Supporting Information

Nickel-Catalyzed Regio- and Diastereoselective Arylamination of Unactivated Alkenes

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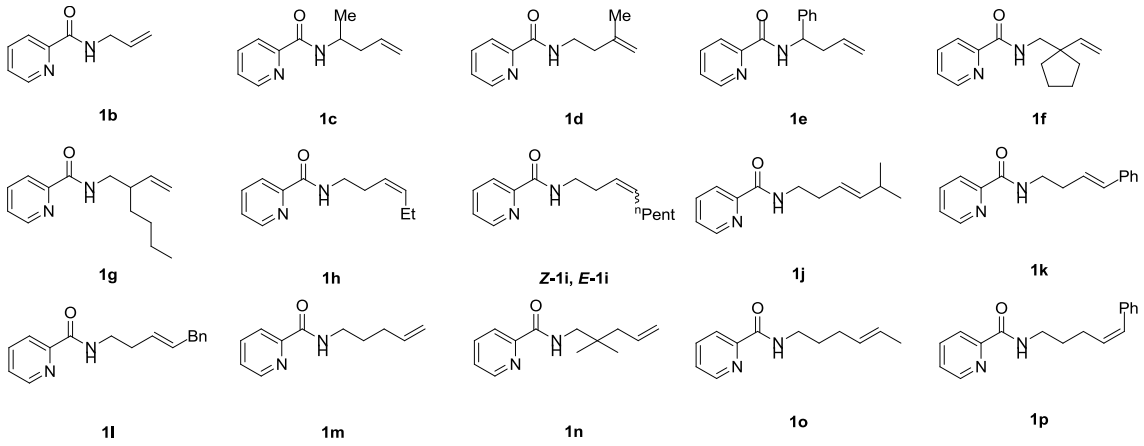
1. General Remarks

All the manipulations were performed in an argon-filled glovebox, unless mentioned otherwise. Anhydrous solvent was purchased from commercial sources and transferred under argon atmosphere. Alkene substrates and Amine benzoate substrates were prepared according to previously reported procedures, all arylboronic acids were purchased from commercial sources and used without further purification. All reagents were purchased from Energy Chemicals and used as received.

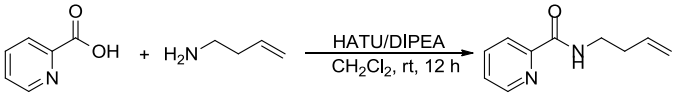
^{1}H NMR, ^{13}C NMR spectra were recorded using Bruker 400 MHz NMR spectrometer. ^{1}H NMR and ^{13}C NMR spectra were referenced to resonances of the residual protons in the deuterated solvents. Multiplicities are recorded as: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, br = broad singlet and m = multiplet. GC-MS analysis was performed on Shimadzu GC-2010 gas chromatography coupled to a Shimadzu QP2010 mass selective detector. Analytical HPLC/MS was performed with an Agilent 6520 Series HPLC. Agilent 1200 Series HPLC.

2. Alkene Substrate Synthesis

Table S1. Picolinamide-containing alkene substrates **1b-1k.**



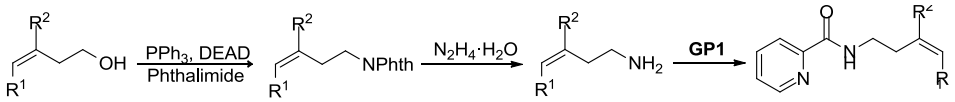
General Procedure for Amide Coupling (GP1):



Compound **1b, 1m** were synthesized from amines[1].

To a 50 mL flask was added alkenyl carboxylic amide (10 mmol, 1.0 eq), picolinic acid (12 mmol, 1.2 eq), HATU (11 mmol, 1.1 eq), DIPEA (20 mmol, 2.0 eq) and CH_2Cl_2 (30 mL). The reaction mixture was left to stir for 12 h. Upon completion, the reaction was quenched with brine (10 mL), and extracted with CH_2Cl_2 (50 mL × 4). The organic layers were combined, and the solvent was removed in vacuo to yield a yellow residue. Purification using column chromatography gave the pure product.

General Procedure for Amide Coupling(GP2)：



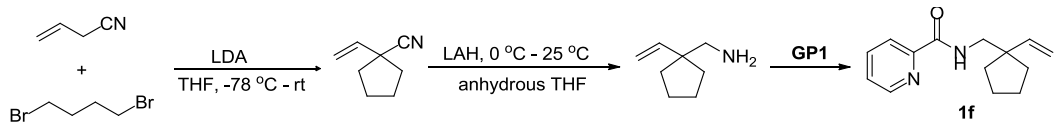
Compound **1c-1e, 1h-1i, 1o** were synthesized from enols[2].

To a mixture of triphenylphosphine (25 mmol, 1.0 eq), phthalimide (25 mmol, 1.0 eq) and the corresponding allyl alcohol (25 mmol, 1.0 eq) in THF (30 mL) was slowly added diethyl azodicarboxylate (DEAD) (25 mmol, 1.0 eq) at 0 ℃. The mixture was stirred at 0 ℃ for 3 h. After the completion of the reaction, the reaction mixture was diluted with *n*-hexane and filtered. The filtrate was dried over Na_2SO_4 and concentrated in vacuo to give the crude product, which was used without further purification.

To the solution of phthalimide product in ethanol (100 mL) was added hydrazine monohydrate (25 mmol) at 50 ℃. The mixture was stirred for 1 h and quenched with 6 M HCl (20 mL). The precipitates formed were removed by filtration, and the resultant filtrate was dried over Na_2SO_4 and concentrated in vacuo to give an unsaturated amine hydrochloride. Aqueous NaOH (6.0 M, 10 mL) was added to the amine salt, and the resulting solution was extracted with CH_2Cl_2 (25 mL × 3). The combined organic extracts was then washed again with brine (10 mL), dried over Na_2SO_4, and filtered. The amine solution was used without further purification.

To the solution of amine (25 mmol, 1.0 eq) was successively added picolinic acid (30 mmol, 1.2 eq), HATU (27.5 mmol, 1.1 eq) and DIPEA (50 mmol, 2.0 eq). The resultant mixture was stirred at room temperature overnight. Water was added and the mixture was extracted with CH_2Cl_2 (50 mL × 3). The combined organic layers were washed with water and brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The resulting residue was purified by alumina gel flash chromatography (ethyl acetate:hexanes = 1:8) to give the desired product.

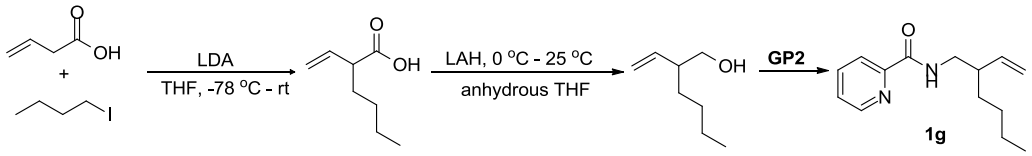
Synthesis of compound **1f**.



To a 100 mL schlenk flask was added lithium diisopropylamide (44 mmol, 1.1 eq, 2.0 M in THF), anhydrous THF (10 mL) under Ar atmosphere. The resulting solution was submerged in a -78 ℃ dry ice bath. A solution of acrylonitrile (40 mmol, 1.0 eq) in 10 mL THF was added dropwise over 5 min, and a solution of 1,4-dibromobutane (38 mmol, 0.95 eq) in 10 mL THF was added dropwise over 0.5 h. After 4 h at this temperature, the solution was quenched slowly with water (10 mL). The aqueous layer was transferred to a separatory funnel and washed with Et_2O (50 mL × 2) before being charged back into the schlenk flask. Hydrochloric acid was added dropwise into the vigorously stirring solution at 0 ℃ until pH = 3. The milky solution was then extracted with EtOAc (100 mL × 2). The combined organic extracts were washed with brine (50 mL), dried over Na_2SO_4, concentrated, and carried forward to the next step without further purification.

To a 250 mL oven-dried flask under Ar atmosphere was added anhydrous THF (100 mL) followed by $LiAlH_4$ (15 mL, 2.4 M in THF). A solution of nitrile (40 mmol, 1.0 eq) in THF (50 mL) was added dropwise at 0 ℃. The reaction vessel was allowed to warm to room temperature and left to stir for 3 h. After this time, the reaction mixture was diluted with Et_2O, washed with 1 M HCl, sat. $NaHCO_3$ solution, brine and extracted with EtOAc (10 mL × 5)[3]. The organic solvent is removed to get a yellow oil which is used to synthesize the final product **1f** through the process **GP1**.

Synthesis of compound **1g**.

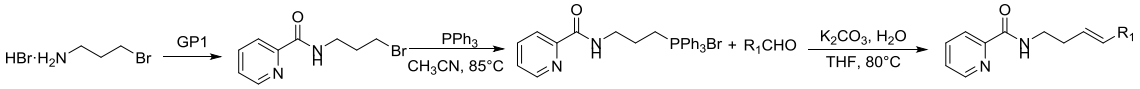


To a 100 mL schlenk flask was added LDA (55 mmol, 2.2 eq, 2.0 M in THF), anhydrous THF (10 mL) under Ar atmosphere. The resulting solution was submerged in a -78 ℃ dry ice bath. A solution of 3-butenoic acid (25 mmol, 1.0 eq) in 5 mL THF was added dropwise over 5 min, and

a solution of 1-iodobutane (25 mmol, 1.0 eq) in 5 mL THF was added dropwise over 5 min. After 4 h at this temperature, the solution was quenched slowly with water (10 mL). The aqueous layer was transferred to a separatory funnel and washed with Et_2O (50 mL × 2) before being charged back into the schlenk flask. Hydrochloric acid was added dropwise into the vigorously stirring solution at 0 ℃ until pH = 3. The milky solution was then extracted with EtOAc (100 mL × 2). The combined organic extracts were washed with brine (50 mL), dried over Na_2SO_4, concentrated, and carried forward to the next step without further purification.

To a 250 mL oven-dried flask under Ar atmosphere was added anhydrous THF (100 mL) followed by LAH (15 mL, 2.4 M in THF). A solution of acid (25 mmol, 1.0 eq) in THF (50 mL) was added dropwise at 0 ℃. The reaction vessel was allowed to warm to room temperature and left to stir for 3 h. After this time, the reaction mixture was diluted with Et_2O, washed with 1 M HCl, sat. $NaHCO_3$ solution, brine and extracted with EtOAc (10 mL × 5)[3]. The organic solvent is removed to get a yellow oil which is used to synthesize the final product **1g** through the process **GP2**.

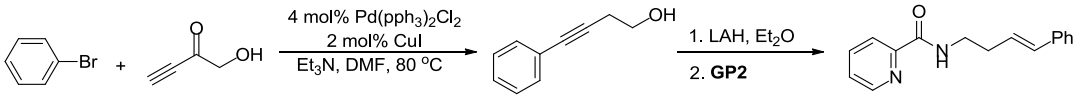
Synthesis of compound **1j, 1l**.



To a 100 mL schlenk flask was added 3-Bromopropylamine hydrobromide (30 mmol, 1.0 eq), then through the process **GP1**. After that, the reaction mixture was diluted with EtOAc (100 mL) and washed with brine (3×100 mL). The organic layer was separated, dried over Na_2SO_4 , and concentrated, and carried forward to the next step without further purification.

PPh_3 (30 mmol, 1.0 eq), CH_3CN (150 mL) was added to the resulting solution and then the reaction vessel was allowed to heat to reflux under argon for 48 h. After this time, the reaction vessel was cooled to room temperature, removed solvent by vacuum. Then added aldehyde (30 mmol, 1.0 eq), K_2CO_3 (45 mmol, 1.5 eq), H_2O (30 mmol, 1.0 eq) in THF (80 mL) was stirred at 80 ℃ for 12 h. After that, the combined organic layers were washed with water and brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography (ethyl acetate:hexanes = 1:8) to give the desired product.

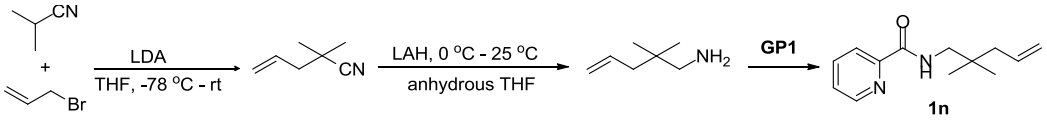
Synthesis of compound **1k**.



To a 100 mL flask was added bromobenzene (50 mmol, 1.0 eq), 1-hydroxybut-3-yn-2-one (55 mmol, 1.1 eq), $Pd(PPh_3)_2Cl_2$ (0.008 mmol, 4 mol%), CuI (0.004 mmol, 2 mol%), Et_3N (60 mmol, 1.2 eq) and DMF (50 mL). The reaction mixture was left to stir for 12 h at 80 ℃. Upon completion, the reaction was quenched with brine (10 mL), and extracted with CH_2Cl_2 (50 mL × 4). The organic layers were combined, and the solvent was removed in vacuo to yield a brown residue. Purification using column chromatography gave the pure product.[4]

To a suspension of LAH (75 mmol, 1.5 eq) in diethyl ether (0.3 M) under N_2 at 0 ℃ was slowly added a solution of 4-phenylbut-3-yn-1-ol (50 mmol, 1.0 eq). After 15 min the reaction was allowed to warm to room temperature and stirred for an additional 3 h. The reaction was re-cooled to 0 ℃, diluted with wet Et_2O, quenched by slow addition of aq. NaOH (1 M), stirred for an additional 0.5 h, filtered through celite and concentrated under reduced pressure. The resulting crude product was used to synthesize the final product **1k** through the process **GP2**.

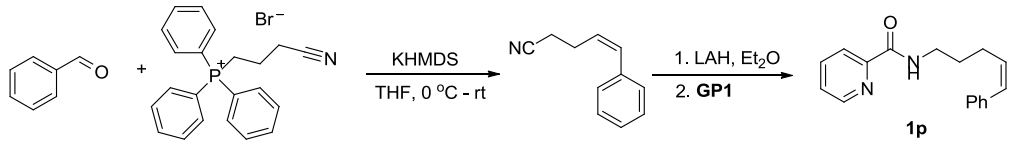
Synthesis of compound **1n**.



To a 100 mL schlenk flask was added LDA (66 mmol, 1.1 eq, 2.0 M in THF), anhydrous THF (10 mL) under Ar atmosphere. The resulting solution was submerged in a -78 ℃ dry ice bath. A solution of isobutyronitrile (60 mmol, 1.0 eq) in 10 mL THF was added dropwise over 5 min, and a solution of allyl bromide (72 mmol, 1.2 eq) in 10 mL THF was added dropwise over 0.5 h. After 4 h at this temperature, the solution was quenched slowly with water (10 mL). The aqueous layer was transferred to a separatory funnel and washed with Et_2O (50 mL × 2) before being charged back into the schlenk flask. Hydrochloric acid was added dropwise into the vigorously stirring solution at 0 ℃ until pH = 3. The milky solution was then extracted with EtOAc (100 mL × 2). The combined organic extracts were washed with brine (50 mL × 1), dried over Na_2SO_4, concentrated, and carried forward to the next step without further purification.

To a 250 mL oven-dried flask under Ar atmosphere was added anhydrous THF (100 mL) followed by $LiAlH_4$ (15 mL, 2.4 M in THF). A solution of nitrile (60 mmol, 1.0 eq) in THF (50 mL) was added dropwise at 0 ℃. The reaction vessel was allowed to warm to room temperature and left to stir for 3 h. After this time, the reaction mixture was diluted with Et_2O, washed with 1 M HCl, sat. $NaHCO_3$ solution, brine and extracted with EtOAc (10 mL × 5)[3]. The organic solvent is removed to get a yellow oil which is used to synthesize the final product **1n** through the process **GP1**.

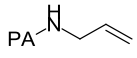
Synthesis of compound **1p**.



To a suspension of (3-cyanopropyl)triphenylphosphonium bromide (7.3 mmol, 1.1 eq) in 20 mL THF at 0 ℃ was added KHMDS (20 wt% in THF, 8.3 mmol, 1.25 eq) slowly over 2 min, which resulted in an orange suspension. The reaction was stirred at 0 ℃ for 15 min before the benzaldehyde (6.65 mmol, 1.0 eq) was added in one portion. The reaction was stirred an additional 0.5 h at 0 ℃, 1.5 h at room temperature. The resulting mixture was filtered through a silica plug, concentrated and purified by flash column silica gel chromatography using the indicated solvent system[5].

To a suspension of LAH (10 mmol, 1.5 eq) in Et_2O (0.3 M) under N_2 at 0 ℃ was slowly added a solution of nitrile (6.65 mmol, 1.0 eq). After 15 min the reaction was allowed to warm to room temperature and stirred for an additional 3 h. The reaction was re-cooled to 0 ℃, diluted with wet Et_2O, quenched by slow addition of aq. NaOH (1 M), stirred for an additional 0.5 h, filtered through celite and concentrated under reduced pressure. The resulting crude product was used to synthesize the final product **1p** through the process **GP1**.

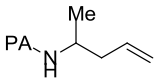
N-allylpicolinamide **(1b)**



The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:6). ^1H NMR (400 MHz, $CDCl_3$) δ 8.55 (m, 1H), 8.21

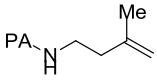
(m, 1H), 8.14 (br, 1H), 7.85 (m, 1H), 7.43 (m, 1H), 6.03–5.87 (m, 1H), 5.31–5.15 (m, 2H), 4.13–4.04 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.31, 150.04, 148.23, 137.50, 134.22, 126.32, 122.44, 116.60, 41.95. GC-MS (EI): Calcd for $C_{11}H_{14}N_2O$: 162.08, found: 162.10.

N-(pent-4-en-2-yl)picolinamide **(1c)**



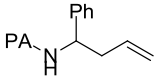
The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (dd, *J* = 2.8, 1.7 Hz, 1H), 8.19–8.12 (m, 1H), 7.92 (br, 1H), 7.80 (m, 1H), 7.41–7.34 (m, 1H), 5.89–5.73 (m, 1H), 5.16–5.01 (m, 2H), 4.23 (m, 1H), 2.33 (d, *J* = 6.7 Hz, 2H), 1.26–1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.55, 150.11, 148.05, 137.38, 134.45, 126.09, 122.23, 117.88, 44.81, 41.02, 20.32. GC-MS (EI): Calcd for $C_{11}H_{14}N_2O$: 190.11, found: 190.15.

N-(3-methylbut-3-en-1-yl)picolinamide **(1d)**



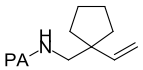
The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57–8.51 (m, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 8.08 (br, 1H), 7.84 (m, 1H), 7.41 (m, 1H), 4.88–4.77 (m, 2H), 3.61 (dd, *J* = 12.9, 6.9 Hz, 2H), 2.35 (t, *J* = 6.9 Hz, 2H), 1.79 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.32, 150.13, 148.18, 142.72, 137.48, 126.19, 122.33, 112.41, 37.62, 37.40, 22.38. GC-MS (EI): Calcd for $C_{11}H_{14}N_2O$: 190.11, found: 190.14.

N-(1-phenylbut-3-en-1-yl)picolinamide **(1e)**



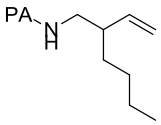
The title compound was isolated as a yellow solid after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (dd, *J* = 4.8, 0.7 Hz, 1H), 8.44 (br, 1H), 8.18 (d, *J* = 7.8 Hz, 1H), 7.83 (m, 1H), 7.46–7.29 (m, 5H), 7.25 (m, 1H), 5.77 (m, 1H), 5.27 (dd, *J* = 15.4, 7.0 Hz, 1H), 5.22–5.04 (m, 2H), 2.77–2.65 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.67, 149.96, 148.18, 141.85, 137.48, 134.08, 128.74, 127.46, 126.70, 126.30, 122.46, 118.38, 52.90, 40.90. GC-MS (EI): Calcd for $C_{16}H_{16}N_2O$: 252.13, found: 252.10.

N-((1-vinylcyclopentyl)methyl)picolinamide **(1f)**



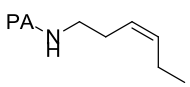
The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (m, 1H), 8.19 (m, 1H), 8.11 (br, 1H), 7.83 (m, 1H), 7.41 (m, 1H), 5.90 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.16–5.07 (m, 2H), 3.47 (d, *J* = 6.1 Hz, 2H), 1.74–1.63 (m, 6H), 1.59 (d, *J* = 6.9 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.51, 150.20, 148.29, 144.41, 137.39, 126.13, 126.10, 122.39, 50.18, 46.55, 35.07, 24.13. GC-MS (EI): Calcd for $C_{14}H_{18}N_2O$: 230.14, found: 230.15.

N-(2-vinylhexyl)picolinamide **(1g)**



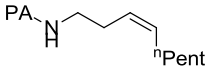
The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (dd, *J* = 2.8, 1.9 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.10 (br, 1H), 7.82 (dd, *J* = 10.7, 4.7 Hz, 1H), 7.48–7.34 (m, 1H), 5.63 (m, 1H), 5.11 (dd, *J* = 13.3, 5.3 Hz, 2H), 3.76–3.49 (m, 1H), 3.34–3.09 (m, 1H), 2.28 (dd, *J* = 8.3, 4.7 Hz, 1H), 1.34 (m, 6H), 0.87 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.29, 150.15, 148.19, 140.49, 137.37, 126.11, 122.29, 116.77, 44.46, 43.35, 32.14, 29.32, 22.79, 14.13. GC-MS (EI): Calcd for $C_{14}H_{20}N_2O$: 232.16, found: 232.15.

(Z)-N-(hex-3-en-1-yl)picolinamide **(1h)**



The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54–8.47 (m, 1H), 8.22–8.15 (m, 1H), 8.11 (br, 1H), 7.89–7.78 (m, 1H), 7.40 (m, 1H), 5.61–5.45 (m, 1H), 5.38 (m, 1H), 3.49 (t, *J* = 6.6 Hz, 2H), 2.51–2.21 (m, 2H), 2.21–1.83 (m, 2H), 0.94 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.34, 150.12, 148.11, 137.45, 134.78, 126.16, 125.14, 122.29, 39.25, 27.51, 20.75, 14.35. GC-MS (EI): Calcd for $C_{12}H_{16}N_2O$: 204.13, found:204.10.

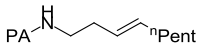
(Z)-N-(non-3-en-1-yl)picolinamide **(Z-1i)**



The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.6 Hz, 1H), 8.16 (dd, *J* = 7.3, 3.4 Hz, 1H), 8.10 (br, 1H), 7.85–7.74 (m, 1H), 7.37 (m, 1H), 5.55–5.32 (m, 2H), 3.52–3.41 (m, 2H), 2.41–2.29 (m, 2H), 2.01 (d, *J* = 6.6 Hz, 2H), 1.24 (dd, *J* = 35.8, 4.7 Hz, 6H), 0.84–0.73 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.27, 150.08, 148.04, 137.34,

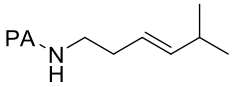
133.12, 126.07, 125.66, 122.20, 39.18, 31.51, 29.36, 27.56, 27.35, 22.59, 14.07. GC-MS (EI): Calcd for $C_{15}H_{22}N_2O$: 246.17, found: 246.15.

(E)-N-(non-3-en-1-yl)picolinamide **(E-1i)**



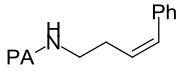
The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 4.7 Hz, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 8.09 (br, 1H), 7.84 (m, 1H), 7.41 (m, 1H), 5.61–5.52 (m, 1H), 5.49–5.39 (m, 1H), 3.50 (dd, *J* = 12.9, 6.8 Hz, 2H), 2.32 (m, 2H), 2.01 (m, 2H), 1.38–1.32 (m, 2H), 1.28–1.22 (m, 4H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.30, 150.24, 148.14, 137.40, 133.86, 126.44, 126.12, 122.28, 39.16, 32.80, 32.73, 31.43, 29.27, 22.67, 14.16. GC-MS (EI): Calcd for $C_{15}H_{22}N_2O$: 246.17, found: 246.20.

(E)-N-(5-methylhex-3-en-1-yl)picolinamide **(1j)**



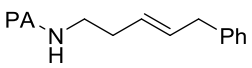
The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 4.7 Hz, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 8.12 (br, 1H), 7.84 (m, 1H), 7.44–7.39 (m, 1H), 5.31 (m, 2H), 3.50 (dd, *J* = 13.1, 6.7 Hz, 2H), 2.61 (m, 1H), 2.39 (m, 2H), 0.93 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.35, 150.15, 148.11, 140.65, 137.40, 126.15, 123.35, 122.25, 39.34, 27.73, 26.72, 23.22. GC-MS (EI): Calcd for $C_{13}H_{18}N_2O$: 218.14, found:218.18.

(Z)-N-(4-phenylbut-3-en-1-yl)picolinamide **(1k)**



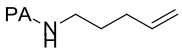
The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (m, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 8.17 (br, 1H), 7.84 (m, 1H), 7.42–7.38 (m, 1H), 7.38–7.33 (m, 2H), 7.30 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.24–7.19 (m, 1H), 6.51 (d, *J* = 15.9 Hz, 1H), 6.24 (m, 1H), 3.63 (m, 2H), 2.56 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.45, 150.08, 148.18, 137.46, 137.44, 132.44, 128.64, 127.34, 127.02, 126.25, 126.21, 122.30, 39.13, 33.33. GC-MS (EI): Calcd for $C_{16}H_{16}N_2O$: 252.13, found: 252.15.

***(E)-N-(5-phenylpent-3-en-1-yl)picolinamide* (1l)**



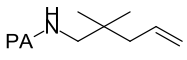
The title compound was isolated as a yellow oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, CDCl3) δ 8.54 (d, *J* = 4.7 Hz, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 8.16 (br, 1H), 7.85 (m, 1H), 7.44–7.41 (m, 1H), 7.22 (dd, *J* = 8.6, 6.1 Hz, 2H), 7.18–7.13 (m, 3H), 5.73 (m, 1H), 5.61–5.54 (m, 1H), 3.57 (dd, *J* = 13.2, 6.8 Hz, 2H), 3.43 (d, *J* = 7.3 Hz, 2H), 2.52 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.42, 150.10, 148.15, 140.74, 137.43, 131.20, 128.51, 128.45, 126.90, 126.18, 126.01, 122.31, 39.18, 33.68, 27.75. GC-MS (EI): Calcd for $C_{15}H_{22}N_2O$: 266.14, found: 266.20.

***N-(pent-4-en-1-yl)picolinamide* (1m)**



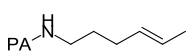
The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:6). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (m, 1H), 8.20 (m, 1H), 8.09 (br, 1H), 7.84 (m, 1H), 7.42 (m, 1H), 5.84 (m, 1H), 5.13–4.94 (m, 2H), 3.49 (dd, *J* = 13.4, 7.0 Hz, 2H), 2.22–2.12 (m, 2H), 1.78–1.70 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.35, 150.13, 148.11, 137.87, 137.52, 126.20, 122.35, 115.37, 39.03, 31.27, 28.93. GC-MS (EI): Calcd for $C_{11}H_{14}N_2O$: 190.11, found: 190.15.

***N-(2,2-dimethylpent-4-en-1-yl)picolinamide* (1n)**



The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:6). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (m, 1H), 8.20 (m, 2H), 7.86–7.81 (m, 1H), 7.42 (m, 1H), 5.92–5.81 (m, 1H), 5.11–5.06 (m, 2H), 3.31 (d, *J* = 6.7 Hz, 2H), 2.06 (d, *J* = 7.5 Hz, 2H), 0.97 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.48, 150.20, 148.19, 137.47, 134.93, 126.17, 122.43, 117.76, 49.10, 44.70, 35.23, 25.17. GC-MS (EI): Calcd for $C_{13}H_{18}N_2O$: 218.14, found: 218.15.

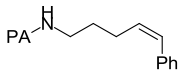
***(E)-N-(hex-4-en-1-yl)picolinamide* (1o)**



The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57–8.51 (m, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 8.07 (br, 1H), 7.84 (m, 1H), 7.41 (m, 1H), 5.57–5.36 (m, 2H),

3.47 (m, 2H), 2.09 (m, 2H), 1.75–1.67 (m, 2H), 1.65 (d, *J* = 4.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.32, 150.21, 148.11, 137.42, 130.36, 126.12, 125.90, 122.27, 39.08, 30.15, 29.49, 18.03. GC-MS (EI): Calcd for $C_{12}H_{16}N_2O$: 204.13, found: 204.15.

***(Z)-N-(5-phenylpent-4-en-1-yl)picolinamide* (1p)**

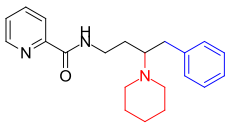


The title compound was isolated as a colorless oil after chromatography on silica with ethyl acetate/hexane (1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.8 Hz, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 8.06 (br, 1H), 7.83 (m, 1H), 7.41 (dd, *J* = 7.5, 4.8 Hz, 1H), 7.28 (m, 4H), 7.20 (t, *J* = 6.9 Hz, 1H), 6.47 (d, *J* = 11.6 Hz, 1H), 5.69 (m, 1H), 3.50 (m, 2H), 2.52–2.38 (m, 2H), 1.83–1.75 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.39, 150.13, 148.12, 137.57, 137.44, 131.70, 129.89, 128.84, 128.30, 126.72, 126.17, 122.29, 39.12, 29.96, 26.11. GC-MS (EI): Calcd for $C_{17}H_{18}N_2O$:266.14, found: 266.20.

3. General Procedure for the Ni-Catalyzed Arylamination of Alkenyl Amines

In an argon-filled glovebox, $NiBr_2$•DME (0.03 mmol, 15 mol%), K_3PO_4 (0.6 mmol, 3.0 eq), alkene substrate (0.2 mmol, 1.0 eq), appropriate amine benzoate electrophile (0.4 mmol, 2 eq), appropriate aryl boronic nucleophile (0.6 mmol, 3.0 eq), *t*-BuOH (2 mL) were added to a 10 mL schlenk flask. The reaction mixture was stirred at 80 ℃ for 24 h and the resulting solution was concentrated in vacuum. The crude product was purified by column chromatography on alumina gel with a mixture of ethyl acetate and hexane as eluent. The conditions for flash chromatography and data for characterization of the products are listed below.

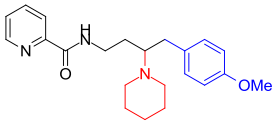
***N-(4-phenyl-3-(piperidin-1-yl)butyl)picolinamide* (2a)**



The title compound was isolated as a colorless oil (80% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.22 (br, 1H), 8.48–8.42 (m, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 7.72 (m, 1H), 7.32–7.28 (m, 1H), 7.19 (t, *J* = 7.4 Hz, 2H), 7.11 (d, *J* = 7.3 Hz, 1H), 7.05 (d, *J* =

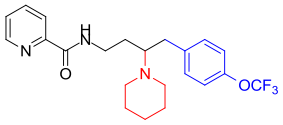
7.2 Hz, 2H), 3.59 (m, 1H), 3.11 (m, 1H), 2.98 (dd, *J* = 13.0, 3.2 Hz, 1H), 2.74 (m, 3H), 2.44 (m, 2H), 2.23 (dd, *J* = 13.0, 10.5 Hz, 1H), 1.73–1.60 (m, 5H), 1.55–1.41 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.60, 150.46, 147.94, 140.57, 137.20, 129.28, 128.54, 126.00, 125.94, 122.23, 68.14, 49.89, 39.53, 34.53, 28.15, 26.01, 25.15. HRMS (ESI) m/z calculated for $C_{21}H_{27}N_3O$ $[M+H]^+$ 338.2232, found 338.2235.

N-(4-(4-methoxyphenyl)-3-(piperidin-1-yl)butyl)picolinamide **(2b)**



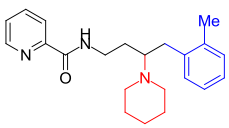
The title compound was isolated as a colorless oil (67% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.30 (br, 1H), 8.56–8.49 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.06 (d, *J* = 7.7 Hz, 2H), 6.85–6.79 (m, 2H), 3.79 (s, 3H), 3.68 (s, 1H), 3.20 (m, 1H), 3.00 (d, *J* = 10.4 Hz, 1H), 2.79 (s, 3H), 2.50 (s, 2H), 2.24 (s, 1H), 1.73 (s, 6H), 1.51 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.68, 156.11, 156.01, 146.03, 135.26, 135.20, 128.23, 123.99, 120.28, 112.14, 53.48, 48.01, 47.95, 37.59, 31.71, 26.34, 24.24, 23.25. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O_2$ $[M+H]^+$ 368.2338, found 368.2339.

N-(3-(piperidin-1-yl)-4-(4-(trifluoromethoxy)phenyl)butyl)picolinamide **(2c)**



The title compound was isolated as a white solid (71% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.14 (br, 1H), 8.52 (d, *J* = 4.6 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.82 (m, 1H), 7.42–7.36 (m, 1H), 7.13 (m, 4H), 3.68 (m, 1H), 3.26 (m, 1H), 3.04 (dd, *J* = 13.2, 3.5 Hz, 1H), 2.78 (dd, *J* = 10.4, 4.5 Hz, 3H), 2.51 (m, 2H), 2.33 (dd, *J* = 13.1, 10.2 Hz, 1H), 1.73 (m, 5H), 1.54 (dd, *J* = 13.8, 5.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.56, 150.53, 147.99, 147.59, 139.58, 137.28, 137.22, 130.51, 130.45, 126.00, 122.31, 121.91, 121.11, 119.36, 67.82, 49.92, 39.30, 34.05, 28.43, 26.20, 25.24. HRMS (ESI) m/z calculated for $C_{22}H_{26}F_3N_3O_2$ $[M+H]^+$ 422.2055, found 422.2060.

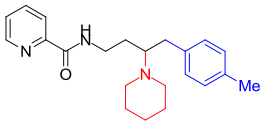
N-(3-(piperidin-1-yl)-4-(o-tolyl)butyl)picolinamide **(2d)**



The title compound was isolated as a colorless oil (84% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400

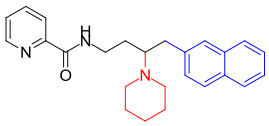
MHz, $CDCl_3$) δ 9.32 (br, 1H), 8.55–8.48 (m, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.15–7.09 (m, 3H), 7.06 (dd, *J* = 6.5, 2.5 Hz, 1H), 3.78–3.66 (m, 1H), 3.19 (m, 1H), 3.07 (dd, *J* = 13.2, 3.0 Hz, 1H), 2.88 (m, 2H), 2.83–2.75 (m, 1H), 2.55 (m, 2H), 2.41–2.34 (m, 1H), 2.33 (s, 3H), 1.76 (m, 5H), 1.61–1.49 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.46, 150.71, 147.91, 138.68, 137.20, 136.17, 130.56, 130.51, 126.19, 126.01, 125.88, 122.28, 66.72, 49.81, 39.76, 31.58, 28.00, 26.21, 25.36, 19.73. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found 352.2389.

N-(3-(piperidin-1-yl)-4-(p-tolyl)butyl)picolinamide **(2e)**



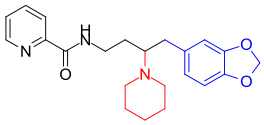
The title compound was isolated as a white solid (75% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.33 (br, 1H), 8.52 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.11–7.02 (m, 4H), 3.69 (dd, *J* = 13.3, 6.9 Hz, 1H), 3.24–3.14 (m, 1H), 3.02 (d, *J* = 12.5 Hz, 1H), 2.87–2.72 (m, 3H), 2.51 (s, 2H), 2.31 (s, 3H), 2.26 (t, *J* = 11.6 Hz, 1H), 1.74 (s, 6H), 1.55–1.48 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.53, 150.66, 147.94, 137.21, 135.45, 129.26, 129.20, 125.91, 122.28, 68.38, 49.93, 39.71, 34.08, 28.09, 26.16, 25.32, 21.15. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found 352.2389.

N-(4-(naphthalen-2-yl)-3-(piperidin-1-yl)butyl)picolinamide **(2f)**



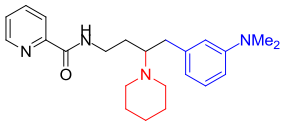
The title compound was isolated as a yellow solid (65% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.13 (br, 1H), 8.51–8.46 (m, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.79–7.74 (m, 3H), 7.61 (s, 1H), 7.50–7.27 (m, 5H), 3.64 (m, 1H), 3.28 (dd, *J* = 13.1, 3.3 Hz, 1H), 3.21 (m, 1H), 3.06 (m, 1H), 2.91 (m, 2H), 2.66 (m, 2H), 2.54 (dd, *J* = 13.0, 10.3 Hz, 1H), 1.95–1.86 (m, 1H), 1.86–1.76 (m, 4H), 1.70–1.63 (m, 1H), 1.58–1.51 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.60, 150.42, 147.96, 137.18, 133.69, 132.15, 128.27, 128.03, 127.75, 127.67, 127.61, 127.50, 126.15, 125.94, 125.42, 122.22, 67.47, 49.92, 39.30, 35.20, 28.53, 25.73, 24.92. HRMS (ESI) m/z calculated for $C_{25}H_{29}N_3O$ $[M+H]^+$ 388.2389, found 388.2390.

N-(4-(benzo[d][1,3]dioxol-5-yl)-3-(piperidin-1-yl)butyl)picolinamide **(2g)**



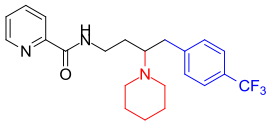
The title compound was isolated as a yellow solid (53% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.28 (br, 1H), 8.52 (d, *J* = 4.4 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (dd, *J* = 6.7, 4.9 Hz, 1H), 6.71 (d, *J* = 7.9 Hz, 1H), 6.62 (s, 1H), 6.58 (d, J = 7.9 Hz, 1H), 5.92 (s, 2H), 3.70 (m, 1H), 3.22 (m, 1H), 2.99–2.94 (m, 1H), 2.83–2.69 (m, 3H), 2.53–2.42 (m, 2H), 2.21 (dd, *J* = 13.0, 10.5 Hz, 1H), 1.77–1.67 (m, 5H), 1.60–1.48 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.55, 150.60, 147.95, 147.76, 145.82, 137.23, 134.54, 125.93, 122.29, 122.12, 109.52, 108.33, 100.93, 68.28, 49.88, 39.63, 34.32, 28.13, 26.18, 25.30. HRMS (ESI) m/z calculated for $C_{22}H_{27}N_3O_3$ $[M+H]^+$ 382.2131, found 382.2131.

N-(4-(3-(dimethylamino)phenyl)-3-(piperidin-1-yl)butyl)picolinamide **(2h)**



The title compound was isolated as a yellow oil (77% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.35 (br, 1H), 8.52 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.38 (m, 1H), 7.14 (t, *J* = 7.9 Hz, 1H), 6.60–6.55 (m, 1H), 6.53 (d, *J* = 6.8 Hz, 2H), 3.71 (m, 1H), 3.20 (m, 1H), 3.05 (dd, *J* = 12.9, 3.0 Hz, 1H), 2.93 (s, 6H), 2.84 (m, 3H), 2.59–2.49 (m, 2H), 2.26 (dd, *J* = 12.8, 10.6 Hz, 1H), 1.83–1.71 (m, 5H), 1.66–1.49 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.54, 150.85, 150.61, 147.92, 141.50, 137.18, 129.18, 125.88, 122.25, 117.71, 113.62, 110.41, 68.20, 49.89, 40.72, 39.76, 34.98, 28.18, 26.03, 25.23. HRMS (ESI) m/z calculated for $C_{23}H_{32}N_4O$ $[M+H]^+$ 381.2654, found 381.2660.

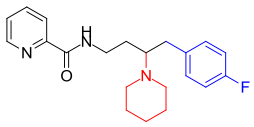
N-(3-(piperidin-1-yl)-4-(4-(trifluoromethyl)phenyl)butyl)picolinamide **(2i)**



The title compound was isolated as a white solid (71% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (br, 1H), 8.49–8.37 (m, 1H), 8.08 (d, *J* = 7.8 Hz, 1H), 7.73 (m, 1H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.32 (m, 1H), 7.21–7.15 (m, 2H), 3.60 (m, 1H), 3.17 (m, 1H), 3.01 (dd, *J* = 13.0, 3.2 Hz, 1H), 2.72 (m, 3H), 2.44 (m, 2H), 2.31 (dd, *J* = 13.0, 10.1 Hz, 1H), 1.75–1.61 (m, 5H), 1.49–1.39 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.57, 150.39, 147.98, 145.06, 137.28, 129.58, 128.51, 128.18, 126.03, 125.76, 125.46, 125.42, 123.06, 122.26,

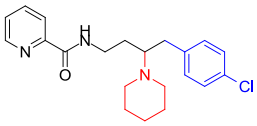
67.72, 49.93, 39.25, 34.64, 28.42, 26.12, 25.17. HRMS (ESI) m/z calculated for $C_{22}H_{26}F_3N_3O$ $[M+H]^+$ 406.2106, found 406.2110.

***N-(4-(4-fluorophenyl)-3-(piperidin-1-yl)butyl)picolinamide* (2j)**



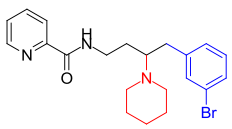
The title compound was isolated as a white solid (78% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.06 (br, 1H), 8.51 (d, *J* = 4.3 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.40–7.36 (m, 1H), 7.11 (dd, *J* = 8.4, 5.5 Hz, 2H), 6.95 (t, *J* = 8.7 Hz, 2H), 3.63 (m, 1H), 3.23 (m, 1H), 3.07 (dd, *J* = 13.2, 3.3 Hz, 1H), 2.85 (m, 3H), 2.57 (m, 2H), 2.34 (dd, *J* = 13.2, 10.2 Hz, 1H), 1.85–1.70 (m, 5H), 1.56 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.59, 150.48, 148.00, 137.27,135.99, 131.70, 130.69, 130.61,129.85, 128.14, 126.00, 122.29, 115.51, 115.30, 67.55, 49.86, 39.28, 34.25, 28.41, 25.86, 25.01. HRMS (ESI) m/z calculated for $C_{21}H_{26}FN_3O$ $[M+H]^+$ 356.2138, found 356.2138.

***N-(4-(4-chlorophenyl)-3-(piperidin-1-yl)butyl)picolinamide* (2k)**



The title compound was isolated as a white solid (79% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.14 (br, 1H), 8.55–8.48 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.3 Hz, 2H), 3.66 (m, 1H), 3.23 (m, 1H), 3.00 (dd, *J* = 13.1, 3.6 Hz, 1H), 2.77 (dd, *J* = 10.7, 5.6 Hz, 3H), 2.50 (m, 2H), 2.29 (dd, *J* = 13.1, 10.1 Hz, 1H), 1.73 (m, 5H), 1.59–1.48 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.58, 150.50, 147.99, 139.22, 137.28, 131.79, 130.63, 128.68, 126.00, 122.29, 67.89, 49.95, 39.36, 34.13, 28.39, 26.15, 25.22. HRMS (ESI) m/z calculated for $C_{21}H_{26}ClN_3O$ $[M+H]^+$ 372.1843, found 372.1842.

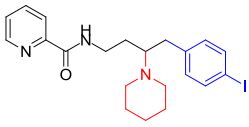
***N-(4-(3-bromophenyl)-3-(piperidin-1-yl)butyl)picolinamide* (2l)**



The title compound was isolated as a colorless oil (80% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.17 (br, 1H), 8.52 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.31 (d, *J* = 8.7 Hz, 2H), 7.13 (t, *J* = 7.7 Hz, 1H), 7.06 (d, *J* = 7.6 Hz, 1H),

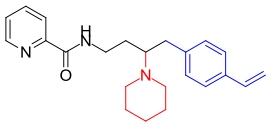
3.69 (m, 1H), 3.24 (m, 1H), 3.02 (d, *J* = 12.5 Hz, 1H), 2.88–2.66 (m, 3H), 2.50 (s, 2H), 2.35–2.22 (m, 1H), 1.73 (s, 6H), 1.54–1.48 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.53, 150.50, 147.97, 137.30, 137.26, 132.24, 130.09, 129.15, 127.99, 125.98, 122.61, 122.28, 67.85, 49.91, 39.35, 34.42, 28.34, 26.14, 25.22. HRMS (ESI) m/z calculated for $C_{21}H_{26}BrN_3O$ $[M+H]^+$ 416.1338, found 416.1337.

N-(4-(4-iodophenyl)-3-(piperidin-1-yl)butyl)picolinamide **(2m)**



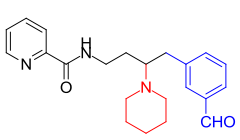
The title compound was isolated as a yellow solid (64% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (br, 1H), 8.52 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.82 (d, *J* = 1.7 Hz, 1H), 7.61–7.54 (m, 2H), 7.42–7.37 (m, 1H), 6.90 (d, *J* = 8.2 Hz, 2H), 3.66 (m, 1H), 3.22 (m, 1H), 3.00 (dd, *J* = 13.1, 3.3 Hz, 1H), 2.78 (dd, *J* = 10.8, 5.3 Hz, 3H), 2.56–2.45 (m, 2H), 2.27 (dd, *J* = 13.0, 10.2 Hz, 1H), 1.84–1.67 (m, 5H), 1.53 (dd, *J* = 10.9, 5.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.61, 150.42, 148.01, 137.61, 137.31, 131.43, 126.05, 122.30, 91.12, 67.81, 49.90, 39.39, 34.37, 28.31, 26.04, 25.15, 1.15. HRMS (ESI) m/z calculated for $C_{21}H_{26}IN_3O$ $[M+H]^+$ 464.1199, found 464.1197.

N-(3-(piperidin-1-yl)-4-(4-vinylphenyl)butyl)picolinamide **(2n)**



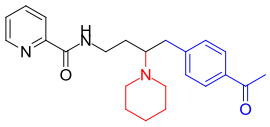
The title compound was isolated as a colorless oil (47% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.28 (br, 1H), 8.52 (d, *J* = 4.1 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.41–7.37 (m, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 6.68 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.70 (d, *J* = 17.6 Hz, 1H), 5.20 (d, *J* = 11.2 Hz, 1H), 3.68 (m, 1H), 3.20 (m, 1H), 3.06 (dd, J = 13.0, 2.8 Hz, 1H), 2.87–2.75 (m, 3H), 2.58–2.46 (m, 2H), 2.35–2.27 (m, 1H), 1.81–1.69 (m, 5H), 1.61–1.50 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.61, 150.51, 147.97, 137.26, 136.68, 136.66, 135.48, 129.55, 129.49, 126.44, 125.98, 122.30, 68.14, 49.93, 39.62, 34.43, 28.20, 26.04, 25.21. HRMS (ESI) m/z calculated for $C_{23}H_{29}N_3O$ $[M+H]^+$ 364.2389, found 364.2389.

N-(4-(3-formylphenyl)-3-(piperidin-1-yl)butyl)picolinamide **(2o)**



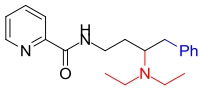
The title compound was isolated as a blue oil (50% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (br, 1H), 9.00 (br, 1H), 8.50 (d, *J* = 4.6 Hz, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.80 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.69 (d, *J* = 4.4 Hz, 2H), 7.44 (d, *J* = 4.8 Hz, 2H), 7.40–7.37 (m, 1H), 3.62 (dd, *J* = 13.4, 6.7 Hz, 1H), 3.26 (m, 1H), 3.21–3.16 (m, 1H), 2.91 (d, *J* = 9.4 Hz, 1H), 2.83 (dd, *J* = 10.5, 5.2 Hz, 2H), 2.65–2.57 (m, 2H), 2.48 (dd, *J* = 12.8, 10.3 Hz, 1H), 1.87 (dd, *J* = 14.5, 5.9 Hz, 1H), 1.76 (d, *J* = 5.1 Hz, 4H), 1.58–1.49 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.57, 164.63, 150.29, 148.03, 137.32, 136.76, 135.57, 130.11, 130.06, 129.31, 128.10, 126.09, 122.28, 67.30, 49.94, 39.05, 34.85, 28.49, 25.82, 24.93. HRMS (ESI) m/z calculated for $C_{23}H_{29}N_3O$ $[M+H]^+$ 366.2182, found 366.2185.

N-(4-(4-acetylphenyl)-3-(piperidin-1-yl)butyl)picolinamide **(2p)**



The title compound was isolated as a white solid (76% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.00 (br, 1H), 8.51 (d, *J* = 4.2 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.88 (s, 1H), 7.82 (m, 1H), 7.49–7.42 (m, 1H), 7.40 (m, 1H), 7.27 (d, *J* = 4.4 Hz, 2H), 3.62 (dd, *J* = 13.6, 6.8 Hz, 1H), 3.25 (m, 1H), 3.17 (dd, *J* = 12.9, 3.5 Hz, 1H), 2.97 (t, *J* = 9.7 Hz, 1H), 2.92–2.82 (m, 2H), 2.63 (d, *J* = 5.2 Hz, 2H), 2.58 (s, 3H), 2.48 (dd, *J* = 12.9, 10.2 Hz, 1H), 1.91–1.73 (m, 5H), 1.62–1.52 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.75, 164.73, 148.10, 137.38, 137.33, 130.11, 129.59, 128.85, 128.38, 126.14, 122.28, 66.83, 50.02, 38.82, 29.83, 28.95, 26.68, 26.62, 25.42. HRMS (ESI) m/z calculated for $C_{23}H_{29}N_3O_2$ $[M+H]^+$ 380.2338, found: 380.2431.

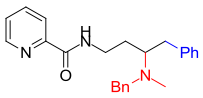
N-(3-(diethylamino)-4-phenylbutyl)picolinamide **(3a)**



The title compound was isolated as a yellow oil (70% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.02 (br, 1H), 8.54–8.47 (m, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.79 (m, 1H), 7.36 (m, 1H), 7.28–7.23 (m, 2H), 7.15 (dd, *J* = 19.4, 7.2 Hz, 3H), 3.59 (m, 1H), 3.35–3.24 (m, 1H), 2.97 (d, *J* = 10.5 Hz, 2H), 2.76 (m, 2H), 2.47 (dd, *J* = 12.8, 6.6 Hz, 2H), 2.31 (t, *J* = 12.3 Hz, 1H), 1.75–1.65 (m, 1H), 1.60 (m, 1H), 1.14 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.37,

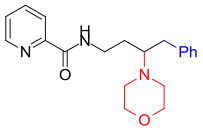
150.57, 148.01, 137.22, 137.16, 129.32, 128.56, 125.98, 125.85, 122.20, 62.15, 43.46, 39.14, 35.18, 29.23, 14.41. HRMS (ESI) m/z calculated for $C_{20}H_{27}N_3O$ $[M+H]^+$ 326.2232, found: 326.2240.

N-(3-(benzyl(methyl)amino)-4-phenylbutyl)picolinamide **(3b)**



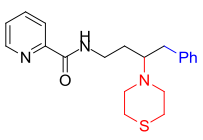
The title compound was isolated as a yellow oil (75% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (br, 1H), 8.47–8.42 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.40–7.36 (m, 3H), 7.29 (d, *J* = 6.9 Hz, 2H), 7.25–7.20 (m, 3H), 7.17 (dd, *J* = 8.4, 6.3 Hz, 1H), 7.12 (d, *J* = 7.0 Hz, 2H), 3.82 (d, *J* = 13.2 Hz, 1H), 3.70 (d, *J* = 13.2 Hz, 1H), 3.57 (m, 1H), 3.40–3.32 (m, 1H), 3.08 (dd, *J* = 13.0, 3.9 Hz, 1H), 2.94 (m, 1H), 2.40 (dd, *J* = 13.0, 9.8 Hz, 1H), 2.32 (s, 3H), 1.79 (m, 1H), 1.61 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.25, 150.43, 148.12, 140.44, 137.25, 129.34, 129.09, 128.54, 128.39, 127.08, 126.96, 126.03, 125.91, 122.19, 63.71, 59.19, 38.34, 35.86, 34.26, 29.93. HRMS (ESI) m/z calculated for $C_{24}H_{27}N_3O$ $[M+H]^+$ 374.2232, found: 374.2232.

N-(3-morpholino-4-phenylbutyl)picolinamide **(3c)**



The title compound was isolated as a yellow oil (82% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.09 (br, 1H), 8.59–8.52 (m, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.83 (m, 1H), 7.42 (s, 1H), 7.28 (t, *J* = 7.4 Hz, 2H), 7.20 (d, *J* = 7.3 Hz, 1H), 7.15 (d, *J* = 7.1 Hz, 2H), 3.89 (t, *J* = 4.4 Hz, 4H), 3.76–3.65 (m, 2H), 3.27 (m, 1H), 3.10 (d, *J* = 13.1 Hz, 1H), 2.87 (dd, *J* = 10.3, 5.3 Hz, 2H), 2.68–2.58 (m, 2H), 2.42–2.32 (m, 1H), 1.84–1.70 (m, 1H), 1.64 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.52, 150.30, 148.09, 137.41, 129.29, 128.68, 128.48, 126.25, 126.15, 122.37, 67.54, 67.13, 48.96, 39.24, 34.72, 28.27. HRMS (ESI) m/z calculated for $C_{20}H_{25}N_3O_2$ $[M+H]^+$ 340.2025, found: 340.2030.

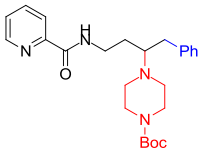
N-(4-phenyl-3-thiomorpholinobutyl)picolinamide **(3d)**



The title compound was isolated as a colorless oil (60% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400

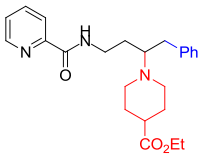
MHz, $CDCl_3$) δ 9.33 (br, 1H), 8.66–8.62 (m, 1H), 8.18 (d, *J* = 7.8 Hz, 1H), 8.15–8.05 (m, 1H), 7.83 (m, 1H), 7.42 (m, 1H), 7.28 (s, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 7.0 Hz, 2H), 3.76–3.67 (m, 1H), 3.18 (m, 3H), 3.06 (dd, *J* = 13.1, 3.6 Hz, 1H), 2.88 (s, 7H), 2.35 (dd, *J* = 13.1, 10.3 Hz, 1H), 1.77 (m, 1H), 1.59 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.52, 150.25, 148.11, 137.43, 130.21, 129.30, 128.67, 128.51, 126.18, 122.33, 69.59, 51.61, 39.67, 35.16, 35.13, 27.98. HRMS (ESI) m/z calculated for $C_{20}H_{25}N_3OS$ $[M+H]^+$ 356.1797, found: 356.1796.

tert-butyl 4-(1-phenyl-4-(picolinamido)butan-2-yl)piperazine-1-carboxylate **(3e)**



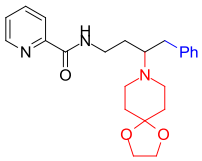
The title compound was isolated as a white solid (61% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (br, 1H), 8.50–8.43 (m, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.80 (m, 1H), 7.42–7.34 (m, 2H), 7.24 (d, *J* = 1.5 Hz, 1H), 7.20–7.14 (m, 1H), 7.14–7.08 (m, 2H), 3.75–3.50 (m, 5H), 3.24 (m, 1H), 3.00 (dd, *J* = 13.1, 3.5 Hz, 1H), 2.94–2.66 (m, 3H), 2.62–2.43 (m, 2H), 2.31 (dd, *J* = 13.0, 10.2 Hz, 1H), 1.76 (dd, *J* = 10.0, 4.9 Hz, 1H), 1.61 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.58, 155.06, 150.13, 148.02, 137.47, 137.42, 129.23, 128.66, 126.25, 126.19, 122.34, 79.85, 67.48, 48.46, 48.38, 39.31, 34.78, 28.55, 28.35. HRMS (ESI) m/z calculated for $C_{25}H_{34}N_4O_3$ $[M+H]^+$ 439.2709, found: 439.2709.

ethyl 1-(1-phenyl-4-(picolinamido)butan-2-yl)piperidine-4-carboxylate **(3f)**



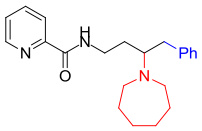
The title compound was isolated as a colorless oil (70% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.47–9.33 (m, 1H), 8.63 (d, *J* = 4.1 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.80 (m, 1H), 7.39 (m, 1H), 7.28 (d, *J* = 7.5 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.1 Hz, 2H), 4.19 (m, 2H), 3.84–3.71 (m, 1H), 3.19–3.01 (m, 3H), 2.94–2.82 (m, 2H), 2.73 (m, 1H), 2.39–2.24 (m, 3H), 2.09 (m, 2H), 1.95 (dd, *J* = 24.1, 12.3 Hz, 2H), 1.75 (m, 1H), 1.61 (d, *J* = 3.1 Hz, 1H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.59, 164.51, 150.50, 148.37, 140.50, 137.12, 129.29, 128.60, 126.09, 125.93, 122.14, 68.35, 60.38, 51.77, 45.17, 42.12, 34.57, 28.84, 27.96, 14.41. HRMS (ESI) m/z calculated for $C_{24}H_{31}N_3O_3$ $[M+H]^+$ 410.2444, found: 410.2444.

***N-(4-phenyl-3-(1,4-dioxa-8-azaspiro[4.5]decan-8-yl)butyl)picolinamide* (3g)**



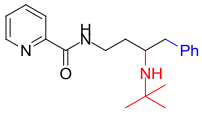
The title compound was isolated as a white solid (69% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76–9.62 (m, 1H), 8.64 (d, *J* = 4.2 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.80 (m, 1H), 7.42–7.36 (m, 1H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 4.00 (s, 4H), 3.83–3.76 (m, 1H), 3.15–3.05 (m, 2H), 2.99 (m, 2H), 2.89 (t, *J* = 10.6 Hz, 1H), 2.71–2.58 (m, 2H), 2.31 (dd, *J* = 12.8, 10.9 Hz, 1H), 2.07–1.93 (m, 4H), 1.73 (m, 1H), 1.60 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.51, 150.54, 148.26, 140.53, 137.15, 129.31, 128.61, 126.07, 125.96, 122.11, 108.02, 68.41, 64.37, 40.17, 40.12, 34.98, 34.61, 27.59. HRMS (ESI) m/z calculated for $C_{23}H_{29}N_3O_3$ $[M+H]^+$ 396.2287, found: 396.2287.

***N-(3-(azepan-1-yl)-4-phenylbutyl)picolinamide* (3h)**



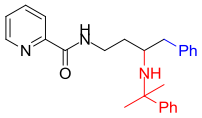
The title compound was isolated as a colorless oil (70% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (br, 1H), 8.55–8.49 (m, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.26 (d, *J* = 14.6 Hz, 2H), 7.19–7.12 (m, 3H), 3.55 (m, 1H), 3.42 (m, 1H), 2.98 (dd, *J* = 13.0, 3.9 Hz, 1H), 2.92–2.84 (m, 3H), 2.67 (m, 2H), 2.39 (dd, *J* = 13.0, 9.7 Hz, 1H), 1.71 (m, 6H), 1.66–1.60 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.47, 150.44, 148.02, 137.28, 137.21, 129.31, 128.49, 128.42, 125.96, 122.27, 67.99, 51.75, 38.89, 35.73, 30.25, 29.43, 27.03. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found: 352.2391.

***N-(3-(tert-butylamino)-4-phenylbutyl)picolinamide* (3i)**



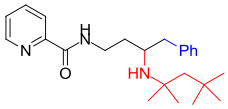
The title compound was isolated as a yellow oil (76% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.26 (br, 1H), 8.52 (m, 1H), 8.19 (m, 1H), 7.82 (m, 1H), 7.38 (m, 1H), 7.31–7.26 (m, 2H), 7.25–7.14 (m, 3H), 3.63–3.52 (m, 2H), 3.13–2.99 (m, 1H), 2.81 (dd, *J* = 13.3, 6.2 Hz, 1H), 2.69 (dd, *J* = 13.3, 7.4 Hz, 1H), 1.86–1.74 (m, 1H), 1.56 (dd, *J* = 13.9, 6.2 Hz, 1H), 1.08 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.28, 150.61, 148.11, 139.46, 137.26, 129.53, 128.56, 126.44, 125.91, 122.28, 53.04, 51.34, 43.84, 37.46, 34.99, 29.95. HRMS (ESI) m/z calculated for $C_{20}H_{27}N_3O$ $[M+H]^+$ 326.2232, found: 326.2333.

***N-(4-phenyl-3-((2-phenylpropan-2-yl)amino)butyl)picolinamide* (3j)**



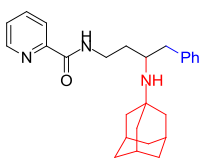
The title compound was isolated as a yellow oil (68% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (br, 1H), 8.55 (m, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 7.84 (m, 1H), 7.43 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.42–7.38 (m, 1H), 7.30–7.24 (m, 3H), 7.22–7.16 (m, 3H), 7.03–6.88 (m, 2H), 3.62–3.36 (m, 2H), 2.91–2.80 (m, 1H), 2.49 (dd, *J* = 11.3, 7.0 Hz, 2H), 1.58 (m, 1H), 1.51 (d, *J* = 10.1 Hz, 6H), 1.46–1.39 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.26, 150.56, 148.10, 147.80, 139.40, 137.30, 129.39, 128.47, 128.15, 126.54, 126.25, 126.20, 125.95, 122.28, 56.00, 53.42, 42.77, 36.85, 33.94, 30.46. HRMS (ESI) m/z calculated for $C_{25}H_{29}N_3O$ $[M+H]^+$ 388.2389, found: 388.2389.

***N-(4-phenyl-3-((2,4,4-trimethylpentan-2-yl)amino)butyl)picolinamide* (3k)**



The title compound was isolated as a colorless oil (80% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (br, 1H), 8.46 (d, *J* = 4.7 Hz, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 7.75 (m, 1H), 7.41–7.24 (m, 2H), 7.22–7.18 (m, 2H), 7.15–7.09 (m, 3H), 3.55–3.42 (m, 2H), 3.04 (m, 1H), 2.71 (dd, *J* = 13.5, 6.3 Hz, 1H), 2.63 (dd, *J* = 13.3, 7.3 Hz, 1H), 1.77–1.70 (m, 1H), 1.53 (dd, *J* = 13.9, 6.3 Hz, 1H), 1.41 (d, *J* = 14.4 Hz, 1H), 1.28 (d, *J* = 14.3 Hz, 1H), 1.19 (d, *J* = 6.0 Hz, 1H), 1.07 (s, 3H), 1.00 (s, 3H), 0.88 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.42, 150.37, 148.11, 139.35, 137.29, 129.51, 128.56, 126.47, 126.00, 122.28, 55.96, 52.32, 43.55, 37.10, 35.23, 32.02, 31.60, 29.05, 27.90. HRMS (ESI) m/z calculated for $C_{24}H_{35}N_3O$ $[M+H]^+$ 382.2858, found: 388.2866.

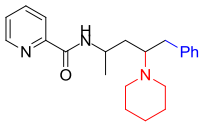
***N-(3-(((3s,5s,7s)-adamantan-1-yl)amino)-4-phenylbutyl)picolinamide* (3l)**



The title compound was isolated as a yellow oil (61% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.24 (br, 1H), 8.55–8.49 (m, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 7.83 (m, 1H), 7.40 (m, 1H), 7.30 (t, *J* = 7.2 Hz, 2H), 7.24–7.17 (m, 3H), 3.57 (m, 2H), 3.20 (m, 1H), 2.81 (dd, *J* = 13.3, 6.1 Hz, 1H), 2.68 (dd, *J* = 13.3, 7.4 Hz, 1H), 2.02 (s,

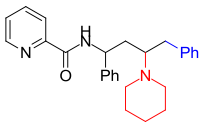
3H), 1.82–1.75 (m, 1H), 1.59 (dd, *J* = 30.3, 12.6 Hz, 14H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.27, 150.63, 148.10, 139.42, 137.29, 129.58, 128.56, 126.46, 125.94, 122.31, 51.59, 50.91, 43.69, 43.60, 37.57, 36.76, 35.26, 29.76. HRMS (ESI) m/z calculated for $C_{26}H_{33}N_3O$ $[M+H]^+$ 404.2702, found: 404.2702.

N-(5-phenyl-4-(piperidin-1-yl)pentan-2-yl)picolinamide **(4a)**



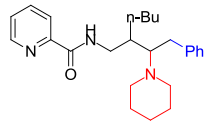
The title compound was isolated as a colorless oil (51% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.70 (d, *J* = 7.4 Hz, 1H), 8.52 (d, *J* = 4.2 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.41–7.36 (m, 1H), 7.27 (d, *J* = 6.1 Hz, 2H), 7.17 (dd, *J* = 18.8, 7.2 Hz, 3H), 4.30 (m, 1H), 3.05 (t, J = 12.4 Hz, 2H), 2.83 (m, 2H), 2.62–2.42 (m, 2H), 2.32–2.20 (m, 1H), 1.88 (m, 1H), 1.79–1.74 (m, 3H), 1.56–1.49 (m, 2H), 1.40–1.34 (m, 1H), 1.27–1.25 (m, 1H), 1.08 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.69, 150.67, 148.06, 141.80, 137.21, 128.99, 128.41, 125.95, 125.88, 122.40, 71.50, 52.32, 46.59, 34.55, 26.20, 24.94, 21.63, 12.44. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found: 352.2390.

N-(1,4-diphenyl-3-(piperidin-1-yl)butyl)picolinamide **(4b)**



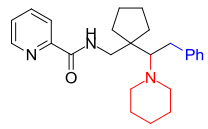
The title compound was isolated as a yellow solid (41% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.45 (d, *J* = 7.6 Hz, 1H), 8.59 (d, *J* = 4.4 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.83 (m, 1H), 7.43 (dd, *J* = 6.7, 4.8 Hz, 1H), 7.14 (d, *J* = 6.3 Hz, 6H), 6.99 (d, *J* = 6.4 Hz, 2H), 6.86–6.77 (m, 2H), 5.37 (m, 1H), 2.94 (dd, *J* = 13.2, 3.0 Hz, 1H), 2.85–2.74 (m, 2H), 2.69 (t, *J* = 10.4 Hz, 1H), 2.49–2.37 (m, 2H), 2.25 (dd, *J* = 13.0, 11.2 Hz, 1H), 2.11 (m, 1H), 1.93–1.78 (m, 4H), 1.76–1.70 (m, 1H), 1.57 (d, *J* = 5.5 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.40, 150.74, 147.93, 141.98, 140.16, 137.17, 129.02, 128.35, 128.24, 126.41, 126.03, 125.96, 125.78, 122.56, 62.94, 52.36, 49.99, 33.57, 33.44, 26.03, 25.49. HRMS (ESI) m/z calculated for $C_{27}H_{31}N_3O$ $[M+H]^+$ 414.2545, found: 414.2545.

N-(2-(2-phenyl-1-(piperidin-1-yl)ethyl)hexyl)picolinamide **(4c)**



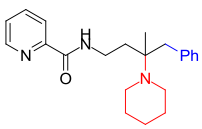
The title compound was isolated as a colorless oil (62% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 1.9:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. The following analytical data correspond to the mixture. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.56 (s, 0.35H), 8.89 (s, 0.65H), 8.54 (d, *J* = 4.7 Hz, 0.35H), 8.51 (d, *J* = 4.2 Hz, 0.65H), 8.17 (d, *J* = 7.9 Hz, 2H), 7.82 (m, 1H), 7.47 (s, 1H), 7.41–7.38 (m, 1H), 7.23–7.16 (m, 3H), 3.90 (dd, *J* = 13.6, 7.3 Hz, 0.36H), 3.61–3.53 (m, 0.65H), 3.45–3.36 (m, 0.65H), 3.18–3.12 (m, 0.35H), 3.06–2.98 (m, 1H), 2.98–2.75 (m, 2H), 2.74–2.53 (m, 4H), 1.75 (dd, *J* = 10.7, 5.8 Hz, 3H), 1.52–1.40 (m, 3H), 1.38–1.20 (m, 7H), 0.89–0.84 (m, 2H), 0.82 (d, *J* = 7.1 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.53, 165.18, 158.68, 158.56, 148.33, 148.09, 137.42, 137.29, 135.39, 135.14, 130.00, 129.67, 129.12, 129.01, 128.67, 127.91, 127.29, 126.30, 122.41, 122.34, 69.30, 69.26, 52.85, 52.79, 41.70, 41.63, 39.06, 39.04, 33.90, 33.86, 32.36, 32.33, 30.24, 29.90, 29.83, 29.55, 25.88, 25.64, 23.00, 22.93, 14.15, 14.07. HRMS (ESI) m/z calculated for $C_{25}H_{35}N_3O$ $[M+H]^+$ 394.2858, found: 394.2859.

***N*-((1-(2-phenyl-1-(piperidin-1-yl)ethyl)cyclopentyl)methyl)picolinamide (4d)**



The title compound was isolated as a colorless oil (44% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (br, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 7.7 Hz, 1H), 7.71 (dd, *J* = 5.6, 3.4 Hz, 1H), 7.53 (dd, *J* = 5.6, 3.3 Hz, 1H), 7.44–7.35 (m, 2H), 7.20 (dd, *J* = 13.3, 6.5 Hz, 3H), 4.21 (d, *J* = 6.2 Hz, 1H), 3.57–3.47 (m, 2H), 2.81 (t, *J* = 6.6 Hz, 1H), 2.66 (d, *J* = 7.5 Hz, 1H), 1.69 (dd, *J* = 13.6, 7.7 Hz, 4H), 1.30 (dd, *J* = 22.6, 8.9 Hz, 8H), 0.95–0.87 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.88, 148.15, 142.29, 137.46, 131.00, 128.96, 128.58, 128.48, 126.17, 122.35, 68.33, 60.52, 38.93, 30.55, 29.44, 29.10, 28.89, 23.94, 23.13. HRMS (ESI) m/z calculated for $C_{25}H_{33}N_3O$ $[M+H]^+$ 392.2702, found: 392.2710.

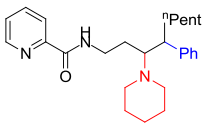
***N*-(3-methyl-4-phenyl-3-(piperidin-1-yl)butyl)picolinamide (4e)**



The title compound was isolated as a yellow oil (79% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.00 (br, 1H), 8.52 (d, *J* = 4.5 Hz, 1H), 8.18 (d, *J* = 7.8 Hz, 1H),

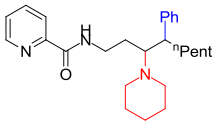
7.82 (m, 1H), 7.39 (dd, *J* = 6.9, 4.5 Hz, 1H), 7.26–7.13 (m, 5H), 3.58 (m, 1H), 3.46–3.36 (m, 1H), 2.90–2.74 (m, 2H), 2.69 (t, *J* = 9.1 Hz, 4H), 2.05 (dd, *J* = 14.9, 7.4 Hz, 1H), 1.75–1.64 (m, 4H), 1.48 (dd, *J* = 10.3, 4.2 Hz, 3H), 1.08 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.05, 150.98, 147.82, 140.93, 137.14, 129.25, 128.54, 125.94, 125.82, 122.32, 63.67, 49.91, 44.22, 34.29, 33.26, 26.03, 25.41, 19.53. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found: 352.2384.

***N-(4-phenyl-3-(piperidin-1-yl)nonyl)picolinamide* (4f)**



The title compound was isolated as a colorless oil (70% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.3 Hz, 1H), 8.22 (br, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.81 (m, 1H), 7.40–7.37 (m, 1H), 7.25 (t, *J* = 7.3 Hz, 2H), 7.20–7.11 (m, 3H), 3.22 (m, 2H), 2.74–2.65 (m, 5H), 1.73–1.66 (m, 1H), 1.63–1.58 (m, 3H), 1.54–1.45 (m, 3H), 1.35 (d, *J* = 15.2 Hz, 1H), 1.27 (d, *J* = 12.1 Hz, 2H), 1.23–1.14 (m, 4H), 1.02–0.94 (m, 2H), 0.90–0.84 (m, 1H), 0.80 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.18, 150.39, 148.00, 137.33, 132.29, 128.78, 128.57, 126.17, 125.99, 122.26, 68.42, 50.39, 39.19, 34.32, 32.07, 29.83, 29.08, 27.28, 26.95, 25.35, 22.68, 14.19. HRMS (ESI) m/z calculated for $C_{26}H_{37}N_3O$ $[M+H]^+$ 408.3015, found: 408.3023.

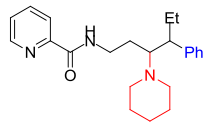
***N-(4-phenyl-3-(piperidin-1-yl)nonyl)picolinamide* (4g)**



The title compound was isolated as a colorless oil (72% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (br, 1H), 8.53–8.50 (m, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 7.82 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.41–7.37 (m, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 7.2 Hz, 3H), 3.66 (dd, *J* = 13.4, 6.6 Hz, 1H), 3.43 (dd, *J* = 11.9, 6.8 Hz, 1H), 2.83 (dd, *J* = 10.2, 4.8 Hz, 1H), 2.79–2.70 (m, 1H), 2.57 (m, 2H), 2.47–2.36 (m, 2H), 1.84 (m, 2H), 1.63 (d, *J* = 8.9 Hz, 2H), 1.54–1.49 (m, 3H), 1.41–1.36 (m, 2H), 1.29–1.25 (m, 2H), 1.19 (dd, *J* = 7.2, 2.7 Hz, 3H), 1.05 (d, *J* = 6.9 Hz, 2H), 0.82–0.78 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.42,

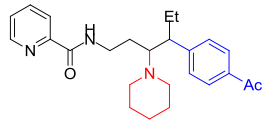
147.89, 145.29, 137.18, 137.13, 128.31, 128.11, 125.86, 125.75, 122.16, 70.01, 50.60, 45.93, 39.35, 32.05, 31.82, 27.39, 27.22, 26.51, 25.07, 22.55, 14.06. HRMS (ESI) m/z calculated for $C_{26}H_{37}N_3O$ $[M+H]^+$ 408.3015, found: 408.3019.

N-(4-phenyl-3-(piperidin-1-yl)hexyl)picolinamide **(4h)**



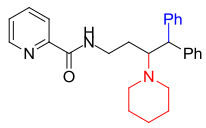
The title compound was isolated as a colorless oil (79% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 4.2 Hz, 1H), 8.15 (br, 1H), 8.06 (d, *J* = 7.8 Hz, 1H), 7.72 (m, 1H), 7.30 (m, 1H), 7.18 (t, *J* = 7.4 Hz, 2H), 7.10 (d, *J* = 7.3 Hz, 1H), 7.07–7.01 (m, 2H), 3.13 (m, 2H), 2.75–2.43 (m, 6H), 1.63 (m, 1H), 1.52 (m, 4H), 1.41 (m, 3H), 1.28 (m, 1H), 1.24–1.15 (m, 1H), 0.56 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.43, 150.18, 148.05, 137.43, 137.33, 128.84, 128.50, 126.32, 126.06, 122.24, 68.23, 51.47, 50.33, 39.06, 28.96, 27.25, 26.73, 25.15, 12.23. HRMS (ESI) m/z calculated for $C_{23}H_{31}N_3O[M+H]^+$ 366.2545, found: 366.2555.

N-(4-(4-acetylphenyl)-3-(piperidin-1-yl)hexyl)picolinamide **(4i)**



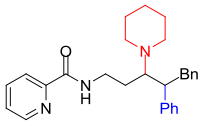
The title compound was isolated as a white solid (51% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 15:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.2 Hz, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 8.12–8.09 (m, 1H), 7.85 (d, *J* = 8.2 Hz, 2H), 7.80 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.39 (dd, *J* = 7.0, 5.2 Hz, 1H), 7.23 (d, *J* = 8.2 Hz, 2H), 3.28 (dd, *J* = 18.1, 6.9 Hz, 2H), 2.86–2.59 (m, 6H), 2.57 (s, 3H), 2.11–2.02 (m, 1H), 1.69 (dd, *J* = 14.8, 7.1 Hz, 1H), 1.58 (d, *J* = 4.9 Hz, 4H), 1.54–1.50 (m, 1H), 1.48 (d, *J* = 5.5 Hz, 2H), 1.29 (m, 2H), 0.63 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.96, 164.21, 150.29, 150.23, 148.04, 137.39, 135.44, 129.04, 128.68, 126.09, 122.25, 68.01, 51.95, 50.45, 39.02, 29.37, 27.10, 26.96, 26.65, 25.29, 12.21. HRMS (ESI) m/z calculated for $C_{25}H_{33}N_3O_2$ $[M+H]^+$ 408.2651, found: 408.2650.

N-(4,4-diphenyl-3-(piperidin-1-yl)butyl)picolinamide **(4j)**



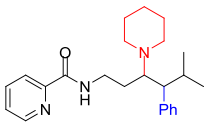
The title compound was isolated as a colorless oil (72% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (br, 1H), 8.50 (s, 1H), 8.31 (d, *J* = 2.7 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.84 (d, *J* = 6.9 Hz, 1H), 7.39 (d, *J* = 7.3 Hz, 2H), 7.30 (d, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.4 Hz, 3H), 7.14 (m, 3H), 4.06 (d, *J* = 10.3 Hz, 1H), 3.71 (m, 1H), 3.53 (d, *J* = 6.5 Hz, 1H), 3.45 (d, *J* = 6.2 Hz, 1H), 2.62 (s, 2H), 2.48 (s, 2H), 1.45 (s, 5H), 1.29–1.25 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.00, 149.75, 148.24, 146.66, 137.45, 129.07, 128.60, 128.23, 127.74, 122.36, 67.30, 38.27, 32.69, 30.93, 30.41, 29.83, 26.51. HRMS (ESI) m/z calculated for $C_{27}H_{31}N_3O$ $[M+H]^+$ 414.2545, found: 414.2540.

N-(4,5-diphenyl-3-(piperidin-1-yl)pentyl)picolinamide **(4k)**



The title compound was isolated as a colorless oil (71% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 8.05 (br, 1H), 7.81 (m, 1H), 7.39 (m, 1H), 7.19–7.01 (m, 8H), 6.87 (d, *J* = 6.8 Hz, 2H), 3.50 (dd, *J* = 13.3, 3.2 Hz, 1H), 3.29 (dd, *J* = 13.3, 7.1 Hz, 1H), 3.15 (dd, *J* = 13.6, 7.1 Hz, 1H), 2.93 (d, *J* = 7.0 Hz, 1H), 2.85 (s, 1H), 2.76 (m, 5H), 1.79 (dd, *J* = 14.2, 6.7 Hz, 1H), 1.64 (d, *J* = 4.8 Hz, 4H), 1.56–1.48 (m, 2H), 1.44–1.36 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.17, 150.25, 148.00, 143.24, 141.38, 137.35, 129.14, 129.02, 128.33, 127.93, 126.33, 126.04, 125.52, 122.23, 67.13, 52.17, 50.31, 41.02, 39.04, 28.69, 26.99, 25.31. HRMS (ESI) m/z calculated for $C_{28}H_{33}N_3O$ $[M+H]^+$ 428.2702, found: 428.2712.

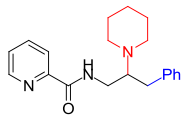
N-(5-methyl-4-phenyl-3-(piperidin-1-yl)hexyl)picolinamide **(4l)**



The title compound was isolated as a colorless oil (71% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 10:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. The following analytical data correspond to the mixture. ^{1}H NMR (400 MHz,

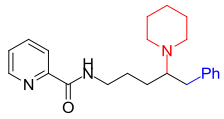
$CDCl_3$) δ 8.53 (d, *J* = 4.7 Hz, 0.09H), 8.50 (d, *J* = 4.6 Hz, 0.91H), 8.21 (d, *J* = 7.8 Hz, 0.09H), 8.16 (d, *J* = 7.8 Hz, 0.91H), 8.13 (br, 0.74H), 7.85 (dd, *J* = 7.8, 1.7 Hz, 0.09H), 7.81 (m, 0.91H), 7.43–7.40 (m, 0.09H), 7.38 (m, 0.91H), 7.25–7.16 (m, 3H), 7.10 (d, *J* = 6.9 Hz, 2H), 3.48–3.22 (m, 2H), 3.03 (m, 1H), 2.67 (t, *J* = 4.8 Hz, 5H), 2.27 (dd, *J* = 11.9, 6.7 Hz, 1H), 1.74 (dd, *J* = 14.5, 6.3 Hz, 1H), 1.66–1.49 (m, 4H), 1.45 (t, *J* = 14.2 Hz, 2H), 1.40 – 1.33 (m, 1H), 1.25 (s, 1H), 0.77 (d, *J* = 6.8 Hz, 3H), 0.72 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.19, 150.34, 148.01, 141.17, 137.33, 130.18, 127.87, 126.18, 126.02, 122.24, 63.94, 54.89, 50.36, 39.31, 29.26, 27.06, 25.36, 21.99, 17.58. HRMS (ESI) m/z calculated for $C_{24}H_{33}N_3O$ $[M+H]^+$ 380.2702, found: 380.2720.

N-(3-phenyl-2-(piperidin-1-yl)propyl)picolinamide **(4m)**



The title compound was isolated as a colorless oil (46% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54–8.49 (m, 1H), 8.20–8.16 (m, 1H), 8.01 (br, 1H), 7.83 (m, 1H), 7.40 (m, 1H), 7.27–7.21 (m, 2H), 7.20–7.07 (m, 3H), 3.37 (d, *J* = 6.9 Hz, 1H), 3.07–2.92 (m, 1H), 2.66 (s, 2H), 2.45 (s, 1H), 2.34–2.22 (m, 1H), 1.79–1.65 (m, 2H), 1.54 (dd, *J* = 13.0, 6.6 Hz, 5H), 1.48–1.36 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.29, 150.28, 148.05, 137.47, 137.38, 129.30, 128.36, 126.19, 126.06, 122.28, 66.64, 49.71, 39.57, 35.34, 27.98, 26.97. HRMS (ESI) m/z calculated for $C_{20}H_{25}N_3O$ $[M+H]^+$ 324.2076, found: 324.2080.

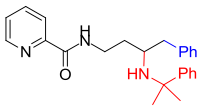
N-(5-phenyl-4-(piperidin-1-yl)pentyl)picolinamide **(4n)**



The title compound was isolated as a colorless oil (81% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.6 Hz, 1H), 8.18 (d, *J* = 7.9 Hz, 1H), 8.01 (br, 1H), 7.83 (m, 1H), 7.40 (dd, *J* = 6.9, 5.1 Hz, 1H), 7.26–7.20 (m, 2H), 7.18–7.08 (m, 3H), 3.37 (m, 2H), 3.00 (dd, *J* = 13.1, 4.1 Hz, 1H), 2.76–2.58 (m, 3H), 2.53–2.41 (m, 2H), 2.30 (dd, *J* = 13.0, 9.4 Hz, 1H), 1.71–1.52 (m, 6H), 1.48–1.22 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.30, 150.30, 148.05, 137.37, 129.30, 128.36, 126.05, 125.74, 122.29, 122.24, 66.64, 49.68, 39.58,

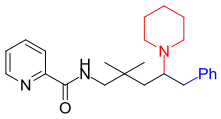
35.37, 27.99, 26.98, 26.68, 25.18. HRMS (ESI) m/z calculated for $C_{22}H_{29}N_3O$ $[M+H]^+$ 352.2389, found: 352.2390.

***N-(4-(tert-butylamino)-5-phenylpentyl)picolinamide* (4o)**



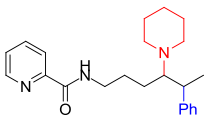
The title compound was isolated as a yellow oil (71% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 4.7 Hz, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 8.07 (br, 1H), 7.76 (m, 1H), 7.33 (m, 1H), 7.20–7.16 (m, 2H), 7.10 (d, *J* = 6.4 Hz, 3H), 3.37 (m, 2H), 3.23 (s, 1H), 2.80 (m, 1H), 2.63 (dd, *J* = 9.4, 6.9 Hz, 2H), 1.72–1.58 (m, 2H), 1.52–1.35 (m, 2H), 0.94 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.78, 154.84, 148.05, 139.07, 136.94, 129.62, 128.35, 124.64, 124.22, 123.79, 59.68, 46.76, 40.13, 39.92, 29.86, 28.70, 24.62. HRMS (ESI) m/z calculated for $C_{21}H_{29}N_3O$ $[M+H]^+$ 340.4824, found: 340.2394.

***N-(2,2-dimethyl-5-phenyl-4-(piperidin-1-yl)pentyl)picolinamide* (4p)**



The title compound was isolated as a colorless oil (45% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (br, 1H), 8.54 (d, *J* = 4.1 Hz, 1H), 8.17 (s, 1H), 8.11 (s, 1H), 7.84 (m, 1H), 7.45–7.41 (m, 2H), 7.22 (s, 1H), 7.17 (d, *J* = 7.3 Hz, 1H), 7.09 (d, *J* = 7.1 Hz, 1H), 3.21 (s, 1H), 3.06 (dd, *J* = 13.6, 6.1 Hz, 1H), 2.98 (dd, *J* = 12.9, 3.4 Hz, 1H), 2.87–2.71 (m, 3H), 2.58 (s, 2H), 2.29–2.23 (m, 1H), 1.82–1.66 (m, 4H), 1.57–1.43 (m, 3H), 1.10 (d, *J* = 14.9 Hz, 1H), 0.88 (m, 1H), 0.78 (s, 3H), 0.35 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.48, 148.28, 137.59, 135.17, 129.66, 128.76, 128.47, 127.55, 126.63, 122.55, 64.63, 47.52, 39.28, 35.56, 34.81, 29.83, 27.14, 24.95, 24.49. HRMS (ESI) m/z calculated for $C_{24}H_{33}N_3O$ $[M+H]^+$ 380.2702, found: 380.2710.

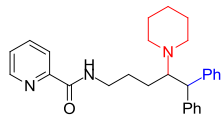
***N-(5-phenyl-4-(piperidin-1-yl)hexyl)picolinamide* (4q)**



The title compound was isolated as a colorless oil (48% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by

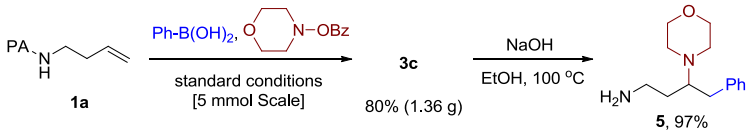
^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 4.4 Hz, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 8.04 (br, 1H), 7.84 (m, 1H), 7.42 (m, 1H), 7.19 (m, 5H), 3.44 (m, 2H), 3.01–2.90 (m, 1H), 2.61 (s, 1H), 2.45–2.34 (m, 3H), 1.72–1.59 (m, 3H), 1.36–1.19 (m, 10H), 0.91 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.33, 150.30, 148.12, 147.26, 137.44, 128.04, 127.91, 126.12, 125.71, 122.32, 70.05, 50.87, 40.85, 39.72, 28.10, 27.03, 25.66, 25.21, 18.85. HRMS (ESI) m/z calculated for $C_{23}H_{31}N_3O$ $[M+H]^+$ 366.2545, found: 366.2545.

***N-(5,5-diphenyl-4-(piperidin-1-yl)pentyl)picolinamide* (4r)**



The title compound was isolated as a colorless oil (65% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). This product was isolated as a 20:1 mixture of diastereomers. The reported dr was determined by ^{1}H NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 4.7 Hz, 1H), 8.20 (d, *J* = 7.8 Hz, 1H), 7.93 (br, 1H), 7.85 (m, 1H), 7.44–7.40 (m, 1H), 7.33 (d, *J* = 7.5 Hz, 2H), 7.28 (s, 2H), 7.24–7.16 (m, 3H), 7.15–7.11 (m, 2H), 7.05 (t, *J* = 7.3 Hz, 1H), 3.93 (d, *J* = 10.5 Hz, 1H), 3.50–3.24 (m, 3H), 2.69–2.07 (m, 4H), 1.77–1.40 (m, 4H), 1.18 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.20, 150.07, 147.96, 137.32, 128.57, 128.15, 127.96, 126.01, 125.82, 122.18, 66.98, 50.31, 50.21, 39.15, 29.73, 27.82, 26.88, 25.00. HRMS (ESI) m/z calculated for $C_{28}H_{33}N_3O$ $[M+H]^+$ 428.2702, found: 428.2710.

4. Gram-scale Reaction and PA Removal

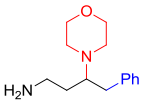


General Procedure for Removal of Picolinamide Directing Group(GP3):

Removal of picolinic acid directing group was carried out by adapting a literature procedure[6]. To an oven-dried schlenk flask was added the aryl amination product **3c** (0.2 mmol, 1.0 eq), NaOH (1 mmol, 5 eq), and EtOH (1 mL). The resulting mixture was stirred at 100 ℃ for 12 h. After this time, the reaction mixture was allowed to cool to room temperature, diluted by addition of EtOAc (5 mL) and H_2O (2 mL ×2). The aqueous layers were combined and extracted

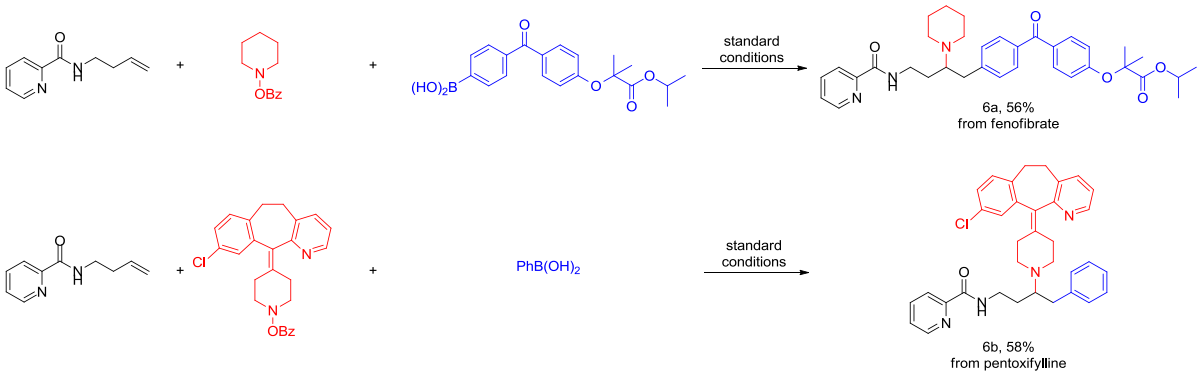
with EtOAc (10 mL × 2). The organic layers were combined, dried over Na_2SO_4, and concentrated in vacuo to give pure orimary amine product.

3-morpholino-4-phenylbutan-1-amine **(5)**

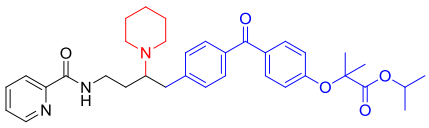


The title compound was isolated as a yellow oil (97% yield) after chromatography on alumina with ethyl acetate/hexane (1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, *J* = 7.4 Hz, 2H), 7.09 (dd, *J* = 18.3, 7.2 Hz, 3H), 3.68–3.54 (m, 4H), 2.93 (dd, *J* = 13.1, 4.0 Hz, 1H), 2.65 (m, 5H), 2.48–2.39 (m, 2H), 2.25 (dd, *J* = 13.0, 9.6 Hz, 1H), 1.92 (s, 2H), 1.53 (m, 1H), 1.34 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.53, 129.19, 128.38, 125.88, 67.51, 65.44, 48.76, 40.31, 40.18, 35.02, 33.06. HRMS (ESI) m/z calculated for $C_{14}H_{22}N_2O$ $[M+H]^+$ 235.1810, found: 235.1820.

5. Late-stage Functionalization of Biorelevant Molecules



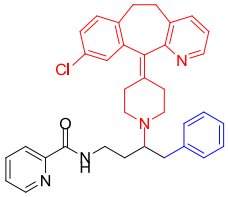
Isopropyl 2-methyl-2-(4-(4-(4-(picolinamido)-2-(piperidin-1-yl)butyl)benzoyl)phenoxy)propanoate (6a)



The title compound was isolated as a yellow oil (56% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (br, 1H), 8.20 (d, *J* = 7.4 Hz, 1H), 8.07 (s, 1H), 7.85 (t, *J* = 7.7 Hz, 1H), 7.77–7.66 (m, 5H), 7.42 (t, *J* = 7.2 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 5.11–5.06 (m, 1H), 3.72 (d, *J* = 6.9 Hz, 1H), 3.69–3.63 (m, 1H), 3.52 (d,

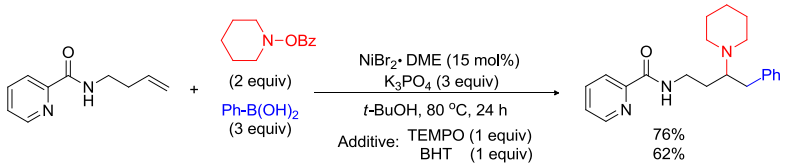
J = 6.5 Hz, 1H), 2.75 (t, *J* = 7.3 Hz, 1H), 2.61 (d, *J* = 6.9 Hz, 1H), 1.75 (m, 4H), 1.66 (s, 6H), 1.26 (t, *J* = 6.6 Hz, 6H), 1.20 (d, *J* = 6.2 Hz, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.24, 164.44, 164.38, 159.52, 159.48, 150.12, 148.11, 147.95, 146.86, 137.43, 137.21, 135.93, 132.00, 131.05, 130.97, 130.21, 130.15, 129.14, 128.37, 126.16, 125.93, 122.27, 117.30, 79.46, 69.32, 39.24, 29.39, 28.51, 25.48, 21.63, 21.59. HRMS (ESI) m/z calculated for $C_{35}H_{43}N_3O_5$ $[M+H]^+$ 586.3281, found: 586.3288.

N-(3-(4-(9-chloro-5H-benzo[5,6]cyclohepta[1,2-b]pyridin-11(6H)-ylidene)piperidin-1-yl)-4-phenylbutyl)picolinamide **(6b)**



The title compound was isolated as a brown oil (58% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.08 (m, 1H), 8.47 (t, *J* = 5.1 Hz, 1H), 8.33 (d, *J* = 4.9 Hz, 1H), 8.06 (dd, *J* = 7.8, 2.3 Hz, 1H), 7.71 (dd, *J* = 8.7, 6.5 Hz, 1H), 7.36–7.33 (m, 1H), 7.30 (t, *J* = 6.2 Hz, 1H), 7.16 (t, *J* = 7.4 Hz, 2H), 7.09–6.99 (m, 7H), 3.58 (m, 1H), 3.41–3.28 (m, 2H), 3.28–3.14 (m, 2H), 2.93 (m, 2H), 2.76–2.70 (m, 2H), 2.68–2.63 (m, 2H), 2.56–2.50 (m, 1H), 2.34 (m, 2H), 2.20 (dd, *J* = 13.0, 10.0 Hz, 1H), 1.65 (m, 1H), 1.56–1.47 (m, 1H), 1.17 (d, *J* = 2.1 Hz, 1H), 1.15 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.05, 164.27, 157.89, 150.30, 147.92, 146.60, 140.32, 139.65, 139.44, 139.40, 137.81, 137.75, 137.12, 133.41, 133.37, 132.54, 132.39, 130.96, 129.11, 128.39, 125.88, 122.06, 67.13, 51.33, 39.14, 34.79, 31.92, 31.35, 31.08, 28.48. HRMS (ESI) m/z calculated for $C_{35}H_{35}ClN_4O$ $[M+H]^+$ 563.2578, found: 563.2585.

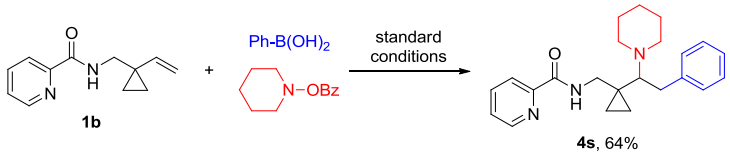
6. Radical Trapping Experiment



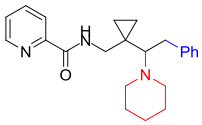
Procedure: To a 25 mL Schlenk tube were added $NiBr_2$•DME (0.03 mmol, 15 mol%), K_3PO_4 (0.6 mmol, 3 eq), alkene substrate (0.2 mmol, 1.0 eq), amine benzoate substrates (0.4 mmol, 2 eq), phenylboronic acid (0.6 mmol, 3 eq), Additive (0.2mmol, 1 equiv) and *t*-BuOH (2 mL). The

resulting mixture was stired for 24 h at 80 ℃. The product were separately obtained with a isolated yield of 76% and 62%. This result indicates that the reaction likely did not involve a radical process.

7. Radical Clock Experiment



To a 25 mL Schlenk tube were added $NiBr_2$•DME (0.03 mmol, 15 mol%), K_3PO_4 (0.5 mmol, 2.5 eq), alkene substrate (0.2 mmol, 1.0 eq), phenylboronic acid (0.4 mmol, 2 eq), *t*-BuOH (2 mL). The resulting mixture was stired for 24 h at 80 ℃. Finally, only cyclopropane remained product 4s was formed in 64% yield, implying that the cyclopropylmethyl radical intermediate known to ring rupture might not be generated in the catalytic cycle.



***N-((1-(2-phenyl-1-(piperidin-1-yl)ethyl)cyclopropyl)methyl)picolinamide* (4s)**

The title compound was isolated as a white oil (64% yield) after chromatography on alumina with ethyl acetate/hexane (1:3). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.53 (br, 1H), 8.56 (d, *J* = 4.1 Hz, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 7.83 (m, 1H), 7.42–7.38 (m, 1H), 7.28 (s, 2H), 7.21–7.16 (m, 3H), 3.66 (dd, *J* = 14.2, 4.2 Hz, 1H), 3.32 (dd, *J* = 14.1, 6.5 Hz, 1H), 3.05 (dd, *J* = 14.4, 5.4 Hz, 1H), 2.68–2.53 (m, 6H), 1.68 (dd, *J* = 8.9, 5.4 Hz, 3H), 1.46–1.40 (m, 2H), 1.27 (d, *J* = 12.0 Hz, 1H), 0.60 (dd, *J* = 9.2, 5.2 Hz, 1H), 0.52 (dd, *J* = 9.2, 4.5 Hz, 1H), 0.41 (dd, *J* = 9.6, 4.5 Hz, 1H), 0.36 (dd, *J* = 9.5, 5.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.68, 150.67, 148.05, 141.81, 137.21, 128.98, 128.41, 125.95, 125.87, 122.39, 71.49, 52.33, 34.53, 26.21, 24.94, 21.63, 12.44, 8.76. HRMS (ESI) m/z calculated for $C_{23}H_{29}N_3O$ $[M+H]^+$ 364.2389, found: 364.2400.

8. X-ray Crystallographic Data

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **4b** in a mixture of petroleum ether and ethyl acetate at room temperature. X-Ray structural analysis

of single crystal **4b** was obtained to confirm the absolute configuration. The X-ray data of **4b** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2054628. Crystal Data for $C_{27}H_{31}N_3O$ (M =413.55 g/mol): triclinic, space group P-1, a = 11.5615(3) Å, b = 12.3247(3) Å, c = 18.0802(4) Å, V = 2284.03(10) $Å^3$, Z = 4, T = 150.00(10) K, μ(MoKα) = 0.573 mm^{-1}, *Dcalc* = 1.203 g/cm^3, 34752 reflections measured (5.136° ≤ 2Θ ≤ 134.132 °), 8156 unique (R_{int} = 0.0478, R_{sigma} = 0.0303) which were used in all calculations. The final R_1 was 0.0411 (I > 2σ (I)) and *w*R_2 was 0.1155 (all data).

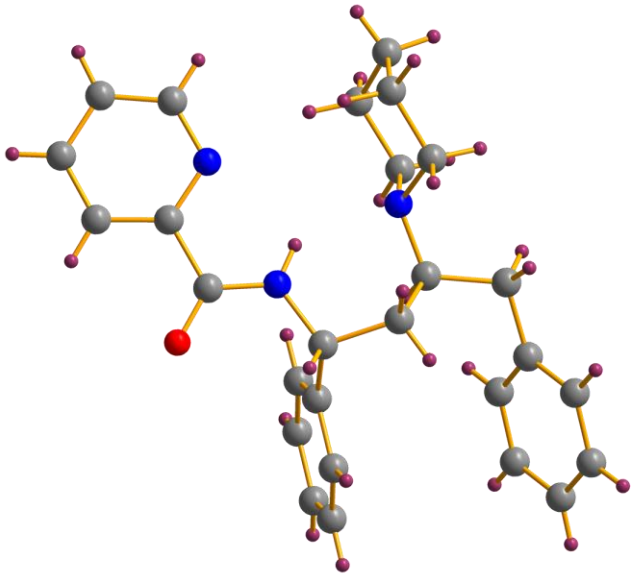


Figure S2. X-ray structure of compound **4b**.

Table S2. Crystal data and structure refinement for **4b**.

Identification code	4b
Empirical formula	$C_{27}H_{31}N_3O$
Formula weight	413.55
Temperature/K	150.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	11.5615(3)

b/Å	12.3247(3)
c/Å	18.0802(4)
α/°	76.633(2)
β/°	74.160(2)
γ/°	68.705(2)
Volume/Å^3	2284.03(10)
Z	4
ρ_{calc}g/cm^3	1.203
μ/mm^{-1}	0.573
F(000)	888.0
Crystal size/mm^3	0.24 × 0.23 × 0.2
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	5.136 to 134.132
Index ranges	-13 ≤ h ≤ 13, -14 ≤ k ≤ 14, -21 ≤ l ≤ 21
Reflections collected	34752
Independent reflections	8156 [R_{int} = 0.0478, Rsigma = 0.0303]
Data/restraints/parameters	8156/0/560
Goodness-of-fit on F^2	1.038
Final R indexes [I>=2σ (I)]	R_1 = 0.0411, wR_2 = 0.1105
Final R indexes [all data]	R_1 = 0.0449, wR_2 = 0.1155
Largest diff. peak/hole / e Å^{-3}	0.25/-0.23

Table S3. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **4b**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
O(1)	3140.6(8)	7328.6(8)	3523.3(5)	29.8(2)
N(1)	3070.6(10)	9420.1(9)	4604.0(6)	30.2(2)
N(2)	2128.6(9)	7661.4(8)	4754.2(6)	22.3(2)
N(3)	990.2(9)	8118.0(8)	6332.7(6)	20.4(2)
C(1)	3499.7(11)	8783.8(10)	4020.8(7)	23.7(3)
C(2)	3573.6(14)	10264.7(12)	4545.1(9)	37.6(3)

C(3)	4495.5(14)	10505.4(13)	3924.3(9)	40.3(3)
C(4)	4946.7(13)	9830.5(13)	3334.8(8)	37.7(3)
C(5)	4442.5(12)	8946.5(12)	3384.4(7)	29.8(3)
C(6)	2907.0(11)	7854.8(10)	4075.2(7)	22.4(2)
C(7)	1417.8(11)	6847.1(10)	4877.9(7)	20.7(2)
C(8)	2223(1)	5556(1)	4994.3(6)	20.4(2)
C(9)	1838.3(11)	4729.3(11)	4801.4(7)	25.2(3)
C(10)	2504.3(13)	3538.7(11)	4925.9(8)	31.7(3)
C(11)	3563.7(13)	3150.4(11)	5252.3(8)	31.6(3)
C(12)	3954.3(12)	3963.1(11)	5449.8(7)	28.8(3)
C(13)	3292.1(11)	5160.1(11)	5316.1(7)	24.4(3)
C(14)	291.1(10)	7132.1(10)	5568.7(7)	21.2(2)
C(15)	673.3(10)	7064.7(10)	6332.1(7)	19.8(2)
C(16)	-110.0(11)	9186.9(10)	6454.5(8)	26.3(3)
C(17)	335.2(13)	10252.2(11)	6299.7(8)	32.3(3)
C(18)	1231.8(13)	10087.8(11)	6830.5(8)	33.4(3)
C(19)	2310.7(12)	8928.7(11)	6754.3(8)	29.1(3)
C(20)	1791.6(11)	7913.5(10)	6888.0(7)	25.2(3)
C(21)	-300.5(11)	6747.8(10)	7048.4(7)	25.0(3)
C(22)	-364.9(11)	5537.1(10)	7076.1(7)	23.6(3)
C(23)	-1500.0(12)	5377.6(11)	7088.5(7)	26.7(3)
C(24)	-1544.6(13)	4275.3(12)	7063.9(7)	31.0(3)
C(25)	-450.8(14)	3315.9(11)	7022.6(7)	31.2(3)
C(26)	683.0(13)	3455.8(11)	7021.7(8)	33.9(3)
C(27)	724.4(12)	4554.3(11)	7053.7(8)	30.8(3)
O(2)	4502.9(9)	2304.6(8)	2227.4(5)	31.7(2)
N(4)	6300.7(12)	4271.9(10)	1628.4(7)	35.5(3)
N(5)	5890.1(9)	2597.8(8)	1102.1(6)	22.5(2)
N(6)	7553.9(9)	3157.3(8)	-333.1(6)	20.6(2)
C(28)	5501.3(11)	3735.9(10)	2113.2(7)	25.0(3)

C(29)	6478.9(16)	5121.9(13)	1884.4(9)	44.2(4)
C(30)	5882.4(15)	5471.8(13)	2602.8(10)	43.2(4)
C(31)	5076.7(13)	4902.1(13)	3097.7(9)	38.1(3)
C(32)	4890.0(12)	4008.2(12)	2852.8(8)	30.7(3)
C(33)	5256.7(11)	2807.6(10)	1821.1(7)	23.2(3)
C(34)	5680.9(11)	1775.4(10)	730.6(7)	20.9(2)
C(35)	6394.9(11)	494.1(10)	990.6(6)	21.1(2)
C(36)	7461.8(11)	147.9(11)	1309.6(7)	25.1(3)
C(37)	8099.9(12)	-1038.7(12)	1509.1(8)	32.0(3)
C(38)	7683.0(13)	-1890.5(12)	1394.0(8)	35.8(3)
C(39)	6619.5(14)	-1555.4(12)	1077.1(9)	35.9(3)
C(40)	5983.4(12)	-374.9(11)	879.2(8)	28.4(3)
C(41)	6023.7(11)	2091.7(10)	-158.6(7)	21.0(2)
C(42)	7395.2(10)	2095.3(10)	-479.3(6)	20.5(2)
C(43)	6966.6(12)	4253.7(10)	-816.8(7)	25.6(3)
C(44)	7016.2(12)	5307.3(11)	-537.4(8)	31.8(3)
C(45)	8376.3(13)	5201.9(11)	-558.1(9)	34.9(3)
C(46)	9016.2(13)	4022.2(11)	-108.4(9)	33.7(3)
C(47)	8892.4(11)	3019.5(11)	-403.8(8)	28.6(3)
C(48)	7862.5(12)	1857.9(10)	-1333.4(7)	24.8(3)
C(49)	7932.5(11)	637.8(10)	-1399.2(7)	23.3(2)
C(50)	8734.6(13)	-355.0(12)	-1029.3(8)	33.8(3)
C(51)	8718.9(15)	-1471.3(12)	-1038.1(8)	39.3(3)
C(52)	7916.3(13)	-1618.3(11)	-1422.8(7)	32.0(3)
C(53)	7148.5(12)	-648.0(11)	-1811.7(8)	30.4(3)
C(54)	7161.4(12)	470.7(11)	-1800.5(7)	26.9(3)

Table S4. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for **4b**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	34.7(5)	28.8(5)	25.8(4)	-7.0(4)	-0.1(4)	-12.9(4)

N(1)	33.0(6)	25.1(5)	34.9(6)	-5.0(4)	-2.8(5)	-14.9(5)
N(2)	27.1(5)	20.9(5)	22.1(5)	-4.7(4)	-2.7(4)	-12.2(4)
N(3)	19.7(5)	16.7(5)	25.9(5)	-4.0(4)	-5.3(4)	-6.1(4)
C(1)	23.2(6)	20.7(6)	26.0(6)	2.6(5)	-8.0(5)	-7.4(5)
C(2)	44.8(8)	30.1(7)	44.6(8)	-6.9(6)	-6.8(6)	-20.6(6)
C(3)	44.5(8)	35.0(7)	49.1(9)	5.7(6)	-13.1(7)	-26.6(7)
C(4)	36.5(7)	43.3(8)	35.2(7)	10.4(6)	-7.7(6)	-24.5(6)
C(5)	28.8(6)	33.4(7)	26.4(6)	3.1(5)	-6.4(5)	-13.0(5)
C(6)	22.3(6)	19.1(6)	24.1(6)	-0.8(5)	-5.8(5)	-5.4(4)
C(7)	24.2(6)	19.4(6)	21.7(6)	-2.1(4)	-6.0(4)	-10.6(5)
C(8)	22.4(6)	21.3(6)	17.6(5)	-4.6(4)	-0.1(4)	-9.1(5)
C(9)	26.5(6)	24.9(6)	27.4(6)	-4.7(5)	-8.0(5)	-9.8(5)
C(10)	38.4(7)	23.0(6)	39.7(7)	-9.0(5)	-12.2(6)	-11.2(5)
C(11)	33.4(7)	20.1(6)	39.1(7)	-6.6(5)	-10.3(6)	-2.7(5)
C(12)	25.3(6)	28.5(7)	32.5(7)	-7.8(5)	-8.7(5)	-4.5(5)
C(13)	24.3(6)	24.5(6)	26.4(6)	-7.4(5)	-3.6(5)	-9.3(5)
C(14)	20.3(5)	18.4(5)	27.3(6)	-3.7(4)	-5.9(5)	-7.9(4)
C(15)	19.2(5)	15.9(5)	24.1(6)	-4.9(4)	-2.0(4)	-5.9(4)
C(16)	23.4(6)	19.5(6)	36.6(7)	-8.0(5)	-7.6(5)	-4.1(5)
C(17)	36.0(7)	17.7(6)	44.3(8)	-4.8(5)	-13.4(6)	-6.1(5)
C(18)	41.0(7)	23.8(6)	43.0(8)	-9.8(6)	-11.4(6)	-14.2(6)
C(19)	30.8(6)	28.4(7)	34.9(7)	-6.9(5)	-11.3(5)	-12.9(5)
C(20)	26.8(6)	21.0(6)	29.2(6)	-4.2(5)	-9.5(5)	-6.3(5)
C(21)	26.5(6)	22.3(6)	25.8(6)	-6.6(5)	0.7(5)	-10.2(5)
C(22)	29.0(6)	23.4(6)	18.0(5)	-1.5(4)	-0.2(4)	-12.1(5)
C(23)	28.5(6)	25.8(6)	25.1(6)	-2.4(5)	-1.9(5)	-11.3(5)
C(24)	38.9(7)	32.2(7)	27.4(6)	-0.4(5)	-5.8(5)	-21.2(6)
C(25)	50.6(8)	22.0(6)	23.4(6)	-0.5(5)	-4.6(5)	-18.2(6)
C(26)	40.2(7)	20.5(6)	32.5(7)	1.7(5)	-3.0(6)	-6.9(5)
C(27)	29.9(6)	25.8(6)	35.0(7)	1.2(5)	-5.8(5)	-11.1(5)

O(1)	37.9(5)	33.2(5)	25.2(4)	-5.9(4)	0.8(4)	-17.1(4)
N(4)	50.3(7)	29.5(6)	31.2(6)	-7.5(5)	-4.3(5)	-19.3(5)
N(5)	25.2(5)	21.2(5)	22.9(5)	-6.0(4)	-1.7(4)	-10.4(4)
N(6)	21.4(5)	16.9(5)	24.9(5)	-1.4(4)	-7.0(4)	-7.2(4)
C(28)	26.8(6)	20.1(6)	26.8(6)	-4.7(5)	-8.8(5)	-3.0(5)
C(29)	63.3(10)	34.2(8)	44.8(9)	-10.5(6)	-7.4(7)	-26.7(7)
C(30)	52.7(9)	32.0(8)	52.8(9)	-20.9(7)	-17.3(7)	-9.2(7)
C(31)	32.5(7)	41.7(8)	40.3(8)	-23.9(6)	-10.8(6)	1.3(6)
C(32)	24.6(6)	34.8(7)	31.5(7)	-13.6(5)	-6.7(5)	-2.1(5)
C(33)	24.2(6)	19.9(6)	23.2(6)	-3.1(5)	-6.3(5)	-3.6(5)
C(34)	20.9(5)	20.7(6)	23.0(6)	-5.9(4)	-2.8(4)	-8.7(4)
C(35)	22.6(5)	21.8(6)	18.2(5)	-4.9(4)	1.2(4)	-9.2(5)
C(36)	26.2(6)	25.9(6)	24.3(6)	-4.4(5)	-3.1(5)	-10.8(5)
C(37)	28.5(6)	31.4(7)	33.5(7)	-2.4(5)	-9.7(5)	-5.7(5)
C(38)	38.0(7)	21.5(6)	42.5(8)	-1.1(6)	-9.0(6)	-5.0(5)
C(39)	41.3(8)	24.1(7)	46.8(8)	-4.8(6)	-10.4(6)	-14.9(6)
C(40)	28.5(6)	25.8(6)	34.0(7)	-4.8(5)	-7.4(5)	-11.3(5)
C(41)	23.3(6)	19.8(6)	22.4(6)	-4.2(4)	-5.6(4)	-8.5(4)
C(42)	23.0(6)	17.3(5)	21.5(6)	-1.1(4)	-5.1(4)	-7.4(4)
C(43)	27.6(6)	20.3(6)	30.8(6)	1.4(5)	-12.2(5)	-8.5(5)
C(44)	34.4(7)	18.6(6)	43.1(8)	-0.8(5)	-15.4(6)	-6.3(5)
C(45)	41.2(7)	23.0(6)	47.8(8)	1.2(6)	-18.4(6)	-16.1(6)
C(46)	33.0(7)	26.5(7)	49.8(8)	-0.1(6)	-20.5(6)	-13.7(5)
C(47)	23.4(6)	21.6(6)	42.2(7)	-2.2(5)	-11.6(5)	-7.0(5)
C(48)	29.9(6)	21.8(6)	22.5(6)	-2.1(5)	-2.7(5)	-10.7(5)
C(49)	25.5(6)	22.4(6)	19.2(5)	-4.8(4)	1.6(4)	-7.9(5)
C(50)	37.5(7)	28.6(7)	36.3(7)	-7.7(6)	-14.6(6)	-4.9(6)
C(51)	50.8(8)	22.6(7)	38.2(8)	-2.6(6)	-15.7(7)	-1.0(6)
C(52)	44.6(8)	21.8(6)	27.5(6)	-7.5(5)	2.8(6)	-13.2(6)
C(53)	32.3(7)	31.3(7)	30.4(7)	-10.3(5)	-2.0(5)	-13.3(5)

C(54)	29.6(6)	24.7(6)	25.2(6)	-4.4(5)	-5.5(5)	-6.9(5)

Table S5. Bond Lengths for **4b**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O(1)	C(6)	1.2305(15)	O(2)	C(33)	1.2313(15)
N(1)	C(1)	1.3369(17)	N(4)	C(28)	1.3388(17)
N(1)	C(2)	1.3370(17)	N(4)	C(29)	1.3368(18)
N(2)	C(6)	1.3384(15)	N(5)	C(33)	1.3383(15)
N(2)	C(7)	1.4587(14)	N(5)	C(34)	1.4556(15)
N(3)	C(15)	1.4716(14)	N(6)	C(42)	1.4756(14)
N(3)	C(16)	1.4717(15)	N(6)	C(43)	1.4708(14)
N(3)	C(20)	1.4667(15)	N(6)	C(47)	1.4667(14)
C(1)	C(5)	1.3855(17)	C(28)	C(32)	1.3854(18)
C(1)	C(6)	1.5070(16)	C(28)	C(33)	1.5045(17)
C(2)	C(3)	1.381(2)	C(29)	C(30)	1.379(2)
C(3)	C(4)	1.375(2)	C(30)	C(31)	1.375(2)
C(4)	C(5)	1.3864(19)	C(31)	C(32)	1.3809(19)
C(7)	C(8)	1.5228(16)	C(34)	C(35)	1.5224(16)
C(7)	C(14)	1.5370(16)	C(34)	C(41)	1.5389(15)
C(8)	C(9)	1.3914(16)	C(35)	C(36)	1.3888(17)
C(8)	C(13)	1.3884(16)	C(35)	C(40)	1.3915(17)
C(9)	C(10)	1.3849(18)	C(36)	C(37)	1.3895(18)
C(10)	C(11)	1.3842(19)	C(37)	C(38)	1.3788(19)
C(11)	C(12)	1.3844(18)	C(38)	C(39)	1.385(2)
C(12)	C(13)	1.3916(17)	C(39)	C(40)	1.3825(19)
C(14)	C(15)	1.5365(16)	C(41)	C(42)	1.5338(15)
C(15)	C(21)	1.5451(15)	C(42)	C(48)	1.5494(16)
C(16)	C(17)	1.5196(16)	C(43)	C(44)	1.5223(17)
C(17)	C(18)	1.5289(18)	C(44)	C(45)	1.5209(18)
C(18)	C(19)	1.5227(18)	C(45)	C(46)	1.5241(18)
C(19)	C(20)	1.5201(16)	C(46)	C(47)	1.5193(17)

C(21)	C(22)	1.5095(16)	C(48)	C(49)	1.5071(16)
C(22)	C(23)	1.3896(17)	C(49)	C(50)	1.3930(18)
C(22)	C(27)	1.3935(18)	C(49)	C(54)	1.3852(18)
C(23)	C(24)	1.3893(18)	C(50)	C(51)	1.3863(19)
C(24)	C(25)	1.381(2)	C(51)	C(52)	1.382(2)
C(25)	C(26)	1.382(2)	C(52)	C(53)	1.3760(19)
C(26)	C(27)	1.3869(19)	C(53)	C(54)	1.3892(18)

Table S6. Bond Angles for **4b**.

Atom	Atom	Atom	**Angle/**°	Atom	Atom	Atom	**Angle/**°
C(1)	N(1)	C(2)	116.94(11)	C(29)	N(4)	C(28)	116.69(12)
C(6)	N(2)	C(7)	121.67(10)	C(33)	N(5)	C(34)	121.16(10)
C(15)	N(3)	C(16)	114.79(9)	C(43)	N(6)	C(42)	114.66(9)
C(20)	N(3)	C(15)	112.28(9)	C(47)	N(6)	C(42)	111.93(9)
C(20)	N(3)	C(16)	109.76(9)	C(47)	N(6)	C(43)	109.46(9)
N(1)	C(1)	C(5)	123.14(11)	N(4)	C(28)	C(32)	123.28(12)
N(1)	C(1)	C(6)	117.01(10)	N(4)	C(28)	C(33)	117.19(11)
C(5)	C(1)	C(6)	119.85(11)	C(32)	C(28)	C(33)	119.52(11)
N(1)	C(2)	C(3)	123.81(14)	N(4)	C(29)	C(30)	123.86(14)
C(4)	C(3)	C(2)	118.72(13)	C(31)	C(30)	C(29)	118.72(13)
C(3)	C(4)	C(5)	118.50(13)	C(30)	C(31)	C(32)	118.64(13)
C(1)	C(5)	C(4)	118.86(13)	C(31)	C(32)	C(28)	118.78(13)
O(1)	C(6)	N(2)	123.89(11)	O(2)	C(33)	N(5)	123.83(11)
O(1)	C(6)	C(1)	120.88(10)	O(2)	C(33)	C(28)	120.63(11)
N(2)	C(6)	C(1)	115.23(10)	N(5)	C(33)	C(28)	115.53(10)
N(2)	C(7)	C(8)	113.71(9)	N(5)	C(34)	C(35)	113.19(9)
N(2)	C(7)	C(14)	109.16(9)	N(5)	C(34)	C(41)	109.80(9)
C(8)	C(7)	C(14)	110.96(9)	C(35)	C(34)	C(41)	110.93(9)
C(9)	C(8)	C(7)	118.50(10)	C(36)	C(35)	C(34)	123.25(10)
C(13)	C(8)	C(7)	122.99(10)	C(36)	C(35)	C(40)	118.35(11)
C(13)	C(8)	C(9)	118.45(11)	C(40)	C(35)	C(34)	118.36(10)

C(10)	C(9)	C(8)	120.94(11)	C(35)	C(36)	C(37)	120.50(11)
C(11)	C(10)	C(9)	120.26(12)	C(38)	C(37)	C(36)	120.53(12)
C(10)	C(11)	C(12)	119.40(12)	C(37)	C(38)	C(39)	119.46(12)
C(11)	C(12)	C(13)	120.25(11)	C(40)	C(39)	C(38)	120.05(12)
C(8)	C(13)	C(12)	120.69(11)	C(39)	C(40)	C(35)	121.11(12)
C(15)	C(14)	C(7)	114.05(9)	C(42)	C(41)	C(34)	114.40(9)
N(3)	C(15)	C(14)	110.87(9)	N(6)	C(42)	C(41)	110.91(9)
N(3)	C(15)	C(21)	115.62(9)	N(6)	C(42)	C(48)	115.33(9)
C(14)	C(15)	C(21)	111.66(9)	C(41)	C(42)	C(48)	111.39(9)
N(3)	C(16)	C(17)	109.94(10)	N(6)	C(43)	C(44)	110.11(10)
C(16)	C(17)	C(18)	110.54(10)	C(45)	C(44)	C(43)	111.05(11)
C(19)	C(18)	C(17)	110.04(11)	C(44)	C(45)	C(46)	109.82(10)
C(20)	C(19)	C(18)	110.61(10)	C(47)	C(46)	C(45)	110.42(11)
N(3)	C(20)	C(19)	110.25(9)	N(6)	C(47)	C(46)	110.83(10)
C(22)	C(21)	C(15)	111.62(10)	C(49)	C(48)	C(42)	111.53(9)
C(23)	C(22)	C(21)	121.00(11)	C(50)	C(49)	C(48)	120.92(11)
C(23)	C(22)	C(27)	118.04(11)	C(54)	C(49)	C(48)	121.01(11)
C(27)	C(22)	C(21)	120.89(11)	C(54)	C(49)	C(50)	118.01(11)
C(24)	C(23)	C(22)	121.05(12)	C(51)	C(50)	C(49)	120.67(12)
C(25)	C(24)	C(23)	120.17(12)	C(52)	C(51)	C(50)	120.46(12)
C(24)	C(25)	C(26)	119.52(12)	C(53)	C(52)	C(51)	119.45(12)
C(25)	C(26)	C(27)	120.26(12)	C(52)	C(53)	C(54)	120.05(12)
C(26)	C(27)	C(22)	120.93(12)	C(49)	C(54)	C(53)	121.29(12)

Table S7. Torsion Angles for **4b.**

A	**B**	**C**	**D**	**Angle/°**	**A**	**B**	**C**	**D**	**Angle/°**
N(1)	C(1)	C(5)	C(4)	-1.80(18)	N(4)	C(28)	C(32)	C(31)	2.18(19)
N(1)	C(1)	C(6)	O(1)	171.41(11)	N(4)	C(28)	C(33)	O(2)	-177.80(11)
N(1))	C(1)	C(6)	N(2)	-8.49(15)	N(4)	C(28)	C(33)	N(5)	1.14(15)
N(1)	C(2)	C(3)	C(4)	-1.2(2)	N(4)	C(29)	C(30)	C(31)	1.5(2)
N(2)	C(7)	C(8)	C(9)	-153.89(10)	N(5)	C(34)	C(35)	C(36)	-22.14(15)

N(2)	C(7)	C(8)	C(13)	29.06(15)	N(5)	C(34)	C(35)	C(40)	160.09(10)
N(2)	C(7)	C(14)	C(15)	-58.34(12)	N(5)	C(34)	C(41)	C(42)	58.06(12)
N(3)	C(15)	C(21)	C(22)	-168.43(9)	N(6)	C(42)	C(48)	C(49)	167.50(9)
N(3)	C(16)	C(17)	C(18)	-58.24(14)	N(6)	C(43)	C(44)	C(45)	58.07(14)
C(1)	N(1)	C(2)	C(3)	0.0(2)	C(28)	N(4)	C(29)	C(30)	-0.7(2)
C(2)	N(1)	C(1)	C(5)	1.54(18)	C(29)	N(4)	C(28)	C(32)	-1.19(19)
C(2)	N(1)	C(1)	C(6)	-178.29(11)	C(29)	N(4)	C(28)	C(33)	177.87(12)
C(2	C(3)	C(4)	C(5)	0.9(2)	C(29)	C(30)	C(31)	C(32)	-0.5(2)
C(3)	C(4)	C(5)	C(1)	0.50(19)	C(30)	C(31)	C(32)	C(28)	-1.25(19)
C(5)	C(1)	C(6)	O(1)	-8.42(17)	C(32)	C(28)	C(33)	O(2)	1.30(17)
C(5)	C(1)	C(6)	N(2)	171.68(10)	C(32)	C(28)	C(33)	N(5)	-179.77(10)
C(6)	N(2)	C(7)	C(8)	75.64(13)	C(33)	N(5)	C(34)	C(35)	-80.54(13)
C(6)	N(2)	C(7)	C(14)	-159.88(10)	C(33)	N(5)	C(34)	C(41)	154.91(10)
C(6)	C(1)	C(5)	C(4)	178.03(11)	C(33)	C2(8)	C(32)	C(31)	-176.86(11)
C(7)	N(2)	C(6)	O(1)	-4.08(17)	C(34)	N(5v	C(33)	O(2)	2.81(17)
C(7)	N(2)	C(6)	C(1)	175.82(9)	C(34)	N(5)	C(33)	C(28)	-176.09(9)
C(7)	C(8)	C(9)	C(10)	-177.25(11)	C(34)	C(35)	C(36)	C(37)	-177.59(11)
C(7)	C(8)	C(13)	C(12)	176.39(11)	C(34)	C(35)	C(40)	C(39)	177.63(12)
C(7)	C(14)	C(15)	N(3)	77.55(11)	C(34)	C(41)	C(42)	N(6)	-75.71(11)
C(7)	C(14)	C(15)	C(21)	-151.96(9)	C(34)	C(41)	C(42)	C(48)	154.39(9)
C(8)	C(7)	C(14)	C(15)	67.74(12)	C(35)	C(34)	C(41)	C(42)	-67.79(12)
C(8)	C(9)	C(10)	C(11)	0.5(2)	C(35)	C(36)	C(37)	C(38)	-0.07(19)
C(9)	C(8)	C(13)	C(12)	-0.66(17)	C(36)	C(35)	C(40)	C(39)	-0.25(19)
C(9)	C(10)	C(11)	C(12)	-0.3(2)	C(36)	C(37)	C(38)	C(39)	0.0(2)
C(10)	C(11)	C(12)	C(13)	-0.4(2)	C(37)	C(38)	C(39)	C(40)	-0.1(2)
C(11)	C(12)	C(13)	C(8)	0.92(19)	C(38)	C(39)	C(40)	C(35)	0.2(2)
C(13)	C(8)	C(9)	C(10)	-0.07(18)	C(40)	C(35)	C(36)	C(37)	0.19(18)
C(14)	C(7)	C(8)	C(9)	82.60(13)	C(41)	C(34)	C(35)	C(36)	101.80(12)
C(14)	C(7)	C(8)	C(13)	-94.45(12)	C(41)	C(34)	C(35)	C(40)	-75.98(13)
C(14)	C(15)	C(21)	C(22)	63.58(12)	C(41)	C(42)	C(48)	C(49)	-64.95(12)

C(15)	N(3)	C(16)	C(17)	-169.84(10)	C(42)	N(6)	C(43)	C(44)	171.62(10)
C(15)	N(3)	C(20)	C(19)	168.57(10)	C(42)	N(6)	C(47)	C(46)	-169.58(10)
C(15)	C(21)	C(22)	C(23)	-122.17(12)	C(42)	C(48)	C(49)	C(50)	-61.56(15)
C(15)	C(21)	C(22)	C(27)	54.84(15)	C(42)	C(48)	C(49)	C(54)	115.58(12)
C(16)	N(3)	C(15)	C(14)	75.14(12)	C(43)	N(6)	C(42)	C(41)	-72.51(12)
C(16)	N(3)	C(15)	C(21)	-53.24(13)	C(43)	N(6)	C(42)	C(48)	55.28(13)
C(16)	N(3)	C(20)	C(19)	-62.48(13)	C(43)	N(6)	C(47)	C(46)	62.16(13)
C(16)	C(17)	C(18)	C(19)	53.55(15)	C(43)	C(44)	C(45)	C(46)	-53.57(16)
C(17)	C(18)	C(19)	C(20)	-53.29(15)	C(44)	C(45)	C(46)	C(47)	53.27(16)
C(18)	C(19)	C(20)	N(3)	58.04(14)	C(45)	C(46)	C(47)	N(6)	-58.23(15)
C(20)	N(3)	C(15)	C(14)	-158.59(9)	C(47)	N(6)	C(42)	C(41)	162.04(10)
C(20)	N(3)	C(15)	C(21)	73.03(12)	C(47)	N(6)	C(42)	C(48)	-70.16(12)
C(20)	N(3)	C(16)	C(17)	62.59(13)	C(47)	N(6)	C(43)	C(44)	-61.65(13)
C(21)	C(22)	C(23)	C(24)	175.85(11)	C(48)	C(49)	C(50)	C(51)	174.64(12)
C(21)	C(22)	C(27)	C(26)	-175.24(11)	C(48)	C(49)	C(54)	C(53)	-174.83(11)
C(22)	C(23)	C(24)	C(25)	-0.32(19)	C((49)	C(50)	C(51)	C(52)	0.8(2)
C(23)	C(22)	C(27)	C(26)	1.85(19)	C(50)	C(49)	C(54)	C(53)	2.38(18)
C(23)	C(24)	C(25)	C(26)	1.29(19)	C(50)	C(51)	C(52)	C(53)	1.3(2)
C(24)	C(25)	C(26)	C(27)	-0.68(19)	C(51)	C(52)	C(53)	C(54)	-1.5(2)
C(25)	C(26)	C(27)	C(22)	-0.9(2)	C(52)	C(53)	C(54)	C(49)	-0.36(19)
C(27)	C(22)	C(23)	C(24)	-1.24(18)	C(54)	C(49)	C(50)	C(51)	-2.6(2)

Table S8. Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **4b.**

Atom	x	y	z	U(eq)
H(2)	2045	8027	5127	27
H(2A)	3286	10716	4946	45
H(3)	4805	11112	3905	48
H(4)	5576	9964	2912	45
H(5)	4732	8471	2997	36
H(7)	1072	6986	4414	25

H(9)	1124	4980	4586	30
H(10)	2239	2998	4790	38
H(11)	4009	2351	5338	38
H(12)	4662	3708	5673	35
H(13)	3569	5700	5444	29
H(14A)	-275	7919	5439	25
H(14B)	-176	6587	5648	25
H(15)	1464	6403	6339	24
H(16A)	-587	9103	6985	32
H(16B)	-664	9296	6107	32
H(17A)	-393	10951	6389	39
H(17B)	770	10361	5761	39
H(18A)	1576	10735	6690	40
H(18B)	767	10089	7366	40
H(19A)	2848	8972	6239	35
H(19B)	2825	8792	7131	35
H(20A)	2490	7185	6826	30
H(20B	1297	7838	7414	30
H(21A	-65	6776	7518	30
H(21B)	-1132	7325	7031	30
H(23)	-2242	6019	7114	32
H(24)	-2313	4184	7075	37
H(25)	-477	2581	6996	37
H(26)	1421	2811	7000	41
H(27)	1490	4636	7060	37
H(5A)	6430	2955	855	27
H(29)	7038	5500	1559	53
H(30)	6022	6081	2750	52
H(31)	4666	5114	3587	46
H(32)	4364	3597	3178	37

H(34)	4772	1871	876	25
H(36)	7751	715	1390	30
H(37)	8814	-1260	1722	38
H(38)	8113	-2684	1528	43
H(39)	6333	-2125	997	43
H(40)	5268	-158	668	34
H(41A)	5884	1533	-396	25
H(41B)	5454	2865	-311	25
H(42)	7920	1426	-175	25
H(43A)	6091	4331	-788	31
H(43B)	7413	4230	-1355	31
H(44A)	6641	6024	-866	38
H(44B)	6524	5357	-11	38
H(45A)	8381	5838	-329	42
H(45B)	8841	5263	-1093	42
H(46A)	9907	3923	-168	40
H(46B)	8626	4008	440	40
H(47A)	9327	3006	-944	34
H(47B)	9291	2276	-107	34
H(48A)	7288	2433	-1646	30
H(48B)	8696	1948	-1536	30
H(50)	9286	-268	-774	41
H(51)	9252	-2126	-783	47
H(52)	7895	-2367	-1419	38
H(53)	6621	-741	-2082	36
H(54)	6642	1120	-2068	32

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **4i** in a mixture of petroleum ether and ethyl acetate at room temperature. X-Ray structural analysis

of single crystal **4i** was obtained to confirm the absolute configuration. The X-ray data of **4i** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2054629.

Crystal Data for $C_{25}H_{33}N_3O_2$ (M =407.54 g/mol): monoclinic, space group $P2_1/c$, a = 21.748(3) Å, b = 5.4836(6) Å, c = 19.4942(13) Å, V = 2291.3(4) $Å^3$, Z = 4, T = 293(2) K, μ(MoKα) = 0.593 mm^{-1}, *Dcalc* = 1.181 g/cm^3, 7506 reflections measured (8.25° ≤ 2Θ ≤ 134.122 °), 4044 unique (R_{int} = 0.0533, R_{sigma} = 0.0617) which were used in all calculations. The final R_1 was 0.0787 (I > 2σ (I)) and *w*R_2 was 0.2353 (all data).

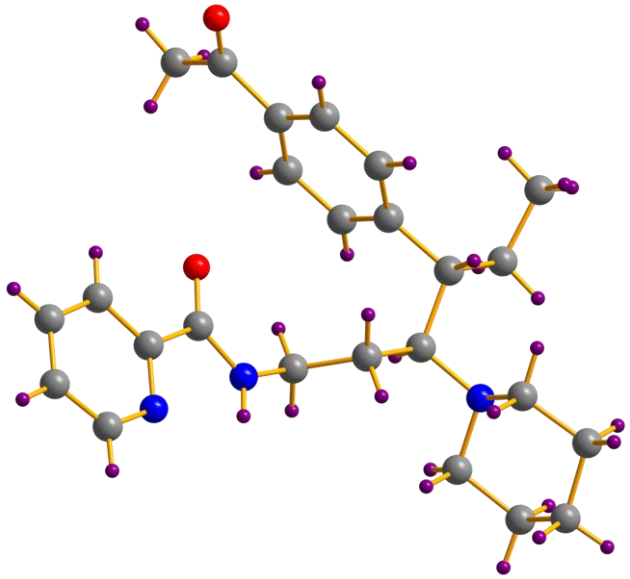


Figure S3. X-ray structure of compound **4i**.

Table S9. Crystal data and structure refinement for **4i**.

Identification code	4i
Empirical formula	$C_{25}H_{33}N_3O_2$
Formula weight	407.54
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	21.748(3)

b/Å	5.4836(6)
c/Å	19.4942(13)
α/°	90
β/°	99.738(10)
γ/°	90
Volume/$Å^3$	2291.3(4)
Z	4
$\rho_{calc}g/cm^3$	1.181
μ/mm^{-1}	0.593
F(000)	880.0
Crystal size/mm^3	0.2 × 0.15 × 0.4
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.25 to 134.122
Index ranges	$-24 \le h \le 25$, $-4 \le k \le 6$, $-21 \le l \le 23$
Reflections collected	7506
Independent reflections	4044 [R_{int} = 0.0533, Rsigma = 0.0617]
Data/restraints/parameters	4044/0/273
Goodness-of-fit on F^2	1.072
Final R indexes [I>=2σ (I)]	R_1 = 0.0787, wR_2 = 0.1919
Final R indexes [all data]	R_1 = 0.1287, wR_2 = 0.2353
Largest diff. peak/hole / e $Å^{-3}$	0.22/-0.30

Table S10. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **4i**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
O(1)	6302.2(19)	7341(9)	4638.9(18)	116.3(14)
O(2)	6847.6(16)	9790(7)	7094.0(15)	93.3(11)
N(1)	5720.6(18)	3411(9)	3245.0(19)	89.4(13)
N(2)	6548.4(15)	7067(8)	3558.6(19)	81.9(11)
N(3	8573.8(13)	10317(6)	3350.3(12)	49.6(7)
C(1)	5769.7(18)	4386(9)	3870(2)	72.5(12)

C(2)	5293(3)	1684(14)	3070(3)	118(2)
C(3)	4897(3)	933(14)	3489(4)	124(2)
C(4)	4947(3)	1900(16)	4128(4)	137(3)
C(5)	5398(3)	3662(15)	4338(3)	123(2)
C(6)	6230.1(19)	6408(10)	4061(2)	77.8(13)
C(7)	6992(2)	9091(10)	3642(3)	88.0(14)
C(8)	7650.0(17)	8269(7)	3758.6(17)	57.9(9)
C(9)	8131.4(16)	10365(7)	3848.4(14)	48.6(8)
C(10)	8264.7(18)	10872(8)	2644.6(15)	60.3(10)
C(11)	8739(2)	11116(9)	2152.8(17)	69.7(12)
C(12)	9133.4(19)	8867(9)	2167.6(17)	68.8(11)
C(13)	9435.0(18)	8309(9)	2907.1(18)	71.1(12)
C(14)	8941.4(18)	8085(8)	3369.7(17)	61.4(10)
C(15)	8491.2(16)	10517(7)	4605.5(15)	53.3(9)
C(16)	8969.6(19)	12585(9)	4697.9(17)	69.2(12)
C(17)	9397(2)	12568(12)	5399(2)	100.6(19)
C(18)	8044.5(16)	10732(7)	5126.8(15)	50.5(8)
C(19)	7637.5(19)	12682(7)	5108.8(16)	61.2(10)
C(20)	7238.0(19)	12905(8)	5594.7(17)	62.2(10)
C(21)	7241.3(17)	11196(7)	6110.9(15)	54.4(9)
C(22)	7639.8(18)	9224(8)	6124.2(16)	59.5(10)
C(23)	8039.4(18)	9022(7)	5640.3(15)	56.9(9)
C(24)	6832.9(18)	11364(9)	6656.7(19)	64.8(11)
C(25)	6427(2)	13553(10)	6656(3)	89.9(15)

Table S11. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for **4i**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	118(3)	148(4)	81(2)	-32(2)	13(2)	-14(3)
O(2)	108(2)	111(3)	69.9(17)	17.9(19)	39.2(17)	-3(2)
N(1)	80(3)	117(4)	70(2)	3(2)	9.6(18)	-30(3)

N(2)	57(2)	107(3)	81(2)	-9(2)	11.8(17)	-22(2)
N(3)	52.0(16)	61.6(19)	34.0(12)	4.4(12)	3.6(11)	0.5(14)
C(1)	50(2)	95(4)	72(2)	16(2)	9.4(18)	0(2)
C(2)	96(4)	155(6)	102(4)	-13(4)	19(3)	-53(4)
C(3)	91(4)	143(6)	142(5)	4(5)	29(4)	-38(4)
C(4)	113(5)	173(7)	140(6)	24(5)	63(4)	-40(5)
C(5)	104(4)	177(7)	98(4)	4(4)	47(3)	-30(5)
C(6)	56(2)	101(4)	73(2)	-4(3)	2.7(19)	-5(2)
C(7)	60(3)	89(4)	113(4)	-4(3)	8(2)	-13(3)
C(8)	62(2)	63(2)	47.6(16)	2.0(16)	4.7(15)	-8.8(19)
C(9)	52.5(19)	55(2)	37.7(14)	3.7(14)	5.2(13)	-0.5(17)
C(10)	65(2)	76(3)	37.7(15)	3.2(16)	2.9(15)	11(2)
C(11)	83(3)	85(3)	41.7(16)	11.9(18)	13.5(17)	3(2)
C(12)	65(2)	95(3)	47.6(17)	-3(2)	13.3(16)	-1(2)
C(13)	58(2)	100(3)	54.8(19)	1(2)	9.7(16)	10(2)
C(14)	61(2)	74(3)	47.6(17)	11.3(18)	6.9(15)	11(2)
C(15)	59(2)	63(2)	36.5(14)	3.3(15)	6.1(14)	-4.7(18)
C(16)	68(2)	93(3)	45.6(17)	-5.5(19)	8.1(16)	-18(2)
C(17)	80(3)	166(6)	54(2)	-20(3)	6(2)	-33(4)
C(18)	61(2)	51(2)	37.3(14)	1.9(14)	3.5(14)	-2.9(18)
C(19)	81(3)	61(3)	42.5(16)	13.4(17)	13.4(16)	0(2)
C(20)	71(2)	64(3)	52.0(18)	-1.8(18)	9.7(16)	3(2)
C(21)	58(2)	64(2)	40.6(15)	-0.1(16)	6.8(14)	-10.6(19)
C(22)	73(2)	63(3)	42.5(16)	11.6(16)	9.1(16)	-6(2)
C(23)	67(2)	63(2)	40.4(15)	7.2(16)	6.4(15)	3(2)
C(24)	61(2)	80(3)	54.3(19)	-7(2)	11.6(17)	-12(2)
C(25)	80(3)	105(4)	92(3)	-12(3)	35(3)	-7(3)

Table S12. Bond Lengths for **4i**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O(1)	C(6)	1.222(5)	C9	C15	1.551(4)

O(2)	C(24)	1.210(5)	C10	C11	1.528(5)
N(1)	C(1)	1.318(6)	C11	C12	1.499(6)
N(1)	C(2)	1.331(7)	C12	C13	1.511(5)
N(2)	C(6)	1.342(5)	C13	C14	1.519(5)
N(2)	C(7)	1.461(6)	C15	C16	1.529(5)
N(3)	C(9)	1.478(4)	C15	C18	1.524(4)
N(3)	C(10)	1.458(4)	C16	C17	1.518(5)
N(3	C(14)	1.459(5)	C18	C19	1.385(5)
C(1)	C(5)	1.375(6)	C18	C23	1.373(5)
C(1)	C(6)	1.498(7)	C19	C20	1.395(5)
C(2)	C(3)	1.347(8)	C20	C21	1.374(5)
C(3)	C(4)	1.341(9)	C21	C22	1.383(5)
C(4)	C(5)	1.389(9)	C21	C24	1.500(5)
C(7)	C(8)	1.481(6)	C22	C23	1.391(5)
C(8)	C(9)	1.545(5)	C24	C25	1.490(7)

Table S13. Bond Angles for **4i**.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C(1)	N(1)	C(2)	118.0(4)	C(12)	C(11)	C(10)	111.1(3)
C(6)	N(2)	C(7)	122.4(4)	C(11)	C(12)	C(13)	109.9(3)
C(10)	N(3)	C(9)	111.6(3)	C(12)	C(13)	C(14)	110.3(3)
C(10)	N(3)	C(14)	111.3(3)	N(3)	C(14)	C(13)	110.9(3)
C(14)	N(3)	C(9)	114.4(3)	C(16)	C(15)	C(9)	112.1(3)
N(1)	C(1)	C(5)	122.1(5)	C(18)	C(15)	C(9)	111.3(3)
N(1)	C(1)	C(6)	118.5(4)	C(18)	C(15)	C(16)	111.2(3)
C(5)	C(1)	C(6)	119.4(5)	C(17)	C(16)	C(15)	113.7(4)
N(1)	C(2)	C(3)	123.6(6)	C(19)	C(18)	C(15)	121.3(3)
C(4)	C(3)	C(2)	118.8(6)	C(23)	C(18)	C(15)	121.2(3)
C(3)	C(4)	C(5)	119.5(5)	C(23)	C(18)	C(19)	117.5(3)
C(1)	C(5)	C(4)	118.0(6)	C(18)	C(19)	C(20)	121.3(3)
O(1)	C(6)	N(2)	124.0(5)	C(21)	C(20)	C(19)	120.7(4)

O(1)	C(6)	C(1)	121.1(4)	C(20)	C(21)	C(22)	118.2(3)
N(2)	C(6)	C(1)	115.0(4)	C(20)	C(21)	C(24)	123.2(4)
N(2)	C(7)	C(8)	112.9(4)	C(22)	C(21)	C(24)	118.6(3)
C(7)	C(8)	C(9)	114.2(4)	C(21)	C(22)	C(23)	120.7(3)
N(3)	C(9)	C(8)	114.6(3)	C(18)	C(23)	C(22)	121.5(4)
N(3)	C(9)	C(15)	110.3(3)	O(2)	C(24)	C(21)	120.5(4)
C(8)	C(9)	C(15)	111.8(3)	O(2)	C(24)	C(25)	121.3(4)
N(3)	C(10)	C(11)	111.0(3)	C(25)	C(24)	C(21)	118.1(4)

Table S14. Torsion Angles for **4i.**

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
N(1)	C(1)	C(5)	C(4)	-2.0(10)	C(9)	C(15)	C(18)	C(23)	120.4(4)
N(1)	C(1)	C(6)	O(1)	-178.6(5)	C(10)	N(3)	C(9)	C(8)	69.2(4)
N(1)	C(1)	C(6)	N(2)	1.0(7)	C(10)	N(3)	C(9)	C(15)	-163.7(3)
N(1)	C(2)	C(3)	C(4)	-2.6(12)	C(10)	N(3)	C(14)	C(13)	59.1(4)
N(2)	C(7)	C(8)	C(9)	180.0(3)	C(10)	C(11)	C(12)	C(13)	-54.6(5)
N(3)	C(9)	C(15)	C(16)	51.2(4)	C(11)	C(12)	C(13)	C(14)	55.6(5)
N(3)	C(9)	C(15)	C(18)	176.4(3)	C(12)	C(13)	C(14)	N(3)	-57.9(5)
N(3)	C(10)	C(11)	C(12)	55.8(5)	C(14)	N(3)	C(9)	C(8)	-58.3(4)
C(1)	N(1)	C(2)	C(3)	2.0(10)	C(14)	N(3)	C(9)	C(15)	68.8(4)
C(2)	N(1)	C(1)	C(5)	0.4(8)	C(14)	N(3)	C(10)	C(11)	-57.7(4)
C(2)	N(1)	C(1)	C(6)	-177.4(5)	C(15)	C(18)	C(19)	C(20)	-178.6(3)
C(2)	C(3)	C(4)	C(5)	0.8(13)	C(15)	C(18)	C(23)	C(22)	179.0(3)
C(3)	C(4)	C(5)	C(1)	1.4(12)	C(16)	C(15)	C(18)	C(19)	64.9(4)
C(5)	C(1)	C(6)	O(1)	3.6(8)	C(16)	C(15)	C(18)	C(23)	-113.8(4)
C(5)	C(1)	C(6)	N(2)	-176.8(5)	C(18)	C(15)	C(16)	C(17)	63.2(5)
C(6)	N(2)	C(7)	C(8)	105.1(5)	C(18)	C(19)	C(20)	C(21)	0.5(6)
C(6)	C(1)	C(5)	C(4)	175.7(6)	C(19)	C(18)	C(23)	C(22)	0.3(5)
C(7)	N(2)	C(6)	O(1)	-3.8(8)	C(19)	C(20)	C(21)	C(22)	-1.6(6)
C(7)	N(2)	C(6)	C(1)	176.7(4)	C(19)	C(20)	C(21)	C(24)	178.7(4)
C(7)	C(8)	C(9)	N(3)	-123.1(4)	C(20)	C(21)	C(22)	C(23)	2.0(5)

C(7)	C(8)	C(9)	C(15)	110.5(4)	C(20)	C(21)	C(24)	O(2)	178.9(4)
C(8)	C(9)	C(15)	C(16)	179.9(3)	C(20)	C(21)	C(24)	C(25)	-3.0(6)
C(8)	C(9)	C(15)	C(18)	-54.9(4)	C(21)	C(22)	C(23)	C(18)	-1.3(6)
C(9)	N(3)	C(10)	C(11)	173.2(3)	C(22)	C(21)	C(24)	O(2)	-0.8(6)
C(9)	N(3)	C(14)	C(13)	-173.2(3)	C(22)	C(21)	C(24)	C(25)	177.2(4)
C(9)	C(15)	C(16)	C(17)	-171.5(4)	C(23)	C(18)	C(19)	C(20)	0.1(5)
C(9)	C(15)	C(18)	C(19)	-60.8(5)	C(24)	C(21)	C(22)	C(23)	-178.3(3)

Table S15. Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for **4i.**

Atom	x	y	z	U(eq)
H(2)	6489	6269	3173	98
H(2A)	5266	955	2635	141
H(3)	4594	-235	3337	149
H(4)	4682	1395	4427	165
H(5)	5448	4333	4782	148
H(7A)	6922	10085	4033	106
H(7B)	6916	10102	3228	106
H(8A)	7717	7269	3368	69
H(8B)	7723	7253	4172	69
H(9)	7894	11884	3762	58
H(10A)	8033	12384	2646	72
H(10B)	7971	9584	2480	72
H(11A)	8521	11390	1682	84
H(11B)	9005	12514	2289	84
H(12A)	9453	9115	1883	83
H(12B)	8876	7501	1978	83
H(13A)	9725	9601	3080	85
H(13B)	9668	6796	2918	85
H(14A)	8668	6724	3214	74
H(14B)	9141	7762	3844	74

H(15)	8721	8985	4703	64
H(16A)	9222	12466	4334	83
H(16B)	8750	14131	4641	83
H(17A)	9151	12715	5763	151
H(17B)	9682	13912	5425	151
H(17C)	9626	11066	5454	151
H(19)	7630	13866	4766	73
H(20)	6966	14225	5569	75
H(22)	7641	8020	6460	71
H(23)	8309	7698	5665	68
H(25A)	6195	13434	7032	135
H(25B)	6144	13643	6222	135
H(25C)	6682	14993	6715	135

9. References

[1] J. A. Gurak, K. S. Yang, Z. Liu, K. M. Engle, *J. Am. Chem. Soc.* **2016**, *138*, 5805.

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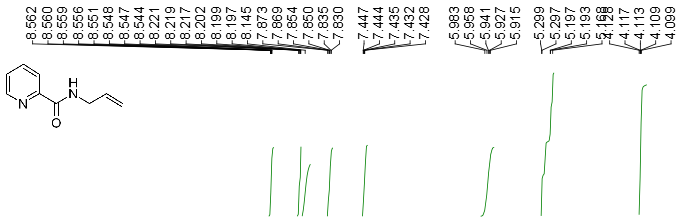
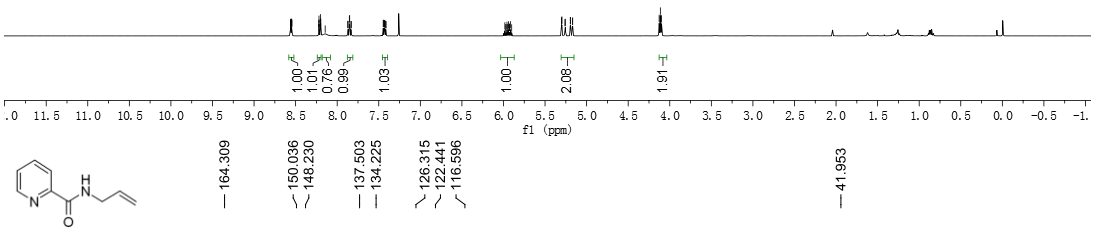
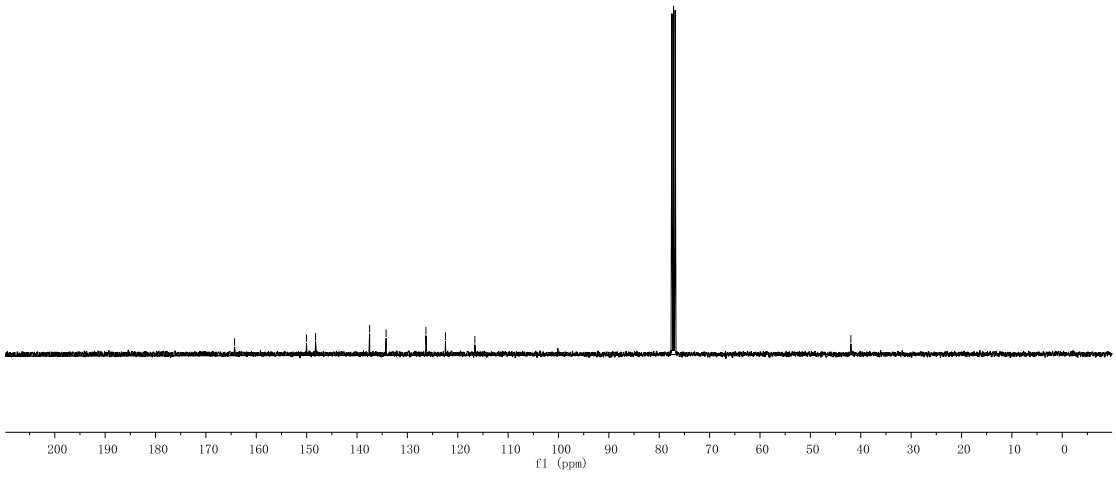
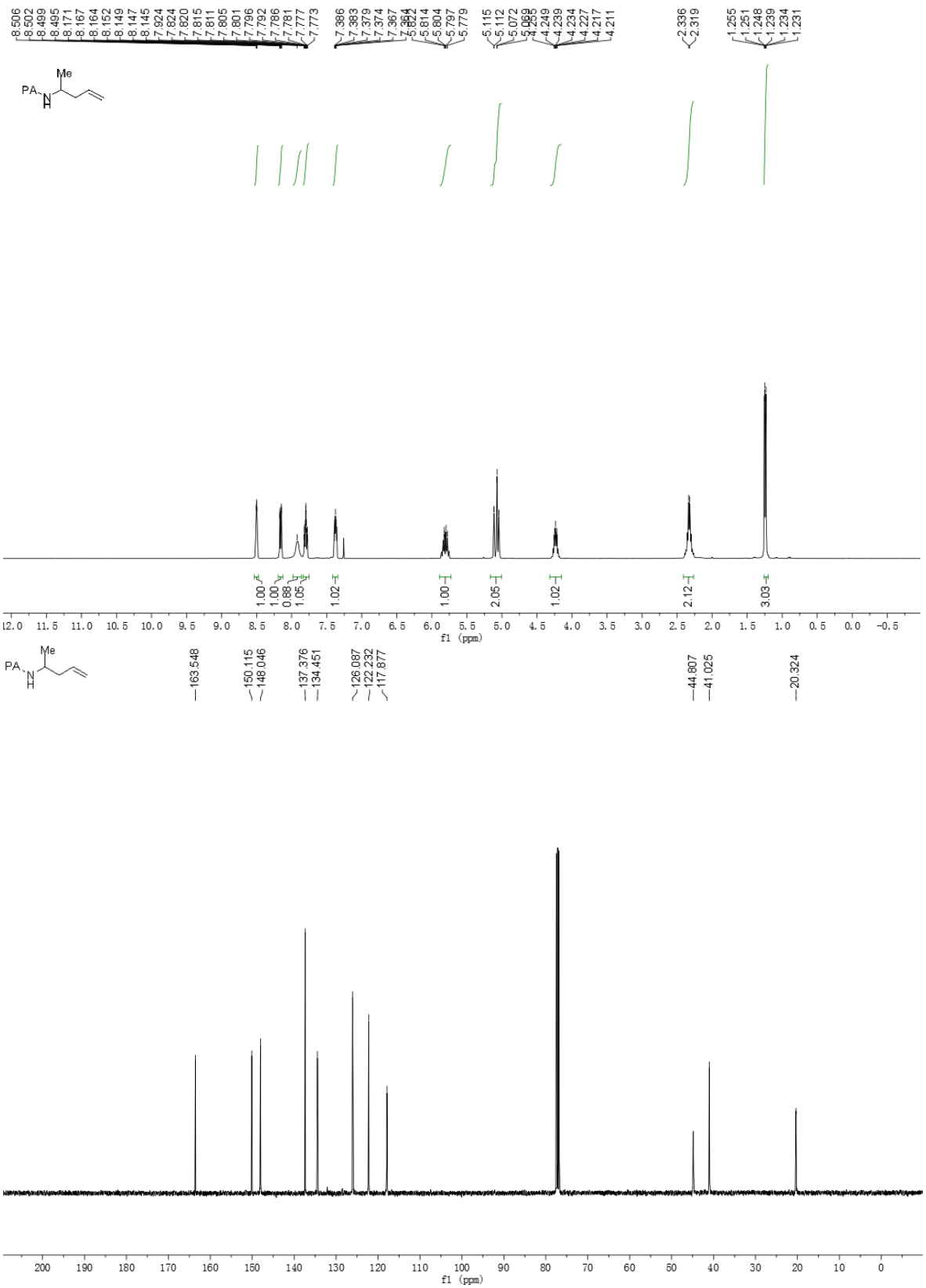
10. NMR Spectra





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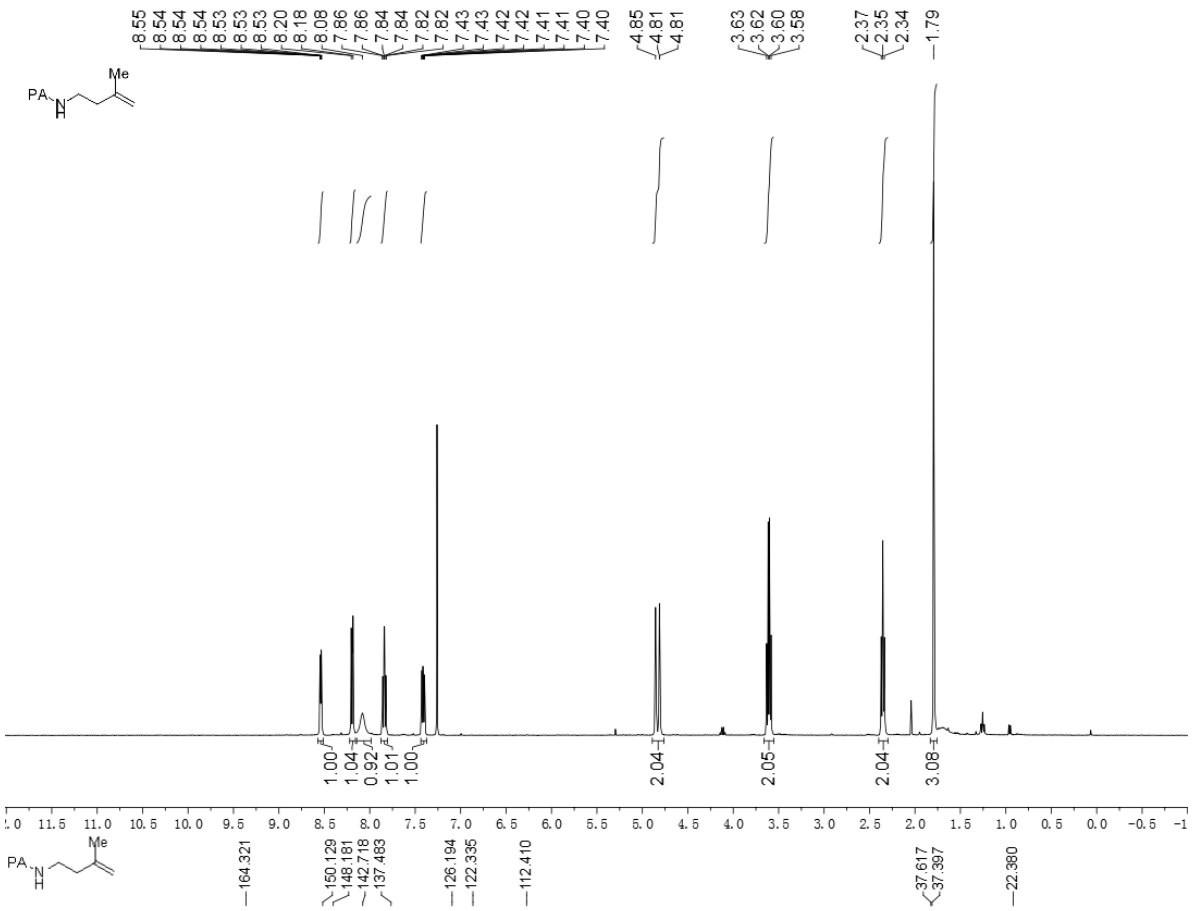
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7.43
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7.40
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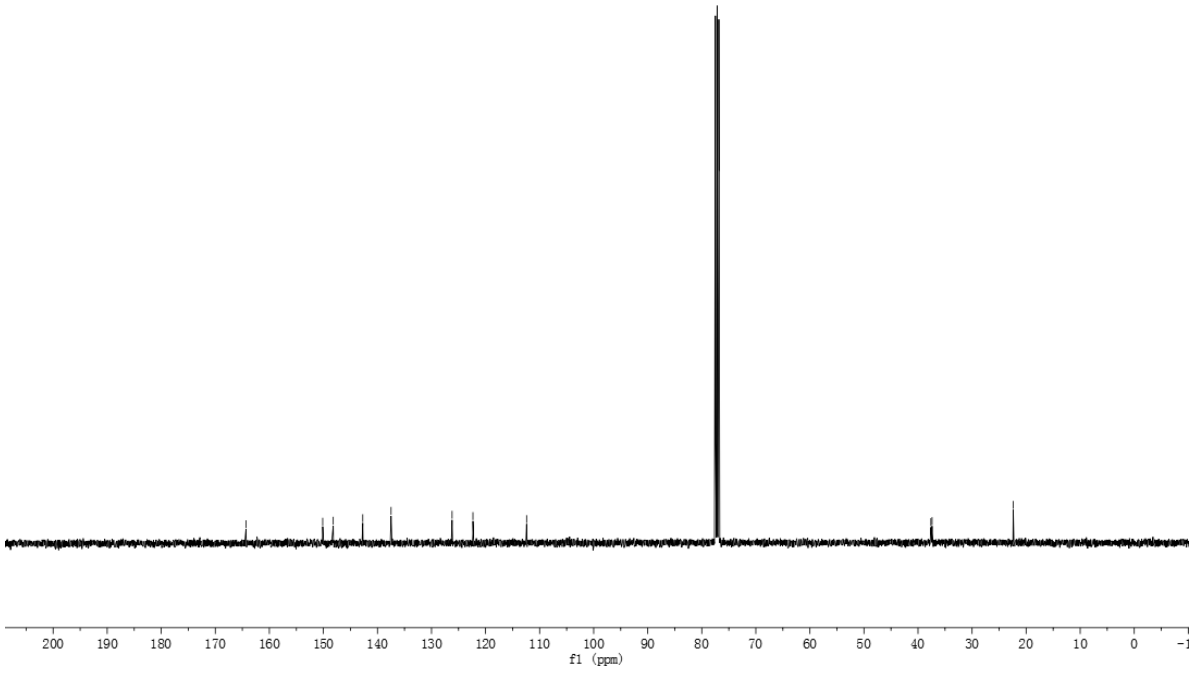
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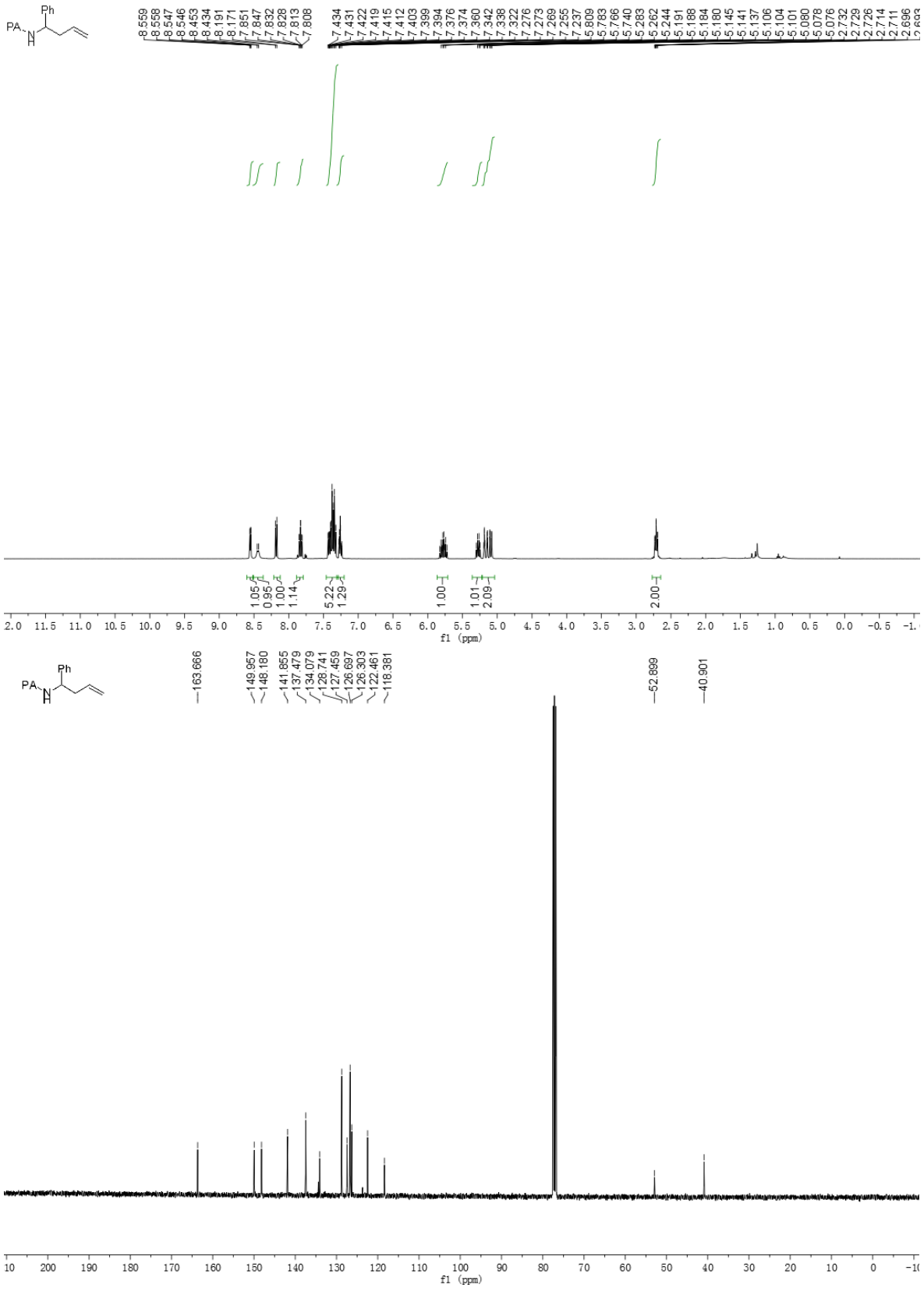
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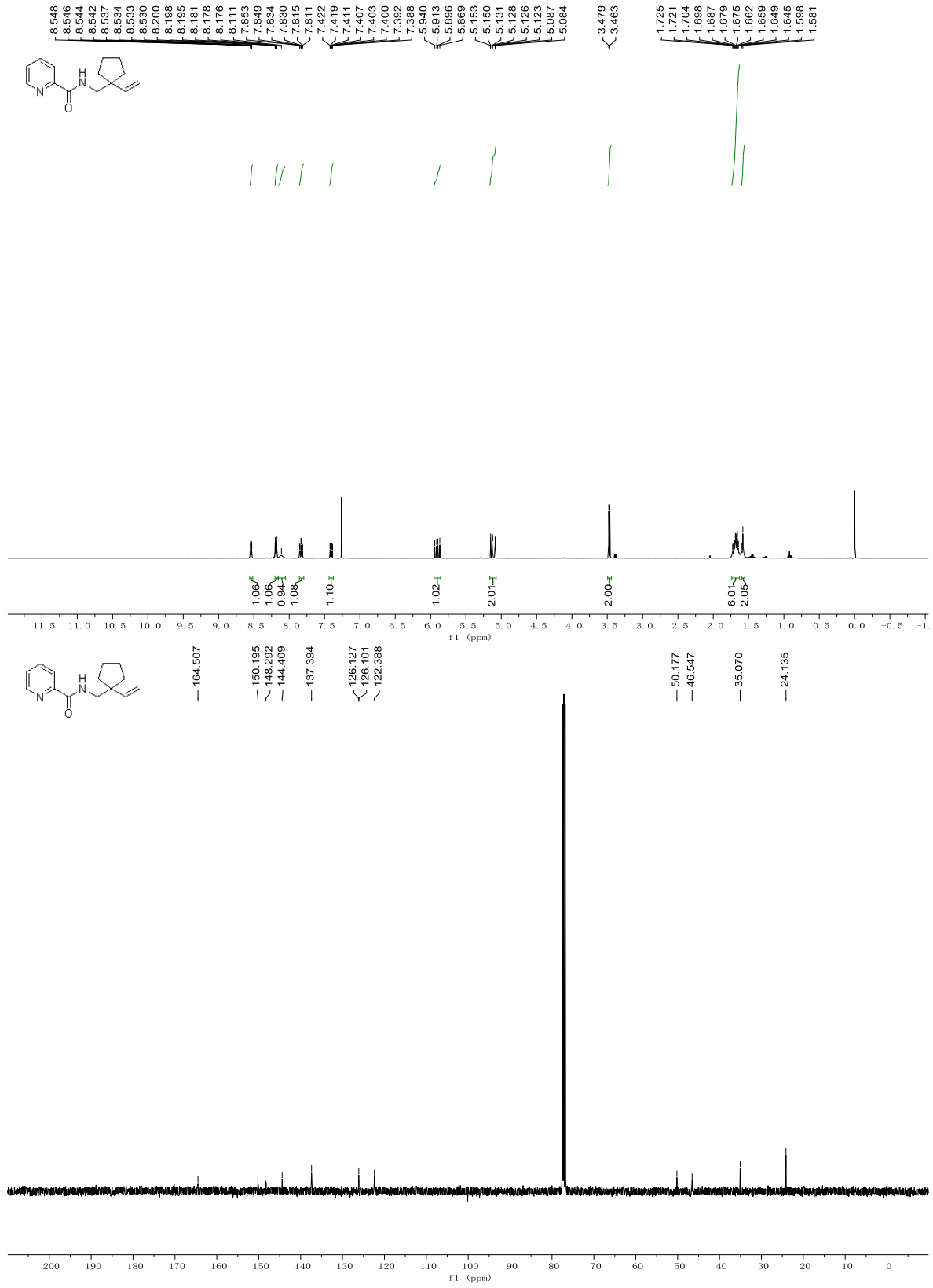
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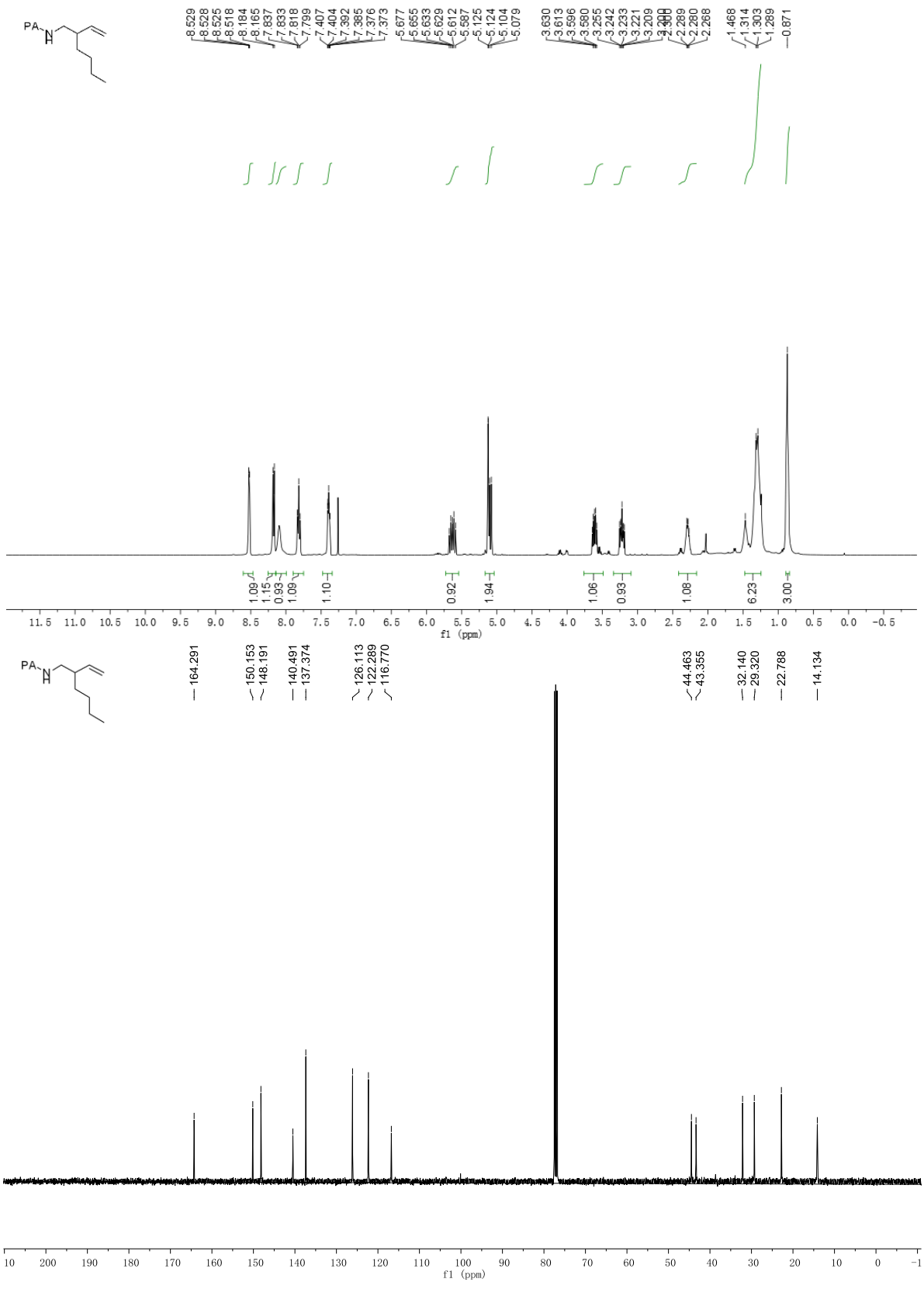
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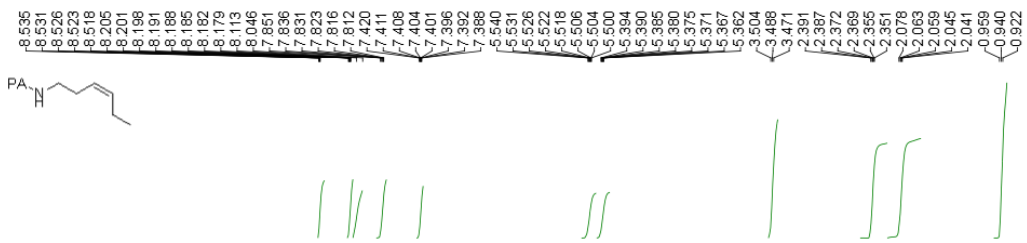
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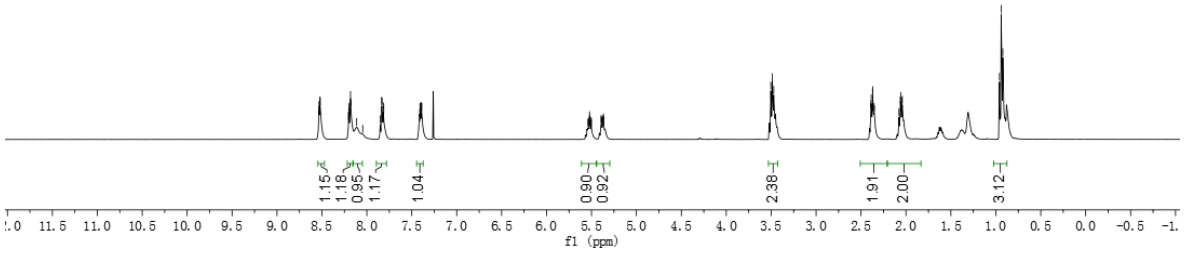
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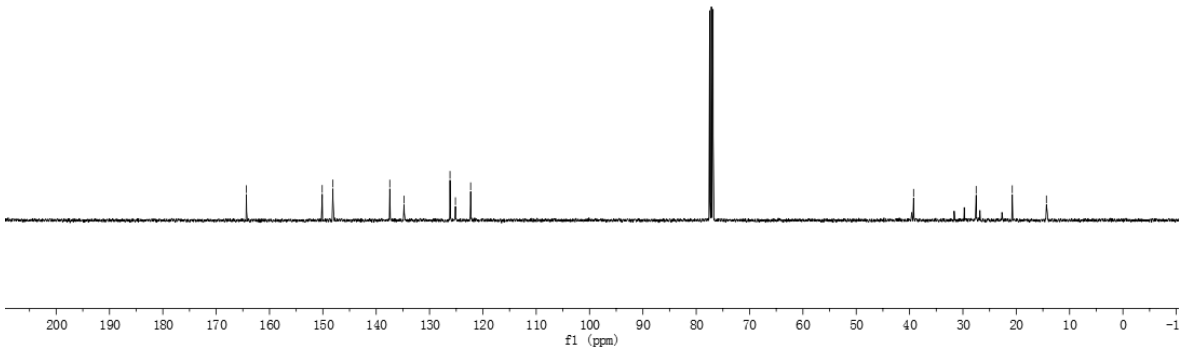
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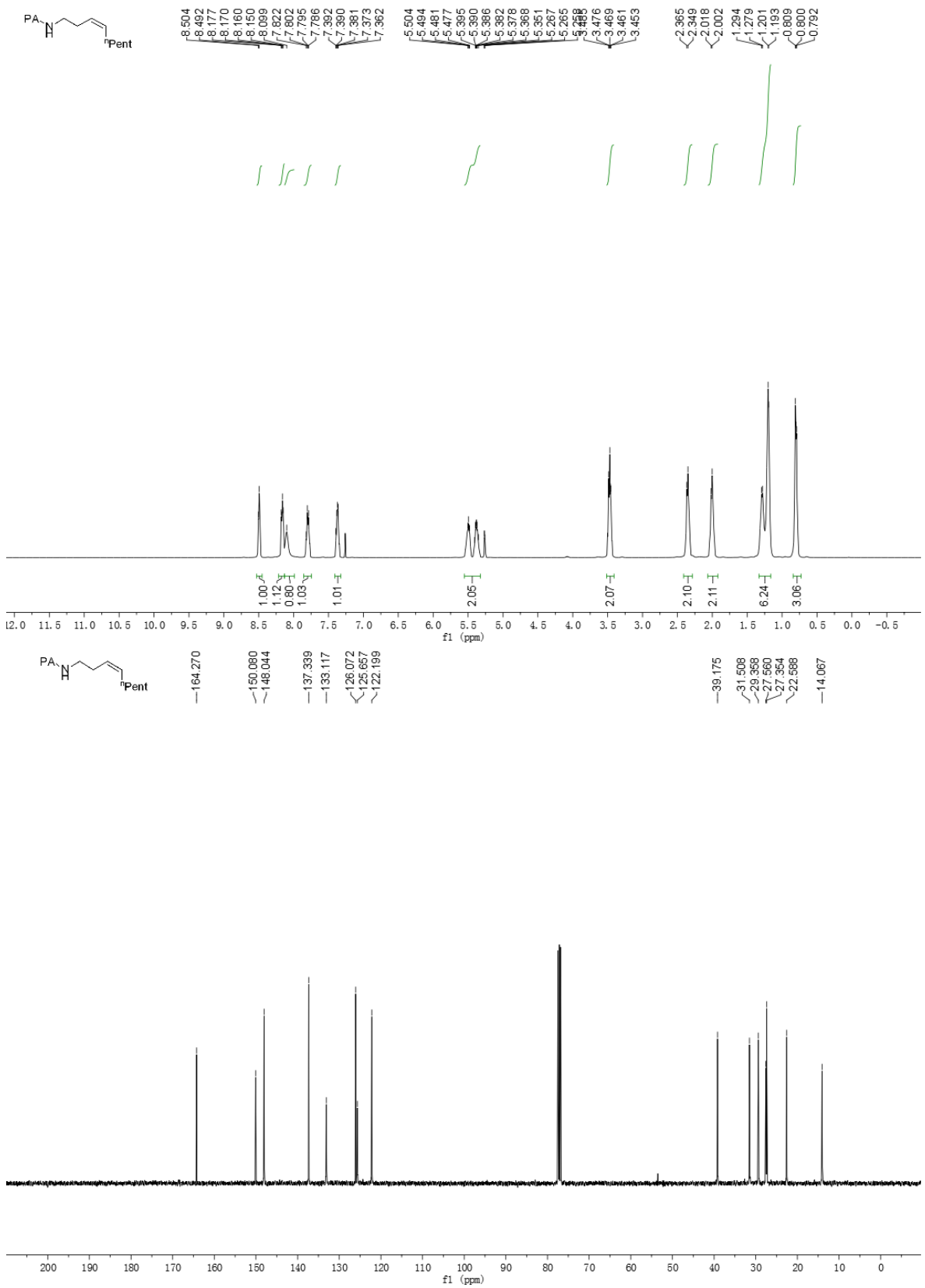
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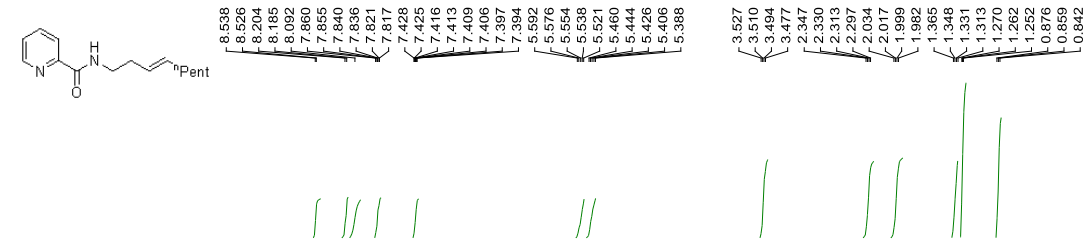
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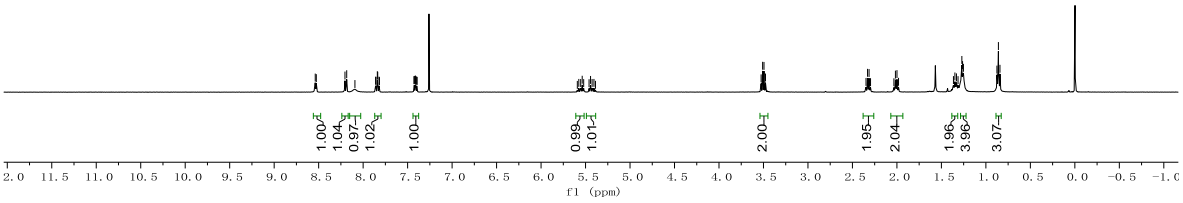
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f1 (ppm)


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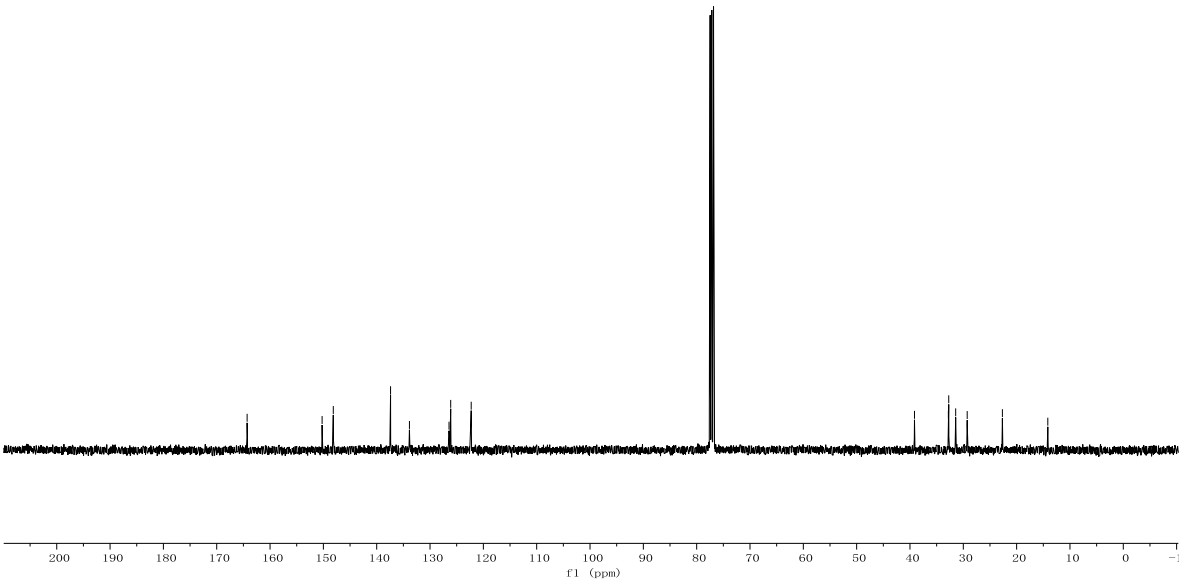
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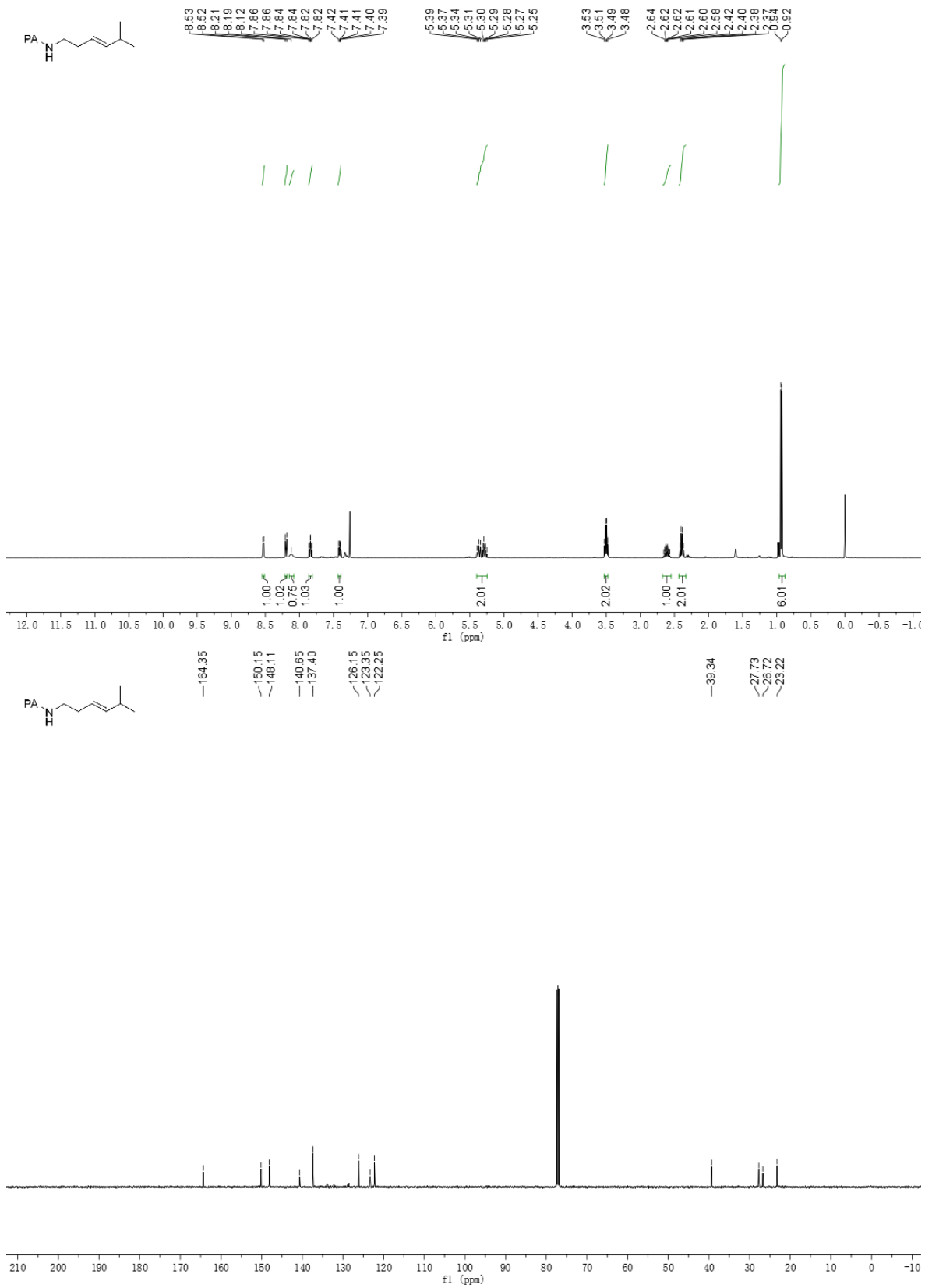
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H
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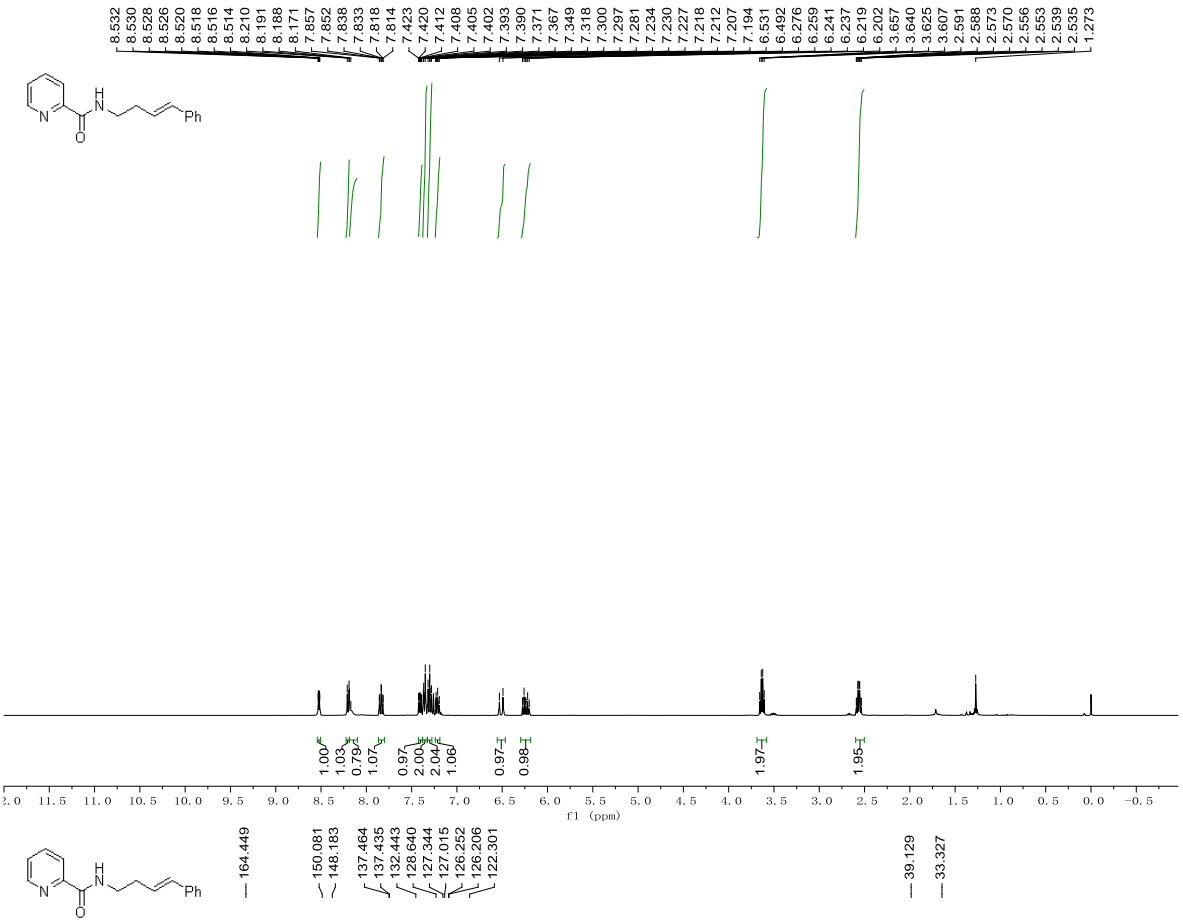
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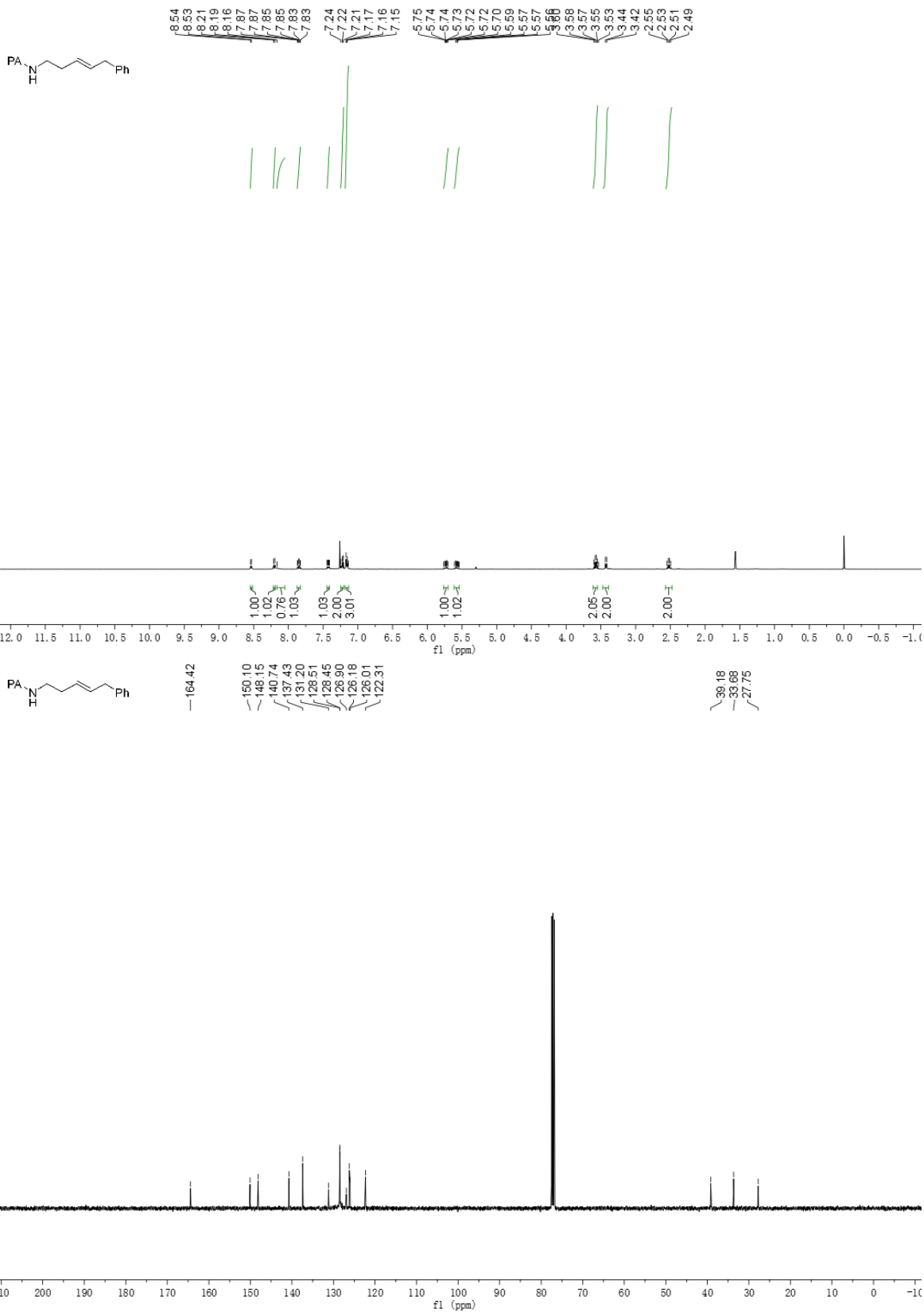
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1.02
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f1 (ppm)
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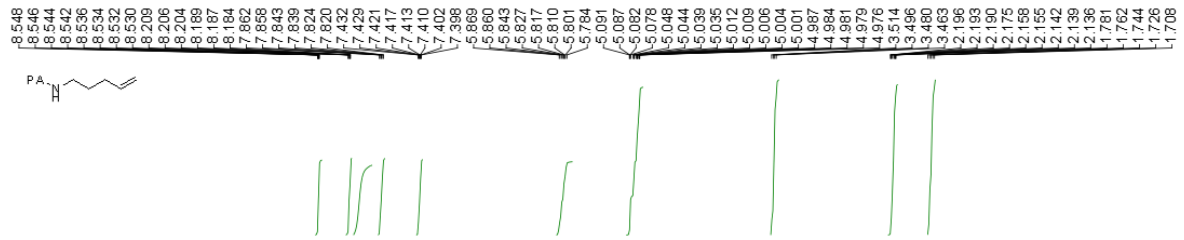
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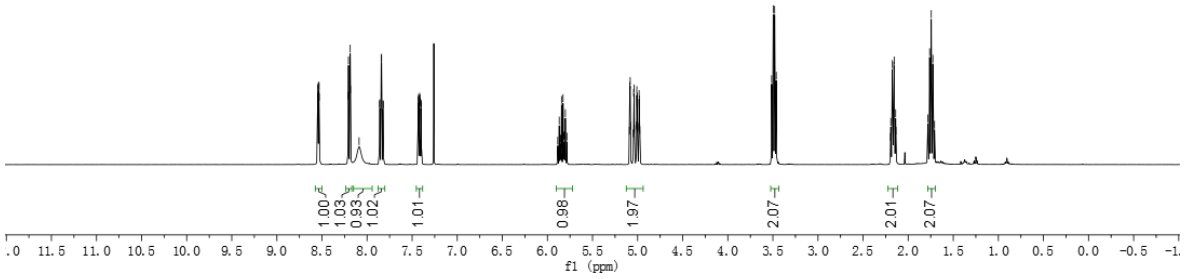
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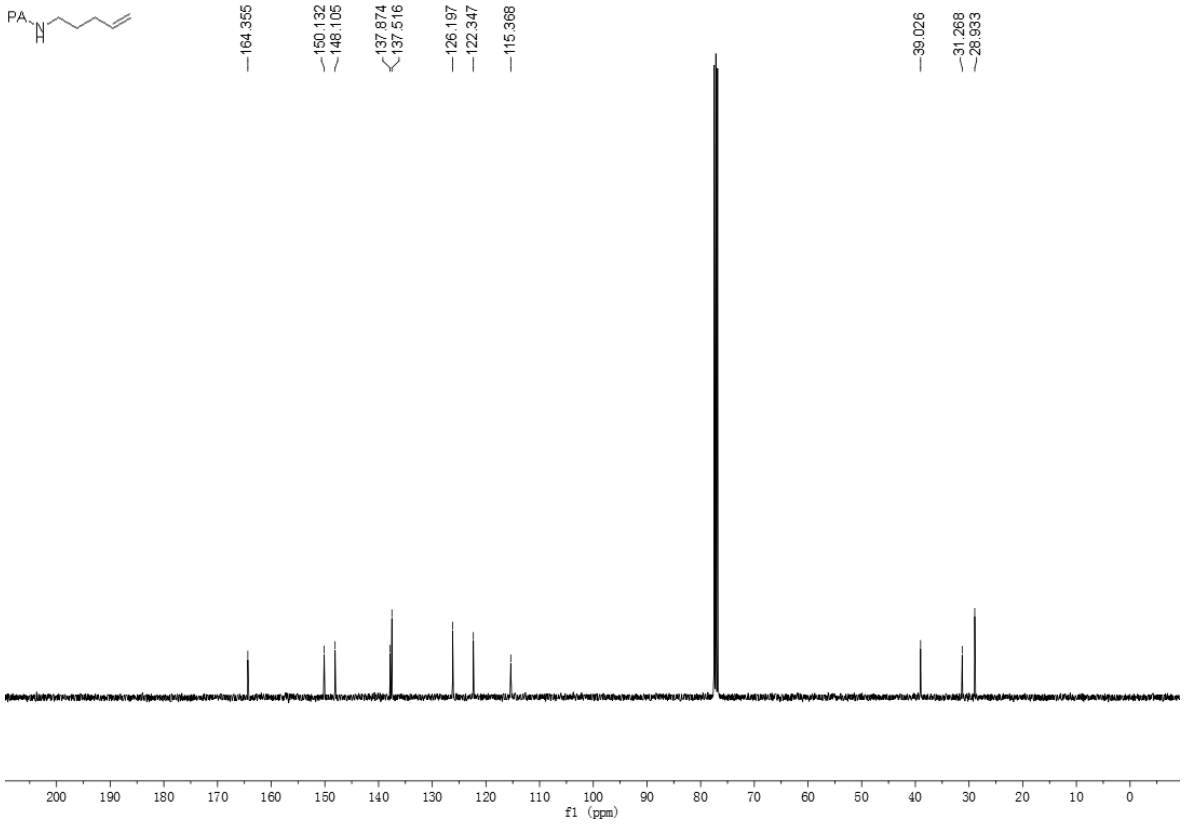
f1 (ppm)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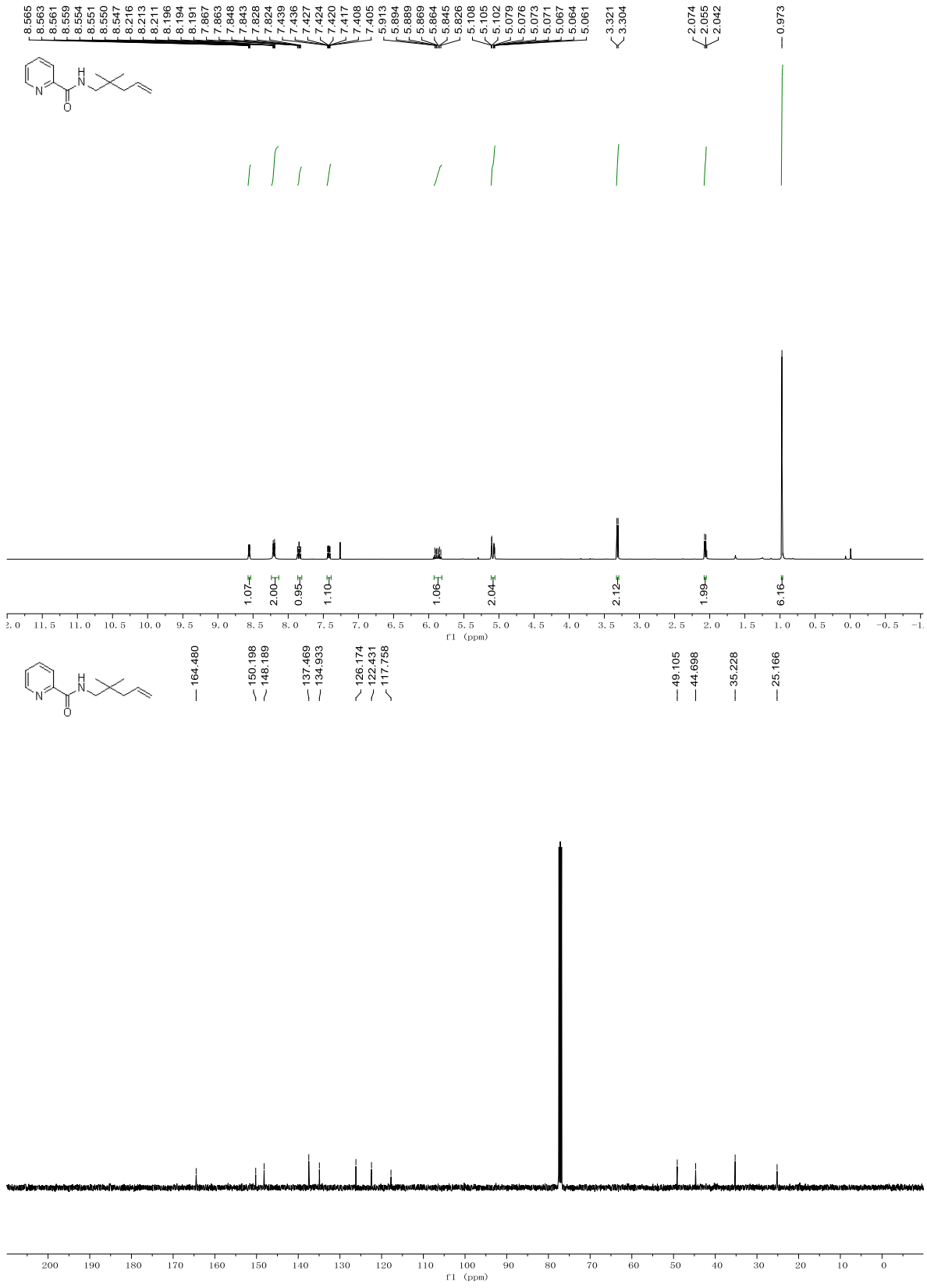
f1 (ppm)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7.420
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5.067
5.064
5.061
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3.304
2.074
2.055
2.042
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1.10
1.06
2.04
2.12
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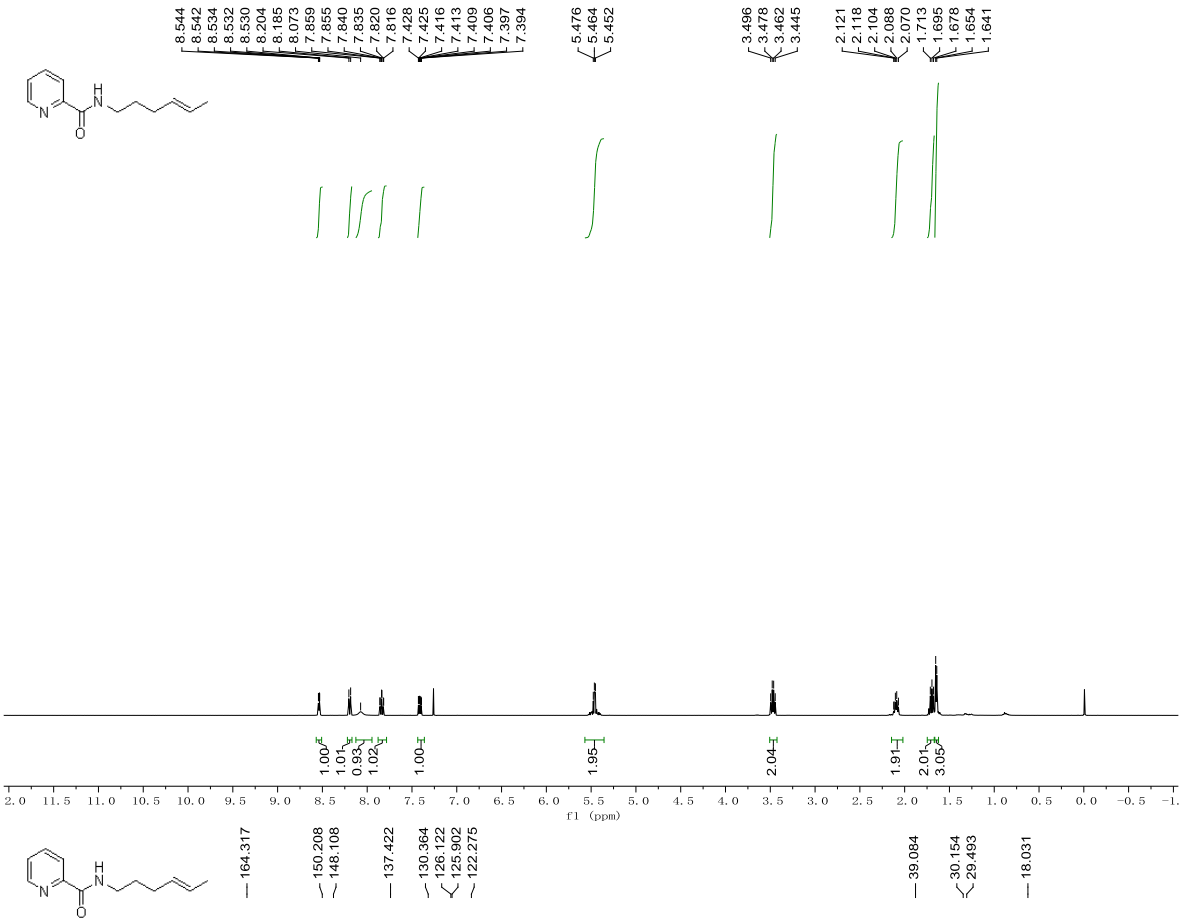
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3.445
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2.070
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2.04
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3.05
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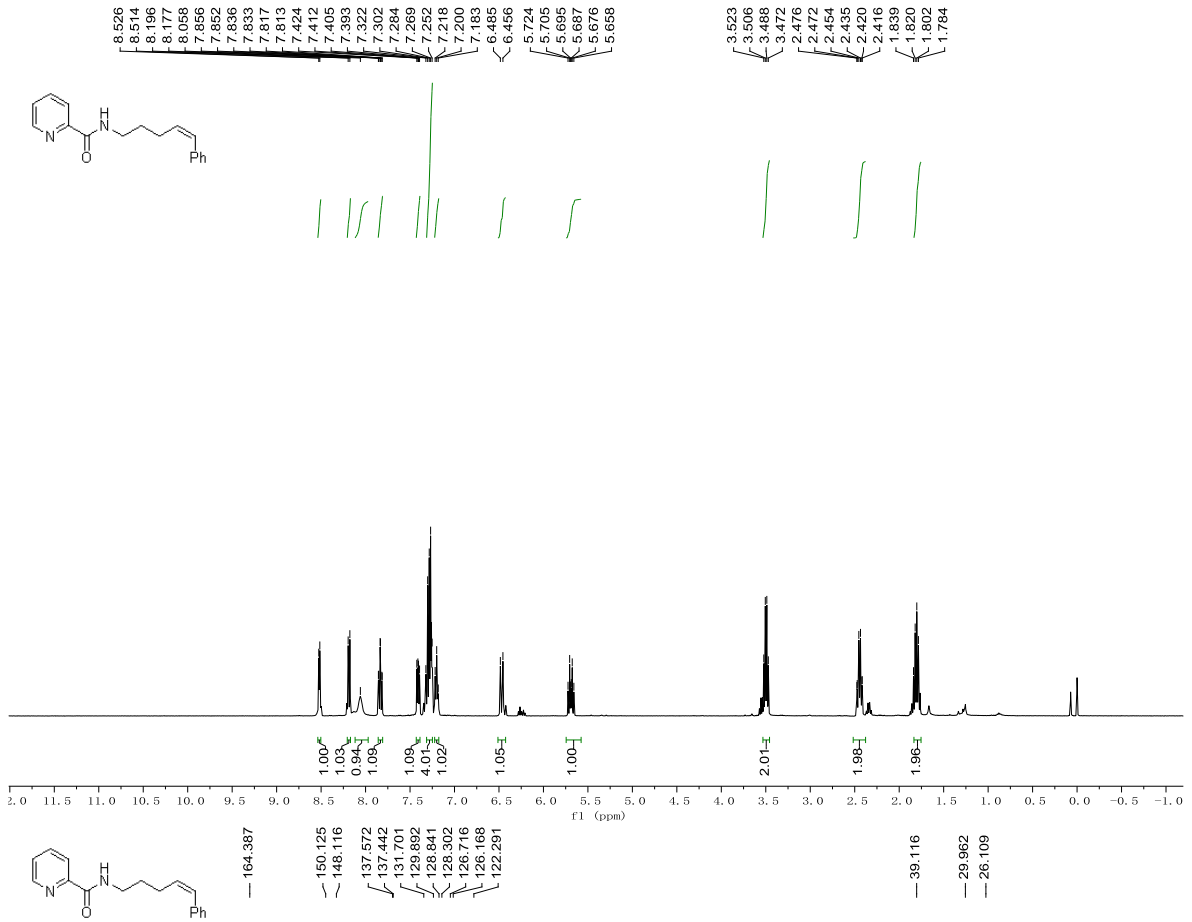
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1.802
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N
O
Ph
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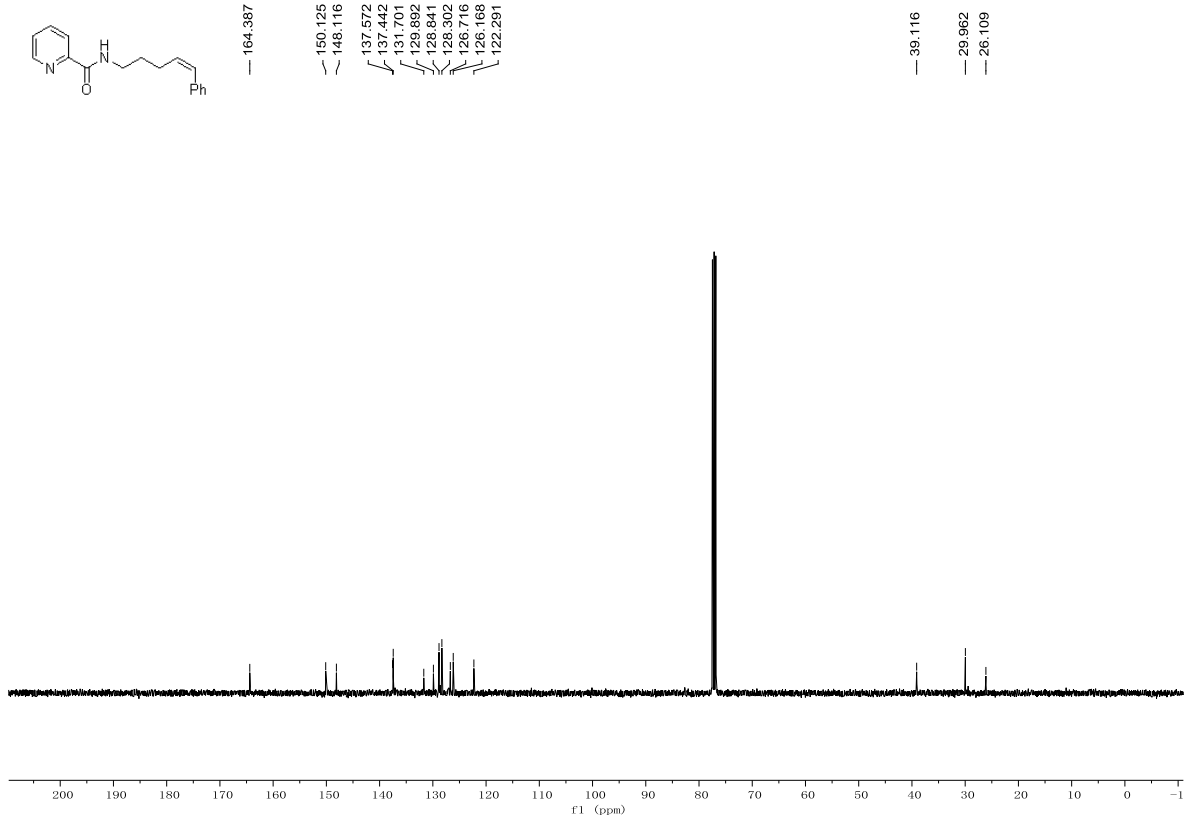
f1 (ppm)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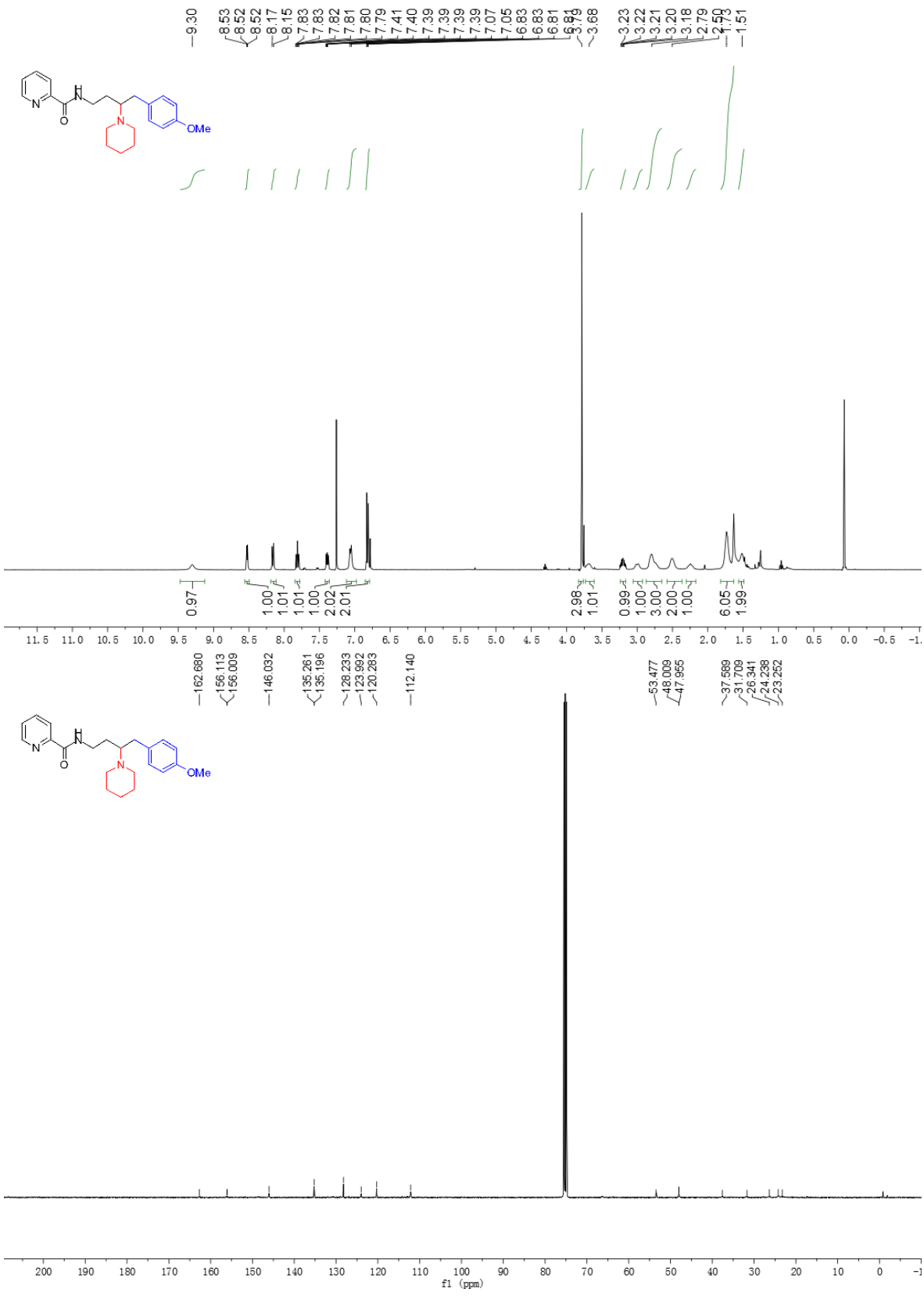
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7.39
7.39
7.39
7.39
7.07
7.05
6.83
6.83
6.81
6.81
3.79
3.68
3.23
3.22
3.21
3.20
3.18
2.79
2.50
1.73
1.51
OMe
0.97
1.00
1.01
1.01
1.00
2.02
2.01
2.98
1.01
0.99
1.00
3.00
2.00
1.00
6.05
1.99
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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135.261
135.196
128.233
123.992
120.283
112.140
53.477
48.009
47.955
37.589
31.709
26.341
24.238
23.252
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.135
8.528
8.516
8.174
8.154
7.837
7.833
7.817
7.813
7.798
7.794
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7.407
7.397
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7.390
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7.376
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7.141
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3.674
3.657
3.273
3.262
3.251
3.056
3.048
3.023
3.015
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2.777
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2.506
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2.325
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1.00
2.97
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5.10
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f1 (ppm)
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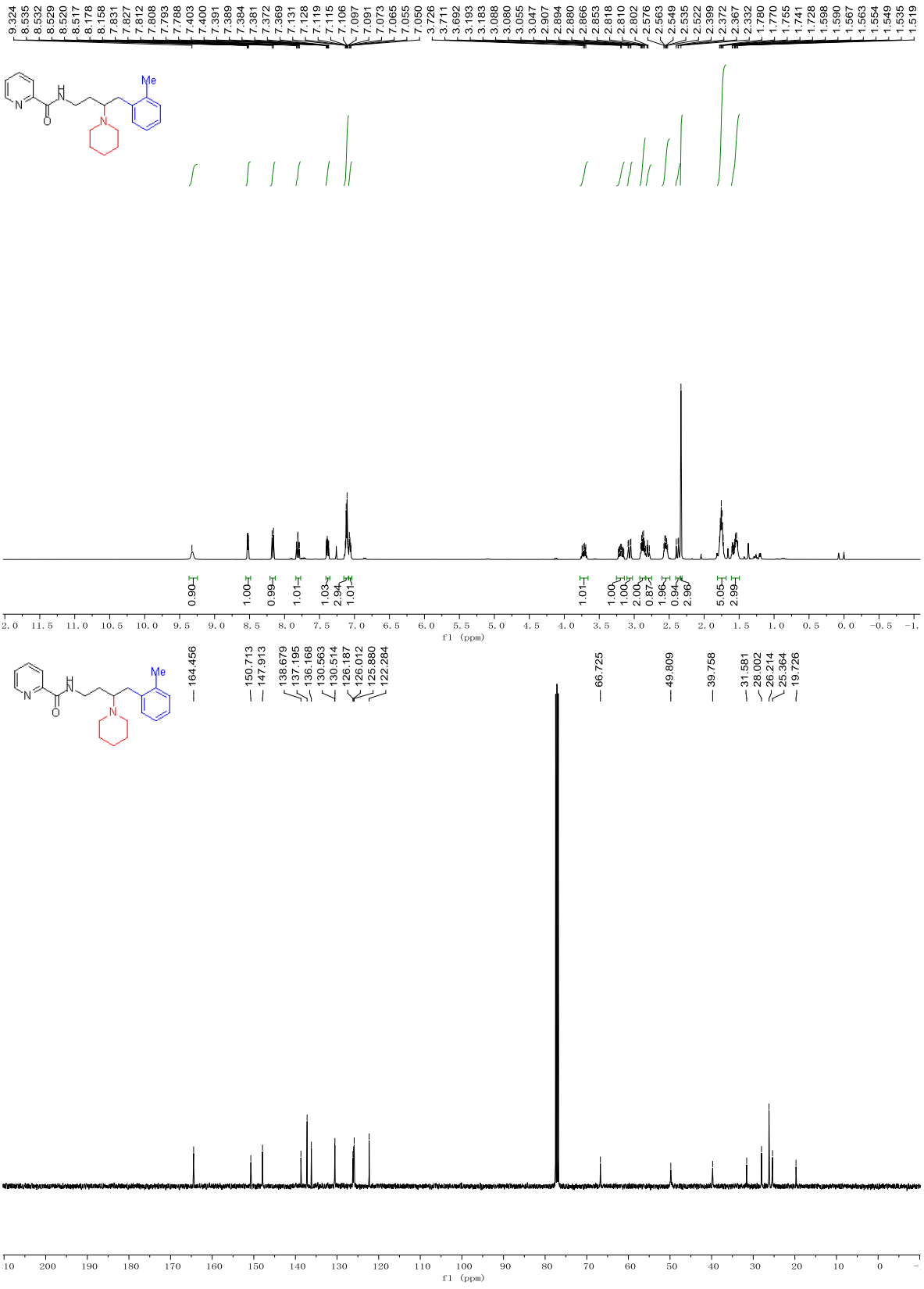
OCF3
f1 (ppm)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7.381
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3.055
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1.535
1.519
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O
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1.01
1.00
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0.94
2.96
5.05
2.99
f1 (ppm)
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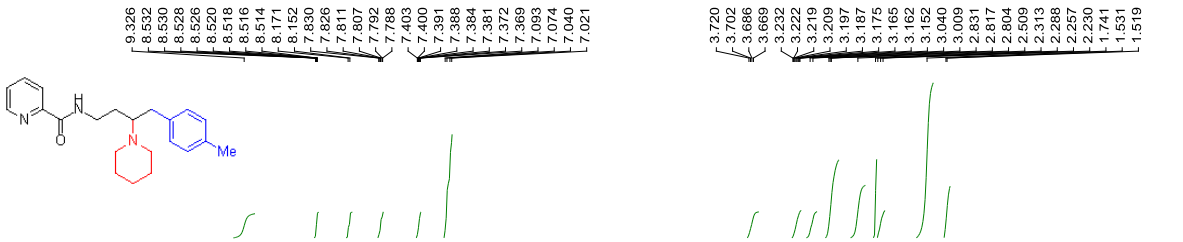
19.726
f1 (ppm)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7.792
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7.040
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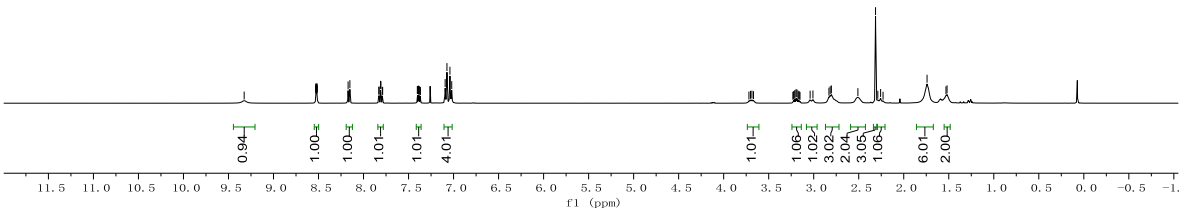
Me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1.06
6.01
2.00
f1 (ppm)


164.531
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137.213
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125.906
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N
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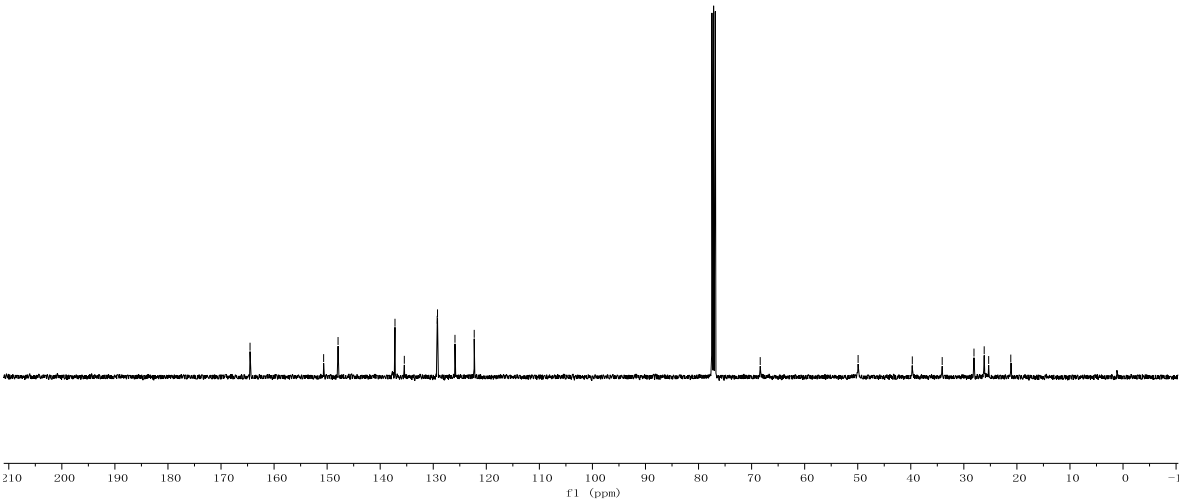
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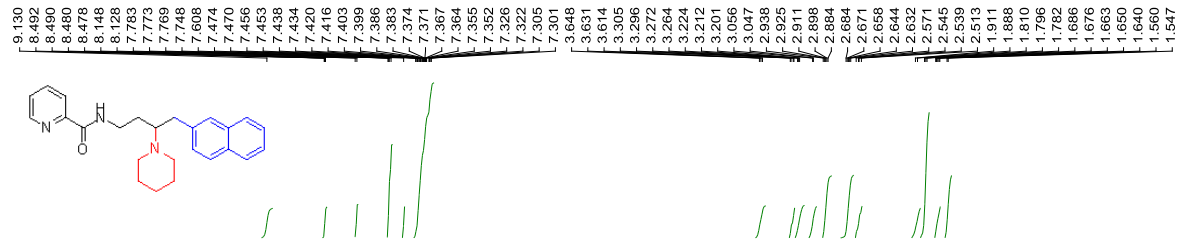
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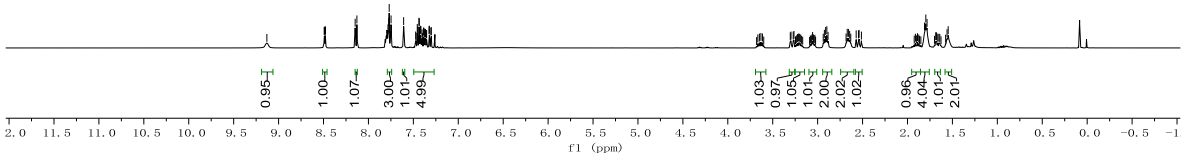
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4.04
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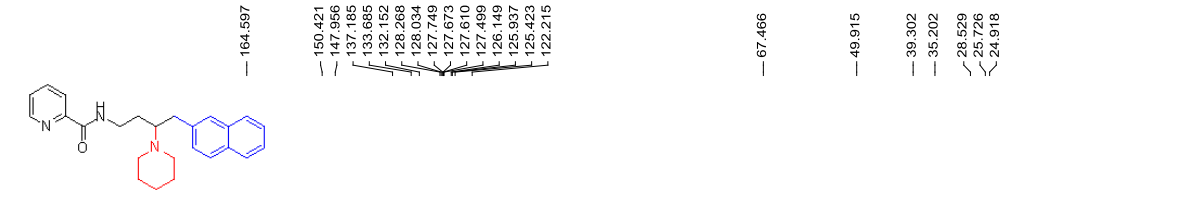
2.01
f1 (ppm)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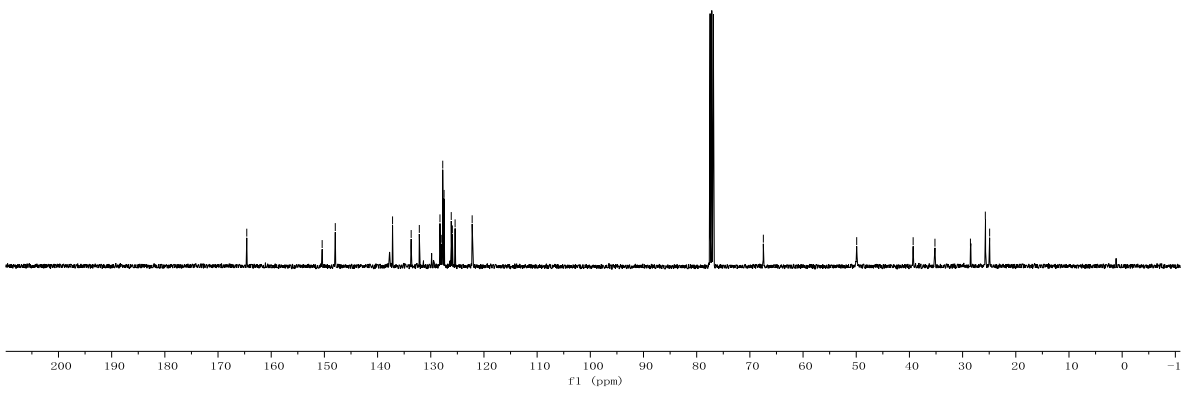
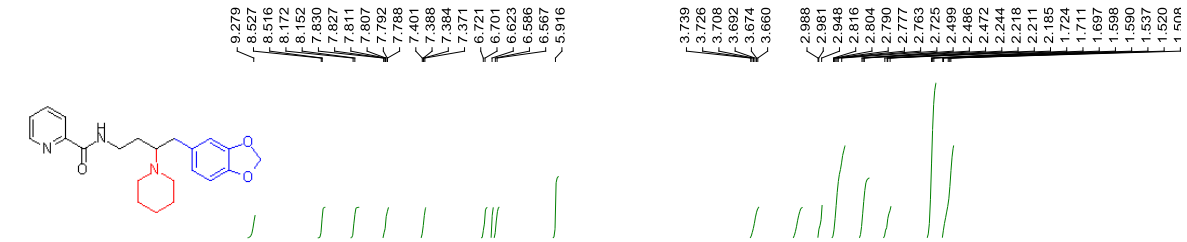

f1 (ppm)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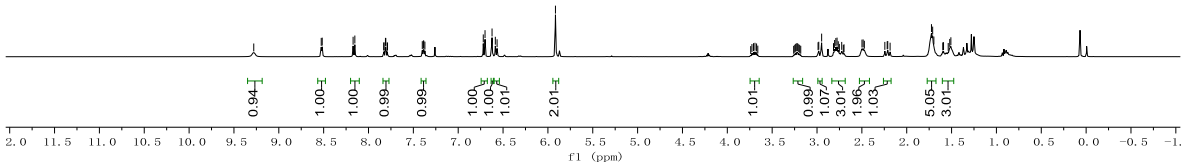
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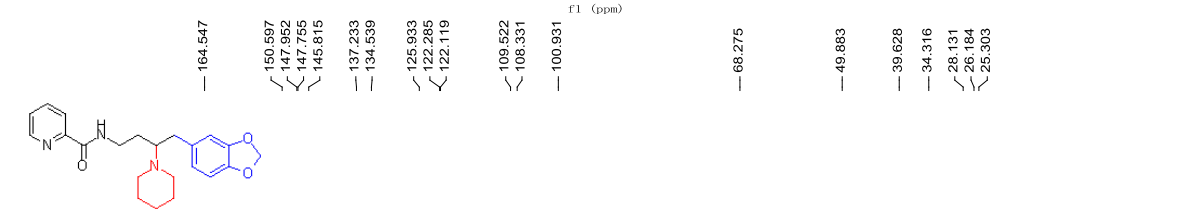
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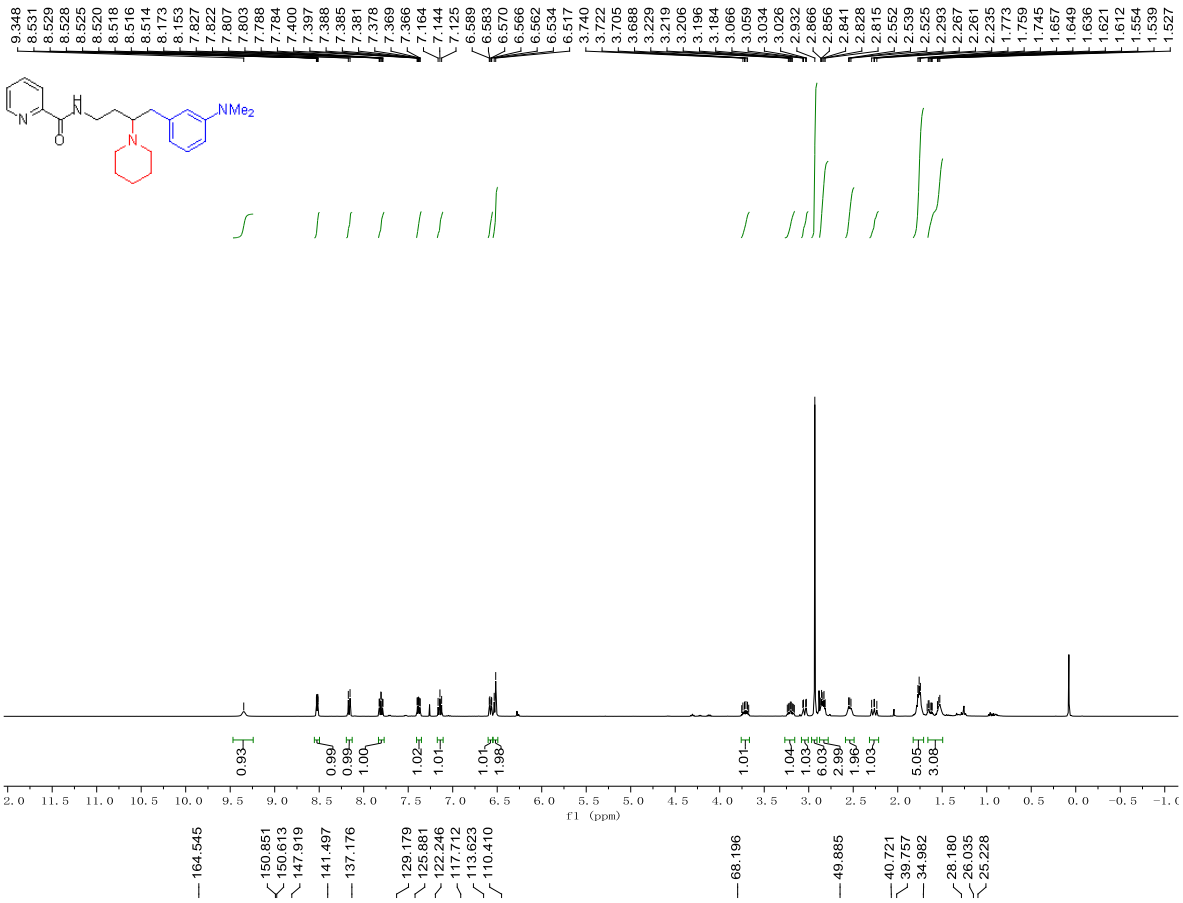
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6.03
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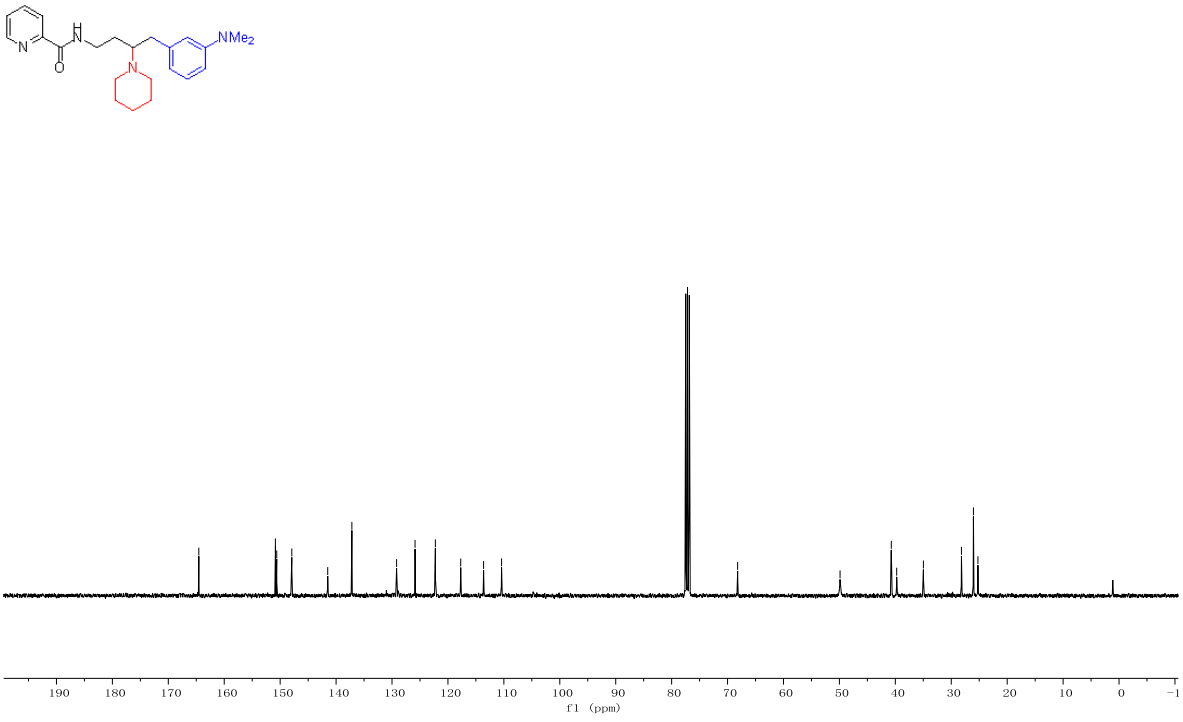
1.03
5.05
3.08
f1 (ppm)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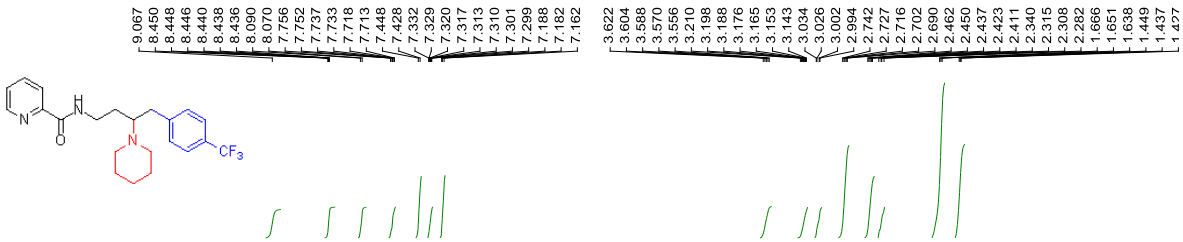
NMe2
f1 (ppm)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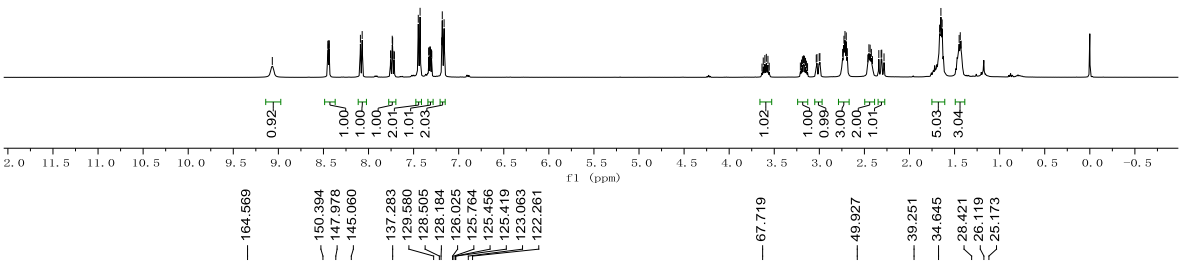
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5.03
3.04
f1 (ppm)


164.569
150.394
147.978
145.060
137.283
129.580
128.505
128.184
126.025
125.764
125.456
125.419
123.063
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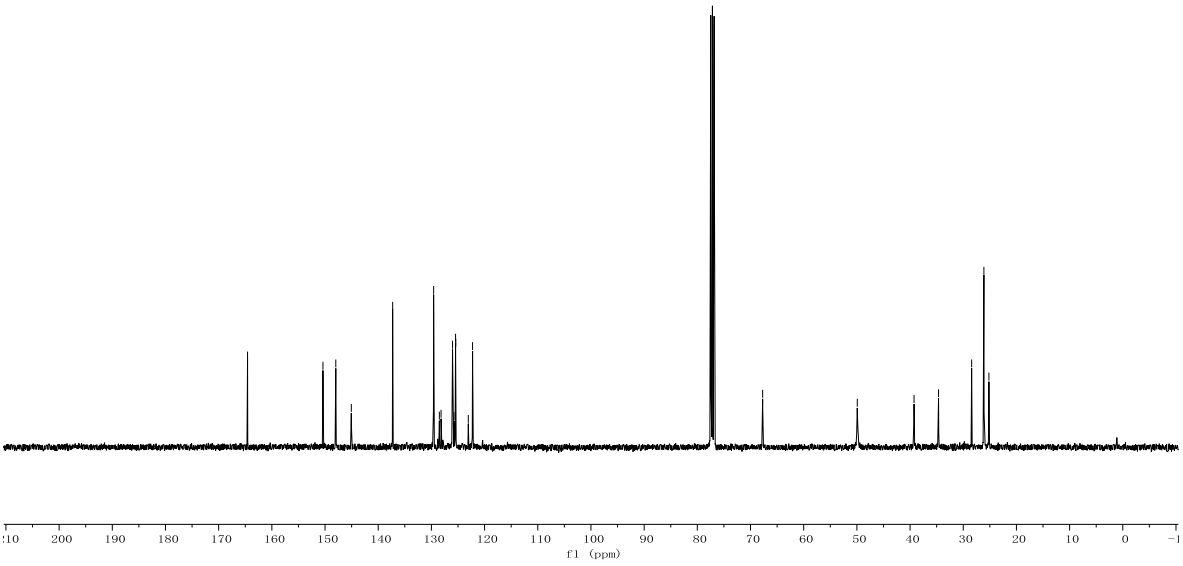
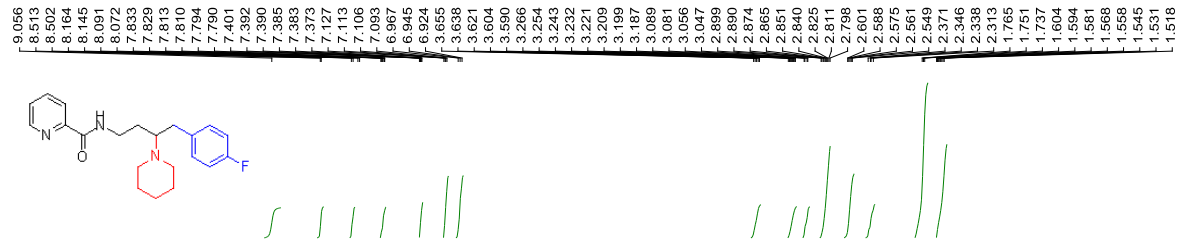
H
O
N
CF3


f1 (ppm)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7.790
7.401
7.392
7.390
7.385
7.383
7.373
7.127
7.113
7.106
7.093
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6.924
3.655
3.638
3.621
3.604
3.590
3.266
3.254
3.243
3.232
3.221
3.209
3.199
3.187
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3.056
3.047
2.899
2.890
2.874
2.865
2.851
2.840
2.825
2.811
2.798
2.601
2.588
2.575
2.561
2.549
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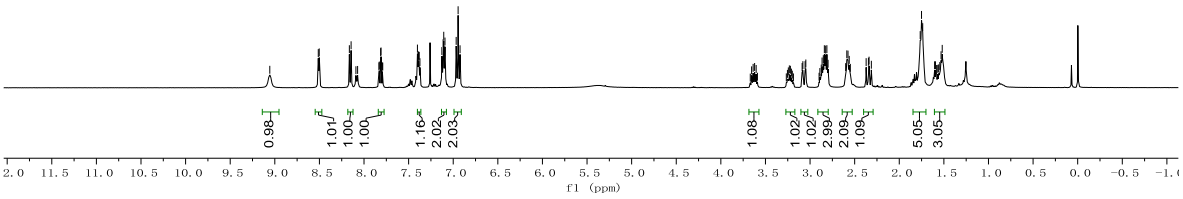
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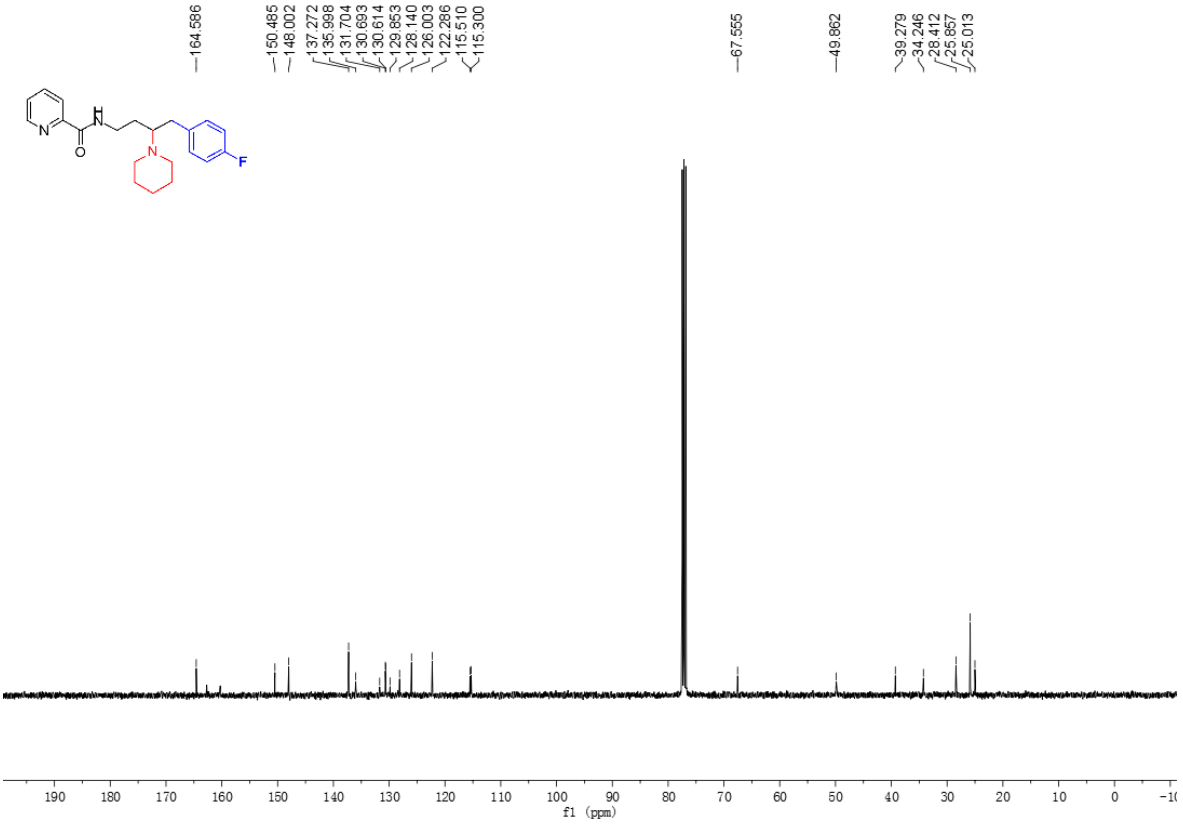
1.02
1.02
2.99
2.09
1.09
5.05
3.05
f1 (ppm)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115.300
67.555
49.862
39.279
34.246
28.412
25.857
25.013
f1 (ppm)


9.139
8.526
8.524
8.516
8.514
8.169
8.149
7.835
7.831
7.816
7.812
7.797
7.793
7.408
7.405
7.396
7.393
7.389
7.386
7.377
7.375
7.239
7.218
7.074
7.053
3.673
3.657
3.639
3.248
3.237
3.226
3.024
3.015
2.991
2.982
2.793
2.779
2.766
2.753
2.524
2.511
2.498
2.484
2.471
2.320
2.295
2.288
2.262
1.762
1.748
1.736
1.720
1.706
1.693
1.557
1.548
1.544
1.535
1.521
Cl
0.94
1.00
1.01
1.07
1.03
1.96
2.00
1.02
1.03
1.00
3.07
2.00
1.13
5.07
3.07
f1 (ppm)
164.577
150.500
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139.224
137.276
131.786
130.627
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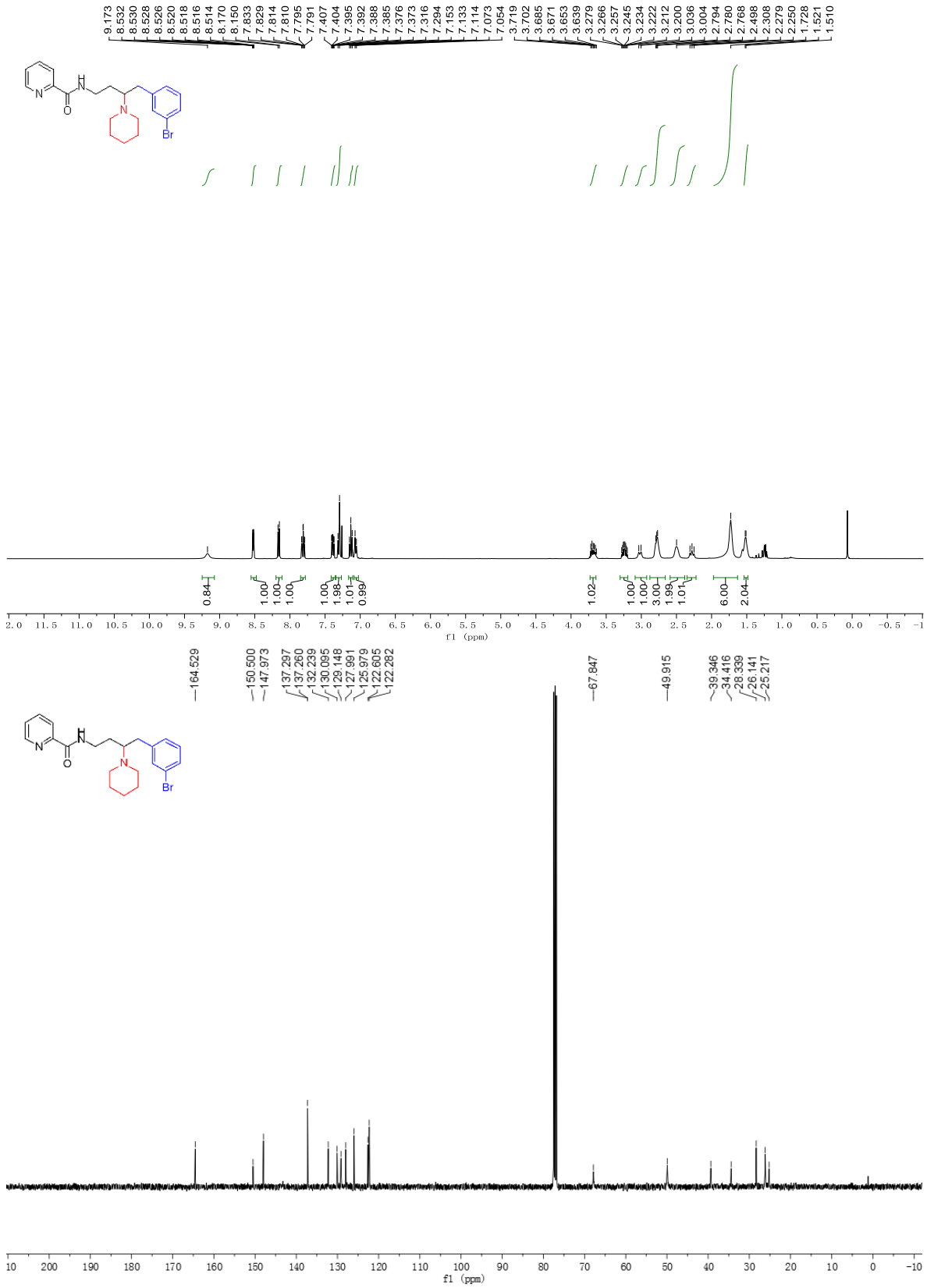
Cl
f1 (ppm)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7.833
7.829
7.814
7.810
7.795
7.791
7.407
7.404
7.395
7.392
7.388
7.385
7.376
7.373
7.316
7.294
7.153
7.133
7.114
7.073
7.054
3.719
3.702
3.685
3.671
3.653
3.639
3.279
3.266
3.257
3.245
3.234
3.222
3.212
3.200
3.036
3.004
2.794
2.780
2.768
2.498
2.308
2.279
2.250
1.728
1.521
1.510
0.84
1.00
1.00
1.00
1.00
1.98
1.01
0.99
1.02
1.00
1.00
3.00
1.99
1.01
6.00
2.04
f1 (ppm)
164.529
150.500
147.973
137.297
137.260
132.239
130.095
129.148
127.991
125.979
122.605
122.282
67.847
49.915
39.346
34.416
28.339
26.141
25.217
f1 (ppm)


9.159
8.533
8.531
8.529
8.527
8.521
8.519
8.517
8.515
8.166
8.146
7.820
7.816
7.587
7.583
7.571
7.566
7.414
7.411
7.402
7.399
7.395
7.392
6.909
6.888
3.703
3.689
3.671
3.655
3.637
3.623
3.224
3.021
3.013
2.988
2.980
2.803
2.789
2.775
2.762
2.525
2.512
2.499
2.303
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2.245
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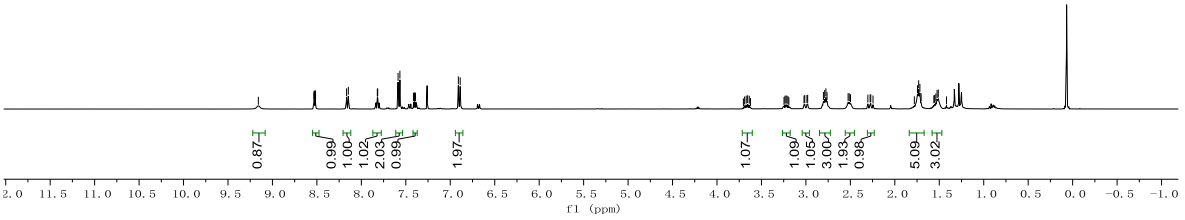
1.538
1.525
1.511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1.07
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1.05
3.00
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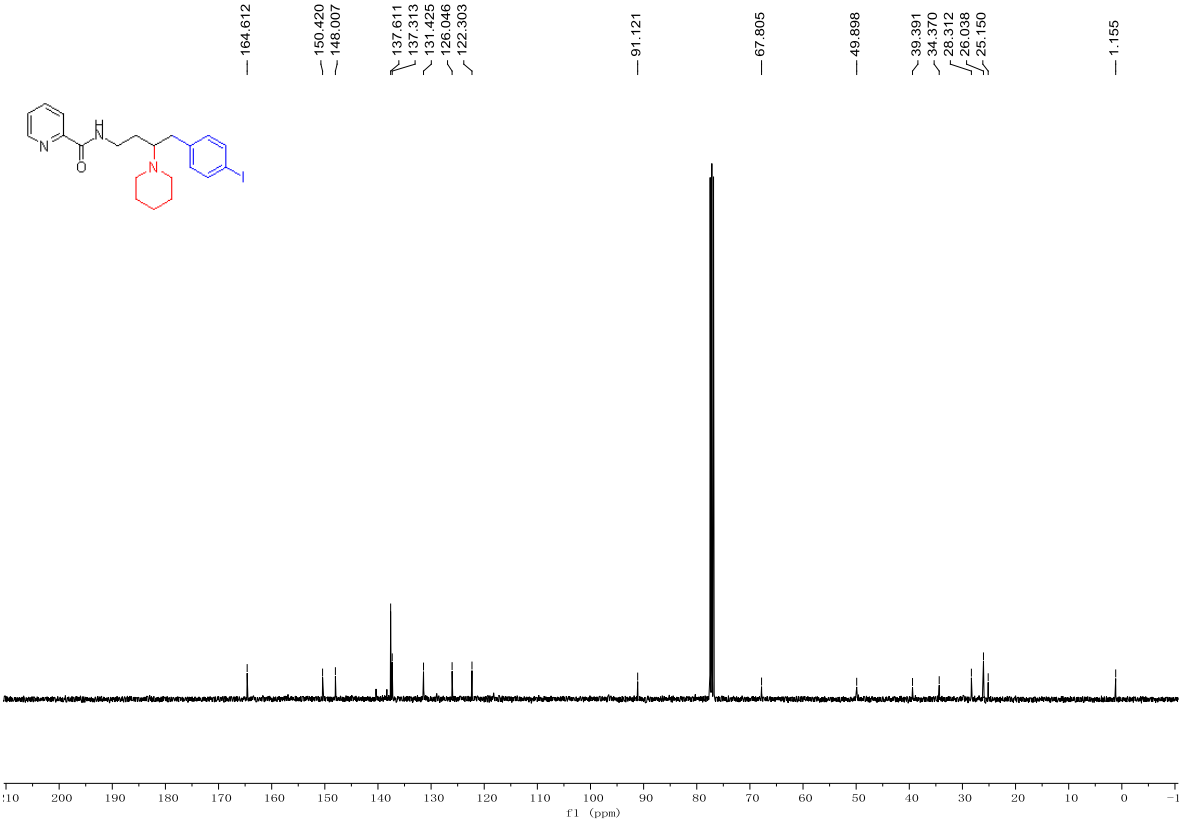
0.98
5.09
3.02
f1 (ppm)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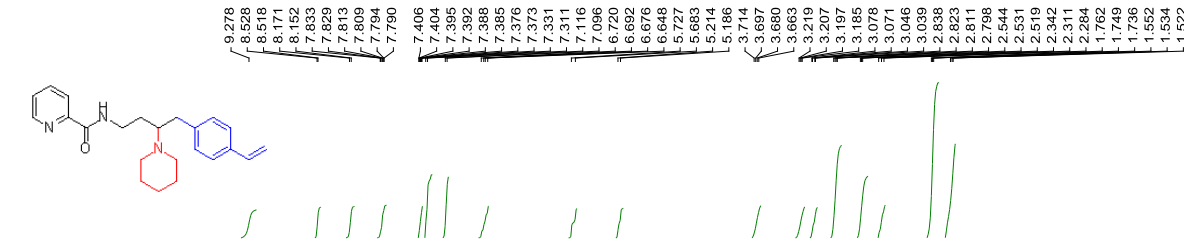
1.155
f1 (ppm)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8.171
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7.813
7.809
7.794
7.790
7.406
7.404
7.395
7.392
7.388
7.385
7.376
7.373
7.331
7.311
7.116
7.096
6.720
6.692
6.676
6.648
5.727
5.683
5.214
5.186
3.714
3.697
3.680
3.663
3.219
3.207
3.197
3.185
3.078
3.071
3.046
3.039
2.838
2.823
2.811
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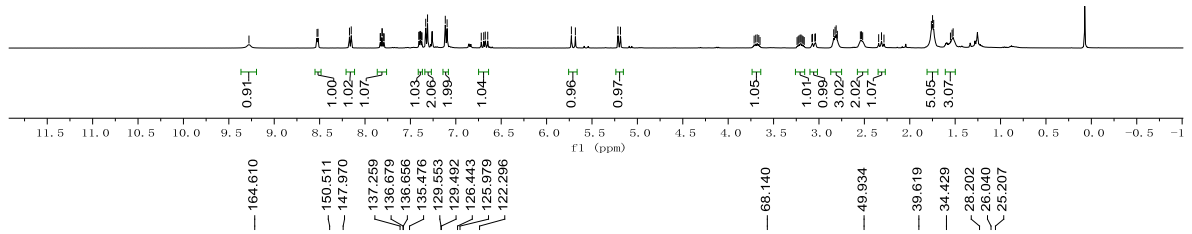
1.534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0.97
1.05
1.01
0.99
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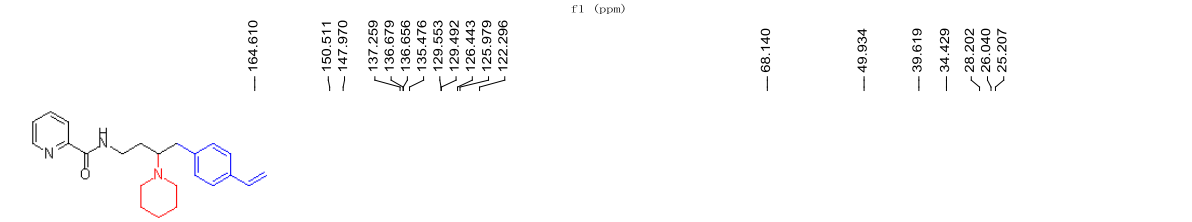
2.02
1.07
5.05
3.07
f1 (ppm)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68.140
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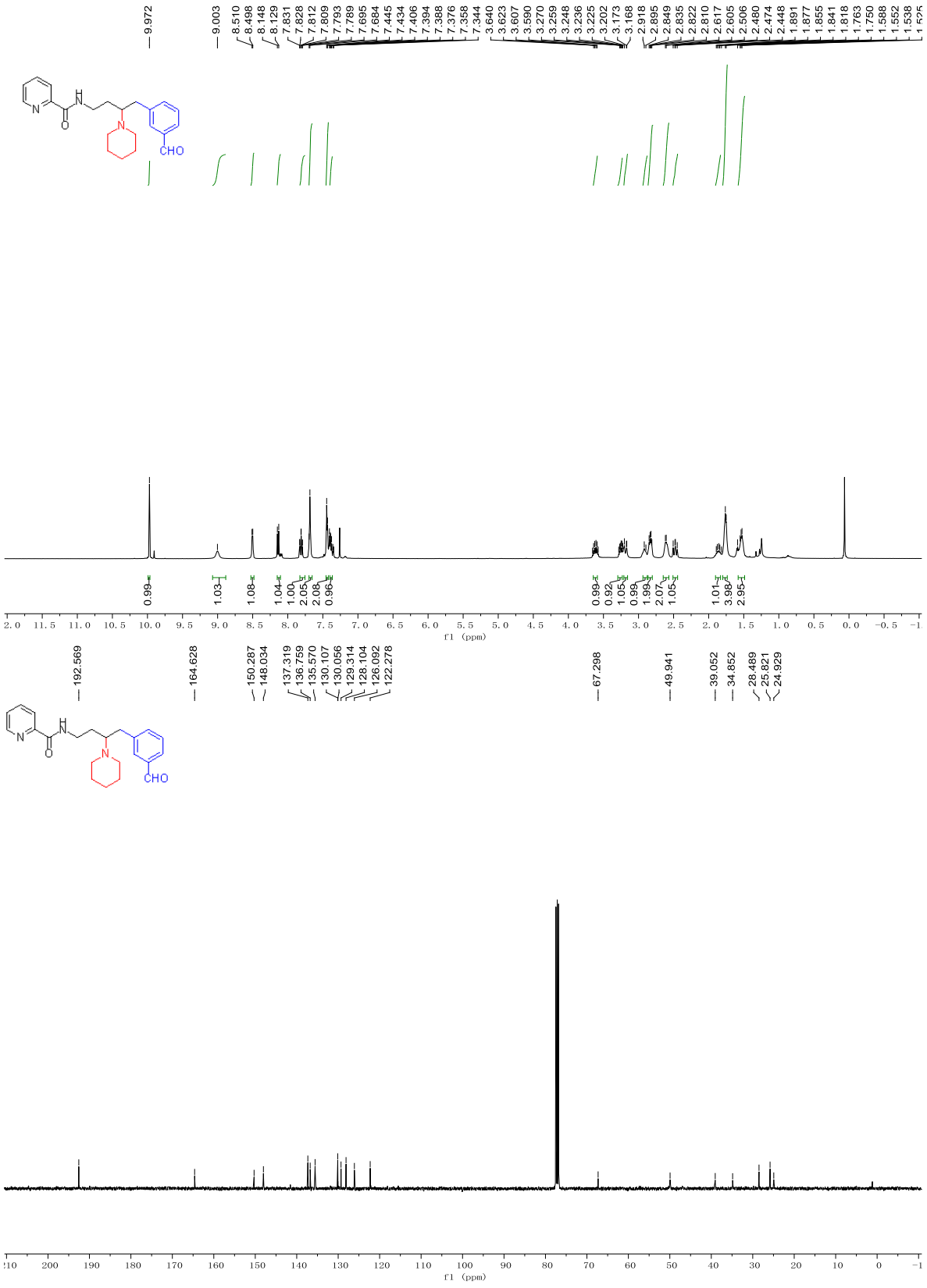
f1 (ppm)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7.809
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7.695
7.684
7.445
7.434
7.406
7.394
7.388
7.376
7.358
7.344
3.640
3.623
3.607
3.590
3.270
3.259
3.248
3.236
3.225
3.202
3.173
3.168
2.918
2.895
2.849
2.835
2.822
2.810
2.617
2.605
2.506
2.480
2.474
2.448
1.891
1.877
1.855
1.841
1.818
1.763
1.750
1.588
1.552
1.538
N
H
O
N
CHO
0.99
1.03
1.08
1.04
1.00
2.05
2.08
0.96
0.99
0.92
1.05
0.99
1.99
2.07
1.05
1.01
3.98
2.95
f1 (ppm)
192.569
164.628
150.287
148.034
137.319
136.759
135.570
130.107
130.056
129.314
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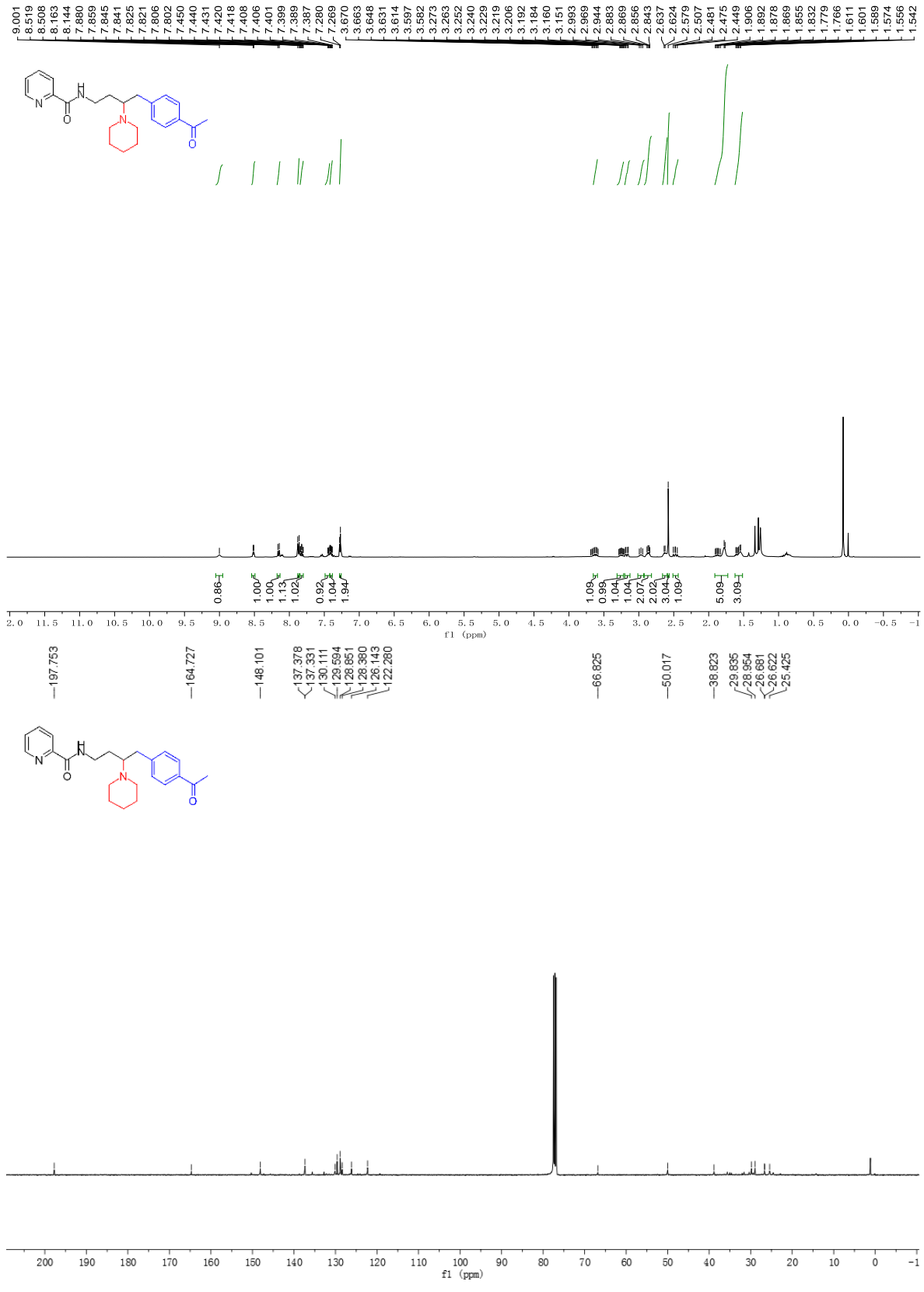
f1 (ppm)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7.825
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7.802
7.450
7.440
7.431
7.420
7.418
7.408
7.406
7.401
7.399
7.389
7.387
7.280
7.269
3.670
3.663
3.648
3.631
3.614
3.597
3.582
3.273
3.263
3.252
3.240
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3.206
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3.184
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3.151
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2.969
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2.869
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2.843
2.637
2.624
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1.869
1.855
1.832
1.779
1.766
1.611
1.601
1.589
1.574
1.556
1.542
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1.00
1.13
1.02
0.92
1.04
1.94
1.09
0.99
1.04
1.04
2.07
2.02
3.04
1.09
5.09
3.09
f1 (ppm)
197.753
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137.378
137.331
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129.594
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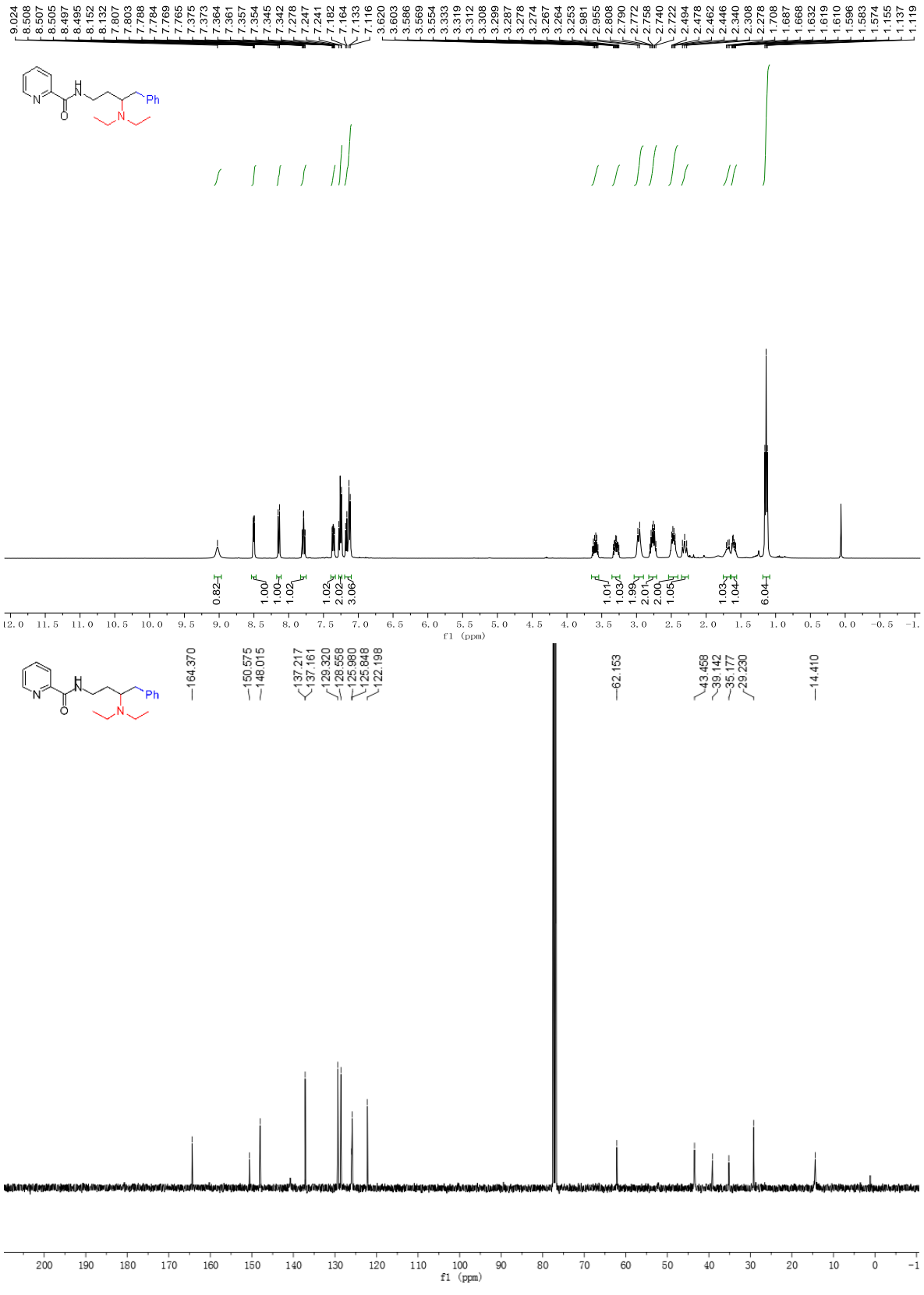
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7.375
7.373
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7.361
7.357
7.354
7.345
7.342
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7.247
7.241
7.182
7.164
7.133
7.116
3.620
3.603
3.586
3.569
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3.278
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3.267
3.264
3.253
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2.772
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2.740
2.722
2.494
2.478
2.462
2.446
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2.278
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1.632
1.619
1.610
1.596
1.583
1.574
1.155
1.137
1.119
Ph
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1.00
1.02
1.02
2.02
3.06
1.01
1.03
1.99
2.01
2.00
1.05
1.03
1.04
6.04
f1 (ppm)
Ph
164.370
150.575
148.015
137.217
137.161
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128.558
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125.848
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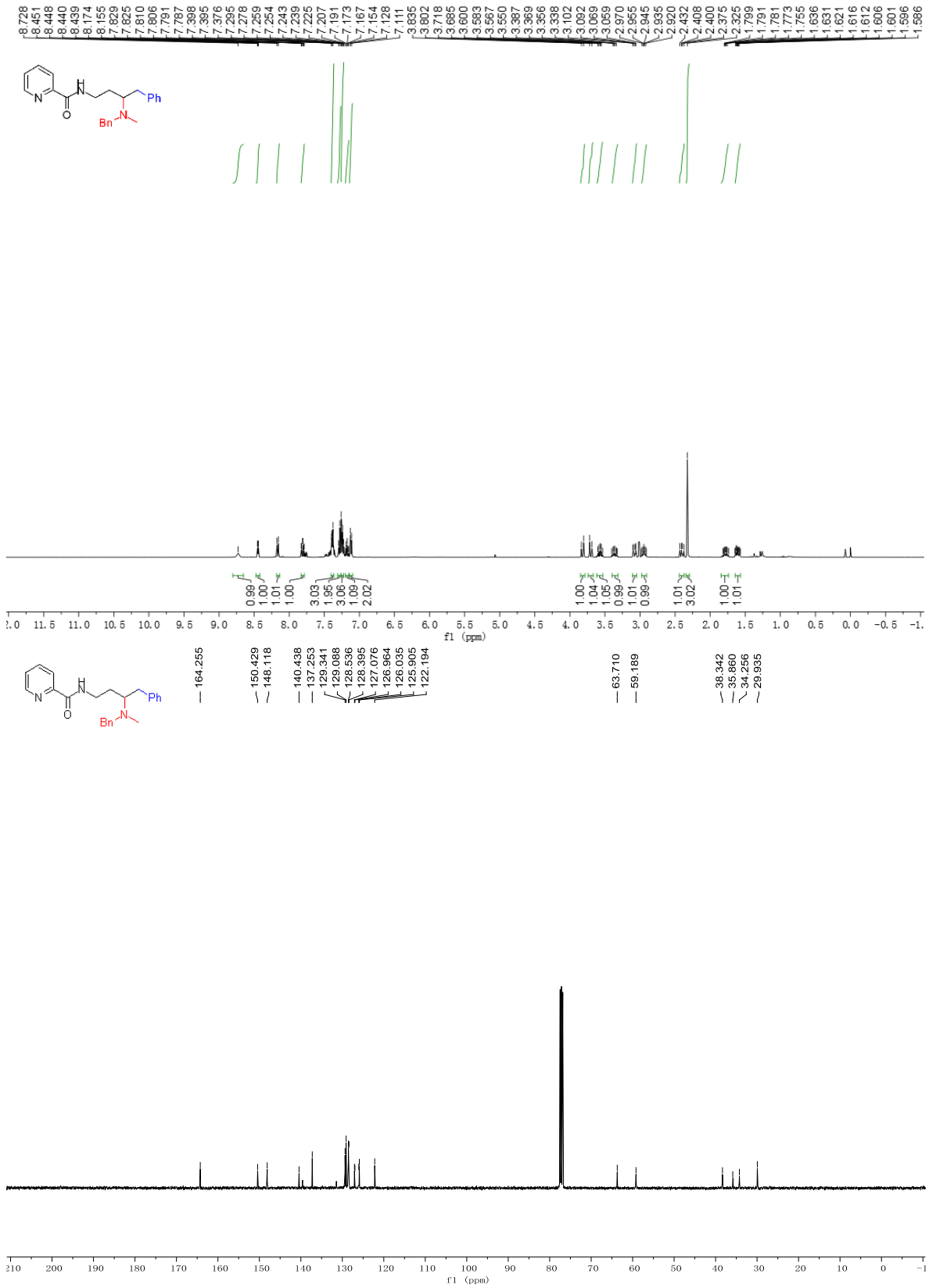
29.230
14.410
f1 (ppm)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7.376
7.295
7.278
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7.243
7.239
7.225
7.207
7.191
7.173
7.167
7.154
7.128
7.111
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3.685
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1.791
1.781
1.773
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1.636
1.631
1.621
1.616
1.612
1.606
1.601
1.596
1.586
Ph
Bn
0.99
1.00
1.01
1.00
3.03
1.95
3.06
1.09
2.02
1.00
1.04
1.05
0.99
1.01
0.99
1.01
3.02
1.00
1.01
f1 (ppm)
164.255
150.429
148.118
140.438
137.253
129.341
129.088
128.536
128.395
127.076
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35.860
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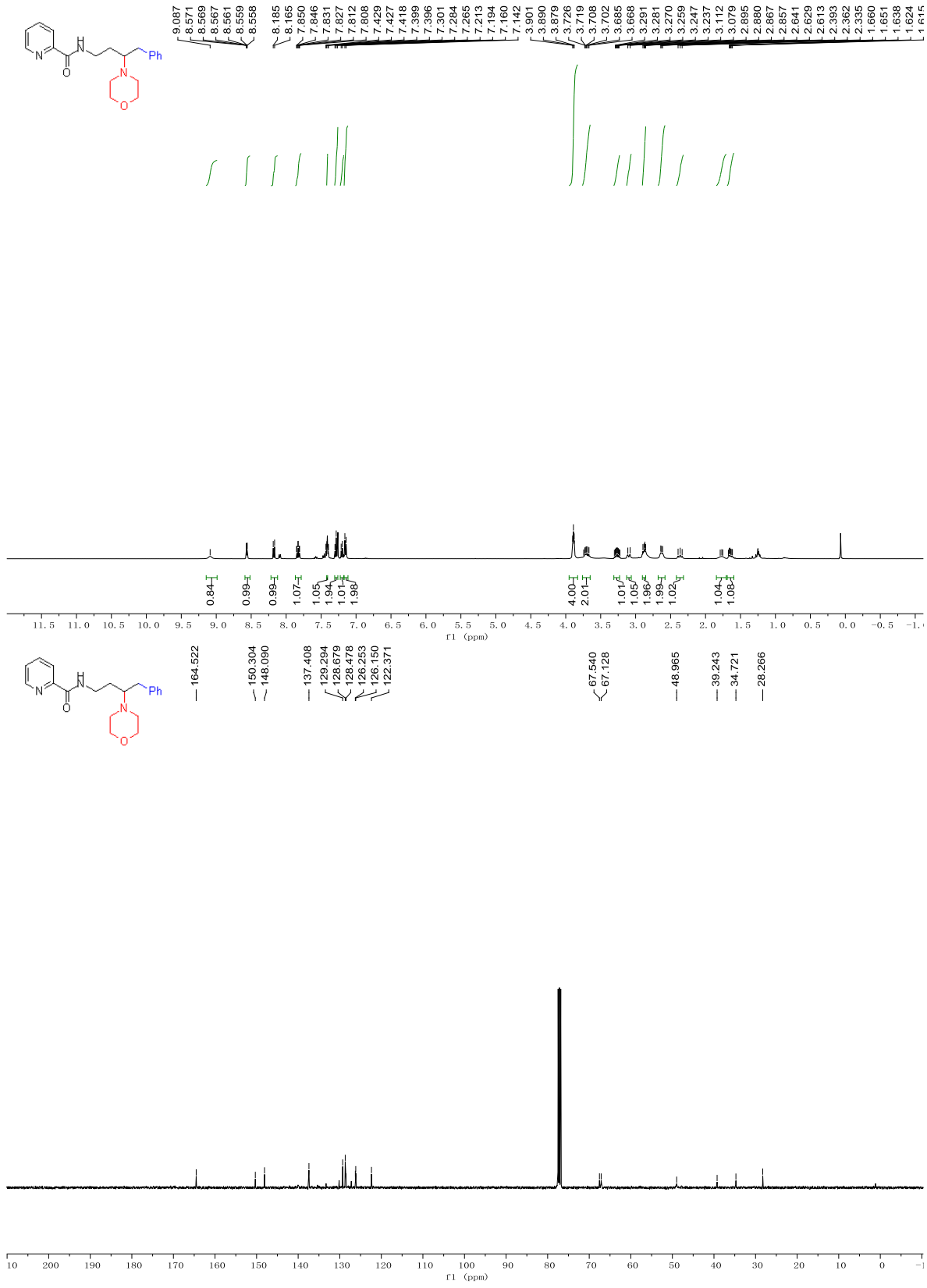
29.935
f1 (ppm)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7.812
7.808
7.429
7.427
7.418
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7.396
7.301
7.284
7.265
7.213
7.194
7.160
7.142
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3.890
3.879
3.726
3.719
3.708
3.702
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3.291
3.281
3.270
3.259
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3.237
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2.857
2.641
2.629
2.613
2.393
2.362
2.335
1.660
1.651
1.638
1.624
Ph
0.84
0.99
0.99
1.07
1.05
1.94
1.01
1.98
4.00
2.01
1.01
1.05
1.96
1.99
1.02
1.04
1.08
f1 (ppm)
164.522
150.304
148.090
137.408
129.294
128.679
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122.371
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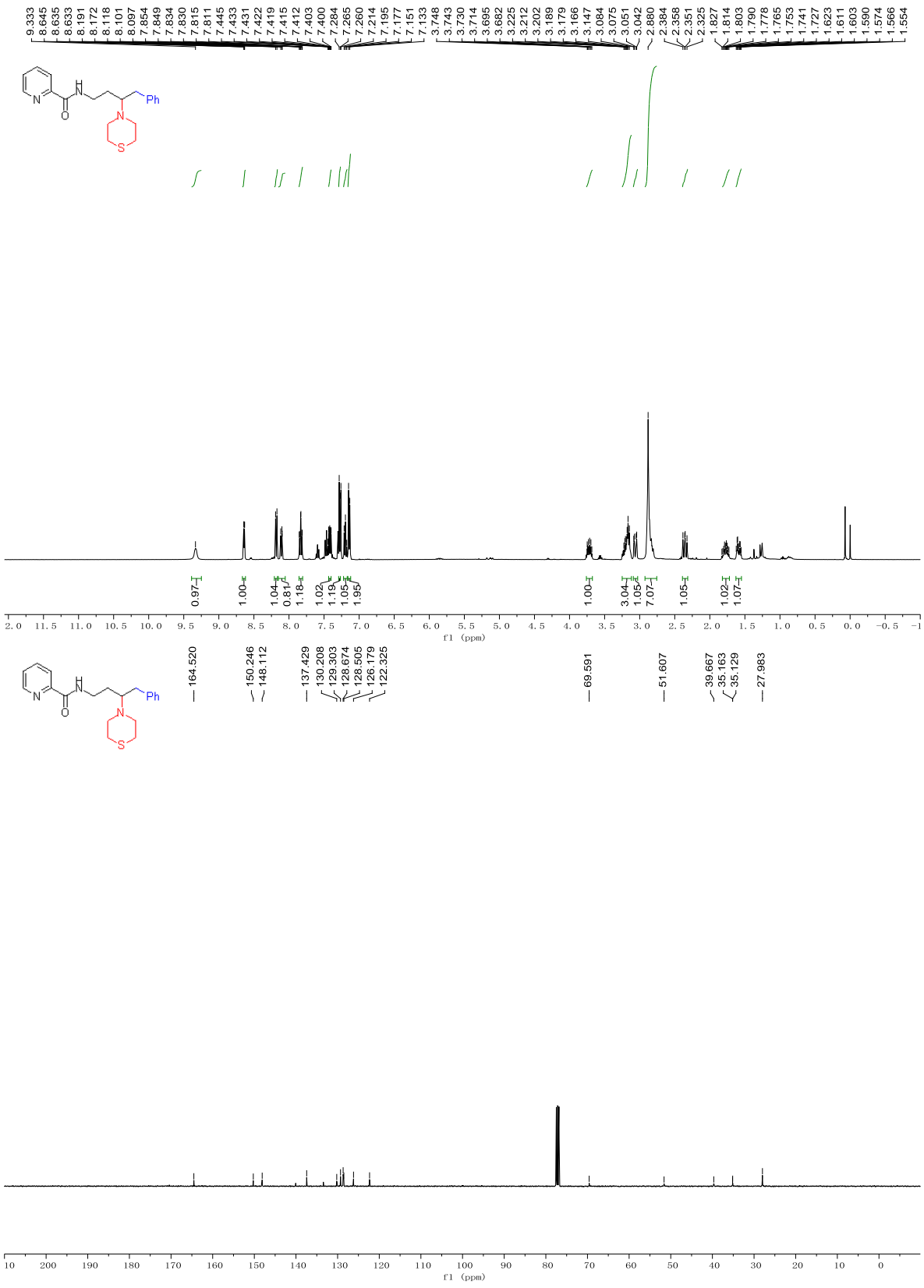
48.965
39.243
34.721
28.266
f1 (ppm)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Ph
0.97 1.00 1.04 0.81 1.18 1.02 1.19 1.05 1.95 1.00 3.04 1.05 7.07 1.05 1.02 1.07
f1 (ppm)
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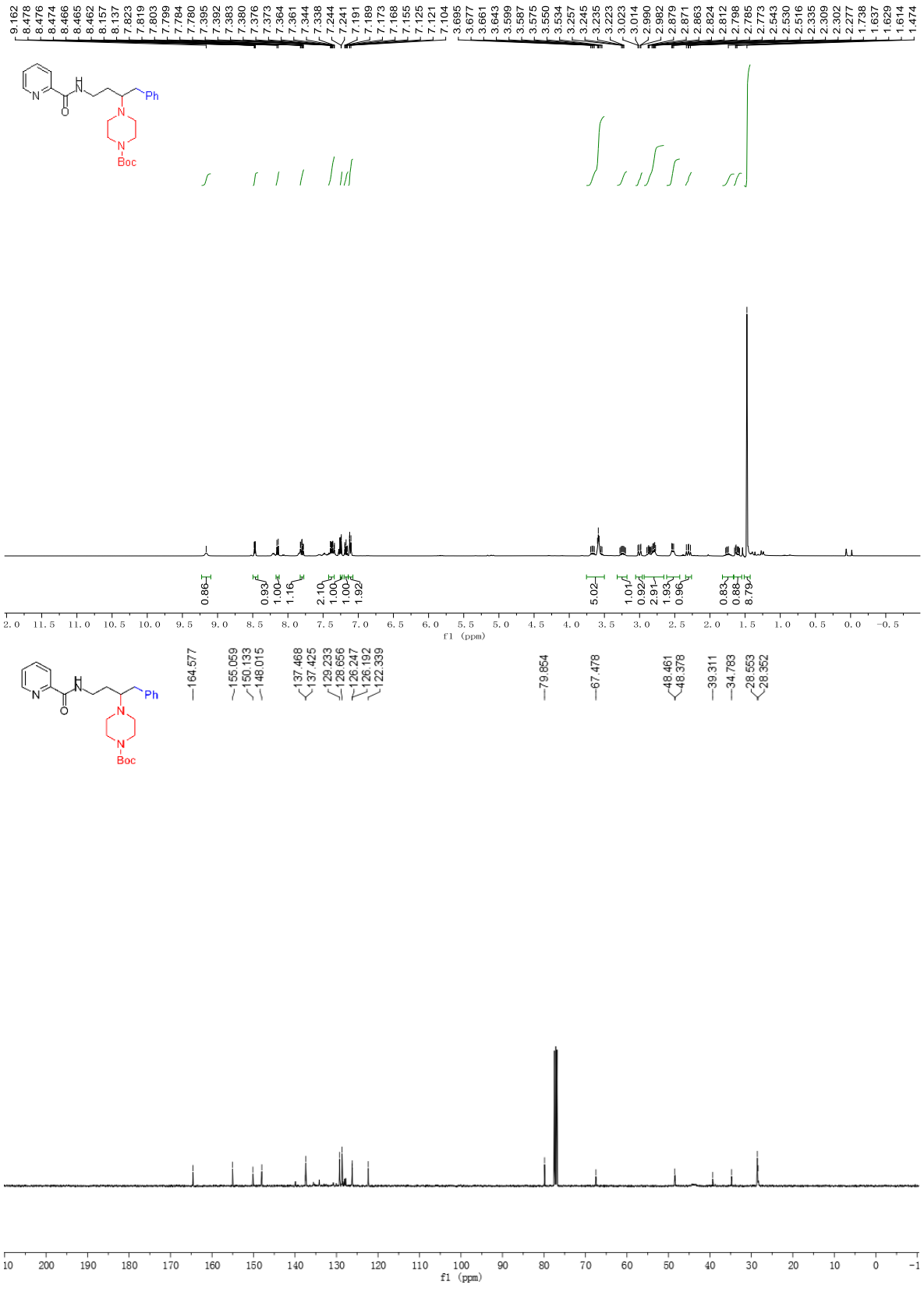
f1 (ppm)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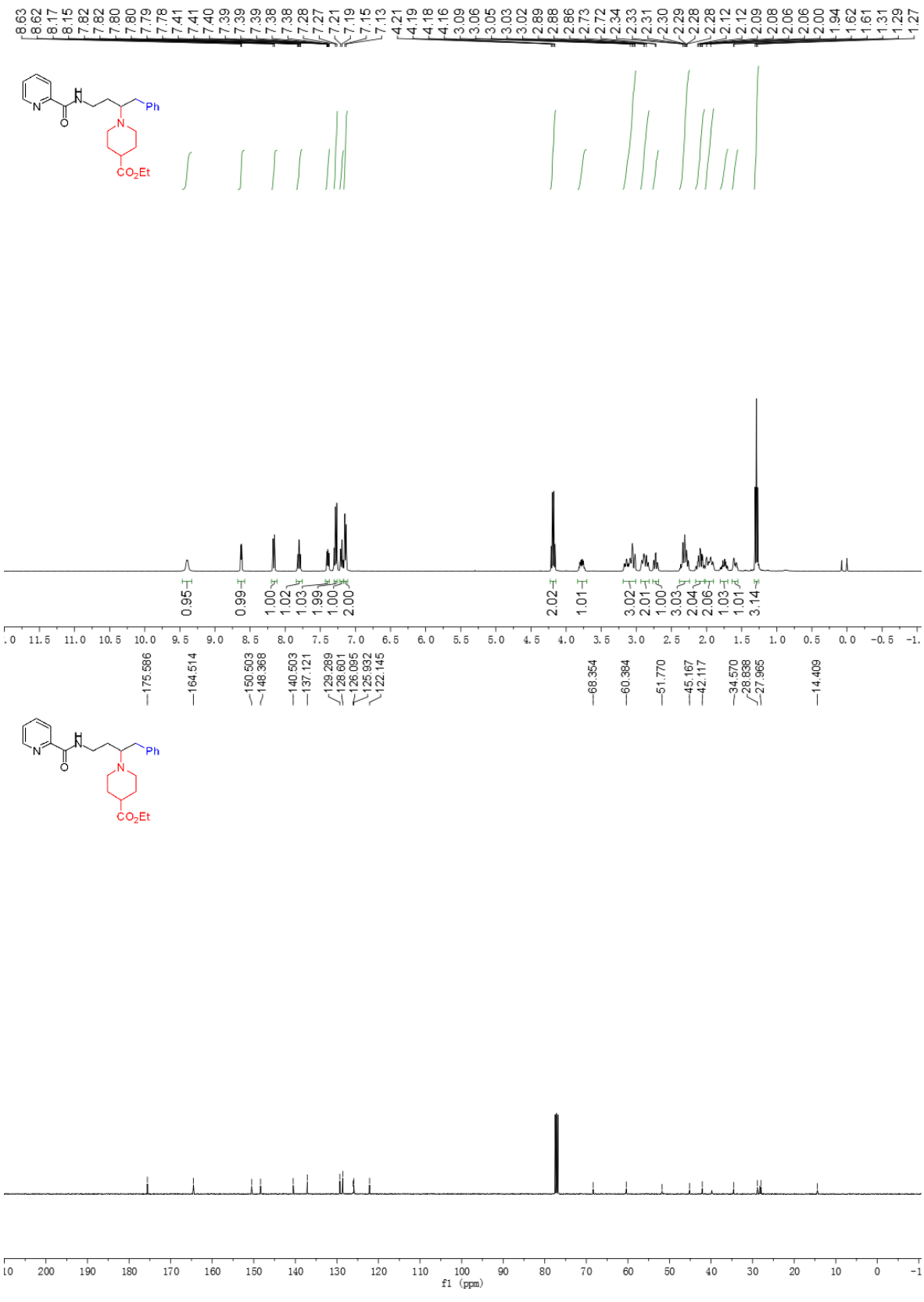
Ph
N
Boc
0.86 0.93 1.00 1.16 2.10 1.00 1.00 1.92 5.02 1.01 0.92 2.91 1.93 0.96 0.83 0.88 8.79
f1 (ppm)
164.577 155.059 150.133 148.015 137.468 137.425 129.233 128.856 126.247 126.192 122.339 79.854 67.478 48.461 48.378 39.311 34.783 28.553 28.352
f1 (ppm)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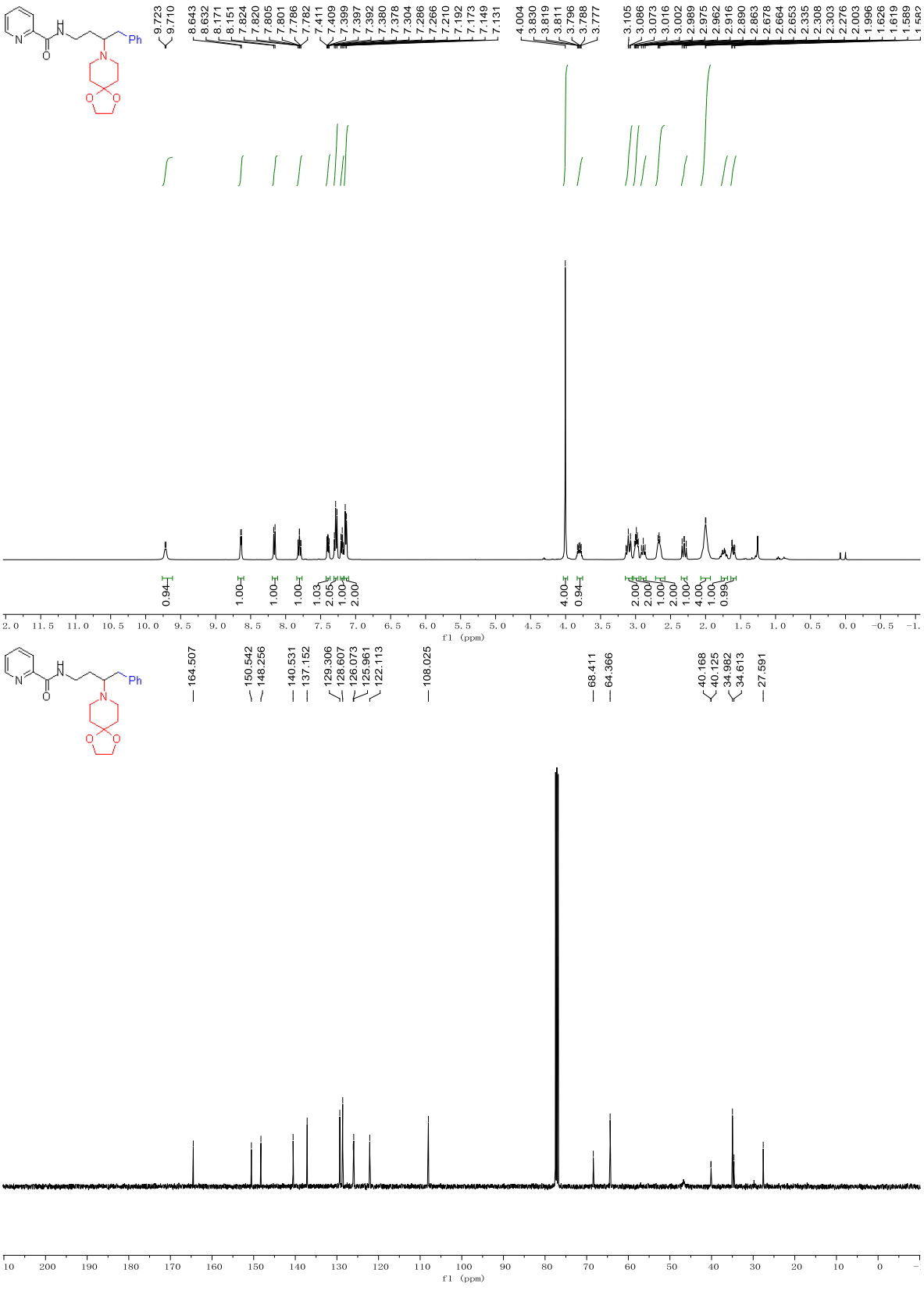
Ph
N
O
N
CO2Et
0.95 0.99 1.00 1.02 1.03 1.99 1.00 2.00 2.02 1.01 3.02 2.01 1.00 3.03 2.04 2.06 1.03 1.01 3.14
175.586 164.514 150.503 148.368 140.503 137.121 129.289 128.601 126.095 125.932 122.145 68.354 60.384 51.770 45.167 42.117 34.570 28.838 27.965 14.409
f1 (ppm)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7.411
7.409
7.399
7.397
7.392
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7.378
7.304
7.286
7.266
7.210
7.192
7.173
7.149
7.131
4.004
3.830
3.819
3.811
3.796
3.788
3.777
3.105
3.086
3.073
3.016
3.002
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2.975
2.962
2.916
2.890
2.863
2.678
2.664
2.653
2.335
2.308
2.303
2.276
2.003
1.996
1.626
1.619
1.589
0.94
1.00
1.00
1.00
1.03
2.05
1.00
2.00
4.00
0.94
2.00
2.00
1.00
2.00
1.00
4.00
1.00
0.99
f1 (ppm)
164.507
150.542
148.256
140.531
137.152
129.306
128.607
126.073
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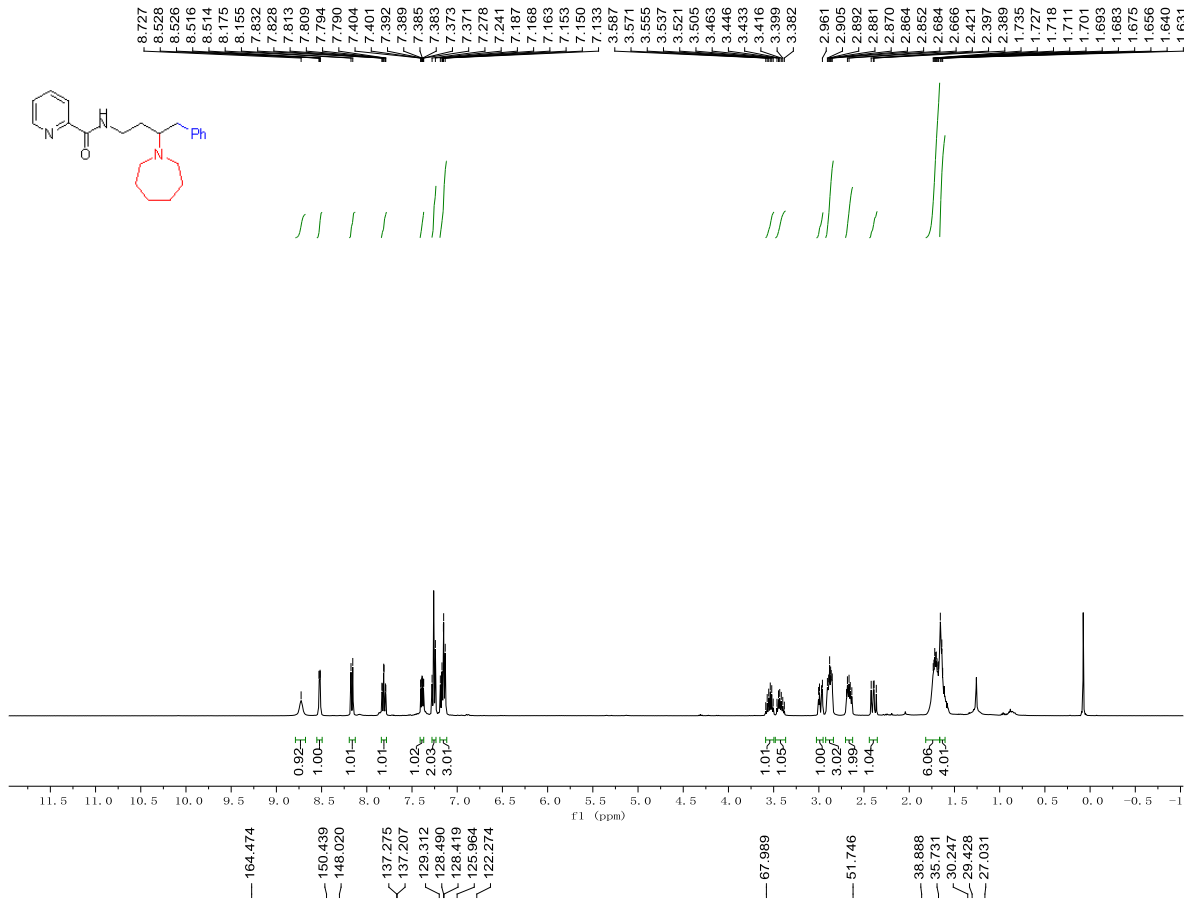
108.025
68.411
64.366
40.168
40.125
34.982
34.613
27.591
f1 (ppm)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7.790
7.404
7.401
7.392
7.389
7.385
7.383
7.373
7.371
7.278
7.241
7.187
7.168
7.163
7.153
7.150
7.133
3.587
3.571
3.555
3.537
3.521
3.505
3.463
3.446
3.433
3.416
3.399
3.382
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2.905
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2.881
2.870
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2.852
2.684
2.666
2.421
2.397
2.389
1.735
1.727
1.718
1.711
1.701
1.693
1.683
1.675
1.656
1.640
Ph
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1.01
1.01
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1.05
1.00
3.02
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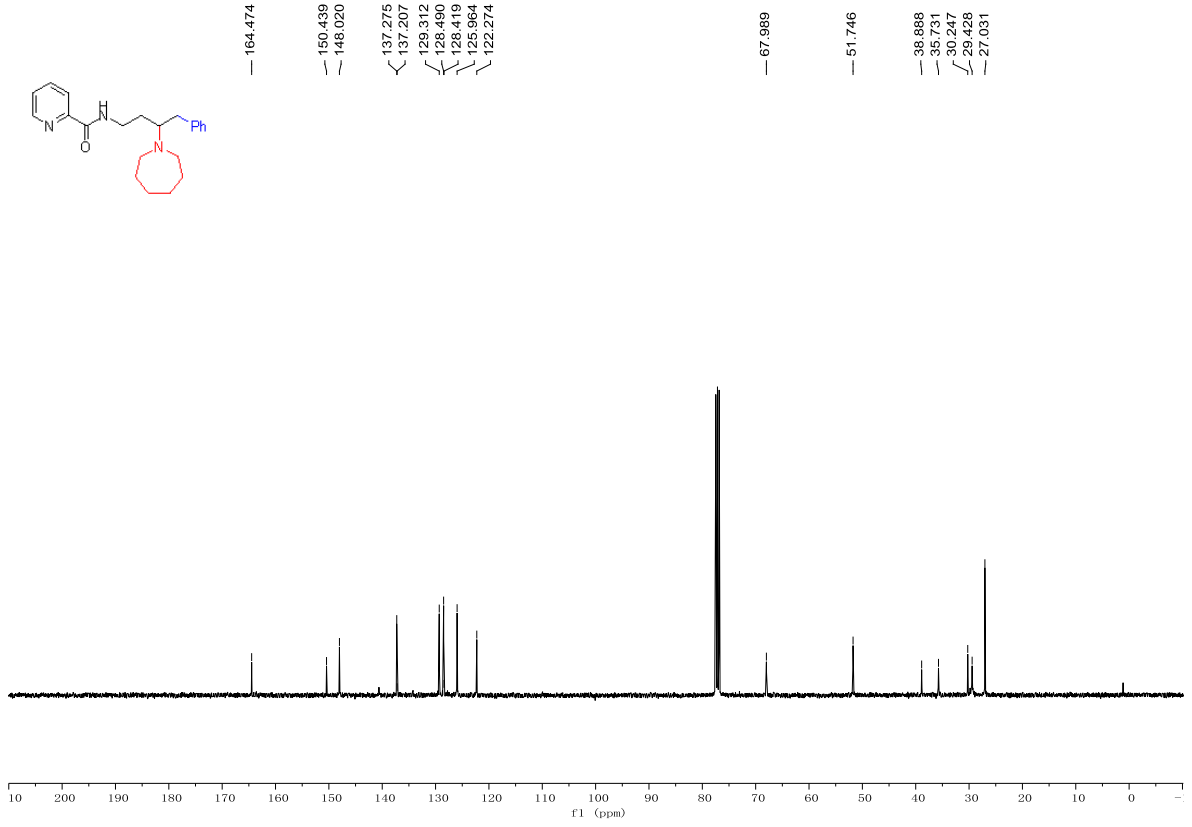
f1 (ppm)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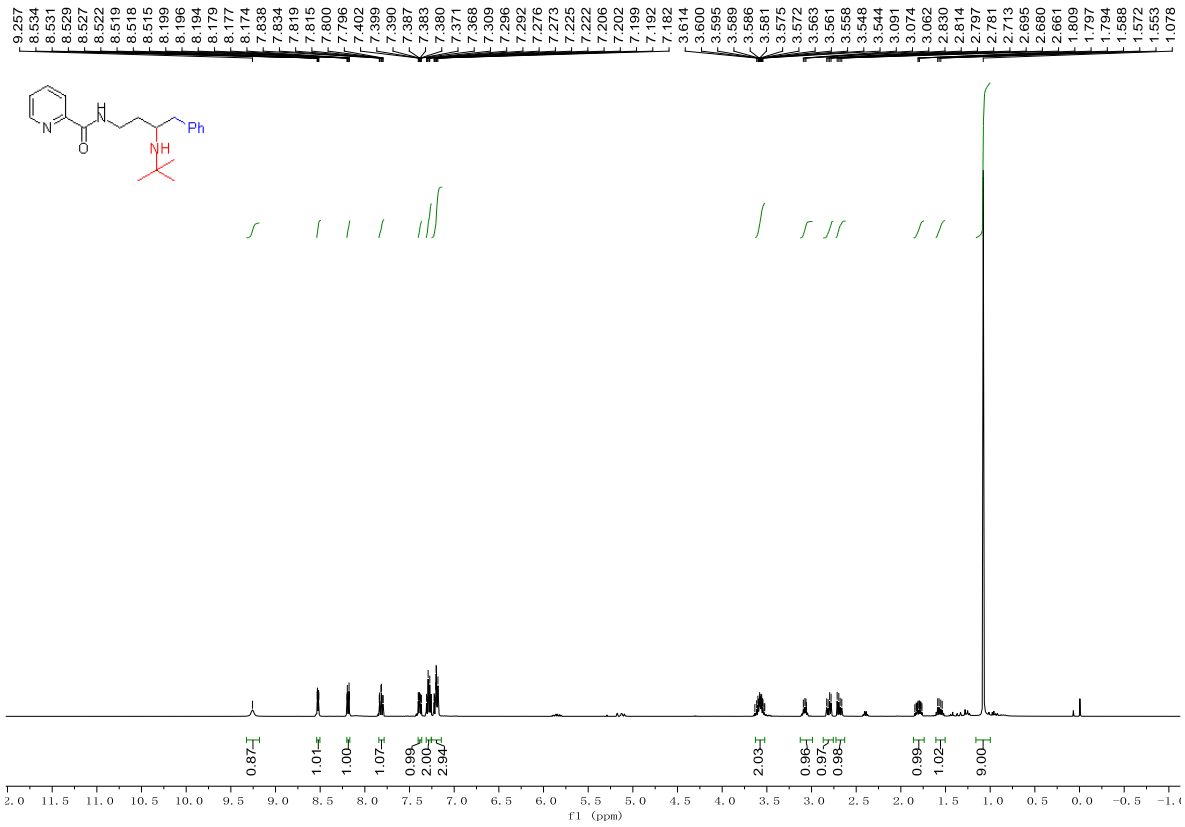
30.247
29.428
27.031
Ph
f1 (ppm)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8.177
8.174
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7.834
7.819
7.815
7.800
7.796
7.402
7.399
7.390
7.387
7.383
7.380
7.371
7.368
7.309
7.296
7.292
7.276
7.273
7.225
7.222
7.206
7.202
7.199
7.192
7.182
3.614
3.600
3.595
3.589
3.586
3.581
3.575
3.572
3.563
3.561
3.558
3.548
3.544
3.091
3.074
3.062
2.830
2.814
2.797
2.781
2.713
2.695
2.680
2.661
1.809
1.797
1.794
1.588
1.572
1.553
1.078
Ph
NH
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1.01
1.00
1.07
0.99
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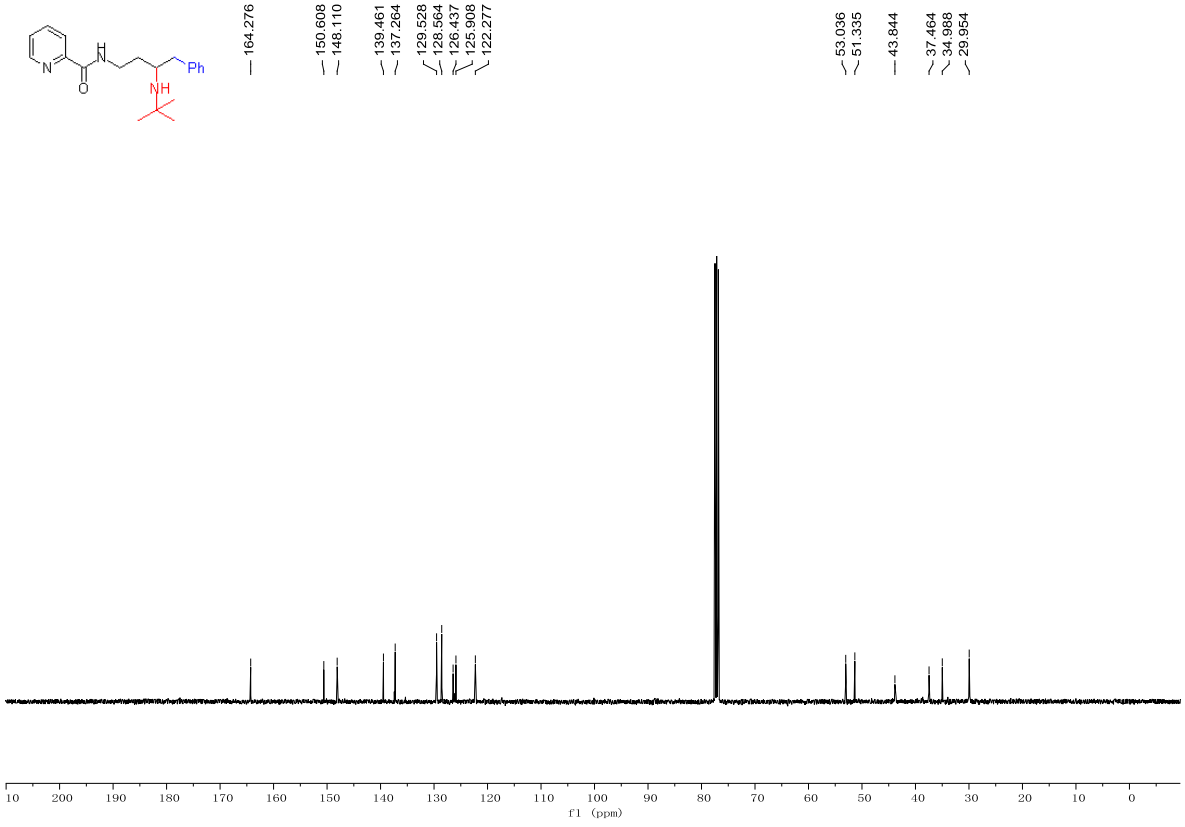
2.94
2.03
0.96
0.97
0.98
0.99
1.02
9.00
f1 (ppm)


Ph
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125.908
122.277
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51.335
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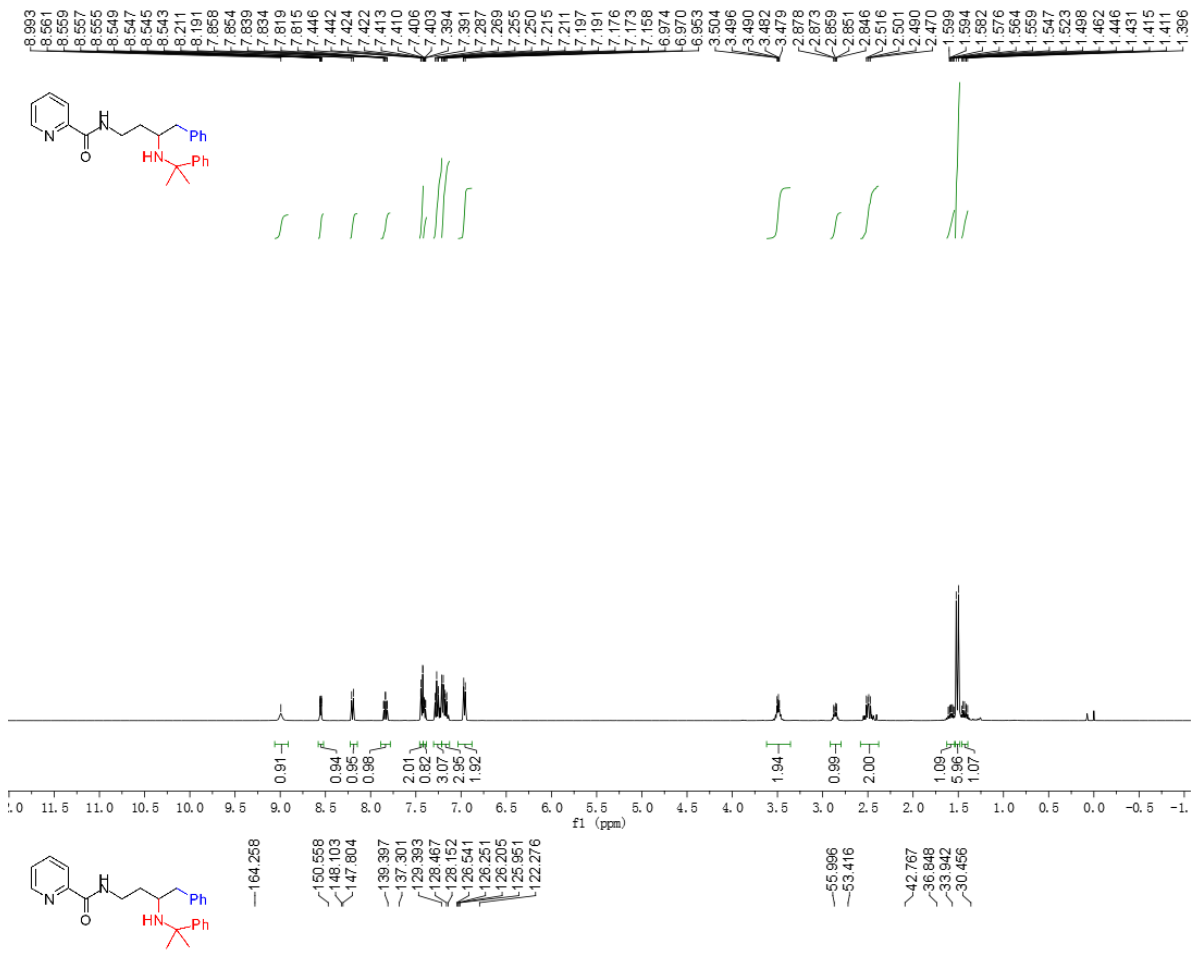
37.464
34.988
29.954
f1 (ppm)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7.839
7.834
7.819
7.815
7.446
7.442
7.424
7.422
7.413
7.410
7.406
7.403
7.394
7.391
7.287
7.269
7.255
7.250
7.215
7.211
7.197
7.191
7.176
7.173
7.158
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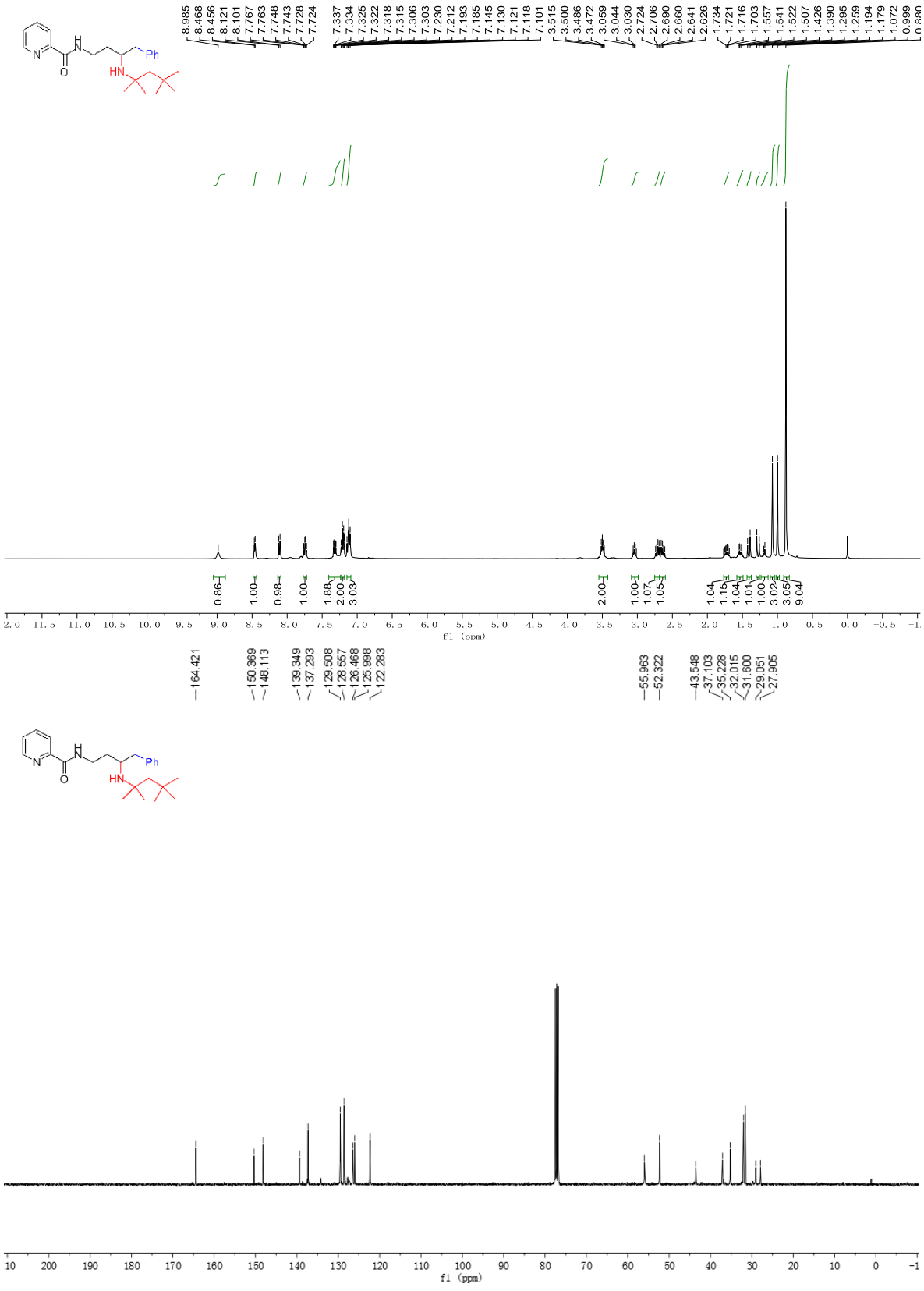
6.970
6.953
3.504
3.496
3.490
3.482
3.479
2.878
2.873
2.859
2.851
2.846
2.516
2.501
2.490
2.470
1.599
1.594
1.582
1.576
1.564
1.559
1.547
1.523
1.498
1.462
1.446
1.431
1.415
1.411
1.396
Ph
HN
Ph
0.91
0.94
0.95
0.98
2.01
0.82
3.07
2.95
1.92
1.94
0.99
2.00
1.09
5.96
1.07
f1 (ppm)
164.258
150.558
148.103
147.804
139.397
137.301
129.393
128.467
128.152
126.541
126.251
126.205
125.951
122.276
55.996
53.416
42.767
36.848
33.942
30.456
f1 (ppm)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7.748
7.743
7.728
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7.337
7.334
7.325
7.322
7.318
7.315
7.306
7.303
7.230
7.212
7.193
7.185
7.145
7.130
7.121
7.118
7.101
3.515
3.500
3.486
3.472
3.059
3.044
3.030
2.724
2.706
2.690
2.660
2.641
2.626
1.734
1.721
1.716
1.703
1.557
1.541
1.522
1.507
1.426
1.390
1.295
1.259
1.194
1.179
1.072
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0.98
1.00
1.88
2.00
3.03
2.00
1.00
1.07
1.05
1.04
1.15
1.04
1.01
1.00
3.02
3.05
9.04
f1 (ppm)
164.421
150.369
148.113
139.349
137.293
129.508
128.557
126.468
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52.322
43.548
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31.600
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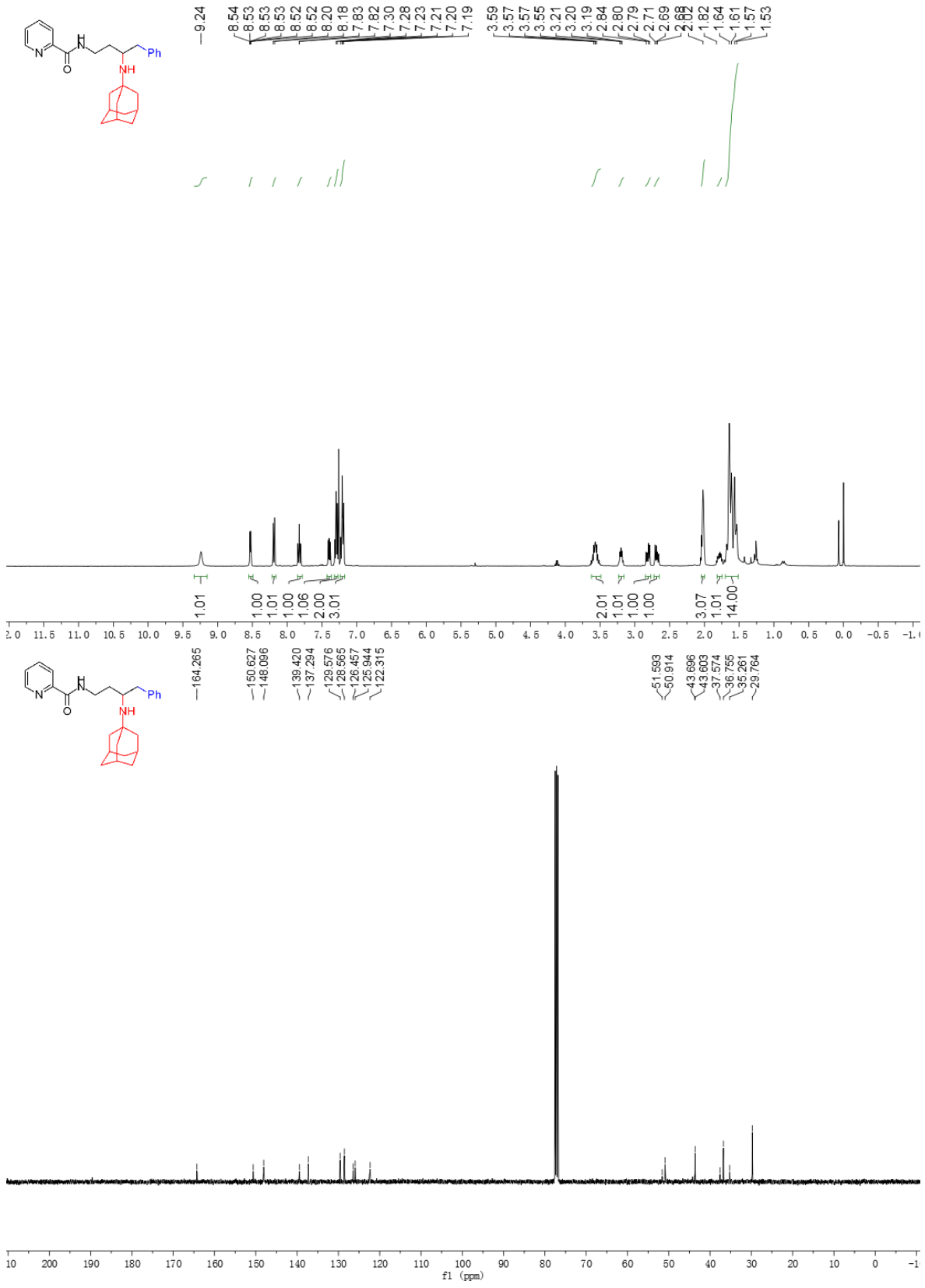
27.905
f1 (ppm)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7.82
7.30
7.28
7.23
7.21
7.20
7.19
3.59
3.57
3.57
3.55
3.21
3.20
3.19
2.84
2.80
2.79
2.71
2.69
1.82
1.64
1.61
1.57
1.53
Ph
NH
1.01
1.00
1.01
1.00
1.06
2.00
3.01
2.01
1.01
1.00
1.00
3.07
1.01
14.00
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150.627
148.096
139.420
137.294
129.576
128.565
126.457
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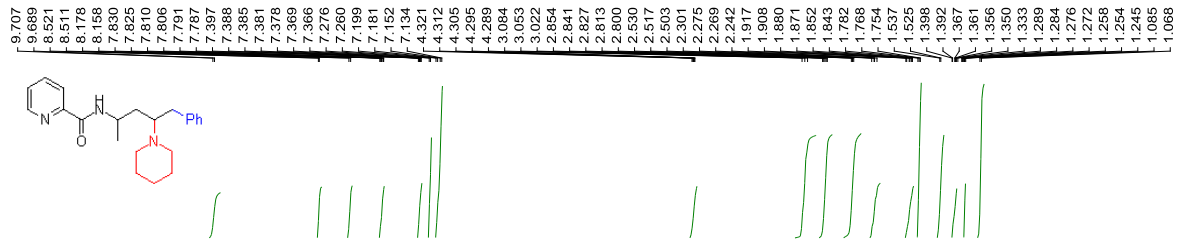
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7.385
7.381
7.378
7.369
7.366
7.276
7.260
7.199
7.181
7.152
7.134
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4.312
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4.295
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4.084
3.053
3.022
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2.841
2.827
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2.517
2.503
2.301
2.275
2.269
2.242
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1.908
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1.852
1.843
1.782
1.768
1.754
1.537
1.525
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1.392
1.367
1.361
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1.254
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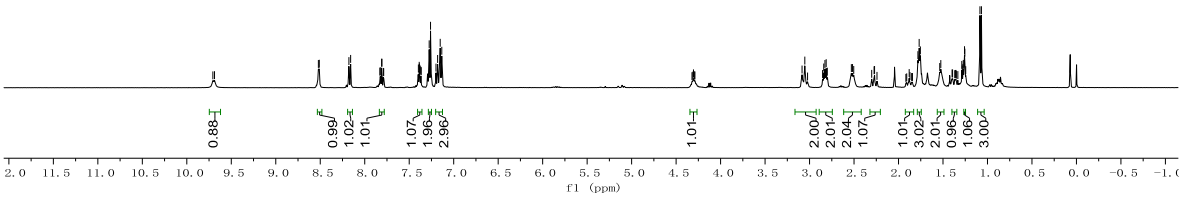
Ph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1.01
2.00
2.01
2.04
1.07
1.01
3.02
2.01
0.96
1.06
3.00
f1 (ppm)


164.689
150.671
148.059
141.805
137.215
128.985
128.412
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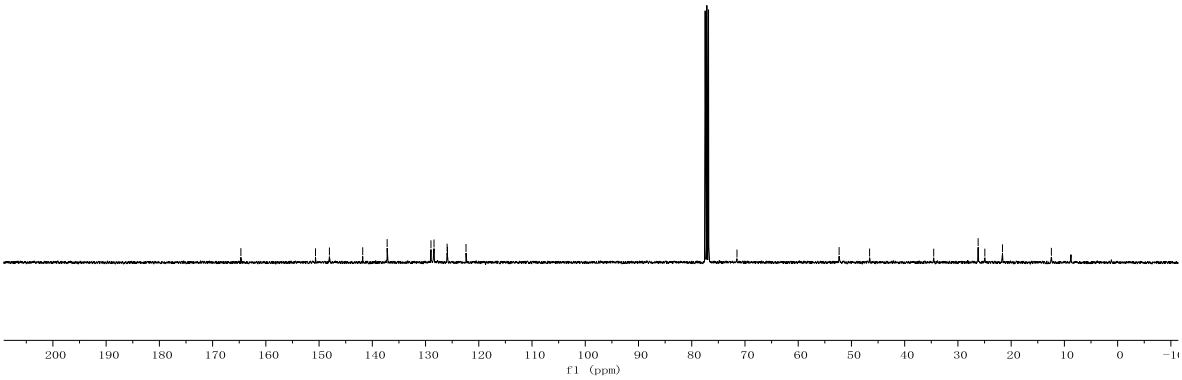
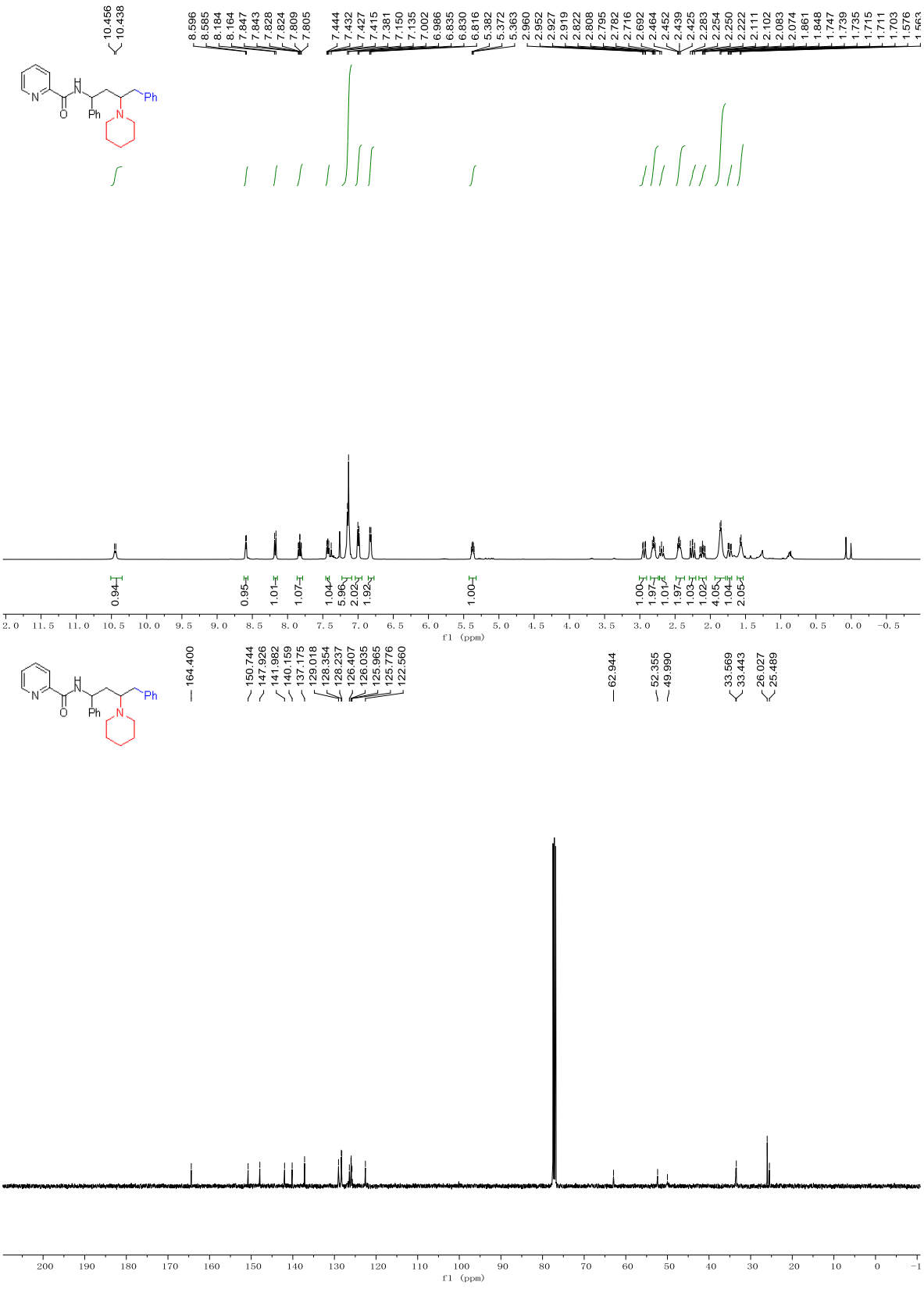
12.445
Ph


f1 (ppm)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2.254
2.250
2.222
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1.848
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1.735
1.715
1.711
1.703
1.576
Ph
Ph
N
O
H
N
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1.07
1.04
5.96
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1.92
1.00
1.00
1.97
1.01
1.97
1.03
1.02
4.05
1.04
2.05
f1 (ppm)
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147.926
141.982
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129.018
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128.237
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126.035
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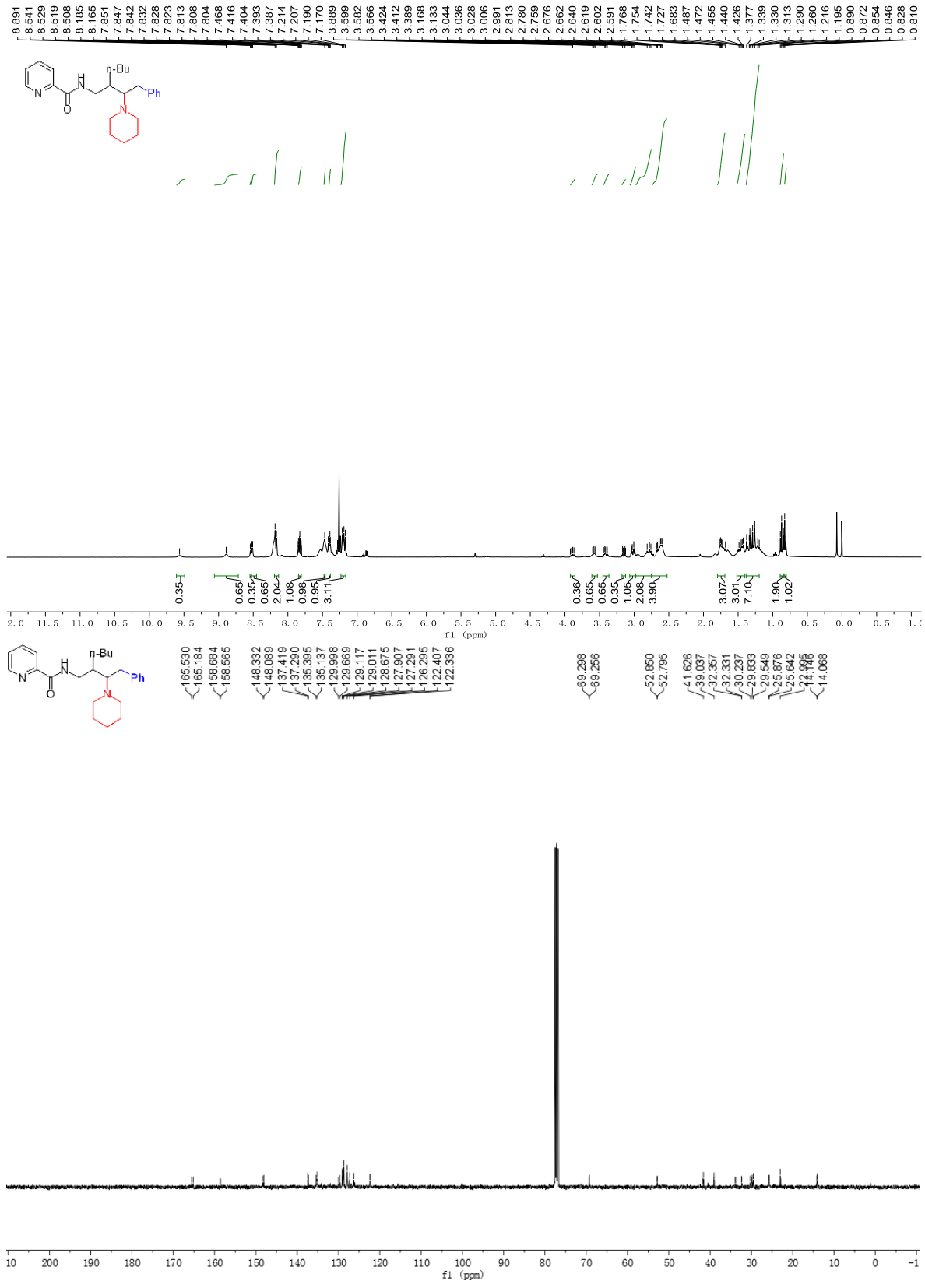
26.027
25.489
f1 (ppm)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0.854
0.846
0.828
0.810
n-Bu
Ph
N
O
H
0.35
0.65
0.35
0.65
2.04
1.08
0.98
0.95
3.11
0.36
0.65
0.65
0.35
1.05
2.08
3.90
3.07
3.01
7.10
1.90
1.02
f1 (ppm)
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165.184
158.684
158.565
148.332
148.089
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137.290
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129.011
128.675
127.907
127.291
126.295
122.407
122.336
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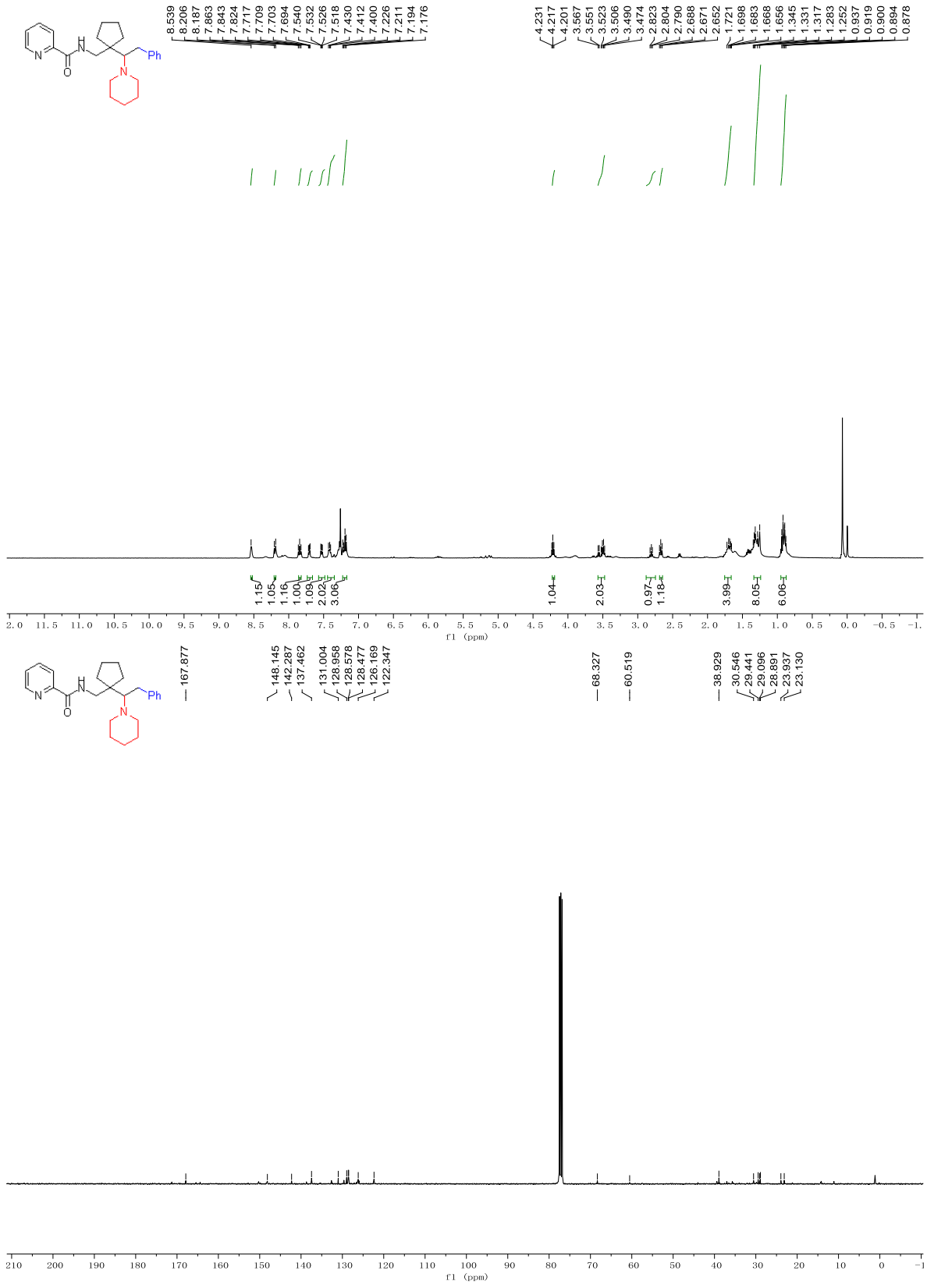
14.068
f1 (ppm)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7.176
4.231
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4.201
3.567
3.551
3.523
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3.490
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1.283
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0.919
0.900
0.894
0.878
Ph
N
O
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1.05
1.16
1.00
1.09
2.02
3.06
1.04
2.03
0.97
1.18
3.99
8.05
6.06
f1 (ppm)
167.877
148.145
142.287
137.462
131.004
128.958
128.578
128.477
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38.929
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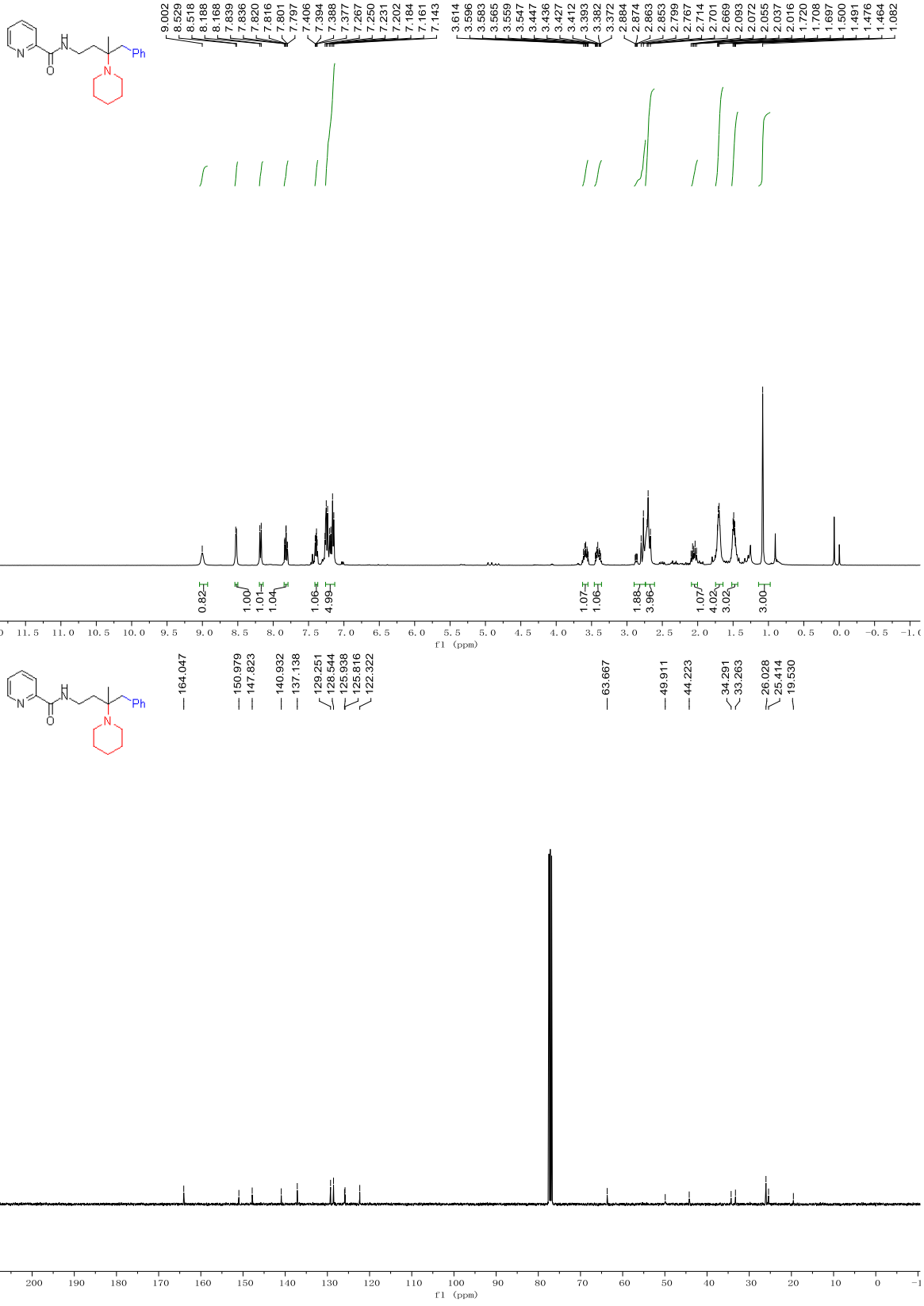
23.130
f1 (ppm)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7.388
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7.267
7.250
7.231
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7.161
7.143
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3.583
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Ph
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1.06
4.99
1.07
1.06
1.88
3.96
1.07
4.02
3.02
3.00
f1 (ppm)
164.047
150.979
147.823
140.932
137.138
129.251
128.544
125.938
125.816
122.322
63.667
49.911
44.223
34.291
33.263
26.028
25.414
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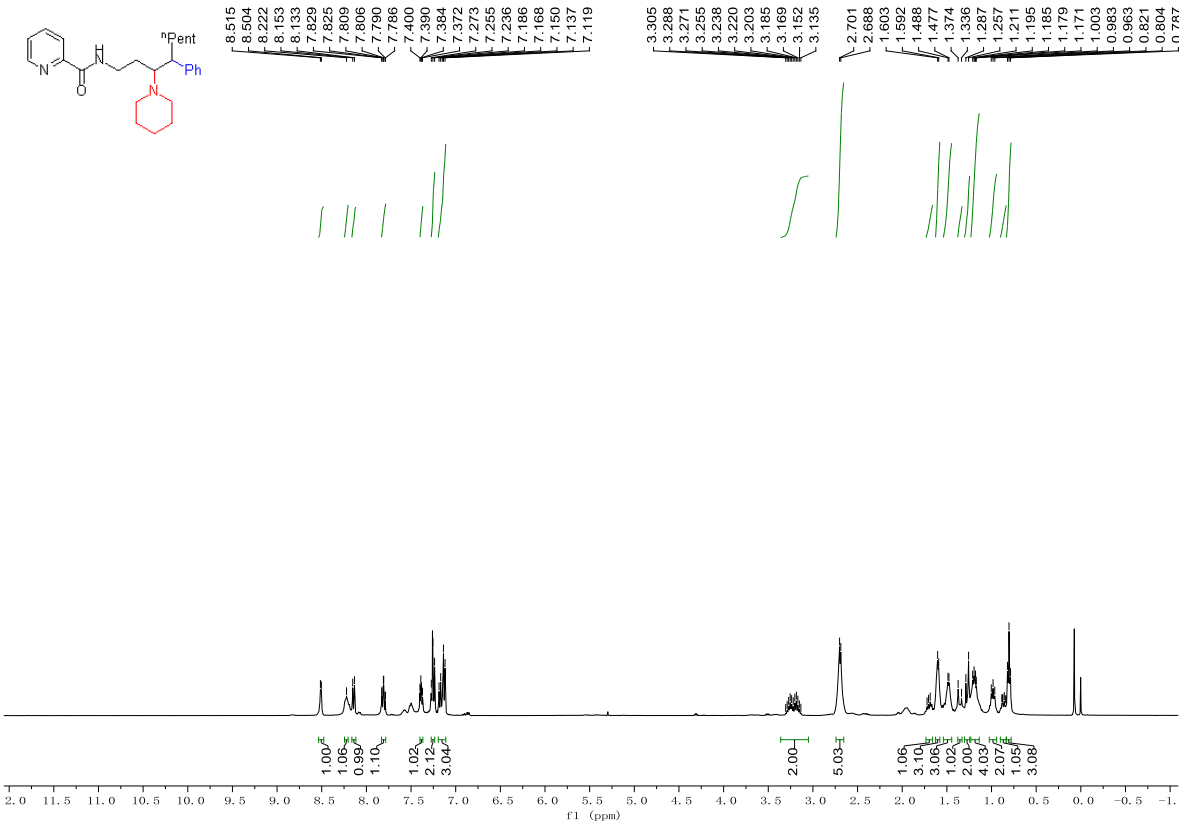
Ph
f1 (ppm)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7.806
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7.786
7.400
7.390
7.384
7.372
7.273
7.255
7.236
7.186
7.168
7.150
7.137
7.119
3.305
3.288
3.271
3.255
3.238
3.220
3.203
3.185
3.169
3.152
3.135
2.701
2.688
1.603
1.592
1.488
1.477
1.374
1.336
1.287
1.257
1.211
1.195
1.185
1.179
1.171
1.003
0.983
0.963
0.821
0.804
1.00
1.06
0.99
1.10
1.02
2.12
3.04
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1.06
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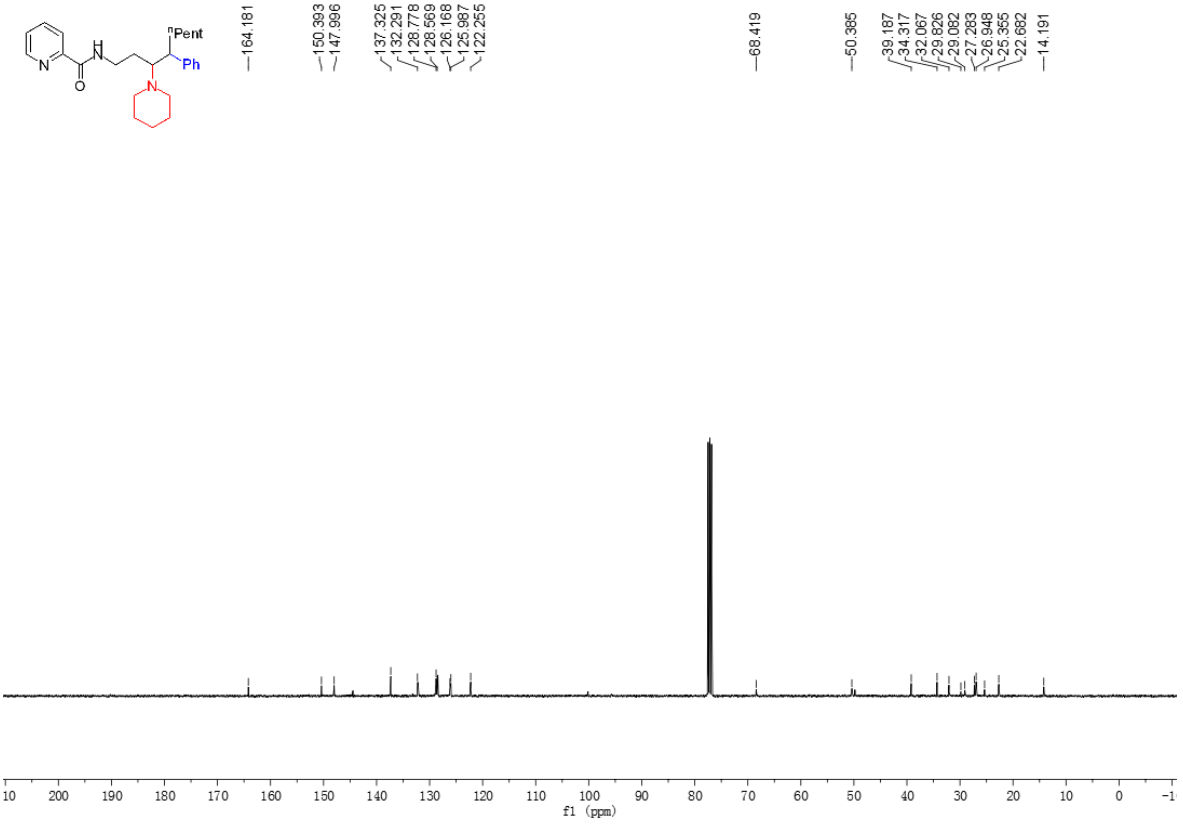
1.02
2.00
4.03
2.07
1.05
3.08
f1 (ppm)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125.987
122.255
68.419
50.385
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29.826
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25.355
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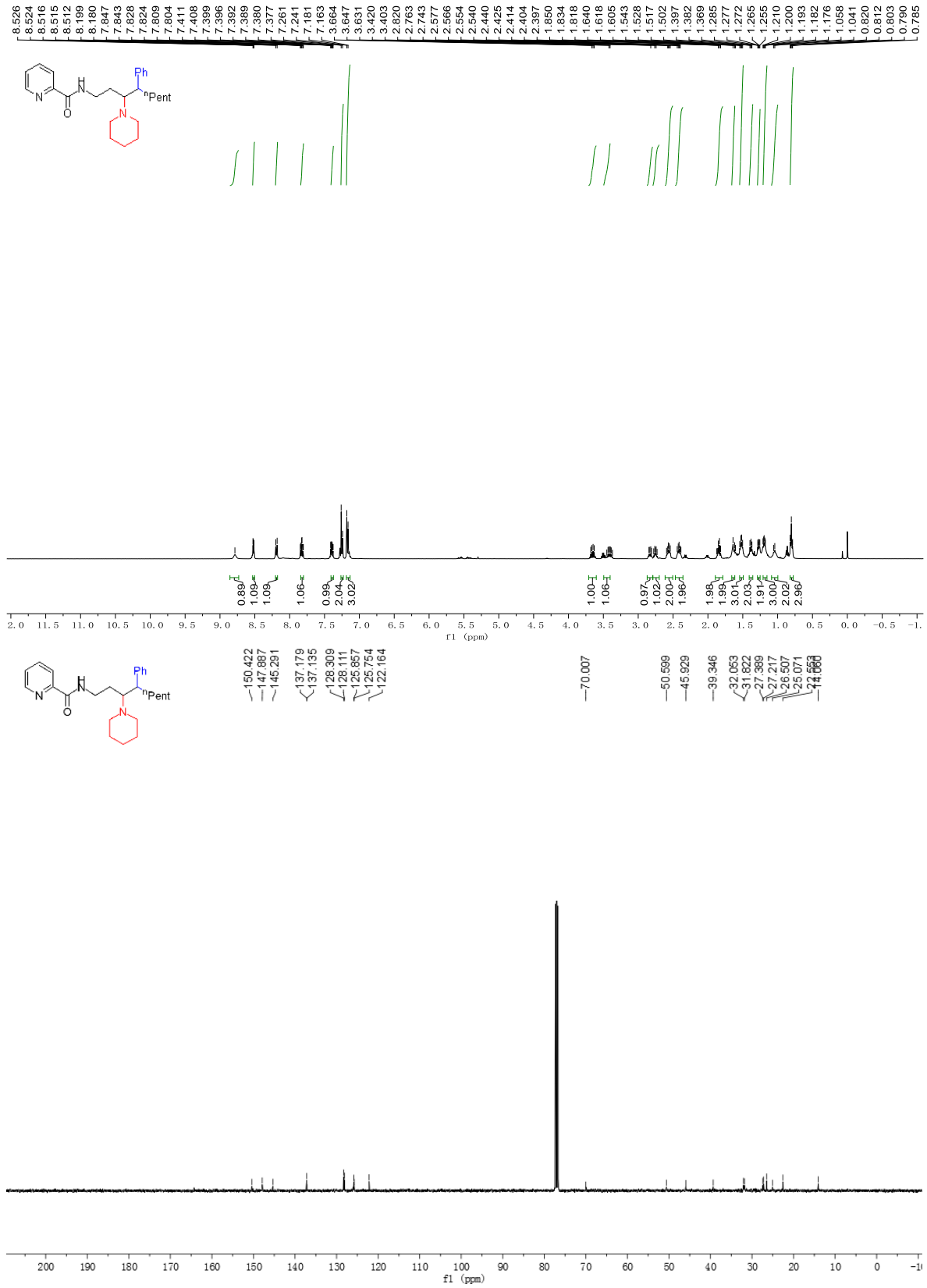
14.191
f1 (ppm)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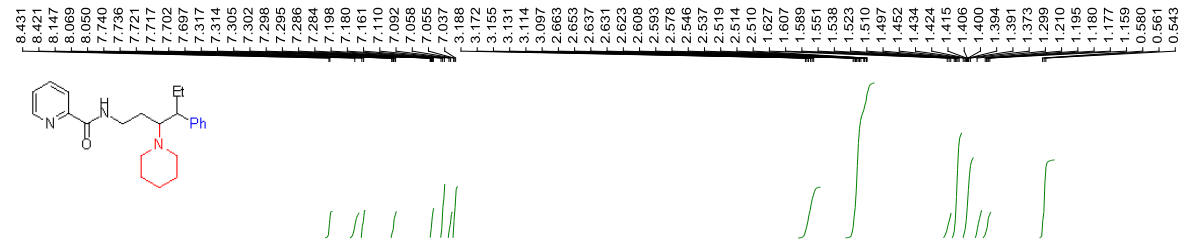
Ph
nPent
0.89 1.09 1.09 1.06 0.99 2.04 3.02 1.00 1.06 0.97 1.02 2.00 1.96 1.98 1.99 3.01 2.03 1.91 3.00 2.02 2.96
f1 (ppm)
150.422 147.887 145.291 137.179 137.135 128.309 128.111 125.857 125.754 122.164 70.007 50.599 45.929 39.346 32.053 31.822 27.369 27.217 26.507 25.071 22.553 14.060
f1 (ppm)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7.305
7.302
7.298
7.295
7.286
7.284
7.198
7.180
7.161
7.110
7.092
7.058
7.055
7.037
3.188
3.172
3.155
3.131
3.114
3.097
2.663
2.653
2.637
2.631
2.623
2.608
2.593
2.578
2.546
2.537
2.519
2.514
2.510
1.627
1.607
1.589
1.551
1.538
1.523
1.510
1.497
1.452
1.434
1.424
1.415
1.406
1.400
1.394
1.391
1.373
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1.180
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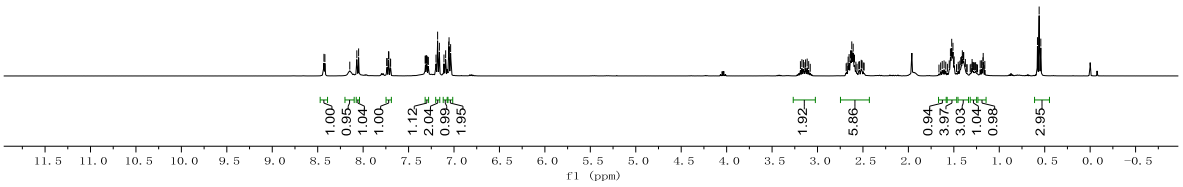
0.580
0.561
0.543
Et
Ph
N
H
O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1.04
1.00
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0.99
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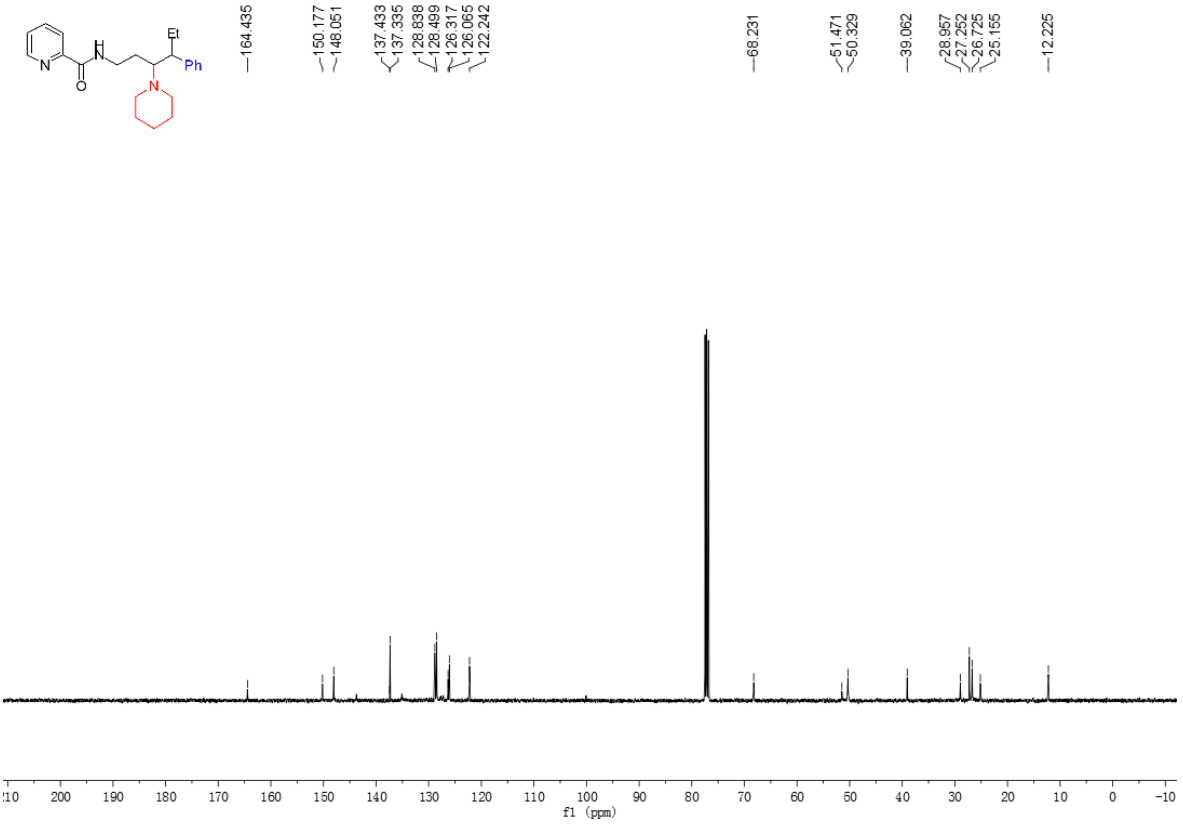
5.86
0.94
3.97
3.03
1.04
0.98
2.95
f1 (ppm)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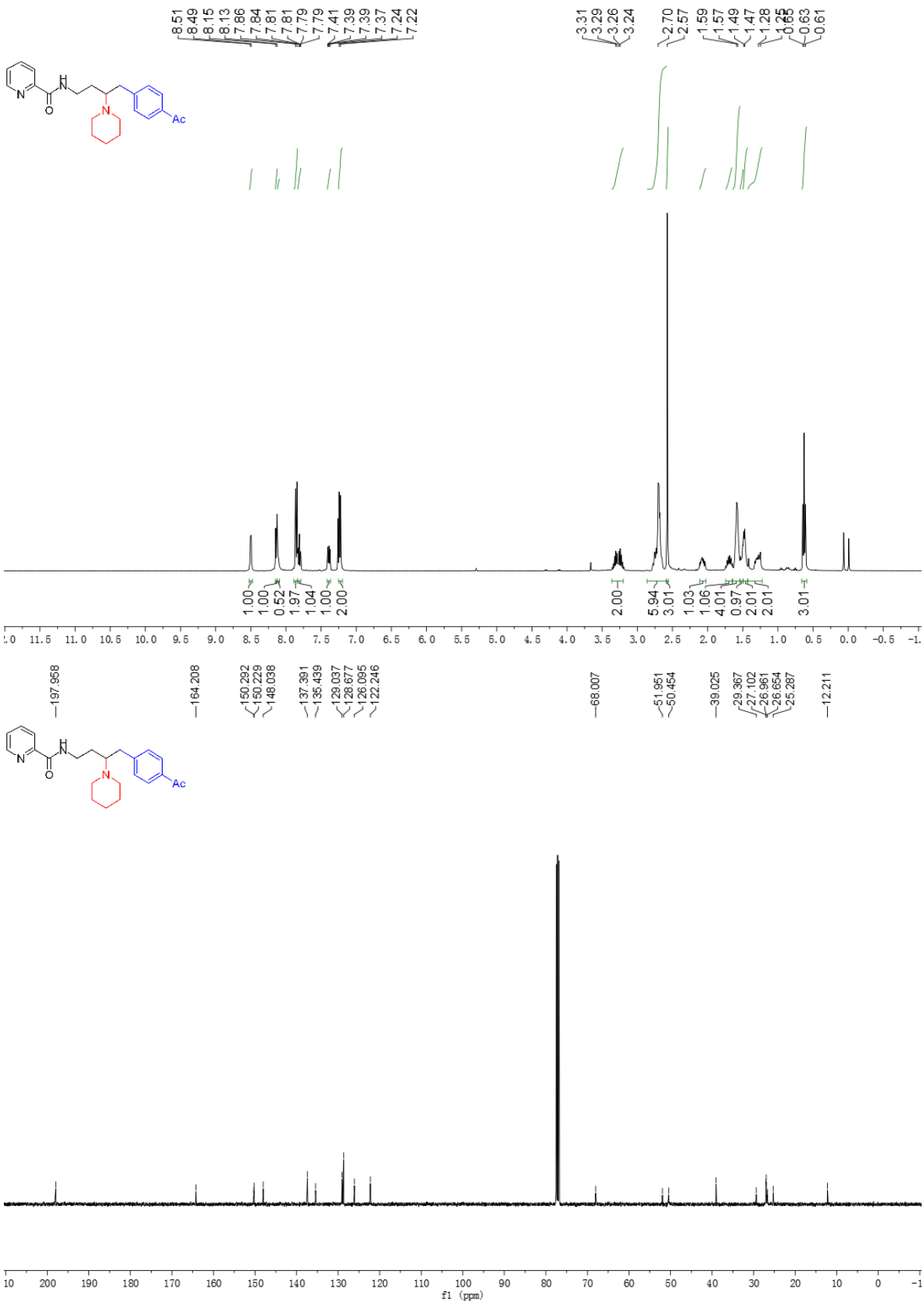
148.051
137.433
137.335
128.838
128.499
126.317
126.065
122.242
68.231
51.471
50.329
39.062
28.957
27.252
26.725
25.155
12.225
Et
Ph
N
H
O
f1 (ppm)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7.84
7.81
7.81
7.79
7.79
7.41
7.39
7.39
7.37
7.24
7.22
3.31
3.29
3.26
3.24
2.70
2.57
1.59
1.57
1.49
1.47
1.28
0.65
0.63
0.61
Ac
1.00
1.00
0.52
1.97
1.04
1.00
2.00
2.00
5.94
3.01
1.03
1.06
4.01
0.97
2.01
2.01
3.01
197.958
164.208
150.292
150.229
148.038
137.391
135.439
129.037
128.677
126.095
122.246
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39.025
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27.102
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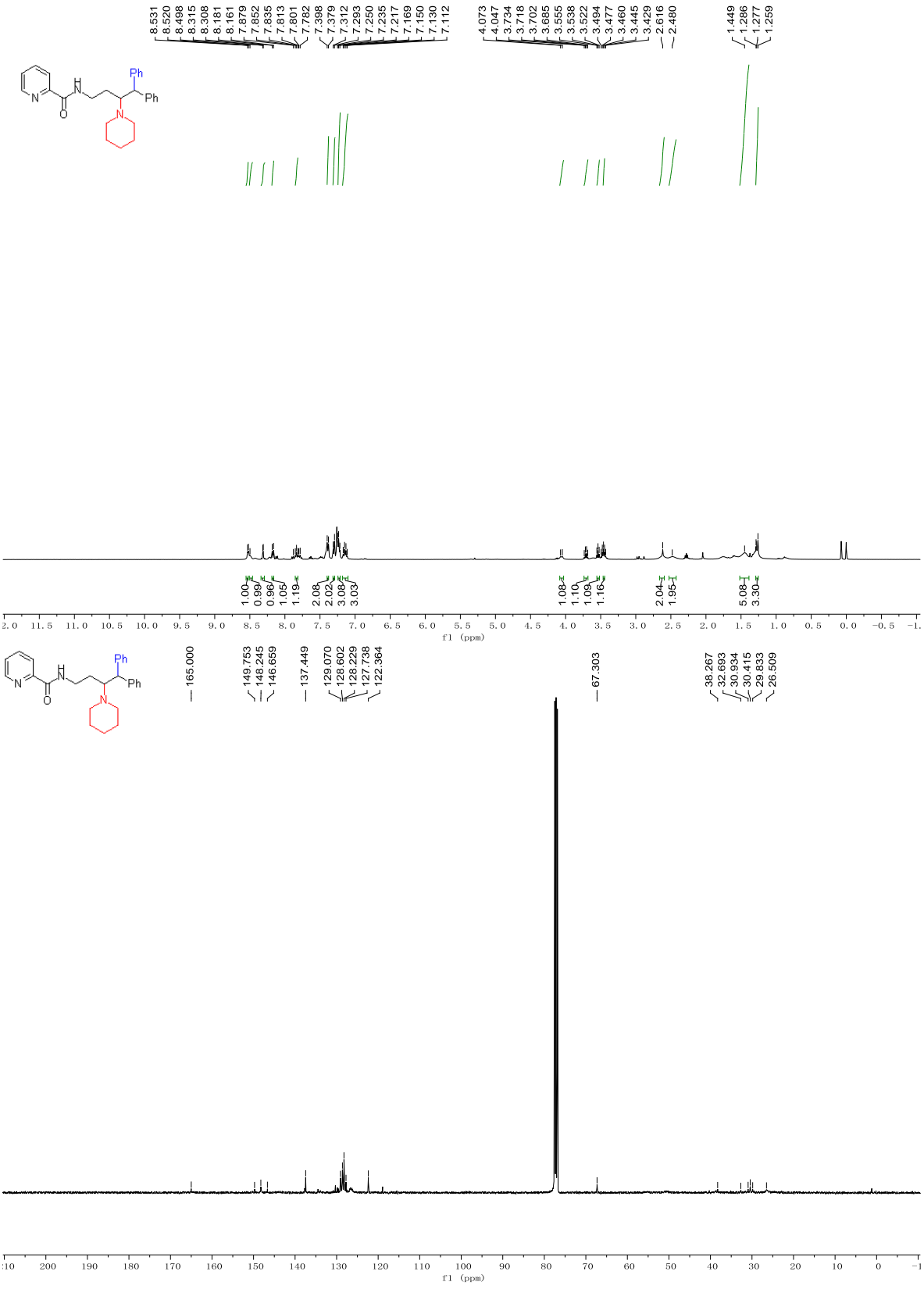
26.654
25.287
12.211
Ac
f1 (ppm)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8.161
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7.835
7.813
7.801
7.782
7.398
7.379
7.312
7.293
7.250
7.235
7.217
7.169
7.150
7.130
7.112
4.073
4.047
3.734
3.718
3.702
3.685
3.555
3.538
3.522
3.494
3.477
3.460
3.445
3.429
2.616
2.480
1.449
1.286
1.277
1.259
Ph
Ph
N
H
O
1.00
0.99
0.96
1.05
1.19
2.08
2.02
3.08
3.03
1.08
1.10
1.09
1.16
2.04
1.95
5.08
3.30
f1 (ppm)
165.000
149.753
148.245
146.659
137.449
129.070
128.602
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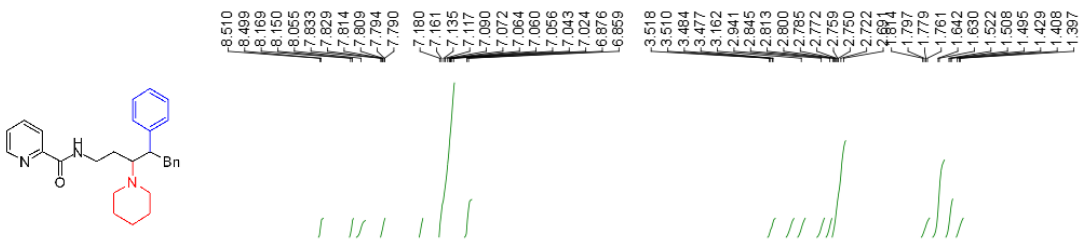
67.303
38.267
32.693
30.934
30.415
29.833
26.509
f1 (ppm)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7.814
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7.794
7.790
7.180
7.161
7.135
7.117
7.090
7.072
7.064
7.060
7.056
7.043
7.024
6.876
6.859
3.518
3.510
3.484
3.477
3.162
2.941
2.845
2.813
2.800
2.785
2.772
2.759
2.750
2.722
1.797
1.779
1.761
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1.630
1.522
1.508
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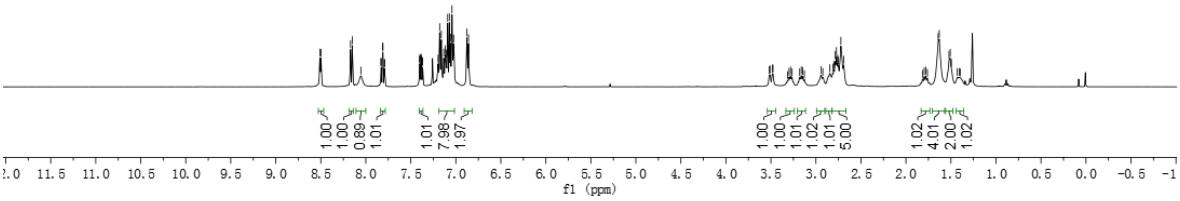
1.408
1.397
N
H
O
Bn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1.02
1.01
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1.02
4.01
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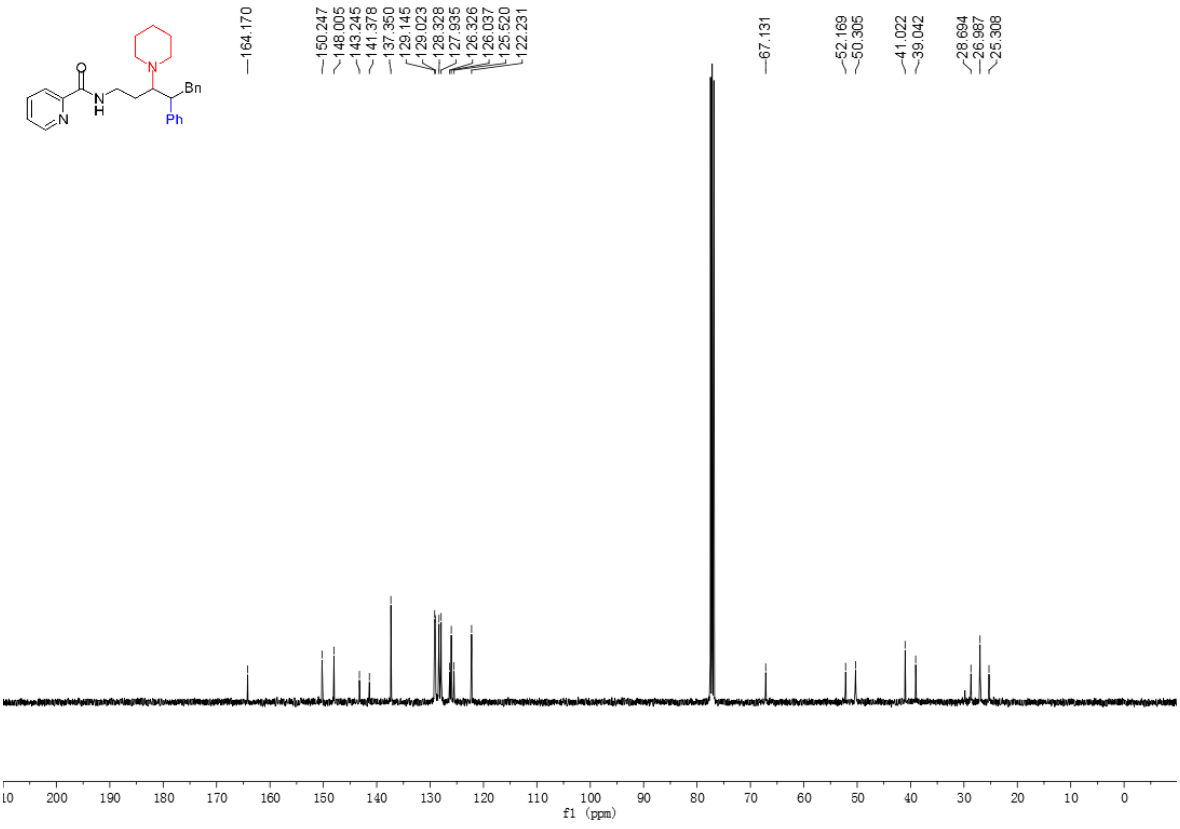
1.02
f1 (ppm)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129.023
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127.935
126.326
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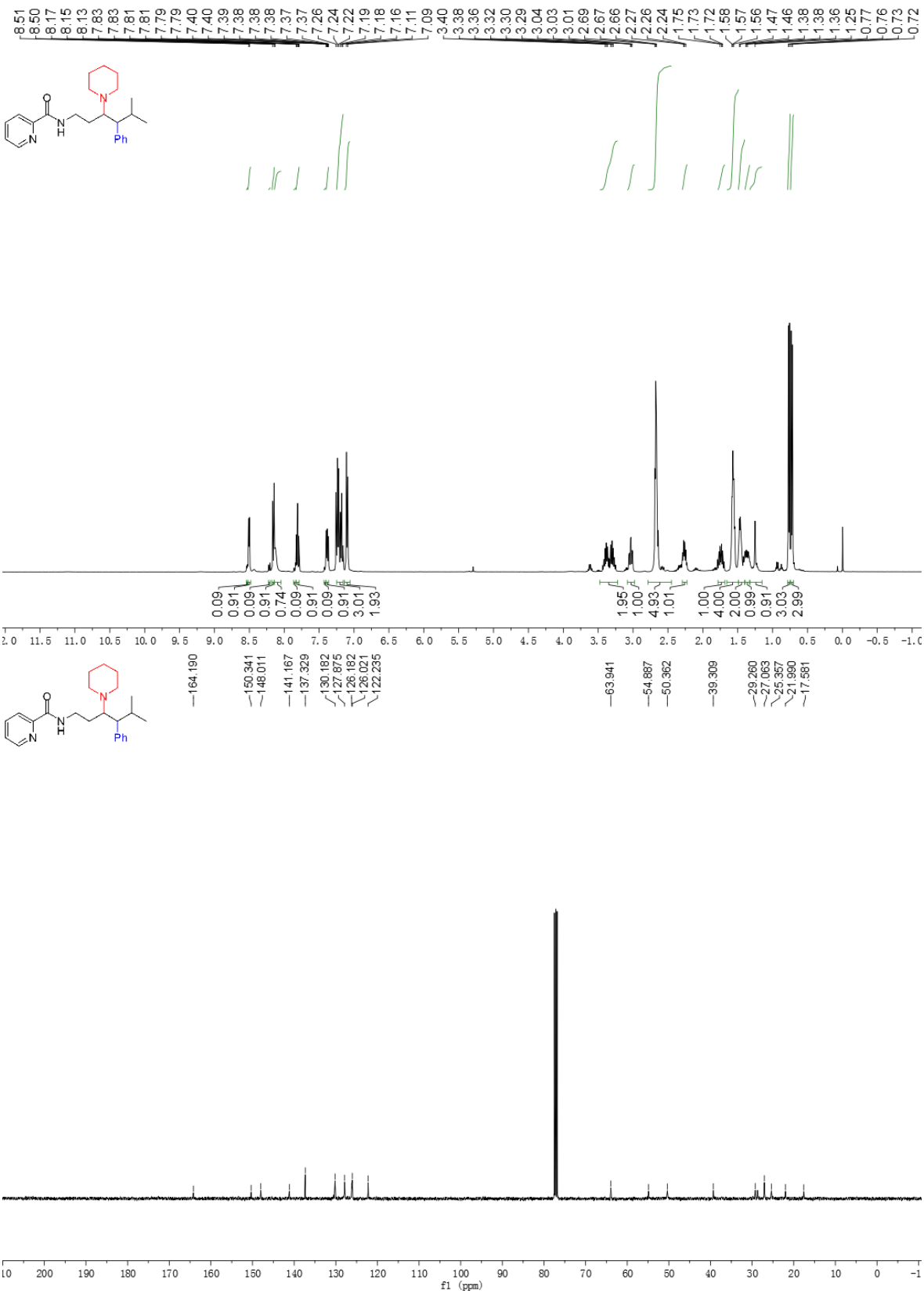
125.520
122.231
67.131
52.169
50.305
41.022
39.042
28.694
26.987
25.308
N
O
N
H
Bn
Ph
f1 (ppm)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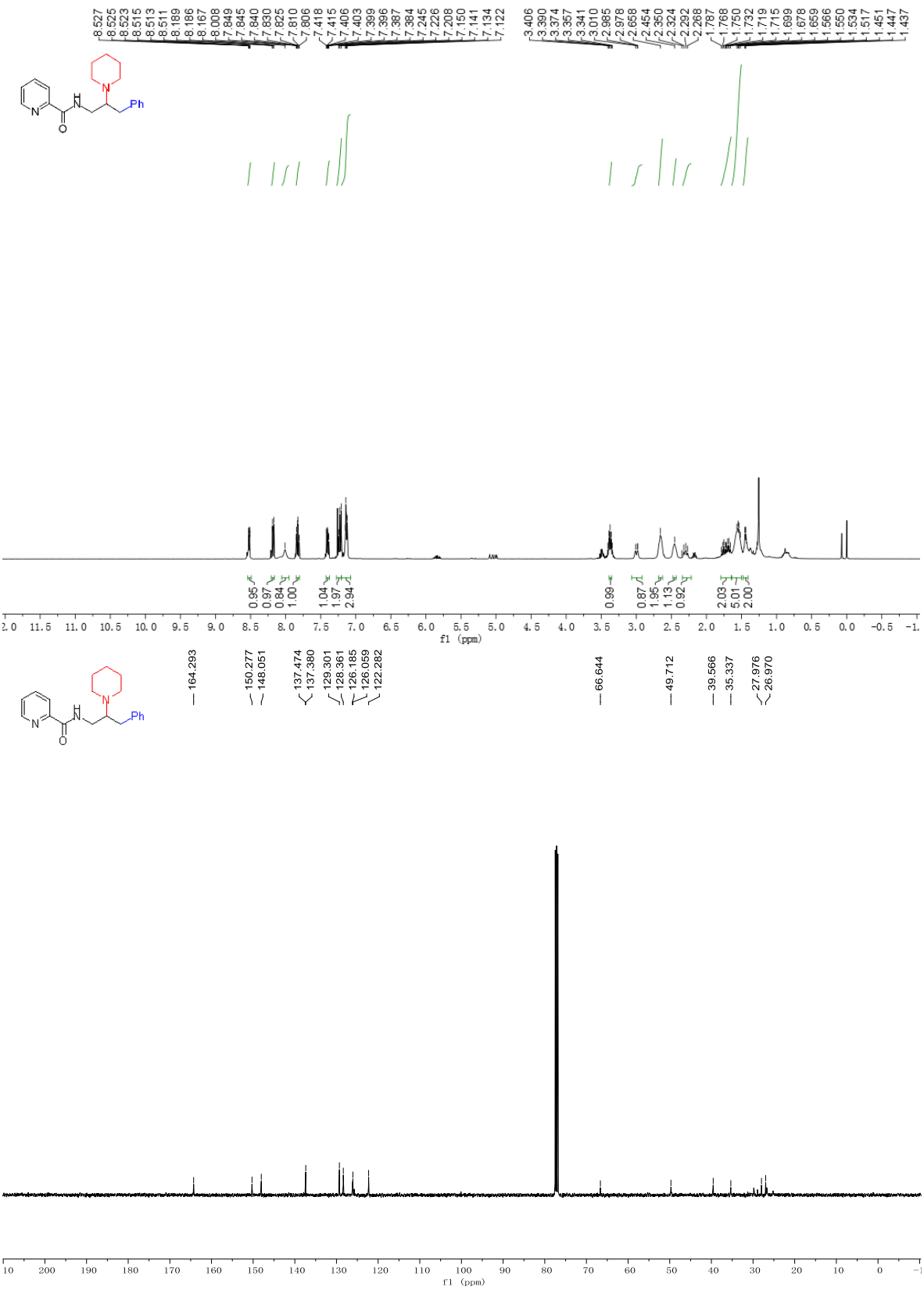
Ph
0.09 0.91 0.09 0.91 0.74 0.09 0.91 0.09 0.91 3.01 1.93
1.95 1.00 4.93 1.01 1.00 4.00 2.00 0.99 0.91 3.03 2.99
164.190 150.341 148.011 141.167 137.329 130.182 127.875 126.182 126.021 122.235
63.941 54.887 50.362 39.309 29.260 27.063 25.357 21.990 17.581
f1 (ppm)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3.406 3.390 3.374 3.357 3.341 3.010 2.985 2.978 2.658 2.454 2.350 2.324 2.292 2.268 1.787 1.768 1.750 1.732 1.719 1.715 1.699 1.678 1.659 1.566 1.550 1.534 1.517 1.451 1.447 1.437
Ph
0.95 0.97 0.84 1.00 1.04 1.97 2.94 0.99 0.87 1.95 1.13 0.92 2.03 5.01 2.00
f1 (ppm)
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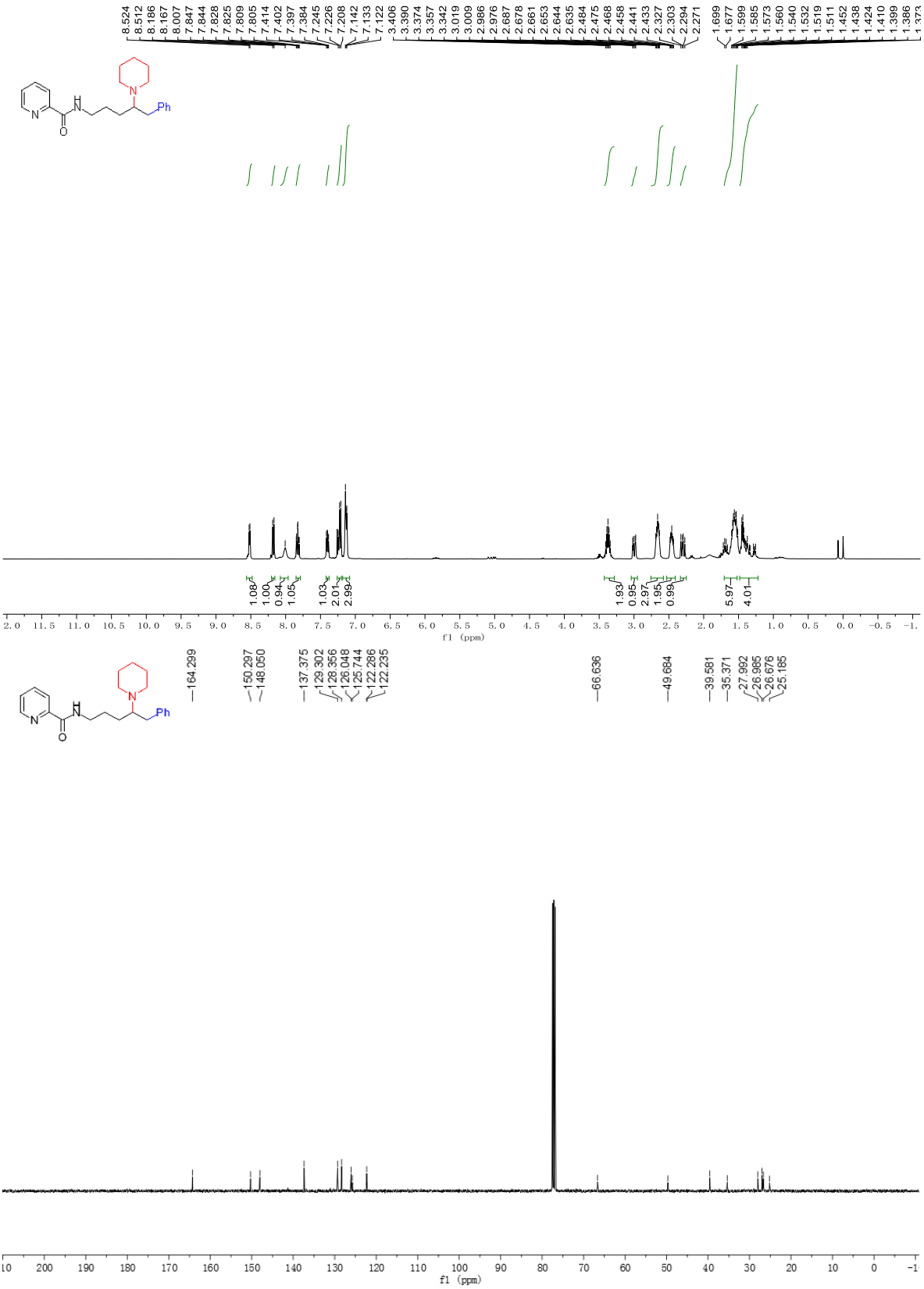
f1 (ppm)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7.805
7.414
7.402
7.397
7.384
7.245
7.226
7.208
7.142
7.133
7.122
3.406
3.390
3.374
3.357
3.342
3.019
3.009
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2.976
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2.678
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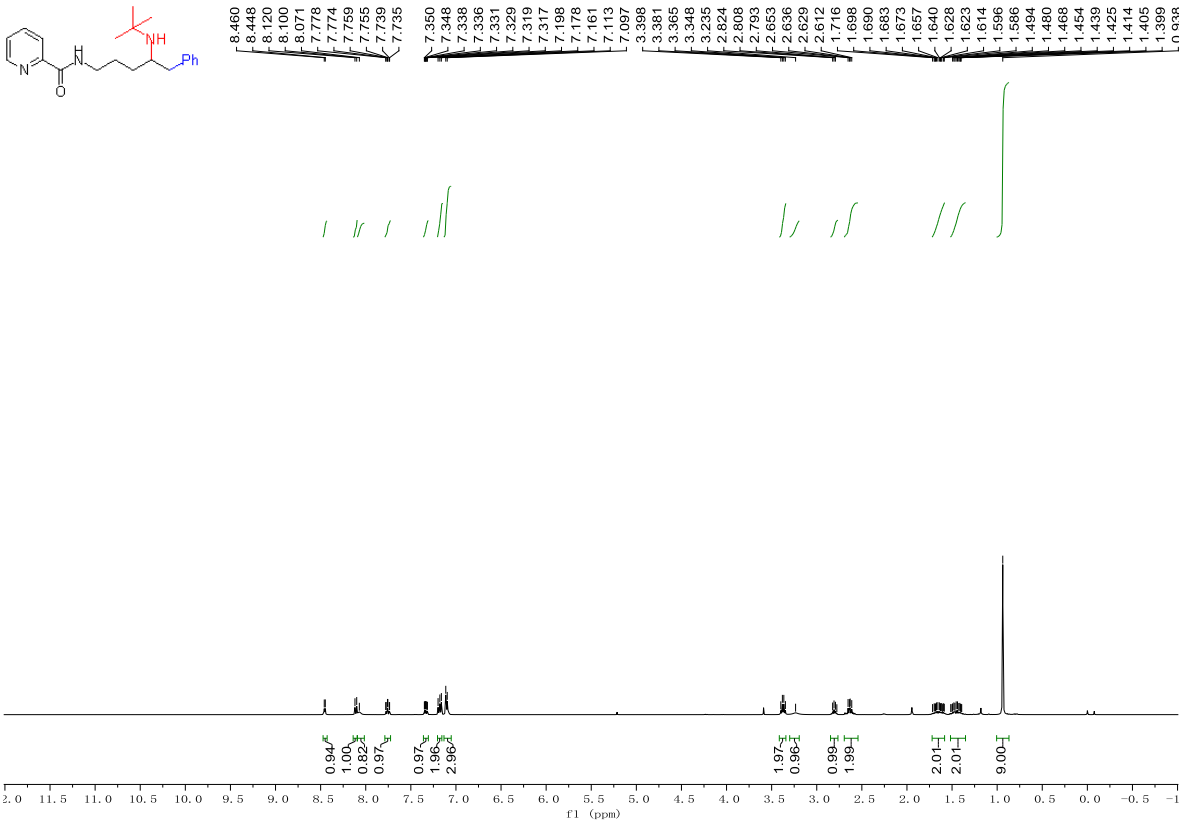
2.635
2.484
2.475
2.468
2.458
2.441
2.433
2.327
2.303
2.294
2.271
1.699
1.677
1.599
1.585
1.573
1.560
1.540
1.532
1.519
1.511
1.452
1.438
1.424
1.410
1.399
1.386
Ph
1.08
1.00
0.94
1.05
1.03
2.01
2.99
1.93
0.95
2.97
1.95
0.99
5.97
4.01
f1 (ppm)
164.299
150.297
148.050
137.375
129.302
128.356
126.048
125.744
122.286
122.235
66.636
49.684
39.581
35.371
27.992
26.985
26.676
25.185
f1 (ppm)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7.755
7.739
7.735
7.350
7.348
7.338
7.336
7.331
7.329
7.319
7.317
7.198
7.178
7.161
7.113
7.097
3.398
3.381
3.365
3.348
3.235
2.824
2.808
2.793
2.653
2.636
2.629
2.612
1.716
1.698
1.690
1.683
1.673
1.657
1.640
1.628
1.623
1.614
1.596
1.586
1.494
1.480
1.468
1.454
1.439
1.425
1.414
1.405
1.399
NH
Ph
N
H
O

0.94
1.00
0.82
0.97
0.97
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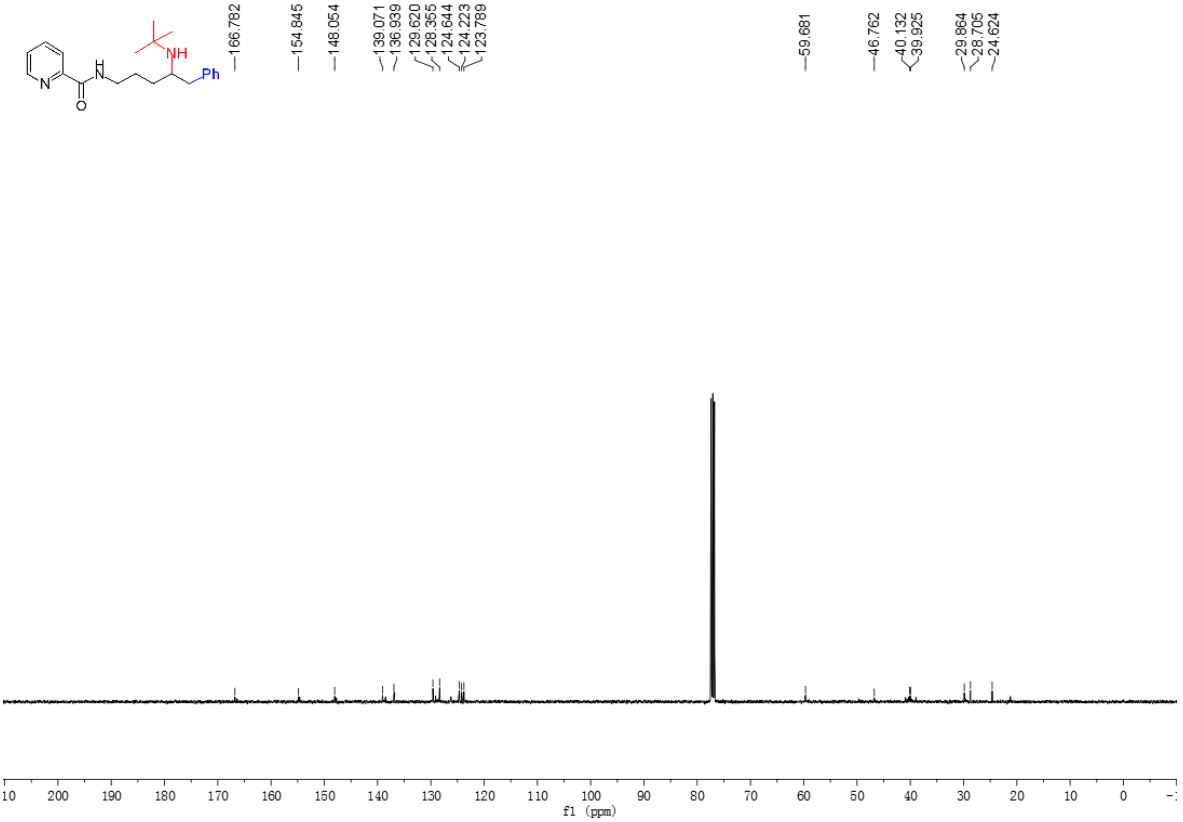
2.96
1.97
0.96
0.99
1.99
2.01
2.01
9.00
f1 (ppm)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40.132
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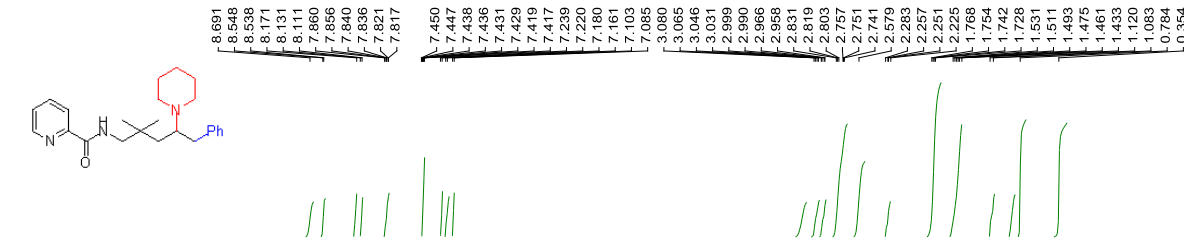
29.864
28.705
24.624
NH
Ph
N
H
O
f1 (ppm)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7.447
7.438
7.436
7.431
7.429
7.419
7.417
7.239
7.220
7.180
7.161
7.103
7.085
3.080
3.065
3.046
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2.831
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2.283
2.257
2.251
2.225
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1.742
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1.083
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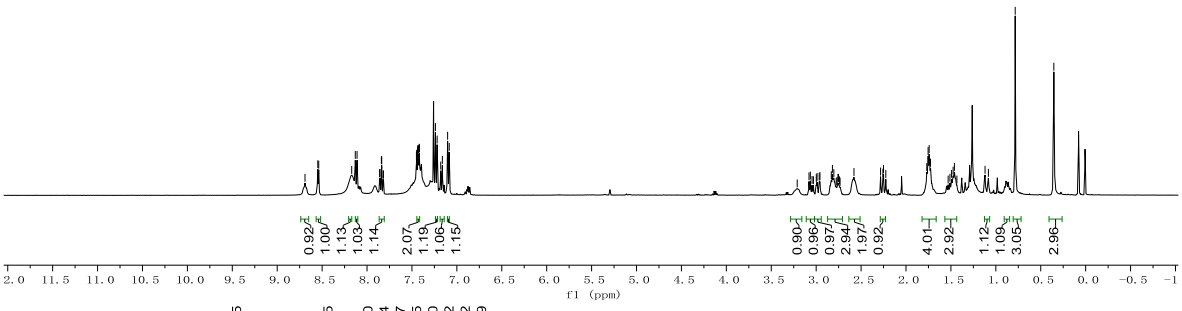
H
O
Ph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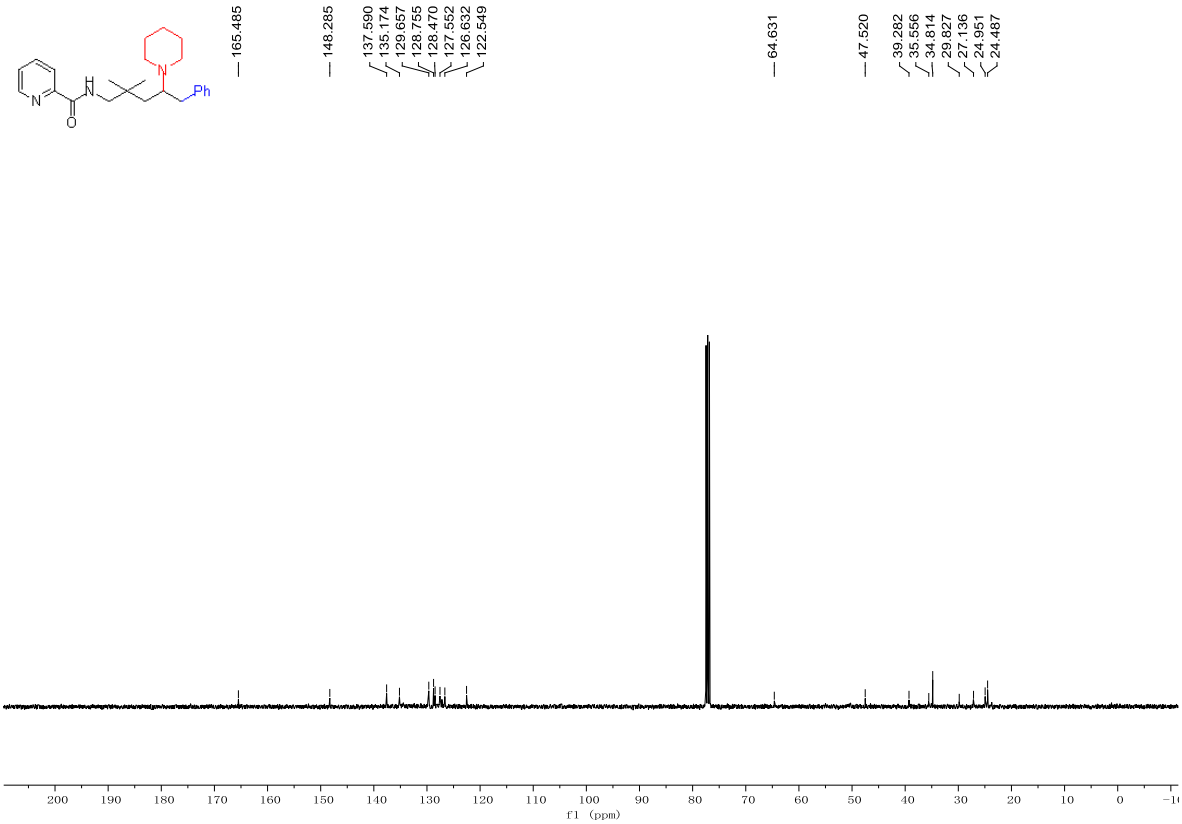
1.14
2.07
1.19
1.06
1.15
0.90
0.96
0.97
2.94
1.97
0.92
4.01
2.92
1.12
1.09
3.05
2.96
f1 (ppm)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34.814
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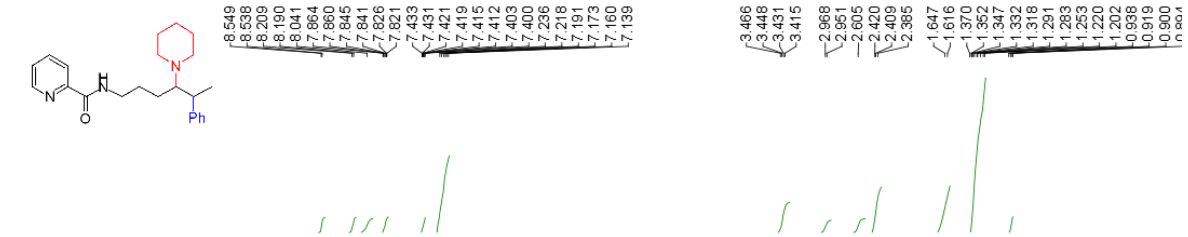
24.487
N
H
O
Ph
f1 (ppm)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7.821
7.433
7.431
7.421
7.419
7.415
7.412
7.403
7.400
7.236
7.218
7.191
7.173
7.160
7.139
3.466
3.448
3.431
3.415
2.968
2.951
2.605
2.420
2.409
2.385
1.647
1.616
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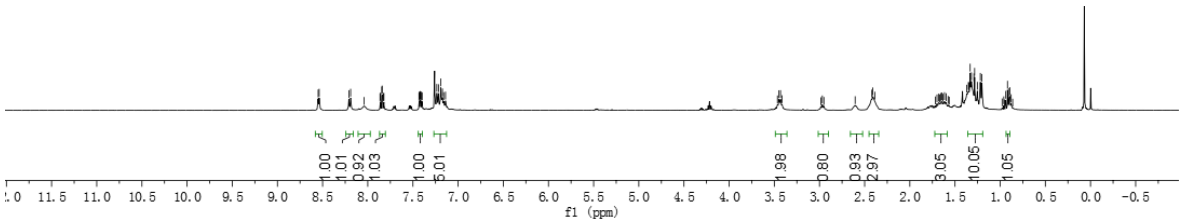
Ph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1.01
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1.00
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0.80
0.93
2.97
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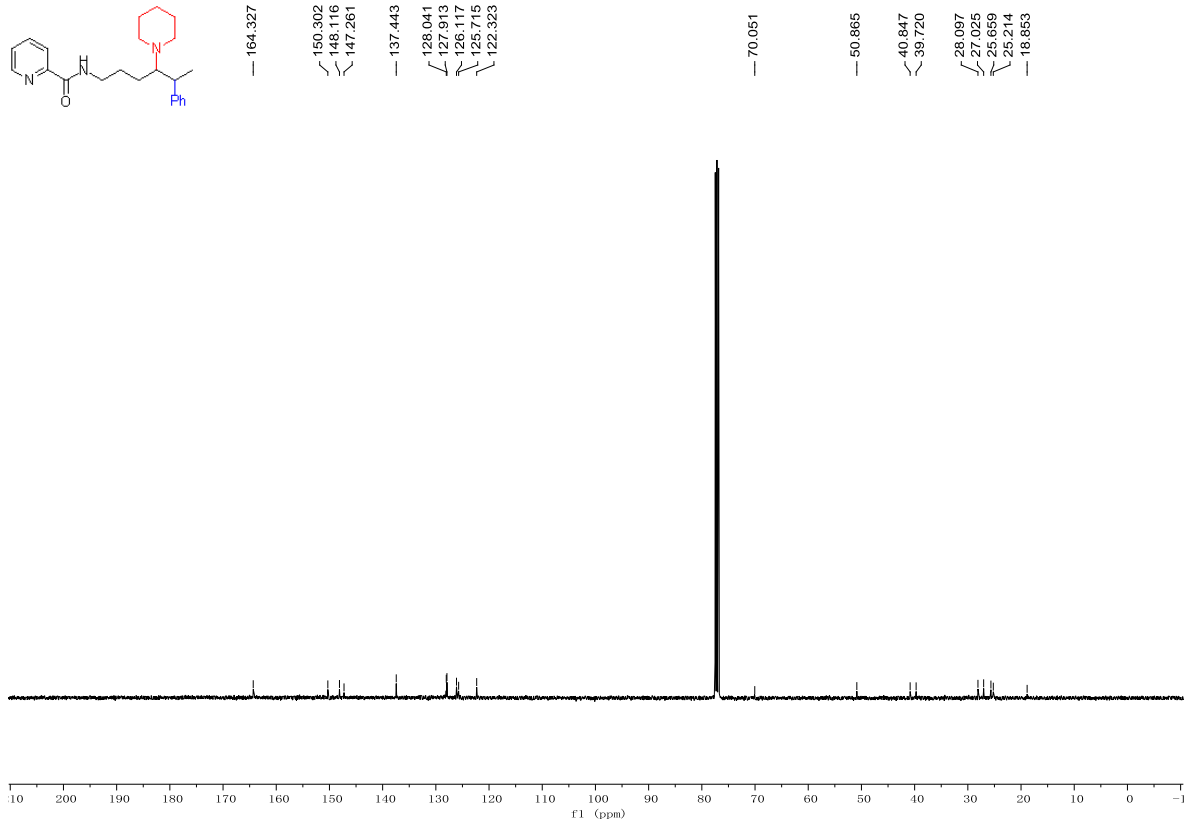
1.05
f1 (ppm)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126.117
125.715
122.323
70.051
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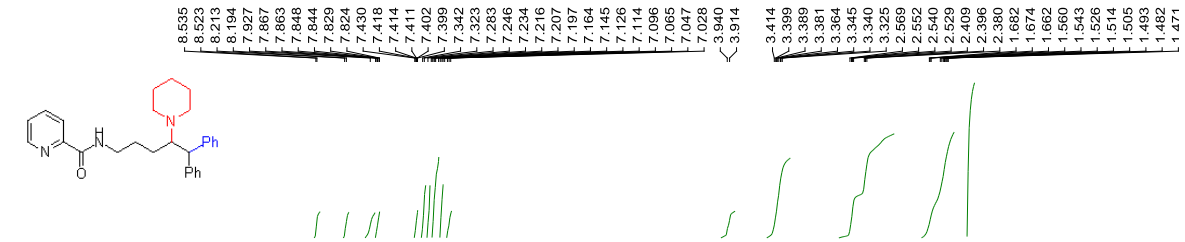
40.847
39.720
28.097
27.025
25.659
25.214
18.853
Ph
f1 (ppm)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8.213
8.194
7.927
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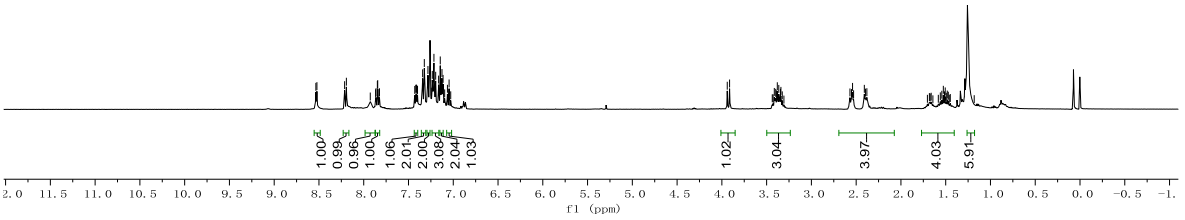
7.848
7.844
7.829
7.824
7.430
7.418
7.414
7.411
7.402
7.399
7.342
7.323
7.283
7.246
7.234
7.216
7.207
7.197
7.164
7.145
7.126
7.114
7.096
7.065
7.047
7.028
3.940
3.914
3.414
3.399
3.389
3.381
3.364
3.345
3.340
3.325
2.569
2.552
2.540
2.529
2.409
2.396
2.380
1.682
1.674
1.662
1.560
1.543
1.526
1.514
1.505
1.493
1.482
1.471
N
H
N
O
Ph
Ph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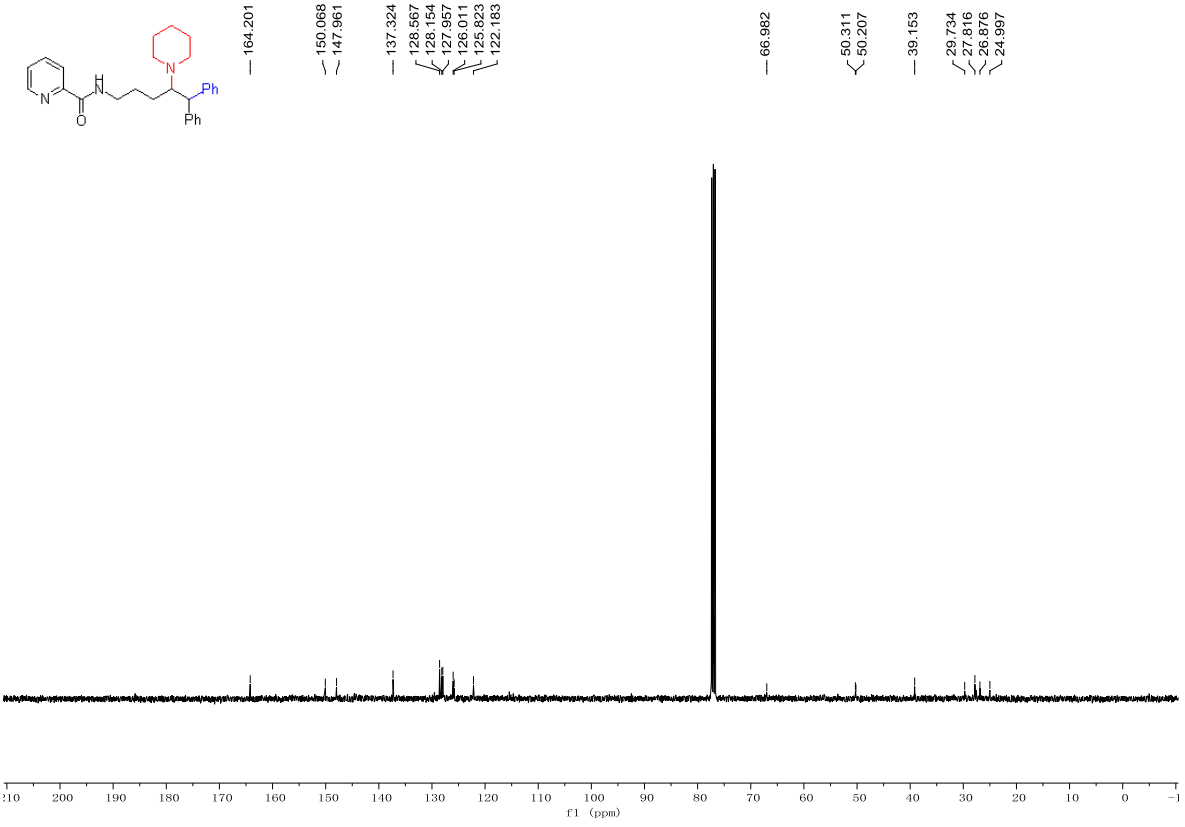
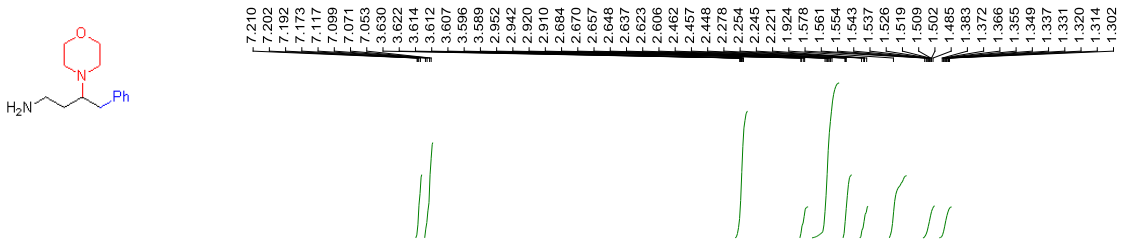
0.99
0.96
1.00
1.06
2.01
2.00
3.08
2.04
1.03
1.02
3.04
3.97
4.03
5.91
f1 (ppm)


164.201
150.068
147.961
137.324
128.567
128.154
127.957
126.011
125.823
122.183
66.982
50.311
50.207
39.153
29.734
27.816
26.876
24.997
N
H
N
O
Ph
Ph
f1 (ppm)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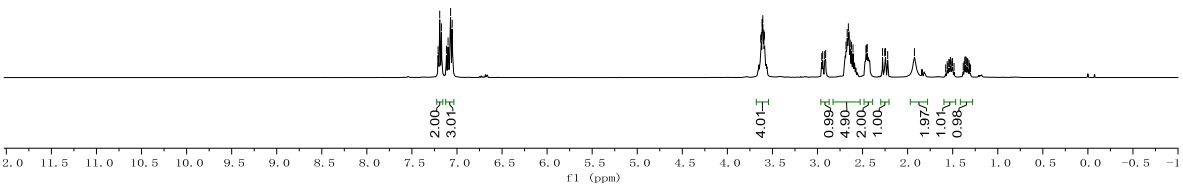
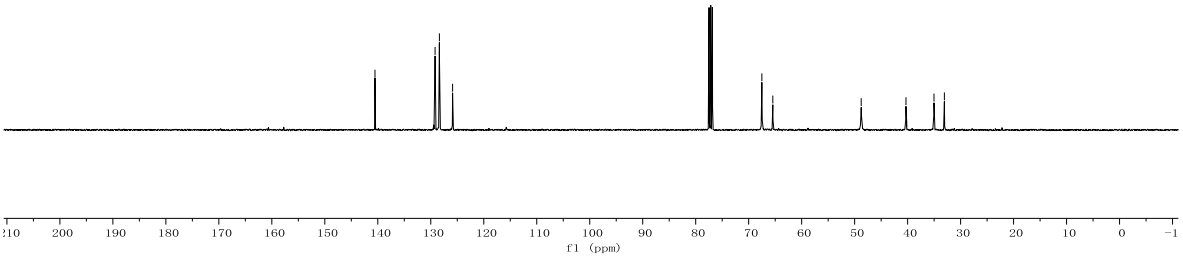
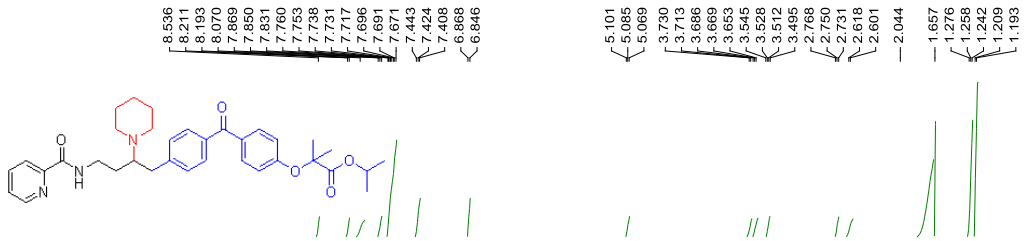
7.202
7.192
7.173
7.117
7.099
7.071
7.053
3.630
3.622
3.614
3.612
3.607
3.596
3.589
2.952
2.942
2.920
2.910
2.684
2.670
2.657
2.648
2.637
2.623
2.606
2.462
2.457
2.448
2.278
2.254
2.245
2.221
1.924
1.578
1.561
1.554
1.543
1.537
1.526
1.519
1.509
1.502
1.485
1.383
1.372
1.366
1.355
1.349
1.337
1.331
1.320
1.314
1.302


2.00
3.01
4.01
0.99
4.90
2.00
1.00
1.97
1.01
0.98
f1 (ppm)


140.532
129.188
128.376
125.881
67.513
65.445
48.762
40.313
40.182
35.023
33.064


f1 (ppm)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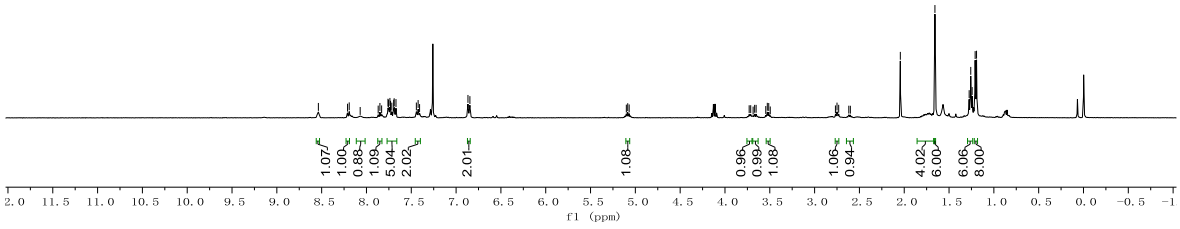
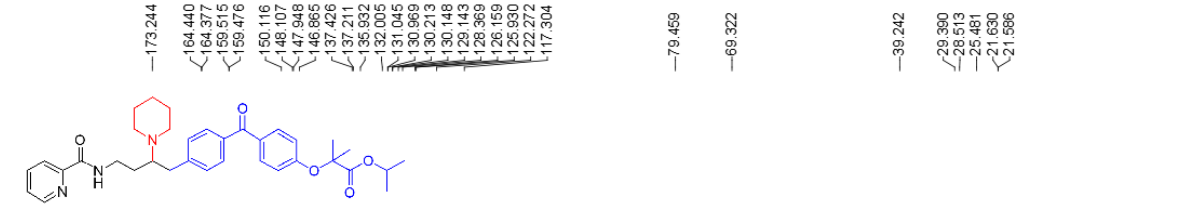
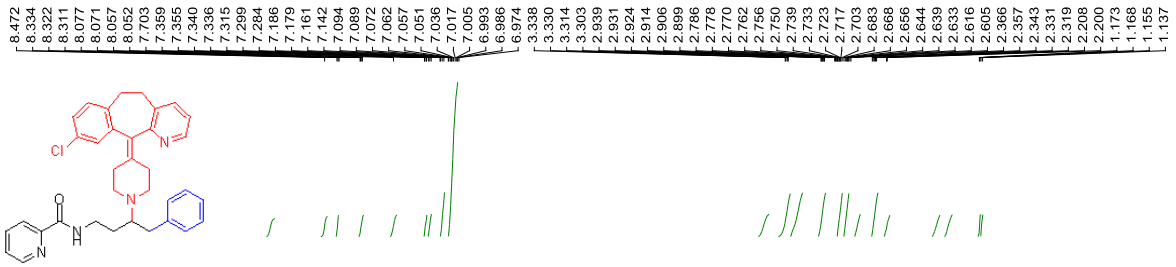
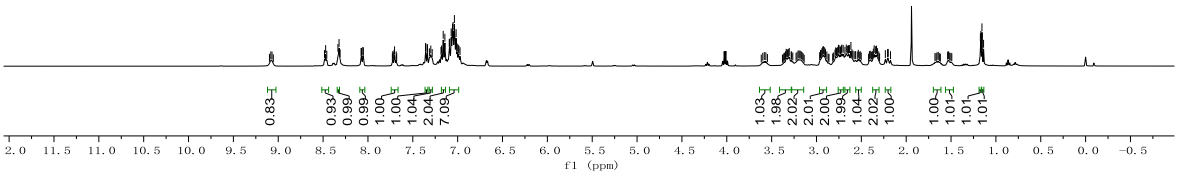
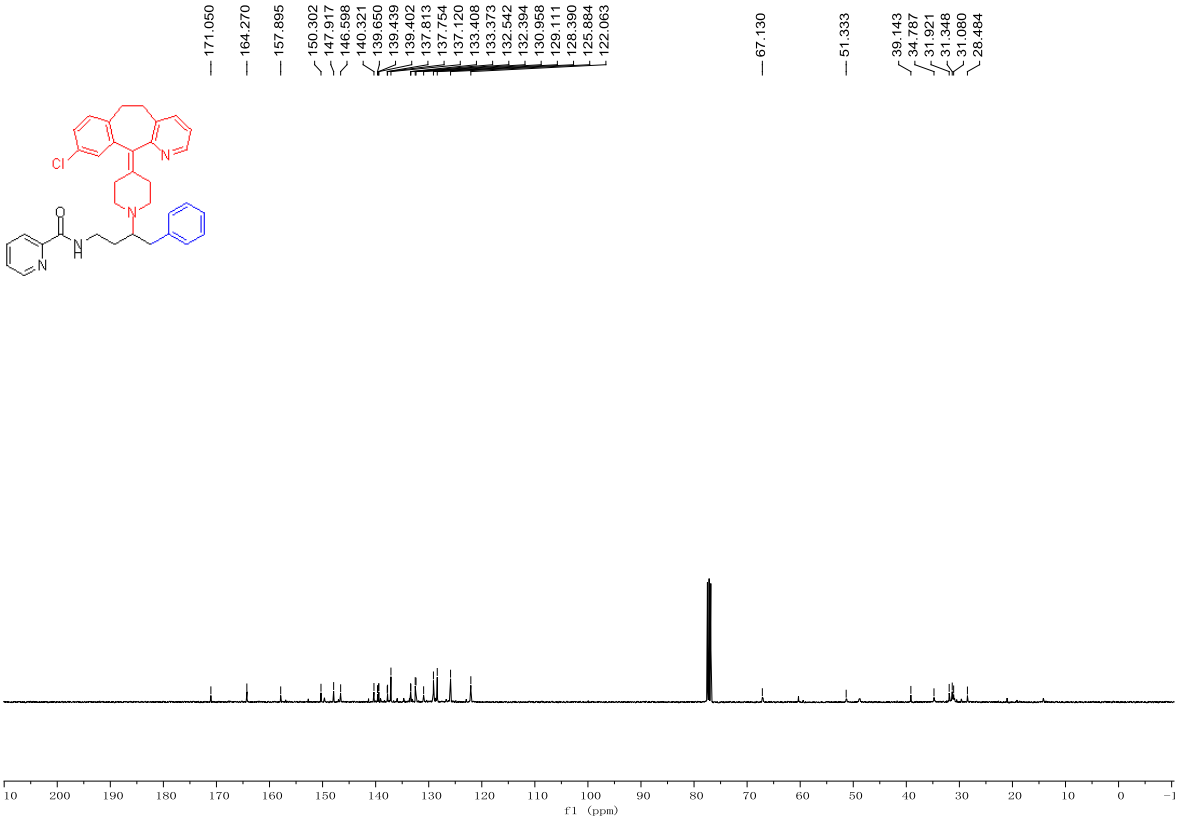
8.211
8.193
8.070
7.869
7.850
7.831
7.760
7.753
7.738
7.731
7.717
7.696
7.691
7.671
7.443
7.424
7.408
6.868
6.846
5.101
5.085
5.069
3.730
3.713
3.686
3.669
3.653
3.545
3.528
3.512
3.495
2.768
2.750
2.731
2.618
2.601
2.044
1.657
1.276
1.258
1.242
1.209
1.193


1.07
1.00
0.88
1.09
5.04
2.02
2.01
1.08
0.96
0.99
1.08
1.06
0.94
4.02
6.00
6.06
8.00
f1 (ppm)


173.244
164.440
164.377
159.515
159.476
150.116
148.107
147.948
146.865
137.426
137.211
135.932
132.005
131.045
130.969
130.213
130.148
129.143
128.369
126.159
125.930
122.272
117.304
79.459
69.322
39.242
29.390
28.513
25.481
21.630
21.586
f1 (ppm)


8.472
8.334
8.322
8.311
8.077
8.071
8.057
8.052
7.703
7.359
7.355
7.340
7.336
7.315
7.299
7.284
7.186
7.179
7.161
7.142
7.094
7.089
7.072
7.062
7.057
7.051
7.036
7.017
7.005
6.993
6.986
6.974
3.338
3.330
3.314
3.303
2.939
2.931
2.924
2.914
2.906
2.899
2.786
2.778
2.770
2.762
2.756
2.750
2.739
2.733
2.723
2.717
2.703
2.683
2.668
2.656
2.644
2.639
2.633
2.616
2.605
2.366
2.357
2.343
2.331
2.319
2.208
2.200
1.173
1.168
1.155
1.137
Cl
N
O
N
H
N


0.83
0.93
0.99
0.99
1.00
1.00
1.04
2.04
7.09
1.03
1.98
2.02
2.01
2.00
1.99
1.04
2.02
1.00
1.00
1.01
1.01
1.01
2.0 11.5 11.0 10.5 10.0 9.5 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 −0.5
f1 (ppm)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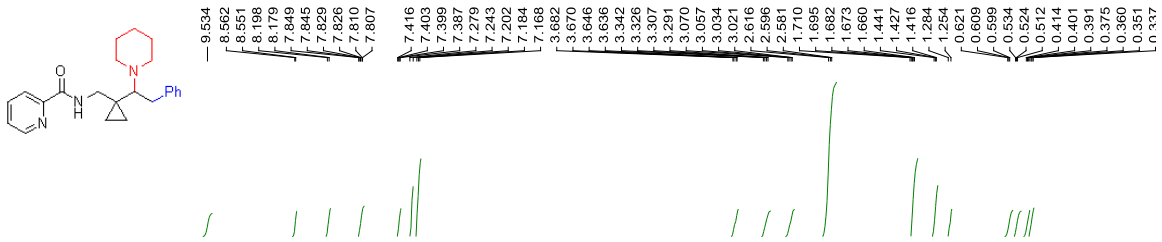
150.302
147.917
146.598
140.321
139.650
139.439
139.402
137.813
137.754
137.120
133.408
133.373
132.542
132.394
130.958
129.111
128.390
125.884
122.063
67.130
51.333
39.143
34.787
31.921
31.348
31.080
28.484
Cl
N
O
N
H
N
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −1
f1 (ppm)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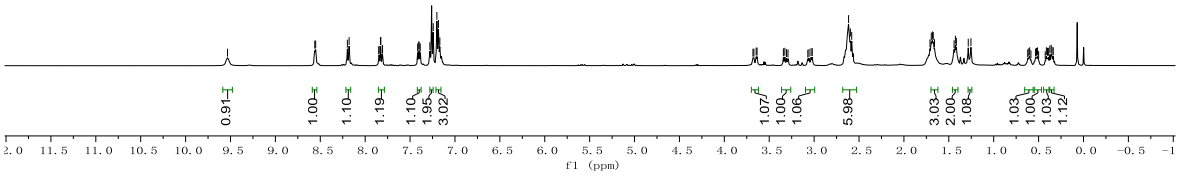
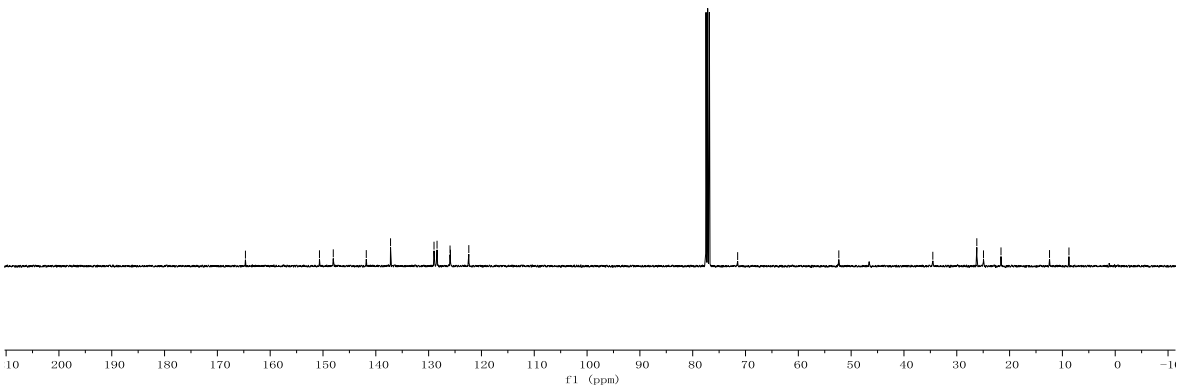
8.551
8.198
8.179
7.849
7.845
7.829
7.826
7.810
7.807
7.416
7.403
7.399
7.387
7.279
7.243
7.202
7.184
7.168
3.682
3.670
3.646
3.636
3.342
3.326
3.307
3.291
3.070
3.057
3.034
3.021
2.616
2.596
2.581
1.710
1.695
1.682
1.673
1.660
1.441
1.427
1.416
1.284
1.254
0.621
0.609
0.599
0.534
0.524
0.512
0.414
0.401
0.391
0.375
0.360
0.351
O
N
H
N
Ph


0.91
1.00
1.10
1.19
1.10
1.95
3.02
1.07
1.00
1.06
5.98
3.03
2.00
1.08
1.03
1.00
1.03
1.12
f1 (ppm)


164.685
150.668
148.055
141.806
137.212
128.980
128.408
125.947
125.869
122.393
71.489
52.335
34.535
26.207
24.944
21.627
12.439
8.761
O
N
H
N
Ph


f1 (ppm)