

Ultrasound-Generated Cyclodextrin–Amino Acid Inclusion Complex with NH₃⁺-Mediated Chalcone Activation: Metal-Free Catalysis Surpassing Nanoparticles

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1. EXPERIMENTAL SECTION

1.1 Materials & Methods

All reagents were used as purchased from commercial suppliers without further purification. Chalcones were synthesized as per the procedure reported in literature.^{1,2} FT-IR spectra were recorded using Bruker alpha Eco-ATR spectrometer in the spectral region of 4000-650 cm⁻¹. ¹H-NMR spectra were recorded on Bruker Avance Neo 500 MHz spectrometer in CDCl₃ and/or D₂O solvents. 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). ¹³C NMR spectra were recorded at 125 MHz in CDCl₃ and/or D₂O relative to trimethylsilane as internal standard. Reference peak for solvents were set as follows: in ¹H-NMR: CDCl₃ = 7.26 ppm, D₂O = 4.79 ppm; in ¹³C-NMR: CDCl₃ = 77.0 ppm. Chiral HPLC analysis was carried out on Shimadzu L202260 instrument by using chiral columns. Pure products were isolated and purified by column chromatography over 60-120 mesh size silica gel using hexane: EtOAc as eluent. The progress of the reaction was monitored by thin layer chromatography (TLC) on silica coated aluminum plates F₂₅₄ and visualized under UV chamber.

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

2. CHARACTERISATION DATA OF DA ADDUCTS

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

Results: 97% yield, pale-yellow oily liquid, dr = 72:28 (*endo:exo*); **IR (ATR):** ν_{\max} 3054, 3026, 2998, 2974, 1665, 1598, 1445, 1331, 1213, 1018, 746, 696, 664 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 7.93-7.89 (m, 1.46H (*endo*)), 7.88-7.86 (m, 0.56H (*exo*)), 7.53-7.47 (m, 1H), 7.44-7.27 (m, 4H), 7.25-7.12 (m, 3H), 6.47-6.43 (m, 1H), 6.14 (dd, $J = 3.0$ Hz, 8.5 Hz, 0.28H (*exo*)), 5.85 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.72H (*endo*)), 3.88 (dd, $J = 3.5$ Hz, 5.0 Hz, 1H), 3.45 (dd, $J = 1.5$ Hz, 4.5 Hz, 0.72H (*endo*)), 3.41 (dd, $J = 1.5$ Hz, 5.5 Hz, 0.28H (*exo*)), 3.32 (s, 0.72H (*endo*)), 3.15 (s, 0.28H (*exo*)), 3.12-3.08 (m, 1H), 2.01 (dd, $J = 1.5$ Hz, 6 Hz, 0.72H (*endo*)), 1.87 (dd, $J = 1.0$ Hz, 6 Hz, 0.28H (*exo*)), 1.62 (dq, $J = 2.0$, 8.5 Hz, 0.72H (*endo*)), 1.51 (dq, $J = 1.5$ Hz, 8.5 Hz, 0.28H (*exo*)) ppm; **$^{13}\text{C NMR}$ (125 MHz, CDCl_3):** δ 201.7, 200.1, 144.8, 143.6, 139.3, 137.4, 137.1, 137.0, 136.6, 133.1, 133.0, 132.9, 128.7, 128.6, 128.5, 128.3, 128.2, 127.6, 126.3, 126.1, 56.3, 54.6, 49.0, 48.9, 48.7, 48.6, 48.1, 48.0, 47.8, 46.0 ppm.

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

Results: 95% yield, colorless oily liquid, dr = 74:26 (*endo:exo*); **IR (ATR):** ν_{\max} 3057, 3029, 2988, 1678, 1499, 1451, 1331, 1216, 1014, 833, 742, 669 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 7.90-7.86 (m, 1.57H (*endo*)), 7.83-7.78 (m, 0.53H (*exo*)), 7.24-7.09 (m, 5H), 7.04-6.93 (m, 2H), 6.41-6.37 (m, 1H), 6.09 (dd, $J = 3.0$ Hz, 5.0 Hz, 0.26H (*exo*)), 5.80 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.74H (*endo*)), 3.78 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.74H (*endo*)), 3.75 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.26H (*exo*)), 3.36 (dd, $J = 5.0$ Hz, 8.5 Hz, 0.74H (*endo*)), 3.29 (dd, $J = 4.0$ Hz, 8.5 Hz, 0.26H (*exo*)), 3.24 (s, 0.74H (*endo*)), 3.09 (s, 0.26H (*exo*)), 3.06-3.02 (m, 1H), 1.95 (d, $J = 8.5$ Hz, 0.74H (*endo*)), 1.85 (d, $J = 9.0$ Hz, 0.26H (*exo*)), 1.58 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.76H (*endo*)), 1.47 (dd, $J = 1.0$ Hz, 8.5 Hz, 0.24H (*exo*)) ppm; **$^{13}\text{C NMR}$ (125 MHz, CDCl_3):** δ 199.8, 198.6, 165.8 (d, $J = 253.7$ Hz), 165.7 (d, $J = 252.5$ Hz), 144.6, 143.4, 139.4, 137.0, 136.6, 133.7 (d, $J = 3.7$ Hz), 133.5 (d, $J = 3.7$ Hz), 133.0, 131.2 (d, $J = 6.2$ Hz), 131.1 (d, $J = 6.2$ Hz), 128.7, 128.3, 128.1, 127.6, 126.5, 126.2, 115.8 (d, $J = 12.5$ Hz), 115.6 (d, $J = 15.0$ Hz), 56.2, 54.6, 49.1, 48.8, 48.7, 48.7, 48.2, 48.1, 46.2 ppm. δ -105.69 (*exo*), -105.80 (*endo*) ppm

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

Results: 92% yield, colorless oily liquid, dr = 71:29 (*endo:exo*); **IR (ATR):** ν_{\max} 3054, 3027, 2994, 2973, 1665, 1594, 1443, 1215, 1015, 743, 691, 664 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 7.85 (dt, $J = 2.5$ Hz, 9.0 Hz, 1.48H (*endo*)), 7.78 (dt, $J = 2.5$ Hz, 9.0 Hz, 0.60H (*exo*)), 7.39

(dt, $J = 2.5$ Hz, 9.0 Hz, 1.43H (*endo*)), 7.33 (dt, $J = 2.5$ Hz, 9.0 Hz 0.63H (*exo*)), 7.31-7.12 (m, 5H), 6.48-6.44 (m, 1H), 6.16 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.29H (*exo*)), 5.87 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.71H (*endo*)), 3.84 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.71H (*endo*)), 3.81 (dd, $J = 4.0$ Hz, 5.0 Hz, 0.29H (*exo*)), 3.43 (dd, $J = 1.5$ Hz, 5.0 Hz, 0.71H (*endo*)), 3.35 (dd, $J = 1.5$ Hz, 5.0 Hz 0.29H (*exo*)), 3.31 (s, 0.71H (*endo*)), 3.16 (s, 0.29H (*exo*)), 3.13-3.10 (m, 1H), 2.02 (d, $J = 8.5$ Hz, 0.71H (*endo*)), 1.87 (d, $J = 9.0$ Hz, 0.29H (*exo*)), 1.65 (dd, $J = 1.5$ Hz, 7.0 Hz 0.71H (*endo*)), 1.54 (d, $J = 1.5$ Hz, 0.29H (*exo*)) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 200.2, 199.0, 144.6, 143.4, 139.5, 139.4, 139.4, 137.0, 136.6, 136.1, 135.6, 135.3, 133.0, 129.9, 129.0, 128.9, 128.7, 128.3, 128.1, 128.0, 127.6, 126.5, 126.2, 56.3, 54.7, 49.1, 48.7, 48.6, 48.2, 48.1, 46.2 ppm.

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

Results: 89% yield, colorless oily liquid, dr = 69:31 (*endo:exo*); **IR (ATR):** ν_{max} 3058, 3021, 2964, 1742, 1678, 1459, 1362, 1223, 1015, 793, 715, 694 cm^{-1} ; **^1H -NMR (500 MHz, CDCl_3):** δ 7.78 (dd, $J = 2.0$ Hz, 5.0 Hz, 1.44H (*endo*)), 7.71 (dd, $J = 2.0$ Hz, 5.0 Hz, 0.64H (*exo*)), 7.56 (dd, $J = 2.0$ Hz, 6.5 Hz, 1.36H (*endo*)) 7.51 (dd, $J = 1.5$ Hz, 6.5 Hz, 0.68H (*exo*)), 7.32-7.27 (m, 3H), 7.25-7.12 (m, 2H), 6.48-6.44 (m, 1H), 6.16 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.31H (*exo*)), 5.87 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.69H (*endo*)), 3.84 (dd, $J = 3.0$ Hz, 5.0 Hz, 0.69H (*endo*)), 3.81 (dd, $J = 3.0$ Hz, 5.0 Hz, 0.31H (*exo*)), 3.43 (dd, $J = 1.0$ Hz, 4.5 Hz, 0.69H (*endo*)), 3.35 (dd, $J = 1.0$ Hz, 4.5 Hz, 0.31H (*exo*)), 3.31 (s, 0.69H (*endo*)), 3.16 (s, 0.31H (*exo*)), 3.14-3.10 (m, 1H), 2.02 (d, $J = 8.5$ Hz, 0.69H (*endo*)), 1.87 (d, $J = 8.5$ Hz, 0.31H (*exo*)), 1.65 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.69H (*endo*)), 1.55 (d, $J = 1.5$ Hz, 0.31H (*exo*)) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 200.4 199.2, 144.5, 143.3, 139.4, 137.0, 136.6, 136.0, 135.8, 133.0, 132.0, 130.2, 130.1, 128.7, 128.4, 128.2, 128.1, 128.1, 127.6, 126.5, 126.2, 56.3, 54.7, 49.1, 48.7, 48.7, 48.6, 48.2, 48.2, 46.2 ppm.

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

Results: 91% yield, yellow oily liquid, dr = 40:60 (*endo:exo*); **IR (ATR):** ν_{max} 3021, 2956, 2922, 1741, 1667, 1607, 1443, 1362, 1225, 1012, 813, 742, 692 cm^{-1} ; **^1H -NMR (500 MHz, CDCl_3):** δ 7.91 (dt, $J = 5.0$ Hz, 9.5 Hz, 0.86H (*endo*)) 7.86 (dd, $J = 5.0$ Hz, 9.5 Hz, 1.21H (*exo*)), 7.31-7.25 (m, 2H), 7.23-7.13 (m, 3H), 6.90 (dd, $J = 2.0$ Hz, 7.0 Hz, 0.84H (*endo*)), 6.85 (dd, $J = 2.0$ Hz, 7.0 Hz, 1.23H (*exo*)), 6.47-6.44 (m, 1H), 6.16 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.60H (*exo*)), 5.88 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.40H (*endo*)), 3.90-3.85 (m, 2.2H {1.2H, -OCH₃ (*endo*) + 1H (*endo* + *exo*)}), 3.83 (s, 1.8H, -OCH₃ (*exo*)), 3.46 (dd, $J = 1.5$ Hz, 5.0 Hz, 0.40H (*endo*)), 3.38 (dd, $J = 1.5$ Hz, 5.0 Hz, 0.60H (*exo*)), 3.32 (s, 0.40H (*endo*)), 3.16 (s, 0.60H (*exo*)), 3.11

(s, 1H), 2.01 (d, $J = 8.5$ Hz, 0.40H(*endo*)), 1.91(d, $J = 8.5$ Hz, 0.60H(*exo*)), 1.63 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.40H(*endo*)), 1.53 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.60H(*exo*)) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 199.8, 198.5, 163.5, 145.0, 143.8, 139.2, 136.9, 136.7, 133.2, 130.9, 130.8, 130.3, 130.1, 128.6, 128.2, 128.2, 127.7, 126.3, 126.1, 113.8 113.8, 55.9, 55.9, 55.6 55.6, 54.2, 49.1, 48.9, 48.6, 48.2, 48.1, 48.0, 46.0 ppm.

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

Results: 93% yield, colorless oily liquid, dr = 68:32 (*endo:exo*); IR (ATR): ν_{max} 3057, 3024, 2984, 1672, 1491, 1452, 1331, 1213, 1014, 832, 743, 701 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 7.92 (dd, $J = 1.5$ Hz, 7.0 Hz, 1.39H (*endo*)), 7.87 (dd, $J = 1.5$ Hz, 7.0 Hz, 0.64H (*exo*)), 7.57-7.50 (m, 1H), 7.47-7.37 (m, 2H), 7.24-7.20 (m, 1.39H), 7.13-7.09 (m, 0.68H), 7.00-6.89 (m, 2H), 6.48 (dd, $J = 3.0$ Hz, 5.0 Hz, 0.32H (*exo*)), 6.45 (dd, $J = 3.0$ Hz, 5.0 Hz, 0.68H (*endo*)), 6.14 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.32H(*exo*)), 5.86(dd, $J = 3.0$ Hz, 5.0 Hz, 0.68H (*endo*)), 3.87 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.32H (*exo*)), 3.83 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.68H (*endo*)), 3.44 (d, $J = 4.5$ Hz, 0.68H (*endo*)), 3.36 (dd, $J = 1.5$ Hz, 5.5 Hz, 0.32H (*exo*)), 3.35 (s, 0.68H (*endo*)), 3.15-3.13 (m, 0.64H), 3.07 (s, 0.68H (*endo*)), 1.98 (d, $J = 8.5$ Hz, 0.55H (*endo*)), 1.87 (d, $J = 9.0$ Hz, 0.45H (*exo*)), 1.65 (dd, $J = 1.5$ Hz, 6.5 Hz, 0.68H (*endo*)), 1.53 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.32H (*exo*)) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 200.8, 200.0, 161.5 (d, $J = 248.7$ Hz), 161.4 (d, $J = 248.7$ Hz), 140.4 (d, $J = 3.7$ Hz), 139.3, 137.3, 137.0, 136.8, 136.7, 133.1, 133.0 (d, $J = 3.7$ Hz), 129.4 (d, $J = 7.5$ Hz), 128.9 (d, $J = 7.5$ Hz), 128.7, 128.7, 128.6, 128.5, 115.4 (d, $J = 20.0$ Hz), 114.9 (d, $J = 20.0$ Hz), 56.6, 54.9, 49.0, 48.7, 48.7 48.1, 48.0, 47.1, 45.2 ppm. ^{19}F NMR (470 MHz, CDCl_3): δ -117.76 (*exo*), -117.48 (*endo*) ppm

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

Results: 92% yield, yellow oily liquid, dr = 63:37 (*endo:exo*); IR (ATR): ν_{max} 2953, 2927, 1731, 1672, 1453, 1254, 1014, 795, 734, 692 cm^{-1} ; ^1H -NMR (500 MHz, CDCl_3): δ 7.92 (dd, $J = 1.5$ Hz, 8.5 Hz, 1.29H (*endo*)), 7.88 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.71H (*exo*)), 7.57- 7.50 (m, 1H), 7.46- 7.38 (m, 2H), 7.27-7.23 (m, 1.41H), 7.21-7.17 (m, 2H), 7.07(dd, $J = 1.5$ Hz, 7.0 Hz, 0.75H), 6.48 (dd, $J = 2.0$ Hz, 7.0 Hz, 0.37H(*exo*)), 6.45 (dd, $J = 2.0$ Hz, 7.0 Hz, 0.63H(*endo*)), 6.14-6.12 (m, 0.37H (*exo*)), 5.86 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.63H (*endo*)), 3.88 (dd, $J = 3.5$ Hz, 8.5 Hz, 0.37H), 3.83 (dd, $J = 3.5$ Hz, 8.5 Hz, 0.63H (*endo*)), 3.44 (dd, $J = 1.5$ Hz, 5.0 Hz, 0.63H (*endo*)), 3.37 (dd, $J = 1.5$ Hz, 5.0 Hz, 0.37H (*exo*)), 3.34 (s, 0.67H), 3.15-3.12 (m, 0.74H), 3.10-3.05 (m, 0.63H), 1.96 (d, $J = 8.5$ Hz, 0.63H (*endo*)), 1.87 (d, $J = 9.0$ Hz, 0.37H (*exo*)), 1.65 (dd, $J = 3.5$ Hz, 8.5 Hz 0.63H (*endo*)), 1.54 (dd, $J = 1.5$ Hz, 8.5 Hz, 0.37H (*exo*)) ppm; ^{13}C

NMR (125 MHz, CDCl₃): δ 200.9, 199.8, 143.3, 142.1, 139.2, 137.2, 136.9, 136.7, 133.1, 133.1, 133.0, 132.1, 131.8, 129.4, 128.9, 128.8, 128.7, 128.6, 128.5, 128.3, 56.5, 54.7, 49.0, 48.9, 48.8, 48.5, 48.1, 48.0, 47.2, 45.3 ppm.

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

Results: 89% yield, yellow oily liquid, dr = 55:45 (*endo:exo*); **IR (ATR):** ν_{\max} 3062, 2964, 2923, 1672, 1592, 1442, 1333, 1257, 1213, 1015, 814, 752, 723, 691 cm⁻¹; **¹H-NMR (500 MHz, CDCl₃):** δ 7.92 (dd, *J* = 2.0 Hz, 7.0 Hz, 1.17H (*endo*)), 7.89 (dd, *J* = 2.0 Hz, 7.0 Hz, 0.88H (*exo*)), 7.57-7.51 (m, 1H), 7.47-7.33 (m, 4H), 7.14 (dd, *J* = 2.5 Hz, 10.5 Hz 1.13H), 7.03 (dd, *J* = 1.0 Hz, 7.0 Hz, 0.91H), 6.48 (dd, *J* = 3.5 Hz, 5.5 Hz, 0.45H (*exo*)), 6.45 (dd, *J* = 3.5 Hz, 6.0 Hz, 0.55H (*endo*)), 6.13 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.45H (*exo*)), 5.86 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.55H (*endo*)), 3.87 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.45H (*exo*)), 3.83 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.55H (*endo*)), 3.43 (d, *J* = 4.0 Hz, 0.55H (*endo*)), 3.37 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.45H (*exo*)), 3.35 (s, 0.55H), 3.15-3.12 (m, 0.85H), 3.08 (d, *J* = 1.5 Hz, 0.55H (*endo*)), 1.96 (d, *J* = 8.5 Hz, 0.55H (*endo*)), 1.87 (d, *J* = 8.5 Hz, 0.45H (*exo*)), 1.66 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.65H (*endo*)), 1.53 (d, *J* = 1.5 Hz, 0.45H (*exo*)) ppm; **¹³C NMR (125 MHz, CDCl₃):** δ 200.9, 199.8, 139.2, 137.2, 136.9, 136.7, 133.2, 133.1, 133.1, 131.7, 131.3, 130.3, 129.8, 128.7, 128.6, 128.5, 56.5, 54.7, 49.1, 48.9, 48.8, 48.5, 48.1, 48.0, 47.2, 45.4 ppm.

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

Results: 91% yield, yellow oily liquid dr = 68:32 (*endo:exo*); **IR (ATR):** ν_{\max} 3051, 2973, 1672, 1592, 1444, 1325, 1204, 1013, 805, 766, 789, 662 cm⁻¹; **¹H-NMR (500 MHz, CDCl₃):** δ 7.95- 7.92 (m, 1.40H (*endo*)), 7.91-7.88 (m, 0.64H (*exo*)), 7.56-7.49 (m, 1H), 7.46-7.37 (m, 2H), 7.18-7.09(m, 3H), 7.05 (s, 1H), 6.50 - 6.45 (m, 1H), 6.18 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.32H (*exo*)), 5.87 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.68H (*endo*)), 3.90 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.68H (*endo*)), 3.87 (dd, *J* = 3.0 Hz, 5.0 Hz, 0.32H(*exo*)) 3.44 (d, *J* = 4.0 Hz, 0.68H (*endo*)), 3.42 (dd, *J* = 1.0 Hz, 5.0 Hz, 0.32H (*exo*)), 3.33 (s, 0.68H (*endo*)), 3.16 (s, 0.32H (*exo*)), 3.13 (s, 0.32H (*exo*)), 3.09 (d, *J* = 1.5 Hz, 0.68H (*endo*)), 2.32 (s, 2H, CH₃ (*endo*)), 2.30 (s, 1H, CH₃ (*exo*)), 2.03 (d, *J* = 10.0 Hz, 0.68H (*endo*)), 1.90 (d, *J* = 10.0 Hz, 0.32H (*exo*)), 1.64 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.68H (*endo*)), 1.54 (dq, *J* = 1.5 Hz, 8.5 Hz, 0.32H (*exo*)) ppm; **¹³C NMR (125 MHz, CDCl₃):** δ 201.4, 200.2, 141.7, 140.6, 139.3, 137.4, 137.1, 137.0, 136.6, 135.8, 135.6, 133.0, 133.0, 132.9, 129.3, 128.9, 128.6, 128.6, 128.5, 128.0, 127.5, 56.3, 54.7, 49.0, 48.9, 48.8, 48.7, 48.1, 47.9, 47.5, 45.6, 46.0, 30.5, 29.8, 21.1, 21.1 ppm.

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

Results: 95% yield, yellow oily liquid, dr = 67:33 (*endo:exo*); **IR (ATR):** ν_{\max} 3052, 2983, 2854, 1678, 1593, 1509, 1448, 1422, 1346, 1271, 1226, 1015, 822, 747, 672 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 8.13 (dt, $J = 2.5$ Hz, 4.0 Hz, 1.32H), 8.09 (dt, $J = 0.5$ Hz, 8.0 Hz, 0.64H), 7.96- 7.90 (m, 2H), 7.59-7.53 (m, 1H), 7.48- 7.40 (m, 3.38H), 7.30 (dt, $J = 2.0$ Hz, 3.5 Hz, 0.68H), 6.54 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.33H), 6.47 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.67H) 6.13 (dd, $J = 3.0$ Hz, 6.0 Hz, 0.33H), 5.87(dd, $J = 3.0$ Hz, 6.0 Hz, 0.67H), 4.10 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.33H), 3.85 (dd, $J = 1.0$ Hz, 5.0 Hz, 0.67H), 3.62 (d, $J = 4$ Hz, 0.67H), 3.45(dd, $J = 1.5$ Hz, 9.0 Hz, 0.33H), 3.41(s, 0.68H), 3.22(s, 0.33H), 3.19- 3.16(m, 1H), 1.97 (d, $J = 8.5$ Hz, 0.68H), 1.89 (d, $J = 8.5$ Hz, 0.33H), 1.71(dq, $J = 1.5$ Hz, 9.0 Hz, 0.67H), 1.57 (dq, $J = 1.5$ Hz, 9.0 Hz, 0.33H) ppm; **$^{13}\text{C NMR}$ (125 MHz, CDCl_3):** δ 200.0, 199.2, 152.9, 151.7, 146.6, 146.4, 139.1, 137.3, 136.9, 136.6, 136.4, 133.4, 133.2, 133.2, 128.8, 128.8, 128.6, 128.4, 128.3, 123.9, 123.5, 56.7, 54.7, 49.3, 49.0, 48.9,, 48.2, 48.1, 47.9, 47.5, 45.8 ppm.

4-(3-Benzoylbicyclo[2.2.1]hept-5-en-2-yl)benzotrile (3k)

Results: 94% yield, yellow oily liquid, dr = 45:55 (*endo:exo*); **IR (ATR):** ν_{\max} 2971, 2980, 2227, 1675, 1607, 1597, 1505, 1448, 1330, 1211, 1017, 833, 761, 695, 563 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 7.95- 7.89 (m, 2H), 7.59-7.51 (m, 3H), 7.50-7.42 (m, 2H) 7.40- 7.35 (m, 1H), 7.26-7.24 (m, 1H), 6.52 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.54H (*exo*)), 6.46 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.46H (*endo*)), 6.12 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.54H(*exo*)), 5.86 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.46H (*endo*)), 4.04 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.54H), 3.83 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.46H) 3.56 (d, $J = 4.0$ Hz, 0.46H), 3.42 (dd, $J = 1.5$ Hz, 5.5 Hz, 0.54H(*exo*)), 3.39 (s, 0.46H (*endo*)), 3.20 (s, 0.56H), 3.17 (s, 0.55H), 3.14 (s, 0.45H), 1.95 (d, $J = 8.5$ Hz, 0.46H (*endo*)), 1.88 (d, $J = 8.5$ Hz, 0.54H (*exo*)), 1.70(dd, $J = 2.0$ Hz, 9.0 Hz, 0.46H), 1.56 (dd, $J = 2.0$ Hz, 9.0 Hz, 0.54H), ppm; **$^{13}\text{C NMR}$ (125 MHz, CDCl_3):** δ 200.2, 199.3, 150.6, 149.4, 139.1, 137.2, 137.0, 136.7, 136.4, 133.3, 133.2, 132.5, 132.1, 128.9, 128.8, 128.6, 128.5, 128.4, 127.3, 119.1, 110.2, 110.0,, 56.6, 54.6, 49.3, 48.9, 48.8, 48.2, 48.0, 47.9, 47.6, 45.9 ppm.

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

Results: 88% yield, yellow oily liquid, dr = 63:37 (*endo:exo*); **IR (ATR):** ν_{\max} 3061, 2963, 2924, 1742, 1672, 1455, 1362, 1223, 1013, 805, 713, 674 cm^{-1} ; **$^1\text{H-NMR}$ (500 MHz, CDCl_3):** δ 7.97-7.93 (m, 1.30H (*endo*)), 7.90-7.86 (m, 0.74H (*exo*)), 7.18-7.02 (m, 6H), 6.48-6.43 (m, 1H), 6.17 (dd, $J = 2.5$ Hz, 5.5 Hz, 0.37H (*exo*)), 5.88 (dd, $J = 3.0$ Hz, 6.0 Hz, 0.63H (*endo*)), 3.84 (dd, $J = 3.5$ Hz, 5.0 Hz, 0.63H (*endo*)), 3.78 (dd, $J = 3.5$ Hz, 5.5 Hz, 0.37H (*exo*)), 3.38 (d, $J = 4.0$ Hz, 0.63H (*endo*)), 3.34 (dd, $J = 1.0$ Hz, 5.0 Hz, 0.37H (*exo*)), 3.31 (s, 0.63H (*endo*)),

3.15-3.10 (m, 0.75H (*exo*)), 3.09- 3.06 (m, 0.63H (*endo*)), 2.32 (s, 2H CH₃ (*endo*)), 2.30 (s, 1H CH₃ (*exo*)), 2.02 (d, *J* = 8.5 Hz, 0.63H (*endo*)), 1.88 (d, *J* = 8.5 Hz, 0.37H (*exo*)), 1.63 (dq, *J* = 1.5 Hz, 3.5 Hz, 0.63H (*endo*)), 1.54 (m, 0.37H (*exo*)) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 200.0, 198.7, 165.7 (d, *J* = 253.7 Hz), 165.6 (d, *J* = 253.7 Hz), 141.6, 139.3, 137.0, 135.7, 133.7 (d, *J* = 2.5 Hz), 133.5 (d, *J* = 2.5 Hz), 133.0, 131.2 (d, *J* = 8.7 Hz), 131.1 (d, *J* = 8.7 Hz), 129.4, 129.0, 128.0, 127.5, 115.8 (d, *J* = 21.2 Hz), 115.6 (d, *J* = 21.2 Hz), 56.3, 54.7, 49.1, 48.9, 48.8, 48.6, 48.2, 48.1, 47.8, 45.9, 21.1 ppm; ¹⁹F NMR (470 MHz, CDCl₃): δ -105.79 (*exo*), -105.89 (*endo*) ppm.

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

Results: 87% yield, yellow oily liquid, dr = 73:27 (*endo:exo*); IR (ATR): ν_{\max} 3052, 2962, 2921, 1731, 1672, 1581, 1362, 1212, 1013, 804, 712, 674 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 7.85 (dt, *J* = 2.5 Hz, 9.0 Hz, 1.54H (*endo*)), 7.78 (dt, *J* = 2.5 Hz, 9.5 Hz, 0.57H (*exo*)), 7.39 (dt, *J* = 2.5 Hz, 9.5 Hz, 1.55H (*endo*)), 7.34 (dt, *J* = 2.5 Hz, 9.5 Hz 0.62H (*exo*)), 7.19- 7.02 (m, 4H), 6.45 (m, 1H), 6.16 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.27H (*exo*)) 5.86 (dd, *J* = 3.0 Hz, 6.0 Hz, 0.73H (*endo*)), 3.82 (dd, *J* = 3.5 Hz, 5.0 Hz, 0.73H (*endo*)), 3.76 (dd, *J* = 3.0 Hz, 5.5 Hz, 0.27H (*exo*)), 3.37 (d, *J* = 4.0 Hz, 0.72H (*endo*)), 3.33 (dd, *J* = 1.5 Hz, 5.5 Hz, 0.27H (*exo*)), 3.29 (s, 0.73H (*endo*)), 3.14-3.10 (m, 0.59H), 3.08-3.06 (m, 0.73H (*endo*)), 2.32 (s, 2H, CH₃ (*endo*)), 2.30 (s, 1H, CH₃ (*exo*)), 2.01 (d, *J* = 8.5 Hz, 0.73H (*endo*)), 1.87 (d, *J* = 9.0 Hz, 0.27H (*exo*)), 1.63 (dd, *J* = 3.5 Hz, 10.5 Hz, 0.72H (*endo*)), 1.42 (s, 0.27H (*exo*)) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 200.5, 199.1, 141.3, 139.4, 139.3, 137.0, 136.5, 135.8, 135.7, 133.0, 130.0, 129.9, 129.4, 129.0, 129.0, 128.9, 128.0, 127.5, 56.4, 54.7, 49.2, 49.0, 48.7, 48.6, 48.2, 48.1, 45.9, 21.1 ppm.

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

Results: 86% yield, yellow oily liquid, dr = 66:34 (*endo:exo*); IR (ATR): ν_{\max} 2951, 2923, 2851, 1741, 1585, 1457, 1371, 1212, 1025, 803, 721, 682 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 7.77 (dt, *J* = 2.0 Hz, 9.0 Hz, 1.37H (*endo*)), 7.70 (dt, *J* = 2.0 Hz, 9.0 Hz, 0.69H (*exo*)), 7.56 (dt, *J* = 2.5 Hz, 9.5 Hz, 1.34H (*endo*)), 7.50 (dt, *J* = 2.0 Hz, 9.0 Hz, 0.68H (*exo*)), 7.17-7.01 (m, 4H), 6.47-6.42 (m, 1H), 6.16 (dd, *J* = 2.8 Hz, 5.6 Hz, 0.34H (*exo*)), 5.86 (dd, *J* = 2.8 Hz, 5.6 Hz, 0.66H (*endo*)), 3.81 (dd, *J* = 3.4 Hz, 5.0 Hz, 0.66H), 3.75 (dd, *J* = 3.5 Hz, 5.5 Hz, 0.34H), 3.37 (d, *J* = 4.0 Hz, 0.66H (*endo*)), 3.31 (dd, *J* = 1.5 Hz, 5.4 Hz, 0.34H (*exo*)), 3.29 (s, 0.66H (*endo*)), 3.11 (d, *J* = 8.0 Hz, 0.90H (*exo*)), 3.06 (s, 0.55H (*endo*)), 2.32 (s, 2H, CH₃ (*endo*)), 2.30 (s, 1H, CH₃ (*exo*)), 2.01 (d, *J* = 8.5 Hz, 0.66H (*endo*)), 1.86 (d, *J* = 8.6 Hz, 0.34H (*exo*)),

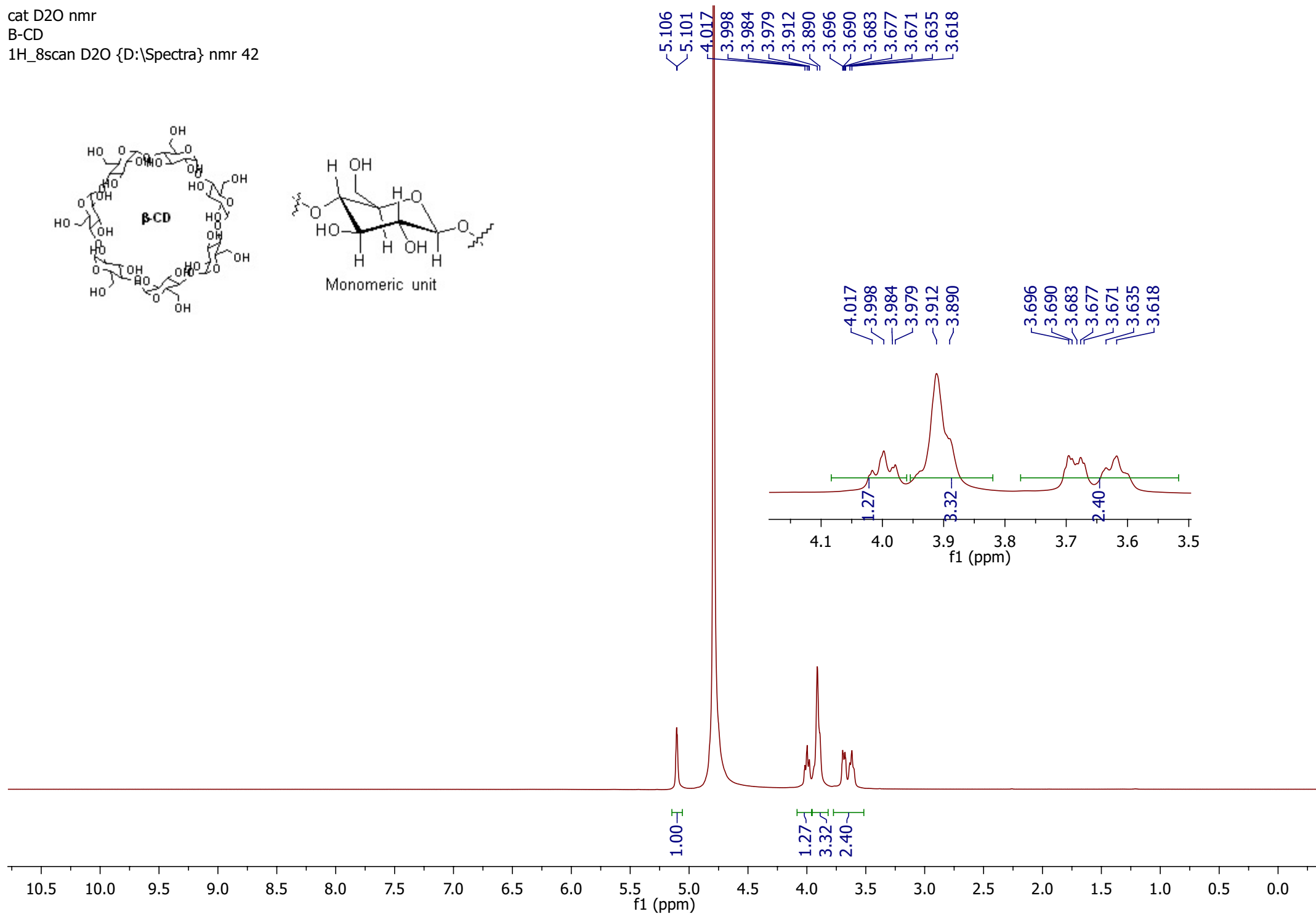
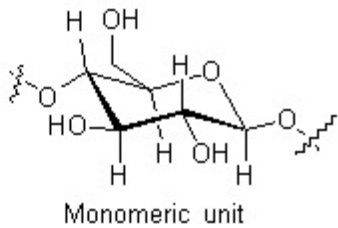
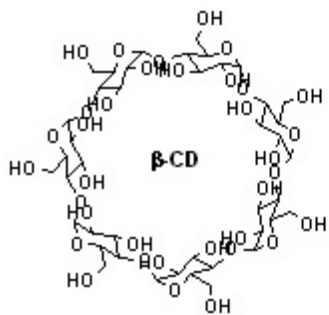
1.63(dd, $J = 3.4$ Hz, 10.5 Hz, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 200.5, 198.7, 141.6, 138.6, 137.1, 136.0, 135.8, 133.0, 132.0, 131.9, 131.9, 130.2, 130.1, 129.4, 129.0, 128.0, 127.5, 56.3, 49.2, 49.0, 48.7, 48.6, 48.6, 48.2, 48.1, 47.9, 45.9, 29.8, 21.1 ppm.

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

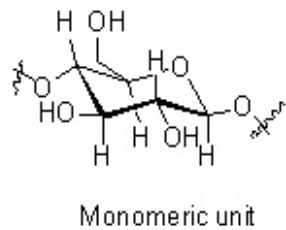
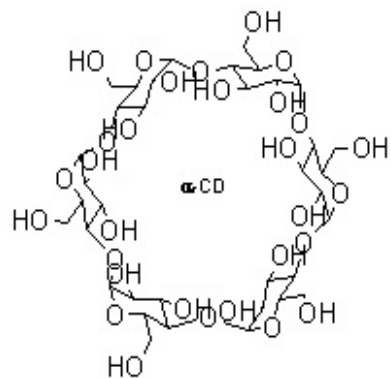
Results: 84% yield, yellow oily liquid, dr = 59:41 (*endo:exo*); **IR (ATR):** ν_{max} 3012, 2963, 2934, 1732, 1669, 1592, 1457, 1367, 1225, 1027, 837, 719, 689 cm^{-1} ; **^1H -NMR (500 MHz, CDCl_3):** δ 7.92 (dt, $J = 2.0, 10.0$ Hz, 1.23H (*endo*)), 7.86 (dt, $J = 2.5$ Hz, 9.5 Hz, 0.84H (*exo*)), 7.19-7.08 (m, 2.5H (*endo*)), 7.04 (s, 1.6H (*exo*)), 6.90 (dd, $J = 2.0$ Hz, 7.0 Hz, 1.25H (*endo*)), 6.86 (dd, $J = 2.5$ Hz, 7.0 Hz, 0.80H (*exo*)), 6.47-6.43 (m, 1H), 6.16 (dd, $J = 3.0$ Hz, 5.5 Hz, 0.41H (*exo*)), 5.88 (dd, $J = 2.5$ Hz, 5.6 Hz, 0.59H (*endo*)), 3.86 (s, 1.8H $-\text{OCH}_3$ (*endo*)), 3.85-3.82 (m, 2.2H {1.2H, $-\text{OCH}_3$, (*exo*) + 1H (*endo* + *exo*)}), 3.42 (d, $J = 4.3$ Hz, 0.59H (*endo*)), 3.35 (dd, $J = 1.4$ Hz, 5.0 Hz, 0.41H (*exo*)), 3.31 (s, 0.59H (*endo*)), 3.13 (s, 0.41H (*exo*)), 3.07 (d, $J = 1.5$ Hz, 0.59H), 2.31 (s, 1.8H, CH_3 (*endo*)), 2.29 (s, 1.2H, CH_3 (*exo*)), 2.01 (d, $J = 8.4$ Hz, 0.59H (*endo*)), 1.90 (d, $J = 8.6$ Hz, 0.41H (*exo*)), 1.62 (dd, $J = 1.6$ Hz, 8.5 Hz, 0.59H), 1.52 (dd, $J = 1.6$ Hz, 8.5 Hz, 0.41H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 200.0, 198.6, 163.4, 163.4, 141.9, 140.7, 139.2, 136.9, 136.7, 135.8, 135.6, 133.1, 130.9, 130.8, 130.3, 130.2, 129.3, 128.9, 128.0, 127.5, 113.8, 113.8, 55.9, 55.6, 55.6, 54.3, 49.1, 48.9, 48.8, 48.8, 48.1, 48.1, 47.6, 45.7, 29.8, 21.1, 21.1 ppm.

**Copies of ^1H -NMR spectra of CDs,
Amino acids and Inclusion Complexes
&
Copies of ^1H and ^{13}C NMR Spectra of
DA adducts**

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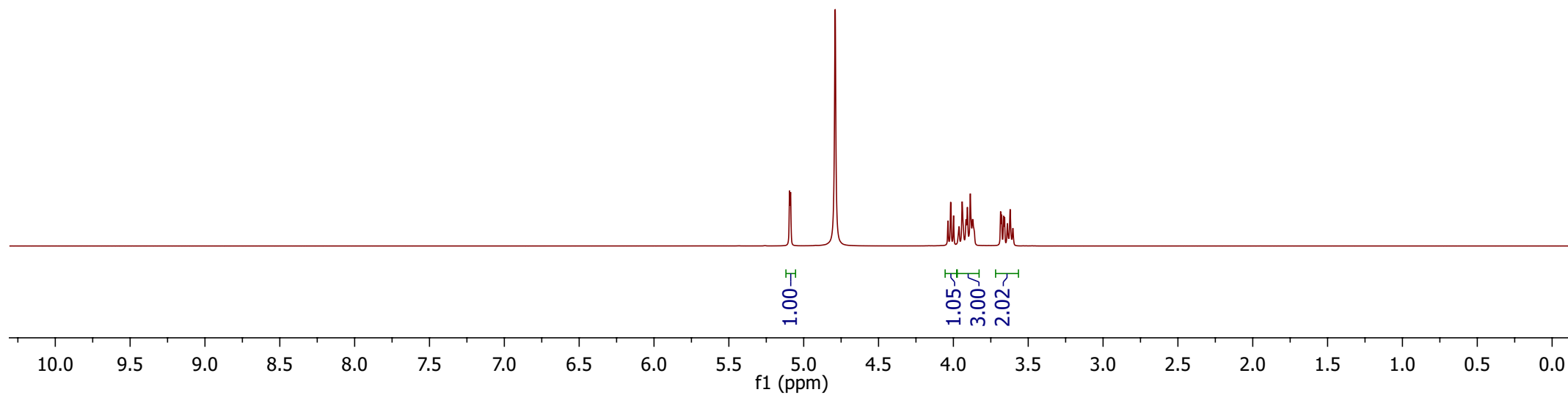
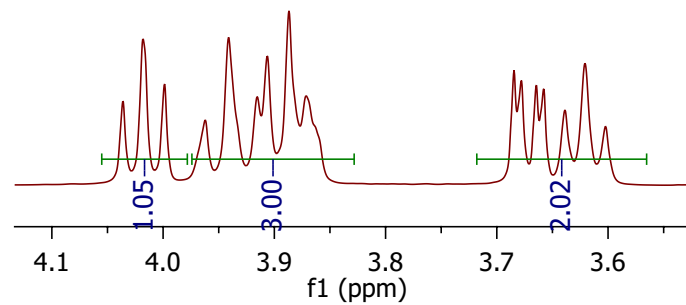


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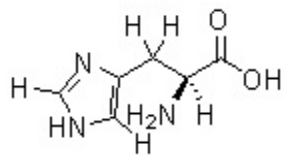


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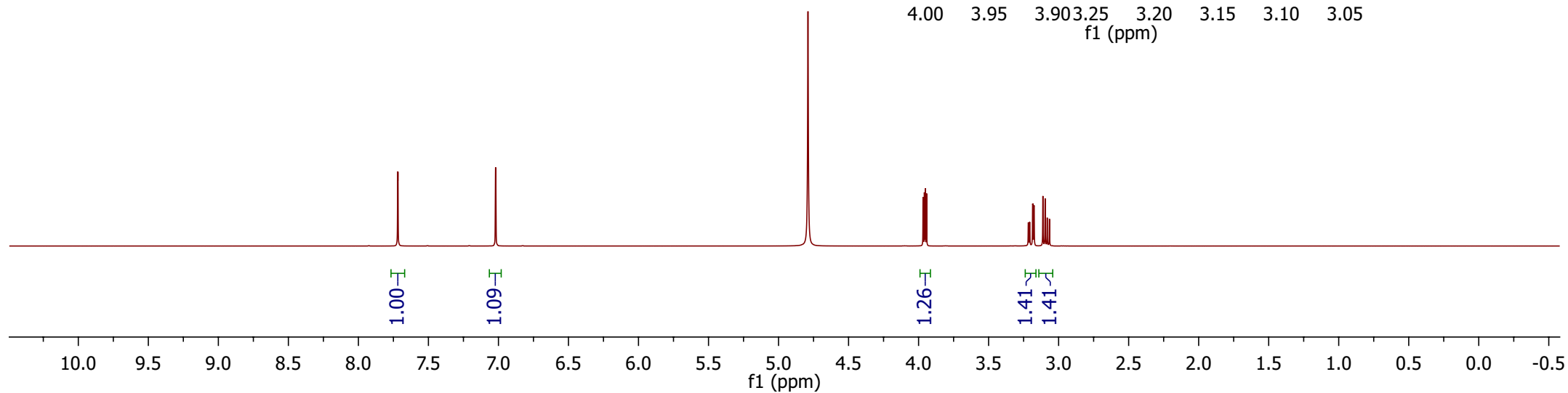
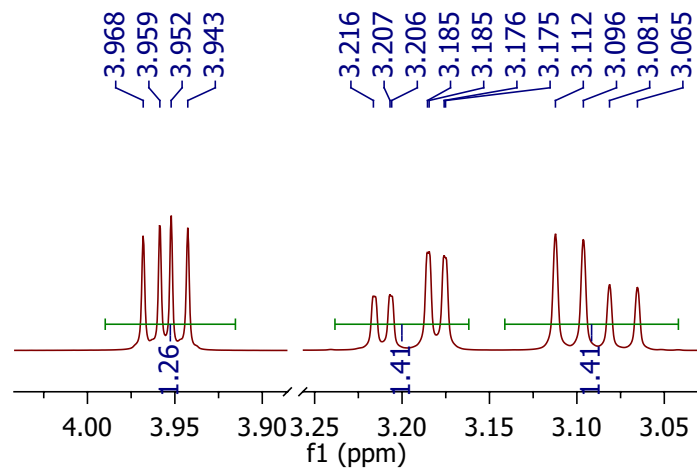
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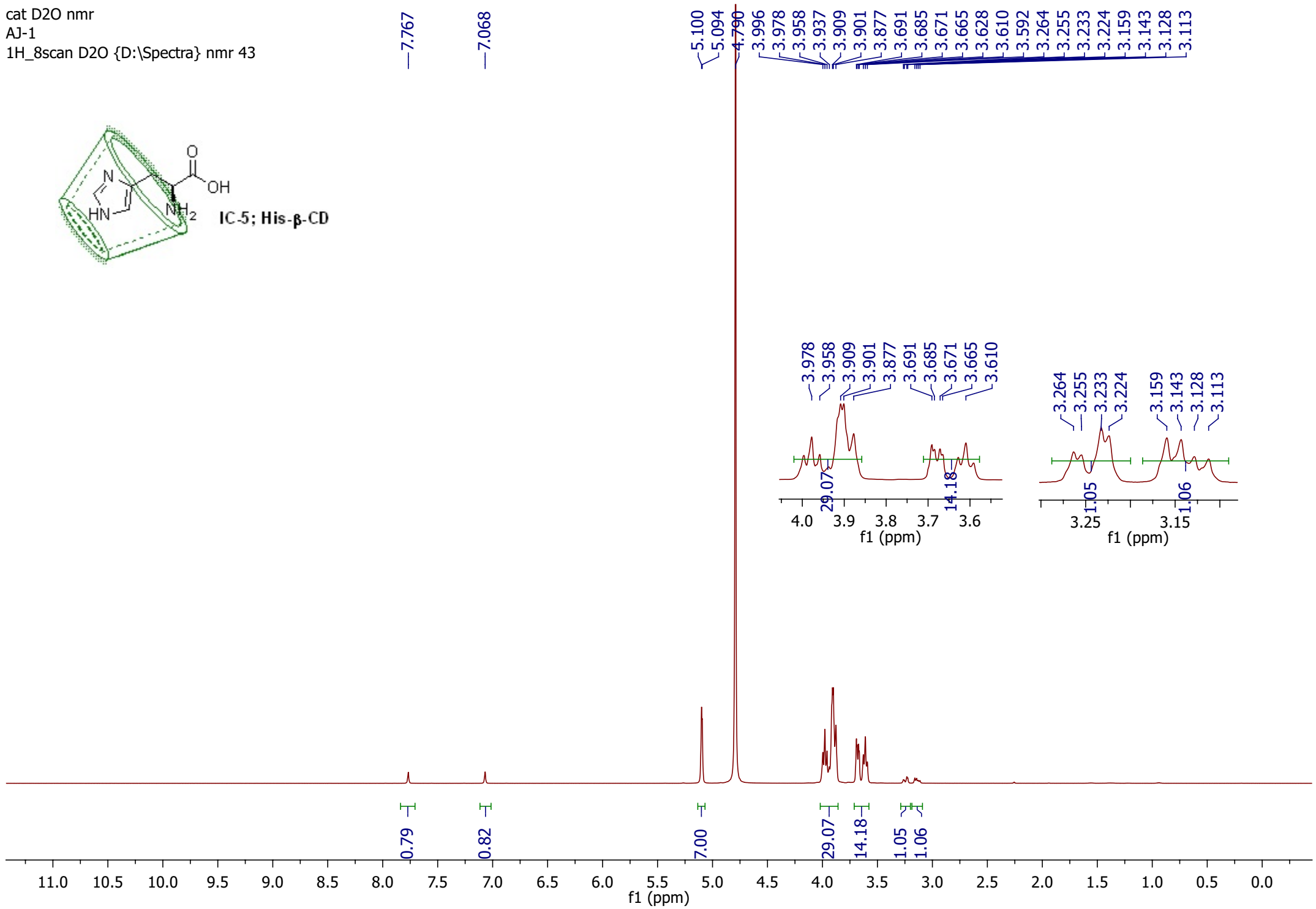
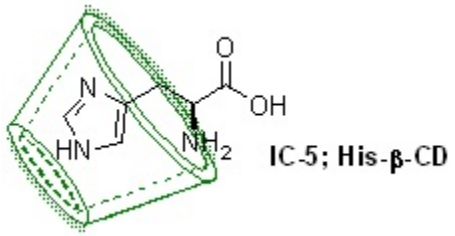
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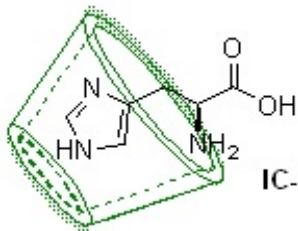
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IC-6; His- α -CD

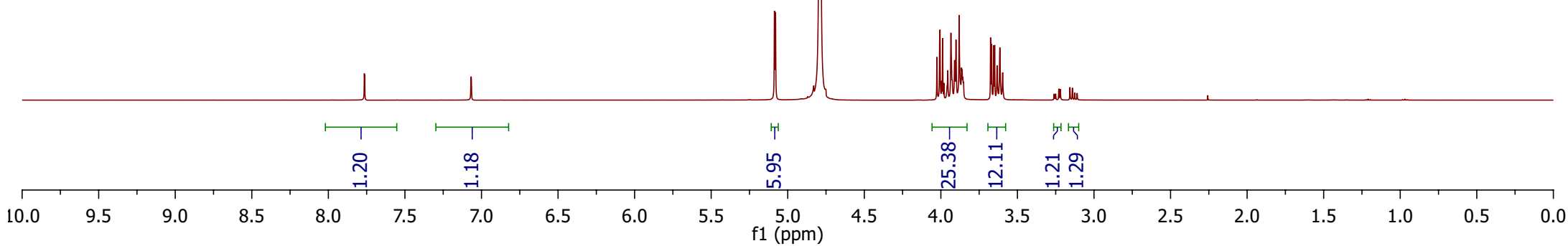
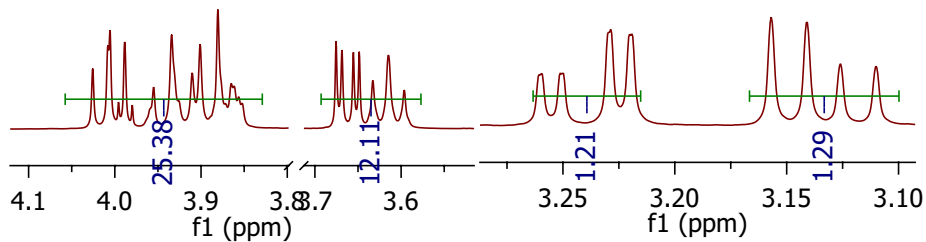
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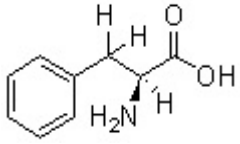
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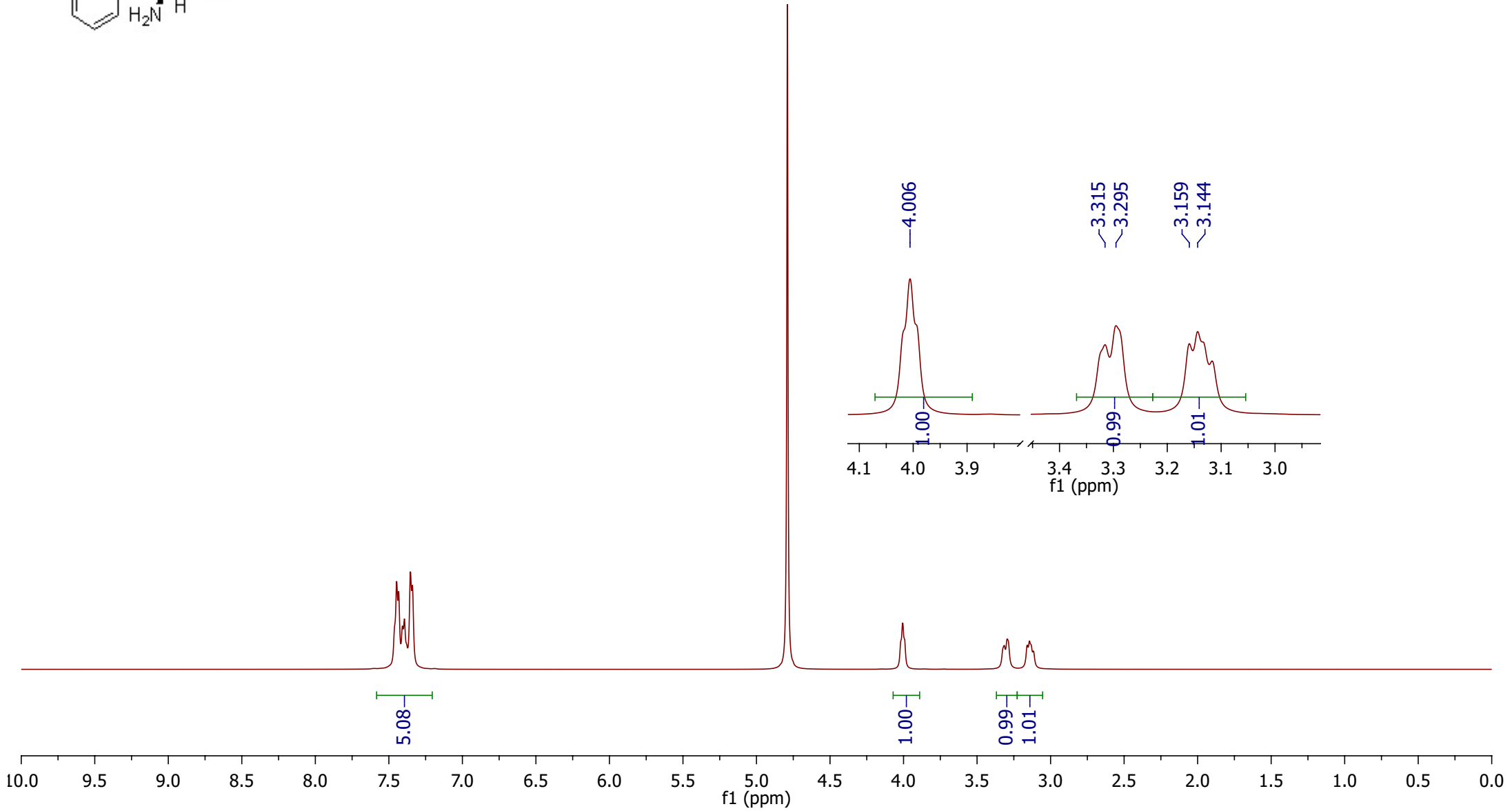


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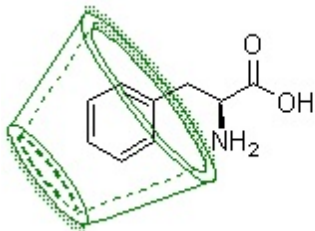
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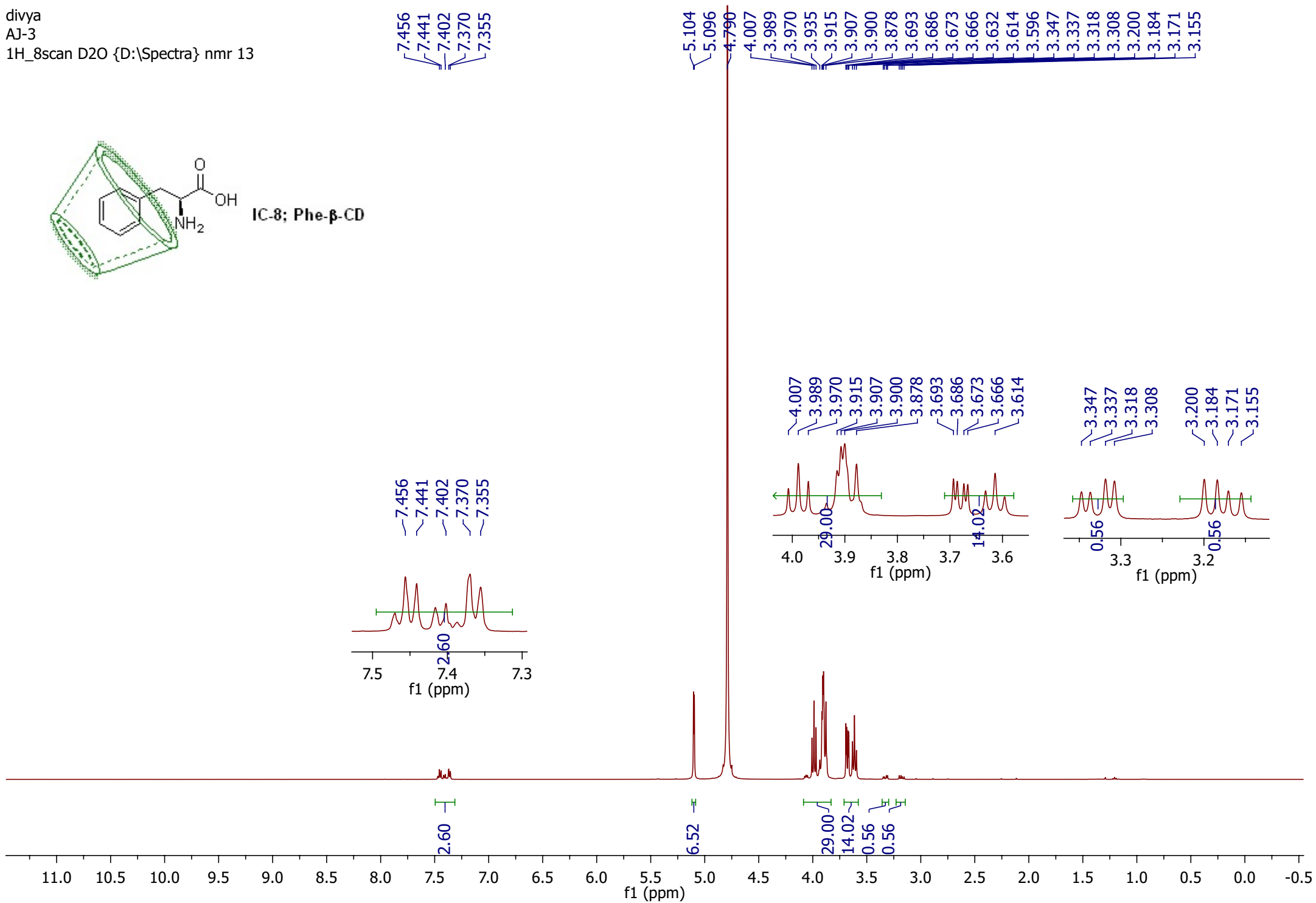
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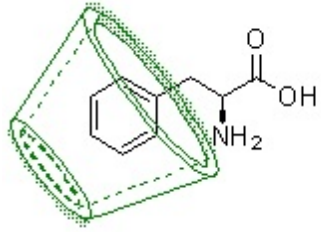
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IC-8; Phe- β -CD



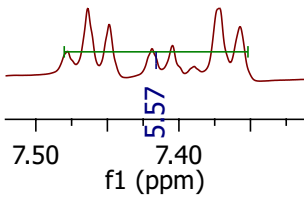
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IC-9; Phe- α -CD

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7.372
7.357

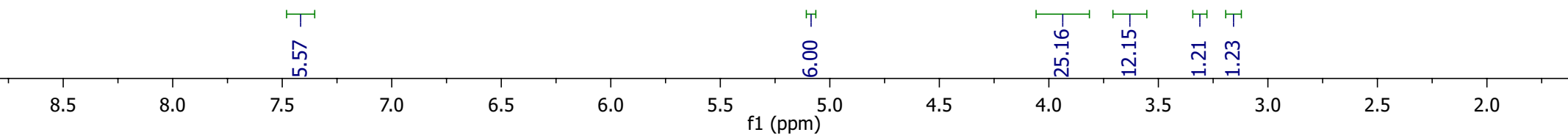
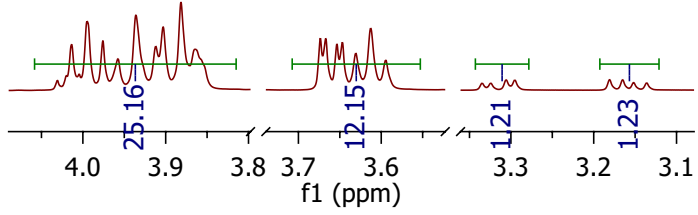
7.478
7.464
7.449
7.419
7.405
7.372
7.357



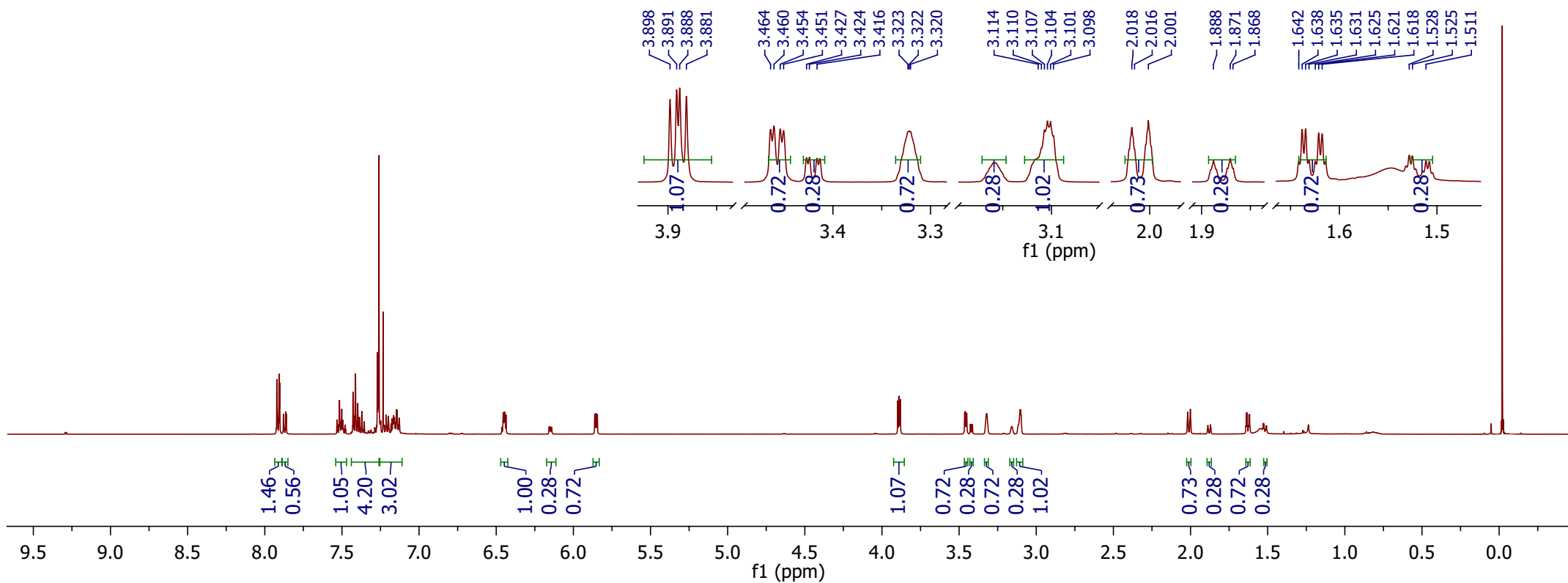
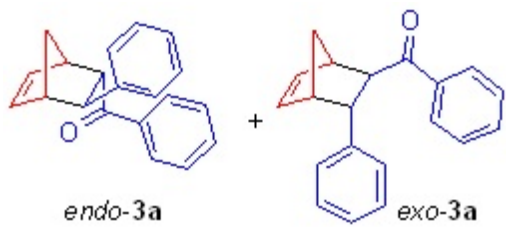
5.085
5.079

4.014
3.995
3.976
3.936
3.912
3.903
3.882
3.865
3.674
3.667
3.654
3.647
3.613
3.324
3.306
3.295
3.181
3.165
3.152
3.136

4.014
3.995
3.976
3.936
3.912
3.903
3.882
3.674
3.667
3.654
3.647
3.613
3.324
3.306
3.295
3.181
3.165
3.152
3.136



Mar 05 2024
D:\198-91103
1H Scan C:\3-D\Spectra\hmr_23

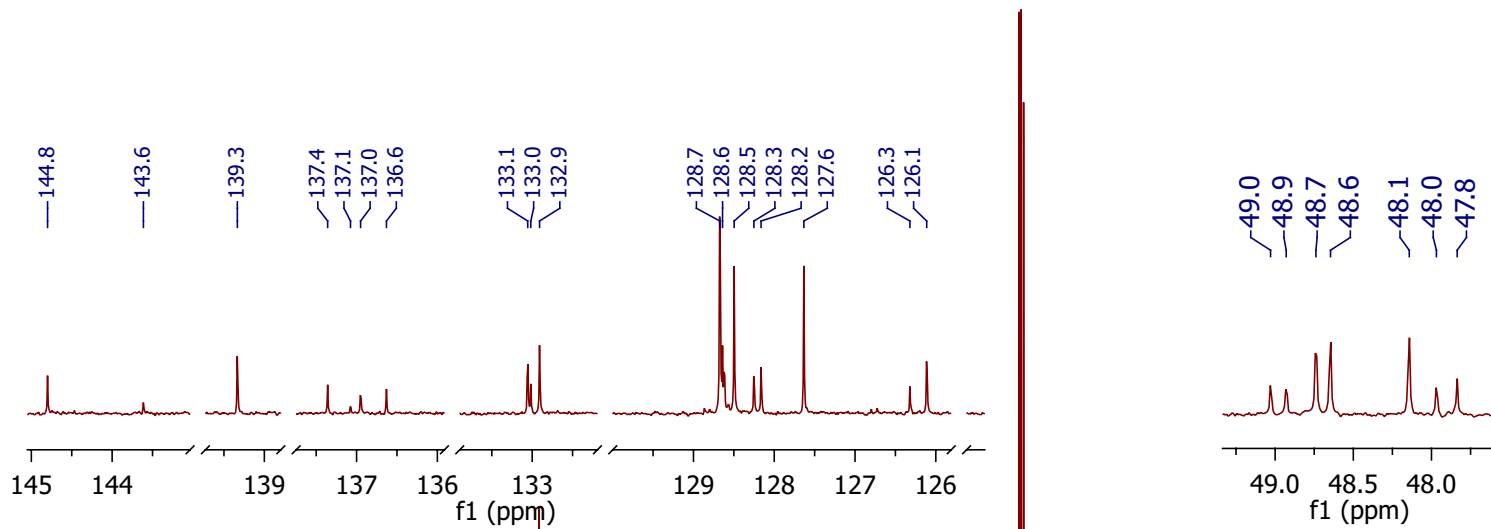
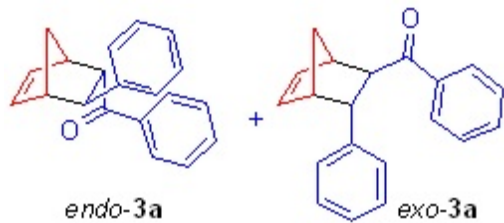


Mar05-2024
DT-198
C13CPD CDCl3 {D:\Spectra} nmr 23

144.8
143.6
139.3
137.4
137.1
137.0
136.6
133.1
133.0
132.9
128.7
128.6
128.5
128.3
128.2
127.6
126.3
126.1

77.4
77.2
76.9

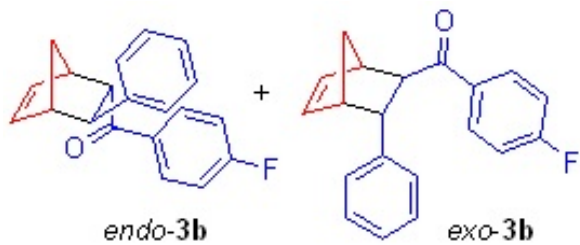
56.3
54.6
49.0
48.9
48.7
48.6
48.1
48.0
47.8
46.0



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

f1 (ppm)

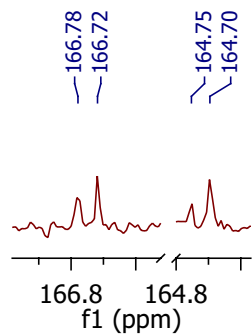
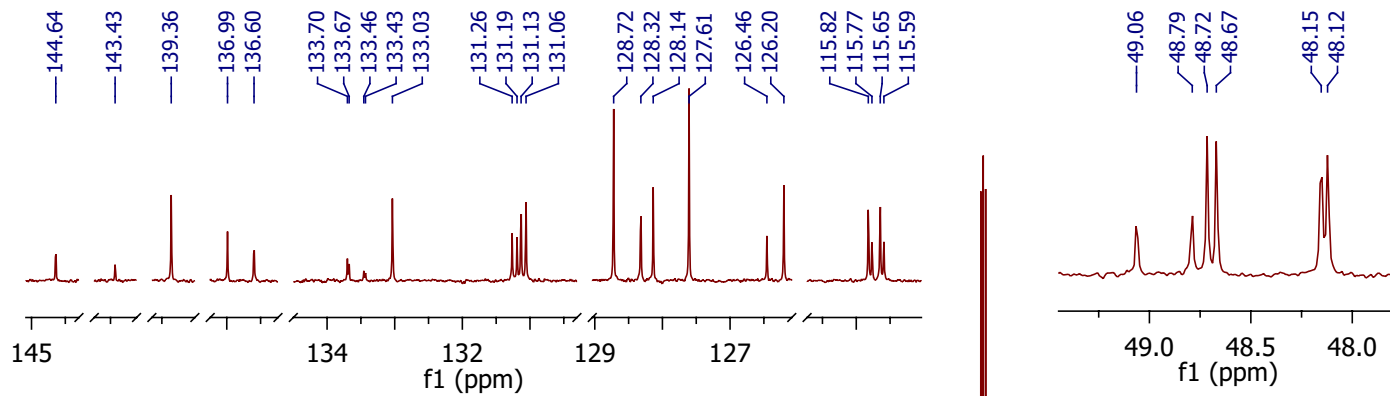
DA SUBSTRATES
 DT-273
 C13CPD CDCI3 (DMSO-d6) Spectra nmr 6



199.99
 198.99
 166.7
 164.7
 139.4
 137.0
 133.0
 131.3
 131.1
 131.1
 128.7
 128.3
 128.1
 127.6
 126.5
 118.8
 115.8
 115.6

77.4
 77.2
 76.9

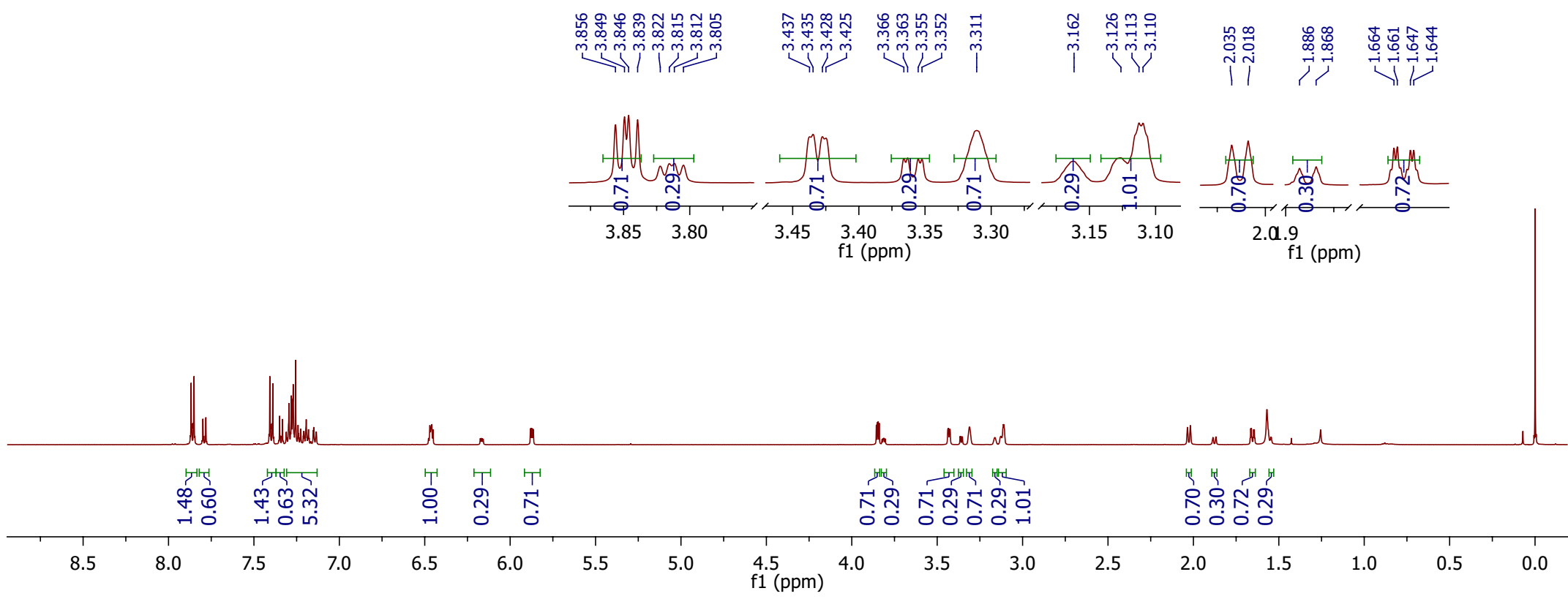
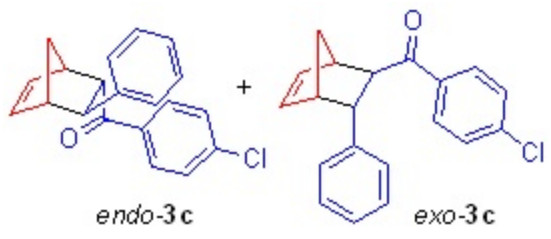
56.2
 54.6
 49.1
 48.8
 48.7
 48.7
 48.2
 48.1
 46.2



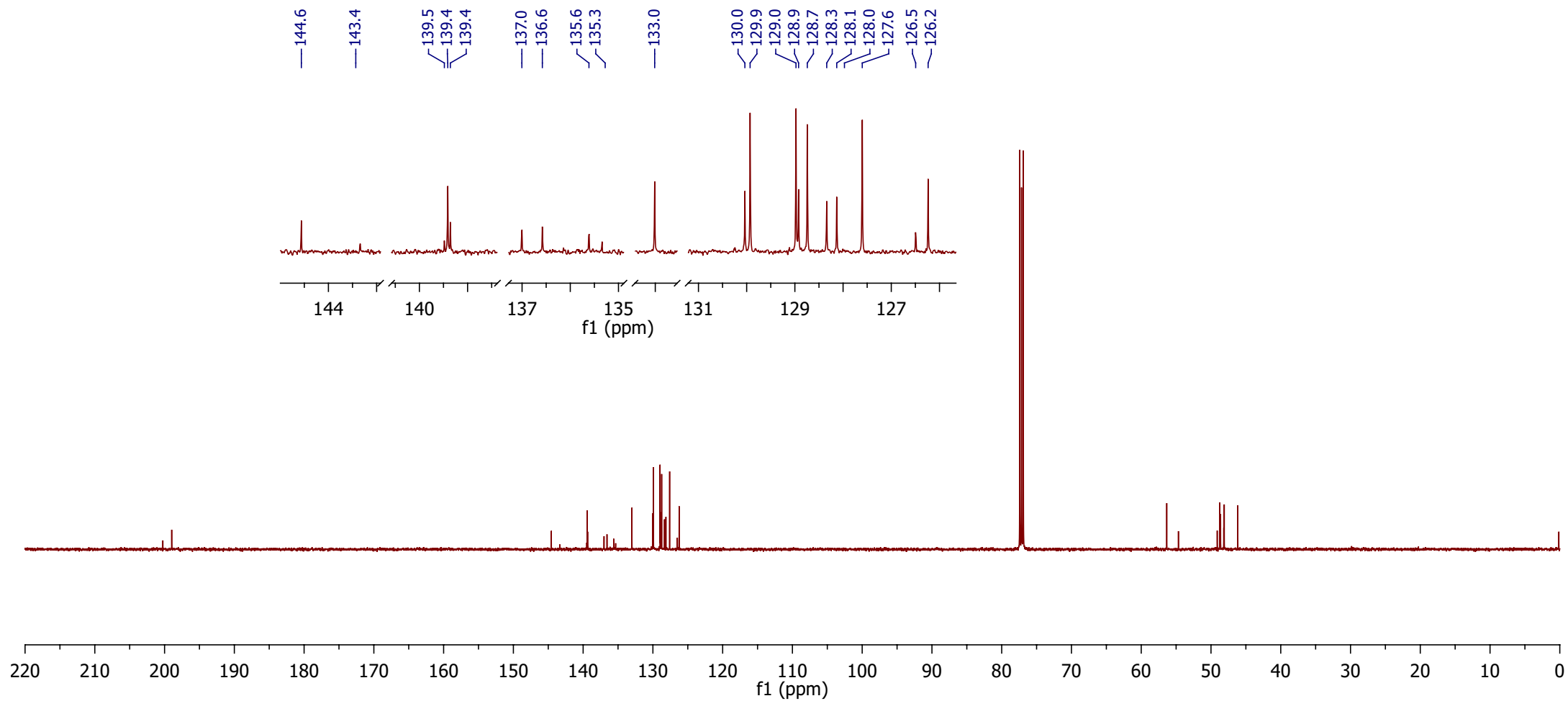
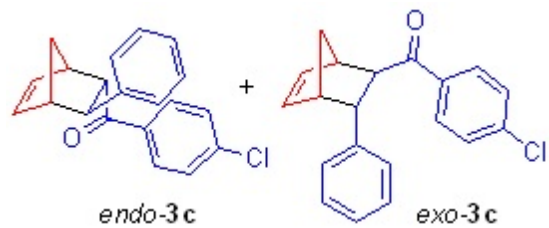
220
 210
 200
 190
 180
 170
 160
 150
 140
 130
 120
 110
 100
 90
 80
 70
 60
 50
 40
 30
 20
 10
 0
 f1 (ppm)

DA SUBSTRATES
D7-26
1H scan CDCl3 (D:\Spectra\hmr 7

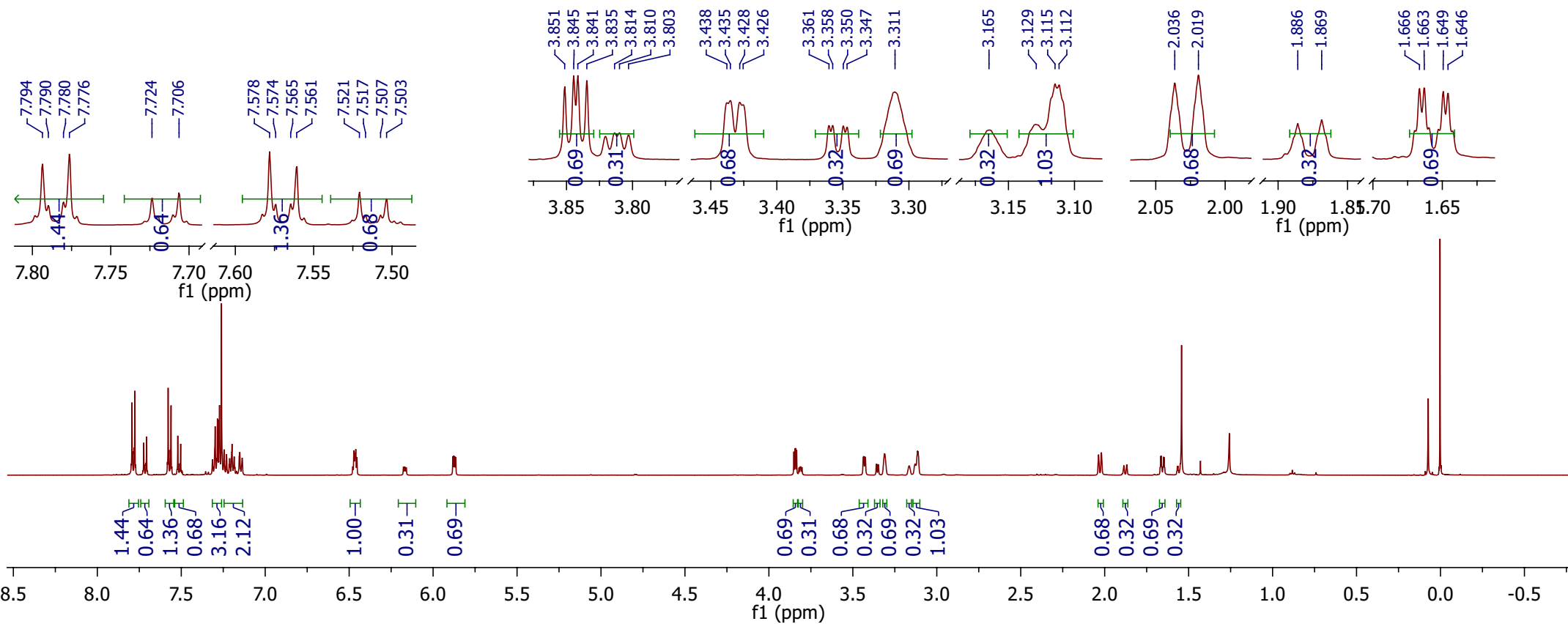
7.856
7.854
7.852
7.848
7.799
7.797
7.783
7.781
7.412
7.407
7.403
7.394
7.390
7.350
7.346
7.337
7.333
7.310
7.295
7.285
7.281
7.273
7.270
7.265
7.256
7.242
7.239
7.227
7.211
7.208
7.194
7.181
7.153
7.150
7.136
7.136
6.470
6.463
6.459
6.452
5.881
5.876
5.870
5.864
3.856
3.849
3.846
3.839
3.822
3.815
3.812
3.805
3.437
3.435
3.428
3.425
3.366
3.363
3.355
3.352
3.311
3.162
3.126
3.113
3.110
2.035
2.018
1.886
1.868
1.664
1.661
1.647
1.644



DA SUBSTRATES asymm Nmrs
DT-274
C13CPD CDCI3 (D) Spectra nmr 7



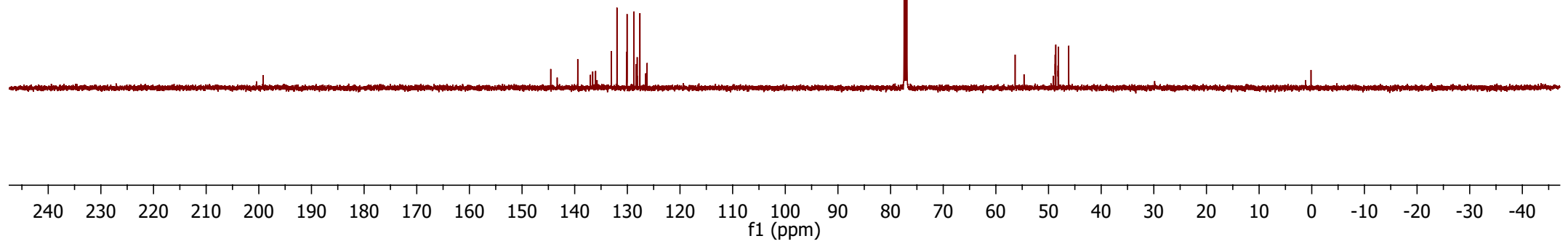
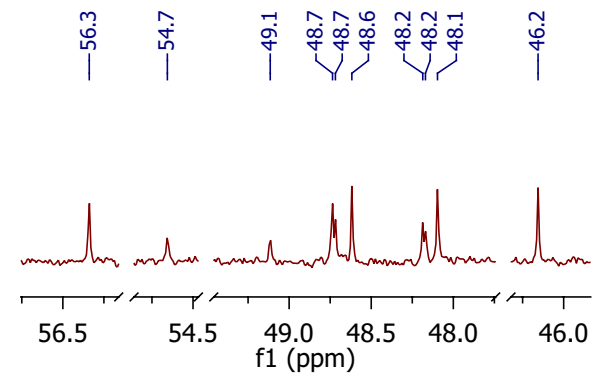
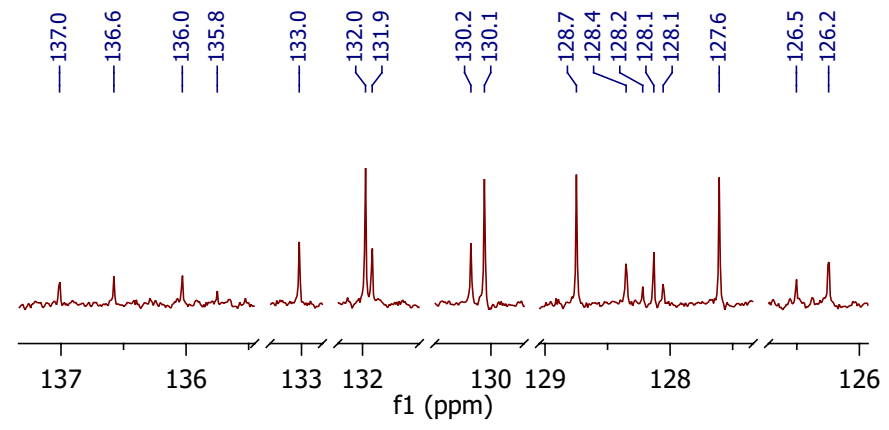
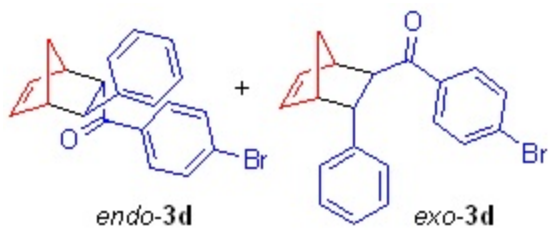
DA SUBSTRATES
DMSO-d₆
1H NMR Spectrum
Scan CDCl₃ (DMSO-d₆)
7.794
7.790
7.780
7.776
7.776
7.724
7.706
7.578
7.574
7.565
7.561
7.521
7.517
7.507
7.503
7.314
7.298
7.289
7.285
7.275
7.272
7.267
7.260
7.245
7.242
7.230
7.212
7.200
7.198
7.185
7.154
7.151
7.137
6.472
6.465
6.461
6.454
5.882
5.876
5.871
5.865
3.851
3.845
3.841
3.835
3.814
3.810
3.803
3.438
3.435
3.428
3.426
3.361
3.358
3.350
3.347
3.311
3.165
3.129
3.115
3.112
2.036
2.019
1.886
1.869
1.666
1.663
1.649
1.646



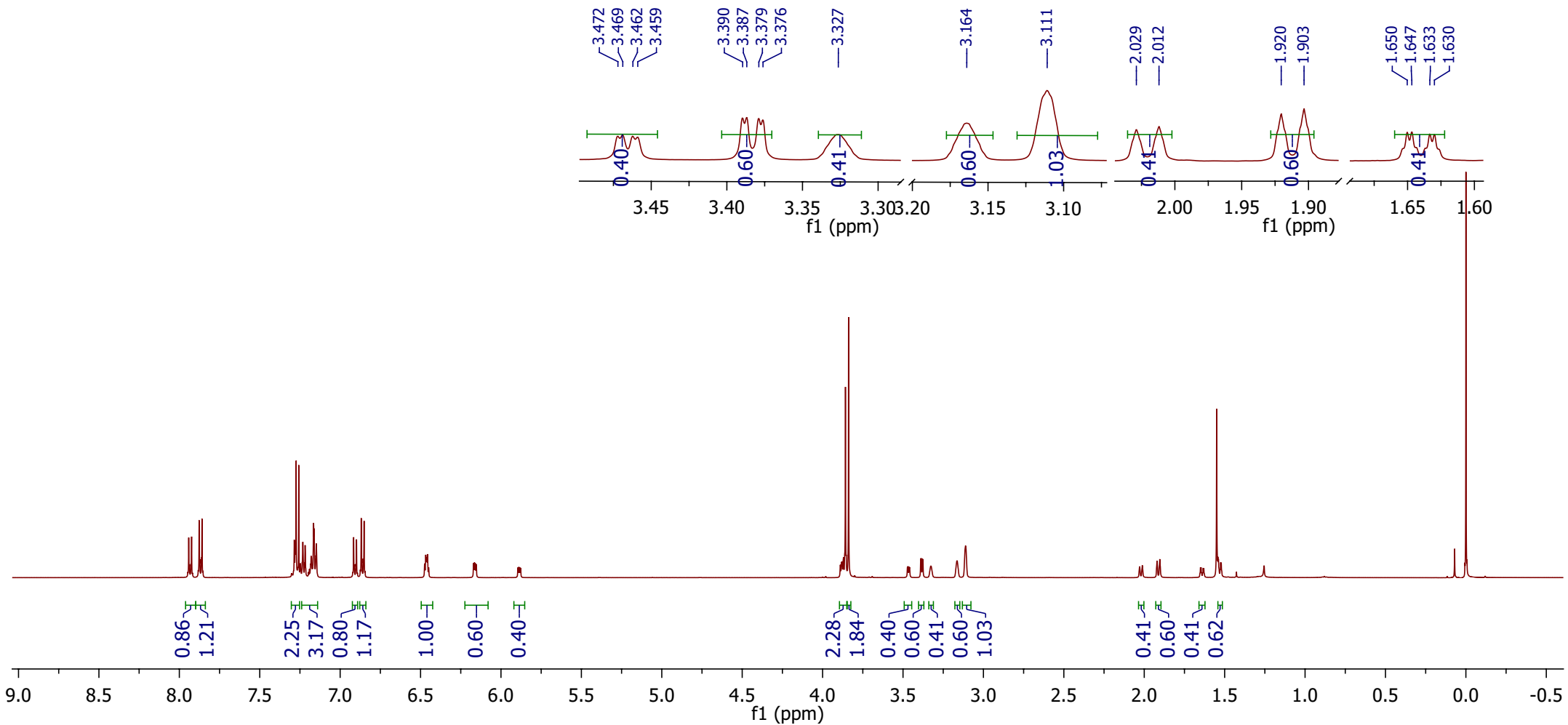
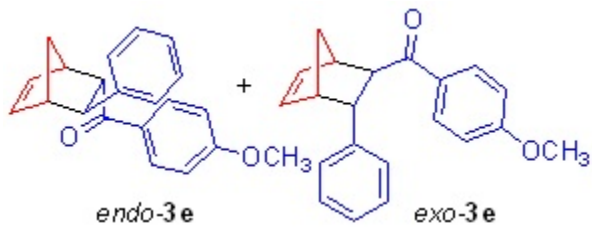
DA SUBSTRATES asymm Nmr
DT-277
C13CPD CDCl3 {D:\Spectra} nmr 3

144.5
143.3
139.4
137.0
136.6
136.0
135.8
133.0
132.0
131.9
130.2
130.1
128.7
128.4
128.2
128.2
128.1
128.1
127.6
126.5
126.2

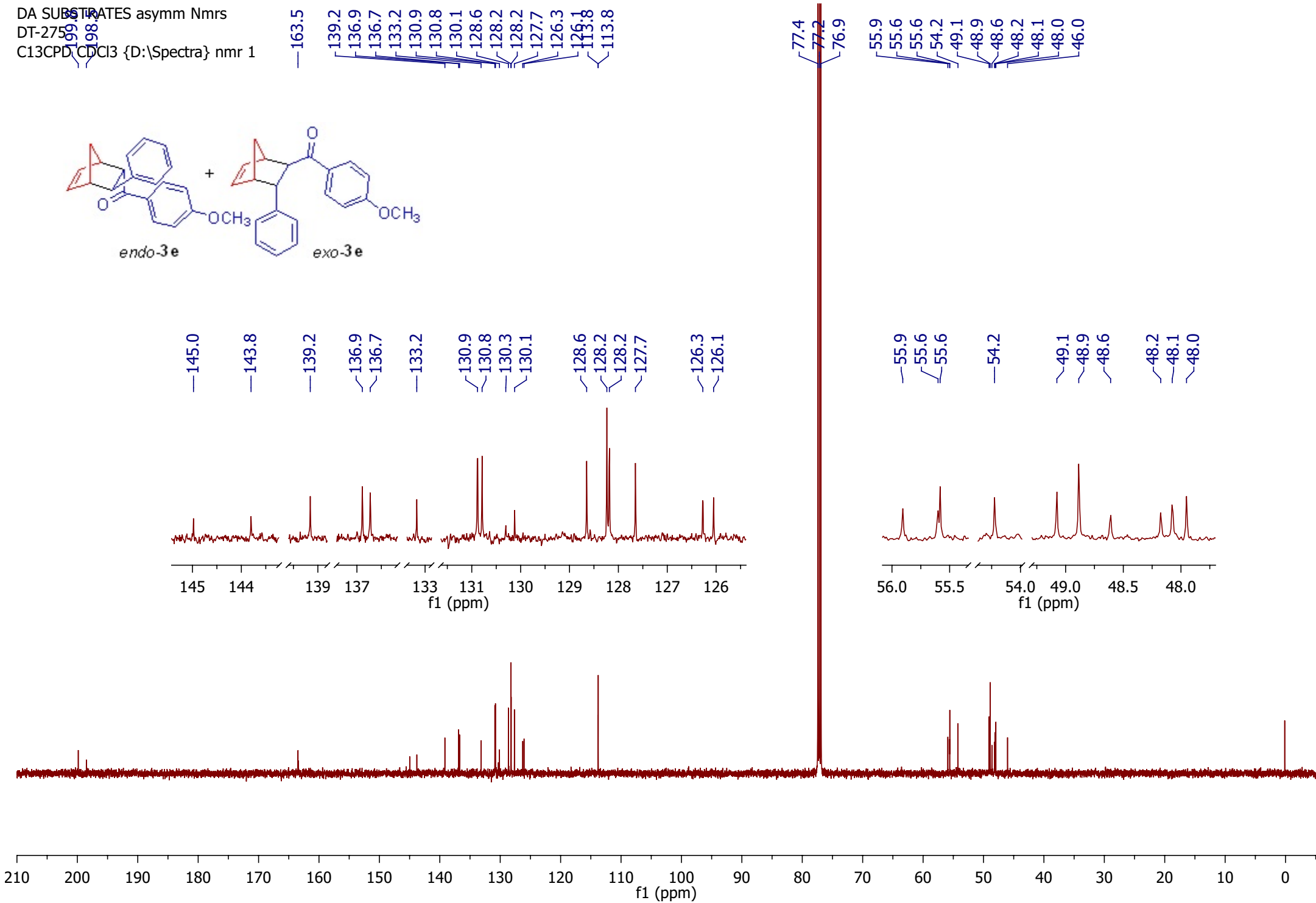
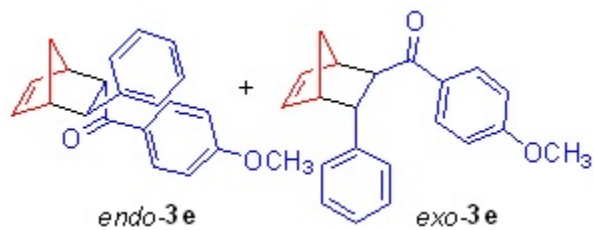
77.4
77.2
76.9
56.3
54.7
49.1
48.7
48.7
48.6
48.2
48.2
48.1
46.2



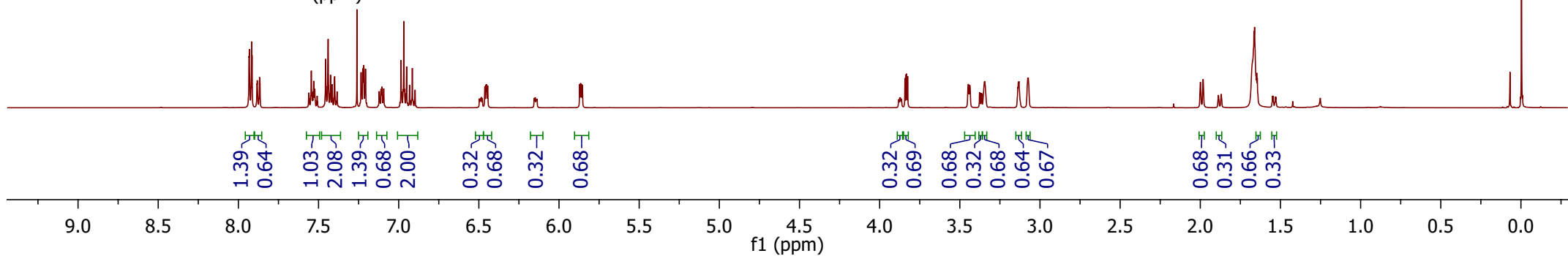
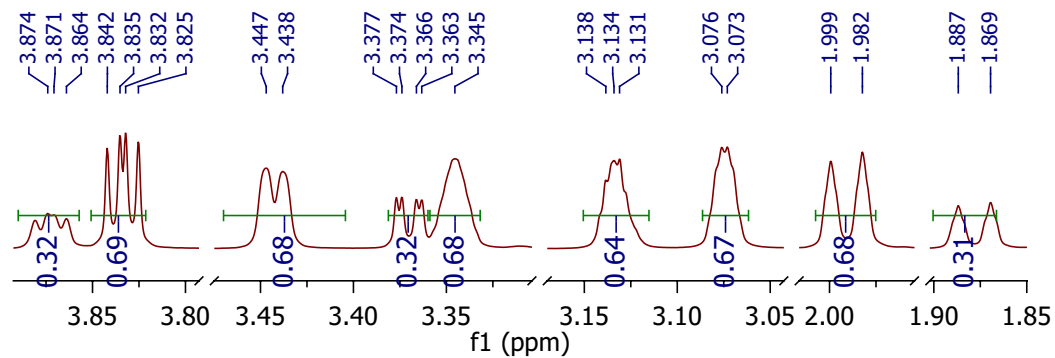
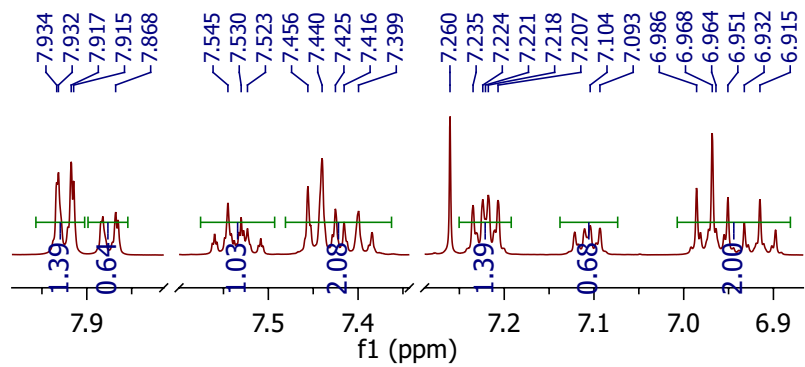
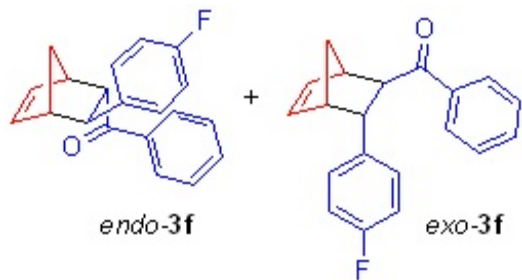
DA SUBSTRATES asymm Nmr
 1H scan CDCl3 (D:\Spectra\hmr1
 7.925
 7.922
 7.875
 7.871
 7.866
 7.855
 7.288
 7.285
 7.274
 7.257
 7.232
 7.218
 7.165
 7.162
 7.151
 7.148
 6.916
 6.912
 6.902
 6.898
 6.869
 6.865
 6.855
 6.851
 6.474
 6.468
 6.463
 6.457
 6.450
 6.170
 6.165
 6.159
 6.154
 5.889
 5.883
 3.890
 3.883
 3.879
 3.870
 3.858
 3.838
 3.472
 3.469
 3.462
 3.459
 3.462
 3.459
 3.390
 3.387
 3.379
 3.376
 3.327
 3.164
 3.111
 2.029
 2.012
 1.920
 1.903
 1.650
 1.647
 1.633
 1.630
 1.920
 1.903
 1.650
 1.647
 1.633
 1.630
 1.542
 1.539
 1.525
 1.521



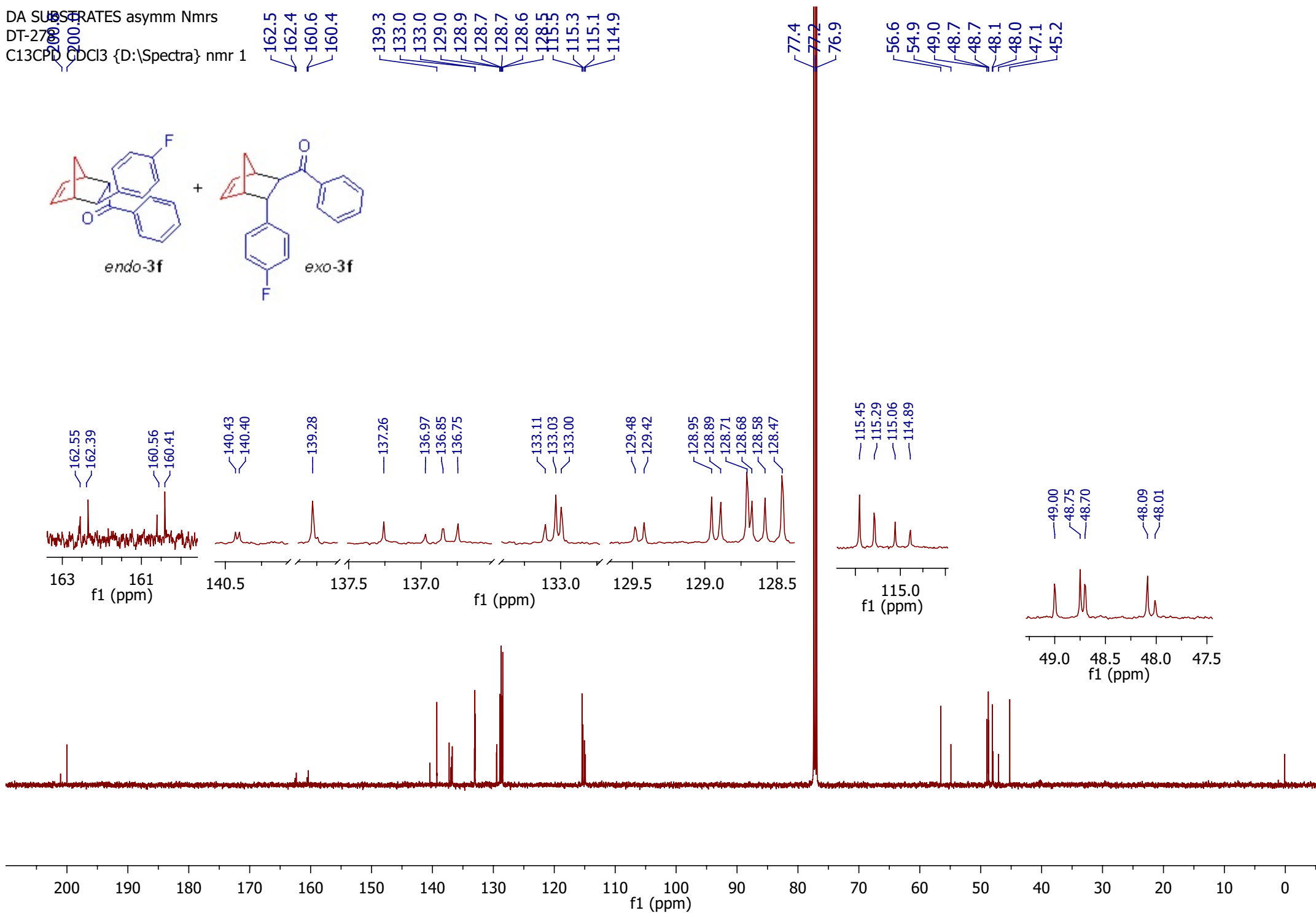
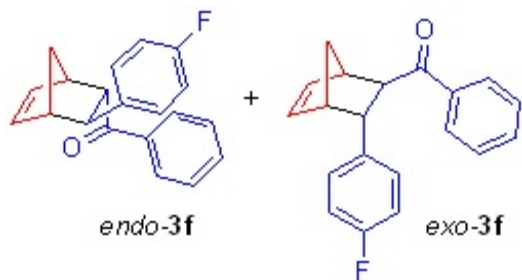
DA SUBSTRATES asymm Nmrs
DT-275
C13CPD, CDCl3 {D:\Spectra} nmr 1



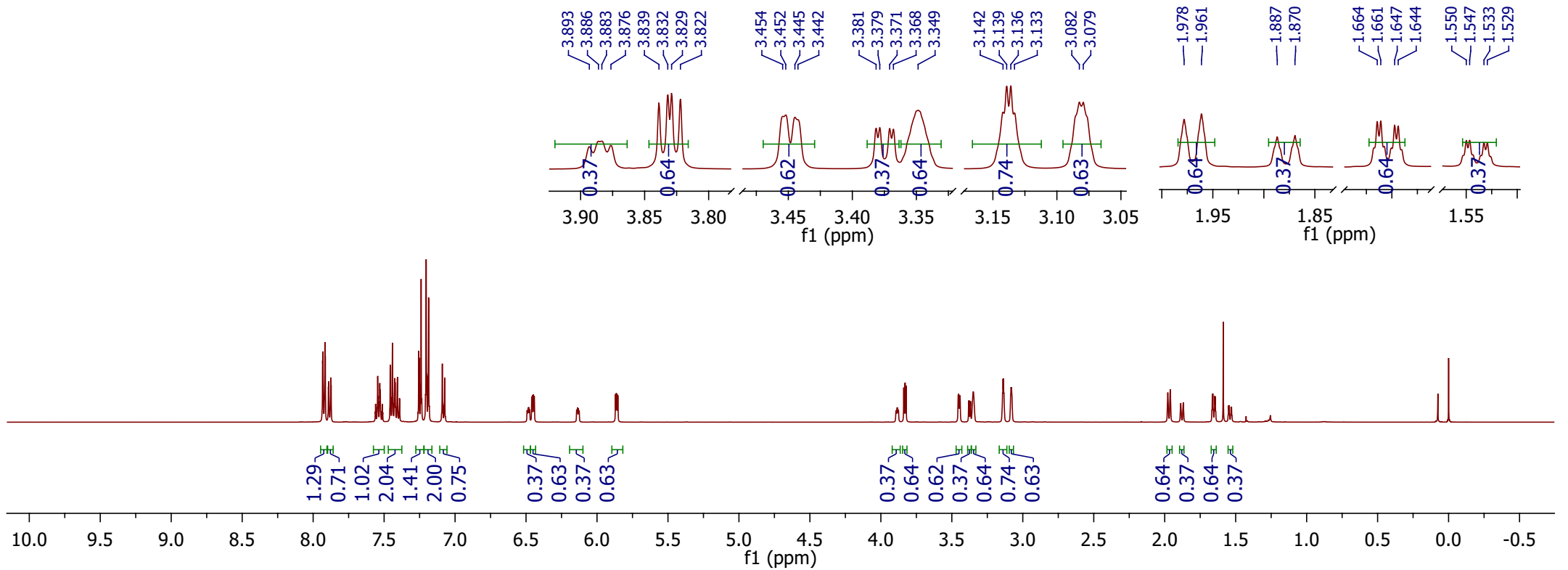
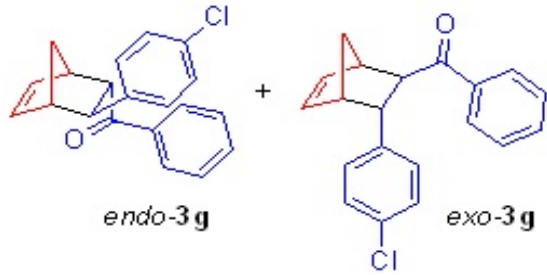
DA SUBSTRATES asynon Nmr1
 1H scan CDCl3 (D:\Spectra\hmr1
 7.934 7.932 7.917 7.915 7.868
 7.545 7.530 7.523 7.456 7.440 7.425 7.416 7.399 7.260 7.235 7.224 7.221 7.218 7.207 7.104 7.093 6.986 6.972 6.968 6.964 6.951 6.932 6.915 6.898 6.462 6.456 6.451 6.445 5.871 5.865 5.860 5.854 3.842 3.835 3.832 3.825 3.447 3.438 3.345 3.138 3.134 3.131 3.076 3.073 1.999 1.982 1.887 1.869



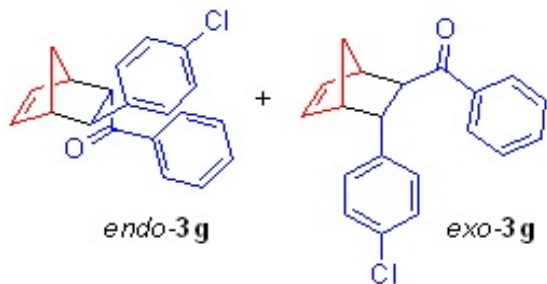
DA SUBSTRATES asymm Nmrs
DT-278
C13CPD CDCl3 {D:\Spectra} nmr 1



DA SUBSTRATES asymmetric Nmr
 D8
 1H Scan CDCl3 3D Spectra
 7.911
 7.899
 7.911
 7.891
 7.891
 7.877
 7.877
 7.545
 7.545
 7.530
 7.528
 7.457
 7.454
 7.441
 7.426
 7.422
 7.407
 7.406
 7.391
 7.256
 7.252
 7.243
 7.239
 7.234
 7.208
 7.204
 7.200
 7.190
 7.187
 7.090
 7.073
 6.459
 6.453
 6.448
 6.442
 5.869
 5.864
 5.858
 5.853
 3.839
 3.832
 3.829
 3.822
 3.454
 3.452
 3.445
 3.442
 3.381
 3.379
 3.371
 3.368
 3.349
 3.142
 3.139
 3.136
 3.133
 3.082
 3.079
 1.978
 1.961
 1.887
 1.870
 1.664
 1.661
 1.647
 1.644
 1.550
 1.547
 1.533
 1.529
 1.588



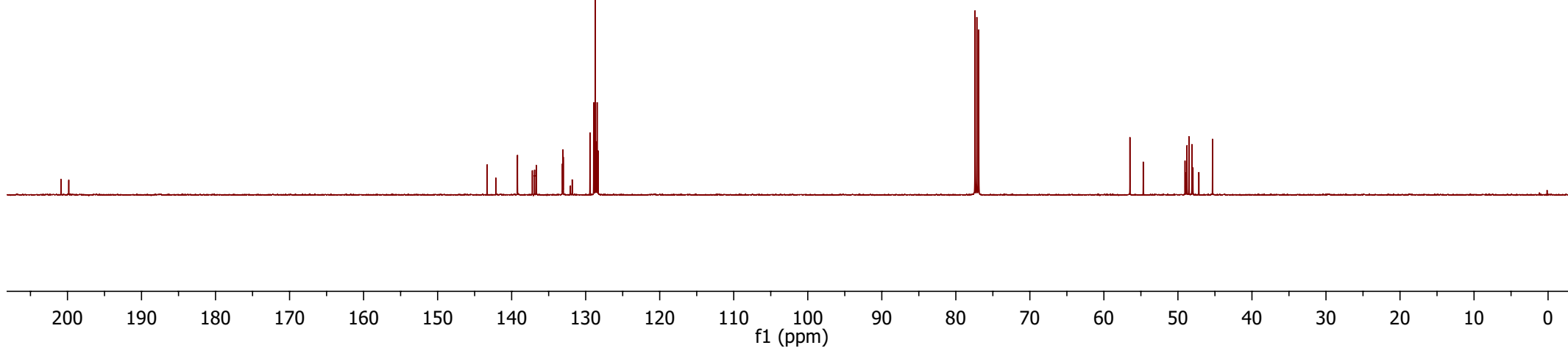
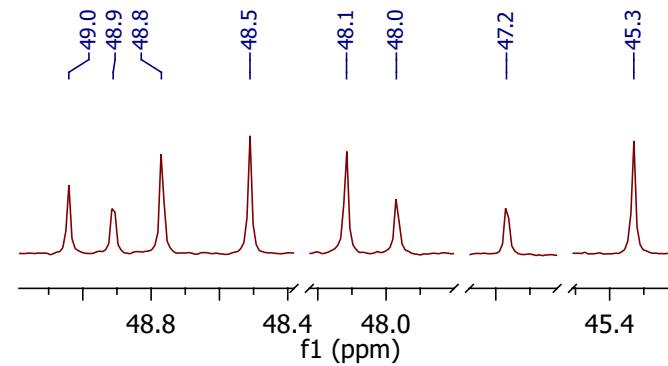
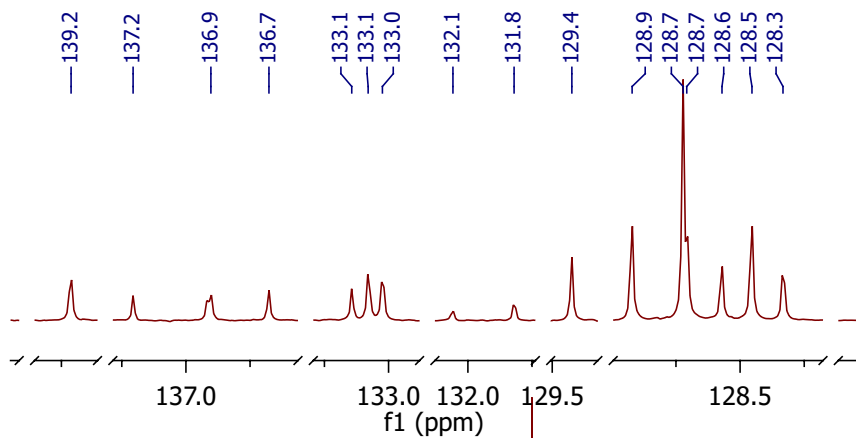
DA SUBSTRATES asymm Nmrs
DT-279
C13CPD CDCI3 {D:\Spectra} nmr 1



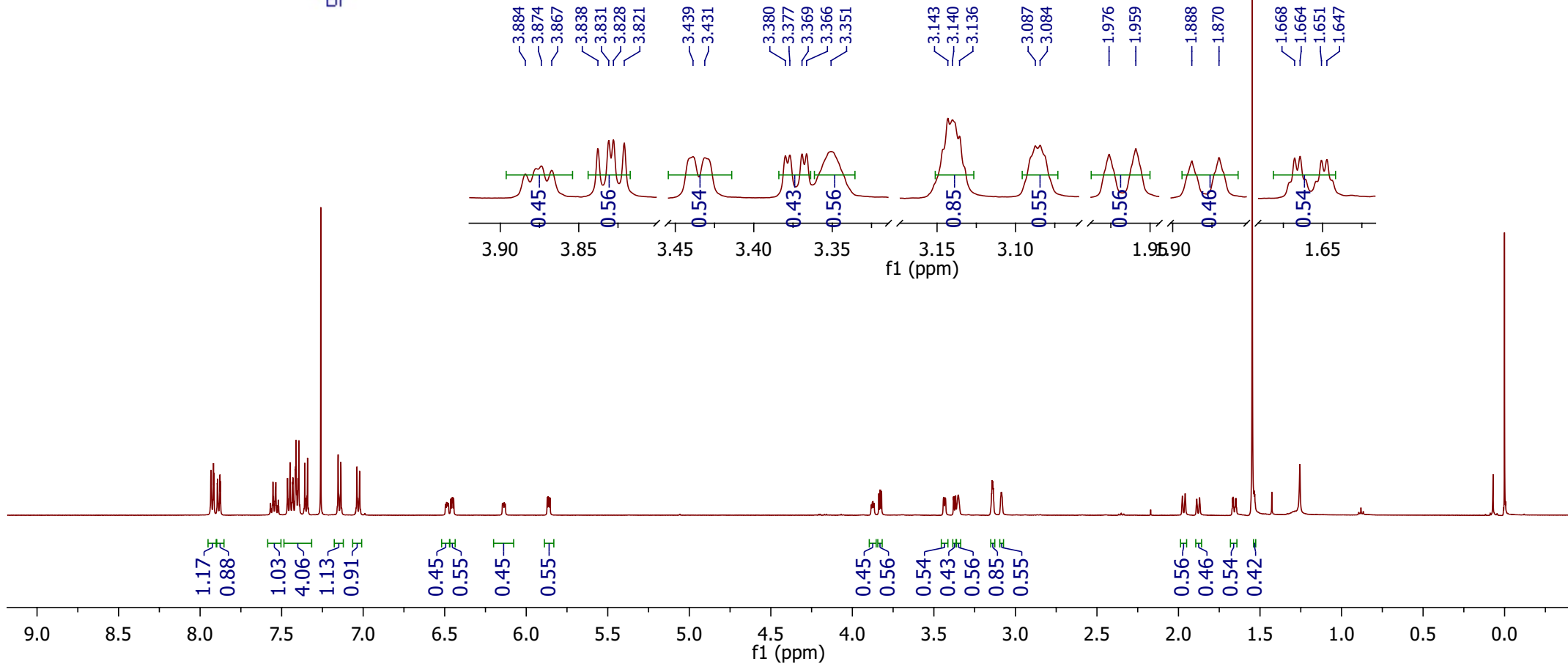
143.3
142.1
139.2
137.2
136.9
136.7
133.1
133.1
133.0
132.1
131.8
129.4
128.9
128.7
128.7
128.6
128.5
128.3

77.4
77.2
76.9

56.5
54.7
49.0
48.9
48.8
48.5
48.1
48.0
47.2
45.3



DA SUBSTRATE
 D81
 1H scan CDCl3 (D:\Spectra\hmr8
 7.984
 7.981
 7.922
 7.918
 7.917
 7.899
 7.895
 7.877
 7.877
 7.555
 7.550
 7.538
 7.536
 7.521
 7.448
 7.433
 7.431
 7.414
 7.411
 7.398
 7.394
 7.358
 7.341
 7.260
 7.153
 7.137
 7.038
 7.036
 7.024
 7.022
 6.463
 6.456
 6.452
 6.445
 5.871
 5.865
 5.860
 5.854
 3.838
 3.831
 3.828
 3.821
 3.439
 3.431
 3.380
 3.377
 3.369
 3.366
 3.351
 3.143
 3.140
 3.136
 3.087
 3.084
 1.976
 1.959
 1.888
 1.870
 1.668
 1.664
 1.651
 1.647
 1.959
 1.870
 1.668
 1.664
 1.651
 1.647
 1.536
 1.532



DA SUBSTRATES asymm Nmrs
DT-28
C13CPD CDCl3 {D:\Spectra} nmr 8



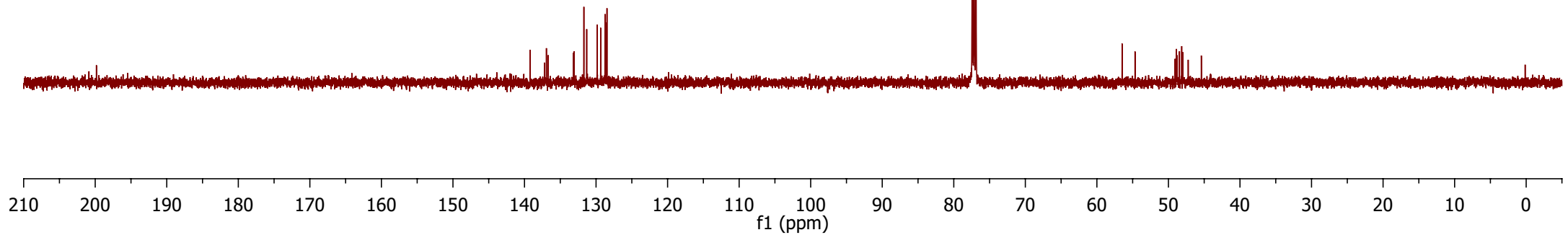
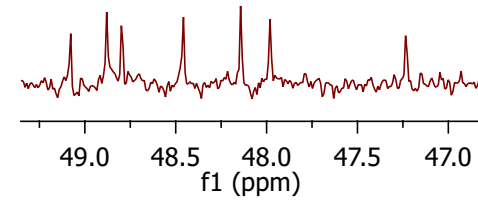
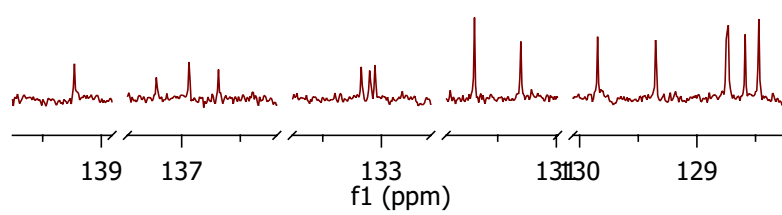
139.2
137.2
136.9
136.7
133.2
133.1
133.1
131.7
131.3
129.8
129.4
128.7
128.6
128.5

77.4
77.2
76.9

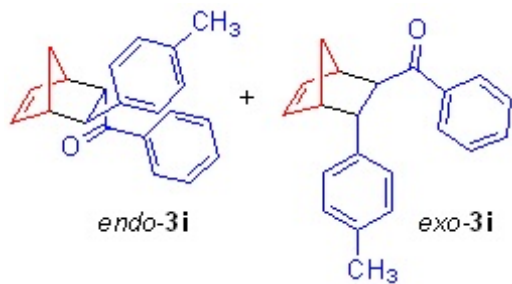
56.5
54.7
49.1
48.9
48.8
48.5
48.1
48.0
47.2
45.4

139.2
137.2
136.9
136.7
133.2
133.1
133.1
131.7
131.3
129.8
129.4
128.7
128.6
128.5

49.1
48.9
48.8
48.5
48.1
48.0
47.2



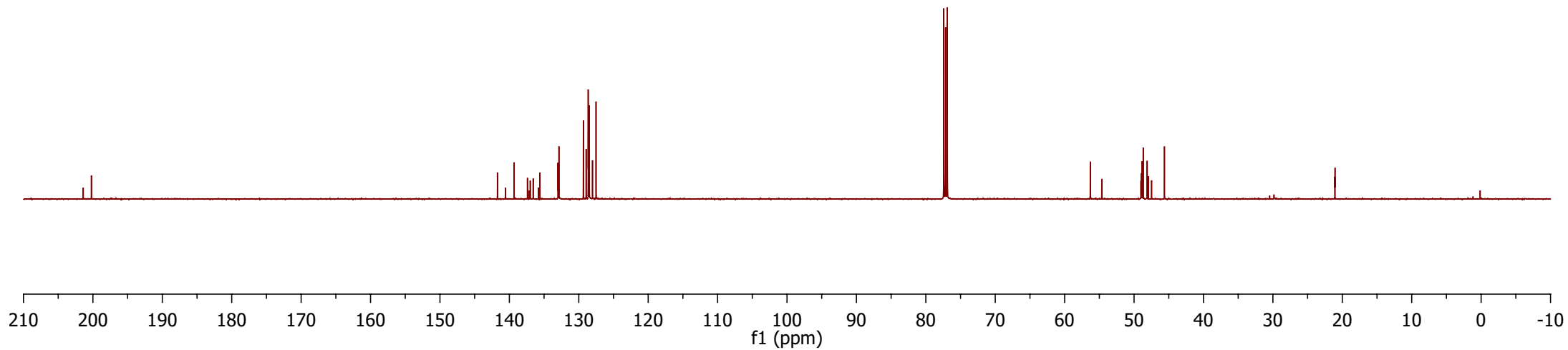
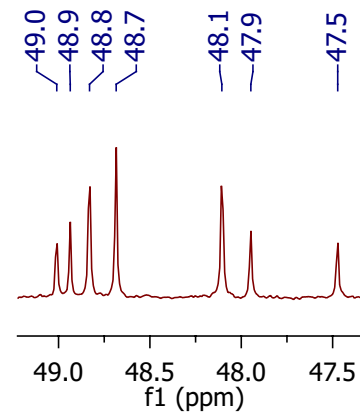
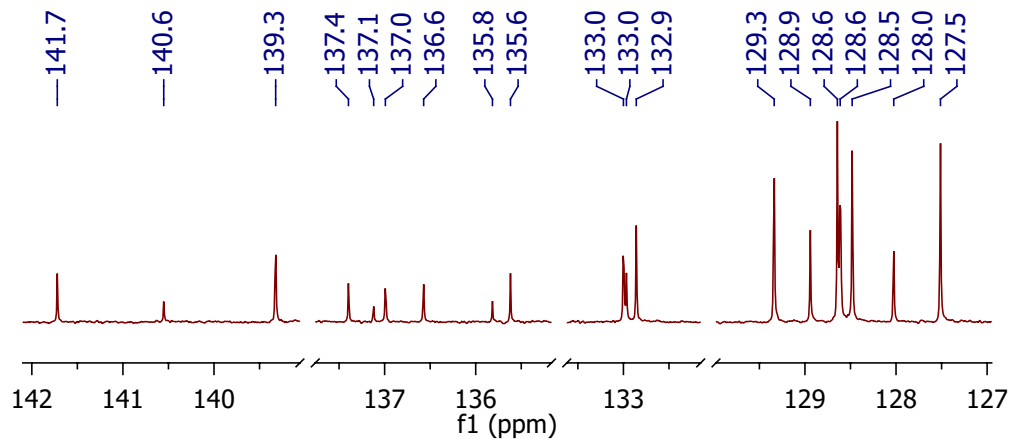
DA SUBSTRATES asymm Nmrs
DT-288
C13CPD CDCl3 {D:\Spectra} nmr 13



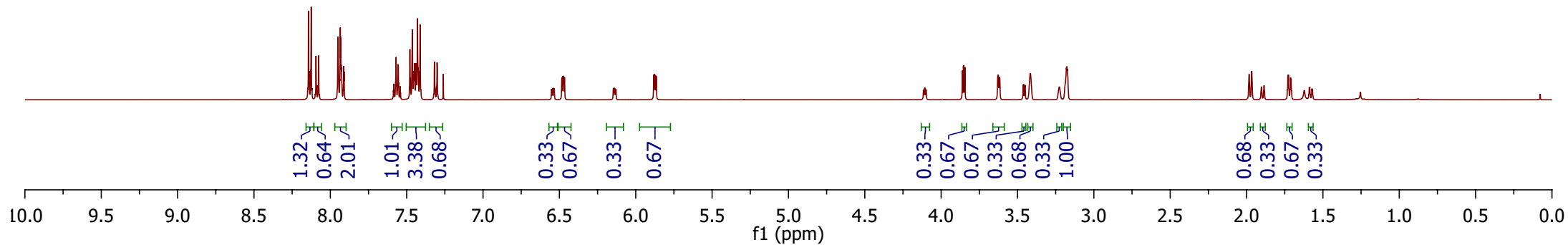
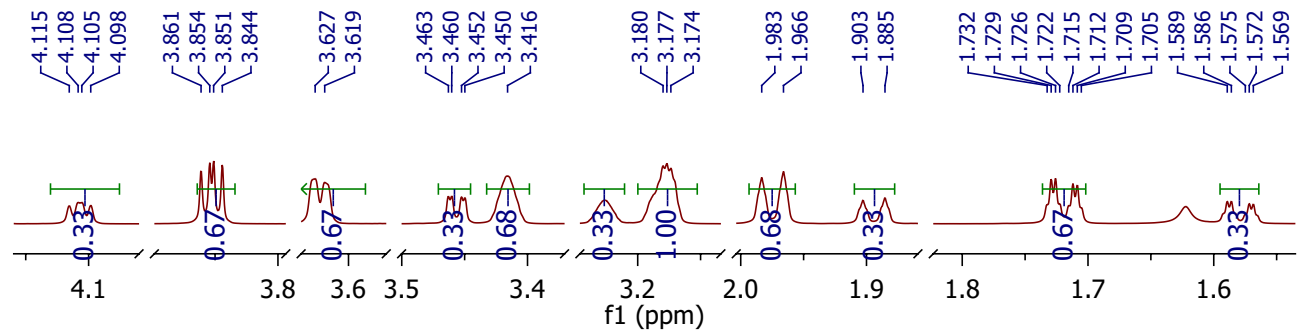
141.7
140.6
139.3
137.4
137.1
137.0
136.6
135.8
135.6
133.0
133.0
132.9
129.3
128.9
128.6
128.6
128.5
128.0
127.5

77.4
77.2
76.9

56.3
54.7
49.0
48.9
48.8
48.7
48.1
47.9
47.5
45.6
30.5
29.8
21.1
21.1



DA SUBSTRATES
D44
1H Scan (CDCl3) (D:\Spectra\hmr1
8.122
8.122
8.122
8.091
8.088
8.074
7.955
7.949
7.933
7.935
7.932
7.929
7.927
7.912
7.910
7.570
7.557
7.555
7.479
7.476
7.463
7.454
7.451
7.448
7.439
7.438
7.436
7.429
7.425
7.415
7.412
7.318
7.301
7.260
6.484
6.477
6.473
6.466
5.882
5.877
5.871
5.865
3.861
3.854
3.851
3.844
3.627
3.619
3.463
3.460
3.452
3.450
3.416
3.180
3.177
3.174
1.983
1.966
1.903
1.885
1.732
1.729
1.726
1.722
1.715
1.712
1.709
1.705
1.589
1.586
1.575
1.572
1.569



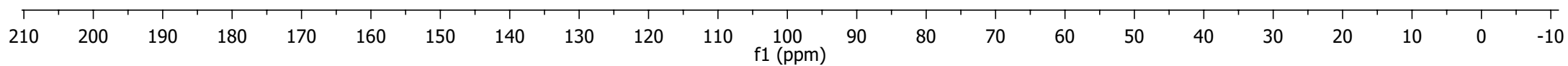
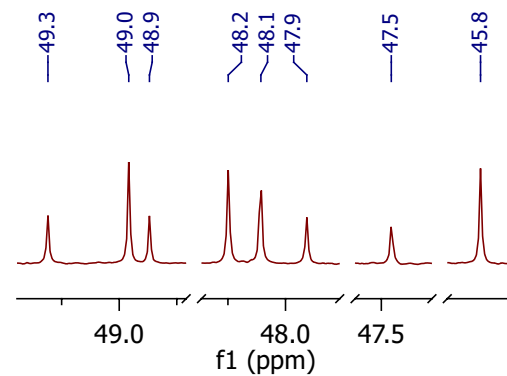
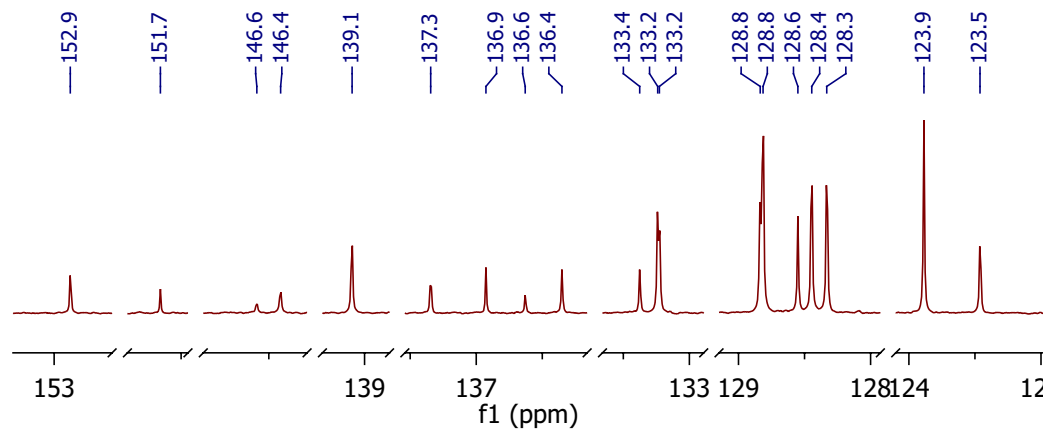
DA SUBSTRATES asymm Nmrs
DT-288
C13CPD CDCl3 {D:\Spectra} nmr 1



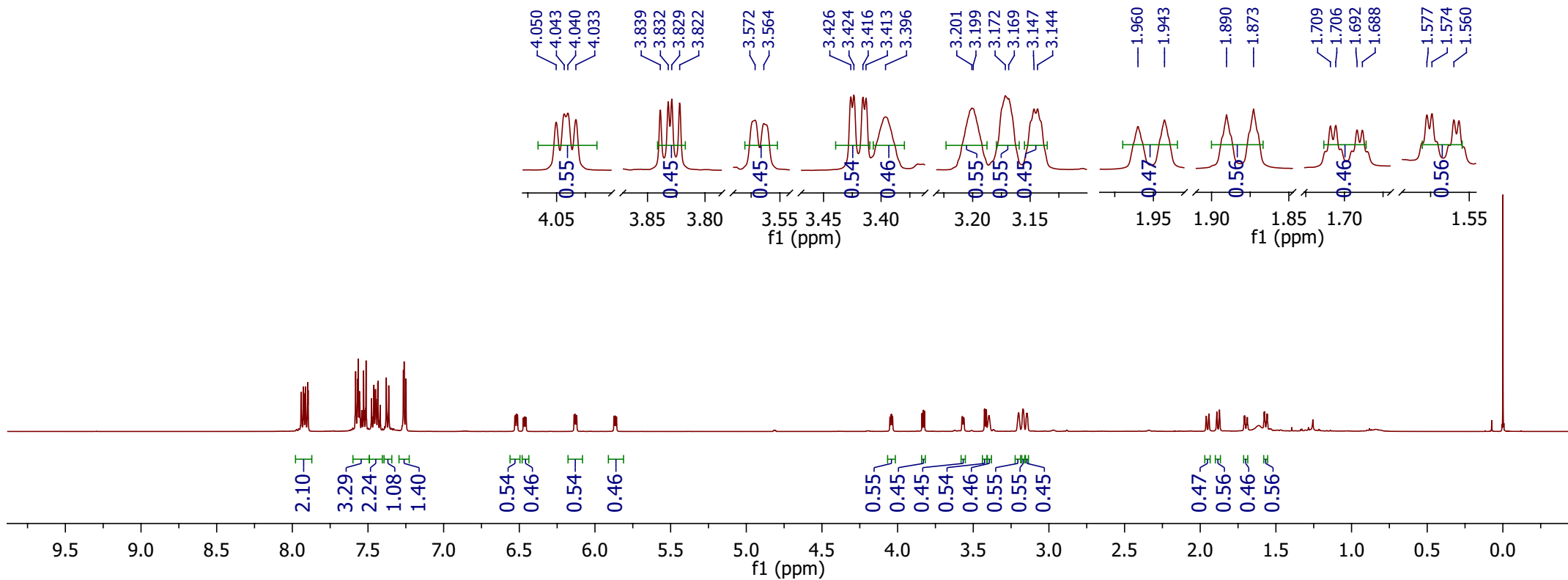
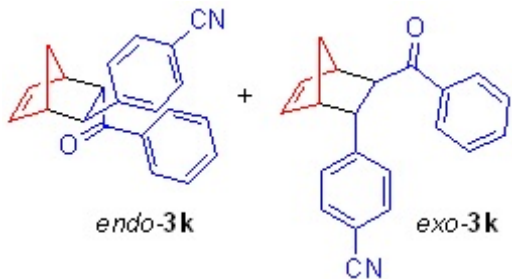
152.9
151.7
146.6
146.4
139.1
137.3
136.9
136.6
136.4
133.4
133.2
133.2
128.8
128.8
128.6
128.4
128.3
123.9
123.5

77.4
77.2
76.9

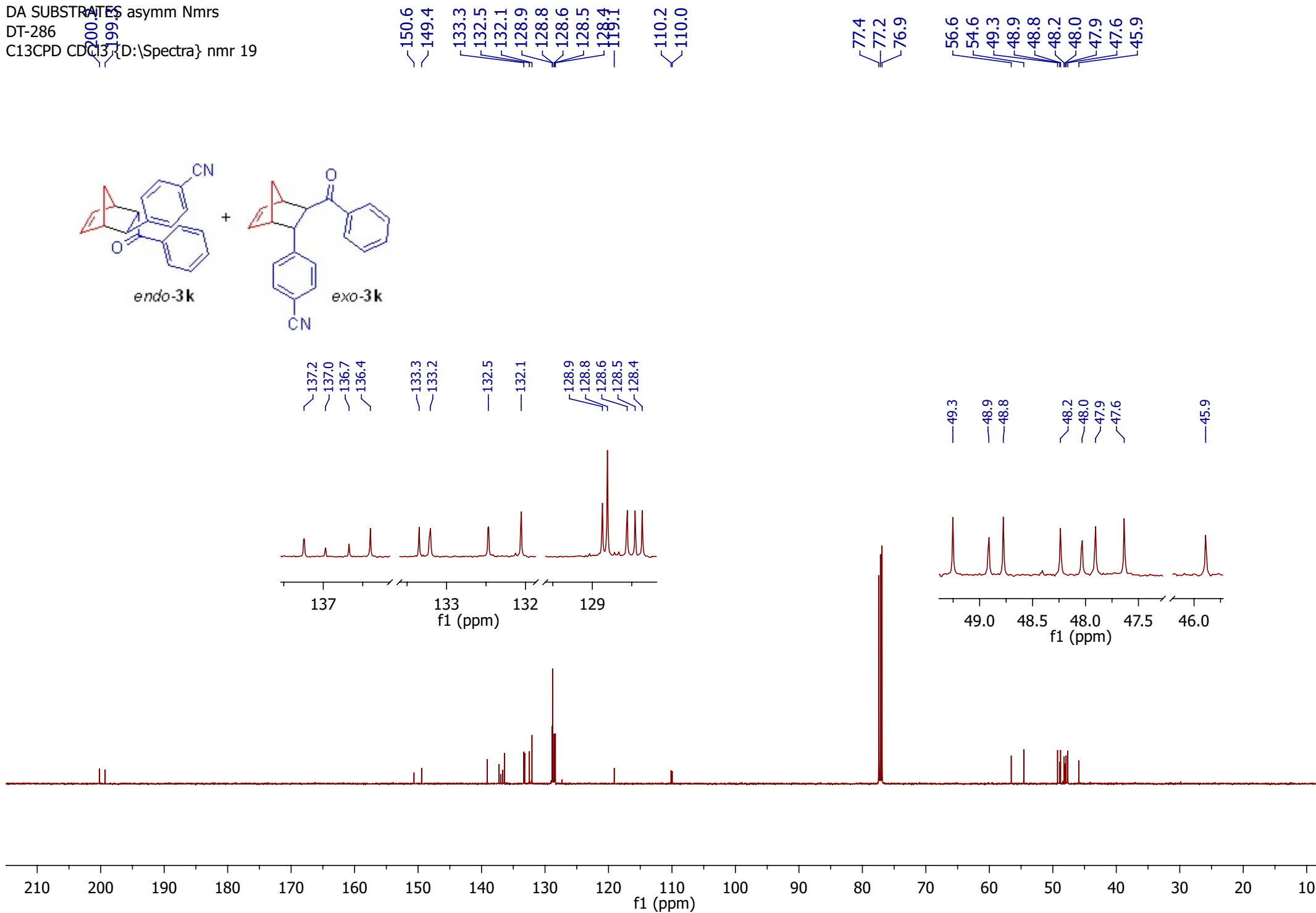
56.7
54.7
49.3
49.0
48.9
48.2
48.1
47.9
47.5
45.8



DA SUBSTRATES asymmetric NMR
 D4 7.846
 1H 7.836
 7.922
 7.922
 7.911
 7.911
 7.898
 7.896
 7.582
 7.565
 7.556
 7.556
 7.529
 7.513
 7.477
 7.461
 7.450
 7.446
 7.435
 7.434
 7.379
 7.363
 7.266
 7.261
 7.250
 6.529
 6.523
 6.518
 6.512
 6.138
 6.132
 6.126
 6.121
 5.874
 5.869
 5.863
 4.043
 4.040
 4.033
 3.839
 3.839
 3.832
 3.829
 3.822
 3.572
 3.564
 3.426
 3.424
 3.416
 3.413
 3.396
 3.201
 3.199
 3.172
 3.169
 3.147
 3.144
 1.960
 1.943
 1.890
 1.873
 1.709
 1.706
 1.692
 1.688
 1.577
 1.574
 1.560

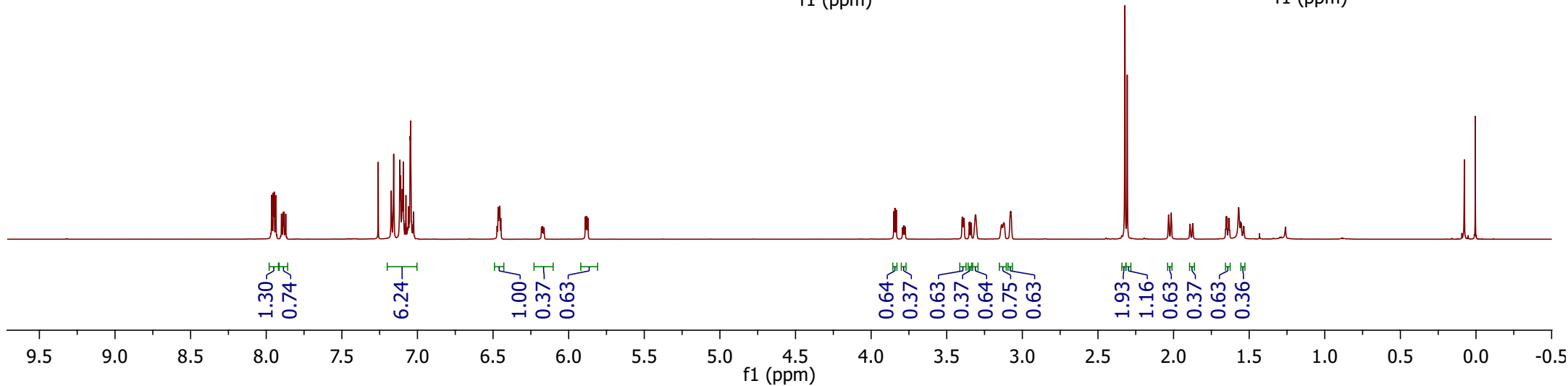
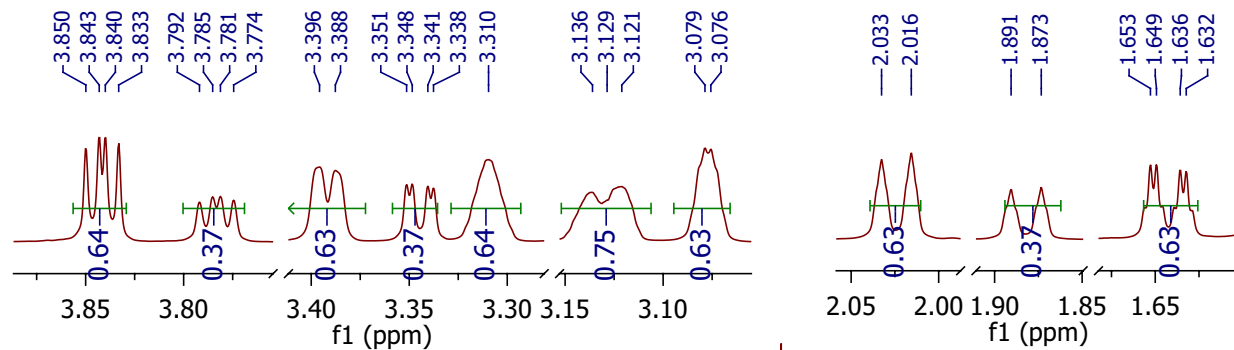
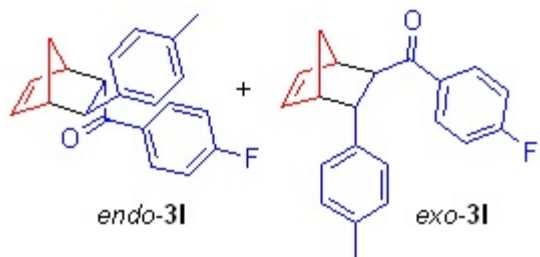


DA SUBSTRATES asymm Nmrs
DT-286
C13CPD CDCl3 (D:\Spectra} nmr 19



DA SUBSTRATE AcEsasymm Nmr
 D6
 1H scan CDCl3 (D) Spectra nmr4

7.764
7.755
7.742
7.746
7.936
7.891
7.888
7.888
7.888
7.888
7.877
7.260
7.173
7.157
7.116
7.111
7.106
7.100
7.094
7.090
7.076
7.060
7.055
7.048
7.045
7.039
7.025
6.466
6.460
6.455
6.449
6.449
5.889
5.884
5.878
5.873
3.850
3.843
3.840
3.833
3.792
3.785
3.781
3.774
3.396
3.388
3.351
3.348
3.341
3.338
3.310
3.136
3.129
3.121
3.079
3.076
2.033
2.016
2.306
2.033
2.016
2.016
1.891
1.873
1.653
1.649
1.636
1.632
1.636
1.632
1.632
1.632
1.569
1.556
1.552



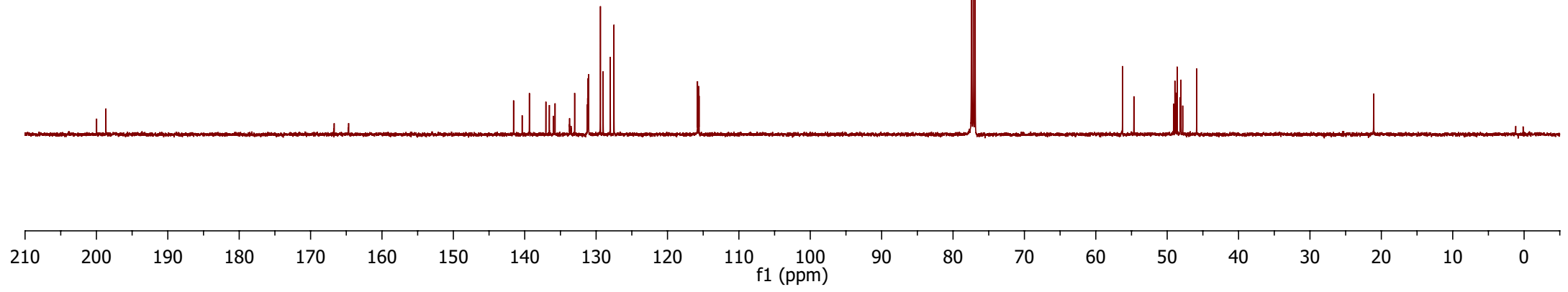
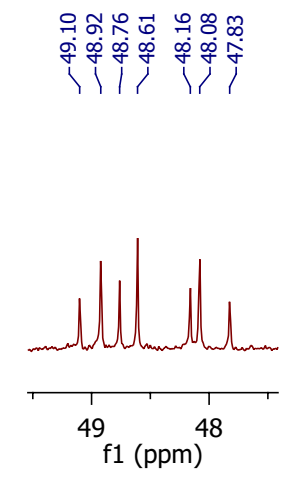
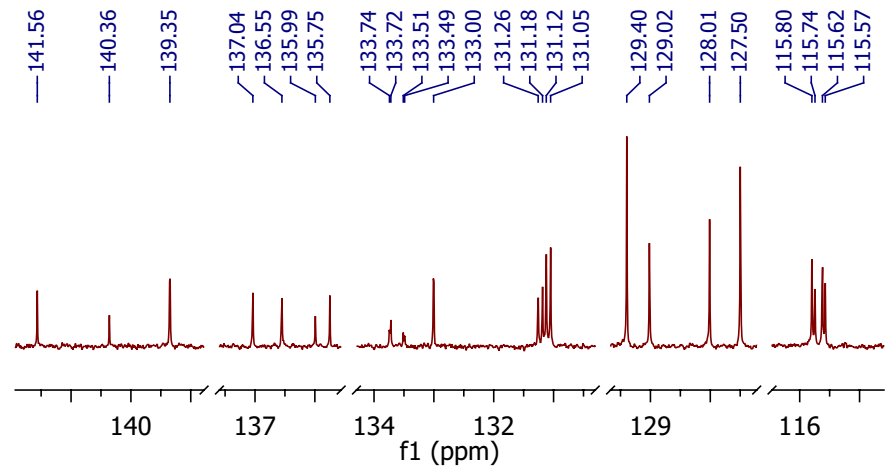
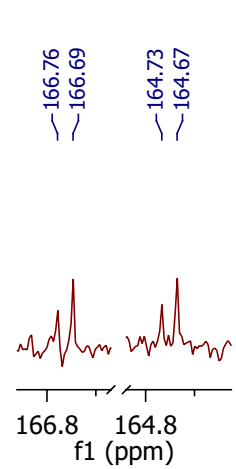
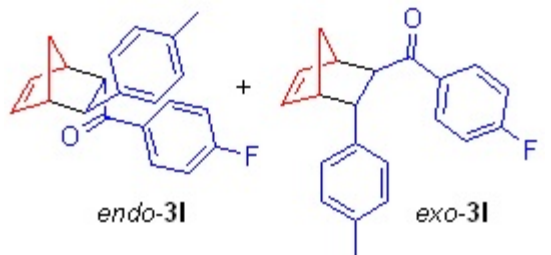
DA SUBSTRATES asymm Nmrs
DT-282
C13CPD CDCl3 {D:\Spectra} nmr 52

200.6
198.7
166.7
164.7
141.6
139.3
137.0
135.7
133.0
131.2
131.1
131.0
129.4
129.0
128.0
127.5
115.8
115.7
115.6
115.6

77.4
77.2
76.9

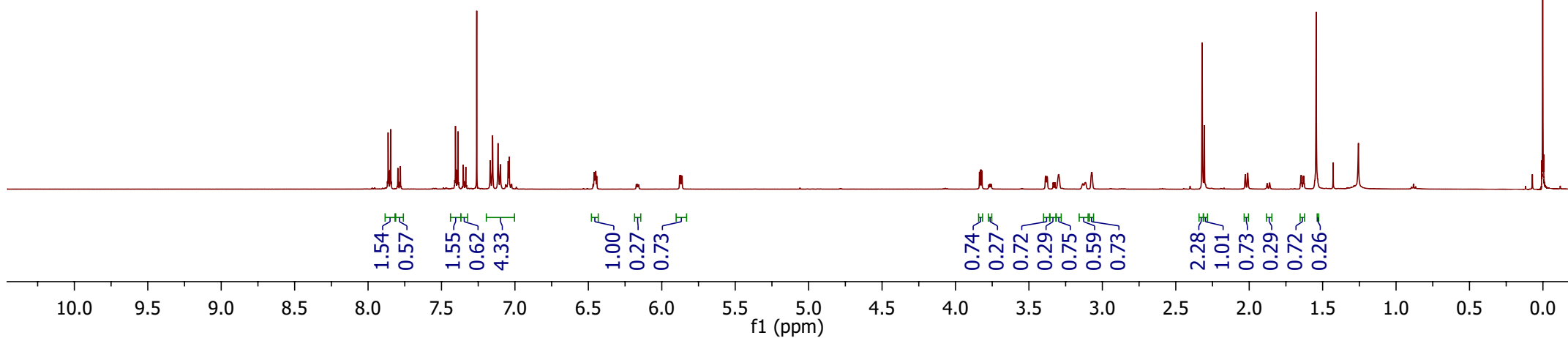
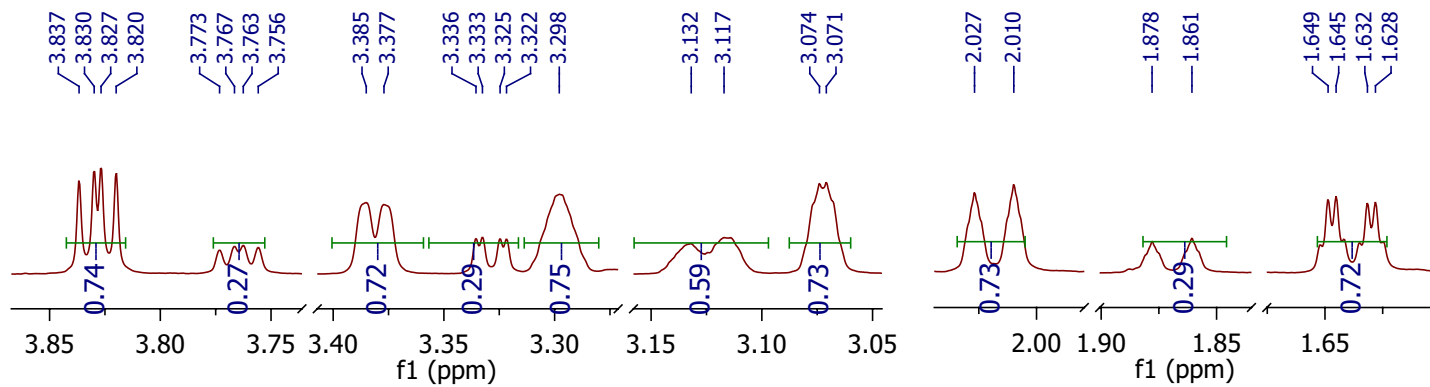
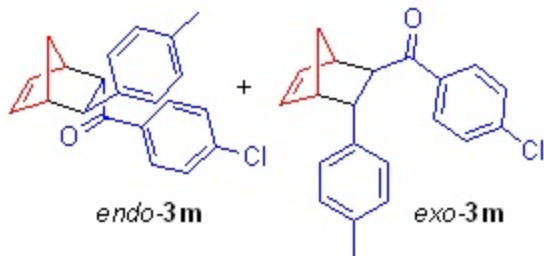
56.3
54.7
49.1
48.9
48.8
48.6
48.2
48.1
47.8
45.9

21.1

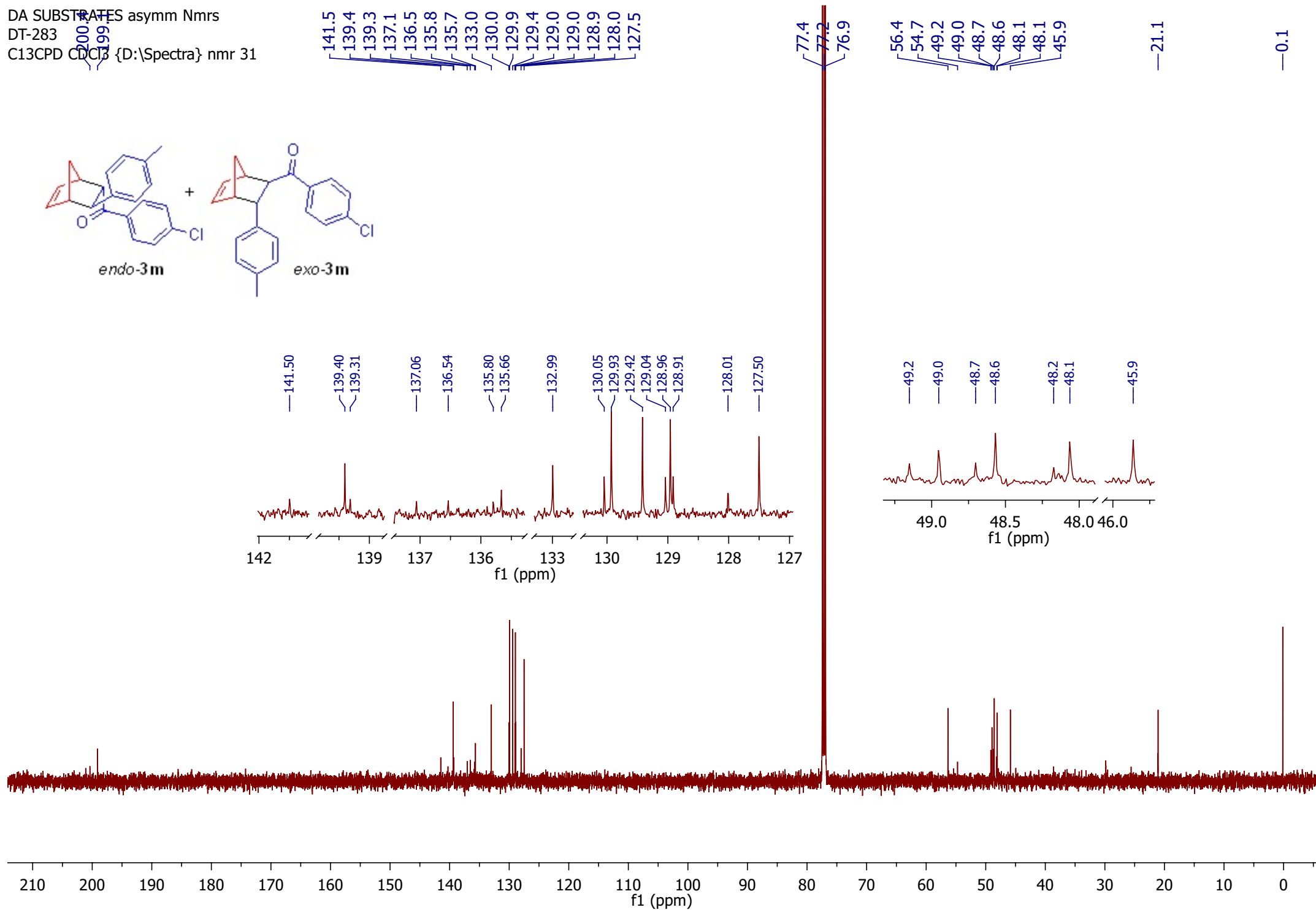
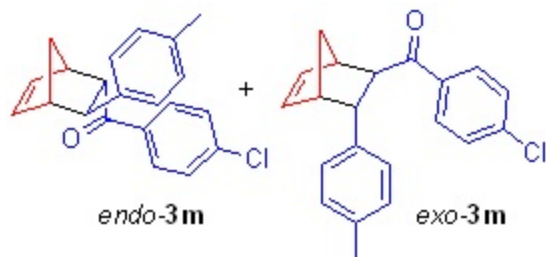


D:\SUBSTRATES\asymm\Nmr\1H scan CDCl3 (D)\Spectra\hmr31

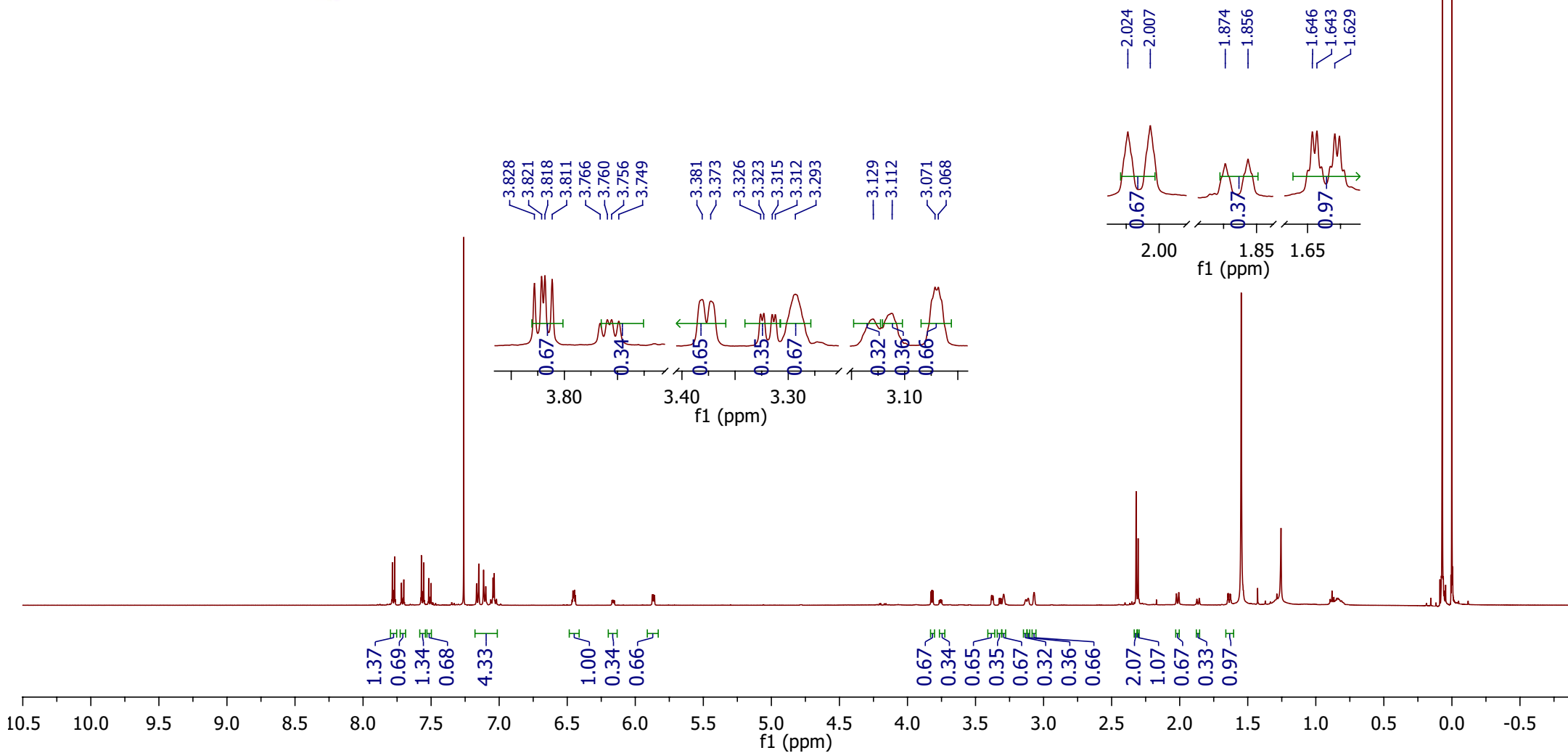
7.866
7.863
7.866
7.858
7.843
7.841
7.799
7.795
7.784
7.788
7.410
7.405
7.401
7.395
7.391
7.387
7.383
7.352
7.348
7.338
7.334
7.260
7.168
7.152
7.115
7.099
7.046
7.039
6.461
6.454
6.449
6.443
5.879
5.873
5.867
5.862
3.837
3.830
3.827
3.820
3.767
3.763
3.385
3.377
3.336
3.333
3.325
3.322
3.298
3.132
3.117
3.074
3.071
2.027
2.010
1.878
1.861
1.649
1.645
1.632
1.628
1.628
1.429



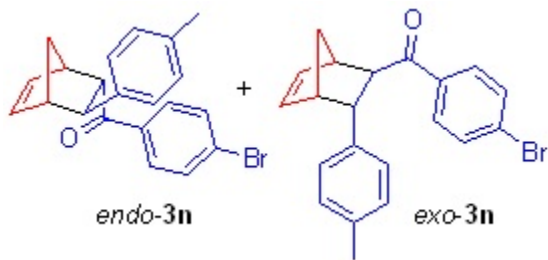
DA SUBSTRATES asymm Nmrs
DT-283
C13CPD CDCl3 {D:\Spectra} nmr 31



DA SUBSTRATES Asymmetric
 1H NMR Scan CDCl3 (DMSO-d6) Spectra pmr 55



DA SUBSTRATES asymm Nmrs
DT-28
C13CPD CDCl3 {D:\Spectra} nmr 55



141.6
139.4
138.6
137.2
137.1
136.0
135.8
135.1
133.0
132.0
131.9
131.9
130.2
130.1
129.4
129.0
128.0
127.5

77.4
77.2
76.9

56.3

49.2
49.0
48.7
48.6
48.6
48.2
48.1
47.9

21.1

141.62

139.41

138.61

137.21

137.06

136.02

135.80

135.07

133.00

131.95

131.91

131.88

130.19

130.08

129.42

129.03

128.01

127.50

49.2

49.0

48.7

48.6

48.6

48.2

48.1

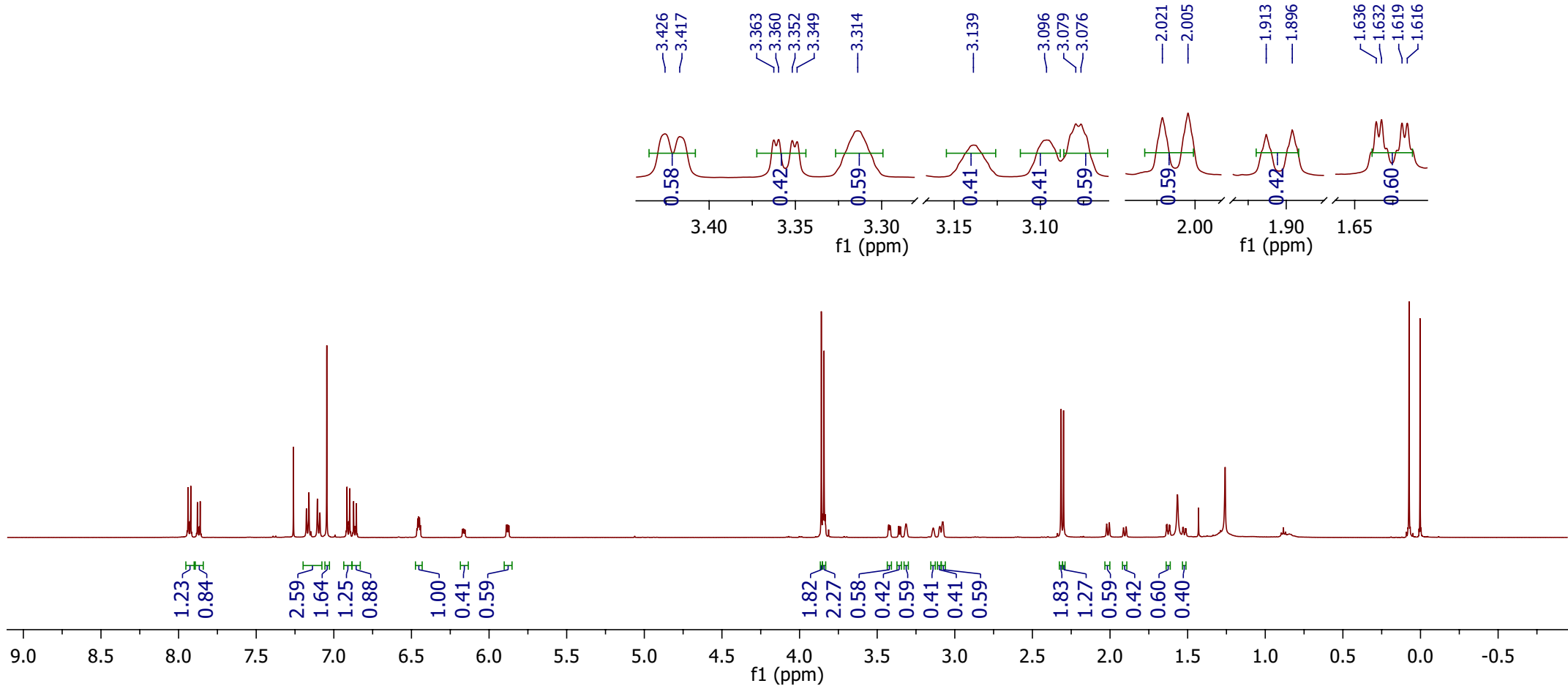
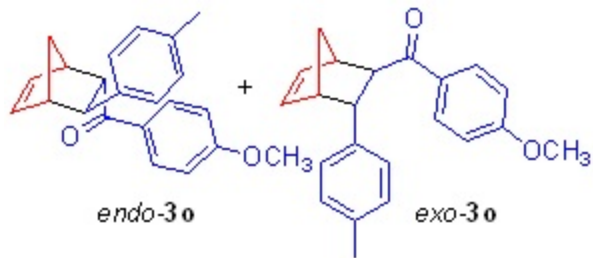
47.9

142 140 138 136 134 f1 (ppm)

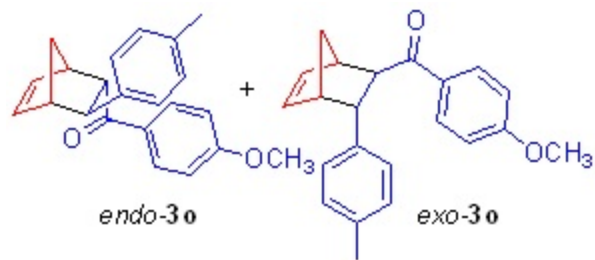
48.5 47.5 f1 (ppm)

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

DA SUBSTRATES asymmetric
D 285 CH₃/OCH₃
1H scan CDCl₃ (D:\Spectra\hmr14



DA SUBSTRATES asymm Nmrs
DT-285 CH3/OCH3
C13CPD CDCl3 {D:\Spectra} nmr 14



163.4
163.4
141.9
139.2
136.9
136.7
135.8
133.1
130.9
130.8
129.3
128.9
128.1
127.5
113.8
113.8

77.4
77.2
76.9
55.9
55.6
55.6
54.3
49.1
48.9
48.8
48.8
48.1
48.1
47.6
45.7
29.8
21.1
21.1

