Supporting Information

Photosensitised Regioselective [2+2]-Cycloaddition of Cinnamates and related Alkenes

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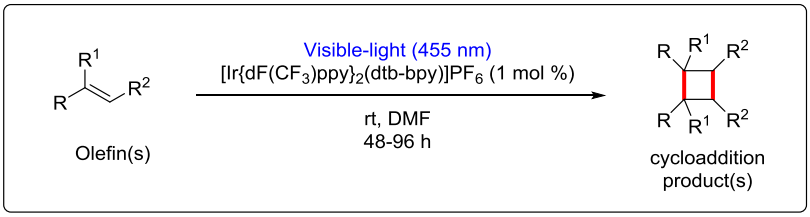
1. General information:

Unless otherwise stated, all commercial chemical materials were used as received without further purification. All reactions were performed using common dry, inert atmosphere techniques. All photochemical reactions were performed under a dry nitrogen atmosphere. The blue light irradiation was done using blue light emitting diodes (700 mA, λ_{max} = 455 nm) produced by Oslon SSL. All the reactions were monitored by TLC and visualized by a dual short/long wavelength UV lamp. Analytical thin layer chromatography was performed on Merck TLC aluminium sheets silica gels 60 F 254. Purifications by column chromatography were performed on silica gel (0.063-0.200 mm). UV-Visible spectra were measured on Varian Cary 50 spectrophotometer. Melting points were recorded on Stanford Research Systems OptiMelt MPA 100 Automated melting point system. All products were characterized by appropriate techniques such as ^{1}H-NMR, ^{19}F-NMR, ^{13}C-NMR, FT-IR and HRMS analysis. FT-IR (Cary 630) spectroscopy was carried out on a spectrometer, equipped with a Diamond Single Reflection ATR-System. NMR spectra were recorded on Bruker Avance 300 and 400 spectrometers. Chemical shifts for ^{1}H-NMR were reported as δ, parts per million, relative to the signal of $CHCl_3$

at 7.26 ppm. Chemical shifts for ^{13}C-NMR were reported as δ, parts per million, relative to the signal of $CHCl_3$ at 77.2 ppm and TMS as an internal standard. Coupling constants (*J*) are given in Hertz (Hz). The following notations indicate the multiplicity of the signals: s = singlet, bs = broad singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, and m = multiplet. Mass spectra were recorded at the Central Analytical Laboratory at the Department of Chemistry of the University of Regensburg on Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS.

2. General procedure (GP-1) for visible-light photocatalysis:



An oven dried 10 mL schlenk flask was charged with olefin (1.00 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in 2.0 mL anhydrous DMF. The resulting suspension was deoxygenated by three freeze-pump-thaw cycles. The reaction mixture was irradiated with blue light emitting diode (LED, λ_{max} = 455 nm) at room temperature for 72 h. Then the reaction mixture was saturated with brine (15 mL) and extracted with ethyl acetate (3 x 20 mL). After drying the combined organic layers on Na_2SO_4, the resulting solution was concentrated *in vacuo*. Purification by silica-gel column chromatography using hexanes and ethyl acetate as eluents afforded cycloaddition product(s).

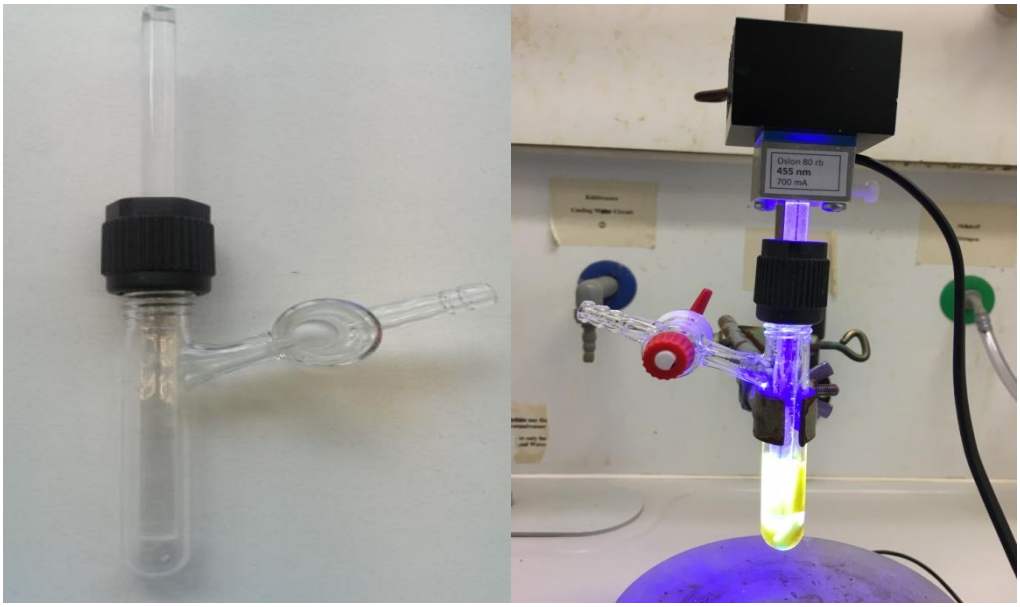
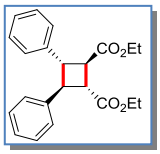


Fig. 1: Experimental set-up for photochemical reaction

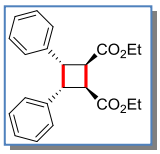
Diethyl (1*R*,2*R*,3*S*,4*S*)-3,4-diphenylcyclobutane-1,2-dicarboxylate (2a):



Following **GP-1**, **2a** was prepared from ethyl cinnamate **1a** (176.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.43) to afford **2a** as a colourless oil (153 mg, 87% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.45 – 7.06 (m, 10H), 4.22 (q, *J* = 7.1 Hz, 4H), 3.79 (d, *J* = 9.6 Hz, 2H), 3.48 (d, *J* = 9.5 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, CDCl3):** δ 172.72, 141.31, 128.73, 127.19, 126.97, 61.13, 47.09, 44.95, 14.37; **IR (neat, cm^{-1}):** 3030, 2981, 2936, 1723, 1602, 1496, 1449, 1416, 1388, 1368, 1317, 1198, 1156, 1095, 1030, 856, 751, 696; **EI-MS:** exact m/z calculated for $C_{22}H_{24}O_4$ $(M)^+$: 352.16691; Found: 352.16799 $(M)^+$.

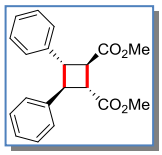
Diethyl (1*R*,2*S*,4*S*)-3,4-diphenylcyclobutane-1,2-dicarboxylate (3a):



Following **GP-1**, **3a** was prepared from ethyl cinnamate **1a** (176.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.39) to afford **3a** as a colourless oil (17 mg, 9% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.37 – 7.15 (m, 4H), 7.12 – 6.95 (m, 4H), 6.94 – 6.85 (m, 2H), 4.40 – 4.32 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 4H), 3.78 (m, 2H), 1.23 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.65, 138.88, 128.15, 127.98, 126.47, 61.16, 45.00, 43.61, 14.39; **IR (neat, cm^{-1}):** 3030, 2981, 2935, 1725, 1602, 1496, 1449, 1267, 1196, 1158, 1095, 1062, 1015, 856, 749, 696; **EI-MS:** exact m/z calculated for $C_{22}H_{24}O_4$ $(M)^+$: 352.16691; Found: 352.16799 $(M)^+$.

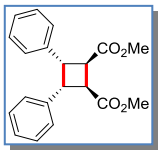
Dimethyl (1*R*,2*R*,3*S*,4*S*)-3,4-diphenylcyclobutane-1,2-dicarboxylate (2b):



Following **GP-1**, **2b** was prepared from methyl cinnamate **1b** (162.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.40) to afford **2b** as a white solid (131 mg, 81% yield); Mp = 69-71 ^{o}C (decomposed).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.40 – 7.22 (m, 10H), 3.78 (m, 2H), 3.75 (s, 6H), 3.56 – 3.50 (m, 2H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 173.12, 141.08, 128.77, 127.29, 126.96, 52.34, 47.47, 44.53; **IR (neat, cm^{-1}):** 3031, 2950, 2923, 2853, 1725, 1602, 1496, 1433, 1313, 1281, 1200, 1167, 1115, 1023, 1006, 905, 775, 753, 733, 696; **HRMS (ESI):** exact m/z calculated for $C_{20}H_{21}O_4$ $(M+H)^+$: 325.1434; Found: 325.1443 $(M+H)^+$.

Dimethyl (1*R*,2*S*,4*S*)-3,4-diphenylcyclobutane-1,2-dicarboxylate (3b):

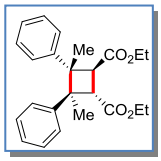


Following **GP-1**, **3b** was prepared from methyl cinnamate **1b** (162.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.36) to afford **3b** as a colourless oil (13 mg, 8% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.15 – 7.01 (m, 6H), 6.95 – 6.89 (m, 4H), 4.41 (dd, *J* = 3.9, 2.3 Hz, 2H), 3.86 (dt, *J* = 3.3, 0.9 Hz, 2H), 3.76 (s, 6H).

^{13}C-NMR (75 MHz, CDCl$_3$): δ 173.13, 138.61, 128.17, 127.92, 126.54, 52.36, 45.08, 43.39; **IR (neat, cm^{-1}):** 3029, 2951, 2923, 1726, 1602, 1496, 1434, 1367, 1270, 1200, 1163, 1062, 1029, 967, 748, 695; **HRMS (ESI):** exact m/z calculated for $C_{20}H_{21}O_4$ $(M+H)^+$: 325.1434; Found: 325.1443 $(M+H)^+$.

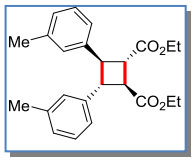
Diethyl (1*S*,2*S*,3*S*,4*S*)-3,4-dimethyl-3,4-diphenylcyclobutane-1,2-dicarboxylate (2c):



Following **GP-1**, **2c** was prepared from ethyl (*E*)-3-phenylbut-2-enoate **1c** (190.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.56) to afford **2c** as a colourless oil (167 mg, 88% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.45 – 7.27 (m, 6H), 7.21 (dd, *J* = 7.7, 1.7 Hz, 4H), 5.92 (d, *J* = 1.3 Hz, 2H), 4.01 (q, *J* = 7.1 Hz, 4H), 2.19 (d, *J* = 1.4 Hz, 6H), 1.09 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 166.08, 155.57, 141.00, 128.03, 127.87, 126.95, 117.90, 59.90, 27.31, 14.10; **IR (neat, cm^{-1}):** 2979, 2937, 1722, 1705, 1638, 1600, 1575, 1492, 1441, 1373, 1273, 1227, 1155, 1095, 1075, 1043, 954, 912, 860, 766, 696; **EI-MS:** exact m/z calculated for $C_{24}H_{28}O_4$ $(M)^+$: 380.19821; Found: 380.19819 $(M)^+$.

Diethyl (1*S*,2*S*,3*R*,4*R*)-3,4-di-*m*-tolylcyclobutane-1,2-dicarboxylate (2d):

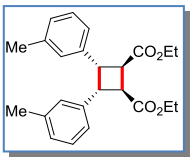


Following **GP-1**, **2d** was prepared from ethyl (*E*)-3-(*m*-tolyl)acrylate **1d** (190.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.49) to afford **2d** as a colourless oil (130 mg, 68% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.24 (t, *J* = 7.5 Hz, 2H), 7.18 – 7.05 (m, 6H), 4.24 (q, *J* = 7.1 Hz, 4H), 3.82 – 3.71 (m, 2H), 3.49 – 3.41 (m, 2H), 2.37 (s, 6H), 1.31 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.79, 141.29, 138.29, 128.58, 127.91, 127.66, 124.08, 61.06, 46.95, 45.02, 21.58, 14.35;

IR (neat, cm^{-1}): 2980, 2924, 2870, 1725, 1606, 1589, 1489, 1460, 1445, 1410, 1385, 1368, 1310, 1197, 1156, 1095, 1021, 857, 778, 698; **EI-MS:** exact m/z calculated for $C_{24}H_{28}O_4$ $(M)^+$: 380.19821; Found: 380.19819 $(M)^+$.

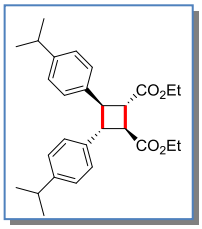
Diethyl (1*R*,2*S*,4*S*)-3,4-di-*m*-tolylcyclobutane-1,2-dicarboxylate (3d):



Following **GP-1**, **3d** was prepared from ethyl (*E*)-3-(*m*-tolyl)acrylate **1d** (190.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.43) to afford **3d** as a colourless oil (43 mg, 23% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.24 – 6.83 (m, 6H), 6.77 – 6.67 (m, 2H), 4.34 (dd, *J* = 3.9, 2.3 Hz, 2H), 4.20 (q, *J* = 7.1 Hz, 4H), 3.85 – 3.78 (m, 2H), 2.19 (s, 6H), 1.29 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.71, 138.82, 137.51, 128.87, 127.92, 127.13, 125.00, 61.09, 44.91, 43.64, 21.49, 14.38; **IR (neat, cm^{-1}):** 2979, 2924, 2856, 1727, 1606, 1589, 1489, 1460, 1446, 1372, 1349, 1300, 1274, 1198, 1159, 1095, 1066, 1024, 878, 857, 776, 698; **EI-MS:** exact m/z calculated for $C_{24}H_{28}O_4$ $(M)^+$: 380.19821; Found: 380.19819 $(M)^+$.

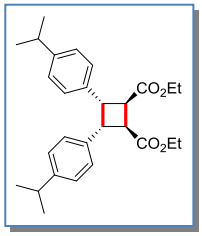
Diethyl (1*S*,2*S*,3*R*,4*R*)-3,4-bis(4-isopropylphenyl)cyclobutane-1,2-dicarboxylate (2e):



Following **GP-1**, **2e** was prepared from ethyl (*E*)-3-(4-isopropylphenyl)acrylate **1e** (218.3 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.52) to afford **2e** as a colourless oil (163 mg, 75% yield).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.32 – 7.26 (m, 4H), 7.24 – 7.18 (m, 4H), 4.23 (q, *J* = 7.1 Hz, 4H), 3.84 – 3.73 (m, 2H), 3.48 – 3.40 (m, 2H), 2.92 (m, 2H), 1.31 (t, *J* = 7.1 6H), 1.27 (d, *J* = 6.9 Hz, 12H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.83, 147.67, 138.82, 126.93, 126.71, 61.03, 46.70, 45.11, 33.88, 24.11, 14.37; **IR (neat, cm^{-1}):** 2960, 2931, 2871, 1726, 1513, 1461, 1409, 1366, 1316, 1263, 1199, 1156, 1097, 1033, 1017, 824; **EI-MS:** exact m/z calculated for $C_{28}H_{36}O_4$ $(M)^+$: 436.26081; Found: 436.25936 $(M)^+$.

Diethyl (1*R*,2*S*,4*S*)-3,4-bis(4-isopropylphenyl)cyclobutane-1,2-dicarboxylate (3e):

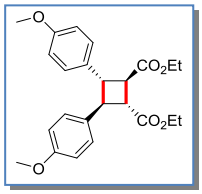


Following **GP-1**, **3e** was prepared from ethyl (*E*)-3-(4-isopropylphenyl)acrylate **1e** (218.3 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.46) to afford

3e as a colourless oil (31 mg, 14% yield).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.23 – 6.90 (m, 4H), 6.88 – 6.78 (m, 4H), 4.42 – 4.26 (m, 2H), 4.19 (q, *J* = 7.1 Hz, 4H), 3.85 – 3.78 (m, 2H), 2.88 – 2.60 (m, 2H), 1.28 (t, *J* = 7.1 Hz, 6H), 1.12 (d, *J* = 7.0 Hz, 12H); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 172.81, 146.95, 136.29, 127.89, 126.03, 61.06, 44.75, 43.62, 33.92, 33.74, 29.88, 24.16, 24.09, 14.39; **IR (neat, cm^{-1}):** 2959, 2925, 2855, 1727, 1606, 1513, 1461, 1373, 1265, 1191, 1158, 1097, 1056, 1017, 827; **EI-MS:** exact m/z calculated for $C_{28}H_{36}O_4$ $(M)^+$: 436.26081; Found: 436.25936 $(M)^+$.

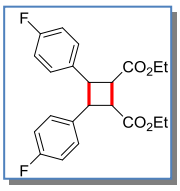
Diethyl (1*R*,2*R*,3*S*,4*S*)-3,4-bis(4-methoxyphenyl)cyclobutane-1,2-dicarboxylate (2f):



Following **GP-1**, **2f** was prepared from ethyl (*E*)-3-(4-methoxyphenyl)acrylate **1f** (206.2 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.40) to afford **2f** as a colourless oil (122 mg, 59% yield) and a mixture of **2f/3f** as yellow oil (49 mg, 27% yield, 1:0.84 d.r.).

^{1}H-NMR (300 MHz, $CDCl_3$, mixture of both isomers): δ 7.24 – 7.17 (m, 2H), 6.88 – 6.81 (m, 4H), 6.66 (d, *J* = 8.8 Hz, 2H), 4.29 (dd, *J*=4.0, 2.2, 1H), 4.19 (qd, *J*=7.1, 1.6, 4H), 3.79 (s, 3H), 3.77 (d, *J* =1.4 Hz, 1H), 3.76 – 3.72 (m, 1H), 3.71 (d, *J* = 2.2 Hz, 2H), 3.64 – 3.59 (m, 1H), 3.39 – 3.34 (m, 1H), 1.27 (td, *J* = 7.1, 4.5 Hz, 6H); **^{1}H-NMR (300 MHz, $CDCl_3$, *trans* isomer):** δ 7.21 – 7.13 (m, 2H), 6.85 – 6.74 (m, 2H), 6.85 – 6.76 (m, 2H), 4.14 (q, *J* = 7.1, 2H), 3.77 – 3.70 (m, 6H), 3.61 – 3.54 (m, 1H), 3.36 – 3.28 (m, 1H), 1.21 (t, *J* = *7.1* Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, *trans* isomer):** δ 172.71, 158.67, 133.38, 127.95, 113.99, 77.38, 77.06, 76.74, 60.94, 55.29, 46.94, 45.01, 14.27; **IR (neat, cm^{-1}):** 2982, 2836, 1722, 1610, 1513, 1461, 1245, 1033, 826, 731; **HRMS (ESI):** exact m/z calculated for mixture: $C_{24}H_{28}O_6$ $(M+H)^+$: 413.1959; Found: 413.1959 $(M+H)^+$; For *trans*: exact m/z calculated for $C_{24}H_{28}O_6$ $(M+H)^+$: 413.1959; Found: 413.1959 $(M+H)^+$; For *cis*: exact m/z calculated for $C_{24}H_{28}O_6$ $(M+H)^+$: 413.1959; Found: 413.1961 $(M+H)^+$.

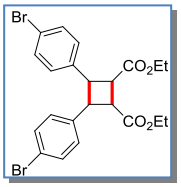
Diethyl-3,4-bis(4-fluorophenyl)cyclobutane-1,2-dicarboxylate (2g/3g):



Following **GP-1**, **2g/3g** was prepared from ethyl (*E*)-3-(4-fluorophenyl)acrylate **1g** (194.2 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.20 (trans), R_f = 0.17 (cis) to afford mixture of **2g/3g** as a white solid (139 mg, 72% yield; d.r. = 6:1); Mp = 60-62 ^{o}C (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.39 – 7.12 (m, 4H), 7.01 (t, *J* = 8.6 Hz, 4H), 4.20 (q, *J* = 7.1 Hz, 4H), 3.75 – 3.55 (m, 2H), 3.47 – 3.31 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 172.52, 162.12 (d, $^{1}J_{C\text{-}F}$ = 245.5 Hz), 136.78 (d, $^{4}J_{C\text{-}F}$ = 3.1 Hz), 128.51 (d, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 115.68 (d, $^{2}J_{C\text{-}F}$ = 21.4 Hz), 61.29, 46.78, 45.05, 14.38; **^{19}F-NMR (282 MHz, $CDCl_3$):** δ -115.85; **IR (neat, cm^{-1}):** 2989, 2944, 1718, 1601, 1509, 1473, 1447, 1393, 1368, 1300, 1215, 1196, 1155, 1109, 1033, 1015, 965, 870, 824, 791, 671; **EI-MS:** exact m/z calculated for $C_{22}H_{22}O_4F_2$ $(M)^+$: 388.14807; Found: 388.14844 $(M)^+$.

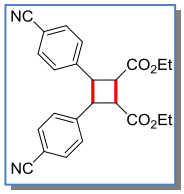
Diethyl-3,4-bis(4-bromophenyl)cyclobutane-1,2-dicarboxylate (2h/3h):



Following **GP-1**, **2h/3h** was prepared from ethyl (*E*)-3-(4-bromophenyl)acrylate **1h** (255.1 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.20 (trans), R_f = 0.17 (cis) to afford mixture of **2h/3h** as a white solid (125 mg, 49% yield; d.r. = 4:1); Mp = 92-94 ^{o}C (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.48 – 7.41 (m, 4H), 7.18 – 7.11 (m, 4H), 4.20 (q, *J* = 7.1 Hz, 4H), 3.66 – 3.58 (m, 2H), 3.42 – 3.34 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 172.35, 139.85, 131.96, 128.69, 121.32, 61.38, 46.73, 44.73, 14.39; **IR (neat, cm^{-1}):** 2985, 2937, 1717, 1588, 1486, 1394, 1364, 1315, 1294, 1273, 1195, 1159, 1111, 1070, 1006, 962, 864, 812, 755, 702; **EI-MS:** exact m/z calculated for $C_{22}H_{22}O_4Br_2$ $(M)^+$: 507.98498; Found: 507.98582 $(M)^+$.

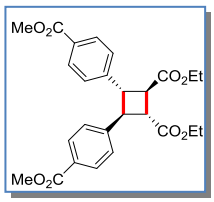
Diethyl-3,4-bis(4-cyanophenyl)cyclobutane-1,2-dicarboxylate (2i/3i):



Following **GP-1**, **2i/3i** was prepared from ethyl (*E*)-3-(4-cyanophenyl)acrylate **1i** (201.2 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.20) to afford a mixture of **2i/3i** as a white solid (171 mg, 85% yield, 1.15:1 d.r.); Mp = 121-122 °C (decomposed).

^{1}H-NMR (400 MHz, $CDCl_3$, *trans* isomer): δ 7.68 – 7.58 (m, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 4.22 (qd, *J* = 7.1 Hz, 1.6, 2H), 3.87 – 3.74 (m, 1H), 3.59 – 3.40 (m, 1H), 1.27 (t, *J* = 7.1 Hz, 3H); **^{13}C-NMR (101 MHz, $CDCl_3$, *trans* isomer):** δ 172.71, 158.67, 133.38, 127.95, 113.99, 77.38, 77.06, 76.74, 60.94, 55.29, 46.94, 45.01, 14.27; **IR (neat, cm^{-1}):** 3064, 2989, 2229, 1715, 1607, 1506, 1372, 1312, 1200, 1163, 1014, 828; **HRMS (ESI):** exact m/z calculated for $C_{24}H_{22}N_2O_4$ $(M+H)^+$: 403.1652; Found: 403.1652 $(M+H)^+$.

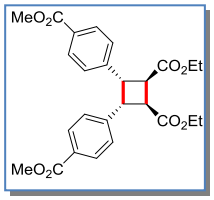
Diethyl (1*S*,2*S*,3*R*,4*R*)-3,4-bis(4-(methoxycarbonyl)phenyl)cyclobutane-1,2-dicarboxylate (2j):



Following **GP-1**, **2j** was prepared from methyl (*E*)-4-(3-ethoxy-3-oxoprop-1-en-1-yl)benzoate **1j** (234.3 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1) to afford **2j** as colourless oil (130 mg, 56% yield).

^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.71 (d, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 4.42 (dd, *J* = 3.9, 2.2, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.84 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.13, 166.79, 145.80, 130.07, 129.18, 126.85, 77.47, 77.05, 76.63, 61.30, 52.17, 46.79, 44.52, 14.22; **IR (neat, cm^{-1}):** 2952, 1718, 1610, 1435, 1159, 1275, 1193, 1103, 1018, 965, 857, 768, 701; **HRMS (ESI):** exact m/z calculated for $C_{26}H_{28}O_8$ $(M+H)^+$: 469.1857; Found: 469.1862 $(M+H)^+$.

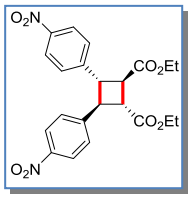
Diethyl (1*R*,2*S*,3*R*,4*S*)-3,4-bis(4-(methoxycarbonyl)phenyl)cyclobutane-1,2-dicarboxylate (3j):



Following **GP-1**, **3j** was prepared from methyl (*E*)-4-(3-ethoxy-3-oxoprop-1-en-1-yl)benzoate **1j** (234.3 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1) to afford **3j** as colourless oil (79 mg, 34% yield).

^{1}H-NMR (400 MHz, CDCl$_3$): δ = 7.71 (d, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 4.42 (dd, *J* = 3.9 Hz, 2.2, 1H), 4.16 (q, *J* =7.1 Hz, 2H), 3.84 (d, *J* = 1.6 Hz, 1H), 3.78 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 172.13, 166.79, 145.80, 130.07, 129.18, 126.85, 77.47, 77.05, 76.63, 61.30, 52.17, 46.79, 44.52, 14.22; **IR (neat, cm^{-1}):** 2952, 1718, 1610,1435, 1159, 1275, 1193, 1103, 1018, 965, 857, 768, 701; **HRMS (ESI):** exact m/z calculated for $C_{26}H_{28}O_8$ $(M+H)^+$: 469.1857; Found: 469.1860 $(M+H)^+$.

Diethyl (1*S*,2*S*,3*R*,4*R*)-3,4-bis(4-nitrophenyl)cyclobutane-1,2-dicarboxylate (2k):

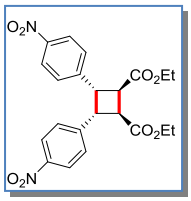


Following **GP-1**, **2k** was prepared from ethyl (*E*)-3-(4-nitrophenyl)acrylate **1k** (221.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 10:1, R_f = 0.2) to afford **2k** as a orange viscous oil (115 mg, 49% yield).

^{1}H-NMR (400 MHz, CDCl$_3$): δ 8.24 – 8.16 (m, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 4.24 (qd, *J* = 7.1 Hz, 1.4, 2H), 3.90 – 3.81 (m, 1H), 3.53 – 3.43 (m, 1H), 1.33 – 1.23 (m, 3H); **^{13}C-NMR (101**

MHz, $CDCl_3$): δ 171.61, 147.50, 147.34, 127.72, 124.17, 77.38, 77.07, 76.75, 61.62, 46.36, 44.60, 14.22; **IR (neat, cm^{-1}):** 2982, 2937, 1722, 1603, 1517, 1342, 1200, 1159, 1111. 1014, 854, 746; **HRMS (ESI):** exact m/z calculated for $C_{22}H_{22}N_2O_8$ $(M+H)^+$: 443.1449; Found: 443.1457 $(M+H)^+$.

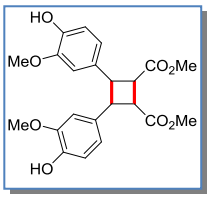
Diethyl (1*R*,2*S*,3*R*,4*S*)-3,4-bis(4-nitrophenyl)cyclobutane-1,2-dicarboxylate (3k):



Following **GP-1**, **3k** was prepared from ethyl (*E*)-3-(4-nitrophenyl)acrylate **1k** (221.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 10:1, R_f = 0.15) to afford **3k** with impurities of **2k** as orange oil. (87 mg, 37% yield).

^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.8 Hz, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 4.57 (d, *J* = 6.2 Hz, 1H), 4.24 (q, *J* =7.1 Hz, 2H), 3.86 (dd, *J* = 3.7, 2.4 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H); **IR (neat, cm^{-1}):** 2982, 2937, 1722, 1603, 1517, 1342, 1200, 1159, 1111. 1014, 854, 746; **HRMS (ESI):** exact m/z calculated for $C_{22}H_{22}N_2O_8$ $(M+H)^+$: 443.1449; Found: 443.1455 $(M+H)^+$.

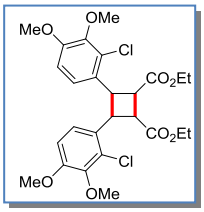
Dimethyl-3,4-bis(4-hydroxy-3-methoxyphenyl)cyclobutane-1,2-dicarboxylate (2l/3l):



Following **GP-1**, **2l/3l** was prepared from methyl (*E*)-3-(4-hydroxy-3-methoxyphenyl)acrylate **1l** (208.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 1:1, R_f = 0.20) to afford mixture of **2l/3l** as a colourless oil (135 mg, 65% yield; d.r. = 2.94:1).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 6.86 (d, *J* = 8.2 Hz, 2H), 6.82 – 6.74 (m, 4H), 5.65 (bs, 2H), 3.84 (s, 6H), 3.74 (s, 6H), 3.61 – 3.53 (m, 2H), 3.45 – 3.38 (m, 2H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 173.28, 146.70, 144.89, 133.08, 119.71, 114.57, 109.48, 56.01, 52.36, 47.98, 44.61; **IR (neat, cm^{-1}):** 3433, 2952, 2844, 1721, 1602, 1514, 1434, 1264, 1235, 1200, 1156, 1121, 1028, 851, 812, 766, 733, 700; **HRMS (ESI):** exact m/z calculated for $C_{22}H_{25}O_8$ $(M+H)^+$: 417.1544; Found: 417.1546 $(M+H)^+$.

Diethyl-3,4-bis(2-chloro-3,4-dimethoxyphenyl)cyclobutane-1,2-dicarboxylate (2m/3m):

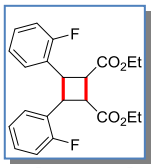


Following **GP-1**, **2m/3m** was prepared from ethyl (*E*)-3-(2-chloro-3,4-dimethoxyphenyl)acrylate **1m** (270.7 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel,

hexanes–EtOAc, 4:1, R_f = 0.16 (trans), R_f = 0.14 (cis) to afford mixture of **2m/3m** as a yellow solid (189 mg, 70% yield; d.r. = 6:1); Mp = 125-127 ^oC (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.26 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 4.23 – 4.19 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 4H), 3.84 (s, 6H), 3.81 (s, 6H), 3.41 – 3.24 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 172.57, 152.89, 145.37, 130.94, 128.54, 123.17, 110.82, 61.18, 60.62, 56.15, 45.45, 43.21, 14.25; **IR (neat, cm^{-1}):** 2934, 2839, 1721, 1594, 1490, 1462, 1439, 1420, 1294, 1267, 1205, 1176, 1149, 1036, 1012, 980, 841, 807, 774, 710, 666; **HRMS (ESI):** exact m/z calculated for $C_{26}H_{31}Cl_2O_8$ $(M+H)^+$: 541.1390; Found: 541.1403 $(M+H)^+$.

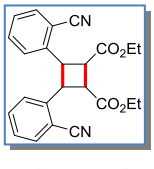
Diethyl-3,4-bis(2-fluorophenyl)cyclobutane-1,2-dicarboxylate (2n/3n):



Following **GP-1**, **2n/3n** was prepared from ethyl (*E*)-3-(2-fluorophenyl)acrylate **1n** (194.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.25 (trans), R_f = 0.20 (cis) to afford mixture of **2n/3n** as a white solid (143 mg, 74% yield; d.r. = 5:1); Mp = 57-59 ^oC (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.42 (td, *J* = 7.5, 1.7 Hz, 2H), 7.23 (tdd, *J* = 7.2, 5.1, 1.8 Hz, 2H), 7.13 (tt, *J* = 9.0, 4.5 Hz, 2H), 7.06 – 6.98 (m, 2H), 4.20 (q, *J* = 7.1 Hz, 4H), 4.13 – 4.04 (m, 2H), 3.53 – 3.43 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 172.35, 160.98 (d, $^1J_{C\text{-}F}$ = 246.2 Hz), 128.90 (d, $^3J_{C\text{-}F}$ = 8.3 Hz), 128.43 (d, $^3J_{C\text{-}F}$ = 4.7 Hz), 127.57 (d, $^2J_{C\text{-}F}$ = 15.1 Hz), 124.46 (d, $^4J_{C\text{-}F}$ = 3.6 Hz), 115.55 (d, $^2J_{C\text{-}F}$ = 22.0 Hz), 61.21, 44.84, 40.09, 14.29; **^{19}F-NMR (282 MHz, $CDCl_3$):** δ -116.94; **IR (neat, cm^{-1}):** 2988, 2944, 1719, 1602, 1509, 1472, 1446, 1393, 1368, 1302, 1216, 1196, 1156, 1107, 1034, 1015, 965, 826, 807, 768, 731, 671; **EI-MS:** exact m/z calculated for $C_{22}H_{22}O_4F_2$ $(M)^+$: 388.14807; Found: 388.14844 $(M)^+$.

Diethyl-3,4-bis(2-cyanophenyl)cyclobutane-1,2-dicarboxylate (2o/3o):

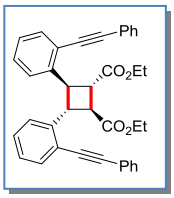


Following **GP-1**, **2o/3o** was prepared from ethyl (*E*)-3-(2-cyanophenyl)acrylate **1o** (201.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 85:15, R_f = 0.40) to afford mixture of **2o/3o** as a white solid (127 mg, 63% yield; d.r. = 7.34:1); Mp = 110-112 ^oC (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.79 (d, *J* = 7.5 Hz, 2H), 7.73 – 7.65 (m, 2H), 7.59 (dd, *J* = 7.8, 1.1 Hz, 2H), 7.42 – 7.35 (m, 2H), 4.22 (q, *J* = 7.1 Hz, 4H), 4.20 – 4.15 (m, 2H),

3.63 – 3.50 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 6H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 171.46, 142.92, 133.83, 133.18, 128.34, 127.67, 117.63, 112.49, 61.74, 14.22; **IR (neat, cm^{-1}):** 2988, 2931, 2222, 1715, 1597, 1473, 1446, 1369, 1320, 1255, 1218, 1193, 1169, 1122, 1103, 1023, 970, 853, 769, 728, 676; **EI-MS:** exact m/z calculated for $C_{24}H_{22}N_2O_4$ $(M)^+$: 402.15607; Found: 402.15624 $(M)^+$.

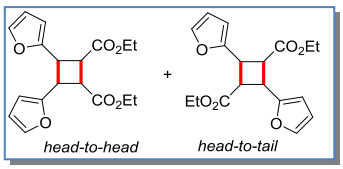
Diethyl (1*S*,2*S*,3*R*,4*R*)-3,4-bis(2-(phenylethynyl)phenyl)cyclobutane-1,2-dicarboxylate (2p):



Following **GP-1**, **2p** was prepared from ethyl (*E*)-3-(2-(phenylethynyl)phenyl)acrylate **1p** (276.3 mg, 1.0 mmol, 1.00 equiv). Purification of the crude product by column chromatography (Hexanes: EtOAc, 9:1, R_f = 0.37) afforded **2p** as a pale yellow oil (174 mg, 63% yield; d.r. = 20:1).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.66 (d, *J* = 7.2 Hz, 1H), 7.52 – 7.41 (m, 3H), 7.37 – 7.30 (m, 3H), 7.27 (td, *J* = 7.6, 1.4 Hz, 1H), 7.14 (td, *J* = 7.5, 1.2 Hz, 1H), 4.55 – 4.51 (m, 1H), 4.02 (q, *J* = 7.1 Hz, 2H), 3.57 – 3.52 (m, 1H), 1.12 (t, *J* = 7.1 Hz, 3H); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 172.71, 142.06, 132.60, 131.79, 129.00, 128.41, 127.08, 127.02, 123.39, 123.05, 93.65, 88.00, 61.05, 45.52, 45.03, 14.20; **IR (neat, cm^{-1}):** 2925, 2854, 1723, 1598, 1493, 1443, 1368, 1318, 1201, 1157, 1095, 1027, 914, 855, 752, 689; **EI-MS:** exact m/z calculated for $C_{38}H_{33}O_4$ $(M+H)^+$: 553.2373; Found: 553.2382 $(M+H)^+$.

Diethyl 3,4-di(furan-2-yl)cyclobutane-1,2-dicarboxylate (2r/3r) and diethyl 2,4-di(furan-2-yl)cyclobutane-1,3-dicarboxylate (2q/3q/4q):

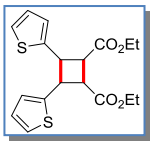


Following **GP-1**, **2q/3q/4q** was prepared from ethyl (*E*)-3-(furan-2-yl)acrylate **1r** (166.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.4-0.2) to afford **4q** as orange oil (27 mg, 16% yield) and a mixture of **2r/3r/4r** as orange oil. (88 mg, 53% yield).

^{1}H-NMR (300 MHz, $CDCl_3$, *trans* isomer): δ 7.36 (dd, *J* = 1.8, 0.8 Hz, 1H), 6.30 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.16 (dd, *J* = 3.2, 0.7 Hz, 1H), 4.18 (qd, *J* =7.1, 0.6 Hz, 2H), 3.82 – 3.72 (m, 1H), 3.53 – 3.44 (m, 1H), 1.25 (t, *J* = 7.1, 3H); **^{1}H-NMR (300 MHz, $CDCl_3$, *cis* isomer**): δ = 7.22 (dd, *J* = 1.8, 0.8 Hz, 1H), 6.20 (dd, *J* = 3.2, 1.8 Hz, 1H), 5.94 (d, *J* = 2.8 Hz, 1H), 4.25 (dd, *J* = 3.8, 2.2, 1H), 4.18 (qd, *J* = 7.1, 1.5 Hz, 2H), 3.85 (dd, *J* = 3.7, 2.2, 1H), 1.26 (td, *J* =7.1, 3.6 Hz, 3H); **^{13}C-NMR (101 MHz, $CDCl_3$, *trans* isomer):** δ 171.88, 153.36, 142.17, 110.37, 106.58, 77.35, 77.03, 76.71, 61.08, 43.37, 39.44, 14.19; **IR (neat, cm^{-1}):** 2982, 2941, 1800, 1726, 1372, 1200, 1096, 1014, 924, 738; **HRMS (ESI):** ***trans***: exact m/z calculated for $C_{18}H_{20}O_6$ $(M+H)^+$:

333.1338; Found: 333.1334 $(M+H)^+$; ***cis***: exact m/z calculated for $C_{18}H_{20}O_6$ $(M+H)^+$: 333.1338; Found: 333.1336 $(M+H)^+$; **head-to-tail conformer**: exact m/z calculated for $C_{18}H_{20}O_6$ $(M+H)^+$: 333.1338; Found: 333.1335 $(M+H)^+$.

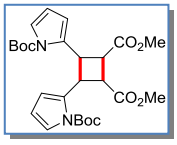
Diethyl-3,4-di(thiophen-2-yl)cyclobutane-1,2-dicarboxylate (2r/3r, 4r):



Following **GP-1**, **2r/3r, 4r** was prepared from methyl (*E*)-3-(thiophen-2-yl)acrylate (188 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.58) to afford mixture of **2r/3r, 4r** as a yellow oil (145.1 mg, 77% yield; d.r. = 1.8:1).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.21 (dd, *J* = 4.2, 2.1 Hz, 2H, *trans*), 7.09 (dd, *J* = 5.1, 1.1 Hz, 1H, *cis*), 7.04 (dd, *J* = 4.9, 0.8 Hz, 0.2H, head-to-tail conformer), 7.00 – 6.93 (m, 4H, *trans*), 6.85 (dd, *J* = 5.0, 3.5 Hz, 1H, *cis*), 6.81 (dd, *J* = 5.1, 3.6 Hz, 0.2H, head-to-tail conformer), 6.73 (dd, *J* = 3.5, 1.0 Hz, 1H, *cis*), 4.52 (dd, *J* = 3.9, 2.2 Hz, 1H, *cis*), 4.26 – 4.13 (m, 7H, *trans/cis*), 3.88 – 3.82 (m, 2H, *trans*), 3.79 (dd, *J* = 3.7, 2.2 Hz, 1H, *cis*), 3.48 – 3.36 (m, 2H, *trans*), 1.29 (t under t, *J* = 7.1, 3.8 Hz, 10H, *trans/cis*), 0.96 (t under t, *J* = 7.1, 4.9 Hz, 0.9H, head-to-tail conformer); **^{13}C-NMR (75 MHz, $CDCl_3$, *trans*/*cis* isomer, head-to-tail conformer):** δ 172.57, 171.76, 171.68, 170.27, 143.92, 141.83, 141.35, 138.63, 127.08, 126.66, 126.62, 126.47, 126.39, 125.46, 125.33, 124.86, 124.62, 124.50, 124.39, 77.55, 77.13, 76.70, 61.23, 61.19, 60.80, 46.06, 45.85, 44.71, 44.63, 43.47, 42.30, 41.39, 41.02, 14.24, 14.21, 13.86; **IR (neat, cm^{-1}):** 3108, 2981, 1722, 1442, 1371, 1297, 1192, 1036, 849, 790, 693; **HRMS (ESI):** ***trans***: exact m/z calculated for $C_{18}H_{21}S_2O_4$ $(M+H)^+$: 365.0876; Found: 365.0880 $(M+H)^+$; ***cis***: exact m/z calculated for $C_{18}H_{21}S_2O_4$ $(M+H)^+$: 365.0876; Found: 365.0880 $(M+H)^+$; **head-to-tail conformer**: exact m/z calculated for $C_{18}H_{21}S_2O_4$ $(M+H)^+$: 365.0876; Found: 365.0880 $(M+H)^+$.

Dimethyl-3,4-bis(1-(*tert*-butoxycarbonyl)-1*H*-pyrrol-2-yl)cyclobutane-1,2-dicarboxylate (2s/3s):

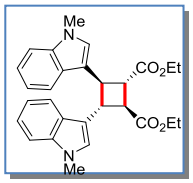


Following **GP-1**, **2s/3s** was prepared from methyl *tert*-butyl (*E*)-2-(3-methoxy-3-oxoprop-1-en-1-yl)-1*H*-pyrrole-1-carboxylate **1s** (263.3 mg, 1.0 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.28) to afford mixture of **2s/3s** as a yellow oil (137.5 mg, 54% yield; d.r. = 1.38:1).

^{1}H-NMR (300 MHz, $CDCl_3$, *trans* isomer): δ 7.10 (dd, *J* = 3.3, 1.7 Hz, 2H), 5.98 (t, *J* = 3.4 Hz, 2H), 5.77 (dd, *J* = 3.4, 1.7 Hz, 2H), 5.02 – 4.92 (m, 2H), 3.71 (s, 6H), 3.60 – 3.54 (m, 2H), 1.53

(s, 18H); **^{1}H-NMR (300 MHz, $CDCl_3$, *cis* isomer):** 7.13 (dd, *J* = 3.3, 1.8 Hz, 2H), 6.29 (dd, *J* = 3.3, 1.7 Hz, 2H), 6.09 (t, *J* = 3.4 Hz, 2H), 4.46 – 4.40 (m, 2H), 3.68 (s, 6H), 3.34 – 3.27 (m, 2H), 1.52 (s, 18H); **^{13}C-NMR (75 MHz, $CDCl_3$, *trans/cis* isomer):** δ 172.84, 172.79, 149.12, 148.96, 135.31, 133.30, 121.72 (*cis*), 121.64, 111.84 (*cis*), 110.98, 110.26 , 109.61, 83.52, 83.42, 51.99, 51.97 (*cis*), 45.33 (*cis*), 44.05, 40.49 (*cis*), 38.44, 27.97, 27.92; **IR (neat, cm^{-1}):** 2981, 1733, 1435, 1319, 1159, 1125, 1066, 846, 723; **HRMS (ESI): trans**: exact m/z calculated for $C_{26}H_{35}N_2O_8$ $(M+H)^+$: 503.2388; Found: 503.2390 $(M+H)^+$; **cis**: exact m/z calculated for $C_{26}H_{35}N_2O_8$ $(M+H)^+$: 503.2388; Found: 503.2390 $(M+H)^+$.

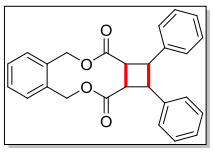
Diethyl (1*S*,3*R*)-3,4-bis(1-methyl-1*H*-indol-3-yl)cyclobutane-1,2-dicarboxylate (2t):



Following **GP-1**, **2t** was prepared from methyl (*E*)-3-(1-methyl-1*H*-indol-3-yl)acrylate **1t** (233.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.23) to afford mixture of **2t** as a yellow oil (51.6 mg, 11% yield).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.69 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.38 – 7.04 (m, 4H), 4.32 – 4.14 (m, 3H), 3.74 (s, *J* = 6.6 Hz, 3H), 3.63 – 3.57 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 173.41, 137.40, 127.02, 126.54, 121.73, 119.89, 119.01, 115.51, 109.33, 77.52, 77.09, 76.67, 60.92, 45.88, 39.83, 32.72, 14.30.; **IR (neat, cm^{-1}):** 3049, 2978, 2929, 1710, 1613, 1550, 1472, 1371, 1315, 1244, 1203, 1177, 1036, 834, 738; **HRMS (ESI):** exact m/z calculated for $C_{28}H_{30}N_2O_4Na$ $(M+Na)^+$: 481.2098; Found: 481.2104 $(M+Na)^+$.

1,2-diphenyl-1,2,2a,5,10,12a-hexahydrobenzo[c]cyclobuta[h][1,6]dioxecine-3,12-dione (2u/3u):

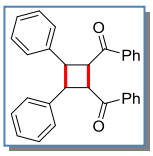


Following **GP-1**, **2u/3u** was prepared from 1,2-phenylenebis(methylene) (2*E*,2'*E*)-bis(3-phenylacrylate) **1u** (398.5 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.3) to afford the major diastereomer of **2u** as white solid (168 mg, 42% yield) and a mixture of both diastereomers of **2u/3u** as colorless oil. (142 mg, 35% yield 1:1.36 d.r.); Mp = 153–154 °C (decomposed).

^{1}H-NMR (400 MHz, $CDCl_3$, major isomer): δ 7.48 – 7.38 (m, 2H), 7.14 – 7.01 (m, 3H), 6.91 (dd, *J* = 5.2, 3.2 Hz, 2H), 5.27 (dd, *J* = 109.2, 12.2 Hz, 2H), 4.46 (dd, *J* = 3.8, 2.3 Hz, 1H), 3.81 (dd, *J* = 3.8, 2.3 Hz, 1H); **^{13}C-NMR (101 MHz, $CDCl_3$, major isomer):** δ 171.47, 138.25, 135.22, 131.58, 129.27, 128.09, 127.75, 126.45, 67.66, 44.31, 43.71; **IR (neat, cm^{-1}):** 3027, 2060, 2963, 1730, 1498, 1446, 1256, 1185, 1163, 1048, 1018, 839, 749, 697; **HRMS (ESI)**

major isomer: exact m/z calculated for $C_{26}H_{22}O_4$ $(M+H)^+$: 399.1591; Found: 399.1590 $(M+H)^+$. **minor isomer:** exact m/z calculated for $C_{26}H_{22}O_4$ $(M+H)^+$: 399.1591; Found: 399.1588 $(M+H)^+$.

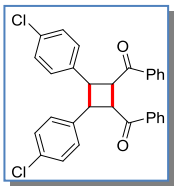
3,4-diphenylcyclobutane-1,2-diyl)bis(phenylmethanone) (2v/3v):



Following **GP-1**, **2v/3v** was prepared from (*E*)-chalcone **1v** (208.2 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.30 (trans), R_f = 0.15 (cis) to afford mixture of **2v/3v** as a white solid (167 mg, 80% yield; d.r. = 3:1); Mp = 54-56 ^oC (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.81 (dt, *J* = 8.5, 1.7 Hz, 4H), 7.48 – 7.40 (m, 2H), 7.33 – 7.17 (m, 14H), 4.63 – 4.57 (m, 2H), 3.99 – 3.93 (m, 2H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 199.20, 141.55, 135.70, 133.63, 129.00, 128.86, 128.69, 127.58, 127.37, 48.07, 47.79; **IR (neat, cm^{-1}):** 2925, 1741, 1663, 1595, 1579, 1491, 1448, 1381, 1293, 1273, 1208, 1178, 1154, 1075, 1020, 985, 910, 857, 802, 775, 749, 689, 661; **HRMS (ESI):** exact m/z calculated for $C_{30}H_{25}O_2$ $(M+H)^+$: 417.1849; Found: 417.1848 $(M+H)^+$.

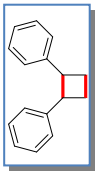
3,4-bis(4-chlorophenyl)cyclobutane-1,2-diyl)bis(phenylmethanone) (2w/3w):



Following **GP-1**, **2w/3w** was prepared from (*E*)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one **1w** (242.7 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography [silica gel, hexanes–EtOAc, 9:1, R_f = 0.28 (*trans*); R_f = 0.17 (*cis*)] to afford mixture of **2w/3w** as a white solid (179 mg, 74% yield; d.r. = 3:1); Mp = 119-121 ^oC (decomposed).

^{1}H-NMR (300 MHz, $CDCl_3$, major isomer): δ 7.85 – 7.78 (m, 4H), 7.49 (ddd, *J* = 6.9, 2.4, 1.2 Hz, 2H), 7.39 – 7.18 (m, 12H), 4.61 – 4.53 (m, 2H), 3.92 – 3.85 (m, 2H); **^{13}C-NMR (75 MHz, $CDCl_3$, major isomer):** δ 198.78, 139.71, 135.45, 133.88, 133.29, 129.10, 128.93, 128.86, 128.83, 47.57, 47.47; **IR (neat, cm^{-1}):** 3058, 2927, 1733, 1670, 1636, 1594, 1579, 1559, 1488, 1446, 1403, 1373, 1316, 1296, 1275, 1230, 1210, 1179, 1089, 1043, 1012, 934, 912, 859, 821, 701, 685; **HRMS (ESI):** exact m/z calculated for $C_{30}H_{23}Cl_2O_2$ $(M+H)^+$: 485.1070; Found: 485.1069 $(M+H)^+$.

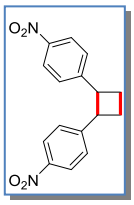
1,2-diphenylcyclobutane (6a):



Following **GP-1**, **6a** was prepared from styrene **5a** (104.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 95:5, R_f = 0.45) to afford *cis/trans* mixture of **6a** as a colourless oil (95 mg, 92% yield; *trans/cis* = 2.57:1).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.43 – 7.24 (m, 7H, *trans/cis*), 7.22 – 7.00 (m, 3H, *trans/cis*), 4.11 (ddd, *J* = 5.6, 3.9, 2.2 Hz, 2H, *cis*), 3.73 – 3.61 (m, 2H, *trans*), 2.55 (ddd, *J* = 6.5, 4.2, 2.6 Hz, 2H, *trans/cis*), 2.47 – 2.33 (m, 4H, *trans/cis*), 2.31 – 2.16 (m, 4H, *trans/cis*); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 144.71 (*trans*), 141.62 (*cis*), 128.47 (*trans*), 128.08 (*cis*), 127.80 (*cis*), 126.78 (*trans*), 126.25 (*trans*), 125.67 (*cis*), 48.02 (*trans*), 45.41 (*cis*), 26.10 (*trans*), 24.34 (*cis*); **IR (neat, cm^{-1}):** 3059, 3026, 2940, 2866, 1685, 1600, 1493, 1446, 1028, 749, 694; **EI-MS:** exact m/z calculated for $C_{16}H_{16}$ $(M)^+$: 208.12465; Found: 208.12450 $(M)^+$.

1,2-bis(4-nitrophenyl)cyclobutane (6b):

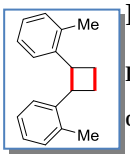


Following **GP-1**, **6b** was prepared from 1-nitro-4-vinylbenzene **5b** (149.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 9:1, R_f = 0.26) to afford *cis/trans* mixture of **6b** as a pale yellow solid (128 mg, 86% yield; *trans/cis* = 5.19:1).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 8.21 – 8.11 (m, 4H, *trans*), 8.01 – 7.90 (m, 4H, *cis*), 7.42 – 7.31 (m, 4H, *trans*), 7.14 – 7.05 (m, 4H, *cis*), 4.26 – 4.17 (m, 2H, *cis*), 3.77 – 3.62 (m, 2H, *trans*), 2.54 – 2.36 (m, 4H, *trans/cis*), 2.34 – 2.15 (m, 4H, *trans/cis*); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 151.05 (*trans*), 146.84 (*cis*), 128.56 (*cis*), 127.52 (*trans*), 124.06 (*trans*), 123.48 (*cis*), 47.82 (*trans*), 45.22 (*cis*), 25.92 (*trans*), 24.14 (*cis*).

The obtained data is in accordance with literature data.[1]

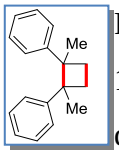
1,2-di-*o*-tolylcyclobutane (6c):



Following **GP-1**, **6c** was prepared from 1-methyl-2-vinylbenzene **5c** (118.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF_3)ppy}$_2$(dtb-bpy)]PF_6 (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 95:5, R_f = 0.43) to afford *cis/trans* mixture of **6c** as a colourless liquid (104 mg, 88% yield; *trans/cis* = 2.44:1).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.28 – 6.93 (m, 10H, *trans/cis*), 4.26 – 4.17 (m, 2H, *cis*), 3.97 – 3.85 (m, 2H, *trans*), 2.55 – 2.35 (m, 4H, *trans*), 2.30 (s, 3H, *trans*), 2.10 (s, 3H, *cis*), 1.96 (dd, *J* = 13.7, 9.1 Hz, 1H, *cis*); **^{13}C-NMR (75 MHz, $CDCl_3$):** δ 142.35 (*trans*), 139.80 (*cis*), 136.38 (*cis*), 136.08 (*trans*), 130.17 (*trans*), 129.86 (*cis*), 126.83 (*cis*), 126.14 (*trans*), 126.09 (*trans*), 125.98 (*trans*), 125.86 (*cis*), 125.43 (*cis*), 43.33 (*trans*), 41.67 (*cis*), 27.09 (*trans*), 24.97 (*cis*), 19.96 (*trans*), 19.83 (*cis*); **IR (neat, cm^{-1}):** 3019, 2965, 2943, 1686, 1602, 1488, 1458, 1378, 1031, 734; **EI-MS:** exact m/z calculated for $C_{18}H_{20}$ $(M)^+$: 236.15595; Found: 236.15646 $(M)^+$.

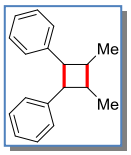
1,2-dimethyl-1,2-diphenylcyclobutane (6d):



Following **GP-1**, **6d** was prepared from prop-1-en-2-ylbenzene **5d** (118.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 95:05, R_f = 0.57) to afford *cis/trans* mixture of **6d** as a colourless oil (99 mg, 84% yield; *trans/cis* = 3.96:1).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.42 – 7.36 (m, 6H, *trans*), 7.36 – 7.16 (m, 4H, *trans*), 7.03 (d, *J* = 4.3 Hz, 6H, *cis*), 6.97 – 6.89 (m, 4H, *cis*), 2.90 – 2.73 (m, 2H, *trans*), 2.68 (dd, *J* = 11.8, 6.3 Hz, 2H, *cis*), 2.15 – 2.01 (m, 2H, *cis*), 1.85 – 1.70 (m, 2H, *trans*), 1.65 (s, 3H, *cis*), 1.17 (s, 3H, *trans*); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 147.44 (*trans*), 147.25 (*cis*), 128.20 (*trans*), 127.45 (*cis*), 126.79 (*trans*), 126.59 (*cis*), 125.81 (*trans*), 125.13 (*cis*), 49.25 (*trans*), 37.00 (*cis*), 29.78 (*cis*), 27.33 (*trans*), 27.00 (*trans*), 26.28 (*cis*); **IR (neat, cm^{-1}):** 2926, 1457, 1421, 1263, 1094, 1029, 895, 804, 733, 703; **EI-MS:** exact m/z calculated for $C_{18}H_{20}$ $(M)^+$: 236.15595; Found: 236.15563 $(M)^+$.

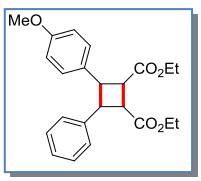
3,4-dimethylcyclobutane-1,2-diyl)dibenzene (6e):



Following **GP-1**, **6e** was prepared from (*E*)-prop-1-en-1-ylbenzene **5e** (118.1 mg, 1.0 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.01 equiv, 1.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 95:05, R_f = 0.59) to afford *cis/trans* mixture of **6e** as a colourless oil (94 mg, 80% yield; *trans/cis* = 3.38:1).

^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.39 – 7.13 (m, 10H, *trans*), 7.13 – 6.90 (m, 10H, *cis*), 3.56 – 3.52 (m, 2H, *cis*), 2.99 – 2.90 (m, 2H, *trans*), 1.91 (td, *J* = 9.4, 3.9 Hz, 2H, *cis*), 1.55 (s, 3H, *cis*), 1.26 (s, 3H, *trans*), 1.23 – 1.18 (m, 2H, *trans*); **^{13}C-NMR (75 MHz, CDCl$_3$):** δ 143.86 (*trans*), 128.48 (*trans*), 128.22 (*cis*), 127.83 (*cis*), 127.00 (*trans*), 126.24 (*trans*), 125.58 (*cis*), 52.83 (*trans*), 43.32 (*trans*), 19.15 (*trans*); **IR (neat, cm^{-1}):** 3058, 2955, 2923, 2861, 1494, 1453, 1261, 1094, 1029, 803, 749, 698; **EI-MS:** exact m/z calculated for $C_{18}H_{20}$ $(M)^+$: 236.15595; Found: 236.15556 $(M)^+$.

Diethyl 3-(4-methoxyphenyl)-4-phenylcyclobutane-1,2-dicarboxylate (7a):

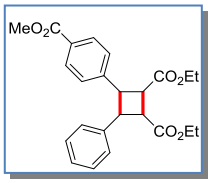


Following **GP-1**, **7a** was prepared from ethyl cinnamate **1a** (88.1 mg, 0.5 mmol, 1.00 equiv), ethyl-3-(4-methoxyphenyl)acrylate **1f** (103.1 mg, 0.5 mmol, 1.00 equiv) and [Ir{dF(CF$_3$)ppy}$_2$(dtb-bpy)]PF$_6$ (11.22 mg, 0.02 equiv, 2.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.3) to afford the desired product **7a** as colorless oil (67

mg, 35% yield), product **2f** as yellow oil (46 mg, 24% yield) and product **2a** (21 mg, 11% yield) as colorless oil.

^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.26 (m, 5H), 7.26 – 7.21 (m, 3H), 6.89 – 6.83 (m, 2H), 4.19 (qt, *J* = 10.8, 5.4 Hz, 4H), 3.79 (s, 3H), 3.76 – 3.63 (m, 2H), 3.47 – 3.35 (m, 2H), 1.27 (td, *J* = 7.1, 3.3 Hz, 6H); **^{13}C-NMR (101 MHz, $CDCl_3$):** δ 172.70, 172.63, 158.71, 141.27, 133.33, 128.59, 128.01, 127.01, 126.81, 114.02, 77.37, 77.05, 76.73, 61.00, 60.96, 55.30, 47.27, 46.67, 45.27, 44.60, 14.26; **IR (neat, cm^{-1}):** 2982, 1722, 1610, 1513, 1457, 1249, 1178, 1033, 828, 701; **HRMS (ESI):** exact m/z calculated for $C_{23}H_{26}O_5$ $(M+H)^+$: 383.1859; Found: 383.1586 $(M+H)^+$.

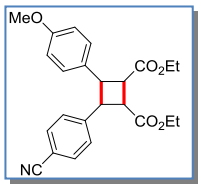
Diethyl 3-(4-(methoxycarbonyl)phenyl)-4-phenylcyclobutane-1,2-dicarboxylate (7b):



Following **GP-1**, **7b** was prepared from ethyl cinnamate **1a** (88.1 mg, 0.5 mmol, 1.00 equiv), methyl-4-(3-ethoxy-3-oxoprop-1-en-1-yl)benzoate **1j** (117.1 mg, 0.5 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.02 equiv, 2.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.4-0.1) to afford the desired product **7b** (76 mg, 32% yield, 1.9:1 d.r.) and product **2j** (25 mg, 14% yield) as colorless oils.

^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.32 (ddd, *J* = 19.6, 15.8, 7.8 Hz, 7H), 4.26 – 4.16 (m, 4H), 3.90 (s, 3H), 3.84 – 3.71 (m, 2H), 3.51 – 3.41 (m, 2H), 1.33 – 1.22 (m, 6H); **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ 172.13, 166.79, 145.82, 130.08, 129.49, 129.22, 127.71, 126.85, 77.36, 77.04, 76.73, 61.29, 52.15, 52.03, 46.82, 44.90, 44.56, 43.24, 14.23; **IR (neat, cm^{-1}):** 2952, 1718, 1610, 1435, 1275, 1185, 1103, 1017, 965, 857, 767, 705; **HRMS (ESI):** exact m/z calculated for $C_{24}H_{26}O_6$ $(M+H)^+$: 411.1808; Found: 411.1807 $(M+H)^+$.

Diethyl 3-(4-cyanophenyl)-4-(4-methoxyphenyl)cyclobutane-1,2-dicarboxylate (7c):



Following **GP-1**, **7c** was prepared from ethyl-3-(4-methoxyphenyl)acrylate **1f** (103.1 mg, 0.5 mmol, 1.00 equiv), ethyl-3-(4-cyanophenyl)acrylate **1i** (100.6 mg, 0.5 mmol, 1.00 equiv) and $[Ir\{dF(CF_3)ppy\}_2(dtb\text{-}bpy)]PF_6$ (11.22 mg, 0.02 equiv, 2.0 mol %) in dry DMF (2 mL). The crude product was purified by column chromatography (silica gel, hexanes–EtOAc, 5:1, R_f = 0.4-0.1) to afford the desired product **7c** (111.3 mg, 54% yield, d.r. 1.75:1) and product **2f** (26.4 mg, 13% yield, 99:1 d.r.) as white solids and product **2i** (17 mg, 14% yield, 90:1 d.r.) as colorless oil; Mp = 54-56 °C (decomposed).

^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.63 – 7.55 (m, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.24 – 7.16 (m, 2H), 6.90 – 6.85 (m, 2H), 4.20 (p, *J* = 7.2 Hz, 4H), 3.80 (s, 3H), 3.77 – 3.56 (m, 2H), 3.44 – 3.38 (m, 2H), 1.27 (dd, *J* = 13.4, 7.1, 6H); **^{13}C-NMR (101 MHz, $CDCl_3$):** δ 172.3, 172.2, 159.0, 146.6, 132.5, 132.4, 127.9, 127.6, 118.8, 114.2, 110.9, 77.4, 77.1, 76.8, 61.3, 61.2, 55.3, 46.9,

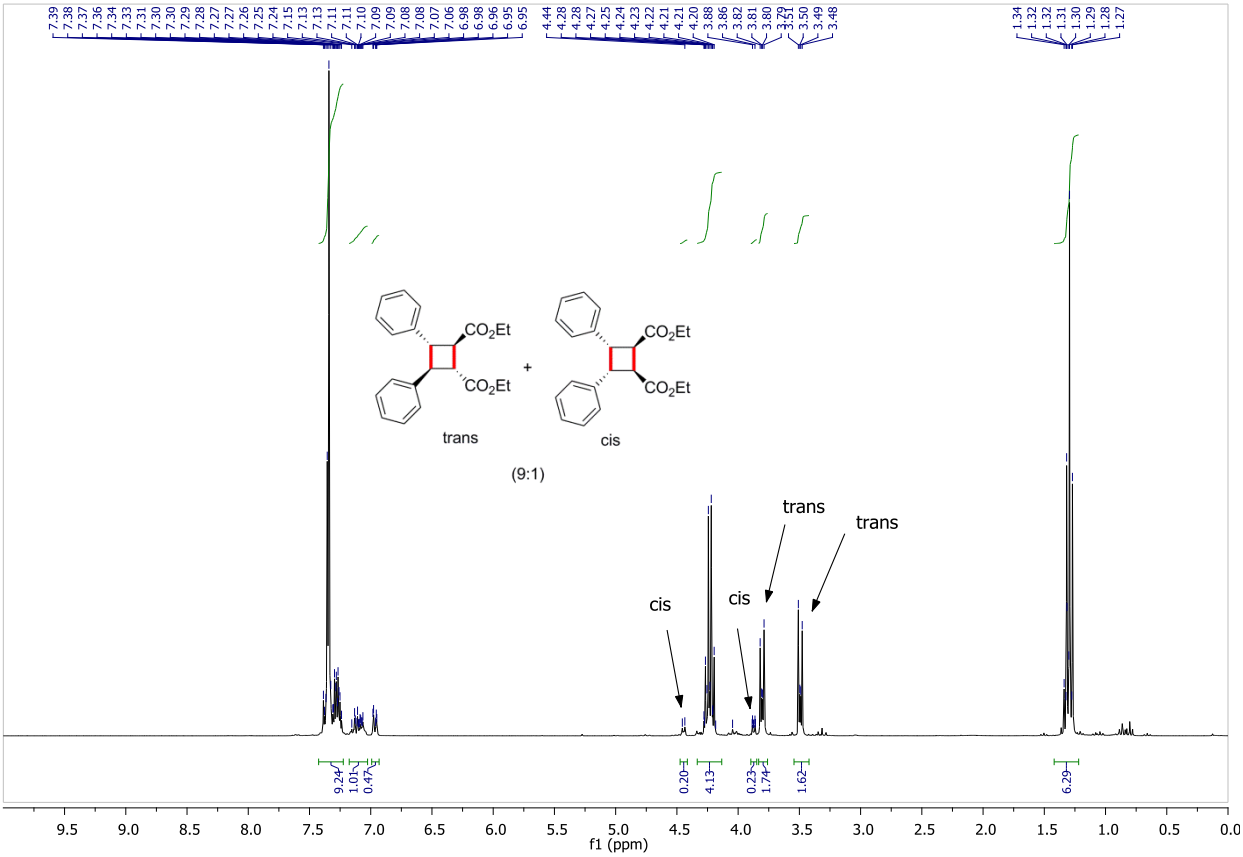
46.7, 45.4, 44.0, 14.2; **IR (neat, cm^{-1}):** 2981, 2937, 2840, 229, 1718, 1610, 1513, 1245, 1178, 114, 1036, 828; **HRMS (ESI):** exact m/z calculated for $C_{24}H_{25}NO_5$ $(M+H)^+$: 408.1811; Found: 408.1805 $(M+H)^+$.

3. References:

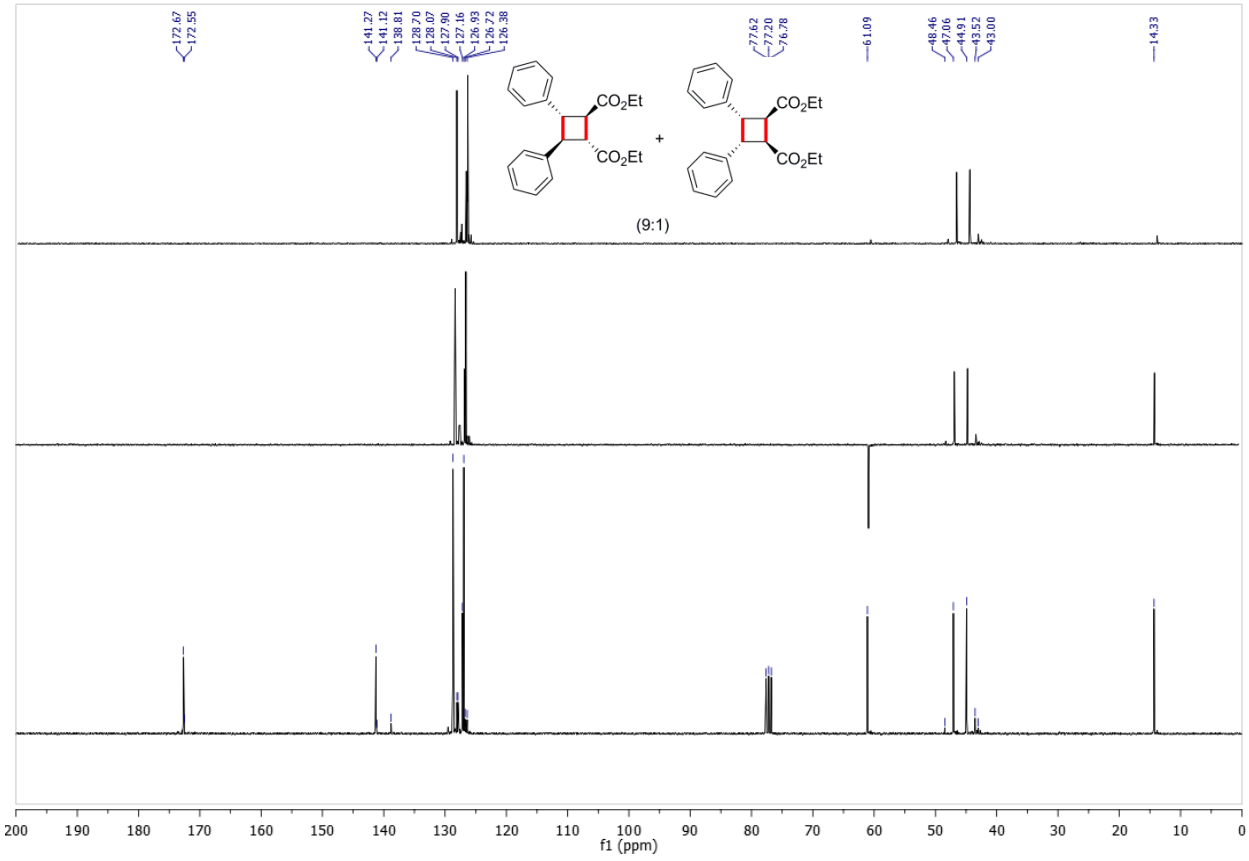
1) J. Herman, E. H. Freed and M. D. Swerdloff, *J. Org. Chem.*, 1971, **36**, 1302-1305

4. NMR spectra:

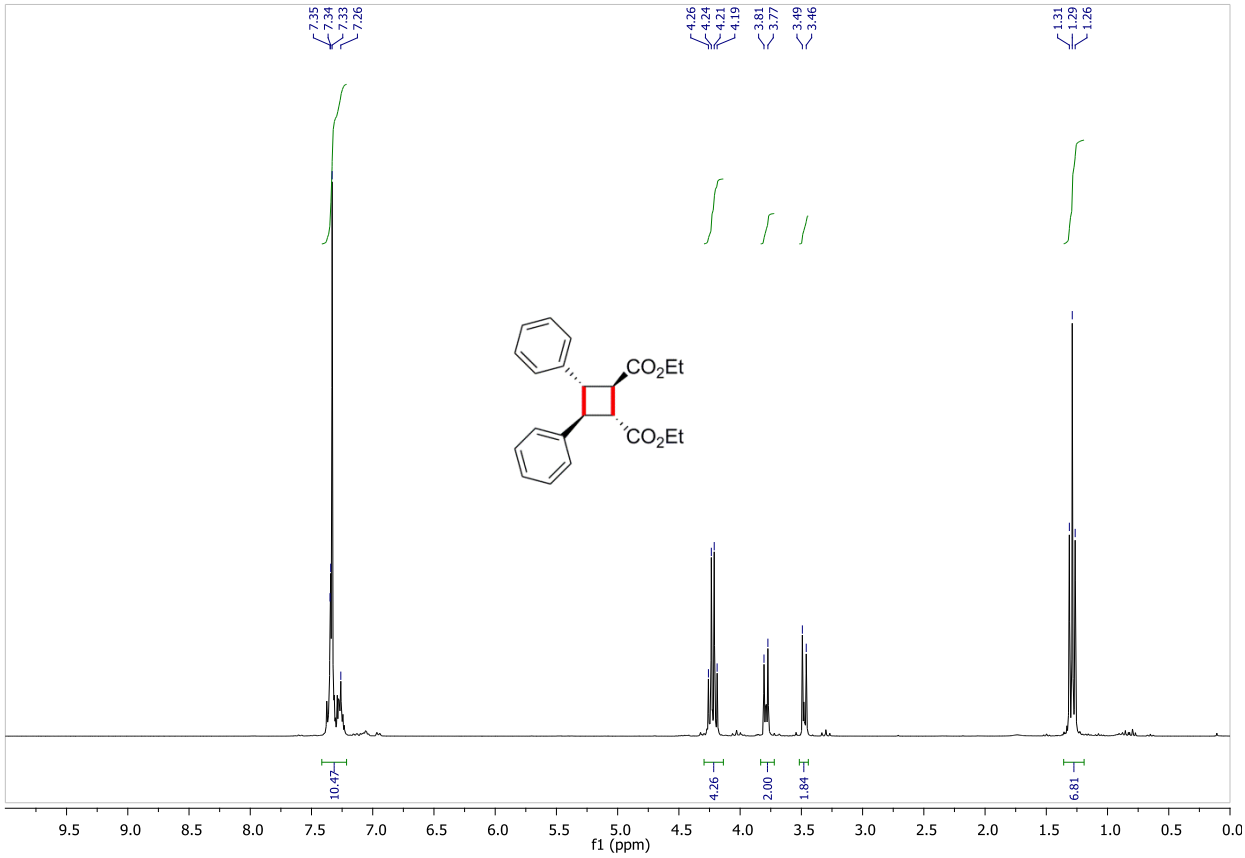
^{1}H-NMR: **2a/3a** (mixture, before separation)



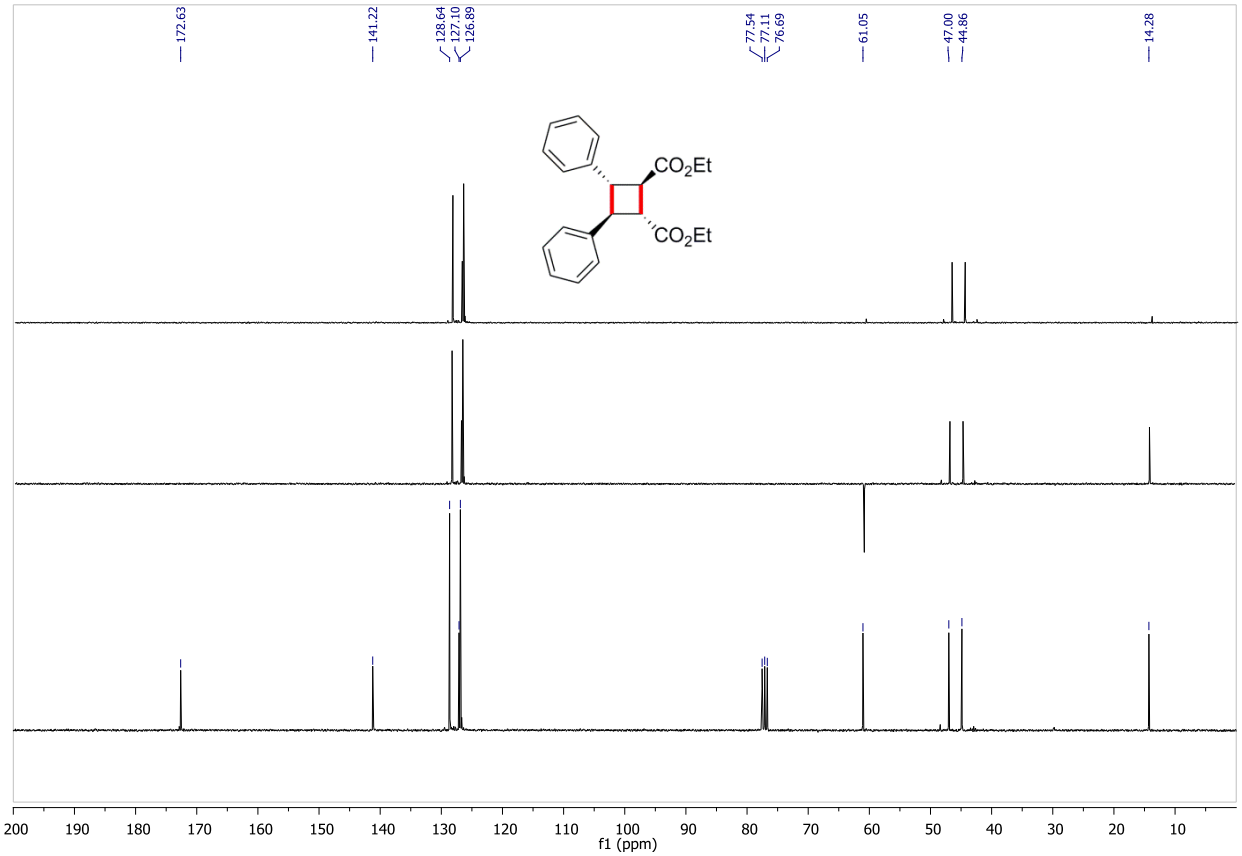
^{13}C-NMR: **2a/3a** (mixture, before separation)



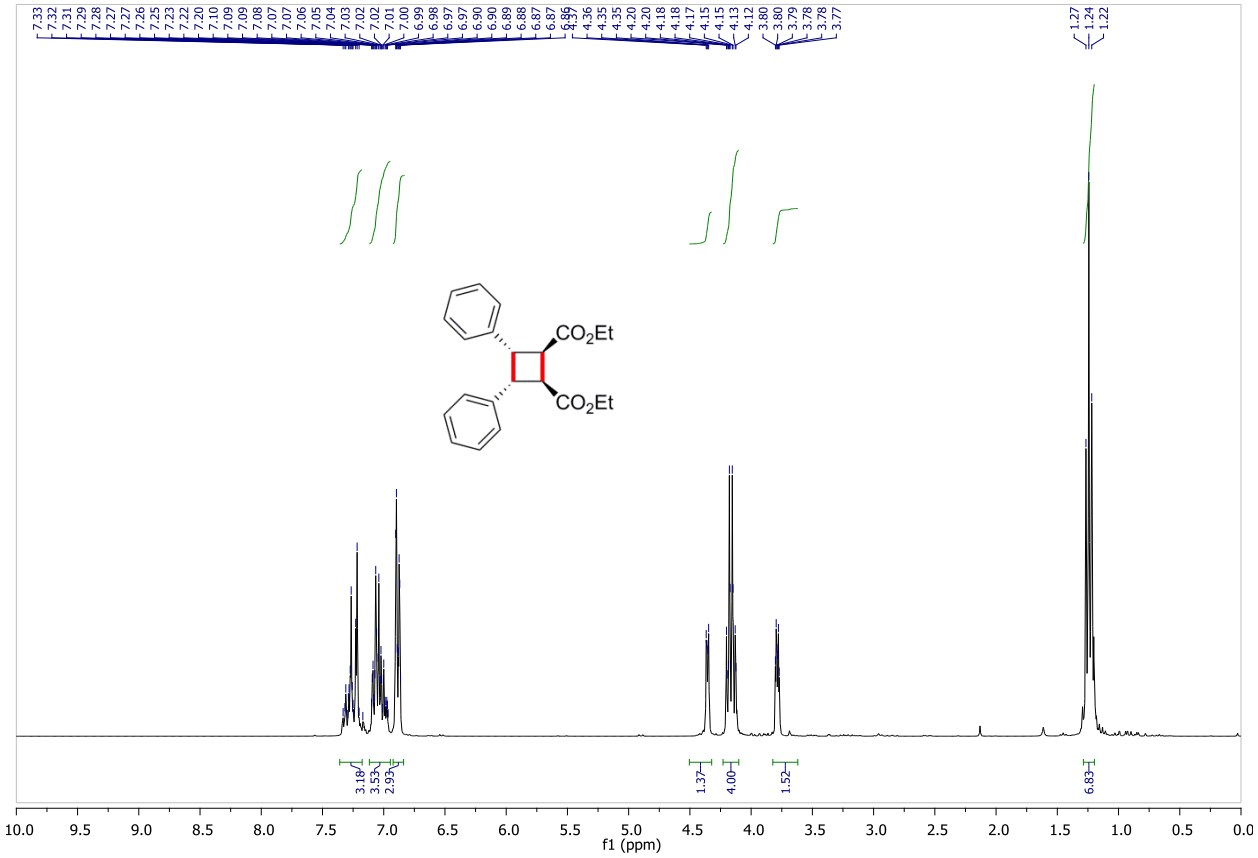
^{1}H-NMR: **2a** (*trans*, after separation)



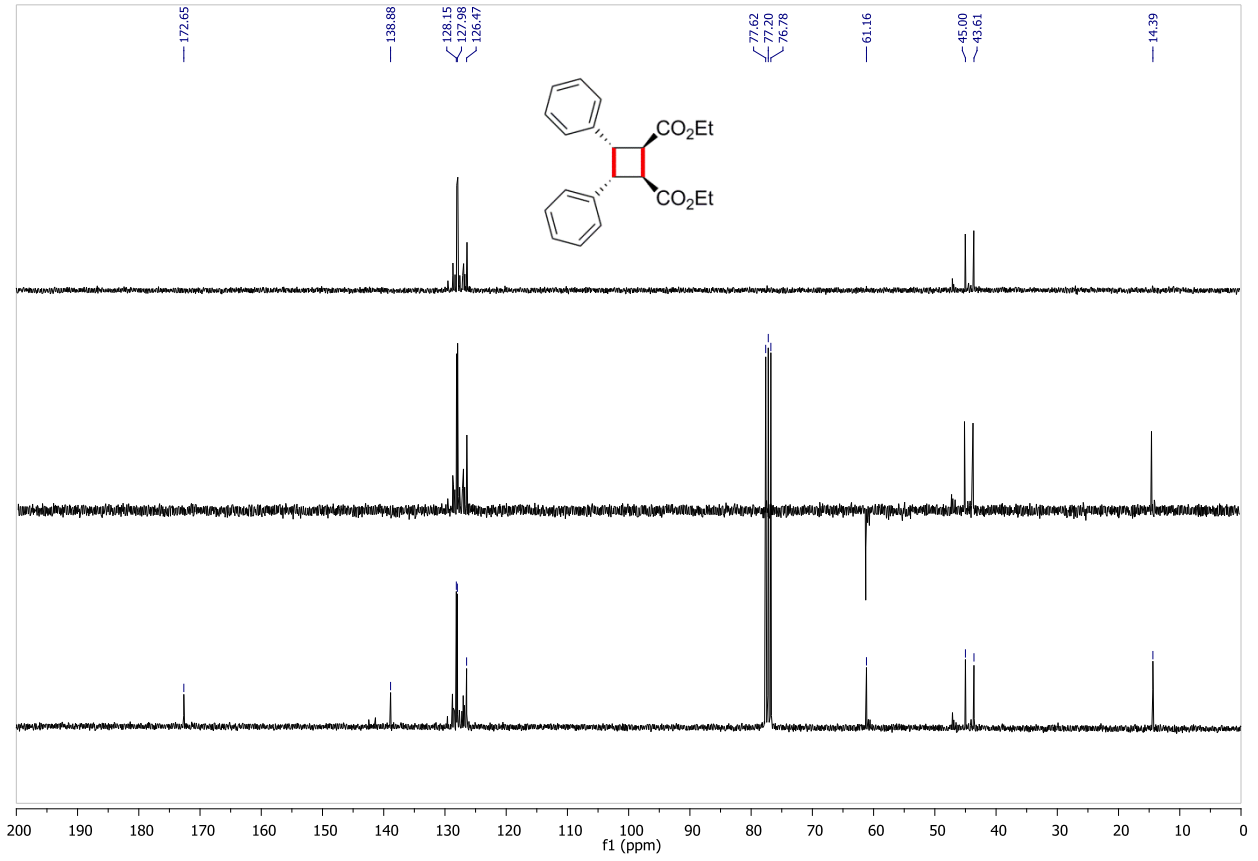
^{13}C-NMR: **2a** (*trans*, after separation)



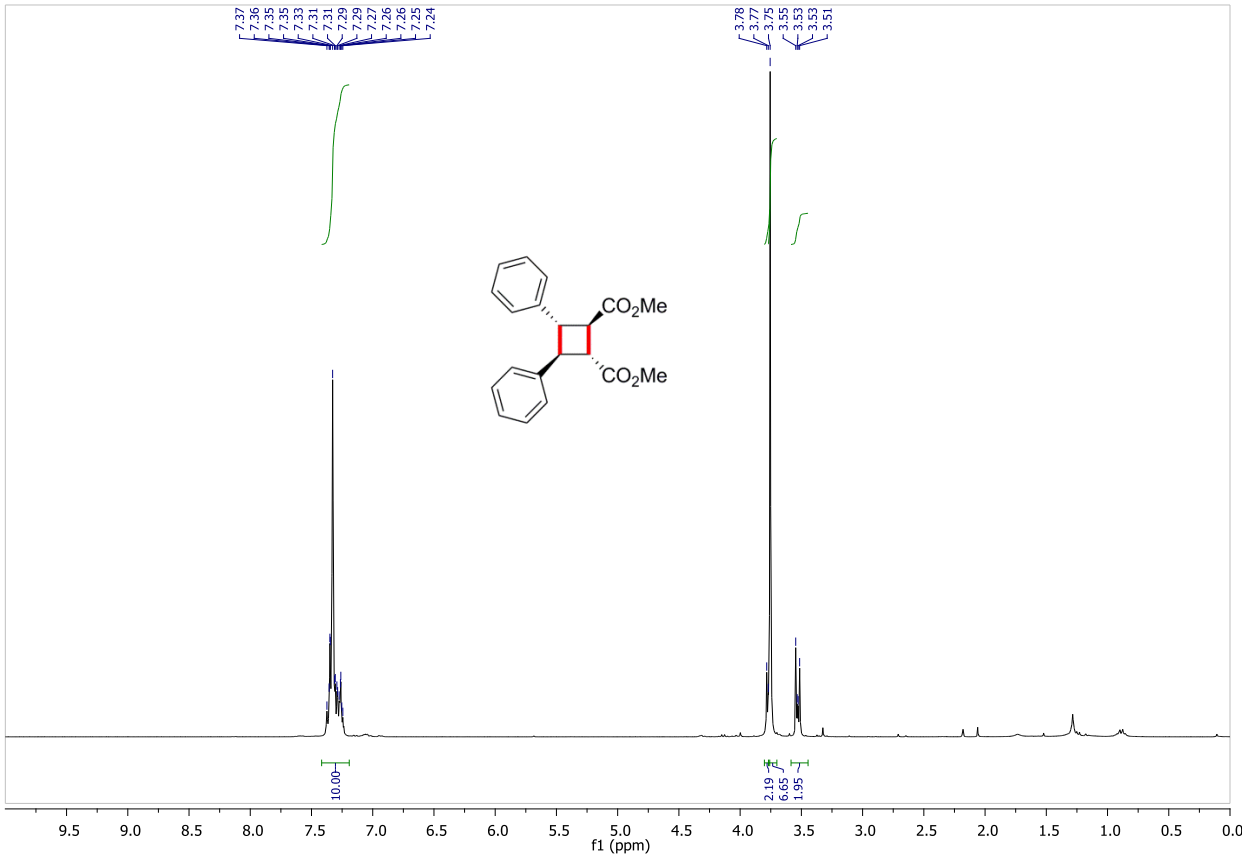
^{1}H-NMR: **3a** (*cis*, after separation)



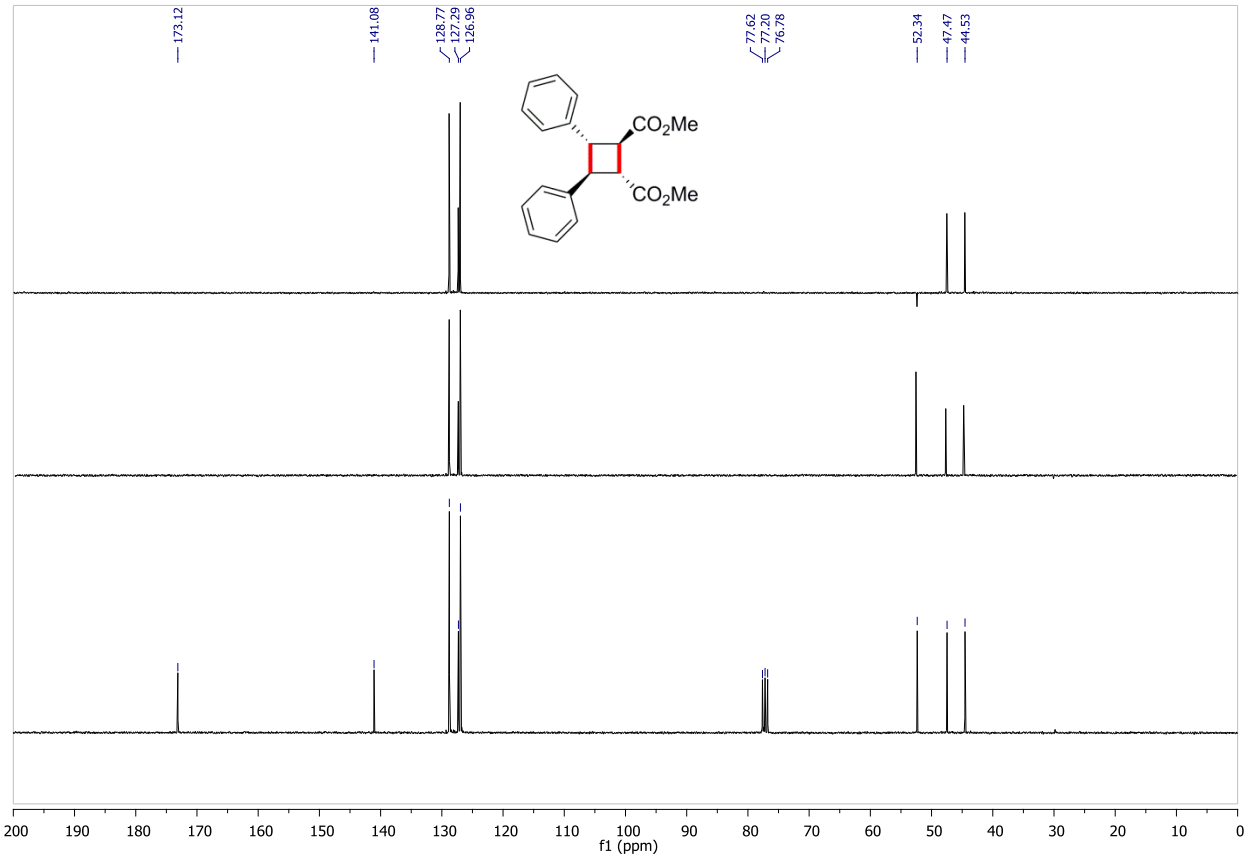
^{13}C-NMR: **3a** (*cis*, after separation)



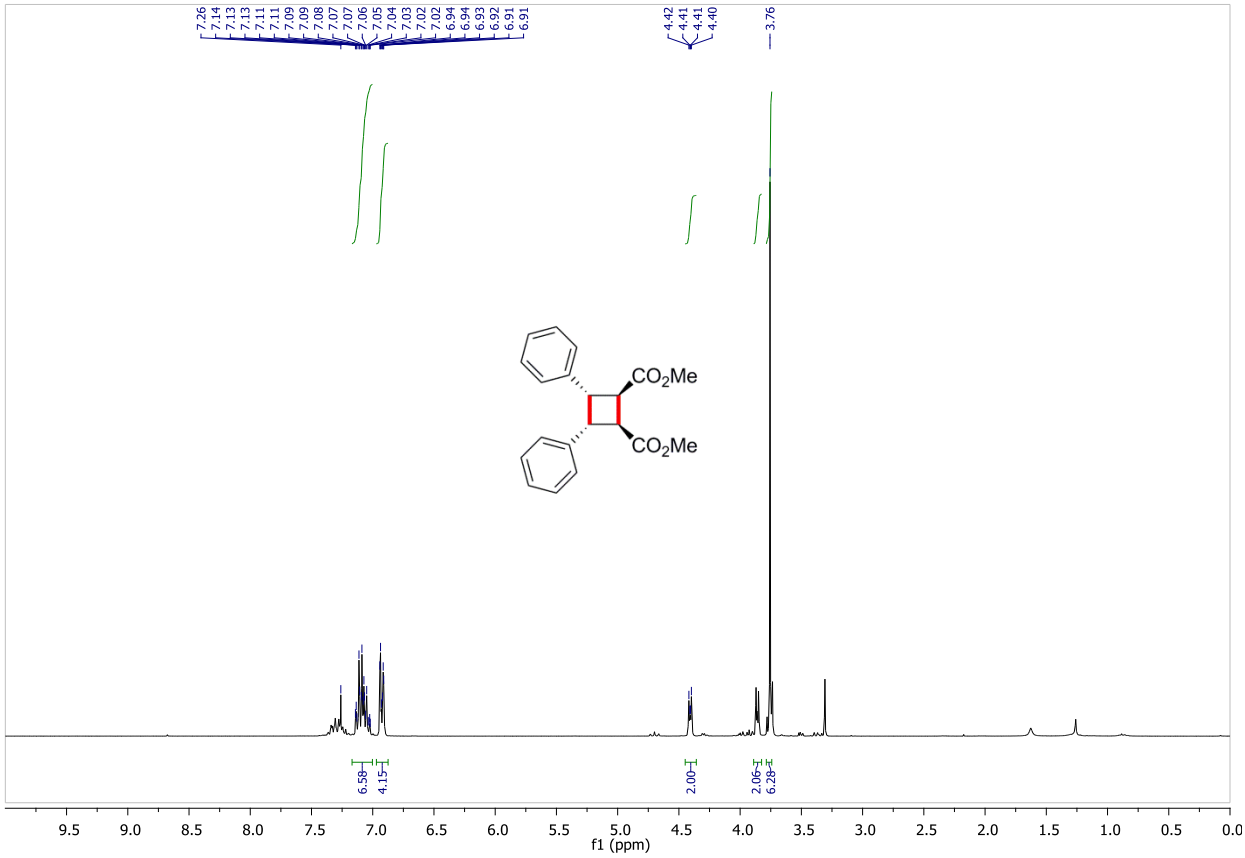
^{1}H-NMR: **2b** (*trans*, after separation)



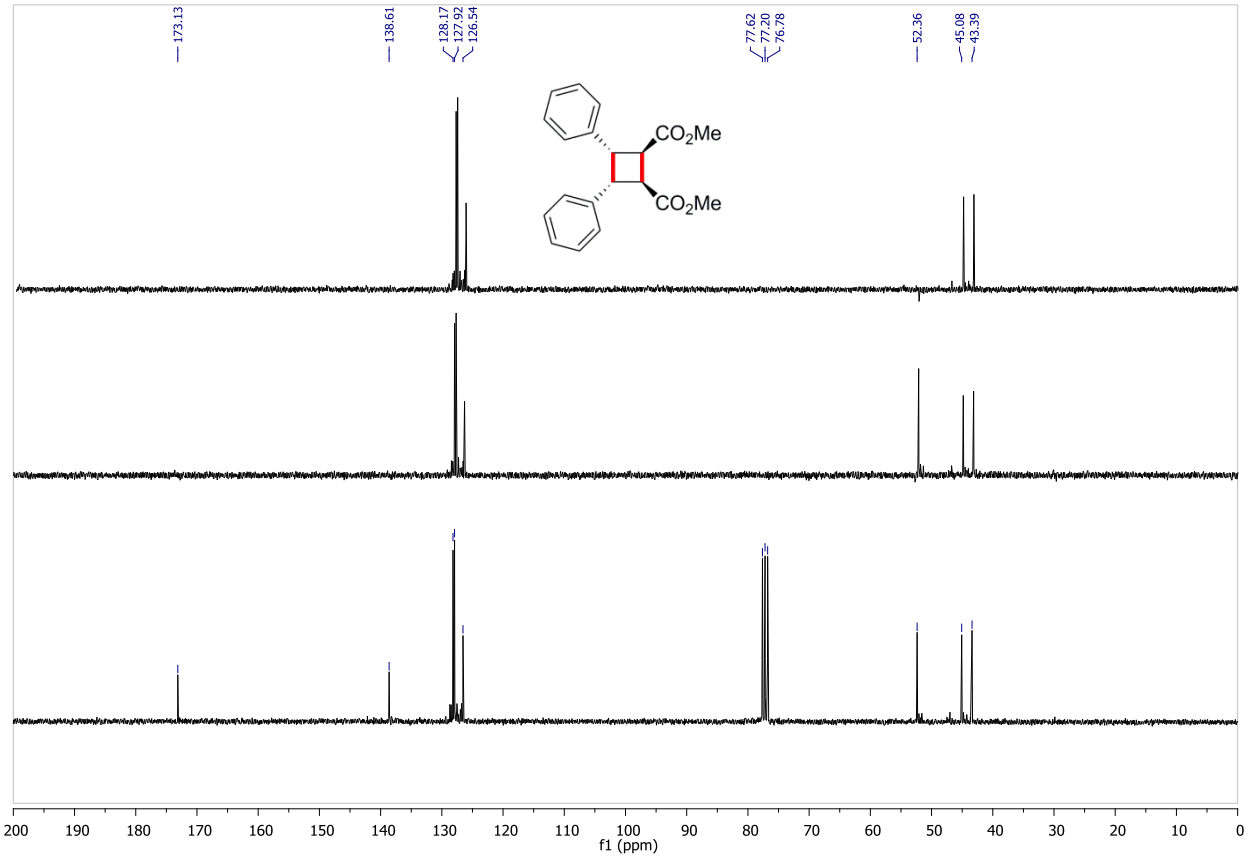
^{13}C-NMR: **2b** (*trans*, after separation)



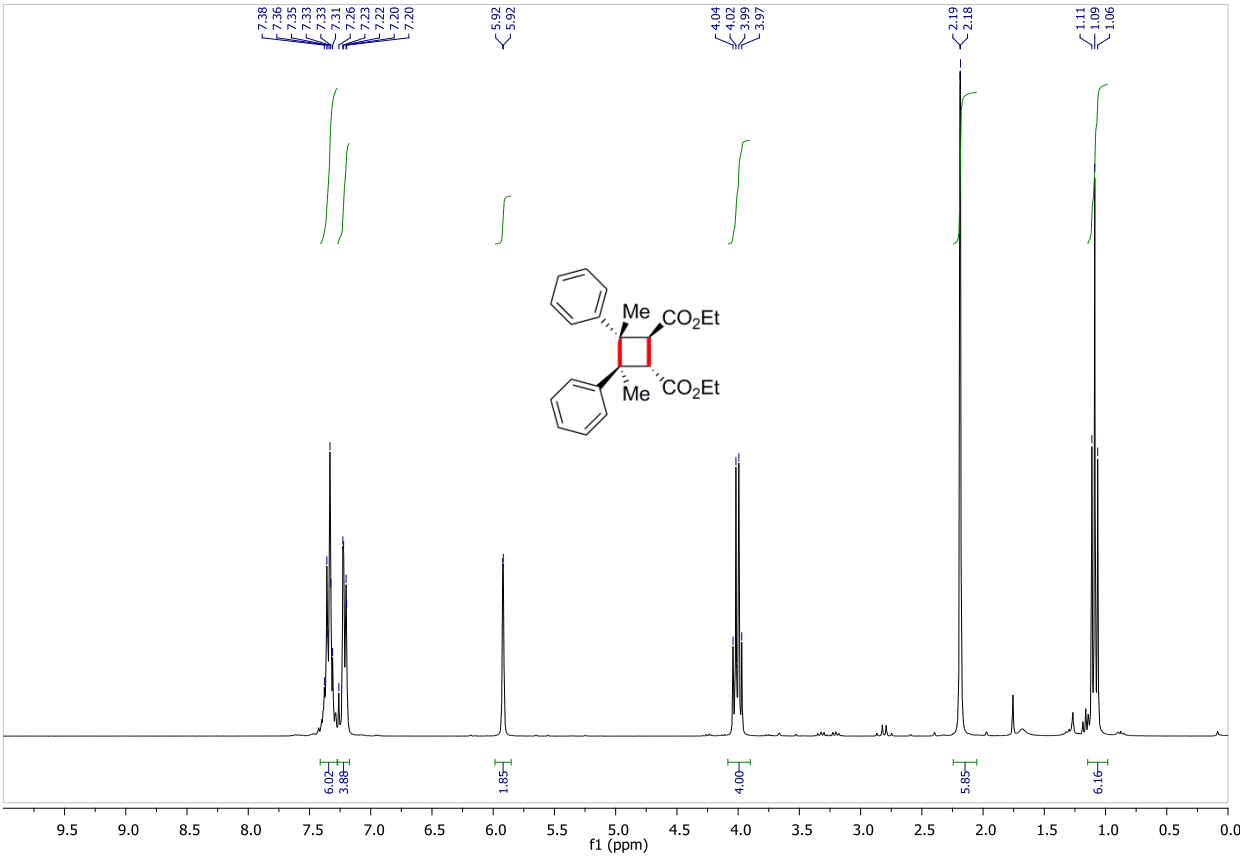
^{1}H-NMR: **3b** (*cis*, after separation)



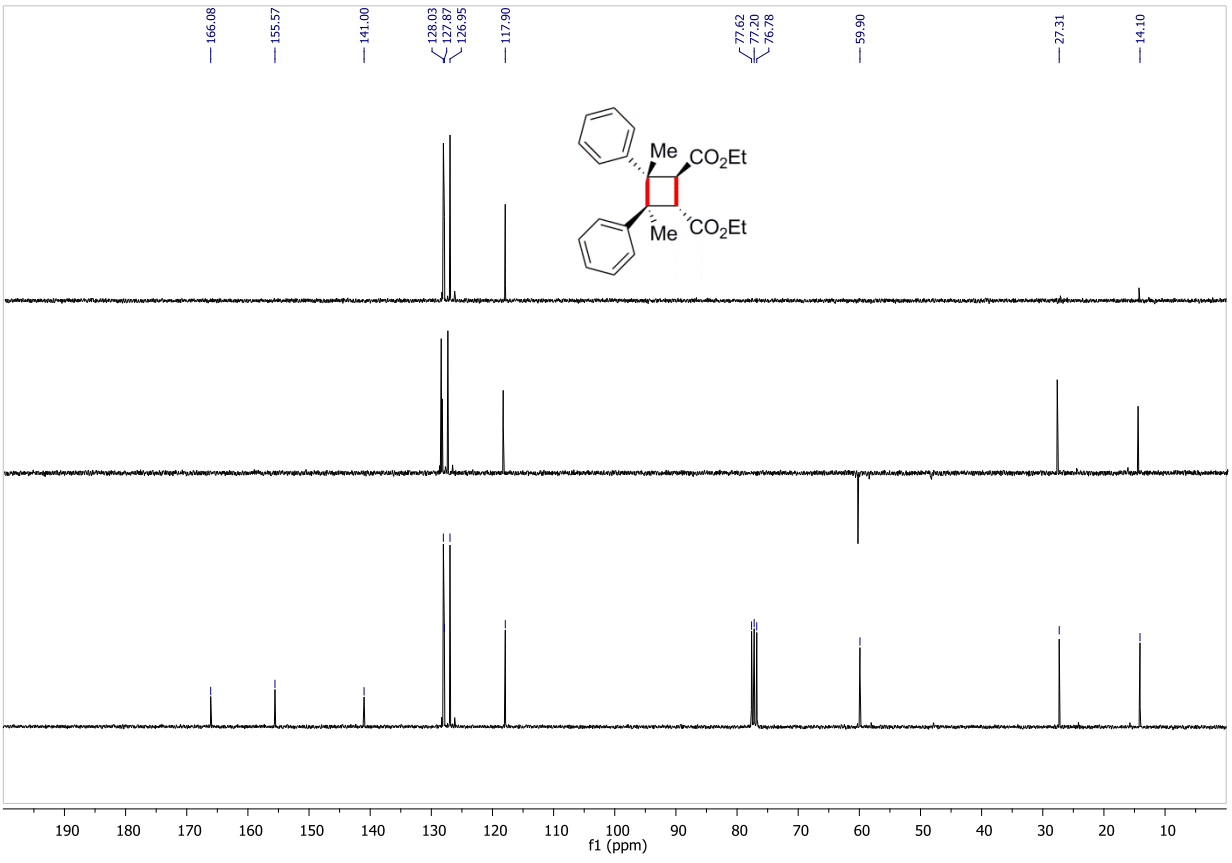
^{13}C-NMR: **3b** (*cis*, after separation)



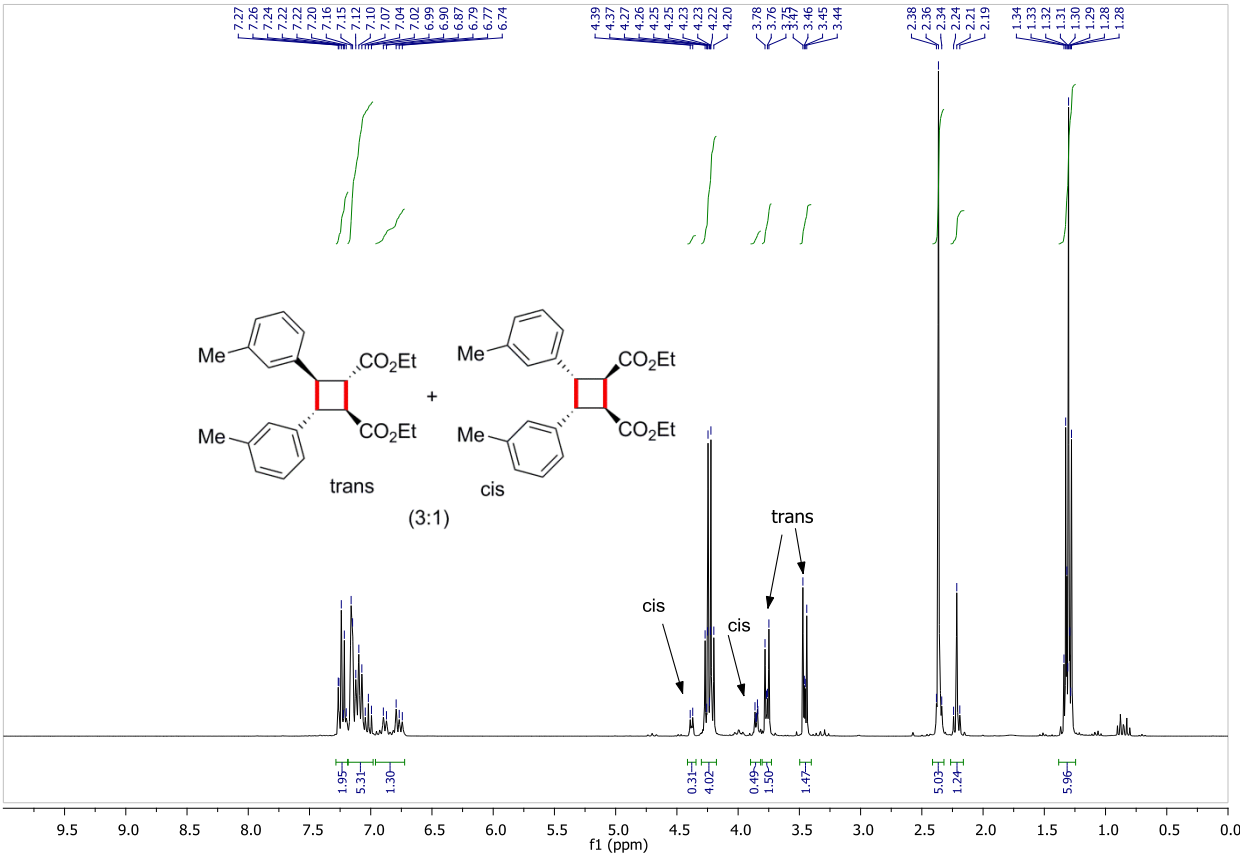
^{1}H-NMR: **2c** (*trans*, isomer only)



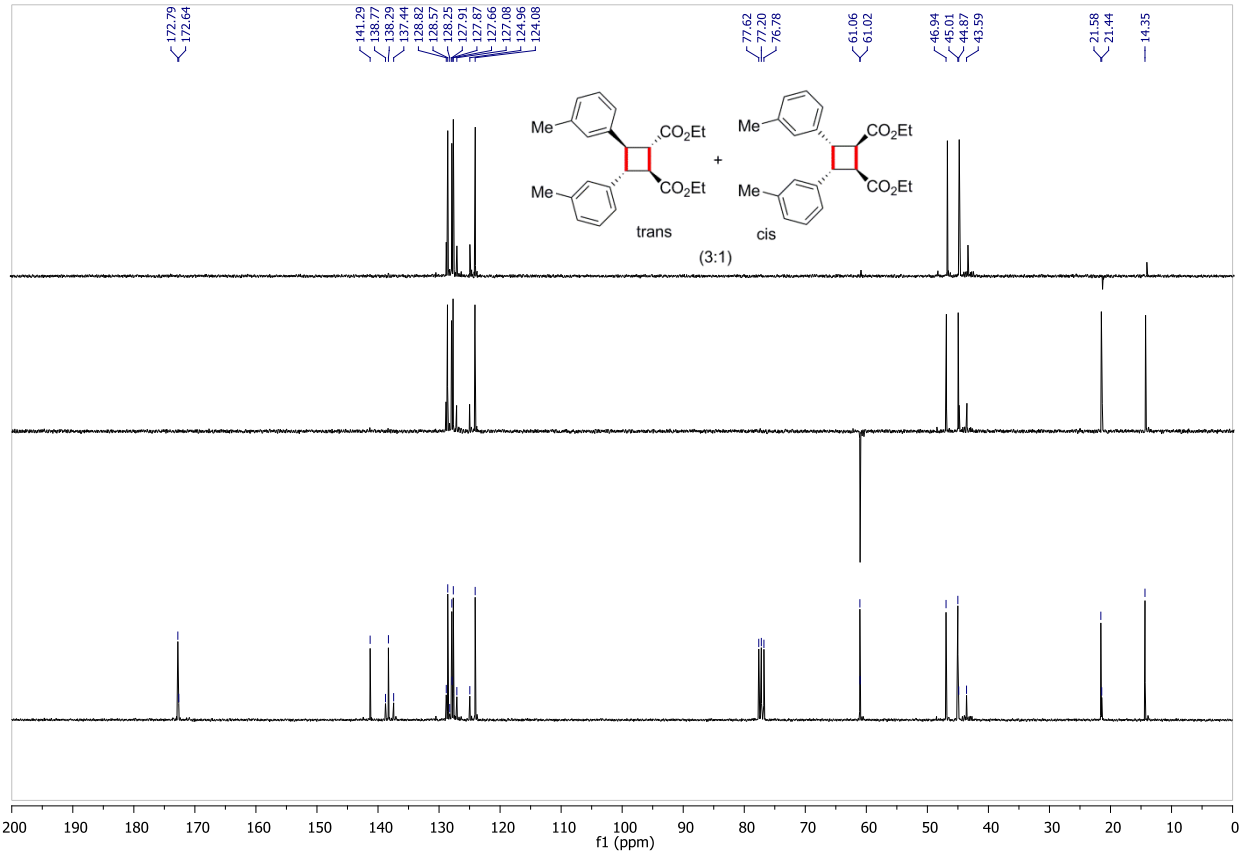
^{13}C-NMR: **2c** (*trans*, isomer only)



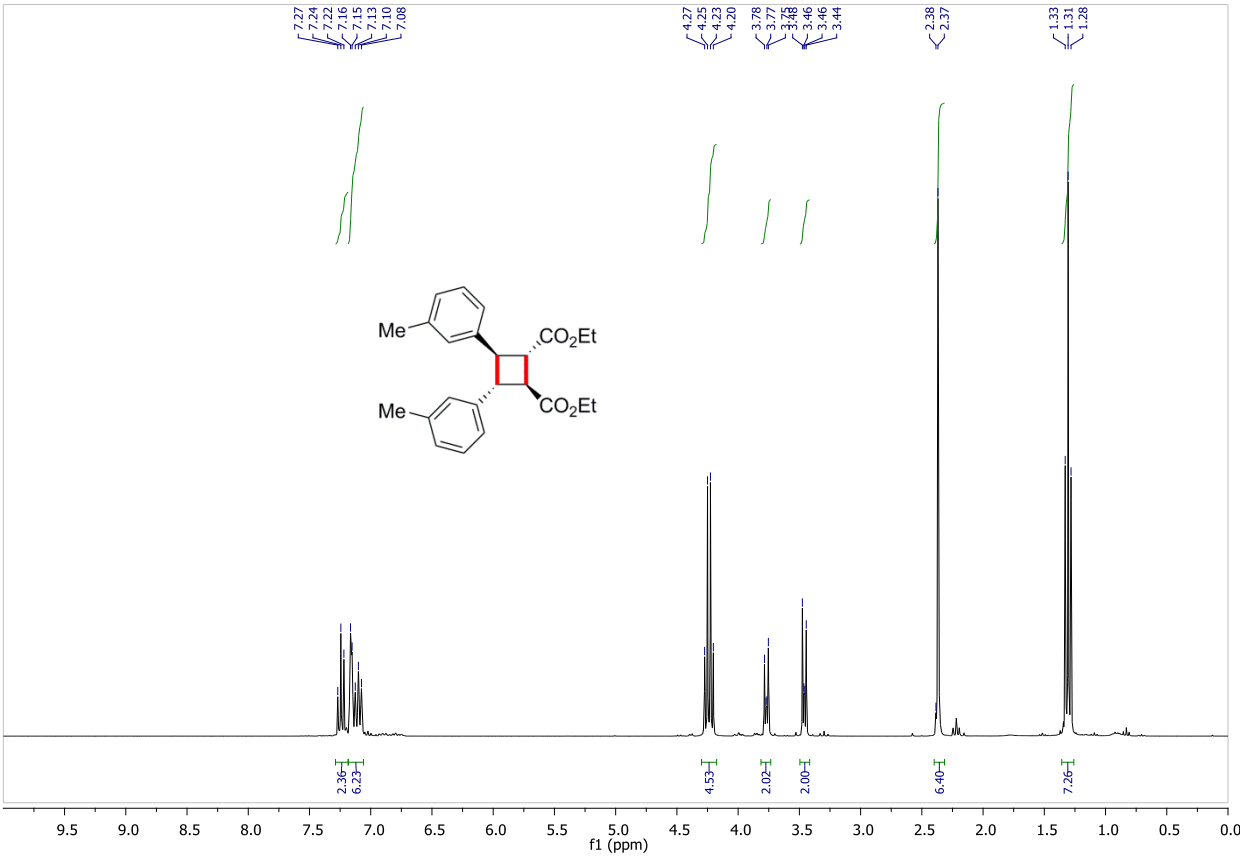
^{1}H-NMR: **2d/3d** (mixture, before separation)



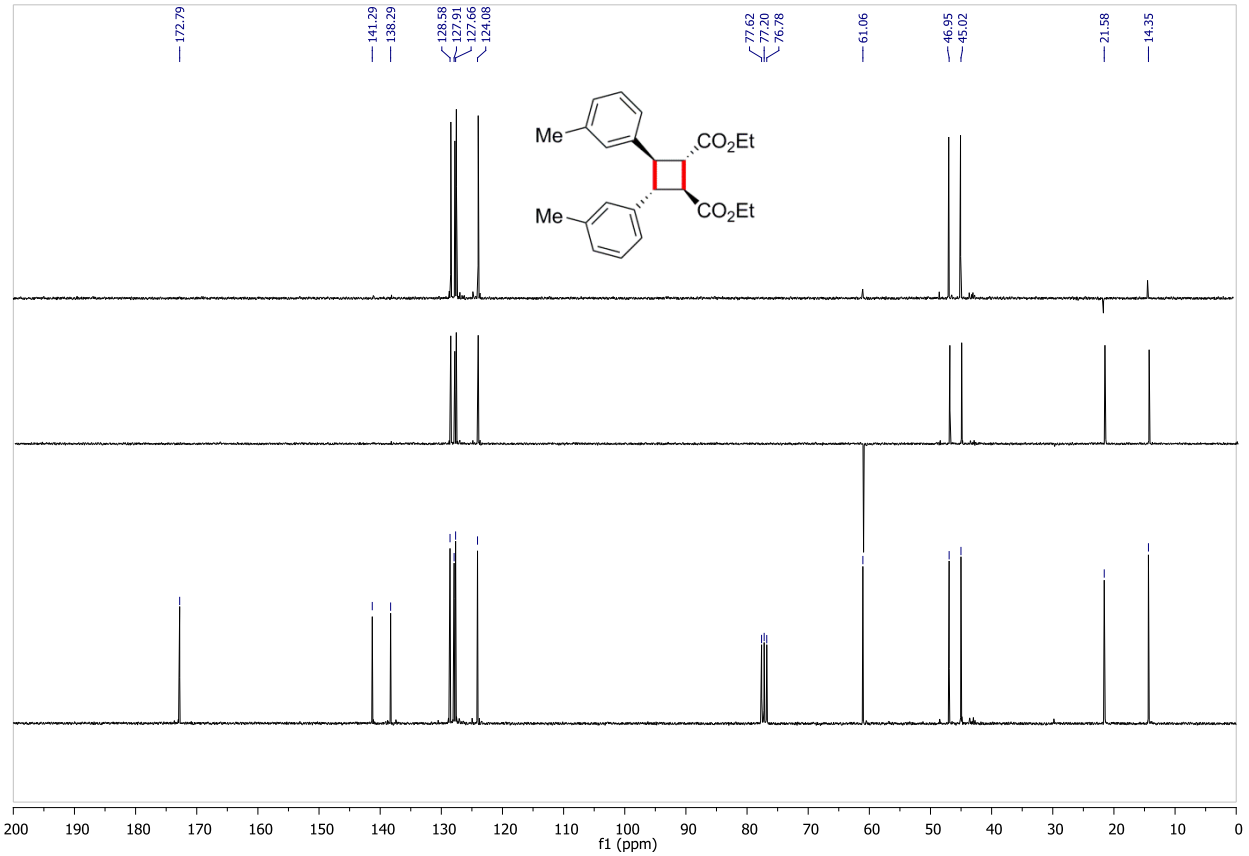
^{13}C-NMR: **2d/3d** (mixture, before separation)



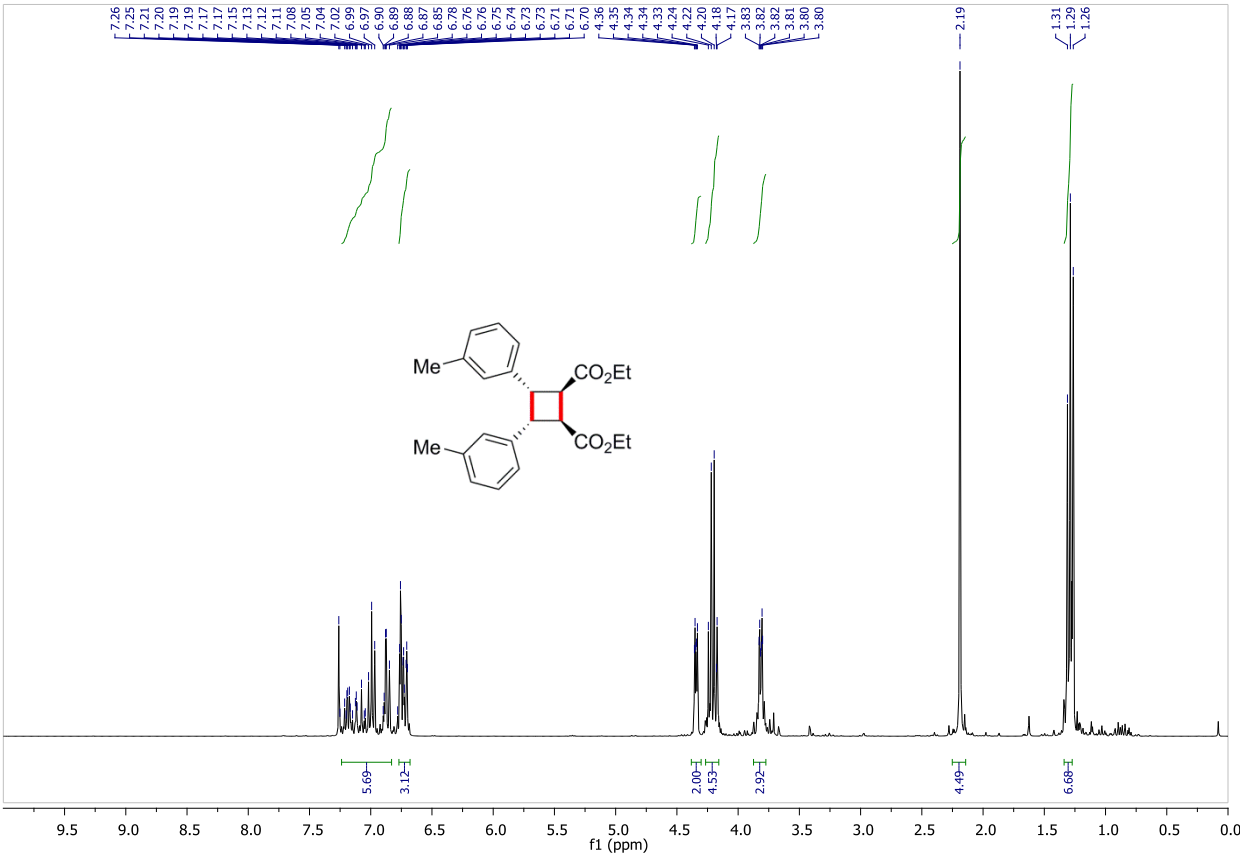
^{1}H-NMR: **2d** (*trans*, after separation)



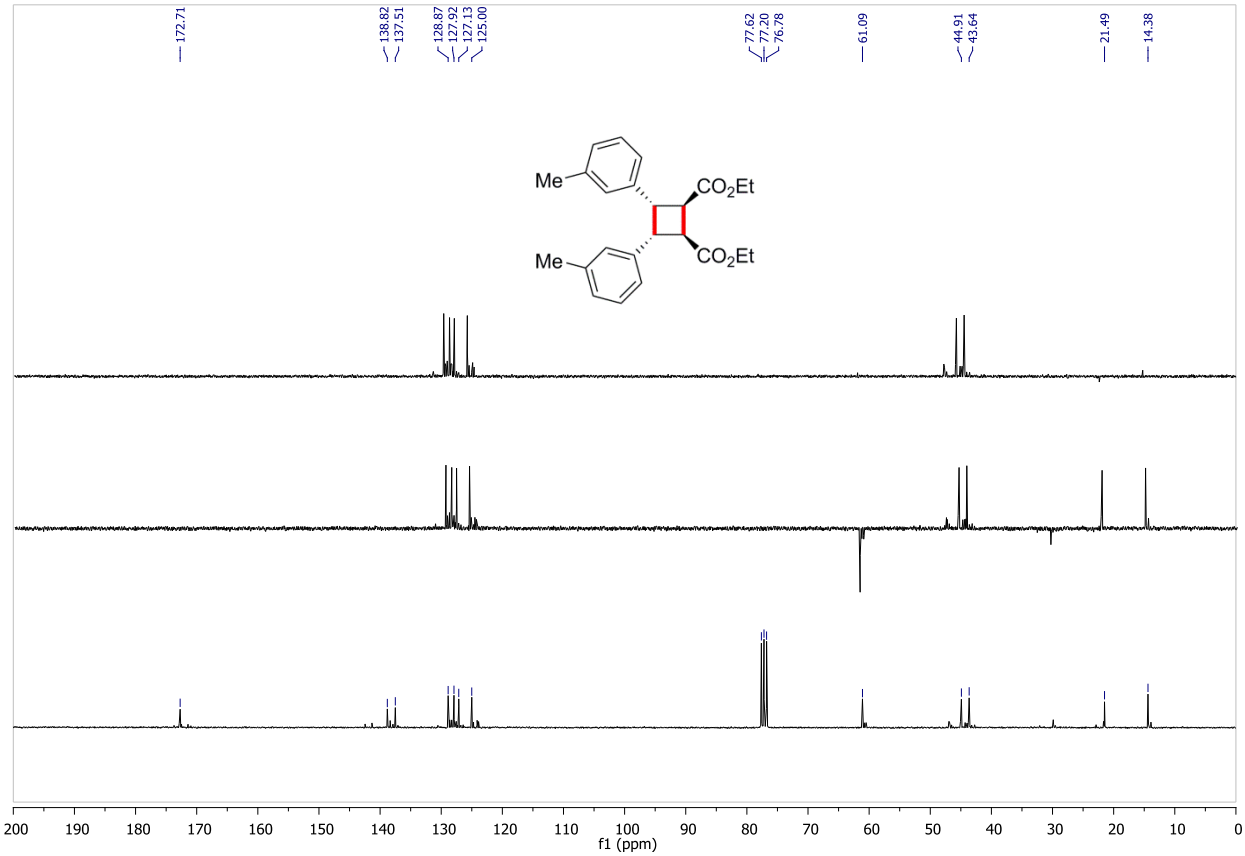
^{13}C-NMR: **2d** (*trans*, after separation)



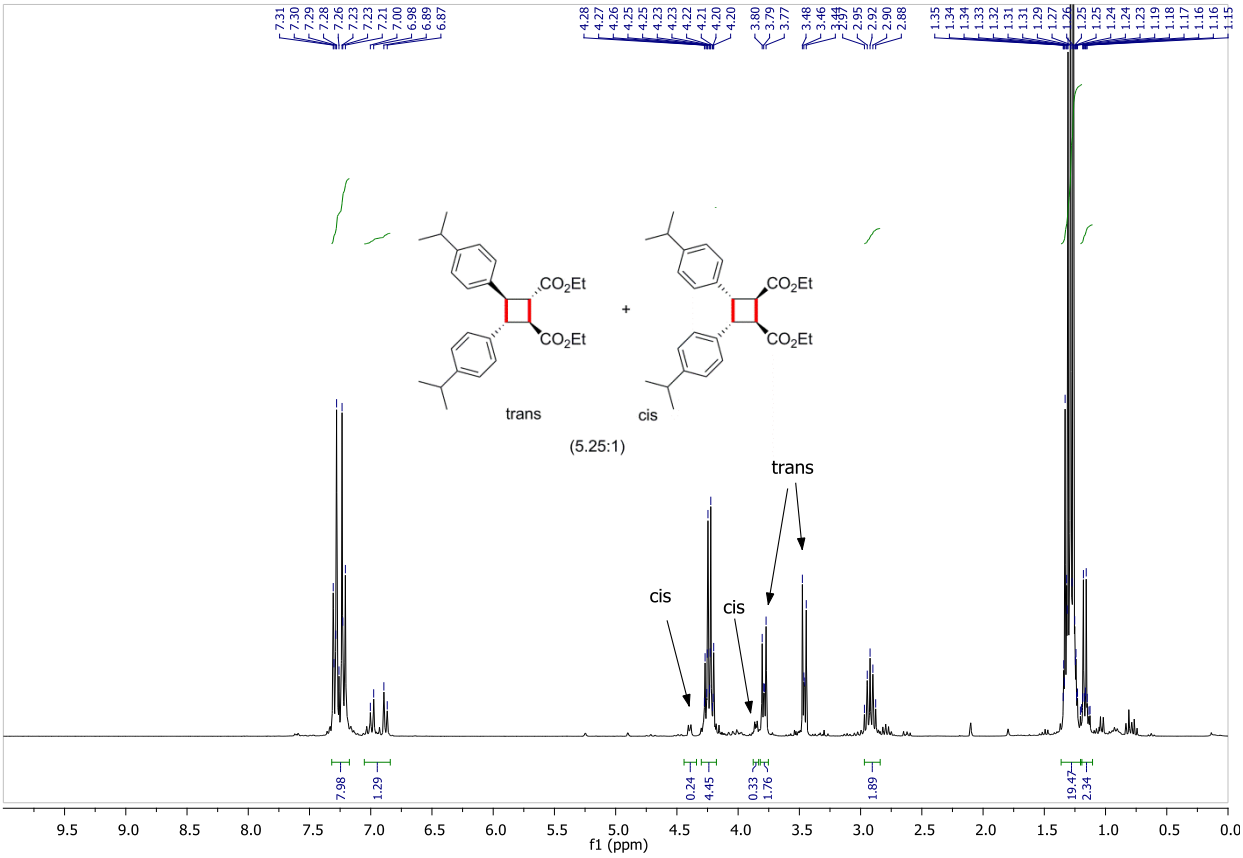
^{1}H-NMR: **3d** (*cis*, after separation)



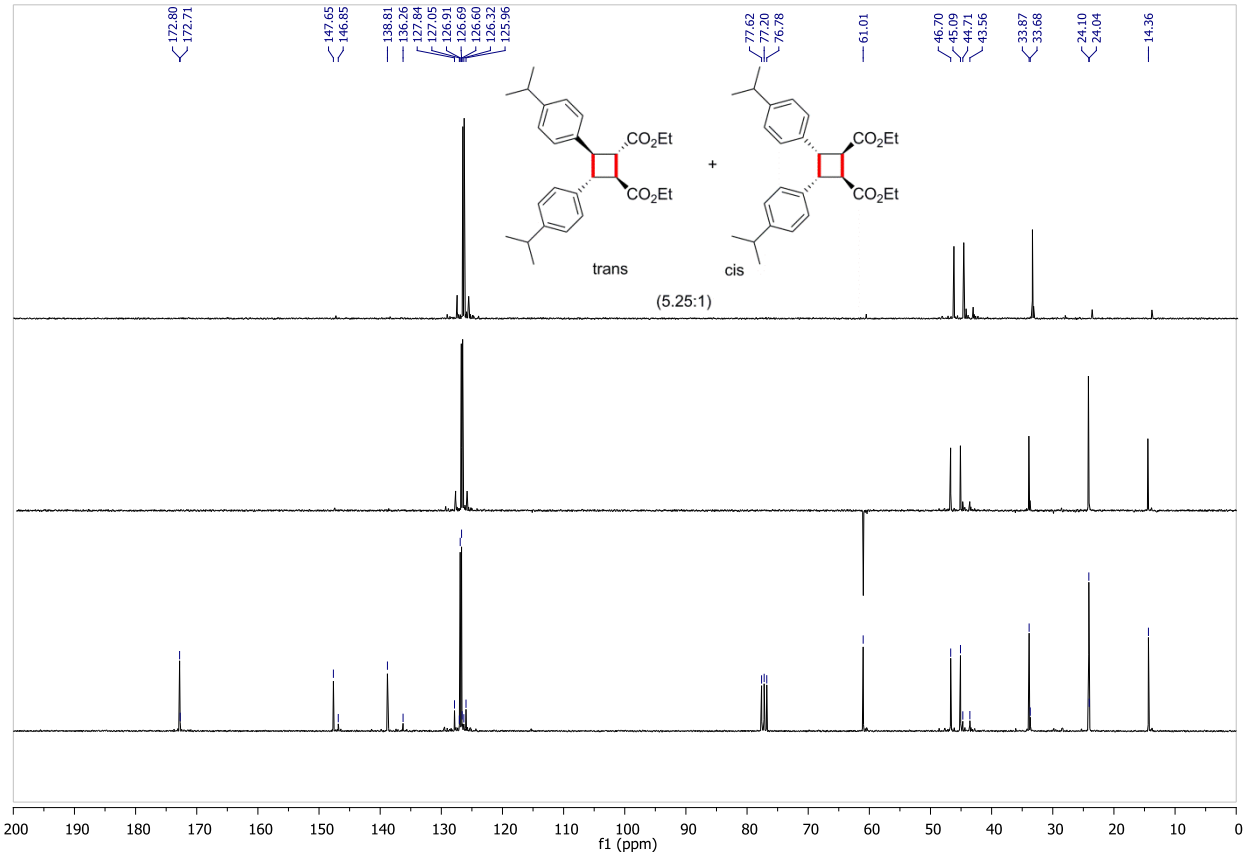
^{13}C-NMR: **3d** (*cis*, after separation)



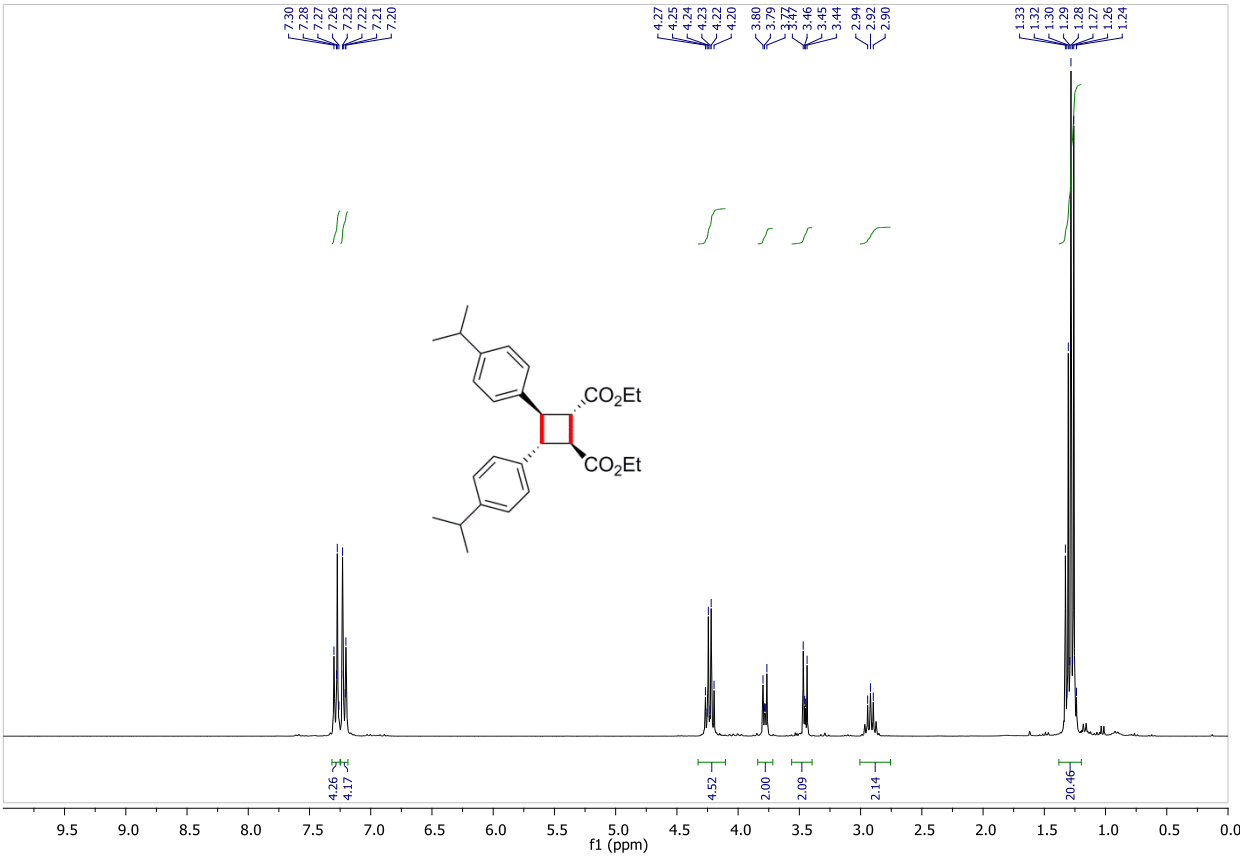
^{1}H-NMR: **2e/3e** (mixture, before separation)



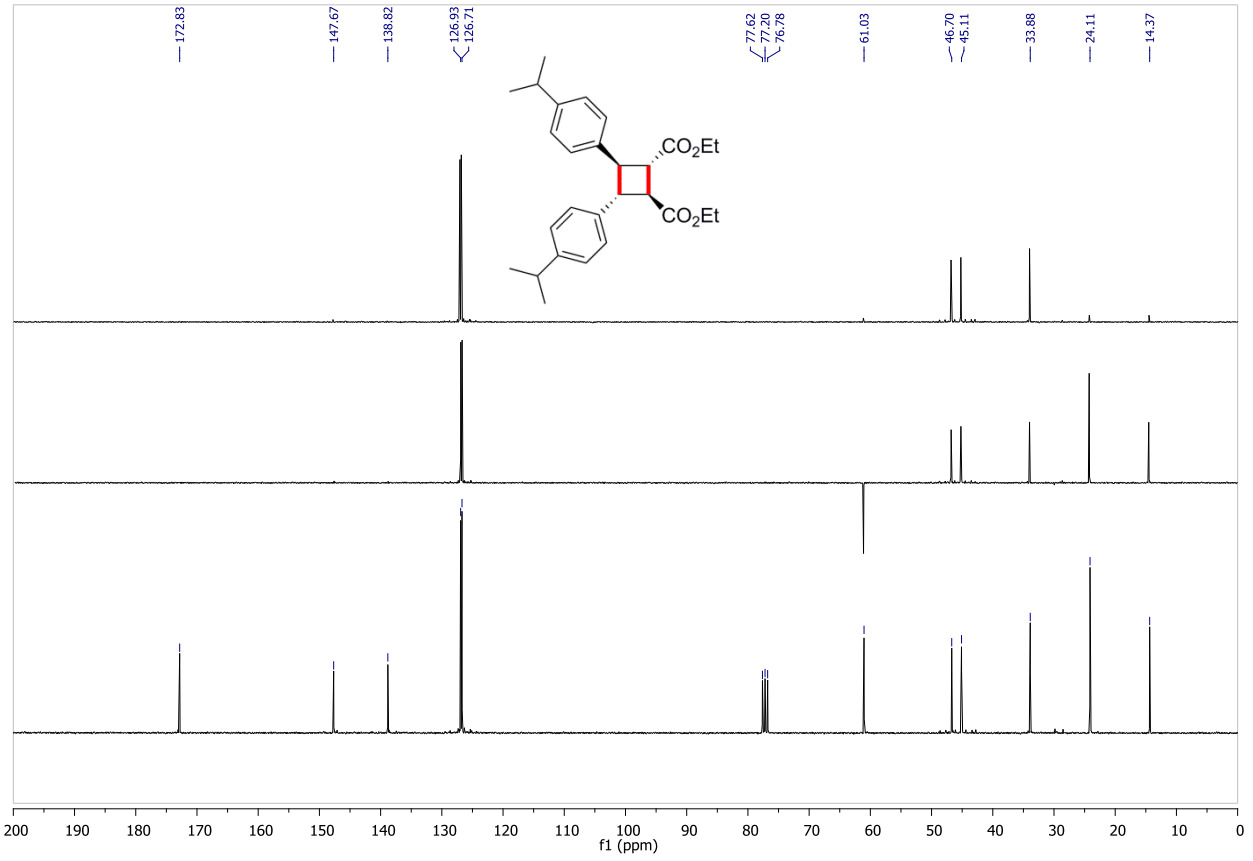
^{13}C-NMR: **2e/3e** (mixture, before separation)



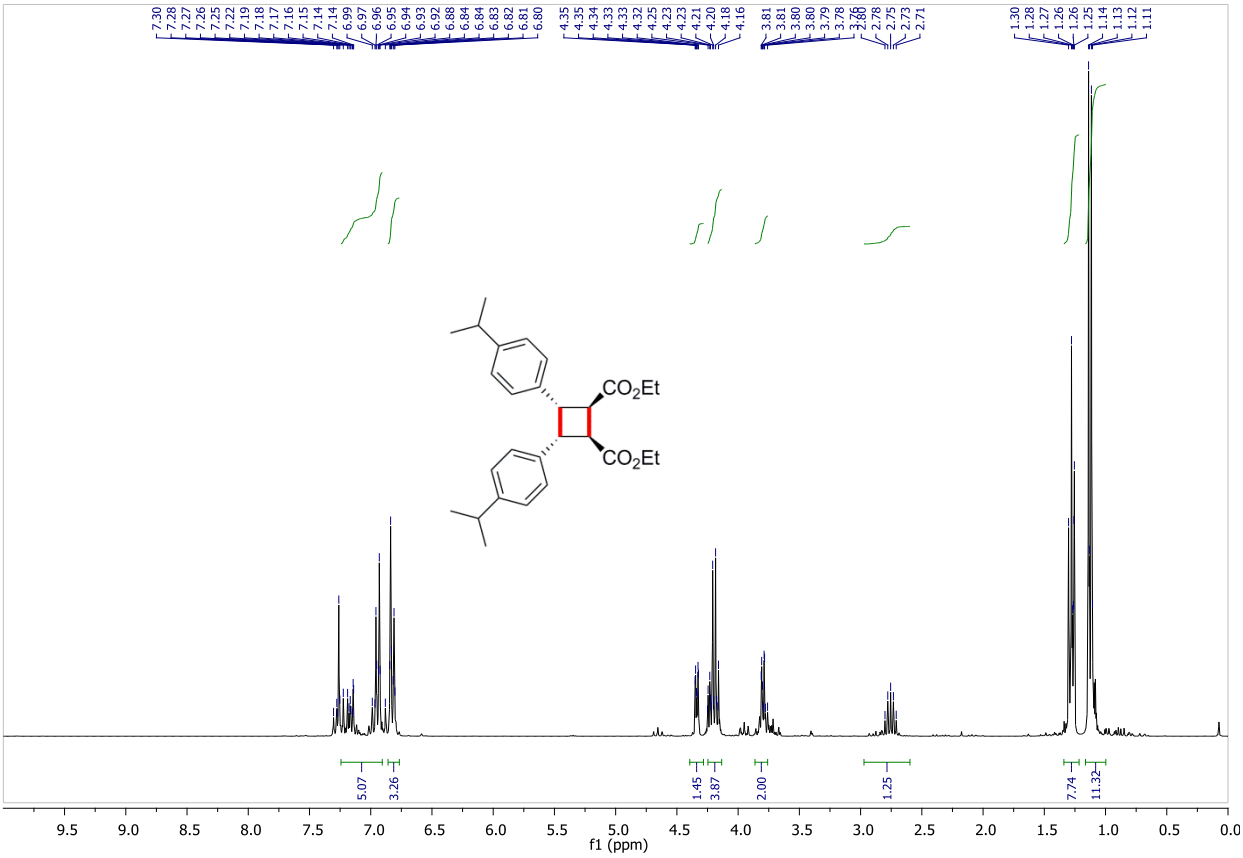
^{1}H-NMR: **2e** (*trans*, after separation)



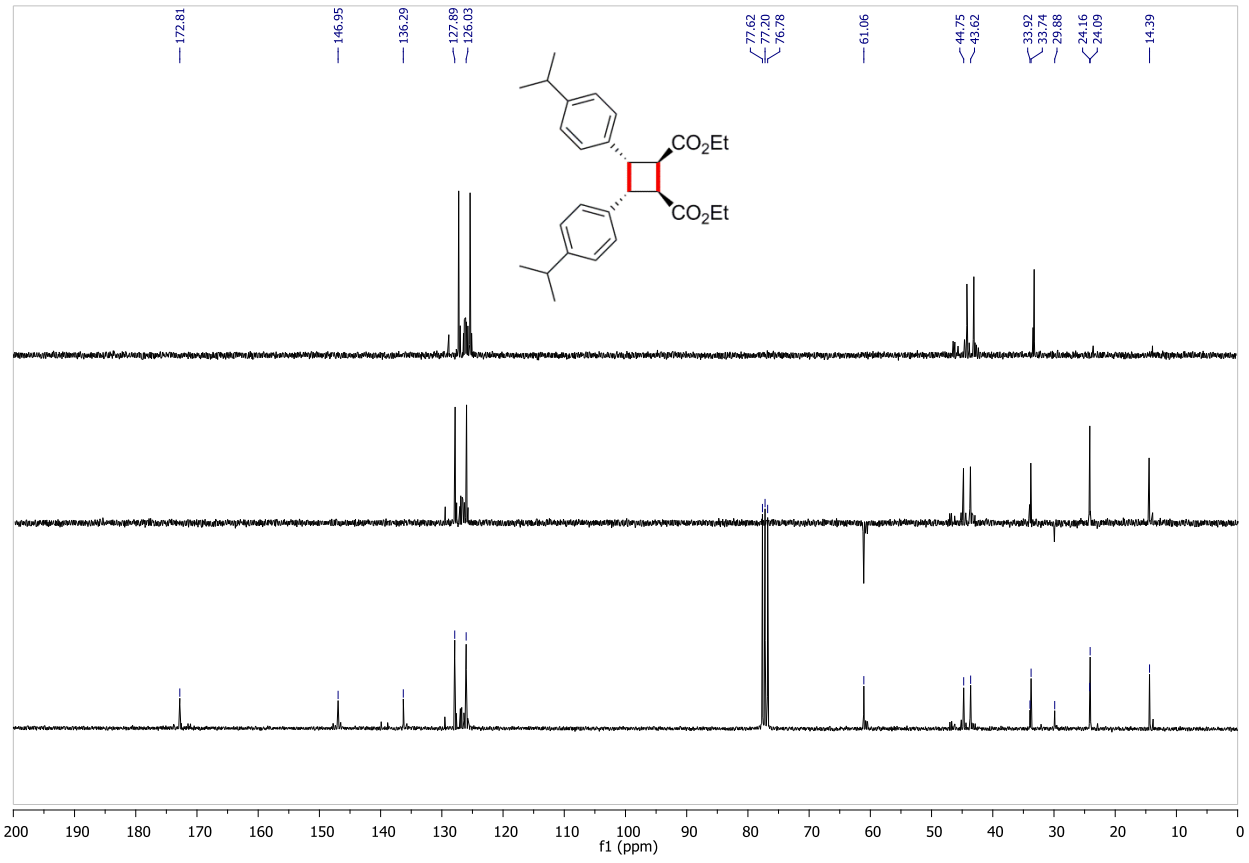
^{13}C-NMR: **2e** (*trans*, after separation)



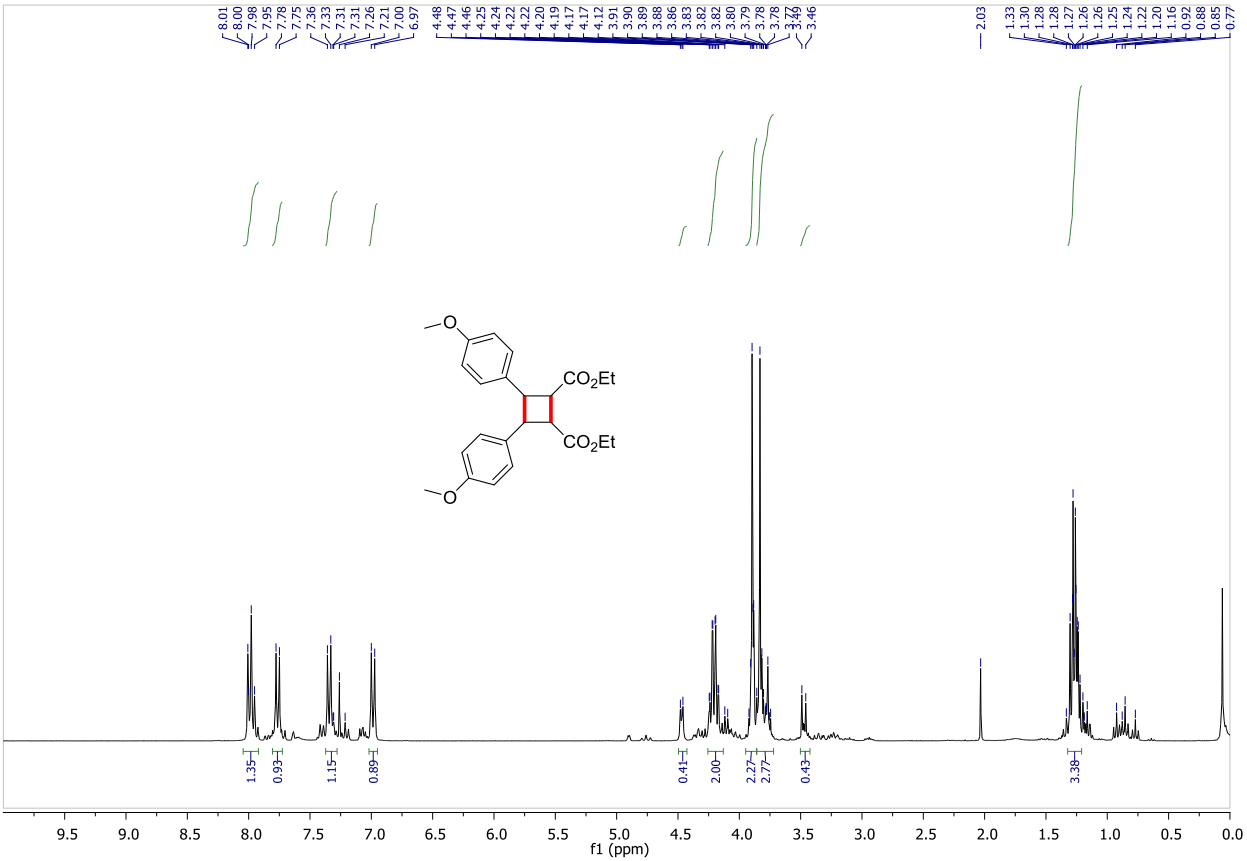
^{1}H-NMR: **3e** (*cis*, after separation)



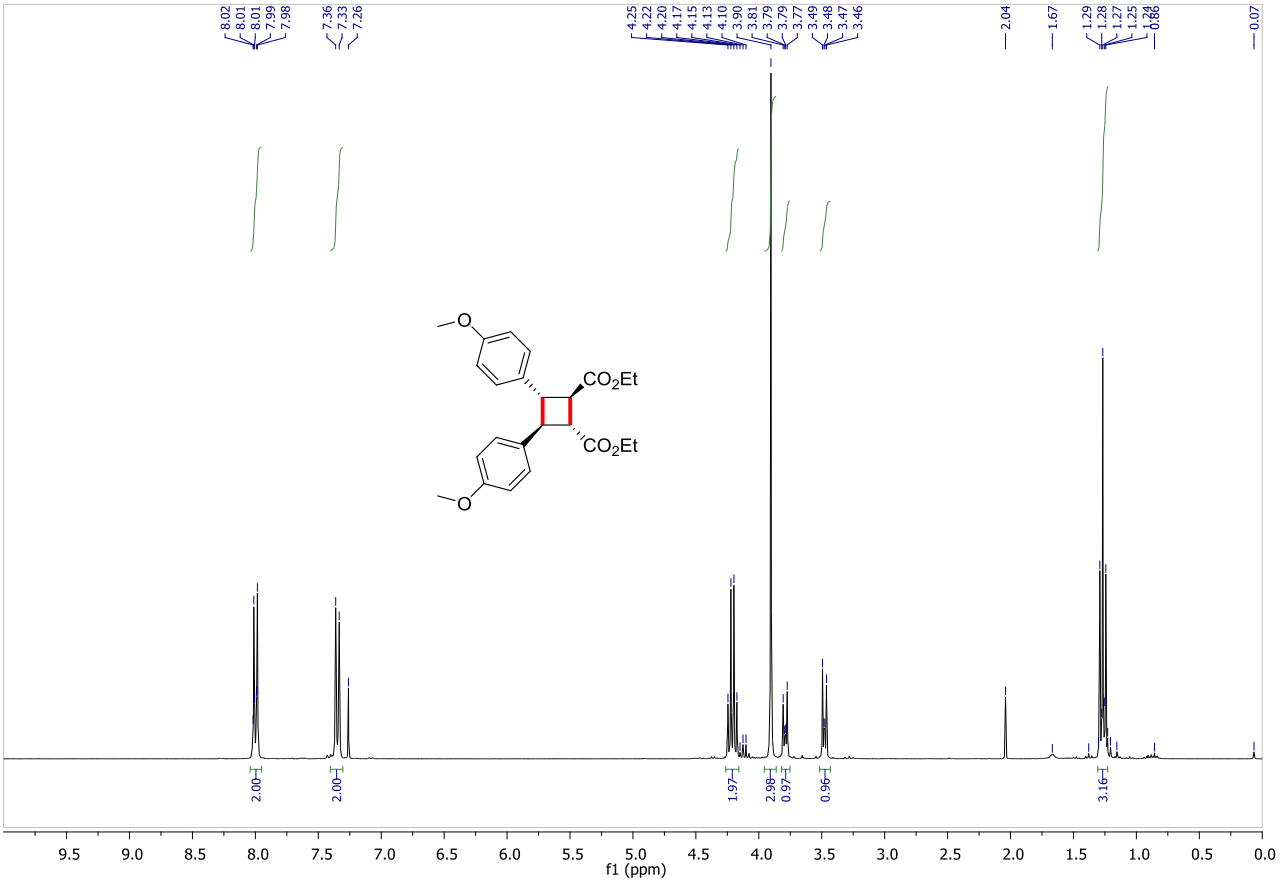
^{13}C-NMR: **3e** (*cis*, after separation)



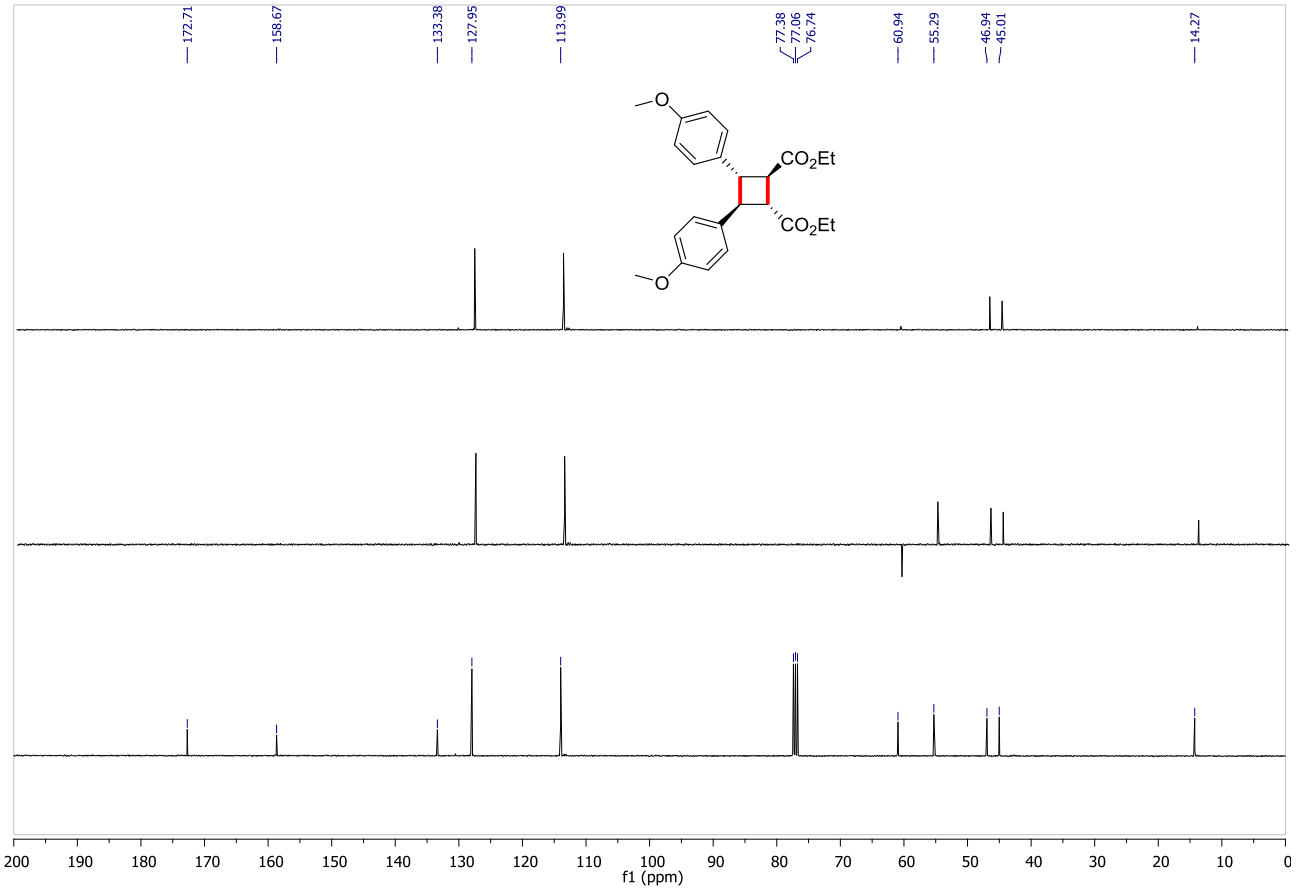
^{1}H-NMR: **2f** (*mixture*, before seperation)



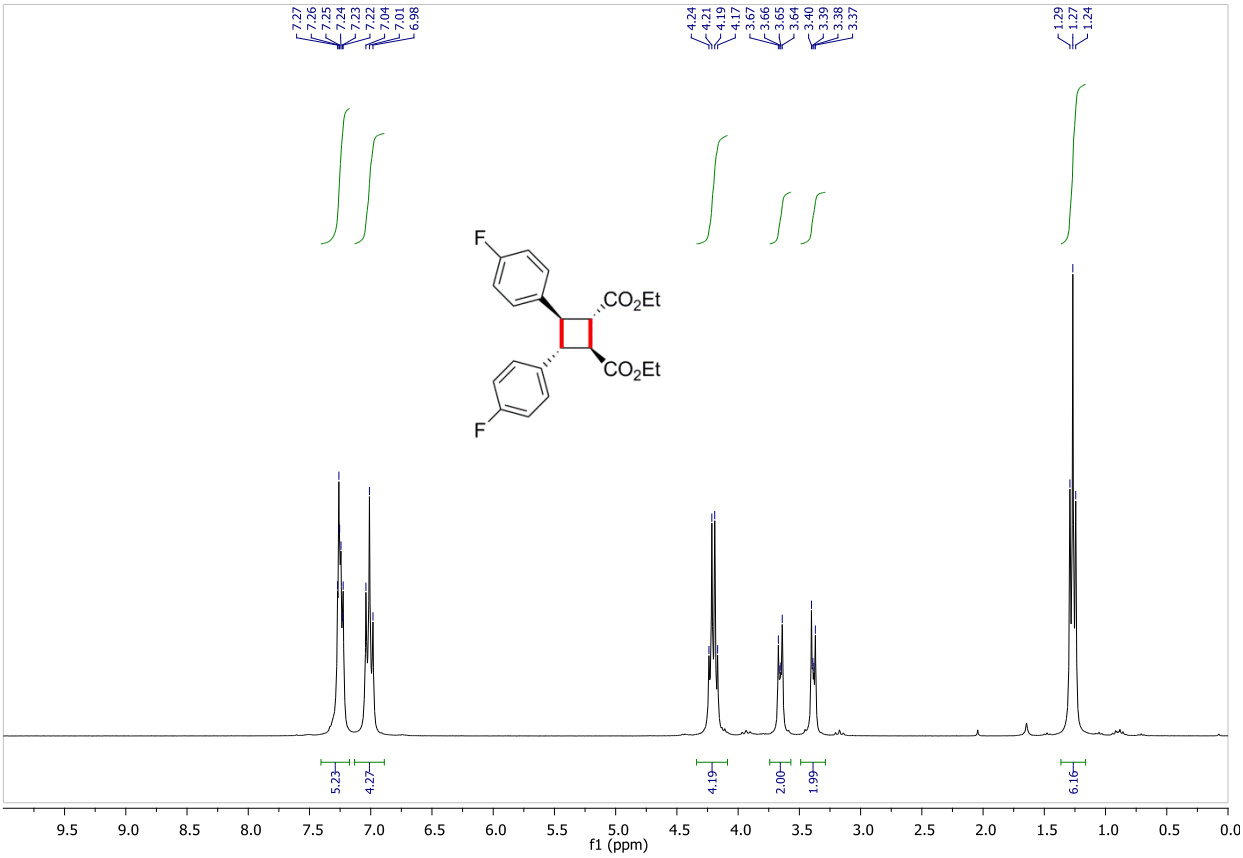
^{1}H-NMR: **2f** (*trans*, after seperation)



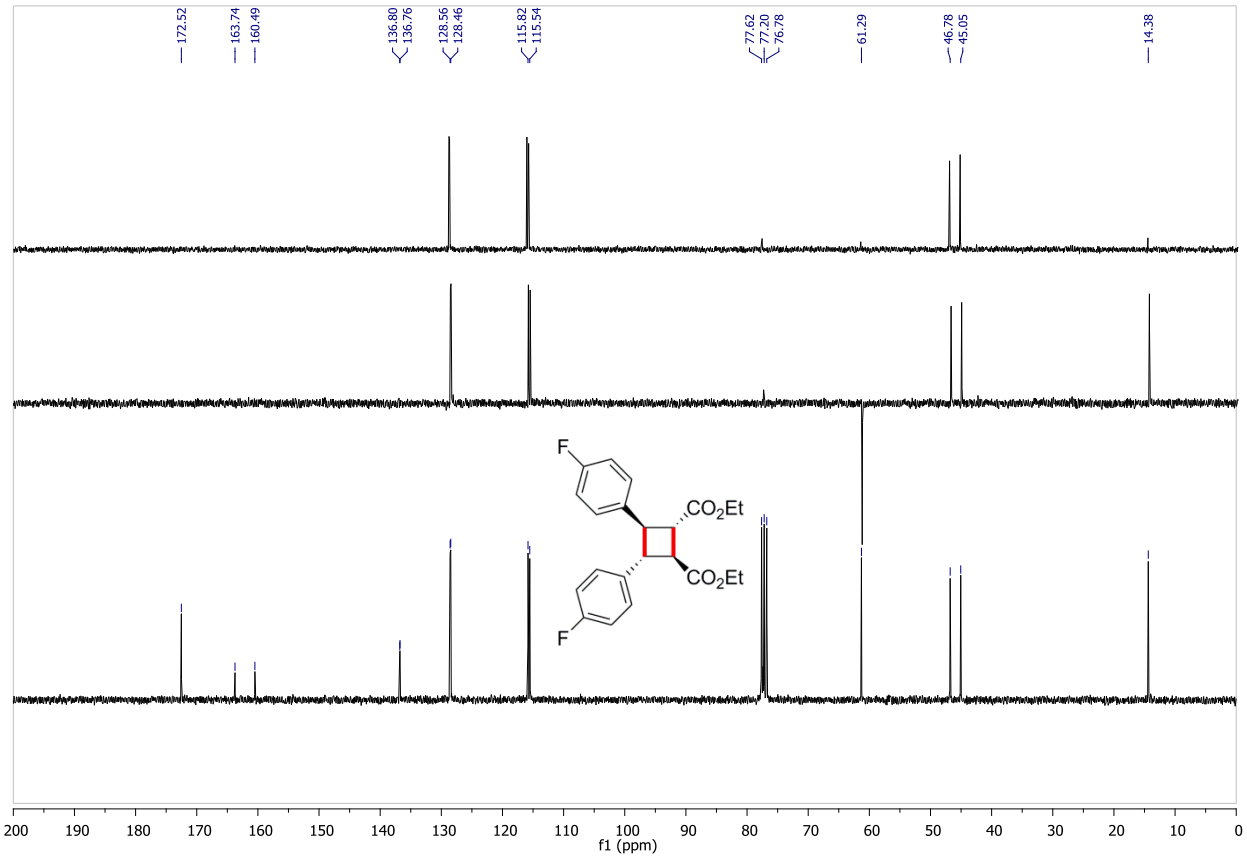
^{13}C-NMR: **2f** (*trans*, after separation)



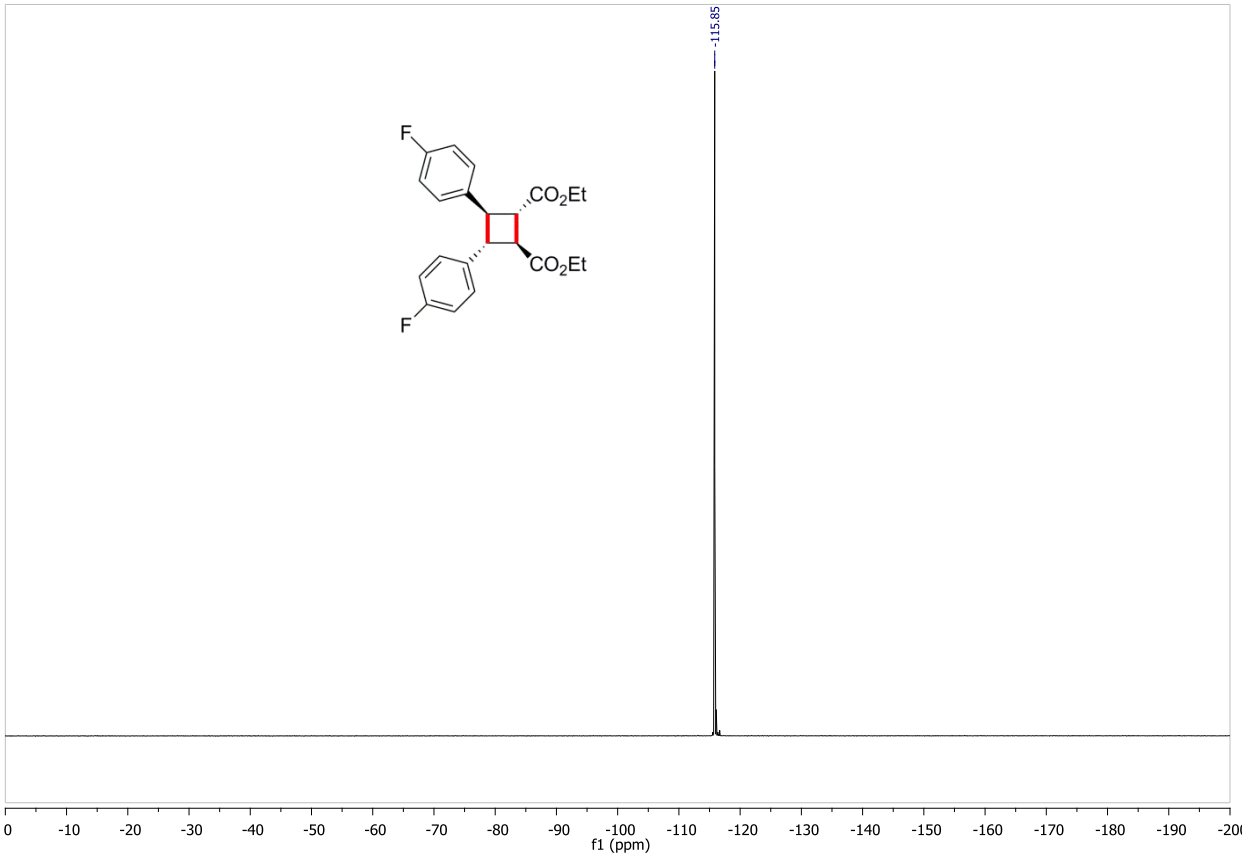
^{1}H-NMR: **2g** (*trans*, after separation)



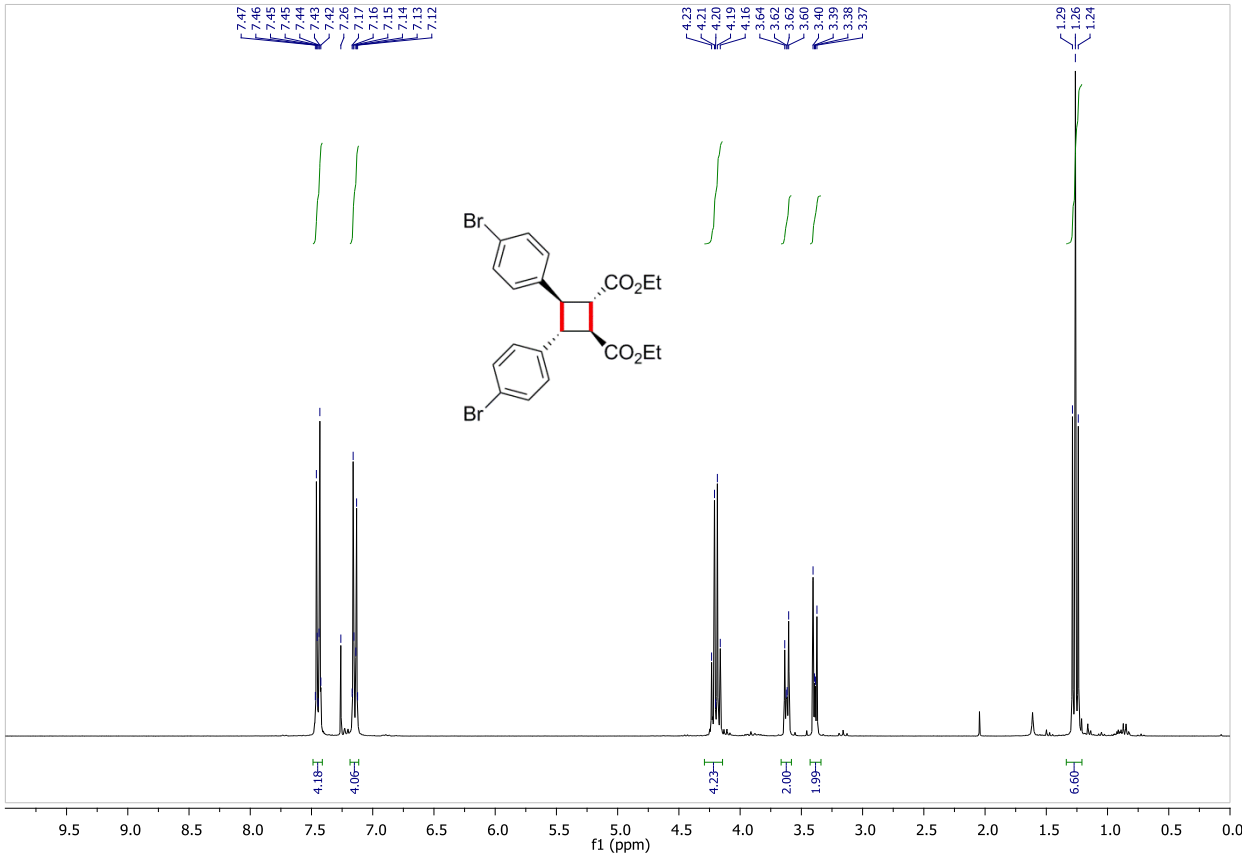
^{13}C-NMR: **2g** (*trans*, after separation)



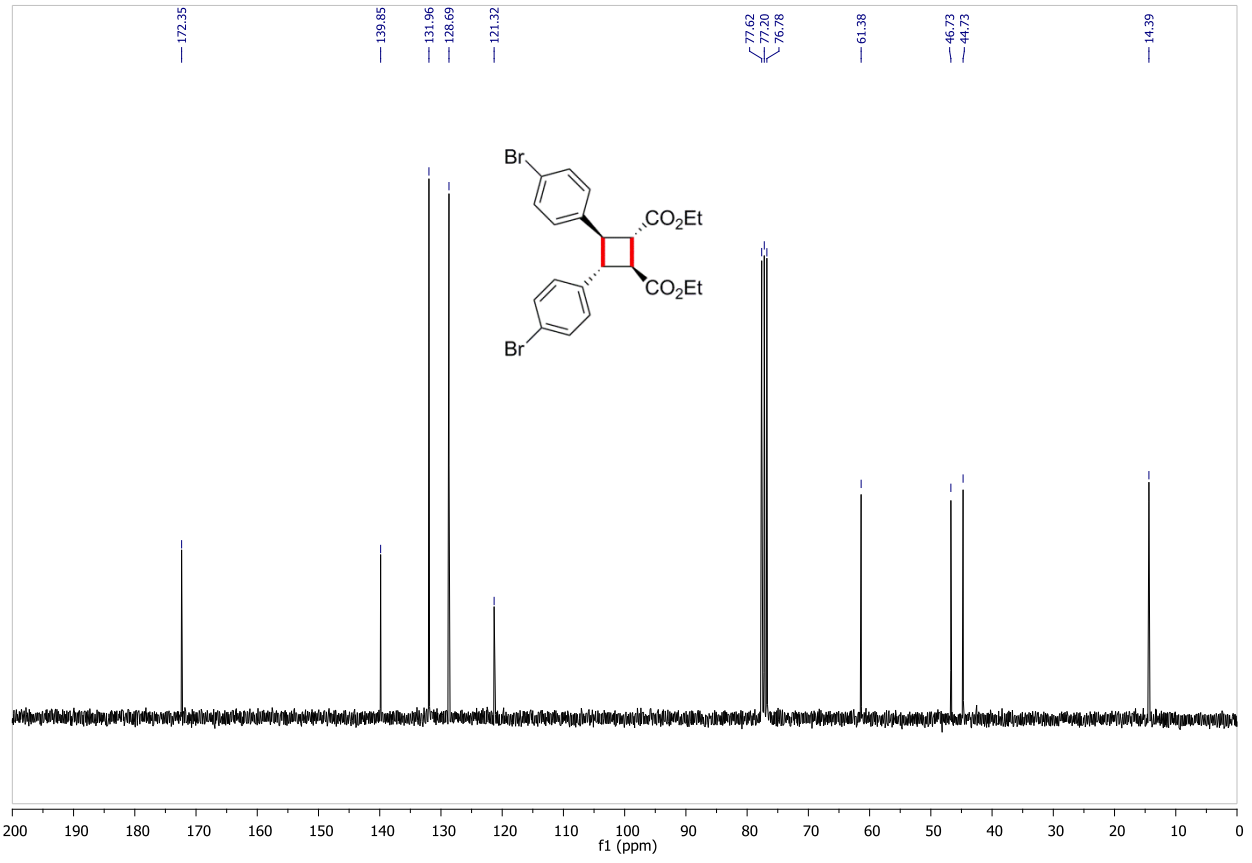
^{19}F-NMR: **2g** (*trans*, after separation)



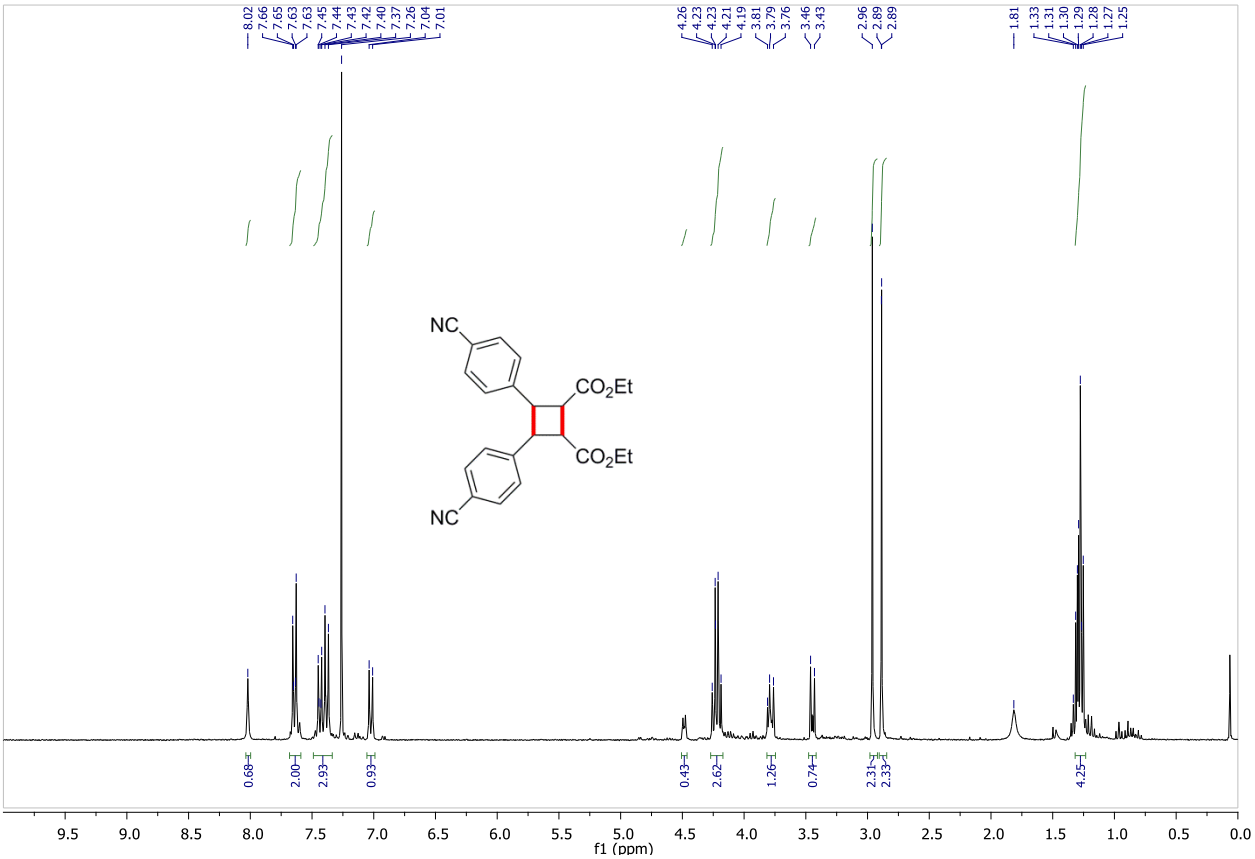
^{1}H-NMR: **2h** (*trans*, after separation)



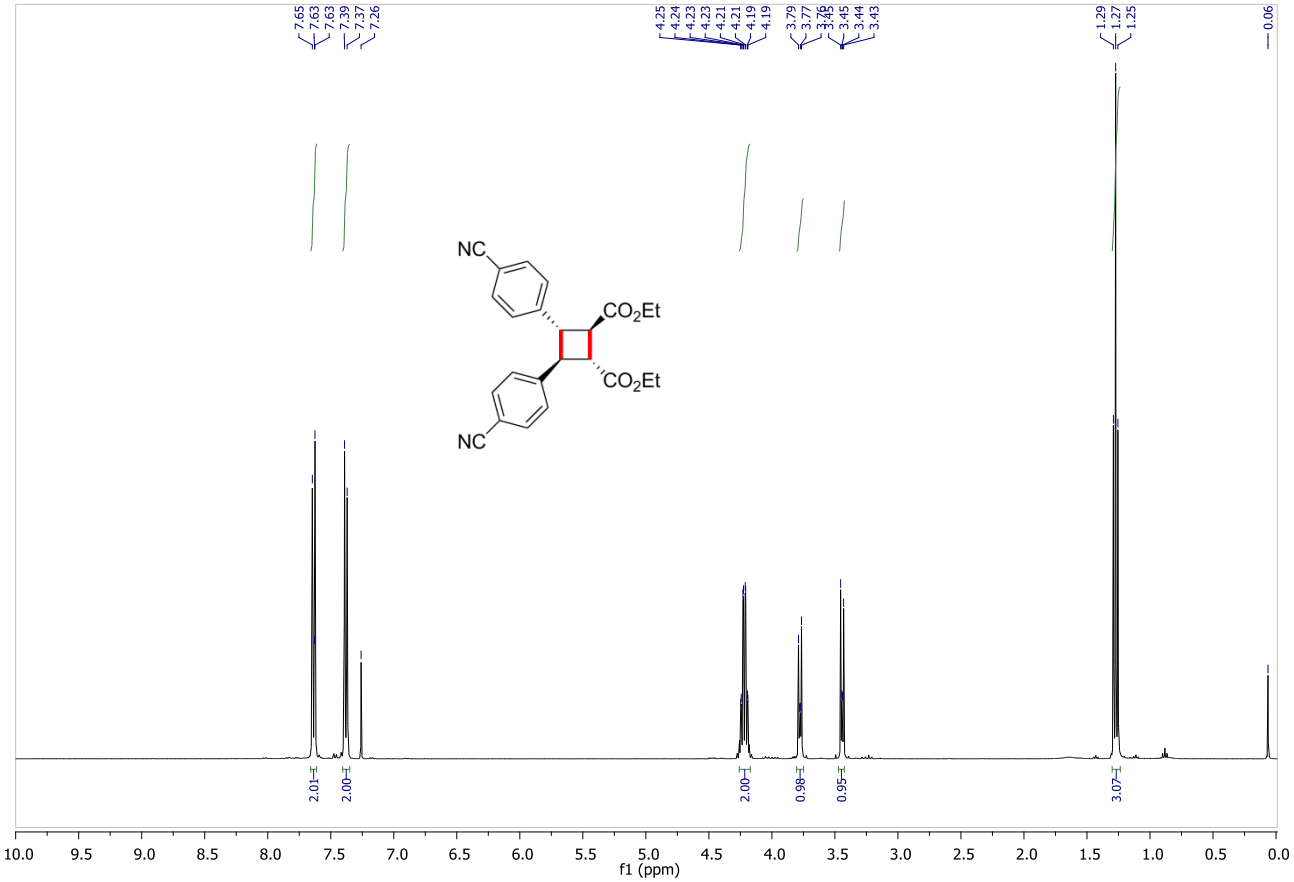
^{13}C-NMR: **2h**(*trans*, after separation)



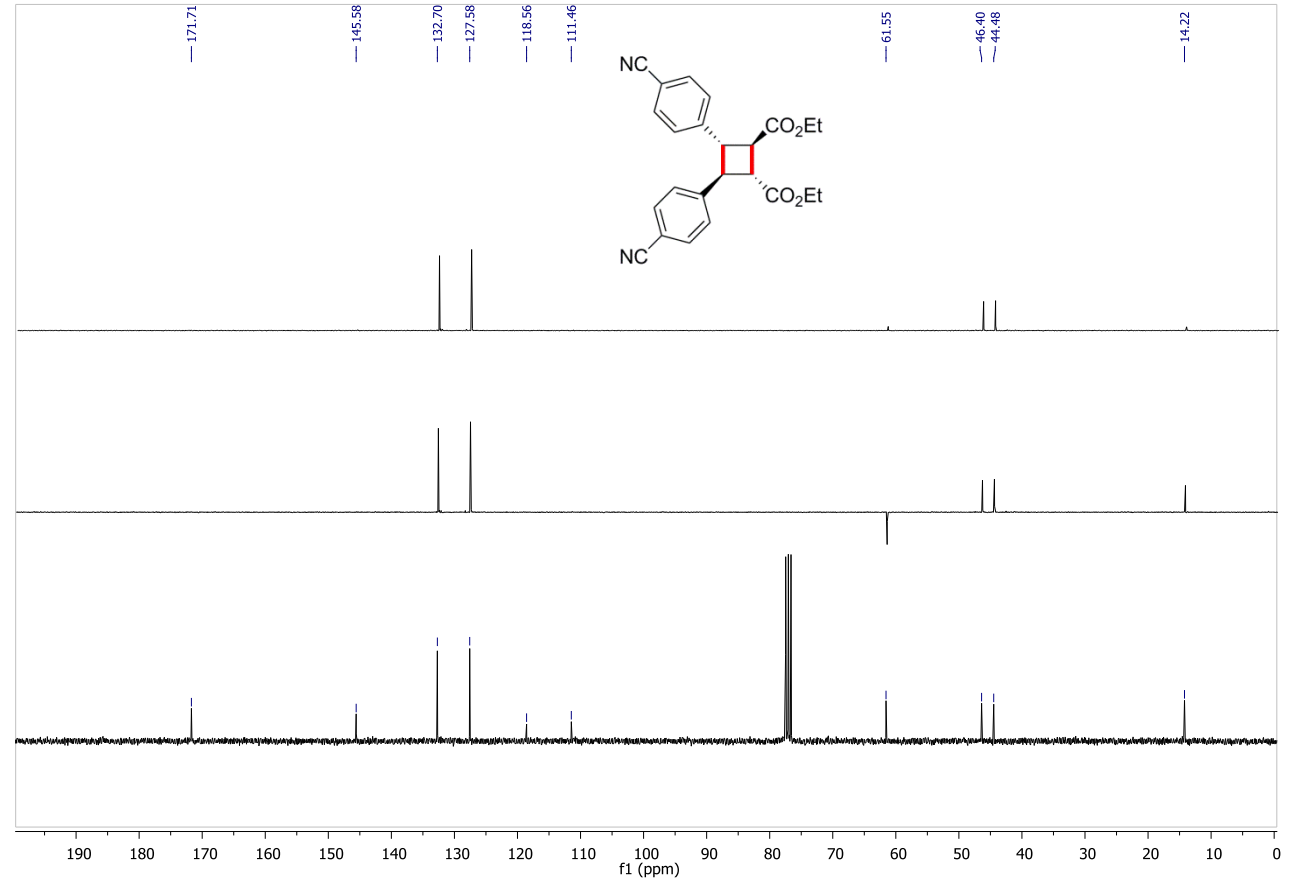
^{1}H-NMR: **2i/3i** (*mixture*, before separation)



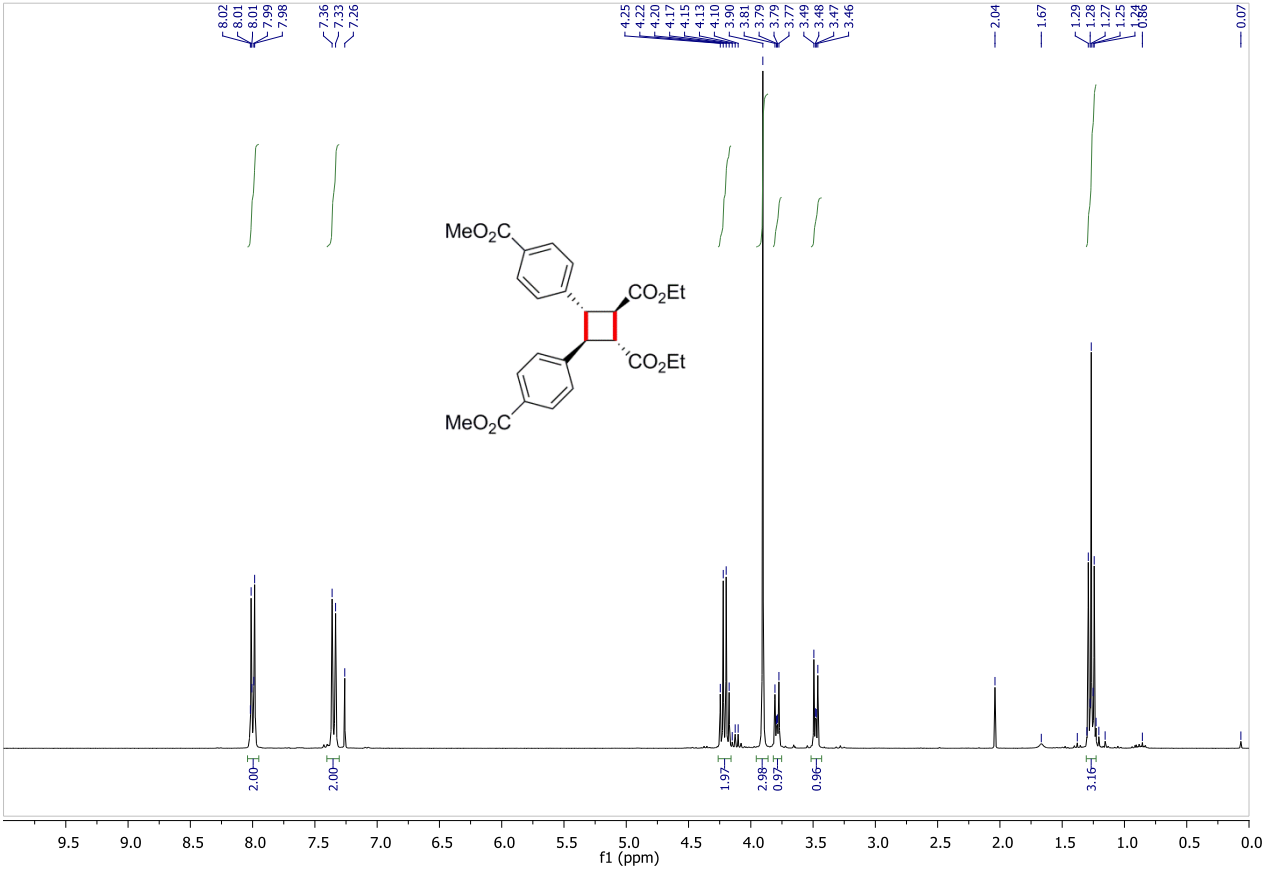
^{1}H-NMR: **2i** (*trans*, after separation)



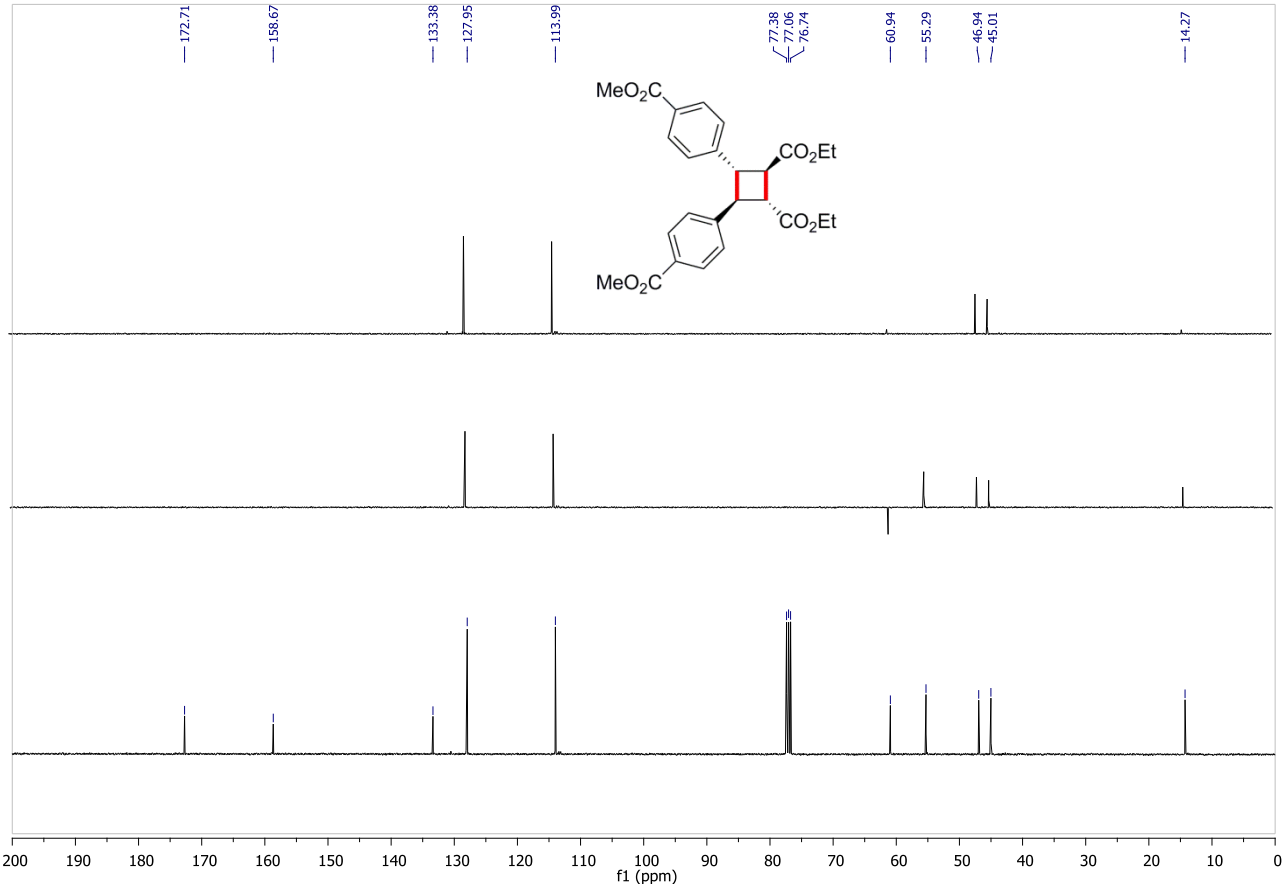
^{13}C-NMR: **2i** (*trans*, after separation)



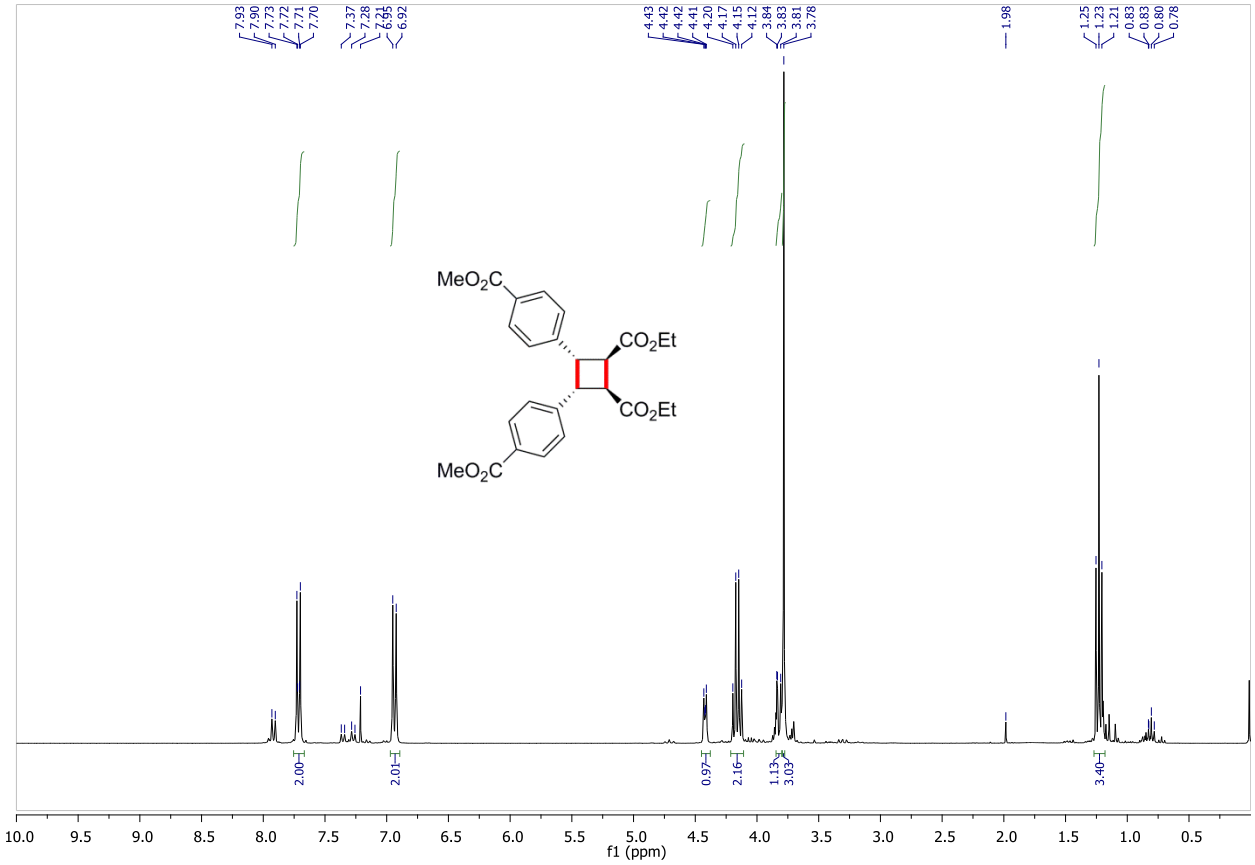
^{1}H-NMR: **2j** (*trans*, after separation)



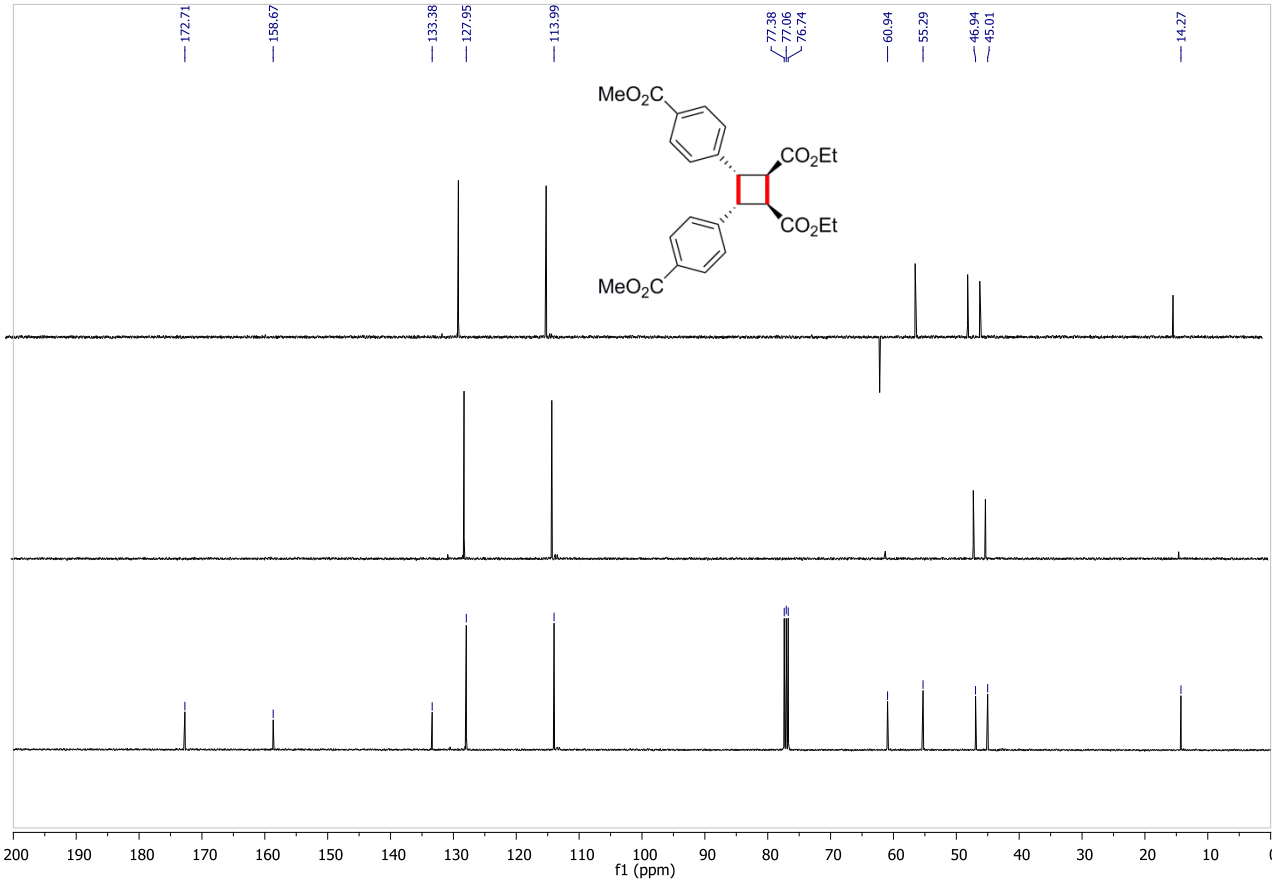
^{13}C-NMR: **2j** (*trans*, after separation)



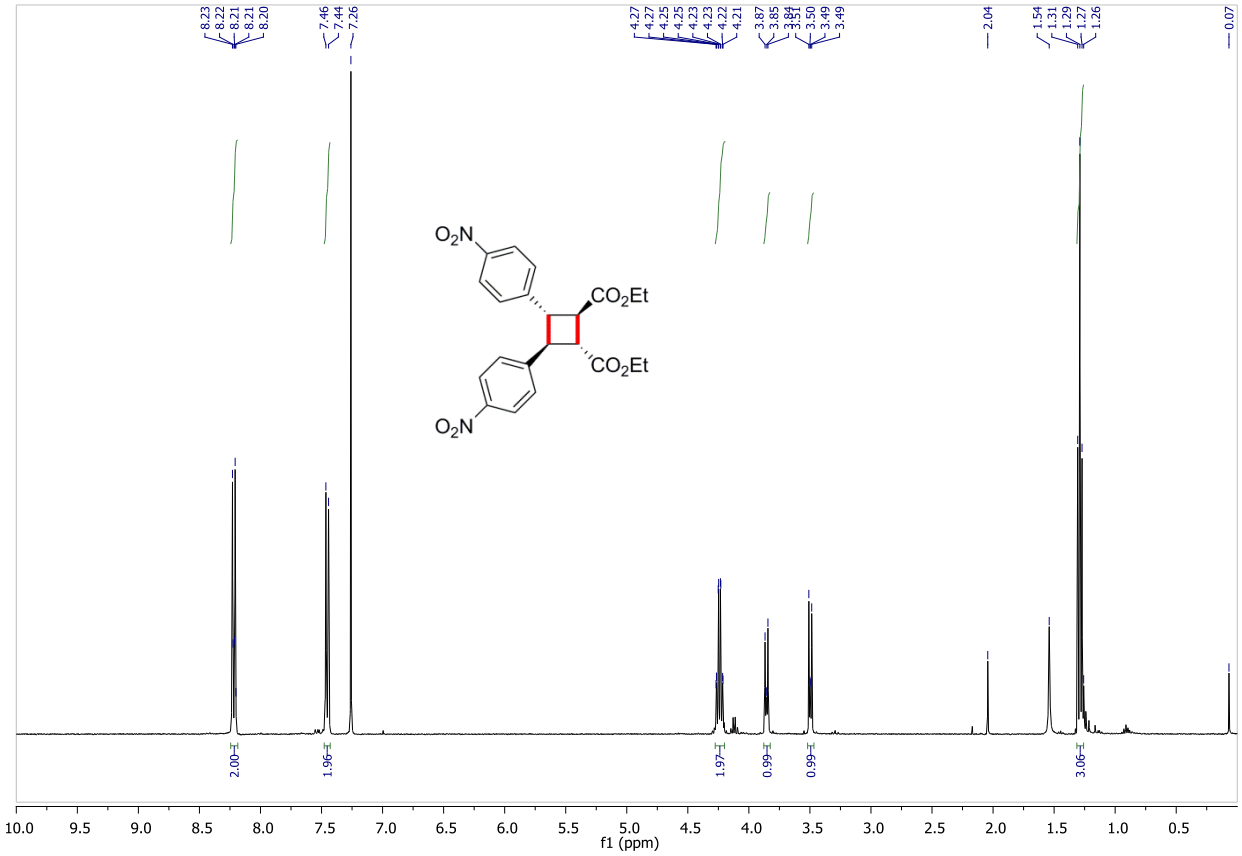
^{1}H-NMR: **3j** (*cis*, after separation)



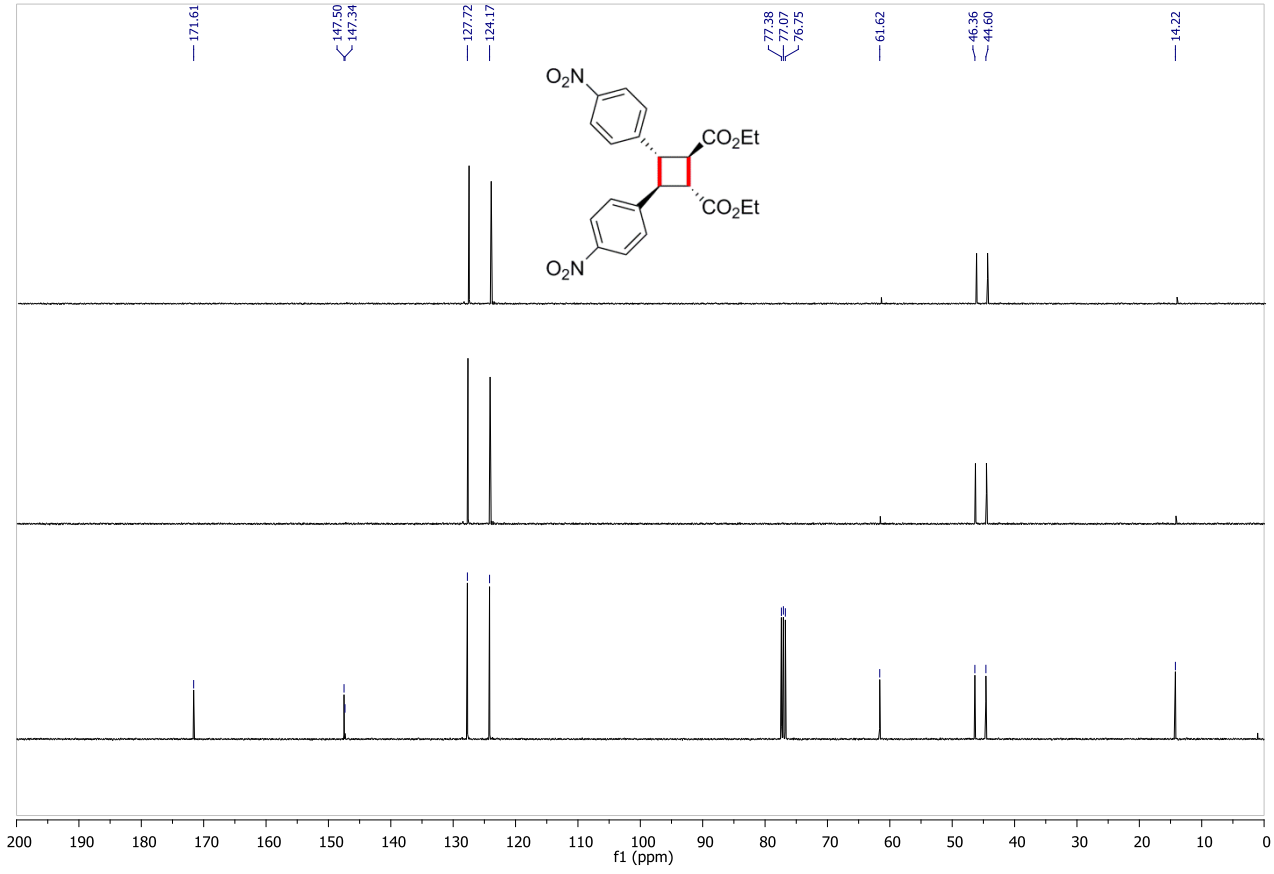
^{13}C-NMR: **3j** (*cis*, after separation)



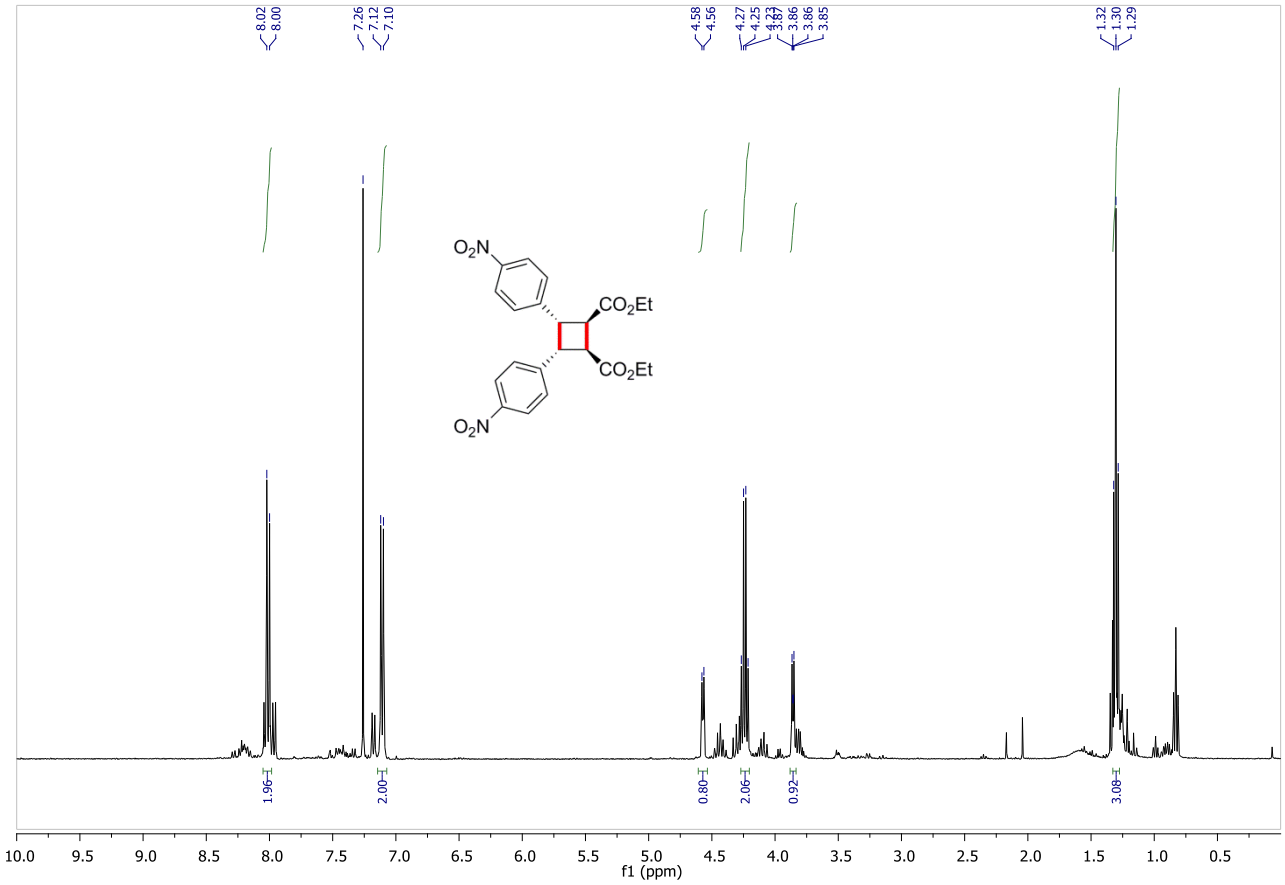
^{1}H-NMR: **2k** (*trans*, after separation)



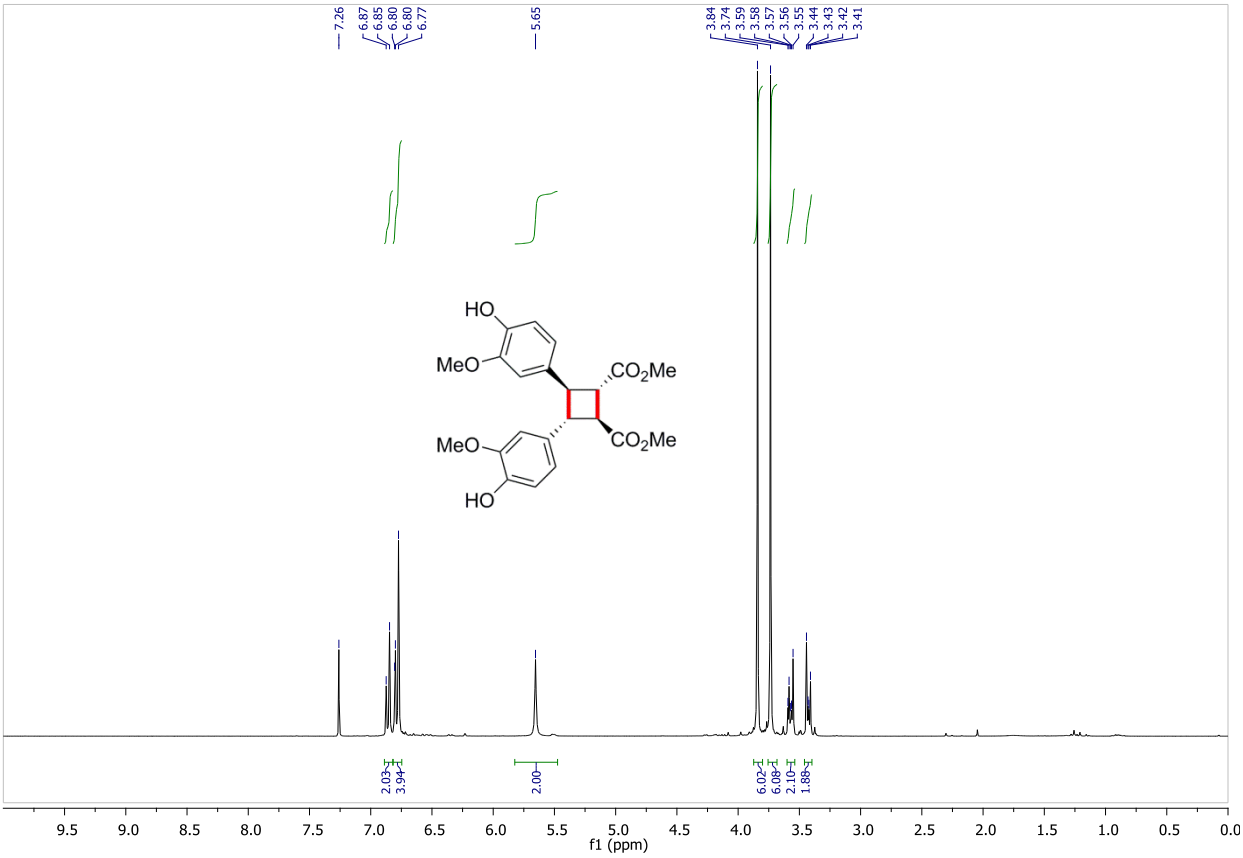
^{13}C-NMR: **2k** (*trans*, after separation)



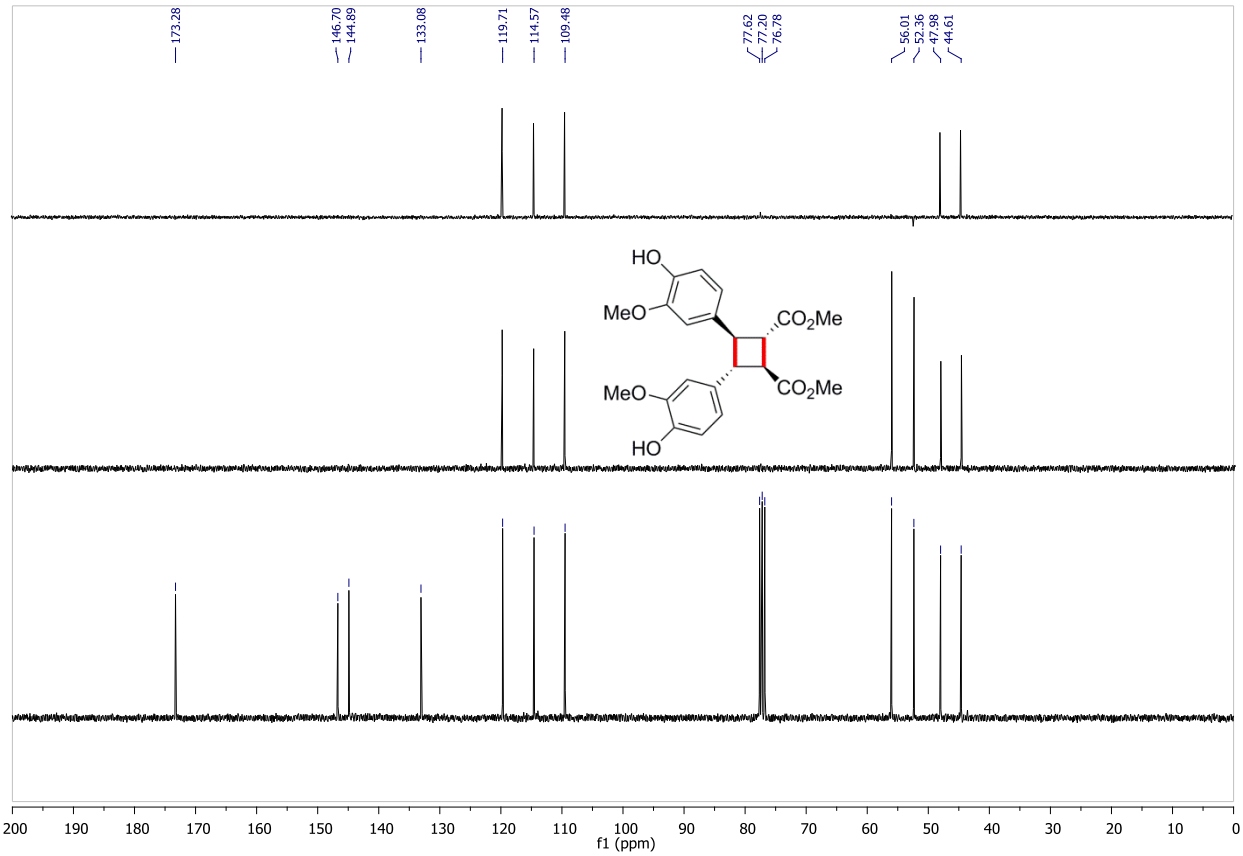
^{1}H-NMR: **3k** (*cis*, after separation)



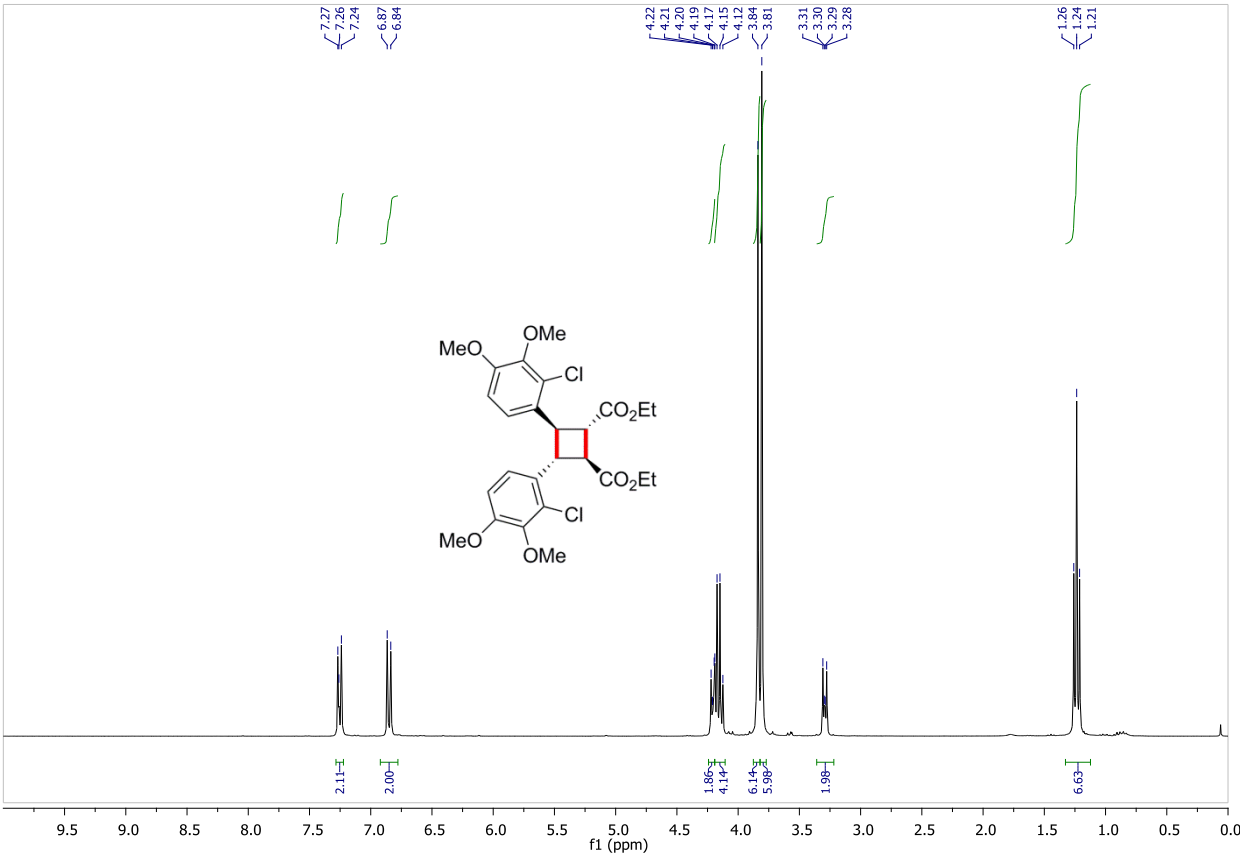
^{1}H-NMR: **2l** (*trans*, after separation)



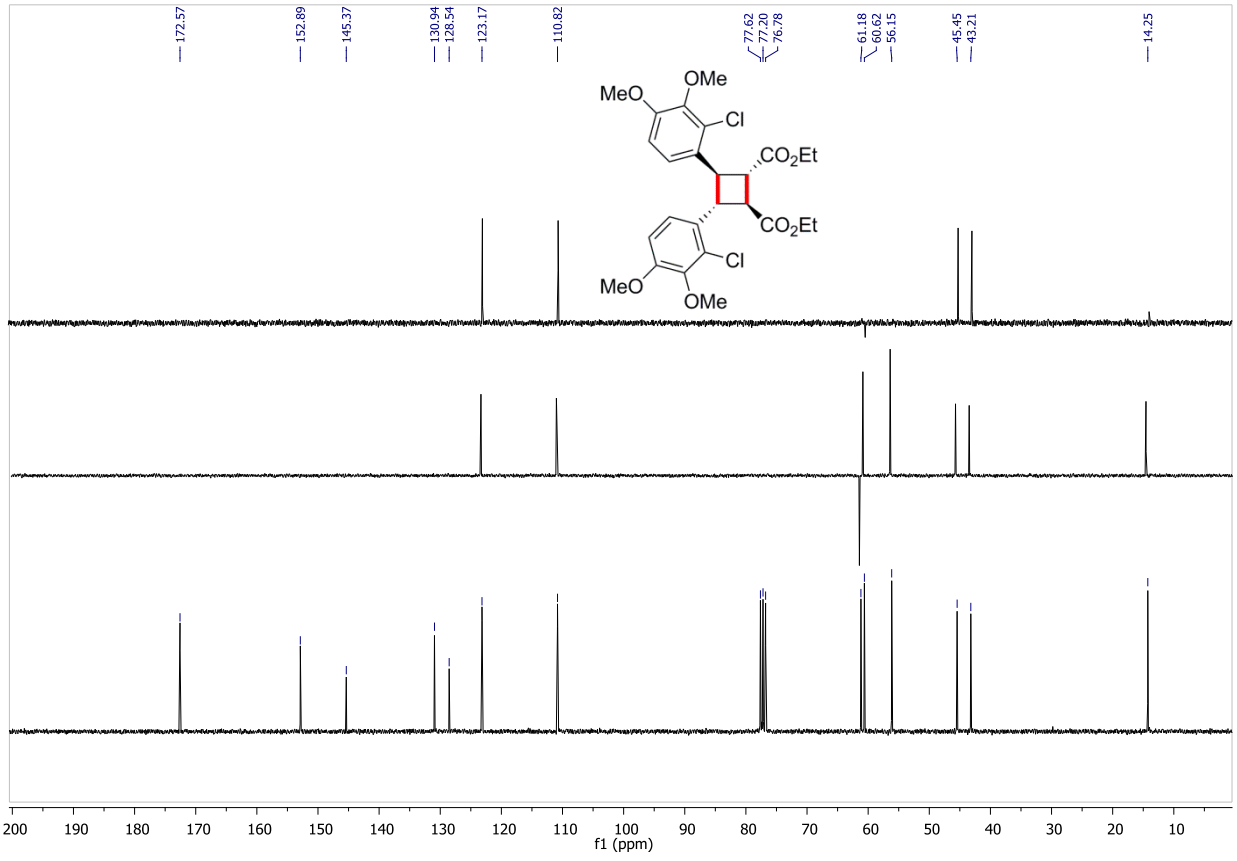
^{13}C-NMR: **2l** (*trans*, after separation)



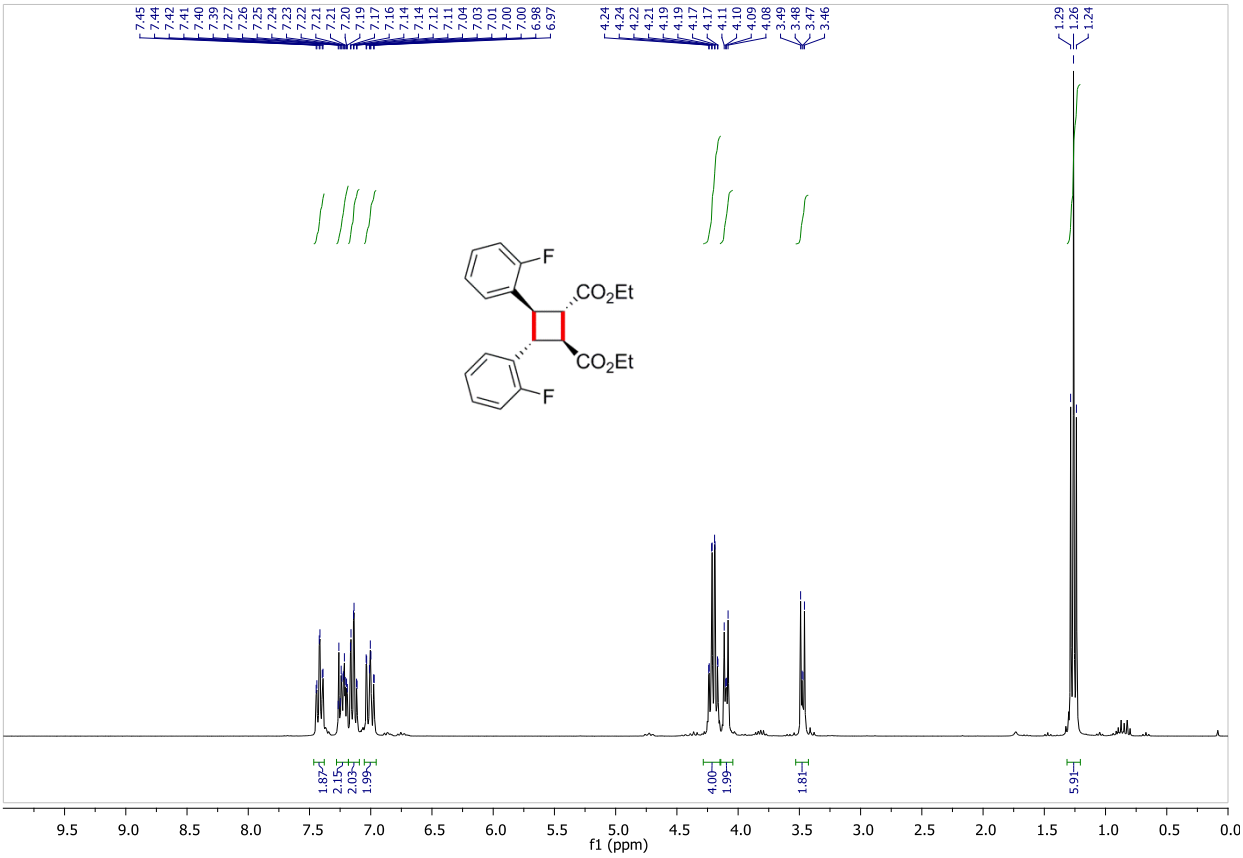
^{1}H-NMR: **2m** (*trans*, after separation)



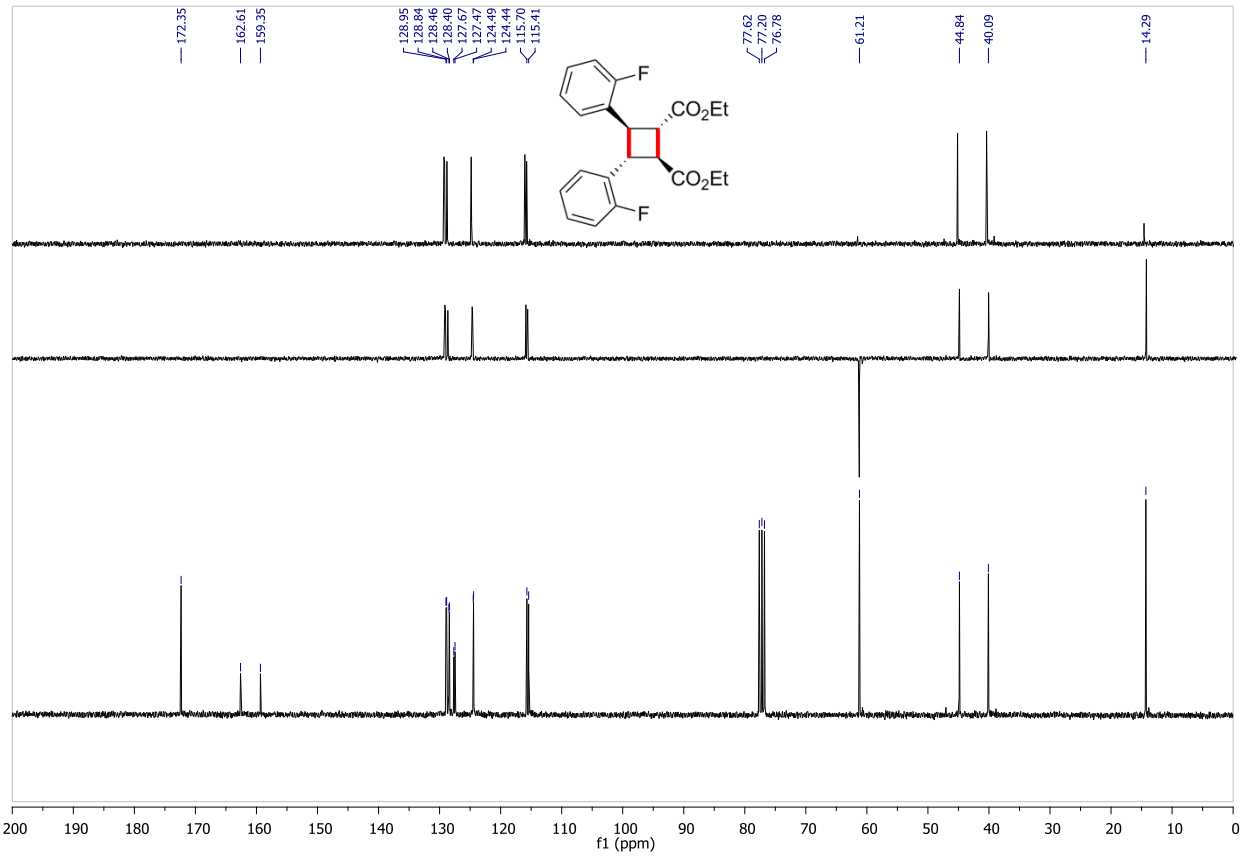
^{13}C-NMR: **2m** (*trans*, after separation)



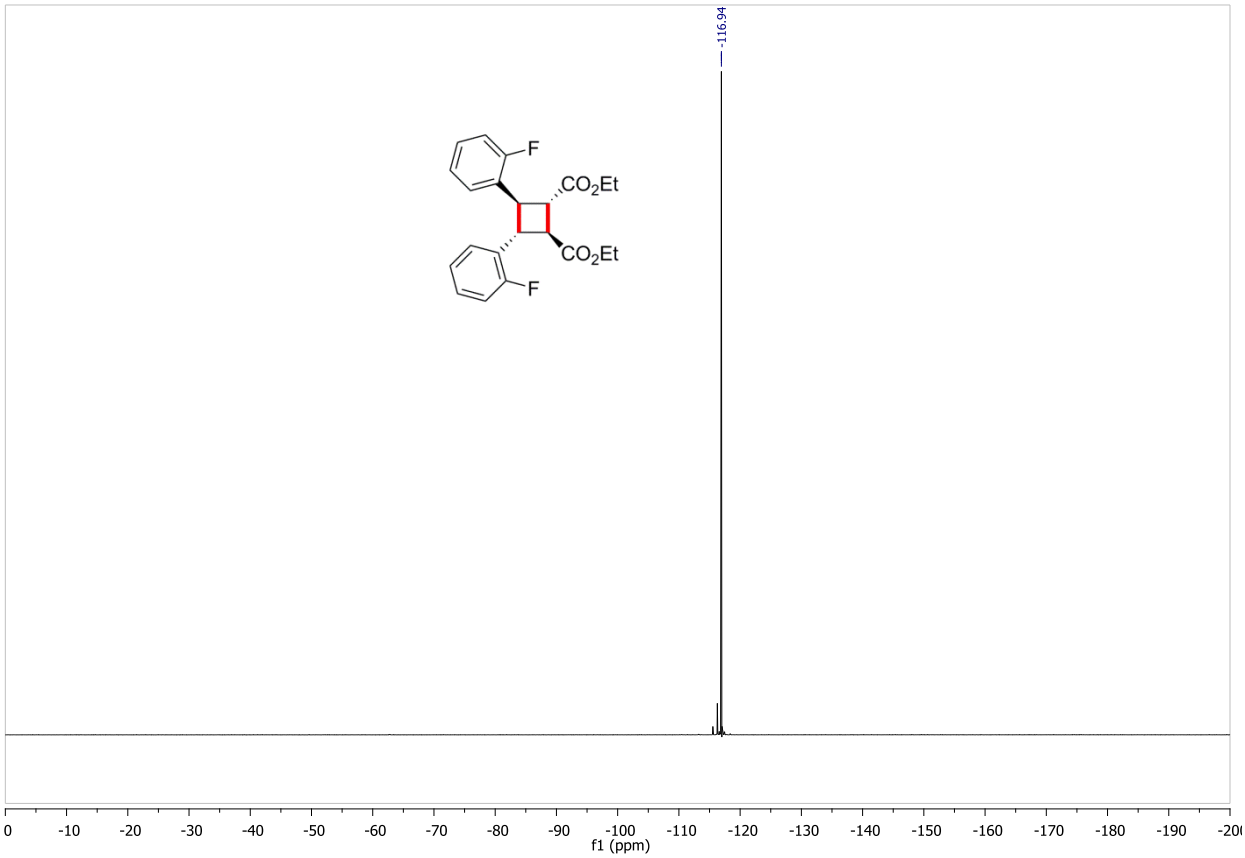
^{1}H-NMR: **2n** (*trans*, after separation)



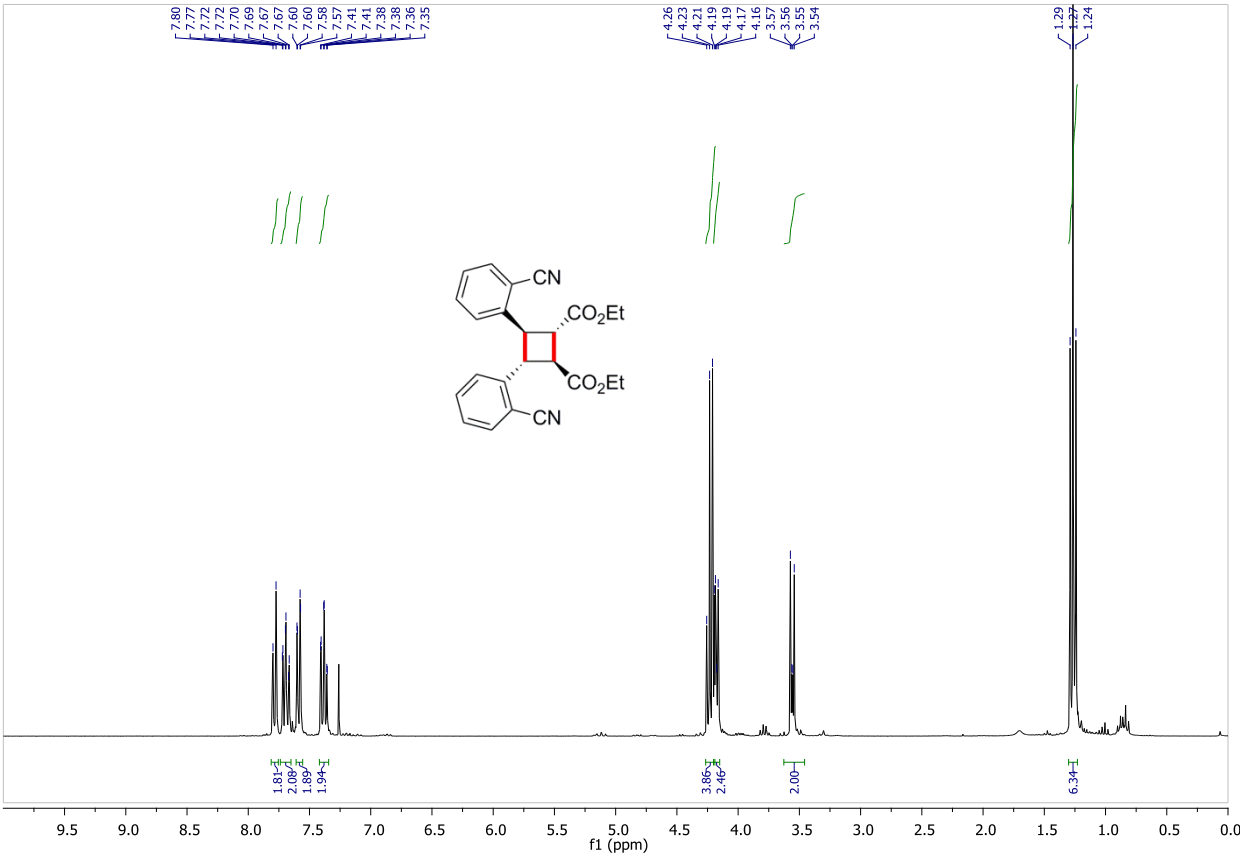
^{13}C-NMR: **2n** (*trans*, after separation)



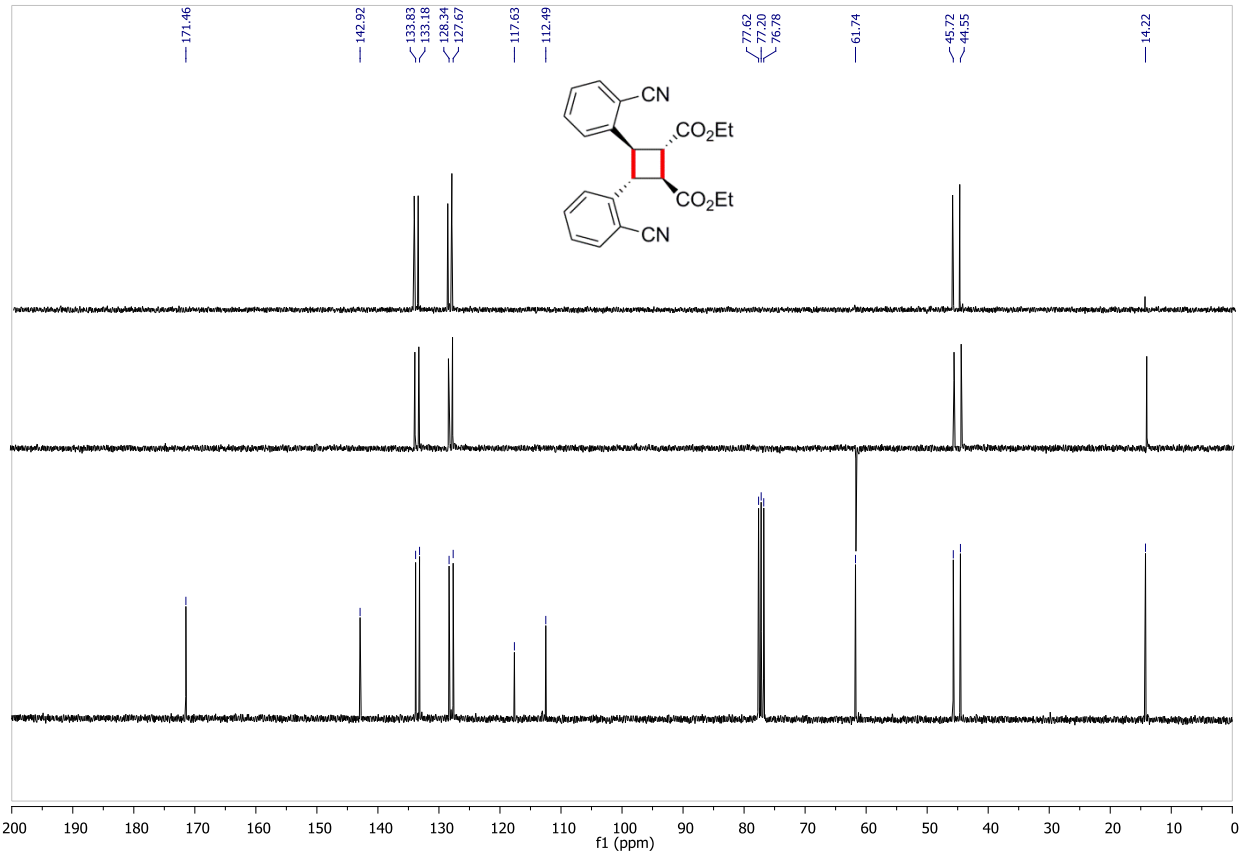
^{19}F-NMR: **2n** (*trans*, after separation)



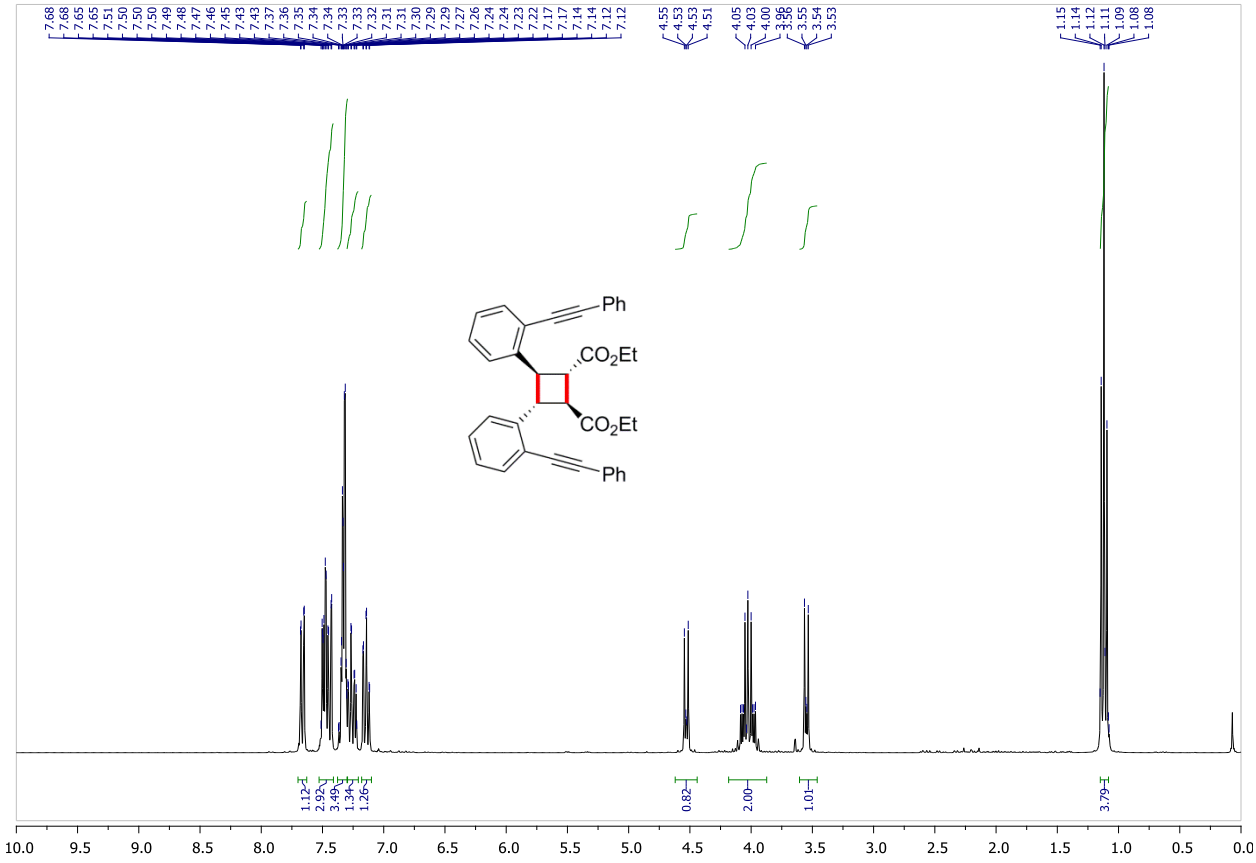
^{1}H-NMR: **2o** (*trans*, after separation)



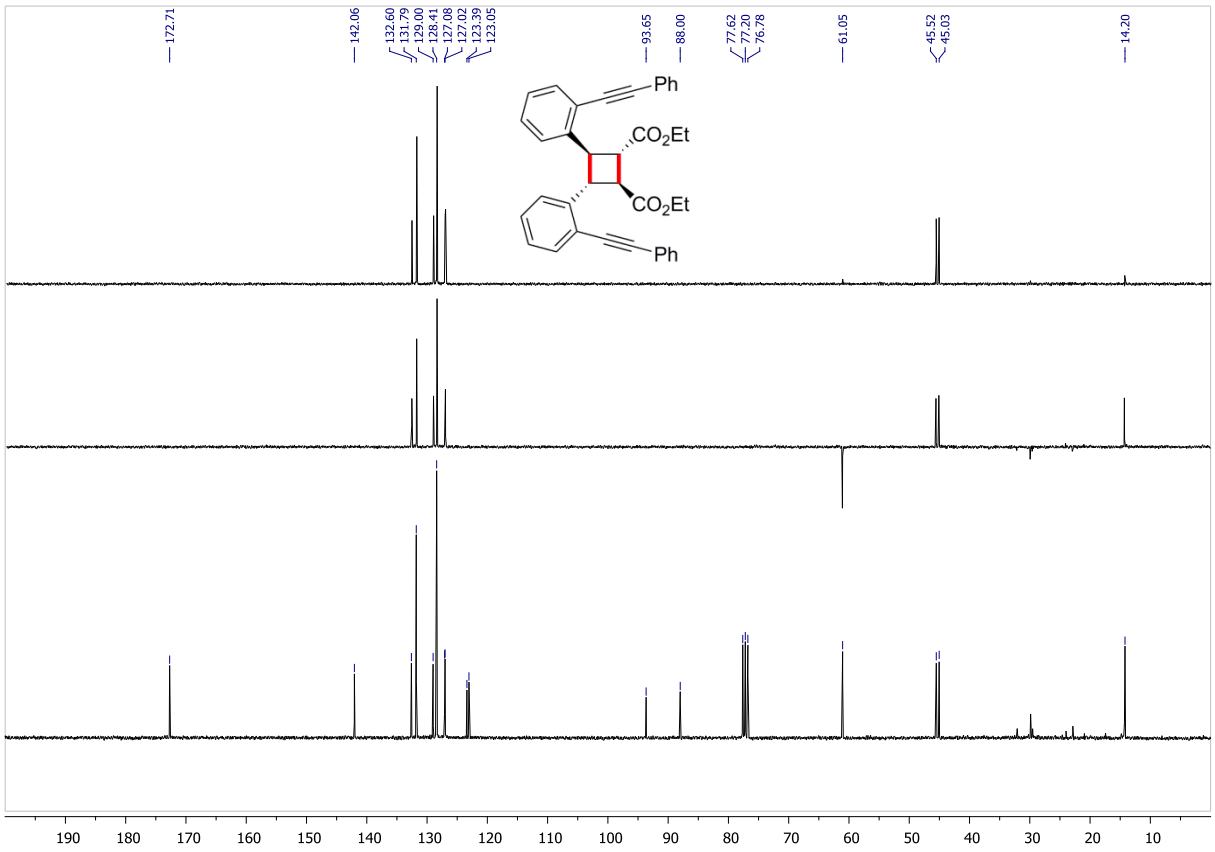
^{13}C-NMR: **2o** (*trans*, after separation)



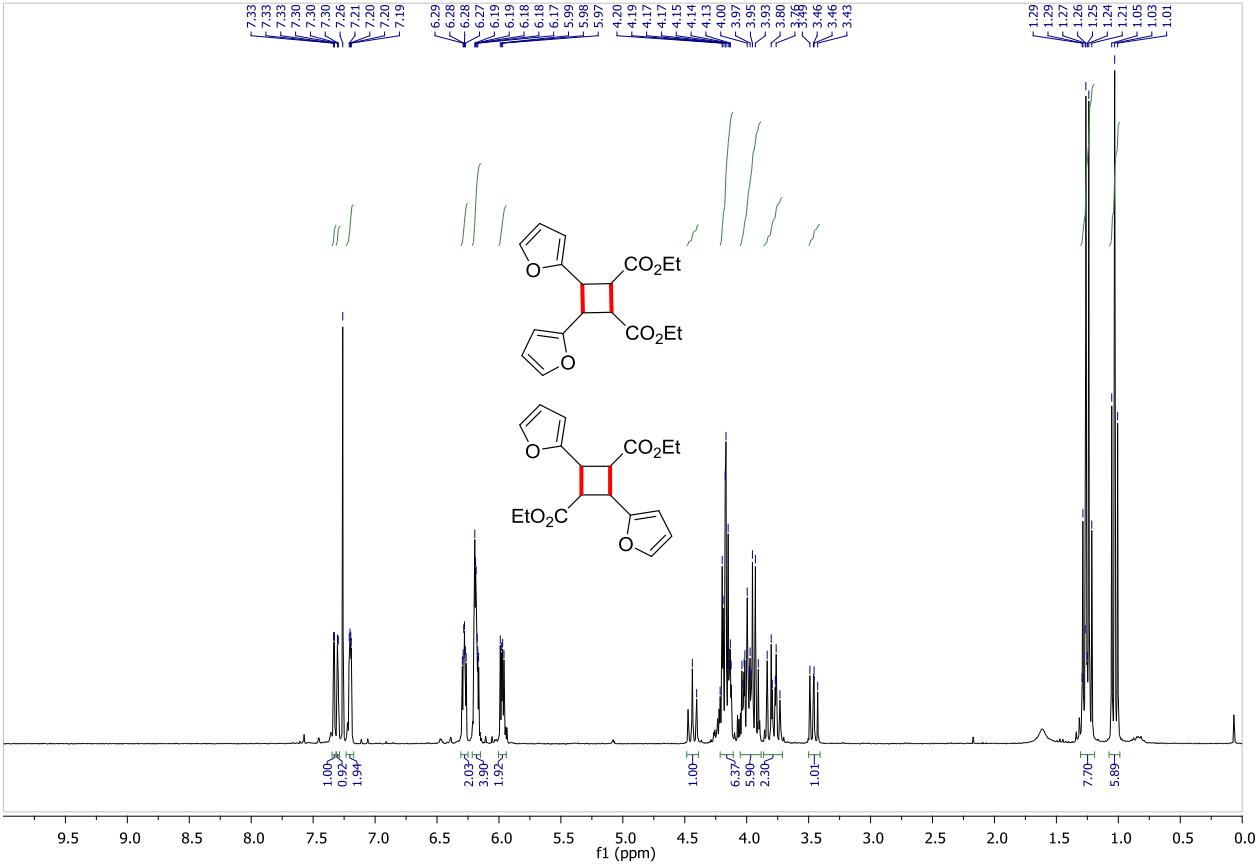
^{1}H-NMR: **2p** (*trans*, after separation)



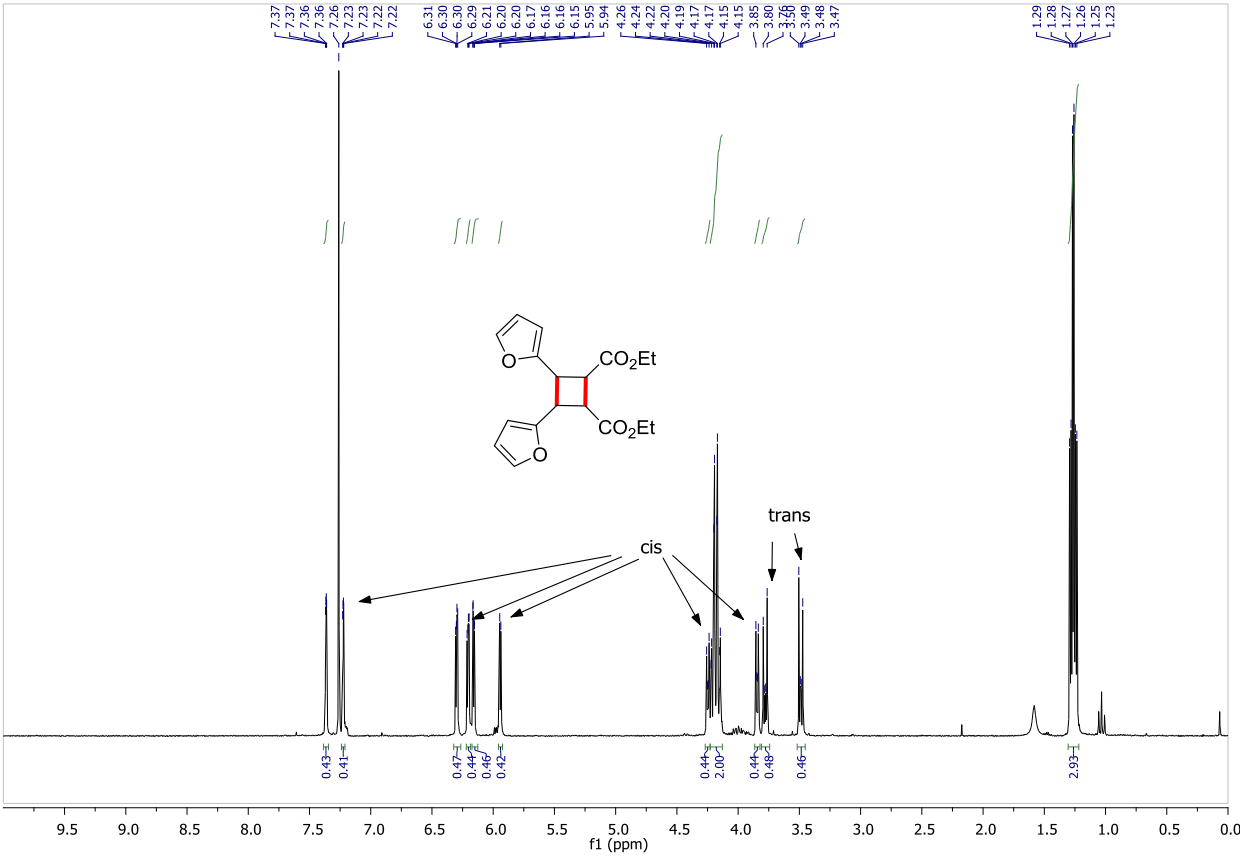
^{13}C-NMR: **2p** (*trans*, after separation)



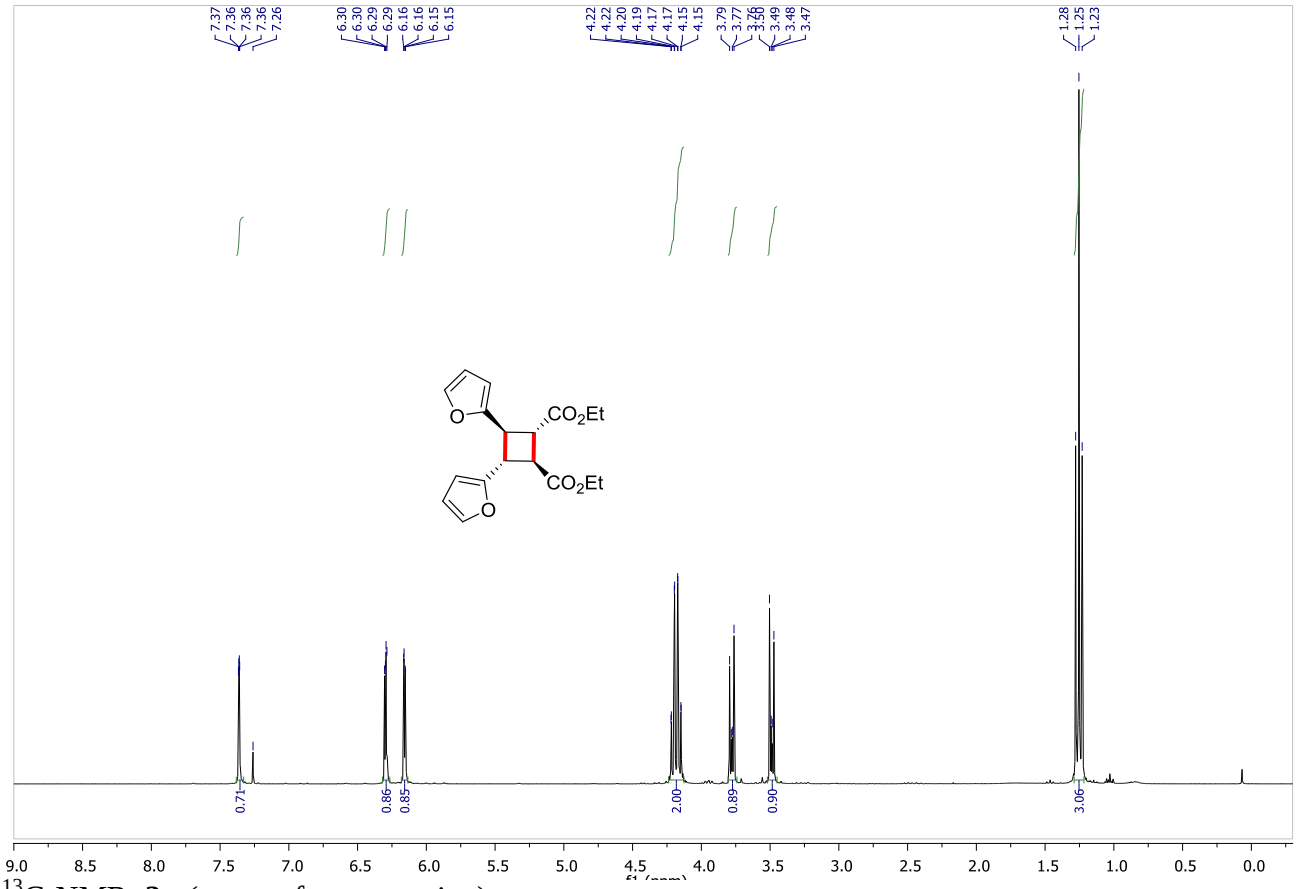
^{1}H-NMR: **2q/3q/4q** (*mixture*, before separation)



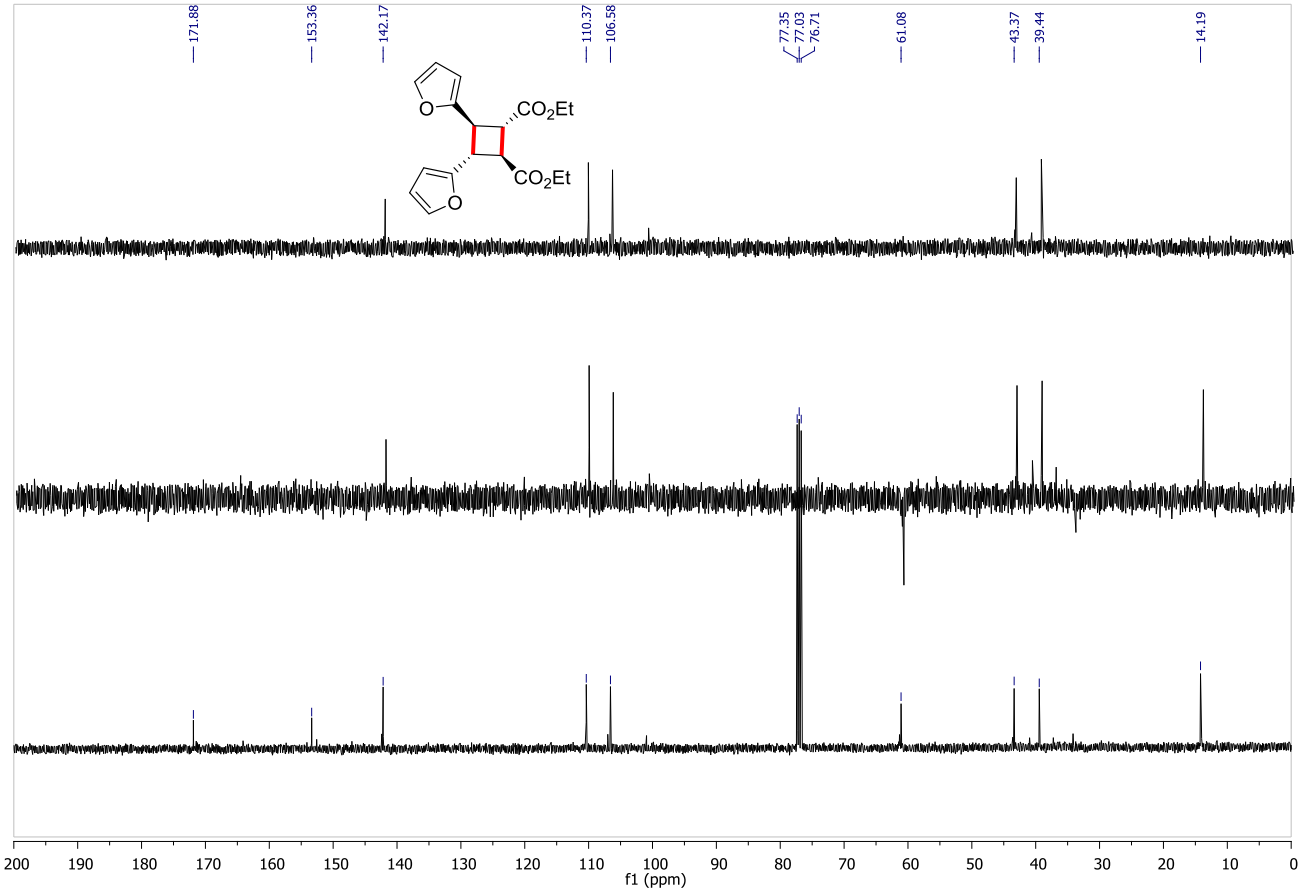
^{1}H-NMR: **2q/3q** (*mixture*, after separation)



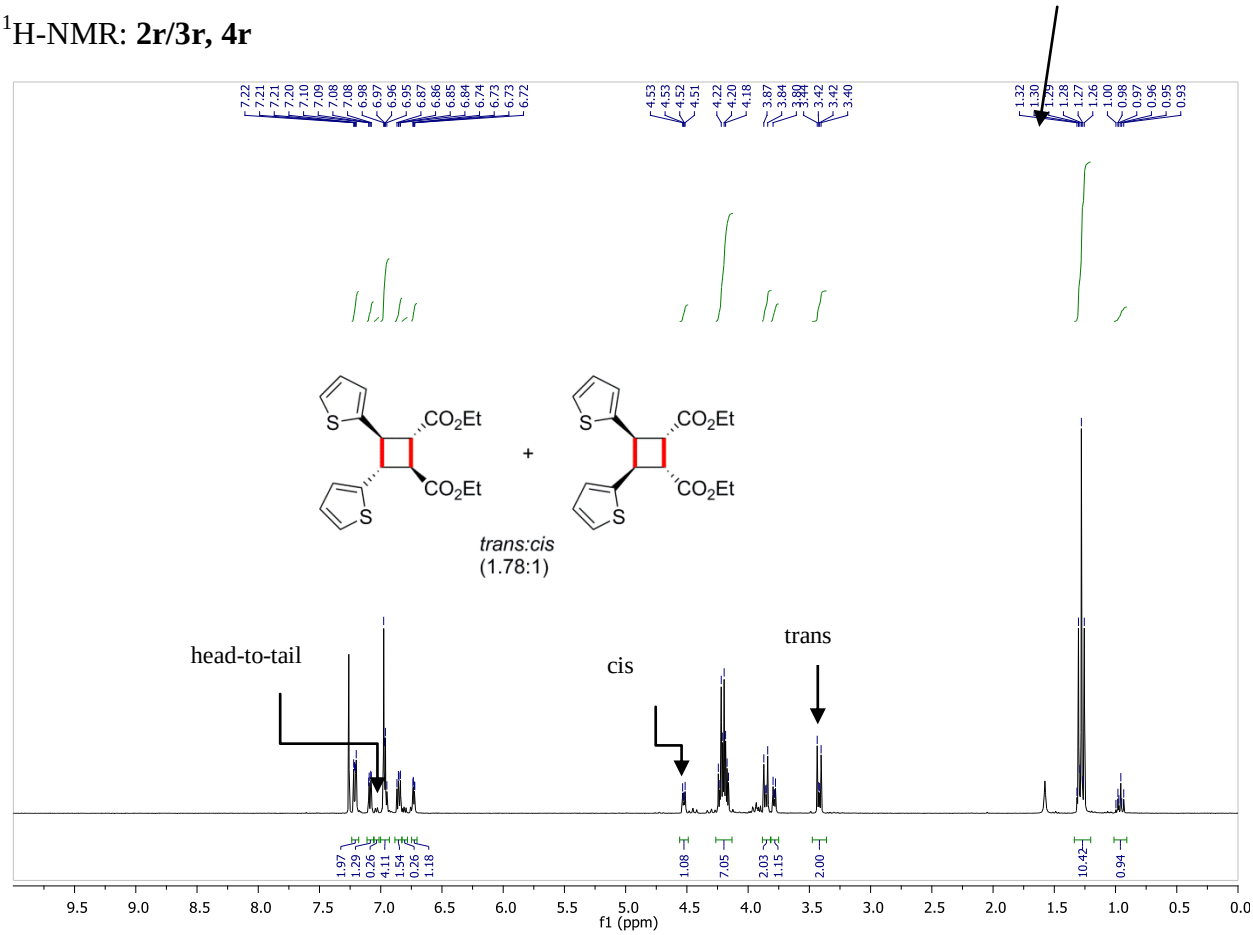
^{1}H-NMR: **2q** (*trans*, after separation)



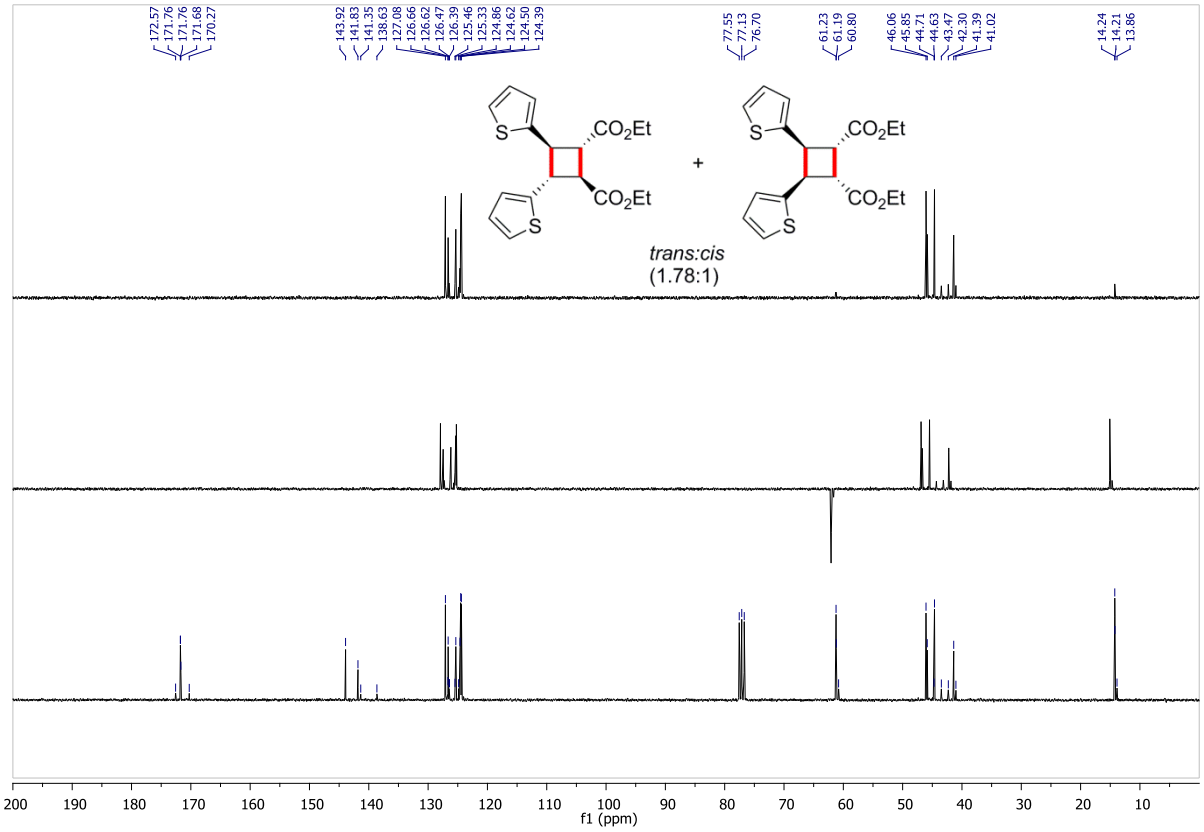
^{13}C-NMR: **2q** (*trans*, after separation)



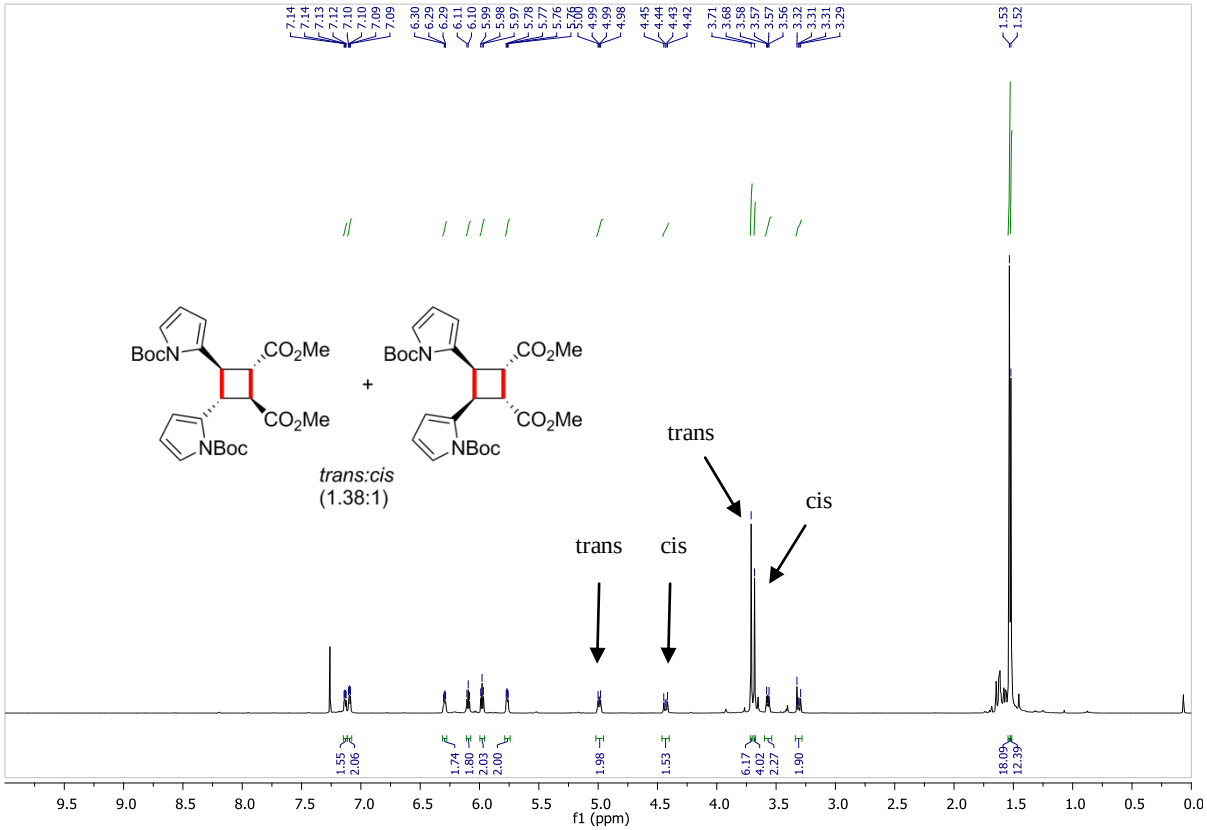
^{1}H-NMR: **2r/3r, 4r**



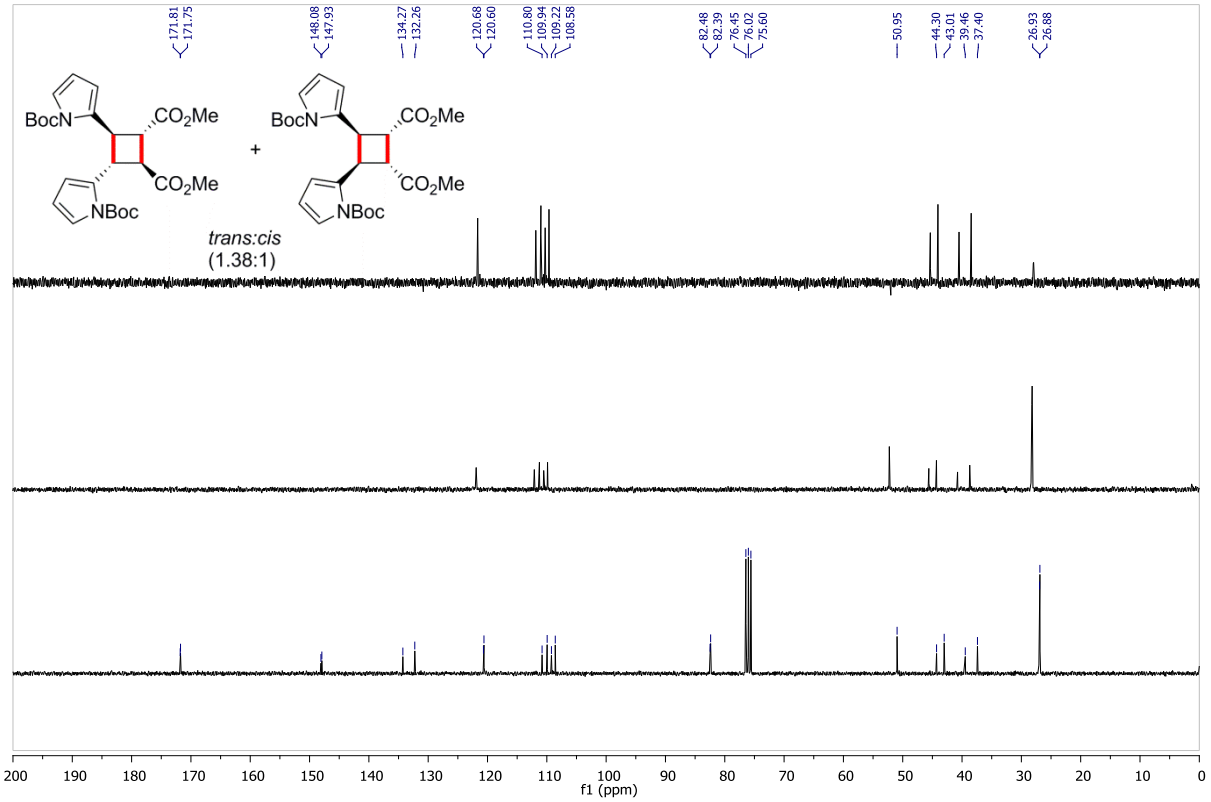
^{13}C-NMR: **2r/3r, 4r**



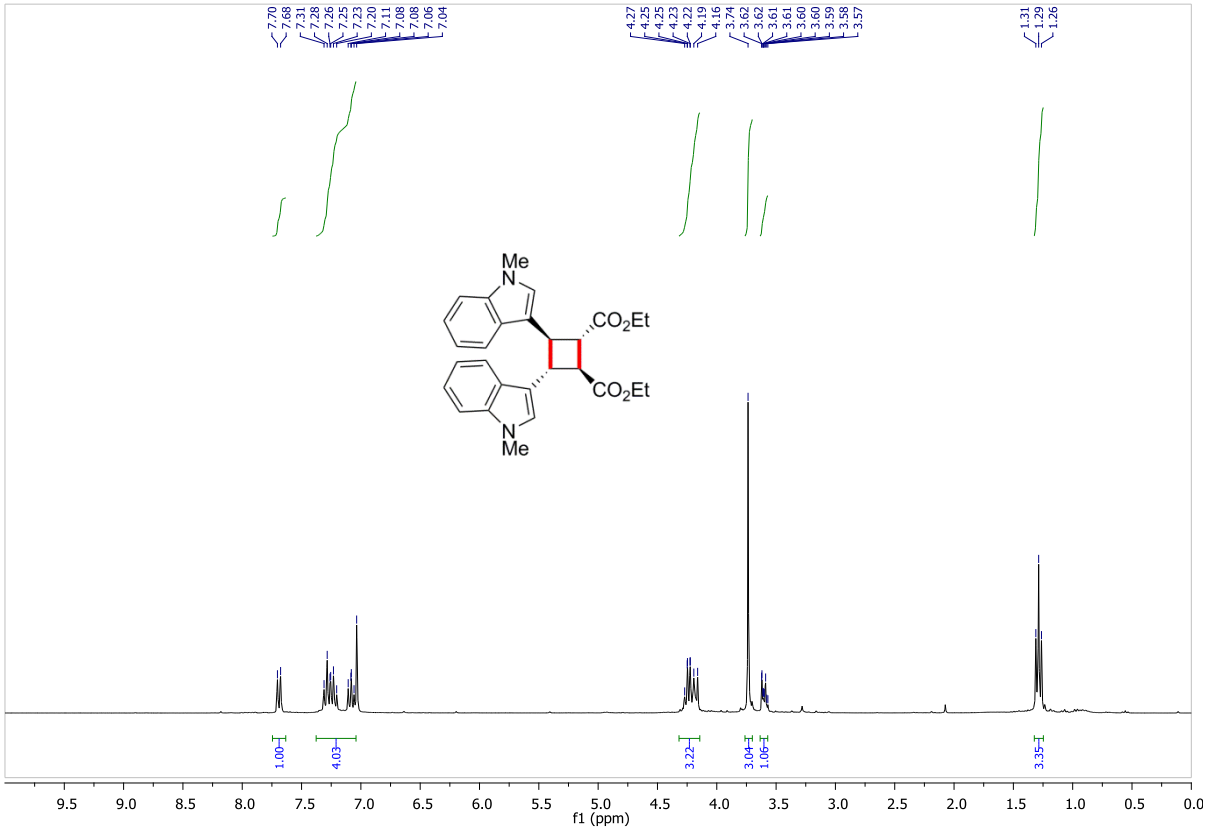
^{1}H-NMR: **2s/3s**



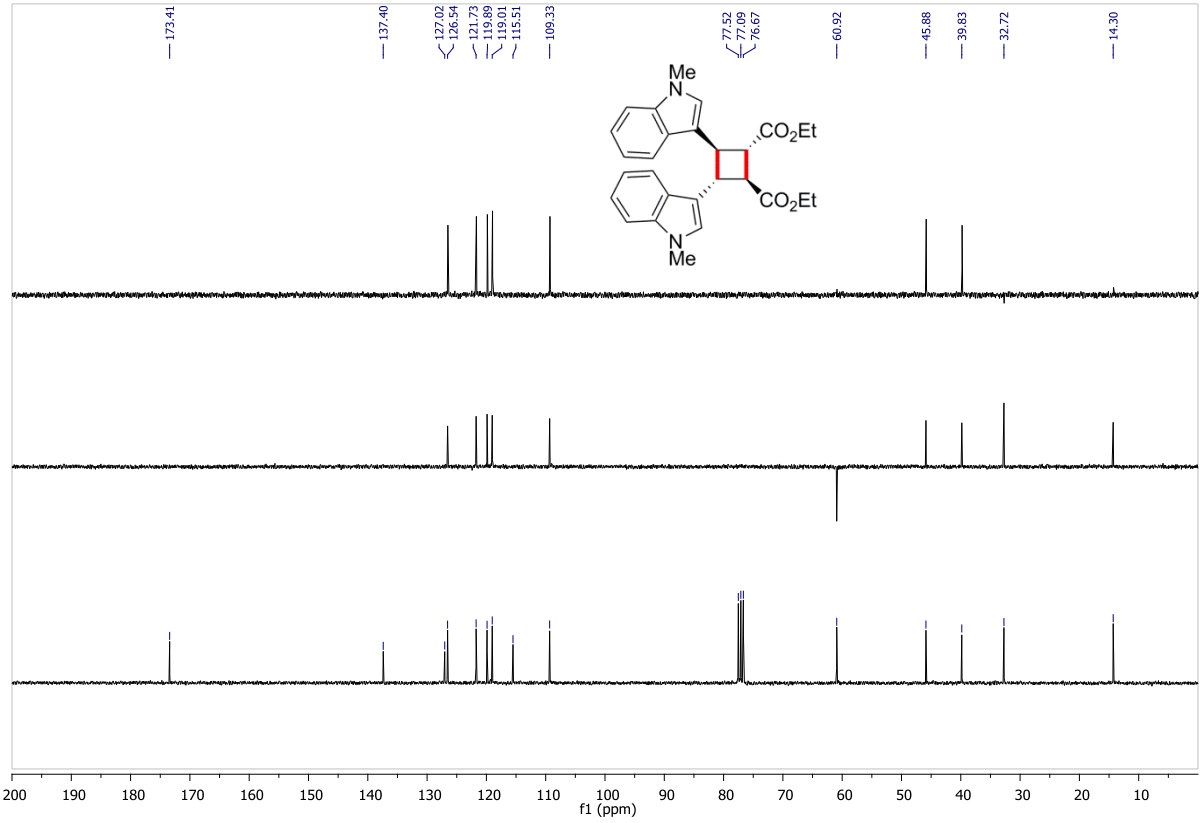
^{13}C-NMR: **2s/3s**



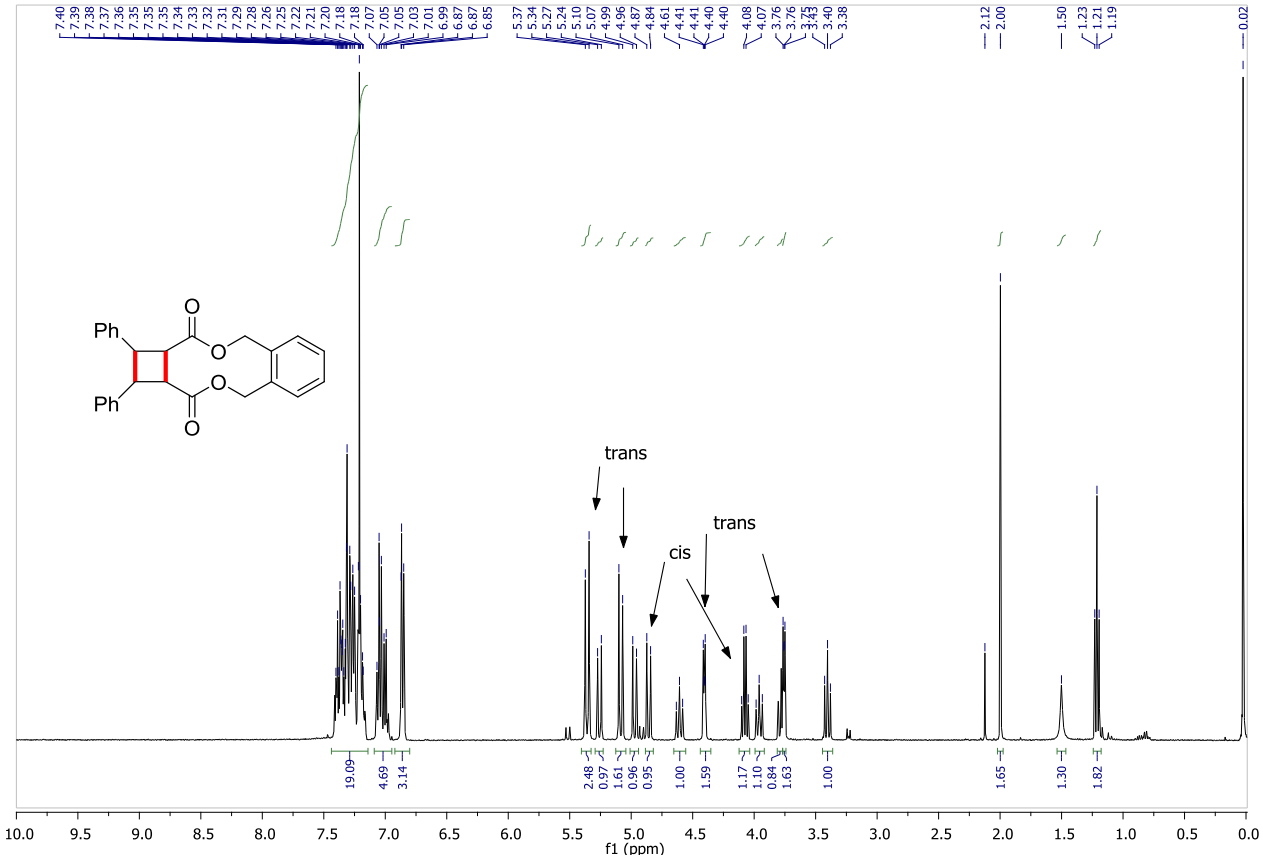
^{1}H-NMR: **2t** (*trans*, after separation)



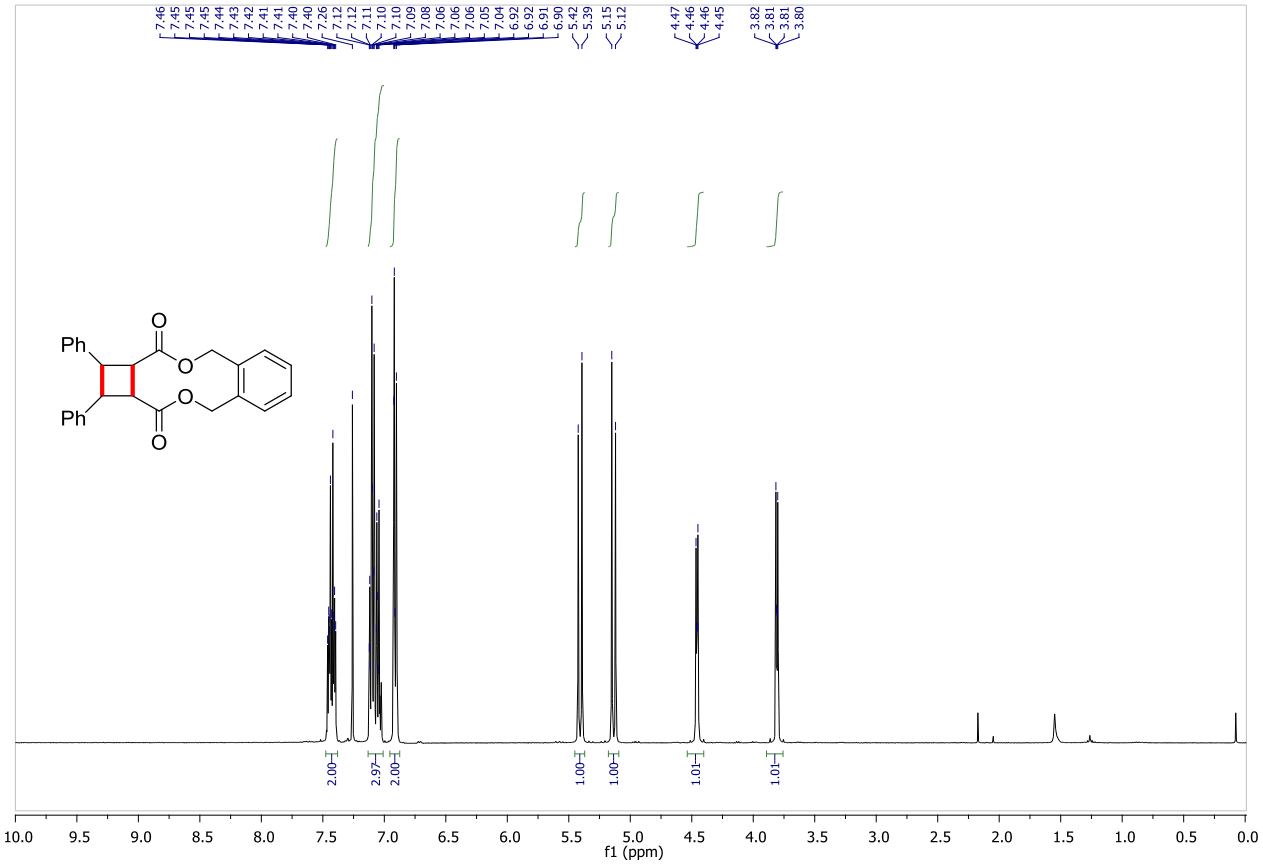
^{13}C-NMR: **2t** (*trans*, after separation)



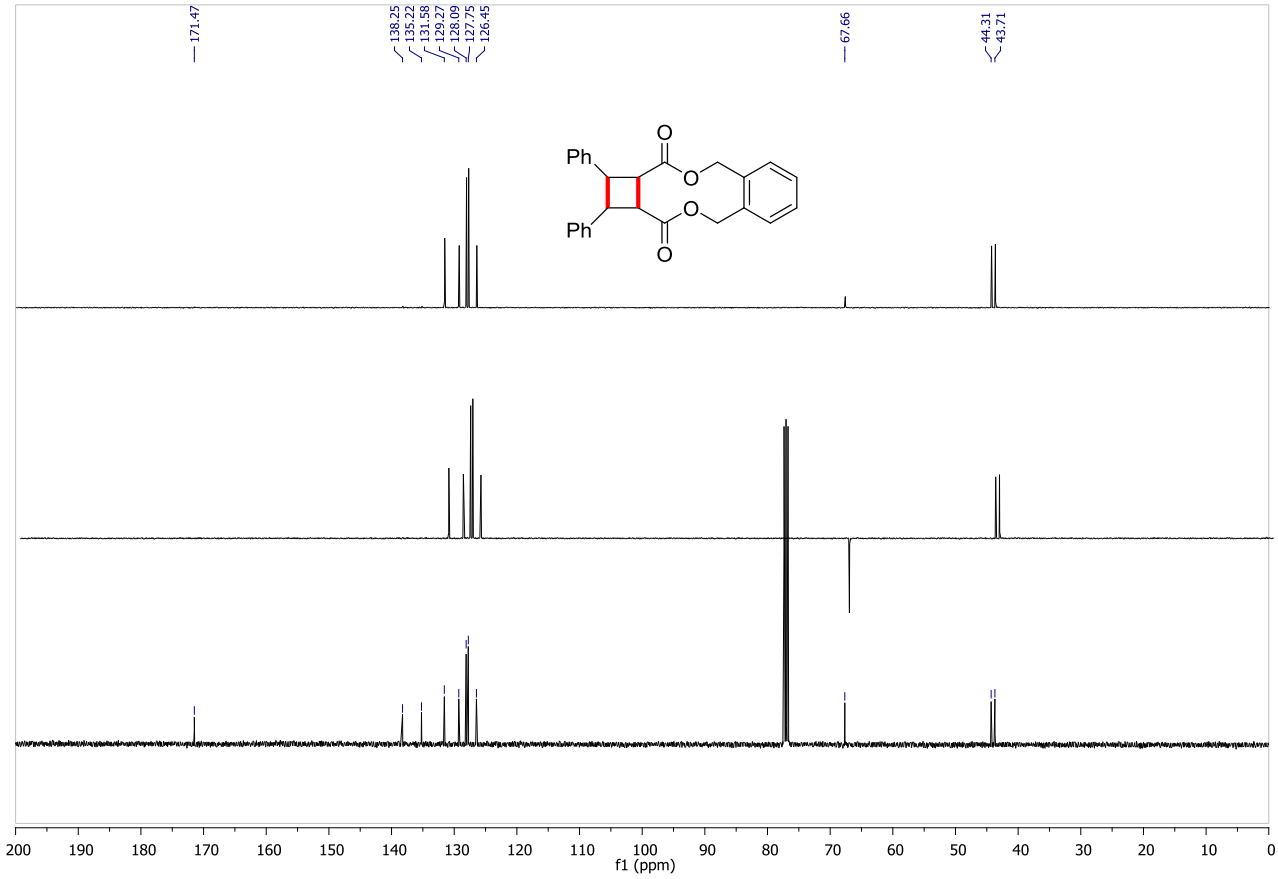
^{1}H-NMR: **2u/3u** (*cis*/*trans*)



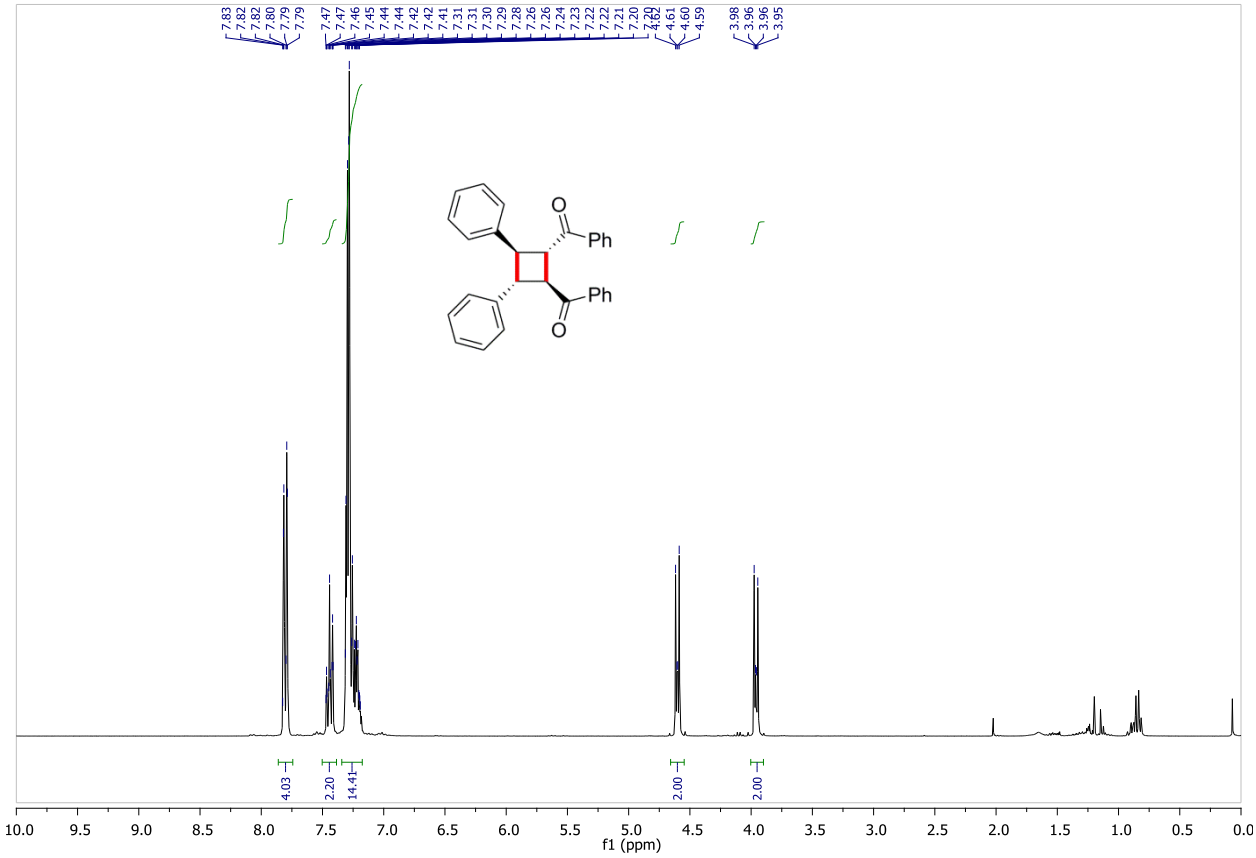
^{1}H-NMR: **2u** (*major isomer*)



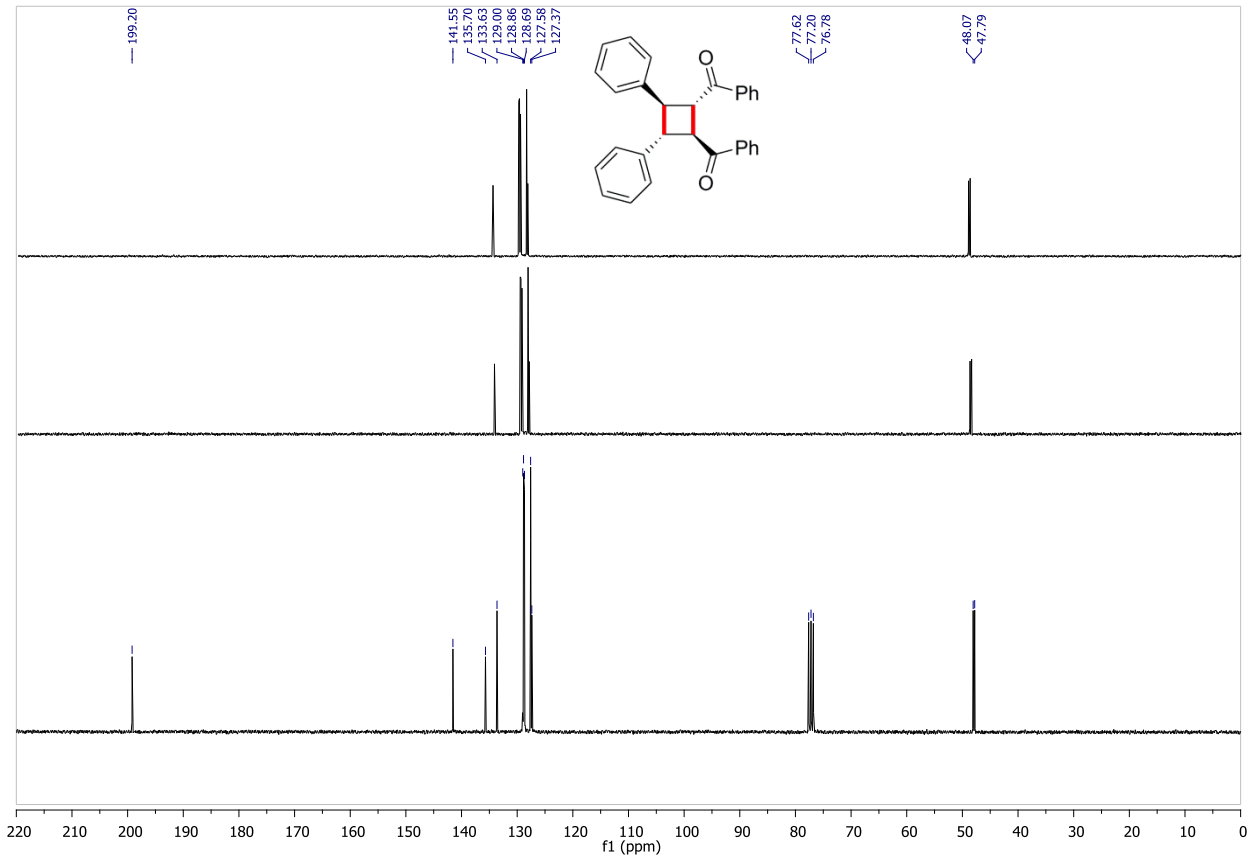
^{13}C-NMR: **2u** (*major isomer*)



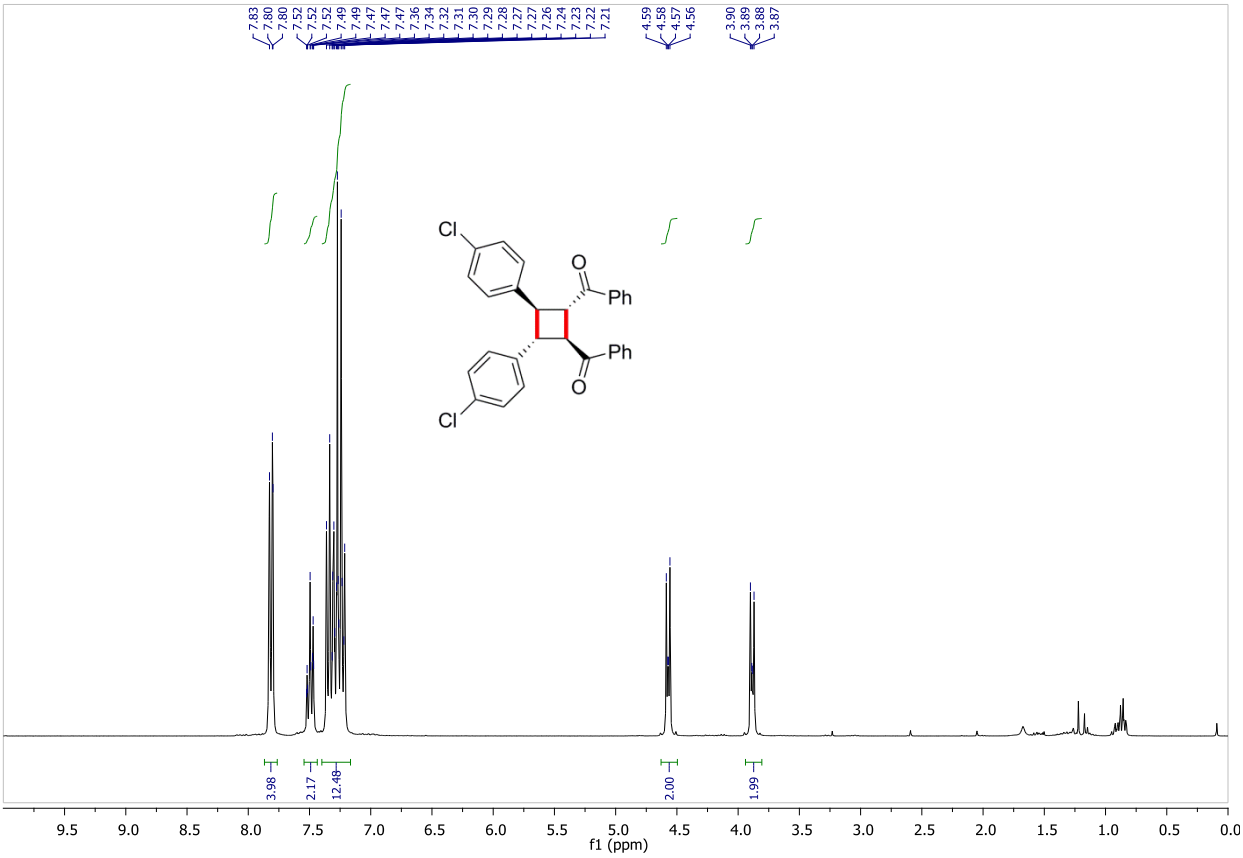
^{1}H-NMR: **2v** (*trans*, after separation)



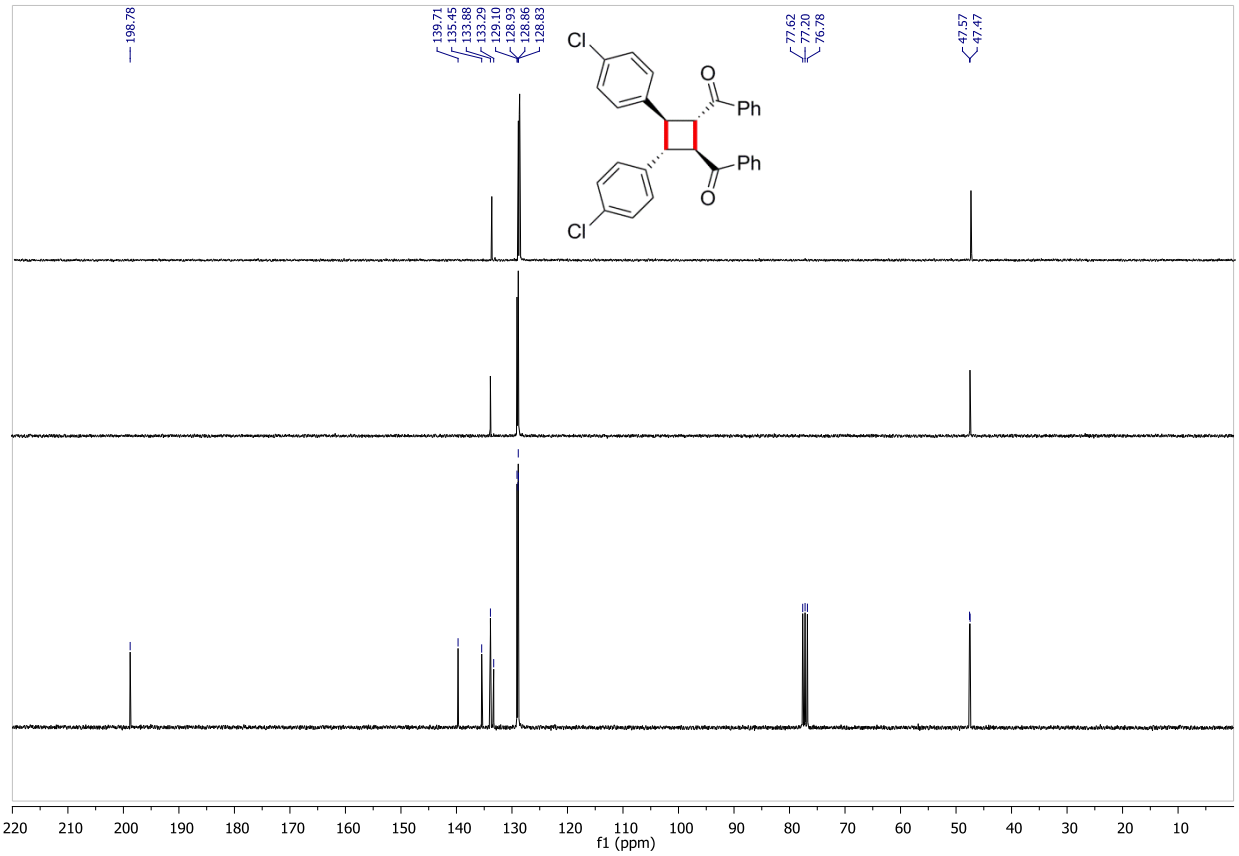
^{13}C-NMR: **2v** (*trans*, after separation)



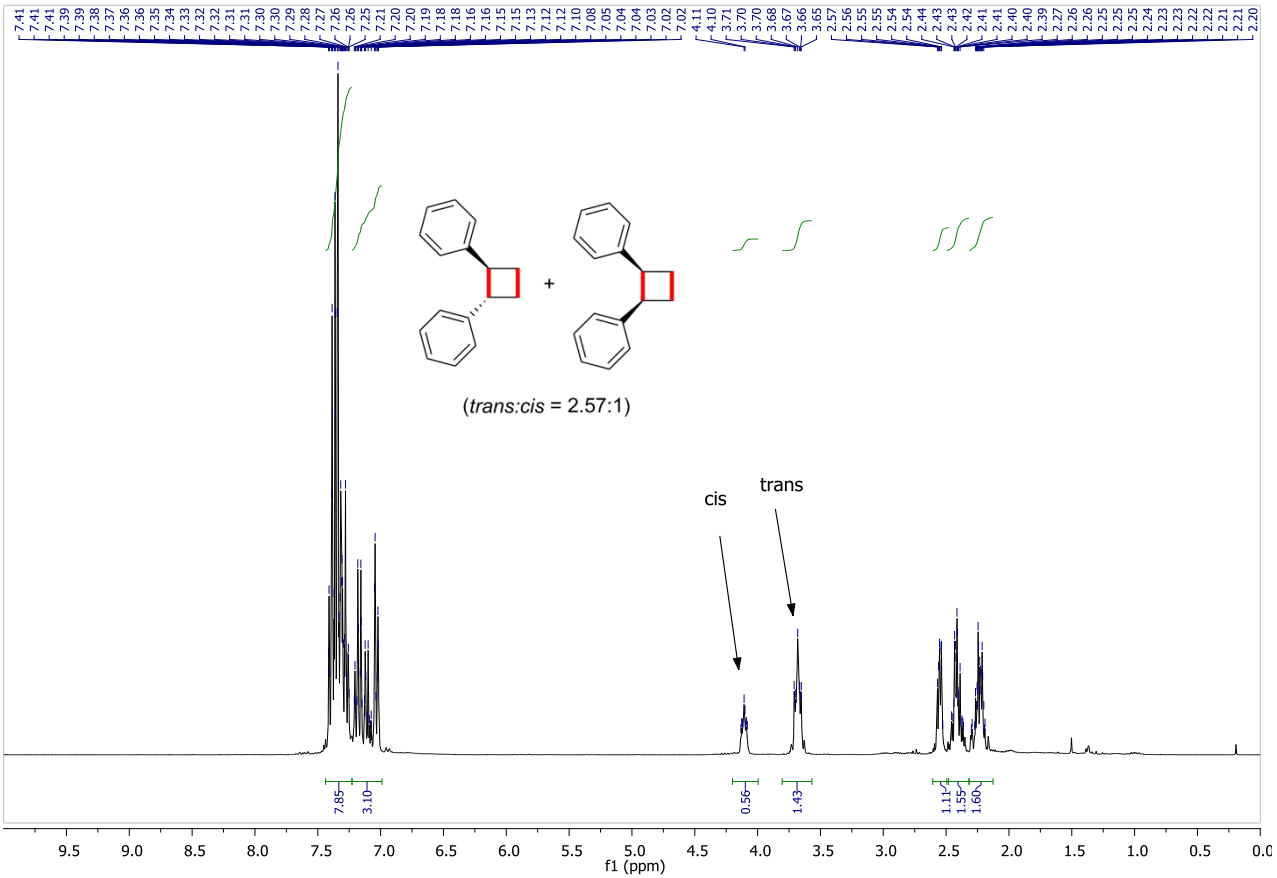
^{1}H-NMR: **2w** (*trans*, after separation)



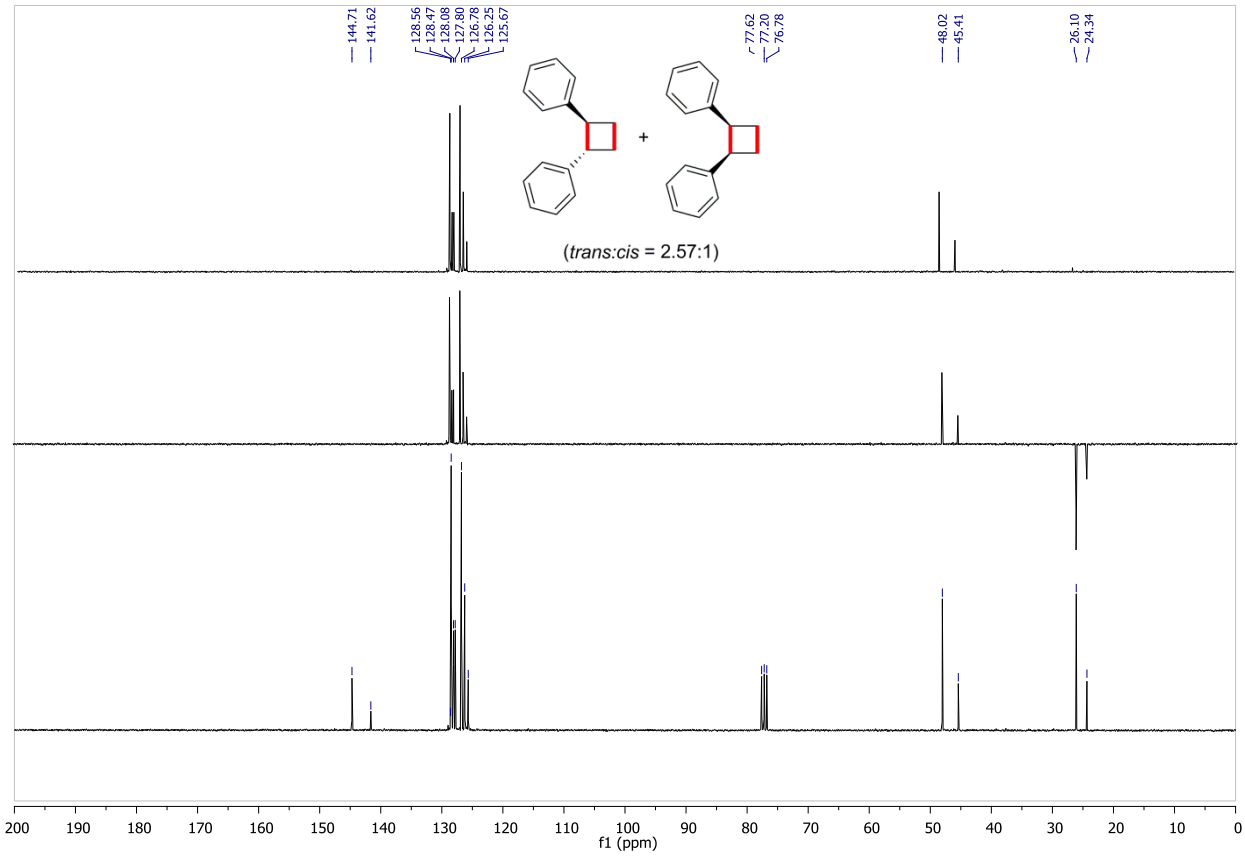
^{13}C-NMR: **2w** (*trans*, after separation)



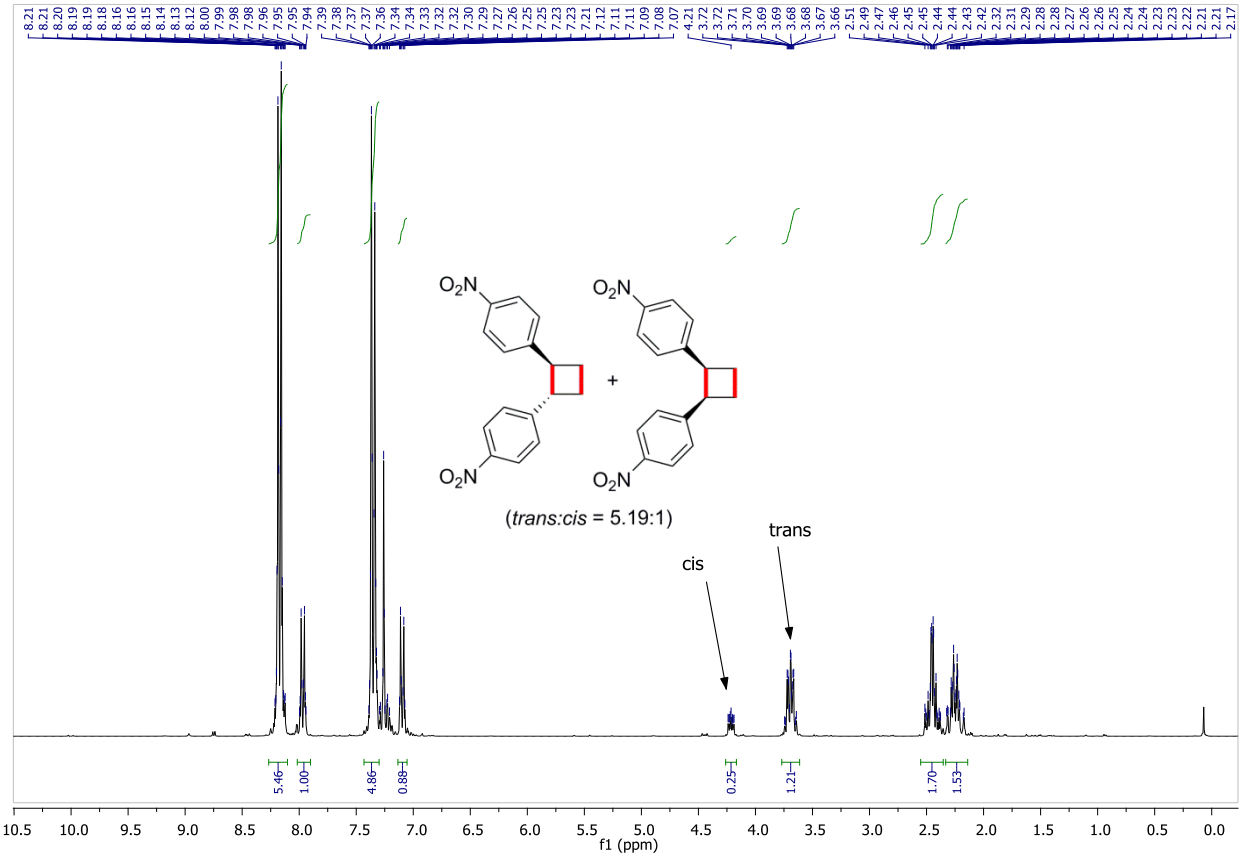
^{1}H-NMR: **6a** (*cis*/*trans*)



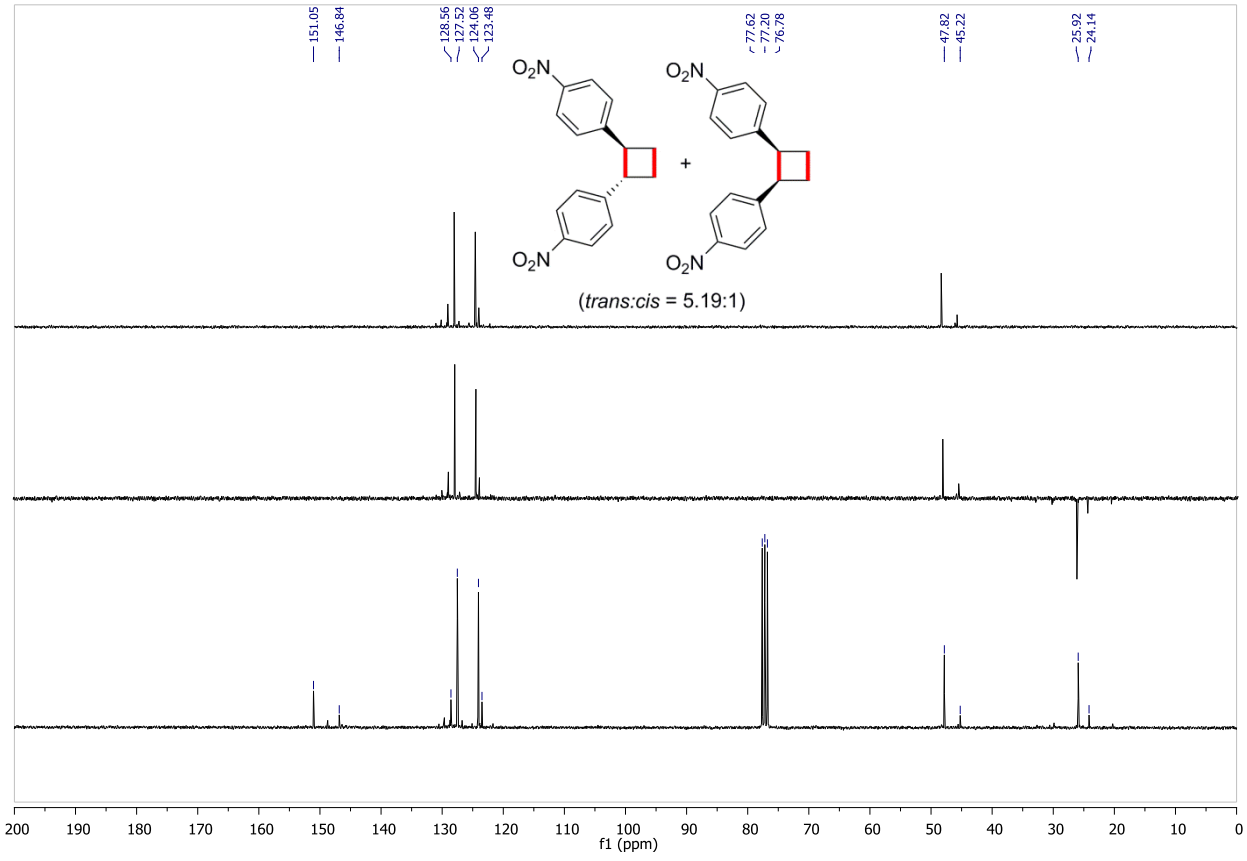
^{13}C-NMR: **6a** (*cis*/*trans*)



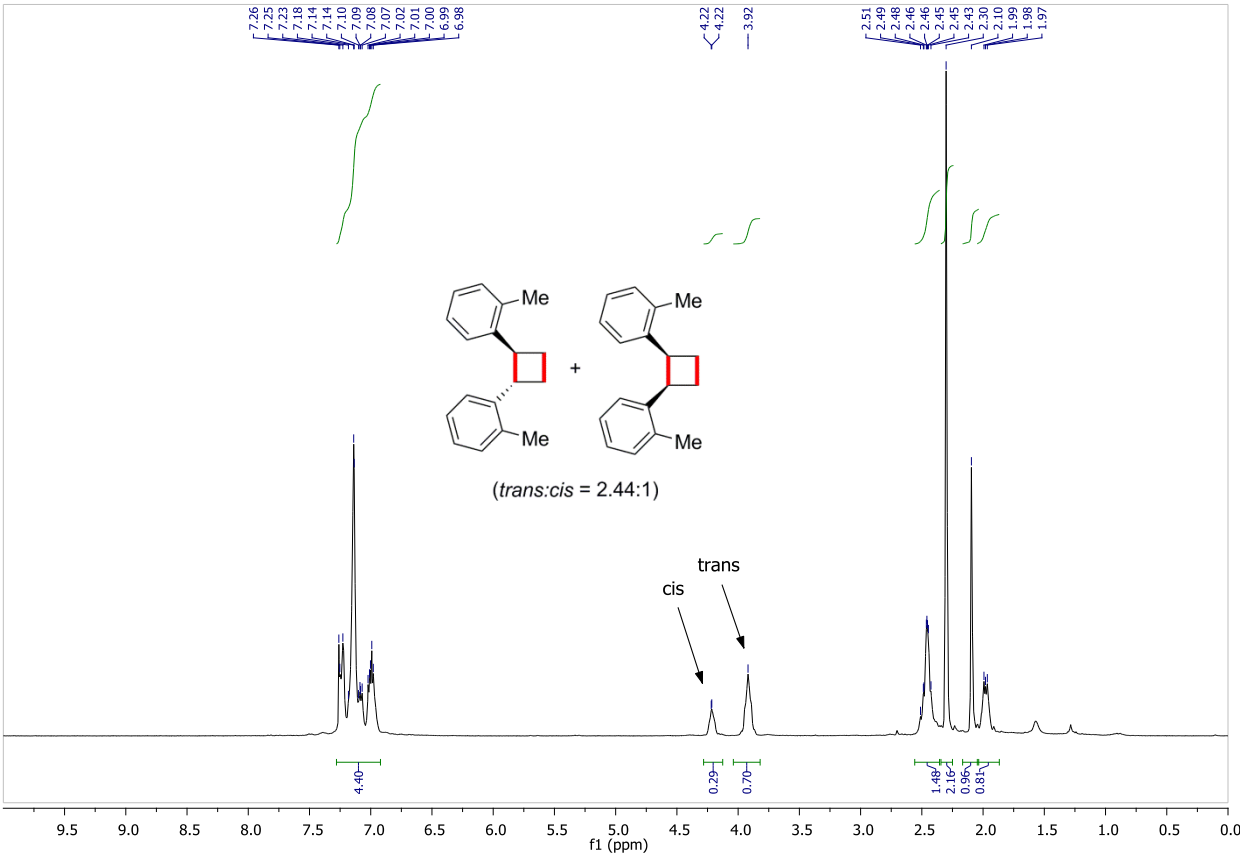
^{1}H-NMR: **6b** (*cis*/*trans*)



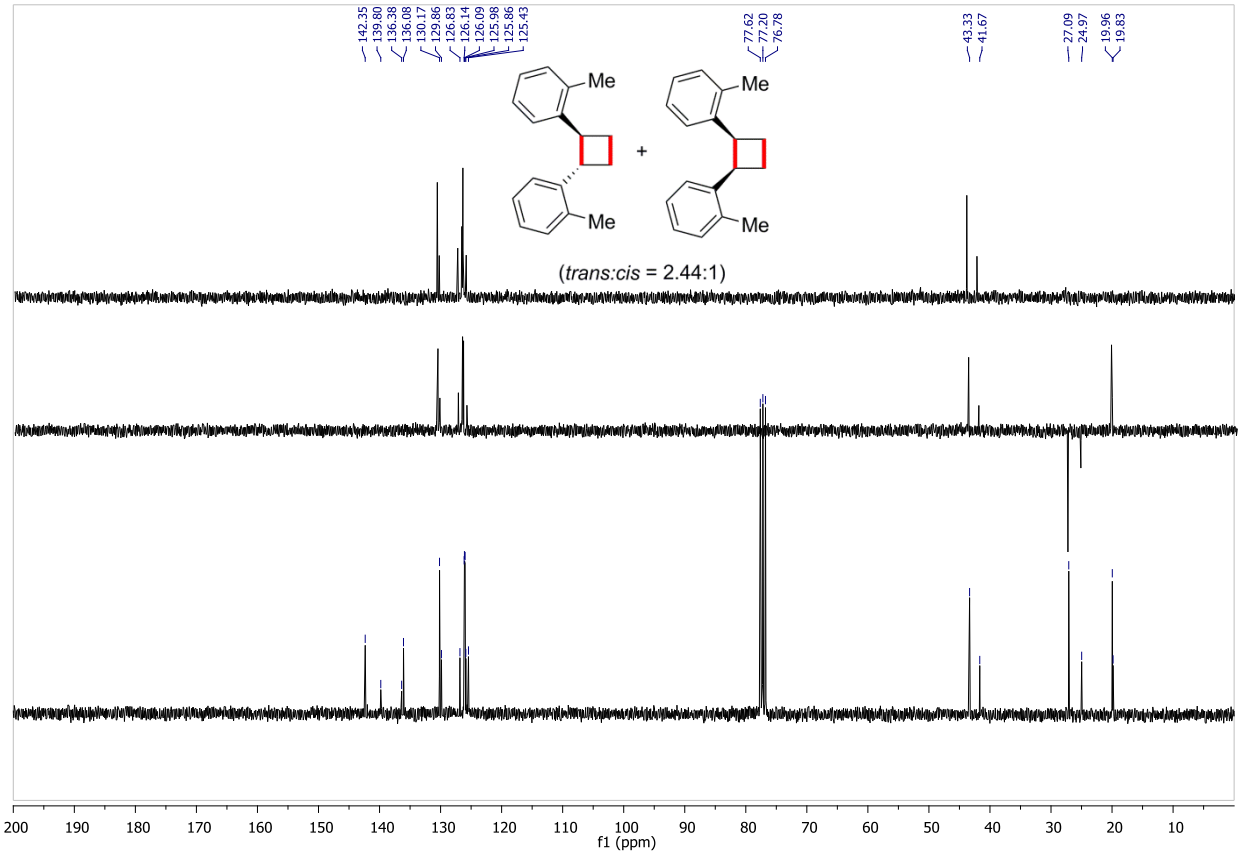
^{13}C-NMR: **6b** (*cis*/*trans*)



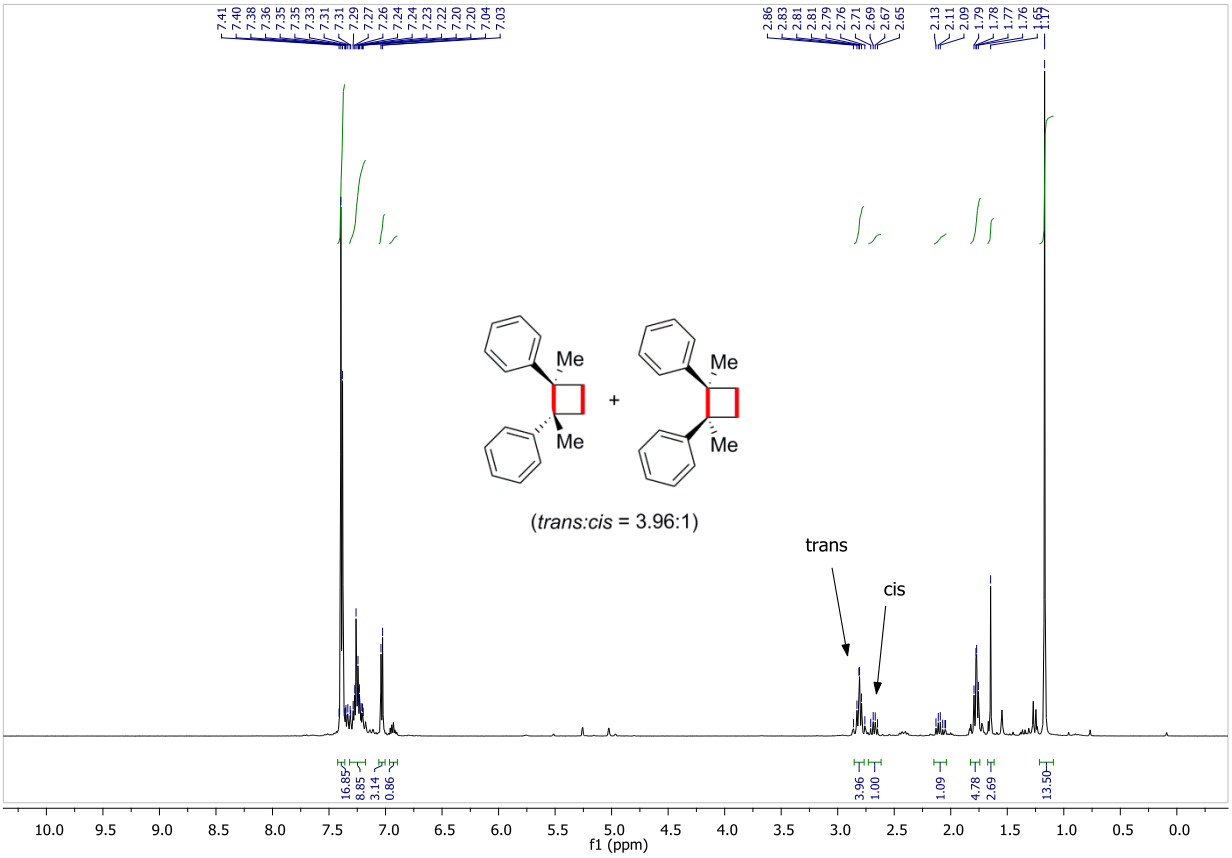
^{1}H-NMR: **6c** (*cis*/*trans*)



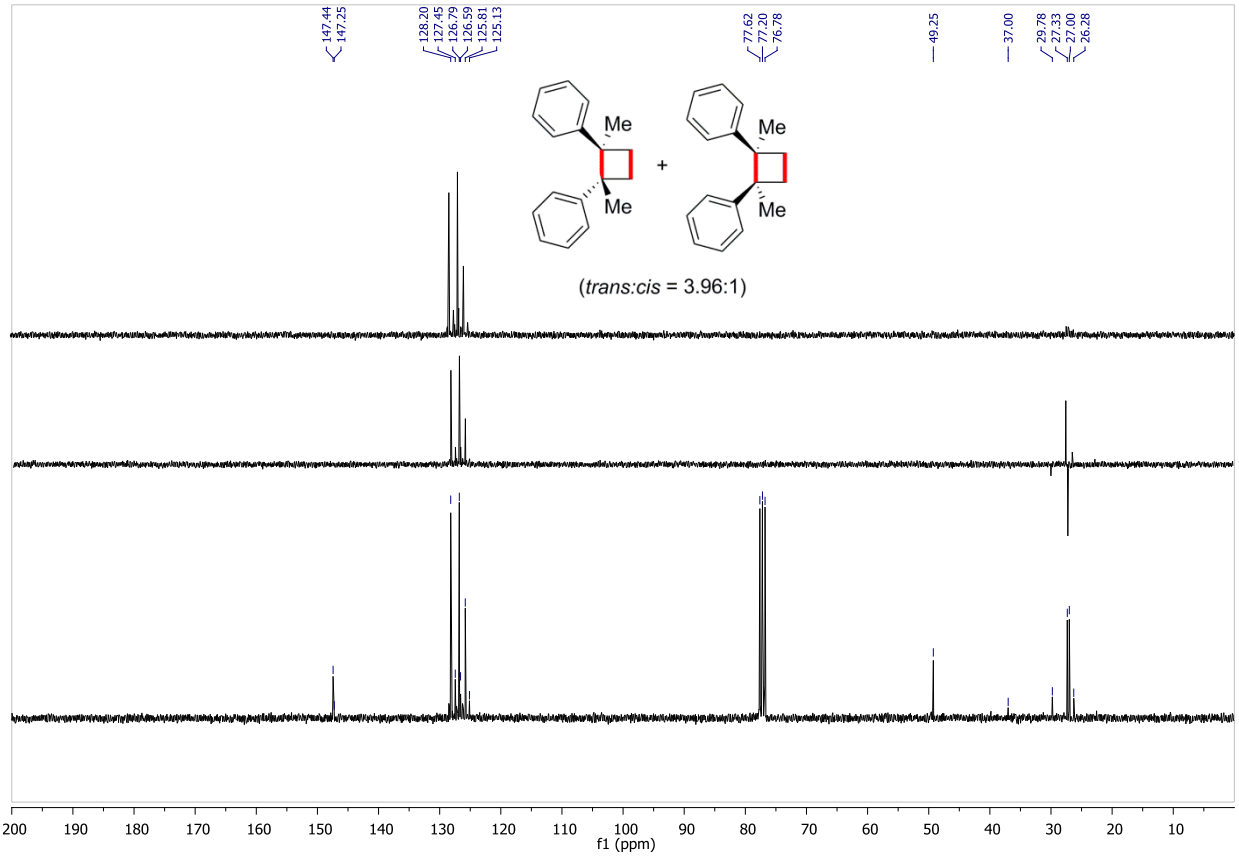
^{13}C-NMR: **6c** (*cis*/*trans*)



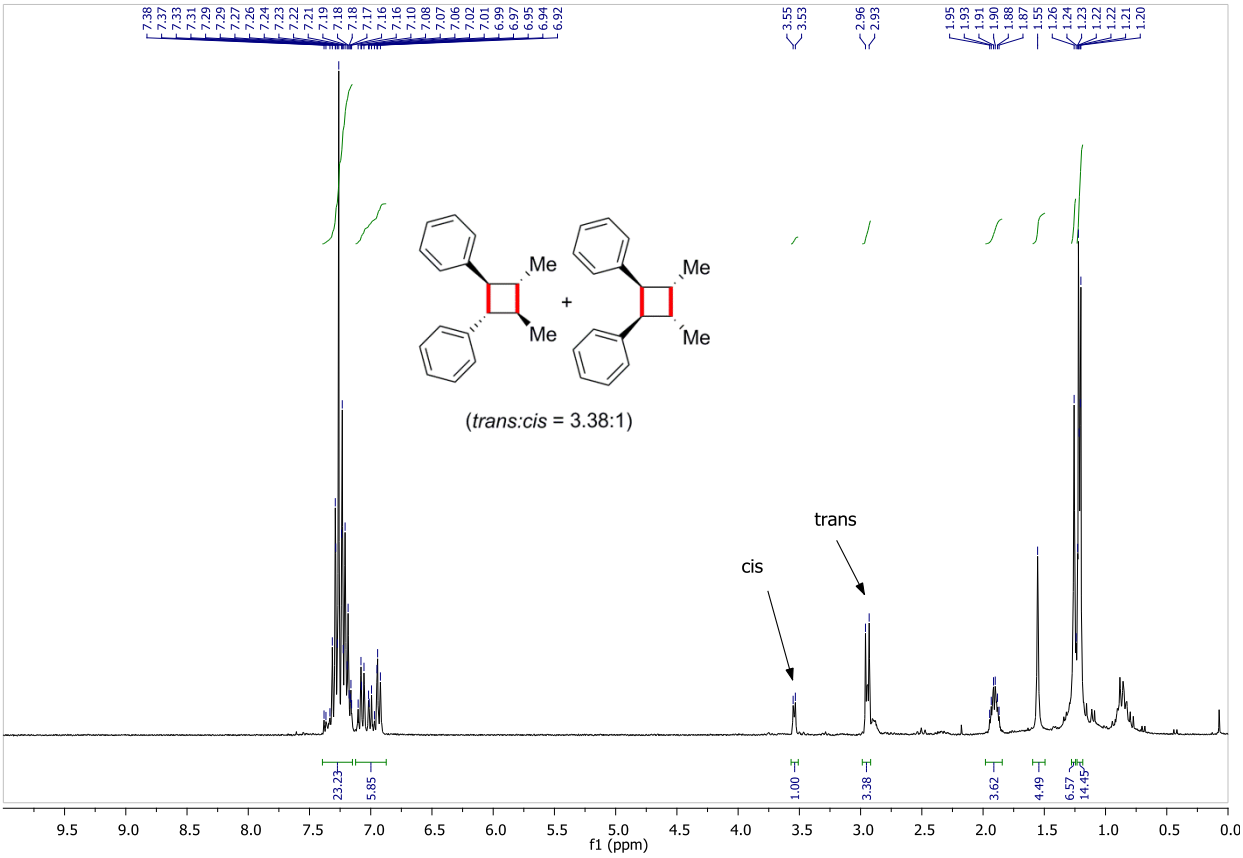
^{1}H-NMR: **6d** (*cis*/*trans*)



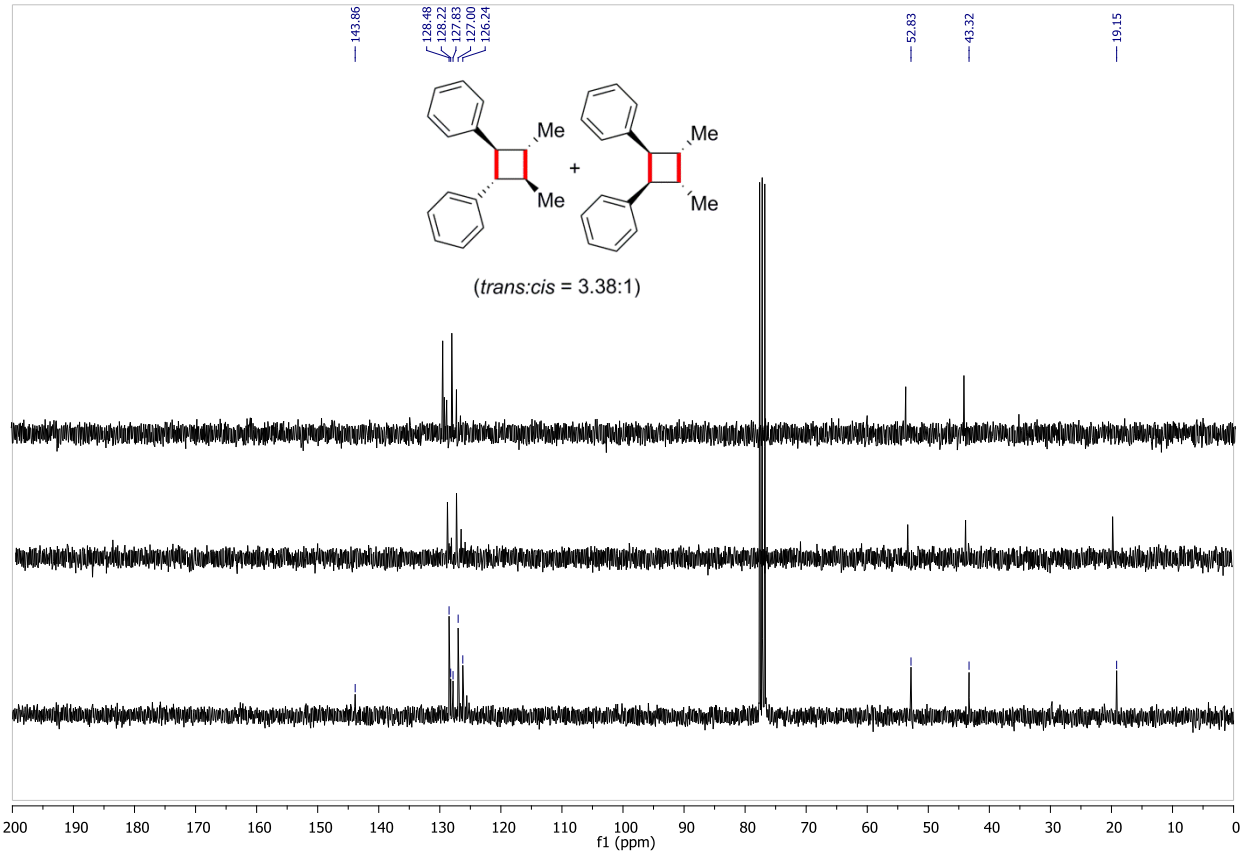
^{13}C-NMR: **6d** (*cis*/*trans*)



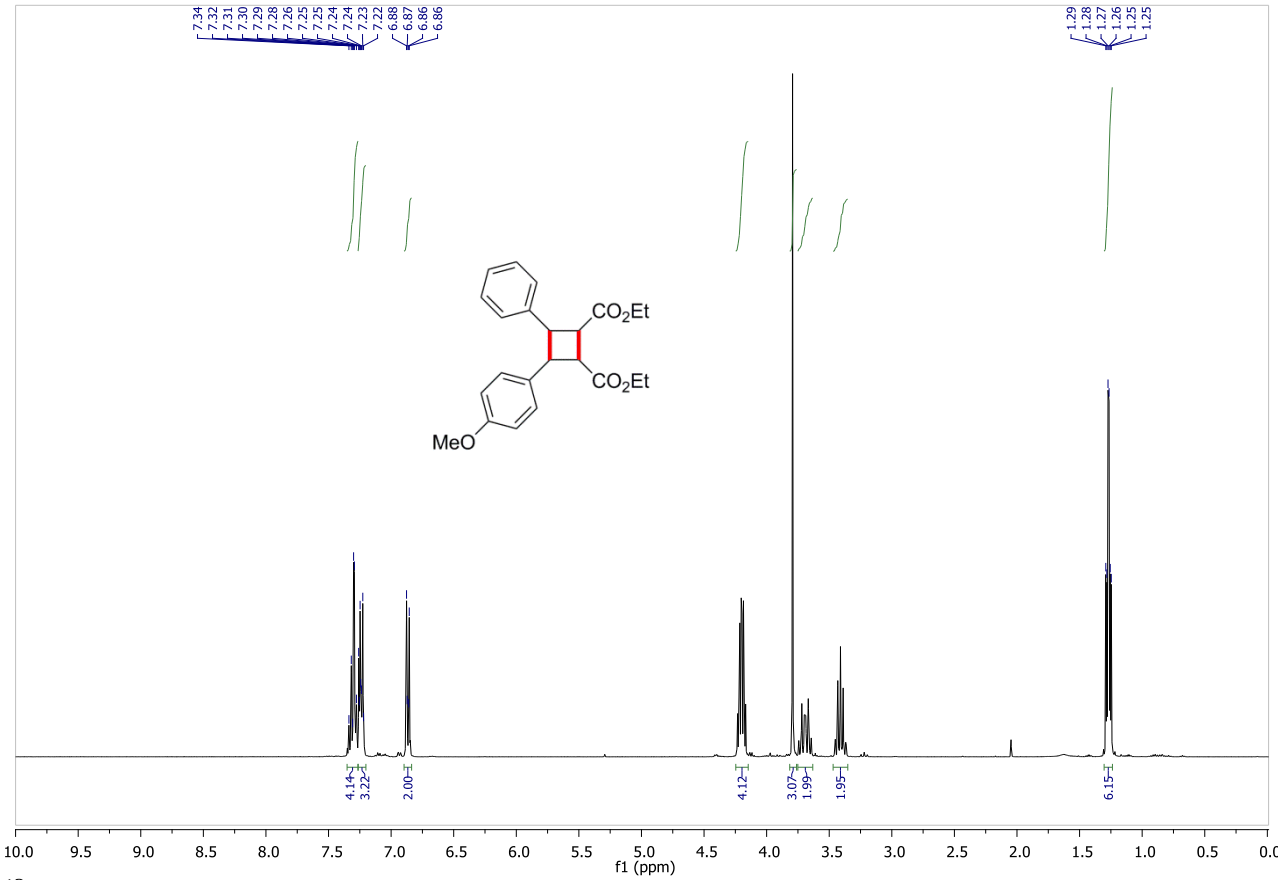
^{1}H-NMR: **6e** (*cis*/*trans*)



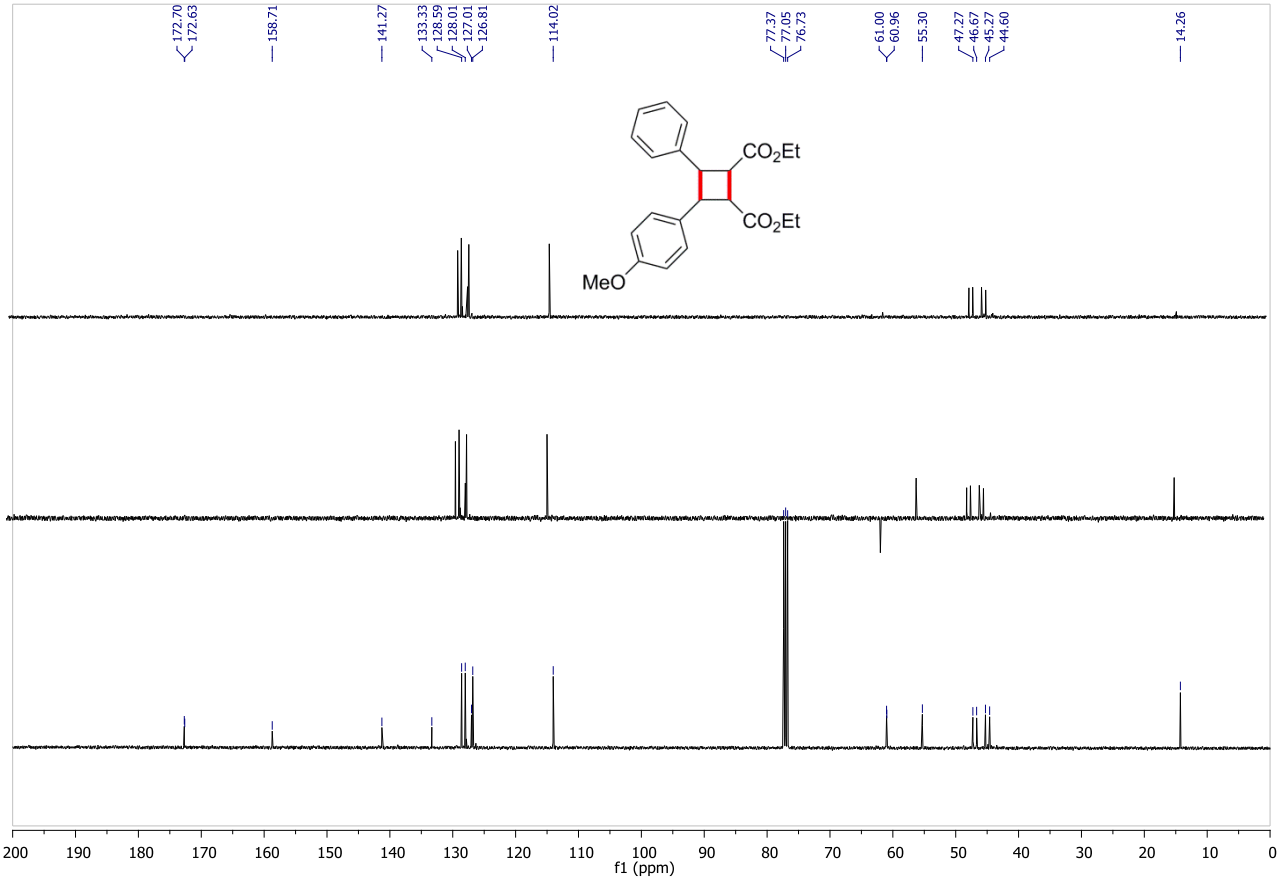
^{13}C-NMR: **6e** (*cis*/*trans*)



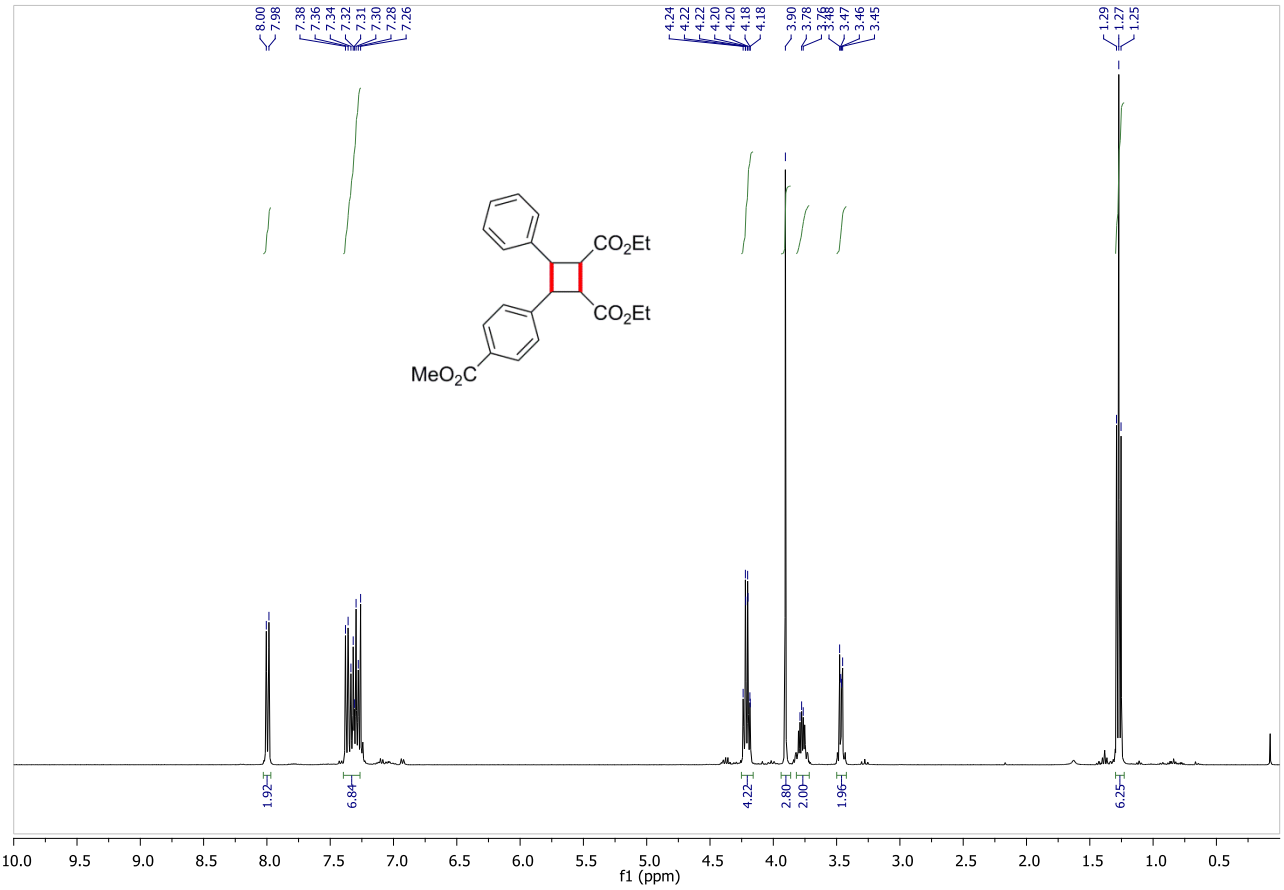
^{1}H-NMR: **7a** (*trans*)



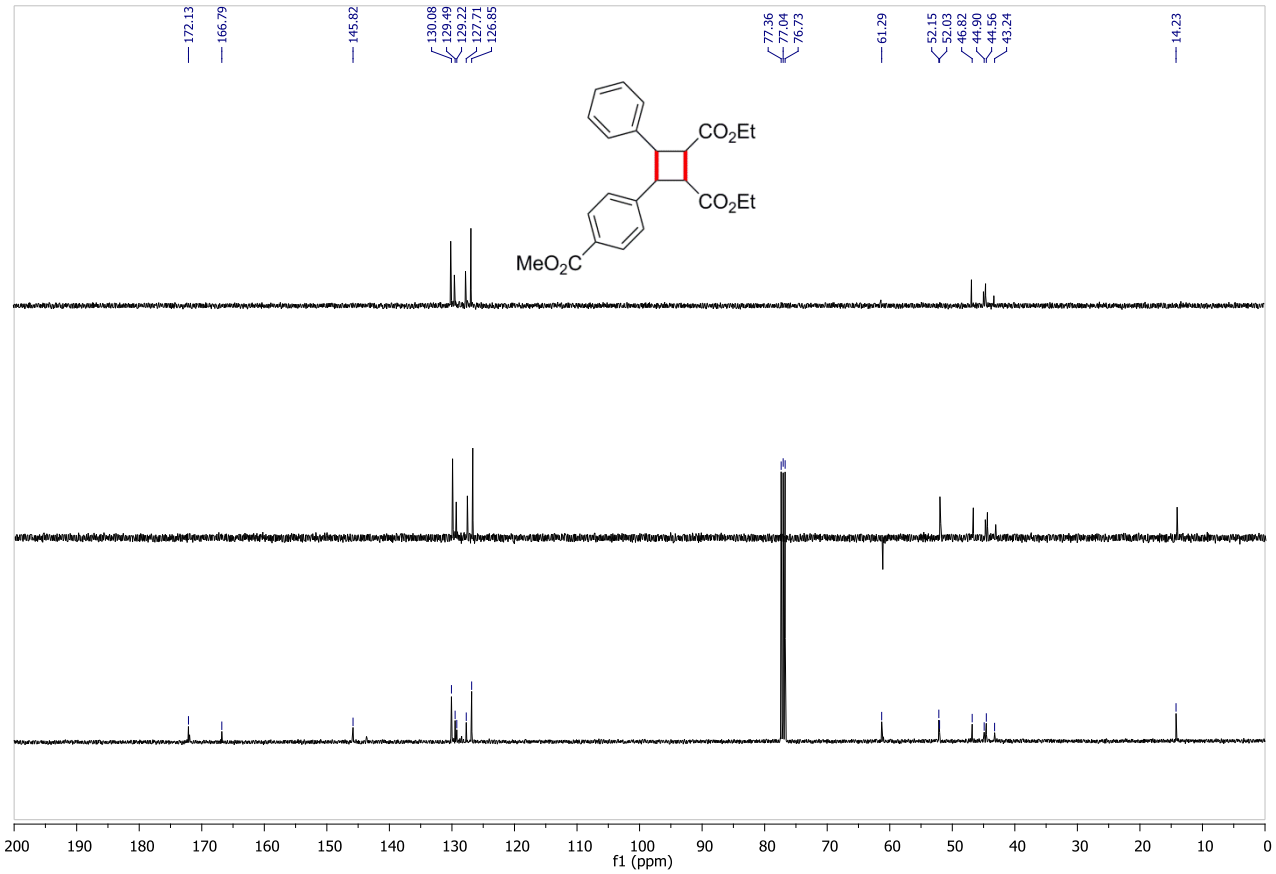
^{13}C-NMR: **7a** (*trans*)



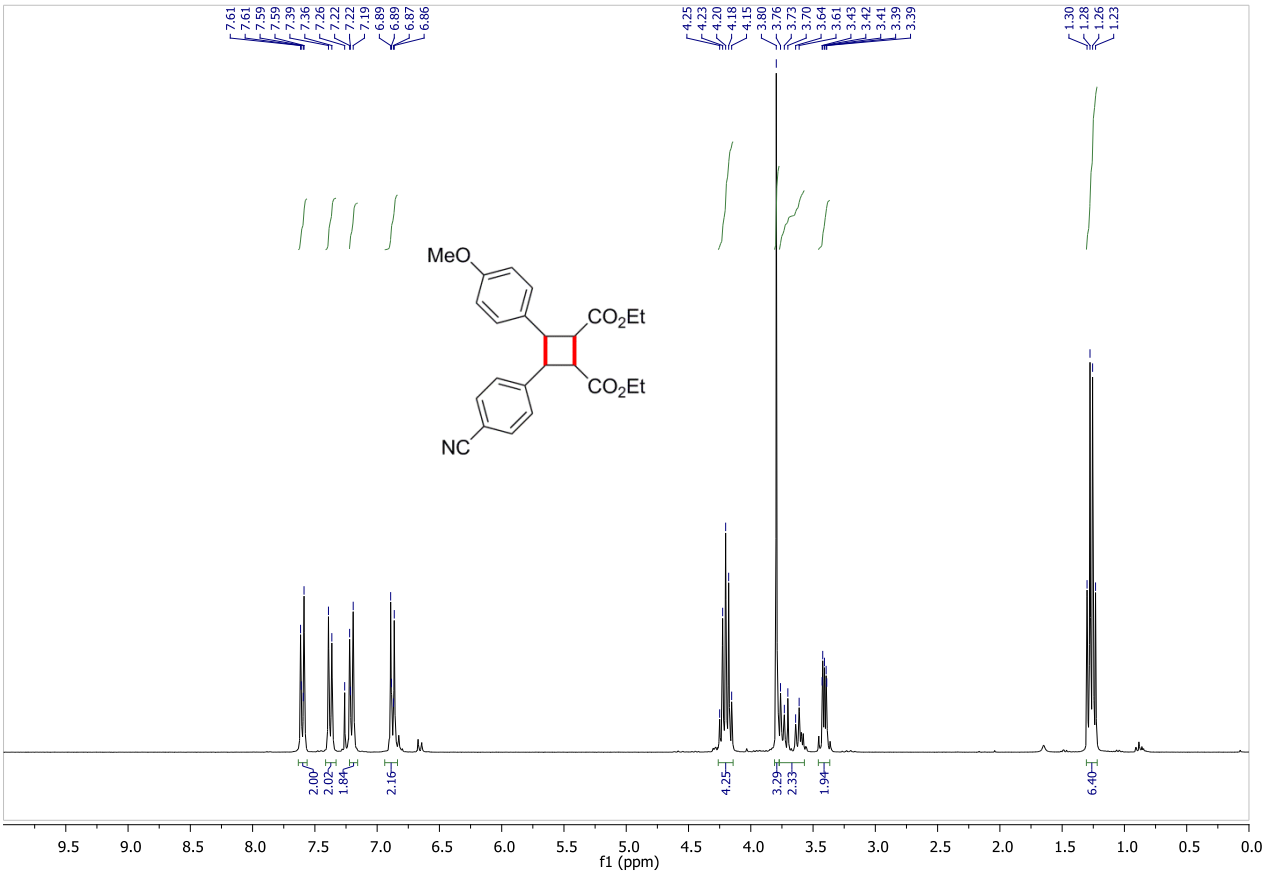
^{1}H-NMR: **7b** (*trans*)



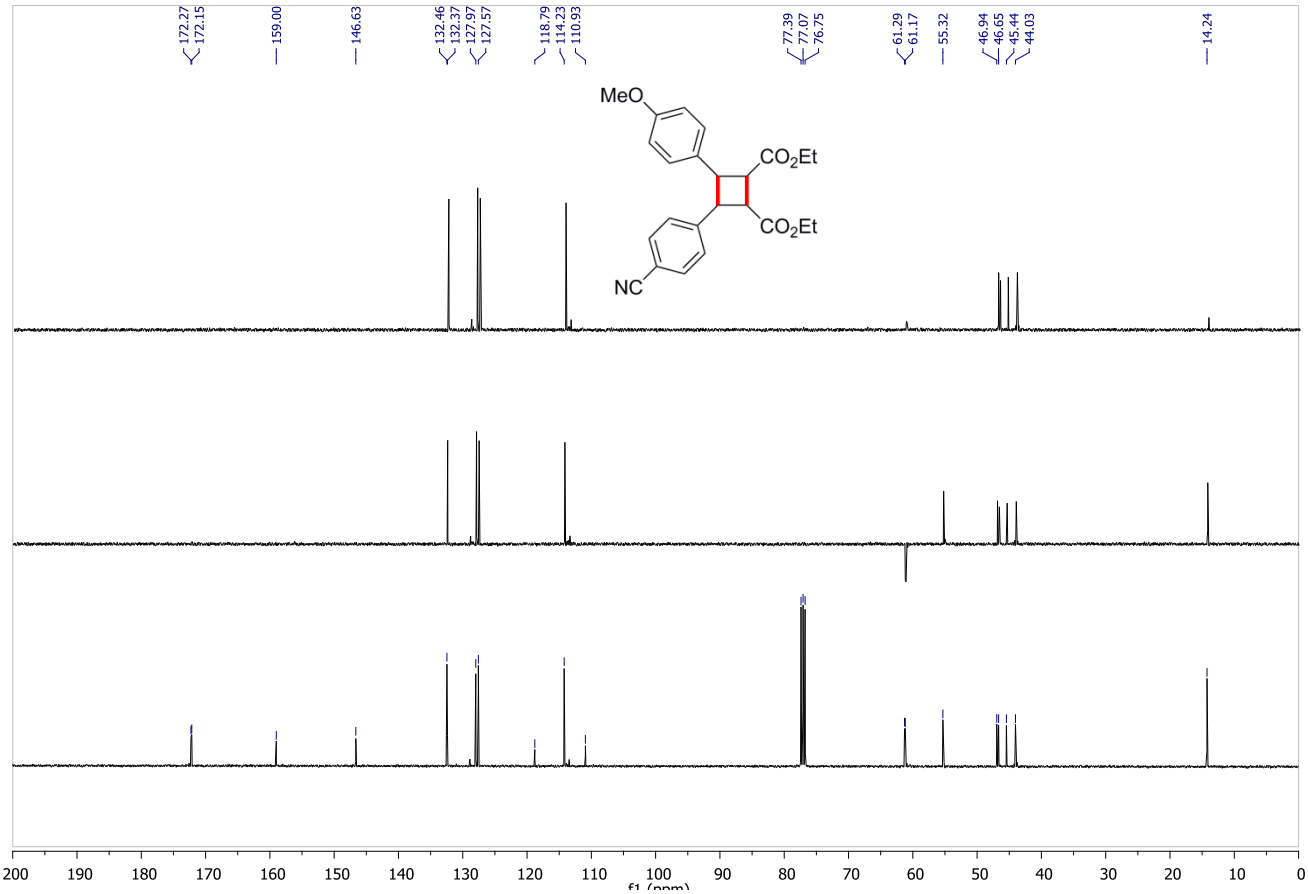
^{13}C-NMR: **7b** (*trans*)



^{1}H-NMR: **7c** (*trans*)



^{13}C-NMR: **7c** (*trans*)



5. Crystal data for 2v:

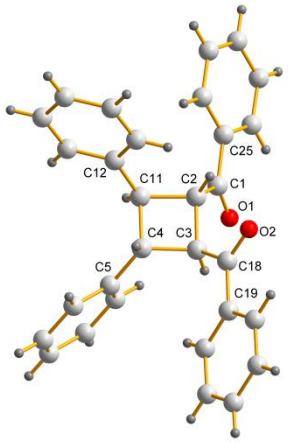


Fig. S2: Crystal structure of **2v**

Experimental: Single clear colourless prism-shaped crystals of (**2v**) were obtained by recrystallisation from DCM/pentane. A suitable crystal (0.27×0.10×0.07) mm^3 was selected and mounted on a MITIGEN holder with inert oil on a SuperNova, Single source at offset, Atlas diffractometer. The crystal was kept at T = 123.01(10) K during data collection. Using **Olex2** (Dolomanov et al., 2009), the structure was solved with the Sheldrick, 2015) structure solution program, using the None methods solution method. The model was refined with version of **olex2.refine** (Bourhis et al., 2015) using Gauss-Newton minimisation.

Crystal Data. $C_{30}H_{24}O_2$, M_r = 416.52, monoclinic, $P2_1/c$ (No. 14), a = 10.8157(1) Å, b = 9.5942(1) Å, c = 21.1289(2) Å, β = 91.349(1)°, α = γ = 90°, V = 2191.90(4) $Å^3$, T = 123.01(10) K, Z = 4, Z' = 1, μ(Cu K_α) = 0.606, 49002 reflections measured, 4378 unique (R_{int} = 0.0317) which were used in all calculations. The final wR_2 was 0.0873 (all data) and R_1 was 0.0338 (I≥σ(I)).

Compound	**2v**
Formula	$C_{30}H_{24}O_2$
$D_{calc.}$/ g cm^{-3}	1.2621
μ/mm^{-1}	0.606
Formula Weight	416.52
Colour	clear colourless
Shape	prism
Size/mm^3	0.27×0.10×0.07
T/K	123.01(10)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	10.8157(1)
b/Å	9.5942(1)
c/Å	21.1289(2)
α/°	90
β/°	91.349(1)
γ/°	90
V/$Å^3$	2191.90(4)
Z	4
Z'	1
Wavelength/Å	1.54184
Radiation type	Cu K_α
Θ_{min}/°	4.09
Θ_{max}/°	73.59
Measured Refl.	49002
Independent Refl.	4378
Reflections Used	4034
R_{int}	0.0317
Parameters	288
Restraints	0
Largest Peak	0.2557
Deepest Hole	-0.1984
GooF	1.0479
wR_2 (all data)	0.0873
wR_2	0.0849
R_1 (all data)	0.0367
R_1	0.0338

Table 1.1: Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for **2v**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
O(1)	2124.4(7)	9112.5(8)	5945.5(4)	30.51(19)
O(2)	3233.2(8)	5262.6(9)	5034.9(4)	33.1(2)
C(5)	2024.0(9)	5363.4(11)	7158.8(5)	21.1(2)
C(3)	2450.1(9)	6193.2(11)	5986.1(5)	21.3(2)
C(2)	3542.5(9)	7222.2(11)	6008.6(5)	21.5(2)
C(4)	2909.1(9)	5523.5(11)	6626.5(5)	21.2(2)
C(19)	1440.8(10)	4123.2(11)	5377.4(5)	23.1(2)
C(12)	5194.4(9)	6212.8(11)	6843.0(5)	22.7(2)
C(25)	4214.1(10)	9794.1(11)	5880.2(5)	24.9(2)
C(11)	3900.2(9)	6717.2(11)	6695.6(5)	21.7(2)
C(6)	2020.8(10)	4149.0(11)	7516.7(5)	26.6(2)
C(1)	3206.6(10)	8745.5(11)	5943.8(5)	23.1(2)
C(10)	1213.5(10)	6430.8(12)	7316.3(5)	25.2(2)
C(18)	2427.1(10)	5206.7(11)	5428.6(5)	23.2(2)
C(20)	1478.8(11)	3171.3(12)	4875.0(5)	29.1(2)
C(24)	491.7(10)	4021.5(11)	5808.6(5)	25.2(2)
C(23)	-399(1)	2981.9(12)	5739.6(5)	28.8(2)
C(13)	5925.2(10)	5643.5(12)	6376.5(5)	28.5(2)
C(9)	411.4(10)	6272.1(13)	7815.1(5)	29.2(2)
C(17)	5649.6(11)	6219.6(12)	7464.6(5)	28.5(2)
C(7)	1217.1(11)	3995.9(13)	8017.1(5)	31.0(3)
C(26)	5453.0(11)	9473.6(13)	6000.3(6)	31.0(3)
C(15)	7534.3(11)	5116.4(12)	7142.4(6)	32.7(3)
C(8)	412.5(10)	5050.6(14)	8166.3(5)	30.7(3)
C(30)	3893.3(11)	11146.9(12)	5698.1(6)	30.7(2)
C(16)	6816.0(11)	5669.8(13)	7611.5(6)	33.6(3)
C(22)	-357.8(11)	2057.6(12)	5239.8(6)	32.0(3)
C(14)	7088.7(11)	5110.7(13)	6524.8(6)	32.6(3)
C(21)	581.6(12)	2156.7(13)	4807.0(6)	34.0(3)
C(29)	4795.9(12)	12149.4(13)	5628.4(6)	35.9(3)
C(28)	6025.4(12)	11824.5(13)	5752.0(6)	36.4(3)
C(27)	6351.6(11)	10494.8(14)	5941.9(6)	37.5(3)

Table 1.2: Anisotropic Displacement Parameters (×10⁴) **2v**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \ldots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	22.7(4)	25.6(4)	43.2(5)	1.1(3)	0.3(3)	-1.3(3)
O(2)	34.5(4)	36.5(5)	28.6(4)	-8.0(4)	9.9(3)	-6.4(3)
C(5)	19.4(5)	22.6(5)	21.3(5)	-3.8(4)	-0.3(4)	-1.6(4)
C(3)	19.3(5)	21.1(5)	23.7(5)	-1.4(4)	1.9(4)	-0.1(4)
C(2)	18.8(5)	22.2(5)	23.4(5)	-1.3(4)	2.0(4)	-0.4(4)
C(4)	20.7(5)	19.8(5)	23.3(5)	-0.2(4)	2.5(4)	-1.2(4)
C(19)	24.4(5)	22.2(5)	22.6(5)	0.8(4)	-3.3(4)	1.2(4)
C(12)	21.6(5)	21.3(5)	25.3(5)	-4.7(4)	0.2(4)	2.2(4)
C(25)	25.8(5)	24.4(5)	24.4(5)	-2.1(4)	0.6(4)	1.4(4)
C(11)	20.9(5)	21.7(5)	22.6(5)	-1.7(4)	2.9(4)	-1.3(4)
C(6)	30.1(6)	22.3(5)	27.3(5)	-1.8(4)	0.6(4)	-0.4(4)
C(1)	22.6(5)	24.3(5)	22.4(5)	-0.2(4)	0.0(4)	-0.3(4)
C(10)	24.1(5)	26.2(5)	25.4(5)	0.9(4)	2.4(4)	1.3(4)
C(18)	23.4(5)	24.3(5)	22.0(5)	1.0(4)	0.4(4)	1.5(4)
C(20)	33.9(6)	27.5(6)	25.9(5)	-0.6(5)	1.1(4)	-1.6(4)
C(24)	24.3(5)	25.1(5)	26.2(5)	-0.3(4)	-1.2(4)	-1.3(4)
C(23)	24.5(5)	29.1(6)	32.7(6)	-1.8(4)	-0.8(4)	3.2(5)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(13)	24.7(5)	33.1(6)	27.4(5)	2.8(4)	-2.4(4)	-1.9(4)
C(9)	22.5(5)	38.7(6)	26.6(5)	3.3(5)	2.4(4)	-2.9(5)
C(17)	29.4(6)	31.9(6)	24.4(5)	-5.6(5)	0.9(4)	3.6(4)
C(7)	35.7(6)	31.0(6)	26.2(5)	-9.7(5)	1.1(5)	5.2(4)
C(26)	25.7(6)	27.2(6)	39.8(6)	-2.3(4)	-0.4(5)	8.4(5)
C(15)	23.7(6)	28.9(6)	45.1(7)	-1.1(4)	-7.2(5)	8.8(5)
C(8)	24.3(6)	45.8(7)	22.0(5)	-9.2(5)	2.3(4)	0.2(5)
C(30)	30.0(6)	27.1(6)	34.8(6)	0.5(5)	-1.2(5)	4.3(5)
C(16)	33.9(6)	36.4(6)	30.1(6)	-7.1(5)	-8.8(5)	10.0(5)
C(22)	33.0(6)	25.8(6)	36.6(6)	-7.6(5)	-8.2(5)	2.0(5)
C(14)	24.8(6)	33.5(6)	39.5(6)	3.8(5)	0.2(5)	-1.3(5)
C(21)	43.9(7)	28.0(6)	29.7(6)	-4.3(5)	-3.9(5)	-5.7(5)
C(29)	42.1(7)	24.6(6)	40.9(7)	-3.6(5)	-0.1(5)	7.6(5)
C(28)	35.3(6)	32.5(6)	41.5(7)	-12.8(5)	0.2(5)	6.9(5)
C(27)	25.8(6)	38.5(7)	48.1(7)	-6.4(5)	-3.0(5)	10.4(6)

Table 1.3: Bond Lengths in Å for **2v**.

Atom	Atom	Length/Å
O(1)	C(1)	1.2224(13)
O(2)	C(18)	1.2204(13)
C(5)	C(4)	1.5018(14)
C(5)	C(6)	1.3890(15)
C(5)	C(10)	1.3935(15)
C(3)	C(2)	1.5396(14)
C(3)	C(4)	1.5681(14)
C(3)	C(18)	1.5108(14)
C(2)	C(11)	1.5699(14)
C(2)	C(1)	1.5114(14)
C(4)	C(11)	1.5732(14)
C(19)	C(18)	1.4916(15)
C(19)	C(20)	1.4016(15)
C(19)	C(24)	1.3919(15)
C(12)	C(11)	1.5066(14)
C(12)	C(13)	1.3896(15)
C(12)	C(17)	1.3919(15)
C(25)	C(1)	1.4915(15)
C(25)	C(26)	1.3923(16)
C(25)	C(30)	1.3952(16)
C(6)	C(7)	1.3923(16)
C(10)	C(9)	1.3890(15)
C(20)	C(21)	1.3796(17)
C(24)	C(23)	1.3919(15)
C(23)	C(22)	1.3805(17)
C(13)	C(14)	1.3872(16)
C(9)	C(8)	1.3871(17)
C(17)	C(16)	1.3955(17)
C(7)	C(8)	1.3761(18)
C(26)	C(27)	1.3876(17)
C(15)	C(16)	1.3799(19)
C(15)	C(14)	1.3805(17)
C(30)	C(29)	1.3807(17)
C(22)	C(21)	1.3862(18)
C(29)	C(28)	1.3846(18)
C(28)	C(27)	1.3807(18)

Table 1.4: Bond Angles in ° for **2v**.

Atom	Atom	Atom	Angle/°
C(6)	C(5)	C(4)	120.22(9)
C(10)	C(5)	C(4)	121.29(9)
C(10)	C(5)	C(6)	118.47(10)
C(4)	C(3)	C(2)	90.51(7)
C(18)	C(3)	C(2)	115.11(8)
C(18)	C(3)	C(4)	114.53(8)
C(11)	C(2)	C(3)	90.17(7)
C(1)	C(2)	C(3)	115.76(8)
C(1)	C(2)	C(11)	115.83(8)
C(3)	C(4)	C(5)	119.59(8)
C(11)	C(4)	C(5)	116.74(8)
C(11)	C(4)	C(3)	89.02(7)
C(20)	C(19)	C(18)	118.39(10)
C(24)	C(19)	C(18)	122.58(9)
C(24)	C(19)	C(20)	119.03(10)
C(13)	C(12)	C(11)	121.39(9)
C(17)	C(12)	C(11)	119.99(10)
C(17)	C(12)	C(13)	118.46(10)
C(26)	C(25)	C(1)	122.45(10)
C(30)	C(25)	C(1)	118.35(10)
C(30)	C(25)	C(26)	119.19(10)
C(4)	C(11)	C(2)	89.22(7)
C(12)	C(11)	C(2)	119.85(8)
C(12)	C(11)	C(4)	114.40(8)
C(7)	C(6)	C(5)	120.77(11)
C(2)	C(1)	O(1)	120.43(9)
C(25)	C(1)	O(1)	120.50(10)
C(25)	C(1)	C(2)	119.07(9)
C(9)	C(10)	C(5)	120.55(10)
C(3)	C(18)	O(2)	120.34(9)
C(19)	C(18)	O(2)	120.15(10)
C(19)	C(18)	C(3)	119.46(9)
C(21)	C(20)	C(19)	120.37(11)
C(23)	C(24)	C(19)	120.06(10)
C(22)	C(23)	C(24)	120.37(11)
C(14)	C(13)	C(12)	120.86(11)
C(8)	C(9)	C(10)	120.39(11)
C(16)	C(17)	C(12)	120.39(11)
C(8)	C(7)	C(6)	120.39(11)
C(27)	C(26)	C(25)	120.06(11)
C(14)	C(15)	C(16)	119.38(11)
C(7)	C(8)	C(9)	119.43(10)
C(29)	C(30)	C(25)	120.37(11)
C(15)	C(16)	C(17)	120.47(11)
C(21)	C(22)	C(23)	119.87(11)
C(15)	C(14)	C(13)	120.44(11)
C(22)	C(21)	C(20)	120.29(11)
C(28)	C(29)	C(30)	120.09(11)
C(27)	C(28)	C(29)	120.03(11)
C(28)	C(27)	C(26)	120.24(12)

Table 1.5: Hydrogen Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for **2v**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(3)	1656.4(9)	6672.0(11)	6030.4(5)	25.6(3)
H(2)	4175.4(9)	6957.9(11)	5706.7(5)	25.8(3)
H(4)	3318.9(9)	4632.5(11)	6545.1(5)	25.5(3)
H(11)	3645.4(9)	7405.0(11)	7009.7(5)	26.0(3)
H(6)	2561.7(10)	3429.9(11)	7420.9(5)	31.9(3)
H(10)	1209.7(10)	7256.4(12)	7085.6(5)	30.2(3)
H(20)	2112.4(11)	3223.9(12)	4585.9(5)	34.9(3)
H(24)	452.6(10)	4648.7(11)	6143.1(5)	30.3(3)
H(23)	-1025.4(10)	2909.6(12)	6031.9(5)	34.5(3)
H(13)	5630.6(10)	5619.4(12)	5959.6(5)	34.2(3)
H(9)	-129.6(10)	6989.0(13)	7914.1(5)	35.1(3)
H(17)	5173.7(11)	6592.9(12)	7784.0(5)	34.3(3)
H(7)	1224.2(11)	3176.0(13)	8252.0(5)	37.1(3)
H(26)	5678.5(11)	8574.2(13)	6119.9(6)	37.1(3)
H(15)	8311.3(11)	4750.6(12)	7241.4(6)	39.2(3)
H(8)	-125.8(10)	4945.5(14)	8499.8(5)	36.8(3)
H(30)	3067.0(11)	11374.7(12)	5623.2(6)	36.8(3)
H(16)	7111.2(11)	5676.4(13)	8028.5(6)	40.3(3)
H(22)	-959.1(11)	1369.8(12)	5193.6(6)	38.4(3)
H(14)	7571.7(11)	4747.2(13)	6205.9(6)	39.2(3)
H(21)	607.2(12)	1536.6(13)	4469.2(6)	40.7(3)
H(29)	4577.7(12)	13044.0(13)	5498.3(6)	43.1(3)
H(28)	6631.7(12)	12502.3(13)	5707.0(6)	43.7(3)
H(27)	7176.5(11)	10282.8(14)	6030.9(6)	45.0(3)