Supporting Information for:

Transition metal-free iodine-promoted haloamination of unfunctionalized olefins

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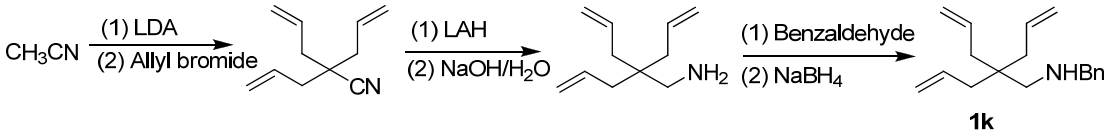
1. Instrumentation and Chemicals

Reactions were carried out using commercially available reagents in oven-dried apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX 400 spectrometer at 298 K using deuterated chloroform as solvent and TMS as the internal reference. Column chromatography was performed employing 200-300 mesh silica gel unless otherwise noted. Thin layer chromatography (TLC) was performed on silica gel GF254. HRMS analyses were carried out with Varian FTICR-MS 7.0T. Unless otherwise indicated, starting materials and reagents used in reactions were purchased from J&K Chemicals (Beijing) or Aladdin Reagents (Shanghai) and were used as received without further purification.

2. Preparation of Substrates

Preparation of Aminoalkenes: aminoalkenes **1a-1j** and **1l-1o** were prepared according to our previous work.[1] Aminoalkenes **1p** and **1q** were obtained according to the reported procedure.[2] Aminoalkenes **1r-1x** were synthesized from 2,2-diphenylhex-5-en-1-amine and the corresponding aldehyde.



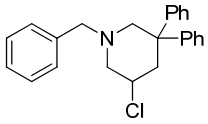
1k

2,2-diallyl-N-benzylpent-4-en-1-amine (1k)[3] A flask was charged with acetonitrile (30 mmol) and THF (50 mL). This solution was cooled to -78 °C. A solution of LDA (32 mmol) was added slowly to the acetonitrile solution. After 30 minutes of stirring at -78 °C, allyl bromide (32 mmol) was added. The reaction mixture was warmed to room temperature with stirring for 1 hour before being cooled to -78 °C again. Another equivalent of LDA solution was added, followed by 30 minutes of stirring, followed by the addition of another equivalent of allyl bromide. The reaction was again warmed to room temperature with stirring for 1 hour before being cooled to -78 °C. The third LDA solution was added, followed by 30 minutes of stirring, followed by the addition of a third equivalent of allyl bromide. Then the reaction was allowed to warm to room temperature with stirring overnight. CH_2Cl_2 (75 mL) was added and the resulting biphasic mixture was washed with water (3×100 mL), dried ($MgSO_4$), and concentrated to give 2,2-diallylpent-4-enenitrile. Conversion of 2,2-diallylpent-4-enenitrile to **1k** was accomplished in a manner similar to that employed for the synthesis **1a-1j**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 6.93 (m, 5H), 6.06 – 5.52 (m, 3H), 4.96 (dd, J = 7.6, 6.9 Hz, 6H), 3.65 (s, 2H), 2.30 (s, 2H), 1.97 (d, J = 7.5 Hz, 6H), 1.11 (brs, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=141.05, 134.82, 128.31, 128.08, 126.82, 117.42, 54.78, 54.70, 40.16, 39.81. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{25}N$, 256.2060; found: 256.2061.

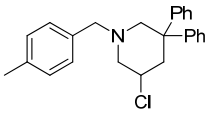
3. Typical Procedure

Typical Procedure for iodine-promoted haloamination reaction: the reaction was carried out in open air system. To a 100 mL flask was added 0.5 mmol alkenylamine, 0.01 mmol iodine, 1.5 mmol LiCl (or Py·HBr) and 20 mL of THF (or CH_2Cl_2). The reaction mixture was stirred at room temperature for an indicated period. Then 30 mL of CH_2Cl_2 was added and the mixture was washed with H_2O, dried over $MgSO_4$ and was concentrated to give an oil which was purified by flash chromatography to give the corresponding product.

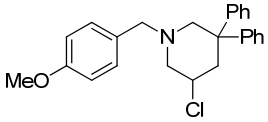
4. Characterization of Products



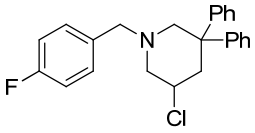
1-benzyl-5-chloro-3,3-diphenylpiperidine (2a) Oil. ^{1}H NMR (400 MHz, CDCl3) δ = 7.17 – 6.98 (m, 15H), 3.77 (t, J = 11.1 Hz, 1H), 3.57 – 3.42 (m, 2H), 3.12 (d, J = 9.8 Hz, 1H), 2.90 (d, J = 12.2 Hz, 1H), 2.23 (dd, J = 27.8, 12.3 Hz, 1H), 2.11 (t, J = 10.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.22, 143.78, 136.48, 128.19, 127.46, 127.26, 126.98, 126.35, 125.32, 125.28, 124.86, 61.43, 60.89, 60.29, 52.51, 47.13, 44.85. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{24}ClN$, 362.1670; found:362.1669.



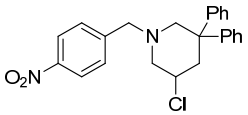
1-(4-methylbenzyl)-5-chloro-3,3-diphenylpiperidine (2b) White solid. M.p.135-136°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.25 – 7.01 (m, 14H), 3.77 (ddd, *J* = 14.8, 8.0, 3.9 Hz, 1H), 3.56 – 3.42 (m, 3H), 3.14 (dd, *J* = 10.3, 3.9 Hz, 1H), 2.90 (d, *J* = 12.4 Hz, 1H), 2.33 – 2.06 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.30, 143.87, 135.94, 133.37, 128.19, 127.95, 127.53, 127.28, 126.98, 125.36, 125.29, 124.85, 61.17, 60.83, 60.29, 52.60, 47.15, 44.91, 20.13. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}ClN$,376.1827; found:376.1826.



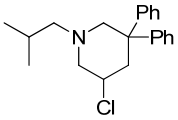
1-(4-methoxybenzyl)-5-chloro-3,3-diphenylpiperidine (2c) White solid. M.p.99-100°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 – 6.47 (m, 14H), 3.81 – 3.68 (m, 4H), 3.51 – 3.42 (m, 3H), 3.13 (dd, *J* = 10.3, 4.2 Hz, 1H), 2.90 (dt, *J* = 23.2, 6.9 Hz, 1H), 2.25 (t, *J* = 12.3 Hz, 1H), 2.18 – 2.04 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.89, 146.30, 143.84, 129.40, 128.51, 127.49, 127.28, 126.99, 125.35, 125.28, 124.86, 112.62, 60.81, 60.69, 60.32, 54.22, 52.62, 47.13, 44.91. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}ClNO$, 392.1776; found:392.1768.



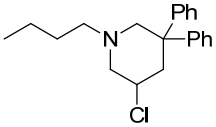
1-(4-fluorolbenzyl)-5-chloro-3,3-diphenylpiperidine(2d) White solid. M.p.109-110°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.23 – 6.85 (m, 14H), 3.76 (tt, *J* = 11.6, 3.9 Hz, 1H), 3.54 – 3.36 (m, 3H), 3.08 (dd, *J* = 10.1, 3.9 Hz, 1H), 2.90 (d, *J* = 12.5 Hz, 1H), 2.21 (dd, *J* = 26.1, 12.3 Hz, 2H), 2.10 (t, *J* = 10.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.34, 159.90, 146.13, 143.68, 132.24, 129.71, 129.64, 127.36, 127.31, 127.03, 125.34, 125.30, 124.93, 114.21, 113.99, 60.79, 60.59, 60.28, 52.39, 47.12, 44.80. ^{19}F NMR (376 MHz, $CDCl_3$) δ = -115.03. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{23}ClFN$, 380.1576; found: 380.1573



1-(4-nitrobenzyl)-5-chloro-3,3-diphenylpiperidine(2e) ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.14 – 8.01 (m, 2H), 7.37 (d, *J* = 8.7 Hz, 2H), 7.22 – 7.00 (m, 10H), 3.87 – 3.77 (m, 1H), 3.70 – 3.52 (m, 2H), 3.48 (d, *J* = 12.0 Hz, 1H), 3.07 (dd, *J* = 10.4, 4.3 Hz, 1H), 2.99 – 2.93 (m, 1H), 2.33 (d, *J* = 12.0 Hz, 1H), 2.21 (dt, *J* = 21.1, 11.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.36, 145.86, 144.43, 143.44, 128.61, 127.40, 127.20, 127.18, 125.48, 125.26, 125.14, 122.59, 61.22, 60.57, 60.29, 52.02, 47.21, 44.58. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{23}ClN_2O_2$, 407.1521; found:407.1520.

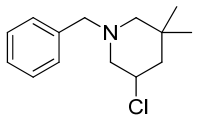


1-i-butyl-5-chloro-3,3-diphenylpiperidine(2f) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 7.30 (m, 2H), 7.17 (dd, *J* = 15.1, 7.1 Hz, 5H), 7.09 – 7.06 (m, 3H), 3.75 (tt, *J* = 10.9, 3.9 Hz, 1H), 3.47 (d, *J* = 12.1 Hz, 1H), 3.15 (dd, *J* = 10.1, 3.7 Hz, 1H), 2.91 – 2.78 (m, 1H), 2.25 (t, *J* = 12.2 Hz, 1H), 2.18 – 1.96 (m, 4H), 1.88 – 1.72 (m, 1H), 0.88 – 0.80 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.51, 144.24, 127.67, 127.28, 126.90, 125.40, 125.28, 124.81, 65.62, 62.56, 60.53, 52.81, 47.27, 45.11, 24.58, 20.10, 19.81. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{26}ClN$,328.1827; found:328.1828.

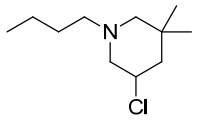


1-n-butyl-5-chloro-3,3-diphenylpiperidine(2g) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.26 – 7.06 (m, 10H), 3.75 (tt, *J* = 11.6, 4.0 Hz, 1H), 3.53 (d, *J* = 12.1 Hz, 1H), 3.15 (dd, *J* = 10.3, 4.1 Hz, 1H), 2.87 (d, *J* = 12.4 Hz, 1H), 2.36 (t, *J* = 7.4 Hz, 2H), 2.30 – 2.19 (m, 1H), 2.14 – 2.04 (m, 2H), 1.54 – 1.38 (m, 2H), 1.33 – 1.21 (m, 2H), 0.84 (dt, *J* = 8.7, 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.50, 144.18, 127.51, 127.28,

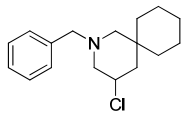
127.03, 125.38, 125.28, 124.86, 61.47, 60.60, 56.81, 52.74, 47.09, 45.04, 27.66, 19.63, 13.01. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{26}ClN$,328.1827; found:328.1830.



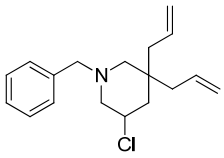
1-benzyl-5-chloro-3,3-dimethylpiperidine(2h) ^1H NMR (400 MHz, $CDCl_3$) δ = 7.31 (d, *J* = 23.2 Hz, 5H), 4.14 (t, *J* = 4.5 Hz, 1H), 3.58 (d, *J* = 13.4 Hz, 2H), 3.19 (d, *J* = 12.3 Hz, 1H), 2.42 (d, *J* = 11.0 Hz, 1H), 2.01 (dd, *J* = 25.3, 14.5 Hz, 2H), 1.78 (d, J = 11.0 Hz, 1H), 1.38 (t, J = 12.3 Hz, 2H), 1.09 (s, 3H), 0.91 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 137.51, 127.61, 127.20, 125.99, 63.48, 61.27, 60.87, 53.21, 47.36, 32.35, 28.25, 24.05. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{20}ClN$, 238.1357; found:238.1360.



1-n-butyl-5-chloro-3,3-dimethylpiperidine(2i) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ = 4.07 (ddt, *J* = 11.8, 10.9, 4.4 Hz, 1H), 3.14 (ddd, *J* = 20.6, 11.5, 9.2 Hz, 1H), 2.40 (d, *J* = 11.1 Hz, 1H), 2.37 – 2.22 (m, 2H), 1.98 – 1.85 (m, 2H), 1.68 (d, *J* = 11.1 Hz, 1H), 1.48 – 1.37 (m, 2H), 1.36 – 1.24 (m, 4H), 1.07 – 0.99 (m, 3H), 0.93 – 0.87 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 63.74, 61.34, 56.67, 53.41, 47.44, 32.22, 28.43, 28.05, 24.19, 19.48, 12.98. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{24}ClN$, 204.1512; found:204.1441.

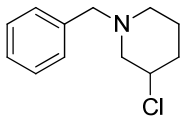


2-benzyl-4-chloro-2-azaspiro[5.5]undecane(2j) ^1H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 7.23 (m, 5H), 4.12 (tt, *J* = 10.8, 4.5 Hz, 1H), 3.59 – 3.40 (m, 2H), 3.23 – 3.12 (m, 1H), 2.69 (d, *J* = 11.3 Hz, 1H), 2.23 – 2.12 (m, 1H), 2.05 (t, *J* = 10.6 Hz, 1H), 1.62 (d, *J* = 11.3 Hz, 2H), 1.40 – 1.15 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.63, 128.58, 128.20, 127.00, 62.58, 62.36, 62.02, 54.01, 46.24, 38.32, 36.04, 32.90, 26.59, 21.54, 21.51. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{24}ClN$, 278.1670; found:278.1672.

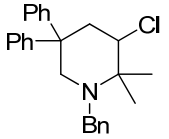


3,3-diallyl-1-benzyl-5-chloropiperidine (2k) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ7.57 – 7.17 (m, 5H), 6.03 – 5.63 (m, 2H), 5.10 (dd, J = 16.0, 10.3 Hz, 4H), 4.18 (ddd, J = 15.7, 11.1, 4.4 Hz, 1H), 3.61 – 3.49 (m, 2H), 3.22 (dd, J = 10.4, 4.0 Hz, 1H), 2.61 (d, J = 11.3 Hz, 1H), 2.47 – 2.31 (m, 2H), 2.21 – 1.94 (m, 4H), 1.85 (d, J = 11.3 Hz, 1H), 1.40 (t, J = 12.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =138.37, 134.19, 133.35,

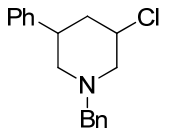
128.86, 128.33, 127.20, 118.27, 118.02, 62.45, 61.84, 61.21, 53.78, 44.21, 42.88, 39.20, 38.24. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{24}ClN$, 290.16; found:290.1664.



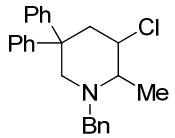
1-benzyl-3-chloropiperidine(2l) Oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ =7.31 – 7.13 (m, 5H), 4.17 – 3.77 (m, 1H), 3.47 (s, 2H), 2.96 (d, *J* = 9.4 Hz, 1H), 2.64 (d, *J* = 11.2 Hz, 1H), 2.07 (dt, *J* = 30.0, 10.1 Hz, 3H), 1.70 (dd, *J* = 8.9, 4.0 Hz, 1H), 1.59 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 136.74, 127.91, 127.15, 126.03, 61.59, 60.15, 55.03, 51.70, 33.75, 23.68. Spectral data was consistent with the known compound.[4]



1-benzyl-3-chloro-2,2-dimethyl-5,5-diphenylpiperidine (2o) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.47 – 6.89 (m, 15H), 3.79 (dd, J = 13.5, 5.0 Hz, 1H), 3.53 (t, J = 11.2 Hz, 1H), 3.41 – 3.35 (m, 2), 3.23 (ddd, J = 13.5, 7.1, 2.1 Hz, 1H), 3.12 (d, J = 14.2 Hz, 1H), 2.24 (dd, J = 13.5, 9.3 Hz, 1H), 1.54(s, 3H), 1.51(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.81, 146.50, 141.29, 128.55, 128.35, 128.33, 128.01, 127.22, 126.85, 126.79, 126.36, 125.97, 74.98, 74.26, 64.12, 62.06, 55.07, 41.04, 30.16, 28.48. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{26}H_{28}ClN$, 390.19; found:390.1976.

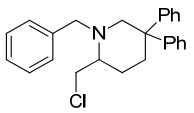


1-benzyl-3-chloro-5-phenylpiperidine (2p) Oil. ^{1}H NMR (400 MHz, $CDCl_3$, major diastereomer) δ = 7.33 – 6.89 (m, 10H), 4.09 – 3.91 (m, 1H), 3.58 – 3.47 (m, 2H), 3.21 – 3.15 (m, 1H), 2.86 (ddd, *J* = 15.7, 10.1, 3.6 Hz, 2H), 2.35 (d, *J* = 12.1 Hz, 1H), 2.13 – 1.96 (m, 2H), 1.75 – 1.64 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 141.38, 136.55, 127.98, 127.52, 127.31, 126.22, 126.11, 125.78, 61.39, 59.97, 58.60, 54.24, 41.72, 41.12. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{20}ClN$, 286.13; found:286.1361.

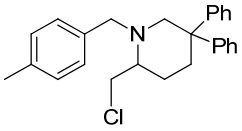


1-benzyl-3-chloro-2-methyl-5,5-diphenylpiperidine (2q) Oil. ^{1}H NMR (400 MHz, $CDCl_3$, major diastereomer) δ = 7.71 – 6.67 (m, 15H), 4.30 (d, J = 13.5 Hz, 1H), 3.80 – 3.71 (m, 1H), 3.64 (dd, J = 12.4, 2.1 Hz, 1H), 3.29 (d, J = 13.5 Hz, 1H), 3.18 – 3.12 (m, 1H), 2.65 – 2.55 (m, 2H), 2.51 (d, J = 12.5 Hz, 1H), 1.49 (d, J = 5.8 Hz, 3H). ^{13}C

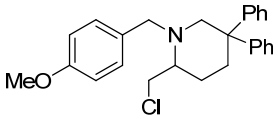
NMR (100 MHz, $CDCl_3$) δ = 147.47, 144.53, 138.62, 129.56, 128.43, 128.36, 128.23, 128.01, 127.28, 126.93, 126.30, 125.84, 64.29, 61.97, 61.59, 58.49, 47.27, 46.25, 18.24. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}ClN$, 376.18; found:376.1820.



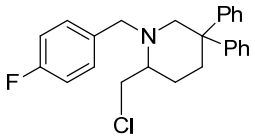
1-benzyl-2-(chloromethyl)-5,5-diphenylpiperidine(2r) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 6.81 (m, 15H), 3.97 (d, *J* = 13.3 Hz, 1H), 3.71 – 3.52 (m, 2H), 3.24 (d, *J* = 13.1 Hz, 2H), 2.65 (s, 1H), 2.51 (d, *J* = 12.4 Hz, 1H), 2.38 (d, *J* = 13.2 Hz, 1H), 2.25 – 2.10 (m, 1H), 1.71 (ddd, J = 27.4, 13.7, 10.5 Hz, 1H), 1.65 – 1.47 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.73, 145.43, 137.47, 128.35, 127.18, 126.98, 126.73, 126.23, 126.11, 124.79, 124.54, 60.11, 59.25, 57.96, 45.18, 43.95, 32.04, 24.57. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}ClN$, 376.1827; found:376.1824.



1-(4-methyl)benzyl-2-(chloromethyl)-5,5-diphenylpiperidine(2s) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ = 7.32 – 7.09 (m, 15H), 3.98 (d, *J* = 13.2 Hz, 1H), 3.76 – 3.58 (m, 2H), 3.37 – 3.14 (m, 2H), 2.70 (t, *J* = 7.6 Hz, 1H), 2.54 (dd, J = 20.6, 7.5 Hz, 1H), 2.44 (d, *J* = 11.0 Hz, 1H), 2.35 (d, *J* = 11.0 Hz, 3H), 2.27 – 2.16 (m, 1H), 1.84 – 1.71 (m, 1H), 1.70 – 1.57 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.80, 146.54, 136.75, 135.30, 129.28, 128.87, 128.23, 127.96, 127.72, 127.16, 125.77, 125.52, 60.98, 60.17, 58.62, 46.18, 44.94, 33.06, 25.55, 21.17. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{26}H_{28}ClN$, 390.1991; found: 390.1910.

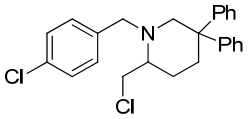


1-(4-methoxyl)benzyl-2-(chloromethyl)-5,5-diphenylpiperidine(2t) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ = 7.31 – 6.89 (m, 15H), 3.98 (d, J = 13.1 Hz, 1H), 3.83 (s, 3H), 3.69 (qd, *J* = 11.2, 4.9 Hz, 2H), 3.27 (dd, *J* = 21.4, 12.8 Hz, 2H), 2.71 (t, *J* = 7.4 Hz, 1H), 2.55 (d, *J* = 12.4 Hz, 1H), 2.44 (d, *J* = 13.3 Hz, 1H), 2.30 – 2.18 (m, 1H), 1.83 – 1.72 (m, 1H), 1.71 – 1.64 (m, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ = 158.85, 147.86, 146.60, 130.51, 128.28, 128.05, 127.80, 127.22, 125.85, 125.60, 113.54, 61.01, 59.97, 58.17, 55.25, 46.18, 44.93, 33.07, 25.57. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{26}H_{28}ClNO$, 406.1859; found: 406.1993.

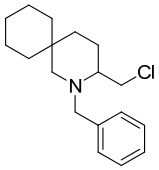


1-(4-fluoro)benzyl-2-(chloromethyl)-5,5-diphenylpiperidine(2u) Oil. ^1H NMR (400 MHz, $CDCl_3$) δ = 7.43 – 6.96 (m, 15H), 4.00 (d, *J* = 13.3 Hz, 1H), 3.68 (d, *J* = 4.7 Hz, 2H), 3.27 (t, *J* = 14.1 Hz, 2H), 2.71 (d, *J* = 4.2 Hz, 1H), 2.56 (d, *J* = 12.3 Hz,

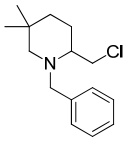
1H), 2.45 (d, *J* = 13.1 Hz, 1H), 2.33 – 2.15 (m, 1H), 1.71 (dd, *J* = 21.3, 12.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.73, 146.34, 130.90, 130.83, 128.13, 128.05, 127.79, 127.07, 125.89, 125.63, 115.13, 114.92, 61.14, 60.17, 58.10, 46.20, 44.94, 33.09, 25.67. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{25}ClFN$, 394.1736; found: 394.1660.



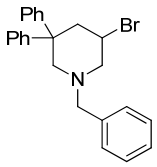
1-(4-chloro)benzyl-2-(chloromethyl)-5,5-diphenylpiperidine(2v) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 – 7.09 (m, 15H), 4.03 – 3.96 (m, 1H), 3.67 (s, 2H), 3.28 (t, *J* = 14.5 Hz, 2H), 2.71 (s, 1H), 2.56 (d, *J* = 10.2 Hz, 1H), 2.46 (d, *J* = 10.2 Hz, 1H), 2.25 (t, *J* = 11.8 Hz, 1H), 1.82 – 1.65 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.63, 146.27, 137.09, 132.95, 130.65, 128.36, 128.12, 128.06, 127.80, 127.04, 125.91, 125.65, 77.33, 77.01, 76.69, 61.19, 60.37, 58.22, 46.20, 44.97, 33.09, 25.69. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{25}Cl_2N$, 410.1440; found: 410.1364.



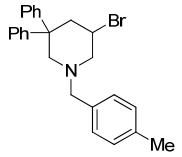
2-benzyl-3-(chloromethyl)-2-azaspiro[5.5]undecane(2w) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ =7.63 – 7.07 (m, 5H), 4.07 (d, *J* = 13.5 Hz, 1H), 3.84 (dd, *J* = 9.8, 4.9 Hz, 1H), 3.73 (dd, *J* = 11.3, 2.6 Hz, 1H), 3.30 (d, *J* = 13.5 Hz, 1H), 2.71 – 2.64 (m, 1H), 2.60 (d, *J* = 11.6 Hz, 1H), 2.02 – 1.93 (m, 1H), 1.91 (d, *J* = 11.6 Hz, 1H), 1.76 (ddd, *J* = 13.5, 9.5, 4.1 Hz, 1H), 1.69 – 1.62 (m, 1H), 1.58 – 1.19 (m, 11H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =139.95, 128.54, 128.21, 126.85, 62.13, 60.48, 58.26, 45.47, 36.95, 33.88, 33.67, 32.97, 26.95, 25.21, 21.72, 21.68. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{26}ClN$, 292.1827; found:292.1830.



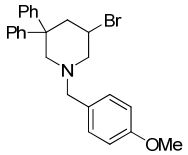
1-benzyl-2-(chloromethyl)-5,5-dimethylpiperidine(2x) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ =7.53 – 6.90 (m, 5H), 4.01 (d, *J* = 13.6 Hz, 1H), 3.78 (dd, *J* = 11.3, 6.5 Hz, 1H), 3.70 (dd, *J* = 11.3, 2.6 Hz, 1H), 3.29 (d, *J* = 13.6 Hz, 1H), 2.65 – 2.54 (m, 1H), 2.34 (t, *J* = 15.0 Hz, 1H), 1.97 – 1.90 (m, 1H), 1.86 (d, *J* = 11.5 Hz, 1H), 1.73 (ddd, *J* = 13.6, 9.7, 4.3 Hz, 1H), 1.44 (dd, *J* = 11.9, 5.8 Hz, 1H), 1.25 (dd, *J* = 13.9, 3.7 Hz, 1H), 0.97 (s, 3H), 0.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.72, 127.39, 127.11, 125.70, 61.62, 60.36, 57.13, 44.12, 34.77, 29.54, 27.22, 24.78, 24.67. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{22}ClN$, 252.1514; found:252.1515.



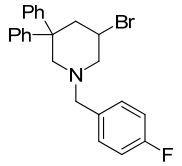
1-benzyl-5-bromo-3,3-diphenylpiperidine (3a) Oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.26– 6.98 (m, 15H), 3.87 (ddd, *J* = 15.3, 8.0, 4.0 Hz, 1H), 3.49 (q, *J* = 13.2 Hz, 3H), 3.18 (dd, *J* = 10.4, 3.8 Hz, 1H), 3.01 (d, *J* = 12.5 Hz, 1H), 2.40 (t, *J* = 12.4 Hz, 1H), 2.30-2.18 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=147.36, 144.80, 137.63, 129.37, 128.71, 128.45, 128.19, 127.54, 126.48, 126.06, 62.57, 62.00, 49.23, 46.80, 45.52. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{24}BrN$, 406.1165; found:406.1161.



5-bromo-1-(4-methylbenzyl)-3,3-diphenylpiperidine (3b) White solid. M.p. = 135-137°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ =7.44 – 6.89 (m, 15H), 4.09 – 3.79 (m, 1H), 3.54 (d, *J* = 12.4 Hz, 1H), 3.49 (s, 2H), 3.20 (dd, *J* = 10.5, 4.3 Hz, 1H), 3.08 – 2.97 (m, 1H), 2.42 (t, *J* = 12.4 Hz, 1H), 2.28 (s, 3H), 2.29 – 2.21 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =147.41, 144.87, 137.07, 134.50, 129.34, 129.11, 128.75, 128.44, 128.16, 126.48, 126.46, 126.02, 62.29, 61.98, 61.92, 49.22, 46.85, 45.59, 21.32. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}BrN$, 420.1321; found:420.1226.

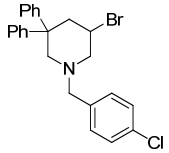


5-bromo-1-(4-methoxybenzyl)-3,3-diphenylpiperidine (3c) White solid. M.p. = 88-91°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ=7.41 – 6.92 (m, 12H), 6.78 (d, *J* = 7.7 Hz, 2H), 3.87 (t, *J* = 11.3 Hz, 1H), 3.71 (s, 3H), 3.51 (d, *J* = 12.1 Hz, 1H), 3.44 (s, 2H), 3.21 – 3.16 (m, 1H), 3.01 (d, *J* = 12.3 Hz, 1H), 2.40 (t, *J* = 12.4 Hz, 1H), 2.30 – 2.14 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=159.00, 147.37, 144.80, 130.52, 129.27, 128.68, 128.41, 128.14, 127.80, 126.44, 125.99, 113.73, 61.97, 61.88, 61.74, 55.34, 49.17, 46.81, 45.59. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}BrNO$, 436.1271; found:436.1263.

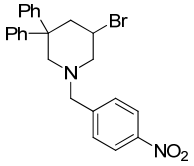


5-bromo-1-(4-fluorobenzyl)-3,3-diphenylpiperidine (3d) White solid. M.p. = 104-107°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46 – 6.62 (m, 14H), 3.83 (dtt, *J* = 18.5, 10.8, 3.8 Hz, 1H), 3.48 (t, *J* = 9.1 Hz, 1H), 3.44 (s, 2H), 3.14 (dd, *J* = 10.4, 4.1 Hz, 1H), 3.01 (d, *J* = 12.6 Hz, 1H), 2.39 (t, *J* = 12.4 Hz, 1H), 2.30 – 2.19 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.50, 161.06, 147.27, 144.69, 133.37, 133.35, 130.88, 130.80, 128.60, 128.48, 128.23, 126.53, 126.45, 126.11, 115.39, 115.17, 61.98, 61.90,

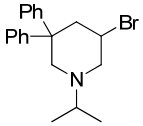
61.71, 49.21, 46.75, 45.32. ^{19}F NMR (376 MHz, $CDCl_3$) δ =-114.95. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{23}BrFN$, 424.1071; found:424.1078.



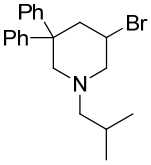
5-bromo-1-(4-chlorobenzyl)-3,3-diphenylpiperidine (3e) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ =7.33 – 6.90 (m, 14H), 4.01 – 3.79 (m, 1H), 3.57 – 3.33 (m, 3H), 3.13 (dd, *J* = 10.4, 4.1 Hz, 1H), 3.01 (d, *J* = 12.6 Hz, 1H), 2.38 (t, *J* = 12.4 Hz, 1H), 2.29 – 2.20 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.06, 143.49, 135.02, 132.08, 129.43, 127.45, 127.40, 127.32, 127.07, 125.36, 125.27, 124.96, 60.84, 60.61, 48.07, 45.57, 44.00. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{23}BrClN$, 440.0775; found:440.0776.



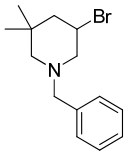
5-bromo-1-(4-nitrobenzyl)-3,3-diphenylpiperidine (3f) White solid. M.p. = 149-151°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ =8.10 (d, *J* = 8.6 Hz, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 7.29 – 7.06(m, 8H), 7.04(d, *J* = 7.3 Hz, 2H), 3.92 (tt, *J* = 11.9, 4.0 Hz, 1H), 3.66 – 3.50 (m, 3H), 3.12 (dd, *J* = 10.2, 4.6 Hz, 1H), 3.06 (d, *J* = 12.8 Hz, 1H), 2.43 – 2.29 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=146.34, 145.82, 144.40, 143.27, 128.60, 127.39, 127.26, 127.20, 125.49, 125.23, 125.14, 122.59, 61.15, 60.80, 60.51, 48.11, 45.34, 43.56. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{23}BrN_2O_2$, 451.1016; found:451.1010.



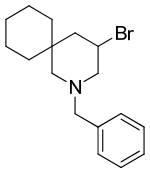
5-bromo-1-isopropyl-3,3-diphenylpiperidine (3g). Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.61 – 7.24 (m, 10H),4.04 – 3.89 (m, 1H), 3.69 (d, *J*=12.1, 1H), 3.32 (d, *J*=6.3, 1H), 3.18 – 3.07 (m, 2H), 2.63 – 2.40 (m, 3H), 1.25 (d, *J*=6.6, 3H), 1.22 (d, *J*=6.6, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =146.7, 144.3, 127.5, 127.3, 127.0, 125.4, 125.3, 124.8, 56.7, 56.1, 53.6, 53.5, 47.0, 45.4, 16.8, 16.5. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{24}BrN$, 358.1165; found:358.1167.



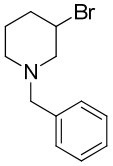
5-bromo-1-isobutyl-3,3-diphenylpiperidine (3h) White solid. M.p. = 83-85°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 7.31 (m, 2H), 7.21 – 7.04 (m, 8H), 3.93 – 3.82 (m, 1H), 3.50 (d, *J* = 12.2 Hz, 1H), 3.23 – 3.18 (m, 1H), 3.00 – 2.92 (m, 1H), 2.41 (t, *J* = 12.3 Hz, 1H), 2.21 – 2.14 (m, 2H), 2.09 (dd, *J* = 5.1, 2.5 Hz, 2H), 1.86 – 1.70 (m, 1H), 0.86 (d, *J* = 4.4 Hz, 3H), 0.84 (d, *J* = 4.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =147.55, 145.17, 128.82, 128.38, 128.02, 126.46, 126.38, 125.91, 66.68, 63.58, 62.16, 49.26, 46.98, 45.86, 25.66, 21.19, 20.91. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{26}BrN$, 372.1321; found:372.1313.



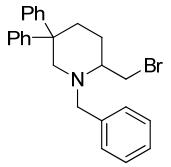
1-benzyl-5-bromo-3,3-dimethylpiperidine(3i) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = =7.29 – 7.16 (m, 5H), 4.16 (ddd, *J* = 15.7, 10.0, 4.5 Hz, 1H), 3.48 (d, *J* = 13.4 Hz, 1H), 3.35 (d, *J* = 13.4 Hz, 1H), 3.15 (d, *J* =6.6 Hz, 1H), 2.35 (d, *J* = 11.1 Hz, 1H), 2.07 (t, *J* = 10.8 Hz, 1H), 1.99 – 1.94 (m, 1H),1.70 (d, *J* = 11.0 Hz, 1H), 1.46 (t, *J* = 12.5 Hz, 1H), 0.99 (s, 3H), 0.80 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=137.44, 127.61, 127.21, 126.01, 63.41, 61.37, 61.20, 48.23, 45.35, 33.36, 28.23, 23.83. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{20}BrN$, 282.0852; found:282.0854.



2-benzyl-4-bromo-2-azaspiro[5.5]undecane (3j). Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ=7.23 – 7.15 (m, 5H), 4.17 (tt, *J* = 12.0, 4.4 Hz, 1H), 3.48 (d, *J* = 13.4 Hz, 1H), 3.35 (dd, *J* = 13.4, 5.4 Hz, 1H), 3.15 (dd, *J* = 10.5, 4.3 Hz, 1H), 2.65 (d, *J* = 11.3 Hz, 1H), 2.19 (dd, *J* = 11.0, 1.8 Hz, 1H), 2.12 (t, *J* = 10.8 Hz, 1H), 1.57 (d, *J* = 11.3 Hz, 2H), 1.36 – 1.08 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ=137.56, 127.54, 127.18, 125.98, 62.11, 61.28, 45.29, 37.31, 36.07, 31.65, 25.56, 20.50, 20.47. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{24}BrN$, 322.1165; found:322.1168.



1-benzyl-3-bromopiperidine(3k) ^{1}H NMR (400 MHz, $CDCl_3$) δ=7.46 – 7.02 (m, 5H), 4.05 (ddd, *J* = 13.7, 9.6, 4.0 Hz, 1H), 3.46 (s, 2H), 3.01 (d, *J* = 9.7 Hz, 1H), 2.66 (d, *J* = 11.2 Hz, 1H), 2.28 (t, *J* = 10.4 Hz, 1H), 2.22 – 2.11 (m, 1H), 2.04 (t, *J* = 9.9 Hz, 1H), 1.77 – 1.50 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ =136.84, 127.95, 127.23, 126.10, 61.60, 60.76, 51.77, 47.34, 34.63, 24.85. Spectral data was consistent with the known compound.[4]



1-benzyl-2-(bromomethyl)-5,5-diphenylpiperidine (3l) Oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.13 (m, 15H), 4.09 (d, *J* = 13.1 Hz, 1H), 3.69 – 3.58 (m, 2H), 3.35 (dd, *J* = 17.1, 12.9 Hz, 2H), 2.75 (s, 1H), 2.63 (d, *J* = 12.4 Hz, 1H), 2.50 (d, *J* = 13.0 Hz, 1H), 2.39 – 2.18 (m, 1H), 1.86 – 1.67 (m, 2H).) δ=147.79, 146.52, 138.41, 129.54, 129.25, 128.31, 128.27, 128.06, 127.79, 127.20, 125.88, 125.63, 60.46, 60.08,

58.95, 46.31, 34.66, 33.10, 26.32. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{26}BrN$, 420.1321; found:420.1225.

5. Reference

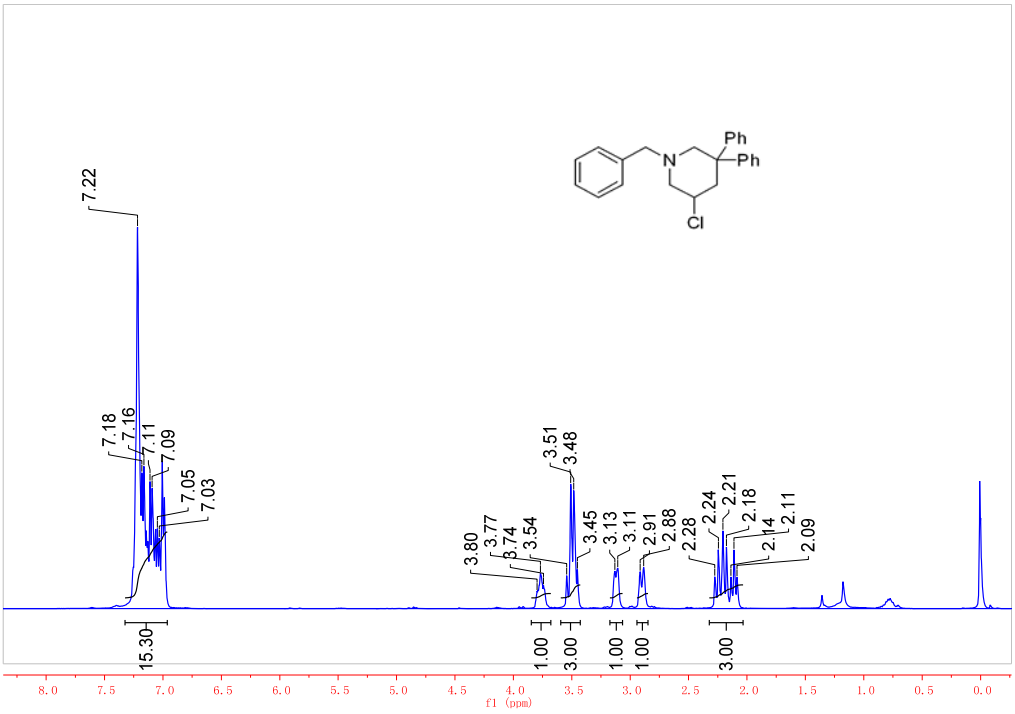
[1] G.-Q. Liu, W. Li, Y.-M. Li, *Adv. Synth. Catal.* **2013**, *355*, 395-402.

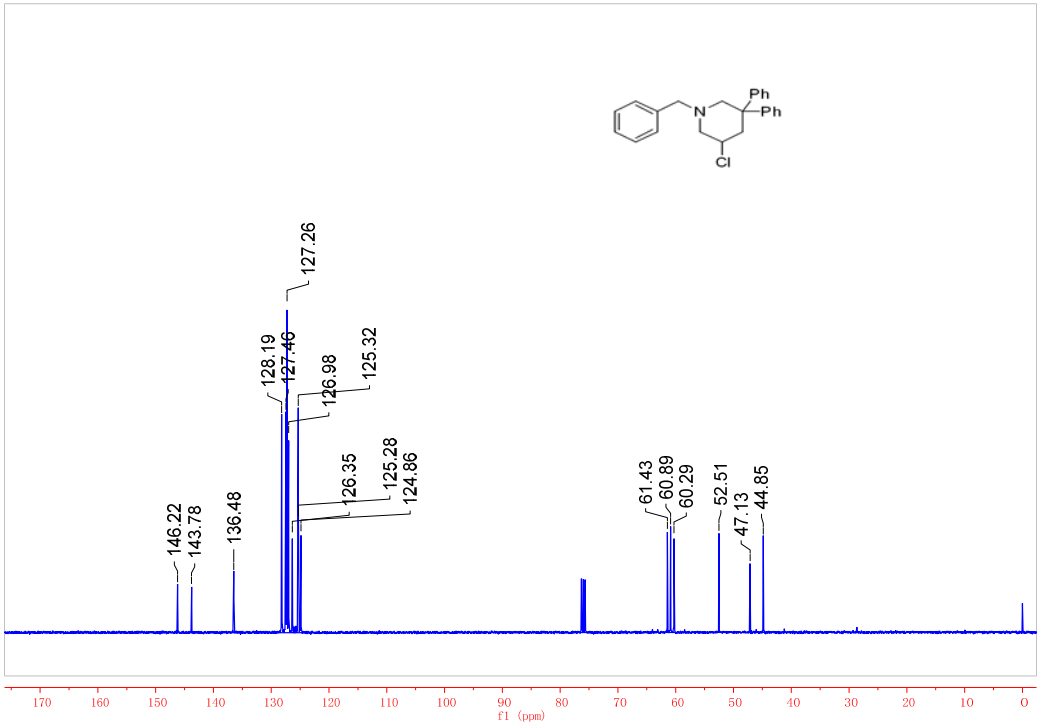
[2] (a)K. D. Hesp, M. Stradiotto, *Organic Letters* **2009**, *11*, 1449-1452; (b)R. Zhang, Q. Xu, L.-Y. Mei, S.-K. Li, M. Shi, *Tetrahedron* **2012**, *68*, 3172-3178.

[3] M. C. Wood, D. C. Leitch, C. S. Yeung, J. A. Kozak, L. L. Schafer, *Angew. Chem. Int. Ed* **2007**, *46*, 354-358.

[4] K. Hayashi, E. Kujime, H. Katayama, S. Sano, M. Shiro, Y. Nagao, *Chem. Pharm. Bull.* **2009**, *57*, 1142-1146.

6. Copies of NMR Spectra

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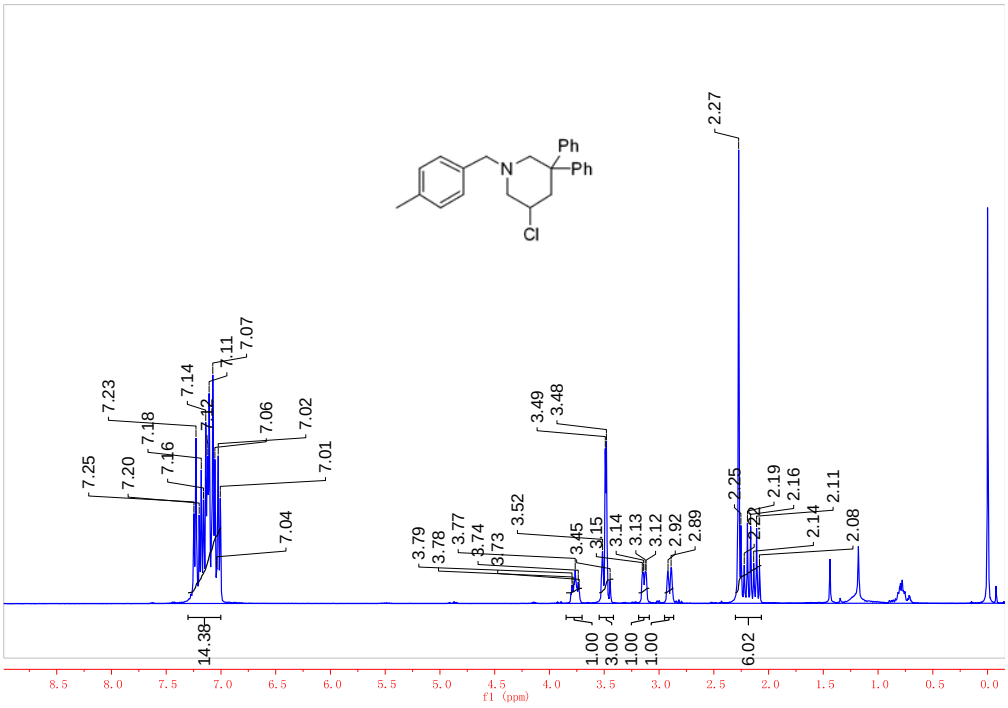
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3.48
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14.38
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1.00
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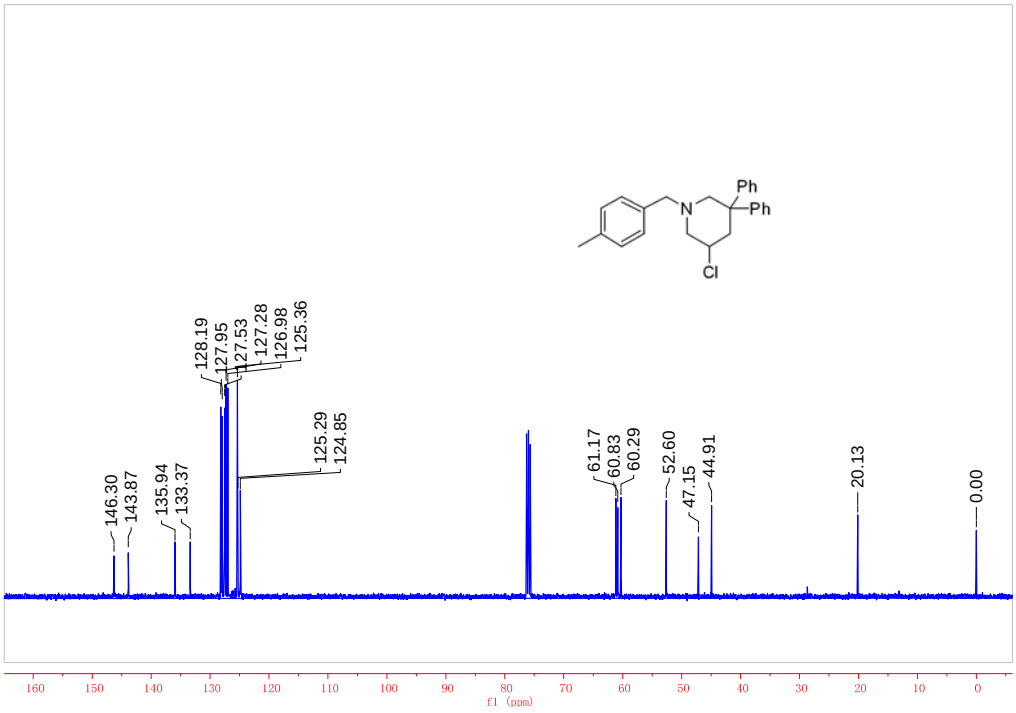
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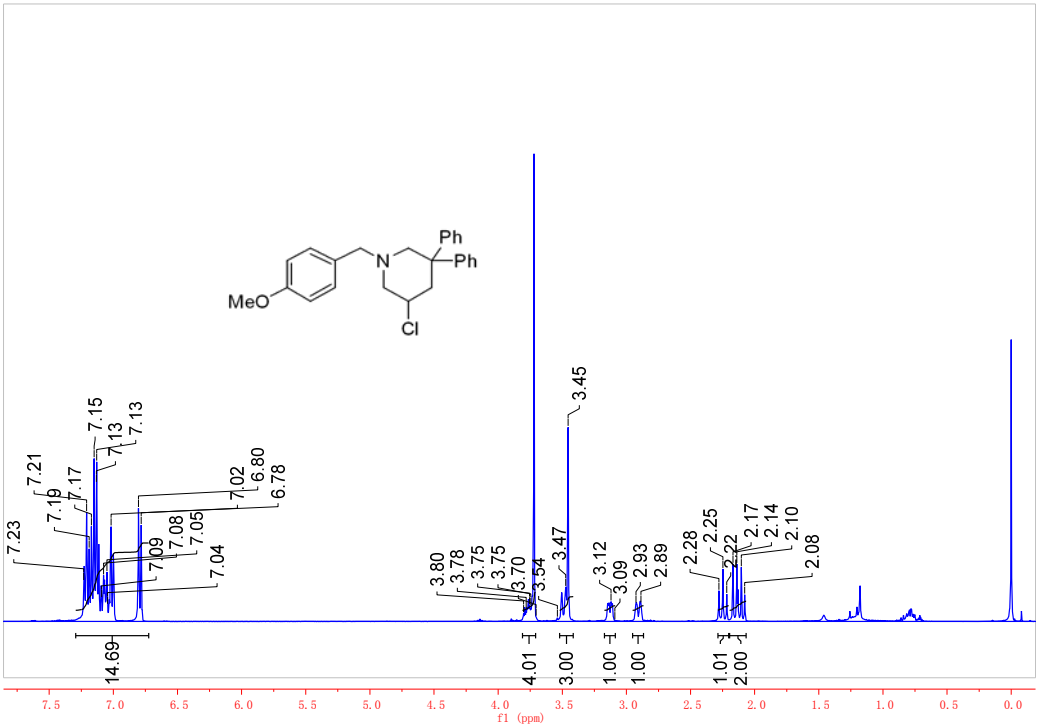
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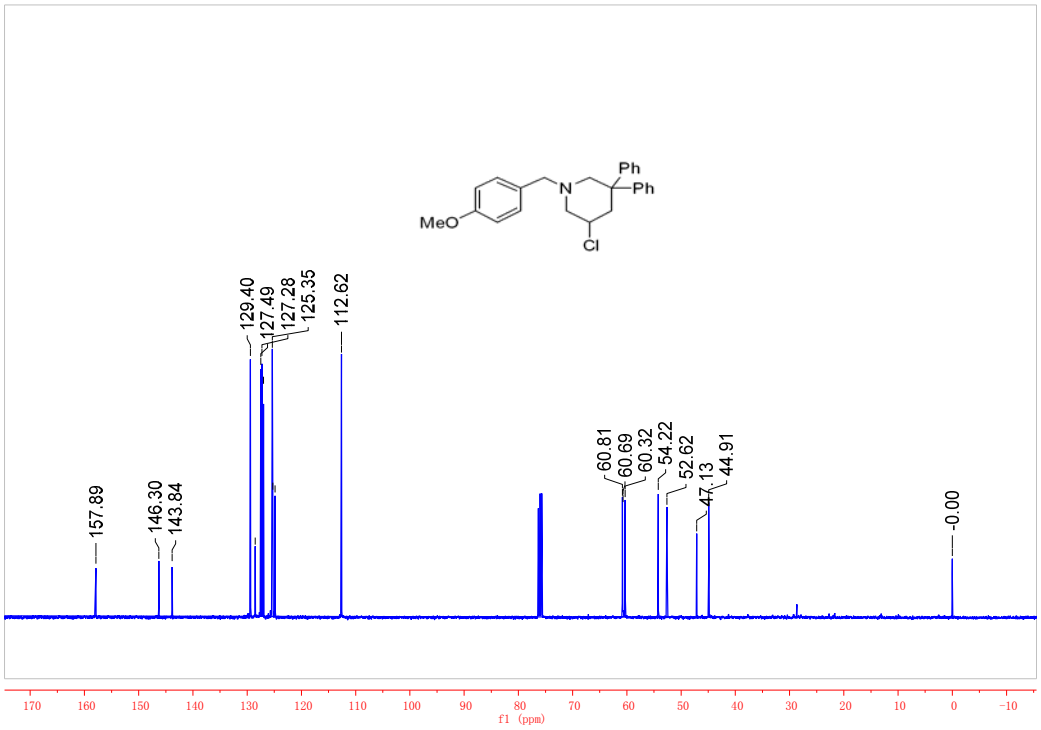
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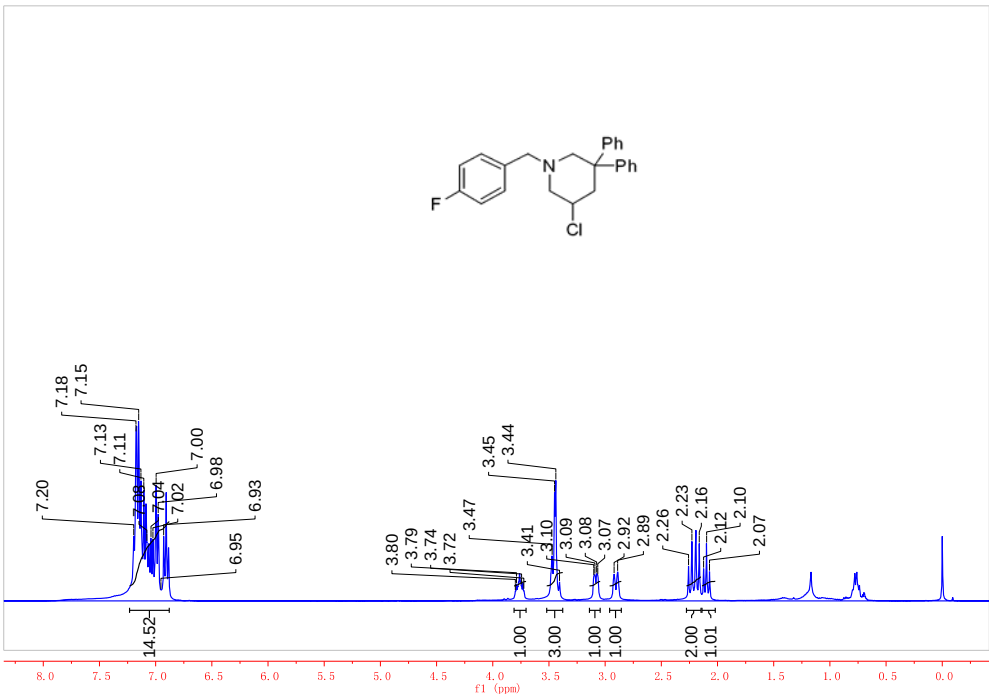
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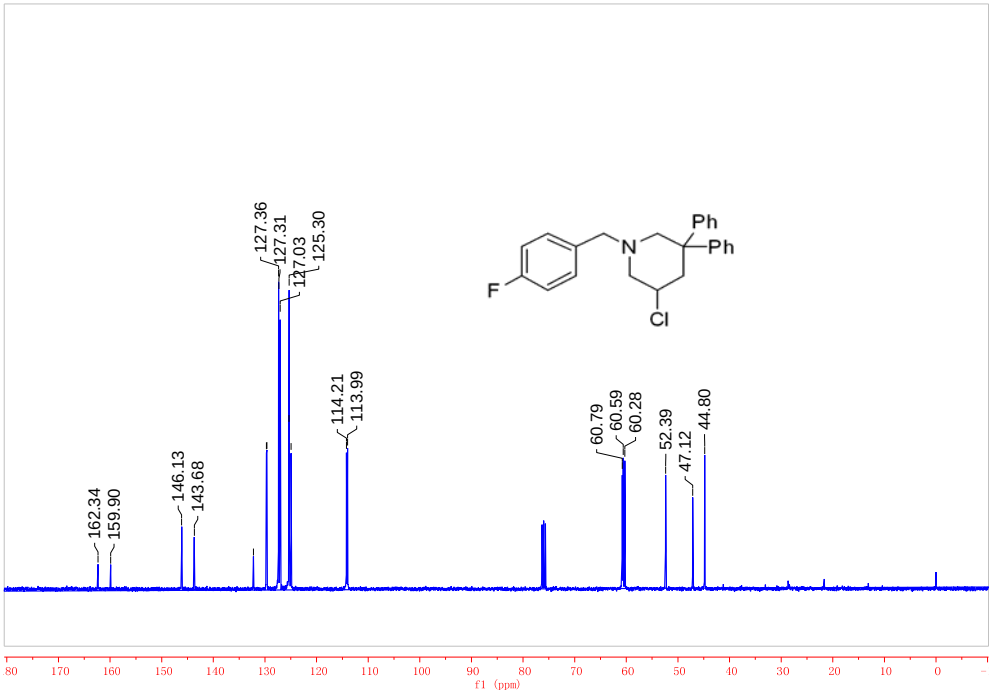
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Cl
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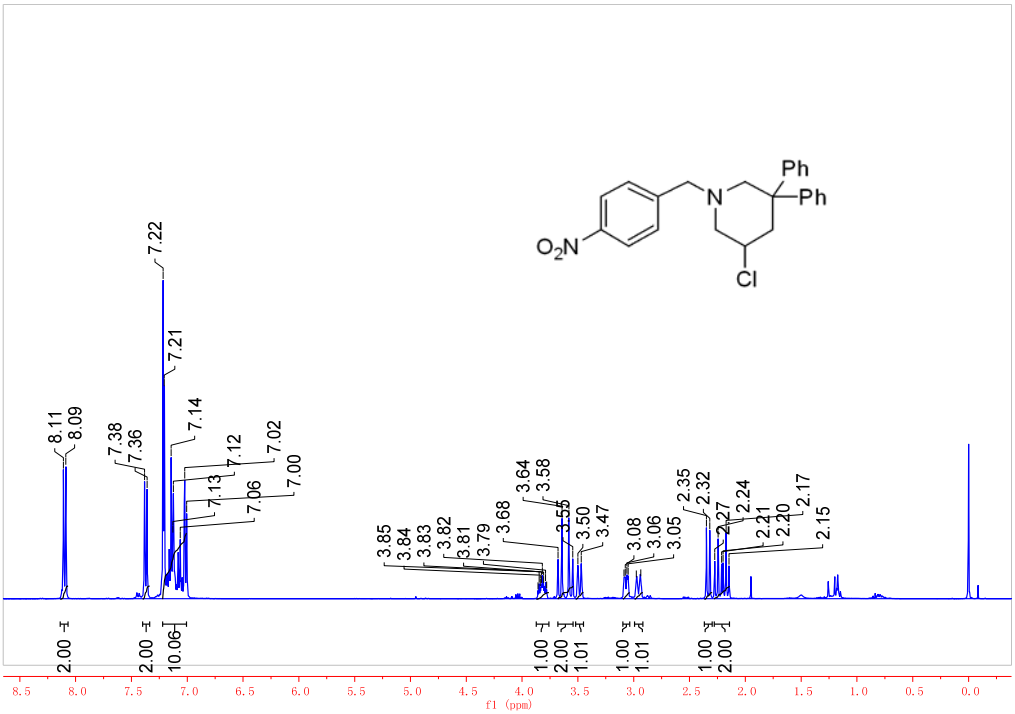
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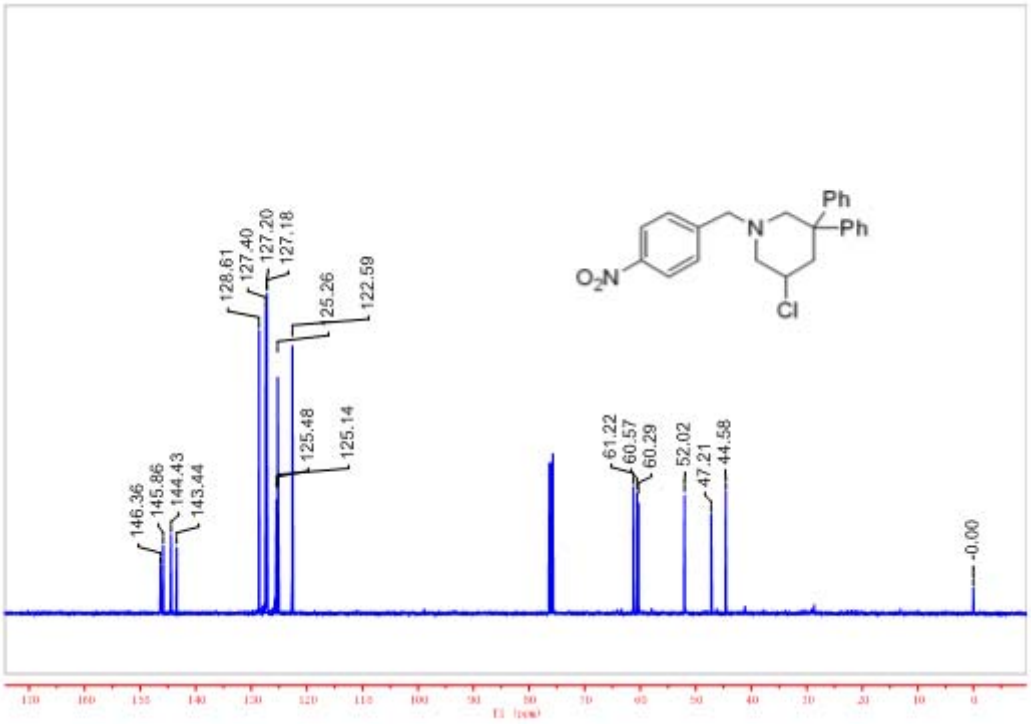
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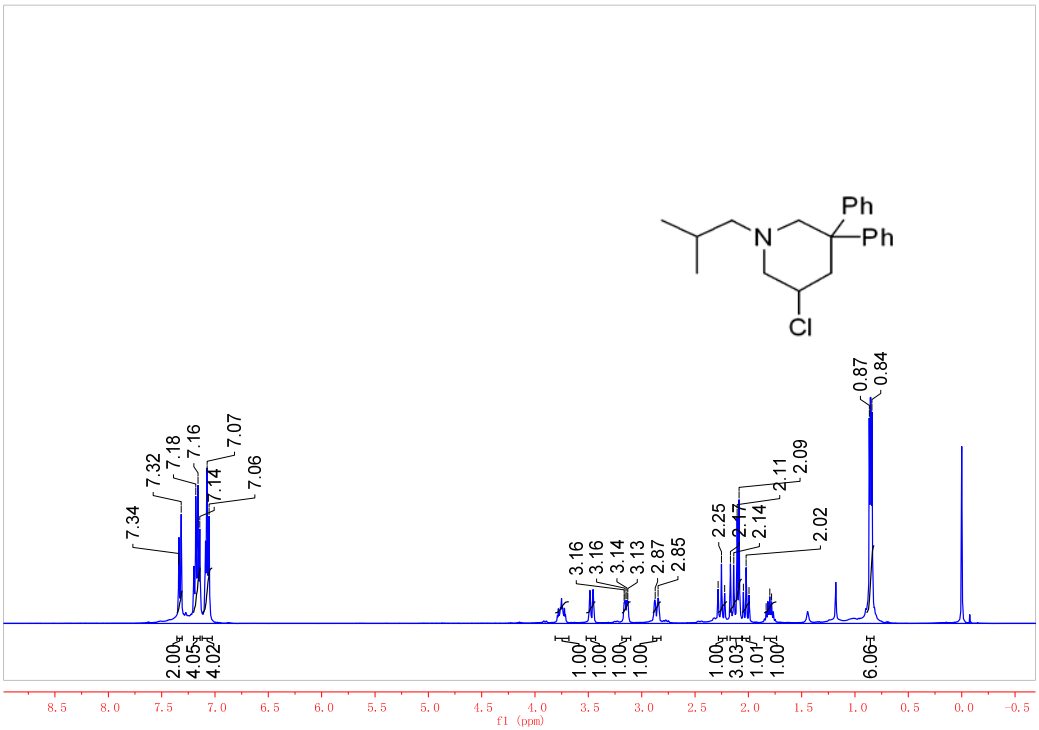
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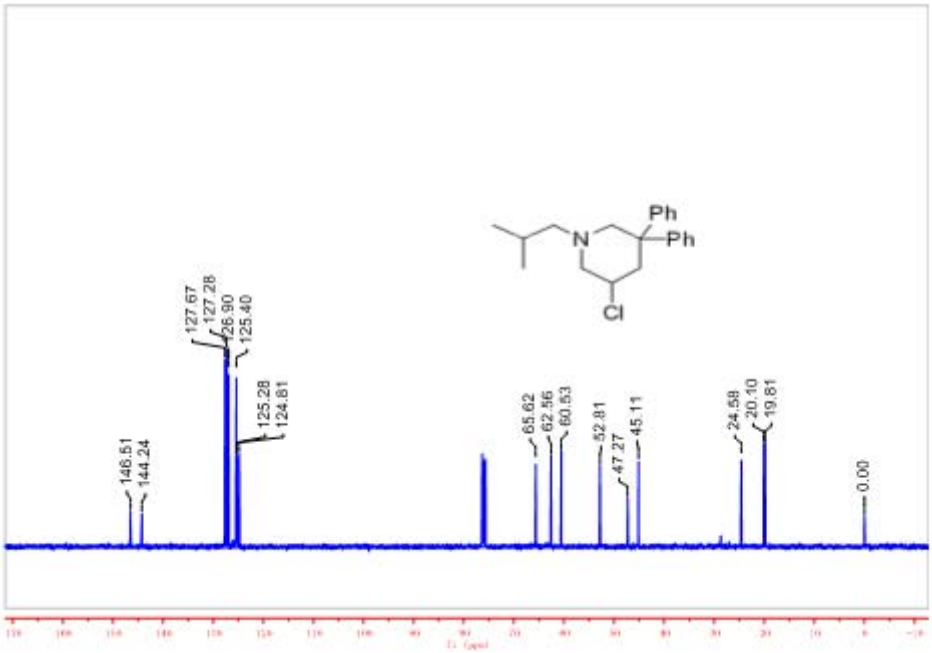
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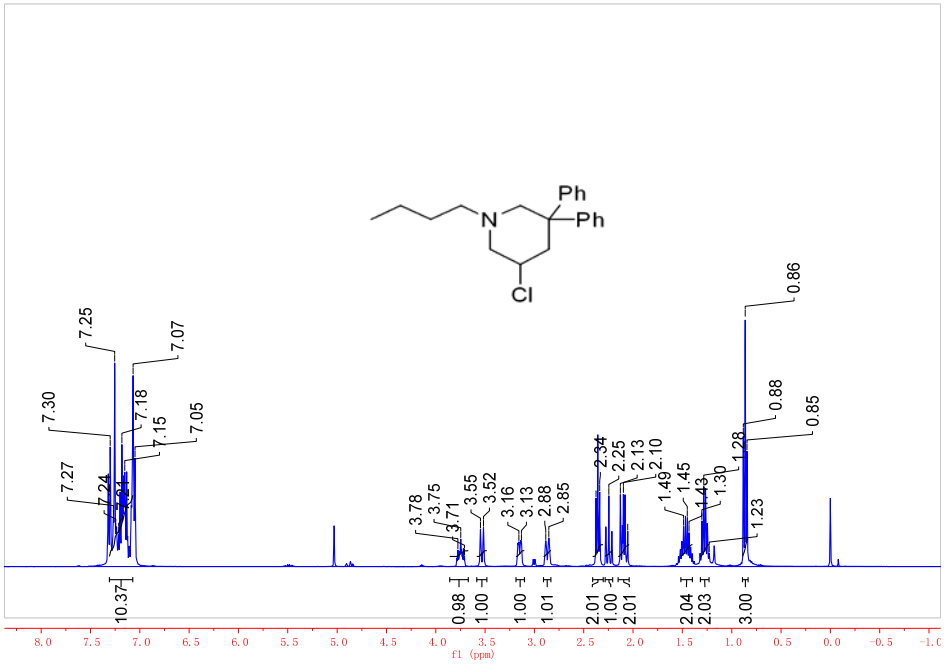
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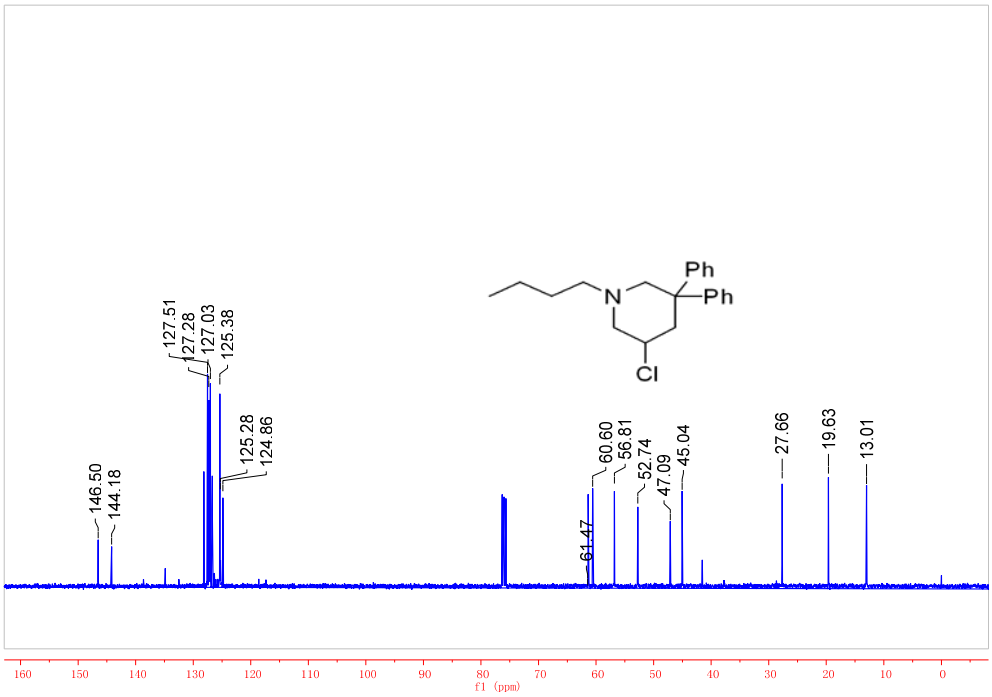
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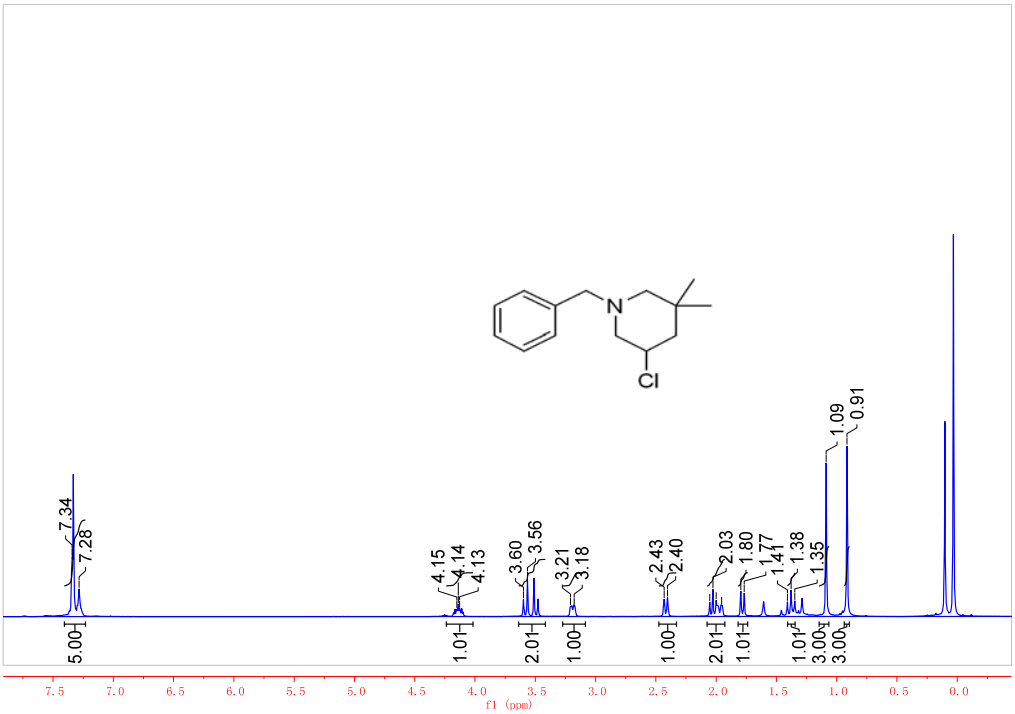
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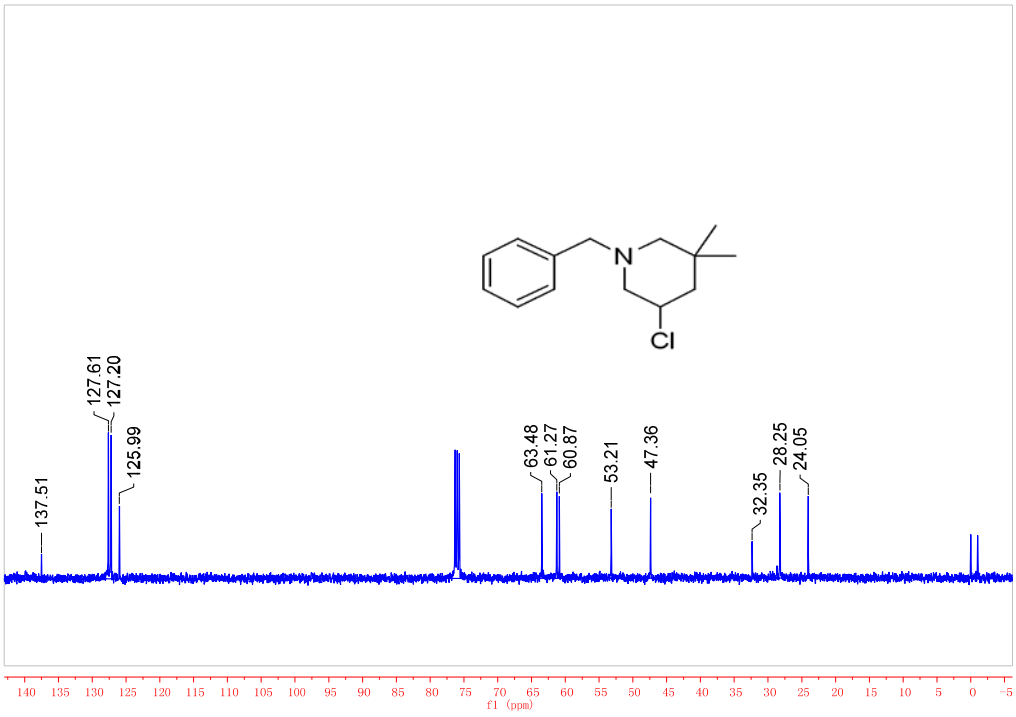
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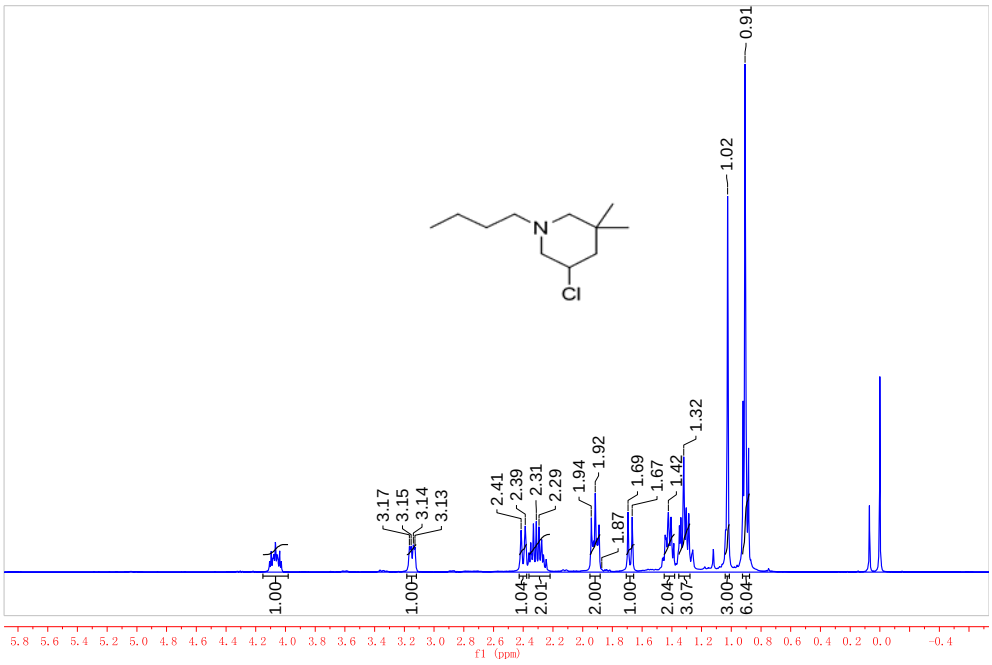
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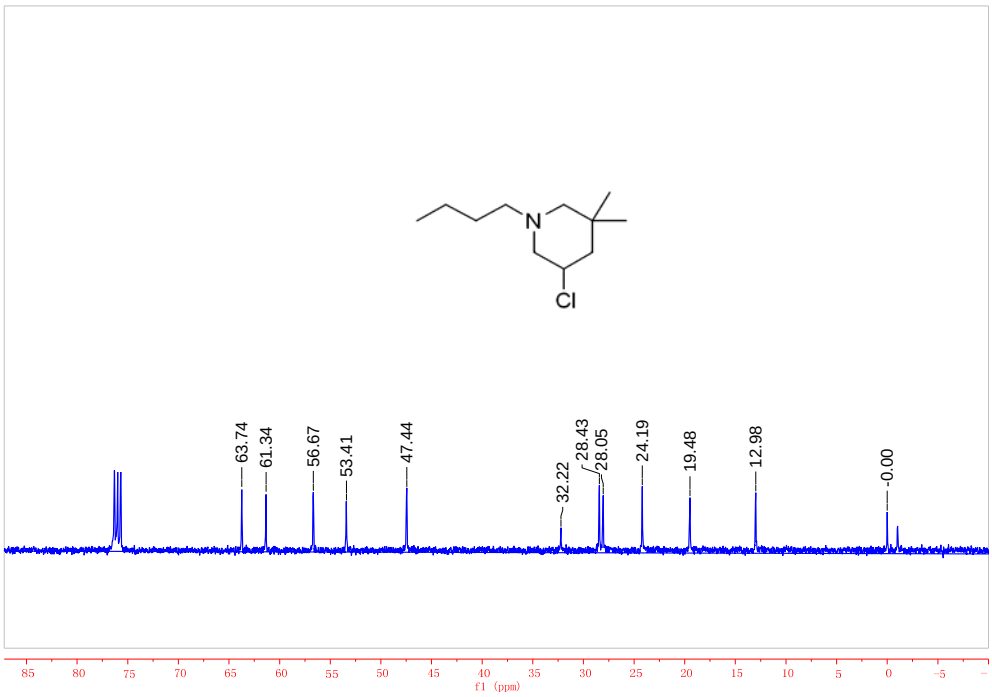
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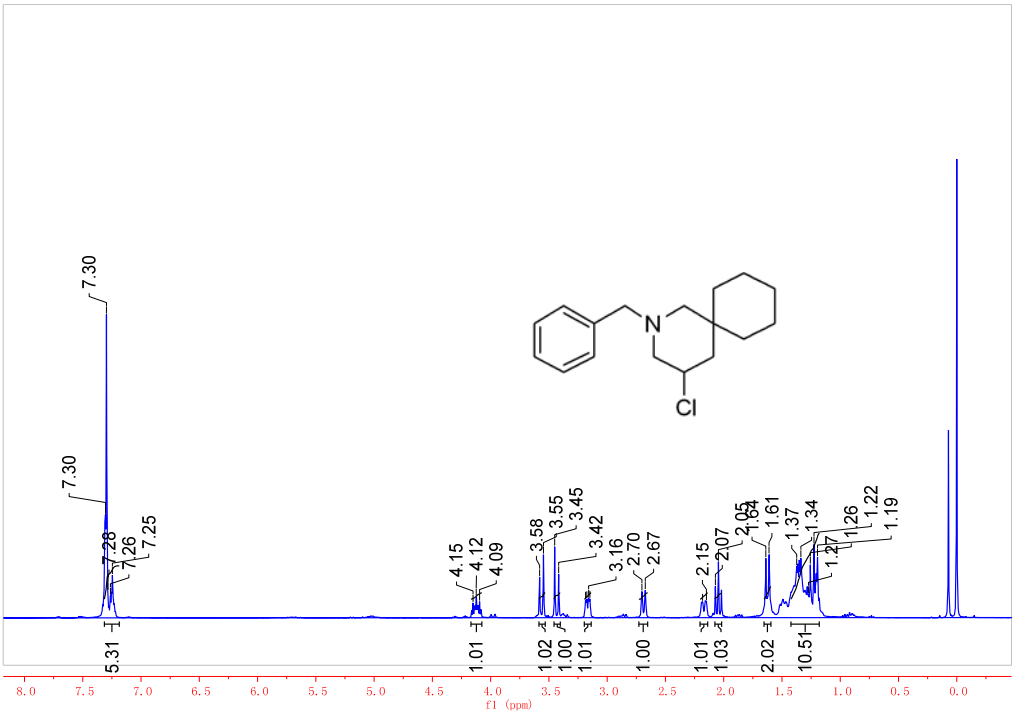
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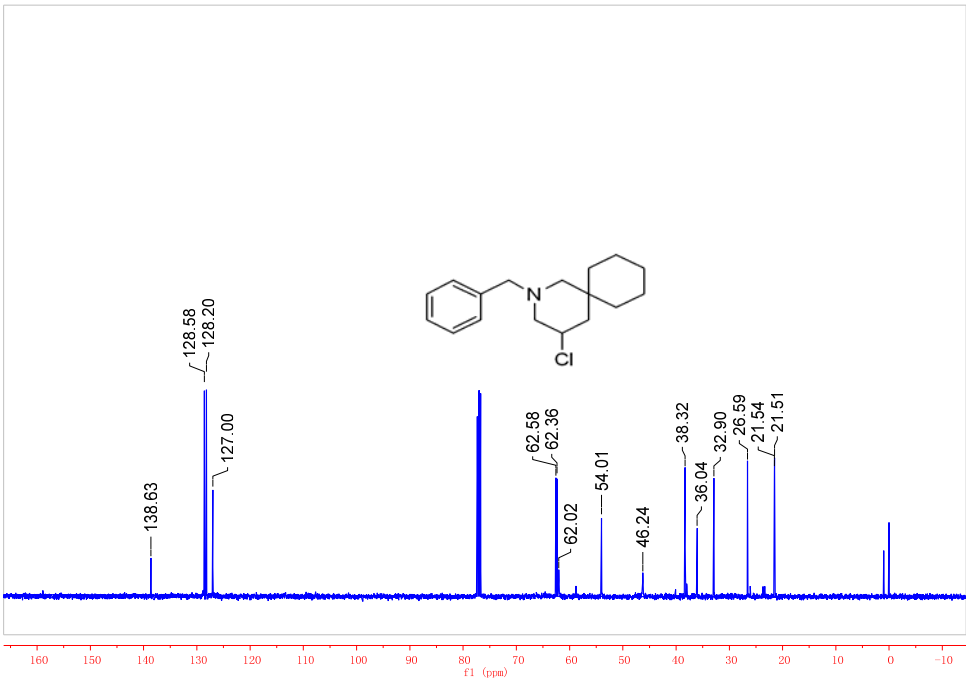
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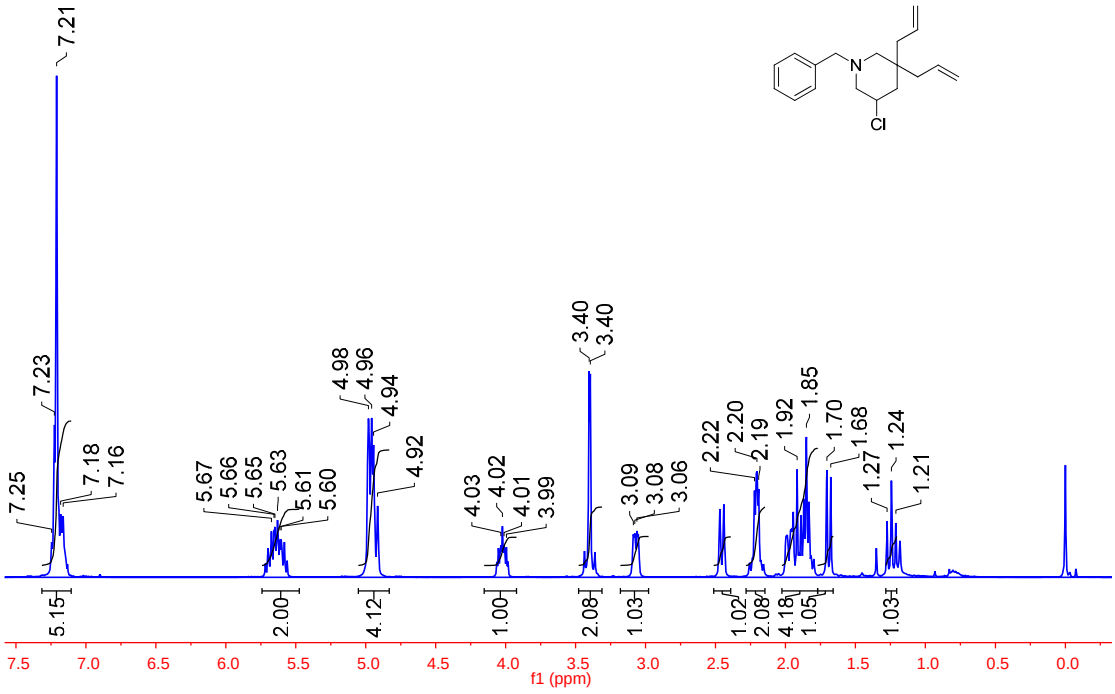
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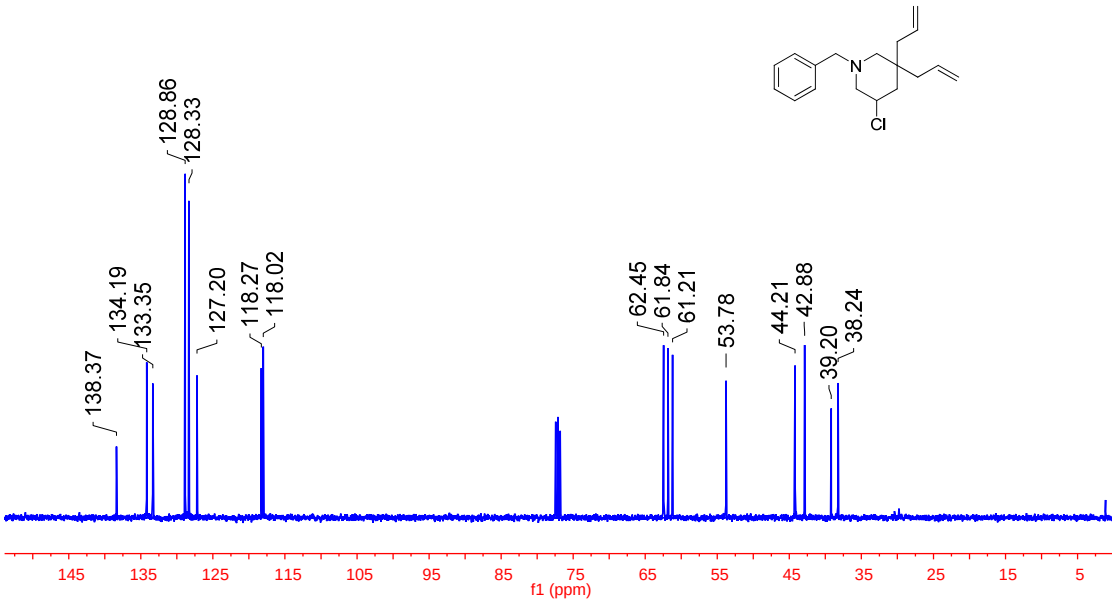
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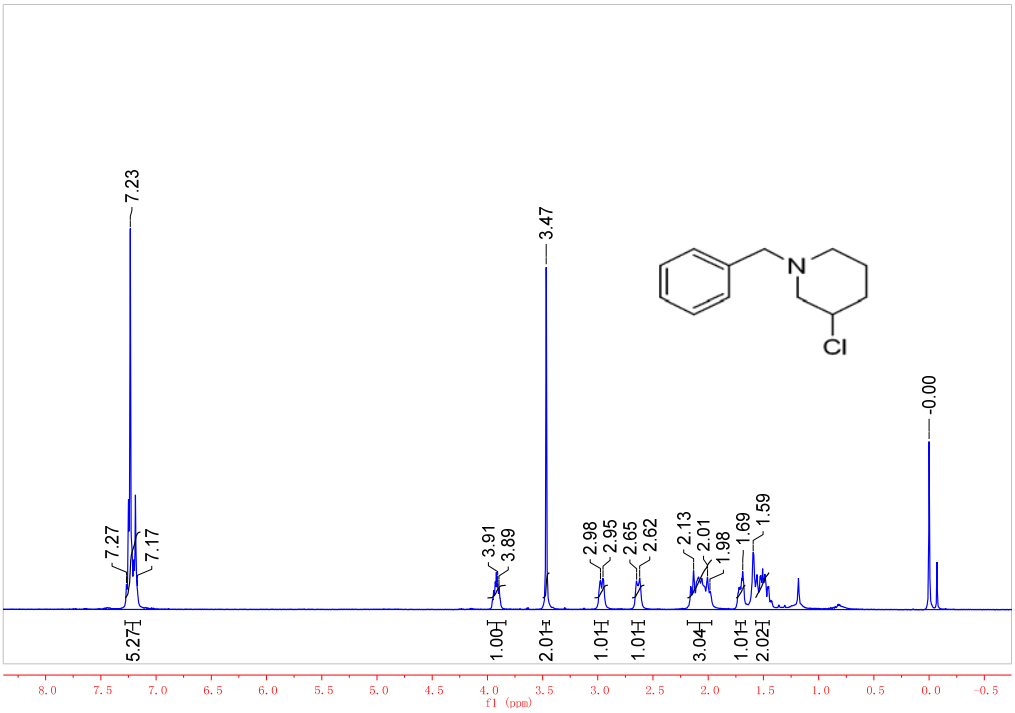
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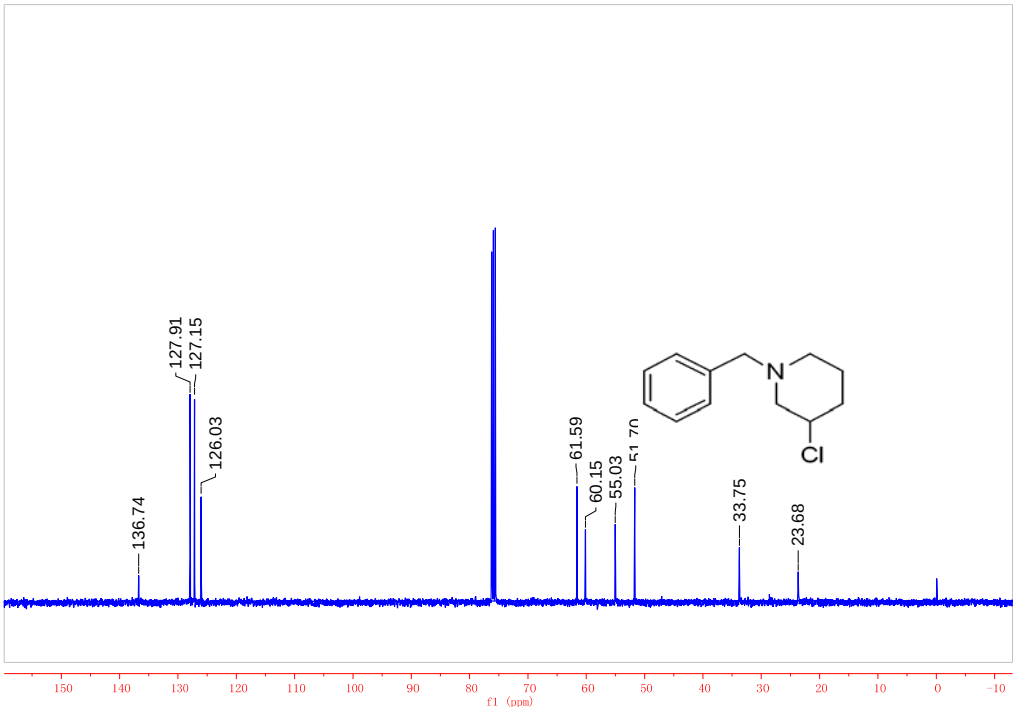
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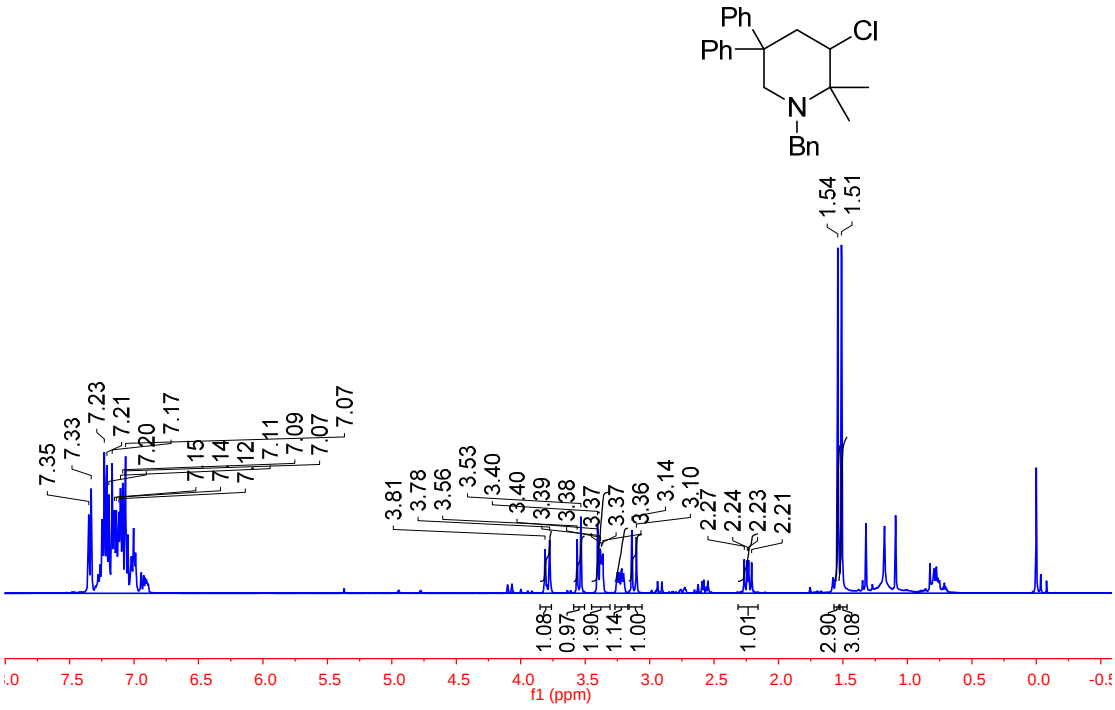
Cl
f1 (ppm)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Ph
Cl
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Bn
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7.23
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7.20
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7.15
7.14
7.12
7.11
7.09
7.07
7.07
3.81
3.78
3.56
3.53
3.40
3.39
3.38
3.37
3.37
3.36
3.14
3.10
2.27
2.24
2.23
2.21
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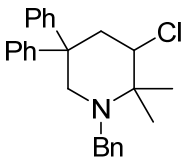
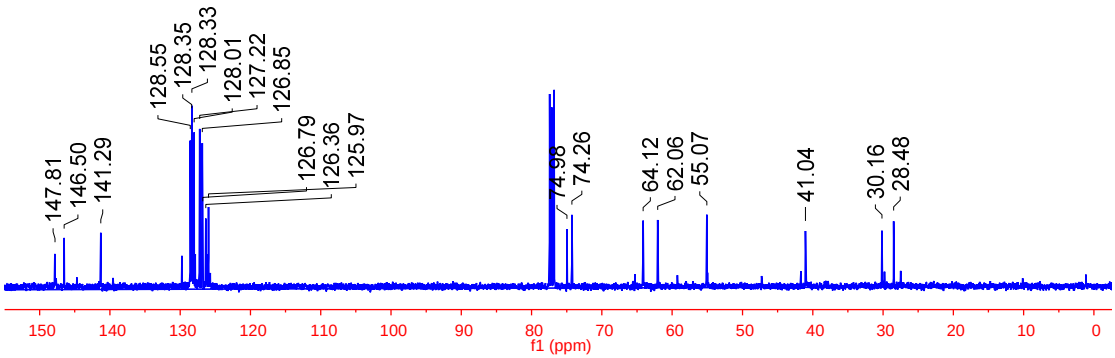
1.90
1.14
1.00
1.01
2.90
3.08
f1 (ppm)


Ph
Ph
Cl
N
Bn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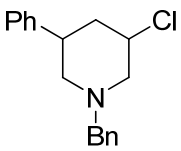
128.35
128.33
128.01
127.22
126.85
126.79
126.36
125.97
74.96
74.26
64.12
62.06
55.07
41.04
30.16
28.48
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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Cl
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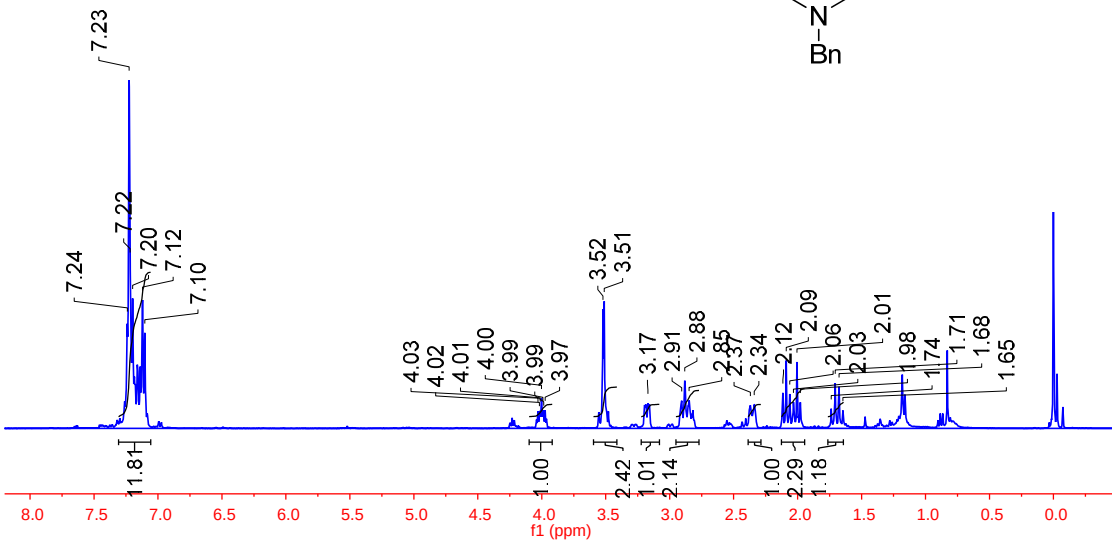
Bn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7.10
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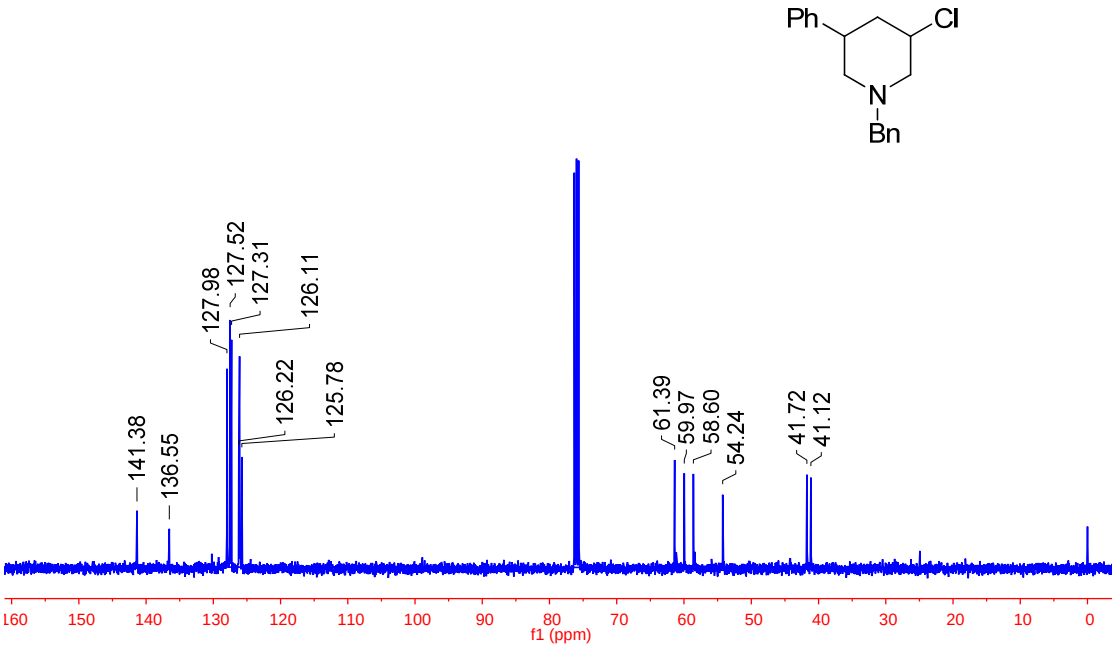
4.02
4.01
4.00
3.99
3.99
3.97
3.52
3.51
3.17
2.91
2.88
2.85
2.37
2.34
2.12
2.09
2.06
2.03
2.01
1.98
1.74
1.71
1.68
1.65
11.81
1.00
2.42
1.01
2.14
1.00
2.29
1.18
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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Cl
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Bn
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127.31
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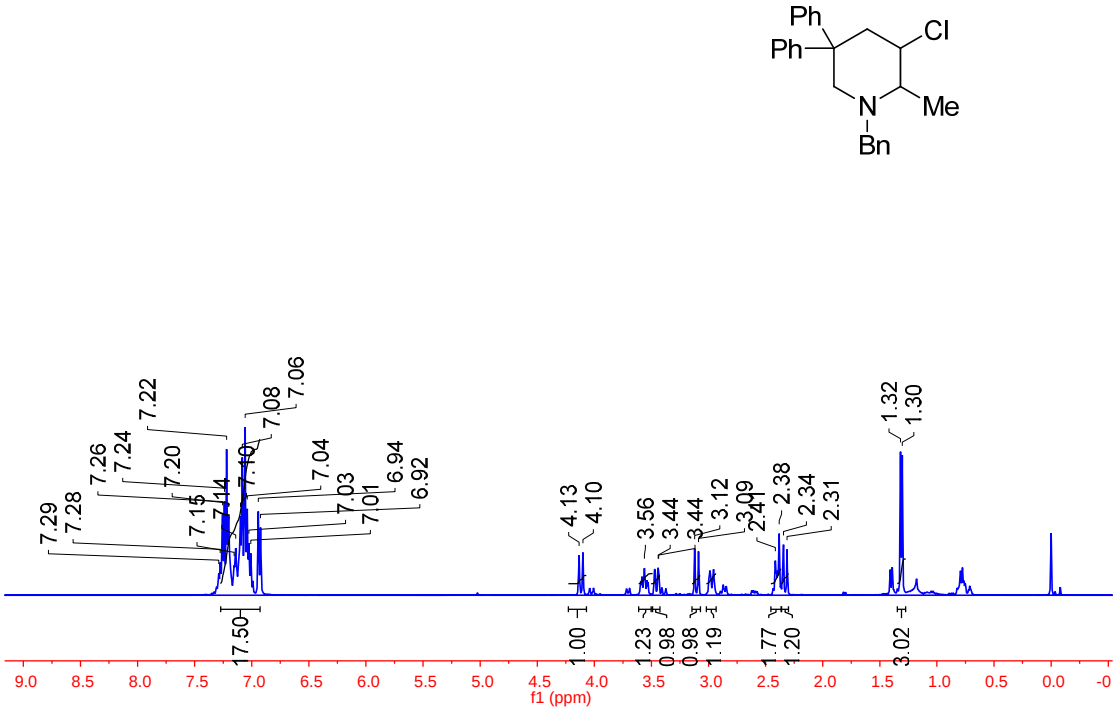
126.22
125.78
61.39
59.97
58.60
54.24
41.72
41.12
f1 (ppm)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Ph
Cl
N
Me
Bn
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7.20
7.15
7.14
7.10
7.08
7.06
7.04
7.03
7.01
6.94
6.92
4.13
4.10
3.56
3.44
3.12
3.09
2.41
2.38
2.34
2.31
1.32
1.30
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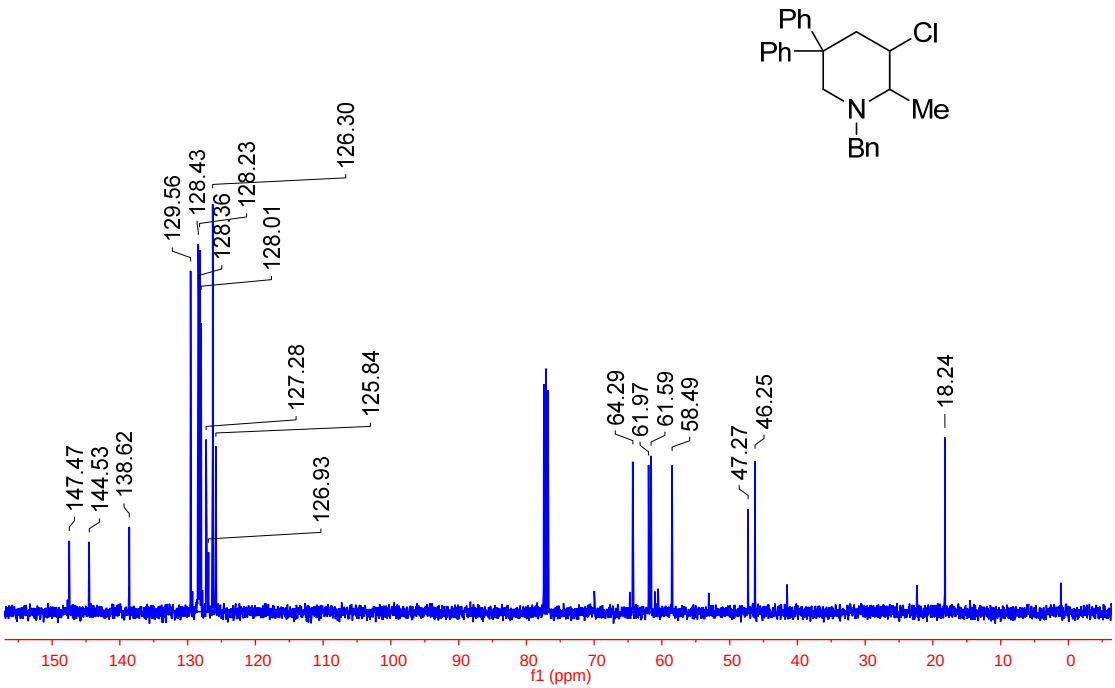
1.23
0.98
0.98
1.19
1.77
1.20
3.02
f1 (ppm)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Me
Bn
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128.23
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126.30
127.28
125.84
126.93
64.29
61.97
61.59
58.49
47.27
46.25
18.24
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140
130
120
110
100
90
80
70
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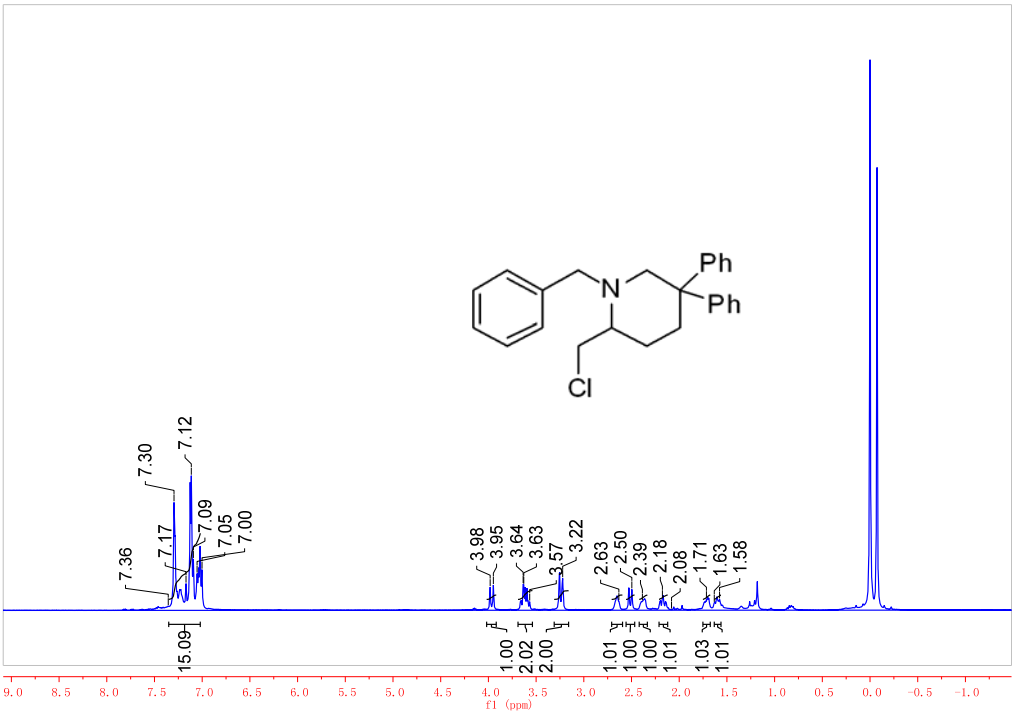
50
40
30
20
10
0
f1 (ppm)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Ph
Cl
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7.12
7.09
7.05
7.00
3.98
3.95
3.64
3.63
3.57
3.22
2.63
2.50
2.39
2.18
2.08
1.71
1.63
1.58
15.09
1.00
2.02
2.00
1.01
1.00
1.00
1.01
1.03
1.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
2.0
1.5
1.0
0.5
0.0
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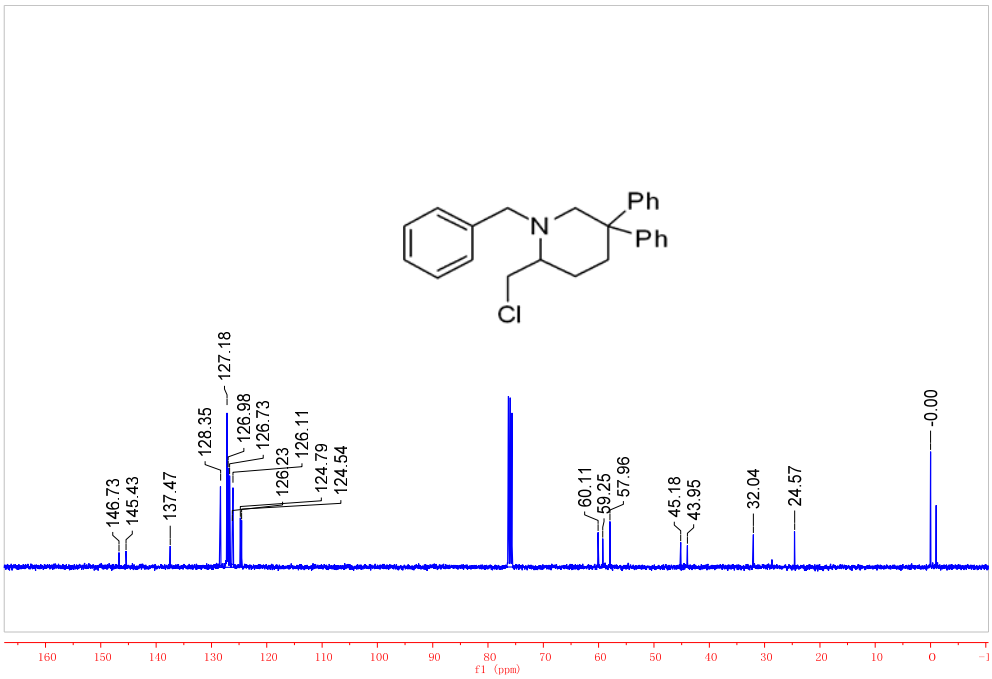
-1.0
f1 (ppm)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Ph
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Cl
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126.98
126.73
126.23
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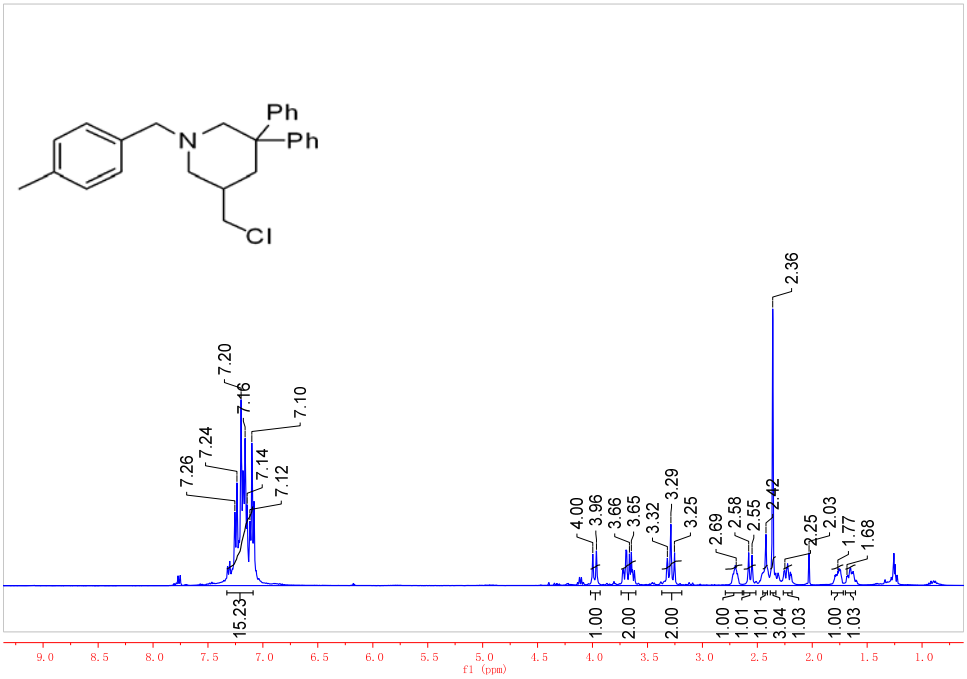
59.25
57.96
45.18
43.95
32.04
24.57
-0.00
f1 (ppm)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Ph
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Cl
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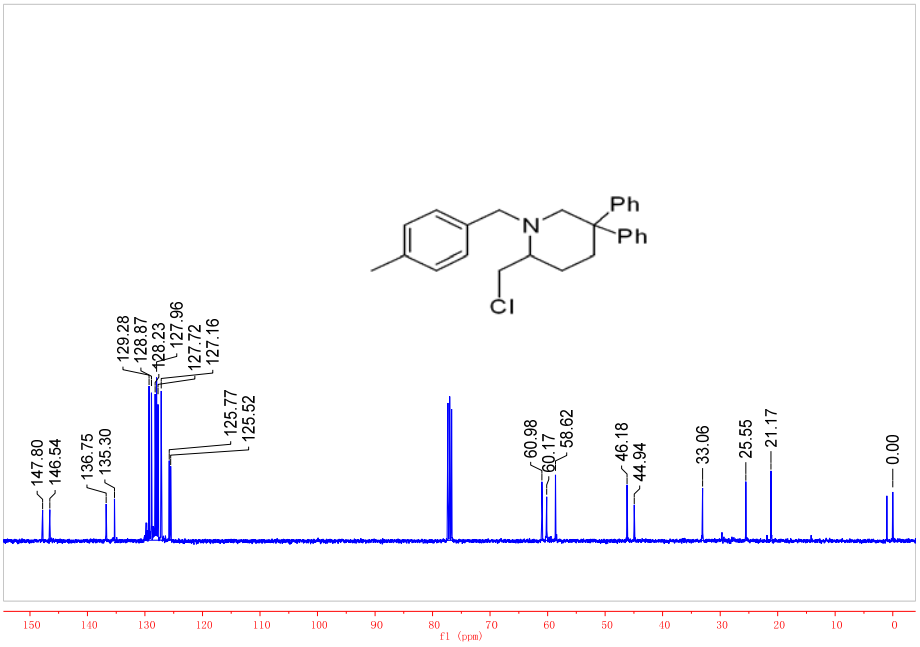
7.20
7.16
7.14
7.12
7.10
4.00
3.96
3.66
3.65
3.32
3.29
3.25
2.69
2.58
2.55
2.42
2.36
2.25
2.03
1.77
1.68
15.23
1.00
2.00
2.00
1.00
1.01
1.01
3.04
1.03
1.00
1.03
f1 (ppm)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Ph
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Cl
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127.96
127.72
127.16
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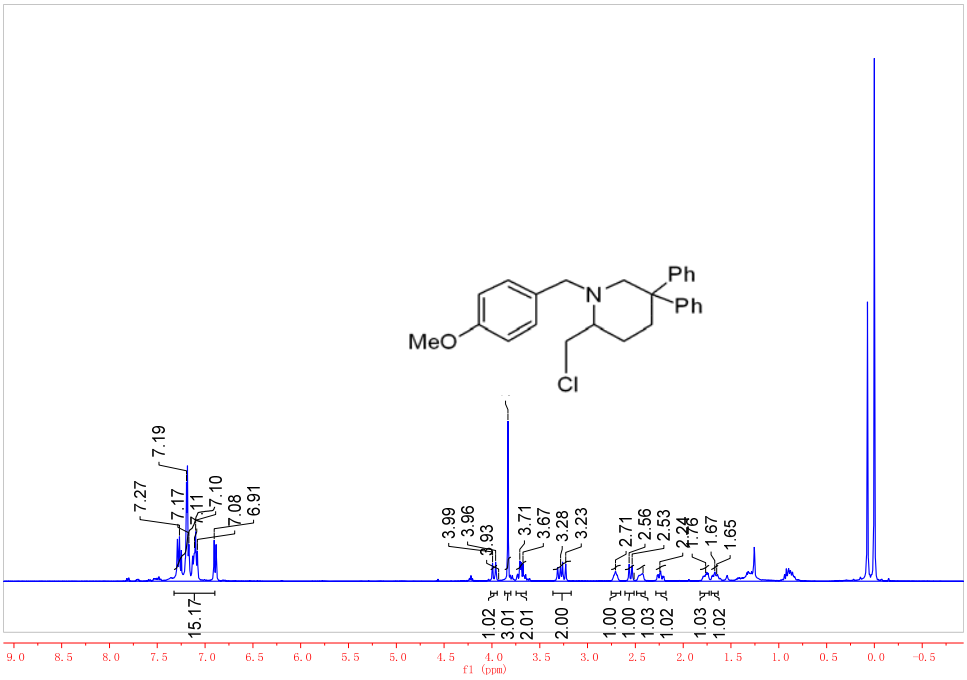
60.17
58.62
46.18
44.94
33.06
25.55
21.17
0.00
f1 (ppm)


MeO
Ph
Ph
N
Cl
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7.17
7.11
7.10
7.08
6.91
3.99
3.96
3.93
3.71
3.67
3.28
3.23
2.71
2.56
2.53
2.24
1.67
1.65
15.17
1.02
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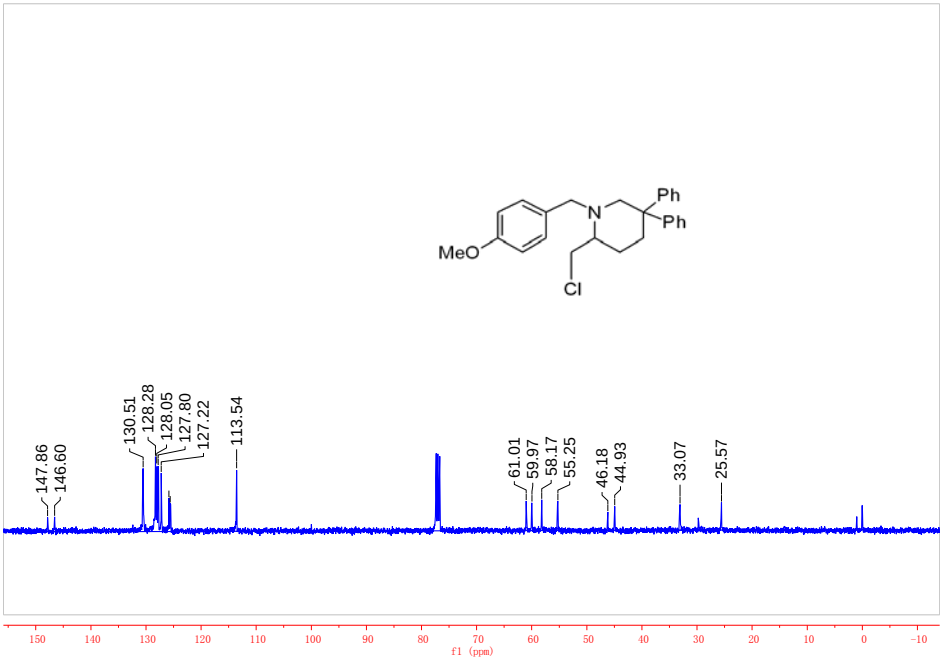
2.01
2.00
1.00
1.00
1.03
1.02
1.03
1.02
f1 (ppm)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MeO
Cl
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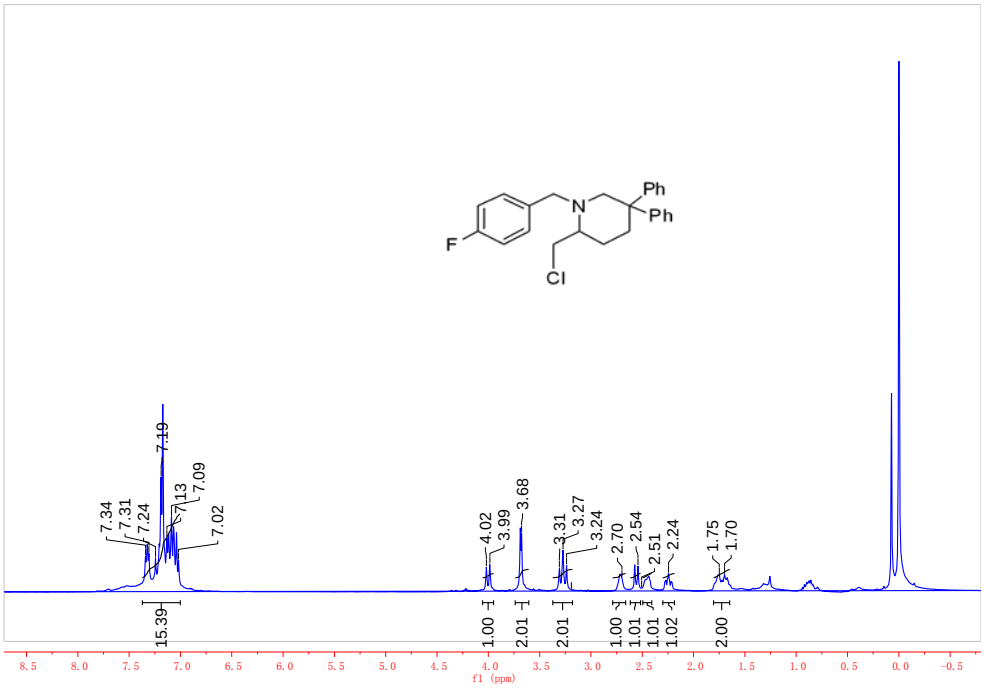
46.18
44.93
33.07
25.57
f1 (ppm)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Ph
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F
Cl
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7.24
7.19
7.13
7.09
7.02
4.02
3.99
3.68
3.31
3.27
3.24
2.70
2.54
2.51
2.24
1.75
1.70
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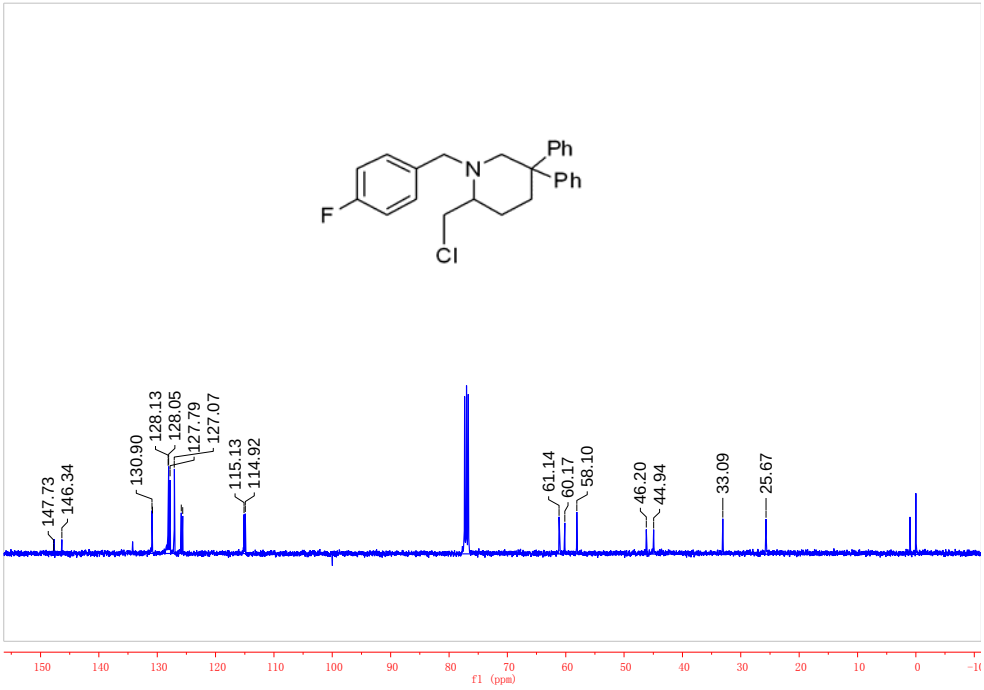
1.00
2.01
2.01
1.00
1.01
1.01
1.02
2.00
f1 (ppm)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Ph
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F
Cl
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60.17
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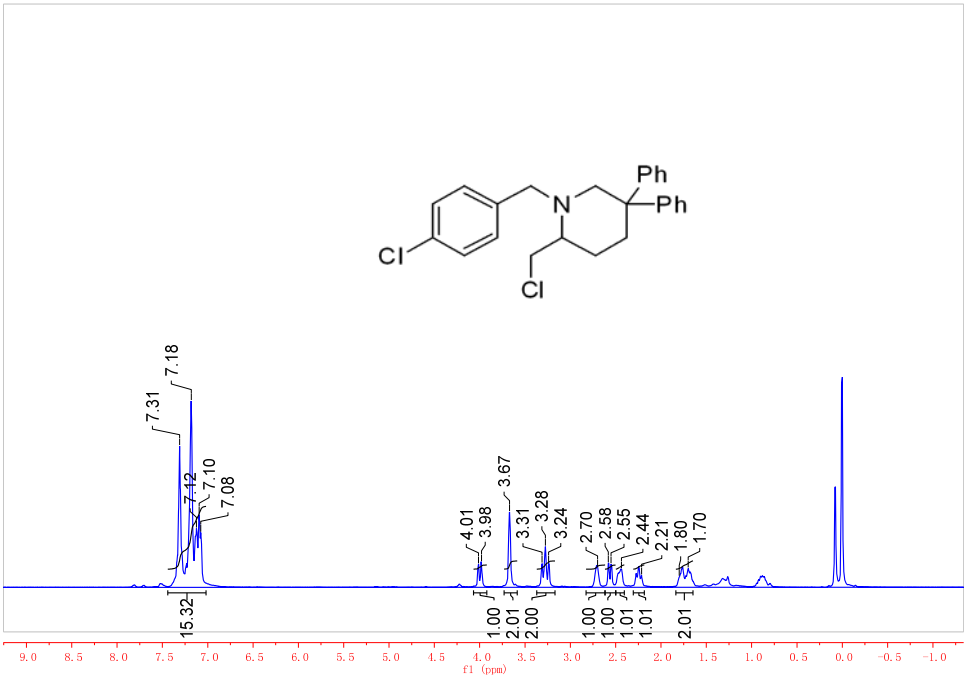
46.20
44.94
33.09
25.67
f1 (ppm)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Ph
N
Cl
Cl
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7.18
7.12
7.10
7.08
4.01
3.98
3.67
3.31
3.28
3.24
2.70
2.58
2.55
2.44
2.21
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1.70
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1.00
2.01
2.00
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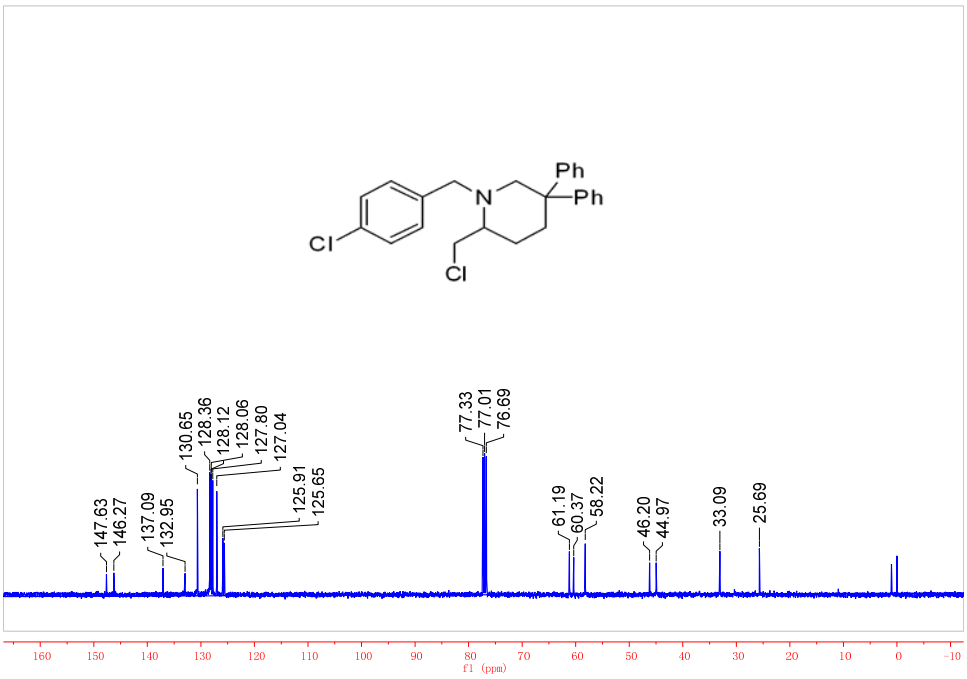
1.00
1.01
1.01
2.01
f1 (ppm)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Cl
Cl
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128.12
128.06
127.80
127.04
125.91
125.65
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76.69
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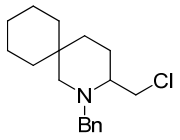
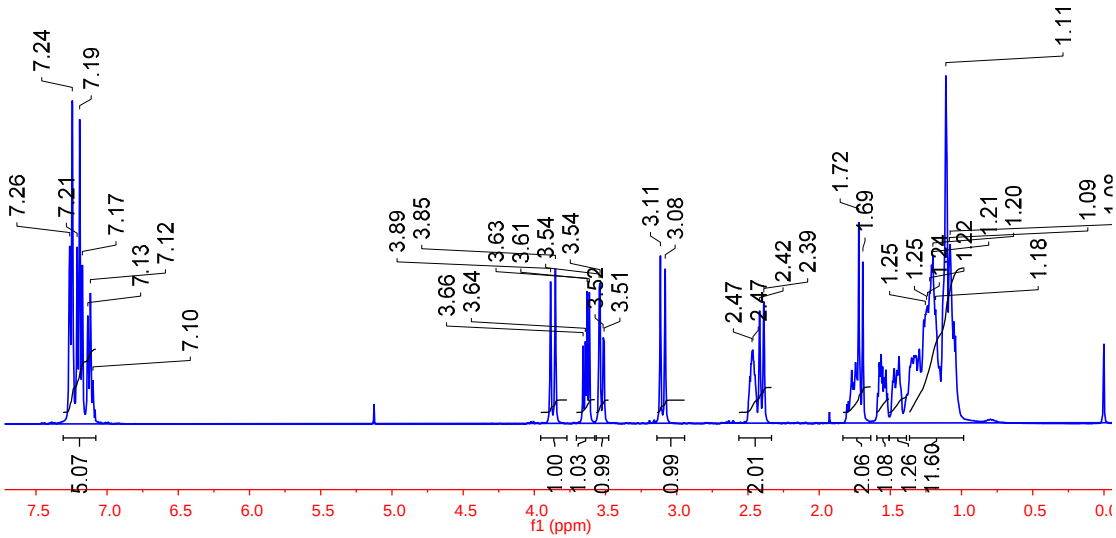
60.37
58.22
46.20
44.97
33.09
25.69
f1 (ppm)


Cl
N
Bn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3.63
3.61
3.54
3.54
3.52
3.51
3.11
3.08
2.47
2.47
2.42
2.39
1.72
1.69
1.25
1.25
1.24
1.22
1.21
1.20
1.18
1.11
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5.07
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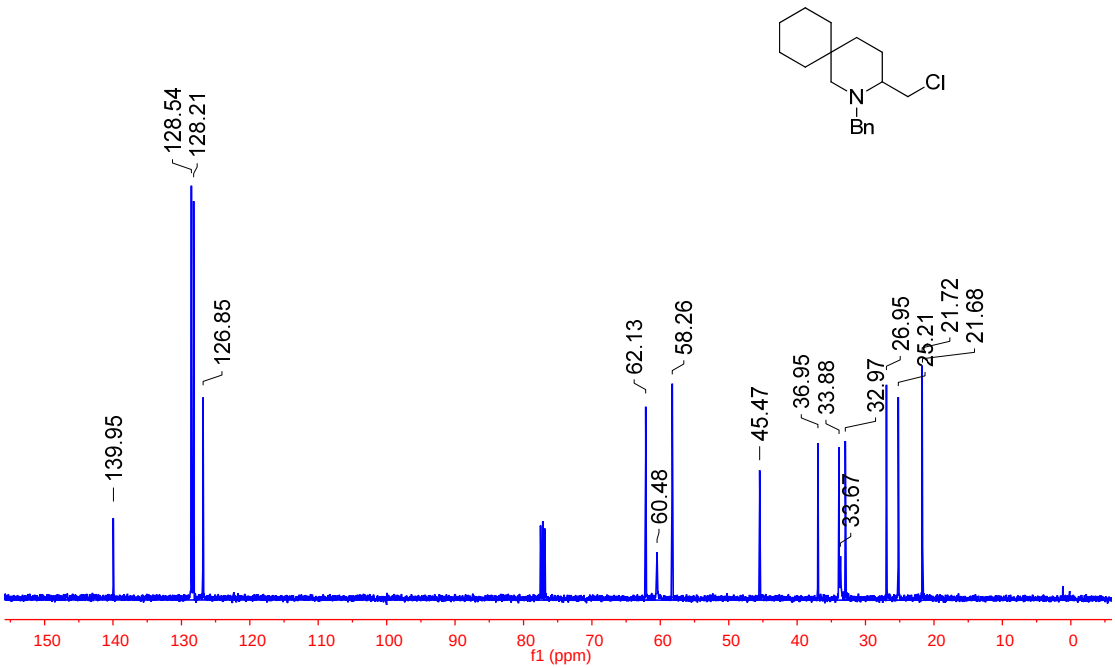
1.03
0.99
0.99
2.01
2.06
1.08
1.26
11.60
f1 (ppm)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Bn
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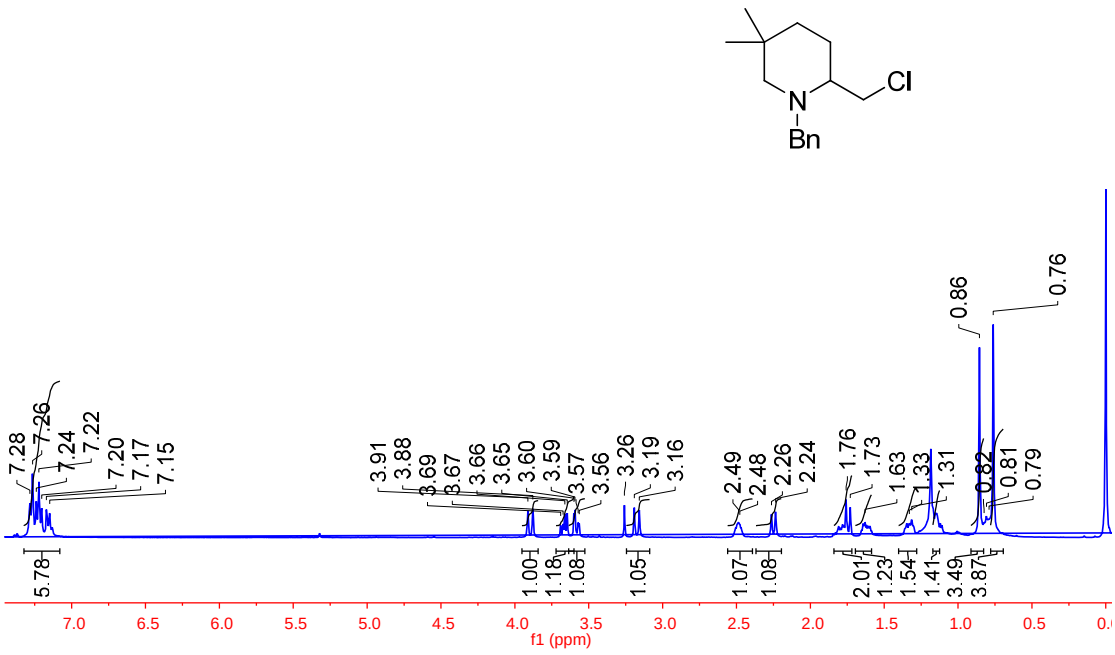
33.88
33.67
32.97
26.95
26.21
21.72
21.68
f1 (ppm)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Bn
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7.24
7.22
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7.17
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3.88
3.69
3.67
3.66
3.65
3.60
3.59
3.57
3.56
3.26
3.19
3.16
2.49
2.48
2.26
2.24
1.76
1.73
1.63
1.33
1.31
0.86
0.82
0.81
0.79
0.76
5.78
1.00
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1.08
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1.07
1.08
2.01
1.23
1.54
1.41
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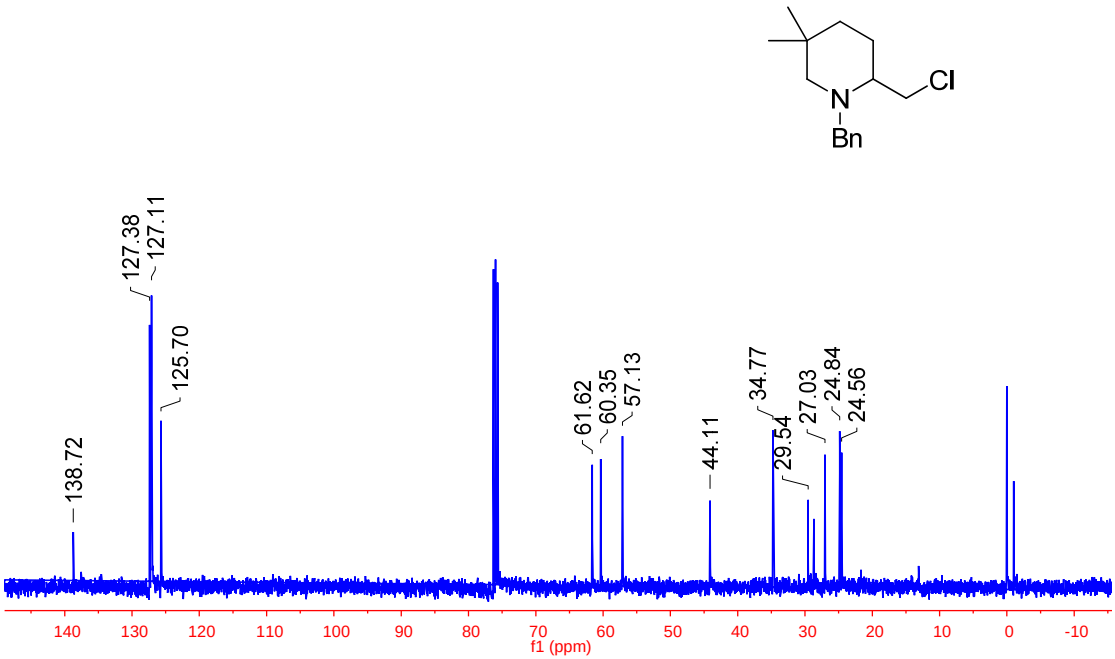
3.87
f1 (ppm)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Bn
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27.03
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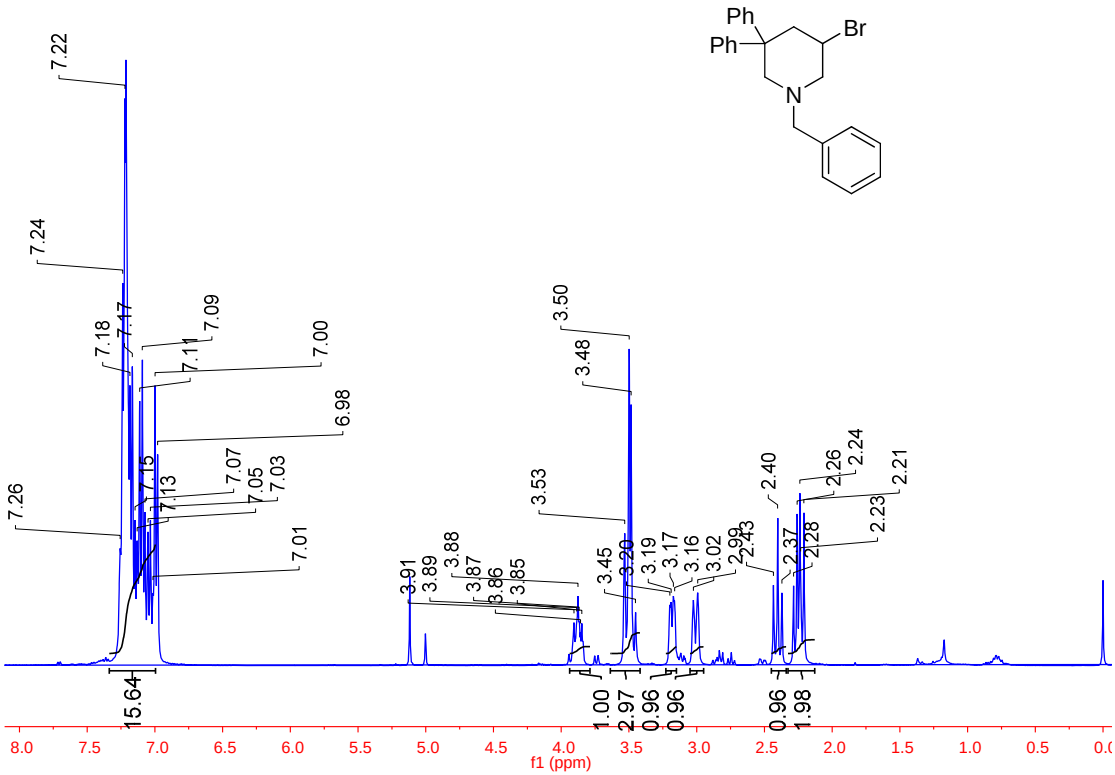
24.56
f1 (ppm)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Ph
Br
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7.15
7.13
7.11
7.09
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6.98
7.01
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3.89
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3.87
3.86
3.85
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3.48
3.45
3.20
3.19
3.17
3.16
3.02
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2.43
2.40
2.37
2.28
2.26
2.24
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2.21
15.64
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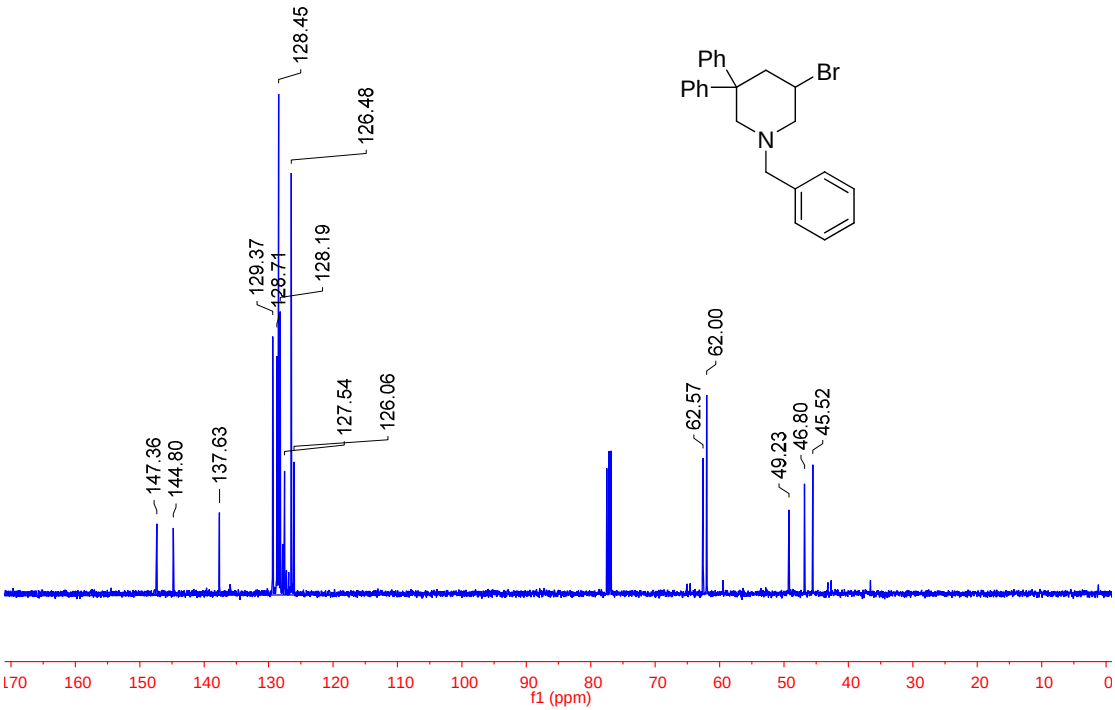
0.96
0.96
1.98
f1 (ppm)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Ph
Br
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126.06
62.57
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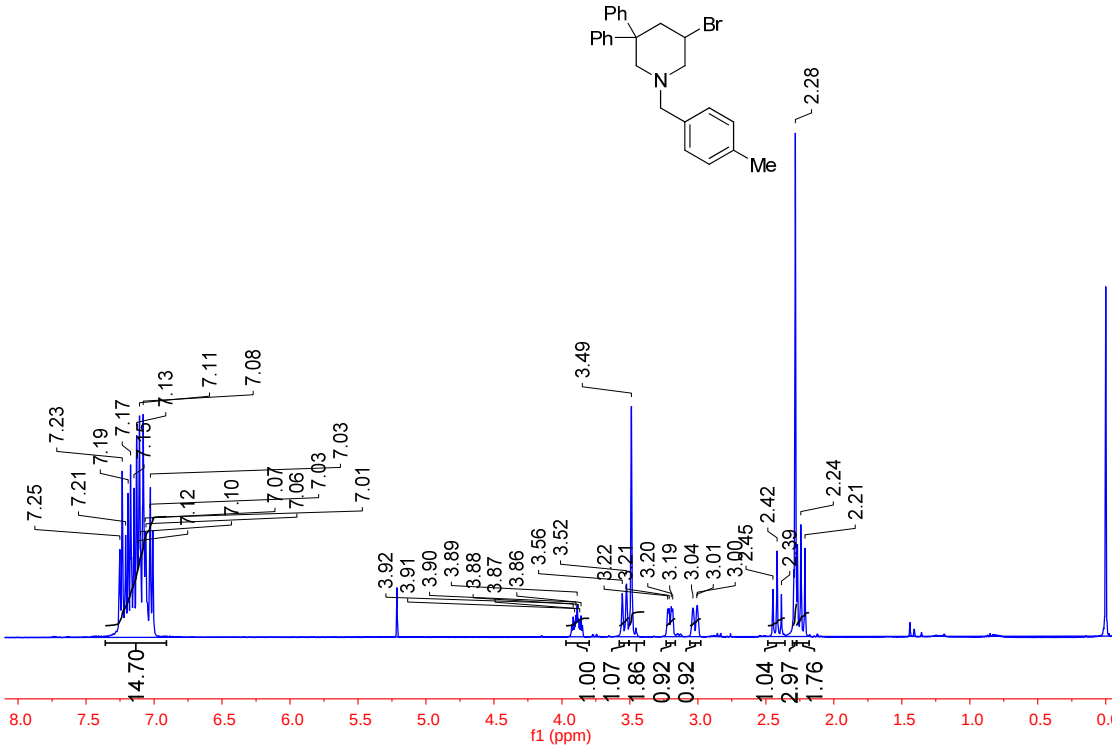
144.80
137.63
49.23
46.80
45.52
f1 (ppm)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Me
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7.13
7.11
7.08
7.25
7.21
7.12
7.10
7.07
7.06
7.03
7.03
7.01
2.24
2.21
2.42
2.39
3.92
3.91
3.90
3.89
3.88
3.87
3.86
3.56
3.52
3.22
3.21
3.20
3.19
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3.01
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1.04
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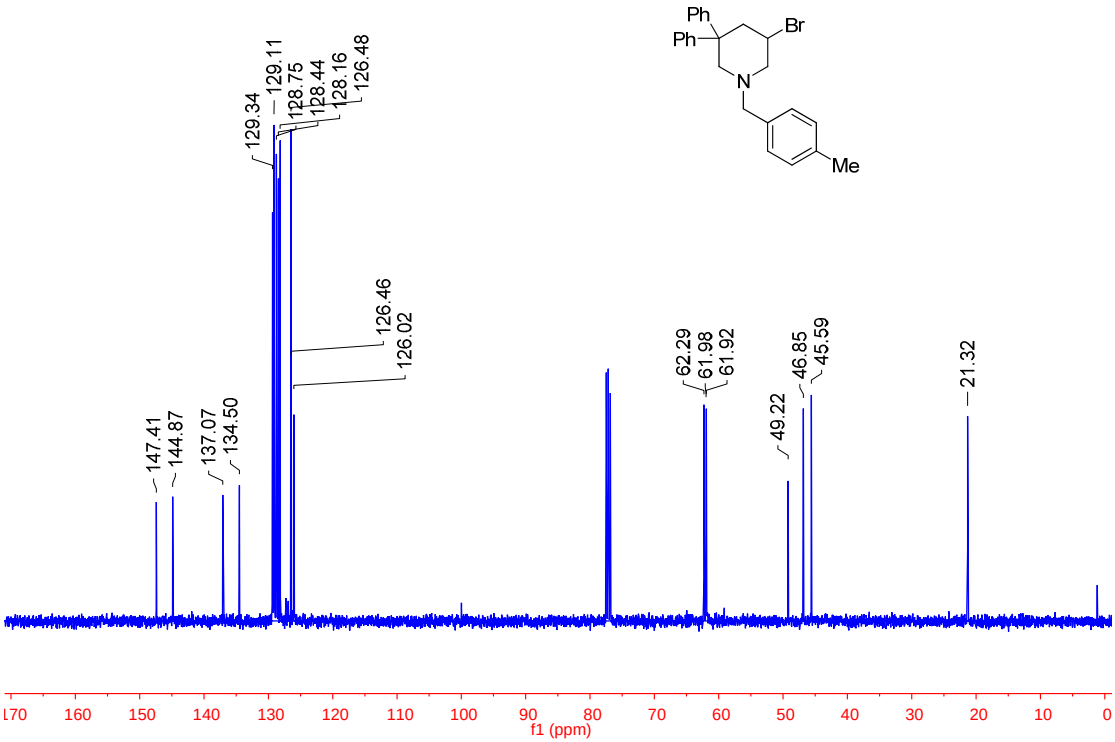
1.76
f1 (ppm)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Br
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Me
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128.44
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126.02
62.29
61.98
61.92
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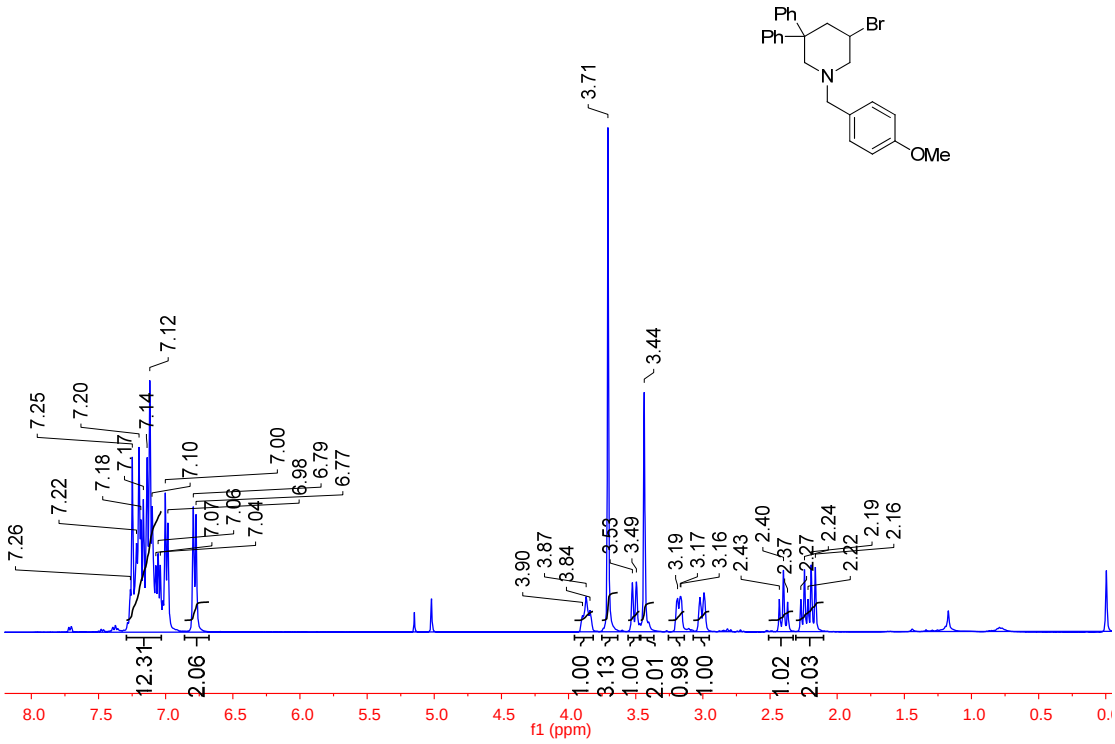
46.85
45.59
21.32
f1 (ppm)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Ph
Br
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OMe
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7.25
7.22
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7.18
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7.14
7.12
7.10
7.07
7.06
7.04
7.00
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6.77
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3.87
3.84
3.71
3.53
3.49
3.44
3.19
3.17
3.16
2.43
2.40
2.37
2.27
2.24
2.22
2.19
2.16
12.31
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1.00
3.13
1.00
2.01
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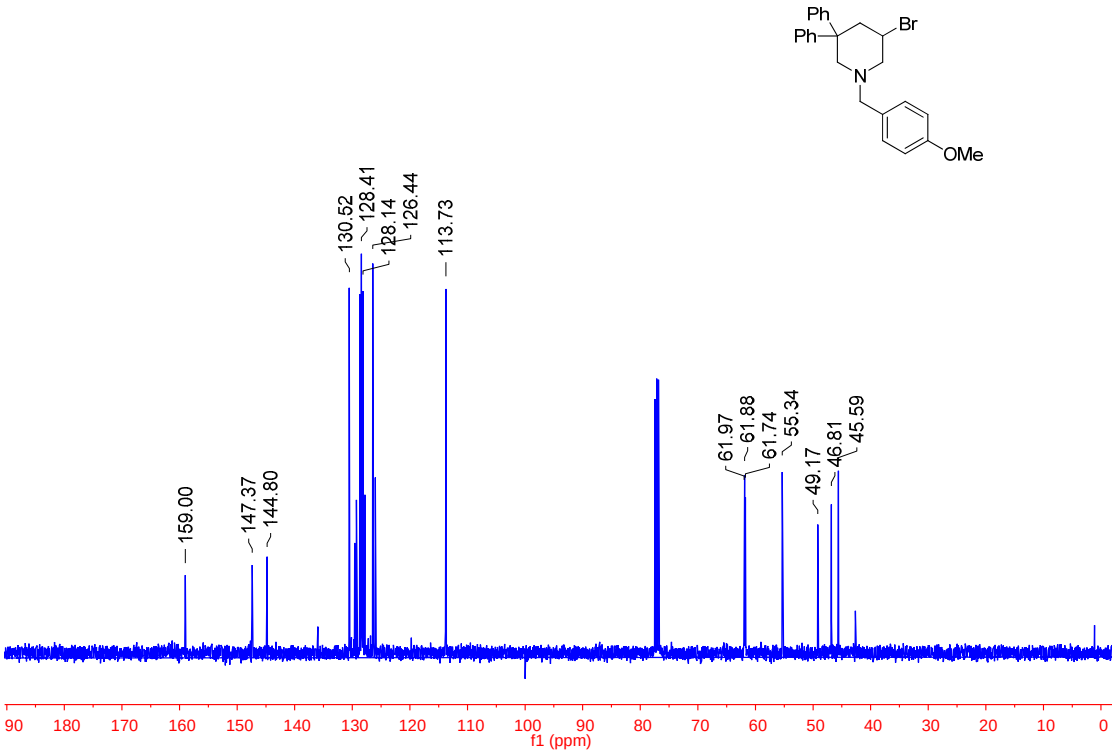
1.00
1.02
2.03
f1 (ppm)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Ph
Br
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OMe
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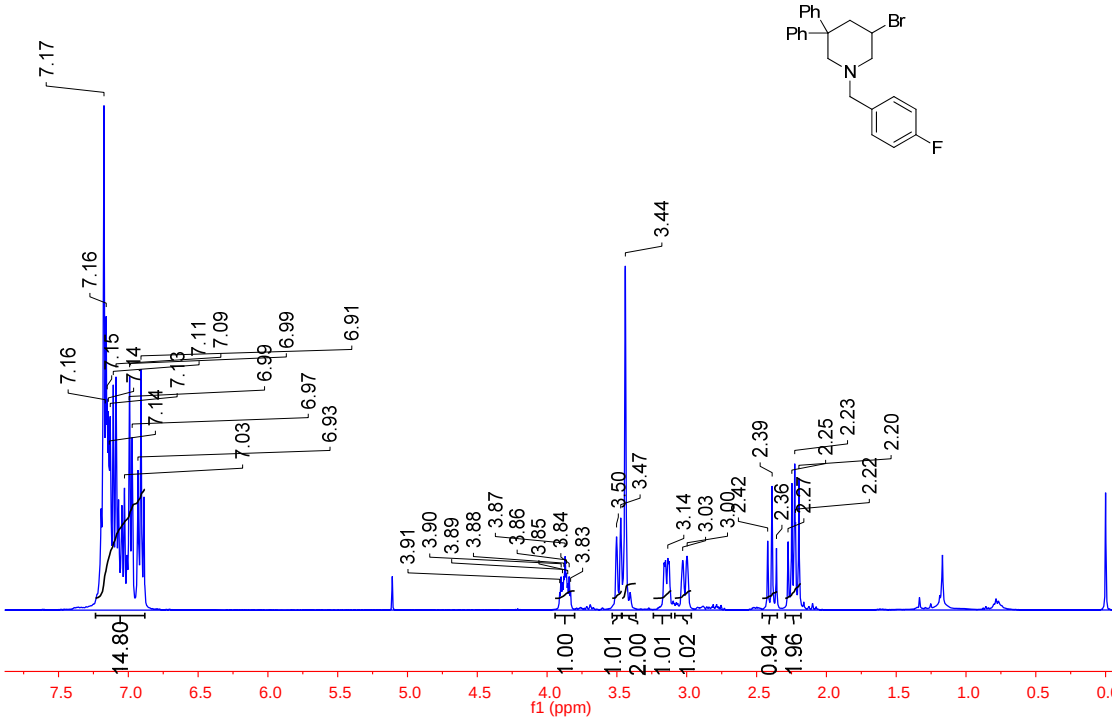
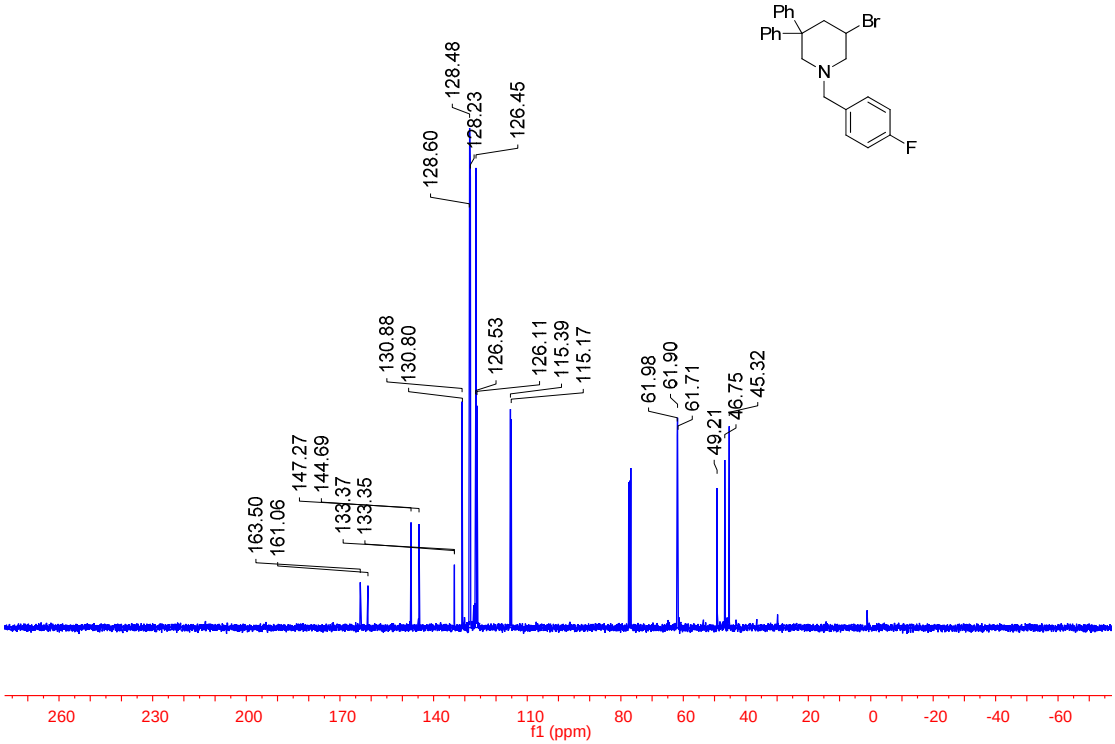
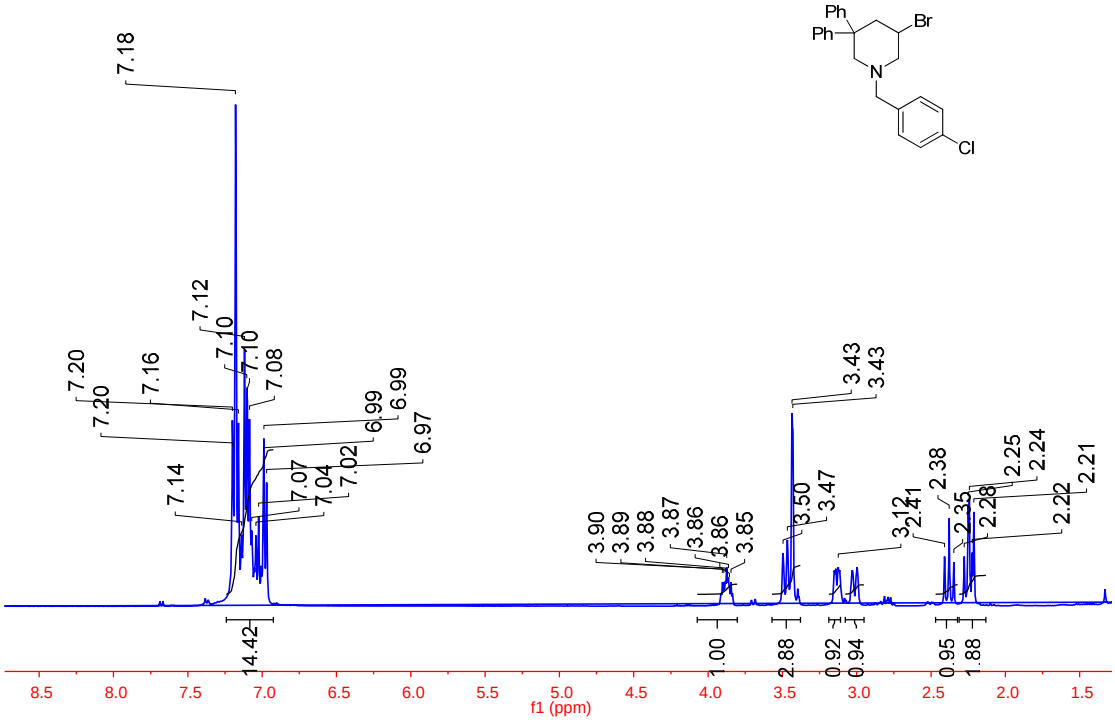
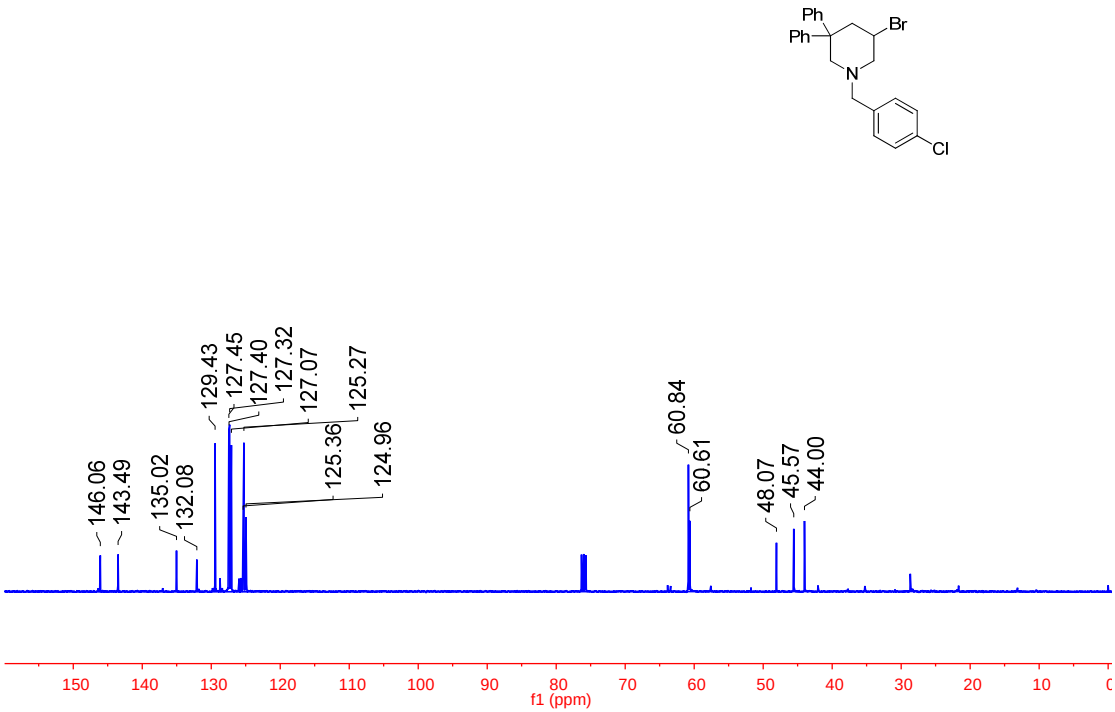
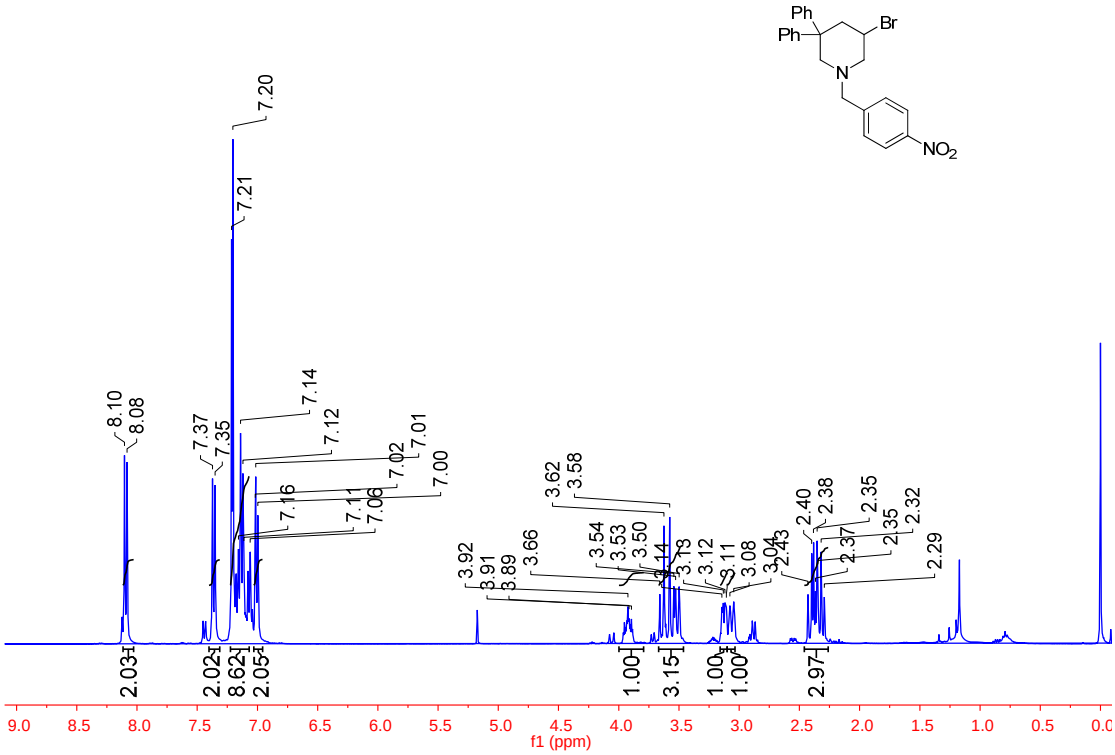
128.41
128.14
126.44
113.73
61.97
61.88
61.74
55.34
49.17
46.81
45.59
f1 (ppm)

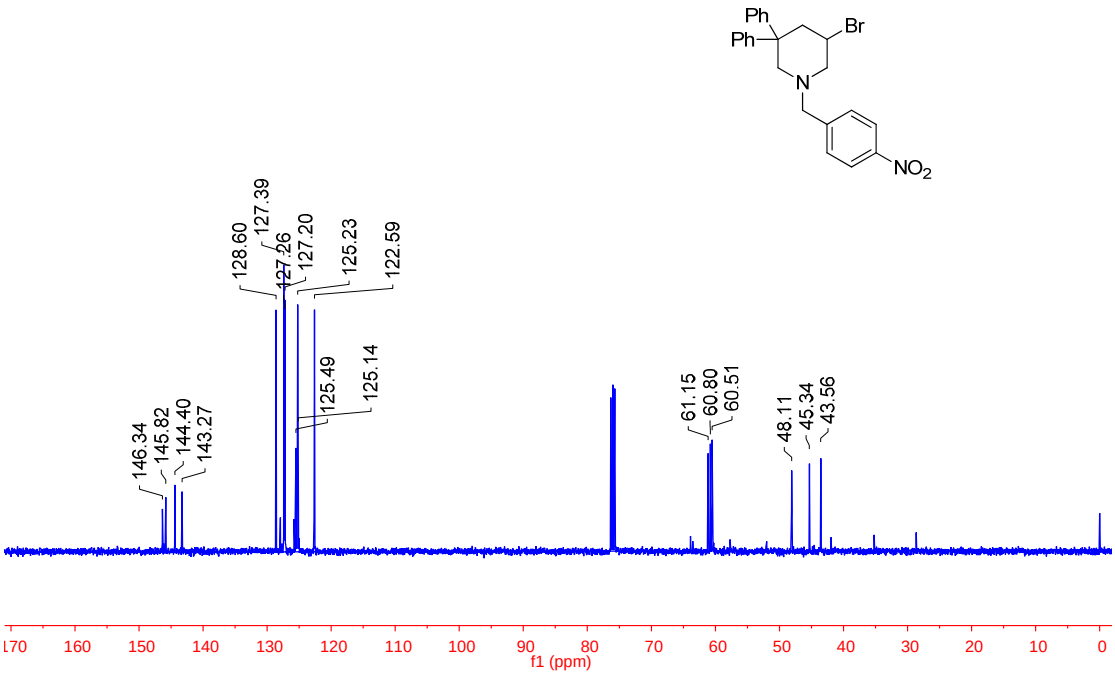

Ph
Ph
Br
N
F
7.17
7.16
6.91
6.99
6.97
6.93
7.03
3.44
3.91
3.90
3.89
3.88
3.87
3.86
3.85
3.84
3.83
3.50
3.47
3.14
3.03
3.00
2.42
2.39
2.36
2.27
2.25
2.23
2.22
2.20
14.80
1.00
1.01
2.00
1.01
1.02
0.94
1.96
f1 (ppm)

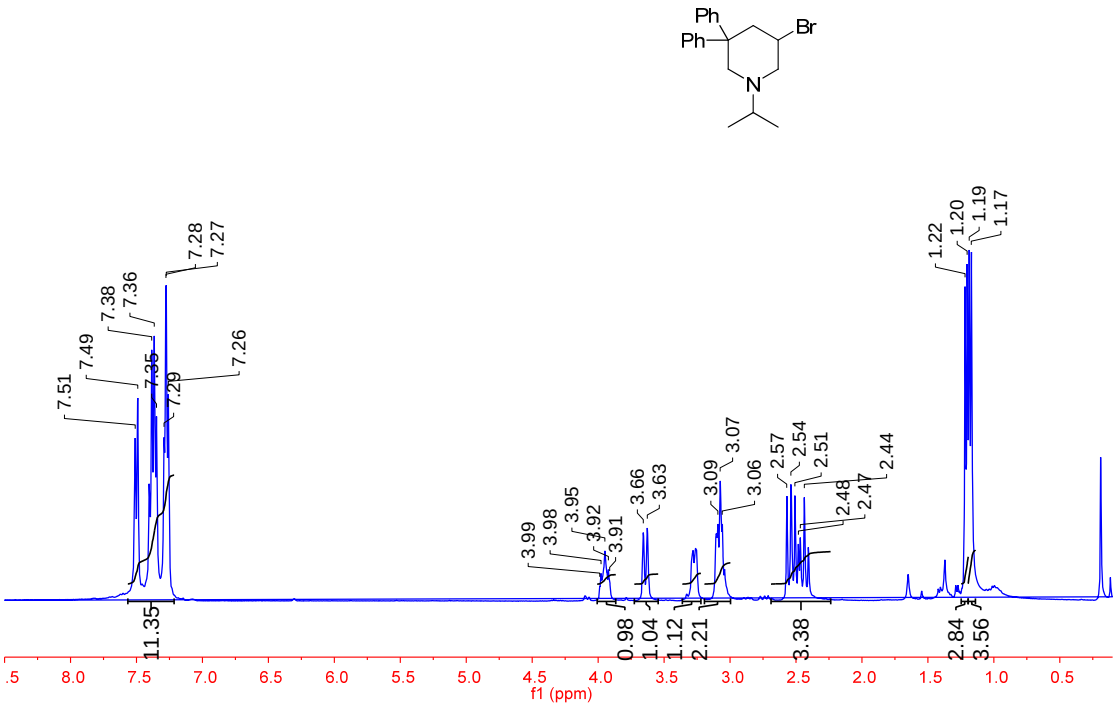

Ph
Ph
Br
N
F
128.48
128.23
126.45
128.60
130.88
130.80
126.53
126.11
115.39
115.17
61.98
61.90
61.71
46.75
45.32
49.21
147.27
144.69
133.37
133.35
163.50
161.06
260
230
200
170
140
110
80
60
40
20
0
-20
-40
-60
f1 (ppm)


Ph
Ph
Br
N
Cl
7.18
7.12
7.10
7.10
7.08
7.20
7.16
7.20
7.14
7.07
7.04
7.02
6.99
6.99
6.97
3.43
3.43
3.90
3.89
3.88
3.87
3.86
3.86
3.85
3.50
3.47
3.12
2.41
2.38
2.35
2.28
2.25
2.24
2.22
2.21
14.42
1.00
2.88
0.92
0.94
0.95
1.88
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)


Ph
Ph
Br
N
Cl
146.06
143.49
135.02
132.08
129.43
127.45
127.40
127.32
127.07
125.27
125.36
124.96
60.84
60.61
48.07
45.57
44.00
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Ph
Ph
Br
N
NO2
8.10
8.08
7.37
7.35
7.20
7.21
7.14
7.12
7.02
7.01
7.00
7.16
7.11
7.06
3.92
3.91
3.89
3.66
3.62
3.58
3.54
3.53
3.50
3.18
3.12
3.11
3.08
3.04
2.43
2.40
2.38
2.37
2.35
2.35
2.32
2.29
2.03
2.02
8.62
2.05
1.00
3.15
1.00
1.00
2.97
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Ph
Ph
Br
N
NO2
146.34
145.82
144.40
143.27
128.60
127.39
127.26
127.20
125.23
122.59
125.49
125.14
61.15
60.80
60.51
48.11
45.34
43.56
f1 (ppm)


Ph
Ph
Br
N
7.51
7.49
7.38
7.36
7.35
7.29
7.28
7.27
7.26
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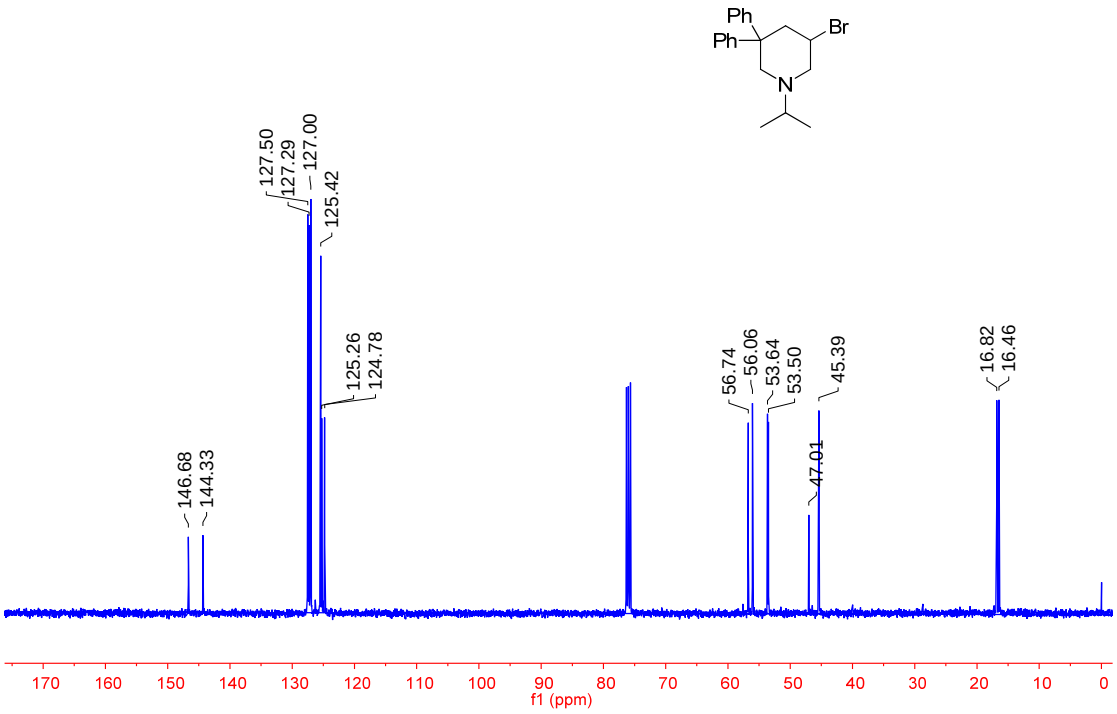
3.98
3.95
3.92
3.91
3.66
3.63
3.09
3.07
3.06
2.57
2.54
2.51
2.48
2.47
2.44
1.22
1.20
1.19
1.17
11.35
0.98
1.04
1.12
2.21
3.38
2.84
3.56
f1 (ppm)


Ph
Ph
Br
N
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144.33
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127.29
127.00
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125.26
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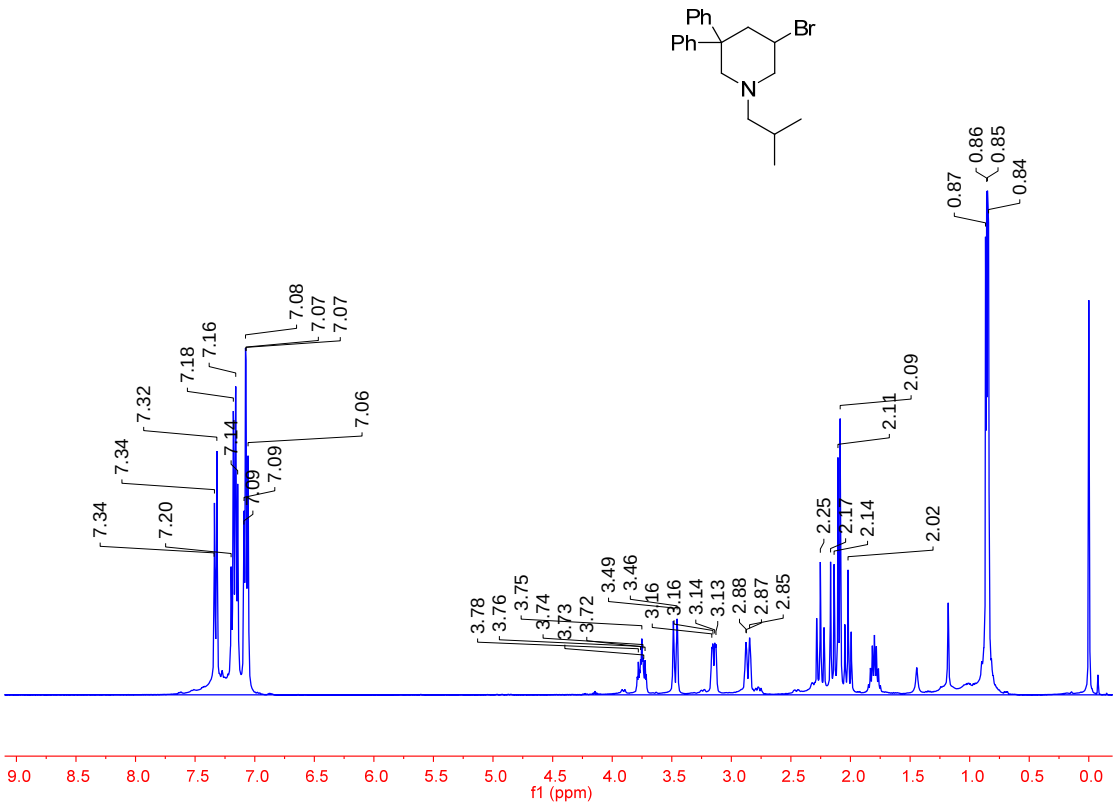
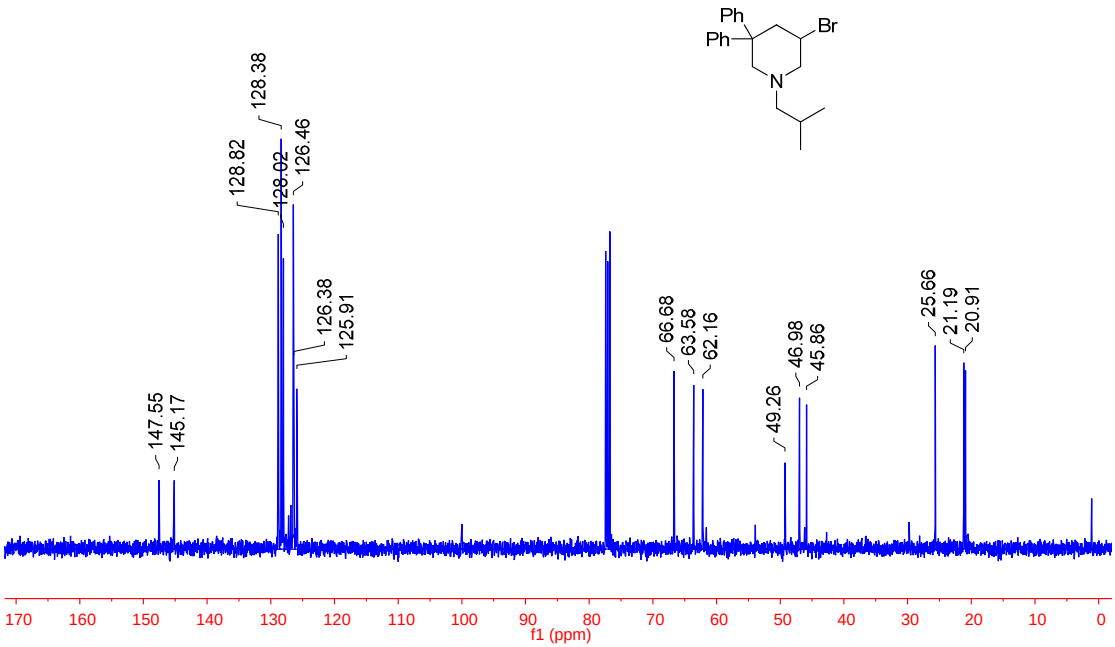
56.74
56.06
53.64
53.50
47.01
45.39
16.82
16.46
f1 (ppm)


Ph
Ph
Br
N
7.34
7.34
7.32
7.20
7.18
7.16
7.14
7.09
7.09
7.08
7.07
7.07
7.06
3.78
3.76
3.75
3.74
3.73
3.72
3.49
3.46
3.16
3.16
3.14
3.13
2.88
2.87
2.85
2.25
2.17
2.14
2.11
2.09
2.02
0.87
0.86
0.85
0.84
f1 (ppm)


Ph
Ph
Br
N
147.55
145.17
128.82
128.38
128.02
126.46
126.38
125.91
66.68
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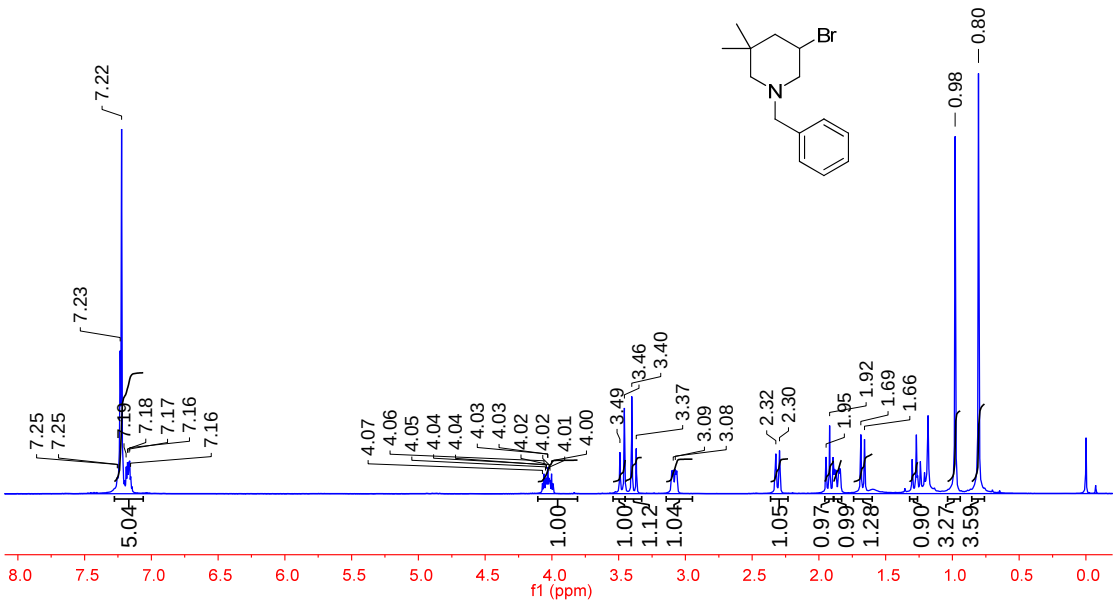
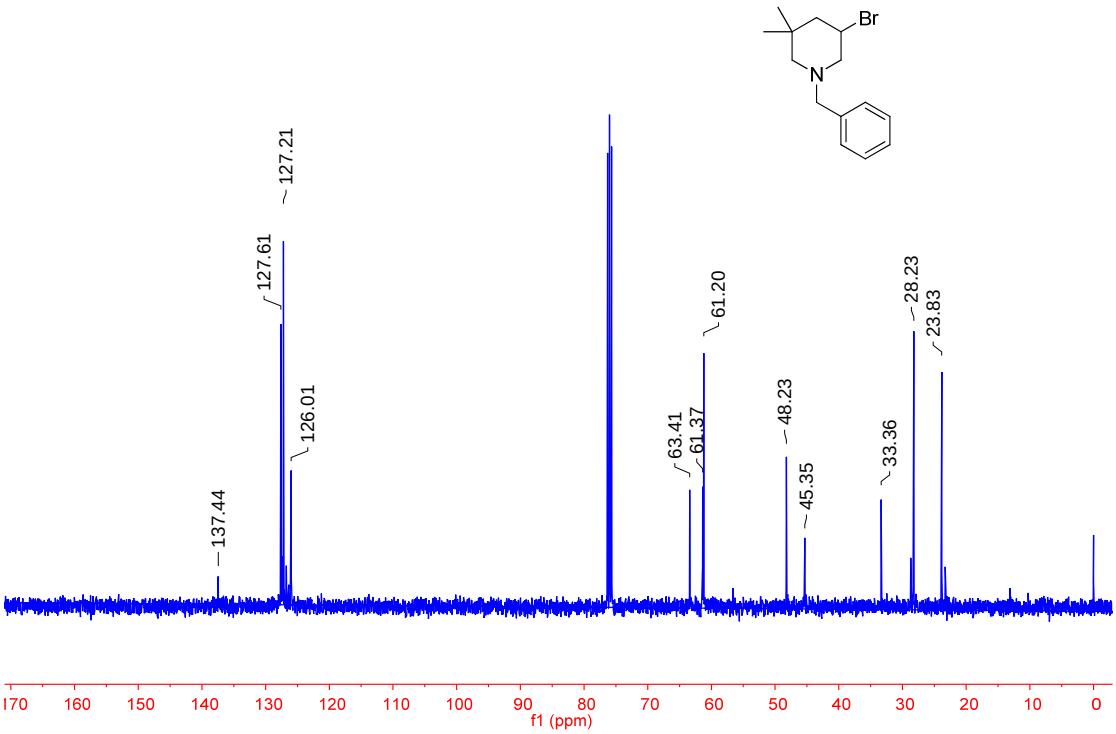
45.86
25.66
21.19
20.91
f1 (ppm)


Br
N
7.25
7.25
7.23
7.22
7.19
7.18
7.17
7.16
7.16
4.07
4.06
4.05
4.04
4.04
4.03
4.03
4.02
4.02
4.01
4.00
3.49
3.46
3.40
3.37
3.09
3.08
2.32
2.30
1.95
1.92
1.69
1.66
0.98
0.80
5.04
1.00
1.00
1.12
1.04
1.05
0.97
0.99
1.28
0.90
3.27
3.59
f1 (ppm)


Br
N
137.44
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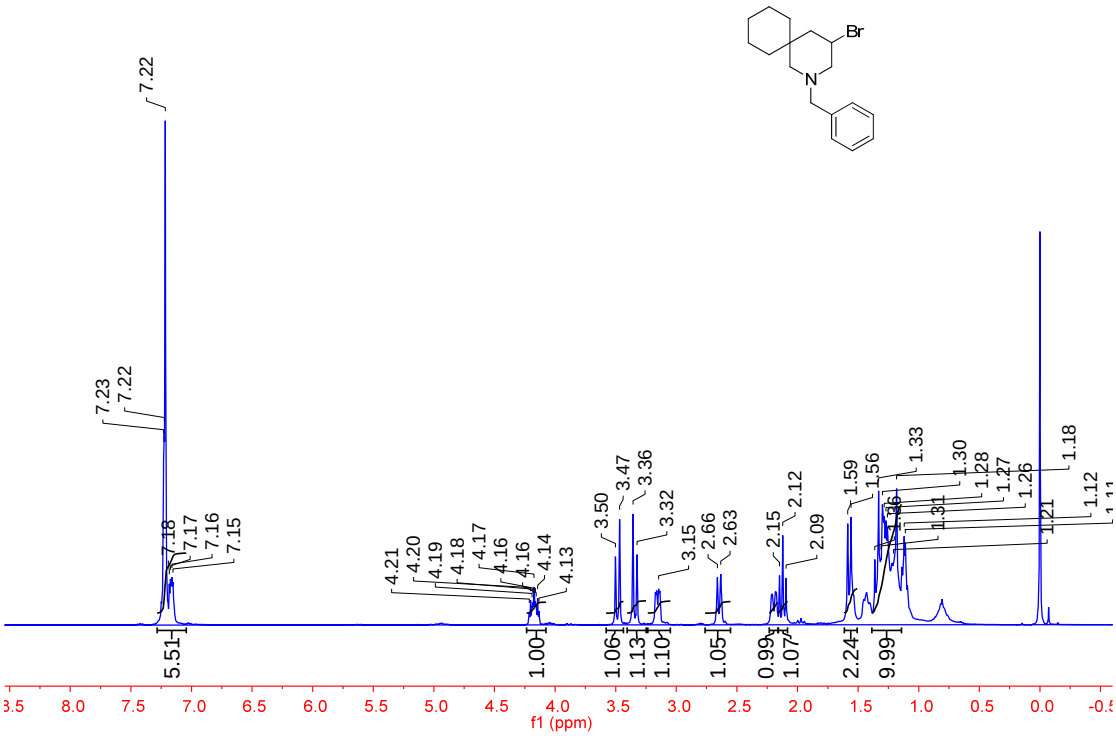
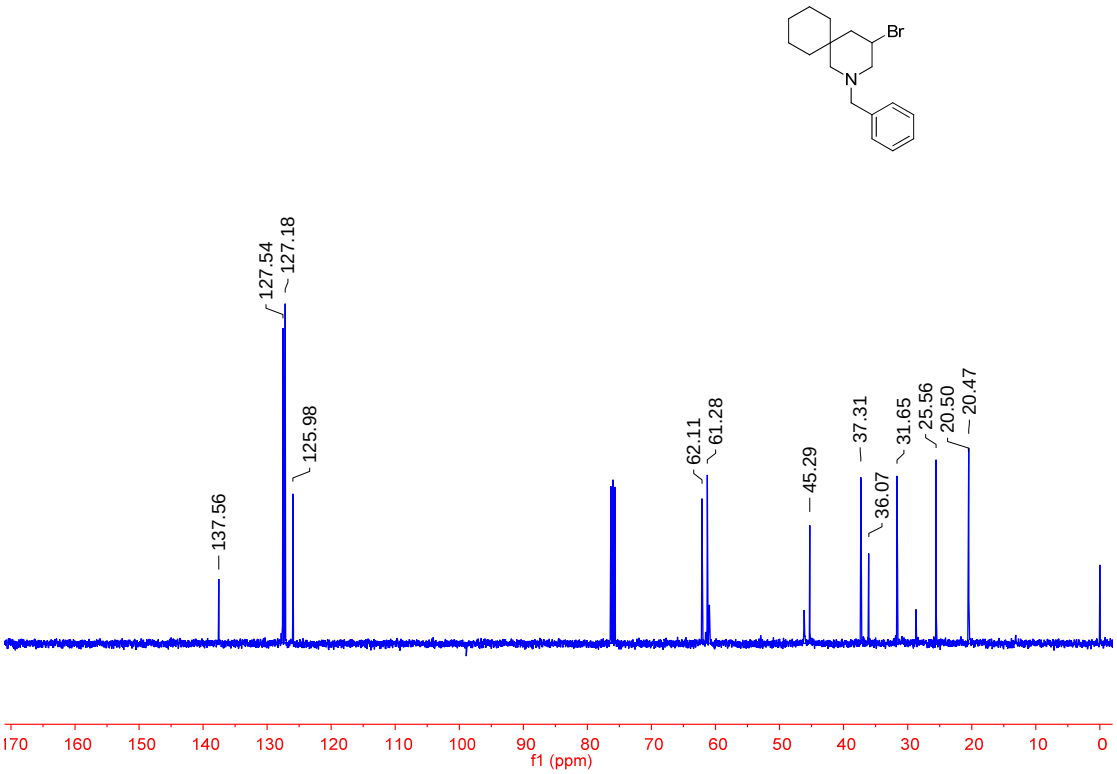
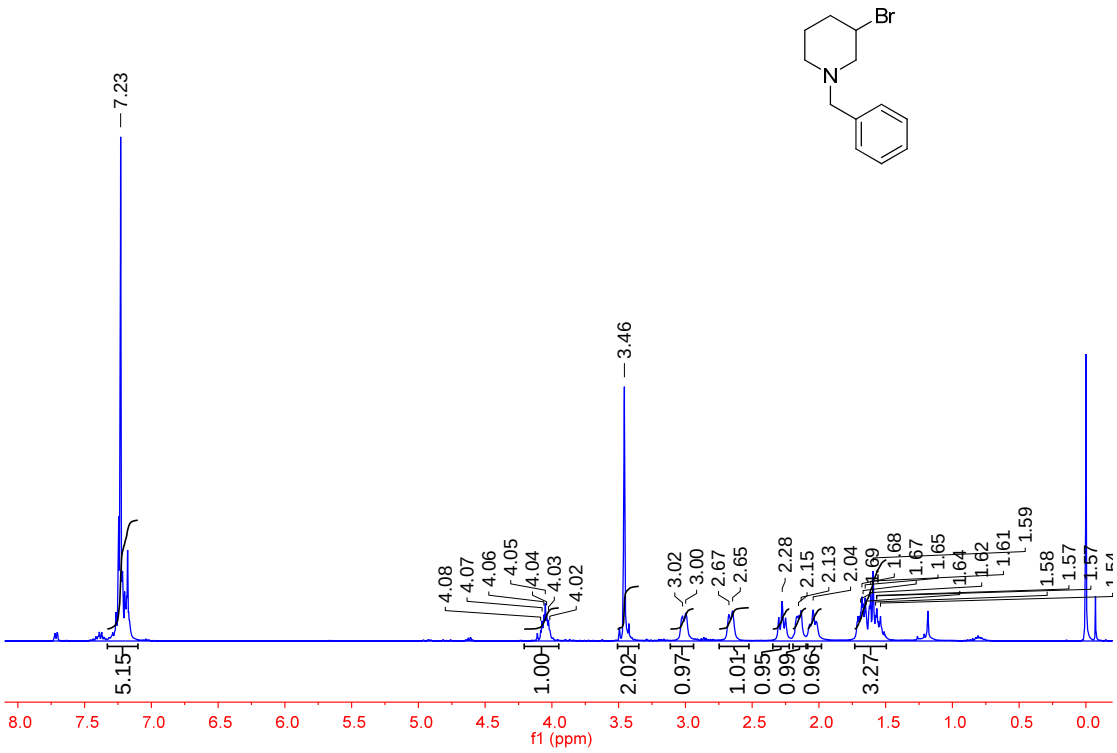
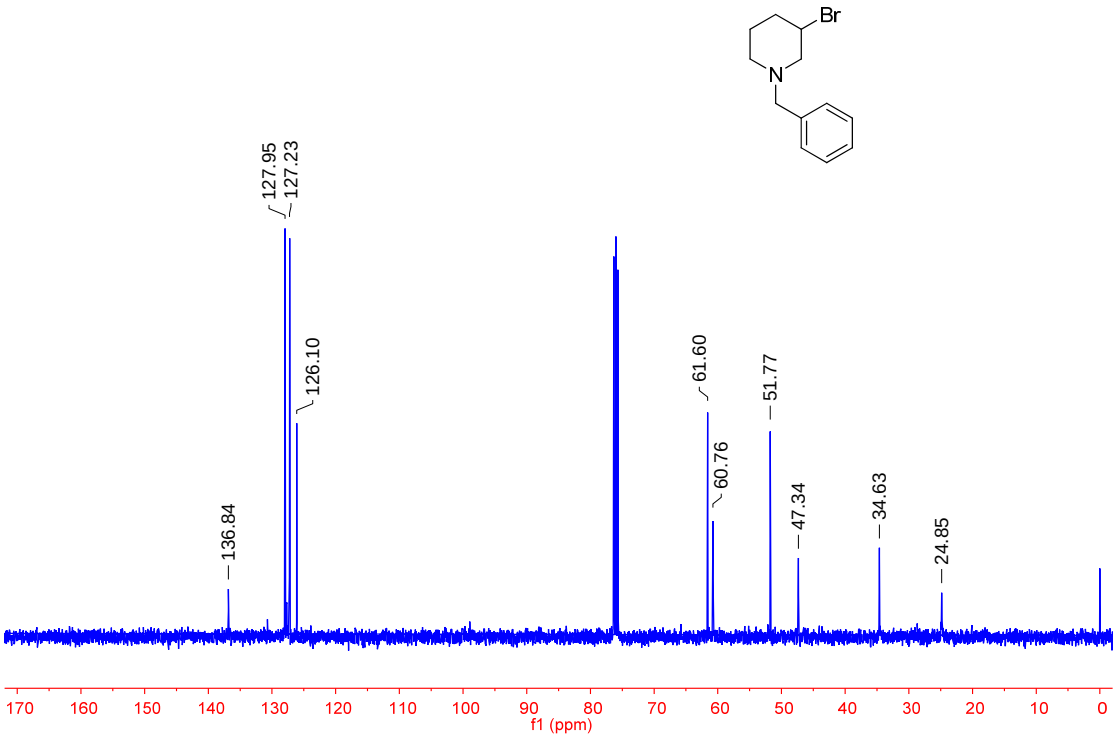
63.41
61.37
61.20
48.23
45.35
33.36
28.23
23.83
f1 (ppm)


Br
N
7.23
7.22
7.18
7.17
7.16
7.15
4.21
4.20
4.19
4.18
4.17
4.16
4.16
4.14
4.13
3.50
3.47
3.36
3.32
3.15
2.66
2.63
2.15
2.12
2.09
1.59
1.56
1.33
1.30
1.28
1.27
1.26
1.21
1.18
1.12
5.51
1.00
1.06
1.13
1.10
1.05
0.99
1.07
2.24
9.99
f1 (ppm)


Br
N
127.54
127.18
125.98
137.56
62.11
61.28
45.29
37.31
36.07
31.65
25.56
20.50
20.47
f1 (ppm)


Br
N
7.23
3.46
4.08
4.07
4.06
4.05
4.04
4.03
4.02
3.02
3.00
2.67
2.65
2.28
2.15
2.13
2.04
1.69
1.68
1.67
1.65
1.64
1.62
1.61
1.59
1.58
1.57
5.15
1.00
2.02
0.97
1.01
0.95
0.99
0.96
3.27
f1 (ppm)


Br
N
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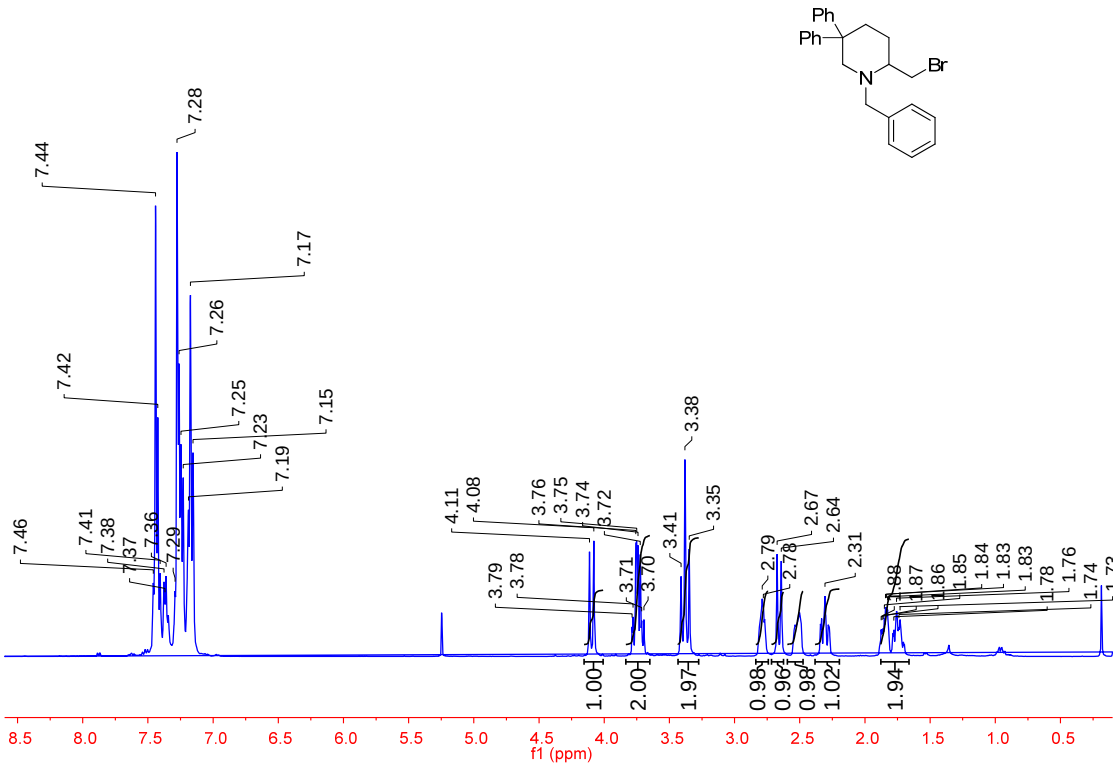
127.23
126.10
136.84
61.60
60.76
51.77
47.34
34.63
24.85
f1 (ppm)


Ph
Ph
N
Br
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7.28
7.17
7.26
7.42
7.25
7.23
7.15
7.19
7.46
7.41
7.38
7.37
7.36
7.29
4.11
4.08
3.76
3.75
3.74
3.72
3.38
3.35
3.41
3.79
3.78
3.71
3.70
2.79
2.78
2.67
2.64
2.31
1.88
1.87
1.86
1.85
1.84
1.83
1.83
1.78
1.76
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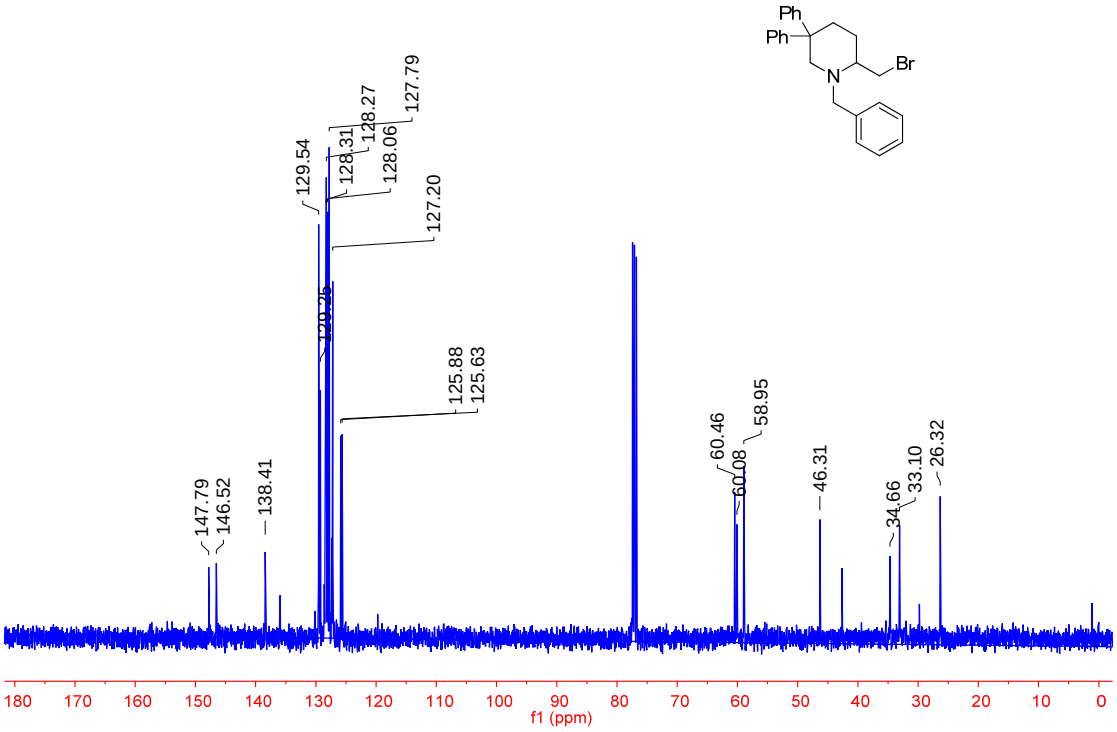
1.00
2.00
1.97
0.98
0.96
0.98
1.02
1.94
f1 (ppm)


Ph
Ph
N
Br
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138.41
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128.27
128.06
127.79
127.20
125.88
125.63
60.46
60.08
58.95
46.31
34.66
33.10
26.32
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)