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Copper Ferrite Nanoparticles Catalyzed Challenging Diels-Alder Reaction of Aromatic Chalcones with Cyclopentadiene

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1. EXPERIMENTAL SECTION

1.1 Materials & Methods

Cyclopentadiene, isoprene and metal ferrites ($CuFe_2O_4$, particle size <100 nm; $CoFe_2O_4$, particle size = 30 nm) were purchased from Sigma Aldrich. All the other metal salts were purchased from AVRA and were used as such without further purification. Chalcones were synthesized as per the procedure reported in literature.[1,2] Solvents were dried and purified according to standard procedures before use. Melting points were obtained on a Thomas-Hoover apparatus in open capillaries and are uncorrected. FTIR spectra were recorded using Bruker alpha Eco-ATR spectrometer in the spectral region of 4000-650 cm^{-1}. ^{1}H-NMR spectra were recorded on Bruker Avance Neo 500 MHz spectrometer in $CDCl_3$ solvent. Spectroscopic data are represented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, dd = doublet of doublets m = multiplet, br = broad singlet, ddd = doublet of doublet of doublet, dddd = doublet of doublet of doublet of doublet, td = triplet of doublet, dt = doublet of triplet, qd = quartet of doublet), integration, coupling constants in Hertz (Hz). ^{13}C NMR spectra were recorded at 125 MHz in $CDCl_3$ relative to trimethylsilane as internal standard. Crude products were isolated and purified by column chromatography over 230-400 mesh size silica gel using hexane:ethyl acetate as eluents. The progress of the reaction was monitored by thin layer chromatography (TLC) on silica coated aluminum plates F_{254} and visualized under UV chamber.

1.2 Characterization Data

Phenyl(3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanone **(3a)**

Results: 89% yield, pale-yellow oily liquid, dr = 65:35 (*endo:exo*); **IR (ATR):** ν_{max} 3058, 3022, 2992, 2970, 1668, 1596, 1447, 1333, 1215, 1014, 745, 692, 665 cm^{-1}; **^{1}H-NMR: (500 MHz, $CDCl_3$)**: δ 7.96-7.92 (m, 1.36H (*endo*)), 7.91-7.88 (m, 0.70H (*exo*)), 7.56-7.49 (m, 1H), 7.46-7.37 (m, 2H), 7.31-7.27 (m, 3H), 7.25-7.15 (m, 2H), 6.49-6.46 (m, 1H), 6.17 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.35H (*exo*)), 5.87 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.65H (*endo*)), 3.91 (dd, *J* = 3.5 Hz, 5.0 Hz, 1H), 3.47 (dd, *J* = 1.5 Hz, 5.0 Hz, 0.65H (*endo*)), 3.44 (dd, *J* = 1.5 Hz, 5.0 Hz, 0.35H (*exo*)), 3.34 (s, 0.65H

(*endo*)), 3.18 (s, 0.35H (*exo*)), 3.11-3.15 (m,1H), 2.03 (d, *J* = 8.5 Hz, 0.65H (*endo*)), 1.90 (d, *J* = 8.5 Hz, 0.35H (*exo*)), 1.65 (dq, *J* = 2.0, 8.5 Hz, 0.65H (*endo*)), 1.54 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.35H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 201.3, 200.1, 144.8, 143.6, 139.3, 137.3, 137.0, 136.9, 136.6, 133.1, 133.0, 132.9, 128.7, 128.6, 128.5, 128.2, 128.1, 127.6, 126.3, 126.1, 56.3, 54.6, 49.0, 48.9, 48.7, 48.6, 48.1, 47.9, 47.8, 45.9 ppm.

4-Fluorophenyl-(3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanone (**3b**)

Results: 68% yield, colorless oily liquid, dr = 70:30 (*endo:exo*); **IR (ATR):** ν_{max} 3059, 3027, 2986, 1676, 1495, 1454, 1333, 1219, 1016, 834, 746, 700 cm^{-1}; **^{1}H-NMR: (500 MHz, $CDCl_3$)**: δ 8.12 (dd, *J* = 5.5 Hz, 8.5 Hz, 1.47H (*endo*)), 8.05 (dd, *J* = 5.5 Hz, 8.5 Hz, 0.63H (*exo*)), 7.48-7.38 (m, 4H), 7.37-7.29 (m, 1H), 7.26 (t, *J* = 8.5 Hz, 1.39H (*endo*)), 7.20 (t, *J* = 8.5 Hz, 0.66H (*exo*)), 6.63 (dd, *J* = 3.0 Hz, 5.0 Hz, 1H), 6.33 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.30H (*exo*)), 6.04 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.70H (*endo*)), 4.02 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.70H (*endo*)), 3.99 (dd, *J* = 5.0 Hz, 8.5 Hz, 0.30H (*exo*)), 3.60 (d, *J* = 4.0 Hz, 0.70H (*endo*)), 3.53 (d, *J* = 4.5 Hz, 0.30H (*exo*)), 3.49 (s, 0.70H (*endo*)), 3.32 (s, 0.30H (*exo*)), 3.29 (s, 0.30H), 3.28 (s, 0.70H), 2.19 (d, *J* = 8.5 Hz, 0.70H (*endo*)), 2.05 (d, *J* = 9.0 Hz, 0.30H (*exo*)), 1.82 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.70H (*endo*)), 1.72 (dd, *J* = 1.0 Hz, 8.5 Hz, 0.30H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 199.8, 198.5, 165.7(d, *J* = 250 Hz) 165.7(d, *J* = 250 Hz), 144.6, 143.4, 139.4, 137.0, 136.6, 133.7 (d, *J* = 2.5 Hz), 133.4 (d, *J* = 2.5 Hz), 133.0, 131.2 (d, *J* = 10.0 Hz), 131.0 (d, *J* = 10.0 Hz), 128.7, 128.3, 128.1, 127.6, 126.5, 126.2, 115.7(d, *J* = 12.5 Hz), 115.7 (d, *J* = 15 Hz), 115.6, 115.6, 56.3, 54.6, 49.1, 48.8, 48.7, 48.7, 48.2, 48.1, 46.2 ppm.

4-Chlorophenyl-(3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanone (**3c**)

Results: 70% yield, colorless oily liquid, dr = 75:25 (*endo:exo*); **IR (ATR):** ν_{max} 3058, 3022, 2992, 2970, 1668, 1596, 1447, 1214, 1014, 745, 692, 665 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.85 (d, *J* = 8.5 Hz, 1.56H (*endo*)), 7.78 (d, *J* = 8.5 Hz, 0.53H (*exo*)), 7.38 (d, *J* = 8.5 Hz, 1.57H (*endo*)), 7.33 (d, *J* = 8.5 Hz, 0.59H (*exo*)), 7.30-7.11 (m, 5H), 6.45 (dd, *J* = 3.5 Hz, 5.0 Hz, 1H), 6.15 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.25H (*exo*)), 5.86 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.75H (*endo*)), 3.84 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.75H (*endo*)), 3.81 (dd, *J* = 4.0 Hz, 5.0 Hz, 0.25H (*exo*)), 3.42 (d, *J* = 4.0 Hz, 0.75H (*endo*)), 3.35 (d, *J* = 4.5 Hz, 0.25H (*exo*)), 3.30 (s, 0.75H (*endo*)), 3.15 (s, 0.25H (*exo*)), 3.12 (s, 0.25H), 3.10 (s, 0.75H), 2.01 (d, *J* = 8.5 Hz, 0.75H

(*endo*)), 1.86 (d, *J* = 8.5 Hz, 0.25H (*exo*)), 1.64 (dd, *J* = 8.5 Hz, 1.0 Hz, 0.75H (*endo*)), 1.54 (dd, *J* = 8.5 Hz, 1.5 Hz, 0.25H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.6, 199.3, 144.9, 143.7, 139.9, 139.8, 139.7, 137.4, 137.0, 136.0, 135.7, 133.4, 130.4, 130.3, 129.4, 129.3, 129.1, 128.7, 128.5, 128.0, 126.9, 126.6, 56.7, 49.1, 49.0, 48.6, 48.5, 46.5 ppm.

4-Bromophenyl(3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanone (**3d**)

Results: 68% yield, colorless oily liquid, dr = 75:25 (*endo:exo*); **IR (ATR):** ν_{max} 3057, 3025, 2966, 1740, 1674, 1451, 1366, 1221, 1013, 797, 718, 699 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.78 (dd, *J* = 2.0 Hz, 7.0 Hz, 1.60H (*endo*)), 7.72 (dd, *J* = 2.0 Hz, 7.0 Hz, 0.57H (*exo*)), 7.56 (dd, *J* = 2.0 Hz, 6.5 Hz, 1.54H (*endo*)) 7.51 (dd, *J* = 1.5 Hz, 6.5 Hz, 0.52H (*exo*)), 7.32-7.27 (m, 3H), 7.25-7.12 (m, 2H), 6.48-6.44 (m, 1H), 6.16 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.25H (*exo*)), 5.87 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.75H (*endo*)), 3.84 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.75H (*endo*)), 3.81 (dd, *J* = 5.0 Hz, 3.5 Hz, 0.25H (*exo*)), 3.43 (d, *J* = 3.5 Hz, 0.75H (*endo*)), 3.35 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.25H (*exo*)), 3.31 (s, 0.75H (*endo*)), 3.16 (s, 0.25H (*exo*)), 3.12 (s, 0.25H (*exo*)), 3.11 (s, 0.75H (*endo*)), 2.02 (d, *J* = 8.5 Hz, 0.75H (*endo*)), 1.87 (d, *J* = 8.5 Hz, 0.25H (*exo*)), 1.65 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.75H (*endo*)), 1.55 (dd, *J* = 2.0 Hz, 9.0 Hz, 0.25H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.3, 199.1, 144.4, 143.2, 139.3, 136.9, 136.5, 135.9, 135.6, 132.9, 131.9, 131.8, 130.0, 129.9, 128.6, 128.2, 128.0, 127.9, 127.5, 126.4, 126.1, 56.2, 54.5, 49.0, 48.7, 48.6, 48.5, 48.2, 48.1, 48.0, 46.1 ppm.

(3-Phenylbicyclo[2.2.1]hept-5-en-2-yl)(*p*-tolyl)methanone (**3e**)

Results: 52% yield, yellow oily liquid, dr = 70:30 (*endo:exo*); **IR (ATR):** ν_{max} 3060, 3024, 2967, 1668, 1598, 1455, 1330, 1256, 1219, 1019, 836, 745, 715cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$):** δ 7.84 (d, *J* = 8.0 Hz 1.47H (*endo*)) 7.79 (d, *J* = 8.5 Hz 0.64H (*exo*)), 7.31-7.15 (m, 7H), 6.46 (dd, *J* = 4.0 Hz, 7.5 Hz, 1H), 6.16 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.30H (*exo*)), 5.87 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.70H (*endo*)), 3.93-3.87 (m, 1H), 3.48 (d, *J* = 3.5 Hz, 0.70H (*endo*)), 3.41 (d, *J* = 4.5 Hz, 0.30H (*exo*)), 3.33 (s, *J* = 4.5 Hz, 0.70H (*endo*)), 3.17 (s, 0.30H (*exo*)), 3.12 (s, 1H), 2.40 (s, 2H, CH_3 (*endo*)), 2.38 (s, 1H, CH_3 (*exo*)) 2.02 (d, *J* = 8.0 Hz, 0.70H (*endo*)), 1.90 (d, *J* = 8.5 Hz, 0.30H (*exo*)), 1.64 (dd, *J* = 1.5 Hz, 8.0 Hz, 1.06H (*endo*)),

1.53 (dd, *J* = 1.0 Hz, 8.5 Hz, 0.48H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 199.7, 198.4, 163.3, 139.0, 130.7, 128.5, 128.2, 128.1, 127.5, 125.9, 113.8, 113.7, 55.8, 55.5, 48.8, 48.5, 45.9, 32.0, 27.1, 26.4, 24.2 ppm.

4-Methoxyphenyl(3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanone (**3f**)

Results: 62% yield, yellow oily liquid , dr = 92:8 (*endo:exo*); **IR (ATR):** ν_{max} 3023, 2954, 2925, 1740, 1669, 1603, 1449, 1367, 1223, 1017, 817, 746, 698 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$):** δ 7.93 (dt, *J* = 3.0 Hz, 10.0 Hz, 1.90H (*endo*)) 7.86 (dd, *J* = 2.0 Hz, 7.0 Hz, 0.18H (*exo*)), 7.31-7.25 (m, 4H), 7.23-7.15 (m, 1H), 6.90 (dd, *J* = 2.0 Hz, 7.0 Hz, 1.90H (*endo*)), 6.85 (dd, *J* = 2.5 Hz, 7.0 Hz, 0.21H (*exo*)), 6.47-6.44 (m, 1H), 6.16 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.08H (*exo*)), 5.88 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.92H (*endo*)), 3.88-3.84 (m, 4H), 3.46 (dd, *J* = 2.0 Hz, 5.0 Hz, 0.92H (*endo*)), 3.38 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.08H (*exo*)), 3.32 (s, 0.92H (*endo*)), 3.16 (s, 0.08H (*exo*)), 3.13-3.10 (m, 1H), 2.02 (d, *J* = 10.0 Hz, 1H), 1.63 (dd, *J* = 1.5 Hz, 8.5 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 198.5, 193.4, 163.4, 144.9, 139.1, 133.1, 130.8, 130.7, 130.3, 128.6, 127.6, 126.0, 113.8, 55.9, 55.6, 48.8, 48.6, 48.1, 46.0 ppm.

(3-(4-Fluorophenyl)bicyclo[2.2.1]hept-5-en-2-yl)(phenyl)methanone (**3g**)

Results: 58% yield, colorless oily liquid, dr = 55:45 (*endo:exo*); **IR (ATR):** ν_{max} 3059, 3027, 2986, 1676, 1495, 1454, 1333, 1219, 1016, 834, 746, 700 cm^{-1}; **^{1}H-NMR: (500 MHz, $CDCl_3$)**: δ 7.93 (dd, *J* = 1.5 Hz, 8.5 Hz, 1H (*endo*)), 7.88 (dd, *J* = 1.5 Hz, 8.5 Hz, 1H (*exo*)), 7.57-7.52 (m, 1H), 7.47-7.39 (m, 2H), 7.24-7.21 (m, 1H), 7.00-6.90 (m, 2H), 6.50-6.44 (m, 1H), 6.15 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.45H (*exo*)), 5.87 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.55H (*endo*)), 3.88 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.45H (*exo*)), 3.84 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.55H (*endo*)), 3.45 (d, *J* = 4.0 Hz, 0.55H (*endo*)), 3.38 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.45H (*exo*)), 3.35 (s, 0.55H (*endo*)), 3.15-3.13 (m,1H), 3.08 (s, 0.55H (*endo*)), 2.00 (d, *J* = 8.5 Hz, 0.55H (*endo*)), 1.88 (d, *J* = 9.0 Hz, 0.45H (*exo*)), 1.66 (dd, *J* = 1.5 Hz, 10.0 Hz, 0.55H (*endo*)), 1.54 (dd, *J* = 1.5 Hz, 3.5 Hz, 0.45H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 201.1, 200.0, 140.4, 139.2, 136.9, 136.8, 136.7, 133.1, 133.0 (d, *J* = 2.5 Hz), 129.5, 129.4, 128.9, 128.9, 128.7(d, *J* = 10.0 Hz), 128.6, 128.4, 115.4, 115.3, 115.0, 114.9, 56.5, 54.9, 49.0, 48.8, 48.7, 48.1, 48.0, 47.0, 45.2 ppm.

(3-(4-Chlorophenyl)bicyclo[2.2.1]hept-5-en-2-yl)(phenyl)methanone (**3h**)

Results: 55% yield, yellow oily liquid, dr = 65:35 (*endo:exo*); **IR (ATR):** ν_{max} 2958, 2923, 1735, 1678, 1451, 1258, 1013, 793, 731, 695 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.92 (dd, *J* = 1.5 Hz, 8.5 Hz, 1.30H (*endo*)), 7.88 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.72H (*exo*)), 7.68- 7.51 (m, 1H), 7.47-7.39 (m, 2H), 7.25-7.18 (m, 3.49H), 7.08 (d, *J* = 8.0 Hz, 0.70H), 6.50-6.43 (m, 1H), 6.14-6.12 (m, 0.35H (*exo*)), 5.86 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.65H (*endo*)), 3.88 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.35H), 3.83 (dd, *J* = 3.0 Hz, 4.5 Hz, 0.65H (*endo*)), 3.44 (dd, *J* = 1.5 Hz, 5.0 Hz, 0.65H (*endo*)), 3.37 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.35H (*exo*)), 3.34 (s, 0.65H), 3.14 (m, 0.70H (*exo*)), 3.09-3.07 (m, 0.65H), 1.96 (d, *J* = 8.5 Hz, 0.65H (*endo*)), 1.87 (d, *J* = 9.0 Hz, 0.35H (*exo*)), 1.65 (dd, *J* = 3.5 Hz, 8.5 Hz 0.65H (*endo*)), 1.54 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.35H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.9, 199.8, 143.3, 142.1, 139.2, 137.2, 136.9, 136.6, 133.1, 133.0, 133.0, 132.0, 131.8, 129.4, 128.9, 128.7, 128.5, 128.4, 128.3, 56.4, 54.6, 49.0, 48.9, 48.7, 48.5, 48.1, 47.9, 47.1, 45.3 ppm.

(3-(4-Bromophenyl)bicyclo[2.2.1]hept-5-en-2-yl)(phenyl)methanone (**3i**)

Results: 65% yield, yellow oily liquid, dr = 65:35 (*endo:exo*); **IR (ATR):** ν_{max} 3061, 2968, 2927, 1676, 1595, 1448, 1330, 1259, 1214, 1011, 818, 759, 724, 692 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.93 (dd, *J* = 1.0 Hz, 8.0 Hz, 1.33H (*endo*)), 7.89 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.72H (*exo*)), 7.57-7.51 (m, 1H), 7.47-7.33 (m, 4H), 7.14 (dd, *J* = 1.0 Hz, 6.5 Hz 1.30H), 7.03 (dd, *J* = 2.5 Hz, 9.5 Hz, 0.70H), 6.48 (dd, *J* = 3.5 Hz, 5.5 Hz, 0.35H (*exo*)), 6.45 (dd, *J* = 3.5 Hz, 6.0 Hz, 0.65H (*endo*)), 6.13 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.35H (*exo*)), 5.86 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.65H (*endo*)), 3.87 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.35H (*exo*)), 3.83 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.65H (*endo*)), 3.44 (d, *J* = 3.5 Hz, 0.65H (*endo*)), 3.38 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.35H (*exo*)), 3.35 (s, 0.70H (*exo*)), 3.14 (dd, *J* = 1.5 Hz, 3.0 Hz, 0.70H (*exo*)), 3.08 (d, *J* = 1.5 Hz, 0.65H (*endo*)), 1.97 (d, *J* = 8.5 Hz, 0.65H (*endo*)), 1.88 (d, *J* = 8.5 Hz, 0.35H (*exo*)), 1.66 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.65H (*endo*)), 1.54 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.35H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.7, 199.7, 143.7, 142.6, 139.1, 137.1, 136.8, 136.5, 133.0, 132.9, 131.6, 131.2, 129.7, 129.2, 128.6, 128.5, 128.3, 120.0, 119.7, 56.3, 54.5, 48.9, 48.8, 48.7, 48.3, 48.0, 47.8, 47.1, 45.3 ppm.

Phenyl-(3-(*p*-tolyl)bicyclo[2.2.1]hept-5-en-2-yl)methanone (**3j**)

Results: 58% yield, yellow oily liquid dr = 55:45 (*endo:exo*); **IR (ATR):** ν_{max} 3058, 2970, 1676, 1596, 1448, 1329, 1209, 1016, 804, 760, 791, 664 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.94 (dd, *J* = 1.0 Hz, 8.0 Hz, 1.10H (*endo*)), 7.90 (dd, *J* = 1.0 Hz, 8.0 Hz, 0.90H (*exo*)), 7.57-7.49 (m, 1H), 7.48-7.37 (m, 2H), 7.18 (d, *J* = 5.0 Hz, 1H), 7.11 (d, *J* = 5.0 Hz, 1H), 7.06 (s, 2H), 6.50 - 6.45 (m, 1H), 6.18 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.45H (*exo*)), 5.88 (dd, *J* = 2.5 Hz, 8.5 Hz, 0.55H (*endo*)), 3.90 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.55H (*endo*)), 3.88 (dd, *J* = 3.0 Hz, 5.0 Hz, 0.45H(*exo*)) 3.44 (d, *J* = 4.0 Hz, 0.55H (*endo*)), 3.42 (dd, *J* = 1.0 Hz, 5.0 Hz, 0.45H (*exo*)), 3.34 (s, 0.55H (*endo*)), 3.16 (s, 0.45H (*exo*)), 3.13 (s, 0.45H (*exo*)), 3.09 (d, *J* = 1.5 Hz, 0.55H (*endo*)), 2.33 (s, 2H, CH_3 (*endo*)), 2.31 (s, 1H, CH_3 (*exo*)), 2.03 (d, *J* = 8.5 Hz, 0.55H (*endo*)), 1.90 (d, *J* = 8.5 Hz, 0.45H (*exo*)), 1.64 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.45H (*exo*)), 1.53 (dd, *J* = 1.5 Hz, 8.5 Hz, 0.55H (*endo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 201.3, 200.1, 141.6, 140.4, 139.2, 137.3, 137.0, 136.9, 136.5, 135.7, 135.5, 132.9, 132.8, 132.7, 129.2, 128.8, 128.6, 128.5, 128.4, 127.9, 127.4, 56.2, 54.5, 48.9, 48.8, 48.7, 48.6, 48.0, 47.8, 47.4, 45.5, 21.0, 20.9 ppm.

(3-(4-Nitrophenyl)bicyclo[2.2.1]hept-5-en-2-yl)(phenyl)methanone (**3k**)

Results: 79% yield, yellow oily liquid, dr = >99:1 (*endo:exo*); **IR (ATR):** ν_{max} 3059, 2922, 2853, 1667, 1598, 1449, 1214, 1014, 843, 724, 693 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 8.08 (dt, *J* = 2.5 Hz, 4.0 Hz, 2H), 7.92 (dd, *J* = 0.5 Hz, 8.0 Hz, 2H), 7.58- 7.53 (m, 1H), 7.46-7.40 (m, 2H), 7.31 (d, *J* = 2.0 Hz, 8.5 H), 6.54 (dd, *J* = 3.0 Hz, 5.5 Hz, 1H), 6.13 (dd, *J* = 3.0 Hz, 6.0 Hz, 1H), 4.10 (dd, *J* = 3.5 Hz, 5.0 Hz, 1H), 3.45 (dd, *J* = 1.0 Hz, 5.0 Hz, 1H), 3.22 (s, 1H), 3.18 (s, 1H), 1.89 (d, *J* = 8.5 Hz, 1H), 1.58 (dd, *J* = 1.5 Hz, 9.0 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 199.2, 152.9, 146.4, 139.1, 136.9, 133.2, 133.2, 128.8, 128.5, 128.4, 128.3, 123.9, 56.7, 48.9, 48.2, 48.1, 45.7 ppm.

4-(3-Benzoylbicyclo[2.2.1]hept-5-en-2-yl)benzonitrile (**3l**)

Results: 75% yield, yellow oily liquid, dr = > 99:1 (*endo:exo*); **IR (ATR):** ν_{max} 3061, 2967, 2226, 1675, 1448, 1329, 1209, 1016, 756, 694, 660 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.90 (dd, *J* = 1.0 Hz, 8.5 Hz, 2H), 7.55 (tt, *J* = 7.0 Hz, 14.5 Hz, 1H), 7.51(dt, *J* = 2.0 Hz, 6.5 Hz, 2H) 7.46-7.41 (m, 2H), 7.27-7.24 (m, 2H), 6.51 (dd, *J* = 3.0 Hz, 5.5 Hz, 1H), 6.12 (dd, *J* = 3.0 Hz, 5.5 Hz, 1H), 4.04 (dd, *J* = 3.5 Hz, 5.0 Hz, 1H), 3.41 (dd,

J = 1.0 Hz, 5.0 Hz, 1H), 3.20 (s, 1H), 3.17 (s, 1H), 1.88 (d, *J* = 8.5 Hz, 1H), 1.56 (dd, *J* = 2.0 Hz, 9.0 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.0, 149.3, 137.1, 136.5, 136.2, 133.2, 131.9, 128.6, 128.4, 118.9, 110.0, 54.4, 49.1, 48.6, 47.7, 47.5 ppm.

(4-Fluorophenyl)(3-(*p*-tolyl)bicyclo[2.2.1]hept-5-en-2-yl)methanone (**3m**)

Results: 70% yield, yellow oily liquid, dr = 65:35 (*endo:exo*); **IR (ATR):** ν_{max} 3063, 2966, 2926, 1740, 1678, 1451, 1368, 1221, 1018, 802, 714, 677 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ7.97-7.93 (m, 1.33H (*endo*)), 7.90-7.86 (m, 0.71H (*exo*)), 7.17-7.02 (m, 6H), 6.48-6.43 (m, 1H), 6.16 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.35H (*exo*)), 5.87 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.65H (*endo*)), 3.83 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.65H (*endo*)), 3.78 (dd, *J* = 3.5 Hz, 5.5 Hz, 0.35H (*exo*)), 3.38 (d, *J* = 4.0 Hz, 0.65H (*endo*)), 3.34 (dd, *J* = 1.0 Hz, 5.0 Hz, 0.35H (*exo*)), 3.30 (s, 0.35H (*exo*)), 3.15-3.10 (m, 0.70H (*exo*)), 3.09- 3.06 (m, 0.65H (*endo*)), 2.31 (s, 2H CH_3 (*endo*)), 2.30 (s, 1H CH_3 (*exo*)), 2.02 (d, *J* = 8.5 Hz, 0.65H (*endo*)), 1.88 (d, *J* = 8.5 Hz, 0.35H (*exo*)), 1.63 (dq, *J* = 1.5 Hz, 3.5 Hz, 0.65H (*endo*)), 1.53 (m, 0.35H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 199.8, 198.5, 139.2, 136.9, 136.4, 135.6, 133.6 (d, *J* = 3.75 Hz), 133.3 (d, *J* = 2.5 Hz), 132.8, 131.1 (d, *J* = 8.75 Hz), 130.9 (d, *J* = 8.75 Hz),, 129.2, 128.8, 127.8, 127.3, 115.7, 115.5 (d, *J* = 21.2 Hz), 115.5 (d, *J* = 21.2 Hz), 115.4, 56.1, 54.5, 48.8 (d, *J* = 42.5 Hz), 48.7 (d, *J* = 38.75 Hz), 48.0, 47.9, 47.6, 45.7, 20.9 ppm.

(4-Chlorophenyl)(3-(*p*-tolyl)bicyclo[2.2.1]hept-5-en-2-yl)methanone (**3n**)

Results: 70% yield, yellow oily liquid, dr = 72:28 (*endo:exo*); **IR (ATR):** ν_{max} 3059, 2966, 2926, 1734, 1678, 1588, 1369, 1214, 1013, 803, 716, 676 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$):** δ 7.85 (dt, *J* = 2.5 Hz, 9.0 Hz, 1.46H (*endo*)), 7.78 (dt, *J* = 2.5 Hz, 9.5 Hz, 0.57H (*exo*)), 7.39 (dt, *J* = 2.5 Hz, 9.5 Hz, 1.41H (*endo*)), 7.33 (dt, *J* = 2.5 Hz, 9.5 Hz 0.56H (*exo*)), 7.19- 7.09 (m, 3H), 7.07 - 7.00 (m, 1H), 6.45 (m, 1H), 6.16 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.28H (*exo)*) 5.86 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.72H (*endo*)), 3.82 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.72H (*endo*)), 3.76 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.28H (*exo*)), 3.37 (d, *J* = 4.0 Hz, 0.72H (*endo*)), 3.32 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.28H (*exo*)), 3.30 (s, 0.72H (*endo*)), 3.14-3.10 (m, 0.56H), 3.08-3.06 (m, 0.72H (*endo*)), 2.32 (s, 2.23H, C*H*$_3$ (*endo*)), 2.30 (s, 0.89H, C*H*$_3$ (*exo*)), 2.01 (d, *J* = 8.5 Hz, 0.72H (*endo*)), 1.87 (d, *J* = 9.0 Hz, 0.28H (*exo*)), 1.63 (dd, *J* = 3.5 Hz, 10.5 Hz, 0.72H (*endo*)), 1.54-1.52 (m, 0.28H (*exo*)) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.8, 199.5, 141.9, 140.7, 139.8,

139.8, 139.7, 137.4, 136.9, 136.4, 136.2, 136.0, 135.8, 135.8, 133.4, 130.4, 130.3, 129.8, 129.4, 129.3, 129.3, 128.4, 127.9, 56.7, 55.1, 49.5, 49.3, 49.1, 48.9, 48.5, 48.4, 48.2, 46.2, 21.4 ppm.

4-Bromophenyl-(3-(*p*-tolyl)bicyclo[2.2.1]hept-5-en-2-yl)methanone (**3o**)

Results: 65% yield, yellow oily liquid, dr = 55:45 (*endo:exo*); **IR (ATR):** ν_{max} 2952, 2921, 2852, 1740, 1584, 1458, 1373, 1214, 1023, 804, 720, 684 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.77 (dt, *J* = 2.0 Hz, 9.0 Hz, 1.21H (*endo*)), 7.70 (dt, *J* = 2.0 Hz, 9.0 Hz, 0.92H (*exo*)), 7.56 (dt, *J* = 2.5 Hz, 9.5 Hz, 1.18H (*endo*)), 7.50 (dt, *J* = 2.0 Hz, 9.0 Hz, 0.94H (*exo*)), 7.17-7.09 (m, 2H), 7.06- 7.01 (m, 2H), 6.47-6.42 (m, 1H), 6.16 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.45H (*exo*)), 5.86 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.55H (*endo*)), 3.81 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.55H), 3.75 (dd, *J* = 3.5 Hz, 5.5 Hz, 0.45H), 3.37 (d, *J* = 3.5 Hz, 0.55H (*endo*)), 3.31 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.45H (*exo*)), 3.29 (s, 0.55H (*endo*)), 3.11 (d, *J* = 8.0 Hz, 0.90H (*exo*)), 3.06 (s, 0.55H (*endo*)), 2.31 (s, 1.70H, CH_3 (*endo*)), 2.30 (s, 1.35H, CH_3 (*exo*)), 2.01 (d, *J* = 8.5 Hz, 0.55H (*endo*)), 1.86 (d, *J* = 8.5 Hz, 0.45H (*exo*)), 1.63(dd, *J* = 3.5 Hz, 10.5 Hz, 0.55H), 1.53(dd, *J* = 1.5 Hz, 3.0 Hz, 0.45H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 200.6, 199.3, 141.4, 140.2, 139.4, 137.0, 136.5, 135.8, 132.9, 131.9, 131.8, 130.1, 130.0, 129.4, 129.0, 129.2, 128.1, 128.0, 127.4, 56.3, 54.7, 49.1, 48.9, 48.6, 48.5, 48.1, 48.0, 47.8, 45.8 21.0 ppm.

4-Methoxyphenyl(3-(*p*-tolyl)bicyclo[2.2.1]hept-5-en-2-yl)methanone (**3p**)

Results: 71% yield, yellow oily liquid, dr = 45:55 (*endo:exo*); **IR (ATR):** ν_{max} 3011, 2964, 2924, 1738, 1671, 1599, 1453, 1369, 1220, 1021, 838, 714, 680 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.92 (dt, *J* = 2.0, 10.0 Hz, 0.92H (*endo*)), 7.86 (dt, *J* = 2.5 Hz, 9.5 Hz, 1.12H (*exo*)), 7.19-7.08 (m, 2H), 7.04 (s, 2H), 6.90 (dd, *J* = 2.0 Hz, 7.0 Hz, 0.96H (*endo*)), 6.86 (dd, *J* = 2.5 Hz, 7.0 Hz, 1.16H (*exo*)), 6.47-6.43 (m, 1H), 6.16 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.55H (*exo*)), 5.88 (dd, *J* = 2.5 Hz, 5.5 Hz, 0.45H (*endo*)), 3.86 (s, 1.20H, OCH_3 (*endo*)), 3.84 (s, 2.20H, OCH_3 (*exo*)) 3.42 (d, *J* = 4.5 Hz, 0.45H (*endo*)), 3.35 (dd, *J* = 1.5 Hz, 5.0 Hz, 0.55H (*exo*)), 3.31 (s, 0.45H (*endo*)), 3.13 (s, 0.55H (*exo*)), 3.07 (d, *J* = 1.5 Hz,1H), 2.31 (s, 1.30H, CH_3 (*endo*)), 2.29 (s, 1.70H, CH_3 (*exo*)), 2.01 (d, *J* = 8.5 Hz, 0.45H (*endo*)), 1.90 (d, *J* = 8.5 Hz, 0.55H (*exo*)), 1.62 (dd, *J* = 3.0 Hz, 8.5 Hz, 1H), 1.52 (dd, *J* = 3.5 Hz, 9.0 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 199.9, 198.6, 163.4, 141.9, 140.7, 139.1, 136.9, 136.6, 135.7, 135.5,

133.1, 130.8, 130.7, 130.3, 130.1, 129.3, 128.9, 127.5, 113.7, 112.5, 113.8, 113.7, 55.9, 55.5, 54.2, 49.0, 48.8, 48.7, 48.1, 48.0, 47.5, 45.6, 21.1, 21.0 ppm.

4-Chlorophenyl(5-methyl-1,2,3,6-tetrahydro-[1,1'-biphenyl]-2-yl)methanone (**5c** and **5c′**)

Results: 56% yield, yellow oily liquid, *Regio* isomers = 70:30 (*para:meta*); **IR (ATR):** ν_{max} 3063, 3026, 2952, 2923, 1740, 1679, 1588, 1450, 1370, 1227, 1011, 839, 797, 699 cm^{-1}; **^{1}H-NMR (500 MHz, $CDCl_3$)**: δ 7.72 (dd, *J* = 2.0 Hz, 8.5 Hz, 2H), 7.32 (dd, *J* = 2.0 Hz, 6.5 Hz, 2H), 7.17-7.13 (m, 4H), 7.08-7.04 (m, 1H), 5.57-5.53 (m, 0.30H), 5.52-5.47 (m, 0.70H) 3.97- 3.92 (m, 0.30H), 3.89-3.82 (m, 0.70H), 3.34-3.26 (m, 0.70H), 3.24-3.17 (m, 0.30H), 2.35-2.27 (m, 4H), 1.73 (s, 3H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 202.7, 202.6, 144.4, 144.2, 139.3, 139.2, 135.7, 134.2, 129.4, 128.8, 128.5, 128.4, 127.6, 127.5, 126.4, 120.9, 119.1, 47.2, 46.7, 42.9, 42.6, 38.8, 35.1, 34.0, 30.8, 29.7, 23.2 ppm.

4-Bromophenyl(5-methyl-1,2,3,6-tetrahydro-[1,1'-biphenyl]-2-yl)methanone (**5d** and **5d′**)

Results: 58% yield, yellow oily liquid, *Regio* isomers = 70:30 (*para:meta*); **IR (ATR):** ν_{max} 3063, 2923, 2853, 1734, 1584, 1451, 1228, 1007, 837, 742, 679 cm^{-1};**^{1}H-NMR (500 MHz, $CDCl_3$):** δ 7.64 (dd, *J* = 2.0 Hz, 7.0 Hz, 2H), 7.49 (dd, *J* = 2.0 Hz, 7.0 Hz, 2H), 7.17-7.14 (m, 4H), 7.09-7.04 (m, 1H), 5.57-5.53 (m, 0.30H), 5.52-5.45 (m, 0.70H), 3.97-3.91 (m, 0.30H), 3.88-3.82 (m, 0.70H), 3.33-3.26 (m, 0.70H), 3.24-3.16 (m, 0.30H), 2.37-2.17 (m, 4H), 1.73(s, 3H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$):** δ 202.8, 144.4, 136.1, 134.2, 131.8, 129.6, 128.5, 128.4, 127.9, 127.5, 127.4, 126.4, 121.0, 119.1, 47.2, 46.7, 42.9, 42.6, 38.8, 38.2, 35.1, 34.0, 30.8, 29.7, 29.4, 23.2 ppm.

2. REFERENCES

1. J. Li, J. Zhang, M. Li, C. Zhang, Y. Yuanb and R. Liu, *Chem. Commun*., 2019, **55**, 2348. DOI: 10.1039/c8cc09369e
2. Y. Zhang, J. Su, W. Niu and Y. Li, *Chem. Asian J*., 2019, **14**, 1477. DOI: 10.1002/asia.201900170

Mar24-2022
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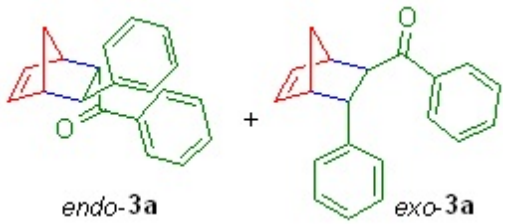

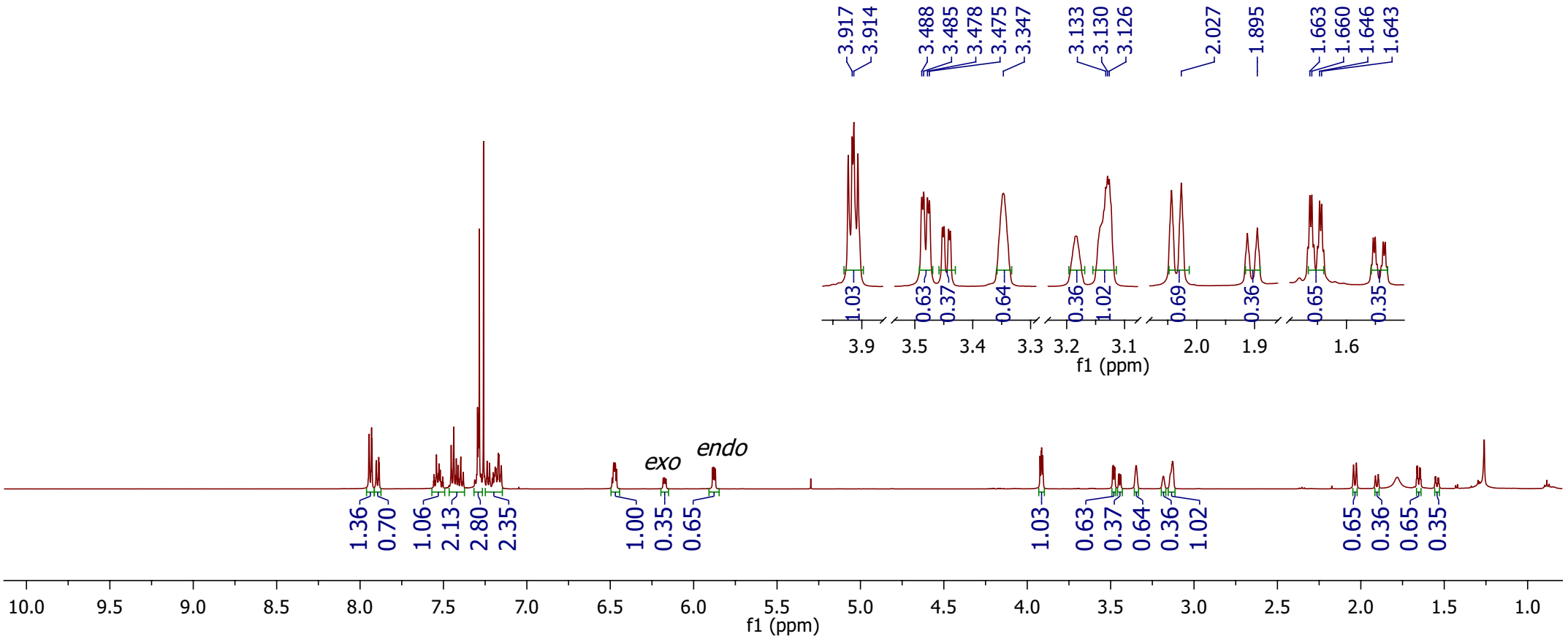

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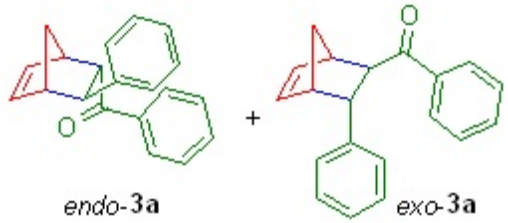

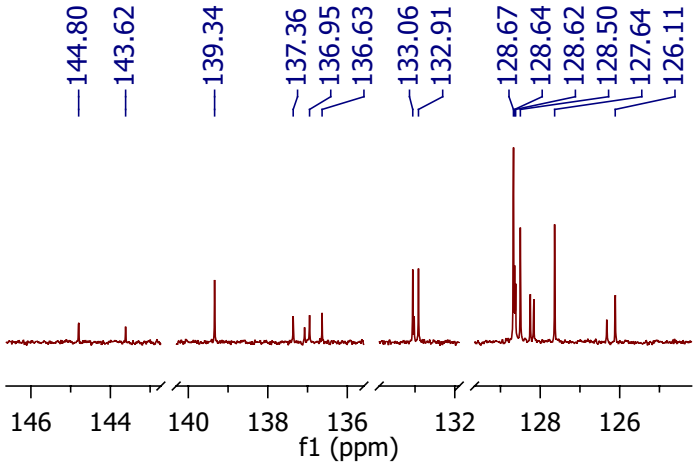

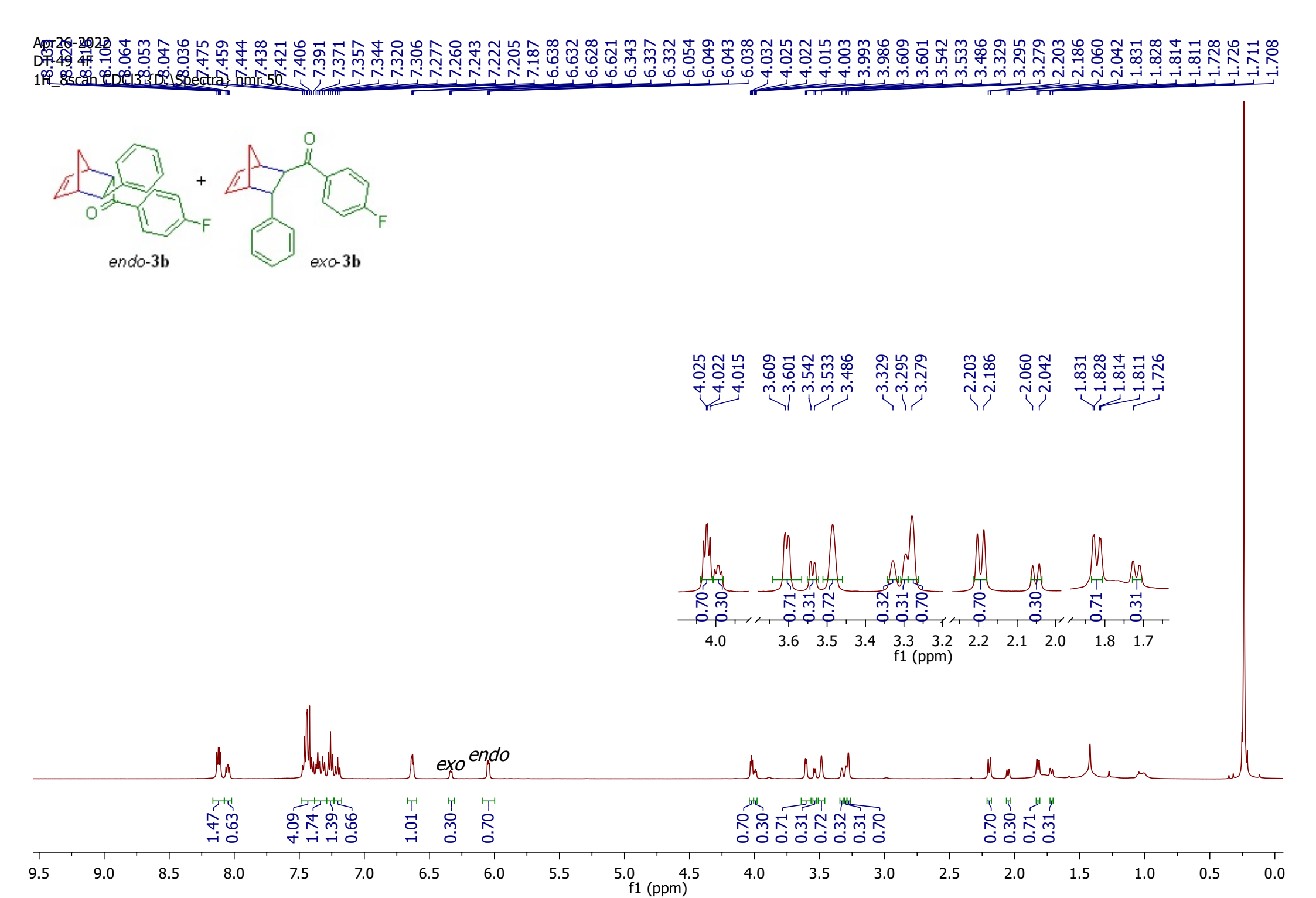
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exo
endo
f1 (ppm)

Apr27-2022
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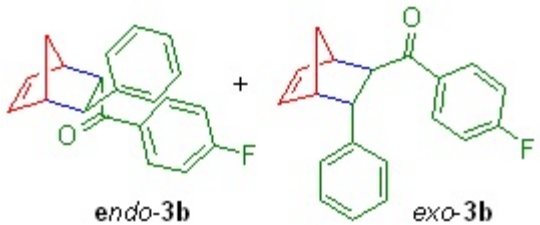

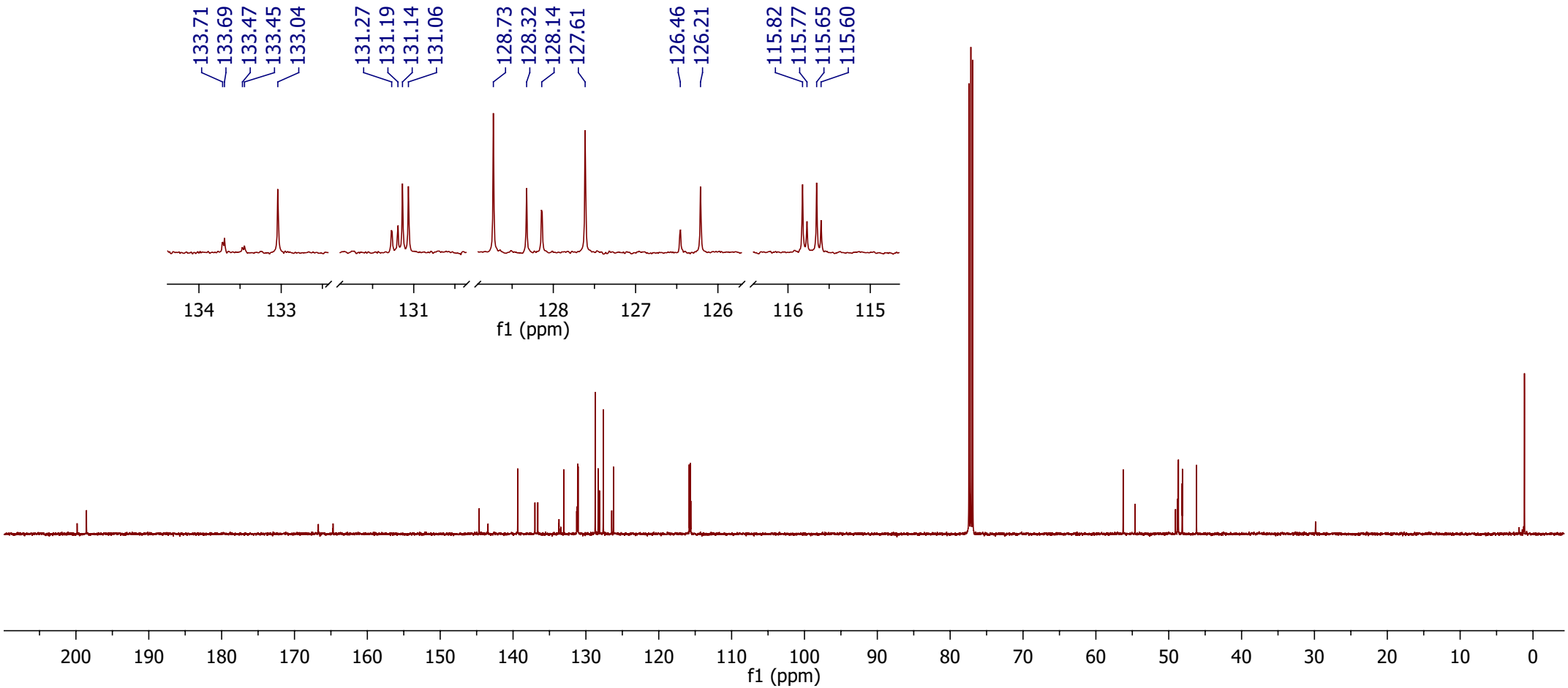

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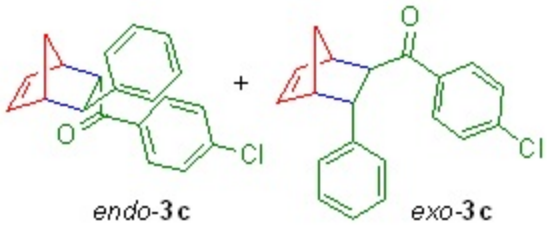

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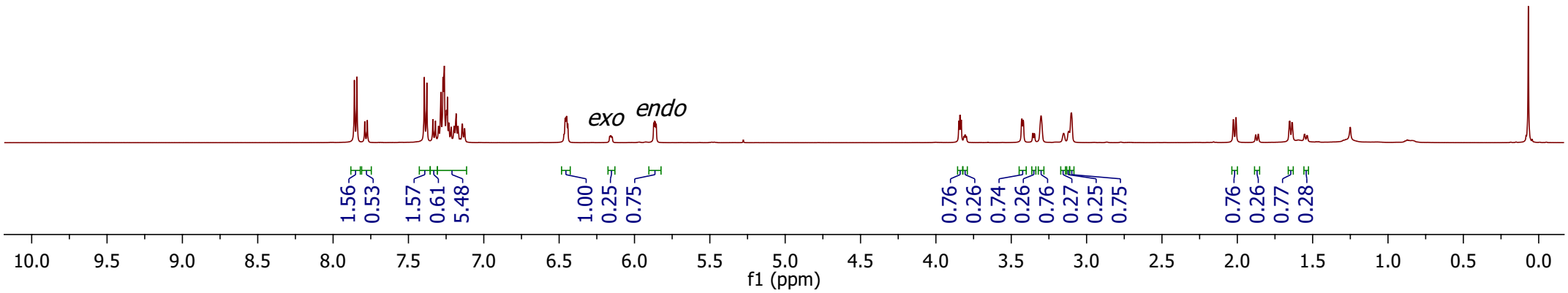

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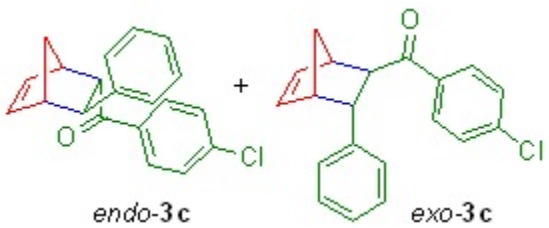

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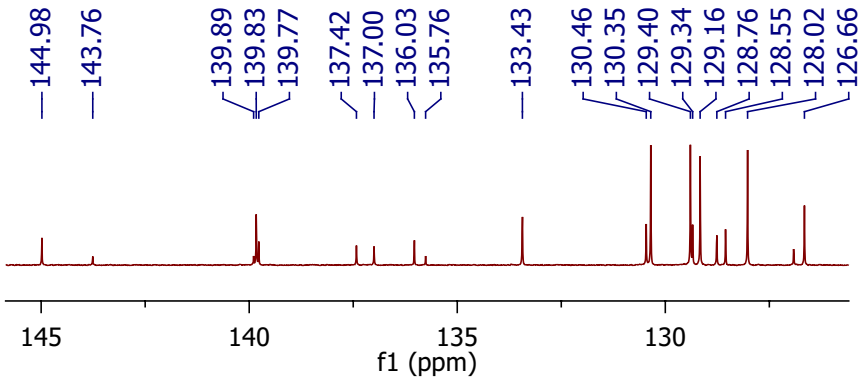

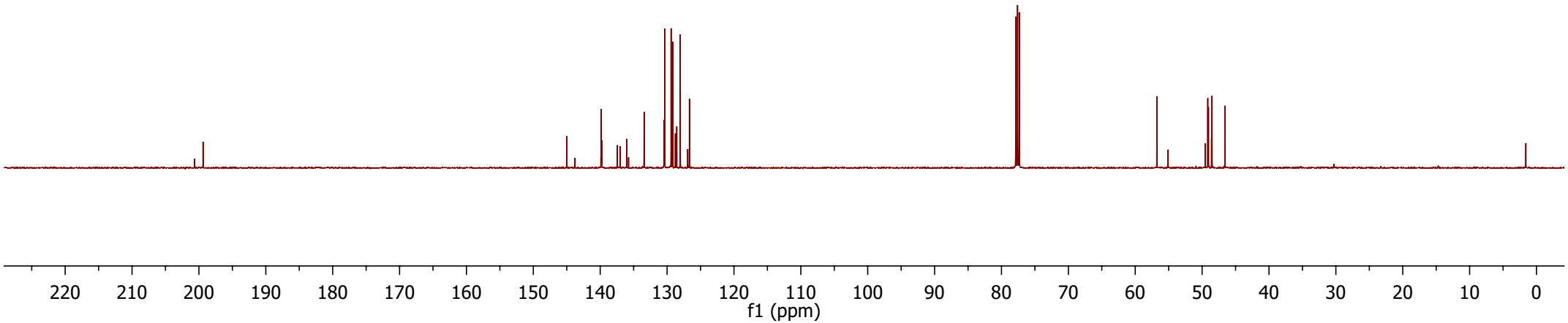

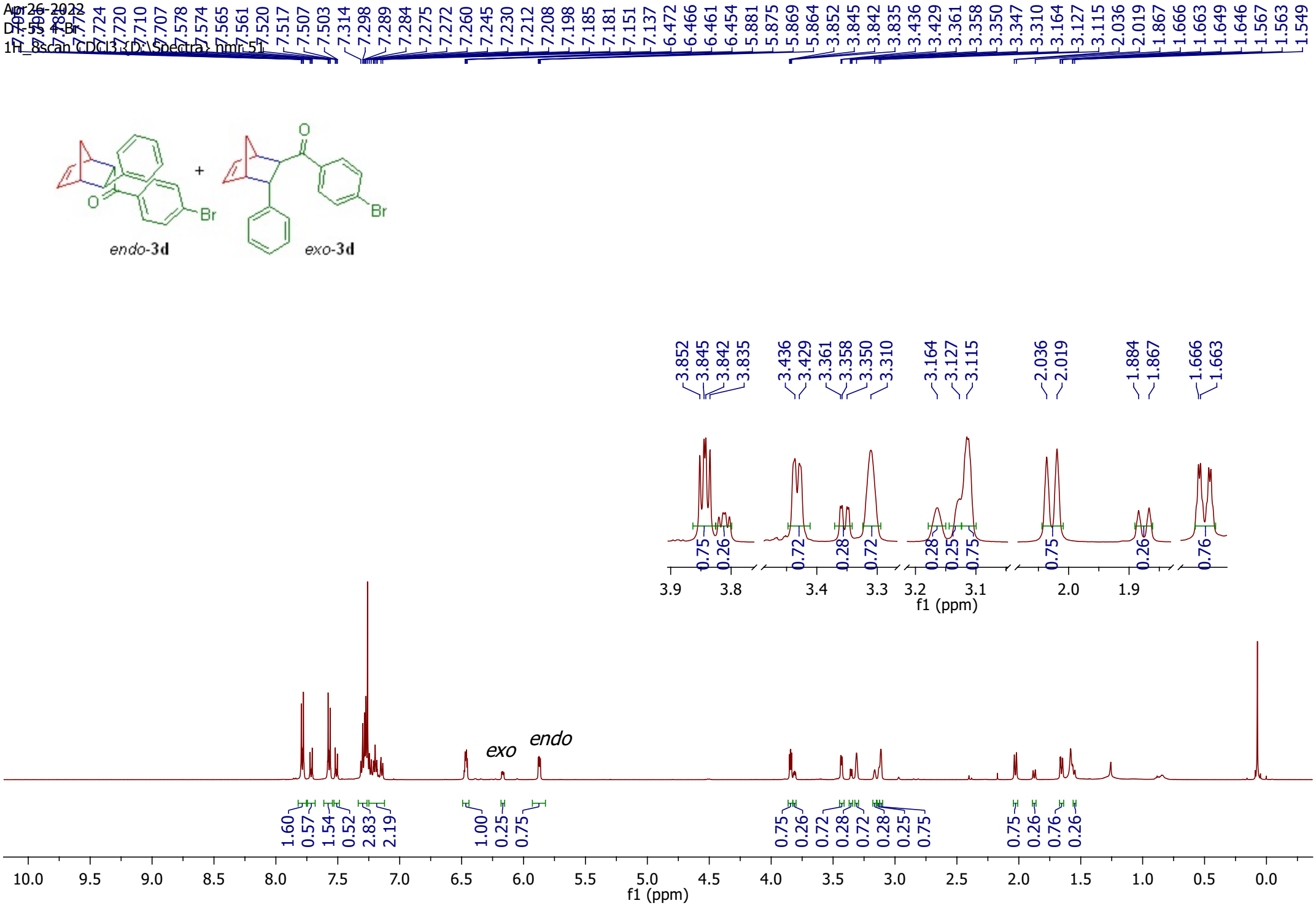
Apr26-2022
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exo
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f1 (ppm)

Apr27-2022
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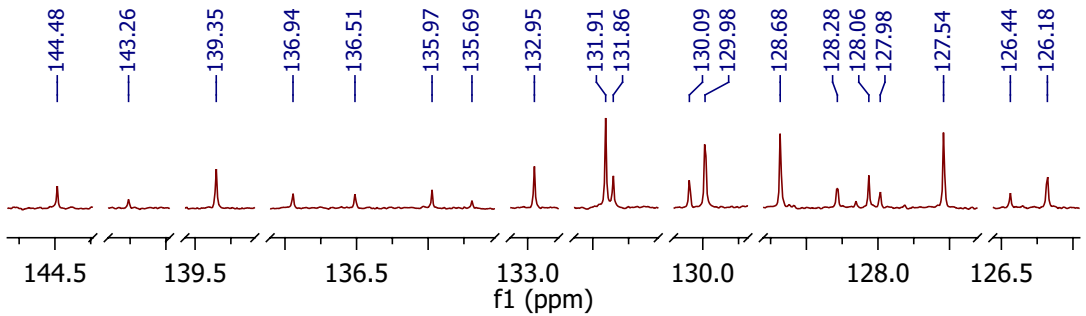
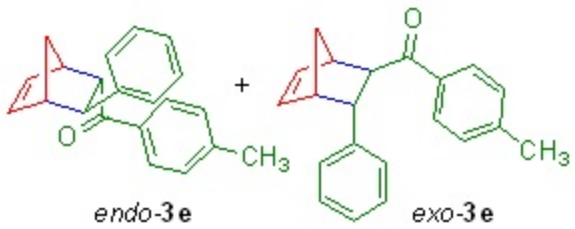
48.65
48.55
48.13
48.10
48.03
46.10
1.10

endo-**3d** *exo*-**3d**

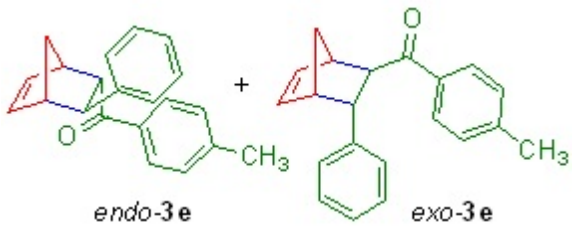


Apr29-2022
DT-53 OCH3
1H_8scan CDCl3 {D:\Spectra} nmr 30

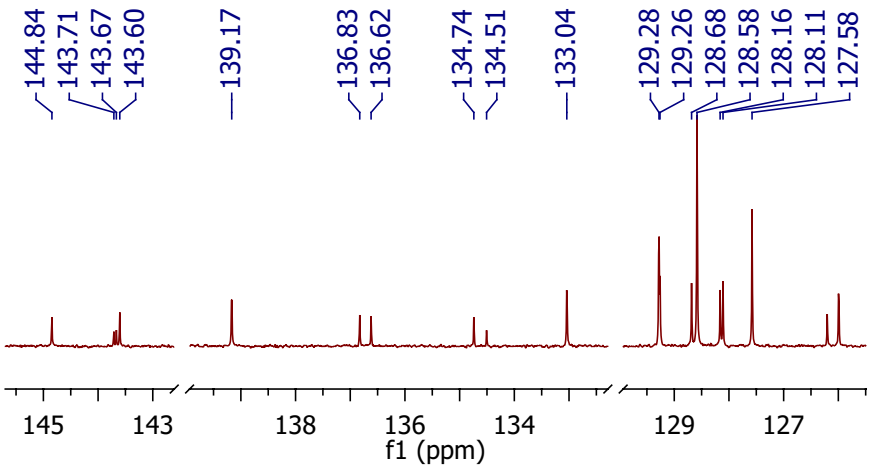


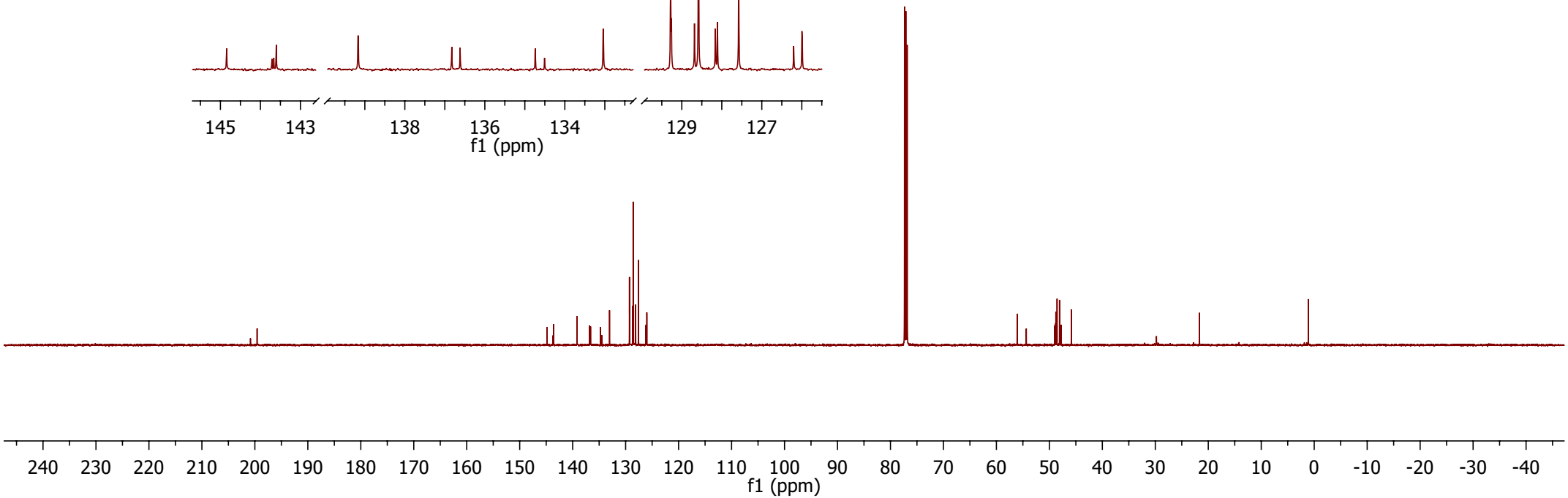
Apr29-2022
DT-53 OCH3
C13CPD CDCl3 {D:\Spectra} nmr 30

200.83
199.58
144.84
143.71
143.67
143.60
139.17
136.83
136.62
134.74
134.51
133.04
129.28
129.26
128.68
128.58
128.16
128.11
127.58
126.20
125.99
77.35
77.09
76.84
56.08
54.36
49.00
48.81
48.77
48.54
48.08
47.91
47.77
45.84
21.67
1.10

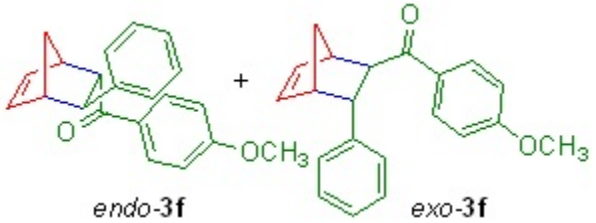


endo-**3e** *exo*-**3e**

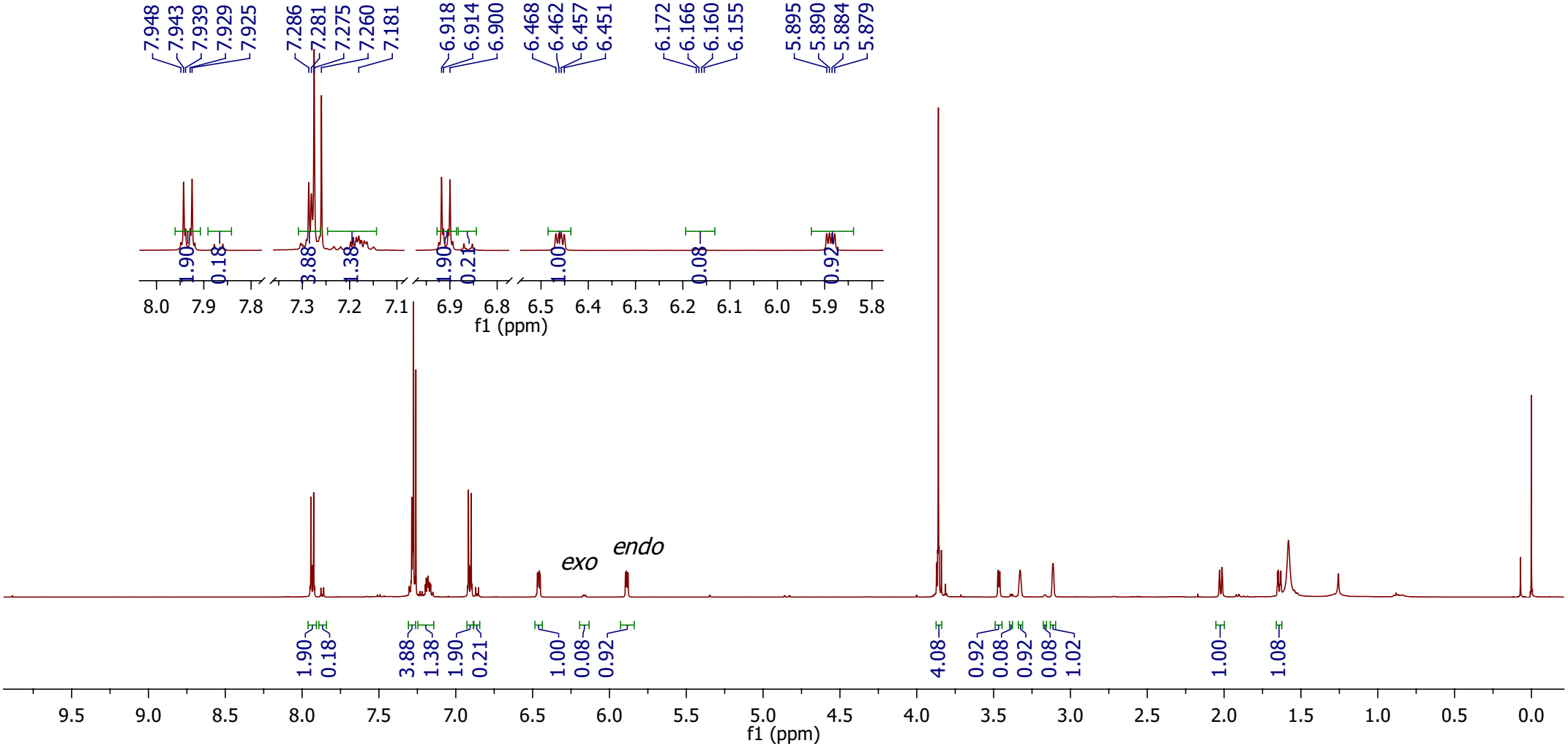




Desktop
DT-53
1H_8scan CDCl3 {D:\Spectra} nmr 8

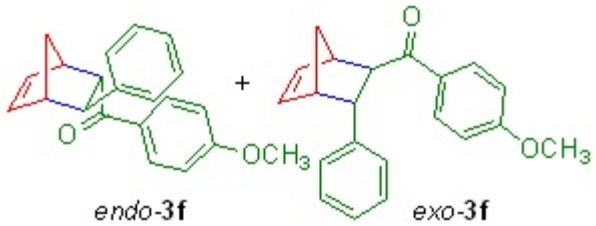


endo-**3f** *exo*-**3f**

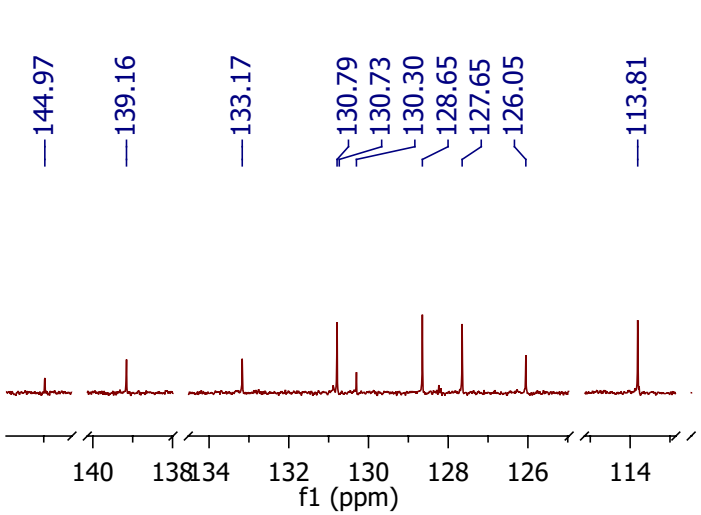


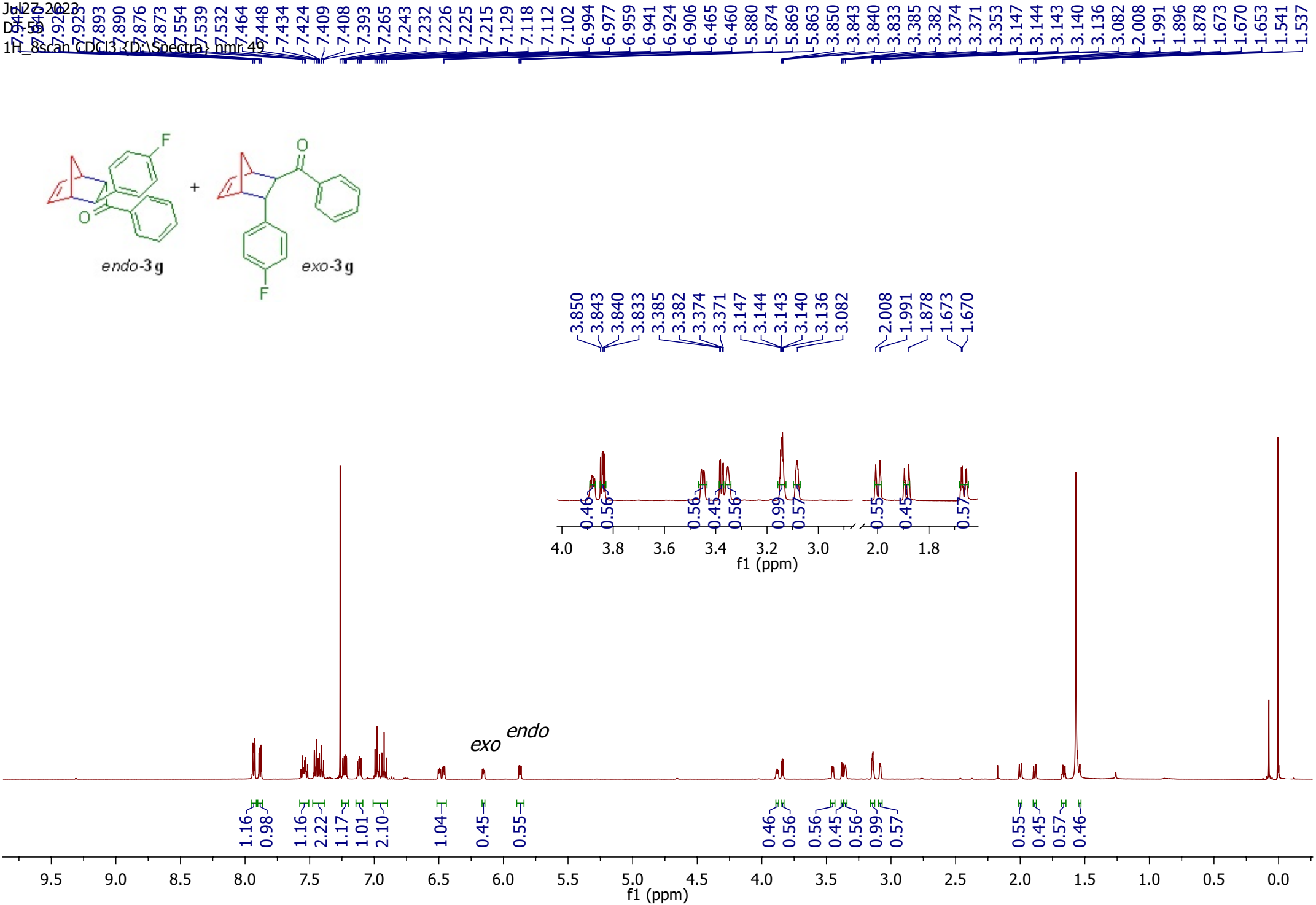
Desktop
DT-53
C13CPD CDCl3 {D:\Spectra} nmr 8

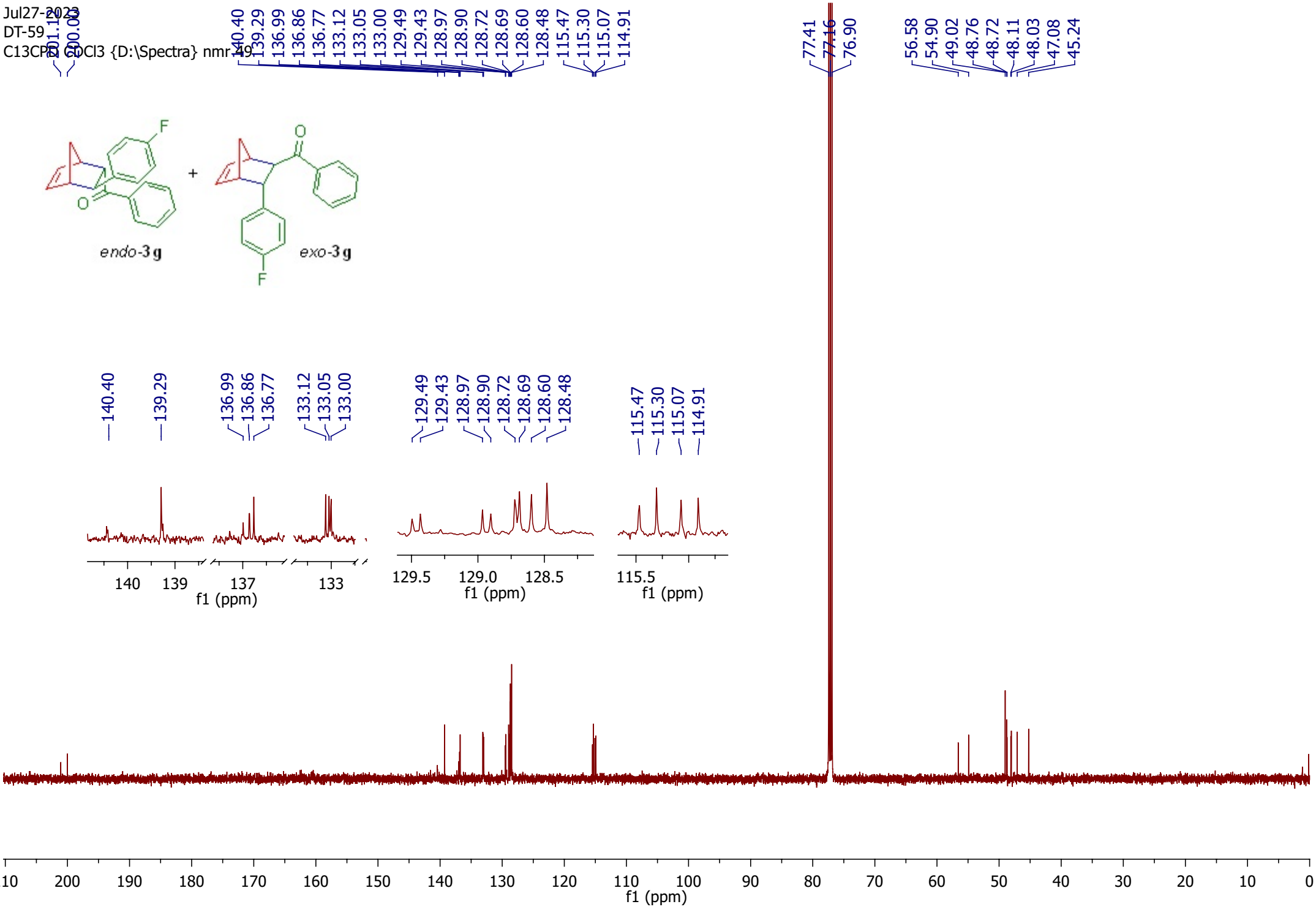
198.53
193.40
163.42
144.97
139.16
133.17
130.79
130.73
130.30
128.65
127.65
126.05
113.81
77.41
77.16
76.91
55.90
55.60
48.89
48.61
48.17
46.02



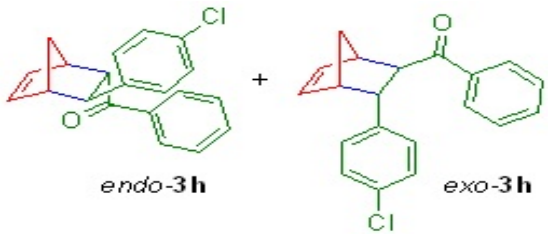
endo-**3f** *exo*-**3f**



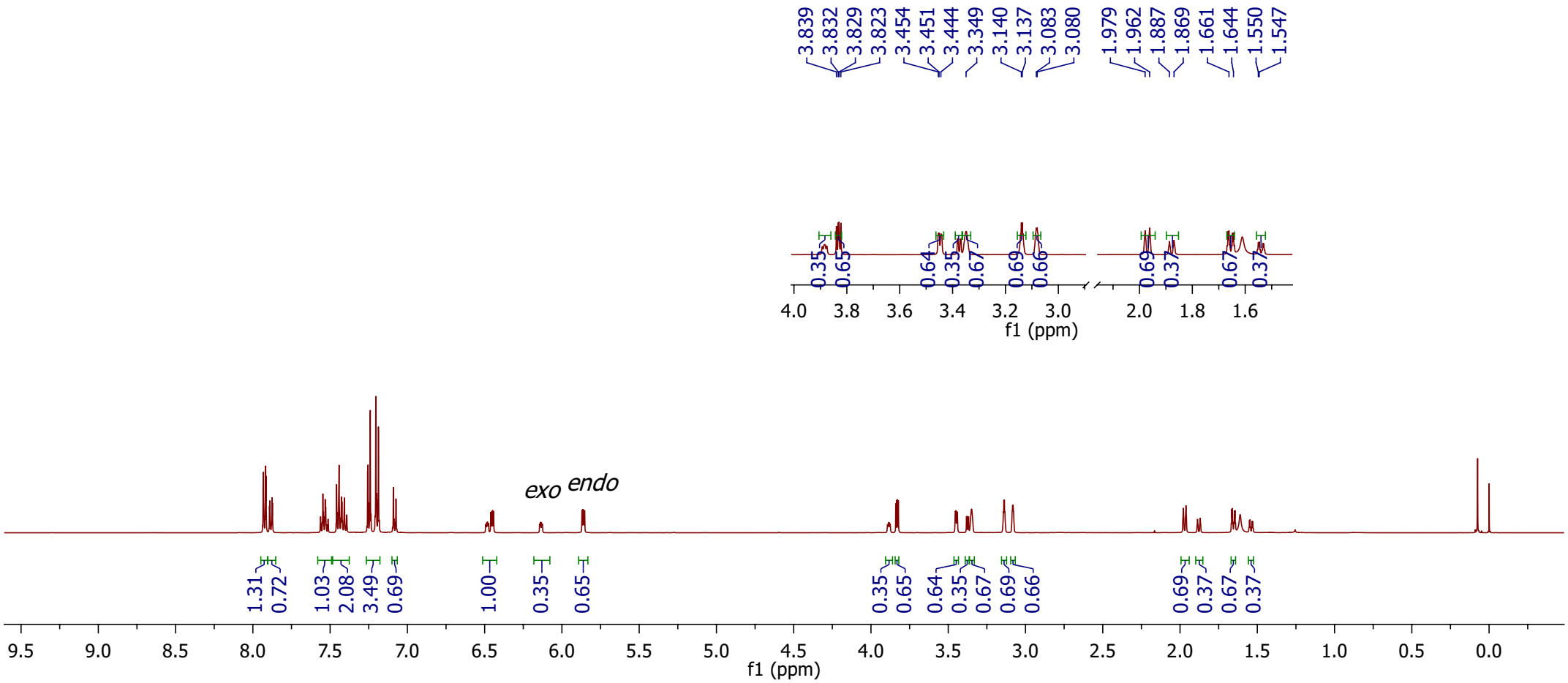

Jul 27 2022
D:\Spectra\nmr\49
1H_8scan CDCl3
endo-3g
exo-3g
exo
endo
f1 (ppm)


Jul27-2023
DT-59
C13CPD CDCl3 {D:\Spectra} nmr-49
201.12
200.00
140.40
139.29
136.99
136.86
136.77
133.12
133.05
133.00
129.49
129.43
128.97
128.90
128.72
128.69
128.60
128.48
115.47
115.30
115.07
114.91
77.41
77.16
76.90
56.58
54.90
49.02
48.76
48.72
48.11
48.03
47.08
45.24
F
O
+
O
F
endo-3g
exo-3g
f1 (ppm)
f1 (ppm)
f1 (ppm)
f1 (ppm)

Jul 26 2023
DMSO
1H_8scan CDCl3 (D:\Spectra) nmr 27

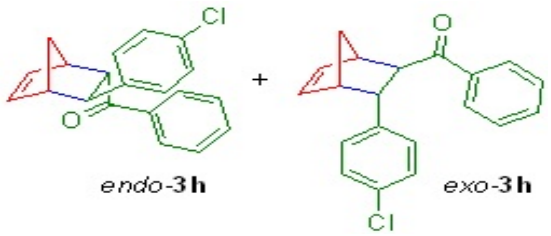


endo-**3h** *exo*-**3h**

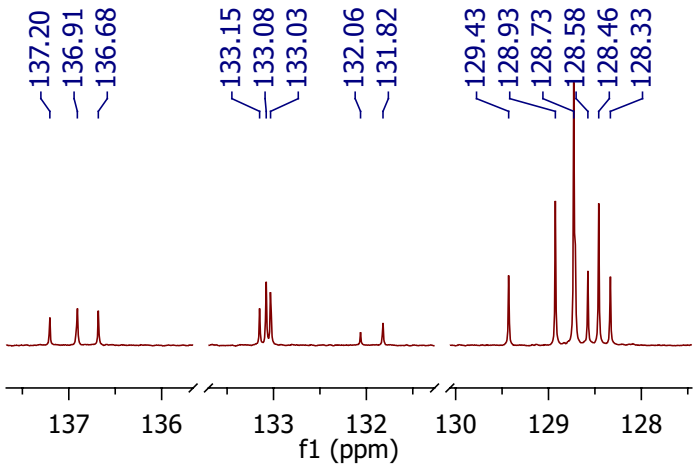


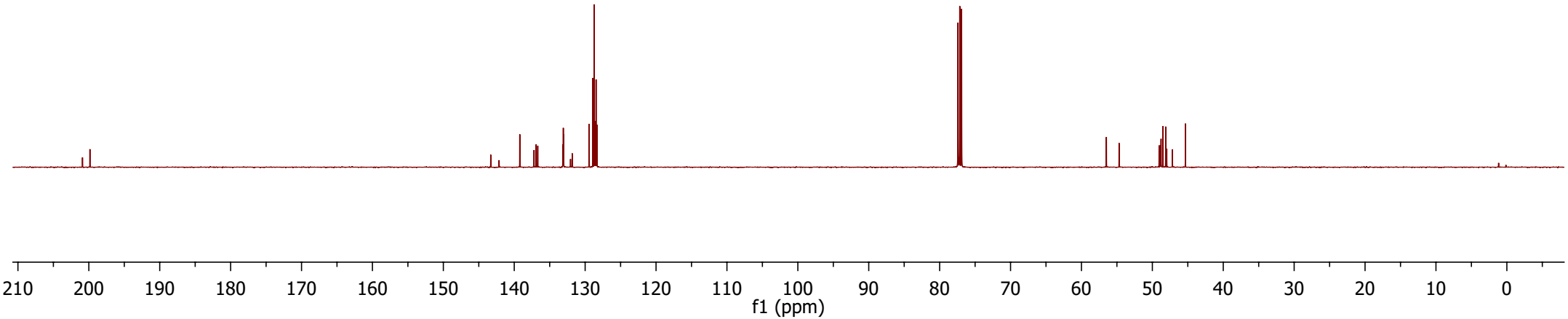
Jul26-2023
DT-61
C13CPD CDCl3 {D:\Spectra} nmr 27

200.90
199.85
143.31
142.13
139.22
137.20
136.91
136.68
133.15
133.08
133.03
132.06
131.82
129.43
128.93
128.73
128.58
128.46
128.33
77.41
77.16
76.91
56.49
54.69
49.05
48.91
48.77
48.52
48.12
47.97
47.17
45.34

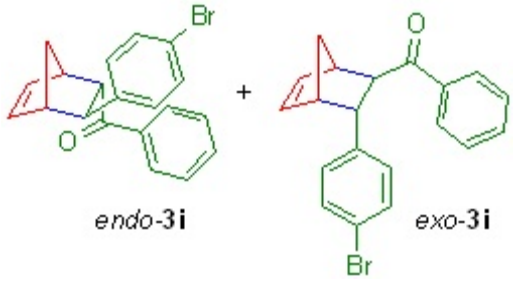


endo-**3h** *exo*-**3h**

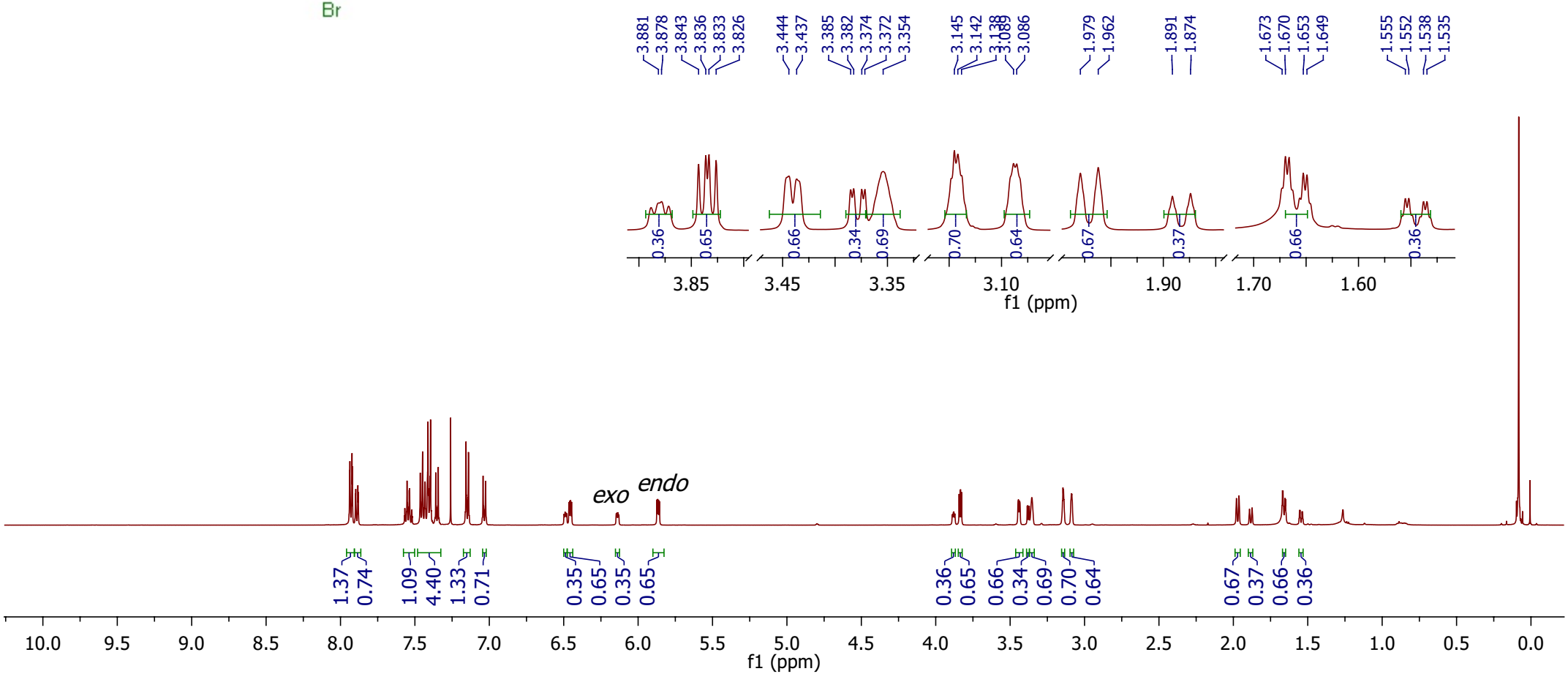




Jul19-2022
DMSO
DMSO-d6 4-Br
1H_8scan CDCl3 (D:\Spectra) nmr 12

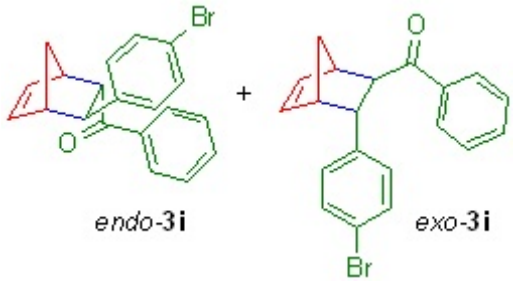


endo-**3i** *exo*-**3i**

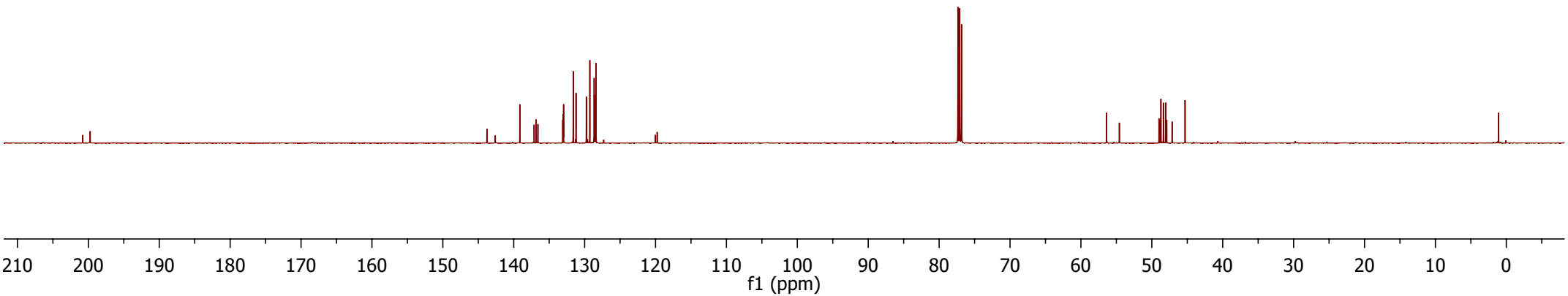


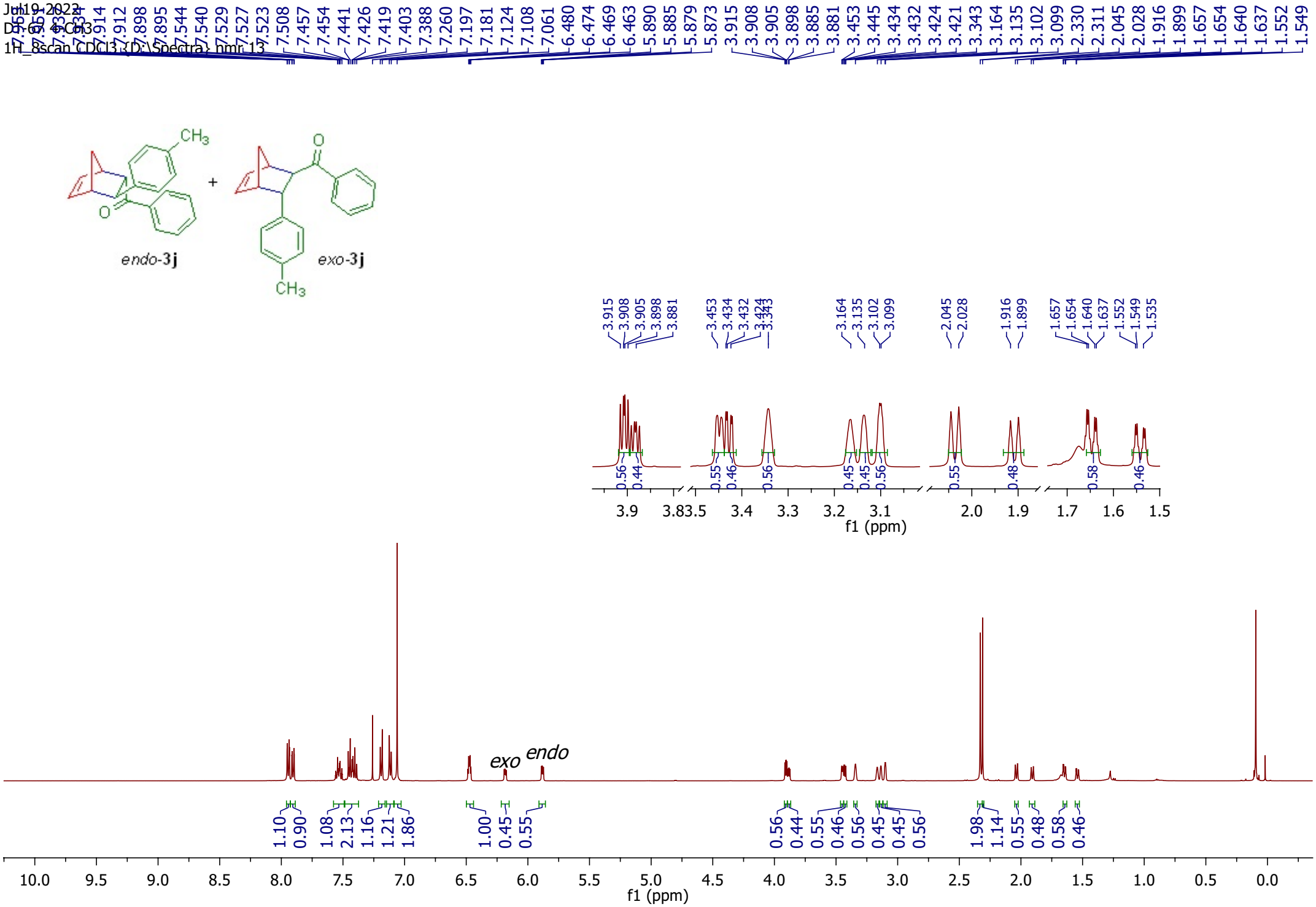
Jul19-2022
DT-66 4-Br
C13CPD CDCl3 {D:\Spectra} nmr 12

200.74
143.78
142.60
139.14
137.12
136.85
136.83
136.59
133.09
133.01
132.97
131.61
131.22
129.76
129.27
128.65
128.50
128.39
120.05
119.77
77.35
77.09
76.84
56.38
54.58
48.99
48.79
48.71
48.37
48.06
47.89
47.14
45.31
1.09

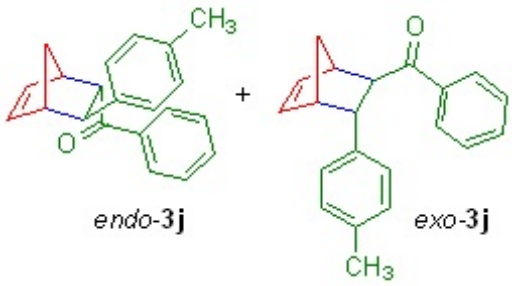


endo-**3i** *exo*-**3i**




Jul 19 2022
D:\6\4\Cm3
1H_8scan CDCl3 (D:\Spectra) nmr 13
endo-3j
exo-3j
exo
endo
f1 (ppm)

Jul19-2022
DT-67 4-CH3
C13CPD CDCl3 {D:\Spectra} nmr 13

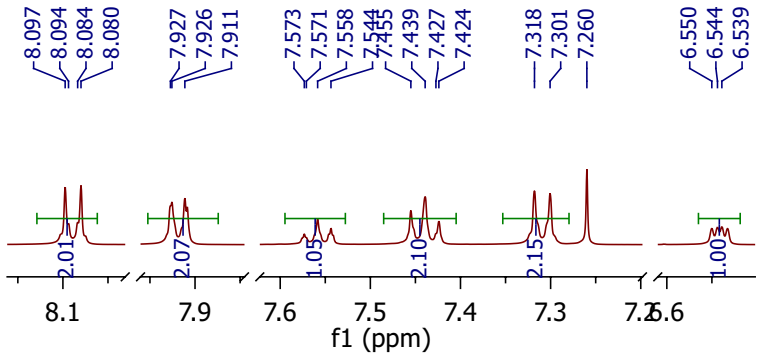


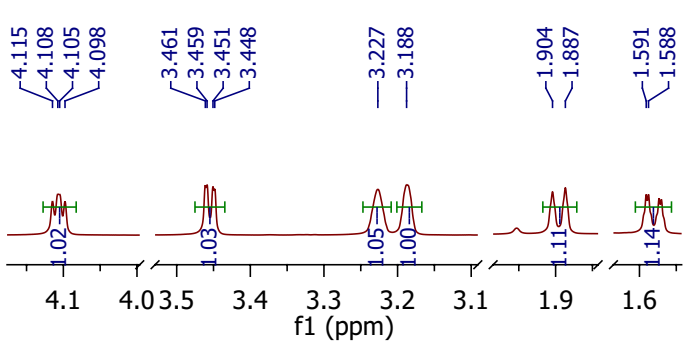
endo-**3j** *exo*-**3j**

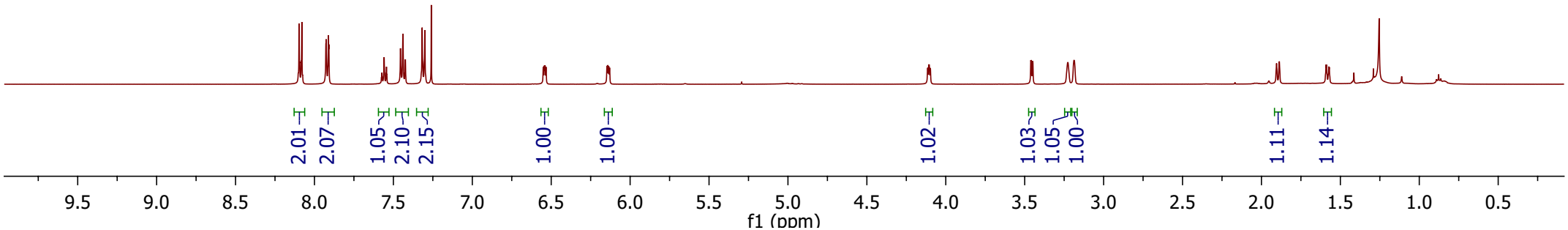
Aug01-2022
DT-69
1H_8scan CDCl3 {D:\Spectra} nmr 2

8.102 8.097 8.094 8.084 8.080 8.075 7.927 7.926 7.911 7.909 7.455 7.439 7.318 7.301 7.260 6.550 6.544 6.539 6.147 6.141 6.135 6.130 4.115 4.108 4.105 4.098 3.461 3.459 3.451 3.448 3.227 3.188 1.904 1.887 1.591 1.588 1.573 1.570

endo-**3k** *exo*-**3k**







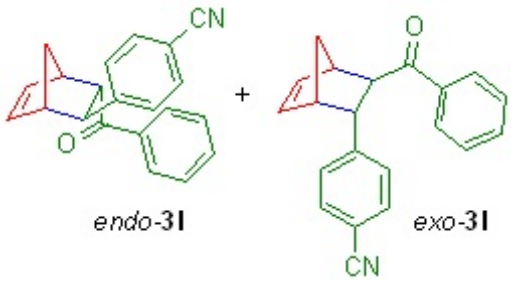
Aug18-2023
DT-68 P.P
C13CPD CDCl3 {D:\Spectra} nmr 35

endo-**3k** *exo*-**3k**

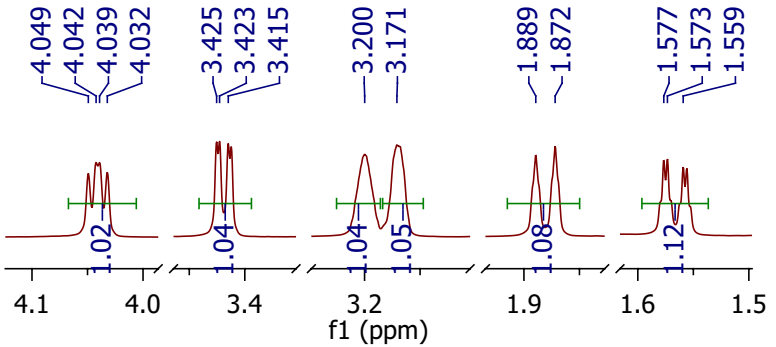
199.23
152.90
146.46
139.13
136.96
133.28
133.26
128.85
128.58
128.48
128.36
123.94
77.41
77.16
76.90
56.70
48.99
48.29
48.13
45.79

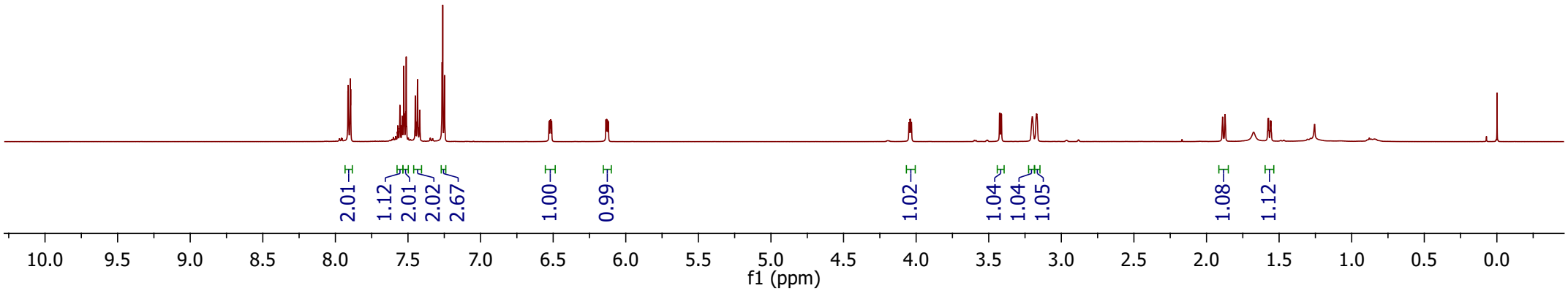
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Jul19-2022
DT-68 4-Cl
1H_8scan CDCl3 {D:\Spectra} nmr 14



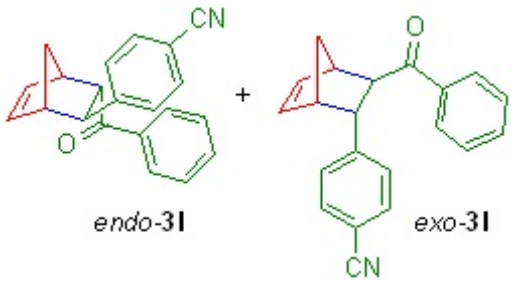
endo-**3l** *exo*-**3l**



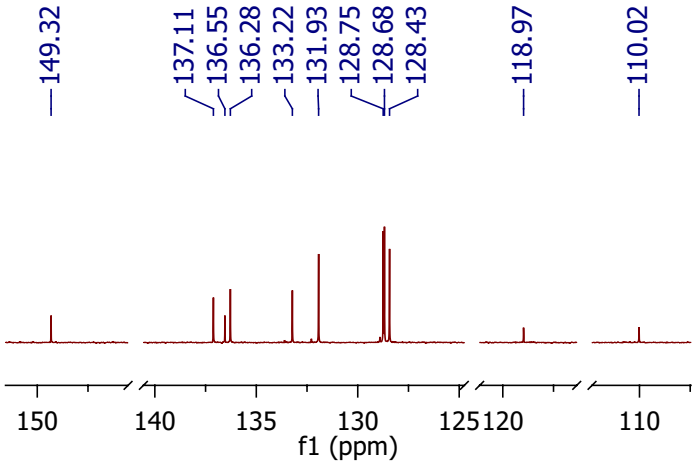


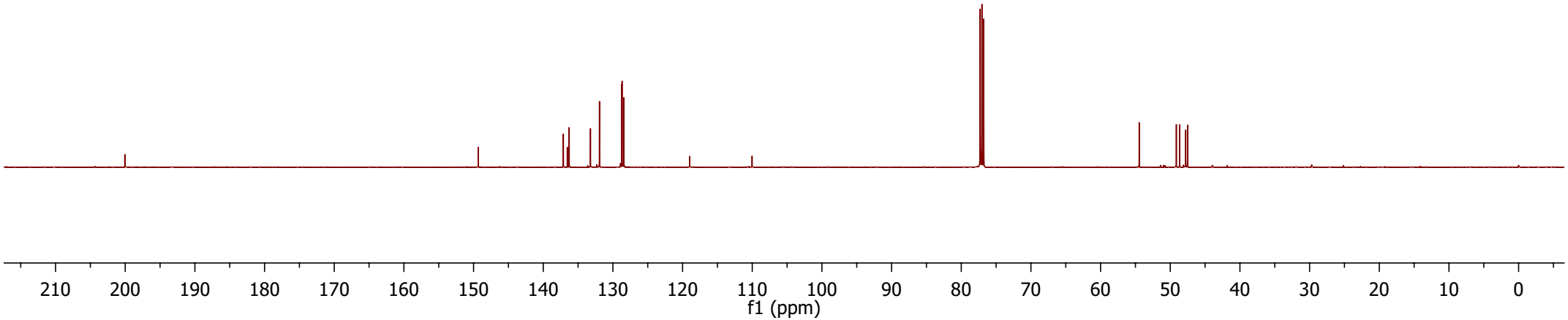
Jul19-2022
DT-68 4-CN
C13CPD CDCl3 {D:\Spectra} nmr 14

200.06 149.32 137.11 136.28 133.22 131.93 128.75 128.68 128.43 118.97 110.02 77.29 77.03 76.78 54.46 49.13 48.65 47.78 47.51

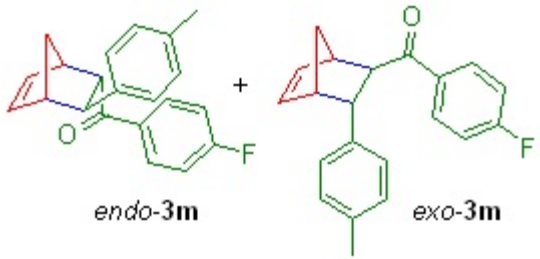


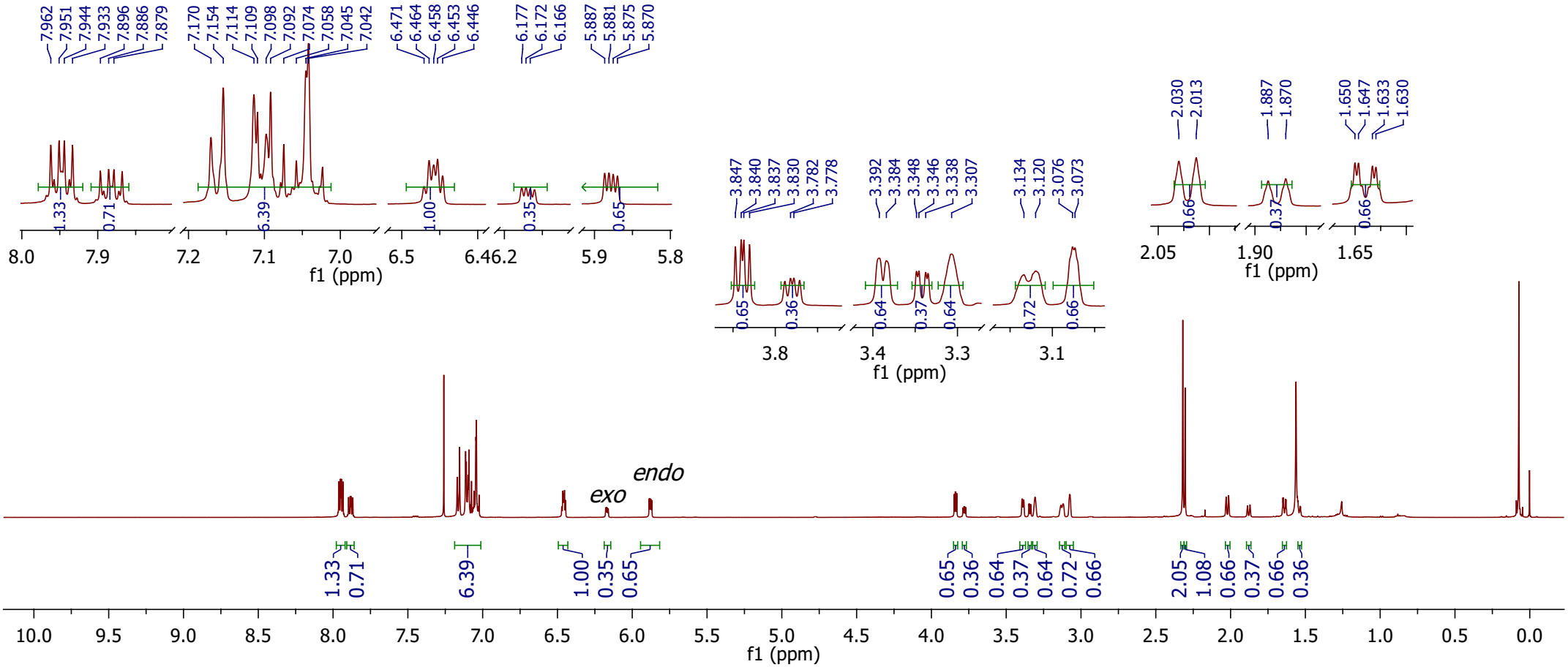
endo-**3l** *exo*-**3l**



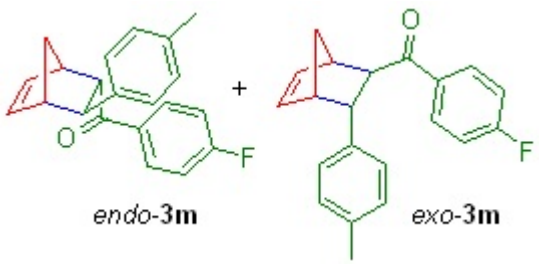


Sep30-2022
D:\8
1H_8scan CDCl3 D:\Spectra\ nmr 6

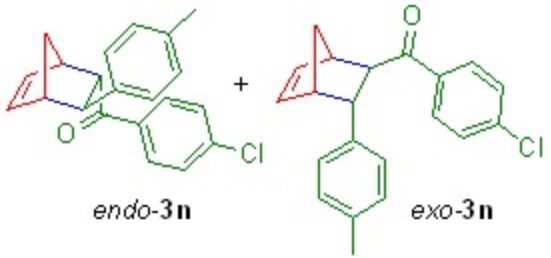




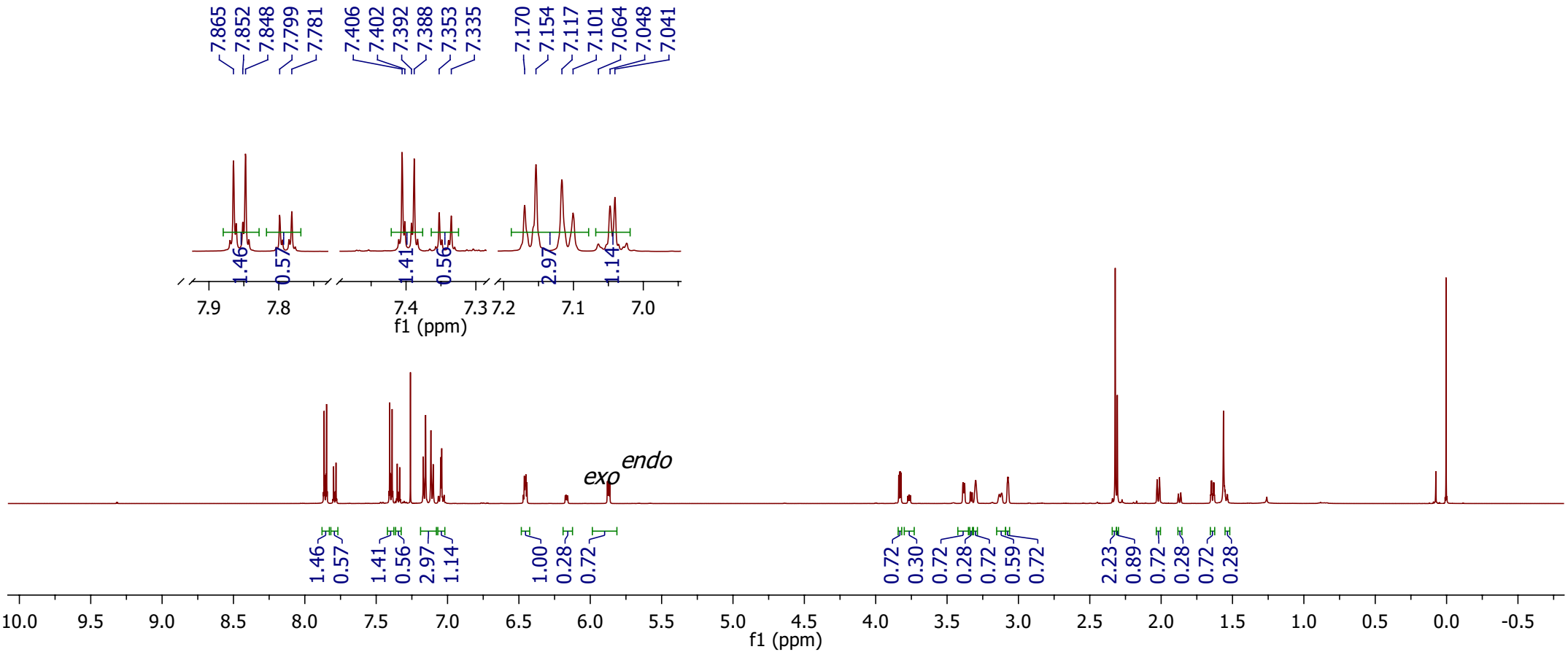
Sep30-2022
DT-86
C13CPD CDCl3 {D:\Spectra} 1m 1 6



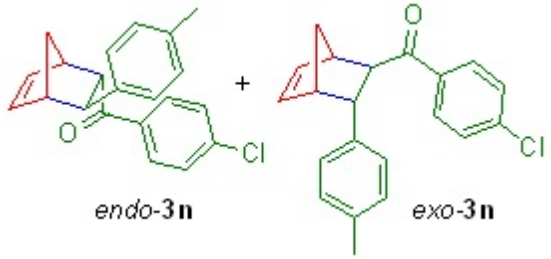
Jul 27 2023
D6-BZ
1H_8scan CDCl3 (D:\Spectra) nmr 48



endo-**3n** *exo*-**3n**



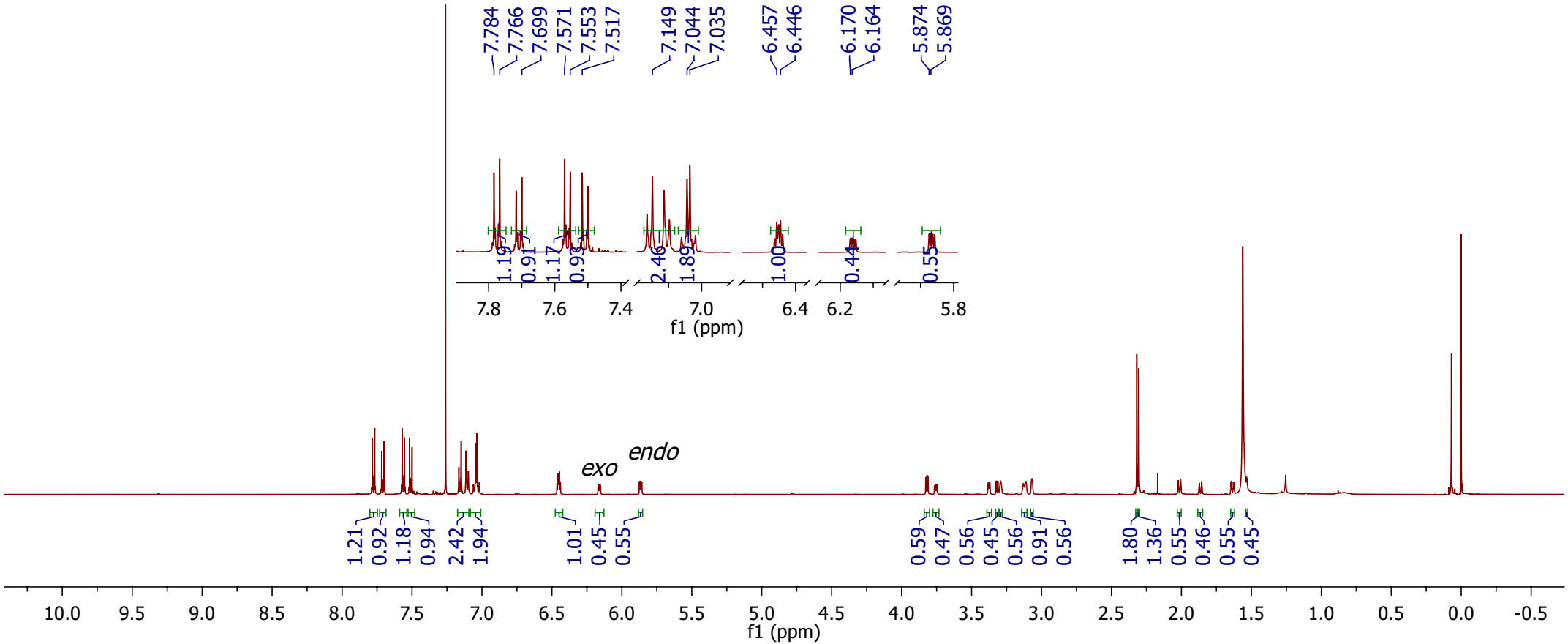
Jul27-2023
DT-87
C13CPD CDCl3 {D:\Spectra} nmr 48



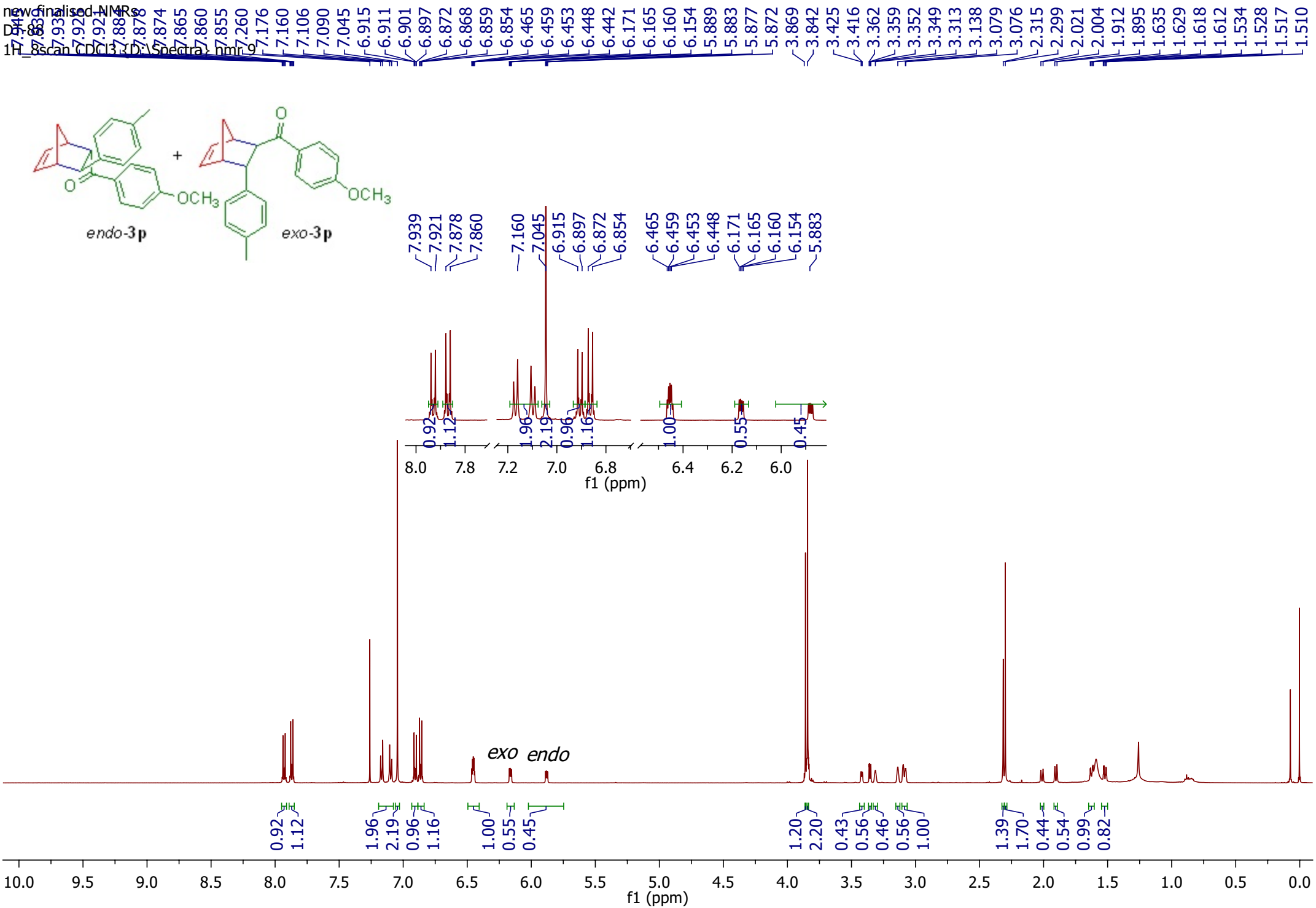
f1 (ppm)

Jul 26 2023
D-91
1H_8scan CDCl3 (D:\Spectra) nmr 26

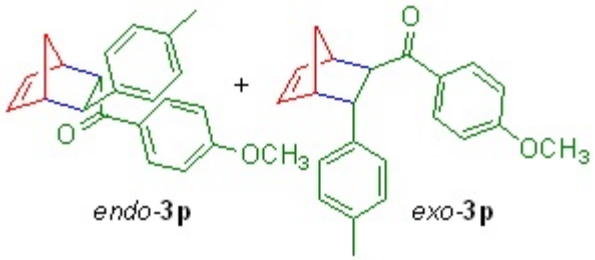
endo-**3o** *exo*-**3o**



Jul26-2023
DT-91
C13CPD CDCl3 {D:\Spectra} nmr 26

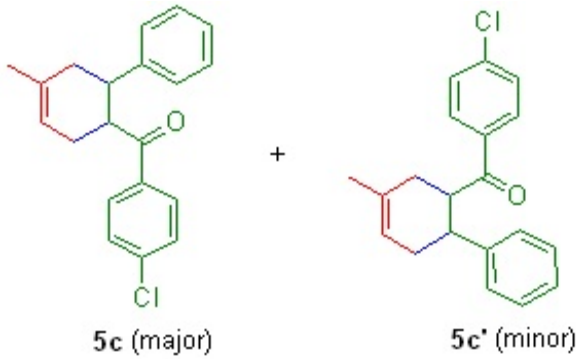

new finalised NMRs
D-88
1H_8scan CDCl3 (D:\Spectra) nmr_9
endo-3p
exo-3p
exo
endo
f1 (ppm)

new finalised NMRs
DT-88
C13CPD CDCl3 {D:\Spectra} nmr 9

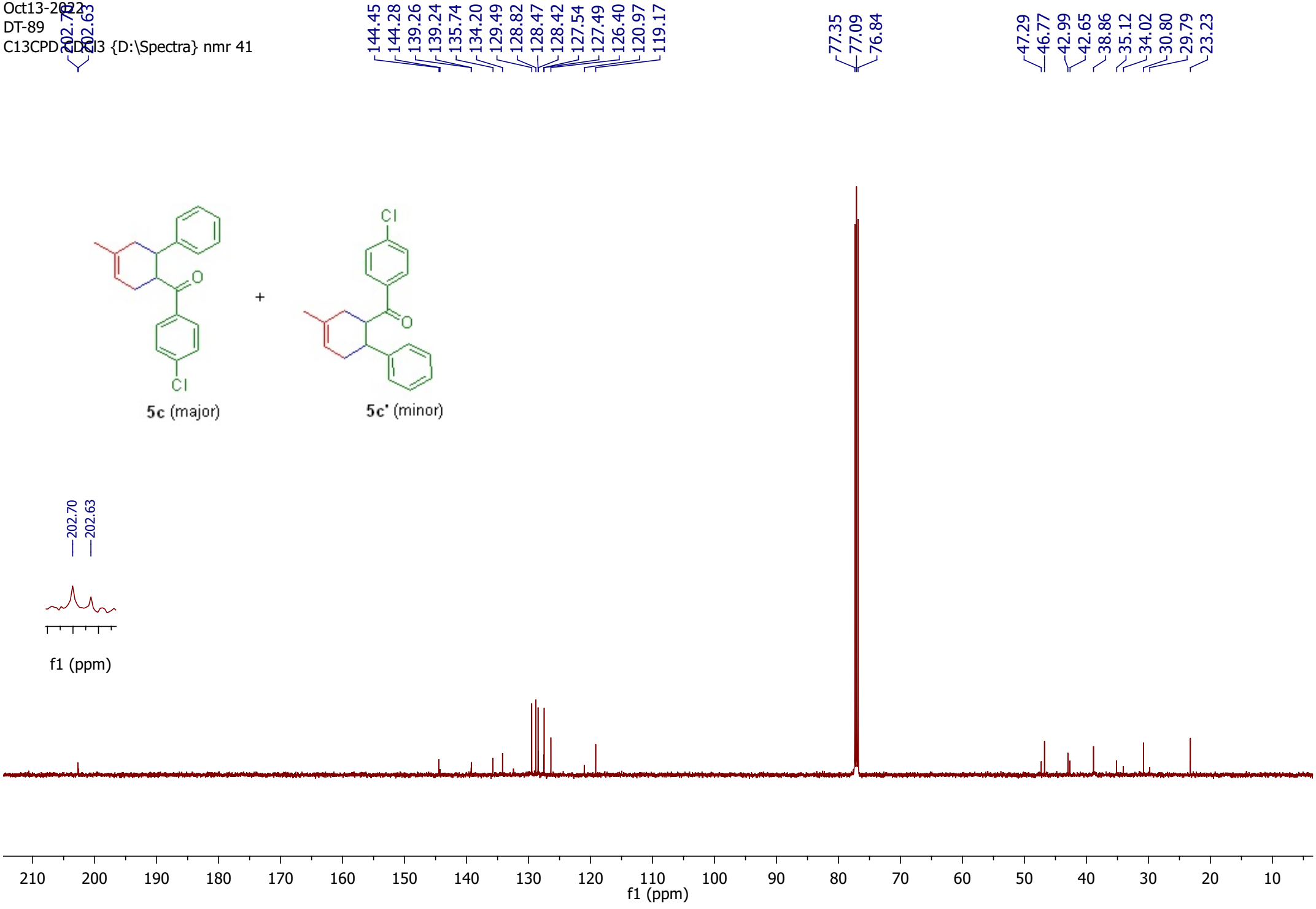


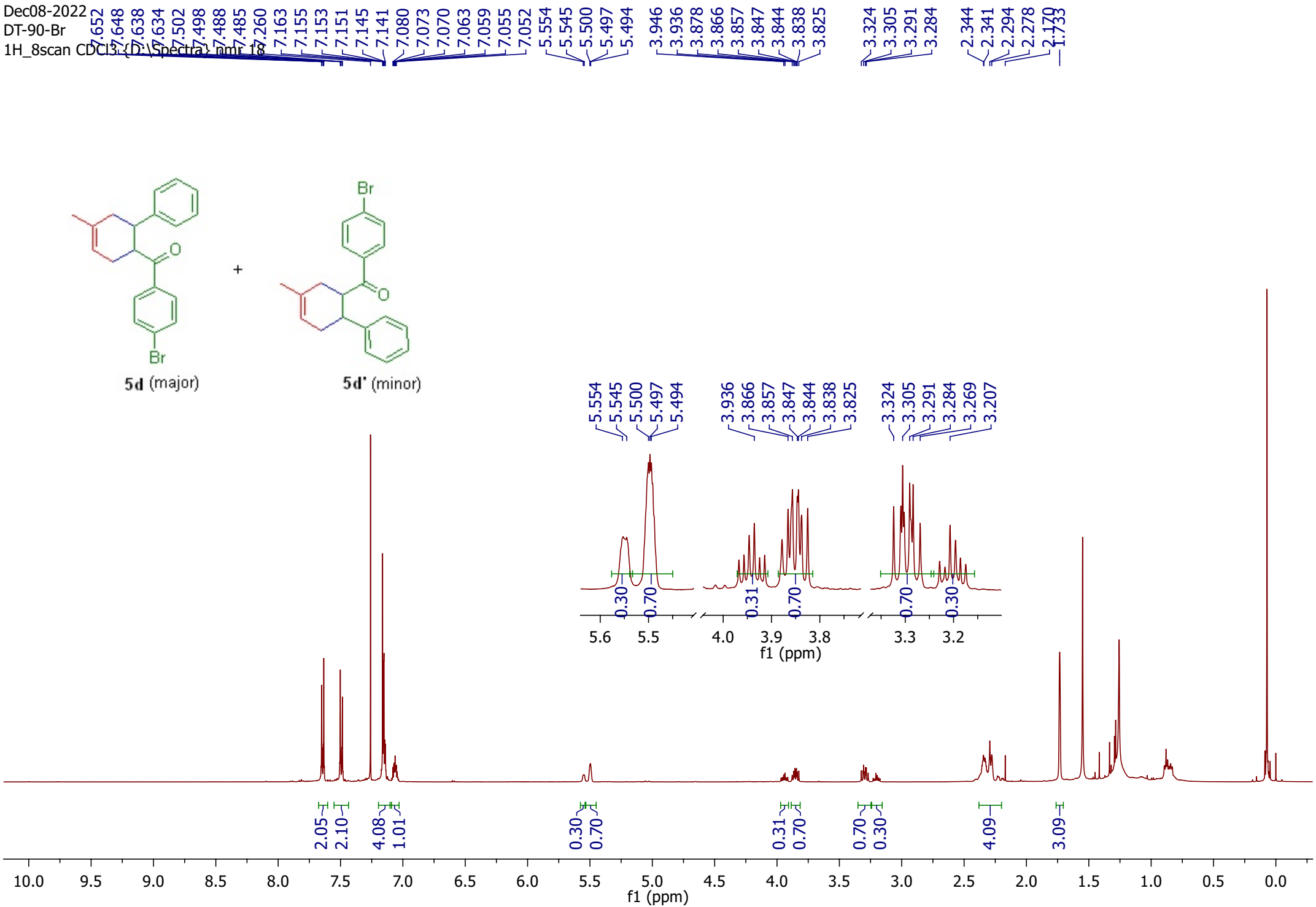
endo-**3p** *exo*-**3p**

Oct13-2022
DT-89
1H_8scan CDCl3 {D:\Spectra} nmr 41



Oct13-2022
DT-89
C13CPD CDCl3 {D:\Spectra} nmr 41




Dec08-2022
DT-90-Br
1H_8scan CDCl3 (D:\Spectra) nmr 18
7.652 7.648 7.638 7.634 7.502 7.498 7.488 7.485 7.260 7.163 7.155 7.153 7.151 7.145 7.141 7.080 7.073 7.070 7.063 7.059 7.055 7.052 5.554 5.545 5.500 5.497 5.494 3.946 3.936 3.878 3.866 3.857 3.847 3.844 3.838 3.825 3.324 3.305 3.291 3.284 2.344 2.341 2.294 2.278 2.170 1.733
Br
O
+
Br
O
5d (major)
5d' (minor)
5.554 5.545 5.500 5.497 5.494 3.936 3.866 3.857 3.847 3.844 3.838 3.825 3.324 3.305 3.291 3.284 3.269 3.207
0.30 0.70 0.31 0.70 0.70 0.30
5.6 5.5 4.0 3.9 3.8 3.3 3.2
f1 (ppm)
2.05 2.10 4.08 1.01 0.30 0.70 0.31 0.70 0.70 0.30 4.09 3.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

Dec08-2022
DT-90-Br
C13CPD CDCl3 {D:\Spectra} nmr 18