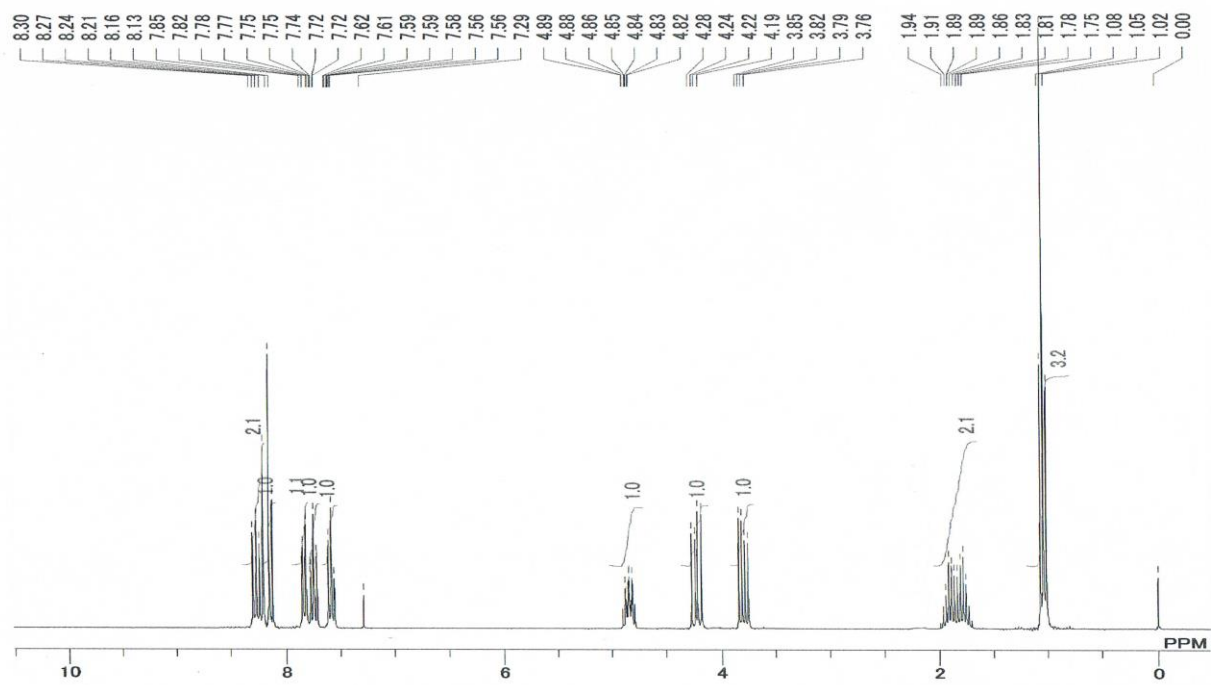
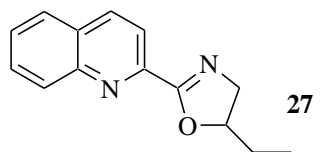
^1H NMR(CDCl_3 , 270.05 MHz)



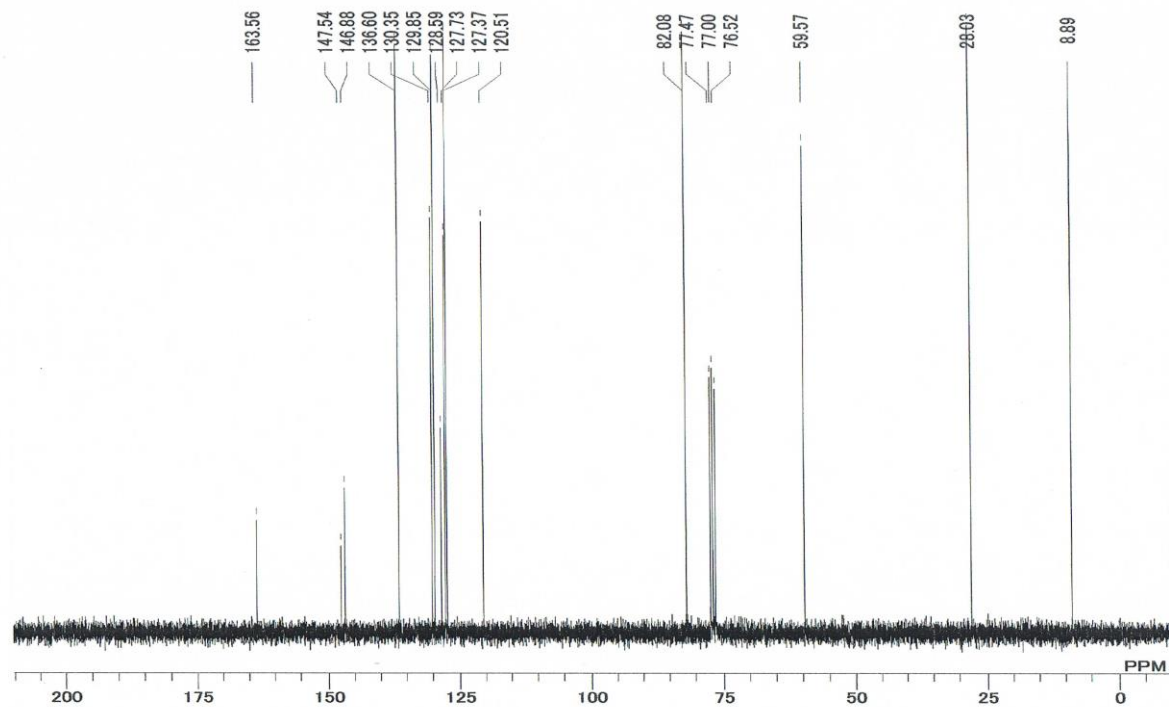
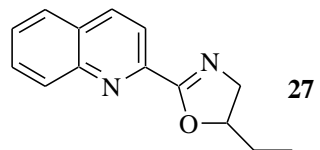
^{13}C NMR(CDCl_3 , 67.80 MHz)



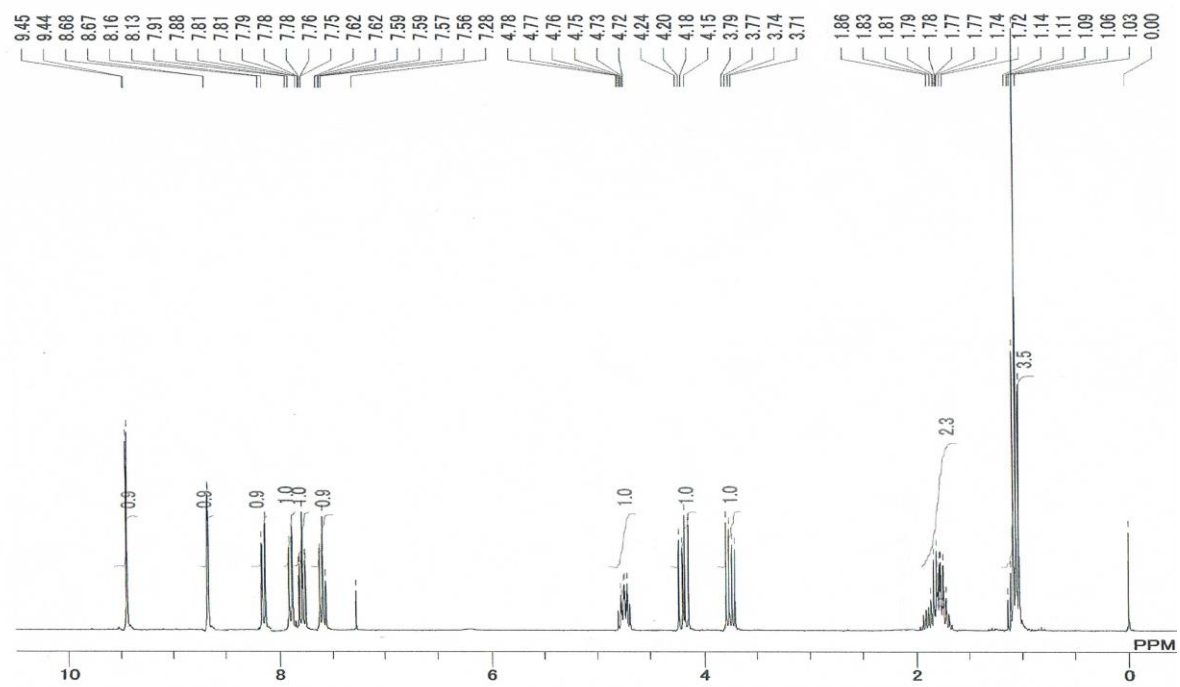
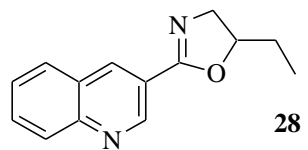
^1H NMR(CDCl_3 , 270.05 MHz)



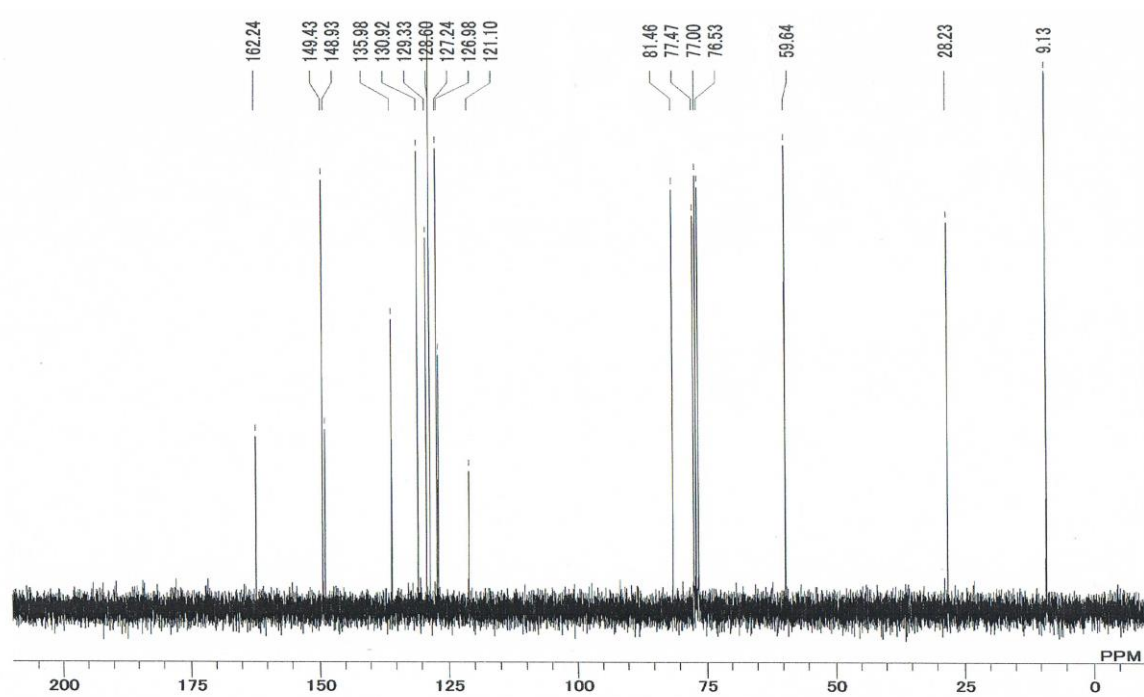
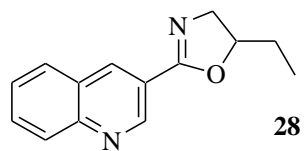
^{13}C NMR(CDCl_3 , 67.80 MHz)



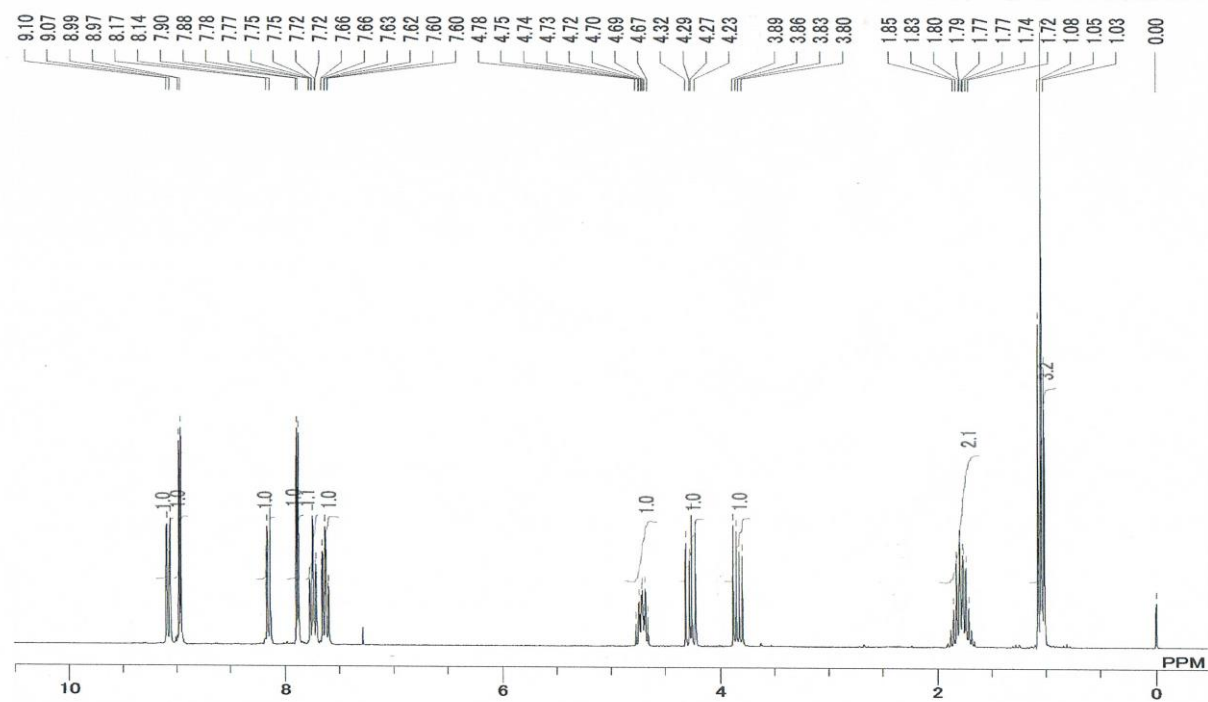
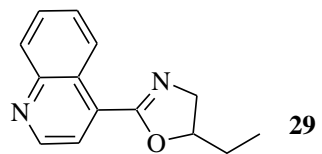
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



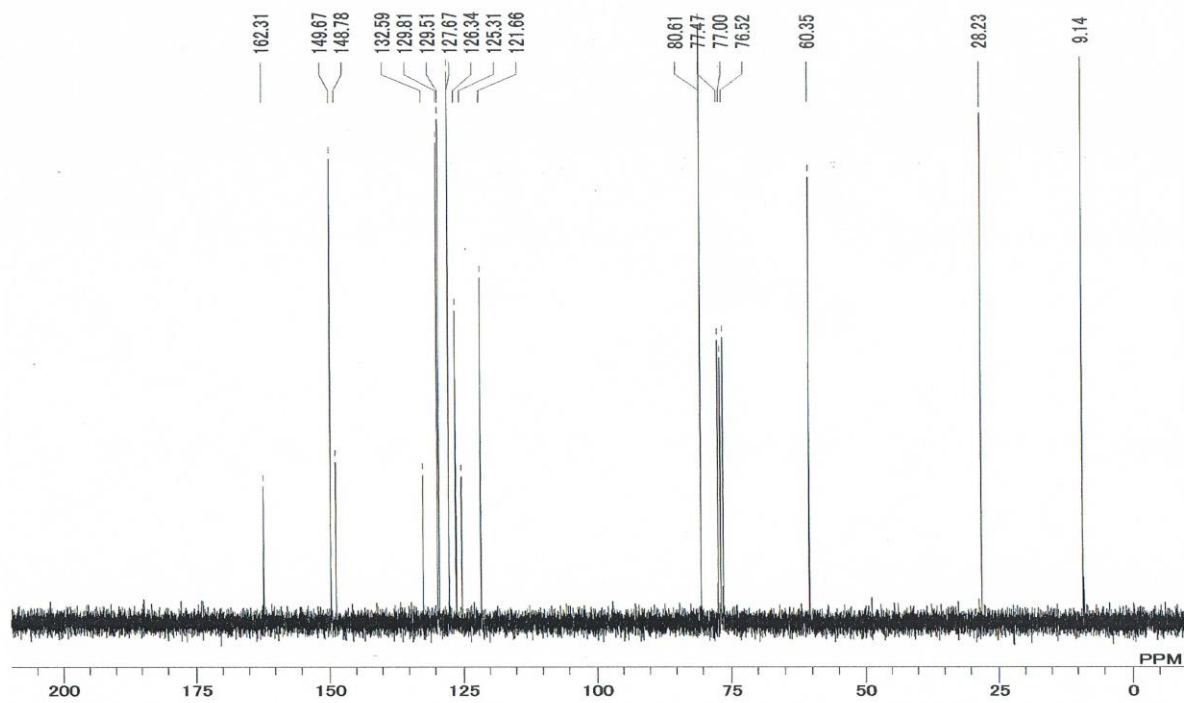
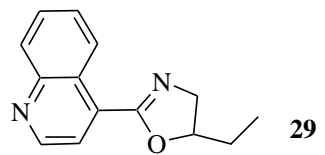
^{13}C NMR(CDCl_3 , 67.80 MHz)



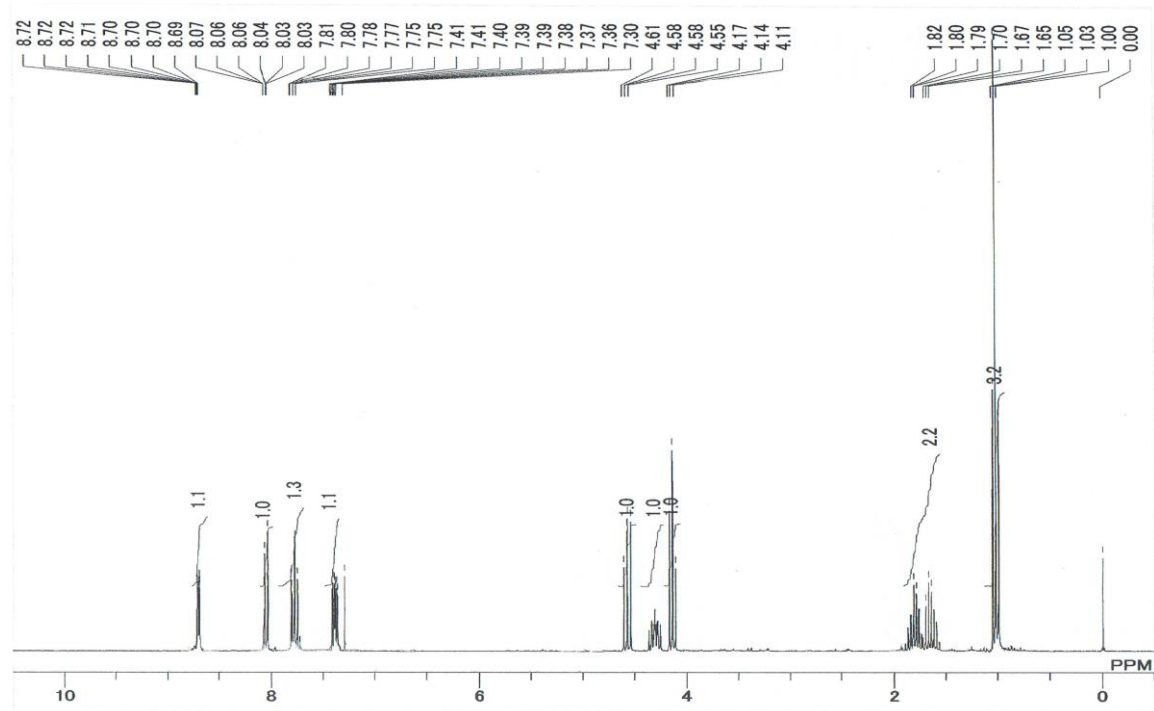
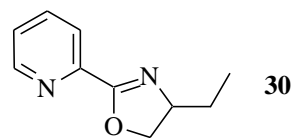
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



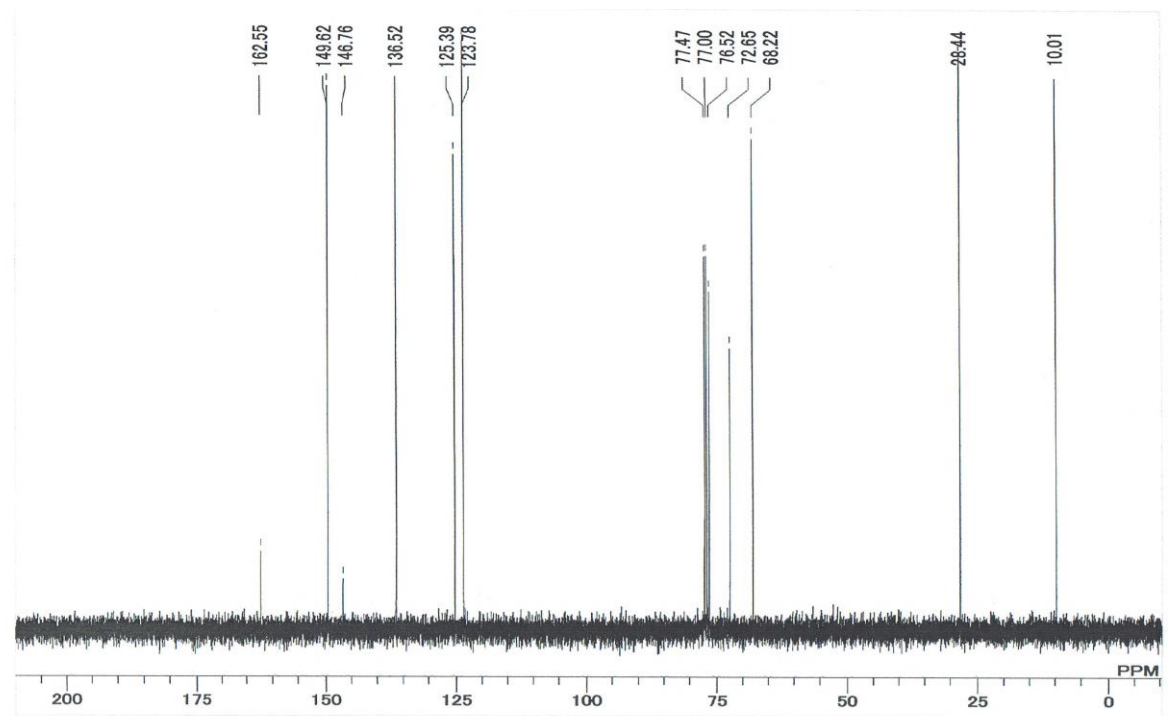
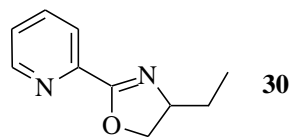
^{13}C NMR(CDCl₃, 67.80 MHz)



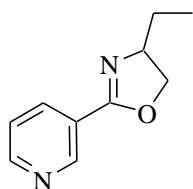
^1H NMR(CDCl_3 , 270.05 MHz)



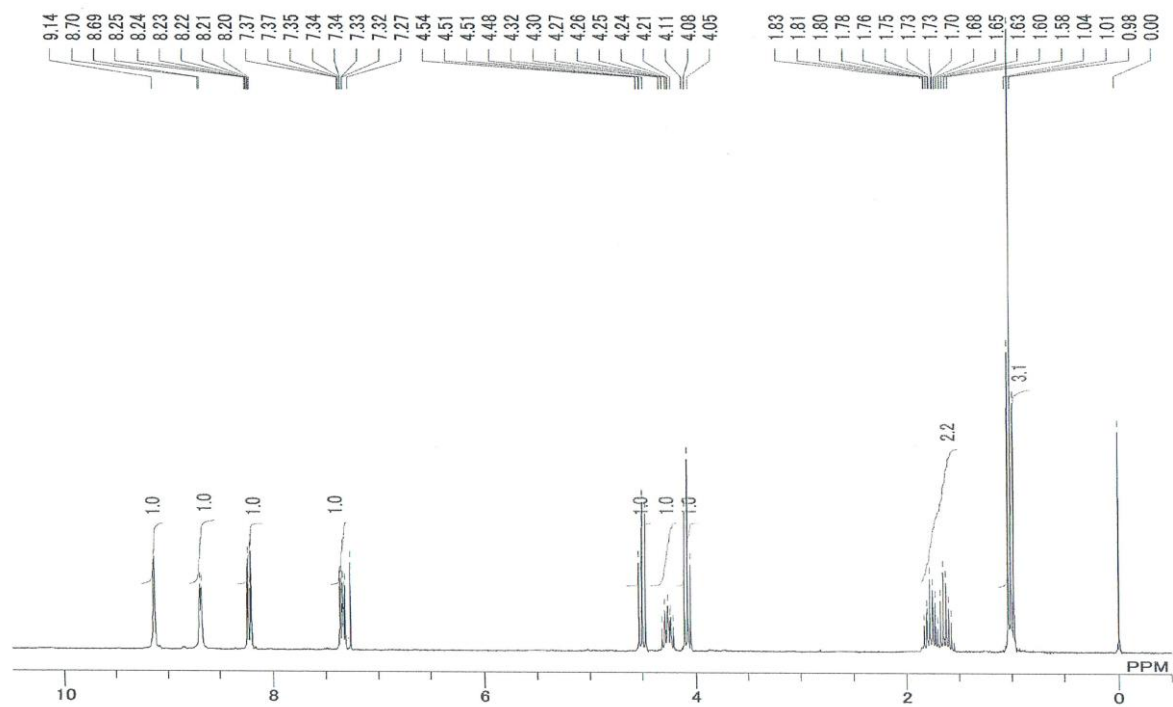
^{13}C NMR(CDCl_3 , 67.80 MHz)



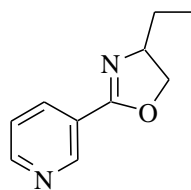
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



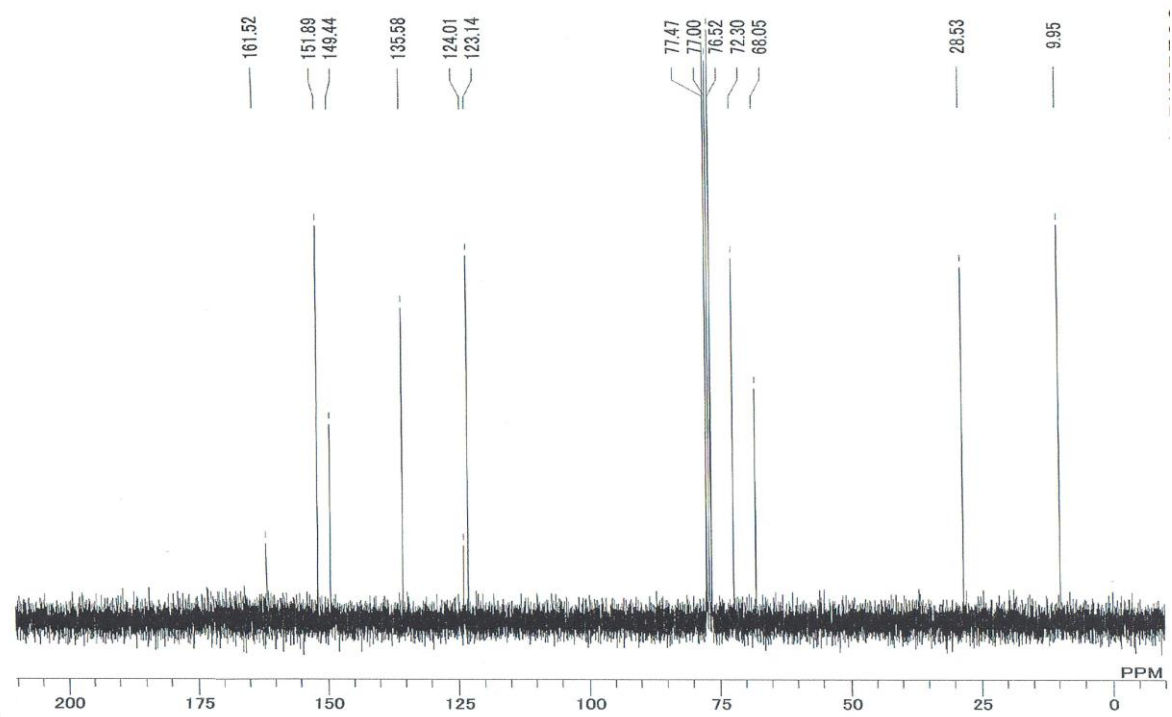
31



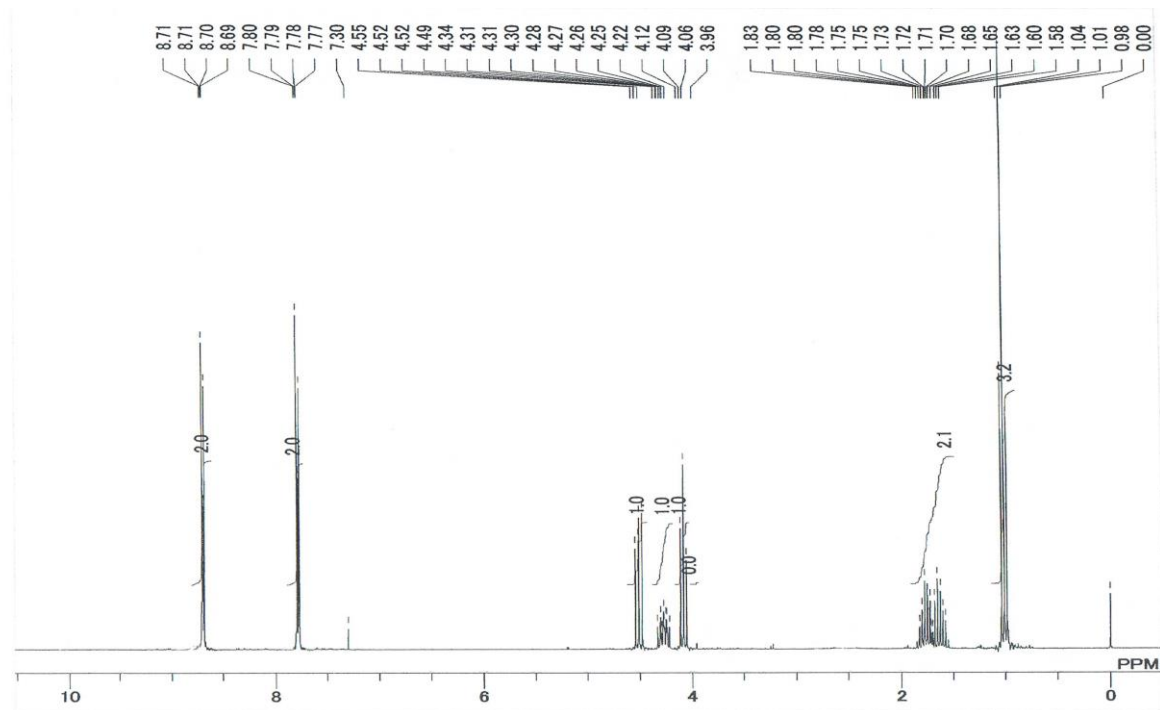
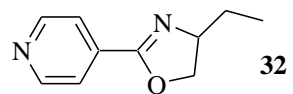
^{13}C NMR(CDCl_3 , 67.80 MHz)



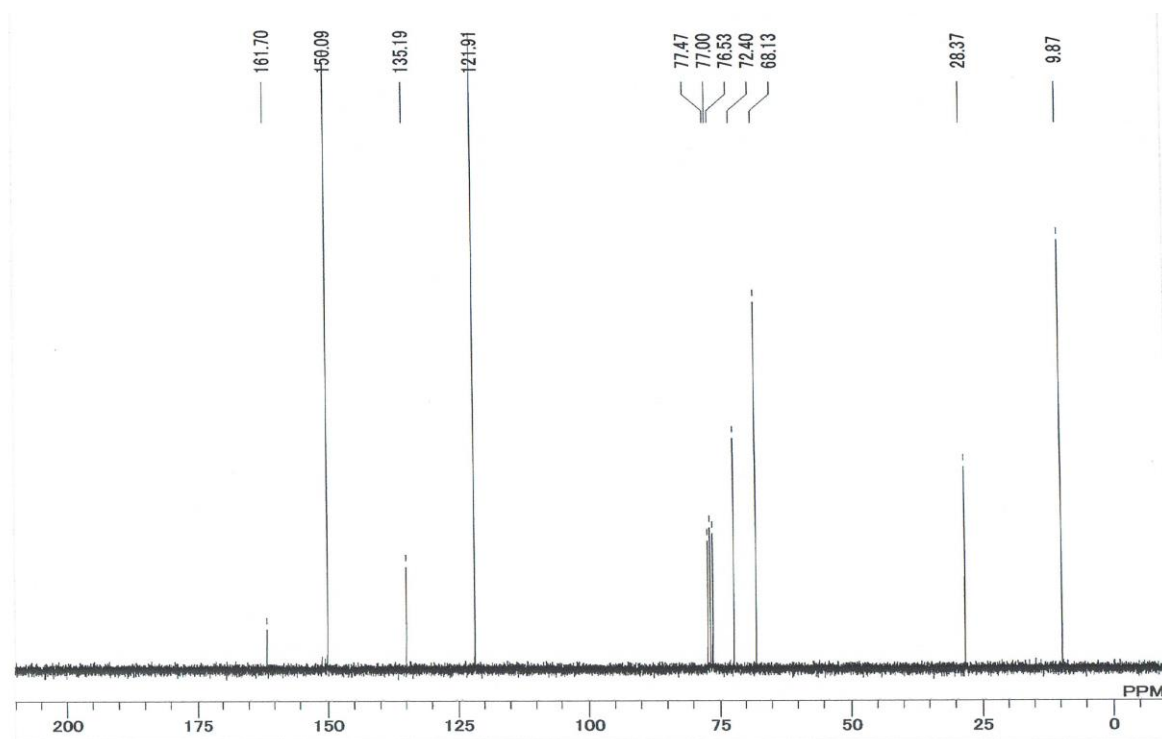
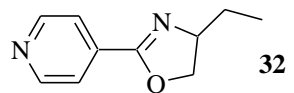
31



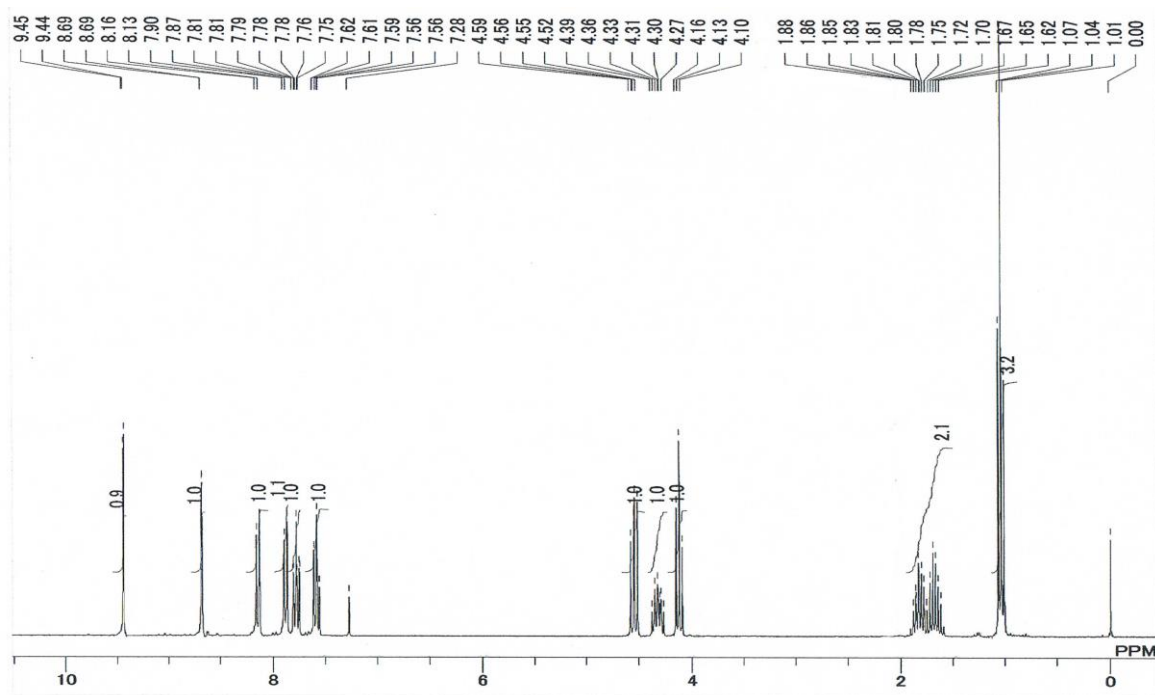
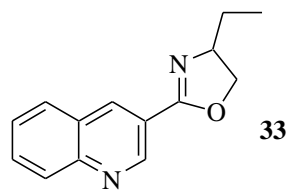
^1H NMR(CDCl_3 , 270.05 MHz)



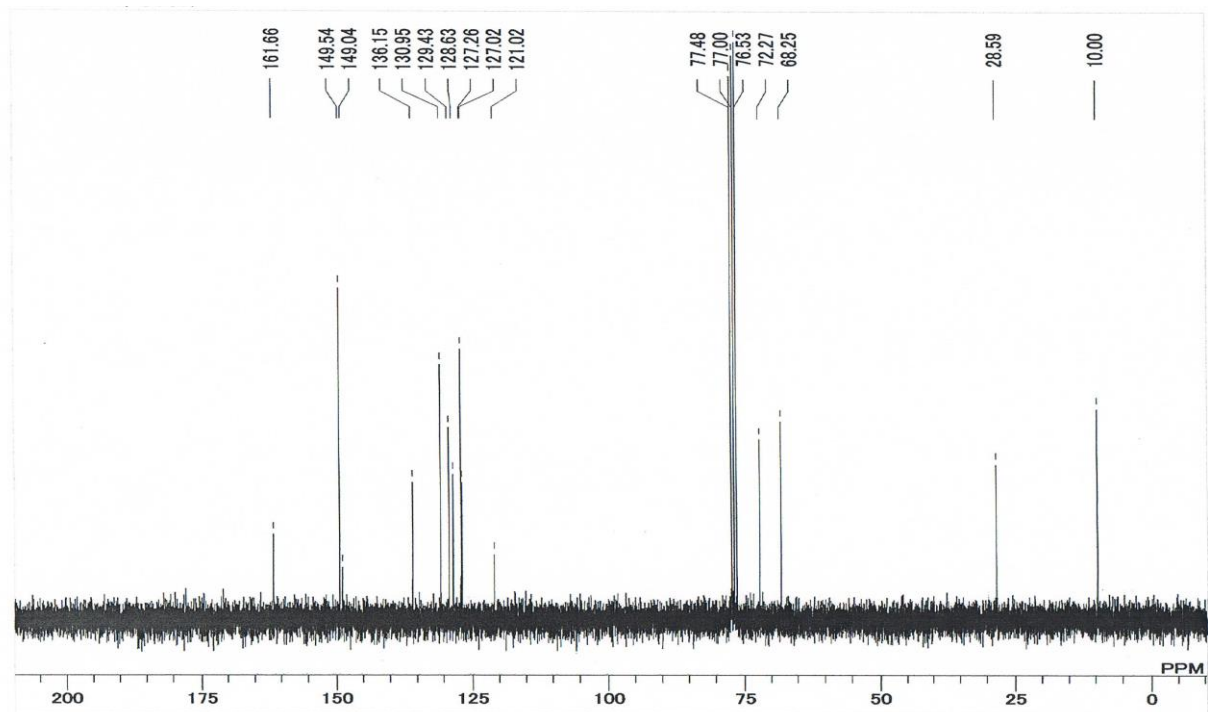
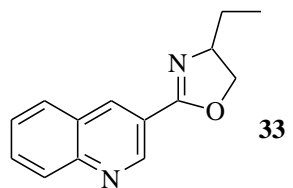
^{13}C NMR(CDCl_3 , 67.80 MHz)



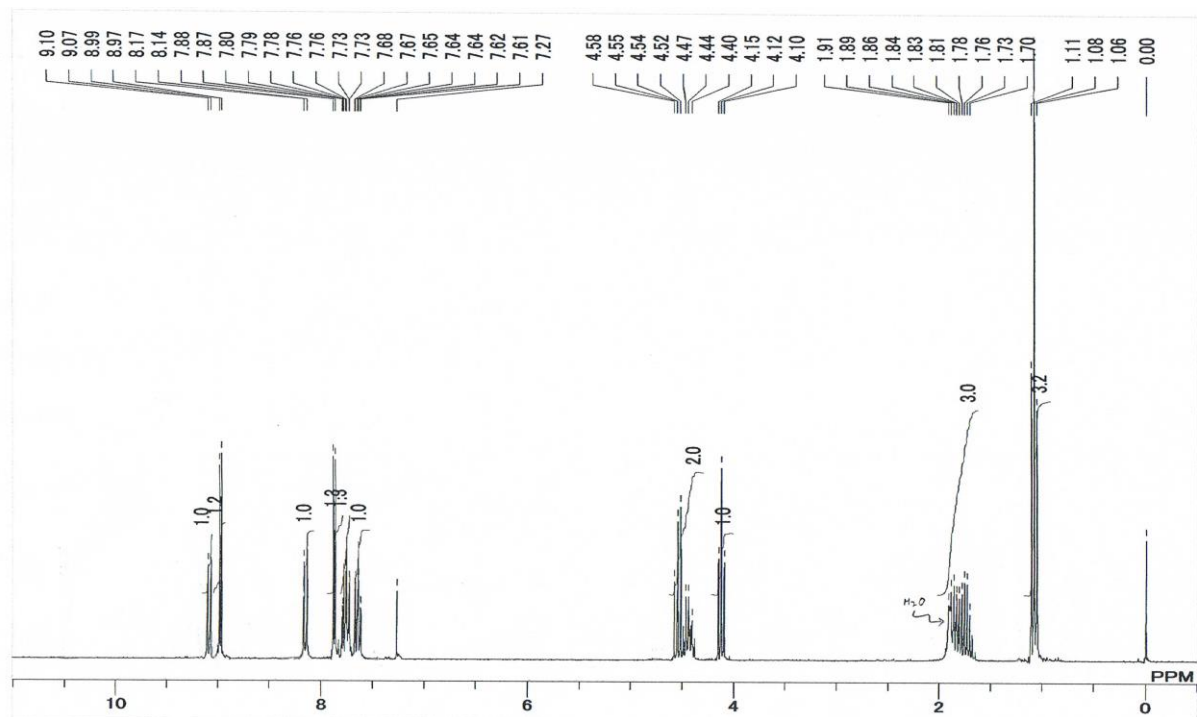
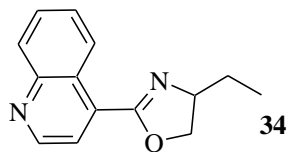
$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



^{13}C NMR(CDCl₃, 67.80 MHz)



$^1\text{H NMR}$ (CDCl_3 , 270.05 MHz)



^{13}C NMR(CDCl₃, 67.80 MHz)

