Supporting Information

Palladium-catalyzed aerobic (1+2) annulation of Csp3-H bonds with olefin for the synthesis of 3-azabicyclo[3.1.0]hex-2-ene

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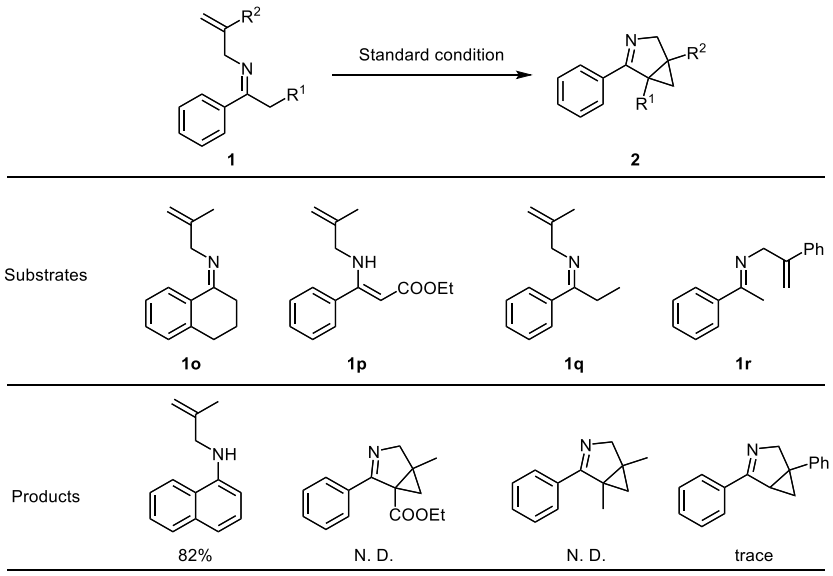
General information

The reactions were conducted under oxygen atmosphere with a balloon fitted on a Schlenk tube. All glassware was oven dried at 110 °C for hours and cooled down under vacuum. DMSO was purified by distillation with calcium hydride. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Imines were prepared following literature procedures. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 100-200 mesh silica gel in petroleum (bp. 60-90 °C). GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. NMR spectra were recorded on a Bruker Advance III spectrometers at 400 MHz (^{1}H NMR), 100 MHz (^{13}C NMR). Tetramethylsilane was used as an internal standard. All ^{1}H NMR spectra were reported in delta (δ) units, parts per million (ppm) downfield from the internal standard. Coupling constants (*J*) are reported in Hertz (Hz). High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT instrument, accurate masses are reported for the molecular ion ([M+H]$^+$). IR spectra were recorded on a Mettler Toledo React IR^{TM} 4000 spectrometer using a diamond comb.

Table S1. Other type substrates for this annulation reaction[a]



[a] Reaction conditions: **1** (0.25 mmol), $Pd(OAc)_2$ (0.025 mmol), Bu_4NBr (0.25 mmol), DMSO 1.0 mL, 80 °C, 15 h, under O_2 (1 atm); Yields shown are of isolated products.

Tetralone-derived imine **1o** underwent dehydrogenative aromatization to afford aminonaphthalene via β-hydride elimination. Enamine **1p** and α-methyl imine **1q**

were not suitable for this annulation reaction. And instead of methyl, phenyl substrate **1r** only gave trace mount of the desired product.

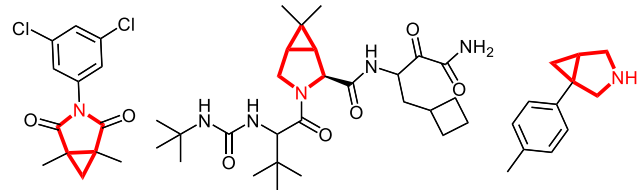
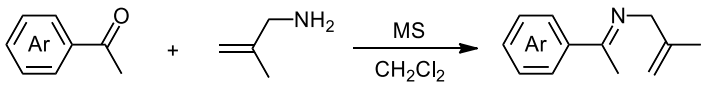


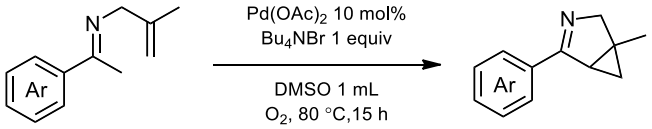
Figure S1. Selected examples of pharmacologically active 3-azabicyclo[3.1.0]hexanes

General procedure for the synthesis of imines 1a-n [1]



A mixture of amine (3.0 mmol), ketone (2.5 mmol), and activiated 4Å molecular sieves (4.0 g) in anhydrous dichloromethane (10 mL) was stirred at room temperature for 24 h, and then filtered through celite. The filtrate was concentrated under vacuum. The residue was subjected to distillation or recrystallation to give pure imine.

General procedure for the [1+2] annulation of Csp3-H bonds with olefin

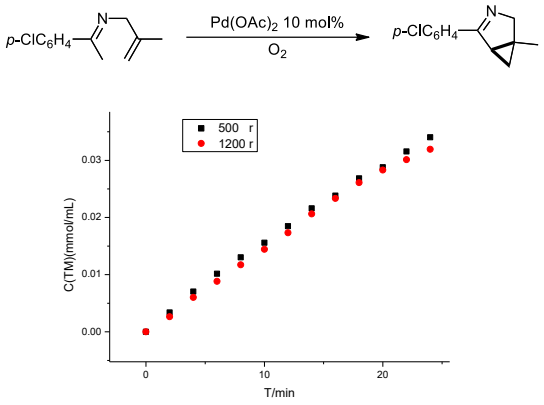


$Pd(OAc)_2$ (5.6 mg, 0.025 mmol), Bu_4NBr (80.5 mg, 0.25 mmol) was added in a Schlenk tube. The Schlenk tube was then sealed with septa and fitted with an oxygen balloon, filled with oxygen. DMSO (1 mL) and imine (0.25 mmol) were injected in the tube via a syringe. The reaction was then heated up to 80 °C. After stirring for 15 hours, it was quenched by water and extracted with ethyl ether (3 * 10 mL). The organic layers were combined and pure product was obtained by flash column chromatography on silica gel (petroleum: ethyl acetate = 40:1).

In-situ IR experiments

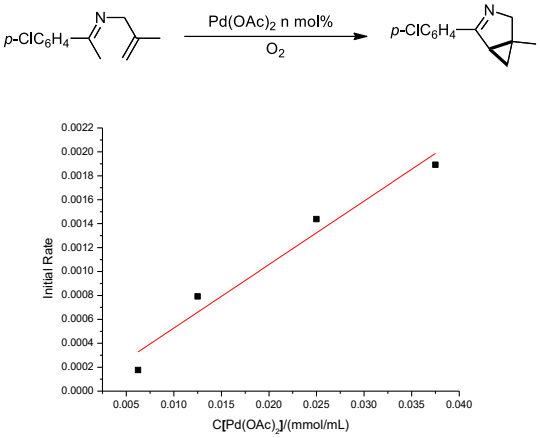
Procedure for the effect of stirring speed

In an oven dried self-prepared three-necked micro reactor with a magnetic stirrer, $Pd(OAc)_2$ (0.05 mmol)and Bu_4NBr (161 mg, 0.5 mmol) were added. The reactor was then put onto in-situ IR and sealed with septa and fitted with an oxygen balloon, filled with oxygen. DMSO (2.0 mL) were injected in the tube via syringe. The mixture was allowed to stir at 80 °C and recorded by React IR. 5 mins later, imine **1j** (0.5 mmol) was added in by a micro syringe. The course of the reaction can be observed from the characteristic IR band of product **2j** at 730 cm^{-1}. When the peak of **2j** has been stable completely, the reaction quenched by EA. The yield was determined by GC with biphenyl as internal standard.



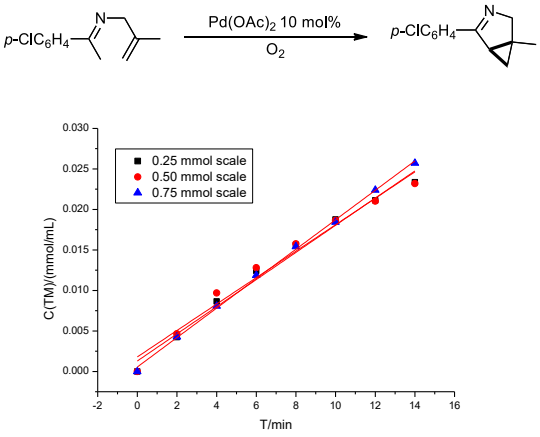
Procedure for the effect of concentration of catalyst

In an oven dried self-prepared three-necked micro reactor with a magnetic stirrer, $Pd(OAc)_2$ (0.0125mmol, 0.025 mmol, 0.05 mmol, 0.75 mmol)and Bu_4NBr (161 mg, 0.5 mmol) were added. The reactor was then put onto in-situ IR and sealed with septa and fitted with an oxygen balloon, filled with oxygen. DMSO (2.0 mL) were injected in the tube via syringe. The mixture was allowed to stir at 80 °C and recorded by React IR. 5 mins later, imine **1j** (0.5 mmol) was added in by a micro syringe. The course of the reaction can be observed from the characteristic IR band of product **2j** at 730 cm^{-1}. When the peak of **2j** has been stable completely, the reaction quenched by EA. The yield was determined by GC with biphenyl as internal standard.



Procedure for the effect of concentration of substrate

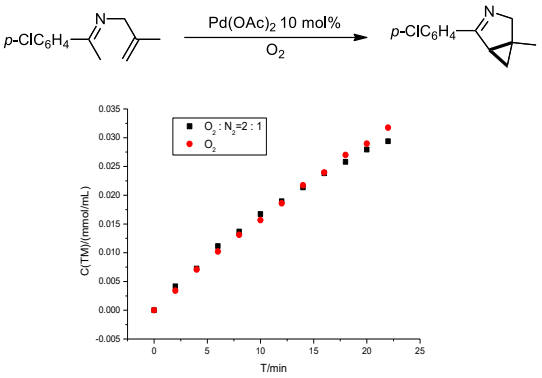
In an oven dried self-prepared three-necked micro reactor with a magnetic stirrer, $Pd(OAc)_2$ (0.05 mmol)and Bu_4NBr (161 mg, 0.5 mmol) were added. The reactor was then put onto in-situ IR and sealed with septa and fitted with an oxygen balloon, filled with oxygen. DMSO (2.0 mL) were injected in the tube via syringe. The mixture was allowed to stir at 80 °C and recorded by React IR. 5 mins later, imine **1j** (0.25 mmol, 0.5 mmol, 0.75 mmol) was added in by a micro syringe. The course of the reaction can be observed from the characteristic IR band of product **2j** at 730 cm^{-1}. When the peak of **2j** has been stable completely, the reaction quenched by EA. The yield was determined by GC with biphenyl as internal standard.



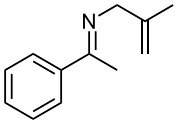
Procedure for the effect of partial pressure of dioxygen

In an oven dried self-prepared three-necked micro reactor with a magnetic stirrer, $Pd(OAc)_2$ (0.05 mmol) and Bu_4NBr (161 mg, 0.5 mmol) were added. The reactor was then put onto in-situ IR and sealed with septa and fitted with an oxygen balloon, filled with oxygen or mixture gas. DMSO (2.0 mL) were injected in the tube via syringe. The mixture was allowed to stir at 80 °C and recorded by React IR. 5 mins later, imine **1j** (0.5 mmol) was added in by a micro syringe. The

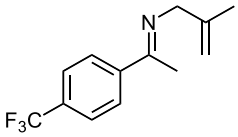
course of the reaction can be observed from the characteristic IR band of product **2j** at 730 cm^{-1}. When the peak of **2j** has been stable completely, the reaction quenched by EA. The yield was determined by GC with biphenyl as internal standard.



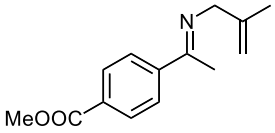
Detail descriptions for substrates and products



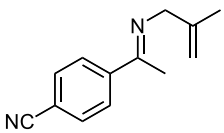
(E)-2-methyl-*N*-(1-phenylethylidene)prop-2-en-1-amine (1a): light yellow liquid, 0.316 g, isolated yield = 73%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.74 (m, 2H), 7.39 – 7.45 (m, 3H), 5.00 (d, *J* = 0.4 Hz, 1H), 4.97 – 4.88 (m, 1H), 4.12 (s, 2H), 2.28 (s, 3H), 1.93 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.0, 143.8, 141.4, 129.7, 128.4, 126.9, 110.7, 58.0, 21.6, 15.6. IR (film): 3063, 3024, 2970, 2914, 2852, 1633, 1446, 1369, 1280, 1132, 1026, 892, 760, 692, 573 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{15}N$ M^+: 173.1204; found: 173.1199.



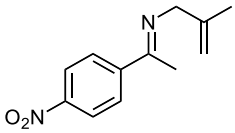
(E)-2-methyl-*N*-(1-(4-(trifluoromethyl)phenyl)ethylidene)prop-2-en-1-amine (1b): colourless liquid, 0.470 g, isolated yield = 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 4.98 (s, 1H), 4.93 (s, 1H), 4.12 (s, 2H), 2.29 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 144.4, 143.6, 131.5 (q, *J* = 32.5 Hz), 127.3, 125.4 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 273.1 Hz), 110.9, 58.2, 21.6, 15.7. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.65. IR (film): 3079, 2976, 2914, 2858, 1636, 1409, 1326, 1167, 1127, 1089, 1067, 1015, 896, 846, 730, 606 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{14}F_3N$ M^+: 241.1078; found: 241.1076.



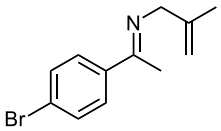
(E)-methyl 4-(1-((2-methylallyl)imino)ethyl)benzoate (1c): white solid, 0.514 g, isolated yield = 89%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 – 7.97 (m, 2H), 7.97 – 7.80 (m, 2H), 4.95 (s, 1H), 4.89 (s, 1H), 4.07 (s, 2H), 3.91 (s, 3H), 2.25 (s, 3H), 1.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 165.1, 145.1, 143.5, 130.9, 129.6, 126.8, 110.8, 58.1, 52.3, 21.5, 15.7. IR (film): 3073, 2952, 2914, 2852, 1724, 1634, 1436, 1276, 1112, 1017, 894, 860, 773, 701, 570 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{17}NO_2$ M^+: 231.1259; found: 231.1260.



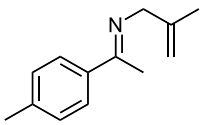
(E)-4-(1-((2-methylallyl)imino)ethyl)benzonitrile (1d): orange liquid, 0.321 g, isolated yield = 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.68 (dd, *J* = 8.4, 2.0 Hz, 2H), 4.95 (s, 1H), 4.91 (s, 1H), 4.10 (s, 2H), 2.27 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3, 145.0, 143.4, 132.2, 127.5, 118.9, 113.1, 111.0, 58.2, 21.5, 15.6. IR (film): 3077, 2973, 2917, 2855, 2228, 1633, 1445, 1403, 1371, 1280, 1088, 1018, 896, 841, 576, 535 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{14}N_2$ M^+: 198.1157; found: 198.1158.



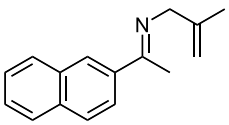
(E)-2-methyl-*N*-(1-(4-nitrophenyl)ethylidene)prop-2-en-1-amine (1e): light yellow liquid, 0.447 g, isolated yield = 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 – 8.16 (m, 2H), 8.14 – 7.85 (m, 2H), 4.97 (s, 1H), 4.93 (s, 1H), 4.12 (s, 2H), 2.31 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 148.7, 146.7, 143.4, 127.8, 123.7, 111.1, 58.4, 21.5, 15.8. IR (film): 3079, 2973, 2917, 2855, 1695, 1636, 1600, 1520, 1348, 1279, 1086, 895, 856, 754, 693, 563 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{14}N_2O_2$ M^+: 218.1055; found: 218.1053.



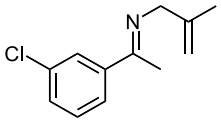
(E)-*N*-(1-(4-bromophenyl)ethylidene)-2-methylprop-2-en-1-amine (1f): colourless liquid, 0.552 g, isolated yield = 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 4.96 (s, 1H), 4.91 (s, 1H), 4.07 (s, 2H), 2.23 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 143.7, 140.0, 131.5, 128.5, 124.2, 110.8, 58.0, 21.6, 15.4. IR (film): 3076, 2969, 2913, 2854, 1633, 1587, 1483, 1394, 1283, 1085, 1009, 895, 828, 566 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{14}BrN$ M^+: 251.0310; found: 251.0305.



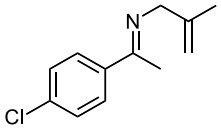
(E)-2-methyl-*N*-(1-(p-tolyl)ethylidene)prop-2-en-1-amine (1g): colourless liquid, 0.407 g, isolated yield = 87%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.97 (s, 1H), 4.91 (s, 1H), 4.10 (s, 2H), 2.41 (s, 3H), 2.26 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 143.9, 139.7, 138.6, 129.1, 126.8, 110.5, 57.8, 21.6, 21.5, 15.5. IR (film): 3073, 3026, 2971, 2920, 2855, 1632, 1445, 1368, 1289, 1183, 1088, 1019, 893, 816, 571 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{17}N$ M^+: 187.1361; found: 187.1357.



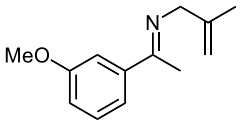
(E)-2-methyl-*N*-(1-(naphthalen-2-yl)ethylidene)prop-2-en-1-amine (1h): white solid, 0.396 g, isolated yield = 71%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 8.22 – 8.11 (m, 1H), 7.98 – 7.92 (m, 1H), 7.88 (d, *J* = 8.8 Hz, 2H), 7.61 – 7.47 (m, 2H), 5.05 (s, 1H), 5.02 – 4.93 (m, 1H), 4.19 (s, 2H), 2.40 (s, 3H), 1.97 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 143.9, 138.6, 134.3, 133.2, 129.0, 128.0, 127.9, 126.9, 126.8, 126.4, 124.5, 110.7, 58.1, 21.7, 15.6. IR (film): 3059, 2971, 2913, 2852, 1627, 1443, 1371, 1290, 1230, 1194, 1128, 890, 859, 823, 748, 476 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{17}N$ M^+: 223.1361; found: 223.1364.



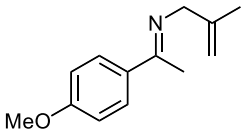
(E)-*N*-(1-(3-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1i): colourless liquid, 0.455 g, isolated yield = 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (t, *J* = 1.8 Hz, 1H), 7.72 (dt, *J* = 7.5, 1.4 Hz, 1H), 7.48 – 7.16 (m, 2H), 4.97 (s, 1H), 4.92 (s, 1H), 4.09 (s, 2H), 2.25 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 143.6, 143.0, 134.5, 129.7, 129.6, 127.1, 125.0, 110.9, 58.0, 21.6, 15.6. IR (film): 3076, 2973, 2914, 2852, 1635, 1568, 1422, 1370, 1286, 1259, 1073, 894, 785, 684 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{14}ClN$ M^+: 207.0815; found: 207.0809.



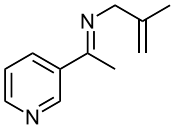
(E)-*N*-(1-(4-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1j): light yellow liquid, 0.378 g, isolated yield = 73%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.6 Hz, 2H), 7.37 (d, *J* = 8.6 Hz, 2H), 4.96 (s, 1H), 4.91 (s, 1H), 4.08 (s, 2H), 2.24 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 143.7, 139.6, 135.8, 128.5, 128.2, 110.8, 58.0, 21.6, 15.5. IR (film): 3072, 2973, 2908, 2852, 1633, 1594, 1488, 1399, 1285, 1093, 1012, 893, 832, 766, 566 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{14}ClN$ M^+: 207.0815; found: 207.0817.



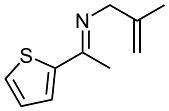
(E)-*N*-(1-(3-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1k): colourless liquid, 0.370 g, isolated yield = 73%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.44 (m, 1H), 7.44 – 7.39 (m, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 6.99 – 6.95 (m, 1H), 4.97 (s, 1H), 4.91 (s, 1H), 4.10 (s, 2H), 3.88 (s, 3H), 2.26 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 159.8, 143.8, 142.9, 129.4, 119.5, 115.7, 112.0, 110.7, 57.9, 55.6, 21.6, 15.9. IR (film): 3076, 2970, 2934, 2834, 1685, 1634, 1580, 1485, 1429, 1368, 1287, 1226, 1044, 892, 784, 691, 571 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{17}NO$ M^+: 203.1310; found: 203.1309.



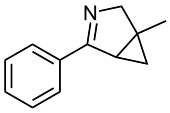
(E)-*N*-(1-(4-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1l): light yellow solid, 0.386 g, isolated yield = 76%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.6 Hz, 2H), 4.97 (s, 1H), 4.90 (s, 1H), 4.08 (s, 2H), 3.84 (s, 3H), 2.23 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.1, 160.9, 144.0, 133.9, 128.3, 113.5, 110.4, 57.7, 55.4, 21.5, 15.3. IR (film): 3073, 2967, 2934, 2910, 2837, 1632, 1605, 1507, 1307, 1251, 1173, 1088, 1032, 892, 833, 805, 571 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{17}NO$ M^+: 203.1310; found: 203.1304.



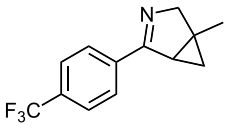
(E)-2-methyl-*N*-(1-(pyridin-3-yl)ethylidene)prop-2-en-1-amine (1m): colourless liquid, 0.339 g, isolated yield = 78%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.03 (d, *J* = 2.0 Hz, 1H), 8.61 (dd, *J* = 4.8, 1.2 Hz, 1H), 8.15 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.40 – 7.24 (m, 1H), 4.95 (s, 1H), 4.89 (s, 1H), 4.07 (s, 2H), 2.26 (s, 3H), 1.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.6, 150.6, 148.3, 143.4, 136.3, 134.1, 123.2, 110.8, 57.9, 21.4, 15.4. IR (film): 3076, 2969, 2911, 2855, 1636, 1586, 1415, 1371, 1287, 1021, 894, 807, 708 cm^{-1}. HRMS (ESI) calcd for $C_{11}H_{14}N_2$ M^+: 174.1157; found: 174.1156.



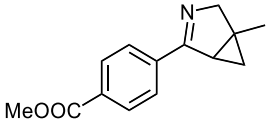
(E)-2-methyl-*N*-(1-(thiophen-2-yl)ethylidene)prop-2-en-1-amine (1n): colourless liquid, 0.304 g, isolated yield = 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.31 (m, 2H), 7.12 – 7.00 (m, 1H), 4.96 (s, 1H), 4.90 (s, 1H), 4.07 (s, 2H), 2.25 (s, 3H), 1.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 148.0, 143.4, 128.8, 127.3, 126.8, 110.5, 57.2, 21.5, 15.4. IR (film): 3076, 2970, 2914, 2855, 1665, 1624, 1431, 1369, 1282, 1234, 1063, 895, 851, 710, 596 cm^{-1}. HRMS (ESI) calcd for $C_{10}H_{13}NS$ M^+: 179.0769; found: 179.0764.



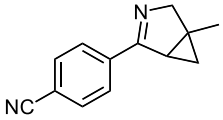
1-Methyl-4-phenyl-3-azabicyclo[3.1.0]hex-3-ene (2a): light yellow liquid, 30.8 mg, isolated yield = 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.85 (m, 2H), 7.52 – 7.35 (m, 3H), 4.09 (dd, *J* = 17.6, 2.4 Hz, 1H), 3.89 (d, *J* = 17.6 Hz, 1H), 2.35 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.43 (s, 3H), 1.11 (dd, *J* = 8.2, 3.8 Hz, 1H), 0.45 (t, *J* = 3.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.9, 134.5, 130.6, 128.6, 128.0, 68.8, 33.4, 27.4, 21.5, 19.1. IR (film): 3061, 2956, 2920, 2867, 2837, 1772, 1599, 1559, 1448, 1372, 1343, 1182, 1051, 1028, 776, 694, 659 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{14}N$ $[M+H]^+$: 172.1126; found: 172.1122.



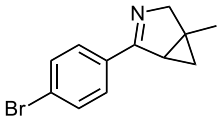
1-Methyl-4-(4-(trifluoromethyl)phenyl)-3-azabicyclo[3.1.0]hex-3-ene (2b): light yellow liquid, 32.3 mg, isolated yield = 54%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 4.13 (dd, *J* = 17.8, 2.6 Hz, 1H), 3.92 (d, *J* = 18.0 Hz, 1H), 2.35 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.44 (s, 3H), 1.15 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.46 (t, *J* = 3.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 137.6, 132.2 (q, *J* = 32.4 Hz), 128.3, 125.6 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 273.2 Hz), 69.0, 33.3, 27.7, 21.5, 19.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.76. IR (film): 3061, 2958, 2926, 2867, 2843, 1727, 1602, 1412, 1324, 1167, 1128, 1069, 1016, 850, 690, 594 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{13}F_3N$ $[M+H]^+$: 240.1000; found: 240.10023.



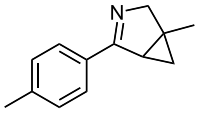
Methyl 4-(5-methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzoate (2c): white solid, 25.2 mg, isolated yield = 44%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.4 Hz, 2H), 7.96 (d, *J* = 8.4 Hz, 2H), 4.12 (dd, *J* = 17.8, 2.6 Hz, 1H), 3.94 (s, 3H), 3.91 (d, *J* = 18.0 Hz, 1H), 2.36 (dd, *J* = 5.6, 2.8 Hz, 1H), 1.43 (s, 3H), 1.13 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.46 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.2, 166.9, 138.4, 131.8, 129.9, 127.94, 69.1, 52.5, 33.4, 27.7, 21.5, 19.1. IR (film): 3061, 2952, 2929, 2867, 2840, 1723, 1598, 1434, 1409, 1277, 1110, 1018, 867, 779, 710 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{16}NO_2$ $[M+H]^+$: 230.1181; found: 230.1180.



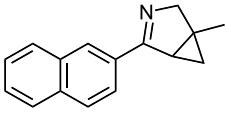
4-(5-Methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzonitrile (2d): white solid, 21.6 mg, isolated yield = 44%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 4.14 (dd, *J* = 18.0, 2.8 Hz, 1H), 3.93 (d, *J* = 18.0 Hz, 1H), 2.34 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.45 (s, 3H), 1.16 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.47 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.5, 138.4, 132.5, 128.5, 118.8, 114.0, 69.2, 33.2, 27.9, 21.5, 19.1. IR (film): 3061, 2952, 2928, 2870, 2228, 1725, 1596, 1456, 1286, 1123, 1074, 848, 747, 550 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{13}N_2$ $[M+H]^+$: 197.1079; found: 197.1082.



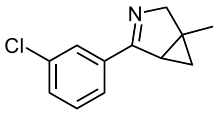
4-(4-Bromophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2f): colourless liquid, 40.5 mg, isolated yield = 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.70 (m, 2H), 7.66 – 7.47 (m, 2H), 4.07 (dd, *J* = 17.6, 2.8 Hz, 1H), 3.86 (d, *J* = 17.6 Hz, 1H), 2.30 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.42 (s, 3H), 1.11 (dd, *J* = 8.2, 3.8 Hz, 1H), 0.44 (t, *J* = 3.4 Hz, 1H). IR (film): 3061, 2952, 2926, 2867, 2840, 1725, 1590, 1487, 1399, 1340, 1177, 1072, 1007, 835, 719, 657, 494, 450 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{13}BrN$ $[M+H]^+$: 250.0231; found: 250.0236.



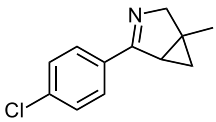
1-Methyl-4-(p-tolyl)-3-azabicyclo[3.1.0]hex-3-ene (2g): light yellow liquid, 35.2 mg, isolated yield = 76%.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 7.6 Hz, 2H), 4.08 (dd, *J* = 17.6, 2.4 Hz, 1H), 3.87 (d, *J* = 17.2 Hz, 1H), 2.41 (s, 3H), 2.34 (dt, *J* = 8.4, 2.8 Hz, 1H), 1.42 (s, 3H), 1.10 (dd, *J* = 8.2, 3.8 Hz, 1H), 0.44 (t, *J* = 3.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 140.8, 131.8, 129.3, 128.0, 68.6, 33.3, 27.3, 21.7, 21.4, 19.1. IR (film): 3058, 3031, 2952, 2920, 2867, 2840, 1728, 1614, 1597, 1343, 1286, 1178, 1058, 826, 717, 666, 496 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{16}N$ $[M+H]^+$: 186.1283; found: 186.1280.



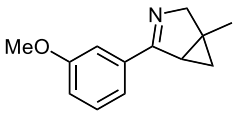
1-Methyl-4-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2h): white solid, 35.9 mg, isolated yield = 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 8.12 (dd, *J* = 8.8, 1.6 Hz, 1H), 8.00 – 7.80 (m, 3H), 7.60 – 7.50 (m, 2H), 4.17 (dd, *J* = 17.6, 2.8 Hz, 1H), 3.96 (d, *J* = 17.6 Hz, 1H), 2.49 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.46 (s, 3H), 1.17 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.52 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 134.6, 133.2, 131.9, 128.9, 128.7, 128.3, 127.9, 127.2, 126.5, 124.6, 68.8, 33.3, 27.4, 21.5, 19.1. IR (film): 3058, 2952, 2923, 2867, 2840, 1728, 1603, 1588, 1569, 1272, 1178, 1125, 1051, 951, 863, 823, 758, 743, 477 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}N$ $[M+H]^+$: 222.1283; found: 222.1285.



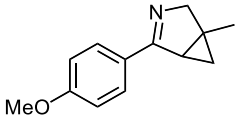
4-(3-Chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2i): colourless liquid, 35.4 mg, isolated yield = 69%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (t, *J* = 1.8 Hz, 1H), 7.79 (dt, *J* = 7.6, 1.4 Hz, 1H), 7.45 – 7.33 (m, 2H), 4.10 (dd, *J* = 17.6, 2.4 Hz, 1H), 3.89 (d, *J* = 17.6 Hz, 1H), 2.32 (dt, *J* = 8.0, 2.8 Hz, 1H), 1.43 (s, 3H), 1.13 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.45 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 136.2, 134.7, 130.6, 130.0, 128.2, 126.1, 68.9, 33.3, 27.6, 21.5, 19.1. IR (film): 3064, 2952, 2926, 2870, 2840, 1728, 1595, 1564, 1428, 1334, 1076, 979, 942, 791, 739, 696 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{12}ClN$ M^+: 205.0658; found: 205.0659.



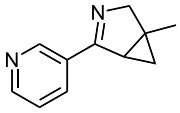
4-(4-Chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2j): colourless liquid, 35.9 mg, isolated yield = 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 4.07 (dd, *J* = 18.2, 2.6 Hz, 1H), 3.86 (d, *J* = 18.0 Hz, 1H), 2.29 (dt, *J* = 8.4, 2.8 Hz, 1H), 1.41 (s, 3H), 1.10 (dd, *J* = 8.2, 3.8 Hz, 1H), 0.43 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 136.6, 132.9, 129.3, 128.8, 68.8, 33.2, 27.6, 21.5, 19.1. IR (film): 3061, 2952, 2923, 2867, 2843, 1725, 1597, 1493, 1404, 1340, 1088, 1057, 1011, 839, 794, 730, 658, 497 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{13}ClN$ $[M+H]^+$: 206.0737; found: 206.0738.



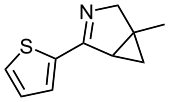
4-(3-Methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2k): colourless liquid, 30.2 mg, isolated yield = 60%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.44 (m, 2H), 7.34 (t, *J* = 8.0 Hz, 1H), 7.00 (dd, *J* = 8.0, 1.4 Hz, 1H), 4.09 (dd, *J* = 17.6, 2.2 Hz, 1H), 3.88 (d, *J* = 18.0 Hz, 1H), 3.86 (s, 3H), 2.34 (dt, *J* = 8.0, 3.0 Hz, 1H), 1.42 (s, 3H), 1.10 (dd, *J* = 7.8, 4.0 Hz, 1H), 0.44 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 159.8, 135.8, 129.6, 120.9, 117.4, 111.8, 68.7, 55.5, 33.4, 27.3, 21.5, 19.1. IR (film): 3061, 2955, 2920, 2870, 2837, 1728, 1602, 1575, 1456, 1337, 1286, 1259, 1042, 980, 876, 838, 792, 701 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{15}NO$ M^+: 201.1154; found: 201.1151 .



4-(4-Methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2l): colourless liquid, 37.7 mg, isolated yield = 75%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 4.05 (dd, *J* = 17.6, 2.4 Hz, 1H), 3.86 (s, 3H), 3.85 (d, *J* = 17.2 Hz, 2H), 2.31 (dt, *J* = 8.4, 2.8 Hz, 1H), 1.41 (s, 3H), 1.09 (dd, *J* = 8.2, 3.8 Hz, 1H), 0.43 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.2, 161.6, 129.6, 127.3, 113.9, 68.5, 55.5, 33.3, 27.3, 21.5, 19.1. IR (film): 3061, 2958, 2926, 2867, 2837, 1728, 1609, 1515, 1420, 1344, 1252, 1170, 1033, 838, 666, 574, 522 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{16}NO$ $[M+H]^+$: 202.1232; found: 202.1230.



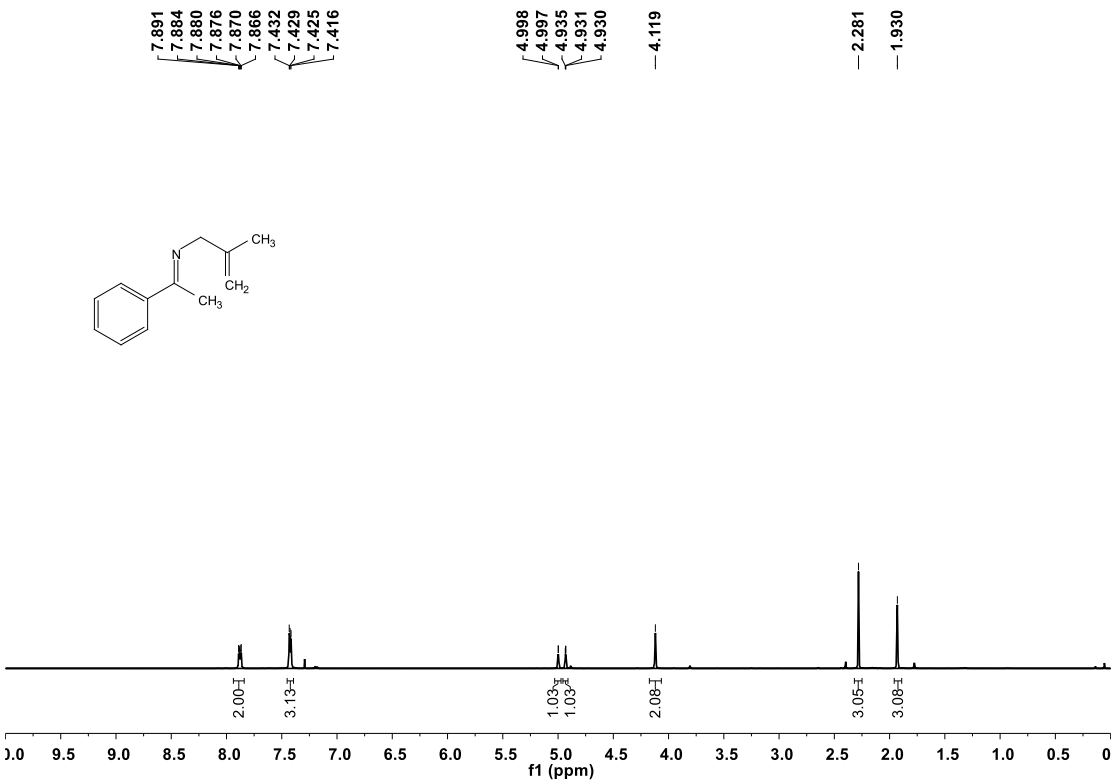
1-Methyl-4-(pyridin-3-yl)-3-azabicyclo[3.1.0]hex-3-ene (2m): colourless liquid, 27.1 mg, isolated yield = 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.08 (d, *J* = 1.6 Hz, 1H), 8.66 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.20 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.35 (ddd, *J* = 7.6, 4.8, 0.8 Hz, 1H), 4.09 (dd, *J* = 18.0, 2.6 Hz, 1H), 3.88 (d, *J* = 18.0 Hz, 1H), 2.35 (dt, *J* = 8.4, 2.8 Hz, 1H), 1.43 (s, 3H), 1.15 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.45 (t, *J* = 3.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.7, 151.5, 149.5, 134.9, 130.1, 123.7, 68.9, 33.0, 27.7, 21.5, 19.0. IR (film): 3034, 2958, 2926, 2870, 1725, 1598, 1457, 1411, 1344, 1285, 1123, 1073, 1025, 816, 796, 747, 708, 621 cm^{-1}. HRMS (ESI) calcd for $C_{11}H_{13}N_2$ $[M+H]^+$: 173.1079; found: 173.1079.



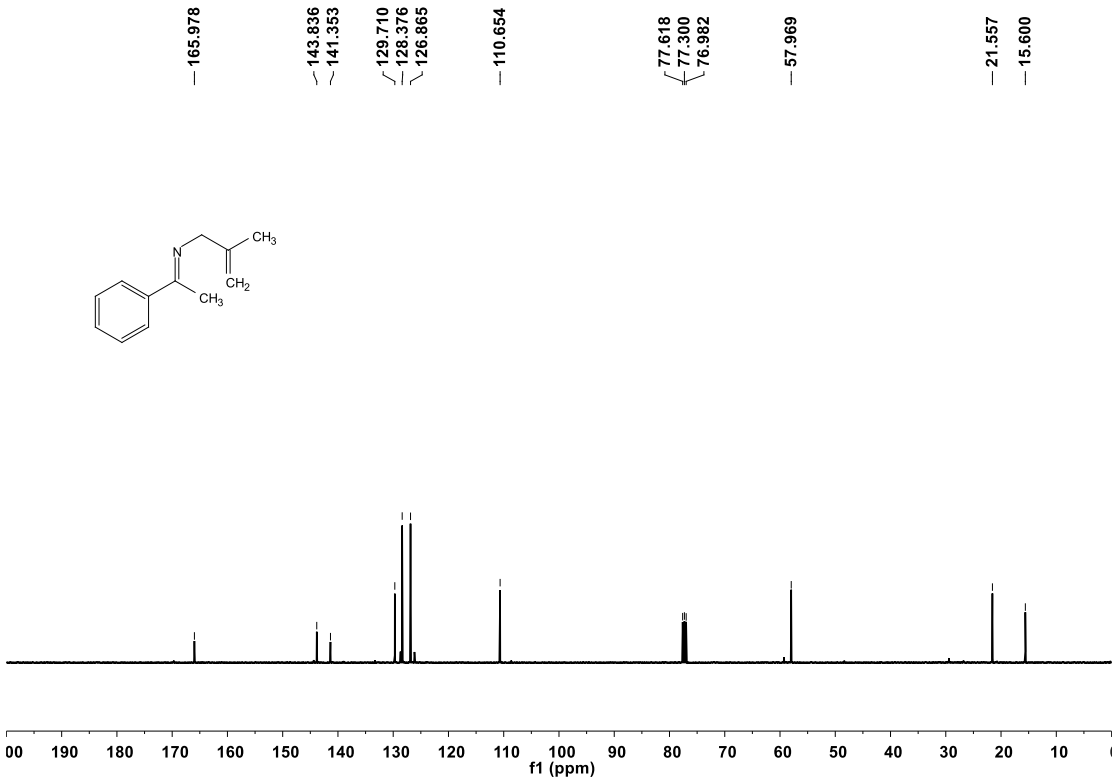
1-Methyl-4-(thiophen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2n): colourless liquid, 20.4 mg, isolated yield = 46%.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 3.6 Hz, 1H), 7.43 (d, *J* = 4.8 Hz, 1H), 7.10 (dd, *J* = 4.8, 3.6 Hz, 1H), 4.03 (dd, *J* = 17.6, 2.4 Hz, 1H), 3.86 (d, *J* = 17.6 Hz, 1H), 2.35 (dt, *J* = 8.4, 2.8 Hz, 1H), 1.42 (s, 3H), 1.09 (dd, *J* = 8.4, 4.0 Hz, 1H), 0.50 (t, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.2, 139.5, 129.7, 129.4, 127.7, 68.5, 33.8, 28.1, 21.4, 19.1. IR (film): 3067, 2958, 2923, 2867, 2840, 1728, 1592, 1436, 1336, 1283, 1121, 1059, 1030, 992, 847, 709 cm^{-1}. HRMS (ESI) calcd for $C_{10}H_{12}NS$ $[M+H]^+$: 178.0690; found:178.0688.

Reference

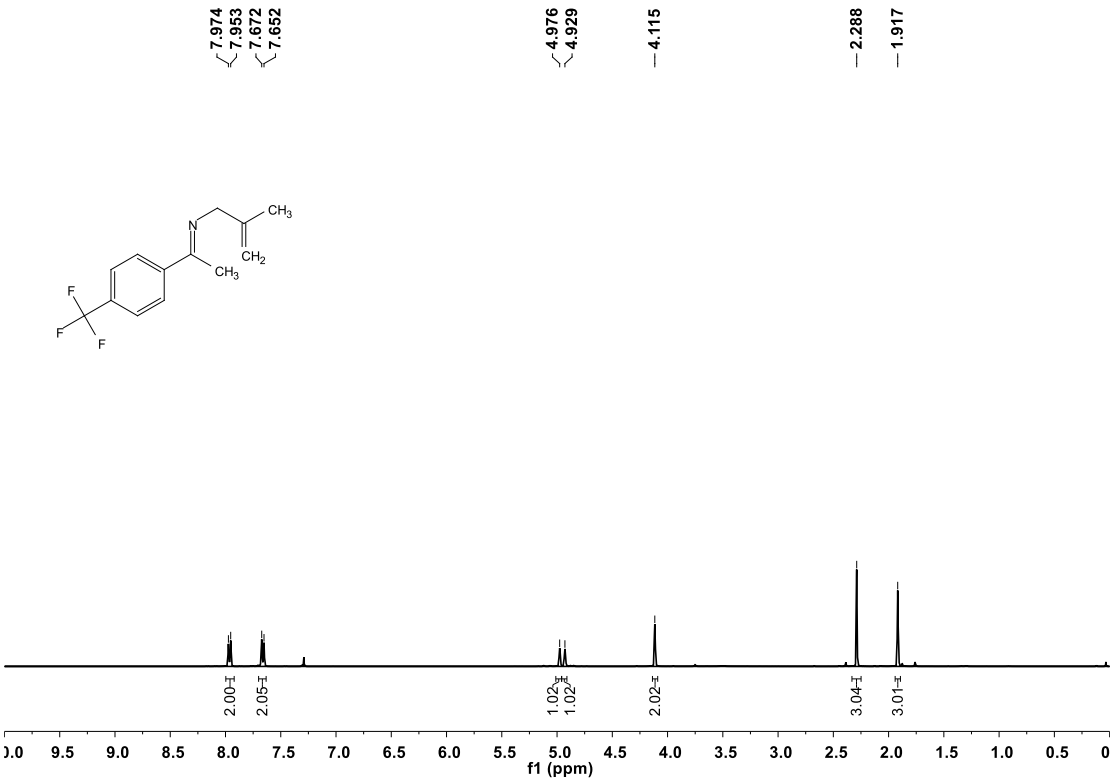
1. Z. Shi, M. Suri and F. Glorius, *Angew. Chem. Int. Ed.*, 2013, **52**, 4892-4896.



^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-phenylethylidene)prop-2-en-1-amine (1a)

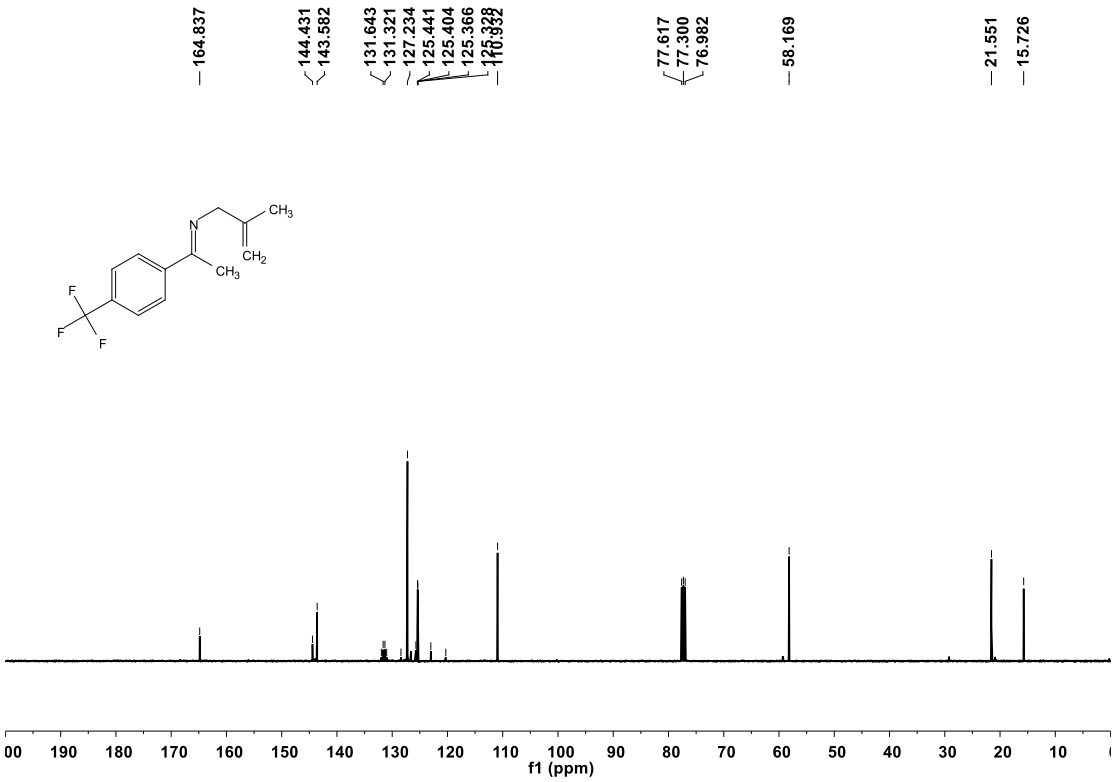


^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-phenylethylidene)prop-2-en-1-amine (1a)



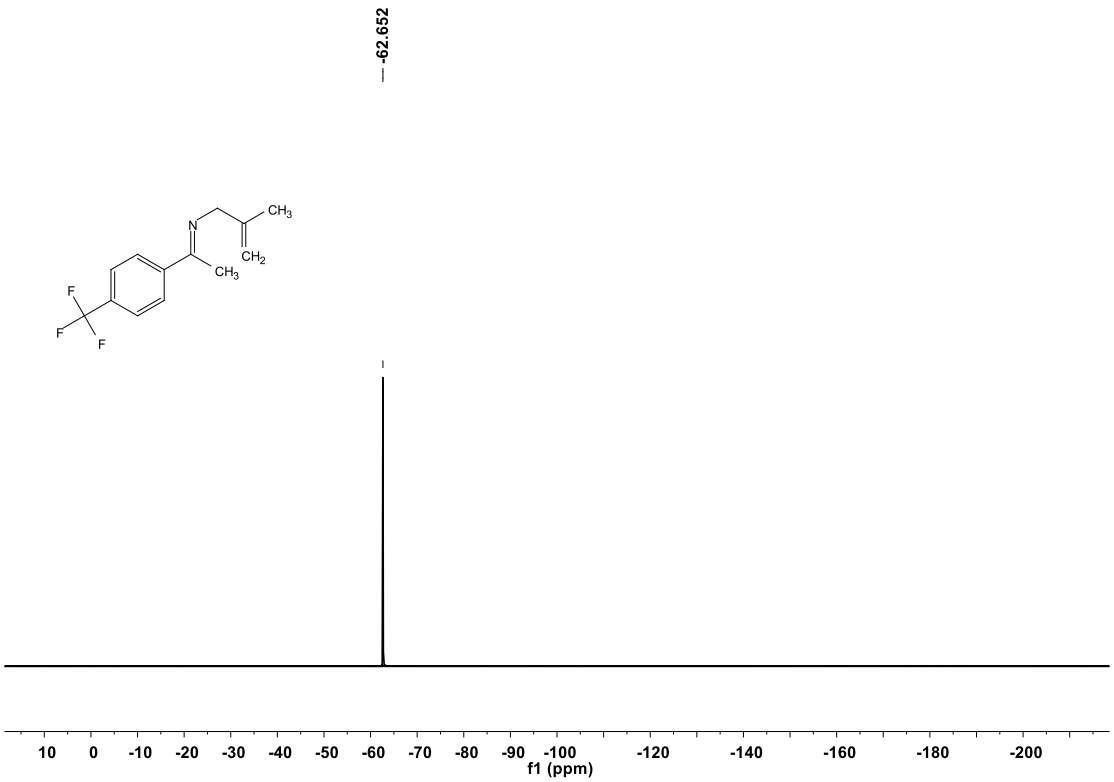
^{1}H NMR spectrum of

(E)-2-methyl-*N*-(1-(4-(trifluoromethyl)phenyl)ethylidene)prop-2-en-1-amine (1b)



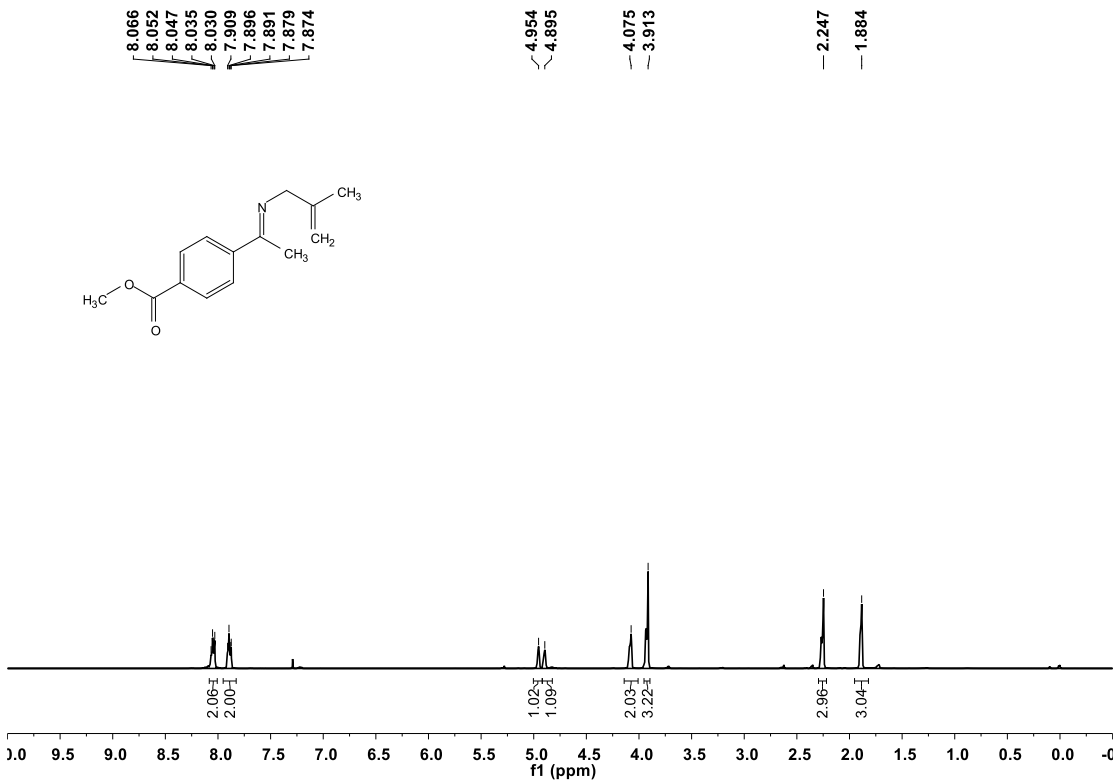
^{13}C NMR spectrum of

(E)-2-methyl-*N*-(1-(4-(trifluoromethyl)phenyl)ethylidene)prop-2-en-1-amine (1b)

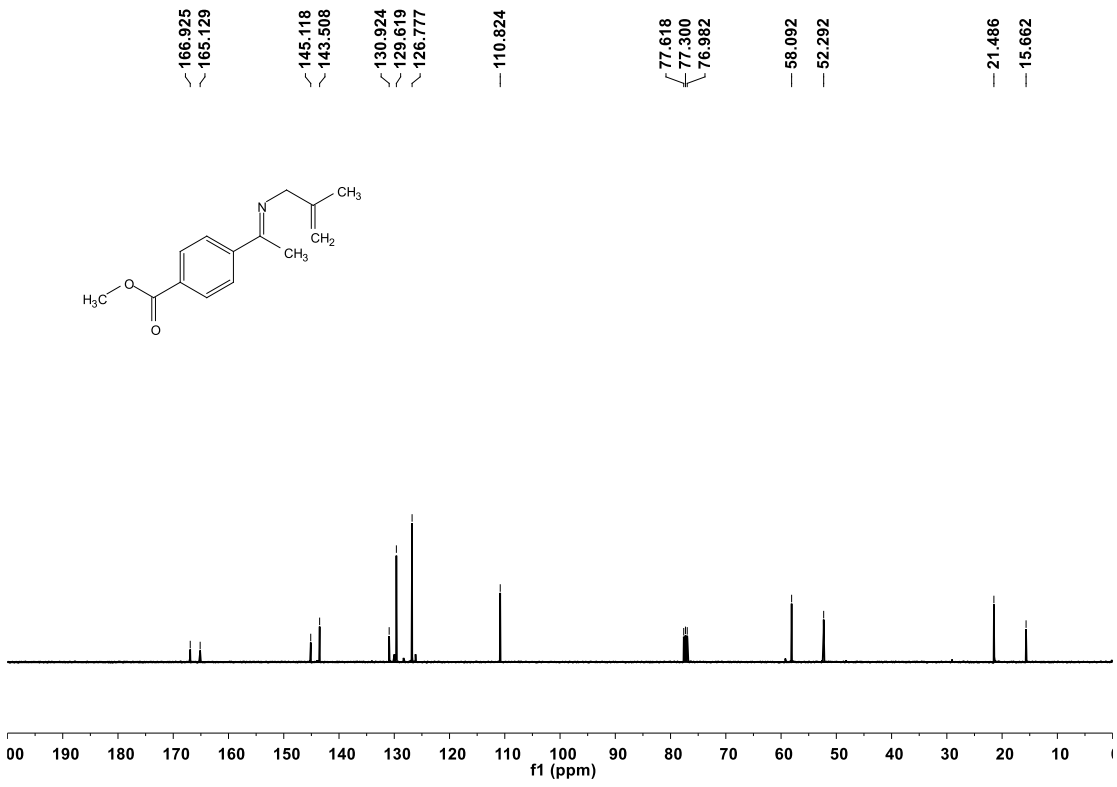


^{19}F NMR spectrum of

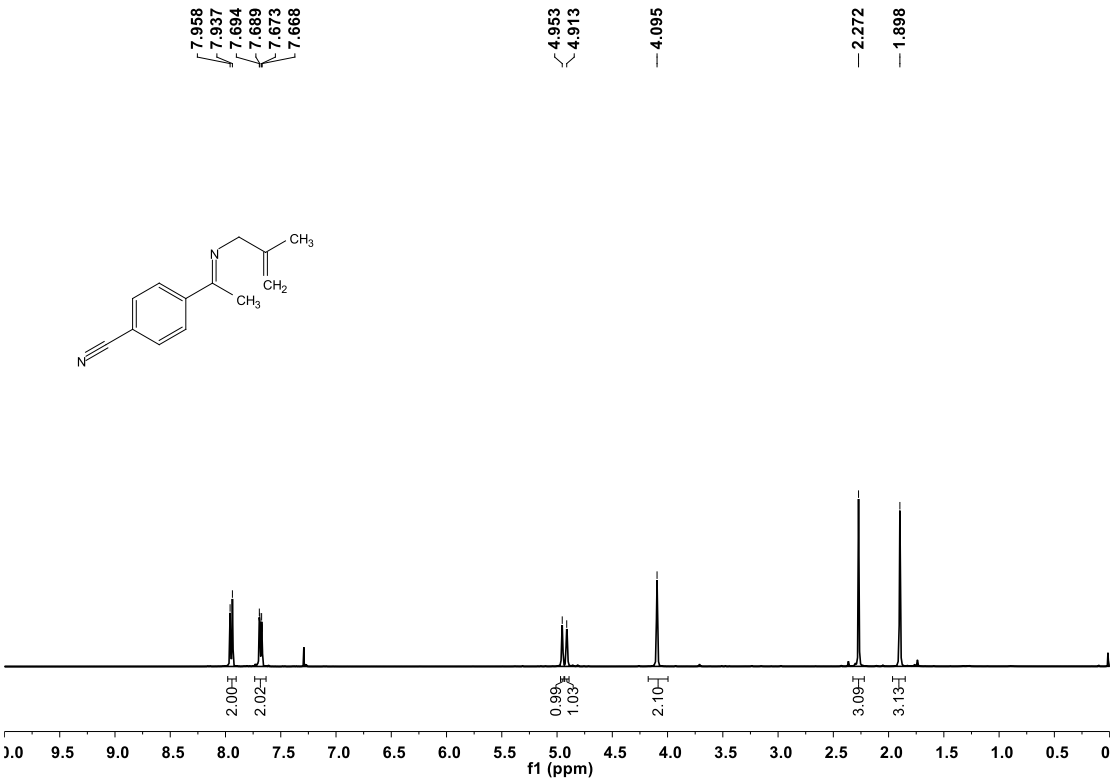
(E)-2-methyl-*N*-(1-(4-(trifluoromethyl)phenyl)ethylidene)prop-2-en-1-amine (1b)



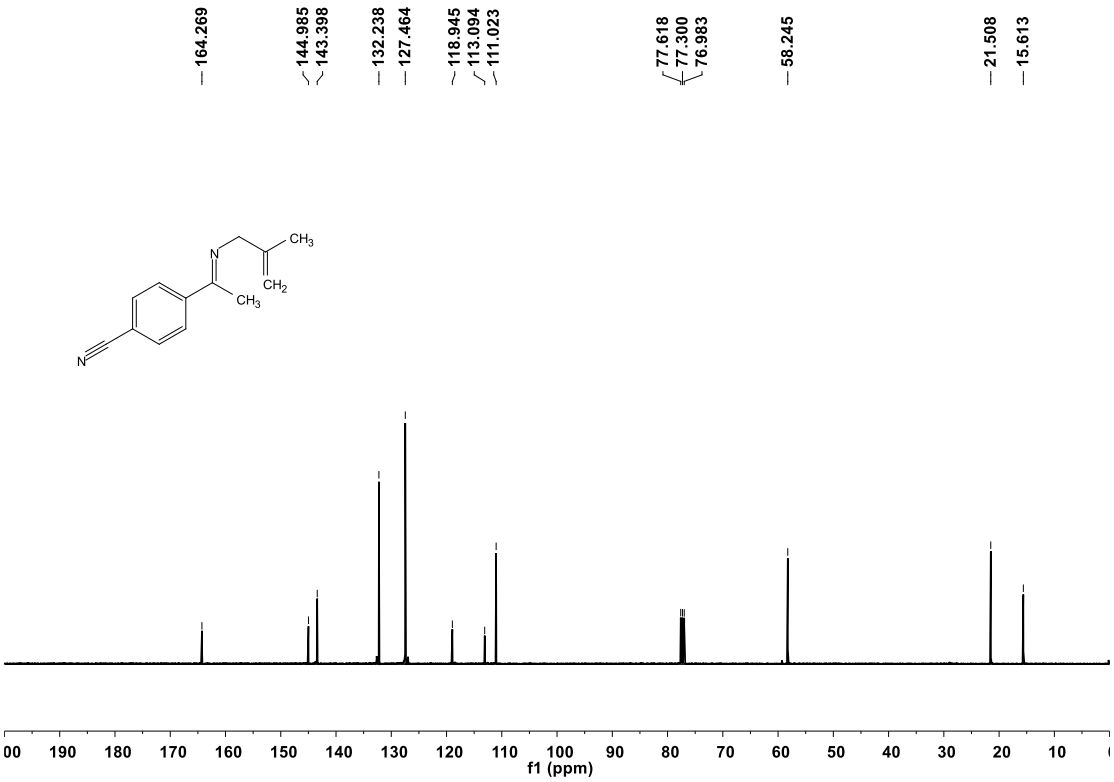
^{1}H NMR spectrum of (E)-methyl 4-(1-((2-methylallyl)imino)ethyl)benzoate (1c)



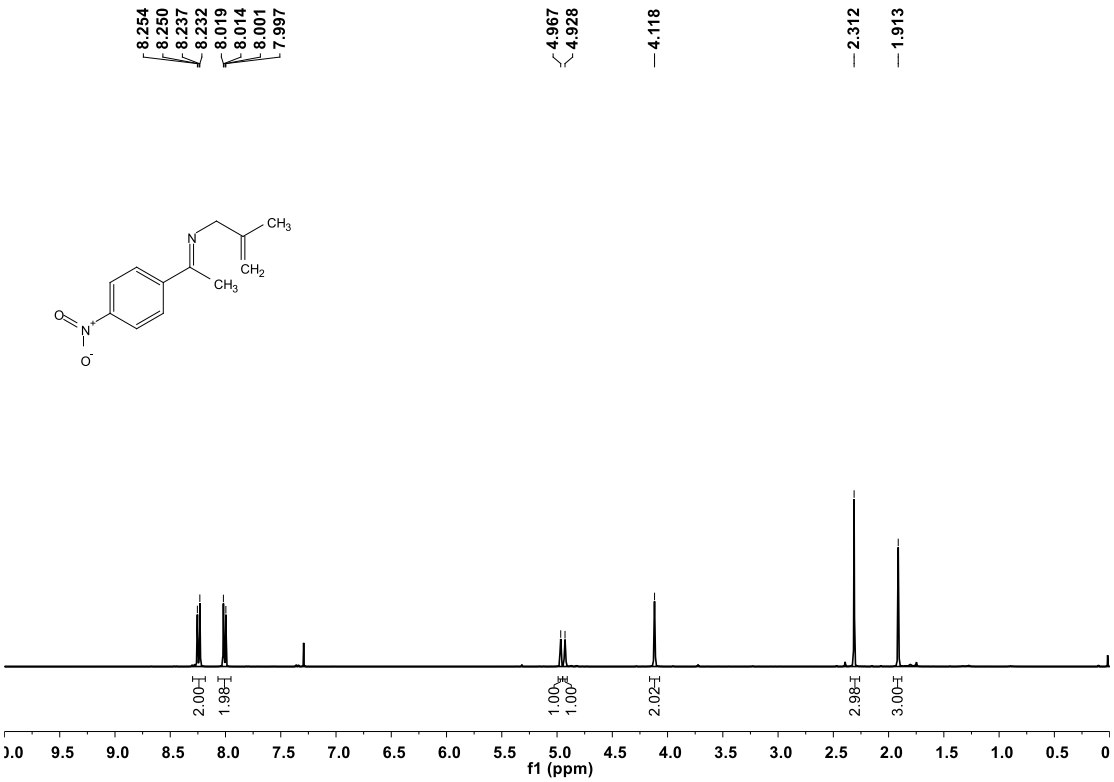
^{13}C NMR spectrum of (E)-methyl 4-(1-((2-methylallyl)imino)ethyl)benzoate (1c)



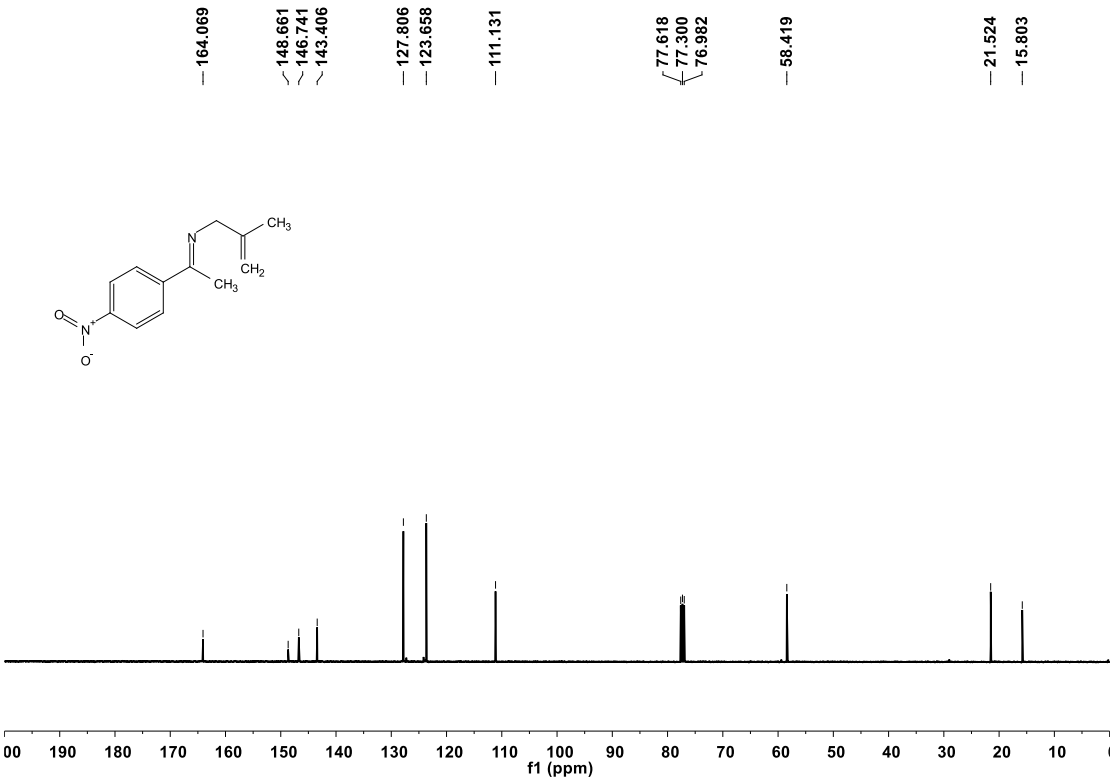
^{1}H NMR spectrum of (E)-4-(1-((2-methylallyl)imino)ethyl)benzonitrile (1d)



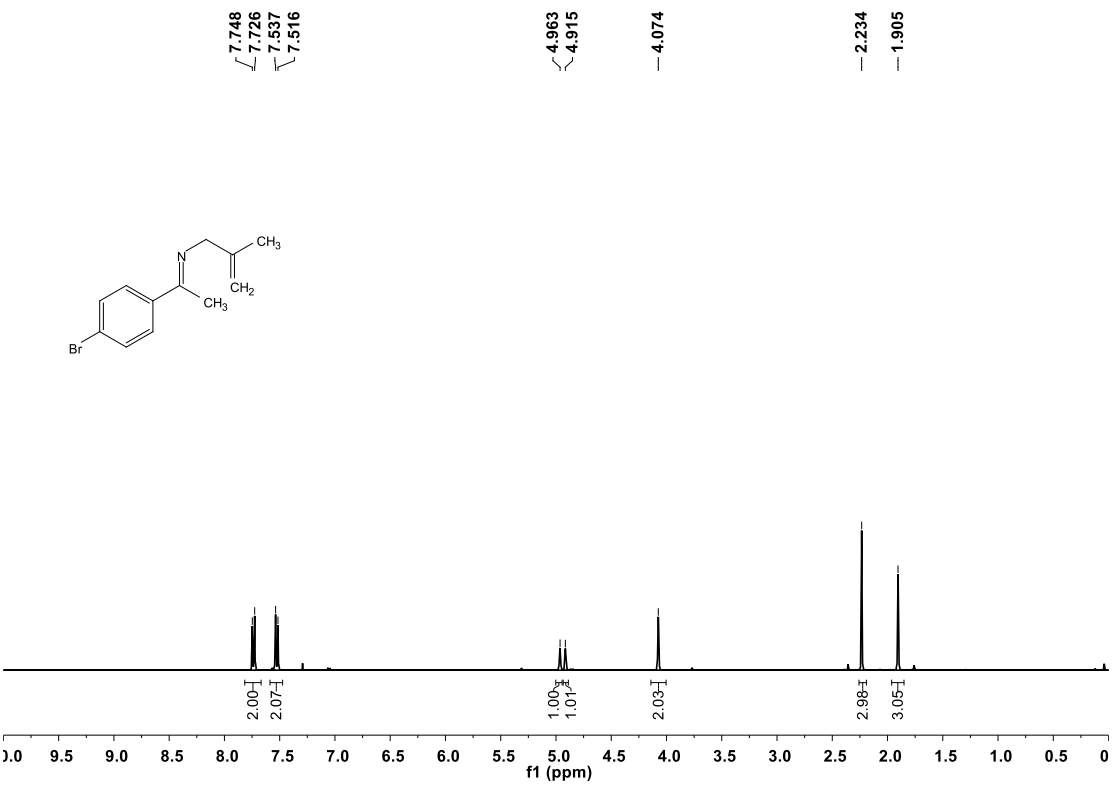
^{13}C NMR spectrum of (E)-4-(1-((2-methylallyl)imino)ethyl)benzonitrile (1d)



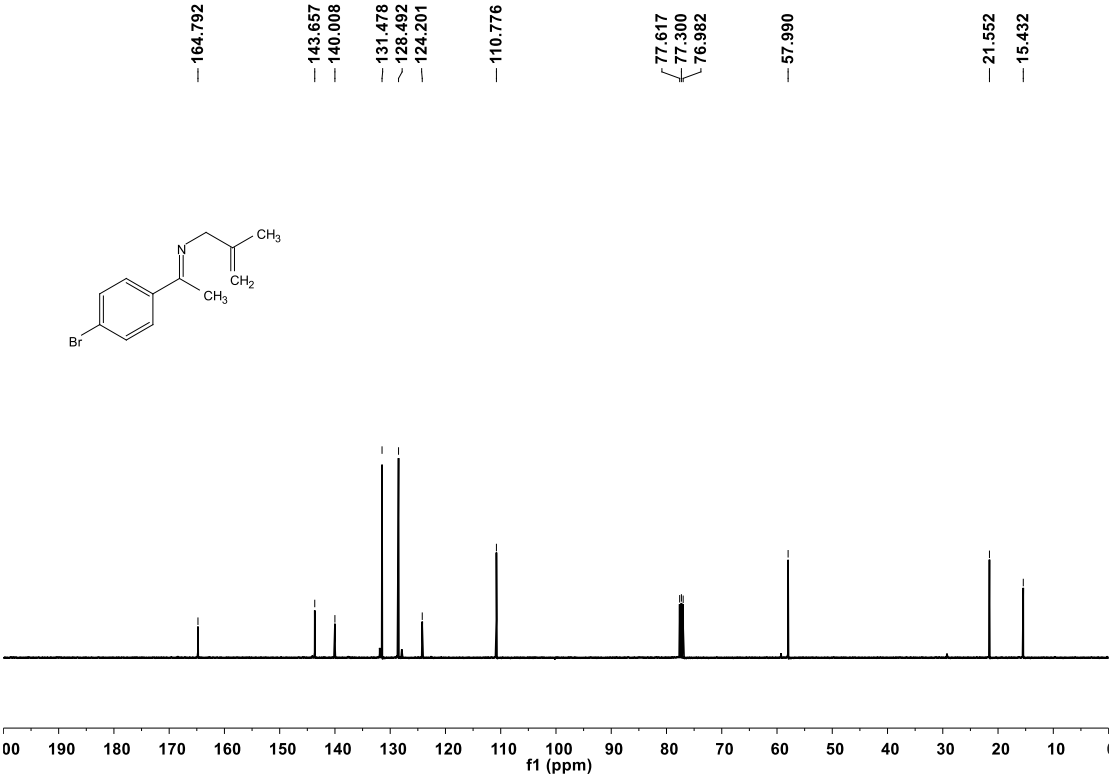
^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-(4-nitrophenyl)ethylidene)prop-2-en-1-amine (1e)



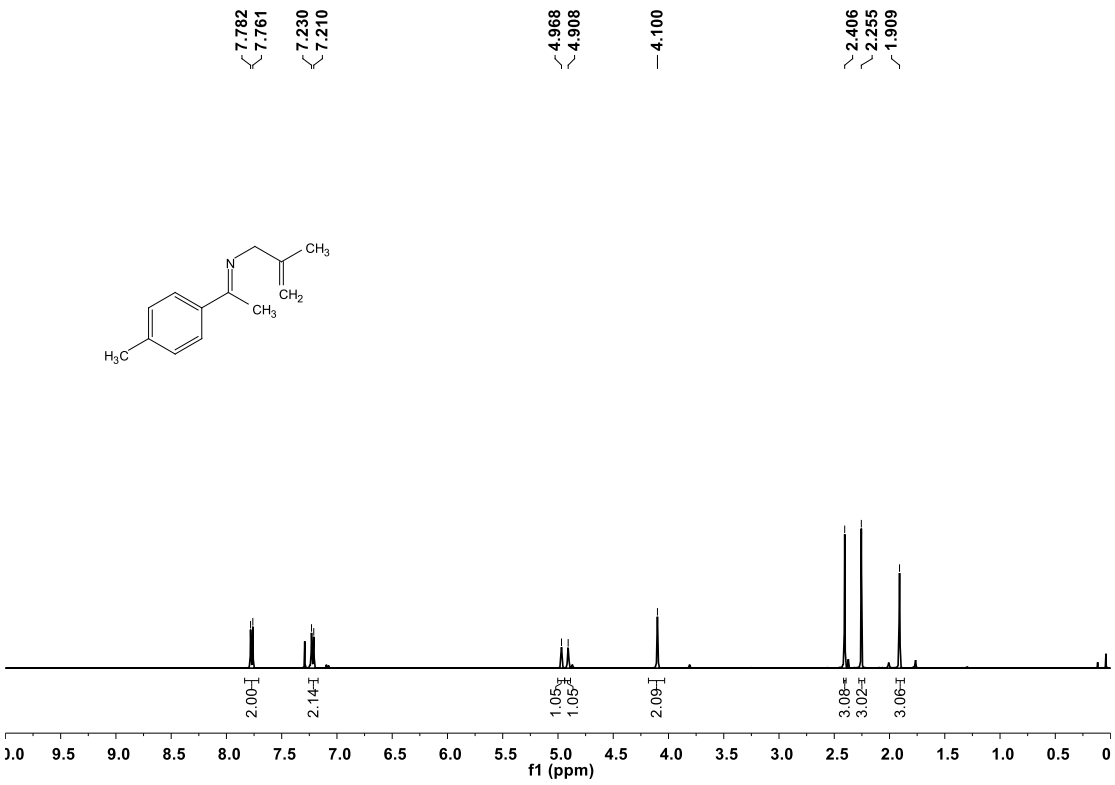
^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-(4-nitrophenyl)ethylidene)prop-2-en-1-amine (1e)



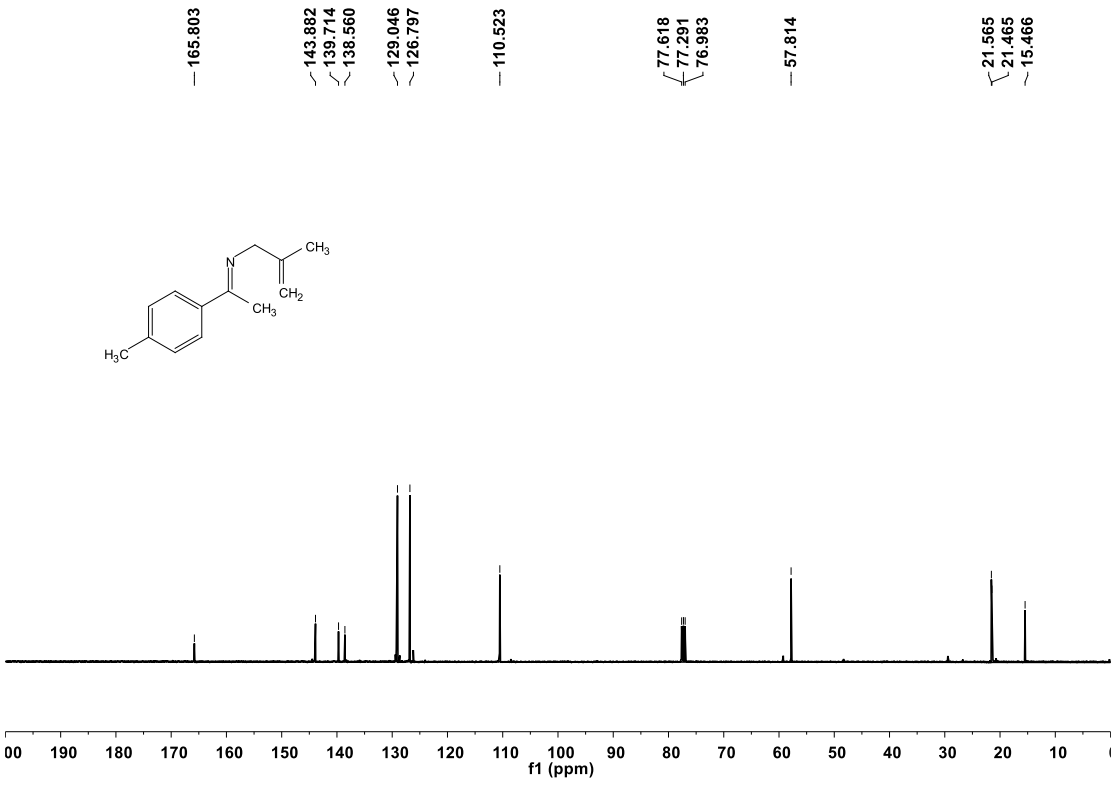
^{1}H NMR spectrum of (E)-*N*-(1-(4-bromophenyl)ethylidene)-2-methylprop-2-en-1-amine (1f)



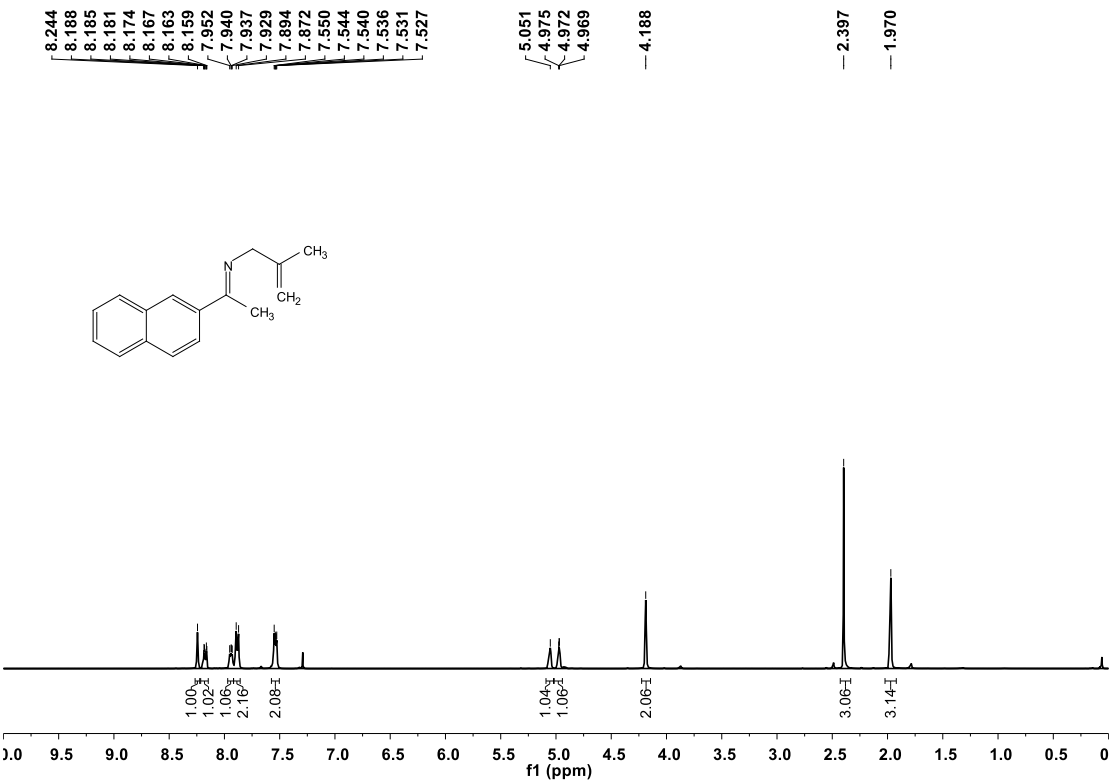
^{13}C NMR spectrum of (E)-*N*-(1-(4-bromophenyl)ethylidene)-2-methylprop-2-en-1-amine (1f)



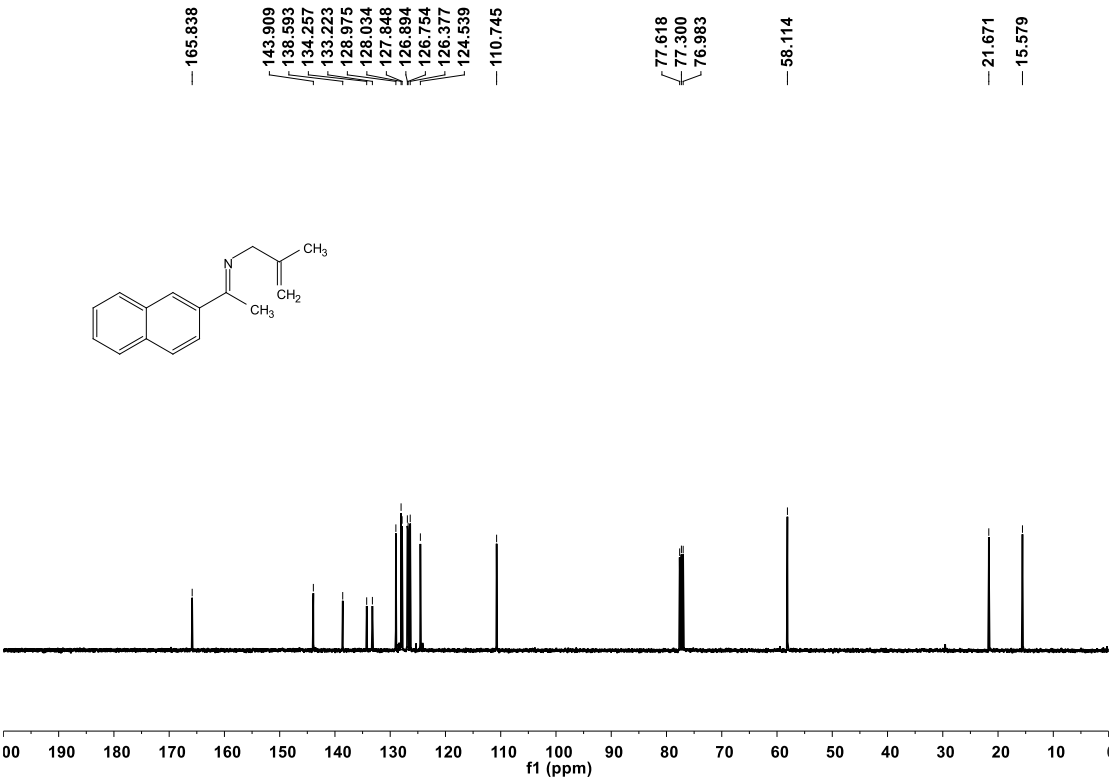
^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-(p-tolyl)ethylidene)prop-2-en-1-amine (1g)



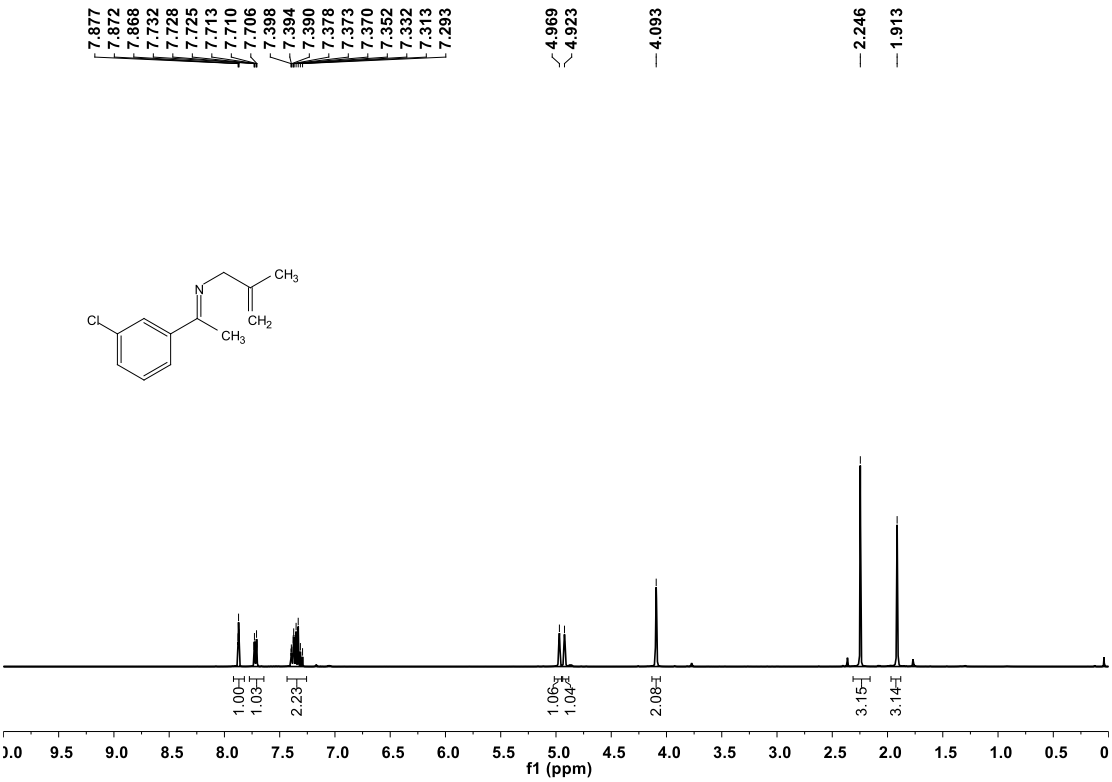
^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-(p-tolyl)ethylidene)prop-2-en-1-amine (1g)



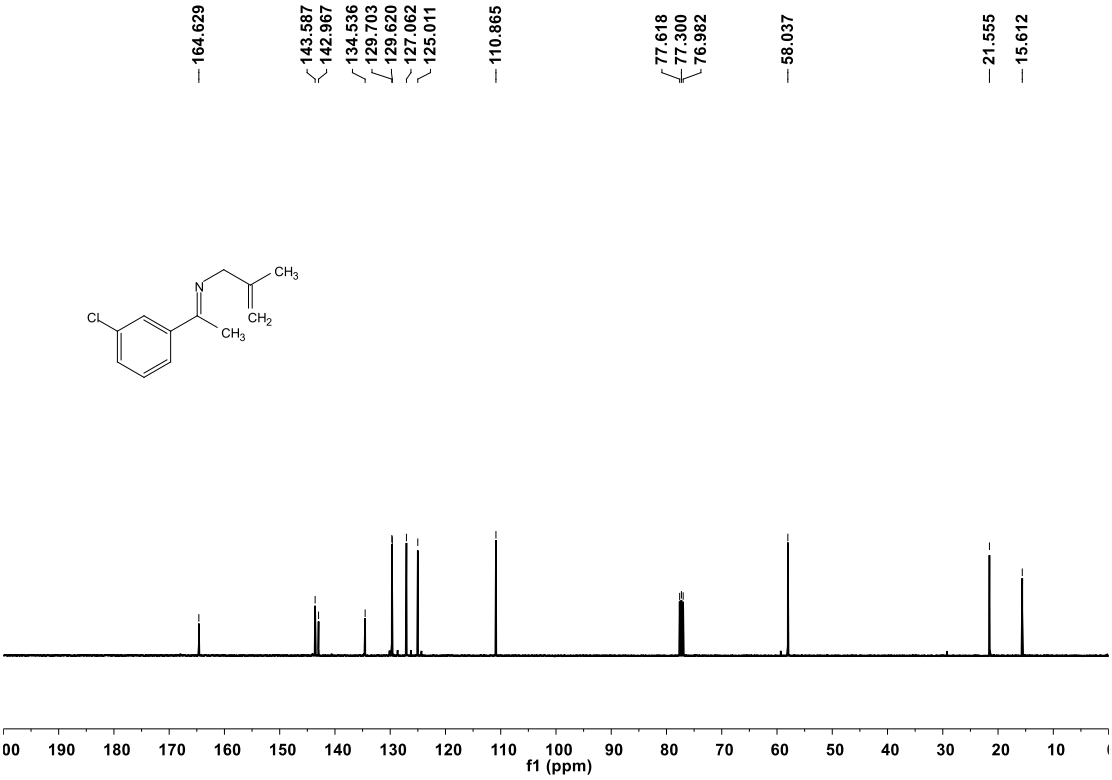
^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-(naphthalen-2-yl)ethylidene)prop-2-en-1-amine (1h)



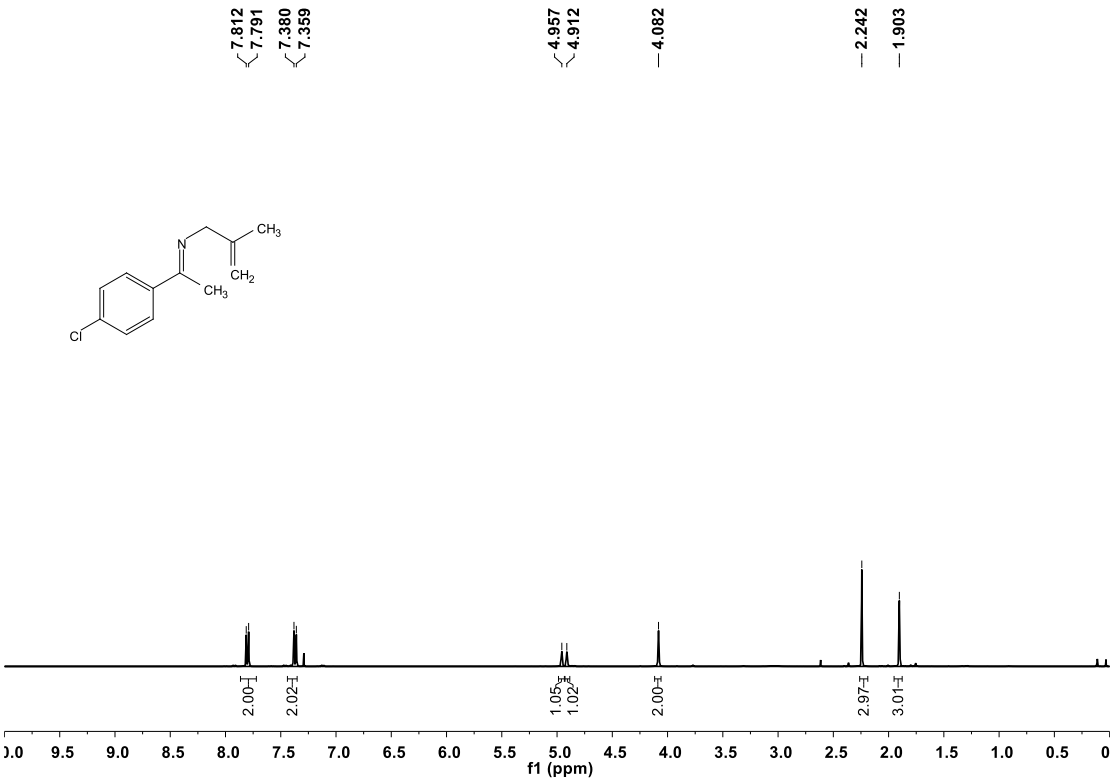
^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-(naphthalen-2-yl)ethylidene)prop-2-en-1-amine (1h)



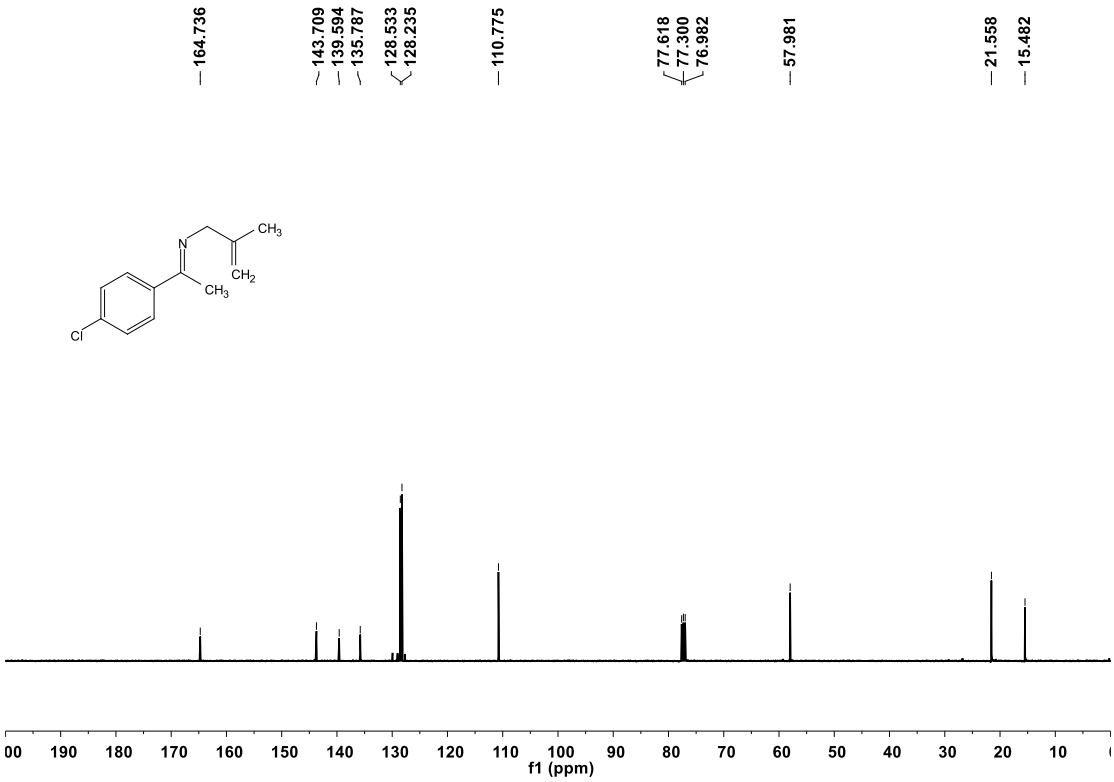
^{1}H NMR spectrum of (E)-*N*-(1-(3-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1i)



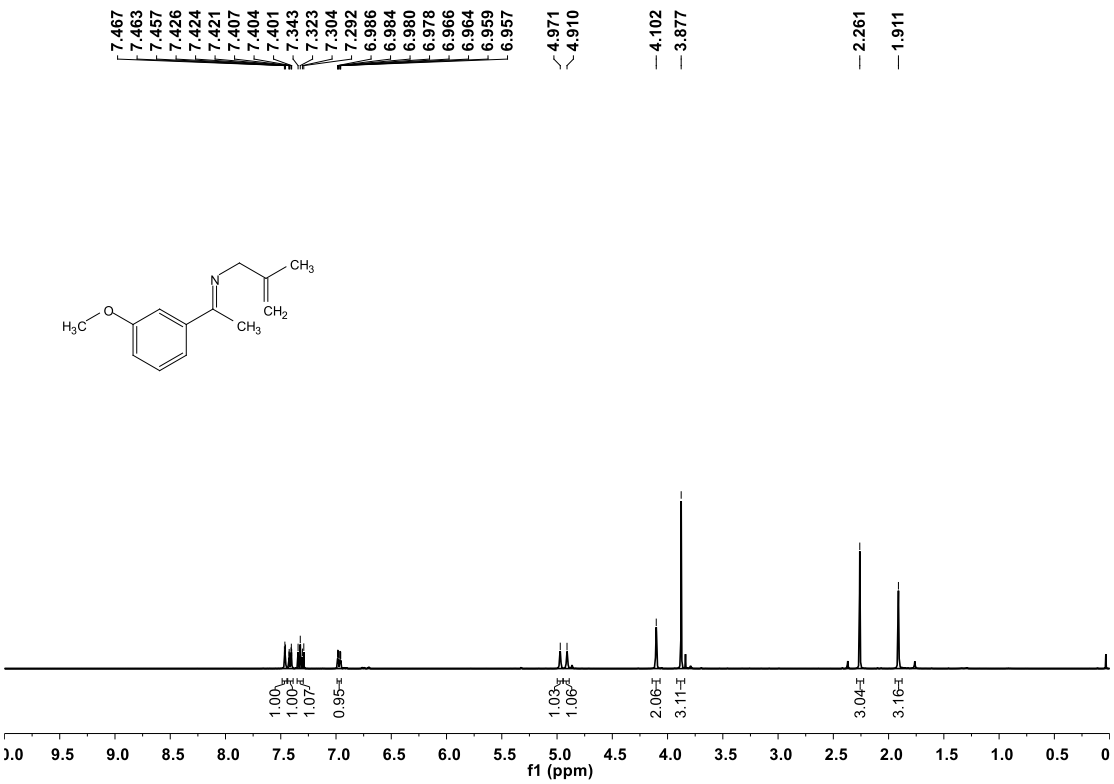
^{13}C NMR spectrum of (E)-*N*-(1-(3-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1i)



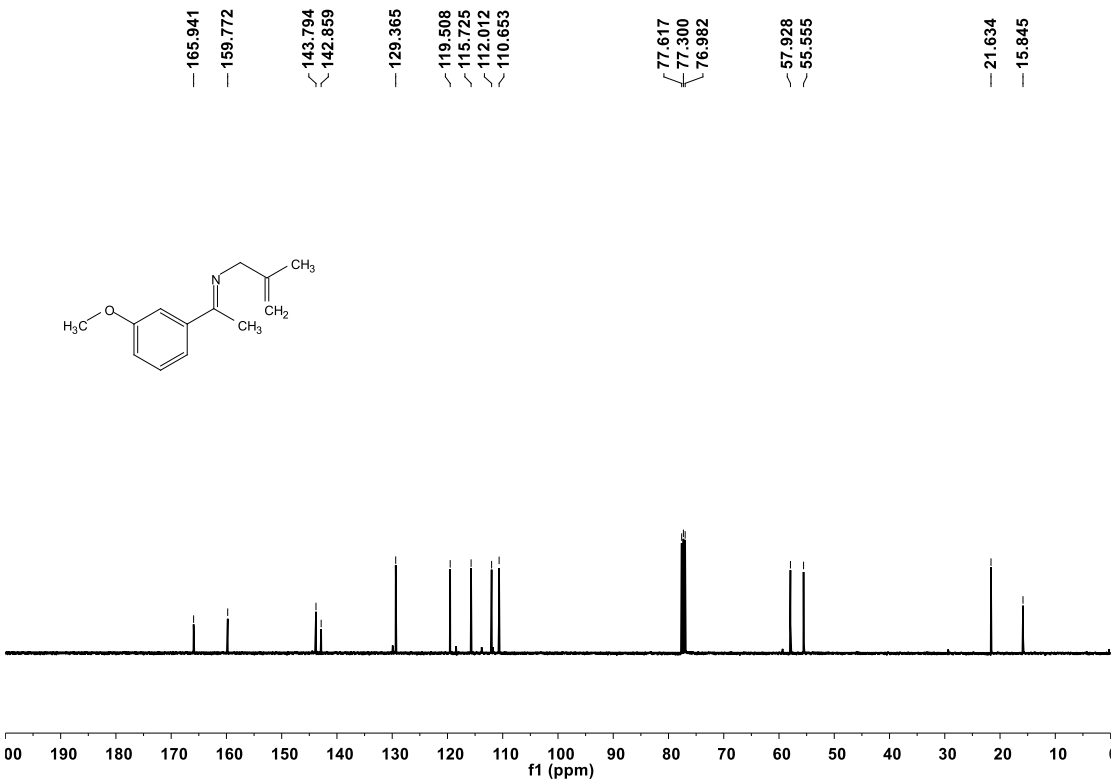
^{1}H NMR spectrum of (E)-*N*-(1-(4-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1j)



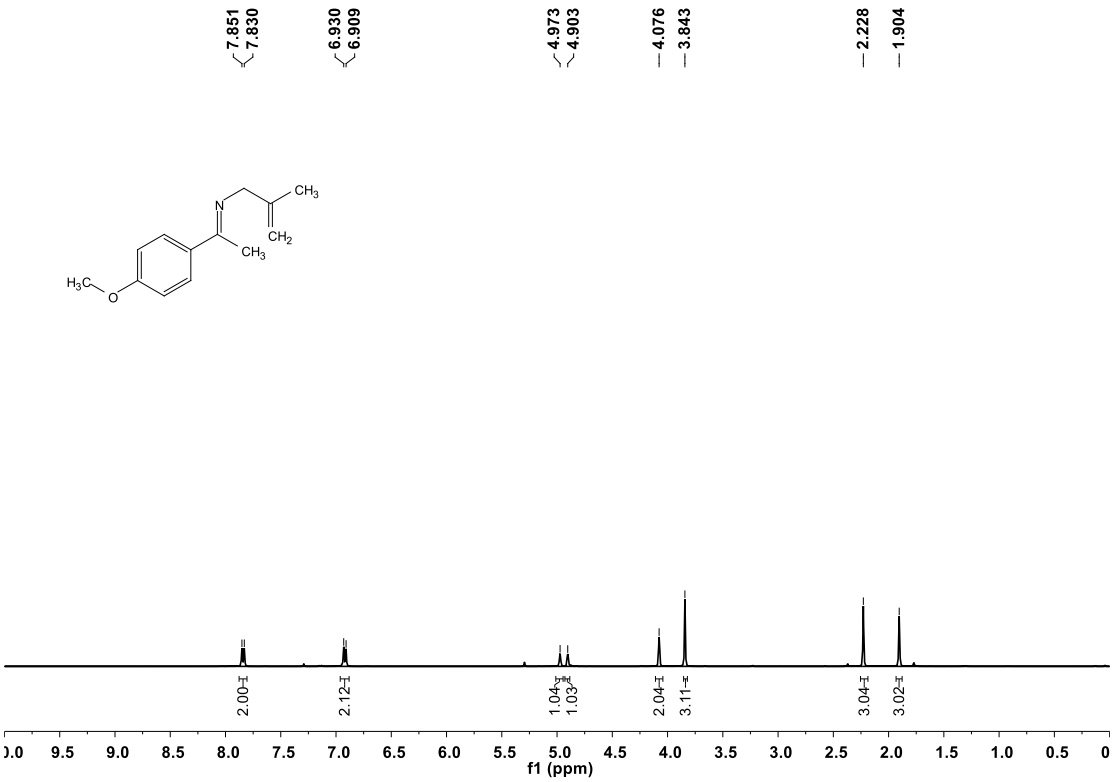
^{13}C NMR spectrum of (E)-*N*-(1-(4-chlorophenyl)ethylidene)-2-methylprop-2-en-1-amine (1j)



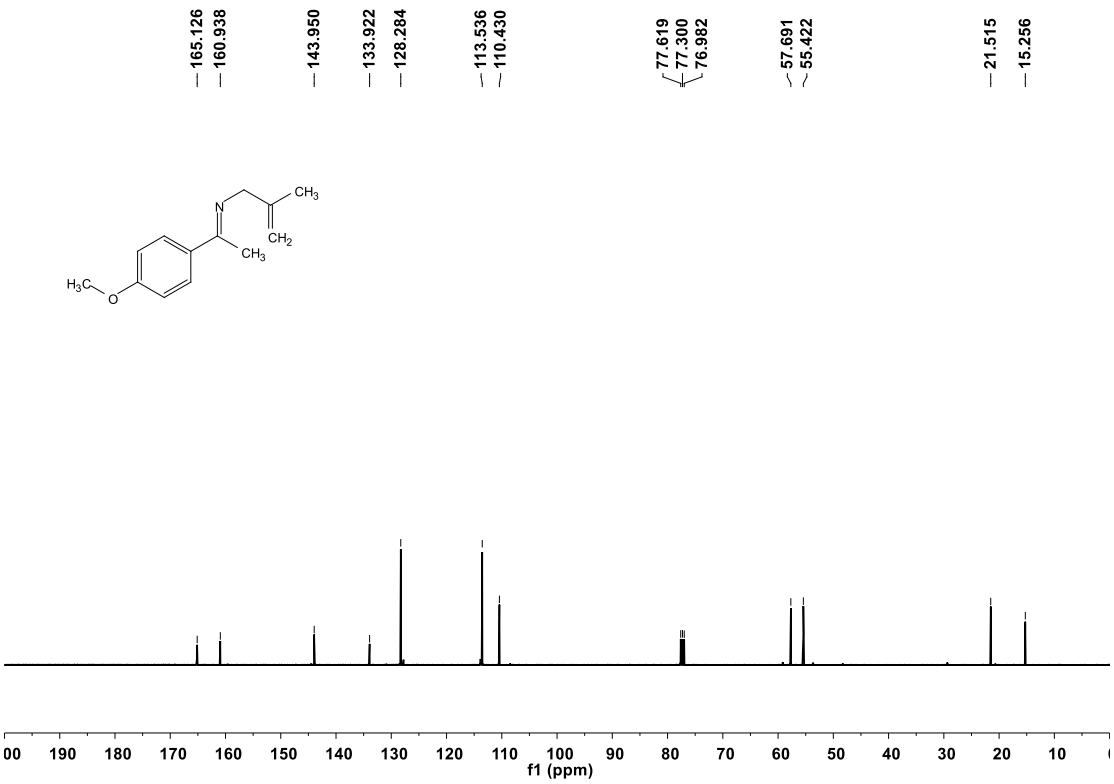
^{1}H NMR spectrum of (E)-*N*-(1-(3-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1k)



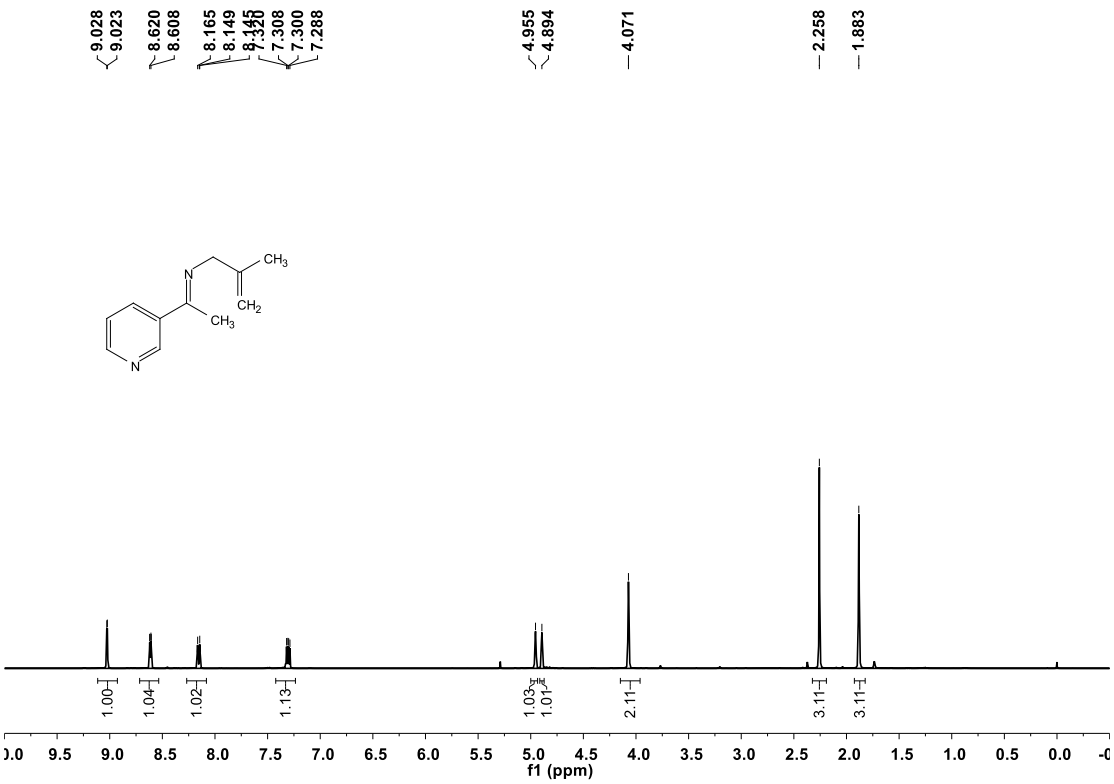
^{13}C NMR spectrum of (E)-*N*-(1-(3-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1k)



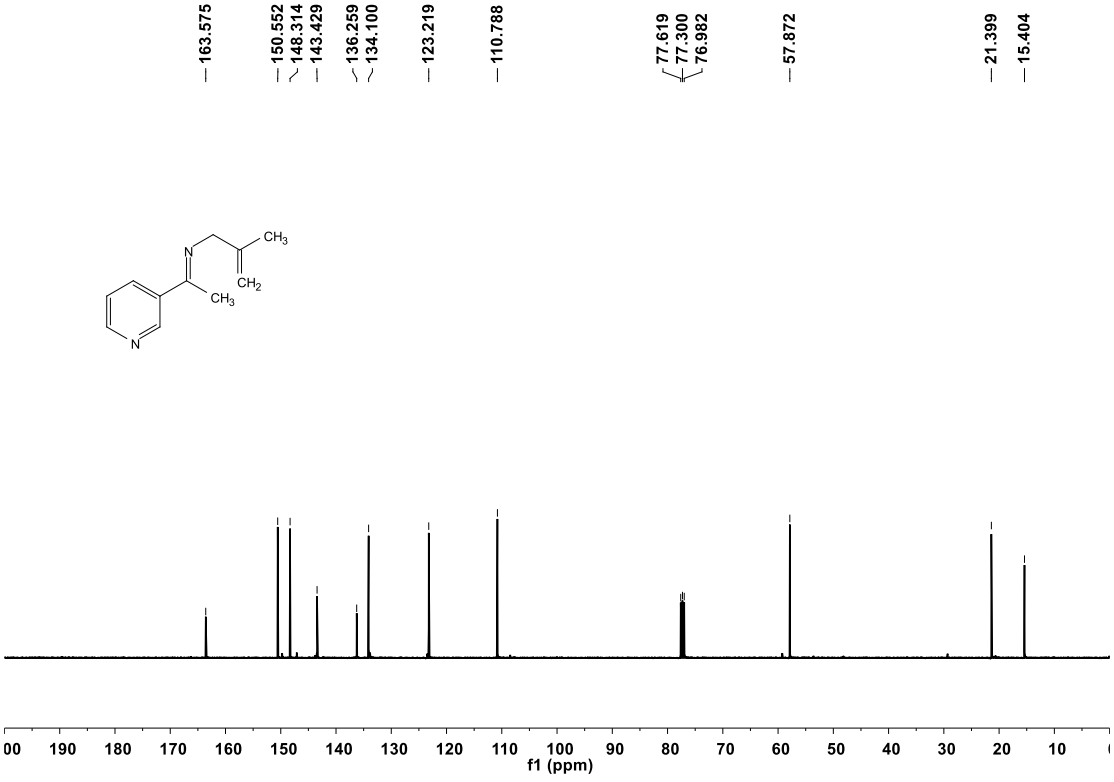
^{1}H NMR spectrum of (E)-*N*-(1-(4-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1l)



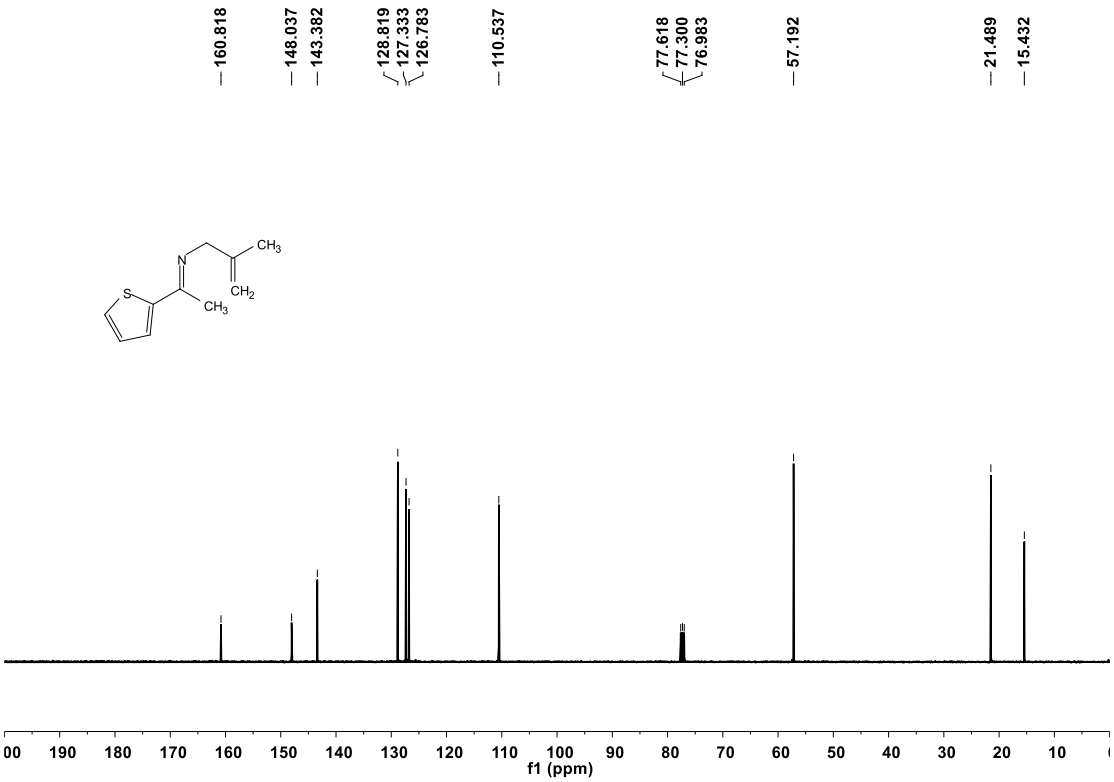
^{13}C NMR spectrum of (E)-*N*-(1-(4-methoxyphenyl)ethylidene)-2-methylprop-2-en-1-amine (1l)



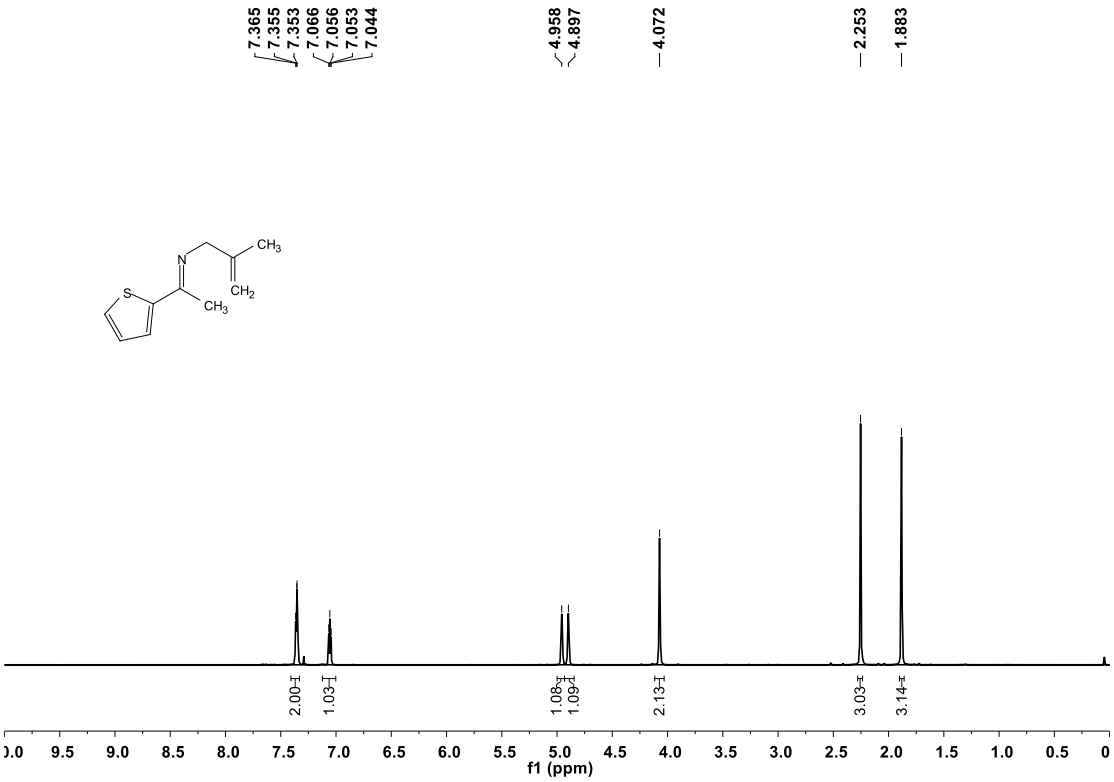
^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-(pyridin-3-yl)ethylidene)prop-2-en-1-amine (1m)



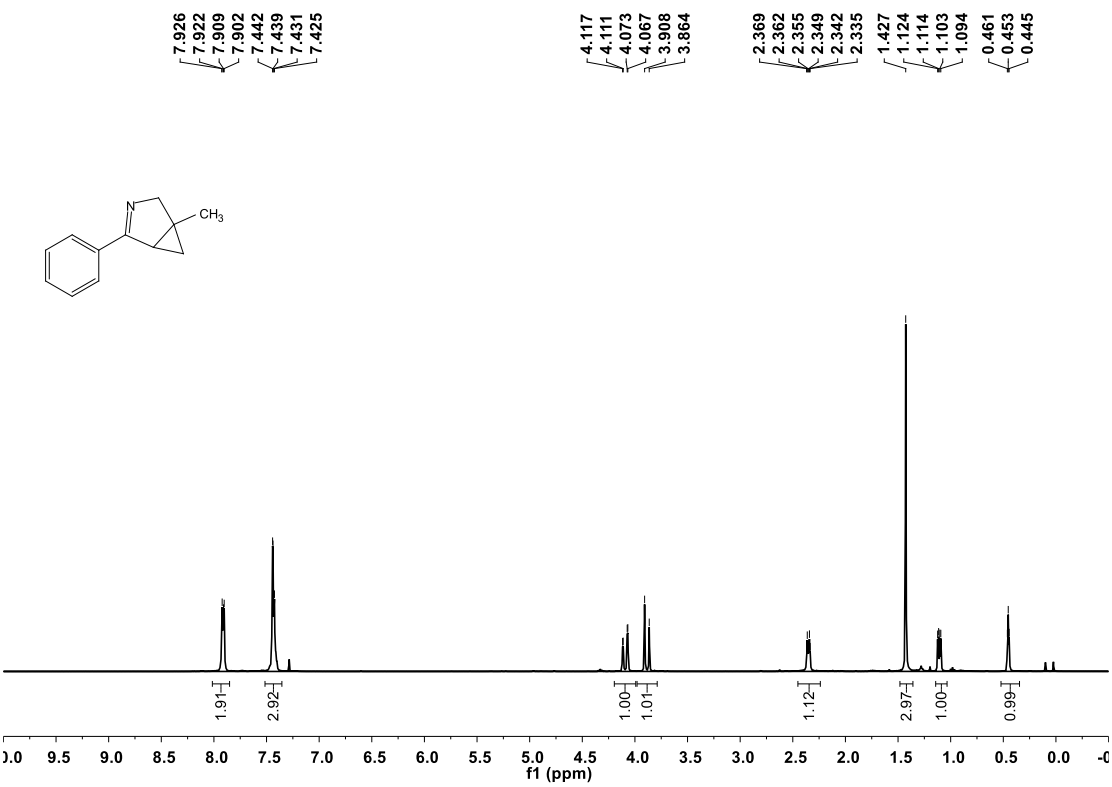
^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-(pyridin-3-yl)ethylidene)prop-2-en-1-amine (1m)



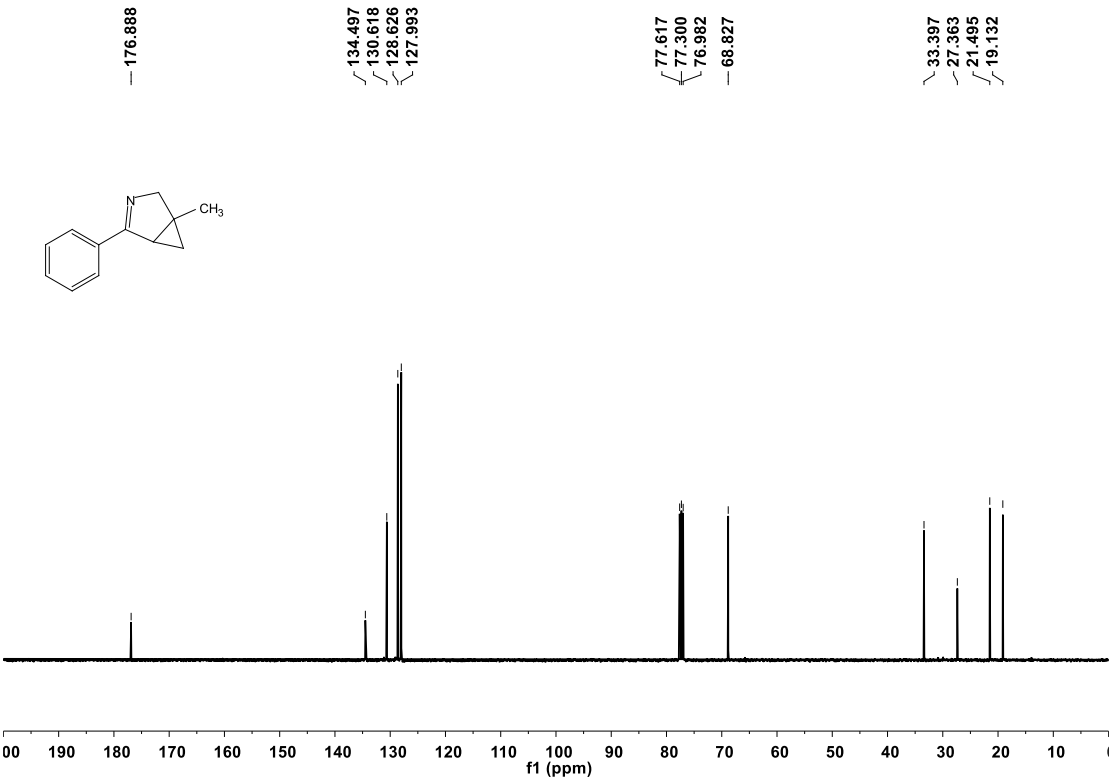
^{1}H NMR spectrum of (E)-2-methyl-*N*-(1-(thiophen-2-yl)ethylidene)prop-2-en-1-amine (1n)



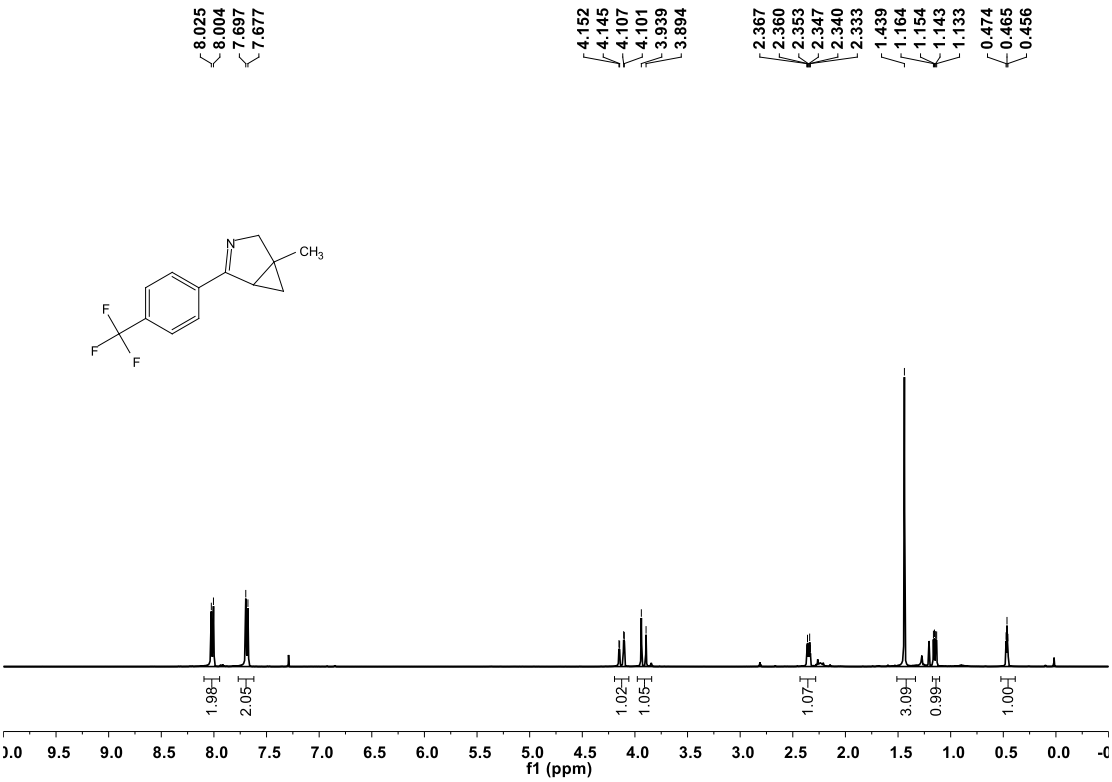
^{13}C NMR spectrum of (E)-2-methyl-*N*-(1-(thiophen-2-yl)ethylidene)prop-2-en-1-amine (1n)



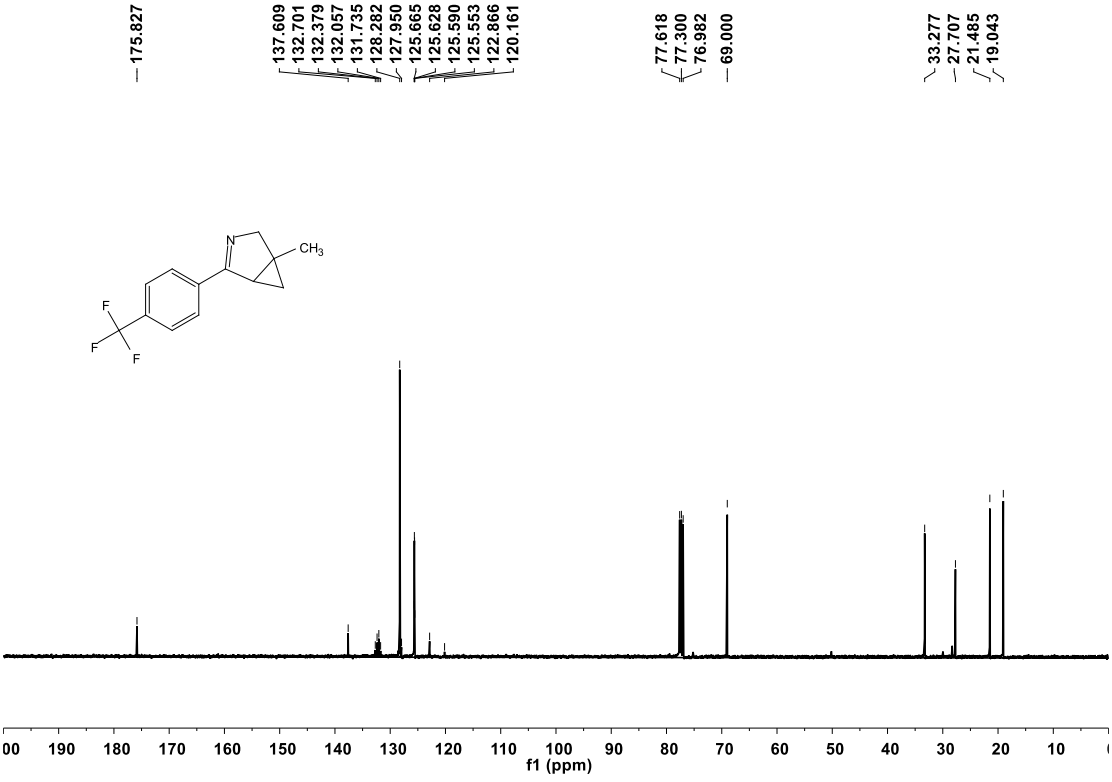
^{1}H NMR spectrum of 1-methyl-4-phenyl-3-azabicyclo[3.1.0]hex-3-ene (2a)



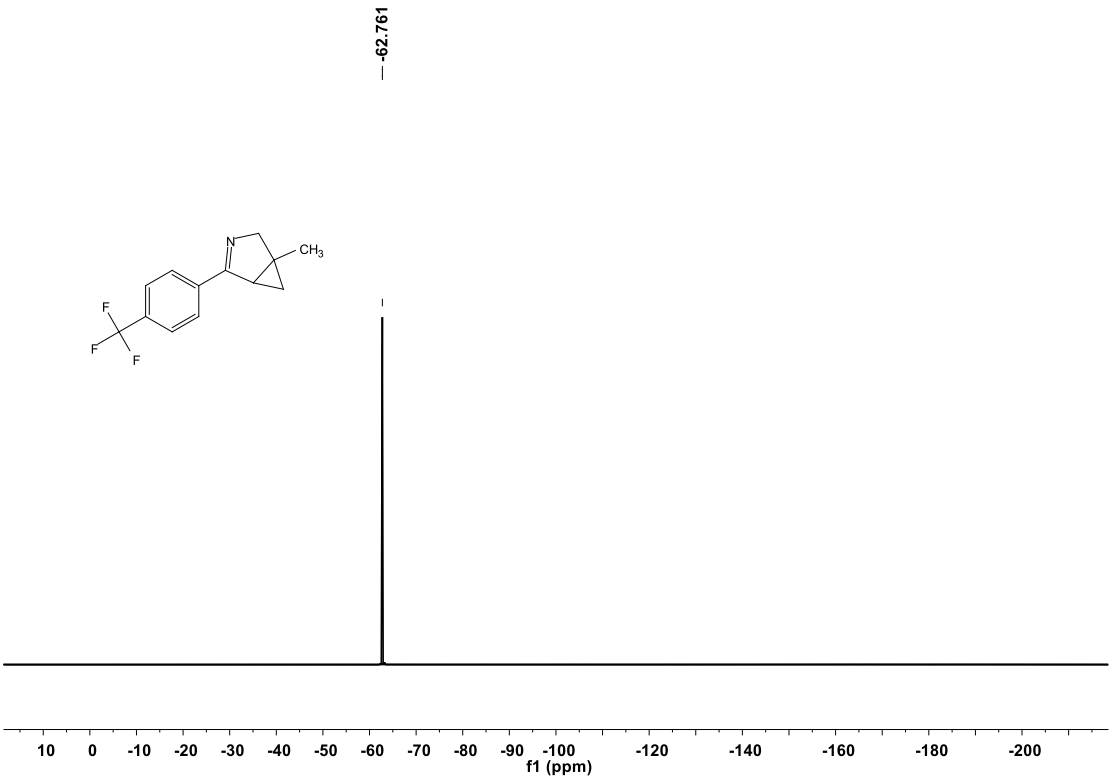
^{13}C NMR spectrum of 1-methyl-4-phenyl-3-azabicyclo[3.1.0]hex-3-ene (2a)



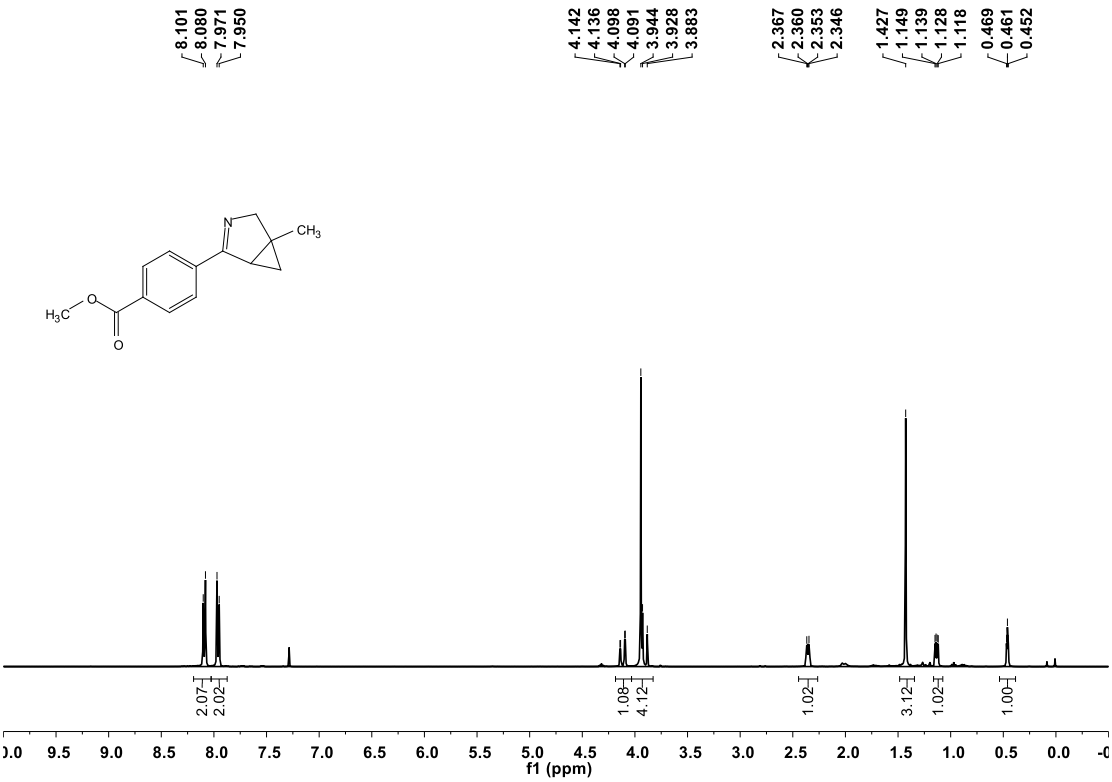
^{1}H NMR spectrum of 1-methyl-4-(4-(trifluoromethyl)phenyl)-3-azabicyclo[3.1.0]hex-3-ene (2b)



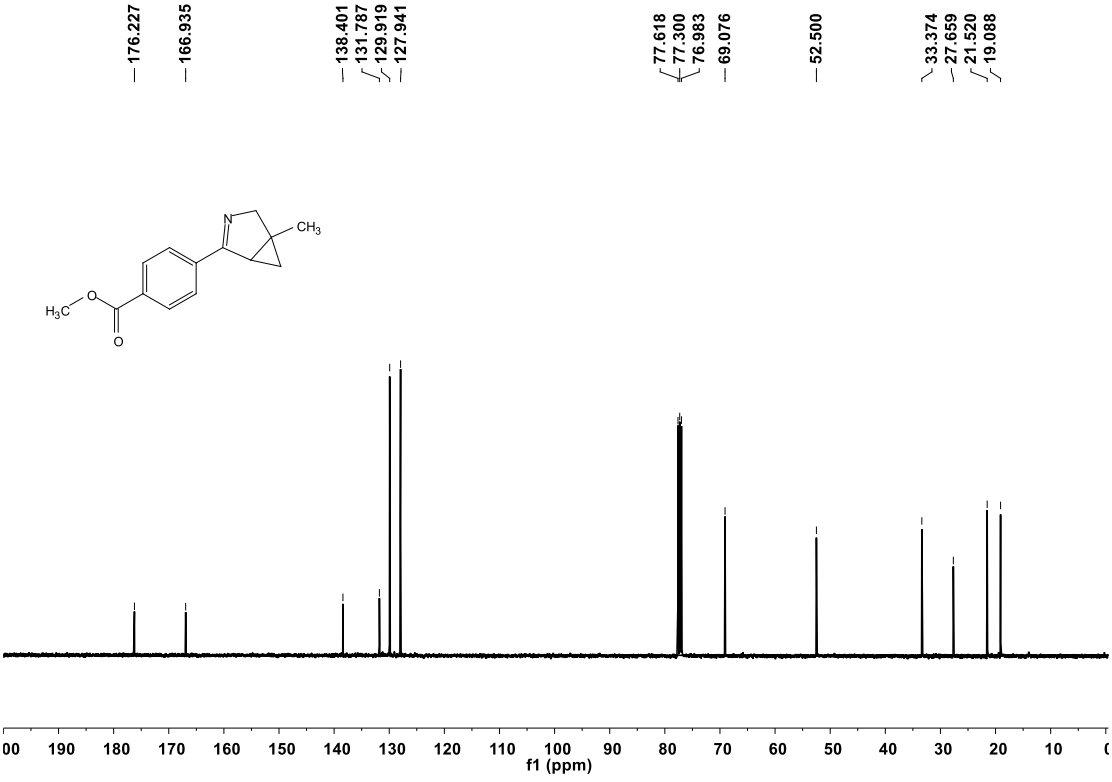
^{13}C NMR spectrum of 1-methyl-4-(4-(trifluoromethyl)phenyl)-3-azabicyclo[3.1.0]hex-3-ene (2b)



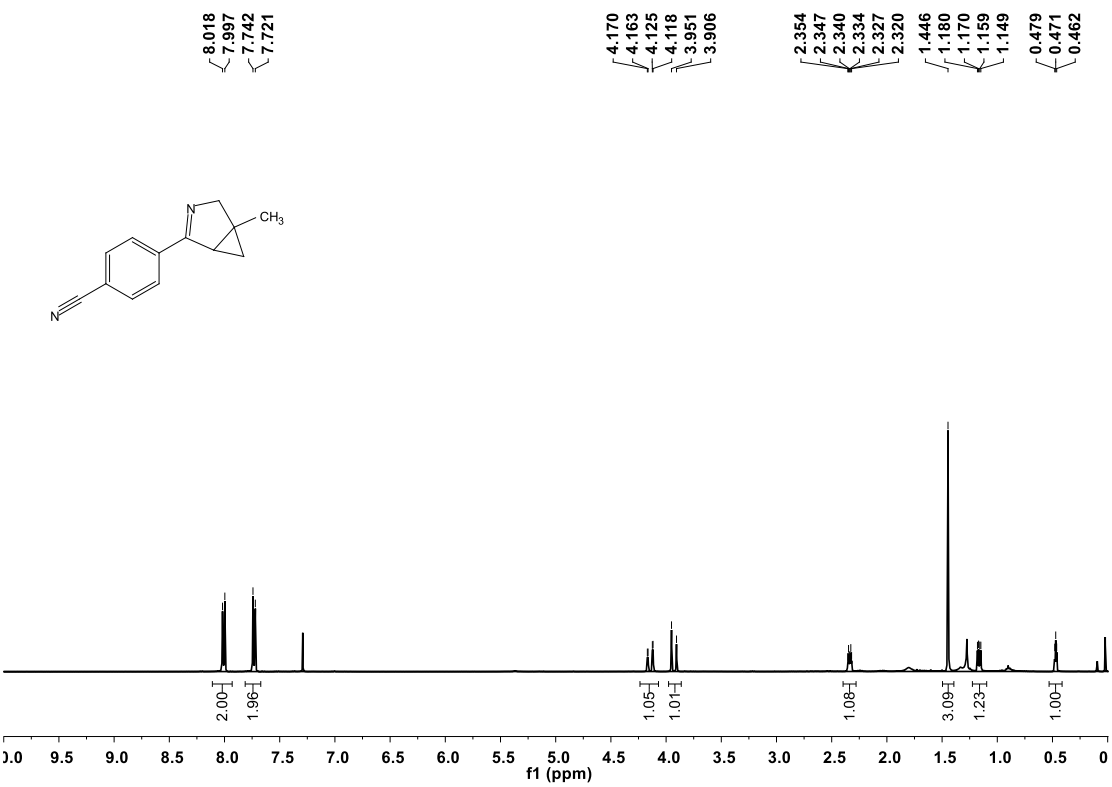
^{19}F NMR spectrum of 1-methyl-4-(4-(trifluoromethyl)phenyl)-3-azabicyclo[3.1.0]hex-3-ene (2b)



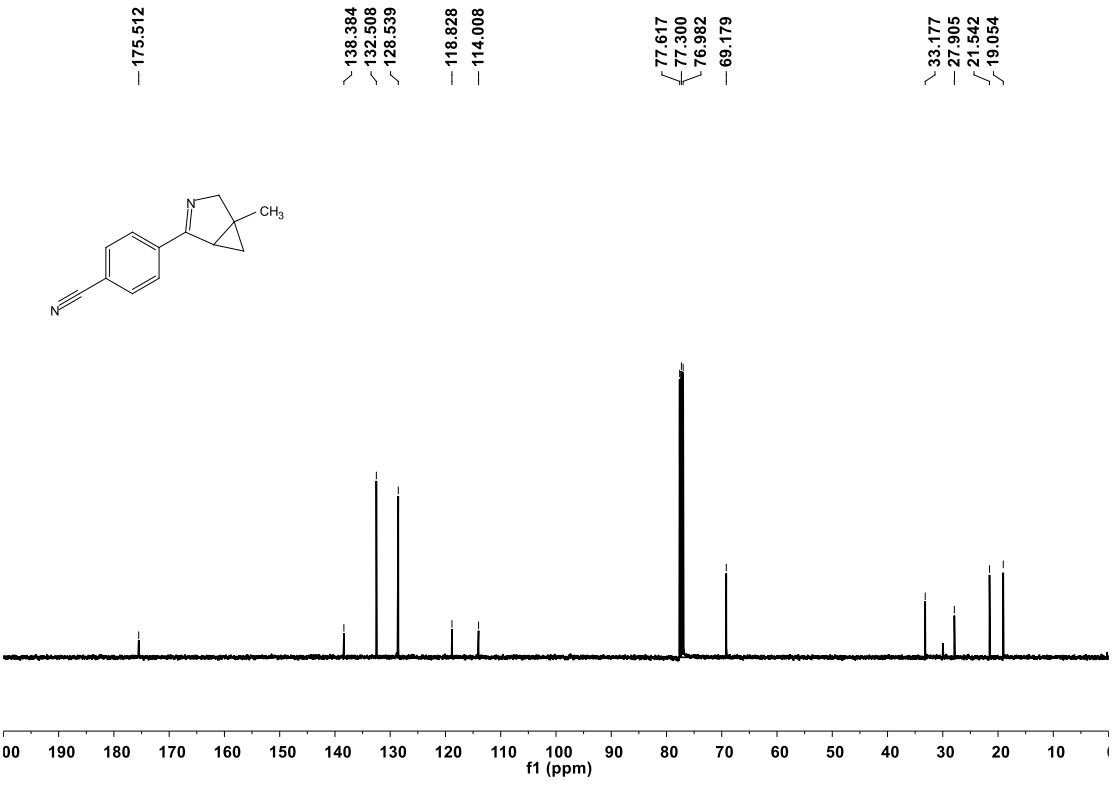
^{1}H NMR spectrum of methyl 4-(5-methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzoate (2c)



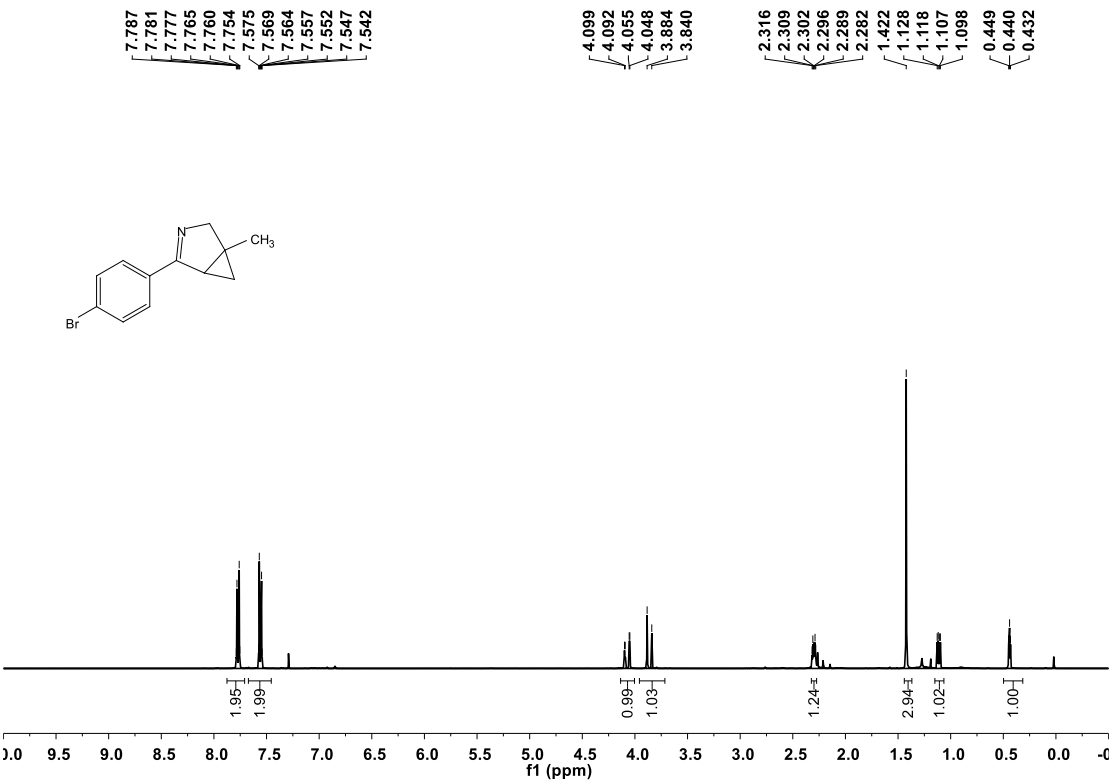
^{13}C NMR spectrum of methyl 4-(5-methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzoate (2c)



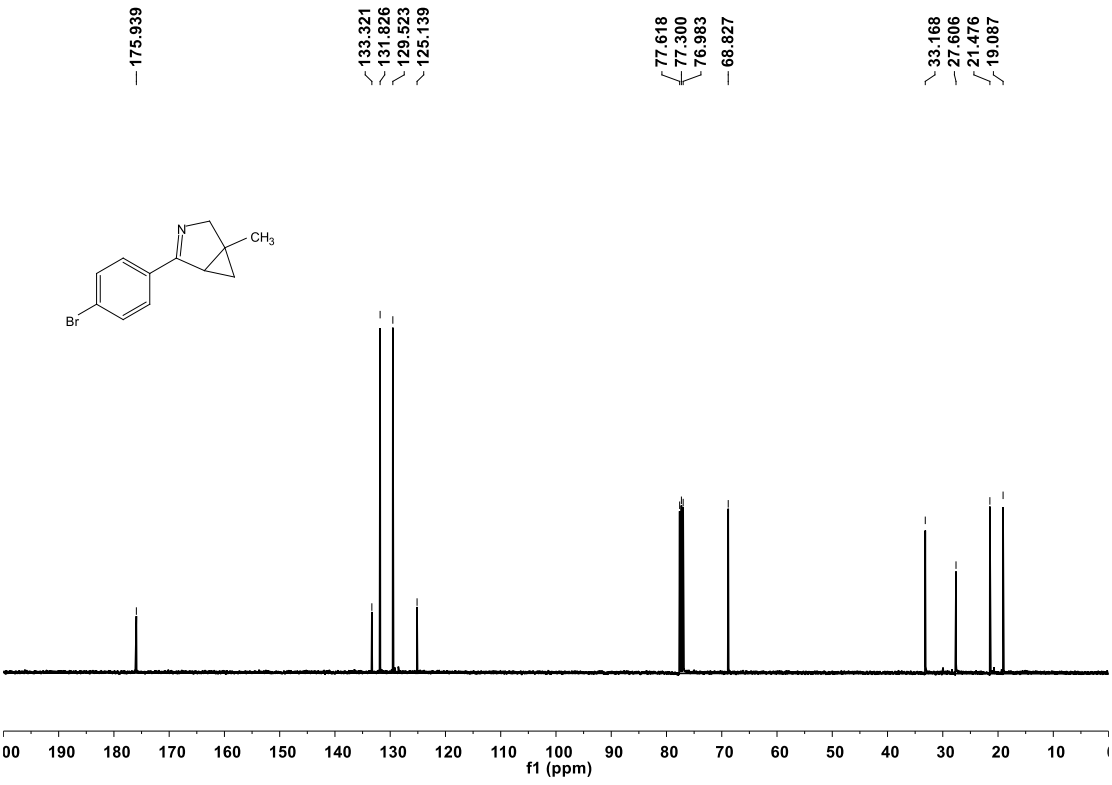
^{1}H NMR spectrum of 4-(5-Methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzonitrile (2d)



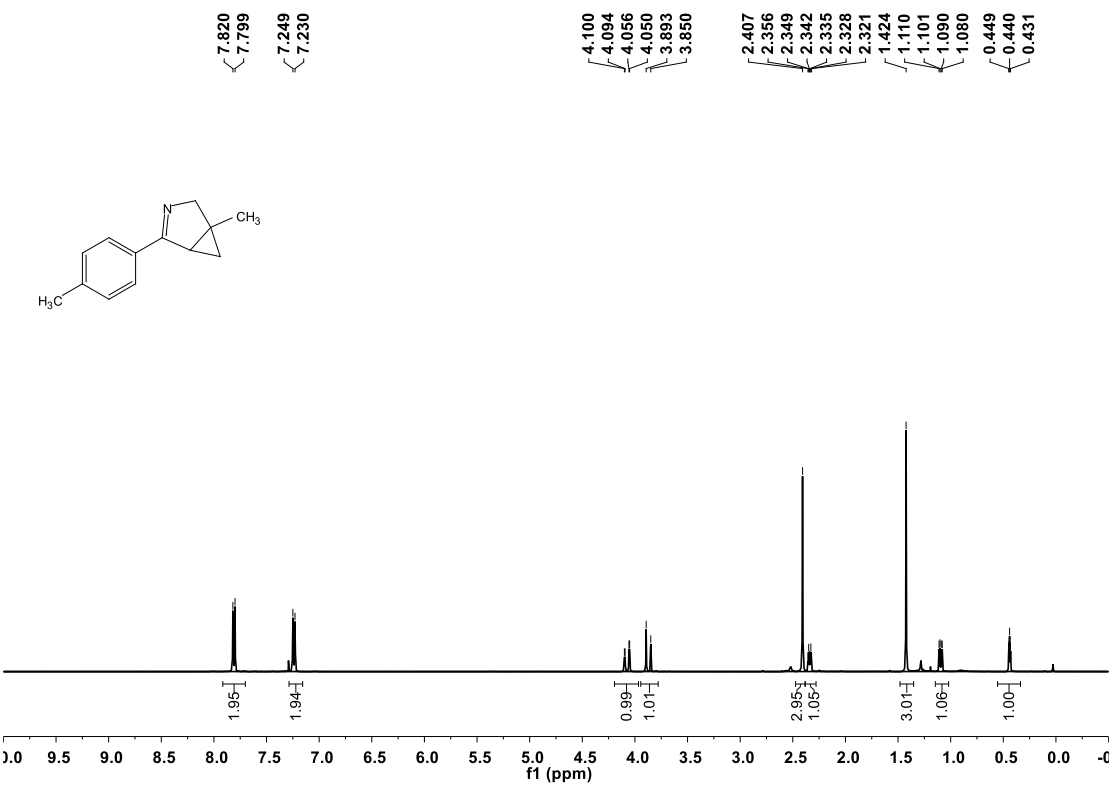
^{13}C NMR spectrum of 4-(5-Methyl-3-azabicyclo[3.1.0]hex-2-en-2-yl)benzonitrile (2d)



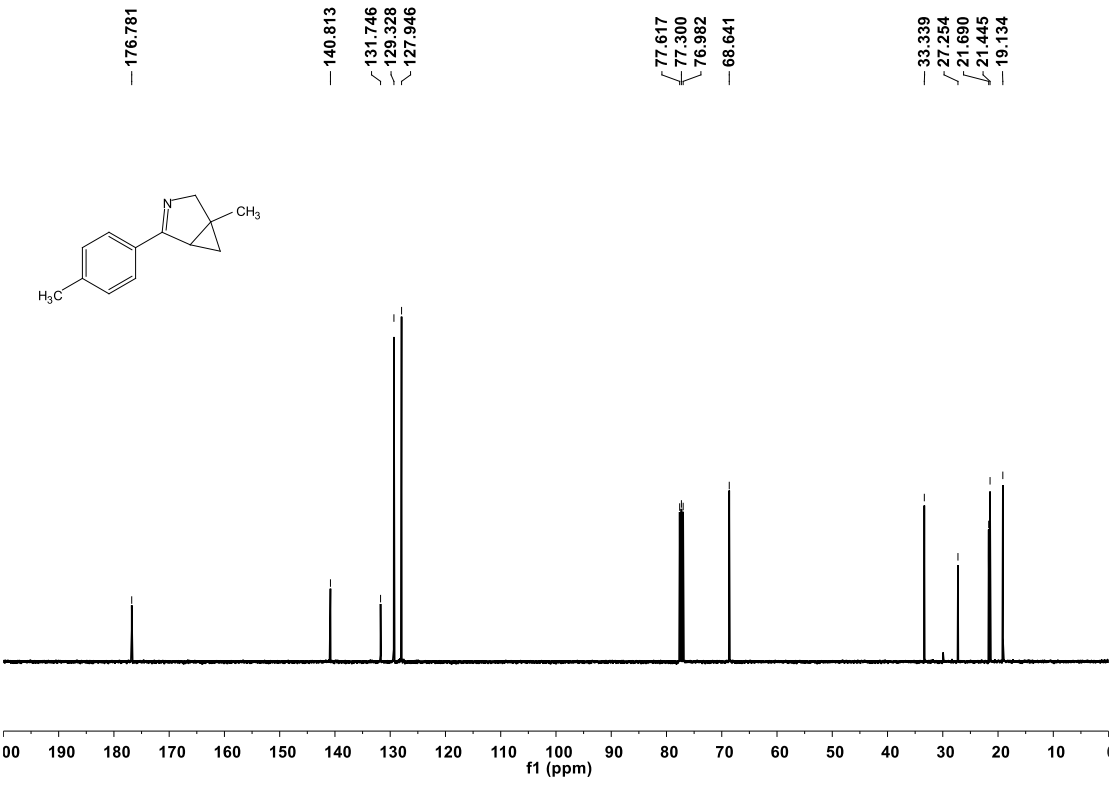
^{1}H NMR spectrum of 4-(4-bromophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2f)



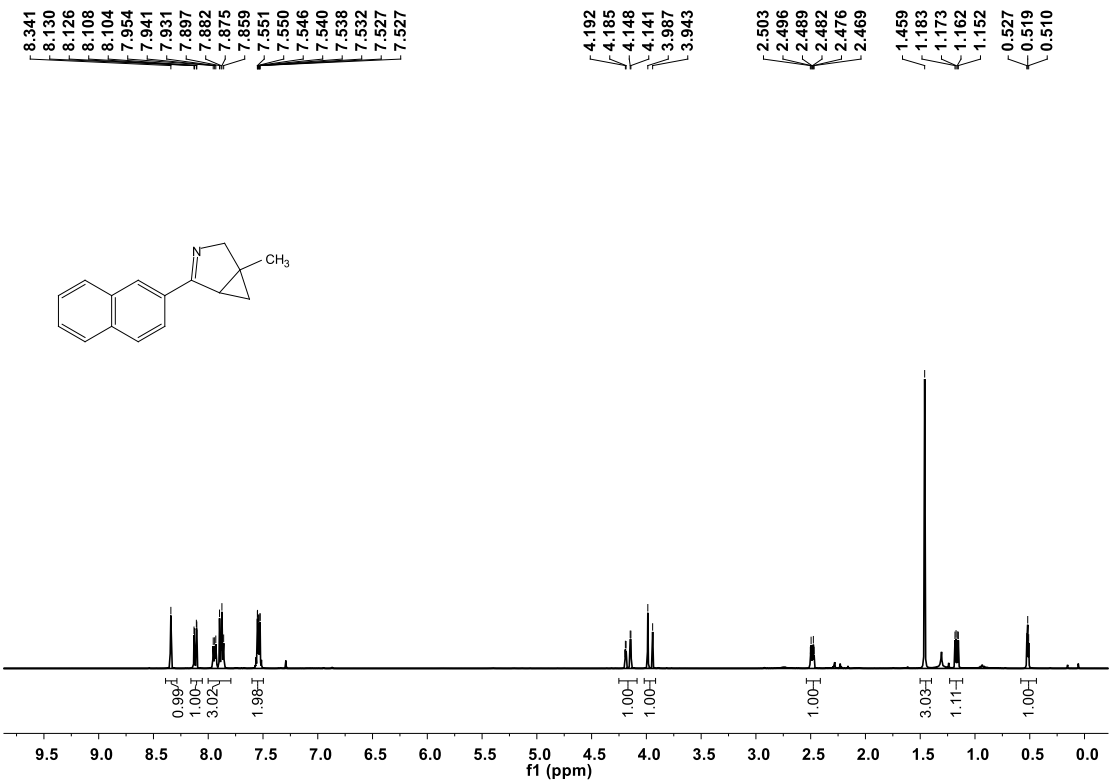
^{13}C NMR spectrum of 4-(4-bromophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2f)



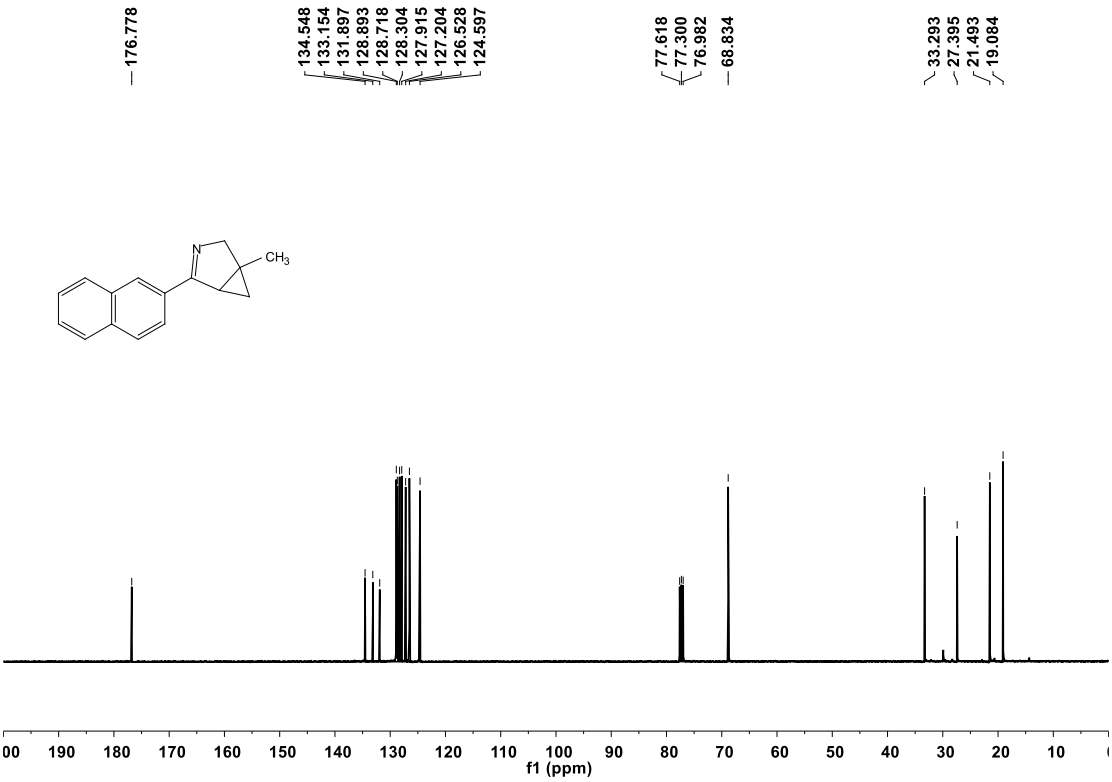
^{1}H NMR spectrum of 1-methyl-4-(p-tolyl)-3-azabicyclo[3.1.0]hex-3-ene (2g)



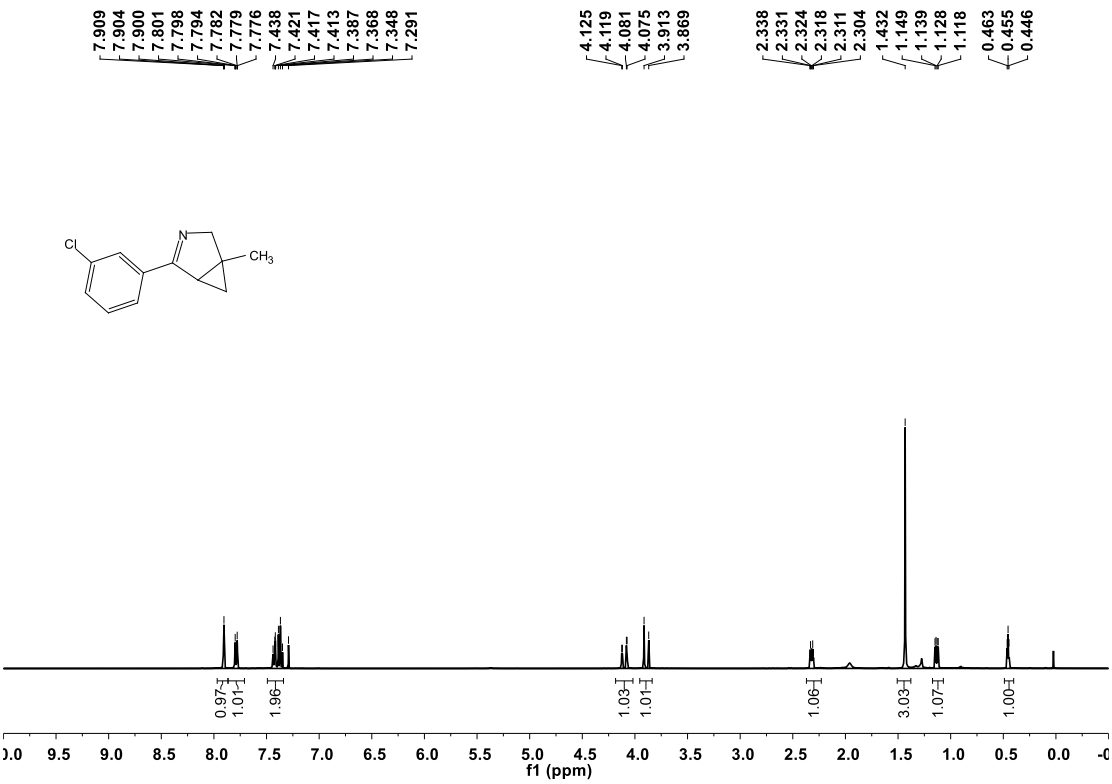
^{13}C NMR spectrum of 1-methyl-4-(p-tolyl)-3-azabicyclo[3.1.0]hex-3-ene (2g)



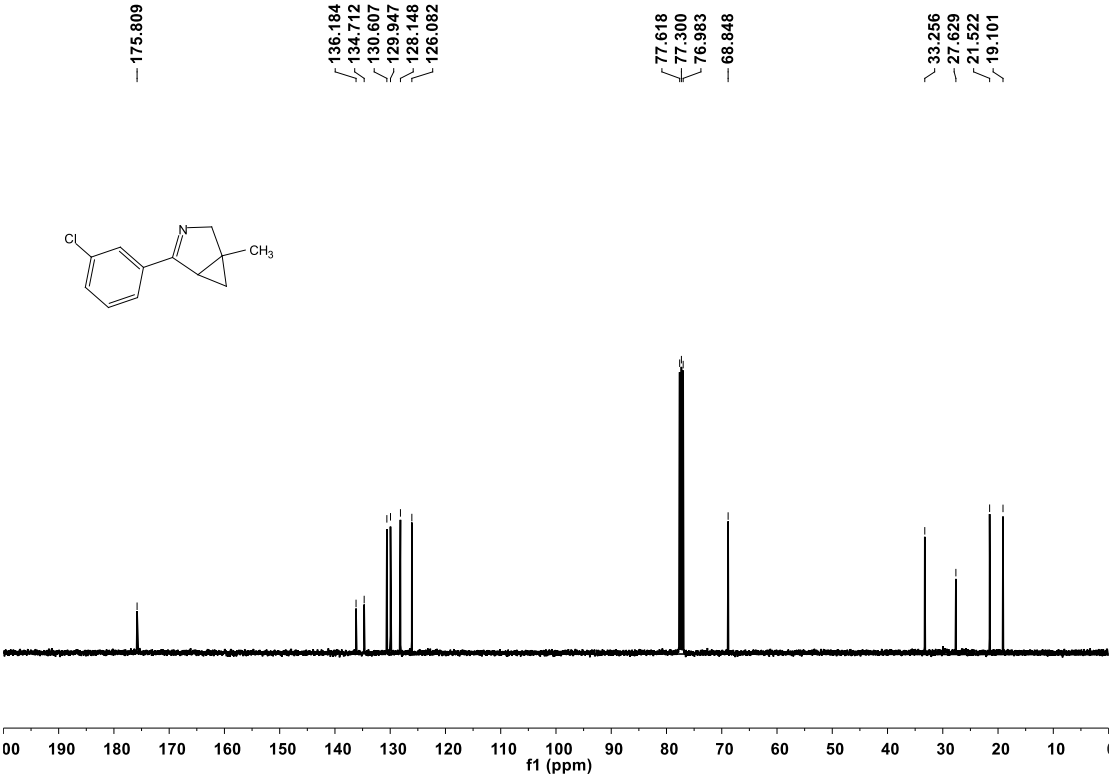
^{1}H NMR spectrum of 1-methyl-4-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2h)



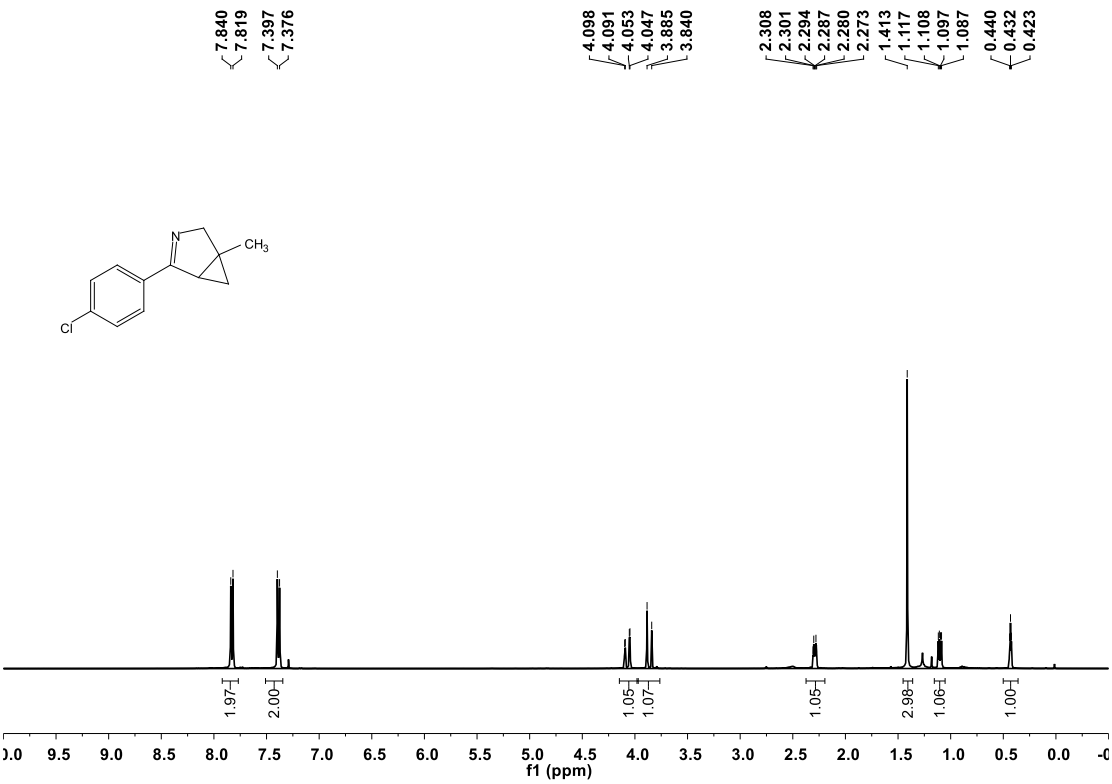
^{13}C NMR spectrum of 1-methyl-4-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2h)



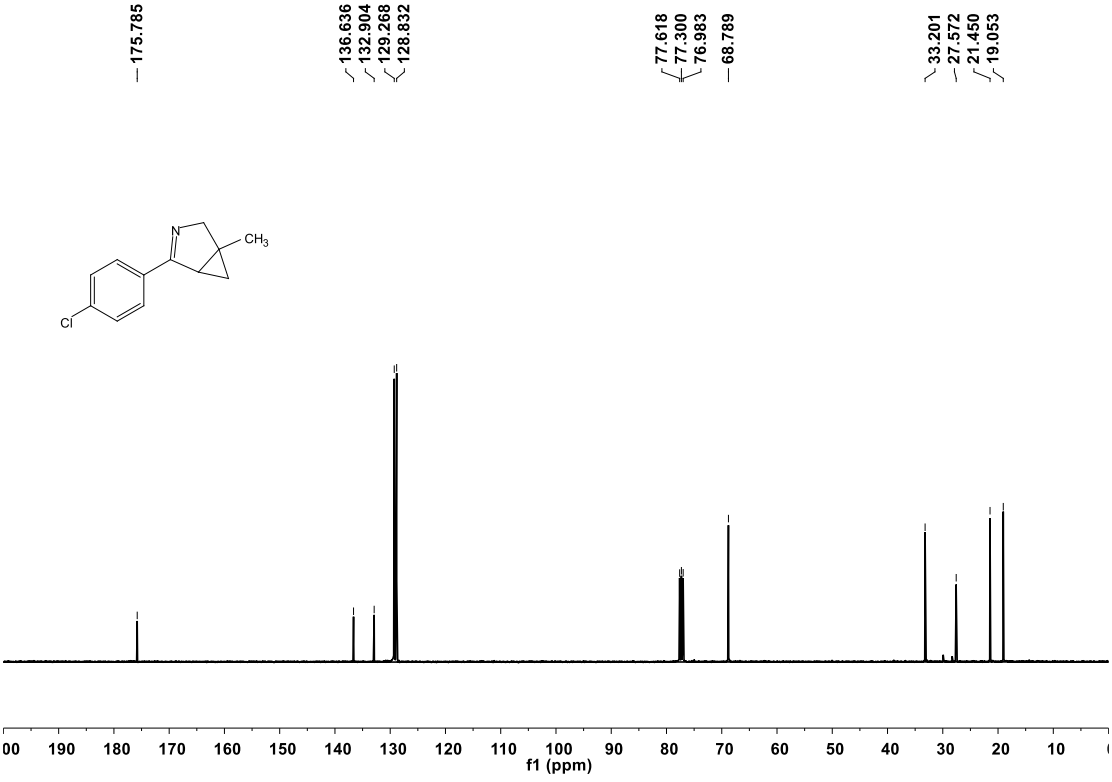
^{1}H NMR spectrum of 4-(3-chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2i)



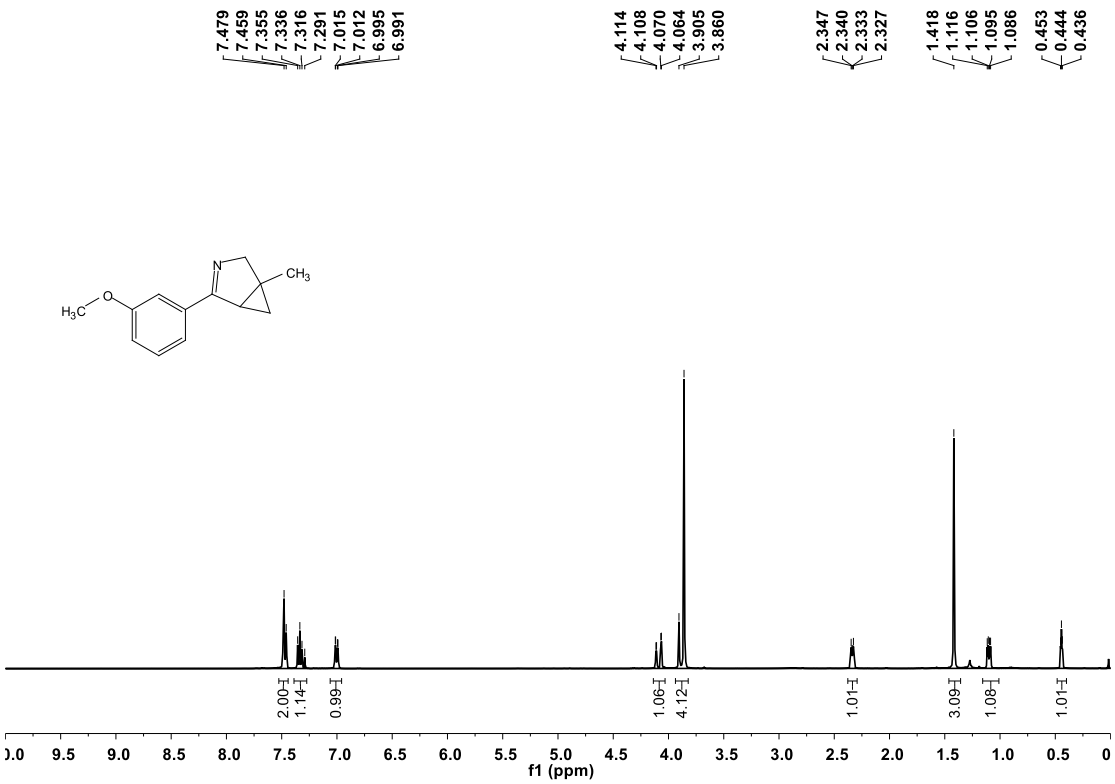
^{13}C NMR spectrum of 4-(3-chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2i)



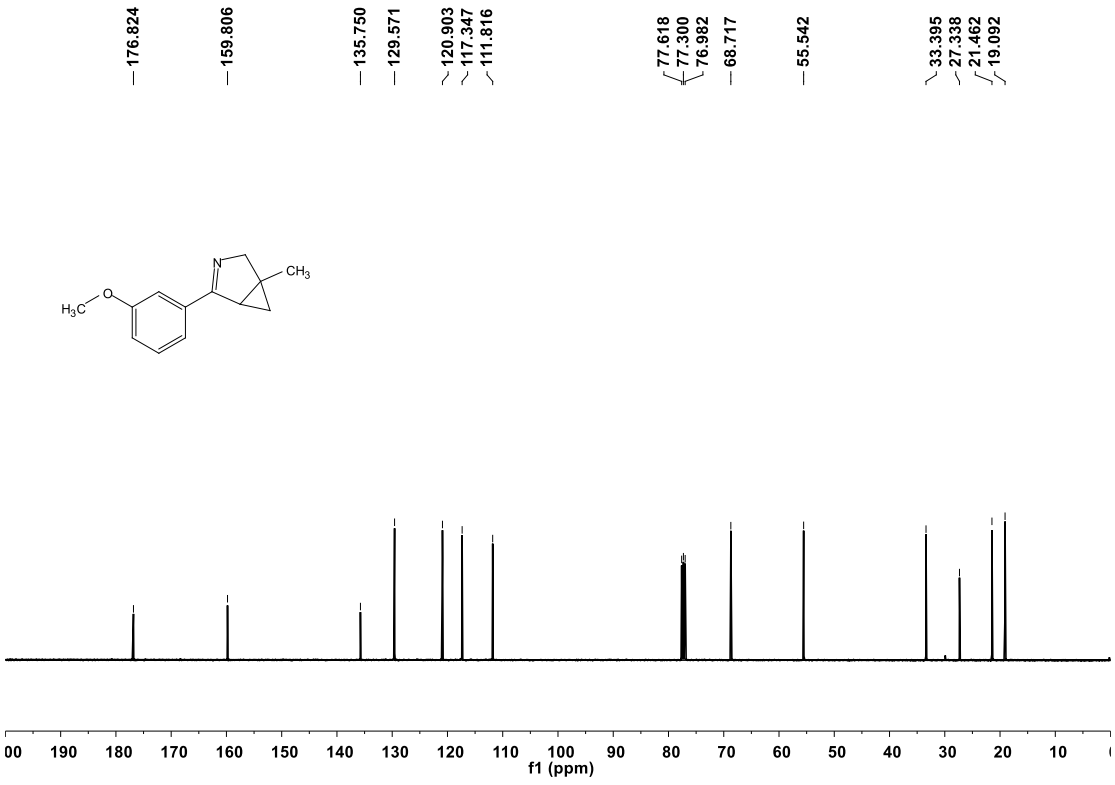
^{1}H NMR spectrum of 4-(4-chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2j)



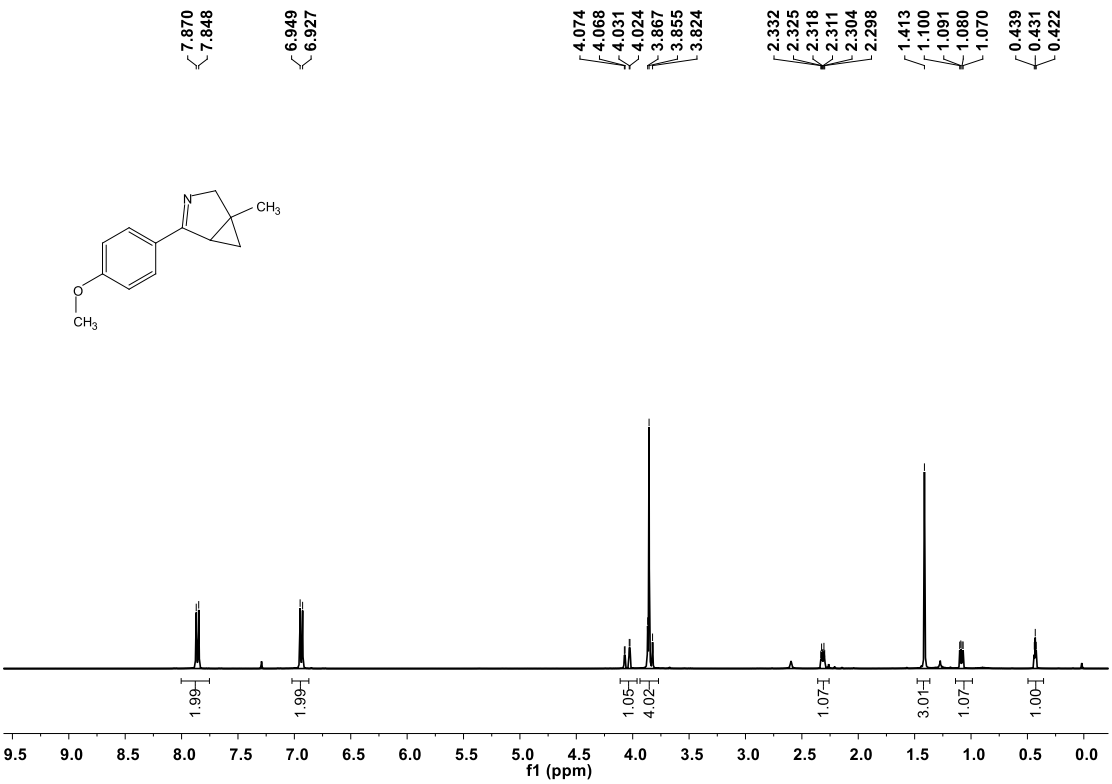
^{13}C NMR spectrum of 4-(4-chlorophenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2j)



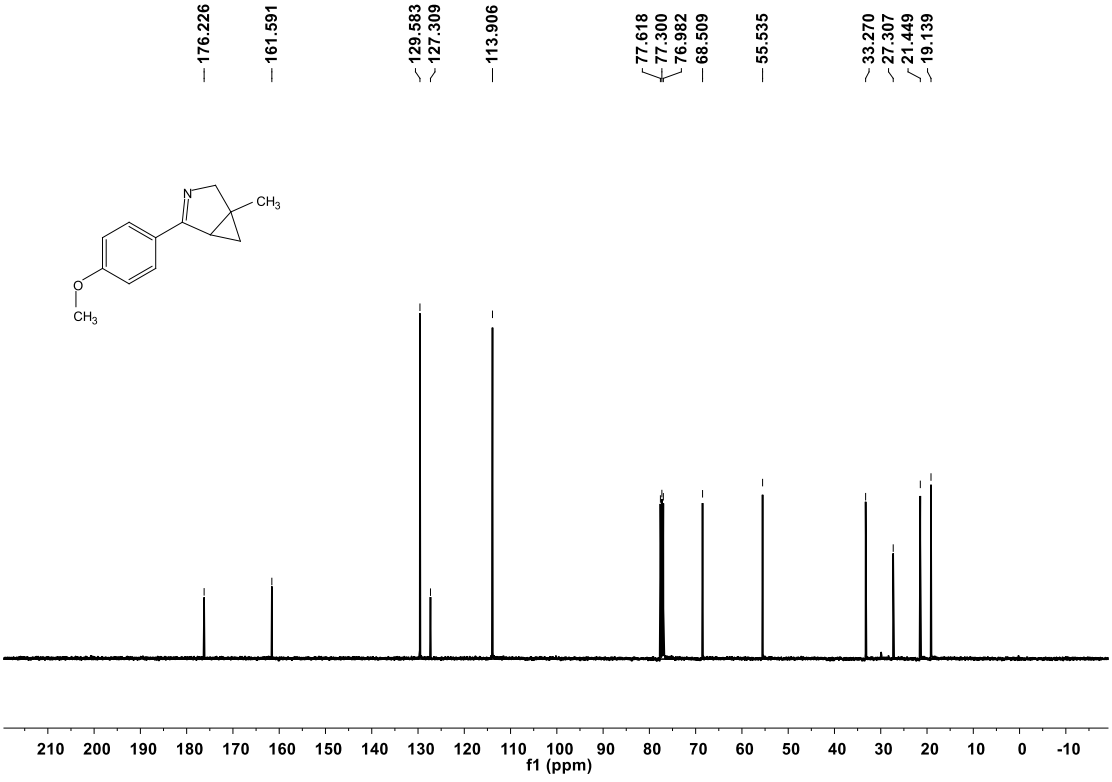
^{1}H NMR spectrum of 4-(3-methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2k)



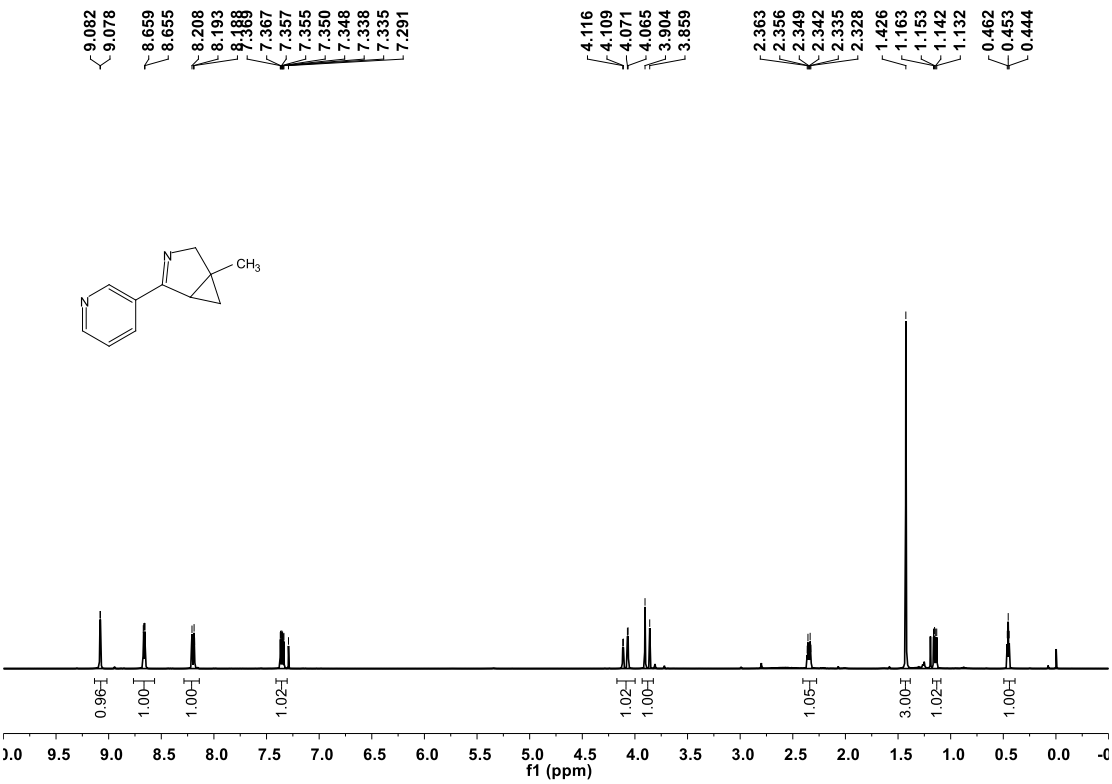
^{13}C NMR spectrum of 4-(3-methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2k)



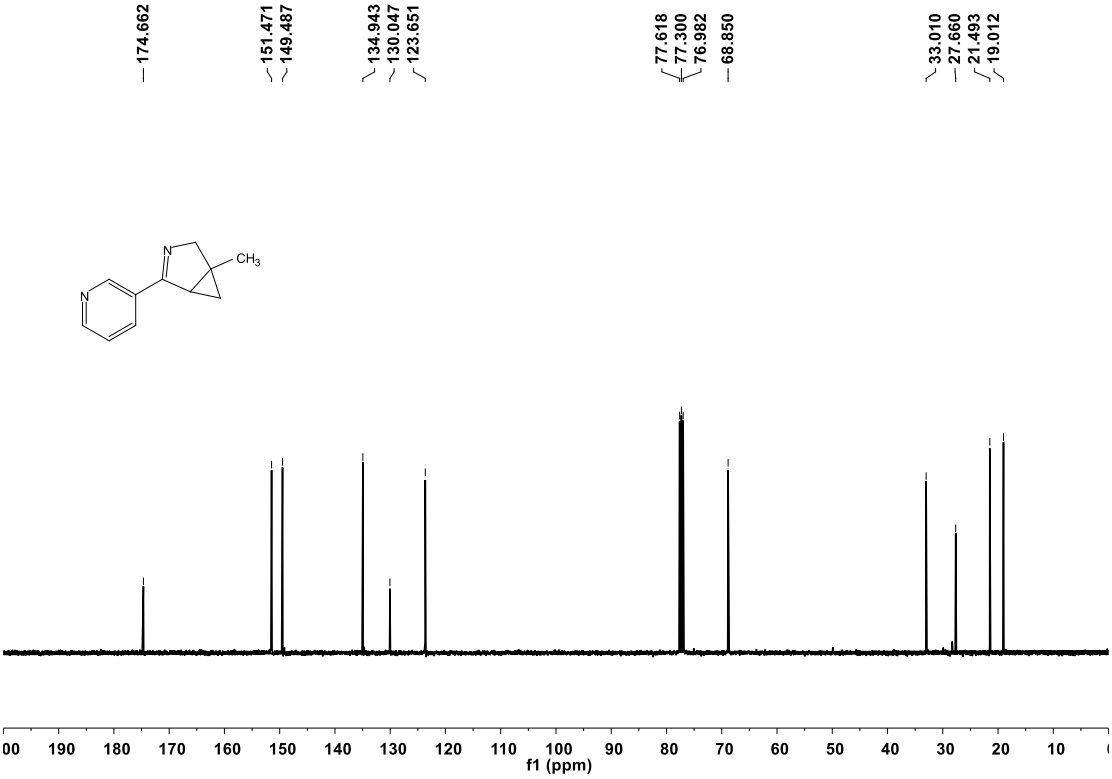
^{1}H NMR spectrum of 4-(4-methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2l)



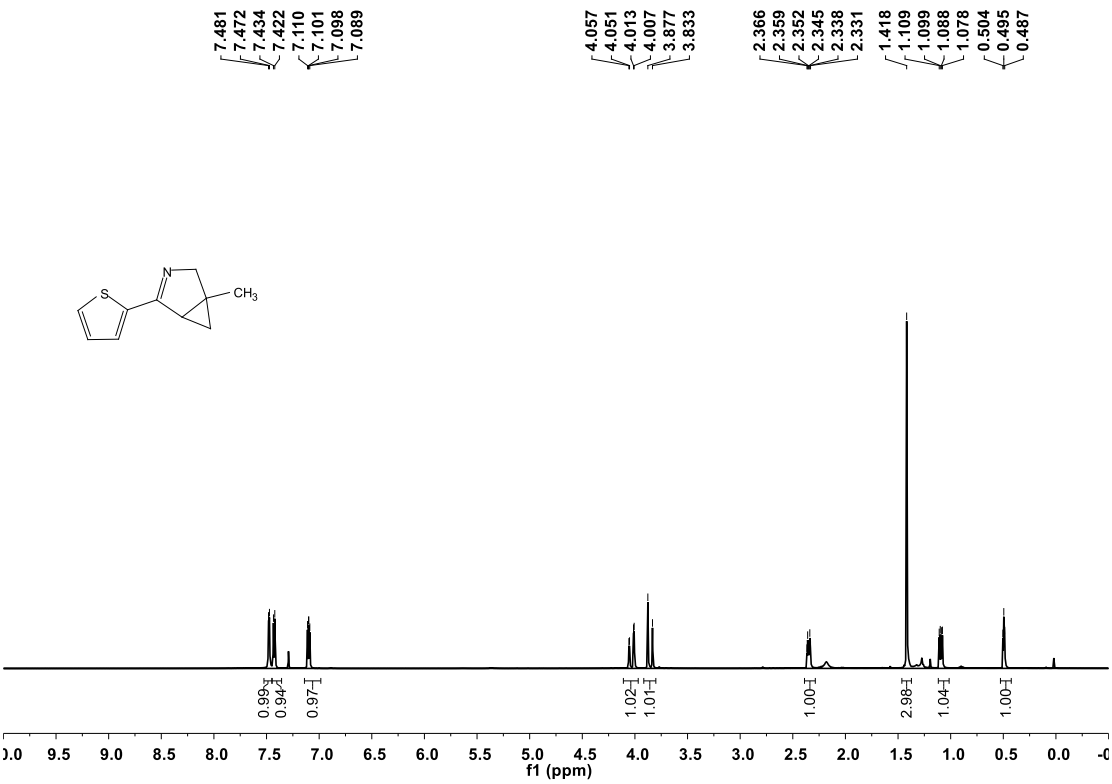
^{13}C NMR spectrum of 4-(4-methoxyphenyl)-1-methyl-3-azabicyclo[3.1.0]hex-3-ene (2l)



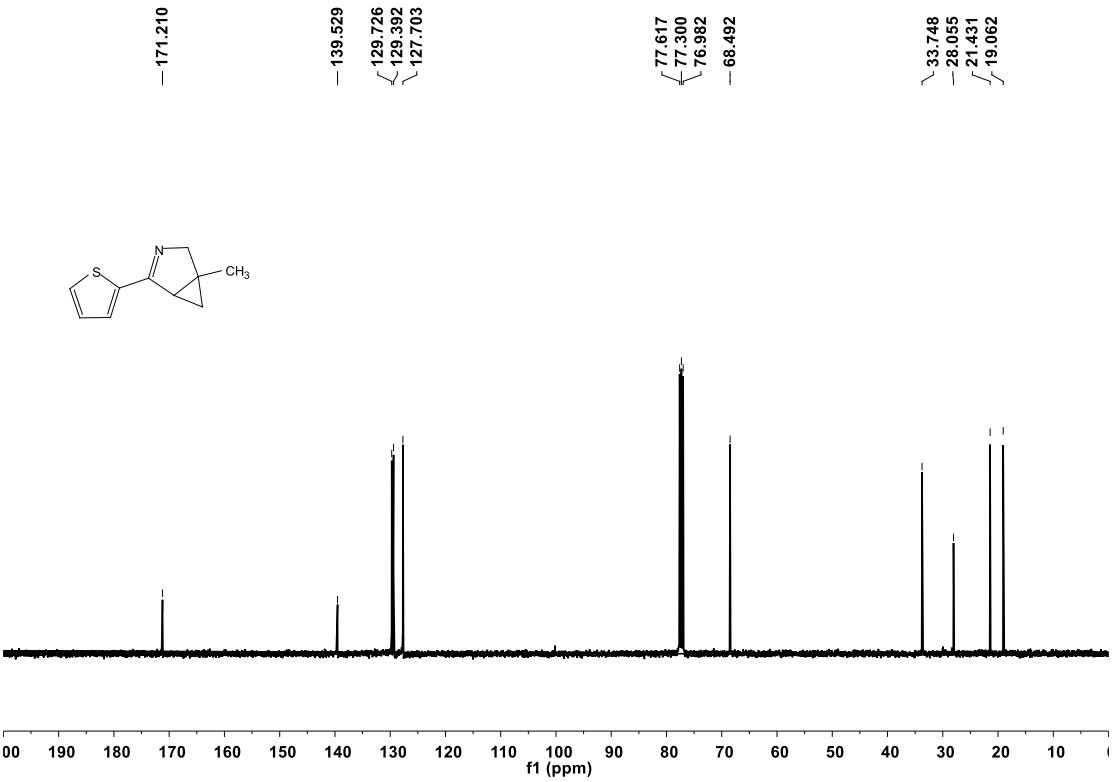
^{1}H NMR spectrum of 1-methyl-4-(pyridin-3-yl)-3-azabicyclo[3.1.0]hex-3-ene (2m)



^{13}C NMR spectrum of 1-methyl-4-(pyridin-3-yl)-3-azabicyclo[3.1.0]hex-3-ene (2m)



^{1}H NMR spectrum of 1-methyl-4-(thiophen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2n)



^{13}C NMR spectrum of 1-methyl-4-(thiophen-2-yl)-3-azabicyclo[3.1.0]hex-3-ene (2n)