Supplementary material

Gold(I)-Catalyzed Tandem Synthesis of Polycyclic Dihydroquinazolinones

Jingyang Sun, Yoona Song and Jae-Sang Ryu *

College of Pharmacy & Graduate School of Pharmaceutical Sciences, Ewha Womans University, 52 Ewhayeodae-gil, Seodaemun-Gu, Seoul 03760, Korea; sunjingyangs@163.com (J.S.); syn3829@naver.com (Y.S.)
* Correspondence: ryuj@ewha.ac.kr; Tel.: +82-2-3277-3008

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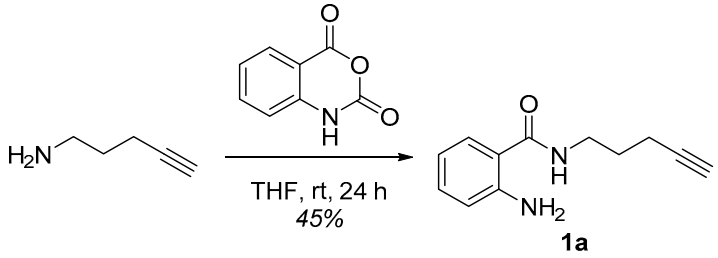
1. General Information

All reactions were performed in oven-dried glassware fitted with glass stoppers under positive pressure of Ar with magnetic stirring, unless otherwise noted. Air- and moisture-sensitive liquids and solutions were transferred via syringe or stainless-steel cannula. TLC was performed on 0.25 mm E. Merck silica gel 60 F_{254} plates and visualized under UV light (254 nm) or by staining with cerium ammonium molybdenate (CAM), potassium permanganate ($KMnO_4$), ninhydrin or *p*-anisaldehyde. Flash chromatography was performed on E. Merck 230–400 mesh silica gel 60. Medium-pressure liquid chromatography (MPLC) was performed on a prepacked column (silica gel, 10 μm) with a UV detector. Reagents were purchased from commercial suppliers, and used without further purification unless otherwise noted. Solvents were distilled from proper drying agents (CaH_2 or Na wire) under Ar atmosphere at 760 mmHg. All moisture- and/or oxygen-sensitive solids were handled and stored in a glovebox under N_2. NMR spectra were recorded on Agilent Unity 400 instruments or Bruker Avance Neo 400 MHz spectrometer system equipped at Drug Development Research Core Center at 24 °C. Chemical shifts are expressed in ppm relative to TMS (^{1}H, 0 ppm), $CDCl_3$ (^{1}H, 7.26 ppm; ^{13}C, 77.2 ppm), DMSO-d_6 (^{1}H, 2.50 ppm; ^{13}C, 39.5 ppm), CD_3CN (^{1}H, 1.94 ppm; ^{13}C, 1.3, 118.3 ppm), CD_3OD (^{1}H, 3.31 ppm; ^{13}C, 49.1 ppm); coupling constants are expressed in Hz. High resolution mass spectra (HRMS) were obtained by electrospray ionization (ESI, TOF), electron ionization (EI, magnetic sector), chemical ionization (CI, magnetic sector), or fast atom bombardment (FAB, magnetic sector). Infrared spectra were recorded with peaks reported in cm^{-1}.

2. Experimental Procedure for the Synthesis of Substrates 1a–u.

2.1. Synthesis of Substrate 1a.



Scheme S1. Synthesis of substrate **1a**.

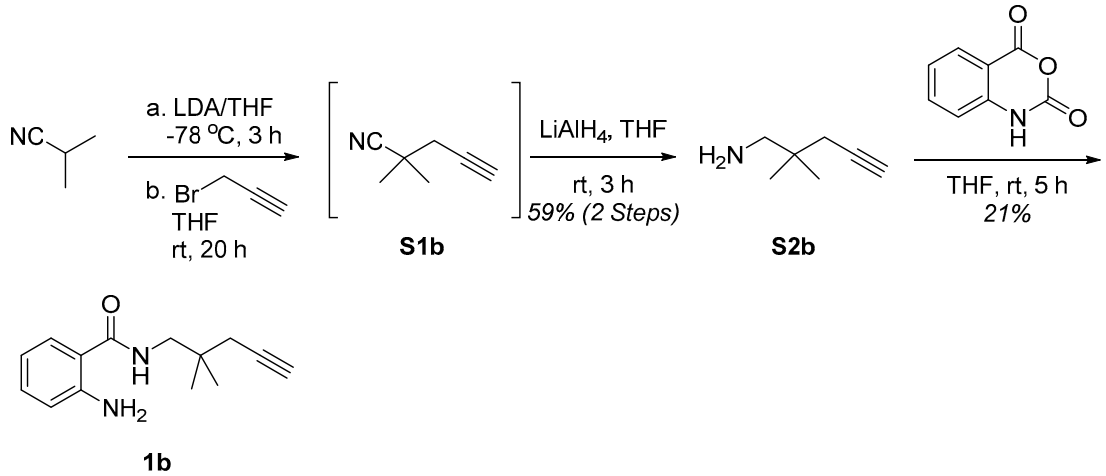
Synthesis of 2-amino-N-(pent-4-yn-1-yl)benzamide (1a).

In a 250 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (2.45 g, 14.4 mmol) and pent-4-yn-1-amine (1.40 mL, 14.4 mmol) were dissolved in anhydrous THF (66 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (2:1 hexanes/EtOAc) to afford **1a** (1.30 g, 6.48 mmol, 45%) as a white solid.

TLC: R_f 0.3 (2:1 hexanes/EtOAc). **mp**: 73.0–75.0 °C. **^{1}H NMR** (400 MHz, C_6D_6) δ 7.02 (ddd, *J* = 8.0, 7.2, 1.6 Hz, 1H), 6.84 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.46 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.23 (dd, *J* = 8.0, 1.2 Hz, 1H), 5.45 (brs, 2H), 5.34 (brs, 1H), 3.18 (td, *J* = 7.0, 6.0 Hz, 2H),

1.83 (td, *J* = 7.0, 2.7 Hz, 2H), 1.72 (t, *J* = 2.7 Hz, 1H), 1.39 (p, *J* = 7.0 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.6, 148.9, 132.4, 127.2, 117.5, 116.7, 116.2, 83.7, 69.5, 39.0, 28.2, 16.4. **HRMS (ESI)** *m/z* calculated for $C_{12}H_{15}N_2O$ $[M + H]^+$ 203.1179, found 203.1181.

2.2. Synthesis of Substrate 1b.



Scheme S2. Synthesis of substrate **1b**.

Synthesis of 2,2-dimethylpent-4-yn-1-amine (S2b).

In a 25 mL oven-dried round-bottom flask with a side arm, *iso*-butyronitrile (390 µL, 4.30 mmol) was dissolved in anhydrous THF (2.5 mL). LDA solution (260 µL, 5.20 mmol, 2.0 M in heptane-THF-ethylbenzene) was added dropwise at 0 °C. After stirred at 0 °C for 3 h, 3-bromo-1-propyne (460 µL, 5.20 mmol) was added dropwise at –78 °C via a syringe and stirred at room temperature for 20 h. Upon completion of the reaction, the reaction mixture was quenched with a saturated aqueous NH_4Cl (20 mL). Two phases were separated, and the aqueous layer was extracted with ether (3 × 60 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, concentrated *in vacuo*. The crude nitrile **S1b** was very volatile and used for the next reaction without purification.

In a 50 mL oven-dried round-bottom flask with a side arm, $LiAlH_4$ (816 mg, 21.5 mmol) was suspended in anhydrous THF (10 mL). A solution of crude nitrile **S1b** in anhydrous THF (7.2 mL) was added dropwise at 0 °C. The resulting grey suspension was stirred at room temperature for 3 h. Upon completion of the reaction, the grey suspension was cooled to 0 °C, diluted with ether (20 mL), and quenched by a sequential addition of water (1.0 mL), a 15% aqueous NaOH (1.0 mL), and water (1.5 mL). The white suspension was then dried over anhydrous Na_2SO_4, filtered, concentrated *in vacuo*. The crude residue was purified by column chromatography (12:1 CH_2Cl_2/MeOH) to afford amine **S2b** (281 mg, 2.53 mmol, 59%, for 2 steps) as a colorless oil.

TLC: R_f 0.20 (12:1 CH_2Cl_2/MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 2.58 (s, 2H), 2.12 (d, *J* = 2.7 Hz, 2H), 1.99 (t, *J* = 2.7 Hz, 1H), 1.30 (brs, 2H), 0.96 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 81.7, 70.6, 51.2, 34.7, 29.5, 24.7. **HRMS (ESI)** *m/z* calcd for $C_7H_{14}N$ $[M + H]^+$ 112.1121, found 112.1121.

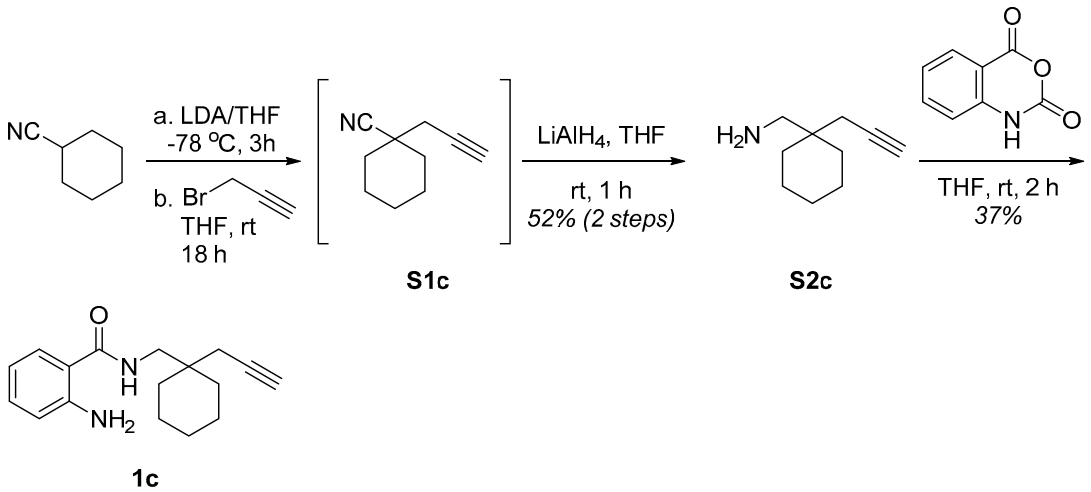
Synthesis of 2-amino-N-(2,2-dimethylpent-4-yn-1-yl)benzamide (1b) (SYN224A)

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (413 mg, 2.53 mmol) and amine **S2b** (260 mg, 2.30 mmol) were dissolved in anhydrous THF

(12 mL) at 0 °C. The reaction mixture was stirred at room temperature for 5 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (15:1 hexanes/EtOAc) to afford **1b** (112 mg, 0.48 mmol, 21%) as a white solid.

TLC: R_f 0.39 (3:1 hexanes/EtOAc). **mp**: 72.2–74.2 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.21 (ddd, *J* = 8.6, 7.2, 1.5 Hz, 1H), 6.72–6.62 (m, 2H), 6.32 (brs, 1H), 5.48 (brs, 2H), 3.39 (d, *J* = 6.4 Hz, 2H), 2.20 (d, *J* = 2.7 Hz, 2H), 2.08 (t, *J* = 2.7 Hz, 1H), 1.07 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.6, 148.8, 132.4, 127.1, 117.5, 116.8, 116.5, 82.0, 71.0, 48.7, 35.1, 30.2, 25.3. **HRMS (ESI)** *m/z* calcd for $C_{14}H_{19}N_2O$ $[M + H]^+$ 231.1492, found 231.1492.

2.3. Synthesis of Substrate 1c.



Scheme S3. Synthesis of substrate **1c**.

Synthesis of (1-(prop-2-yn-1-yl)cyclohexyl)methanamine (S2c).

In a 25 mL oven-dried round-bottom flask with a side arm, *iso*-butyronitrile (540 µL, 4.60 mmol) was dissolved in anhydrous THF (2.7 mL). LDA solution (280 µL, 5.50 mmol, 2.0 M in heptane-THF-ethylbenzene) was added dropwise at 0 °C. After stirred at 0 °C for 3 h, 3-bromo-1-propyne (490 µL, 5.50 mmol) was added dropwise at –78 °C via a syringe and stirred at room temperature for 18 h. Upon completion of the reaction, the reaction mixture was quenched with a saturated aqueous NH_4Cl (20 mL). Two phases were separated, and the aqueous layer was extracted with ether (4 × 40 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, concentrated *in vacuo*. The crude nitrile **S1c** was very volatile and used for the next reaction without purification.

In a 100 mL oven-dried round-bottom flask with a side arm, $LiAlH_4$ (872 mg, 23.0 mmol) was suspended in anhydrous THF (15 mL). A solution of crude nitrile **S1c** in anhydrous THF (8.0 mL) was added dropwise at 0 °C. The resulting grey suspension was stirred at room temperature for 1 h. Upon completion of the reaction, the grey suspension was cooled to 0 °C, diluted with ether (30 mL), and quenched by a sequential addition of water (1.5 mL), a 15% aqueous NaOH (1.5 mL), and water (2.7 mL). The white suspension was then dried over anhydrous Na_2SO_4, filtered, concentrated *in vacuo*. The crude residue was purified by column chromatography (15:1 CH_2Cl_2/MeOH) to afford amine **S2c** (365 mg, 2.41 mmol, 52% for 2 steps) as a colorless oil. Since the amine **S2c** was volatile, it was converted into the corresponding amine salt **S2c′** for characterization: HCl solution (120 µL, 500 µmol, 4 M in dioxane) was added to amine **S2c** (50.0 mg, 330 µmol). After concentrated, Et_2O (0.5 mL) was added. The resulting precipitate was filtered

off and washed with Et_2O (10 mL). The amine salt **S2c'** was obtained as a white solid.

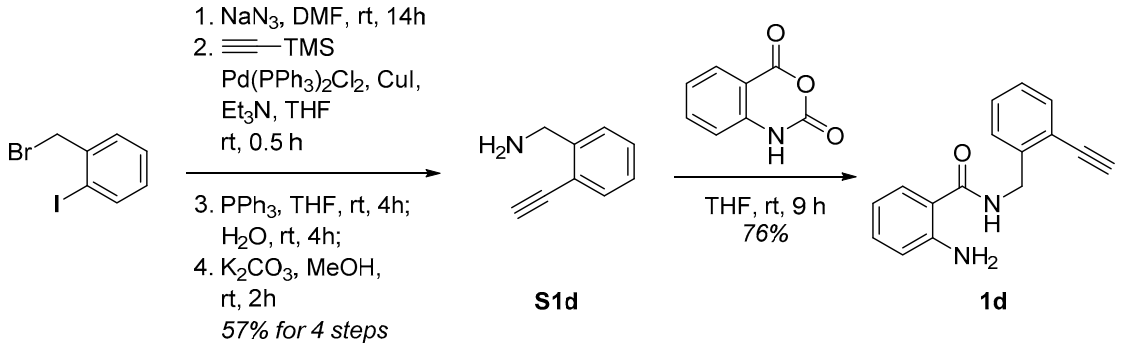
S2c': **TLC**: R_f 0.13 (15:1 CH_2Cl_2/MeOH). **mp:** 192.3–194.3 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.36 (brs, 3H), 3.04 (s, 2H), 2.47 (d, *J* = 2.6 Hz, 2H), 2.08 (t, *J* = 2.6 Hz, 1H), 1.56–1.34 (m, 10H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 80.0, 72.3, 46.8, 35.7, 32.6, 25.6, 25.5, 21.4. **HRMS (ESI)** *m/z* calcd for $C_{10}H_{18}N$ $[M]^+$ 152.1434, found 152.1434.

Synthesis of 2-amino-N-((1-(prop-2-yn-1-yl)cyclohexyl)methyl)benzamide (1c).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (374 mg, 2.20 mmol) and amine **S2c** (302 mg, 2.00 mmol) were dissolved in anhydrous THF (10 mL) at 0 °C. The reaction mixture was stirred at room temperature for 2 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (3:1 hexanes/EtOAc) to afford **1d** (199 mg, 737 μmol, 37%) as a white solid.

TLC: R_f 0.30 (3:1 hexanes/EtOAc). **mp:** 88.5–90.5 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.33 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.20 (ddd, *J* = 8.2, 7.2, 1.5 Hz, 1H), 6.71–6.61 (m, 2H), 6.44 (brs, 1H), 5.50 (brs, 2H), 3.49 (d, *J* = 6.3 Hz, 2H), 2.27 (d, *J* = 2.7 Hz, 2H), 2.10 (t, *J* = 2.7 Hz, 1H), 1.64–1.55 (m, 2H), 1.50–1.40 (m, 8H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.6, 148.9, 132.3, 127.1, 117.4, 116.7, 116.6, 82.1, 71.4, 46.6, 37.2, 33.6, 27.3, 26.1, 21.7. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{23}N_2O$ $[M + H]^+$ 271.1805, found 271.1811.

2.4. Synthesis of Substrate 1d.



Scheme S4. Synthesis of substrate **1d**.

In a 50 mL oven-dried round-bottom flask with a side arm, 2-iodobenyl bromide (1.30 g, 4.38 mmol) and NaN_3 (854 mg, 13.1 mmol) were dissolved in anhydrous DMF (7.3 mL). The reaction mixture was stirred at room temperature for 14 h. Upon completion of the reaction, the mixture was diluted with water (50 mL) and extracted with *n*-hexane (3 × 100 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, and filtered. The concentration by rotary evaporation afforded a volatile crude 1-(azidomethyl)-2-iodobenzene as a colorless liquid, which was used directly in the next step without further purification. In a 50 mL oven-dried round-bottom flask with a side arm, the crude 1-(azidomethyl)-2-iodobenzene, $Pd(PPh_3)_2Cl_2$ (154 mg, 219 μmol), CuI (83.4 mg, 438 μmol), trimethylethynylsilane (473 mg, 4.82 mmol), and Et_3N (21 mL) were dissolved in anhydrous THF (10 mL). The reaction mixture was stirred at room temperature for 30 min. Upon completion of the reaction, the mixture was quenched with saturated aqueous NH_4Cl (3.0 mL) and extracted with ether (3 × 100 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, and filtered. The concentration by rotary evaporation gave a crude ((2-(azidomethyl)phenyl)ethynyl)trimethylsilane as a brown liquid, which was used directly in the next step without further purification. In a 50 mL oven-dried round-bottom flask with a side arm, the crude ((2-(azidomethyl)phenyl)ethynyl)trimethylsilane and PPh_3

(2.30 g, 8.76 mmol) were dissolved in anhydrous THF (5.0 mL). After stirred at room temperature for 4 h, water (5.0 mL) was added, and the mixture was stirred at room temperature for 4 h. Next, K_2CO_3 (1.21 g, 8.76 mmol) and MeOH (1.0 mL) were added, and the mixture was stirred at room temperature for 2 h. The resultant solution was then extracted with CH_2Cl_2 (3 × 100 mL), dried over anhydrous Na_2SO_4, filtered, concentrated *in vacuo*. The crude residue was purified by column chromatography (10:1 CH_2Cl_2/MeOH) to afford amine **S1d** (330 mg, 2.52 mmol, 58% for 4 steps) as a purple oil.

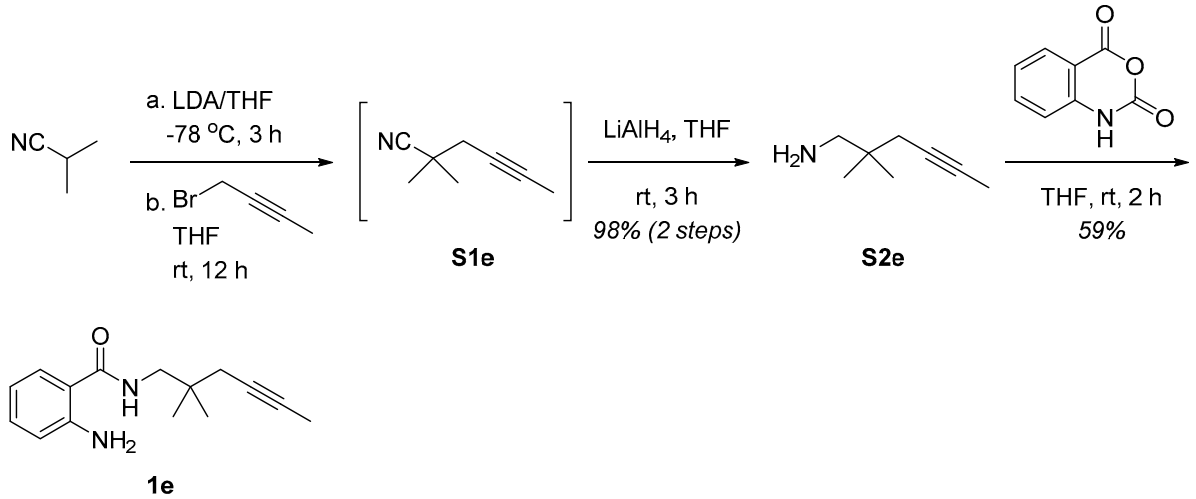
TLC: R_f 0.25 (10:1 CH_2Cl_2/MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.50 (d, *J* = 8.2 Hz, 1H), 7.35 (qd, *J* = 7.6, 1.6 Hz, 2H), 7.23 (td, *J* = 7.4, 2.0 Hz, 1H), 4.01 (s, 2H), 3.33 (s, 1H), 1.92 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 145.9, 133.2, 129.4, 128.2, 127.6, 126.9, 120.9, 81.8, 45.5. **HRMS (ESI)** *m/z* calcd for $C_9H_{10}N$ $[M + H]^+$ 132.0808, found 132.0809.

Synthesis of 2-amino-N-(2-ethynylbenzyl)benzamide (1d).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (424 mg, 2.60 mmol) and amine **S1d** (340 mg, 2.60 mmol) were dissolved in anhydrous THF (13 mL) at 0 °C. The reaction mixture was stirred at room temperature for 9 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (3:1 hexanes/EtOAc) to afford **1d** (495 mg, 1.98 mmol, 76%) as a purple solid.

TLC: R_f 0.23 (3:1 hexanes/EtOAc). **mp**: 104.0–106.0 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.43 (ddd, *J* = 7.7, 1.4, 0.7 Hz, 1H), 7.38–7.30 (m, 2H), 7.29–7.24 (m, 3H), 7.20 (ddd, *J* = 8.5, 7.2, 1.4 Hz, 1H), 6.70–6.62 (m, 2H), 6.58 (s, 1H), 5.53 (s, 2H), 4.75 (d, *J* = 5.9 Hz, 2H), 3.38 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.0, 148.9, 140.7, 133.1, 132.4, 129.3, 128.7, 127.5, 127.1, 121.3, 117.3, 116.6, 115.9, 82.1, 81.6, 42.3. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{15}N_2O$ $[M + H]^+$ 251.1179, found 251.1183.

2.5. Synthesis of Substrate 1e.



Scheme S5. Synthesis of substrate **1e**.

Synthesis of 2,2-dimethylhex-4-yn-1-amine (S2e).

In a 25 mL oven-dried round-bottom flask with a side arm, *iso*-butyronitrile (390 μL, 4.30 mmol) was dissolved in anhydrous THF (2.5 mL). LDA solution (260 μL, 5.20 mmol, 2.0 M in heptane-THF-ethylbenzene) was added dropwise at 0 °C. After stirred at 0 °C for 3 h, 1-bromo-2-butyne (470 μL, 5.20 mmol) was added dropwise at −78 °C via a syringe and stirred at room temperature for 12 h. Upon completion of the reaction, the reaction mixture was quenched with a saturated aqueous

NH_4Cl (20 mL). Two phases were separated, and the aqueous layer was extracted with ether (3 × 40 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, concentrated *in vacuo*. The crude nitrile **S1e** was very volatile and used for the next reaction without purification.

In a 100 mL oven-dried round-bottom flask with a side arm, $LiAlH_4$ (816 mg, 21.5 mmol) was suspended in anhydrous THF (10 mL). A solution of crude nitrile **S1e** in anhydrous THF (7.2 mL) was added dropwise at 0 °C. The resulting grey suspension was stirred at room temperature for 2 h. Upon completion of the reaction, the grey suspension was cooled to 0 °C, diluted with ether (20 mL), and quenched by a sequential addition of water (1.0 mL), a 15% aqueous NaOH (1.0 mL), and water (1.5 mL). The white suspension was then dried over anhydrous Na_2SO_4, filtered, concentrated *in vacuo*. The crude residue was purified by column chromatography (17:1 CH_2Cl_2/MeOH) to afford amine **S2e** (525 mg, 4.20 mmol, 98% for 2 steps) as a colorless oil. Since the amine **S2e** was volatile, it was converted into the corresponding amine salt **S2e′** for characterization: HCl solution (0.11 mL, 0.44 mmol, 4 M in dioxane) was added to amine **S2e** (36.5 mg, 291 μmol). After concentrated, Et_2O (0.5 mL) was added. The resulting precipitate was filtered off and washed with Et_2O (10 mL). The amine salt **S2e′** was obtained as a white solid.

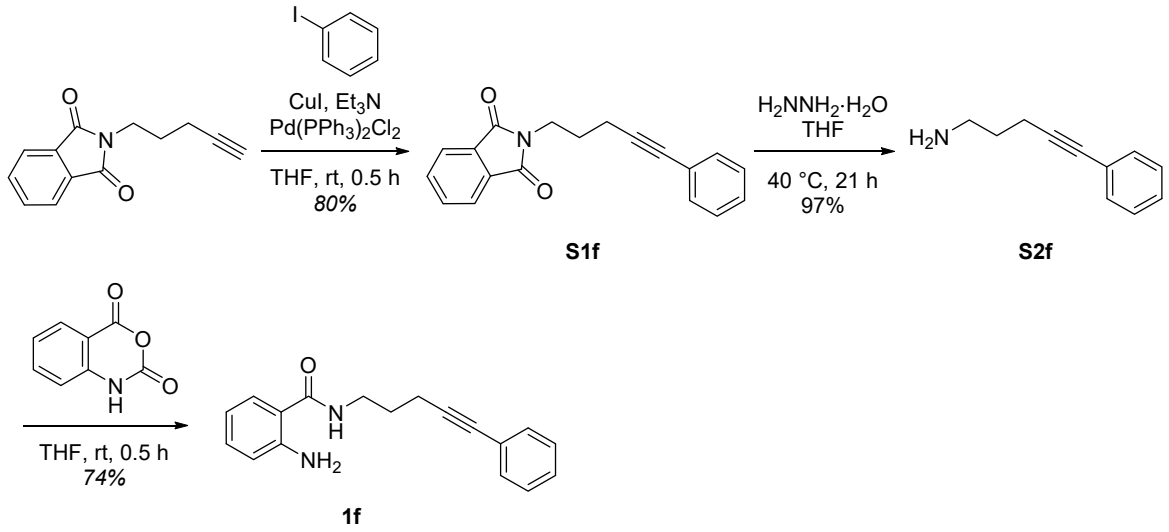
S2e′: **TLC**: R_f 0.10 (17:1 CH_2Cl_2/MeOH). **mp**: 186.8–188.8 °C. **^{1}H NMR** (400 MHz, CD_3CN): δ 8.07 (s, 3H), 2.86 (s, 2H), 2.23 (q, *J* = 2.6 Hz, 2H), 1.77 (t, *J* = 2.6 Hz, 3H), 1.07 (s, 6H). **^{13}C NMR** (100 MHz, CD_3CN): δ 79.6, 76.1, 49.3, 34.1, 30.6, 24.9, 3.4. **HRMS (ESI)** *m/z* calcd for $C_8H_{16}N$ $[M]^+$ 126.1277, found 216.1276.

Synthesis of 2-amino-N-(2,2-dimethylhex-4-yn-1-yl)benzamide (1e).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (431 mg, 2.60 mmol) and amine **S2e** (300 mg, 2.40 mmol) were dissolved in anhydrous THF (12 mL) at 0 °C. The reaction mixture was stirred at room temperature for 2 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (3:1 hexanes/EtOAc) to afford **1e** (377 mg, 1.54 mmol, 59%) as a white solid.

TLC: R_f 0.28 (3:1 hexanes/EtOAc). **mp**: 72.0–74.0 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.34 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.20 (ddd, *J* = 8.1, 7.2, 1.5 Hz, 1H), 6.72–6.61 (m, 2H), 6.55 (s, 1H), 5.48 (s, 2H), 3.36 (d, *J* = 6.2 Hz, 2H), 2.14 (q, *J* = 2.6 Hz, 2H), 1.81 (t, *J* = 2.6 Hz, 3H), 1.03 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.2, 148.5, 132.0, 126.8, 117.1, 116.4, 116.4, 78.2, 77.1, 48.9, 34.7, 30.5, 25.2, 3.4. **HRMS (ESI)** *m/z* calcd for $C_{15}H_{21}N_2O$ $[M + H]^+$ 245.1648, found 245.1650.

2.6. Synthesis of Substrate 1f.



Scheme S6. Synthesis of substrate **1f**.

Synthesis of 2-(5-phenylpent-4-yn-1-yl)isoindoline-1,3-dione (S1f).

In a 100 mL oven-dried round-bottom flask with a side arm, *N*-(4-pentynyl)phthalimide (1.00 g, 4.70 mmol), $Pd(PPh_3)_2Cl$ (165 mg, 235 μmol, 5 mol%) and CuI (90.0 mg, 470 μmol, 10 mol%) were placed. A solution of iodobenzene (520 μL, 4.70 mmol, 1 equiv.) and anhydrous Et_3N (23.5 mL) in anhydrous THF (12 mL) was added via a syringe and stirred at room temperature for 0.5 h. Upon completion of the reaction, the reaction mixture was quenched with a saturated aqueous $NaHCO_3$ (50 mL). Two phases were separated, and the aqueous layer was extracted with CH_2Cl_2 (4 × 50 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography (7:1 hexanes/EtOAc) to afford **S1f** (1.09 g, 3.76 mmol, 80%) as an orange solid.

TLC: R_f 0.20 (6:1 hexanes/EtOAc). **mp**: 129.6–131.6 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.82 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.67 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.30–7.27 (m, 2H), 7.24–7.20 (m, 3H), 3.87 (t, *J* = 7.0 Hz, 2H), 2.50 (t, *J* = 7.0 Hz, 2H), 2.02 (quin, *J* = 7.0 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.6, 134.0, 132.3, 131.6, 128.2, 127.7, 123.8, 123.4, 88.8, 81.4, 37.6, 27.5, 17.5. **HRMS (ESI)** *m*/*z* calculated for $C_{19}H_{16}NO_2$ $[M + H]^+$ 290.1176, found: 290.1178.

Synthesis of 5-phenylpent-4-yn-1-amine (S2f).

In a 25 mL oven-dried round-bottom flask with a side arm, phthalimide **S1f** (1.05 g, 3.60 mmol) was dissolved in anhydrous THF (5.3 mL). Hydrazine monohydrate (1.50 mL, 31.0 mmol) was added dropwise. The reaction mixture was stirred at 40 °C for 21 h. Upon completion of the reaction, the reaction mixture was diluted with ether (100 mL) and the precipitate was filtered off. The organic filtrate was washed with 10% aqueous NaOH solution (20 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo* to afford crude amine **S2f** (548 mg, 97%) as orange oil.

TLC: R_f 0.05 (CH_2Cl_2). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.41–7.37 (m, 2H), 7.29–7.26 (m, 3H), 2.87 (t, *J* = 6.9 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.74 (quin, *J* = 6.9 Hz, 2H), 1.25 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 131.7, 128.3, 127.7, 124.0, 89.7, 81.1, 41.5, 32.6, 17.0. **HRMS (ESI)** *m*/*z* calculated for $C_{11}H_{14}N$ $[M + H]^+$ 160.1121, found 160.1124.

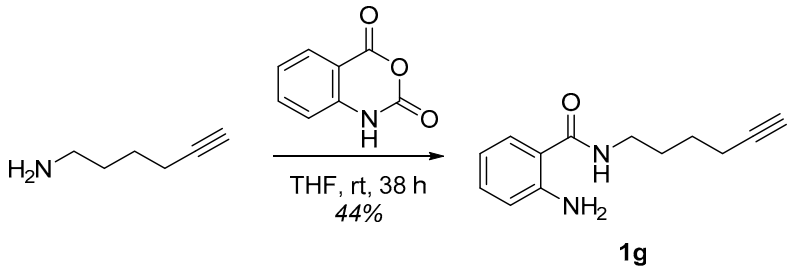
Synthesis of 2-amino-N-(5-phenylpent-4-yn-1-yl)benzamide (1f).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (560 mg, 3.40 mmol) and amine **S2g** (547 mg, 3.40 mmol) were dissolved in anhydrous THF (17 mL) at 0 °C. The reaction mixture was stirred at room temperature for 0.5 h. Upon

completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (3:1 hexanes/EtOAc) to afford **1f** (696 mg, 2.52 mmol, 74%) as a white solid.

TLC: R_f 0.23 (2:1 hexanes/EtOAc). **mp**: 97.9–99.9 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42–7.34 (m, 2H), 7.31–7.27 (m, 4H), 7.17 (ddd, *J* = 8.6, 7.2, 1.5 Hz, 1H), 6.66 (dd, *J* = 8.2, 1.2 Hz, 1H), 6.51 (ddd, *J* = 8.2, 7.2, 1.2 Hz, 1H), 6.39 (s, 1H), 5.52 (s, 2H), 3.61 (td, *J* = 6.8, 5.7 Hz, 2H), 2.56 (t, *J* = 6.8 Hz, 2H), 1.94 (quin, *J* = 6.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.5, 148.9, 132.4, 131.8, 128.4, 128.0, 127.2, 123.6, 117.4, 116.7, 116.2, 89.3, 81.8, 39.5, 28.4, 17.6. **HRMS (ESI)** *m/z* calcd for $C_{18}H_{19}N_2O$ $[M + H]^+$ 279.1492, found 279.1495.

2.7. Synthesis of Substrate 1g.



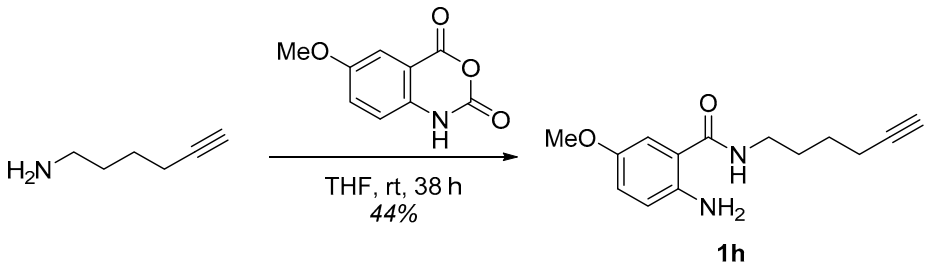
Scheme S7. Synthesis of substrate **1g**.

Synthesis of 2-amino-N-(hex-5-yn-1-yl)benzamide (1g).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (839 mg, 5.14 mmol) and hex-5-yn-1-amine (580 µL, 5.14 mmol) were dissolved in anhydrous THF (26 mL) at 0 °C. The reaction mixture was stirred at room temperature for 38 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (2.5:1 hexanes/EtOAc) to afford **1g** (490 mg, 2.26 mmol, 44%) as a purple solid.

TLC: R_f 0.23 (2.5:1 hexanes/EtOAc). **mp**: 50.6–52.6 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): 7.30 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.20 (td, *J* = 7.6, 1.2 Hz, 1H), 6.71–6.61 (m, 2H), 6.06 (brs, 1H), 5.49 (brs, 2H), 3.45 (td, *J* = 7.0, 5.8 Hz, 2H), 2.26 (td, *J* = 6.8, 2.6 Hz, 2H), 1.97 (t, *J* = 2.6 Hz, 1H), 1.78–1.70 (m, 2H), 1.67–1.60 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.50, 148.79, 132.38, 127.13, 117.46, 116.75, 116.42, 84.18, 68.94, 39.26, 28.87, 25.93, 18.29. **HRMS (ESI)** *m*/*z* calculated for $C_{13}H_{17}N_2O$ $[M + H]^+$ 217.1335, found 217.1339.

2.8. Synthesis of Substrate 1h.



Scheme S8. Synthesis of substrate **1h**.

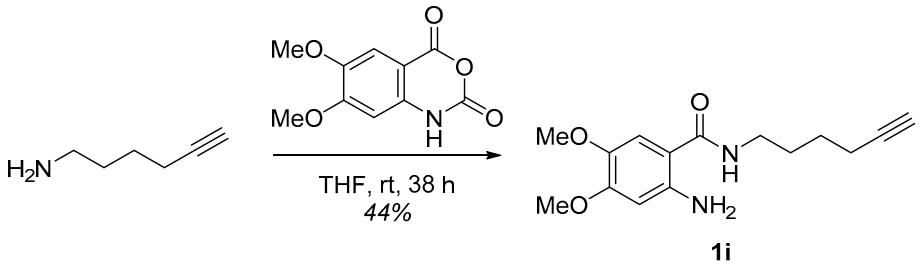
Synthesis of 2-amino-N-(hex-5-yn-1-yl)-5-methoxybenzamide (1h).

In a 25 mL oven-dried round-bottom flask with a side arm, 5-methoxyisatoic anhydride (500 mg, 2.59 mmol) and hex-5-yn-1-amine (876 µL, 7.76 mmol) were dissolved in anhydrous THF (25 mL) at 0 °C. The reaction mixture was stirred at room temperature for

36 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (2.5:1 hexanes/EtOAc) to afford **1h** (593 mg, 2.41 mmol, 93%) as a yellow solid.

TLC: R_f 0.20 (2.5:1 hexanes/EtOAc). **mp**: 61.0–62.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.22 (t, *J* = 5.7 Hz, 1H), 7.03 (d, *J* = 2.9 Hz, 1H), 6.82 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.64 (d, *J* = 8.9 Hz, 1H), 5.91 (s, 2H), 3.68 (s, 3H), 3.21 (td, *J* = 6.9, 5.7 Hz, 2H), 2.76 (t, *J* = 2.7 Hz, 1H), 2.19 (td, *J* = 7.0, 2.7 Hz, 2H), 1.65–1.54 (m, 2H), 1.54–1.42 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 168.49, 149.32, 143.66, 119.14, 117.59, 115.33, 112.13, 84.44, 71.23, 55.57, 38.20, 28.32, 25.53, 17.43. **HRMS (ESI)** *m*/*z* calculated for $C_{14}H_{19}N_2O_2$ $[M + H]^+$ 247.1441, found 247.1440.

2.9. Synthesis of Substrate 1i.



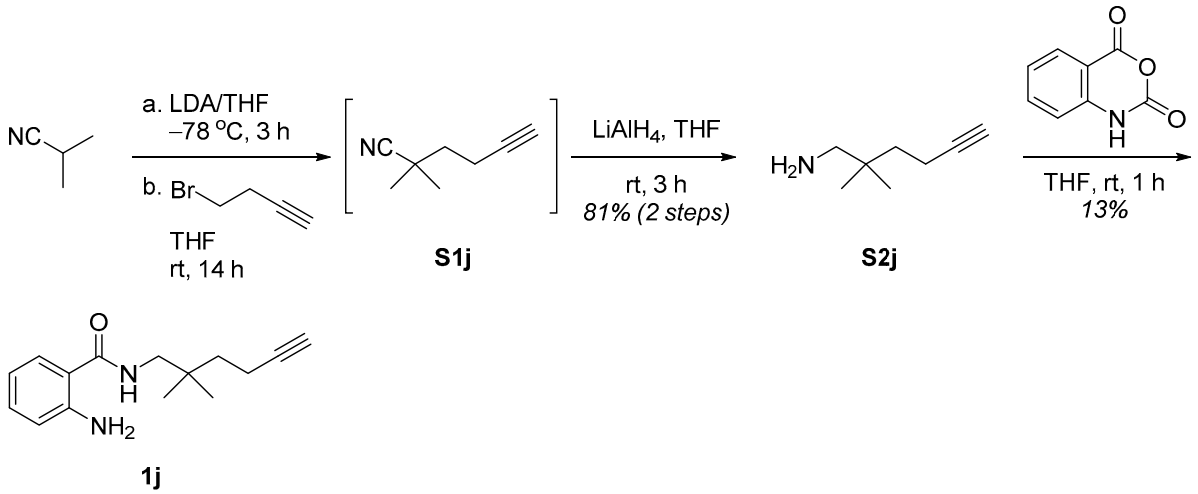
Scheme S9. Synthesis of substrate **1i**.

Synthesis of 2-amino-N-(hex-5-yn-1-yl)benzamide (1i).

In a 25 mL oven-dried round-bottom flask with a side arm, 4,5-dimethoxyisatoic anhydride (500 mg, 2.24 mmol) and hex-5-yn-1-amine (632 μL, 5.60 mmol) were dissolved in anhydrous THF (11 mL) at 0 °C. The reaction mixture was stirred at room temperature for 23 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (2.5:1 hexanes/EtOAc) to afford **1i** (581 mg, 2.10 mmol, 94%) as a white solid.

TLC: R_f 0.21 (2:1 hexanes/EtOAc). **mp**: 75.1–76.3 °C. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.00 (t, *J* = 5.7 Hz, 1H), 7.07 (s, 1H), 6.29 (d, *J* = 2.7 Hz, 3H), 3.70 (s, 3H), 3.67 (s, 3H), 3.19 (td, *J* = 6.9, 5.6 Hz, 2H), 2.76 (t, *J* = 2.6 Hz, 1H), 2.19 (td, *J* = 6.9, 2.7 Hz, 2H), 1.65–1.52 (m, 2H), 1.47 (dtd, *J* = 9.5, 7.0, 4.9 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 168.40, 152.69, 146.21, 138.97, 112.55, 105.25, 99.89, 84.50, 71.27, 56.73, 55.09, 38.18, 28.55, 25.61, 17.48. **HRMS (ESI)** *m*/*z* calculated for $C_{15}H_{21}N_2O_3$ $[M + H]^+$ 277.1547, found 277.1540.

2.10. Synthesis of Substrate 1j.



Scheme S10. Synthesis of substrate **1j**.

Synthesis of 2,2-dimethylhex-5-yn-1-amine (S2j).

In a 25 mL oven-dried round-bottom flask with a side arm, *iso*-butyronitrile (650 μ L, 7.20 mmol) was dissolved in anhydrous THF (4.3 mL). LDA solution (440 μL, 8.70 mmol, 2.0 M in heptane-THF-ethylbenzene) was added dropwise at 0 °C. After stirred at 0 °C for 3 h, 4-bromo-1-butyne (820 μL, 8.70 mmol) was added dropwise at –78 °C via a syringe and stirred at room temperature for 14 h. Upon completion of the reaction, the reaction mixture was quenched with a saturated aqueous NH_4Cl (20 mL). Two phases were separated, and the aqueous layer was extracted with ether (3 × 40 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, concentrated *in vacuo*. The crude nitrile **S1j** was very volatile and used for the next reaction without purification.

In a 50 mL oven-dried round-bottom flask with a side arm, $LiAlH_4$ (391 mg, 10.3 mmol) was suspended in anhydrous THF (5.3 mL). A solution of crude nitrile **S1j** in anhydrous THF (5.0 mL) was added dropwise at 0 °C. The resulting grey suspension was stirred at room temperature for 6 h. Upon completion of the reaction, the grey suspension was cooled to 0 °C, diluted with ether (20 mL), and quenched by a sequential addition of water (1.0 mL), a 15% aqueous NaOH (1.0 mL), and water (1.5 mL). The resulting white suspension was then dried over anhydrous Na_2SO_4, filtered, concentrated *in vacuo*. The crude residue was purified by column chromatography (15:1 CH_2Cl_2/MeOH) to afford amine **S2j** (730 mg, 5.83 mmol, 81% for 2 steps) as a colorless oil. Since the amine **S2j** was volatile, it was converted into the corresponding amine salt **S2j'** for characterization: HCl solution (60.0 μL, 230 μmol, 4 M in dioxane) was added to amine **S2j** (18.7 mg, 150 μmol). After concentrated, Et_2O (0.5 mL) was added. The resulting precipitate was filtered off and washed with Et_2O (10 mL). The amine salt **S2j'** was obtained as white solid.

S2j': **TLC**: R_f 0.15 (15:1 CH_2Cl_2/MeOH). **mp**: 126.1–128.1 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.38 (s, 3H), 2.86 (s, 2H), 2.22 (td, *J* = 7.7, 2.7 Hz, 2H), 1.98 (t, *J* = 2.7 Hz, 1H), 1.66 (t, *J* = 7.7 Hz, 4H), 1.10 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 84.1, 69.2, 49.5, 38.4, 33.4, 24.7, 13.6. **HRMS (ESI)** *m/z* calcd for $C_8H_{16}N$ $[M]^+$ 126.1277, found 126.1279.

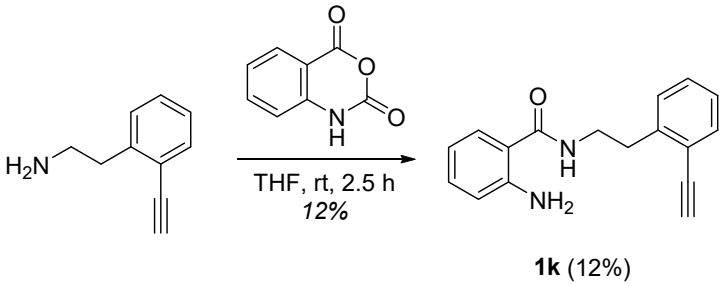
Synthesis of 2-amino-N-(2,2-dimethylhex-5-yn-1-yl)benzamide (1j).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (1.29 g, 7.90 mmol) and amine **S2j** (901 mg, 7.20 mmol) were dissolved in anhydrous THF (36 mL) at 0 °C. The reaction mixture was stirred at room temperature for 1 h. Upon comple-

tion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (4:1 hexanes/EtOAc) to afford **1j** (233 mg, 0.95 mmol, 13%) as a white solid.

TLC: R_f 0.18 (4:1 hexanes/EtOAc). **mp**: 72.7–74.7 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.21 (ddd, *J* = 8.2, 7.2, 1.5 Hz, 1H), 6.72–6.62 (m, 2H), 6.20 (s, 1H), 5.46 (s, 1H), 3.29 (d, *J* = 6.5 Hz, 2H), 2.25 (td, *J* = 7.8, 2.7 Hz, 2H), 1.98 (t, *J* = 2.7 Hz, 1H), 1.63–1.54 (m, 3H), 0.97 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.5, 148.8, 132.4, 127.0, 117. 5, 116.8, 116.7, 85.3, 68.5, 48.3, 38.6, 34.9, 25.1, 13.7. **HRMS (ESI)** *m/z* calcd for $C_{15}H_{21}N_2O$ $[M + H]^+$ 245.1648, found 245.1649.

2.11. Synthesis of Substrate 1k.



1k (12%)

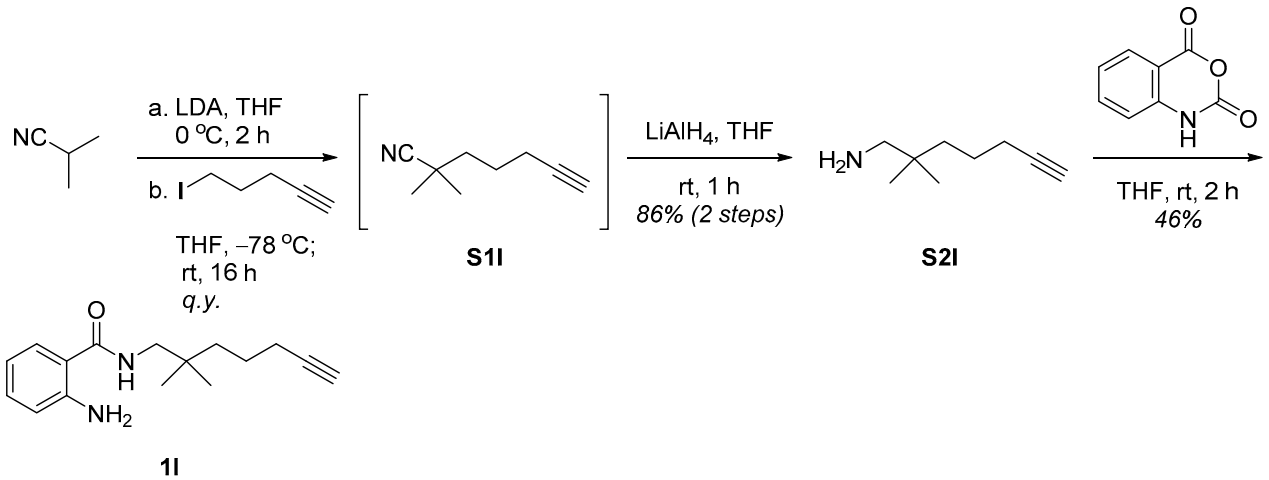
Scheme S11. Synthesis of substrate **1k**.

Synthesis of 2-amino-N-(2-ethynylphenethyl)benzamide (1k).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (556 mg, 3.40 mmol) and 2-(2-ethynylphenyl)ethan-1-amine (450 mg, 3.10 mmol) were dissolved in anhydrous THF (16 mL) at 0 °C. The reaction mixture was stirred at room temperature for 2.5 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by column chromatography (4:1 hexanes/EtOAc) to afford **1k** (97.2 mg, 370 μmol, 12%) as a white solid.

TLC: R_f 0.18 (4:1 hexanes/EtOAc). **mp**: 132.1–134.1 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.55–7.49 (m, 1H), 7.32 (td, *J* = 7.5, 1.5 Hz, 1H), 7.25–7.19 (m, 3H), 7.19–7.14 (m, 1H), 6.70–6.57 (m, 2H), 6.13 (s, 1H), 5.49 (s, 2H), 3.77–3.68 (m, 2H), 3.30 (s, 1H), 3.13 (t, *J* = 6.7 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.5, 148.8, 141.9, 133.3, 132.3, 129.6, 129.4, 127.3, 126.8, 122.0, 117.4, 116.7, 116.4, 82.4, 81.5, 40.5, 34.1. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{17}N_2O$ $[M + H]^+$ 265.1335, found 265.1337.

2.12. Synthesis of Substrate 1l.



1l

Scheme S12. Synthesis of substrate **1l**.

Synthesis of 2,2-dimethylhept-6-yn-1-amine (S2l).

In a 100 mL oven-dried round-bottom flask with a side arm, NaI (8.77 g, 58.5 mmol) and 5-chloro-1-pentyne (3.00 g, 29.3 mmol) were dissolved in anhydrous acetone (29 mL). The reaction mixture was stirred at 80 °C under reflux for 18 h. Upon completion of the reaction, the insoluble NaCl was filtered off and the filtrate was concentrated *in vacuo* to afford crude 5-iodo-1-pentyne (5.68 g, 29.3 mmol, 100%) as dark red liquid. The crude residue was very volatile and used for next reaction without purification.

In a 100 mL oven-dried round-bottom flask with a side arm, *iso*-butyronitrile (1.68 g, 24.3 mmol) was dissolved in anhydrous THF (14.4 mL). LDA solution (14.7 mL, 29.4 mmol, 1.2 equiv. 2.0 M in heptane-THF-ethylbenzene) was added dropwise at 0 °C. After stirred at 0 °C for 2 h, 5-iodo-1-pentyne (5.68 g, 29.3 mmol, 1.2 equiv.) was added dropwise at –78 °C via a syringe and stirred at room temperature for 17 h. Upon completion of the reaction, the reaction mixture was quenched with saturated aqueous NH_4Cl (80 mL). The aqueous layer was extracted with ether (3 × 120 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude nitrile **S1l** as a red oil, which was very volatile and used for the next reaction without purification.

In a 25 mL oven-dried round-bottom flask with a side arm, $LiAlH_4$ (2.31 g, 61.0 mmol, 2.5 equiv.) was suspended in anhydrous THF (40 mL). A solution of crude nitrile in anhydrous THF (40 mL) was added dropwise at 0 °C. The resulting grey suspension was stirred at room temperature for 1 h. Upon completion of the reaction, the grey suspension was cooled to 0 °C, diluted with ether (45 mL), and quenched by a sequential addition of water (3.0 mL), a 15% aqueous NaOH (3.0 mL), and water (9.0 mL). The white suspension was then dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography (10:1 CH_2Cl_2/MeOH) to afford amine **S2l** (2.91 g, 20.9 mmol, 86%) as a colorless oil. Since the amine **S2l** was volatile, it was converted into the corresponding amine salt **S2l'** for characterization: HCl solution (80 μL, 320 μmol, 4 M in dioxane) was added to amine **S2l** (30.0 mg, 220 μmol). After concentrated, Et_2O (0.5 mL) was added. The resulting precipitate was filtered off and washed with Et_2O (10 mL). The amine salt **S2j'** was obtained as white solid.

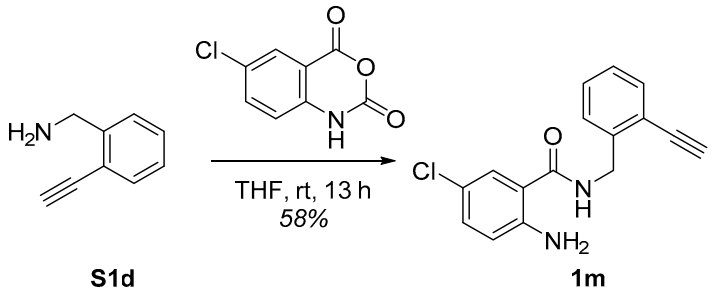
S2l': TLC: R_f 0.15 (10:1 CH_2Cl_2/MeOH). **mp**: 80.5–82.5 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.35 (s, 3H), 2.80 (d, *J* = 5.8 Hz, 2H), 2.22 (td, *J* = 6.6, 2.6 Hz, 2H), 1.96 (t, *J* = 2.6 Hz, 1H), 1.64 – 1.59 (m, 2H), 1.51 – 1.43 (m, 2H), 1.08 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 84.2, 68.8, 49.6, 38.9, 33.1, 25.0, 23.1, 19.0. **HRMS (ESI)** m/z calcd for $C_9H_{18}N$ $[M]^+$ 140.1434, found 140.1435.

Synthesis of 2-amino-N-(2,2-dimethylhept-6-yn-1-yl)benzamide (1l).

In a 25 mL oven-dried round-bottom flask with a side arm, isatoic anhydride (3.91 g, 23.0 mmol, 1.1 equiv.) and amine **S2l** (2.91 g, 20.9 mmol) were dissolved in anhydrous THF (115 mL) at 0 °C. The reaction mixture was stirred at room temperature for 2 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (5:1 hexanes/EtOAc) to afford **1l** (2.47 g, 9.56 mmol, 46%) as a white solid.

TLC: R_f 0.25 (4:1 hexanes/EtOAc). **mp**: 79.6–81.6 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.21 (ddd, *J* = 8.2, 7.2, 1.5 Hz, 1H), 6.72–6.61 (m, 2H), 6.06 (s, 1H), 5.43 (s, 2H), 3.27 (d, *J* = 6.3 Hz, 2H), 2.20 (td, *J* = 6.9, 2.6 Hz, 2H), 1.94 (t, *J* = 2.6 Hz, 1H), 1.60–1.50 (m, 2H), 1.44–1.35 (m, 2H), 0.96 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.5, 148.7, 132.3, 127.1, 117.4, 116.9, 116.7, 84.7, 68.7, 48.8, 39.0, 34.6, 25.3, 23.3, 19.3. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{23}N_2O$ $[M + H]^+$ 259.1805, found: 259.1806.

2.13. Synthesis of Substrate 1m.



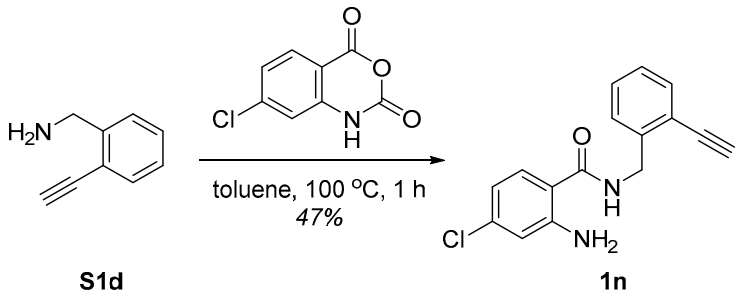
Scheme S13. Synthesis of substrate **1m**.

Synthesis of 2-amino-5-chloro-N-(2-ethynylbenzyl)benzamide (1m).

In a 25 mL oven-dried round-bottom flask with a side arm, 5-chloroisatoic anhydride (431 mg, 2.18 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (286 mg, 2.18 mmol) were dissolved in anhydrous THF (2.5 mL) at 0 °C. The reaction mixture was stirred at room temperature for 13 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (5:1 hexanes/EtOAc) to afford **1m** (359 mg, 1.26 mmol, 58%) as a white solid.

TLC: R_f 0.324:1 hexanes/EtOAc). **mp**: 165.2–166.8 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.54 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.45–7.40 (m, 1H), 7.36 (td, *J* = 7.5, 1.5 Hz, 1H), 7.32–7.28 (m, 2H), 7.15 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.62 (d, *J* = 8.7 Hz, 1H), 6.51 (s, 1H), 5.51 (s, 2H), 4.74 (d, *J* = 5.9 Hz, 2H), 3.39 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.9, 146.7, 140.4, 133.33, 132.3, 129.5, 128.8, 127.7, 126.9, 121.7, 121.4, 119.0, 117.3, 82.4, 81.7, 42.6. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{14}ClN_2O$ $[M + H]^+$ 285.0789, found: 285.0795.

2.14. Synthesis of Substrate 1n.



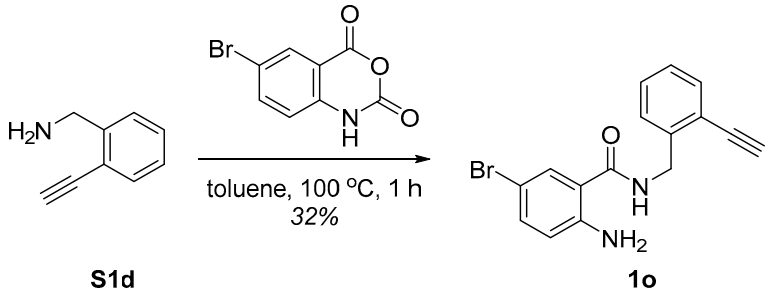
Scheme S14. Synthesis of substrate **1n**.

Synthesis of 2-amino-4-chloro-N-(2-ethynylbenzyl)benzamide (1n).

In a 25 mL oven-dried round-bottom flask with a side arm, 4-chloroisatoic anhydride (549 mg, 2.78 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (365 mg, 2.78 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100 °C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (7:1 hexanes/EtOAc) to afford **1n** (370 mg, 1.30 mmol, 47%) as a white solid.

TLC: R_f 0.30 (5:1 hexanes/EtOAc). **mp**: 112.0–113.7 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.44–7.39 (m, 1H), 7.35 (td, *J* = 7.5, 1.5 Hz, 1H), 7.28 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 1H), 6.67 (d, *J* = 2.0 Hz, 1H), 6.59 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.52 (s, 1H), 5.66 (s, 2H), 4.73 (d, *J* = 5.9 Hz, 2H), 3.38 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.4, 149.8, 140.6, 138.2, 133.2, 129.5, 128.8, 128.5, 127.7, 121.4, 116.9, 114.39, 82.3, 81.7, 42.5. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{14}ClN_2O$ $[M + H]^+$ 285.0789, found: 285.0794.

2.15. Synthesis of Substrate 1o.



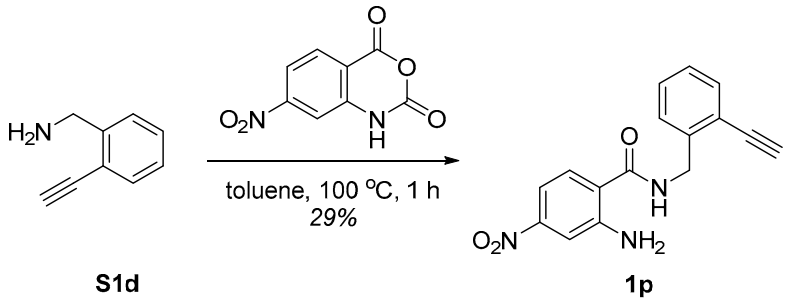
Scheme S15. Synthesis of substrate **1o**.

Synthesis of 2-amino-5-bromo-N-(2-ethynylbenzyl)benzamide (1o).

In a 25 mL oven-dried round-bottom flask with a side arm, 5-bromoisatoic anhydride (581 mg, 2.40 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (315 mg, 2.40 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (5:1 hexanes/EtOAc) to afford **1o** (255 mg, 775 μmol, 32%) as a yellow solid.

TLC: R_f 0.20 (4:1 hexanes/EtOAc). **mp**: 153.2–154.6 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.54 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.44–7.40 (m, 2H), 7.36 (td, *J* = 7.5, 1.5 Hz, 1H), 7.29 (dd, *J* = 7.6, 1.7 Hz, 1H), 6.57 (d, *J* = 8.7 Hz, 1H), 6.50 (s, 1H), 5.53 (s, 2H), 4.74 (d, *J* = 5.8 Hz, 2H), 3.40 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.9, 147.7, 140.4, 135.1, 133.3, 129.8, 129.4, 128.7, 127.7, 121.4, 119.0, 117.5, 107.9, 82.4, 81.7, 42.5. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{14}BrN_2O$ $[M + H]^+$ 329.0284, found: 329.0291.

2.16. Synthesis of Substrate 1p.



Scheme S16. Synthesis of substrate **1p**.

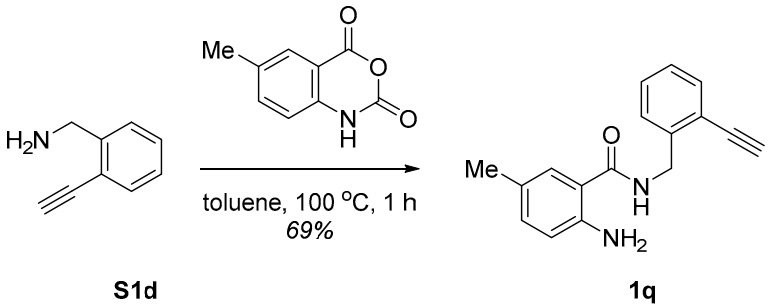
Synthesis of 2-Amino-N-(2-ethynyl-benzyl)-4-nitro-benzamide (1p).

In a 25 mL oven-dried round-bottom flask with a side arm, 4-nitroisatoic anhydride (365 mg, 1.75 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (230 mg, 1.75 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (7:1 hexanes/EtOAc) to afford **1p** (147mg, 498 μmol, 29%) as a yellow solid.

TLC: R_f 0.25 (4:1 hexanes/EtOAc). **mp**: 160.0–161.7 °C. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.08 (t, *J* = 5.8 Hz, 1H), 7.79 (d, *J* = 8.7 Hz, 1H), 7.59 (d, *J* = 2.4 Hz, 1H), 7.49 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.39 (td, *J* = 7.5, 1.4 Hz, 1H), 7.37–7.24 (m, 3H), 6.86 (s, 2H), 4.59 (d, *J* = 5.7 Hz, 2H), 4.48 (s, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 167.5, 150.2, 149.5, 141.0, 132.29,

129.8, 129.0, 126.9, 126.5, 120.2, 119.4, 110.1, 108.2, 85.7, 81.1, 40.9. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{13}N_3O_3$ $[M + H]^+$ 296.1030, found: 296.1031.

2.17. Synthesis of Substrate 1q.

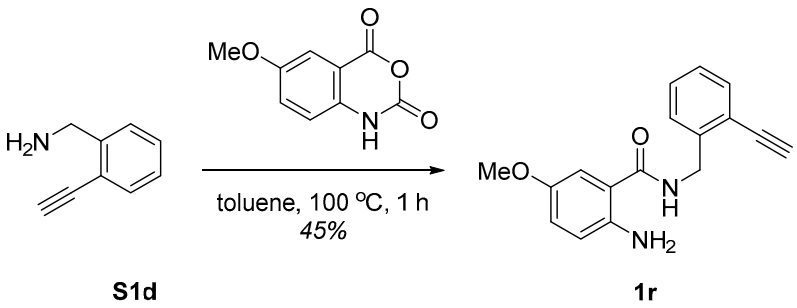


Scheme S17. Synthesis of substrate **1q**.

In a 25 mL oven-dried round-bottom flask with a side arm, 5-methylisatoic anhydride (250 mg, 1.40 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (185 mg, 1.40 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (4:1 hexanes/EtOAc) to afford **1q** (256 mg, 968 μmol, 69%) as a white solid.

TLC: R_f 0.45 (2:1 hexanes/EtOAc). **mp**: 136.0–137.4 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.51 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.40 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.32 (td, *J* = 7.6, 1.4 Hz, 1H), 7.24 (td, *J* = 7.5, 1.4 Hz, 1H), 7.12 (s, 1H), 7.00 (dd, *J* = 8.2, 2.0 Hz, 1H), 6.64 (s, 1H), 6.58 (d, *J* = 8.2 Hz, 1H), 5.31 (s, 2H), 4.73 (d, *J* = 5.9 Hz, 2H), 3.37 (s, 1H), 2.20 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.1, 145.5, 140.8, 133.3, 133.1, 129.4, 128.6, 127.5, 127.4, 126.6, 121.3, 118.0, 116.6, 82.3, 81.7, 42.3, 20.5. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{17}N_2O$ $[M + H]^+$ 265.1335, found: 265.1337.

2.18. Synthesis of Substrate 1r.



Scheme S18. Synthesis of substrate **1r**.

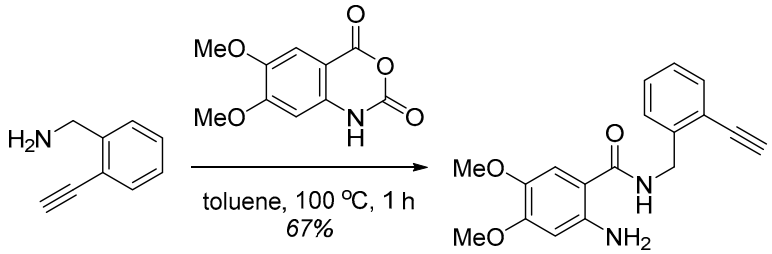
Synthesis of 2-amino-N-(2-ethynylbenzyl)-5-methoxybenzamide (1r).

In a 25 mL oven-dried round-bottom flask with a side arm, 5-methoxyisatoic anhydride (490 mg, 2.54 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (333 mg, 2.54 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (5:1 hexanes/EtOAc) to afford **1r** (323 mg, 1.15 mmol, 45%) as a white solid.

TLC: R_f 0.20 (5:1 hexanes/EtOAc). **mp**: 106.9–108.8 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.43 (ddd, *J* = 7.6, 1.4, 0.6 Hz, 1H), 7.35 (td, *J* = 7.6, 1.5 Hz, 1H),

7.28 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.25 (d, *J* = 1.5 Hz, 4H), 6.91 (d, *J* = 2.8 Hz, 1H), 6.87 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.66 (dd, *J* = 8.7, 0.5 Hz, 2H), 4.75 (d, *J* = 5.9 Hz, 2H), 3.74 (s, 3H), 3.38 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.7, 151.3, 142.4, 140.7, 133.1, 129.4, 128.6, 127.5, 121.3, 119.3, 118.9, 117.4, 112.3, 82.2, 81.7, 56.0, 42.4. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{16}N_2NaO_2$ $[M + Na]^+$ 303.1104, found: 303.1102.

2.19. Synthesis of Substrate 1s.



S1d **1s**

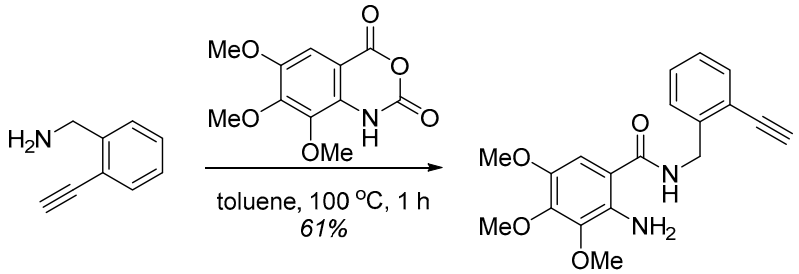
Scheme S19. Synthesis of substrate **1s**.

Synthesis of 2-amino-N-(2-ethynylbenzyl)-4,5-dimethoxybenzamide (1s).

In a 25 mL oven-dried round-bottom flask with a side arm, 4,5-dimethoxyisatoic anhydride (147 mg, 661 μmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (86.7 mg, 661 μmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (2:1 hexanes/EtOAc) to afford **1s** (138 mg, 445 μmol, 67%) as a brown solid.

TLC: R_f 0.35 (2:1 hexanes/EtOAc). **mp**: 119.3–120.8 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.52 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.41 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.33 (td, *J* = 7.5, 1.5 Hz, 1H), 7.25 (td, *J* = 8.9, 7.9, 1.4 Hz, 1H), 6.87 (s, 1H), 6.66 (s, 1H), 6.19 (s, 1H), 5.41 (s, 2H), 4.73 (d, *J* = 5.9 Hz, 2H), 3.84 (s, 3H), 3.79 (s, 3H), 3.38 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.6, 153.4, 144.8, 141.0, 140.9, 133.1, 129.3, 128.6, 127.4, 121.2, 111.0, 107.1, 104.6, 100.8, 82.0, 57.0, 55.7, 42.3. **HRMS (ESI)** *m/z* calcd for $C_{18}H_{19}N_2O_3$ $[M + H]^+$ 311.1390, found: 311.1398.

2.20. Synthesis of Substrate 1t.



S1d **1t**

Scheme S20. Synthesis of substrate **1t**.

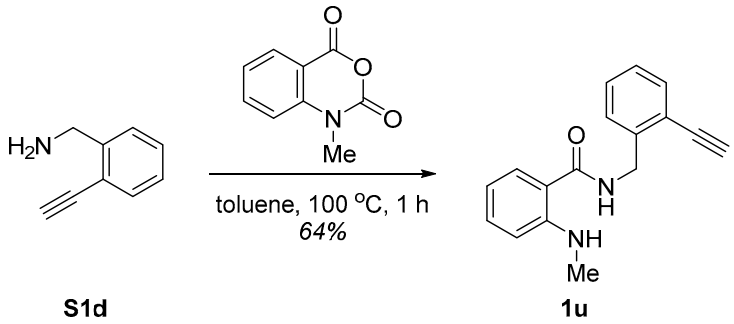
Synthesis of 2-amino-N-(2-ethynylbenzyl)-3,4,5-trimethoxybenzamide (1t).

In a 25 mL oven-dried round-bottom flask with a side arm, 3,4,5-trimethoxyisatoic anhydride (483 mg, 1.90 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (250 mg, 1.90 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was

stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (2:1 hexanes/EtOAc) to afford **1t** (397 mg, 1.17 mmol, 61%) as a white solid.

TLC: R_f 0.35 (2:1 hexanes/EtOAc). **mp**: 118.0–119.6 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.39 (ddd, *J* = 7.7, 1.4, 0.6 Hz, 1H), 7.31 (td, *J* = 7.6, 1.5 Hz, 1H), 7.23 (td, *J* = 7.5, 1.4 Hz, 1H), 6.87 (s, 1H), 6.76 (s, 1H), 4.71 (d, *J* = 5.9 Hz, 2H), 3.90 (s, 3H), 3.85 (s, 3H), 3.77 (s, 3H), 3.37 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.4, 146.0, 144.7, 141.7, 140.9, 137.2, 133.1, 129.4, 128.6, 127.4, 121.2, 111.3, 106.7, 82.2, 81.85, 60.9, 60.5, 57.1, 42.4. **HRMS (ESI)** *m/z* calcd for $C_{19}H_{21}N_2O_4$ $[M + H]^+$ 341.1496, found: 341.1501.

2.21. Synthesis of Substrate 1u.



Scheme S21. Synthesis of substrate **1u**.

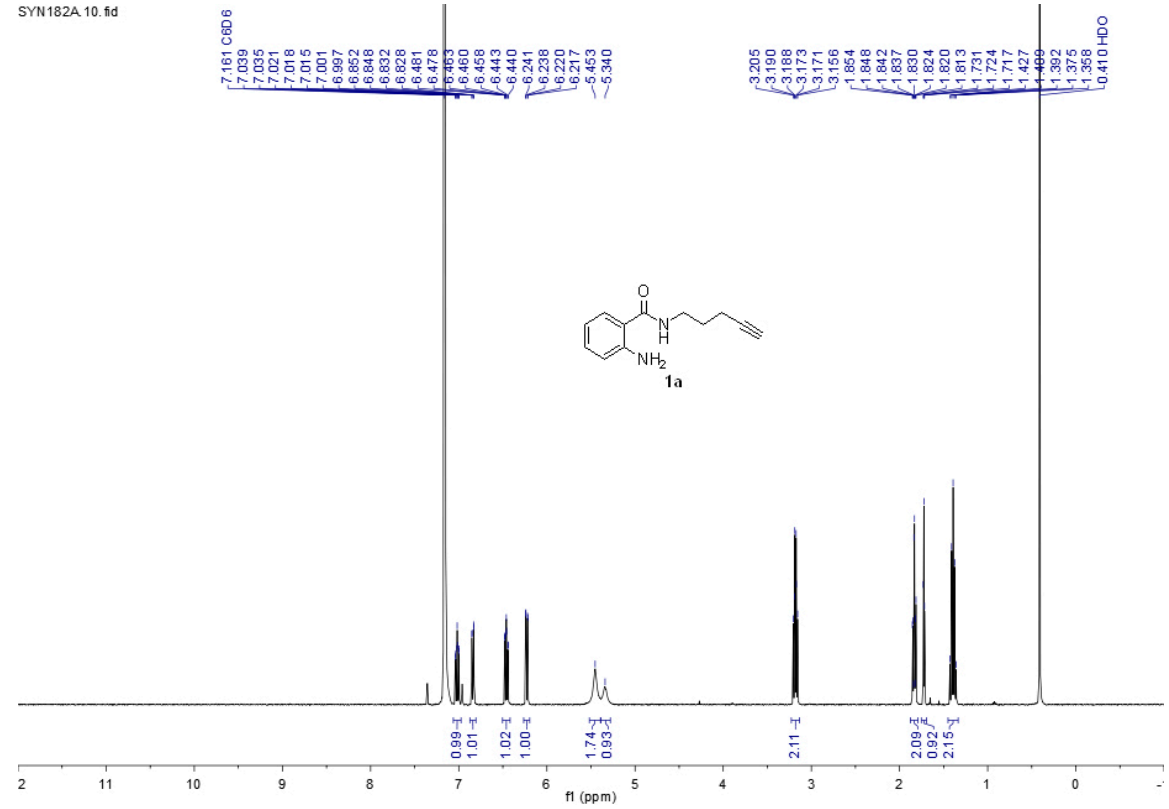
Synthesis of N-(2-ethynylbenzyl)-2-(methylamino)benzamide (1u).

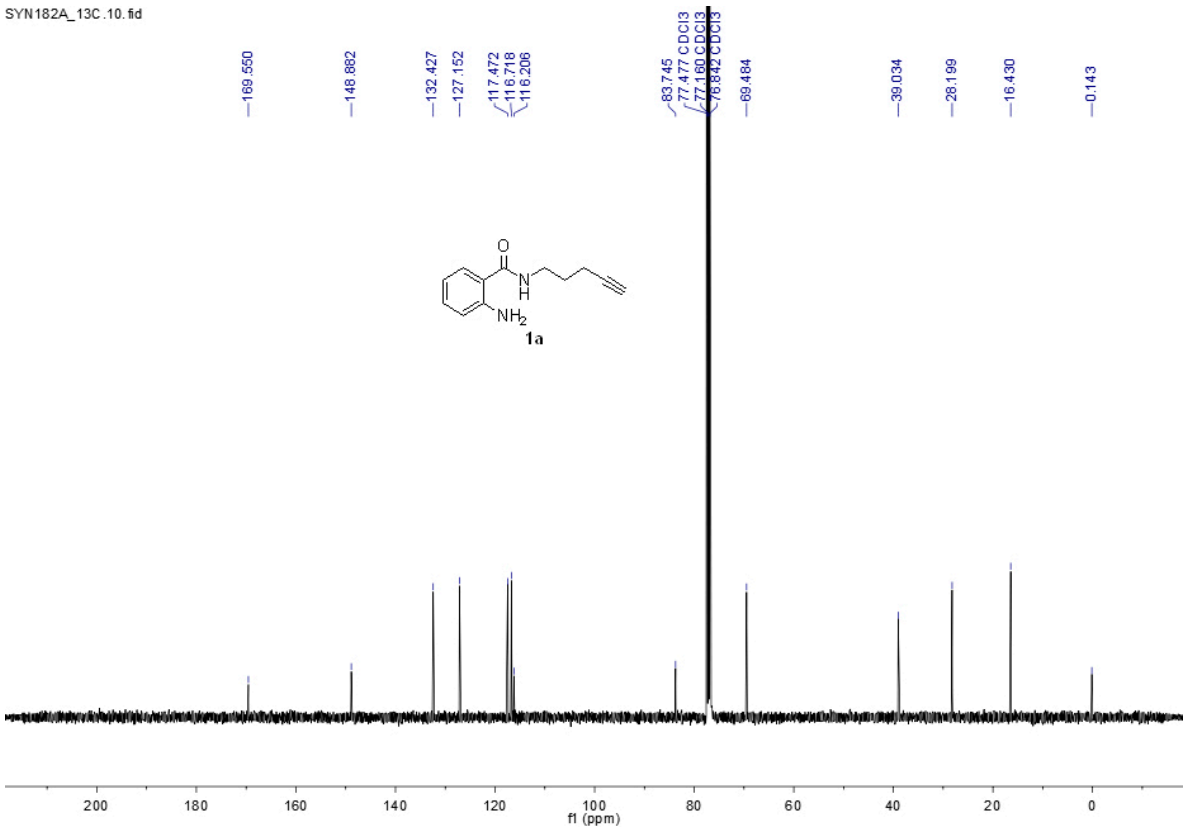
In a 25 mL oven-dried round-bottom flask with a side arm, *N*-methylisatoic (446 mg, 2.52 mmol, 1.0 equiv.) and (2-ethynylphenyl)methanamine (**S1d**) (330 mg, 2.52 mmol) were dissolved in anhydrous toluene (2.0 mL). The reaction mixture was stirred at 100°C for 1 h. Upon completion of the reaction, the reaction mixture was concentrated *in vacuo* and purified by MPLC (10:1 hexanes/EtOAc) to afford **1u** (426 mg, 1.61 mmol, 64%) as a white solid.

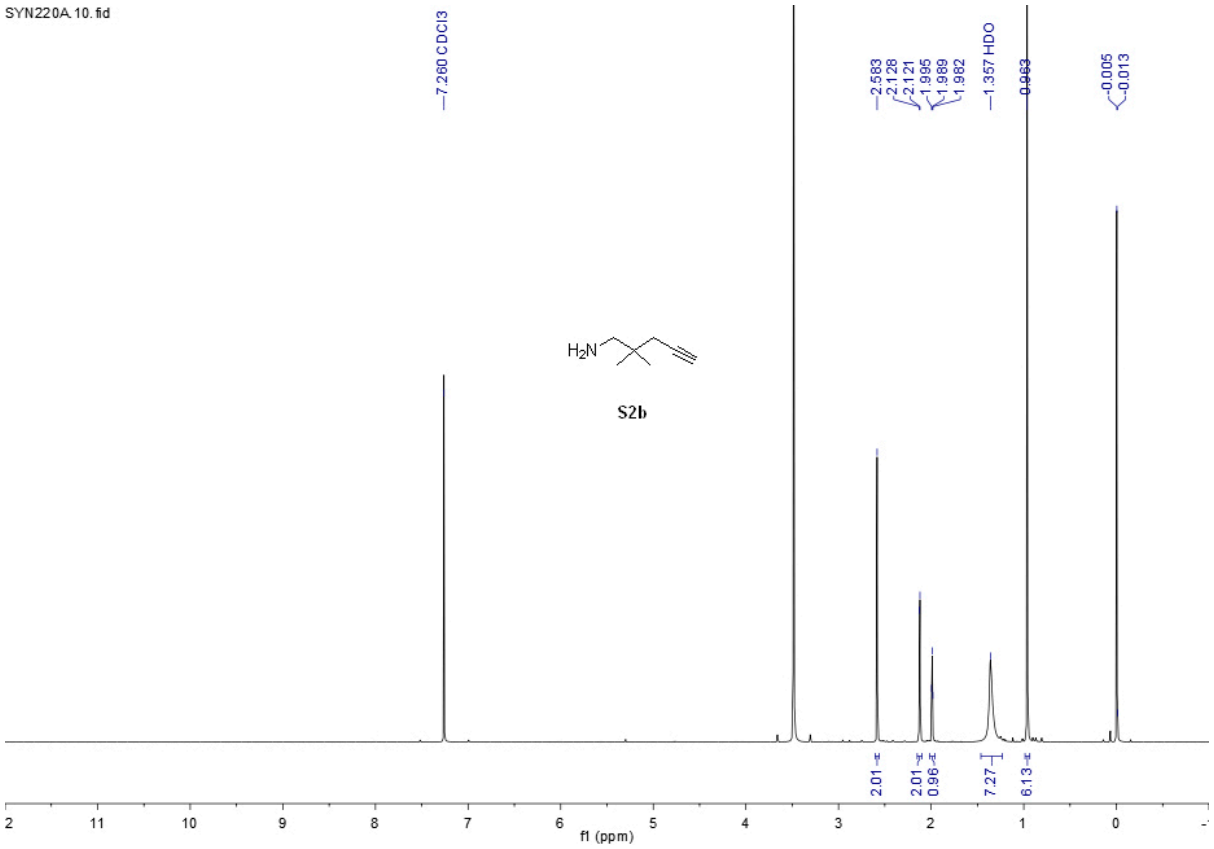
TLC: R_f 0.20 (5:1 hexanes/EtOAc). **mp**: 112.0–113.7 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.42 (ddd, *J* = 7.6, 1.4, 0.6 Hz, 1H), 7.34 (dtd, *J* = 8.6, 4.7, 1.6 Hz, 2H), 7.31–7.23 (m, 2H), 6.66 (d, *J* = 7.8 Hz, 1H), 6.57 (td, *J* = 7.6, 1.1 Hz, 1H), 4.74 (d, *J* = 5.9 Hz, 2H), 3.37 (s, 1H), 2.86 (d, *J* = 5.1 Hz, 2H), 1.56 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.7, 150.7, 140.8, 133.1, 133.0, 129.3, 128.5, 127.4, 127.3, 121.2, 114.8, 114.5, 111.1, 82.2, 81.7, 42.3, 29.7. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{17}N_2O$ $[M + H]^+$ 265.1335, found: 265.1340.

3. ^{1}H and ^{13}C NMR spectra

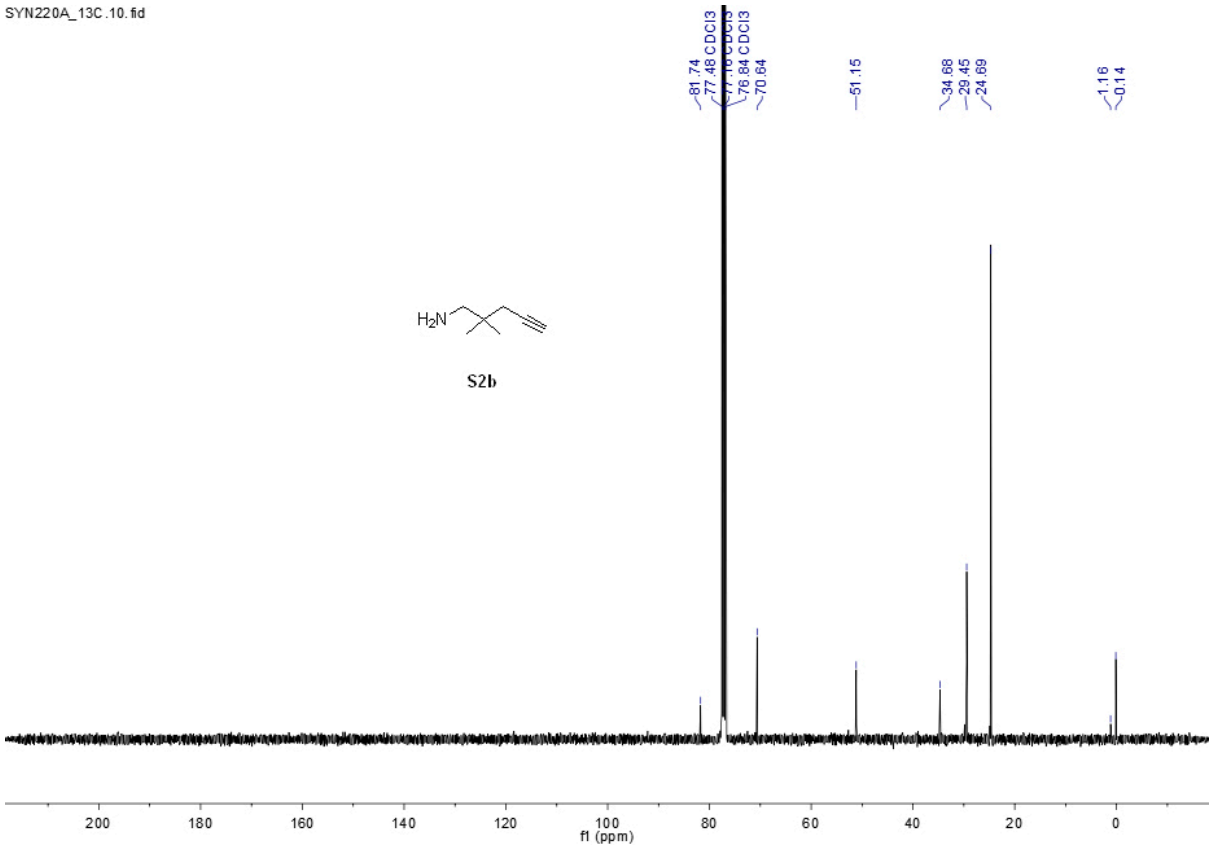
3.1. Spectra of Substrates



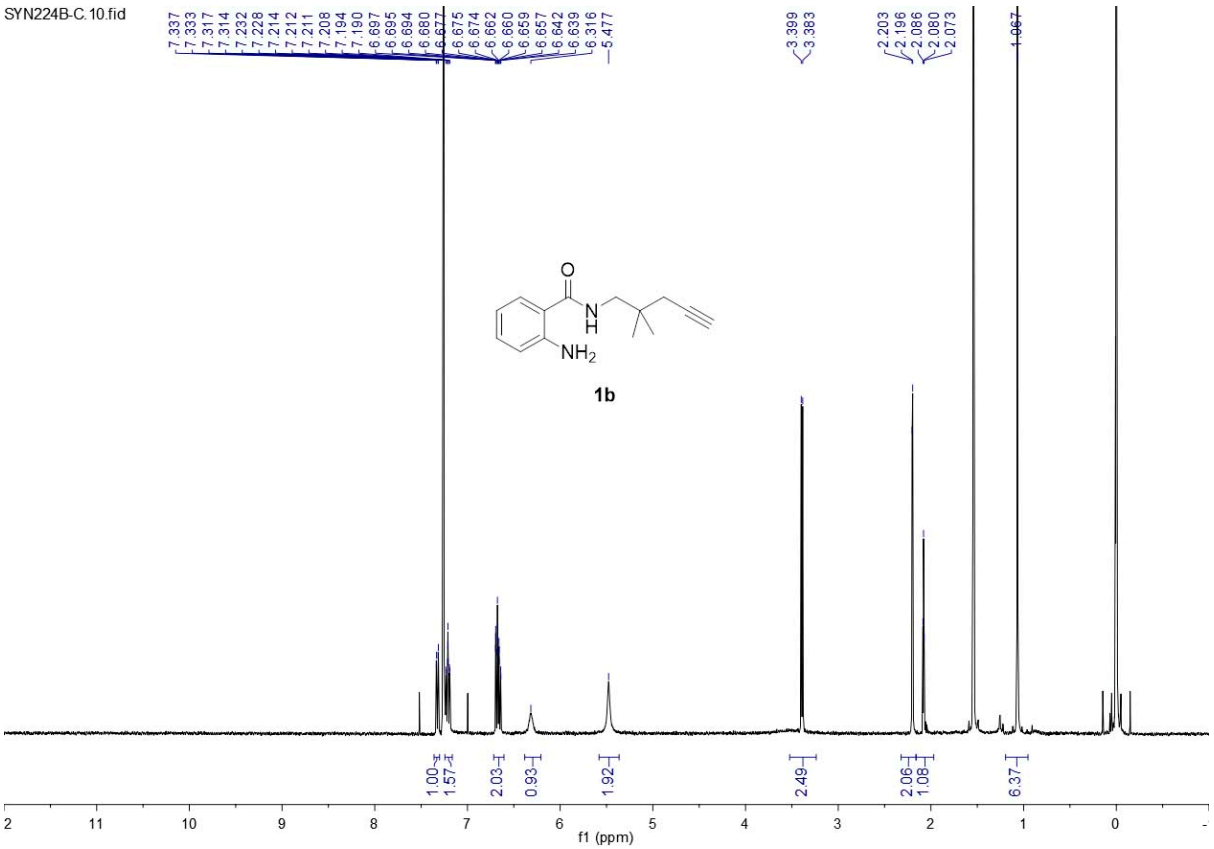



SYN220A.10.fid
7.260 CDCl3
2.583
2.128
2.121
1.995
1.989
1.982
1.357 HDO
0.963
0.005
0.013
2.01
2.01
0.96
7.27
6.13
f1 (ppm)

S2b


SYN220A_13C.10.fid
81.74
77.48 CDCl3
76.84 CDCl3
70.64
51.15
34.68
29.45
24.69
1.16
0.14
f1 (ppm)

S2b


SYN224B-C.10.fid
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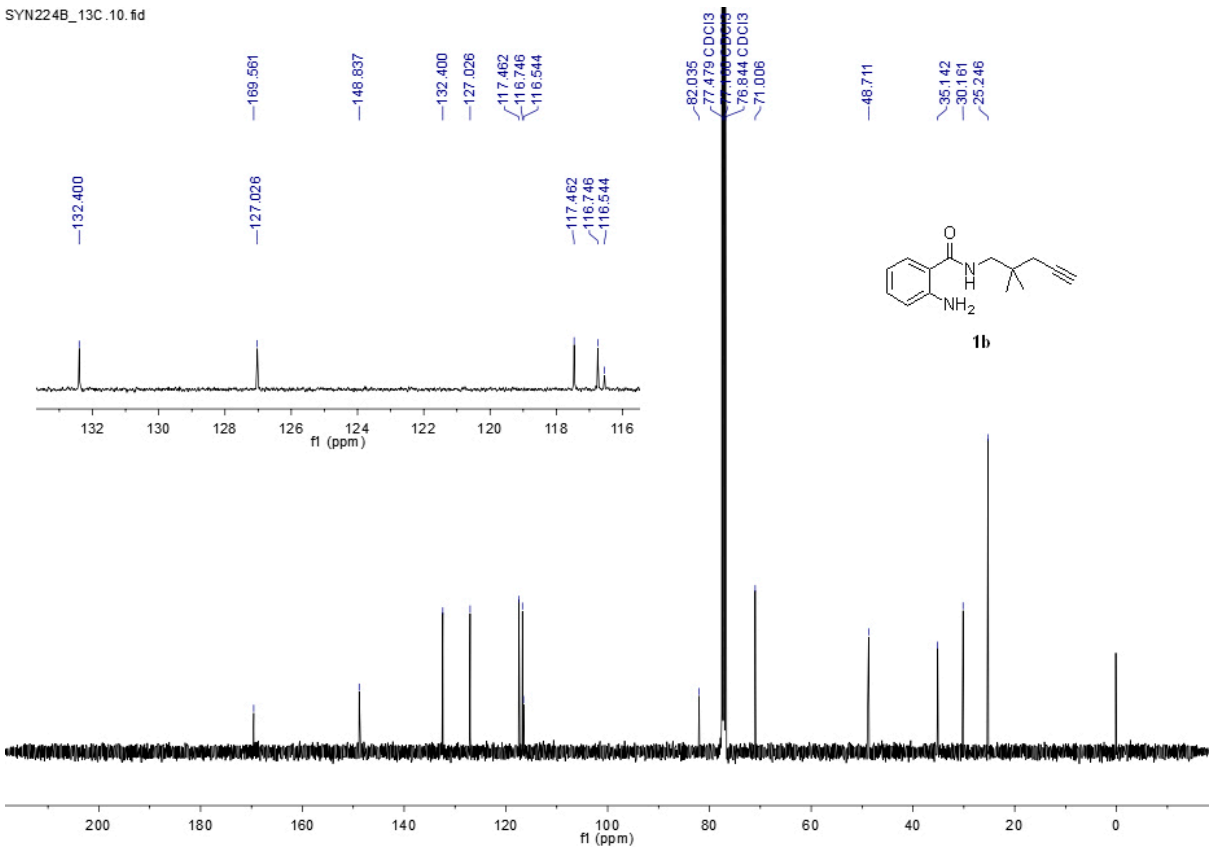
7.333
7.317
7.314
7.232
7.228
7.214
7.212
7.211
7.208
7.194
7.190
6.697
6.695
6.694
6.680
6.677
6.675
6.674
6.662
6.660
6.659
6.657
6.642
6.639
6.316
5.477
3.399
3.383
2.203
2.196
2.086
2.080
2.073
1.067
1.00
1.57
2.03
0.93
1.92
2.49
2.06
1.08
6.37
f1 (ppm)
1b


SYN224B_13C.10.fid
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148.837
132.400
127.026
117.462
116.746
116.544
82.035
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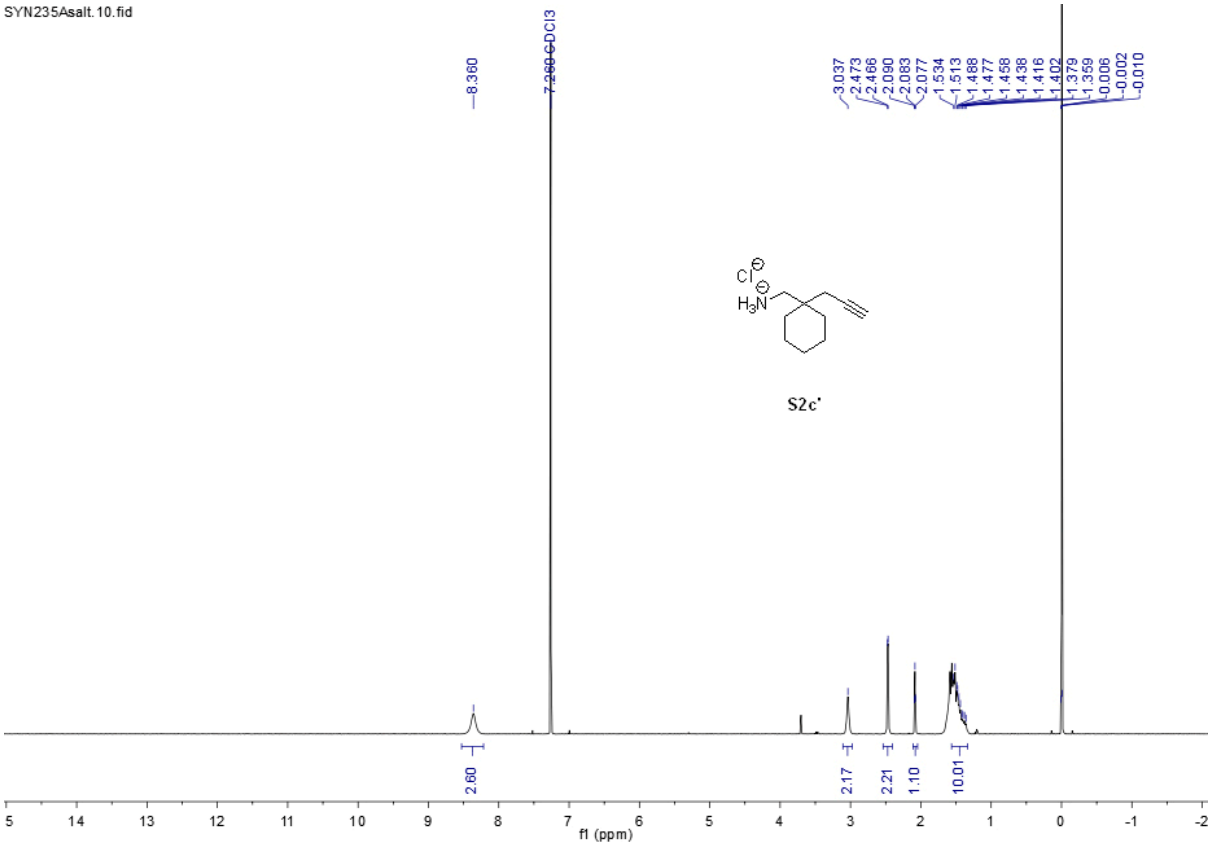
76.844 CDCl3
71.006
48.711
35.142
30.161
25.246
f1 (ppm)
1b


SYN235Asalt.10.fid
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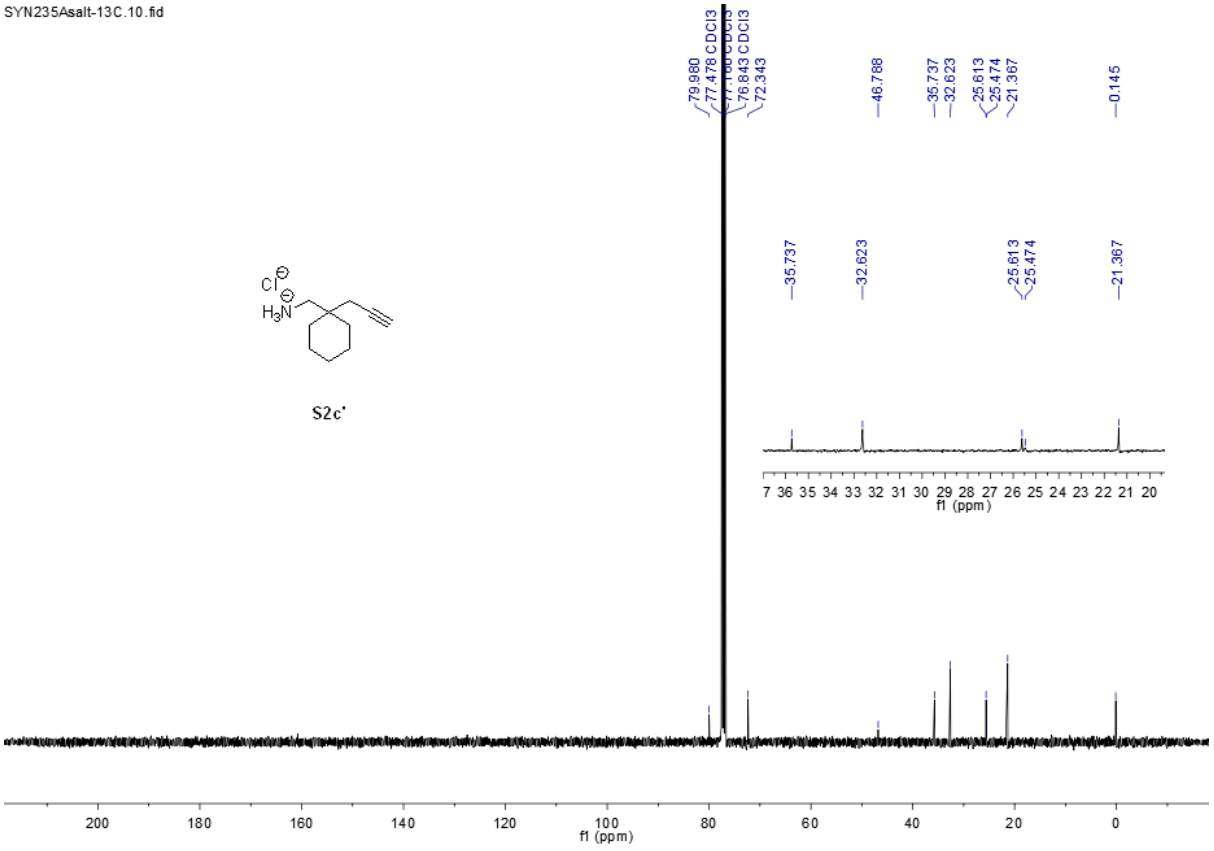
7.260 CDCl3
3.037
2.473
2.466
2.090
2.083
2.077
1.534
1.513
1.488
1.477
1.458
1.438
1.416
1.402
1.379
1.359
0.006
-0.002
-0.010
S2c'
2.60
2.17
2.21
1.10
10.01
f1 (ppm)


SYN235Asalt-13C.10.fid
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77.478 CDCl3
76.843 CDCl3
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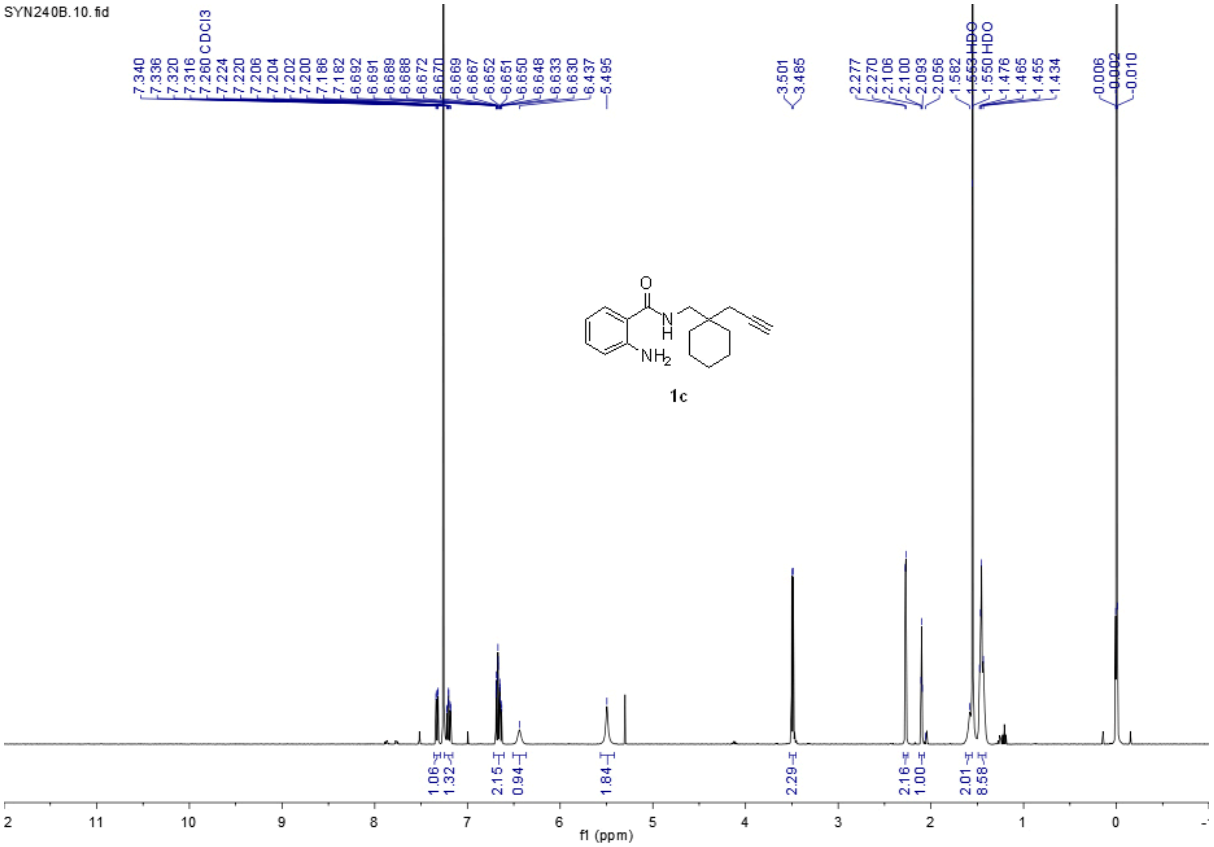
46.788
35.737
32.623
25.613
25.474
21.367
0.145
S2c'
f1 (ppm)


SYN240B.10.fid
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7.320
7.316
7.260 CDCl3
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7.220
7.206
7.204
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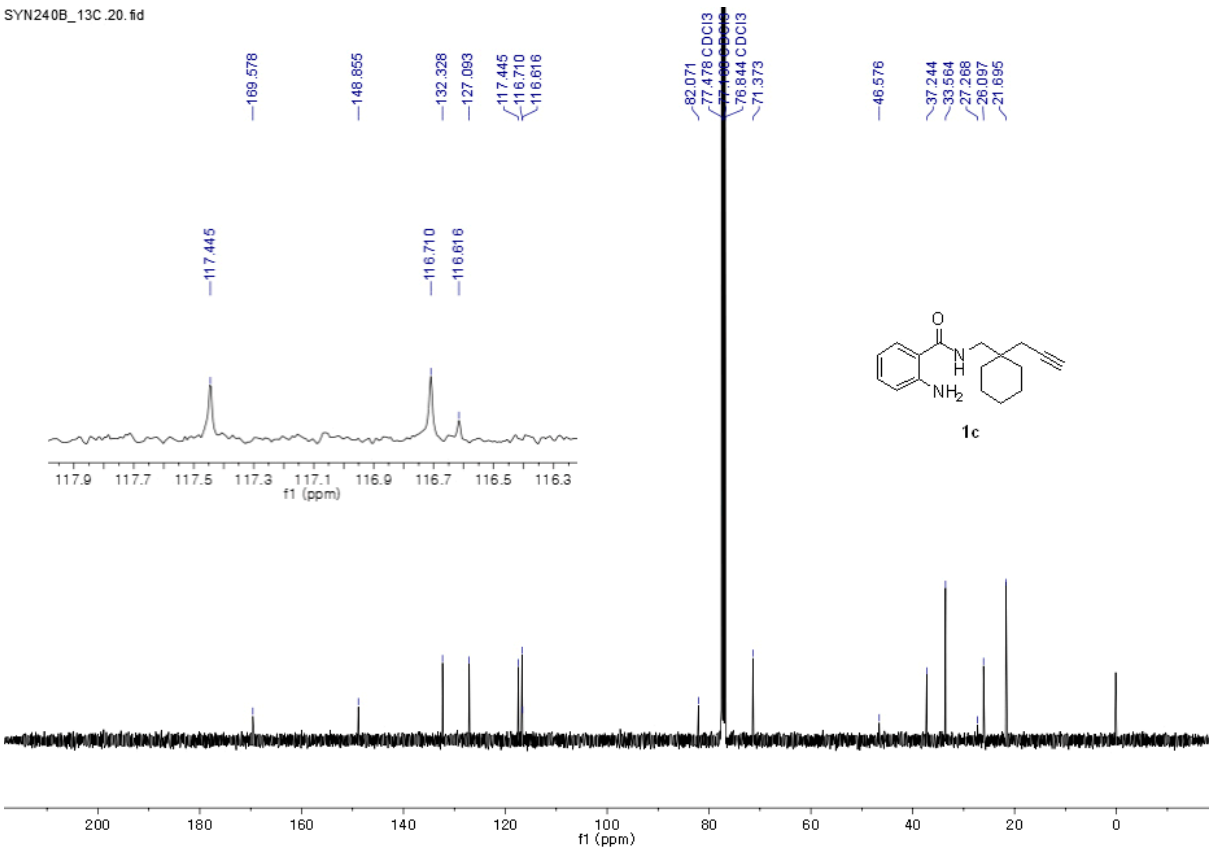
7.186
7.182
6.692
6.691
6.689
6.688
6.672
6.670
6.669
6.667
6.652
6.651
6.650
6.648
6.633
6.630
6.437
5.495
3.501
3.485
2.277
2.270
2.106
2.100
2.093
2.056
1.582
1.563 H2O
1.550 HDO
1.476
1.465
1.455
1.434
0.006
0.002
-0.010
1.06
1.32
2.15
0.94
1.84
2.29
2.16
1.00
2.01
8.58
f1 (ppm)
1c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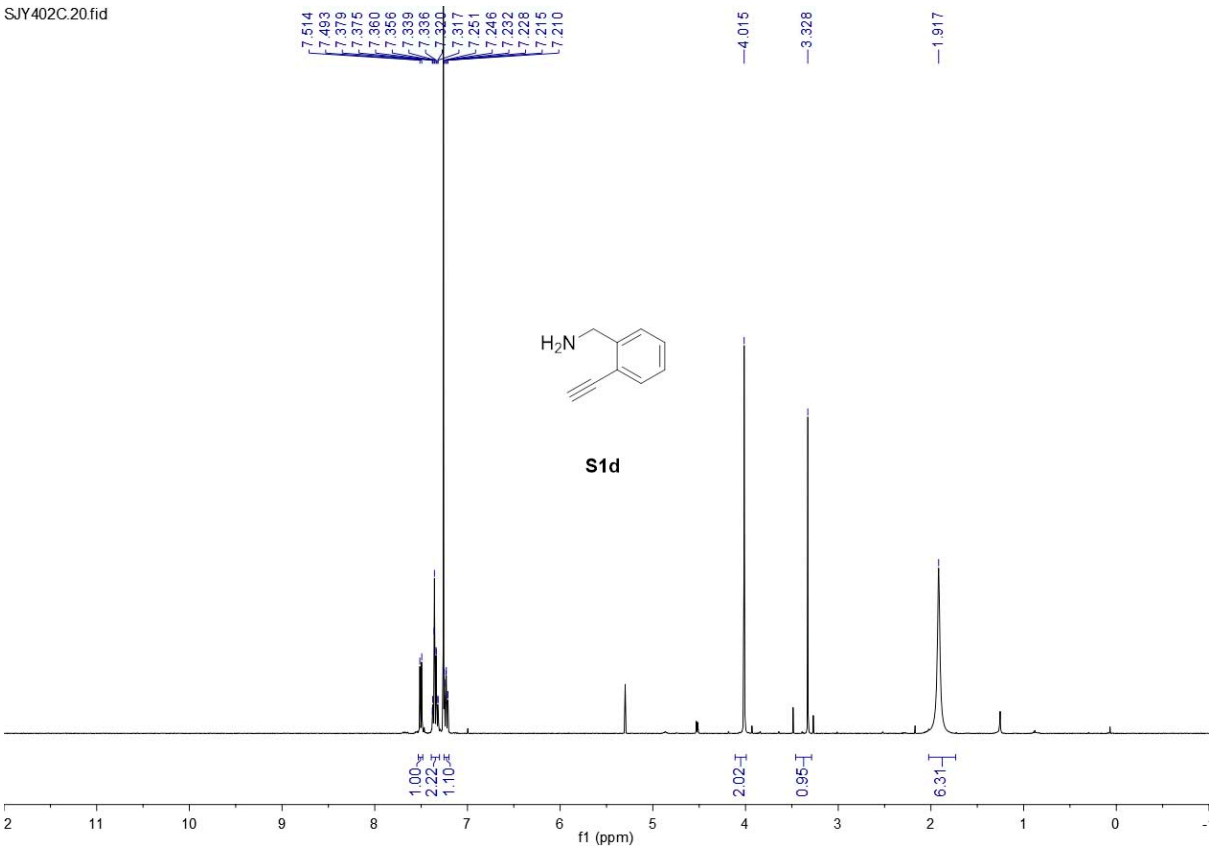
148.855
132.328
127.093
117.445
116.710
116.616
82.071
77.478 CDCl3
76.844 CDCl3
71.373
46.576
37.244
33.564
27.268
26.097
21.695
117.445
116.710
116.616
117.9
117.7
117.5
117.3
117.1
116.9
116.7
116.5
116.3
f1 (ppm)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
1c


SJY402C.20.fid
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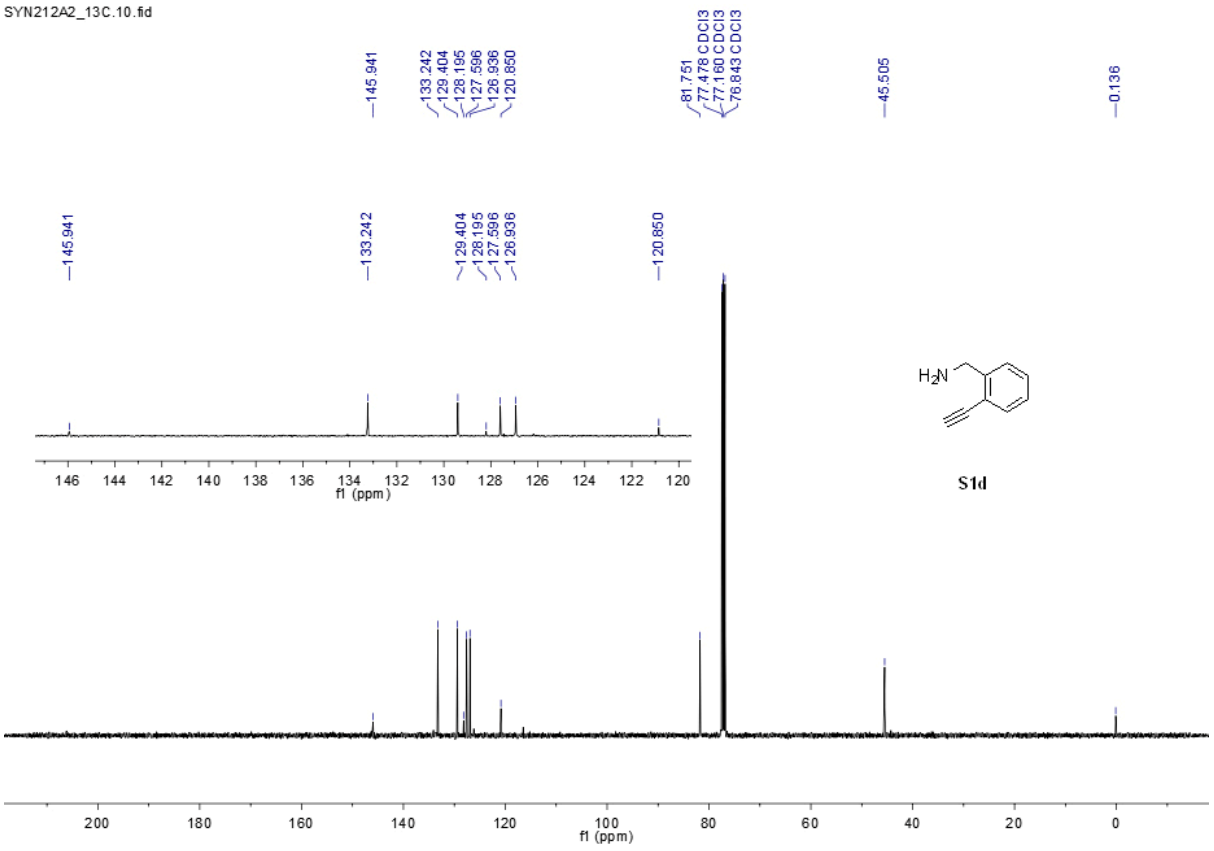
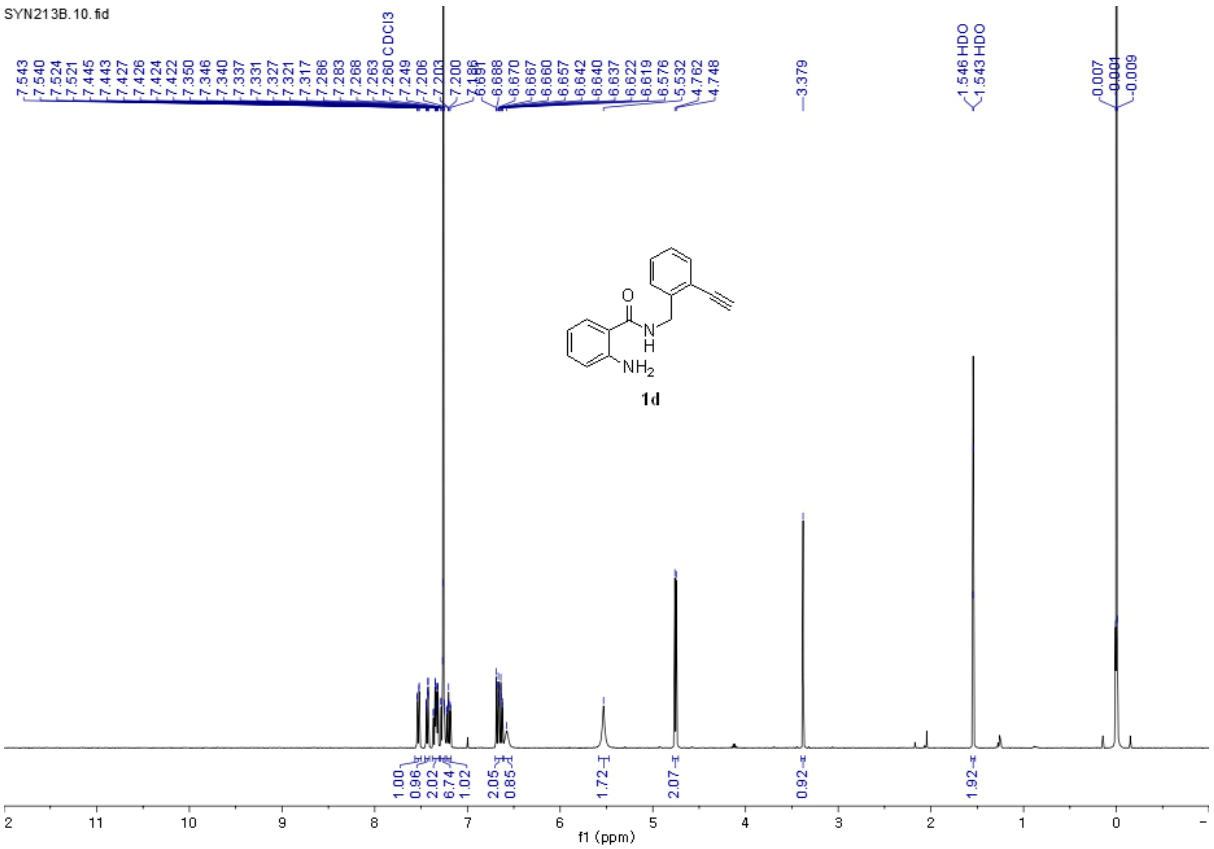
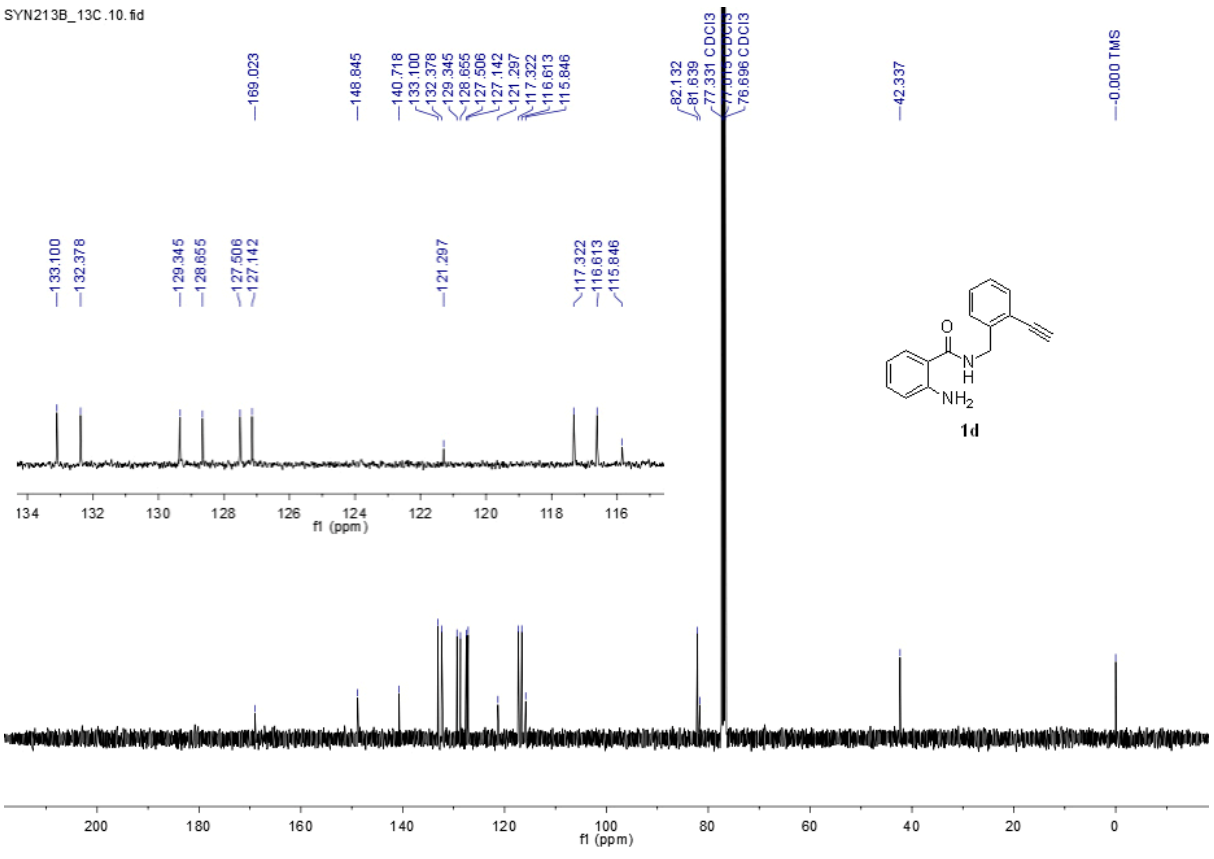
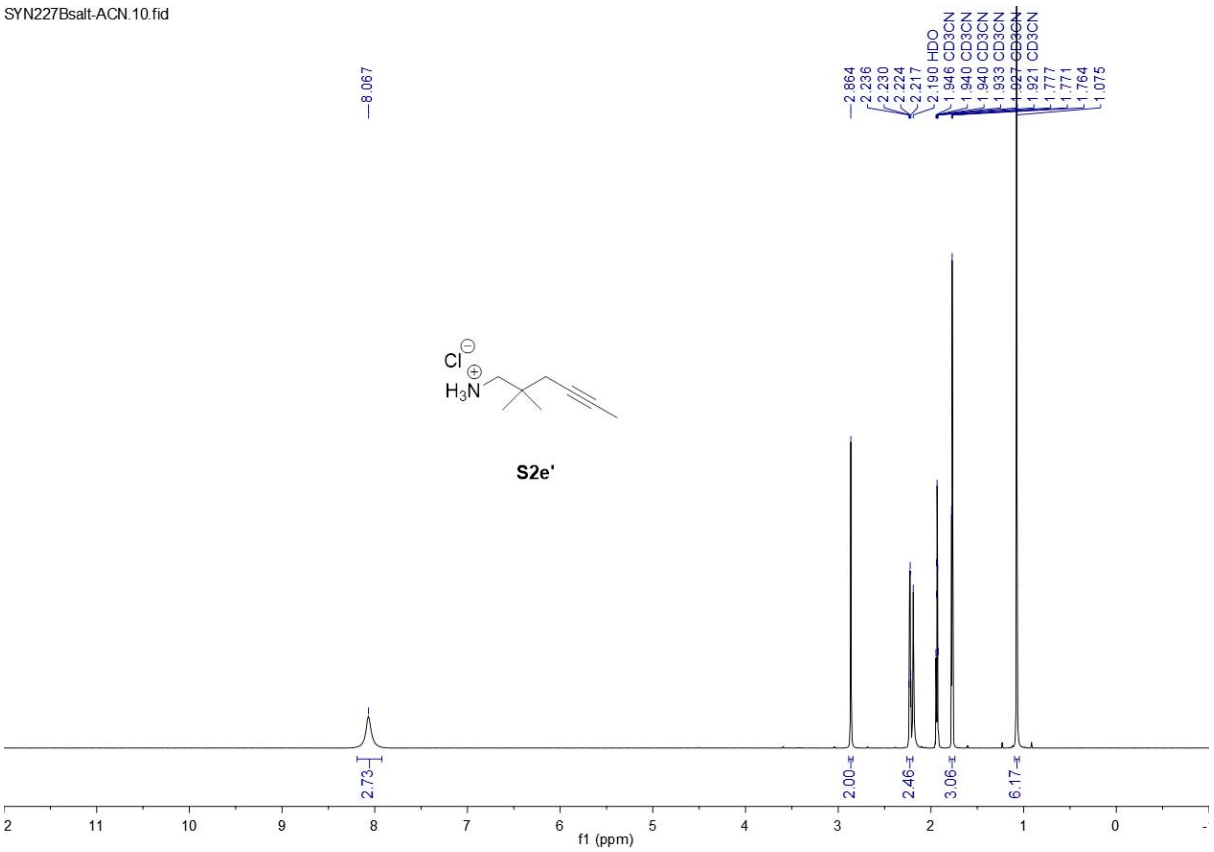
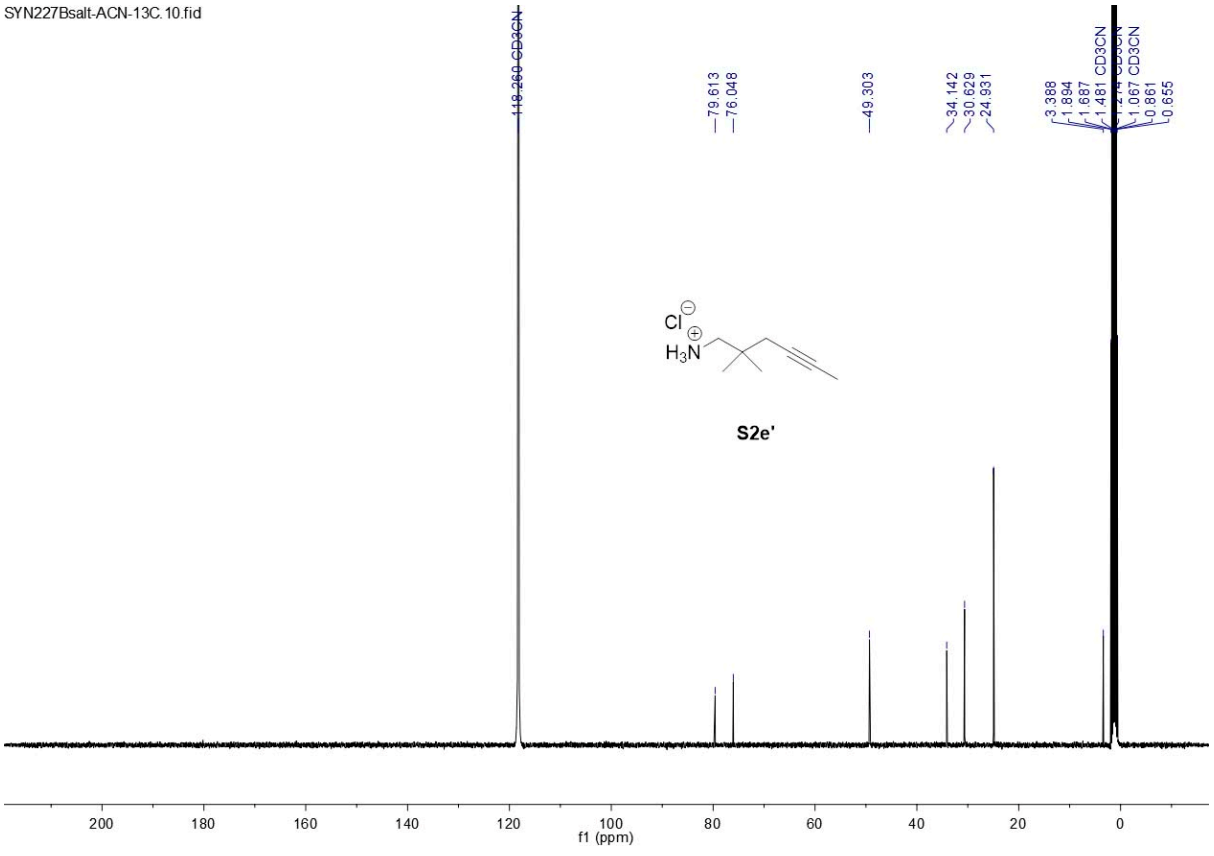
7.493
7.379
7.375
7.360
7.356
7.339
7.336
7.320
7.317
7.251
7.246
7.232
7.228
7.215
7.210
4.015
3.328
1.917
1.00
2.22
1.10
2.02
0.95
6.31
f1 (ppm)
S1d

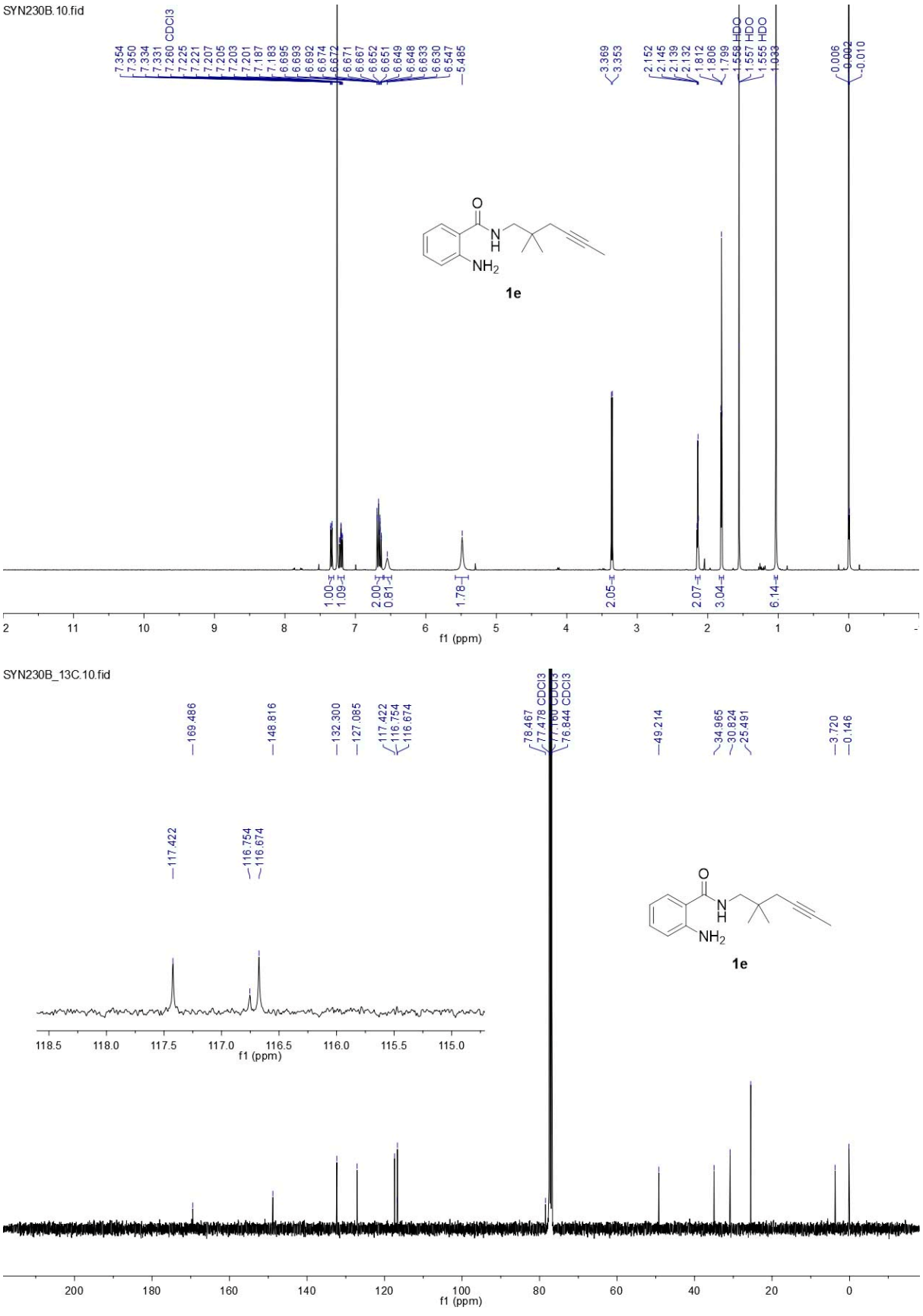

SYN212A2_13C.10.fid
145.941
133.242
129.404
128.195
127.596
126.936
120.850
81.751
77.478 CDCl3
77.160 CDCl3
76.843 CDCl3
45.505
0.136
f1 (ppm)
S1d

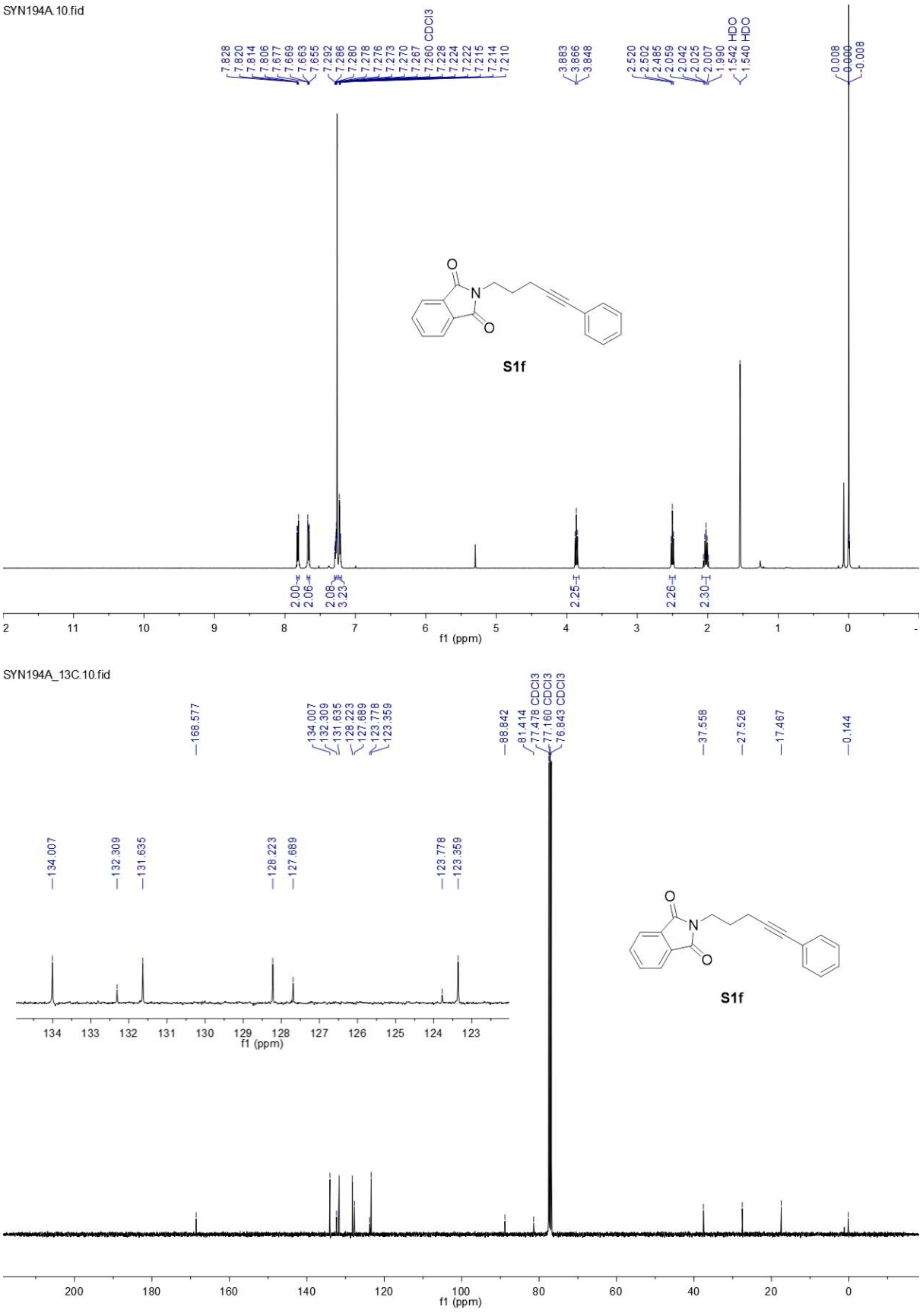

SYN213B.10.fid
7.543
7.540
7.524
7.521
7.445
7.443
7.427
7.426
7.424
7.422
7.350
7.346
7.340
7.337
7.331
7.327
7.321
7.317
7.286
7.283
7.268
7.263
7.260 CDCl3
7.249
7.206
7.203
7.200
6.688
6.670
6.667
6.660
6.657
6.642
6.640
6.637
6.622
6.619
6.576
5.532
4.762
4.748
3.379
1.546 HDO
1.543 HDO
0.007
0.001
-0.009
1.00
0.98
2.02
6.74
1.02
2.05
0.85
1.72
2.07
0.92
1.92
f1 (ppm)
O
N
H
NH2
1d

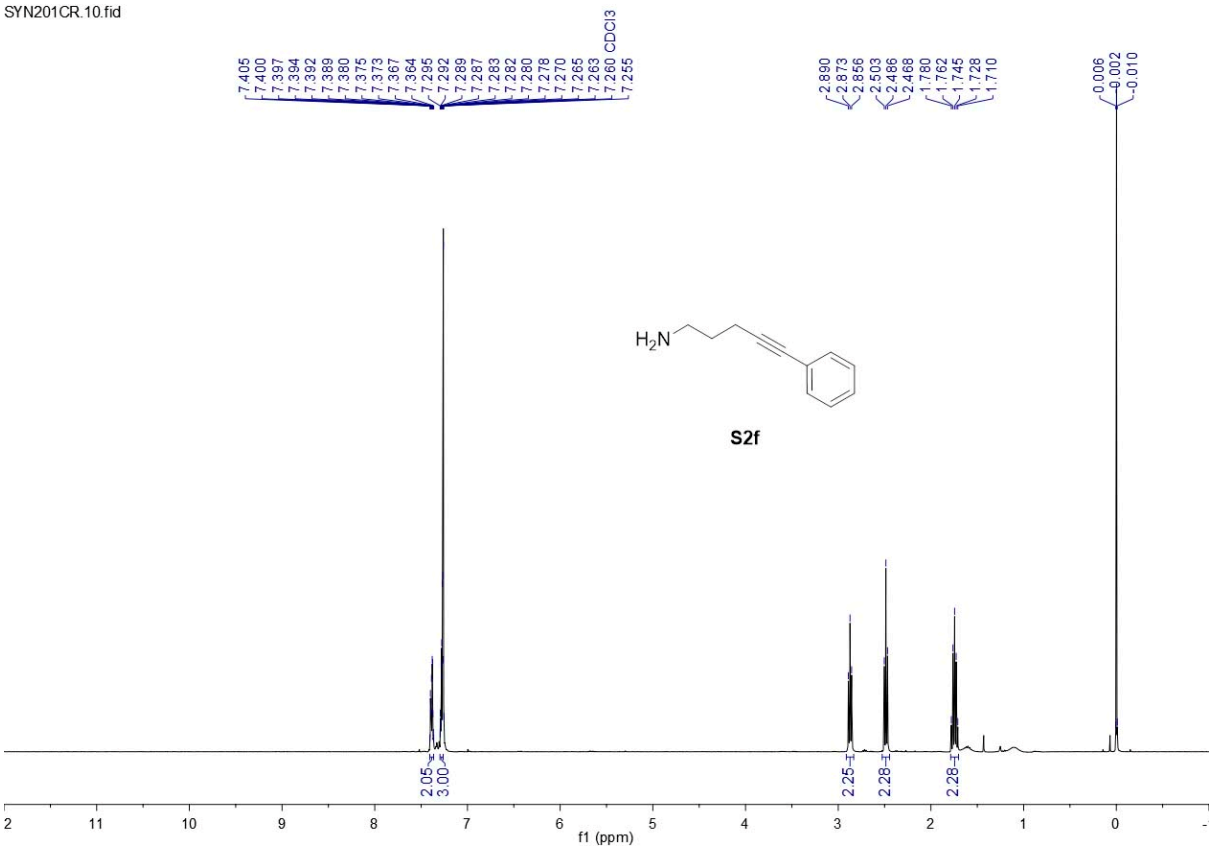

SYN213B_13C.10.fid
169.023
148.845
140.718
133.100
132.378
129.345
128.655
127.506
127.142
121.297
117.322
116.613
115.846
82.132
81.639
77.331 CDCl3
76.696 CDCl3
42.337
0.000 TMS
f1 (ppm)
O
N
H
NH2
1d

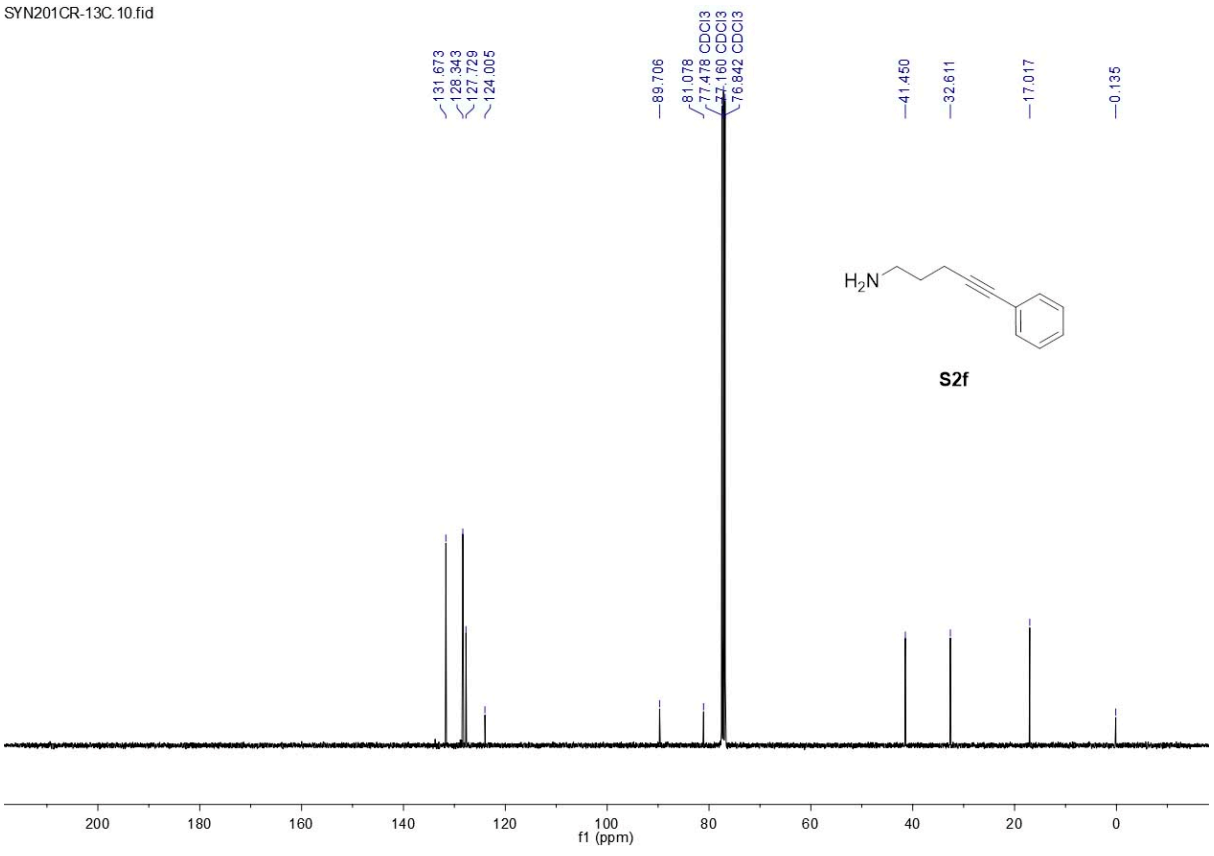

SYN227Bsalt-ACN.10.fid
8.067
2.864
2.236
2.230
2.224
2.217
2.190 HDO
1.946 CD3CN
1.940 CD3CN
1.940 CD3CN
1.933 CD3CN
1.927 CD3CN
1.921 CD3CN
1.777
1.771
1.764
1.075
Cl
H3N
S2e'
2.73
2.00
2.46
3.06
6.17
f1 (ppm)

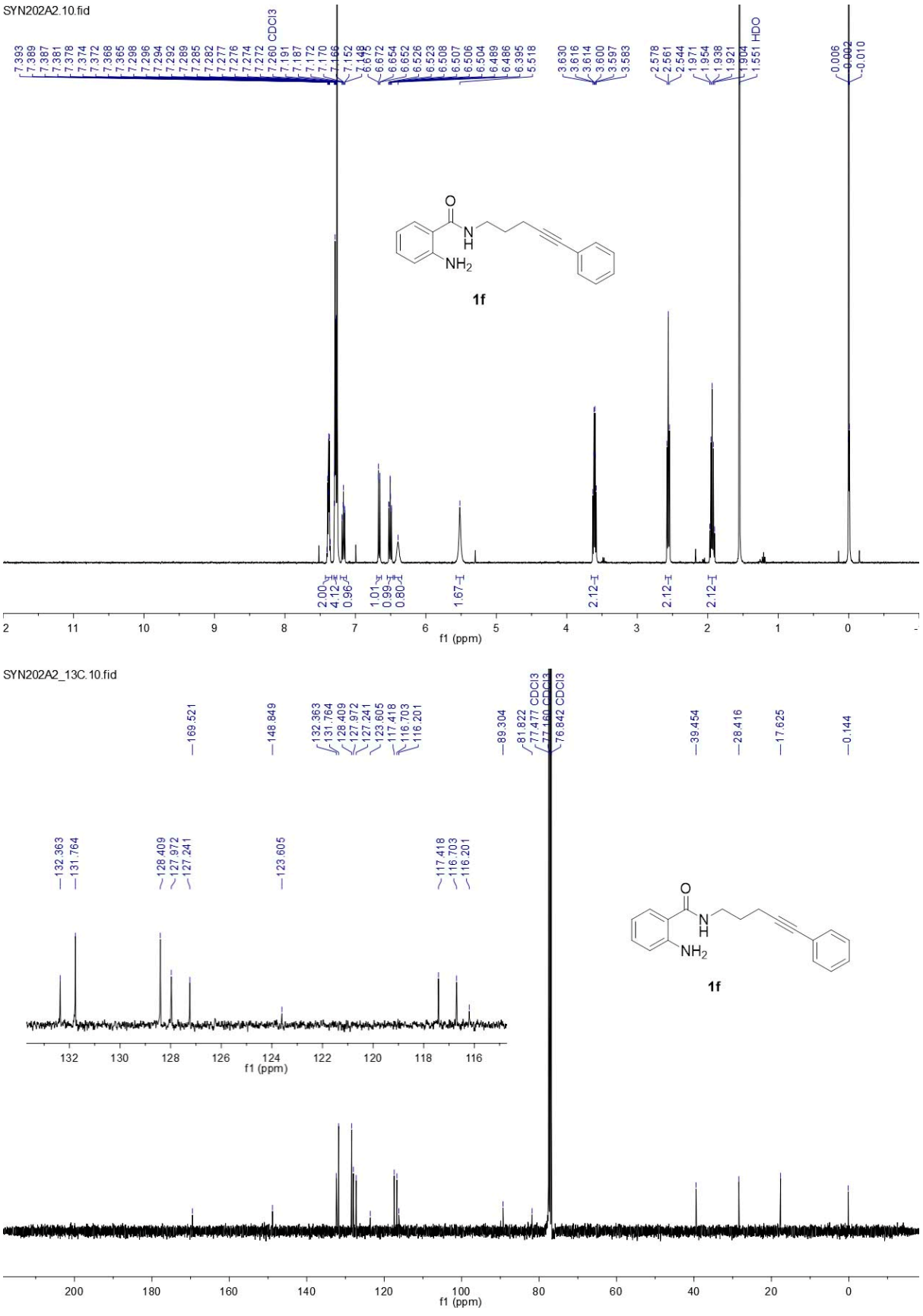

SYN227Bsalt-ACN-13C.10.fid
118.260 CD3CN
79.613
76.048
49.303
34.142
30.629
24.931
3.388
1.894
1.687
1.481 CD3CN
1.067 CD3CN
0.861
0.655
Cl
H3N
S2e'
f1 (ppm)

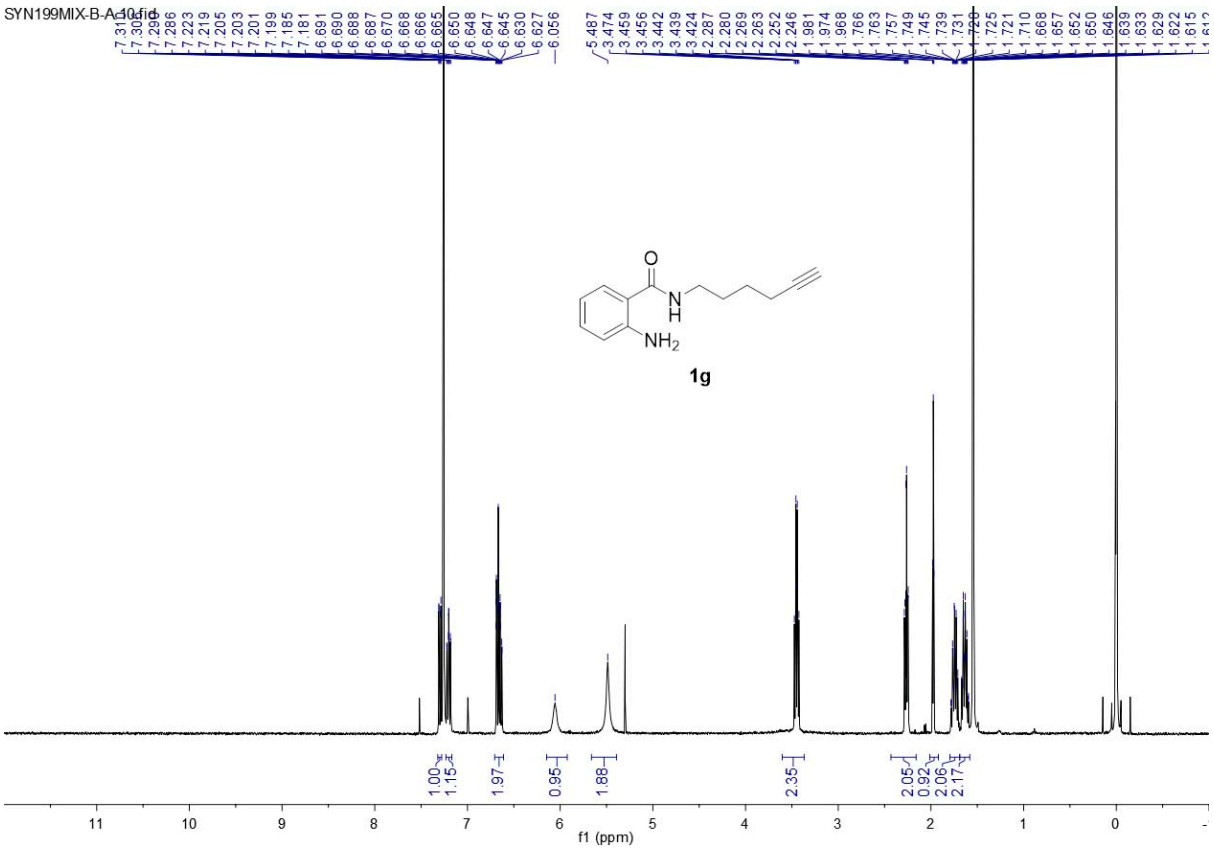

SYN230B.10.fid
7.354
7.350
7.334
7.331
7.260 CDCl3
7.225
7.221
7.207
7.205
7.203
7.201
7.187
7.183
6.695
6.693
6.692
6.674
6.672
6.671
6.667
6.652
6.651
6.649
6.648
6.633
6.630
6.547
5.485
3.369
3.353
2.152
2.145
2.139
2.132
1.812
1.806
1.799
1.558 HDO
1.557 HDO
1.555 HDO
1.003
0.006
0.002
-0.010
1.00
1.09
2.00
0.81
1.78
2.05
2.07
3.04
6.14
f1 (ppm)
1e
SYN230B_13C.10.fid
169.486
148.816
132.300
127.085
117.422
116.754
116.674
78.467
77.478 CDCl3
77.160 CDCl3
76.844 CDCl3
49.214
34.965
30.824
25.491
3.720
0.146
f1 (ppm)
1e

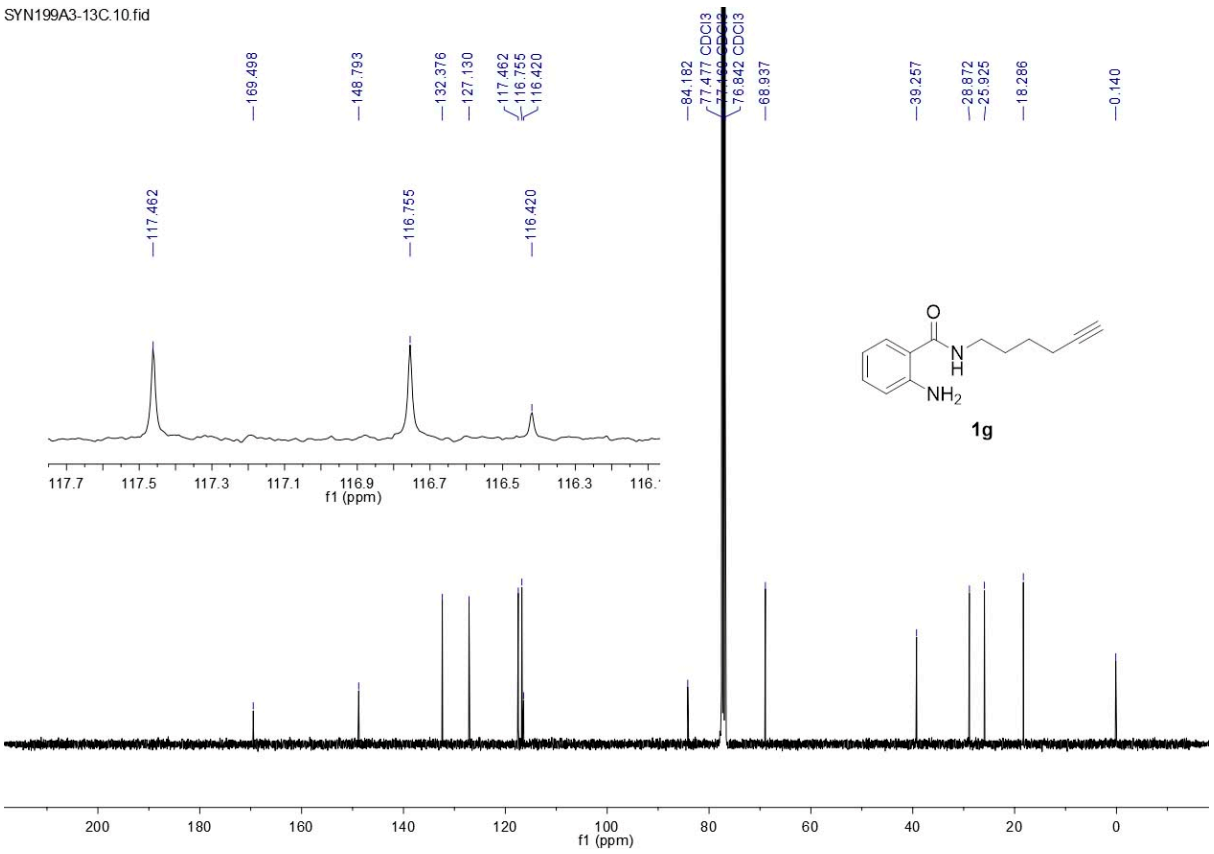

SYN194A.10.fid
7.828
7.820
7.814
7.806
7.677
7.669
7.663
7.655
7.292
7.286
7.280
7.278
7.276
7.273
7.270
7.267
7.260 CDCl3
7.228
7.224
7.222
7.215
7.214
7.210
3.883
3.866
3.848
2.520
2.502
2.485
2.059
2.042
2.025
2.007
1.990
1.542 HDO
1.540 HDO
0.008
0.000
-0.008
2.00
2.06
2.08
3.23
2.25
2.26
2.30
f1 (ppm)
S1f
SYN194A_13C.10.fid
168.577
134.007
132.309
131.635
128.223
127.689
123.778
123.359
88.842
81.414
77.478 CDCl3
77.160 CDCl3
76.843 CDCl3
37.558
27.526
17.467
0.144
f1 (ppm)
S1f

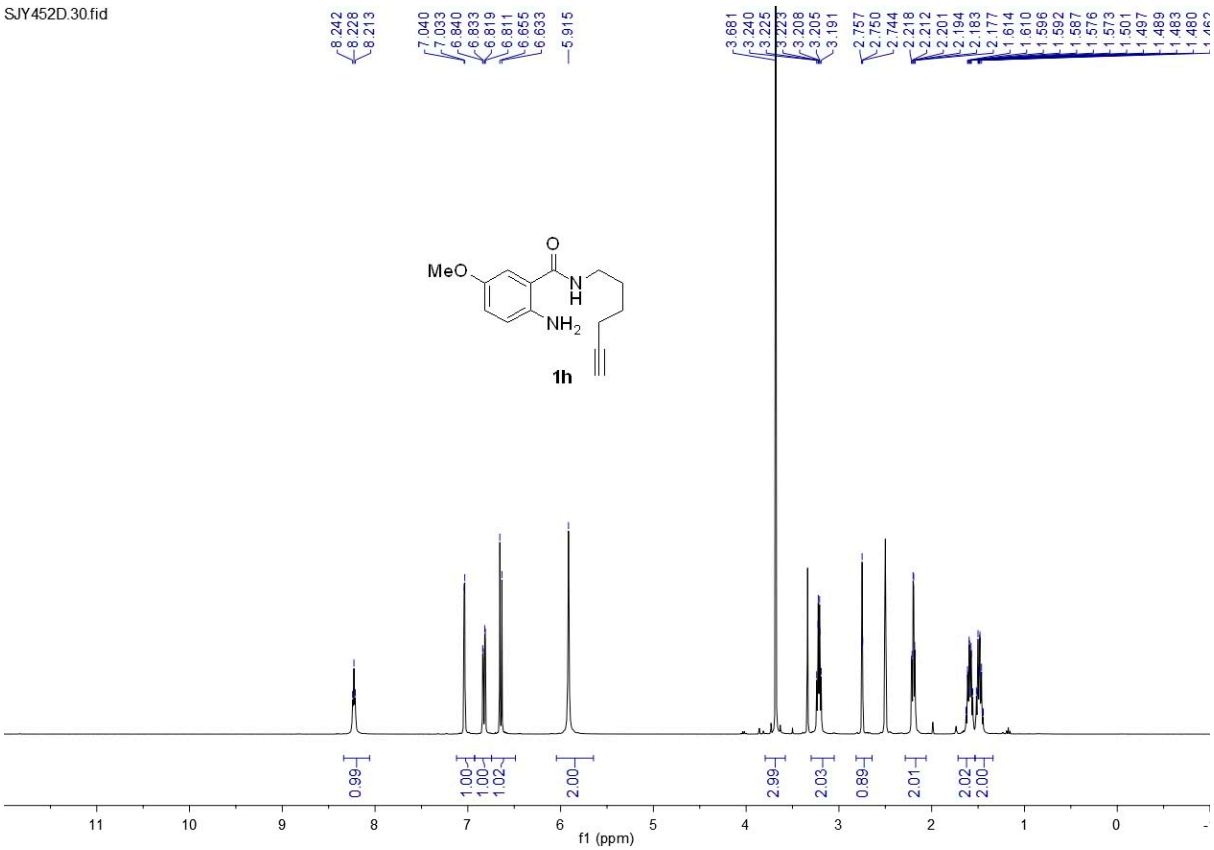

SYN201CR.10.fid
7.405
7.400
7.397
7.394
7.392
7.389
7.380
7.375
7.373
7.367
7.364
7.295
7.292
7.289
7.287
7.283
7.282
7.280
7.278
7.270
7.265
7.263
7.260 CDCl3
7.255
2.890
2.873
2.856
2.503
2.486
2.468
1.780
1.762
1.745
1.728
1.710
0.006
0.002
-0.010
2.05
3.00
2.25
2.28
2.28
f1 (ppm)
S2f

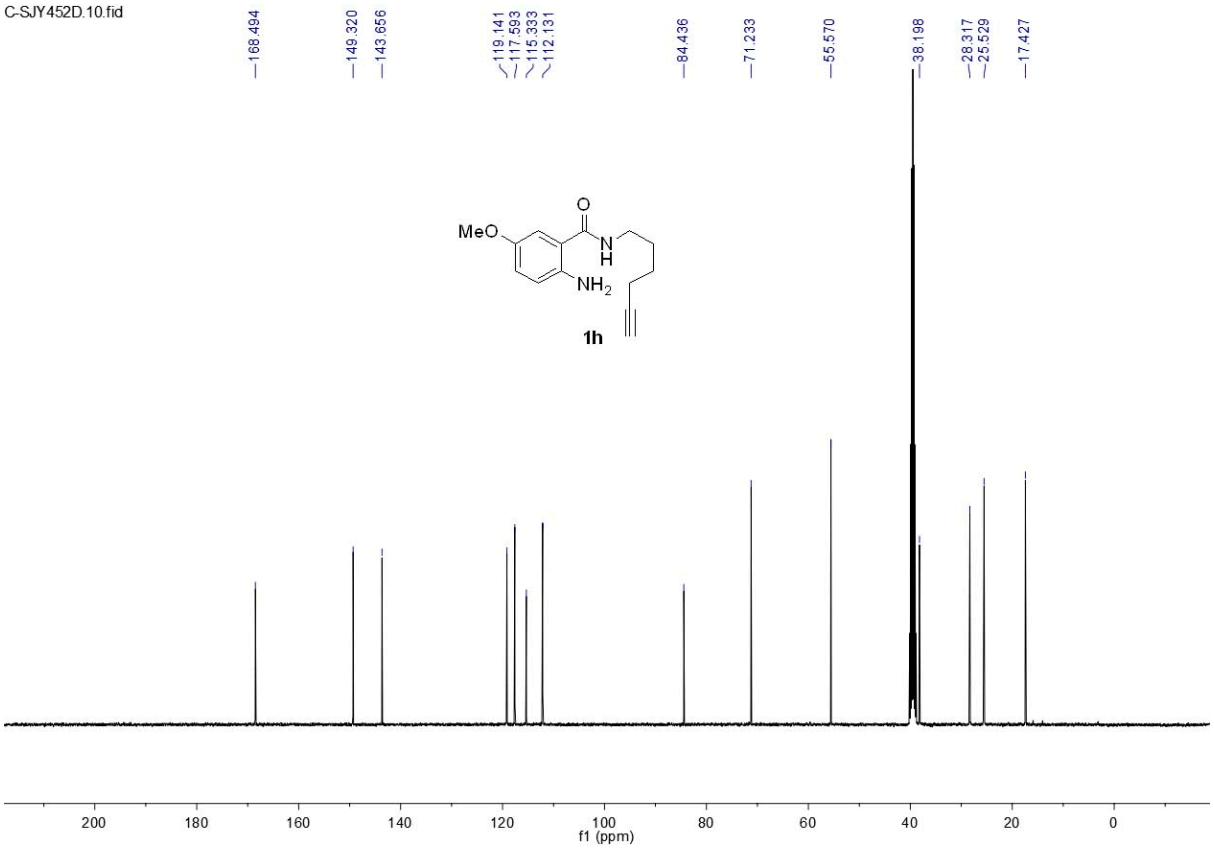

SYN201CR-13C.10.fid
131.673
128.343
127.729
124.005
89.706
81.078
77.478 CDCl3
77.160 CDCl3
76.842 CDCl3
41.450
32.611
17.017
0.135
f1 (ppm)
S2f

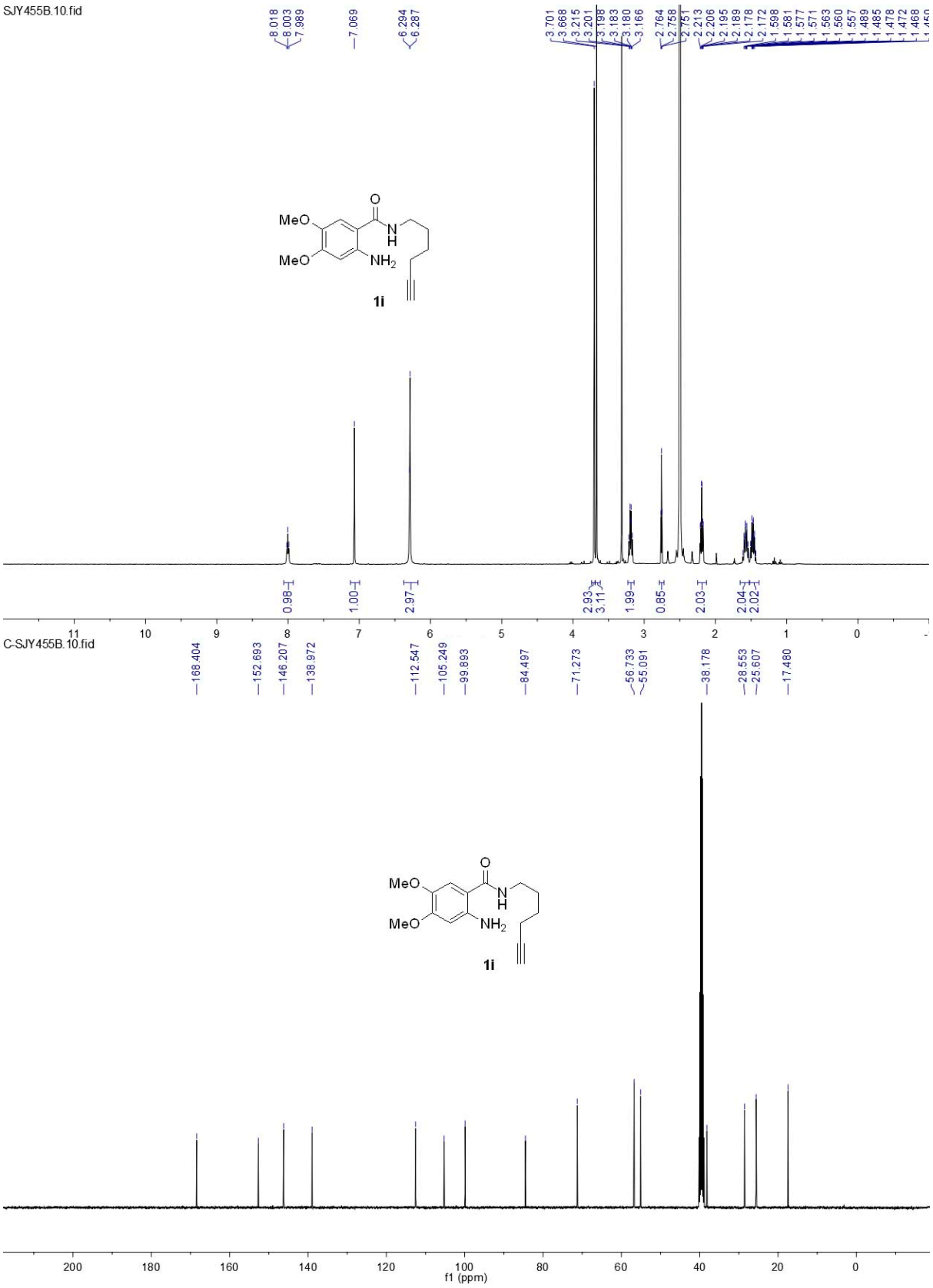

SYN202A2.10.fid
CDCl3
HDO
f1 (ppm)
1f
SYN202A2_13C.10.fid
CDCl3
f1 (ppm)
1f

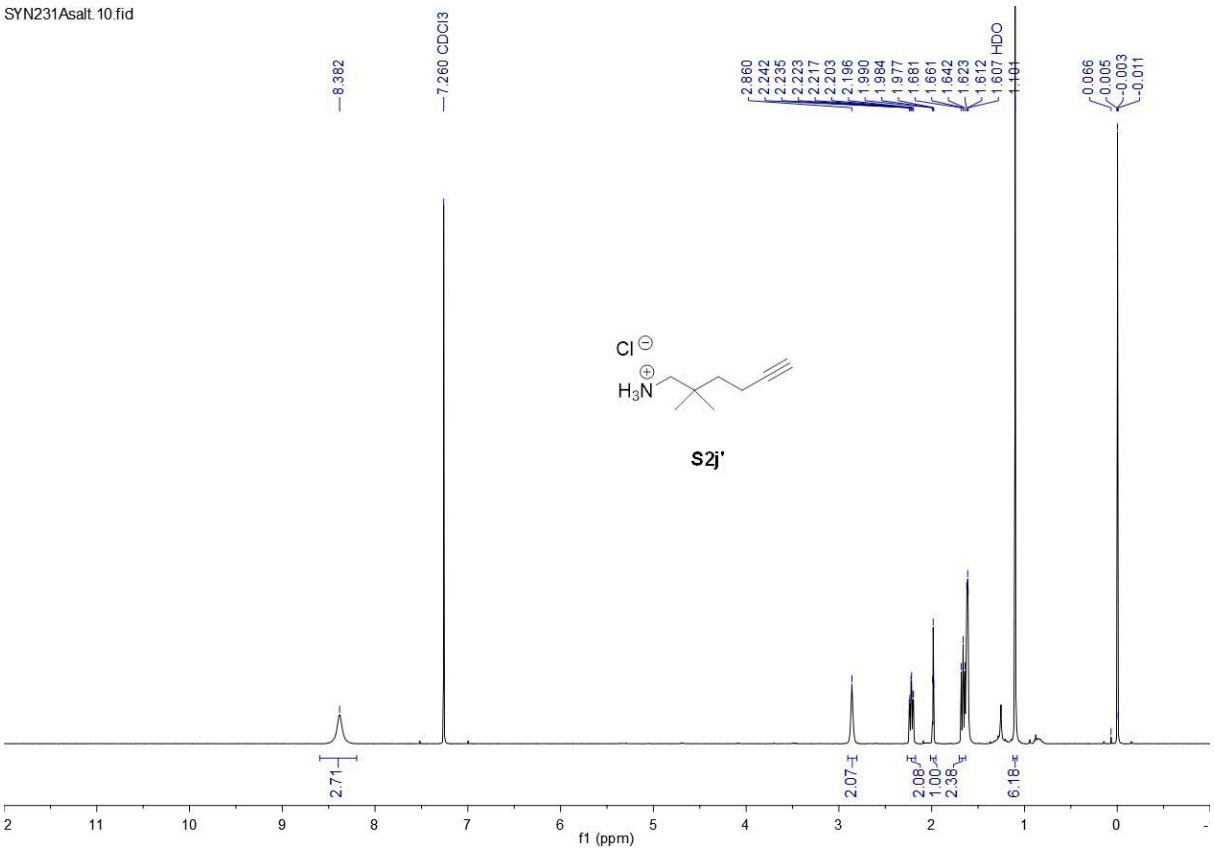

SYN199MIX-B-A.10.fid
1g
f1 (ppm)

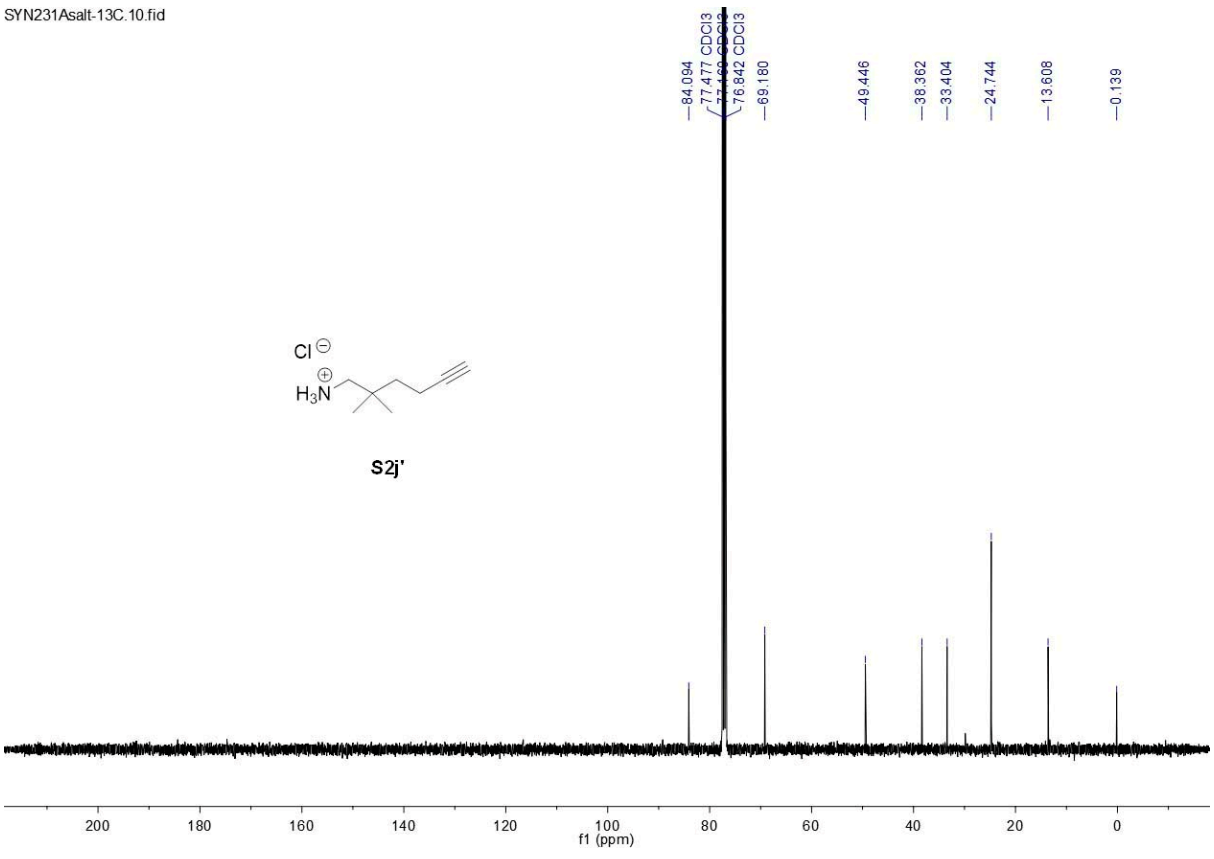

SYN199A3-13C.10.fid
117.462
116.755
116.420
1g
f1 (ppm)

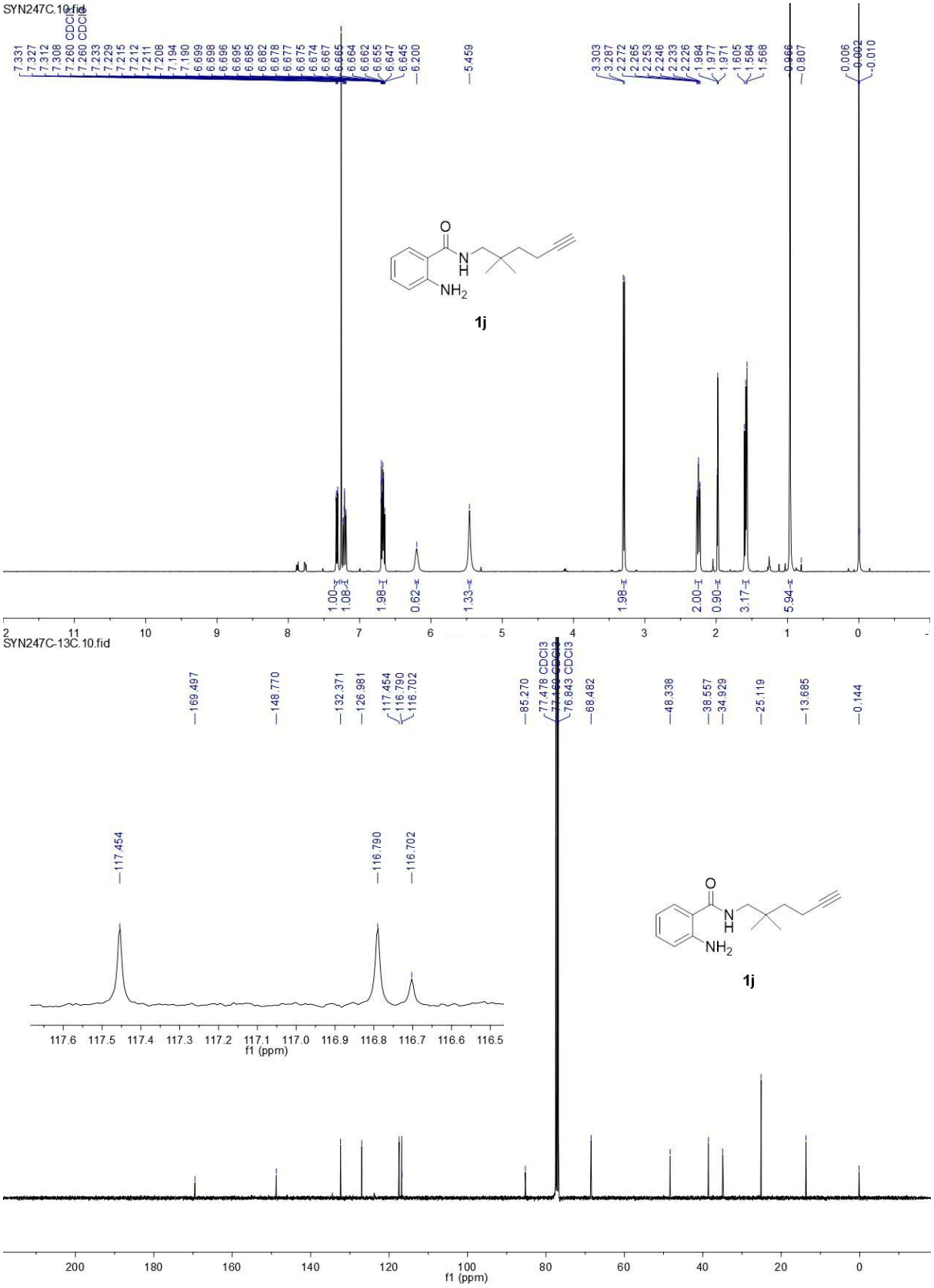

SJY452D.30.fid
8.242
8.228
8.213
7.040
7.033
6.840
6.833
6.819
6.811
6.655
6.633
5.915
3.681
3.240
3.225
3.223
3.208
3.205
3.191
2.757
2.750
2.744
2.218
2.212
2.201
2.194
2.183
2.177
1.614
1.610
1.596
1.592
1.587
1.576
1.573
1.501
1.497
1.489
1.483
1.480
MeO
O
N
H
NH2
1h
0.99
1.00
1.00
1.02
2.00
2.99
2.03
0.89
2.01
2.02
2.00
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

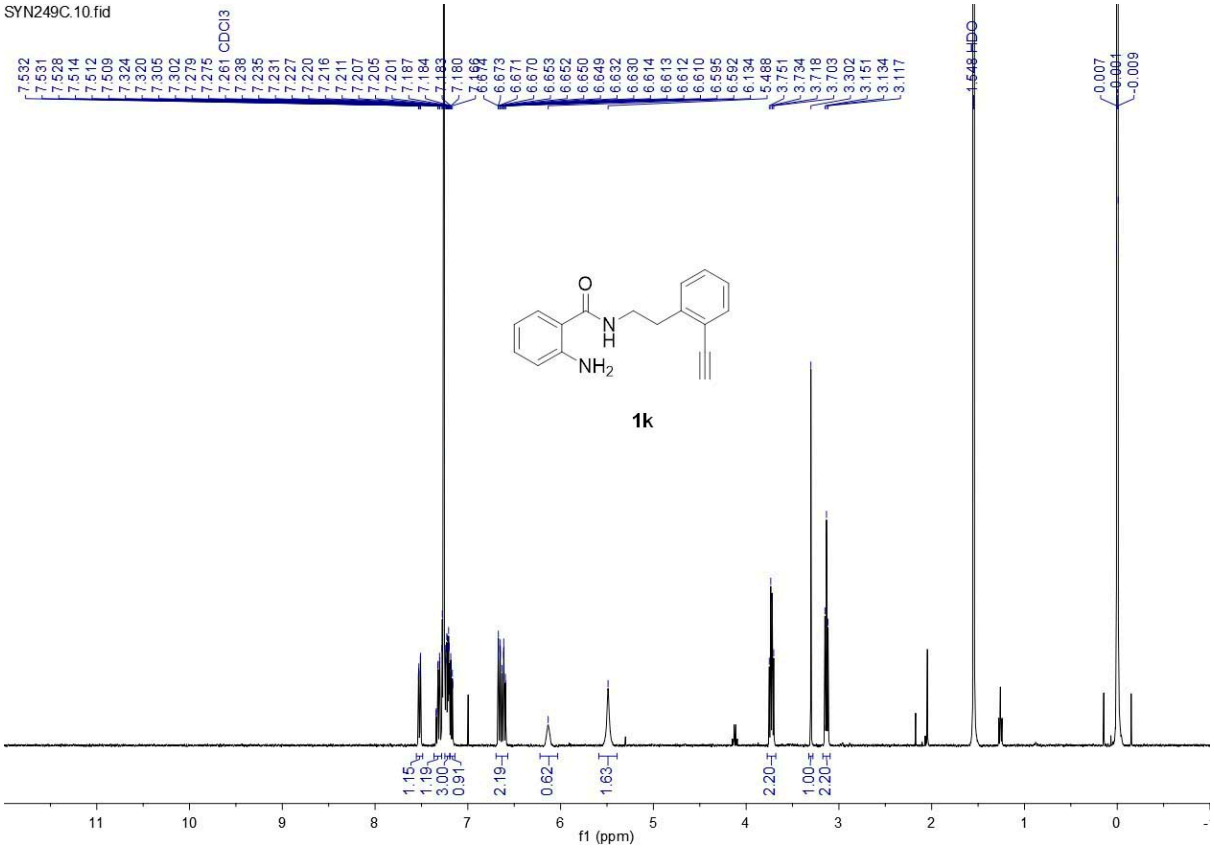

C-SJY452D.10.fid
168.494
149.320
143.656
119.141
117.593
115.333
112.131
84.436
71.233
55.570
38.198
28.317
25.529
17.427
MeO
O
N
H
NH2
1h
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


SJY455B.10.fid
8.018
8.003
7.989
7.069
6.294
6.287
3.701
3.668
3.215
3.201
3.198
3.183
3.180
3.166
2.764
2.758
2.751
2.213
2.206
2.195
2.189
2.178
2.172
1.598
1.581
1.577
1.571
1.563
1.560
1.557
1.489
1.485
1.478
1.472
1.468
MeO
MeO
O
N
H
NH2
1i
0.98
1.00
2.97
2.93
3.11
1.99
0.85
2.03
2.04
2.02
C-SJY455B.10.fid
168.404
152.693
146.207
138.972
112.547
105.249
99.893
84.497
71.273
56.733
55.091
38.178
28.553
25.607
17.480
MeO
MeO
O
N
H
NH2
1i
f1 (ppm)

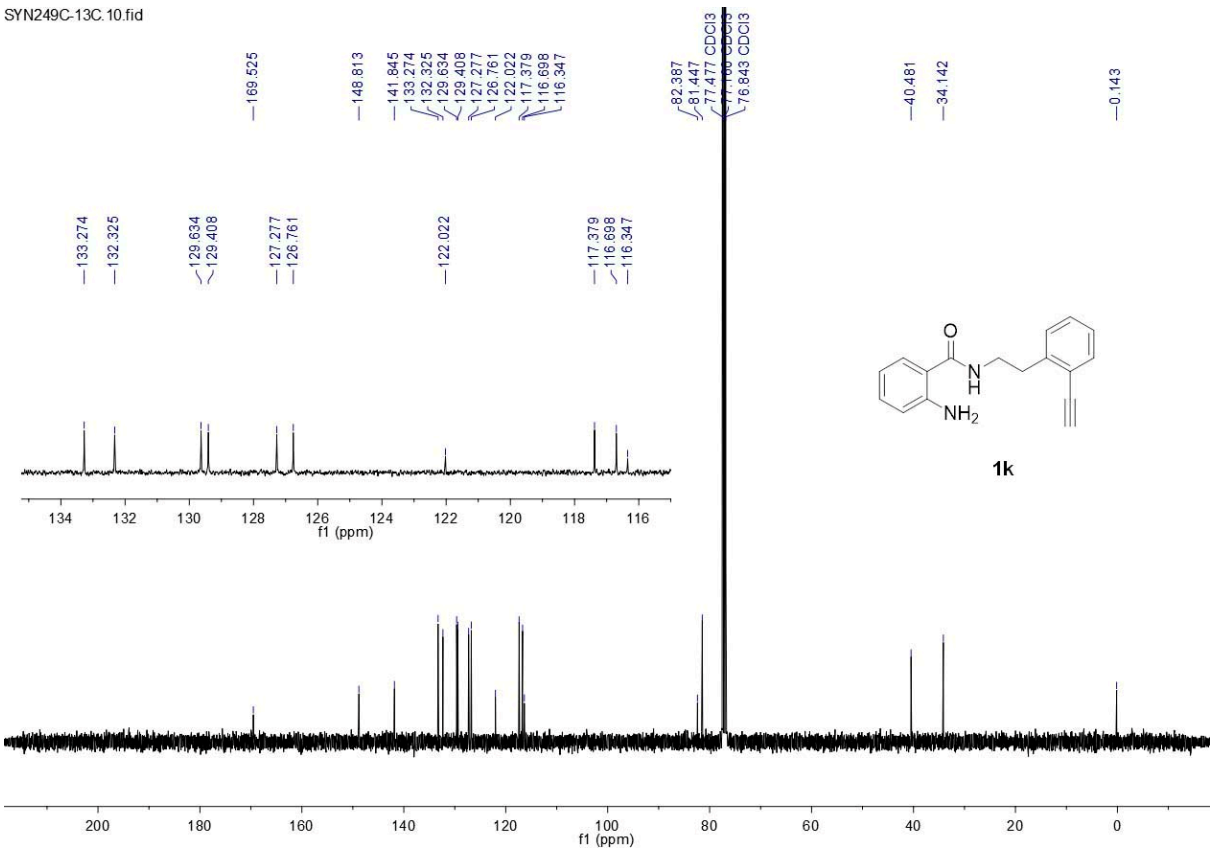

SYN231Asalt.10.fid
8.382
7.260 CDCl3
2.860
2.242
2.235
2.223
2.217
2.203
2.196
1.990
1.984
1.977
1.681
1.661
1.642
1.623
1.612
1.607 HDO
1.101
0.066
0.005
-0.003
-0.011
Cl
H3N
S2j'
2.71
2.07
2.08
1.00
2.38
6.18
f1 (ppm)


SYN231Asalt-13C.10.fid
84.094
77.477 CDCl3
76.842 CDCl3
69.180
49.446
38.362
33.404
24.744
13.608
0.139
Cl
H3N
S2j'
f1 (ppm)

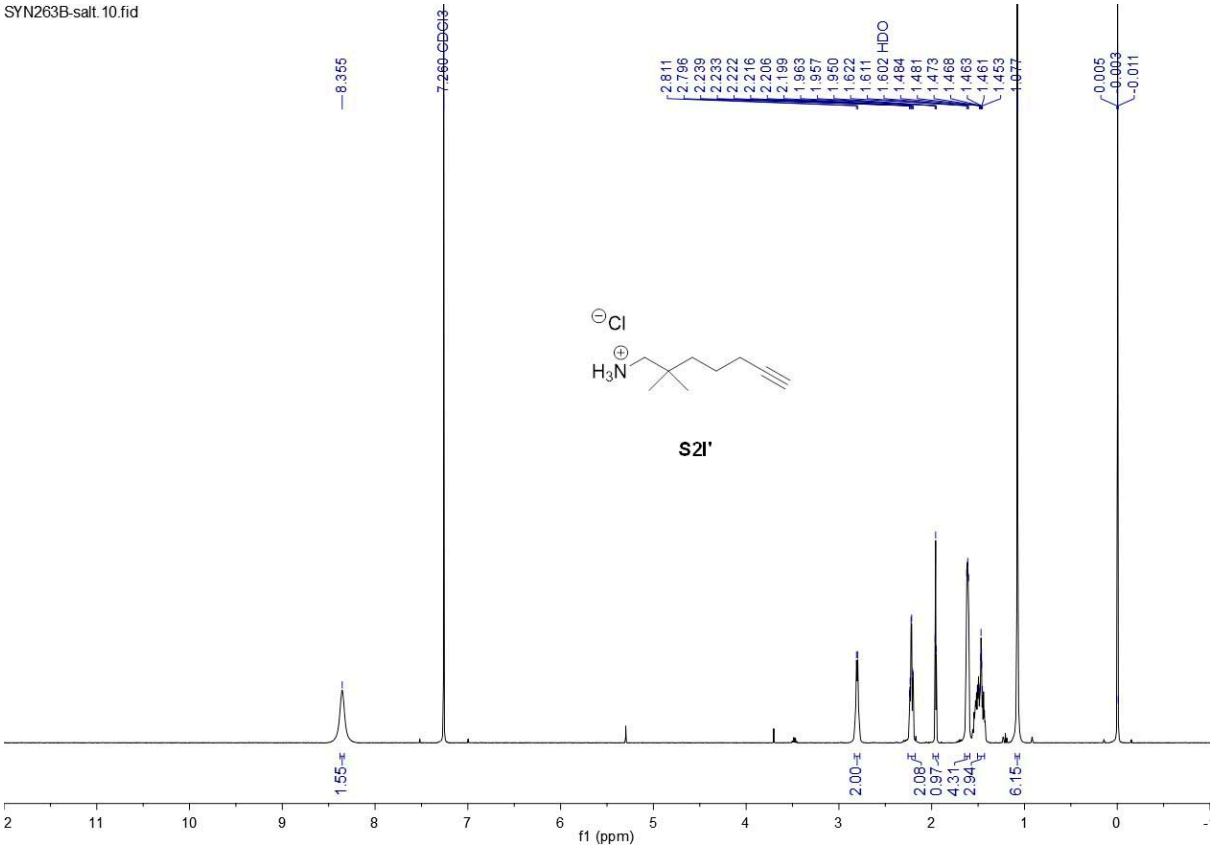

SYN247C.10.fid
7.331
7.327
7.312
7.308
7.260 CDCl3
7.260 CDCl3
7.233
7.229
7.215
7.212
7.211
7.208
7.194
7.190
6.699
6.698
6.696
6.695
6.685
6.682
6.678
6.677
6.675
6.674
6.667
6.665
6.664
6.662
6.655
6.647
6.645
6.200
5.459
3.303
3.287
2.272
2.265
2.253
2.246
2.233
2.226
1.984
1.977
1.971
1.605
1.584
1.568
0.966
0.807
0.006
0.002
-0.010
1.00
1.08
1.98
0.62
1.33
1.98
2.00
0.90
3.17
5.94
O
N
H
NH2
1j
SYN247C-13C.10.fid
169.497
148.770
132.371
126.981
117.454
116.790
116.702
85.270
77.478 CDCl3
77.160 CDCl3
76.843 CDCl3
68.482
48.338
38.557
34.929
25.119
13.685
0.144
117.454
116.790
116.702
f1 (ppm)
f1 (ppm)


SYN249C.10.fid
CDCl3
1.548 HDO
0.007
0.001
-0.009
1.15
1.19
3.00
0.91
2.19
0.62
1.63
2.20
1.00
2.20
f1 (ppm)

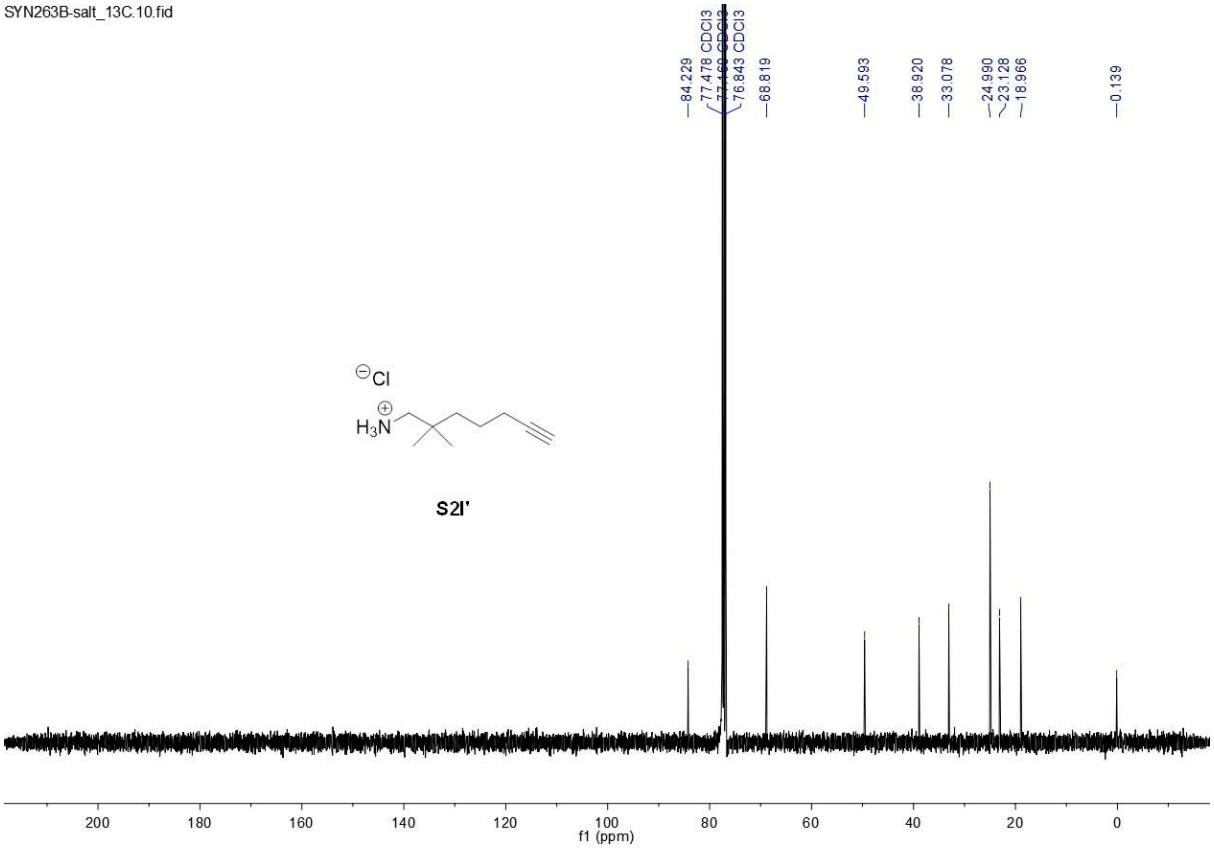
1k


SYN249C-13C.10.fid
169.525
148.813
141.845
133.274
132.325
129.634
129.408
127.277
126.761
122.022
117.379
116.698
116.347
82.387
81.447
77.477 CDCl3
76.843 CDCl3
40.481
34.142
0.143
f1 (ppm)

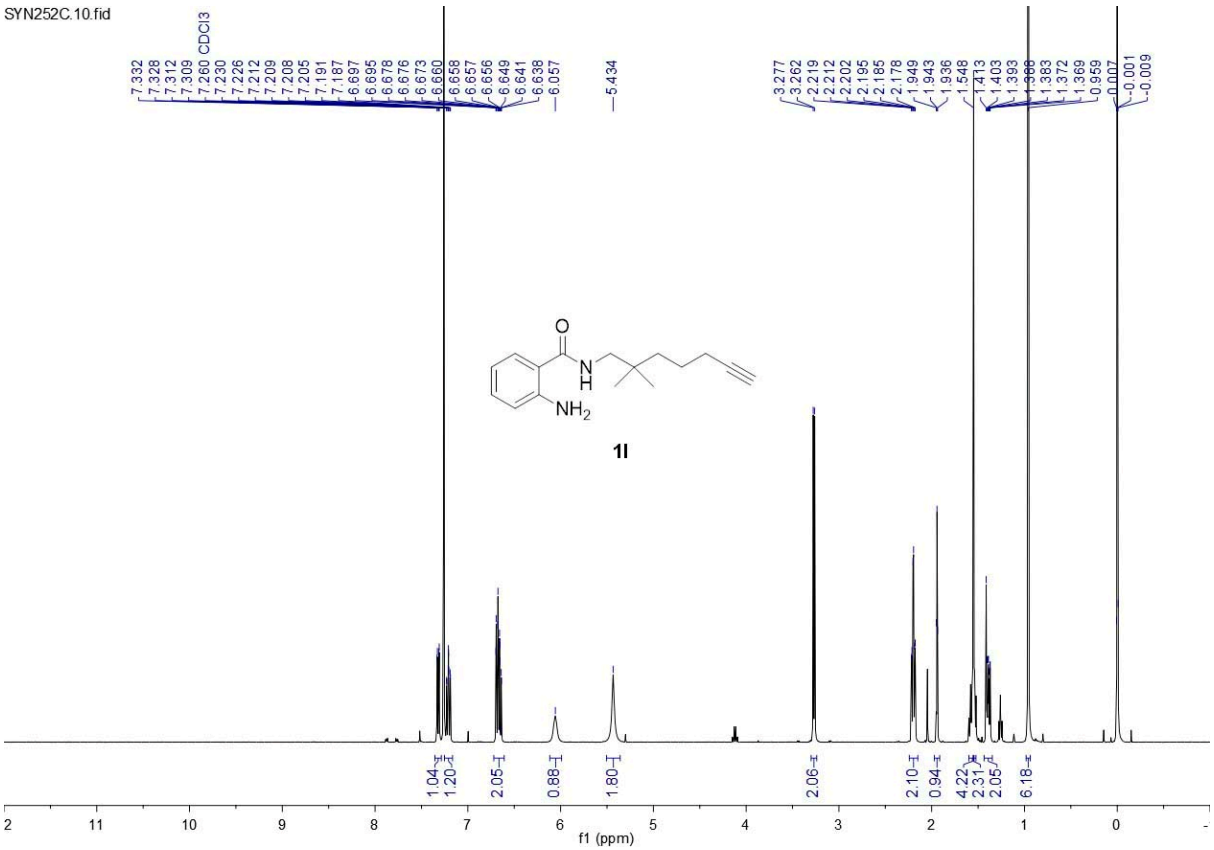
1k

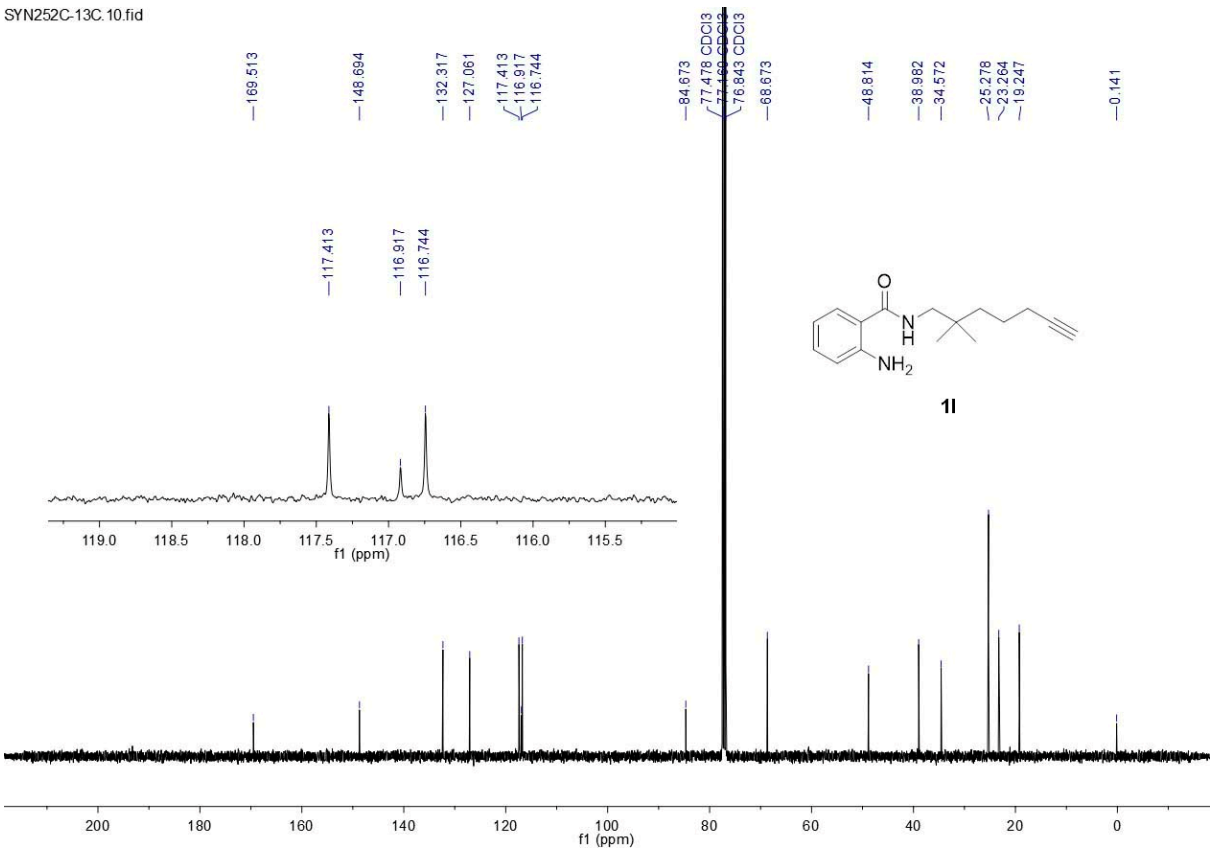

SYN263B-salt.10.fid
8.355
7.260 CDCl3
2.811
2.796
2.239
2.233
2.222
2.216
2.206
2.199
1.963
1.957
1.950
1.622
1.611
1.602 HDO
1.484
1.481
1.473
1.468
1.463
1.461
1.453
1.077
0.005
0.003
-0.011
1.55
2.00
2.08
0.97
4.31
2.94
6.15
f1 (ppm)

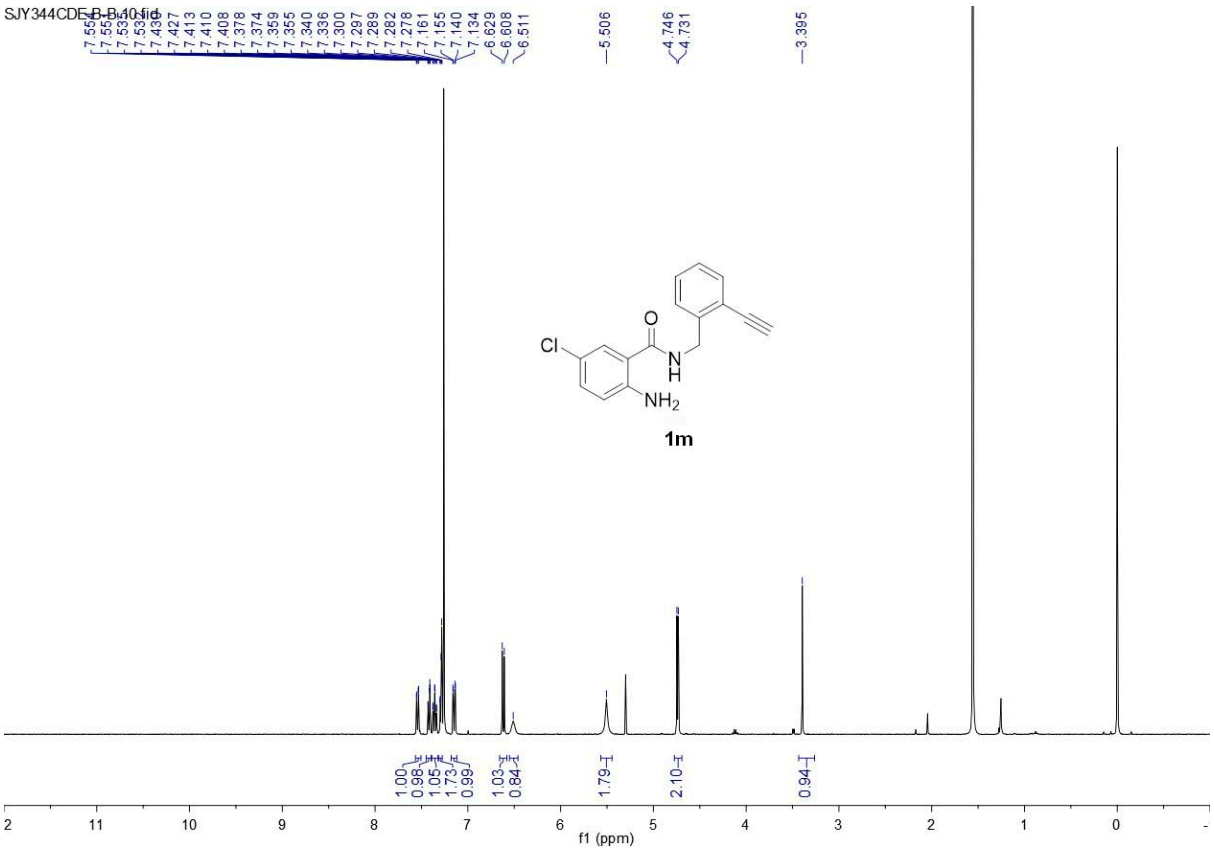
S2l'

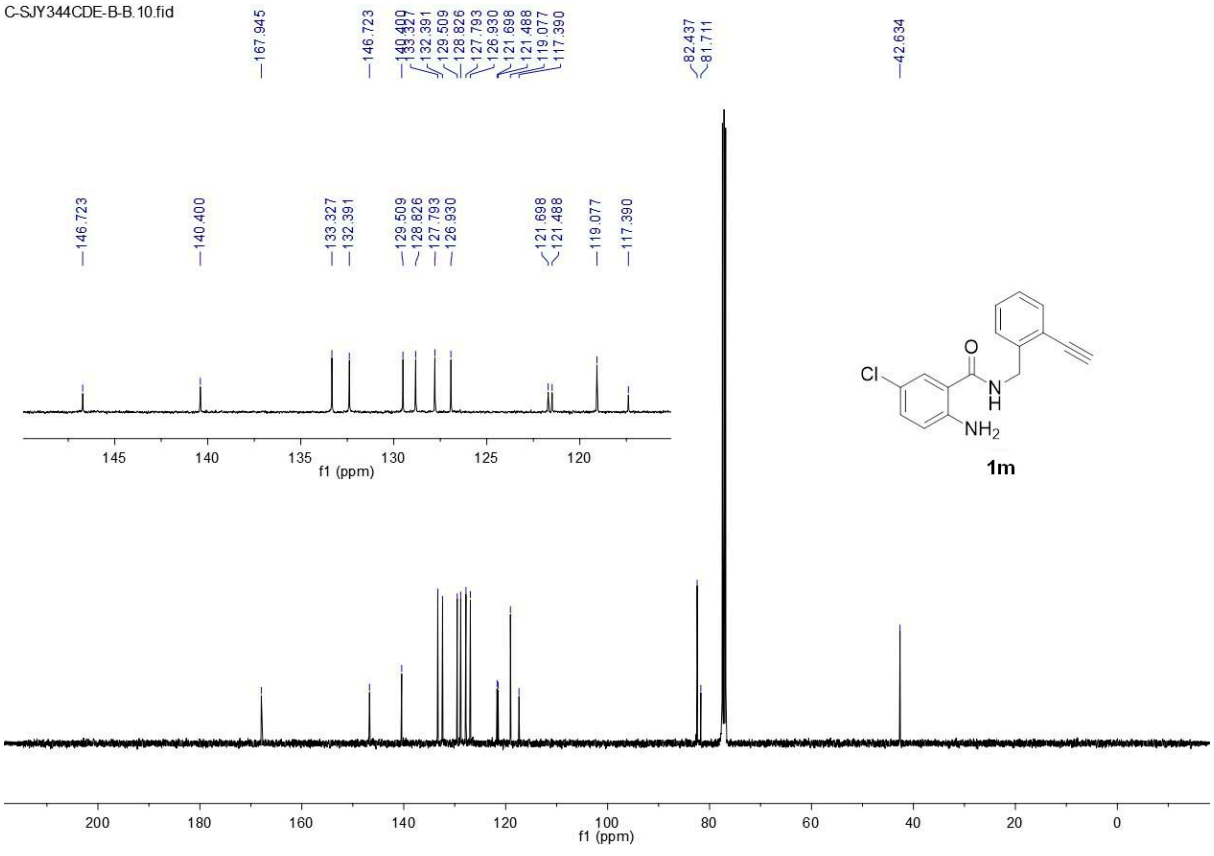

SYN263B-salt_13C.10.fid
84.229
77.478 CDCl3
76.843 CDCl3
68.819
49.593
38.920
33.078
24.990
23.128
18.966
0.139
f1 (ppm)

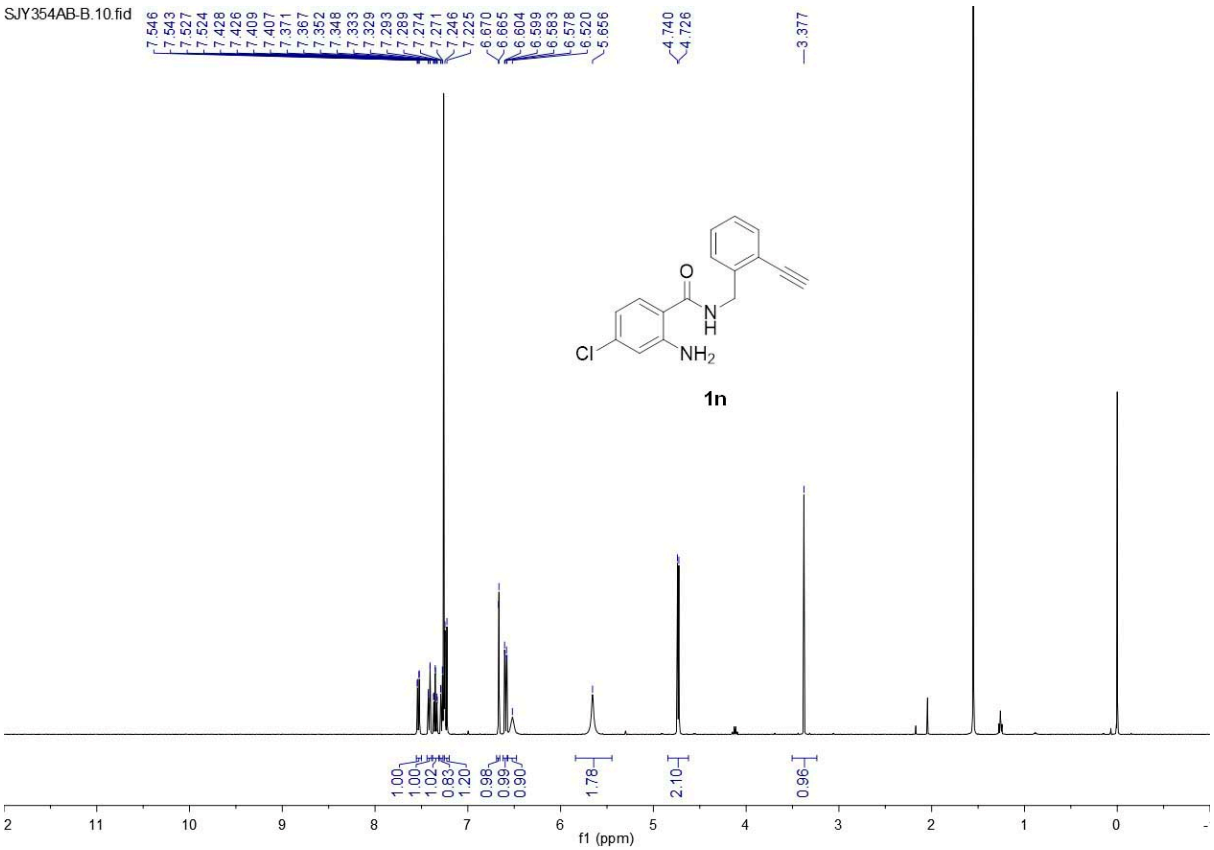
S2l'

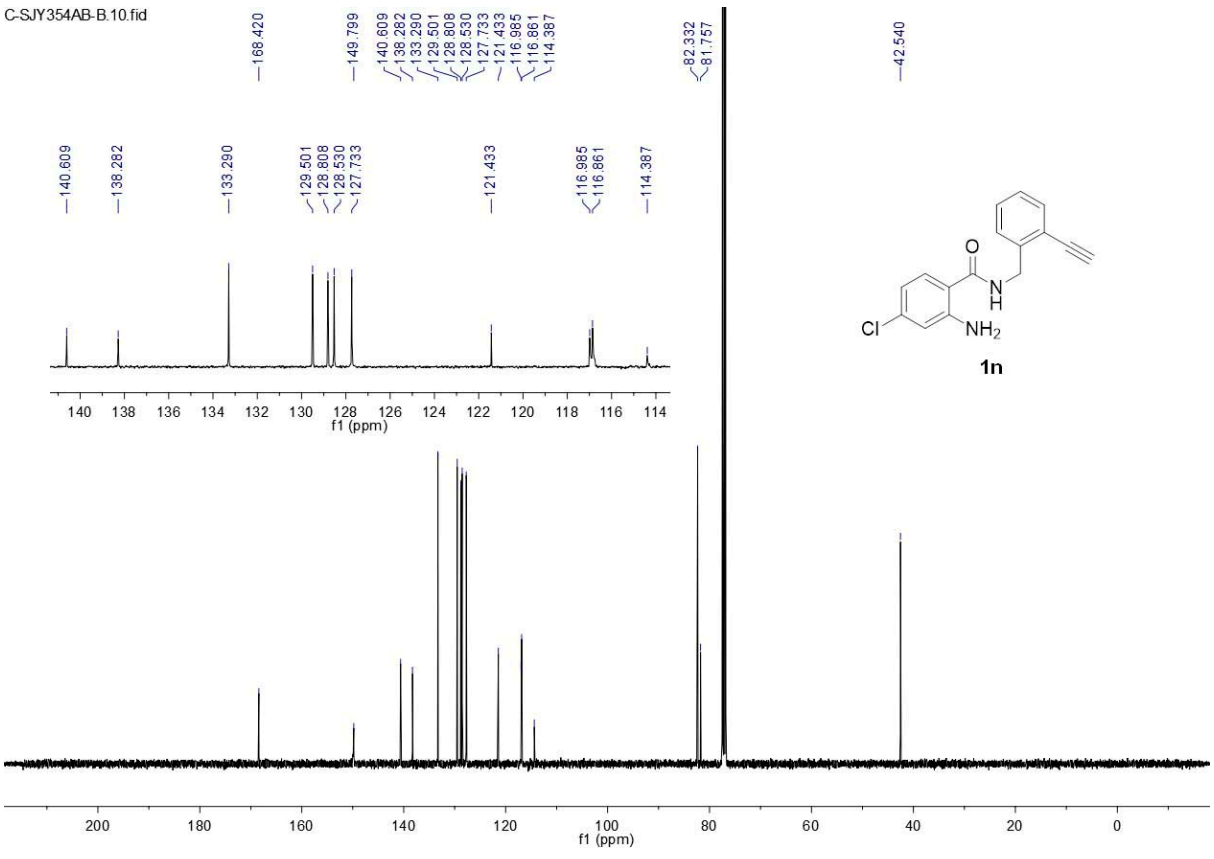

SYN252C.10.fid
7.332
7.328
7.312
7.309
7.260 CDCl3
7.230
7.226
7.212
7.209
7.208
7.205
7.191
7.187
6.697
6.695
6.678
6.676
6.673
6.660
6.658
6.657
6.656
6.649
6.641
6.638
6.057
5.434
3.277
3.262
2.219
2.212
2.202
2.195
2.185
2.178
1.949
1.943
1.936
1.548
1.413
1.403
1.393
1.383
1.372
1.369
0.959
0.007
-0.001
-0.009
1.04
1.20
2.05
0.88
1.80
2.06
2.10
0.94
4.22
2.31
2.05
6.18
f1 (ppm)
1l

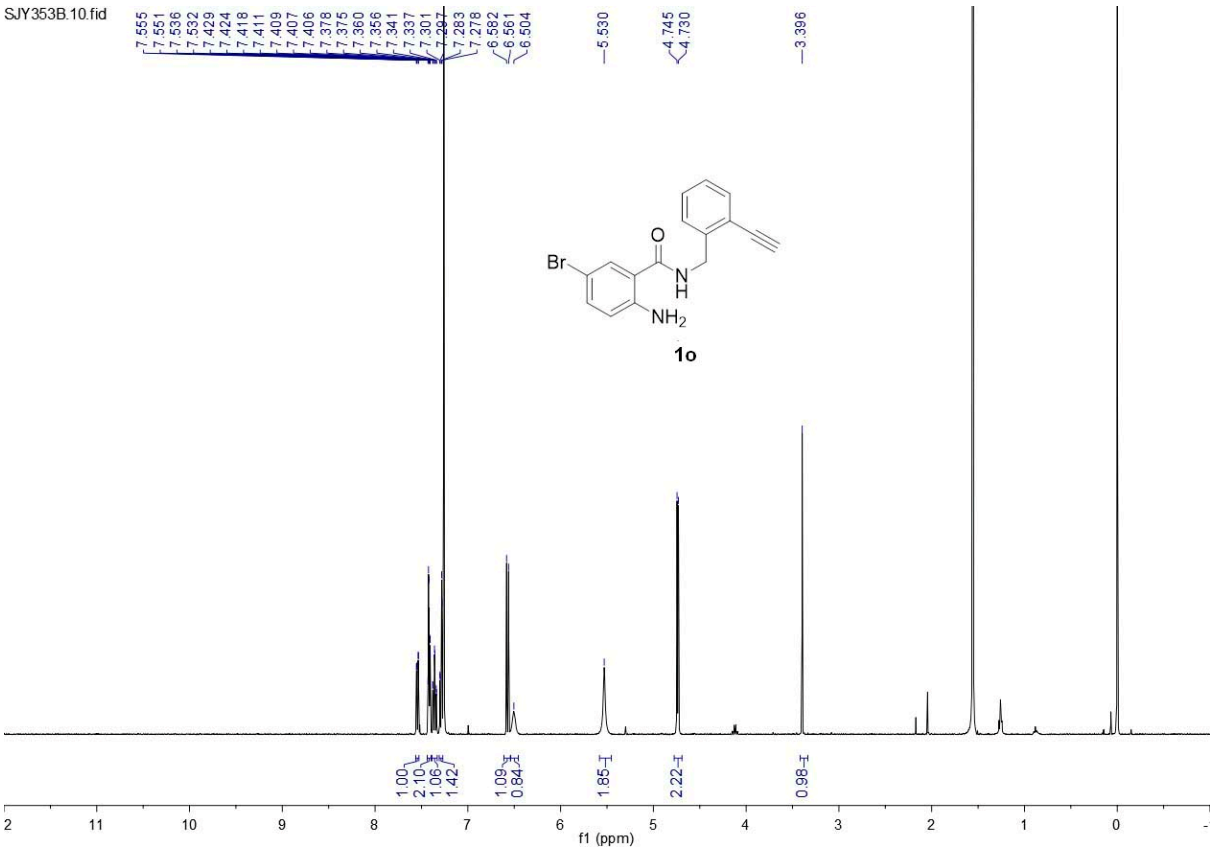

SYN252C-13C.10.fid
169.513
148.694
132.317
127.061
117.413
116.917
116.744
84.673
77.478 CDCl3
77.163 CDCl3
76.843 CDCl3
68.673
48.814
38.982
34.572
25.278
23.264
19.247
0.141
f1 (ppm)
1l

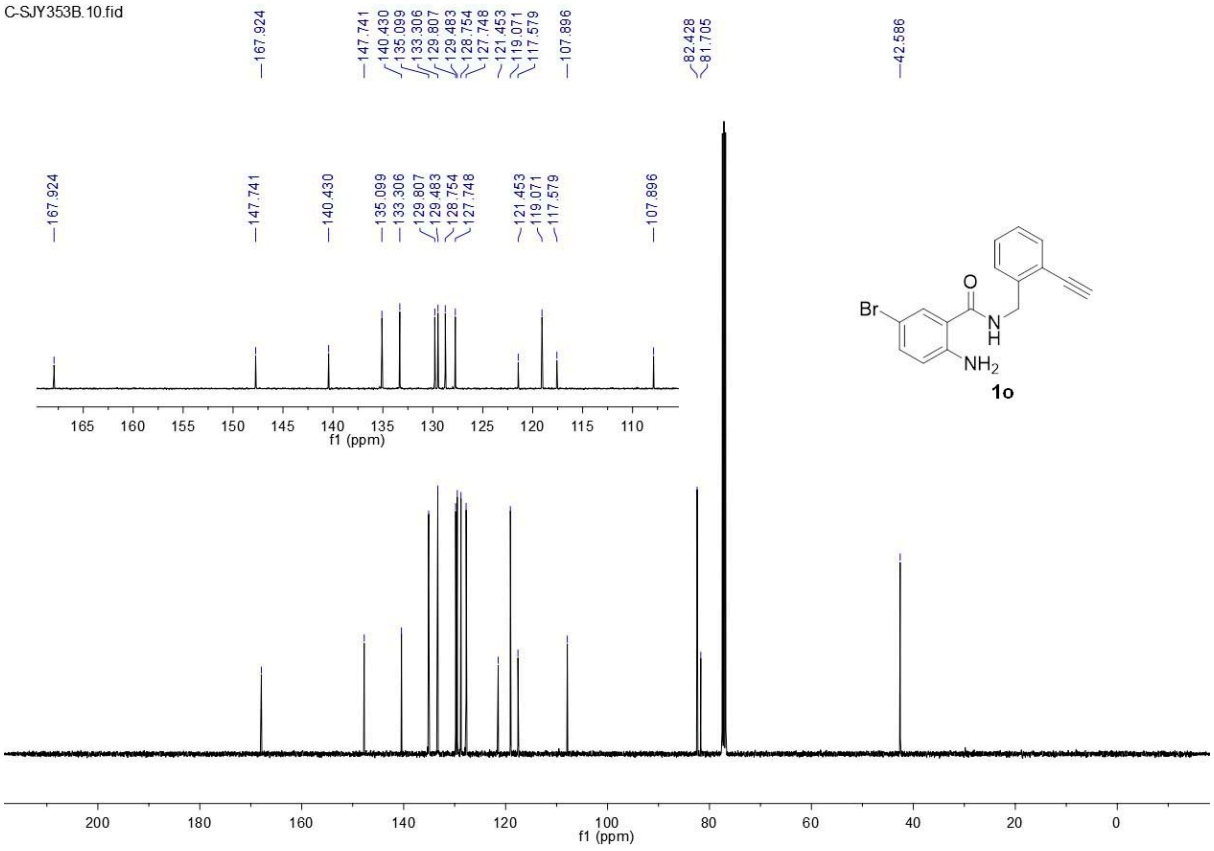

SJY344CDE-B-B.10.fid
7.556
7.555
7.553
7.532
7.439
7.427
7.413
7.410
7.408
7.378
7.374
7.359
7.355
7.340
7.336
7.300
7.297
7.289
7.282
7.278
7.161
7.155
7.140
7.134
6.629
6.608
6.511
5.506
4.746
4.731
3.395
Cl
O
N
H
NH2
1m
1.00
0.98
1.05
1.73
0.99
1.03
0.84
1.79
2.10
0.94
2
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

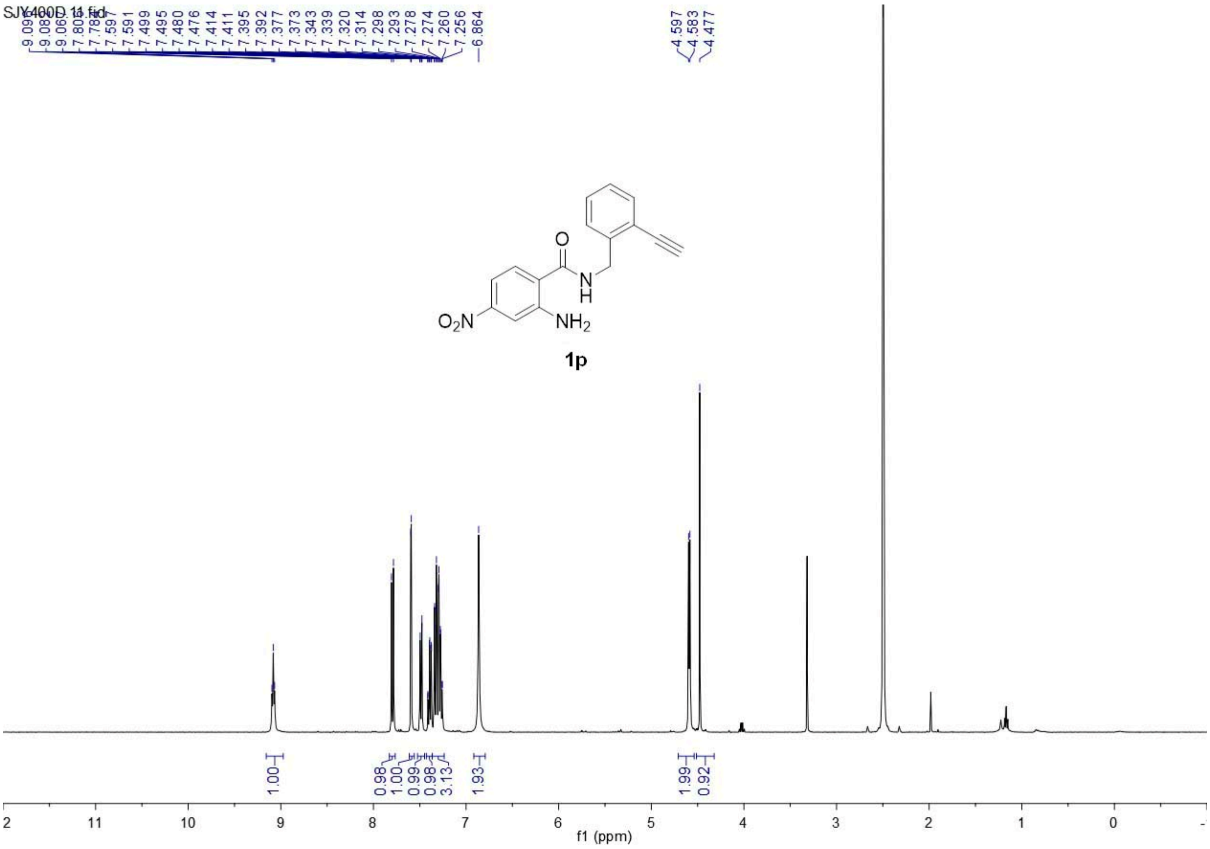

C-SJY344CDE-B-B.10.fid
167.945
146.723
140.400
133.327
132.391
129.509
128.826
127.793
126.930
121.698
121.488
119.077
117.390
82.437
81.711
42.634
146.723
140.400
133.327
132.391
129.509
128.826
127.793
126.930
121.698
121.488
119.077
117.390
145
140
135
130
125
120
f1 (ppm)
Cl
O
N
H
NH2
1m
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

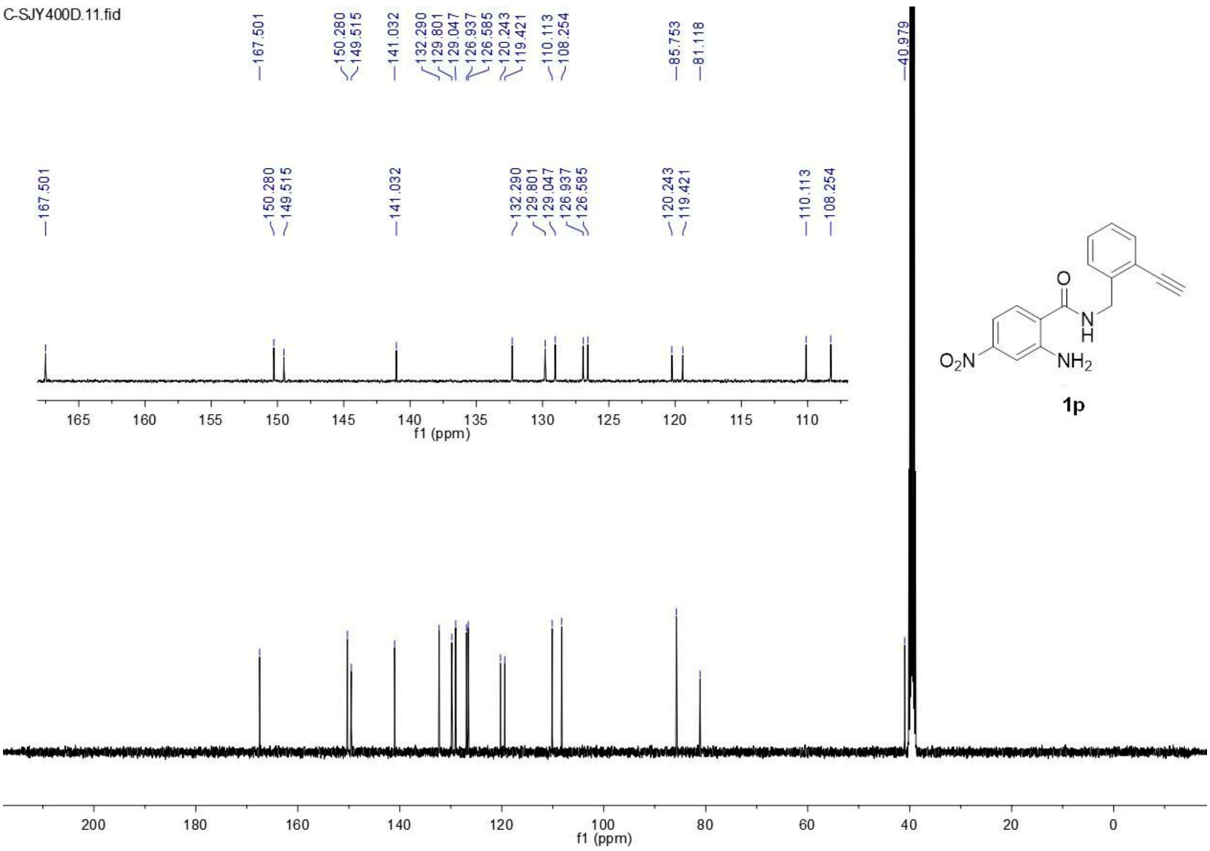

SJY354AB-B.10.fid
7.546
7.543
7.527
7.524
7.428
7.426
7.409
7.407
7.371
7.367
7.352
7.348
7.333
7.329
7.293
7.289
7.274
7.271
7.246
7.225
6.670
6.665
6.604
6.599
6.583
6.578
6.520
5.656
4.740
4.726
3.377
1.00
1.00
1.02
0.83
1.20
0.98
0.99
0.90
1.78
2.10
0.96
f1 (ppm)
Cl
NH2
O
N
H
1n

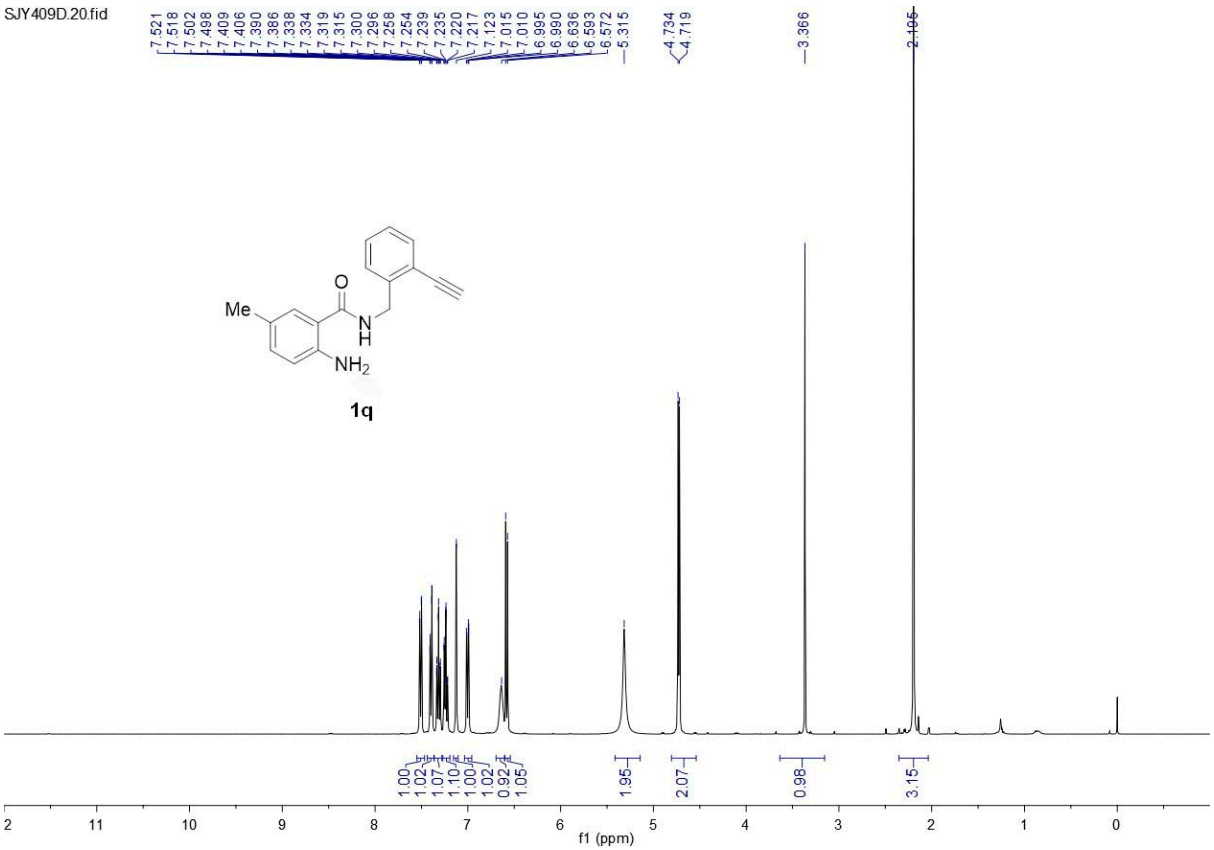

C-SJY354AB-B.10.fid
168.420
149.799
140.609
138.282
133.290
129.501
128.808
128.530
127.733
121.433
116.985
116.861
114.387
82.332
81.757
42.540
f1 (ppm)
Cl
NH2
O
N
H
1n

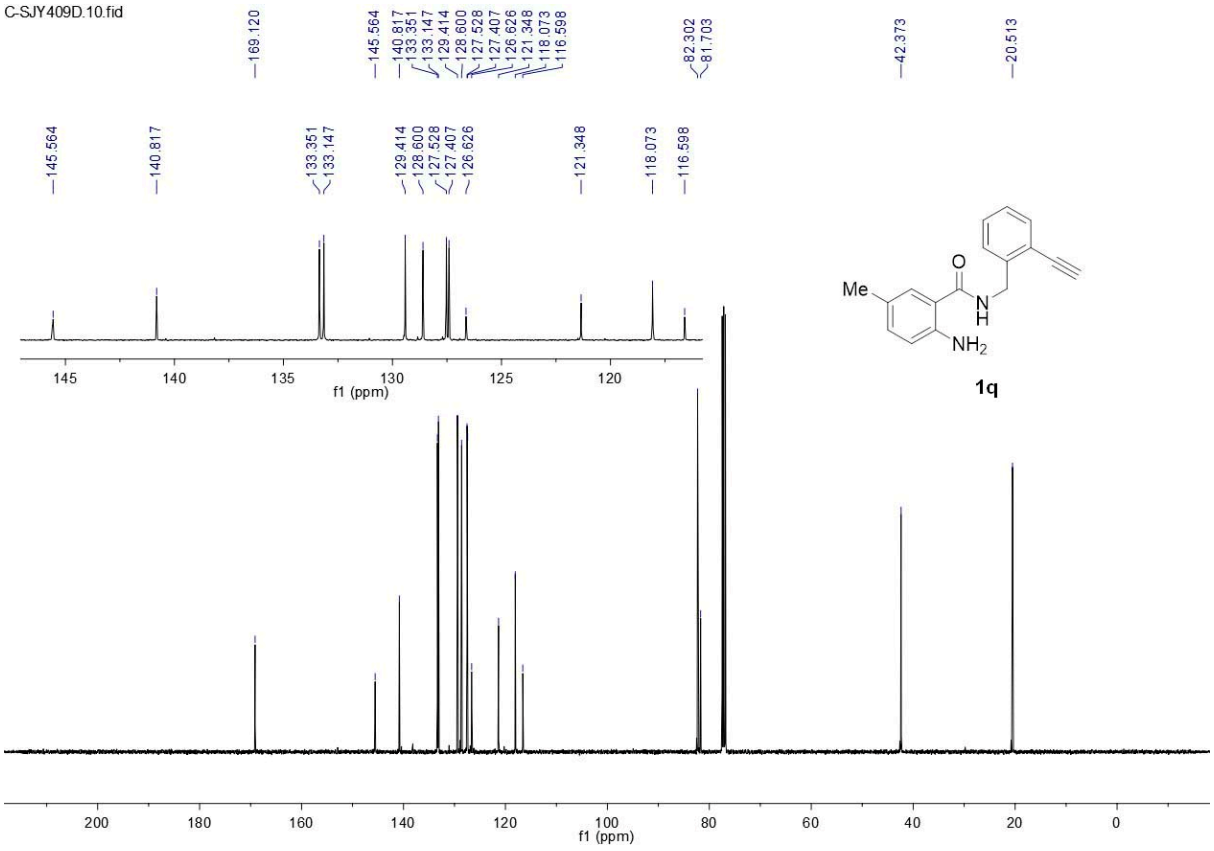

SJY353B.10.fid
7.555
7.551
7.536
7.532
7.429
7.424
7.418
7.411
7.409
7.407
7.406
7.378
7.375
7.360
7.356
7.341
7.337
7.301
7.287
7.283
7.278
6.582
6.561
6.504
5.530
4.745
4.730
3.396
Br
O
N
H
NH2
1o
1.00
2.10
1.06
1.42
1.09
0.84
1.85
2.22
0.98
f1 (ppm)

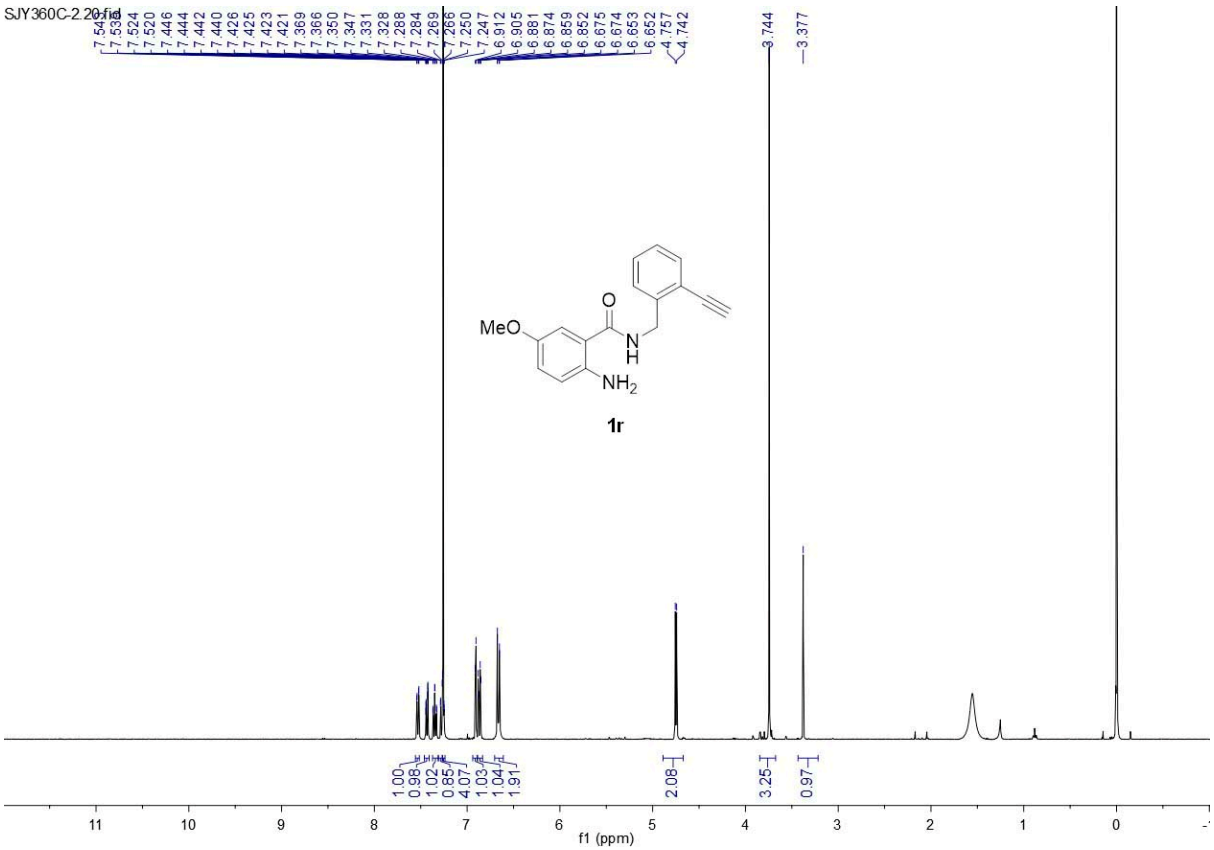

C-SJY353B.10.fid
167.924
147.741
140.430
135.099
133.306
129.807
129.483
128.754
127.748
121.453
119.071
117.579
107.896
82.428
81.705
42.586
Br
O
N
H
NH2
1o
f1 (ppm)

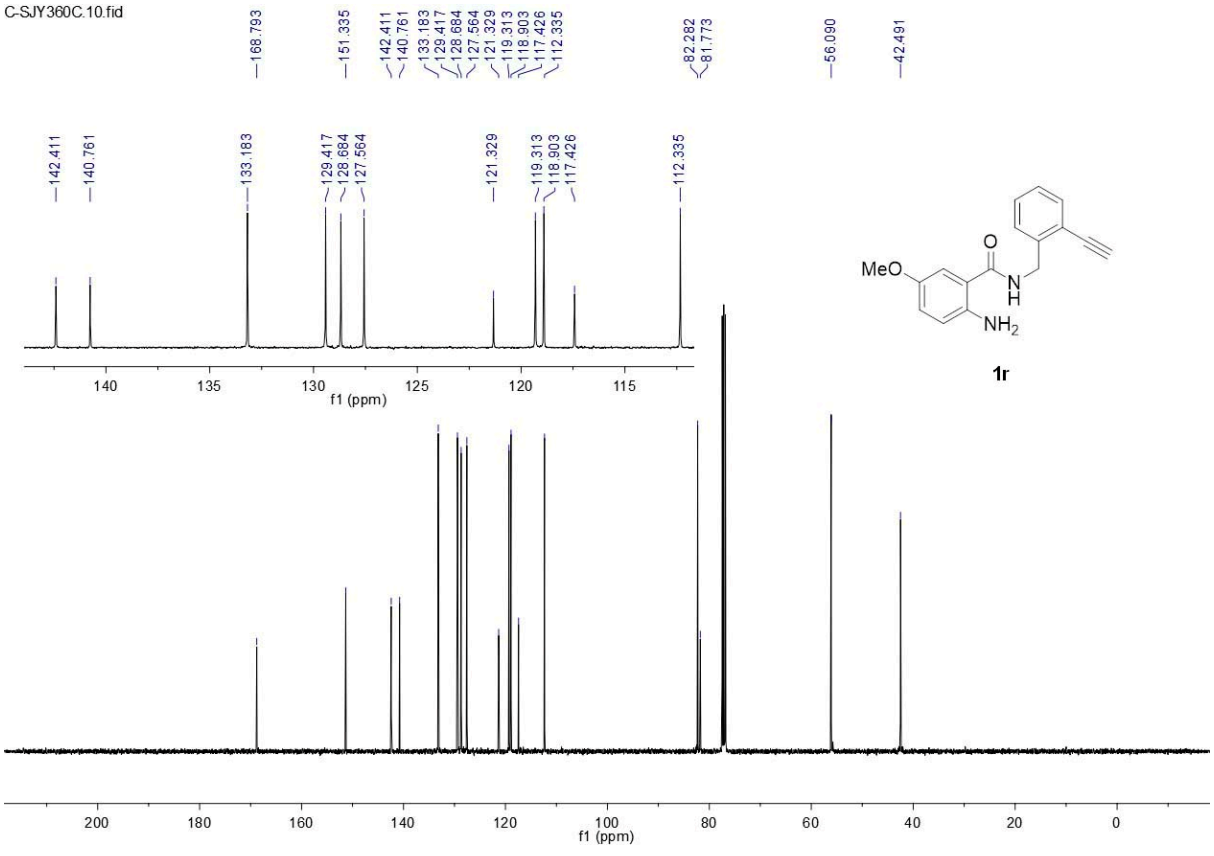

SJY400D.11.fid
9.099
9.085
9.061
7.808
7.788
7.597
7.591
7.499
7.495
7.480
7.476
7.414
7.411
7.395
7.392
7.377
7.373
7.343
7.339
7.320
7.314
7.298
7.293
7.278
7.274
7.260
7.256
6.864
4.597
4.583
4.477
O
N
H
O_2N
NH_2
1p
1.00
0.98
1.00
0.99
0.98
3.13
1.93
1.99
0.92
f1 (ppm)

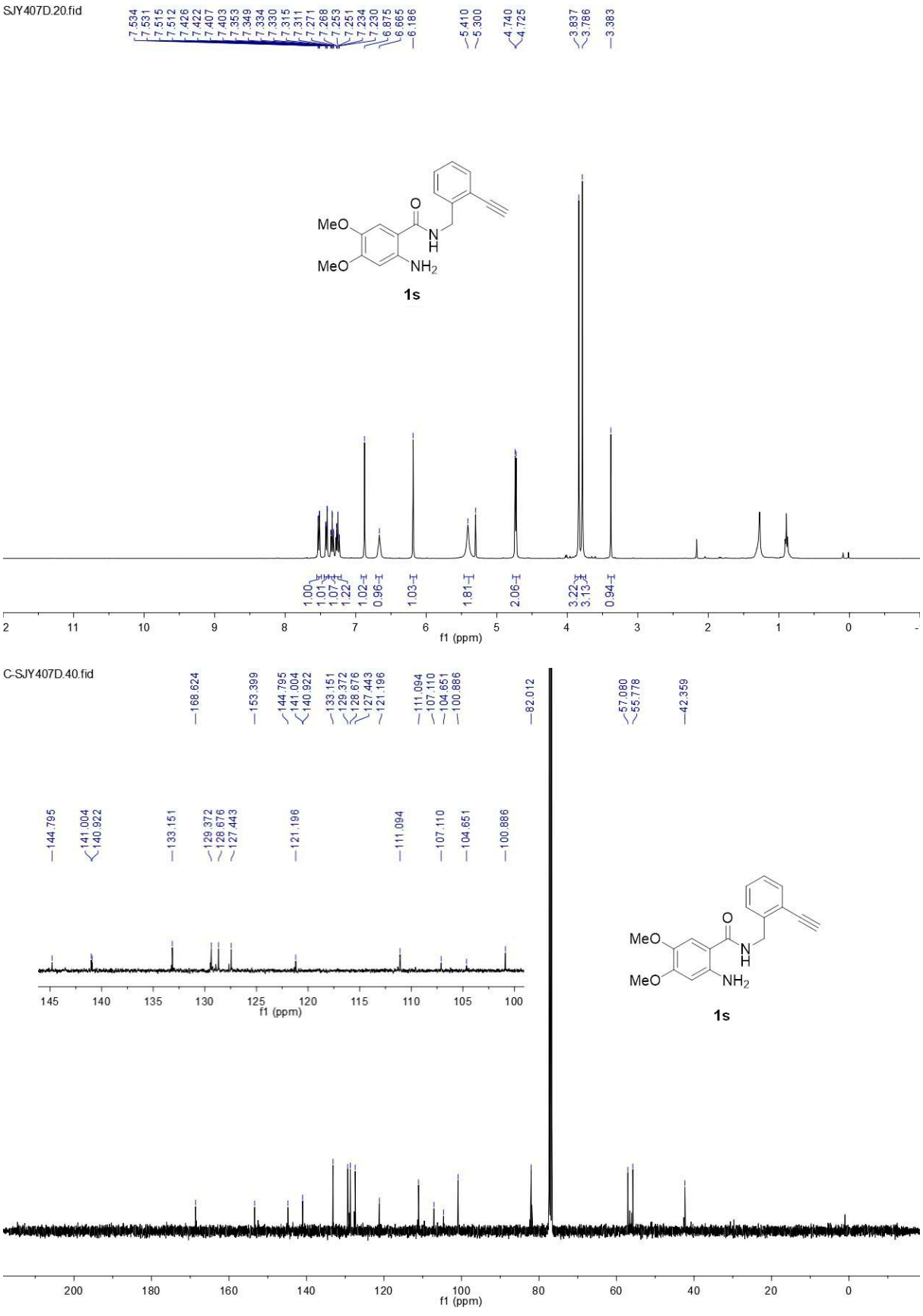

C-SJY400D.11.fid
167.501
150.280
149.515
141.032
132.290
129.801
129.047
126.937
126.585
120.243
119.421
110.113
108.254
85.753
81.118
40.979
O
N
H
O_2N
NH_2
1p
f1 (ppm)

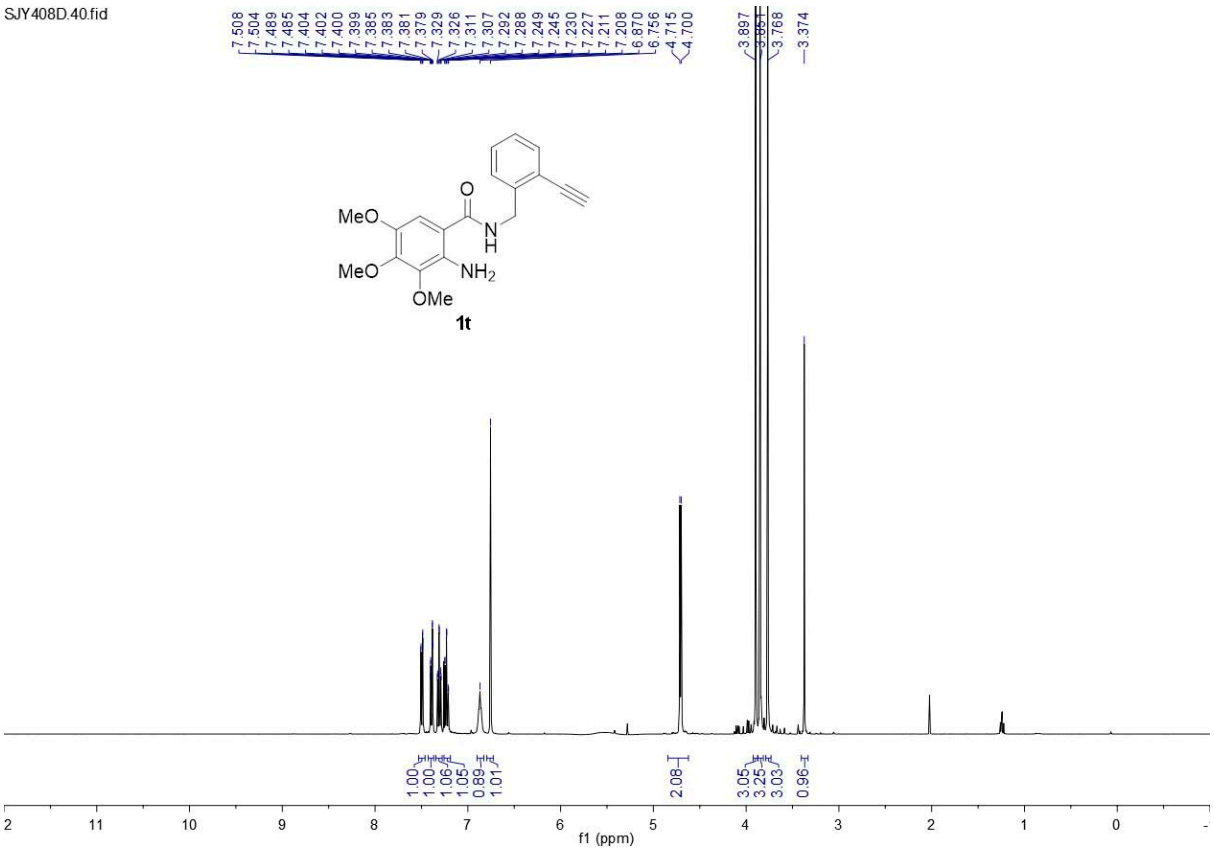

SJY409D.20.fid
7.521
7.518
7.502
7.498
7.409
7.406
7.390
7.386
7.338
7.334
7.319
7.315
7.300
7.296
7.258
7.254
7.239
7.235
7.220
7.217
7.123
7.015
7.010
6.995
6.990
6.636
6.593
6.572
5.315
4.734
4.719
3.366
2.195
Me
O
N
H
NH2
1q
1.00
1.02
1.07
1.10
1.00
1.02
0.92
1.05
1.95
2.07
0.98
3.15
f1 (ppm)

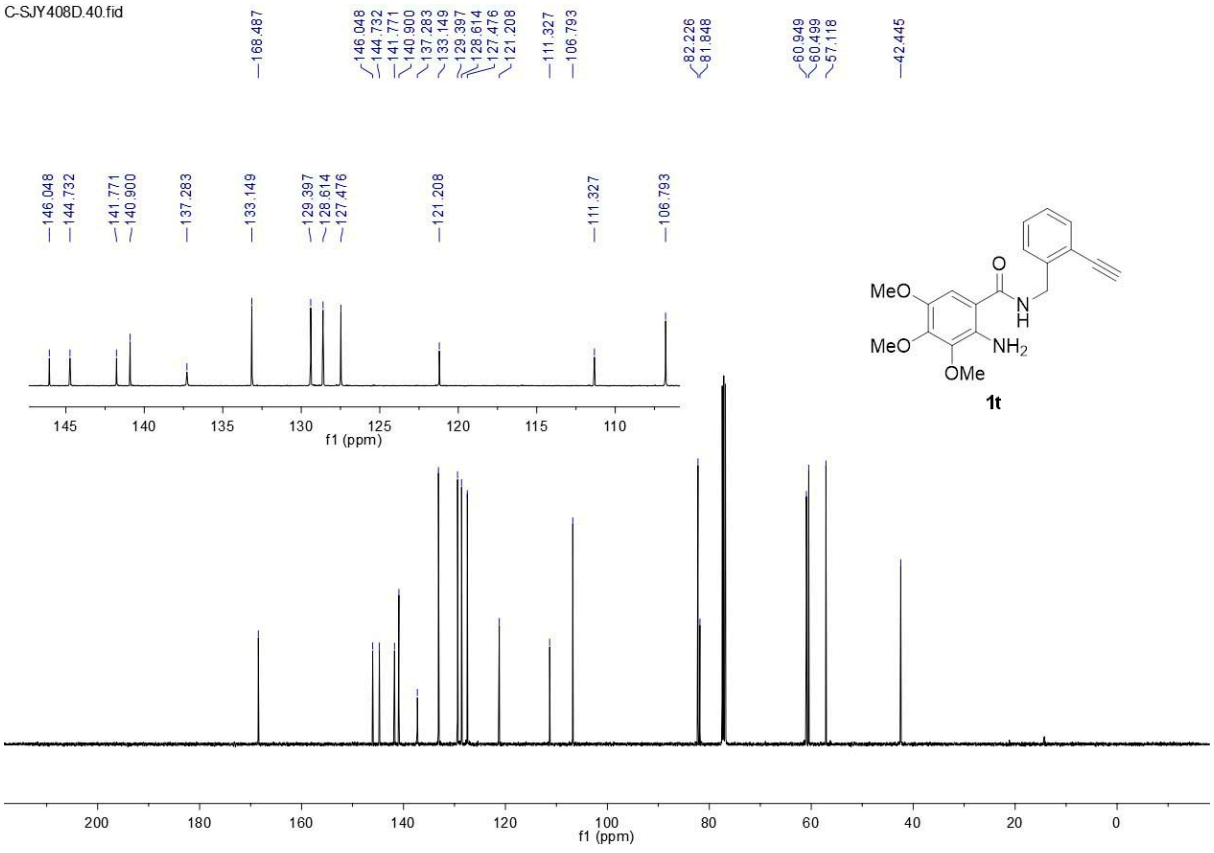

C-SJY409D.10.fid
169.120
145.564
140.817
133.351
133.147
129.414
128.600
127.528
127.407
126.626
121.348
118.073
116.598
82.302
81.703
42.373
20.513
Me
O
N
H
NH2
1q
f1 (ppm)

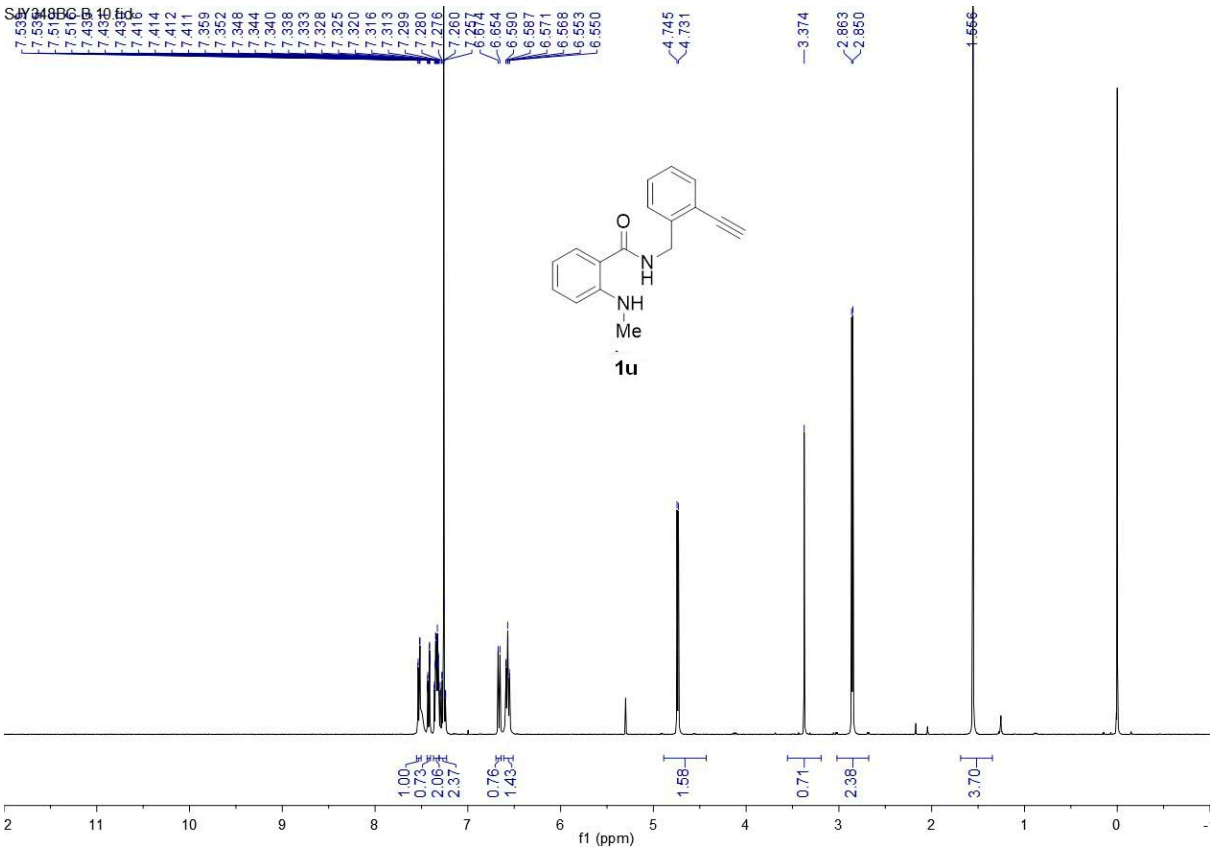

SJY360C-2.20.fid
7.544
7.538
7.524
7.520
7.446
7.444
7.442
7.440
7.426
7.425
7.423
7.421
7.369
7.366
7.350
7.347
7.331
7.328
7.288
7.284
7.269
7.266
7.250
7.247
6.912
6.905
6.881
6.874
6.859
6.852
6.675
6.674
6.653
6.652
4.757
4.742
3.744
3.377
1.00
0.98
1.02
0.85
4.07
1.03
1.04
1.91
2.08
3.25
0.97
MeO
O
N
H
NH2
1r
f1 (ppm)

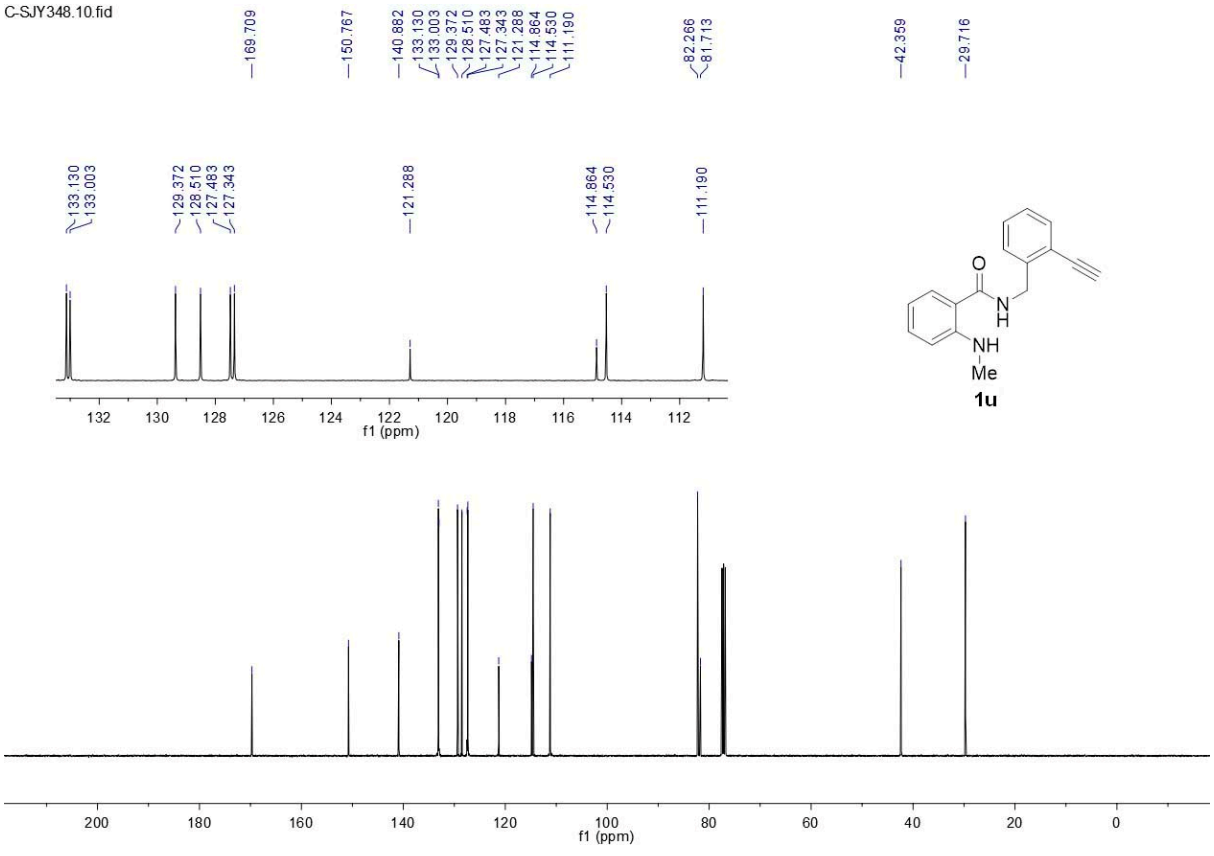

C-SJY360C.10.fid
168.793
151.335
142.411
140.761
133.183
129.417
128.684
127.564
121.329
119.313
118.903
117.426
112.335
82.282
81.773
56.090
42.491
MeO
O
N
H
NH2
1r
f1 (ppm)


SJY407D.20.fid
7.534
7.531
7.515
7.512
7.426
7.422
7.407
7.403
7.353
7.349
7.334
7.330
7.315
7.311
7.271
7.268
7.253
7.251
7.234
7.230
6.875
6.665
6.186
5.410
5.300
4.740
4.725
3.837
3.786
3.383
MeO
MeO
O
N
H
NH2
1s
1.00
1.01
1.07
1.22
1.02
0.96
1.03
1.81
2.06
3.22
3.13
0.94
f1 (ppm)
C-SJY407D.40.fid
168.624
153.399
144.795
141.004
140.922
133.151
129.372
128.676
127.443
121.196
111.094
107.110
104.651
100.886
82.012
57.080
55.778
42.359
f1 (ppm)
MeO
MeO
O
N
H
NH2
1s
f1 (ppm)

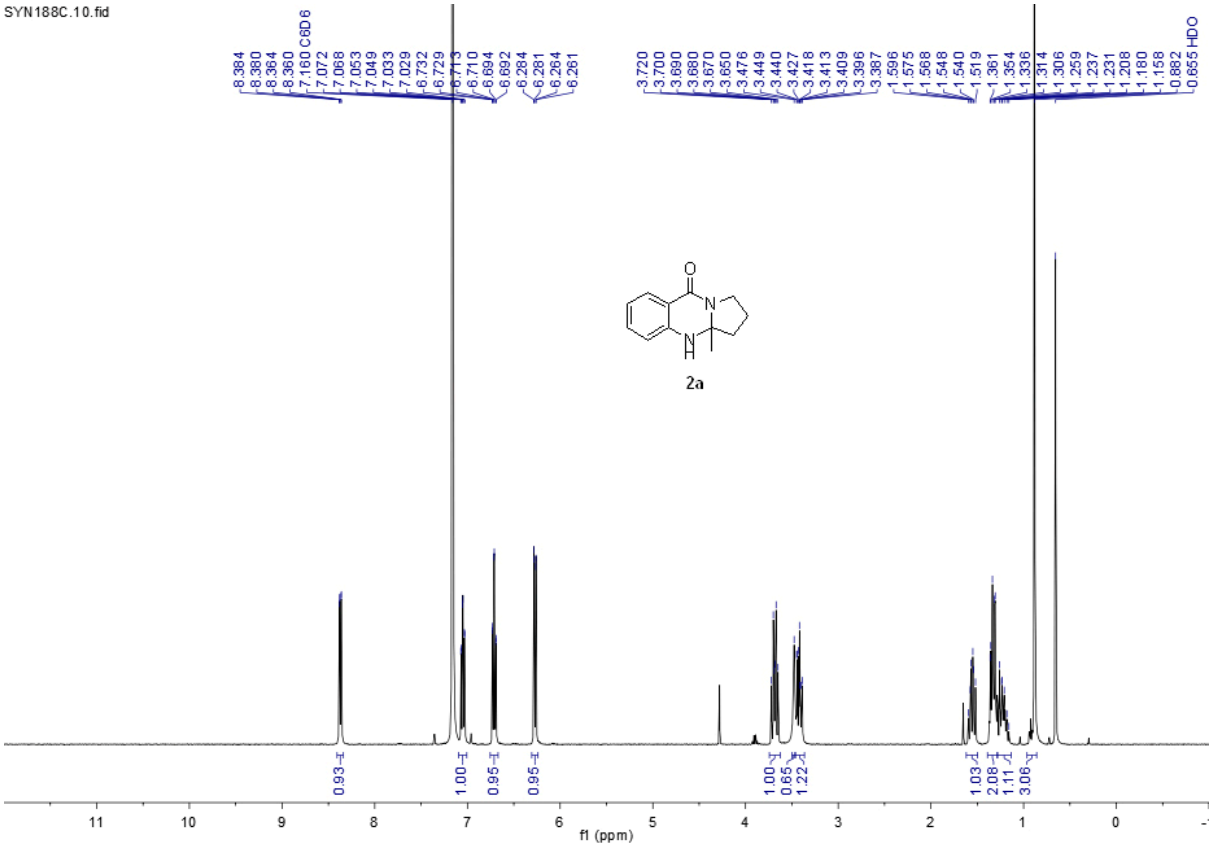

SJY408D.40.fid
7.508
7.504
7.489
7.485
7.404
7.402
7.400
7.399
7.385
7.383
7.381
7.379
7.329
7.326
7.311
7.307
7.292
7.288
7.249
7.245
7.230
7.227
7.211
7.208
6.870
6.756
4.715
4.700
3.897
3.861
3.768
3.374
MeO
MeO
OMe
NH2
O
N
H
1t
1.00
1.00
1.06
1.05
0.89
1.01
2.08
3.05
3.25
3.03
0.96
f1 (ppm)


C-SJY408D.40.fid
168.487
146.048
144.732
141.771
140.900
137.283
133.149
129.397
128.614
127.476
121.208
111.327
106.793
82.226
81.848
60.949
60.499
57.118
42.445
MeO
MeO
OMe
NH2
O
N
H
1t
f1 (ppm)

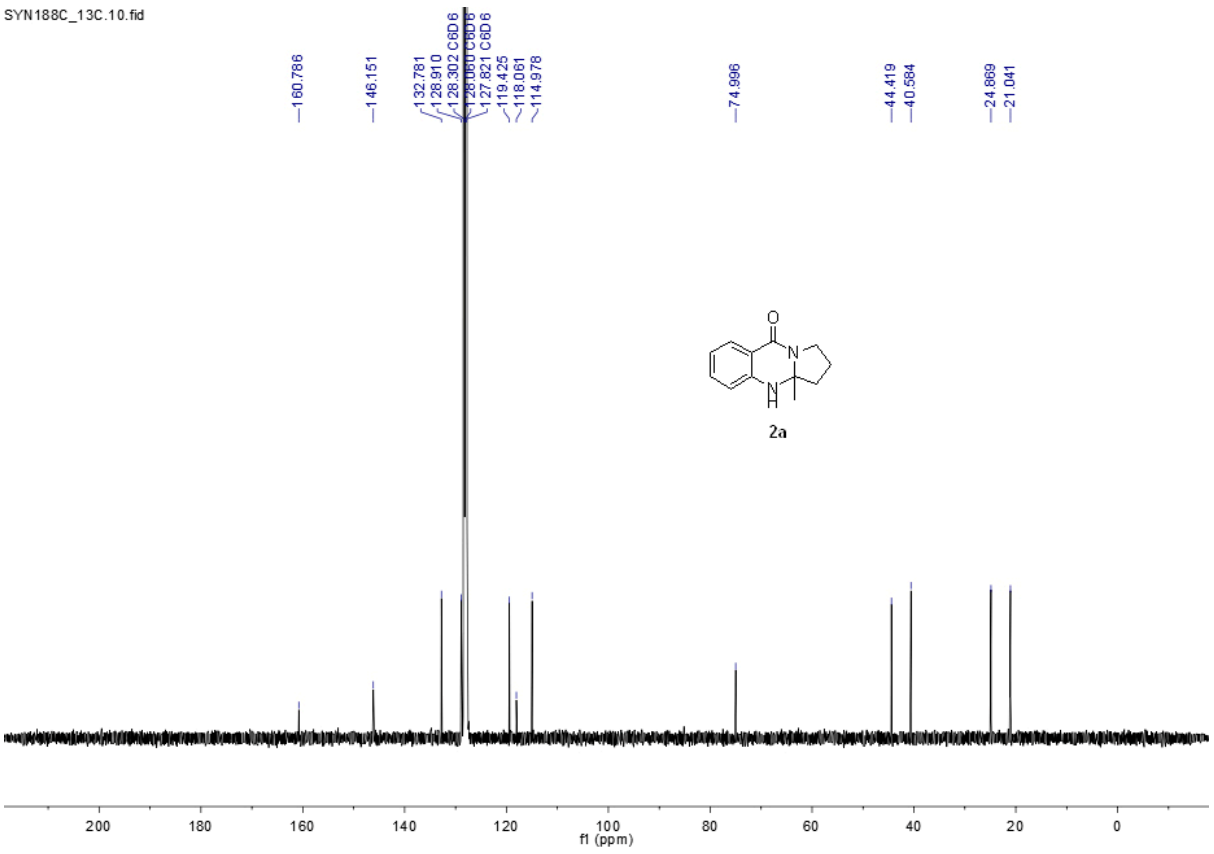




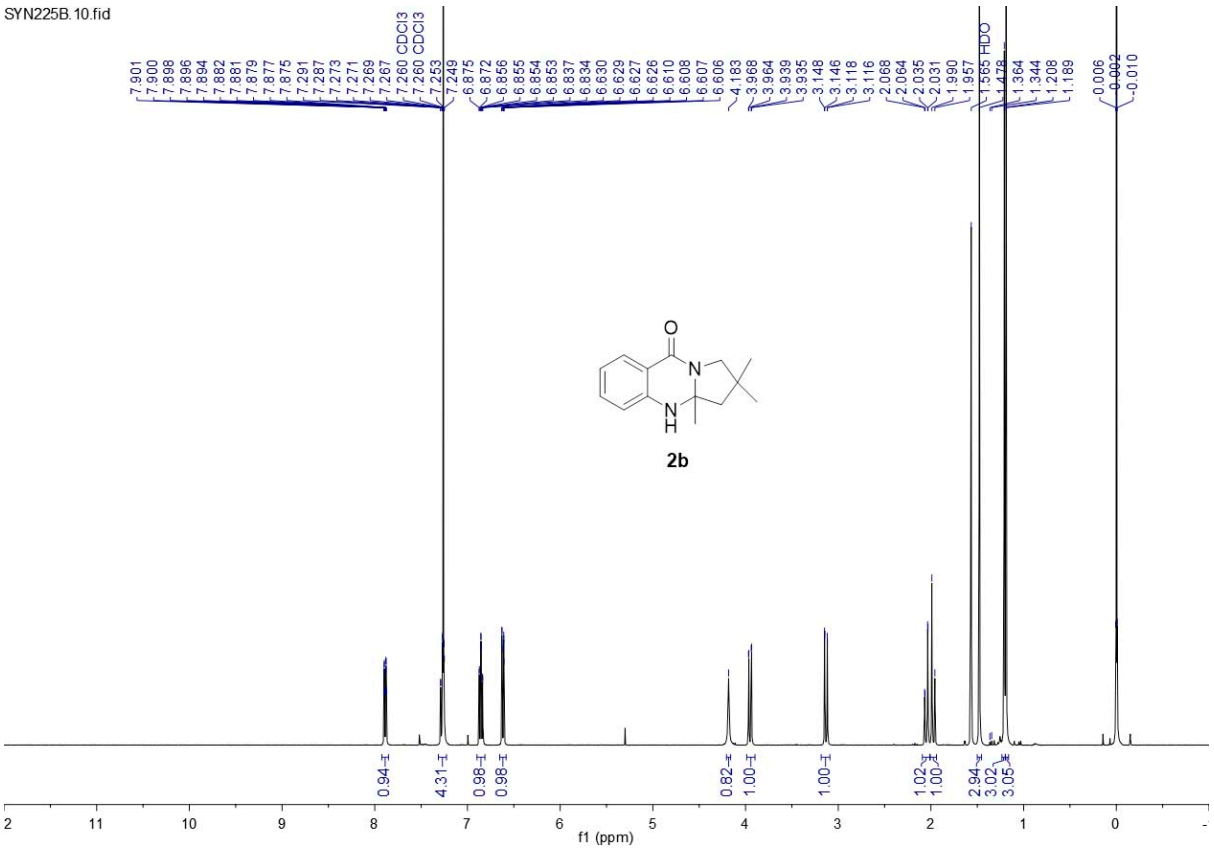
3.2. Spectra of Products 2a–s

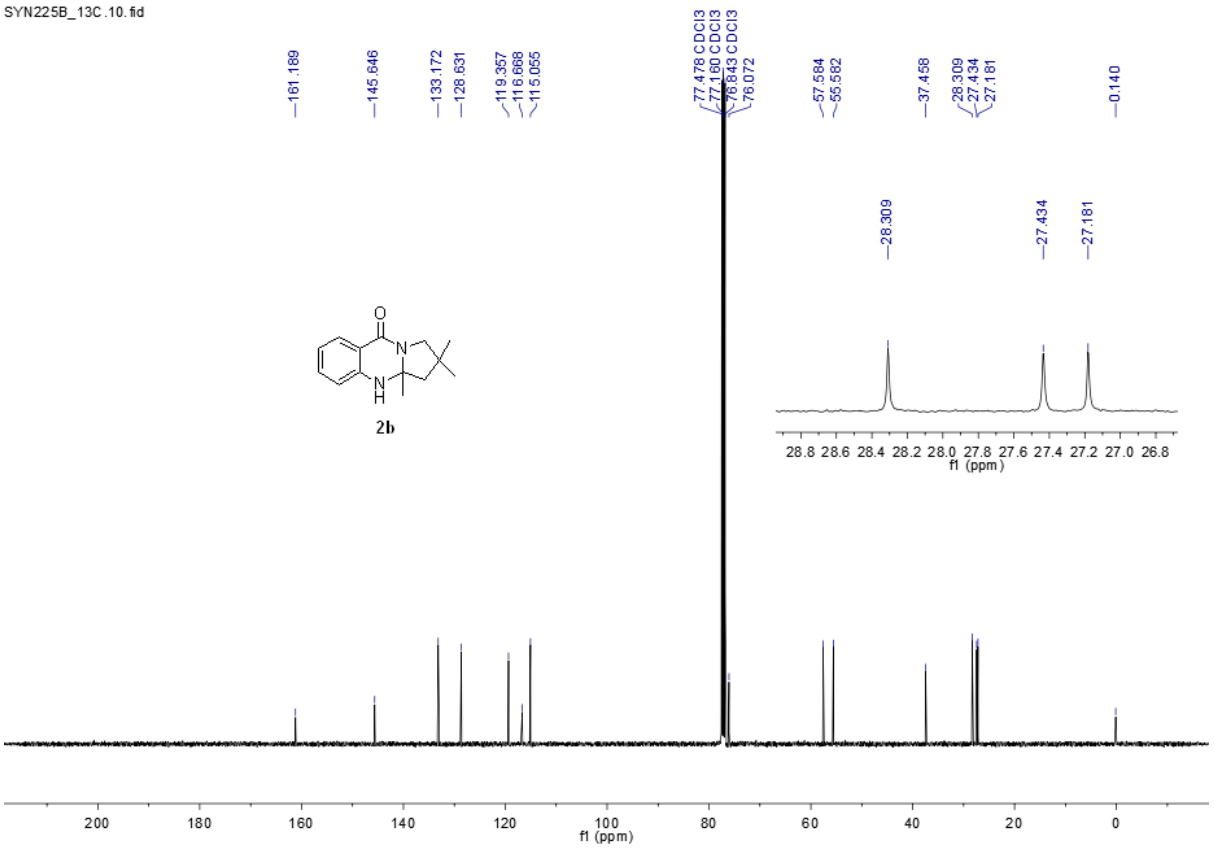

SYN188C.10.fid
8.384
8.380
8.364
8.360
7.160 C6D6
7.072
7.068
7.053
7.049
7.033
7.029
6.732
6.729
6.713
6.710
6.694
6.692
6.284
6.281
6.264
6.261
3.720
3.700
3.690
3.680
3.670
3.650
3.476
3.449
3.440
3.427
3.418
3.413
3.409
3.396
3.387
1.596
1.575
1.568
1.548
1.540
1.519
1.361
1.354
1.336
1.314
1.306
1.259
1.237
1.231
1.208
1.180
1.158
0.882
0.655 HDO
0.93
1.00
0.95
0.95
1.00
0.85
1.22
1.03
2.08
1.11
3.06
f1 (ppm)

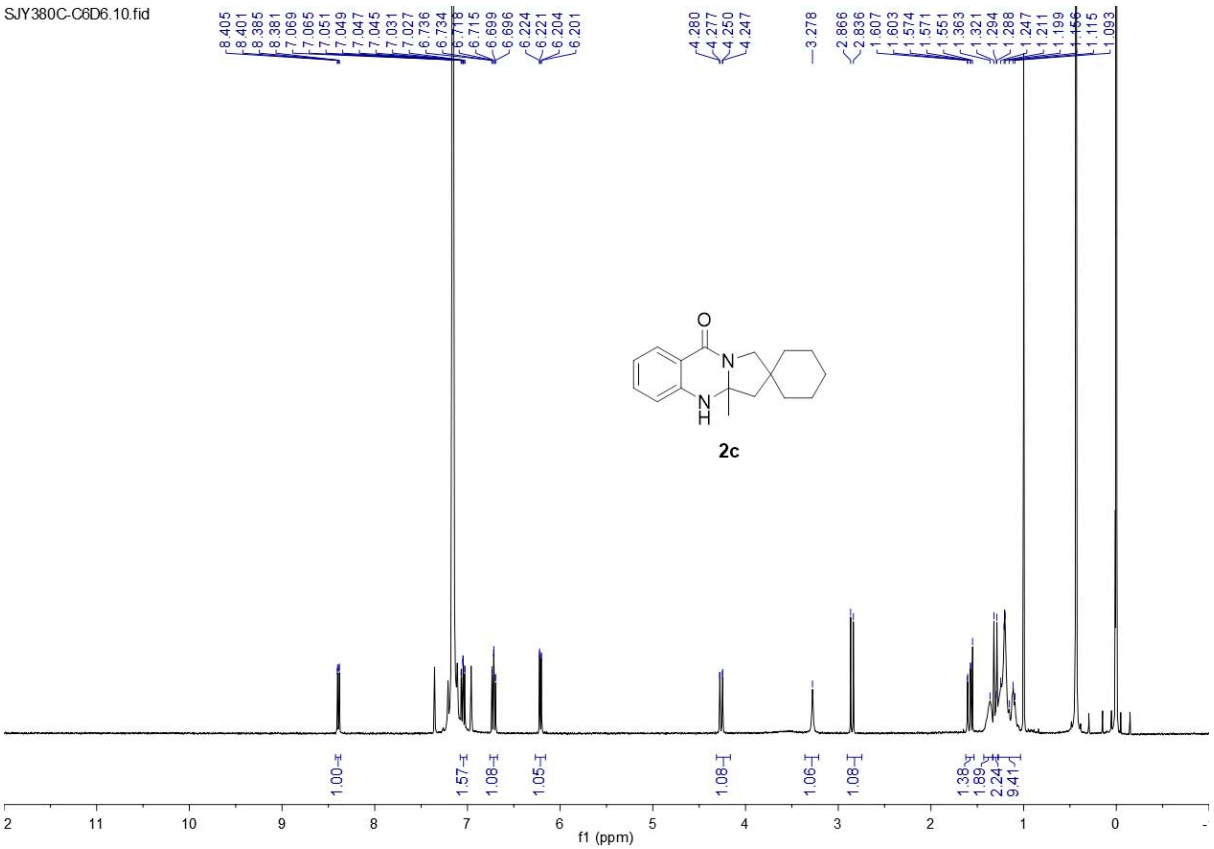
2a

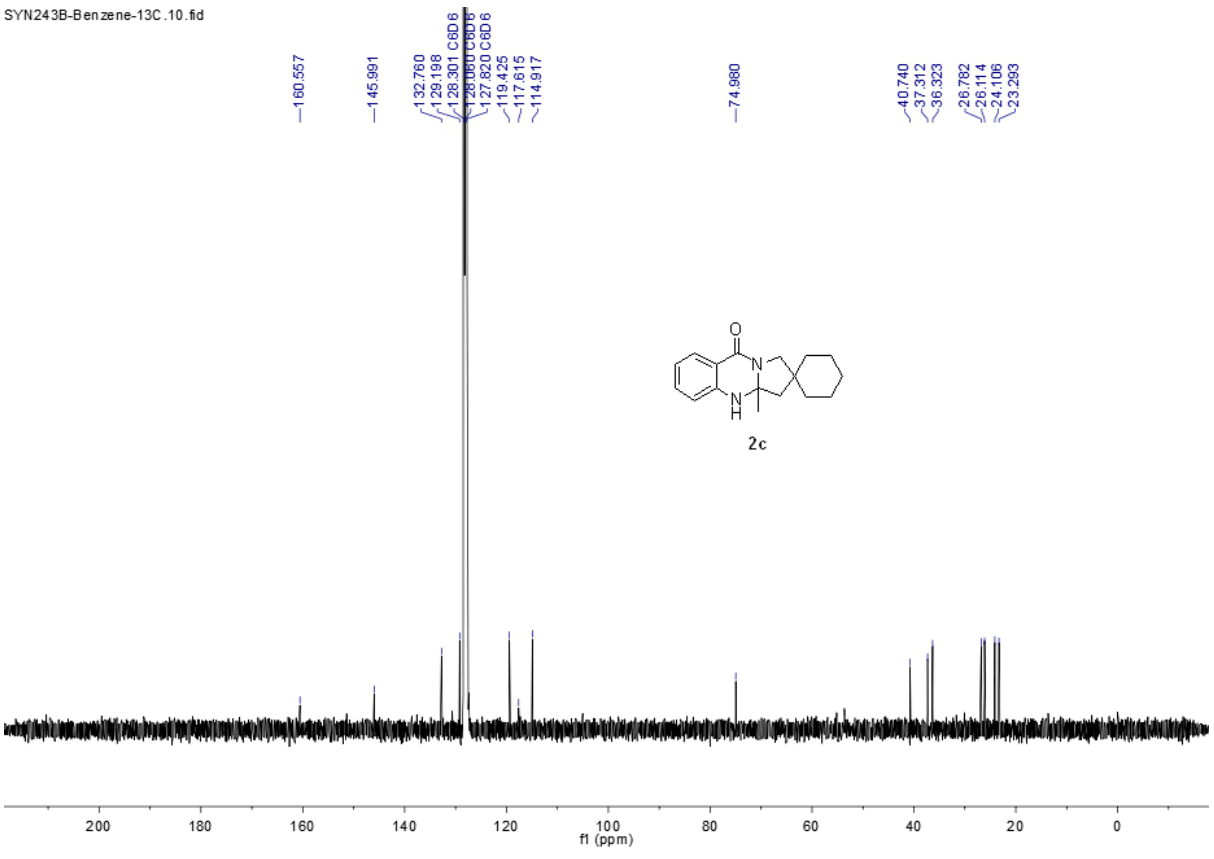

SYN188C_13C.10.fid
160.786
146.151
132.781
128.910
128.302 C6D6
128.060 C6D6
127.821 C6D6
119.425
118.061
114.978
74.996
44.419
40.584
24.869
21.041
f1 (ppm)

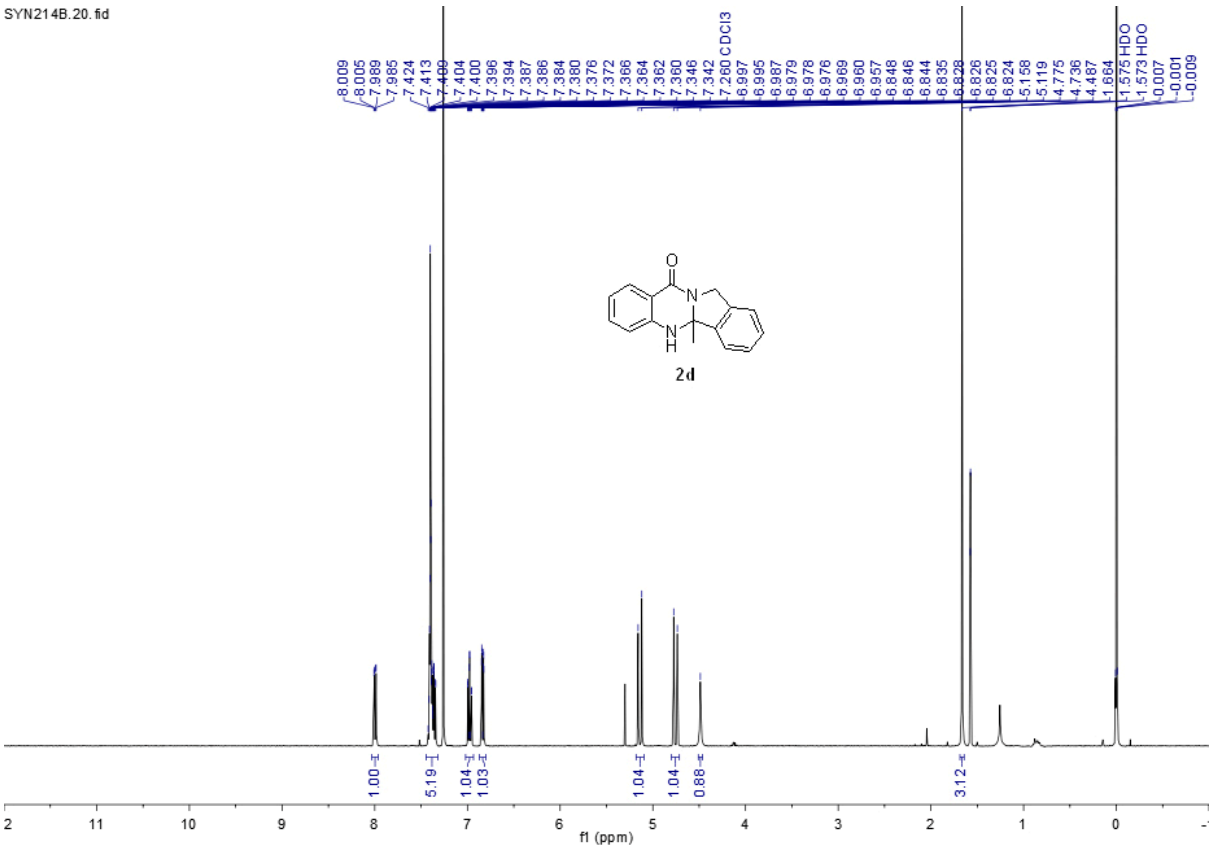
2a

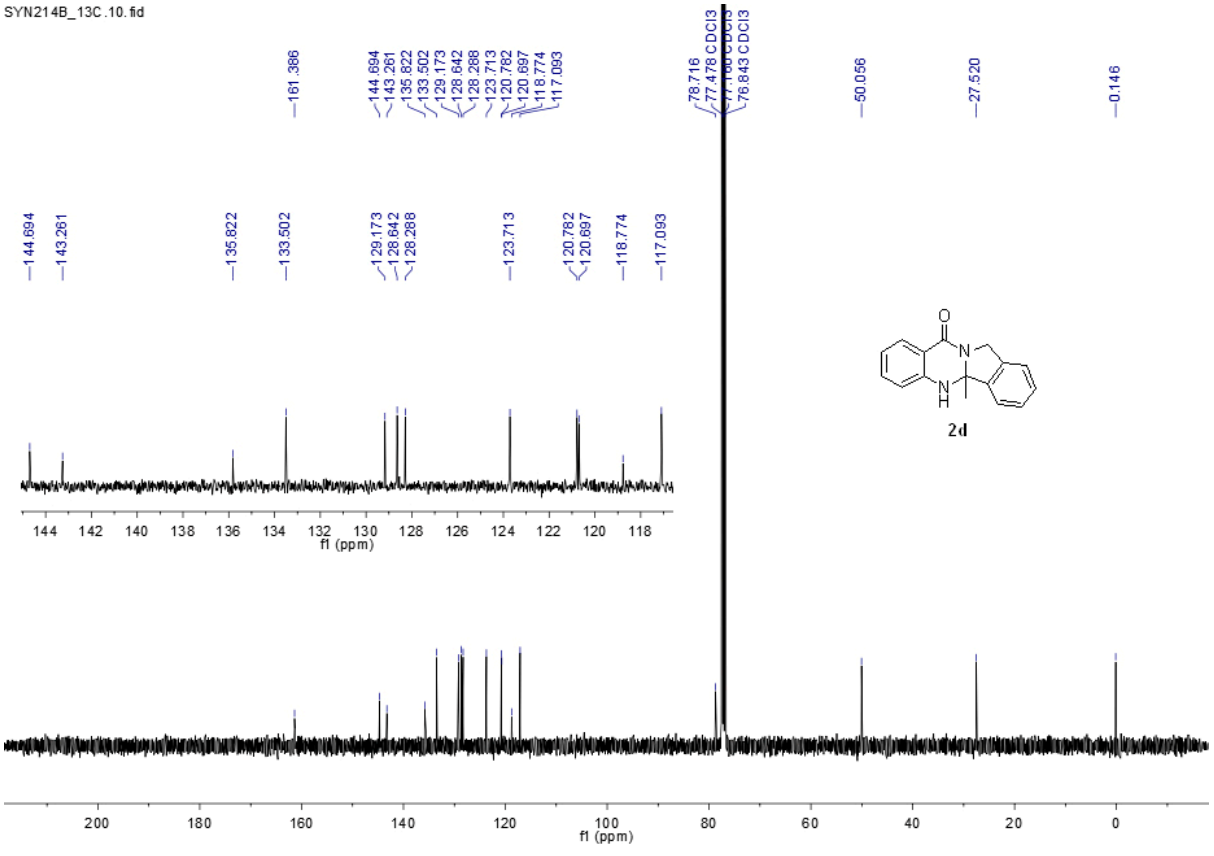

SYN225B.10.fid
7.901
7.900
7.898
7.896
7.894
7.882
7.881
7.879
7.877
7.875
7.291
7.287
7.273
7.271
7.269
7.267
7.260 CDCl3
7.260 CDCl3
7.253
7.249
6.875
6.872
6.856
6.855
6.854
6.853
6.837
6.834
6.630
6.629
6.627
6.626
6.610
6.608
6.607
6.606
4.183
3.968
3.964
3.939
3.935
3.148
3.146
3.118
3.116
2.068
2.064
2.035
2.031
1.990
1.957
1.565 HDO
1.478
1.364
1.344
1.208
1.189
0.006
0.002
-0.010
0.94
4.31
0.98
0.98
0.82
1.00
1.00
1.02
1.00
2.94
3.02
3.05
f1 (ppm)
2b

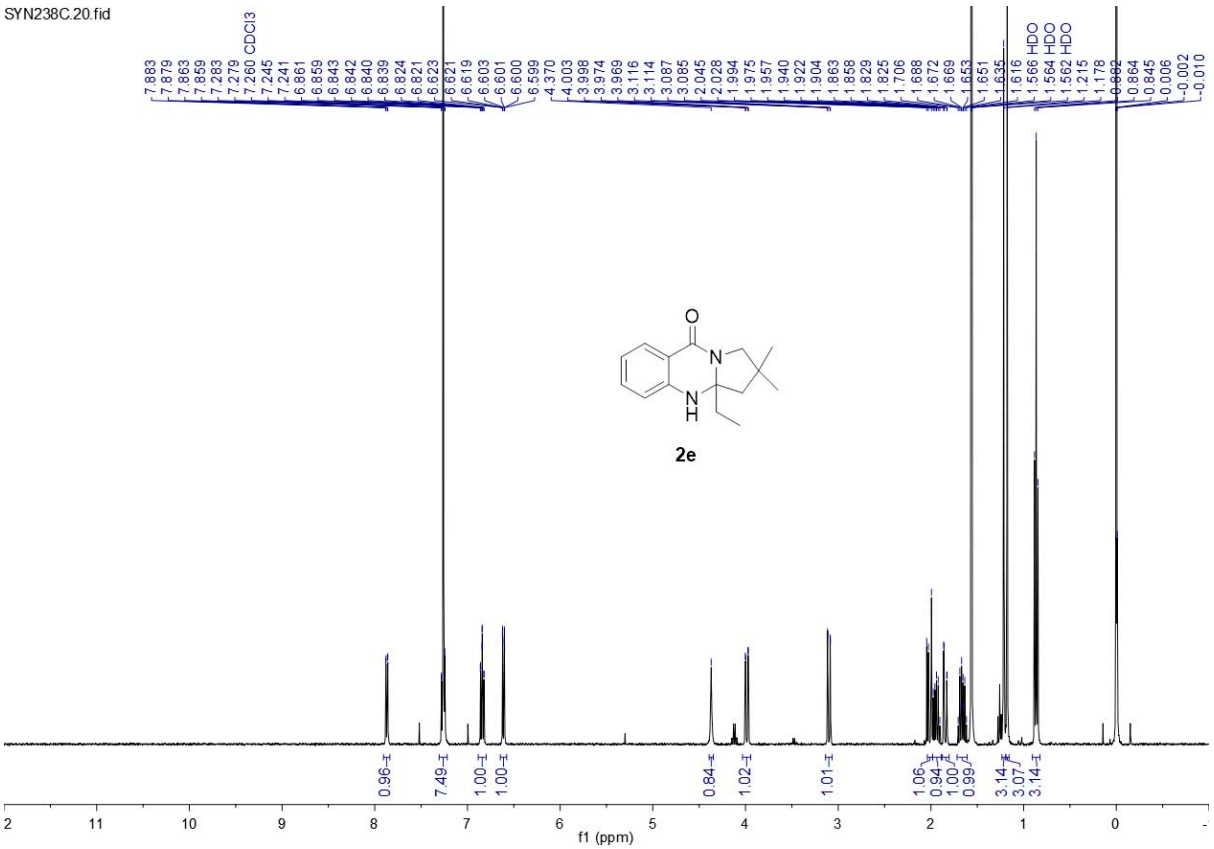

SYN225B_13C.10.fid
161.189
145.646
133.172
128.631
119.357
116.668
115.055
77.478 CDCl3
77.160 CDCl3
76.843 CDCl3
76.072
57.584
55.582
37.458
28.309
27.434
27.181
0.140
f1 (ppm)
2b


SJY380C-C6D6.10.fid
8.405
8.401
8.385
8.381
7.069
7.065
7.051
7.049
7.047
7.045
7.031
7.027
6.736
6.734
6.718
6.715
6.699
6.696
6.224
6.221
6.204
6.201
4.280
4.277
4.250
4.247
3.278
2.866
2.836
1.607
1.603
1.574
1.571
1.551
1.363
1.321
1.294
1.288
1.247
1.211
1.199
1.166
1.115
1.093
1.00
1.57
1.08
1.05
1.08
1.06
1.08
1.38
1.89
2.24
9.41
f1 (ppm)
2c

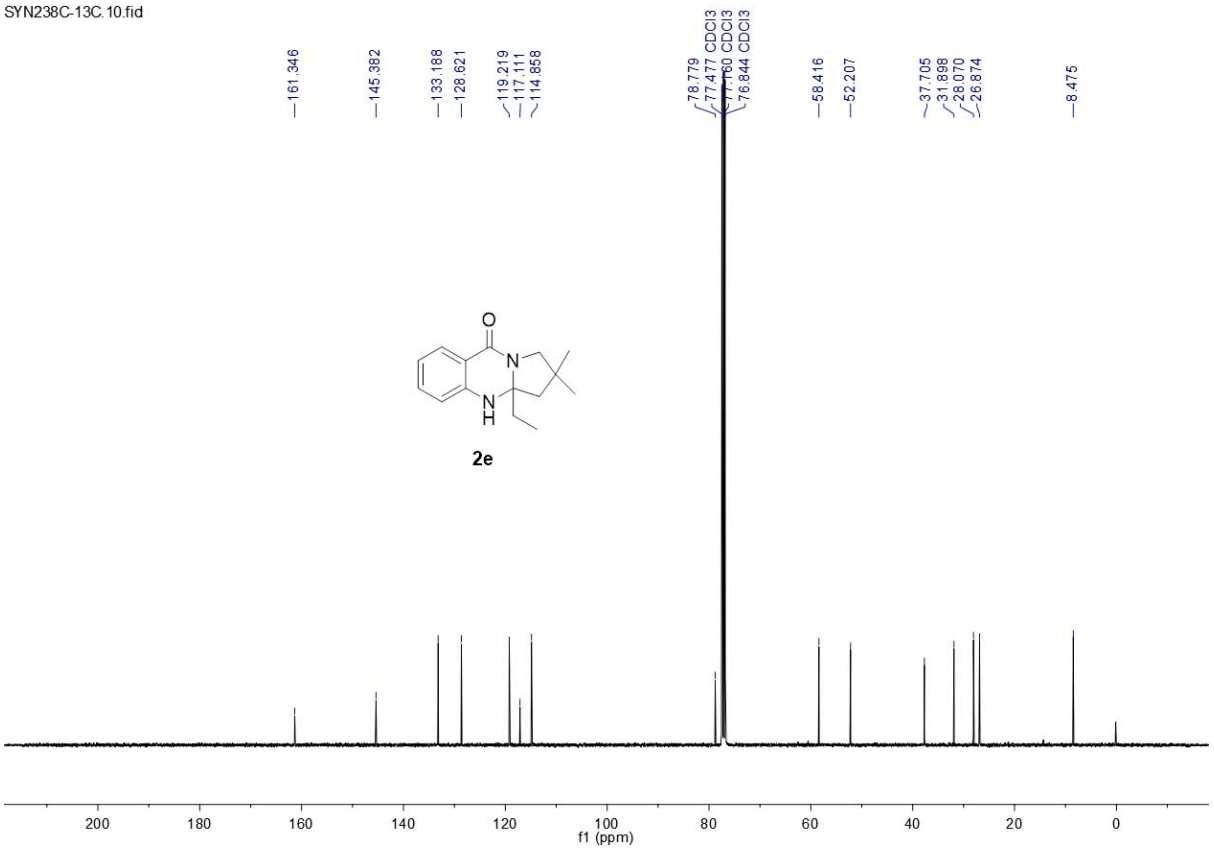

SYN243B-Benzene-13C.10.fid
160.557
145.991
132.760
129.198
128.301 C6D6
128.060 C6D6
127.820 C6D6
119.425
117.615
114.917
74.980
40.740
37.312
36.323
26.782
26.114
24.106
23.293
f1 (ppm)
2c


SYN214B.20.fid
8.009
8.005
7.989
7.985
7.424
7.413
7.409
7.404
7.400
7.396
7.394
7.387
7.386
7.384
7.380
7.376
7.372
7.366
7.364
7.362
7.360
7.346
7.342
7.260 CDCl3
6.997
6.995
6.987
6.979
6.978
6.976
6.969
6.960
6.957
6.848
6.846
6.844
6.835
6.828
6.826
6.825
6.824
5.158
5.119
4.775
4.736
4.487
1.664
1.575 HDO
1.573 HDO
0.007
-0.001
-0.009
1.00
5.19
1.04
1.03
1.04
1.04
0.88
3.12
f1 (ppm)
2d

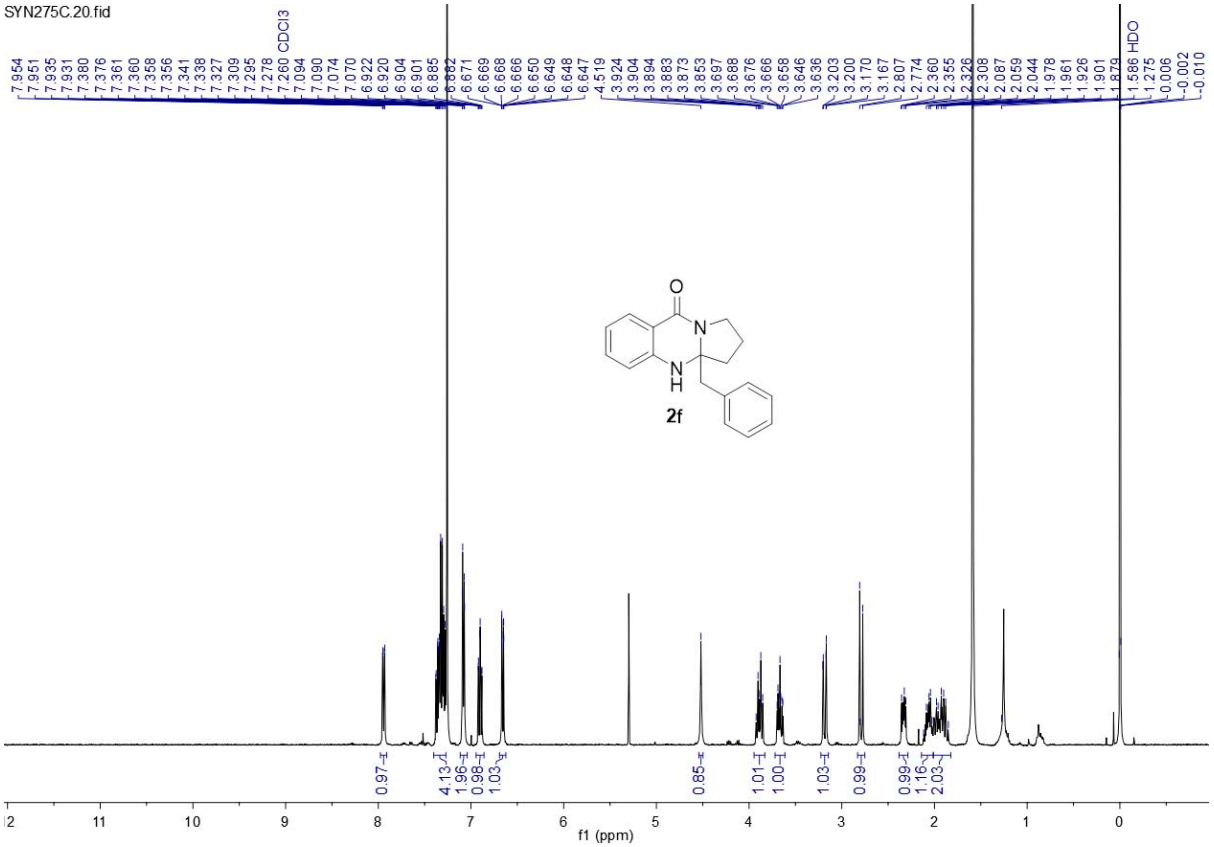

SYN214B_13C.10.fid
161.386
144.694
143.261
135.822
133.502
129.173
128.642
128.288
123.713
120.782
120.697
118.774
117.093
78.716
77.478 CDCl3
76.843 CDCl3
50.056
27.520
0.146
f1 (ppm)
2d

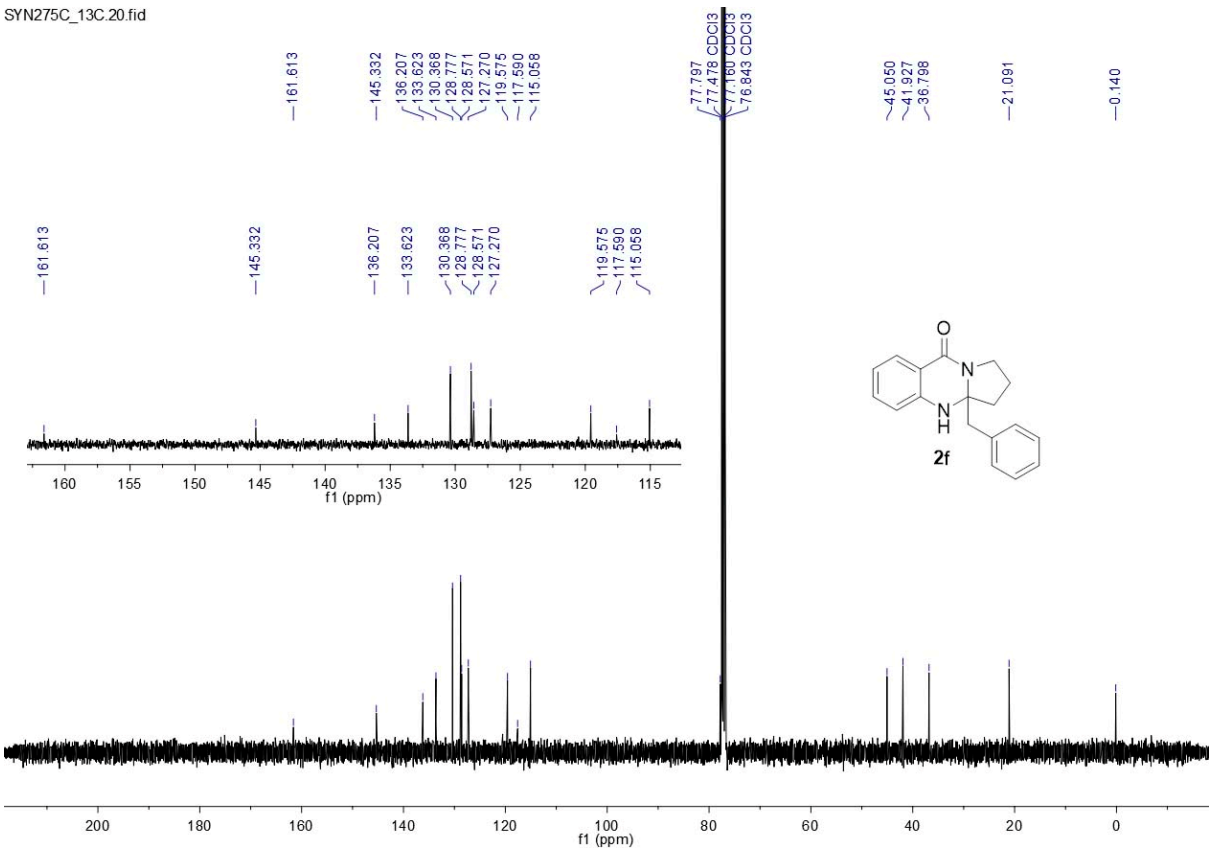

SYN238C.20.fid
7.883
7.879
7.863
7.859
7.283
7.279
7.260 CDCl3
7.245
7.241
6.861
6.859
6.843
6.842
6.840
6.839
6.824
6.821
6.623
6.621
6.619
6.603
6.601
6.600
6.599
4.370
4.003
3.998
3.974
3.969
3.116
3.114
3.087
3.085
2.045
2.028
1.994
1.975
1.957
1.940
1.922
1.904
1.863
1.858
1.829
1.825
1.706
1.688
1.672
1.669
1.653
1.651
1.635
1.616
1.566 HDO
1.564 HDO
1.562 HDO
1.215
1.178
0.882
0.864
0.845
0.006
-0.002
-0.010
0.96
7.49
1.00
1.00
0.84
1.02
1.01
1.06
0.94
1.00
0.99
3.14
3.07
3.14
f1 (ppm)

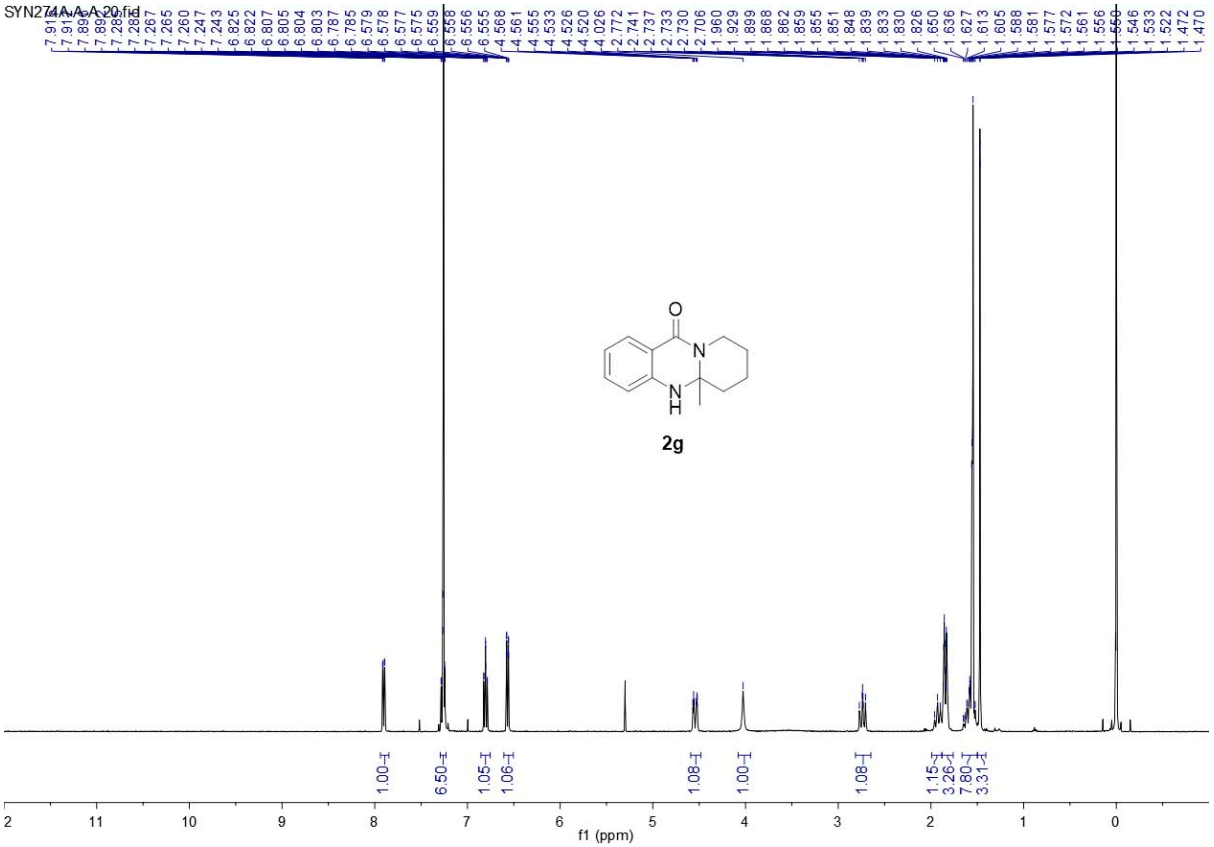
2e

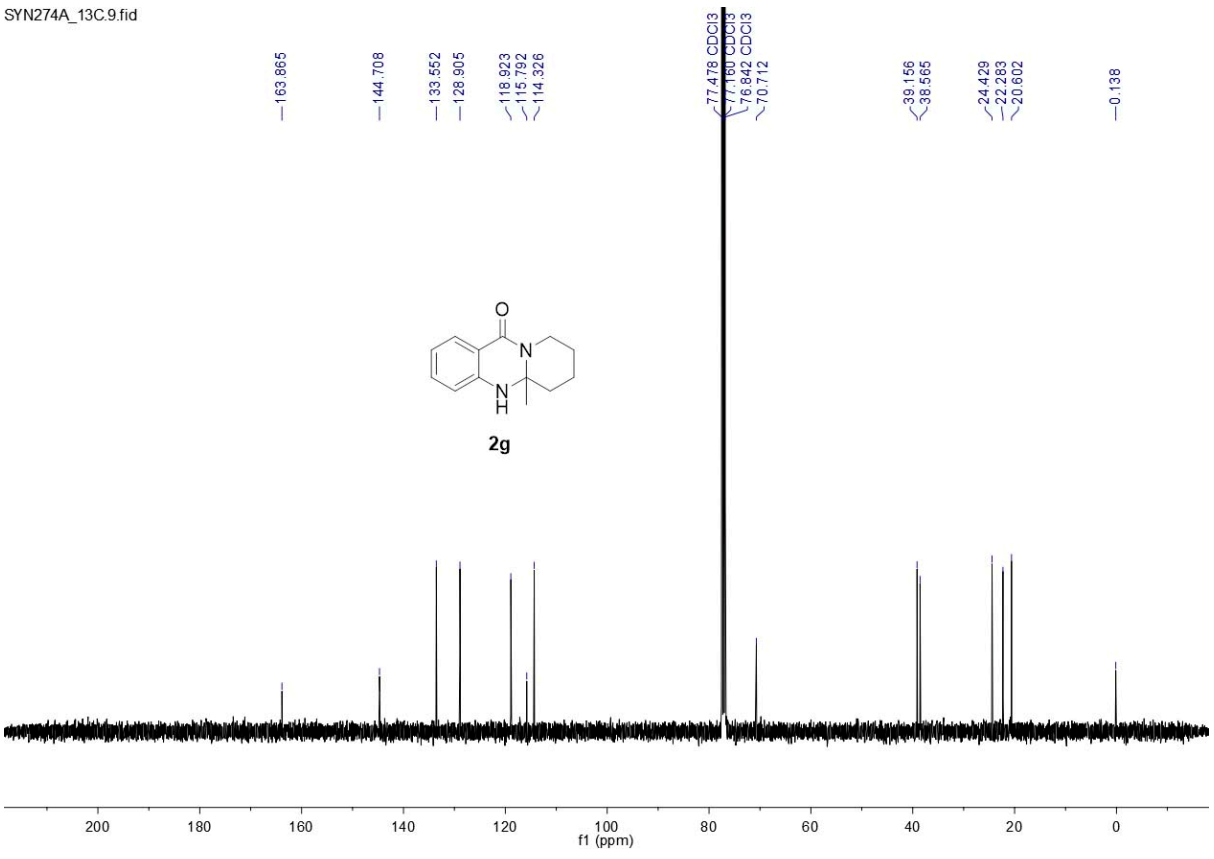

SYN238C-13C.10.fid
161.346
145.382
133.188
128.621
119.219
117.111
114.858
78.779
77.477 CDCl3
77.160 CDCl3
76.844 CDCl3
58.416
52.207
37.705
31.898
28.070
26.874
8.475
f1 (ppm)

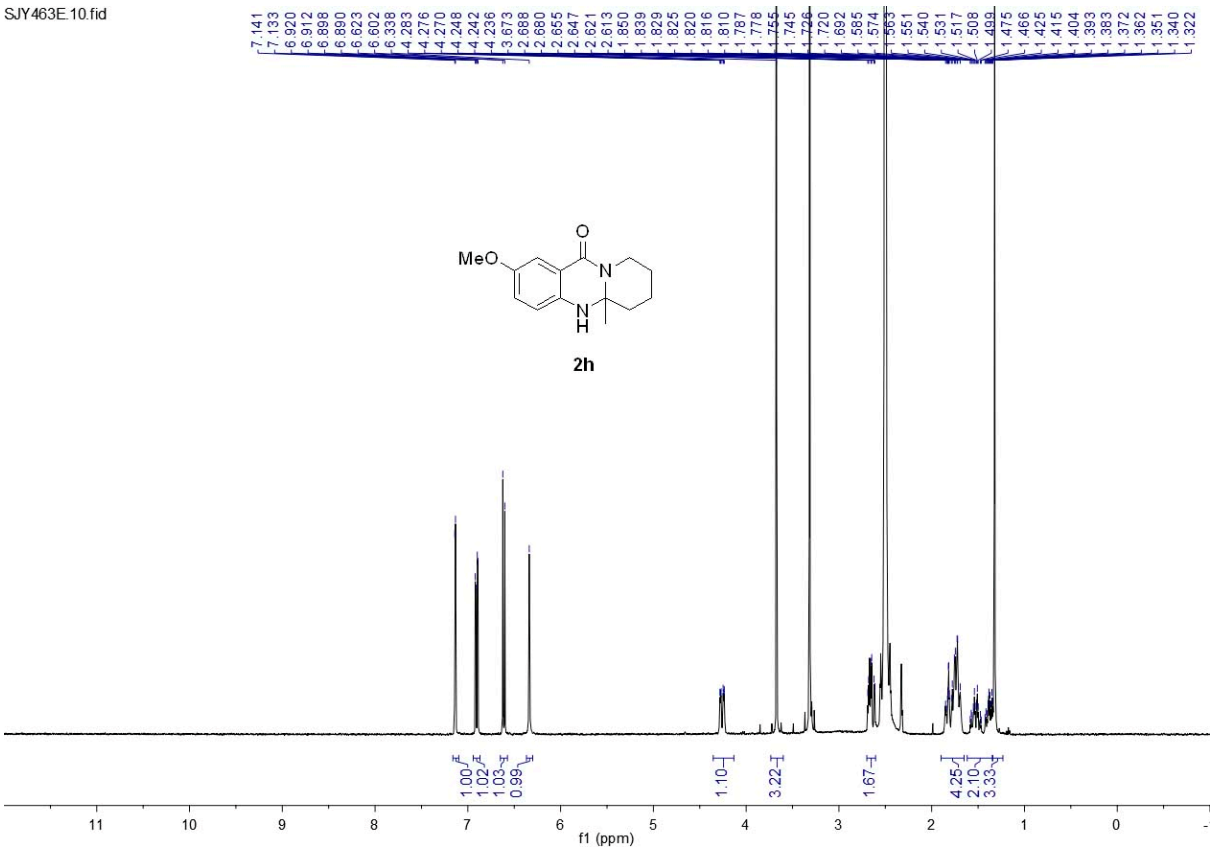
2e

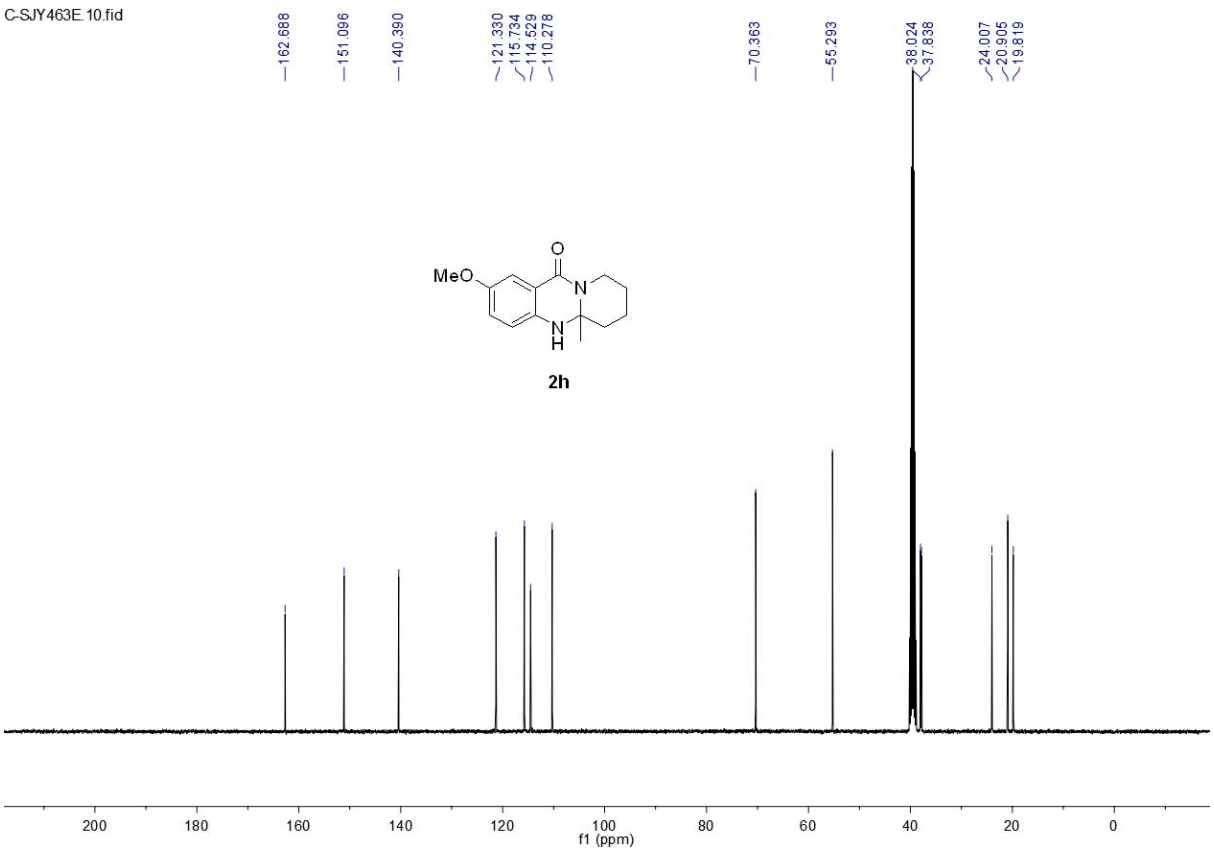

SYN275C.20.fid
7.954
7.951
7.935
7.931
7.380
7.376
7.361
7.360
7.358
7.356
7.341
7.338
7.327
7.309
7.295
7.278
7.260 CDCl3
7.094
7.090
7.074
7.070
6.922
6.920
6.904
6.901
6.885
6.882
6.671
6.669
6.668
6.666
6.650
6.649
6.648
6.647
4.519
3.924
3.904
3.894
3.883
3.873
3.853
3.697
3.688
3.676
3.666
3.658
3.646
3.636
3.203
3.200
3.170
3.167
2.807
2.774
2.360
2.355
2.326
2.308
2.087
2.059
2.044
1.978
1.961
1.926
1.901
1.879
1.586 HDO
1.275
0.006
-0.002
-0.010
0.97
4.13
1.96
0.98
1.03
0.85
1.01
1.00
1.03
0.99
0.99
1.16
2.03
f1 (ppm)
2f

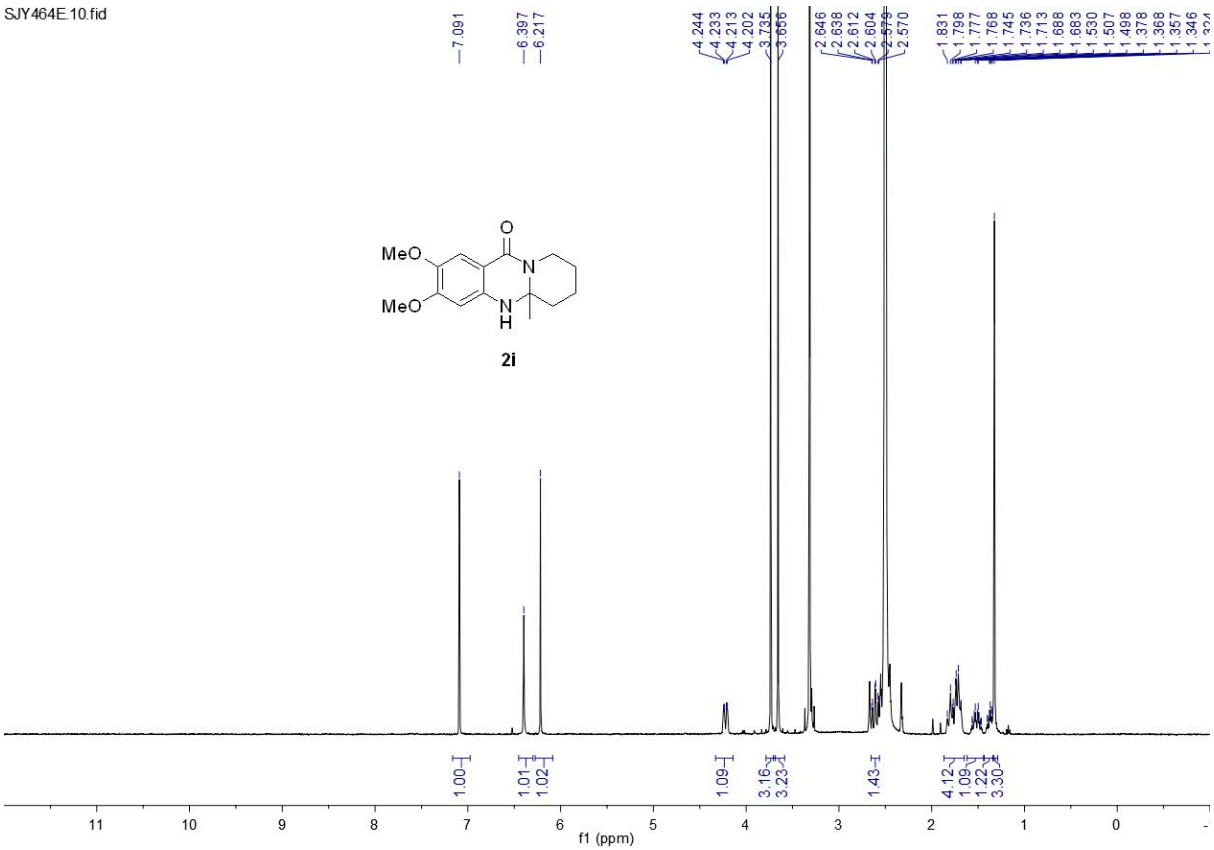

SYN275C_13C.20.fid
161.613
145.332
136.207
133.623
130.368
128.777
128.571
127.270
119.575
117.590
115.058
77.797
77.478 CDCl3
77.160 CDCl3
76.843 CDCl3
45.050
41.927
36.798
21.091
0.140
f1 (ppm)
2f

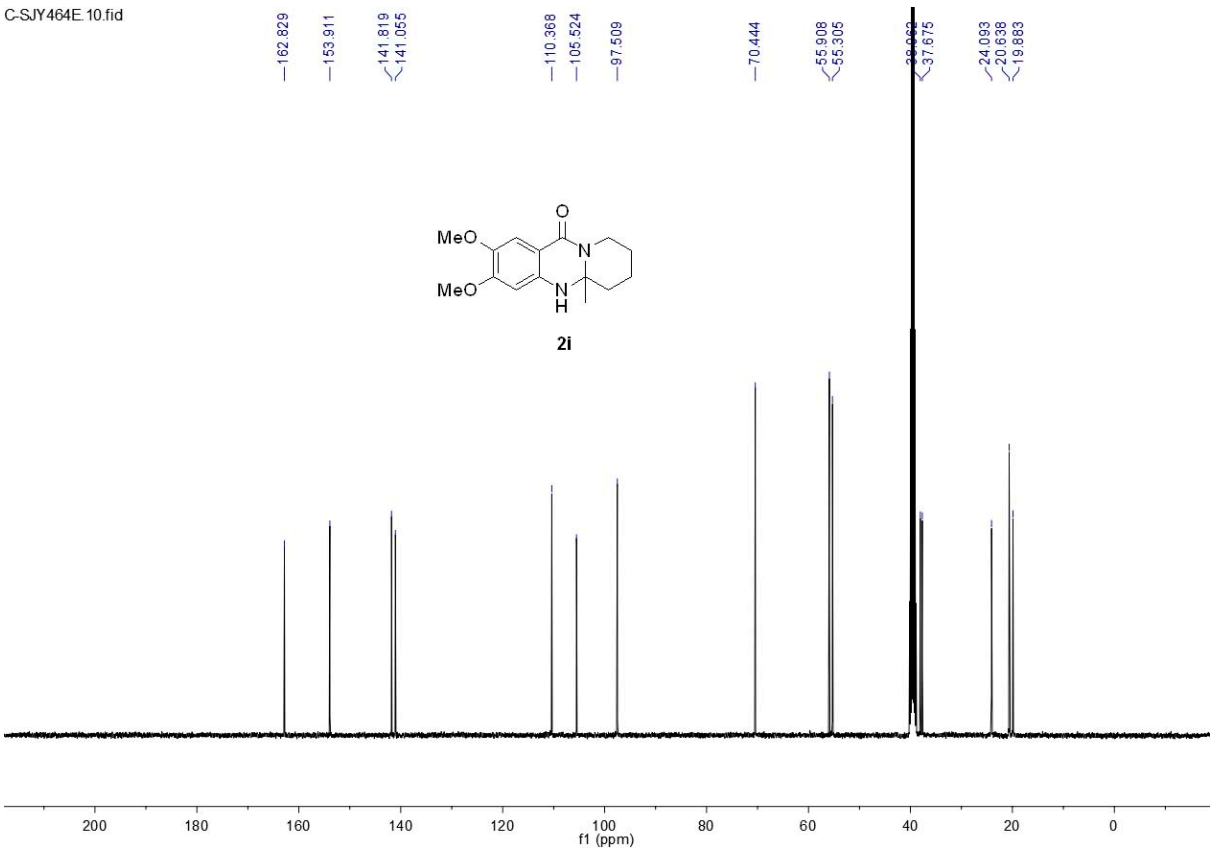

SYN274A-A-A-20.fid
2g
f1 (ppm)

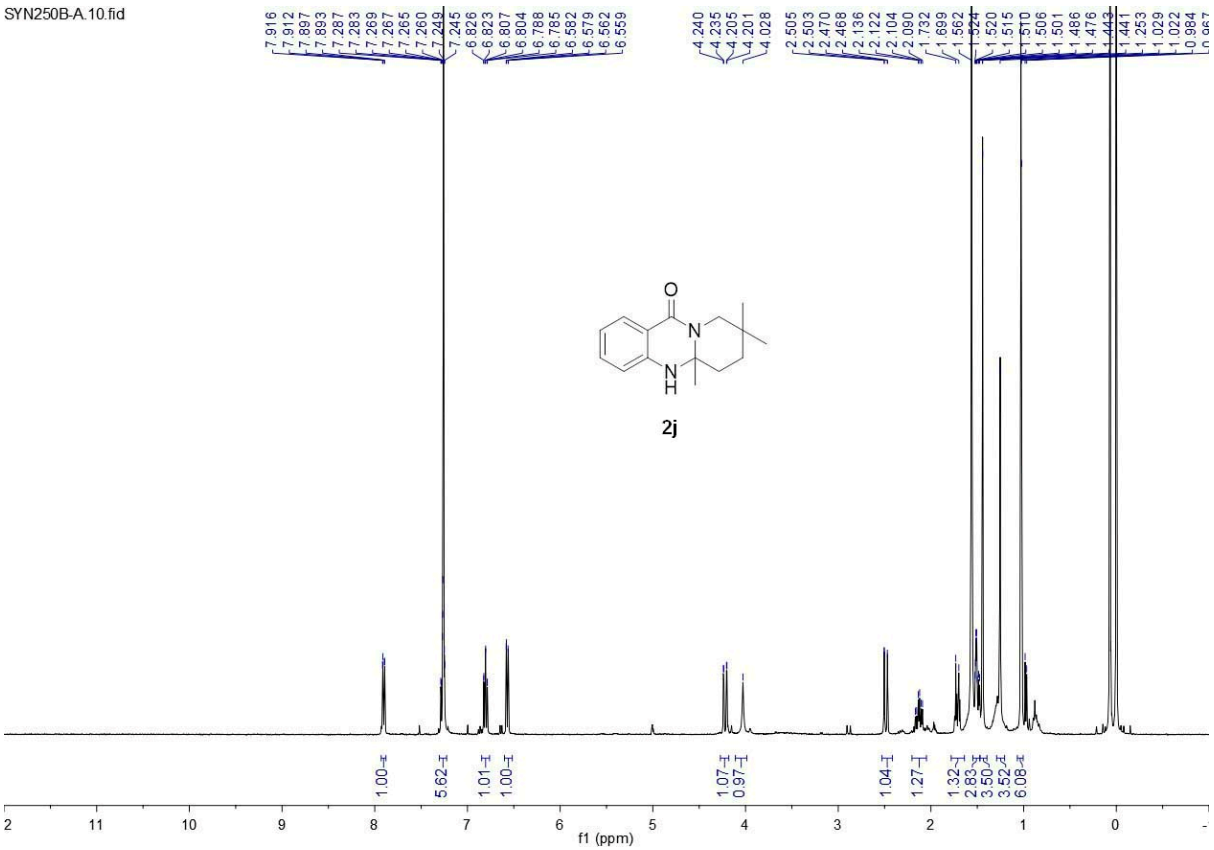

SYN274A_13C.9.fid
2g
f1 (ppm)

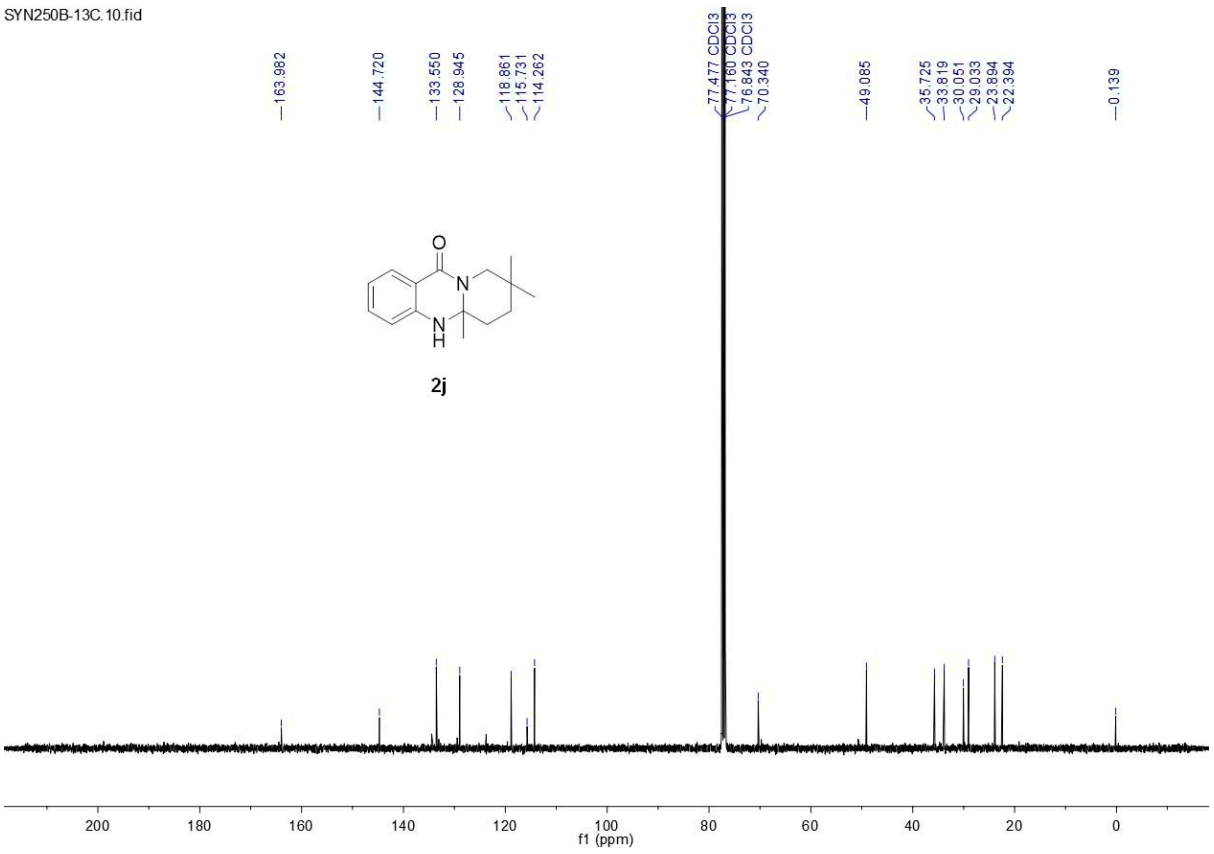

SJY463E.10.fid
MeO
O
N
N
H
2h
f1 (ppm)

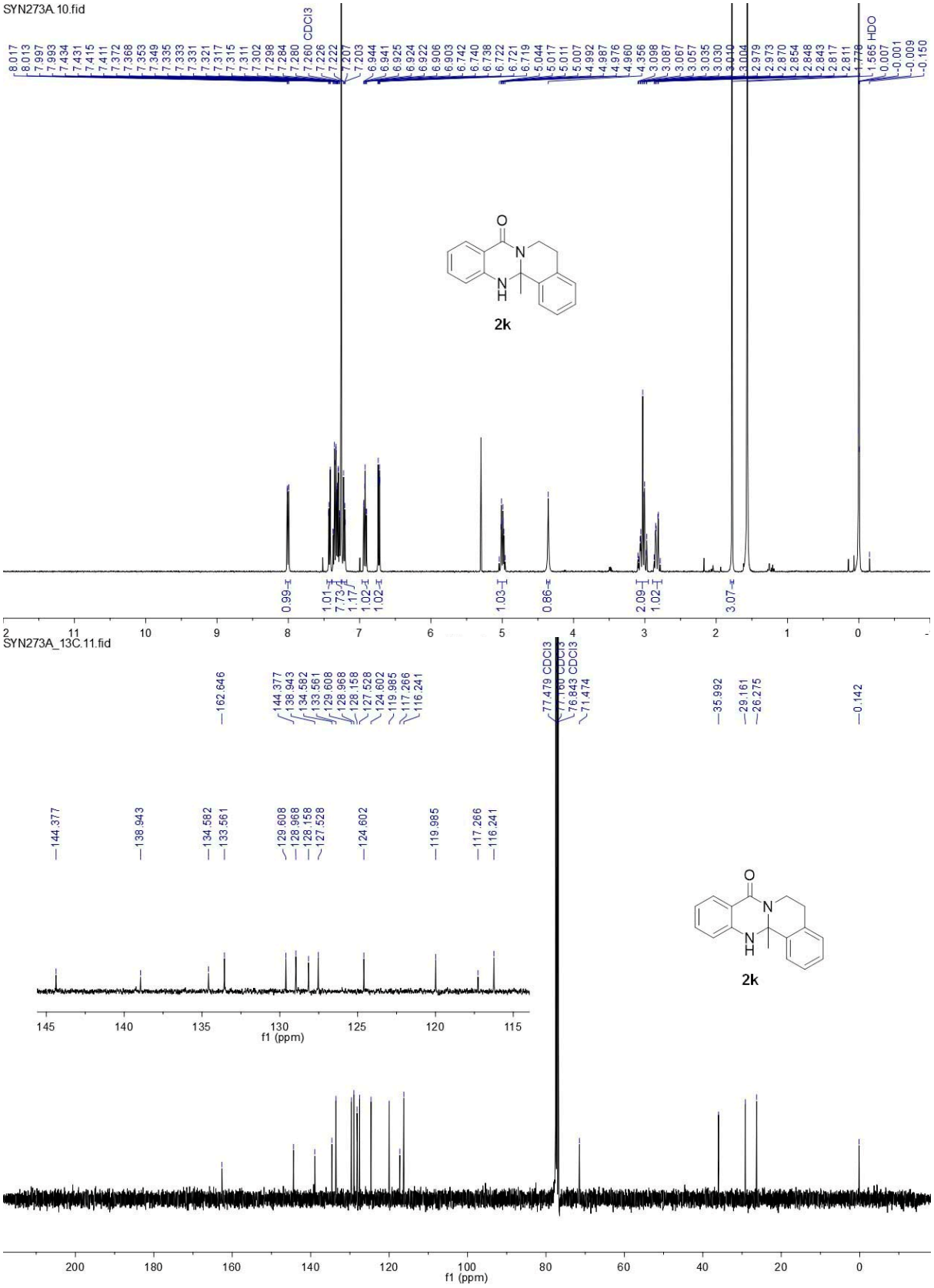

C-SJY463E.10.fid
MeO
O
N
N
H
2h
f1 (ppm)

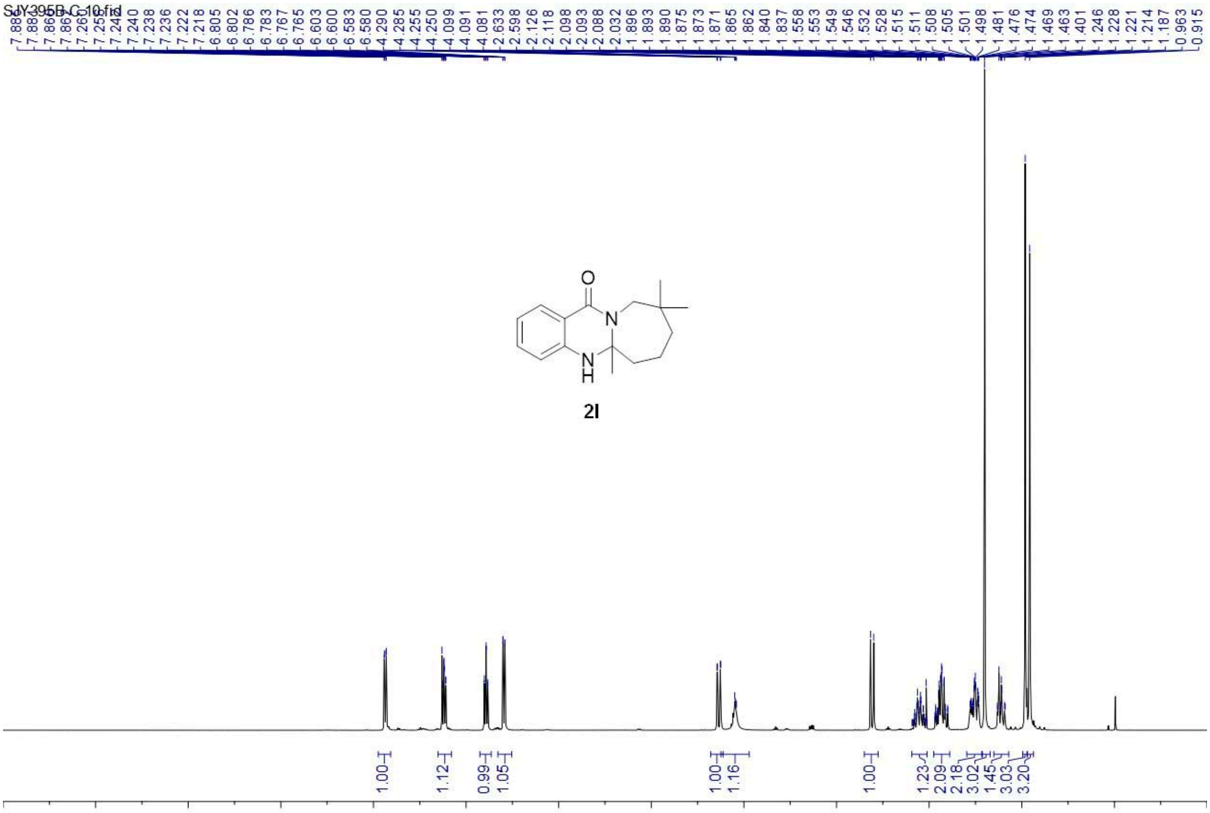

SJY464E.10.fid
7.091
6.397
6.217
4.244
4.233
4.213
4.202
3.735
3.656
2.646
2.638
2.612
2.604
2.578
2.570
1.831
1.798
1.777
1.768
1.745
1.736
1.713
1.688
1.683
1.530
1.507
1.498
1.378
1.368
1.357
1.346
MeO
MeO
O
N
N
H
2i
1.00
1.01
1.02
1.09
3.16
3.23
1.43
4.12
1.09
1.22
3.30
f1 (ppm)

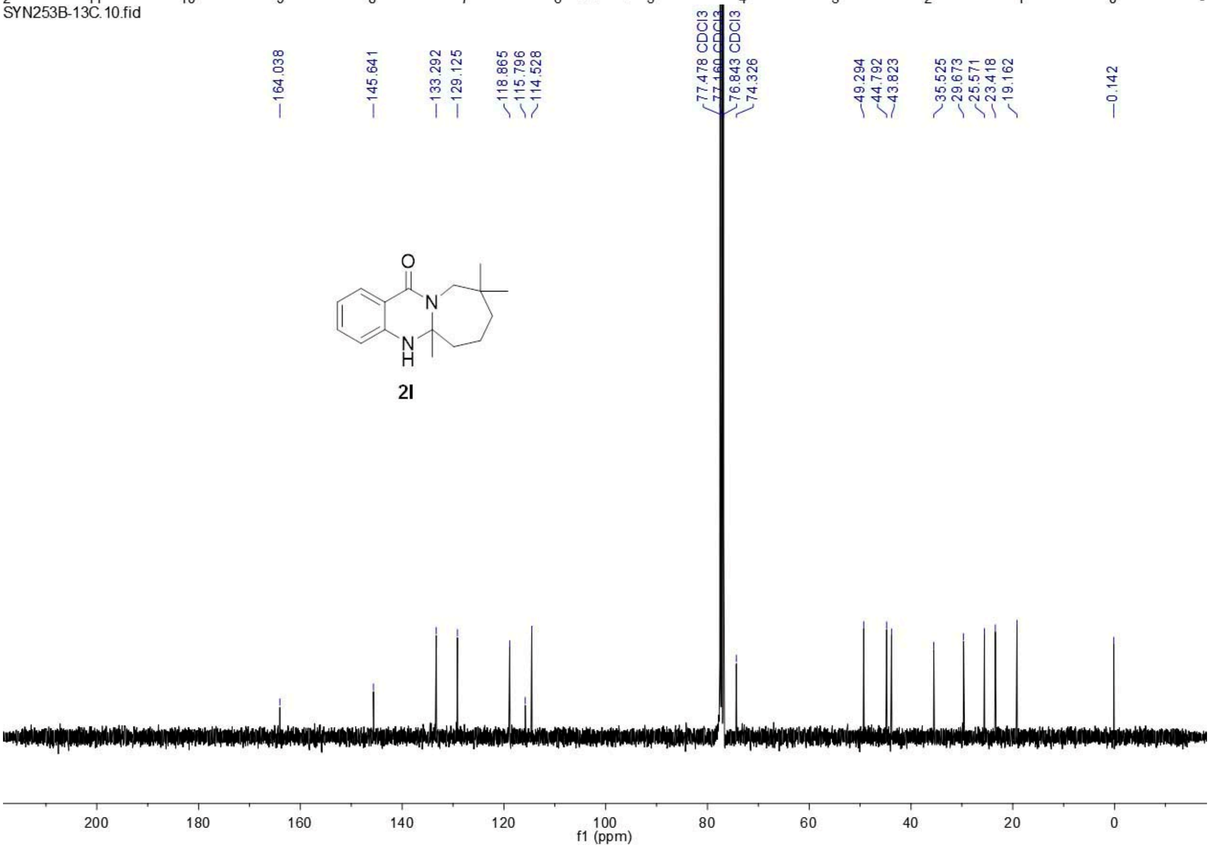

C-SJY464E.10.fid
162.829
153.911
141.819
141.055
110.368
105.524
97.509
70.444
55.908
55.305
38.062
37.675
24.093
20.638
19.883
MeO
MeO
O
N
N
H
2i
f1 (ppm)

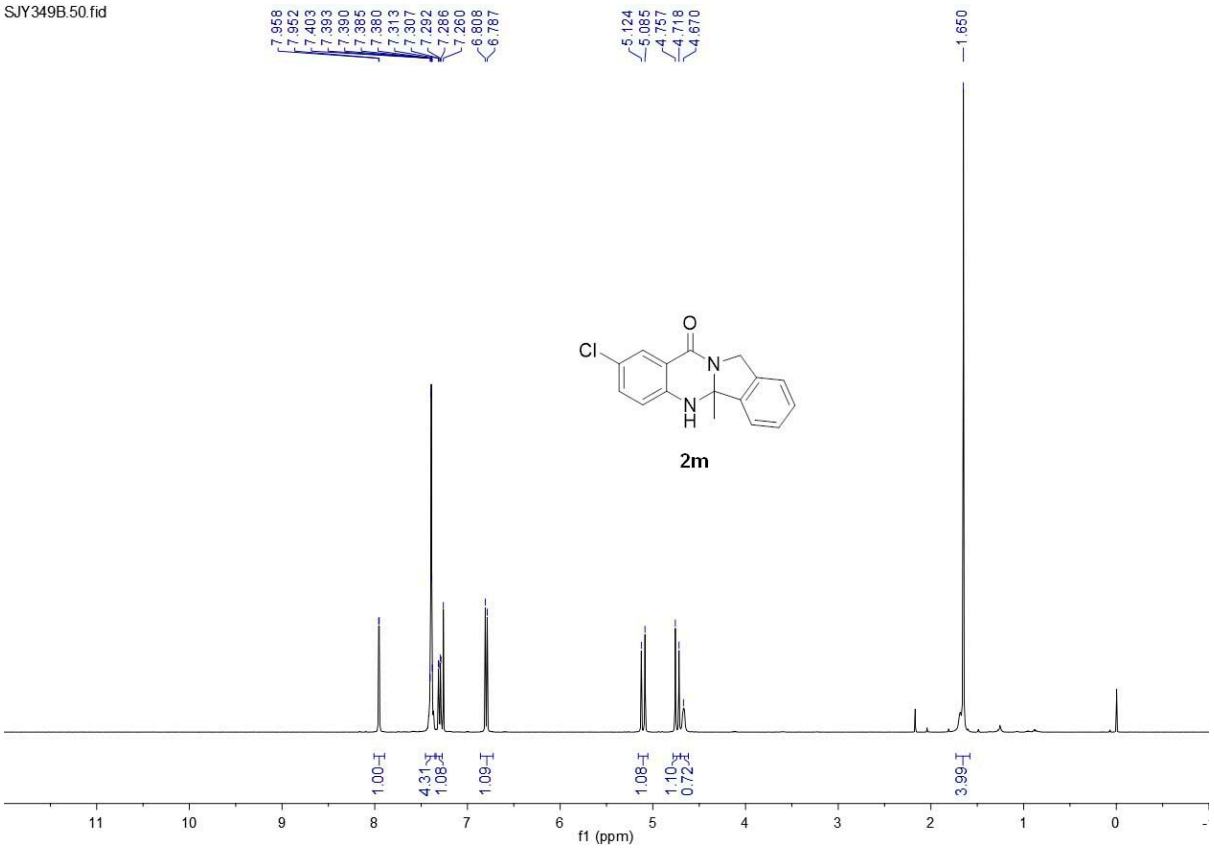

SYN250B-A.10.fid
7.916
7.912
7.897
7.893
7.287
7.283
7.269
7.267
7.265
7.260
7.249
7.245
6.826
6.823
6.807
6.804
6.788
6.785
6.582
6.579
6.562
6.559
4.240
4.235
4.205
4.201
4.028
2.505
2.503
2.470
2.468
2.136
2.122
2.104
2.090
1.732
1.699
1.562
1.524
1.520
1.515
1.510
1.506
1.501
1.486
1.476
1.443
1.441
1.253
1.029
1.022
0.984
1.00
5.62
1.01
1.00
1.07
0.97
1.04
1.27
1.32
2.83
3.50
3.52
6.08
f1 (ppm)
2j

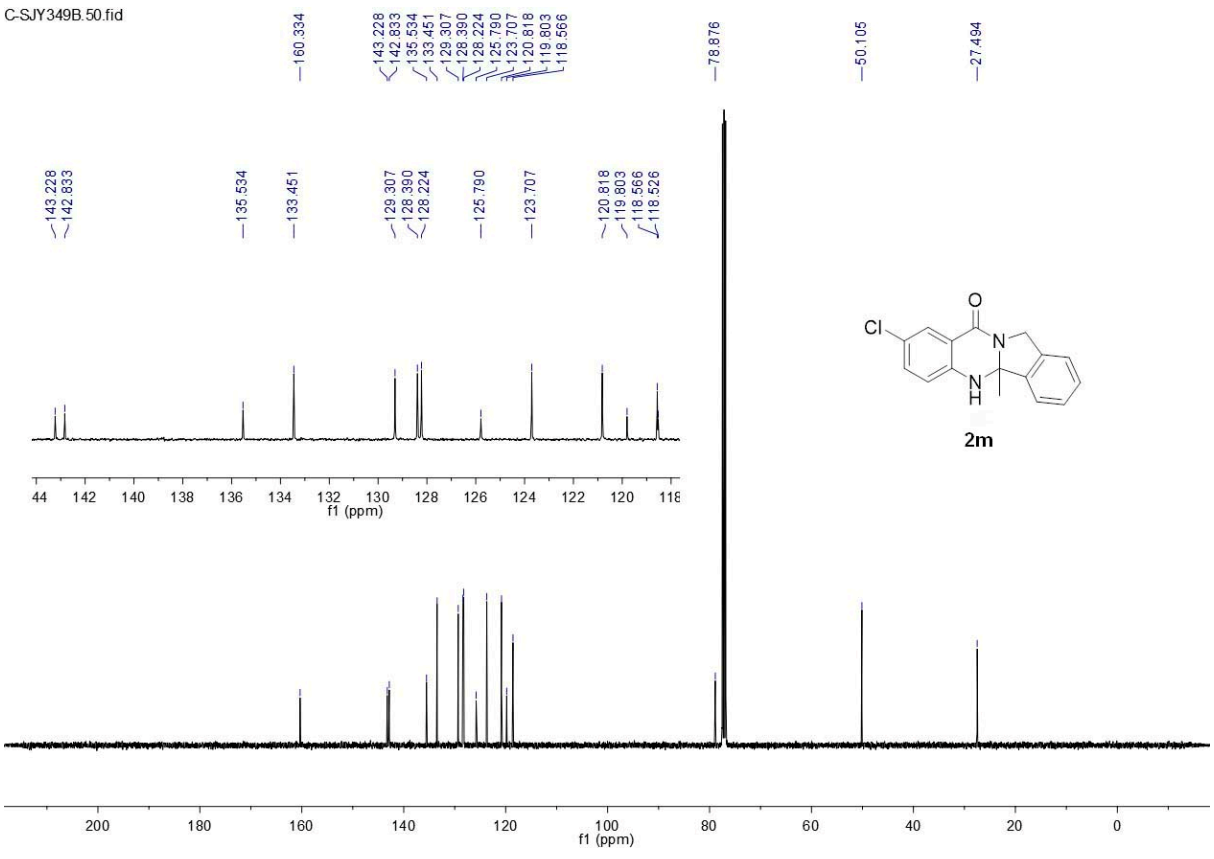

SYN250B-13C.10.fid
163.982
144.720
133.550
128.945
118.861
115.731
114.262
77.477 CDCl3
77.160 CDCl3
76.843 CDCl3
70.340
49.085
35.725
33.819
30.051
29.033
23.894
22.394
0.139
f1 (ppm)
2j

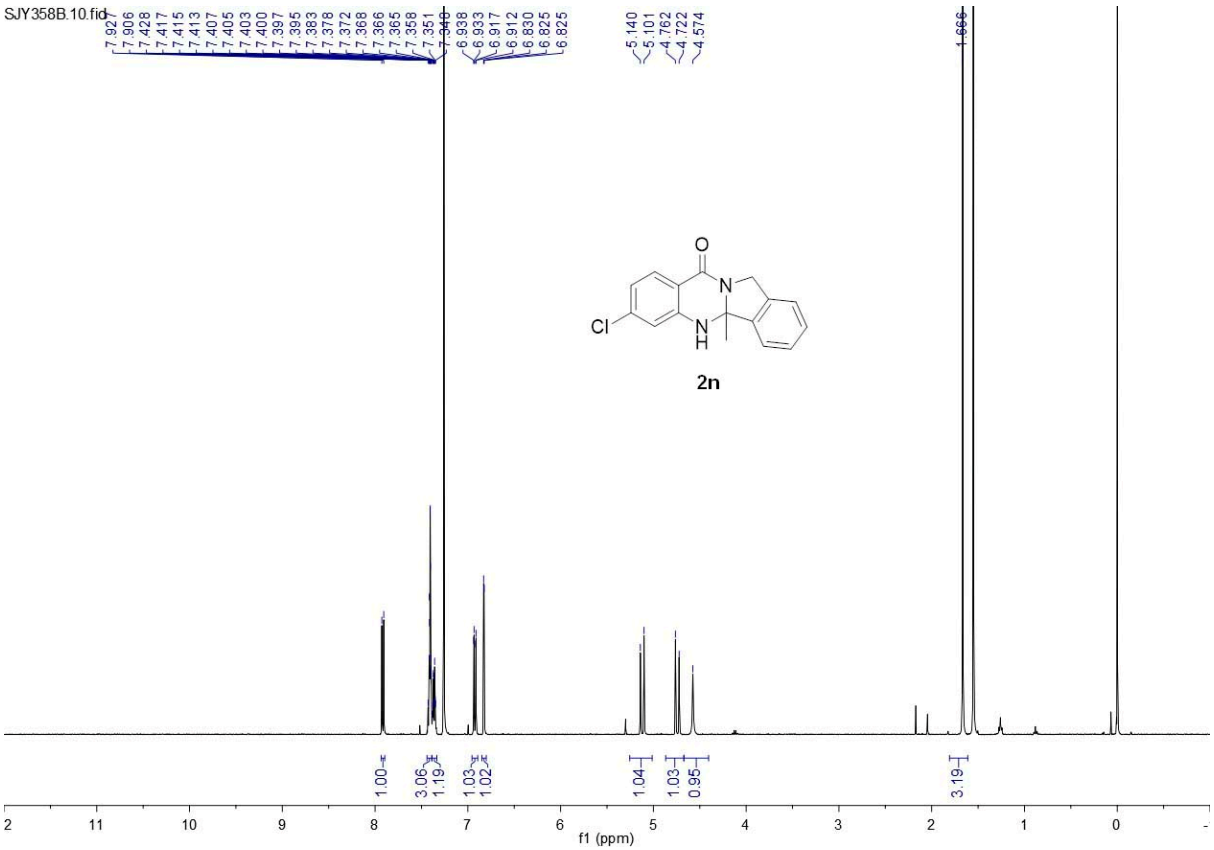

SYN273A.10.fid
CDCl3
HDO
2k
SYN273A_13C.11.fid
CDCl3
f1 (ppm)
2k
f1 (ppm)

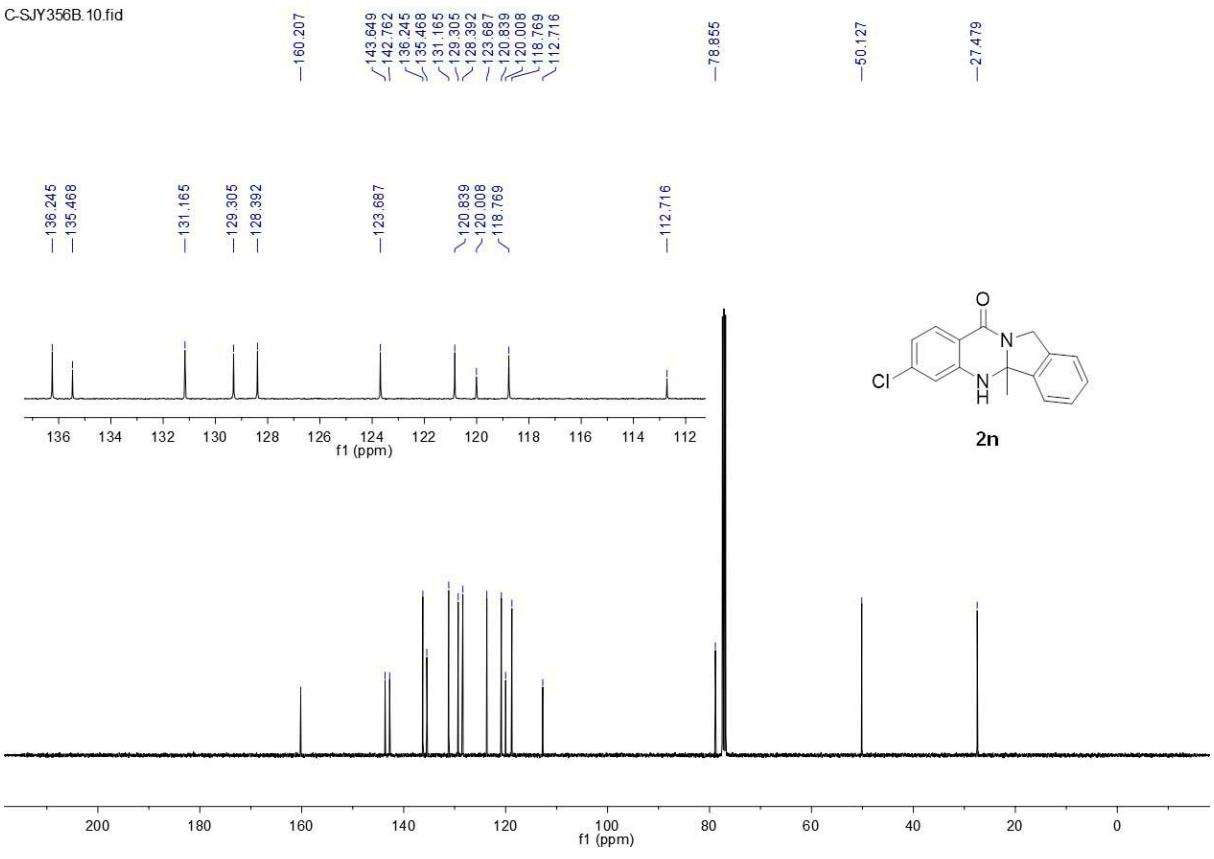

7.240
7.238
7.236
7.222
7.218
6.805
6.802
6.786
6.783
6.767
6.765
6.603
6.600
6.583
6.580
4.290
4.285
4.255
4.250
4.099
4.091
4.081
2.633
2.598
2.126
2.118
2.098
2.093
2.088
2.032
1.896
1.893
1.890
1.875
1.873
1.871
1.865
1.862
1.840
1.837
1.558
1.553
1.549
1.546
1.532
1.528
1.515
1.511
1.508
1.505
1.501
1.498
1.481
1.476
1.474
1.469
1.463
1.401
1.246
1.228
1.221
1.214
1.187
0.963
0.915
2l
1.00
1.12
0.99
1.05
1.00
1.16
1.00
1.23
2.09
2.18
3.02
1.45
3.03
3.20
11
10
9
8
7
6
5
4
3
2
1
0

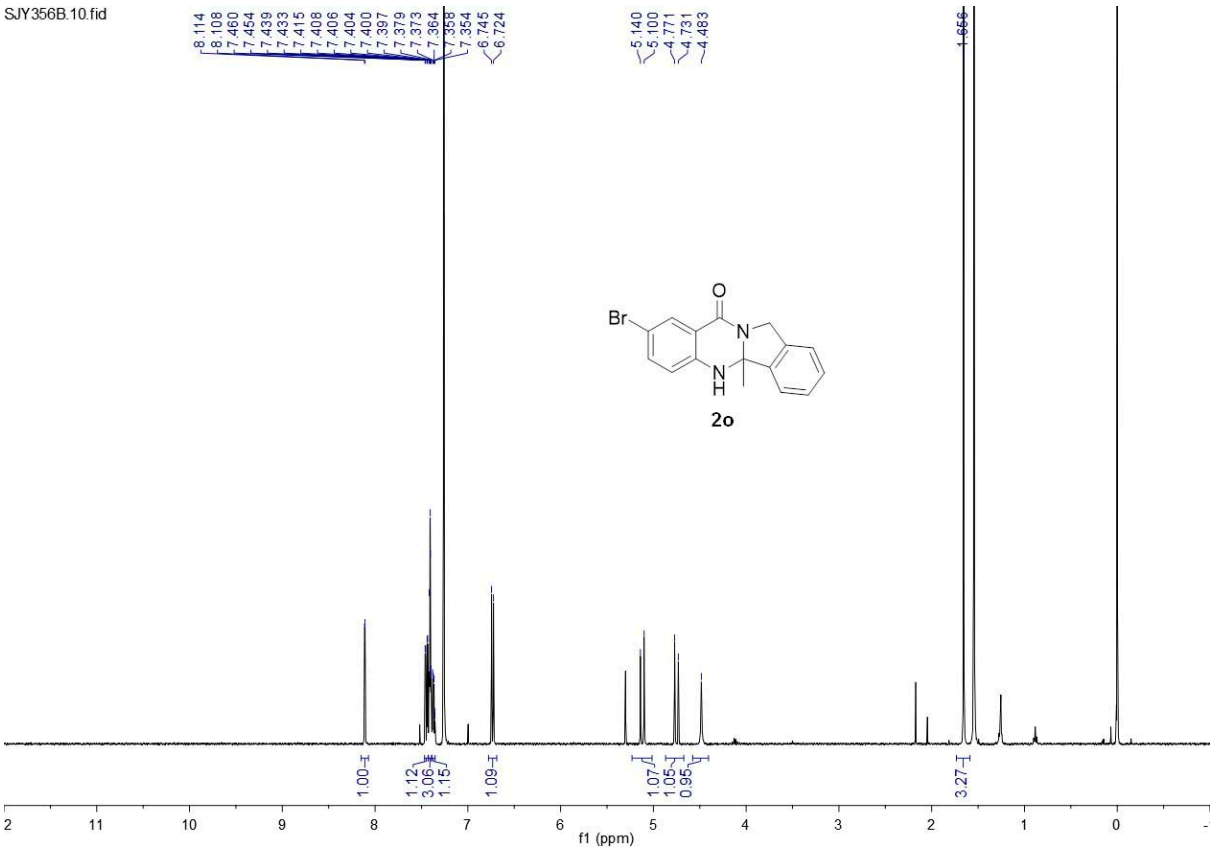

SYN253B-13C.10.fid
164.038
145.641
133.292
129.125
118.865
115.796
114.528
77.478 CDCl3
76.843 CDCl3
74.326
49.294
44.792
43.823
35.525
29.673
25.571
23.418
19.162
0.142
2l
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

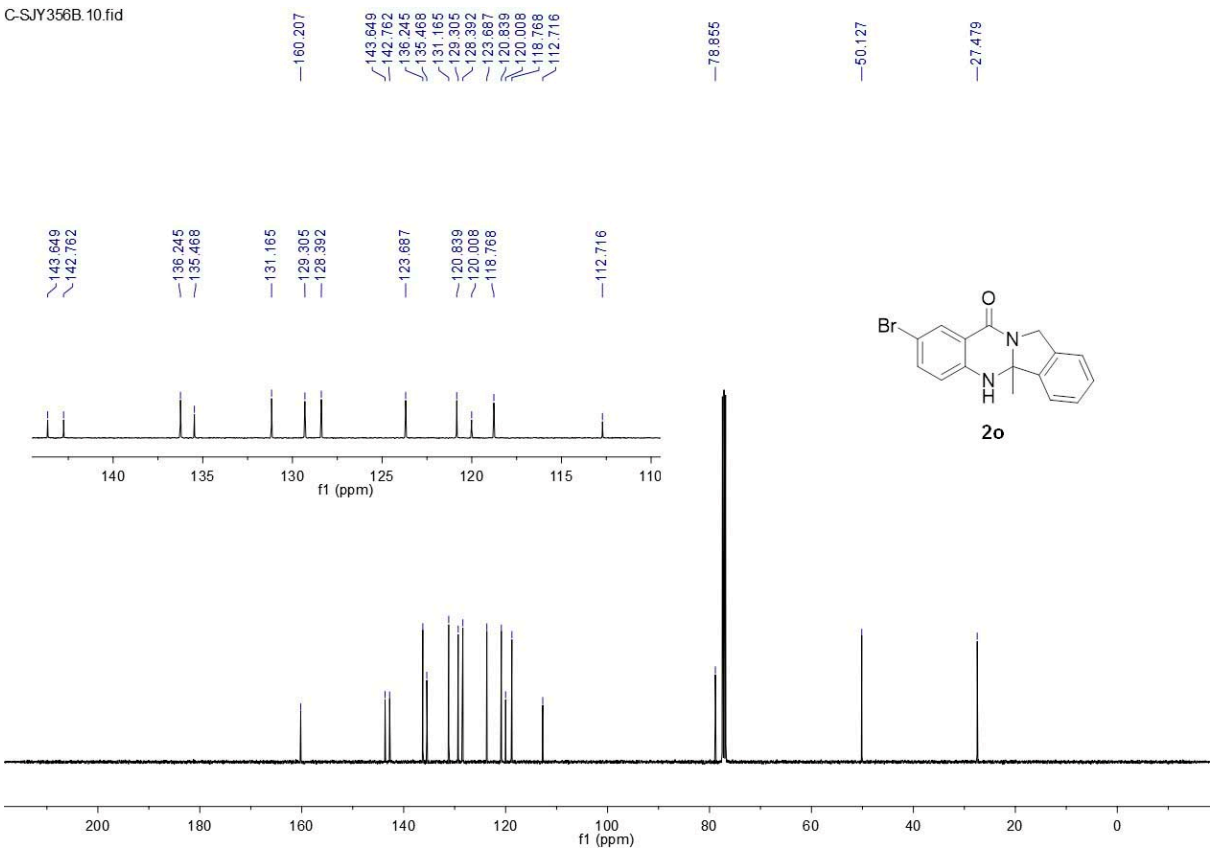

SJY349B.50.fid
7.958
7.952
7.403
7.393
7.390
7.385
7.380
7.313
7.307
7.292
7.286
7.260
6.808
6.787
5.124
5.085
4.757
4.718
4.670
1.650
Cl
O
N
N
H
2m
1.00
4.31
1.08
1.09
1.08
1.10
0.72
3.99
f1 (ppm)

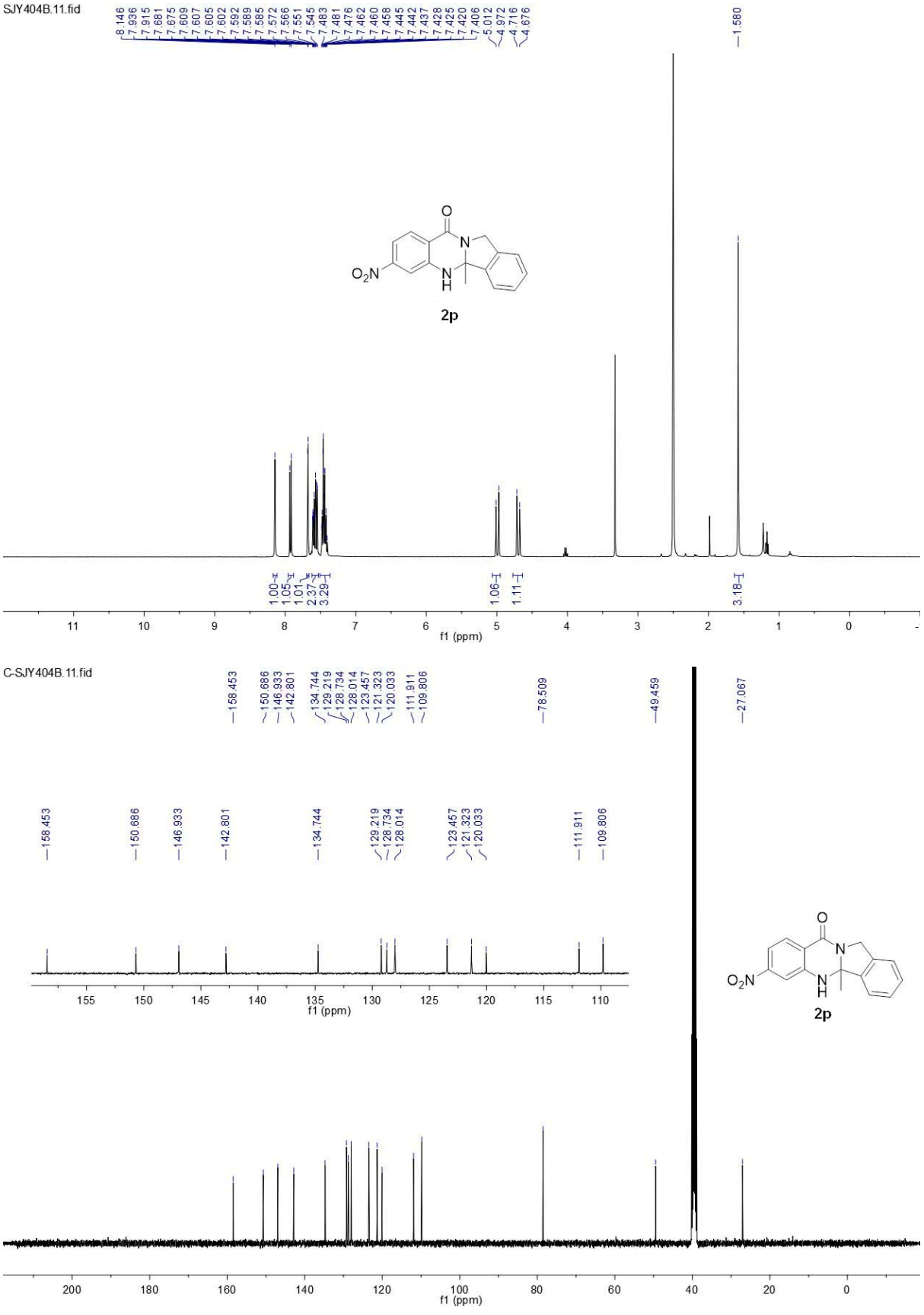

C-SJY349B.50.fid
160.334
143.228
142.833
135.534
133.451
129.307
128.390
128.224
125.790
123.707
120.818
119.803
118.566
78.876
50.105
27.494
118.526
Cl
O
N
N
H
2m
f1 (ppm)

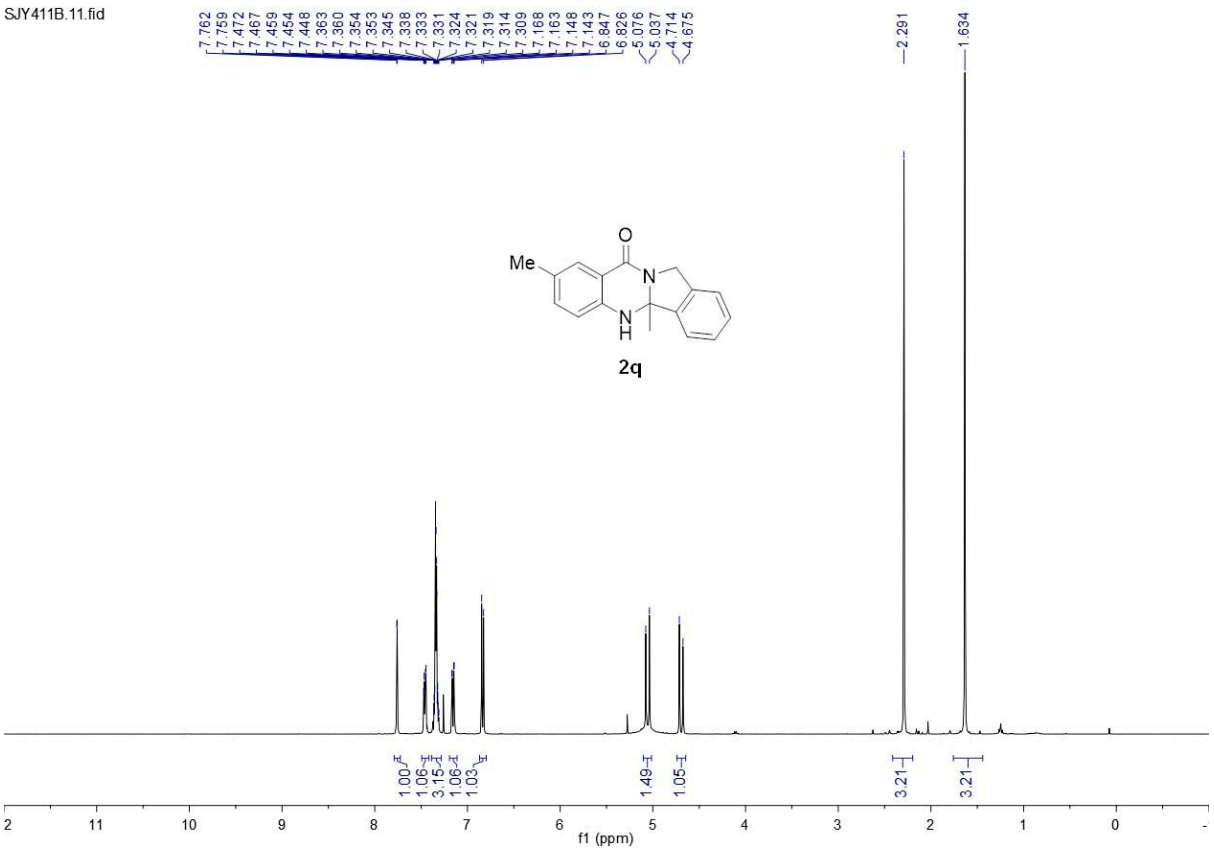

SJY358B.10.fid
7.927
7.906
7.428
7.417
7.415
7.413
7.407
7.405
7.403
7.400
7.397
7.395
7.383
7.378
7.372
7.368
7.366
7.365
7.358
7.351
7.340
6.938
6.933
6.917
6.912
6.830
6.825
5.140
5.101
4.762
4.722
4.574
1.666
1.00
3.06
1.19
1.03
1.02
1.04
1.03
0.95
3.19
f1 (ppm)
O
N
Cl
N
H
2n

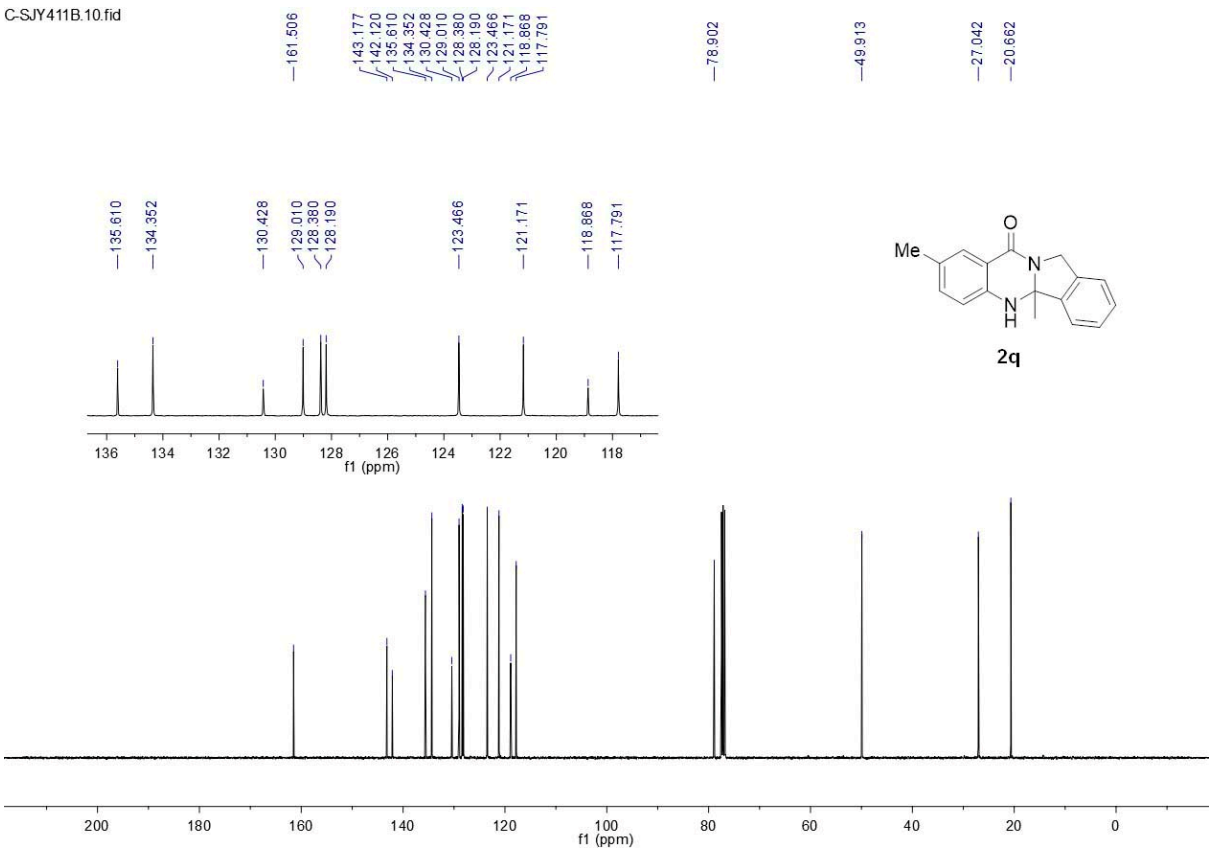

C-SJY356B.10.fid
160.207
143.649
142.762
136.245
135.468
131.165
129.305
128.392
123.687
120.839
120.008
118.769
112.716
78.855
50.127
27.479
f1 (ppm)
O
N
Cl
N
H
2n

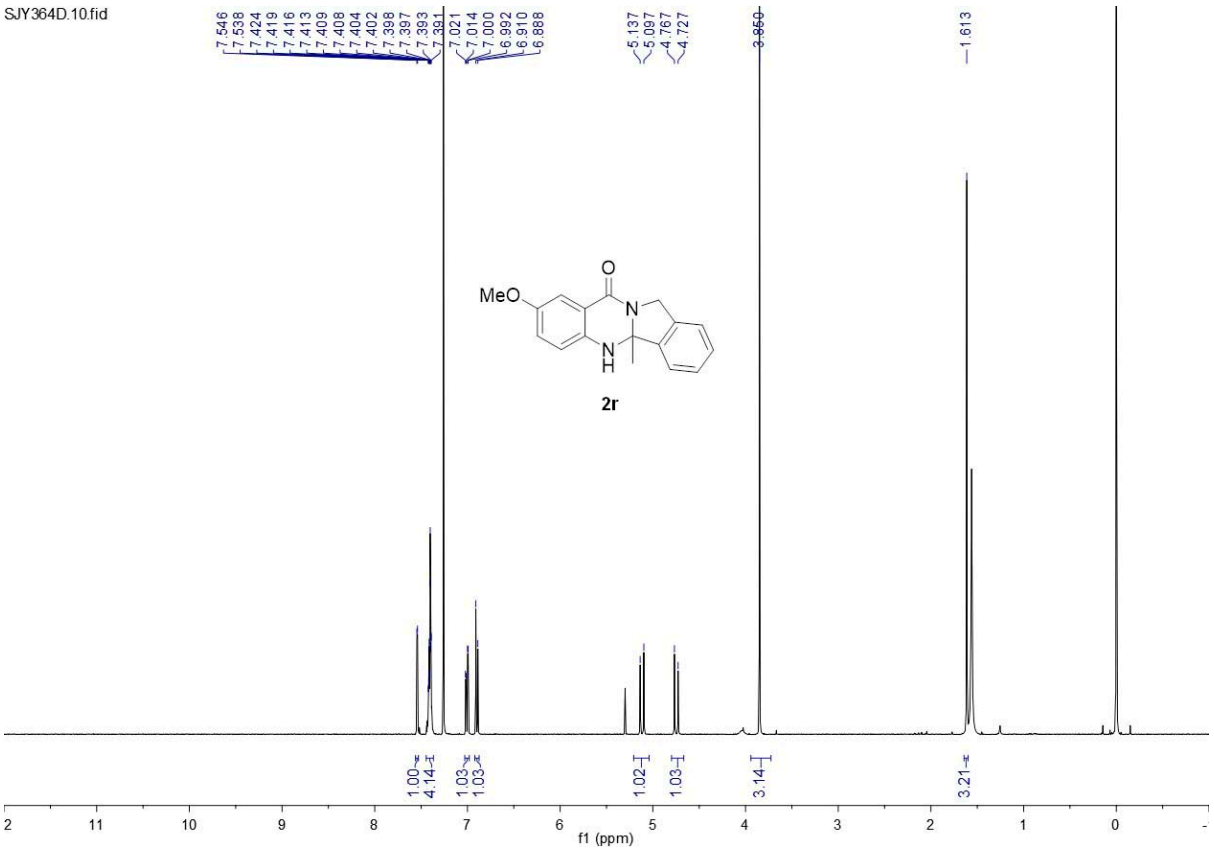

SJY356B.10.fid
8.114
8.108
7.460
7.454
7.439
7.433
7.415
7.408
7.406
7.404
7.400
7.397
7.379
7.373
7.364
7.358
7.354
6.745
6.724
5.140
5.100
4.771
4.731
4.483
1.655
1.00
1.12
3.06
1.15
1.09
1.07
1.05
0.95
3.27
f1 (ppm)
Br
O
N
N
H
2o

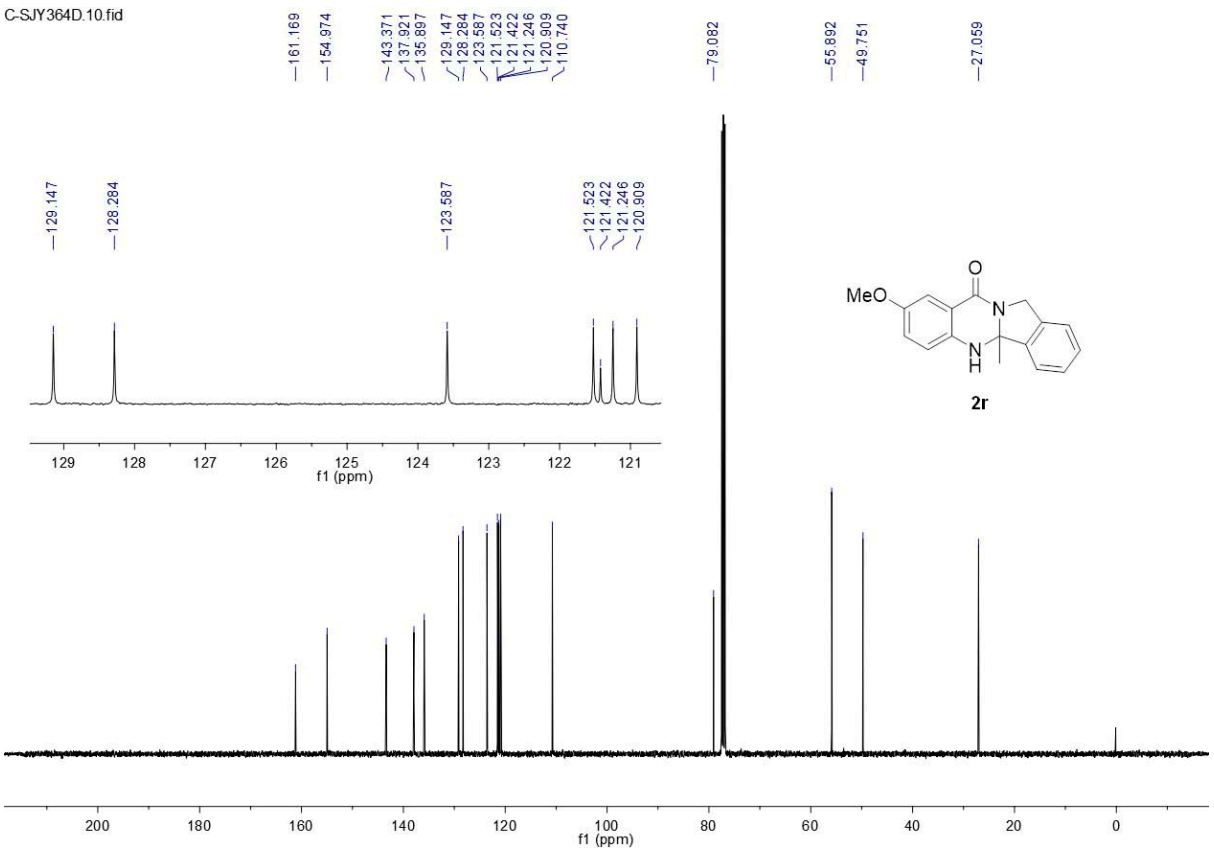

C-SJY356B.10.fid
160.207
143.649
142.762
136.245
135.468
131.165
129.305
128.392
123.687
120.839
120.008
118.768
112.716
78.855
50.127
27.479
f1 (ppm)
Br
O
N
N
H
2o

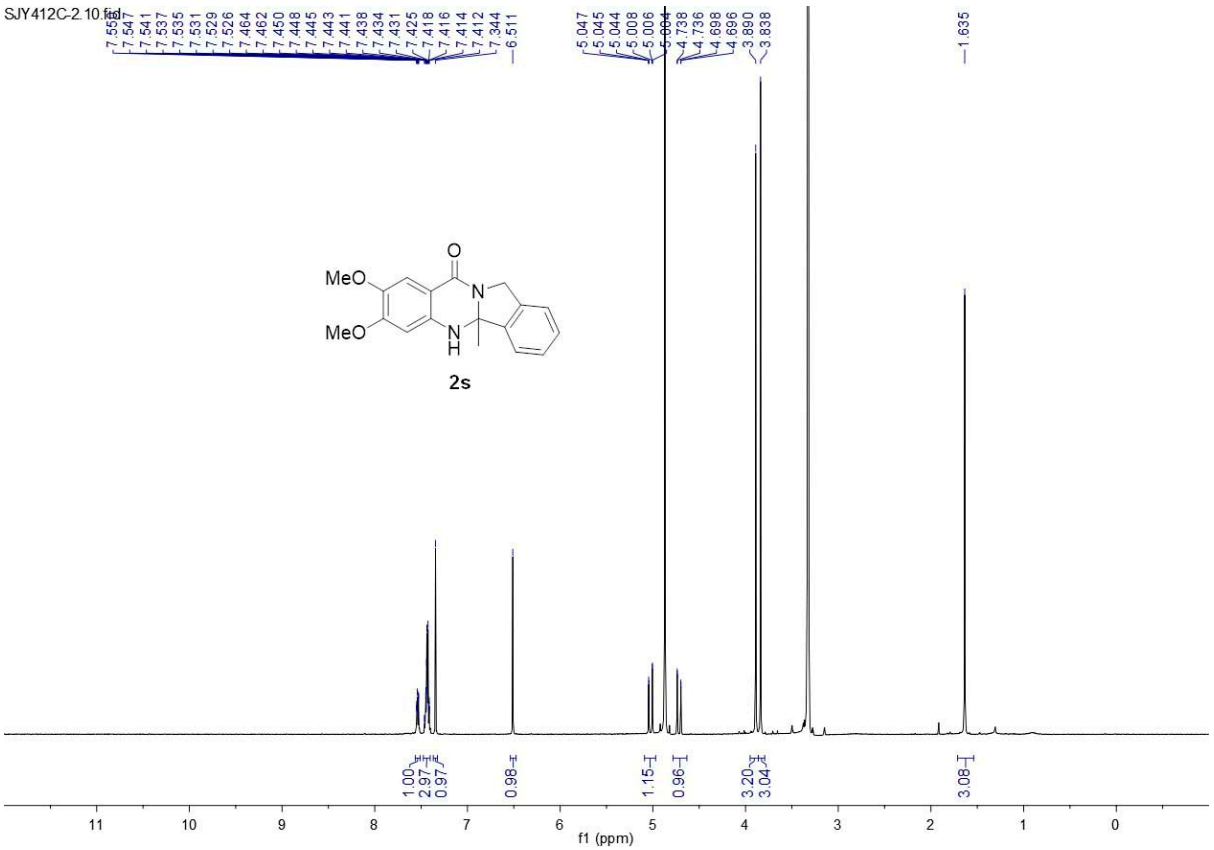

SJY404B.11.fid
8.146
7.936
7.915
7.681
7.675
7.609
7.607
7.605
7.602
7.592
7.589
7.585
7.572
7.566
7.551
7.545
7.483
7.481
7.476
7.462
7.460
7.458
7.445
7.442
7.437
7.428
7.425
7.420
7.406
5.012
4.972
4.716
4.676
1.580
1.00
1.05
1.01
2.37
3.29
1.06
1.11
3.18
f1 (ppm)
O
N
O2N
N
H
2p
C-SJY404B.11.fid
158.453
150.686
146.933
142.801
134.744
129.219
128.734
128.014
123.457
121.323
120.033
111.911
109.806
78.509
49.459
27.067
f1 (ppm)
2p

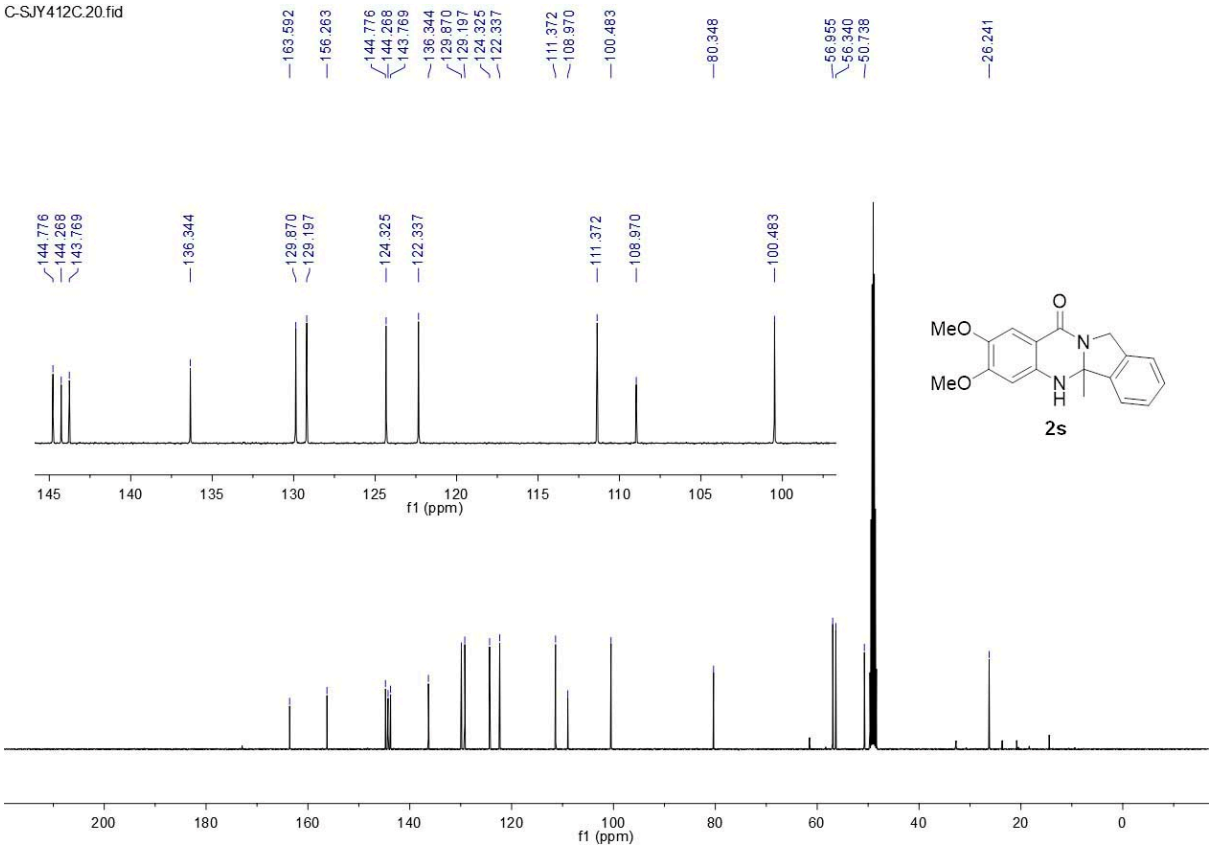

SJY411B.11.fid
7.762
7.759
7.472
7.467
7.459
7.454
7.448
7.363
7.360
7.354
7.353
7.345
7.338
7.333
7.331
7.324
7.321
7.319
7.314
7.309
7.168
7.163
7.148
7.143
6.847
6.826
5.076
5.037
4.714
4.675
2.291
1.634
Me
O
N
N
H
2q
1.00
1.06
3.15
1.06
1.03
1.49
1.05
3.21
3.21
f1 (ppm)

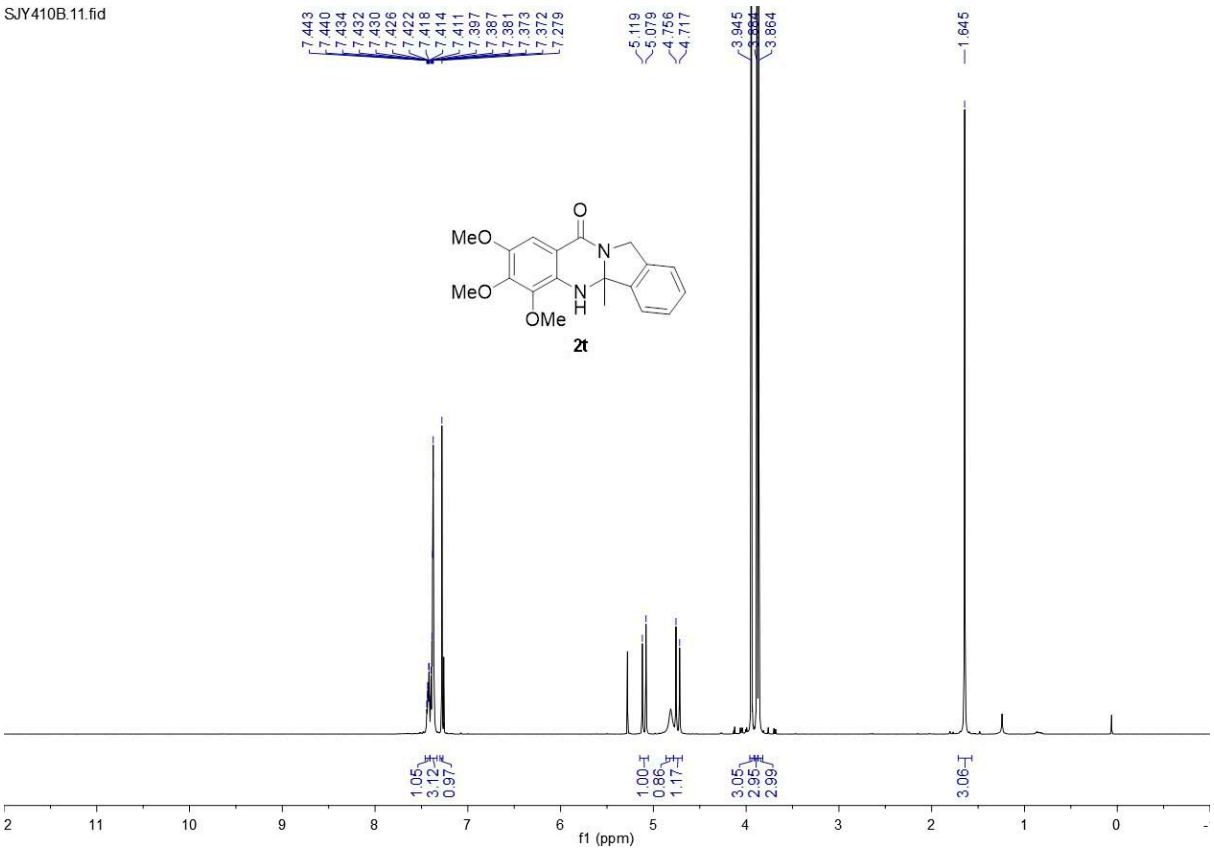

C-SJY411B.10.fid
161.506
143.177
142.120
135.610
134.352
130.428
129.010
128.380
128.190
123.466
121.171
118.868
117.791
78.902
49.913
27.042
20.662
Me
O
N
N
H
2q
f1 (ppm)
f1 (ppm)

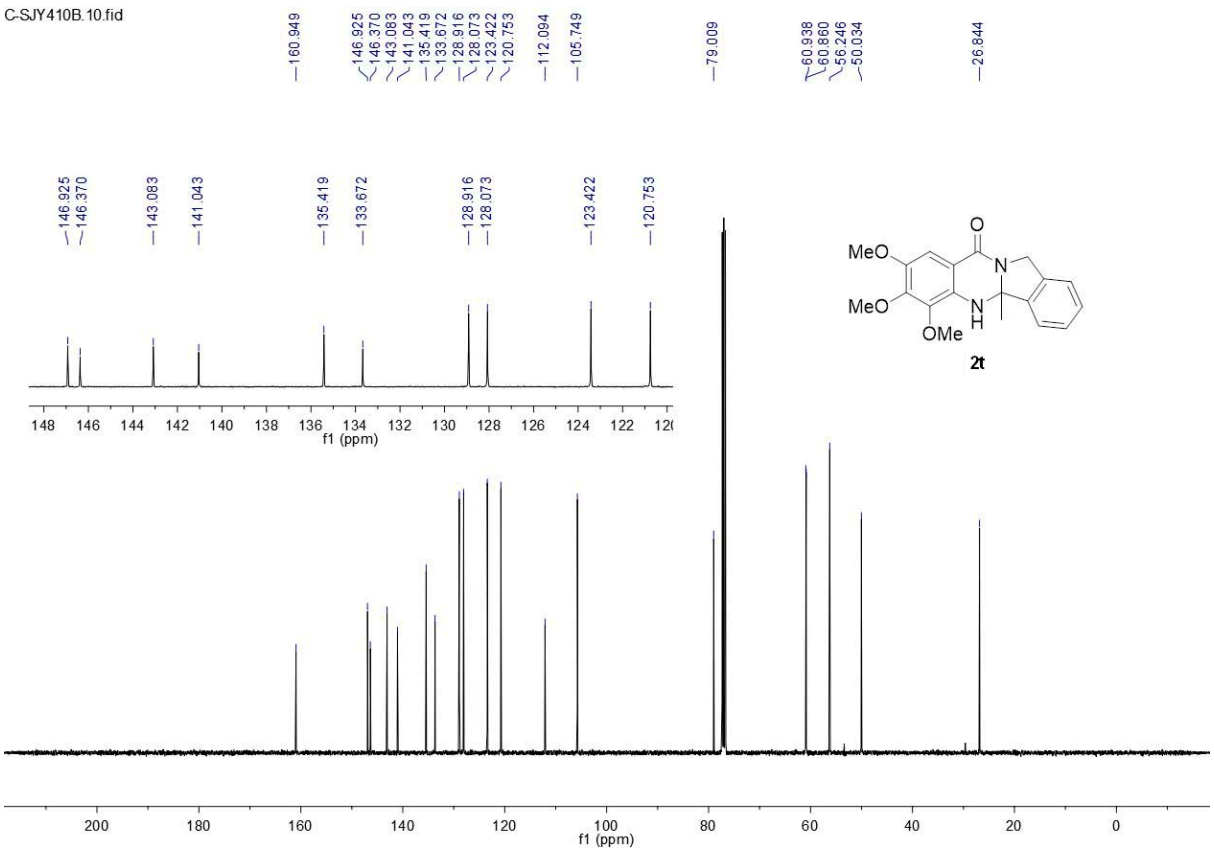

SJY364D.10.fid
7.546
7.538
7.424
7.419
7.416
7.413
7.409
7.408
7.404
7.402
7.398
7.397
7.393
7.391
7.021
7.014
7.000
6.992
6.910
6.888
5.137
5.097
4.767
4.727
3.860
1.613
1.00
4.14
1.03
1.03
1.02
1.03
3.14
3.21
f1 (ppm)
MeO
O
N
N
H
2r

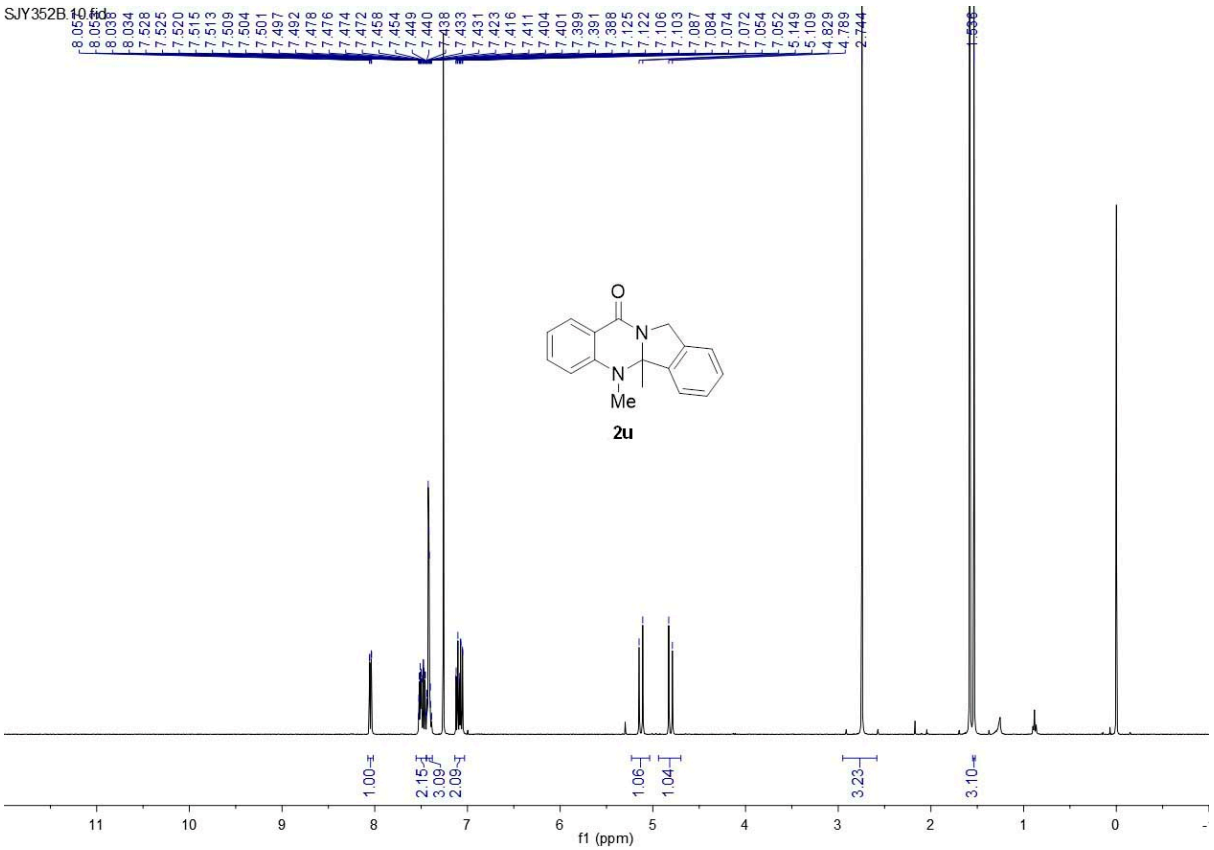

C-SJY364D.10.fid
161.169
154.974
143.371
137.921
135.897
129.147
128.284
123.587
121.523
121.422
121.246
120.909
110.740
79.082
55.892
49.751
27.059
f1 (ppm)
MeO
O
N
N
H
2r

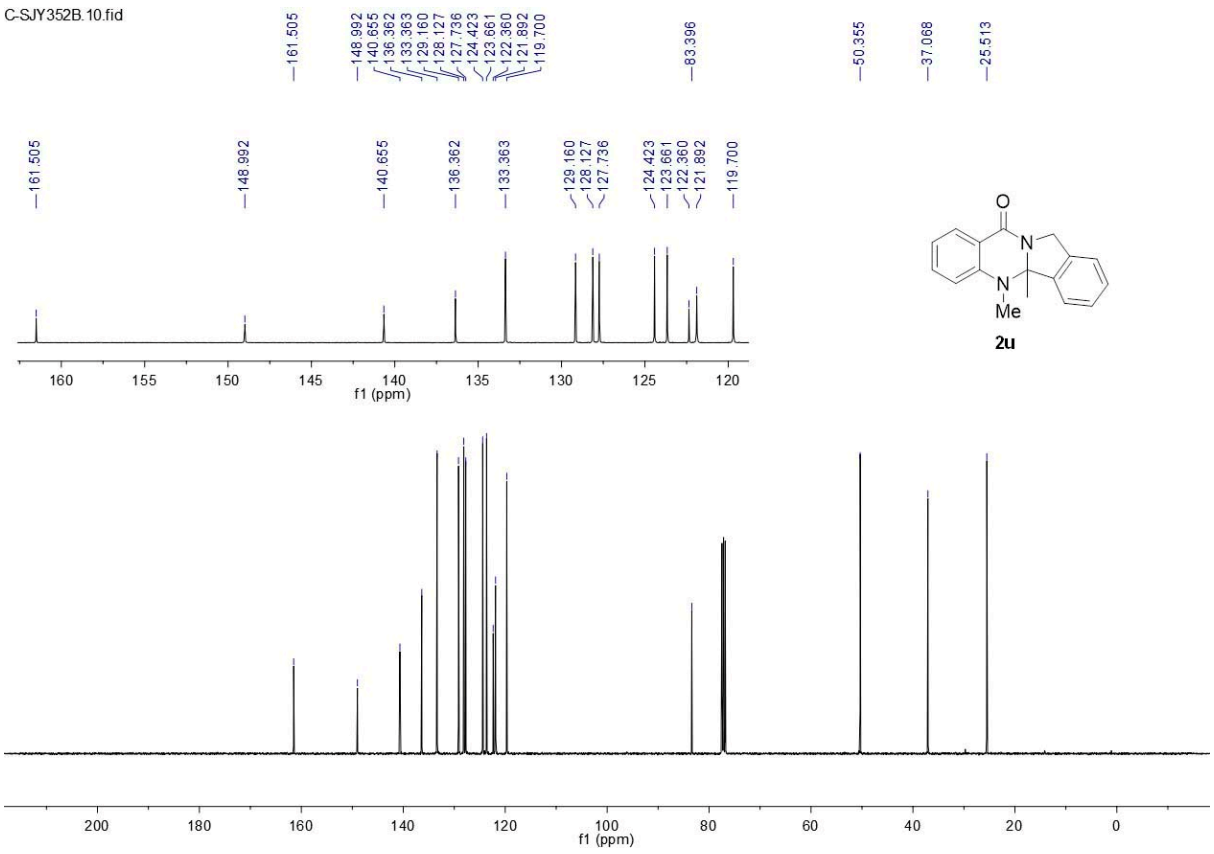

SJY412C-2.10.fid
7.558
7.547
7.541
7.537
7.535
7.531
7.529
7.526
7.464
7.462
7.450
7.448
7.445
7.443
7.441
7.438
7.434
7.431
7.425
7.418
7.416
7.414
7.412
7.344
6.511
5.047
5.045
5.044
5.008
5.006
4.738
4.736
4.698
4.696
3.890
3.838
1.635
MeO
MeO
O
N
N
H
2s
1.00
2.97
0.97
0.98
1.15
0.96
3.20
3.04
3.08
f1 (ppm)


C-SJY412C.20.fid
163.592
156.263
144.776
144.268
143.769
136.344
129.870
129.197
124.325
122.337
111.372
108.970
100.483
80.348
56.955
56.340
50.738
26.241
MeO
MeO
O
N
N
H
2s
f1 (ppm)


SJY410B.11.fid
7.443
7.440
7.434
7.432
7.430
7.426
7.422
7.418
7.414
7.411
7.397
7.387
7.381
7.373
7.372
7.279
5.119
5.079
4.756
4.717
3.945
3.864
1.645
MeO
MeO
OMe
O
N
H
2t
1.05
3.12
0.97
1.00
0.86
1.17
3.05
2.95
2.99
3.06
f1 (ppm)


C-SJY410B.10.fid
160.949
146.925
146.370
143.083
141.043
135.419
133.672
128.916
128.073
123.422
120.753
112.094
105.749
79.009
60.938
60.860
56.246
50.034
26.844
MeO
MeO
OMe
O
N
H
2t
f1 (ppm)


SJY352B.10.fid
f1 (ppm)
2u


C-SJY352B.10.fid
161.505
148.992
140.655
136.362
133.363
129.160
128.127
127.736
124.423
123.661
122.360
121.892
119.700
83.396
50.355
37.068
25.513
f1 (ppm)
2u