

Supplementary Information

Visible light-induced oxidant/additive-free atom-economic synthesis of multifunctionalized cyclopropanes via energy transfer

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1. MATERIALS AND METHODS

All air- and moisture-insensitive reactions were carried out under ambient atmosphere and monitored by thin layer chromatography (TLC). Concentration under reduced pressure was performed by rotary evaporation at 35-45 °C at an appropriate pressure. Purified compounds were further dried under high vacuum. Yields refer to purified and spectroscopically pure compounds, unless otherwise stated. Mass spectra and High-Resolution Mass spectral (HRMS) data was carried out using an Agilent6890N GC (JEOL JMS-700) TOF instrument, and electronic ionization (EI), Chemical ionization (CI), and FAB as ion source at National Taiwan Normal University, Taipei City, Taiwan. Melting points were determined on an EZ-Melt (Automated melting point apparatus).

Solvents

Bottle grade DMA, PEG-400, CAN, Toluene, MeOH, DMSO were purchased from Alfa acer. Anhydrous DCM was obtained from Phoenix Solvent Drying Systems. All deuterated solvents were purchased from Sigma-Aldrich.

Chromatography

Thin layer chromatography (TLC) was performed using Merck TLC aluminum sheets (silica gel 60 F254) and visualized by fluorescence quenching under UV light. Flash column chromatography was performed using silica gel (40-63 μm particle size) purchased from Merck.

Photochemistry

All reactions with blue light were carried out using 1 x 40 W Kessil Blue LED (Kessil A160WE Controllable LED Aquarium Light) lamp purchased from Amazon (Taiwan) with an output centered at a wavelength of approximately $\lambda_{\text{max}} = 462$ nm. The reaction vials were cooled with a fan while being irradiated under blue light.

See the following links for more details.

https://www.kessil.com/aquarium/saltwater_A160.php

https://www.kessil.com/support/downloadfiles/aquarium/A160WE_UserManual.pdf

Spectroscopy and Instruments

^1H , ^{13}C , and DEPT NMR spectra were recorded on a 400 MHz Varian Unity Plus or Varian Mercury plus spectrometer or JEOL ECS-400. The chemical shift (δ) values are reported in ppm, and the coupling constants (J)

are given in Hz. For ^1H NMR: CDCl_3 , δ 7.26; CD_3CN , δ 1.96; CD_2Cl_2 , δ 5.32; $(\text{CD}_3)_2\text{SO}$, δ 2.50; For ^{13}C NMR: CDCl_3 , δ 77.16; 1.32; CD_2Cl_2 , δ 53.84; $(\text{CD}_3)_2\text{SO}$, δ 39.52.¹ The abbreviations used are as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublet, ddd = doublet of doublet of doublet, dt = doublet of triplets, td = triplet of doublet, m = multiplet, br = broad; coupling constants in Hz; integration.

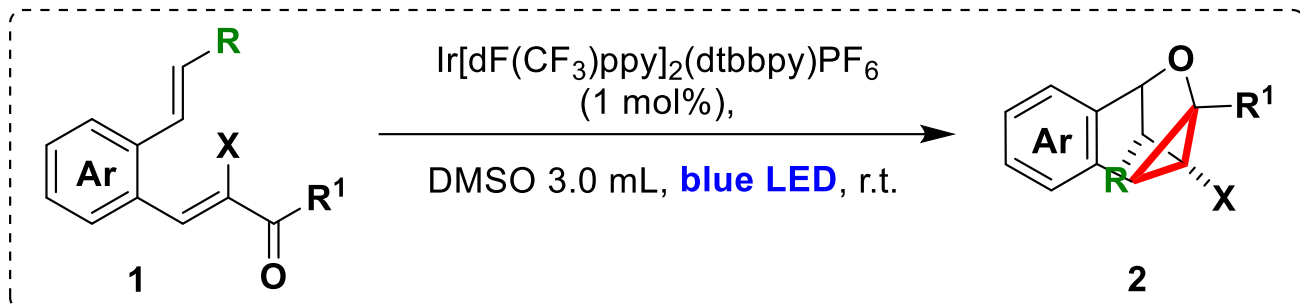
Starting materials

All substrates were used as received from commercial suppliers, or prepared according to published procedures, respectively, unless otherwise stated. Nickel, Copper, Iron, Ir (III) catalysts, were purchased from Alfa Aesar, and stored in an under vacuum/dark hood cabinet. Aldehydes, Alkenes, N-substituted amines were purchased from Sigma-Aldrich, Acros, TCI, or Alfa Aesar.

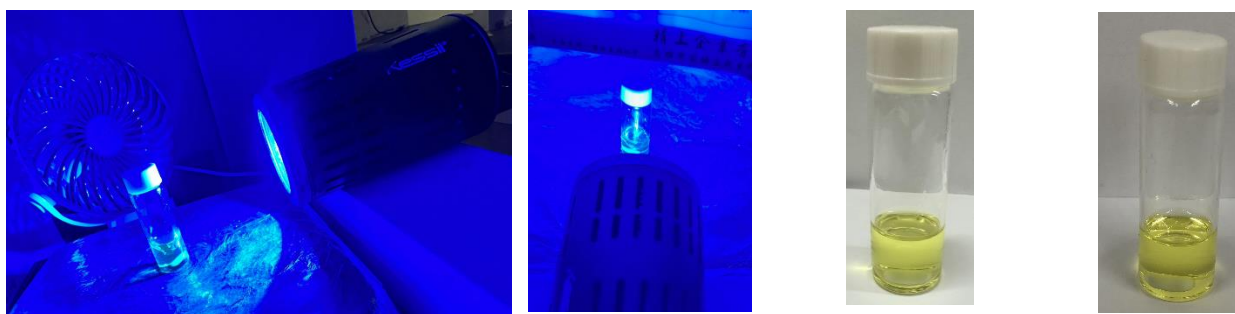
2. EXPERIMENTAL DATA

General procedure and reaction condition optimization for cascade cyclopropanation

a) General procedure for cyclopropanation



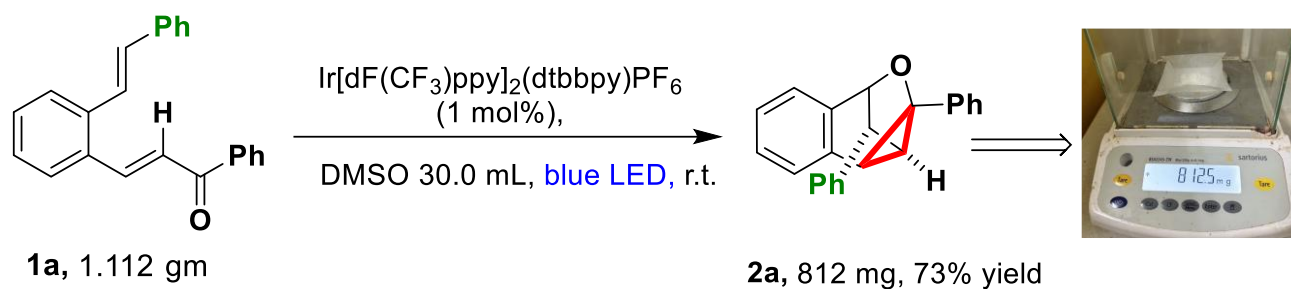
A clean vial (5 mL) equipped with a magnetic stir bar were added to **1** (0.2 mmol), (Ir[dF(CF₃)ppy]₂(dtbbpy))PF₆ (1 mol%) and DMSO (3.0 mL) at room temperature, and then placed at a distance of approx. 3 cm from a 40 W blue LED lamp, and the solution was stirred at room temperature under visible-light irradiation for 4-7 h. The progress of the reaction was monitored by thin layer chromatography. When the reaction was complete, water was added to quench the reaction mixture, followed by extraction with ethyl acetate (3×10 mL). Finally, the combined organic layer was dried over Na₂SO₄, filtered, and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel eluting with Hex/EA = 97:3 to afford the cyclopropanation product **2** derivatives.



Before reaction **1a** After reaction

Figure S1: Reaction setup

b) General procedure for gram scale synthesis of **2a**.



A clean vial (5 mL) equipped with a magnetic stir bar were added to **1a** (3.587 mmol), (Ir[dF(CF₃)ppy]₂(dtbbpy))PF₆ (1 mol%) and DMSO (30.0 mL) at room temperature, and then placed at a distance of approx. 3 cm from a 40 W blue LED lamp, and the solution was stirred at room temperature under visible-light irradiation for 10 h. The progress of the reaction was monitored by thin layer chromatography. When the reaction was complete, cold water was added to quench the reaction mixture, followed by extraction with ethyl acetate (10×12 mL). Finally, the combined organic layer was dried over Na₂SO₄, filtered, and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel eluting with Hex/EA = 97:3 to afford the cyclopropanation product **2a** derivative in 73% (812 mg) yield.

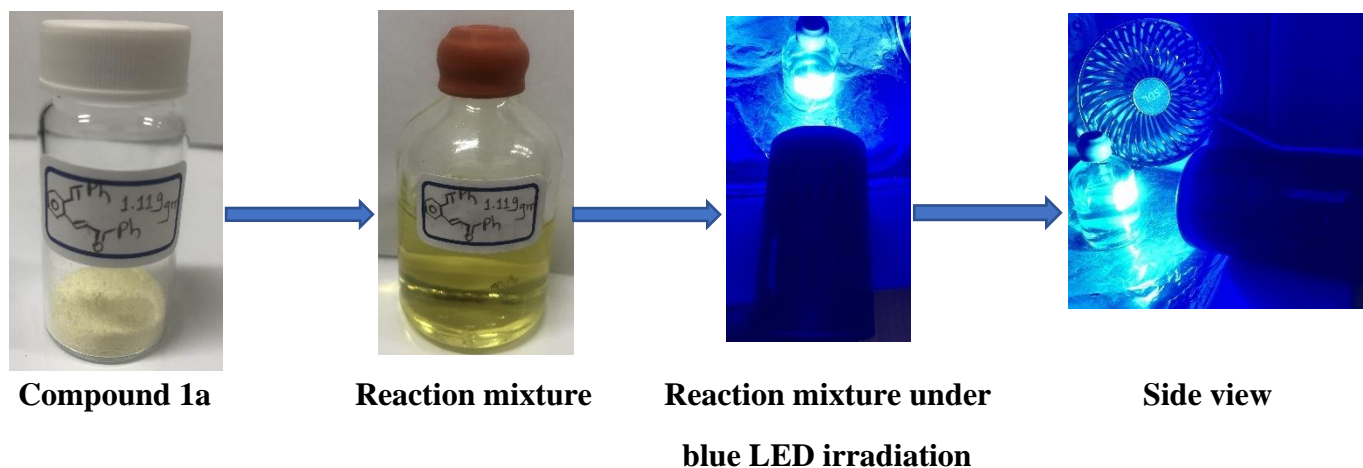
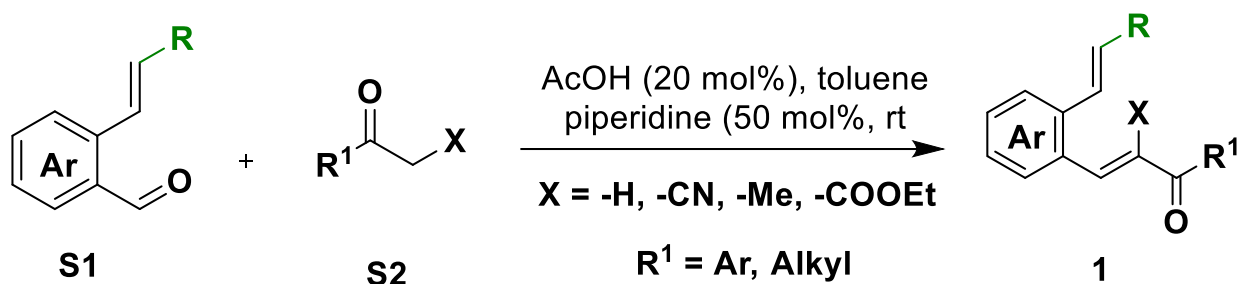


Figure S2. Representation of the reaction setup

c) Substrates synthesis and cyclization reactions

1. General Procedure for the Synthesis of analogous (1).¹



A reaction tube was charged with (*E*)-1-Formyl-2-(2-aryl-(alkene) alkenyl) benzene (**S1**) (1.0 equiv), **S2** derivatives (1.2 equiv), acetic acid (20 mol%) and piperidine (20 mol%), in 3.0 mL of toluene. The reaction suspension was stirred at room temperature and progress of reaction was monitored by TLC. Upon completion water was added to quench the reaction mixture and then extracted with the ethyl acetate (3 × 20 mL). Finally combined organic layer was dried over sodium sulphate, filtered and concentrated in vacuum. The residue was purified by column chromatography on silica gel to afford the corresponding **1** derivative.

3. CONTROL STUDIES

a. Stern-Volmer Quenching Studies

Further insights into the reaction mechanism were gained by examining the controlling features of visible light-mediated cyclopropanation and dearomatization. Initially, a Stern-Volmer analysis was performed² (Figure S3), and the photosensitizer Ir[dF(CF₃)ppy]₂(bpy)PF₆ was quenched efficiently with the addition of compound **1q''** (C) or **1q** (D), whereas compound **1q'** (B) devoid of olefin moiety showed no quenching even at an extremely high concentration (500 μL).

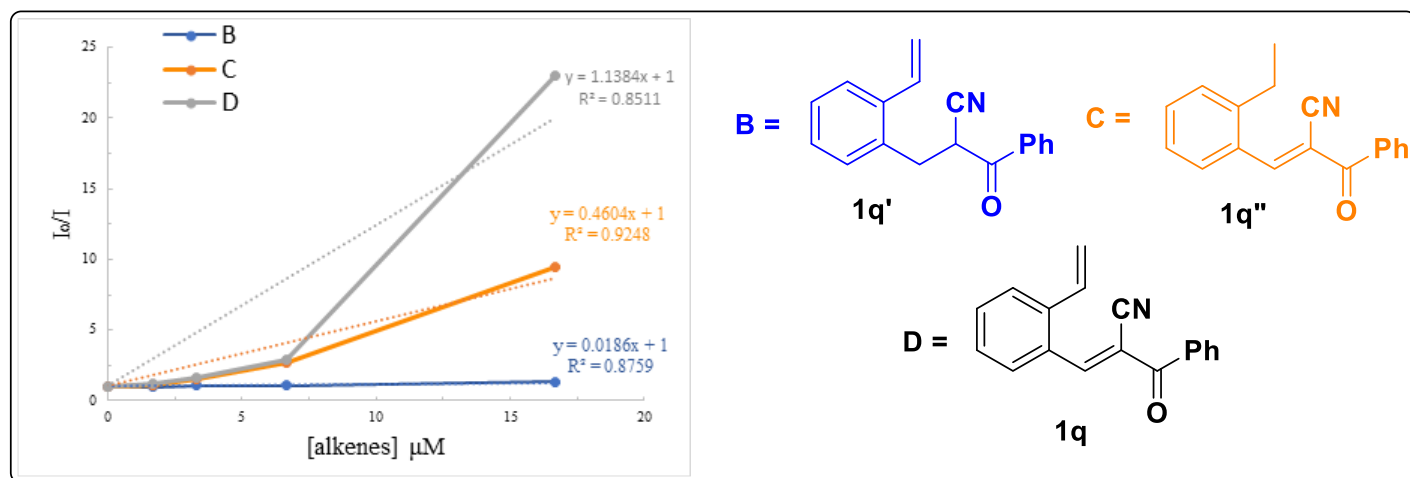


Figure S3: Stern-Volmer quenching study of [Ir(dF(CF₃)ppy)₂(dtbbpy)]PF₆. The data were fitted through linear regression: **1q'** (B) ($y = 1.1384x + 1$; $R^2 = 0.8511$); **1q''** (C) ($y = 0.4604x + 1$; $R^2 = 0.9248$); **1q** (D) ($y = 0.0186x + 1$; $R^2 = 0.8759$); blue oval = (**1q'** (B)); orange oval = **1q''** (C); Gray oval = **1q** (D).

b. Fluorescence Quenching Study

A solution of Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ in acetonitrile at 25 °C was excited at 320 nm and the intensity of emission spectrum was measured at 480 nm (emission maximum). The fluorescence quenching experiments demonstrated that substrates **1a** efficiently quenches the excited state of the Ir photocatalyst under the standard reaction conditions respectively. (Figure S4).

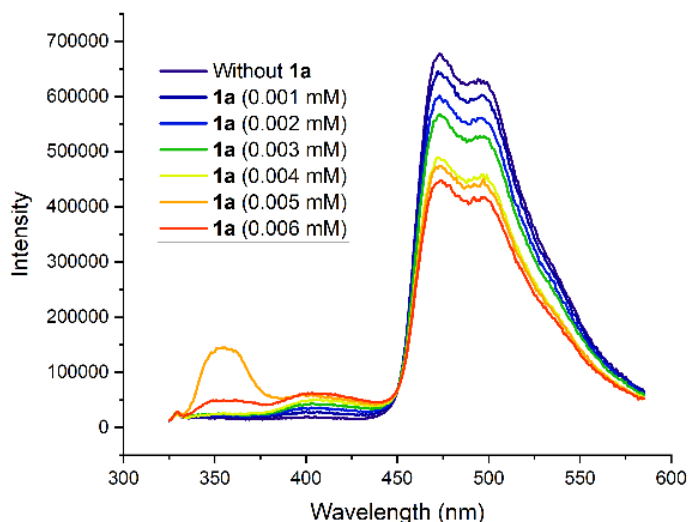
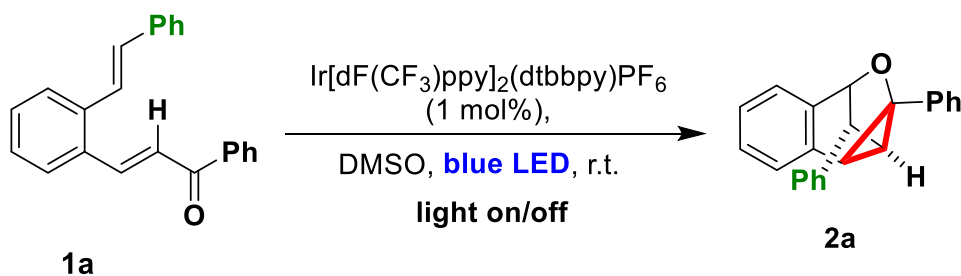


Figure S4. Fluorescence quenching of Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (0.05 mM) with **1a** in acetonitrile.

c. Light On-Off Experiment (Scheme S1)



A light on/off experiment was performed separately by using the model reaction of **1a** irradiated under the standard catalytic system respectively. The reaction proceeded over a period of time under alternating cycles of irradiation and darkness (0-200 minutes for product **2a**), and the progress was monitored by TLC analysis. As shown in Figure S5, cyclization product **2a** was only formed under irradiation with blue LEDs (Scheme S1).

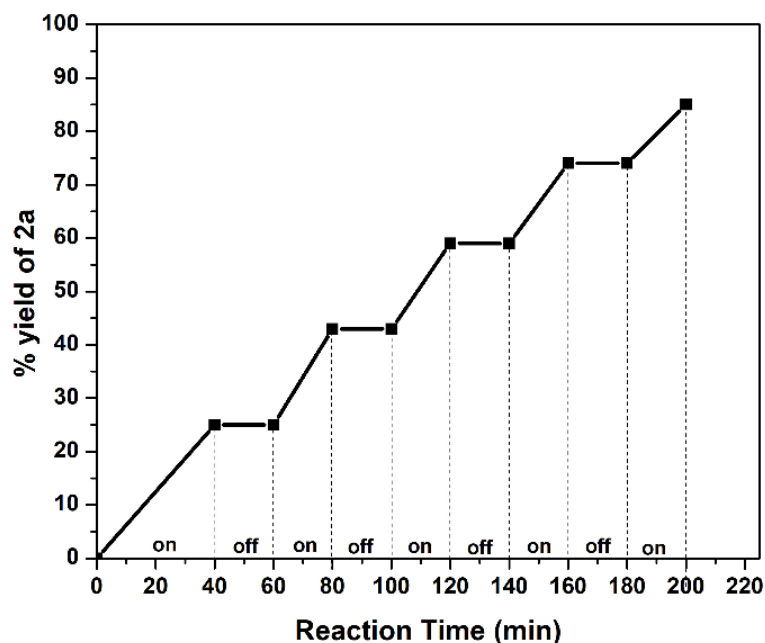
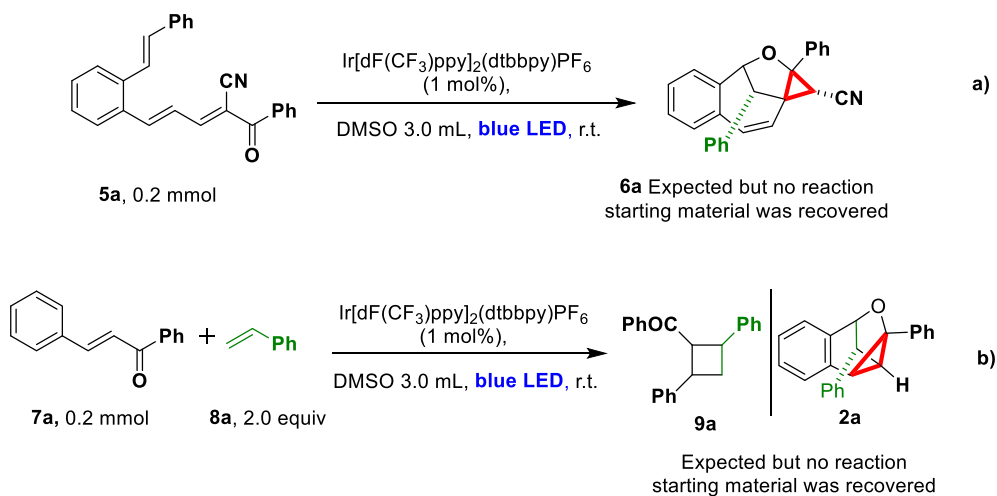


Figure S5. Visible-light Irradiation ON/OFF Experiments for product **2a**.

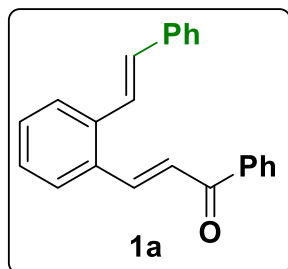
d. Control Reactions



Reaction was carried out according to general procedure a on 0.2 mmol scale. No formation of cyclopropanation fused product **6a** or **9a** or **2a** were observed, and only unreacted starting materials were isolated from the reaction mixture.

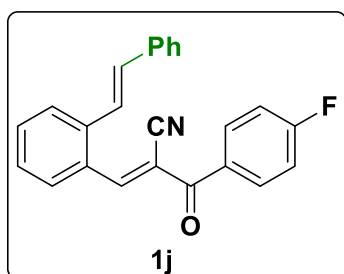
4. SPECTRAL CHARACTERIZATION

(E)-1-phenyl-3-(2-((E)-styryl)phenyl)prop-2-en-1-one (1a): Yield: 91 % (282 mg), pale yellow solid, m.p. 71-



73 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.25 (d, $J = 15.5$ Hz, 1H), 8.06-7.99 (m, 2H), 7.70-7.65 (m, 1H), 7.62 (d, $J = 7.6$ Hz, 1H), 7.58-7.43 (m, 7H), 7.41-7.25 (m, 5H), 6.99 (d, $J = 16.1$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 190.10, 142.43, 137.99, 136.93, 133.29, 132.78, 132.59, 130.19, 128.66, 128.56, 128.46, 128.02, 127.67, 127.39, 126.93, 126.72, 125.48, 124.21; **HRMS (EI):** m/z calcd. for $[\text{M}]^+$ $[\text{C}_{23}\text{H}_{18}\text{NO}]^+$ 310.1358, found 310.1360.

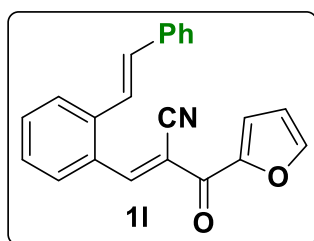
(E)-2-(4-fluorobenzoyl)-3-(2-((E)-styryl)phenyl)acrylonitrile (1j): Yield: 83 % (293 mg) yellow solid, m.p.



98-100 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.48 (d, $J = 2.1$ Hz, 1H), 8.18 (t, $J = 5.4$ Hz, 1H), 7.94 (s, 1H), 7.64 (dd, $J = 10.6, 7.6$ Hz, 1H), 7.55 (dd, $J = 14.5, 6.8$ Hz, 1H), 7.47 (s, 2H), 7.43 (dd, $J = 12.9, 5.3$ Hz, 1H), 7.41-7.36 (m, 2H), 7.31 (d, $J = 5.9$ Hz, 1H), 7.29-7.21 (m, 1H), 7.12 (d, $J = 8.3$ Hz, 2H), 6.91 (dd, $J = 15.9, 2.2$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 187.17, 167.10, 164.56

(d, $J_F = 254.6$ Hz), 156.04, 154.59, 139.75, 136.40, 135.51, 132.68, 132.09, 132.00 (d, $J_F = 9.2$ Hz), 129.87, 128.95, 128.85, 128.69 (d, $J_F = 16$ Hz), 127.96, 127.72 (d, $J_F = 24$ Hz), 126.84, 124.70, 116.57, 116.07, 115.85 (d, $J_F = 22$ Hz), 111.74; **HRMS (EI):** m/z calcd. for $[\text{M}]^+$ $[\text{C}_{24}\text{H}_{16}\text{FNO}]^+$ 353.1216, found 353.1214.

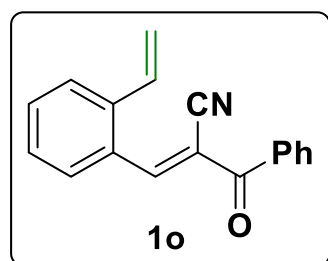
(E)-2-(furan-2-carbonyl)-3-(2-((E)-styryl)phenyl)acrylonitrile (1i): Yield: 88 % (286 mg), yellow solid, m.p.



125-127 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.82 (s, 1H), 8.15 (d, $J = 7.8$ Hz, 1H), 7.75 (d, $J = 3.7$ Hz, 1H), 7.70-7.62 (m, 2H), 7.53 (dd, $J = 14.2, 7.4$ Hz, 3H), 7.47-7.29 (m, 5H), 6.92 (d, $J = 16.0$ Hz, 1H), 6.62 (dd, $J = 3.6, 1.6$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 173.04, 154.90, 150.70, 150.63, 148.02, 139.76,

136.50, 135.44, 132.53, 130.03, 129.02, 128.78, 128.55, 127.79, 127.68, 126.89, 124.81, 121.46, 116.78, 114.35, 112.81, 109.97, 109.92; **HRMS (EI):** m/z calcd. For $[\text{M}]^+$ $[\text{C}_{22}\text{H}_{15}\text{NO}_2]^+$ 325.1103, found 325.1103.

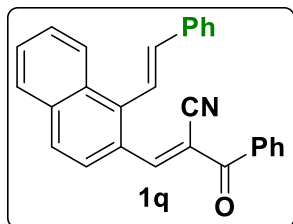
(E)-2-benzoyl-3-(2-vinylphenyl)acrylonitrile (1o) : Yield: 70 % (181 mg), pale yellow solid, m.p. 100-102 °C.



Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.42 (s, 1H), 8.21-8.15 (m, 1H), 7.93-7.89 (m, 2H), 7.67-7.61 (m, 1H), 7.57-7.49 (m, 4H), 7.44 (ddd, $J = 8.4, 6.2, 2.4$ Hz, 1H), 6.90 (dd, $J = 17.2, 11.0$ Hz, 1H), 5.68-5.44 (m, 2H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 188.66, 154.44, 139.95, 135.72, 133.56, 133.50, 132.55, 129.29,

128.71, 128.67, 128.18, 127.59, 120.64, 116.54, 112.21; **HRMS (EI)**: m/z calcd. for $[M]^+$ $[C_{18}H_{13}NO]^+$ 259.0997, found 259.0996.

(E)-2-benzoyl-3-(1-((E)-styryl)naphthalen-2-yl)acrylonitrile (1q): Yield: 85 % (327 mg), yellow solid, m.p.

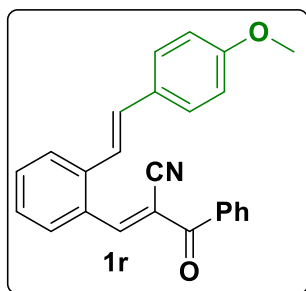


112-114 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.3/9.7). **¹H NMR** (400 MHz, CDCl₃) δ 8.52 (s, 1H), 8.33 (d, $J = 8.8$ Hz, 1H), 8.21-8.13 (m, 1H), 7.95-7.88 (m, 2H), 7.80-7.74 (m, 2H), 7.71-7.53 (m, 5H), 7.48-7.38 (m, 4H), 7.24-7.16 (m, 2H), 6.69 (d, $J = 16.1$ Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 189.86, 156.27, 141.32, 140.52, 136.20, 135.82, 134.83, 132.85, 131.75,

129.02, 128.96, 128.59, 128.52, 128.47, 128.36, 127.89, 127.20, 126.96, 125.88, 124.68, 123.55, 116.70, 111.76;

HRMS (EI): m/z calcd. for $[M]^+$ $[C_{28}H_{19}NO]^+$ 385.1467, found 385.1470.

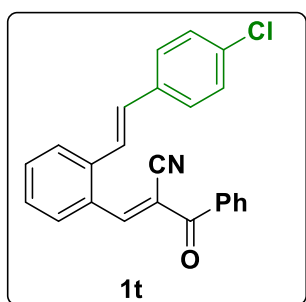
(E)-2-benzoyl-3-(2-((E)-4-methoxystyryl)phenyl)acrylonitrile (1r): Yield: 92 % (336 mg) yellow solid, m.p.



122-124 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.7/9.3). **¹H NMR** (400 MHz, CDCl₃) δ 8.47 (s, 1H), 8.19 (d, $J = 7.8$ Hz, 1H), 7.95-7.87 (m, 2H), 7.61 (ddd, $J = 7.0, 5.1, 4.0$ Hz, 2H), 7.56-7.41 (m, 6H), 7.11 (d, $J = 16.0$ Hz, 1H), 6.94-6.83 (m, 3H), 3.86 (d, $J = 7.9$ Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 189.06, 160.05, 154.74, 140.05, 135.85, 134.99, 133.35, 132.55, 129.73, 129.48, 129.31, 129.26, 128.97, 128.66, 128.22, 127.56, 127.46,

122.50, 116.53, 114.25, 112.05, 77.31, 76.99, 76.68, 55.37; **HRMS (EI)**: m/z calcd. for $[M]^+$ $[C_{25}H_{19}NO_2]^+$ 365.1416, found 365.1414.

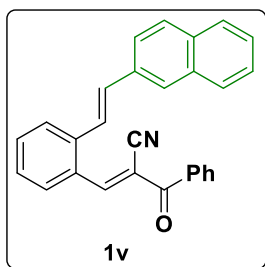
(E)-2-benzoyl-3-(2-((E)-4-chlorostyryl)phenyl)acrylonitrile (1t): Yield: 86 % (317 mg), yellow solid, m.p.



131-133 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). **¹H NMR** (400 MHz, CDCl₃) δ 8.46 (s, 1H), 8.19 (d, $J = 7.7$ Hz, 1H), 7.95-7.88 (m, 2H), 7.68-7.60 (m, 2H), 7.55 (td, $J = 7.7, 1.1$ Hz, 1H), 7.51-7.45 (m, 3H), 7.43-7.38 (m, 2H), 7.37-7.33 (m, 2H), 7.26-7.20 (m, 1H), 6.87 (d, $J = 16.0$ Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 188.77, 154.32, 139.20, 135.74, 134.96, 134.30, 133.87, 133.49, 132.60, 130.02, 129.47, 129.27, 129.01, 128.68,

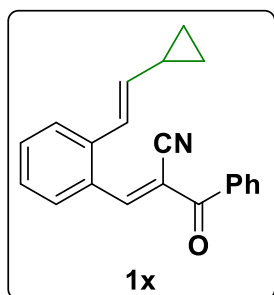
128.17, 128.01, 127.61, 125.30, 116.44, 112.41; **HRMS (EI)**: m/z calcd. for $[M]^+$ $[C_{24}H_{16}ClNO]^+$ 369.0920, found 369.0918.

(E)-2-benzoyl-3-(2-((E)-2-(naphthalen-2-yl)vinyl)phenyl)acrylonitrile (1f) : Yield: 90 % (346.5 mg), yellow



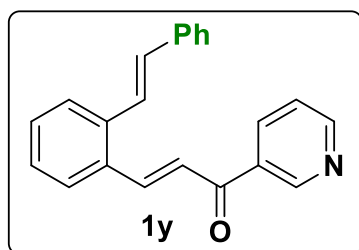
solid, m.p. 138-139 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.3/9.7). $^1\text{H NMR}$ (400 MHz, CDCl_3) : δ 8.47 (s, 1H), 8.19 (dd, $J = 7.9, 1.2$ Hz, 1H), 7.93-7.76 (m, 5H), 7.70-7.60 (m, 2H), 7.597.38 (m, 8H), 7.31 (d, $J = 15.9$ Hz, 1H), 7.04 (d, $J = 16.0$ Hz, 1H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 189.02, 154.42, 139.53, 135.74, 135.21, 133.84, 133.45, 133.33, 132.56, 129.86, 129.22, 128.95, 128.63, 128.47, 128.10, 127.91, 127.69, 127.52, 127.49, 126.54, 126.43, 124.78, 123.20, 116.41, 112.29; **HRMS (EI)**: m/z calcd. for $[\text{M}]^+$ $[\text{C}_{28}\text{H}_{19}\text{NO}]^+$ 385.1467, found 385.1469.

(E)-2-benzoyl-3-(2-((E)-2-cyclopropylvinyl)phenyl)acrylonitrile (1x): Yield: 62 % (185.4 mg), pale yellow



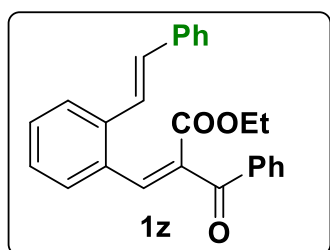
solid, m.p. 88-89 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). $^1\text{H NMR}$ (400 MHz, CDCl_3) : δ 8.43 (s, 1H), 8.15 (ddd, $J = 7.9, 1.4, 0.7$ Hz, 1H), 7.95-7.88 (m, 2H), 7.68-7.59 (m, 1H), 7.57-7.50 (m, 2H), 7.49-7.44 (m, 1H), 7.44-7.40 (m, 1H), 7.35 (td, $J = 7.5, 6.9, 1.8$ Hz, 1H), 6.61 (d, $J = 15.5$ Hz, 1H), 5.48 (dd, $J = 15.5, 9.2$ Hz, 1H), 1.65-1.52 (m, 1H), 0.93-0.80 (m, 2H), 0.56-0.47 (m, 2H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 188.90, 155.06, 142.81, 140.25, 135.89, 133.36, 132.49, 129.25, 129.06, 128.71, 128.62, 127.32, 126.98, 123.62, 116.70, 111.39, 15.22, 7.82; **HRMS (ESI)**: m/z calcd. for $[\text{M}+\text{H}]^+$ $[\text{C}_{21}\text{H}_{18}\text{NO}]^+$ 300.1388, found 300.1390.

(E)-1-(pyridin-3-yl)-3-(2-((E)-styryl)phenyl)prop-2-en-1-one (1y): Yield: 75 % (286.5 mg), light yellow solid,



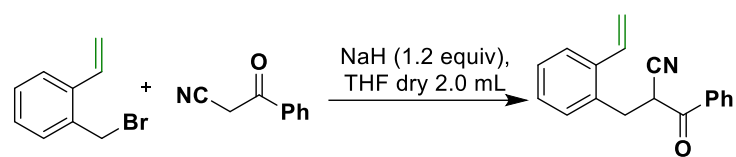
m.p. 125-130 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 1/9). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.30-9.21 (m, 1H), 8.80 (dd, $J = 4.8, 1.7$ Hz, 1H), 8.39-8.24 (m, 2H), 7.68 (dd, $J = 23.1, 7.7$ Hz, 2H), 7.58-7.27 (m, 10H), 7.01 (d, $J = 16.1$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 188.68, 153.19, 149.76, 143.60, 138.39, 136.86, 135.84, 133.36, 133.07, 132.83, 130.68, 128.73, 128.17, 127.78, 127.39, 127.15, 126.77, 125.27, 123.61, 123.22; **HRMS (EI)**: m/z calcd. for $[\text{M}]^+$ $[\text{C}_{22}\text{H}_{17}\text{NO}]^+$ 311.3840, found 311.3838.

1-phenyl-2-((E)-2-((E)-styryl)benzylidene)pentane-1,3-dione (1z): Yield: 75 % (286.5 mg), light yellow solid,



m.p. 125-130 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 1/9). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.36 (s, 1H), 7.91-7.83 (m, 2H), 7.60-7.46 (m, 4H), 7.44-7.29 (m, 6H), 7.26-7.17 (m, 2H), 7.06-6.95 (m, 2H), 4.25 (q, $J = 7.1$ Hz, 2H), 1.19 (t, $J = 7.1$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 194.76, 164.84, 141.80, 137.42, 136.99, 136.45, 133.57, 133.20, 132.82, 131.58, 129.96, 129.32, 129.04, 128.76, 128.58, 128.18, 127.45, 126.84, 126.46, 125.48, 61.57, 14.01; **HRMS (EI)**: m/z calcd. for $[\text{M}]^+$ $[\text{C}_{26}\text{H}_{22}\text{O}_2]^+$ 366.1620, found 366.1617.

Synthesis of 3-oxo-3-phenyl-2-(2-vinylbenzyl)propanenitrile (1q'): To a stirred solution of

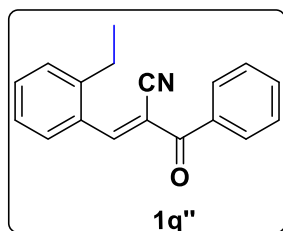


1q' mixture of isomers

of cyanoacetophenone (0.4 mmol, 1.2 equiv.) in THF (2 mL, dry) was added NaH (60% in mineral oil). The mixture was stirred for 20 min at 0 °C.

1-(bromomethyl)-2-vinylbenzene (0.5 mmol) in THF (dry) was added and the reaction mixture was stirred for another 8 h. Upon completion, the reaction was quenched with aqueous NH₄Cl and extracted with ethyl acetate three times. The organics were combined, dried over anhydrous Na₂SO₄, concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with a gradient eluent of petroleum ether/ethyl acetate to affording the final product (**1q'**) (38% yield). ¹H NMR (597 MHz, CDCl₃) δ 7.58 (d, *J* = 7.6 Hz, 1H), 7.54-7.36 (m, 3H), 7.33 (ddt, *J* = 15.0, 8.5, 4.3 Hz, 2H), 7.27-7.19 (m, 3H), 7.01 (dt, *J* = 11.0, 7.3 Hz, 1H), 6.97 (d, *J* = 11.0 Hz, 1H), 5.72 (dd, *J* = 17.3, 1.1 Hz, 1H), 5.60-5.53 (m, 1H), 5.38 (dd, *J* = 11.0, 1.1 Hz, 1H), 5.31 (dd, *J* = 10.9, 1.3 Hz, 1H), 5.04 (s, 1H), 4.94 (s, 1H), 3.62 (d, *J* = 14.1 Hz, 1H), 3.39 (d, *J* = 14.1 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 197.37, 138.27, 136.63, 134.53, 133.45, 132.77, 132.22, 131.92, 131.18, 131.11, 129.57, 129.20, 128.68, 128.52, 128.09, 127.90, 127.88, 126.74, 126.64, 126.25, 120.91, 117.34, 117.09, 72.72, 72.08, 55.04, 39.95, 29.68; Yield: 92 % (181.4 mg), colorless liquid; HRMS (EI): *m/z* calcd. for [M]⁺ [C₁₈H₁₅NO]⁺ 261.1154, found 261.1156.

(E)-2-benzoyl-3-(2-ethylphenyl)acrylonitrile (1q''): A reaction tube was charged with 2-ethylbenzaldehyde

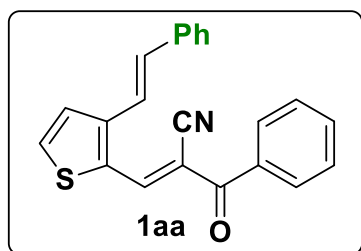


(1.0 equiv), benzoyacetonitrile (1.5 equiv), AcOH (20 mol%) and piperidine (20 mol%) in 5.0 mL of toluene. The reaction suspension was stirred at room temperature, and the progress of the reaction was monitored by TLC. Upon completion, water was added to quench the reaction mixture, which was then extracted with ethyl acetate (3×15 mL).

Finally, the combined organic layer was dried over sodium sulfate, filtered, and concentrated under vacuum. The residue was purified by column chromatography on silica gel to afford the **1q''**.

¹H NMR (400 MHz, CDCl₃) δ 8.39 (d, *J* = 6.6 Hz, 1H), 8.22 (d, *J* = 7.5 Hz, 1H), 7.91 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.68-7.60 (m, 1H), 7.57-7.43 (m, 3H), 7.39-7.30 (m, 2H), 2.73 (q, *J* = 7.6 Hz, 2H), 1.25-1.15 (m, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 188.90, 153.91, 145.79, 135.74, 133.41, 132.92, 130.09, 129.40, 129.22, 128.68, 128.61, 126.67, 116.63, 111.79, 26.64, 15.81; Yield: 95 %, pale yellow solid, m.p. 134-136 °C; HRMS (EI): *m/z* calcd. for [M]⁺ [C₁₈H₁₅NO]⁺ 261.1154, found 261.1155.

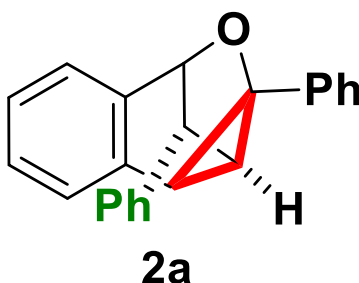
(E)-2-benzoyl-3-(3-((E)-styryl)thiophen-2-yl)acrylonitrile (1aa): Yield: 75 % (286.5 mg), light yellow solid,



m.p. 125-130 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 1/9). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.70 (s, 1H), 7.93 (d, $J = 7.4$ Hz, 2H), 7.75 (d, $J = 5.1$ Hz, 1H), 7.67-7.28 (m, 9H), 7.16 (d, $J = 16.0$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 188.32, 149.37, 144.99, 136.38, 136.03, 134.96, 134.48, 133.09, 131.54, 129.03, 129.00, 128.88, 128.50, 127.02,

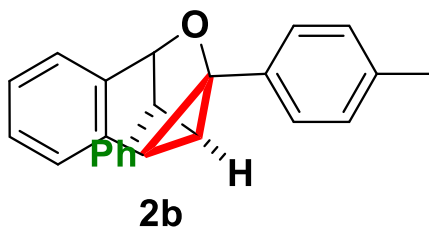
126.27, 118.97, 117.97, 104.58, 24.60; **HRMS (EI):** m/z calcd. for $[\text{M}]^+$ $[\text{C}_{22}\text{H}_{15}\text{NOS}]^+$ 341.4280, found 341.4277.

1,2-diphenyl-1a,2,3,7b-tetrahydro-1H-1,3-epoxycyclopropa[a]naphthalene (2a)



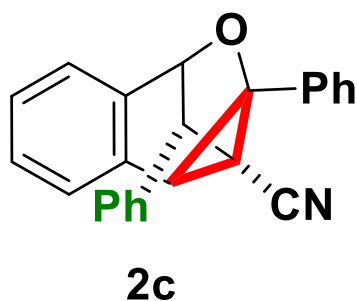
Yield: 91% (56.42 mg), white solid, m.p. 87-89 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44 (ddd, $J = 4.1, 3.3, 1.6$ Hz, 2H), 7.41-7.35 (m, 2H), 7.34-7.26 (m, 2H), 7.18 (td, $J = 7.5, 1.3$ Hz, 1H), 7.08-6.96 (m, 4H), 6.95-6.91 (m, 2H), 6.85-6.81 (m, 1H), 5.31 (d, $J = 6.1$ Hz, 1H), 3.93 (dd, $J = 6.1, 2.8$ Hz, 1H), 2.90 (d, $J = 7.9$ Hz, 1H), 2.57 (ddd, $J = 7.9, 2.8, 0.6$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 137.91, 136.50, 133.02, 131.97, 128.36, 127.88, 127.80, 127.68, 126.93, 126.37, 125.88, 125.04, 124.93, 124.24, 110.46, 79.01, 65.53, 43.31, 27.67, 25.30; **HRMS (EI):** m/z calc'd for $[\text{M}]^+$ $[\text{C}_{23}\text{H}_{18}\text{O}]^+$ 310.1358, found, 310.1360.

2-phenyl-1-(p-tolyl)-1a,2,3,7b-tetrahydro-1H-1,3-epoxycyclopropa[a]naphthalene (2b)



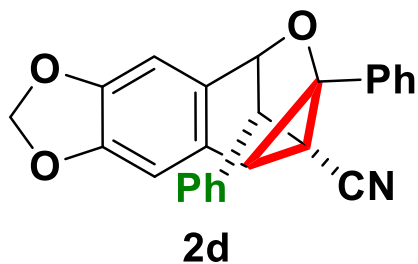
Yield: 93% (60.26 mg), Off-white solid, m.p. 98-100 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.32 (ddd, $J = 6.0, 3.8, 1.2$ Hz, 3H), 7.21-7.14 (m, 3H), 7.08-6.95 (m, 4H), 6.94-6.90 (m, 2H), 6.84-6.79 (m, 1H), 5.29 (d, $J = 6.1$ Hz, 1H), 3.92 (dd, $J = 6.1, 2.7$ Hz, 1H), 2.85 (d, $J = 7.8$ Hz, 1H), 2.52 (ddd, $J = 7.8, 2.8, 0.6$ Hz, 1H), 2.36 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 136.69, 136.59, 134.72, 133.15, 131.98, 129.04, 127.85, 127.80, 127.63, 126.33, 125.84, 125.05, 124.94, 124.22, 78.99, 65.49, 43.31, 27.32, 25.06, 21.12; **HRMS (EI):** m/z calc'd for $[\text{M}]^+ [\text{C}_{24}\text{H}_{20}\text{O}]^+$ 324.1514, found, 324.1452.

1,2-diphenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2c)



Yield: 87% (58.29 mg), light-brown solid, m.p.= 150-152 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.6/9.4). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.70-7.63 (m, 2H), 7.51-7.38 (m, 4H), 7.29 (td, $J = 7.6, 1.2$ Hz, 1H), 7.13-7.06 (m, 4H), 6.92-6.81 (m, 3H), 5.42-5.35 (m, 1H), 4.23 (dd, $J = 6.1, 0.6$ Hz, 1H), 3.59 (s, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 132.62, 132.26, 131.51, 129.36, 128.94, 128.72, 128.66, 128.37, 127.45, 127.34, 127.00, 126.46, 126.35, 124.70, 118.50, 79.12, 69.46, 46.92, 31.03, 24.01; **HRMS (EI):** m/z calc'd for $[\text{M}]^+ [\text{C}_{24}\text{H}_{17}\text{NO}]^+$ 335.1310; found, 335.1307.

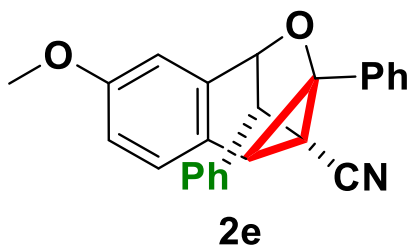
1,2-diphenyl-1,2,3,8b-tetrahydro-1aH-1,3-epoxycyclopropa[5,6]naphtho[2,3-d][1,3]dioxole-1a-carbonitrile (2d)



Yield: 91% (68.9 mg), light-brown solid, m.p.= 195-197 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 1/9). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.69-7.59 (m, 2H), 7.45 (dt, $J = 15.8, 7.1$ Hz, 3H), 7.20-7.08 (m, 3H), 6.97-6.87 (m, 3H), 6.36 (s, 1H), 5.88 (q, $J = 1.5$ Hz, 2H), 5.27 (d, $J = 6.1$

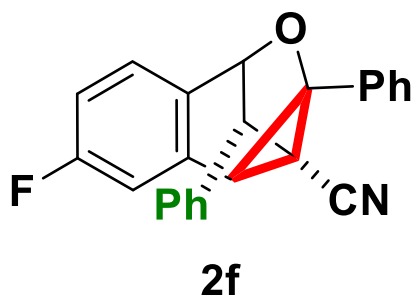
Hz, 1H), 4.16 (d, $J = 5.7$ Hz, 1H), 3.49 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 147.71, 146.78, 132.67, 132.26, 128.90, 128.71, 128.50, 127.51, 127.39, 126.29, 125.32, 122.79, 118.55, 107.15, 106.14, 101.04, 78.84, 68.83, 46.89, 31.22, 23.67; **HRMS (EI)**: m/z calc'd for $[\text{M}]^+$ $[\text{C}_{25}\text{H}_{17}\text{NO}_3]^+$ 379.1208, found, 379.1206.

5-methoxy-1,2-diphenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2e)



Yield: 85% (62.5 mg), Off-white-brown solid, m.p.= 154-156 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.6/9.4). ^1H NMR (400 MHz, CDCl_3) δ 7.69-7.63 (m, 2H), 7.50-7.39 (m, 3H), 7.14-7.07 (m, 3H), 7.01 (d, $J = 2.4$ Hz, 1H), 6.92-6.86 (m, 2H), 6.76 (d, $J = 8.2$ Hz, 1H), 6.62 (dd, $J = 8.2, 2.5$ Hz, 1H), 5.33 (d, $J = 6.0$ Hz, 1H), 4.19 (d, $J = 6.0$ Hz, 1H), 3.78 (s, 3H), 3.53 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 159.85, 132.86, 132.33, 130.64, 128.90, 128.70, 128.40, 127.48, 127.29, 126.33, 125.87, 124.05, 118.53, 112.33, 112.09, 78.73, 69.25, 55.31, 47.29, 31.33, 24.20; **HRMS (EI)**: m/z calc'd for $[\text{M}]^+$ $[\text{C}_{25}\text{H}_{19}\text{NO}_2]^+$ 365.1416, found, 365.1415.

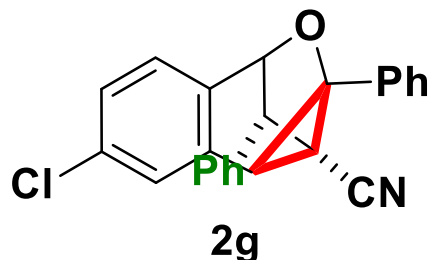
6-fluoro-1,2-diphenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2f)



Yield: 88% (62.13 mg), brown solid, m.p.= 187-189 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). ^1H NMR (400 MHz, CDCl_3) δ 7.71-7.61 (m, 2H), 7.51 – 7.41 (m, 3H), 7.25-7.06 (m, 4H), 6.95-6.74 (m, 4H), 5.38 (d, $J = 6.1$ Hz, 1H), 4.24 (d, $J = 6.1$ Hz, 1H), 3.57 (d, $J = 12.4$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 163.85, 161.39 (d, $J_F = 246$ Hz), 132.39, 131.85, 131.45, 131.36 (d, $J_F = 9$ Hz), 129.12, 129.06, 128.79, 128.54, 127.59, 127.49, 127.36, 127.33, 126.46, 126.38, 126.32, 118.10, 115.81,

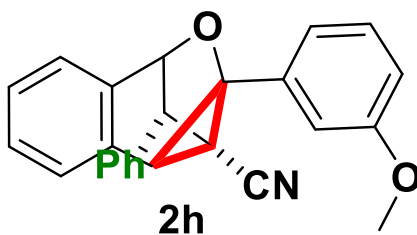
115.52 (d, $J_F = 29$ Hz), 114.07, 113.85, 113.62 (t, $J_F = 23$ Hz), 112.54, 112.25(d, $J_F = 29$ Hz), 78.53, 69.35, 46.94, 46.70, 30.91, 30.88, 30.37, 24.09; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{24}H_{16}FNO]^+$ 353.1216, found, 353.1215.

6-chloro-1,2-diphenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2g)



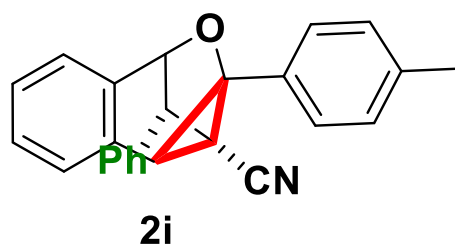
Yield: 79% (58.3 mg), brown solid, m.p. = 124-126 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). **1H NMR** (400 MHz, $CDCl_3$) δ 7.68-7.61 (m, 2H), 7.52-7.38 (m, 4H), 7.30-7.26 (m, 1H), 7.13 (dd, $J = 5.1, 1.9$ Hz, 3H), 6.94-6.76 (m, 3H), 5.35 (d, $J = 5.9$ Hz, 1H), 4.23 (d, $J = 6.1$ Hz, 1H), 3.55 (d, $J = 10.9$ Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 133.05, 133.02, 132.08, 131.85, 129.17, 129.11, 128.81, 128.80, 128.63, 127.94, 127.73, 127.67, 127.56, 127.37, 127.34, 127.18, 126.59, 126.44, 126.35, 126.01, 125.05, 118.06, 78.51, 69.52, 46.80, 46.70, 30.60, 30.50, 24.04; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{24}H_{16}ClNO]^+$ 369.0920, found, 369.0921.

1-(3-methoxyphenyl)-2-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2h)



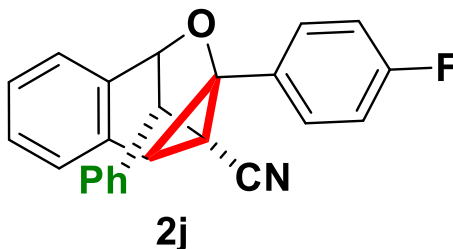
Yield: 90% (65.7 mg), white solid, m.p. = 158-160 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.6/9.4). **1H NMR** (400 MHz, $CDCl_3$) δ 7.47 (d, $J = 7.6$ Hz, 1H), 7.39 (t, $J = 8.2$ Hz, 1H), 7.29 (td, $J = 7.6, 1.2$ Hz, 1H), 7.25-7.19 (m, 2H), 7.10 (ddd, $J = 7.1, 6.3, 1.5$ Hz, 4H), 6.95 (ddd, $J = 8.3, 2.4, 1.1$ Hz, 1H), 6.91-6.81 (m, 3H), 5.38 (d, $J = 6.1$ Hz, 1H), 4.22 (d, $J = 5.7$ Hz, 1H), 3.86 (s, 3H), 3.57 (s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.84, 133.81, 132.59, 131.49, 129.80, 129.34, 128.68, 128.38, 127.45, 127.35, 127.02, 126.48, 124.71, 118.45, 118.28, 114.74, 111.95, 110.46, 79.12, 69.37, 55.36, 46.89, 31.24, 24.17; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{25}H_{19}NO_2]^+$ 365.1416, found, 365.1412.

2-phenyl-1-(p-tolyl)-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2i)



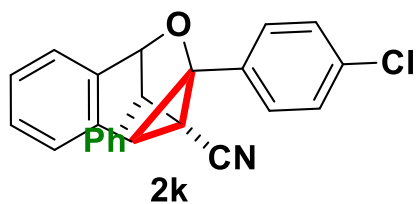
Yield: 89% (62.12 mg), brown solid, m.p. = 146-148 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.59-7.51 (m, 2H), 7.47 (d, $J = 7.5$ Hz, 1H), 7.32-7.26 (m, 3H), 7.13-7.04 (m, 4H), 6.92-6.85 (m, 2H), 6.83 (d, $J = 7.4$ Hz, 1H), 5.36 (d, $J = 6.1$ Hz, 1H), 4.22 (d, $J = 6.1$ Hz, 1H), 3.56 (s, 1H), 2.39 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.95, 132.74, 131.56, 129.47, 129.43, 129.12, 128.62, 128.35, 127.45, 127.30, 126.93, 126.50, 126.43, 124.69, 118.68, 79.12, 69.56, 46.91, 30.81, 23.80, 21.26; **HRMS (EI):** (m/z) calc'd for $[\text{M}]^+$ $[\text{C}_{25}\text{H}_{19}\text{NO}]^+$ 349.1467, found, 349.1465.

1-(4-fluorophenyl)-2-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2j)



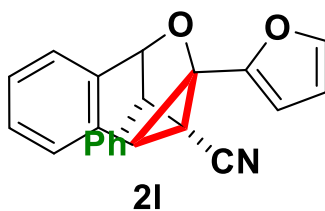
Yield: 83%, (58.6 mg), light-brown solid, m.p.= 141-142 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.68-7.61 (m, 2H), 7.50-7.45 (m, 1H), 7.30 (td, $J = 7.6, 1.2$ Hz, 1H), 7.21-7.06 (m, 6H), 6.91-6.81 (m, 3H), 5.37 (dd, $J = 6.1, 0.4$ Hz, 1H), 4.23 (dd, $J = 6.1, 0.7$ Hz, 1H), 3.55 (s, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 164.32, 161.85 (d, $J_F = 247$ Hz), 132.48, 131.42 (d, $J_F = 106$ Hz), 129.16, 128.73, 128.63, 128.54, 128.41 (t, $J_F = 9$ Hz), 128.08 (d, $J_F = 3$ Hz), 127.41, 127.11 (d, $J_F = 30$ Hz), 126.47, 124.74, 118.45, 115.95, 115.73 (d, $J_F = 22$ Hz),, 79.19, 69.11, 46.86, 30.83, 23.82; **HRMS (EI):** m/z calc'd for $[\text{M}]^+$ $[\text{C}_{24}\text{H}_{16}\text{FNO}]^+$ 353.1216, found, 353.1218.

1-(4-chlorophenyl)-2-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2k)



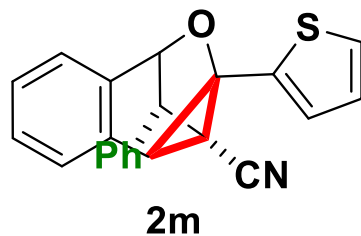
Yield: 78% (57.56 mg), Off-white solid, m.p.= 212-214 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). **¹H NMR** (400 MHz, CDCl₃) δ 7.65-7.57 (m, 2H), 7.52-7.42 (m, 3H), 7.32-7.27 (m, 1H), 7.14-7.07 (m, 4H), 6.90-6.82 (m, 3H), 5.38 (d, *J* = 6.1 Hz, 1H), 4.21 (d, *J* = 5.6 Hz, 1H), 3.56 (s, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 134.95, 132.39, 131.39, 130.93, 129.43, 129.06, 128.97, 128.77, 128.42, 127.67, 127.43, 127.41, 127.17, 126.49, 124.75, 118.27, 79.19, 68.98, 46.85, 31.04, 24.15; **HRMS (EI):** m/z calc'd for [M]⁺ [C₂₄H₁₆ClNO]⁺ 369.0920; found, 369.0922.

1-(furan-2-yl)-2-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2l)



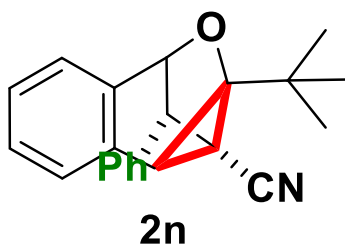
Yield: 77%, (50.1 mg), White solid, m.p. = 105-107 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). **¹H NMR** (400 MHz, CDCl₃) δ 7.57-7.54 (m, 1H), 7.52 (d, *J* = 7.2 Hz, 1H), 7.47-7.39 (m, 5H), 7.36 (ddd, *J* = 7.5, 5.0, 1.4 Hz, 2H), 7.24 (d, *J* = 7.2 Hz, 1H), 6.80-6.70 (m, 1H), 6.49 (dd, *J* = 3.3, 1.9 Hz, 1H), 5.15 (s, 1H), 3.65 (s, 1H), 2.94 (s, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 145.50, 144.01, 136.82, 135.69, 129.14, 129.07, 128.18, 127.82, 127.71, 127.52, 127.32, 122.74, 117.83, 110.94, 110.73, 82.44, 64.99, 48.64, 32.43, 24.63; **HRMS (EI):** m/z calc'd for [M]⁺ [C₂₂H₁₅NO₂]⁺ 325.1103, found, 325.1105.

2-phenyl-1-(thiophen-2-yl)-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2m)



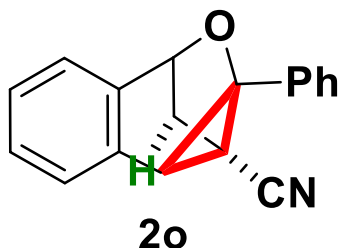
Yield: 83% (56.61 mg), Light-brown solid, m.p.= 144-146 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.8). **¹H NMR** (400 MHz, CDCl₃) δ 7.53-7.45 (m, 2H), 7.44-7.37 (m, 7H), 7.37-7.26 (m, 5H), 7.13-7.06 (m, 6H), 6.90-6.79 (m, 3H), 5.36 (d, *J* = 6.1 Hz, 1H), 5.21 (s, 1H), 4.25 (d, *J* = 6.1 Hz, 1H), 3.57 (d, *J* = 4.8 Hz, 2H), 2.97 (s, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 136.81, 135.63, 134.94, 134.53, 132.41, 131.31, 129.14, 129.05, 128.90, 128.76, 128.40, 128.17, 127.84, 127.44, 127.40, 127.34, 127.30, 127.20, 127.17, 126.86, 126.78, 126.52, 125.86, 125.83, 124.82, 122.74, 118.28, 117.85, 82.52, 79.42, 67.38, 67.03, 48.85, 46.56, 34.73, 32.05, 26.95, 24.84; **HRMS (EI)**: m/z calc'd for [M]⁺ [C₂₂H₁₅NOS]⁺ 341.0874, found, 341.0872.

1-(tert-butyl)-2-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2n)



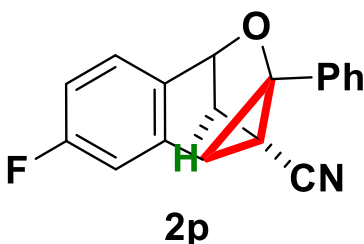
Yield: 65% (40.95 mg), White solid, m.p.= 90-92 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). **¹H NMR** (400 MHz, CDCl₃) δ 7.44-7.37 (m, 1H), 7.27-7.23 (m, 1H), 7.09-7.00 (m, 4H), 6.82-6.71 (m, 3H), 5.15 (dd, *J* = 6.2, 0.5 Hz, 1H), 4.00 (dd, *J* = 6.2, 0.6 Hz, 1H), 3.25 (s, 1H), 1.26 (s, 9H); **¹³C NMR** (101 MHz, CDCl₃) δ 132.86, 131.83, 130.36, 128.34, 128.22, 127.43, 127.15, 126.55, 126.08, 124.50, 119.75, 78.23, 75.07, 47.49, 32.34, 27.61, 26.74, 18.88; **HRMS (EI)**: m/z calc'd for [M]⁺ [C₂₁H₂₂NO]⁺ 315.1623, found, 315.1623.

1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2o)



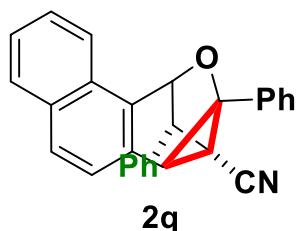
Yield: 76% (39.37 mg), brown solid, m.p.= 88-90 °C; Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). **¹H NMR** (400 MHz, CDCl₃) δ 7.63-7.56 (m, 2H), 7.48-7.36 (m, 5H), 7.33 (td, *J* = 7.4, 1.5 Hz, 1H), 7.25-7.21 (m, 1H), 5.34 (d, *J* = 6.1 Hz, 1H), 3.41 (s, 1H), 2.64 (ddd, *J* = 11.8, 6.0, 0.8 Hz, 1H), 1.73 (d, *J* = 11.8 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 134.98, 132.22, 128.77, 128.73, 128.65, 128.16, 127.25, 127.00, 126.29, 122.57, 118.95, 75.09, 68.87, 31.70, 31.06, 19.26; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₁₈H₁₃NO] 259.0997, found, 259.0998.

6-fluoro-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2p)



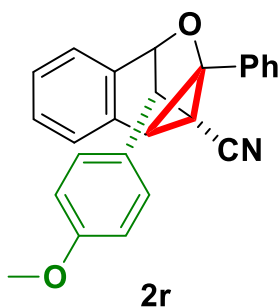
Yield: 71% (39.33 mg), brown solid, m.p.= 114-146 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). **¹H NMR** (400 MHz, CDCl₃) δ 7.62-7.55 (m, 2H), 7.44 (dddd, *J* = 8.6, 7.0, 4.6, 2.3 Hz, 3H), 7.19 (ddd, *J* = 11.1, 8.4, 3.9 Hz, 2H), 7.01 (ddd, *J* = 9.1, 8.3, 2.5 Hz, 1H), 5.34 (d, *J* = 6.0 Hz, 1H), 3.38 (s, 1H), 2.65 (ddd, *J* = 11.8, 6.0, 0.7 Hz, 1H), 1.72 (d, *J* = 11.8 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 164.01, 161.56 (d, *J_F* = 245 Hz), 131.80, 130.89 (d, *J_F* = 3 Hz), 130.32, 130.24 (d, *J_F* = 8 Hz), 128.95, 128.73, 126.33, 124.38, 124.30 (d, *J_F* = 8 Hz), 118.56, 114.58, 114.35 (d, *J_F* = 22 Hz), 113.96, 113.75 (d, *J_F* = 21 Hz), 74.48, 68.75, 31.84, 30.97, 30.95, 19.42; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₁₈H₁₂FNO]⁺ 277.0903, found, 277.0901.

1,9-diphenyl-1,1a,8,9-tetrahydro-9aH-1,8-epoxycyclopropa[a]phenanthrene-9a-carbonitrile (2q)



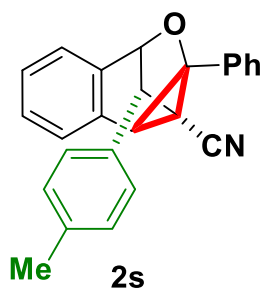
Yield: 72% (55.44 mg), Light-brown solid, m.p.= 110-112 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.3/9.7). **¹H NMR** (597 MHz, CDCl₃) δ 8.04 (d, *J* = 7.8 Hz, 1H), 7.96-7.90 (m, 2H), 7.75-7.70 (m, 2H), 7.61-7.56 (m, 2H), 7.54-7.47 (m, 3H), 7.46-7.34 (m, 6H), 6.06 (s, 1H), 3.72 (s, 1H), 2.99 (s, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 137.18, 132.54, 132.01, 131.56, 129.21, 128.93, 128.86, 128.82, 128.12, 127.88, 127.24, 127.07, 126.05, 126.01, 125.81, 125.15, 121.46, 118.17, 77.77, 69.50, 48.75, 34.51, 26.42; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₂₈H₁₉NO]⁺ 385.1467, found, 385.1465.

2-(4-methoxyphenyl)-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2r)



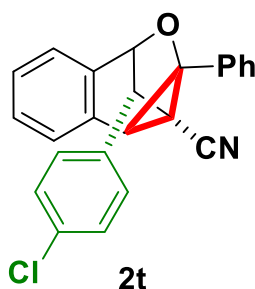
Yield: 91% (66.43 mg), Off-white solid, m.p.= 158-160 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.6/9.4). **¹H NMR** (400 MHz, CDCl₃) δ 7.71-7.61 (m, 2H), 7.53-7.37 (m, 4H), 7.31 (td, *J* = 7.6, 1.2 Hz, 1H), 7.13 (td, *J* = 7.5, 1.0 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 1H), 6.77 (d, *J* = 8.6 Hz, 2H), 6.66-6.59 (m, 2H), 5.33 (d, *J* = 6.1 Hz, 1H), 4.16 (d, *J* = 6.1 Hz, 1H), 3.68 (s, 3H), 3.56 (s, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 158.69, 132.34, 131.65, 129.47, 128.89, 128.71, 128.62, 128.59, 127.05, 126.42, 126.33, 124.84, 124.47, 118.56, 113.82, 79.09, 69.33, 55.08, 46.27, 31.00, 24.41; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₂₅H₁₉NO₂]⁺ 365.1416, found, 365.1414.

1-phenyl-2-(p-tolyl)-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2s)



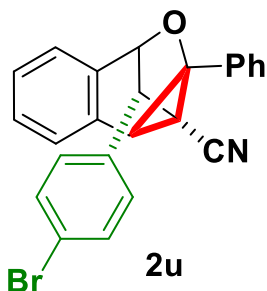
Yield: 87% (60.73), White solid, m.p. = 154-156 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). **¹H NMR** (400 MHz, CDCl₃) δ 7.69-7.62 (m, 2H), 7.51-7.44 (m, 3H), 7.42 (dd, *J* = 4.9, 3.6 Hz, 1H), 7.30 (td, *J* = 7.6, 1.2 Hz, 1H), 7.12 (td, *J* = 7.5, 1.1 Hz, 1H), 6.88 (dd, *J* = 12.4, 7.7 Hz, 3H), 6.75 (d, *J* = 8.1 Hz, 2H), 5.35 (d, *J* = 6.1 Hz, 1H), 4.18 (d, *J* = 6.1 Hz, 1H), 3.56 (s, 1H), 2.19 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 136.98, 132.35, 131.63, 129.47, 129.44, 129.08, 128.89, 128.70, 128.61, 127.36, 126.98, 126.43, 126.34, 124.79, 118.55, 79.09, 69.33, 46.63, 31.01, 24.23, 20.96; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₂₄H₂₀O]⁺ 324.1514, found, 324.1516.

2-(4-chlorophenyl)-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2t)



Yield: 76% (58.6 mg), Off-white solid, m.p.= 175-177 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). **¹H NMR** (400 MHz, CDCl₃) δ 7.70-7.62 (m, 2H), 7.54-7.38 (m, 4H), 7.32 (td, *J* = 7.6, 1.2 Hz, 1H), 7.14 (td, *J* = 7.5, 1.1 Hz, 1H), 7.10-7.02 (m, 2H), 6.83 (dd, *J* = 18.4, 7.9 Hz, 3H), 5.36 (d, *J* = 6.1 Hz, 1H), 4.18 (d, *J* = 6.1 Hz, 1H), 3.58 (s, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 133.29, 132.02, 131.26, 131.20, 129.20, 129.04, 128.89, 128.81, 128.76, 128.63, 127.24, 126.52, 126.35, 124.78, 118.23, 78.90, 69.53, 46.42, 30.98, 24.02; **HRMS (EI)**: *m/z* calc'd for [M]⁺ [C₂₄H₁₆ClNO]⁺ 369.0920, found, 369.0921.

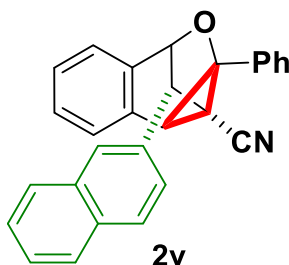
2-(4-bromophenyl)-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2u)



Yield: 79% (56.1), Off-white solid, m.p.= 116-118 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.4/9.6). **¹H NMR** (400 MHz, CDCl₃) δ 7.69-7.61 (m, 2H), 7.53-7.39 (m, 4H),

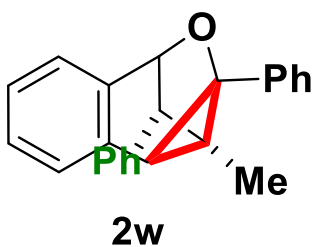
7.32 (d, $J = 1.2$ Hz, 1H), 7.25-7.19 (m, 2H), 7.18-7.11 (m, 1H), 6.86 (d, $J = 7.4$ Hz, 1H), 6.78-6.72 (m, 2H), 5.36 (d, $J = 6.1$ Hz, 1H), 4.17 (d, $J = 6.1$ Hz, 1H), 3.58 (s, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 132.00, 131.79, 131.58, 131.17, 129.15, 129.05, 128.91, 128.76, 127.27, 126.51, 126.35, 124.79, 121.43, 118.21, 109.89, 78.83, 69.29, 46.49, 30.98, 23.98; **HRMS (EI)**: m/z calc'd for $[\text{M}]^+$ $[\text{C}_{24}\text{H}_{16}\text{ClNO}]^+$ 413.0415, found, 413.0415.

2-(naphthalen-2-yl)-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2v)



Yield: 88% (67.76), Off-white solid, m.p.= 110-112 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.3/9.7). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.69 (ddd, $J = 7.4, 4.8, 3.2$ Hz, 3H), 7.65-7.57 (m, 2H), 7.49 (ddd, $J = 8.2, 3.5, 1.9$ Hz, 3H), 7.46-7.33 (m, 4H), 7.28-7.24 (m, 1H), 7.06-6.95 (m, 2H), 6.81 (d, $J = 7.4$ Hz, 1H), 5.48 (d, $J = 6.1$ Hz, 1H), 4.38 (d, $J = 6.1$ Hz, 1H), 3.67 (s, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 133.07, 132.44, 132.26, 131.46, 130.21, 129.39, 128.98, 128.75, 128.73, 128.14, 127.66, 127.46, 127.10, 126.62, 126.45, 126.39, 126.12, 125.92, 125.24, 124.74, 118.54, 79.13, 69.55, 47.12, 31.14, 24.18; **HRMS (EI)**: m/z calc'd for $[\text{M}]^+$ $[\text{C}_{28}\text{H}_{19}\text{NO}]^+$ 385.1467, found, 385.1465.

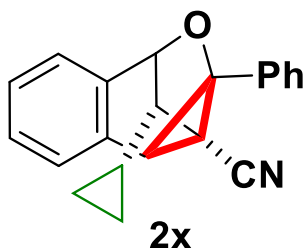
1a-methyl-1,2-diphenyl-1a,2,3,7b-tetrahydro-1H-1,3-epoxycyclopropa[a]naphthalene (2w)



Yield: 80% (51.84), White solid, m.p.= 111-113 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 (dd, $J = 8.3, 1.3$ Hz, 2H), 7.41 (ddd, $J = 6.2, 3.9, 3.4$ Hz, 3H), 7.33 (d, $J = 7.4$ Hz, 1H), 7.22 (d, $J = 1.3$ Hz, 1H), 7.08-7.02 (m, 3H), 6.98 (d, $J = 1.2$ Hz, 1H), 6.80 (dd, $J = 7.4, 2.3$ Hz, 3H), 5.19 (d, $J = 6.1$ Hz, 1H), 3.65 (d, $J = 6.1$ Hz, 1H), 2.80 (s, 1H), 1.25 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 135.65, 135.22, 134.37, 131.76, 128.41, 128.24, 127.90, 127.68, 127.64,

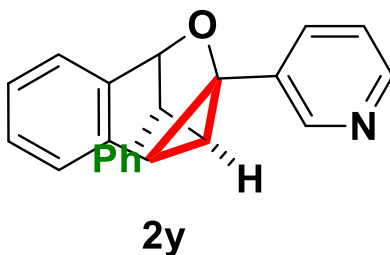
126.40, 125.63, 124.94, 124.35, 79.30, 69.06, 49.23, 29.69, 29.29, 13.78; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{24}H_{20}O]^+$ 324.1514, found, 324.1515.

2-cyclopropyl-1-phenyl-1,2,3,7b-tetrahydro-1aH-1,3-epoxycyclopropa[a]naphthalene-1a-carbonitrile (2x)



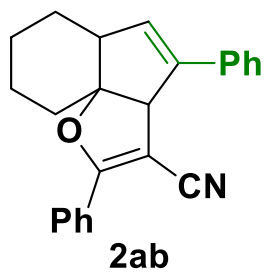
Yield: 82% (49 mg), White solid, m.p.= 150-152 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.2/9.8). **¹H NMR** (400 MHz, $CDCl_3$) δ 7.56 (d, J = 7.2 Hz, 2H), 7.47-7.33 (m, 6H), 7.28 (d, J = 7.0 Hz, 1H), 5.16 (d, J = 5.9 Hz, 1H), 3.37 (s, 1H), 2.09 (dd, J = 10.1, 5.9 Hz, 1H), 0.51-0.43 (m, 2H), 0.35-0.28 (m, 1H), 0.15 (ddd, J = 11.3, 9.3, 4.9 Hz, 1H), 0.29 (tdd, J = 7.8, 6.6, 4.0 Hz, 1H); **¹³C NMR** (101 MHz, $CDCl_3$) δ 132.84, 132.48, 129.26, 128.66, 128.59, 126.90, 126.88, 126.13, 124.39, 118.88, 77.80, 69.59, 47.87, 30.72, 24.09, 6.58, 3.63, 2.34; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{21}H_{17}NO]^+$ 299.1310, found, 299.1310.

3-(2-phenyl-1a,2,3,7b-tetrahydro-1H-1,3-epoxycyclopropa[a]naphthalen-1-yl)pyridine (2y)



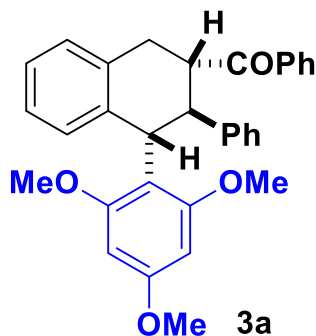
Yield: 88% (53.49 mg), White solid, m.p.= 138-140 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 1.2/8.8). **¹H NMR** (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.53 (d, J = 3.6 Hz, 1H), 7.83 – 7.69 (m, 1H), 7.37-7.17 (m, 3H), 7.11-6.97 (m, 4H), 6.96-6.89 (m, 2H), 6.84 (d, J = 7.3 Hz, 1H), 5.32 (d, J = 6.1 Hz, 1H), 3.94 (dd, J = 6.0, 2.7 Hz, 1H), 2.93 (d, J = 7.9 Hz, 1H), 2.61 (dd, J = 7.9, 2.7 Hz, 1H); **¹³C NMR** (101 MHz, $CDCl_3$) δ 148.13, 146.41, 135.98, 133.90, 132.74, 132.36, 131.80, 127.92, 127.85, 127.72, 126.49, 125.94, 125.34, 124.28, 123.08, 78.91, 63.70, 43.31, 27.40, 25.20; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{22}H_{17}NO]^+$ 311.1310, found, 311.1312.

2,4-diphenyl-3a,5a,6,7,8,9-hexahydroindeno[7a,1-b]furan-3-carbonitrile (2ab)



Yield: 94% (63.7 mg), White solid, m.p. = 144-146 °C. Purification by flash column chromatography on silica gel (eluent: EtOAc/Petroleum ether = 0.5/9.5). $^1\text{H NMR}$ (597 MHz, CDCl_3) δ 8.04-7.92 (m, 2H), 7.50-7.40 (m, 3H), 7.34 (ddd, $J = 7.8, 4.5, 1.7$ Hz, 2H), 7.27-7.22 (m, 3H), 5.57 (t, $J = 2.1$ Hz, 1H), 4.10 (dd, $J = 5.4, 2.3$ Hz, 1H), 3.31 (d, $J = 2.3$ Hz, 1H), 2.65-2.52 (m, 1H), 2.41-2.30 (m, 1H), 2.24 (d, $J = 13.8$ Hz, 1H), 2.05-1.90 (m, 2H), 1.82 (dd, $J = 10.6, 3.2$ Hz, 1H), 1.60 (td, $J = 13.6, 4.2$ Hz, 1H), 1.53-1.45 (m, 1H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 164.48, 143.97, 143.60, 131.15, 128.83, 128.59, 128.55, 127.22, 127.13, 126.84, 126.71, 118.17, 100.96, 84.01, 60.59, 57.53, 38.05, 26.92, 26.34, 22.39; **HRMS (EI)**: m/z calc'd for $[\text{M}]^+$ $[\text{C}_{24}\text{H}_{21}\text{NO}]^+$ 339.1623, found, 339.1621.

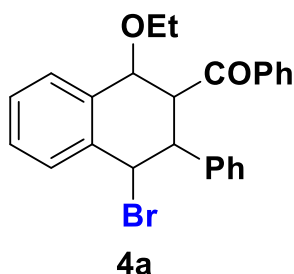
phenyl(3-phenyl-4-(2,4,6-trimethoxyphenyl)-1,2,3,4-tetrahydronaphthalen-2-yl)methanone (3a)



To a 25 mL Schlenk tube with a magnetic bar were added **2a** (1.0 eq, 0.1 mmol), 1,3,5-trimethoxy benzene (1.5 equiv) the corresponding solvent 1,2-DCE (2.0 mL) and TfOH (0.3 equiv) at r.t. The reaction mixture was stirred at room temperature. After the reaction was finished (5h), the reaction mixture was concentrated in vacuo. The crude product was purified by column chromatography (silica gel, EtOAc/petroleum ether = 1:5) to afford **3a** as off-white solid (89%, 42.5 mg), m.p.= 182-184 °C. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.84 (dt, $J = 8.5, 1.7$ Hz, 2H), 7.76 (dd, $J = 8.4, 1.3$ Hz, 1H), 7.66 (dd, $J = 8.4, 7.1$ Hz, 1H), 7.61 (s, 1H), 7.57-7.50 (m, 2H), 7.48-7.41 (m, 3H), 7.40-7.29 (m, 3H), 7.20 (ddt, $J = 8.1, 6.2, 1.8$ Hz, 3H), 7.17-7.10 (m, 1H), 7.09-7.03 (m, 2H), 4.75 (d, $J = 3.5$ Hz, 2H), 4.39 (ddd, $J = 14.9, 9.3, 5.5$ Hz, 1H), 4.01 (dd, $J = 8.3, 3.5$ Hz, 1H), 3.39 (ddt, $J = 21.0, 9.4, 7.0$ Hz, 1H), 3.11 (dq, $J = 9.2, 7.0$ Hz, 1H), 0.95 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 202.89, 159.58, 159.11,

143.20, 141.18, 137.36, 134.93, 132.65, 128.37, 128.12, 128.06, 127.63, 127.31, 126.00, 125.64, 124.71, 113.71, 92.09, 90.58, 55.73, 55.07, 47.76, 47.50, 42.21, 35.36; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{30}H_{26}N_2O_3]^+$ 478.2144, found, 478.2131.

(4-bromo-1-ethoxy-3-phenyl-1,2,3,4-tetrahydronaphthalen-2-yl)(phenyl)methanone (4a)



To a 25 mL Schlenk tube with a magnetic bar were added **2a** (1.0 eq, 0.1 mmol), the corresponding solvent ethanol (2.0 mL) and NBS (1.0 equiv) at r.t. The reaction mixture was stirred at room temperature under dark. After the reaction was finished (10h), the reaction mixture was concentrated in vacuo. The crude product was purified by column chromatography (silica gel, EtOAc/petroleum ether = 1:3) to afford **4a** (d.r. = 5:1) as light-purple solid (91%, 39.5 mg), m.p.= 180-182 °C. **¹H NMR** (400 MHz, CDCl₃) δ 7.86 – 7.82 (m, 2H), 7.58 – 7.50 (m, 2H), 7.48-7.43 (m, 3H), 7.39-7.35 (m, 1H), 7.34-7.29 (m, 1H), 7.23-7.17 (m, 2H), 7.14 (ddd, J = 7.2, 3.7, 1.4 Hz, 1H), 7.08-7.04 (m, 2H), 4.79-4.71 (m, 2H), 4.42 (dd, J = 8.3, 3.7 Hz, 1H), 4.01 (dd, J = 8.3, 3.5 Hz, 1H), 3.74 (d, J = 10.0 Hz, 1H), 3.41-3.33 (m, 1H), 3.11 (dq, J = 9.2, 7.0 Hz, 1H), 0.96 (t, J = 7.0 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 197.41, 144.46, 139.81, 137.04, 135.22, 132.84, 132.67, 129.74, 129.06, 128.67, 128.65, 128.47, 128.28, 128.23, 128.14, 127.95, 127.70, 127.55, 127.44, 127.24, 126.87, 126.50, 125.81, 79.60, 74.71, 72.97, 66.62, 64.56, 55.29, 54.27, 52.11, 45.80, 15.35, 14.73; **HRMS (EI)**: m/z calc'd for $[M]^+$ $[C_{25}H_{23}BrO_2]^+$ 434.0881, found, 434.0883.

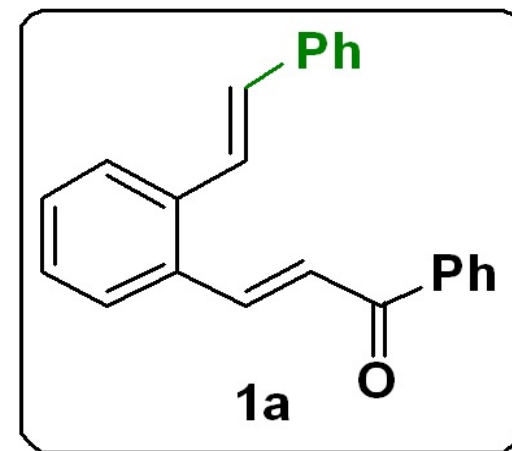
5. REFERENCE

1. B. S. Gore, C.-H. Chiang, C. C. Lee, Y.-L. Shih, J.-J. Wang, *Org. Lett.* **2020**, *22*, 7848-7852.
2. M. R. Becker, A. D. Richardson and C. S. Schindler, *Nat Commun.*, 2019, **10**, 5095.

U0376-1
TPG-476

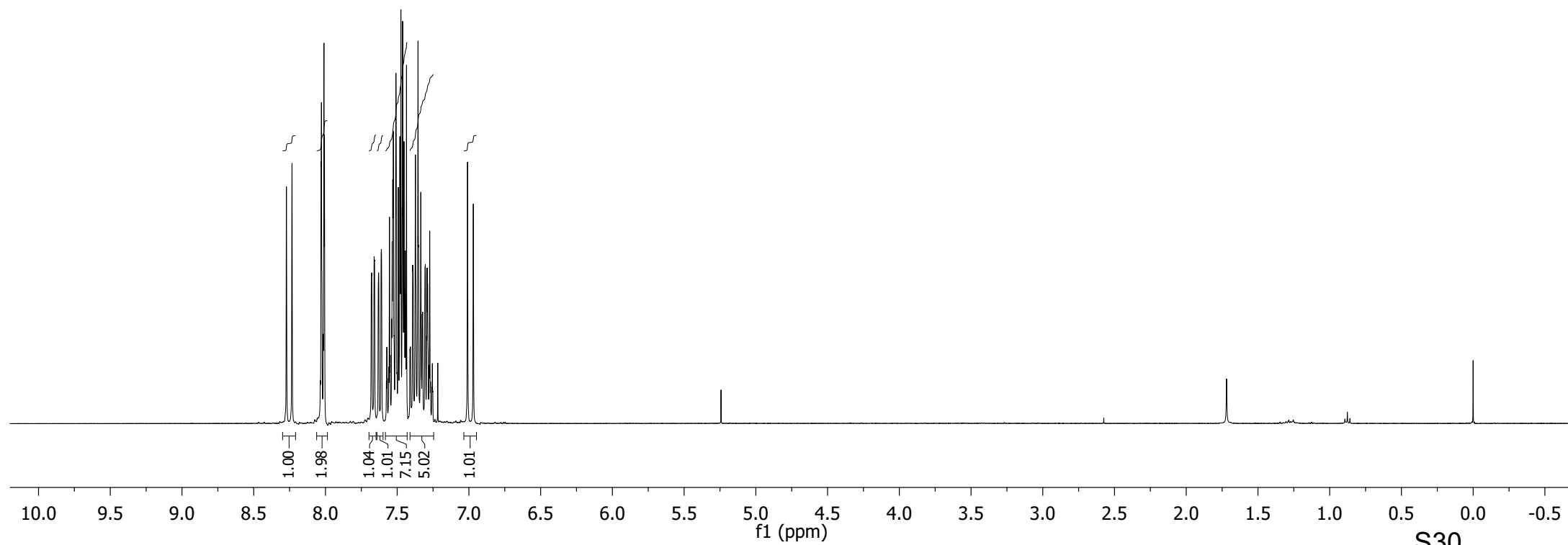
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8.01
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7.51
7.49
7.48
7.47
7.46
7.46
7.45
7.44
7.44
7.37
7.35
7.35
7.34
7.27
7.01
7.01
6.94

Solvent CDCl_3
Spectrometer Frequency 400.28



1.72

0.00



S30

U0376-2
TPG-476

—190.10

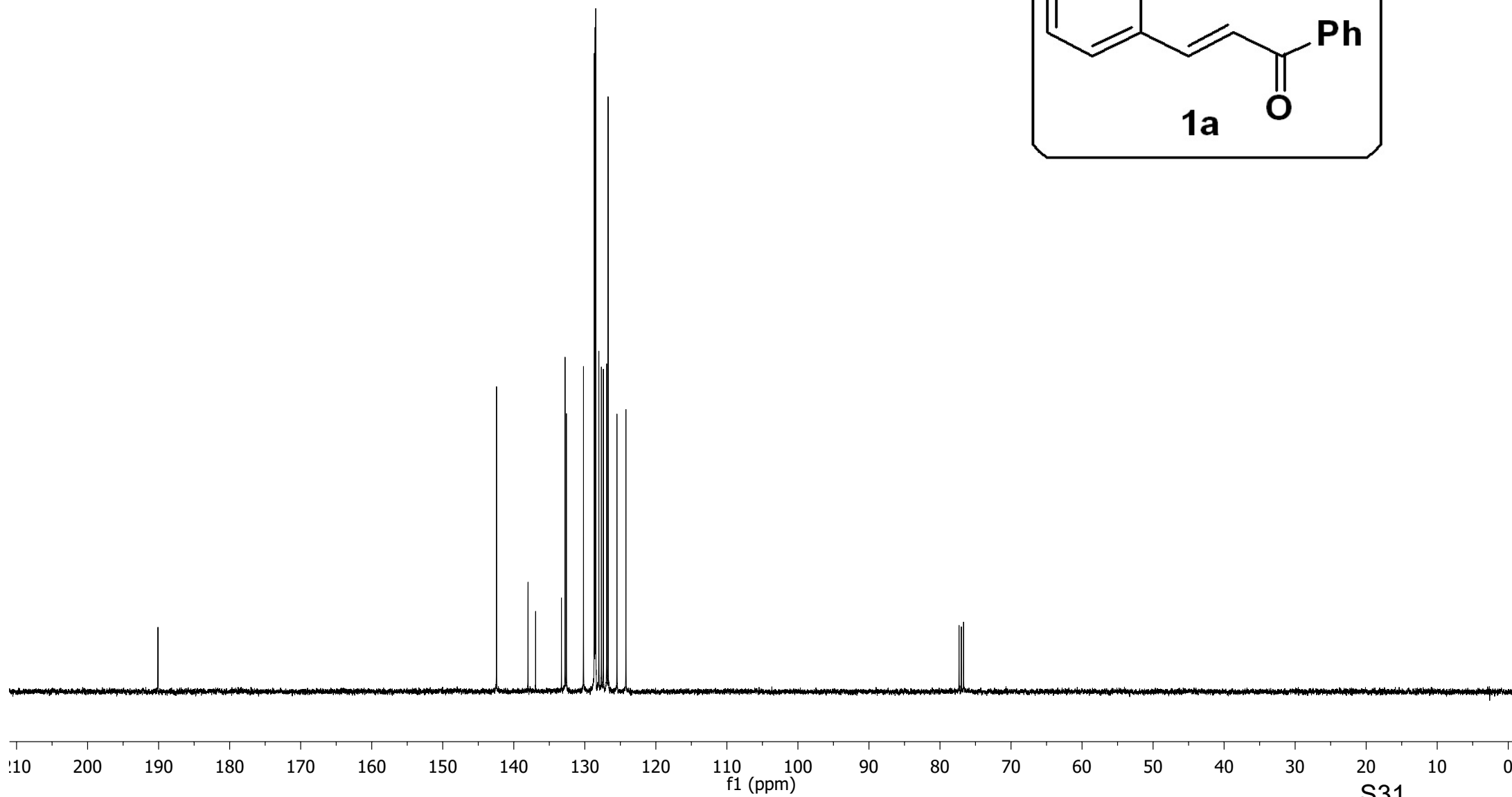
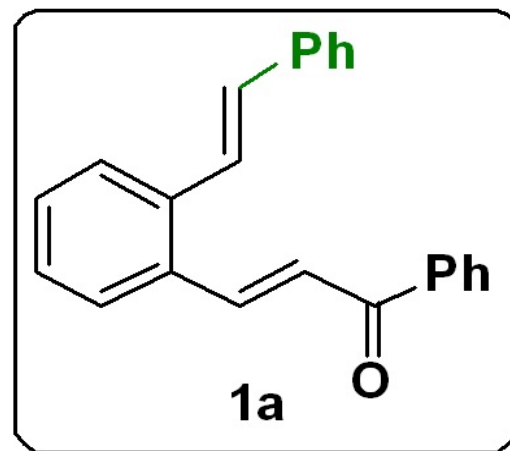
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130.19
128.66
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127.39
126.93
126.72
125.48
124.21

77.32
77.00
76.68

Solvent

CDCl₃

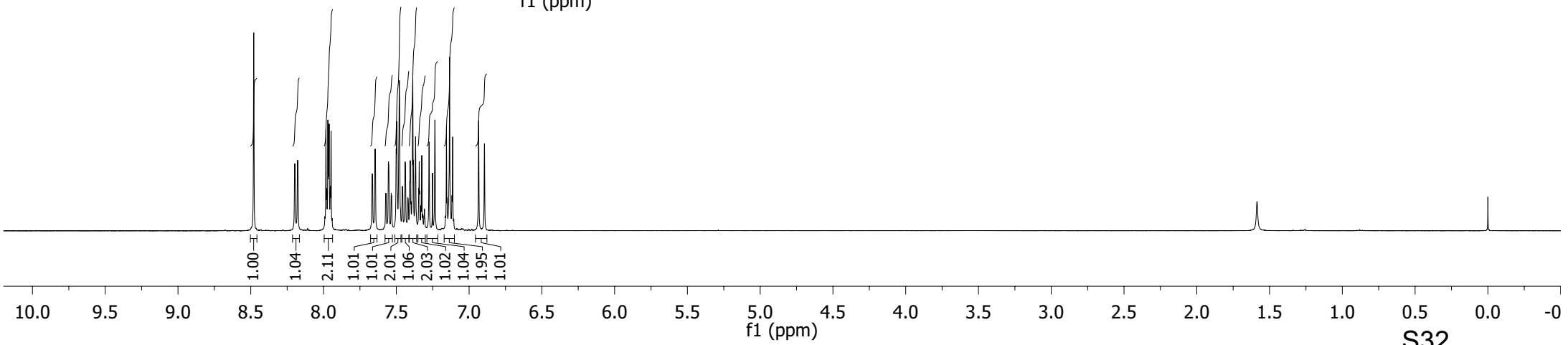
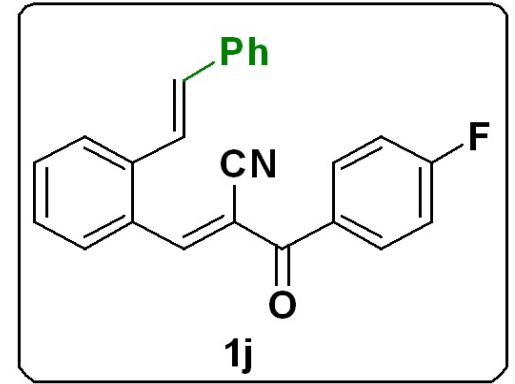
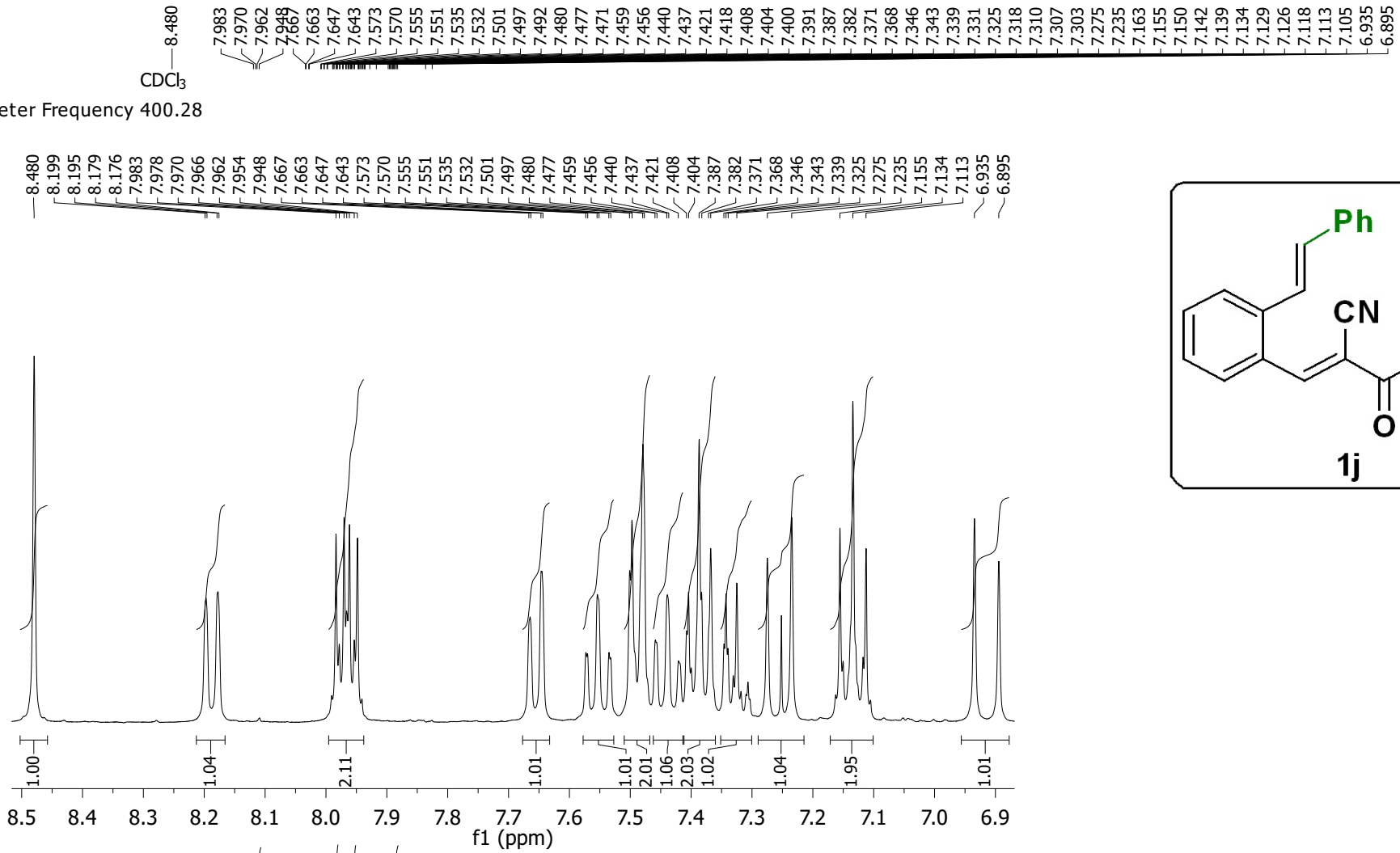
Spectrometer Frequency 100.66



S31

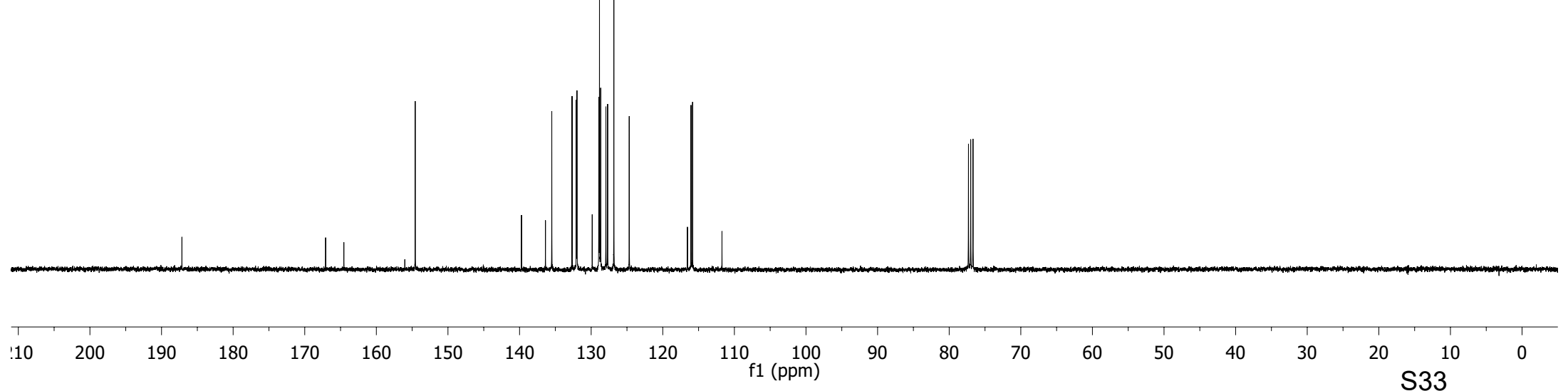
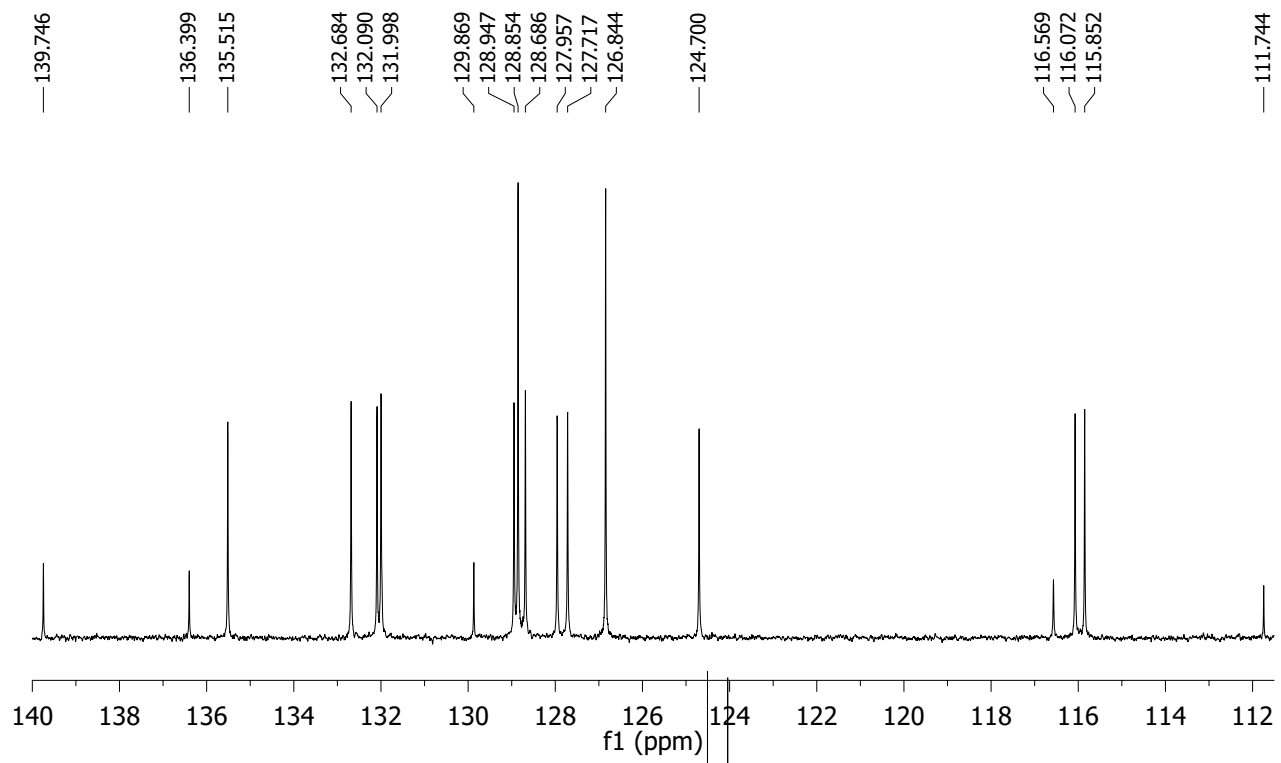
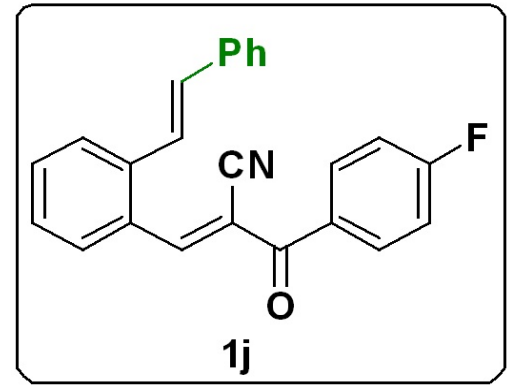
Solvent
Spectrometer Frequency 400.28

CDCl₃



Solvent CDCl_3
Spectrometer Frequency 100.66

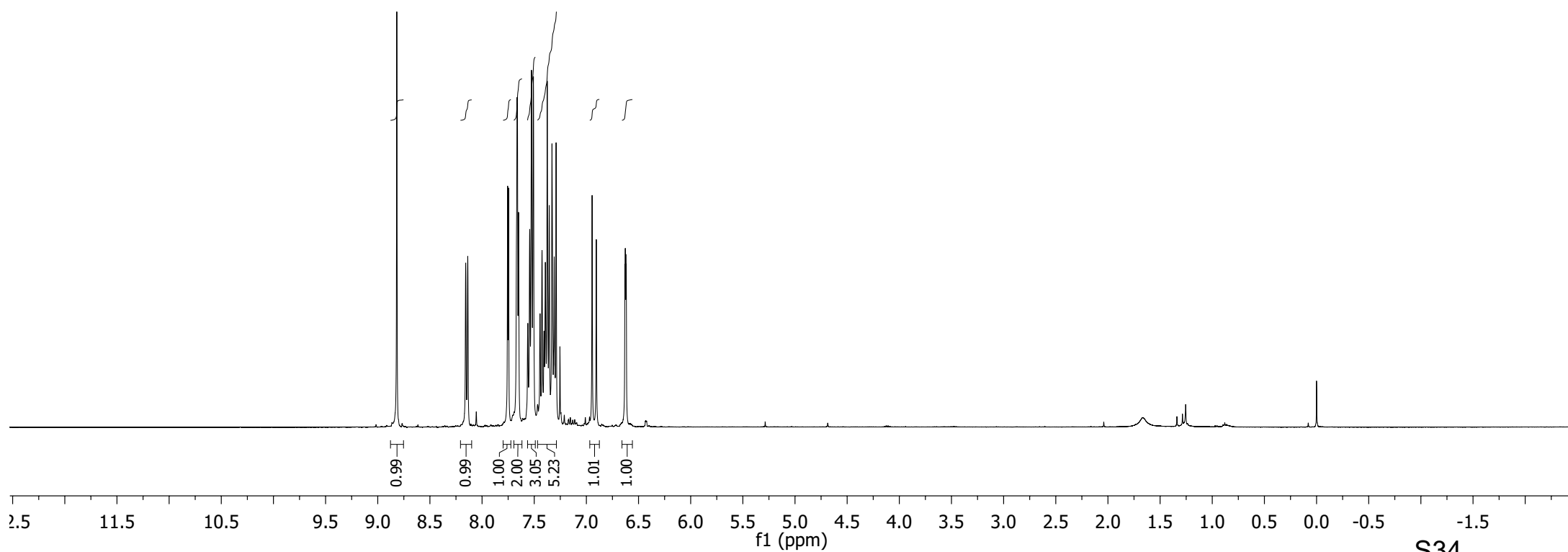
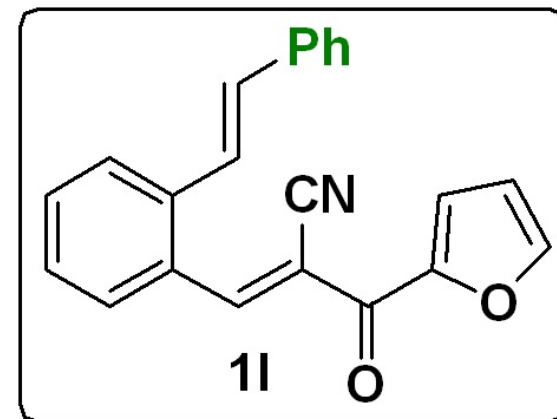
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154.591
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128.854
128.686
127.957
127.717
126.844
124.700
116.072
115.852
111.744



U0013-4
LW-ds-2

Solvent CDCl_3
Spectrometer Frequency 400.28

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8.16
8.14
7.75
7.75
7.66
7.66
7.65
7.56
7.54
7.53
7.51
7.44
7.43
7.41
7.39
7.38
7.36
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6.63
6.62
6.62
0.00



S34

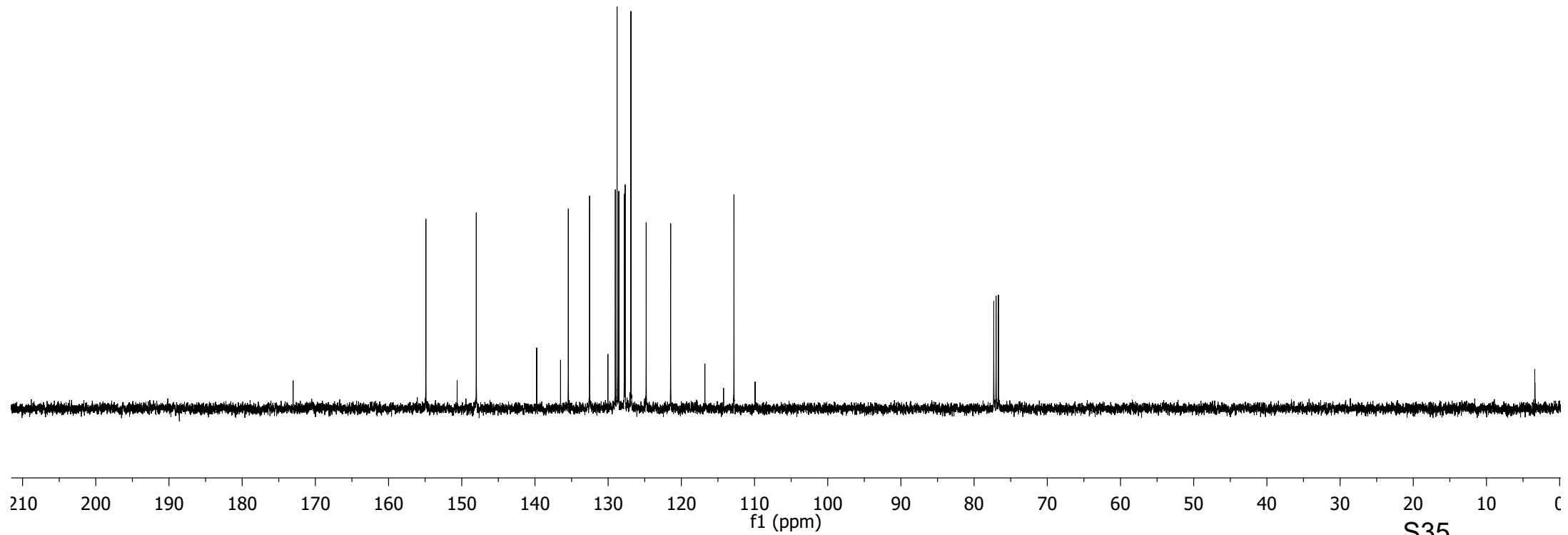
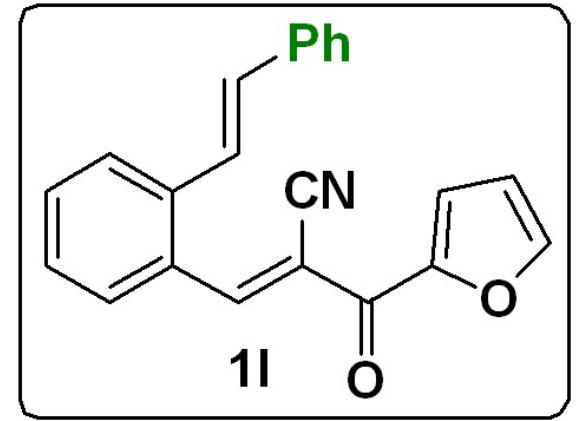
U0013-5
LW-ds-2

Solvent CDCl_3
Spectrometer Frequency 100.66

173.04

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150.63
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136.50
135.44
132.53
130.03
129.02
128.78
128.55
127.79
127.68
126.89
124.81
121.46
116.78
114.35
112.81
109.97
109.92

77.31
77.00
76.68



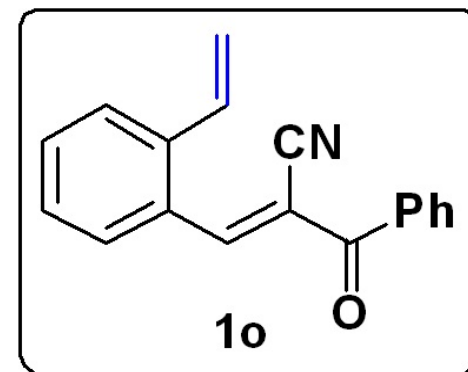
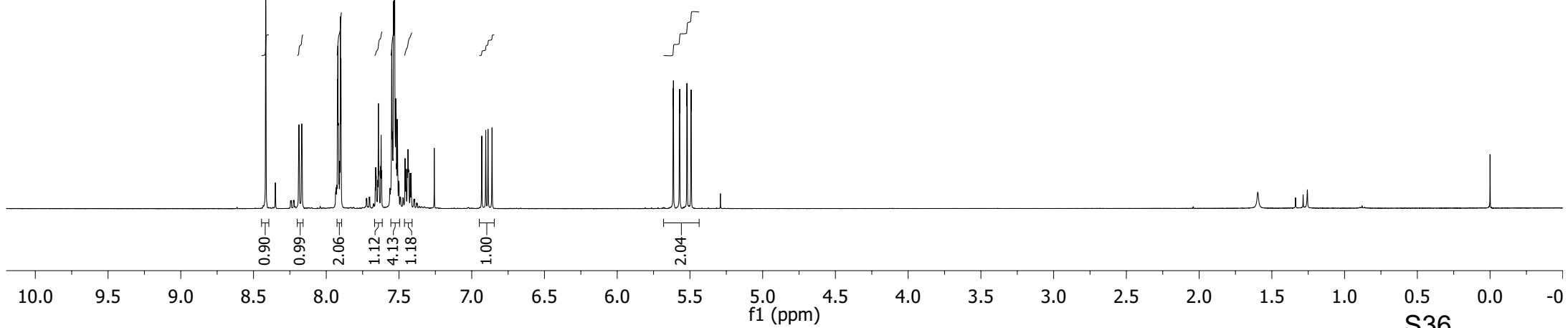
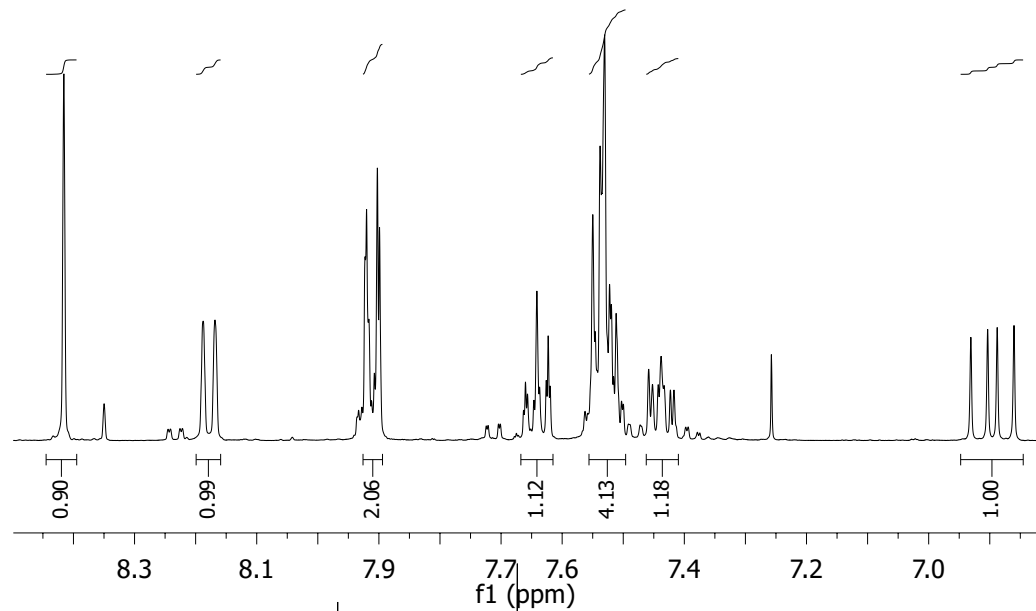
S35

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8.187
8.170
8.167
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7.903
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7.899
7.660
7.660
7.623
7.550
7.546
7.538
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6.888
6.860

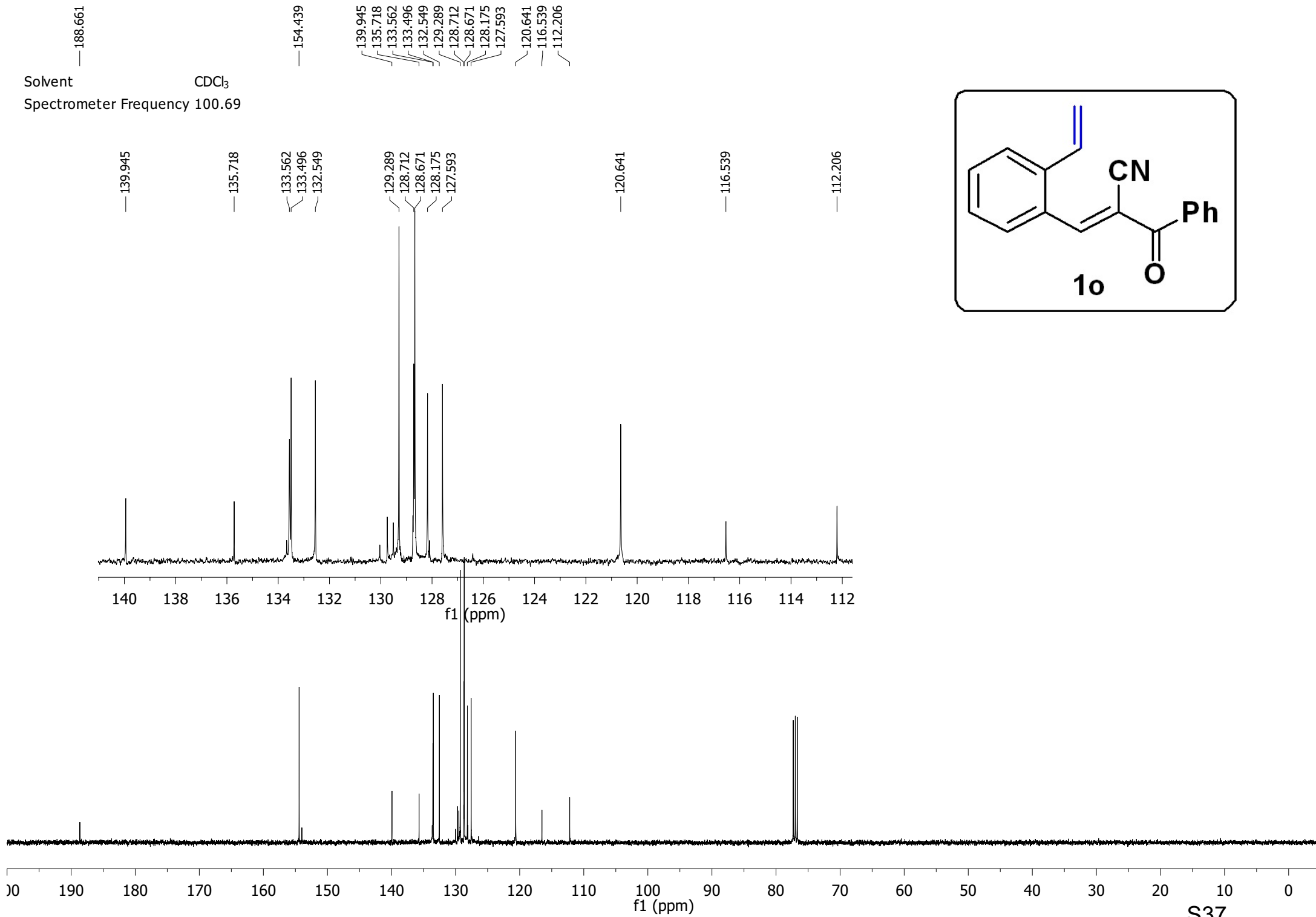
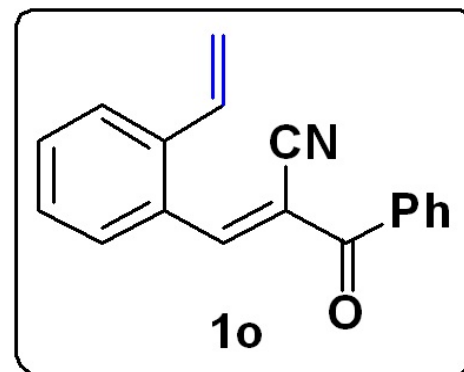
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5.519
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5.491

Solvent CDCl_3
Spectrometer Frequency 400.40

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8.189
8.187
8.170
8.167
7.923
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7.916
7.903
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7.899
7.660
7.660
7.623
7.550
7.546
7.538
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7.523
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7.510
7.458
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6.931
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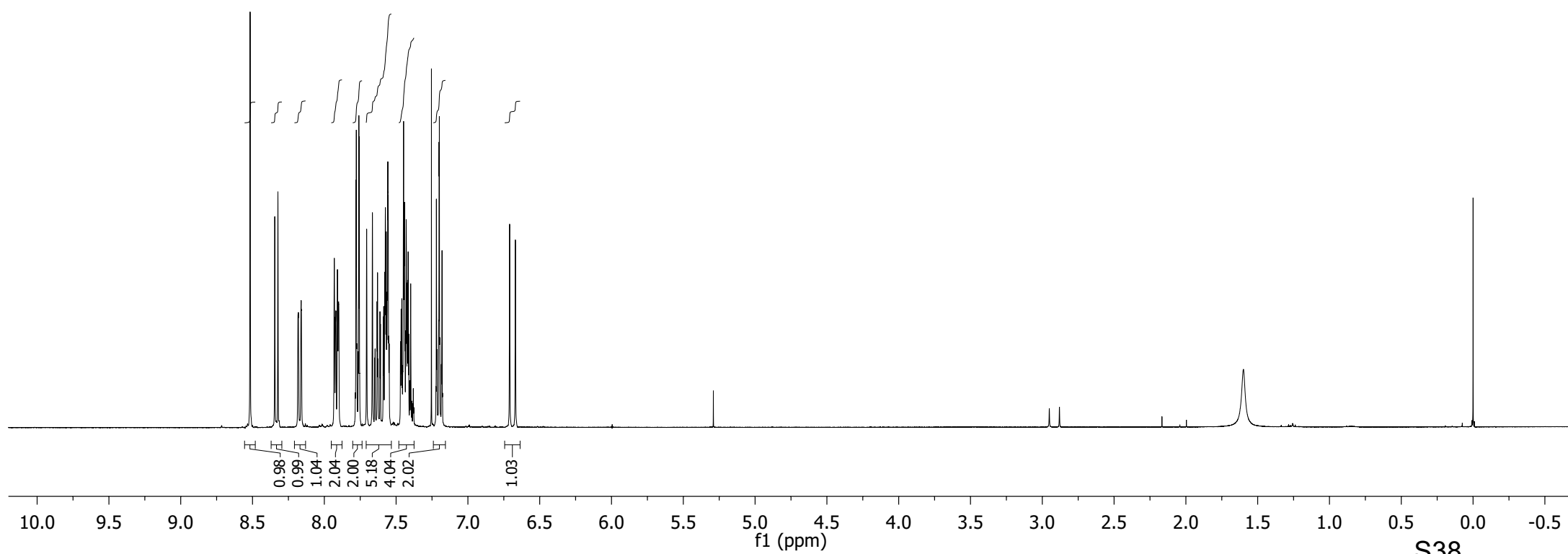
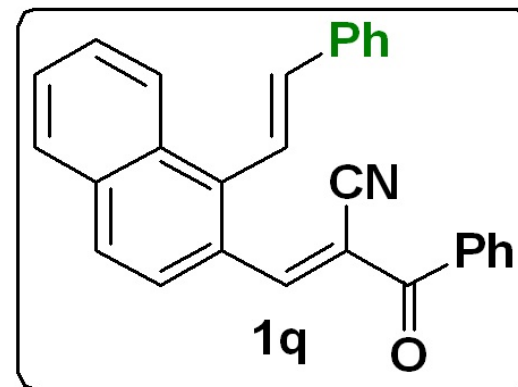


Solvent CDCl_3
Spectrometer Frequency 100.69



MCP
LW
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7.91
7.78
7.78
7.76
7.76
7.71
7.66
7.63
7.57
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7.44
7.44
7.43
7.42
7.25
7.22
7.20
7.20
7.18
6.71
6.67

Solvent CDCl₃
Spectrometer Frequency 400.40



S38

M0036-2
LW-ds-5

— 189.86

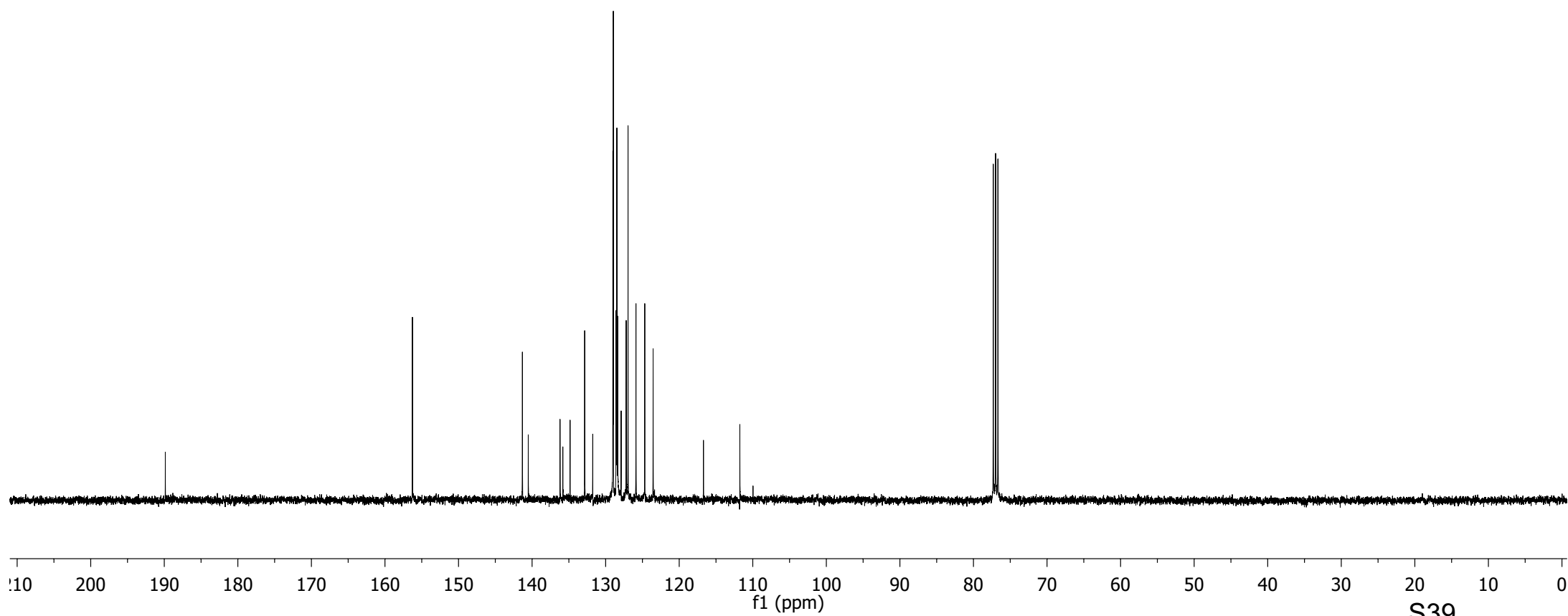
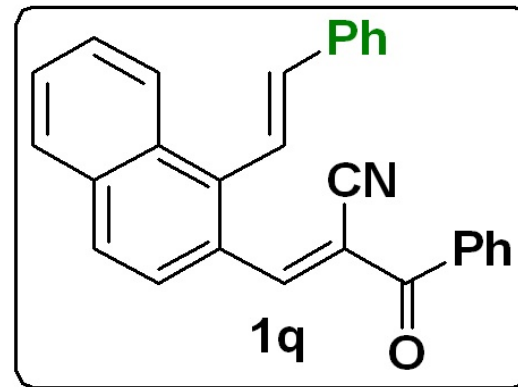
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— 127.89
— 127.20
— 126.96
— 125.88
— 124.68
— 123.36
— 111.76

{ 77.31
— 76.99
— 76.67

Solvent

CDCl₃

Spectrometer Frequency 100.69

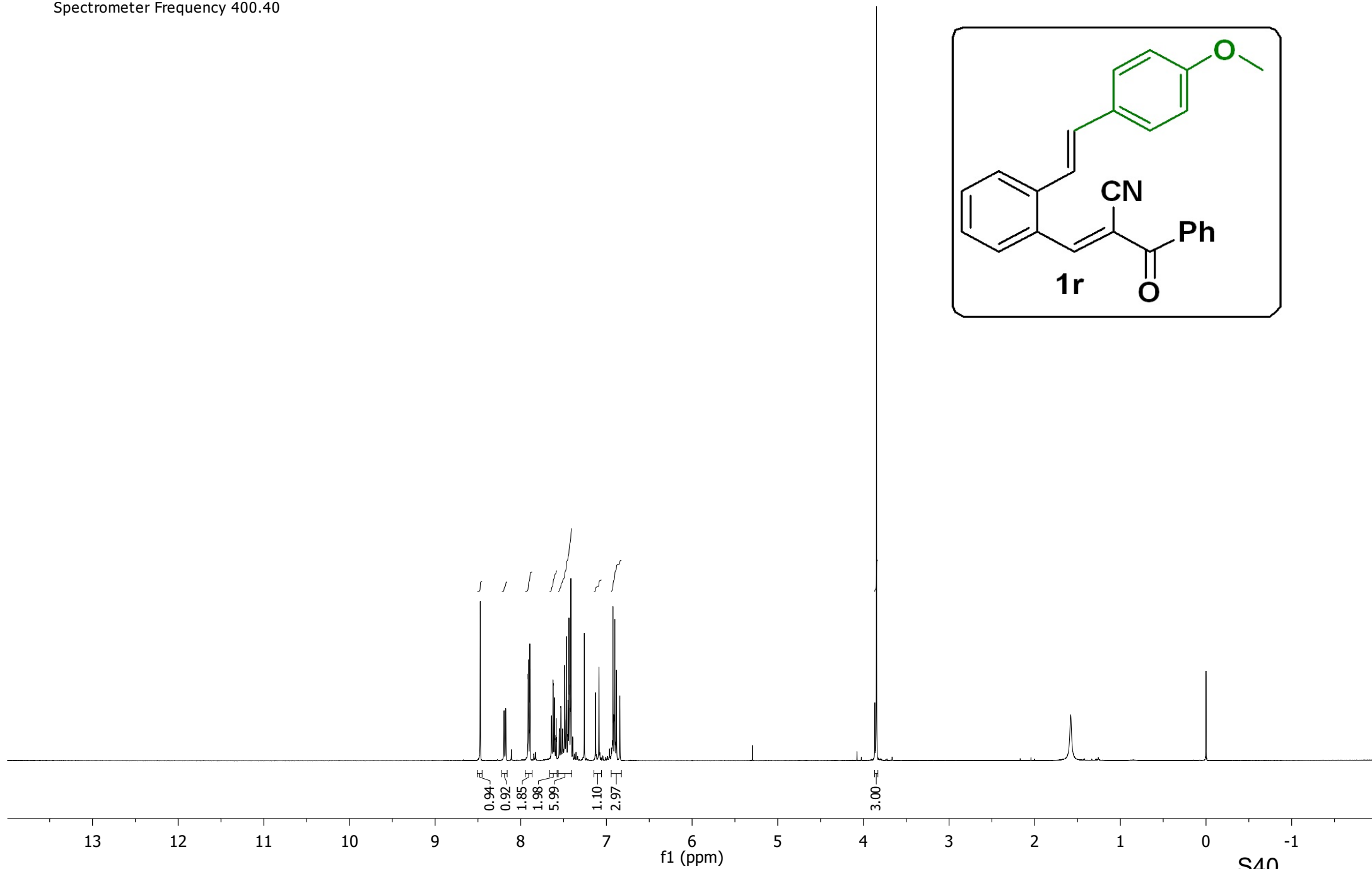
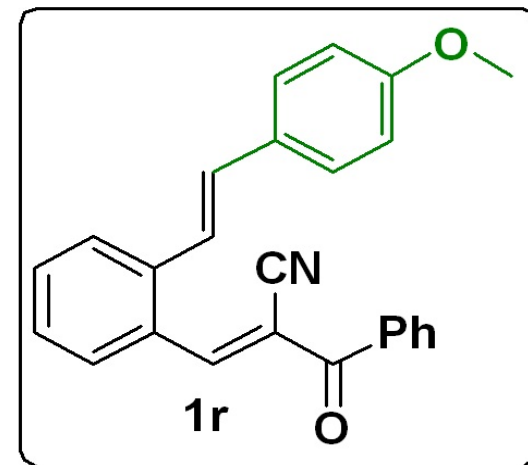


S39

M0027-16
LW-ds-4

Solvent CDCl_3
Spectrometer Frequency 400.40

8.47 8.20 8.18 7.91 7.91 7.90 7.89 7.89 7.64 7.62 7.62 7.61 7.60 7.59 7.59 7.58 7.55 7.55 7.53 7.51 7.51 7.49 7.47 7.47 7.45 7.45 7.44 7.43 7.42 7.42 7.39 7.39 7.26 7.26 7.13 7.13 7.09 7.09 6.93 6.93 6.92 6.92 6.91 6.91 6.88 6.88 6.84 6.84 3.87 3.85



1r

S40

M0027-17
LW-ds-4

—189.06

—160.05

—154.74

—140.05

—135.85

—134.99

—133.35

—132.55

—129.73

—129.31

—129.26

—128.97

—128.66

—128.22

—127.56

—127.46

—122.59

—116.53

—114.25

—112.05

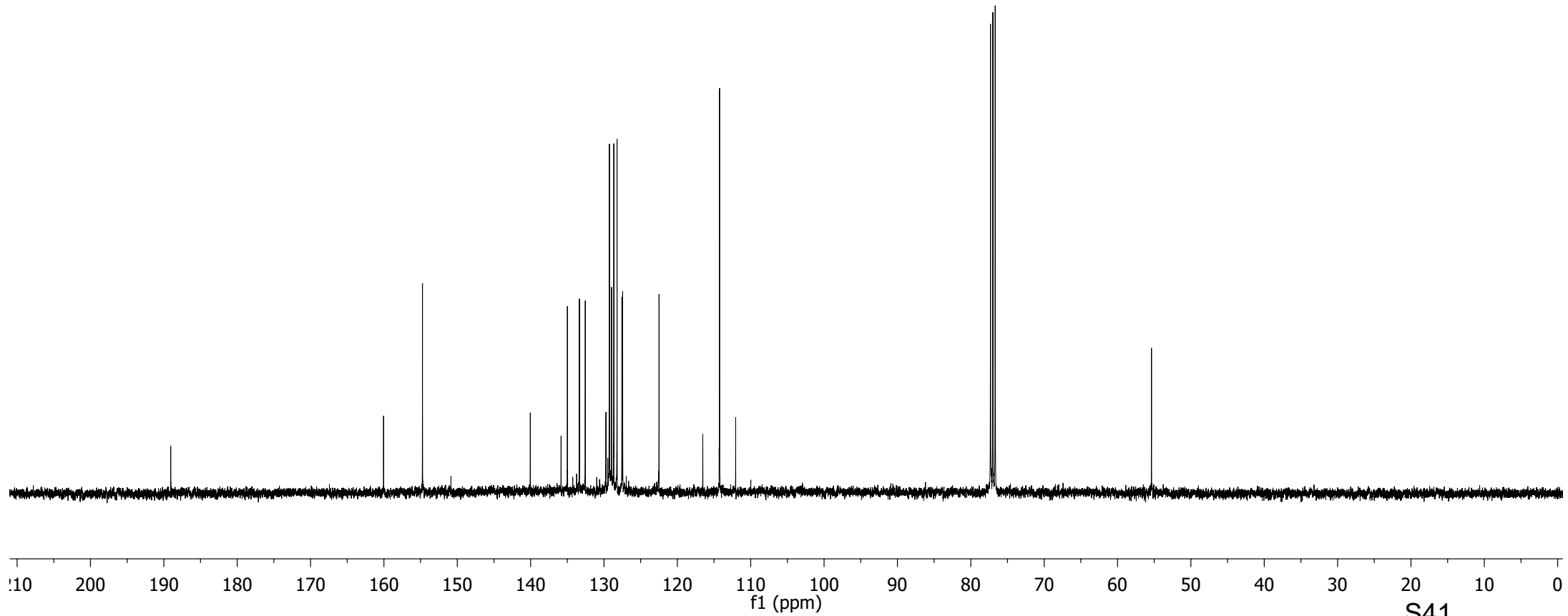
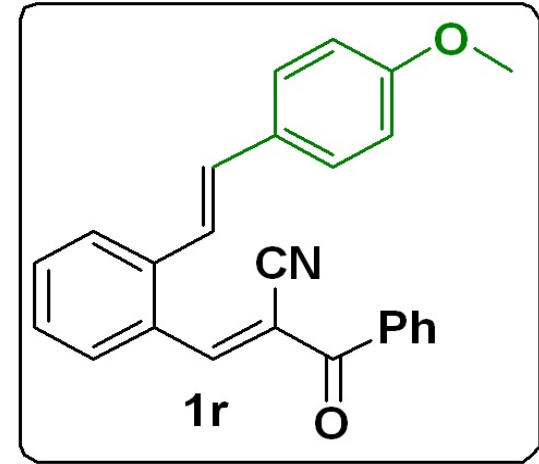
—77.31

—76.99

—76.68

—55.37

Solvent CDCl_3
Spectrometer Frequency 100.69



S41

M0024-7
LW-ds-3

8.46
8.20
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7.33
6.89
6.85

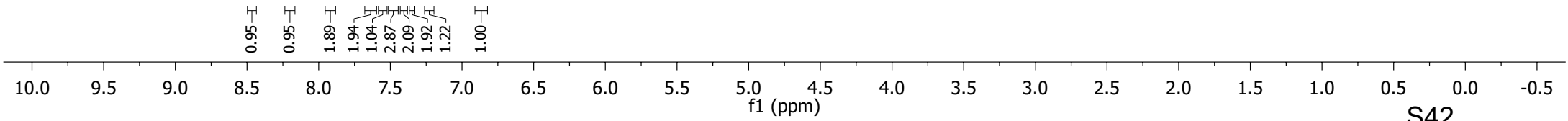
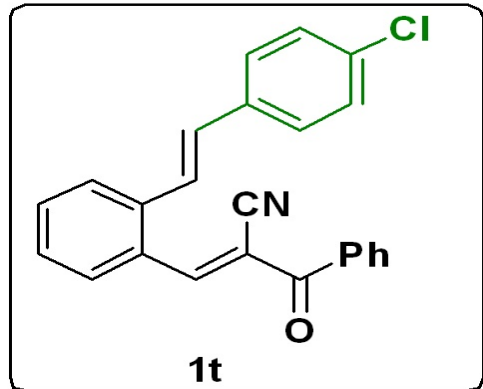
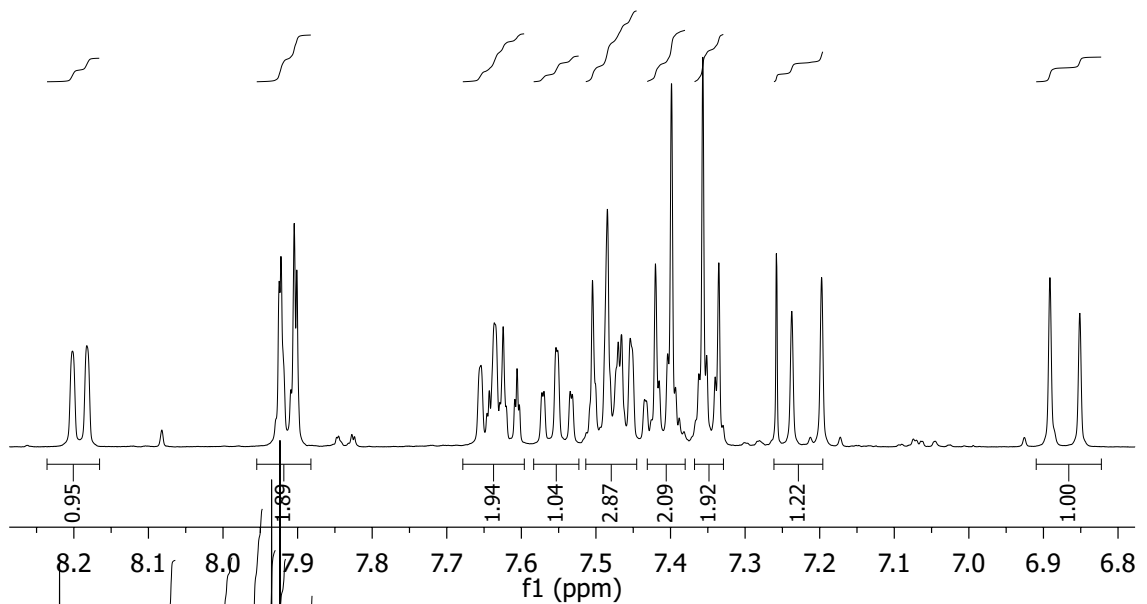
Solvent CDCl₃
Spectrometer Frequency 400.40

M0024-7
LW-ds-3

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7.45
7.42
7.40
7.40
7.36
7.35
7.34
7.26
7.24
7.20

6.89
6.85

1.58
-0.00



M0024-8
LW-ds-3

— 188.77

— 154.32

139.20

135.74

134.30

133.87

133.49

132.60

130.02

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128.01

127.61

126.44

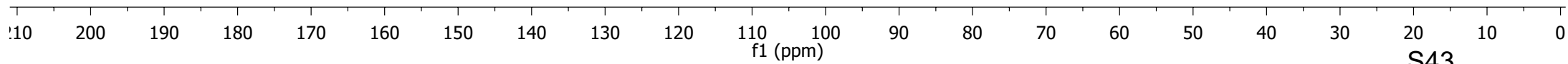
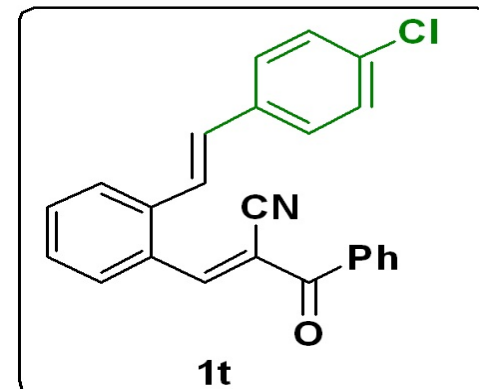
— 112.41

77.31

76.99

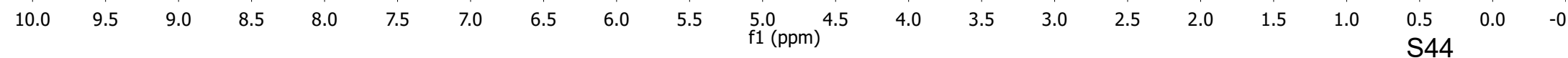
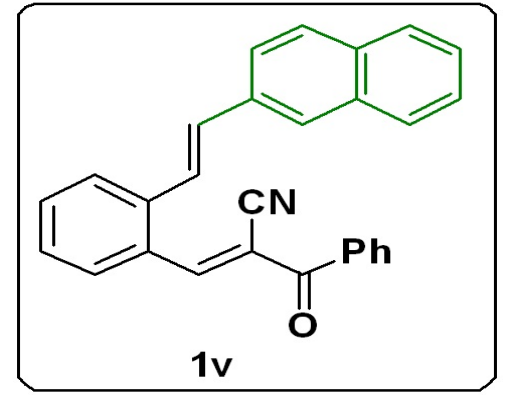
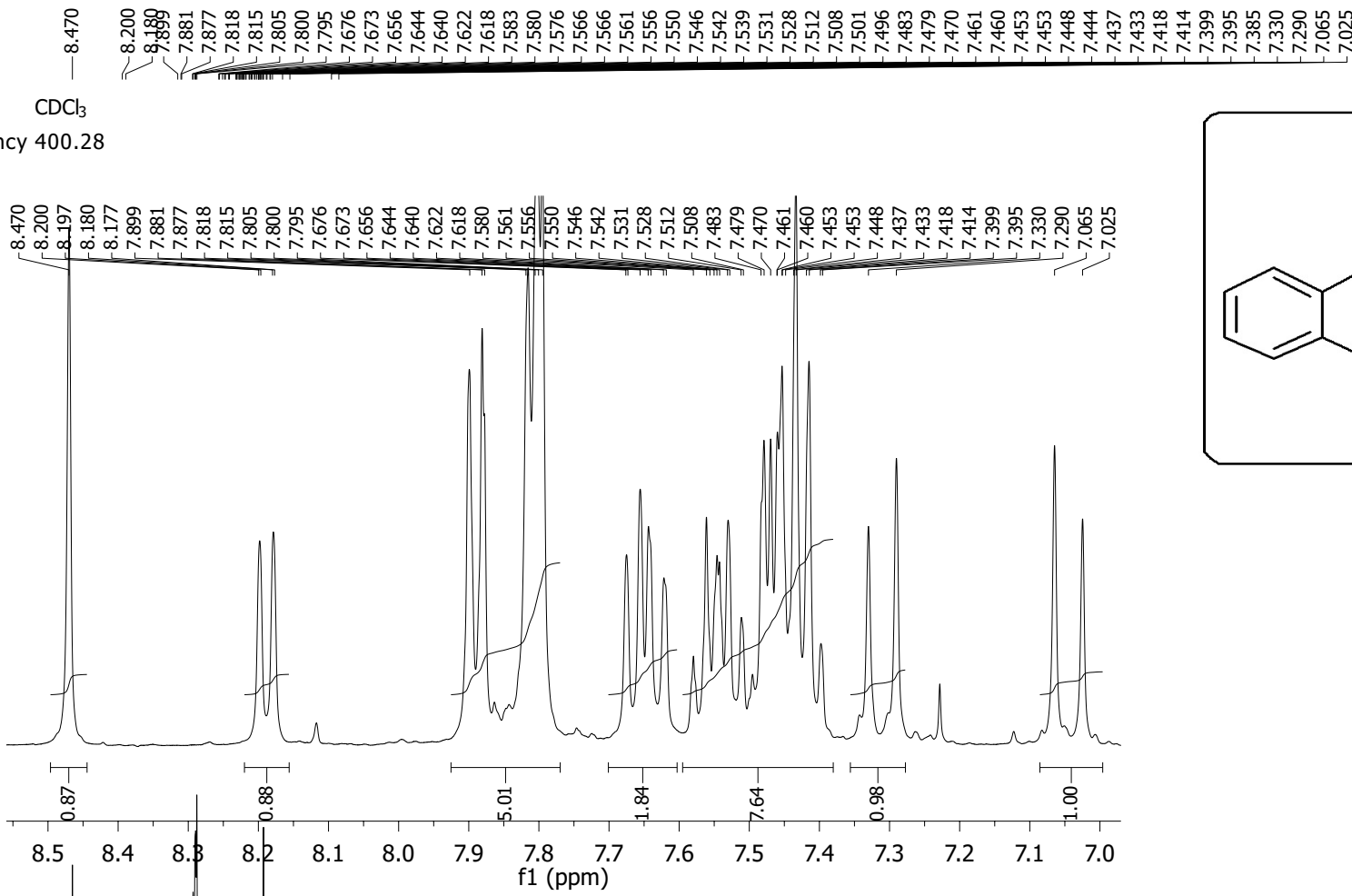
76.68

Solvent CDCl_3
Spectrometer Frequency 400.40

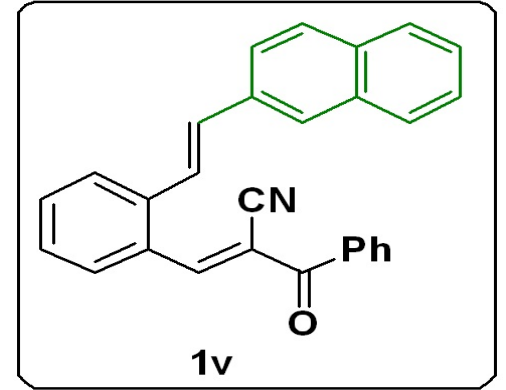
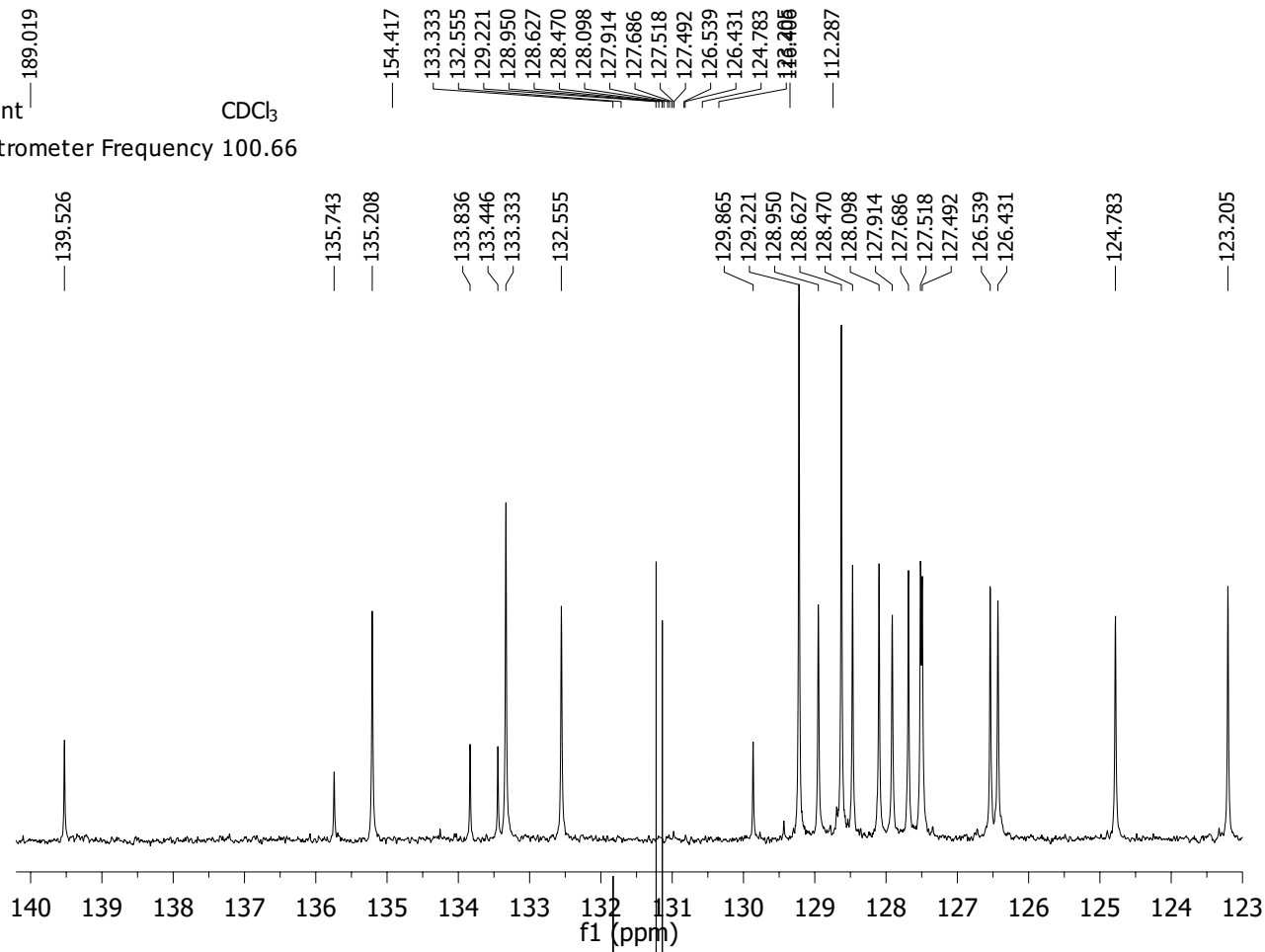


S43

Solvent CDCl₃
Spectrometer Frequency 400.28

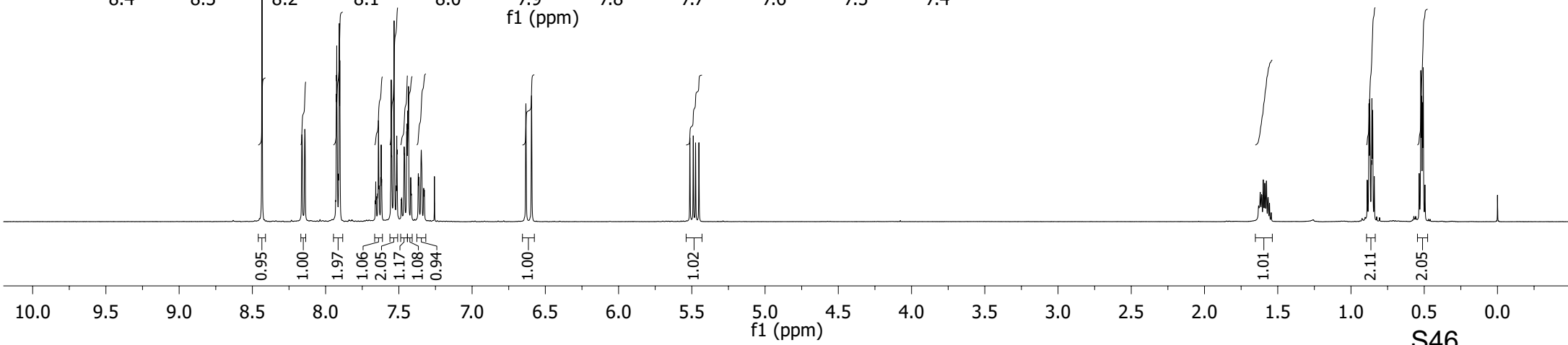
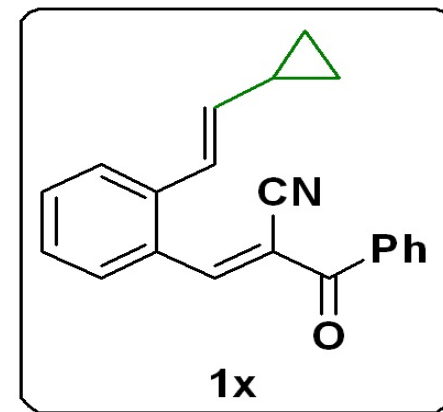
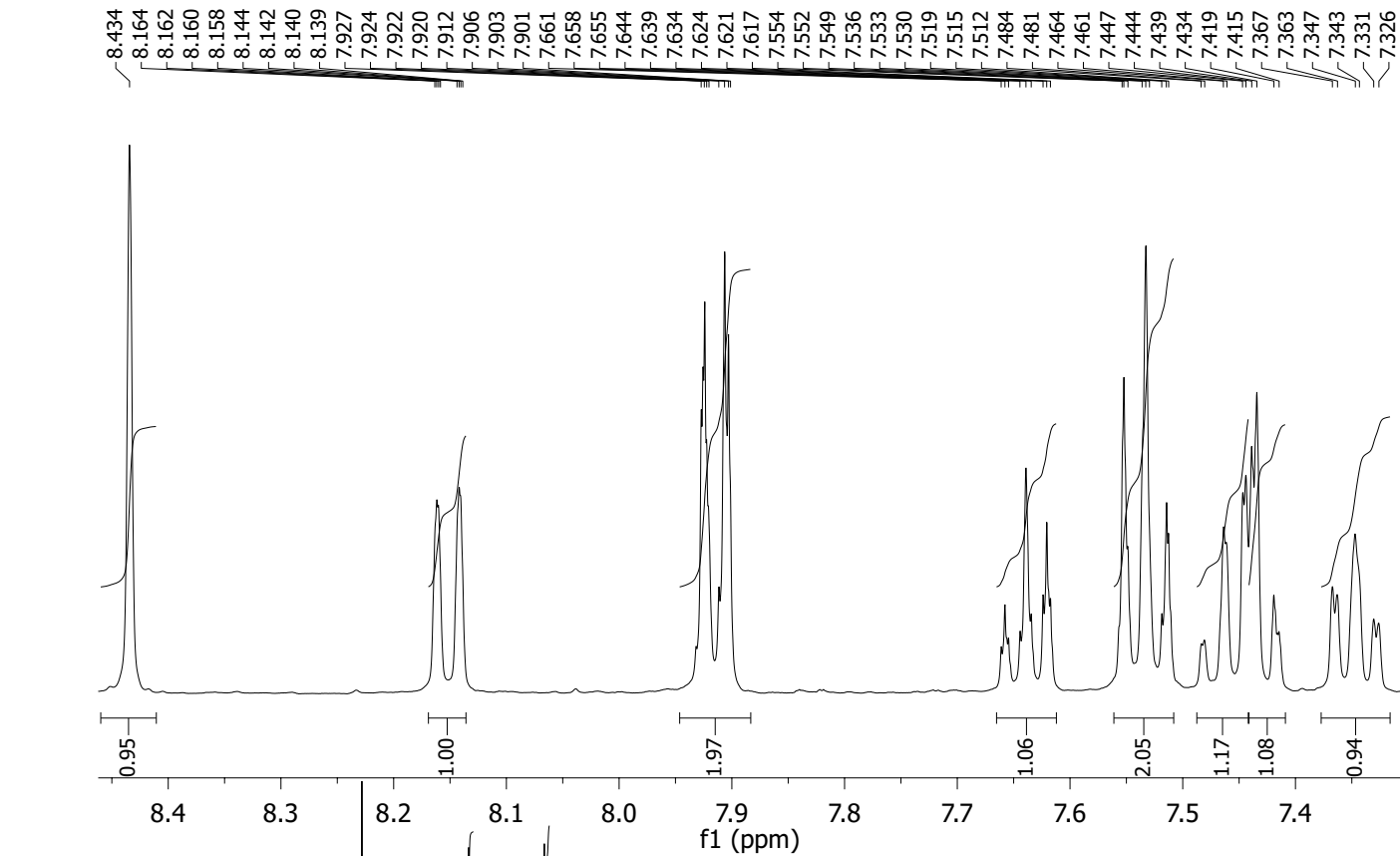


Solvent — 189.019
CDCl₃
Spectrometer Frequency 100.66

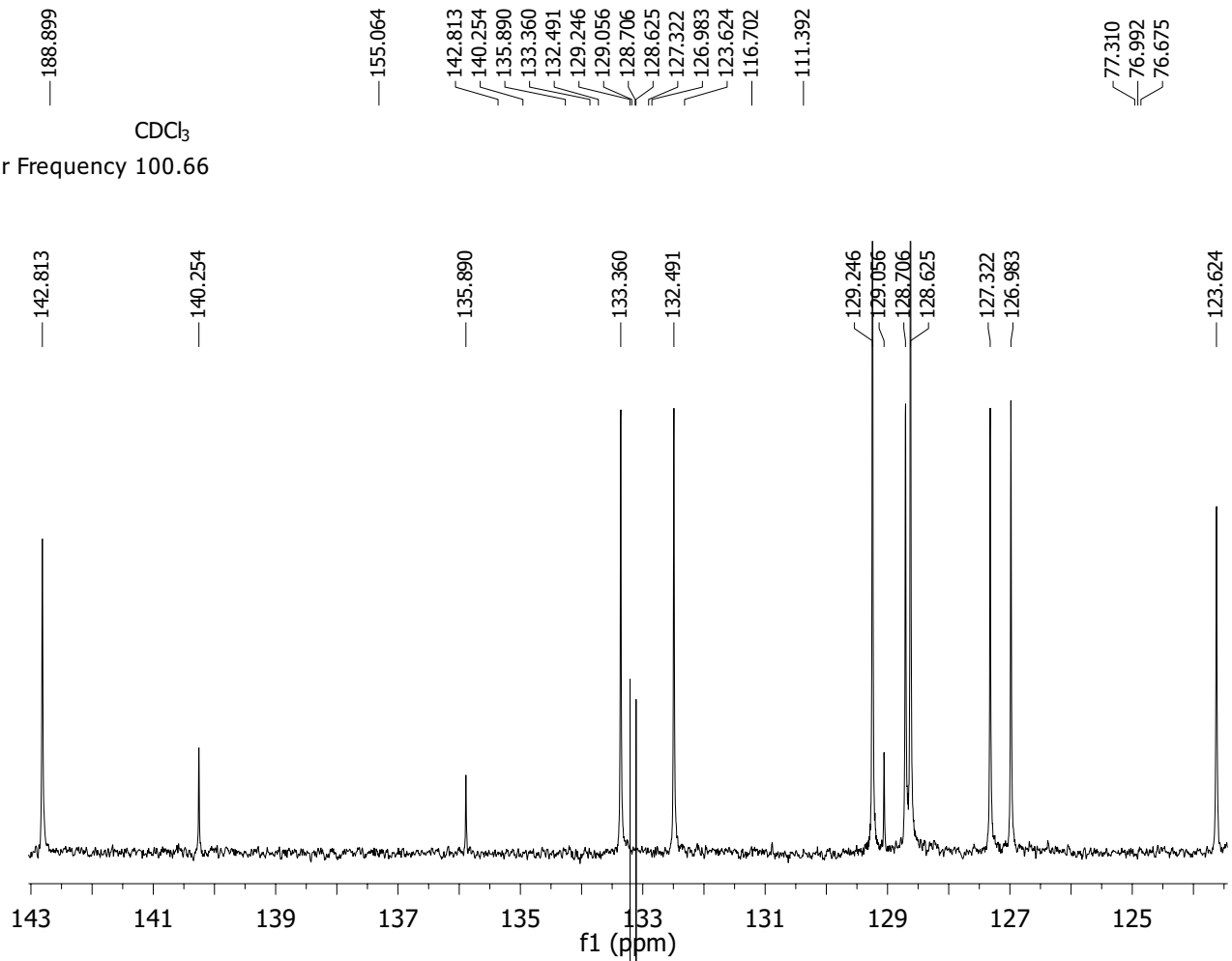


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Solvent CDCl_3
Spectrometer Frequency 400.28



Solvent CDCl_3
Spectrometer Frequency 100.66

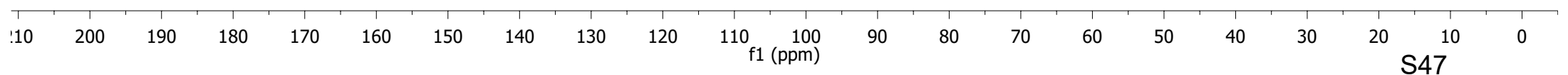
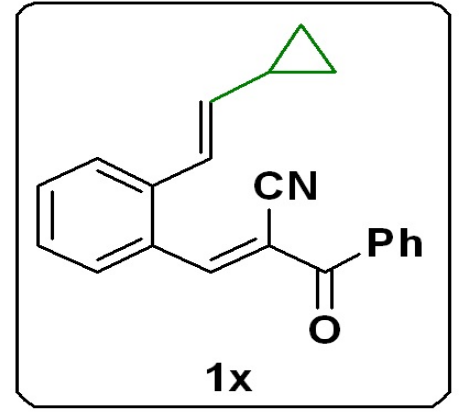


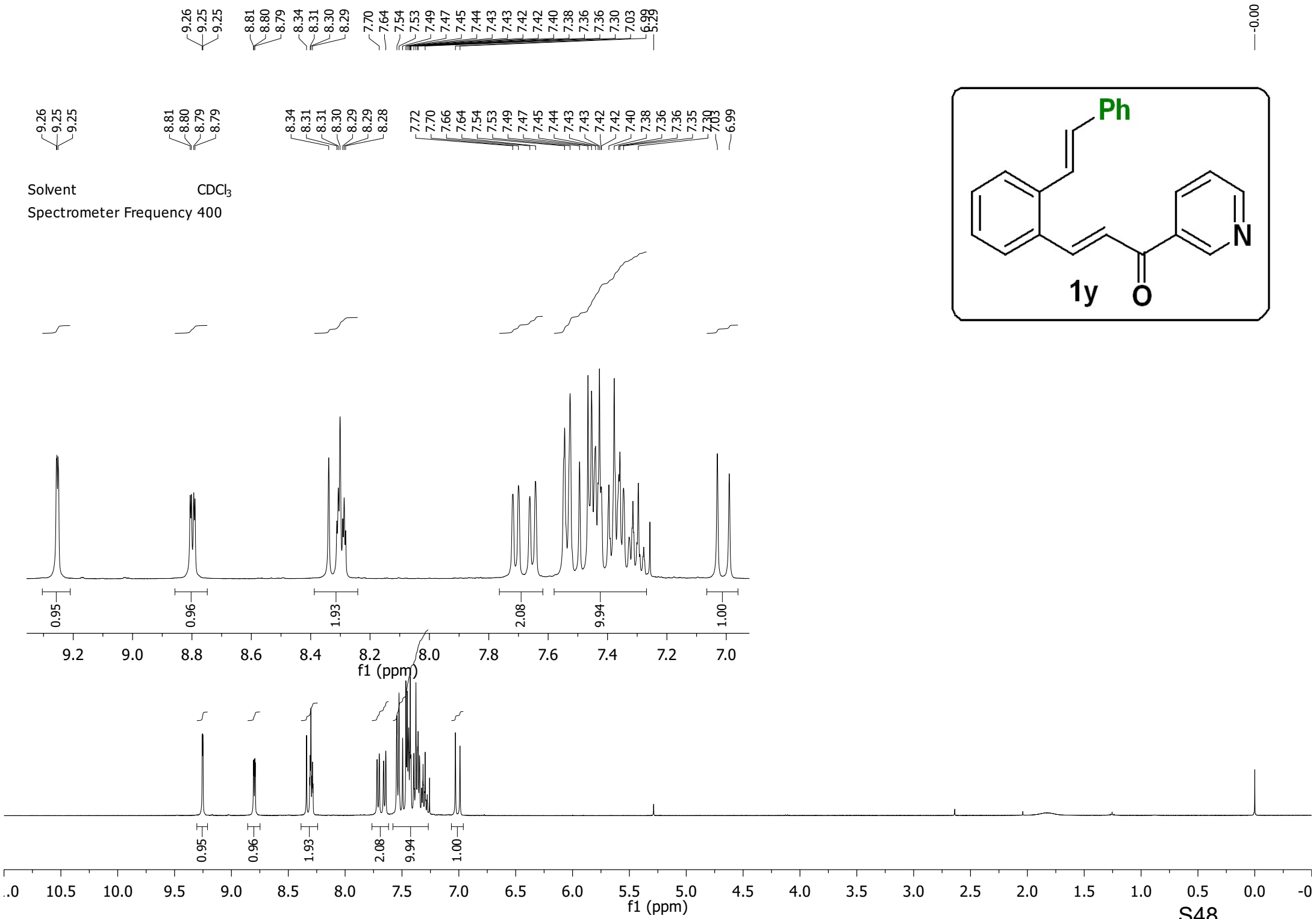
155.064
142.813
140.254
135.890
133.360
132.491
129.246
129.056
128.706
128.625
127.322
126.983
123.624
116.702
111.392

77.310
76.992
76.675

15.221

7.815

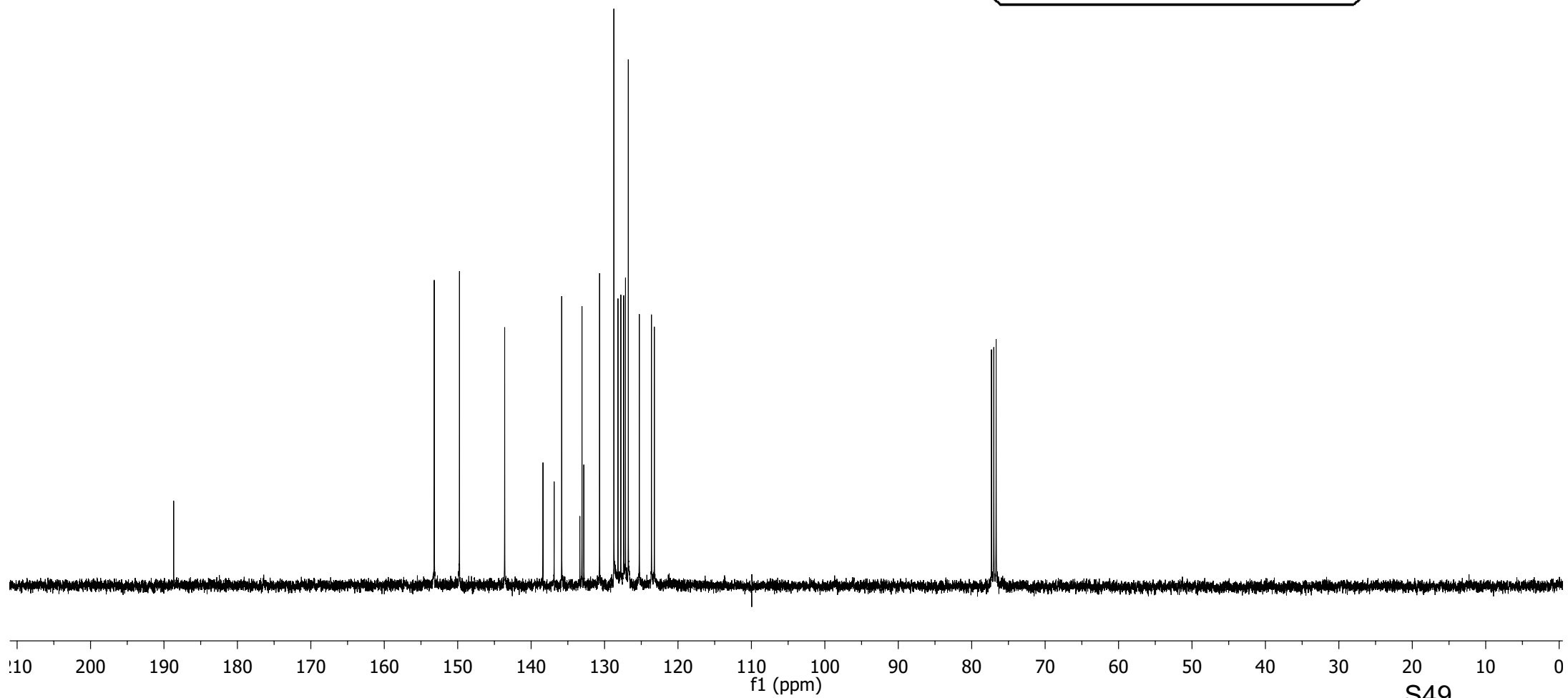
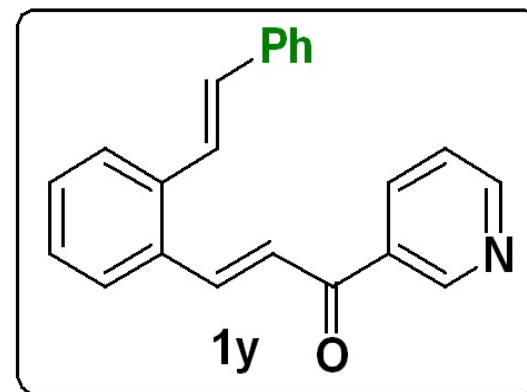




Solvent CDCl_3
Spectrometer Frequency 100

153.19
149.76
143.60
138.39
136.86
135.84
133.36
133.07
132.83
130.68
128.73
128.17
127.78
127.39
127.15
126.77
125.27
123.61
123.22

77.31
76.99
76.67

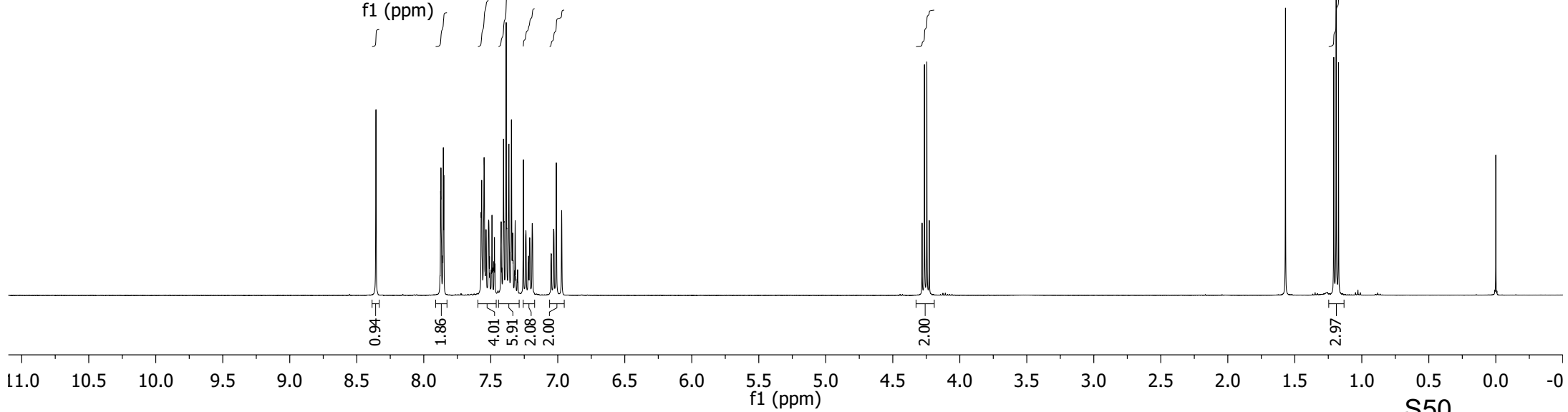
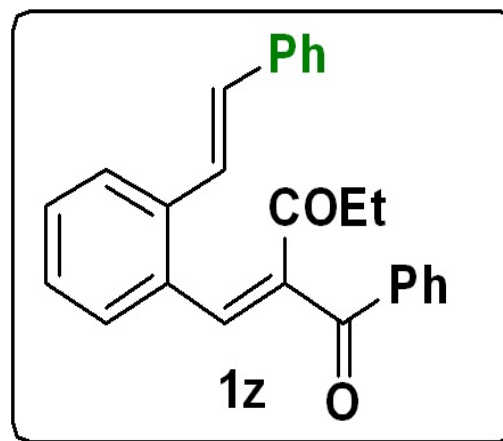
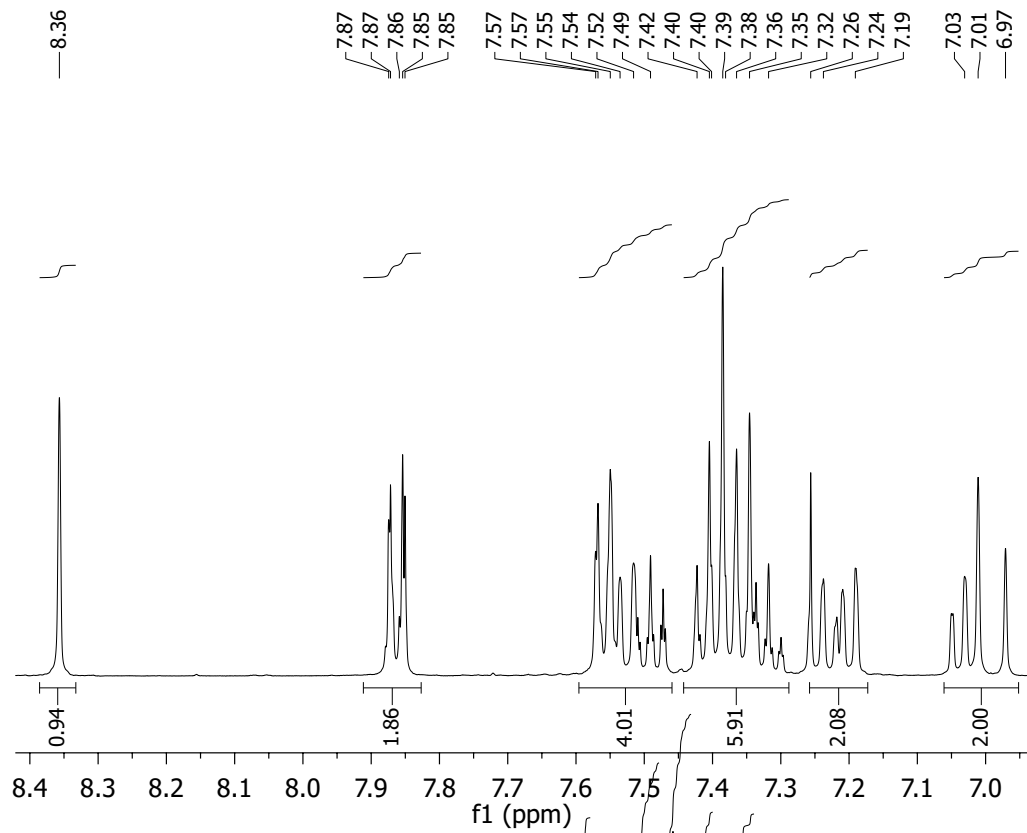


Solvent CDCl_3
Spectrometer Frequency 400

8.36
7.87
7.87
7.85
7.85
7.57
7.57
7.55
7.54
7.52
7.49
7.42
7.40
7.40
7.39
7.38
7.36
7.35
7.34
7.32
7.26
7.24
7.21
7.19
7.03
7.01
6.97
6.96
4.26
4.25
4.23

1.57
1.21
1.19
1.17

-0.00



Solvent CDCl_3
Spectrometer Frequency 100

—194.76

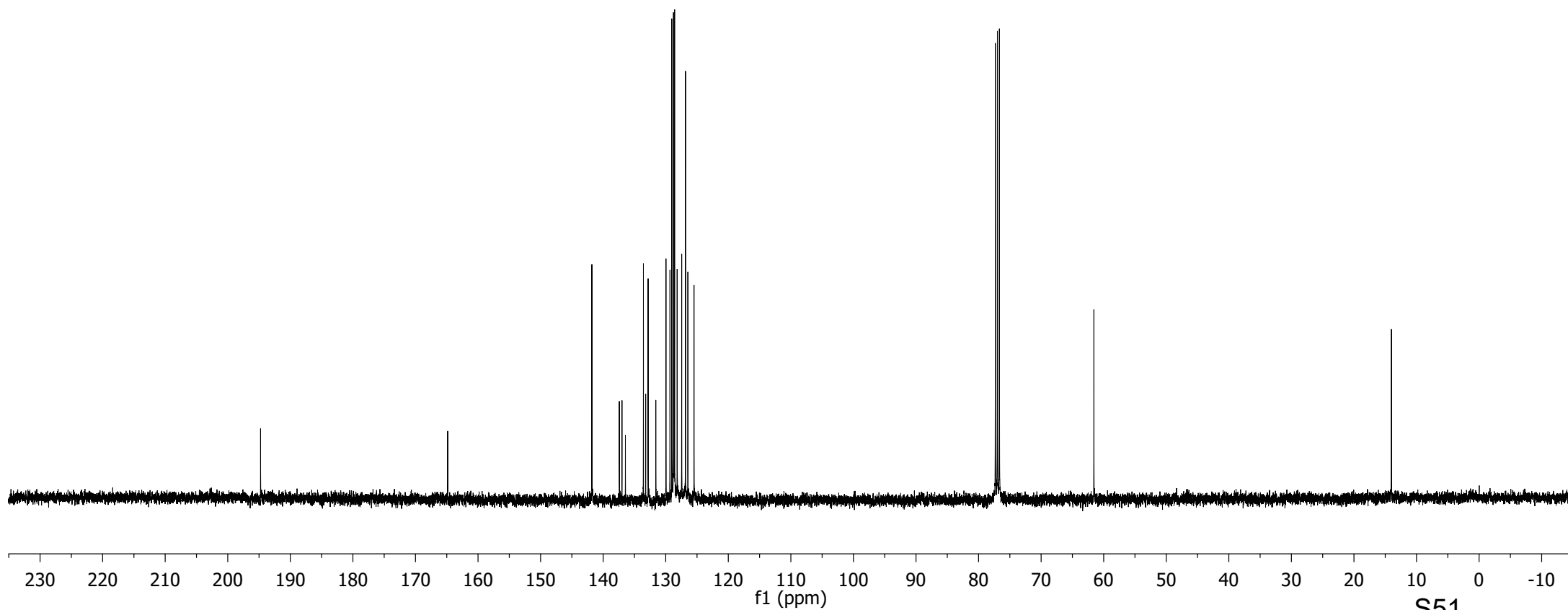
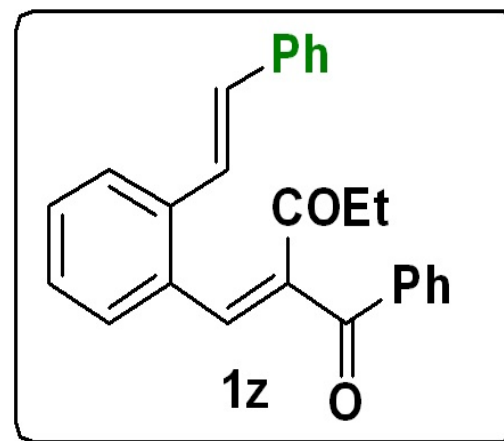
—164.84

141.80
137.42
136.99
136.45
133.57
133.20
132.82
131.58
129.96
129.32
129.04
128.76
128.58
128.18
127.45
126.84
126.46
125.48

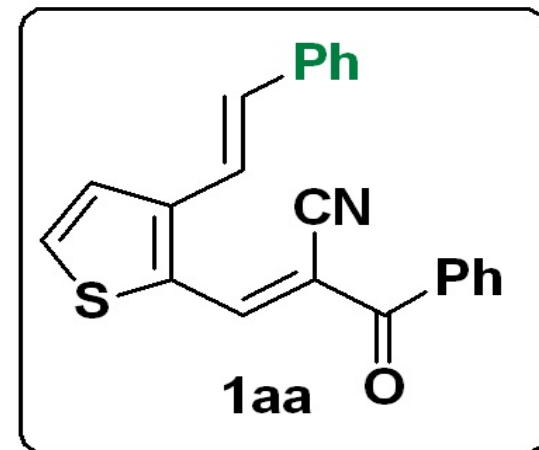
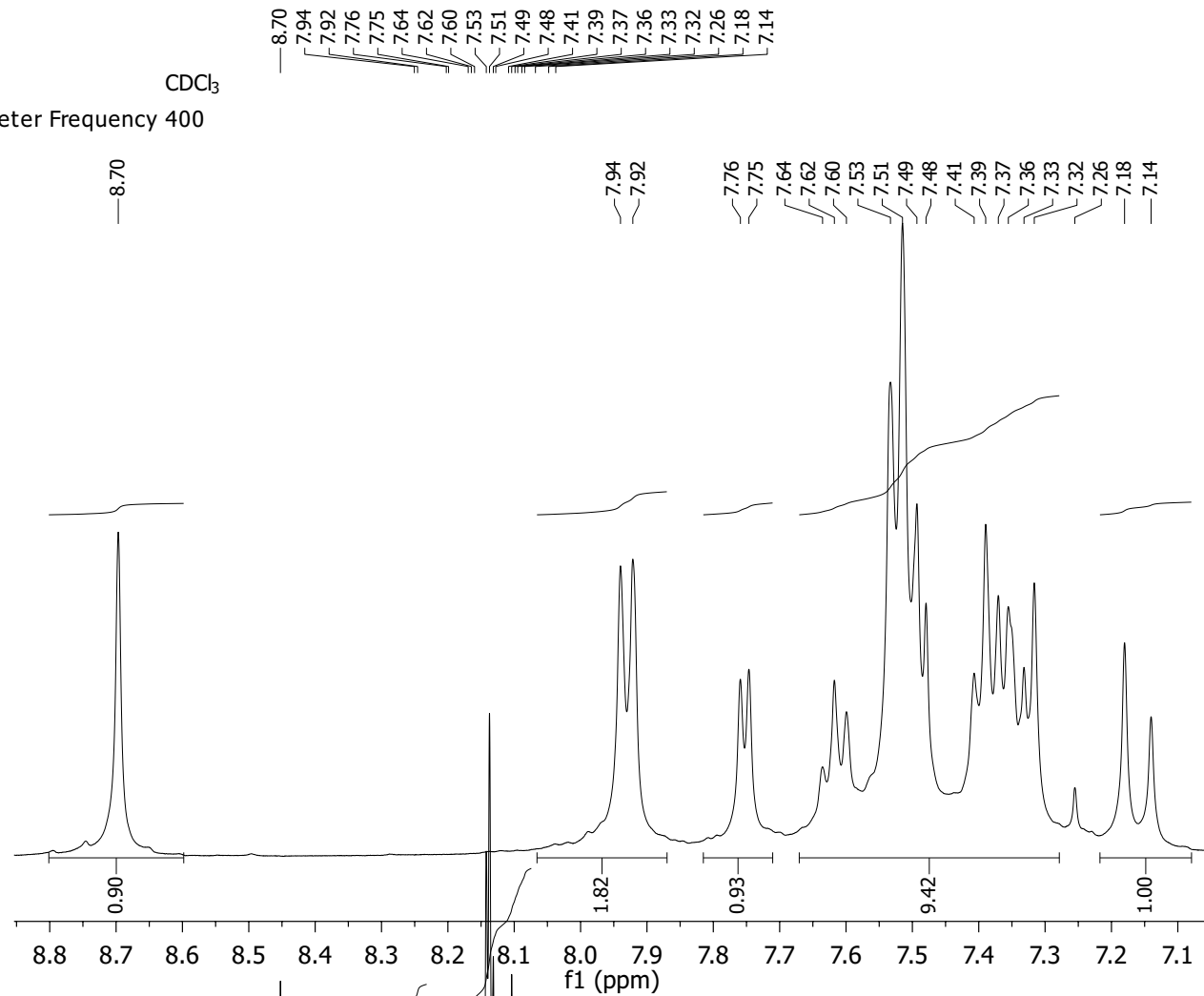
77.31
76.99
76.68

—61.57

—14.01



Solvent CDCl_3
Spectrometer Frequency 400



11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

f1 (ppm)

Solvent CDCl_3
Spectrometer Frequency 100

— 149.37

— 188.32

— 144.99

— 136.38

— 136.03

— 134.96

— 134.48

— 133.09

— 131.54

— 129.03

— 129.00

— 128.88

— 128.50

— 127.02

— 126.27

— 104.58

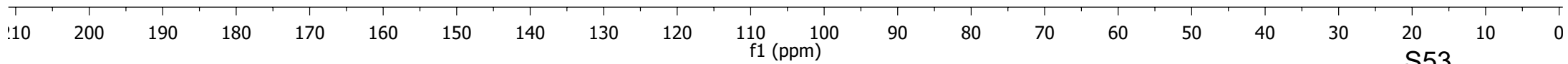
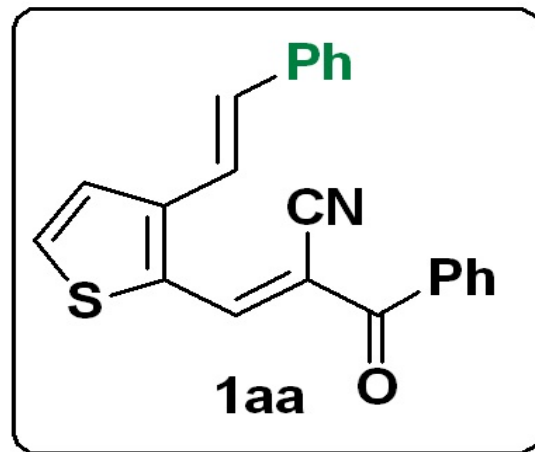
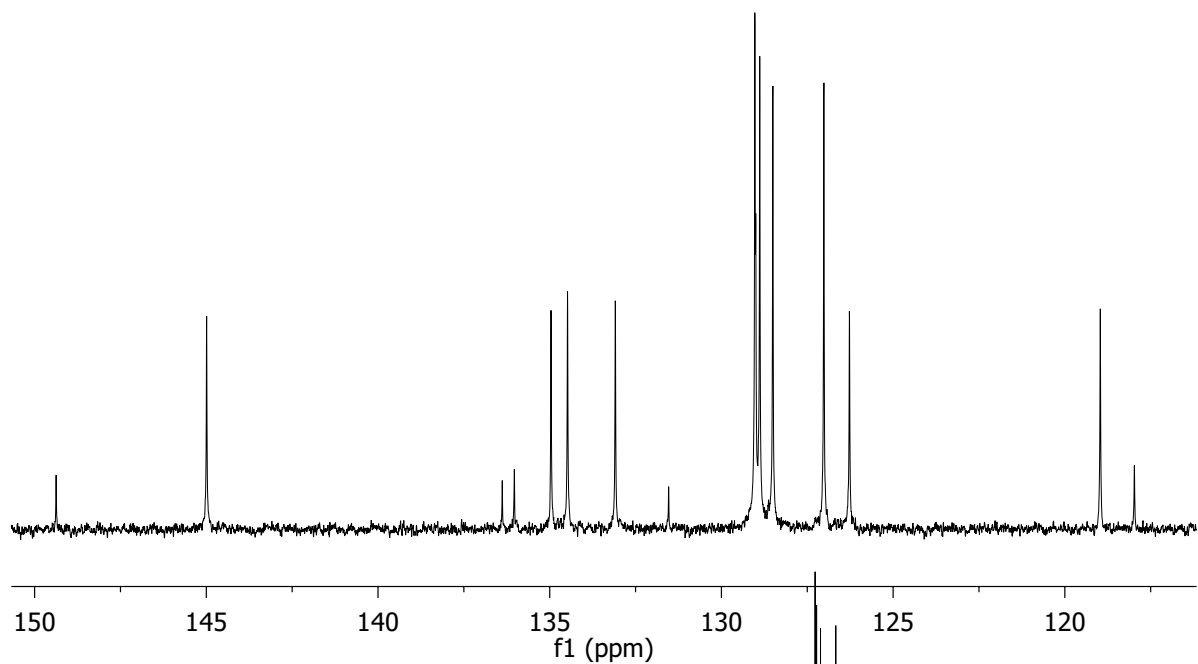
— 118.97

— 117.97

— 77.31

— 76.99

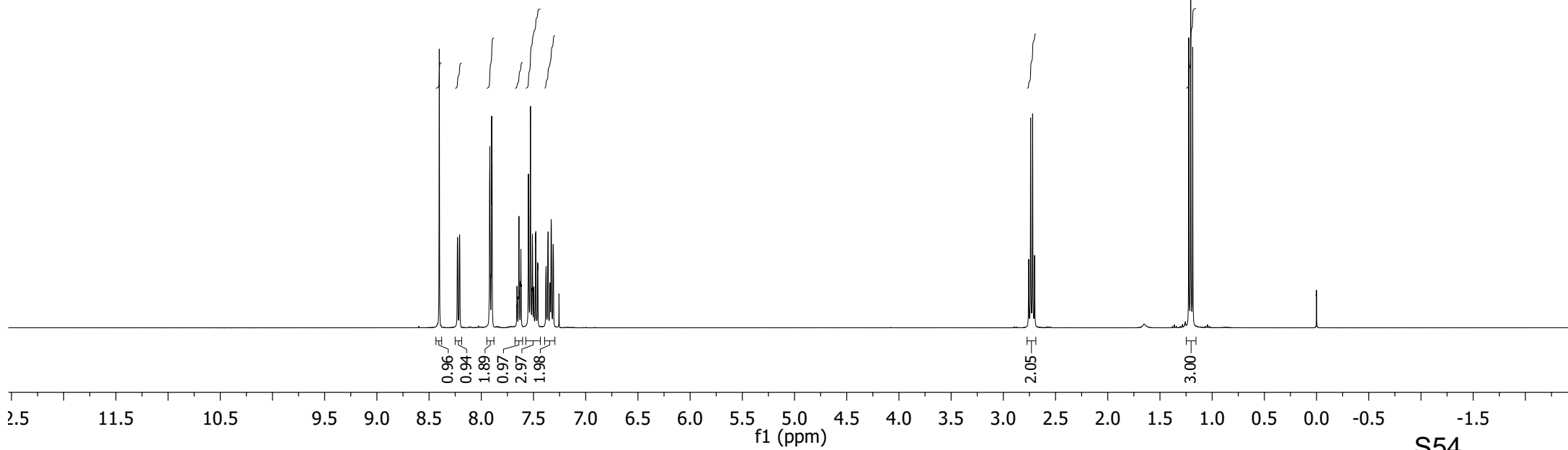
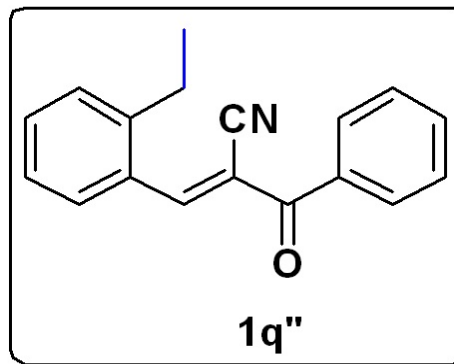
— 76.68



U0009
TPG-463

2.90
2.88
2.76
2.74
2.72
2.70
2.58
2.56
1.65
1.36
1.28
1.26
1.22
1.21
1.19
1.16
1.06
1.04
0.00

Solvent CDCl_3
Spectrometer Frequency 400.28



U0362-4
TPG-463

—188.90

Solvent

CDCl₃

Spectrometer Frequency 100.66

—153.91

—145.79

—135.74

—133.41

—132.92

—130.09

—129.40

—129.22

—128.68

—128.61

—126.67

—116.63

—111.79

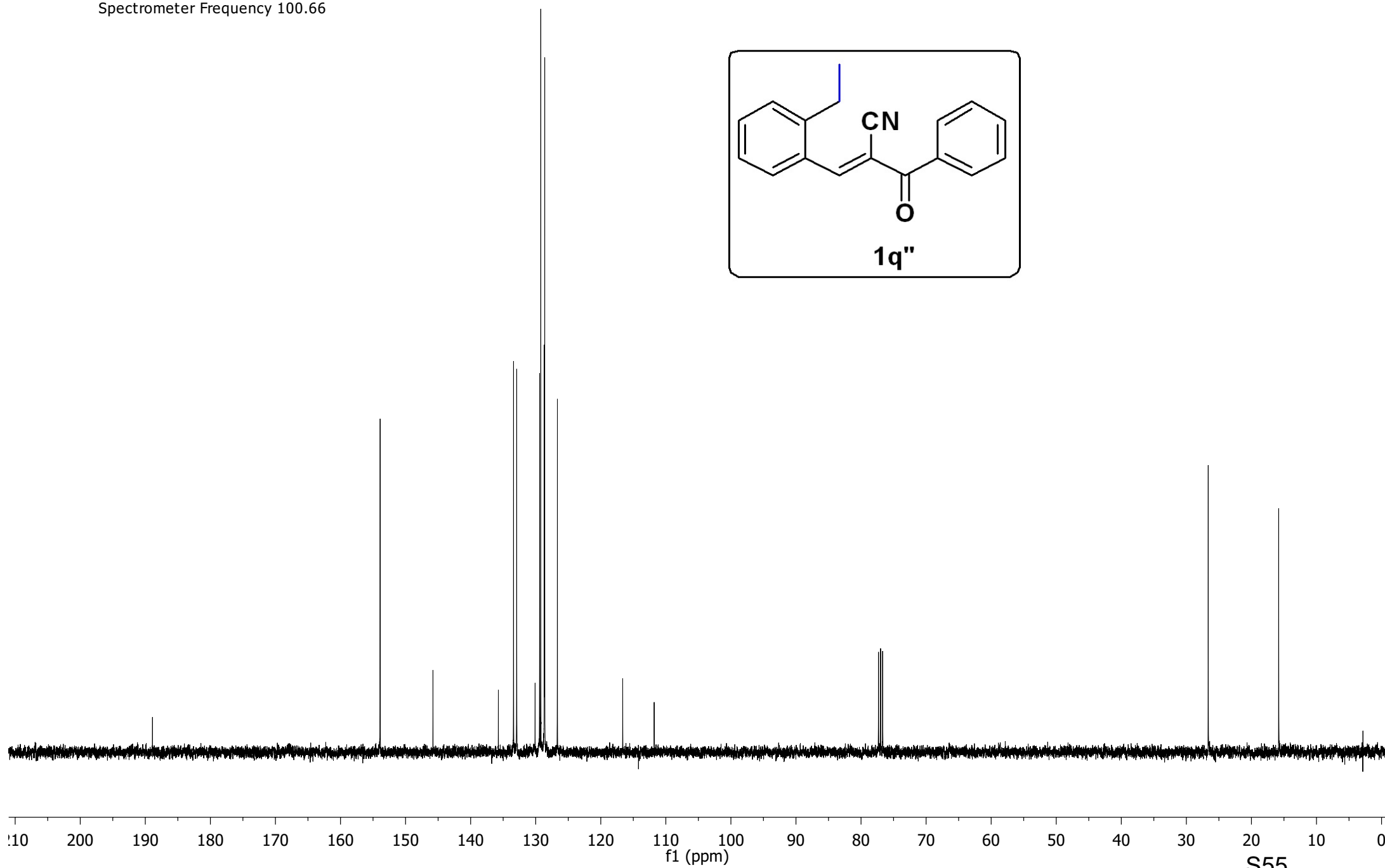
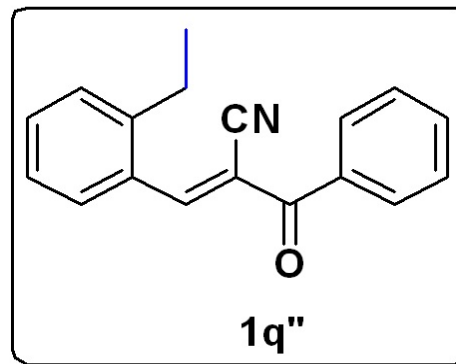
—77.31

—77.00

—76.68

—26.64

—15.81



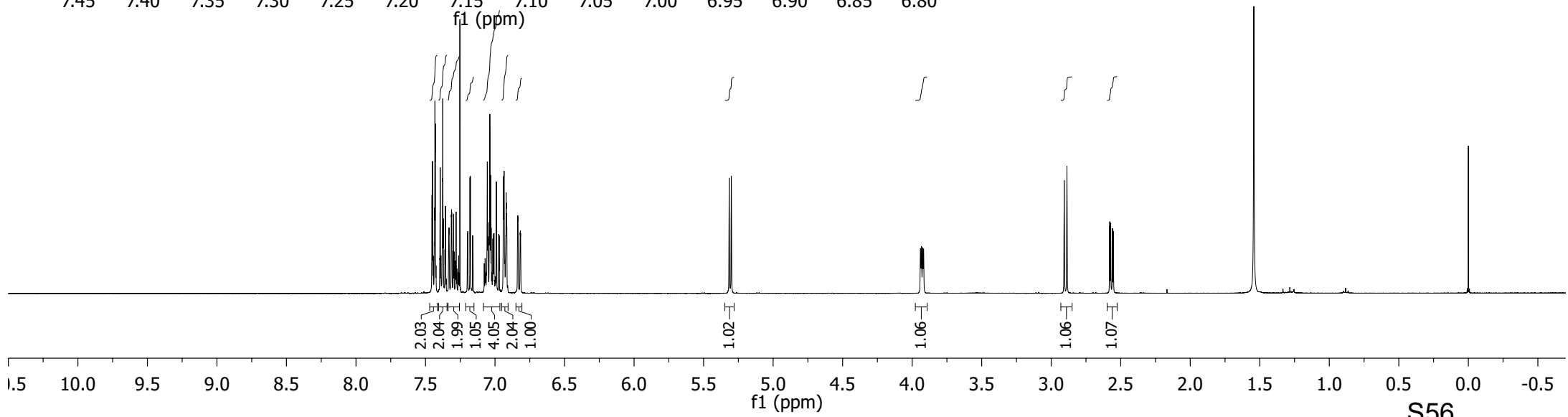
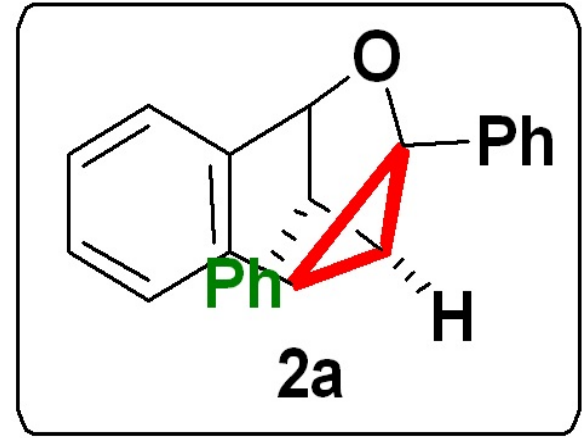
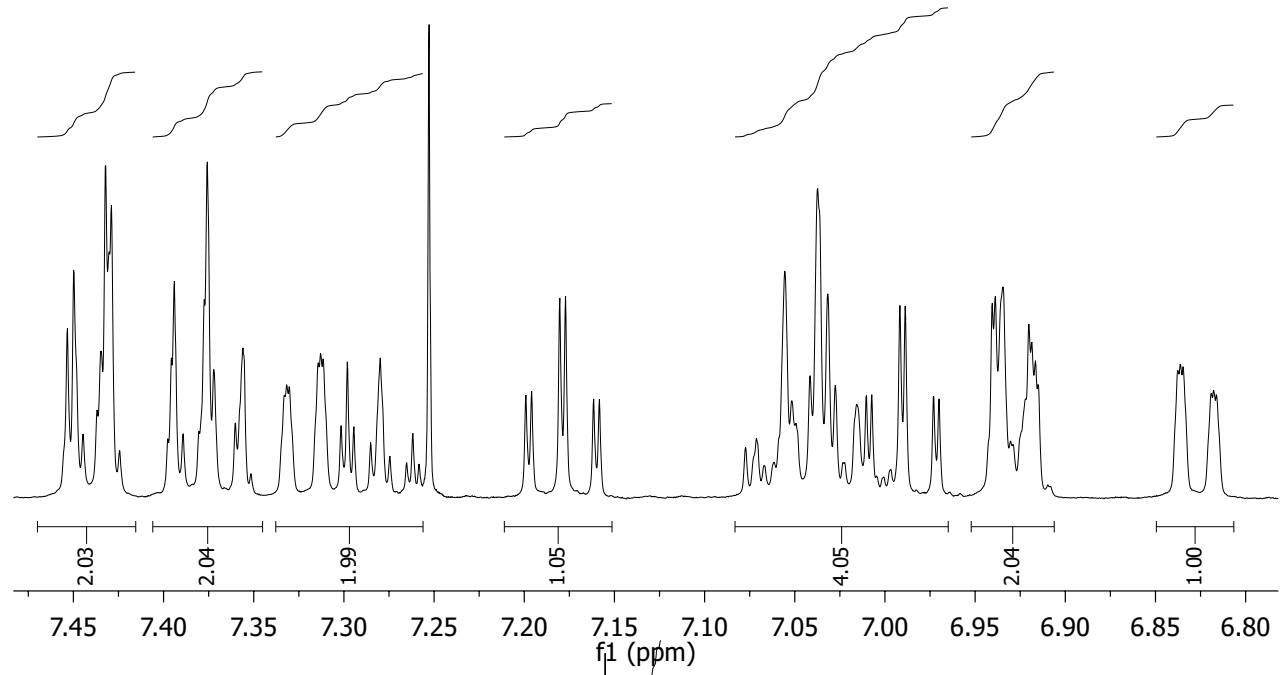
M025719
 TPG-363
 7.457, 7.451, 7.443, 7.43, 7.40, 7.39, 7.38, 7.37, 7.36, 7.33, 7.33, 7.31, 7.31, 7.31, 7.30, 7.28, 7.25, 7.18, 7.18, 7.06, 7.04, 7.04, 7.03, 7.03, 6.99, 6.99, 6.94, 6.94, 6.93, 6.92, 6.92, 6.92, 6.92, 6.84, 6.84, 6.83, 6.83, 5.30, 3.94, 3.93, 3.92, 3.92

2.91, 2.89, 2.58, 2.58, 2.57, 2.57, 2.56, 2.56, 2.55, 2.55, 1.54

-0.00

Solvent CDCl₃
 Spectrometer Frequency 400.40

M025719
 TPG-363
 7.457, 7.451, 7.443, 7.43, 7.40, 7.39, 7.38, 7.37, 7.36, 7.33, 7.33, 7.31, 7.31, 7.31, 7.30, 7.28, 7.25, 7.18, 7.18, 7.06, 7.04, 7.04, 7.03, 7.03, 6.99, 6.99, 6.94, 6.94, 6.93, 6.92, 6.92, 6.92, 6.92, 6.84, 6.84, 6.83, 6.83, 6.82, 6.82, 6.82, 6.82



M0257-14
TPG-363 Solvent CDCl_3
Spectrometer Frequency 100.69

137.91
136.50
133.02
131.97
128.36
127.88
127.80
127.68
126.93
126.37
125.88
125.04
124.93
124.24
110.46

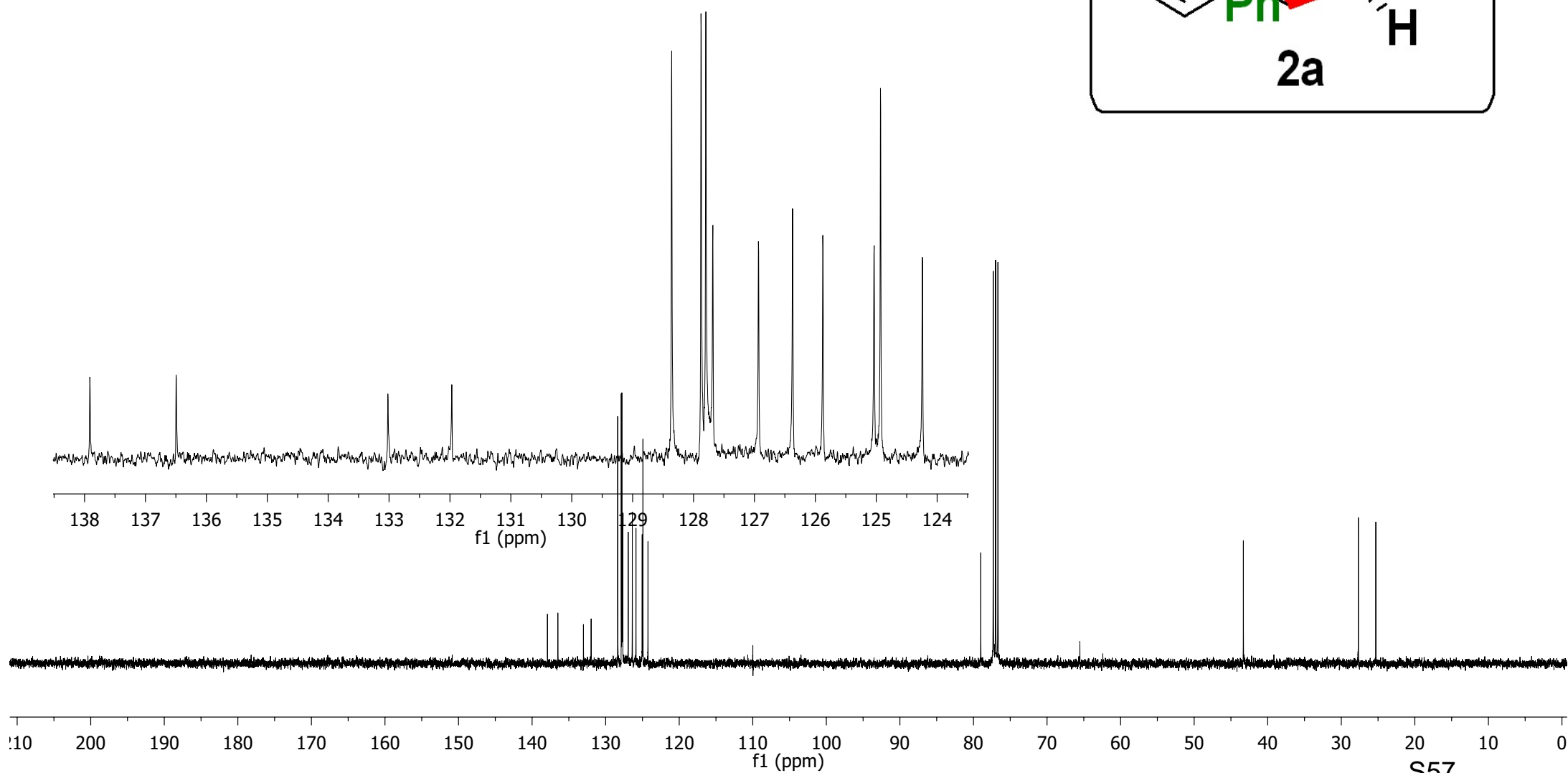
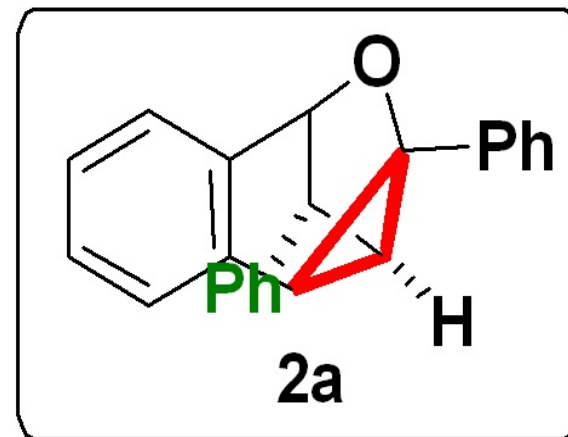
79.01
77.31
76.99
76.67

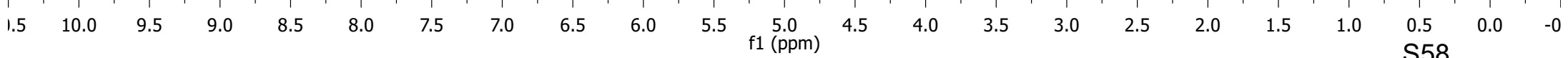
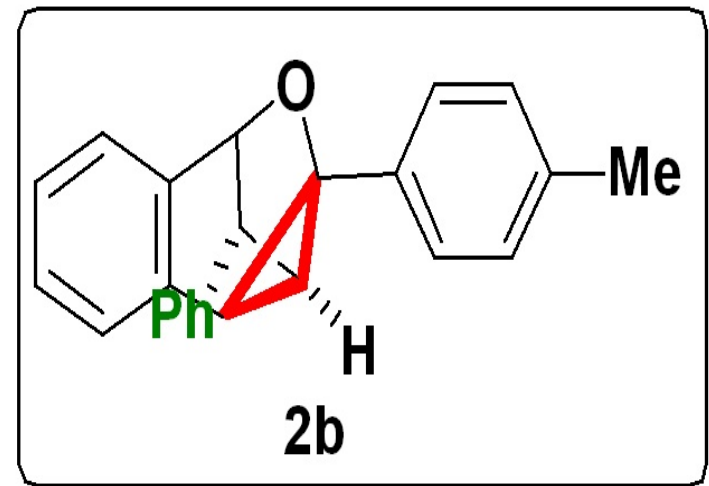
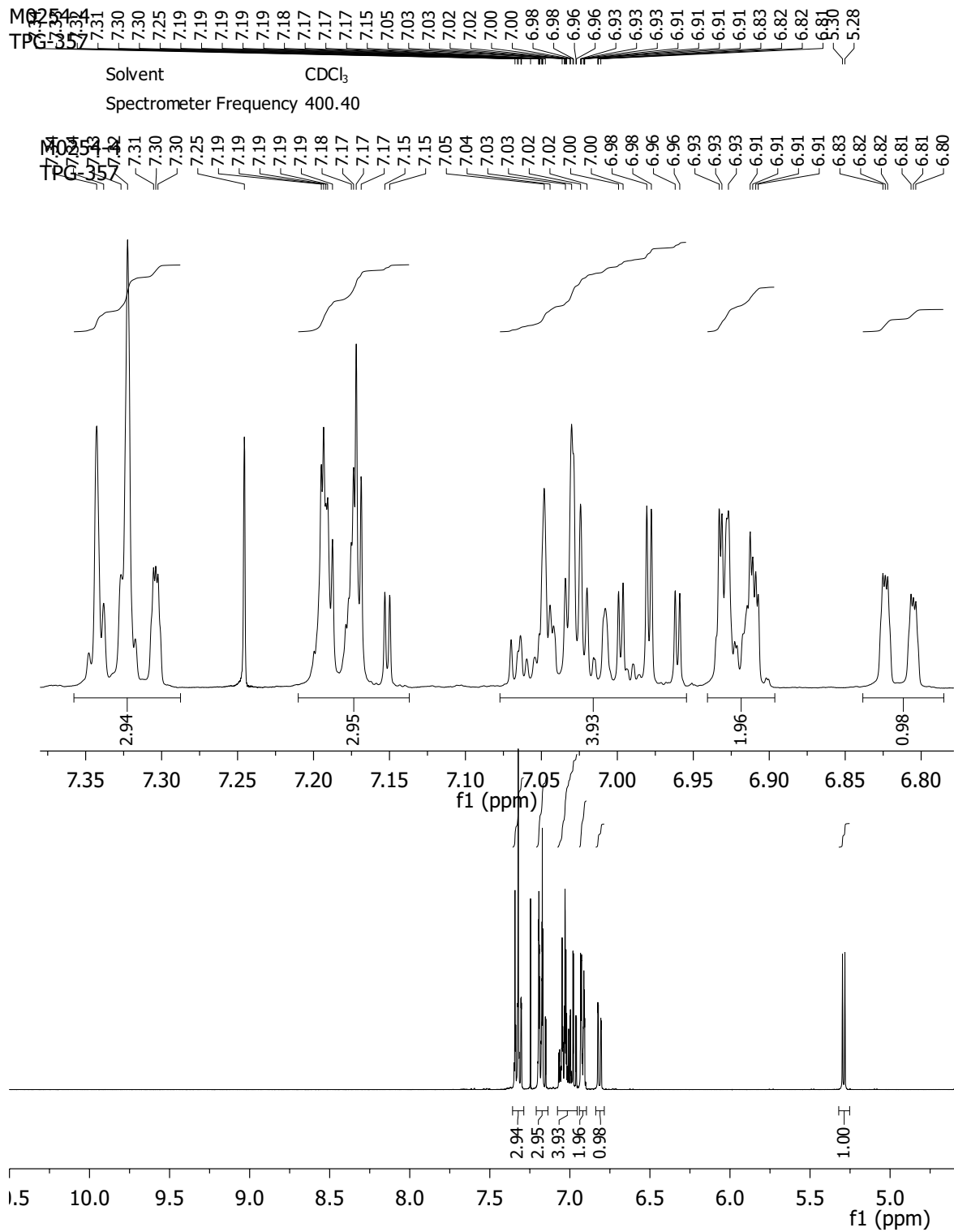
65.53

43.31

27.67
25.30

M0257-14
TPG-363 136.50
133.02
131.97
128.36
127.88
127.80
127.68
126.93
126.37
125.88
125.04
124.93
124.24





M0254-5
TPG-357

Solvent CDCl_3
Spectrometer Frequency 400.40

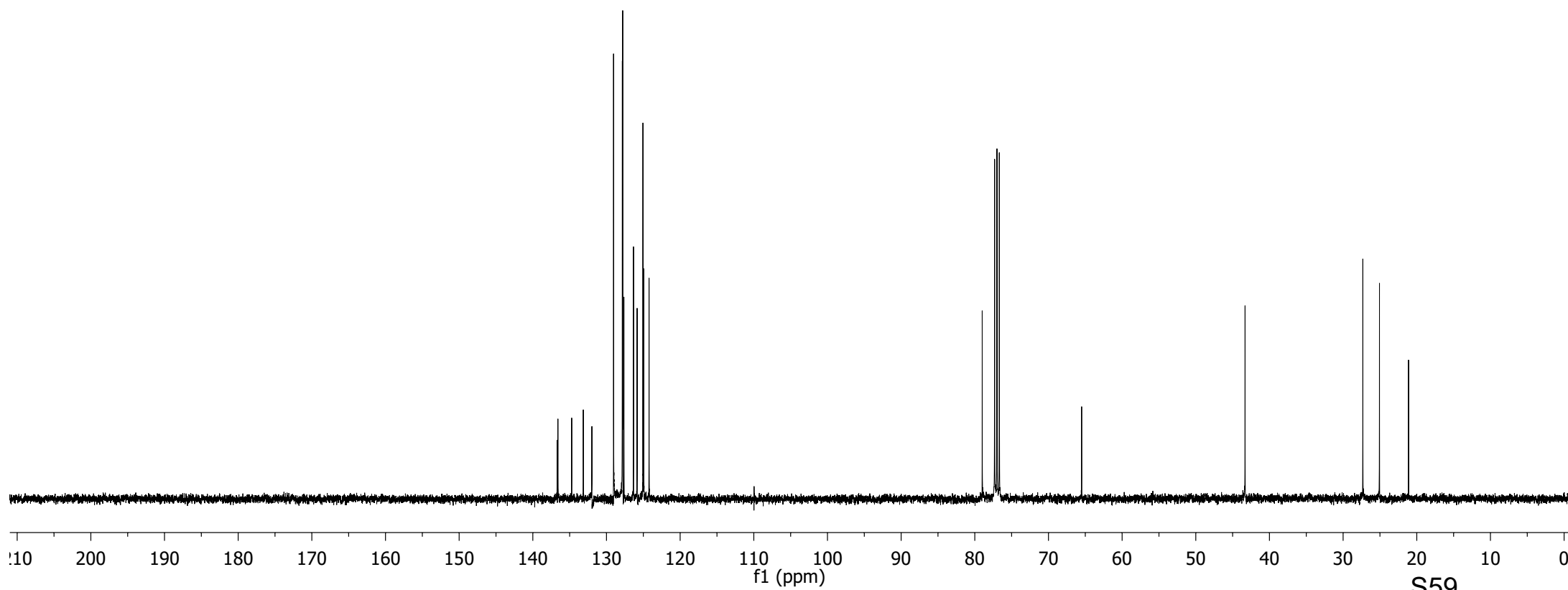
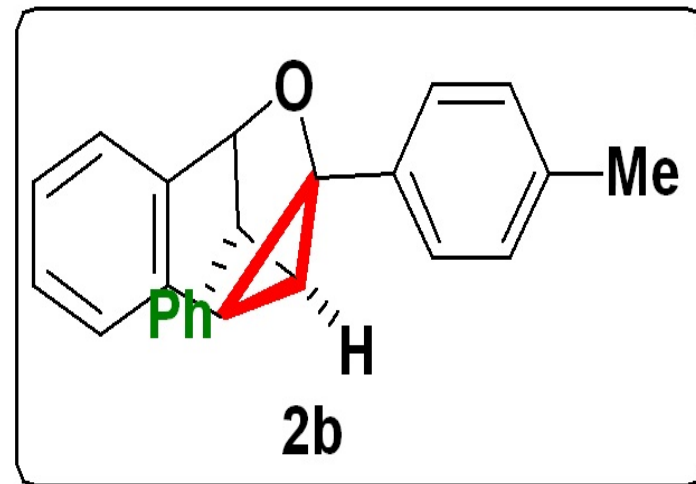
136.69
136.59
134.72
133.15
131.98
129.04
127.85
127.80
127.63
126.33
125.84
125.05
124.94
124.22

78.99
77.31
76.99
76.67

65.49

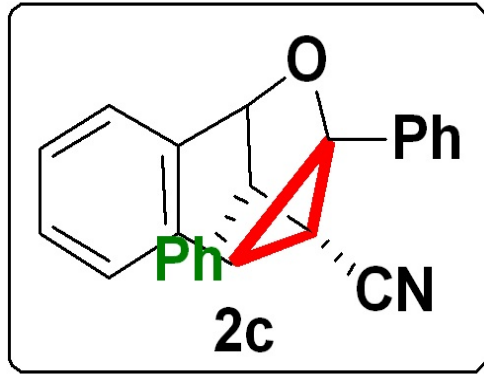
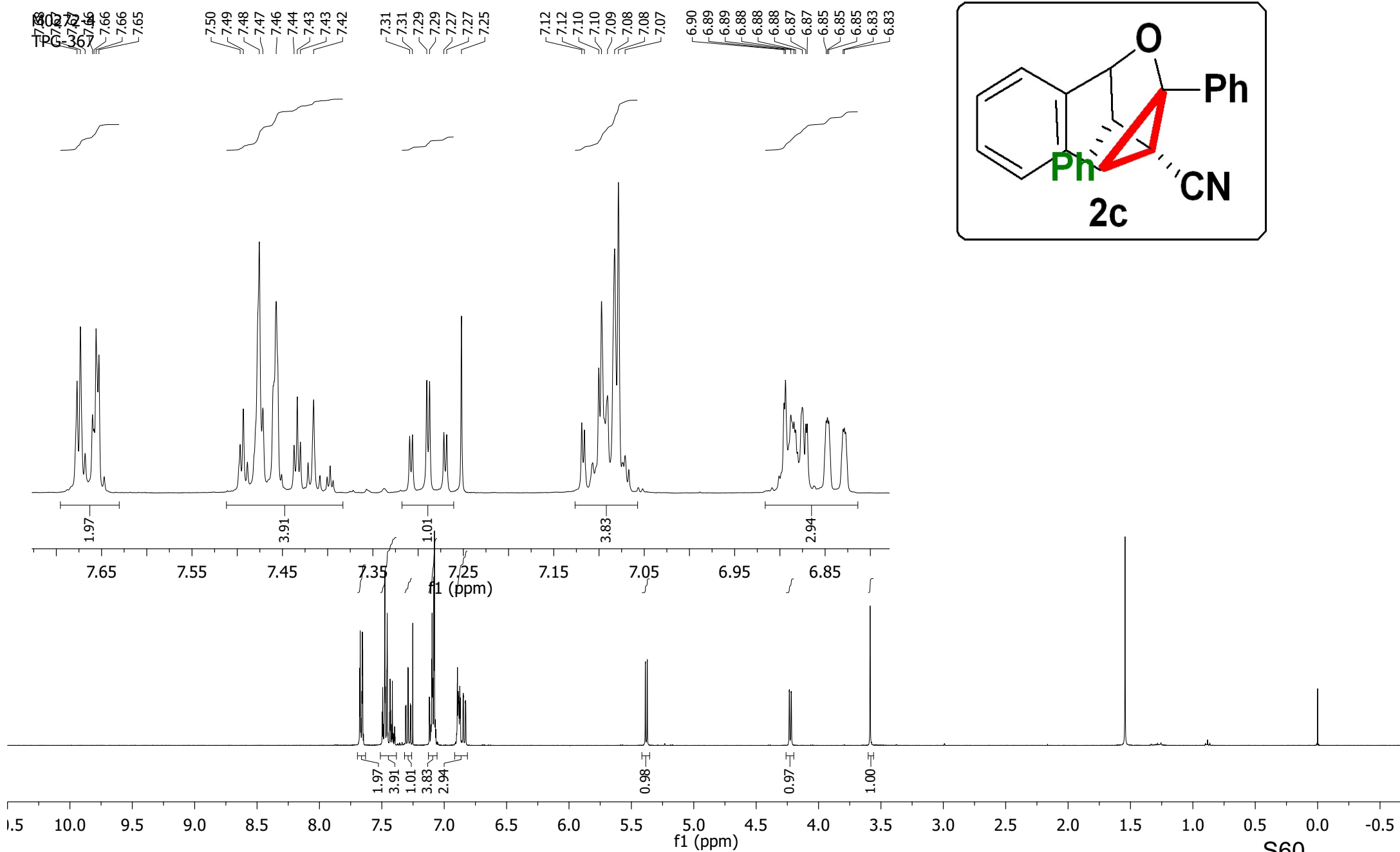
43.31

27.32
25.06
21.12



M0278
TPG-367

Solvent CDCl₃
Spectrometer Frequency 400.40



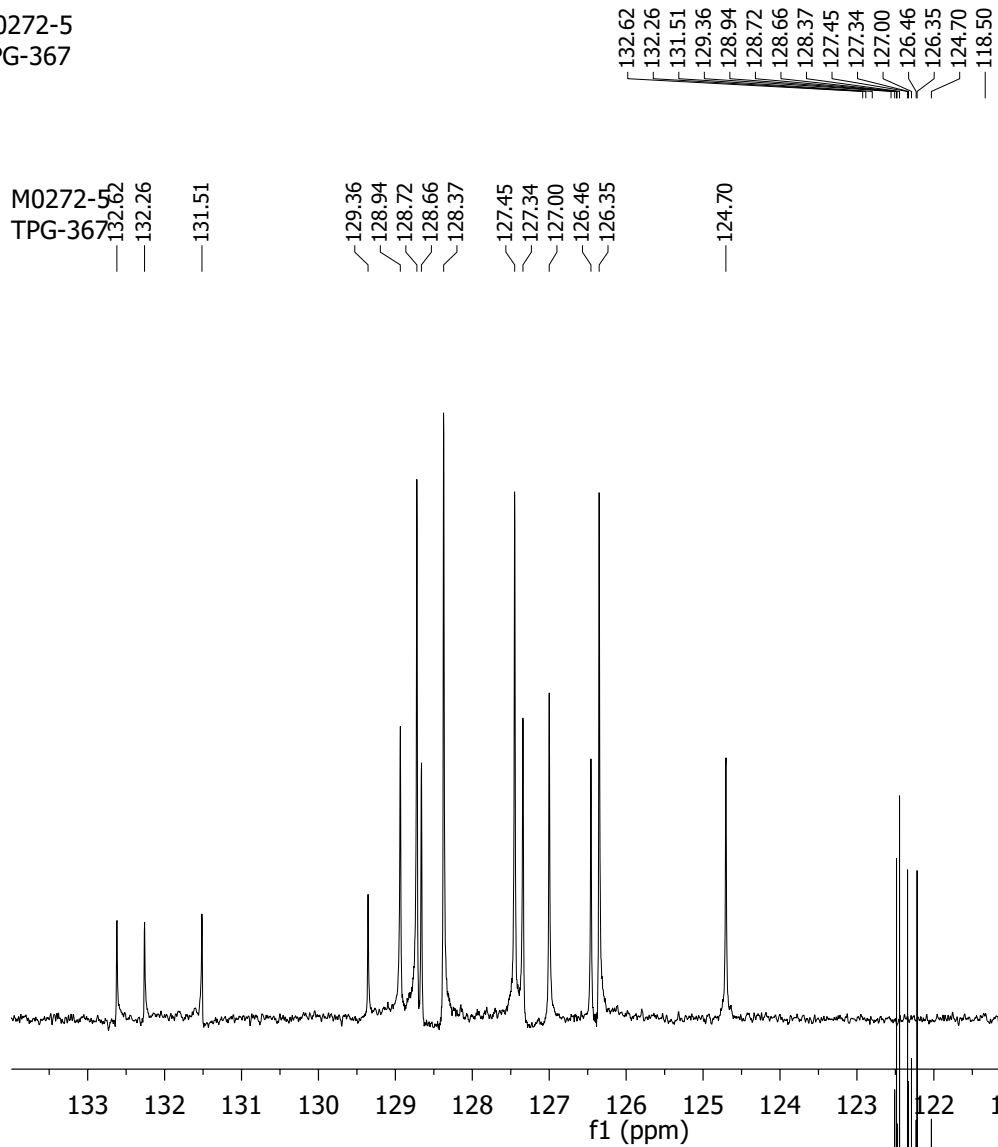
M0272-5
TPG-367

M0272-5
TPG-367
—132.62
—132.26
—131.51

—129.36
—128.94
—128.72
—128.66
—128.37
—127.45
—127.34
—127.00
—126.46
—126.35

—124.70

—118.50

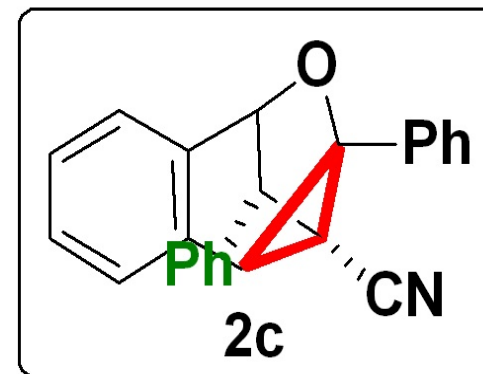


—79.12
—77.31
—76.99
—76.67
—69.46

—46.92

—31.03

—24.01



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

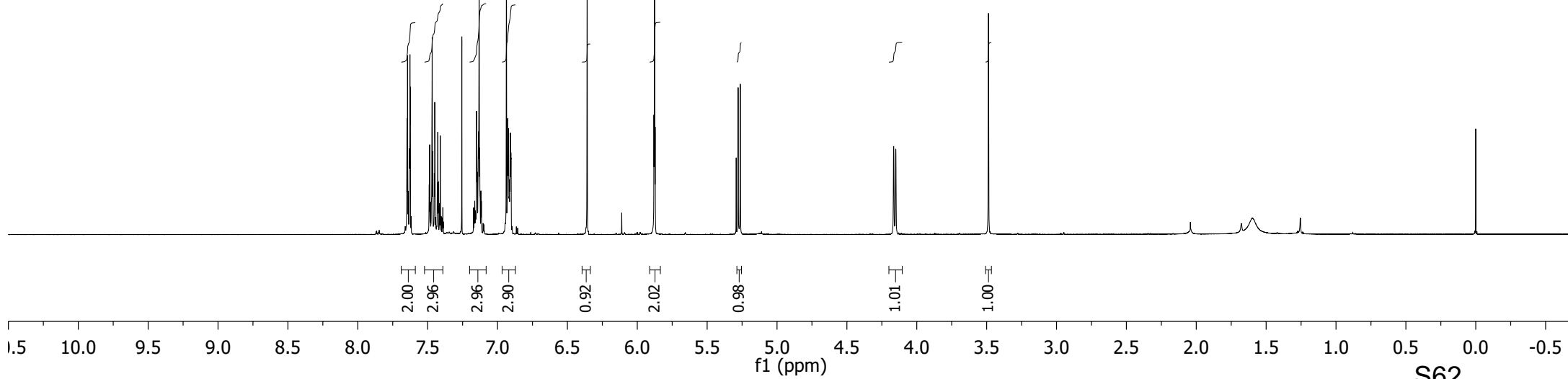
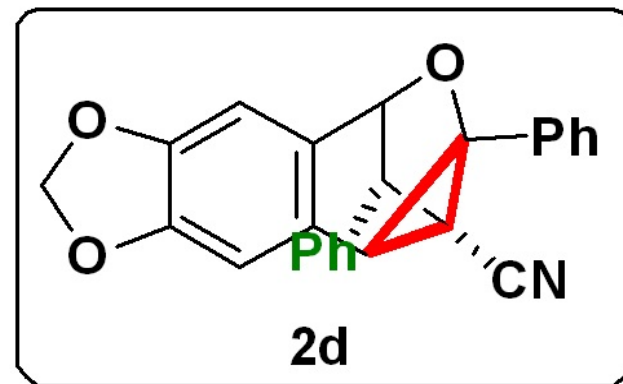
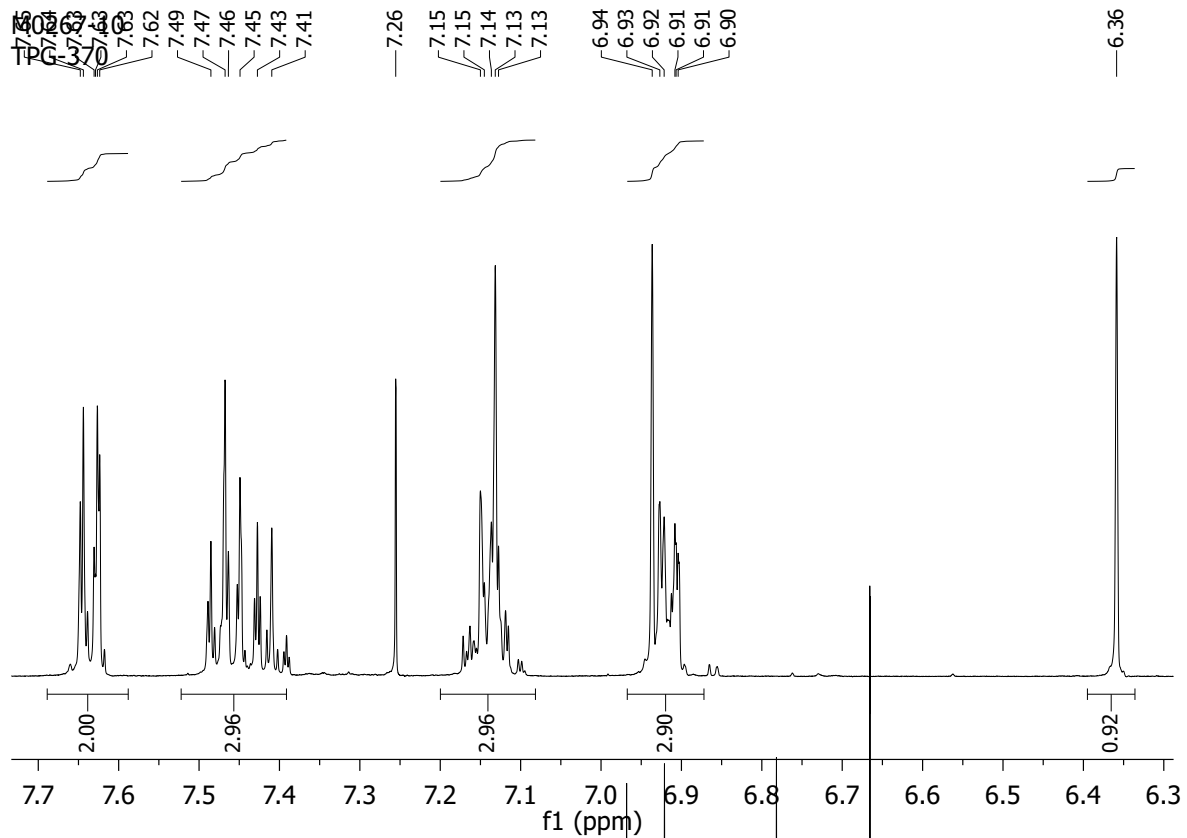
S61

M0267-10
TPG-370

Solvent

CDCl₃

Spectrometer Frequency 400.40



M0267-11
TPG-370

Solvent CDCl_3
Spectrometer Frequency 100.69

147.71
146.78
132.67
132.26
128.90
128.71
128.50
127.51
127.39
126.29
125.32
122.79
118.55
107.15
106.14
101.04

78.84
77.31
76.99
76.67

68.83

46.89

31.22

23.67

M0267-11
TPG-370

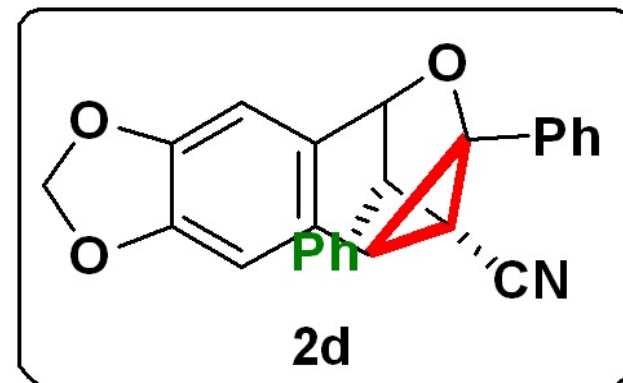
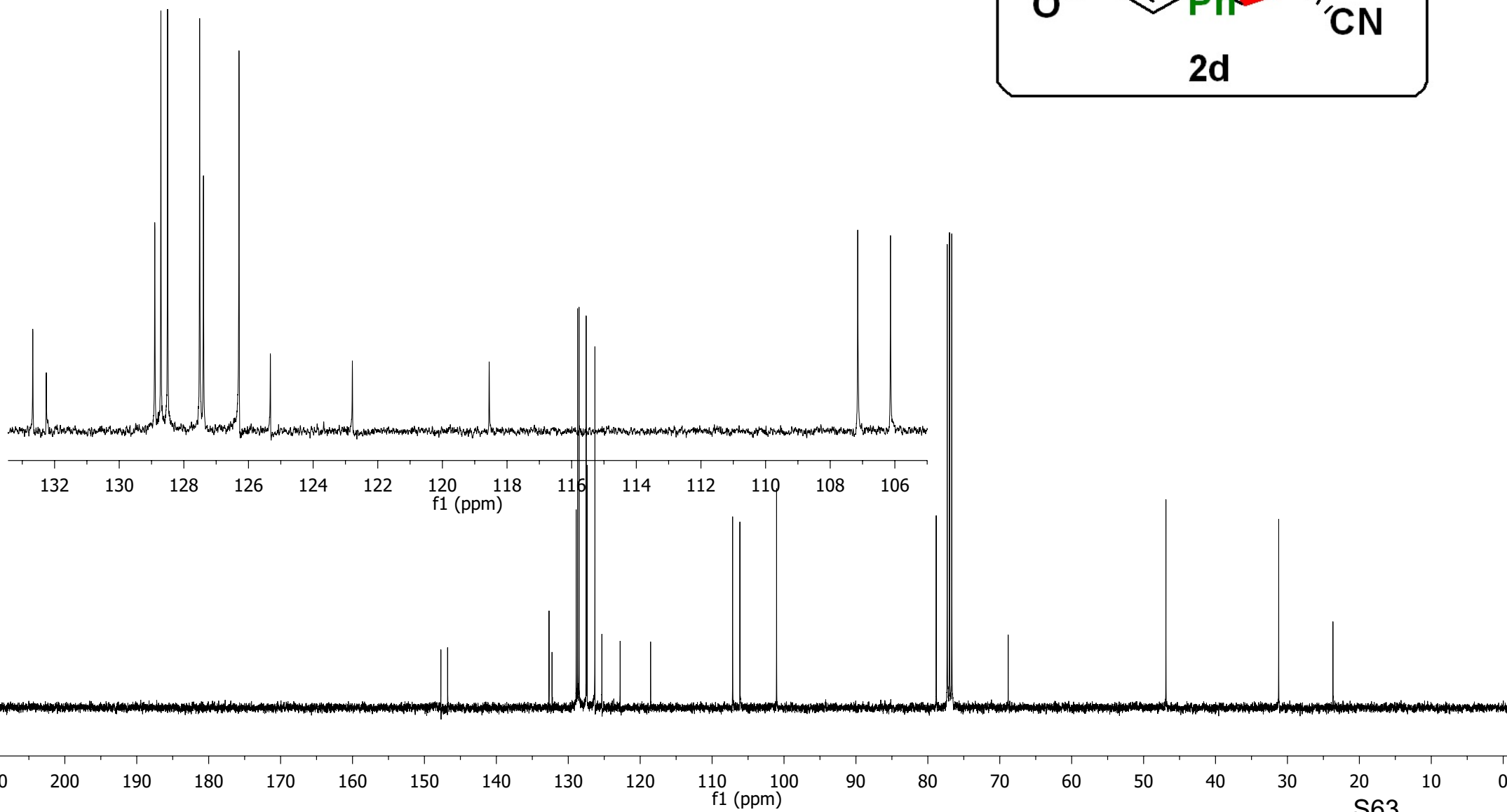
128.90
128.71
128.50
127.51
127.39
126.29
125.32

122.79

118.55

107.15

106.14



M0257-7
TPG-361

Solvent CDCl₃
Spectrometer Frequency 400.28

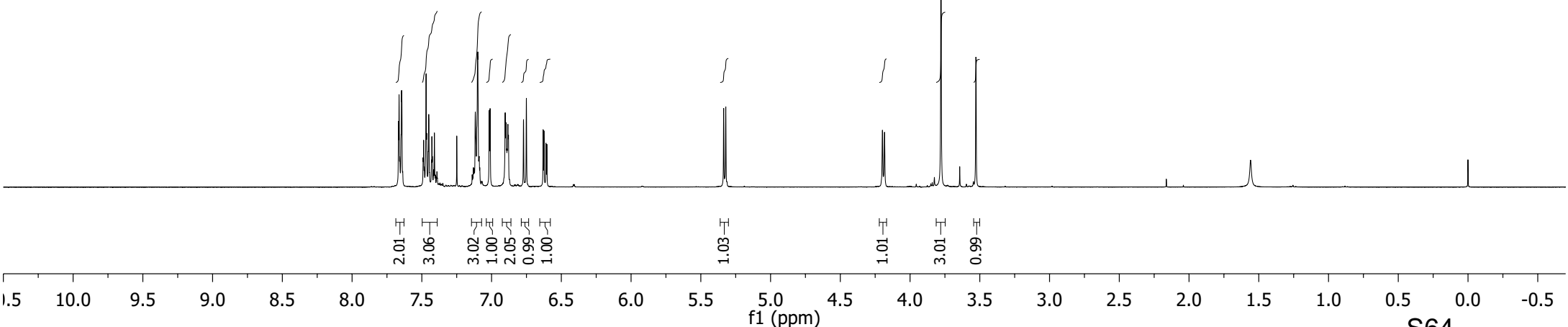
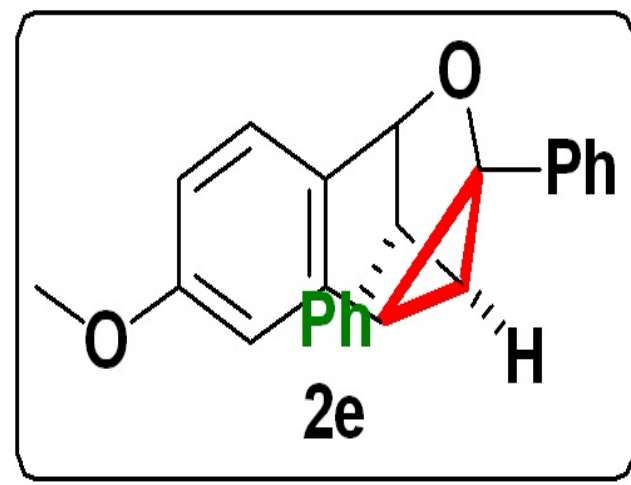
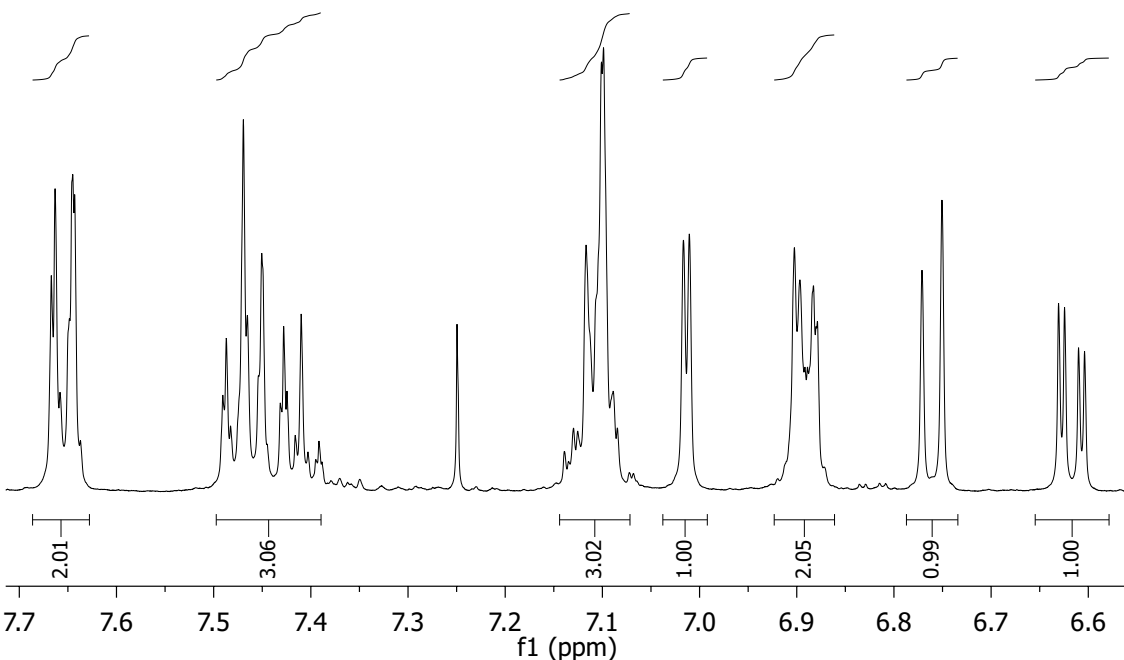
7.67 7.66 7.66 7.65 7.64 7.49 7.49 7.47 7.47 7.45 7.43 7.43 7.42 7.41 7.25 7.12 7.10 7.10 7.09 7.02 7.01 6.90 6.90 6.89 6.89 6.88 6.88 6.77 6.75 6.63 6.62 6.61 6.60 5.34 5.32

4.20 4.18 3.78 3.53

1.56

0.00

7.67 7.66 7.66 7.65 7.64 7.49 7.49 7.47 7.47 7.45 7.43 7.43 7.42 7.41 7.25 7.12 7.10 7.10 7.09 7.02 7.01 6.90 6.90 6.89 6.89 6.88 6.88 6.77 6.75 6.63 6.62 6.61 6.60

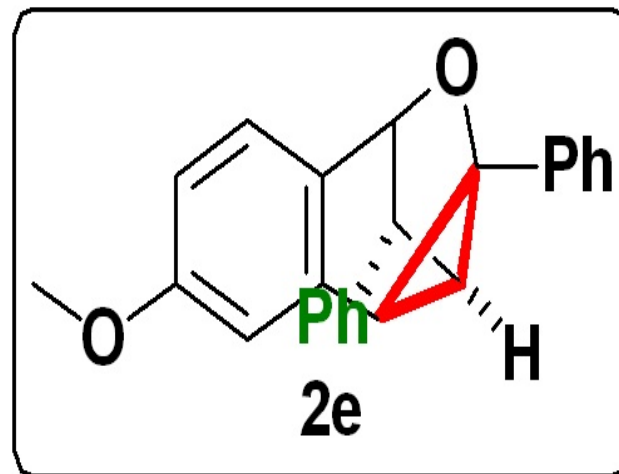
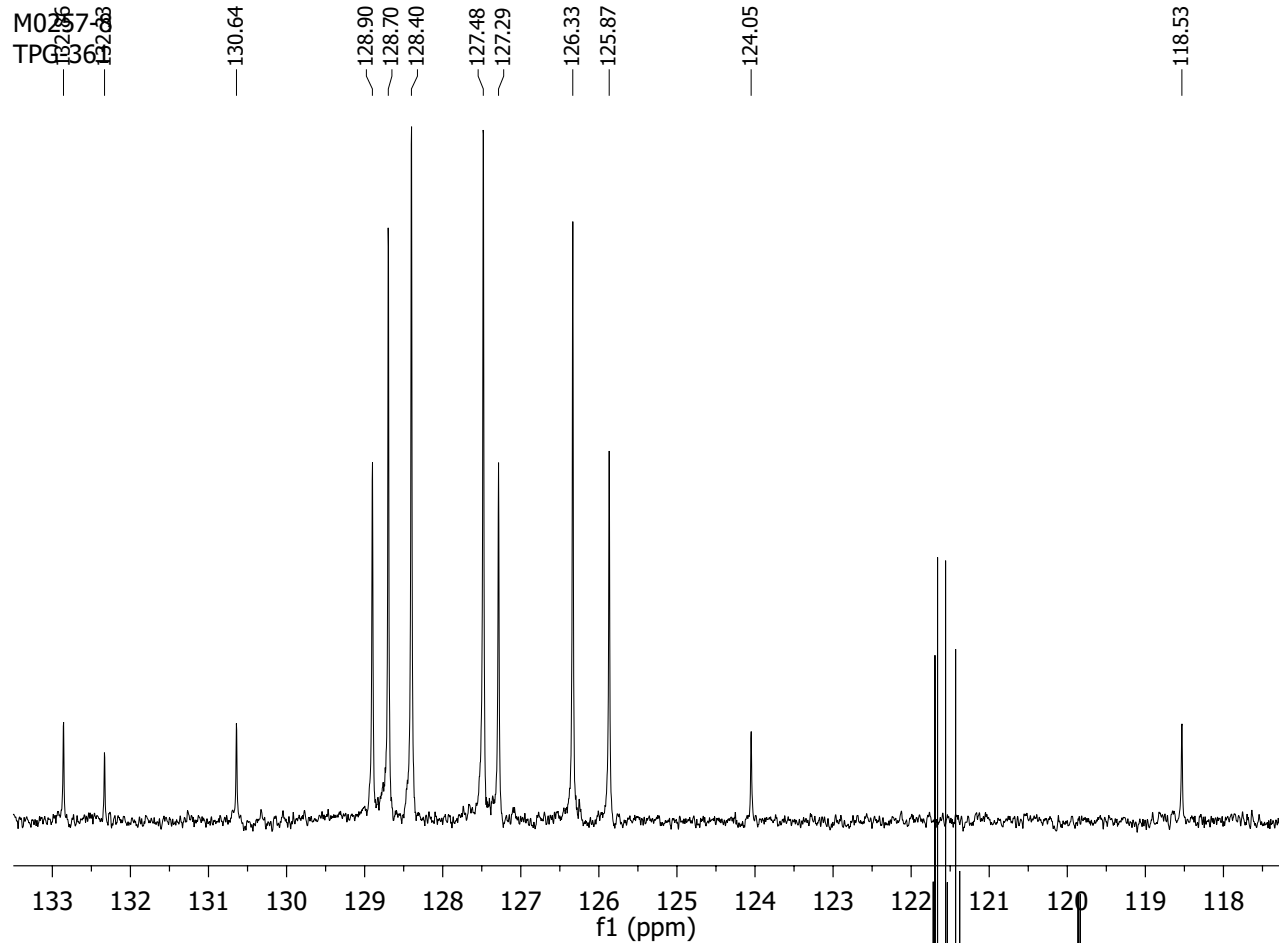


M0257-8
TPG-361

Solvent

CDCl_3

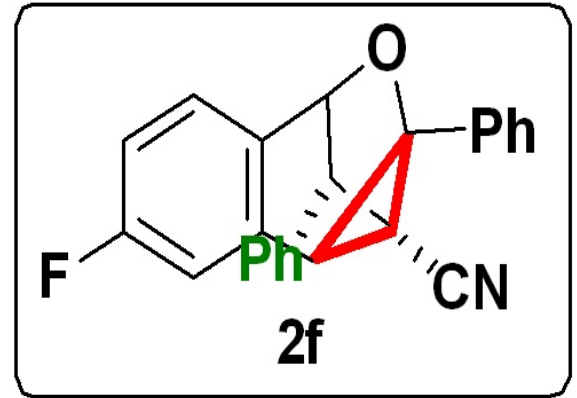
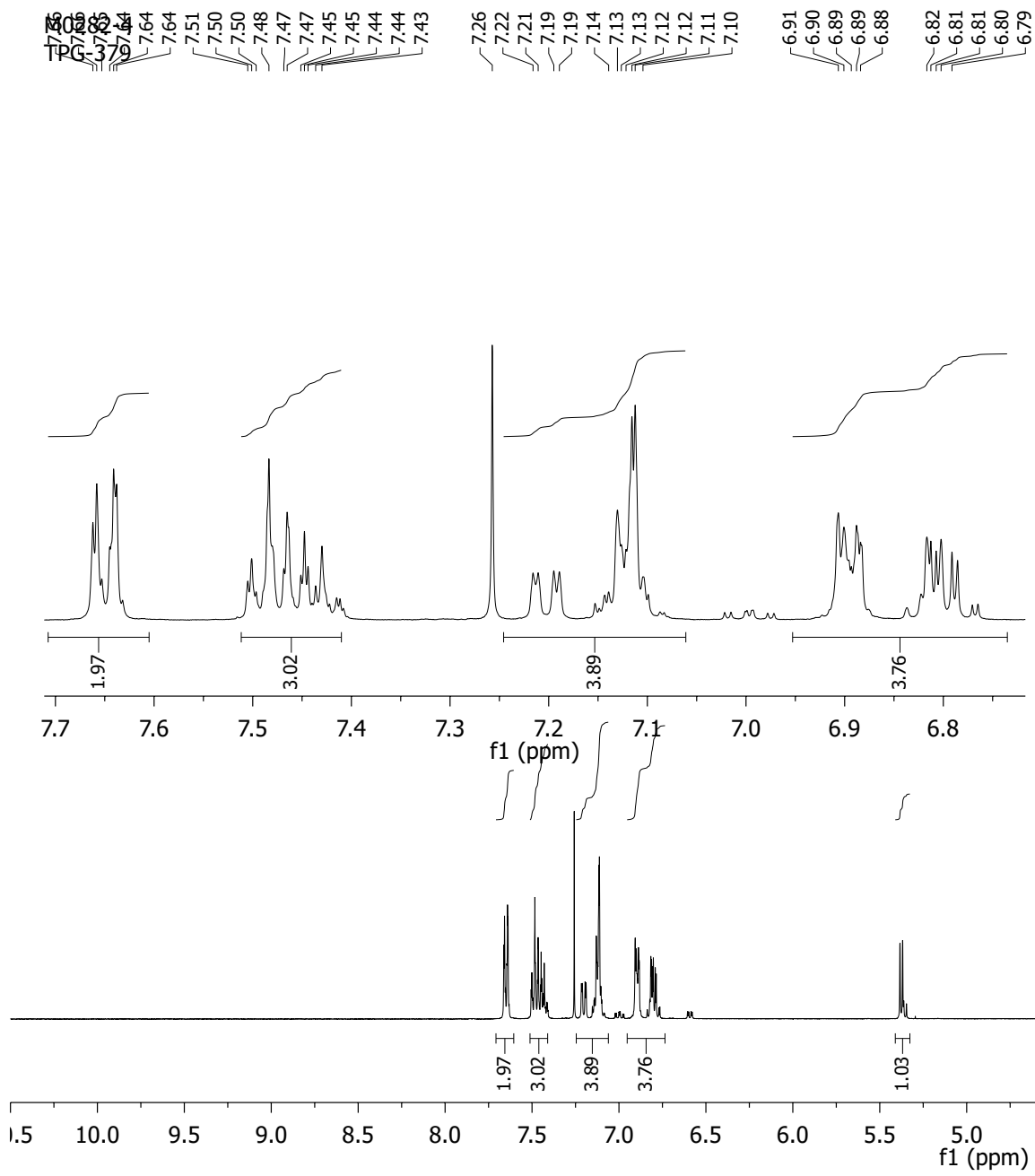
Spectrometer Frequency 100.66



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

S65

M02894
TPG-379
Solvent CDCl₃
Spectrometer Frequency 400.40



M0282-5
TPG-379

Solvent

CDC₃

Spectrometer Frequency 100.69

163.85
161.39
132.39
131.85
131.45
131.36
129.12
129.06
128.79
128.54
128.54
127.59
127.49
127.36
127.33
126.46
126.38
126.32
118.10
115.81
115.52
114.07
113.85
113.62
112.54
112.25

78.53
77.31
76.99
76.67
69.35

46.94
46.70

30.91
30.88
30.37

24.09

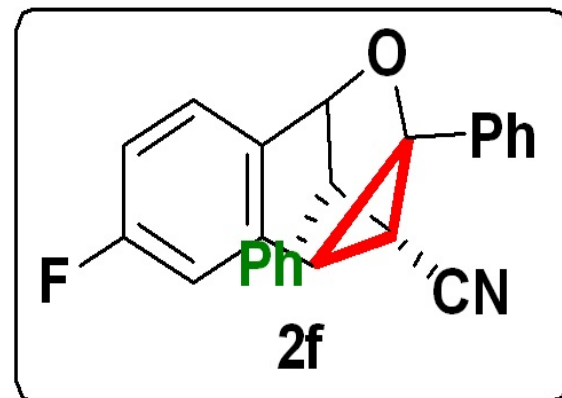
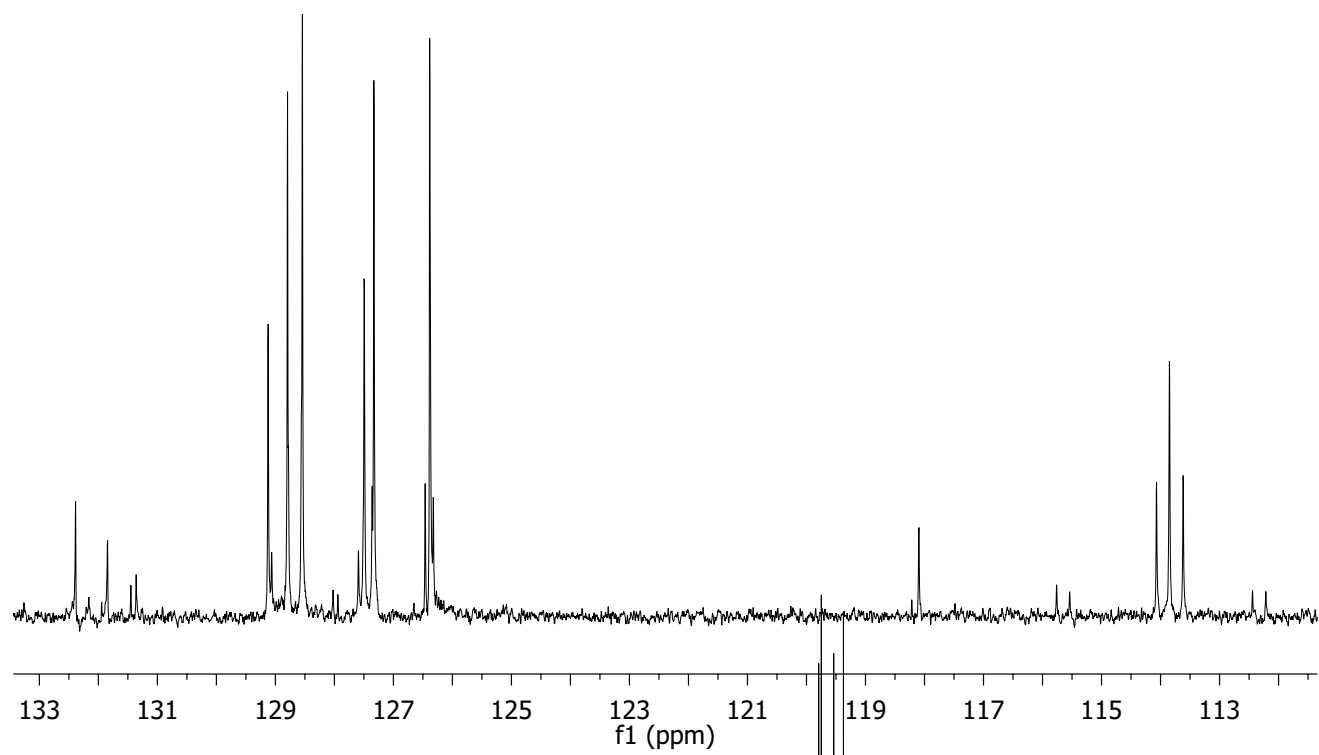
208.25
207.25
131.45
131.36
129.12
129.06
128.79
128.54
127.49
127.33
126.46
126.38
126.32

118.10

115.81
115.52

114.07
113.85
113.62

112.54
112.25



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

S67

M0282-1-1
TPG-378

7.65
7.65
7.64
7.63
7.63
7.50
7.50
7.48
7.48
7.46
7.45
7.44
7.44
7.43
7.42
7.40
7.29
7.28
7.27
7.26
7.14
7.14
7.13
7.12
6.91
6.90
6.89
6.89
6.87
6.86
5.36
5.34

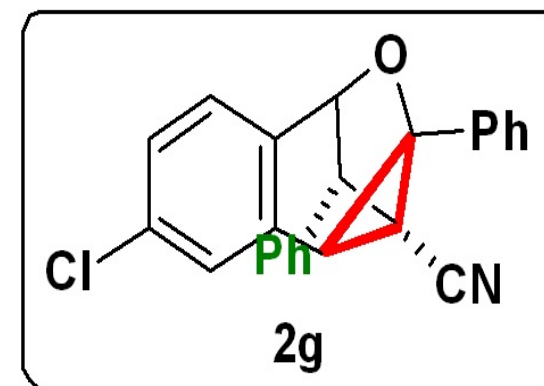
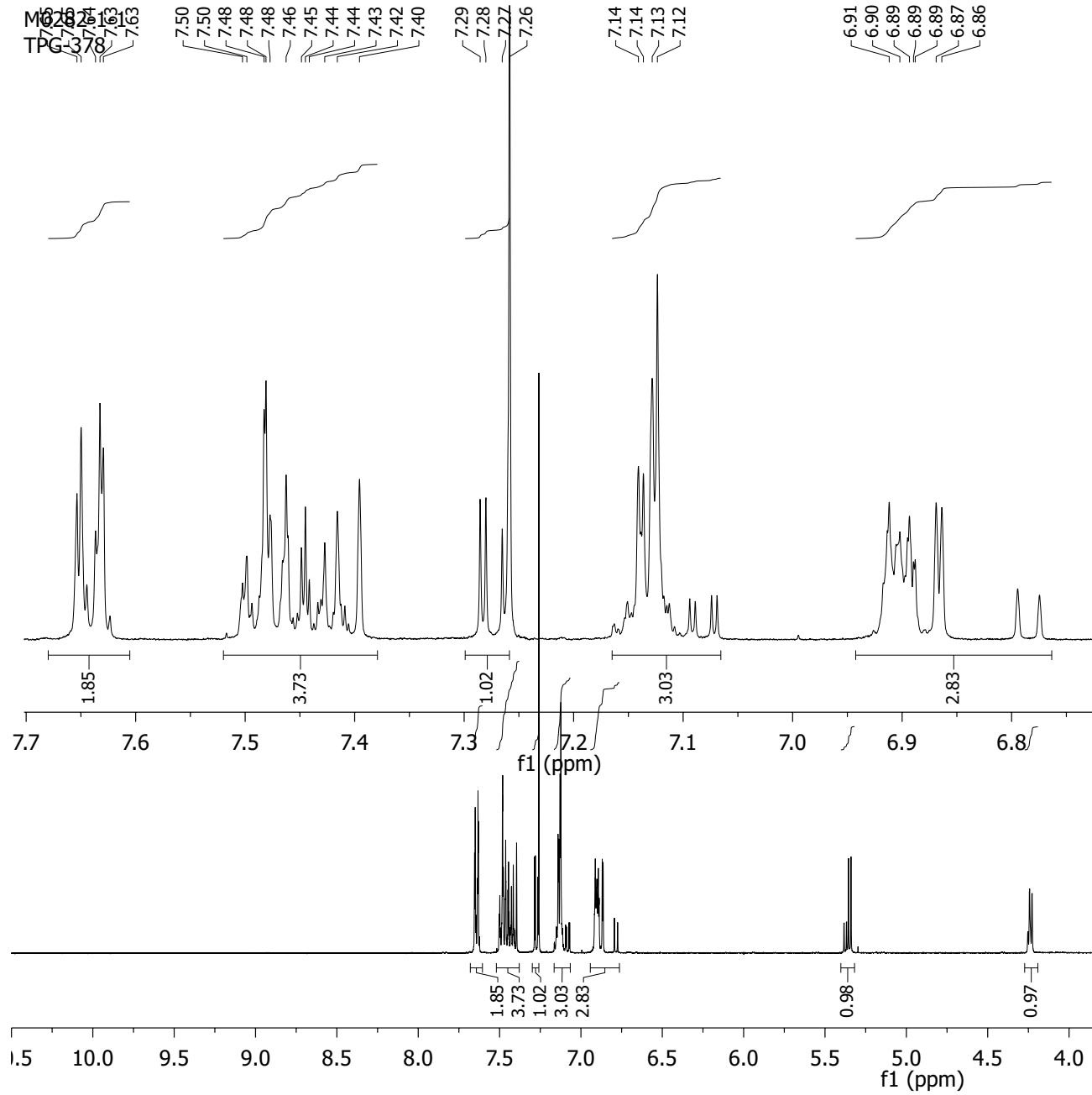
4.24
4.23

3.57
3.54

1.54

-0.00

Solvent CDCl₃
Spectrometer Frequency 400.40



S68

M0282-2
TPG-378

Solvent

CDCl₃

Spectrometer Frequency 150.19

M0282-2

TPG-378

129.17

129.11

128.81

128.80

128.63

127.94

127.73

127.67

127.56

127.37

127.34

127.18

126.59

126.44

126.35

126.01

125.05

133.05

133.02

132.08

131.85

129.17

129.11

128.81

128.80

128.63

127.94

127.73

127.67

127.56

127.37

127.34

127.18

126.59

126.44

126.35

126.01

125.05

118.06

78.51

77.20

76.99

76.78

69.52

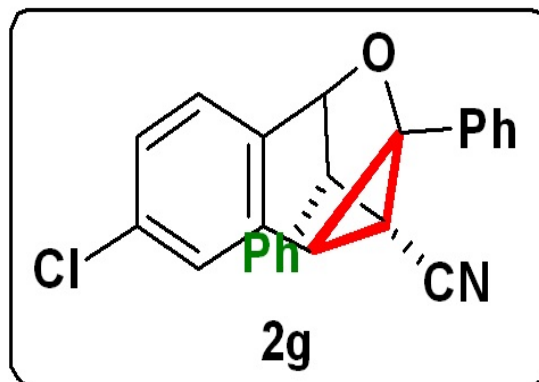
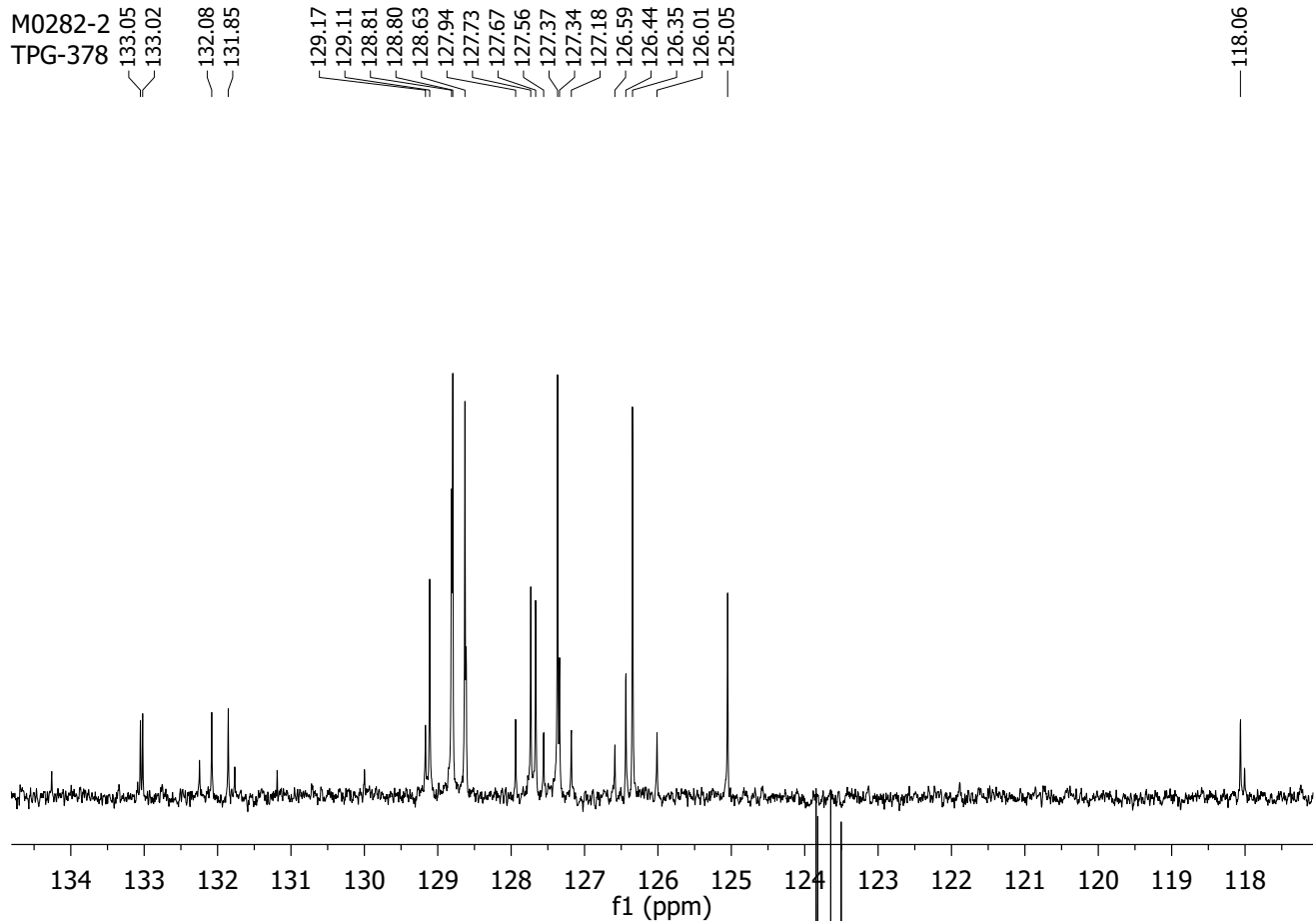
46.80

46.70

30.60

30.50

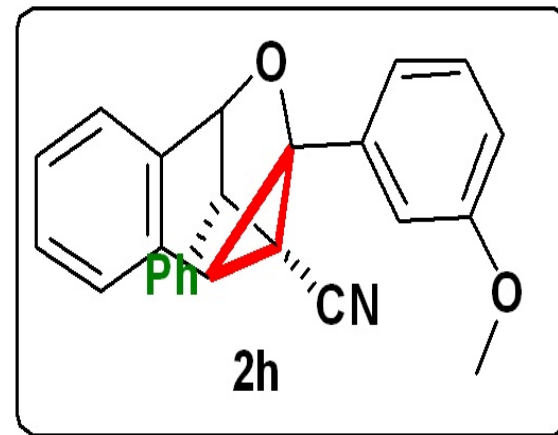
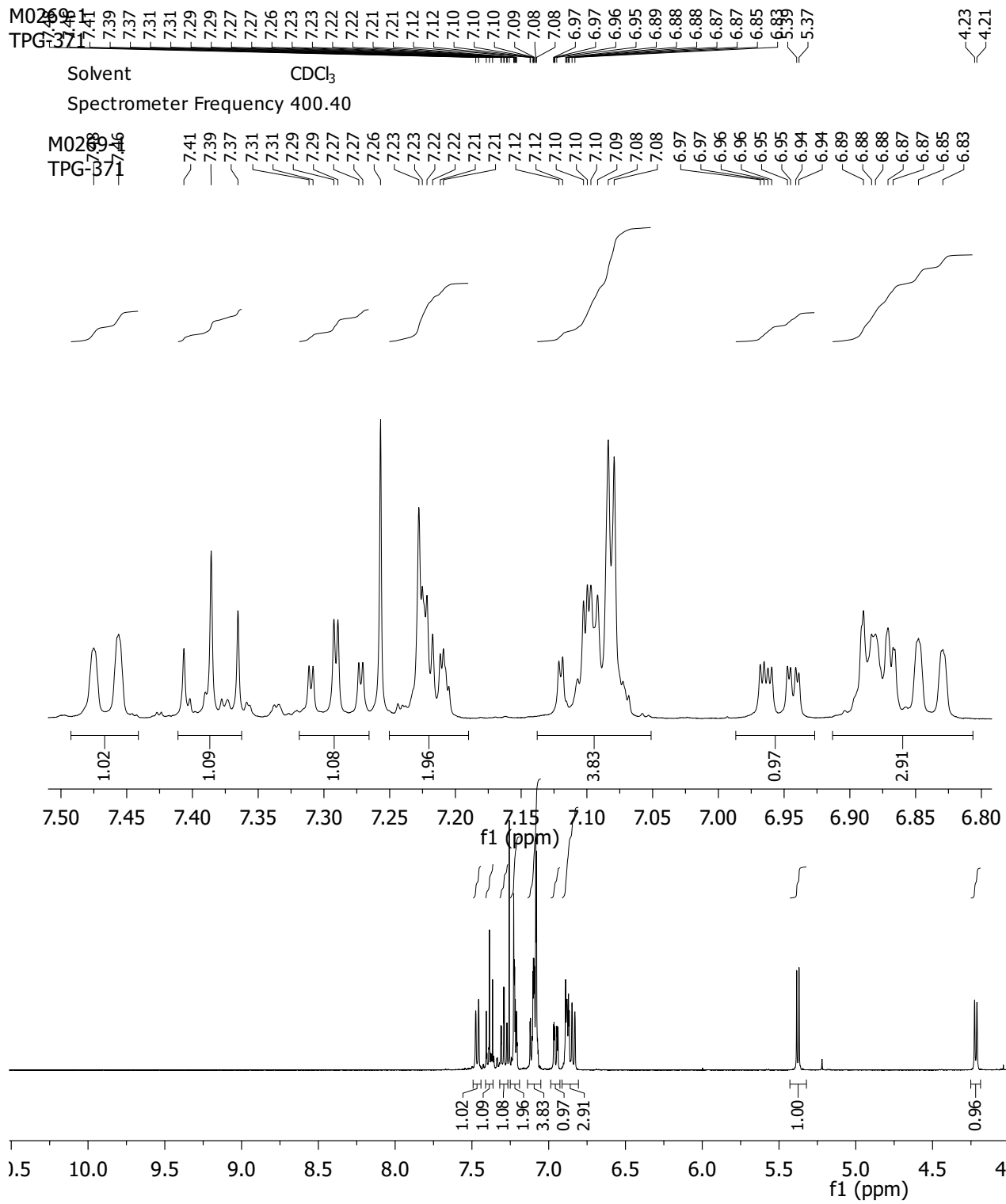
24.04



10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

S69



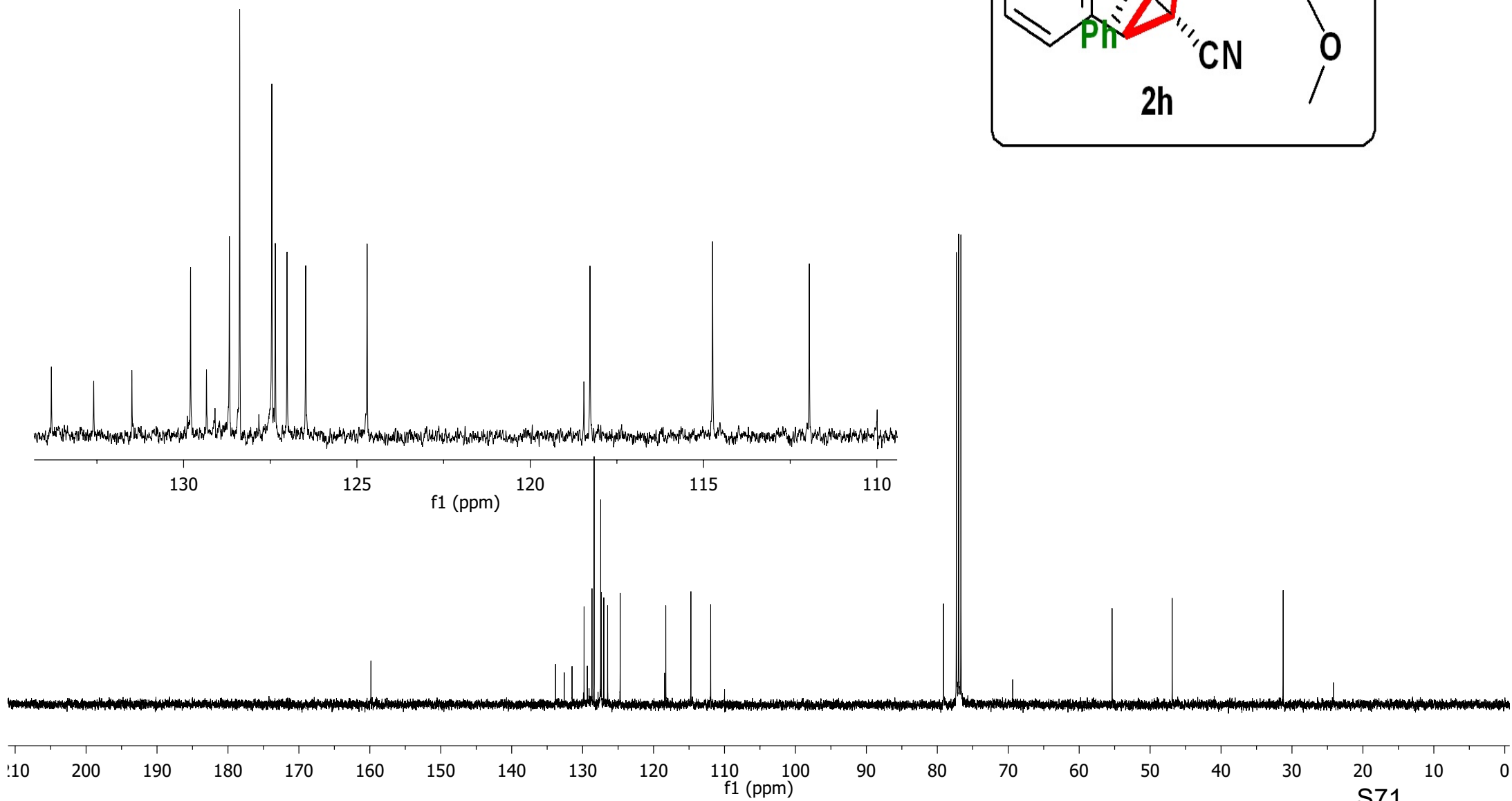
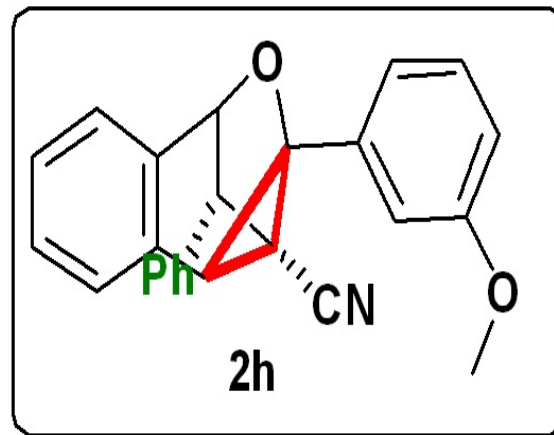
M0269-2
TPG-371 Solvent CDCl_3
Spectrometer Frequency 100.69

133.81
132.59
131.49
129.80
129.34
128.68
128.38
127.45
127.35
127.02
126.48
124.71
118.45
118.28
114.74
111.95
110.46

79.12
77.31
76.99
76.68
69.37
55.36
46.89
31.24
24.17

M0269-2
TPG-371
131.49
129.80
129.34
128.68
128.38
127.45
127.35
127.02
126.48
124.71

118.45
118.28
114.74
111.95
110.46



M0252-15
TPG-355
Solvent CDCl_3
Spectrometer Frequency 400.40

7.56
7.55
7.54
7.53
7.48
7.46
7.31
7.30
7.29
7.29
7.27
7.27
7.26
7.11
7.11
7.10
7.10
7.09
7.09
7.08
7.08
7.07
7.06
6.89
6.88
6.88
6.87
6.87
6.87
6.84
6.82
5.37
5.36

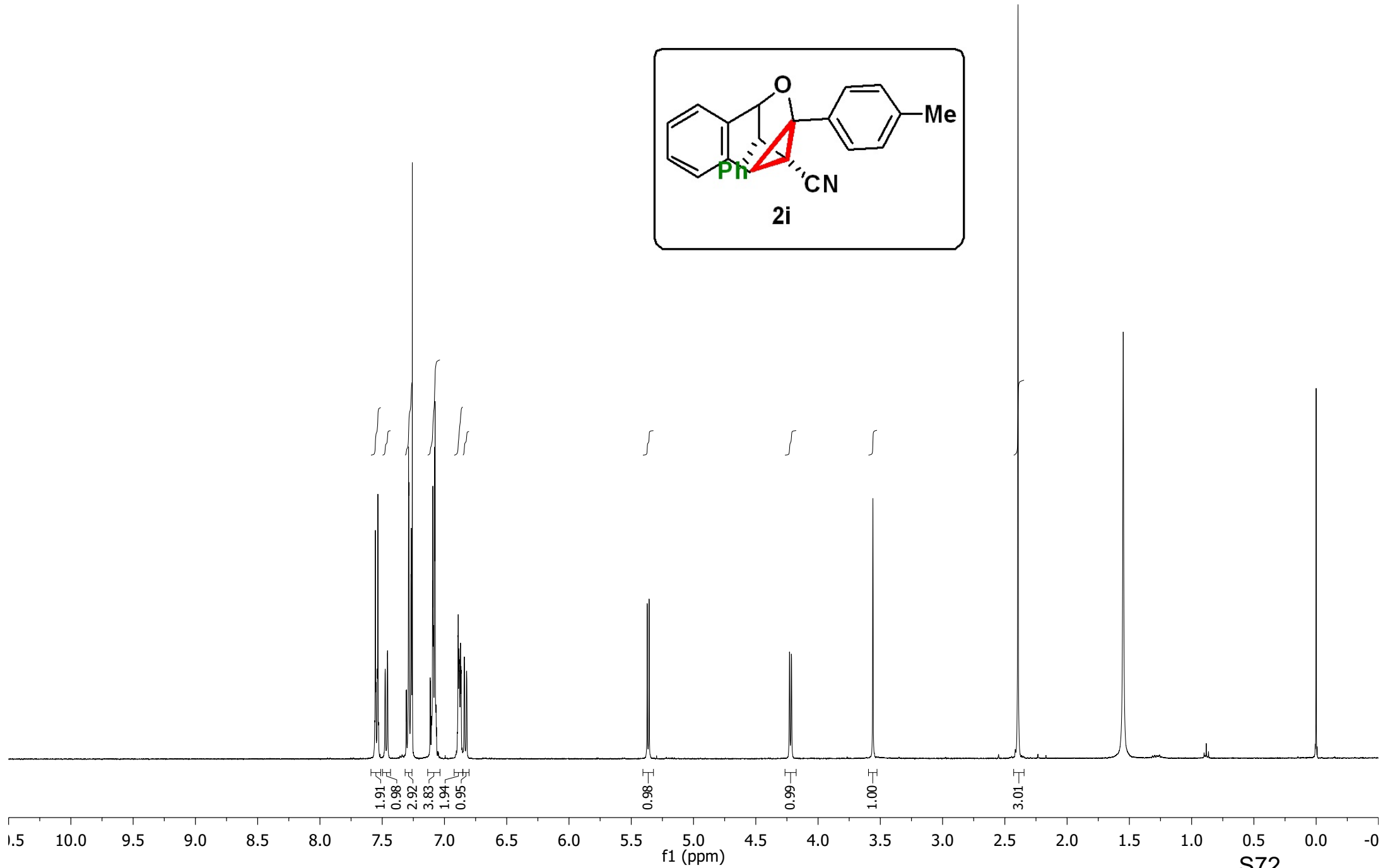
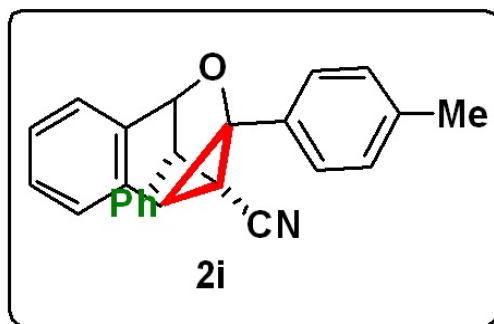
4.23
4.21

3.56

2.39

1.55

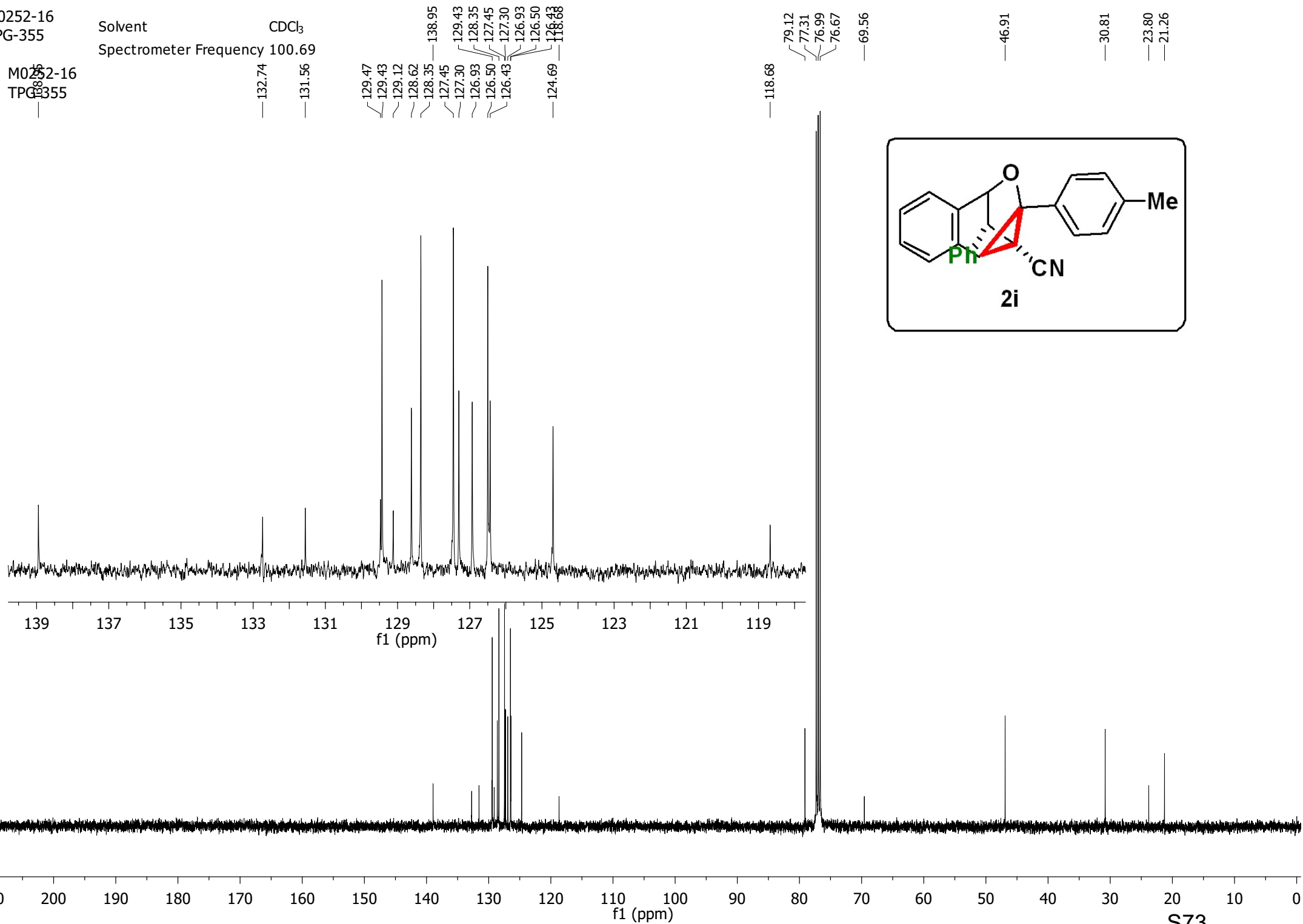
0.00



M0252-16
TPG-355

Solvent CDCl_3
Spectrometer Frequency 100.69

M0252-16
TPG-355

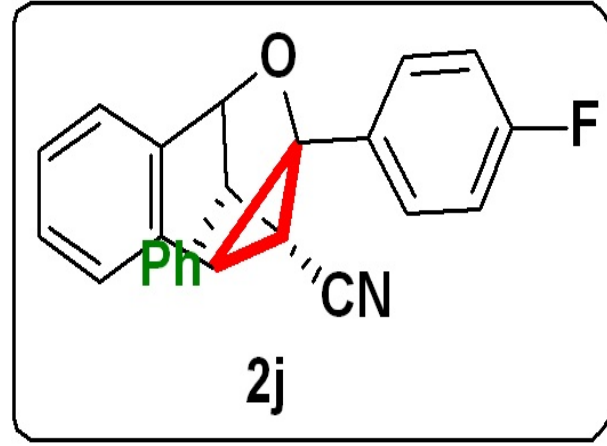
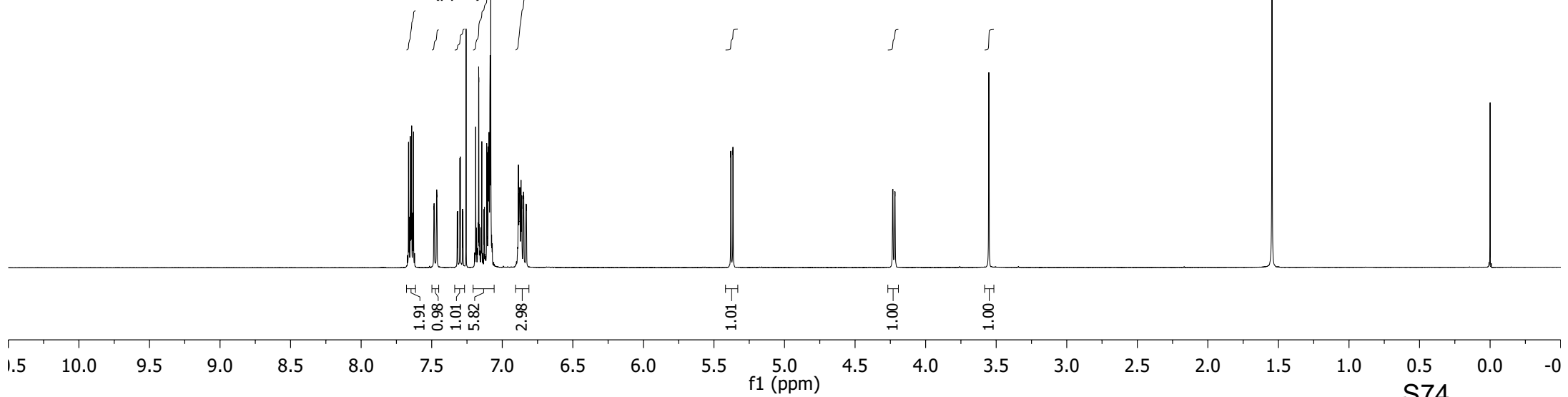
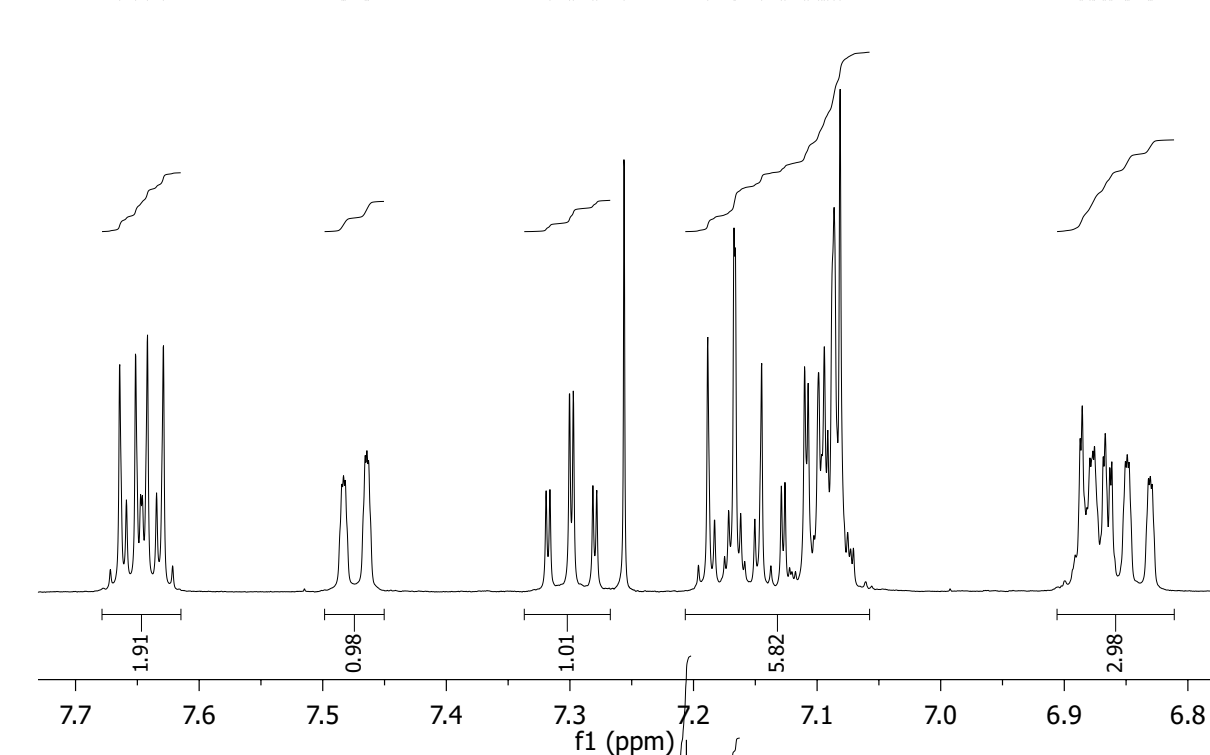


MO-55
TPG-356

Solvent CDCl_3

Spectrometer Frequency 400.40

MO-55
TPG-356



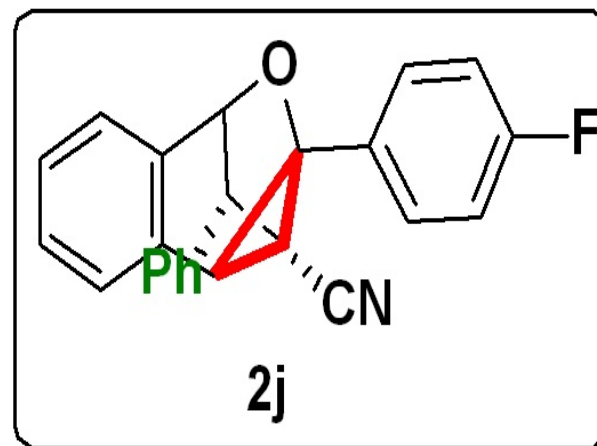
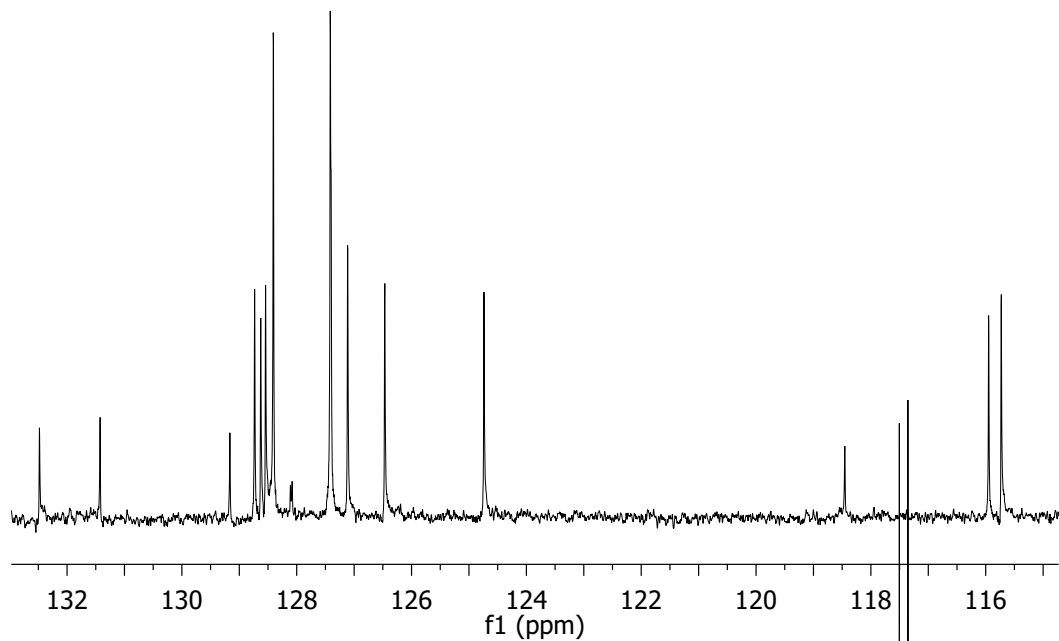
M0254-2
TPG-356

Solvent CDCl_3
Spectrometer Frequency 100.69

164.32
161.85
132.48
131.42
129.16
128.73
128.63
128.54
128.41
128.08
127.41
127.11
126.47
124.74
118.45
115.95
115.73

79.19
77.31
76.99
76.67
69.11
46.86
30.83
23.82

129.16
128.73
128.63
128.54
128.41
128.08
127.41
127.11
126.47
124.74
118.45
115.95
115.73

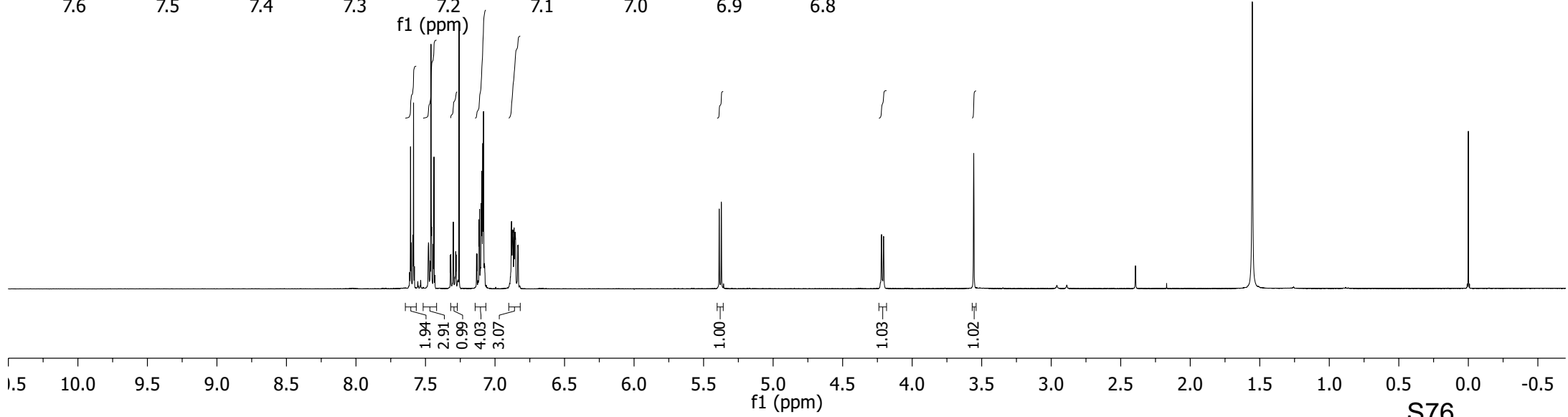
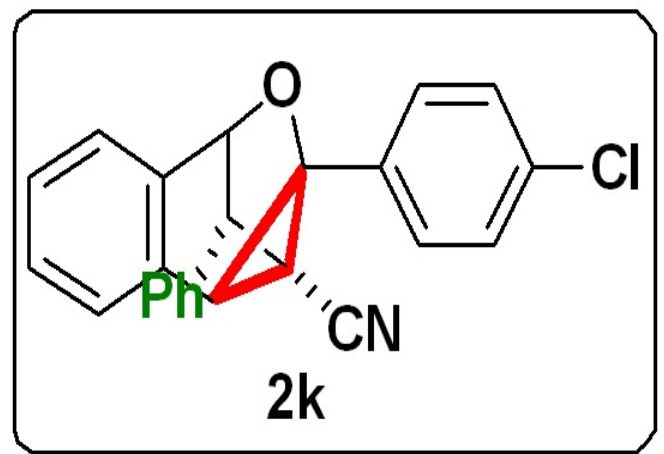
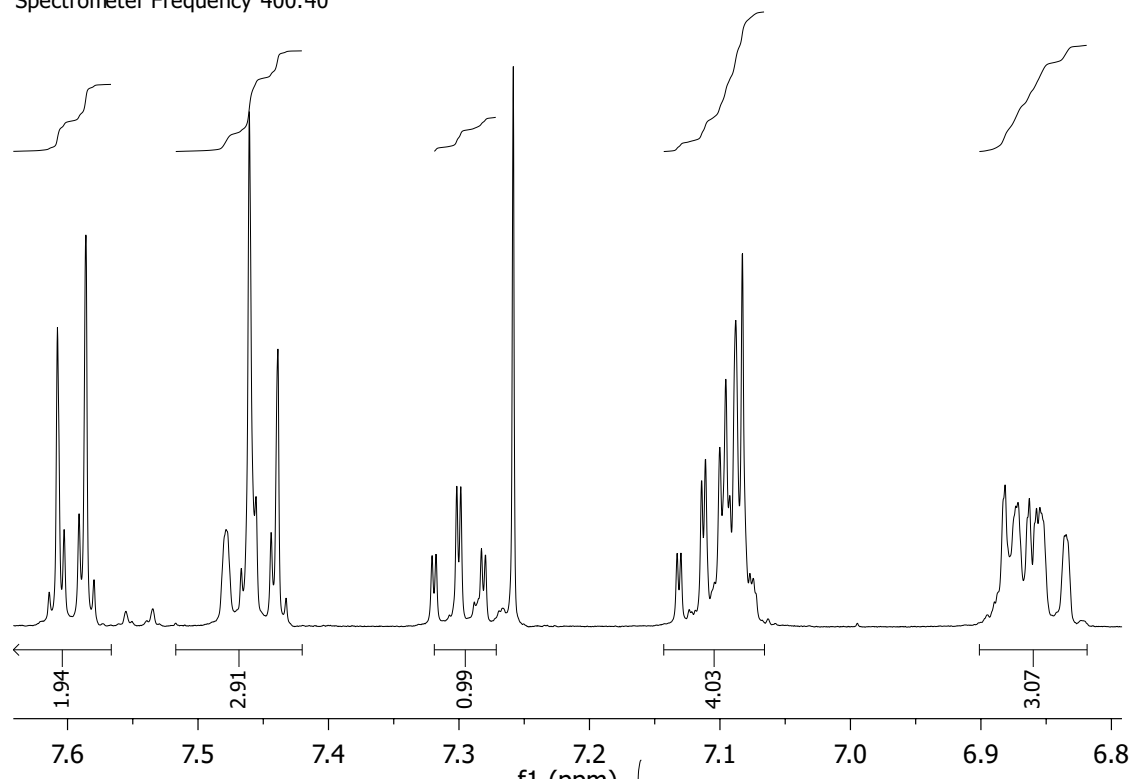


230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
S75

MQZ-51
 TPG-358
 TPG-358
 7.6251
 7.6164
 7.6059
 7.5959
 7.58748
 7.5747
 7.5746
 7.5746
 7.5744
 7.5744
 7.5744
 7.5743
 7.5732
 7.5730
 7.5730
 7.5728
 7.5728
 7.5726
 7.5713
 7.5713
 7.5711
 7.5711
 7.5710
 7.5710
 7.5709
 7.5709
 7.5708
 7.5708
 7.5707
 7.5707
 7.5688
 7.5687
 7.5686
 7.5686
 7.5685
 7.5685
 7.5683
 7.5683
 6.9137
 6.8837
 6.8736
 6.8686
 6.8686
 6.8585
 6.8585

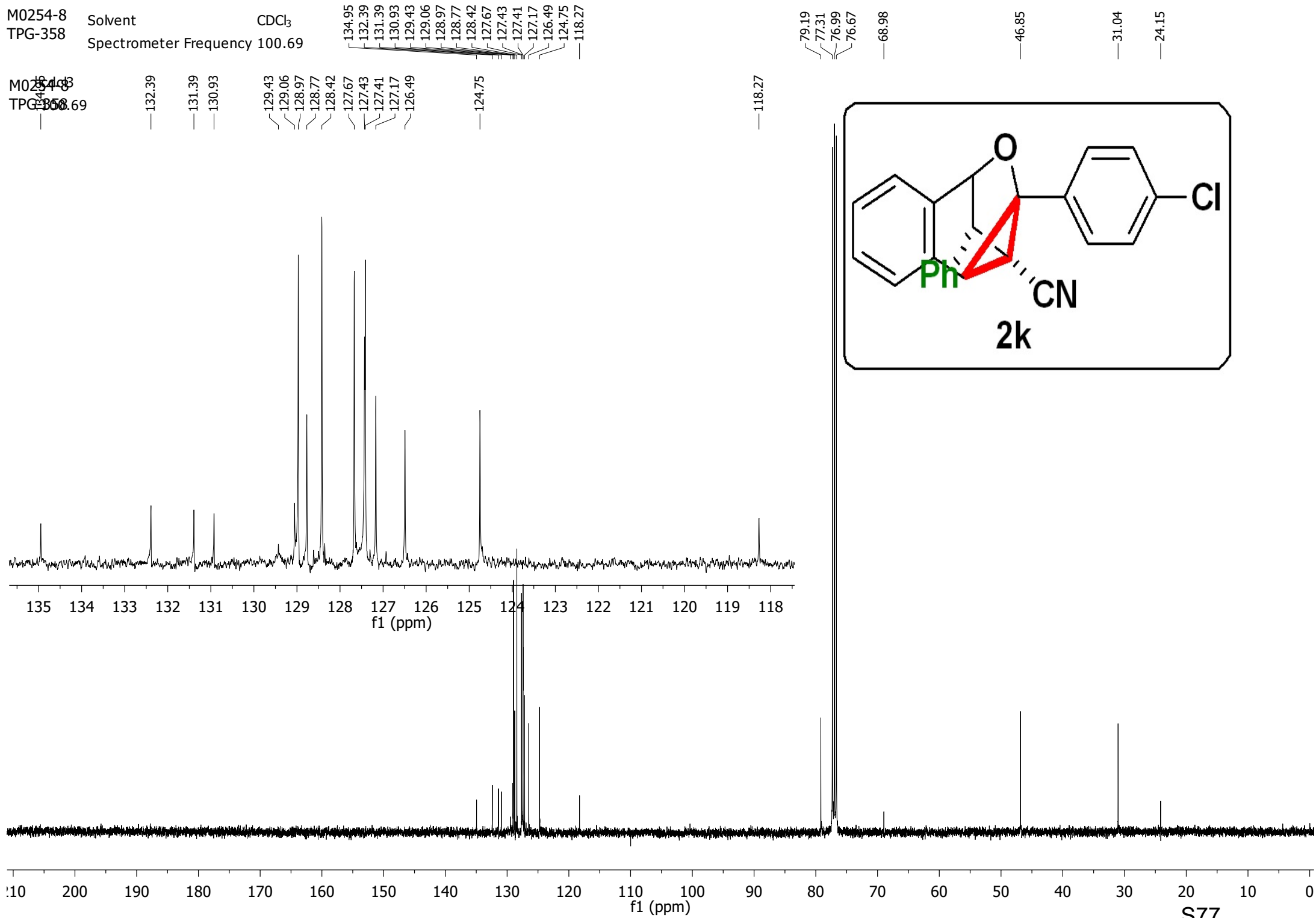
4.22
 4.21
 3.56
 2.96
 2.89
 2.39
 2.17
 1.55
 1.26
 0.88
 0.01
 -0.00
 -0.01

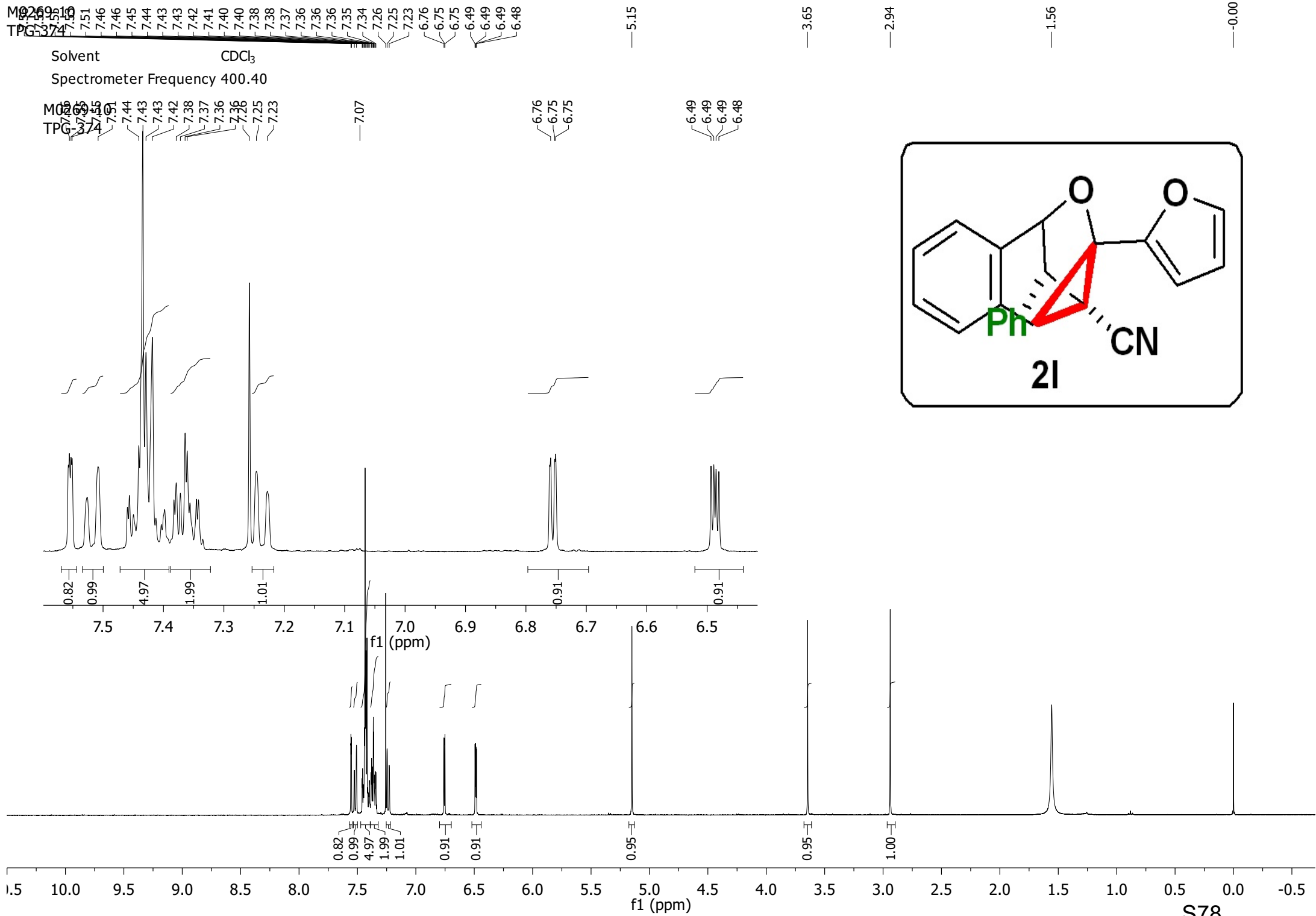
Solvent CDCl₃
 Spectrometer Frequency 400.40



M0254-8
TPG-358
Solvent
Spectrometer Frequency 100.69

M0254-8
TPG-358

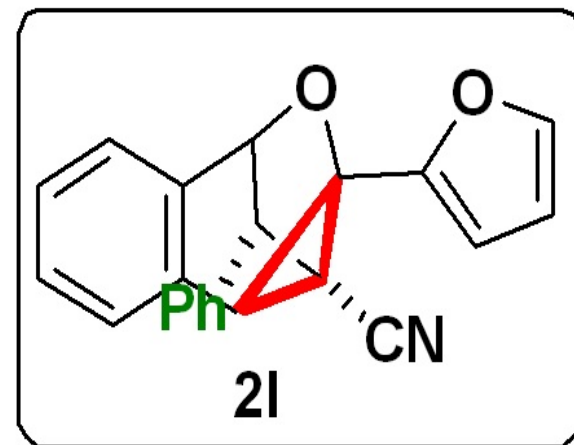
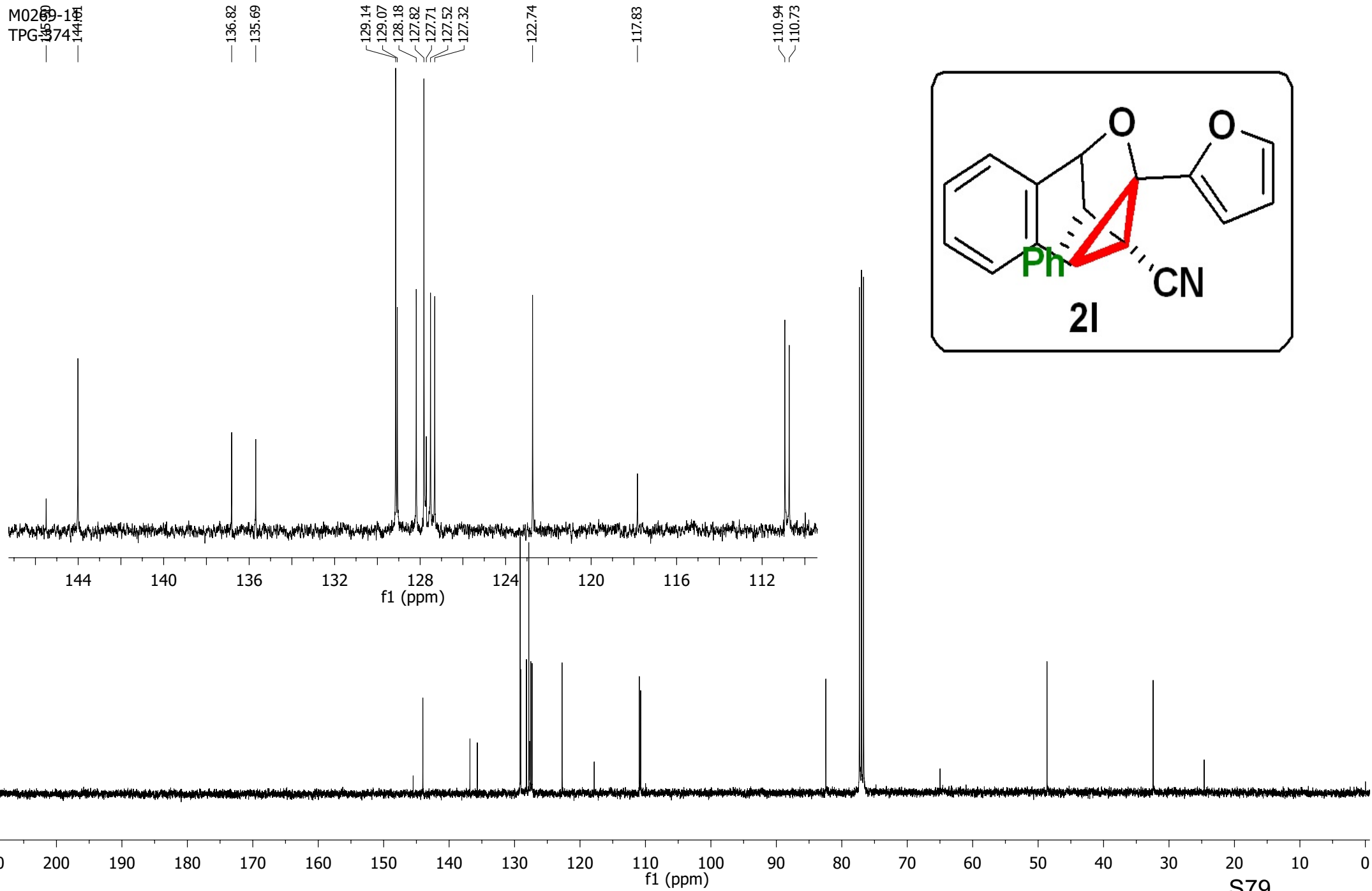




M0269-11
TPG-374

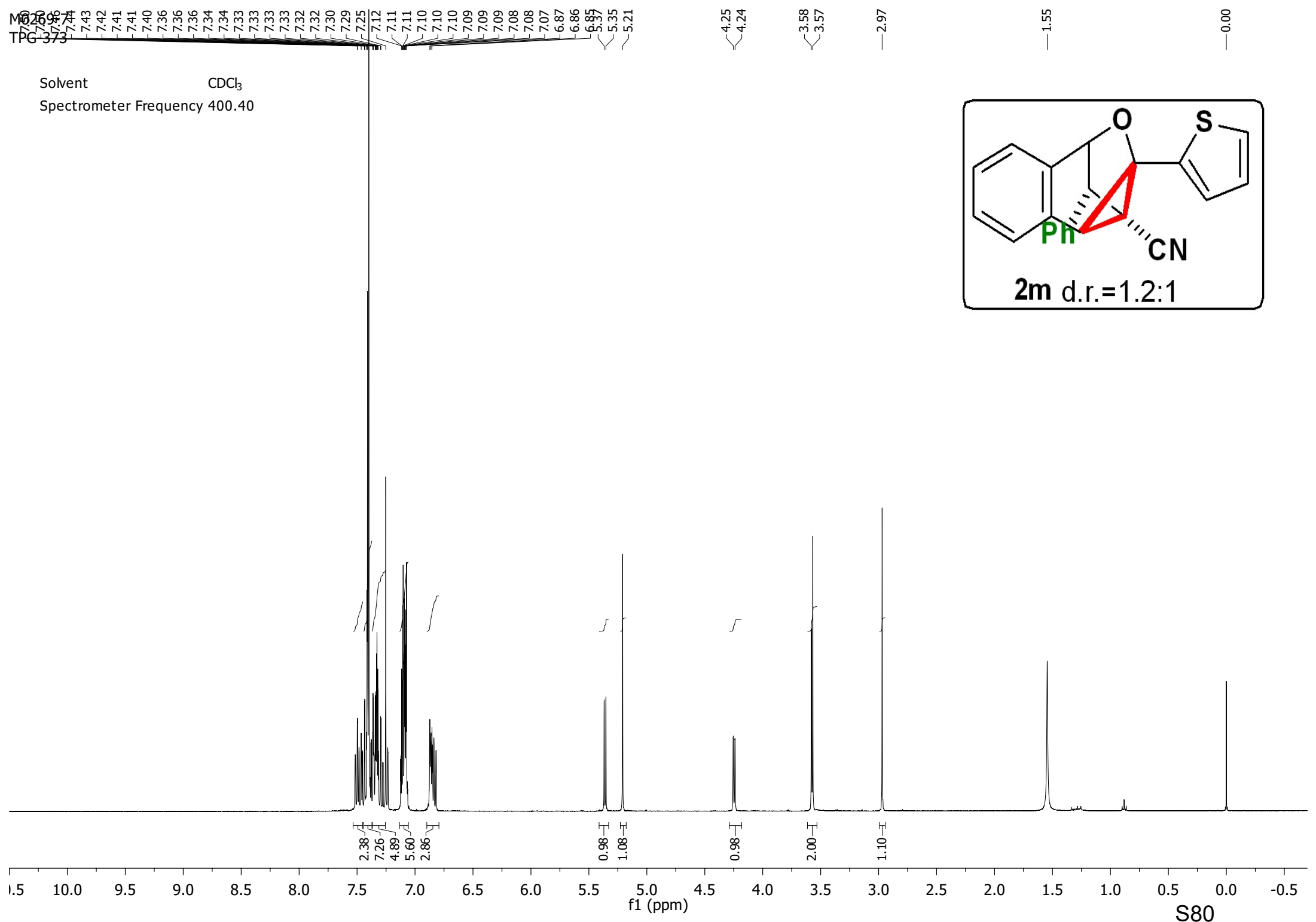
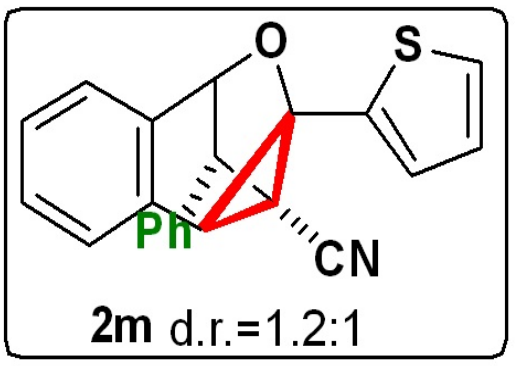
Solvent CDCl_3
Spectrometer Frequency 100.69

M0269-11
TPG-374



MW 269.4
TPG 373

Solvent CDCl_3
Spectrometer Frequency 400.40



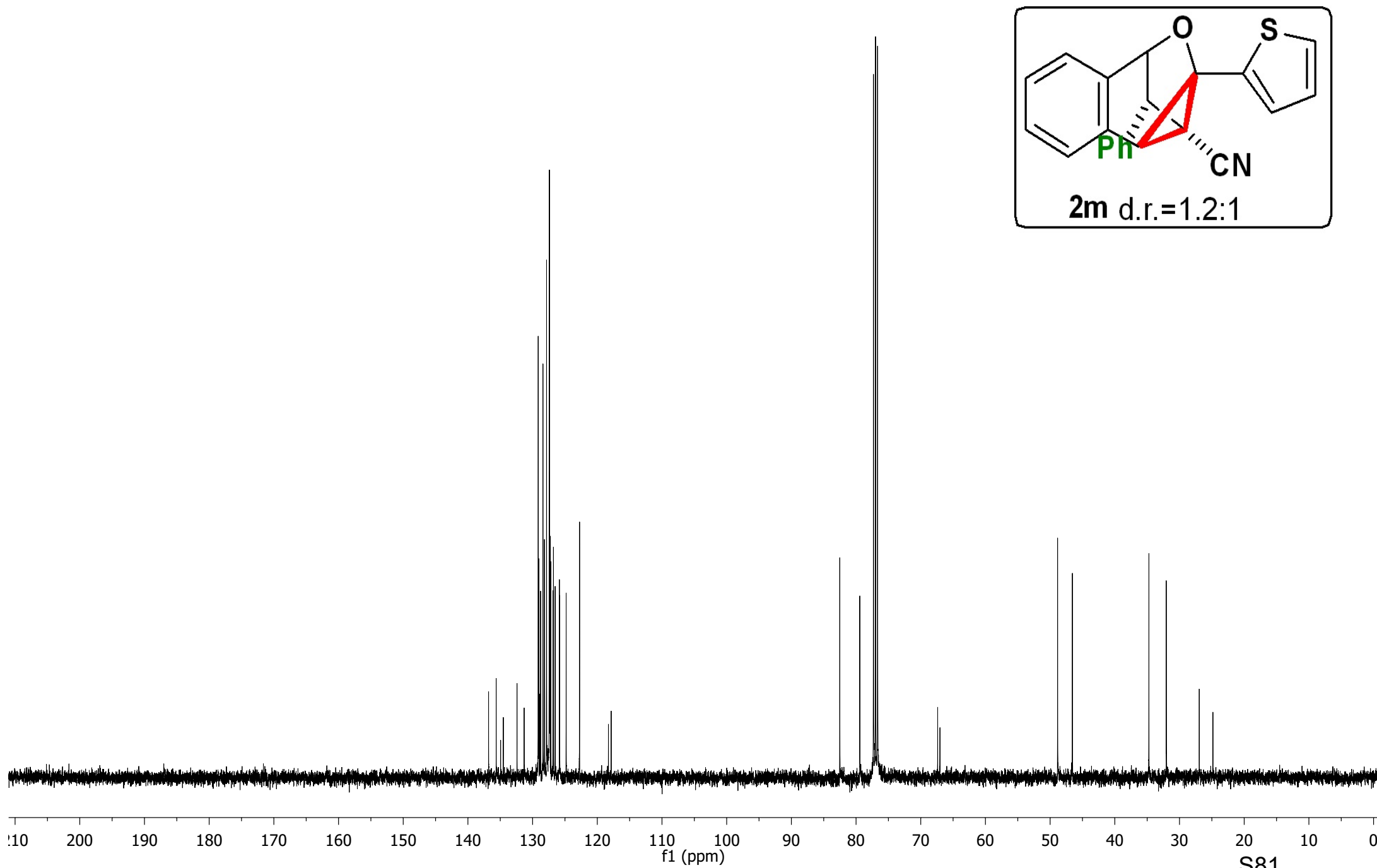
M0269-8
TPG-373

Solvent CDCl_3
Spectrometer Frequency 100.69

136.81
135.63
134.94
134.53
132.41
131.31
129.14
129.05
128.90
128.76
128.40
128.17
127.84
127.44
127.40
127.34
127.30
127.20
127.17
126.86
126.78
126.52
125.86
125.83
124.82
122.74
118.28
117.85

82.52
79.42
77.31
76.99
76.68
67.38
67.03

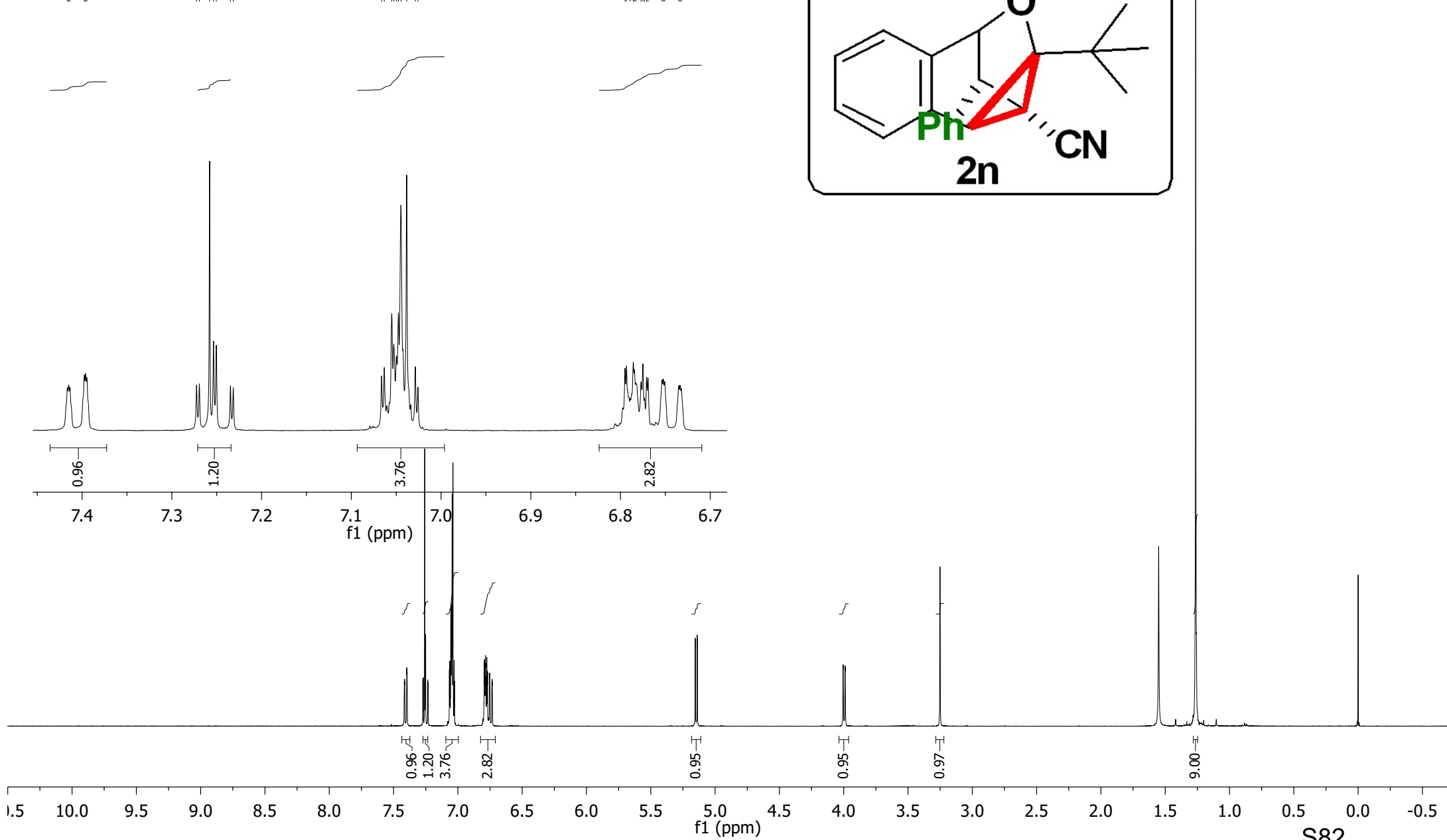
48.85
46.56
34.73
32.05
26.95
24.84



M0269-13
TPG-375

Solvent CDCl_3
Spectrometer Frequency 400.40

M0269-13
TPG-375



M0269-14
TPG-375
Solvent CDCl_3
Spectrometer Frequency 100.69

132.86
131.83
130.36
128.34
128.22
127.43
127.15
126.55
126.08
124.50
119.75

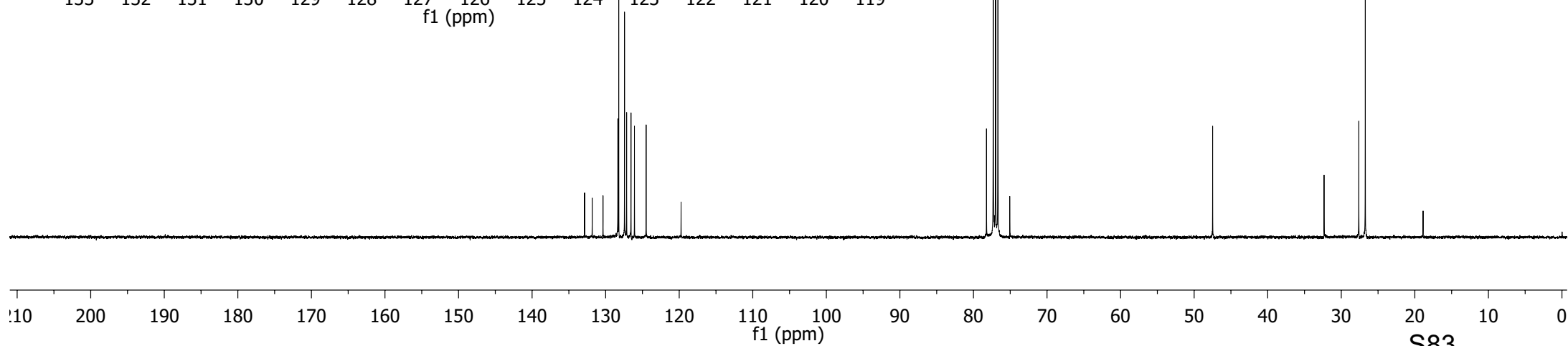
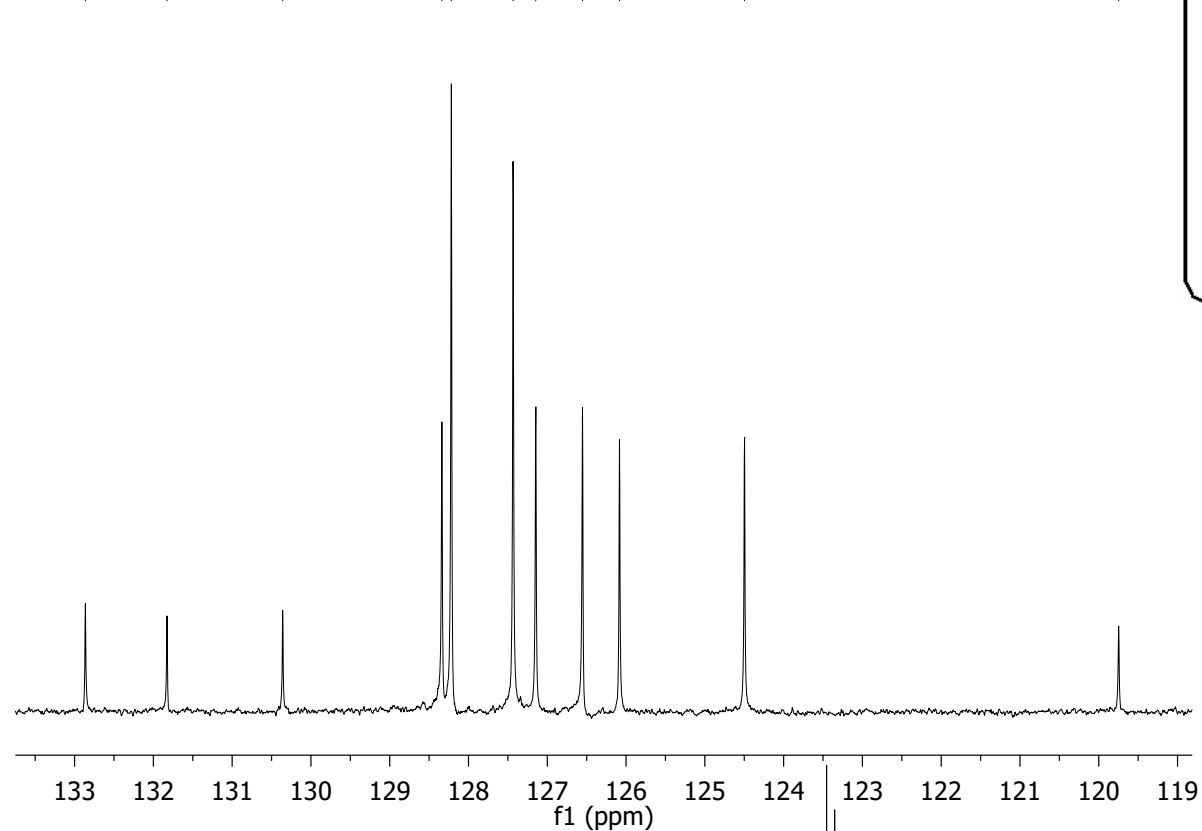
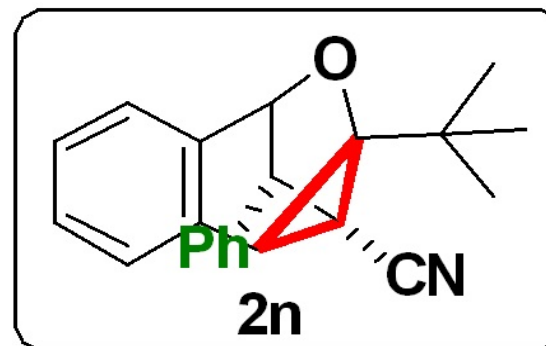
78.23
77.31
77.19
76.99
76.67
75.07

47.49

32.34
27.61
26.74

18.88

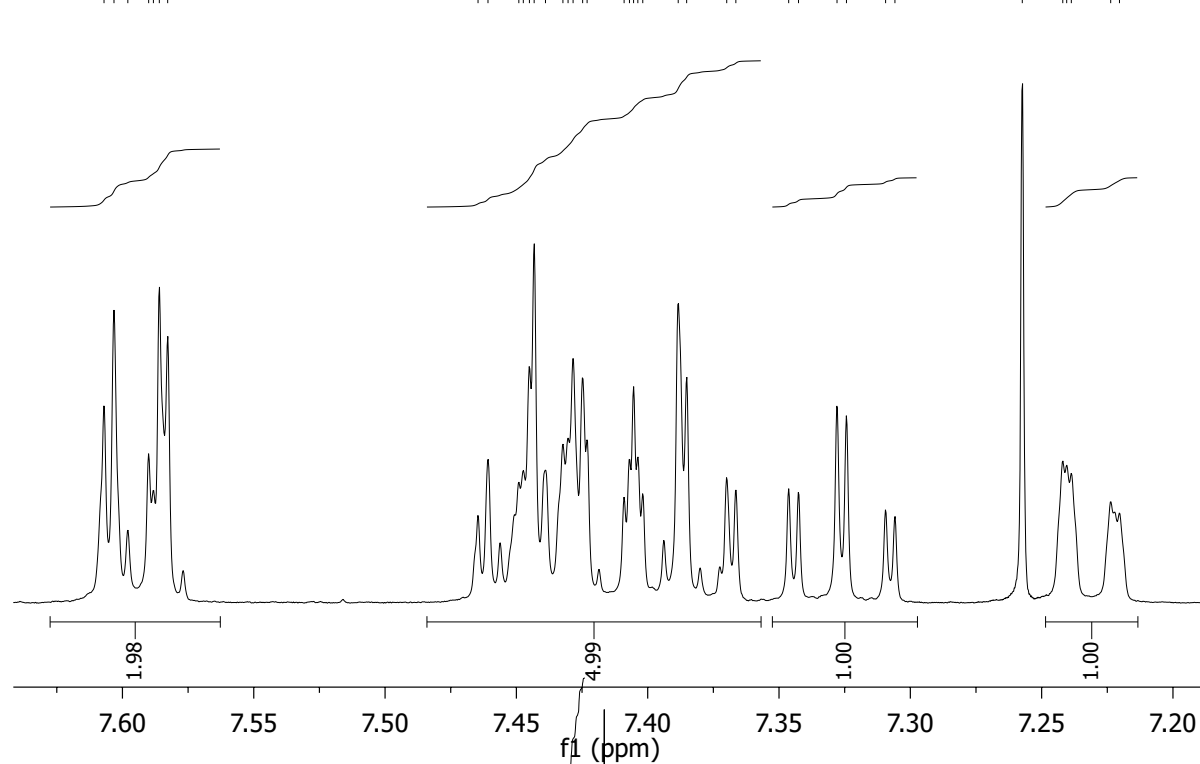
M0269-14
TPG-375
131.83
130.36
128.34
128.22
127.43
127.15
126.55
126.08
124.50
119.75



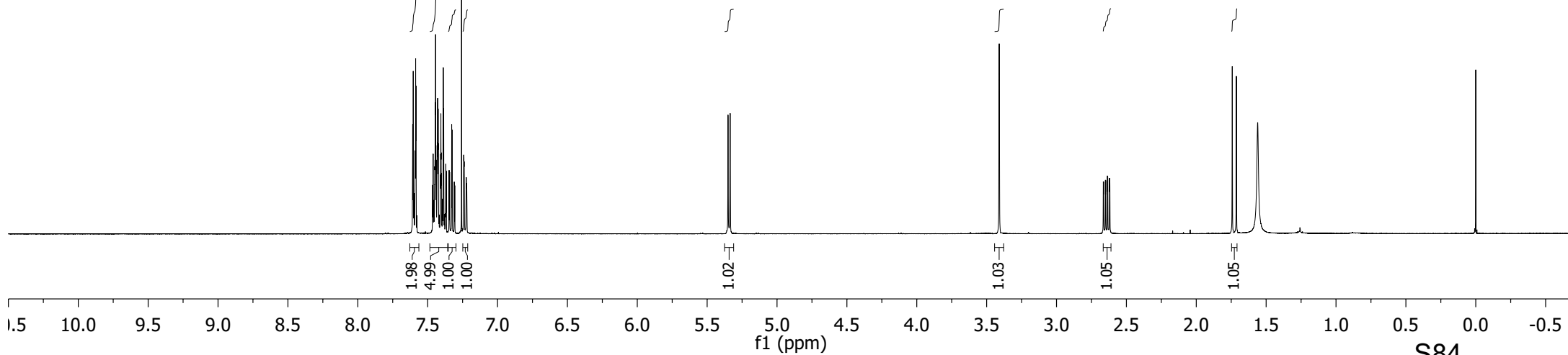
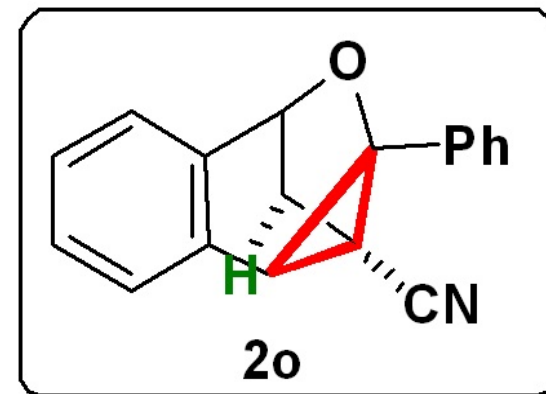
M025719
TPG-362

Solvent CDCl_3
Spectrometer Frequency 400.40

M025719
TPG-362



3.41 2.67 2.66 2.65 2.65 2.64 2.63 2.62 2.62 1.74 1.71 1.56 0.00



M0257-11
TPG-362 Solvent CDCl_3
Spectrometer Frequency 100.69

M0257-11
TPG-362

132.22

128.77
128.73
128.65
128.16

127.25
127.00
126.29

134.98
132.22
128.77
128.73
128.65
128.16
127.25
127.00
126.29
122.57
118.95

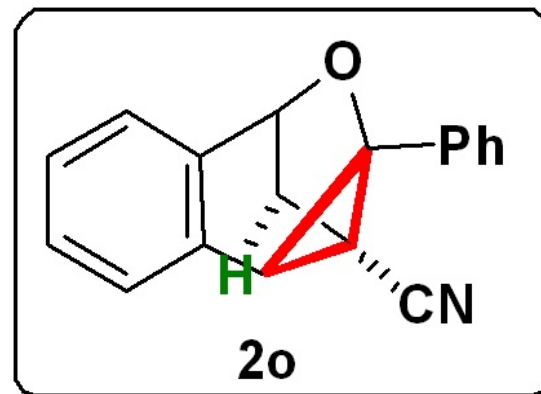
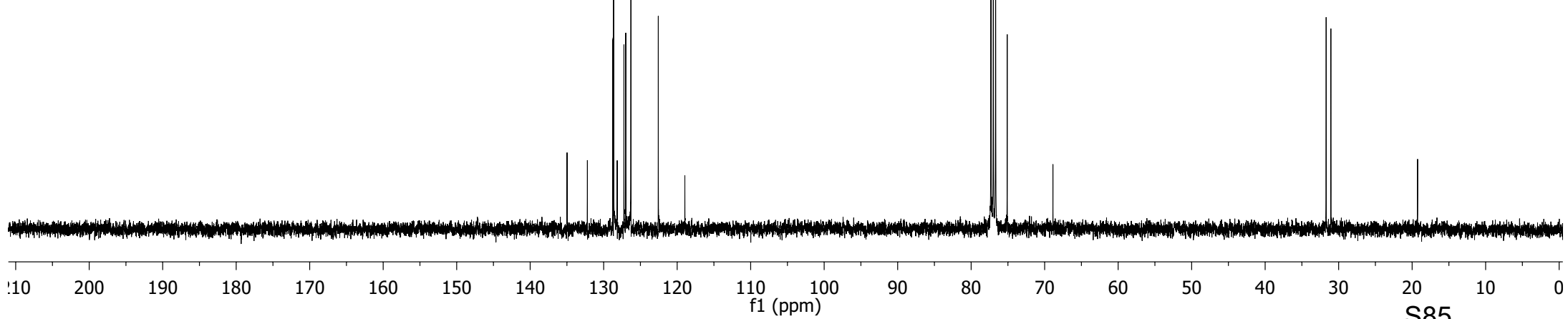
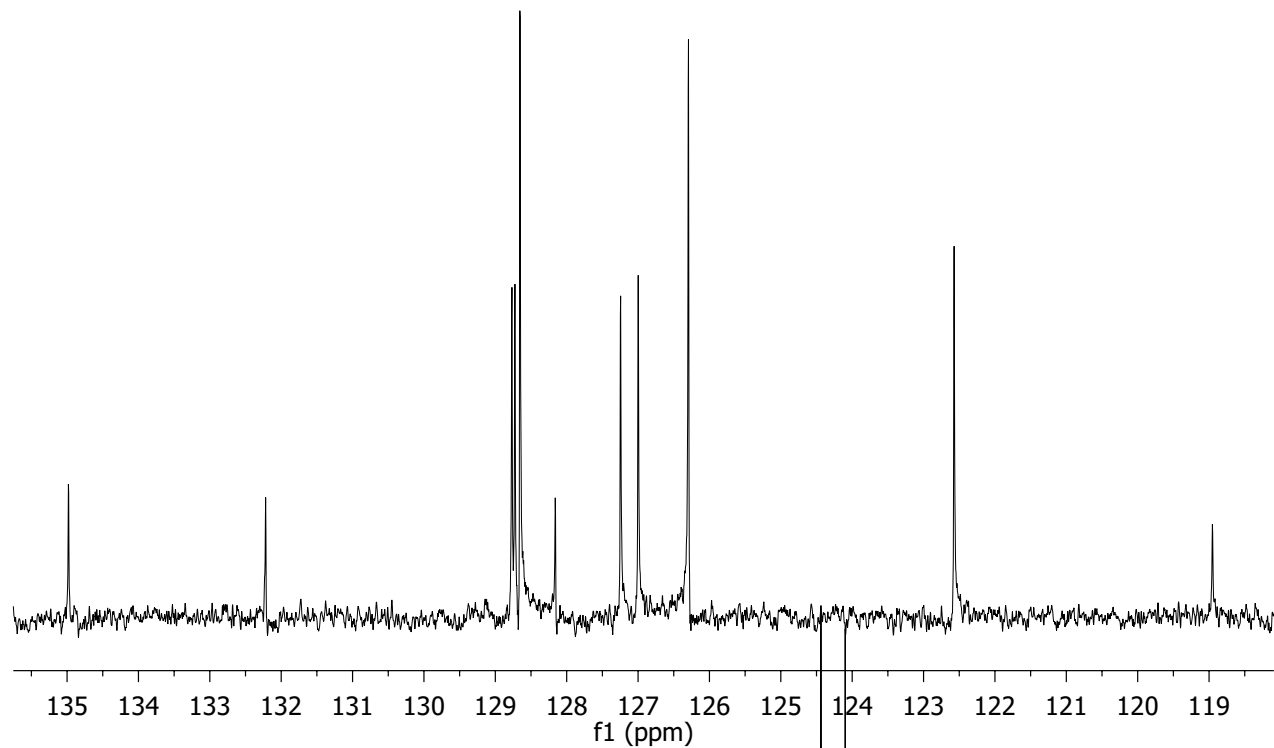
122.57

118.95

77.31
76.99
76.67
75.09
68.87

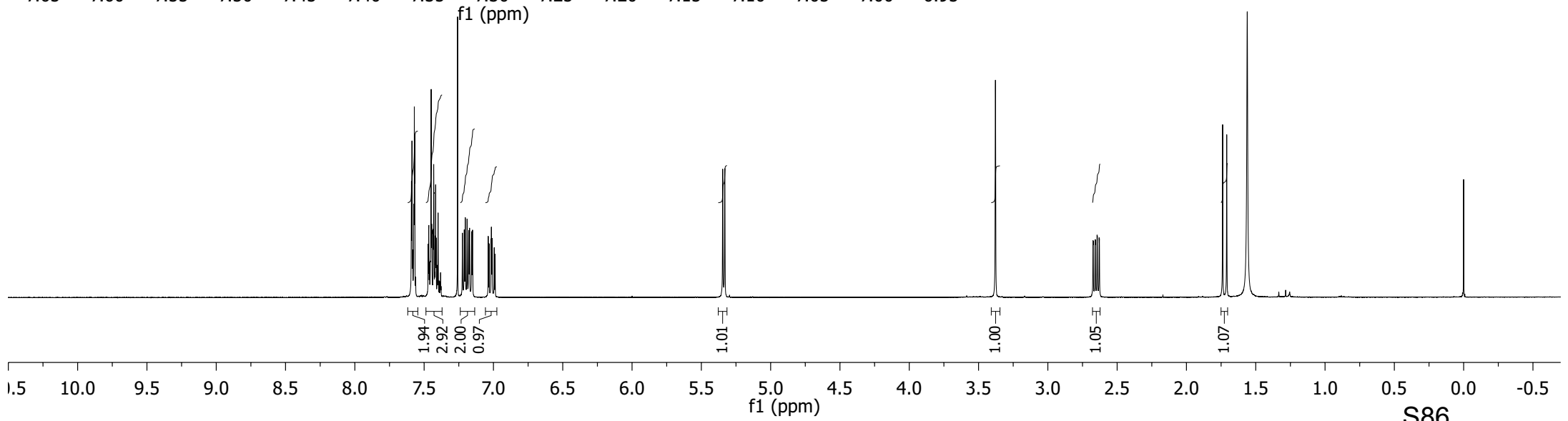
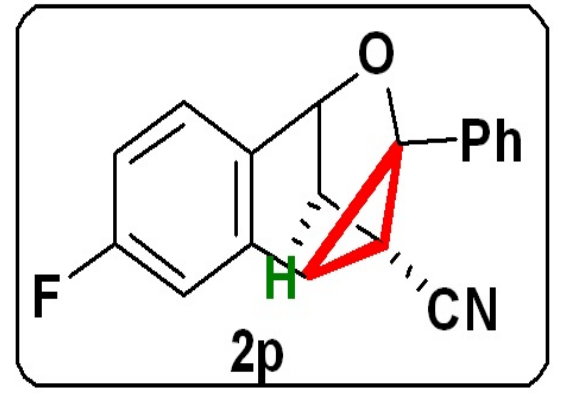
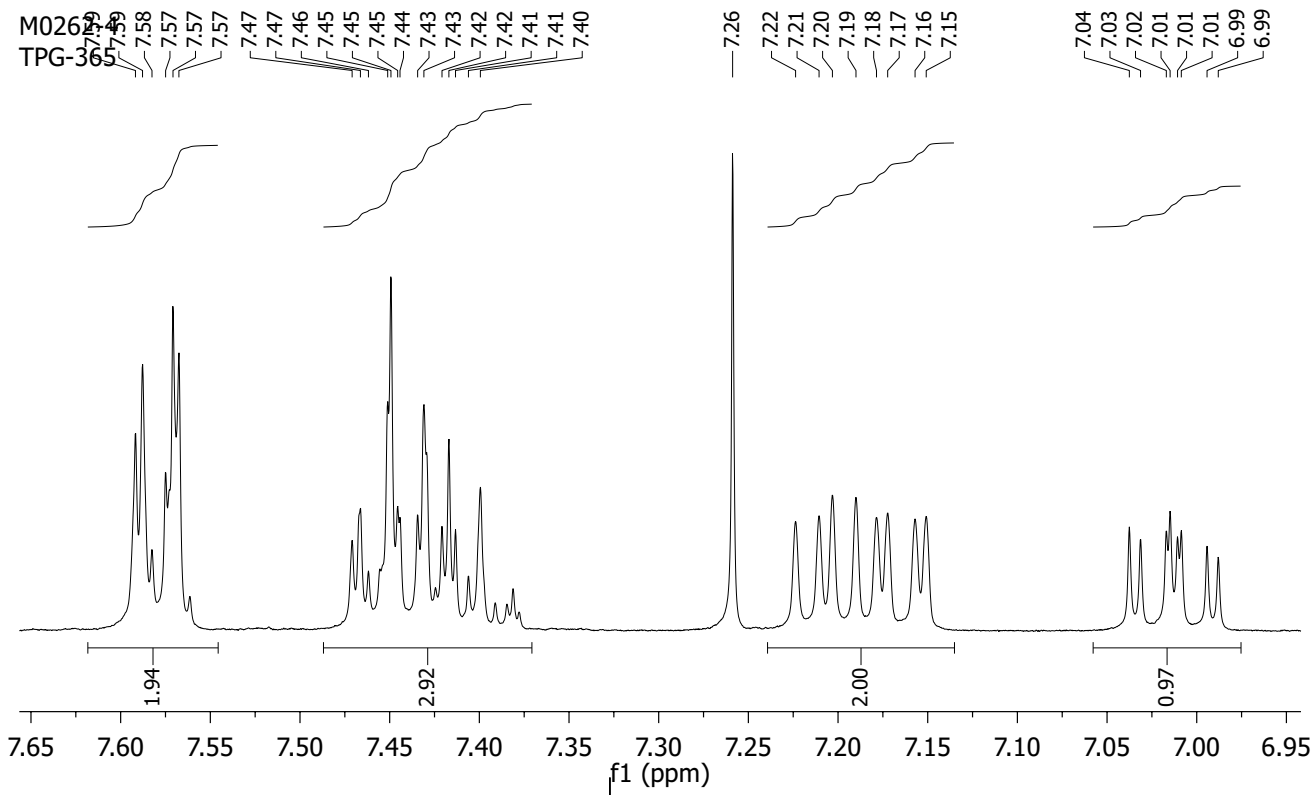
31.70
31.06

19.26



M0269
TPG-365
Solvent
Spectrometer Frequency 400.40

3.38
2.67
2.67
2.66
2.64
2.64
2.63
2.63
1.74
1.71
1.56
0.00



M0262-5
TPG-365

Solvent

CDCl₃

Spectrometer Frequency 100.69

—164.01
—161.56

—131.80
—130.89
—130.32
—130.24
—128.95
—128.73
—126.33
—124.38
—124.30
—118.56
—114.58
—114.35
—113.96
—113.75

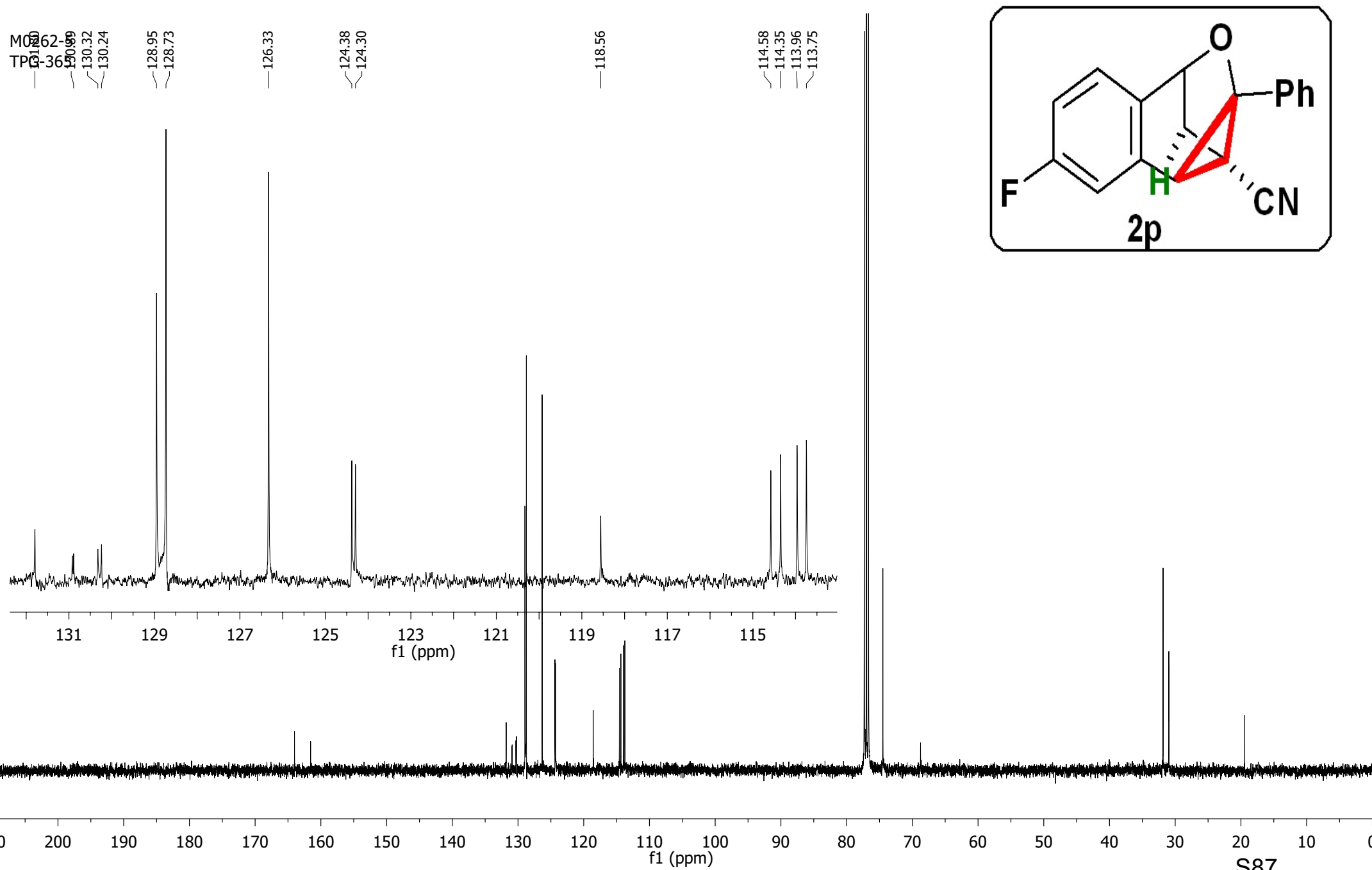
—77.31
—76.99
—76.68
—74.48
—68.75

—31.84
—30.97
—30.95

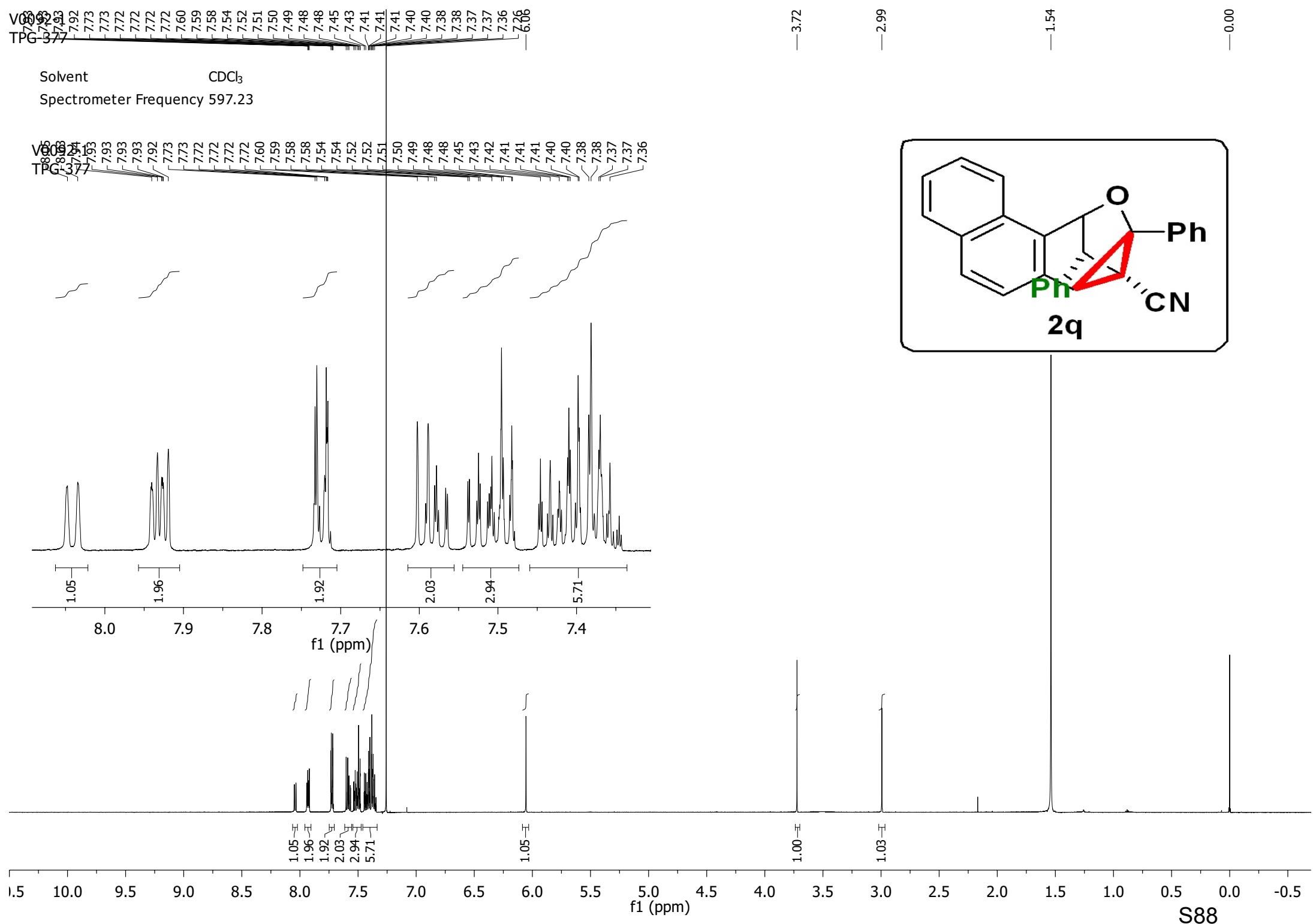
—19.42

M0262-5
TPG-365
—131.80
—130.89
—130.32
—130.24
—128.95
—128.73
—126.33
—124.38
—124.30
—118.56

—114.58
—114.35
—113.96
—113.75



S87



V0092-2
TPG-377

Solvent CDCl_3
Spectrometer Frequency 150.19

137.18
132.54
132.01
131.56
129.21
128.93
128.86
128.82
128.12
127.88
127.24
127.07
126.05
126.01
125.81
125.15
121.46
118.17

77.77
77.20
76.99
76.78
69.50

48.75
34.51
26.42

V0092-2
TPG-377

132.54
132.01
131.56

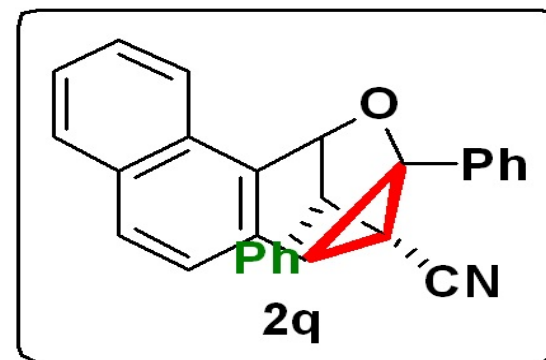
129.21
128.93
128.86
128.82
128.12
127.88
127.24
127.07
126.05
126.01
125.81
125.15

121.46

118.17

138 136 134 132 130 128 126 124 122 120 118
f1 (ppm)

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



M0267-76
TPG-369

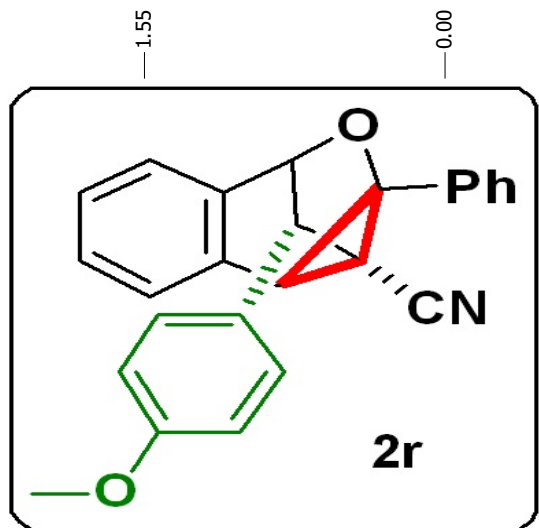
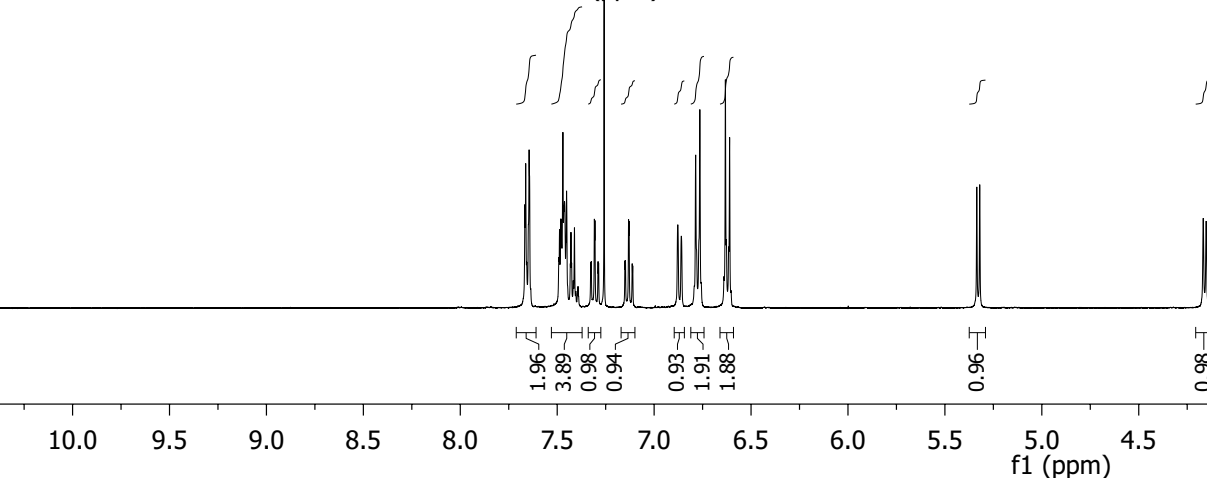
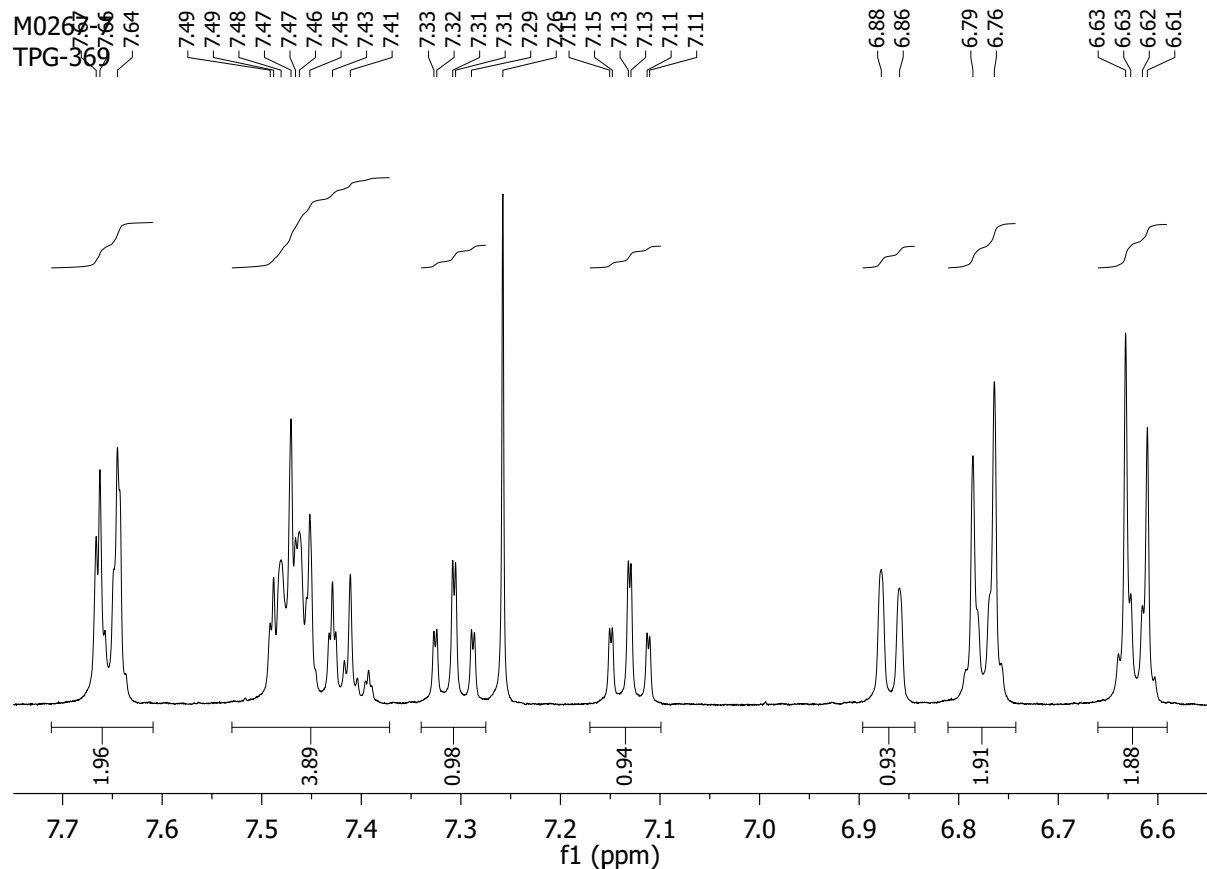
Solvent CDCl₃
Spectrometer Frequency 400.40

M0267-76
TPG-369

7.66 7.64 7.49 7.48 7.47 7.46 7.45 7.43 7.43 7.41 7.33 7.32 7.31 7.31 7.29 7.26 7.15 7.15 7.13 7.13 7.11 7.11 6.88 6.86 6.79 6.76 6.63 6.63 6.62 6.61 5.34 5.32

4.17 4.15

3.68 3.56



M0267-8
TPG-369

Solvent

CDCl_3

Spectrometer Frequency 100.69

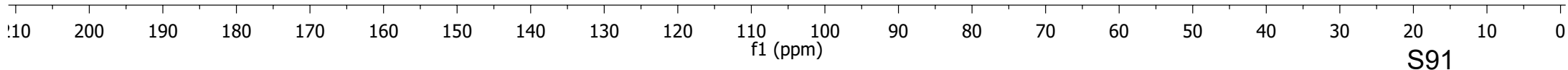
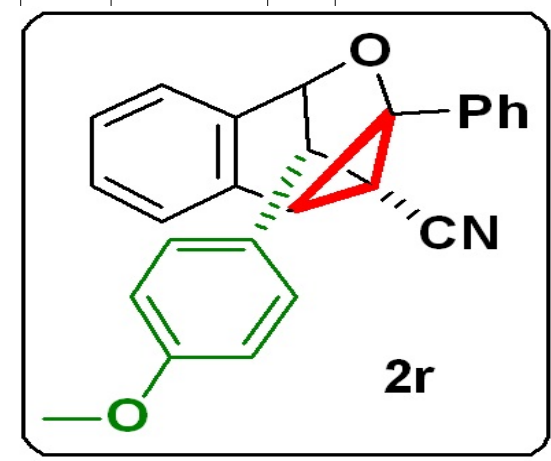
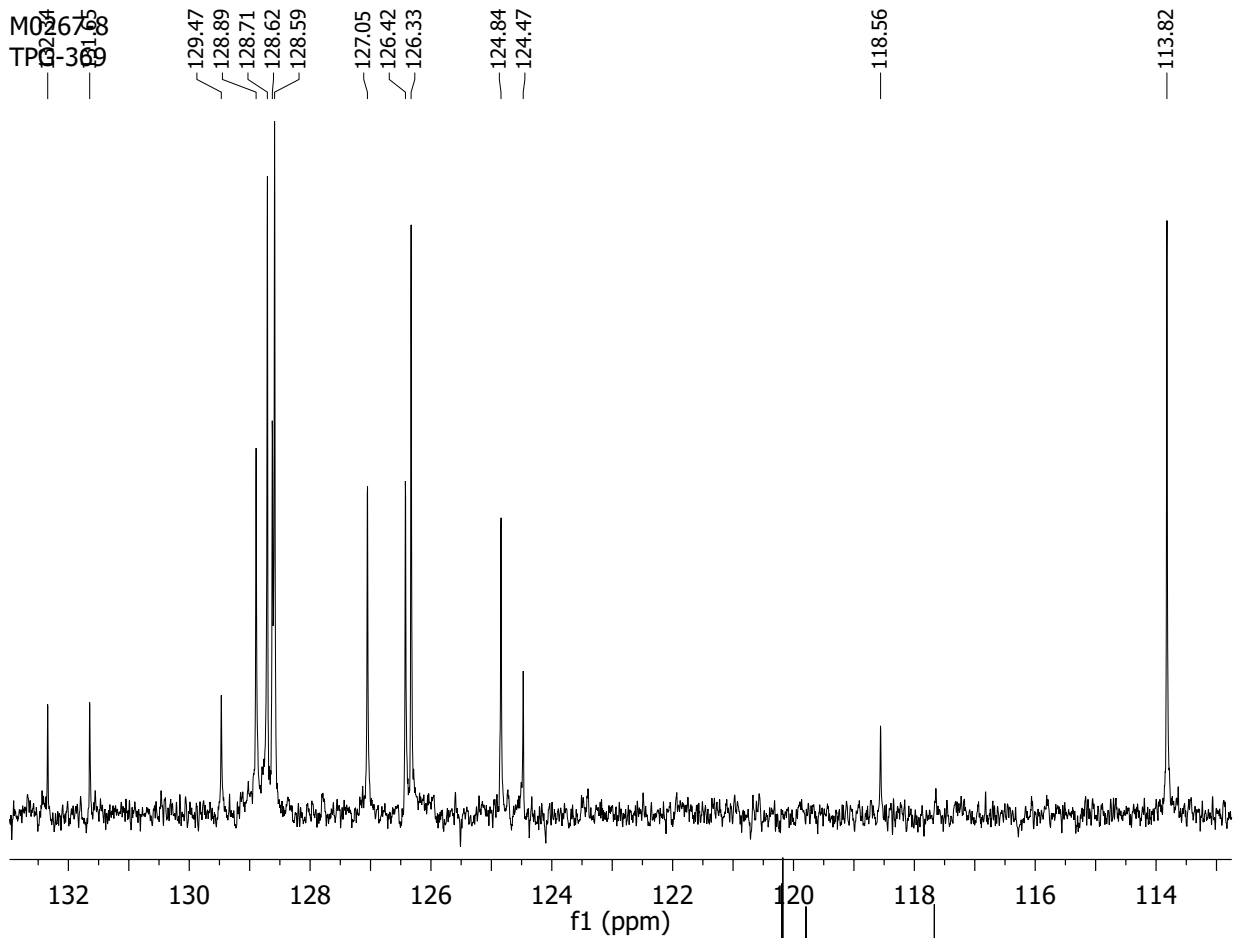
158.69

132.34
131.65
129.47
128.89
128.71
128.62
128.59
127.05
126.42
126.33
124.84
124.47
118.56

113.82

79.09
77.31
76.99
76.67
69.33

55.08
46.27
31.00
24.41

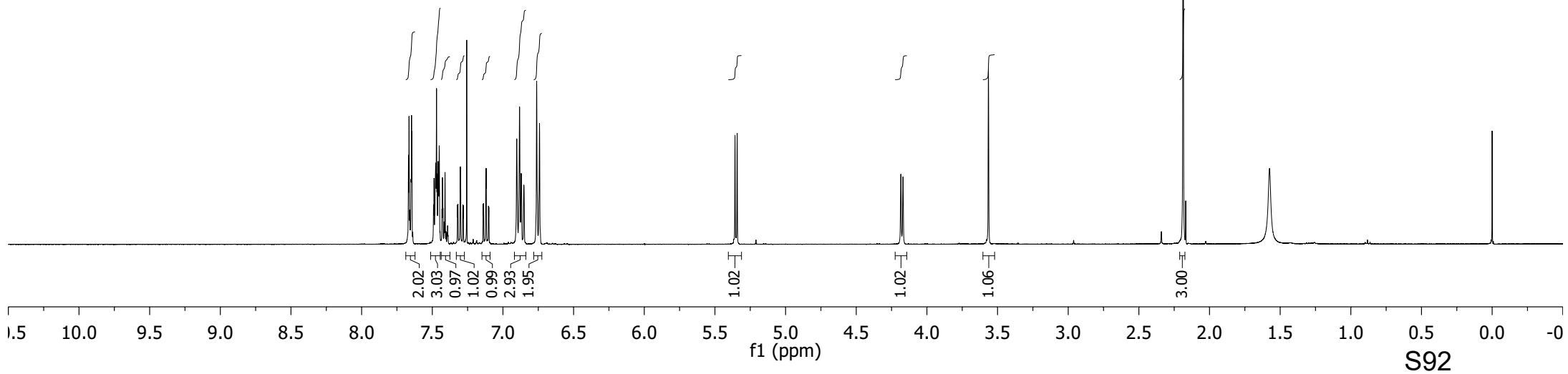
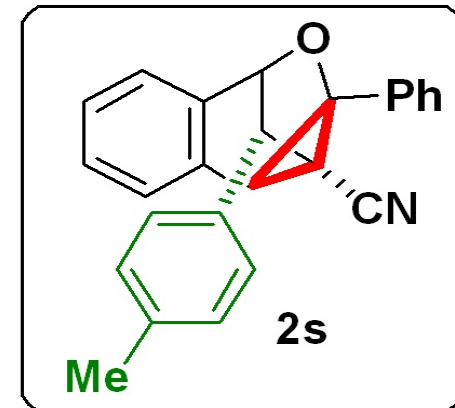
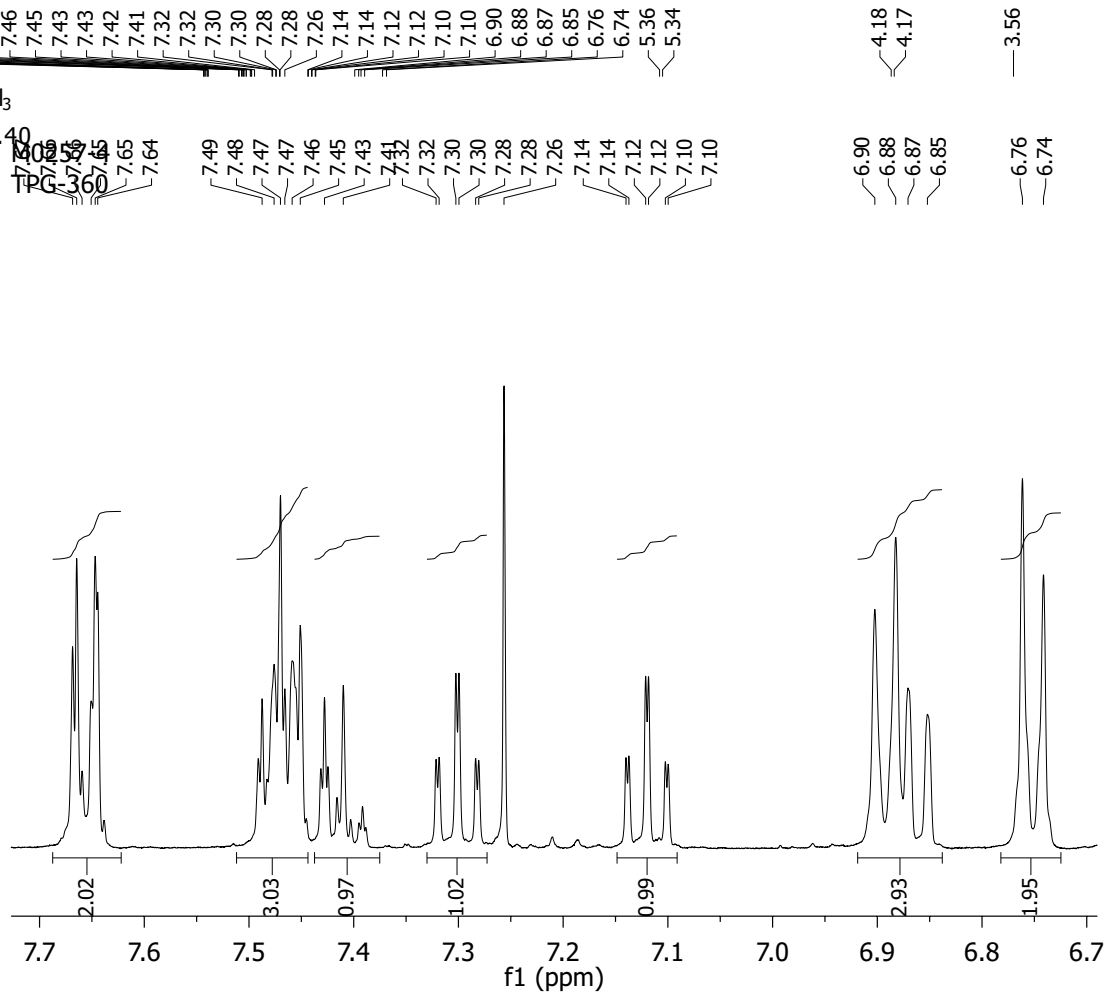


M02574
TPG-360

Solvent

CDCl₃

Spectrometer Frequency 400.40



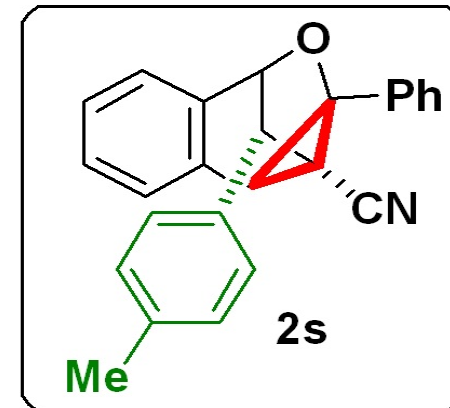
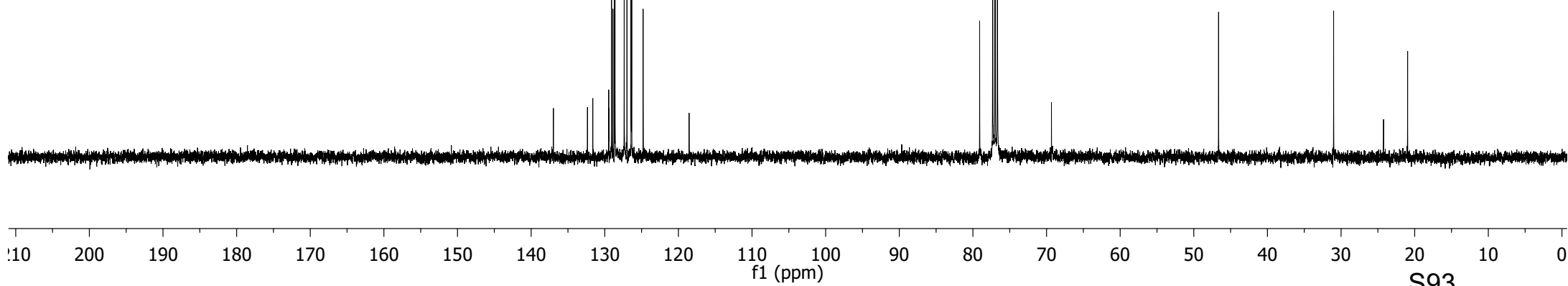
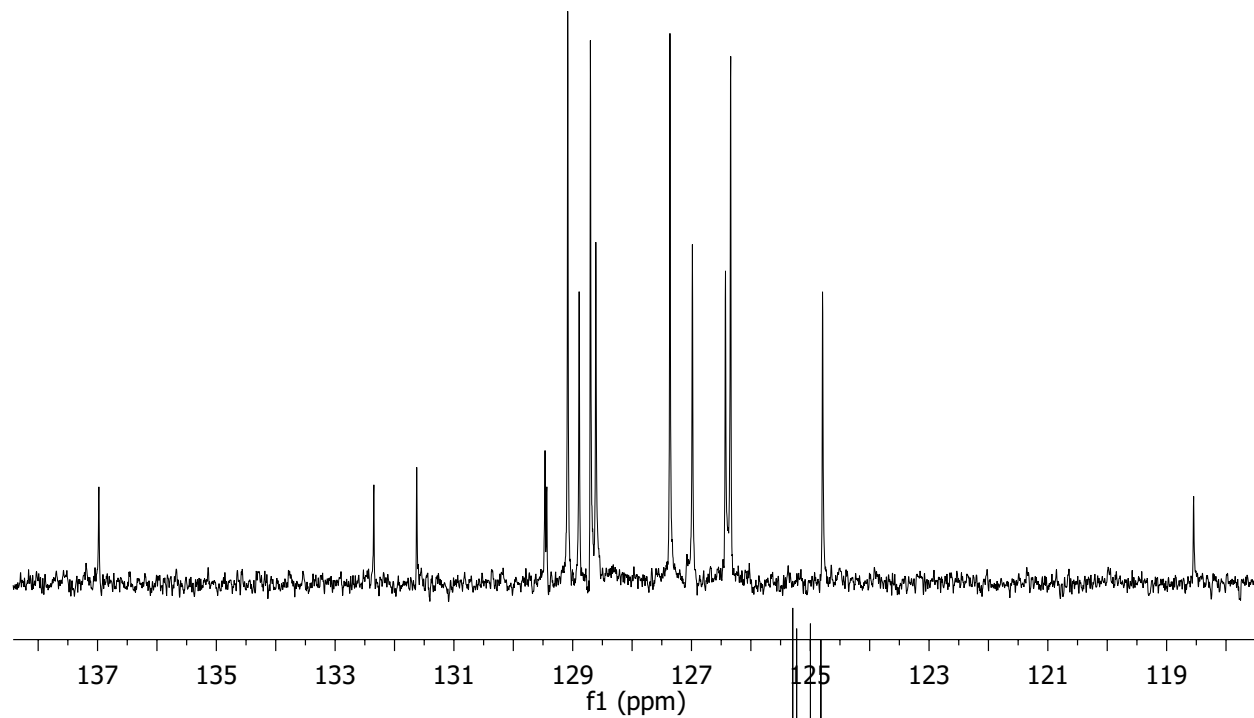
M0257-5
TPG-360 Solvent CDCl_3
Spectrometer Frequency 100.69

136.98
132.35
131.63
129.47
129.44
129.08
128.89
128.70
128.61
127.36
126.98
126.43
126.34
124.79
118.55

79.09
77.31
76.99
76.68
69.33

46.63
31.01
24.23
20.96

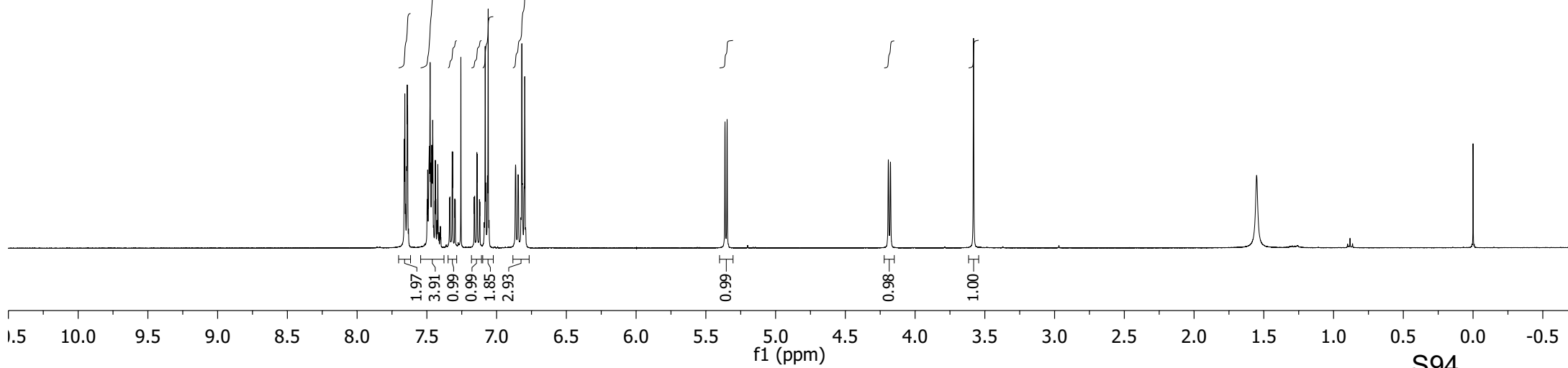
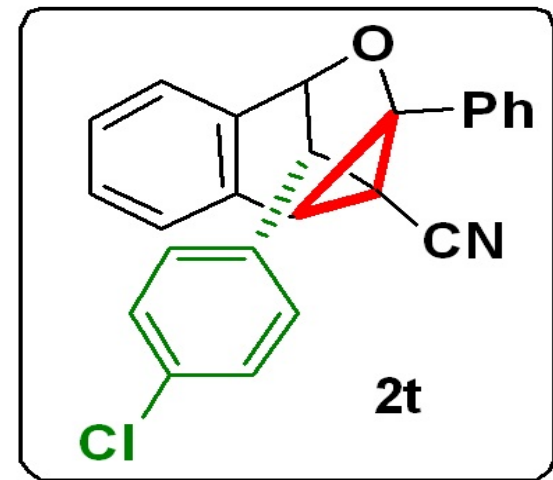
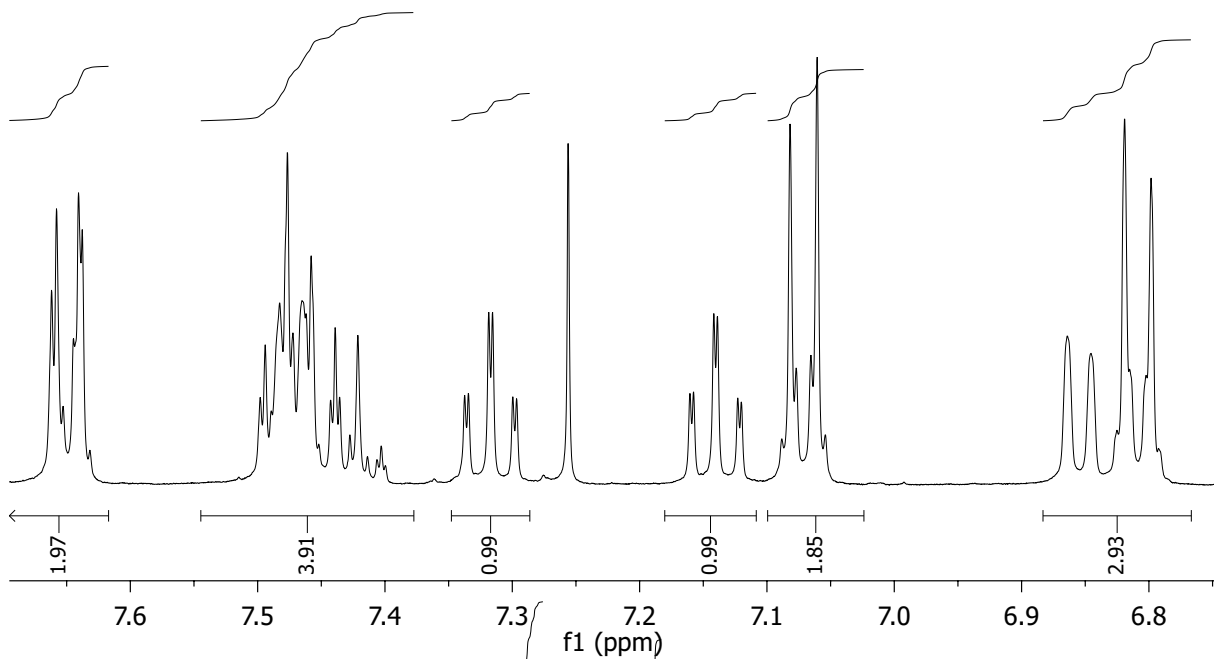
M0257-5
TPG-360
132.35
131.63
129.47
129.44
129.08
128.89
128.70
128.61
127.36
126.98
126.43
126.34
124.79
118.55



M 2694
TPG-372

Solvent CDCl_3
Spectrometer Frequency 400.40

7.56
7.50
7.48
7.47
7.46
7.46
7.44
7.44
7.44
7.42
7.34
7.33
7.32
7.32
7.30
7.30
7.26
7.16
7.16
7.14
7.14
7.12
7.12
7.08
7.08
7.07
7.06
6.86
6.85
6.82
6.82
5.86
5.35
4.19
4.18
3.58
1.55
0.00



M0269-5
TPG-372

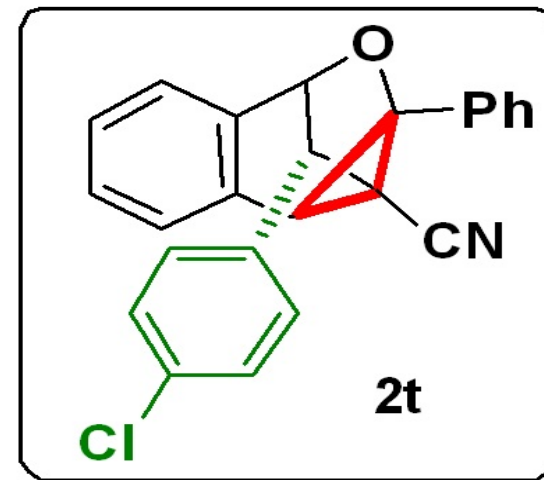
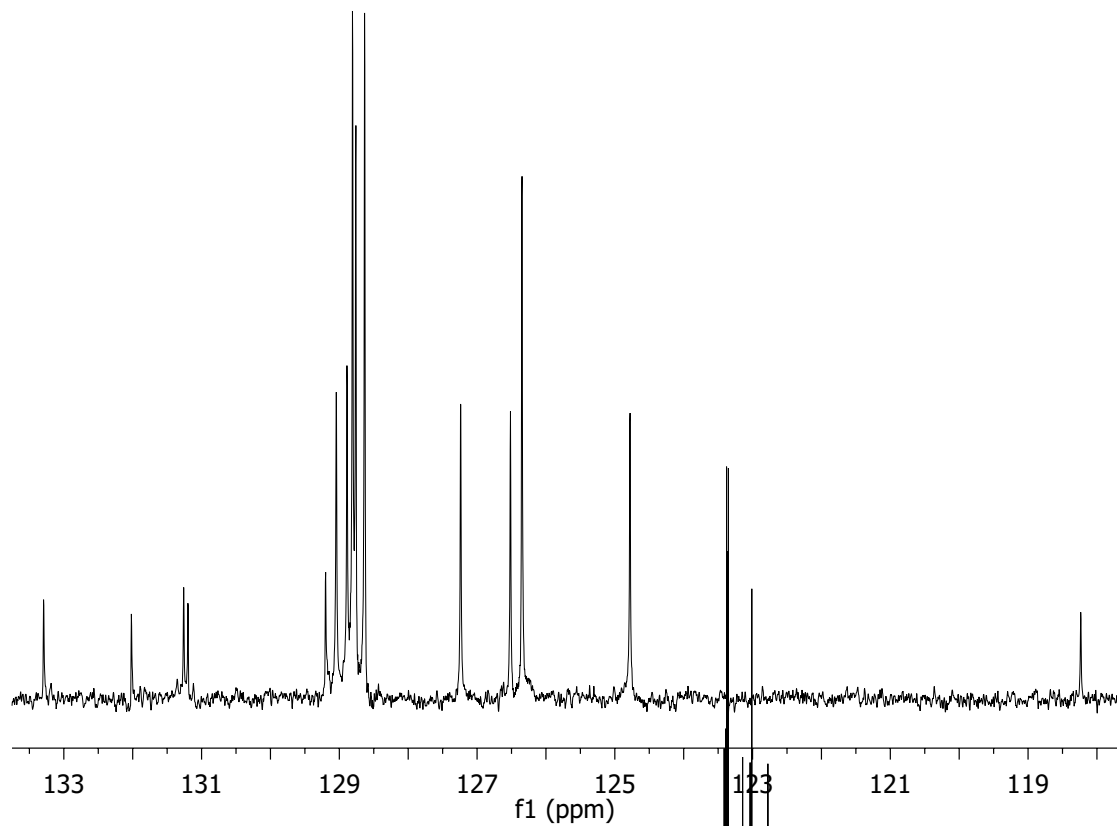
Solvent CDCl₃
Spectrometer Frequency 100.69

133.29
132.02
131.26
131.20
129.20
129.04
128.89
128.81
128.76
128.63
127.24
126.52
126.35
124.78
118.23

78.90
77.31
76.99
76.67
69.53

46.42
30.98
24.02

M0269-5
TPG-372
132.02
131.26
131.20
129.20
129.04
128.89
128.81
128.76
128.63
127.24
126.52
126.35
124.78
118.23



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

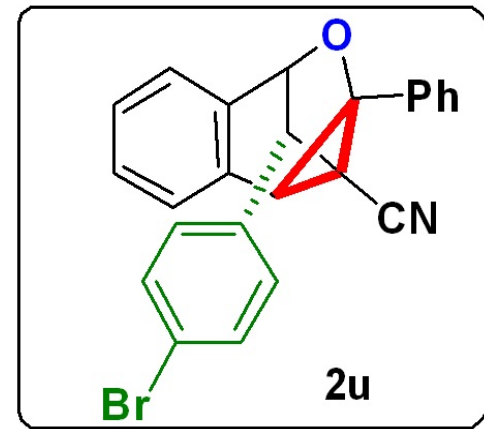
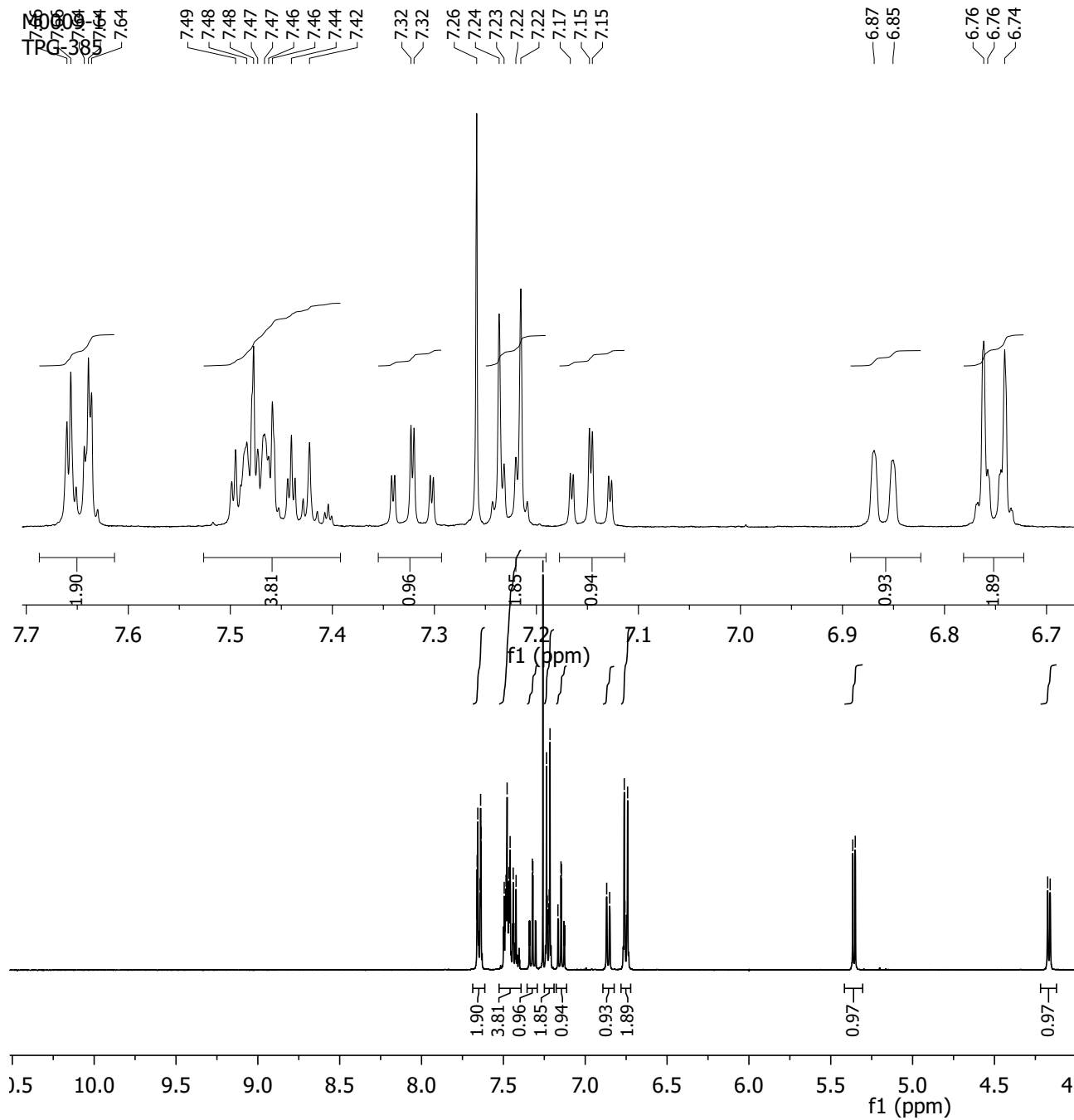
M0009-4
TPG-385

Solvent CDCl₃
Spectrometer Frequency 400.40

7.766
7.764
7.764
7.749
7.748
7.747
7.747
7.746
7.746
7.744
7.742
7.732
7.732
7.726
7.724
7.723
7.722
7.722
7.717
7.715
7.715
7.687
7.685
7.676
7.676
7.674

5.37
5.35
4.17
4.16
3.58

1.55
0.00



M0009-2
TPG-385

Solvent CDCl₃
Spectrometer Frequency 100.69

M0009-2
TPG-385

129.15
129.05
128.91
128.76

127.27
126.51
126.35

124.79

121.43

118.21

132.00
131.79
131.58
131.17
129.15
129.05
128.91
128.76
127.27
126.51
126.35
124.79
121.43
118.21
109.89

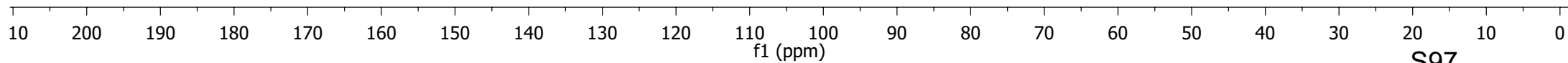
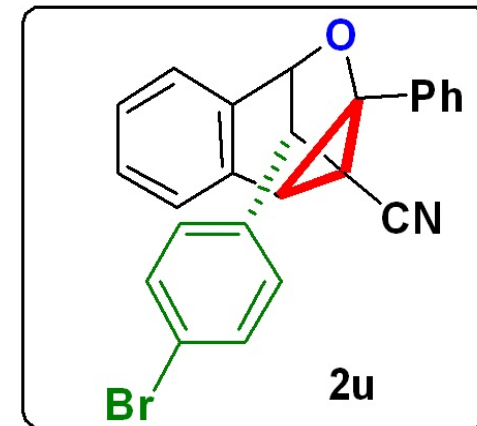
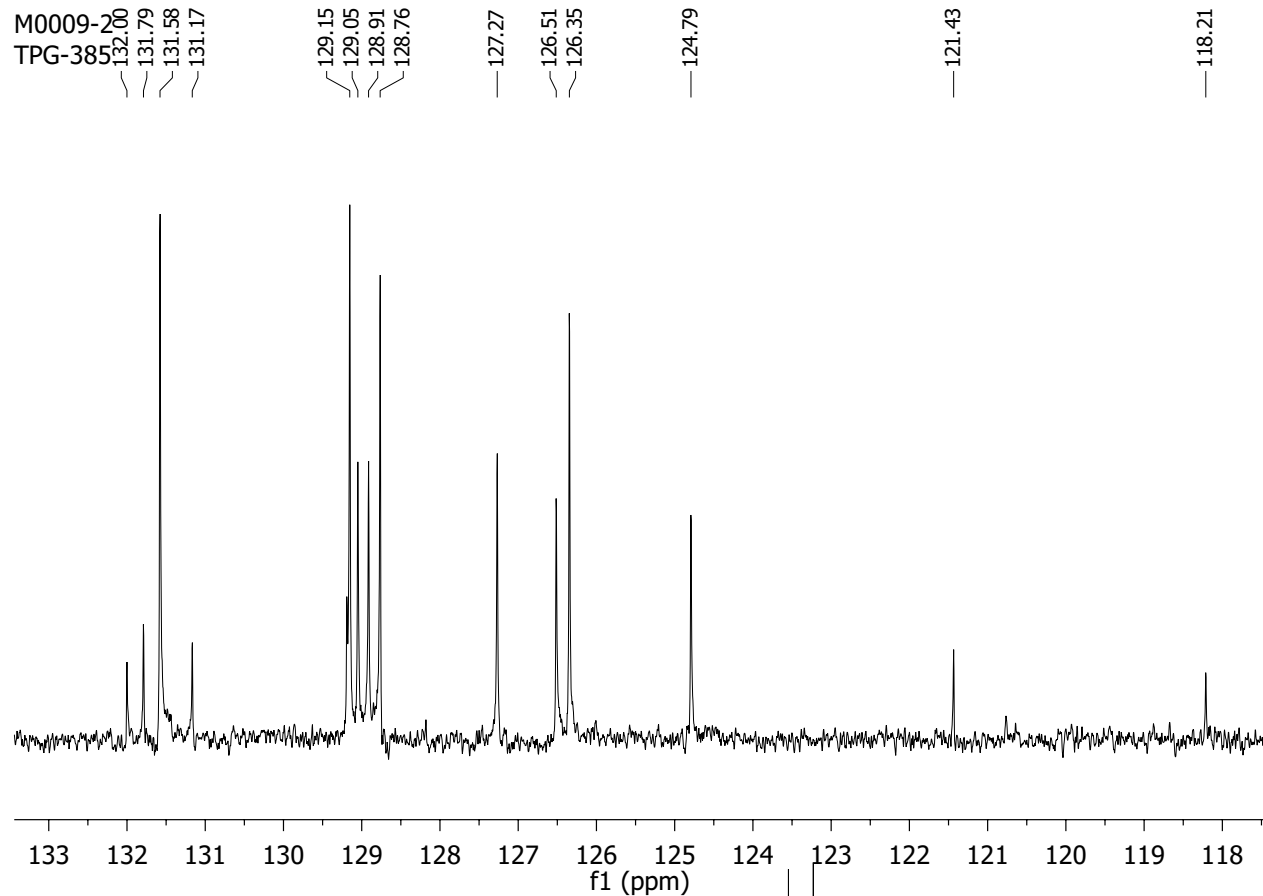
78.83
77.31
76.99
76.68

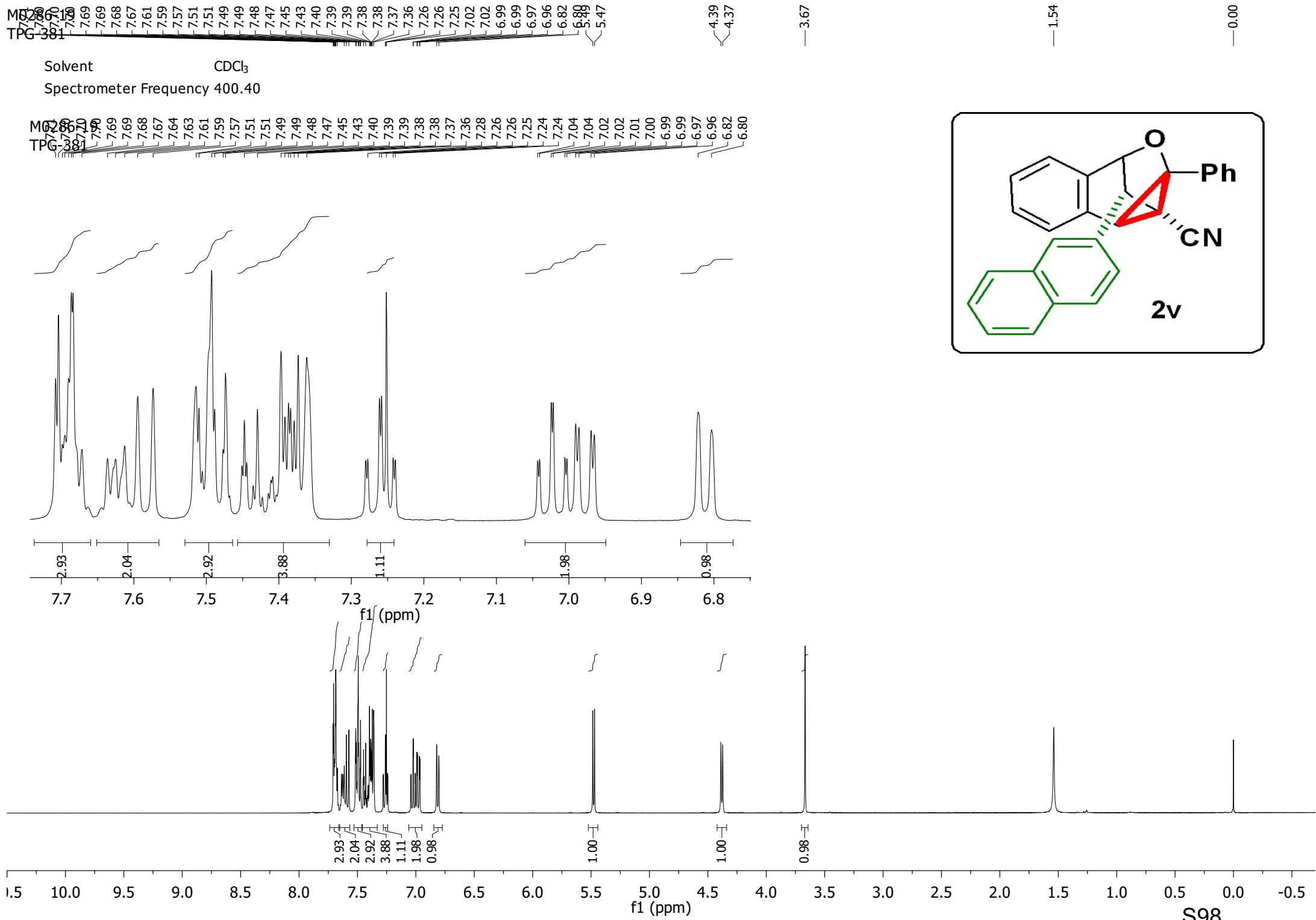
69.29

46.49

30.98

23.98





M0286-20
TPG-381

Solvent CDCl_3
Spectrometer Frequency 100.69

133.07
132.44
132.26
131.46
130.21
129.39
128.98
128.75
128.73
128.14
127.66
127.46
127.10
126.62
126.45
126.39
126.12
125.92
125.24
124.74
118.54

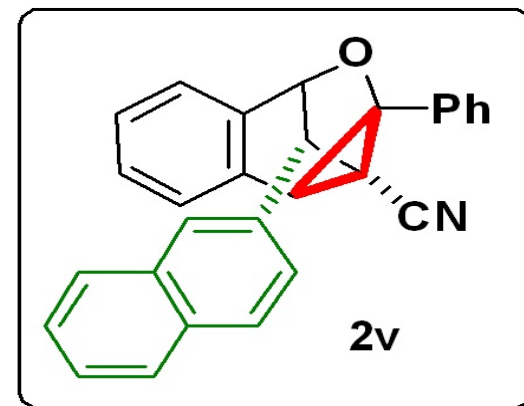
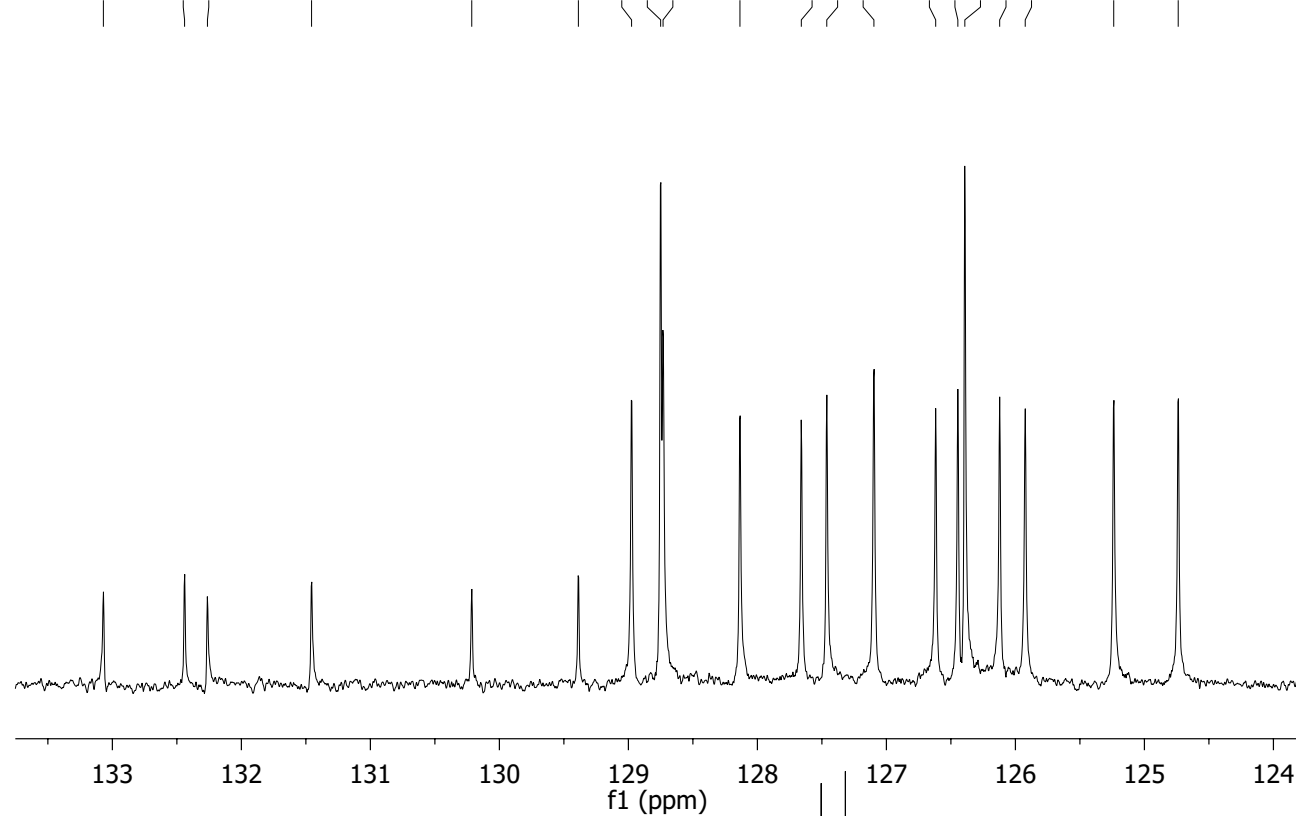
79.13
77.31
76.99
76.67
69.55

47.12

31.14

24.18

M0286-20
TPG-381
133.20
132.44
132.26
131.46
130.21
129.39
128.98
128.75
128.73
128.14
127.66
127.46
127.10
126.62
126.45
126.39
126.12
125.92
125.24
124.74



200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

S99

M0262-7
TPG-366

Solvent

CDCl₃

Spectrometer Frequency 400.40

M0262-7
TPG-366

7.59
7.58
7.57
7.56
7.42
7.42
7.42
7.41
7.40
7.40
7.40
7.39
7.34
7.33
7.26
7.23
7.22
7.06
7.05
7.05
7.04
7.04
6.99
6.98
6.81
6.80
6.79
6.78
5.20
5.19

3.66
3.65

2.80

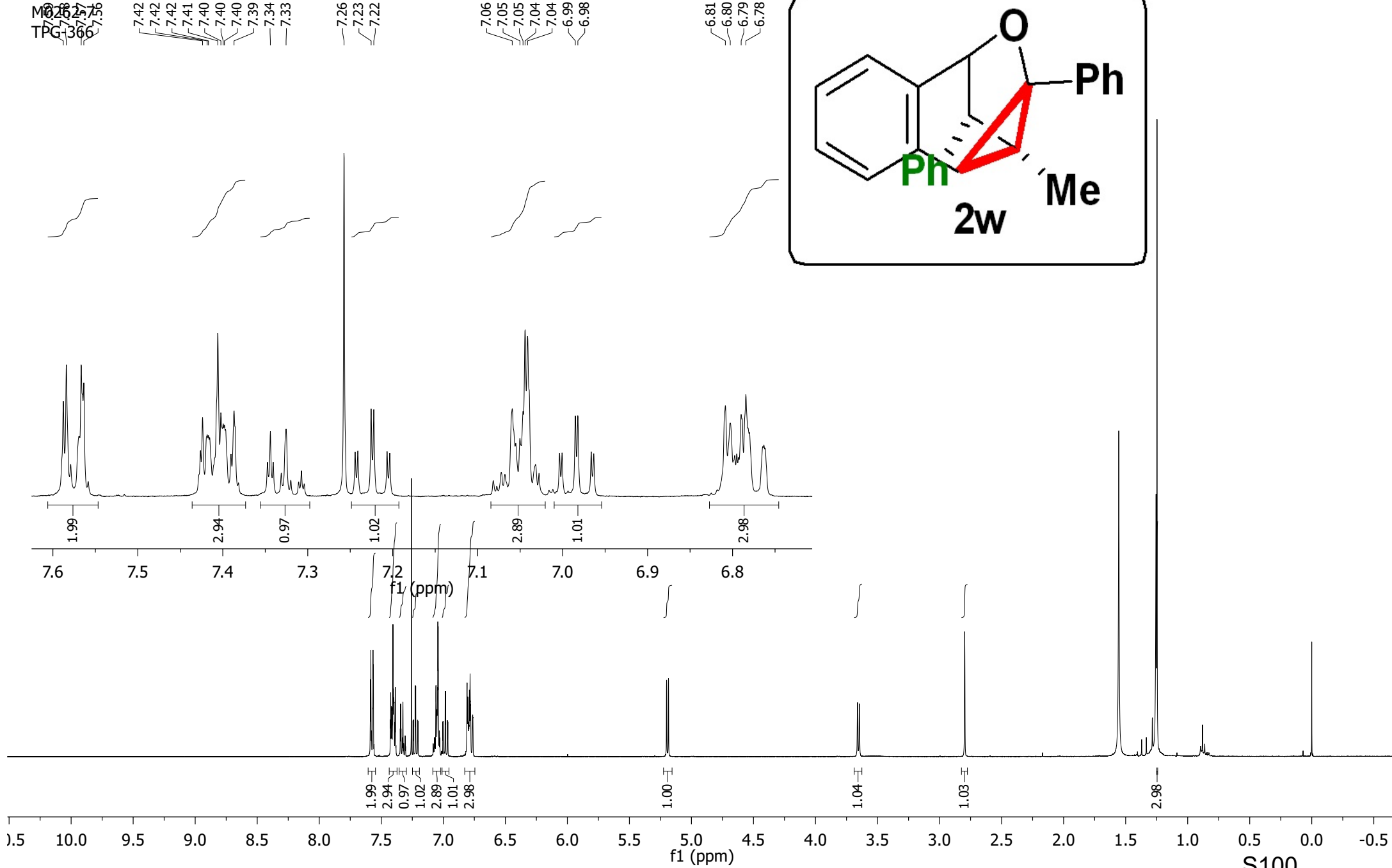
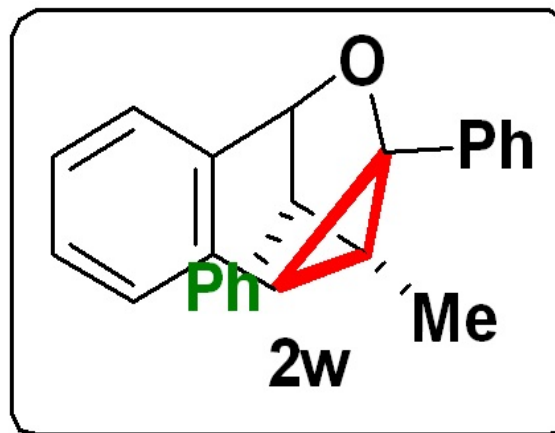
1.56

1.25

-0.00

7.42
7.42
7.41
7.40
7.40
7.40
7.39
7.34
7.33
7.26
7.23
7.22
7.06
7.05
7.05
7.04
7.04
6.99
6.98
6.81
6.80
6.79
6.78

6.81
6.80
6.79
6.78



S100

M0262-8
TPG-366

Solvent

CDCl_3

Spectrometer Frequency 100.69

135.65
135.22
134.37
131.76
128.41
128.24
127.90
127.68
127.64
126.40
125.63
124.94
124.35

79.30
77.31
76.99
76.68

69.06

49.23

29.69
29.29

13.78

M0262-8
TPG-366

134.37

131.76

128.41

128.24

127.90

127.68

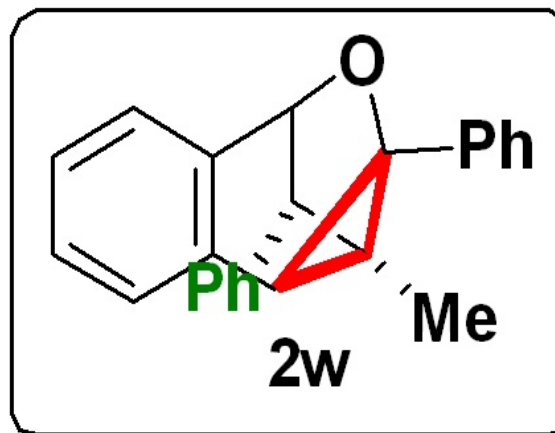
127.64

126.40

125.63

124.94

124.35



136 135 134 133 132 131 130 129 128 127 126 125 124

f1 (ppm)

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

S101

U0311-4

TPG-383

Solvent

Spectrometer Frequency 400.28

CDCl₃

7.57
7.55
7.46
7.45
7.43
7.41
7.39
7.38
7.37
7.36
7.35
7.34
7.33
7.32
7.29
7.27
7.25

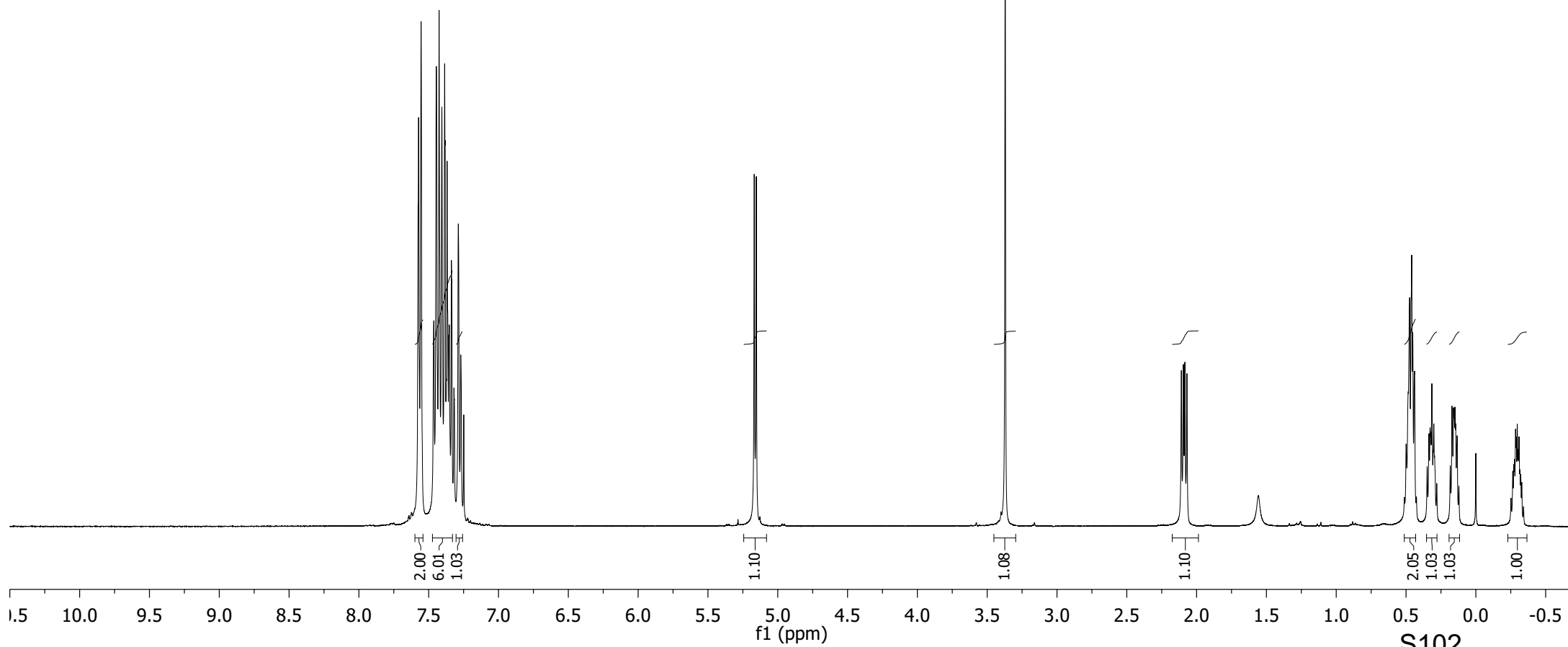
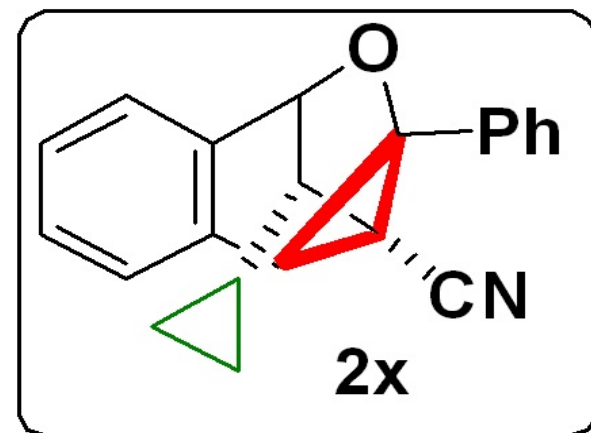
5.17
5.15

3.37

2.11
2.09
2.08
2.07

1.56

0.47
0.46
0.45
0.44
0.34
0.33
0.32
0.31
0.30
0.17
0.16
0.16
0.15
0.14
-0.28
-0.20



S102

U0311-6-1

Solvent

CDCl_3

Spectrometer Frequency 100.69

132.84
132.48
129.26
128.66
128.59
126.90
126.88
126.13
124.39
118.88

77.80
77.31
76.99
76.67
69.59

47.87

30.72

24.09

6.58
3.63
2.34

U0311-6-1

132.84
132.48

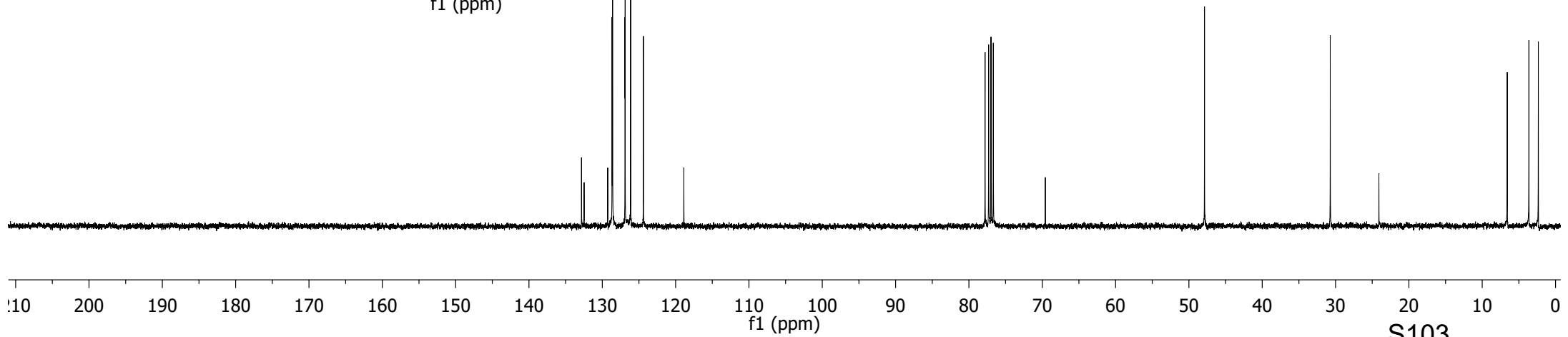
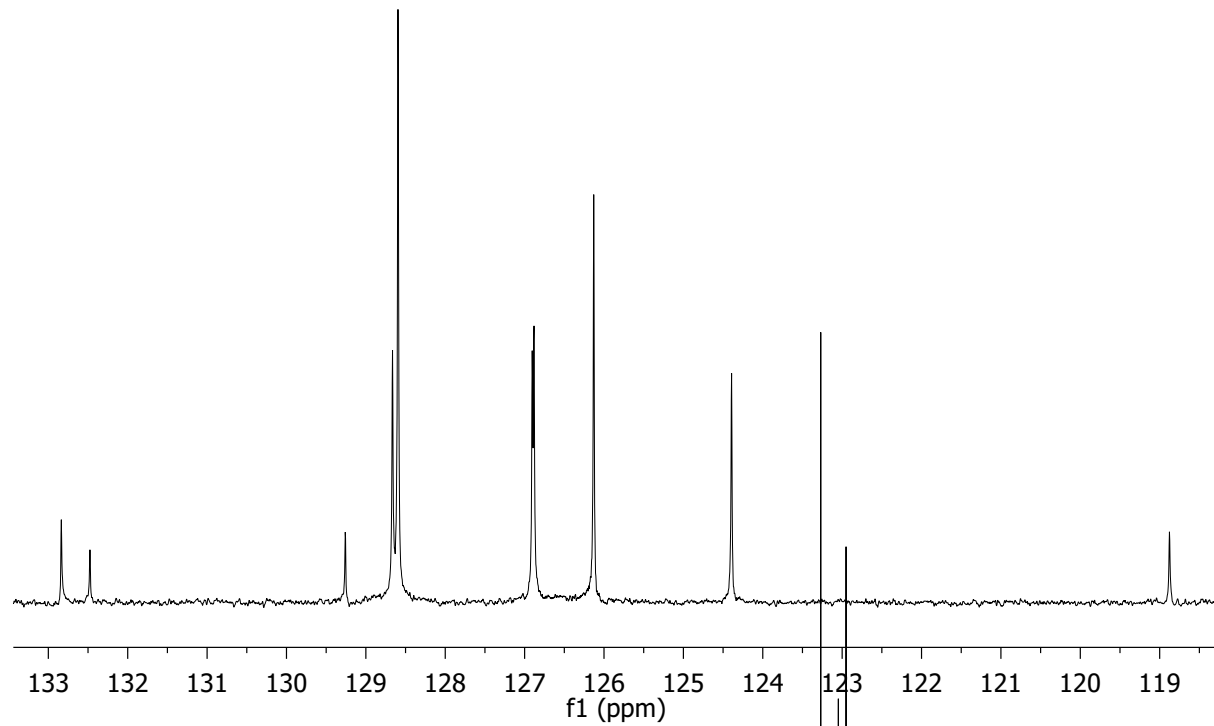
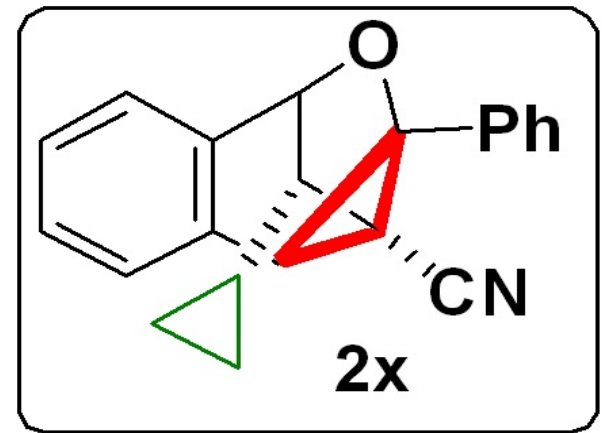
129.26
128.66
128.59

126.90
126.88

126.13

124.39

118.88



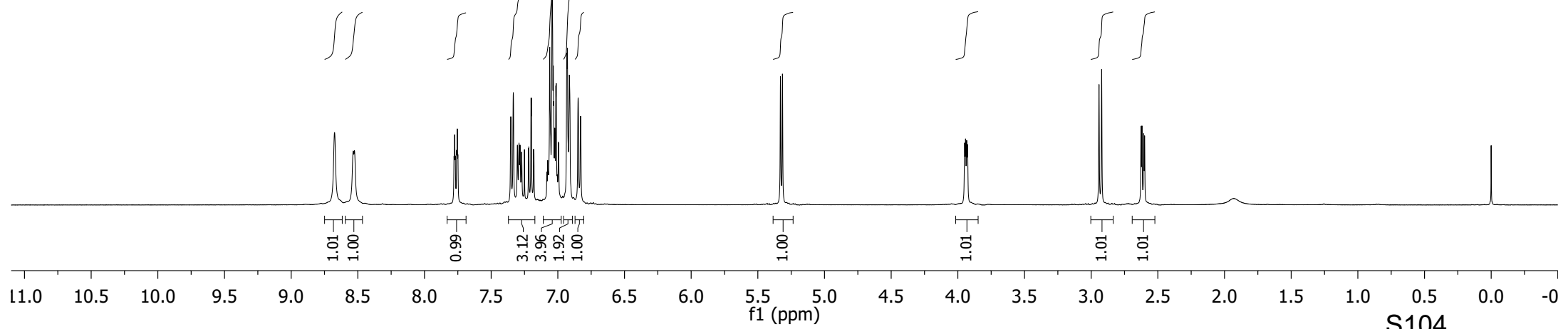
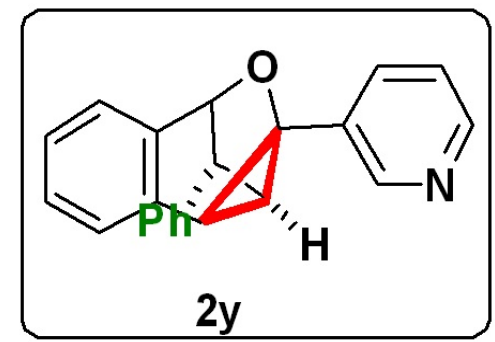
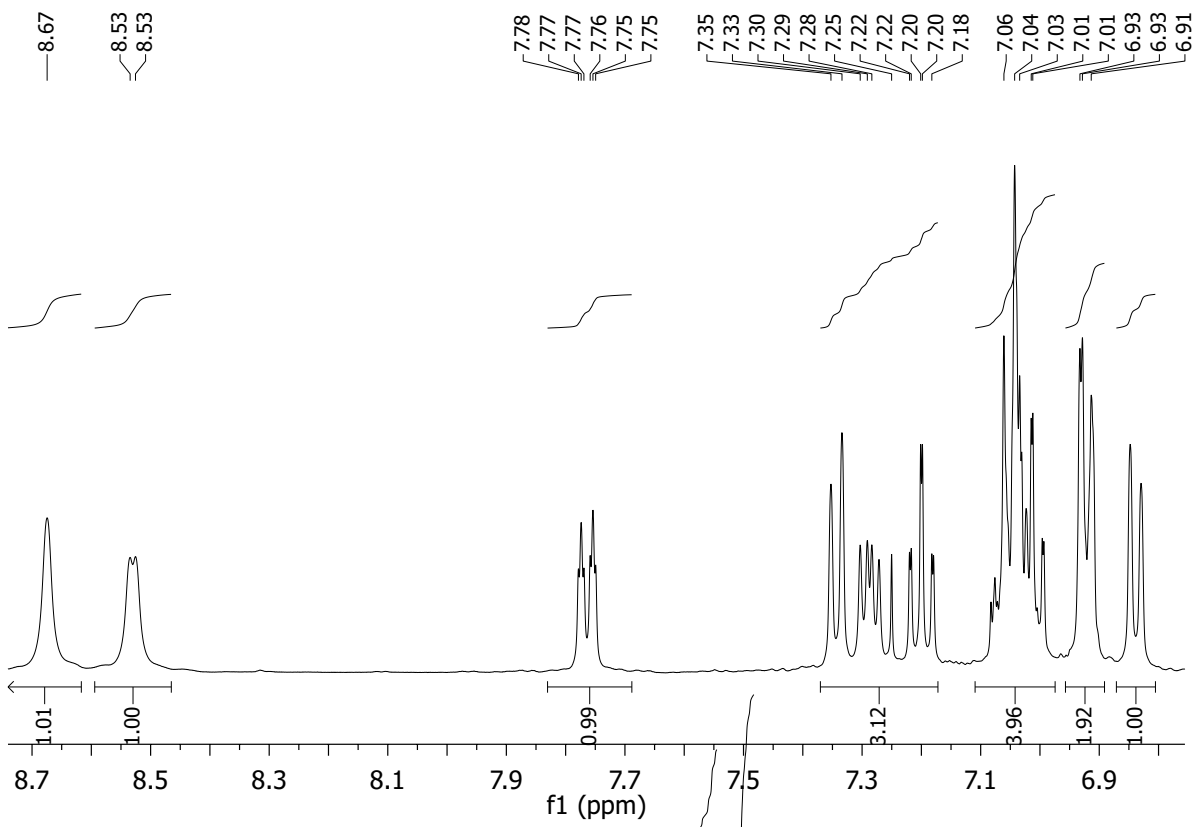
S103

Solvent CDCl_3
Spectrometer Frequency 400

8.67
8.53
8.53
7.78
7.77
7.77
7.76
7.75
7.75
7.35
7.33
7.20
7.20
7.06
7.04
7.03
7.03
7.01
7.01
6.93
6.93
6.91
6.85
6.83
5.32
3.95
3.94
3.93
3.93

2.94
2.92
2.63
2.62
2.61
2.60

0.00



Solvent CDCl_3
Spectrometer Frequency 100

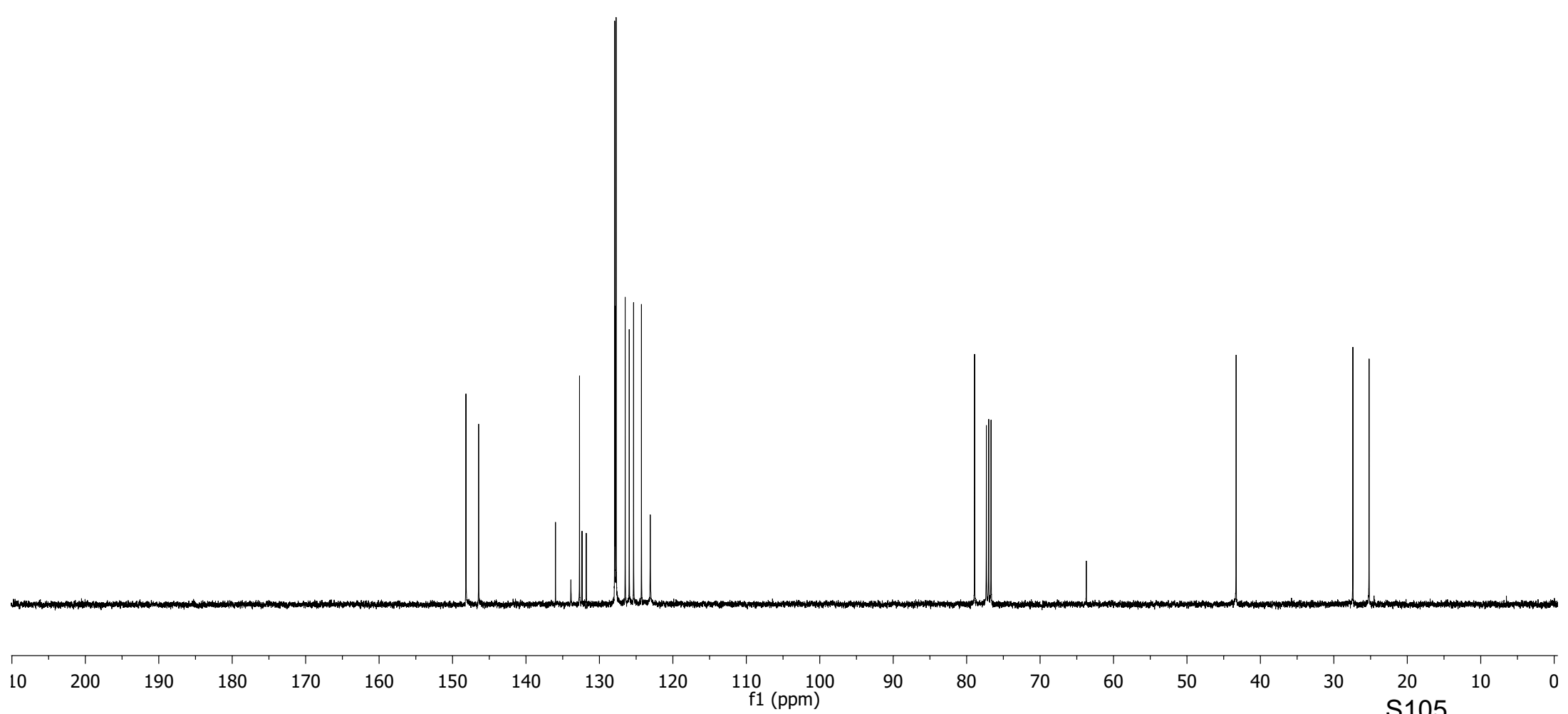
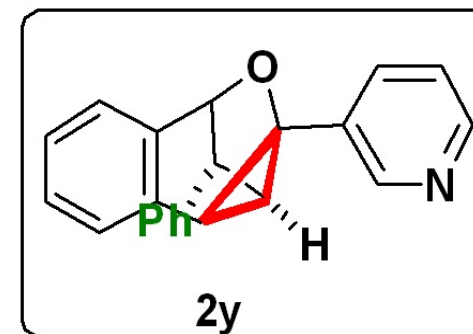
148.18
146.45
135.98
133.88
132.71
132.37
131.79
127.93
127.86
127.73
126.49
125.94
125.35
124.28
123.08

78.92
77.31
76.99
76.68

63.71

43.31

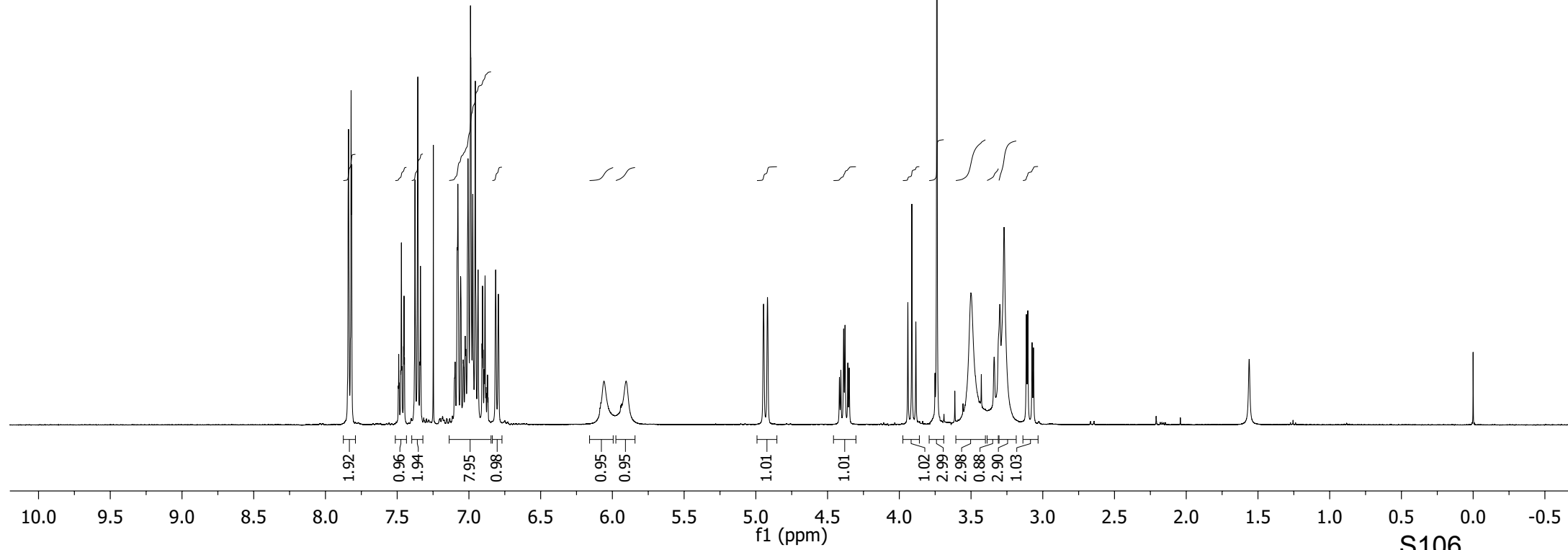
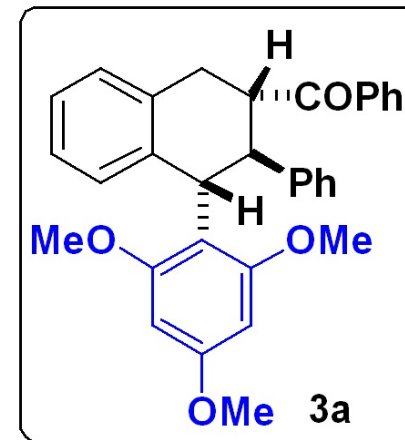
27.41
25.20



U0066-1
TPG-412

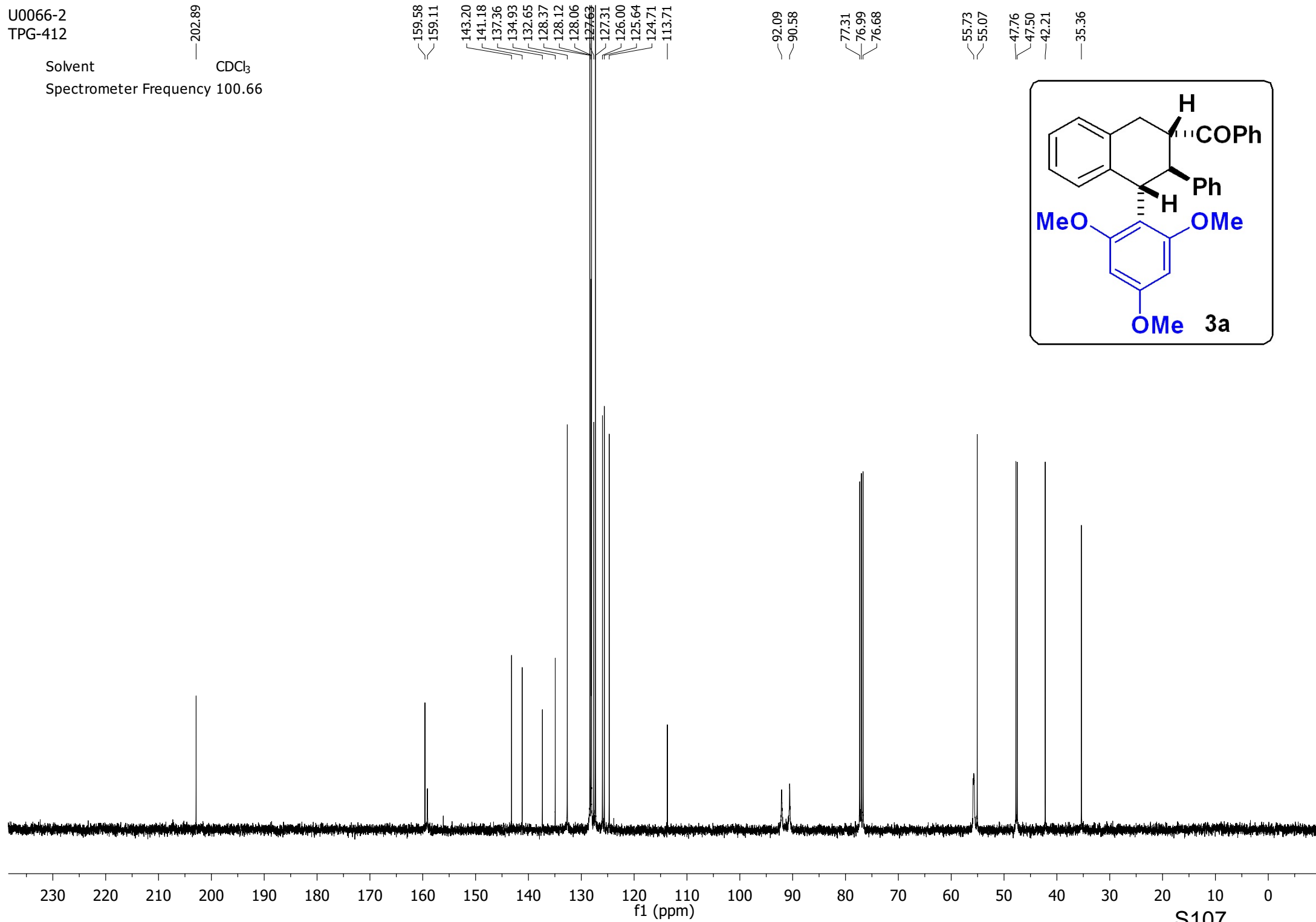
Solvent CDCl₃
Spectrometer Frequency 400.28

7.84 7.82 7.82 7.47 7.38 7.36 7.34 7.25 7.08 7.01 6.99 6.97 6.96 6.94 6.81 6.81 5.90 4.95 4.92 4.41 4.39 4.38 4.36 4.35 3.94 3.91 3.88 3.75 3.74 3.61 3.50 3.43 3.34 3.30 3.27 3.11 3.10 3.07 3.06 1.56 0.00



U0066-2
TPG-412

Solvent CDCl_3
Spectrometer Frequency 100.66



S107

V0038-15
TPG-413

Solvent CDCl_3
Spectrometer Frequency 150

164.48
143.97
143.60
131.15
128.83
128.59
128.55
127.22
127.13
126.84
126.71
118.17

100.96

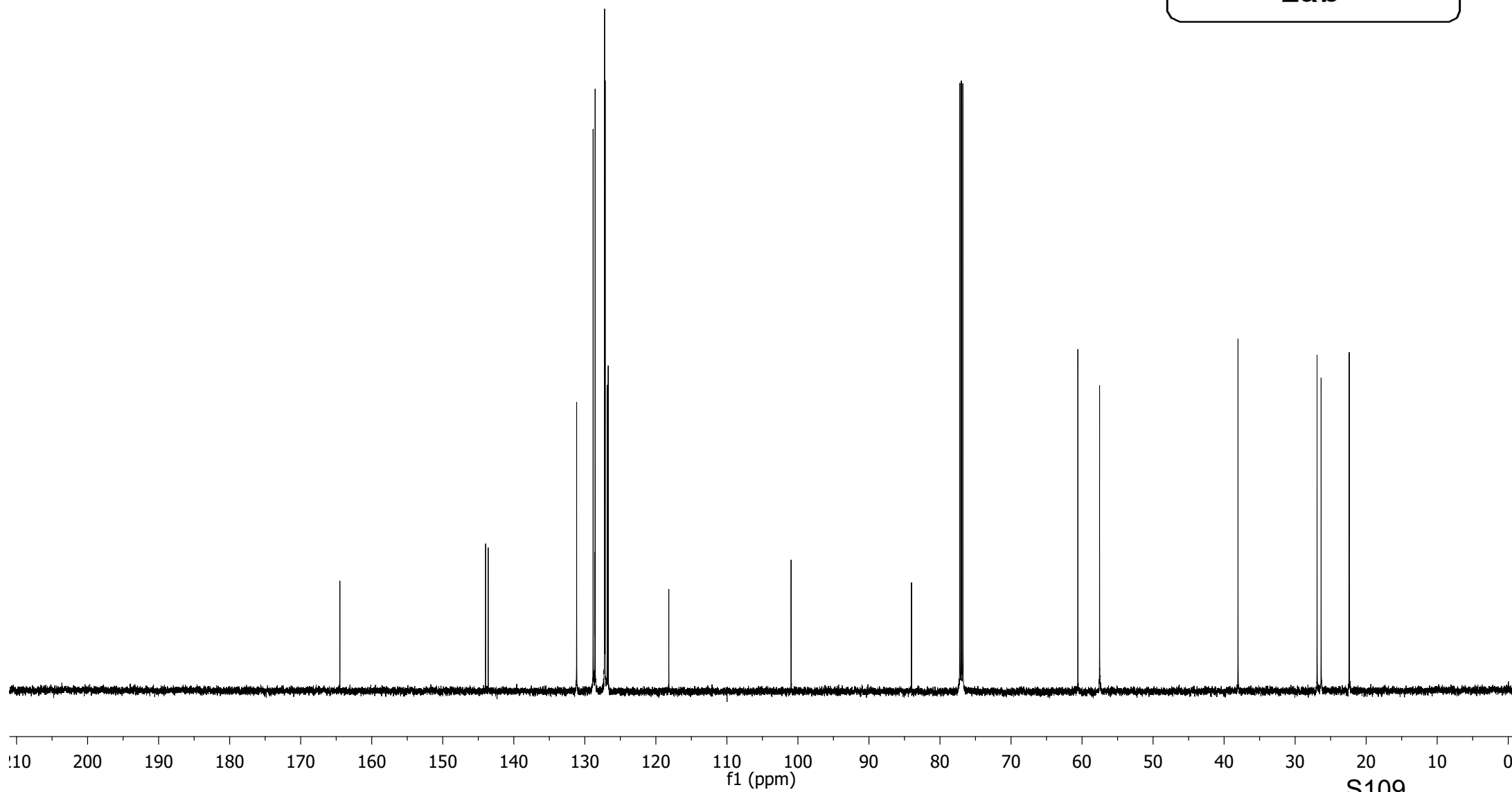
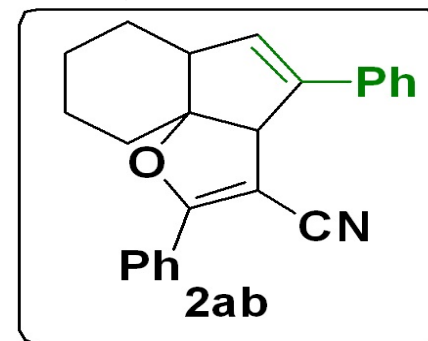
84.01

77.20
76.99
76.78

60.59
57.53

38.05

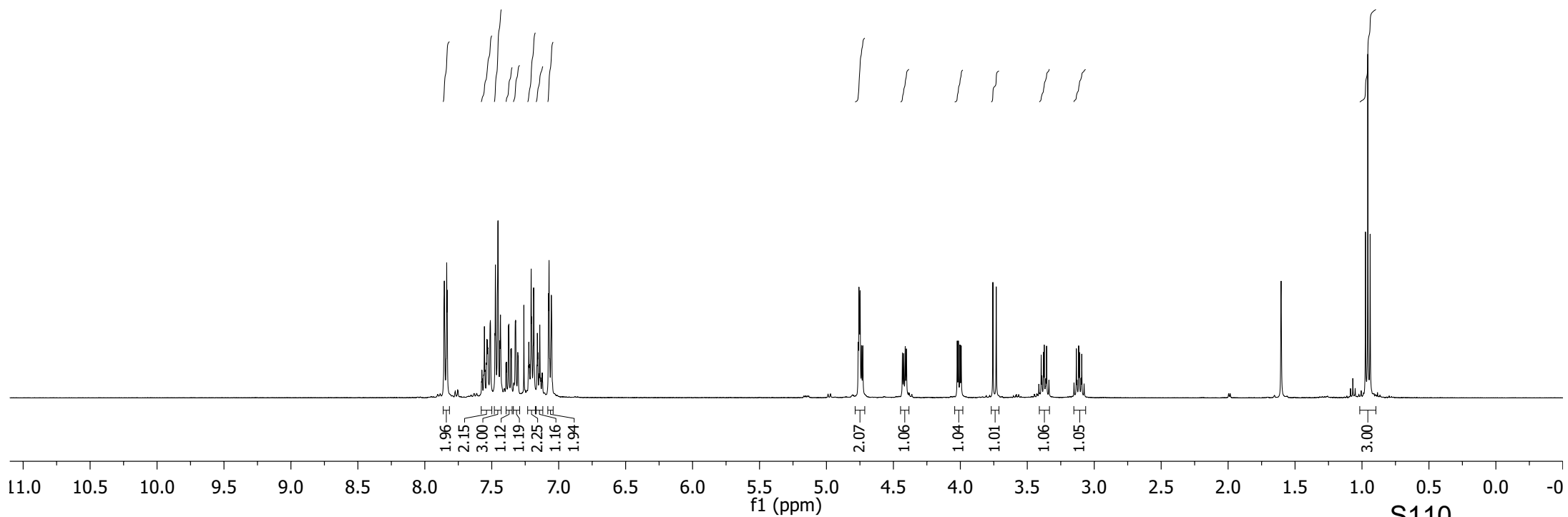
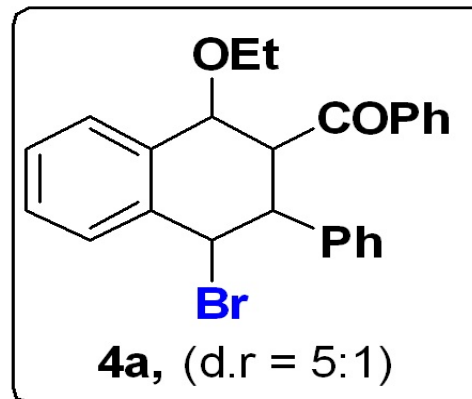
26.92
26.34
22.39



S109

7.85
7.84
7.83
7.57
7.56
7.55
7.54
7.54
7.53
7.53
7.51
7.48
7.47
7.45
7.44
7.43
7.39
7.39
7.38
7.37
7.36
7.35
7.32
7.32
7.31
7.26
7.23
7.22
7.22
7.21
7.20
7.19
7.16
7.16
7.16
7.15
7.14
7.12
7.08
7.07
4.95
4.76
4.76
4.75
4.74
4.73
4.41
4.40
4.02
4.01
3.76
3.73
3.40
3.39
3.38
3.37
3.36
3.36
3.34
3.13
3.13
3.12
3.11
3.10
3.08
1.07
0.97
0.96
0.94

Solvent CDCl₃
Spectrometer Frequency 400



Solvent CDCl_3
Spectrometer Frequency 100.66

144.48
139.83
137.04
135.23
132.69
129.76
129.08
128.69
128.66
128.49
127.97
127.71
127.56
126.51

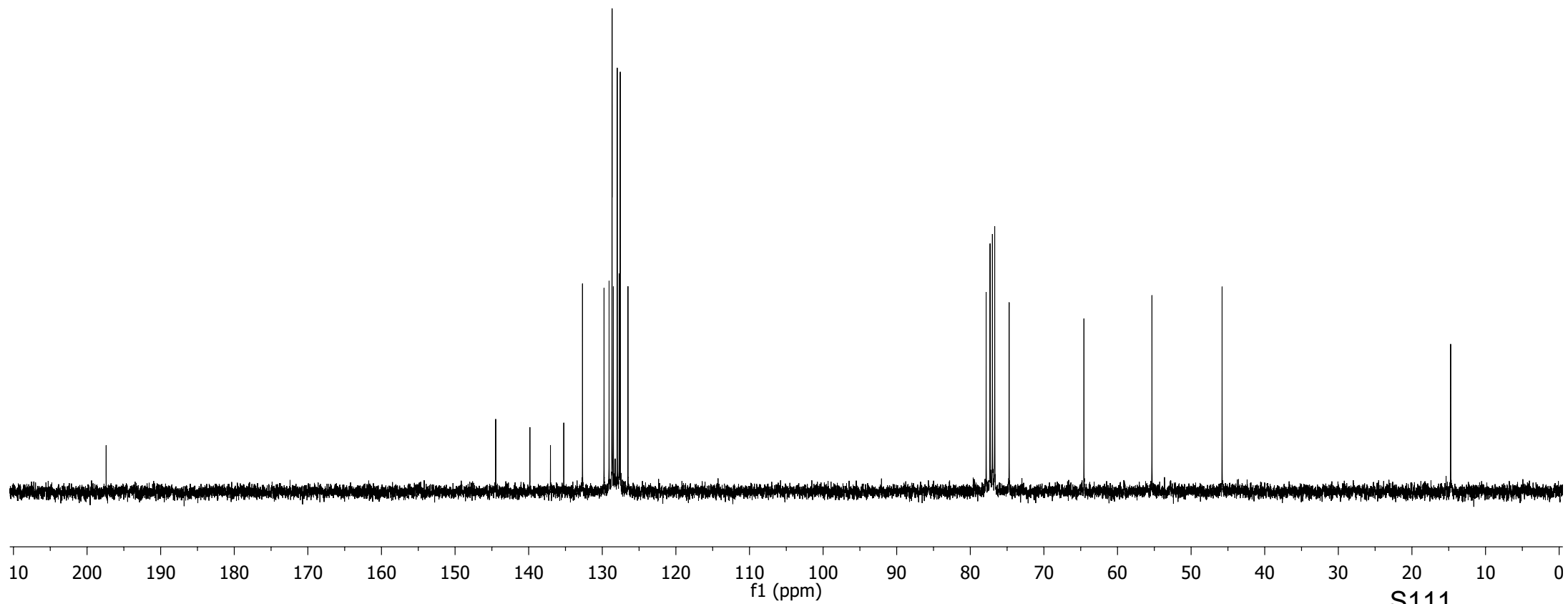
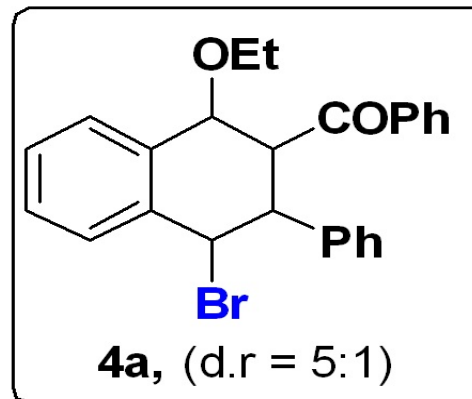
77.85
77.32
77.00
76.68
74.72

64.56

55.32

45.79

14.74



S111

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level G

PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	24	Report
PLAT005_ALERT_5_G	No Embedded Refinement Details Found in the CIF		Please Do !
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O1	105.1	Degree
PLAT793_ALERT_4_G	Model has Chirality at C1	(Centro SPGR)	S Verify
PLAT793_ALERT_4_G	Model has Chirality at C8	(Centro SPGR)	R Verify
PLAT793_ALERT_4_G	Model has Chirality at C9	(Centro SPGR)	R Verify
PLAT793_ALERT_4_G	Model has Chirality at C16	(Centro SPGR)	R Verify
PLAT793_ALERT_4_G	Model has Chirality at C17	(Centro SPGR)	S Verify
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	684	Note

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 0 **ALERT level B** = A potentially serious problem, consider carefully
- 0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
- 9 **ALERT level G** = General information/check it is not something unexpected

- 0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 2 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 1 ALERT type 3 Indicator that the structure quality may be low
 - 5 ALERT type 4 Improvement, methodology, query or suggestion
 - 1 ALERT type 5 Informative message, check
-

checkCIF publication errors

Alert level A

PUBL004_ALERT_1_A The contact author's name and address are missing,
_publ_contact_author_name and _publ_contact_author_address.

PUBL005_ALERT_1_A _publ_contact_author_email, _publ_contact_author_fax and
_publ_contact_author_phone are all missing.
At least one of these should be present.

PUBL006_ALERT_1_A _publ_requested_journal is missing
e.g. 'Acta Crystallographica Section C'

PUBL008_ALERT_1_A _publ_section_title is missing. Title of paper.

PUBL009_ALERT_1_A _publ_author_name is missing. List of author(s) name(s).

PUBL010_ALERT_1_A _publ_author_address is missing. Author(s) address(es).

PUBL012_ALERT_1_A _publ_section_abstract is missing.
Abstract of paper in English.

- 7 **ALERT level A** = Data missing that is essential or data in wrong format
 - 0 **ALERT level G** = General alerts. Data that may be required is missing
-

Publication of your CIF

You should attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the nature of your study may justify the reported deviations from journal submission requirements and the more serious of these should be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. *checkCIF* was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

If level A alerts remain, which you believe to be justified deviations, and you intend to submit this CIF for publication in a journal, you should additionally insert an explanation in your CIF using the Validation Reply Form (VRF) below. This will allow your explanation to be considered as part of the review process.

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

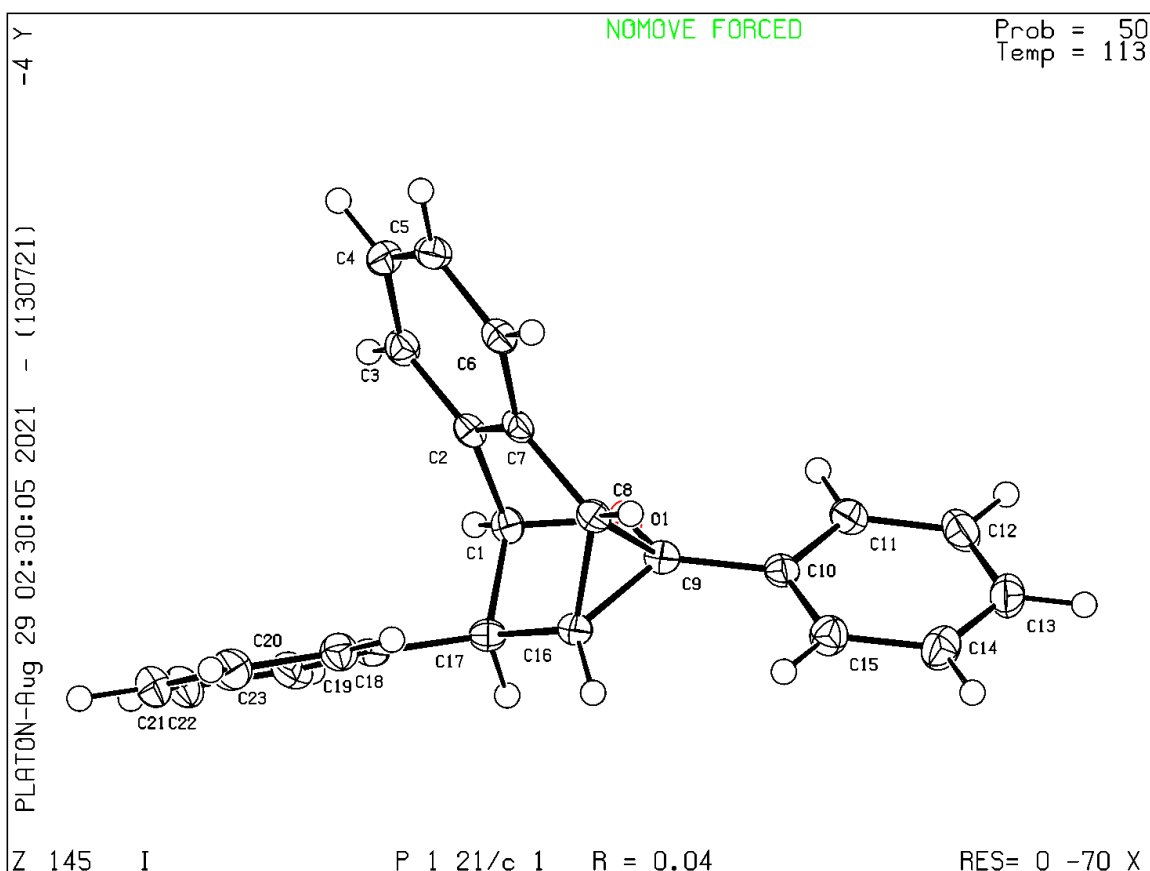
```
# start Validation Reply Form
_vrf_PUBL004_GLOBAL
;
PROBLEM: The contact author's name and address are missing,
RESPONSE: ...
;
_vrf_PUBL005_GLOBAL
;
PROBLEM: _publ_contact_author_email, _publ_contact_author_fax and
RESPONSE: ...
;
_vrf_PUBL006_GLOBAL
;
PROBLEM: _publ_requested_journal is missing
RESPONSE: ...
;
_vrf_PUBL008_GLOBAL
;
PROBLEM: _publ_section_title is missing. Title of paper.
RESPONSE: ...
;
_vrf_PUBL009_GLOBAL
;
PROBLEM: _publ_author_name is missing. List of author(s) name(s).
RESPONSE: ...
;
_vrf_PUBL010_GLOBAL
;
PROBLEM: _publ_author_address is missing. Author(s) address(es).
```

```
RESPONSE: ...
;
_vrf_PUBL012_GLOBAL
;
PROBLEM: _publ_section_abstract is missing.
RESPONSE: ...
;
# end Validation Reply Form
```

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF via the web. If you wish to submit your CIF for publication in IUCrData you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 13/07/2021; check.def file version of 13/07/2021

Datablock I - ellipsoid plot



checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: I

Bond precision:	C-C = 0.0022 A	Wavelength=0.71073	
Cell:	a=8.2282(3)	b=14.8841(7)	c=14.9762(5)
	alpha=90	beta=101.424(3)	gamma=90
Temperature:	113 K		
	Calculated	Reported	
Volume	1797.79(12)	1797.79(12)	
Space group	P 21/c	P 1 21/c 1	
Hall group	-P 2ybc	-P 2ybc	
Moiety formula	C24 H16 Cl N O	2(C24 H16 Cl N O)	
Sum formula	C24 H16 Cl N O	C48 H32 Cl2 N2 O2	
Mr	369.83	739.65	
Dx,g cm-3	1.366	1.366	
Z	4	2	
Mu (mm-1)	0.226	0.226	
F000	768.0	768.0	
F000'	768.88		
h,k,lmax	9,17,17	9,17,17	
Nref	3165	3163	
Tmin,Tmax	0.917,0.934	0.554,1.000	
Tmin'	0.914		

Correction method= # Reported T Limits: Tmin=0.554 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 24.999

R(reflections)= 0.0362(2786) wR2(reflections)= 0.0863(3163)

S = 1.056 Npar= 244

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

PLAT761_ALERT_1_C CIF Contains no X-H Bonds Please Check
PLAT762_ALERT_1_C CIF Contains no X-Y-H or H-Y-H Angles Please Check

Alert level G

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 27 Report
PLAT005_ALERT_5_G No Embedded Refinement Details Found in the CIF Please Do !
PLAT042_ALERT_1_G Calc. and Reported Moiety Formula Strings Differ Please Check
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 2.00 Check
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O1 106.3 Degree
PLAT793_ALERT_4_G Model has Chirality at C1 (Centro SPGR) R Verify
PLAT793_ALERT_4_G Model has Chirality at C8 (Centro SPGR) R Verify
PLAT793_ALERT_4_G Model has Chirality at C10 (Centro SPGR) S Verify
PLAT793_ALERT_4_G Model has Chirality at C17 (Centro SPGR) S Verify
PLAT793_ALERT_4_G Model has Chirality at C24 (Centro SPGR) S Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints 768 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
11 **ALERT level G** = General information/check it is not something unexpected

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
5 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

checkCIF publication errors

Alert level A

PUBL004_ALERT_1_A The contact author's name and address are missing,
_publ_contact_author_name and _publ_contact_author_address.
PUBL005_ALERT_1_A _publ_contact_author_email, _publ_contact_author_fax and
_publ_contact_author_phone are all missing.
At least one of these should be present.
PUBL006_ALERT_1_A _publ_requested_journal is missing
e.g. 'Acta Crystallographica Section C'
PUBL008_ALERT_1_A _publ_section_title is missing. Title of paper.
PUBL009_ALERT_1_A _publ_author_name is missing. List of author(s) name(s).
PUBL010_ALERT_1_A _publ_author_address is missing. Author(s) address(es).
PUBL012_ALERT_1_A _publ_section_abstract is missing.
Abstract of paper in English.

7 **ALERT level A** = Data missing that is essential or data in wrong format
0 **ALERT level G** = General alerts. Data that may be required is missing

Publication of your CIF

You should attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the nature of your study may justify the reported deviations from journal submission requirements and the more serious of these should be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. *checkCIF* was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

If level A alerts remain, which you believe to be justified deviations, and you intend to submit this CIF for publication in a journal, you should additionally insert an explanation in your CIF using the Validation Reply Form (VRF) below. This will allow your explanation to be considered as part of the review process.

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

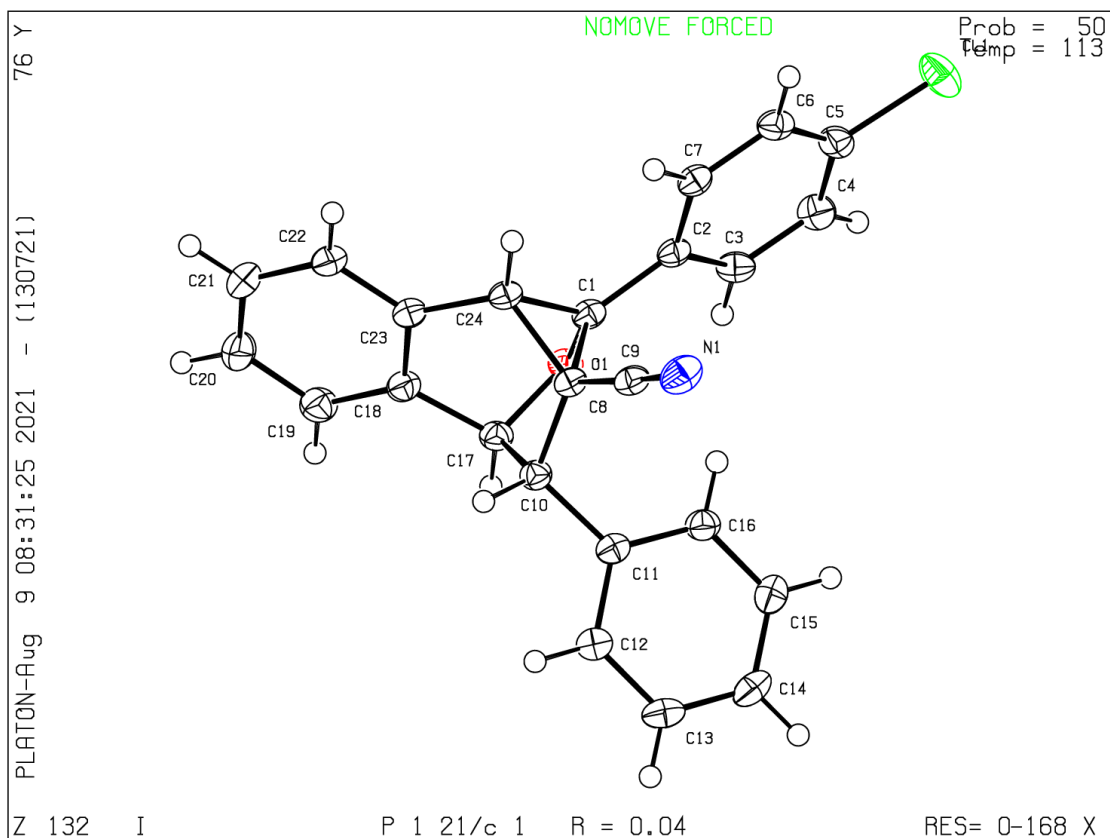
```
# start Validation Reply Form
_vrf_PUBL004_GLOBAL
;
PROBLEM: The contact author's name and address are missing,
RESPONSE: ...
;
_vrf_PUBL005_GLOBAL
;
PROBLEM: _publ_contact_author_email, _publ_contact_author_fax and
RESPONSE: ...
;
_vrf_PUBL006_GLOBAL
;
PROBLEM: _publ_requested_journal is missing
RESPONSE: ...
;
_vrf_PUBL008_GLOBAL
;
PROBLEM: _publ_section_title is missing. Title of paper.
RESPONSE: ...
;
_vrf_PUBL009_GLOBAL
;
PROBLEM: _publ_author_name is missing. List of author(s) name(s).
RESPONSE: ...
;
_vrf_PUBL010_GLOBAL
;
PROBLEM: _publ_author_address is missing. Author(s) address(es).
RESPONSE: ...
;
_vrf_PUBL012_GLOBAL
;
```

PROBLEM: _publ_section_abstract is missing.
RESPONSE: ...
;
end Validation Reply Form

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF via the web. If you wish to submit your CIF for publication in IUCrData you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 13/07/2021; check.def file version of 13/07/2021

Datablock I - ellipsoid plot



The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level G

PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	36	Report
PLAT005_ALERT_5_G	No Embedded Refinement Details Found in the CIF		Please Do !
PLAT793_ALERT_4_G	Model has Chirality at C8 (Centro SPGR)	S	Verify
PLAT793_ALERT_4_G	Model has Chirality at C9 (Centro SPGR)	S	Verify
PLAT793_ALERT_4_G	Model has Chirality at C10 (Centro SPGR)	S	Verify
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	1014	Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
6 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

checkCIF publication errors

Alert level A

PUBL004_ALERT_1_A The contact author's name and address are missing,
_publ_contact_author_name and _publ_contact_author_address.
PUBL005_ALERT_1_A _publ_contact_author_email, _publ_contact_author_fax and
_publ_contact_author_phone are all missing.
At least one of these should be present.
PUBL006_ALERT_1_A _publ_requested_journal is missing
e.g. 'Acta Crystallographica Section C'
PUBL008_ALERT_1_A _publ_section_title is missing. Title of paper.
PUBL009_ALERT_1_A _publ_author_name is missing. List of author(s) name(s).
PUBL010_ALERT_1_A _publ_author_address is missing. Author(s) address(es).
PUBL012_ALERT_1_A _publ_section_abstract is missing.
Abstract of paper in English.

7 **ALERT level A** = Data missing that is essential or data in wrong format
0 **ALERT level G** = General alerts. Data that may be required is missing

Publication of your CIF

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Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PUBL004_GLOBAL
;
PROBLEM: The contact author's name and address are missing,
RESPONSE: ...
;
_vrf_PUBL005_GLOBAL
;
PROBLEM: _publ_contact_author_email, _publ_contact_author_fax and
RESPONSE: ...
;
_vrf_PUBL006_GLOBAL
;
PROBLEM: _publ_requested_journal is missing
RESPONSE: ...
;
_vrf_PUBL008_GLOBAL
;
PROBLEM: _publ_section_title is missing. Title of paper.
RESPONSE: ...
;
_vrf_PUBL009_GLOBAL
;
PROBLEM: _publ_author_name is missing. List of author(s) name(s).
RESPONSE: ...
;
_vrf_PUBL010_GLOBAL
;
PROBLEM: _publ_author_address is missing. Author(s) address(es).
```

```

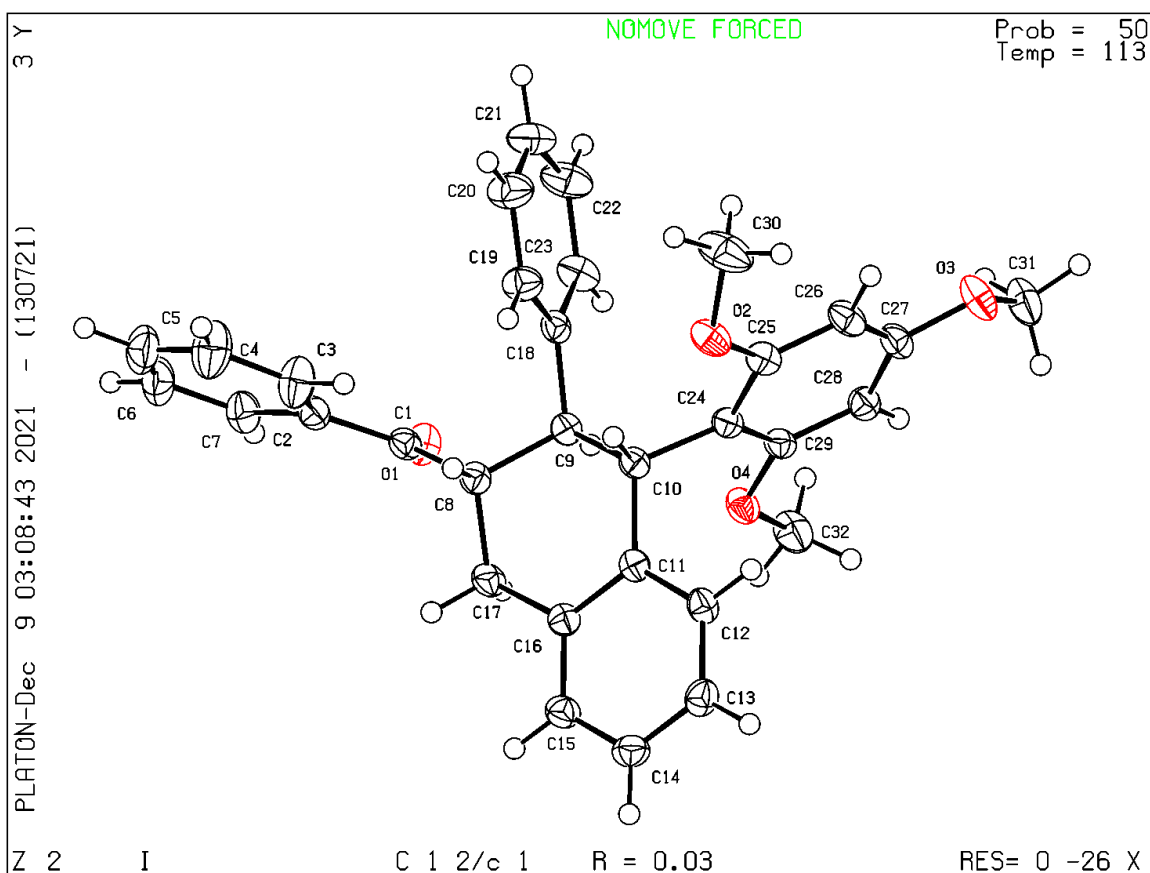
RESPONSE: ...
;
_vrf_PUBL012_GLOBAL
;
PROBLEM: _publ_section_abstract is missing.
RESPONSE: ...
;
# end Validation Reply Form

```

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF via the web. If you wish to submit your CIF for publication in IUCrData you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 13/07/2021; check.def file version of 13/07/2021

Datablock I - ellipsoid plot



The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.



Alert level G

PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	2	Report
PLAT005_ALERT_5_G	No Embedded Refinement Details Found in the CIF		Please Do !
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O1	108.8	Degree
PLAT793_ALERT_4_G	Model has Chirality at C1	(Centro SPGR)	S Verify
PLAT793_ALERT_4_G	Model has Chirality at C2	(Centro SPGR)	R Verify
PLAT793_ALERT_4_G	Model has Chirality at C3	(Centro SPGR)	R Verify

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 0 **ALERT level B** = A potentially serious problem, consider carefully
- 0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
- 6 **ALERT level G** = General information/check it is not something unexpected

- 0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 2 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 0 ALERT type 3 Indicator that the structure quality may be low
 - 3 ALERT type 4 Improvement, methodology, query or suggestion
 - 1 ALERT type 5 Informative message, check
-

checkCIF publication errors



Alert level A

PUBL004_ALERT_1_A The contact author's name and address are missing,
_publ_contact_author_name and _publ_contact_author_address.

PUBL005_ALERT_1_A _publ_contact_author_email, _publ_contact_author_fax and
_publ_contact_author_phone are all missing.
At least one of these should be present.

PUBL006_ALERT_1_A _publ_requested_journal is missing
e.g. 'Acta Crystallographica Section C'

PUBL008_ALERT_1_A _publ_section_title is missing. Title of paper.

PUBL009_ALERT_1_A _publ_author_name is missing. List of author(s) name(s).

PUBL010_ALERT_1_A _publ_author_address is missing. Author(s) address(es).

PUBL012_ALERT_1_A _publ_section_abstract is missing.
Abstract of paper in English.

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-

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PROBLEM: _publ_contact_author_email, _publ_contact_author_fax and
RESPONSE: ...
;
_vrf_PUBL006_GLOBAL
;
PROBLEM: _publ_requested_journal is missing
RESPONSE: ...
;
_vrf_PUBL008_GLOBAL
;
PROBLEM: _publ_section_title is missing. Title of paper.
RESPONSE: ...
;
_vrf_PUBL009_GLOBAL
;
PROBLEM: _publ_author_name is missing. List of author(s) name(s).
RESPONSE: ...
;
_vrf_PUBL010_GLOBAL
;
PROBLEM: _publ_author_address is missing. Author(s) address(es).
```

```
RESPONSE: ...
;
_vrf_PUBL012_GLOBAL
;
PROBLEM: _publ_section_abstract is missing.
RESPONSE: ...
;
# end Validation Reply Form
```

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF via the web. If you wish to submit your CIF for publication in IUCrData you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 19/02/2022; check.def file version of 19/01/2022

Datablock I - ellipsoid plot

