

Supporting Information for

**Copper(0)-Induced Deselenative Insertion of *N,N*-Disubstituted Selenoamides
into Acetylenic C–H Bond Leading to Propargylamines**

*Takenori Mitamura and Akiya Ogawa**

*Department of Applied Chemistry, Graduate School of Engineering, Osaka Prefecture University,
1-1 Gakuen-cho, Nakaku, Sakai, Osaka 599-8531, Japan*

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General Comments.

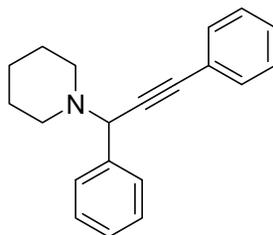
¹H NMR spectra were recorded on a JEOL JNM-AL spectrometer using CDCl₃ as the solvent with tetramethylsilane (TMS) as the internal standard. ¹³C NMR spectra were obtained on a JEOL JNM-AL spectrometer using CDCl₃ as the solvent. Chemical shifts in ¹³C NMR were measured relative to CDCl₃ by using δ 77.0 ppm. Infrared spectra were determined on a Shimadzu FTIR-8400 spectrometer. Melting points were determined on a Yanagimoto micro melting point apparatus. Conventional mass spectra were recorded with a Shimadzu GCMS-QP5000 gas chromatograph mass spectrometer. High resolution mass spectra were obtained on a JEOL JMS-DX303 instrument in the analytical section of Osaka University. Elemental analyses were also performed there. 4-Methylphenylacetylene (**2b**),^{S1} 4-pentanylphenylacetylene (**2c**),^{S1} 4-methoxyphenylacetylene (**2d**),^{S1} 4-chlorophenylacetylene (**2e**),^{S1} 4-nitrophenylacetylene (**2f**),^{S2} and phenylacetylene-*d*' (**2i**)^{S3} were prepared according to the literature procedures. Other reactants and reagents were purchased from commercial source and used without further purification.

Experimental Procedure and Characterization Data of Products.

General procedure for the copper(0)-induced reaction of selenoamide **1** with acetylene **2**.

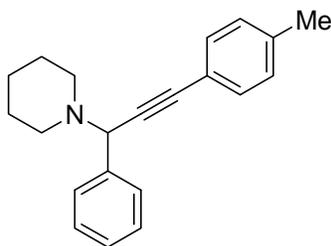
The reaction of *N*-(selenobenzoyl)piperidine (**1a**, 127 mg, 0.5 mmol) with phenylacetylene (**2a**, 1 mL) in the presence of copper(0) (127 mg, 2.0 mmol) was performed at 110 °C for 4 h. After heating, the reaction mixture was treated through a Celite pad and evaporation. The crude mixture was purified by PTLC on silica gel (Hex:Et₂O = 9:1) to give *N*-(1,3-diphenyl-2-propynyl)piperidine (**3a**, 134 mg, 0.49 mmol, 99%) as a colorless oil.

N-(1,3-Diphenyl-2-propynyl)piperidine (**3a**)^{S4}:



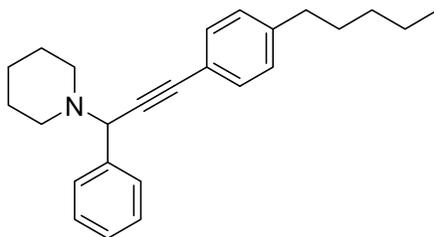
Colorless oil; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.38–1.50 (m, 2H), 1.51–1.68 (m, 4H), 2.56 (t, *J* = 5.3 Hz, 4H), 4.79 (s, 1H), 7.26–7.40 (m, 5H), 7.47–7.56 (m, 3H), 7.63 (d, *J* = 7.3 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 24.4, 26.2, 50.7, 62.4, 86.1, 87.8, 123.4, 127.4, 128.0, 128.3, 128.5, 131.8, 138.7; MS(EI) *m/z* 275 (M⁺, 14).

N-(3-(4-Methylphenyl)-1-phenyl-2-propynyl)piperidine (**3b**)^{S5}:



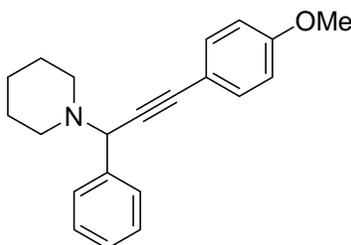
A pale yellow oil; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.36–1.48 (m, 2H), 1.50–1.68 (m, 4H), 2.32 (s, 1H), 2.55 (t, *J* = 5.1 Hz, 4H), 4.77 (s, 1H), 7.11 (d, *J* = 7.5 Hz, 2H), 7.22–7.46 (m, 5H), 7.63 (d, *J* = 6.9 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 21.4, 24.4, 26.2, 50.6, 62.3, 85.2, 87.9, 120.2, 127.3, 127.9, 128.4, 128.9, 131.6, 138.0, 138.7; MS(EI) *m/z* 289 (M⁺, 24).

N-(3-(4-*n*-Pentylphenyl)-1-phenyl-2-propynyl)piperidine (**3c**)^{S5}:



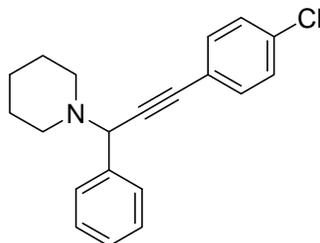
A pale yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 0.88 (t, $J = 7.4$ Hz, 3H), 1.22–1.37 (m, 4H), 1.38–1.49 (m, 2H), 1.50–1.69 (m, 6H), 2.50–2.63 (m, 6H), 4.79 (s, 1H), 7.11 (d, $J = 5.1$ Hz, 2H), 7.19–7.37 (m, 3H), 7.42 (d, $J = 8.4$ Hz, 2H), 7.63 (d, $J = 7.0$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 14.0, 22.5, 24.4, 26.1, 30.9, 31.4, 35.8, 50.6, 62.4, 85.2, 88.0, 120.5, 127.4, 128.0, 128.3, 128.5, 131.7, 138.7, 143.1; MS(EI) m/z 345 (M^+ , 36).

***N*-(3-(4-Methoxyphenyl)-1-phenyl-2-propynyl)piperidine (3d)^{S5}:**



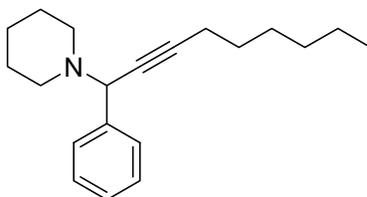
Slight yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.30–1.42 (m, 2H), 1.45–1.63 (m, 4H), 2.50 (t, $J = 5.3$ Hz, 4H), 3.73 (s, 3H), 4.72 (s, 1H), 6.78 (d, $J = 8.8$ Hz, 2H), 7.17–7.34 (m, 3H), 7.39 (d, $J = 8.8$ Hz, 2H), 7.57 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.4, 26.1, 50.7, 55.2, 62.4, 84.5, 87.6, 113.8, 115.5, 127.3, 128.0, 128.5, 133.1, 138.8, 159.4; MS (EI) m/z 305 (M^+ , 29).

***N*-(3-(4-Chlorophenyl)-1-phenyl-2-propynyl)piperidine (3e)^{S6}:**



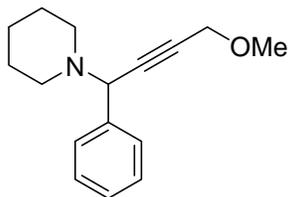
Gray solid; mp 51–52 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.37–1.49 (m, 2H), 1.50–1.69 (m, 4H), 2.55 (t, $J = 2.55$ Hz, 4H), 4.78 (s, 1H), 7.22–7.47 (m, 7H), 7.60 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.4, 26.1, 50.7, 62.3, 86.7, 87.2, 121.7, 127.5, 128.0, 128.4, 128.5, 133.0, 134.0, 138.2; MS (EI) m/z 309 (M^+ , 32).

***N*-(1-Phenyl-2-nonyl)piperidine (3g)^{S5}:**



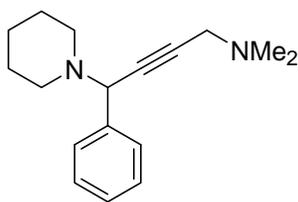
Colorless oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 0.90 (t, $J = 7.0$ Hz, 3H), 1.23–1.64 (m, 16H), 2.31 (dt, $J = 2.2, 7.0$ Hz, 2H), 2.45 (t, $J = 5.3$ Hz, 2H), 4.53 (s, 1H), 7.20–7.35 (m, 3H), 7.55 (d, $J = 7.3$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 14.0, 18.8, 22.6, 24.5, 26.2, 28.6, 29.0, 31.3, 50.5, 62.0, 76.2, 87.9, 127.1, 127.8, 128.5, 139.2; MS (EI) m/z 283 (M^+ , 10).

***N*-(4-Methoxy-1-phenyl-2-butynyl)piperidine (3h):**



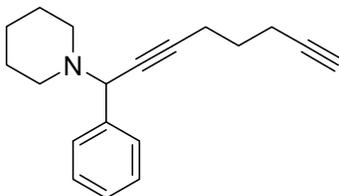
Colorless oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.37–1.47 (m, 2H), 1.50–1.64 (m, 4H), 2.48 (t, $J = 5.3$ Hz, 4H), 3.44 (s, 3H), 4.25 (d, $J = 1.8$ Hz, 2H), 4.62 (s, 1H), 7.22–7.38 (m, 3H), 7.52–7.59 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.4, 26.1, 50.6, 57.5, 60.1, 61.9, 82.9, 83.2, 127.4, 128.0, 128.4, 138.4; IR (NaCl, cm^{-1}) 3029, 3014, 2934, 2851, 2806, 2771, 1493, 1450, 1356, 1269, 1186, 1155, 1105, 1084, 1030, 989, 908, 789, 731, 698, 640; MS (EI) m/z 243 (M^+ , 12); Anal. Calcd. for $\text{C}_{16}\text{H}_{21}\text{NO}$: C, 78.97; H, 8.70; N, 5.76. Found: C, 78.81; H, 8.46; N, 5.66.

***N,N*-Dimethyl-*N*-(4-phenyl-4-(*N'*-piperidyl)-2-butynyl)amine (3i):**



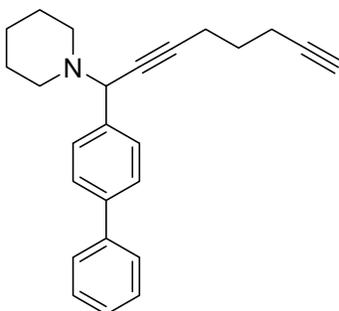
A pale yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.33–1.49 (m, 2H), 1.50–1.71 (m, 4H), 2.37 (s, 6H), 2.49 (t, $J = 5.1$ Hz, 4H), 3.42 (s, 2H), 4.62 (s, 1H), 7.15–7.44 (m, 3H), 7.57 (d, $J = 6.9$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.4, 26.1, 44.2, 48.2, 50.6, 62.0, 81.4, 82.2, 127.3, 127.9, 128.4, 138.7; IR (NaCl, cm^{-1}) 3057, 3029, 2934, 2853, 2777, 1493, 1450, 1356, 1319, 1269, 1151, 1097, 1067, 1030, 989, 837, 789, 731, 698, 669, 638; MS (EI) m/z 211 ($\text{M}^+ - \text{C}_2\text{H}_6\text{NH}$, 9); HRMS (EI) calcd for $\text{C}_{17}\text{H}_{24}\text{N}_2$ (M^+) 256.1939, found 256.1926.

***N*-(2,7-Nonadiynyl-1-phenyl)piperidine (3k):**



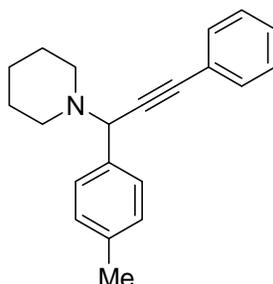
Slight yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.32–1.45 (m, 2H), 1.46–1.64 (m, 4H), 1.79 (q, $J = 7.0$ Hz, 2H), 1.97 (t, $J = 2.7$ Hz, 1H), 2.35 (dt, $J = 2.7, 7.0$ Hz, 2H), 2.39–2.53 (m, 6H), 4.52 (s, 1H), 7.19–7.36 (m, 3H), 7.53 (d, $J = 7.0$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 17.6, 17.9, 24.4, 26.1, 27.9, 50.5, 61.9, 68.8, 77.1, 83.5, 86.4, 127.2, 127.8, 128.4, 140.0; IR (NaCl, cm^{-1}) 3300, 3028, 2932, 2851, 2804, 1454, 1435, 1269, 1204, 1155, 1114, 989, 727, 698, 638; MS (EI) m/z 264 ($\text{M}^+ - \text{H}$, 6); Anal. calcd. for $\text{C}_{19}\text{H}_{23}\text{N}$: C, 85.99; H, 8.74; N, 5.28. Found: C, 85.71; H, 8.75; N, 5.28.

***N*-(1-(4-Biphenyl)-2,7-nonadiynyl)piperidine (3l):**



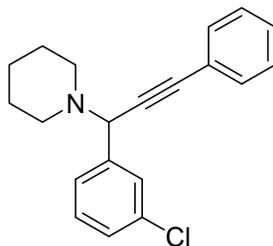
Slight yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.39–1.51 (m, 2H), 1.53–1.68 (m, 4H), 1.82 (q, J = 7.0 Hz, 2H), 1.99 (t, J = 2.8 Hz, 1H), 2.40 (dt, J = 2.7, 7.0 Hz, 2H), 2.44–2.58 (m, 6H), 4.66 (s, 1H), 7.34 (t, J = 7.1 Hz, 1H), 7.44 (t, J = 7.5 Hz, 2H), 7.54–7.68 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 17.6, 17.9, 24.4, 26.0, 27.9, 50.5, 61.6, 68.8, 76.9, 83.5, 86.6, 126.6, 127.0, 127.1, 128.6, 128.8, 137.9, 140.1, 140.9; IR (NaCl, cm^{-1}) 3300, 3055, 3028, 2934, 2853, 2804, 1599, 1487, 1450, 1404, 1319, 1275, 1155, 1111, 1087, 1038, 1009, 989, 856, 756, 698, 637; MS (EI) m/z 341 (M^+ , 100); HRMS (FAB) calcd for $\text{C}_{25}\text{H}_{28}\text{N}$ ($\text{M}^+\text{+H}$) 342.2223, found 342.2227.

***N*-(1-(4-Methylphenyl)-3-phenyl-2-propynyl)piperidine (3m)^{S7}:**



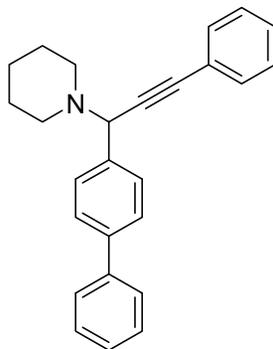
Colorless oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.36–1.48 (m, 2H), 1.50–1.69 (m, 4H), 2.34 (s, 3H), 2.57 (t, J = 5.4 Hz, 4H), 4.77 (s, 1H), 7.15 (d, J = 7.8 Hz, 2H), 7.24–7.33 (m, 3H), 7.45–7.54 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 21.0, 24.4, 26.1, 50.6, 62.0, 86.2, 87.6, 123.3, 127.9, 128.2, 128.5, 128.7, 131.7, 135.4, 137.1; MS(EI) m/z 289 (M^+ , 15).

***N*-(1-(3-Chlorophenyl)-3-phenyl-2-propynyl)piperidine (3n)^{S8}:**



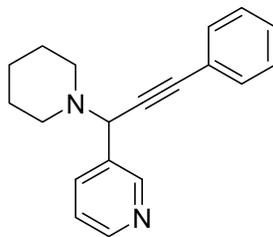
Slight yellow oil; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.38–1.50 (m, 2H), 1.51–1.71 (m, 4H), 2.54 (t, J = 5.1 Hz, 4H), 4.76 (s, 1H), 7.21–7.28 (m, 5H), 7.29–7.36 (m, 3H), 7.64 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.3, 26.1, 50.7, 61.9, 85.1, 88.3, 123.0, 126.6, 127.6, 128.2, 128.3, 128.5, 129.2, 131.8, 134.0, 141.0; MS(EI) m/z 309 (M^+ , 12).

***N*-(1-(4-biphenyl)-3-phenyl-2-propynyl)piperidine (3o)^{S8}:**



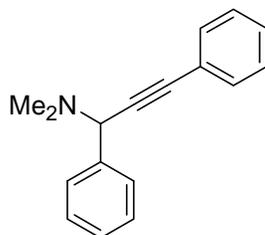
White solid; mp 115 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.38–1.50 (m, 2H), 1.52–1.70 (m, 4H), 2.60 (t, J = 5.3 Hz, 4H), 4.82 (s, 1H), 7.25–7.35 (m, 4H), 7.36–7.46 (m, 2H), 7.48–7.63 (m, 6H), 7.70 (d, J = 8.1 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.4, 26.2, 50.7, 62.1, 86.0, 87.9, 123.3, 126.8, 127.0, 127.2, 127.3, 128.0, 128.2, 128.7, 128.9, 129.0, 130.2, 131.8, 137.8, 140.3, 140.9; IR (NaCl, cm^{-1}) 3028, 2932, 2851, 2804, 1599, 1487, 1443, 1314, 1277, 1153, 1092, 968, 854, 754, 691, 667; MS(EI) m/z 351 (M^+ , 20).

***N*-(3-Phenyl-1-(3-pyridyl)-2-propynyl)piperidine (3p)^{S9}:**



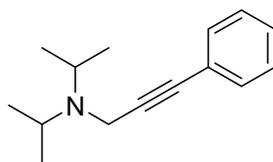
Colorless oil; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.37–1.50 (m, 2H), 1.51–1.72 (m, 4H), 2.56 (t, *J* = 5.1 Hz, 4H), 4.83 (s, 1H), 7.23–7.37 (m, 4H), 7.47–7.56 (m, 2H), 7.95 (d, *J* = 8.1 Hz, 1H), 8.54 (d, *J* = 4.2 Hz, 1H), 8.88 (s, 1H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 24.3, 26.1, 50.6, 60.2, 84.3, 88.6, 122.8, 123.0, 128.3, 131.8, 134.3, 136.0, 148.7, 150.0; IR (NaCl, cm⁻¹) 3032, 2934, 2853, 2806, 2750, 1574, 1489, 1475, 1443, 1421, 1315, 1285, 1155, 1092, 1026, 991, 968, 756, 712, 692, 660, 530; MS(EI) *m/z* 276 (M⁺, 12).

***N,N*-Dimethyl-*N*-(1,3-diphenyl-2-propynyl)amine (3q)^{S10}:**



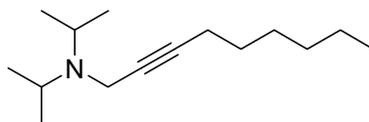
Colorless oil; ¹H NMR (300 MHz, CDCl₃, ppm) δ 2.33 (s, 6H), 4.83 (s, 1H), 7.25–7.41 (m, 5H), 7.48–7.56 (m, 2H), 7.61 (d, *J* = 6.6 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 41.6, 62.2, 84.8, 88.4, 123.2, 127.7, 128.1, 128.2, 128.3, 128.5, 131.8, 138.7; IR (NaCl, cm⁻¹) 3059, 3030, 2941, 2858, 2823, 2777, 1599, 1489, 1470, 1450, 1325, 1275, 1172, 1157, 1070, 1018, 968, 916, 833, 756, 729, 692, 598; MS(EI) *m/z* 235 (M⁺, 14).

***N,N*-Diisopropyl-*N*-(3-phenyl-2-propynyl)amine (3s)^{S11}:**



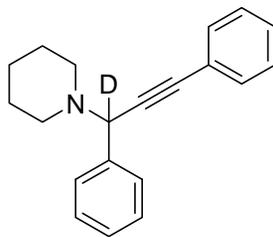
Slight yellow oil; ¹H NMR (400 MHz, CDCl₃, ppm) δ 1.15 (d, *J* = 6.3 Hz, 12H), 3.26 (q, *J* = 6.6 Hz, 2H), 3.65 (s, 2H), 7.24–7.31 (m, 3H), 7.37–7.43 (m, 2H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 20.6, 34.7, 48.4, 83.4, 89.0, 123.7, 127.6, 128.1, 131.4; IR (NaCl, cm⁻¹) 3057, 3022, 2966, 2932, 2874, 1599, 1489, 1464, 1443, 1381, 1362, 1325, 1204, 1177, 1119, 1070, 1028, 756, 691, 633, 525; MS(EI) *m/z* 215 (M⁺, 3).

***N,N*-Diisopropyl-*N*-(2-nonyl)amine (3t):**



Colorless oil; ¹H NMR (400 MHz, CDCl₃, ppm) δ 0.89 (t, *J* = 6.8 Hz, 3H), 1.09 (d, *J* = 6.3 Hz, 12H), 1.21–1.56 (m, 8H), 2.15 (t, *J* = 7.1 Hz, 2H), 3.19 (sep, *J* = 6.6 Hz, 2H), 3.39 (s, 2H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 14.0, 18.8, 20.5, 22.6, 28.5, 28.8, 31.3, 34.3, 48.2, 78.7, 83.4; IR (NaCl, cm⁻¹) 2963, 2932, 2858, 1464, 1435, 1379, 1361, 1325, 1204, 1177, 1140, 1119, 1026, 933, 885, 719, 614, 538; MS(EI) *m/z* 223 (M⁺, 5); HRMS (FAB) calcd for C₁₅H₃₀N (M⁺+H) 224.2380, found 224.2371.

N-(1,3-Diphenyl-2-propynyl)piperidine-*d*¹ (**3u**):



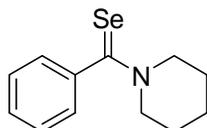
Slight yellow oil; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.37–1.49 (m, 2H), 1.50–1.69 (m, 4H), 2.56 (t, *J* = 5.1 Hz, 4H), 4.79 (s, 5%), 7.21–7.40 (m, 6H), 7.46–7.55 (m, 2H), 7.59–7.57 (m, 2H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 24.4, 26.2, 50.6, 86.1, 87.8, 123.3, 127.4, 128.0, 128.2, 128.5, 131.8, 138.6; IR (NaCl, cm⁻¹) 3042, 3029, 2932, 2853, 2804, 1599, 1489, 1447, 1244, 1153, 1099, 1070, 1028, 1004, 756, 718, 691; MS (EI) *m/z* 276 (M⁺, 10); HRMS (FAB) calcd for C₂₀H₂₁DN (M⁺+H) 277.1832, found 277.1836.

Synthesis and Spectral Data of Selenoamides.

General procedure for the synthesis of selenoamides **1a**, **1b**, **1c**, **1d**, **1e**, **1f**, and **1g**.

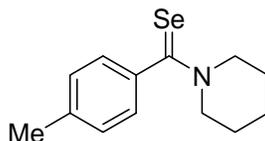
A mixture of benzonitrile (1.2 mL, 11.8 mmol), selenium (1.05 g, 13.1 mmol), H₂O (2.4 mL, 133 mmol), triethyl amine (2.4 mL, 17.3 mmol), and THF (10 mL) in a 50 mL stainless steel autoclave was stirred at 110 °C under the pressure of CO (1.5 MPa: initial pressure at 25 °C) for overnight. After the reaction, CO was purged in the well-ventilated hood, and to the reaction mixture was added piperidine (2.0 mL, 19.7 mmol), the resulting mixture, then, was heated in an autoclave at 110 °C for 4 h. After stirring, the reaction mixture was slightly acidified with aqueous 2 N HCl and extracted with diethyl ether. The combined extracts were dried over anhydrous MgSO₄, filtered, and evaporated. The crude material was purified by flash column chromatography on silica gel (Hex:Et₂O = 7:3) to give *N*-(selenobenzoyl)piperidine (**1a**, 1.31 g, 5.2 mmol, 44%) as a yellow solid.

N-(Selenobenzoyl)piperidine (**1a**)^{7d}:



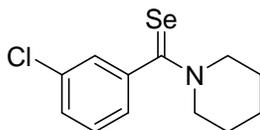
Yellow solid; mp 89 °C; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.52–1.64 (m, 2H), 1.71–1.95 (m, 4H), 3.50 (t, *J* = 5.7 Hz, 2H), 4.49 (t, *J* = 5.6 Hz, 2H), 7.21–7.39 (m, 5H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 23.9, 25.5, 26.7, 53.9, 55.0, 124.3, 128.1, 128.2, 146.0, 203.4; MS (EI) *m/z* 253 (M⁺, 30).

N-(*p*-Methyl-selenobenzoyl)piperidine (**1b**)⁵ⁱ:



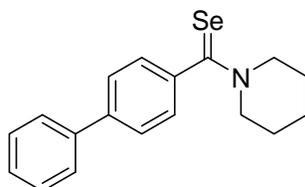
A pale orange solid; mp 109–110 °C; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.51–1.65 (m, 2H), 1.70–1.94 (m, 4H), 2.33 (s, 3H), 3.53 (t, *J* = 5.7 Hz, 2H), 4.48 (t, *J* = 5.7 Hz, 2H), 7.09–7.20 (m, 4H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 21.2, 24.0, 25.5, 26.8, 53.9, 55.2, 124.6, 128.8, 138.4, 143.4, 203.8; IR (NaCl, cm⁻¹) 3015, 2945, 2918, 2856, 1643, 1516, 1493, 1443, 1286, 1238, 1200, 1134, 1099, 1016, 1001, 947, 883, 854, 822, 795; MS (EI) *m/z* 267 (M⁺, 63); HRMS (EI) calcd for C₁₃H₁₇NSe (M⁺) 267.0526, found 267.0528.

N-(*m*-Chloro-selenobenzoyl)piperidine (**1c**):



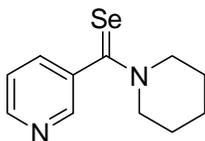
Yellow solid; mp 69–70 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.53–1.66 (m, 2H), 1.71–1.94 (m, 4H), 3.49 (t, $J = 5.7$ Hz, 2H), 4.45 (t, $J = 5.0$ Hz, 2H), 7.07–7.16 (m, 1H), 7.22–7.29 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 23.7, 25.4, 26.6, 54.0, 54.9, 122.4, 124.4, 128.2, 129.5, 133.9, 147.4, 201.0; IR (NaCl, cm^{-1}) 2939, 2856, 1636, 1589, 1562, 1495, 1446, 1406, 1350, 1285, 1238, 1200, 1132, 1092, 1076, 1003, 966, 950, 889, 854, 781, 731, 689; MS (EI) m/z 287 (M^+ , 21); Anal. Calcd. for $\text{C}_{12}\text{H}_{14}\text{ClNSe}$: C, 50.28; H, 4.92; N, 4.89. Found: C, 50.22; H, 4.84; N, 4.86.

***N*-(*p*-Phenyl-selenobenzoyl)piperidine (1d):**



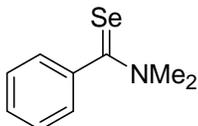
Light yellow solid; mp 186–187 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.56–1.68 (m, 2H), 1.73–1.84 (m, 2H); 1.85–1.96 (m, 2H), 3.58 (t, $J = 5.7$ Hz, 2H), 4.50 (t, $J = 5.7$ Hz, 2H), 7.31–7.64 (m, 9H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 24.0, 25.5, 26.8, 54.0, 55.2, 125.0, 126.9, 127.0, 127.1, 127.4, 127.6, 141.2, 144.9, 198.4; IR (NaCl, cm^{-1}) 3014, 2948, 2928, 2902, 2857, 2853, 1658, 1499, 1483, 1443, 1237, 1215, 1194, 1132, 1003, 837, 760, 743, 689; MS (EI) m/z 329 (M^+ , 18); Anal. Calcd. for $\text{C}_{18}\text{H}_{19}\text{NSe}$: C, 65.85; H, 5.83; N, 4.27. Found: C, 65.64; H, 5.87; N, 4.54.

***N*-(3-Pyridyl-selenobenzoyl)piperidine (1e):**



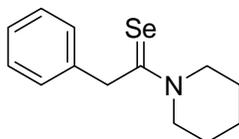
Yellow solid; mp 86 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 1.50–1.70 (m, 2H), 1.70–2.09 (m, 4H), 3.53 (brs, 2H), 4.49 (brs, 2H), 7.18–7.39 (m, 1H), 7.55–7.77 (m, 1H), 8.42–8.65 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 23.8, 25.4, 26.8, 54.1, 55.1, 122.9, 132.4, 141.9, 144.4, 149.2, 199.5; IR (NaCl, cm^{-1}) 3416, 2939, 2856, 1634, 1582, 1564, 1504, 1446, 1408, 1286, 1240, 1207, 1184, 1132, 1107, 1024, 1003, 943, 883, 854, 818, 795, 710, 689; MS (EI) m/z 254 (M^+ , 21); Anal. Calcd. for $\text{C}_{11}\text{H}_{14}\text{N}_2\text{Se}$: C, 52.18; H, 5.57; N, 11.06. Found: C, 51.99; H, 5.51; N, 11.03.

***N,N*-Dimethyl benzeneselenoamide (1f)^{5c}:**



Yellow solid; mp 76 °C; ^1H NMR (300 MHz, CDCl_3 , ppm) δ 3.10 (s, 3H), 3.70 (s, 3H), 7.19–7.42 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3 , ppm) δ 44.7, 47.3, 124.7, 128.1, 128.4, 146.2, 205.4; MS (EI) m/z 213 (M^+ , 64).

Benzyl piperidyl selenone (1g)^{S12}:

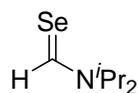


Light yellow solid; mp 107–108 °C; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.23–1.35 (m, 2H), 1.57–1.68 (m, 2H), 1.69–1.79 (m, 2H), 3.55 (t, *J* = 5.6 Hz, 2H), 4.40 (t, *J* = 5.5 Hz, 2H), 4.52 (s, 2H), 7.20–7.43 (m, 5H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 23.5, 25.2, 25.9, 52.4, 54.6, 56.4, 126.9, 127.7, 128.8, 135.3, 201.6; MS (EI) *m/z* 267 (M⁺, 31).

Preparation of Selenoformamide **1h**.^{S13}

The treatment of *N,N*-diisopropylformamide (0.75 mL, 5 mmol) with oxalyl chloride (0.5 mmol, 0.45 mL) in diethyl ether (25 mL) under N₂ atmosphere was stirred at 0 °C for 1 h. Moreover, the reaction mixture was stirred at 3 h at room temperature. The mixture was added to the solution of LiAlHSeH (6 mmol), prepared in situ. The combined mixture was stirred at room temperature for 3 h. After the reaction, to the mixture was added H₂O and was extracted with AcOEt. The organic layer was dried over anhydrous MgSO₄ and evaporated. The residue was purified by flash chromatography on silica gel (Hex; AcOEt = 9:1) to give *N,N*-diisopropyl selenoformamide (**1h**, 592 mg, 62%) as a yellow solid.

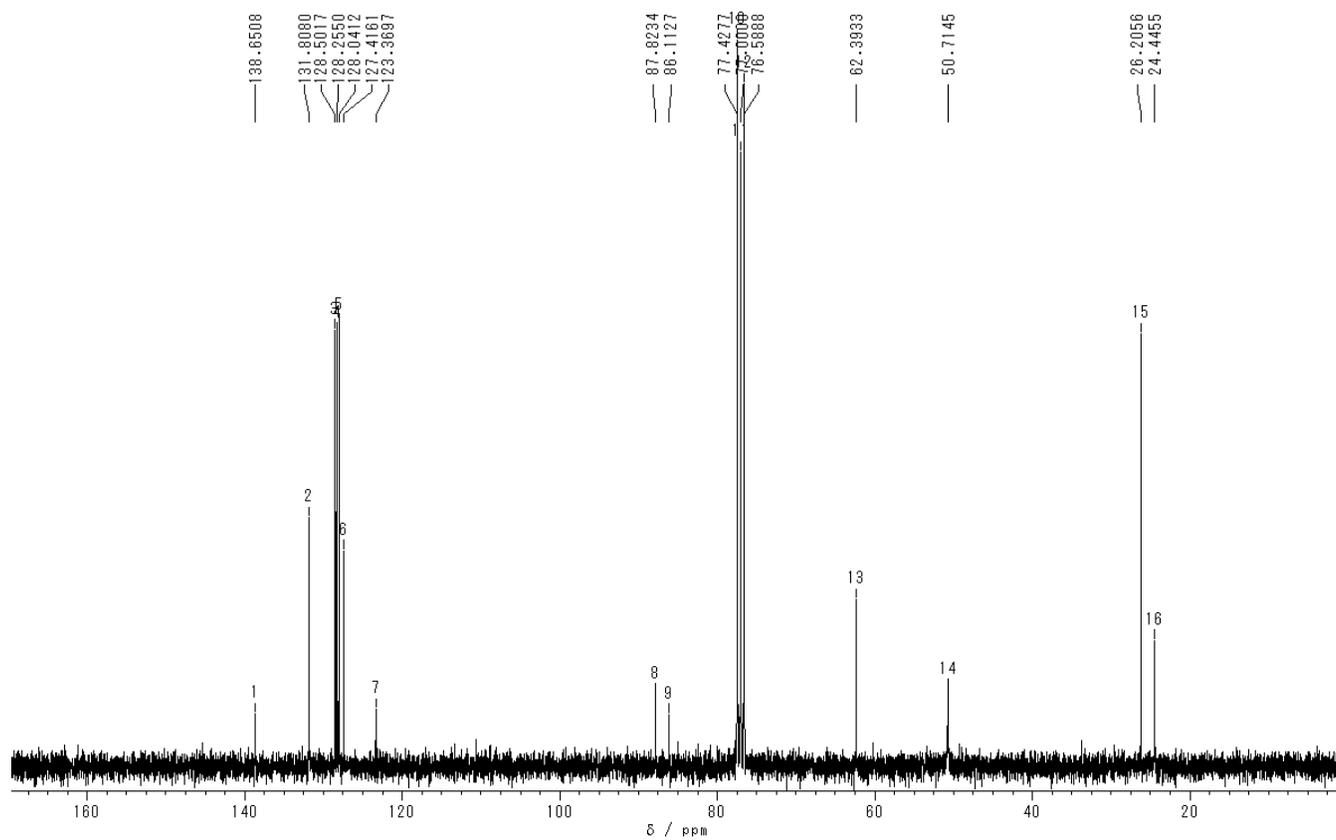
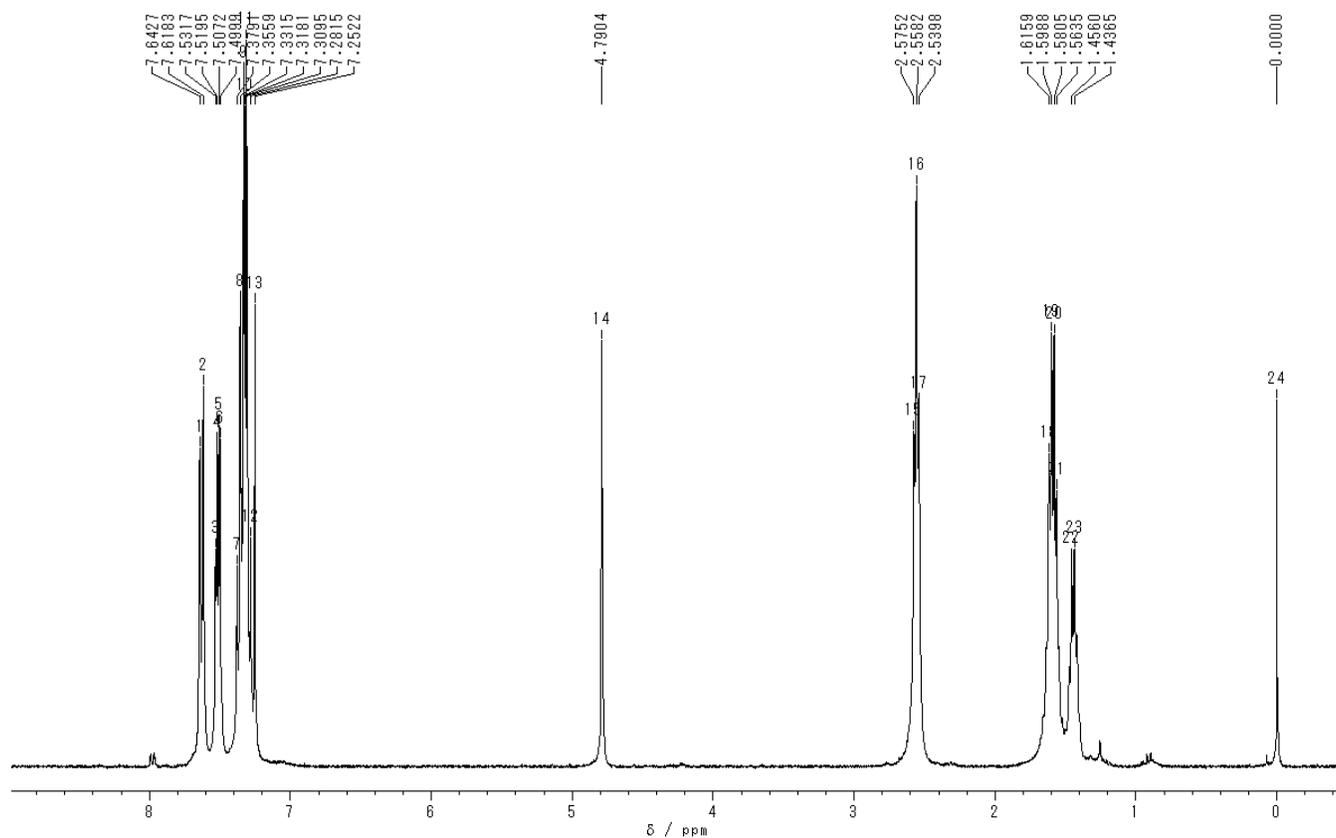
N,N-Diisopropyl selenoformamide (**1h**)^{S14}:



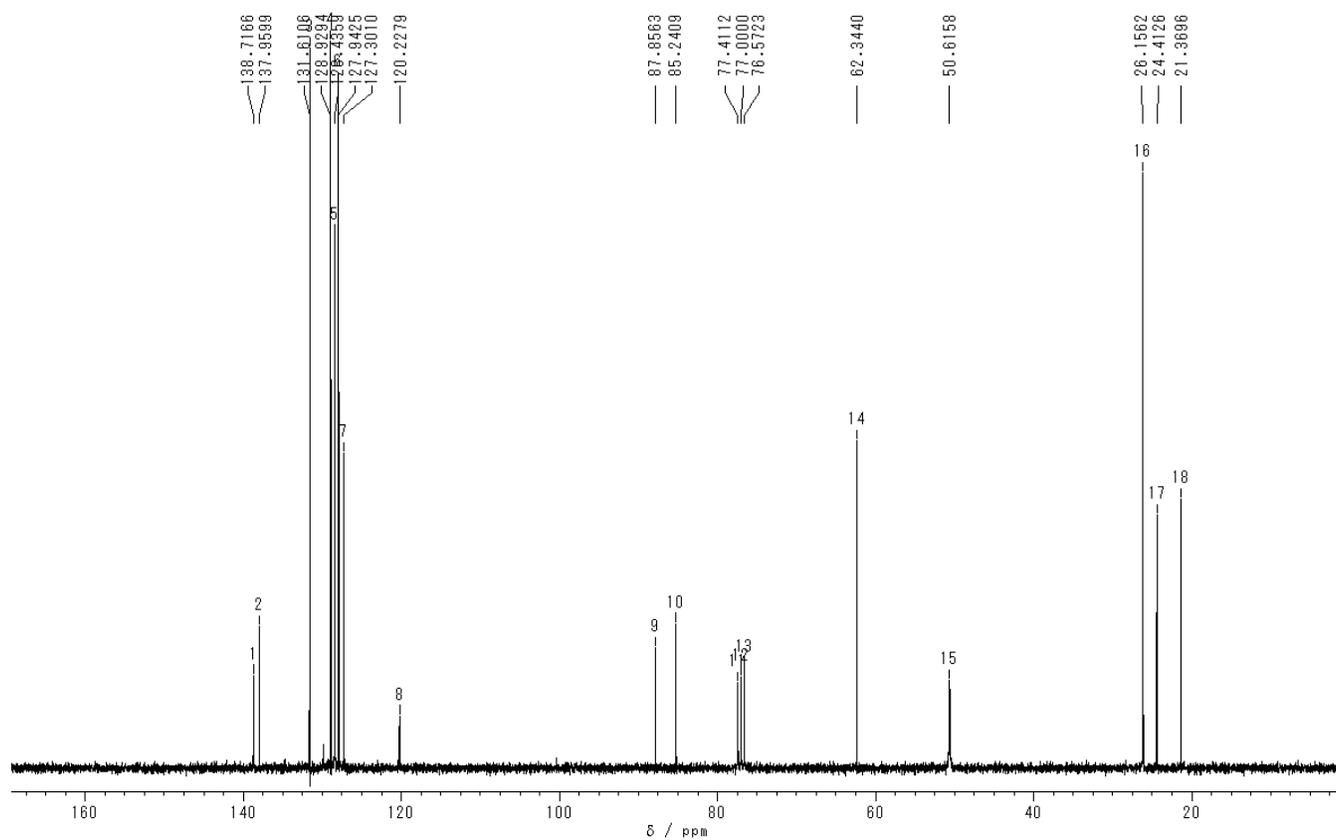
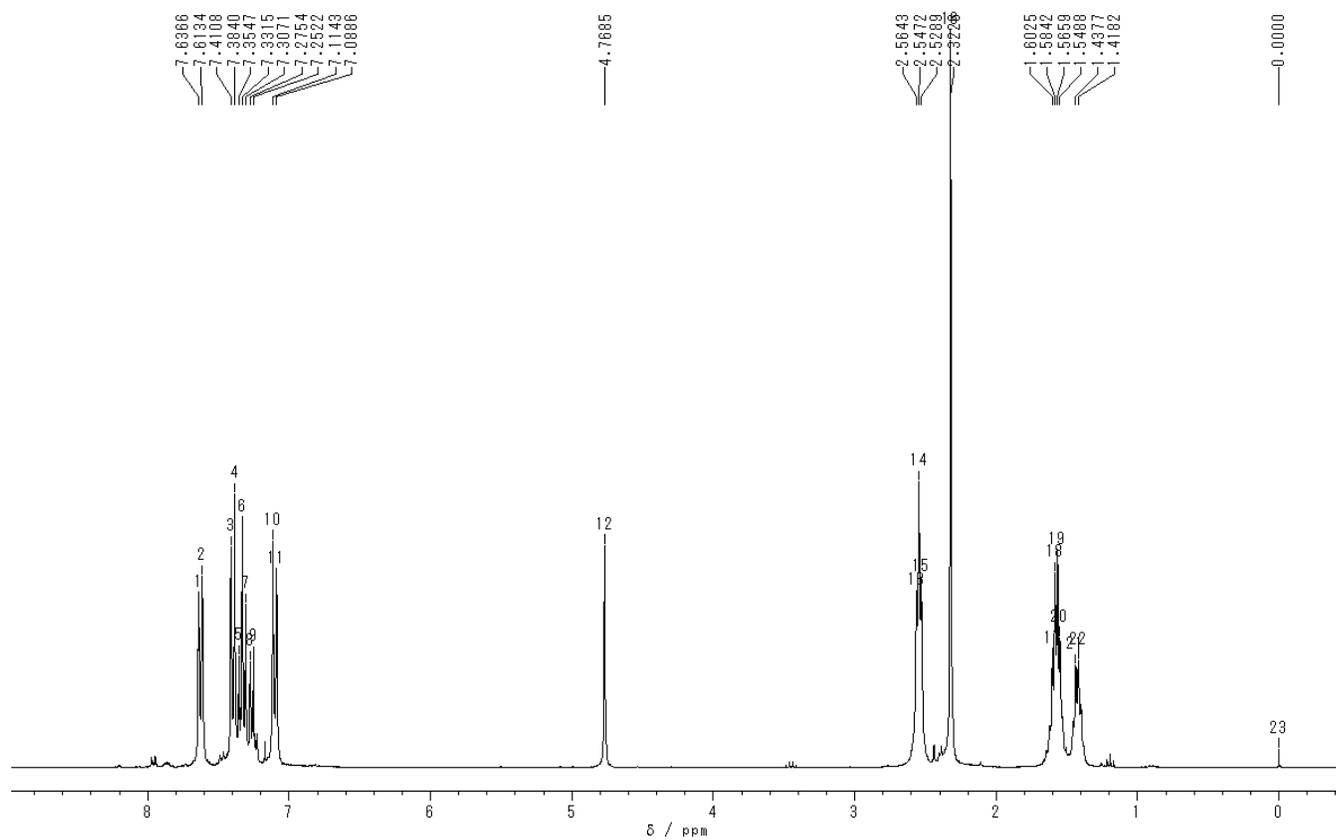
Yellow solid; mp 82–83 °C; ¹H NMR (300 MHz, CDCl₃, ppm) δ 1.28 (d, *J* = 6.6 Hz, 6H), 1.33 (d, *J* = 6.6 Hz, 6H), 3.96 (sep, *J* = 6.6 Hz, 1H), 5.81 (sep, *J* = 6.6 Hz, 1H), 10.77 (s, 1H); ¹³C NMR (75 MHz, CDCl₃, ppm) δ 18.9, 23.9, 50.2, 52.4, 186.2; IR (NaCl, cm⁻¹) 2972, 2932, 2870, 1504, 1456, 1371, 1298, 1184, 1138, 1111, 1003, 920, 816, 731, 687, 644, 573; MS (EI) *m/z* 193 (M⁺, 43).

^1H and ^{13}C Spectra

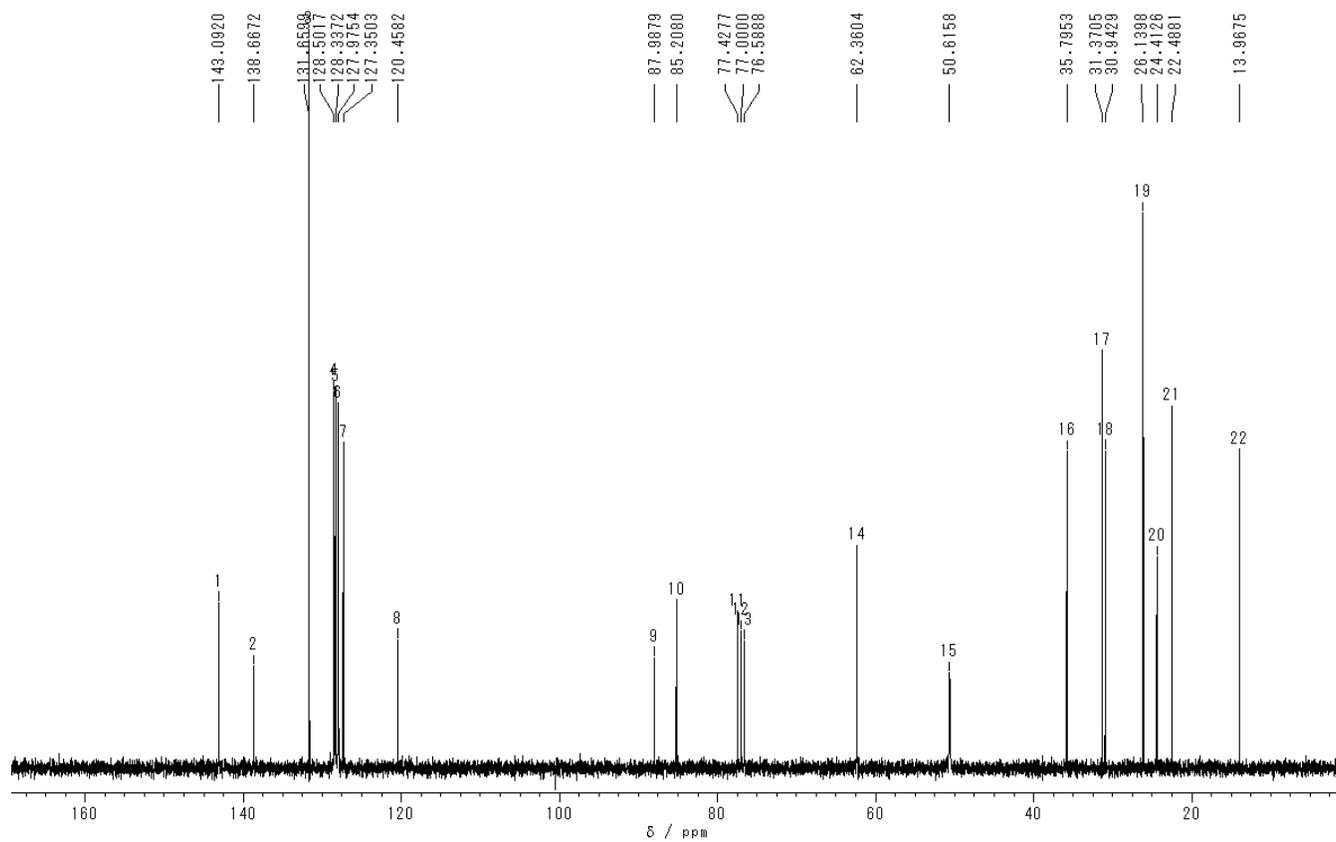
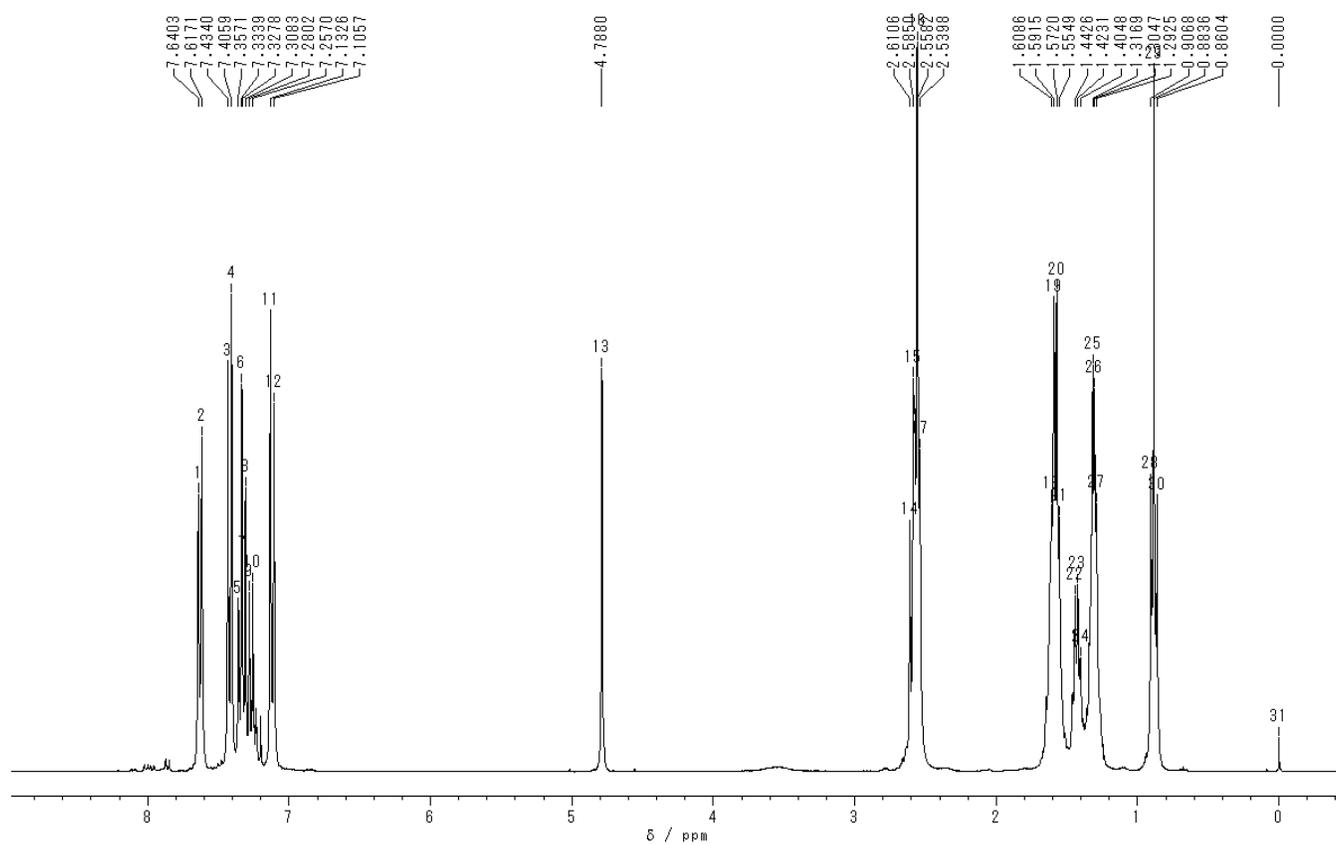
N-(1,3-Diphenyl-2-propynyl)piperidine (3a)



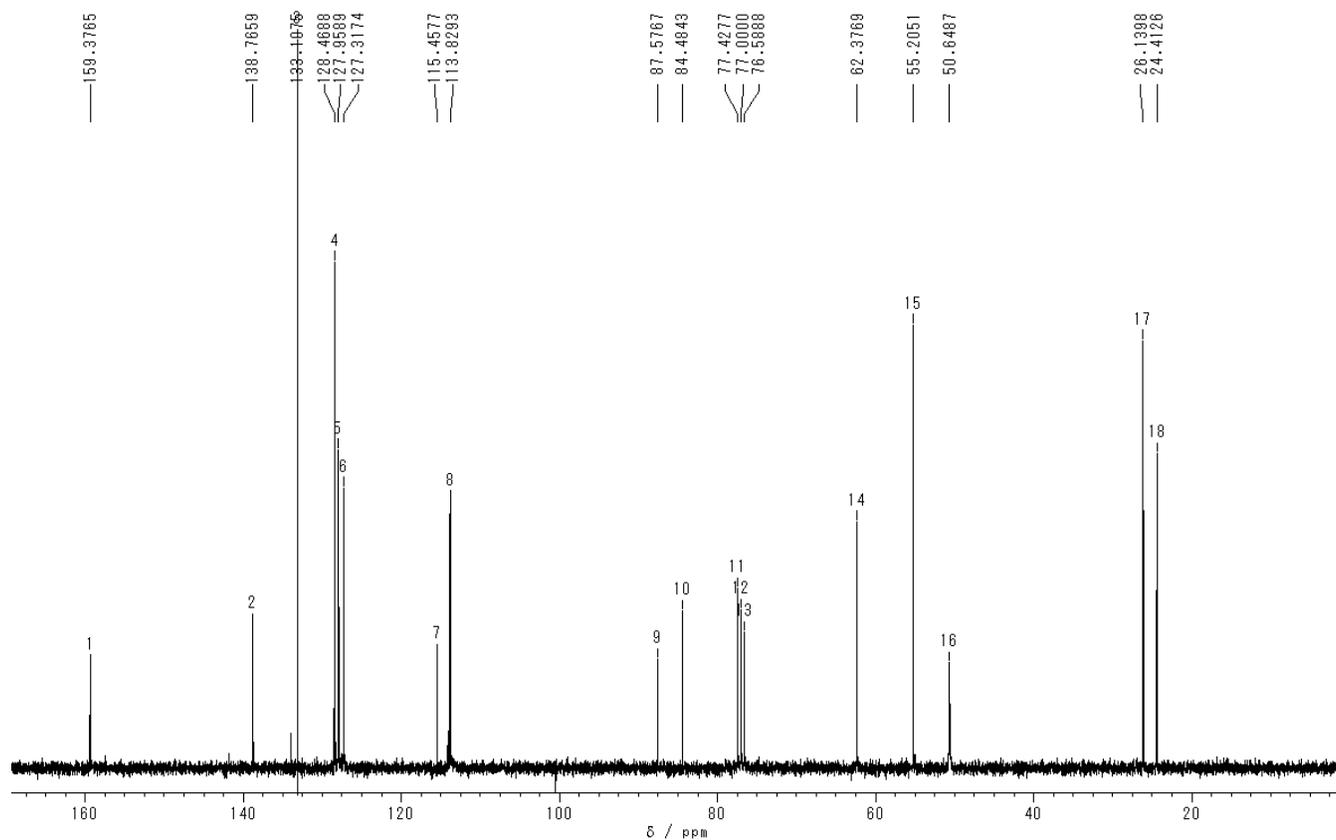
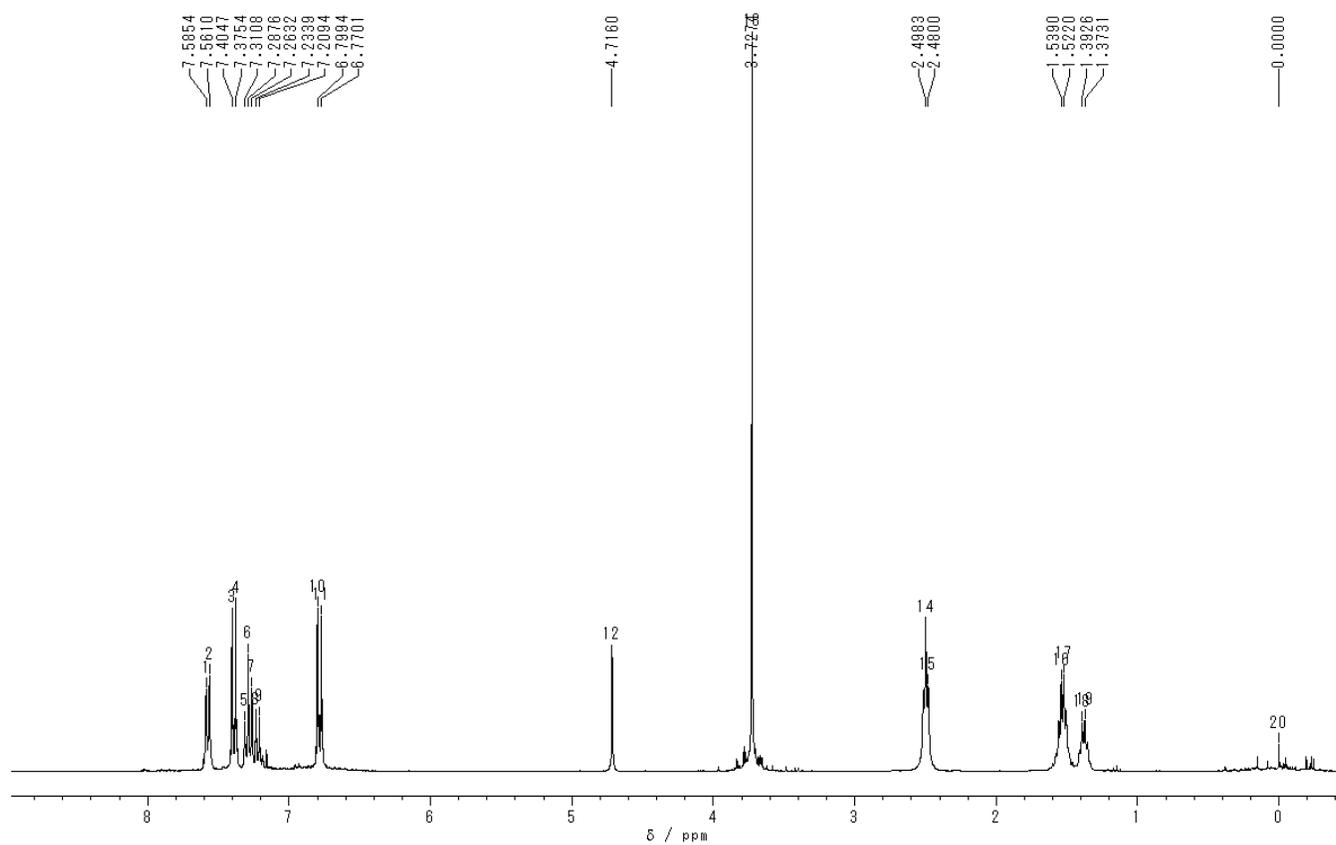
***N*-3-(4-Methylphenyl)-1-phenyl-2-propynylpiperidine (3b)**



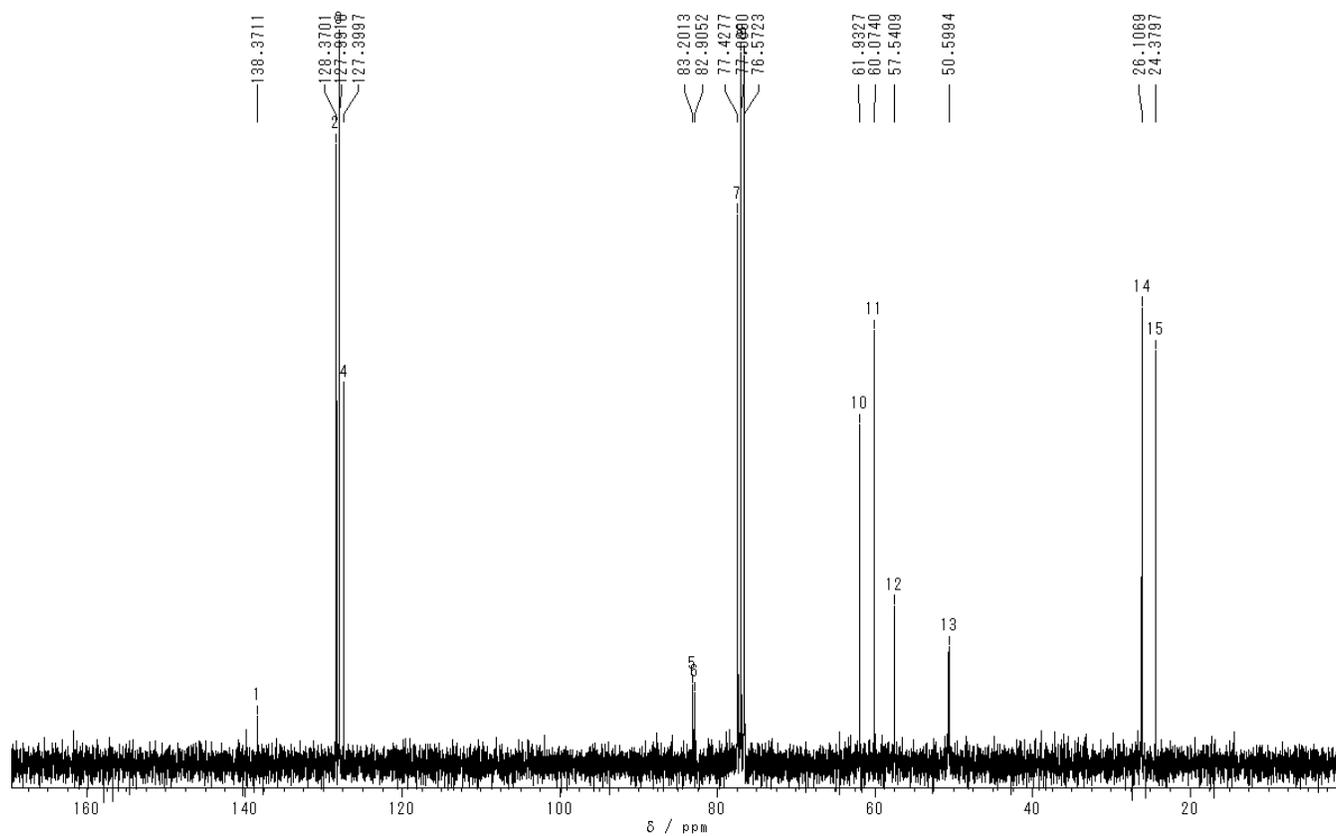
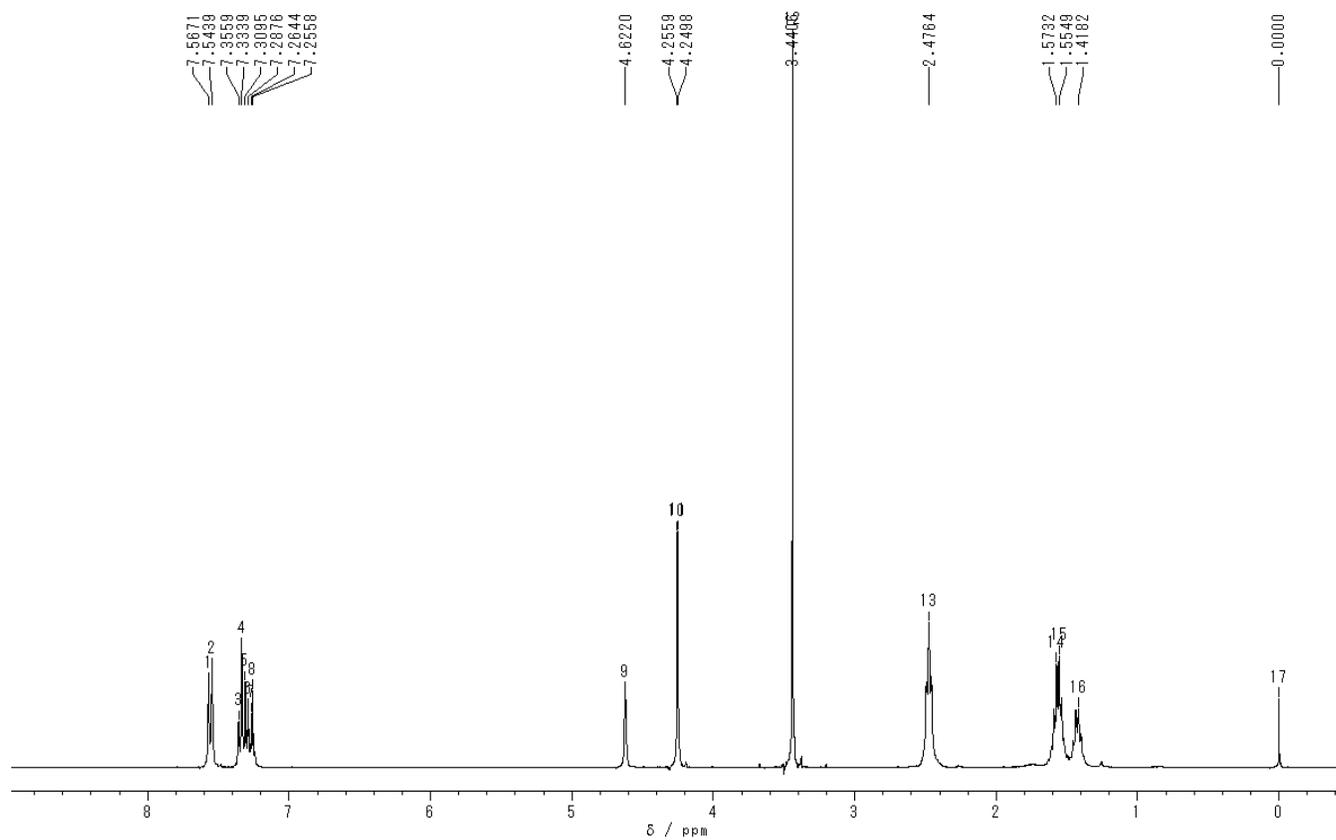
***N*-[3-(4-*n*-Pentylphenyl)-1-phenyl-2-propynyl]piperidine (3c)**



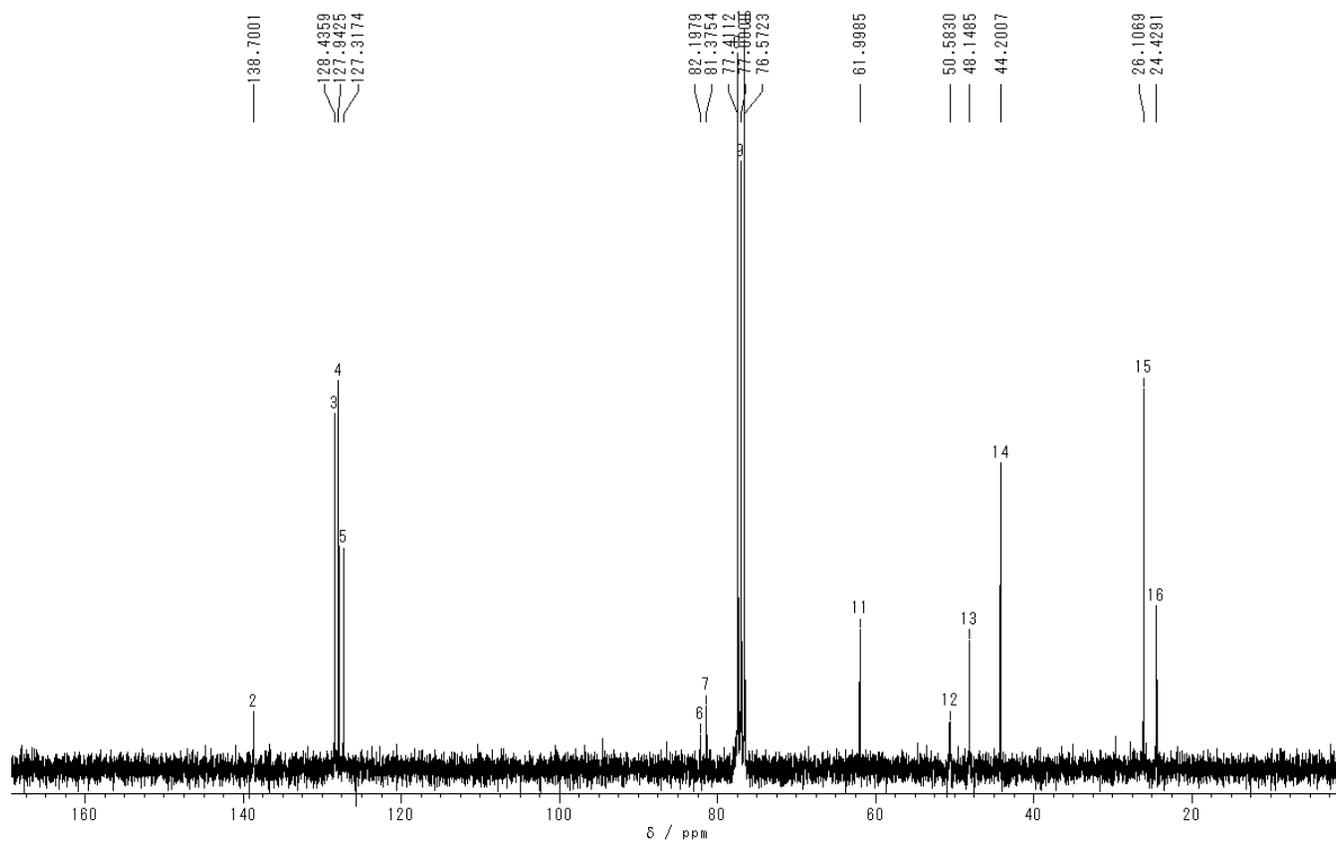
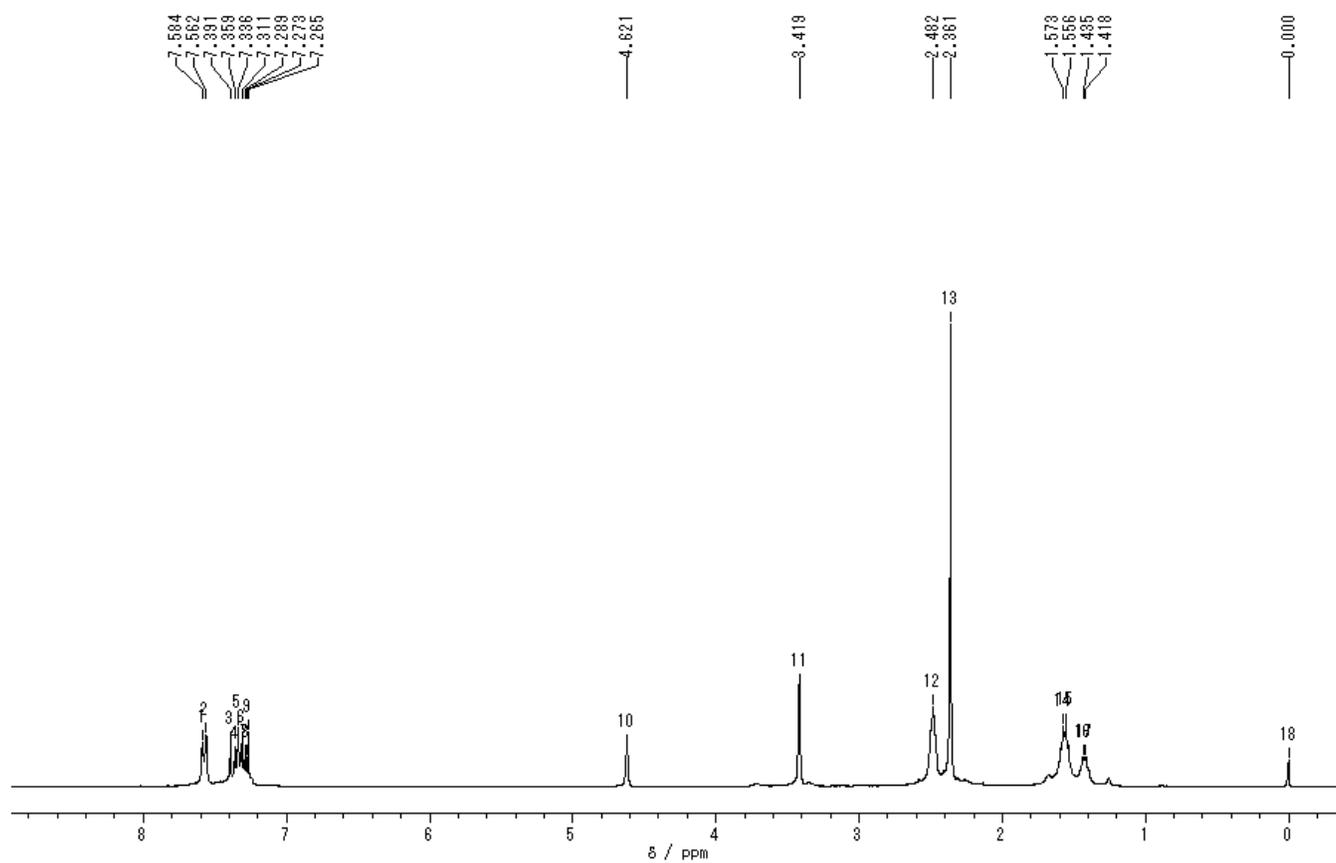
***N*-3-(4-Methoxyphenyl)-1-phenyl-2-propynylpiperidine (3d)**



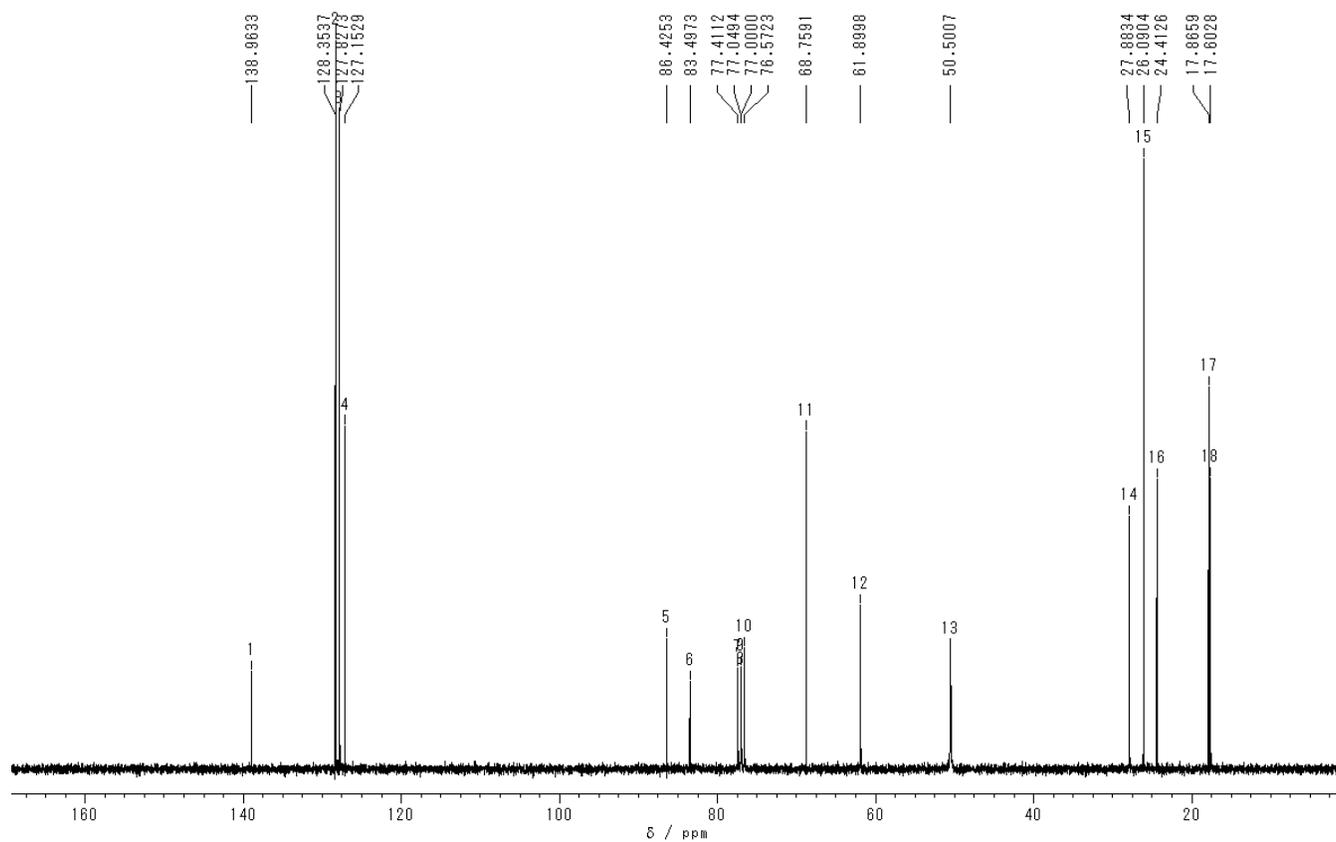
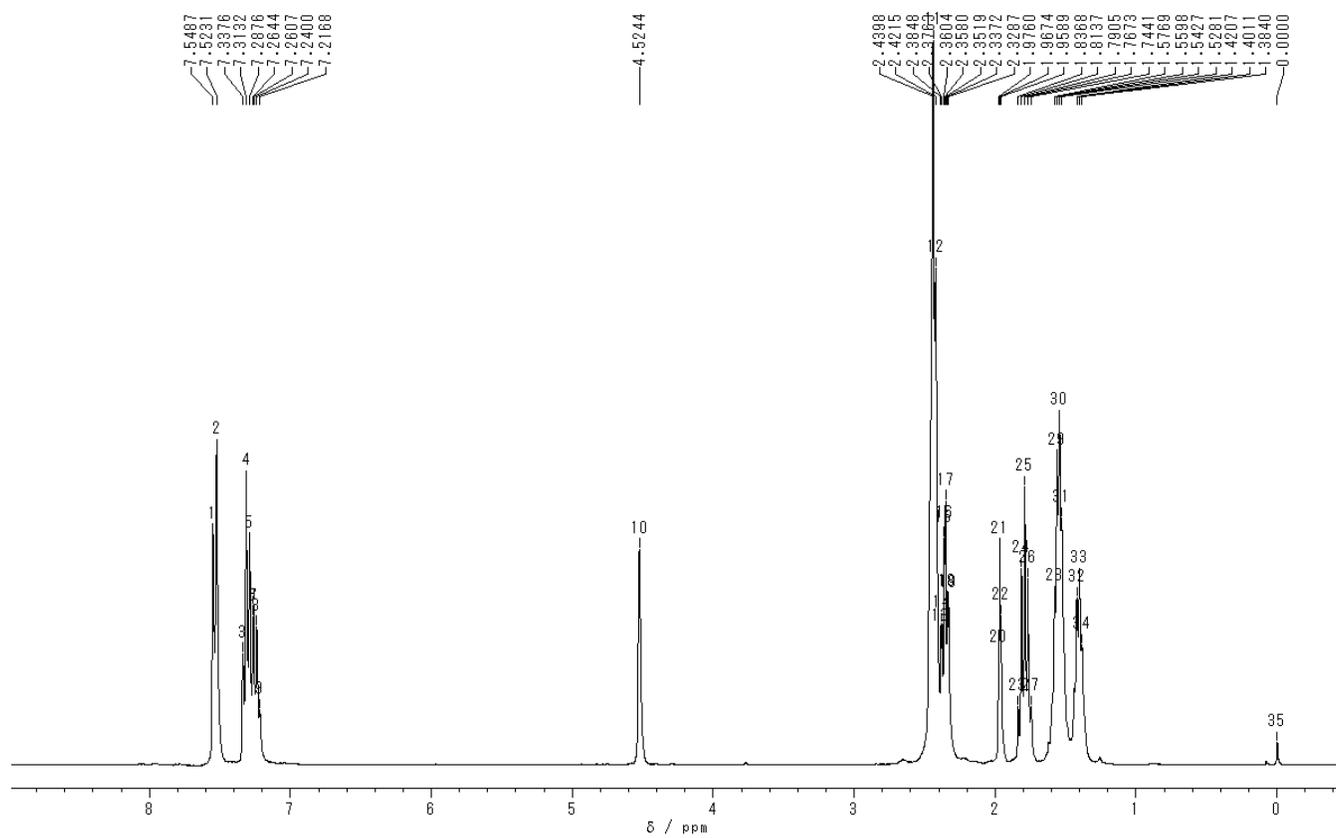
N-(4-Methoxy-1-phenyl-2-butynyl)piperidine (3h)



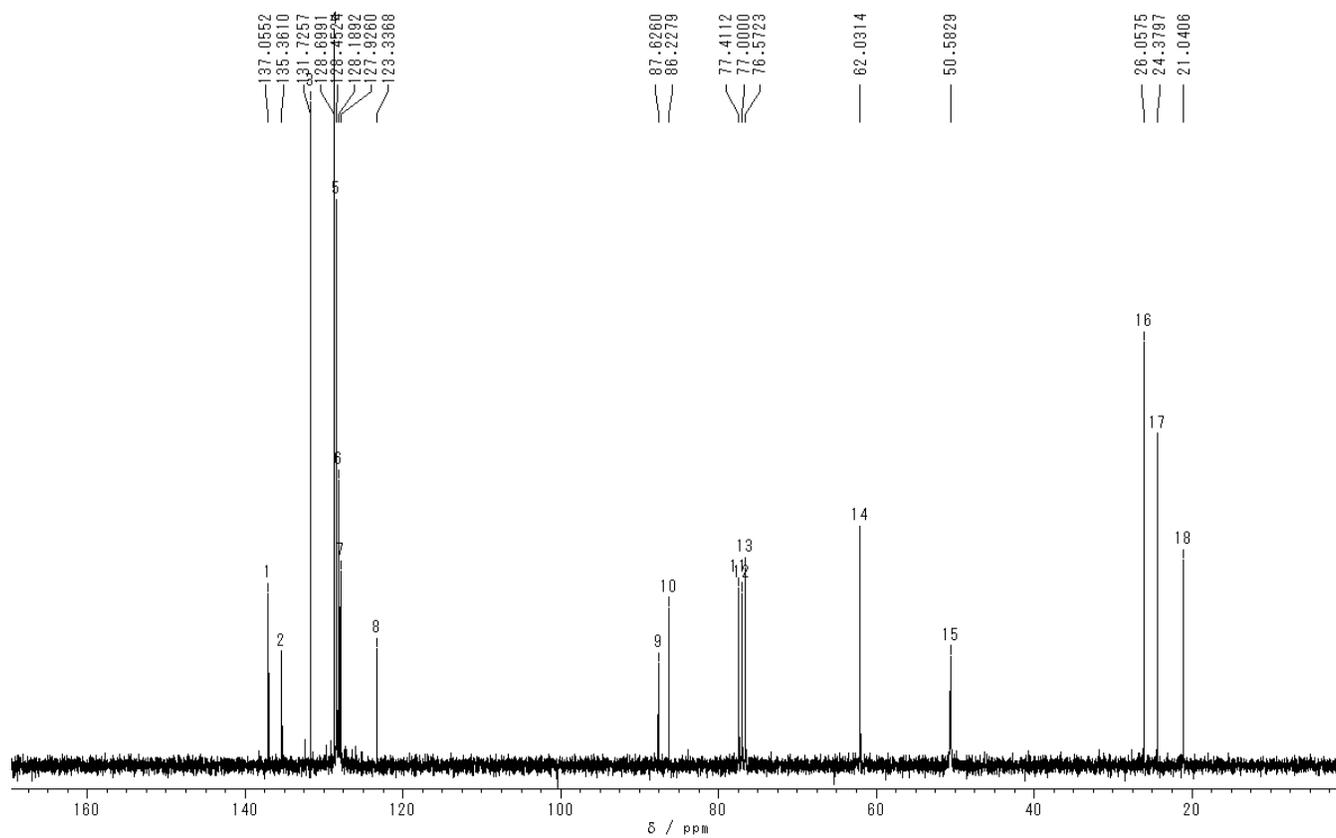
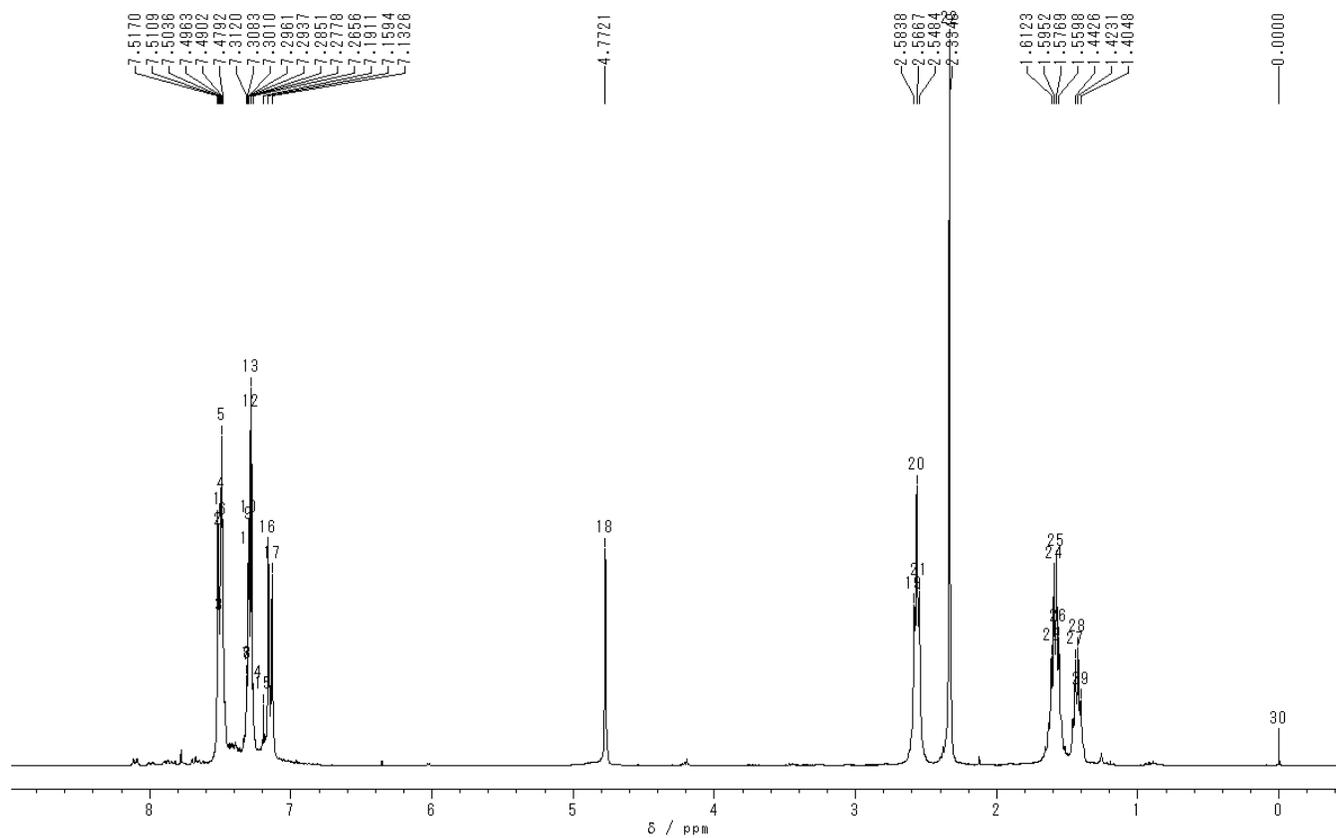
***N,N*-Dimethyl-*N*-(4-phenyl-4-(*N'*-piperidyl)-2-butynyl)amine (3i)**



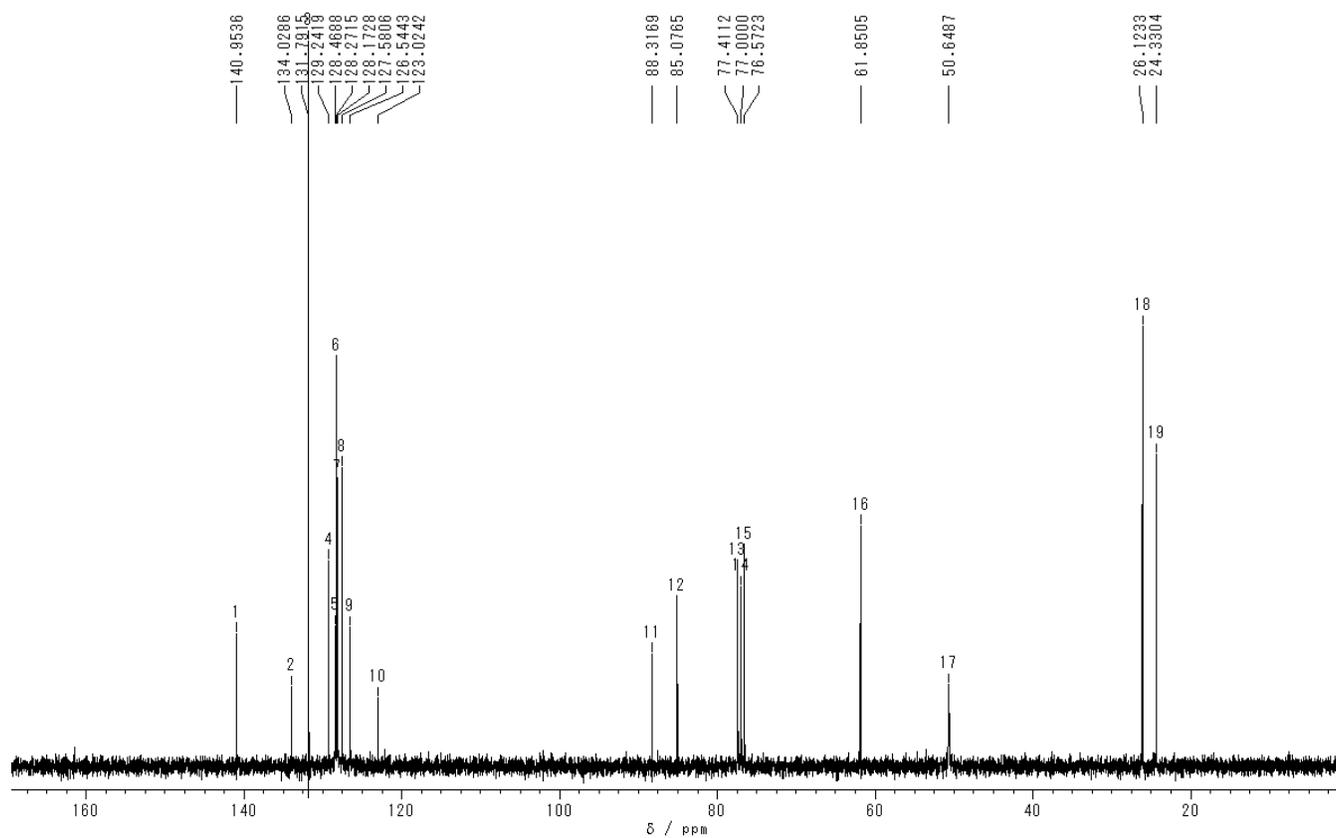
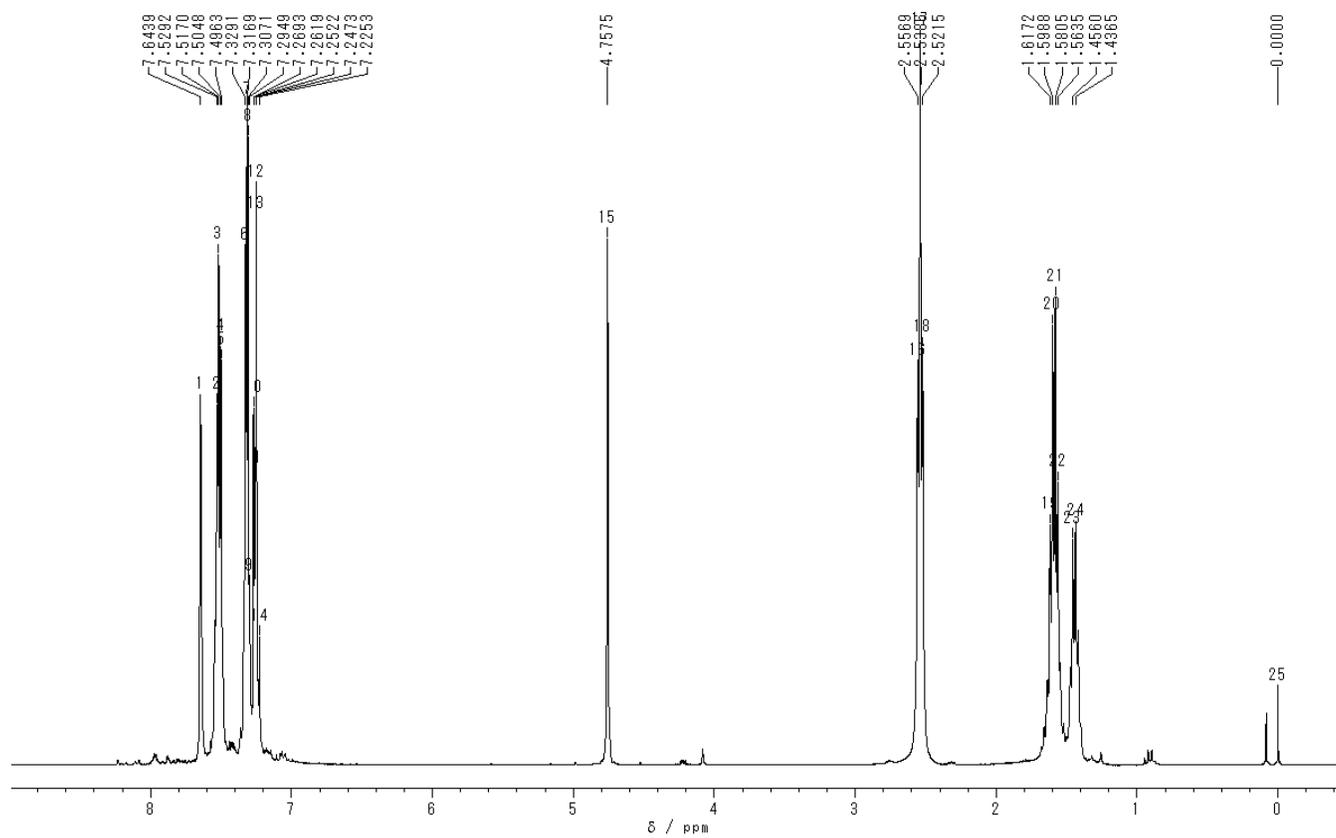
N-(2,7-Nonadiynyl-1-phenyl)piperidine (3k)



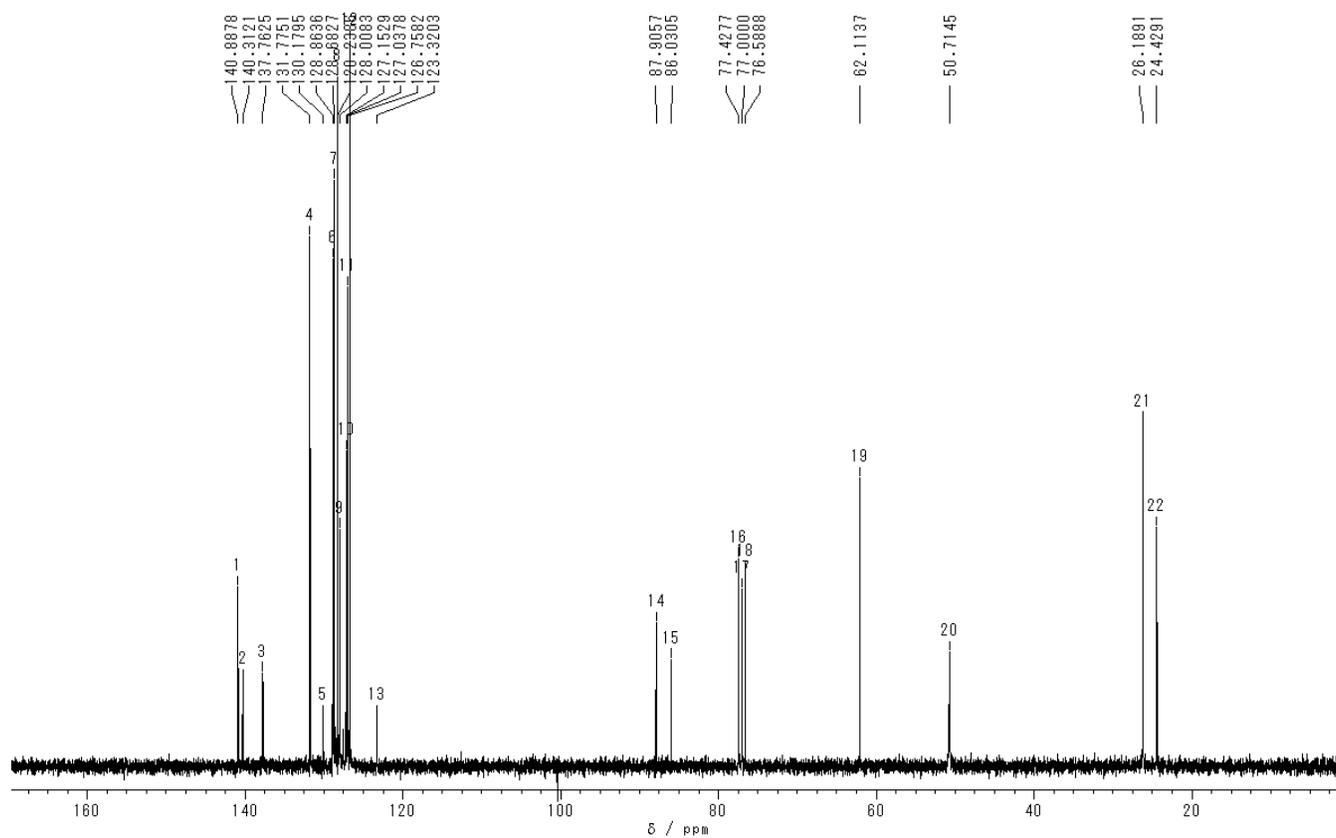
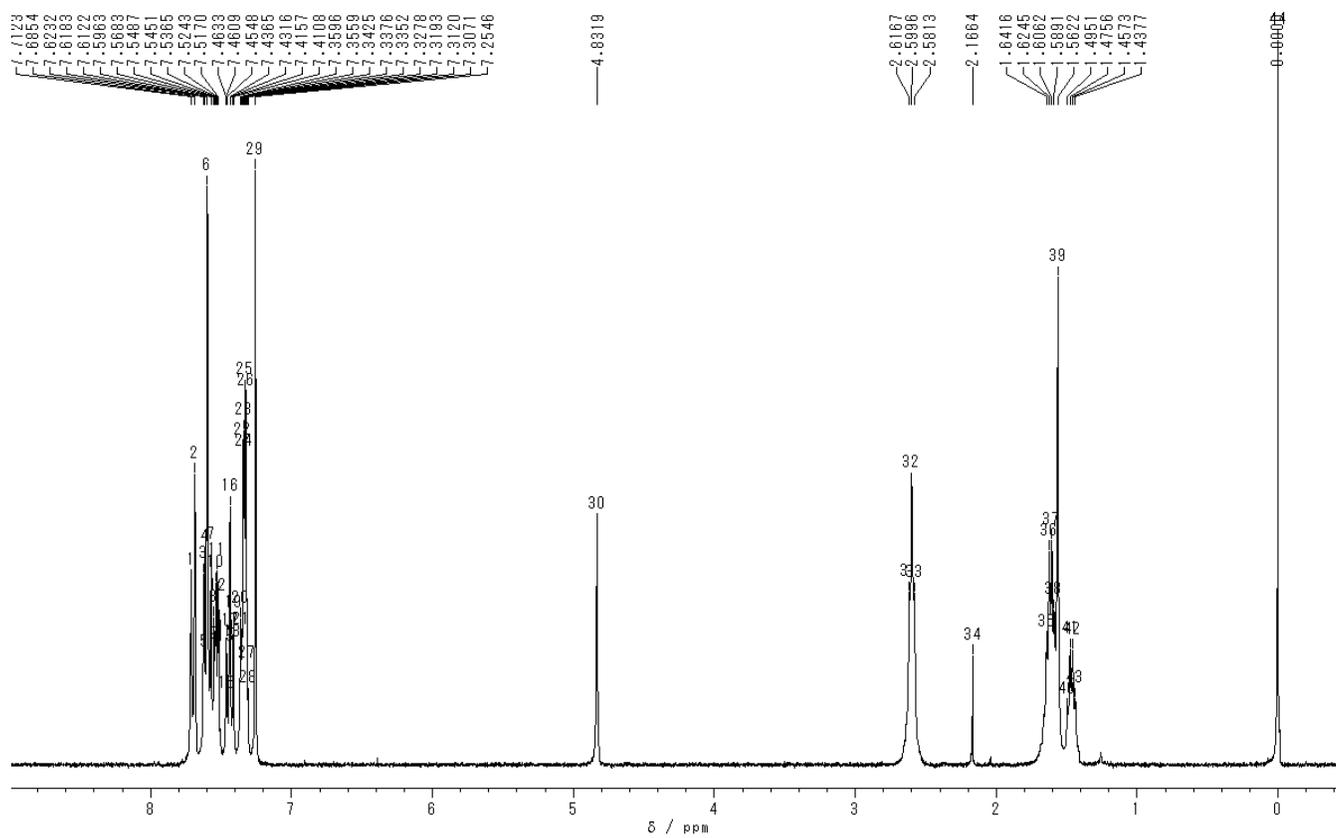
***N*-(1-(4-Methylphenyl)-3-phenyl-2-propynyl)piperidine (3m)**



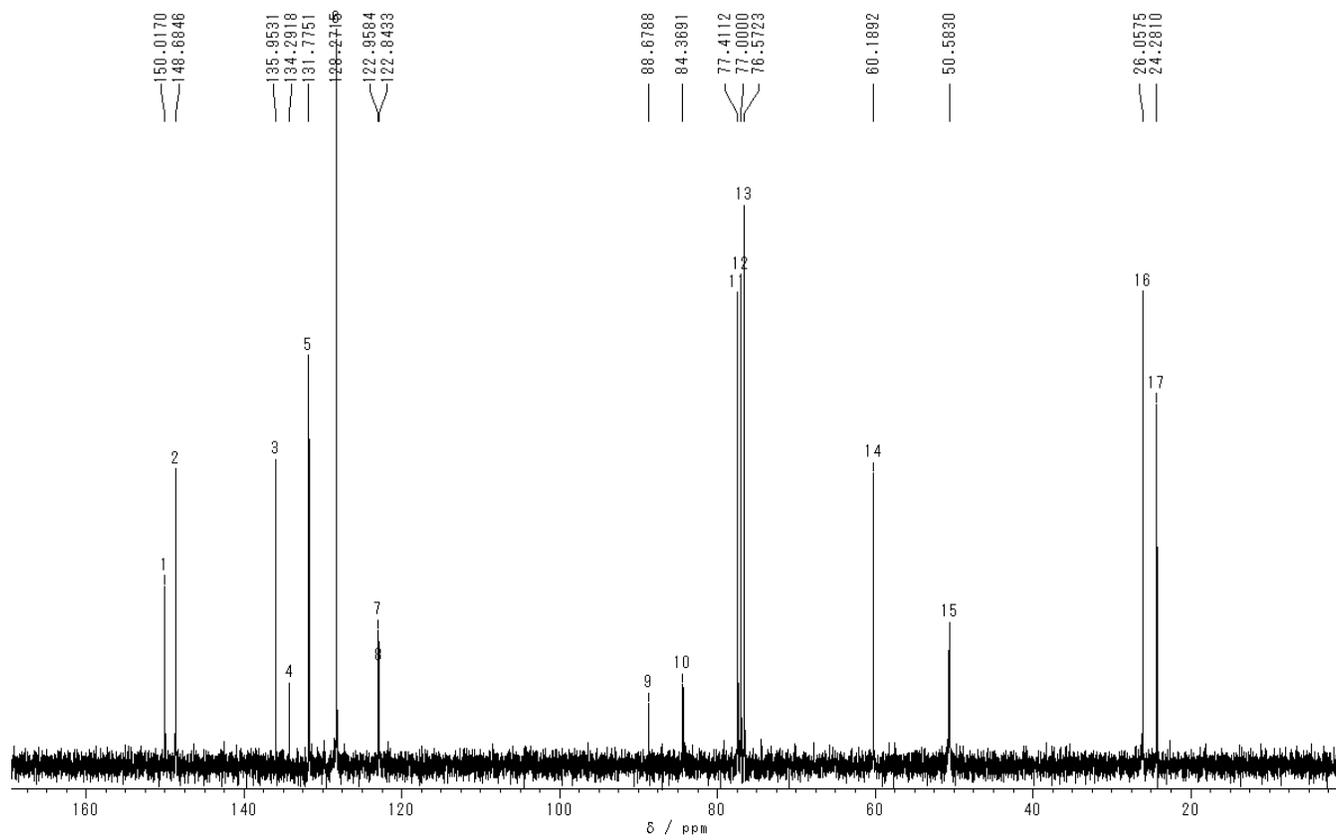
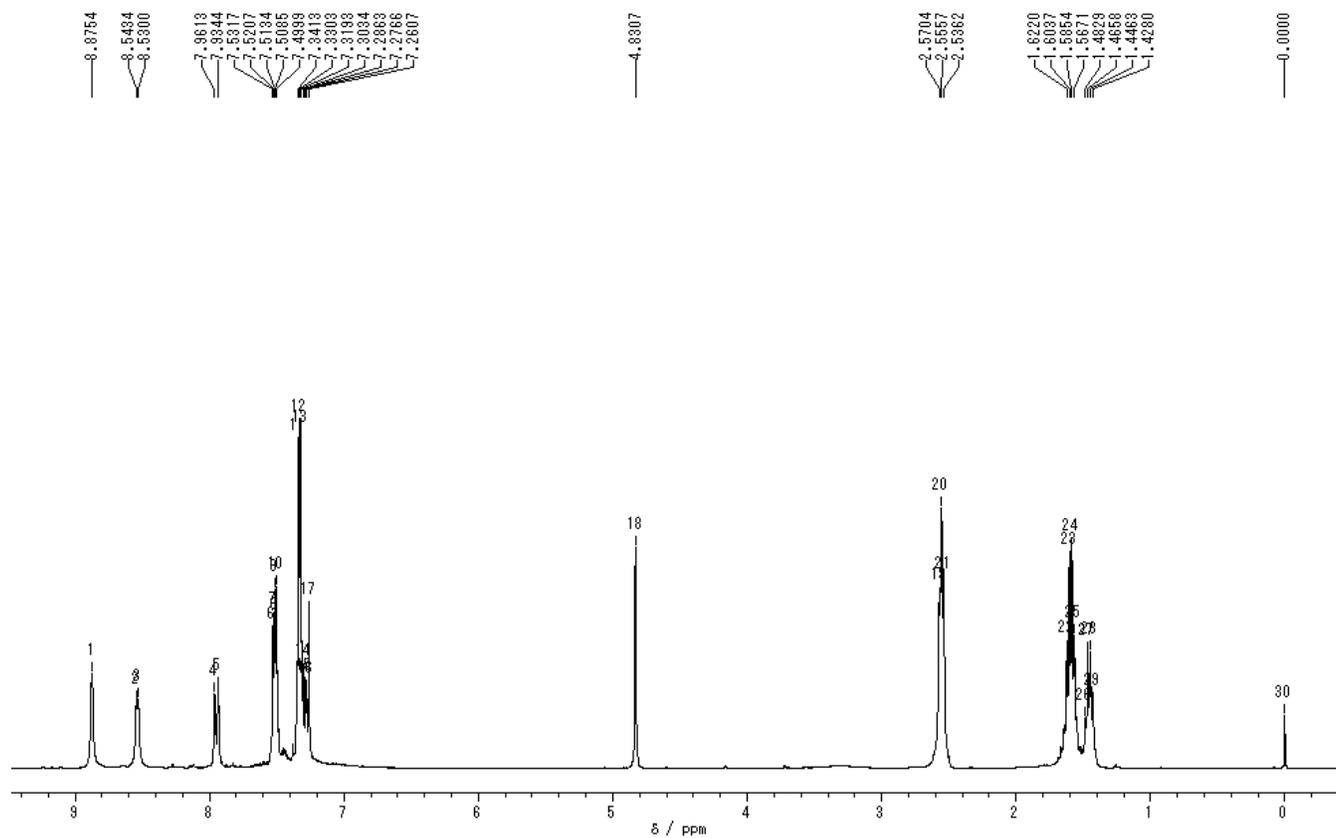
N-(1-(3-Chlorophenyl)-3-phenyl-2-propynyl)piperidine (3n)



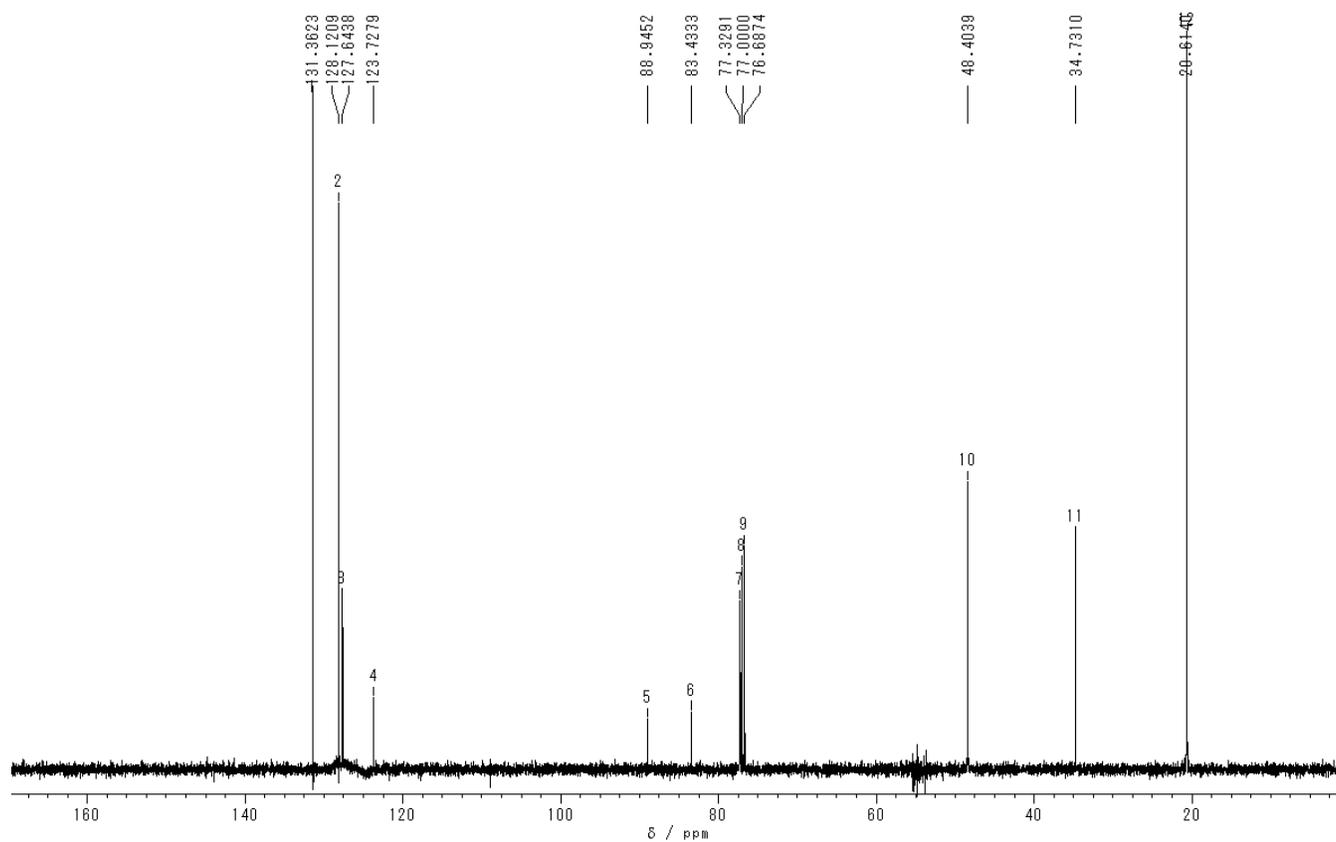
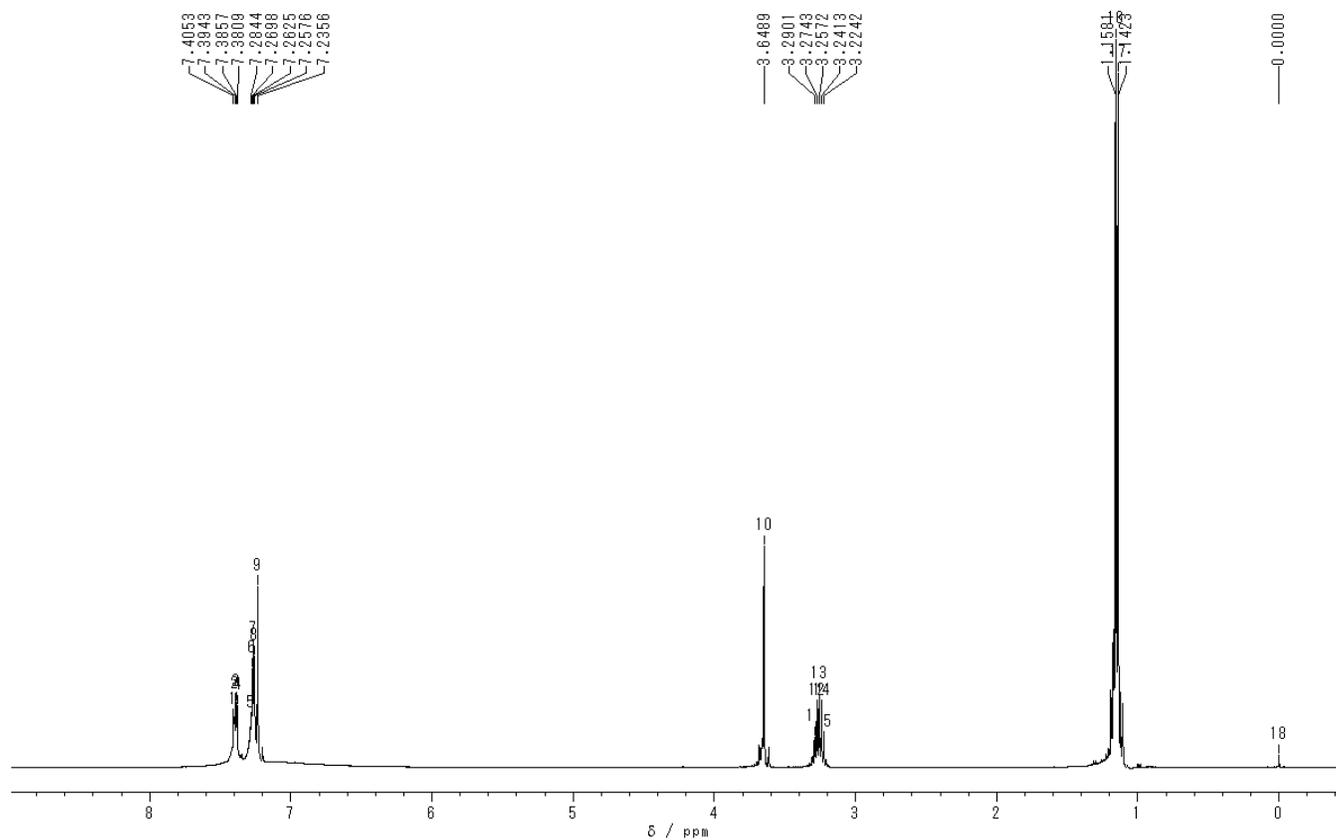
N-(1-(4-Biphenyl)-3-phenyl-2-propynyl)piperidine (30)



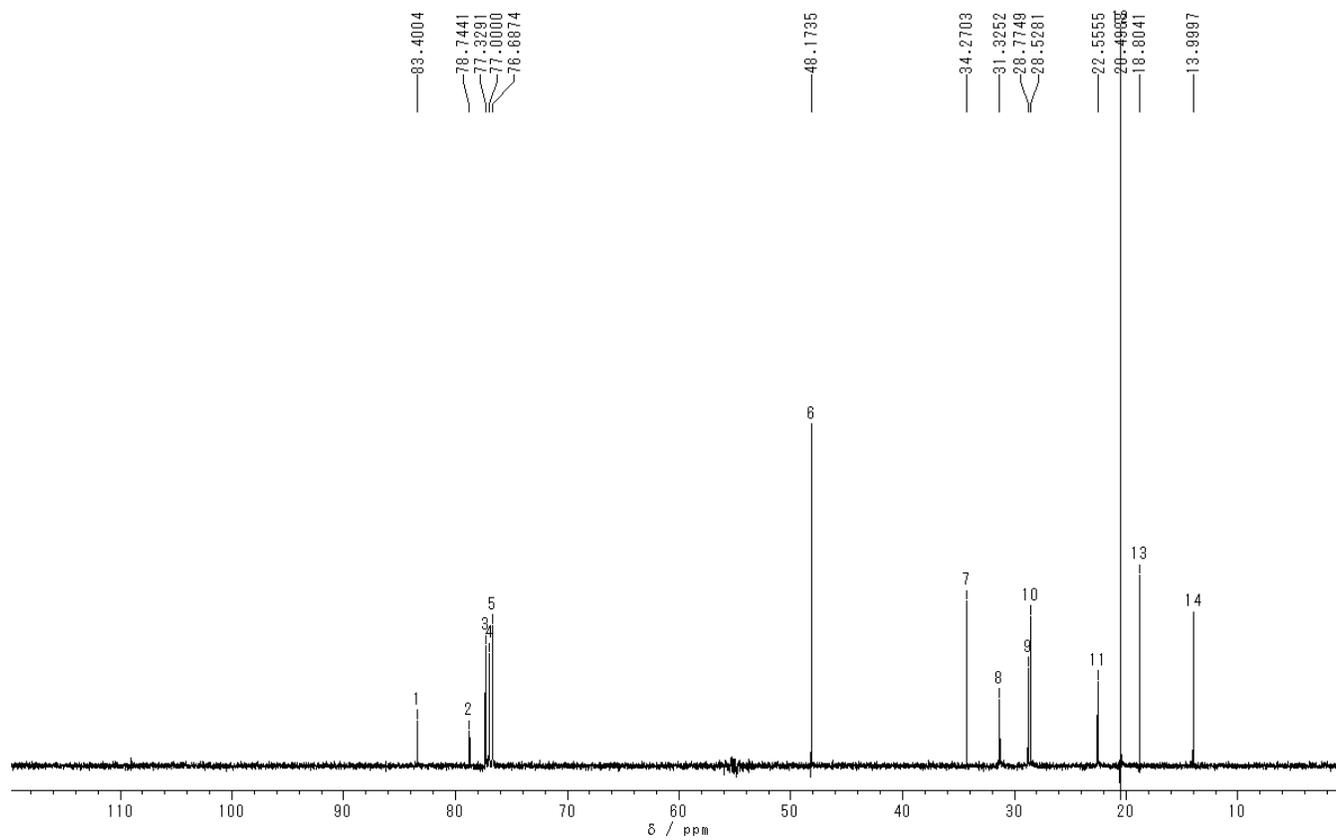
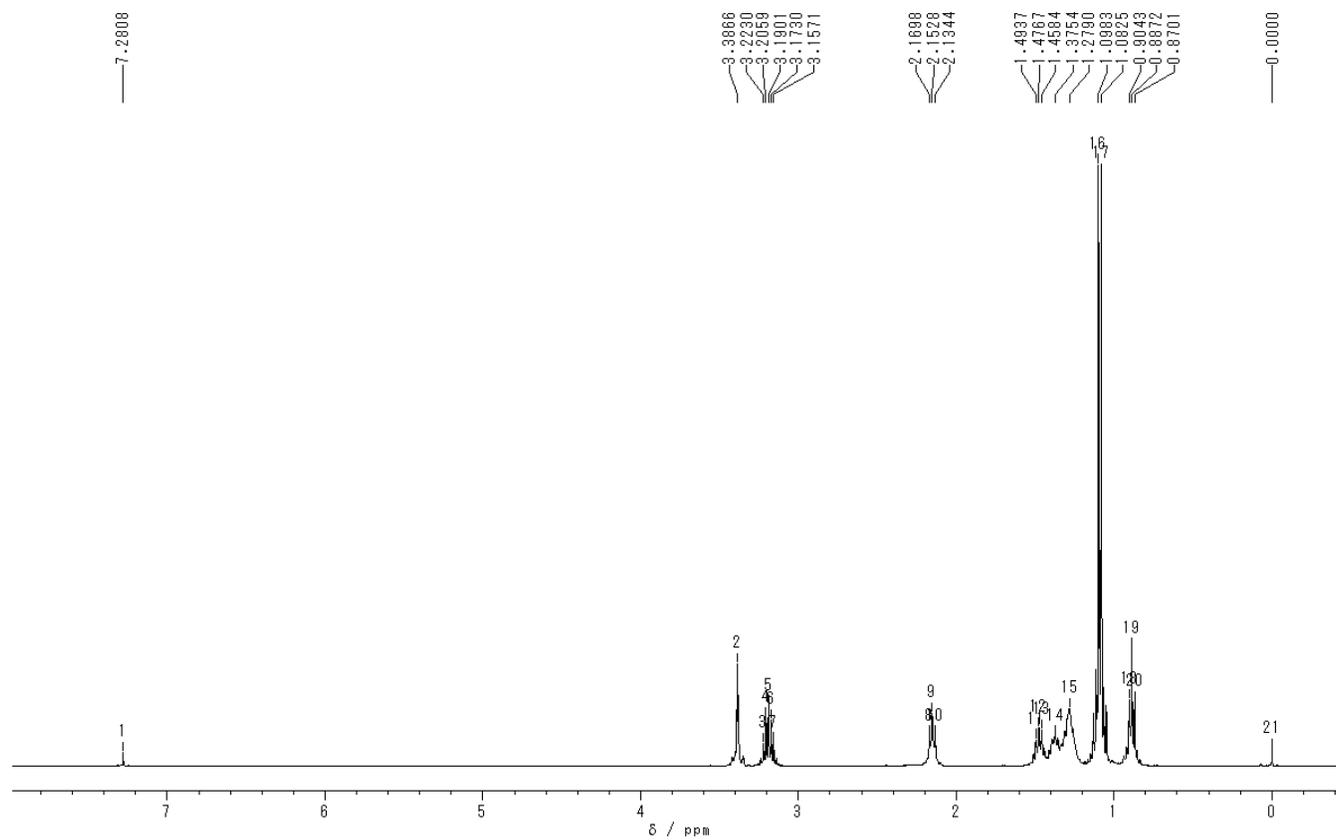
N-(3-Phenyl-1-(3-pyridyl)-2-propynyl)piperidine (3p)



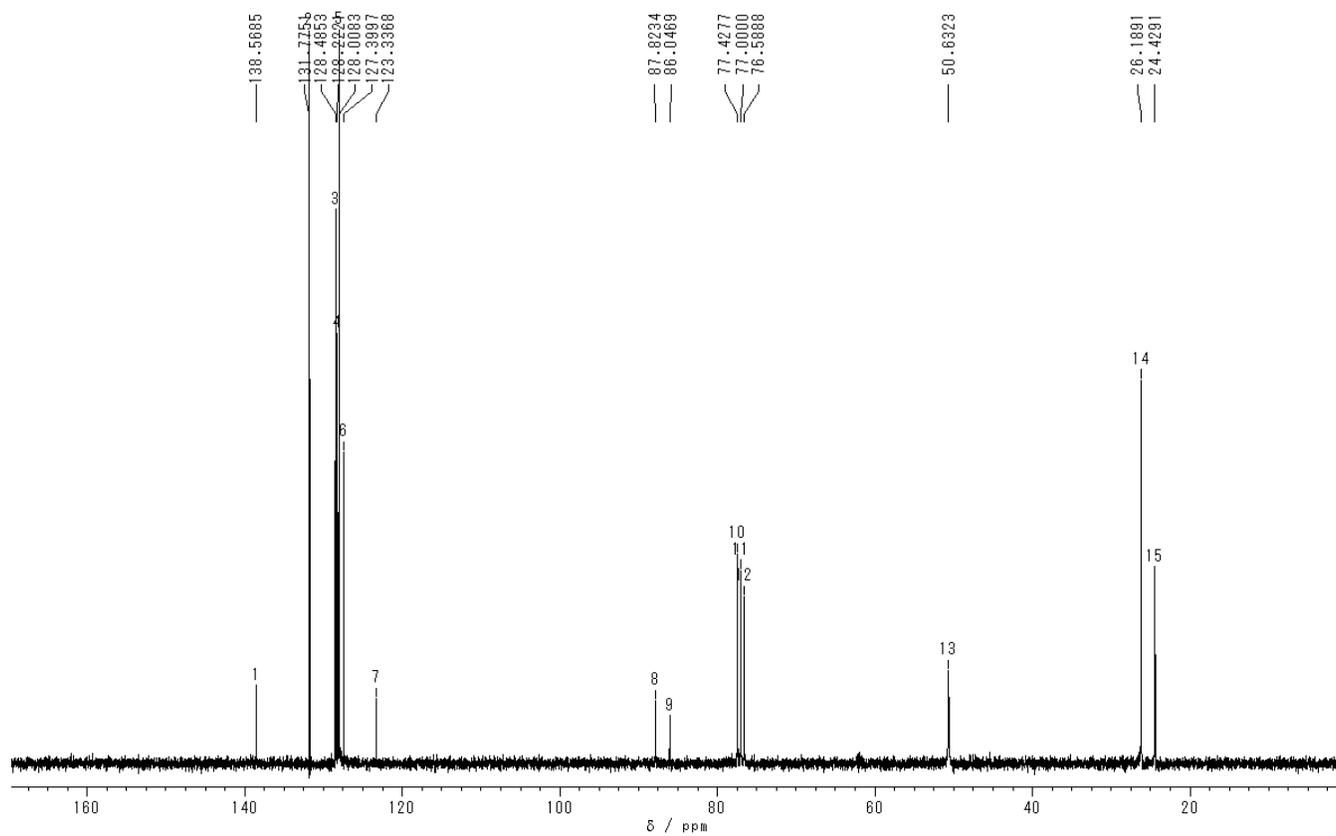
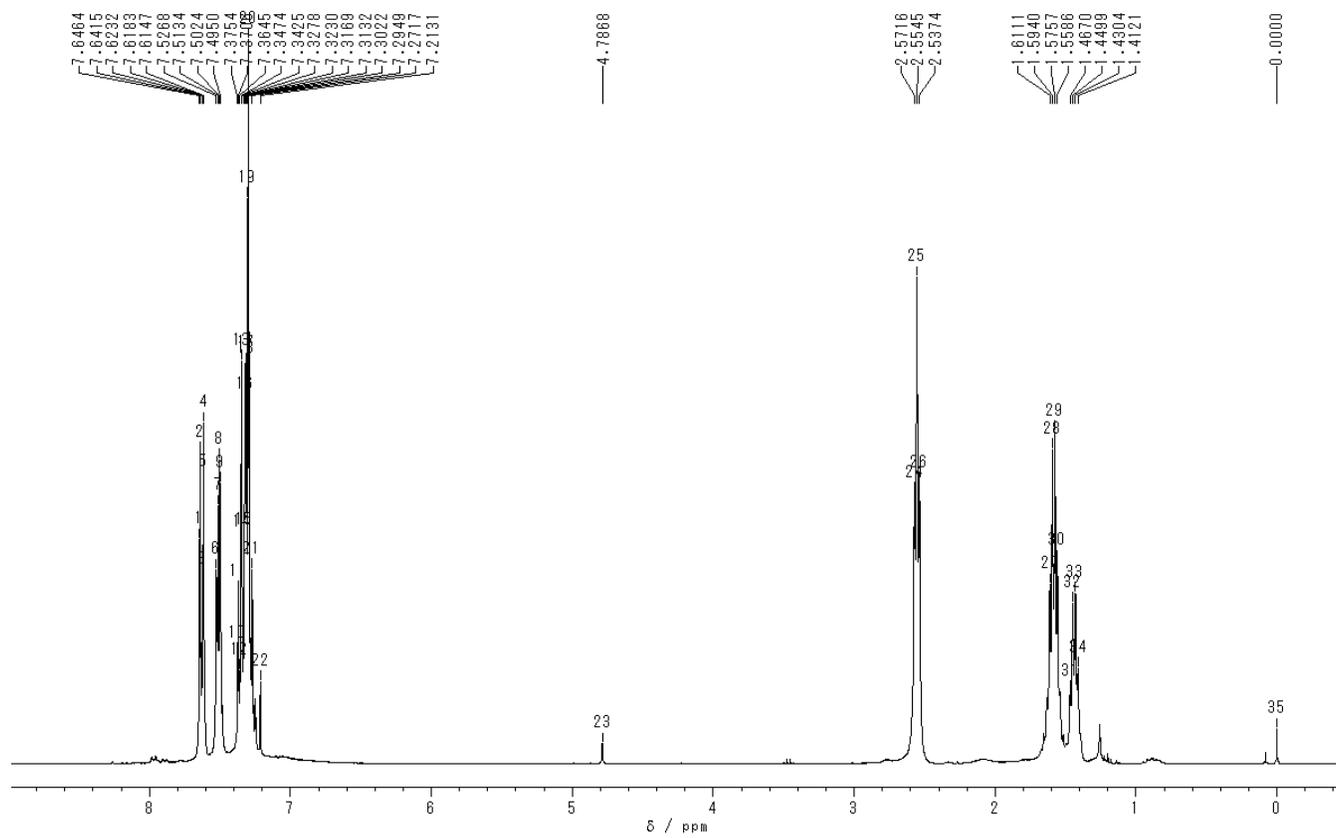
***N,N*-Diisopropyl-*N*-(3-phenyl-2-propynyl)amine (3s)**



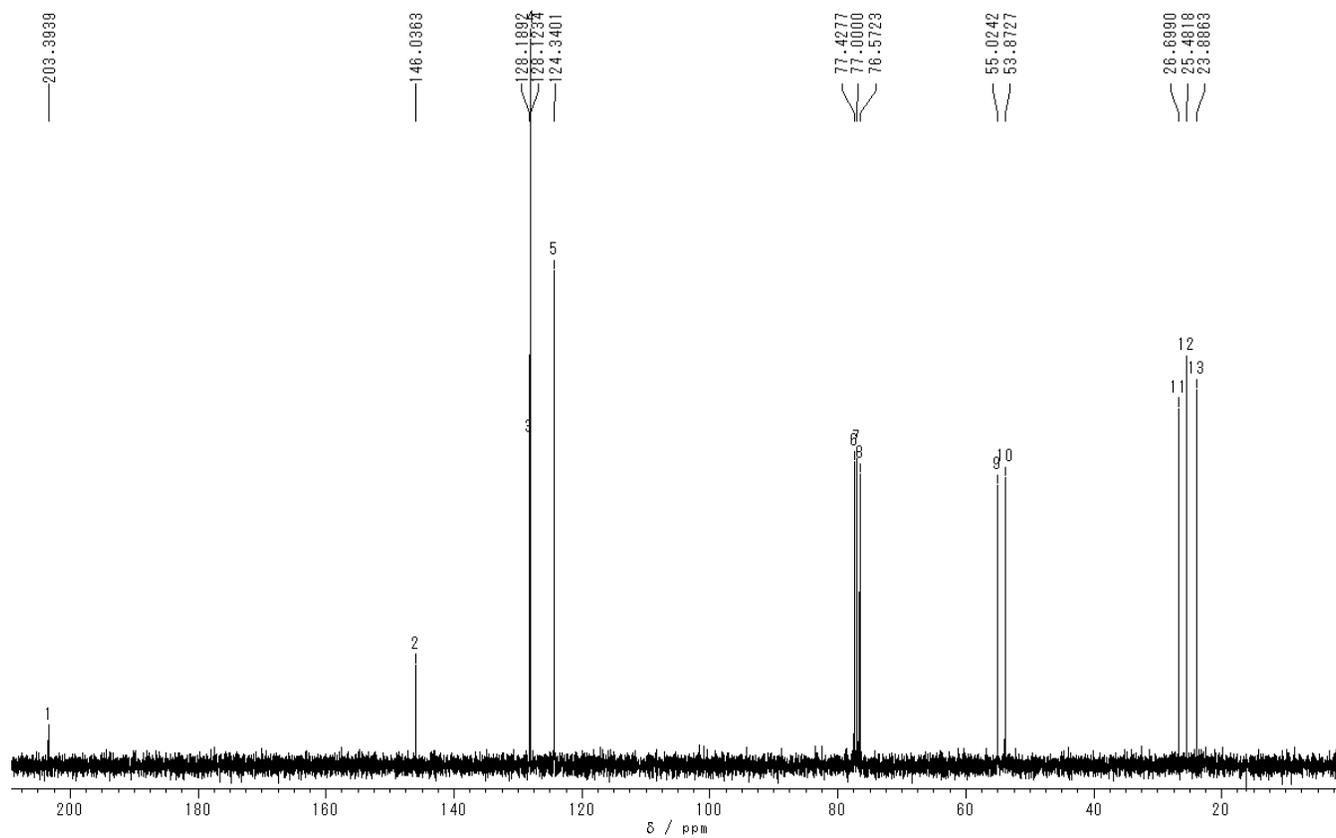
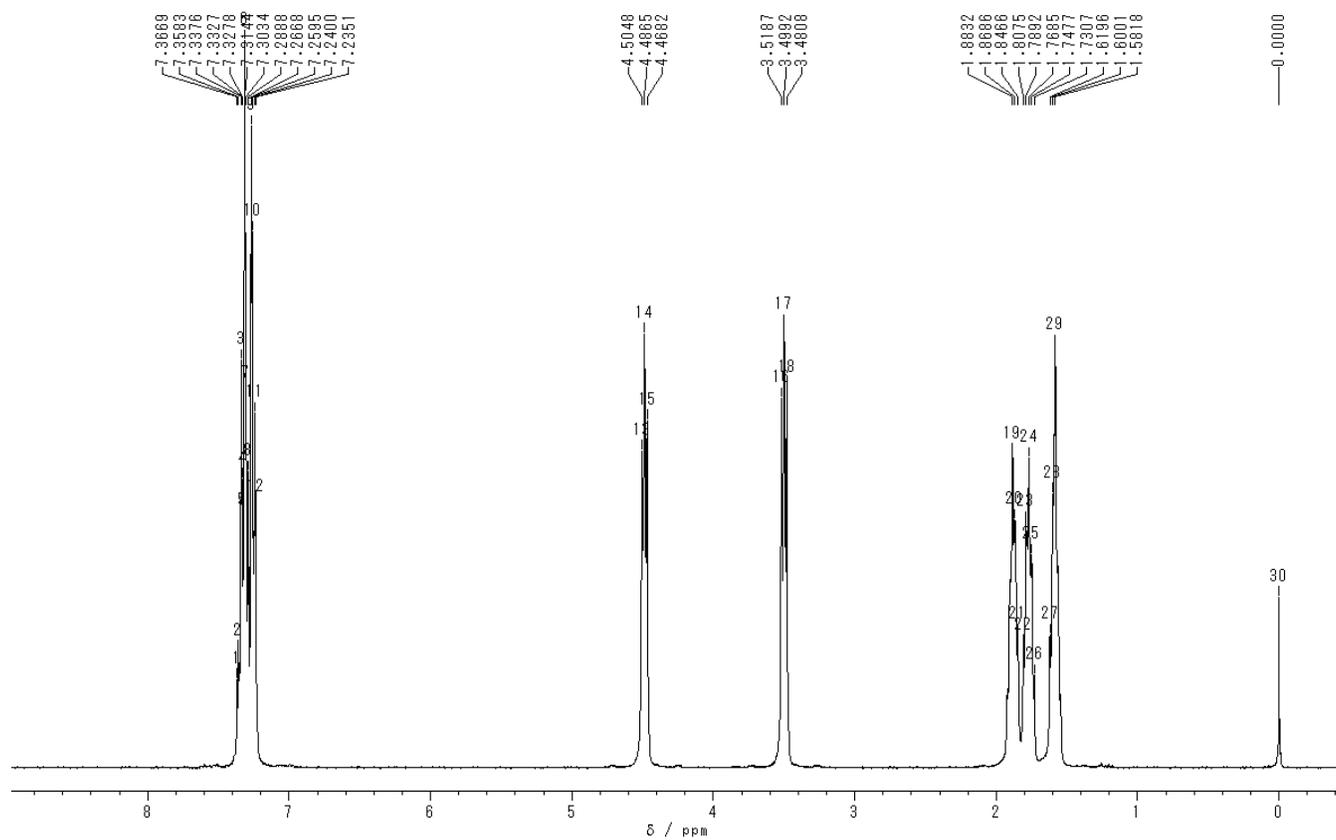
***N,N*-Diisopropyl-*N*-(2-nonyl)amine (3t)**



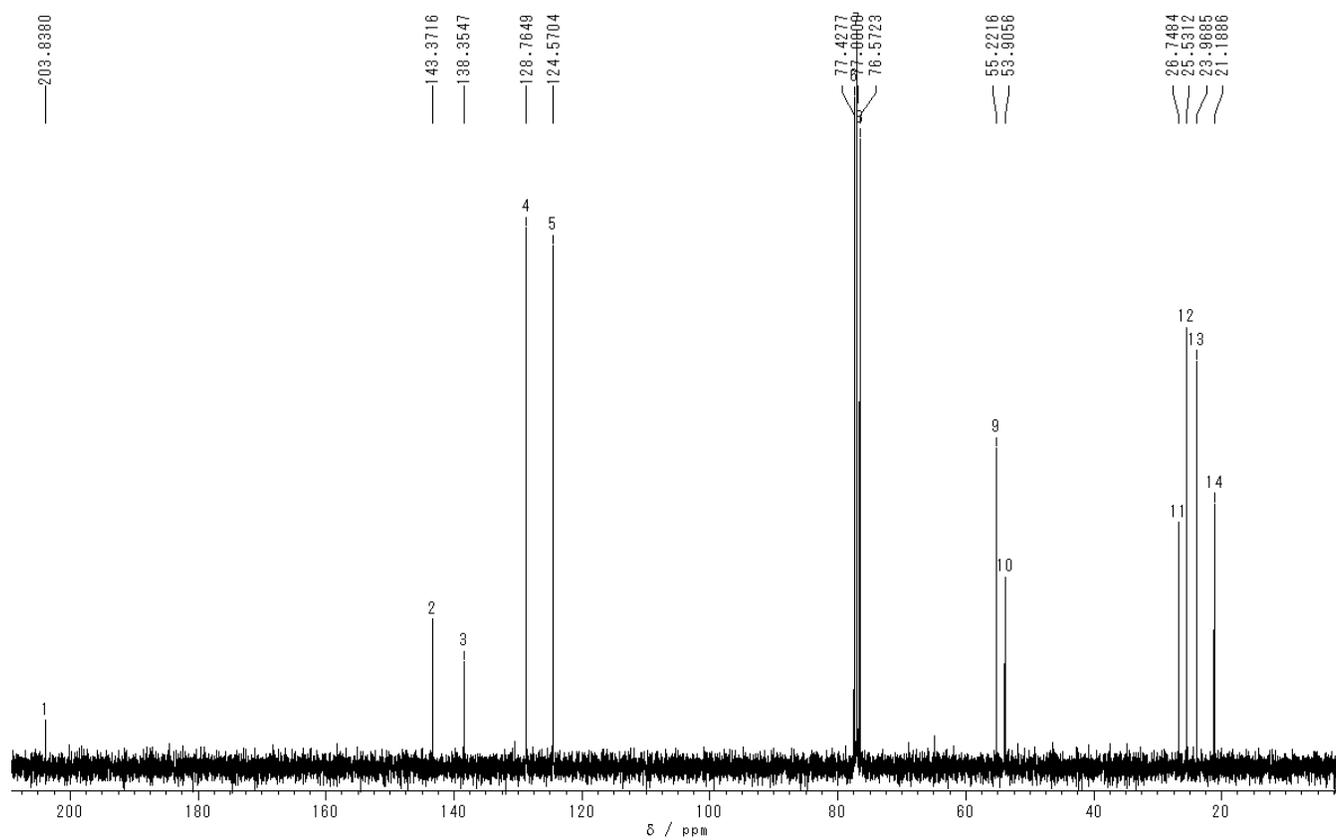
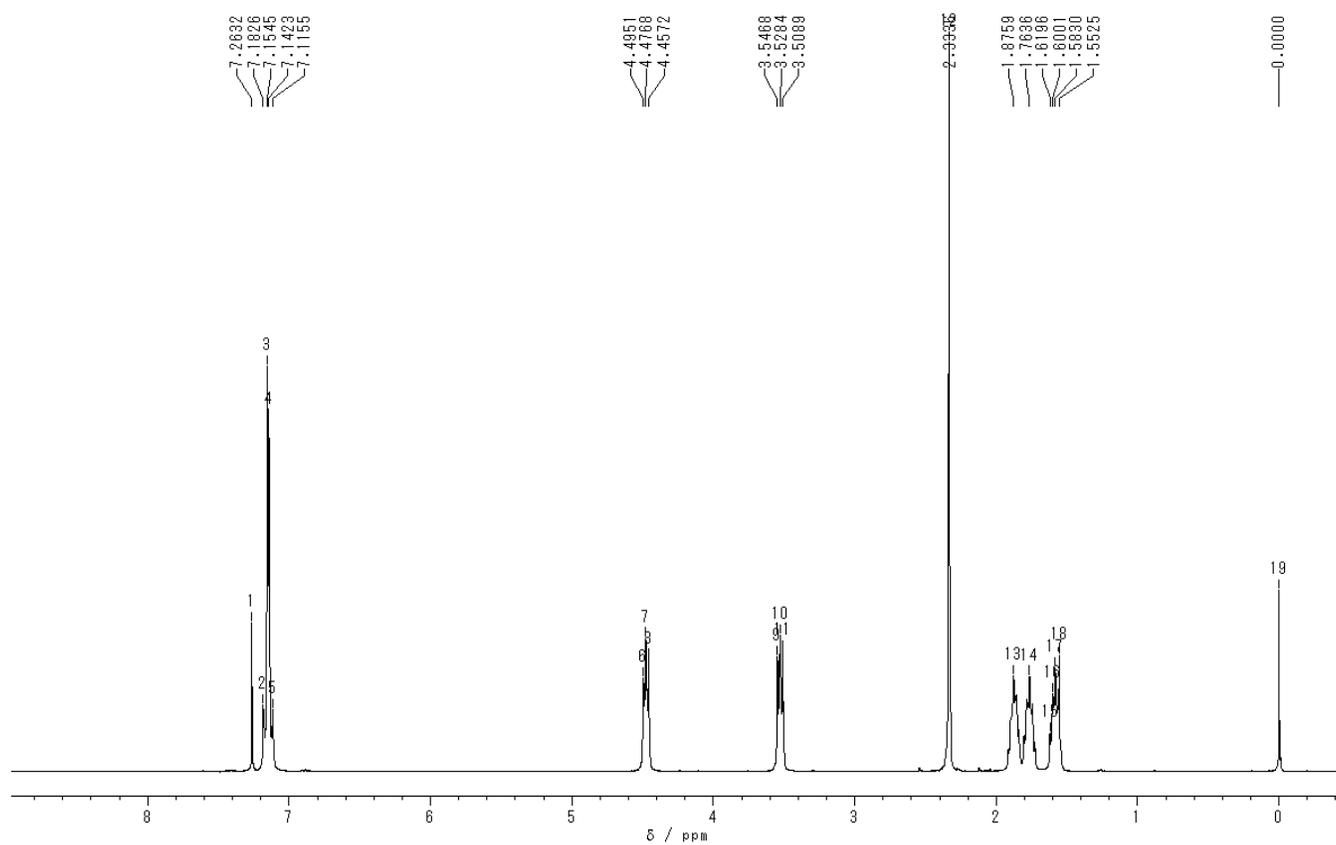
***N*-(1,3-Diphenyl-2-propynyl)piperidine-*d*¹ (3u)**



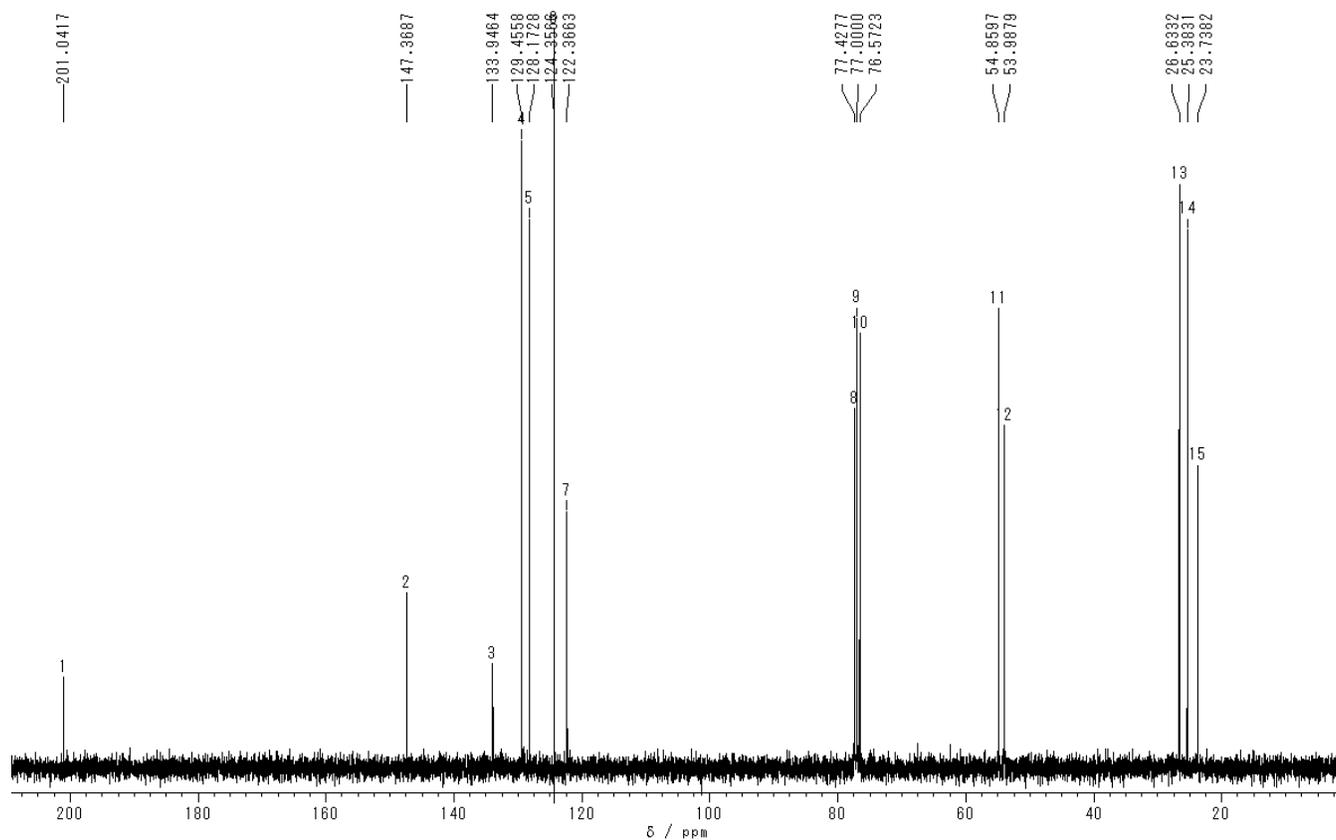
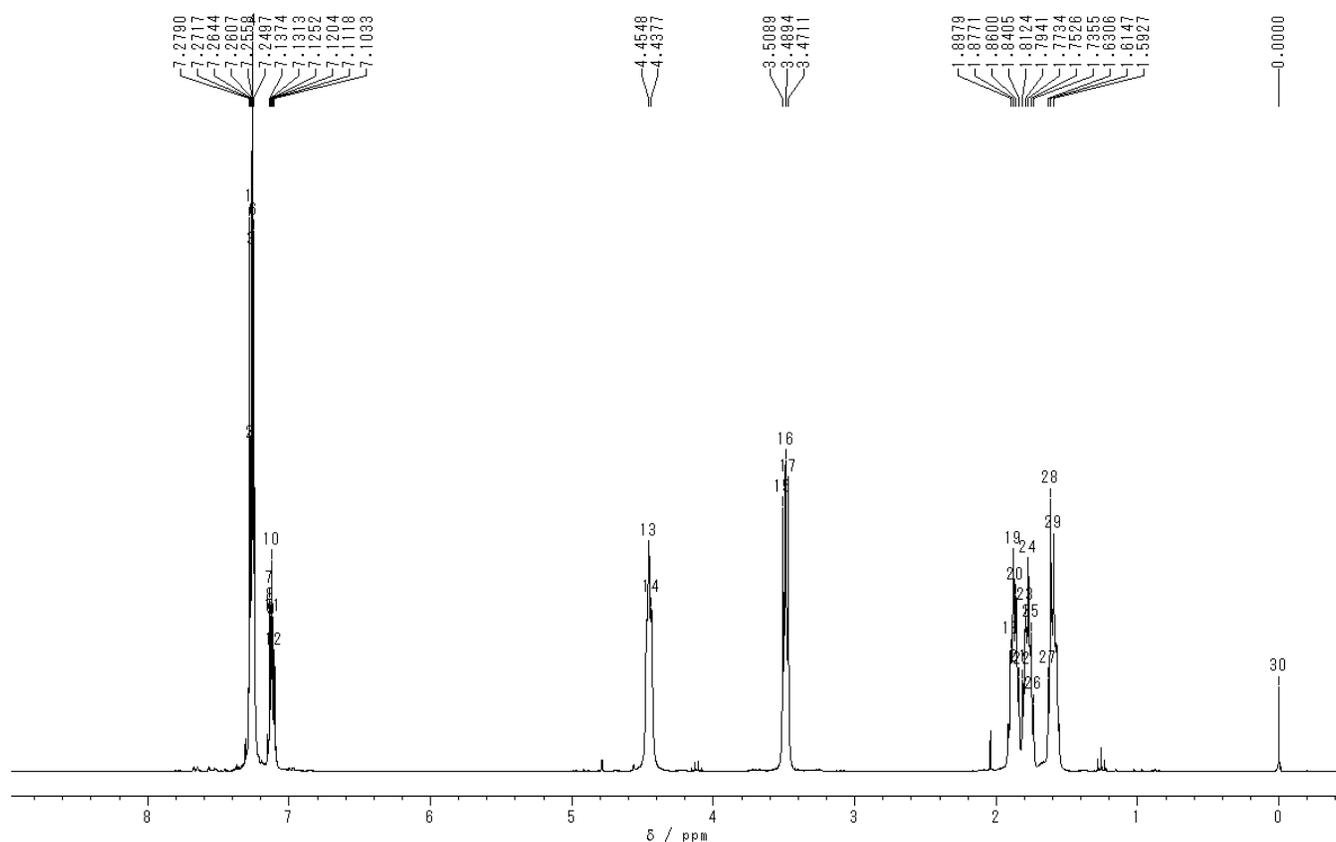
N-(Selenobenzoyl)piperidine (1a)



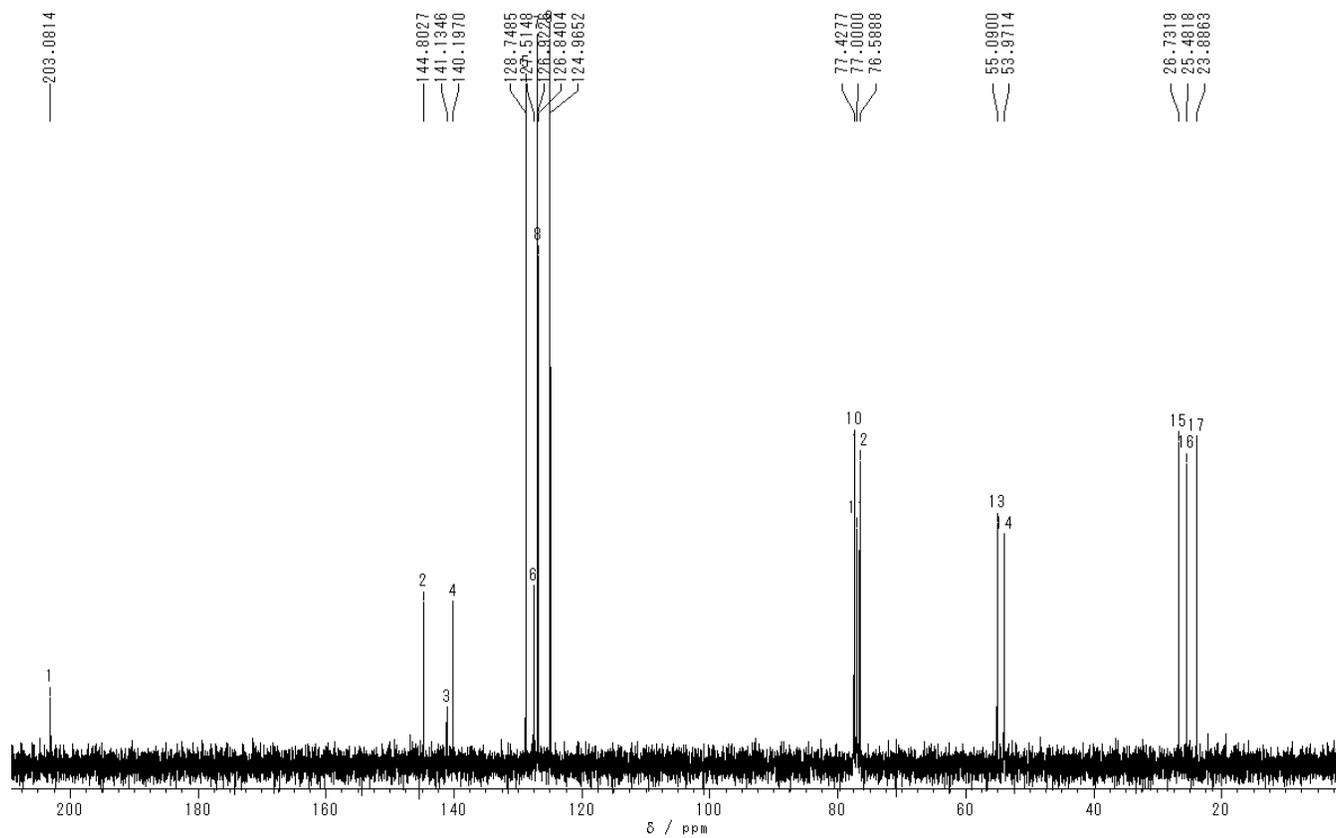
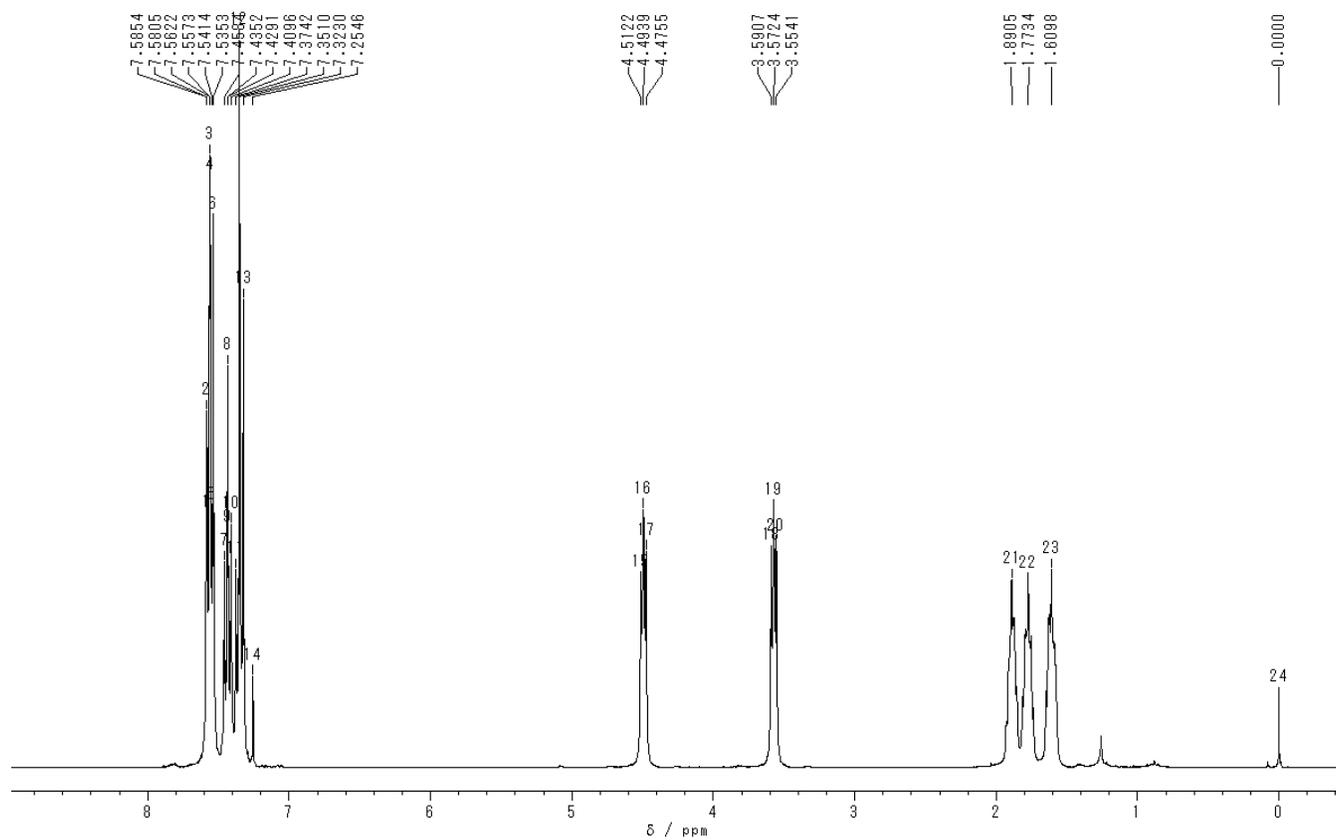
***N*-(*p*-Methyl-selenobenzoyl)piperidine (1b)**



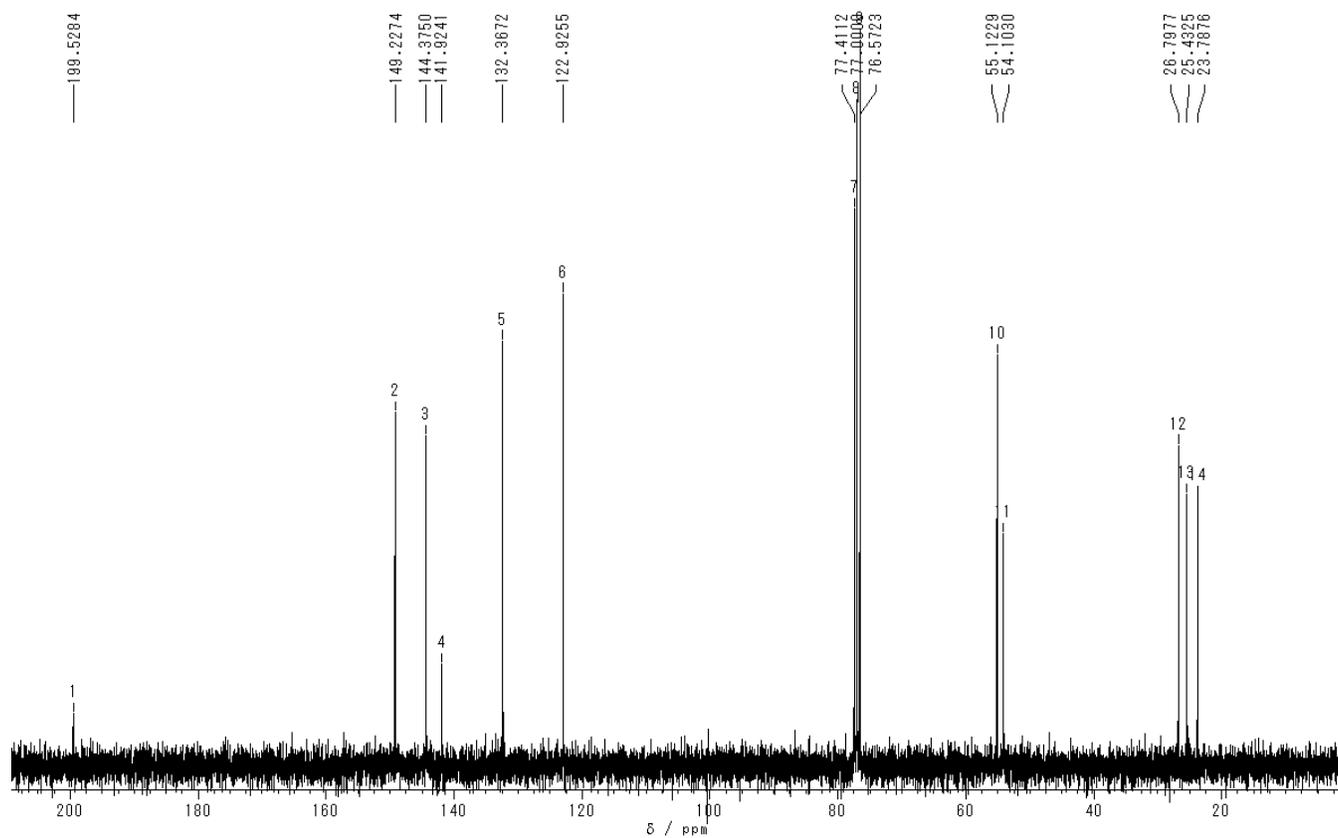
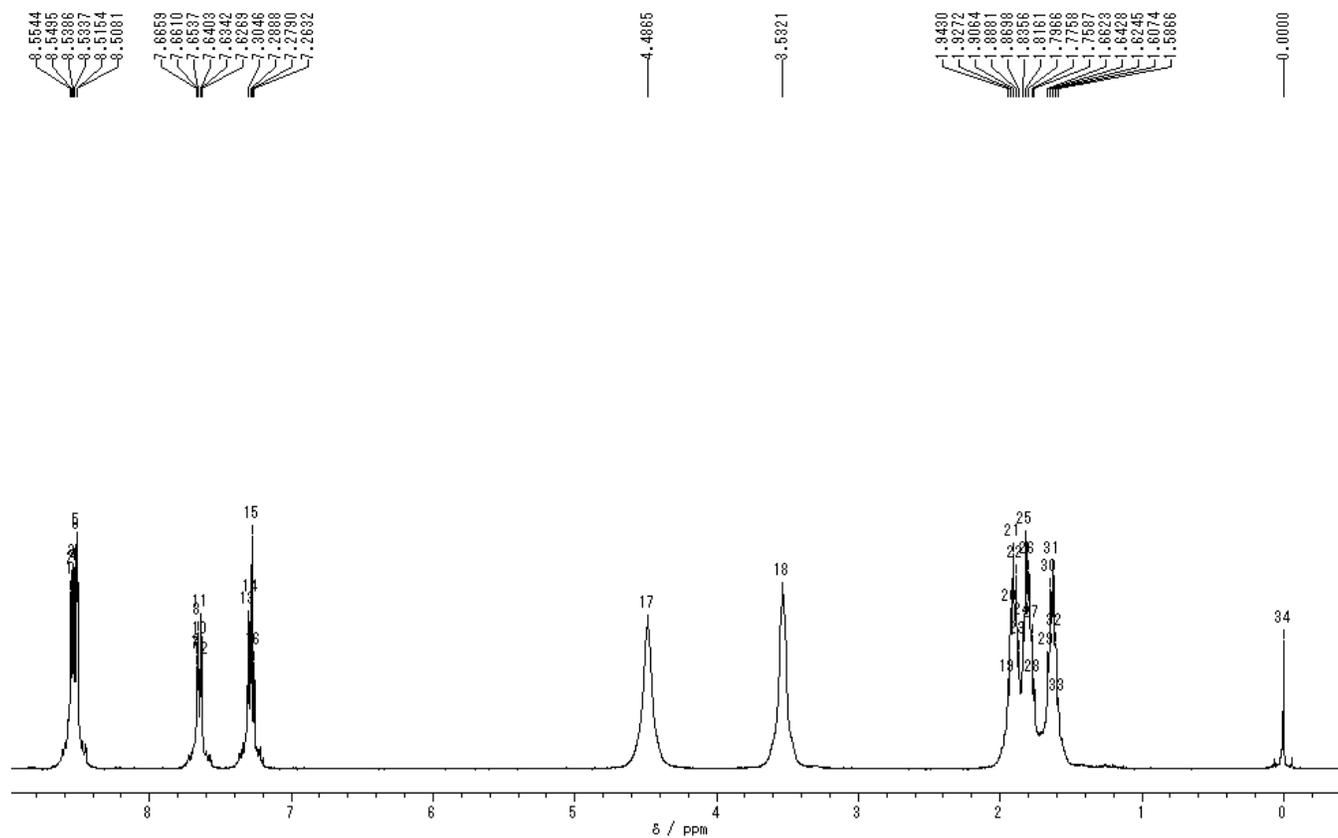
***N*-(*m*-Chloro-selenobenzoyl)piperidine (1c)**



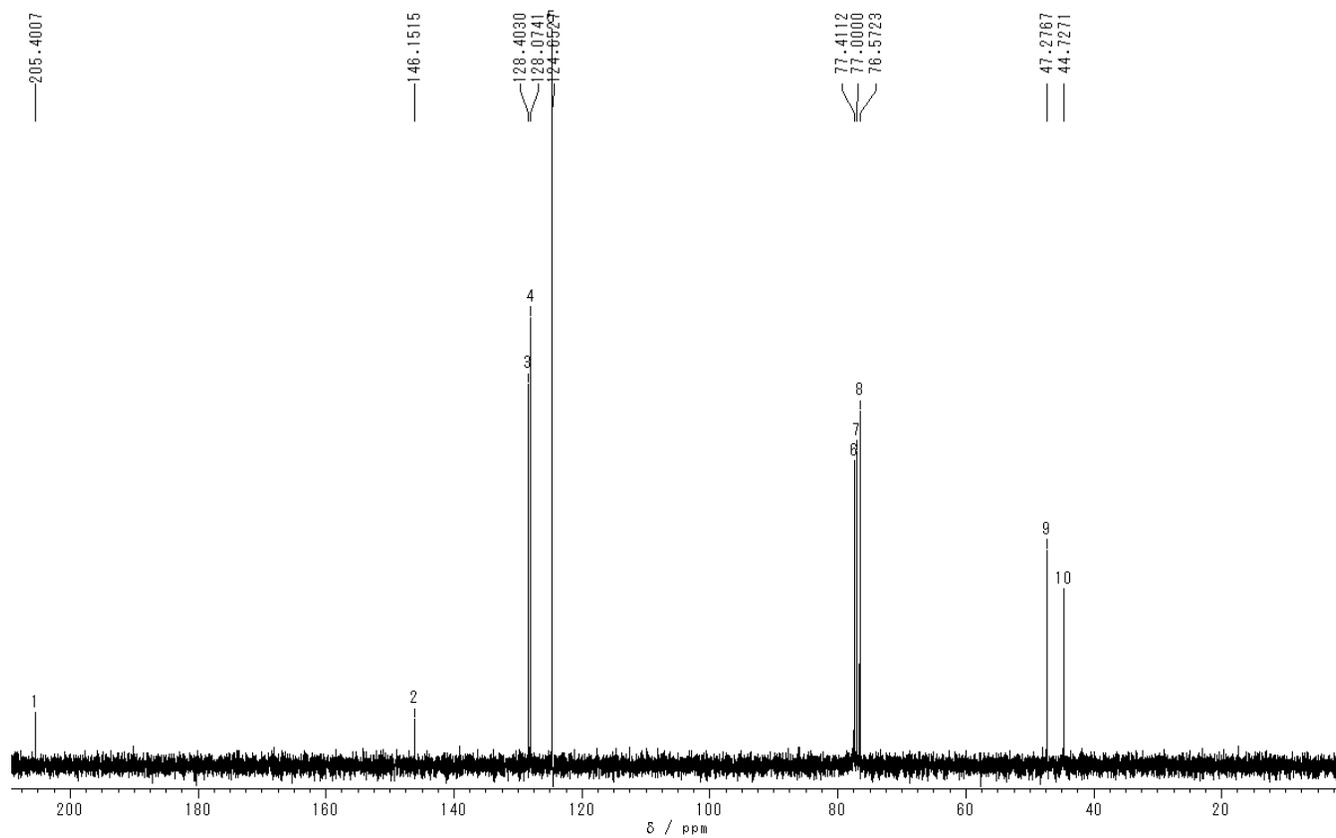
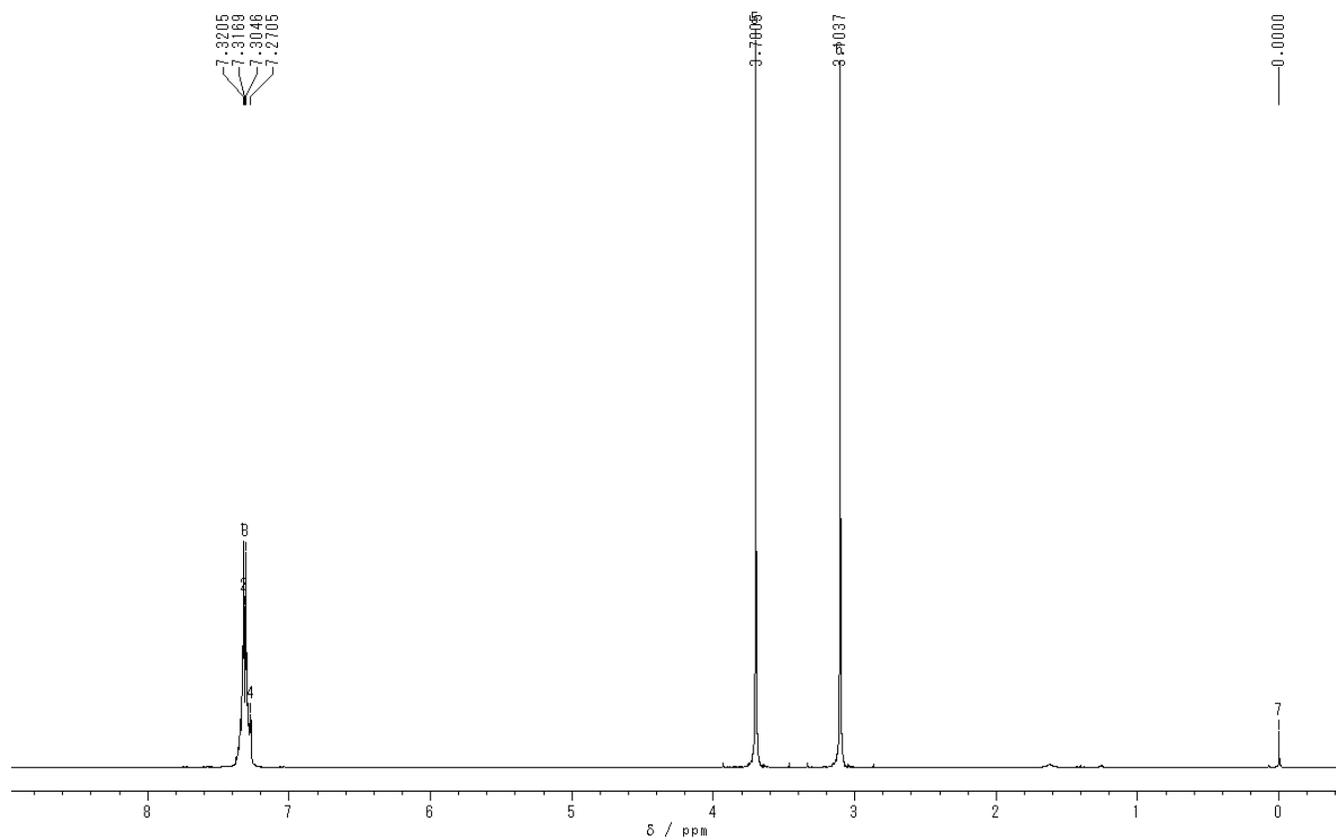
***N*-(*p*-Phenyl-selenobenzoyl)piperidine (1d)**



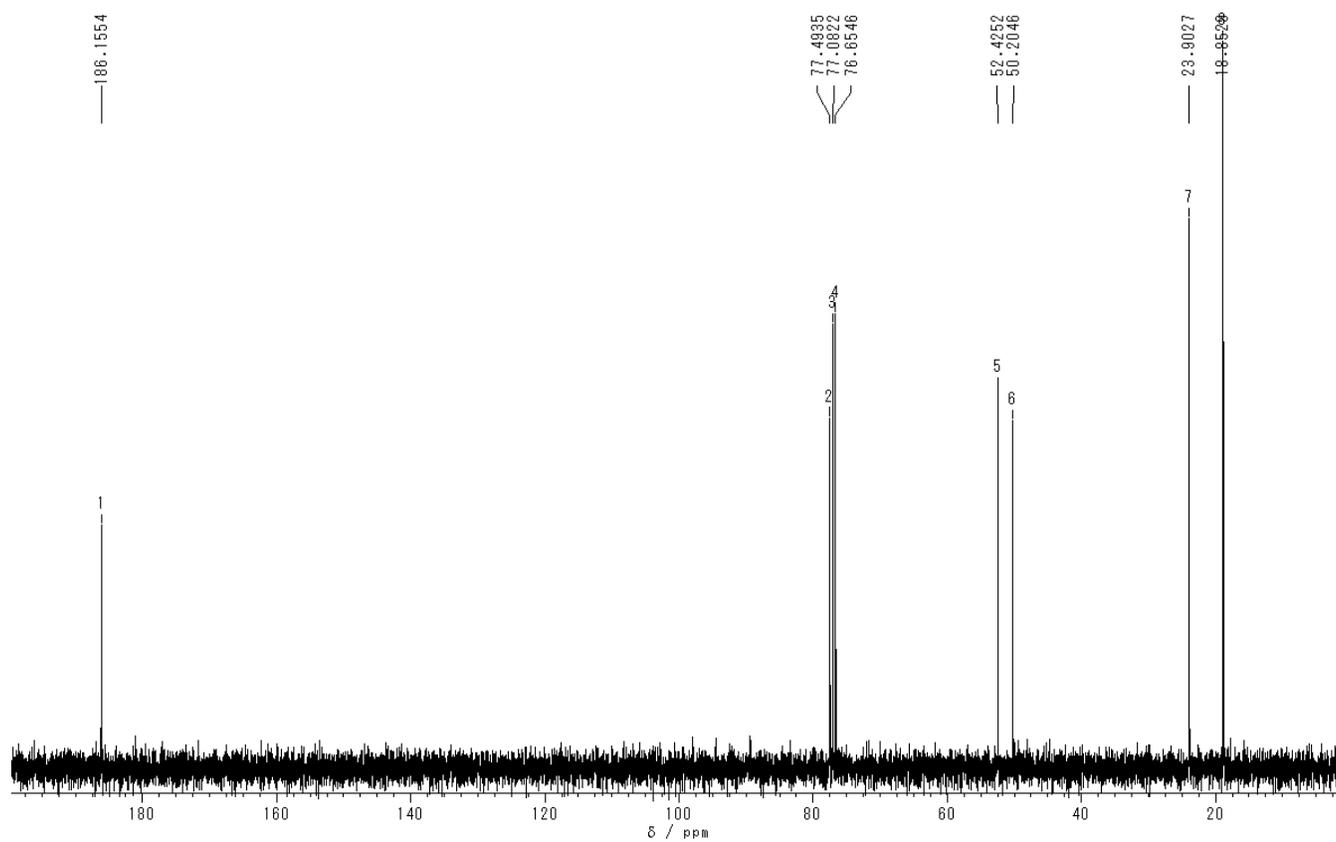
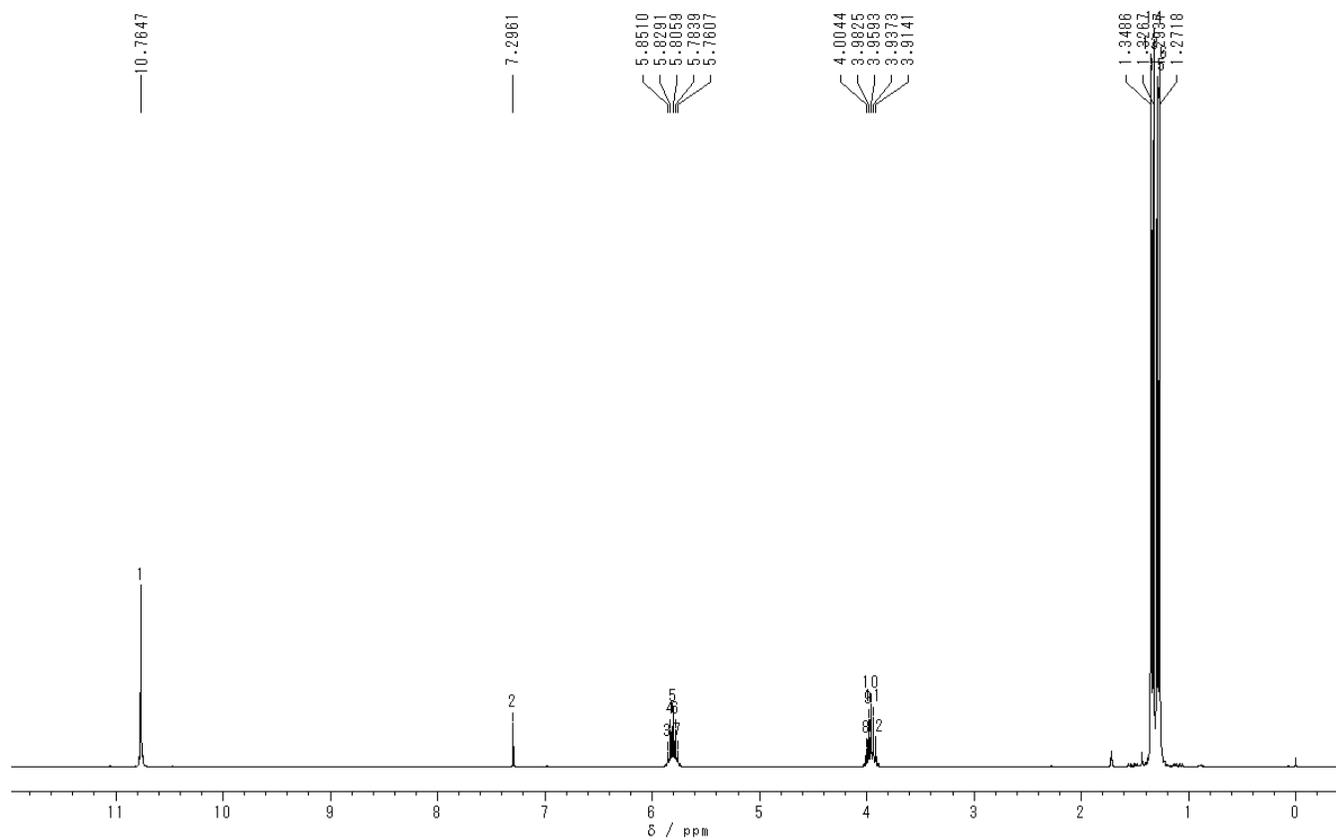
N-(3-Pyridyl-selenobenzoyl)piperidine (1e)



N,N-Dimethyl benzeneselenoamide (1f)



***N,N*-Diisopropyl selenoformamide (1h):**



References and Notes

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