Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting information for the article entitled

Palladium-catalysed α and β C−H allylation of aryl alkenes

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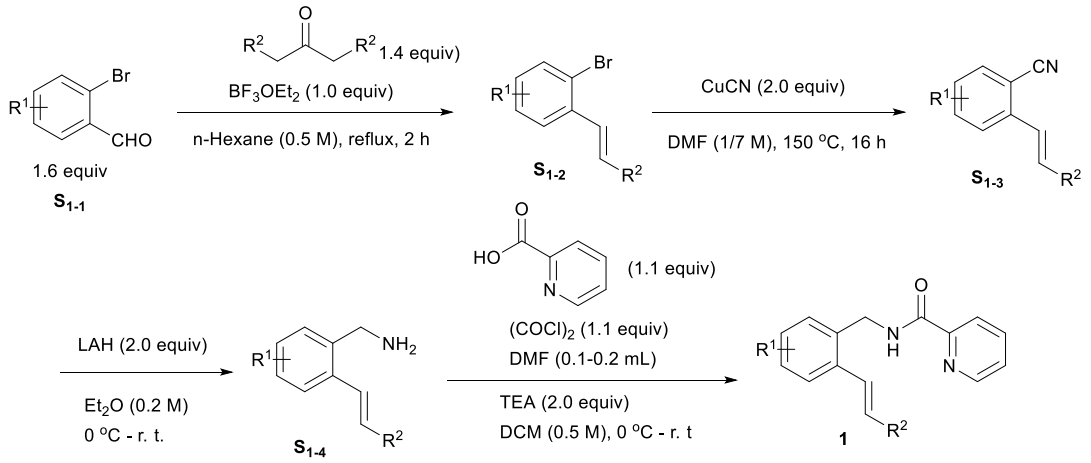
Content

General Methods

Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with basic solution of potassium permanganate or acidic solution of ceric molybdate. Flash column chromatography was performed using Merck aluminum oxide 90 active neutral with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use. Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on Bruker AMX 400 spectrophotometer ($CDCl_3$ as solvent)，and Bruker AMX 500 spectrophotometer ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 7.26, singlet). Multiplicities were given as: s (singlet), d (doublet), t (triplet), dd (doublets of doublet) or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million(ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 77.0, triplet). Mass spectrometry was performed by Waters Q-Tof Premier Micromass instrument, using Electro Spray Ionization (ESI) mode. IR spectra were recorded as thin films on KBr plates on a Bio-Rad FTS 165 FTIR spectrometer and are reported in frequency of absorption (cm^{-1}). $Pd(OAc)_2$ was purchased from Energy Chemical and used directly. Other reagents, unless otherwise noted below, are commercially available from TCI, Energy Chemical, Alfa Aesar (China) Chemical Co. Ltd. and used without further purification.

2. Substrate Synthesis

2.1 General Procedure A for Substrate Synthesis



General Procedure for Tandem Aldol-Grob Reaction[1]: An oven-dried Schlenk flask was charged with 2-bromobenzaldehyde (**$S_{1\text{-}1}$**) (1.6 equiv, 8.0 mmol), 5-nonanone (1.0 equiv, 5.0 mmol) and dry *n*-hexane (0.5 M, 10.0 mL). A solution of boron trifluoride diethyl etherate (BF_3•OEt_2) (1.0 equiv, 5.0 mmol) was added to the Schlenk flask, and the mixture was heated to reflux with stirring for 2 hours. The reaction was cooling down to room temperature and quenched with water. The aqueous layer was extracted three times with diethyl ether. The combined organic phase was dried over anhydrous Na_2SO_4. The organic phase was filtrated and concentrated in vacuo, and the crude product was purified by silica gel chromatography to obtain compound **($S_{1\text{-}2}$)** as a colorless liquid.

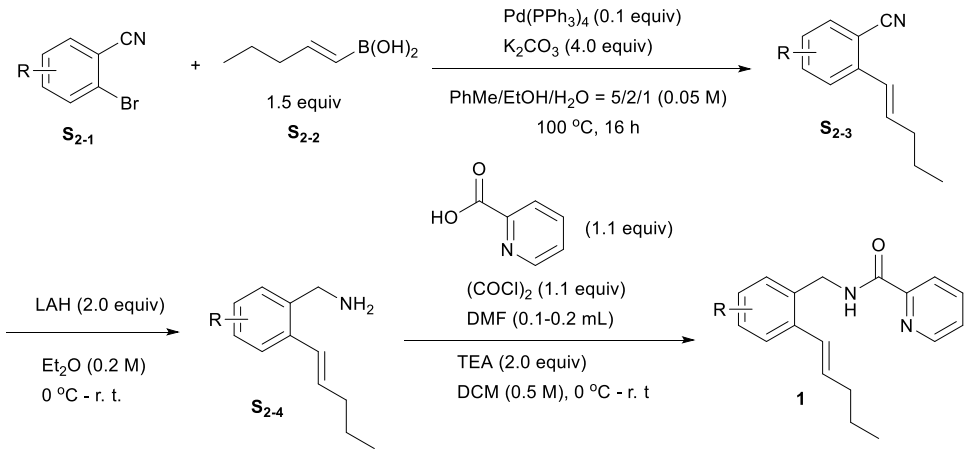
General Procedure for Cyanation Reaction[2]: To a mixture of CuCN (2.0 equiv, 10.0 mmol) in DMF (0.14 M, 35.7 mL) was added 1-bromo-2-(pent-1-yn-1-yl) benzene **($S_{1\text{-}2}$)** (1.0 equiv, 5.0 mmol) at room temperature. The reaction was heated to 150 °C with stirring for 16 h until the substrate was completely consumed. After that, the reaction was cooled down to room temperature, the mixture was diluted with H_2O (20 mL) and extracted with EtOAc (30 mL × 3). The organic layer was combined and dried over anhydrous Na_2SO_4. The organic phase was filtrated and concentrated in vacuo, and the resulting residue was purified by silica gel column chromatography

(PE / EA) to give compound **($S_{1\text{-}3}$)**.

General Procedure for Benzonitrile Reduction[3]: A solution of substituted benzonitrile **($S_{1\text{-}3}$)** in Et_2O (0.2 M, 15.0 mL) was added $LiAlH_4$ (2.0 equiv, 1.0 M in THF) over 30 min at 0°C and stirred for 2 h at room temperature. Then, 2 N aq. NaOH was added slowly until a clear solution was obtained. The Et_2O layer was separated and the aqueous phase was extracted with Et_2O (20 mL × 3). The organic layers were combined and dried over anhydrous Na_2SO_4. After removing the solvent under reduced pressure, the resulting amine **($S_{1\text{-}4}$)** was obtained and used in the next step without further purification.

General Procedure for Amide Preparation[4]: A 50 mL round-bottomed flask immersed in a 0 °C bath (ice and water) was charged with picolinic acid (1.0 equiv, 5.0 mmol) and CH_2Cl_2 (0.5 M, 10.0 mL). The stirred suspension was added oxalyl chloride (1.1 equiv, 2.0 M in DCM) in dropwise over a 15-minute period followed by the addition of DMF (50 M, catalytic amount) in one portion, producing a rust-red color with the evolution of a gas. The mixture was kept in the cooling bath for 1 h and then allowed to warm to room temperature. After gas evolution ceased, the mixture was again cooled to 0 °C, and NEt_3 (2.0 equiv, 10.0 mmol) was added in dropwise over a 15 minute period followed by dropwise addition of benzylamine (1.1 equiv, 5.5 mmol) over a 15 minute period. The brown mixture was kept in the cooling bath for 30 minutes and then allowed to warm to room temperature. Stirring was continued at room temperature for 8 h. After the reaction was finished, the solvent was removed under vacuo to give the crude product as a brown solid which was extracted with CH_2Cl_2 (10 mL × 3). The organic phases were combined and dried over anhydrous Na_2SO_4. The organic phase was filtrated and concentrated in vacuo, and the resulting residue was purified by silica gel column chromatography (PE/EA = 8/1) to obtain the corresponding amide product.

2.2 General Procedure B for Substrate Synthesis



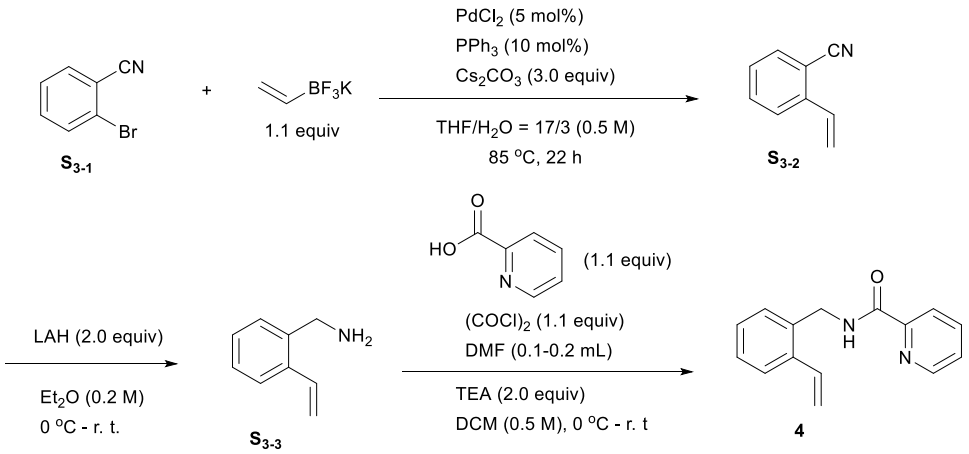
Synthesis of (*E*)-alkenyl boronic acid[5]: To a solution of terminal alkyl alkyne (1.0 equiv, 2.0 mmol) in DCM (1 M, 2.0 mL) was added $HBBr_2Me_2S$ (1 M in DCM, 2.0 mL) at 0 °C. After addition, the mixture was warm to room temperature and stirred for 4 h. The resulting solution was then dropwisely added to an ice-cooled mixture of Et_2O/H_2O (*v:v* = 2:1, 3.0 mL) and continued stirring for 15 minutes. The aqueous layer was extracted with Et_2O (3 × 10 mL). The organic phase was combined and dried over anhydrous Na_2SO_4. After filtrated and concentrated in vacuo to afford (*E*)-alkenyl boronic acid **($S_{2\text{-}2}$)** which was used directly.

Suzuki Reaction[6]: A solution of (*E*)-pent-1-en-1-ylboronic acid (1.5 equiv, 7.5 mmol), $Pd(PPh_3)_4$ (0.1 equiv, 0.05 mmol), K_2CO_3 (4.0 equiv, 20.0 mmol), K_3PO_4 (3.0 equiv, 15.0 mmol), and ortho-bromoarene (1.0 equiv, 5.0 mmol) in toluene/EtOH/H_2O (100 mL, *v:v:v* = 5/2/1, 0.05 M) was heated to 100 °C with stirring for 16 h in a sealed tube under argon atmosphere. Then the reaction was cooled to room temperature and diluted with H_2O (20 mL) followed by extraction with EtOAc (3 × 30 mL). The organic phase was combined and dried over anhydrous Na_2SO_4. After

filtration and concentrated in vacuo, the crude product was purified by silica gel chromatography (SiO_2, PE/EA) to obtain the corresponding product.

Benzonitrile Reduction and Amide Preparation was performed following **the general procedure A**.

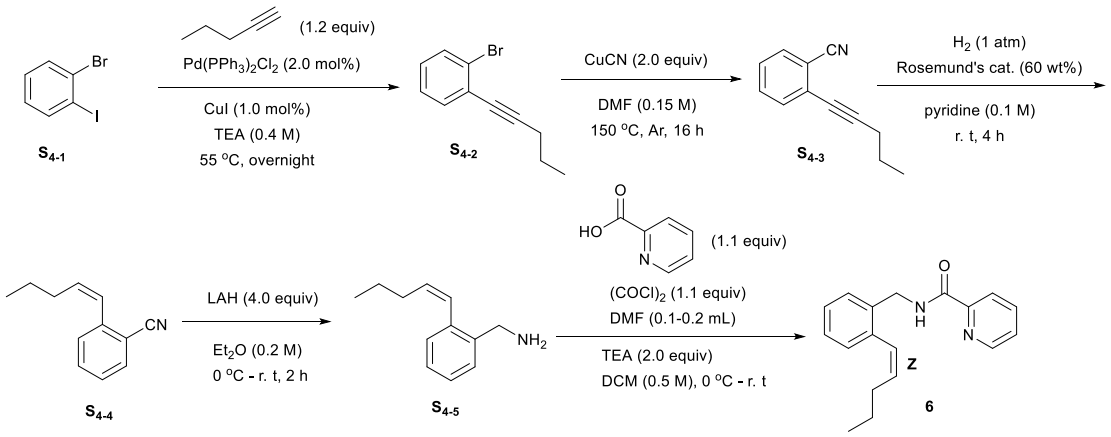
2.3 General Procedure C for Substrate Synthesis



General Procedure for Heck Reaction: A solution of potassium vinyl trifluoroborate (1.1 equiv, 5.5 mmol), $PdCl_2$ (5 mol%, 0.25 mmol), PPh_3 (10 mol%, 0.5 mmol), Cs_2CO_3 (3.0 equiv, 15.0 mmol), and substituted 2-bromobenzaldehyde **($S_{3\text{-}1}$)** (1.0 equiv, 5.0 mmol) in THF/H_2O (0.5 M , *v:v* = 17:3, 10.0 mL) was heated at 85 °C under Ar atmosphere in a sealed tube. The reaction mixture was stirred at 85 °C for 22 h, then cooled to r.t. and diluted with H_2O (3 mL), which was extracted by EtOAc (30 mL × 3). The organic layer was concentrated in vacuo, and the crud product was purified by silica gel chromatography (SiO_2, PE/EA = 98/2) to obtain the corresponding product **($S_{3\text{-}2}$)**.

Benzonitrile Reduction and Amide Preparation was performed following **the general procedure A**.

2.4 General Procedure D for Substrate Synthesis



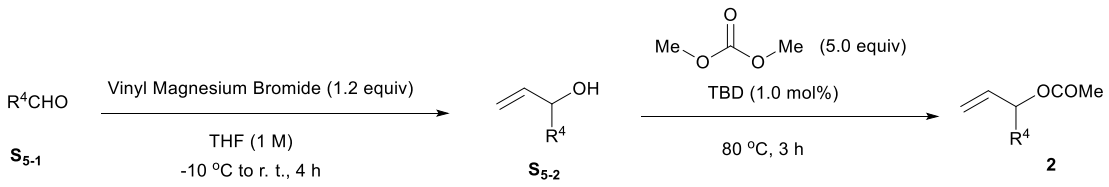
Alkynylation Reaction[7]: A solution of $Pd(PPh_3)_2Cl_2$ (2.0 mol%, 0.1 mmol), CuI (1.0 mol%, 0.05 mmol), 1-bromo-2-iodobenzene (1.0 equiv, 5.0 mmol) and 1-pentyne (1.2 mmol, 6.0 mmol) in TEA (0.4 M, 12.5 mL) was heated to 55 °C with stirring for overnight. Then, the reaction was cooled to room temperature and diluted with H_2O (20 mL), and extracted with EtOAc (3 × 30 mL). The organic phase was combined and dried over anhydrous Na_2SO_4. After filtration and concentration in vacuo, the crude product was purified by silica gel chromatography (SiO_2, PE / EA) to obtain the corresponding product **($S_{4\text{-}2}$)**.

Cyanation Reaction was performed following **the general procedure A** to obtain the corresponding product (**$S_{4\text{-}3}$)**

Hydrogenation Reaction: Following the previously reported procedure with a slight modification, a two-necked flask was changed with Rosenmund's catalyst (5% Pd on $BaSO_4$, 521.0 mg), alkyne **($S_{4\text{-}3}$)** (1.0 equiv, 3.9 mmol) and pyridine (0.1 M, 39 mL). The reaction bottle was vacuumed three times and then backfilled with H_2 three times. And then, the solution was allowed to stir at room temperature for 4 h until the reaction was completed (monitored by TLC). The reaction solution was diluted with ethyl acetate (30 mL) and washed with HCl (2 M, 3 × 30 mL), water (1 × 50 mL) and brine (1 × 50 mL). The organic phase was then dried over Na_2SO_4. After filtration and concentration in vacuo, the crude product was purified by silica gel chromatography (SiO_2, PE / EA) to obtain olefin **($S_{4\text{-}4}$)**.

Benzonitrile Reduction and Amide Preparation was performed following **the general procedure A**.

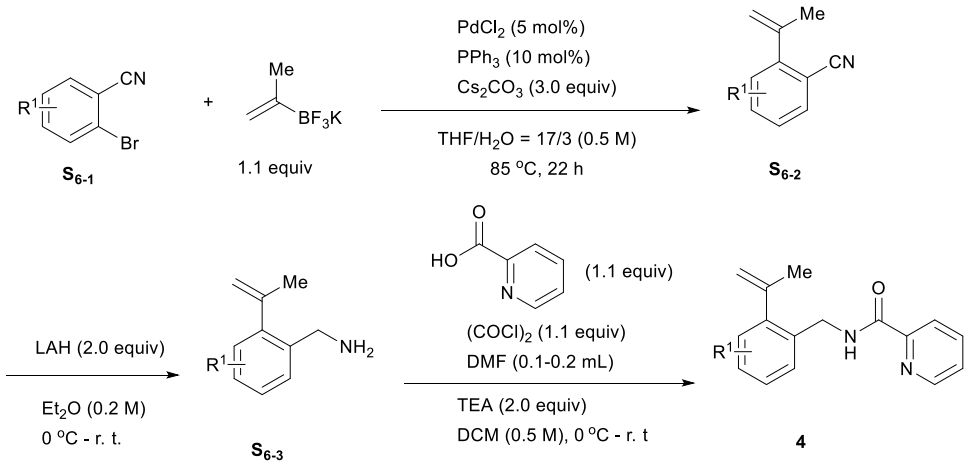
2.5 General Procedure E for Substrate Synthesis



Synthesis of allyl alcohol: Corresponding aldehyde (1.0 equiv, 10.0 mmol) was added to THF (1 M, 10.0 mL) in a dry 50 mL two-port flask at 0 °C. Then a solution of vinyl magnesium bromide (1.2 equiv, 12.0 mmol) in THF (1 M, 10.0 mL) was slowly dropped into the reaction. After stirring for 15 min at 0 °C, the reaction was gradually raised to room temperature and stirred for 4 h. The reaction was quenched with sat. aq NH_4Cl (10 mL) and then extracted with EA (3 × 20 mL). The combined organic layer was washed with brine (20 mL) and dried over anhydrous Na_2SO_4. The solvent was removed by vacuum and the residue was purified by silica gel column chromatography (PE/EA = 4/1) to obtain the corresponding allyl alcohol **($S_{5\text{-}2}$)**.

Allyl carbonate synthesis[7]: In a dry 100 mL round-bottled flask, the allyl alcohol **($S_{5\text{-}2}$)** (1.0 equiv, 3.0 mmol), dimethyl carbonate (5.0 equiv, 15.0 mmol) and 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD) (0.01 equiv, 0.03.0 mmol) were stirred at 80 °C under argon protection for 3 h, the heating was stopped, and the reaction mixture was cooled to room temperature. The mixture was purified by silica gel column chromatography (PE/EA = 10/1) to obtain the required methyl allyl carbonate Compounds.

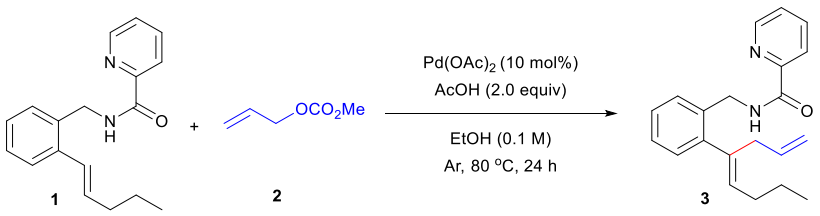
2.6 General Procedure F for Substrate Synthesis



General Procedure for Heck Reaction: A solution of potassium isopropenyl trifluoroborate (1.1 equiv, 5.5 mmol), $PdCl_2$ (5.0 mol%, 0.25 mmol), PPh_3 (10 mol%, 0.5 mmol), Cs_2CO_3 (3.0 equiv, 15.0 mmol), and bromobenzene (**$S_{6\text{-}1}$**) (1.0 equiv, 5.0 mmol) in THF/H_2O (0.5 M, *v:v* = 17:3, 10.0 mL) was heated at 85 °C under Ar atmosphere in a sealed tube. The reaction mixture was stirred at 85 °C for 22 h, cooled to r.t. and diluted with H_2O (3 mL). After extraction with EtOAc (30 mL × 3), the organic layer was combined and dried over anhydrous Na_2SO_4. After filtration and concentration, the solvent was removed in vacuo, and the crud product was purified by silica gel chromatography (SiO_2, PE/EA = 98/2) to obtain the corresponding product **($S_{6\text{-}2}$)**.

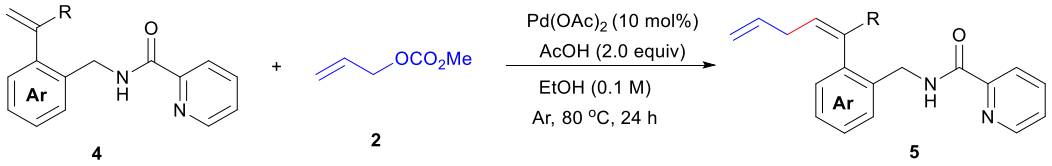
Benzonitrile Reduction and Amide Preparation was performed following **the general procedure A**.

3. General Procedure for the Allylation

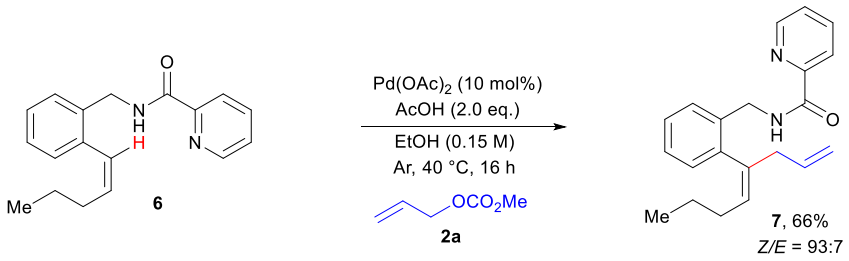


A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and

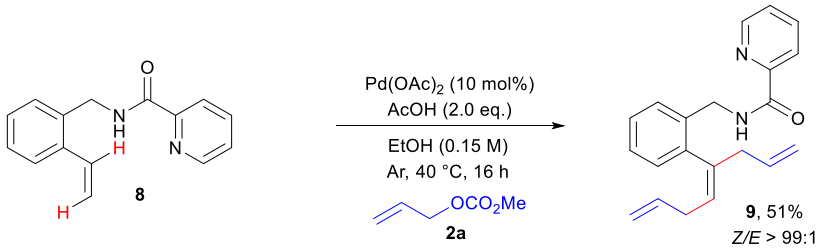
ethanol (0.1 M, 1.5 mL). Then, styrene **1** (1.0 equiv, 0.15 mmol), allyl methyl carbonate **2** (4.0 equiv, 0.6 mmol) and AcOH (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 80 ℃ in oil bath with stirring for 24 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (PE/EA) to provide product **3**.



A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and ethanol (0.1 M, 1.5 mL). Then, styrene **4** (1.0 equiv, 0.15 mmol), allyl methyl carbonate **2** (4.0 equiv, 0.6 mmol) and AcOH (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 80 ℃ in oil bath with stirring for 24 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (PE/EA) to provide product **5**.

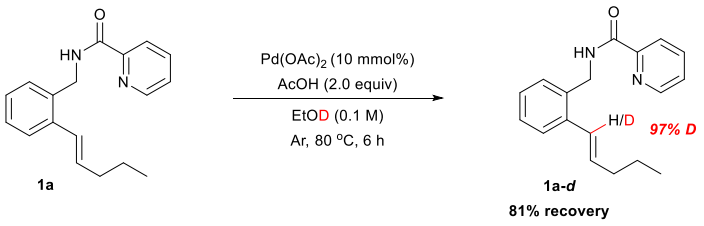


A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and ethanol (0.15 M, 1.0 mL). Then, styrene **6** (1.0 equiv, 0.15 mmol), allyl methyl carbonate **2a** (4.0 equiv, 0.6 mmol) and AcOH (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 40 ℃ in oil bath with stirring for 16 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (PE/EA) to provide product **7**.

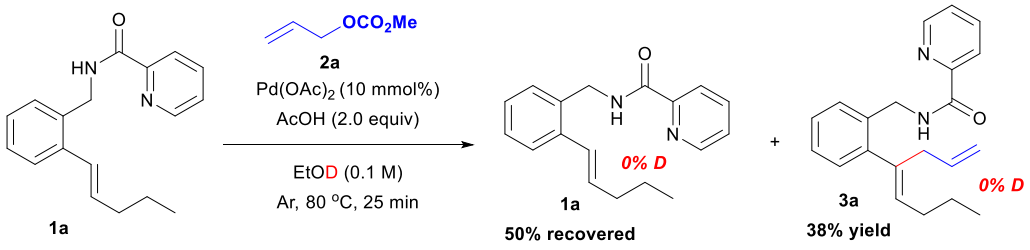


A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and ethanol (0.15 M, 1.0 mL). Then, styrene **8** (1.0 equiv, 0.15 mmol), allyl methyl carbonate **2a** (4.0 equiv, 0.6 mmol) and AcOH (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 40 ℃ in oil bath with stirring for 16 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (PE/EA) to provide product **9**.

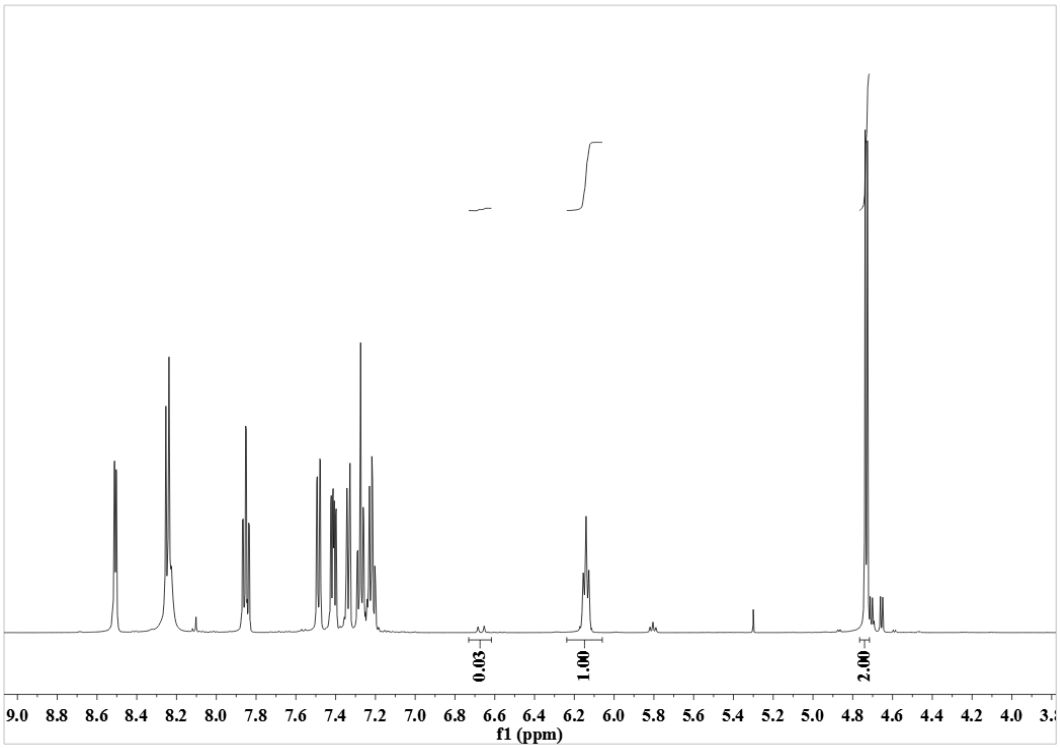
4. Deuterium-Labelled Experiment



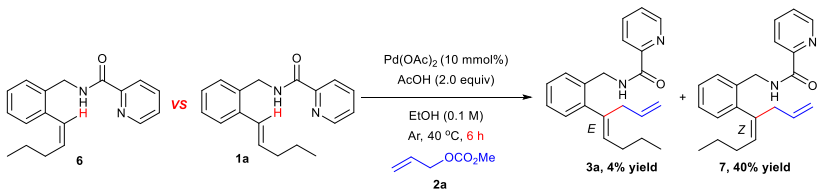
A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and EtOD (0.1 M, 1.5 mL). Then, acrylamide **1a** (1.0 equiv, 0.15 mmol) and acid (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated in oil bath to 80 ℃ with stirring for 6 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide the product. The product yields were estimated by ^1H NMR.



A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.015 mmol) and EtOD (0.1 M, 1.5 mL). Then, **1a** (1.0 equiv, 0.15 mmol), allyl methyl carbonate **2a** (4.0 equiv, 0.6 mmol) and AcOH (2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 80 ℃ with stirring for 25 min. After cooling down, the mixture was directly applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide product **1a**. The product yields were estimated by ^{1}H NMR.



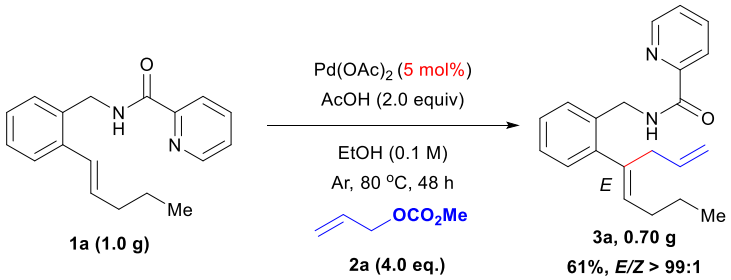
5. Competition Experiments



A dry screw-cap vial was charged with $Pd(OAc)_2$ (10 mol%, 0.01 mmol) and ethanol (0.1 M, 2.0 mL). Then, acrylamide **6** (1.0 equiv, 0.1 mmol), acrylamide **1a** (1.0 equiv, 0.1 mmol), allyl methyl carbonate **2a** (4.0 equiv, 0.6 mmol) and AcOH

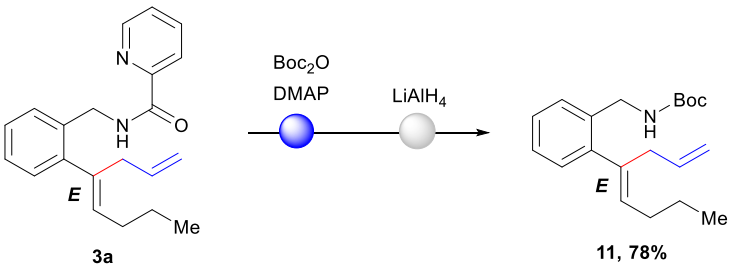
(2.0 equiv, 0.3 mmol) were added into the solution in sequence. The vial was sealed under argon and heated to 40 ℃ with stirring for 6 h. After cooling down, the mixture was directly applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide product **3a** (4% yield) and **7** (40% yield).

6. Gram-Scaled Synthesis



A dry screw-cap vial was charged with $Pd(OAc)_2$ (5 mol%, 0.18 mmol) and ethanol (0.1 M, 30 mL). Then, acrylamide **1a** (1.0 equiv, 3.57 mmol), allyl methyl carbonate **2a** (4.0 equiv, 14.27 mmol) and AcOH (2.0 equiv, 7.13 mmol) were added into the solution in sequence. The vial was sealed under argon and heated in oil bath to 80 ℃ with stirring for 48 h. After cooling down, the mixture was concentrated and directly applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide product **3a** (61% yield).

7. Directing Group Removal



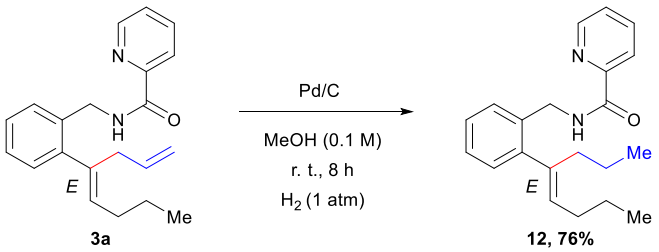
DG removal was performed according to previous literature[8]:

Step 1 Boc-anhydride (5.0 equiv, 1.0 mmol) was added to a solution of **3a** (1.0 equiv, 0.2 mmol) and DMAP (2.0 equiv, 0.4 mmol) in MeCN (0.1 M, 2.0 mL) and the reaction mixture was stirred for 4 h. The reaction mixture was quenched with sat. aq NH_4Cl (10 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic

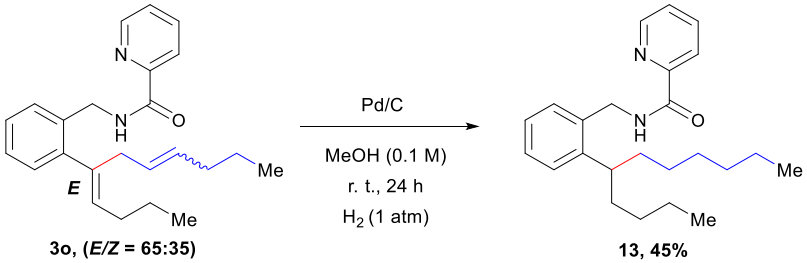
extracts were dried over Na_2SO_4, concentrated under reduced pressure and purified by column chromatography (SiO_2, PE / EA=10:1 to 2: 1) to give N-Boc-amide (82% yield).

Step 2 To a solution of N-Boc-amide in THF/^{t}BuOH (1:1, 0.2 M) was added dropwisely $LiAlH_4$ (2.0 equiv，2.5 M in THF) over 30 min at 0 °C and stirred at room temperature for 2 h, and 2 N NaOH was added slowly at 0 °C until a clear solution was obtained. The organic layer was separated and the aqueous phase was extracted with Et_2O (20 mL × 3). The organic layer was combined and dried over anhydrous Na_2SO_4. After removing the solvent under reduced pressure, the residue was purified by column chromatography on silica gel with PE/EA to give amine **11** as a clear liquid (95% yield and 78% yield for two steps respectively).

8. Hydrogenation



Pd/C (20 wt%) was added to a solution of amide **3a** (1.0 equiv, 0.1 mmol) in MeOH (0.1 M, 1.0 mL). The reaction mixture was vacuumed and then backfilled with H_2 (1 atm) for three times, and then the reaction mixture was stirred at room temperature for 8 h. The reaction mixture was filtered, concentrated in vacuo and applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide product **12** (76% yield).



Pd/C (20 wt%) was added to a solution of amide **3o** (1.0 equiv, 0.1 mmol) in

MeOH (0.1 M, 1.0 mL). The reaction mixture was vacuumed and then backfilled with H_2 (1 atm) for three times, and then the reaction mixture was stirred at room temperature for 8 h. The reaction mixture was filtered, concentrated in vacuo and applied to a flash column chromatography for separation (ethyl acetate/petroleum ether mixtures) to provide product **13** (45% yield).

9. References

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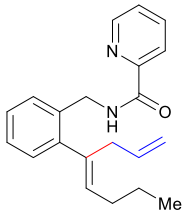
11. Characterization Data



3a
(*E*/*Z* = 93:7)

(*E*)-*N*-(2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3a):

Following the general procedure 1, **3a** was obtained as a yellow oil (37.0 mg, 77% yield, *E*/*Z* = 93:7).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (ddd, *J* = 4.7, 1.6, 0.9 Hz, 1H), 8.26 (s, 1H), 8.23 – 8.21 (m, 1H), 7.84 (td, *J* = 7.7, 1.7 Hz, 1H), 7.43 – 7.37 (m, 2H), 7.24 – 7.20 (m, 2H), 7.14 – 7.11 (m, 1H), 5.76 – 5.68 (m, 1H), 5.42 (t, *J* = 7.2 Hz, 1H), 5.02 – 4.94 (m, 2H), 4.65 (d, *J* = 5.9 Hz, 2H), 3.16 (d, *J* = 6.7 Hz, 2H), 2.19 (q, *J* = 7.3 Hz, 2H), 1.50 – 1.43 (m, 2H), 0.95 (t, *J* = 7.4 Hz,3H).

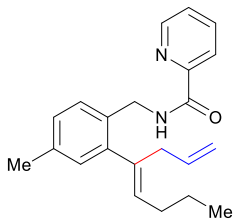
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.00, 148.93, 147.01, 142.98, 136.28, 135.79, 134.25, 134.19, 130.87, 128.39, 127.71, 126.03, 125.99, 125.06, 121.25, 114.83, 40.36, 36.12, 29.28, 21.75, 12.93.

HRMS (ESI) for $C_{21}H_{24}N_2O$ $[M+H]^+$: 321.1961, found: 321.1969.

FTIR (KBr, cm^{-1})

3854.21, 3739.25, 3444.93, 2959.81, 2934.58, 2829.82, 2715.89, 1602.46, 1364.10, 1126.17, 1078.50, 774.84.



3b
(E/Z > 99:1)

(*E*)-*N*-(4-methyl-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3b)

Following the general procedure 1, **3b** was obtained as a ye llow liquid (34.1 mg, 68% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.50 (d, *J* = 4.2 Hz, 1H), 8.21 (d, *J* = 7.8 Hz, 2H), 7.84 (td, *J* = 7.7, 1.7 Hz, 1H), 7.41 – 7.39 (m, 1H), 7.28 (d, *J* = 7.8 Hz, 1H),

7.04 (d, *J* = 7.8 Hz, 1H), 6.94 (s, 1H), 5.76 – 5.68 (m, 1H), 5.40 (t, *J* = 7.2 Hz, 1H), 5.02 – 4.95 (m, 2H), 4.60 (d, *J* = 5.8 Hz, 2H), 3.15 (d, *J* = 6.7 Hz, 2H), 2.32 (s, 3H), 2.18 (q, *J* = 7.3 Hz, 2H), 1.49 – 1.42 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H).

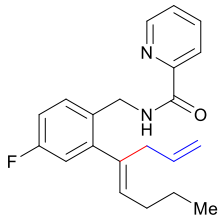
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.93, 150.01, 148.00, 143.96, 137.26, 136.90, 136.70, 135.36, 132.20, 131.72, 129.98, 128.88, 127.75, 126.02, 122.22, 115.75, 41.17, 37.16, 30.31, 22.79, 21.08, 13.97.

HRMS (ESI) for $C_{22}H_{26}N_2O$ $[M+H]^+$: 335.2118, found: 335.2114.

FTIR (KBr, cm^{-1})

3848.60 3750.47 3440.67 2962.62 2928.97, 2830.40, 2724.30 2359.81 2314.95 1606.77, 1362.57, 1235.51 1182.24 1068.85 774.50 640.21 607.48 563.35.



3c
(E/Z > 99:1)

(*E*)-*N*-(4-fluoro-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3c)

Following the general procedure 1, **3c** was obtained as a yell ow liquid (36.0 mg, 71% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.52 (ddd, *J* = 4.7, 1.5, 0.8 Hz, 1H), 8.24 – 8.21 (m, 2H), 7.87 – 7.83 (m, 1H), 7.43 – 7.40 (m, 1H), 7.36 (dd, *J* = 8.5, 5.9 Hz, 1H), 6.93 – 6.89 (m, 1H), 6.83 (dd, *J* = 9.6, 2.7 Hz, 1H), 5.75 – 5.67 (m, 1H), 5.44 (t, *J* = 7.3 Hz, 1H), 5.03 – 4.97 (m, 2H), 4.59 (d, *J* = 6.0 Hz, 2H), 3.14 (d, *J* = 6.8 Hz, 2H), 2.19 (q, *J* = 7.3 Hz, 2H), 1.50 – 1.43 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$)

δ 164.02, 161.58 (d, J_{C-F} = 246.2 Hz), 149.85, 148.05, 145.97 (d, J_{C-F} = 7.6 Hz), 137.34, 136.00, 134.90, 132.45, 131.16 (d, J_{C-F} = 3.1 Hz), 130.47 (d, J_{C-F} = 8.4 Hz), 126.15, 122.27, 116.22, 116.04 (d, J_C

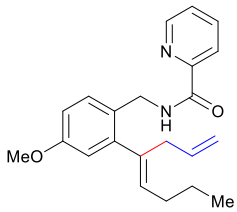
$_{-F}$ = 20.8 Hz), 113.80 (d, J_{C-F} = 21.1 Hz), 40.73, 36.90, 30.27, 22.70, 13.95.

^{19}F NMR (471 MHz, $CDCl_3$) δ -115.70.

HRMS (ESI) for $C_{21}H_{23}FN_2O$ $[M+H]^+$: 339.1867, found: 339.1866.

FTIR (KBr, cm^{-1})

3852.67 3744.86 3624.30 2968.22 2934.58 2829.98 2721.50 2359.81 2328.97 1606.45 1362.38 1227.10 1190.65 1075.70 774. 36, 643.93 607.48 562.62.



3d
(*E*/*Z* = 90:10)

(*E*)-*N*-(4-methoxy-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3d)

Following the general procedure 1, **3d** was obtained as a yellow liquid (35.7 mg, 68% yield, *E*/*Z* = 90:10).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.50 (d, *J* = 4.1 Hz, 1H), 8.21 (d, *J* = 7.8 Hz, 1H), 8.17 (s, 1H), 7.86 – 7.82 (m, 1H), 7.40 (ddd, *J* = 7.6, 4.7, 1.2 Hz, 1H), 7.31 (d, *J* = 8.5 Hz, 1H), 6.77 (dd, *J* = 8.5, 2.7 Hz, 1H), 6.68 (d, *J* = 2.7 Hz, 1H), 5.77 – 5.69 (m, 1H), 5.43 (t, *J* = 7.2 Hz, 1H), 5.03 – 4.95 (m, 2H), 4.57 (d, *J* = 5.8 Hz, 2H), 3.79 (s, 3H), 3.14 (d, *J* = 6.7 Hz, 2H), 2.20 – 2.16 (m, 2H), 1.49 – 1.42 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H).

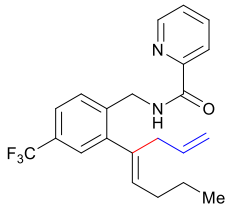
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.88, 158.44, 150.02, 148.01, 145.44, 137.27, 136.74, 135.31, 131.83, 130.30, 127.50, 126.02, 122.21, 115.87, 114.92, 112.27, 55.27, 40.94, 37.07, 30.28, 22.76, 13.96.

HRMS (ESI) for $C_{22}H_{26}N_2O_2$ $[M+H]^+$: 351.2067, found: 351.2069.

FTIR (KBr, cm^{-1})

3843.06, 3739.25, 2962.62, 2833.64, 2713.08, 2357.01, 1606.50, 1362.22, 1232.71, 1182.24, 1067.29, 772.90, 641.12, 607.48, 562.62.



3e

(*E*/*Z* > 99:1)

(*E*)-*N*-(2-(octa-1,4-dien-4-yl)-4-(trifluoromethyl)benzyl) picolinamide (3e)

Following the general procedure 1, **3e** was obtained as a brown oil (33.8 mg, 58% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.57 – 8.52 (m, 1H), 8.33 (s, 1H), 8.23 – 8.21 (m, 1H), 7.88 – 7.84 (m, 1H), 7.52 – 7.50 (m, 1H), 7.47 – 7.42 (m, 2H), 7.37 (s, 1H), 5.75 – 5.67 (m, 1H), 5.46 (t, *J* = 7.3 Hz, 1H), 5.02 – 4.99 (m, 2H), 4.69 (d, *J* = 6.1 Hz, 2H), 3.17 (d, *J* = 6.8 Hz, 2H), 2.23 (q, *J* = 7.3 Hz, 2H), 1.53 – 1.45 (m, 2H), 0.97 (q, *J* = 7.0 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$)

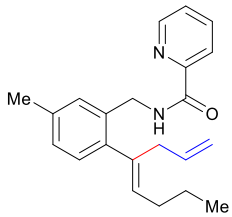
δ 163.22, 148.64, 147.08, 143.28, 138.46, 136.38, 134.78, 133.64, 131.97, 128.19 (q, $J_{C\text{-}F}$ = 32.3 Hz), 127.79, 125.27, 125.14 (d, $J_{C\text{-}F}$ = 3.7 Hz), 123.09 (d, $J_{C\text{-}F}$ = 270.5 Hz), 122.73 (q, $J_{C\text{-}F}$ = 3.8 Hz), 121.31, 115.43, 39.91, 35.91, 29.29, 21.66, 12.95.

^{19}F NMR (471 MHz, $CDCl_3$) δ -62.4.

HRMS (ESI) for $C_{22}H_{23}F_3N_2O$ $[M+H]^+$: 389.1835, found: 389.1835.

FTIR (KBr, cm^{-1})

3848.72, 3742.18, 3444.99, 2954.34, 2830.18, 2713.22, 2354.34, 1606.54, 1362.27, 1232.86, 1187.90, 1070.24, 774.30, 649.69, 618.85, 565.58.



3f

(*E*/*Z* = 90:10)

(*E*)-*N*-(5-methyl-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3f)

Following the general procedure 1, **3f** was obtained as a yel low liquid (24.6 mg, 49% yield, *E*/*Z* = 90:10).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.44 (dd, *J* = 2.7, 1.9 Hz, 1H), 8.17 (t, *J* = 13.1 Hz, 2H), 7.78 – 7.75 (m, 1H), 7.34 – 7.32 (m, 1H), 7.12 (s, 1H), 6.97 – 6.93 (m,

2H), 5.68 – 5.60 (m, 1H), 5.33 (t, *J* = 7.2 Hz, 1H), 4.94 – 4.87 (m, 2H),4.53 (d, *J* = 5.9 Hz, 1H), 3.07 (d, *J* = 6.7 Hz, 2H), 2.23 (s, 3 H), 2.11 (q, *J* = 7.3 Hz, 2H), 1.42 – 1.34 (m, 2H), 0.94 – 0.82 (m, 3H).

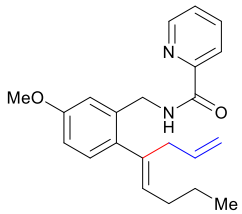
^{13}C NMR (125 MHz, $CDCl_3$)

δ 162.94, 148.99, 147.00, 140.16, 136.25, 135.69, 135.59, 134.38, 13 3.98, 130.78, 128.49, 128.31, 126.81, 125.02, 121.24, 114.74, 40.36, 36.20, 29.30, 21.77, 20.06, 12.92.

HRMS (ESI) for $C_{22}H_{26}N_2O$ $[M+H]^+$: 335.2118, found: 335.2121.

FTIR (KBr, cm^{-1})

3854.21, 3742.06, 3442.06, 2962.62, 2828.04, 2713.08, 2357.01, 1606.83, 1362.33, 1227.10, 1185.05, 1070.09, 771.53, 604.67, 560.14.



3g

(*E*/*Z* > 99:1)

(*E*)-*N*-(5-methoxy-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3g)

Following the general procedure 1, **3g** was obtained as a ye llow liquid (31.0 mg, 59% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (ddd, *J* = 4.8, 1.6, 0.9 Hz, 1H), 8.26 (s, 1H), 8.22 (dt, *J* = 7.8, 1.0 Hz, 1H), 7.84 (td, *J* = 7.7, 1.7 Hz, 1H), 7.41 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.93 (d, *J* = 2.7 Hz, 1H), 6.77 (dd, *J* = 8.4, 2.7 Hz, 1H), 5.76 – 5.68 (m, 1H), 5.40 (t, *J* = 7.2 Hz, 1H), 5.01 – 4.94 (m, 2H), 4.61 (d, *J* = 5.9 Hz, 2H), 3. 77 (s, 3H), 3.13 (d, *J* = 6.7 Hz, 2H), 2.18 (q, *J* = 7.3 Hz, 2H), 1.4 9 – 1.42 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H).

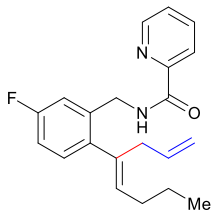
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.01, 157.39, 148.89, 147.01, 136.26, 135.56, 135.47, 135.38, 13 4.39, 130.94, 129.43, 125.06, 121.23, 114.73, 112.87, 111.47, 54.23, 40.47, 36.30, 29.33, 21.78, 12.93.

HRMS (ESI) for $C_{22}H_{26}N_2O_2$ $[M+H]^+$: 351.2067, found: 351.2070.

FTIR (KBr, cm^{-1})

3854.21, 3743.77, 3456.07, 2965.42, 2830.76, 2713.08, 2351.40, 1606. 84, 1362.39, 1235.51, 1185.05, 1072.90, 774.45, 646.62, 607.48, 56 8.22.



3h
(*E*/*Z* > 99:1)

(*E*)-*N*-(5-fluoro-2-(octa-1,4-dien-4-yl)benzyl)picolinamide (3h)

Following the general procedure 1, **3h** was obtained as a yell ow oil (37.6 mg, 74% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.53 (ddd, *J* = 4.7, 1.6, 0.9 Hz, 1H), 8.30 (s, 1H), 8.23 (dt, *J* = 7.8, 1.0 Hz, 1H), 7.86 (td, *J* = 7.7, 1.7 Hz, 1H), 7.44 – 7.42 (m, 1H), 7.09 – 7.06 (m, 2H), 6.92 – 6.88 (m, 1H), 5.75 – 5.66 (m, 1 H), 5.41 (t, *J* = 7.3 Hz, 1H), 5.01 – 4.96 (m, 2H), 4.62 (d, *J* = 6. 1 Hz, 2H), 3.13 (d, *J* = 6.7 Hz, 2H), 2.19 (q, *J* = 7.3 Hz, 2H), 1. 51 – 1.43 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$)

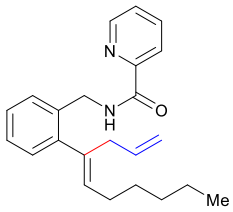
δ 163.17, 160.67 (d, J_{C-F} = 245.2 Hz), 148.73, 147.07, 138.56 (d, J_{C-F} = 3.2 Hz), 136.69 (d, J_{C-F} = 7.0 Hz), 136.36, 134.94, 134.05, 131.40, 129.88 (d, J_{C-F} = 7.9 Hz), 125.22, 121.31, 115.05, 113.83 (d, J_{C-F} = 21.8 Hz), 112.73 (d, J_{C-F} = 20.9 Hz), 40.01, 36.15, 29. 28, 21.73, 12.93.

^{19}F NMR (471 MHz, $CDCl_3$) δ -115.58.

HRMS (ESI) for $C_{21}H_{23}FN_2O$ $[M+H]^+$: 339.1867, found: 339.1869.

FTIR (KBr, cm^{-1})

3854.21, 3739.25, 3436.45, 2968.22, 2829.86, 2713.08, 2357.01, 1606. 41, 1362.55, 1227.10, 1187.85, 1070.09, 774.15, 646.73, 607.48, 559. 81.



3i
(*E*/*Z* > 99:1)

(*E*)-*N*-(2-(deca-1,4-dien-4-yl)benzyl)picolinamide(3i)

Following the general procedure 1, **3i** was obtained as a yel low oil (42.5 mg, 61% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (ddd, *J* = 4.7, 1.6, 0.9 Hz, 1H), 8.23 (ddd, *J* = 7.9, 4.3, 1.0 Hz, 2H), 7.86 – 7.82 (m, 1H), 7.42 – 7.38 (m, 2H), 7.23 – 7.21 (m, 2H), 7.14 – 7.10 (m, 1H), 5.72 (ddt, *J* = 16.9, 10.0, 6.8 Hz, 1 H), 5.42 (t, *J* = 7.2 Hz, 1H), 5.02 – 4.95 (m, 2H), 4.64 (d, *J* = 5.9 Hz, 2H), 3.15 (d, *J* = 6.7 Hz, 2H), 2.20 (dd, *J* = 14.7, 7.3 Hz, 2 H), 1.46 – 1.40 (m, 2H), 1.35 – 1.28 (m, 4H), 0.91 – 0.84 (m, 3 H).

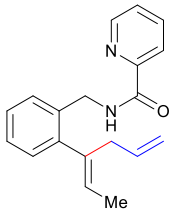
^{13}C NMR (125 MHz, $CDCl_3$)

δ 162.96, 148.94, 146.99, 142.99, 136.26, 135.54, 134.25, 134.20, 13 1.10, 128.38, 127.76, 126.02, 125.97, 125.04, 121.23, 114.81, 40.38, 36.12, 30.62, 28.25, 27.21, 21.53, 13.02.

HRMS (ESI) for $C_{23}H_{28}N_2O$ $[M+H]^+$: 349.2274, found: 349.2278.

FTIR (KBr, cm^{-1})

3851.40, 3742.06, 3442.06, 2954.21, 2825.23, 2718.69, 2357.01 1606. 03, 1361.99, 1224.30, 1185.05, 1081.31, 775.70, 649.53, 615.89, 557. 09.



3j
(*E*/*Z* = 93:7)

(*E*)-*N*-(2-(hexa-2,5-dien-3-yl)benzyl)picolinamide (3j)

Following the general procedure 1, **3j** was obtained as yellow liquid (14.5 mg, 33% yield, *E*/*Z* = 93:7).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.52 (d, *J* = 4.3 Hz, 1H), 8.26 (s,1H), 8.23 (d, *J* = 7.8 Hz, 1H), 7.85 (td, *J*

= 7.7, 1.6 Hz, 1H), 7.42 – 7.37 (m, 2H), 7.24 – 7.20 (m, 2H), 7.13 – 7.10 (m, 1H), 5.77 – 5.69 (m, 1H), 5.50 (q, *J* = 6.8 Hz, 1H), 5.03 – 4.95 (m, 2H), 4.64 (d, *J* = 5.9 Hz, 2H), 3.16 (d, *J* = 6.7 Hz, 2H), 1.80 (d, *J* = 6.8 Hz, 3H).

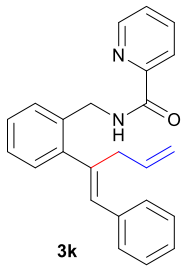
^{13}C NMR (125 MHz, $CDCl_3$)

δ 164.05, 149.94, 148.06, 144.04, 137.53, 137.33, 135.20, 134.95, 129.35, 128.63, 127.06, 127.02, 126.10, 125.92, 122.29, 115.80, 41.37, 36.85, 13.82.

HRMS (ESI) for $C_{19}H_{20}N_2O$ $[M+H]^+$: 293.1648 found: 293.1649.

FTIR (KBr, cm^{-1})

3851.40, 3742.06, 3444.60, 2954.21, 2830.16, 2721.50, 2354.21, 2321.71, 1606.62, 1362.60, 1224.30, 1184.70, 1068.76, 774.32, 646.73, 615.89, 573.83.



3k
(*E*/*Z* > 99:1)

(*E*)-*N*-(2-(1-phenylpenta-1,4-dien-2-yl)benzyl)picolinamide (3k)

Following the general procedure 1, **3k** was obtained as a yellow oil (12.2 mg, 23% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.50 (ddd, *J* = 4.8, 1.6, 0.9 Hz, 1H), 8.34 (s, 1H), 8.22 – 8.20 (m, 1H), 7.85 – 7.82 (m, 1H), 7.47 – 7.44 (m, 1H), 7.42 – 7.39 (m, 1H), 7.37 – 7.34 (m, 4H), 7.29 (dd, *J* = 5.7, 3.4 Hz, 2H), 7.26 – 7.24 (m, 2H), 6.5 (s, 1H), 5.83 – 5.75 (m, 1H), 5.06 – 5.01 (m, 2H), 4.74 (d, *J* = 5.9 Hz, 2H), 3.39 (dd, *J* = 6.7, 1.0 Hz, 2H).

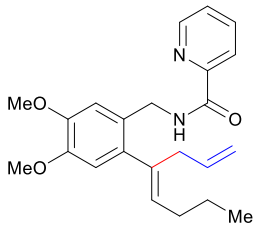
^{13}C NMR (125 MHz, $CDCl_3$)

δ 13C NMR (126 MHz, CDCl3) δ 164.09, 149.88, 148.04, 143.35, 139.63, 137.33, 137.14, 135.32, 134.91, 131.21, 129.28, 128.94, 128.83, 128.29, 127.47, 127.19, 126.93, 126.14, 122.31, 116.82, 41.39, 37.83.

HRMS (ESI) for $C_{24}H_{22}N_2O$ $[M+H]^+$: 355.1805, found: 355.1807.

FTIR (KBr, cm^{-1})

3848.60, 3747.66, 3440.71, 2965.42, 2830.31, 2715.89, 2351.40, 1606.68, 1362.52, 1229.91, 1179.44, 1068.74, 774.29, 646.73, 618.69, 559.81.



3l

(*E*/*Z* > 99:1)

(*E*)-*N*-(4,5-dimethoxy-2-(octa-1,4-dien-4-yl)benzyl) picolin amide (3l)

Following the general procedure 1, **3l** was obtained as bro wn oil (32.5 mg, 57% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (d, *J* = 4.2 Hz, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 8.19 (s, 1H), 7.85 (td, *J* = 7.7, 1.6 Hz, 1H), 7.41 (ddd, *J* = 7.5, 4.8, 1.0 Hz, 1 H), 6.91 (s, 1H), 6.64 (s, 1H), 5.78 – 5.70 (m, 1H), 5.42 (t, *J* = 7. 2 Hz, 1H), 5. 03 – 4.98 (m,2H), 4.57 (d, *J* = 5.8 Hz, 2H), 3.85 (d, *J* = 5.8 Hz, 6H), 3.14 (d, *J* = 6.8 Hz, 2H), 2.22 – 2.17 (m, 2H), 1.51 – 1.43 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

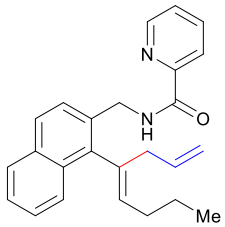
^{13}C NMR (125 MHz, $CDCl_3$)

δ 162.91, 148.96, 147.01, 146.73, 146.72, 136.26, 135.76, 135.50, 13 4.47, 130.92, 126.44, 125.03, 121.21, 114.84, 111.28, 111.15, 54.93, 54.92, 40.20, 36.40, 29.33, 21.79, 12.96.

HRMS (ESI) for $C_{23}H_{28}N_2O_3$ $[M+H]^+$: 381.2173, found: 381.2173.

FTIR (KBr, cm^{-1})

3851.40, 3739.25, 3450.47, 2959.81, 2828.04, 2721.50, 2357.01, 1606. 09, 1362. 38, 1226.33, 1173.83, 1072.60, 778.50, 649.53, 610.28, 55 9.81.



3m

(*E*/*Z* > 99:1)

(*E*)-*N*-((1-(octa-1,4-dien-4-yl)naphthalen-2-yl)methyl) picolinamide (3m)

Following the general procedure 1, **3m** was obtained as a ye llow liquid (32.8 mg, 59% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.48 (d, *J* = 4.7 Hz, 1H), 8.27 (s, 1H), 8.24 (d, *J* = 7.9 Hz, 1 H), 7.90 – 7.88 (m, 1H), 7.85 – 7.82 (m, 1H), 7.81 – 7.78 (m, 1 H), 7.73 (d, *J* = 8.5 Hz, 1H), 7.54 (d, *J* = 8.5 Hz, 1H), 7.48 – 7. 43 (m, 2H), 7.40 – 7.37 (m, 1H), 5.78 – 5.69 (m, 1H), 5.54 (t, *J* =7.3 Hz, 1H), 4.95 – 4.91 (m, 2H), 4.78 (t, *J*=6.3 Hz, 2H), 3.43 (dd, *J* = 14.2, 6.8 Hz, 1H), 3.14 (dd, *J* = 14.4, 7.7 Hz, 1H), 2.40 – 2.36 (m, 2H),1.59 – 1.52 (m, 2H), 1.02 (t, *J* = 7.4 Hz, 3H).

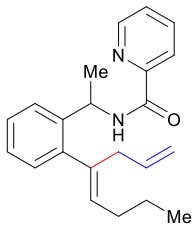
^{13}C NMR (125 MHz, $CDCl_3$)

δ 164.88, 144.14, 141.42, 136.69, 135.21, 134.56, 130.67, 129.59, 128.70, 128.59, 128.23, 127.49, 126.92, 126.72, 120.08, 35.80, 20.6 9, 19.71, 19.26.

HRMS (ESI) for $C_{25}H_{26}N_2O$ $[M+H]^+$: 371.2118, found: 371.2119.

FTIR (KBr, cm^{-1})

3848.60, 3744.86, 3425.23, 2954.21, 2833.64, 2721.50, 2357.01, 16 06.57, 1362.41, 1232.71, 1182.24, 1067.29, 775.70, 652.34, 610.28, 565.42.



3n

(*E*/*Z* > 99:1)

(*E*)-*N*-(1-(2-(octa-1,4-dien-4-yl)phenyl)ethyl)picolinamide (3n)

Following the general procedure 1, **3n** was obtained as a yello w oil (33.1 mg, 66% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.54 – 8.52 (m, 1H), 8.32 (dd, *J* = 45.0, 7.5 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 7.82 (td, *J* = 7.7 Hz, 1.7 Hz, 1H), 7.45 (dd, *J* = 7. 8, 1.0 Hz, 1H), 7.41 – 7.38 (m, 1H), 7.28 (dt, *J* = 5.8, 2.9 Hz, 1H), 7.19 (td, *J* = 7.5, 1.3 Hz, 1H), 7.08 (dd, *J* = 7.6, 1.3 Hz, 1H), 5.7 9 – 5.71 (m, 1H), 5.43 – 5.37 (m, 2H), 5.00 – 4.89 (m, 2H), 3.24 (ddd, *J* = 37.8, 14.8, 6.7 Hz, 2H), 2.24 – 2.16 (m, 2H), 1.54 (d, *J*

= 6.8 Hz, 3H), 1.48 – 1.43 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

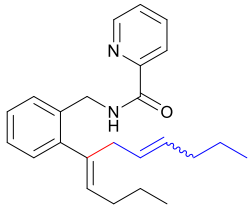
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.04, 150.10, 147.96, 143.28, 140.97, 137.28, 136.96, 135.61, 13 1.87, 129.77, 127.21, 126.64, 126.02, 125.08, 122.15, 115.46, 46.32, 37.17, 30.30, 23.11, 22.81, 13.95.

HRMS (ESI) for $C_{22}H_{26}N_2O$ $[M+H]^+$: 335.2118, found: 335.2117.

FTIR (KBr, cm^{-1})

3851.40, 3742.06, 3430.84, 2959.81, 2830.84, 2727.10, 2357.01, 1606. 79, 1362.05, 1221.50, 1182.24, 1067.29, 772.90, 649.53, 610.28, 571. 03.



3o
(*E*/*Z* = 65:35)

***N*-(2-((4*E*)-undeca-4,7-dien-5-yl)benzyl)picolinamide (3o)**

Following the general procedure 1, **3o** was obtained as a y ellow liquid (38.1 mg, 70% yield, *E*/*Z* = 65:35).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (d, *J* = 4.6 Hz, 1H), 8.22 (d, *J* = 7.8 Hz, 2H), 7.84 (td, *J* = 7.7, 1.7 Hz, 1H), 7.42 – 7.36 (m, 2H), 7.23 – 7.19 (m, 2H), 7.14 – 7.10 (m, 1H), 5.39 – 5.26 (m, 3H), 4.64 (t, *J* = 6.3 Hz, 2 H), 3.15 (d, *J* = 7.1 Hz, 2H), 2.23 – 2.17 (m, 2H), 1.87 – 1.83 (m, 2H), 1.50 – 1.42 (m, 2H), 1.31 – 1.26 (m, 1H), 1.21 – 1.16 (m, 1H), 0.97 – 0.93 (m, 3H), 0.77 (t, *J* = 6.7 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$)

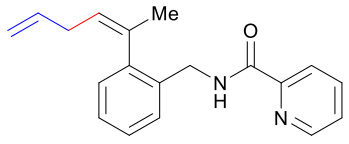
δ 163.98, 149.99, 148.01, 144.11, 137.79, 137.28, 135.31, 135.21, 131.96, 130.87, 129.40, 128.74, 127.00, 126.93, 126.05, 122.25, 41. 32, 35.95, 34.54, 30.76, 30.32, 29.26, 22.59, 13.97.

HRMS (ESI) for $C_{24}H_{30}N_2O$ $[M+H]^+$: 363.2431, found: 363.2433.

FTIR (KBr, cm^{-1})

3853.03, 3740.57, 3447.66, 2951.40, 2830.84, 2724.30, 2354.21, 16

11.21, 1370.09, 1207.48, 1086.92, 777.94, 630.60, 598.58, 537.72.



5a

(*E*/*Z* > 99:1)

(*Z*)-*N*-(2-(hexa-2,5-dien-2-yl)benzyl)picolinamide (5a)

Following the general procedure 1, **5a** was obtained as a yellow oil (33.3 mg, 76% yield, *E*/*Z*>99:1).

^{1}H NMR (500 MHz, Chloroform-d)

δ 8.52 (dt, *J* = 4.7, 1.6, 0.9 Hz, 1H), 8.27 (s, 1H), 8.23 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.85 (td, *J* = 7.7, 1.7 Hz, 1H), 7.45 – 7.39 (m, 2H), 7.29 – 7.23 (m, 2H), 7.08 – 7.04 (m, 1H), 5.79 – 5.70 (m, 1H), 5.61 (tq, *J* = 7.4, 1.6 Hz, 1H), 4.99 – 4.91 (m, 2H), 4.69 – 4.49 (m, 2H), 2.55 – 2.49 (m, 2H), 2.03 (s, 3H).

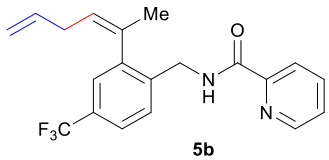
^{13}C NMR (126 MHz, Chloroform-d)

δ 164.11 , 149.87 , 148.05 , 141.43 , 137.34 , 136.89 , 136.58 , 135.05 , 128.63 , 128.53 , 127.62 , 127.27 , 126.13 , 125.96 , 122.29 , 114.67 , 41.01 , 33.61 , 25.88 .

HRMS (ESI) for $C_{19}H_{20}N_2O$ $[M+H]^+$: 293.1648, found: 293.1651.

FTIR (KBr, cm^{-1})

3436.52, 2957.01, 2830.84, 2715.89, 2354.21, 1600.00, 1367.29, 1199.07, 1072.90, 775.70.



5b

(*E*/*Z* > 99:1)

(*Z*)-*N*-(2-(hexa-2,5-dien-2-yl)-4-(trifluoromethyl)benzyl) picolinamide (5b)

Following the general procedure 1, **5b** was obtained as a yellow oil (18.2 mg, 51% yield, *E*/*Z*>99:1).

^{1}H NMR (400 MHz, Chloroform-d)

δ 8.54 (d, *J* = 4.7 Hz, 1H), 8.37 (s, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 7.87 (td, *J* = 7.7, 1.7 Hz, 1H), 7.59 – 7.45 (m, 2H), 7.45 (m, *J* = 7.6, 4.8, 1.3 Hz, 1H), 7.32 (d, *J* = 1.8 Hz, 1H), 5.81 – 5.72 (m, 1H), 5.71 – 5.65 (t, 1H), 4.99 – 4.91 (dt, 2H), 4.74 – 4.52 (m, 2H), 2.50 (q, *J* = 7.4 Hz, 2H), 2.05 (s, 3H).

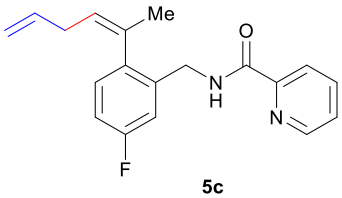
^{13}C NMR (101 MHz, Chloroform-d)

δ 164.35 , 149.55 , 148.13 , 141.84 , 141.72 (d, J_{C-F} = 1.8 Hz), 139.38 , 137.46 , 136.30 , 135.30 , 128.67 , 127.03 , 126.37 , 125.47 (q, J_{C-F} = 3.8 Hz), 124.12 (q, J_{C-F} = 3.6 Hz), 124.06 (d, J_{C-F} = 272.6 Hz), 122.36 , 115.04 , 40.56 , 33.60 , 25.54 .19F NMR (471 MHz, Chloroform-d) δ -62.49.

HRMS (ESI) for $C_{20}H_{19}F_3N_2O$ $[M+H]^+$: 361.1522, found: 361.1523.

FTIR (KBr, cm^{-1})

3449.57, 2957.01, 2830.84, 2721.50, 2351.40, 1605.61, 1367.29, 1210.28, 1072.90, 775.70.



5c

(*E*/*Z* = 93:7)

(*Z*)-*N*-(5-fluoro-2-(hexa-2,5-dien-2-yl)benzyl)picolinamide (5c)

Following the general procedure 1, **5c** was obtained as a yellow oil (19.0 mg, 61% yield, *E*/*Z* = 93:7).

^{1}H NMR (400 MHz, Chloroform-d)

δ 8.55 (dt, *J* = 4.7, 1.3 Hz, 1H), 8.33 (s, 1H), 8.23 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.87 (td, 1H), 7.47 – 7.41 (m, 1H), 7.12 (dd, *J* = 9.8, 2.6 Hz, 1H), 7.01 (td, *J* = 8.4, 5.9 Hz, 1H), 6.98 – 6.92 (m, 1H), 5.80 – 5.68 (m, 1H), 5.63 (td, 1H), 4.99 – 4.92 (m, 2H), 4.67 – 4.46 (m, 2H), 2.54 – 2.47 (m, 2H), 2.03 – 1.99 (m, 3H).

^{13}C NMR (101 MHz, Chloroform-d)

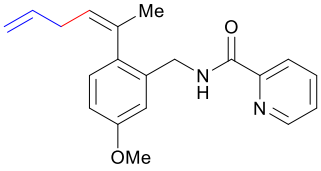
δ 164.29 , 161.93 (d, J_{C-F} = 245.4 Hz), 149.63 , 148.12 , 137.54 (d, J_{C-F} = 7.1 Hz), 137.43 , 136.87 (d, J_{C-F} = 3.4 Hz), 136.68 , 135.69 , 130.10 (d, J_{C-F} = 7.9 Hz), 126.61 , 126.31 , 122.36 , 114.85 (d, J_{C-F} = 21.9 Hz), 114.79 ,114.40 (d, J_{C-F} = 21.0 Hz), 40.64 , 33.55 , 25.86 .

^{19}F NMR (376 MHz, Chloroform-d) δ -100.01.

HRMS (ESI) for $C_{19}H_{19}N_2O$ $[M+H]^+$: 311.1554, found: 311.1558.

FTIR (KBr, cm^{-1})

3452.17, 2957.01, 2830.84, 2713.08, 2354.21, 1600.00, 1358.88, 1204.67, 1067.29, 772.90, 604.67.



5d
(*E*/*Z* = 94:6)

(*Z*)-*N*-(2-(hexa-2,5-dien-2-yl)-5-methoxybenzyl)picolinamide (5d)

Following the general procedure 1, **5d** was obtained as a yellow oil (19.5 mg, 61% yield, *E*/*Z* = 94:6).

^{1}H NMR (400 MHz, Chloroform-d)

δ 8.52 (d, *J* = 3.8 Hz, 1H), 8.27 (s, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 7.85 (td, *J* = 7.7, 1.7 Hz, 1H), 7.46 – 7.38 (m, 1H), 7.01 – 6.95 (m, 2H), 6.82 (dd, *J* = 8.3, 2.7 Hz, 1H), 5.80 – 5.69 (m, 1H), 5.59 (td, *J* = 7.5, 1.6 Hz, 1H), 4.99 – 4.90 (m, 2H), 4.66 – 4.45 (m, 2H), 3.79 (s, 3H), 2.52 (d, *J* = 6.2 Hz, 2H), 2.00 (s, 3H).

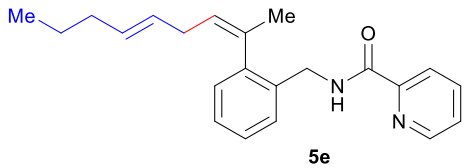
^{13}C NMR (101 MHz, Chloroform-d)

δ 164.13 , 158.67 , 149.81 , 148.07 , 137.34 , 137.00 , 136.43 , 136.28 , 133.64 , 129.68 , 126.28 , 126.16 , 122.29 , 114.61 , 113.86 , 113.01 , 55.27 , 41.13 , 33.63 , 26.12.

HRMS (ESI) for $C_{20}H_{22}N_2O_2$ $[M+H]^+$: 323.1754, found: 323.1758.

FTIR (KBr, cm^{-1})

3453.27, 2962.62, 2830.84, 2713.08, 2348.60, 1600.00, 1356.07, 1207.48, 1067.29, 772.90, 613.08.



5e
(*E*/*Z* = 64:36)

***N*-(2-((*2Z*,5E)-nona-2,5-dien-2-yl)benzyl)picolinamide (5e)**

Following the general procedure 1, **5e** was obtained as a yellow oil (27.7 mg, 83% yield, *E*/*Z* = 64:36).

^{1}H NMR (400 MHz, Chloroform-d)

δ 8.51 (s, 1H), 8.27 (s, 1H), 8.21 (s, 1H), 7.85 (t, *J* = 7.7 Hz, 1H), 7.45 –

7.37 (m, 2H), 7.26 (tq, *J* = 3.9, 2.1 Hz, 2H), 7.10 – 7.02 (m, 1H), 5.56 – 5.49 (m, 1H), 5.35 – 5.26 (m, 2H), 4.66 (dd, *J* = 14.7, 6.2 Hz, 1H), 4.52 (dd, *J* = 13.5, 6.9 Hz, 1H), 2.50 – 2.37 (m, 2H), 2.01 (s, 3H), 1.93 – 1.88 (m, 1H), 1.84 – 1.74 (m, 1H), 1.32 – 1.21 (m, 2H), 0.81 (t, *J* = 7.4 Hz, 3H).

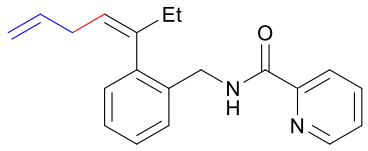
^{13}C NMR (101 MHz, Chloroform-d)

δ 164.09 , 148.05 , 141.62 , 137.35 , 135.65 , 135.06 , 130.75 , 130.08 , 128.72 , 128.60 , 127.66 , 127.46 , 127.23 , 127.12 , 126.14 , 122.30 , 41.10 , 34.67 , 29.14 , 25.84 , 22.73 , 13.75.

HRMS (ESI) for $C_{22}H_{26}N_2O$ $[M+H]^+$: 335.2118, found: 335.2115.

FTIR (KBr, cm^{-1})

3431.30, 2954.21, 2828.04, 2713.08, 1600.00, 1364.49, 1207.48, 1070.09, 775.70.



5f

(*E*/*Z* > 99:1)

(Z)-N-(2-(hepta-3,6-dien-3-yl)benzyl)picolinamide

Following the general procedure 1, **5f** was obtained as a yellow oil (34.0 mg, 74% yield, *E*/*Z* > 99:1).

^{1}H NMR (500 MHz, Chloroform-*d*)

δ 8.53 (dt, *J* = 4.6, 1.3 Hz, 1H), 8.26 (s, 1H), 8.23 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.85 (td, *J* = 7.7, 1.7 Hz, 1H), 7.45 – 7.40 (m, 2H), 7.28 – 7.26 (m, 2H), 7.05 – 7.02 (m, 1H), 5.76 (dd, *J* = 16.9, 10.4 Hz, 1H), 5.58 (t, *J* = 7.3 Hz, 1H), 4.99 – 4.91 (m, 2H), 4.58 (dd, *J* = 46.3, 6.0 Hz, 1H), 2.57 – 2.50 (m, 2H), 2.33 (m, *J* = 19.3, 7.5, 1.4 Hz, 1H), 1.05 (t, *J* = 7.4 Hz, 3H).

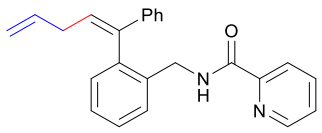
^{13}C NMR (126 MHz, Chloroform-*d*)

δ 163.10 , 148.88 , 147.01 , 141.36 , 139.64 , 136.28 , 136.01 , 134.33 , 128.17 , 127.30 , 126.24 , 125.08 , 123.10 , 121.25 , 113.57 , 39.89 , 32.38 , 30.99 , 11.58 , 0.00 .

HRMS (ESI) for $C_{22}H_{26}N_2O$ $[M+H]^+$: 307.1805, found: 307.1808.

FTIR (KBr, cm^{-1})

3444.04, 2825.76, 1597.20, 1372.90, 1088.09, 772.90.



5g

(*E*/*Z* > 99:1)

(Z)-N-(2-(1-phenylpenta-1,4-dien-1-yl)benzyl)picolinamide Following the general procedure 1, **5g** was obtained as a yellow oil (34.5 mg, 65% yield, *E*/*Z* > 99:1).

^{1}H NMR (500 MHz, Chloroform-*d*)

δ 8.45 (dt, *J* = 4.9, 1.7, 0.9 Hz, 1H), 8.14 (dt, *J* = 7.7, 1.1 Hz, 1H), 8.03 (s, 0H), 7.80 (td, *J* = 7.7, 1.7 Hz, 1H), 7.50 (dd, *J* = 5.5, 3.6 Hz, 1H), 7.40 – 7.33 (m, 3H), 7.26 (td, *J* = 6.8, 1.4 Hz, 4H), 7.23 – 7.16 (m, 2H), 6.34 (t, *J* = 7.5 Hz, 1H), 5.84 (ddt, *J* = 16.5, 10.1, 6.2 Hz, 1H), 5.07 – 4.98 (m, 2H), 4.42 (dd, *J* = 14.4, 6.1 Hz, 2H), 2.75 (td, *J* = 7.6, 4.6, 1.6 Hz, 2H).

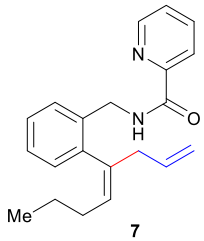
^{13}C NMR (126 MHz, Chloroform-*d*)

δ 163.92 , 149.82 , 147.88 , 140.85 , 140.72 , 138.68 , 137.18 , 136.54 , 136.38 , 130.54 , 129.14 , 128.46 , 127.97 , 127.59 , 127.58 , 127.19 , 126.21 , 125.97 , 122.15 , 115.30 , 41.27 , 34.23 .

HRMS (ESI) for $C_{21}H_{24}N_2O$ $[M+H]^+$: 355.1805, found: 355.1804.

FTIR (KBr, cm^{-1})

3472.90, 2839.25, 1597.20, 1361.68, 1215.89, 1072.90, 781.31.



7

(*E*/*Z* = 7:93)

(*Z*)-*N*-(2-(octa-1,4-dien-4-yl)benzyl)picolinamide (7)

Following the general procedure 1, **7** was obtained as a yellow oil (42.3 mg, 66% yield, *E*/*Z* = 7:93).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.51 (ddd, *J* = 4.7, 1.5, 0.9 Hz, 1H), 8.22 (dd, *J* = 9.8, 9.0 Hz, 2H), 7.84 (td, *J* = 7.7, 1.7 Hz, 1H), 7.43 – 7.39 (m, 2H), 7.26 – 7.24 (m, 2H), 7.04 – 7.03 (m, 1H), 5.87 – 5.79 (m, 1H), 5.59 – 5.56 (m, 1H), 5.03 – 4.96 (m, 2H), 4.58 (dd, *J* = 8.5, 6.0 Hz, 2H), 3.07 – 2.97 (m, 2H), 1.84 – 1.67 (m,

2H), 1.37 – 1.28 (m, 2H), 0.80 (t, *J* = 7.4 Hz 3H).

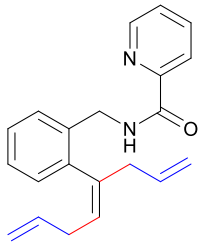
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.04, 148.89, 146.99, 139.72, 136.82, 136.27, 134.92, 134.35, 128.28, 128.25, 127.42, 126.27, 126.15, 125.06, 121.24, 115.39, 42.73, 39.98, 30.23, 21.62, 12.78.

HRMS (ESI) for $C_{21}H_{24}N_2O$ $[M+H]^+$: 321.1961, found: 321.1960.

FTIR (KBr, cm^{-1})

3846.26, 3742.06, 3436.45, 2957.01, 2830.84, 2718.69, 2351.40, 1606.40, 1362.05, 1232.71, 1179.44, 1070.09, 772. 90, 649.53, 610.28, 571.03.



9

(*Z*)-*N*-(2-(octa-1,4,7-trien-4-yl)benzyl)picolinamide (9)

Following the general procedure 1, **9** was obtained as a yellow liquid (24.4 mg, 51% yield).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.52 (ddd, *J* = 4.7, 1.6, 0.9 Hz, 1H), 8.25 (s, 1H), 8.24 – 8.21 (m, 1H), 7.85 (td, *J* = 7.7, 1.7 Hz, 1H), 7.44 – 7.40 (m, 2H), 7.28 – 7.24 (m, 2H), 7.05 – 7.02 (m, 1H), 5.89 – 5.80 (m, 1H), 5.78 – 5.70 (m, 1H), 5.63 – 5.60 (m, 1H), 5.05 – 4.91 (m, 4H), 4.57 (qt, *J* = 15.1, 7.6 Hz, 2H), 3.10 – 2.99 (m, 2H), 2.59 – 2.48 (m, 2H).

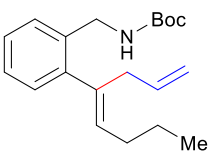
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.09, 148.85, 147.02, 139.16, 138.20, 136.30, 135.67, 134.59, 134.38, 128.15, 127.46, 126.36, 126.35, 125.19, 125.10, 121.27, 115.68, 113.78, 42.61, 39.92, 32.45.

HRMS (ESI) for $C_{21}H_{22}N_2O$ $[M+H]^+$: 319.1805, found: 319.1802.

FTIR (KBr, cm^{-1})

3851.40, 3747.66, 3447.66, 2962.62, 2833.64, 2724.30, 2357.01, 1606.17, 1362.19, 1229.91, 1187.85, 1078.50, 768.33, 652.34, 615.89, 565.42.



11

Tert-butyl(*E*)-(2-(octa-1,4-dien-4-yl)benzyl)carbamate (11)

Following the general procedure 3, **11** was obtained as a yello w oil (49.4 mg, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 7.32 – 7.30 (m, 1H), 7.23 – 7.17 (m, 2H), 7.09 – 7.07 (m, 1H), 5.72 – 5.64 (m, 1H), 5.34 (t, *J* = 7.3 Hz, 1H), 4.99 – 4.93 (m, 2H), 4.70 (s, 1H), 4.29 (d, *J* = 5.4 Hz, 2H), 3.11 (d, *J* = 6.7 Hz, 2H), 2.18 (q, *J* = 7.3 Hz, 2H), 1.45 (s, 9H), 0.96 (t, *J* = 7.4 Hz, 3H).

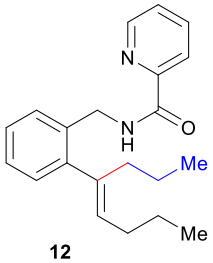
^{13}C NMR (125 MHz, $CDCl_3$)

δ 154.72, 142.61, 135.73, 134.75, 134.31, 130.57, 128.27, 127.26, 125.85, 114.73, 78.20, 41.41, 36.03, 29.25, 27.41, 21.78, 12.93.

HRMS (ESI) for $C_{20}H_{29}NO_2$ $[M+Na]^+$: 338.2091, found: 338.2089.

FTIR (KBr, cm^{-1})

3854.21, 3744.86, 3447.66, 2965.42, 2830.84, 2713.08, 2351.40, 16 06.4, 1361.88, 1229.91, 1185.05, 1075.70, 781.31.



12

(*E*)-*N*-(2-(oct-4-en-4-yl)benzyl)picolinamide (12)

Following the general procedure 1, **12** was obtained as a yel low liquid (24.5 mg, 76% yield).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.50 – 8.49(d, *J* = 4.7 Hz, 1H), 8.27 (s, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 7.82 (td, *J* = 7.7, 1.7 Hz, 1H), 7.41 – 7.38 (m, 2H), 7.2 3 – 7.21 (m, 2H), 7.12 – 7.10 (m, 1H), 6.84 – 6.73 (m, 3H), 5.3 5 (t, *J* = 7.2 Hz, 1H), 4.66 (d, *J* = 5.9 Hz, 2H), 2.38 – 2.35 (m, 2H), 2.39 – 2.34 (m, 2H), 2.17 (q, *J* = 7.3 Hz, 2H), 1.48 – 1.41 (m, 2H), 1.36 – 1.31 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H), 0.90 –

0.87 (m, 3H).

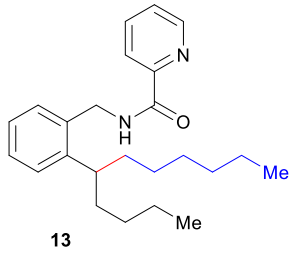
^{13}C NMR (125 MHz, $CDCl_3$)

δ 163.03, 148.93, 147.00, 143.43, 138.46, 136.27, 134.19, 130.02, 128.41, 127.56, 125.97, 125.84, 125.05, 121.23, 40.34, 33.35, 29.20, 21.91, 20.38, 13.18, 12.93.

HRMS (ESI) for $C_{21}H_{26}N_2O$ $[M+H]^+$: 323.2118, found: 323.2119.

FTIR (KBr, cm^{-1})

3854.21, 3742.06, 3433.64, 2955.87, 2827.76, 2724.30, 2362.62, 16 06.67, 1362. 42, 1235.94, 1175.09, 1072.90, 774.49, 638.32, 610.28, 571.03.



13

***N*-(2-(undecan-5-yl)benzyl)picolinamide (13)**

Following the general procedure 1, **13** was obtained as a yellow oil (15.6 mg, 45% yield).

^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.47 (d, *J* = 4.6 Hz, 1H), 8.24 (d, *J* = 7.8 Hz, 1H), 8.13 (s, 1 H), 7.84 (td, *J* = 7.7 Hz, 1.7 Hz, 1H), 7.40 (dd, *J* = 7.4, 4.8 Hz, 1H), 7.33 – 7.29 (m, 2H), 7.26 (d, *J* = 6.0 Hz, 1H), 7.17 (t, *J* = 7.3 Hz, 1H), 4.68 (t, *J* = 5.3 Hz, 2H), 2.87 – 2.82 (m, 1H), 1. 66 – 1.59 (m, 3H), 1.55 – 1.47 (m, 2H), 1.20 – 1.01 (m, 12H), 0. 79 (t, *J* = 7.0 Hz, 3H), 0.73 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$)

δ 162.57, 148.80, 146.98, 144.74, 136.28, 134.49, 128.68, 127.29, 125.07, 124.69, 121.21, 40.85, 36.42, 36.11, 30.71, 29.01, 28.65, 2 6.78, 22.01, 21.92, 21.54, 13.05, 12.96.

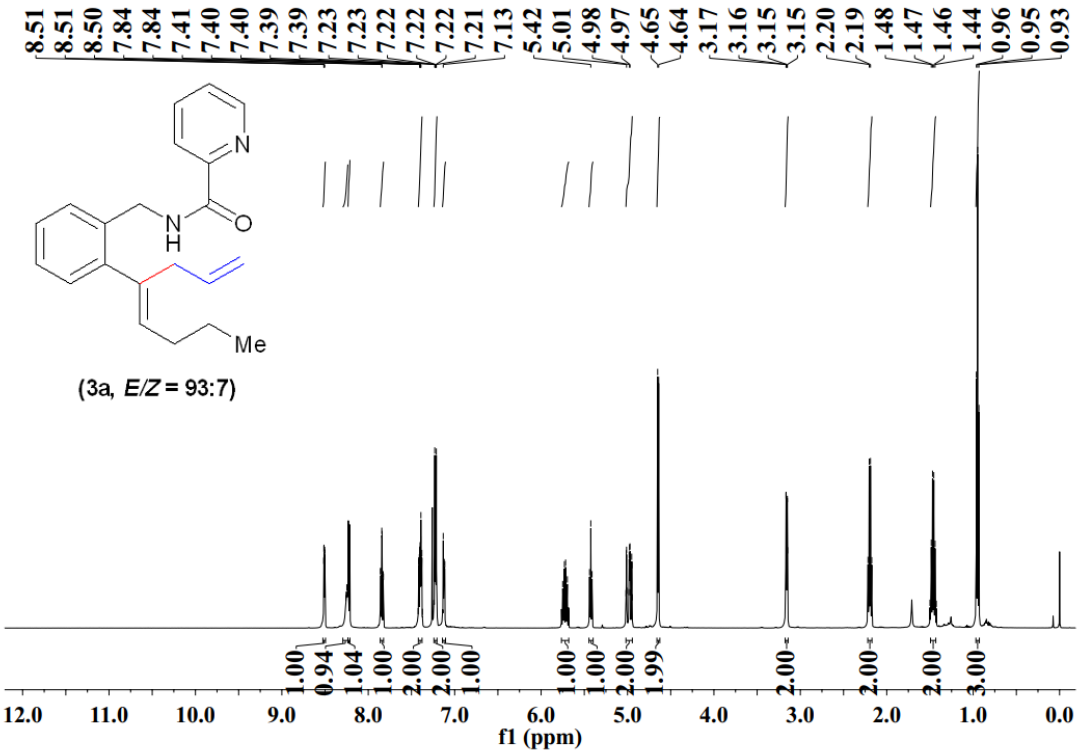
HRMS (ESI) for $C_{24}H_{34}N_2O$ $[M+H]^+$: 367.2744, found: 367.2742.

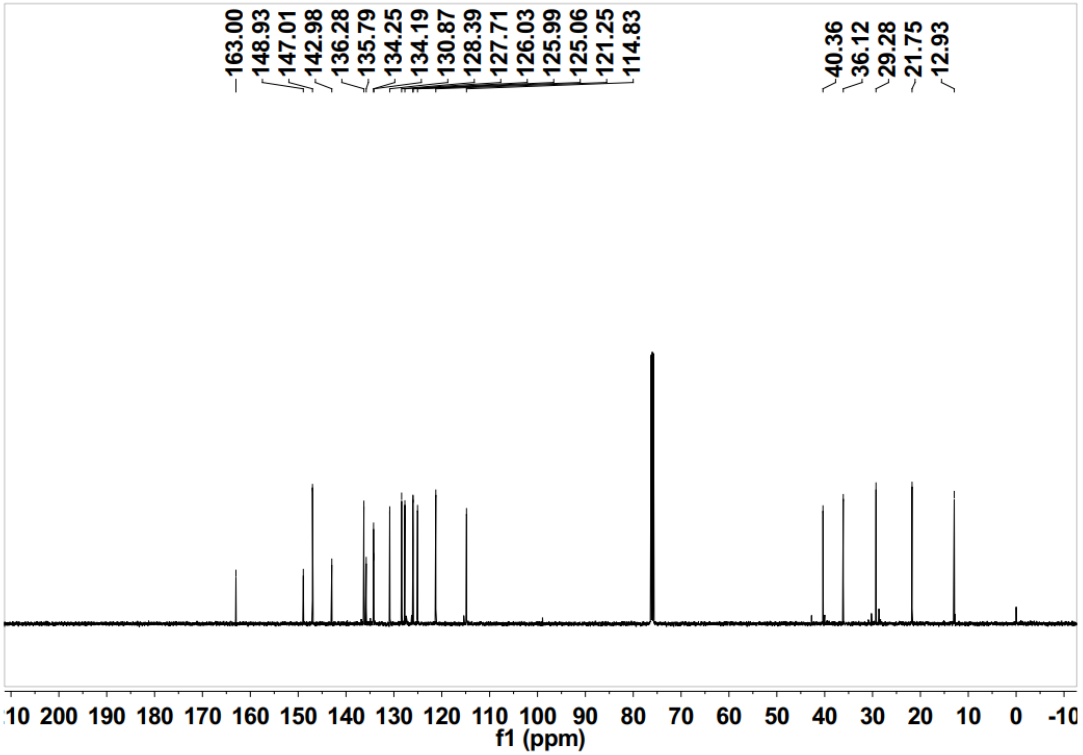
FTIR (KBr, cm^{-1})

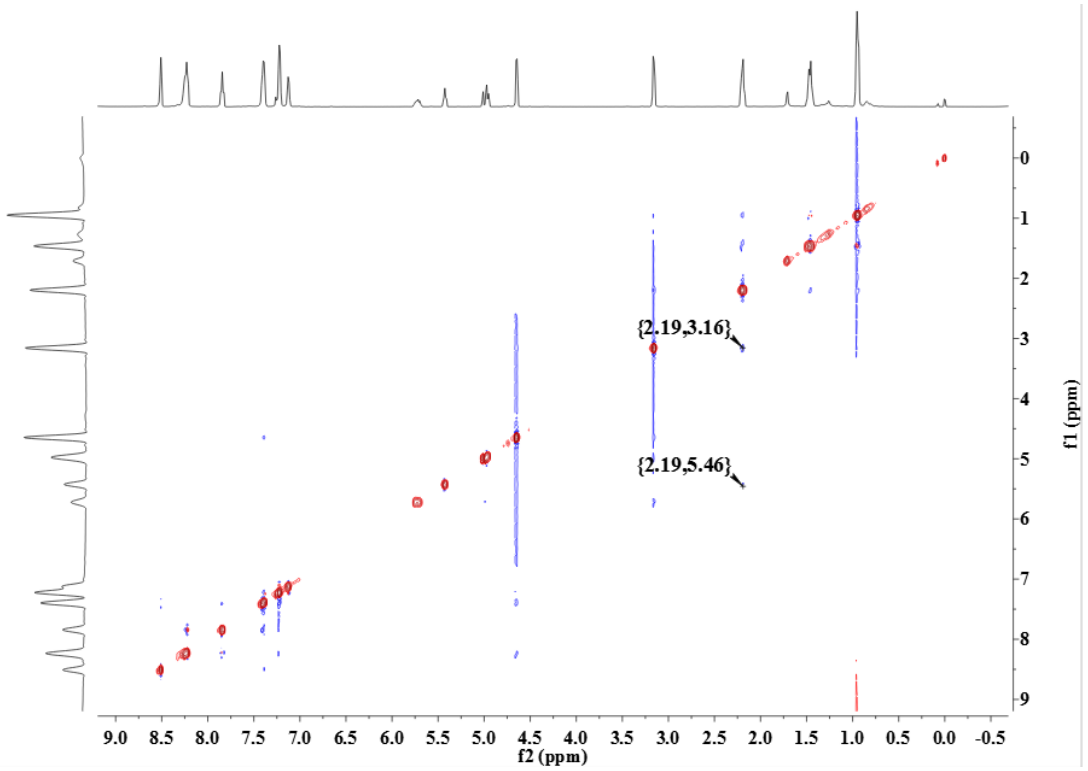
3851.53, 3742.18, 3473.02, 2959.95, 2829.09, 2716.02, 2359.95,

1606.63, 1362.77, 1230.06, 1182.39, 1070.24, 774.17, 644.08, 604.83, 562.77.

12. $^{1}H/^{13}C$ NMR Charts



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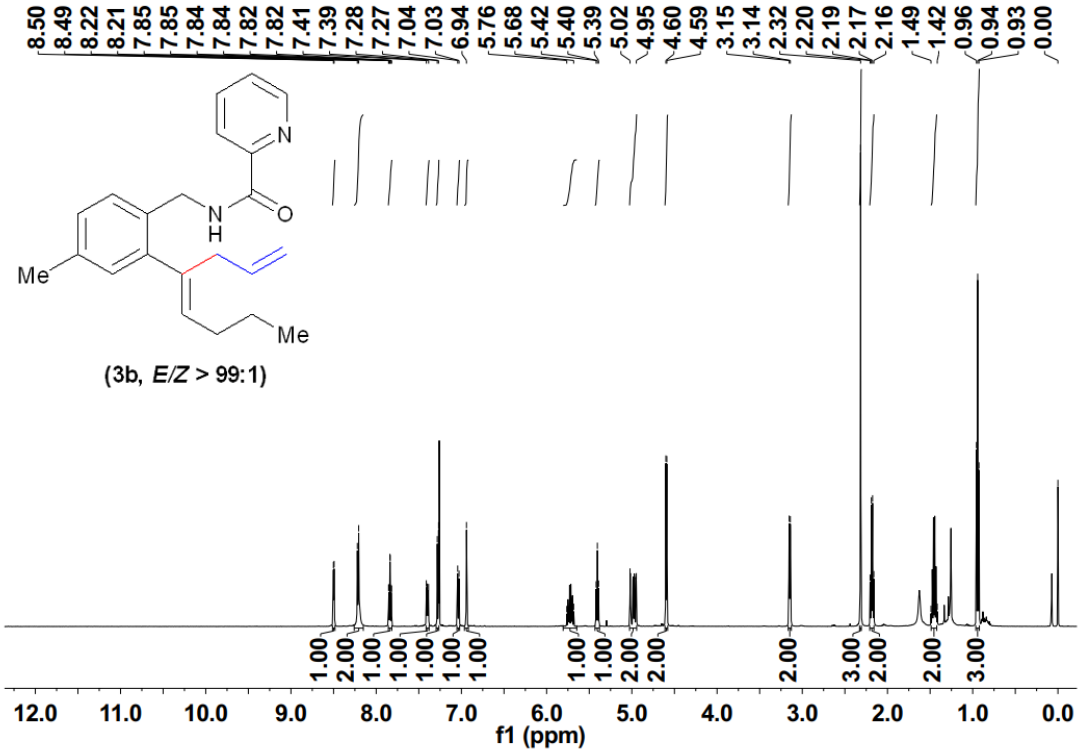
{2.19,5.46}
f2 (ppm)
f1 (ppm)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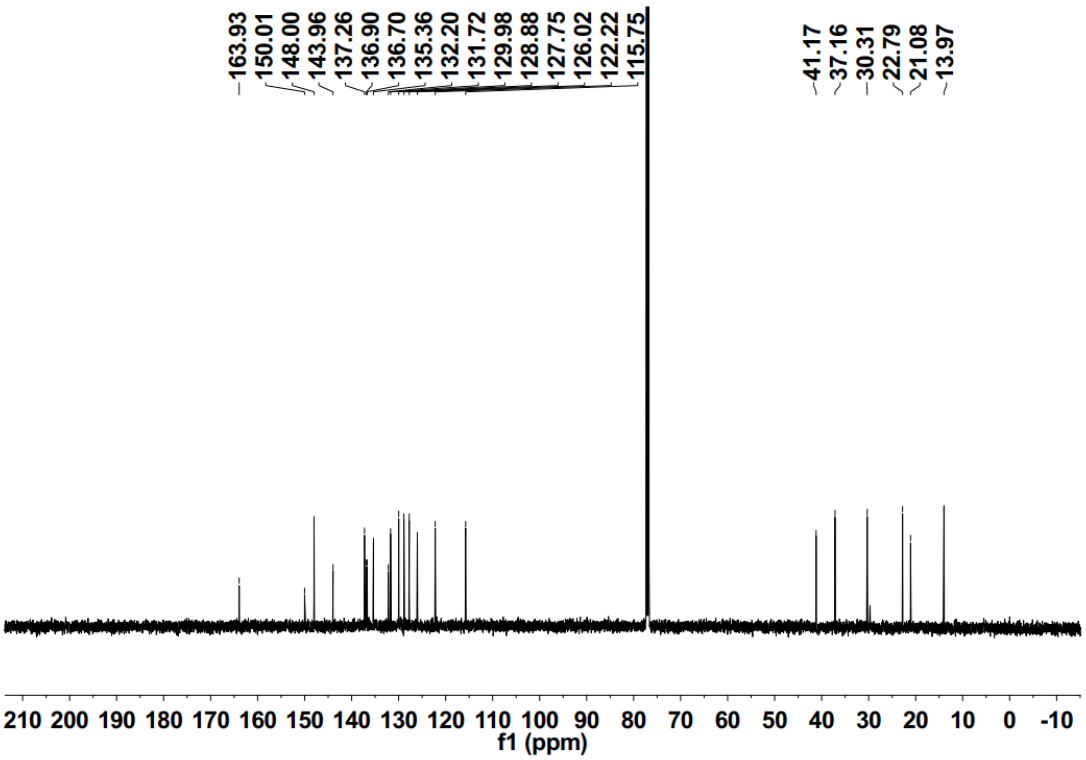
N
N
H
O
Me
Me
1.00 2.00 1.00 1.00 1.00 1.00 1.00 1.00 2.00 2.00 2.00 3.00 2.00 2.00 3.00
f1 (ppm)

(3b, *E*/*Z* > 99:1)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126.02
122.22
115.75
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21.08
13.97
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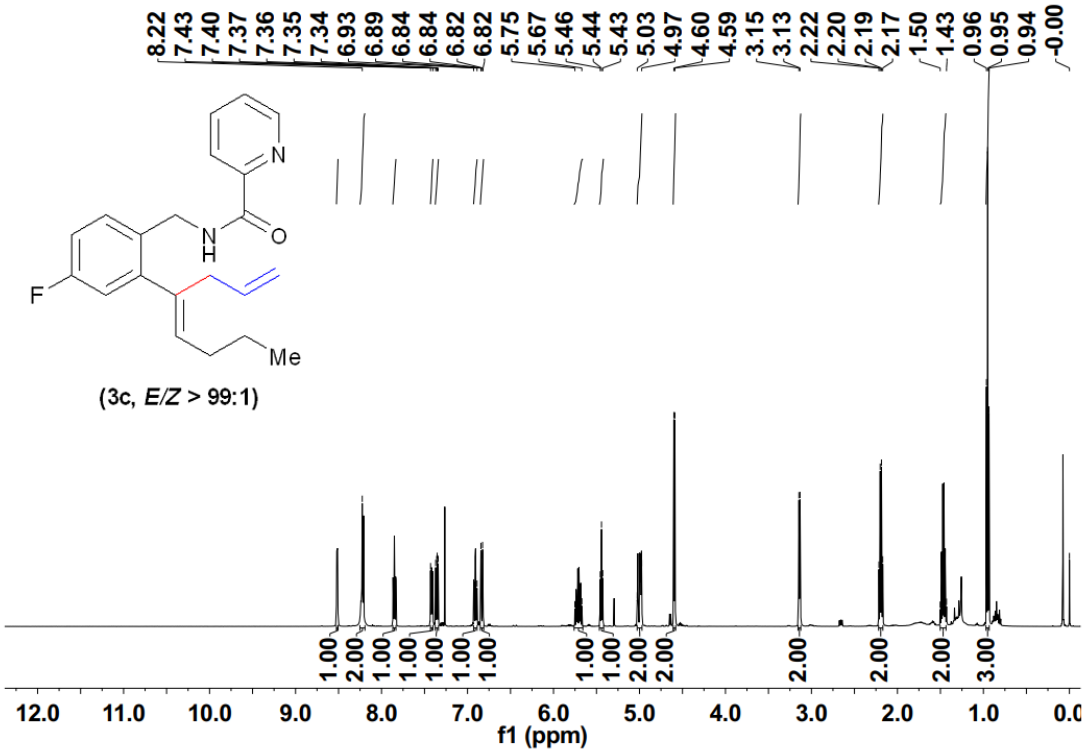
f1 (ppm)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2.00
2.00
2.00
3.00
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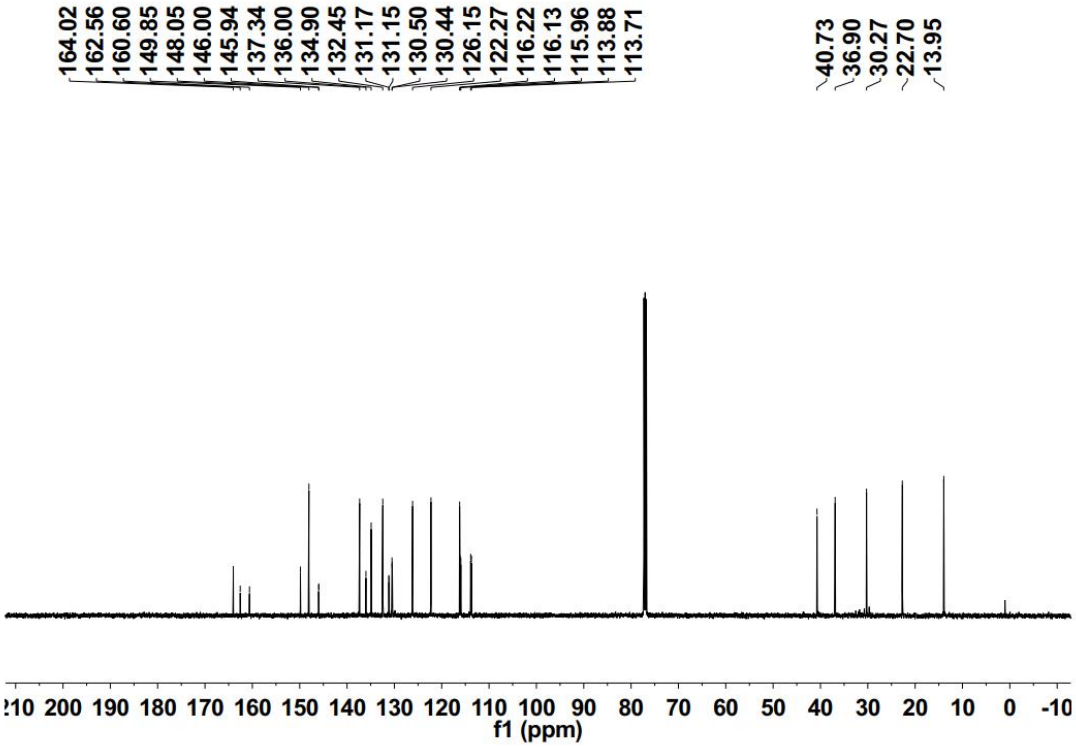
f1 (ppm)

(3c, *E*/*Z* > 99:1)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126.15
122.27
116.22
116.13
115.96
113.88
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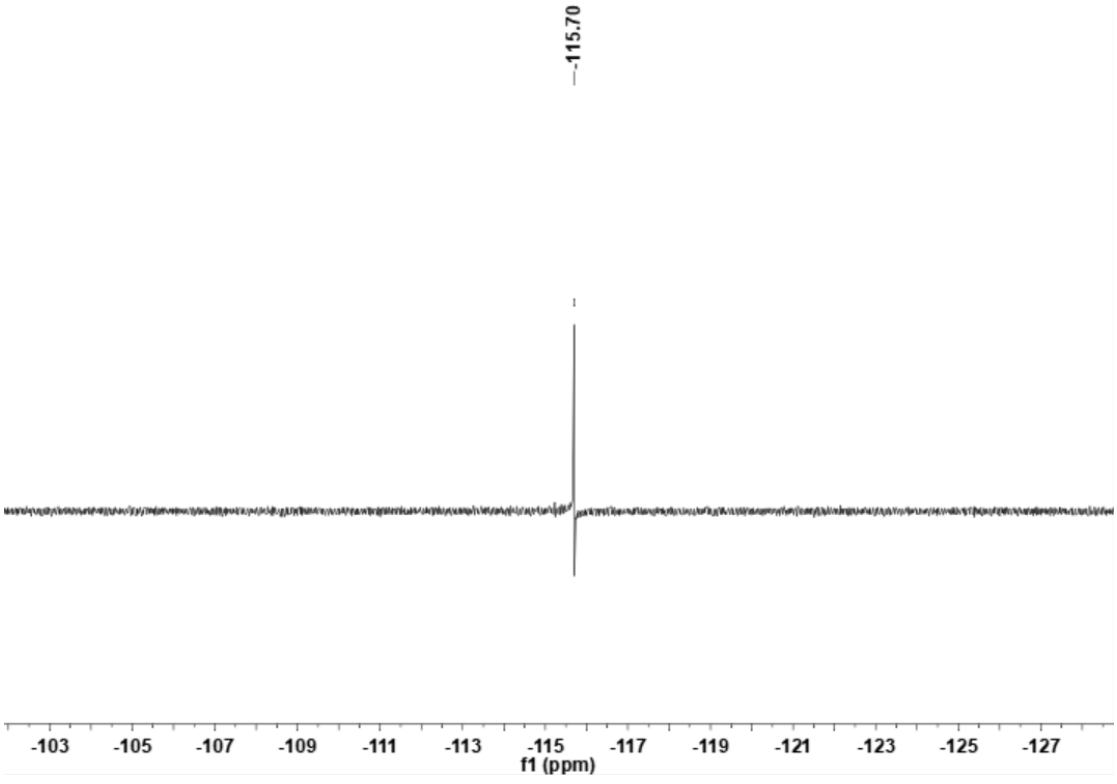
f1 (ppm)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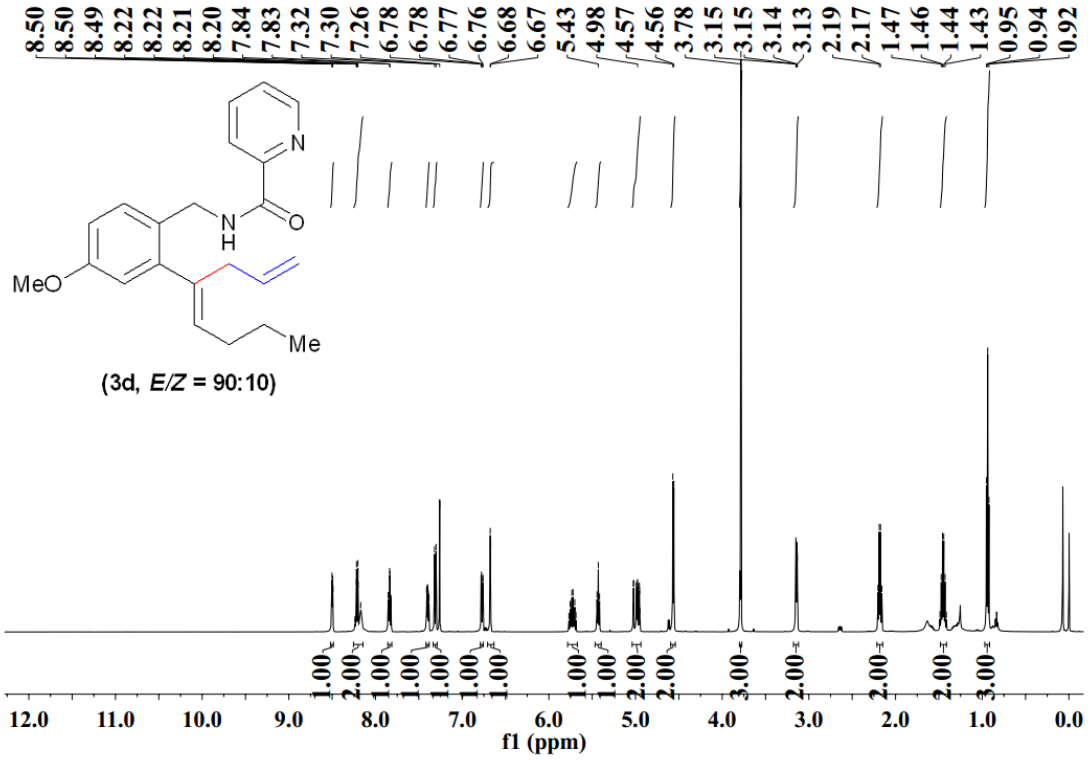
f1 (ppm)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12.0
11.0
10.0
9.0
8.0
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4.0
3.0
2.0
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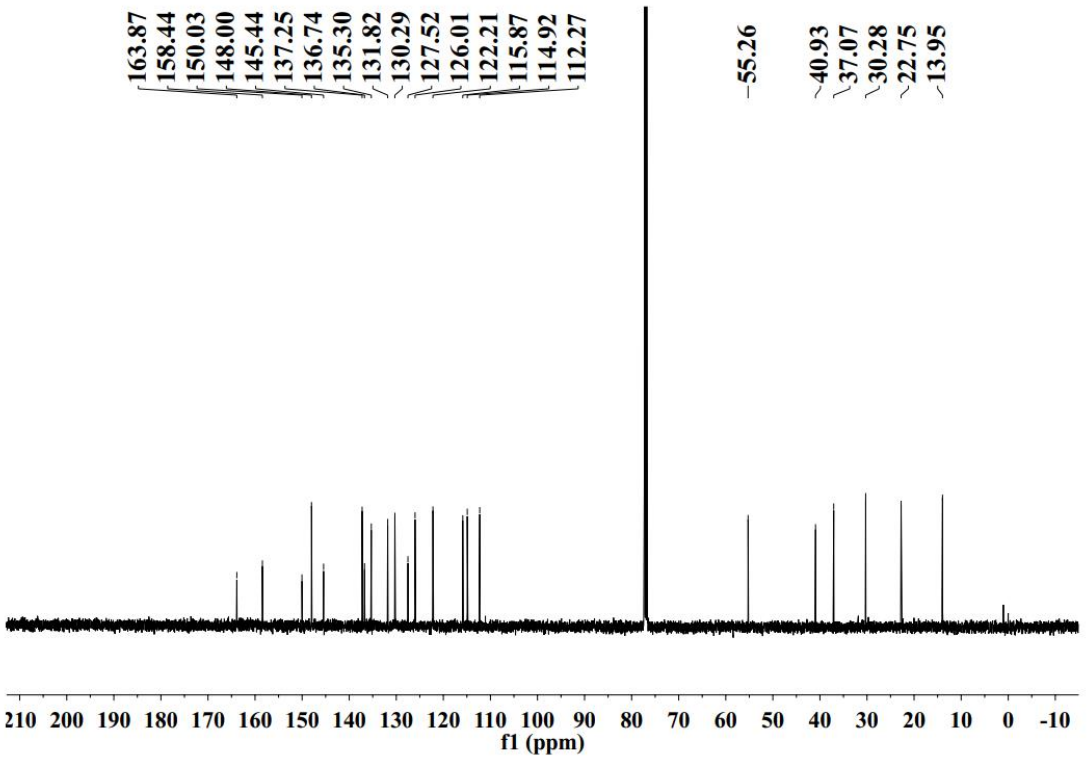
f1 (ppm)

(3d, *E*/*Z* = 90:10)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114.92
112.27
55.26
40.93
37.07
30.28
22.75
13.95
210
200
190
180
170
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100
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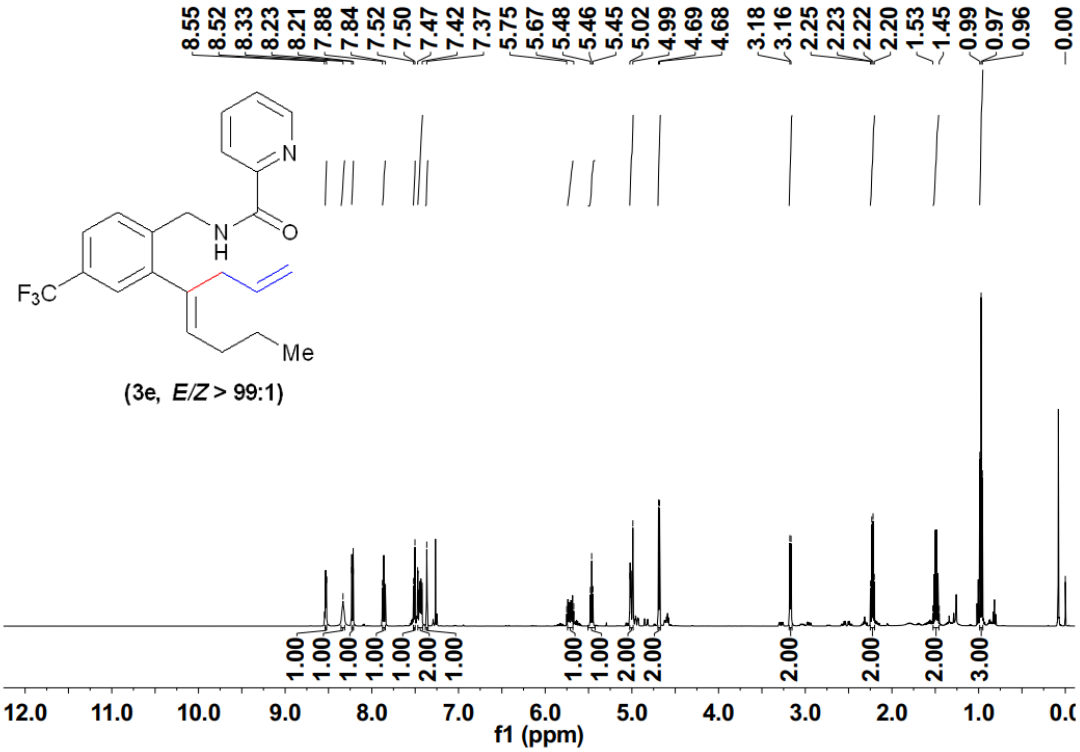
f1 (ppm)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3.00
12.0
11.0
10.0
9.0
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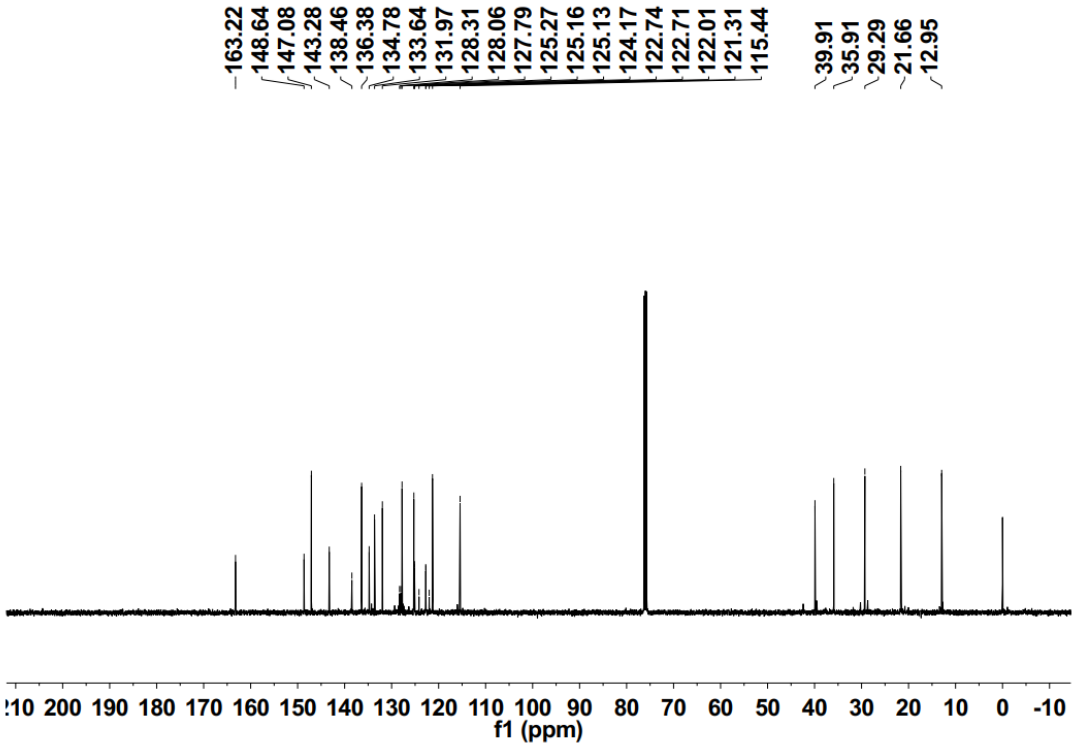
f1 (ppm)

(3e, *E*/*Z* > 99:1)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125.16
125.13
124.17
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122.71
122.01
121.31
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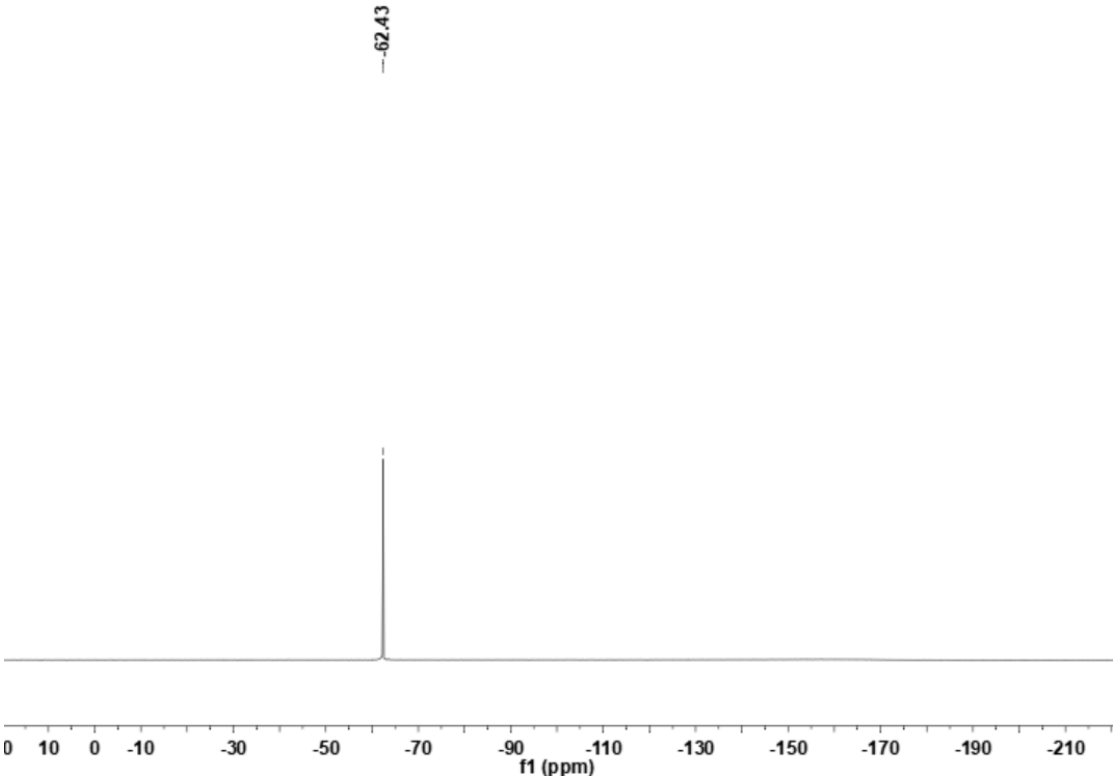
f1 (ppm)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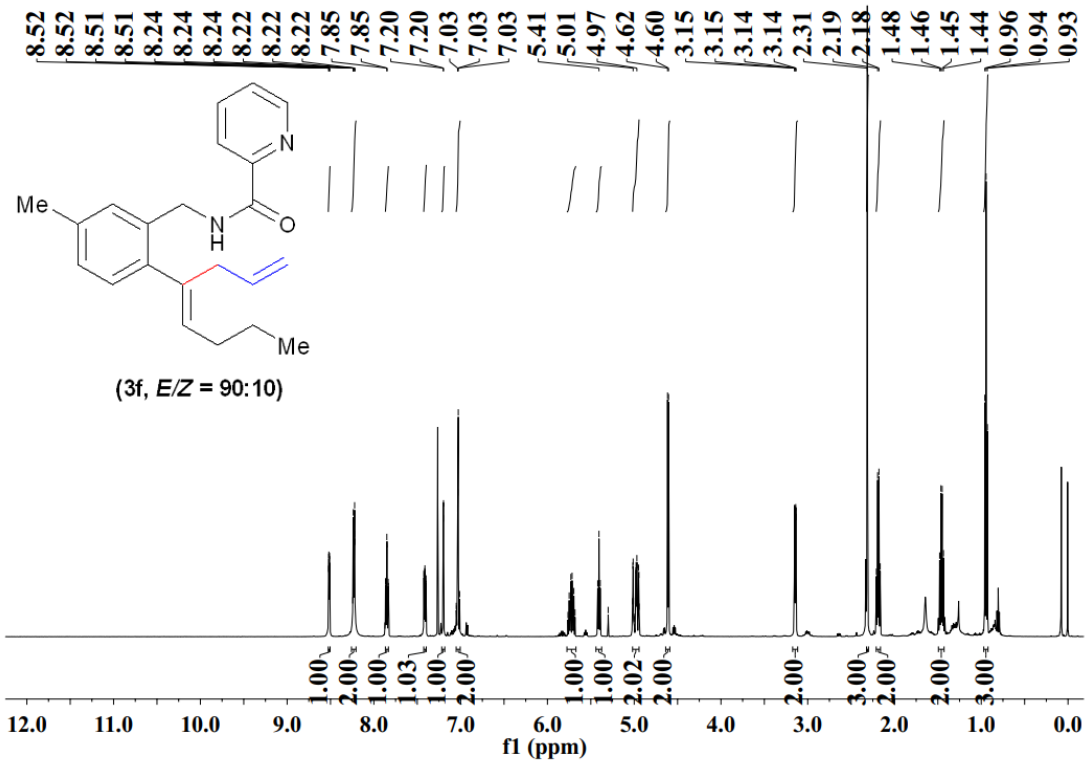
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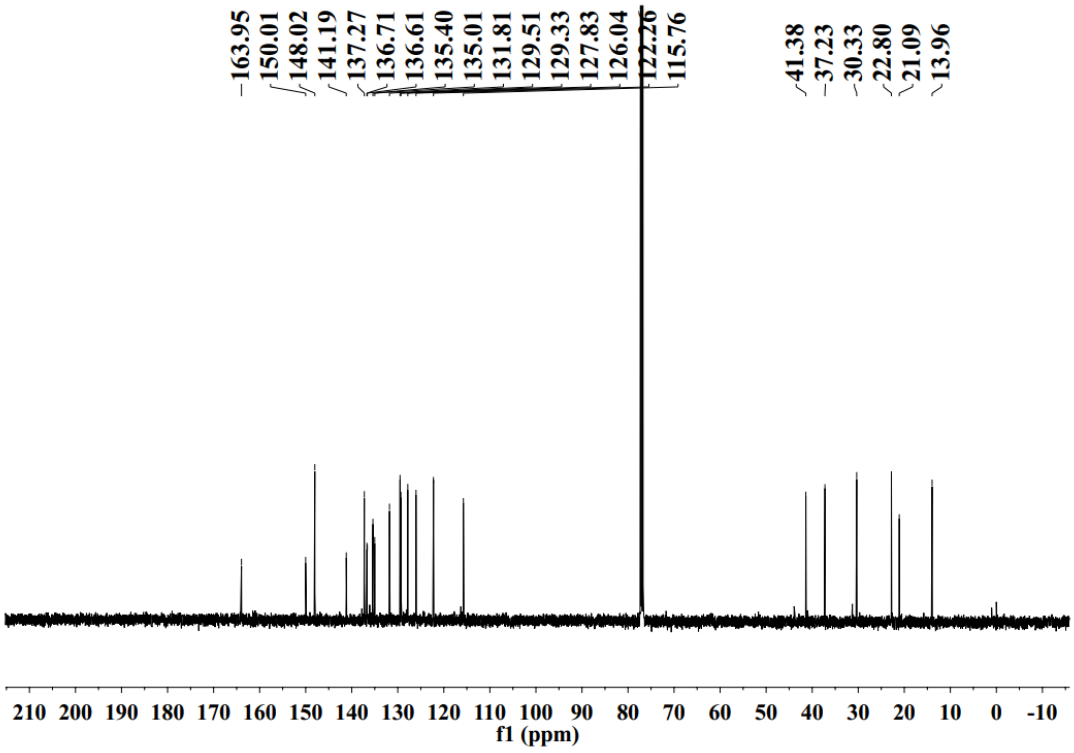
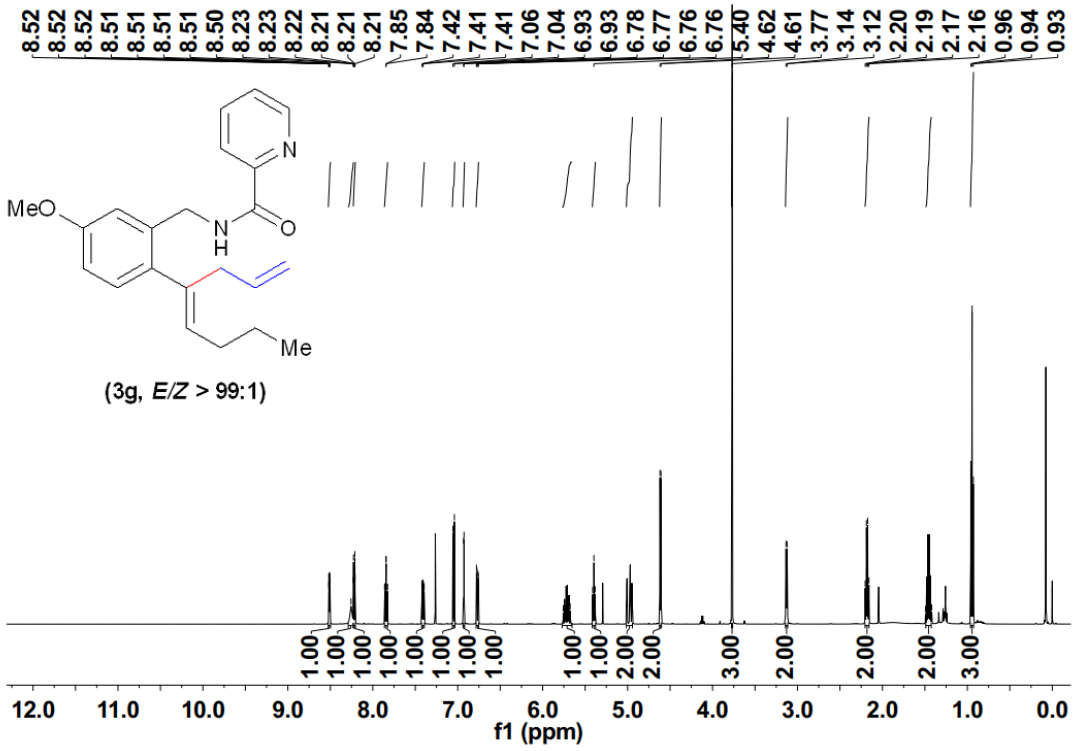
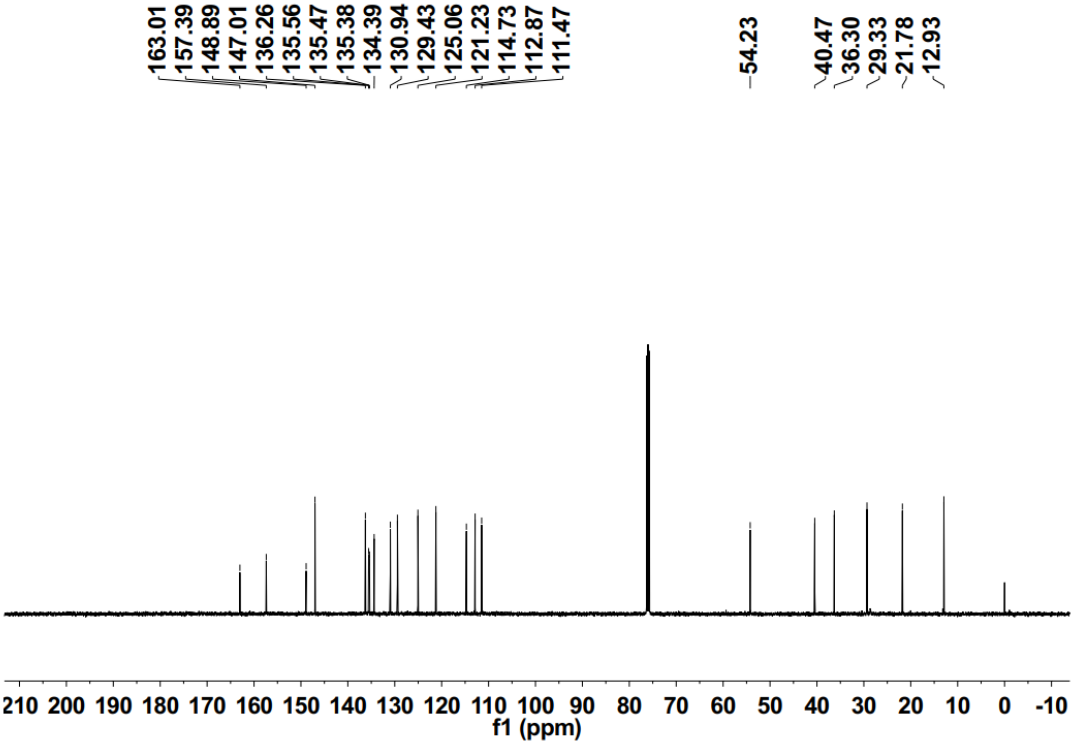
N
Me
N
H
O
Me
1.00 2.00 1.00 1.03 1.00 2.00 1.00 1.00 2.02 2.00 2.00 3.00 2.00 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

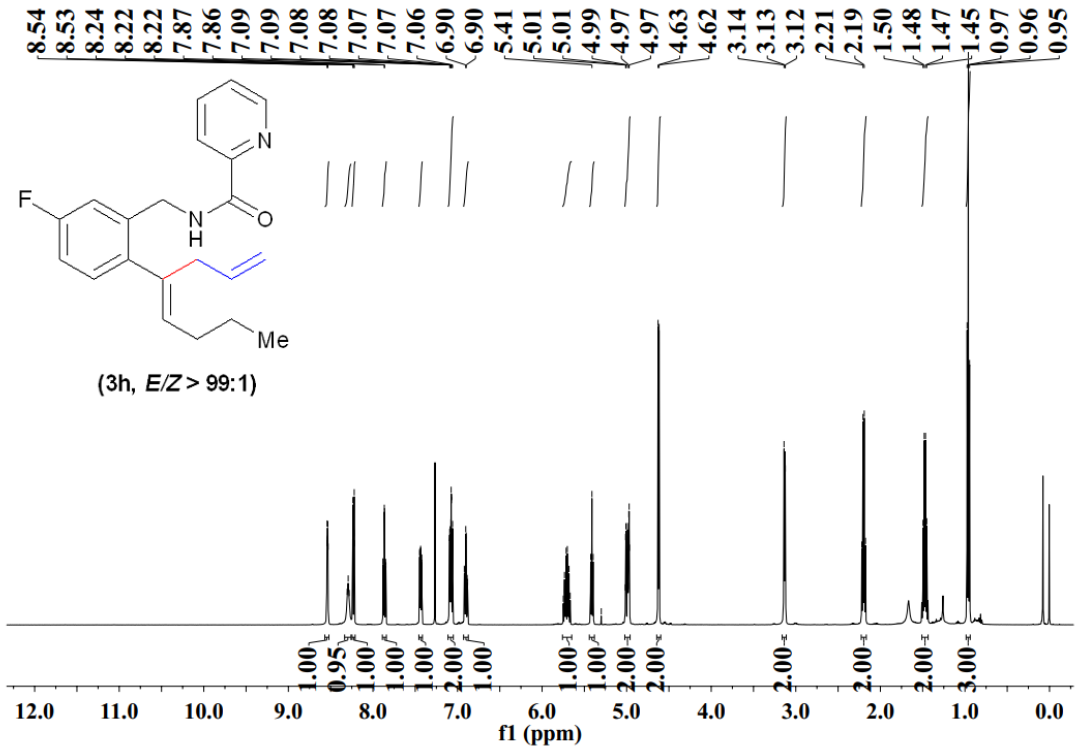
(3f, *E*/*Z* = 90:10)

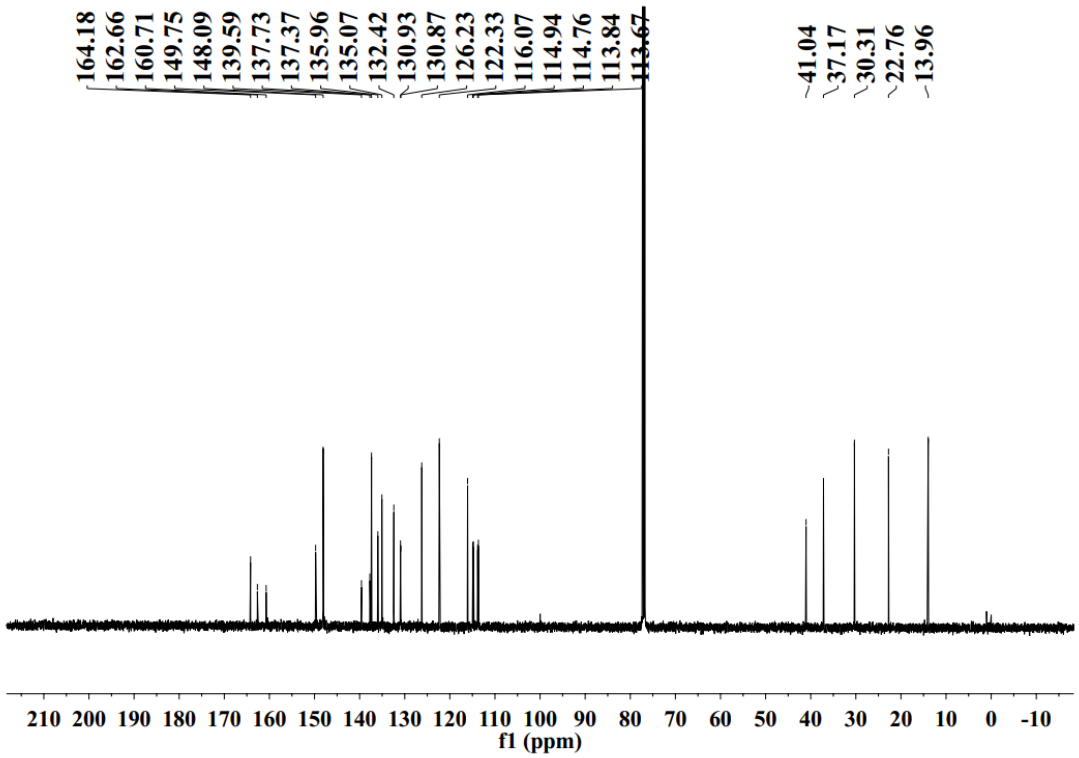

163.95
150.01
148.02
141.19
137.27
136.71
136.61
135.40
135.01
131.81
129.51
129.33
127.83
126.04
122.26
115.76
41.38
37.23
30.33
22.80
21.09
13.96
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

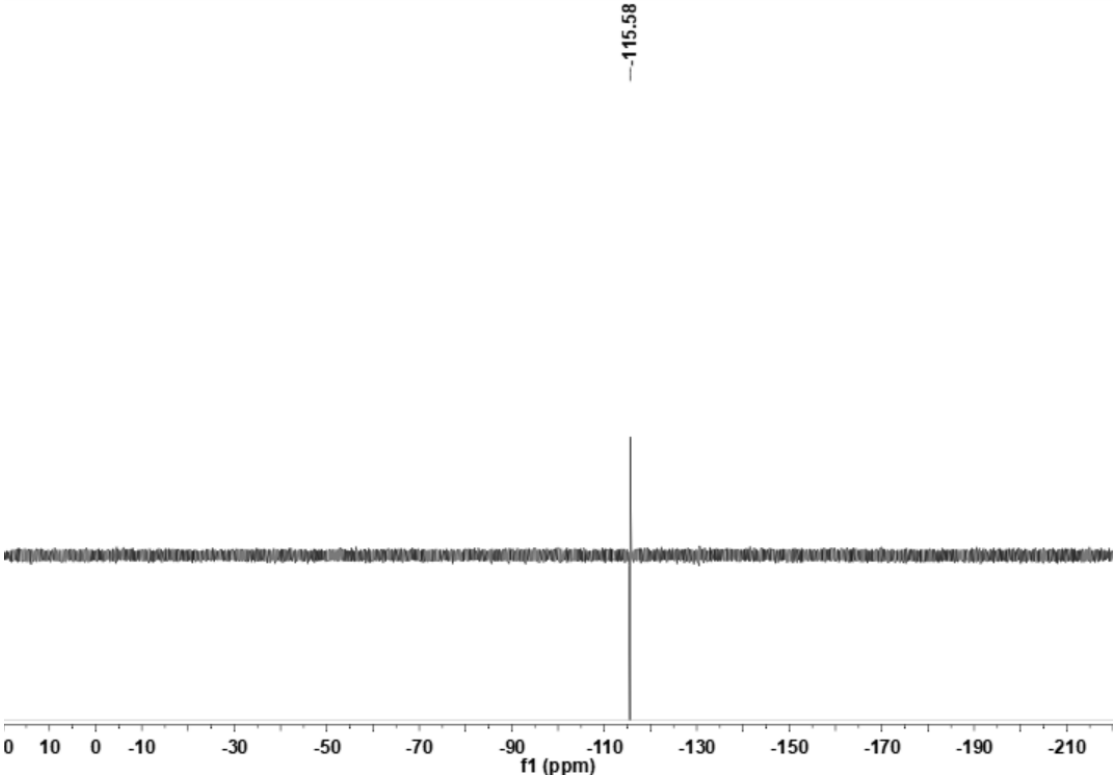

8.52
8.52
8.52
8.51
8.51
8.51
8.51
8.50
8.23
8.23
8.22
8.21
8.21
8.21
7.85
7.84
7.42
7.41
7.41
7.06
7.04
6.93
6.93
6.78
6.77
6.76
6.76
5.40
4.62
4.61
3.77
3.14
3.12
2.20
2.19
2.17
2.16
0.96
0.94
0.93
MeO
N
H
O
Me
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
2.00
2.00
3.00
2.00
2.00
2.00
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

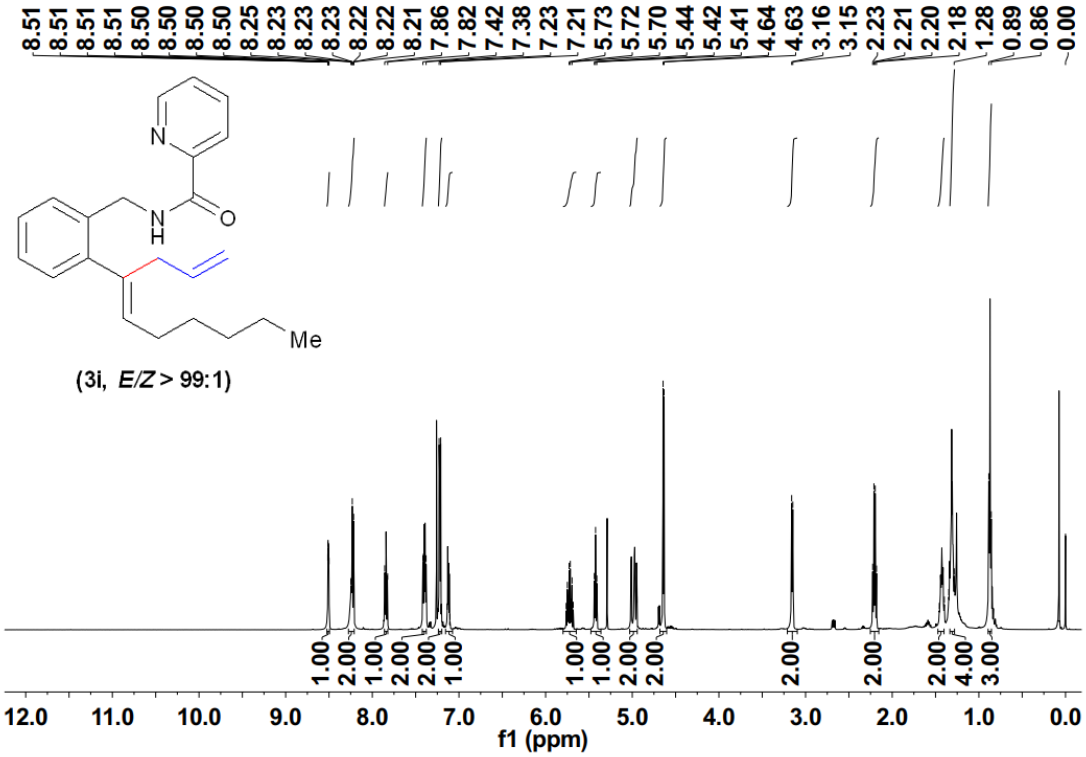
(3g, *E/Z* > 99:1)

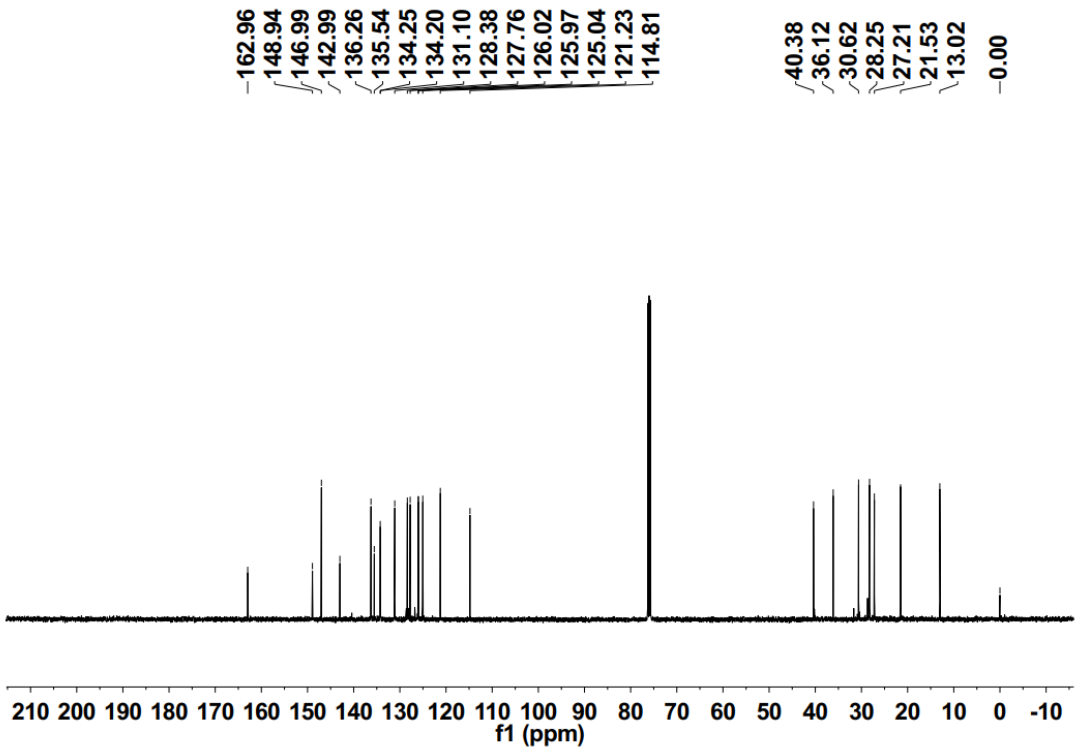

163.01
157.39
148.89
147.01
136.26
135.56
135.47
135.38
134.39
130.94
129.43
125.06
121.23
114.73
112.87
111.47
54.23
40.47
36.30
29.33
21.78
12.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

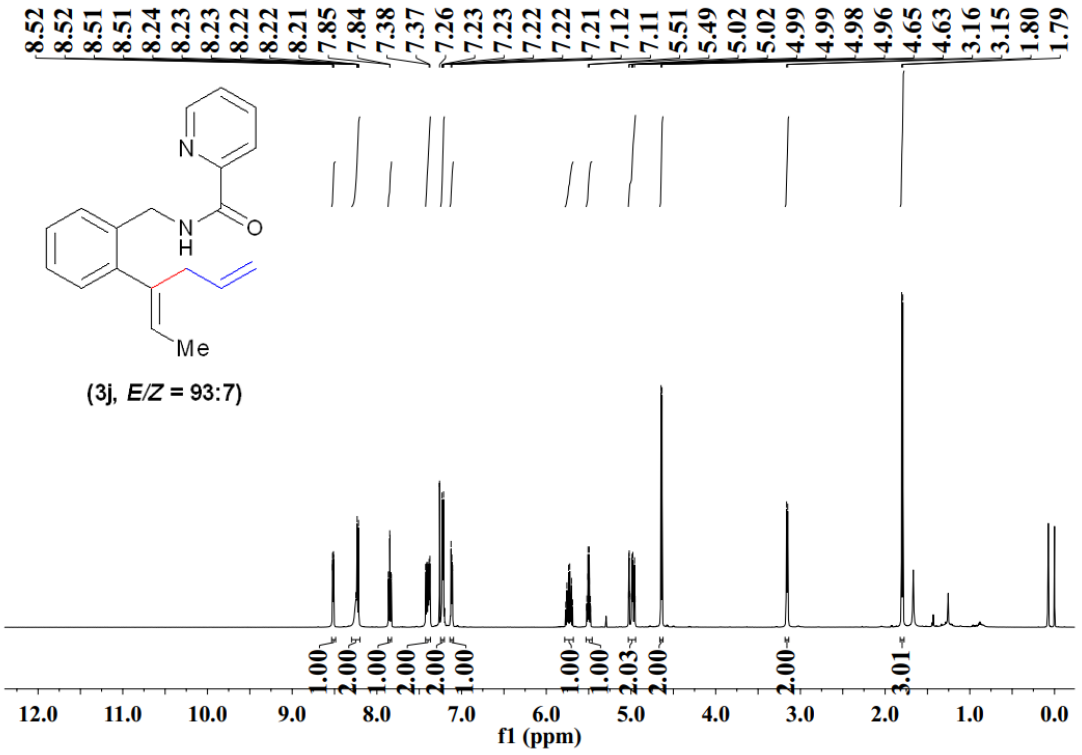

8.54
8.53
8.24
8.22
8.22
7.87
7.86
7.09
7.09
7.08
7.08
7.07
7.07
7.06
6.90
6.90
5.41
5.01
5.01
4.99
4.97
4.97
4.63
4.62
3.14
3.13
3.12
2.21
2.19
1.50
1.48
1.47
1.45
0.97
0.96
0.95
F
N
H
O
N
Me
(3h, E/Z > 99:1)
1.00
0.95
1.00
1.00
1.00
2.00
1.00
1.00
1.00
2.00
2.00
2.00
2.00
2.00
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

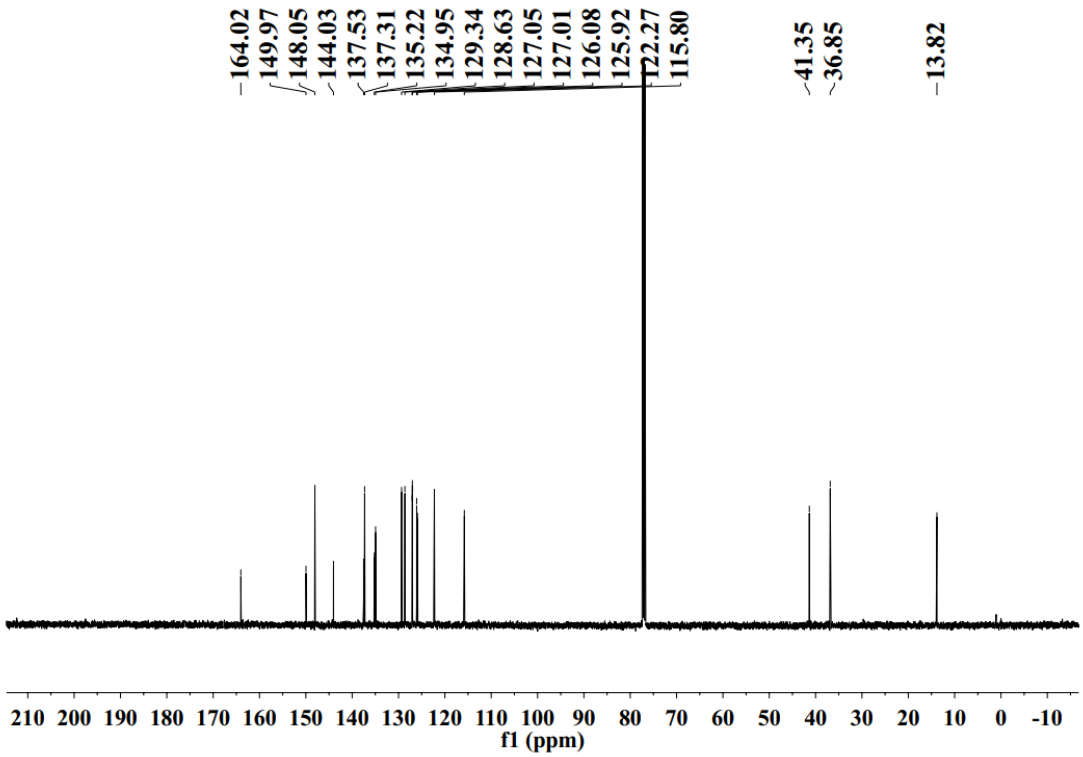

164.18
162.66
160.71
149.75
148.09
139.59
137.73
137.37
135.96
135.07
132.42
130.93
130.87
126.23
122.33
116.07
114.94
114.76
113.84
113.67
41.04
37.17
30.31
22.76
13.96
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

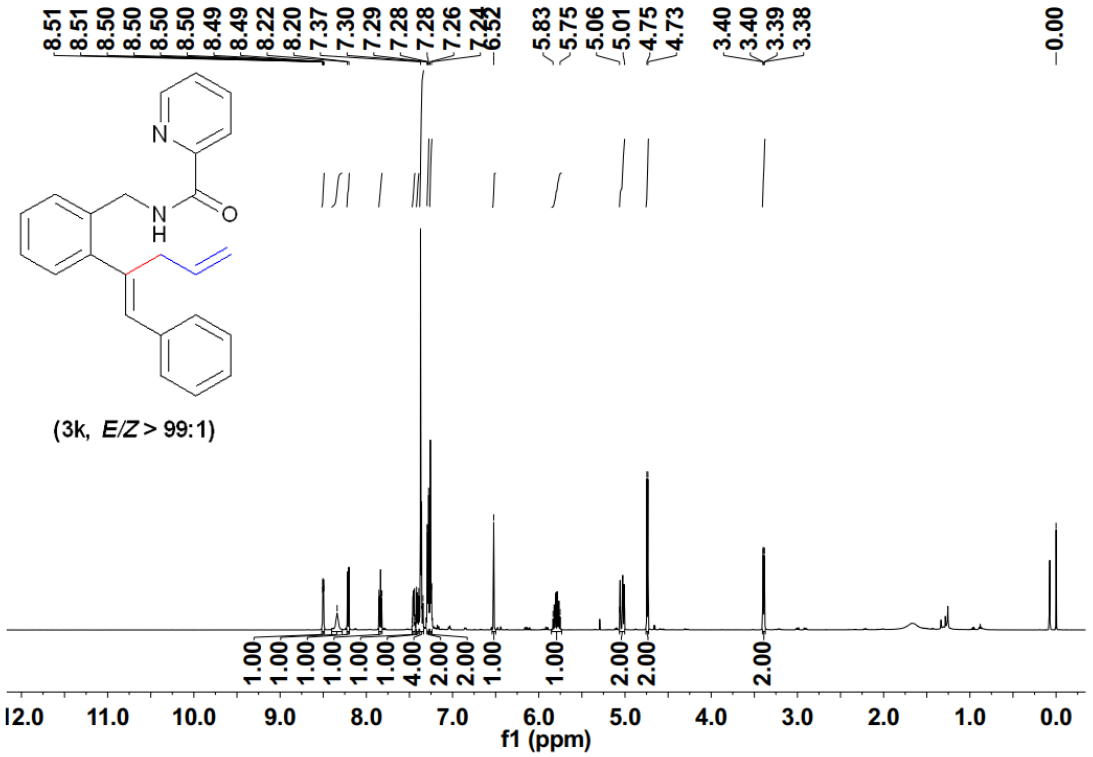

-115.58
0 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

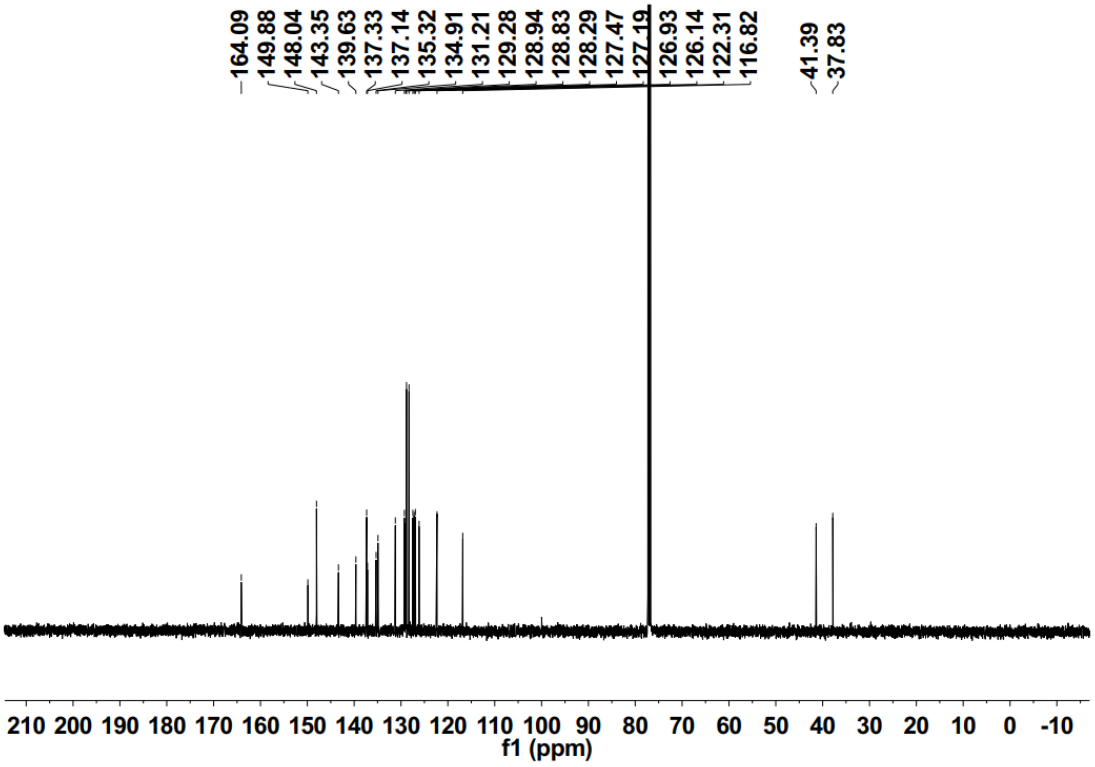

8.51 8.51 8.51 8.51 8.50 8.50 8.50 8.50 8.25 8.23 8.23 8.23 8.22 8.22 8.21 7.86 7.82 7.42 7.38 7.23 7.21 5.73 5.72 5.70 5.44 5.42 5.41 4.64 4.63 3.16 3.15 2.23 2.21 2.20 2.18 1.28 0.89 0.86 0.00
N
N
H
O
Me
(3i, E/Z > 99:1)
1.00 2.00 1.00 2.00 2.00 1.00 1.00 1.00 2.00 2.00 2.00 2.00 2.00 4.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

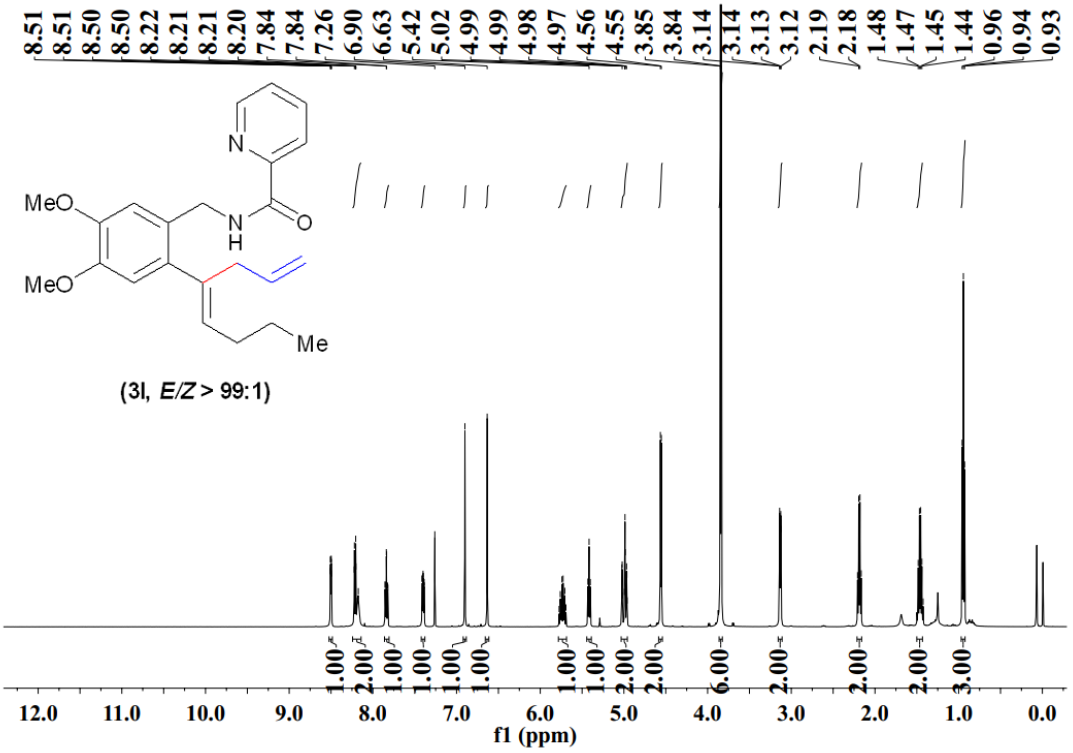

162.96 148.94 146.99 142.99 136.26 135.54 134.25 134.20 131.10 128.38 127.76 126.02 125.97 125.04 121.23 114.81
40.38 36.12 30.62 28.25 27.21 21.53 13.02 0.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.52 8.52 8.51 8.51 8.24 8.23 8.23 8.22 8.22 8.21 7.85 7.84 7.38 7.37 7.26 7.23 7.23 7.22 7.22 7.21 7.12 7.11 5.51 5.49 5.02 5.02 4.99 4.99 4.98 4.96 4.65 4.63 3.16 3.15 1.80 1.79
N
N
H
O
Me
(3j, E/Z = 93:7)
1.00 2.00 1.00 2.00 2.00 1.00 1.00 1.00 2.03 2.00 2.00 3.01
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

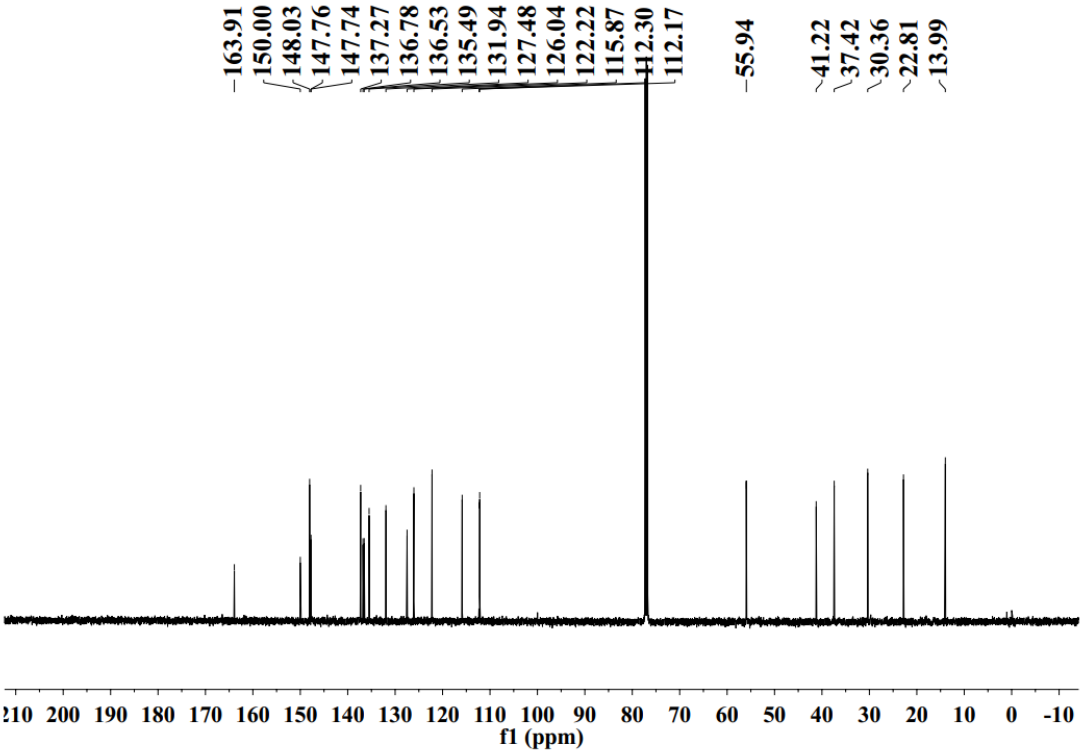

164.02 149.97 148.05 144.03 137.53 137.31 135.22 134.95 129.34 128.63 127.05 127.01 126.08 125.92 122.27 115.80
41.35 36.85
13.82
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

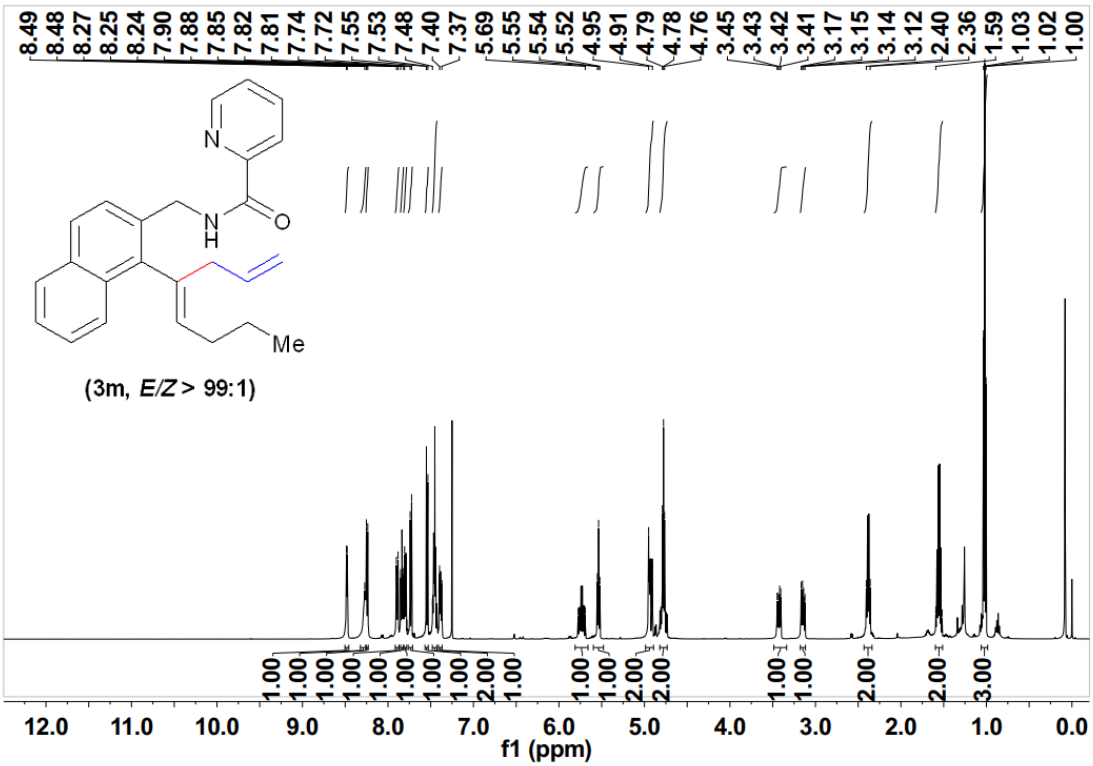

8.51
8.51
8.50
8.50
8.50
8.50
8.49
8.49
8.22
8.20
7.37
7.30
7.29
7.28
7.28
7.26
7.24
6.52
5.83
5.75
5.06
5.01
4.75
4.73
3.40
3.40
3.39
3.38
0.00
N
N
H
O
(3k, E/Z > 99:1)
1.00
1.00
1.00
1.00
1.00
1.00
4.00
2.00
2.00
1.00
1.00
2.00
2.00
2.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

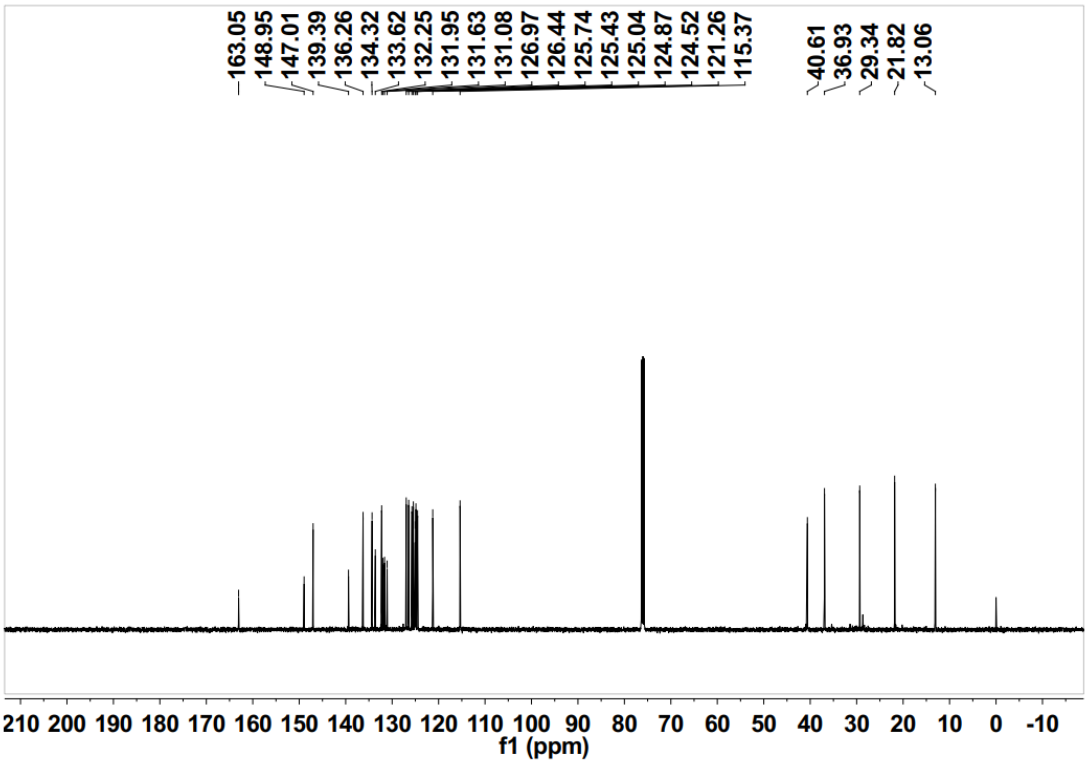

164.09
149.88
148.04
143.35
139.63
137.33
137.14
135.32
134.91
131.21
129.28
128.94
128.83
128.29
127.47
127.19
126.93
126.14
122.31
116.82
41.39
37.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

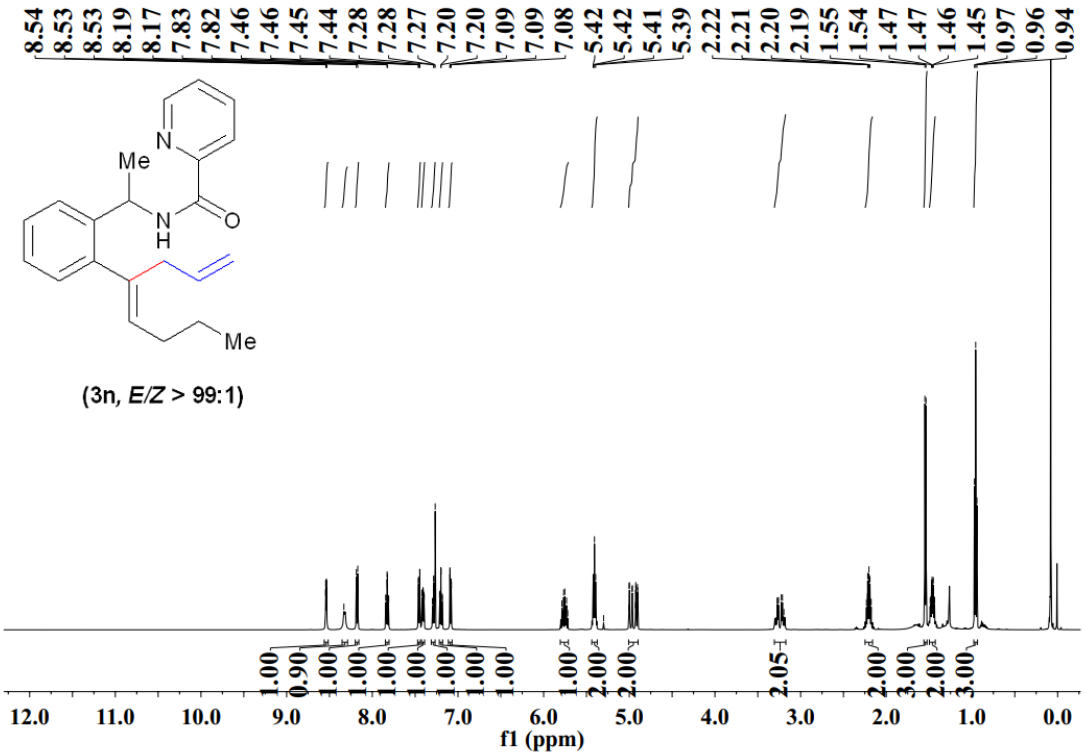

8.51 8.51 8.50 8.50 8.22 8.21 8.21 8.20 7.84 7.84 7.26 6.90 6.63 5.42 5.02 4.99 4.99 4.98 4.97 4.56 4.55 3.85 3.84 3.14 3.14 3.13 3.12 2.19 2.18 1.48 1.47 1.45 1.44 0.96 0.94 0.93
MeO
MeO
N
H
O
Me
1.00 2.00 1.00 1.00 1.00 1.00 1.00 1.00 2.00 2.00 6.00 2.00 2.00 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

(3l, *E*/*Z* > 99:1)

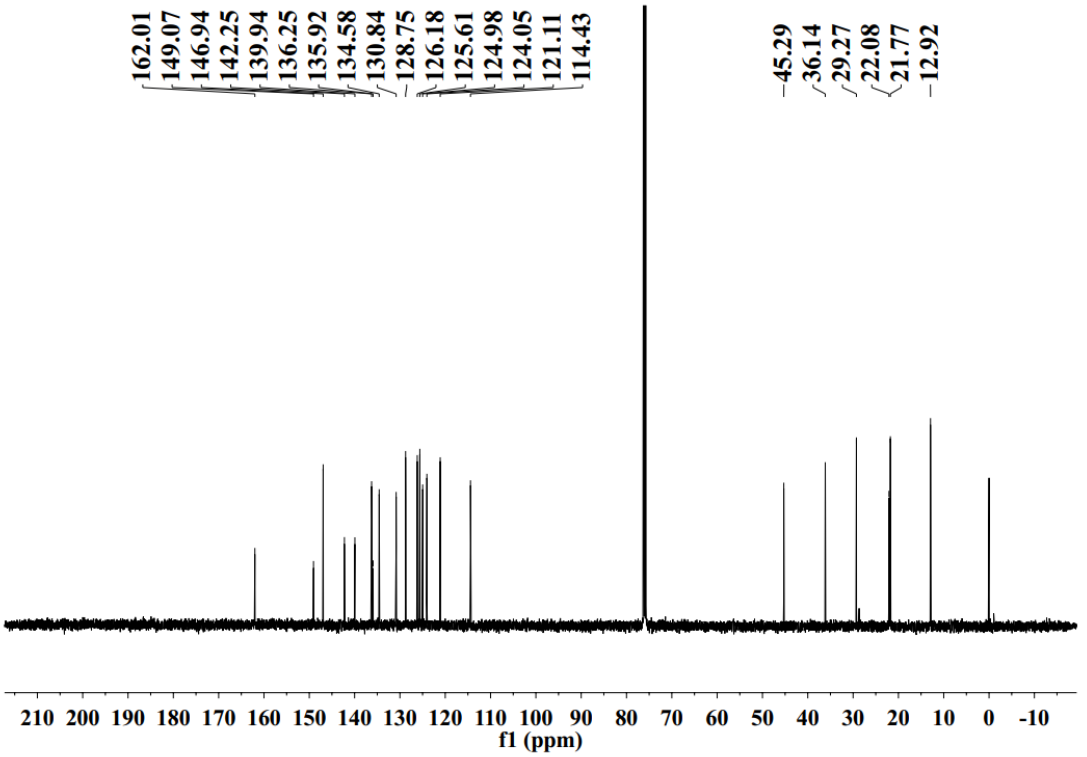

163.91 150.00 148.03 147.76 147.74 137.27 136.78 136.53 135.49 131.94 127.48 126.04 122.22 115.87 112.30 112.17
55.94
41.22 37.42 30.36 22.81 13.99
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

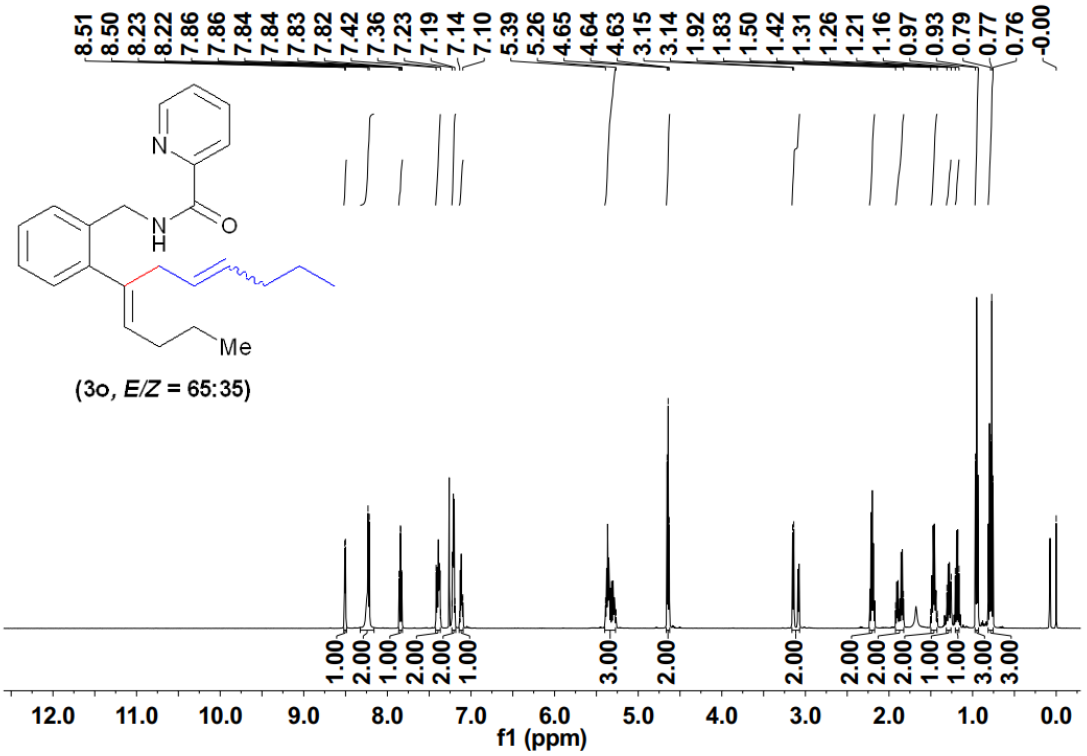

8.49 8.48 8.27 8.25 8.24 7.90 7.88 7.85 7.82 7.81 7.74 7.72 7.55 7.53 7.48 7.40 7.37 5.69 5.55 5.54 5.52 4.95 4.91 4.79 4.78 4.76 3.45 3.43 3.42 3.41 3.17 3.15 3.14 3.12 2.40 2.36 1.59 1.03 1.02 1.00
N
N
H
O
Me
(3m, E/Z > 99:1)
1.00 1.00 1.00 1.00 1.00 1.00 1.00 2.00 1.00 1.00 1.00 2.00 2.00 1.00 1.00 2.00 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


163.05 148.95 147.01 139.39 136.26 134.32 133.62 132.25 131.95 131.63 131.08 126.97 126.44 125.74 125.43 125.04 124.87 124.52 121.26 115.37
40.61 36.93 29.34 21.82 13.06
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

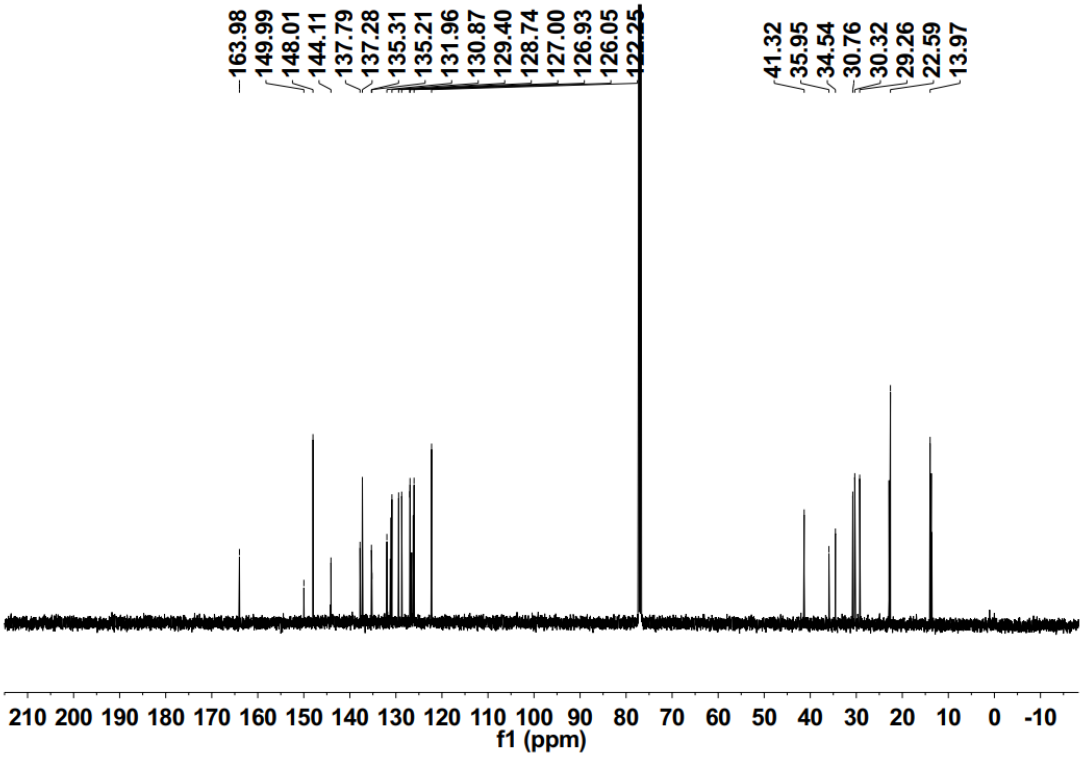

8.54 8.53 8.53 8.19 8.17 7.83 7.82 7.46 7.46 7.45 7.44 7.28 7.28 7.27 7.20 7.20 7.09 7.09 7.08 5.42 5.42 5.41 5.39 2.22 2.21 2.20 2.19 1.55 1.54 1.47 1.47 1.46 1.45 0.97 0.96 0.94
N
Me
N
H
O
Me
1.00 0.90 1.00 1.00 1.00 1.00 1.00 1.00 1.00 1.00 2.00 2.00 2.05 2.00 3.00 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

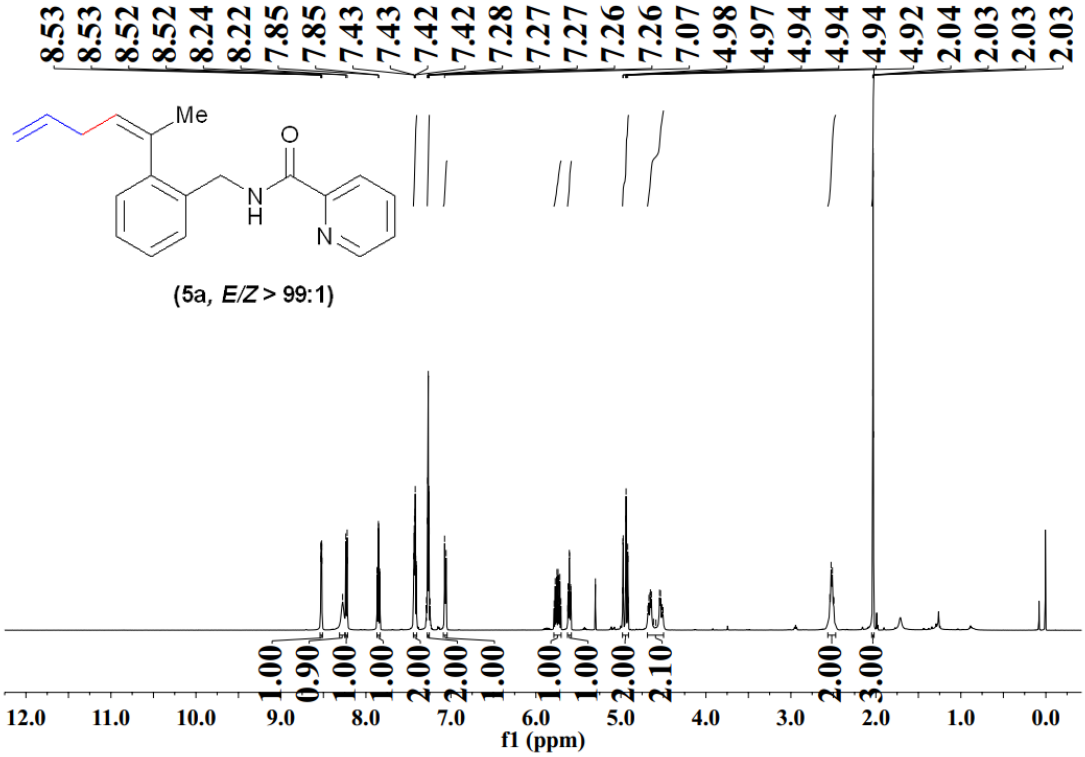
(3n, *E*/*Z* > 99:1)


162.01 149.07 146.94 142.25 139.94 136.25 135.92 134.58 130.84 128.75 126.18 125.61 124.98 124.05 121.11 114.43
45.29 36.14 29.27 22.08 21.77 12.92
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

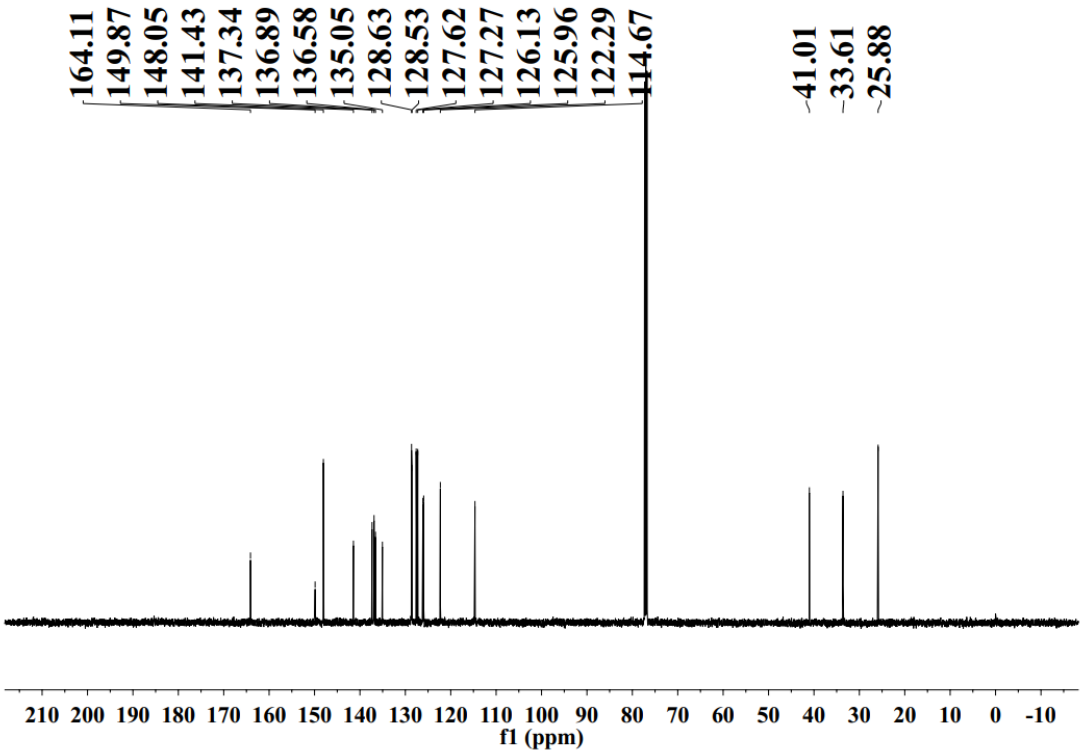

8.51
8.50
8.23
8.22
7.86
7.86
7.84
7.84
7.83
7.82
7.42
7.36
7.23
7.19
7.14
7.10
5.39
5.26
4.65
4.64
4.63
3.15
3.14
1.92
1.83
1.50
1.42
1.31
1.26
1.21
1.16
0.97
0.93
0.79
0.77
0.76
-0.00
N
N
H
O
Me
1.00
2.00
1.00
2.00
2.00
1.00
3.00
2.00
2.00
2.00
2.00
2.00
1.00
1.00
3.00
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

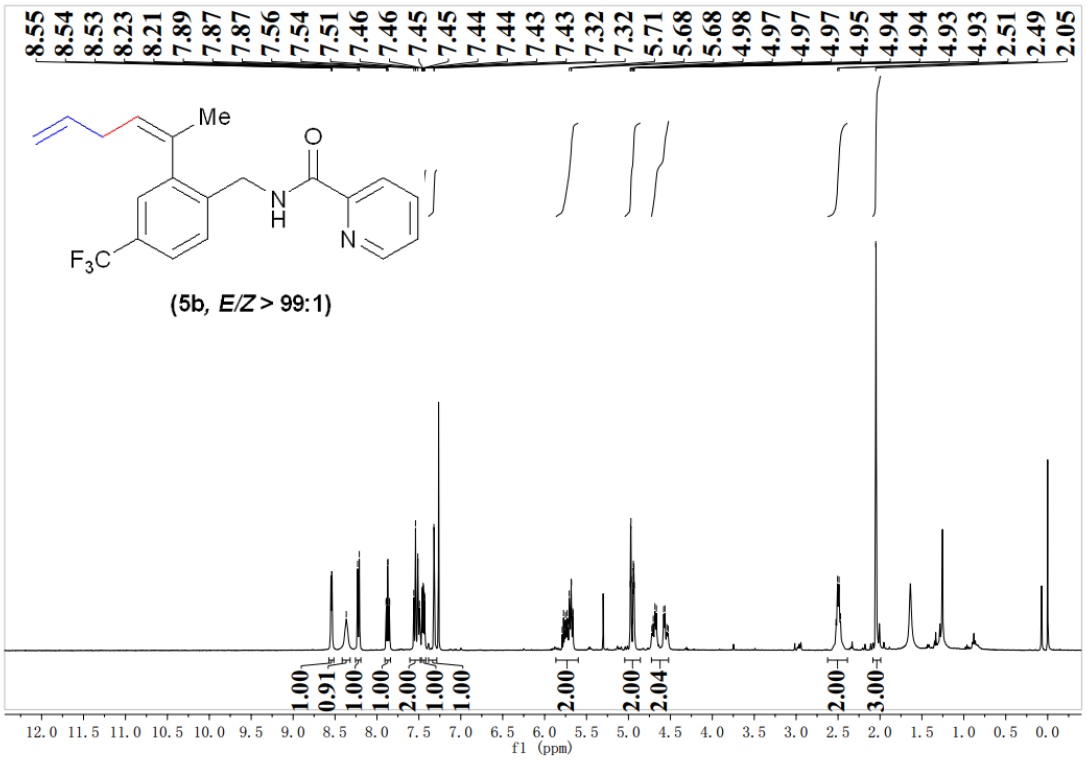
(3o, *E*/*Z* = 65:35)

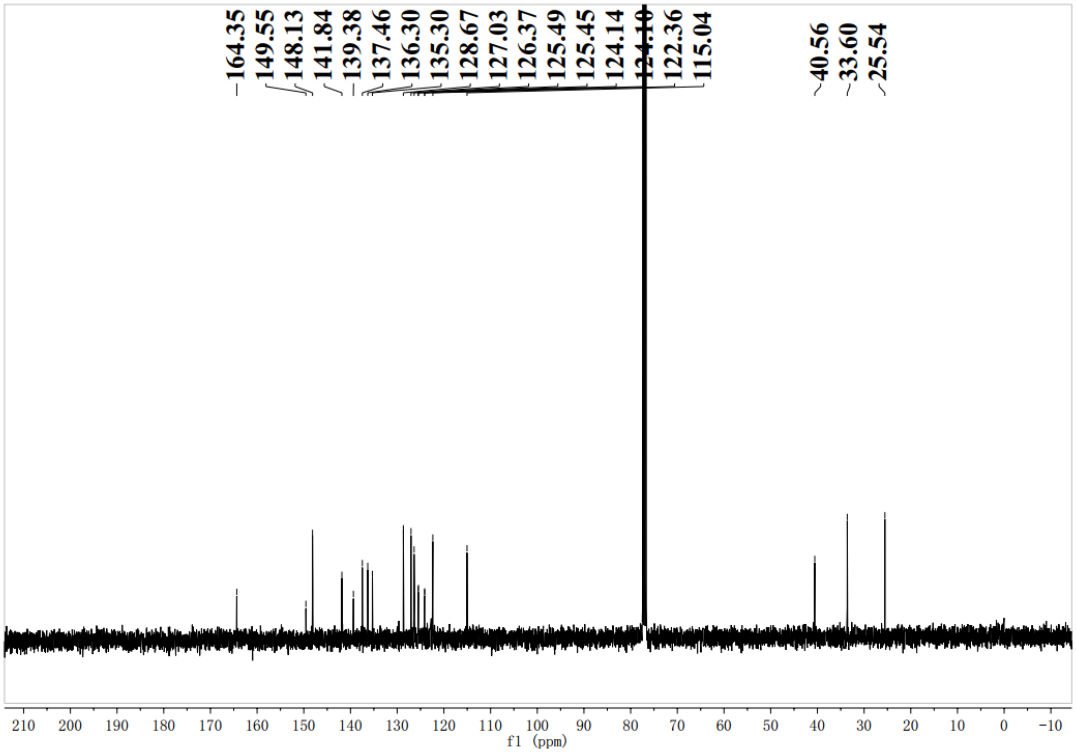

163.98
149.99
148.01
144.11
137.79
137.28
135.31
135.21
131.96
130.87
129.40
128.74
127.00
126.93
126.05
122.25
41.32
35.95
34.54
30.76
30.32
29.26
22.59
13.97
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

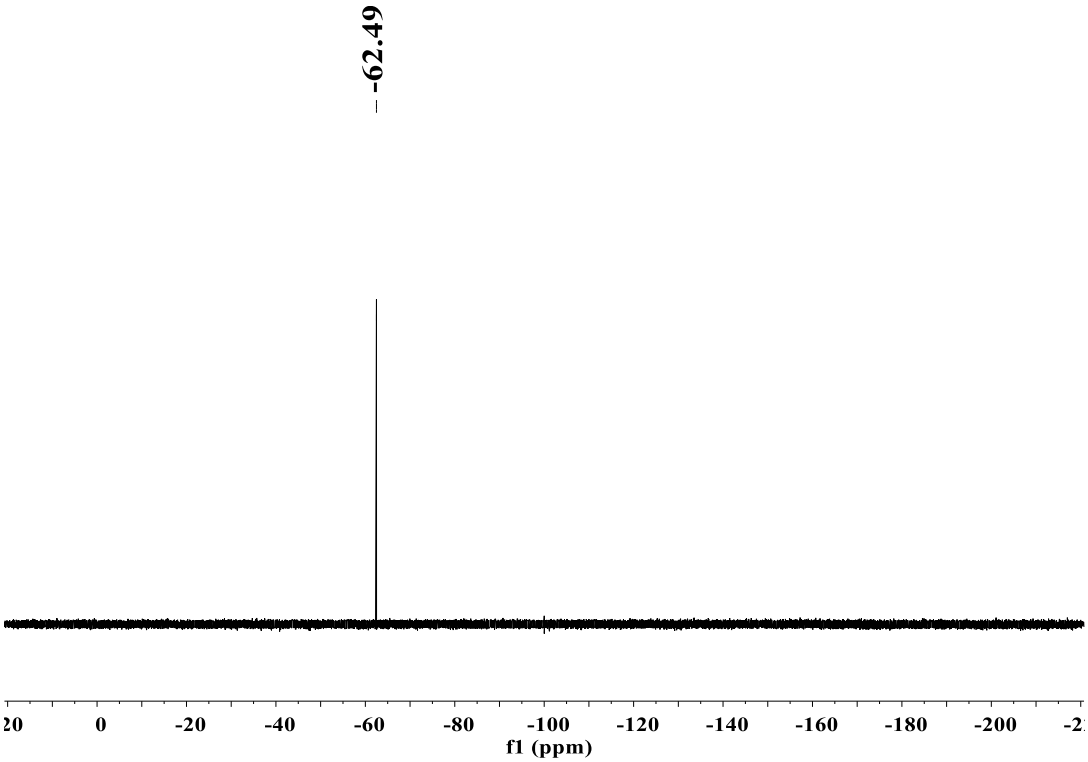

8.53 8.53 8.52 8.52 8.24 8.22 7.85 7.85 7.43 7.43 7.42 7.42 7.28 7.27 7.27 7.26 7.26 7.07 4.98 4.97 4.94 4.94 4.94 4.92 2.04 2.03 2.03 2.03
Me
O
N
H
N
1.00 0.90 1.00 1.00 2.00 2.00 1.00 1.00 1.00 2.00 2.10 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

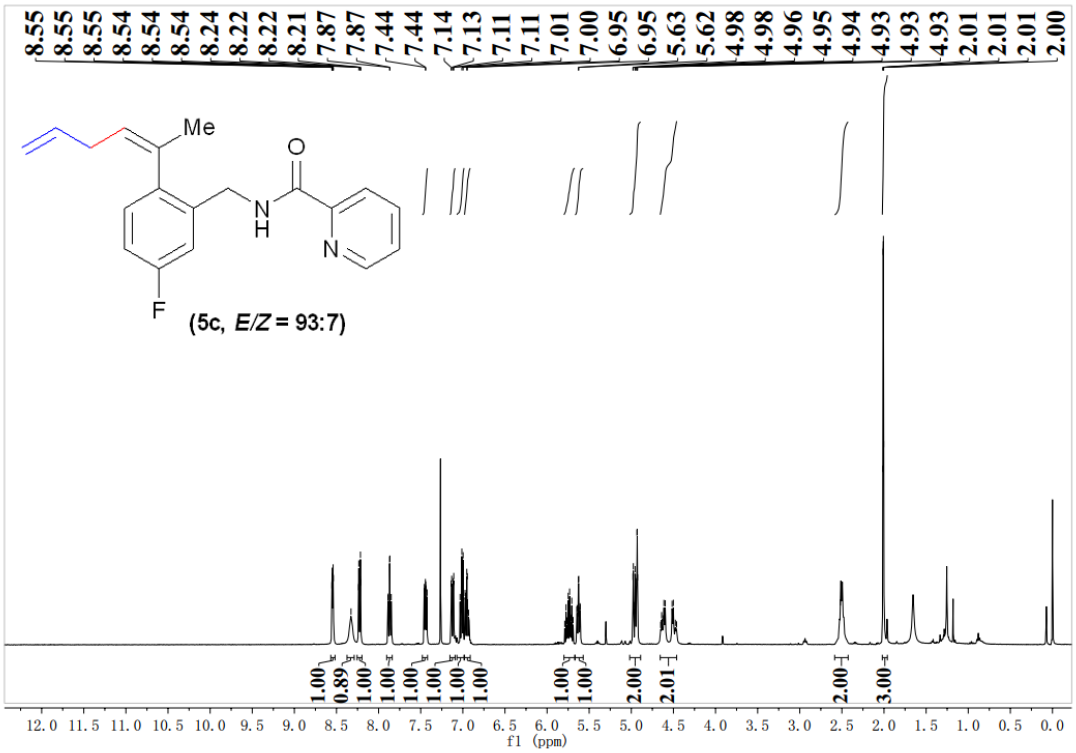
(5a, *E*/*Z* > 99:1)

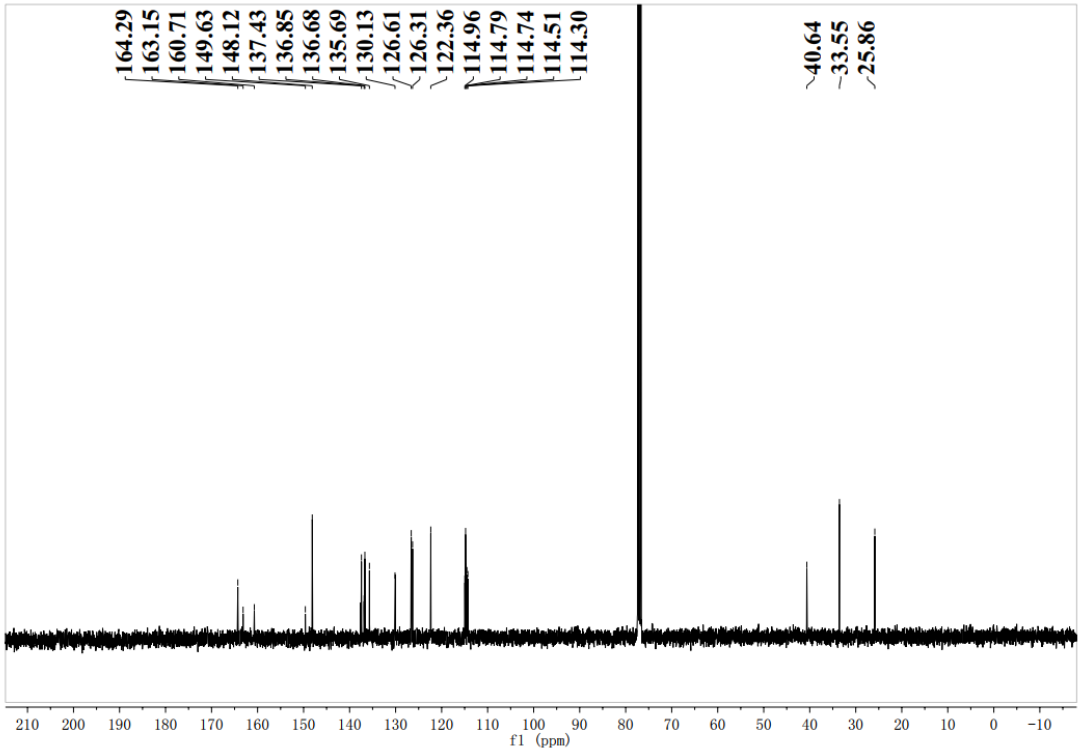

164.11 149.87 148.05 141.43 137.34 136.89 136.58 135.05 128.63 128.53 127.62 127.27 126.13 125.96 122.29 114.67
41.01 33.61 25.88
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

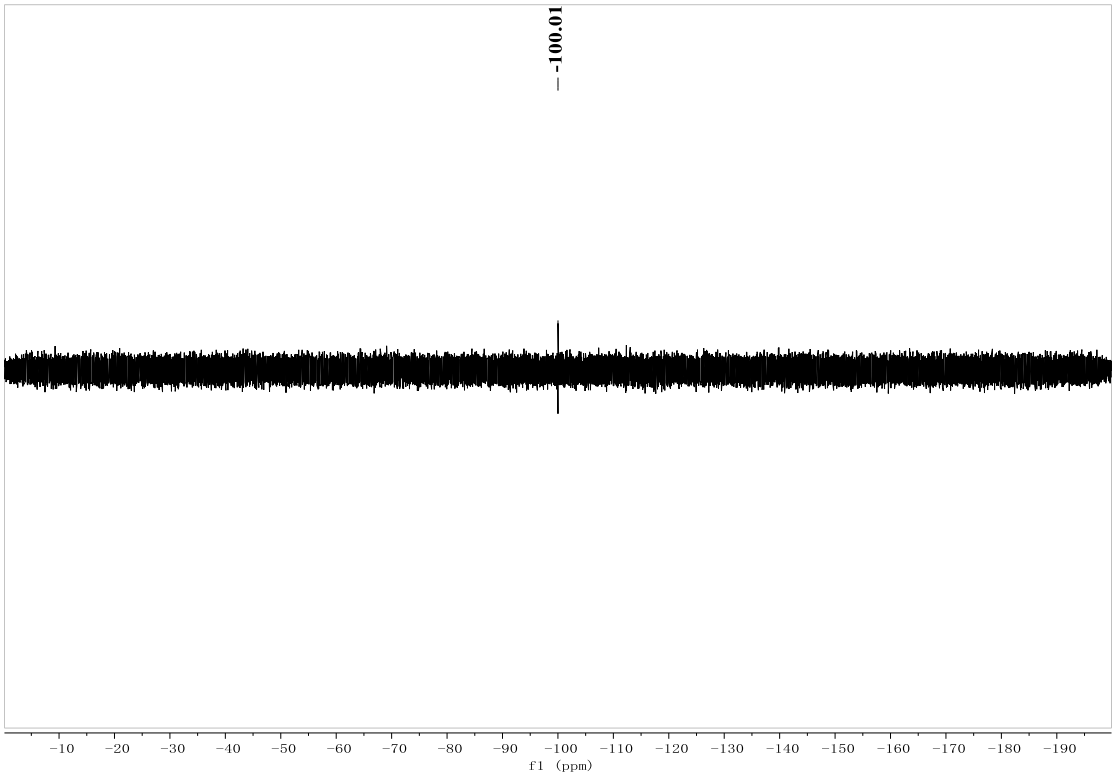

8.55 8.54 8.53 8.23 8.21 7.89 7.87 7.87 7.56 7.54 7.51 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.32 7.32 5.71 5.68 5.68 4.98 4.97 4.97 4.97 4.95 4.94 4.94 4.93 4.93 2.51 2.49 2.05
Me
O
N
H
N
F3C
(5b, E/Z > 99:1)
1.00 0.91 1.00 1.00 2.00 1.00 1.00 2.00 2.00 2.04 2.00 3.00
f1 (ppm)

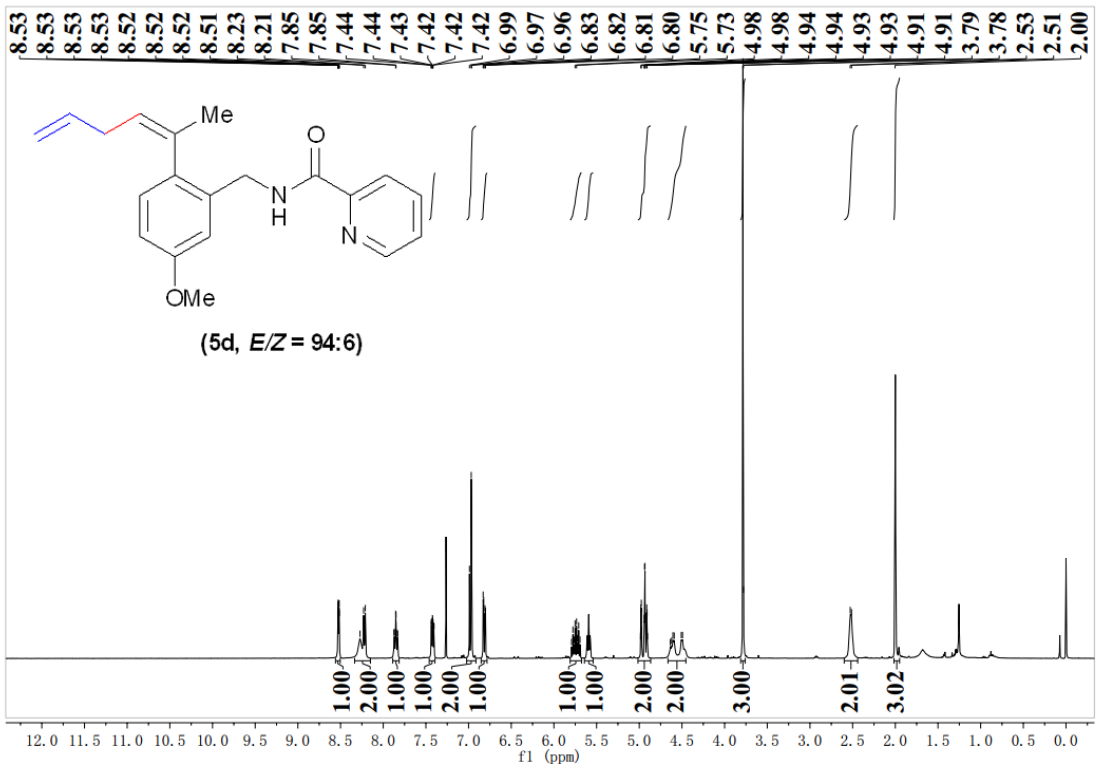

164.35 149.55 148.13 141.84 139.38 137.46 136.30 135.30 128.67 127.03 126.37 125.49 125.45 124.14 124.10 122.36 115.04
40.56 33.60 25.54
f1 (ppm)

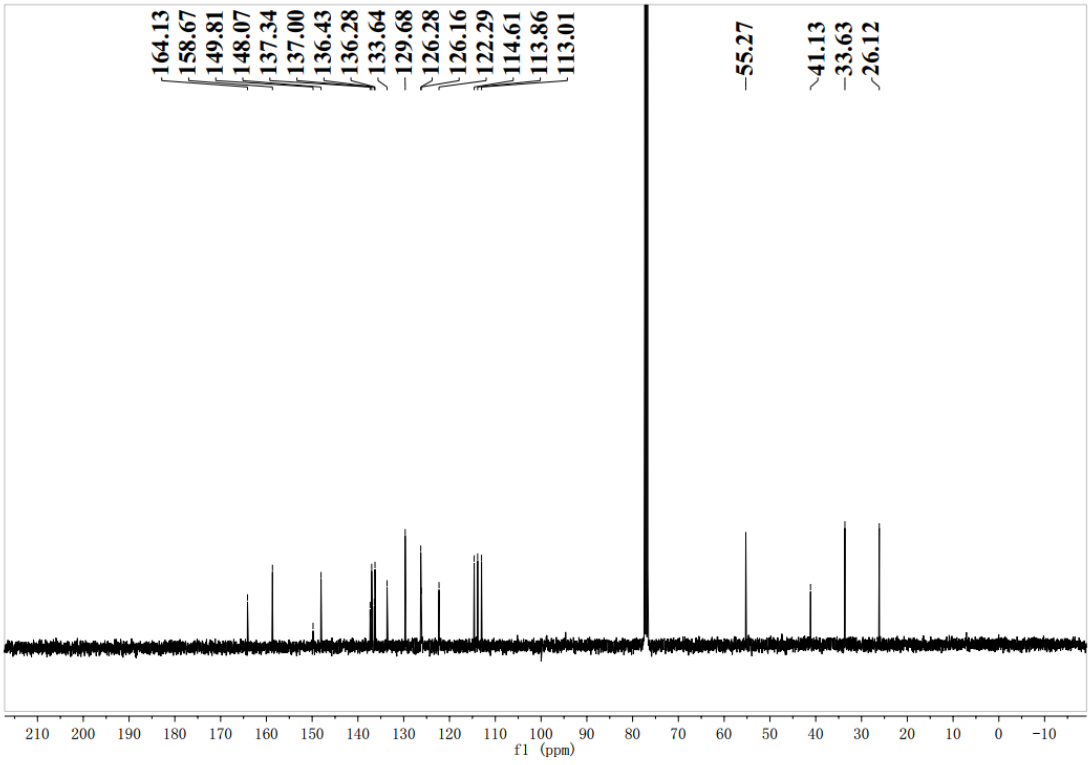

-62.49
20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)

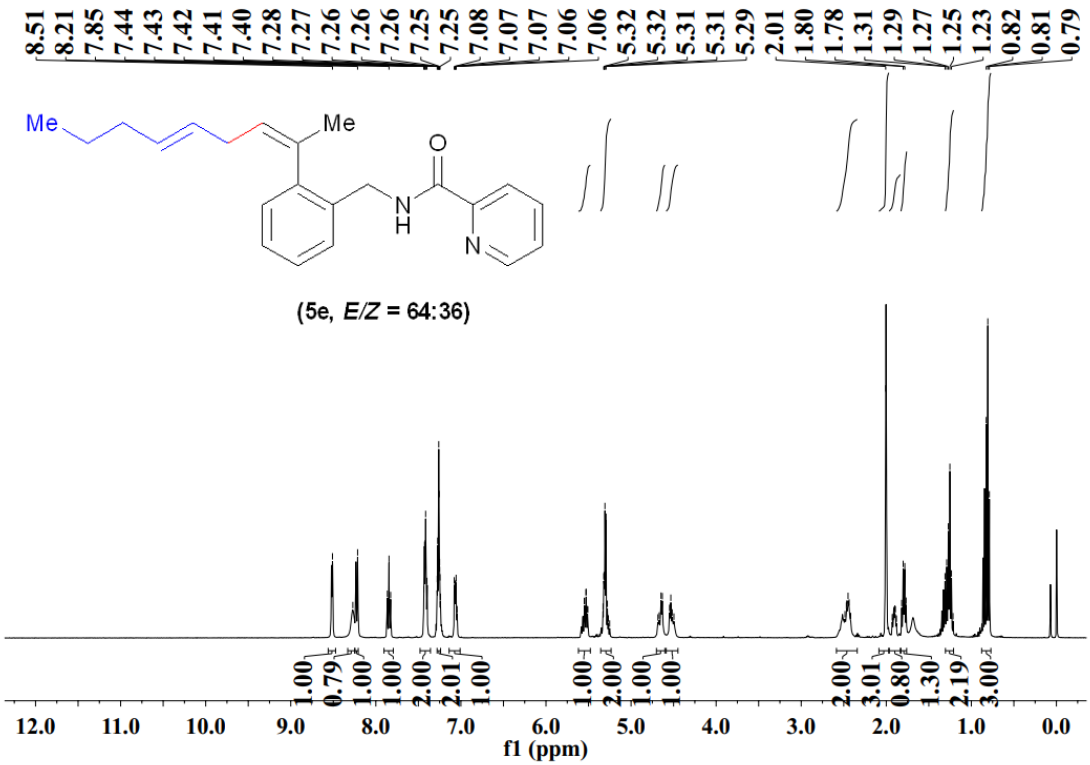

8.55 8.55 8.55 8.54 8.54 8.54 8.24 8.22 8.22 8.21 7.87 7.87 7.44 7.44 7.14 7.13 7.11 7.11 7.01 7.00 6.95 6.95 5.63 5.62 4.98 4.98 4.96 4.95 4.94 4.93 4.93 4.93 2.01 2.01 2.01 2.00
Me
O
N
H
N
F
(5c, E/Z = 93:7)
1.00 0.89 1.00 1.00 1.00 1.00 1.00 1.00 1.00 1.00 2.00 2.01 2.00 3.00
f1 (ppm)

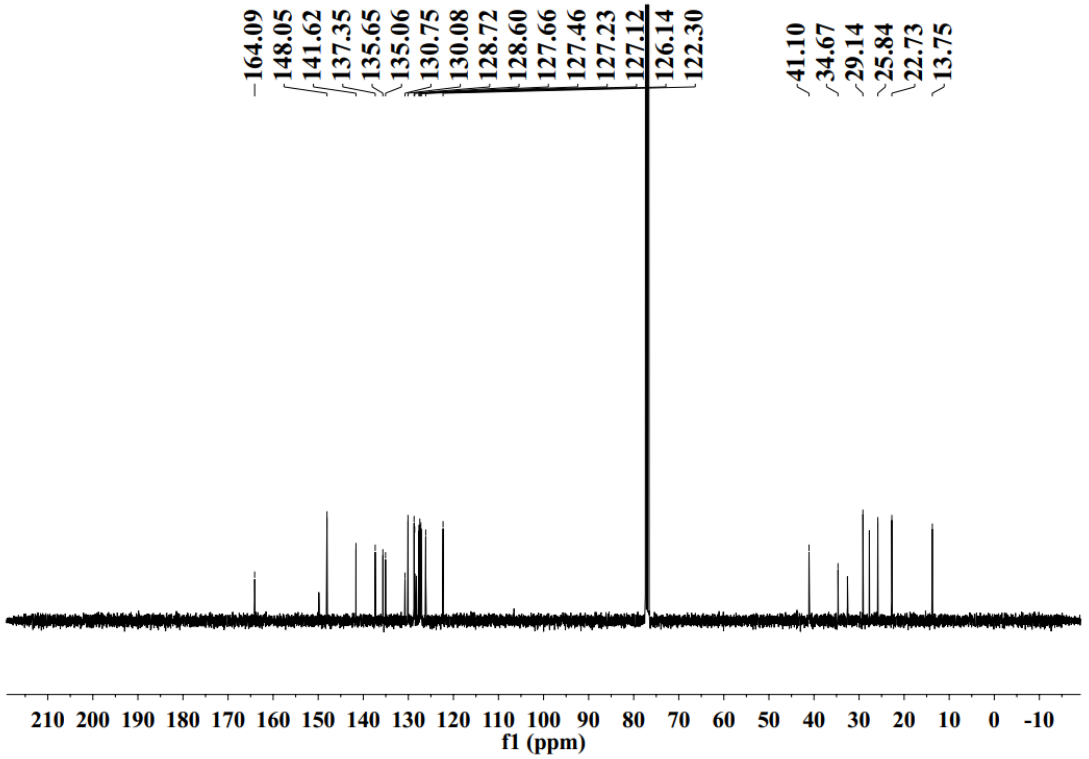

164.29
163.15
160.71
149.63
148.12
137.43
136.85
136.68
135.69
130.13
126.61
126.31
122.36
114.96
114.79
114.74
114.51
114.30
40.64
33.55
25.86
f1 (ppm)

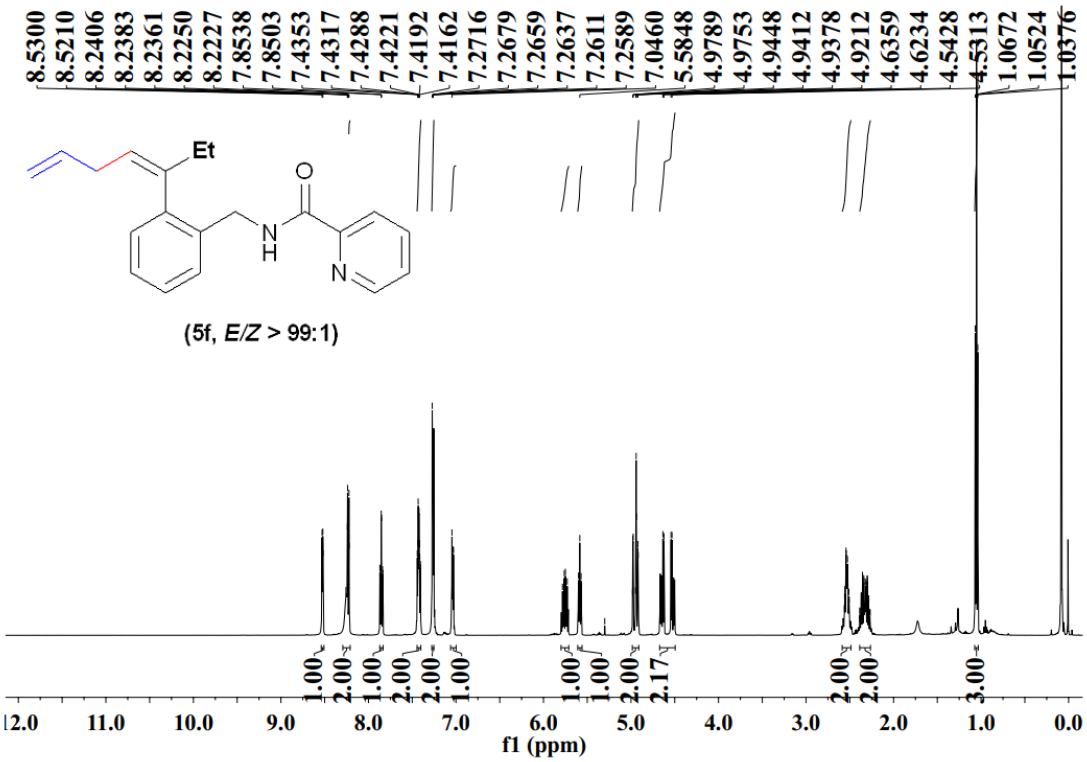

-100.01
f1 (ppm)

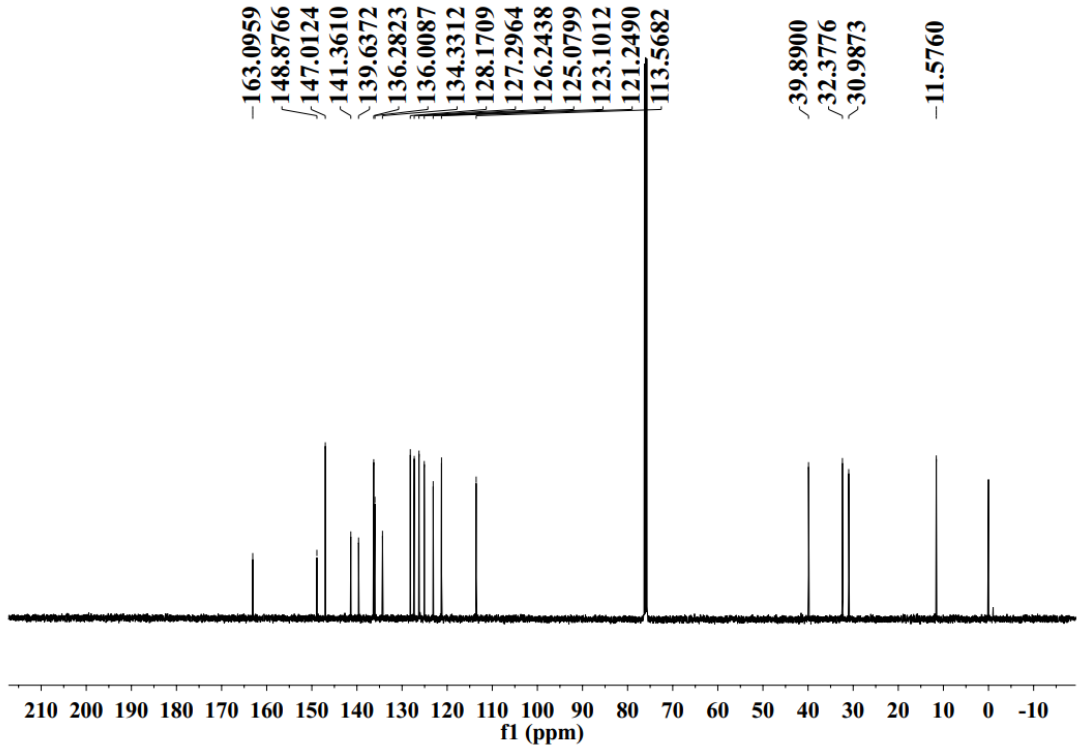

8.53 8.53 8.53 8.53 8.52 8.52 8.52 8.51 8.23 8.21 7.85 7.85 7.44 7.44 7.43 7.42 7.42 7.42 6.99 6.97 6.96 6.83 6.82 6.81 6.80 5.75 5.73 4.98 4.98 4.94 4.94 4.93 4.93 4.91 4.91 3.79 3.78 2.53 2.51 2.00
Me
O
N
H
N
OMe
(5d, E/Z = 94:6)
1.00 2.00 1.00 1.00 2.00 1.00 1.00 1.00 2.00 2.00 3.00 2.01 3.02
12.0 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
f1 (ppm)

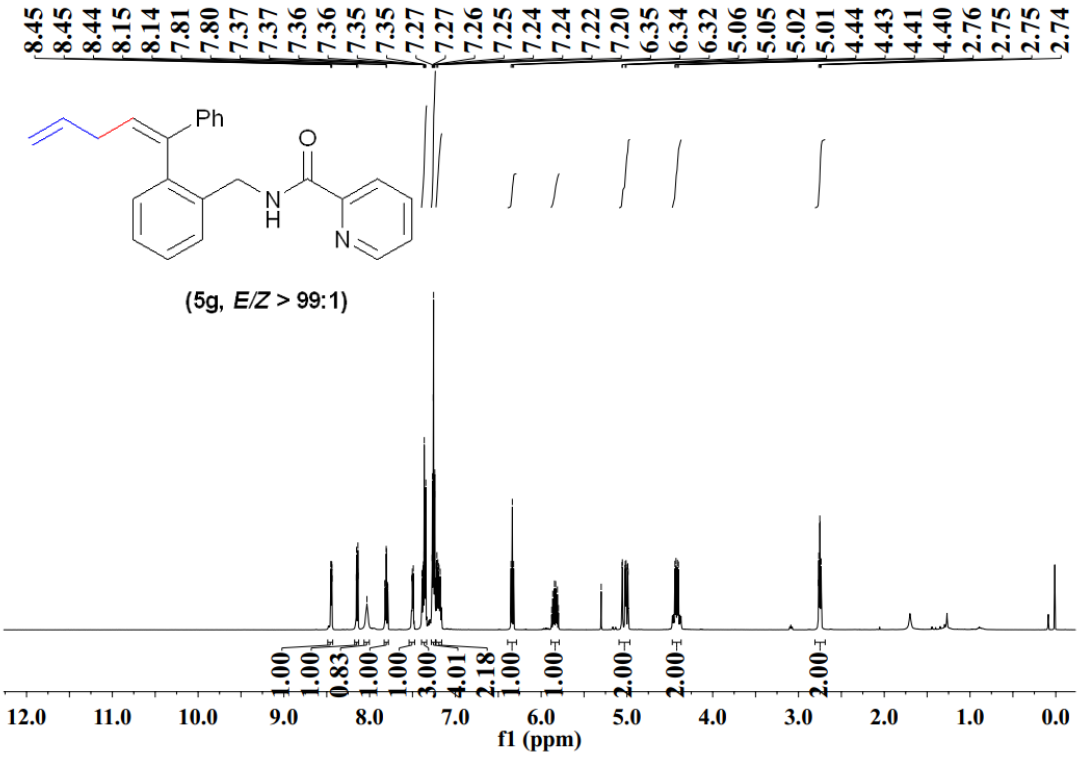

164.13 158.67 149.81 148.07 137.34 137.00 136.43 136.28 133.64 129.68 126.28 126.16 122.29 114.61 113.86 113.01
55.27
41.13 33.63 26.12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.51 8.21 7.85 7.44 7.43 7.42 7.41 7.40 7.28 7.27 7.26 7.26 7.26 7.25 7.25 7.08 7.07 7.07 7.06 7.06 5.32 5.32 5.31 5.31 5.29 2.01 1.80 1.78 1.31 1.29 1.27 1.25 1.23 0.82 0.81 0.79
Me
Me
O
N
H
N
(5e, E/Z = 64:36)
1.00 0.79 1.00 1.00 2.00 2.01 1.00 1.00 2.00 1.00 1.00 2.00 3.01 0.80 1.30 2.19 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

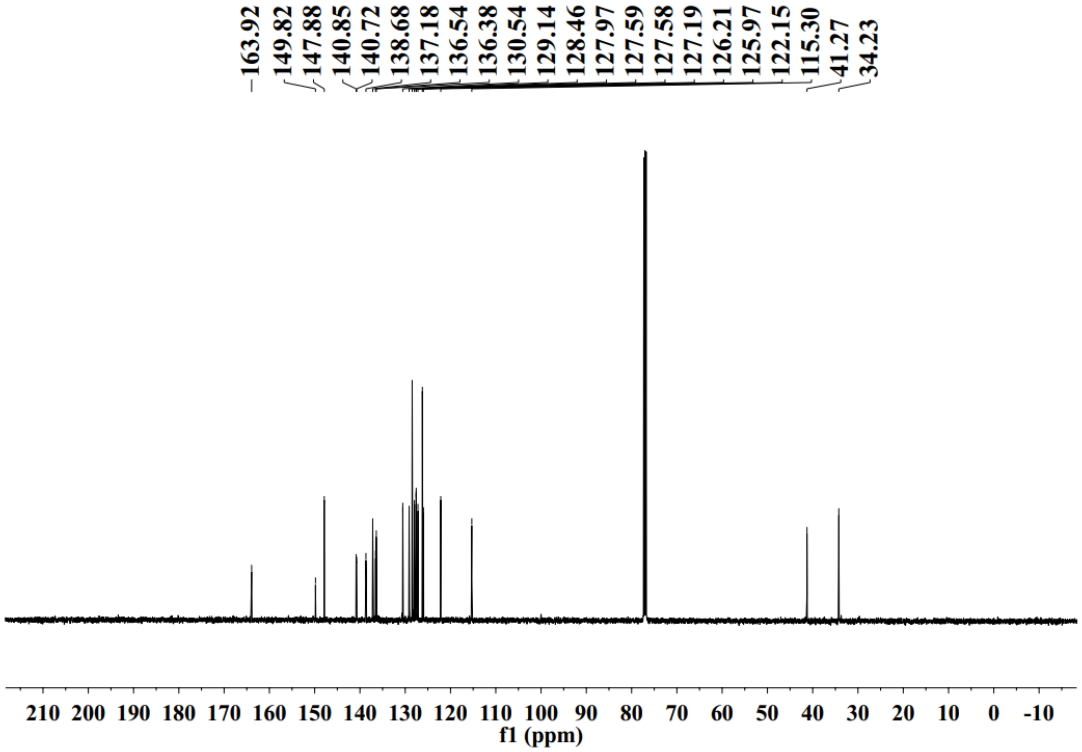

164.09 148.05 141.62 137.35 135.65 135.06 130.75 130.08 128.72 128.60 127.66 127.46 127.23 127.12 126.14 122.30
41.10 34.67 29.14 25.84 22.73 13.75
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

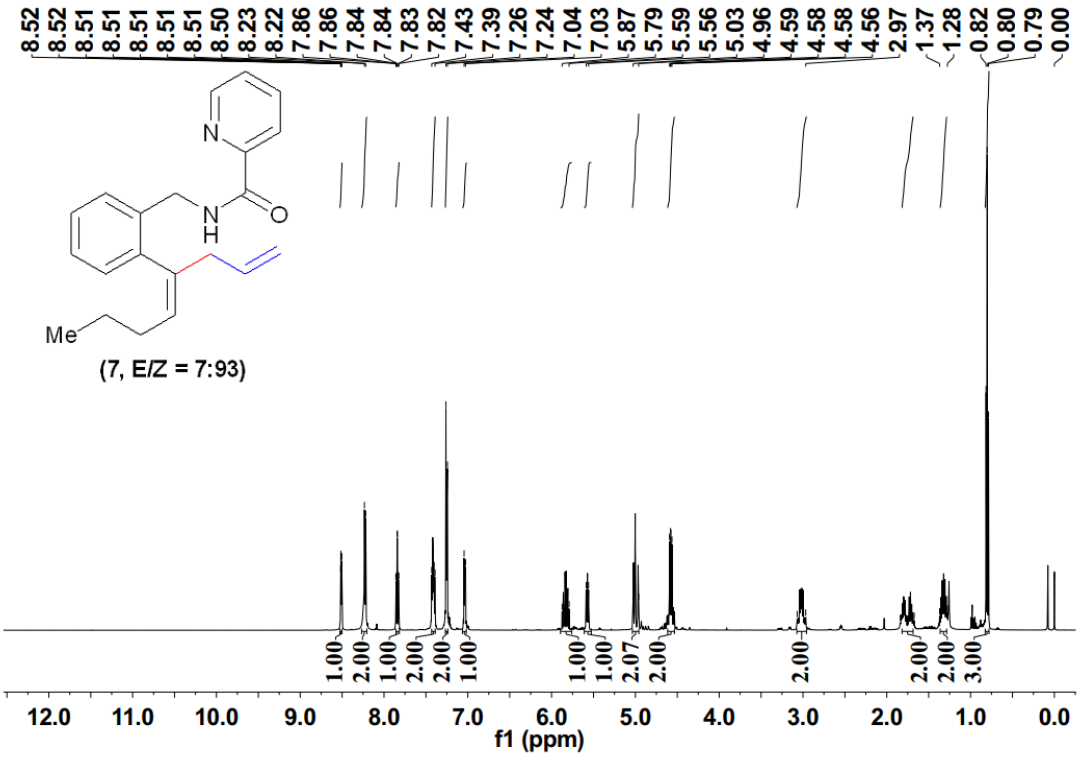

8.5300
8.5210
8.2406
8.2383
8.2361
8.2250
8.2227
7.8538
7.8503
7.4353
7.4317
7.4288
7.4221
7.4192
7.4162
7.2716
7.2679
7.2659
7.2637
7.2611
7.2589
7.0460
5.5848
4.9789
4.9753
4.9448
4.9412
4.9378
4.9212
4.6359
4.6234
4.5428
4.5313
1.0672
1.0524
1.0376
Et
O
N
H
N
(5f, E/Z > 99:1)
1.00
2.00
1.00
2.00
2.00
1.00
1.00
1.00
2.00
2.17
2.00
2.00
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

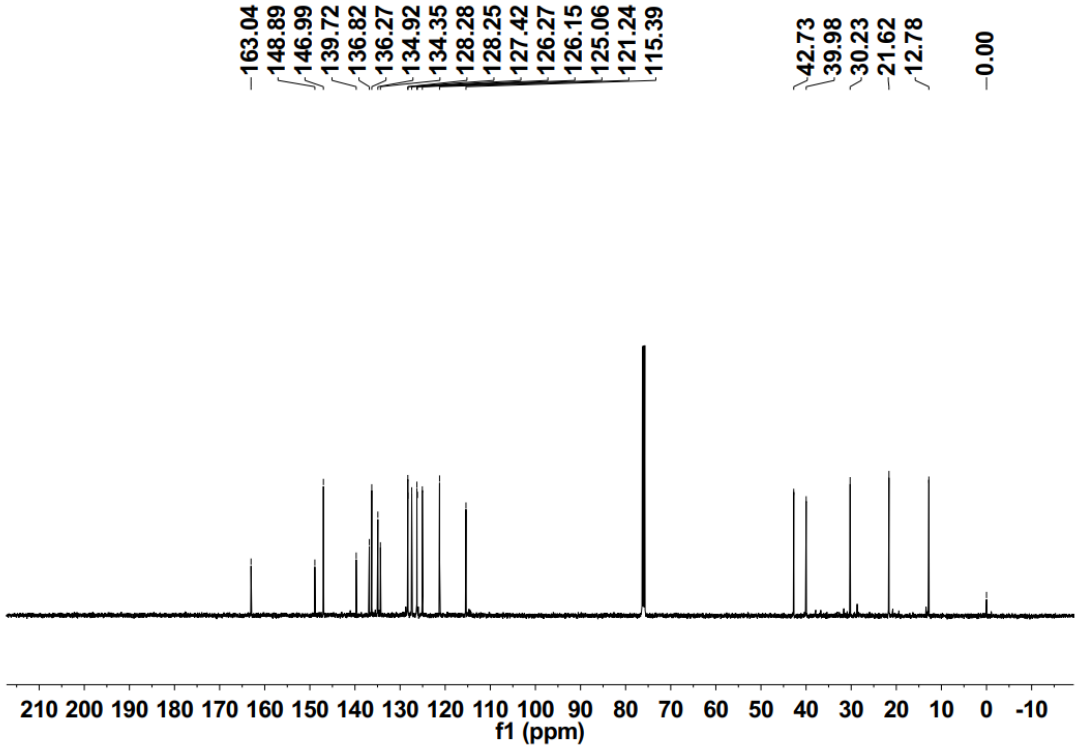

163.0959
148.8766
147.0124
141.3610
139.6372
136.2823
136.0087
134.3312
128.1709
127.2964
126.2438
125.0799
123.1012
121.2490
113.5682
39.8900
32.3776
30.9873
11.5760
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

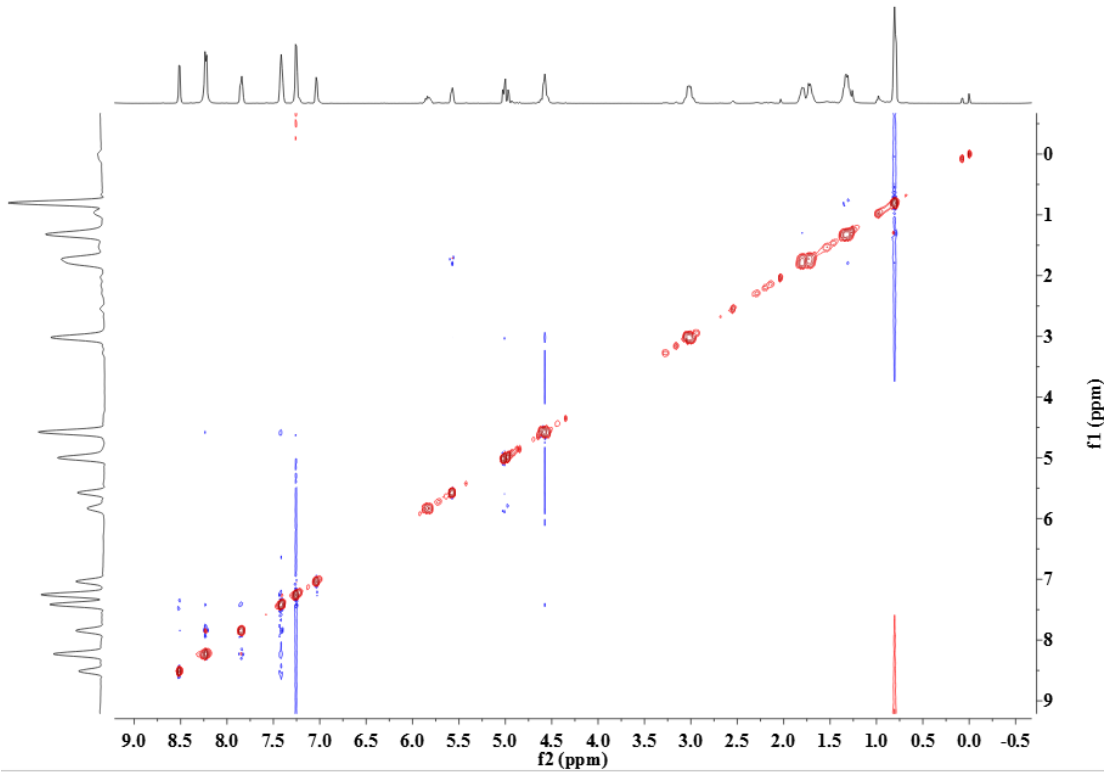

8.45
8.45
8.44
8.15
8.14
7.81
7.80
7.37
7.37
7.36
7.36
7.35
7.35
7.27
7.27
7.26
7.25
7.24
7.24
7.22
7.20
6.35
6.34
6.32
5.06
5.05
5.02
5.01
4.44
4.43
4.41
4.40
2.76
2.75
2.75
2.74
Ph
O
N
H
N
1.00
1.00
0.83
1.00
1.00
3.00
4.01
2.18
1.00
1.00
2.00
2.00
2.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

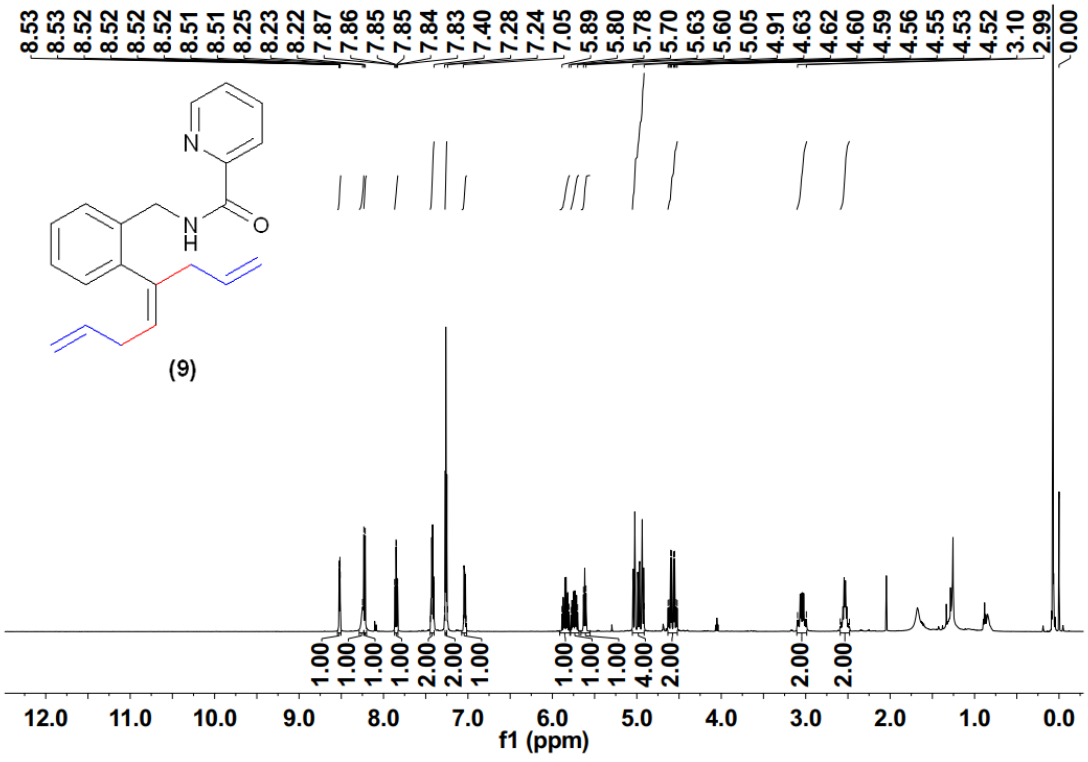
(5g, *E*/*Z* > 99:1)

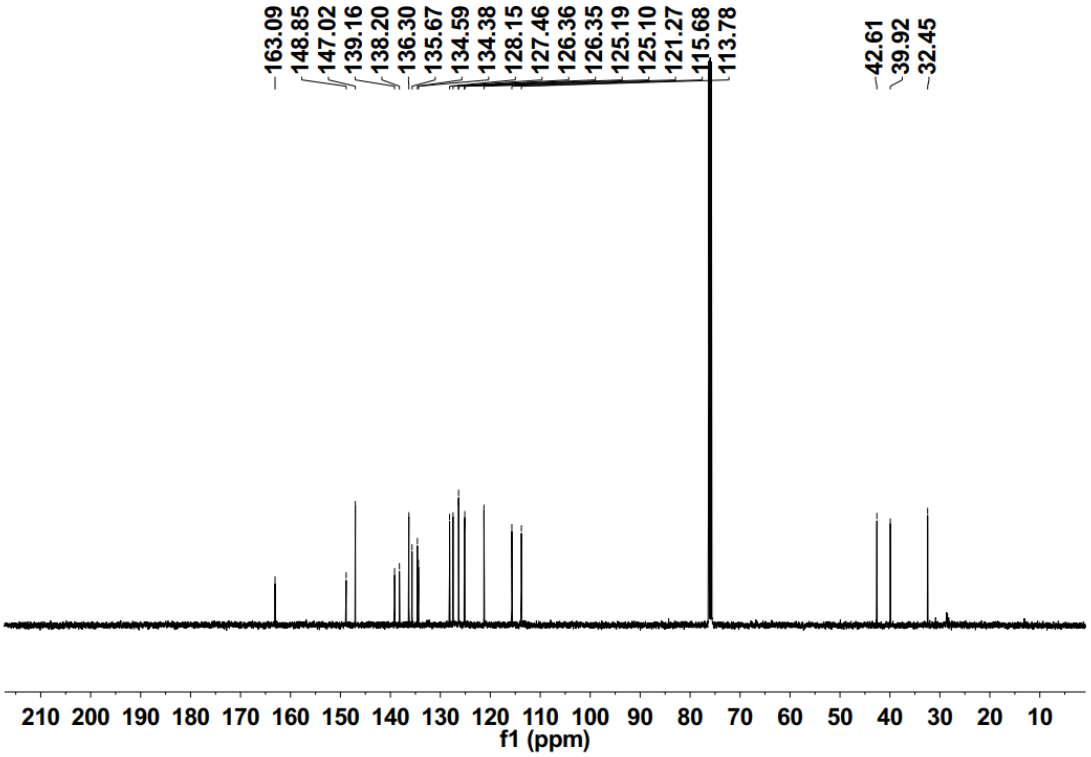

163.92
149.82
147.88
140.85
140.72
138.68
137.18
136.54
136.38
130.54
129.14
128.46
127.97
127.59
127.58
127.19
126.21
125.97
122.15
115.30
41.27
34.23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

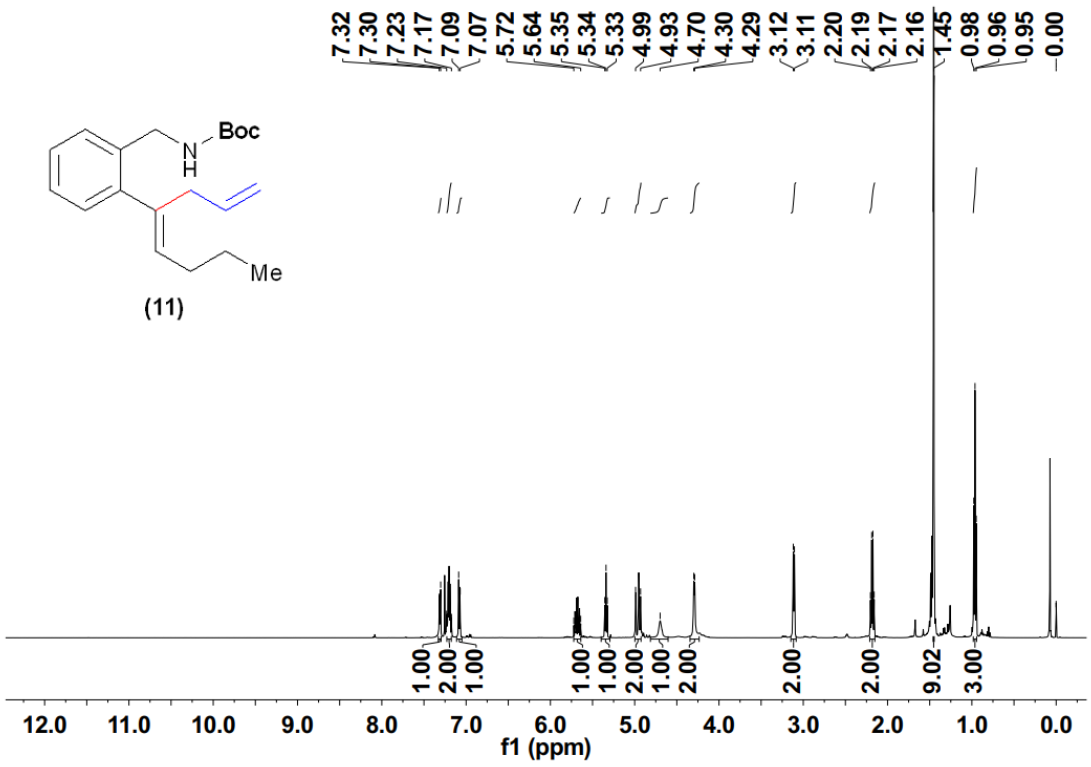

8.52 8.52 8.51 8.51 8.51 8.51 8.51 8.50 8.23 8.22 7.86 7.86 7.84 7.84 7.83 7.82 7.43 7.39 7.26 7.24 7.04 7.03 5.87 5.79 5.59 5.56 5.03 4.96 4.59 4.58 4.58 4.56 2.97 1.37 1.28 0.82 0.80 0.79 0.00
N
N
H
O
Me
(7, E/Z = 7:93)
1.00 2.00 1.00 2.00 2.00 1.00 1.00 1.00 2.07 2.00 2.00 2.00 2.00 3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

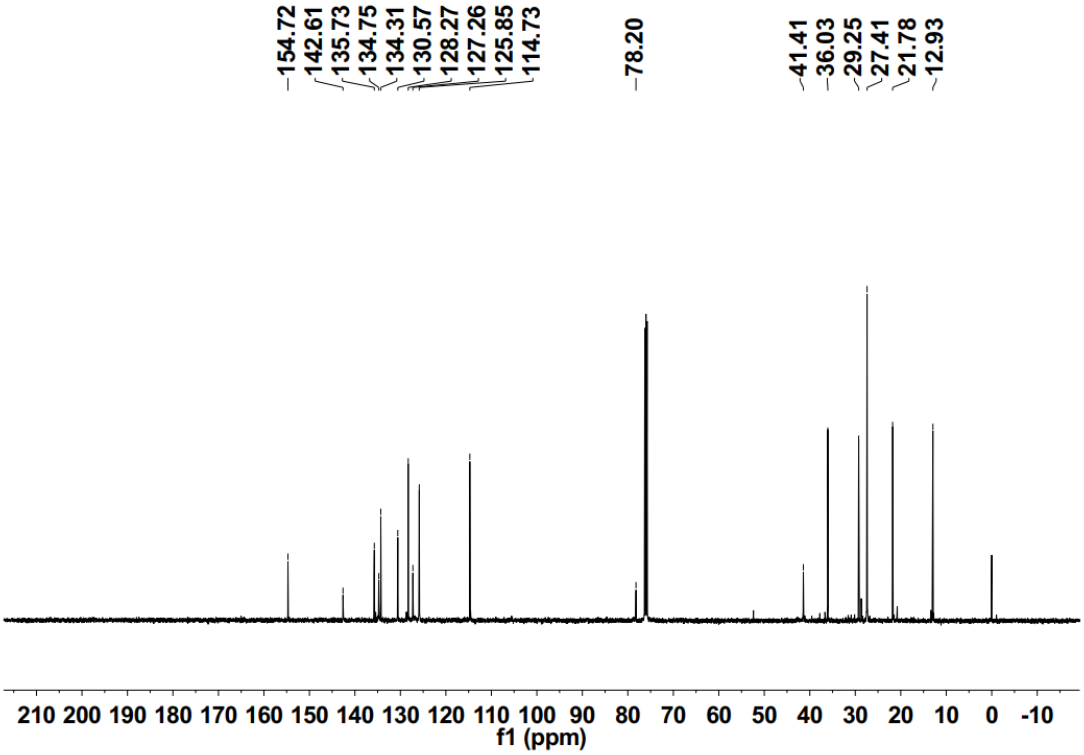

163.04 148.89 146.99 139.72 136.82 136.27 134.92 134.35 128.28 128.25 127.42 126.27 126.15 125.06 121.24 115.39
42.73 39.98 30.23 21.62 12.78
0.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

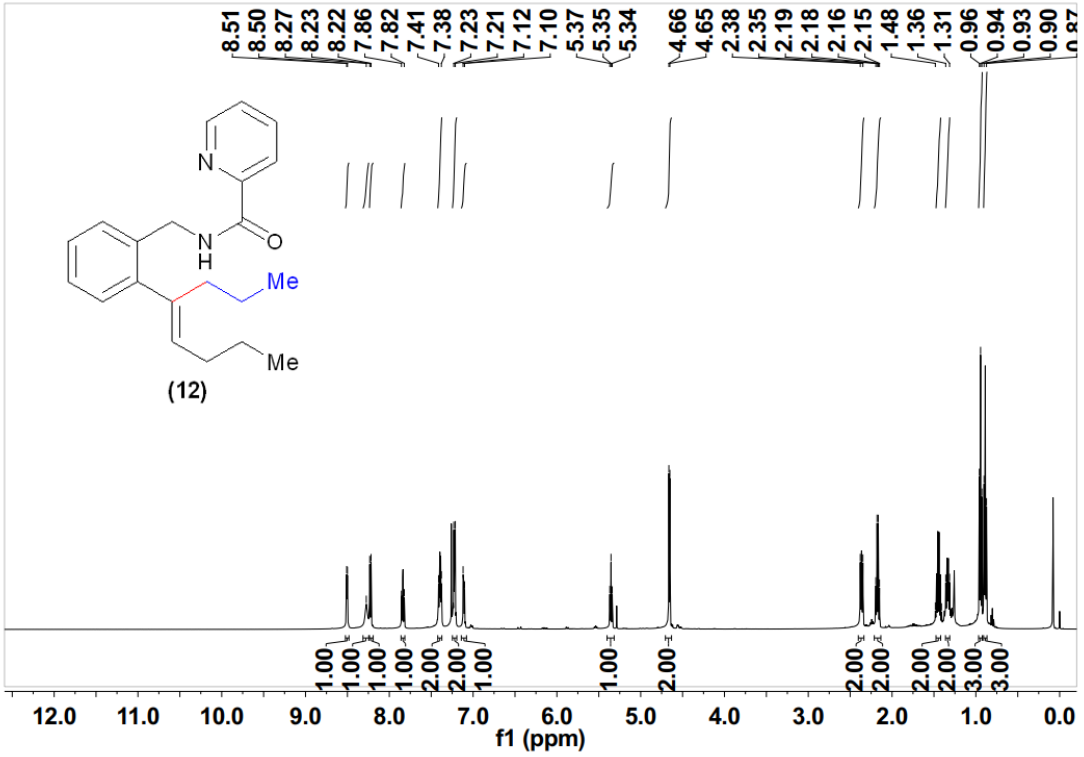

f2 (ppm)
f1 (ppm)

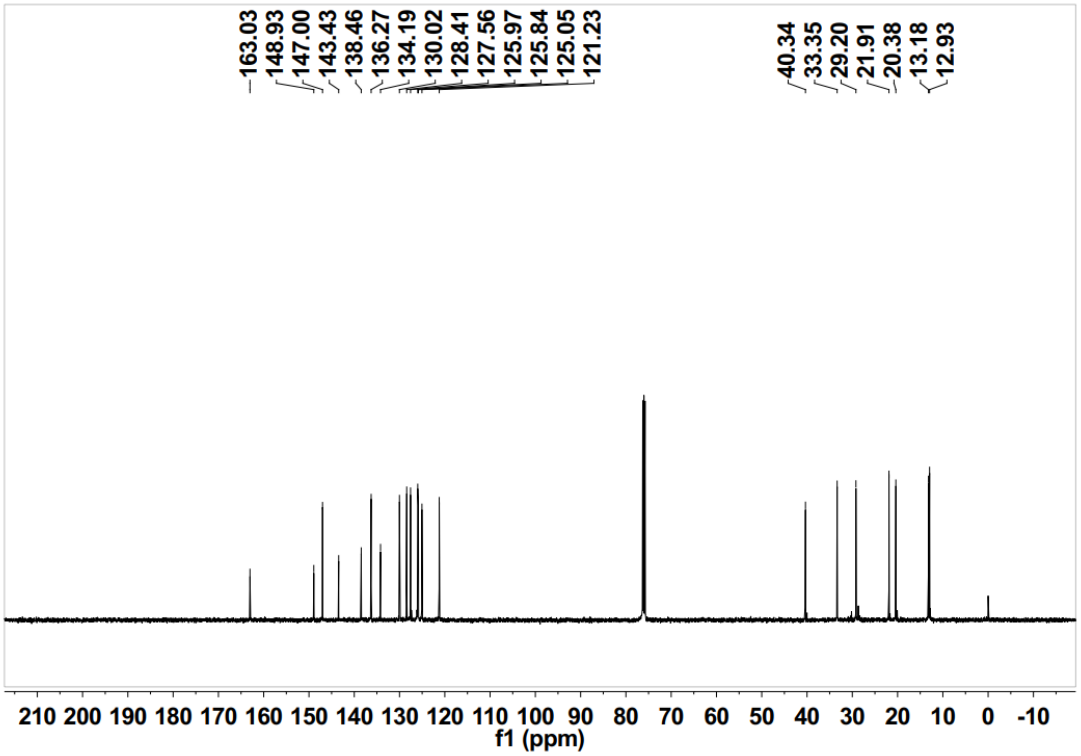

(9)
f1 (ppm)

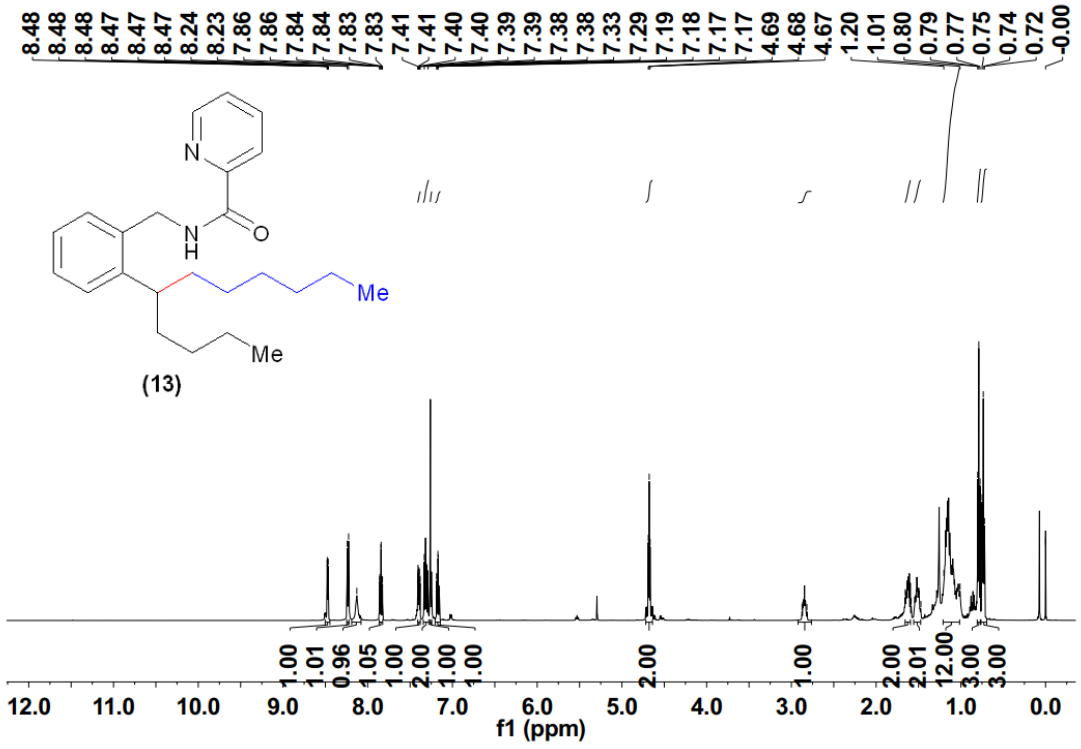

163.09
148.85
147.02
139.16
138.20
136.30
135.67
134.59
134.38
128.15
127.46
126.36
126.35
125.19
125.10
121.27
115.68
113.78
42.61
39.92
32.45
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

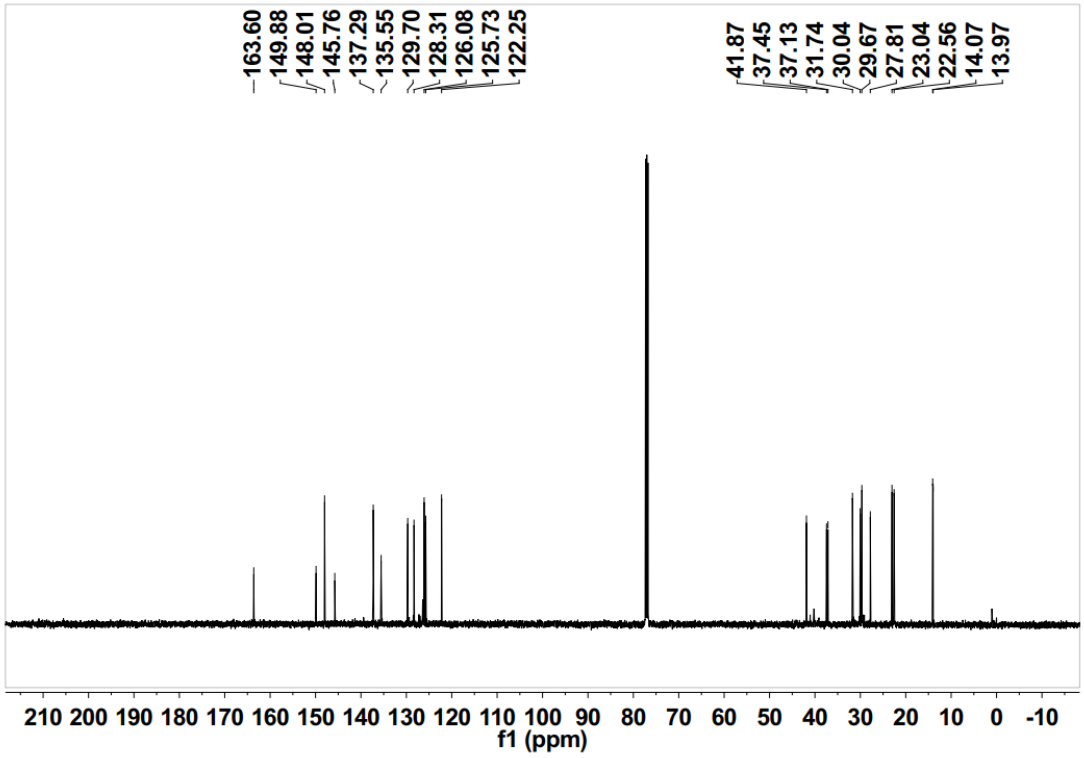

7.32
7.30
7.23
7.17
7.09
7.07
5.72
5.64
5.35
5.34
5.33
4.99
4.93
4.70
4.30
4.29
3.12
3.11
2.20
2.19
2.17
2.16
1.45
0.98
0.96
0.95
0.00
Boc
N
H
Me
(11)
1.00
2.00
1.00
1.00
1.00
2.00
1.00
2.00
2.00
2.00
9.02
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


154.72
142.61
135.73
134.75
134.31
130.57
128.27
127.26
125.85
114.73
78.20
41.41
36.03
29.25
27.41
21.78
12.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.51
8.50
8.27
8.23
8.22
7.86
7.82
7.41
7.38
7.23
7.21
7.12
7.10
5.37
5.35
5.34
4.66
4.65
2.38
2.35
2.19
2.18
2.16
2.15
1.48
1.36
1.31
0.96
0.94
0.93
0.90
N
N
H
O
Me
Me
(12)
1.00
1.00
1.00
1.00
2.00
2.00
1.00
1.00
2.00
2.00
2.00
2.00
2.00
3.00
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


163.03
148.93
147.00
143.43
138.46
136.27
134.19
130.02
128.41
127.56
125.97
125.84
125.05
121.23
40.34
33.35
29.20
21.91
20.38
13.18
12.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.48
8.48
8.48
8.47
8.47
8.47
8.24
8.23
7.86
7.86
7.84
7.84
7.83
7.83
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.38
7.33
7.29
7.19
7.18
7.17
7.17
4.69
4.68
4.67
1.20
1.01
0.80
0.79
0.77
0.75
0.74
0.72
-0.00
N
N
H
O
Me
Me
(13)
1.00
1.01
0.96
1.05
1.00
2.00
1.00
1.00
2.00
1.00
2.00
2.01
12.00
3.00
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


163.60
149.88
148.01
145.76
137.29
135.55
129.70
128.31
126.08
125.73
122.25
41.87
37.45
37.13
31.74
30.04
29.67
27.81
23.04
22.56
14.07
13.97
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)