Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Iodine(III)-mediated oxidative intramolecular arene-alkene coupling exemplified in the synthesis of phenanthrenes

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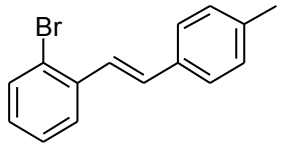
General Methods and materials: Chemicals were obtained from commercial sources and were used without further purification. Yields correspond to isolated compounds unless indicated otherwise. TLC: MACHEREY-NAGEL, TLC plates Alugram® Sil G/UV254. Visualization of the developed chromatogram was performed by fluorescence quenching at 254 nm and staining with aqueous ceric ammonium molybdate, *p*-anisaldehyde, or potassium permanganate. Chromatography: separations were carried out on Merck Silica 60 (0.063-0.200 mm, 70-230 mesh ASTM) using forced flow. IR: Bruker FT-IR Alpha-spectrometer with ATR sampling module. High-resolution mass spectrometry (HR-MS): APEX IV 7T FTICR, BRUKER Daltonic. M.p.: BÜCHI 540 capillary melting point apparatus, values are uncorrected. NMR (^{1}H, ^{13}C) spectra were recorded at 300, 400, and 500 MHz (^{1}H) as well as 101 and 126 MHz (^{13}C), respectively, on VARIAN Unity-300, Bruker Avance III 400 and Varian Inova 500 instruments in $CDCl_3$ solutions, if not otherwise specified. Chemical shifts (δ) are given in ppm. Multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sex = sextet, sept = septet, m = multiplet.

General procedure A: Suzuki coupling of boronic acid (-esters) with 1-bromo-2-iodobenzene[1]

To a degassed solution of 1-bromo-2-iodobenzene, K_2CO_3 (3.00 eq.) and $Pd(PPh_3)_4$ (0.05 eq.) in PhMe/EtOH/H_2O (3:2:1, ~0.5 M) under an argon atmosphere is added the boronic acid or its pinacol ester (1.20 eq.) and the resulting mixture is stirred at 90 °C for 16 h. H_2O and DCM are added and the phases are separated. The aquaeous layer is extracted with DCM (3 x). The combined organic layers are washed with brine, dried over anhydrous Na_2SO_4, filtered and the solvent is removed under reduced pressure. The residue is purified on silica gel to yield the title compound.

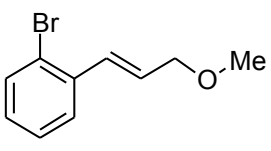
(*E*)-1-Bromo-2-(4-methylstyryl)benzene



Following general procedure A: 1-bromo-2-iodobenzene (635 mg, 2.24 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (130 mg, 112 µmol, 0.05 eq.), K_2CO_3 (931 mg, 6.73 mmol, 3.00 eq.), (*E*)-(4-methylstyryl)boronic acid (400 mg, 2.47 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc, 10:1; yield: 341 mg, 1.25 mmol, 56%, yellow liquid.

DC: R_f = 0.56 (pentane); **IR** (ATR): ν = 3049, 2918, 1512, 1464, 1436, 1022, 960, 801, 746, 610, 500 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.67 (dd, $^{3}J_{HH}$ = 7.9 Hz, $^{4}J_{HH}$ = 1.6 Hz, 1H), 7.58 (dd, $^{3}J_{HH}$ = 8.0 Hz, $^{4}J_{HH}$ = 1.3 Hz, 1H), 7.48-7.38 (m, 3H), 7.35-7.27 (m, 1H), 7.19 (d, $^{3}J_{HH}$ = 7.9 Hz, 2H), 7.11 (ddd, $^{3}J_{HH}$ = 7.9, 7.2 Hz, $^{4}J_{HH}$ = 1.6 Hz, 1H), 7.02 (d, $^{3}J_{HH}$ = 16.2 Hz, 1H), 2.38 (s, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 138.11, 137.38, 134.34, 133.11, 131.45, 129.52, 128.64, 127.58, 126.83, 126.68, 126.55, 124.13, 21.53.; **HR-MS** (EI): $[C_{15}H_{13}Br]^+$ ($[M]^+$): calcd.: 272.0201, obs.: 272.0211.

(*E*)-1-Bromo-2-(3-methoxyprop-1-en-1-yl)benzene

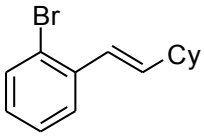


Following general procedure A: 1-bromo-2-iodobenzene (641 mg, 2.27 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (131 mg, 113 µmol, 0.05 eq.), K_2CO_3 (940 mg, 6.80 mmol, 3.00 eq.), (E)-(3-methoxyprop-1-en-1-yl)boronic acid (494 mg, 2.49 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with

pentane/EtOAc, 100:1→30:1; yield: 381 mg, 1.68 mmol, 74%, yellow liquid.

DC: R_f = 0.16 (pentane); **IR** (ATR): ν = 2925, 2821, 1466, 1436, 1191, 1125, 1109, 1023, 963, 747 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.52 (ddd, $^3J_{HH}$ = 9.3, 8.0 Hz, $^4J_{HH}$ = 1.5 Hz, 2H), 7.25 (td, $^3J_{HH}$ = 7.6 Hz, $^4J_{HH}$ = 1.2 Hz, 1H), 7.15-7.04 (m, 1H), 6.93 (d, $^3J_{HH}$ = = 15.6 Hz, 1H), 6.20 (dt, $^3J_{HH}$ = 15.9, 5.9 Hz, 1H), 4.11 (dd, $^3J_{HH}$ = 5.9 Hz, $^4J_{HH}$ = 1.6 Hz, 2H), 3.39 (s, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 136.6, 132.9, 131.1, 129.0, 128.9, 127.5, 127.1, 123.6, 72.9, 58.1; **HR-MS** (EI): $[C_{10}H_{11}BrO]^+$ ($[M]^+$): calcd.: 255.9993, obs.: 225.9996.

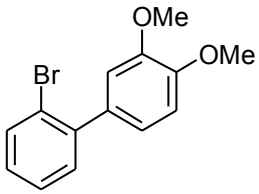
(*E*)-1-Bromo-2-(2-cyclohexylvinyl)benzene



Following general procedure A: 1-bromo-2-iodobenzene (943 mg, 3.33 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (193 mg, 167 µmol, 0.05 eq.), K_2CO_3 (1.38 g, 10.0 mmol, 3.00 eq.), (*E*)-(2-cyclohexylvinyl)boronic acid (565 mg, 3.67 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 9 mL); eluting with pentane; yield: 829 mg, 3.13 mmol, 94%, colorless liquid. The sample is an inseparable mixture with 8% 1-bromo-2-iodobenzene

DC: R_f = 0.90 (pentane); **IR** (ATR): ν = 2922, 2850, 1466, 1448, 1023, 964, 746 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.49 (ddd, $^3J_{HH}$ = 12.5, 7.9 Hz, $^4J_{HH}$ = 1.5 Hz, 2H), 7.30-7.17 (m, 1H), 7.03 (ddd, $^3J_{HH}$ = 8.0, 7.3 Hz, $^4J_{HH}$ = 1.7 Hz, 1H), 6.66 (dd, $^3J_{HH}$ = 15.9 Hz, $^4J_{HH}$ = 1.4 Hz, 1H), 6.10 (dd, $^3J_{HH}$ = 15.9, 7.0 Hz, 1H), 2.23-2.11 (m, 1H), 1.89-1.60 (m, 5H), 1.40-1.08 (m, 5H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 139.8, 137.7, 132.8, 128.0, 127.3, 126.7, 126.2, 123.3, 41.2, 32.8, 26.1, 26.0; **HR-MS** (EI): $[C_{14}H_{17}Br]^+$ ($[M]^+$): calcd.: 264.0514, obs.: 264.0521.

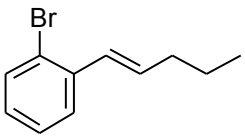
2-Bromo-3',4'-dimethoxy-1,1'-biphenyl



Following general procedure A: 1-bromo-2-iodobenzene (1.46 g, 5.16 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (298 mg, 258 µmol, 0.05 eq.), K_2CO_3 (2.14 g, 15.5 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.50 g, 5.68 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 12 mL); eluting with pentane/ EtOAc 15:1; yield: 987 mg, 3.74 mmol, 72%, light yellow solid.

DC: R_f = 0.21 (pentane/EtOAc 15:1); **Smp.**: 66 °C; **IR** (ATR): ν = 2934, 2834, 1519, 1468, 1246, 1215, 1172, 1026, 755 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.70-7.64 (m, 1H), 7.37-7.32 (m, 2H), 7.19 (ddd, $^3J_{HH}$ = 8.0, 5.3, 3.8 Hz, 1H), 6.99-6.91 (m, 3H), 3.93 (s, 3H), 3.91 (s, 3H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.5, 148.2, 142.3, 133.8, 133.1, 131.3, 128.5, 127.3, 122.8, 121.7, 112.8, 110.6, 55.9, 55.9; **HR-MS** (ESI): $[C_{14}H_{13}BrO_2Na]^+$ ($[M+Na]^+$): calcd.: 314.9981, obs.: 314.9991.

(*E*)-1-Bromo-2-(pent-1-en-1-yl)benzene

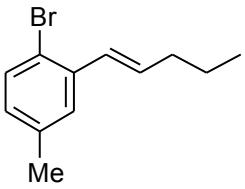


Following general procedure A: 1-bromo-2-iodobenzene (1.13 g, 3.99 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (231 mg, 200 µmol, 0.05 eq.), K_2CO_3 (1.66 g, 12.0 mmol, 3.00 eq.), (*E*)-pent-1-en-1-ylboronic acid (500 mg, 4.39 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 12 mL); eluting with pentane; yield: 1.01 g, 3.20 mmol, 72%, colorless liquid. The sample contains 15 mol% of inseparable 1-bromo-2-iodobenzene.

DC: R_f = 0.74 (pentane); **IR** (ATR): ν = 2958, 2928, 2871, 1464, 1435, 1022, 1002, 962, 744, 667 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.59-7.46 (m, 2H), 7.26

(tdd, $^3J_{HH}$ = 7.9 Hz, $^4J_{HH}$ = 1.3, 0.6 Hz, 1H), 7.07 (ddd, $^3J_{HH}$ = 7.7, 7.2 Hz, $^4J_{HH}$ = 1.6 Hz, 1H), 6.73 (dt, $^3J_{HH}$ = 15.7 Hz, $^3J_{HH}$ = 1.5 Hz, 1H), 6.19 (dt, $^3J_{HH}$ = 15.7, 6.9 Hz, 1H), 2.26 (qd, $^3J_{HH}$ = 7.2 Hz, $^4J_{HH}$ = 1.5 Hz, 2H), 1.53 (h, $^3J_{HH}$ = 7.3 Hz, 2H), 1.00 (t, $^3J_{HH}$ = 7.4 Hz, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 137.7, 134.0, 132.8, 128.8, 128.1, 127.3, 126.8, 123.1, 35.1, 22.4, 13.7; **HR-MS** (EI): $[C_{11}H_{13}Br]^+$ ($[M]^+$): calcd.: 224.0201, obs.: 224.0194.

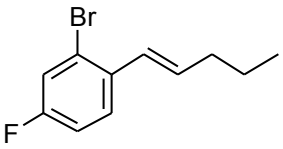
(*E*)-1-Bromo-4-methyl-2-(pent-1-en-1-yl)benzene



Following general procedure A: 4-bromo-3-iodotoluene (1.18 g, 3.99 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (231 mg, 200 µmol, 0.05 eq.), K_2CO_3 (1.66 g, 12.0 mmol, 3.00 eq.), (*E*)-pent-1-en-1-ylboronic acid (500 mg, 4.39 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 12 mL); eluting with pentane; yield: 915 mg, 3.16 mmol, 79%, liquid. The sample contains 15 mol% of inseparable 4-bromo-2-iodotoluene.

DC: R_f = 0.74 (pentane); **IR** (ATR): ν = 2958, 2927, 1693, 1602, 1485, 1458, 1379, 1038, 964, 865, 824, 673 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.38 (d, $^3J_{HH}$ = = 8.0 Hz, 2H), 7.36 (dd, $^4J_{HH}$ = 1.7, 0.8 Hz, 1H), 7.04 (ddd, $^3J_{HH}$ = 8.0, $^4J_{HH}$ = 1.1, 0.6 Hz, 1H), 6.67 (d, $^3J_{HH}$ = 15.7 Hz, 1H), 6.12 (dt, $^3J_{HH}$ = 15.7, 7.0 Hz, 1H), 2.30 (s, 3H), 2.22 (qd, $^3J_{HH}$ = 7.3, $^4J_{HH}$ = 1.6 Hz, 2H), 1.51 (h, $^3J_{HH}$ = 7.3 Hz, 2H), 0.97 (t, $^3J_{HH}$ = 7.3 Hz, 3H). **^{13}C-NMR** (101 MHz, $CDCl_3$): δ (ppm) = 139.9, 138.2, 134.8, 133.1, 133.0, 128.5, 128.2, 126.4, 35.1, 22.4, 20.6, 13.7; **HR-MS** (EI): $[C_{12}H_{15}Br]^+$ ($[M]^+$): calcd.: 238.0357, obs.: 238.0357.

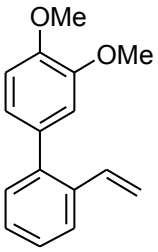
(*E*)-2-Bromo-4-fluoro-1-(pent-1-en-1-yl)benzene



Following general procedure A: 1-Bromo-4-fluoro-2-iodobenzene (600 mg, 1.99 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (115 mg, 100 µmol, 0.05 eq.), K_2CO_3 (827 mg, 5.98 mmol, 3.00 eq.), (*E*)-pent-1-en-1-ylboronic acid (250 mg, 2.19 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 9 mL); eluting with pentane; yield: 439 mg, 1.81 mmol, 91%, colorless liquid.

DC: R_f = 0.99 (pentane); **IR** (ATR): ν = cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.45 (dd, $^3J_{HH}$ = 8.7 Hz, $^3J_{HF}$ = 6.0 Hz, 1H), 7.27 (dd, $^3J_{HF}$ = 8.3 Hz, $^3J_{HH}$ = 2.6 Hz, 1H), 6.97 (dddd, $^3J_{HH}$ = 8.0, 2.6 Hz, $^3J_{HF}$ = 8.7 Hz, $^4J_{HH}$ = 0.6 Hz, 1H), 6.63 (dd, $^3J_{HH}$ = 15.7 Hz, $^4J_{HH}$ = 0.5 Hz, 1H), 6.09 (dt, $^3J_{HH}$ = 15.7, 6.9 Hz, 1H), 2.22 (qd, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 1.5 Hz, 2H), 1.52 (sex, $^3J_{HH}$ = 7.3 Hz, 2H), 0.97 (t, $^3J_{HH}$ = 7.3 Hz, 3H). **^{13}C-NMR** (101 MHz, $CDCl_3$): δ (ppm) = 161.2 (d, $^1J_{CF}$ = 250.2 Hz), 134.1 (d, $^4J_{CF}$ = 3.9 Hz), 133.9 (d, $^5J_{CF}$ = 2.0 Hz), 127.7 , 127.6 (d, $^3J_{CF}$ = 8.1 Hz), 122.8 (d, $^3J_{CF}$ = 9.5 Hz), 119.7 (d, $^2J_{CF}$ = 24.0 Hz), 114.7 (d, $^2J_{CF}$ = 21.1 Hz), 35.1, 22.4, 13.7; **^{19}F-NMR** (283 MHz, $CDCl_3$) = -114.22 (ddd, $^3J_{HF}$ = 8.1, 6.0 Hz). **HR-EI-MS**: $[C_{11}H_{12}FBr]^+$ ($[M]^+$): calcd.: 242.0106, obs.: 242.0101.

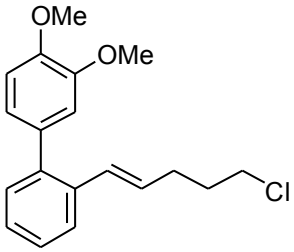
3',4'-Dimethoxy-2-vinyl-1,1'-biphenyl



Following general procedure A: 2-bromo-3',4'-dimethoxy-1,1'-biphenyl (400 mg, 1.36 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (79 mg, 68.0 µmol, 0.05 eq.), K_2CO_3 (566 mg, 4.09 mmol, 3.00 eq.), vinylboronicacid pinacolester (243 mg, 1.58 µmol, 1.16 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 15:1; yield: 116 mg, 483 µmol, 35%, colorless oil.

DC: R_f = 0.51 (Hexane/EtOAc 5:1); **IR** (ATR): ν = cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ (ppm) = 7.67-7.60 (m, 1H), 7.37-7.28 (m, 3H), 6.95-6.86 (m, 3H), 6.75 (dd, $^3J_{HH}$ = 17.5, 11.0 Hz, 1H), 5.69 (dd, $^3J_{HH}$ = 17.5, $^4J_{HH}$ = 1.4 Hz, 1H), 5.19 (dd, $^3J_{HH}$ = 11.0 Hz, $^4J_{HH}$ = 1.3 Hz, 1H), 3.93 (s, 3H), 3.88 (s, 3H); **^{13}C-NMR** (101 MHz, $CDCl_3$): δ (ppm) = 148.4, 148.2, 140.6, 136.1, 135.8, 133.5, 130.0, 127.6, 127.2, 125.8, 122.0, 114.4, 113.2, 110.8, 55.9, 55.9; **HR-MS** (ESI): $[C_{16}H_{17}O_2]^+$ ($[M+H]^+$): calcd.: 241.1223, obs.: 241.1217.

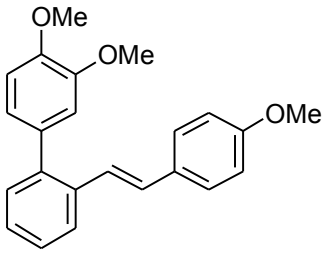
(*E*)-2-(5-Chloropent-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl



Following general procedure A: 2-bromo-3',4'-dimethoxy-1,1'-biphenyl (100 mg, 341 µmol, 1.00 eq.), $Pd(PPh_3)_4$ (20 mg, 17.1 µmol, 0.05 eq.), K_2CO_3 (141 mg, 1.02 mmol, 3.00 eq.), (*E*)-5-chloro-1-pentene-1-yl)boronic acid (87 mg, 375 µmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3.5 mL); eluting with pentane/EtOAc 15:1; yield: 44 mg, 139 mmol, 41%, colorless liquid.

DC: R_f = 0.10 (Hexane/EtOAc 15:1); **IR** (ATR): ν = 2934, 2835, 1519, 1477, 1464, 1440, 1246, 1218, 1172, 1140, 1028, 756 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.58-7.52 (m, 1H), 7.32-7.27 (m, 3H), 6.93 (d, $^3J_{HH}$ = 8.1 Hz, 1H), 6.90-6.86 (m, 2H), 6.45 (d, $^3J_{HH}$ = 15.8 Hz, 1H), 6.09 (dt, $^3J_{HH}$ = 15.8, 7.0 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H), 3.55 (t, $^3J_{HH}$ = 6.6 Hz, 2H), 2.30 (qd, $^3J_{HH}$ = 7.1, $^4J_{HH}$ = 1.5 Hz, 2H), 1.90 (dq, $^3J_{HH}$ = 8.0, 6.7 Hz, 2H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.3, 148.1, 140.1, 135.6, 133.7, 130.3, 130.1, 129.4, 127.2, 127.0, 125.9, 122.0, 113.1, 110.8, 55.9, 55.9, 44.3, 32.1, 30.2; **HR-MS** (EI): $[C_{19}H_{21}O_2Cl]^+$ ($[M]^+$): calcd.: 316.1230, obs.: 316.1225.

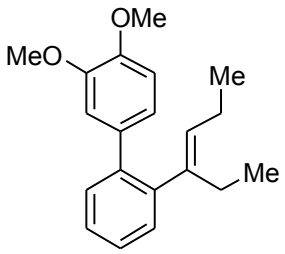
(*E*)-3',4'-Dimethoxy-2-(4-methoxystyryl)-1,1'-biphenyl



Following general procedure A: 2-bromo-3',4'-dimethoxy-1,1'-biphenyl (400 mg, 1.36 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (79 mg, 68.0 µmol, 0.05 eq.), K_2CO_3 (566 mg, 4.09 mmol, 3.00 eq.), ((*E*)-(3-methoxy-1-propen-1-yl)boronic acid (317 mg, 1.77 mmol, 1.30 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 15:1→5:1; yield: 369 mg, 1.06 mmol, 78%, light yellow solid.

DC: R_f = 0.24 (Hexane/EtOAc 5:1); **mp.:** 91°C; **IR** (ATR): ν = 2932, 2834, 1605, 1509, 1462, 1440, 1247, 1219, 1173, 1140, 1028, 819, 755 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.80-7.65 (m, 1H), 7.41-7.28 (m, 5H), 7.00 (d, J = 1.8 Hz, 2H), 6.95 (s, 3H), 6.92-6.80 (m, 2H), 3.95 (s, 3H), 3.84 (s, 3H), 3.81 (s, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 159.1, 148.3, 148.0, 140.5, 135.7, 133.6, 130.4, 130.0, 128.7, 127.6, 127.2, 127.0, 125.8, 125.6, 122.1, 114.0, 113.2, 110.8, 56.9, 55.9, 55.3; **HR-MS** (EI): $[C_{23}H_{22}O_3]^+$ ($[M]^+$): calcd.: 346.1569, obs.: 346.1570.

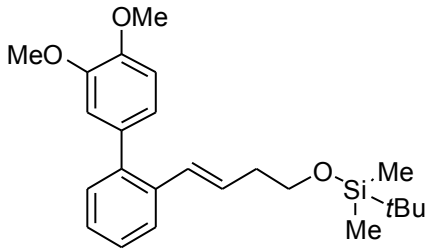
(*E*)-2-(Hex-3-en-3-yl)-3',4'-dimethoxy-1,1'-biphenyl



Following general procedure A: 2-bromo-3',4'-dimethoxy-1,1'-biphenyl (400 mg, 1.36 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (79 mg, 68.0 µmol, 0.05 eq.), K_2CO_3 (566 mg, 4.09 mmol, 3.00 eq.), (*Z*)-2-(Hex-3-en-3-yl)benzo[d][1,3,2]dioxaborole (358 mg, 1.77 mmol, 1.30 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 30:1; yield: 295 mg, 996 µmol, 73%, colorless oil. Reaction time = 64 h.

DC: R_f = 0.32 (Hex/EtOAc 15:1); **IR** (ATR): ν = 2962, 1519, 1464, 1406, 1324, 1245, 1213, 1172, 1140, 1030, 859, 810, 757 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.29-7.22 (m, 3H), 7.20-7.17 (m, 1H), 7.00 (d, $^3J_{HH}$ = 2.0 Hz, 1H), 6.96 (dd, $^3J_{HH}$ = 8.2, 2.0 Hz, 1H), 6.85 (d, $^3J_{HH}$ = 8.2 Hz, 1H), 5.42 (t, $^3J_{HH}$ = 7.3 Hz, 1H), 3.90 (s, 3H), 3.82 (s, 3H), 2.10 (dq, $^3J_{HH}$ = 7.5 Hz, 2H), 1.87 (q, $^3J_{HH}$ = 7.5 Hz, 2H), 0.99 (t, $^3J_{HH}$ = 7.5 Hz, 3H), 0.69 (t, $^3J_{HH}$ = 7.5 Hz, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.1, 147.8, 143.2, 142.9, 139.3, 135.0, 132.0, 130.6, 129.7, 126.7, 126.7, 121.1, 112.5, 110.7, 55.8, 55.7, 23.7, 21.4, 14.3, 13.1; **HR-MS** (ESI): $[C_{20}H_{24}O_2Na]^+$ ([M+Na]$^+$): calcd.: 319.1669, obs.: 319.1672.

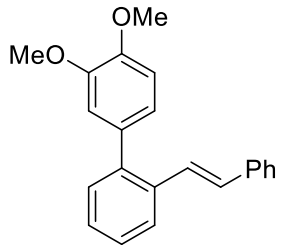
(*E*)-*tert*-butyl((4-(3',4'-dimethoxy-[1,1'-biphenyl]-2-yl)but-3-en-1-yl)oxy)dimethyl-silane



Following general procedure A: 2-bromo-3',4'-dimethoxy-1,1'-biphenyl (400 mg, 1.36 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (79 mg, 68.0 µmol, 0.05 eq.), K_2CO_3 (566 mg, 4.09 mmol, 3.00 eq.), (*E*)-*tert*-butyldimethyl ((4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-en-1-yl)oxy)silane (556 mg, 1.77 mmol, 1.30 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 30:1; yield: 432 mg, 1.08 mmol, 79%, colorless oil. Reaction time = 64 h

DC: R_f = 0.36 (Hex/EtOAc 15:1); **IR** (ATR): ν = 2930, 2856, 1519, 1463, 1406, 1247, 1218, 1172, 1140, 1097, 1030, 835, 775, 755 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.55 (d, $^3J_{HH}$ = 7.2 Hz, 1H), 7.31-7.22 (m, 3H), 6.90 (d, $^3J_{HH}$ = 8.1 Hz, 1H), 6.88 (d, $^4J_{HH}$ = 1.8 Hz, 1H), 6.89-6.84 (m, 1H), 6.44 (d, $^3J_{HH}$ = 15.9 Hz, 1H), 6.14 (dt, $^3J_{HH}$ = 15.8, 7.0 Hz, 1H), 3.92 (s, 3H), 3.86 (s, 3H), 3.66 (t, $^3J_{HH}$ = 6.8 Hz, 2H), 2.34 (dq, $^3J_{HH}$ = 6.9, 1.5 Hz, 2H), 0.87 (s, 9H), 0.03 (s, 6H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.3, 148.0, 140.1, 135.8, 133.8, 130.7, 130.0, 127.8, 127.2, 126.9, 125.8, 122.0, 113.2, 110.7, 63.1, 55.9, 55.8, 36.8, 25.9, 18.3, -5.3; **^{29}Si-NMR** (99.4 MHz, $CDCl_3$) = 19.02; **HR-MS** (ESI): $[C_{24}H_{34}O_3SiNa]^+$ ([M+Na]$^+$): calcd.: 421.2169, obs.: 421.2162.

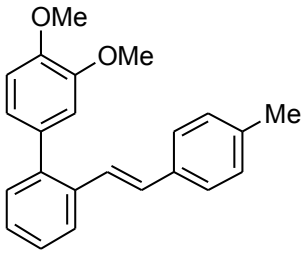
(*E*)-2-(Hex-3-en-3-yl)-3',4'-dimethoxy-1,1'-biphenyl



2-Bromo-3',4'-dimethoxy-1,1'-biphenyl (200 mg, 682 µmol, 1.00 eq.), $Pd(PPh_3)_4$ (39 mg, 31.1 µmol, 0.05 eq.), K_2CO_3 (283 mg, 2.05 mmol, 3.00 eq.), (*E*)-styrylboronic acid (111 mg, 750 µmol, 1.30 eq.), PhMe/EtOH/H_2O (3:2:1, 5.25 mL); eluting with Pentane/EtOAc 30:1; yield: 112 mg, 354 µmol, 52 %, colorless oil. Reaction time = 24 h

DC: R_f = (Hex/EtOAc 15:1); **IR** (ATR): ν = 2933, 2832, 1518, 1462, 1440, 1246, 1218, 1171, 1139, 1026, 966, 857, 812, 758, 692, 599 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.75 (m, 1H), 7.42 – 7.20 (m, 8H), 7.16 (d, $^3J_{HH}$ = 16.4 Hz, 1H), 7.02 (d, $^3J_{HH}$ = 16.3 Hz, 1H), 6.94 (m, 3H), 3.94 (s, 3H), 3.83 (s, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.3, 148.1, 140.8, 137.5, 135.4, 133.5, 130.0, 129.1, 128.6, 127.9, 127.4, 127.4, 127.3, 126.4, 125.8, 122.1, 113.2, 110.9, 56.0, 55.9; **HR-MS** (EI): $[C_{22}H_{20}O_2]^+$ ($[M]^+$): calcd.:316.1463, obs.: 316.1460.

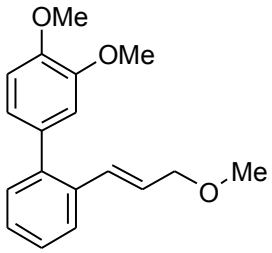
(*E*)-3',4'-Dimethoxy-2-(4-methylstyryl)-1,1'-biphenyl



Following general procedure A: (*E*)-1-bromo-2-(4-methylstyryl)benzene (341 mg, 1.25 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (72 mg, 62.4 µmol, 0.05 eq.), K_2CO_3 (518 mg, 3.74 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (363 mg, 1.37 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3.0 mL); eluting with pentane/EtOAc 30:1→15:1; yield: 284 mg, 859 µmol, 69%, yellow oil.

DC: R_f = 0.29 (pentane/EtOAc 15:1); **IR** (ATR): ν = 2932, 2833, 1511, 1462, 1439, 1245, 1218, 1171, 1026, 967, 806, 754, 731 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.79-7.71 (m, 1H), 7.42-7.27 (m, 4H), 7.20-7.03 (m, 3H), 7.02-6.91 (m, 3H), 3.96 (s, 3H), 3.85 (s, 3H), 2.35 (s, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.3, 148.0, 140.6, 137.2, 135.5, 134.7, 133.6, 130.0, 129.2, 129.1, 127.2, 127.2, 126.9, 126.3, 125.7, 122.1, 113.2, 110.8, 55.9, 55.9, 21.3; **HR-MS** (EI): $[C_{23}H_{22}O_2]^+$ ($[M]^+$): calcd.: 330.1620, obs.: 330.1621.

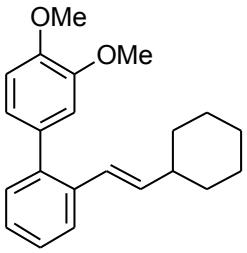
(*E*)-3',4'-Dimethoxy-2-(3-methoxyprop-1-en-1-yl)-1,1'-biphenyl



Following general procedure A: (*E*)-1-bromo-2-(3-methoxyprop-1-en-1-yl)benzene (369 mg, 1.62 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (94 mg, 81.2 µmol, 0.05 eq.), K_2CO_3 (673 mg, 4.87 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (472 mg, 1.79 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3.0 mL); eluting with pentane/EtOAc 30:1→10:1; yield: 282 mg, 992 µmol, 61%, yellow oil.

DC: R_f = 0.24 (pentane/EtOAc 15:1); **IR** (ATR): ν = 2931, 2832, 1518, 1440, 1406, 1324, 1244, 1216, 1138, 1025, 969, 754 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.61-7.57 (m, 1H), 7.34-7.26 (m, 3H), 6.90 (t, $^3J_{HH}$ = 8.1 Hz, 1H), 6.88-6.85 (m, 2H), 6.61 (d, $^3J_{HH}$ = 15.9 Hz, 1H), 6.20 (dt, $^3J_{HH}$ = 15.9, 6.2 Hz, 1H), 3.99 (dd, $^3J_{HH}$ = 6.3 Hz, $^4J_{HH}$ = 1.4 Hz, 2H), 3.92 (s, 3H), 3.86 (s, 3H), 3.31 (s, 3H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.3, 148.1, 140.6, 134.8, 133.5, 132.0, 130.1, 127.5, 127.3, 126.7, 126.2, 122.0, 113.2, 110.7, 73.3, 57.9, 55.9, 55.9; **HR-MS** (ESI): $[C_{18}H_{20}O_3Na]^+$ ($[M+Na]^+$): calcd.: 307.1306, obs.: 307.1305.

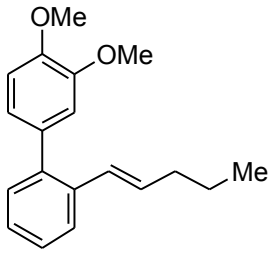
(*E*)-2-(2-Cyclohexylvinyl)-3',4'-dimethoxy-1,1'-biphenyl



Following general procedure A: (*E*)-1-bromo-2-(2-cyclohexylvinyl)benzene (339 mg, 1.28 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (74 mg, 64.0 µmol, 0.05 eq.), K_2CO_3 (532 mg, 3.85 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (373 mg, 1.41 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3.0 mL); eluting with pentane/EtOAc 15:1; yield: 252 mg, 781 µmol, 61%, colorless oil.

DC: R_f = (pentane/EtOAc 15:1); **IR** (ATR): ν = 2923, 2849, 1519, 1463, 1441, 1246, 1217, 1172, 1139, 1028, 968, 755 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.60-7.53 (m, 1H), 7.35-7.22 (m, 3H), 6.98-6.87 (m, 3H), 6.36 (dd, $^3J_{HH}$ = 16.0 Hz, $^4J_{HH}$ = 0.9 Hz, 1H), 6.09 (dd, $^3J_{HH}$ = 15.9, 6.9 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H), 2.05 (q, $^3J_{HH}$ = 3.3 Hz, 1H), 1.77-1.60 (m, 5H), 1.34-1.21 (m, 2H), 1.21-1.06 (m, 3H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.1, 147.9, 140.0, 137.5, 136.2, 133.9, 130.0, 127.2, 126.7, 126.5, 125.9, 121.9, 113.3, 110.7, 55.9, 55.8, 41.2, 33.0, 26.1, 26.0; **HR-MS** (ESI): $[C_{22}H_{27}O_2]^+$ ($[M+H]^+$): calcd.: 323.2006, obs.: 323.2006.

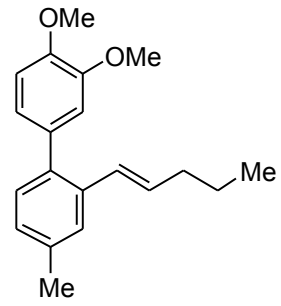
(*E*)-3',4'-Dimethoxy-2-(pent-1-en-1-yl)-1,1'-biphenyl



Following general procedure A: (*E*)-1-bromo-2-(pent-1-en-1-yl)benzene (338 mg, 1.50 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (87 mg, 75.0 µmol, 0.05 eq.), K_2CO_3 (622 mg, 4.50 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (435 mg, 1.65 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 15:1; yield: 227 mg, 803 µmol, 54%, colorless liquid.

DC: R_f = 0.29 (Hex:EtOAc 15:1); **IR** (ATR): ν = 2959, 2931, 2834, 1519, 1477, 1463, 1440, 1246, 1217, 1172, 1139, 1028, 969, 755 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) =7.57 (d, $^3J_{HH}$ = 7.6 Hz, 1H), 7.34-7.22 (m, 3H), 6.97-6.85 (m, 3H), 6.39 (d, $^3J_{HH}$ = 15.8 Hz, 1H), 6.15 (dt, $^3J_{HH}$ = 15.7, 6.9 Hz, 1H), 3.94 (s, 3H), 3.87 (s, 3H), 2.12 (qd, $^3J_{HH}$ = 7.1, $^4J_{HH}$ = 1.5 Hz, 2H), 1.44 (h, $^3J_{HH}$ = 7.3 Hz, 2H), 0.92 (t, $^3J_{HH}$ = 7.4 Hz, 3H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.2, 148.0, 139.9, 136.0, 133.9, 131.7, 130.0, 129.1, 127.2, 126.7, 125.9, 121.9, 113.2, 110.7, 55.9 55.8, 35.2, 22.6, 13.7; **HR-MS** (EI): $[C_{19}H_{22}O_2]^+$ ($[M]^+$): calcd.: 282.1620, obs.: 282.1633.

(*E*)-3',4'-Dimethoxy-4-methyl-2-(pent-1-en-1-yl)-1,1'-biphenyl

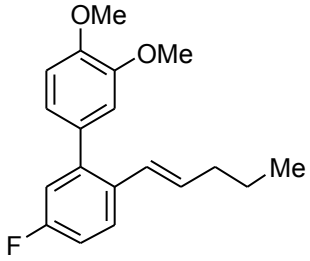


Following general procedure A: (*E*)-4-bromo-3-(pent-1-en-1-yl)toluene (359 mg, 1.50 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (87 mg, 75.0 µmol, 0.05 eq.), K_2CO_3 (622 mg, 4.50 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (435 mg, 1.65 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 6 mL); eluting with pentane/EtOAc 30:1; yield: 192 mg, 648 µmol, 43%, colorless liquid.

DC: R_f = 0.22 (Hex:EtOAc 15:1); **IR** (ATR): ν = 2956, 2931, 2870, 2834, 1518, 1463, 1248, 1225, 1167, 1139, 1029, 970, 808, 763 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.47 (d, $^3J_{HH}$ = 7.9 Hz, 1H), 7.11 (d, $^3J_{HH}$ = 7.9 Hz, 1H), 7.09 (s, 1H), 6.95-6.86 (m, 3H), 6.35 (d, $^3J_{HH}$ = 15.8 Hz, 1H), 6.10 (dt, $^3J_{HH}$ = 15.7, 6.9 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H), 2.36 (s, 3H), 2.10 (qd, $^3J_{HH}$ = 7.1, $^4J_{HH}$ = 1.5 Hz, 2H), 1.44 (h, $^3J_{HH}$ = 7.3 Hz, 2H), 0.91 (t, $^3J_{HH}$ = 7.4 Hz, 3H); **^{13}C-NMR** (126 MHz,

$CDCl_3$): δ (ppm) = 148.2, 147.9, 139.8, 136.4, 134.0, 133.2, 130.8, 130.6, 128.8, 128.0, 125.8, 121.9, 113.2, 110.7, 55.9, 55.8, 35.2, 22.6, 21.1, 13.7.; **HR-MS** (EI): $[C_{20}H_{24}O_2]^+$ ($[M]^+$): calcd.: 296.1776, obs.: 296.1784.

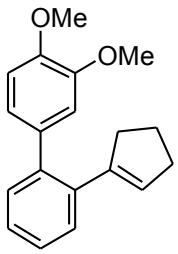
(*E*)-5-Fluoro-3',4'-dimethoxy-2-(pent-1-en-1-yl)-1,1'-biphenyl



Following general procedure A: (E)-2-bromo-4-fluoro-1-(pent-1-en-1-yl)benzene (429 mg, 1.76 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (102 mg, 88.2 µmol, 0.05 eq.), K_2CO_3 (732 mg, 5.29 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaboro-lane (513 mg, 1.94 mmol, 1.10 eq.), PhMe/EtOH/ H_2O (3:2:1, 9 mL); eluting with pentane/EtOAc 30:1; yield: 380 mg, 1.27 mmol, 72%, colorless oil.

DC: R_f = 0.38 (Hex:EtOAc 15:1); **IR** (ATR): ν = 2957, 1605, 1573, 1518, 1483, 1464, 1324, 1248, 1228, 1164, 1140, 1029, 970, 859, 810, 764 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.57-7.45 (m, 1H), 7.03-6.86 (m, 5H), 6.32 (dt, $^3J_{HH}$ = 15.7 Hz, $^4J_{HH}$ = 1.4 Hz, 1H), 6.07 (dt, $^3J_{HH}$ = 15.7, 6.9 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H), 2.16-2.04 (m, 2H), 1.44 (h, $^3J_{HH}$ = 7.3 Hz, 2H), 0.92 (t, $^3J_{HH}$ = 7.3 Hz, 3H).; **^{13}C-NMR** (101 MHz, $CDCl_3$): δ (ppm) = 161.53 (d, $^1J_{CF}$ = 245.9 Hz), 148.36 , 141.61 (d, $^3J_{CF}$ = 7.6 Hz), 132.80 (d, $^5J_{CF}$ = 1.9 Hz), 132.27 (d, $^4J_{CF}$ = 3.2 Hz), 131.42 (d, J = 1.8 Hz), 128.19 , 127.60 (d, $^3J_{CF}$ = 8.2 Hz), 121.87 , 116.36 (d, $^2J_{CF}$ = 21.3 Hz), 114.05 (d, $^2J_{CF}$ = 21.1 Hz), 113.01 , 110.82 , 55.89, 55.9, 35.13 , 22.57 , 13.71 . **^{19}F-NMR** (282 MHz, $CDCl_3$) δ -116.30--116.45 (m); **HR-MS** (ESI): $[C_{19}H_{21}O_2FNa]^+$ ($[M+Na]^+$): calcd.: 323.1418, obs.: 323.1421.

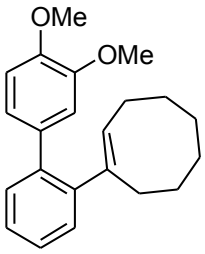
2-(Cyclopent-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl



(1-Bromo-2-(cyclopent-1-en-1-yl)benzene (102 mg, 457 µmol, 1.00 eq.), $Pd(PPh_3)_4$ (26 mg, 23.0 µmol, 0.05 eq.), K_2CO_3 (190 mg, 1.37 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (133 mg, 503 µmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3.5 mL); eluting with pentane/EtOAc 15:1; yield: 90 mg, 321 µmol, 70%, colorless oil.

DC: R_f = 0.39 (pentane/EtOAc 15:1); **IR** (ATR): ν = 2949, 2836, 1519, 1463, 1439, 1406, 1246, 1213, 1171, 1139, 1028, 756 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.35-7.26 (m, 4H), 6.96-6.86 (m, 3H), 5.67 (p, $^3J_{HH}$ = 2.3 Hz, 1H), 3.92 (s, 3H), 3.84 (s, 3H), 2.41-2.32 (m, 2H), 2.19-2.11 (m, 2H), 1.79 (p, $^3J_{HH}$ = 7.4 Hz, 2H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.2, 147.9, 144.7, 139.9, 137.6, 135.1, 130.0, 129.7, 129.0, 126.9, 126.8, 121.0, 112.4, 110.7, 55.8, 55.8, 35.5, 33.2, 24.3; **HR-MS** (ESI): $[C_{19}H_{21}O_2]^+$ ($[M+H]^+$): calcd.: 281.1536, obs.: 281.1537.

(*E*)-2-(Cyclooct-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl

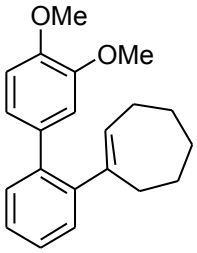


Following general procedure A: (1-bromo-2-(cyclooct-1-en-1-yl) benzene (300 mg, 1.13 mmol, 1.00 eq.), $Pd(PPh_3)_4$ (65 mg, 56.6 µmol, 0.05 eq.), K_2CO_3 (469 mg, 3.39 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (327 mg, 1.24 mmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 3 mL); eluting with pentane/EtOAc 30:1; yield: 221 mg, 685 µmol, 61%, colorless oil.

DC: R_f = 0.24 (pentane/EtOAc 30:1); **IR** (ATR): ν = 2922, 2849, 1519, 1465, 1439, 1248, 1214, 1172, 1141, 1029, 757 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) =7.34-

7.17 (m, 4H), 7.09 (d, $^3J_{HH}$ = 2.0 Hz, 1H), 7.01 (dd, $^3J_{HH}$ = 8.2, 2.0 Hz, 1H), 6.88 (d, $^3J_{HH}$ = 8.3 Hz, 1H), 5.77 (t, $^3J_{HH}$ = 8.2 Hz, 1H), 3.92 (s, 3H), 3.84 (s, 3H), 2.22 (td, $^3J_{HH}$ = 8.0, 4.7 Hz, 2H), 2.03-1.91 (m, 2H), 1.64-1.40 (m, 6H), 1.31-1.21 (m, 2H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.1, 147.8, 143.4, 143.3, 138.8, 134.8, 130.2, 129.9, 129.6, 126.8, 126.6, 121.0, 112.6, 110.8, 55.9, 55.8, 30.1, 30.0, 28.3, 27.0, 26.7, 26.5.; **HR-MS** (ESI): $[C_{22}H_{27}O_2]^+$ ($[M+H]^+$): calcd.: 323.2006, obs.: 323.2005.

2-(Cyclohept-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl

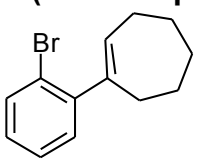


Following general procedure A: (1-bromo-2-(cyclohept-1-en-1-yl) benzene (222 mg, 884 µmol, 1.00 eq.), $Pd(PPh_3)_4$ (51 mg, 44.0 µmol, 0.05 eq.), K_2CO_3 (366 mg, 2.65 mmol, 3.00 eq.), 2-(3,4-dimethoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (257 mg, 957 µmol, 1.10 eq.), PhMe/EtOH/H_2O (3:2:1, 7 mL); eluting with pentane/EtOAc 30:1; yield: 183 mg, 593 µmol, 67%, colorless oil. Reaction time = 20 h.

DC: R_f = 0.59 (pentane/EtOAc 15:1); **IR** (ATR): ν = 2920, 2846, 1519, 1475, 1439, 1247, 1214, 1172, 1029, 756 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.30-7.21 (m, 4H), 7.00 (d, $^3J_{HH}$ = 2.0 Hz, 1H), 6.97 (dd, $^3J_{HH}$ = 8.2, 2.0 Hz, 1H), 6.89 (d, $^3J_{HH}$ = 8.2 Hz, 1H), 5.93 (t, $^3J_{HH}$ = 6.5 Hz, 1H), 3.93 (s, 3H), 3.87 (s, 3H), 2.26-2.17 (m, 2H), 1.99 (m, 2H), 1.69-1.62 (m, 2H), 1.54-1.47 (m, 2H), 1.31-1.23 (m, 2H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 148.2, 147.8, 147.0, 145.3, 138.9, 135.1, 131.8, 129.8, 129.5, 126.8, 126.7, 121.3, 112.7, 110.7, 55.8, 55.7, 34.8, 32.5, 29.2, 26.9, 26.5; **HR-MS** (EI): $[C_{21}H_{24}O_2]^+$ ($[M]^+$): calcd.: 308.1776, obs.: 308.1773.

General procedure B:[2] synthesis of 1-(2-bromophenyl)cycloalkenes: Under an argon atmosphere 1-bromo-2-iodobenzene (616 µL, 1.35 g, 4.80 mmol, 1.00 equiv) in dry THF (20 mL) was cooled to –40 °C and treated with isopropylmagnesium chloride (2 M in THF, 2 mL, 4 mmol, 0.8 equiv) and stirred at –20 °C for 1.5 h. Then the mixture is cooled to –40 °C, the ketone (9.8 mmol) was added, and the reaction was slowly warmed to room temperature over 16 h. Then water was added, the phases were separated, the aqueous phase was extracted with DCM (3 x 20 mL), the combined organic phases were dried over Na_2SO_4, and the solution was concentrated to dryness. The alcohol was obtained by passing it through a short plug of silica (pentane→pentane : EtOAc) and directly used without further purification. The alcohol was dissolved in toluene (20 mL), treated with *p*-toluenesulfonic acid (4.80 mmol) and stirred at 110 °C for 16 h. Water was added, the phases were separated and the aqueous phase was extracted with DCM (3 x 20 mL). The combined organic phases were washed with sat. aq. $NaHCO_3$-soln. (2 x 20 mL), dried over Na_2SO_4 and concentrated. Column chromatography (SiO_2, pentane) provided the 1-(2-bromophenyl)cycloalkenes as colorless liquids.

1-(2-Bromophenyl)cyclohept-1-ene

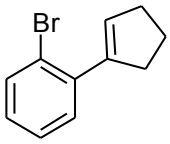


Cyclohepatone (1.13 mL, 1.08 g, 9.60 mmol, 2.00 equiv);
Yield: 230 mg, 916 µmol, 19%

DC: R_f = 0.90 (pentane); **IR** (ATR): ν = 2918, 2848, 1464, 1433, 1023, 853, 750, cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.52 (dd, $^3J_{HH}$ = 7.9 Hz, $^4J_{HH}$ = 1.2 Hz, 1H), 7.22 (ddd, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 1.3 Hz, 1H), 7.15 (dd, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 2.0 Hz, 1H), 7.06 (ddd, $^3J_{HH}$ = 7.9, 7.3 Hz, $^4J_{HH}$ = 2.0 Hz, 1H), 5.81 (t, $^3J_{HH}$ = 6.5 Hz, 1H), 2.52-2.43 (m, 2H), 2.38-2.21 (m, 2H), 1.89-1.76 (m, 2H), 1.65 (m, 4H); **^{13}C-NMR** (76 MHz, $CDCl_3$): δ (ppm) = 147.24, 145.71, 132.54,

132.44, 130.23, 127.70, 127.03, 122.17, 34.51, 32.44, 28.97, 27.00, 26.91; **HR-MS** (EI): $[C_{13}H_{15}Br]^+$ ($[M]^+$): calcd.: 250.0357, obs.: 250.0366.

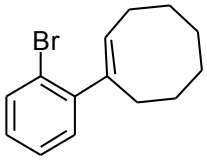
1-Bromo-2-(cyclopent-1-en-1-yl)benzene



Cyclopentanone (850 µL, 808 mg, 9.60 mmol, 2.00 equiv)
Yield: 104 mg, 466 µmol, 10%

DC: R_f = 0.90 (pentane); **IR** (ATR): ν = 2927, 2845, 1697, 1467, 1433, 1023, 750 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.61-7.51 (m, 1H), 7.27-7.21 (m, 2H), 7.08 (ddd, $^3J_{HH}$ = 7.9, 5.8, 3.3 Hz, 1H), 5.97 (p, $^3J_{HH}$ = 2.2 Hz, 1H), 2.79-2.69 (m, 2H), 2.58-2.49 (m, 2H), 2.09-1.96 (m, 2H).; **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 142.9, 139.6, 133.0, 131.0, 129.8, 127.9, 126.9, 122.1, 36.2, 33.5, 23.9; **HR-MS** (EI): $[C_{11}H_{11}Br]^+$ ($[M]^+$): calcd.: 222.0044, obs.: 222.0039.

(*E*)-1-(2-Bromophenyl)cyclooct-1-ene

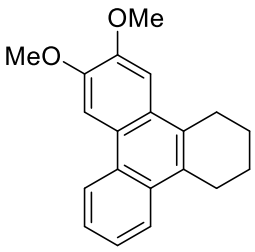


Cycloctanone (1.21 g, 9.60 mmol, 2.00 equiv)
Yield: 72 mg, 272 µmol, 6%

DC: R_f = 0.90 (pentane); **IR** (ATR): ν = 2920, 2849, 1465, 1446, 1022, 751 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.54 (dd, $^3J_{HH}$ = 7.9 Hz, $^4J_{HH}$ = 1.3 Hz, 1H), 7.24 (ddd, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 1.3 Hz, 1H), 7.15 (dd, $^3J_{HH}$ = 7.6, 2.0 Hz, 1H), 7.08 (ddd, $^3J_{HH}$ = 7.6, 1.9 Hz, 1H), 5.60 (t, $^3J_{HH}$ = 8.2 Hz, 1H), 2.61-2.49 (m, 2H), 2.36-2.23 (m, 2H), 1.68-1.56 (m, 6H), 1.54-1.43 (m, 2H); **^{13}C-NMR** (76 MHz, $CDCl_3$): δ (ppm) = 145.5, 141.7, 132.6, 130.6, 130.1, 127.9, 126.9, 122.5, 30.0, 29.8, 28.1, 26.7, 26.6, 26.5; **HR-MS** (EI): $[C_{14}H_{17}Br]^+$ ($[M]^+$): calcd.: 264.0514, obs.: 264.0511.

General Procedure C: Synthesis of Phenanthrenes 2: To a solution of the alkene (1.0 equiv) in $MeNO_2$ (0.3 M) with activated molecular sieves (4 Å, powder, spatula tip) was added a solution of PIFA (1.3 equiv) in $MeNO_2$ (0.19 M) at 10 °C *via* syringe pump (0.25 ml/h). The reaction was stirred for a total of 16 h. Sat. aq. $Na_2S_2O_3$ solution was added and the reaction was extracted with DCM (3×20 mL). The combined organic layers were dried over Na_2SO_4. Column chromatography furnished the cyclization products **2**.

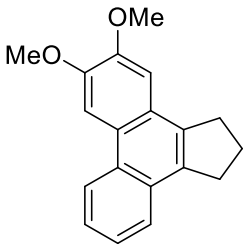
6,7-Dimethoxy-1,2,3,4-tetrahydrotriphenylene (2a)



Phenanthrene **2a** was synthesized following the general procedure C using the corresponding alkene (100 mg, 340 µmol, 1.00 equiv) and PIFA (175 mg, 442 µmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a brown solid (77 mg, 265 µmol, 78%).

R_f = 0.42 (pentane/EtOAc 20:1); **IR** (ATR): ν (cm^{-1}) = 2927, 2832, 1612, 1524, 1503, 1460, 1434, 1397, 1253, 1205, 1164, 1135, 1046, 1022, 844, 824, 766, 749, 718, 629, 605; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.55 (m, 1H), 8.12-7.98 (m, 2H), 7.64-7.47 (m, 2H), 7.39 (s, 1H), 4.12 (s, 3H), 4.06 (s, 3H), 3.24-3.02 (m, *J* = 14.9 Hz, 4H), 2.01 (t, *J* = 3.0 Hz, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.1, 148.3, 131.2, 129.5, 128.7, 128.7, 127.0, 125.5, 125.1, 123.8, 123.4, 122.2, 103.9, 103.6, 55.9, 55.8, 27.2, 26.7, 23.0; **HR-EI-MS**: $[C_{20}H_{20}O_2]^+$ ($[M]^+$): calcd.: 292.1462, obs.: 292.1468; **Mp**. = 155 °C.

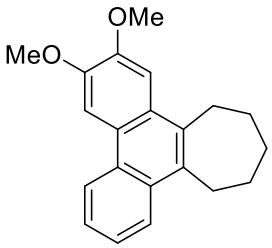
5,6-Dimethoxy-2,3-dihydro-1H-cyclopenta[l]phenanthrene (2b)



Phenanthrene **2b** was synthesized following the general procedure C using the corresponding alkene (90 mg, 321 μmol, 1.00 equiv) and PIFA (166 mg, 444 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (68 mg, 244 μmol, 76%).

R_f = 0.45 (pentane/EtOAc 20:1); **IR** (ATR): ν (cm^{-1}) = 2960, 2904, 2848, 1507, 1473, 1461, 1449, 1434, 1416, 1400, 1252, 1207, 1191, 1163, 1039, 1021, 844, 783, 772, 745, 619, 424; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.55 (d, $^3J_{HH}$ = 9.5 Hz, 1H), 8.06 (s, 1H), 7.86 (m, 1H), 7.65-7.51 (m, 2H), 7.19 (s, 1H), 4.12 (s, 3H), 4.06 (s, 3H), 3.34 (m, 4H), 2.37 (m, 2H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 149.3, 148.4, 136.8, 135.8, 129.6, 129.3, 125.6, 125.1, 124.5, 122.6, 105.1, 104.1, 56.0, 55.9, 32.5, 32.1, 23.5; **HR-ESI-MS**: calc. ($C_{19}H_{18}O_2Na$, $[M+Na]^+$): 301.1199; obs.: 301.1206, **Mp** = 175 °C (degr.).

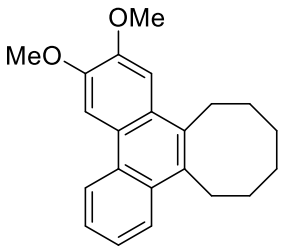
2,3-Dimethoxy-10,11,12,13-tetrahydro-9*H*-cyclohepta[l]phenanthrene (2c)



Phenanthrene **2c** was synthesized following the general procedure C using the corresponding alkene (100 mg, 342 μmol, 1.00 equiv) and PIFA PIFA (153 mg, 357 μmol, 1.10 equiv). Column chromatography (SiO_2, Pentane/EtOAc 20:1) delivered the product as a yellow resin (73 mg, 243 μmol, 73%).

R_f = 0.40 (pentane/EtOAc 20:1); **IR** (ATR): ν (cm^{-1}) = 2911, 2847, 1617, 1527, 1507, 1460, 1432, 1394, 1256, 1226, 1205, 1172, 1130, 1040, 1024, 845, 821, 756, 737, 719, 601, 445; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.60 (m, 1H), 8.23 (m, 1H), 8.11 (s, 1H), 7.65-7.54 (m, 3H), 4.15 (s, 3H), 4.10 (s, 3H), 3.45-3.29 (m, 4H), 1.94 (m 2H), 1.86-1.71 (m, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.3, 148.3, 136.1, 135.1, 130.3, 129.0, 126.1, 125.6, 124.9, 124.3, 124.0, 122.6, 104.5, 103.8, 55.9, 55.9, 31.3, 28.5, 27.9, 26.2, 26.2; **HR-ESI-MS**: calc. ($C_{21}H_{22}O_2Na$, $[M+Na]^+$): 329.1512; obs.: 329.1512, **Mp** = 145 °C.

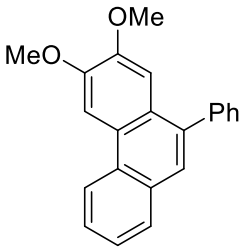
2,3-Dimethoxy-9,10,11,12,13,14-hexahydrocycloocta[l]phen-anthrene (2d)



Phenanthrene **2d** was synthesized following the general procedure C using the corresponding alkene (100 mg, 326 μmol, 1.00 equiv) and PIFA (169 mg, 424 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a white solid (80 mg, 252 μmol, 77%).

R_f = 0.39 (pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2912, 2843, 1529, 1507, 1464, 1433, 1397, 1257, 1211, 1193, 1168, 1129, 1046, 1027, 926, 913, 845, 822, 793, 755, 721, 661, 644, 600, 401; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.58 (m, 1H), 8.14 (m, 1H), 8.07 (s, 1H), 7.56 (m, 2H), 7.49 (s, 1H), 4.12 (s, 3H), 4.06 (s, 3H), 3.56-3.16 (m, 4H), 1.99-1.77 (m, 4H), 1.48-1.35 (m, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.2, 148.3, 133.3, 132.5, 130.2, 129.3, 125.9, 125.6, 125.0, 124.7, 124.5, 122.5, 105.1, 103.8, 55.9, 55.9, 30.6, 30.2, 27.7, 27.3, 26.9, 26.7; **HR-ESI-MS**: calc. ($C_{22}H_{24}O_2Na$, $[M+Na]^+$): 343.1669; obs.: 343.1668; **Mp**. = 150 °C.

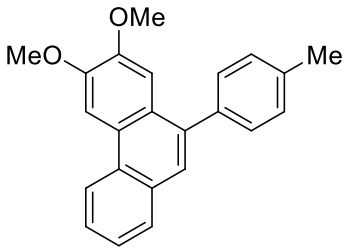
2,3-Dimethoxy-10-phenylphenanthrene (2e)



Phenanthrene **2e** was synthesized following the general procedure C using the corresponding alkene (100 mg, 316 μmol, 1.00 equiv) and PIFA (158 mg, 411 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (42 mg, 133 μmol, 42%).

R_f = 0.34 (Pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2933, 1524, 1499, 1463, 1435, 1259, 1205, 1151, 1024, 857, 760, 700, 585, 571; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.55 (d, $^3J_{HH}$ = 8.0 Hz, 1H), 8.08 (s, 1H), 7.86 (dd, $^3J_{HH}$ = 7.9 Hz, $^4J_{HH}$ = 1.4 Hz, 1H), 7.65-7.41 (m, 8H), 7.33 (m, 1H), 4.13 (s, 3H), 3.82 (s, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.2, 149.1, 141.1, 138.0, 131.1, 129.8, 129.4, 128.8, 128.4, 127.4, 126.2, 126.1, 125.9, 125.9, 122.0, 107.1, 103.5, 56.0, 55.7; **HR-EI-MS**: calc. ($C_{22}H_{18}O_2$, $[M]^+$): 314.1307; obs.: 314.1304,

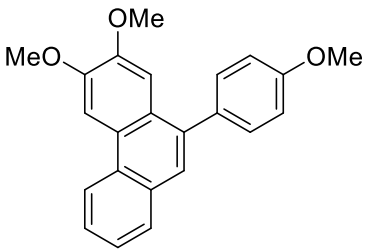
2,3-Dimethoxy-10-(p-tolyl)phenanthrene (2f)



Phenanthrene **2f** was synthesized following the general procedure C using the corresponding alkene (100 mg, 302 μmol, 1.00 equiv) and PIFA (169 mg, 393 μmol, 1.30 equiv). Column chromatography (SiO_2, Pentane/ EtOAc 20:1) delivered the product as a yellow resin (43 mg, 130 μmol, 43%, ratio of 3-methoxy- vs. 1-methoxy-isomer = 92:8).

R_f = 0.48 (pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2933, 1797, 1617, 1526, 1502, 1464, 1436, 1252, 1208, 1151, 1126, 1041, 1023, 908, 836, 820, 760, 726, 689, 646, 607, 580, 544, 515; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.61 (d, $^3J_{HH}$ = 8.3 Hz, 1H), 8.05 (s, 1H), 7.94(m, 1H), 7.68-7.56 (m, 2H), 7.47 (m, 4H), 7.37-7.29 (m, 3H), 4.15 (s, 3H), 4.05 (s, 3H), 2.48 (s, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.3, 136.8, 130.4, 130.0, 129.0, 127.0, 126.8, 126.6, 126.0, 125.4, 122.4, 108.3, 103.2, 56.0, 55.9, 21.3; **HR-ESI-MS**: calc. ($C_{23}H_{20}O_2$, $[M]^+$): 328.1463; obs.: 328.1455,

2,3-Dimethoxy-10-(4-methoxyphenyl)phenanthrene (2g)

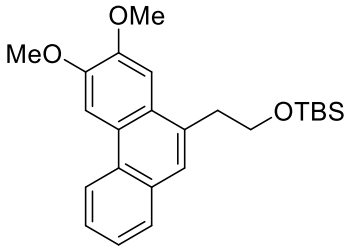


The reaction was carried out in DCM instead of $MeNO_2$. Phenanthrene **2g** was synthesized following the general procedure C using the corresponding alkene (100 mg, 289 μmol, 1.00 equiv) and PIFA (144 mg, 375 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 15:1) delivered the product as a yellow resin (44 mg, 127 μmol, 44%).

R_f = 0.34 (pentane/EtOAc 15:1); **IR**: ν (cm^{-1}) = 2933, 1502, 1463, 1436, 1287, 1242, 1210, 1160, 1026, 907, 836, 792, 726, 608, 553; **^{1}H-NMR** δ (400 MHz, $CDCl_3$) = 8.59 (dd, $^3J_{HH}$ = 8.3 Hz, $^4J_{HH}$ = 0.5 Hz, 1H), 8.02 (s, 1H), 7.92 (dd, $^3J_{HH}$ = 8.3 Hz, $^4J_{HH}$ = 1.0 Hz, 1H), 7.60 (m, 1H), 7.56 (s, 1H), 7.50-7.40 (m, 3H), 7.22 (s, 1H), 7.08-6.98 (m, 2H), 4.12 (s, 3H), 4.02 (s, 3H), 3.89 (s, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 154.5, 149.4, 147.3, 133.6, 132.3, 131.2, 131.0, 130.1, 128.0, 126.9, 126.7, 126.3, 125.6, 125.1, 122.7, 121.6, 113.7, 111.3, 107.9, 102.9, 70.6, 56.5,

56.0, 56.0, 55.9, 55.4, 55.3, 55.1; **HR-ESI-MS**: calc. ($C_{23}H_{21}O_3$, $[M+H]^+$): 345,1485; obs.: 345,1483,

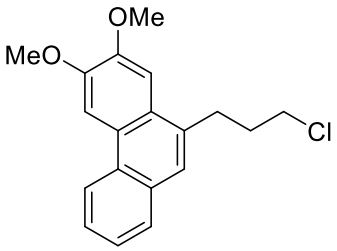
***tert*-Dutyl(2-(6,7-dimethoxyphenanthren-9-yl)ethoxy)dimethylsilane (2h)**



Phenanthrene **2h** was synthesized following the general procedure C using the corresponding alkene (100 mg, 250 μmol, 1.00 equiv) and PIFA (130 mg, 326 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a yellow resin (66 mg, 166 μmol, 66%).

R_f = 0.39 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2952, 2929, 2856, 1526, 1502, 1472, 1435, 1389, 1269, 1230, 1210, 1166, 1093, 1040, 1026, 878, 835, 806, 776, 745; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.50 (d, $^3J_{HH}$ = 8.1 Hz, 1H), 8.06 (s, 1H), 7.81 (m, 1H), 7.62-7.47 (m, 3H), 7.46 (s, 1H), 4.13 (s, 3H), 4.07 (s, 3H), 4.03 (t, $^3J_{HH}$ = 7.4 Hz, 2H), 3.33 (t, $^3J_{HH}$ = 7.5 Hz, 2H), 0.89 (s, 9H), 0.00 (s, 6H); **^{13}C-NMR** (76 MHz, $CDCl_3$): δ (ppm) = 149.2, 148.9, 132.4, 131.3, 129.2, 128.2, 126.5, 125.8, 125.7, 125.6, 125.4, 122.0, 104.7, 103.8, 63.6, 56.0, 37.2, 26.0, 18.4, –5.3; **HR-ESI-MS**: calc. ($C_{24}H_{33}O_3$, $[M+H]^+$): 397.2193; obs.: 397.2176,

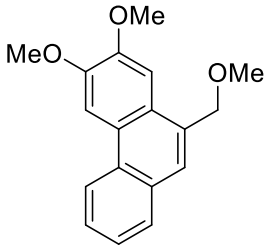
10-(3-Chloropropyl)-2,3-dimethoxyphenanthrene (2i)



Phenanthrene **2i** was synthesized following the general procedure C using the corresponding alkene (100 mg, 315 μmol, 1.00 equiv) and PIFA (157 mg, 410 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (64 mg, 202 μmol, 64%).

R_f = 0.34 (pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2934, 1527, 1502, 1462, 1435, 1389, 1266, 1230, 1209, 1166, 1038, 1023, 909, 850, 804, 778,727, 646, 570; **^{1}H-NMR** δ (400 MHz, $CDCl_3$) = 8.50 (dd, $^3J_{HH}$ = 8.2 Hz, $^4J_{HH}$ = 0.5 Hz, 1H), 8.05 (s, 1H), 7.81 (dd, $^3J_{HH}$ = 7.8, $^4J_{HH}$ = 1.4 Hz, 1H), 7.61-7.49 (m, 3H), 7.46 (s, 1H), 4.12 (s, 3H), 4.07 (s, 3H), 3.67 (t, $^3J_{HH}$ = 6.1 Hz, 2H), 3.32-3.18 (m, 2H), 2.35-2.23 (m, 2H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.3, 149.0, 134.1, 131.2, 129.2, 128.2, 126.0, 125.8, 125.7, 125.5, 125.0, 122.0, 104.7, 103.9, 77.3, 77.0, 76.7, 56.0, 55.9, 44.9, 32.8, 30.5; **HR-ESI-MS**: calc. ($C_{19}H_{19}O_2ClNa$, $[M+Na]^+$): 337.0966; obs.: 337.0954,

2,3-Dimethoxy-10-(methoxymethyl)phenanthrene (2j)

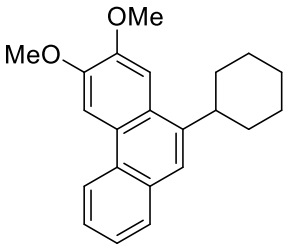


Phenanthrene **2j** was synthesized following the general procedure C using the corresponding alkene (100 mg, 352 μmol, 1.00 equiv) and PIFA (182 mg, 457 μmol, 1.30 equiv). Column chromatography (SiO_2, Pentane/EtOAc 15:1) delivered the product as a yellow resin (55 mg, 193 μmol, 55%).

R_f = 0.38 (pentane/EtOAc 15:1); **IR**: ν (cm^{-1}) = 2992, 2931, 2833, 1615, 1503, 1464, 1435, 1390, 1377, 1267, 1231, 1207, 1168, 1115, 1094, 1037, 1021, 957, 882, 853, 842, 803, 775, 743, 569, 544, 467; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.52 (d, $^3J_{HH}$ = 8.1 Hz, 1H), 8.04 (s, 1H), 7.86 (m, 1H), 7.69-7.50 (m, 3H), 4.93 (s, 2H), 4.12 (s, 3H), 4.07 (s, 3H), 3.46 (s, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$):

δ (ppm) = 149.3, 149.1, 131.0, 130.7, 130.0, 128.7, 126.4, 125.9, 125.8, 125.7, 125.5, 122.0, 105.0, 103.6, 74.0, 57.8, 55.9, 55.9; **HR-ESI-MS**: calc. ($C_{18}H_{18}O_3Na$, $[M+Na]^+$): 305.1148; obs.: 305.1145,

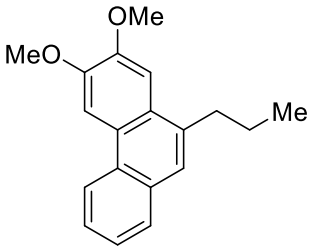
10-Cyclohexyl-2,3-dimethoxyphenanthrene (2k)



Phenanthrene **2k** was synthesized following the general procedure C using the corresponding alkene (80 mg, 261 μmol, 1.00 equiv) and PIFA (135 mg, 339 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (49 mg, 154 μmol, 59%).

R_f = 0.43 (pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2928, 2848, 1615, 1527, 1503, 1465, 1454, 1434, 1389, 1262, 1230, 1209, 1161, 1131, 1043, 1020, 888, 874, 849, 806, 781, 743, 637, 576; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.50 (d, $^3J_{HH}$ = 7.5 Hz, 1H), 8.07 (s, 1H), 7.84 (m, 1H), 7.62-7.46 (m, 4H), 4.13 (s, 3H), 4.08 (s, 3H), 3.20 (m, 1H), 2.25-2.11 (m, 2H), 2.02-1.82 (m, 3H), 1.73-1.50 (m, 5H); **^{13}C-NMR** (76 MHz, $CDCl_3$): δ (ppm) = 149.1, 148.8, 140.9, 133.2, 131.5, 131.4, 128.7, 128.5, 128.5, 125.9, 125.6, 125.5, 121.9, 121.2, 104.4, 103.9, 77.5, 77.0, 76.6, 55.9, 55.9, 55.6, 39.9, 34.0, 27.4, 26.7; **HR-ESI-MS**: calc. ($C_{22}H_{24}O_2Na$, $[M+Na]^+$): 343.1669; obs.: 343.1656

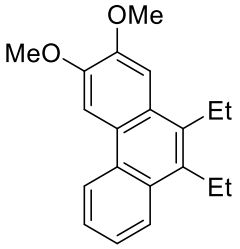
2,3-Dimethoxy-10-propylphenanthrene (2l)



Phenanthrene **2l** was synthesized following the general procedure C using the corresponding alkene (70 mg, 248 μmol, 1.00 equiv) and PIFA (128 mg, 322 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a yellow resin (47 mg, 166 μmol, 67%).

R_f = 0.43 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2956, 1616, 1528, 1503, 1465, 1436, 1340, 1269, 1230, 1209, 1166, 1039, 851, 805, 746; **^{1}H-NMR** δ (500 MHz, $CDCl_3$) = 8.48 (d, $^3J_{HH}$ = 8.2 Hz, 1H), 8.04 (s, 1H), 7.79 (dd, $^3J_{HH}$ = 7.8 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 7.58-7.46 (m, 3H), 7.41 (s, 1H), 4.11 (s, 3H), 4.05 (s, 3H), 3.07-3.00 (m, 2H), 1.92-1.78 (m, 2H), 1.06 (t, $^3J_{HH}$ = 7.4 Hz, 3H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ (ppm) = 149.0, 148.8, 135.8, 131.4, 129.0, 128.1, 126.4, 125.6, 125.5, 125.4, 124.4, 121.9, 104.8, 103.8, 55.9, 55.9, 35.7, 22.9, 14.3; **HR-ESI-MS**: calc. ($C_{19}H_{20}O_2Na$, $[M+Na]^+$): 303.1356; obs.: 33.1354.

9,10-Diethyl-2,3-dimethoxyphenanthrene (2m)

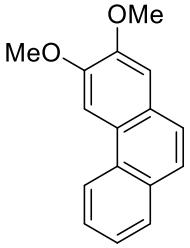


Phenanthrene **2m** was synthesized following the general procedure C using the corresponding alkene (100 mg, 337 μmol, 1.00 equiv) and PIFA (175 mg, 439 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (75 mg, 253 μmol, 75%).

R_f = 0.43 (pentane/EtOAc 20:1); **^{1}H-NMR** δ (300 MHz, $CDCl_3$) 8.58 (m, 1H), 8.13 (m 1H), 8.07 (s, 1H), 7.64-7.54 (m, 2H), 7.49 (s, 1H), 4.14 (s, 3H), 4.09 (s, 3H), 3.27-3.11 (m, 4H), 1.39 (m, 6H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 149.2, 148.4, 134.1, 133.3, 130.4, 129.3, 126.2, 125.6, 125.1, 124.7, 124.5, 122.5, 105.2, 103.8, 77.4, 77.2, 77.0, 76.6, 55.9, 55.9, 22.5, 22.1, 15.2, 14. 8; **IR**: ν (cm^{-1}) = 2964,

1617, 1530, 1505, 1464, 1435, 1394, 1259, 1223, 1204, 1167, 1079, 1044, 1026, 847, 796, 758, 729; **HR-ESI-MS**: calc. ($C_{20}H_{22}O_2Na$, $[M+Na]^+$): 317.1512; obs.: 317.1512,

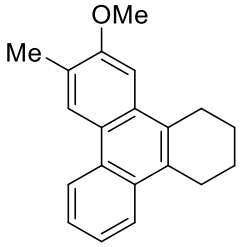
2,3-Dimethoxyphenanthrene (2n)



Phenanthrene **2n** was synthesized following the general procedure C using the corresponding alkene (100 mg, 449 μmol, 1.00 equiv) and PIFA (231 mg, 584 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 15:1) delivered the product as a white resin (42 mg, 176 μmol, 39%).

R_f = 0.38 (pentane/EtOAc 15:1); **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.54 (dd, *J* = 8.3, 0.5 Hz, 1H), 8.02 (s, 1H), 7.88 (ddd, *J* = 7.9, 1.4, 0.5 Hz, 1H), 7.66 (s, 1H), 7.66 (s, 2H), 7.63 (ddd, *J* = 8.4, 5.3, 1.5 Hz, 1H), 7.54 (ddd, *J* = 8.0, 5.5, 1.2 Hz, 1H), 4.13 (s, 3H), 4.05 (s, 3H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 149.3, 131.3, 129.7, 128.7, 127.1, 126.2, 125.9, 125.5, 125.2, 124.8, 122.1, 108.3, 103.3, 56.0, 55.9; **IR**: ν (cm^{-1}) = 3001, 2961, 1615, 1523, 1506, 1462, 1435, 1391, 1373, 1268, 1219, 1193, 1155, 1105, 1037, 1022, 855, 801, 779, 743; **HR-EI-MS**: $[C_{16}H_{14}O_2]^+$ ($[M]^+$): calcd.: 238.0994, obs.: 238.0997.

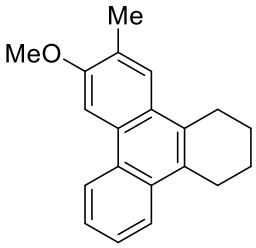
6-Methoxy-7-methyl-1,2,3,4-tetrahydrotriphenylene (2o)



Phenanthrene **2n** was synthesized following the general procedure C using the corresponding alkene (100 mg, 359 μmol, 1.00 equiv) and PIFA (186 mg, 467 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a brown resin (70 mg, 253 μmol, 71%, ratio of 7-methyl- vs. 5-methyl-isomer = 85:15).

R_f = 0.44 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2933, 2835, 1622, 1500, 1452, 1435, 1246, 1221, 1193, 1156, 1135, 1050, 1018, 998, 868, 855, 823, 749, 717, 697, 627, 596, 557, 547, 459; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.63 (m, 1H), 8.46 (s, 1H), 8.06 (m, 1H), 7.63-7.52 (m, 2H), 7.33 (s, 1H), 4.02 (s, 3H), 3.21 (m, 4H), 2.49 (s, 3H), 2.03 (m, 4H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 131.7, 130.9, 129.8, 129.6, 129.5, 129.2, 128.7, 126.8, 126.5, 126.2, 125.3, 124.6, 123.3, 123.1, 122.3, 110.2, 102.0, 77.4, 77.2, 77.0, 76.6, 55.4, 55.3, 27.1, 26.8, 23.0, 16.9, 16.4; **HR-EI-MS**: calc. ($C_{20}H_{20}O$, $[M]^+$): 276.1514, obs.: 276.1523.

7-Methoxy-6-methyl-1,2,3,4-tetrahydrotriphenylene (2p)

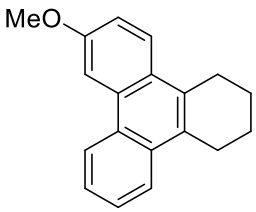


Phenanthrene **2o** was synthesized following the general procedure C using the corresponding alkene (100 mg, 359 μmol, 1.00 equiv) and PIFA (186 mg, 467 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a yellow resin (73 mg, 262 μmol, 73%).

R_f = 0.46 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2931, 2864, 1673, 1603, 1498, 1485, 1441, 1394, 1297, 1248, 1222, 1159, 1033, 909, 842, 768, 753, 730; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.62 (m, 1H), 8.05 (m, 1H), 7.98 (s, 1H), 7.83 (d, $^4J_{HH}$ = 0.6 Hz, 1H), 7.64-7.55 (m, 2H), 4.07 (s, 3H), 3.22-3.07 (m, 4H), 2.47 (d, $^4J_{HH}$ = 0.6 Hz, 3H), 2.06-1.94 (m, 4H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 156.4, 132.0, 129.9, 128.9, 128.8, 127.7, 127.3, 126.3, 126.1, 125.1, 125.0, 123.4, 122.5,

101.7, 55.4, 27. 0, 26.7, 23.1, 23.0, 17.0; **HR-EI-MS**: calc. ($C_{20}H_{20}O$, $[M]^+$): 276.1514; obs.: 276.1509,

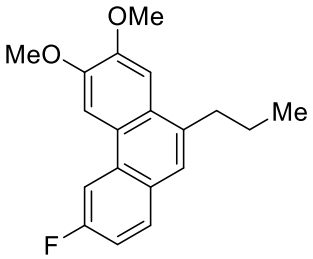
7-Methoxy-1,2,3,4-tetrahydrotriphenylene (2q)



Phenanthrene **2p** was synthesized following the general procedure C using the corresponding alkene (100 mg, 378 μmol, 1.00 equiv) and PIFA (189 mg, 492 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a yellow resin (78 mg, 299 μmol, 79%).

R_f = 0.41 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2930, 2834, 1618, 1530, 1504, 1435, 1362, 1282, 1228, 1177, 1044, 1018, 840, 807, 752; **^{1}H-NMR** δ (300 MHz, $CDCl_3$) = 8.63 (m, 1H), 8.07 (m, 2H), 7.98 (d, $^3J_{HH}$ = 9.1 Hz, 1H), 7.61 (m, 2H), 7.26 (m, 1H), 4.03 (s, 3H), 3.22-3.02 (m, *J* = 7.6, 5.3 Hz, 4H), 2.05-1.92 (m, 4H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 157.6, 132.5, 130.7, 130.2, 128.8, 127.9, 126.7, 126.6, 125.2, 124.9, 123.4, 122.8, 116.1, 104.5, 55.5, 26.9, 26.7, 23.0, 23.0; **HR-EI-MS**: calc. ($C_{19}H_{18}O$, $[M]^+$): 262.1358; obs.: 262.1365.

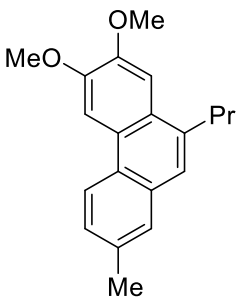
6-Fluoro-2,3-dimethoxy-10-propylphenanthrene (2r)



Phenanthrene **2q** was synthesized following the general procedure C using the corresponding alkene (100 mg, 333 μmol, 1.00 equiv) and PIFA (186 mg, 433 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 30:1) delivered the product as a yellow resin (65 mg, 218 μmol, 65%).

R_f = 0.32 (pentane/EtOAc 30:1); **IR**: ν (cm^{-1}) = 2962, 2250, 1529, 1505, 1467, 1435, 1415, 1265, 1200, 1155, 1103, 1030, 913, 862, 845, 792, 721, 643, 615, 559; **^{1}H-NMR** δ (400 MHz, $CDCl_3$) = 8.06 (dd, $^3J_{HH}$ = 11.4 Hz, $^4J_{HF}$ = 2.4 Hz, 1H), 7.84 (s, 1H), 7.76 (dd, $^3J_{HF}$ = 8.8 Hz, $^3J_{HH}$ = 6.0 Hz, 1H), 7.45 (s, 1H), 7.38 (s, 1H), 7.28-7.21 (m, 1H), 4.10 (s, 3H), 4.06 (s, 3H), 3.05-2.96 (m, 2H), 1.93-1.79 (m, 2H), 1.08 (t, $^3J_{HH}$ = 7.4 Hz, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 161.0 (d, $^1J_{CF}$ = 243.4 Hz), 149.5 , 148.8, 134.9 (d, $^5J_{CF}$ = 2.6 Hz), 130.2 (d, $^3J_{CF}$ = 8.2 Hz), 130.1 (d, $^3J_{CF}$ = 8.9 Hz), 128.0 (d, $^5J_{CF}$ = 1.4 Hz), 126.7 , 124.7 (d, $^4J_{CF}$ = 4.4 Hz), 123.7 , 114.6 (d, $^2J_{CF}$ = 23.9 Hz), 106.8 (d, $^2J_{CF}$ = 22.2 Hz), 104.7, 103.8, 55.9, 55.8, 35.5, 22.9, 14.3; **HR-ESI-MS**: $[C_{19}H_{20}O_2F]^+$ ($[M+H]^+$): calcd.: 299.1442, obs.: 299.1429.

2,3-Dimethoxy-7-methyl-10-propylphenanthrene (2s)



Phenanthrene **2r** was synthesized following the general procedure C using the corresponding alkene (100 mg, 337 μmol, 1.00 equiv) and PIFA (168 mg, 438 μmol, 1.30 equiv). Column chromatography (SiO_2, pentane/EtOAc 20:1) delivered the product as a yellow resin (66 mg, 223 μmol, 66%).

R_f = 0.34 (pentane/EtOAc 20:1); **IR**: ν (cm^{-1}) = 2956, 1785, 1609, 1520, 1442, 1266, 1207, 1157, 1029, 860, 845, 829, 792; **^{1}H-NMR** δ (400 MHz, $CDCl_3$) = 8.26 (s, 1H), 8.04 (s, 1H), 7.71 (d, $^3J_{HH}$ = 8.1 Hz, 1H), 7.46 (s, 1H), 7.41 (s, 1H), 7.34 (dd, $^3J_{HH}$ = 8.1 Hz, $^4J_{HH}$ = 1.1 Hz, 1H), 4.14 (s, 3H), 4.06 (s, 3H), 3.10-2.98 (m, 2H), 2.61 (s, 3H), 1.92-1.79 (m, 2H),

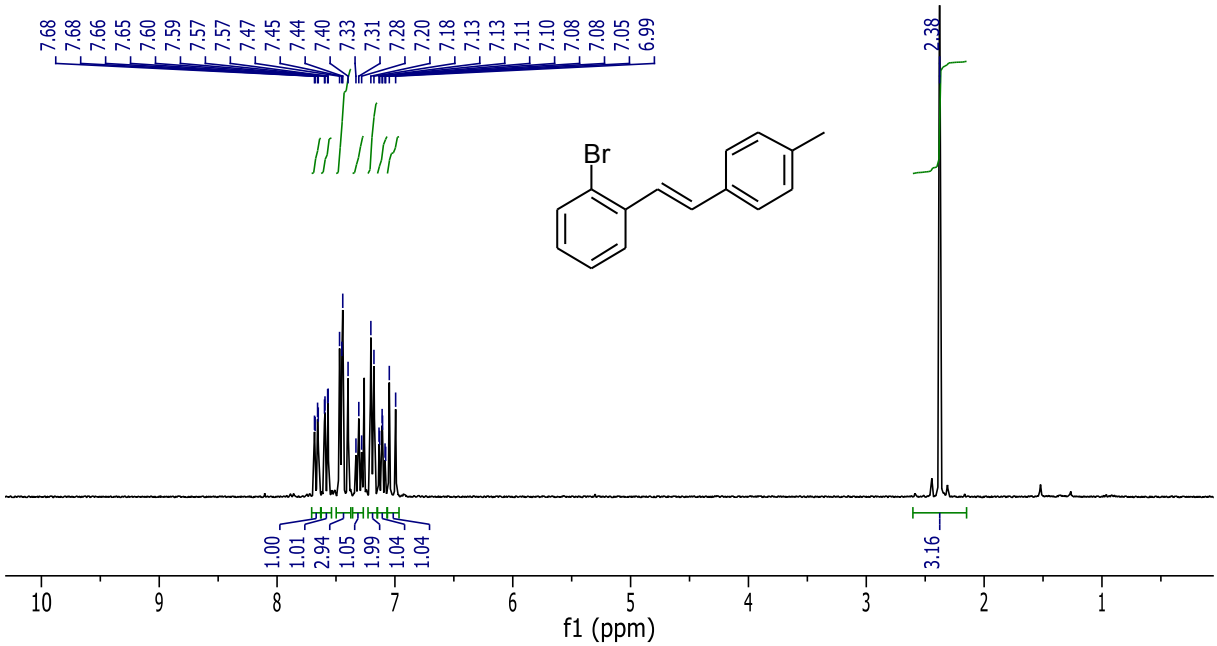
1.27 (t, $^{3}J_{HH}$ = 7.2 Hz, 3H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 149.0, 148.7, 135.0, 134.7, 129.3, 129.1, 128.0, 127.4, 126.5, 125.1, 124.2, 121.6, 104.8, 103.8, 56.0, 55.8, 35.6, 22.9, 22.1, 14.3; **HR-ESI-MS**: calc. ($C_{20}H_{23}O_2$, $[M+H]^+$): 295.1693; obs.: 295.1690,

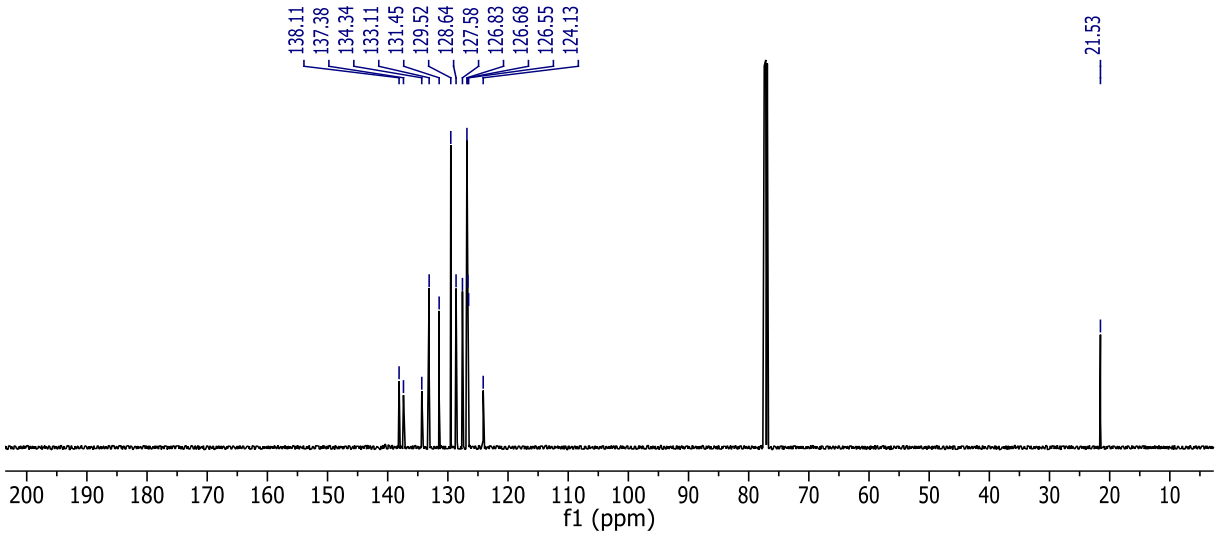
References:

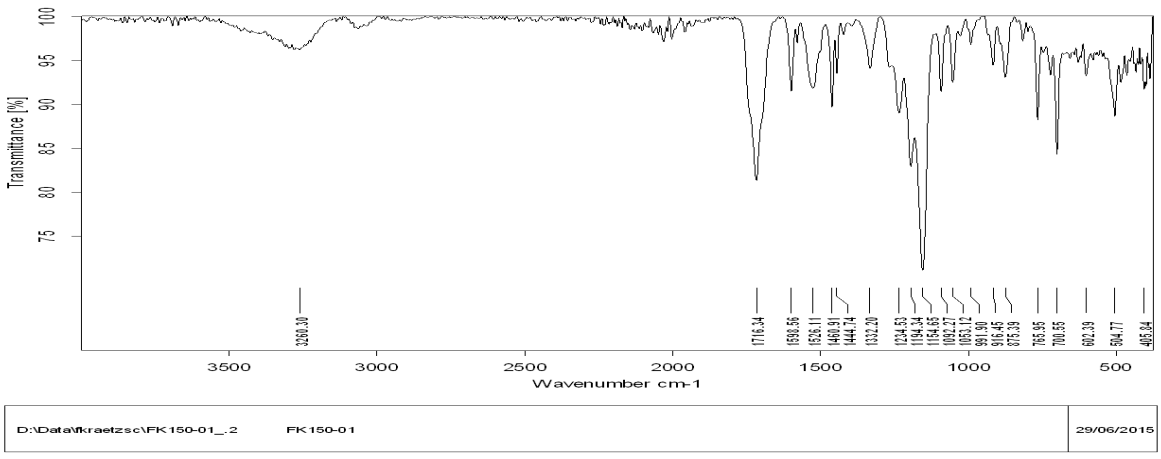
1 S. Maity, N. Zheng, *Angew. Chem. Int. Ed.* 2012, **51**, 9562.

2 M. D. Kennedy, S. J. Bailey, S. M. Wales, P. A. Keller, *J. Org. Chem.* 2015, **80**, 5992.

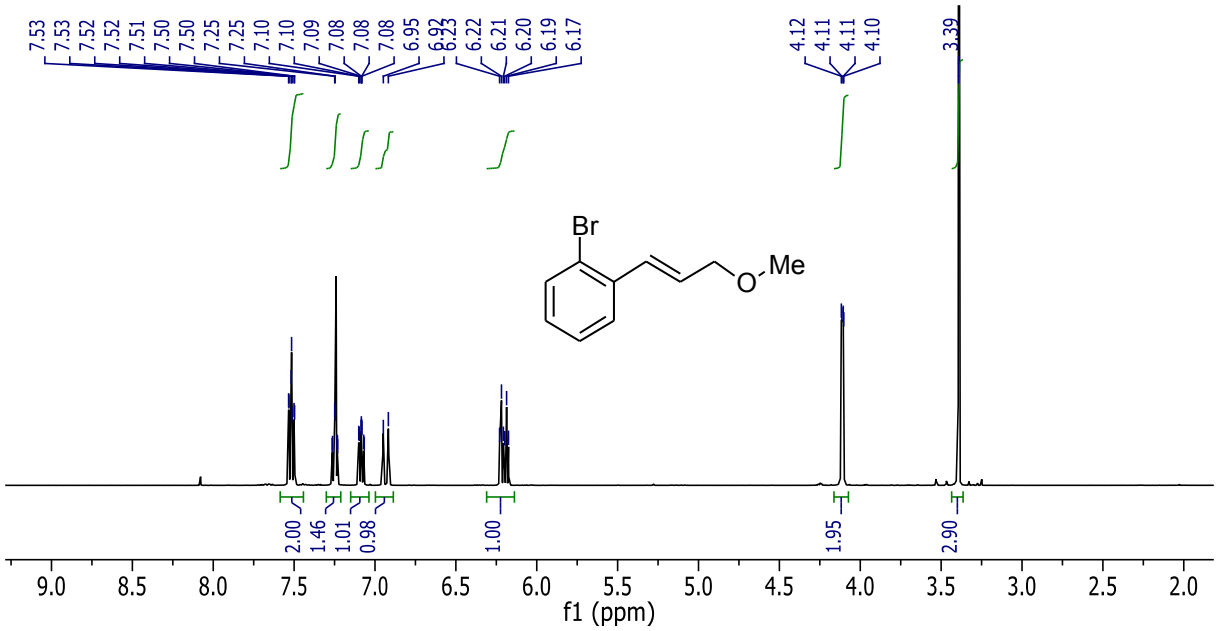
(*E*)-1-Bromo-2-(4-methylstyryl)benzene

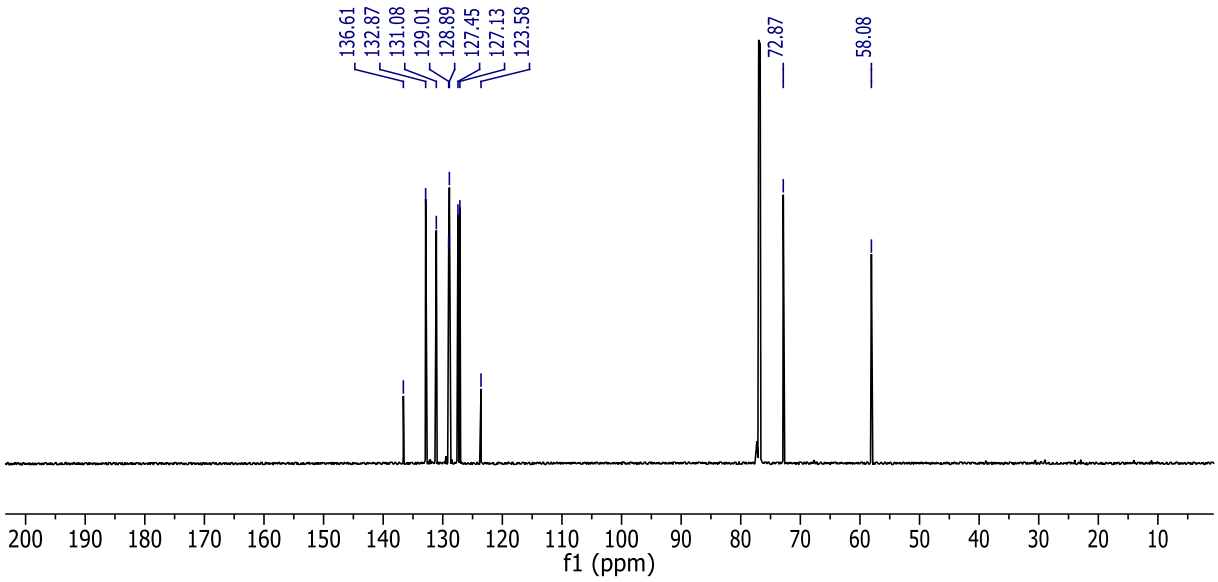


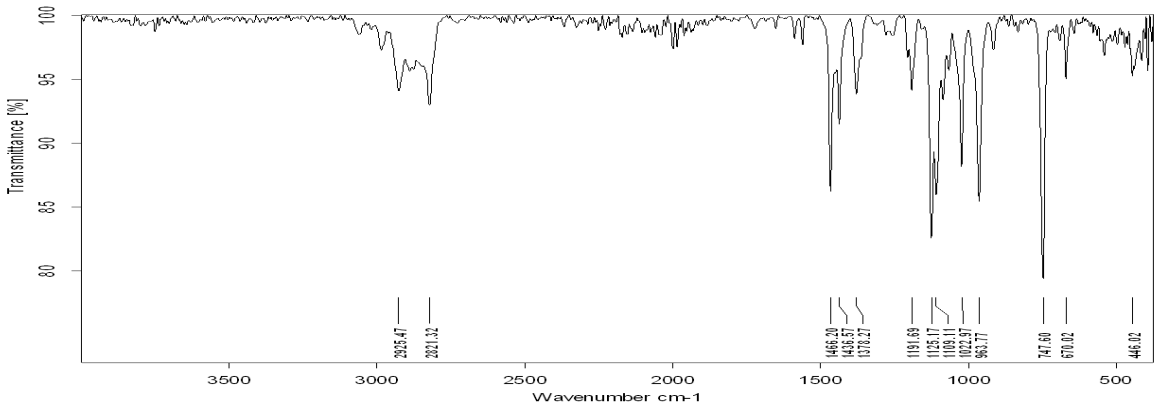




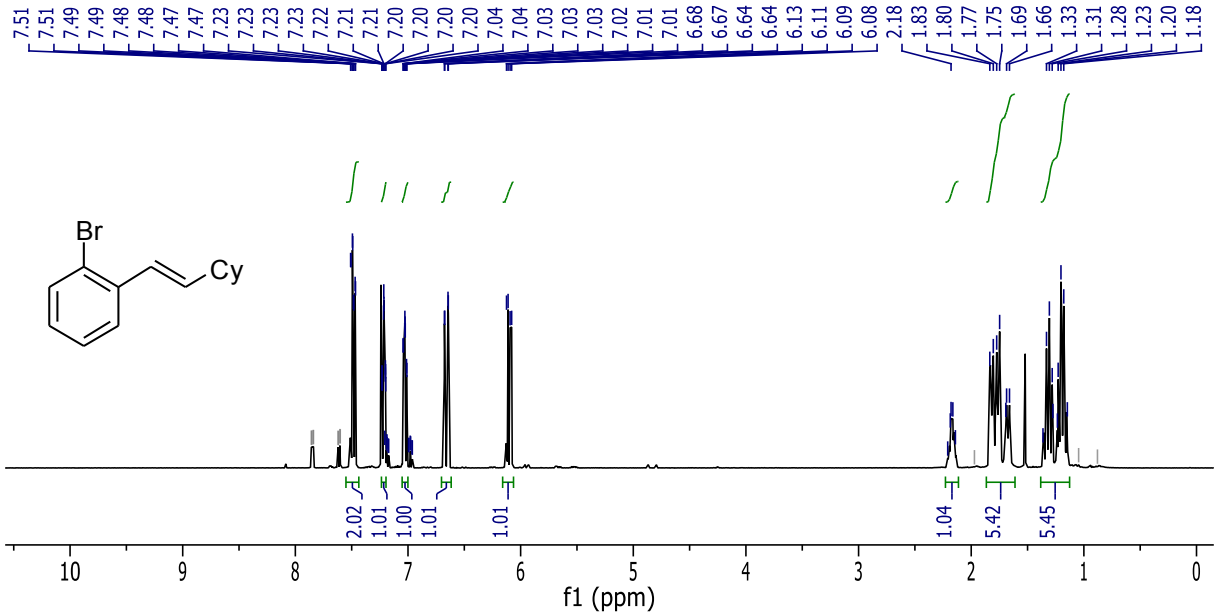
(*E*)-1-Bromo-2-(3-methoxyprop-1-en-1-yl)benzene

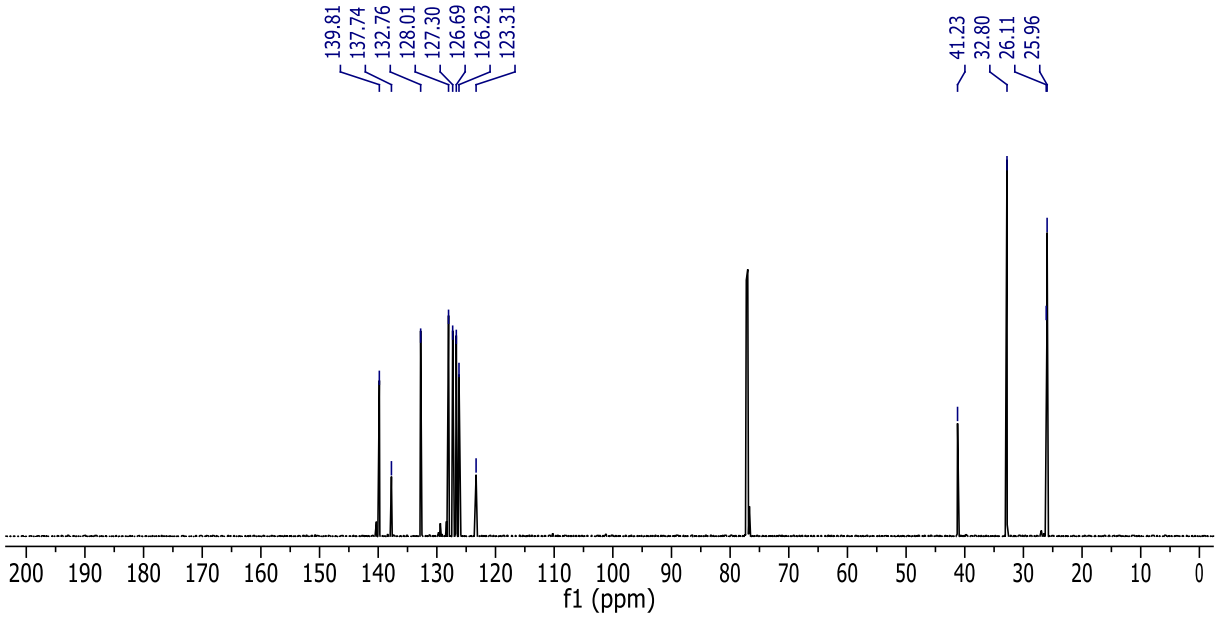


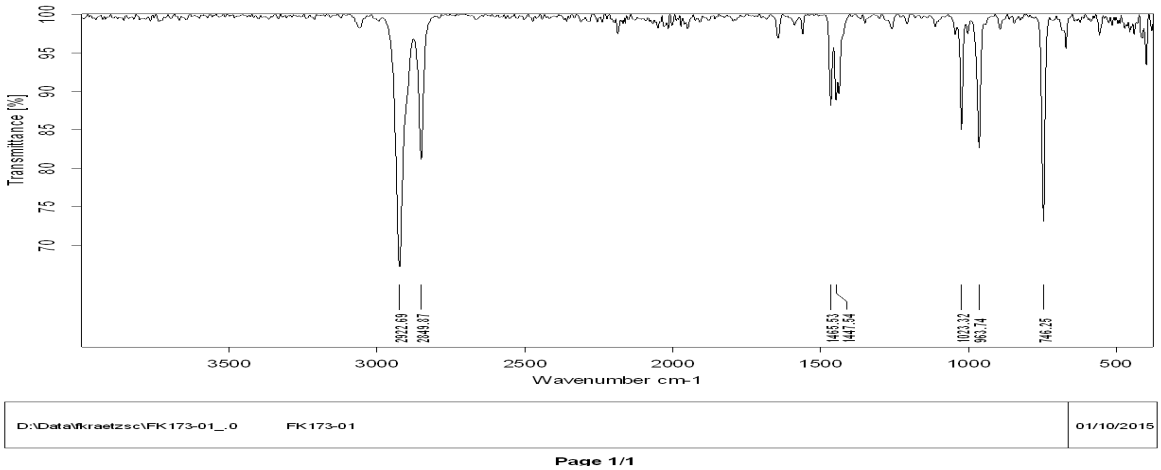




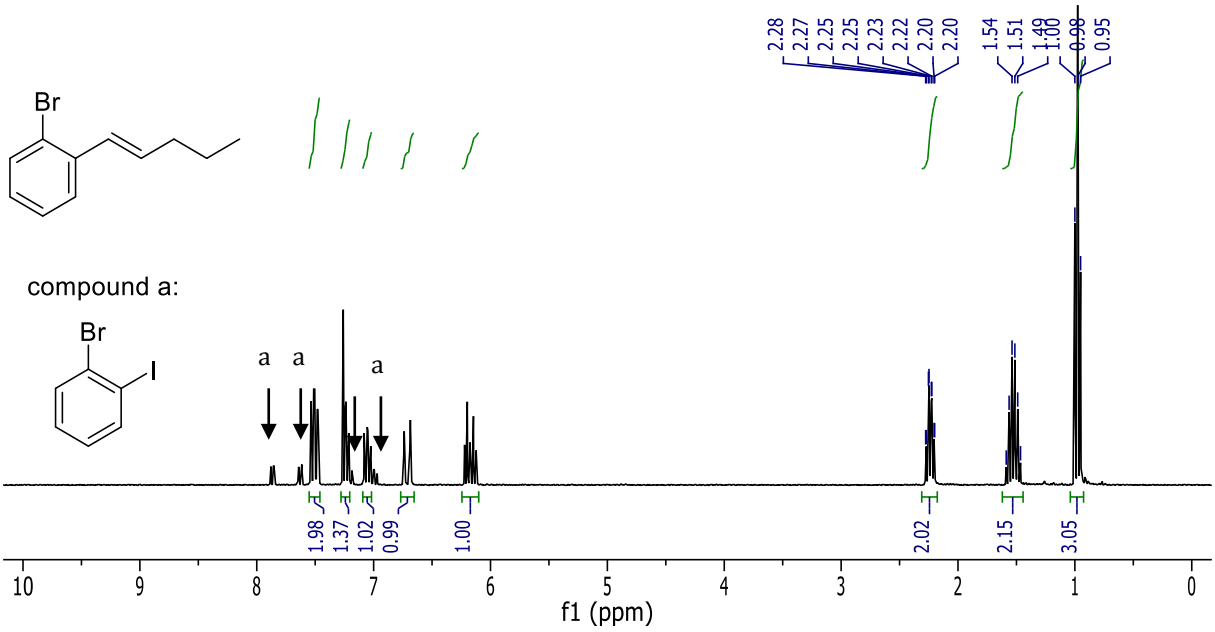
(*E*)-1-Bromo-2-(2-cyclohexylvinyl)benzene

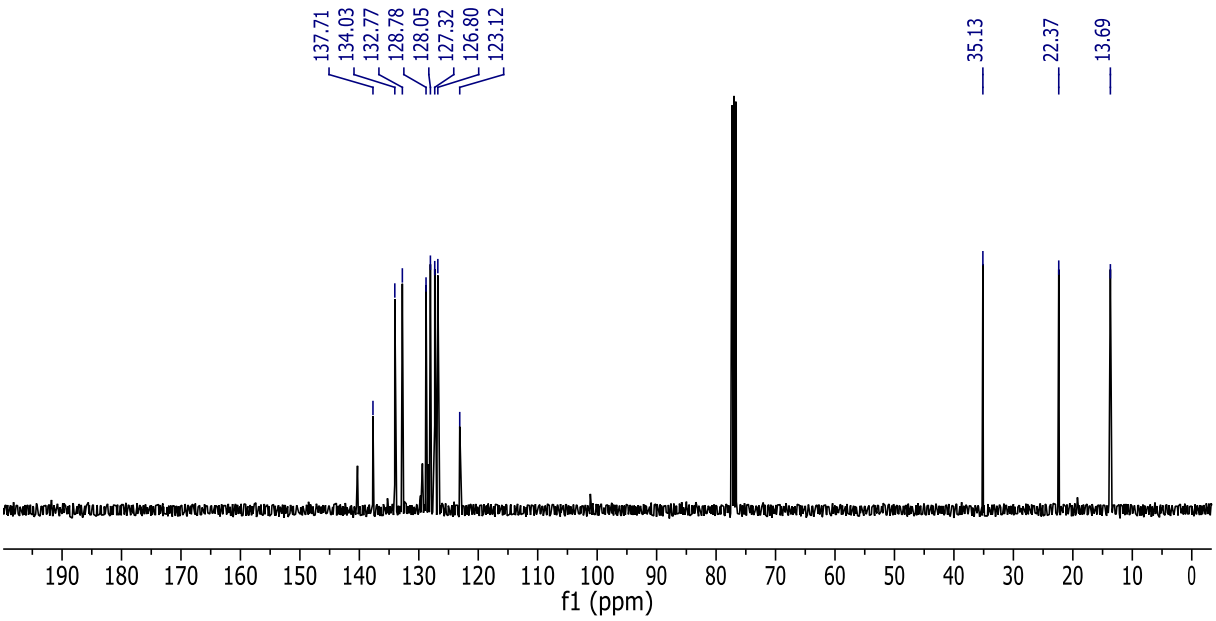


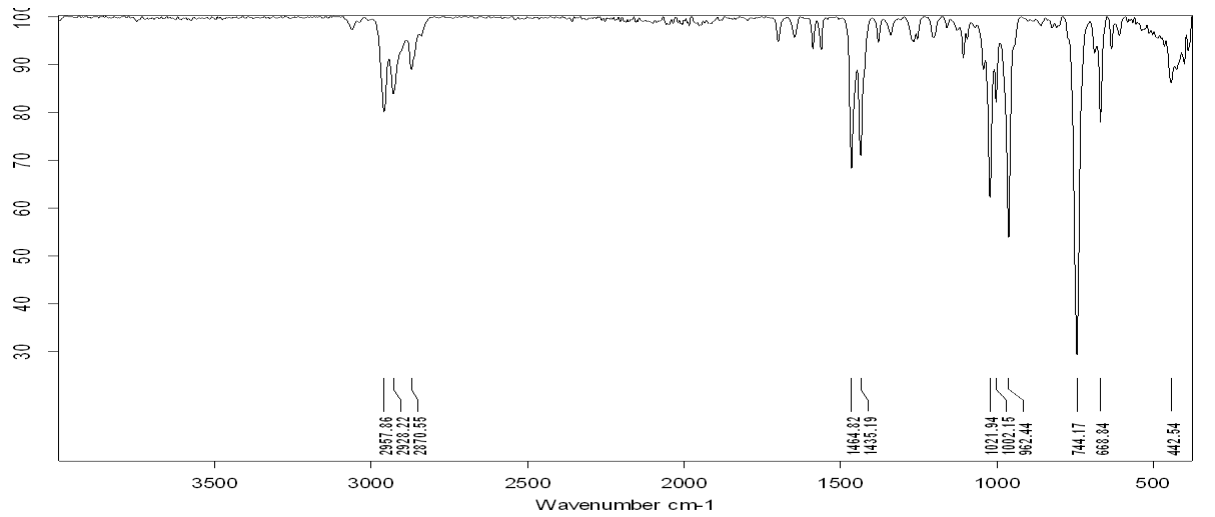




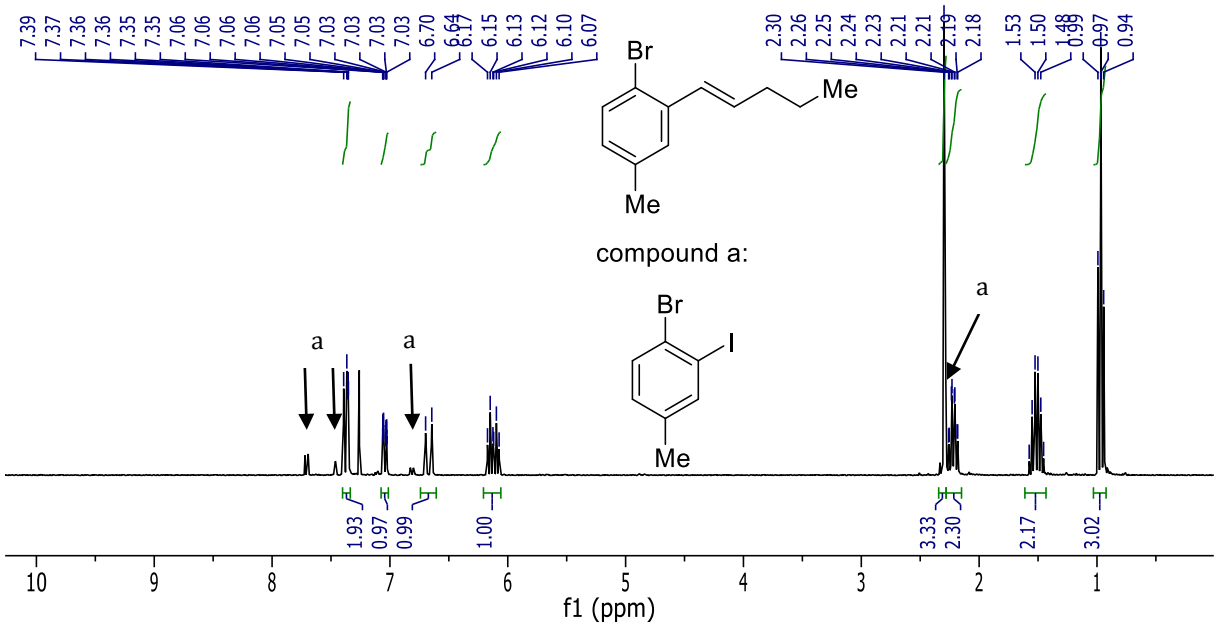
(*E*)-1-Bromo-2-(pent-1-en-1-yl)benzene

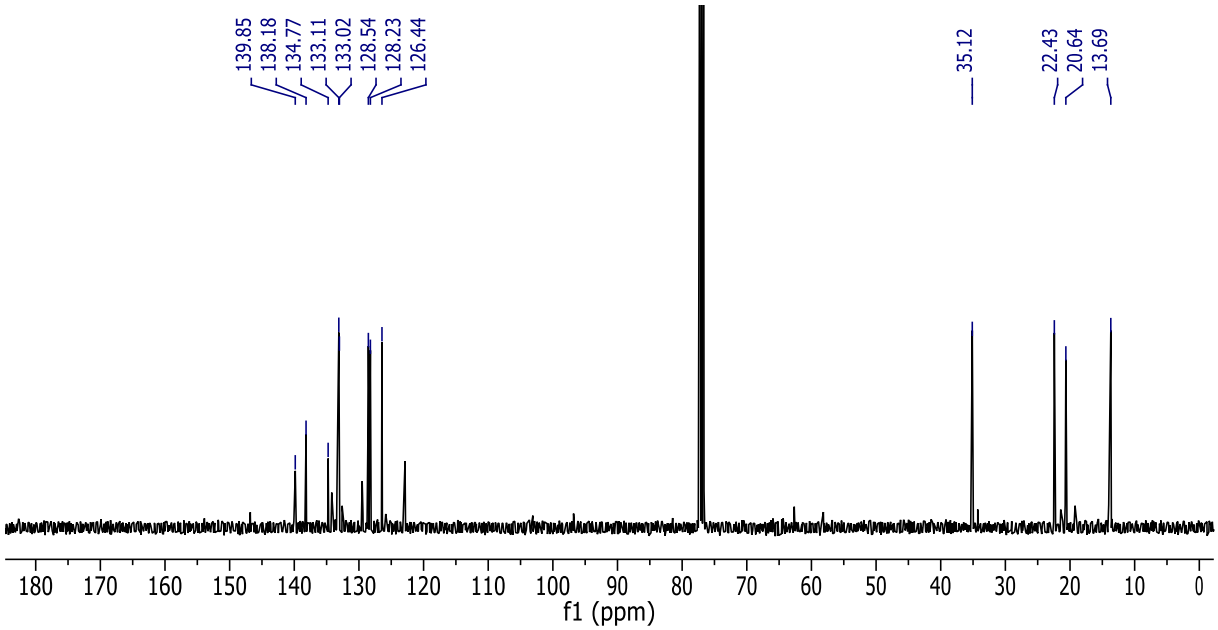


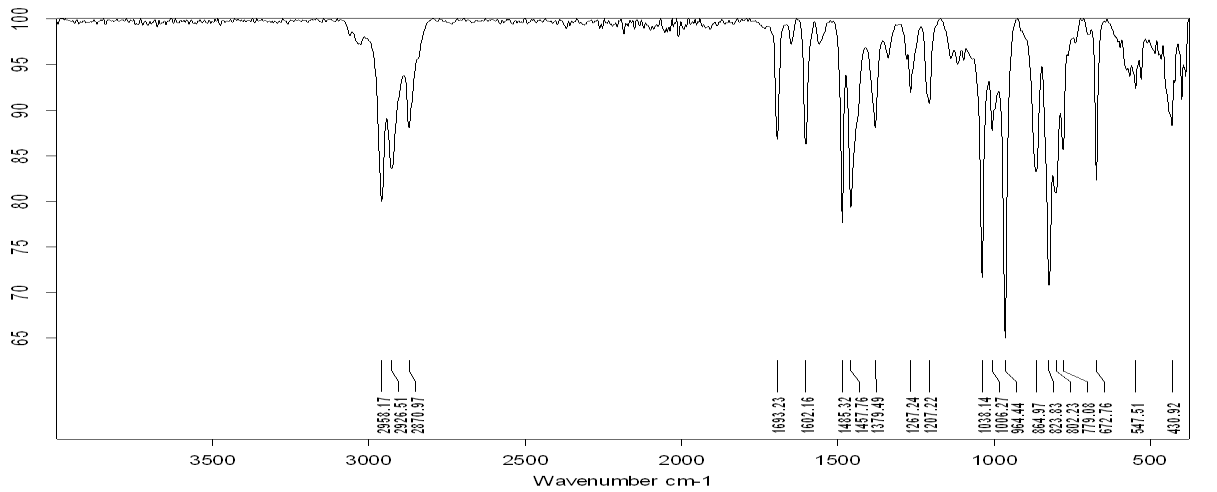




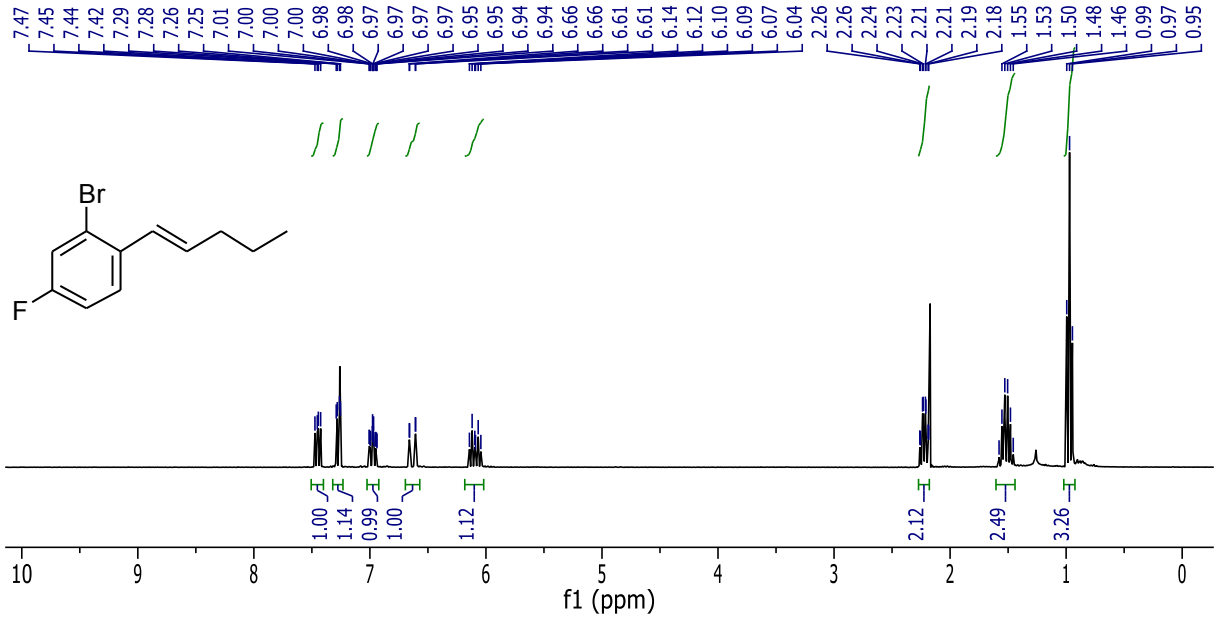
(*E*)-1-Bromo-4-methyl-2-(pent-1-en-1-yl)benzene

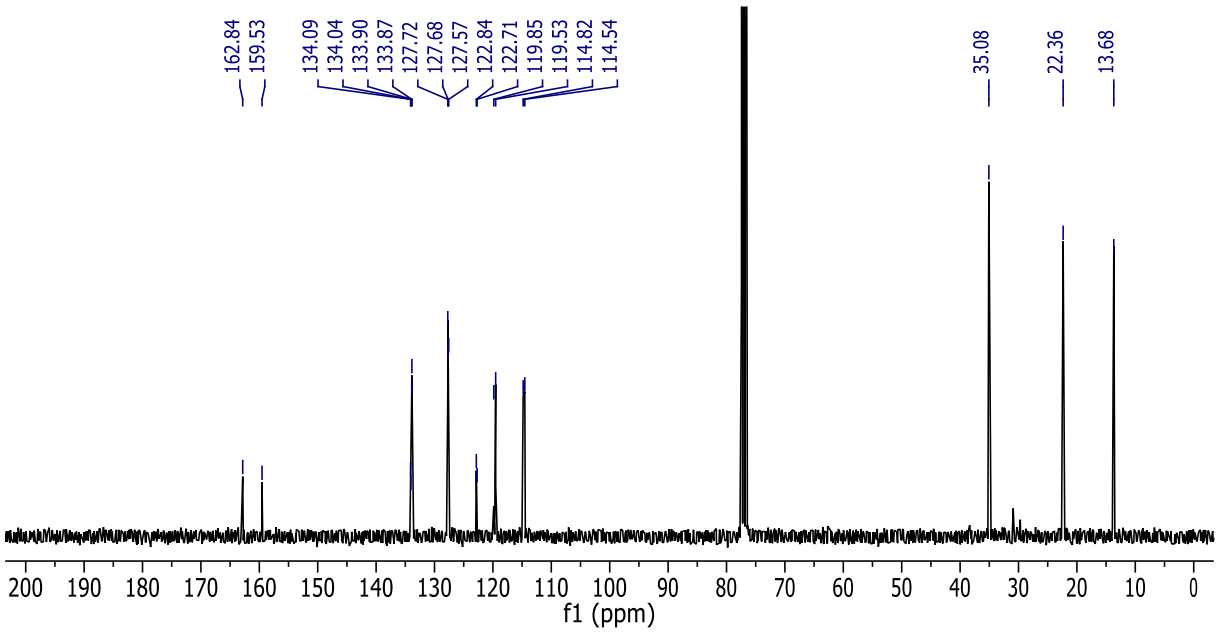


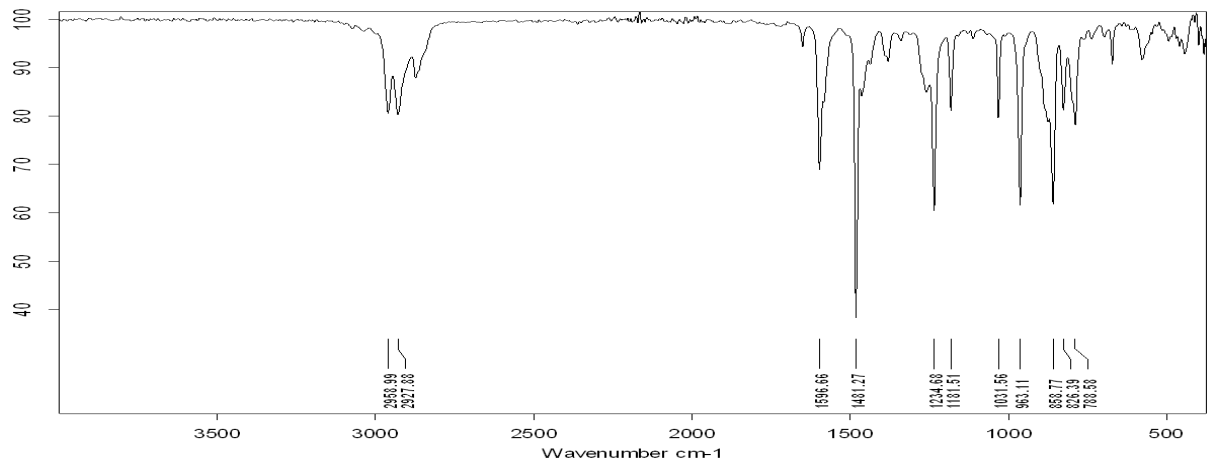




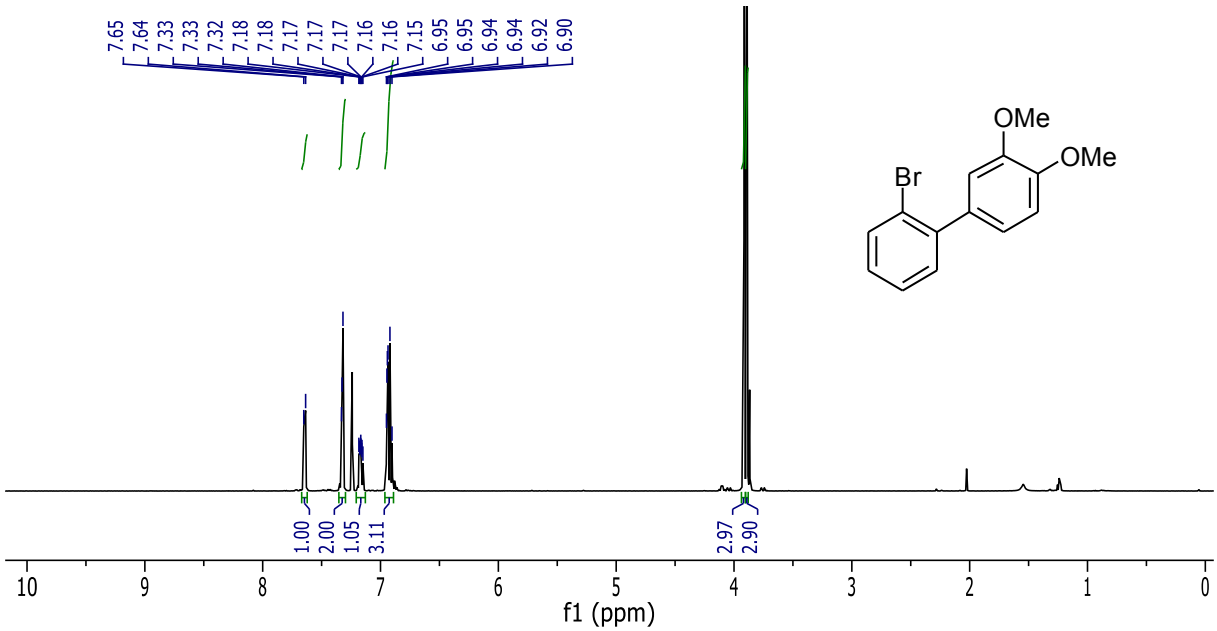
(*E*)-2-Bromo-4-fluoro-1-(pent-1-en-1-yl)benzene

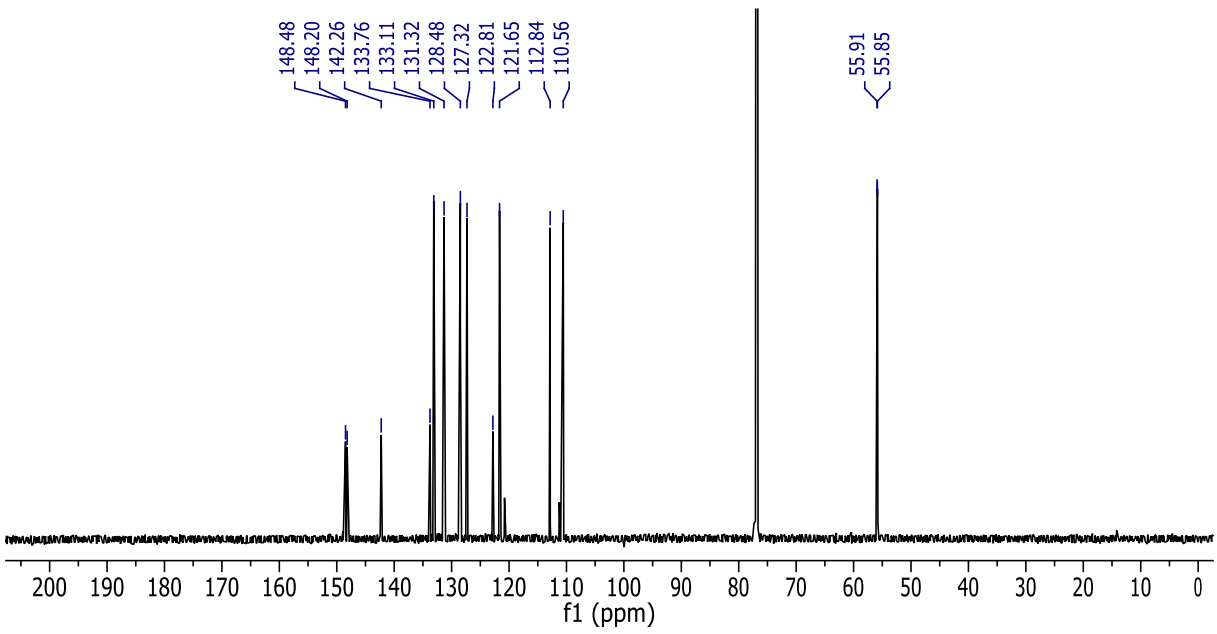


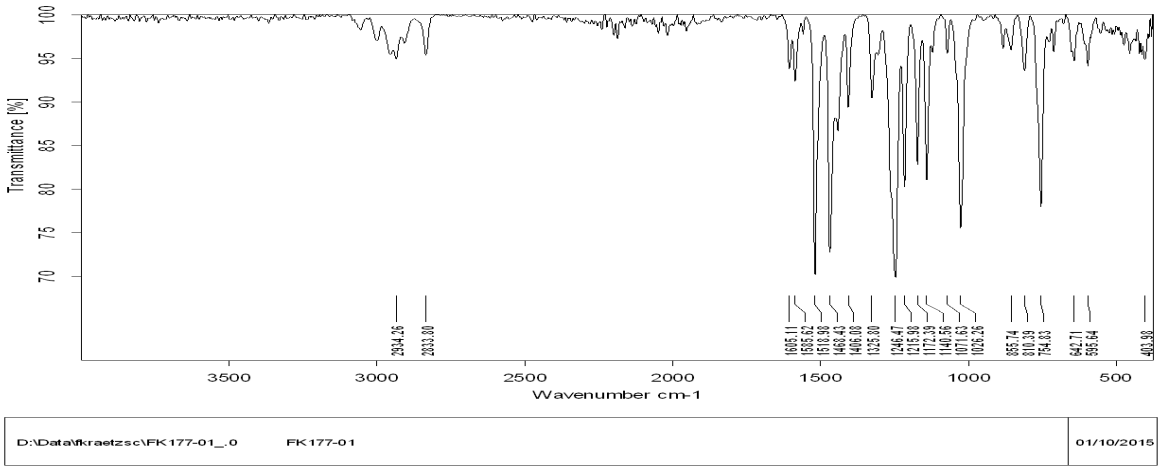




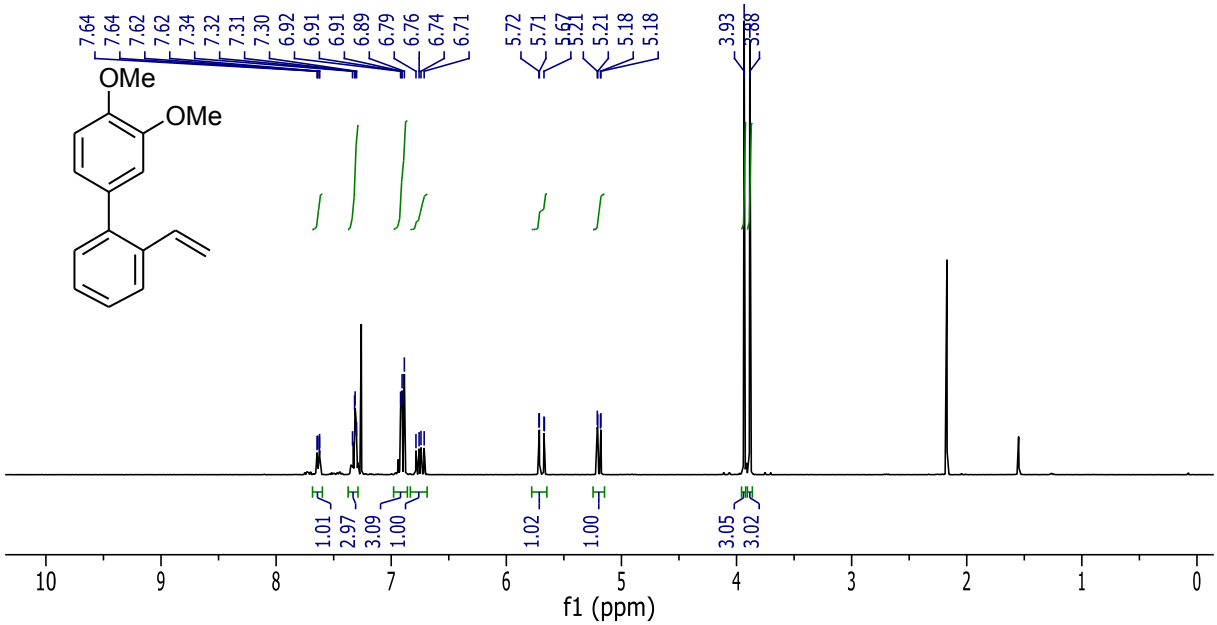
2-Bromo-3',4'-dimethoxy-1,1'-biphenyl

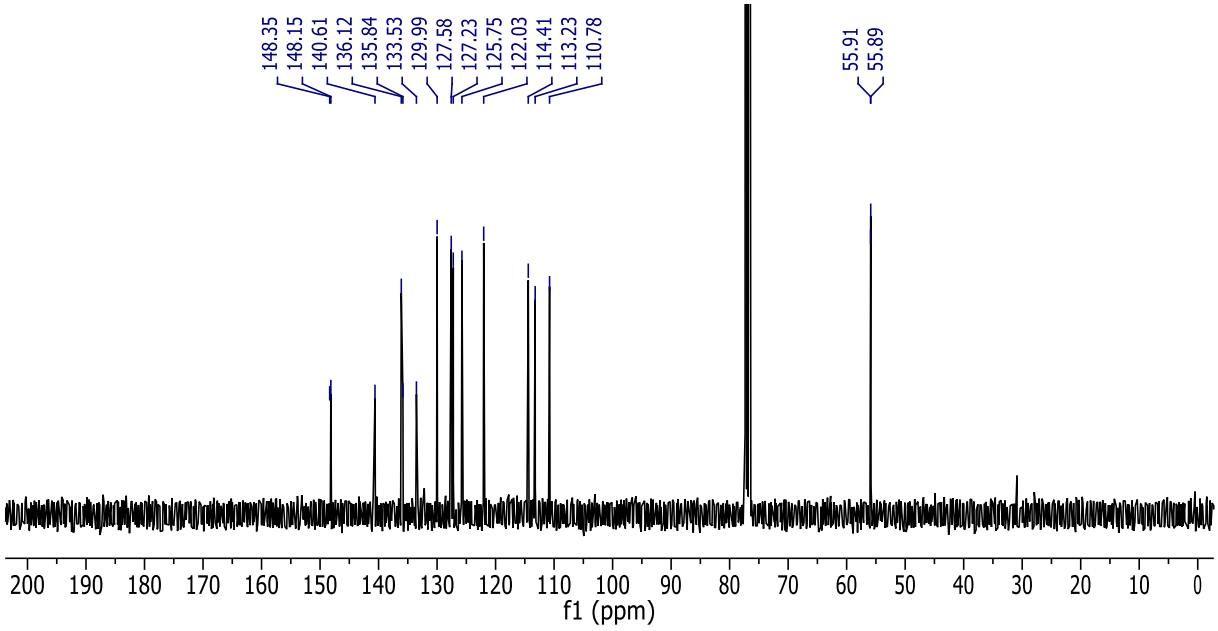


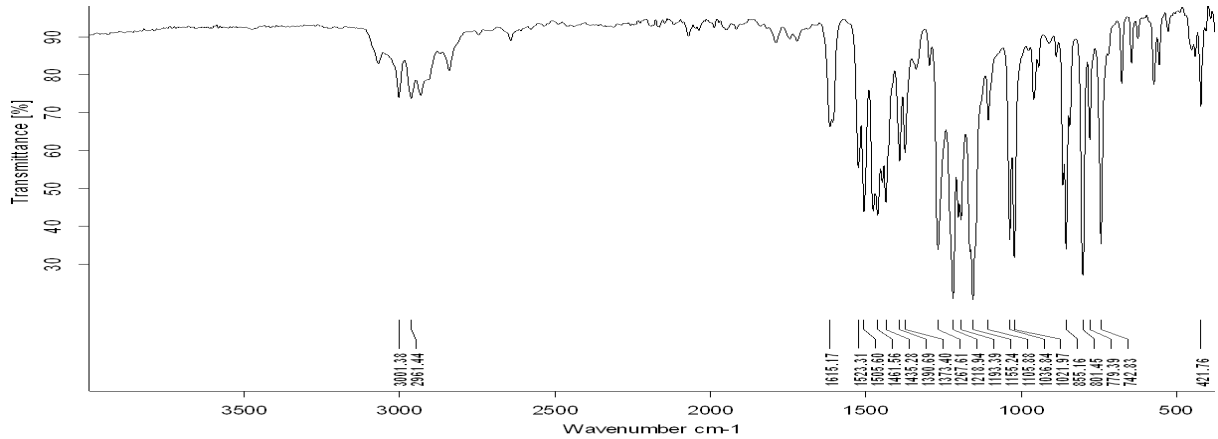




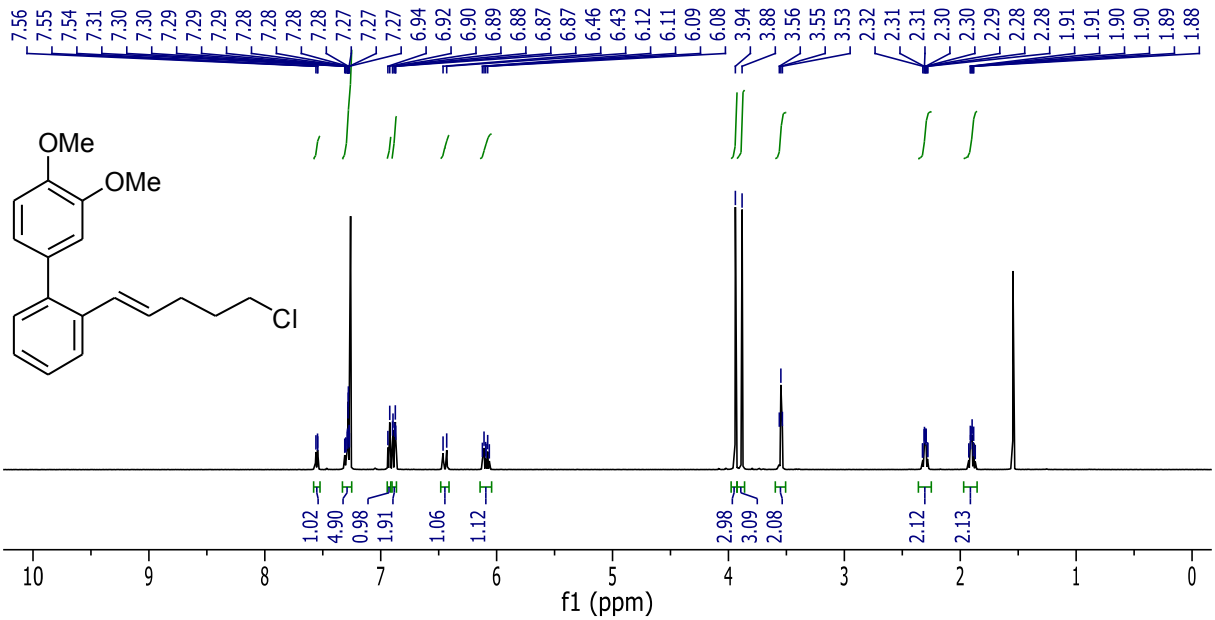
3',4'-Dimethoxy-2-vinyl-1,1'-biphenyl

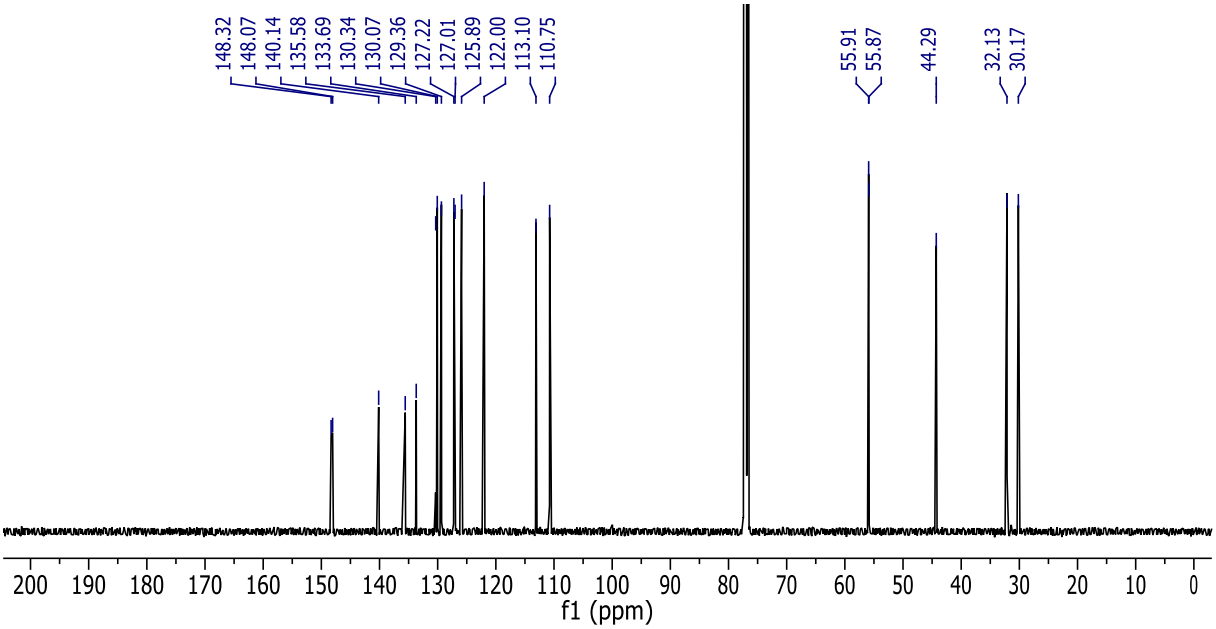


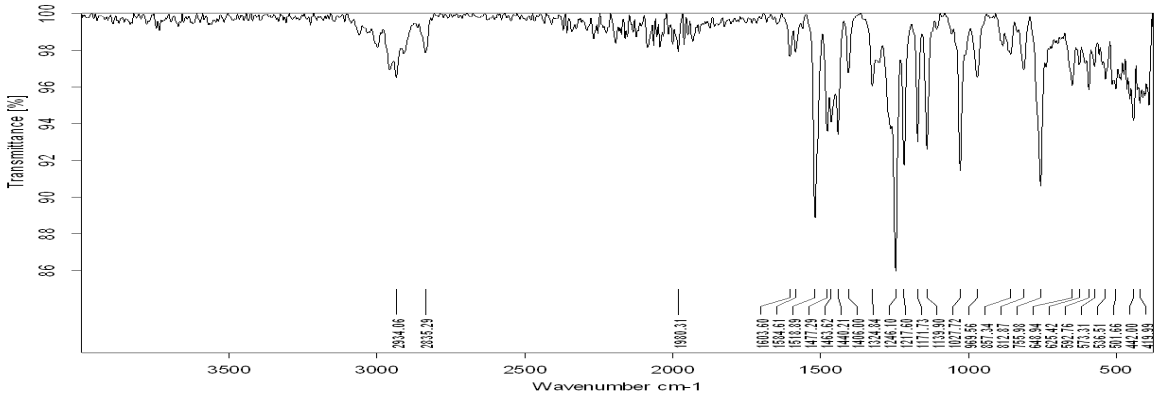




(*E*)-2-(5-Chloropent-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl







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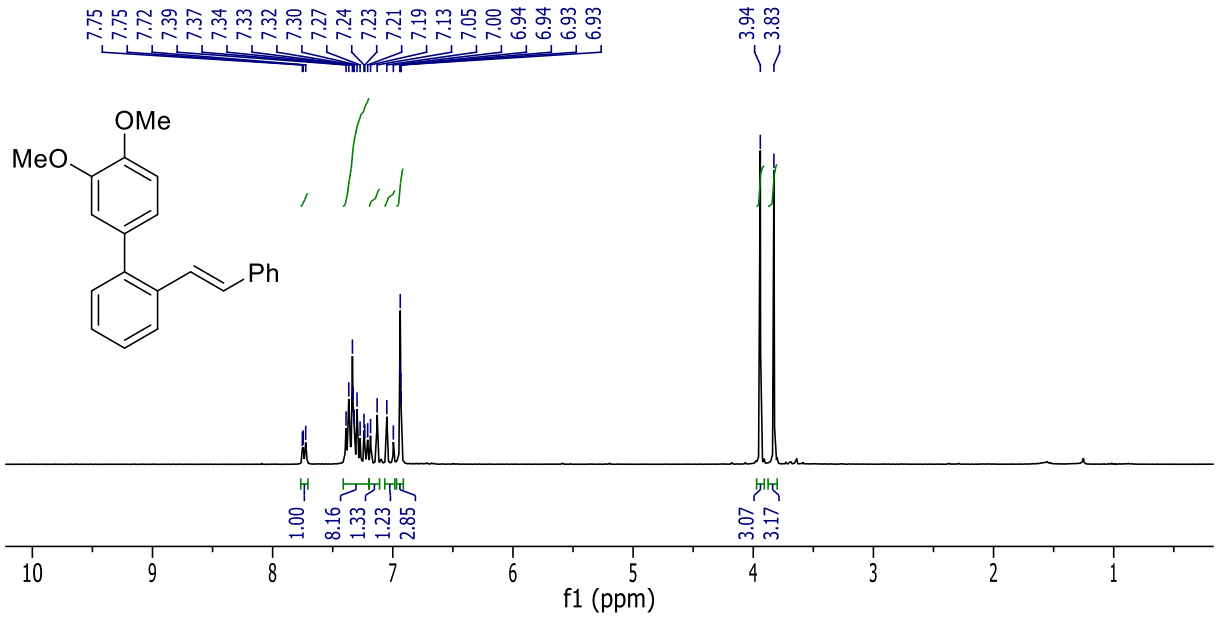
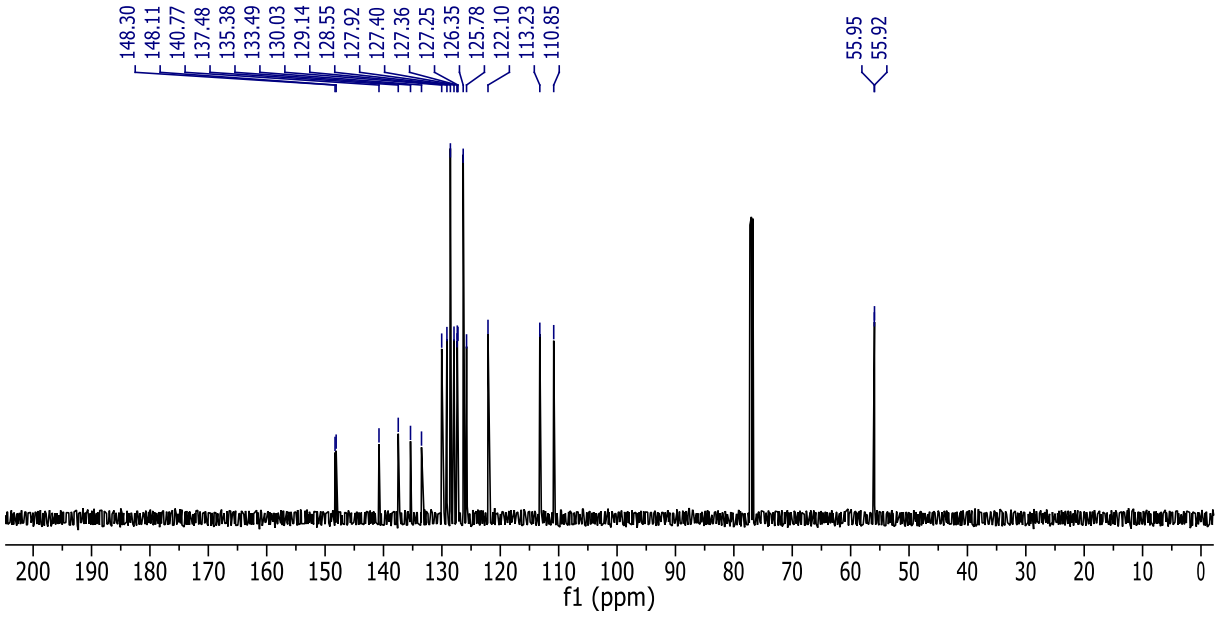
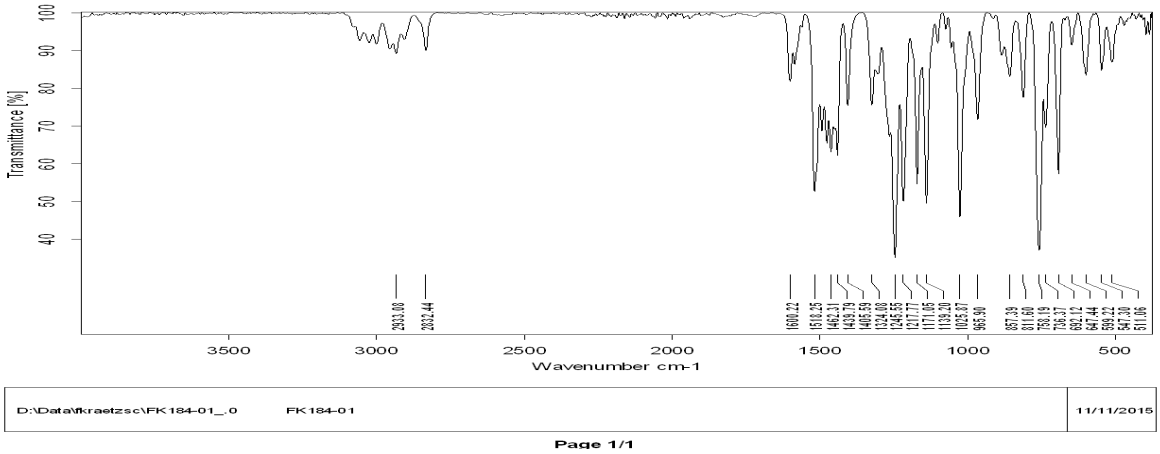
(*E*)-2-(Hex-3-en-3-yl)-3',4'-dimethoxy-1,1'-biphenyl







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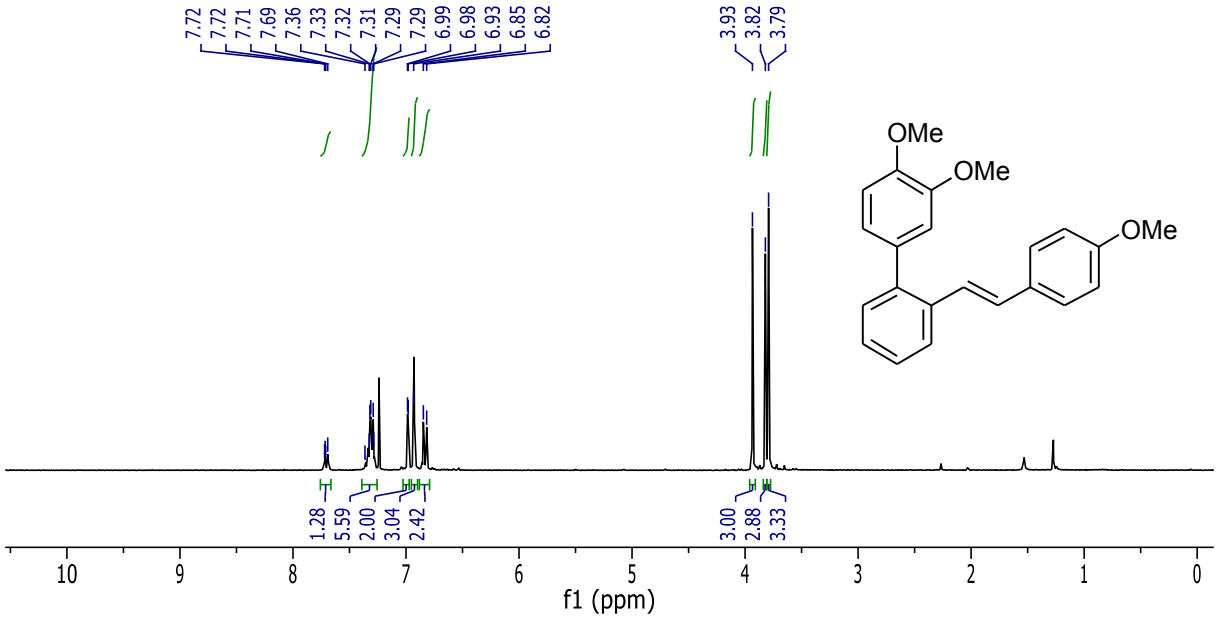
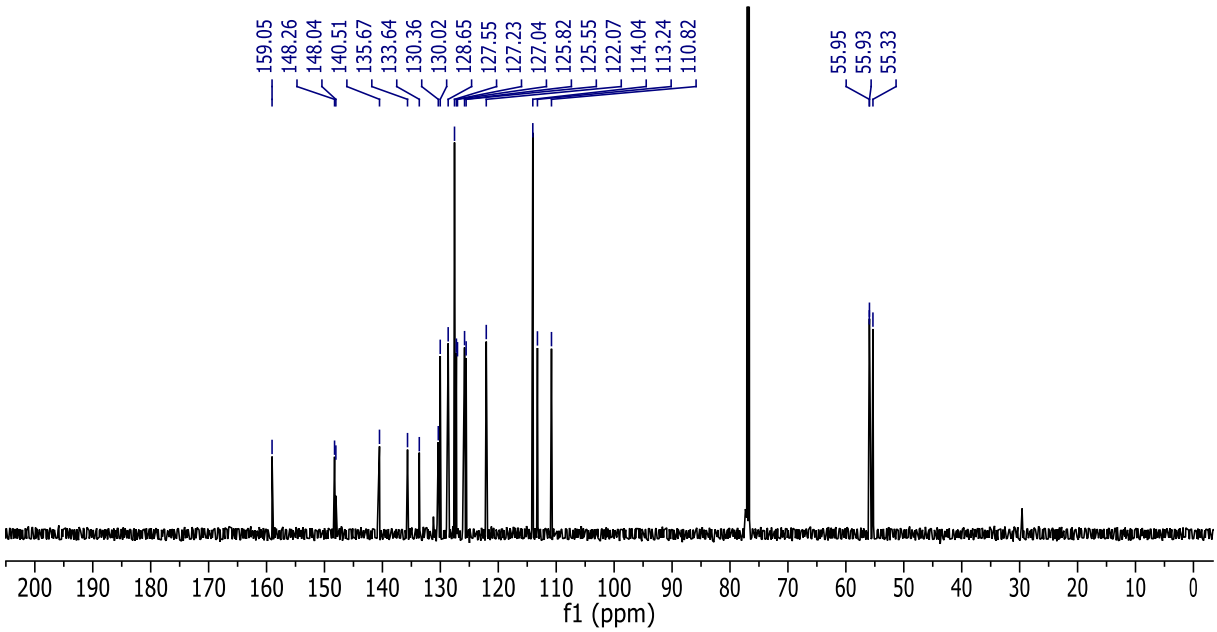
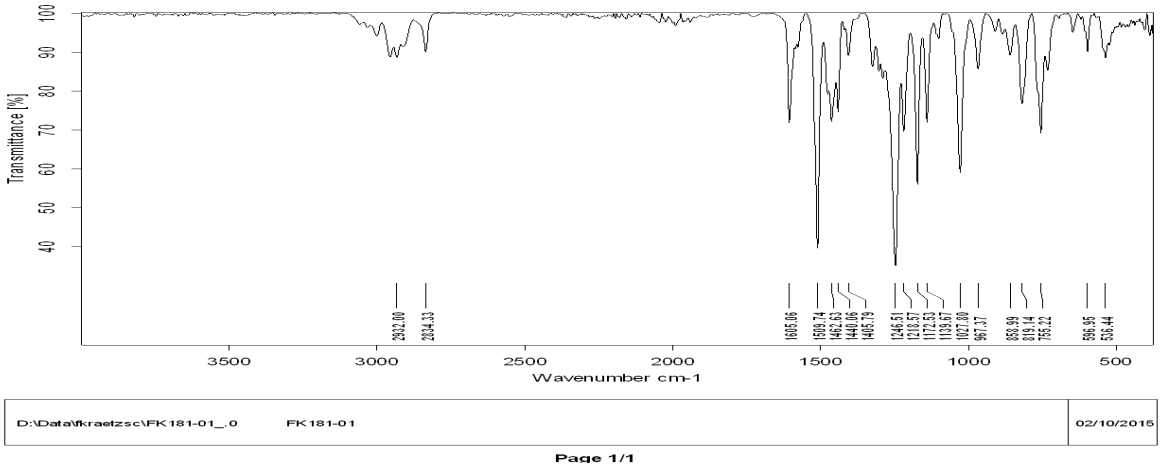
(*E*)-3',4'-Dimethoxy-2-(4-methoxystyryl)-1,1'-biphenyl







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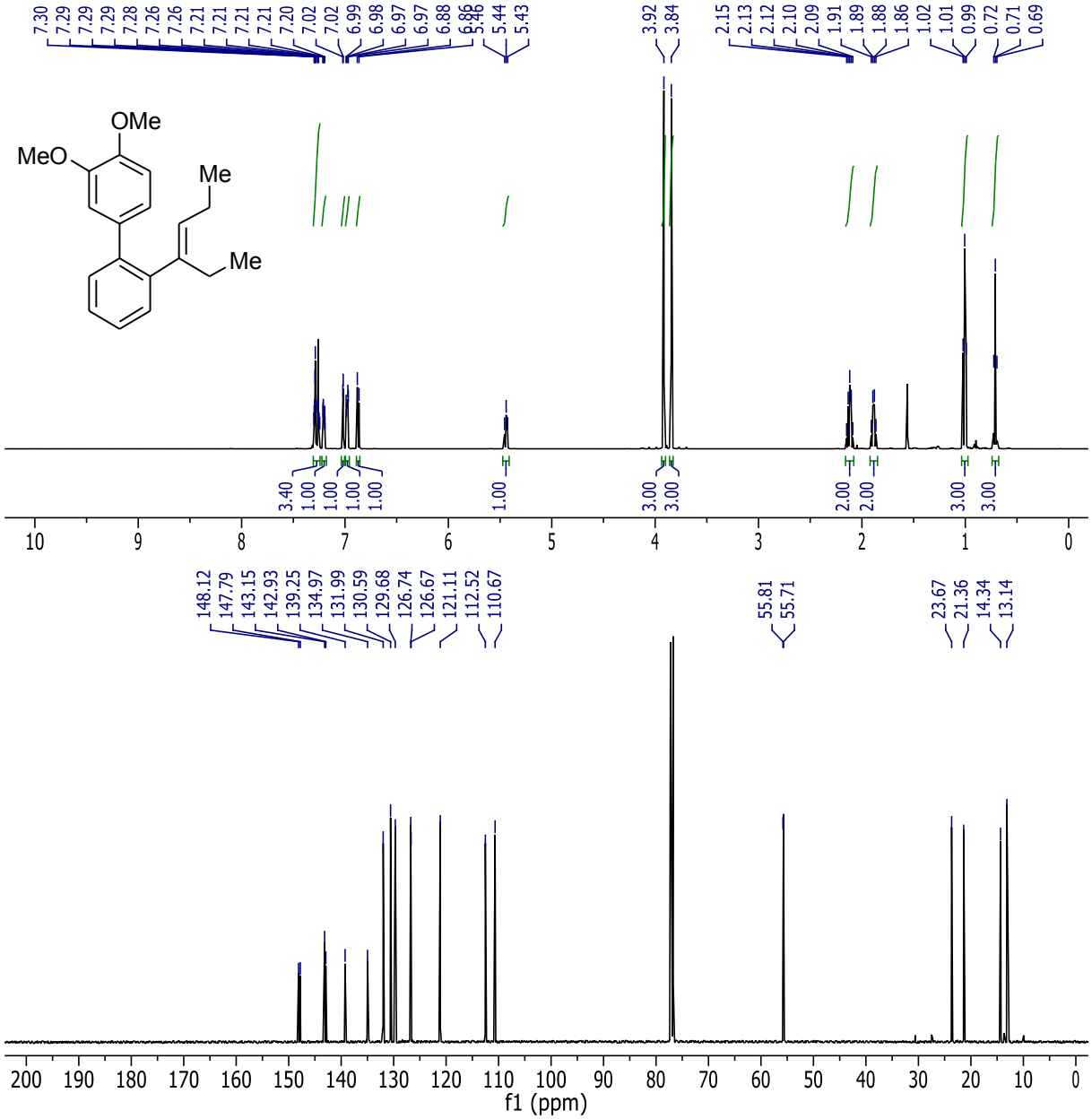
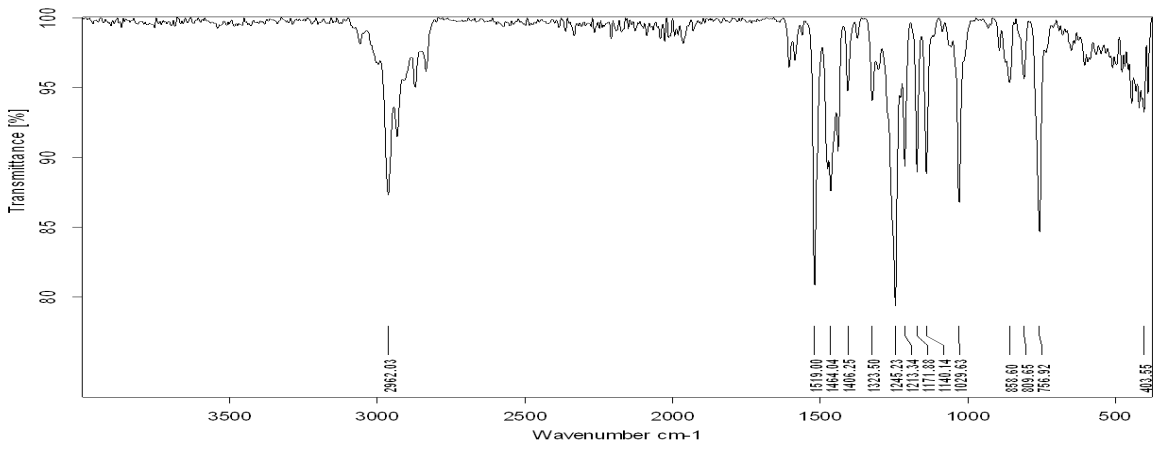
(*E*)-2-(Hex-3-en-3-yl)-3',4'-dimethoxy-1,1'-biphenyl





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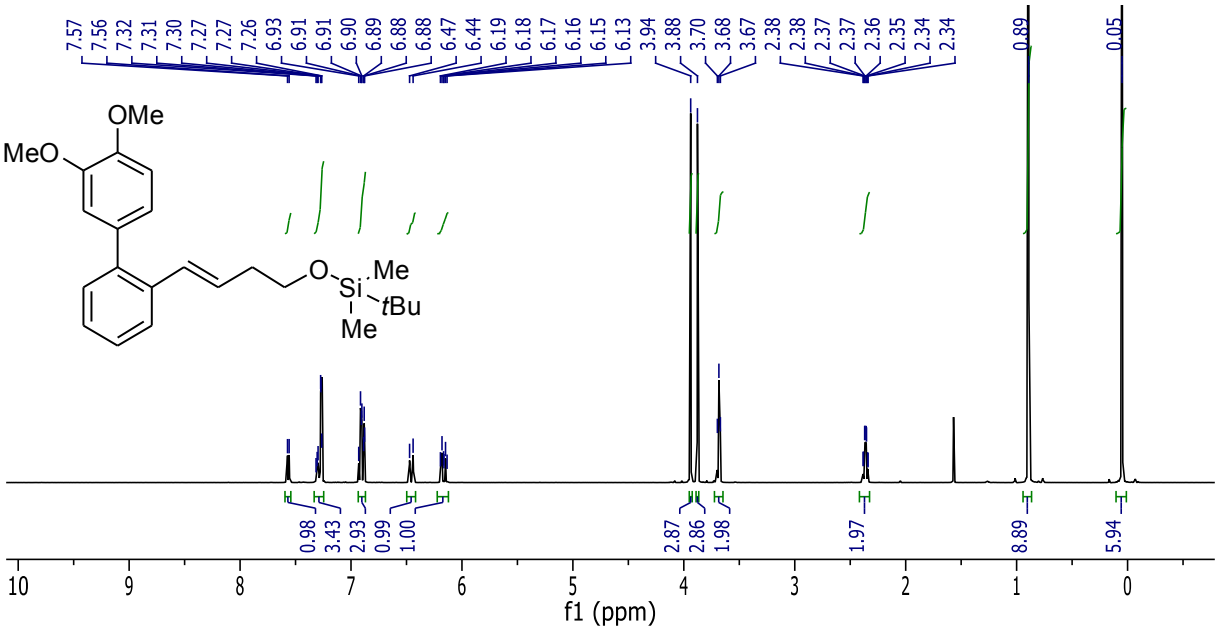
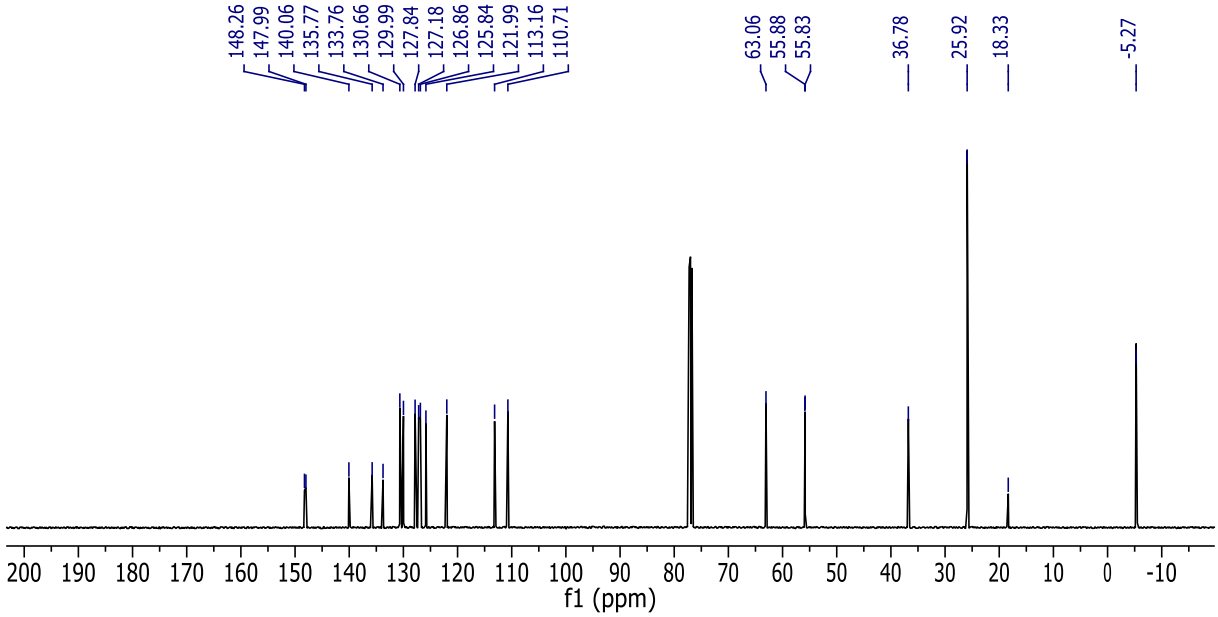
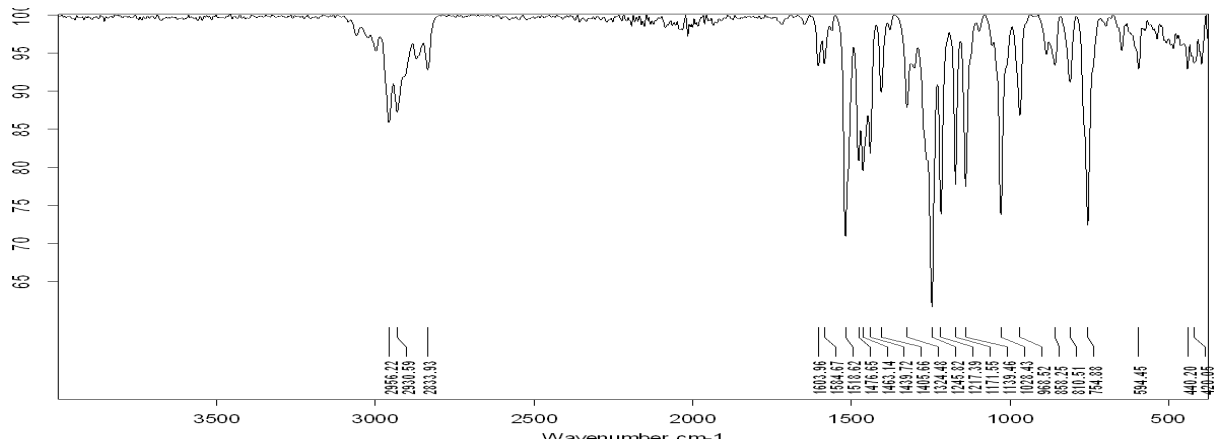
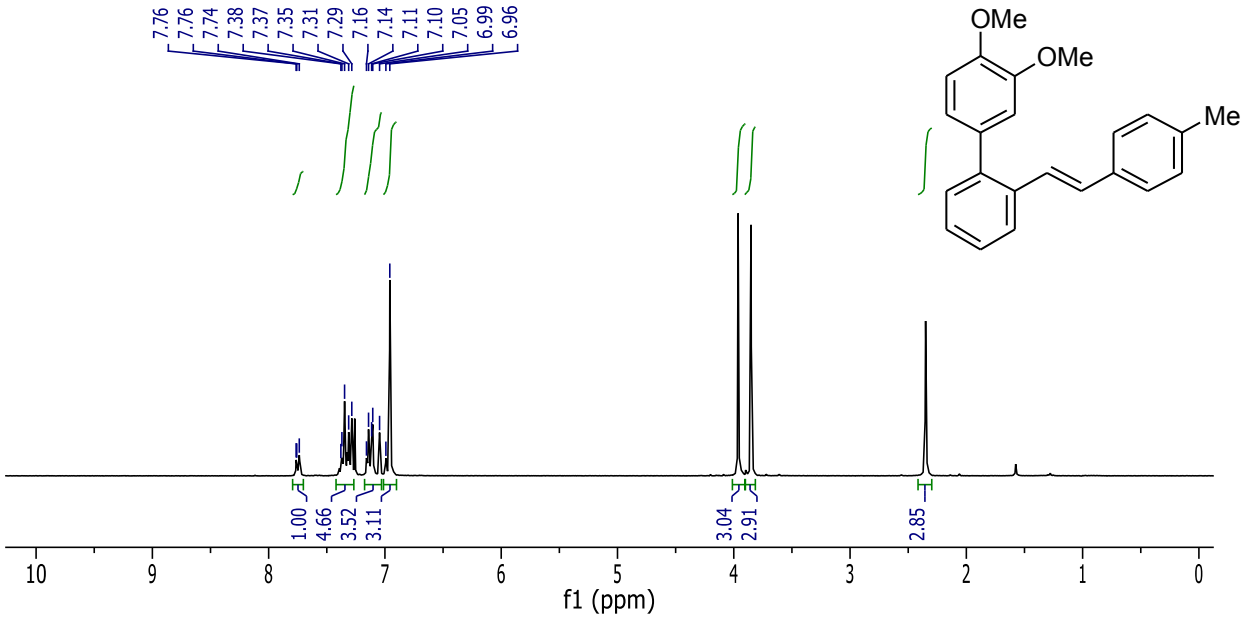
(*E*)-*tert*-Butyl((4-(3',4'-dimethoxy-[1,1'-biphenyl]-2-yl)but-3-en-1-yl)oxy)dimethyl-silane

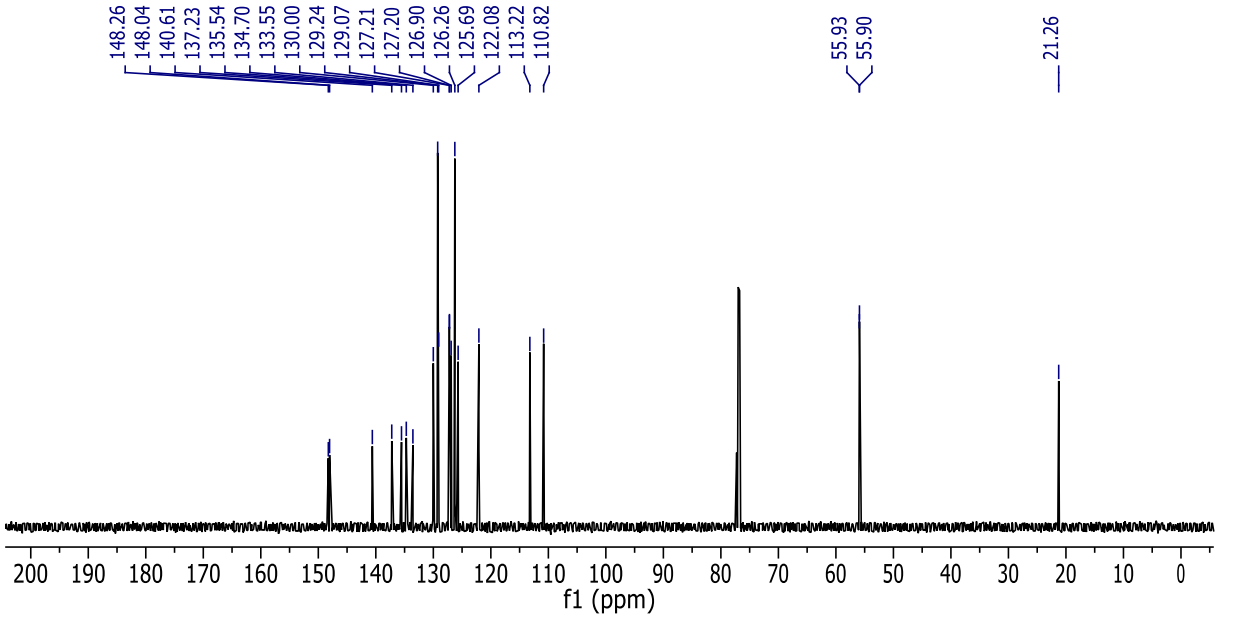


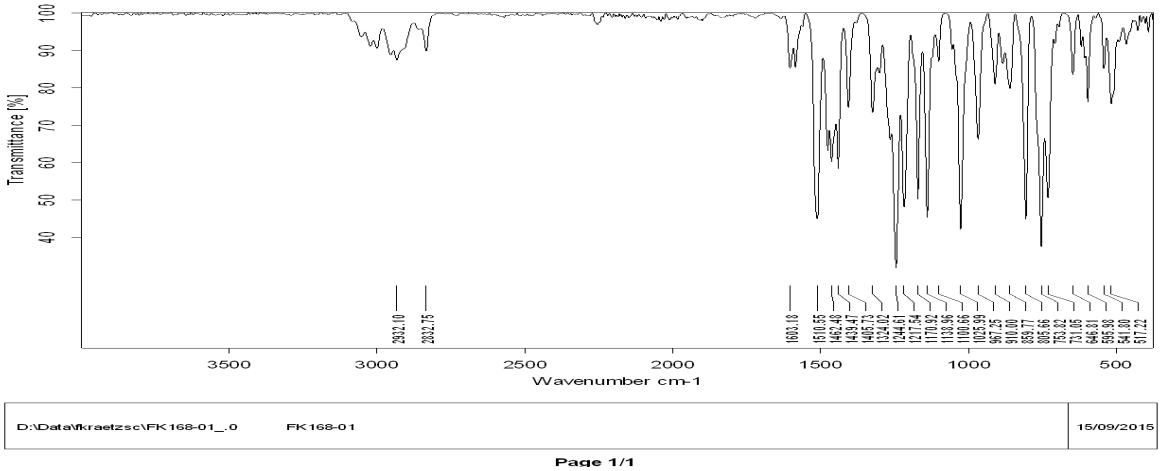




(*E*)-3',4'-Dimethoxy-2-(4-methylstyryl)-1,1'-biphenyl







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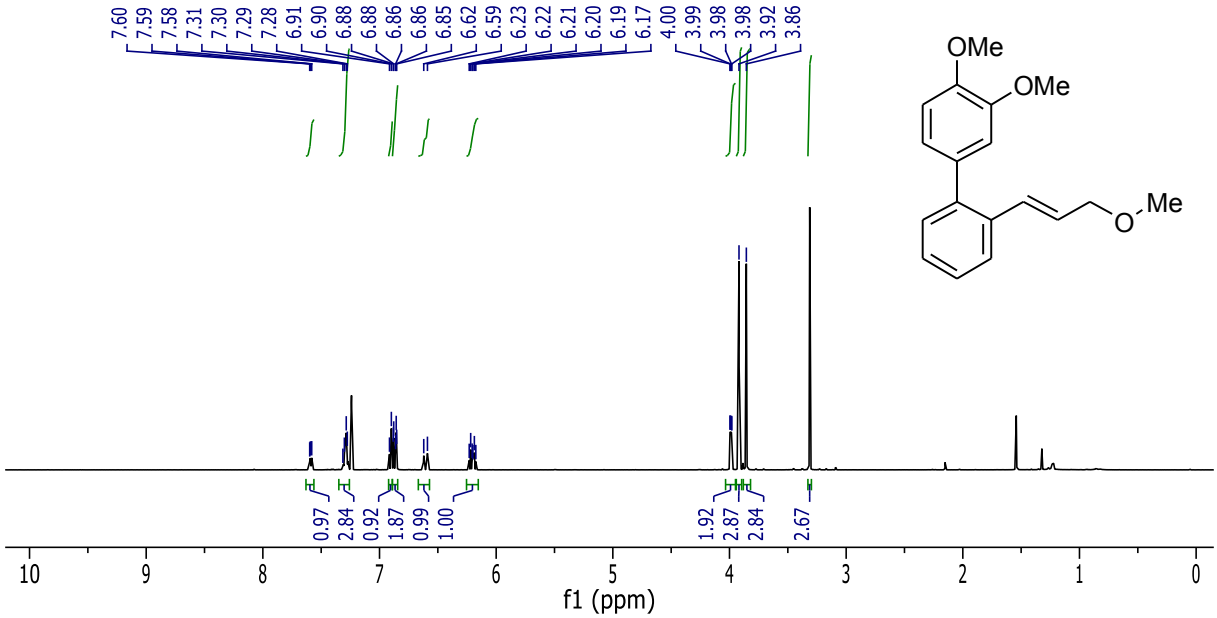
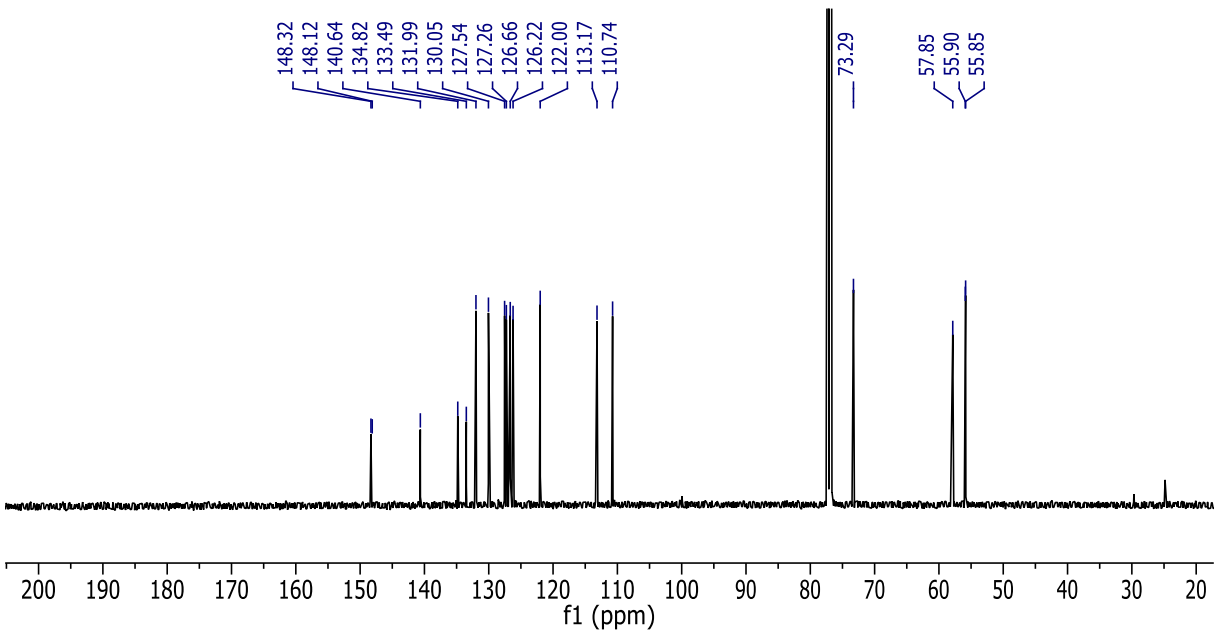
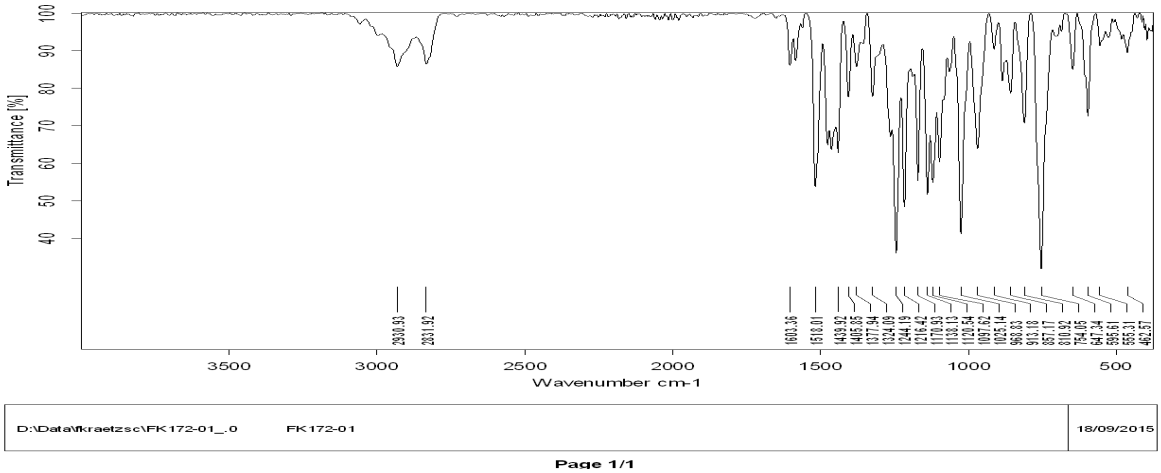
(*E*)-3',4'-Dimethoxy-2-(3-methoxyprop-1-en-1-yl)-1,1'-biphenyl







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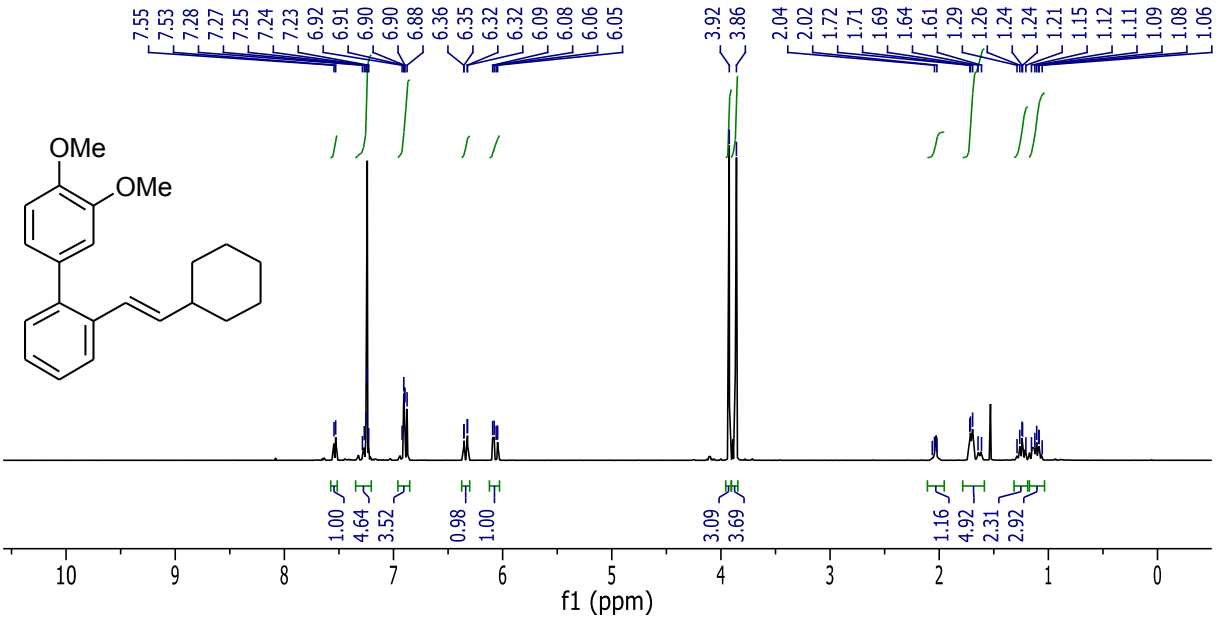
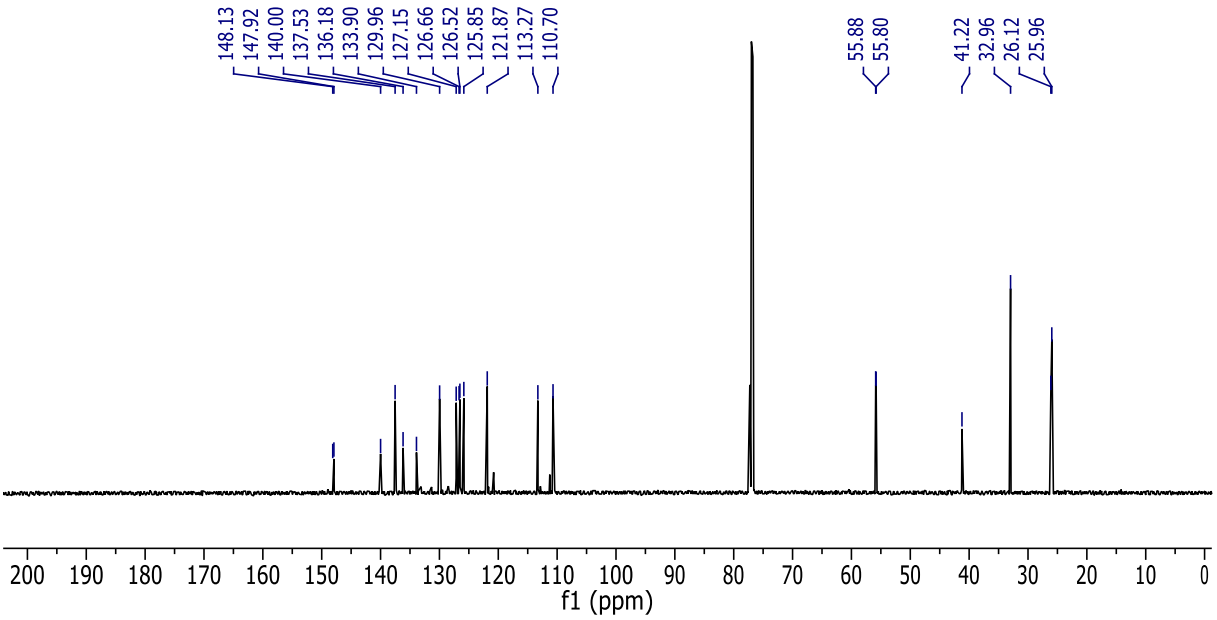
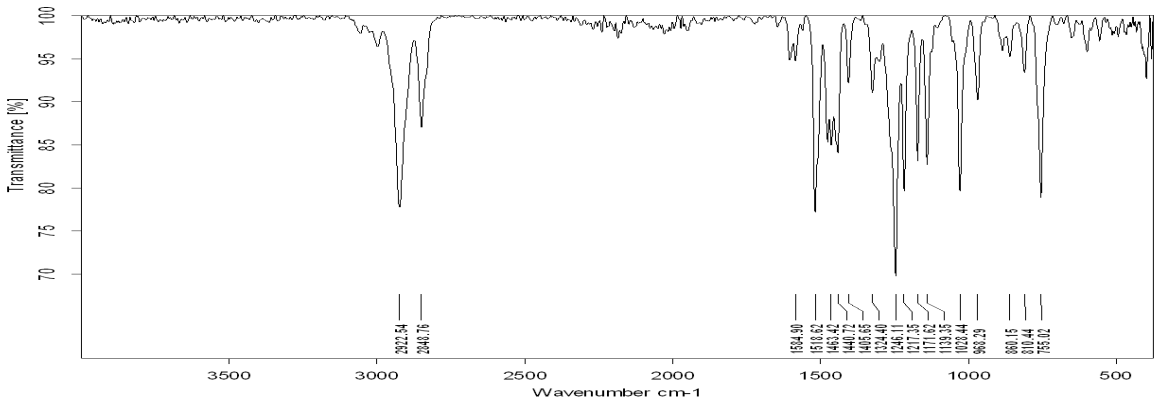
(*E*)-2-(2-Cyclohexylvinyl)-3',4'-dimethoxy-1,1'-biphenyl







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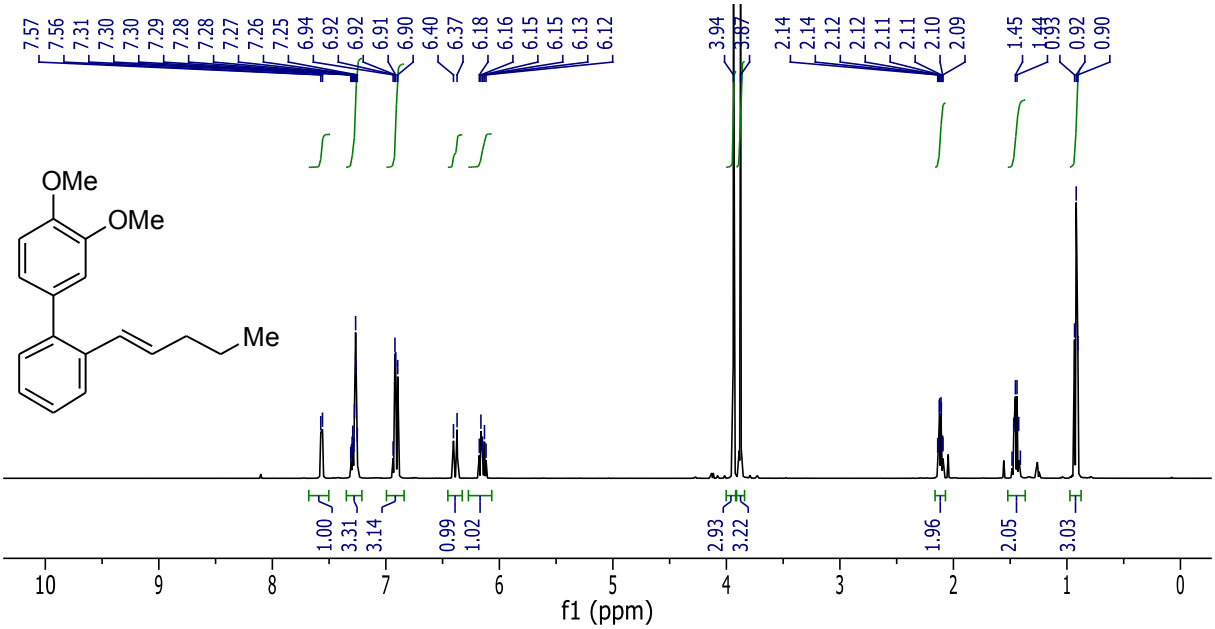
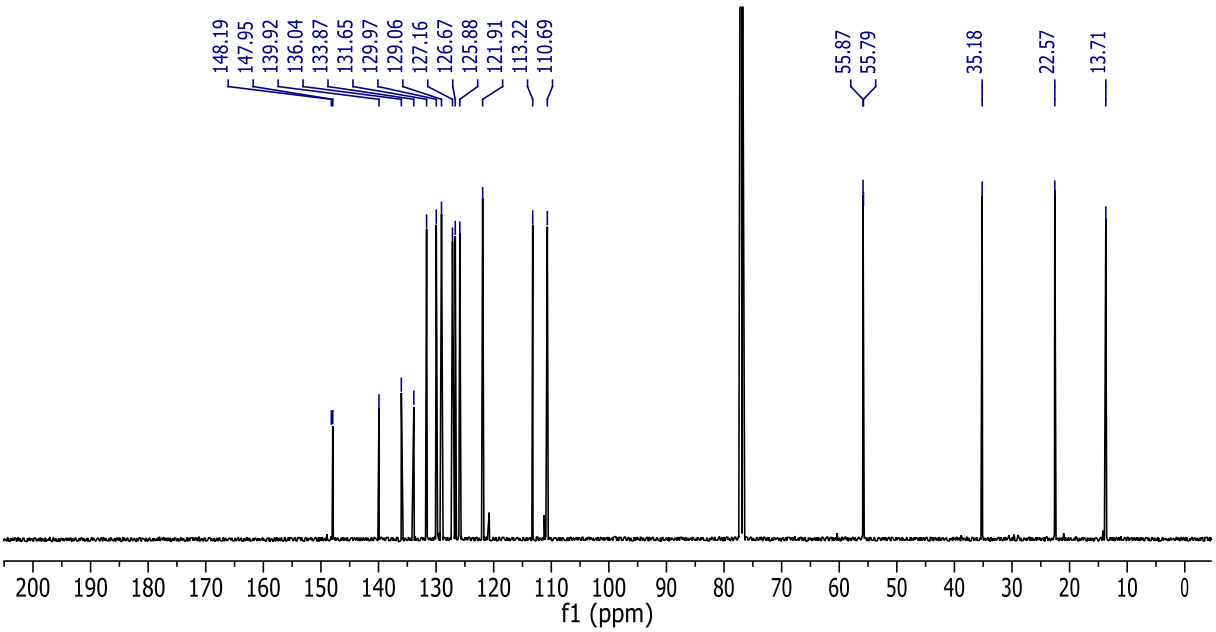
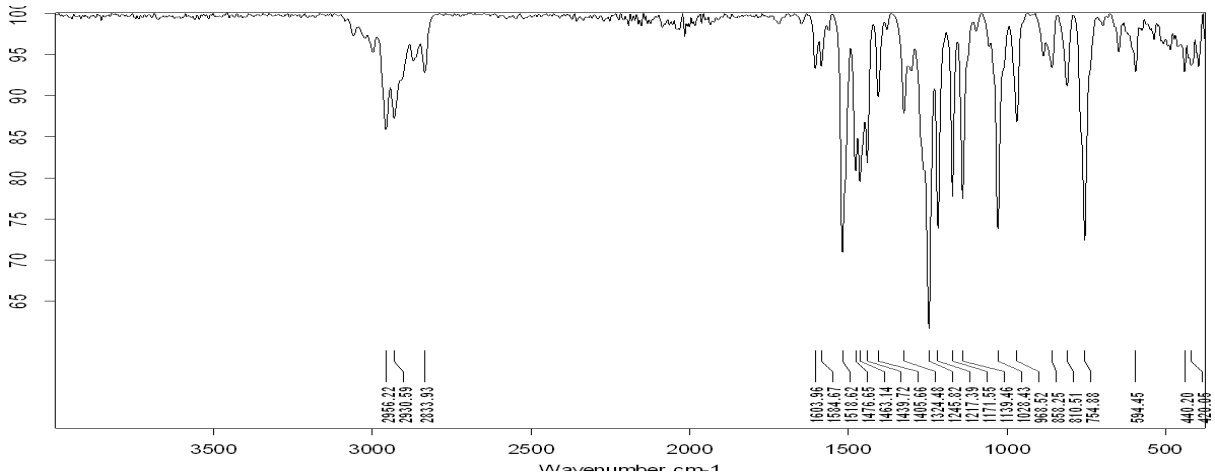
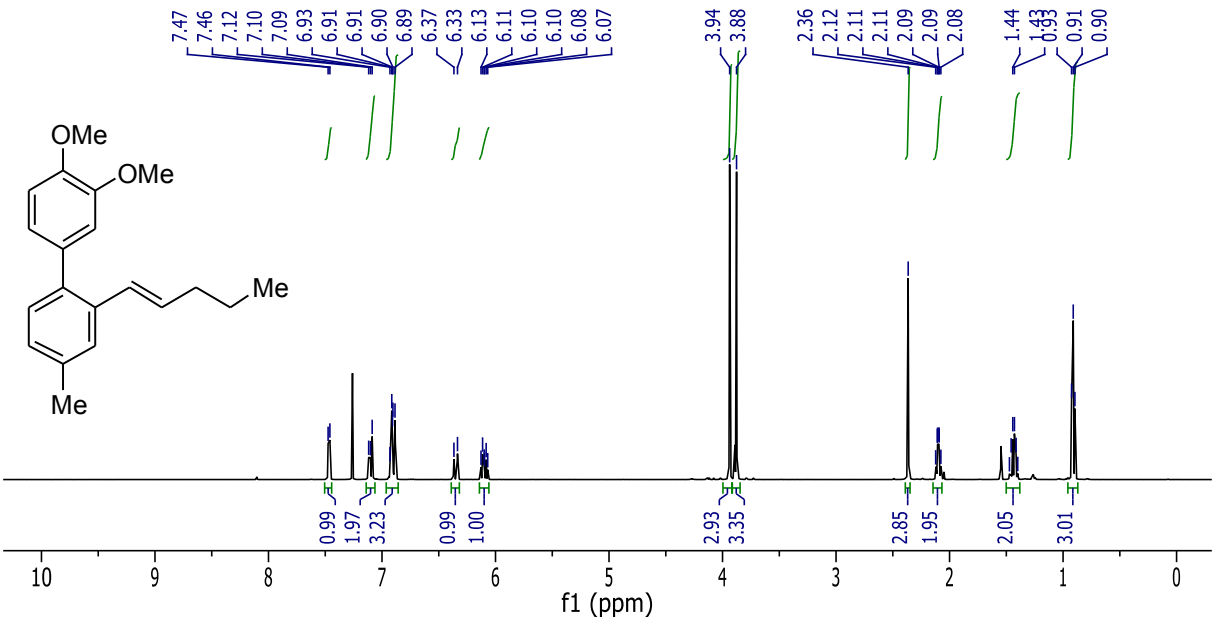
(*E*)-3',4'-Dimethoxy-2-(pent-1-en-1-yl)-1,1'-biphenyl

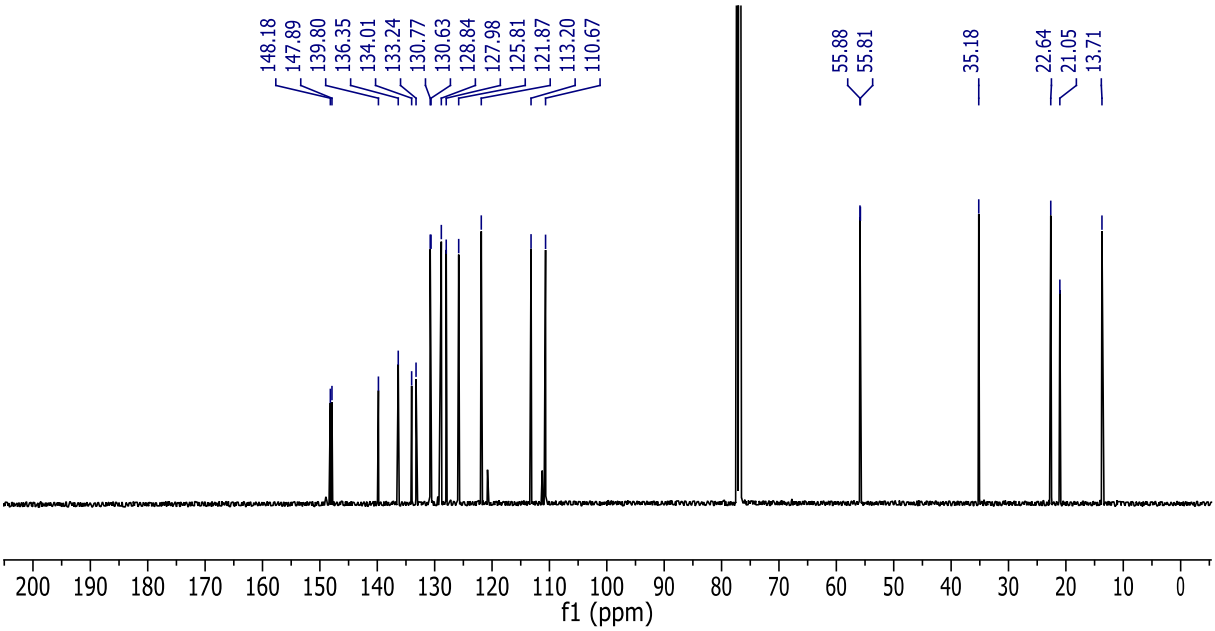


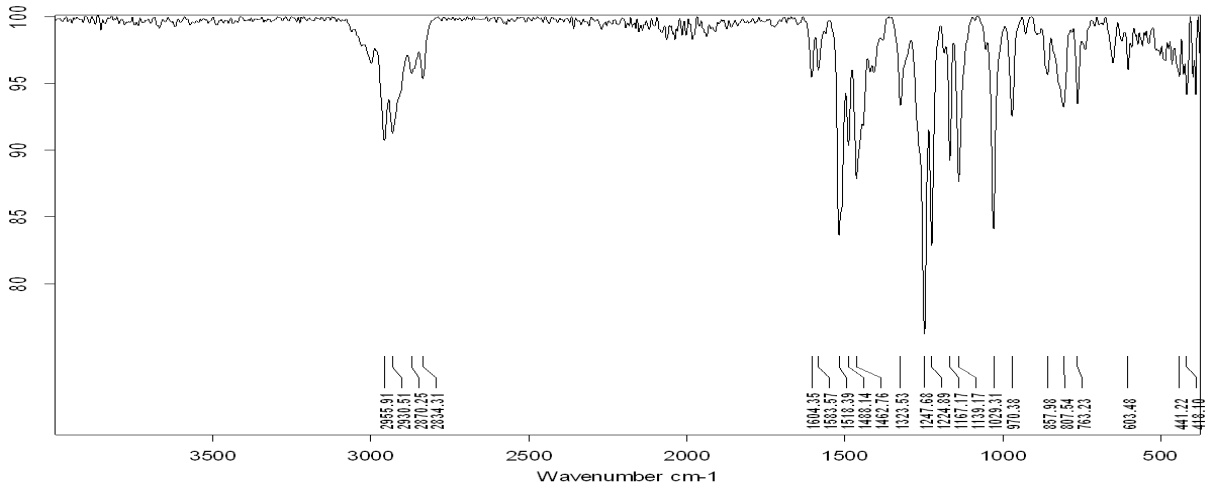




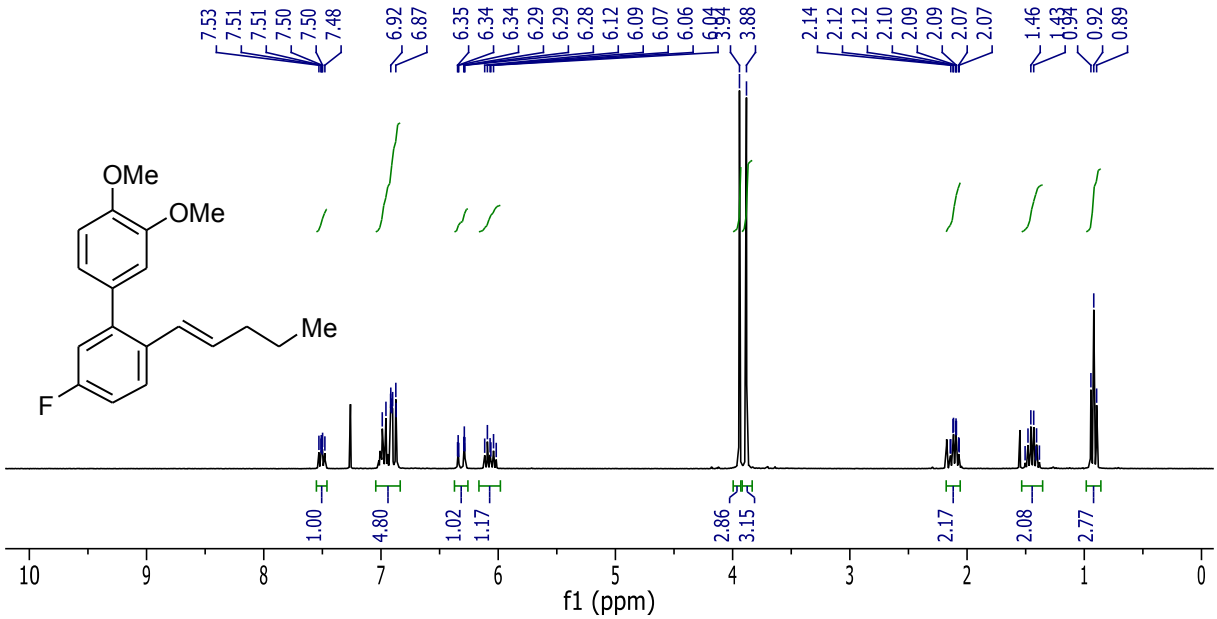
(*E*)-3',4'-Dimethoxy-4-methyl-2-(pent-1-en-1-yl)-1,1'-biphenyl

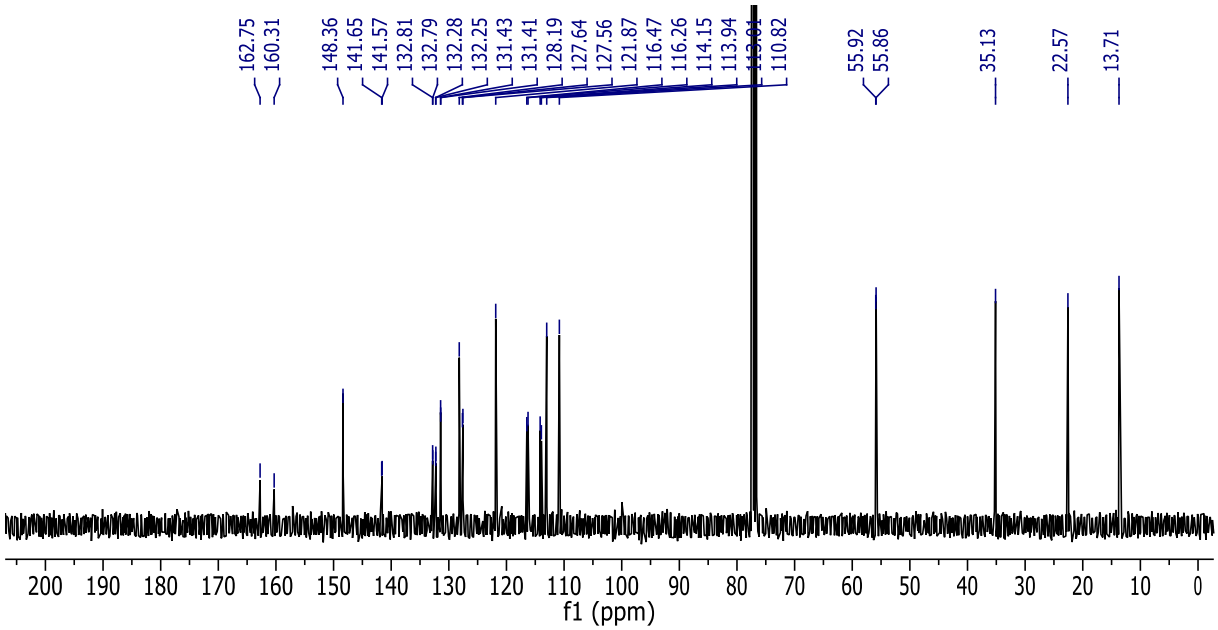


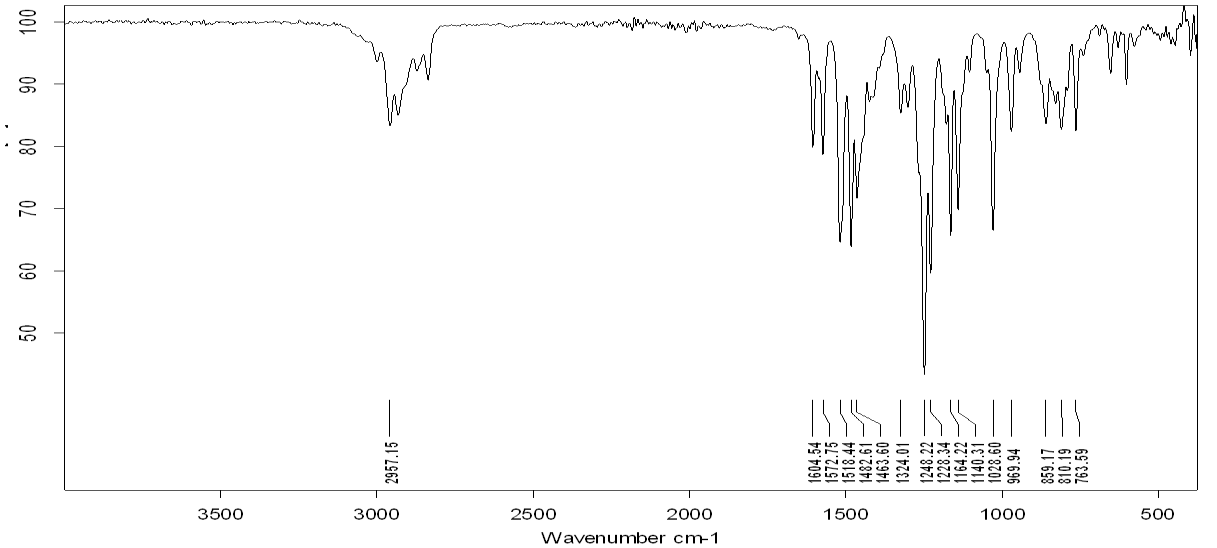




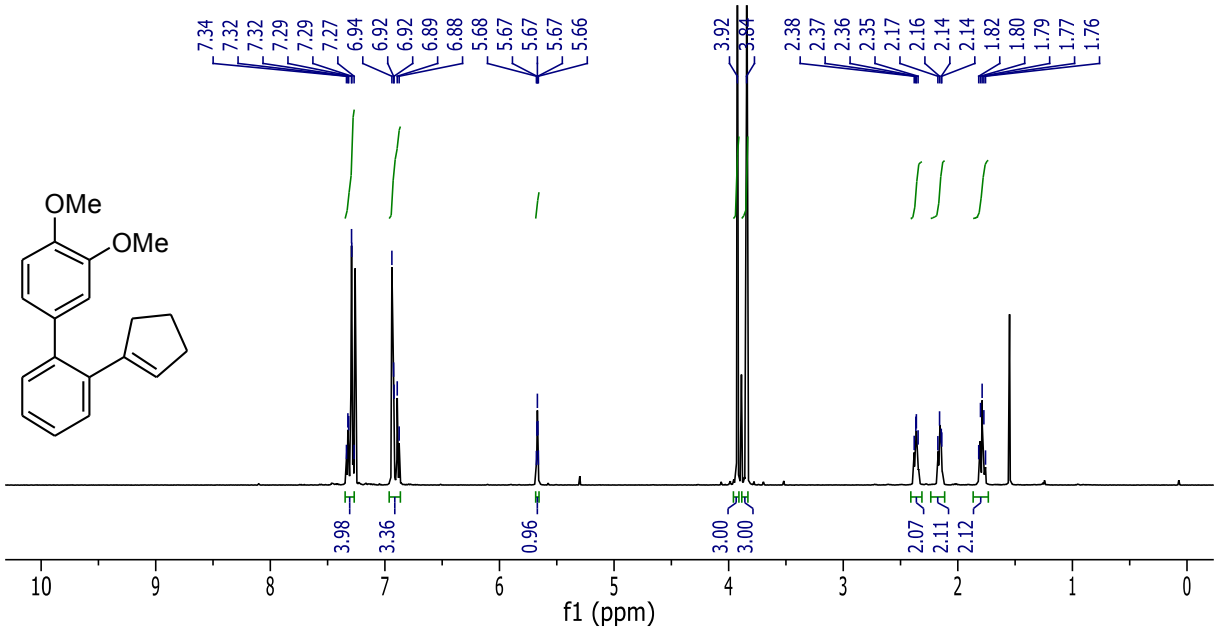
(*E*)-5-Fluoro-3',4'-dimethoxy-2-(pent-1-en-1-yl)-1,1'-biphenyl

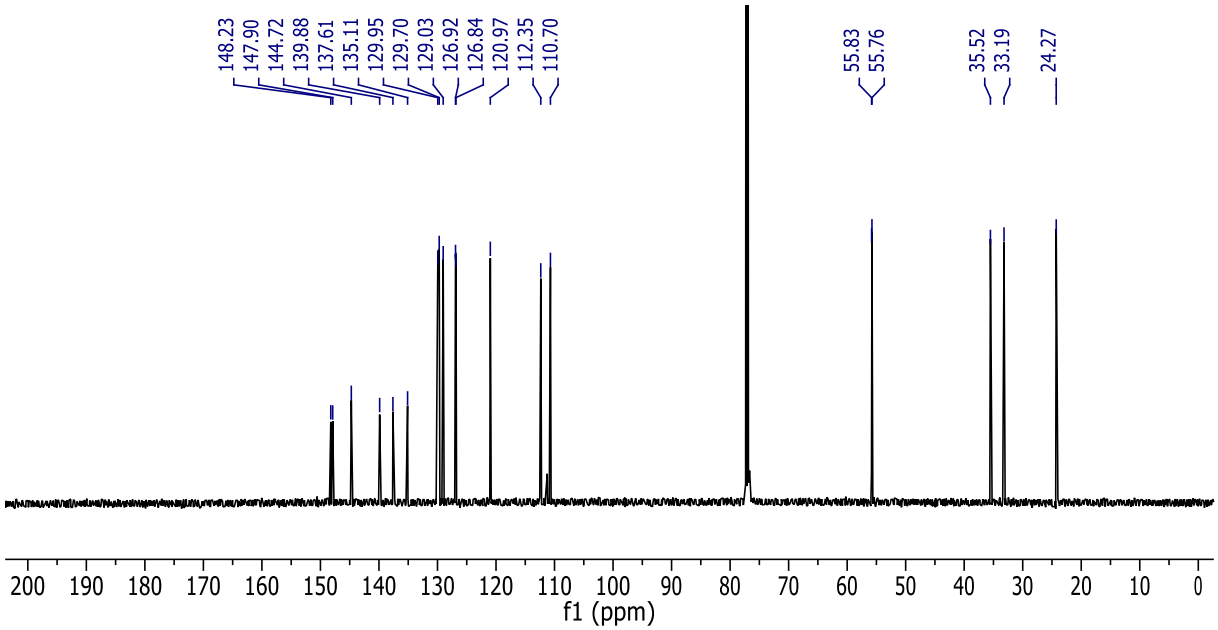


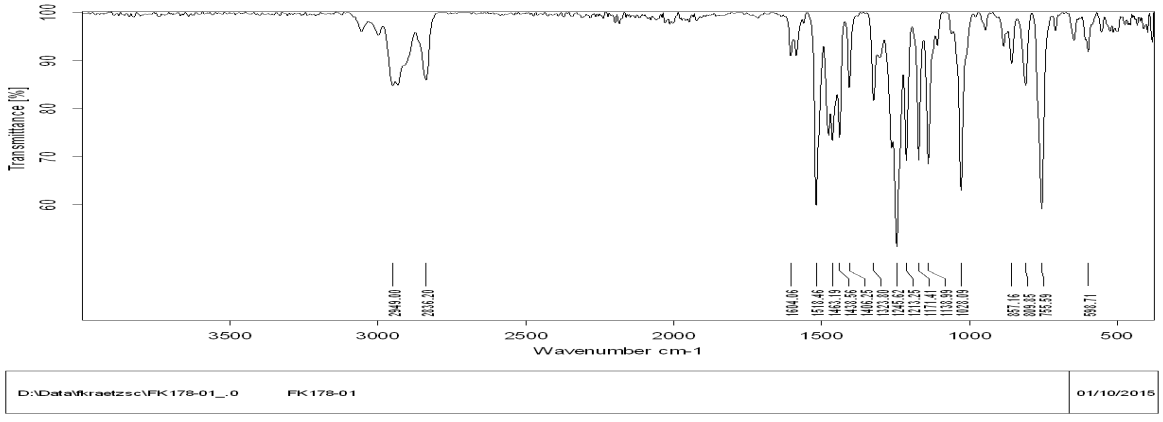




2-(Cyclopent-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl







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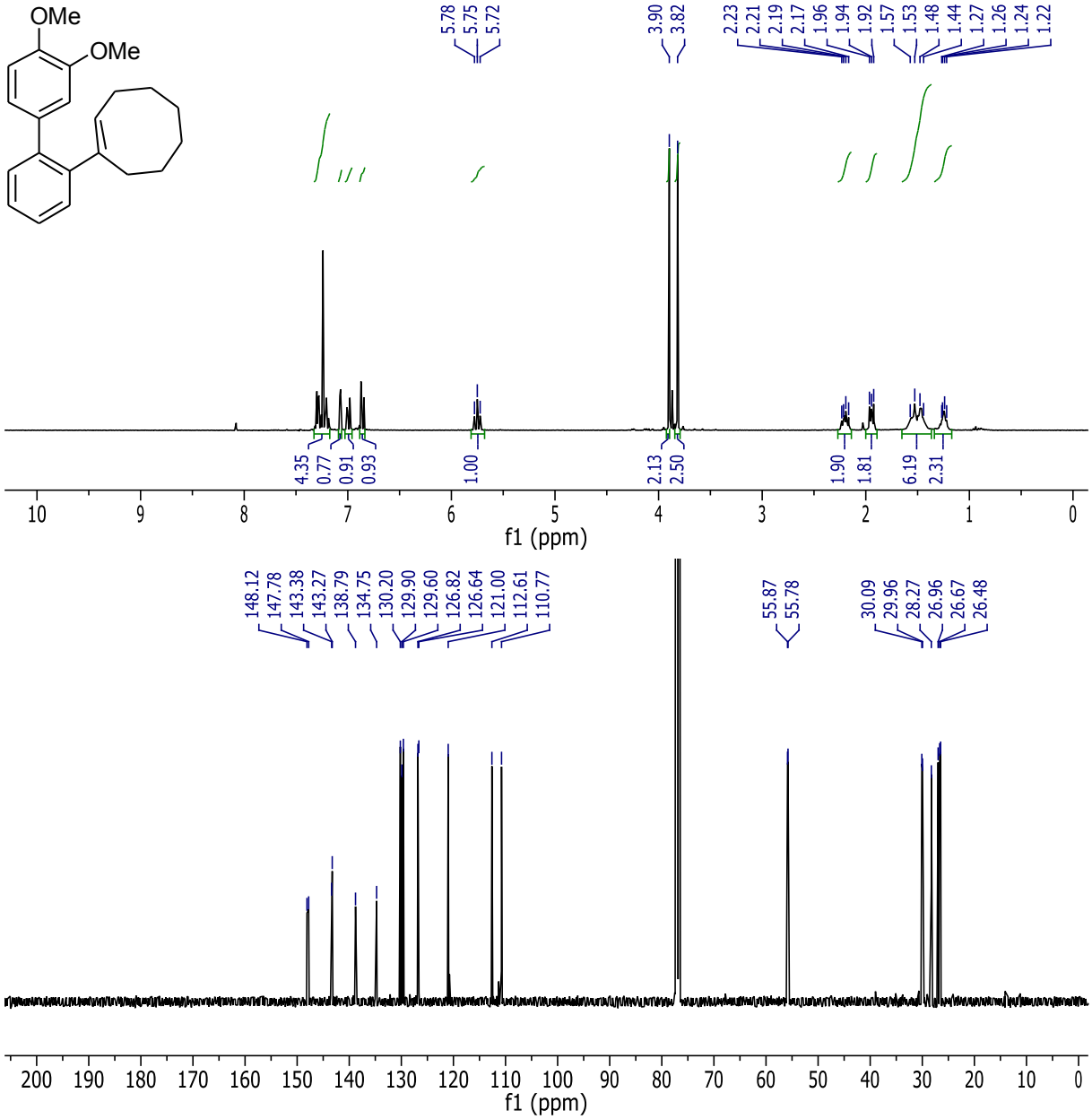
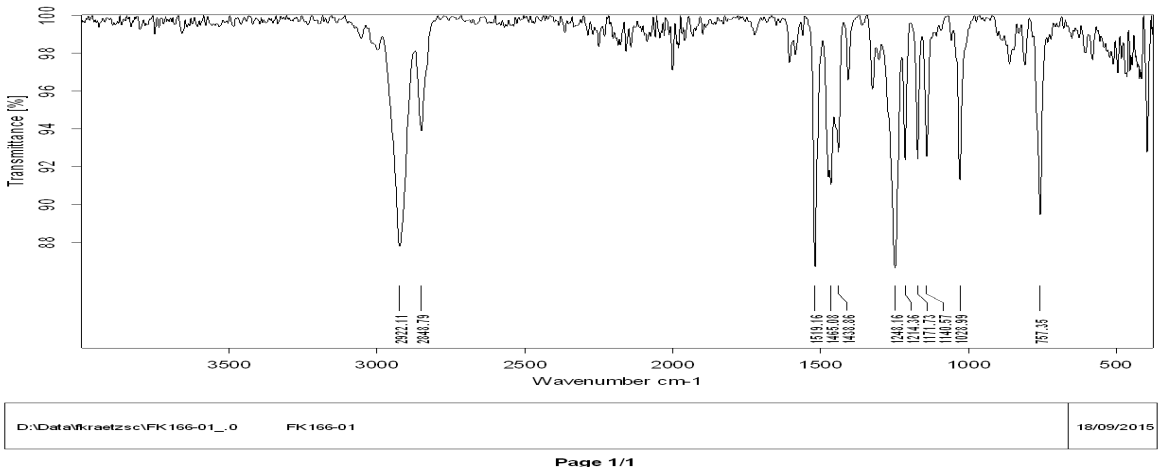
(*E*)-2-(Cyclooct-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl





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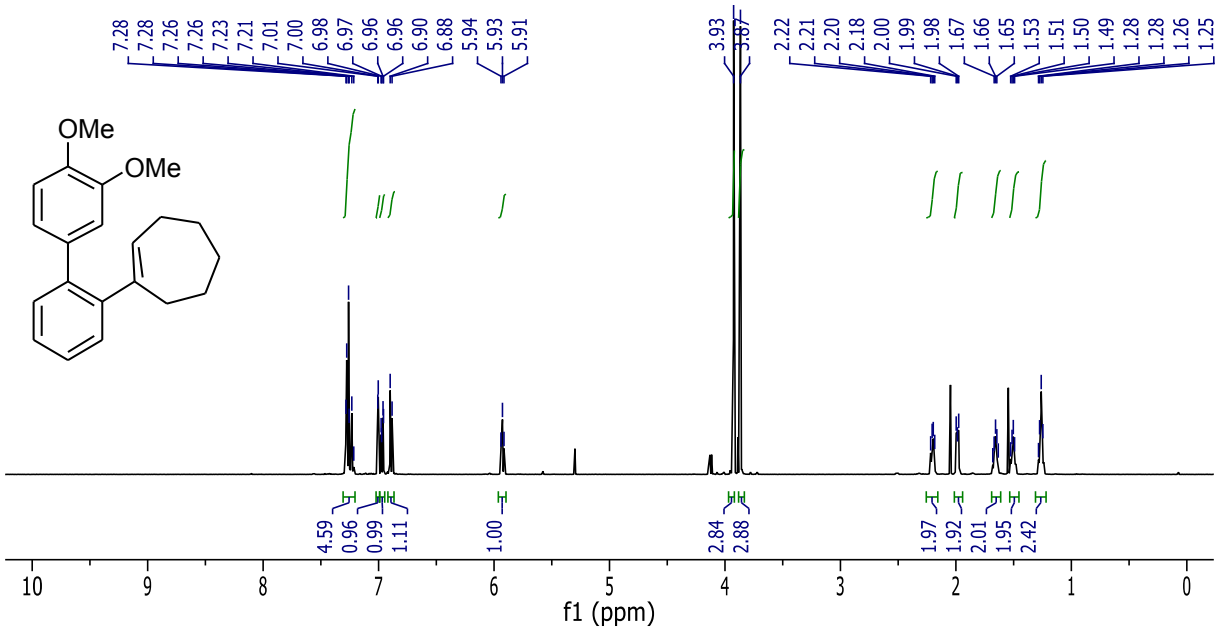
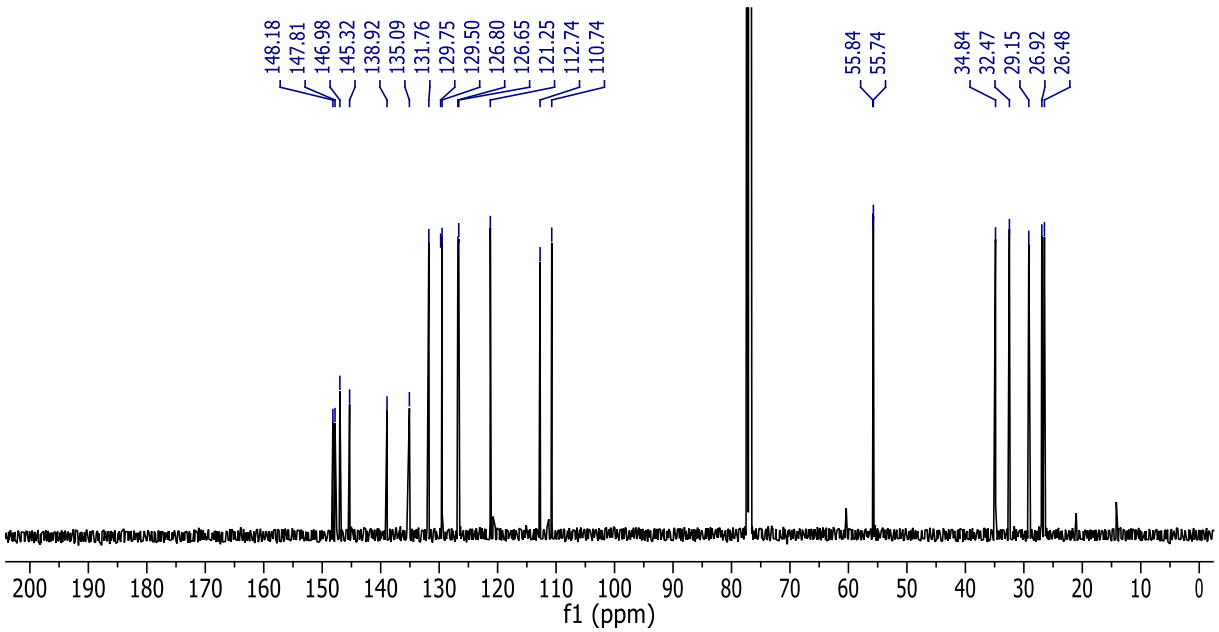
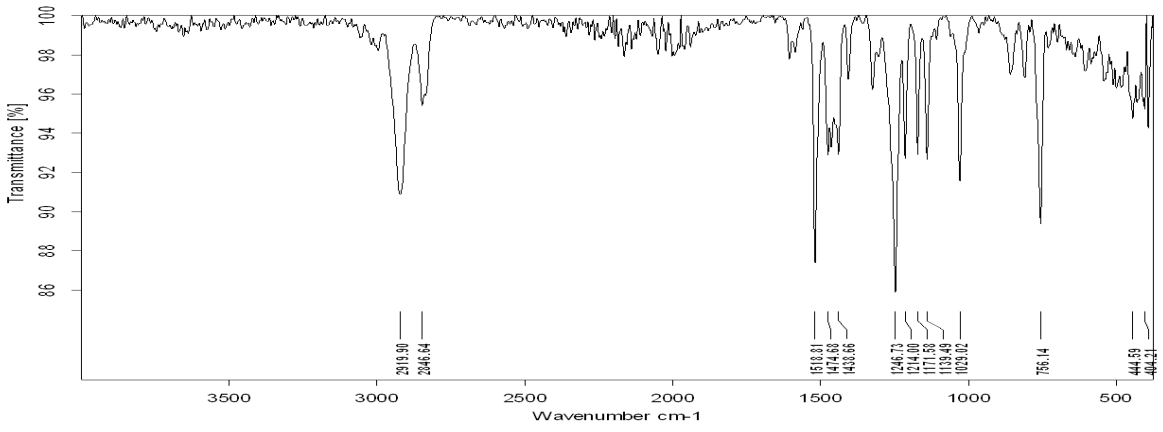
2-(Cyclohept-1-en-1-yl)-3',4'-dimethoxy-1,1'-biphenyl







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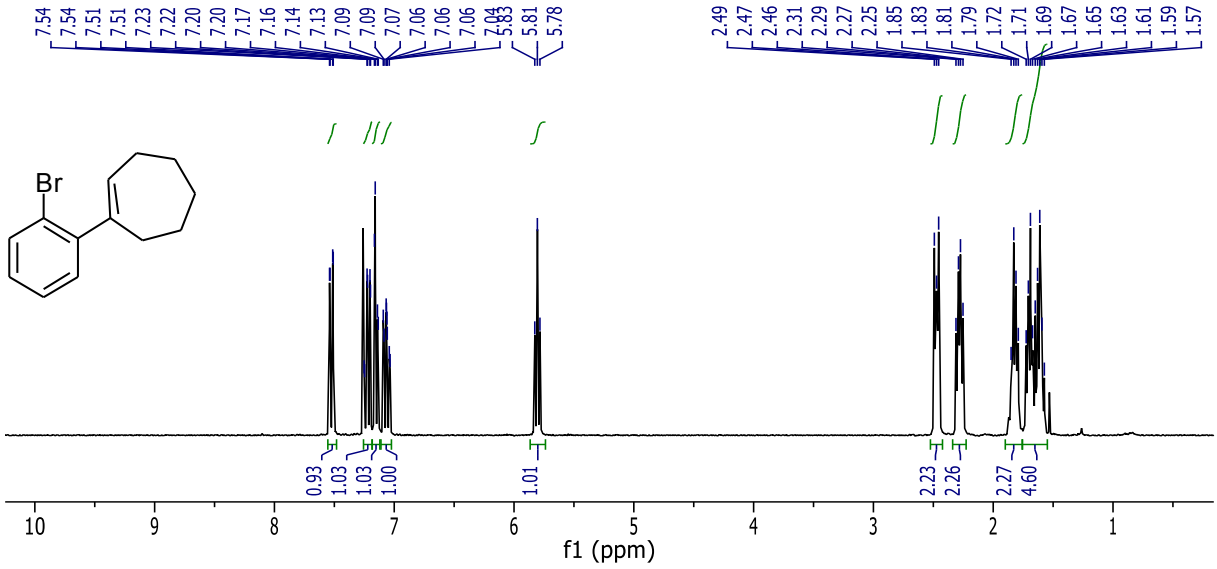
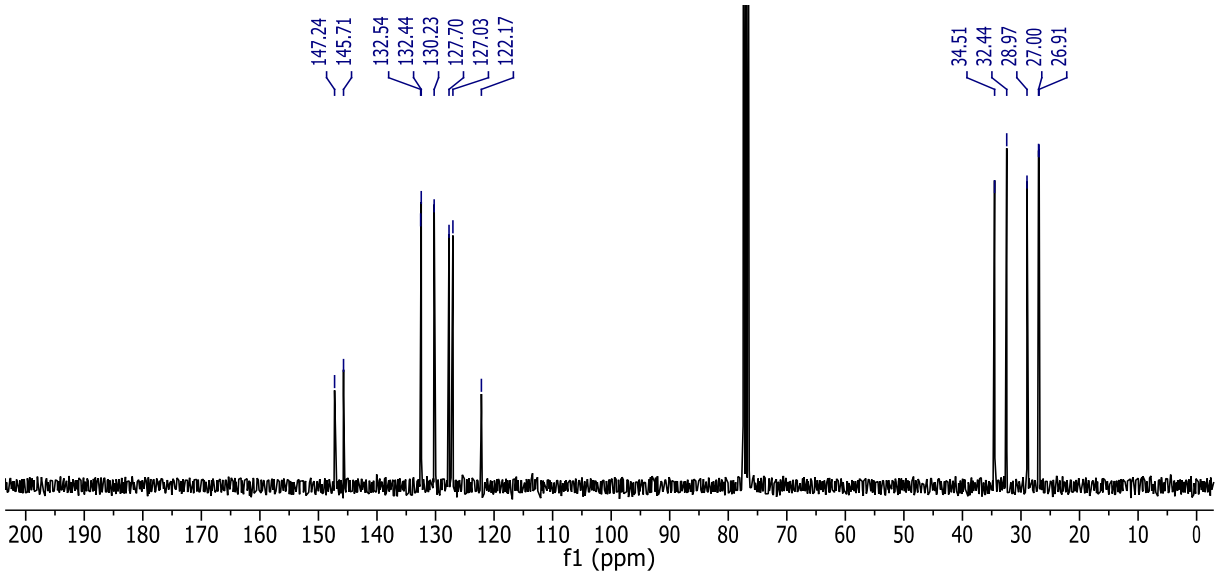
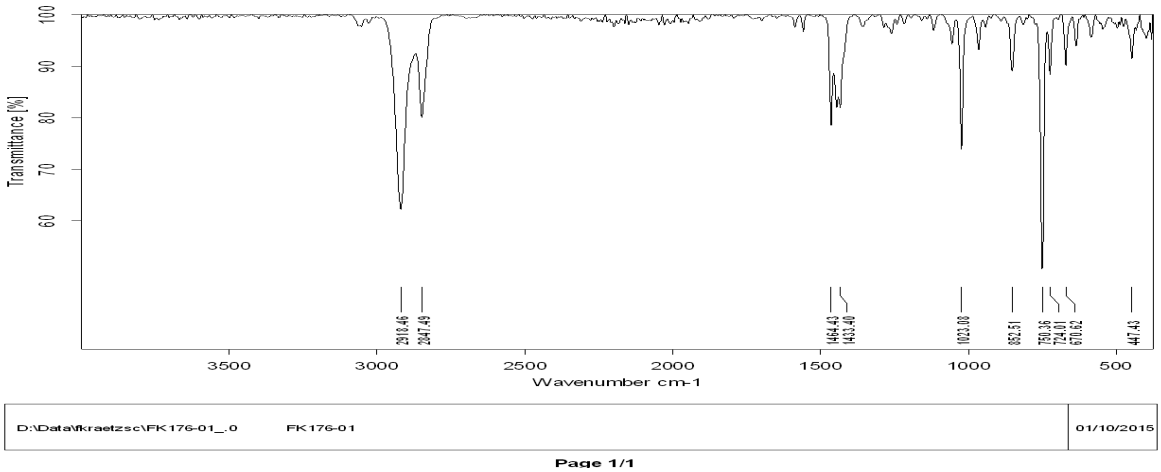
1-(2-Bromophenyl)cyclohept-1-ene







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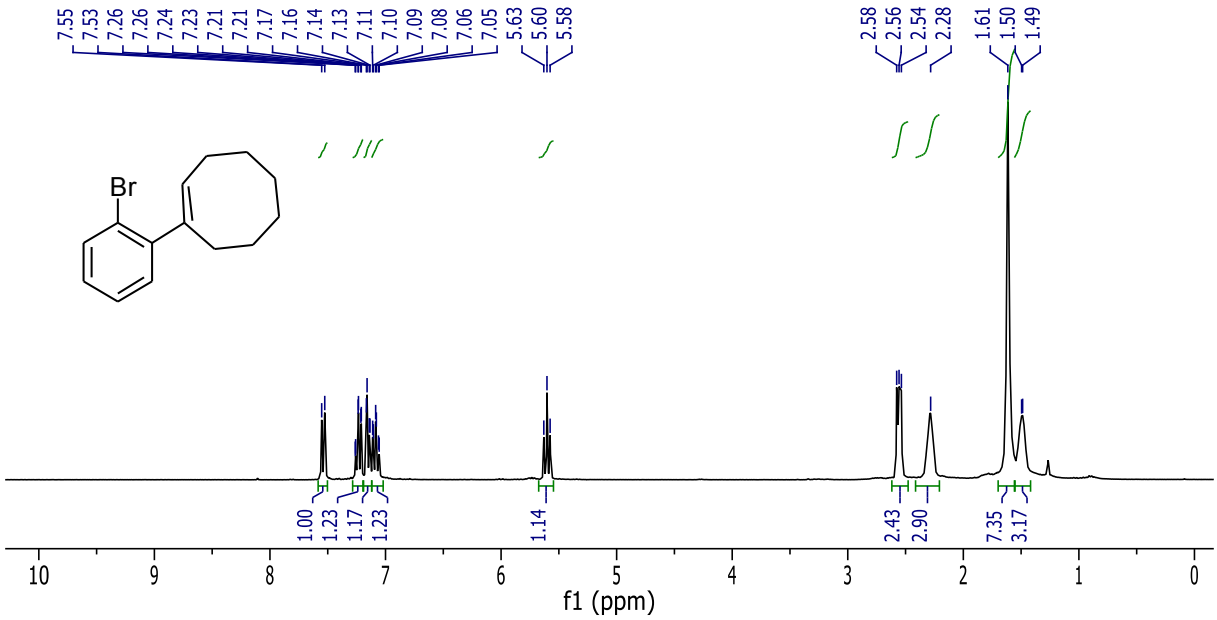
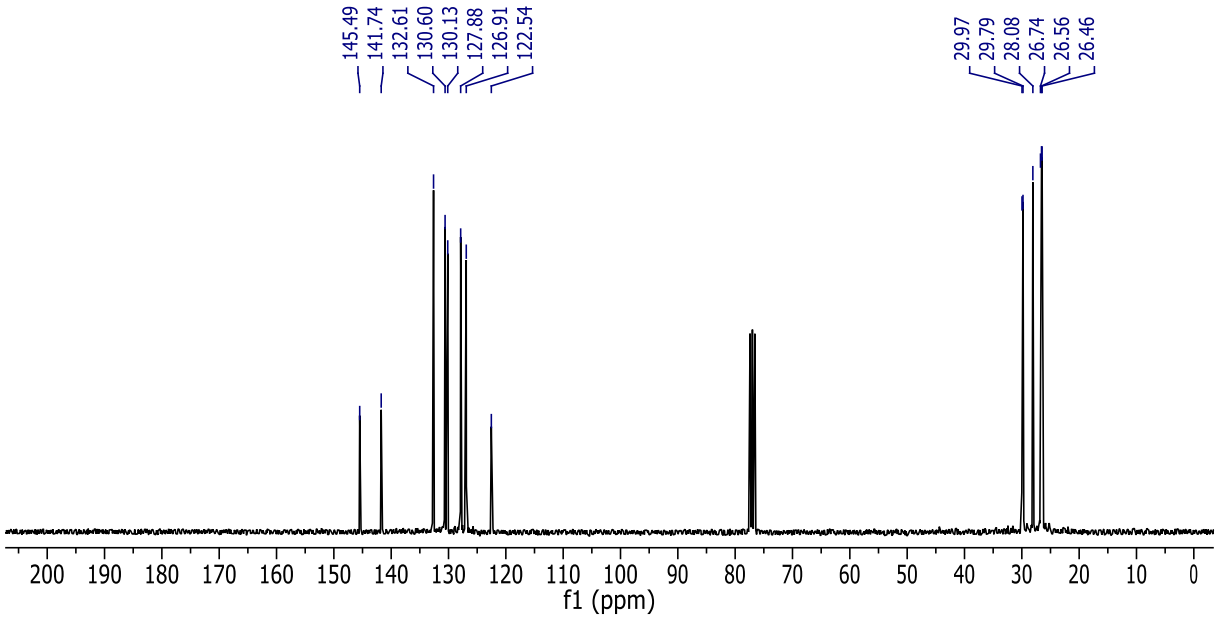
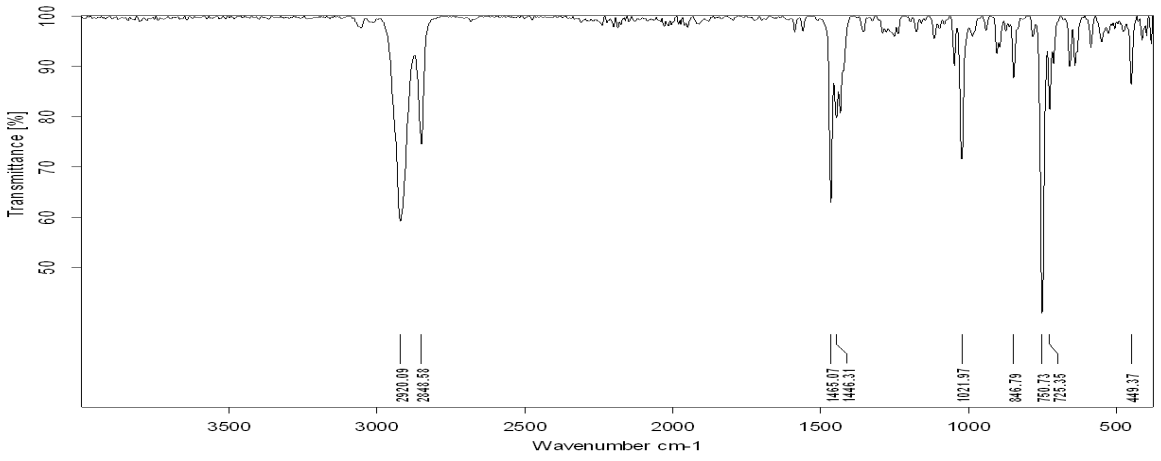
(*E*)-1-(2-Bromophenyl)cyclooct-1-ene







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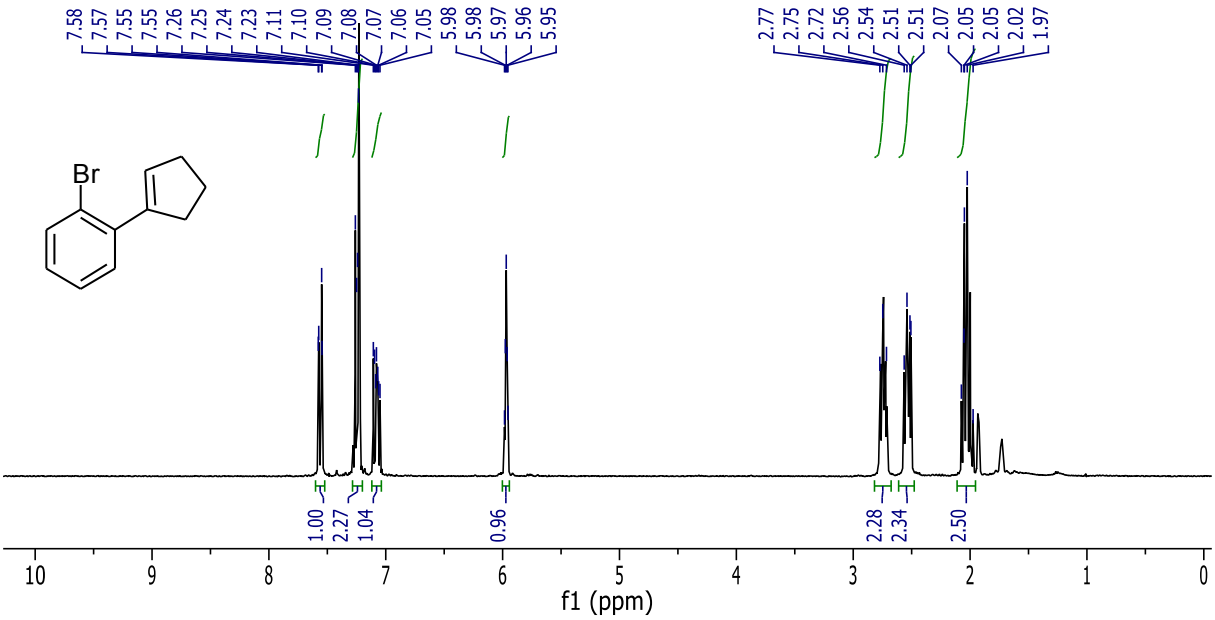
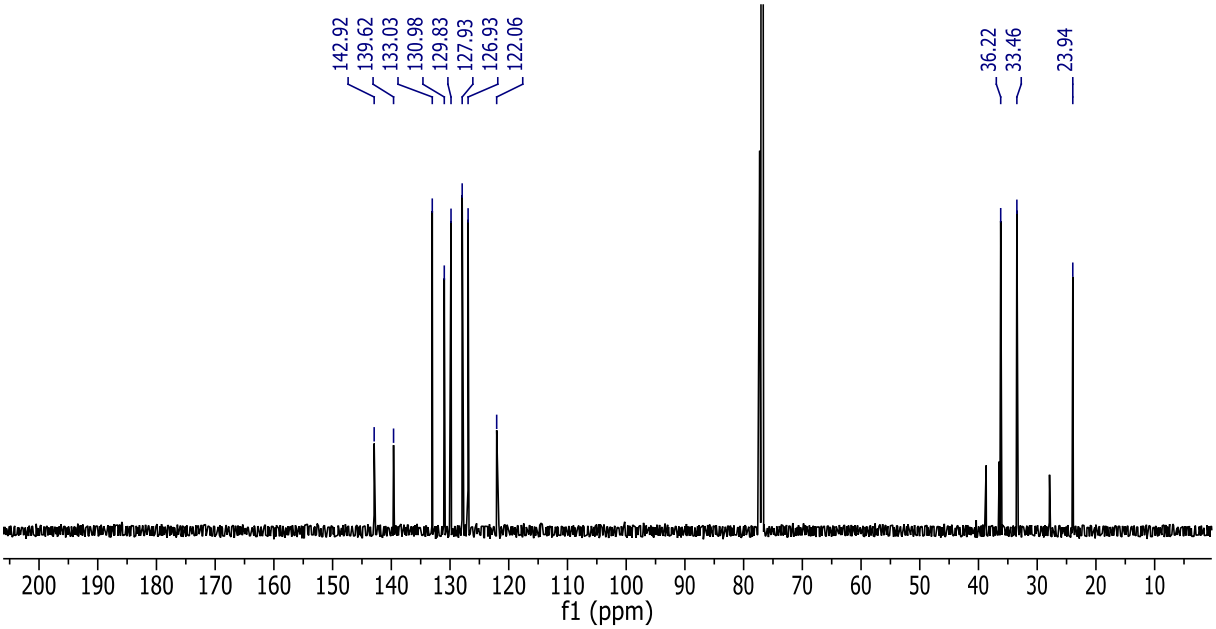
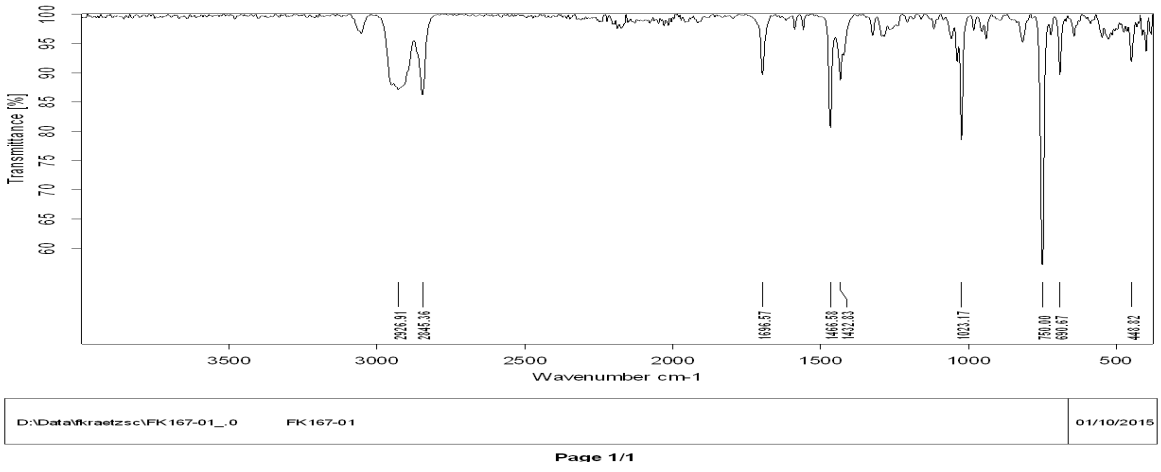
1-Bromo-2-(cyclopent-1-en-1-yl)benzene







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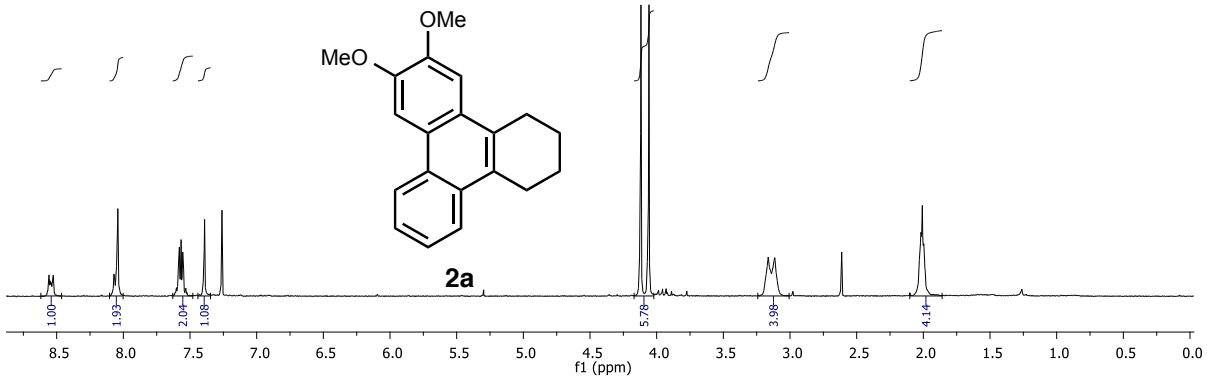
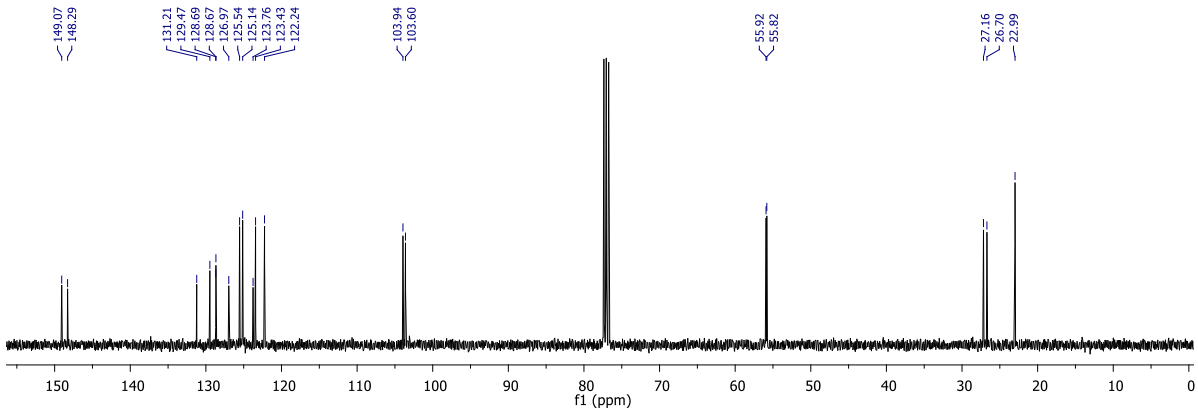
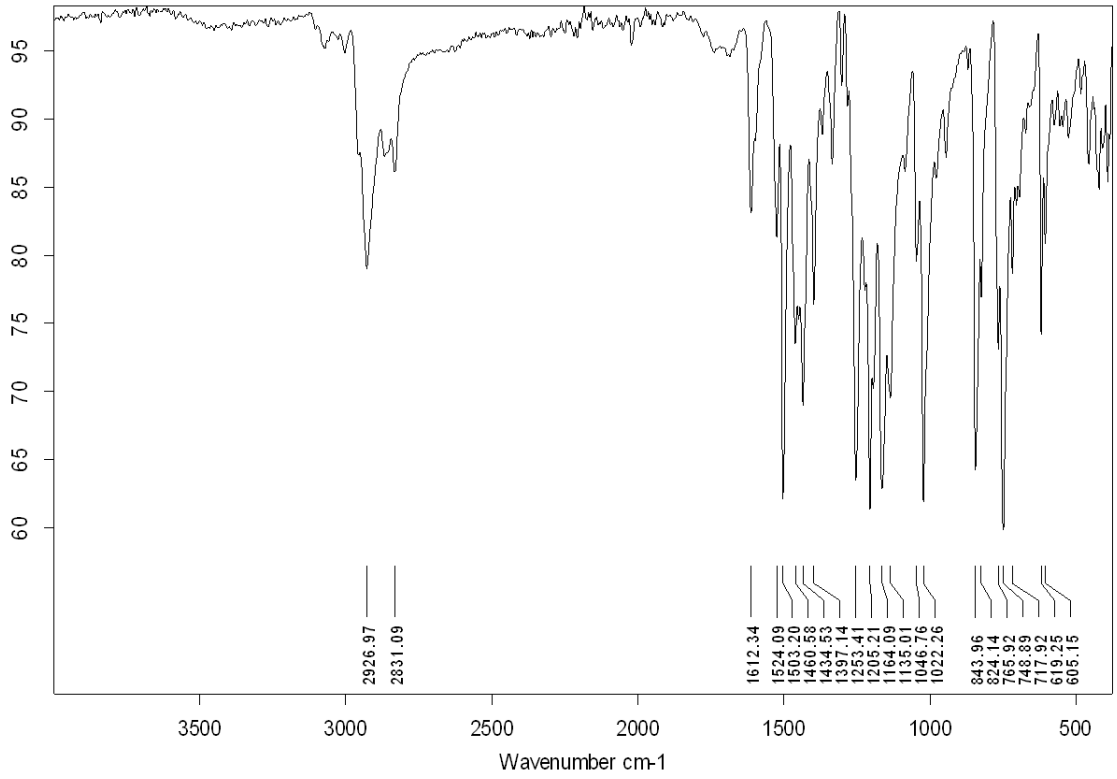
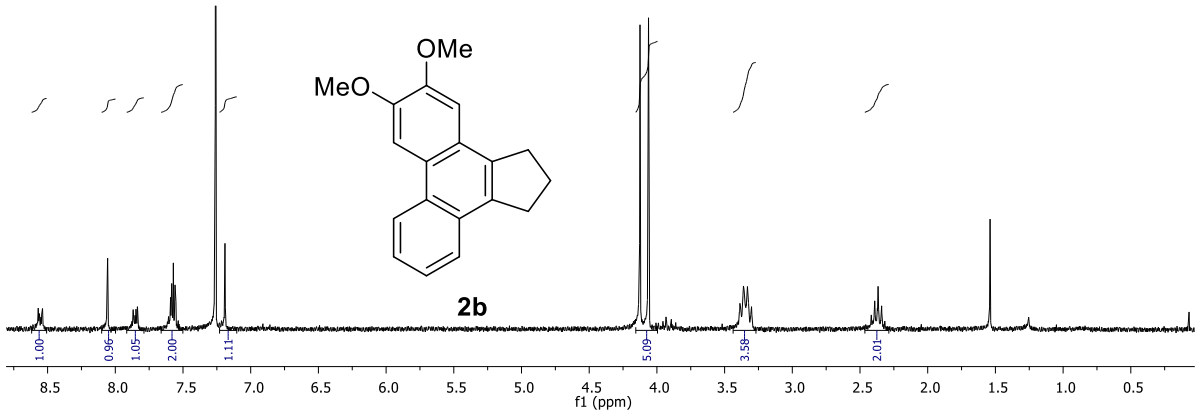
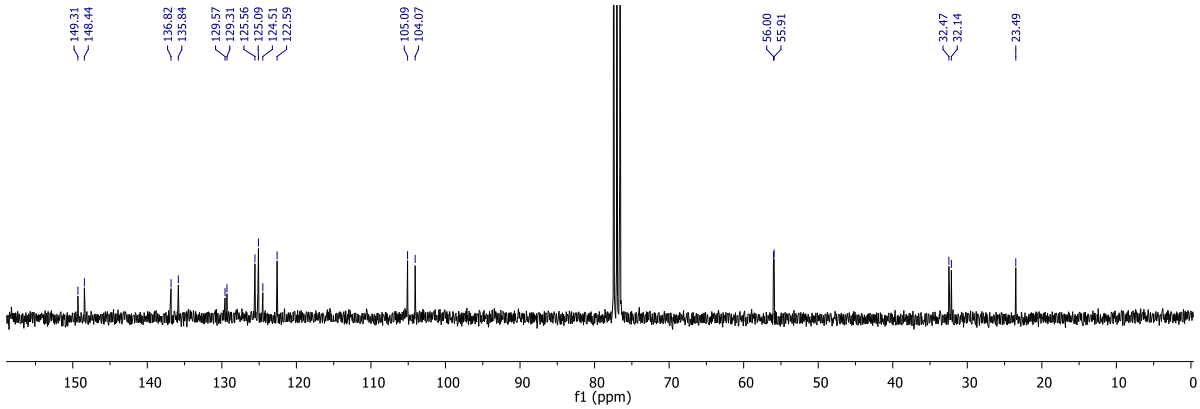
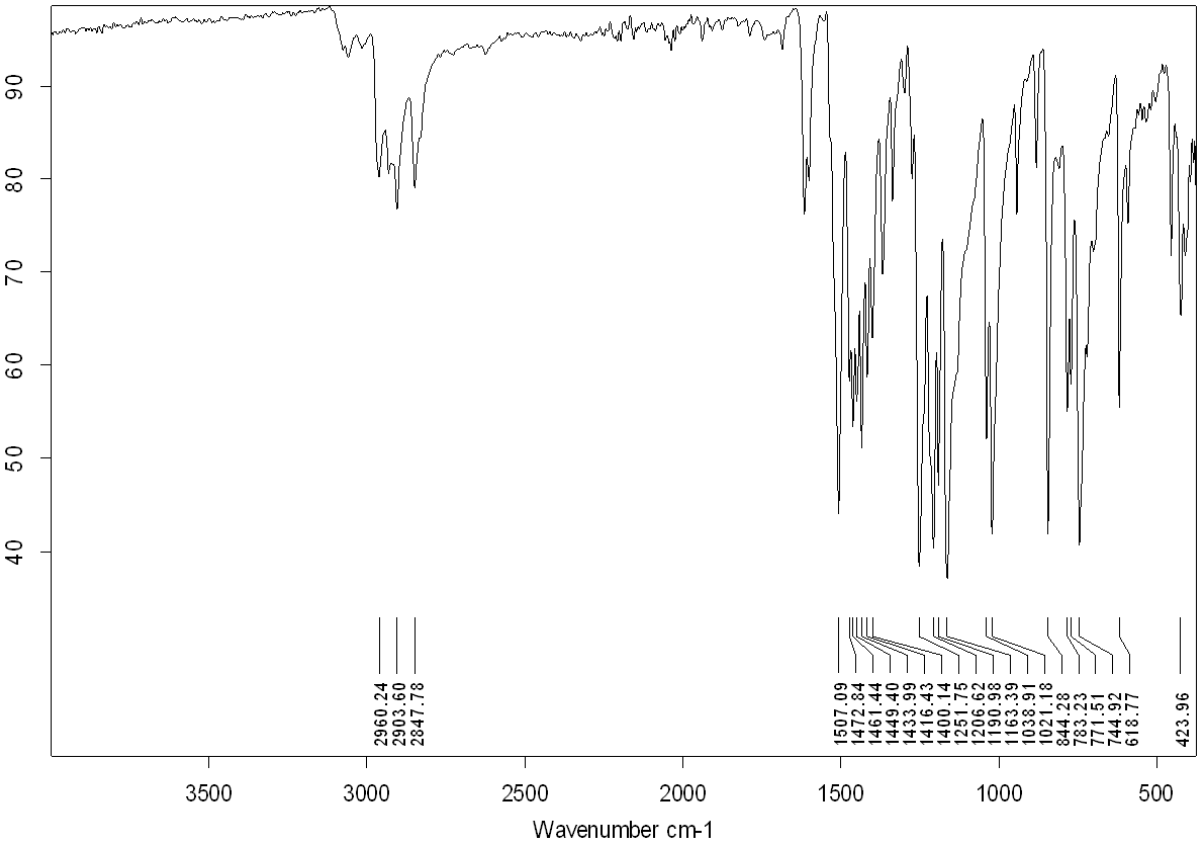

OMe
MeO
2a
1.00
1.93
2.04
1.08
5.78
3.98
4.14
f1 (ppm)

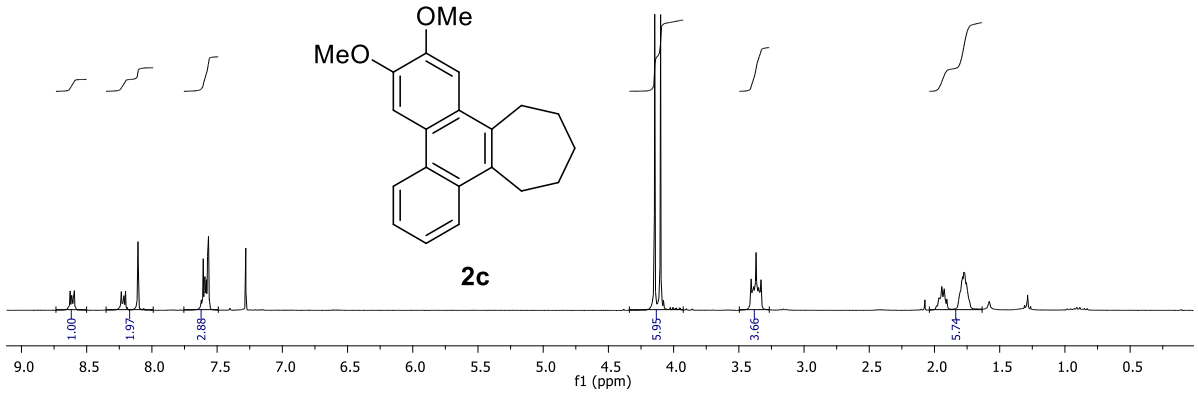

149.07
148.29
131.21
129.47
128.69
128.67
126.97
125.54
125.14
123.76
123.43
122.24
103.94
103.60
55.92
55.82
27.16
26.70
22.99
f1 (ppm)

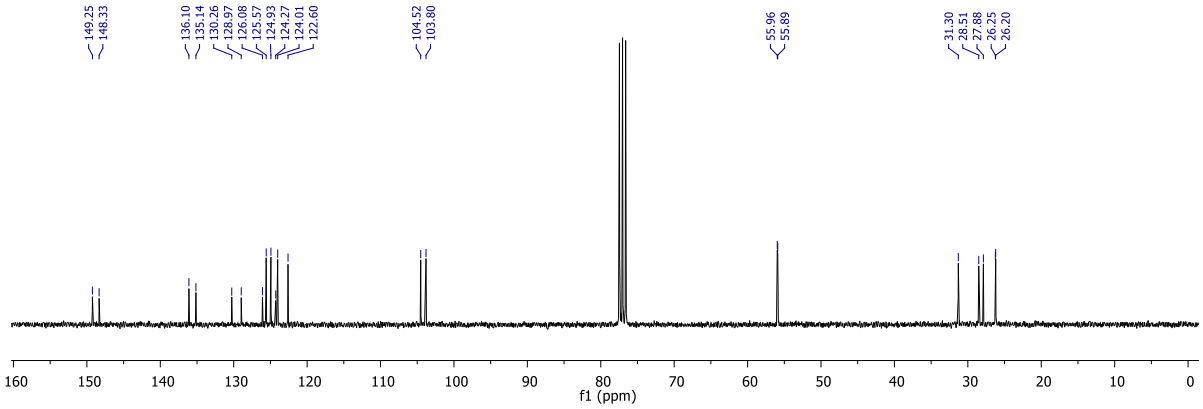

2926.97
2831.09
1612.34
1524.09
1503.20
1460.58
1434.53
1397.14
1253.41
1205.21
1164.09
1135.01
1046.76
1022.26
843.96
824.14
765.92
748.89
717.92
619.25
605.15
Wavenumber cm-1

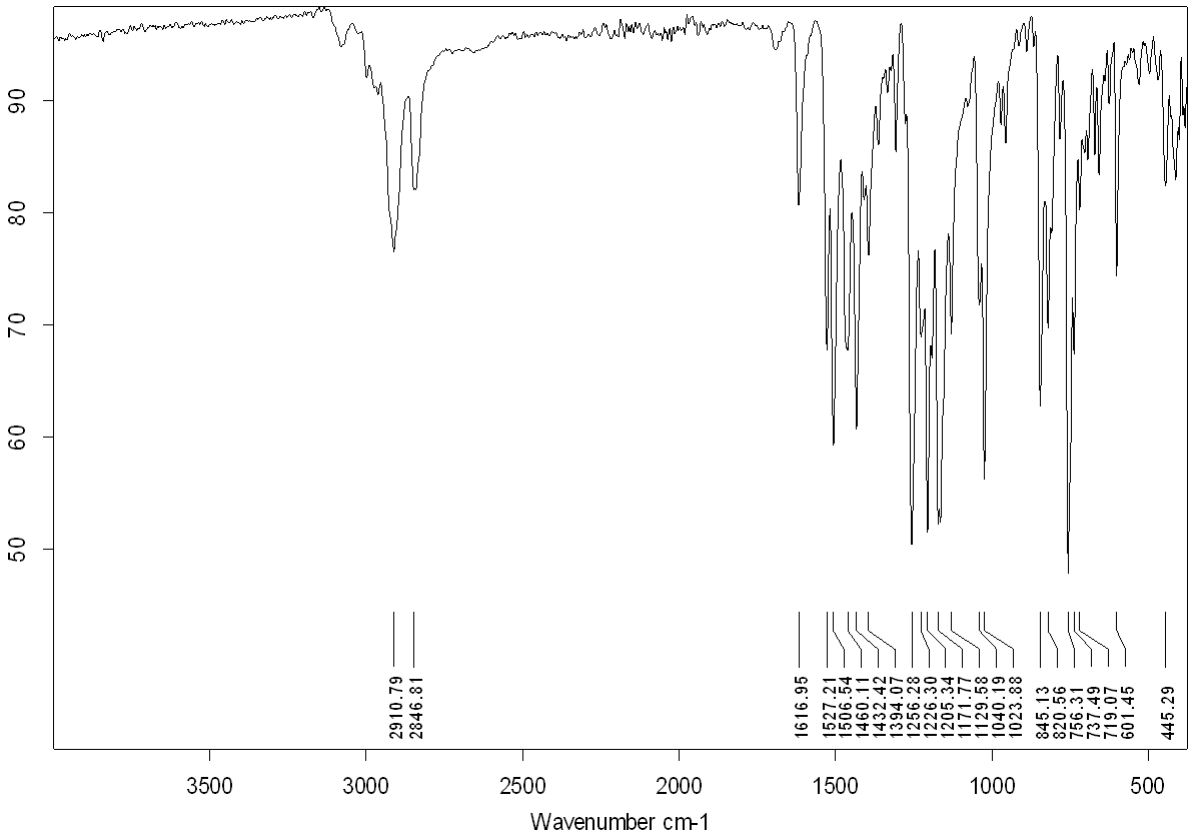

OMe
MeO
2b
1.00
0.96
1.05
2.00
1.11
5.09
3.58
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

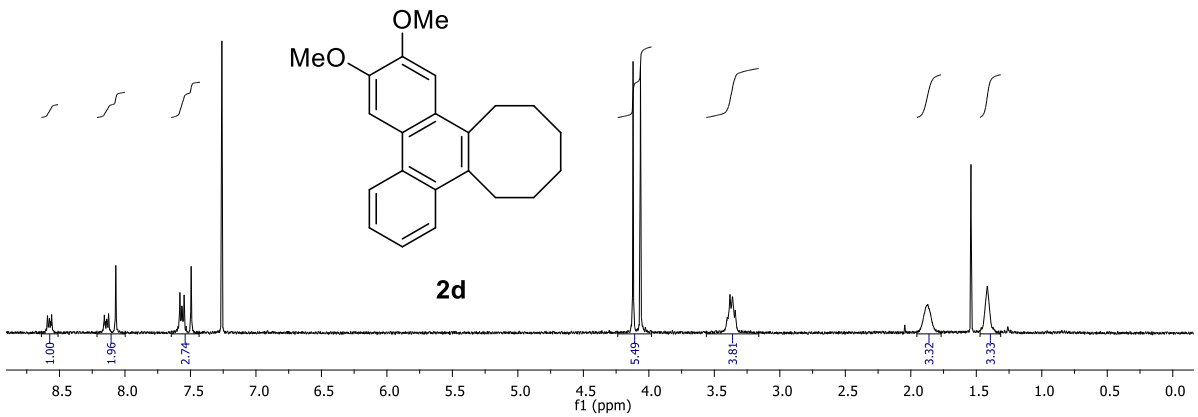

149.31
148.44
136.82
135.84
129.57
129.31
125.56
125.09
124.51
122.59
105.09
104.07
56.00
55.91
32.47
32.14
23.49
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

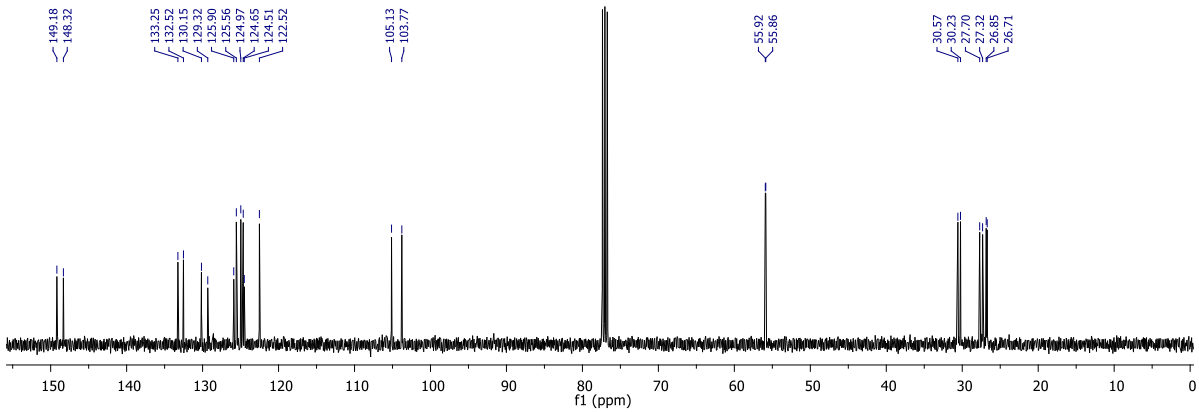

90
80
70
60
50
40
2960.24
2903.60
2847.78
1507.09
1472.84
1461.44
1449.40
1433.99
1416.43
1400.14
1251.75
1206.62
1190.98
1163.39
1038.91
1021.18
844.28
783.23
771.51
744.92
618.77
423.96
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

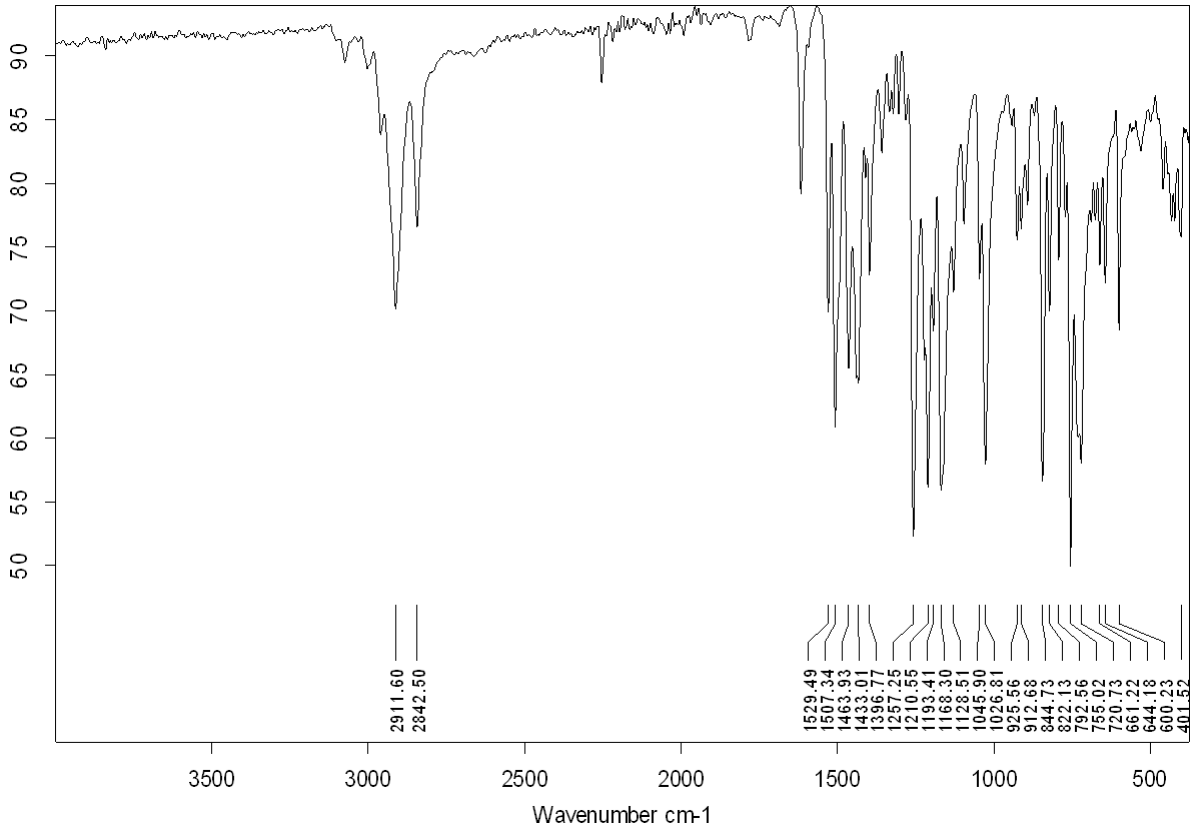

OMe
MeO
2c
1.00
1.97
2.88
5.95
3.66
5.74
f1 (ppm)

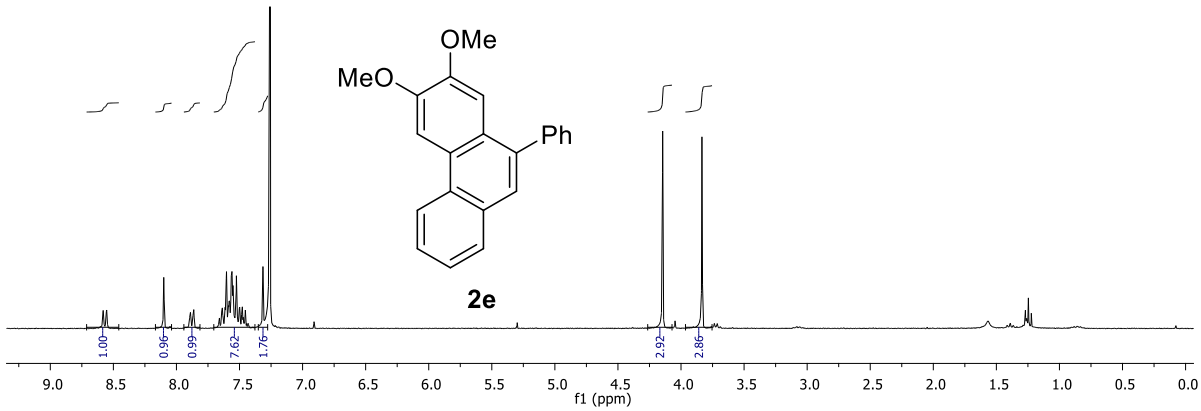

149.25
148.33
136.10
135.14
130.26
128.97
126.08
125.57
124.93
124.27
124.01
122.60
104.52
103.80
55.96
55.89
31.30
28.51
27.88
26.25
26.20
f1 (ppm)

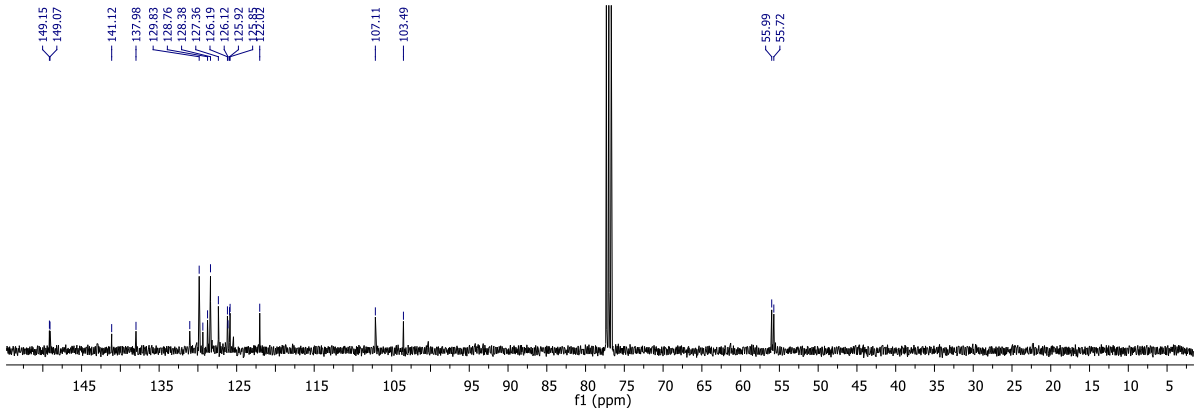

2910.79
2846.81
1616.95
1527.21
1506.54
1460.11
1432.42
1394.07
1256.28
1226.30
1205.34
1171.77
1129.58
1040.19
1023.88
845.13
820.56
756.31
737.49
719.07
601.45
445.29
Wavenumber cm-1

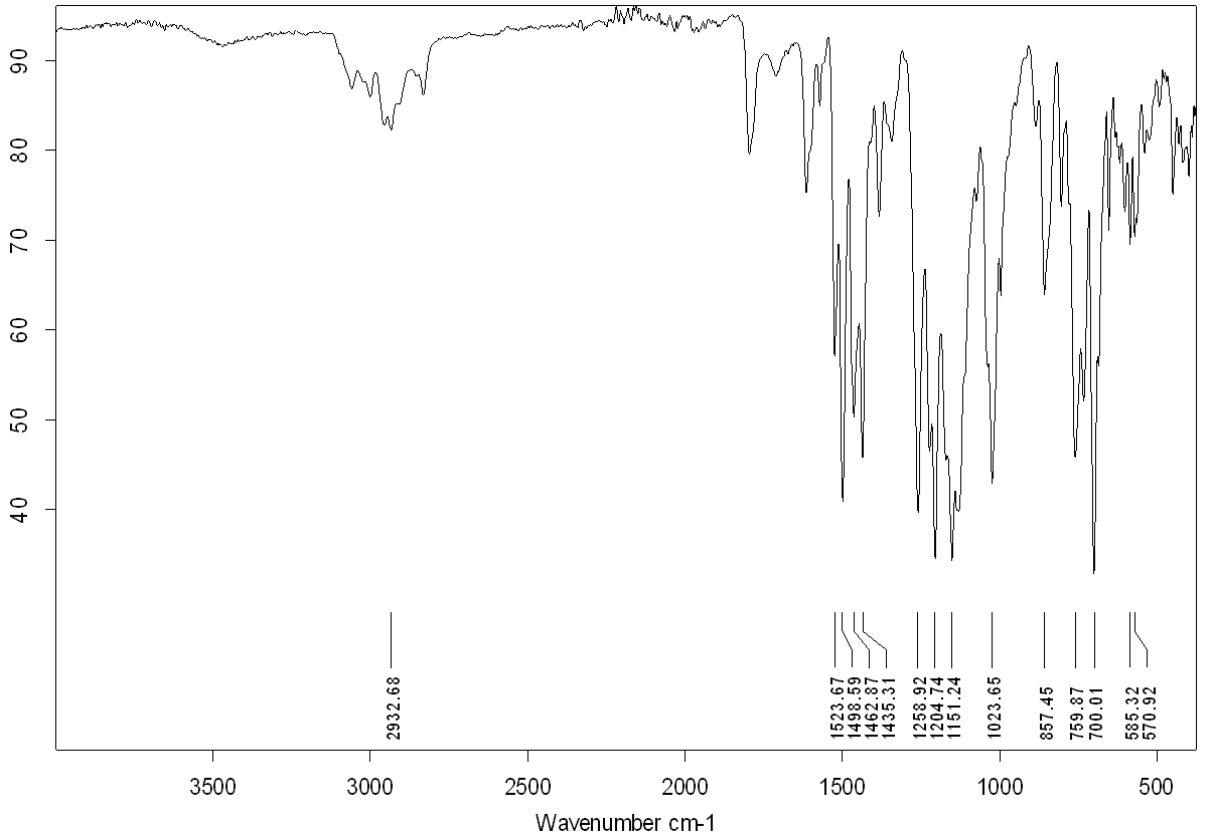

OMe
MeO
2d
1.00
1.96
2.74
5.49
3.81
3.32
3.33
f1 (ppm)

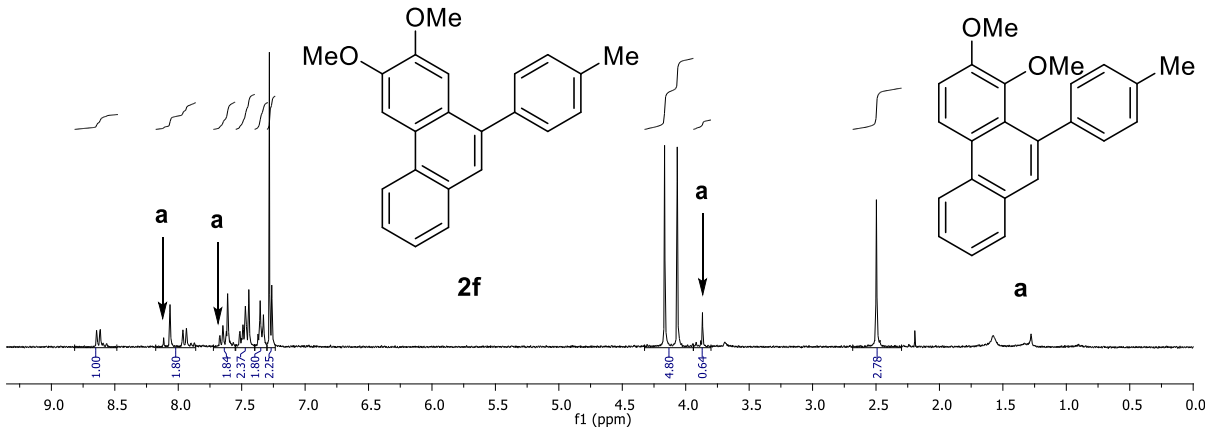

149.18
148.32
133.25
132.52
130.15
129.32
125.90
125.56
124.97
124.65
124.51
122.52
105.13
103.77
55.92
55.86
30.57
30.23
27.70
27.32
26.85
26.71
f1 (ppm)

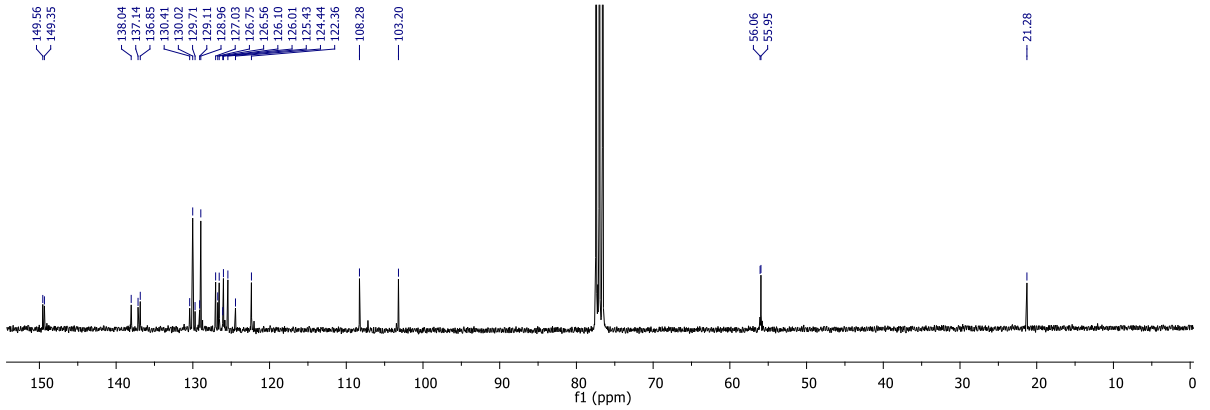

2911.60
2842.50
1529.49
1507.34
1463.93
1433.01
1396.77
1257.25
1210.55
1193.41
1168.30
1128.51
1045.90
1026.81
925.56
912.68
844.73
822.13
792.56
755.02
720.73
661.22
644.18
600.23
401.52
Wavenumber cm-1

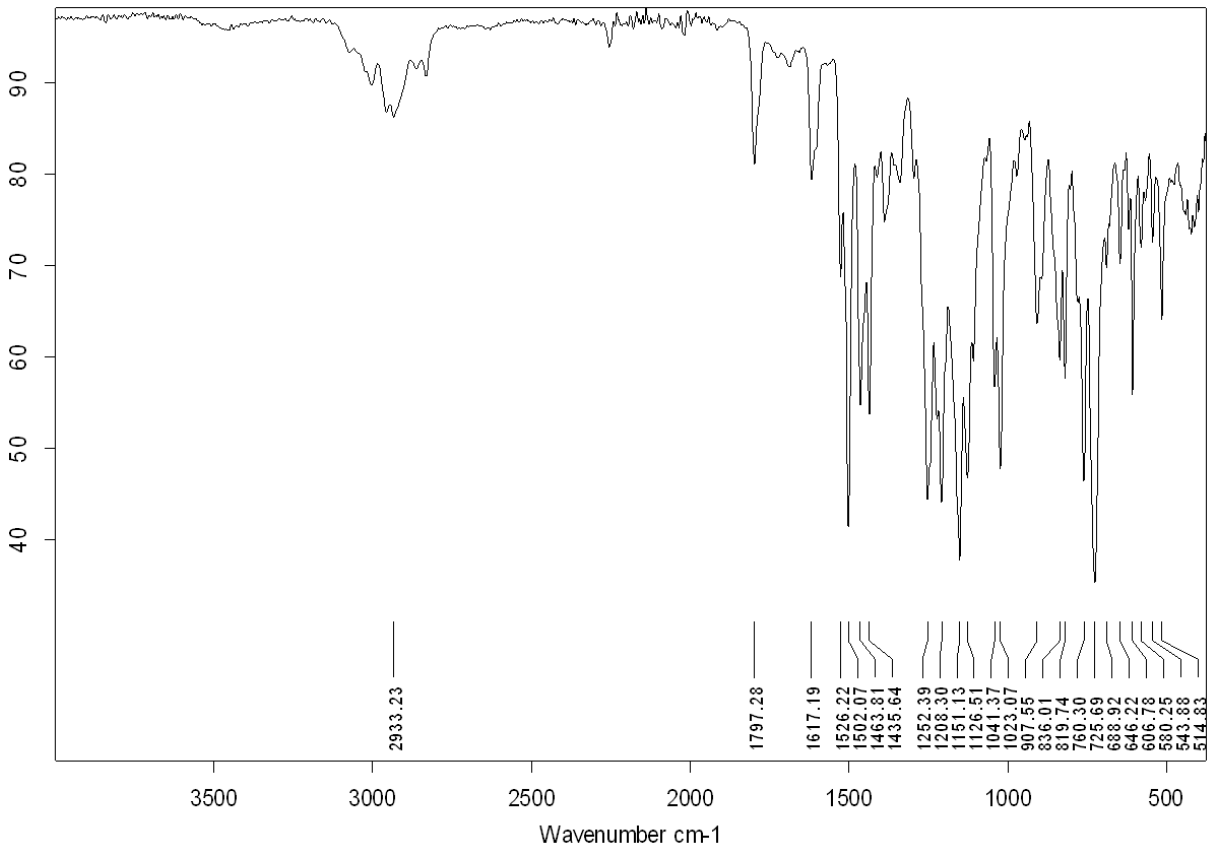

OMe
MeO
Ph
2e
1.00
0.96
0.99
7.62
1.76
2.92
2.86
f1 (ppm)

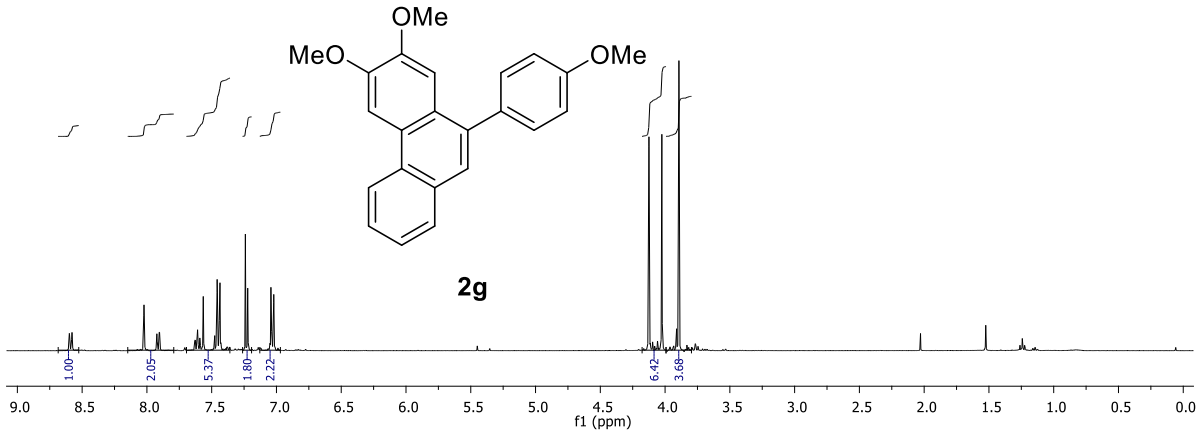

149.15
149.07
141.12
137.98
129.83
128.76
128.38
127.36
126.19
126.12
125.92
107.11
103.49
55.99
55.72
f1 (ppm)

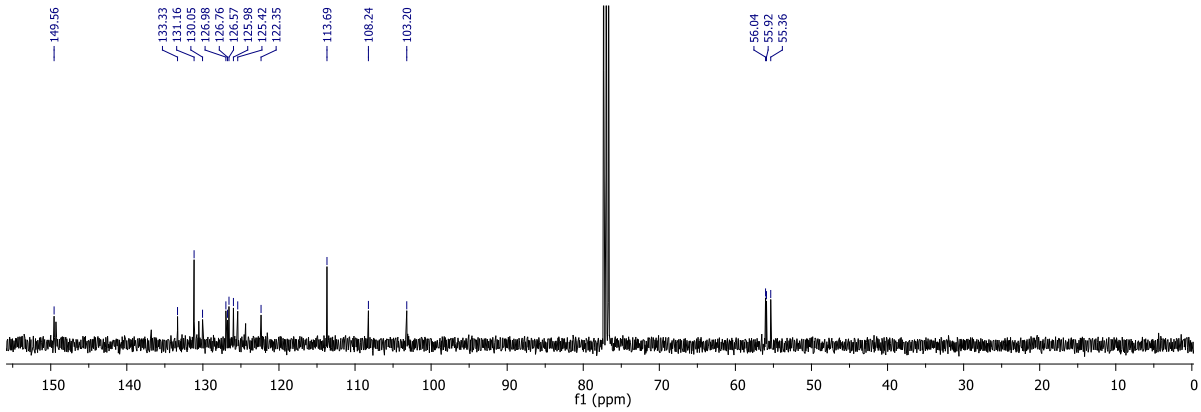

2932.68
1523.67
1498.59
1462.87
1435.31
1258.92
1204.74
1151.24
1023.65
857.45
759.87
700.01
585.32
570.92
Wavenumber cm-1

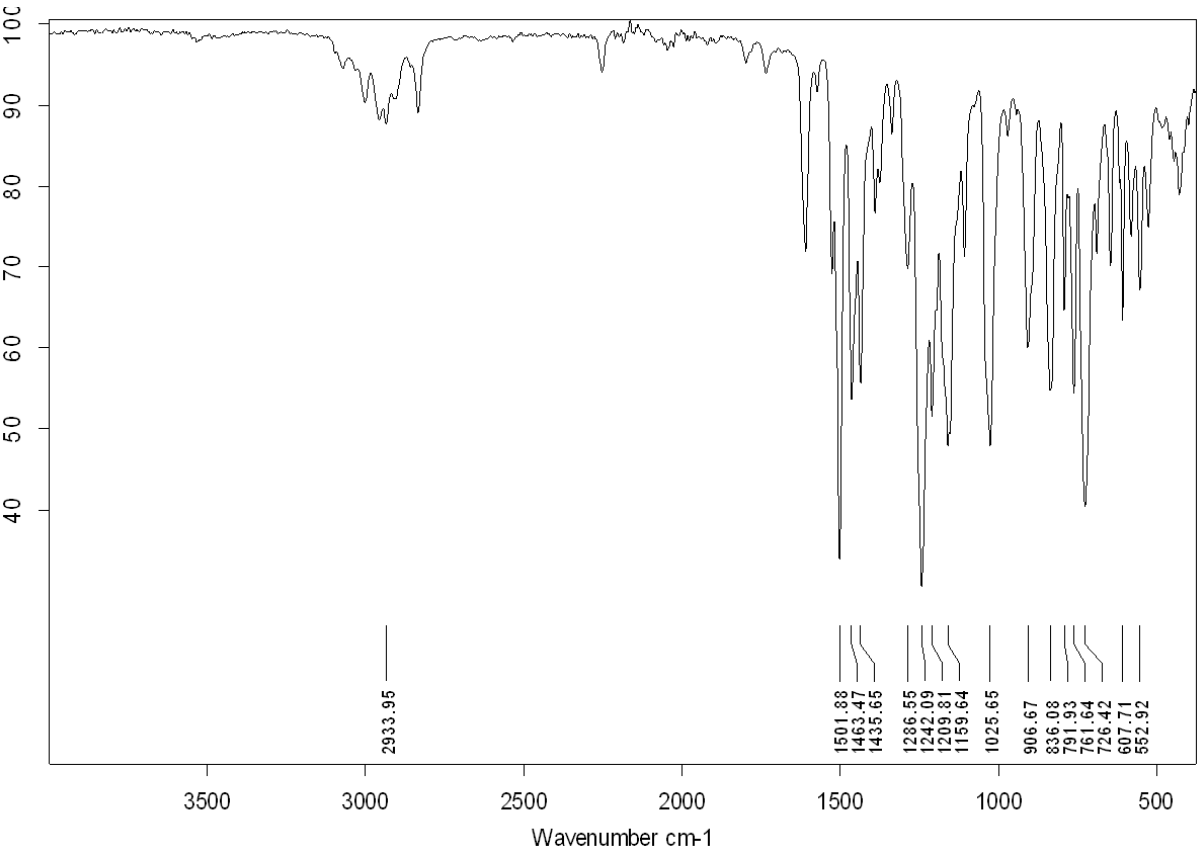

OMe
MeO
Me
a
a
2f
a
OMe
OMe
Me
a
1.00
1.80
1.84
2.37
1.80
2.25
4.80
0.64
2.78
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

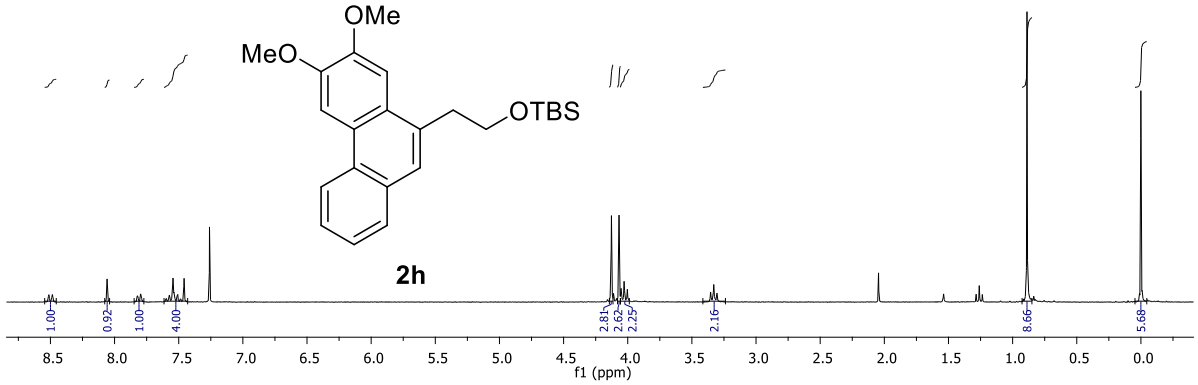

149.56
149.35
138.04
137.14
136.85
130.41
130.02
129.71
129.11
128.96
127.03
126.75
126.56
126.10
126.01
125.43
124.44
122.36
108.28
103.20
56.06
55.95
21.28
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

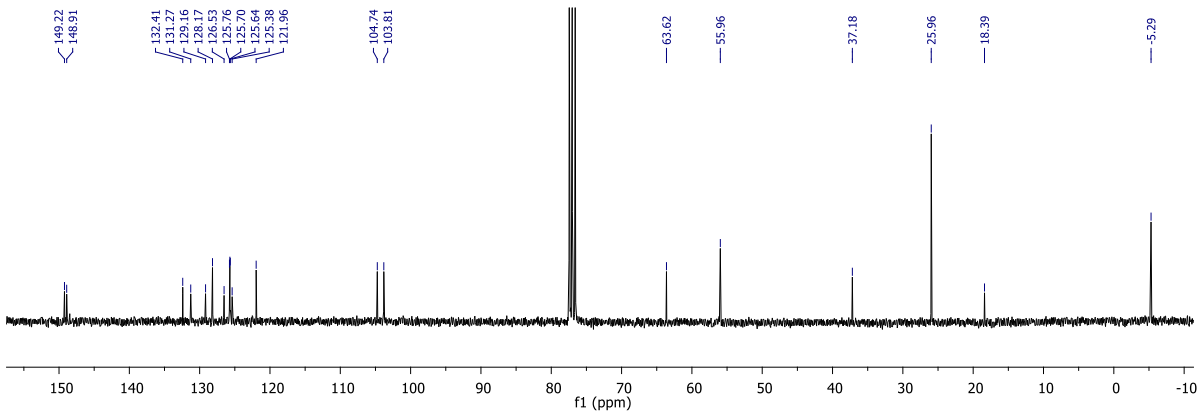

90
80
70
60
50
40
2933.23
1797.28
1617.19
1526.22
1502.07
1463.81
1435.64
1252.39
1208.30
1151.13
1126.51
1041.37
1023.07
907.55
836.01
819.74
760.30
725.69
688.92
646.22
606.78
580.25
543.88
514.83
3500 3000 2500 2000 1500 1000 500
Wavenumber cm-1

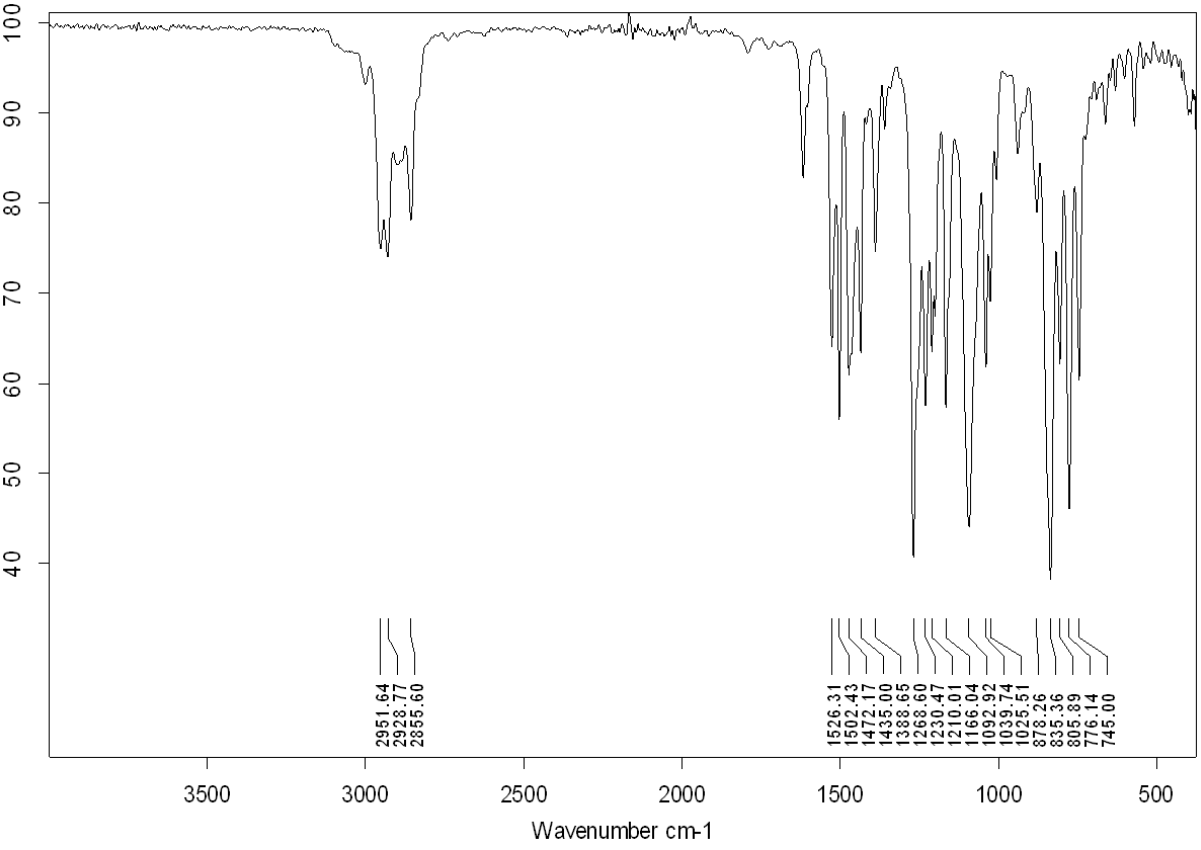

OMe
MeO
OMe
2g
1.00
2.05
5.37
1.80
2.22
6.42
3.68
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

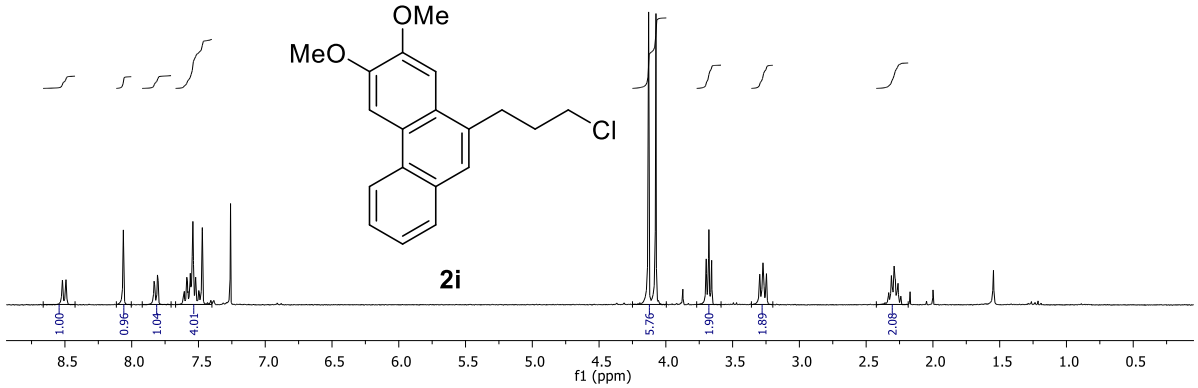

149.56
133.33
131.16
130.05
126.98
126.76
126.57
125.98
125.42
122.35
113.69
108.24
103.20
56.04
55.92
55.36
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

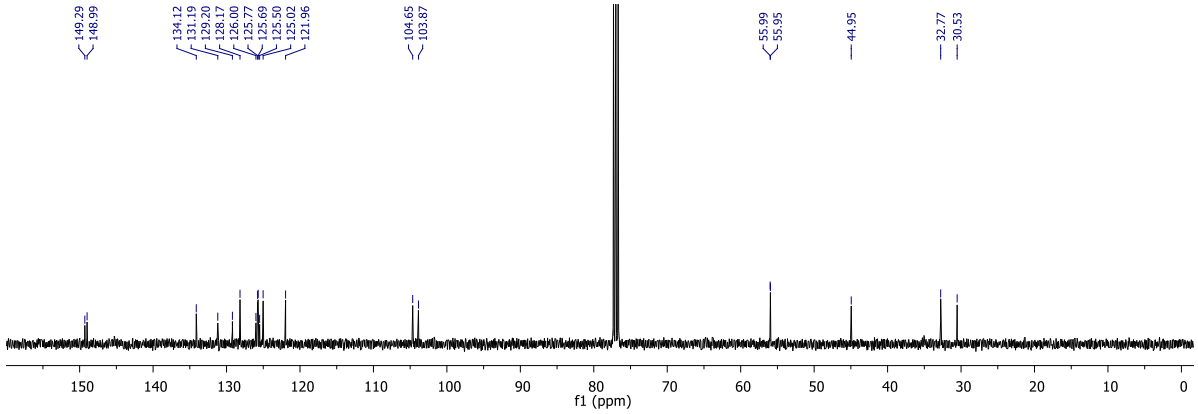

100
90
80
70
60
50
40
2933.95
1501.88
1463.47
1435.65
1286.55
1242.09
1209.81
1159.64
1025.65
906.67
836.08
791.93
761.64
726.42
607.71
552.92
3500 3000 2500 2000 1500 1000 500
Wavenumber cm-1

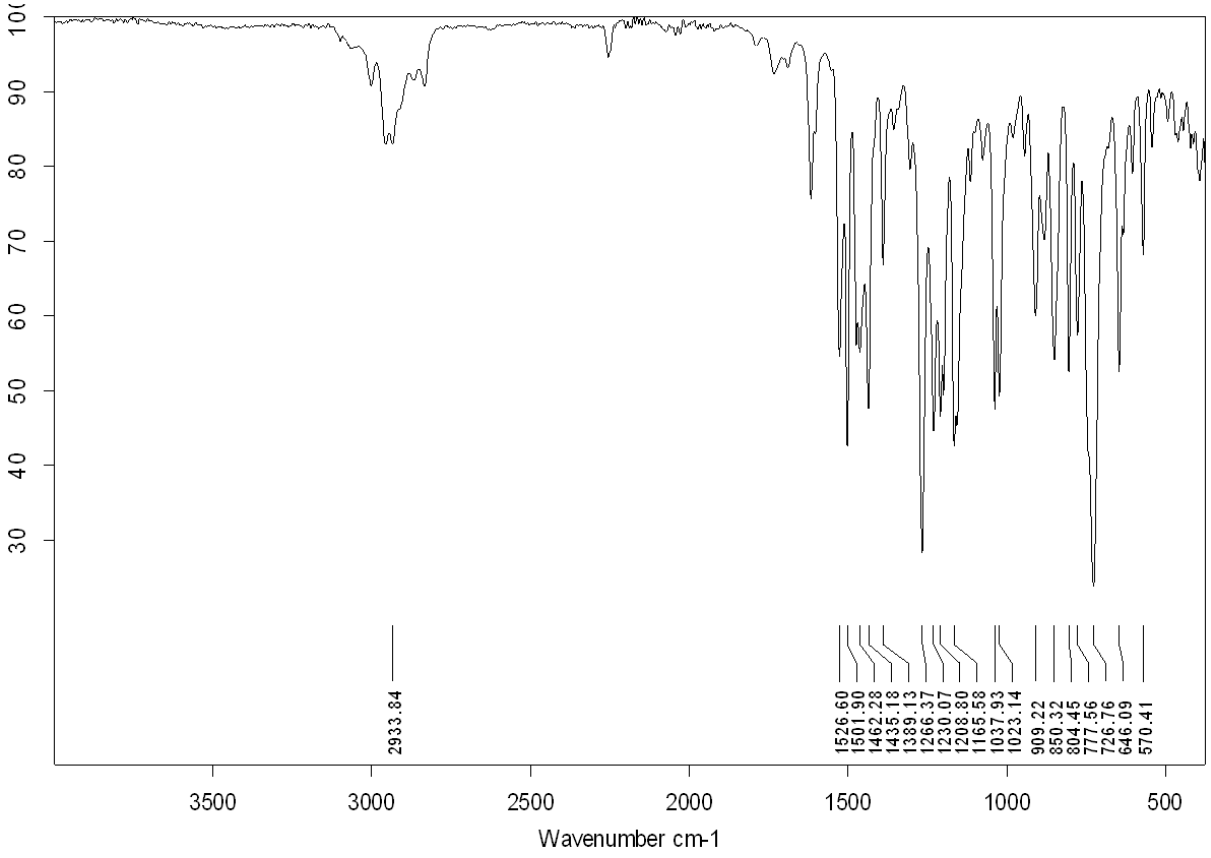

OMe
MeO
OTBS
2h
1.00
0.92
1.00
4.00
2.81
2.62
2.25
2.16
8.66
5.68
f1 (ppm)

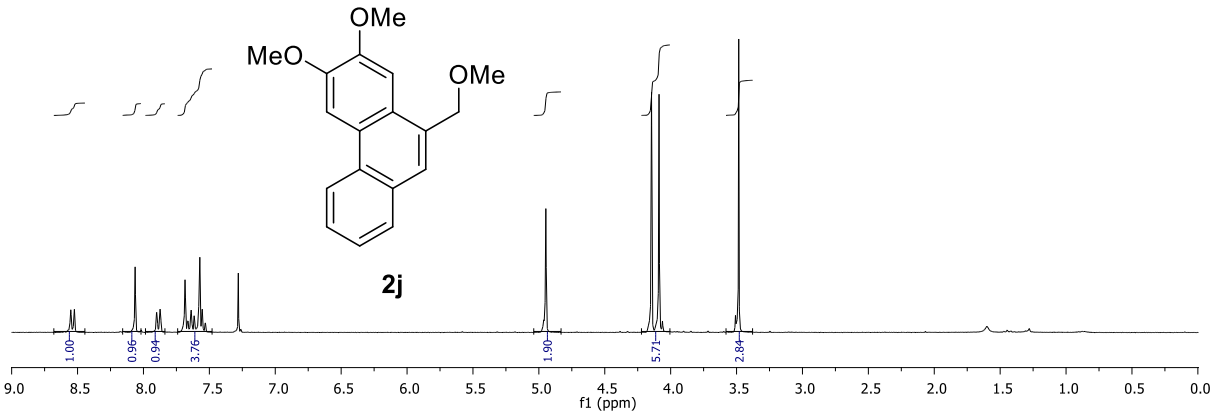

149.22
148.91
132.41
131.27
129.16
128.17
126.53
125.76
125.70
125.64
125.38
121.96
104.74
103.81
63.62
55.96
37.18
25.96
18.39
-5.29
f1 (ppm)

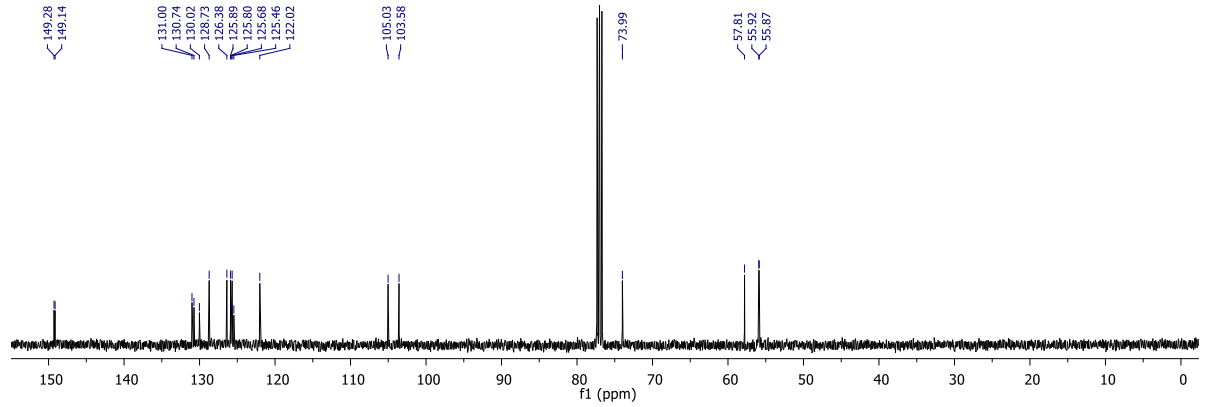

100
90
80
70
60
50
40
2951.64
2928.77
2855.60
1526.31
1502.43
1472.17
1435.00
1388.65
1268.60
1230.47
1210.01
1166.04
1092.92
1039.74
1025.51
878.26
835.36
805.89
776.14
745.00
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

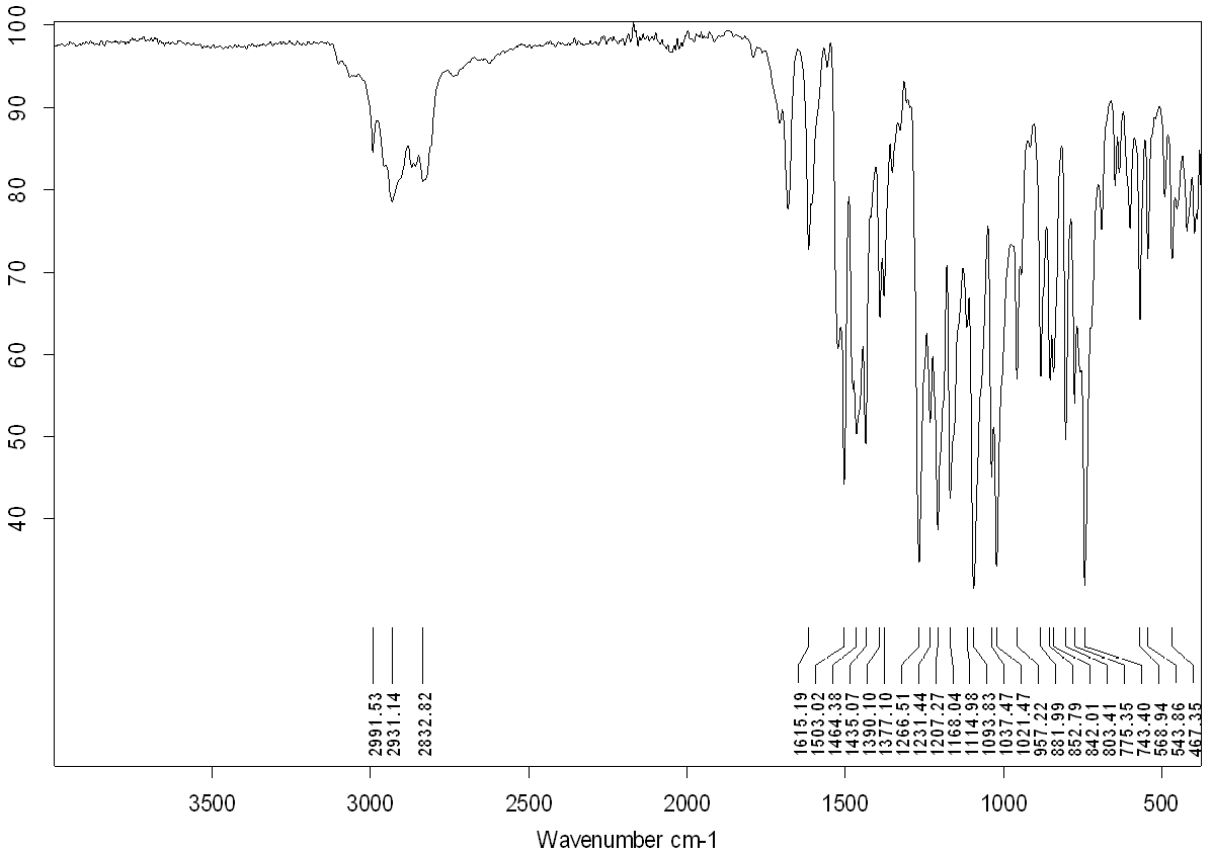

OMe
MeO
Cl
2i
1.00
0.96
1.04
4.01
5.76
1.90
1.89
2.08
f1 (ppm)

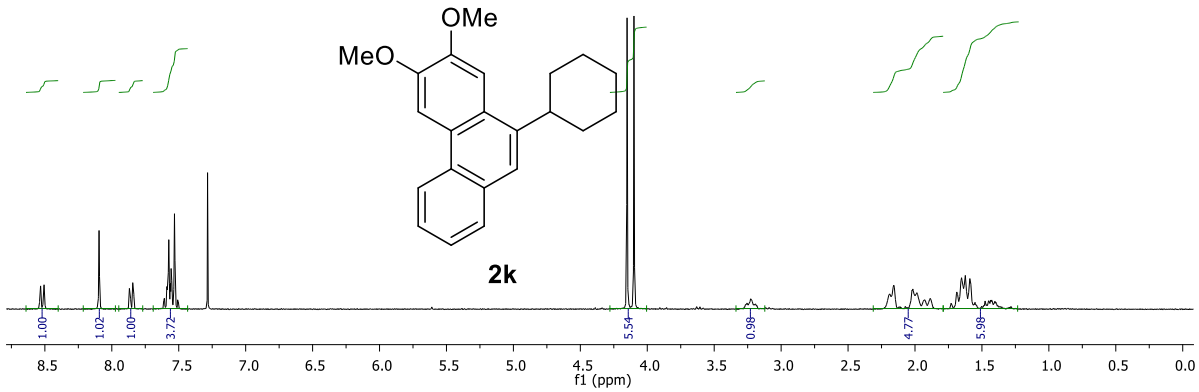

149.29
148.99
134.12
131.19
129.20
128.17
126.00
125.77
125.69
125.50
125.02
121.96
104.65
103.87
55.99
55.95
44.95
32.77
30.53
f1 (ppm)

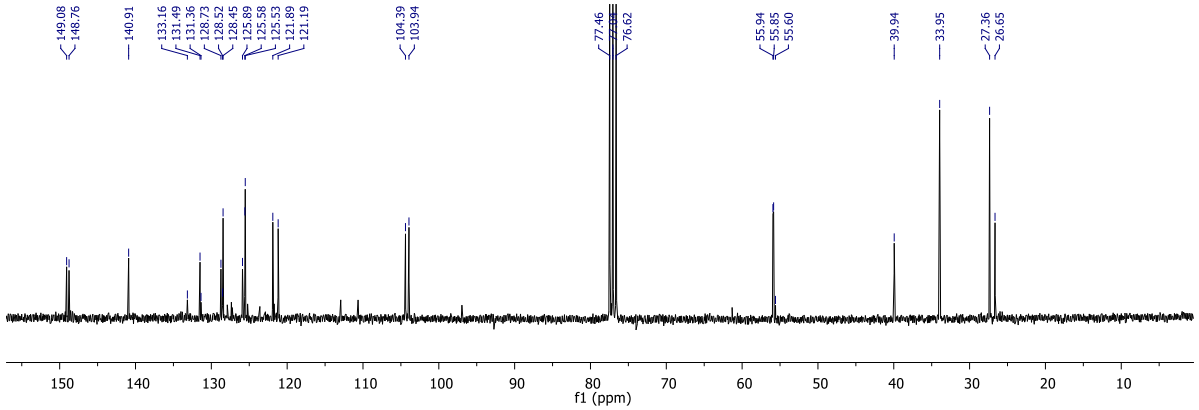

2933.84
1526.60
1501.90
1462.28
1435.18
1389.13
1266.37
1230.07
1208.80
1165.58
1037.93
1023.14
909.22
850.32
804.45
777.56
726.76
646.09
570.41
Wavenumber cm-1

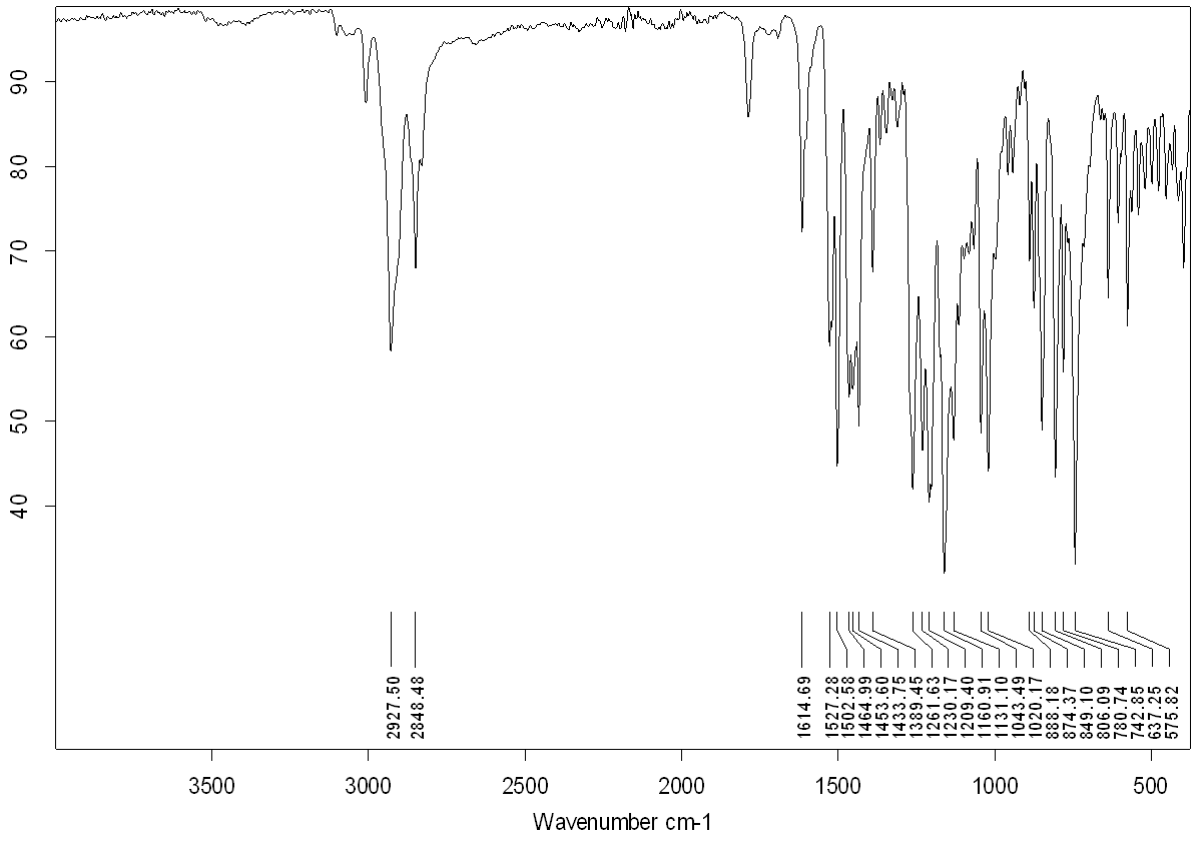

OMe
MeO
OMe
2j
1.00
0.96
0.94
3.76
1.90
5.71
2.84
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

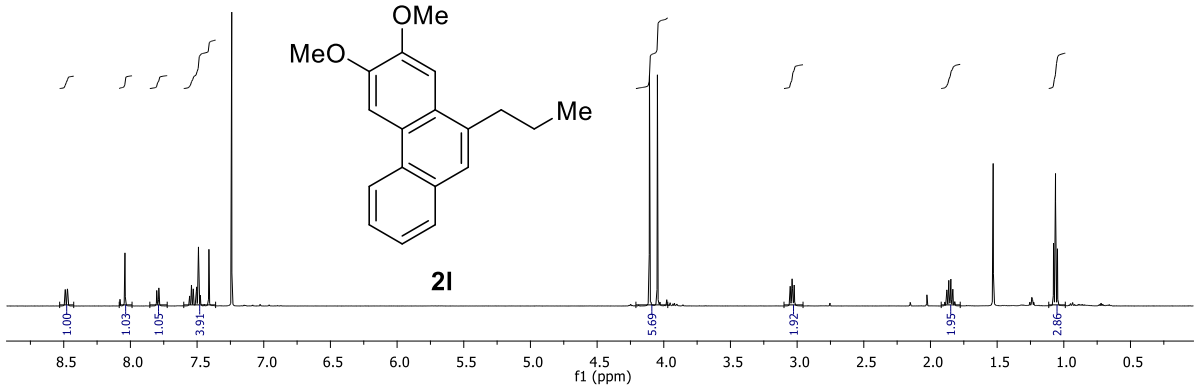

149.28
149.14
131.00
130.74
130.02
128.73
126.38
125.89
125.80
125.68
125.46
122.02
105.03
103.58
73.99
57.81
55.92
55.87
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

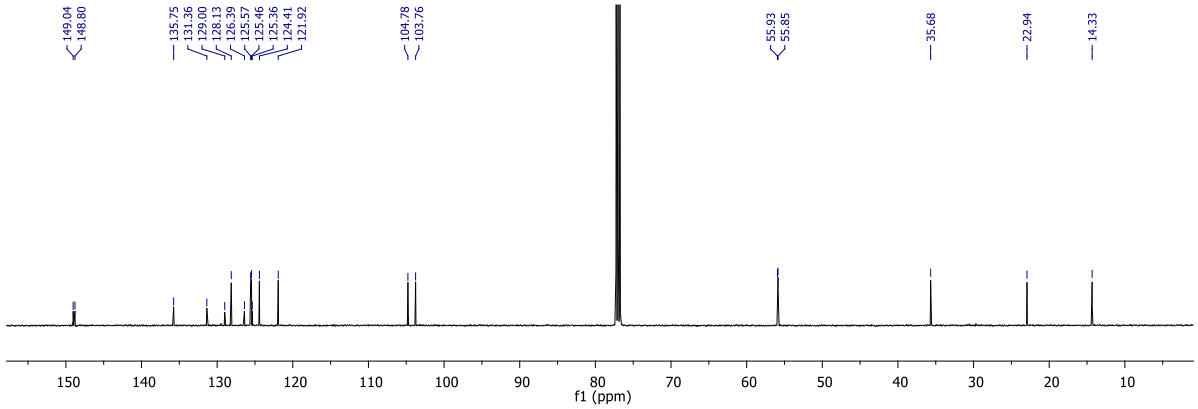

100
90
80
70
60
50
40
2991.53
2931.14
2832.82
1615.19
1503.02
1464.38
1435.07
1390.10
1377.10
1266.51
1231.44
1207.27
1168.04
1114.98
1093.83
1037.47
1021.47
957.22
881.99
852.79
842.01
803.41
775.35
743.40
568.94
543.86
467.35
3500 3000 2500 2000 1500 1000 500
Wavenumber cm-1

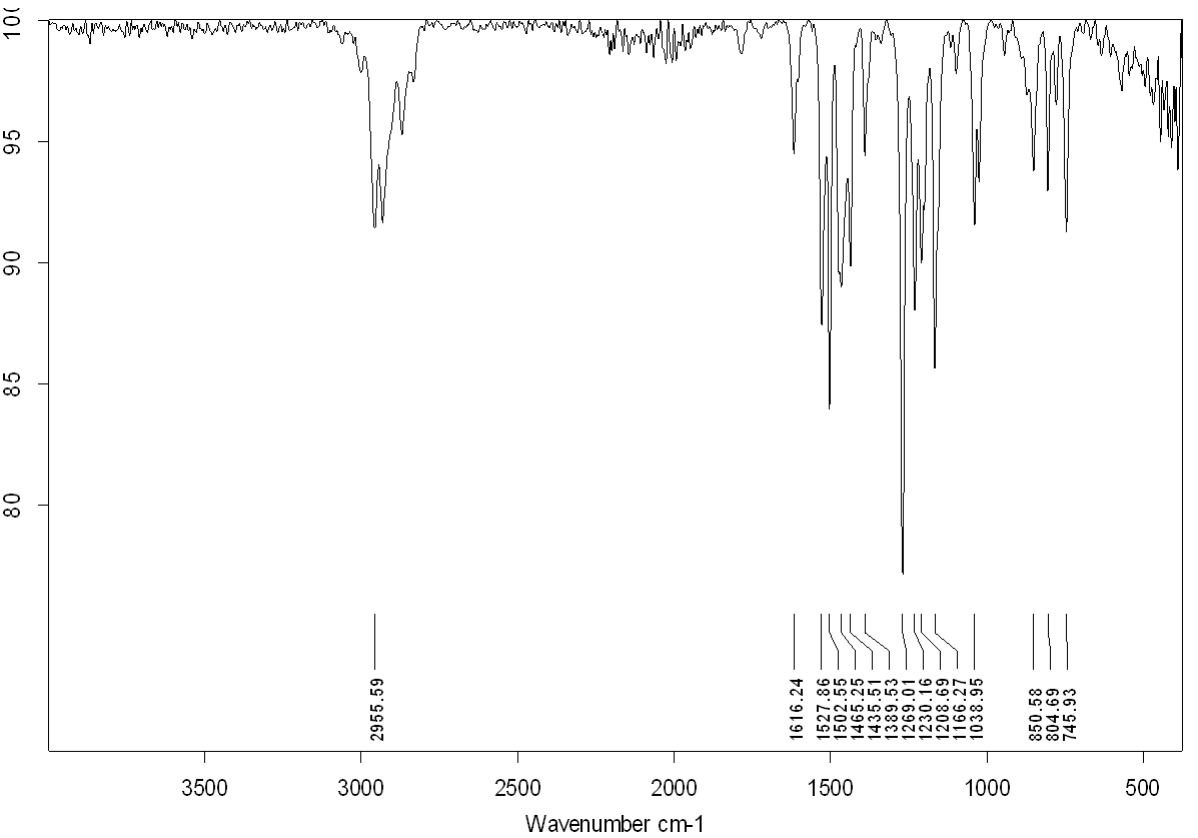

OMe
MeO
2k
1.00
1.02
1.00
3.72
5.54
0.98
4.77
5.98
f1 (ppm)

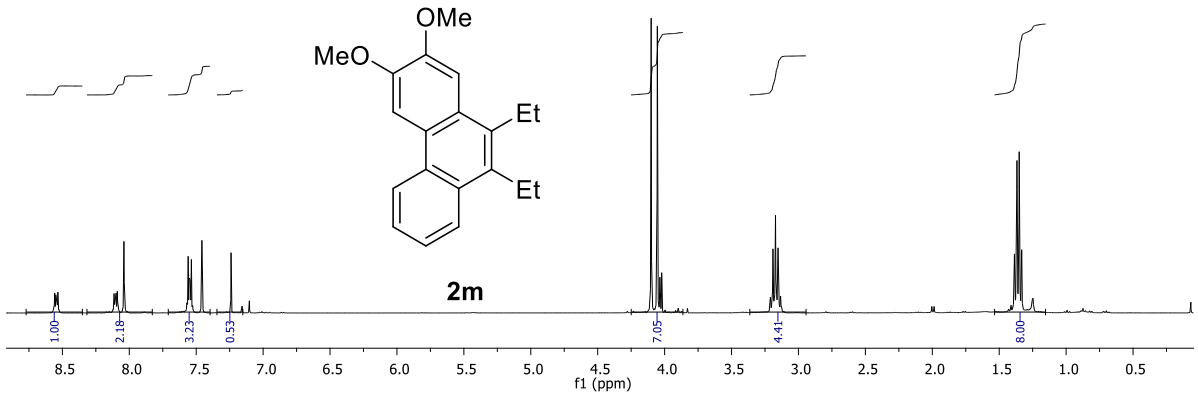

149.08
148.76
140.91
133.16
131.49
131.36
128.73
128.52
128.45
125.89
125.58
125.53
121.89
121.19
104.39
103.94
77.46
76.62
55.94
55.85
55.60
39.94
33.95
27.36
26.65
f1 (ppm)

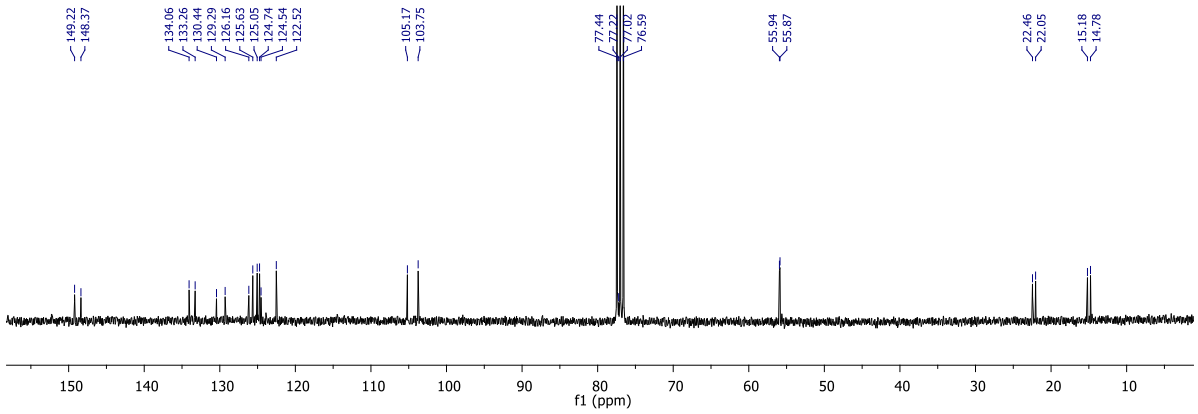

2927.50
2848.48
1614.69
1527.28
1502.58
1464.99
1453.60
1433.75
1389.45
1261.63
1230.17
1209.40
1160.91
1131.10
1043.49
1020.17
888.18
874.37
849.10
806.09
780.74
742.85
637.25
575.82
Wavenumber cm-1

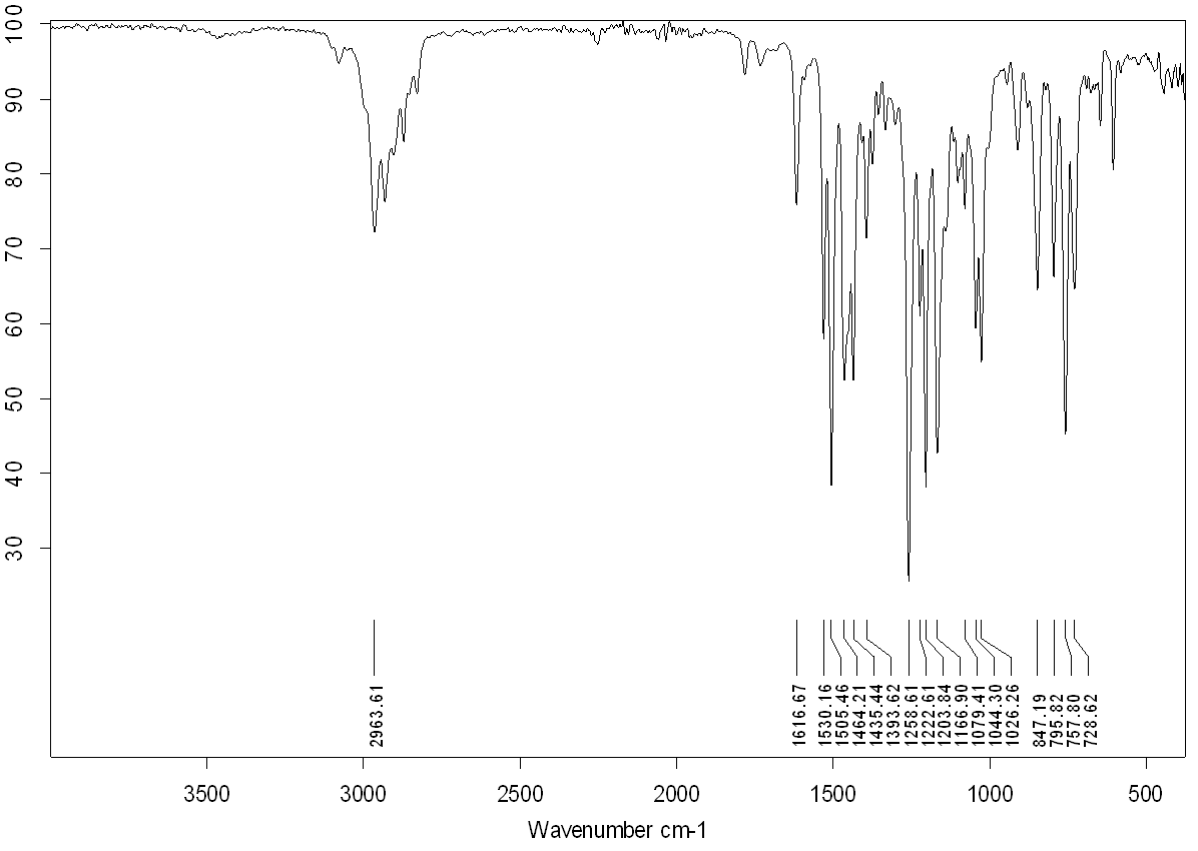

OMe
MeO
Me
2l
1.00
1.03
1.05
3.91
5.69
1.92
1.95
2.86
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

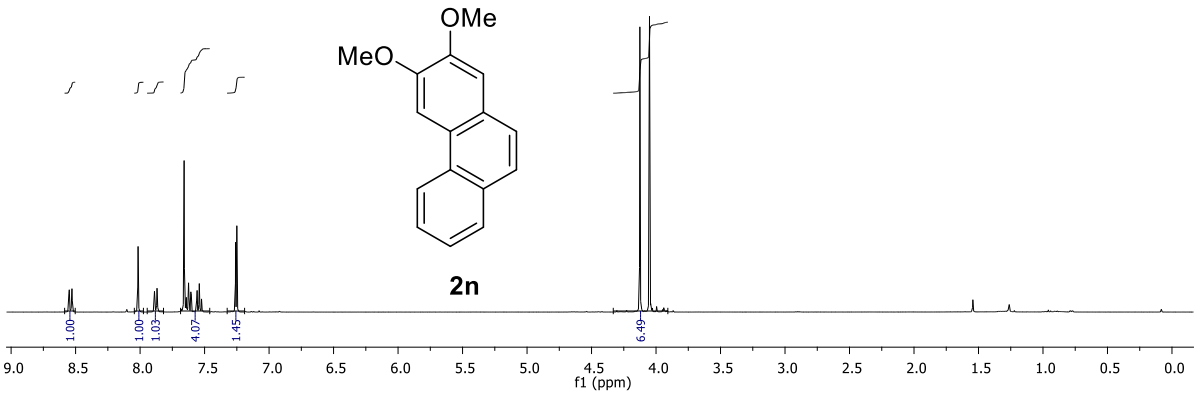

149.04
148.80
135.75
131.36
129.00
128.13
126.39
125.57
125.46
125.36
124.41
121.92
104.78
103.76
55.93
55.85
35.68
22.94
14.33
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

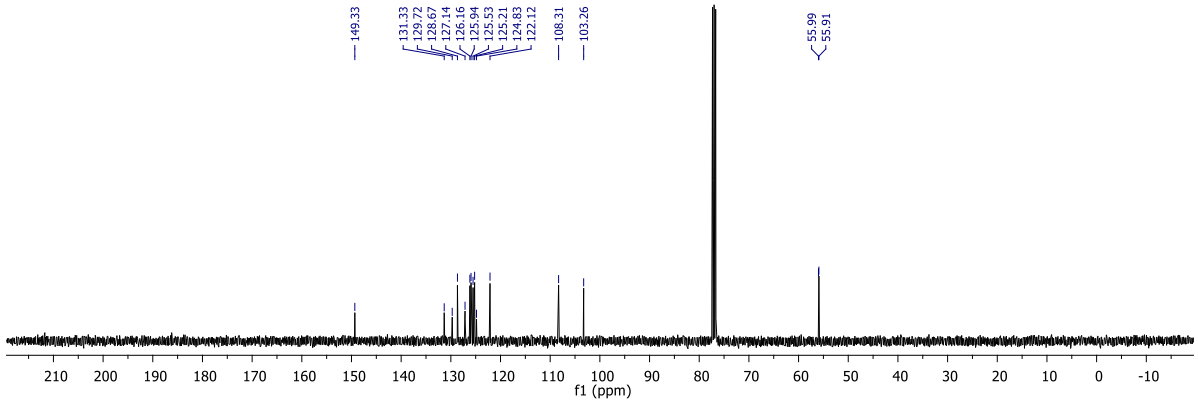

100
95
90
85
80
2955.59
1616.24
1527.86
1502.55
1465.25
1435.51
1389.53
1269.01
1230.16
1208.69
1166.27
1038.95
850.58
804.69
745.93
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

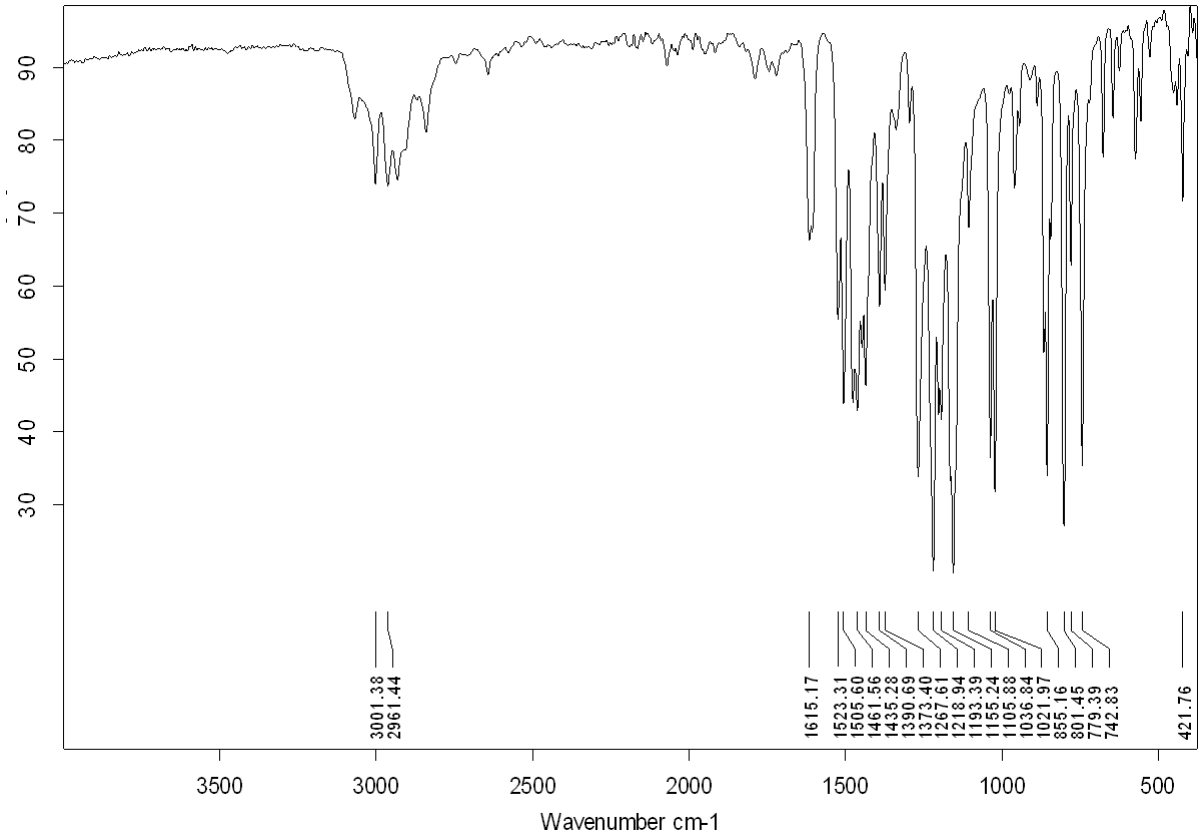

OMe
MeO
Et
Et
2m
1.00
2.18
3.23
0.53
7.05
4.41
8.00
f1 (ppm)

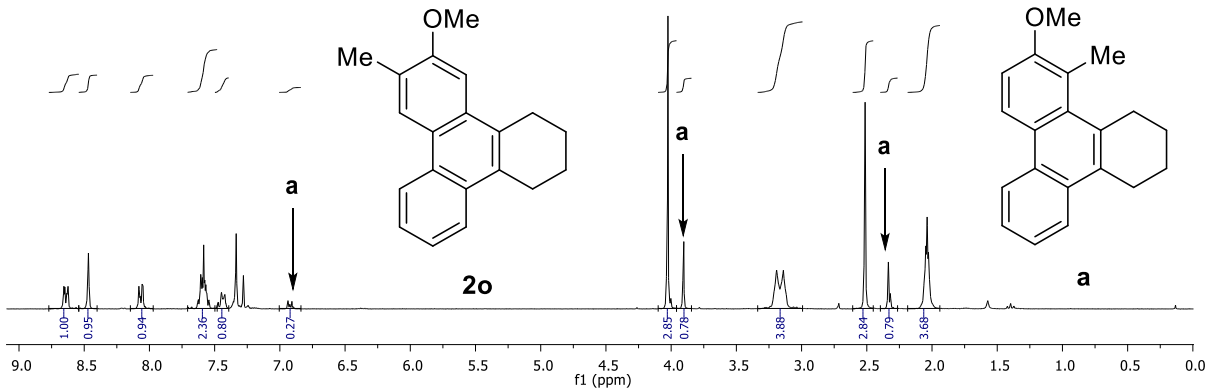

149.22
148.37
134.06
133.26
130.44
129.29
126.16
125.63
125.05
124.74
124.54
122.52
105.17
103.75
77.44
77.22
77.02
76.59
55.94
55.87
22.46
22.05
15.18
14.78
f1 (ppm)

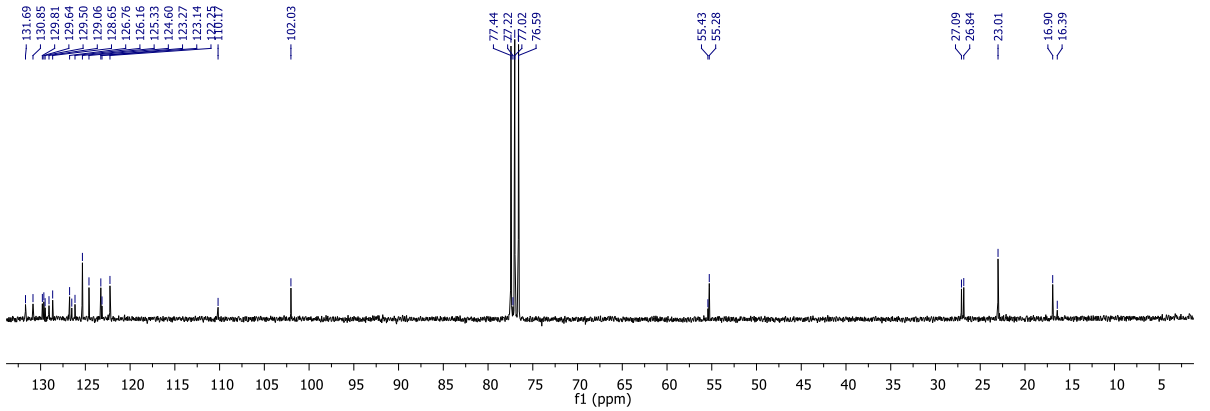

2963.61
1616.67
1530.16
1505.46
1464.21
1435.44
1393.62
1258.61
1222.61
1203.84
1166.90
1079.41
1044.30
1026.26
847.19
795.82
757.80
728.62
Wavenumber cm-1

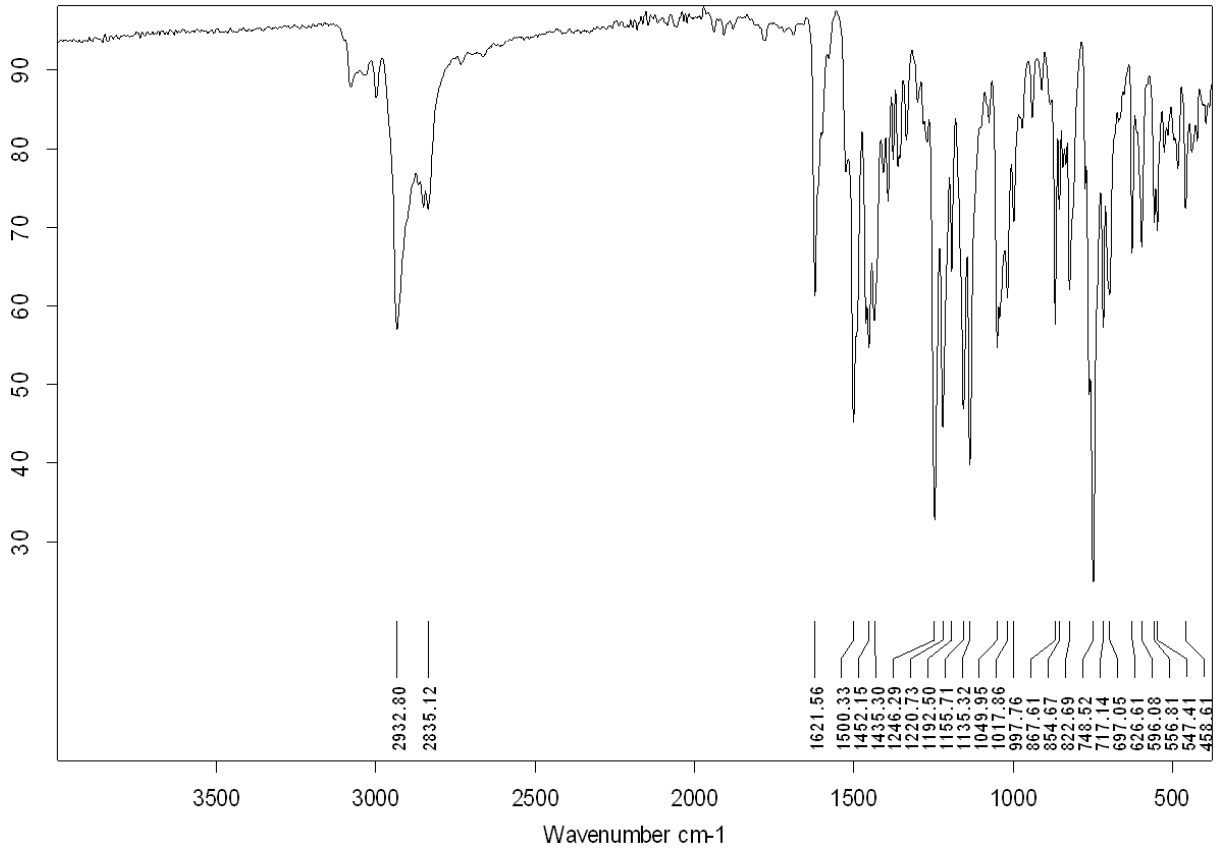

OMe
MeO
2n
1.00
1.00
1.03
4.07
1.45
6.49
f1 (ppm)

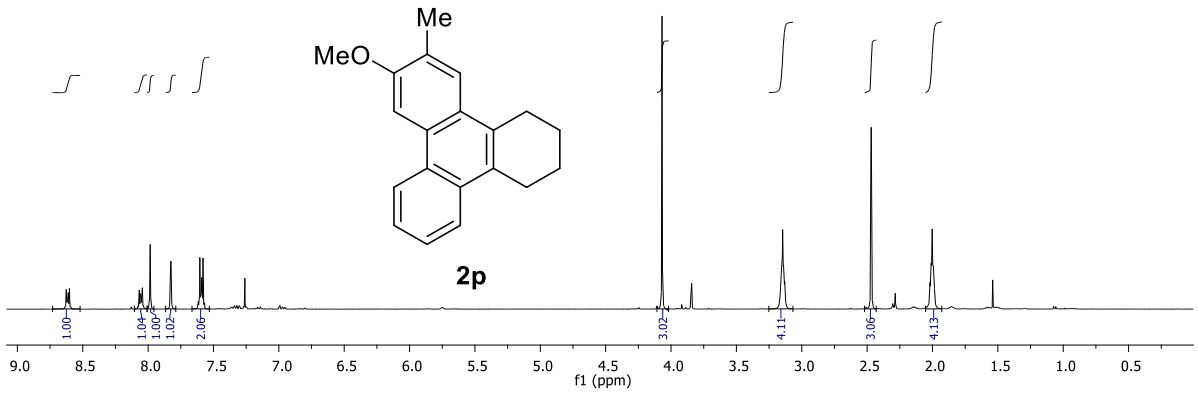

149.33
131.33
129.72
128.67
127.14
126.16
125.94
125.53
125.21
124.83
122.12
108.31
103.26
55.99
55.91
f1 (ppm)

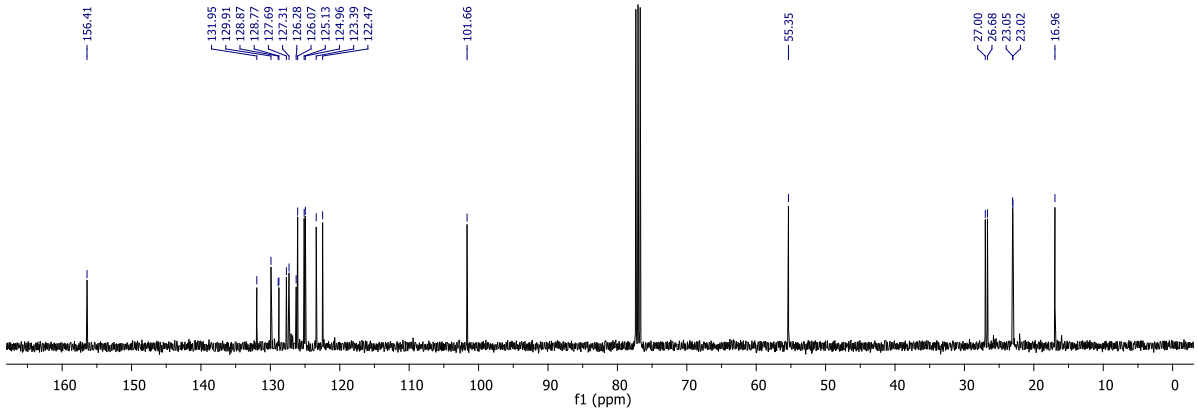

3001.38
2961.44
1615.17
1523.31
1505.60
1461.56
1435.28
1390.69
1373.40
1267.61
1218.94
1193.39
1155.24
1105.88
1036.84
1021.97
855.16
801.45
779.39
742.83
421.76
Wavenumber cm-1

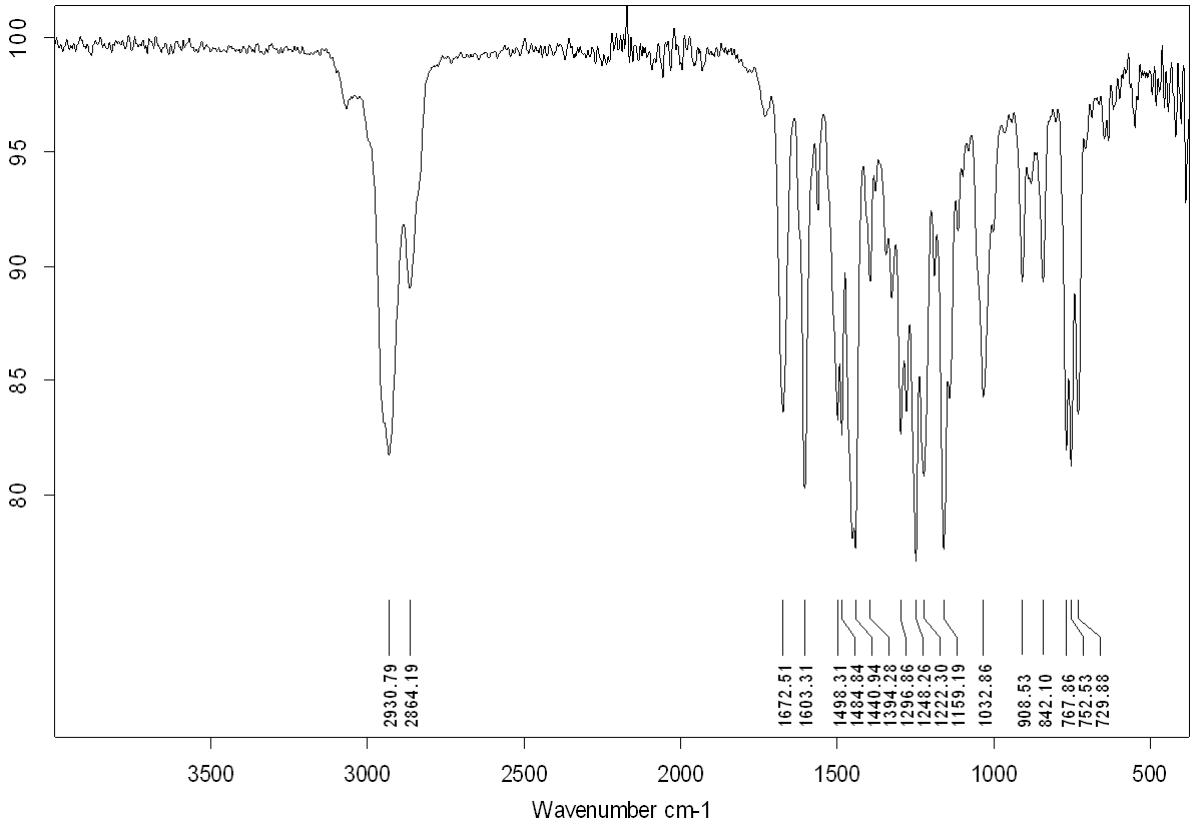

OMe
Me
a
2o
a
a
OMe
Me
a
1.00
0.95
0.94
2.36
0.80
0.27
2.85
0.78
3.88
2.84
0.79
3.68
f1 (ppm)

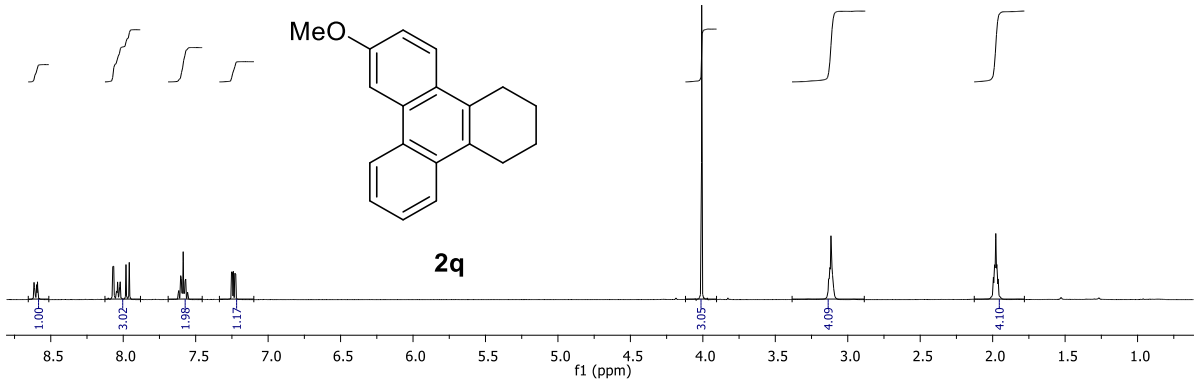

131.69
130.85
129.81
129.64
129.50
129.06
128.65
126.76
126.16
125.33
124.60
123.27
123.14
102.03
77.44
77.22
77.02
76.59
55.43
55.28
27.09
26.84
23.01
16.90
16.39
f1 (ppm)

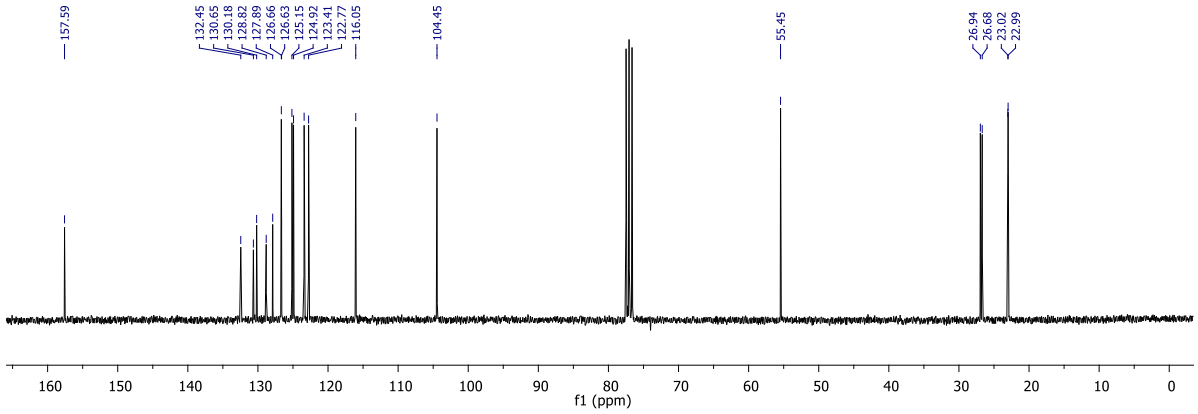

2932.80
2835.12
1621.56
1500.33
1452.15
1435.30
1246.29
1220.73
1192.50
1155.71
1135.32
1049.95
1017.86
997.76
867.61
854.67
822.69
748.52
717.14
697.05
626.61
596.08
556.81
547.41
458.61
Wavenumber cm-1

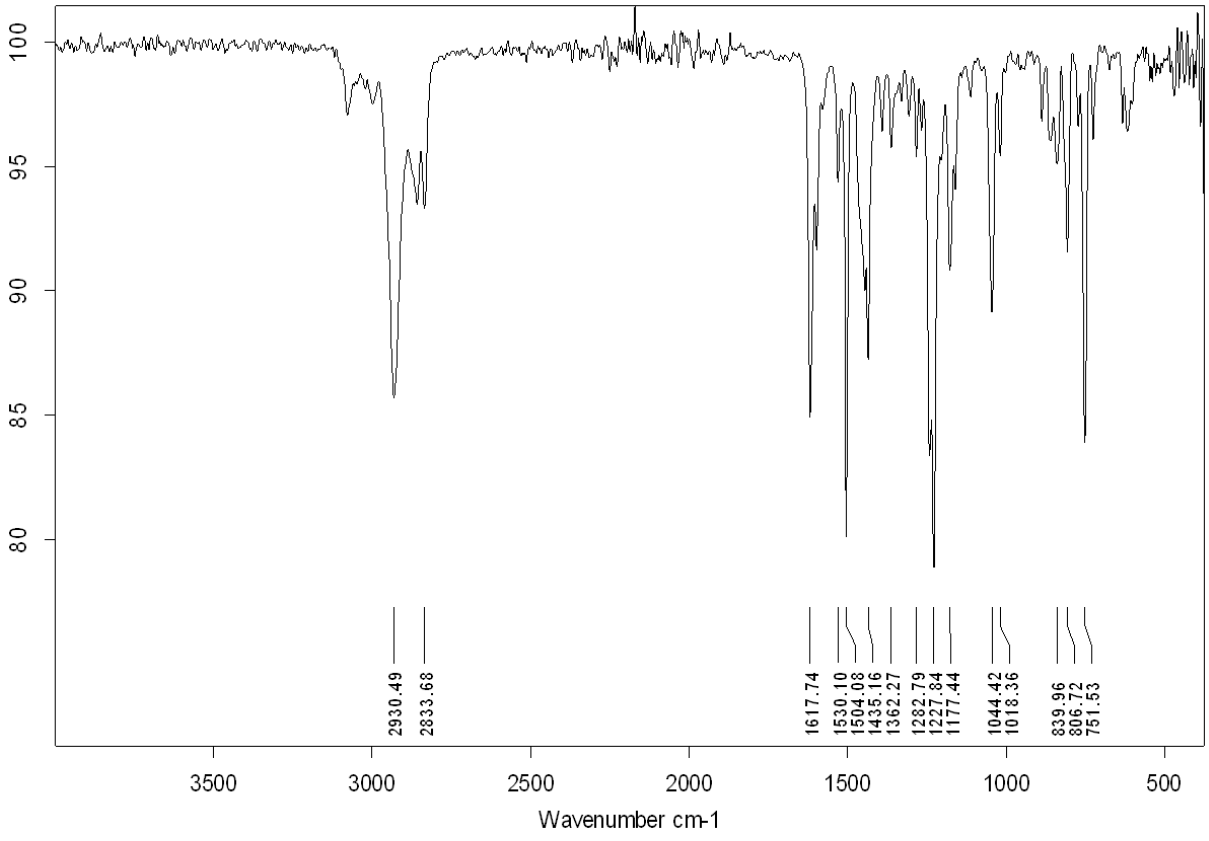

Me
MeO
2p
1.00
1.04
1.00
1.02
2.06
3.02
4.11
3.06
4.13
f1 (ppm)

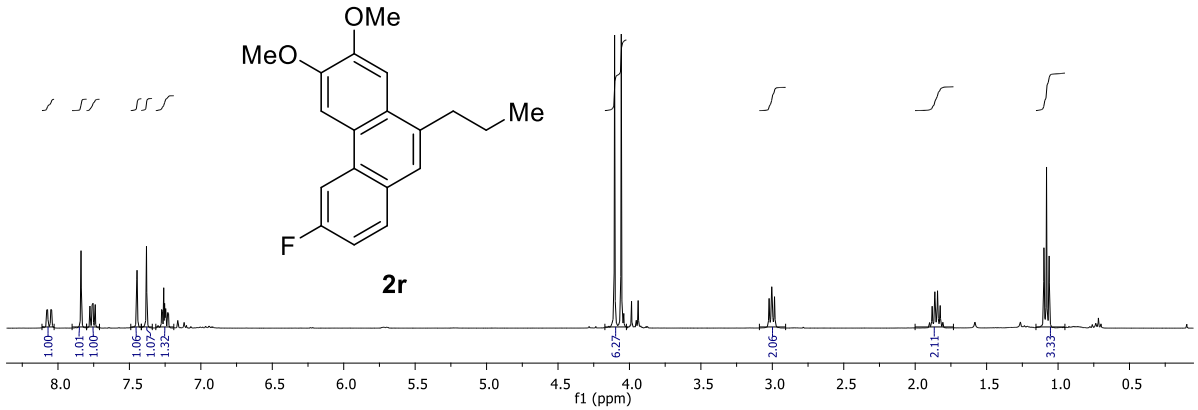

156.41
131.95
129.91
128.87
128.77
127.69
127.31
126.28
126.07
125.13
124.96
123.39
122.47
101.66
55.35
27.00
26.68
23.05
23.02
16.96
f1 (ppm)

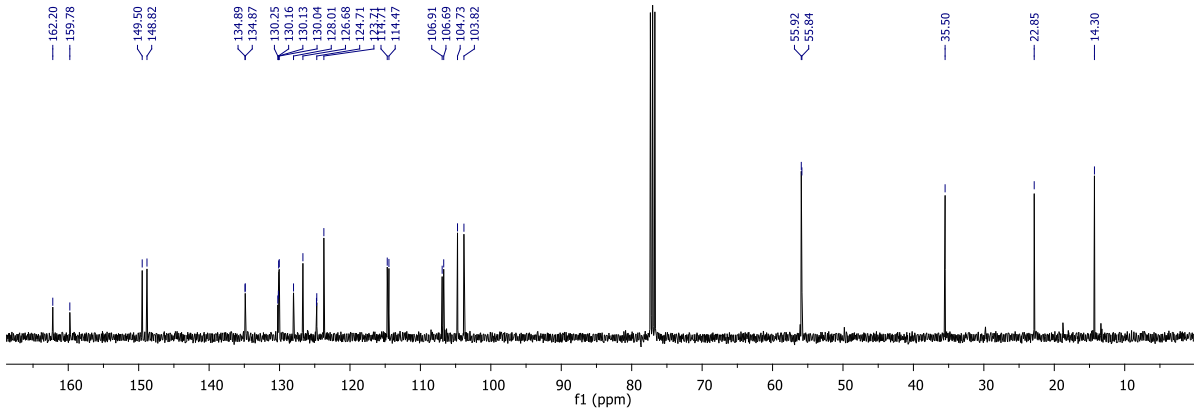

Transmittance
2930.79
2864.19
1672.51
1603.31
1498.31
1484.84
1440.94
1394.28
1296.66
1248.26
1222.30
1159.19
1032.86
908.53
842.10
767.86
752.53
729.88
Wavenumber cm-1

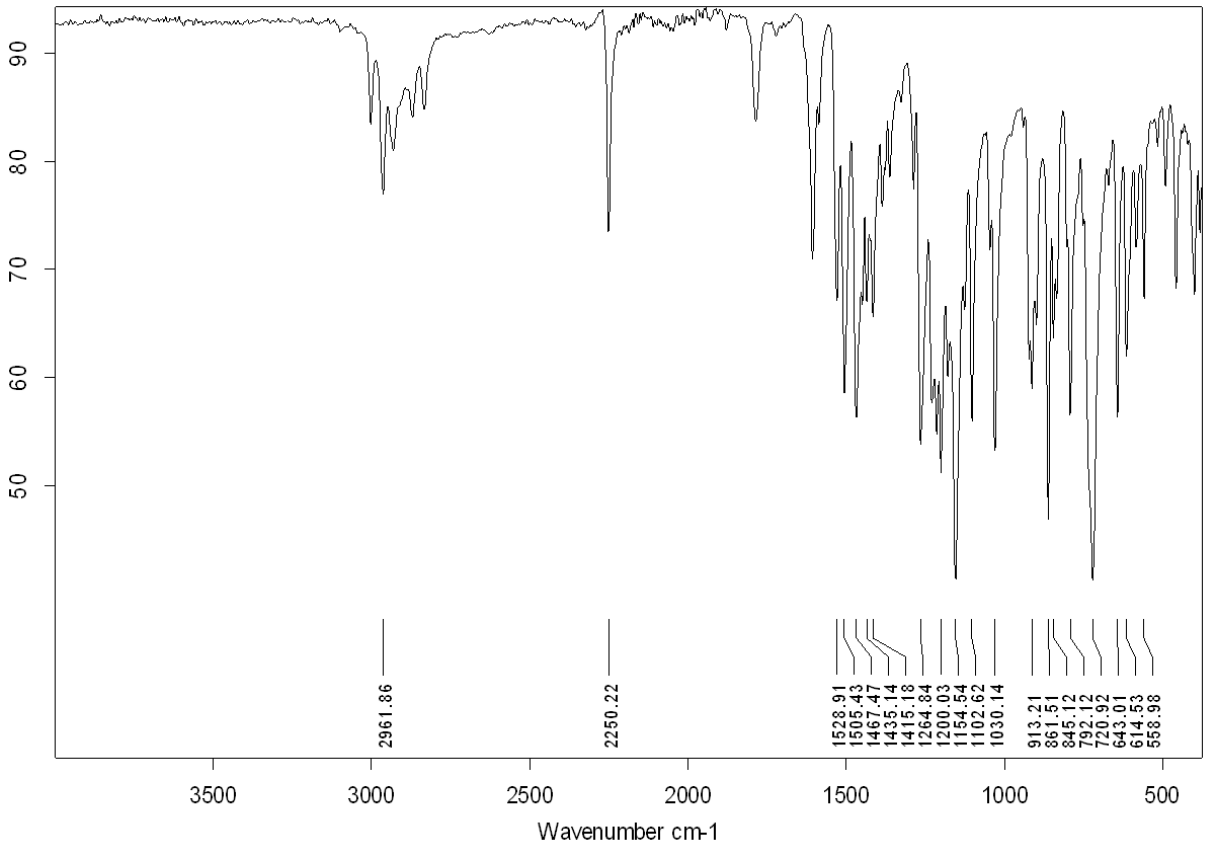

MeO
2q
1.00
3.02
1.98
1.17
3.05
4.09
4.10
f1 (ppm)

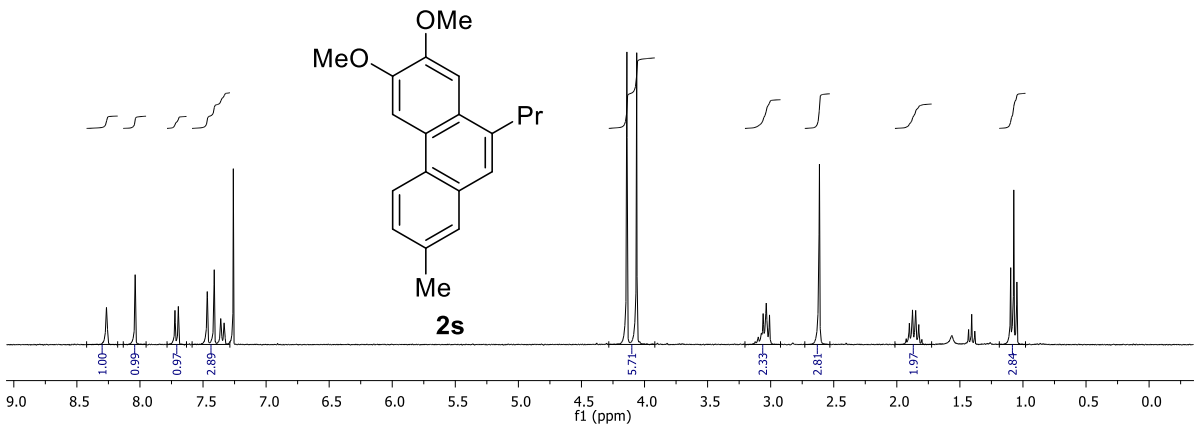

157.59
132.45
130.65
130.18
128.82
127.89
126.66
126.63
125.15
124.92
123.41
122.77
116.05
104.45
55.45
26.94
26.68
23.02
22.99
f1 (ppm)

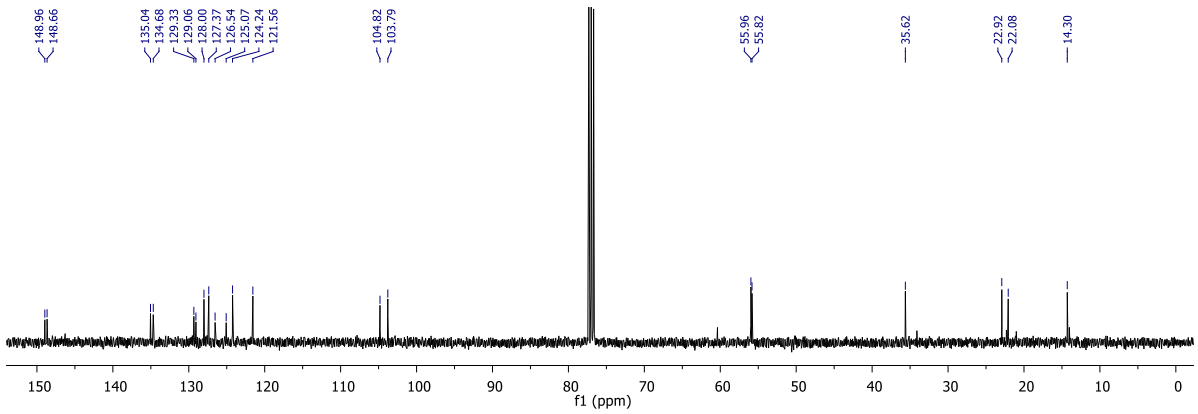

100
95
90
85
80
2930.49
2833.68
1617.74
1530.10
1504.08
1435.16
1362.27
1282.79
1227.84
1177.44
1044.42
1018.36
839.96
806.72
751.53
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

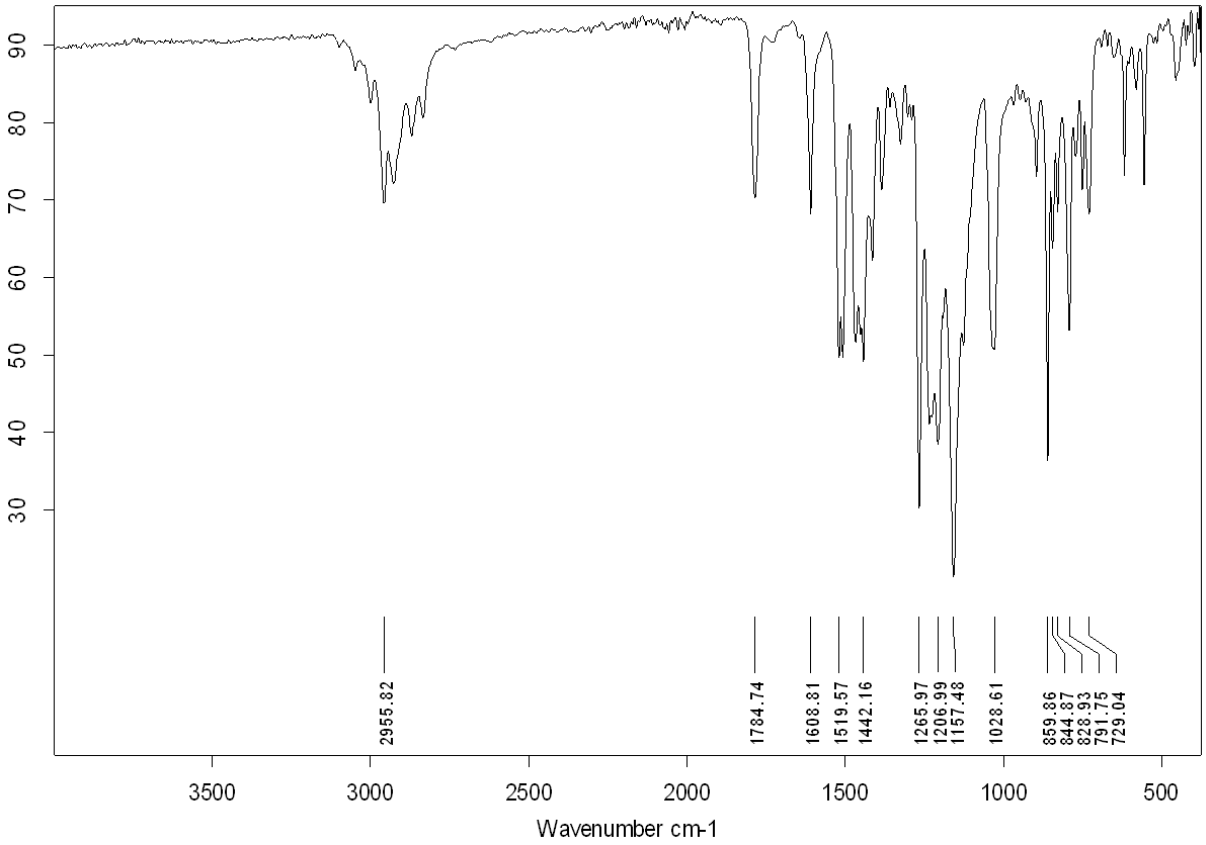

OMe
MeO
Me
F
2r
1.00
1.01
1.00
1.06
1.07
1.32
6.27
2.06
2.11
3.33
f1 (ppm)


162.20
159.78
149.50
148.82
134.89
134.87
130.25
130.16
130.13
130.04
128.01
126.68
124.71
123.71
114.71
114.47
106.91
106.69
104.73
103.82
55.92
55.84
35.50
22.85
14.30
f1 (ppm)


2961.86
2250.22
1528.91
1505.43
1467.47
1435.14
1415.18
1264.84
1200.03
1154.54
1102.62
1030.14
913.21
861.51
845.12
792.12
720.92
643.01
614.53
558.98
Wavenumber cm-1


OMe
MeO
Pr
Me
2s
1.00
0.99
0.97
2.89
5.71
2.33
2.81
1.97
2.84
f1 (ppm)


148.96
148.66
135.04
134.68
129.33
129.06
128.00
127.37
126.54
125.07
124.24
121.56
104.82
103.79
55.96
55.82
35.62
22.92
22.08
14.30
f1 (ppm)


2955.82
1784.74
1608.81
1519.57
1442.16
1265.97
1206.99
1157.48
1028.61
859.86
844.87
828.93
791.75
729.04
Wavenumber cm-1