Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Visible Light Induced Synthesis of (*Z*)-β-iodoenamides from *N*-Vinyl Amides Mediated by the Ion Pair Charge Transfer State

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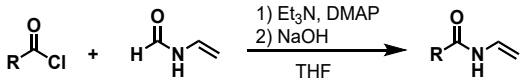
Contents

1. General Information

Unless otherwise noted, the solvents, reagents, and deuterated solvents were purchased from Aladdin, Aldrich, Adamas, and TCI without further purification. Column chromatography was performed with silica gel (Merck, 300-400 mesh). ^{1}H NMR were recorded on Bruker Avance (400 MHz) spectrometers with tetramethylsilane (TMS) as internal standard. Chemical shifts are reported in ppm from TMS. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants, integration. ^{13}C NMR spectra were recorded on Bruker Avance (101 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from TMS with the solvent resonance as the internal standard ($CDCl_3$: δ 77.0). HRMS was recorded on a commercial apparatus (ESI Source, TOF). Melting points were obtained by XT4A Micro Melting-point Measurement Instruments. A UV-3600 spectrophotometer was used as the light source for the UV-Vis data.

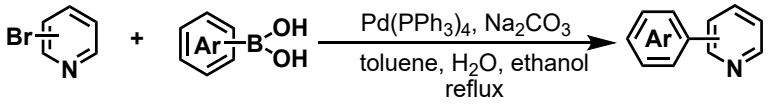
2. Experimental Procedures

General procedure for the synthesis of *N*-vinyl amides

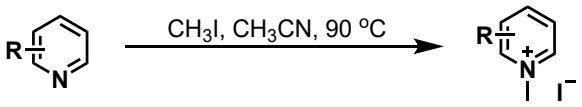


N-vinyl formamide (5 mmol, 1.0 eq), triethylamine (6 mmol, 1.2 eq), DMAP (5 mol%, 30.1 mg) and anhydrous THF (30 mL) were added to a three-necked round-bottomed flask equipped with an overhead stirrer, an addition funnel, and a nitrogen balloon. The mixture was cooled to 0 ℃ in an ice-water bath. Freshly distilled aryl chloride (5.75 mmol, 1.15 eq) was loaded into the addition funnel and slowly added at a rate such that temperature was maintained below 5 ℃ over 1 h. React for 3 h to 48 h. A solution of 5 N NaOH (15 mmol/3 mL H_2O) was then slowly added at 0-5 ℃ over 2 h. The water layer was removed and extracted with EtOAc. The organic layers were combined, washed with a solution of NaCl, and dried over anhydrous Na_2SO_4. After filtering off insoluble, the solvents were evaporated under reduced pressure using rotary evaporation to give the crude product, which purified by flash chromatography on silica gel with petroleum ether/ethyl acetate (PE: EA= 10:1- 3:1) to give a white solid. The solid was further dried in a vacuum oven.

General procedure for the synthesis of pyridinium iodides

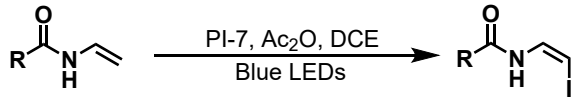


To a solution of arylboronic acid (2.6 mmol) in toluene (7 mL), ethanol (1.5 mL), and H_2O (7 mL) was added Na_2CO_3 (15.0 mmol) followed by $Pd(PPh_3)_4$ (0.06 mmol) and bromo-heterocycle (2.0 mmol) under argon in a two-necked flask. The mixture was refluxed overnight, and then cooled to room temperature. To the reaction mixture was added aqueous NH_4Cl (20 mL), extracted with EtOAc for three times, and dried over $MgSO_4$. After removal of the solvent under reduced pressure to afford the crude product, which was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to give corresponding *N*-heterocyclic compounds.



To a solution of *N*-heterocycle compounds (5.0 mmol, 1.0 equiv) in acetonitrile (5 mL, 1 M) was added CH_3I (2.84 g, 20.0 mmol, 4.0 equiv) under argon in a two-necked flask. The mixture was heated at 90°C for 16 h, and then cooled to room temperature. After removal of the solvent under reduced pressure to afford the crude product, which was purified by recrystallization in CH_3CN/EtOAc co-system.

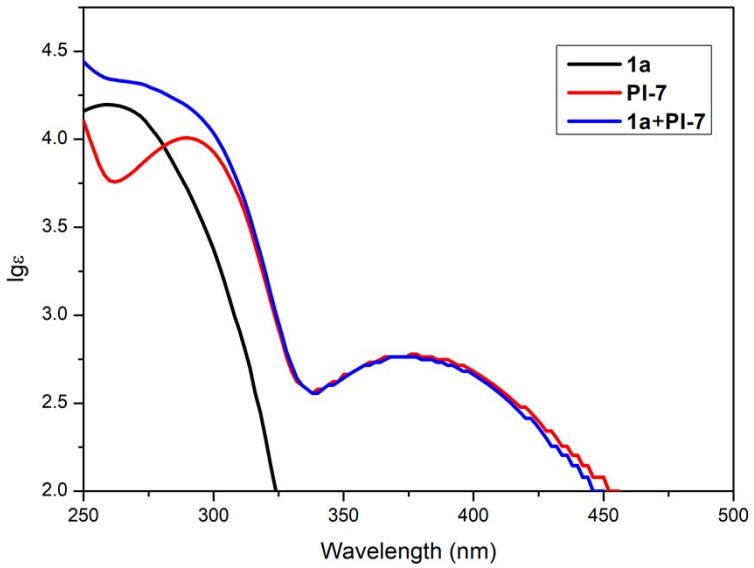
General procedure for the synthesis of (*Z*)-β-iodoenamides



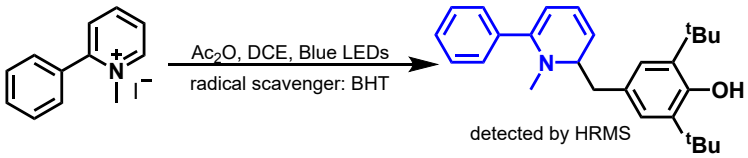
An oven-dried 25 mL Schlenk tube was sequentially charged with *N*-vinyl amide (0.2 mmol), acetic anhydride (1.0 equiv, 19 uL), **PI-7** (1.0 equiv, 59.4 mg) and DCE (3.0 mL). The resulting solution was stirred at room temperature under air and blue LED (λ_{max} = 445 nm) for 16 h. Then, the reaction mixture was filtered, and the solvent was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and toluene (PE: PhMe = 1:1) as eluent to afford the dihydroxazole derivatives.

3. UV−Vis Absorption Spectra

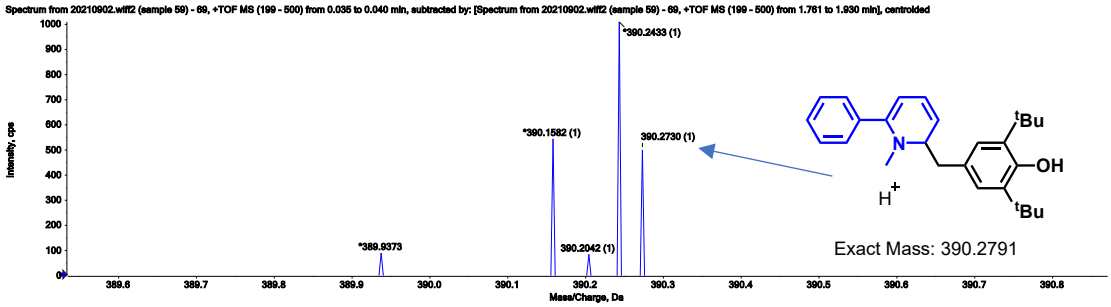
The UV−vis absorption spectra of **1a**, **PI-7, 1a+PI-7**. Measurements were carried out a DCE solution under air at room temperature: **1a** (5.0×10^{-5} M), **PI-7** (5.0×10^{-5} M), **1a** (5.0×10^{-5} M) + **PI-7** (5.0×10^{-5} M).

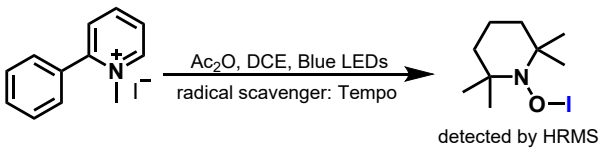


4. Trapping Radical Intermediates

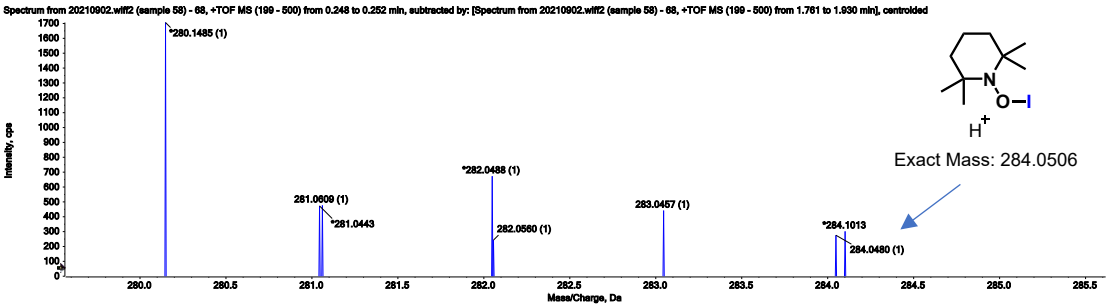


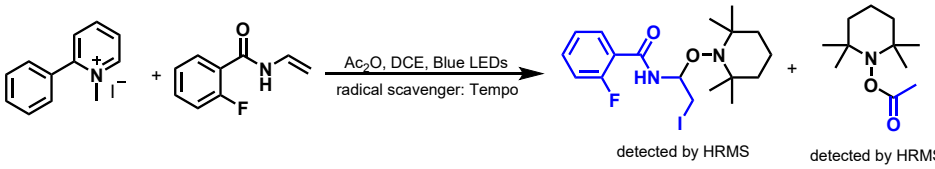
An oven-dried 25 mL Schlenk tube was sequentially charged with **PI-7** (0.2 mmol, 59.4 mg), acetic anhydride (1.0 equiv, 19 uL), DCE (3.0 mL) and radical scavenger BHT (2.0 equiv, 88.0 mg). The resulting solution was stirred at room temperature under air and blue LED (λ_{max} = 445 nm) for 12 h. Then, the reaction mixture was analyzed by HRMS.



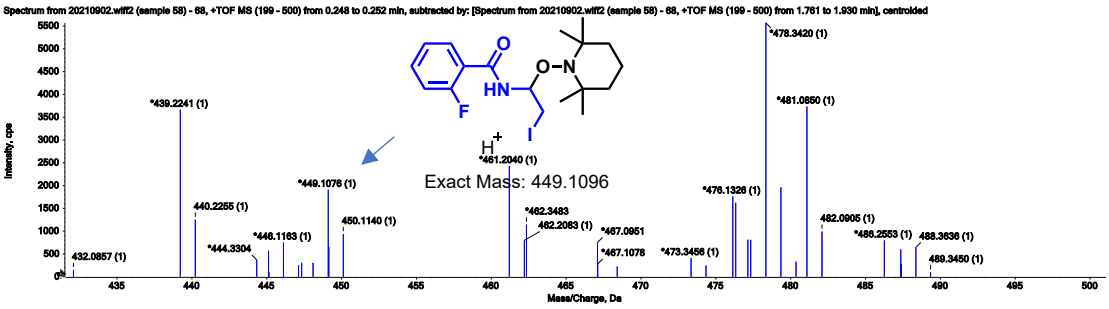


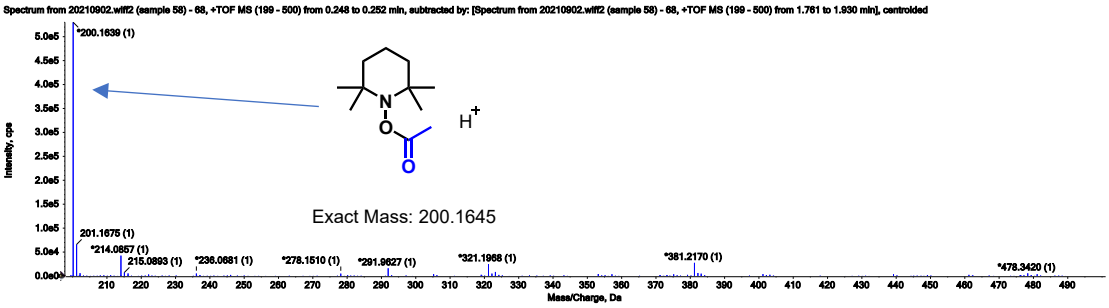
An oven-dried 25 mL Schlenk tube was sequentially charged with **PI-7** (0.2 mmol, 59.4 mg), acetic anhydride (1.0 equiv, 19 uL), DCE (3.0 mL) and radical scavenger Tempo (2.0 equiv, 62.8 mg). The resulting solution was stirred at room temperature under air and blue LED (λ_{max} = 445 nm) for 12 h. Then, the reaction mixture was analyzed by HRMS.





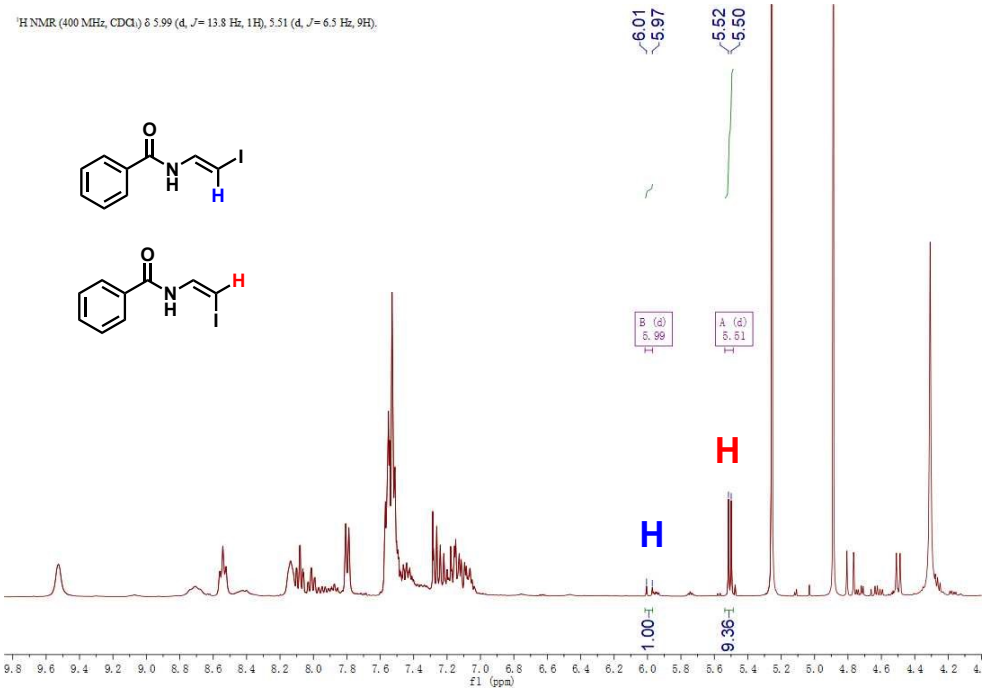
An oven-dried 25 mL Schlenk tube was sequentially charged with 2-fluoro-*N*-vinylbenzamide (0.2 mmol, 33 mg), acetic anhydride (1.0 equiv, 19 uL), **PI-7** (1.0 equiv, 59.4 mg), DCE (3.0 mL) and radical scavenger Tempo (2.0 equiv, 62.8 mg). The resulting solution was stirred at room temperature under air and blue LED (λ_{max} = 445 nm) for 12 h. Then, the reaction mixture was analyzed by HRMS.



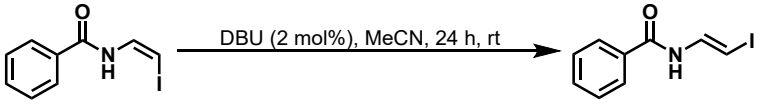


5. ^{1}H NMR analysis

An oven-dried 25 mL Schlenk tube was sequentially charged with 2-fluoro-*N*-vinylbenzamide (0.2 mmol, 33 mg), acetic anhydride (1.0 equiv, 19 uL), **PI-7** (1.0 equiv, 59.4 mg) and DCE (3.0 mL). The resulting solution was stirred at room temperature under air and blue LED (λ_{max} = 445 nm) for 12 h. Then, the reaction mixture was analyzed by ^{1}H NMR.



6. Derivatization of (*Z*)-β-iodoenamides

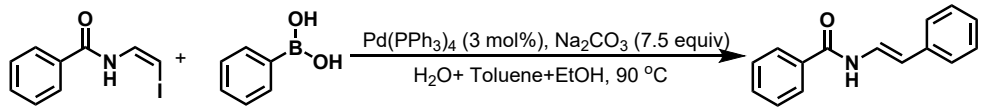


2g **3**, 33%

An oven-dried 25 mL Schlenk tube equipped with magnetic stirring bar was sequentially charged with (*Z*)-*N*-(2-iodovinyl) benzamide (0.1 mmol, 27.3 mg), DBU (2 mol%, 0.6 uL) and MeCN (1.0 mL). The resulting solution was stirred at room temperature under air for 24 h. Then, the reaction mixture was filtered, and the solvent

was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (PE: EA= 1:1) as eluent to afford (*E*)-*N*-(2-iodovinyl) benzamide **3** (9.0 mg, 33%).

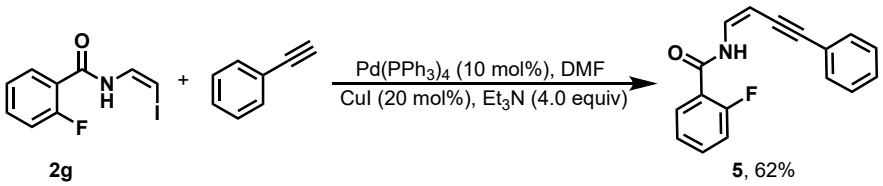
(*E*)-*N*-(2-iodovinyl) benzamide **3**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.81 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.68 (dd, *J* = 13.8, 10.5 Hz, 1H), 7.62 – 7.55 (m, 1H), 7.54 – 7.45 (m, 2H), 5.87 (d, *J* = 13.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.6, 133.9, 132.8, 132.4, 128.9, 127.1, 57.4.



2g **4**, 39%

To a solution of phenylboric acid (1.5 equiv, 18.3 mg) in toluene (0.2 mL), ethanol (0.5 mL), and H_2O (0.2 mL) was added Na_2CO_3 (7.5 equiv, 79.5 mg) followed by $Pd(PPh_3)_4$ (3 mol%, 3.4 mg) and (*Z*)-*N*-(2-iodovinyl) benzamide (0.1 mmol, 27.3 mg) under argon in a two-necked flask. The resulting solution was stirred at 90 ℃ for 16 h. Then, the reaction mixture was filtered, and the solvent was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (PE: EA= 5:1) as eluent to afford (*E*)-*N*-styrylbenzamide **4** (8.7 mg, 39%).

(*E*)-*N*-styrylbenzamide **4**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 10.2 Hz, 1H), 7.93 – 7.86 (m, 2H), 7.77 (dd, *J* = 14.6, 10.8 Hz, 1H), 7.58 (ddd, *J* = 6.4, 3.8, 1.3 Hz, 1H), 7.55 – 7.48 (m, 2H), 7.39 (d, *J* = 7.4 Hz, 2H), 7.33 (t, *J* = 7.7 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 6.29 (d, *J* = 14.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.5, 136.0, 133.5, 132.2, 128.8, 128.7, 127.1, 126.8, 125.7, 123.0, 113.6.

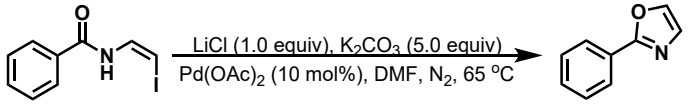


2g **5**, 62%

Treat 0.1 M solution of (*Z*)-2-fluoro-*N*-(2-iodovinyl) benzamide (0.1 mmol, 29.1 mg) (0.1 mmol, 27.3 mg) in DMF with $Pd(PPh_3)_4$ (10 mol%, 11.3 mg) and stir the suspension at room temperature for 10 min. Add the phenyl acetylene (1.0 equiv, 11 uL), CuI (20 mol%, 3.8 mg) and Et_3N (4.0 equiv, 55 uL) sequentially and stir the resulting mixture at room temperature for 72 h. Then, quenching the mixture with distilled water

and extract with Et_2O. The organic layers dried over anhydrous Na_2SO_4, and then was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (PE: EA= 5:1) as eluent to afford (*Z*)-2-fluoro-*N*-(4-phenylbut-1-en-3-yn-1-yl) benzamide **5** (16.3 mg, 62%).

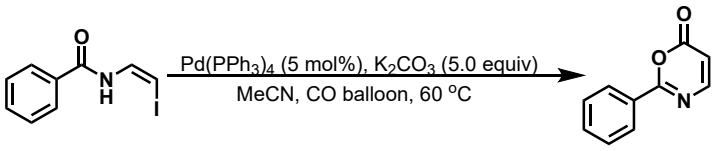
(*Z*)-2-fluoro-*N*-(4-phenylbut-1-en-3-yn-1-yl) benzamide **5**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.44 – 9.25 (m, 1H), 8.23 (td, *J* = 8.0, 1.8 Hz, 1H), 7.61 – 7.49 (m, 4H), 7.42 – 7.32 (m, 4H), 7.24 (ddd, *J* = 12.6, 8.3, 0.8 Hz, 1H), 5.22 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1 (d, *J* = 247.9 Hz). 160.1 (d, *J* = 3.5 Hz), 134.4 (d, *J* = 9.7 Hz), 132.6 (d, *J* = 1.7 Hz), 132.1, 131.3, 128.5, 128.4, 125.2 (d, *J* = 3.2 Hz), 123.1, 119.5 (d, *J* = 10.5 Hz), 116.5, 116.2, 97.6, 90.7, 83.6.



2g **6**, 54%

(*Z*)-*N*-(2-iodovinyl) benzamide (0.1 mmol, 29.1 mg), LiCl (1.0 equiv, 4.3 mg), K_2CO_3 (5.0 equiv, 69 mg) and $Pd(OAc)_2$ (10 mol%, 2.3 mg) dissolved in DMF. The solution was degassed, placed under N_2 atmosphere, and heated to 65 ℃ for 24 h. Then, quenching the mixture with distilled water and extract with Et_2O. The organic layers dried over anhydrous Na_2SO_4, and then was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (PE: EA= 5:1) as eluent to afford 2-phenyl oxazole **6** (7.9 mg, 54%).

2-phenyl oxazole **6**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.02 (m, 2H), 7.73 – 7.70 (m, 1H), 7.51 – 7.43 (m, 3H), 7.24 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.0, 138.6, 130.4, 128.8, 128.4, 127.5, 126.4.



2g **7**, 45%

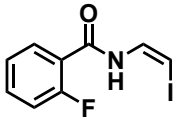
An oven-dried 25 mL Schlenk tube equipped with magnetic stirring bar and a CO balloon was sequentially charged with (*Z*)-*N*-(2-iodovinyl) benzamide (0.1 mmol, 27.3 mg), K_2CO_3 (5.0 equiv, 69 mg) and $Pd(PPh_3)_4$ (10 mol%, 5.7 mg) and CH_3CN (1.0 mL). The resulting solution was stirred at 60 ℃ under air for 24 h. Then, the reaction

mixture was filtered, and the solvent was evaporated in vacuum. The crude product was purified by flash column chromatography on silica gel using petroleum ether and ethyl acetate (PE: EA= 5:1) as eluent to afford 2-phenyl-6H-1,3-oxazin-6-one **7** (7.8 mg, 45%).

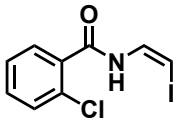
2-phenyl-6H-1,3-oxazin-6-one **7**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.23 (m, 2H), 7.86 (d, *J* = 6.8 Hz, 1H), 7.67 – 7.60 (m, 1H), 7.57 – 7.50 (m, 2H), 6.25 (d, *J* = 6.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 158.4, 154.7, 133.5, 129.6, 128.9, 128.6, 109.6.

7. Characterization Data of Products

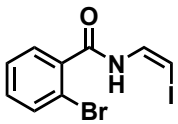
(1) (*Z*)-β-iodoenamides



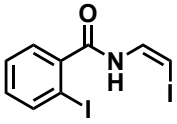
(*Z*)-2-fluoro-*N*-(2-iodovinyl) benzamide (2a): Light yellow solid. M. P. 80.2 -82.0 ℃. (44.0 mg, yield: 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (s, 1H), 8.17 (td, *J* = 7.9, 1.8 Hz, 1H), 7.59 (dddd, *J* = 13.7, 7.3, 5.9, 2.6 Hz, 2H), 7.35 – 7.29 (m, 1H), 7.26 – 7.16 (m, 1H), 5.56 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0 (d, *J* = 248.6 Hz), 160.5 (d, *J* = 3.3 Hz), 134.5 (d, *J* = 9.6 Hz), 132.4 (d, *J* = 1.6 Hz), 130.8, 125.2 (d, *J* = 3.2 Hz), 119.4 (d, *J* = 10.6 Hz), 116.4 (d, *J* = 24.8 Hz), 61.5. HRMS (ESI) m/z calcd. for C_9H_7FINO [M+H]+: 291.9629, found: 291.9633.



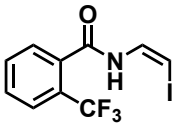
(*Z*)-2-chloro-*N*-(2-iodovinyl) benzamide (2b): White solid. M. P. 80.4 -81.0 ℃. (43.8 mg, yield: 73%).^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (s, 1H), 7.89 (dd, *J* = 7.2, 1.3 Hz, 1H), 7.61 (dd, *J* = 10.8, 6.5 Hz, 1H), 7.51 – 7.37 (m, 3H), 5.59 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.1, 132.5, 132.5, 131.5, 131.0, 130.8, 130.7, 127.4, 61.4. HRMS (ESI) m/z calcd. for C_9H_7ClINO [M+H]+: 307.9334, found: 307.9338.



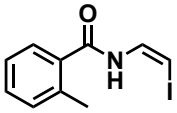
(*Z*)-2-bromo-*N*-(2-iodovinyl) benzamide (2c): Light yellow solid. M. P. 97.3 -98.2 ℃. (46.7 mg, yield: 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.73 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.68 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.58 (dd, *J* = 10.9, 6.5 Hz, 1H), 7.45 (td, *J* = 7.5, 1.2 Hz, 1H), 7.38 (td, *J* = 7.7, 1.8 Hz, 1H), 5.59 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 135.4, 134.0, 132.4, 130.7, 130.5, 127.8, 119.5, 61.4. HRMS (ESI) m/z calcd. for C_9H_7BrINO [M+H]+: 351.8828, found: 351.8830.



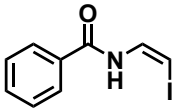
(*Z*)-2-iodo-*N*-(2-iodovinyl) benzamide (2d): Light yellow solid. M. P. 103.8 -104.5 ℃. (42.0 mg, yield: 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.74 (d, *J* = 6.9 Hz, 1H), 7.59 – 7.50 (m, 2H), 7.47 (td, *J* = 7.6, 1.0 Hz, 1H), 7.20 (td, *J* = 7.7, 1.7 Hz, 1H), 5.59 (d, *J* = 6.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 140.6, 140.0, 132.2, 130.3, 129.0, 128.4, 92.4, 61.6. HRMS (ESI) m/z calcd. for $C_9H_7I_2NO$ [M+H]+: 399.8690, found: 399.8686.



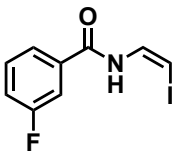
(*Z*)-*N*-(2-iodovinyl)-2-(trifluoromethyl) benzamide (2e): Yellow solid. M. P. 127.7 -128.3 ℃. (38.1 mg, yield: 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.5 Hz, 1H), 7.74 – 7.60 (m, 4H), 7.54 (dd, *J* = 10.9, 6.5 Hz, 1H), 5.59 (d, *J* = 6.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 134.1 (q, *J* = 3.9 Hz), 132.3, 130.8, 130.3, 128.8, 127.7 (q, *J* = 32.2 Hz), 126.7 (q, *J* = 5.0 Hz), 123.4 (q, *J* = 273.7 Hz), 61.8. HRMS (ESI) m/z calcd. for $C_{10}H_7F_3INO$ [M+H]+: 341.9597, found: 341.9597.



(*Z*)-*N*-(2-iodovinyl)-2-methylbenzamide (2f): Brownish yellow oil. (34.9 mg, yield: 61%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.49 – 7.40 (m, 2H), 7.37 – 7.30 (m, 1H), 7.25 – 7.19 (m, 2H), 5.41 (d, *J* = 6.3 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5, 136.3, 133.0, 130.6, 130.1, 129.6, 126.0, 125.1, 59.7, 19.3. HRMS (ESI) m/z calcd. for $C_{10}H_{10}INO$ [M+H]+: 287.9880, found: 287.9882.

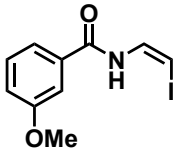


(*Z*)-*N*-(2-iodovinyl) benzamide (2g): Yellow solid. M. P. 78.3 -80.1 ℃. (28.0 mg, yield: 51%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 10.9 Hz, 1H), 7.94 – 7.79 (m, 3H), 7.62 (dd, *J* = 10.6, 4.2 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 132.7, 132.7, 130.7, 129.0, 127.3, 61.1. HRMS (ESI) m/z calcd. for C_9H_8INO [M+H]+: 273.9723, found: 273.9725.

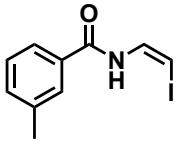


(*Z*)-3-fluoro-*N*-(2-iodovinyl) benzamide (2h): White solid. M. P. 46.3 -48.0 ℃. (23.5 mg, yield: 41%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.62 (dd, *J* = 13.8, 4.8 Hz, 2H), 7.53 (dt, *J* = 14.0, 6.0 Hz, 2H), 7.36 – 7.29 (m, 1H), 5.56 (d, *J* = 6.4 Hz, 1H). ^{13}C NMR (101 MHz,

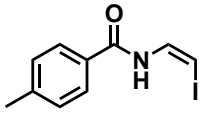
$CDCl_3$) δ 162.9 (d, *J* = 248.8 Hz), 162.9, 135.0 (d, *J* = 6.9 Hz), 130.7 (d, *J* = 7.9 Hz), 130.4, 122.6 (d, *J* = 3.1 Hz), 119.7 (d, *J* = 21.3 Hz), 114.8 (d, *J* = 23.1 Hz), 61.8. HRMS (ESI) m/z calcd. for C_9H_7FINO [M+H]+: 291.9629, found: 291.9630.



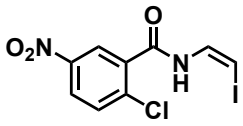
(*Z*)-*N*-(2-iodovinyl)-3-methoxybenzamide (2i): Brown oil. (27.0 mg, yield: 45%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 9.0 Hz, 1H), 7.54 (dd, *J* = 10.9, 6.4 Hz, 1H), 7.50 – 7.37 (m, 3H), 7.14 (ddd, *J* = 7.7, 2.5, 1.6 Hz, 1H), 5.52 (d, *J* = 6.4 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0, 160.1, 134.2, 130.6, 130.0, 118.8, 112.7, 61.1, 55.5. HRMS (ESI) m/z calcd. for $C_{10}H_{10}INO_2$ [M+H]+: 303.9829, found: 303.9830.



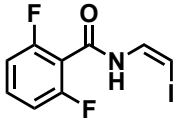
(*Z*)-*N*-(2-iodovinyl)-3-methylbenzamide (2j): Light yellow solid. M. P. 65.2 -66.1 ℃. (28.0 mg, yield: 49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.71 (s, 1H), 7.66 (m, 1H), 7.55 (dd, *J* = 10.9, 6.4 Hz, 1H), 7.45 – 7.39 (m, 2H), 5.51 (d, *J* = 6.4 Hz, 1H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 139.0, 133.4, 132.7, 130.7, 128.8, 128.1, 124.1, 60.8, 21.4. HRMS (ESI) m/z calcd. for $C_{10}H_{10}INO$ [M+H]+: 287.9880, found: 287.9881.



(*Z*)-*N*-(2-iodovinyl)-4-methylbenzamide (2k): White solid. M. P. 54.5 -55.7 ℃. (20.4 mg, yield: 36%).^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 10.9 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.55 (dd, *J* = 10.9, 6.4 Hz, 1H), 7.33 (d, *J* = 7.9 Hz, 2H), 5.49 (d, *J* = 6.4 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 143.4, 130.8, 129.9, 129.6, 127.3, 60.6, 21.6. HRMS (ESI) m/z calcd. for $C_{10}H_{10}INO$ [M+H]+: 287.9880, found: 287.9882.

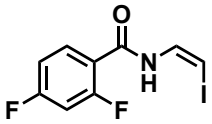


(*Z*)-2-chloro-*N*-(2-iodovinyl)-5-nitrobenzamide (2l): Yellow solid. M. P. 70.3 -71.9 ℃. (28.6 mg, yield: 41%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (d, *J* = 2.7 Hz, 1H), 8.42 (d, *J* = 8.3 Hz, 1H), 8.32 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.60 (dd, *J* = 10.7, 6.5 Hz, 1H), 5.71 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 146.8, 137.5, 134.0, 132.0, 130.3, 126.7, 126.6, 63.2. HRMS (ESI) m/z calcd. for $C_9H_6ClIN_2O_3$ [M+H]+: 352.9184, found: 352.9186.

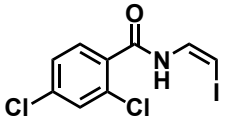


(*Z*)-2,6-difluoro-*N*-(2-iodovinyl) benzamide (2m): Orange oil. (38.1

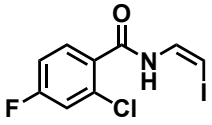
mg, yield: 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.56 (dd, *J* = 10.8, 6.5 Hz, 1H), 7.49 (tt, *J* = 8.4, 6.3 Hz, 1H), 7.10 – 7.01 (m, 3H), 5.59 (d, *J* = 6.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6 (dd, *J* = 254.9, 6.0 Hz), 157.6, 133.1 (t, *J* = 10.7 Hz), 130.1, 112.5 (d, *J* = 26.2 Hz), 112.5 (dd, *J* = 18.0, 2.6 Hz), 61.9. HRMS (ESI) m/z calcd. for $C_9H_6F_2INO$ [M+H]+: 309.9535, found: 309.9531.



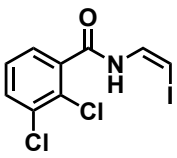
(*Z*)-2,4-difluoro-*N*-(2-iodovinyl) benzamide (2n): White solid. M. P. 68.6 -69.4 ℃. (45.1 mg, yield: 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (s, 1H), 8.22 (td, *J* = 8.9, 6.5 Hz, 1H), 7.67 – 7.55 (m, 1H), 7.12 – 7.02 (m, 1H), 6.96 (ddd, *J* = 12.2, 8.4, 2.4 Hz, 1H), 5.58 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.5 (dd, *J* = 257.4, 13.2 Hz), 161.4 (dd, *J* = 251.0, 12.5 Hz), 159.6 (d, *J* = 3.6 Hz), 134.3 (dd, *J* = 10.4, 3.4 Hz), 130.7, 116.0 (dd, *J* = 11.0, 3.7 Hz), 112.9 (dd, *J* = 21.3, 3.3 Hz), 104.7 (dd, *J* = 28.8, 25.9 Hz), 61.7. HRMS (ESI) m/z calcd. for $C_9H_6F_2INO$ [M+H]+: 309.9535, found: 309.9531.



(*Z*)-2,4-dichloro-*N*-(2-iodovinyl) benzamide (2o): Brown solid. M. P. 53.2 -53.4 ℃. (42.7 mg, yield: 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.59 (dd, *J* = 10.8, 6.5 Hz, 1H), 7.51 (d, *J* = 2.0 Hz, 1H), 7.41 (dd, *J* = 8.4, 2.0 Hz, 1H), 5.62 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.1, 138.3, 132.7, 131.8, 130.8, 130.6, 127.9, 61.9. HRMS (ESI) m/z calcd. for $C_9H_6Cl_2INO$ [M+H]+: 341.8944, found: 341.8946.

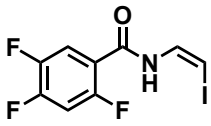


(*Z*)-2-chloro-4-fluoro-*N*-(2-iodovinyl) benzamide (2p): White solid. M. P. 70.8 -71.2 ℃. (36.4 mg, yield: 56 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 5.9 Hz, 1H), 7.96 (dd, *J* = 8.7, 6.1 Hz, 1H), 7.60 (dd, *J* = 10.7, 6.5 Hz, 1H), 7.23 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.18 – 7.10 (m, 1H), 5.60 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.9 (d, *J* = 256.5 Hz), 162.1, 133.6 (d, *J* = 9.5 Hz), 132.4 (d, *J* = 10.8 Hz), 130.7, 128.7 (d, *J* = 3.6 Hz), 118.2 (d, *J* = 25.2 Hz), 115.0 (d, *J* = 21.3 Hz), 61.7. HRMS (ESI) m/z calcd. for $C_9H_6FClINO$ [M+H]+: 325.9239, found: 325.9241.

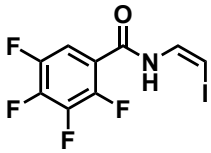


(*Z*)-2,3-dichloro-*N*-(2-iodovinyl) benzamide (2q): White solid. M. P. 65.4 -65.8 ℃. (38.5 mg, yield: 57 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ

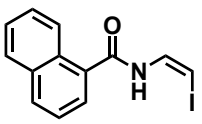
8.16 (d, *J* = 7.7 Hz, 1H), 7.65 (ddd, *J* = 12.2, 7.9, 1.5 Hz, 2H), 7.56 (dd, *J* = 10.8, 6.5 Hz, 1H), 7.35 (t, *J* = 7.9 Hz, 1H), 5.62 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.9, 135.4, 134.4, 133.0, 130.4, 129.5, 128.8, 127.9, 62.1. HRMS (ESI) m/z calcd. for $C_9H_6Cl_2INO$ [M+H]+: 341.8944, found: 341.8946.



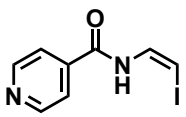
(*Z*)-2,4,5-trifluoro-*N*-(2-iodovinyl) benzamide (2r): White solid. M. P. 89.3 -90.3 ℃. (49.8 mg, yield: 76 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (s, 1H), 8.03 (ddd, *J* = 10.5, 8.9, 7.1 Hz, 1H), 7.64 – 7.53 (m, 1H), 7.10 (ddd, *J* = 11.1, 9.4, 6.0 Hz, 1H), 5.62 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 156.3 (ddd, *J* = 247.6, 9.8, 2.6 Hz), 152.9 (ddd, *J* = 231.7, 16.0, 13.8 Hz), 147.5 (ddd, *J* = 248.3, 12.5, 3.1 Hz), 130.5, 120.3 (ddd, *J* = 21.2, 3.6, 2.2 Hz), 116.3 (ddd, *J* = 13.5, 7.3, 4.3 Hz), 106.6 (dd, *J* = 31.3, 21.6 Hz), 62.4. HRMS (ESI) m/z calcd. for $C_9H_5F_3INO$ [M+H]+: 327.9441, found: 327.9446.



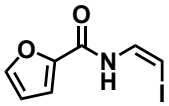
(*Z*)-2,3,4,5-tetrafluoro-*N*-(2-iodovinyl) benzamide (2s): White solid. M. P. 86.9 -88.0 ℃. (45.4 mg, yield: 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 7.91 – 7.79 (m, 1H), 7.58 (ddd, *J* = 10.0, 6.5, 3.1 Hz, 1H), 5.68 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.8 130.3, 113.3 (ddd, *J* = 21.4, 2.9, 1.8 Hz), 63.2. HRMS (ESI) m/z calcd. for $C_9H_4F_4INO$ [M+H]+: 345.9346, found: 345.9350.



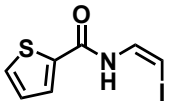
(*Z*)-*N*-(2-iodovinyl)-1-naphthamide (2t): White solid. M. P. 117.9 - 118.2 ℃. (41.3 mg, yield: 64 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.3 Hz, 1H), 8.03 (d, *J* = 8.3 Hz, 1H), 7.98 – 7.84 (m, 2H), 7.81 – 7.75 (m, 1H), 7.70 – 7.52 (m, 4H), 5.57 (d, *J* = 6.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 133.9, 132.2, 132.0, 130.7, 130.2, 128.5, 127.7, 126.8, 125.7, 125.2, 124.7, 61.1. HRMS (ESI) m/z calcd. for $C_{13}H_{10}INO$ [M+H]+: 323.9880, found: 323.9881.



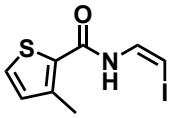
(*Z*)-*N*-(2-iodovinyl) isonicotinamide (2u): Light yellow oil. (23.5 mg, yield: 43 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (s, 2H), 8.08 (s, 1H), 7.72 (d, *J* = 5.5 Hz, 2H), 7.53 (dd, *J* = 10.8, 6.5 Hz, 1H), 5.65 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.4, 151.0, 139.8, 130.0, 120.9, 63.1. HRMS (ESI) m/z calcd. for $C_8H_7IN_2O$ [M+H]+: 274.9676, found: 274.9680.



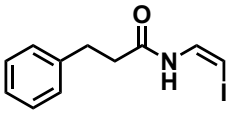
(*Z*)-*N*-(2-iodovinyl) furan-2-carboxamide (2v): Light yellow solid. M. P. 79.3 -81.0 ℃. (35.0 mg, yield: 67 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.58 (dd, *J* = 1.7, 0.7 Hz, 1H), 7.48 (dd, *J* = 11.1, 6.5 Hz, 1H), 7.27 (dd, *J* = 3.5, 0.6 Hz, 1H), 6.59 (dd, *J* = 3.5, 1.7 Hz, 1H), 5.51 (d, *J* = 6.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.0, 146.6,145.1, 129.7, 116.5, 112.8, 61.0. HRMS (ESI) m/z calcd. for $C_7H_6INO_2$ [M+H]+: 263.9516, found: 263.9519.



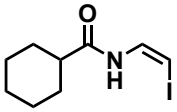
(*Z*)-*N*-(2-iodovinyl) thiophene-2-carboxamide (2w): Brown oil. (19.5 mg, yield: 35 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.67 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.63 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.48 (dd, *J* = 10.9, 6.4 Hz, 1H), 7.18 (dd, *J* = 5.0, 3.8 Hz, 1H), 5.50 (d, *J* = 6.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 137.2, 131.9, 130.3, 129.3, 128.1, 60.8. HRMS (ESI) m/z calcd. for C_7H_6INOS [M+H]+: 279.9288, found: 279.9290.



(*Z*)-*N*-(2-iodovinyl)-3-methylthiophene-2-carboxamide (2x): Brown oil. (24.5 mg, yield: 42 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.52 (dd, *J* = 10.8, 6.4 Hz, 1H), 7.43 (d, *J* = 5.0 Hz, 1H), 6.98 (d, *J* = 5.0 Hz, 1H), 5.49 (d, *J* = 6.4 Hz, 1H), 2.65 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 142.5, 132.6, 130.6, 130.1, 128.7, 60.4, 16.4. HRMS (ESI) m/z calcd. for C_8H_8INOS [M+H]+: 293.9444, found: 293.9446.

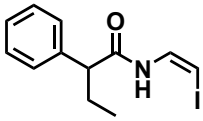


(*Z*)-*N*-(2-iodovinyl)-3-phenylpropanamide (2y): Brown oil. (25.3 mg, yield: 42 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 3H), 7.28 – 7.22 (m, 3H), 7.19 (s, 1H), 5.34 (d, *J* = 6.3 Hz, 1H), 3.04 (t, *J* = 7.7 Hz, 2H), 2.67 (t, *J* = 7.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 140.1, 130.2, 128.7, 128.3, 126.5, 59.6, 38.2, 31.0. HRMS (ESI) m/z calcd. for $C_{11}H_{12}INO$ [M+H]+: 302.0036, found: 302.0040.



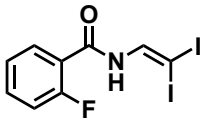
(*Z*)-*N*-(2-iodovinyl) cyclohexanecarboxamide (2z): Brown oil. (17.0 mg, yield: 31 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (s, 2H), 5.40 – 5.31 (m, 1H), 2.24 (tt, *J* = 11.6, 3.5 Hz, 1H), 1.96 (d, *J* = 13.2 Hz, 2H), 1.89 – 1.81 (m, 2H), 1.72 (dd, *J* = 7.8, 6.4 Hz, 1H), 1.51 (ddd, *J* = 24.3, 12.2, 2.9 Hz, 2H), 1.33 (tdd, *J* = 19.5, 8.7, 2.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.3, 130.4,

59.5, 45.3, 29.8, 25.6, 25.5. HRMS (ESI) m/z calcd. for $C_9H_{14}INO$ [M+H]+: 280.0193, found: 280.0197.

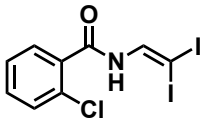


(*Z*)-*N*-(2-iodovinyl)-2-phenylbutanamide (2aa): Yellow oil. (19.5 mg, yield: 31 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.39 (m, 2H), 7.37 – 7.32 (m, 3H), 7.31 – 7.25 (m, 2H), 5.31 (d, *J* = 5.8 Hz, 1H), 3.39 (dd, *J* = 8.6, 6.6 Hz, 1H), 2.34 – 2.20 (m, 1H), 1.97 – 1.83 (m, 1H), 0.94 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 138.4, 130.3, 129.2, 128.2, 127.8, 60.1, 55.2, 25.9, 12.3. HRMS (ESI) m/z calcd. for $C_{12}H_{14}INO$ [M+H]+: 316.0193, found: 316.0195.

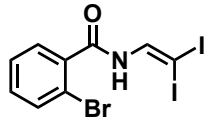
(2) β, β-diiodoenamides:



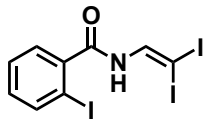
***N*-(2,2-diiodovinyl)-2-fluorobenzamide (2a'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 – 8.55 (m, 1H), 8.21 – 8.10 (m, 2H), 7.63 – 7.55 (m, 1H), 7.34 (td, *J* = 7.9, 1.0 Hz, 1H), 7.21 (ddd, *J* = 12.4, 8.3, 0.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161,0 (d, *J* = 248.5 Hz), 159.1 (d, *J* = 3.1 Hz), 137.7, 134.7 (d, *J* = 9.7 Hz), 132.6, 125.3 (d, *J* = 3.2 Hz), 119.0 (d, *J* = 10.2 Hz), 116.4 (d, *J* = 24.8 Hz). HRMS (ESI) m/z calcd. for $C_9H_6FI_2NO$ [M+H]+: 417.8596, found: 417.8596.



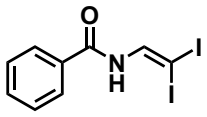
2-chloro-*N*-(2,2-diiodovinyl) benzamide (2b'): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 10.0 Hz, 1H), 8.11 (d, *J* = 10.7 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 3.8 Hz, 2H), 7.45 – 7.39 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.7, 137.6, 132.8, 132.0, 131.7, 131.0, 130.9, 127.5.



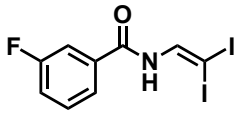
2-bromo-*N*-(2,2-diiodovinyl) benzamide (2c'): ^{1}H NMR (400 MHz, CDCl3) δ 8.09 (d, J = 10.8 Hz, 1H), 7.97 (d, J = 8.7 Hz, 1H), 7.73 (dd, J = 7.6, 1.8 Hz, 1H), 7.68 (dd, J = 7.9, 1.2 Hz, 1H), 7.46 (td, J = 7.5, 1.2 Hz, 1H), 7.39 (td, J = 7.7, 1.8 Hz, 1H). ^{13}C NMR (101 MHz, CDCl3) δ 162.7, 137.3, 134.9, 134.0, 132.6, 130.9, 127.9, 119.4.



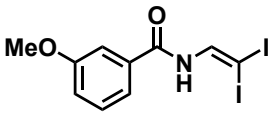
***N*-(2,2-diiodovinyl)-2-iodobenzamide (2d'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 10.9 Hz, 1H), 7.97 (d, *J* = 7.9 Hz, 1H), 7.58 – 7.45 (m, 3H), 7.25 – 7.18 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 140.6, 139.5, 137.1, 132.3, 129.1, 128.5, 92.3.



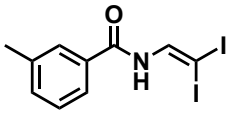
***N*-(2,2-diiodovinyl) benzamide (2g'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 10.9 Hz, 1H), 7.94 – 7.79 (m, 3H), 7.62 (dd, *J* = 10.6, 4.2 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7, 137.5, 132.8, 132.2, 129.0, 127.3.



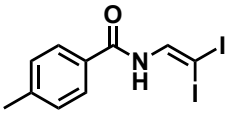
***N*-(2,2-diiodovinyl)-3-fluorobenzamide (2h'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 10.8 Hz, 1H), 7.80 (d, *J* = 9.2 Hz, 1H), 7.64 – 7.56 (m, 2H), 7.51 (td, *J* = 8.1, 5.8 Hz, 1H), 7.36 – 7.29 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.9 (d, *J* = 249.1 Hz), 161.5, 137.2, 134.5 (d, *J* = 6.9 Hz), 130.8 (d, *J* = 7.9 Hz), 122.6 (d, *J* = 3.1 Hz), 119.9 (d, *J* = 21.3 Hz), 114.8 (d, *J* = 23.2 Hz).



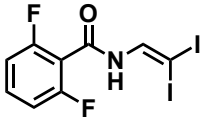
***N*-(2,2-diiodovinyl)-3-methoxybenzamide (2i'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 10.9 Hz, 1H), 7.83 (d, *J* = 10.0 Hz, 1H), 7.43 (dd, *J* = 9.8, 5.9 Hz, 2H), 7.40 – 7.35 (m, 1H), 7.15 (ddd, *J* = 8.1, 2.6, 1.0 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.6, 160.1, 137.4, 133.7, 130.0, 119.0, 118.8, 112.8, 55.6.



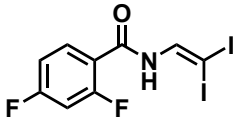
***N*-(2,2-diiodovinyl)-3-methylbenzamide (2j'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 10.9 Hz, 1H), 7.83 (d, *J* = 9.7 Hz, 1H), 7.68 (s, 1H), 7.65 – 7.60 (m, 1H), 7.44 – 7.38 (m, 2H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.9, 139.1, 137.6, 133.6, 132.2, 128.9, 128.1, 124.1, 21.4.



***N*-(2,2-diiodovinyl)-4-methylbenzamide (2k'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 10.9 Hz, 1H), 7.82 (d, *J* = 10.5 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.6, 143.6, 137.6, 129.7, 129.4, 127.3, 21.6.

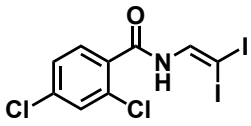


***N*-(2,2-diiodovinyl)-2,6-difluorobenzamide (2m'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 10.8 Hz, 1H), 7.77 (s, 1H), 7.50 (tt, *J* = 8.4, 6.3 Hz, 1H), 7.05 (t, *J* = 8.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6 (d, *J* = 249.5 Hz), 156.1, 136.8, 133.3 (t, *J* = 10.9 Hz), 112.6 (d, *J* = 26.2 Hz).

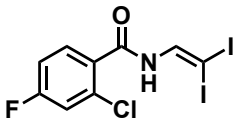


***N*-(2,2-diiodovinyl)-2,4-difluorobenzamide (2n'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 – 8.43 (m, 1H), 8.22 (td, *J* = 9.0, 6.5 Hz, 1H),

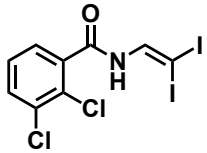
8.10 (dd, *J* = 10.6, 3.4 Hz, 1H), 7.12 – 7.04 (m, 1H), 6.96 (ddd, *J* = 12.2, 8.3, 2.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.2, 137.5, 134.4 (d, *J* = 7.1 Hz), 113.0 (d, *J* = 24.5 Hz), 104.7 (dd, *J* = 28.8, 26.0 Hz).



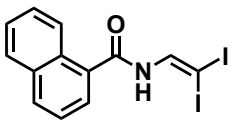
2,4-dichloro-*N*-(2,2-diiodovinyl) benzamide (2o'): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 9.8 Hz, 1H), 8.09 (d, *J* = 10.7 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.51 (d, *J* = 2.0 Hz, 1H), 7.41 (dd, *J* = 8.4, 2.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.64, 138.5, 137.4, 132.8, 131.7, 130.6, 130.2, 128.0.



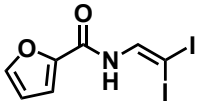
2-chloro-*N*-(2,2-diiodovinyl)-4-fluorobenzamide (2p'): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (d, *J* = 9.2 Hz, 1H), 8.10 (d, *J* = 10.7 Hz, 1H), 7.97 (dd, *J* = 8.8, 6.1 Hz, 1H), 7.23 (dd, *J* = 8.2, 2.4 Hz, 1H), 7.14 (ddd, *J* = 8.8, 7.6, 2.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1 (d, *J* = 257.1 Hz), 160.6, 137.5, 133.9 (d, *J* = 9.6 Hz), 132.3 (d, *J* = 10.6 Hz), 128.1 (d, *J* = 3.5 Hz), 118.2 (d, *J* = 25.1 Hz), 115.2 (d, *J* = 21.3 Hz).



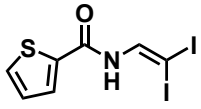
2,3-dichloro-*N*-(2,2-diiodovinyl) benzamide (2q'): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 10.7 Hz, 1H), 8.01 (s, 1H), 7.66 (ddd, *J* = 9.8, 7.9, 1.5 Hz, 2H), 7.36 (t, *J* = 7.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.4, 137.1, 134.9, 134.5, 133.3, 129.5, 129.0, 128.0.



***N*-(2,2-diiodovinyl)-1-naphthamide (2t'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 8.3 Hz, 1H), 8.17 (d, *J* = 11.0 Hz, 1H), 8.04 (d, *J* = 8.3 Hz, 1H), 7.94 (d, *J* = 7.8 Hz, 1H), 7.76 (d, *J* = 7.0 Hz, 1H), 7.70 – 7.52 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 137.6, 133.9, 132.2, 131.7, 130.2, 128.6, 127.8, 126.9, 125.7, 125.1, 124.7.

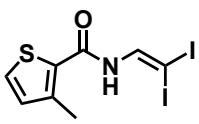


***N*-(2,2-diiodovinyl) furan-2-carboxamide (2v'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.97 (d, *J* = 11.1 Hz, 1H), 7.58 (dd, *J* = 1.7, 0.7 Hz, 1H), 7.30 (dd, *J* = 3.6, 0.7 Hz, 1H), 6.60 (dd, *J* = 3.6, 1.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.5, 146.1, 145.3, 136.5, 117.0, 112.9.

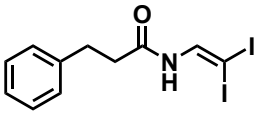


***N*-(2,2-diiodovinyl) thiophene-2-carboxamide (2w'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 10.9 Hz, 1H), 7.66 (dd, *J* = 12.3, 9.1 Hz, 3H), 7.21 – 7.15 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.2,

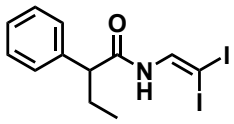
136.9, 136.6, 132.2, 129.5, 128.2.



***N*-(2,2-diiodovinyl)-3-methylthiophene-2-carboxamide (2x'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 10.8 Hz, 1H), 7.61 (d, *J* = 9.8 Hz, 1H), 7.44 (d, *J* = 5.0 Hz, 1H), 6.97 (d, *J* = 5.0 Hz, 1H), 2.63 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.2, 143.1, 137.4, 132.7, 129.4, 128.9, 16.4.



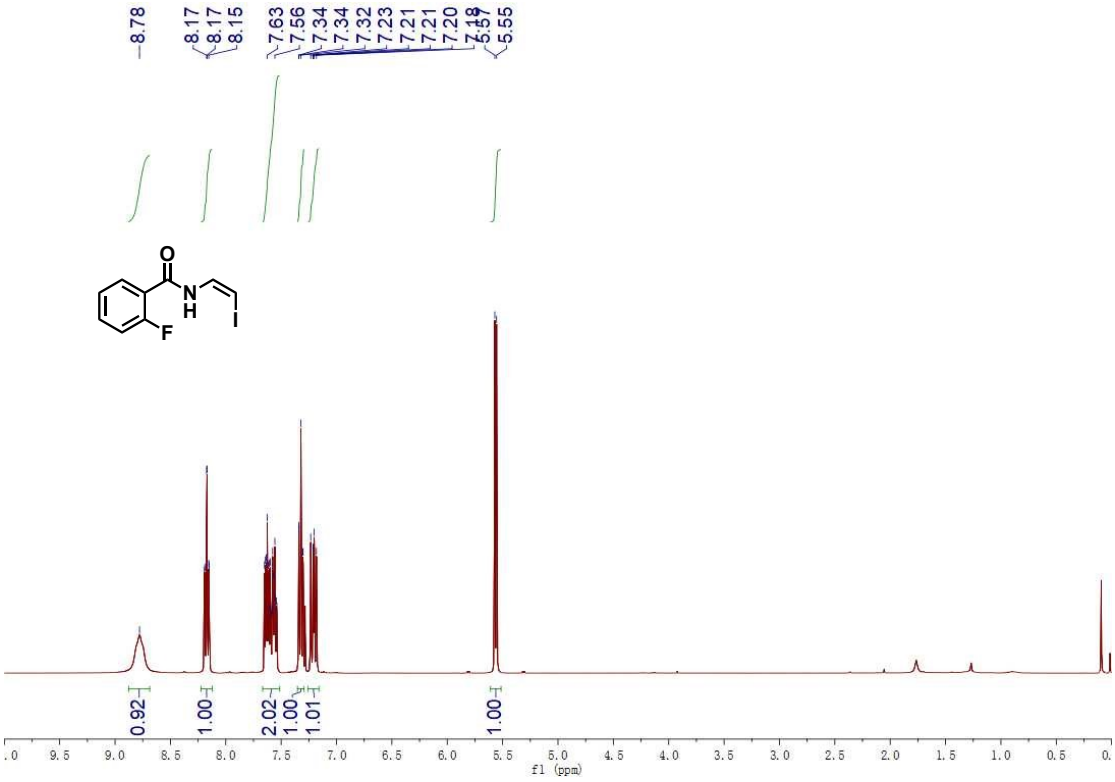
***N*-(2,2-diiodovinyl)-3-phenylpropanamide (2y'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 10.9 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 – 7.22 (m, 3H), 6.93 (d, *J* = 10.0 Hz, 1H), 3.03 (t, *J* = 7.6 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 139.9, 137.1, 128.8, 128.3, 126.6, 38.0, 31.0.



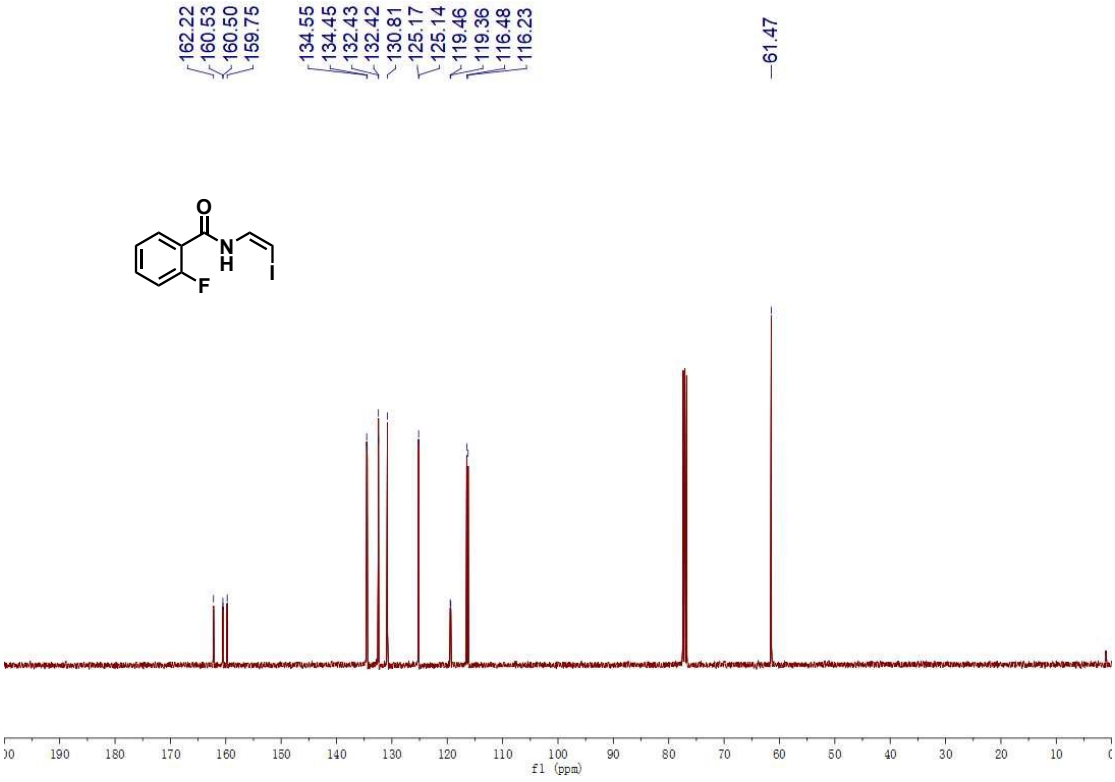
***N*-(2,2-diiodovinyl)-2-phenylbutanamide (2aa'):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 10.9 Hz, 1H), 7.45 – 7.38 (m, 2H), 7.37 – 7.30 (m, 3H), 7.03 (d, J = 10.0 Hz, 1H), 3.36 (dd, J = 8.7, 6.5 Hz, 1H), 2.33 – 2.20 (m, 1H), 1.95 – 1.82 (m, 1H), 0.93 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 138.0, 137.2, 129.3, 128.2, 127.9, 55.0, 25.7, 12.2.

8. ^{1}H NMR and ^{13}C NMR spectra

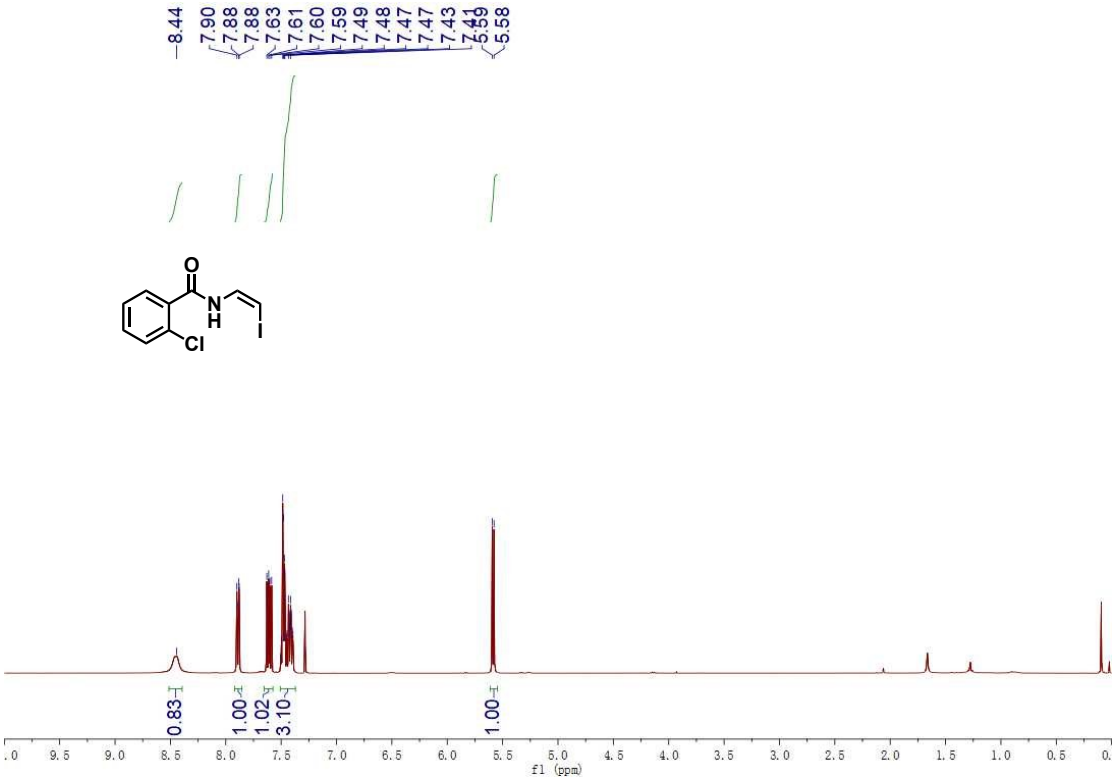
(*Z*)-2-fluoro-*N*-(2-iodovinyl) benzamide (2a): ^{1}H NMR (400 MHz, $CDCl_3$)



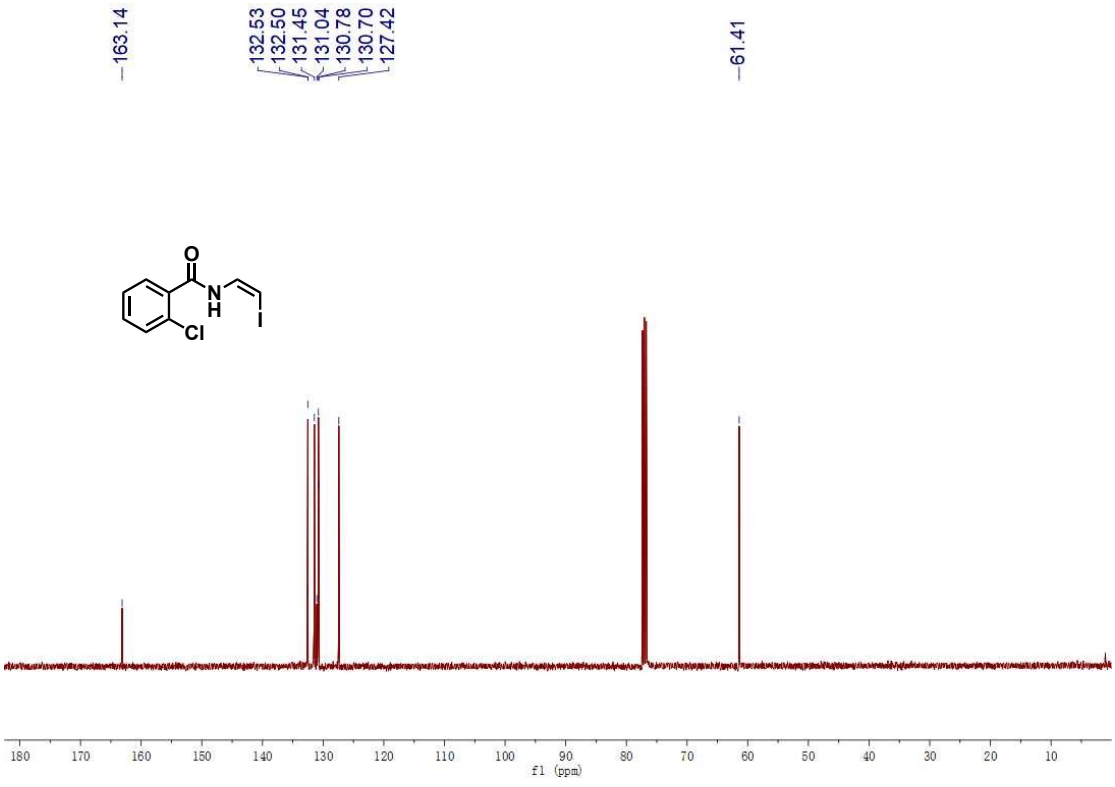
(*Z*)-2-fluoro-*N*-(2-iodovinyl) benzamide (2a): ^{13}C NMR (101 MHz, $CDCl_3$)



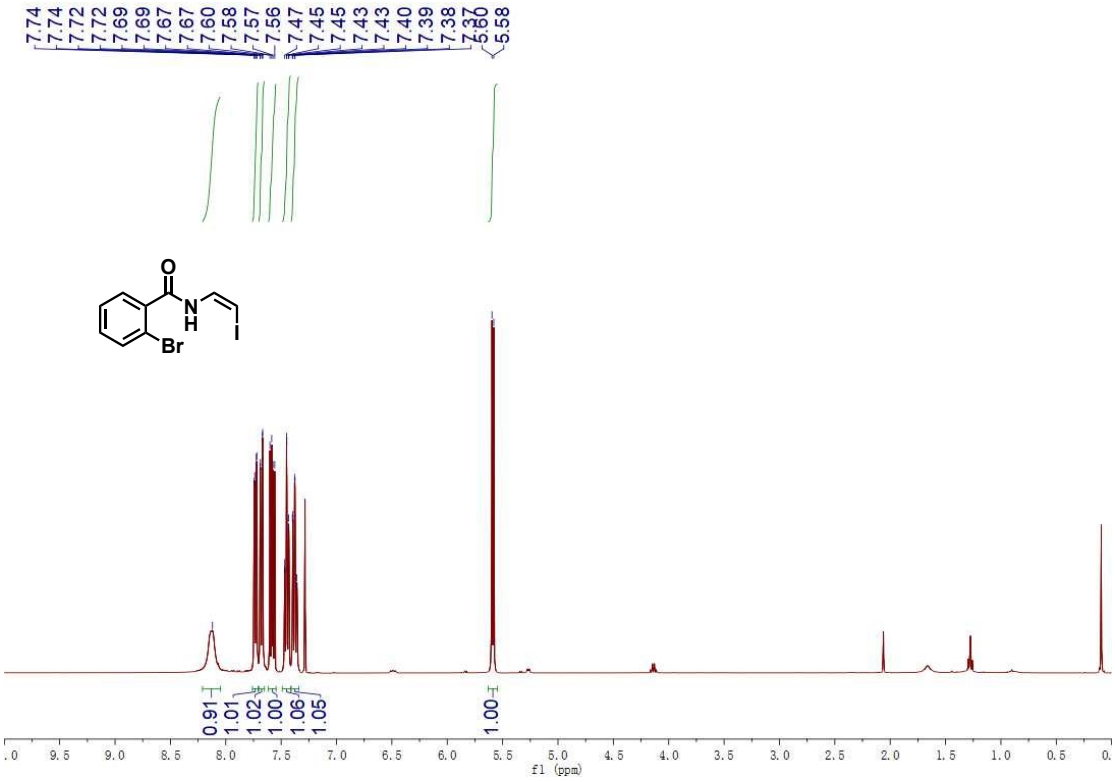
(*Z*)-2-chloro-*N*-(2-iodovinyl) benzamide (2b): ^{1}H NMR (400 MHz, $CDCl_3$)



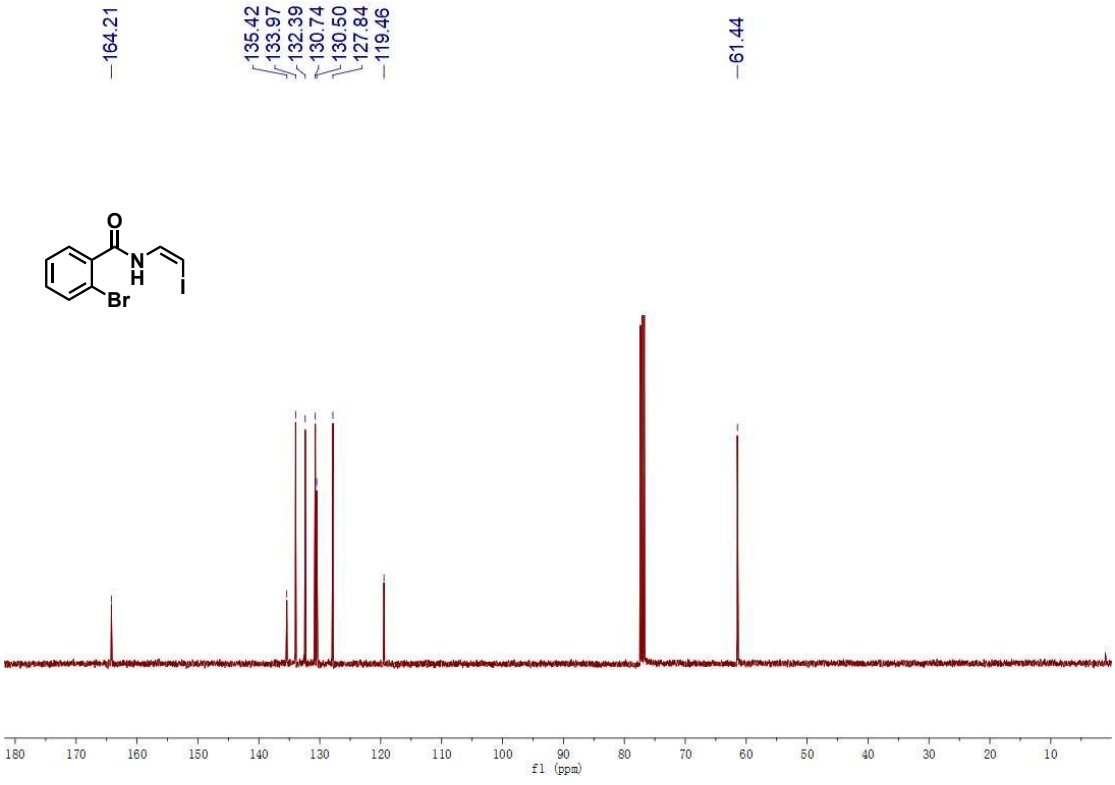
(*Z*)-2-chloro-*N*-(2-iodovinyl) benzamide (2b): ^{13}C NMR (101 MHz, $CDCl_3$)



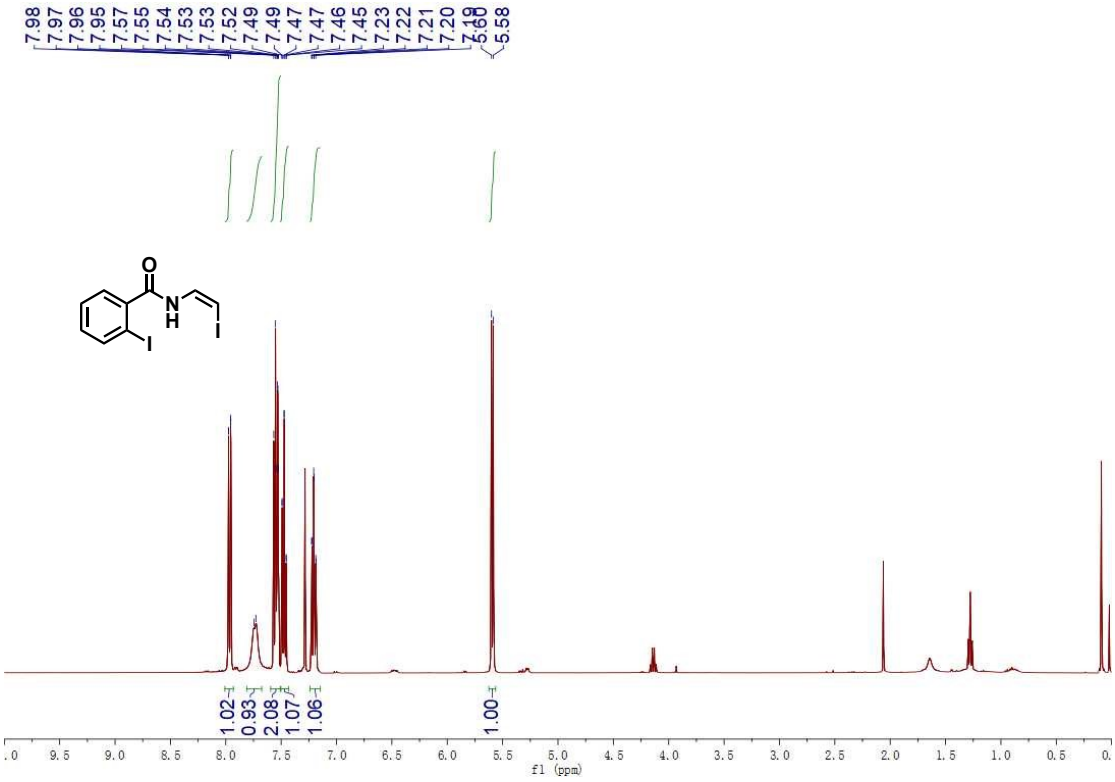
(*Z*)-2-bromo-*N*-(2-iodovinyl) benzamide (2c): ^{1}H NMR (400 MHz, $CDCl_3$)



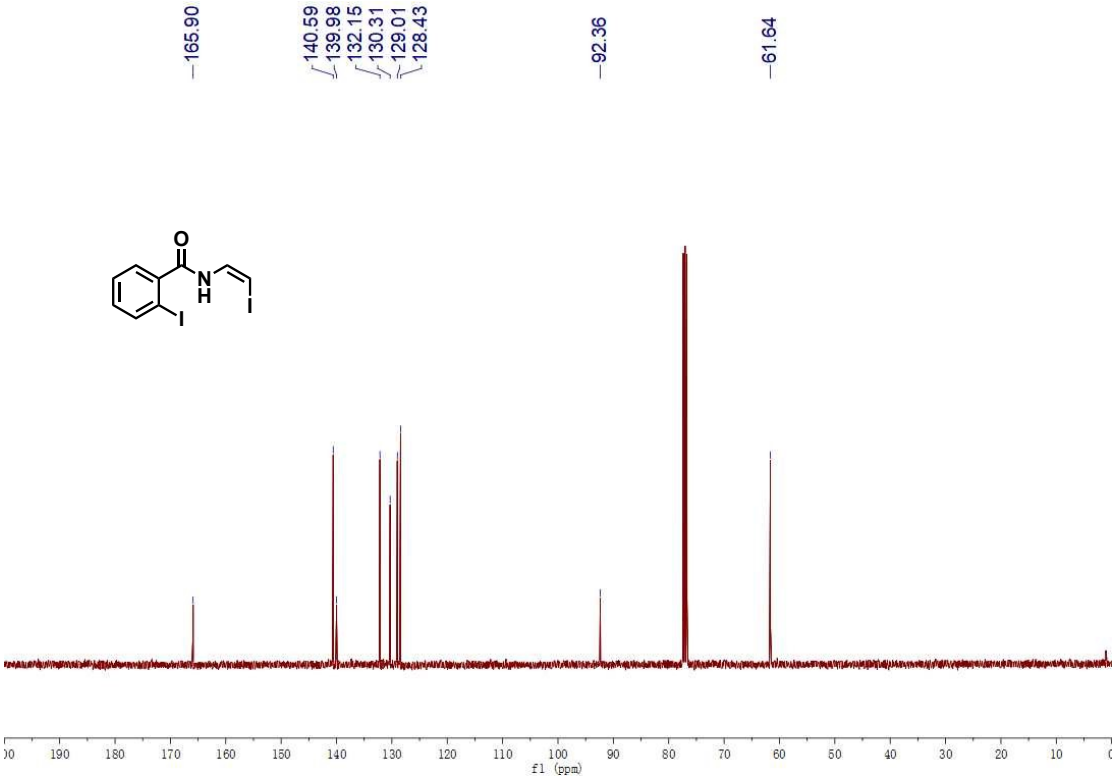
(*Z*)-2-bromo-*N*-(2-iodovinyl) benzamide (2c): ^{13}C NMR (101 MHz, $CDCl_3$)



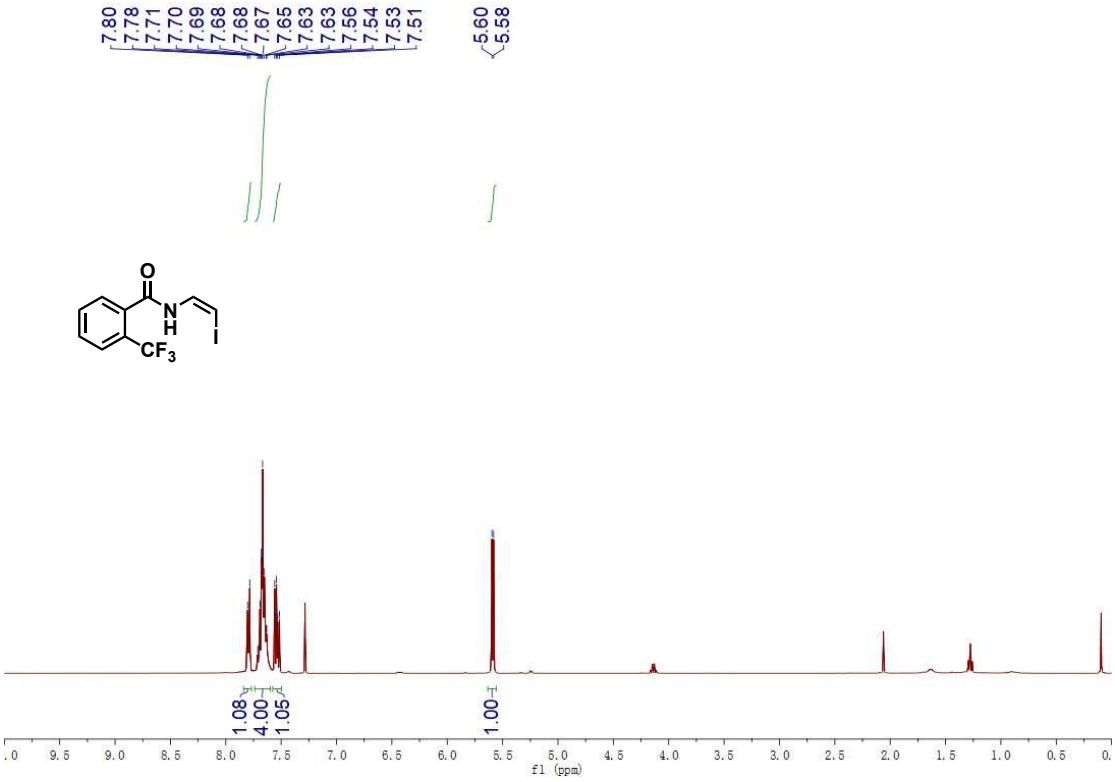
(*Z*)-2-iodo-*N*-(2-iodovinyl) benzamide (2d): ^{1}H NMR (400 MHz, $CDCl_3$)



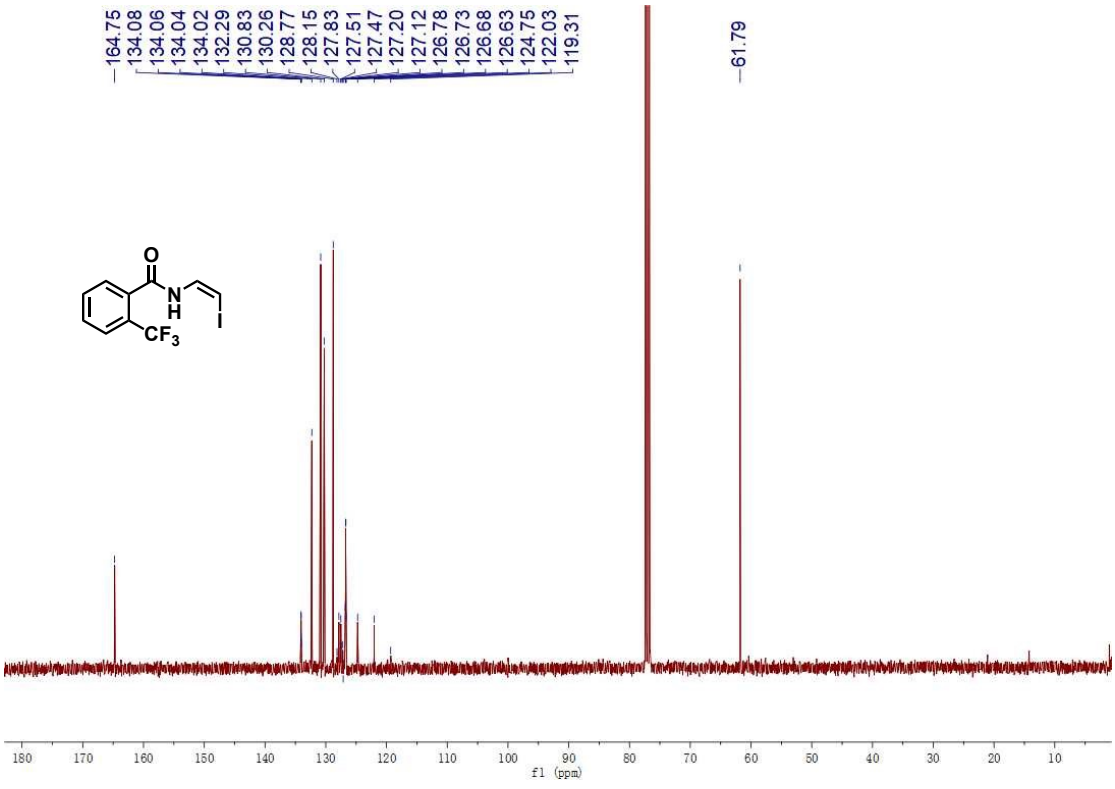
(*Z*)-2-iodo-*N*-(2-iodovinyl) benzamide (2d): ^{13}C NMR (101 MHz, $CDCl_3$)



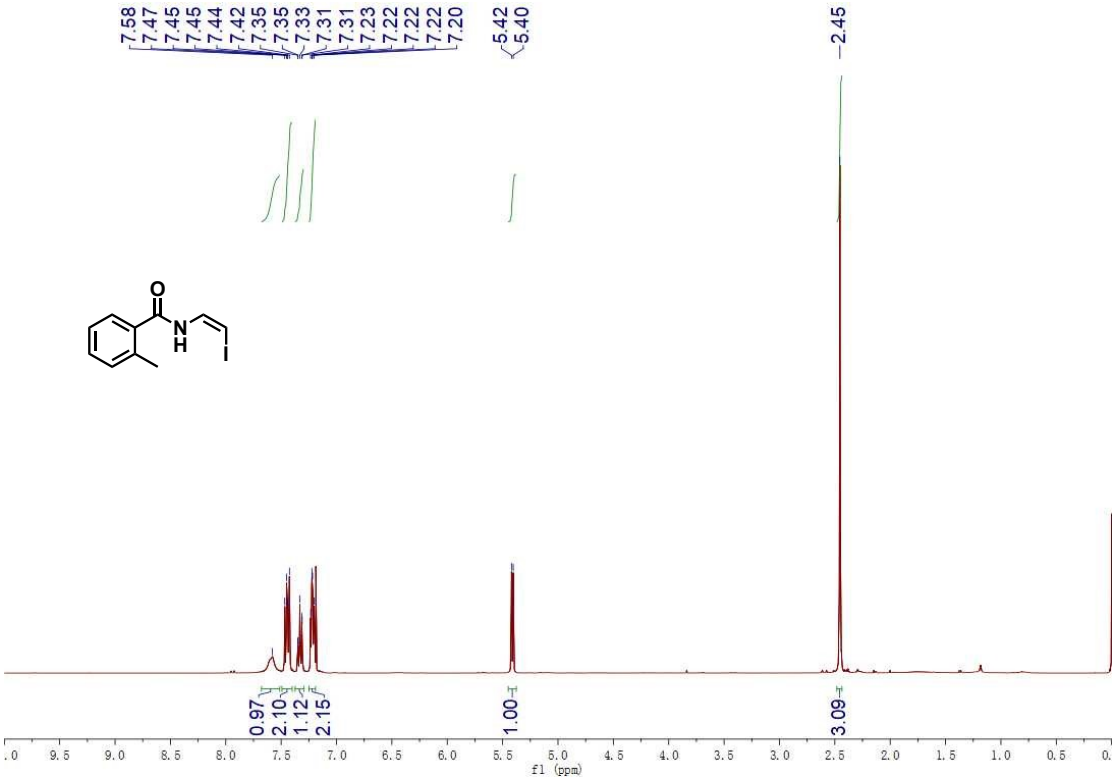
(*Z*)-*N*-(2-iodovinyl)-2-(trifluoromethyl) benzamide (2e): ^{1}H NMR (400 MHz, $CDCl_3$)



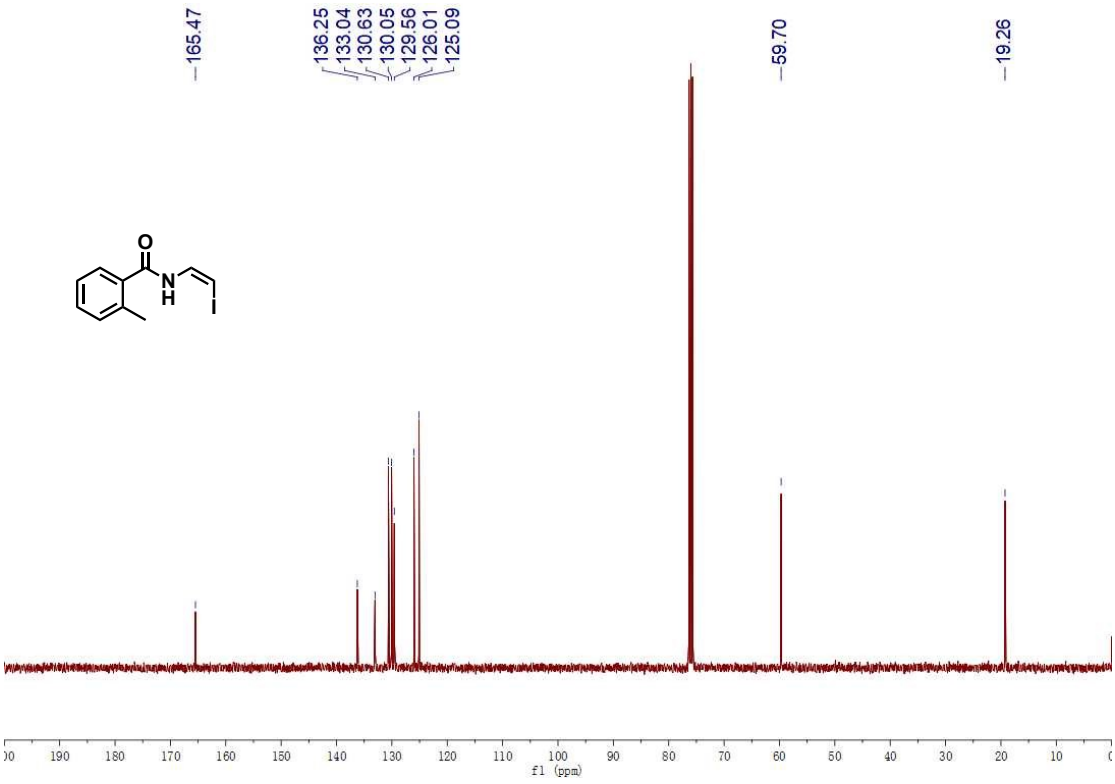
(*Z*)-*N*-(2-iodovinyl)-2-(trifluoromethyl) benzamide (2e): ^{13}C NMR (101 MHz, $CDCl_3$)



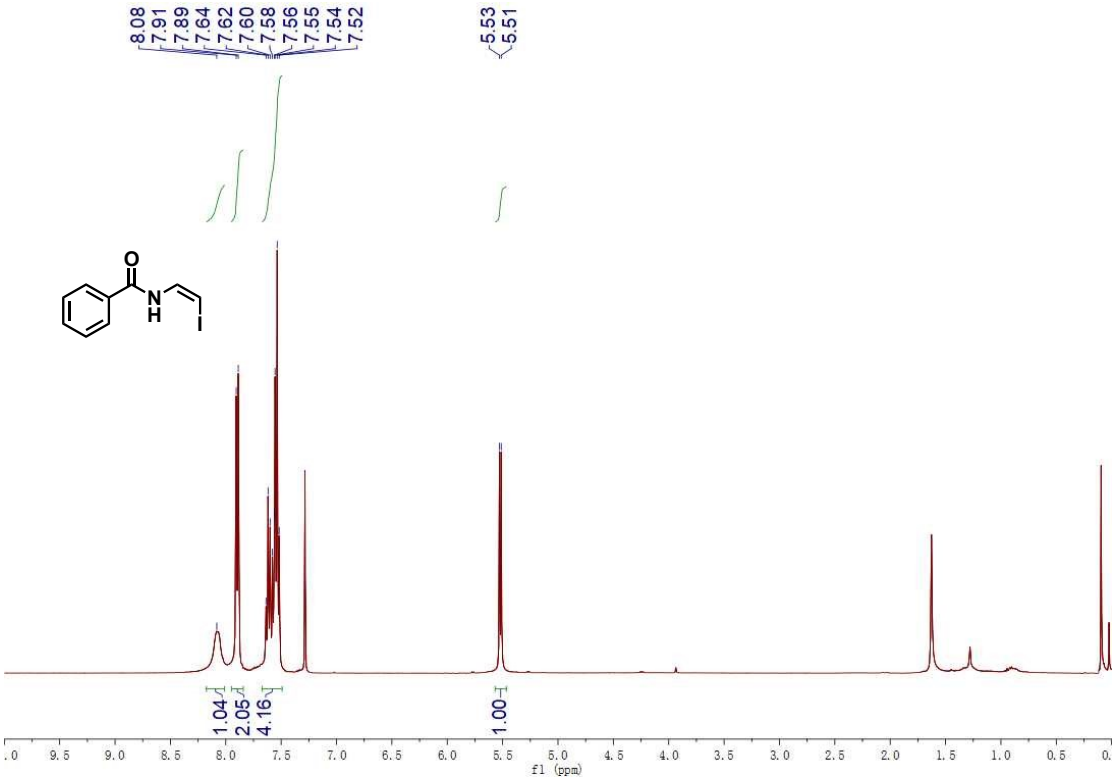
(*Z*)-*N*-(2-iodovinyl)-2-methylbenzamide (2f): ^{1}H NMR (400 MHz, $CDCl_3$)



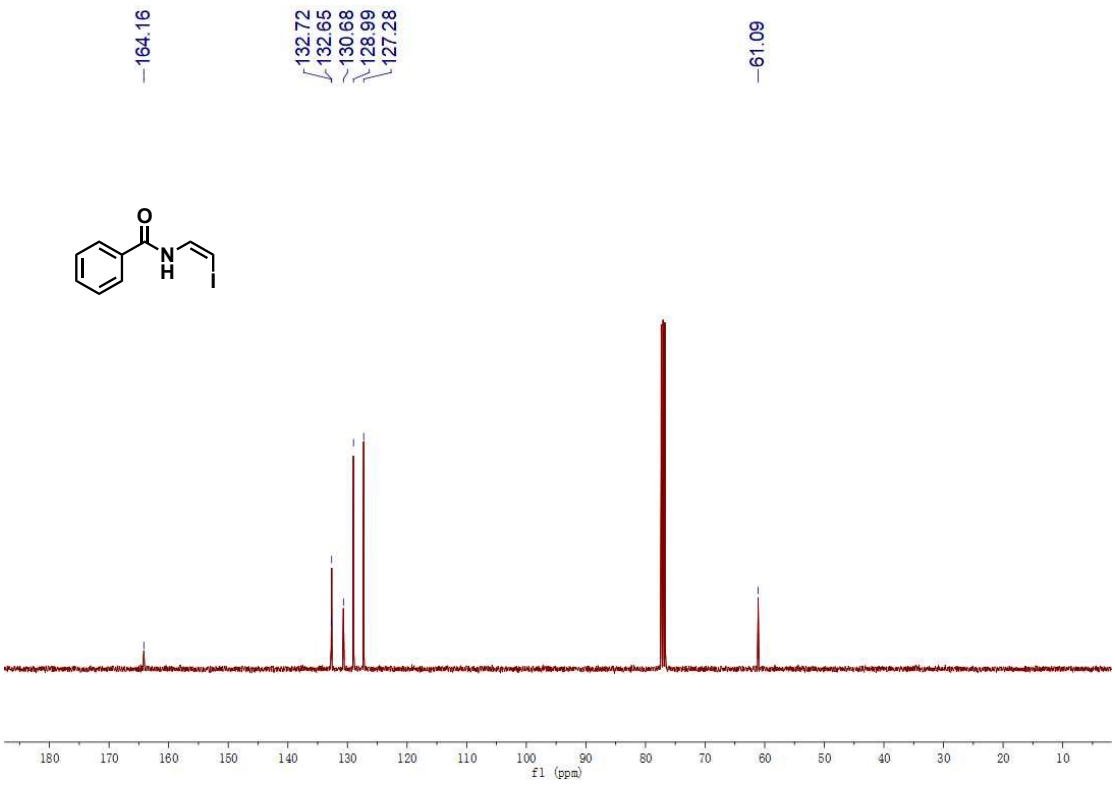
(*Z*)-*N*-(2-iodovinyl)-2-methylbenzamide (2f): ^{13}C NMR (101 MHz, $CDCl_3$)



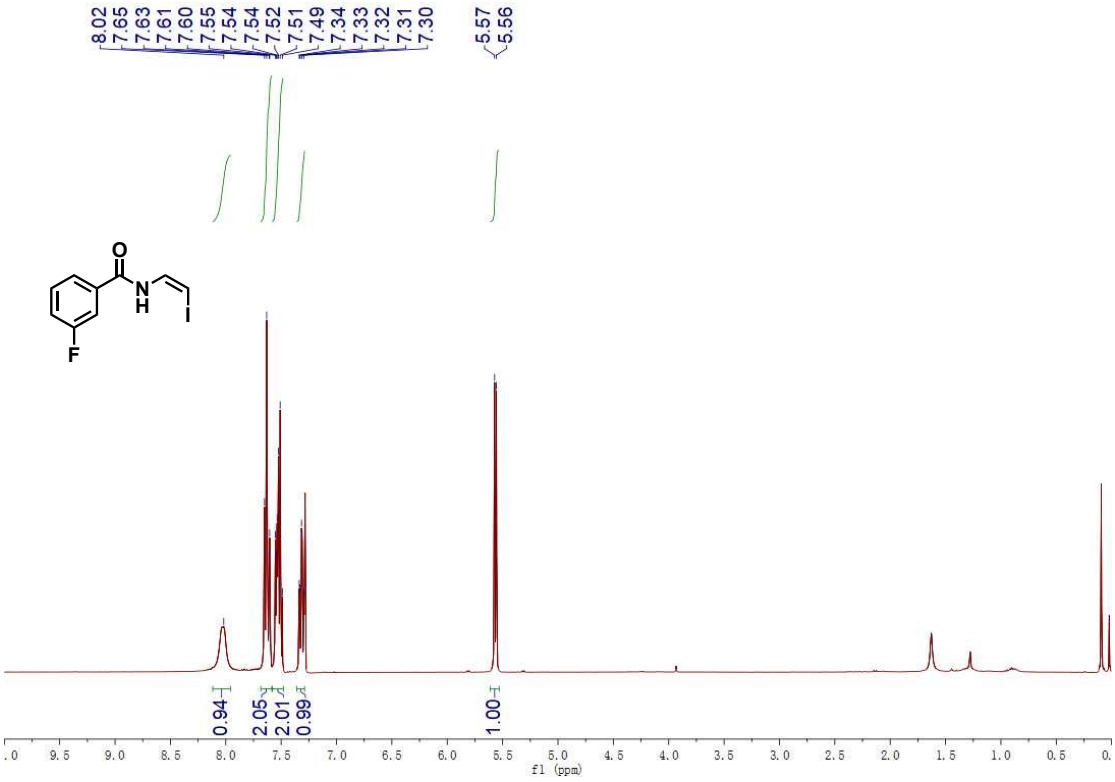
(*Z*)-*N*-(2-iodovinyl) benzamide (2g): ^{1}H NMR (400 MHz, $CDCl_3$)



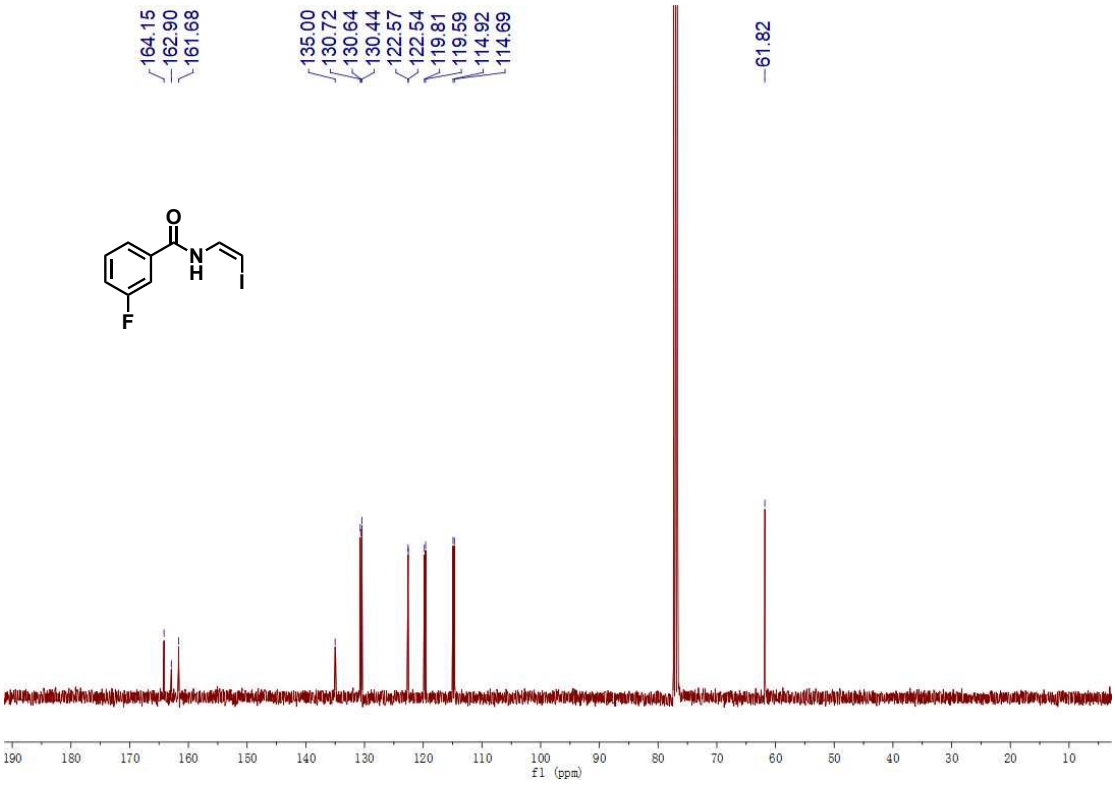
(*Z*)-*N*-(2-iodovinyl) benzamide (2g): ^{13}C NMR (101 MHz, $CDCl_3$)



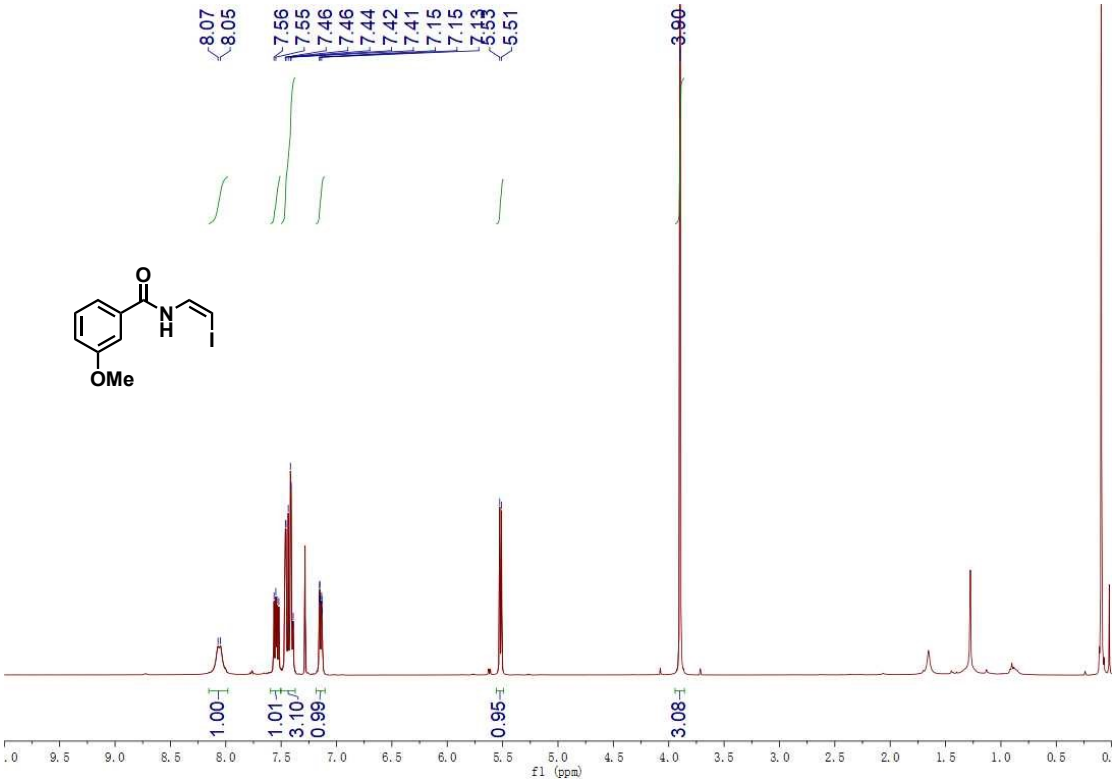
(*Z*)-3-fluoro-*N*-(2-iodovinyl) benzamide (2h): ^{1}H NMR (400 MHz, $CDCl_3$)



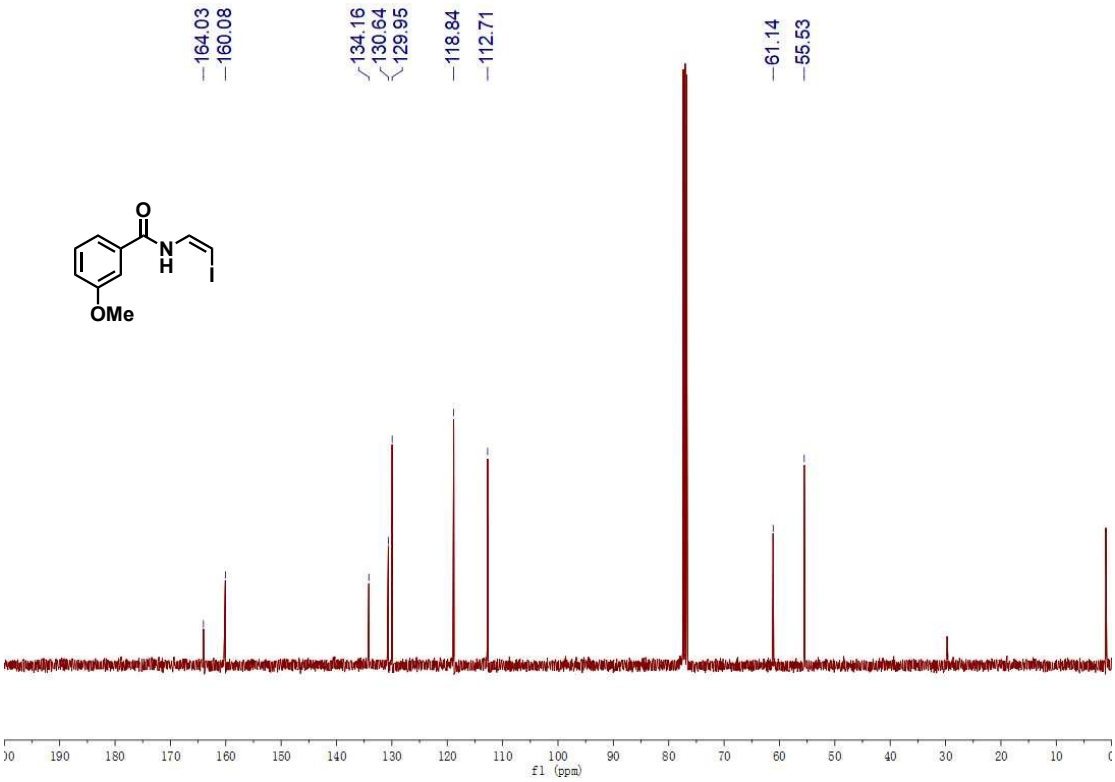
(*Z*)-3-fluoro-*N*-(2-iodovinyl) benzamide (2h): ^{13}C NMR (101 MHz, $CDCl_3$)



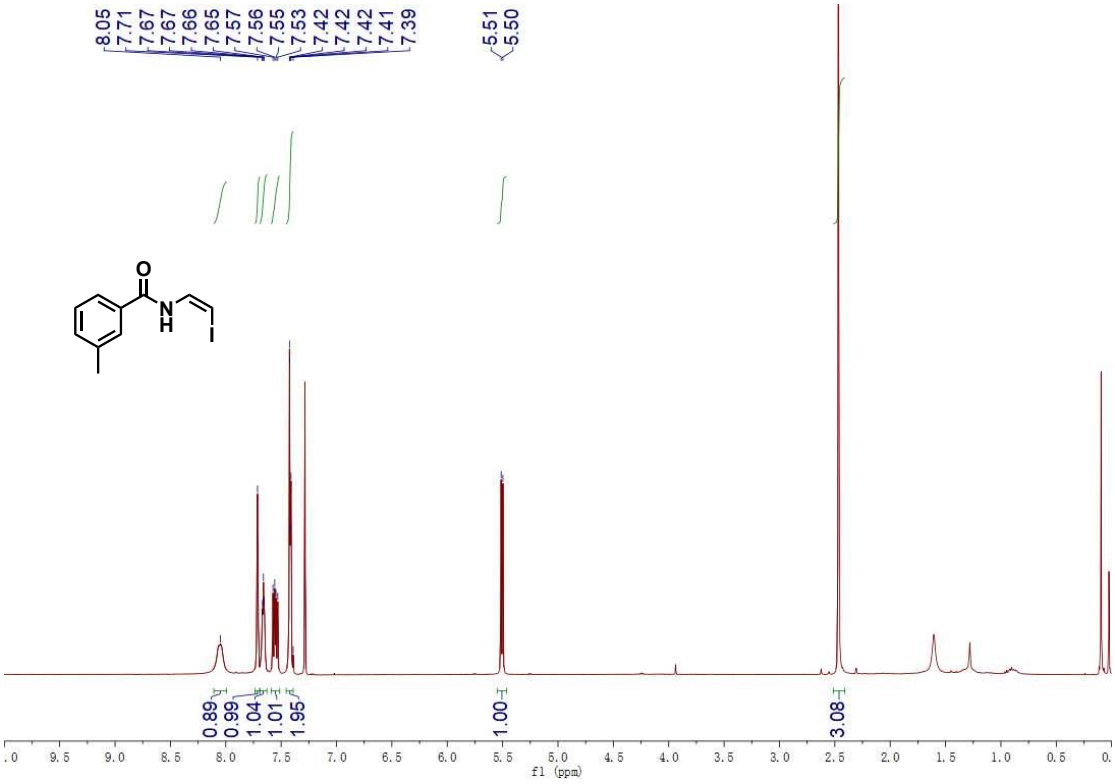
(*Z*)-*N*-(2-iodovinyl)-3-methoxybenzamide (2i): ^{1}H NMR (400 MHz, $CDCl_3$)



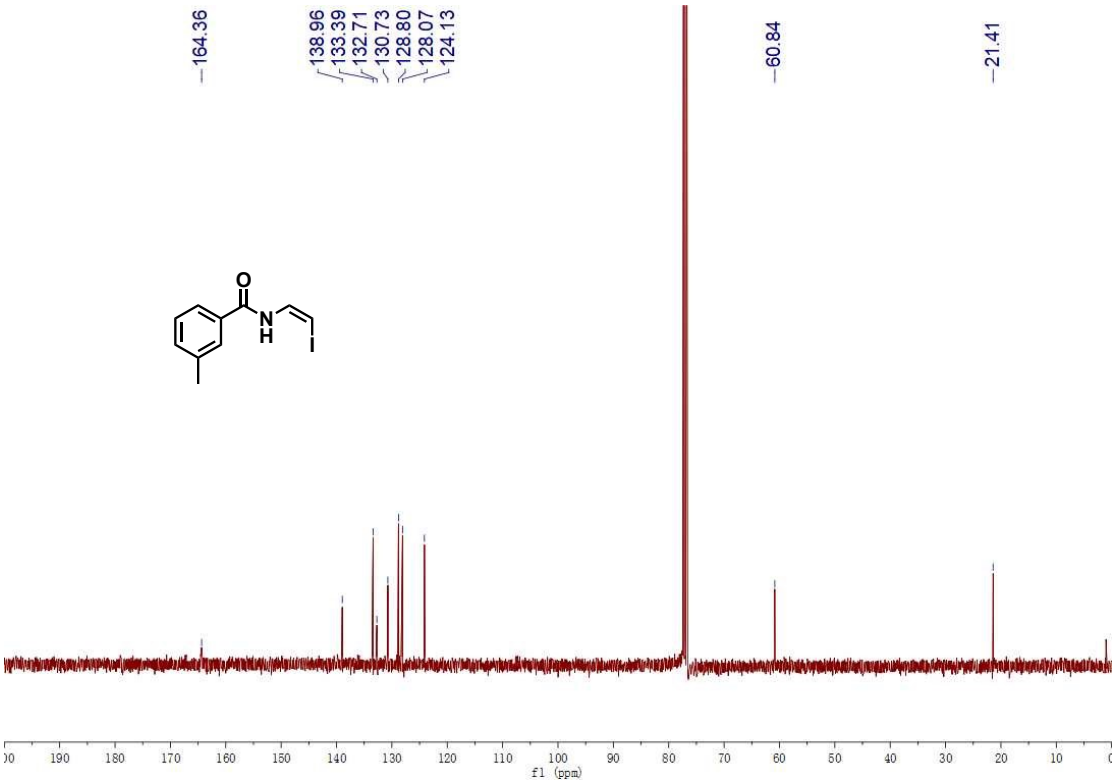
(*Z*)-*N*-(2-iodovinyl)-3-methoxybenzamide (2i): ^{13}C NMR (101 MHz, $CDCl_3$)



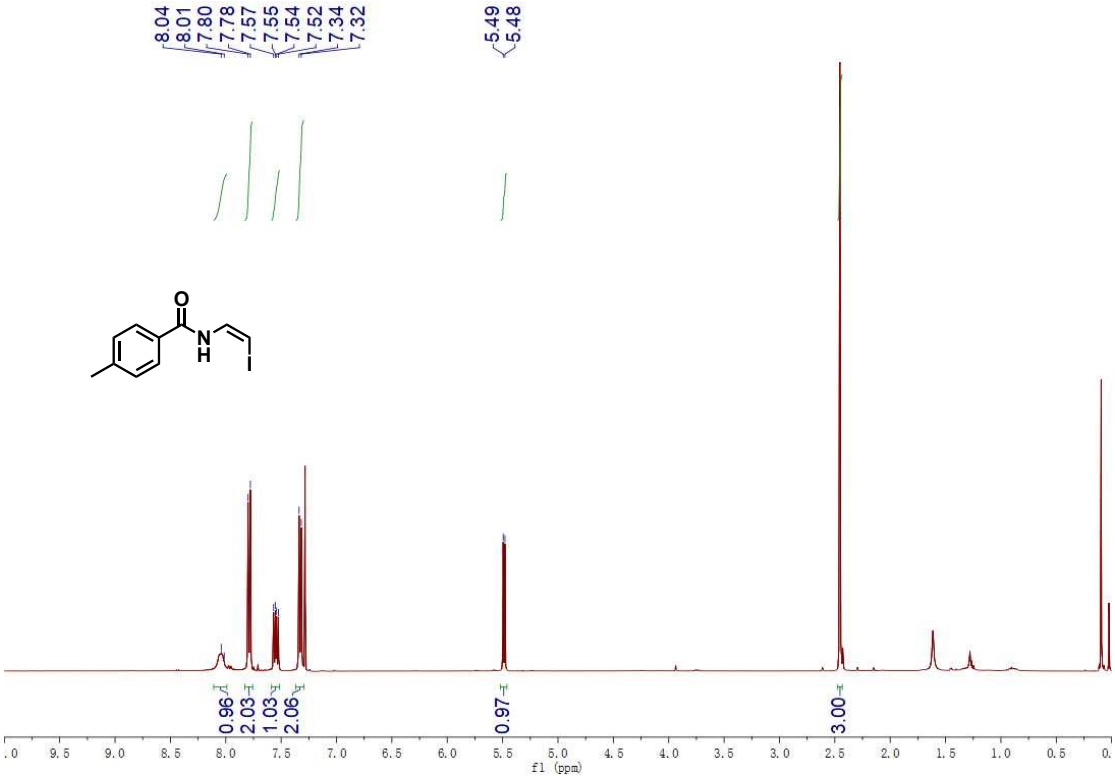
(*Z*)-*N*-(2-iodovinyl)-3-methylbenzamide (2j): ^{1}H NMR (400 MHz, $CDCl_3$)



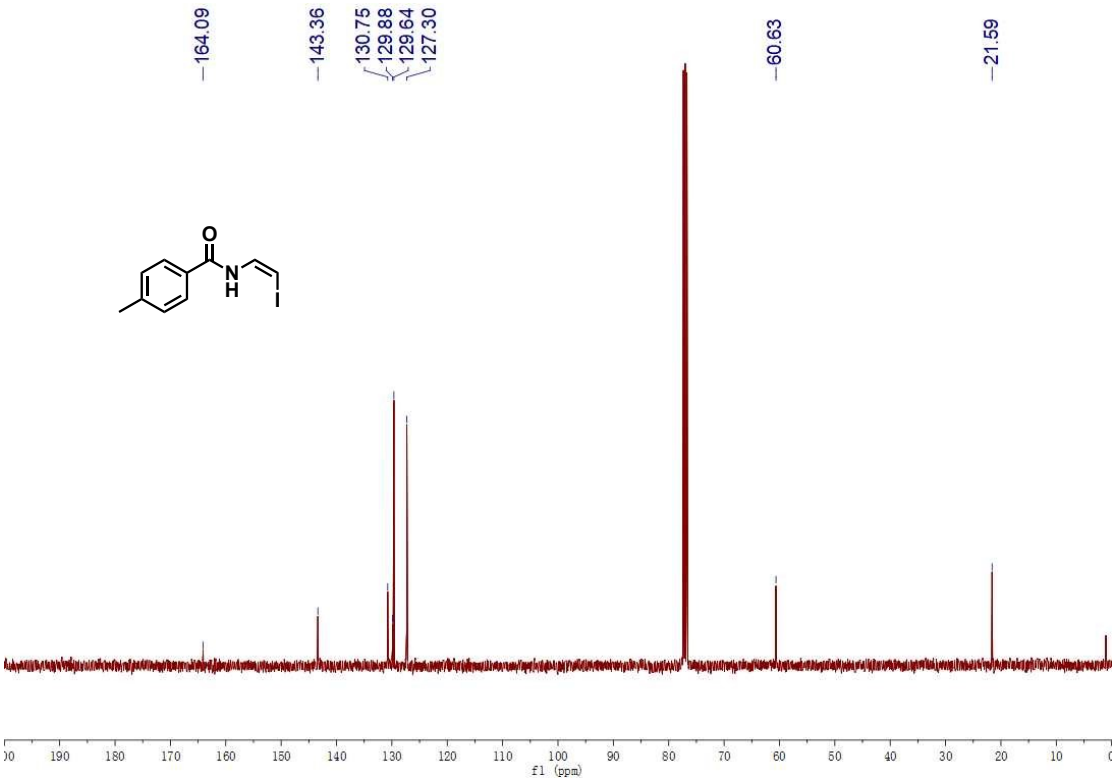
(*Z*)-*N*-(2-iodovinyl)-3-methylbenzamide (2j): ^{13}C NMR (101 MHz, $CDCl_3$)



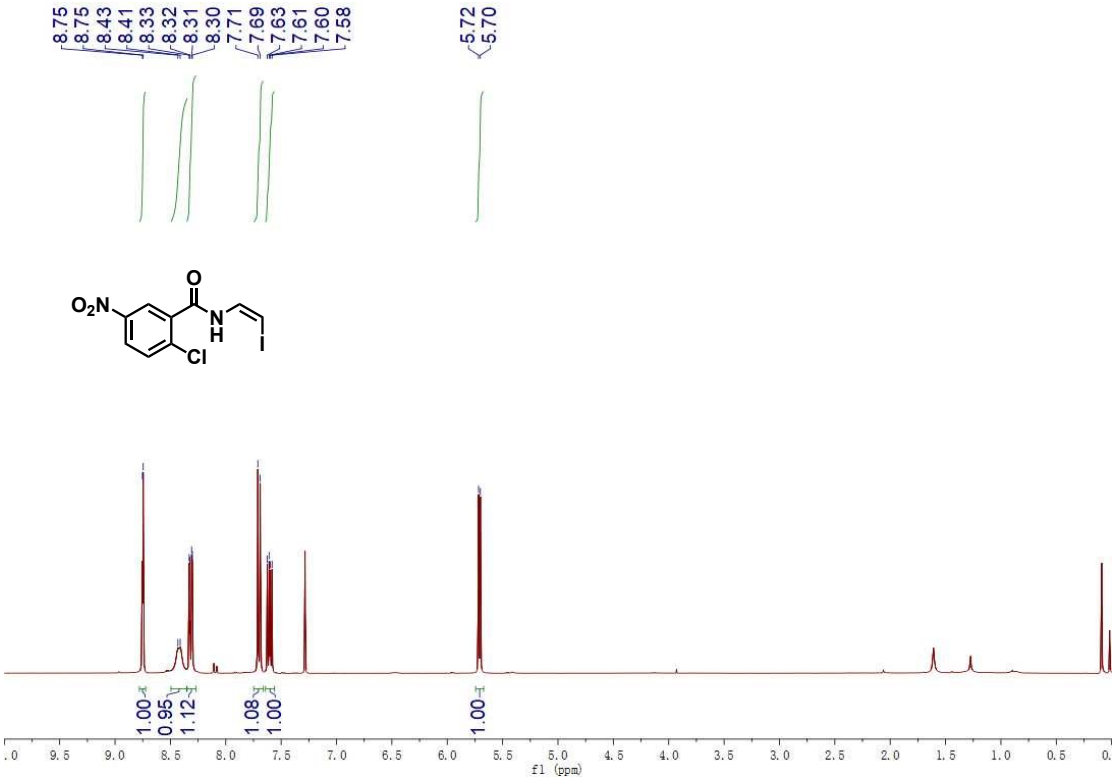
(*Z*)-*N*-(2-iodovinyl)-4-methylbenzamide (2k): ^{1}H NMR (400 MHz, $CDCl_3$)



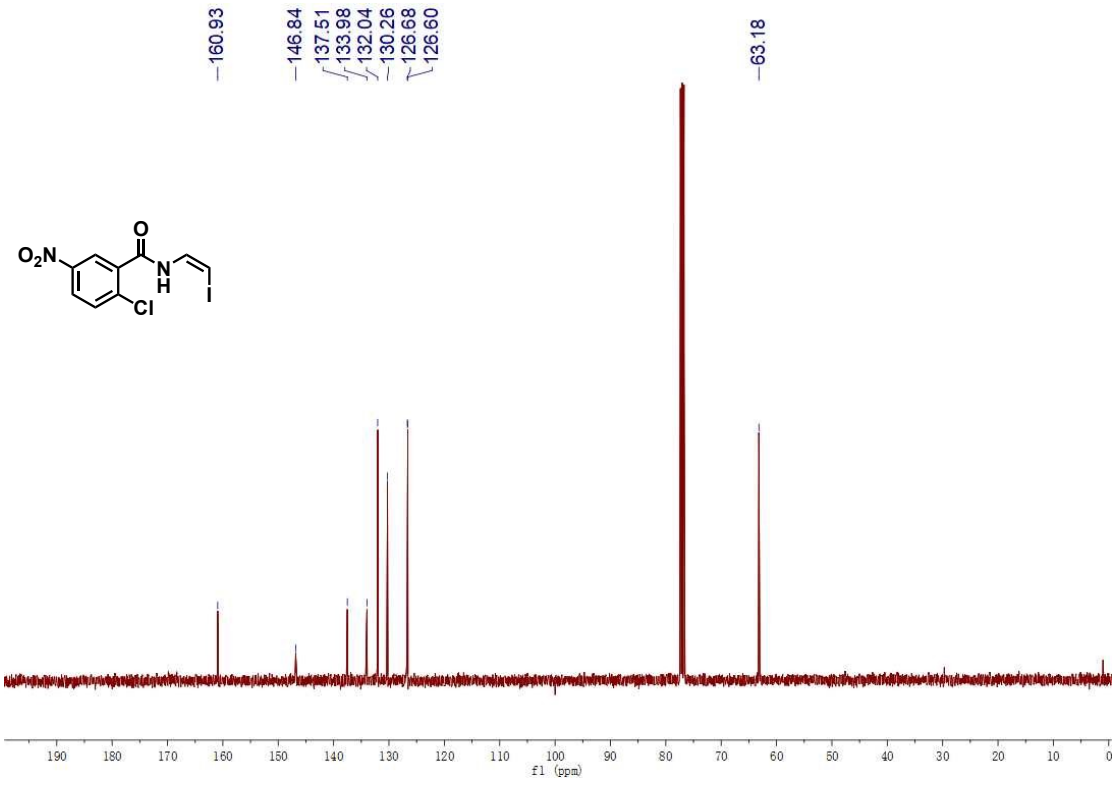
(*Z*)-*N*-(2-iodovinyl)-4-methylbenzamide (2k): ^{13}C NMR (101 MHz, $CDCl_3$)



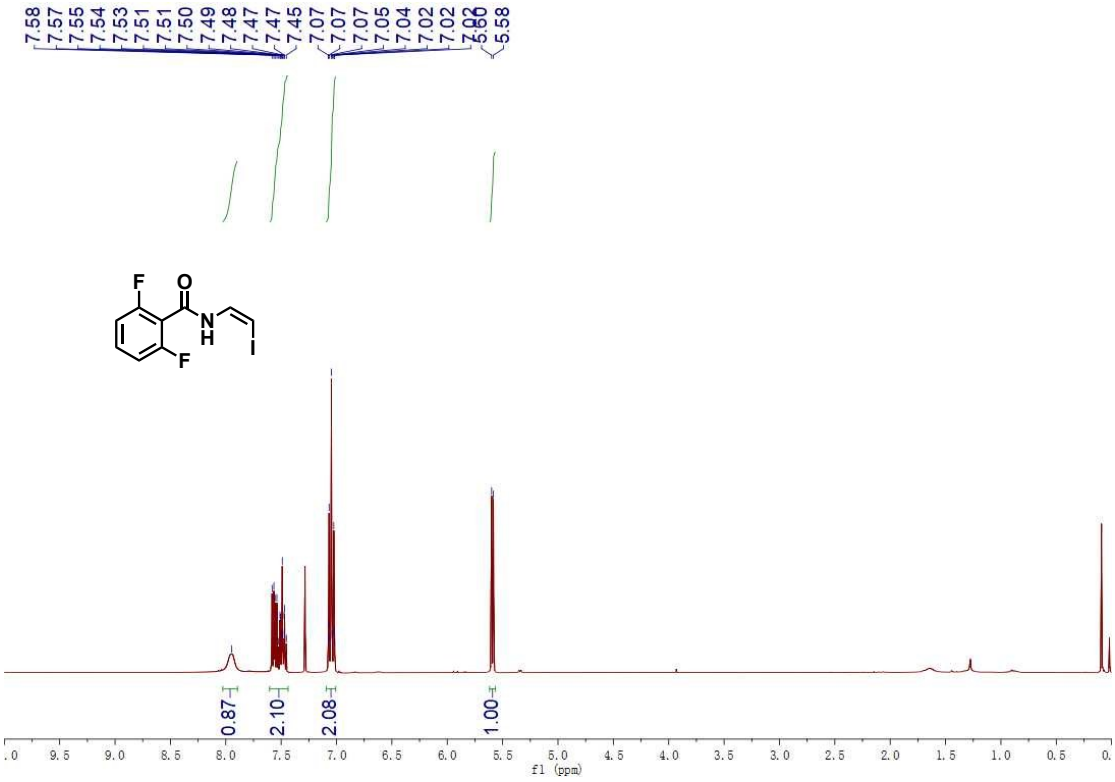
(*Z*)-2-chloro-*N*-(2-iodovinyl)-5-nitrobenzamide (2l): ^{1}H NMR (400 MHz, $CDCl_3$)



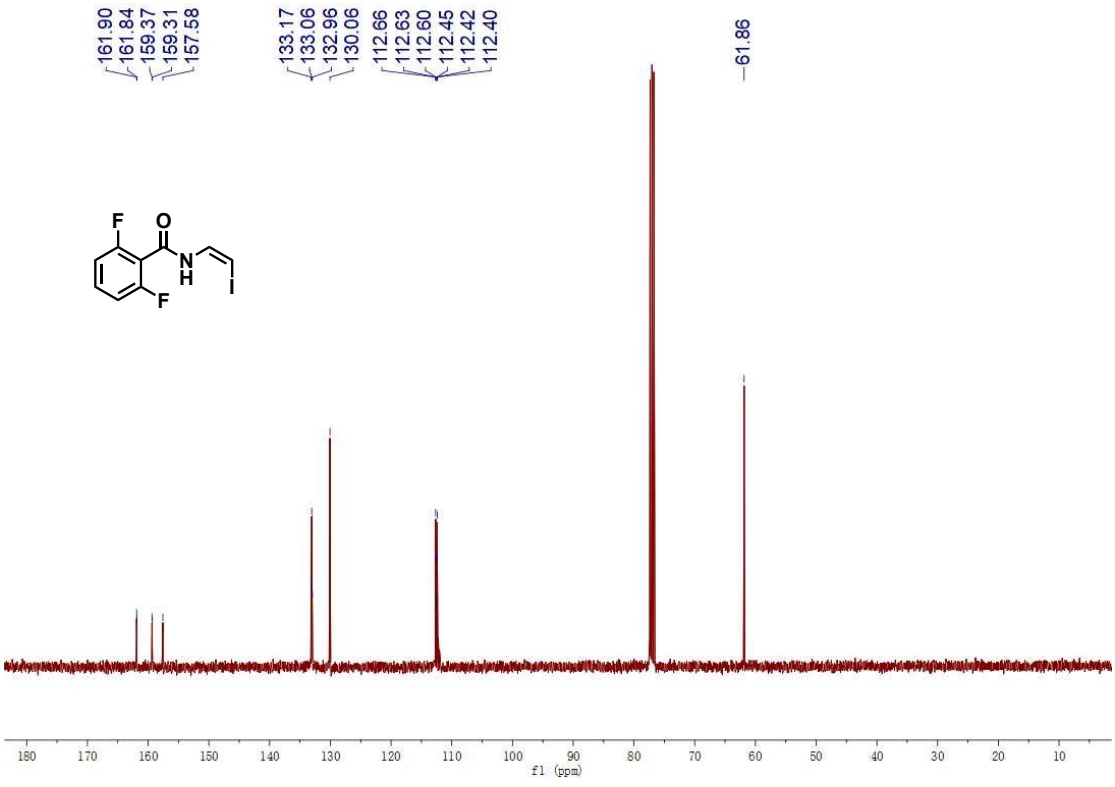
(*Z*)-2-chloro-*N*-(2-iodovinyl)-5-nitrobenzamide (2l): ^{13}C NMR (101 MHz, $CDCl_3$)



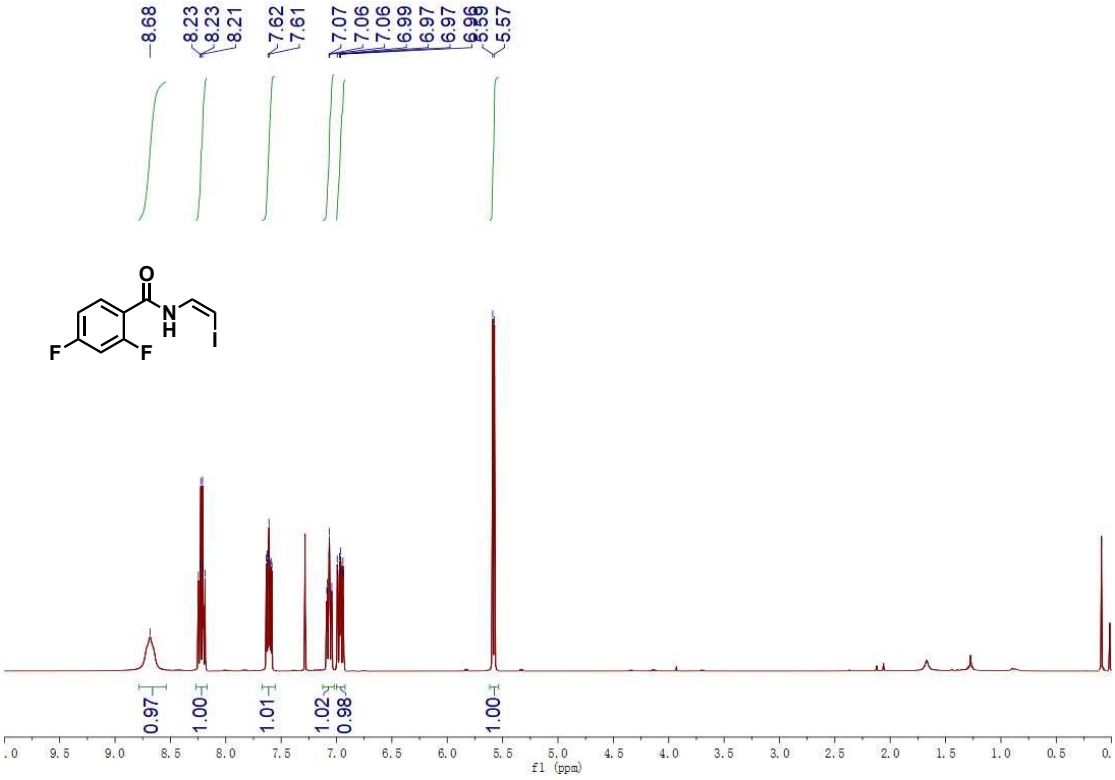
(*Z*)-2,6-difluoro-*N*-(2-iodovinyl) benzamide (2m): ^{1}H NMR (400 MHz, $CDCl_3$)



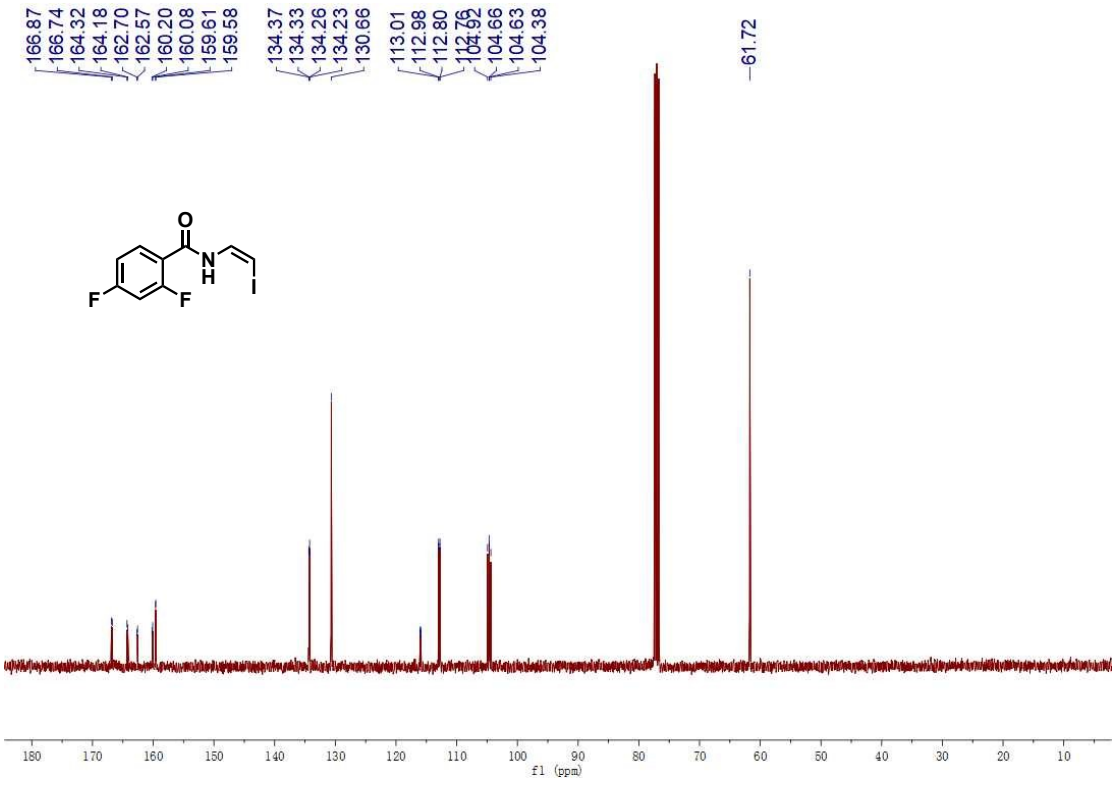
(*Z*)-2,6-difluoro-*N*-(2-iodovinyl) benzamide (2m): ^{13}C NMR (101 MHz, $CDCl_3$)



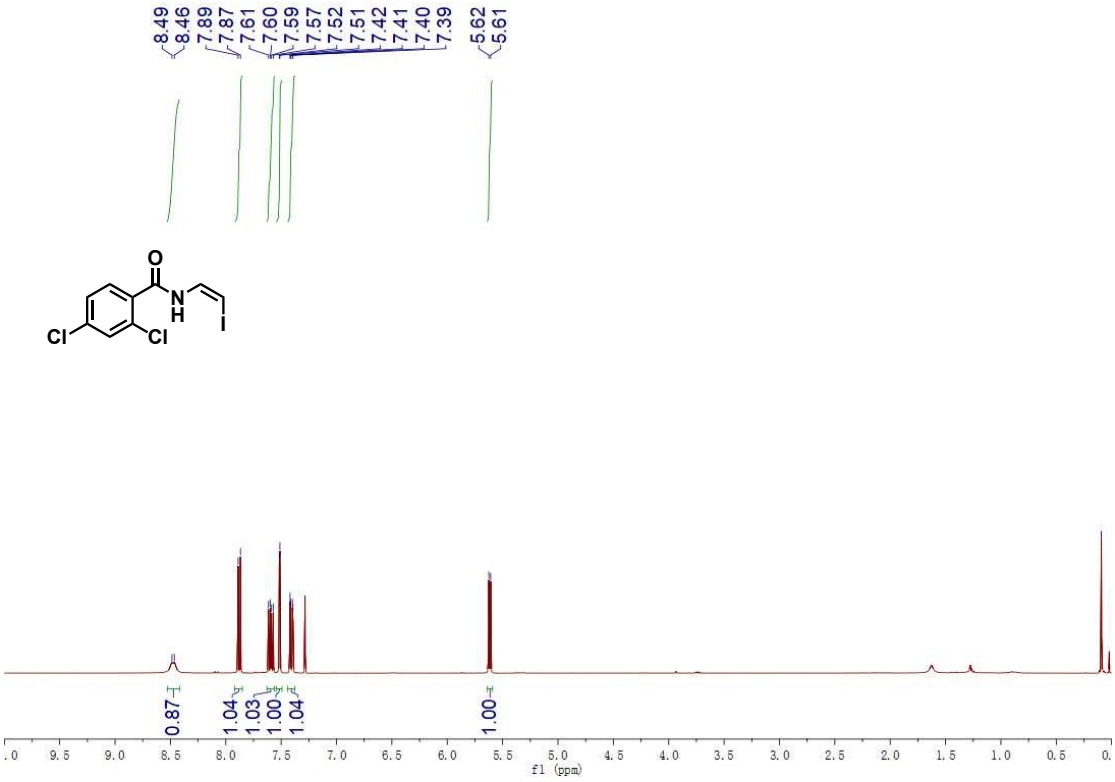
(*Z*)-2,4-difluoro-*N*-(2-iodovinyl) benzamide (2n): ^{1}H NMR (400 MHz, $CDCl_3$)



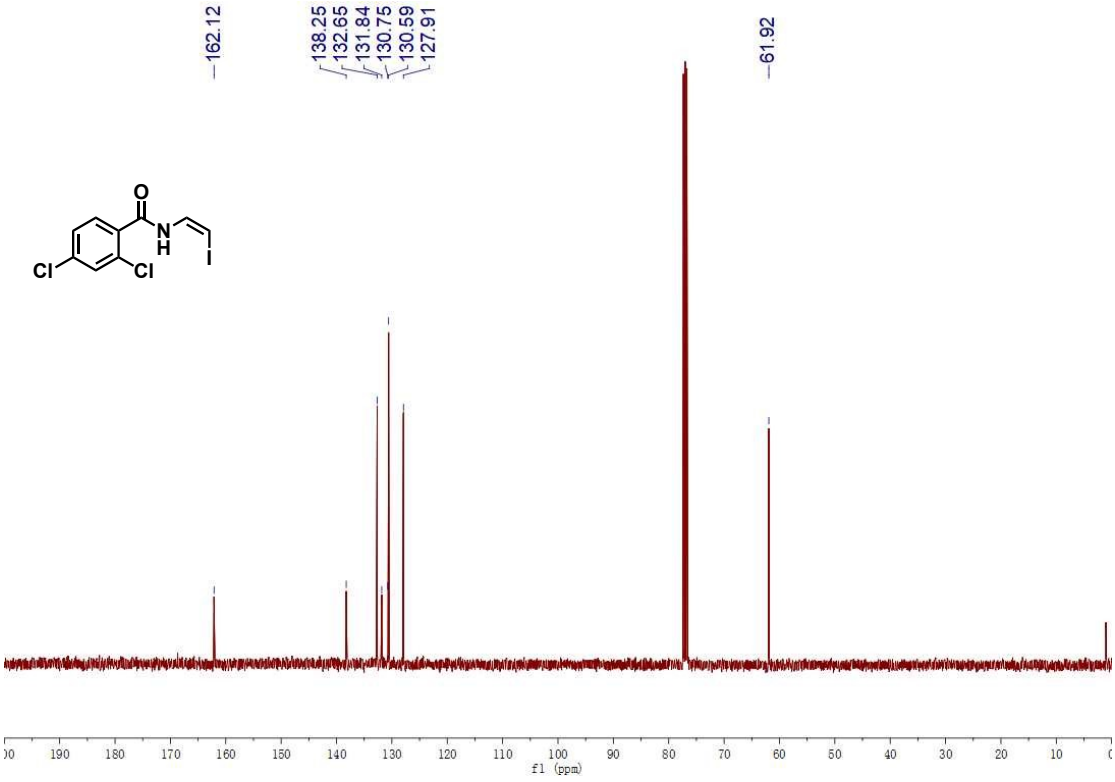
(*Z*)-2,4-difluoro-*N*-(2-iodovinyl) benzamide (2n): ^{13}C NMR (101 MHz, $CDCl_3$)



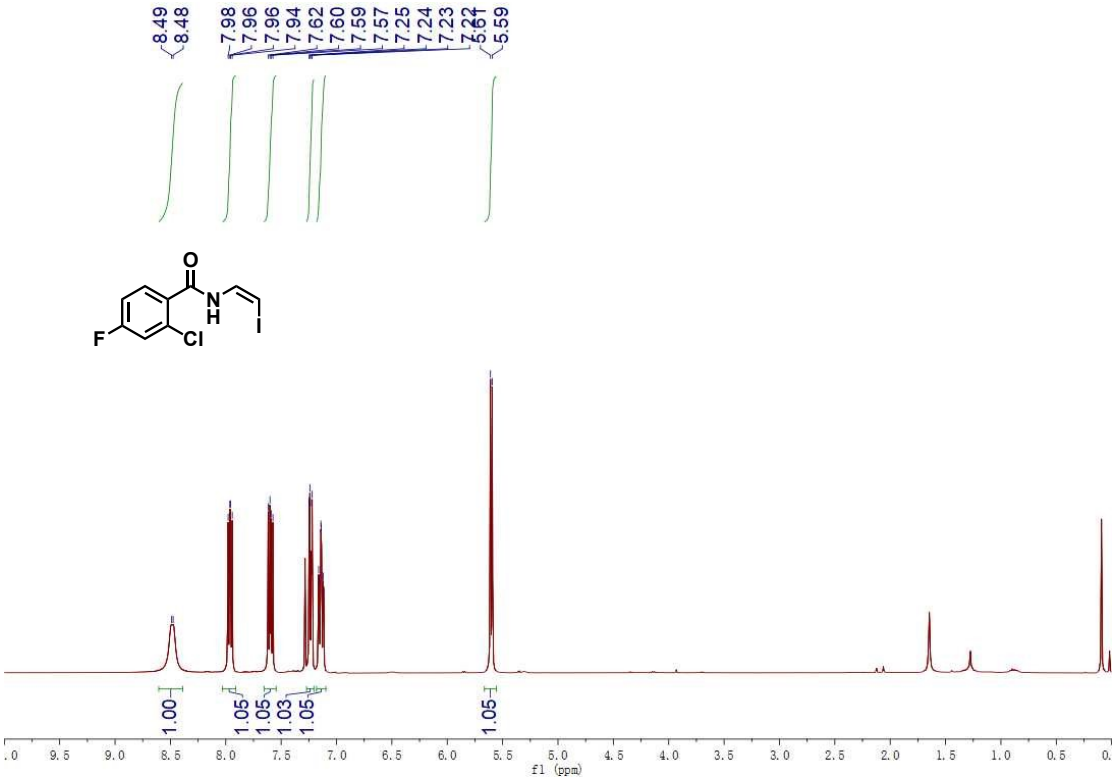
(*Z*)-2,4-dichloro-*N*-(2-iodovinyl) benzamide (2o): ^{1}H NMR (400 MHz, $CDCl_3$)



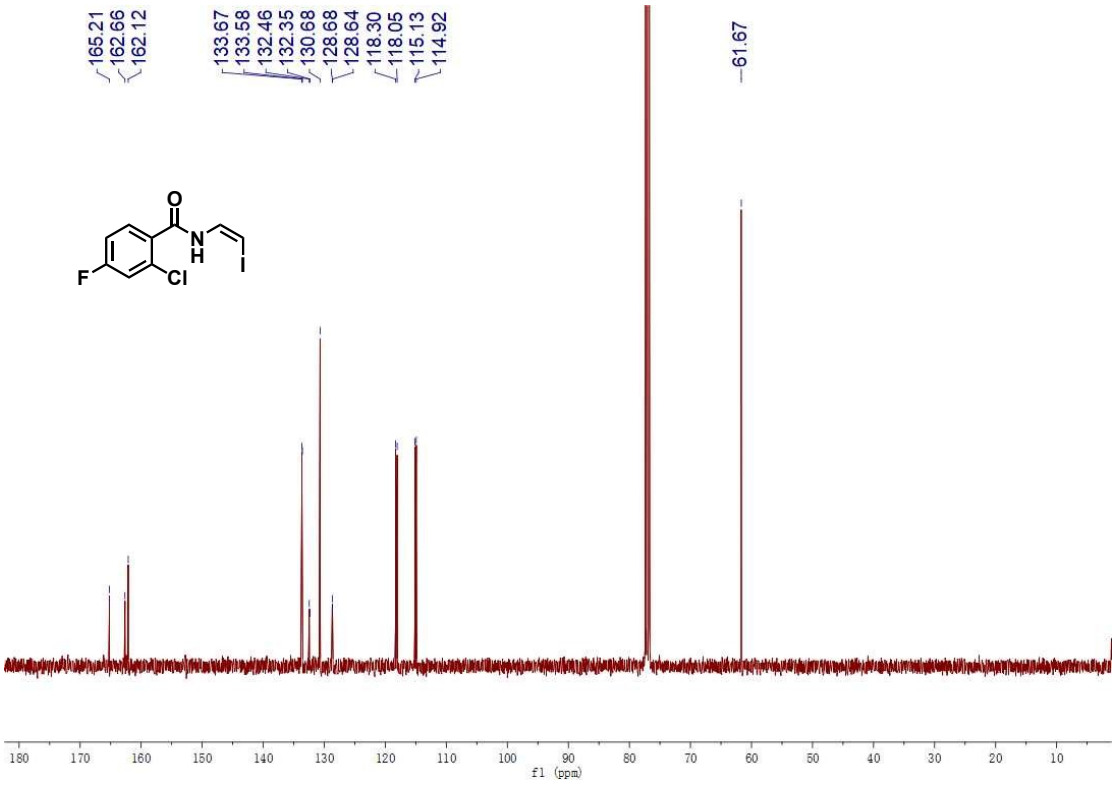
(*Z*)-2,4-dichloro-*N*-(2-iodovinyl) benzamide (2o): ^{13}C NMR (101 MHz, $CDCl_3$)



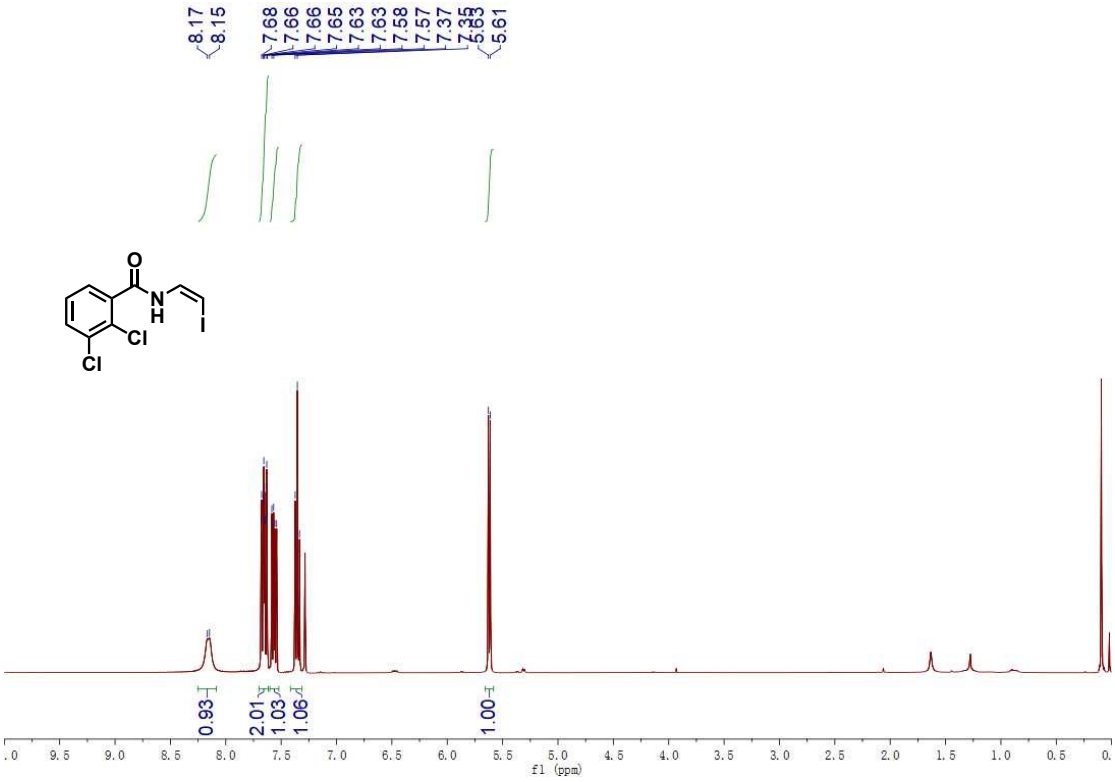
(*Z*)-2-chloro-4-fluoro-*N*-(2-iodovinyl) benzamide (2p): ^{1}H NMR (400 MHz, $CDCl_3$)



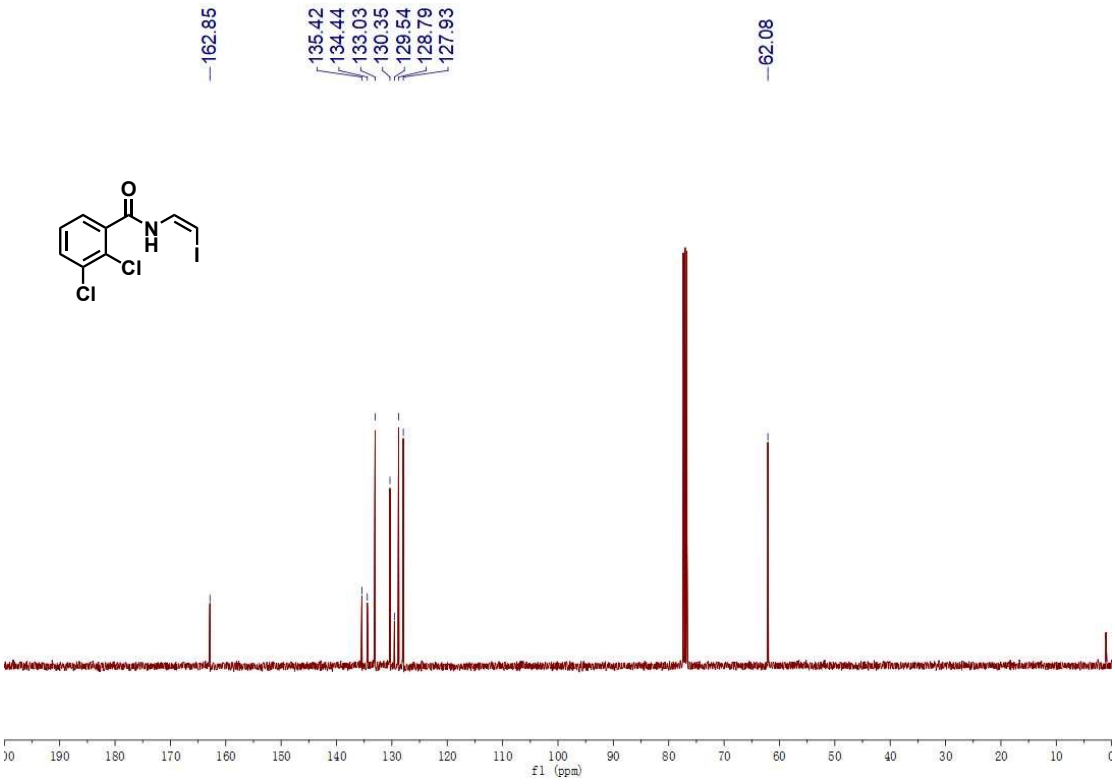
(*Z*)-2-chloro-4-fluoro-*N*-(2-iodovinyl) benzamide (2p): ^{13}C NMR (101 MHz, $CDCl_3$)



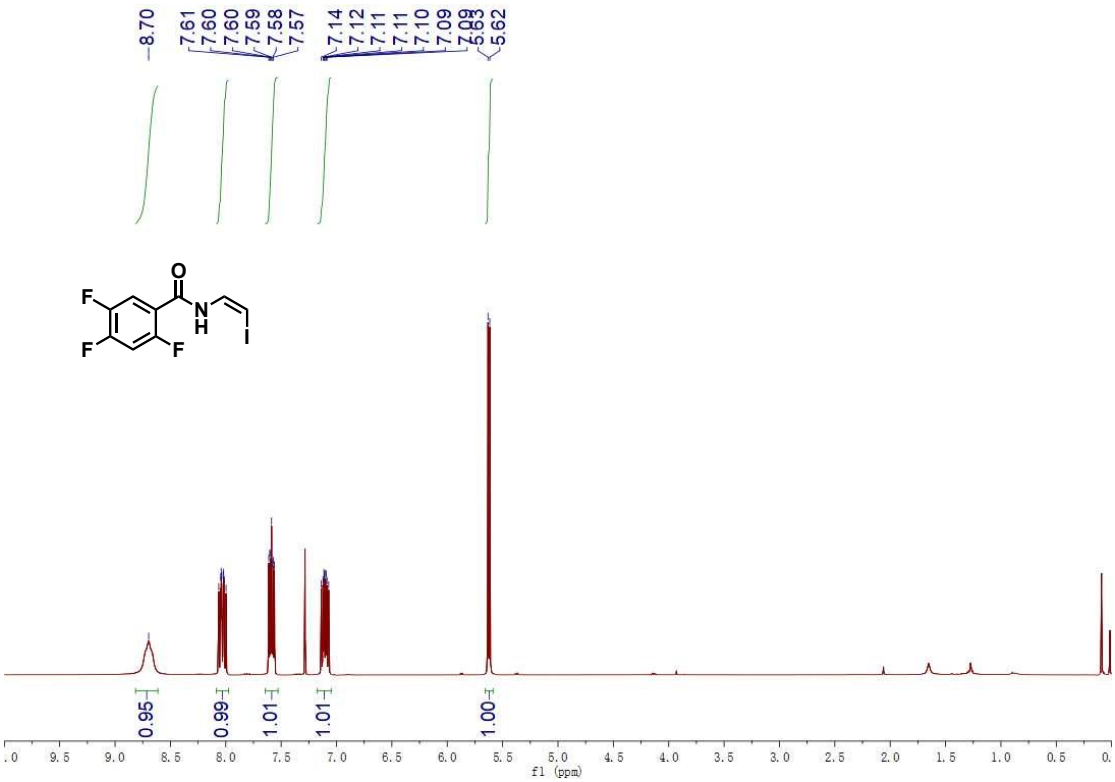
(*Z*)-2,3-dichloro-*N*-(2-iodovinyl) benzamide (2q): ^{1}H NMR (400 MHz, $CDCl_3$)



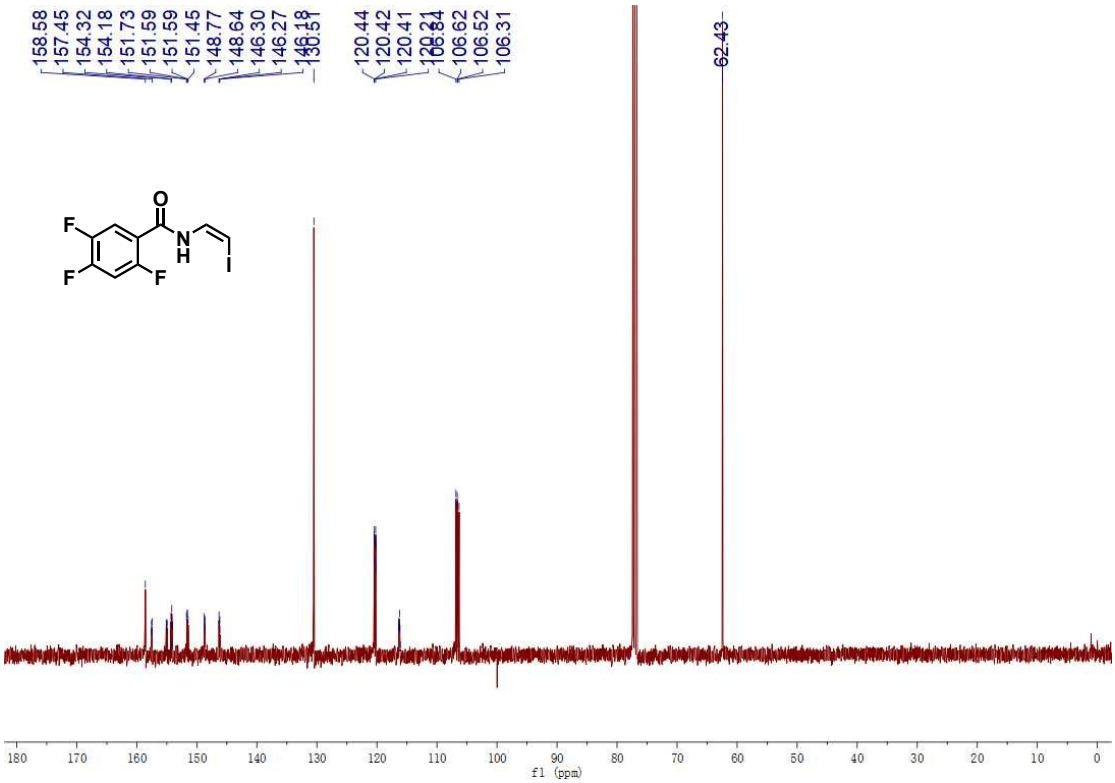
(*Z*)-2,3-dichloro-*N*-(2-iodovinyl) benzamide (2q): ^{13}C NMR (101 MHz, $CDCl_3$)



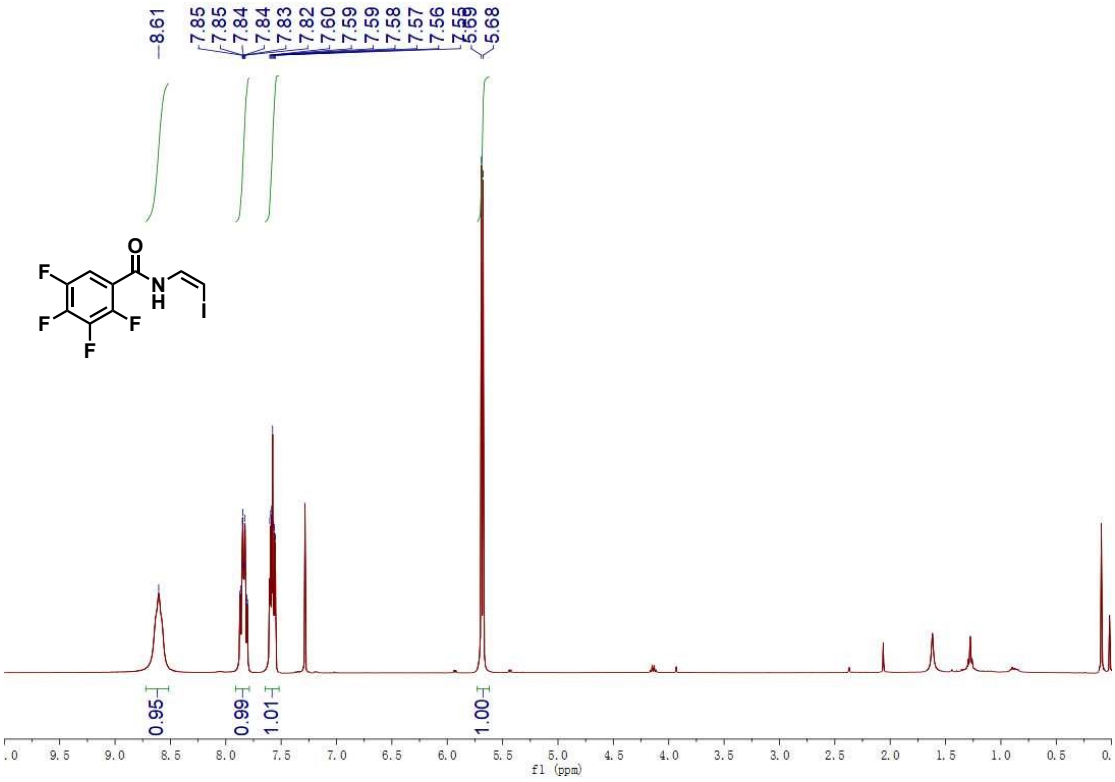
(*Z*)-2,4,5-trifluoro-*N*-(2-iodovinyl) benzamide (2r): ^{1}H NMR (400 MHz, $CDCl_3$)



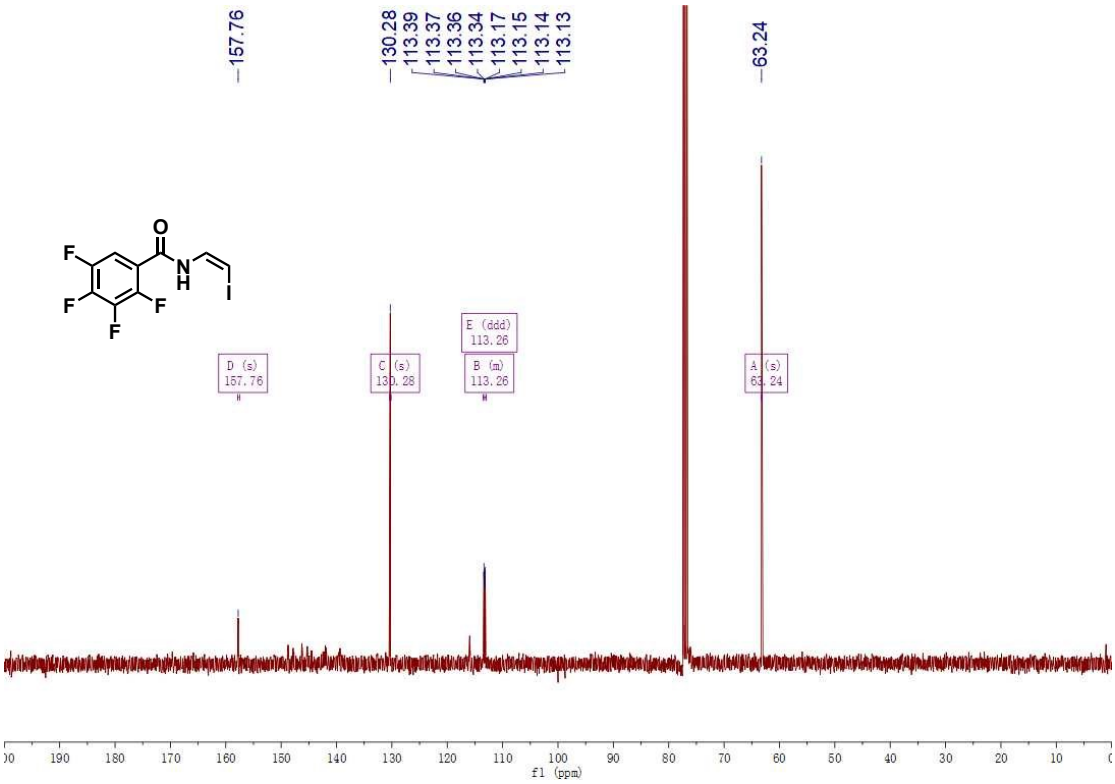
(*Z*)-2,4,5-trifluoro-*N*-(2-iodovinyl) benzamide (2r): ^{13}C NMR (101 MHz, $CDCl_3$)



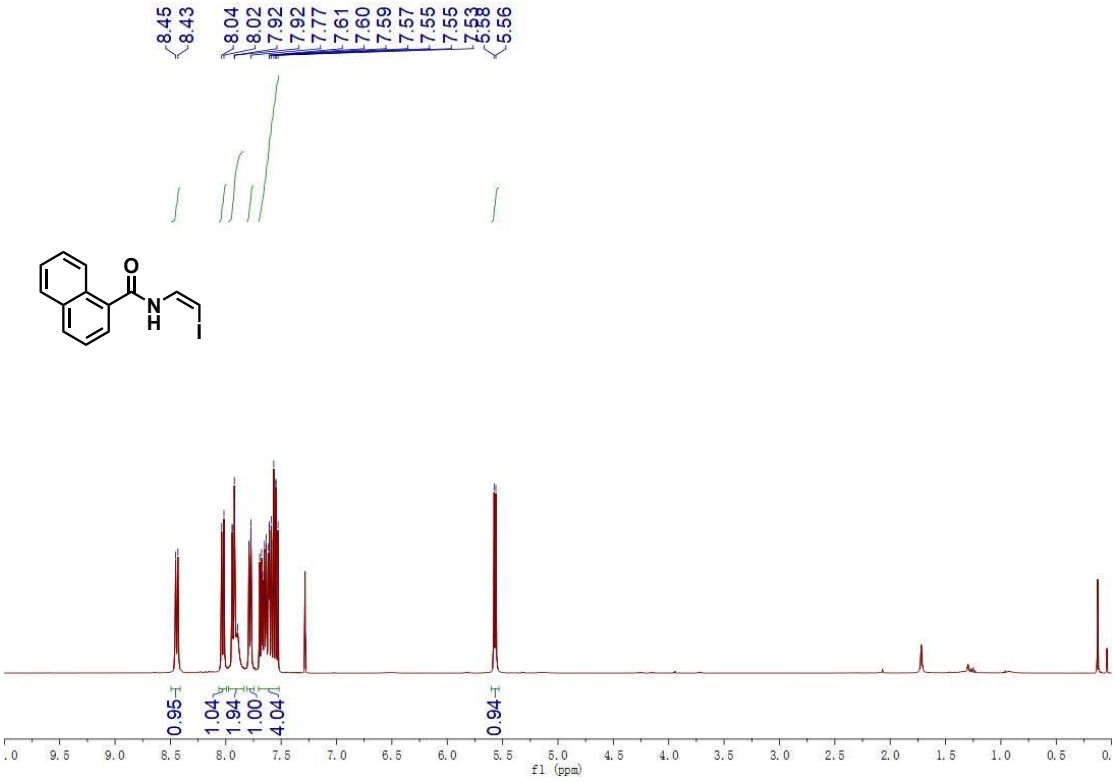
(*Z*)-2,3,4,5-tetrafluoro-*N*-(2-iodovinyl) benzamide (2s): ^{1}H NMR (400 MHz, $CDCl_3$)



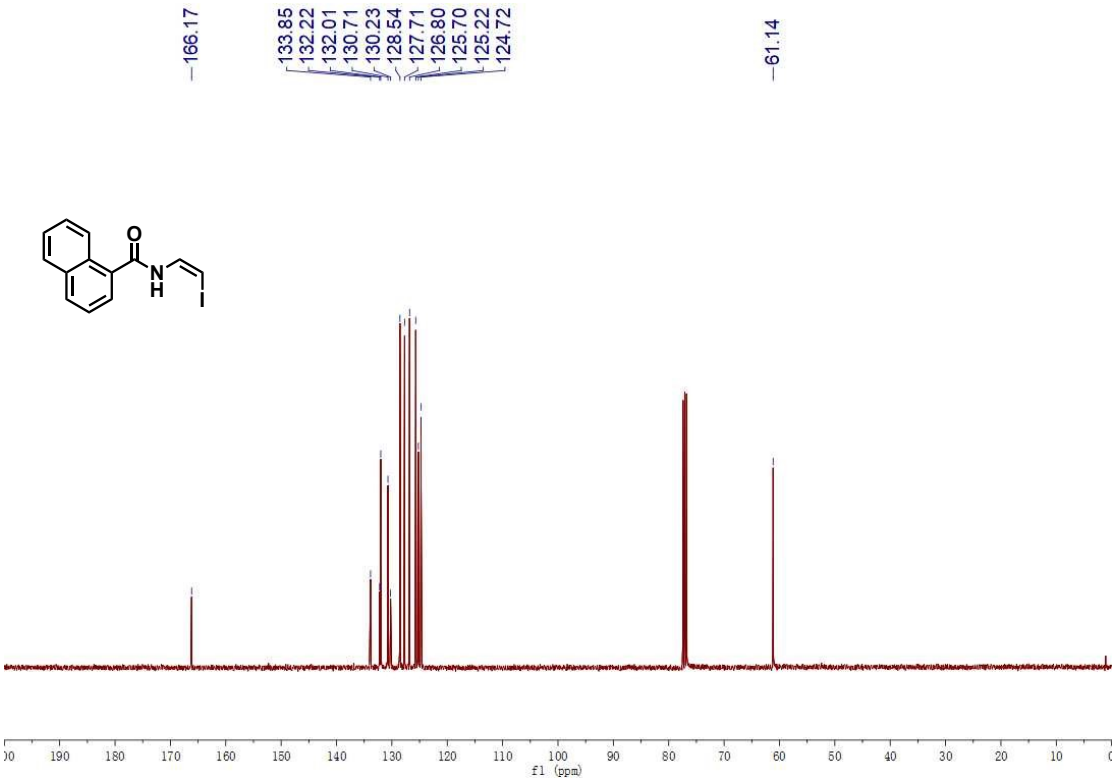
(*Z*)-2,3,4,5-tetrafluoro-*N*-(2-iodovinyl) benzamide (2s): ^{13}C NMR (101 MHz, $CDCl_3$)



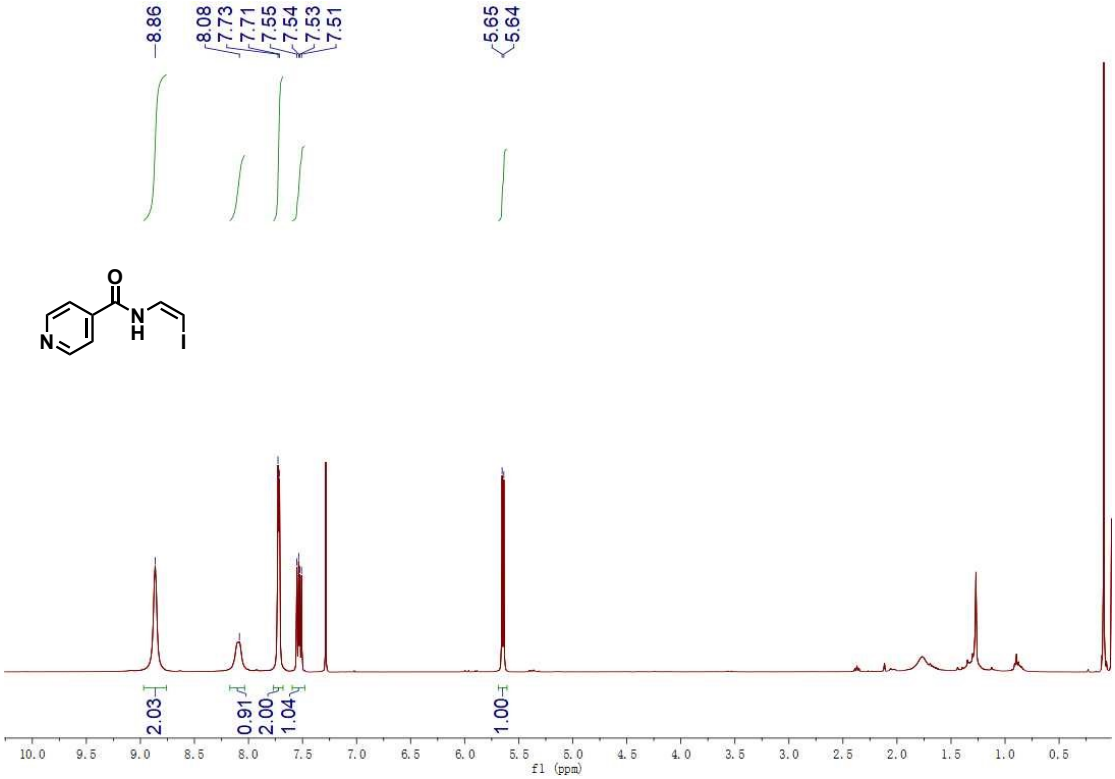
(*Z*)-*N*-(2-iodovinyl)-1-naphthamide (2t): ^{1}H NMR (400 MHz, $CDCl_3$)



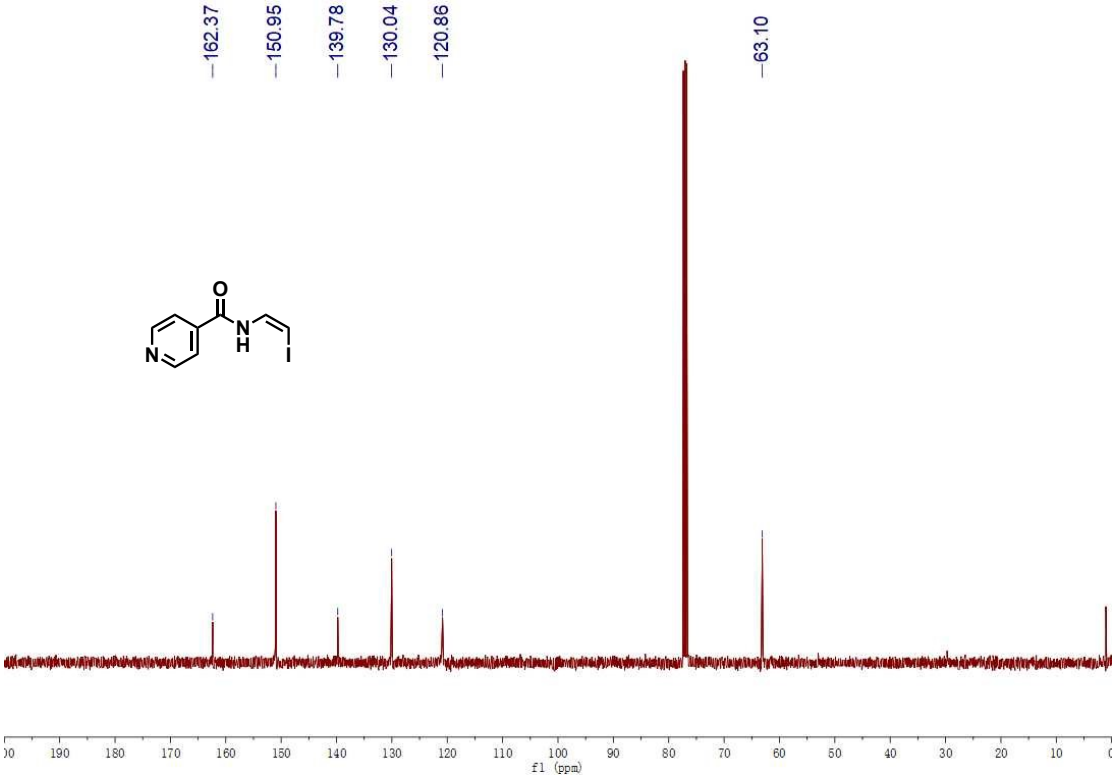
(*Z*)-*N*-(2-iodovinyl)-1-naphthamide (2t):^{13}C NMR (101 MHz, $CDCl_3$)



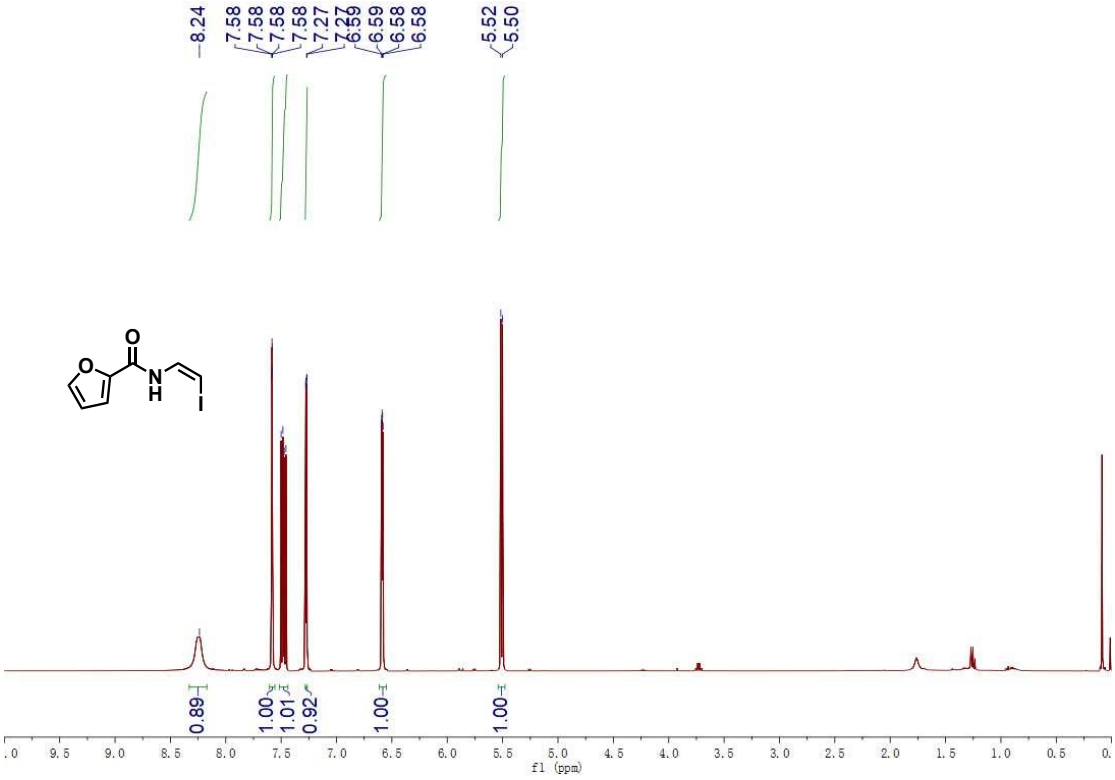
(*Z*)-*N*-(2-iodovinyl) isonicotinamide (2u): ^{1}H NMR (400 MHz, $CDCl_3$)



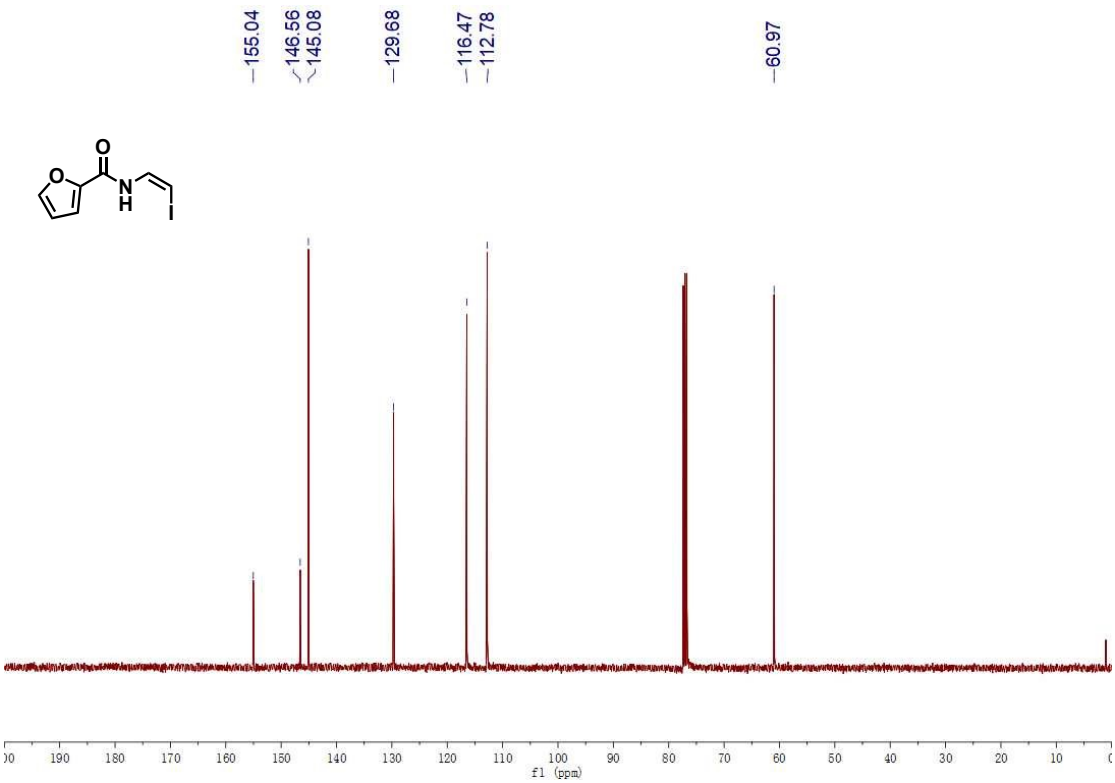
(*Z*)-*N*-(2-iodovinyl) isonicotinamide (2u): ^{13}C NMR (101 MHz, $CDCl_3$)



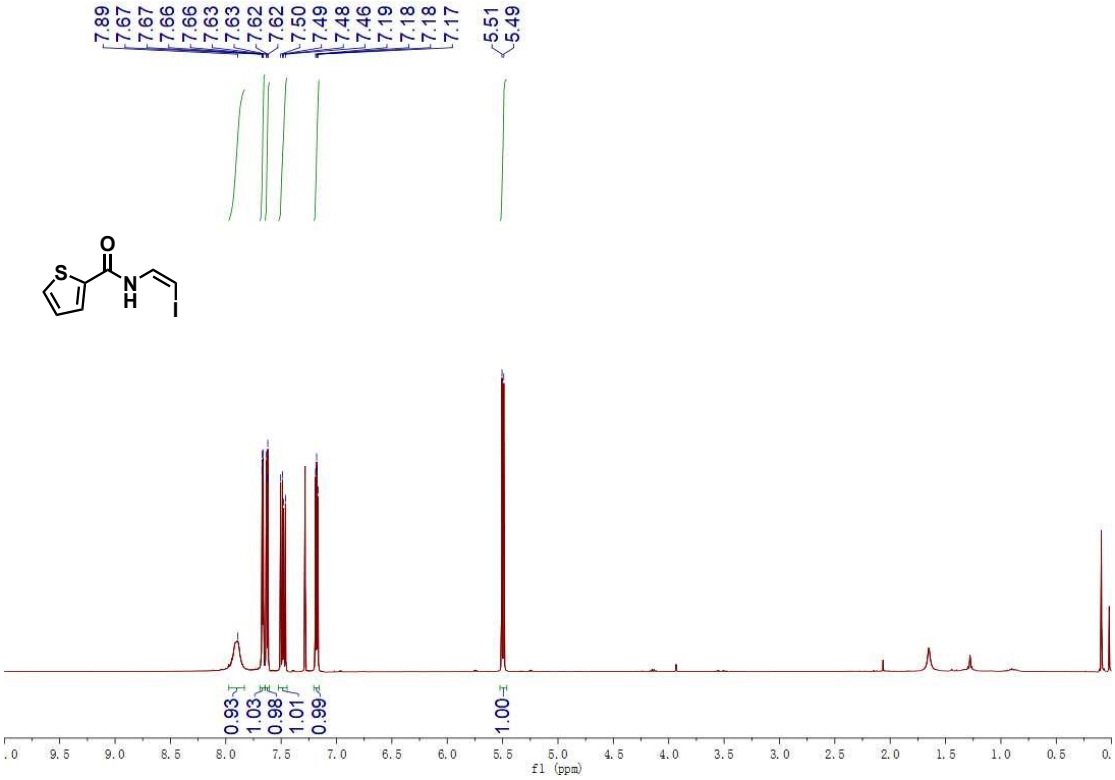
(*Z*)-*N*-(2-iodovinyl) furan-2-carboxamide (2v): ^{1}H NMR (400 MHz, $CDCl_3$)



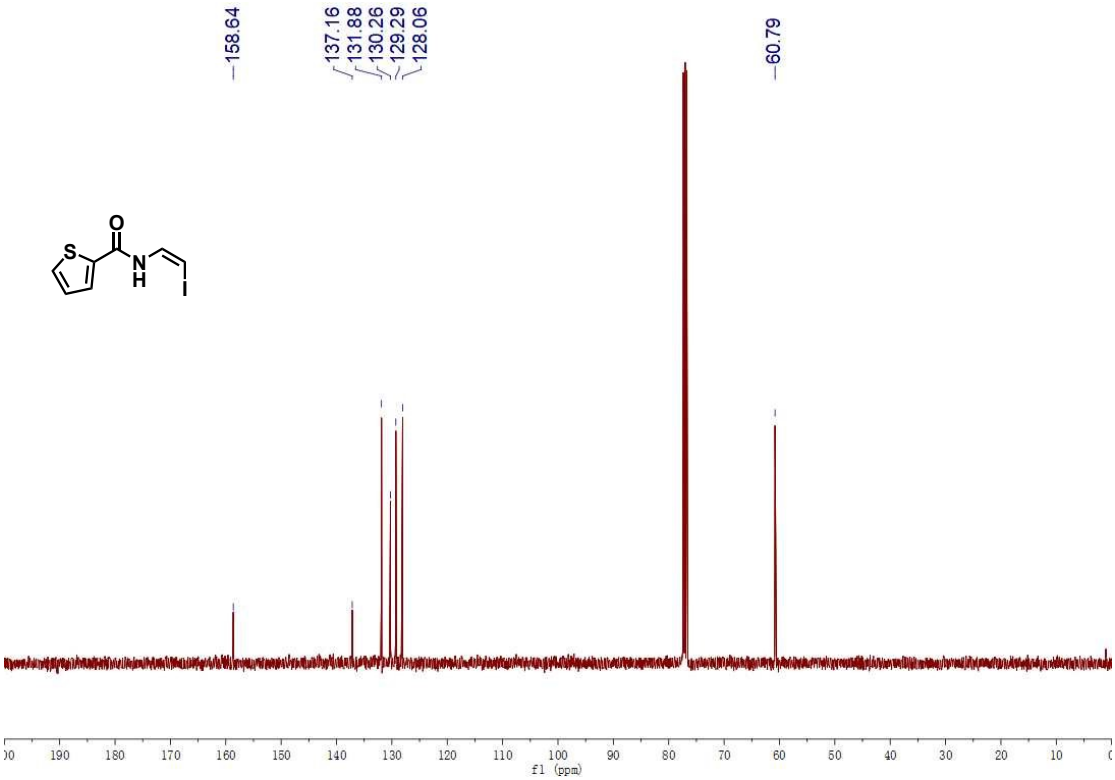
(*Z*)-*N*-(2-iodovinyl) furan-2-carboxamide (2v): ^{13}C NMR (101 MHz, $CDCl_3$)



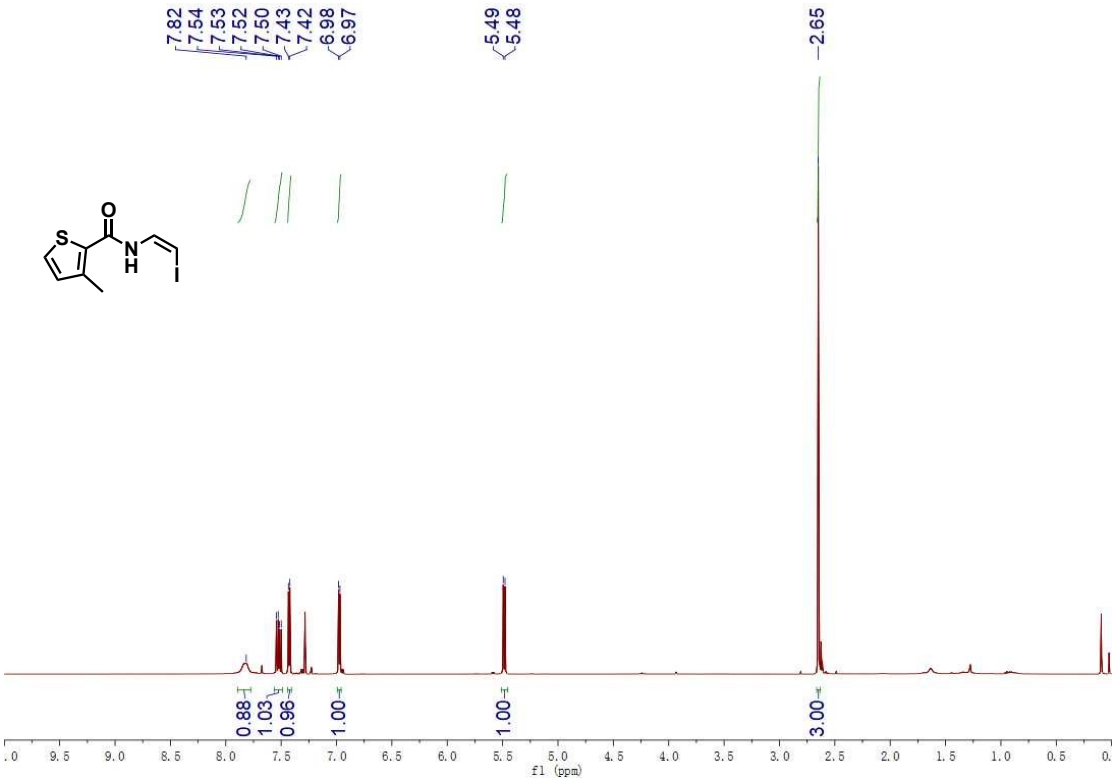
(*Z*)-*N*-(2-iodovinyl) thiophene-2-carboxamide (2w): ^{1}H NMR (400 MHz, $CDCl_3$)



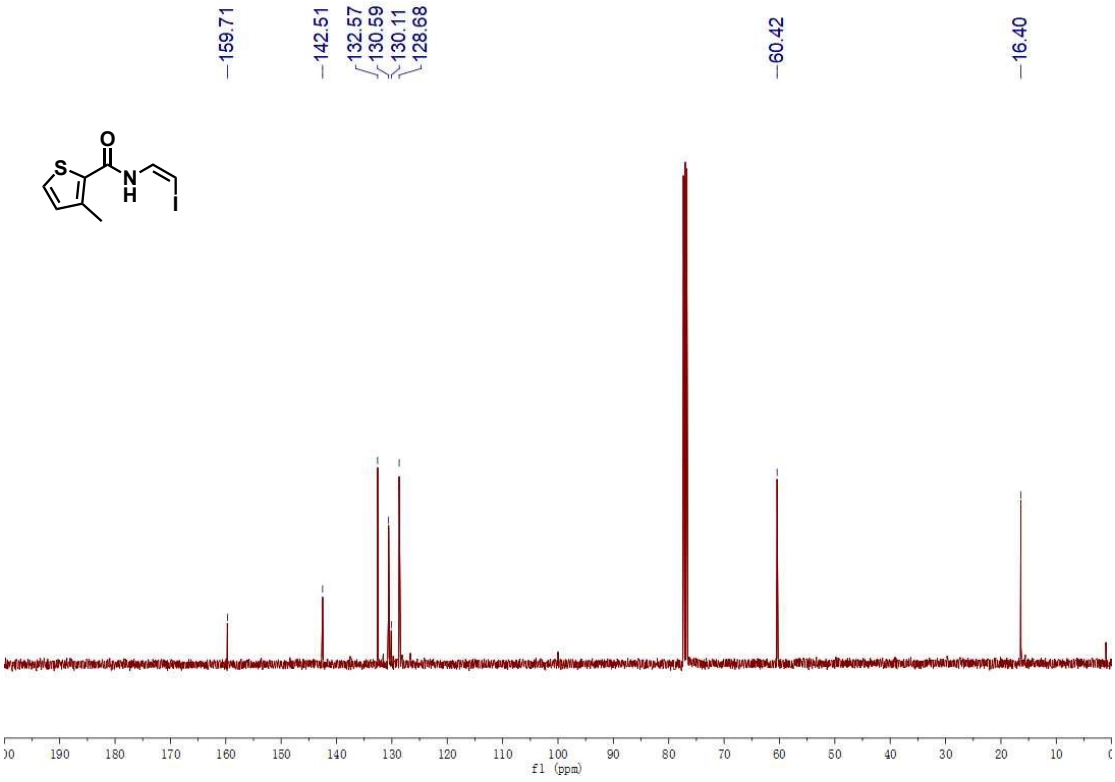
(*Z*)-*N*-(2-iodovinyl) thiophene-2-carboxamide (2w):^{13}C NMR (101 MHz, $CDCl_3$)



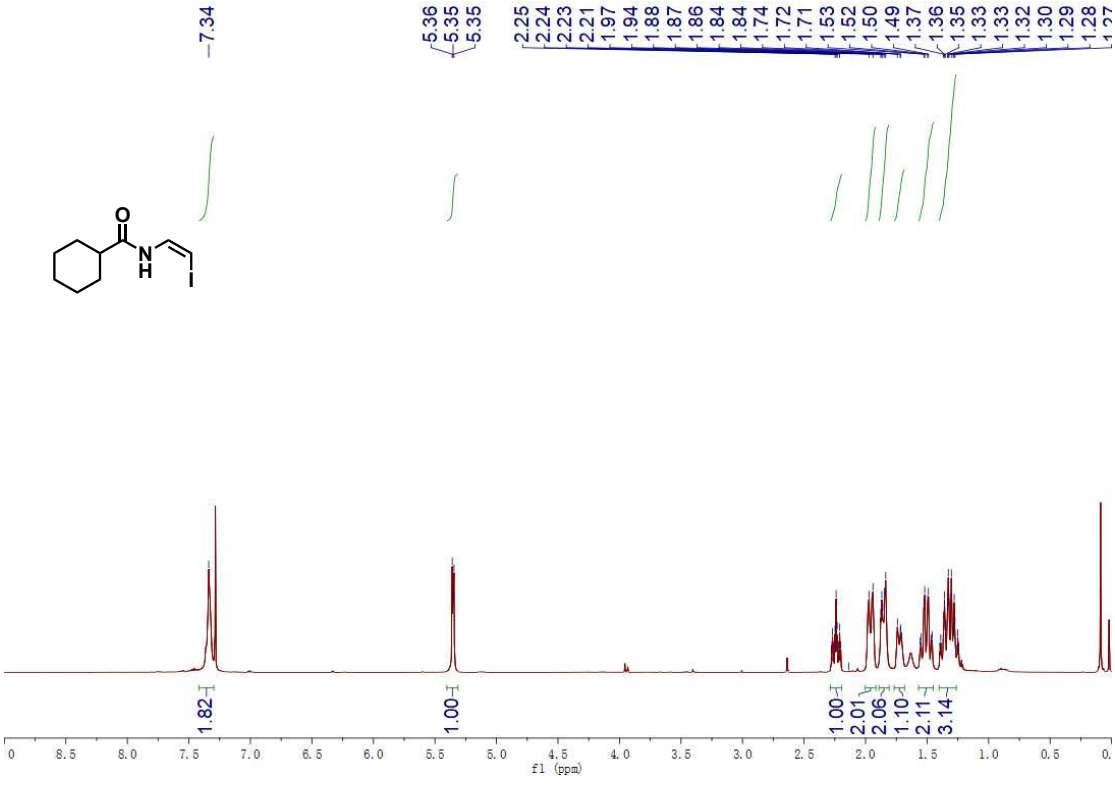
(*Z*)-*N*-(2-iodovinyl)-3-methylthiophene-2-carboxamide (2x): ^{1}H NMR (400 MHz, $CDCl_3$)



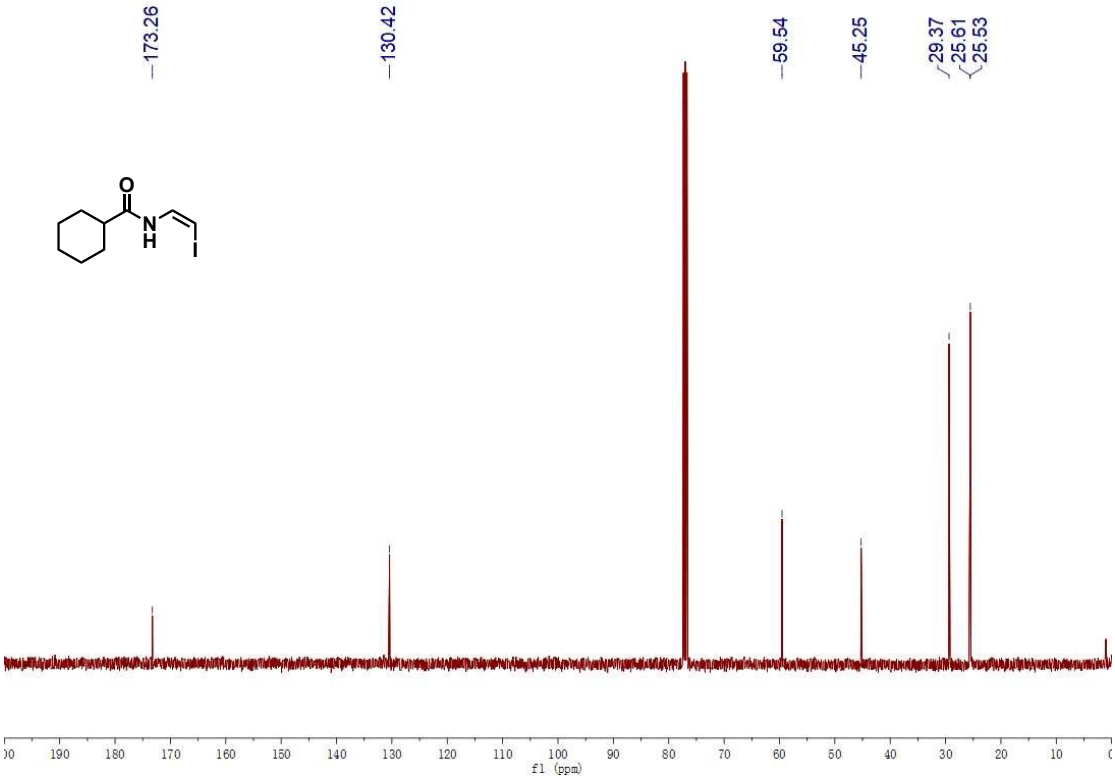
(*Z*)-*N*-(2-iodovinyl)-3-methylthiophene-2-carboxamide (2x):^{13}C NMR (101 MHz, $CDCl_3$)



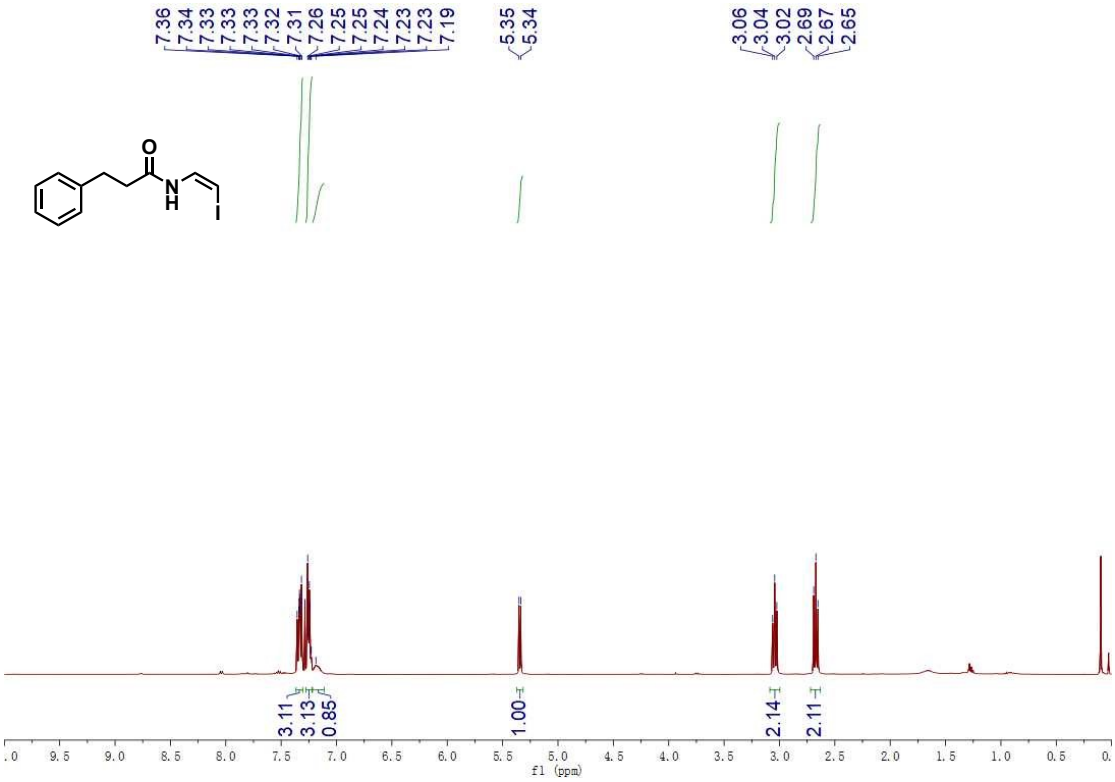
(*Z*)-*N*-(2-iodovinyl) cyclohexanecarboxamide (2y): ^{1}H NMR (400 MHz, $CDCl_3$)



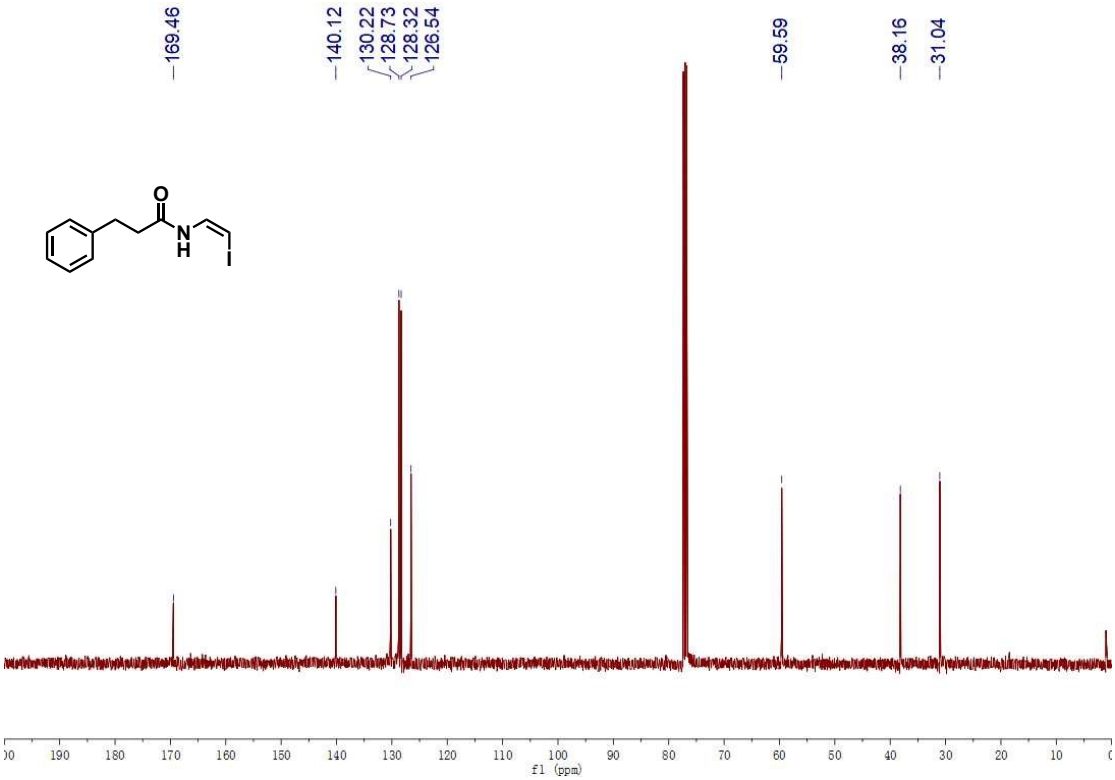
(*Z*)-*N*-(2-iodovinyl) cyclohexanecarboxamide (2y): ^{13}C NMR (101 MHz, $CDCl_3$)



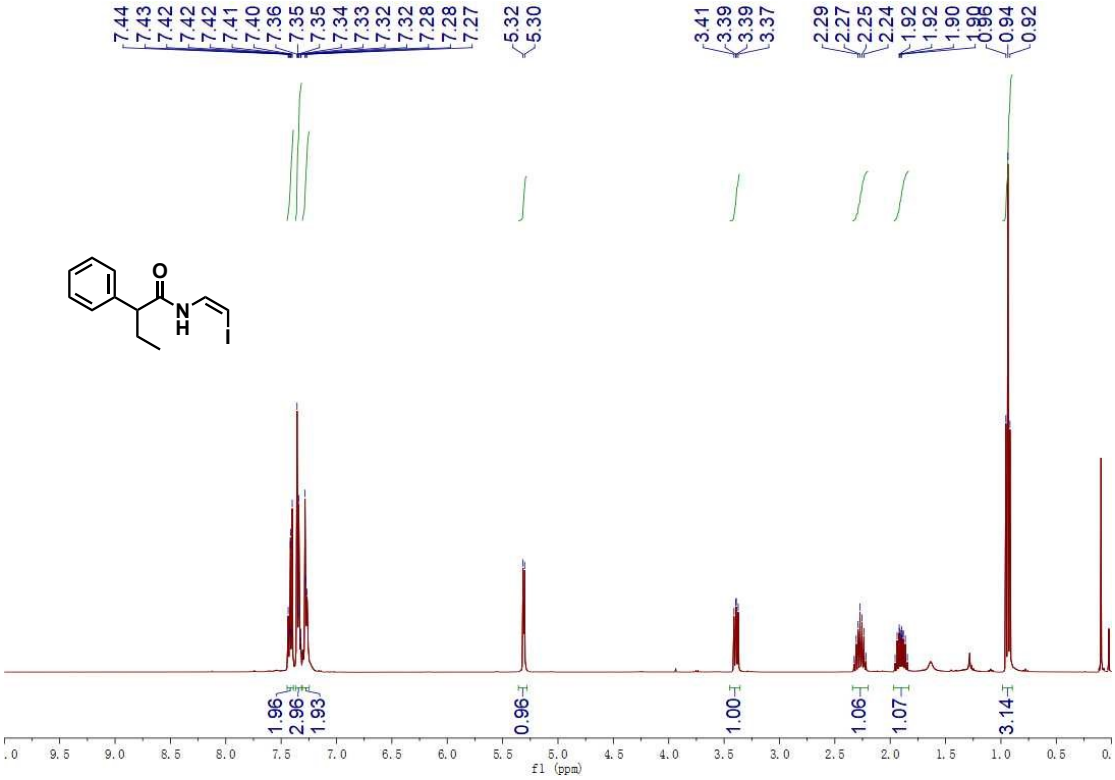
(*Z*)-*N*-(2-iodovinyl)-3-phenylpropanamide (2z): ^{1}H NMR (400 MHz, $CDCl_3$)



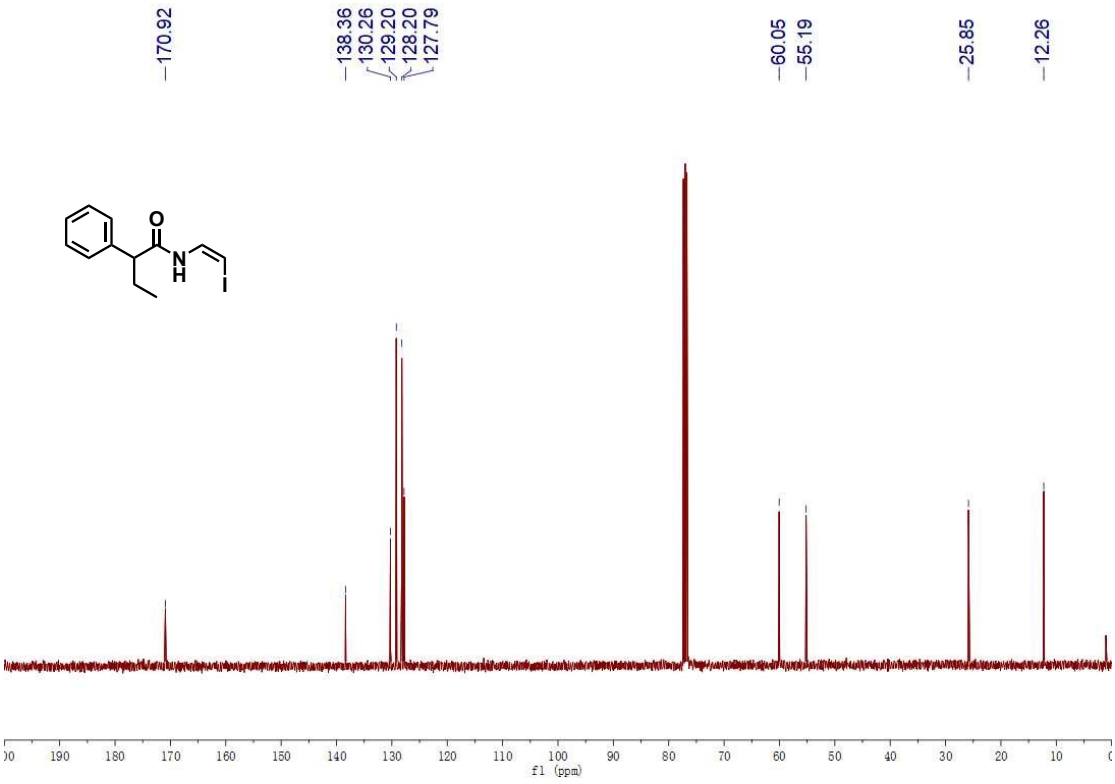
(*Z*)-*N*-(2-iodovinyl)-3-phenylpropanamide (2z): ^{13}C NMR (101 MHz, $CDCl_3$)



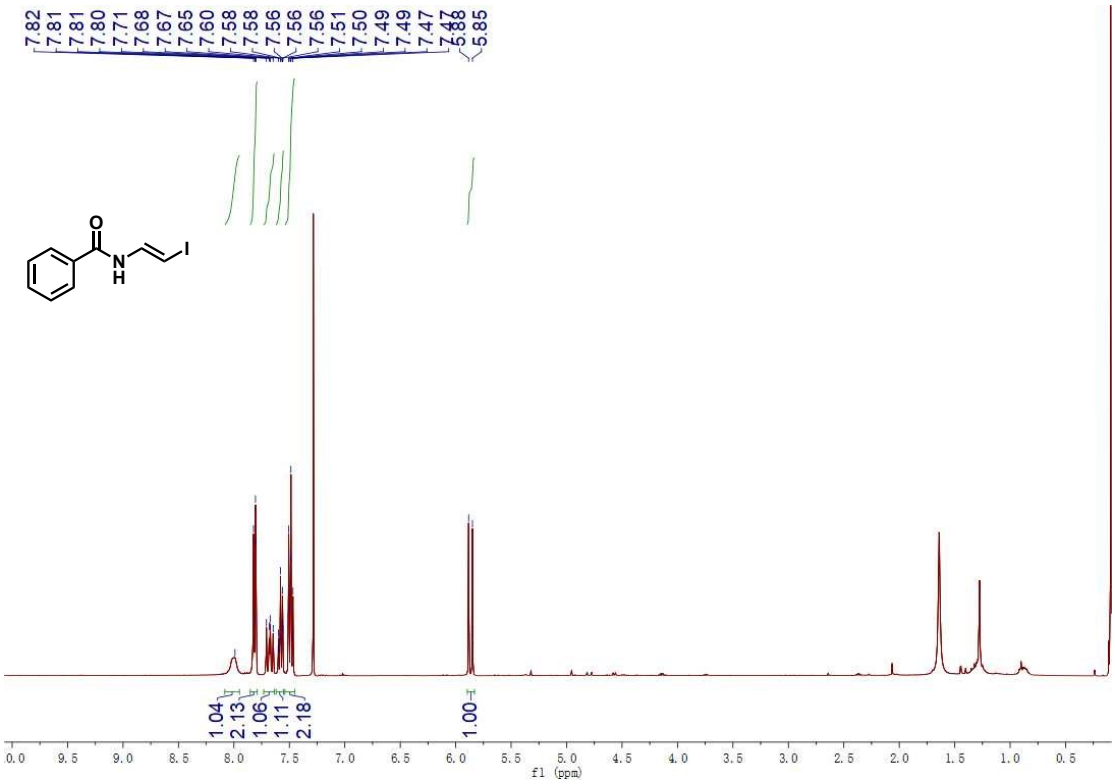
(*Z*)-*N*-(2-iodovinyl)-2-phenylbutanamide (2aa): ^{1}H NMR (400 MHz, $CDCl_3$)



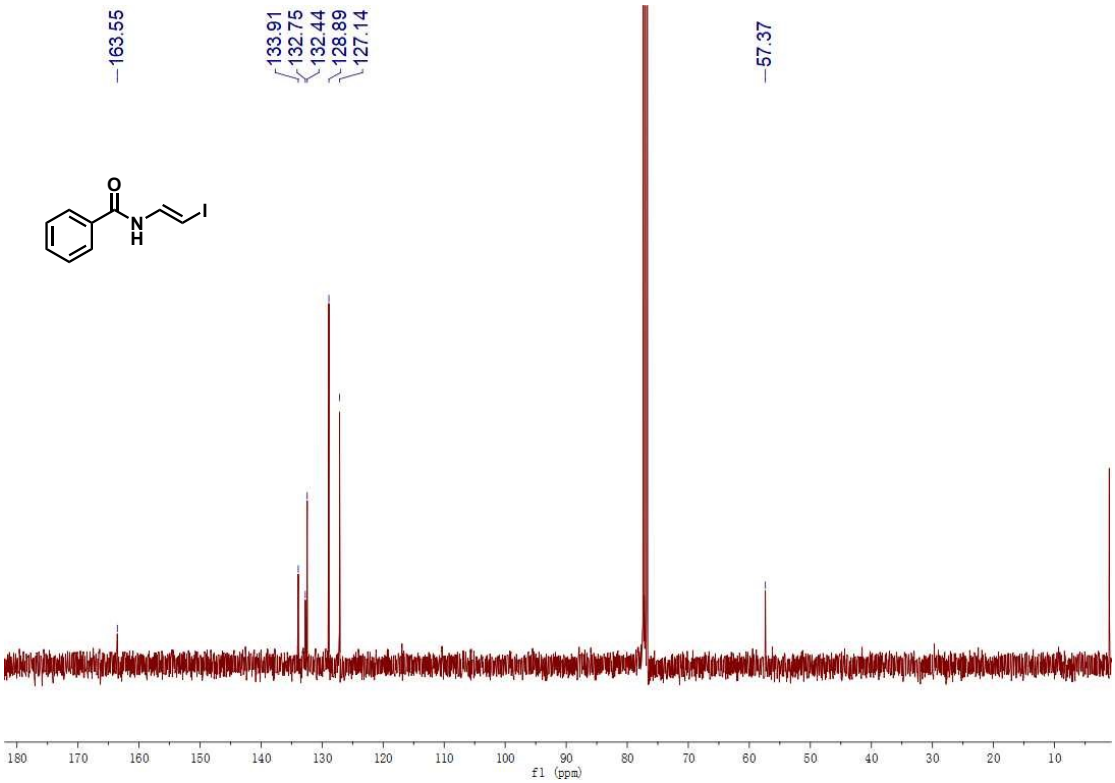
(*Z*)-*N*-(2-iodovinyl)-2-phenylbutanamide (2aa): ^{13}C NMR (101 MHz, $CDCl_3$)



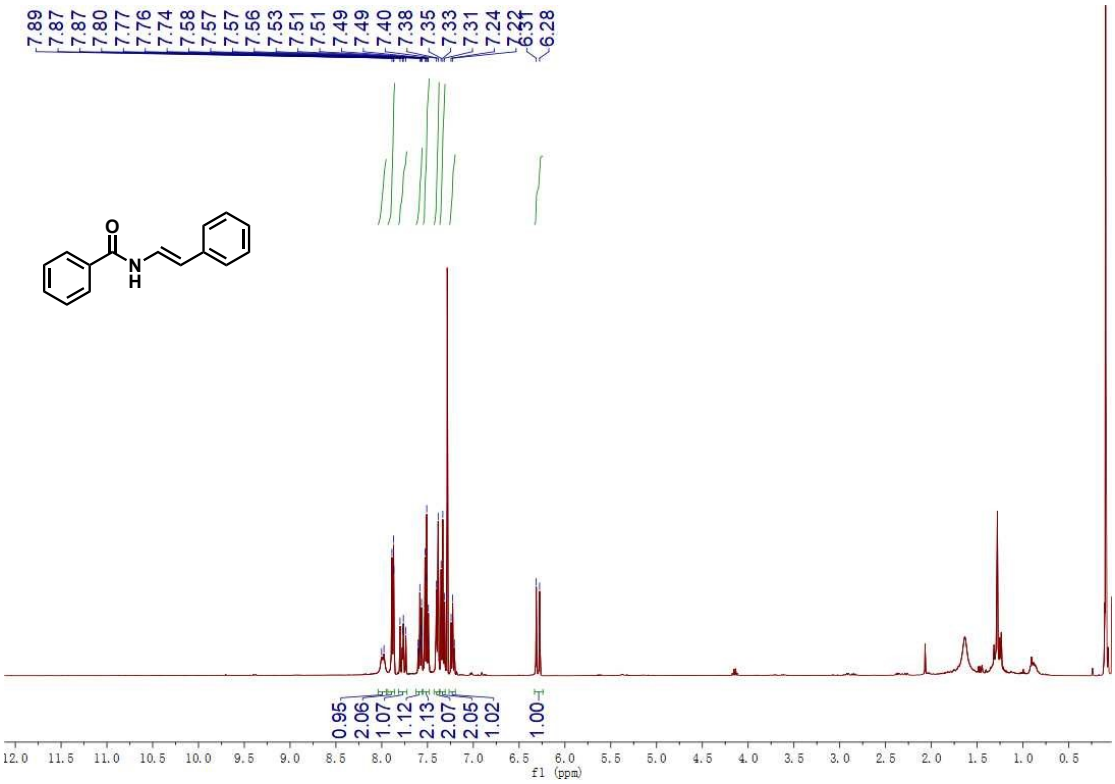
(*E*)-*N*-(2-iodovinyl) benzamide (3): ^{1}H NMR (400 MHz, $CDCl_3$)



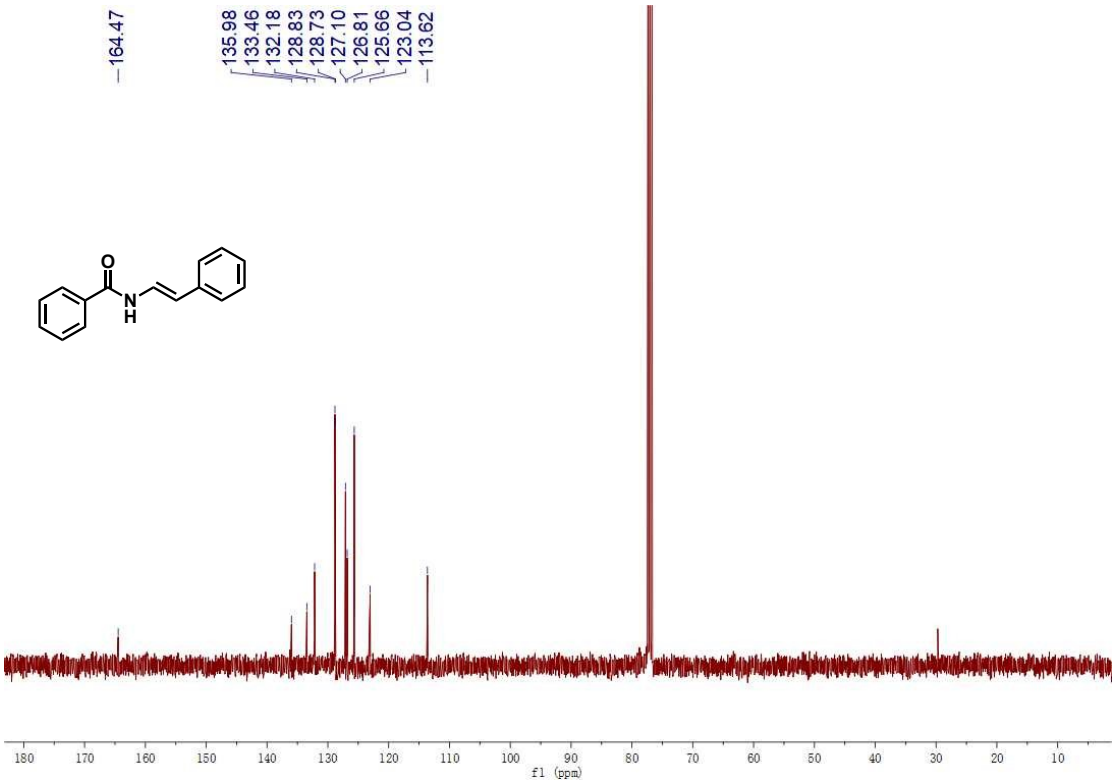
(*E*)-*N*-(2-iodovinyl) benzamide (3): ^{13}C NMR (101 MHz, $CDCl_3$)



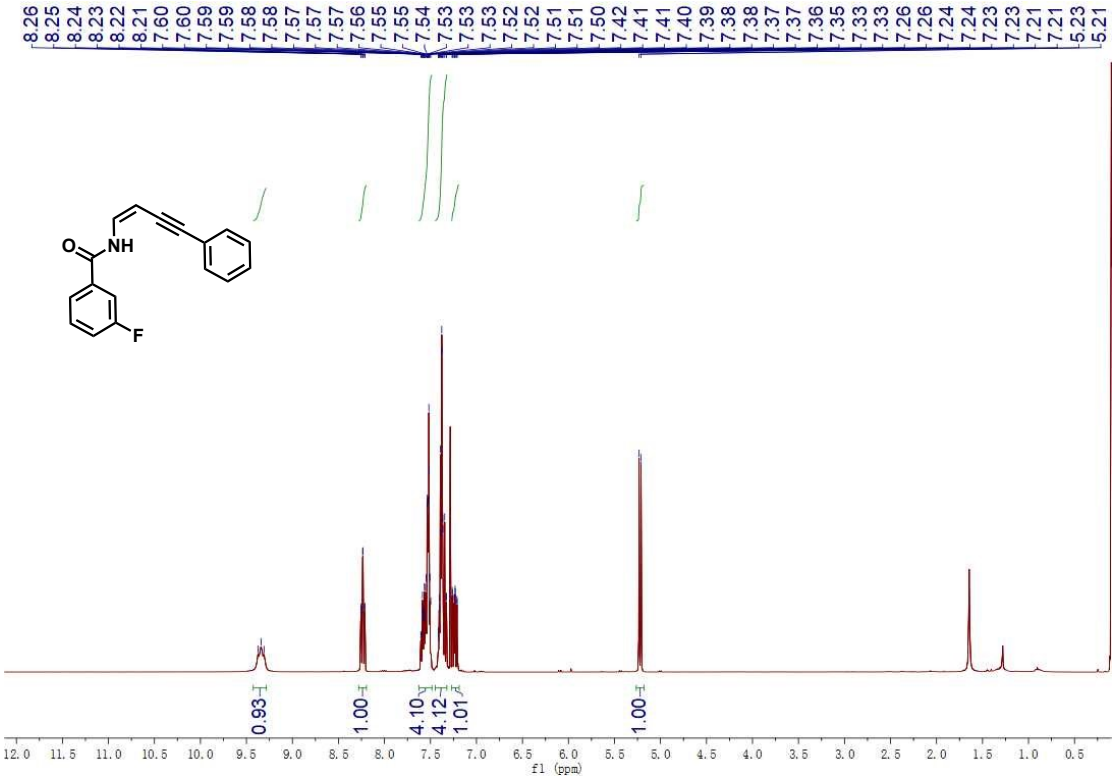
(*Z*)-*N*-styrylbenzamide (4): ^{1}H NMR (400 MHz, $CDCl_3$)



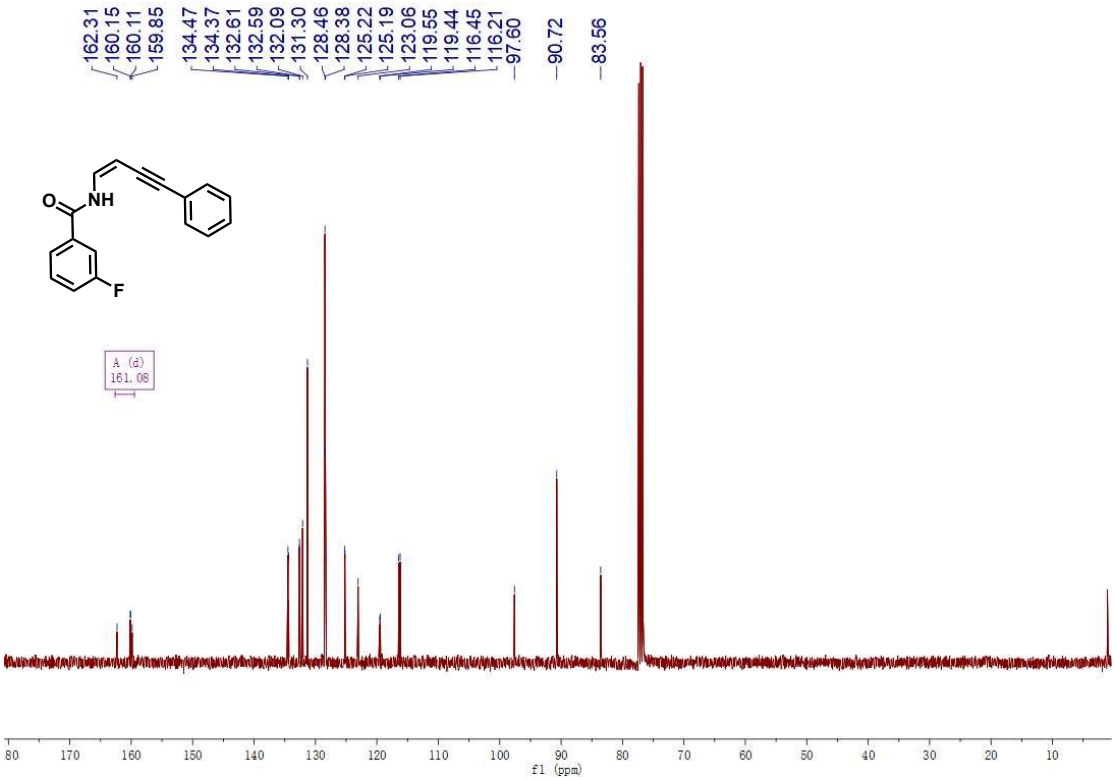
(*Z*)-*N*-styrylbenzamide (4): ^{13}C NMR (101 MHz, $CDCl_3$)



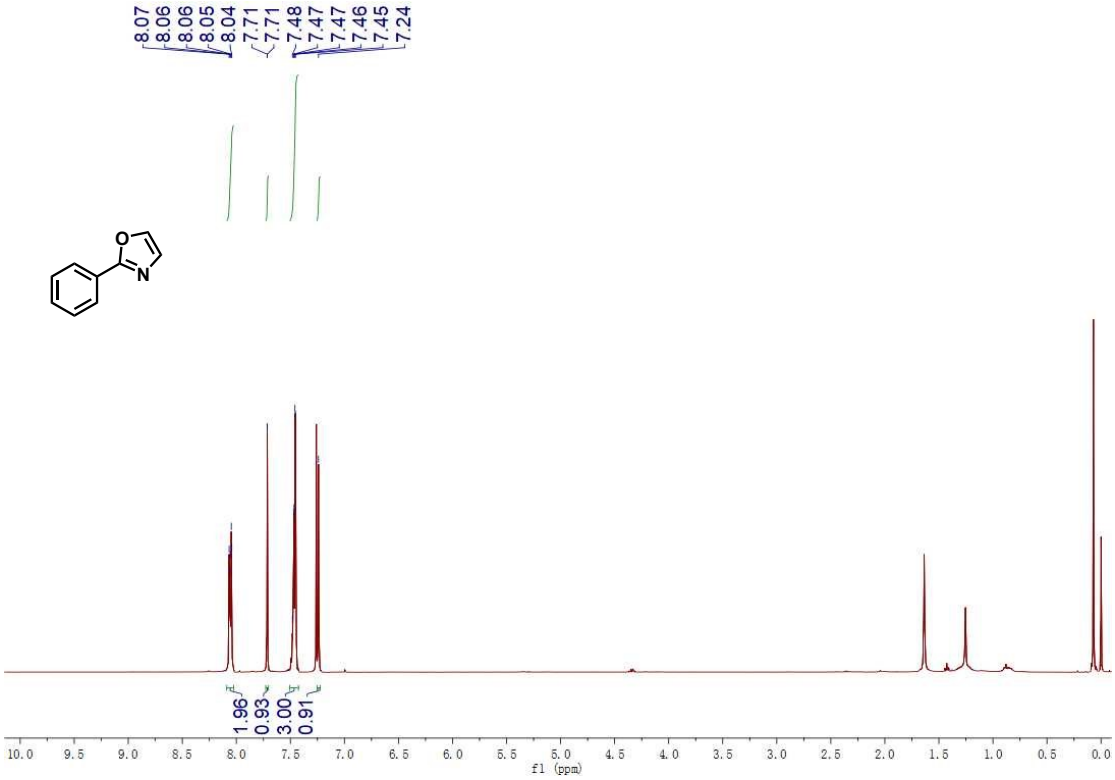
(*Z*)-2-fluoro-*N*-(4-phenylbut-1-en-3-yn-1-yl) benzamide (5): ^{1}H NMR (400 MHz, $CDCl_3$)



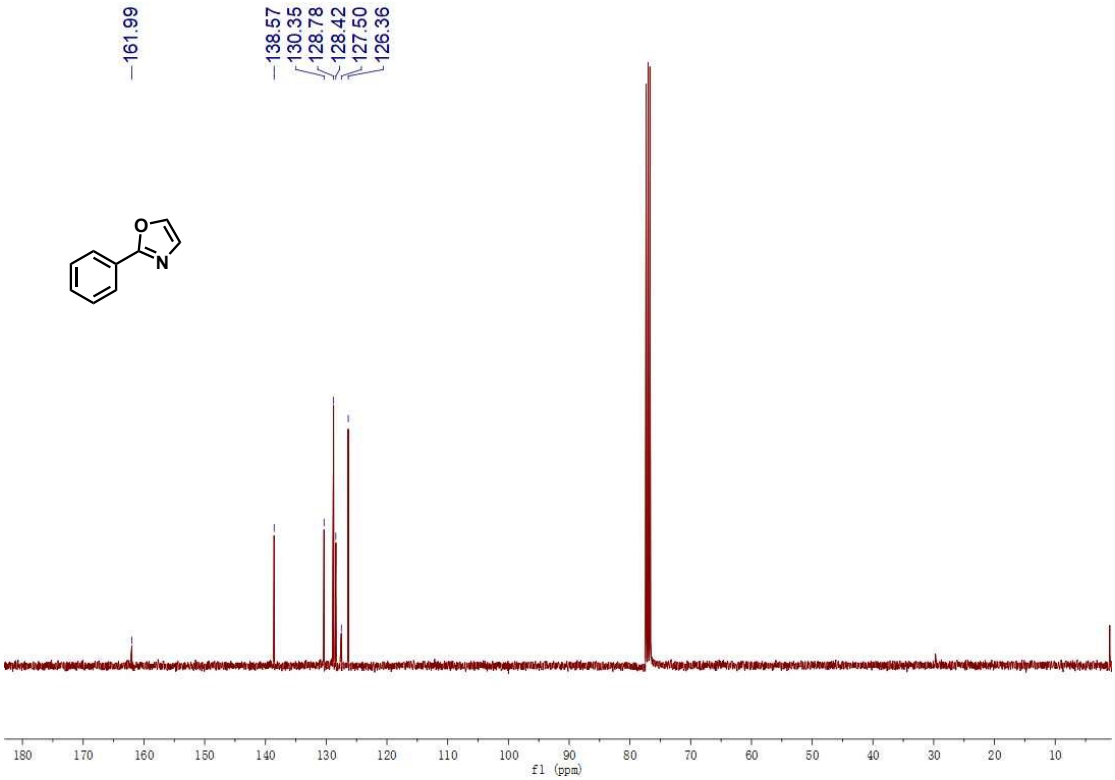
(*Z*)-2-fluoro-*N*-(4-phenylbut-1-en-3-yn-1-yl) benzamide (5): ^{13}C NMR (101 MHz, $CDCl_3$)



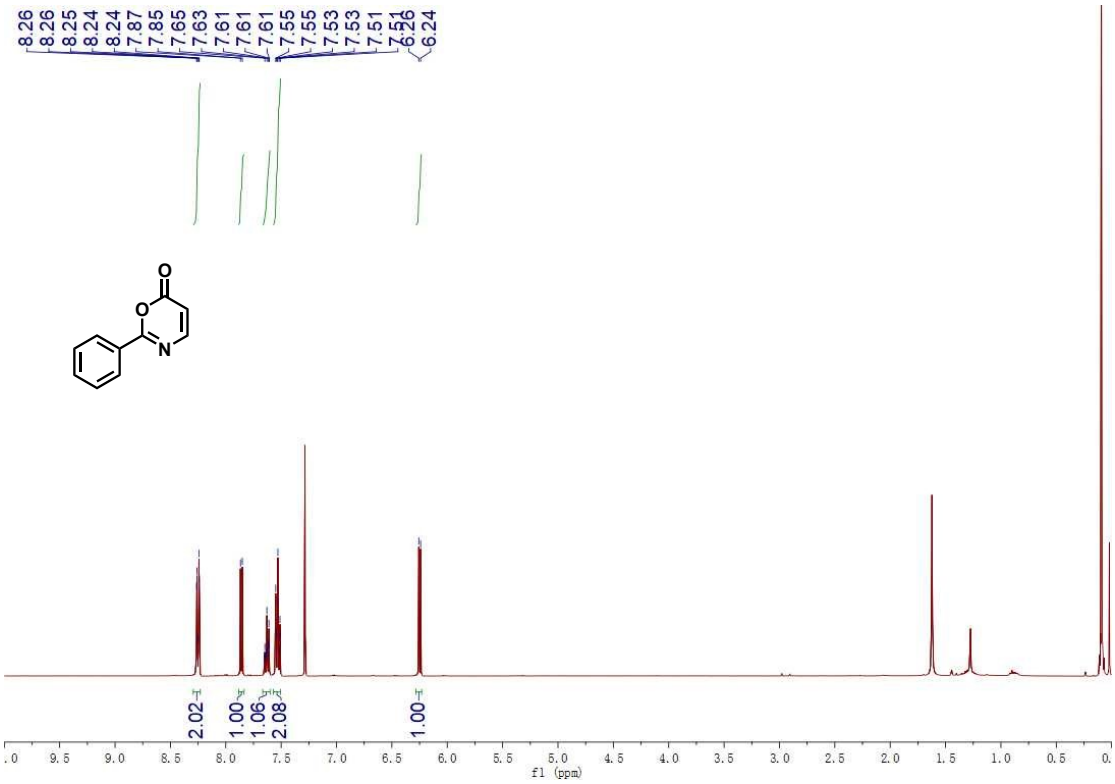
2-phenyl oxazole (6): ^{1}H NMR (400 MHz, $CDCl_3$)



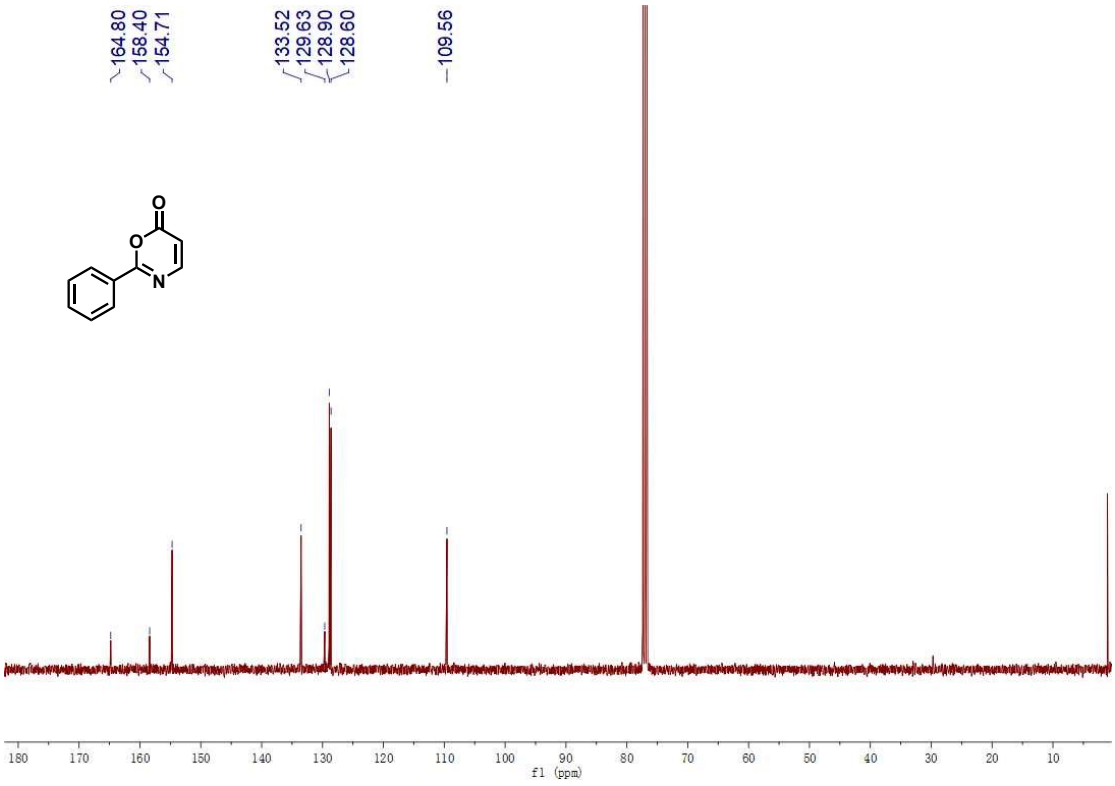
2-phenyl oxazole (6): ^{13}C NMR (101 MHz, $CDCl_3$)



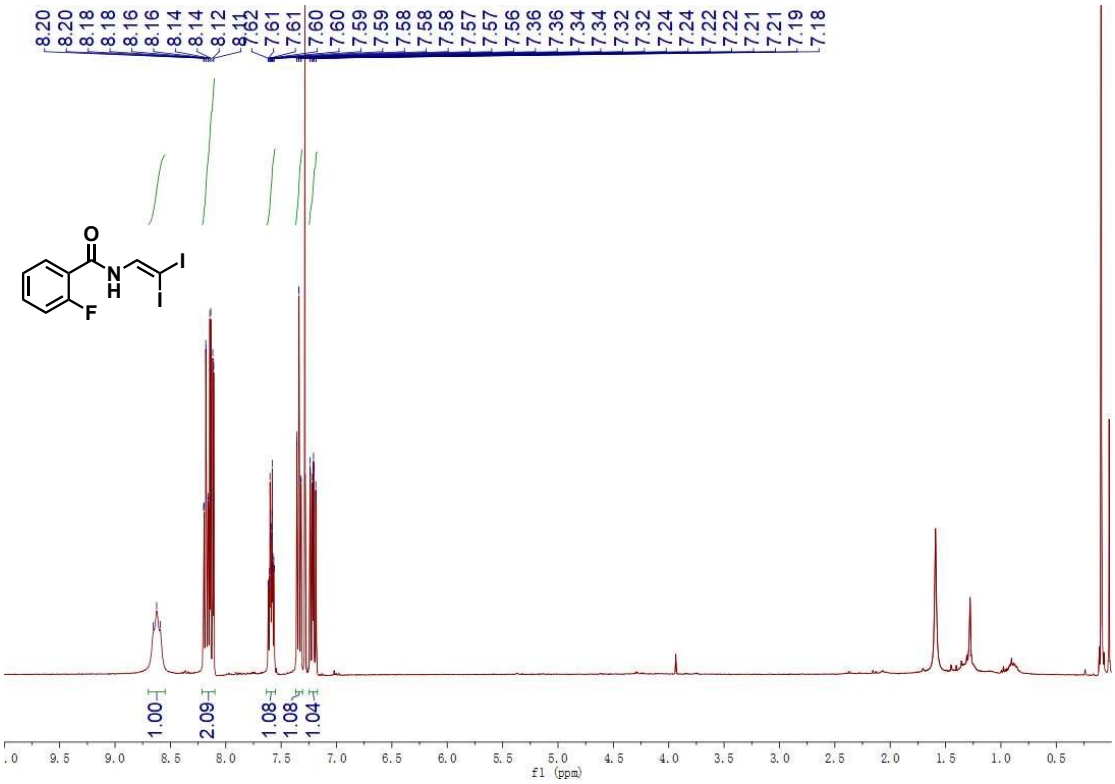
2-phenyl-6H-1,3-oxazin-6-one (7): ^{1}H NMR (400 MHz, $CDCl_3$)



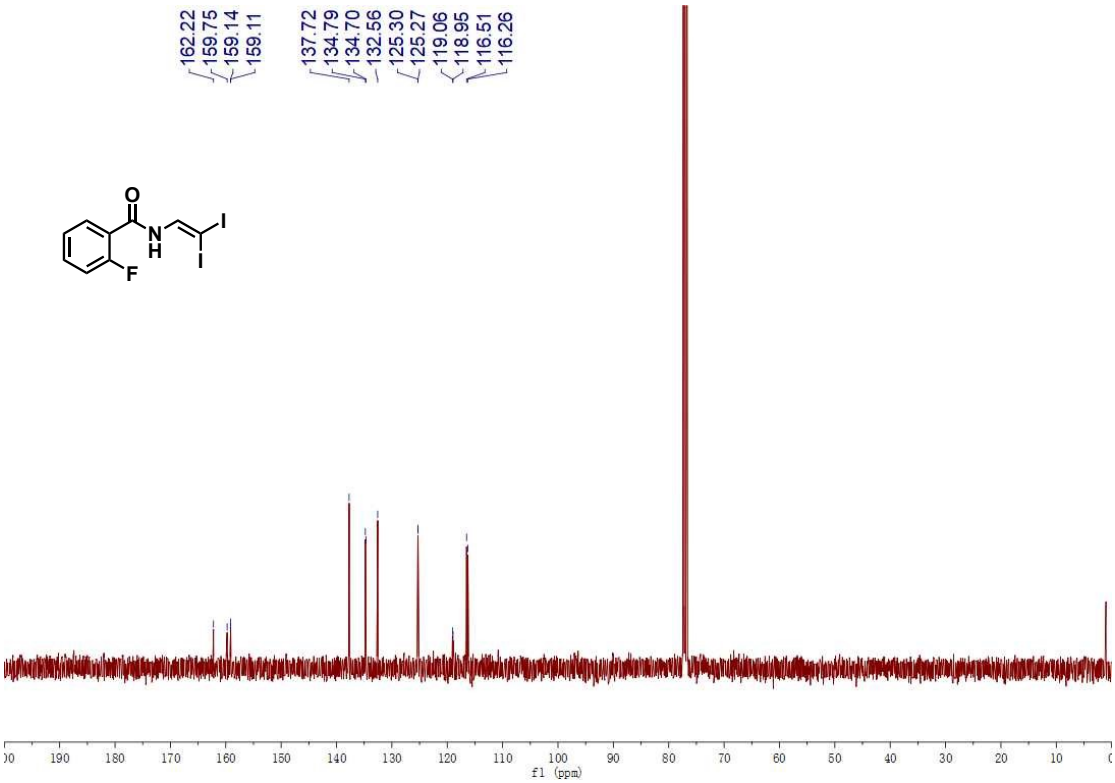
2-phenyl-6H-1,3-oxazin-6-one (7): ^{13}C NMR (101 MHz, $CDCl_3$)



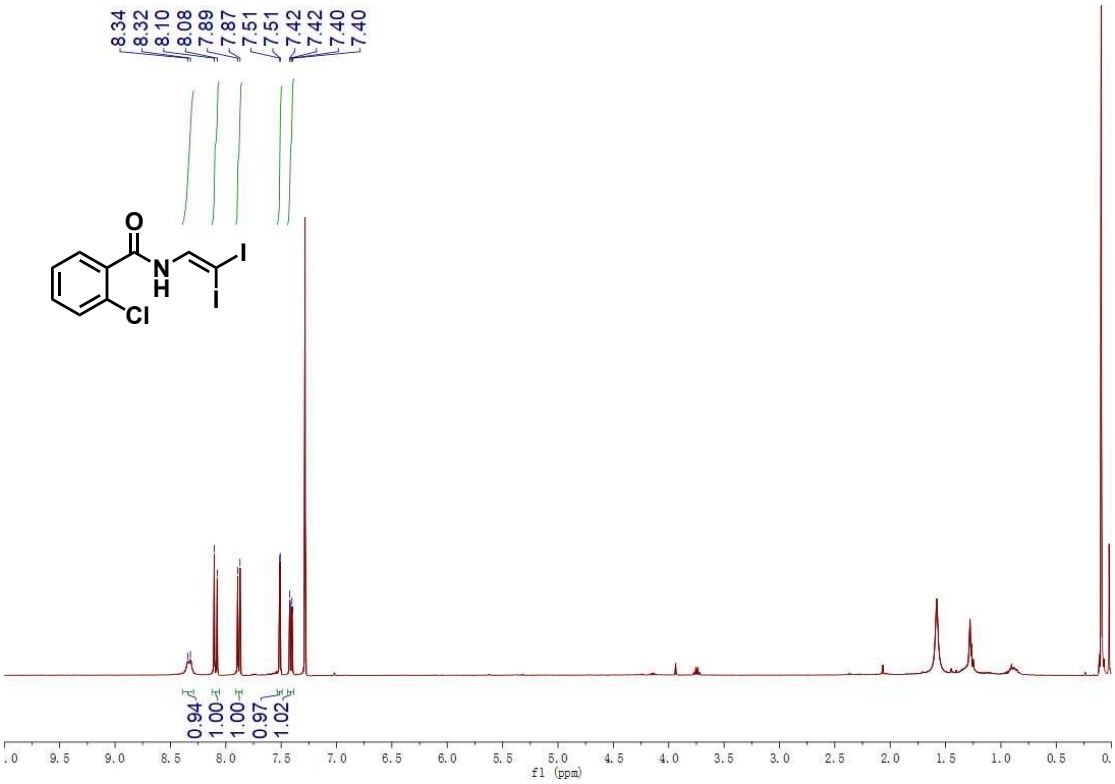
***N*-(2,2-diiodovinyl)-2-fluorobenzamide (2a'):** ^{1}H NMR (400 MHz, $CDCl_3$)



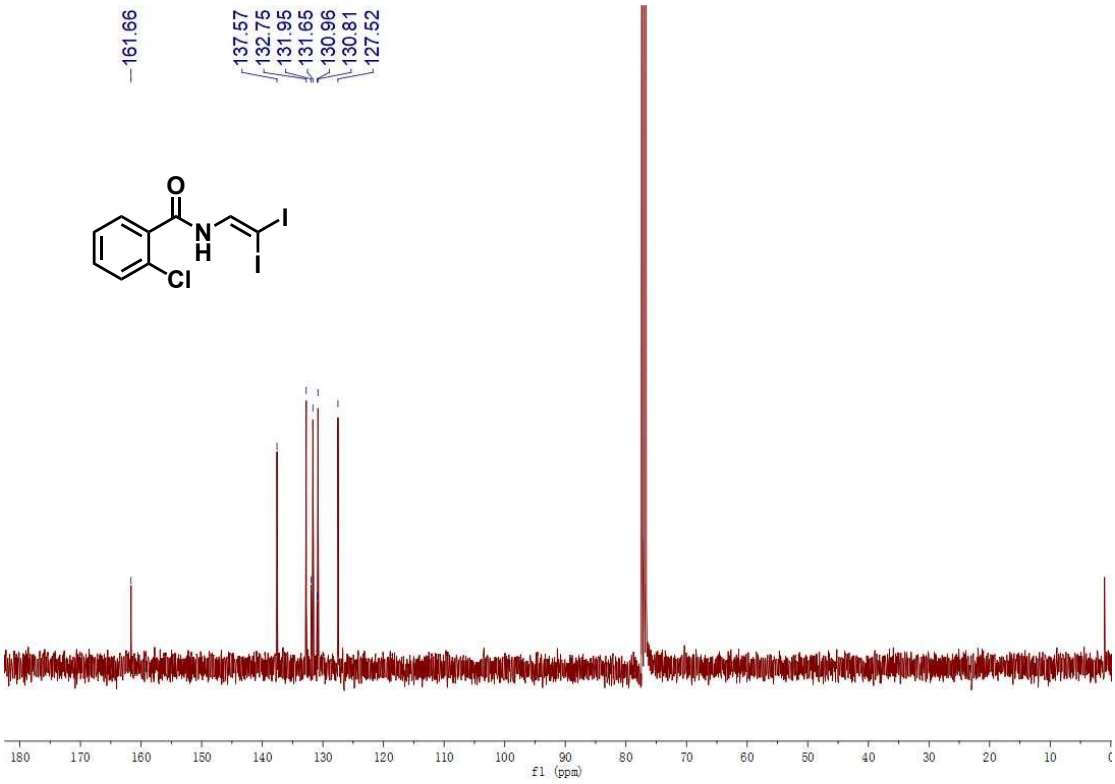
***N*-(2,2-diiodovinyl)-2-fluorobenzamide (2a'):** ^{13}C NMR (101 MHz, $CDCl_3$)



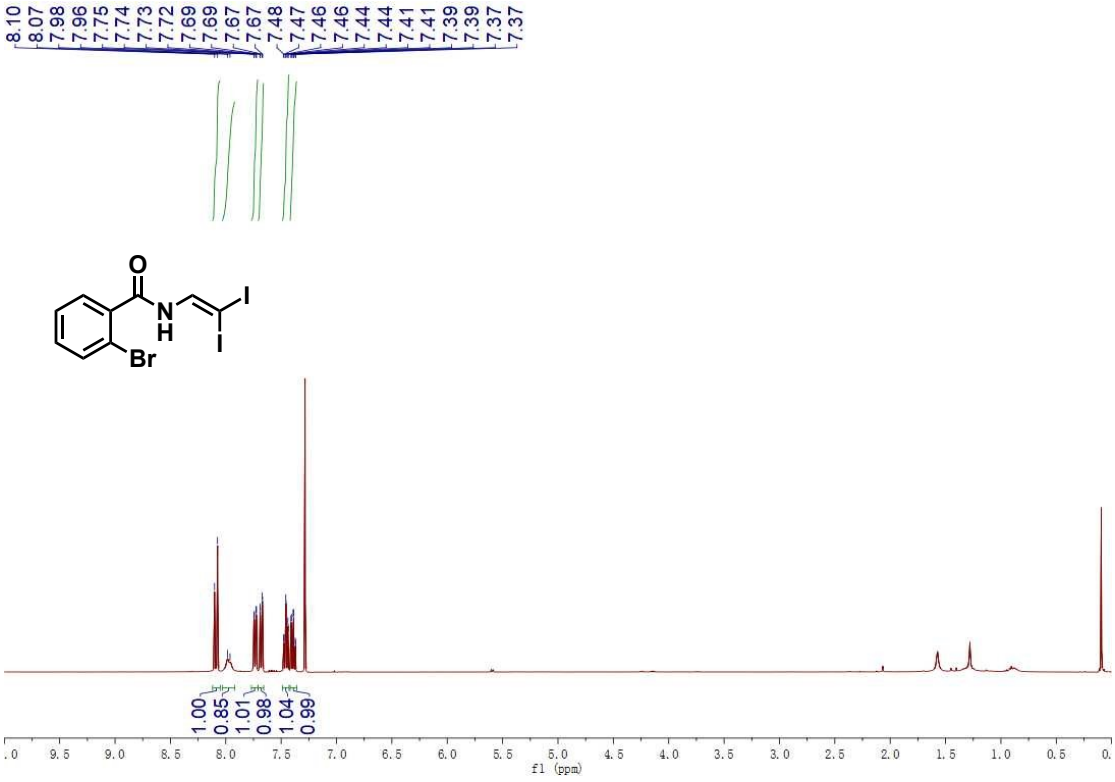
2-chloro-*N*-(2,2-diiodovinyl) benzamide (2b'): ^{1}H NMR (400 MHz, $CDCl_3$)



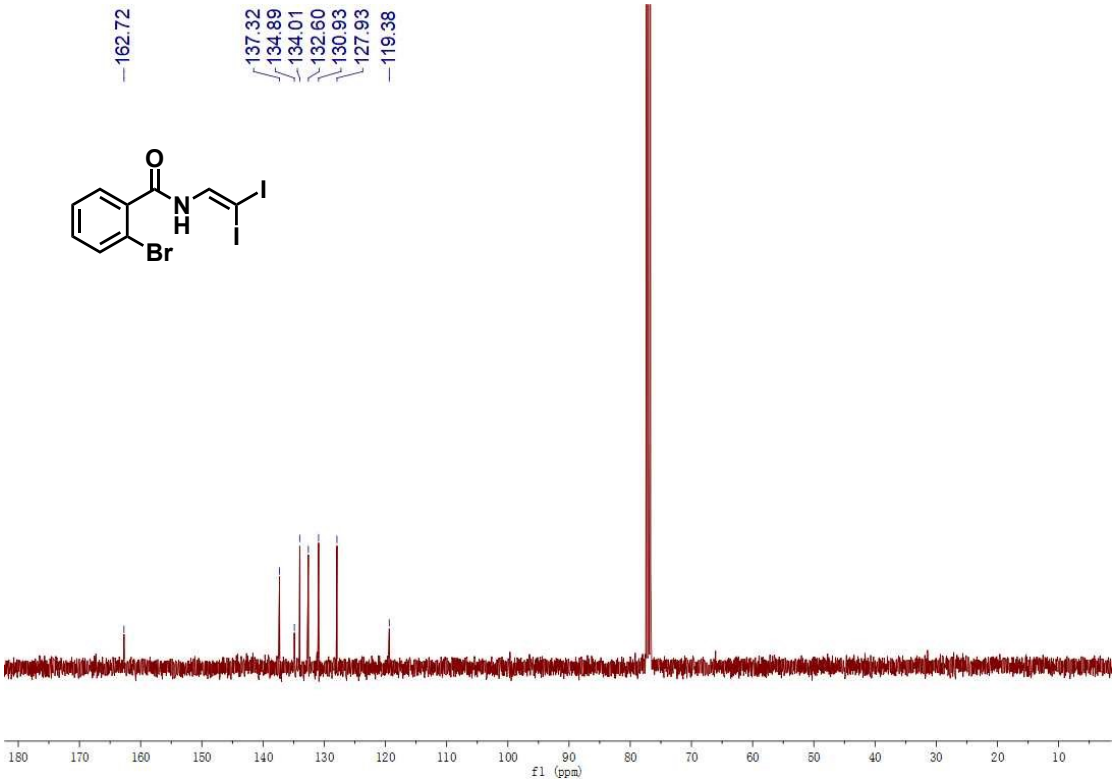
2-chloro-*N*-(2,2-diiodovinyl) benzamide (2b'): ^{13}C NMR (101 MHz, $CDCl_3$)



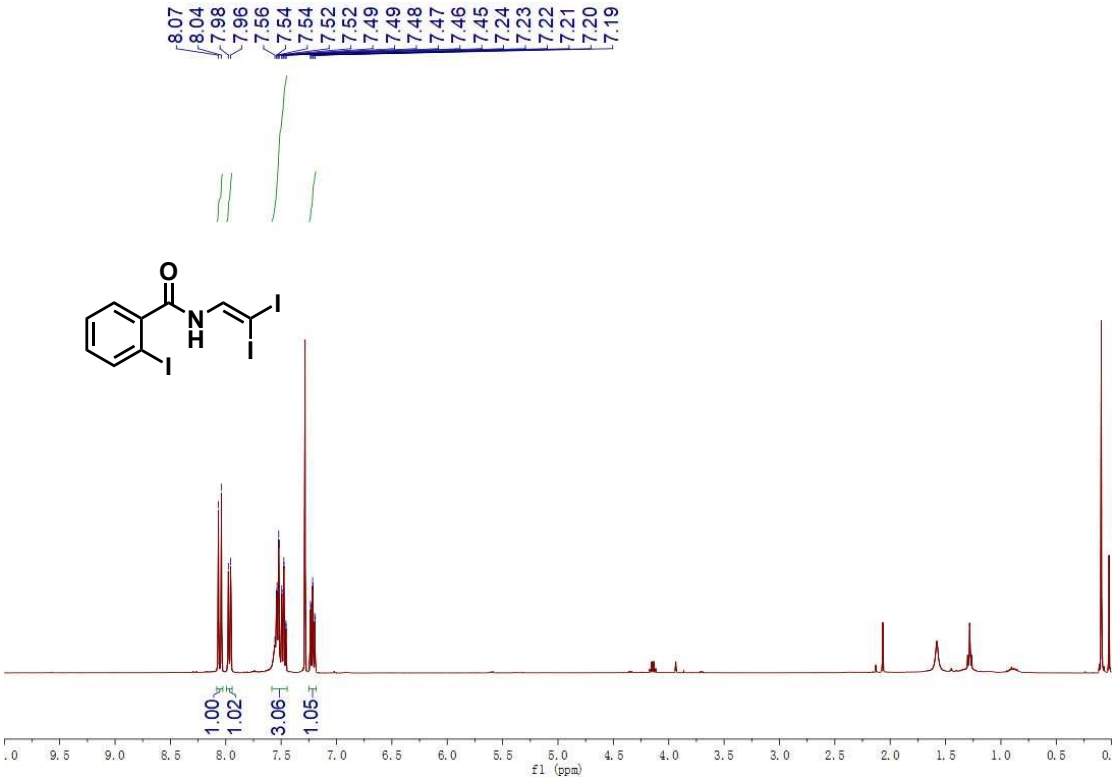
2-bromo-*N*-(2,2-diiodovinyl) benzamide (2c'): ^{1}H NMR (400 MHz, $CDCl_3$)



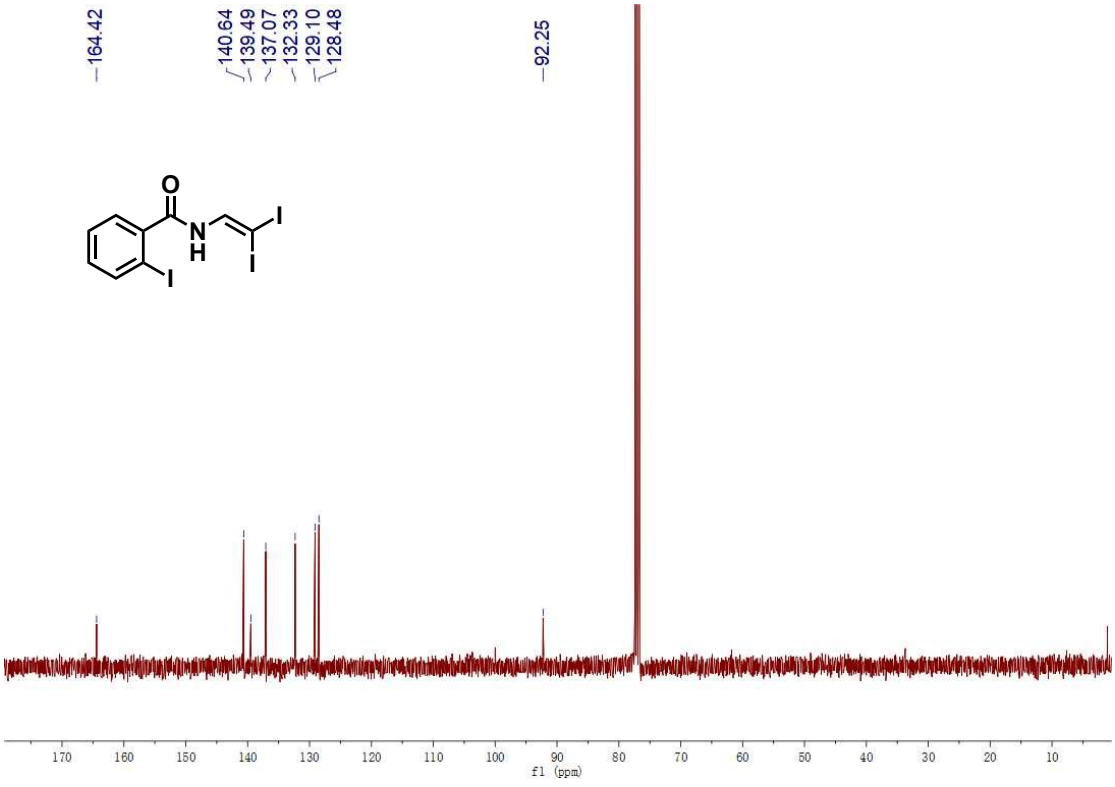
2-bromo-*N*-(2,2-diiodovinyl) benzamide (2c'): ^{13}C NMR (101 MHz, $CDCl_3$)



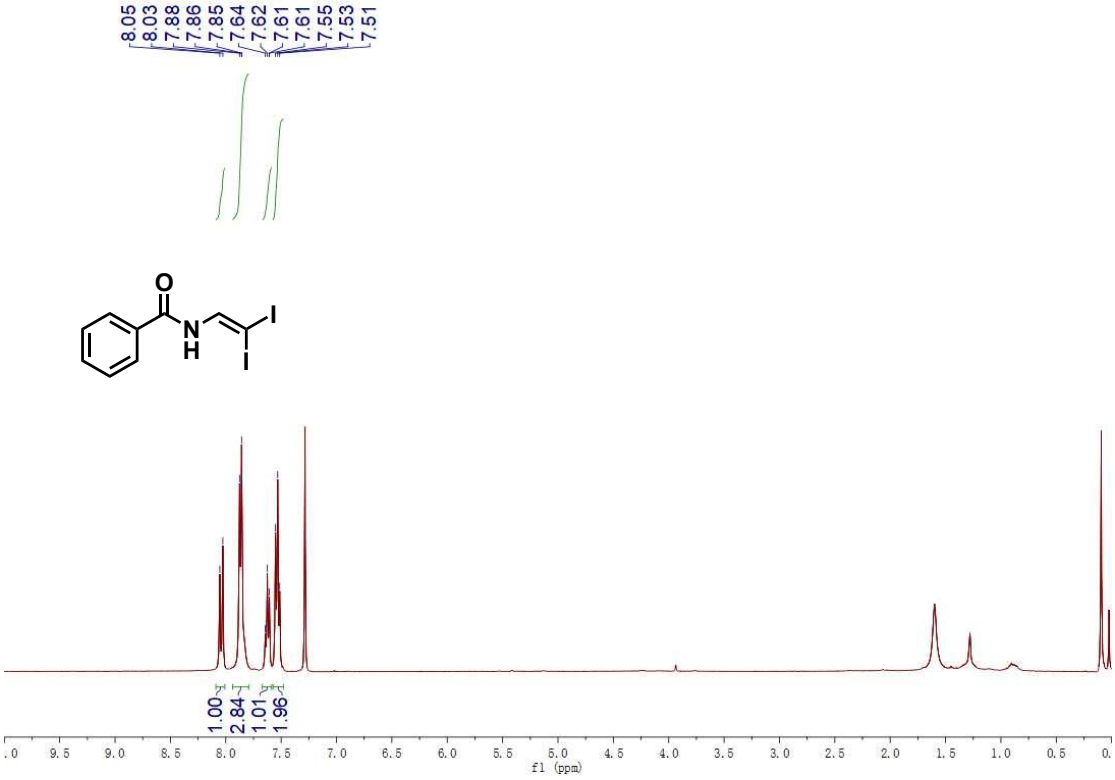
***N*-(2,2-diiodovinyl)-2-iodobenzamide (2d'):** ^{1}H NMR (400 MHz, $CDCl_3$)



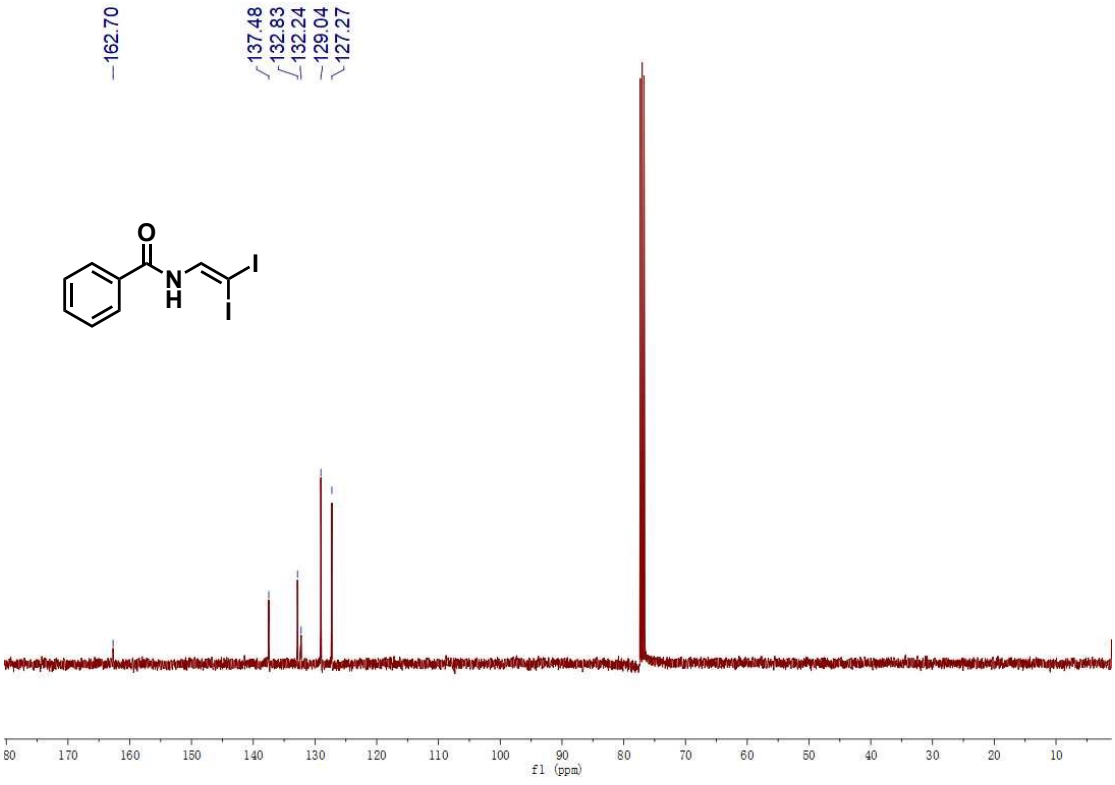
***N*-(2,2-diiodovinyl)-2-iodobenzamide (2d'):** ^{13}C NMR (101 MHz, $CDCl_3$)



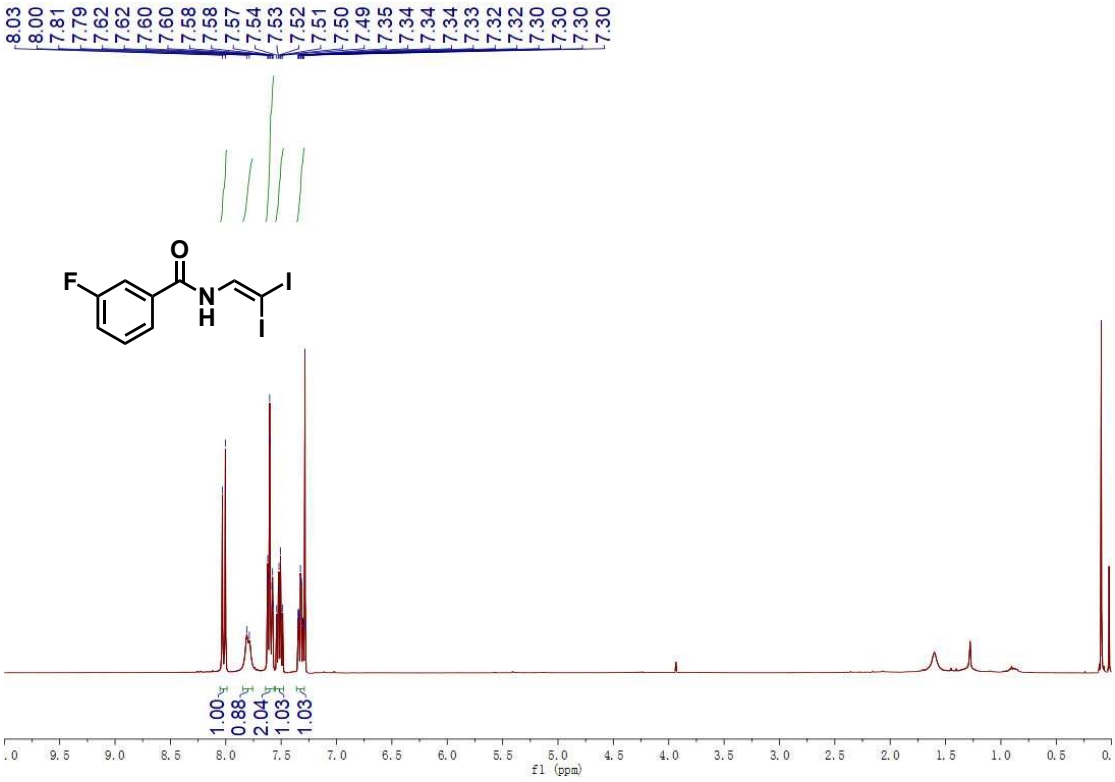
***N*-(2,2-diiodovinyl) benzamide (2g'):** ^{1}H NMR (400 MHz, $CDCl_3$)



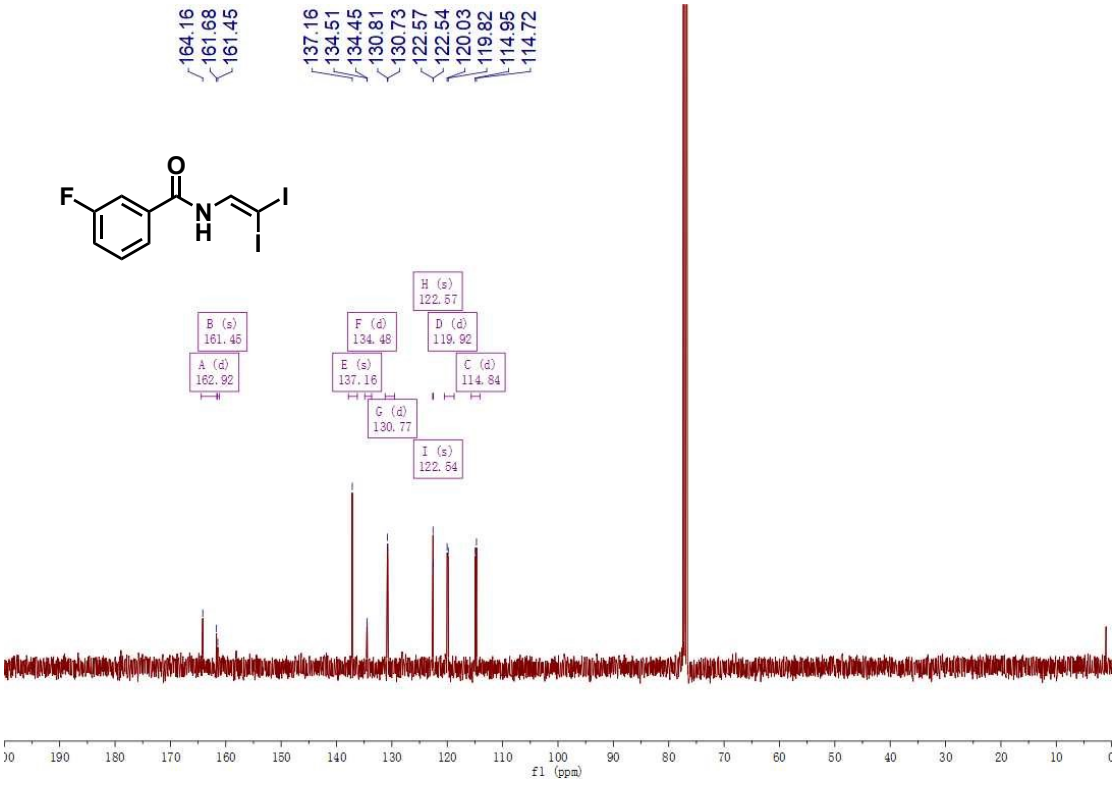
***N*-(2,2-diiodovinyl) benzamide (2g'):** ^{13}C NMR (101 MHz, $CDCl_3$)



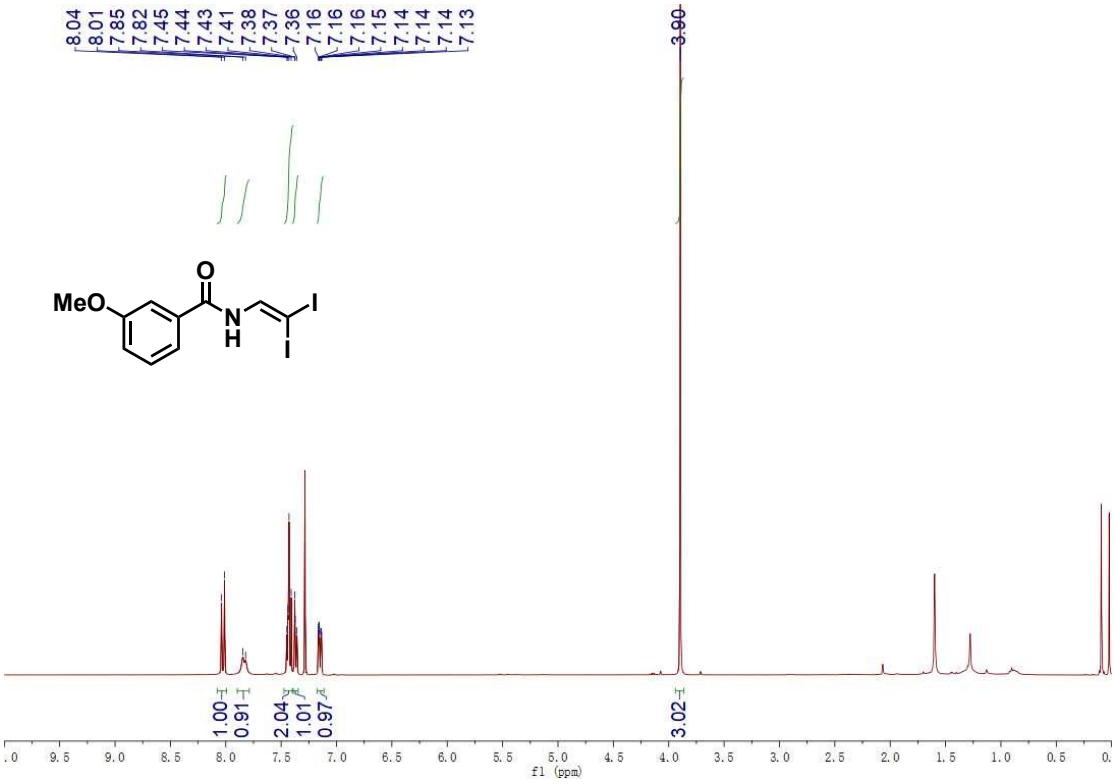
***N*-(2,2-diiodovinyl)-3-fluorobenzamide (2h'):** ^{1}H NMR (400 MHz, $CDCl_3$)



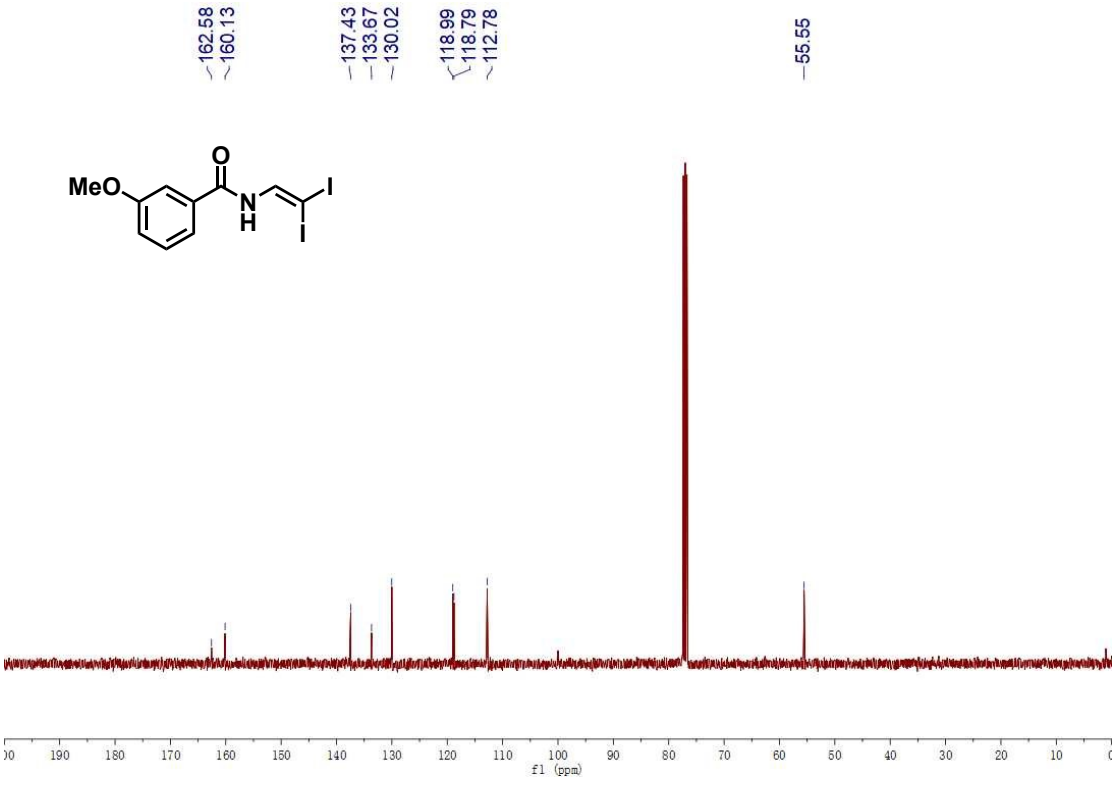
***N*-(2,2-diiodovinyl)-3-fluorobenzamide (2h'):** ^{13}C NMR (101 MHz, $CDCl_3$)



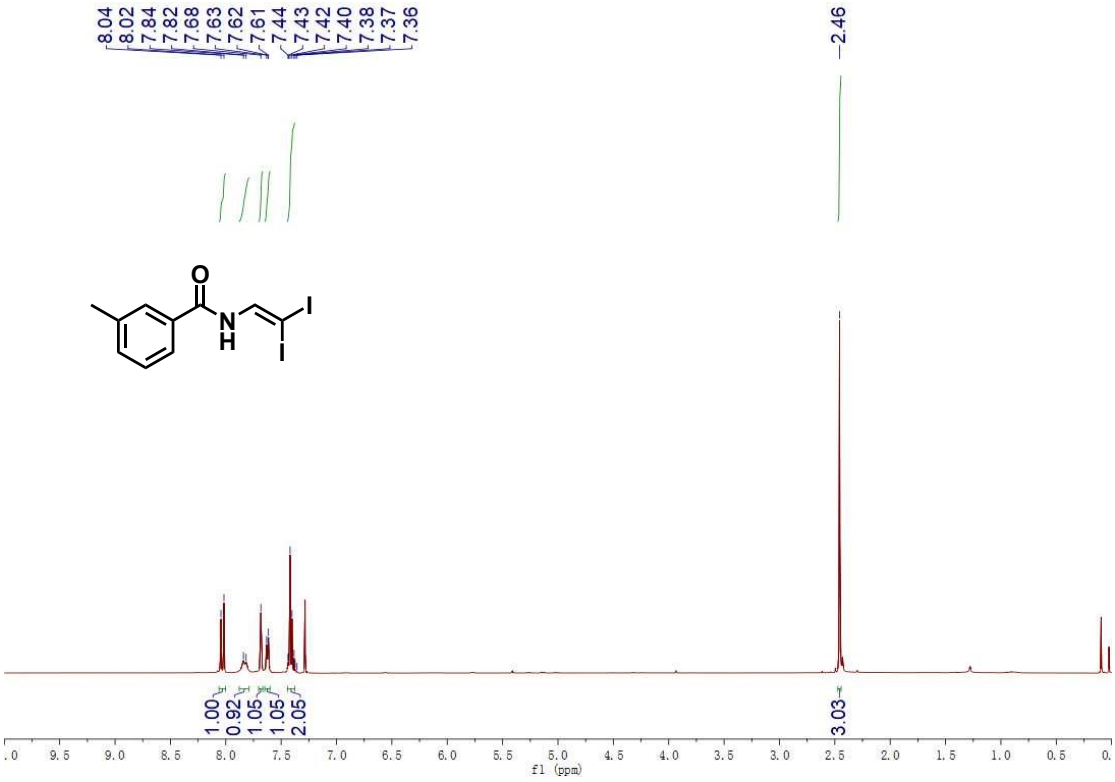
***N*-(2,2-diiodovinyl)-3-methoxybenzamide (2i'):** ^{1}H NMR (400 MHz, $CDCl_3$)



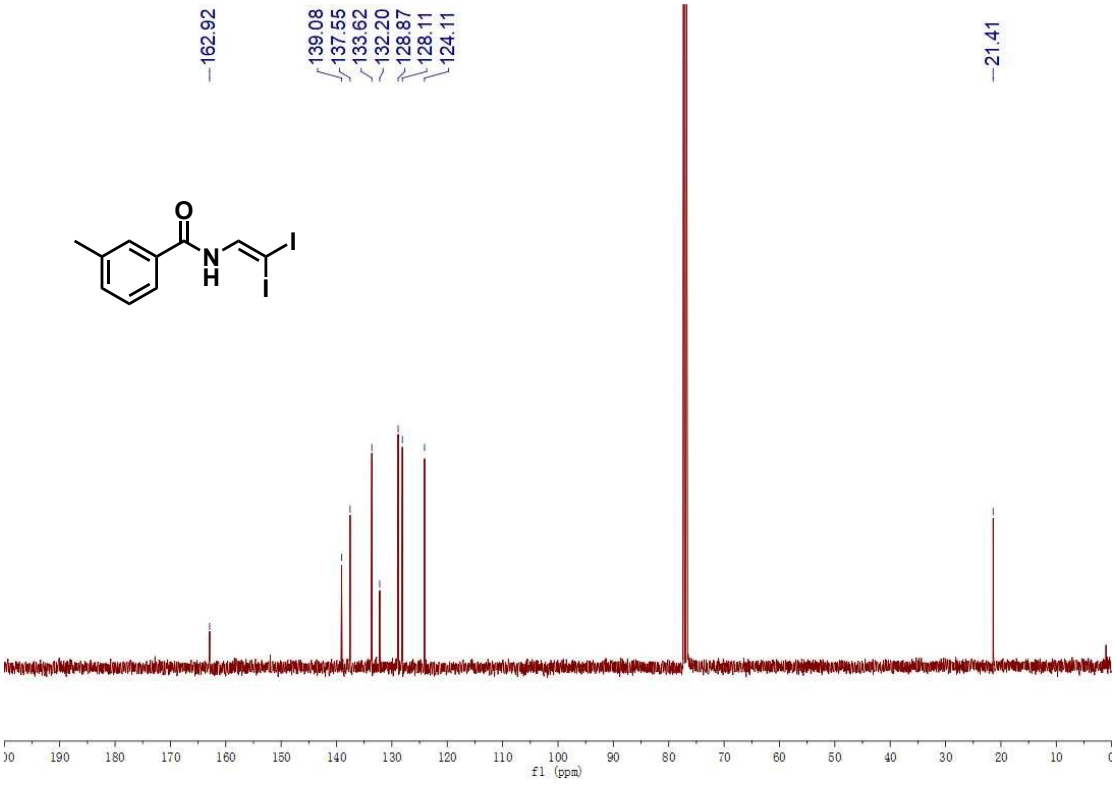
***N*-(2,2-diiodovinyl)-3-methoxybenzamide (2i'):** ^{13}C NMR (101 MHz, $CDCl_3$)



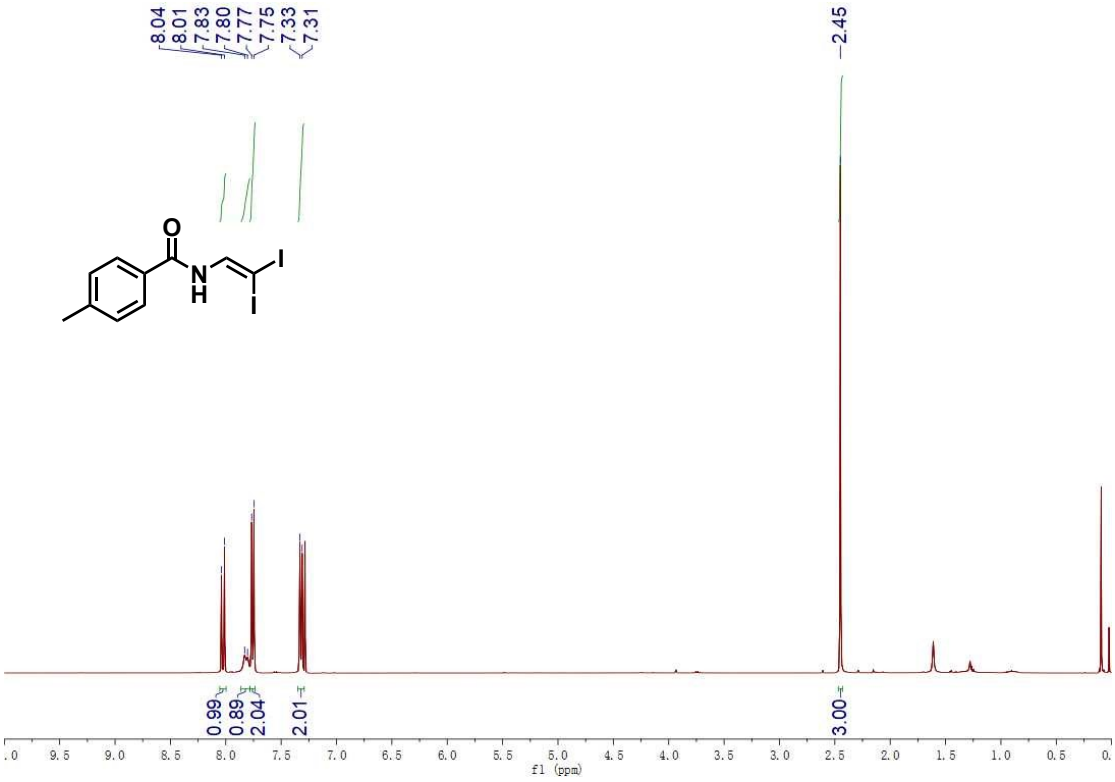
***N*-(2,2-diiodovinyl)-3-methylbenzamide (2j'):** ^{1}H NMR (400 MHz, $CDCl_3$)



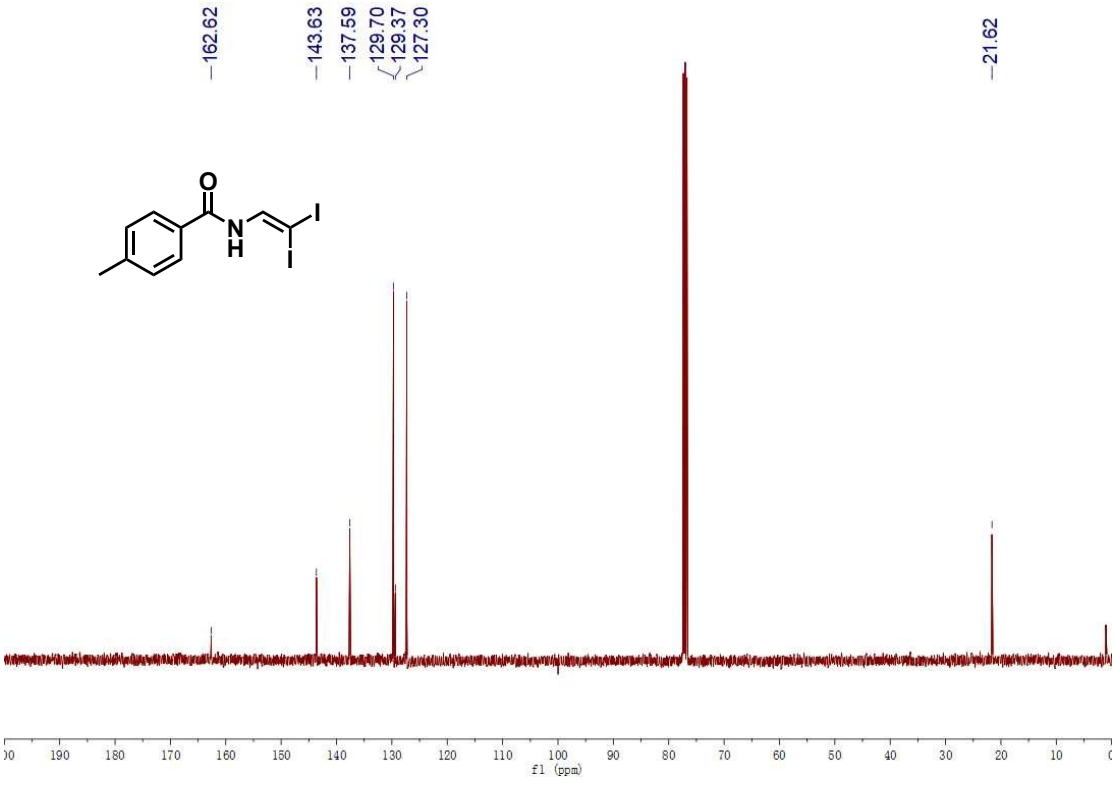
***N*-(2,2-diiodovinyl)-3-methylbenzamide (2j'):** ^{13}C NMR (101 MHz, $CDCl_3$)



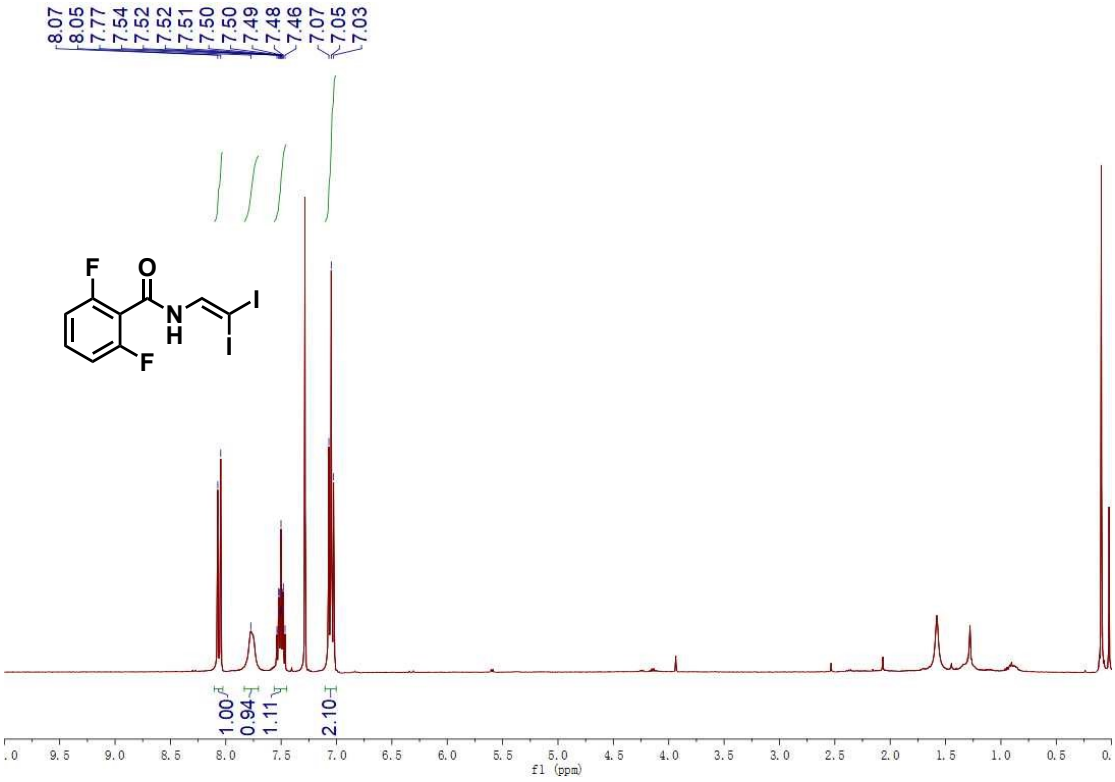
***N*-(2,2-diiodovinyl)-4-methylbenzamide (2k'):** ^{1}H NMR (400 MHz, $CDCl_3$)



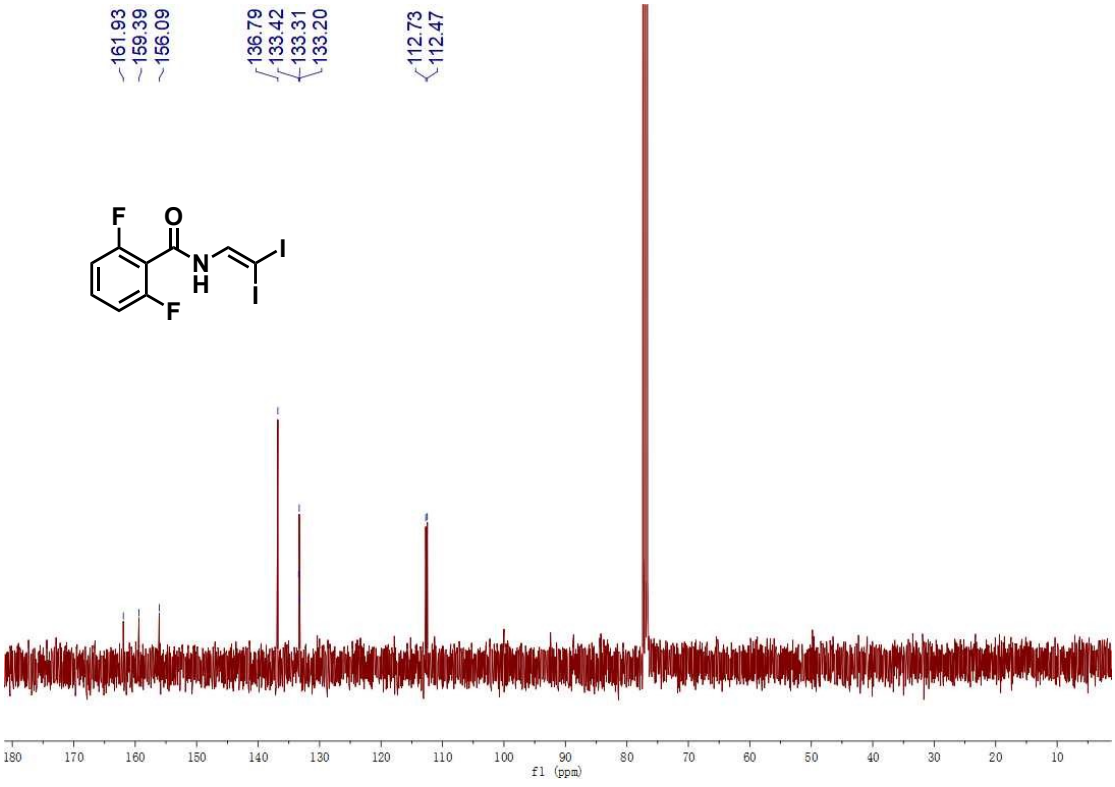
***N*-(2,2-diiodovinyl)-4-methylbenzamide (2k'):** ^{13}C NMR (101 MHz, $CDCl_3$)



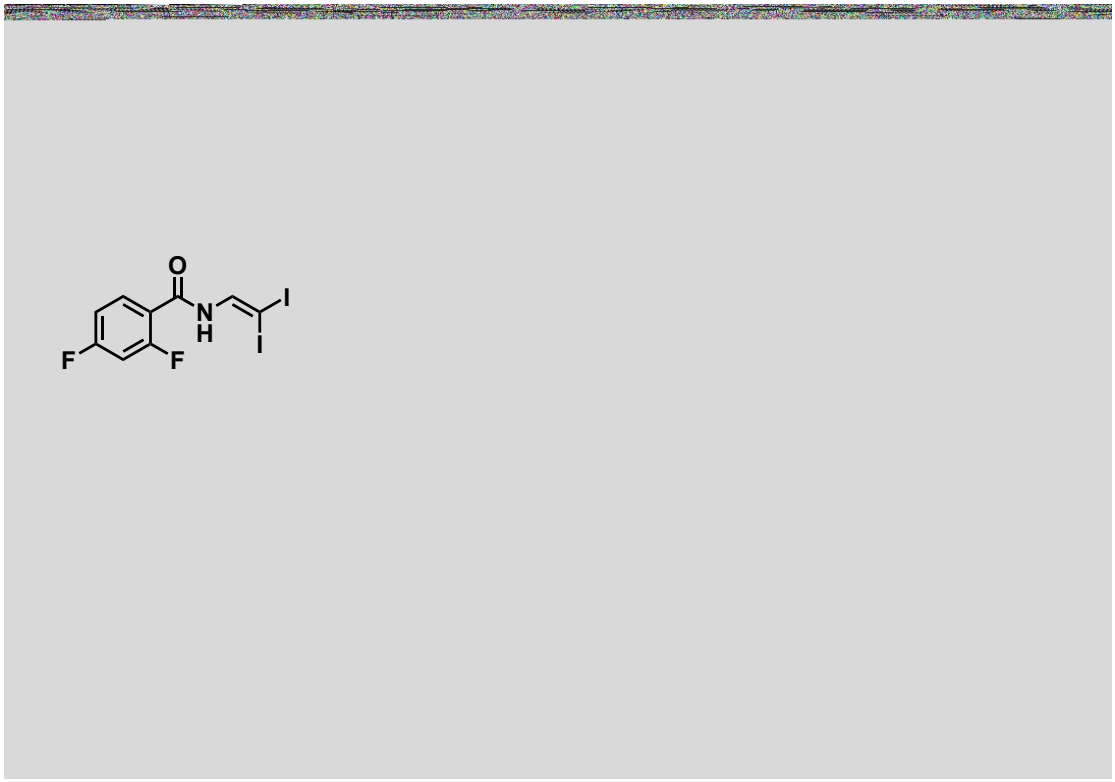
***N*-(2,2-diiodovinyl)-2,6-difluorobenzamide (2m'):** ^{1}H NMR (400 MHz, $CDCl_3$)



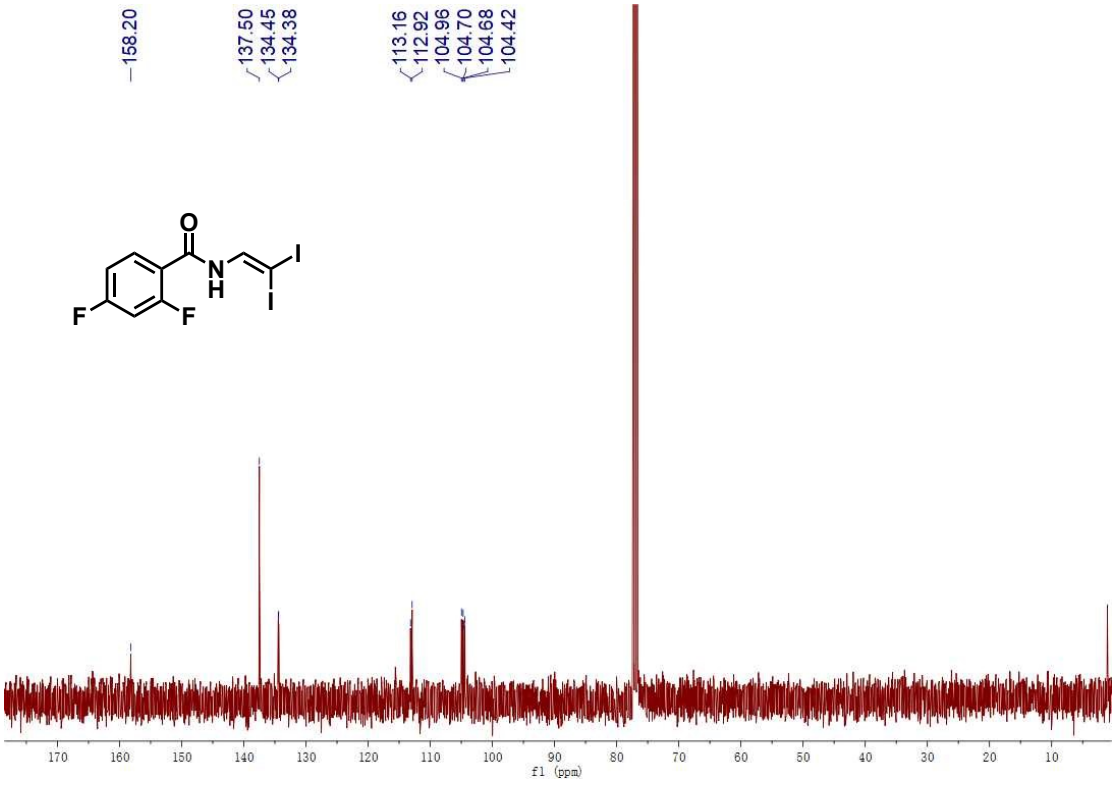
***N*-(2,2-diiodovinyl)-2,6-difluorobenzamide (2m'):** ^{13}C NMR (101 MHz, $CDCl_3$)



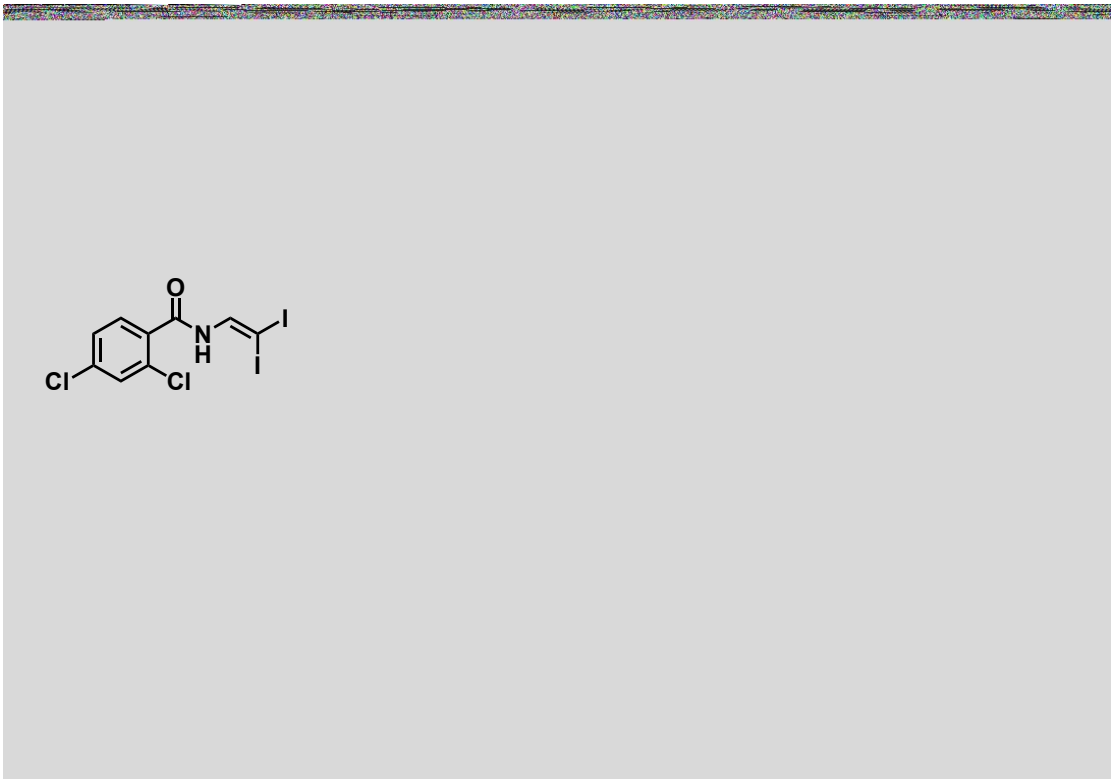
***N*-(2,2-diiodovinyl)-2,4-difluorobenzamide (2n'):** ^{1}H NMR (400 MHz, $CDCl_3$)



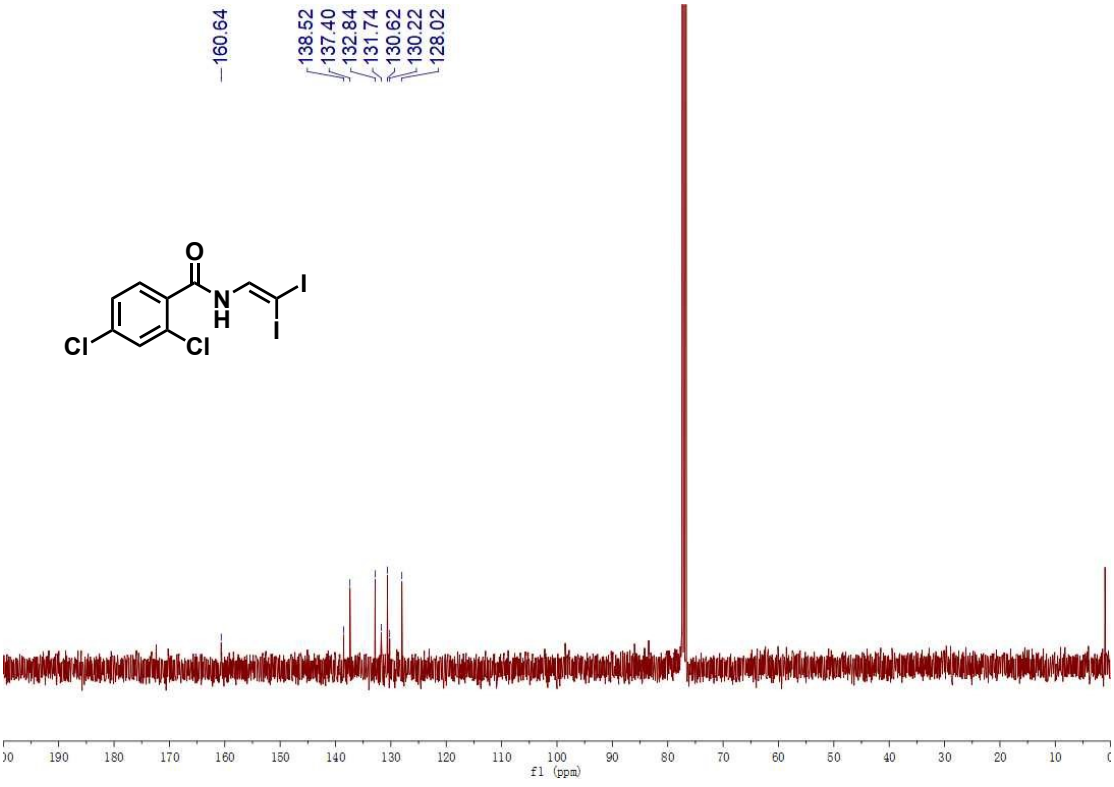
***N*-(2,2-diiodovinyl)-2,4-difluorobenzamide (2n'):** ^{13}C NMR (101 MHz, $CDCl_3$)



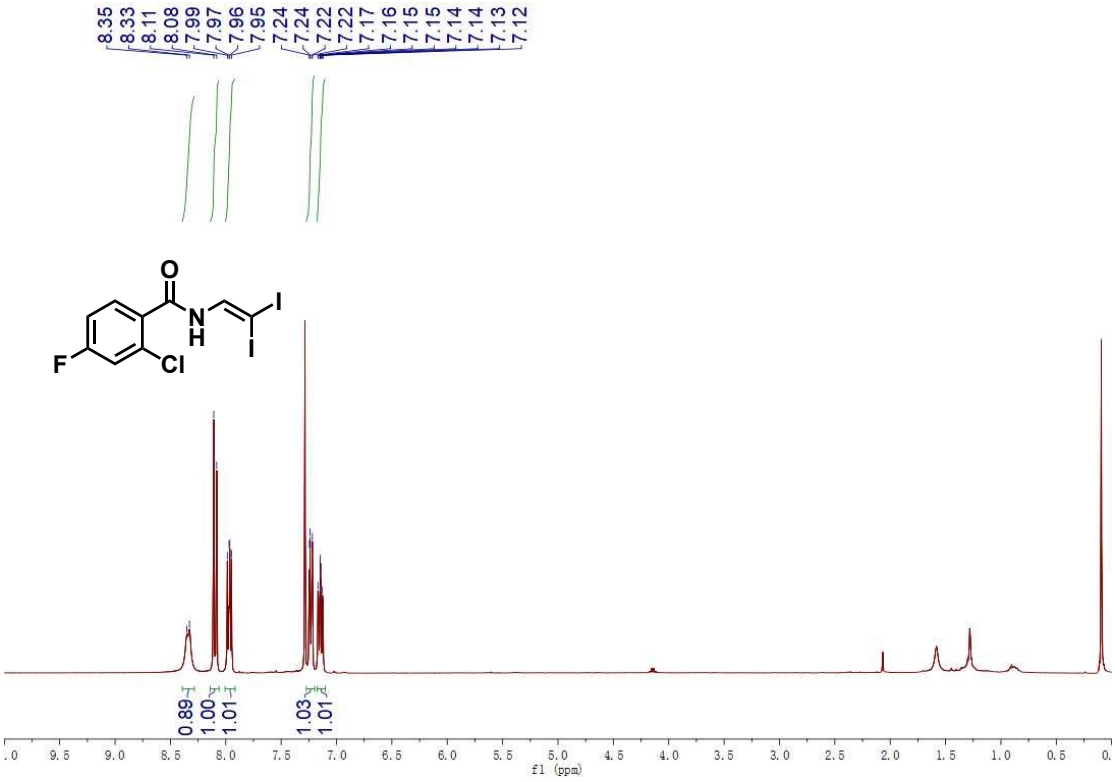
2,4-dichloro-*N*-(2,2-diiodovinyl) benzamide (2o'): ^{1}H NMR (400 MHz, $CDCl_3$)



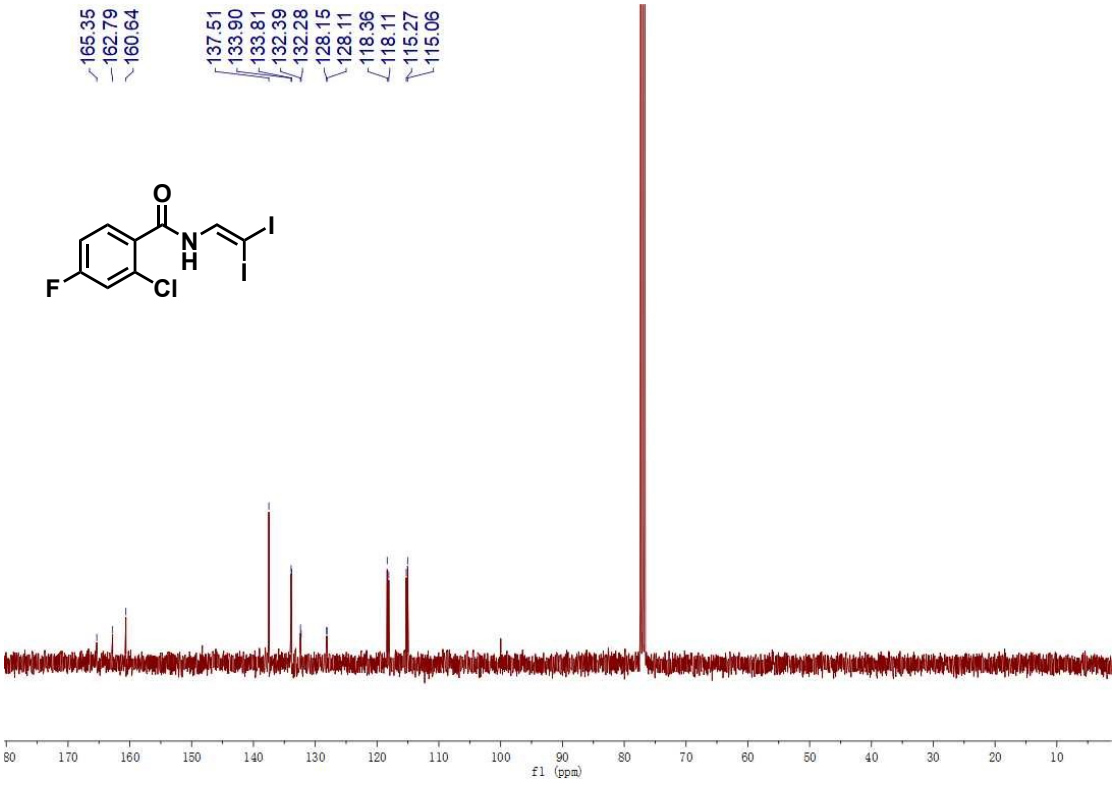
2,4-dichloro-*N*-(2,2-diiodovinyl) benzamide (2o'): ^{13}C NMR (101 MHz, $CDCl_3$)



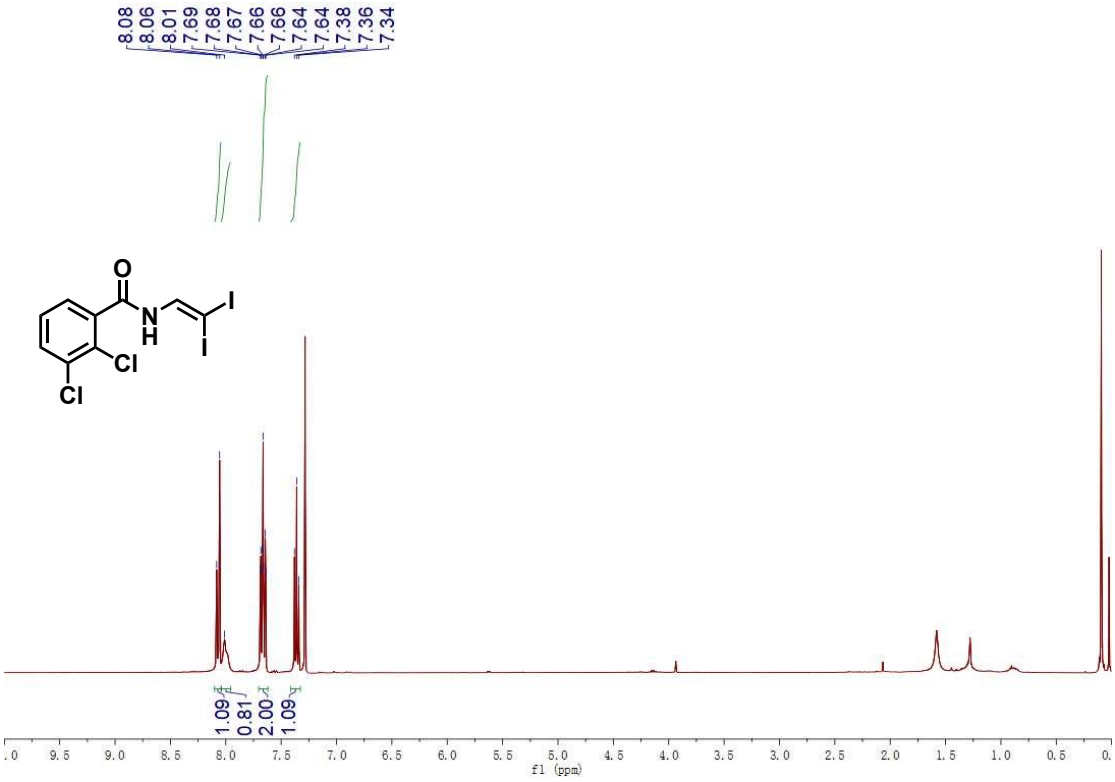
2-chloro-*N*-(2,2-diiodovinyl)-4-fluorobenzamide (2p'): ^{1}H NMR (400 MHz, $CDCl_3$)



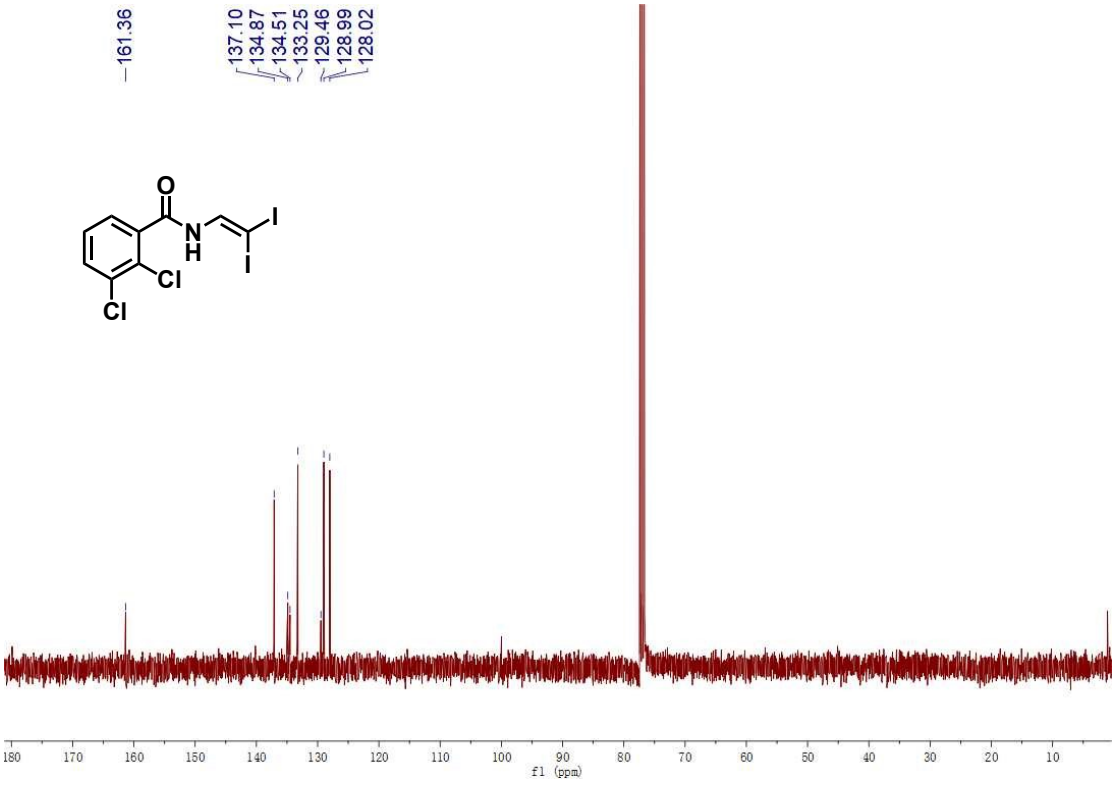
2-chloro-*N*-(2,2-diiodovinyl)-4-fluorobenzamide (2p'): ^{13}C NMR (101 MHz, $CDCl_3$)



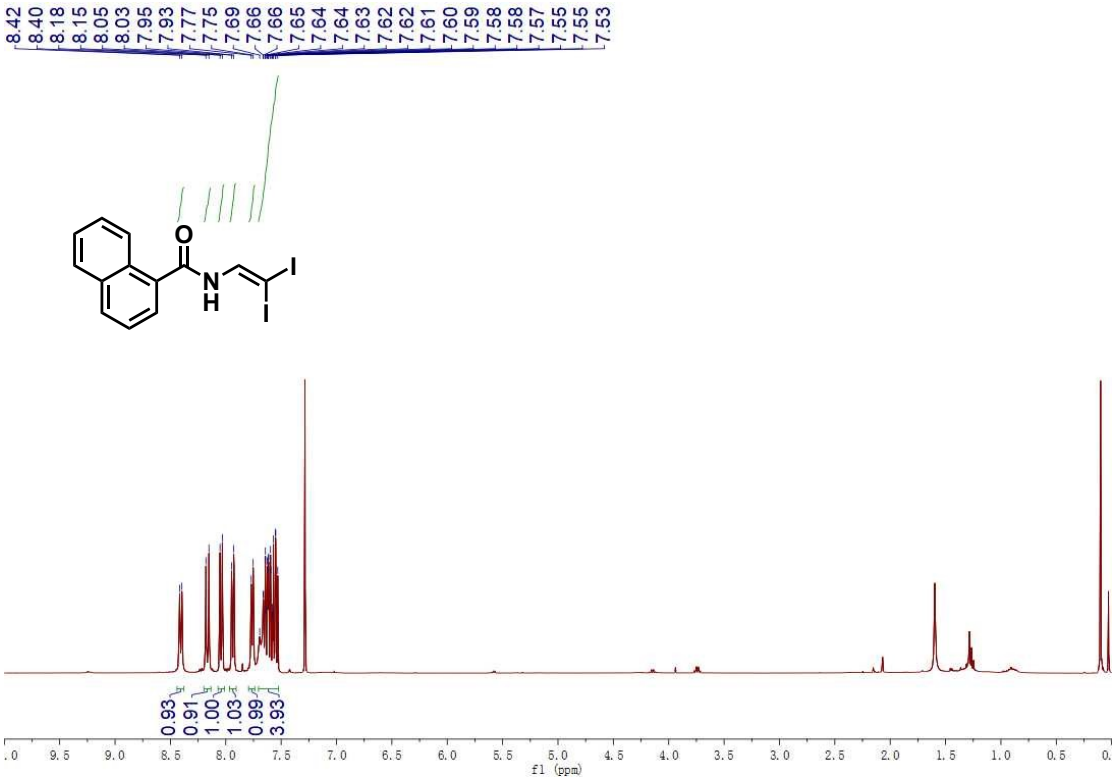
2,3-dichloro-*N*-(2,2-diiodovinyl) benzamide (2q'): ^{1}H NMR (400 MHz, $CDCl_3$)



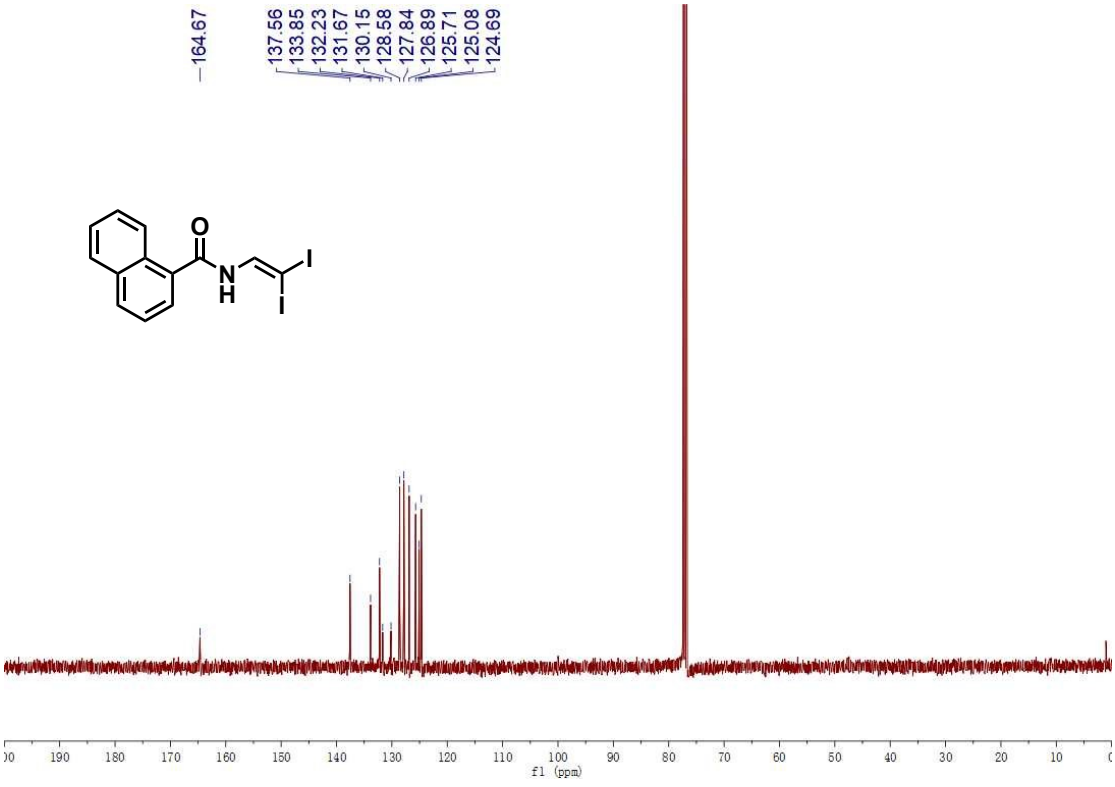
2,3-dichloro-*N*-(2,2-diiodovinyl) benzamide (2q'): ^{13}C NMR (101 MHz, $CDCl_3$)



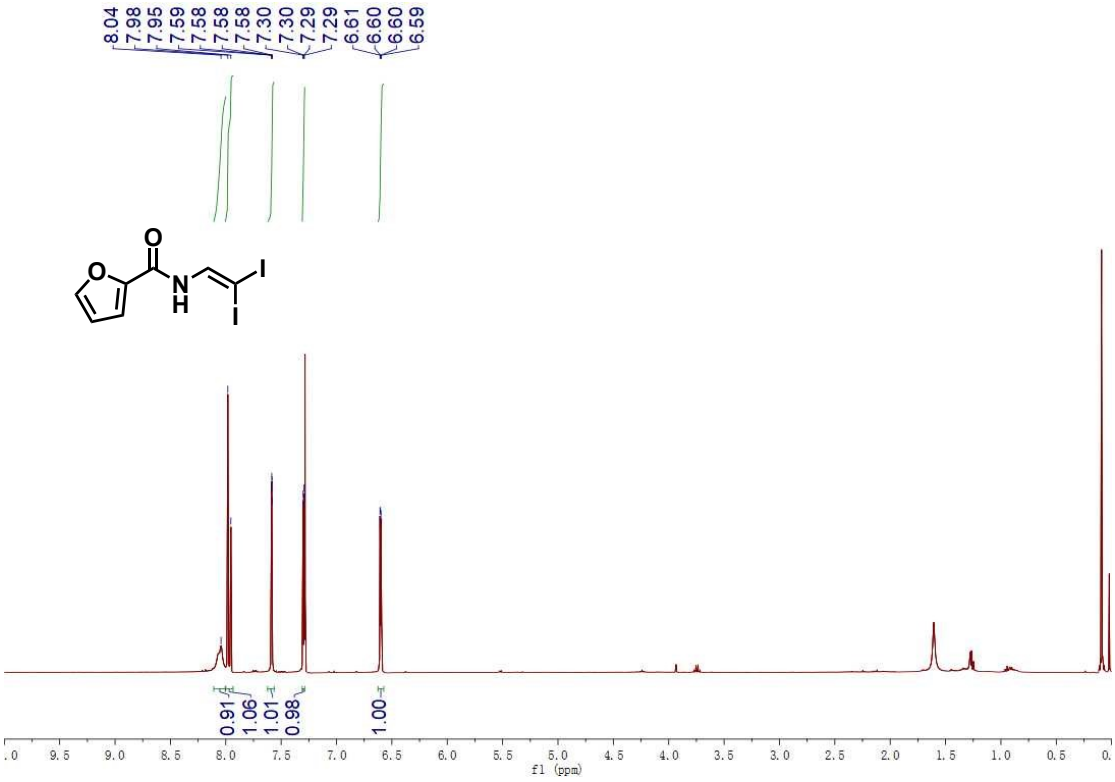
***N*-(2,2-diiodovinyl)-1-naphthamide (2t'):** ^{1}H NMR (400 MHz, $CDCl_3$)



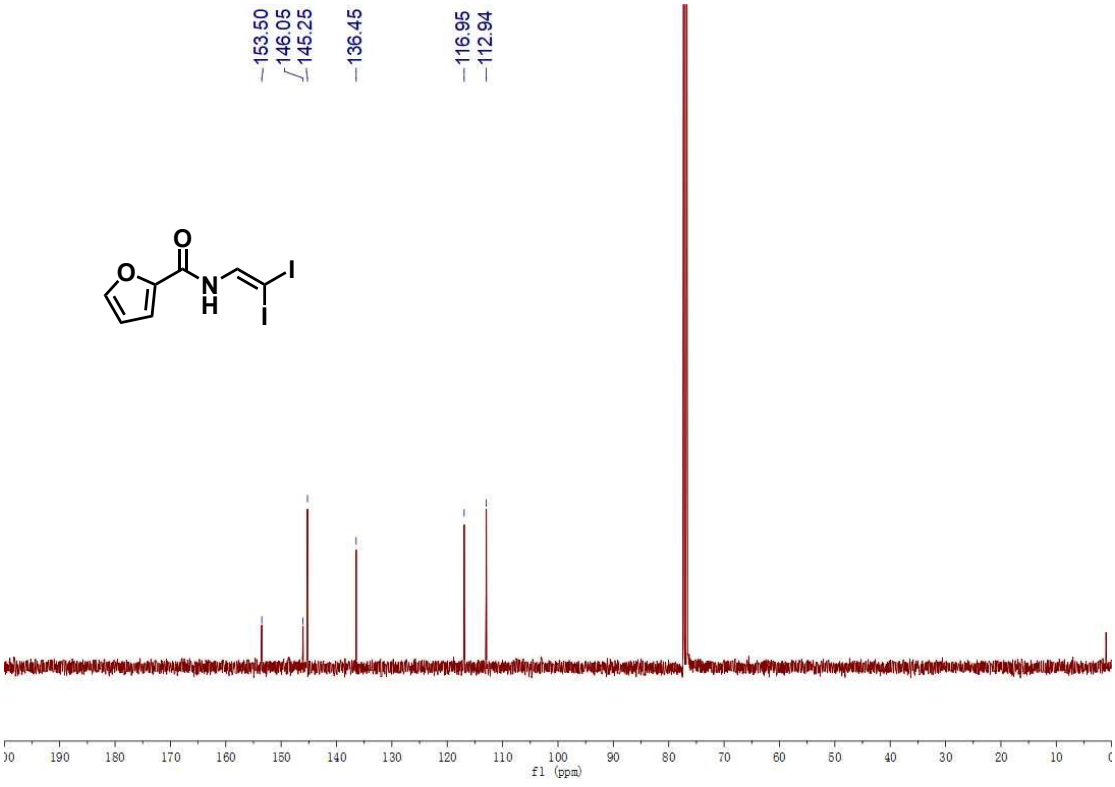
***N*-(2,2-diiodovinyl)-1-naphthamide (2t'):** ^{13}C NMR (101 MHz, $CDCl_3$)



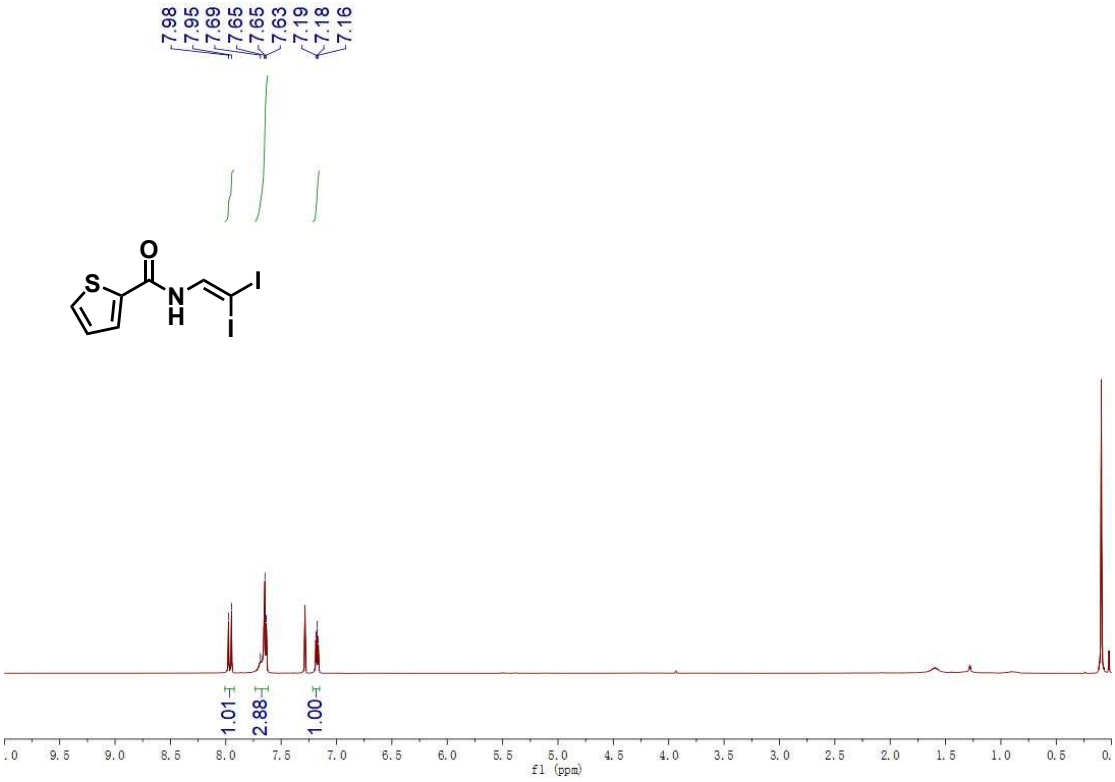
***N*-(2,2-diiodovinyl) furan-2-carboxamide (2v'):** ^{1}H NMR (400 MHz, $CDCl_3$)



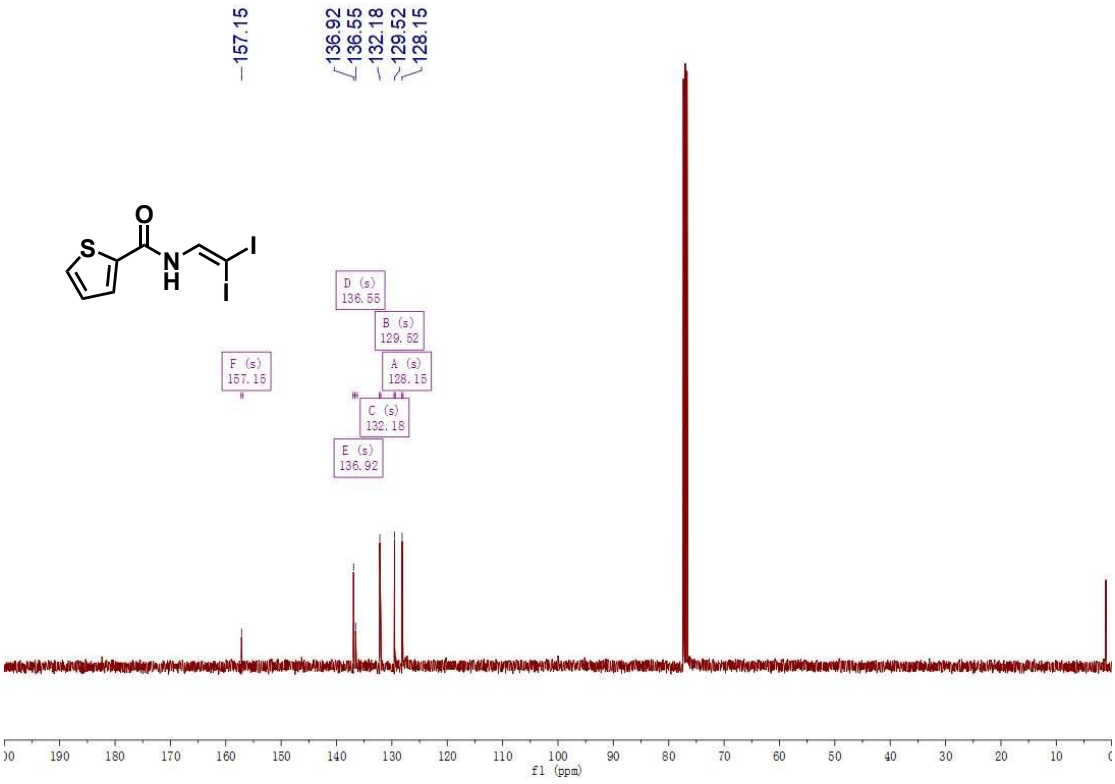
***N*-(2,2-diiodovinyl) furan-2-carboxamide (2v'):** ^{13}C NMR (101 MHz, $CDCl_3$)



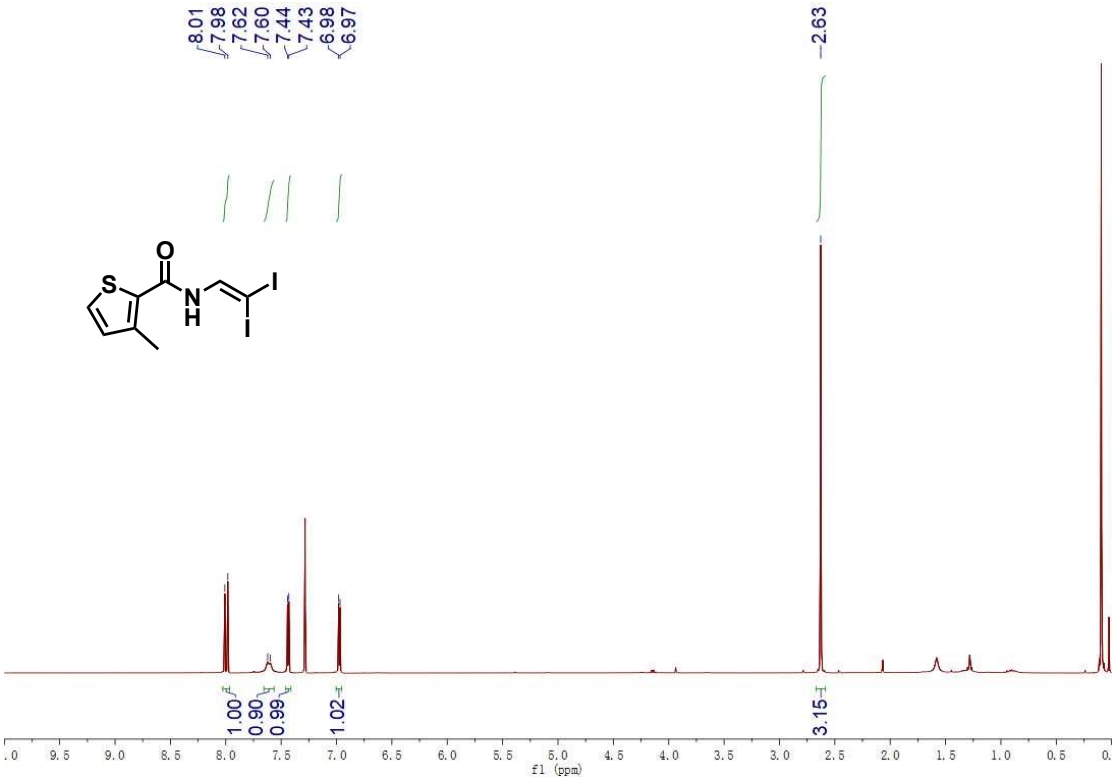
***N*-(2,2-diiodovinyl) thiophene-2-carboxamide (2w'):** ^{1}H NMR (400 MHz, $CDCl_3$)



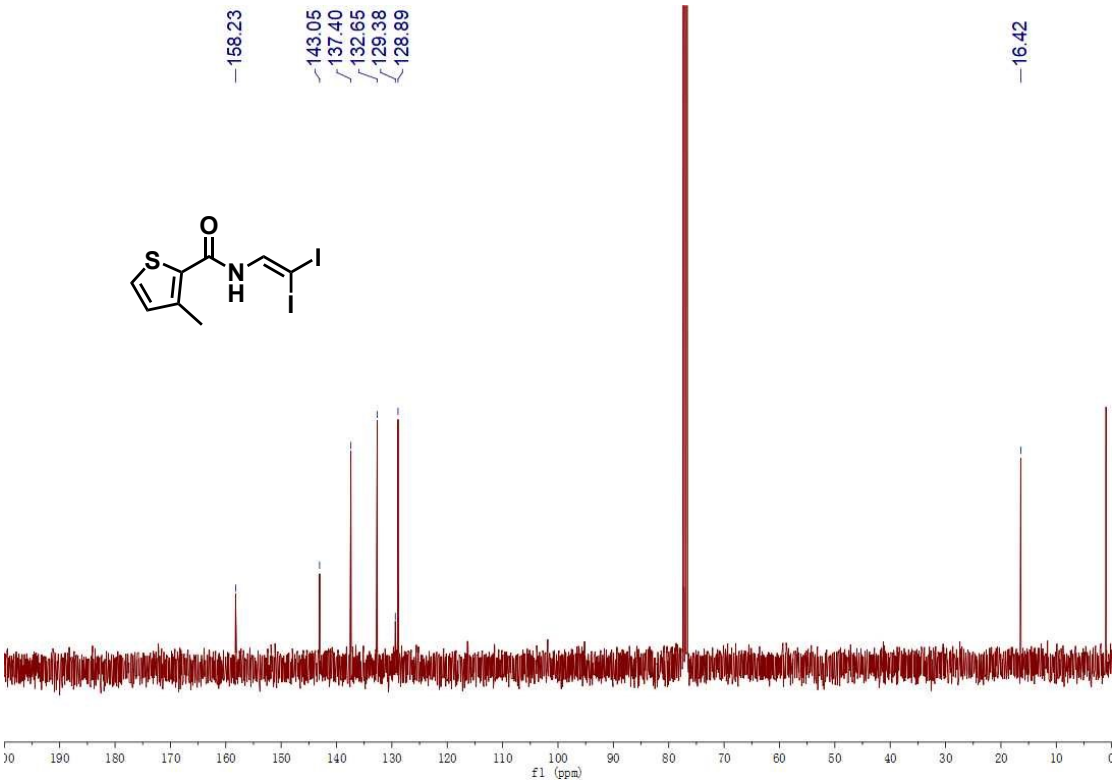
***N*-(2,2-diiodovinyl) thiophene-2-carboxamide (2w'):** ^{13}C NMR (101 MHz, $CDCl_3$)



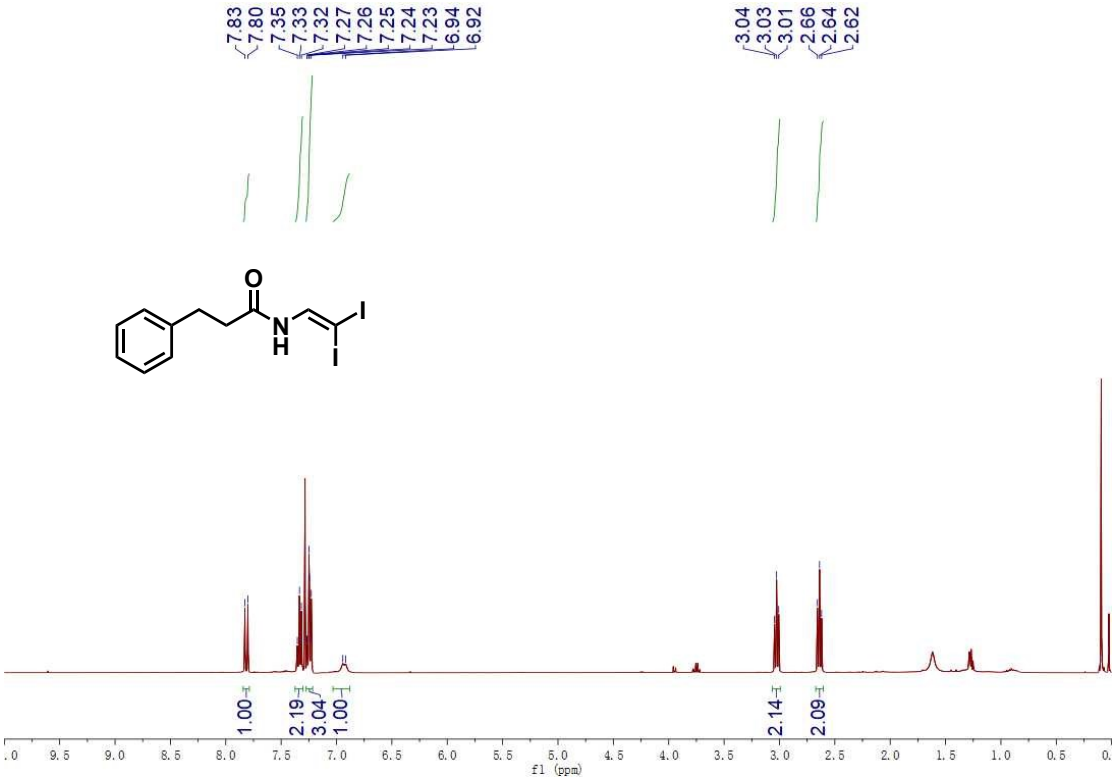
***N*-(2,2-diiodovinyl)-3-methylthiophene-2-carboxamide (2x'):** ^{1}H NMR (400 MHz, $CDCl_3$)



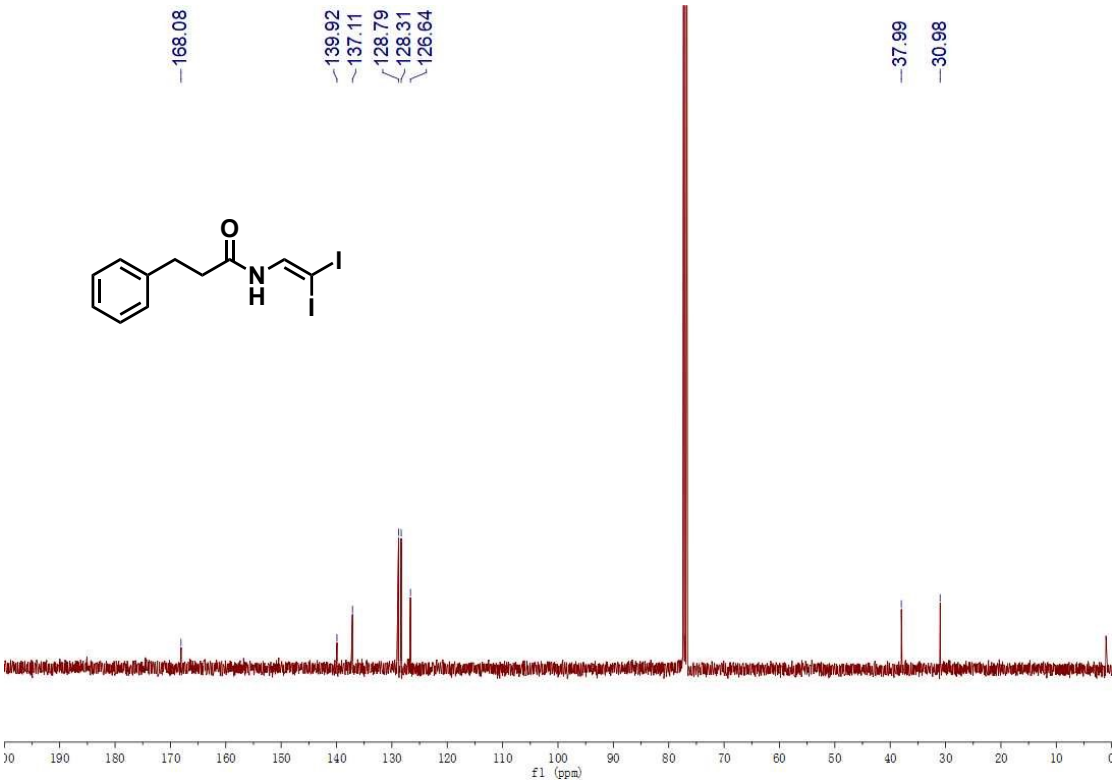
***N*-(2,2-diiodovinyl)-3-methylthiophene-2-carboxamide (2x'):** ^{13}C NMR (101 MHz, $CDCl_3$)



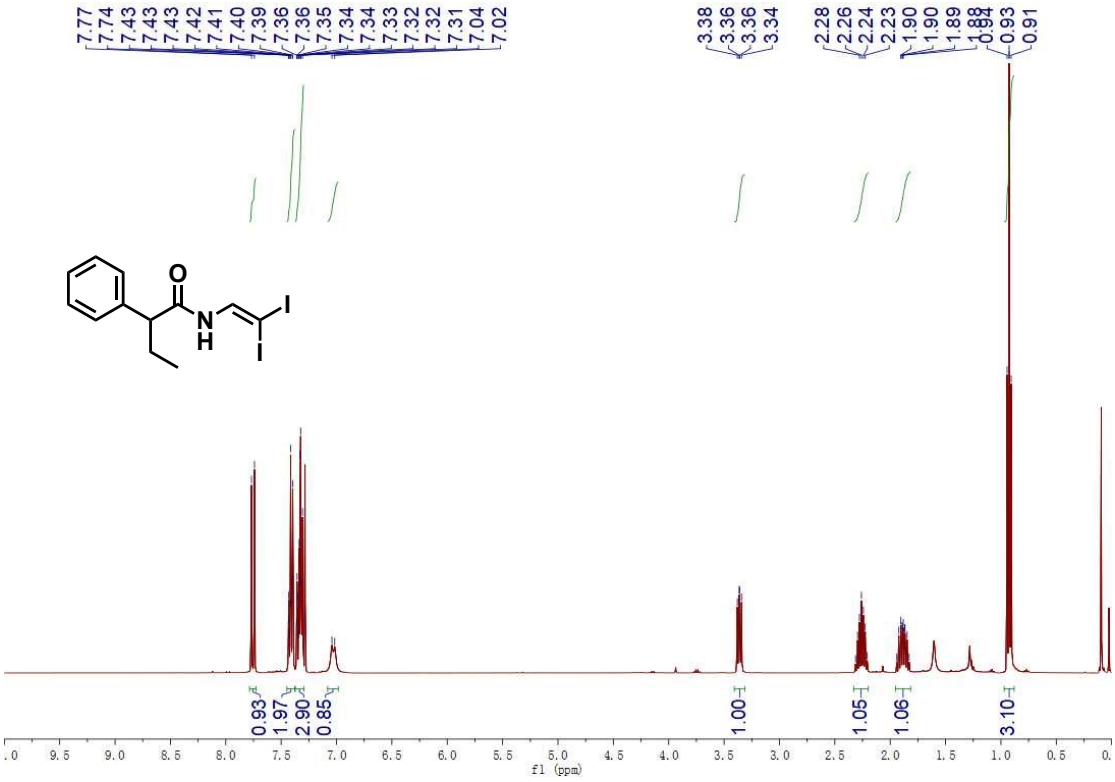
***N*-(2,2-diiodovinyl)-3-phenylpropanamide (2y'):** ^{1}H NMR (400 MHz, $CDCl_3$)



***N*-(2,2-diiodovinyl)-3-phenylpropanamide (2y'):** ^{13}C NMR (101 MHz, $CDCl_3$)



***N*-(2,2-diiodovinyl)-2-phenylbutanamide (2aa'):** ^{1}H NMR (400 MHz, $CDCl_3$)



***N*-(2,2-diiodovinyl)-2-phenylbutanamide (2aa'):** ^{13}C NMR (101 MHz, $CDCl_3$)