Design and Synthesis of a Novel Chiral Triptycene-Based Imine Ligand for Enantioselective Epoxide Ring-Opening Reactions

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1. Experimental Section

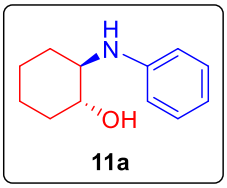
1.1. Materials & Methods

All the solvents and reagents were purchased at the highest commercially quality and used without further purification. 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. Powder X-ray diffraction (PXRD) spectra were recorded by using Panalytical's X'Pert Pro X-ray diffractometer. Field emission scanning electron microscope (FESEM) images and Energy-dispersive X-ray spectroscopy (EDS) data was obtained on a field emission scanning electron microscope (HITACHI Japan SU8010 Series). Crude products were isolated and purified by column chromatography over 60-120 mesh size silica gel using hexanes and 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 in UV chamber.

1.2. General Procedure for Epoxide Ring-Opening Reaction:

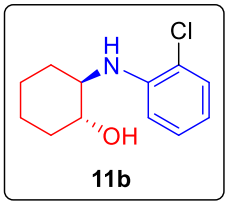
In a 25 mL round-bottom flask, epoxide (0.4 mmol, 1 equiv.) and 5 mol% of the triptycene-based cobalt catalyst (0.02 mmol) were dissolved in 1 mL of *tert*-butyl methyl ether. Subsequently, 5 mol% of an additive (0.02 mmol) was added to the reaction mixture. The mixture was stirred at room temperature, followed by the addition of aniline (0.4 mmol, 1 equiv.) or its derivatives. The reaction was allowed to proceed for the optimized reaction time, and progress was monitored by TLC. Upon completion, the solvent was evaporated under reduced pressure, and the crude product was purified by column chromatography using a gradient of ethyl acetate and hexanes (10:90 to 20:80) as the eluents to afford the pure product. The enantiomeric excess was determined by HPLC analysis using chiral columns Chiralpak IA, IB, ASH and phenomenex Amylose-1 (λ = 254 nm, IPA/hexanes used as the eluent).

1.3. Characterization Data



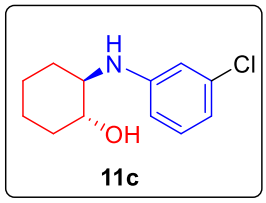
11a

2-(phenylamino)cyclohexan-1-ol (11a): 92% yield, brown oil; **IR (ATR):** ν_{max} 3349, 2958, 2923, 2853, 1611, 1461, 1278, 1261, 764, 750 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.20 (t, *J* = 8Hz, 2H), 6.83-6.77 (m, 3H), 3.41 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.19-3.11 (m, 1H), 2.16-2.05 (m, 2H), 1.82-1.68 (m, 2H), 1.46-1.34 (m, 1H), 1.34- 1.26 (m, 2H), 1.16-1.06 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 146.8, 129.5, 119.5, 115.4, 74.3, 61.2, 33.4, 31.4, 25.1, 24.4, ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 8.02 min, τ_R (major) = 8.6 min, 52% ee.



11b

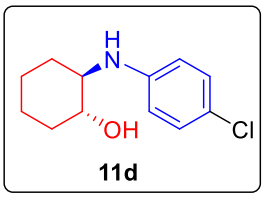
2-((2-chlorophenyl)amino)cyclohexan-1-ol (11b): 99% yield, Light brown oil **IR (ATR):** ν_{max} 3401, 2929, 2856, 1596, 1515, 1325, 1032, 739, 694 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.28-7.25 (m, 1H), 7.15-7.11 (m, 1H), 6.89 (d, *J* = 8 Hz, 1H), 6.68 (td, J_1 = 1.5 Hz, J_2 = 8 Hz, 1H), 3.49 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.25-3.17 (m, 1H), 2.17-2.04 (m, 2H), 1.83-1.71 (m, 2H), 1.46-1.37 (m, 1H), 1.36-1.29 (m, 2H), 1.22-1.12 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 143.4, 129.6, 128.0, 118.8, 115.6, 113.7, 74.5, 60.4, 33.3, 31.6, 25.1, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 6.4 min, τ_R (major) = 9.1 min, 20% ee.



11c

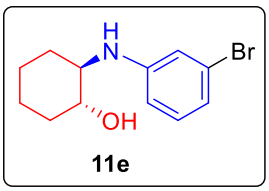
2-((3-chlorophenyl)amino)cyclohexan-1-ol (11c): 93% yield, light brown oil, **IR (ATR):** ν_{max} 3347, 2931, 2857, 1596, 1503, 1483, 1280, 988, 938, 889, 682 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.07 (t, *J* = 8.5 Hz, 1H), 6.72-6.68 (m, 2H), 6.59-6.55 (m, 1H), 3.36 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.13-3.06 (m, 1H), 2.13-2.04 (m, 2H), 1.81-1.67 (m, 2H), 1.44-1.34 (m, 1H), 1.34-1.27 (m, 2H), 1.11-1.01 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 149.0, 135.2, 130.4, 118.4, 114.1, 112.8, 74.5, 60.2, 33.4, 31.6, 25.0, 24.4 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 9.1 min, τ_R (major) = 9.6 min, 62% ee.

2-((4-chlorophenyl)amino)cyclohexan-1-ol (11d): 99% yield, brown solid ; **IR (ATR):** ν_{max} 3403, 2924, 2856, 1671, 1375, 951, 849, 697 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.13 (dt, J_1 = 3 Hz, J_2 = 10 Hz, 2H), 6.70-6.66 (m, 2H), 3.38 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.12-3.05 (m, 1H), 2.14-2.04 (m, 2H), 1.82-1.68 (m, 2H), 1.44-1.34 (m, 1H), 1.34-1.26 (m, 2H), 1.13-1.03 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 145.8, 129.3, 123.7, 116.1, 74.4, 61.0, 33.4, 31.4, 25.0, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 10.6 min, τ_R (major) = 12..2 min, 42% ee.



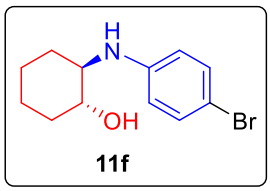
11d

2-((3-bromophenyl)amino)cyclohexan-1-ol (11e): 94% yield, brown oil; **IR (ATR):** ν_{max} 3348, 2930, 2856, 1593, 1500, 1480, 1449, 1325, 1279, 759, 582 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.01 (t, J = 8.5 Hz, 1H), 6.86-6.82 (m, 2H), 6.63-6.60 (m, 1H), 3.35 (td, J_1 = 4 Hz, J_2 = 9.5 Hz, 1H), 3.12-3.05 (m, 1H), 2.13-2.04 (m, 2H), 1.80-1.66 (m, 2H), 1.43-1.33 (m, 1H), 1.33-1.26 (m, 2H), 1.11-1.01 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 149.1, 130.7, 123.4, 121.2, 117.0, 113.2, 74.4, 60.2, 33.4, 31.6, 25.0, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 16.03 min, τ_R (major) = 17.4 min, 54% ee.



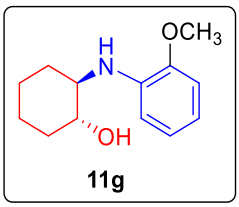
11e

2-((4-bromophenyl) amino) cyclohexan-1-ol (11f): 98% yield, white crystal; mp: 75-77° C,**IR (ATR):** ν_{max} 3375, 2931, 2858, 1594, 1514, 1482, 1280, 1260, 766, 755, 505 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.23 (dt, J_1 = 3.5 Hz, J_2 = 10 Hz, 2H), 6.57 (dt, J_1 = 3.5 Hz, J_2 = 10 Hz, 2H), 3.36-3.30 (m, 1H), 3.08-3.01 (m, 1H), 2.11-2.02 (m, 2H), 1.78-1.66 (m, 2H), 1.42-1.32 (m, 1H), 1.31-1.24 (m, 1H), 1.08-0.97 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 145.8, 132.0, 116.0, 110.0, 74.4, 60.3, 33.4, 31.4, 24.9, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 12.3 min, τ_R (minor) = 16.2 min, 52% ee.



11f

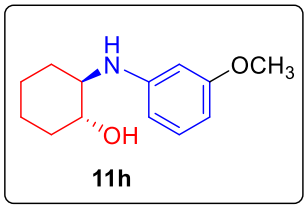
2-((2-methoxyphenyl) amino) cyclohexan-1-ol (11g): 85% yield, Greyish blue solid ; mp: 70-72° C; **IR (ATR):** ν_{max}, 3383, 2926, 1600, 1521, 1451, 1345, 1224, 1048, 725 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 6.88 (td, J_1 = 1 Hz, J_2 = 7.5 Hz 1H), 6.82-6.78 (m, 2H), 6.72 (td, J_1 = 1.5



11g

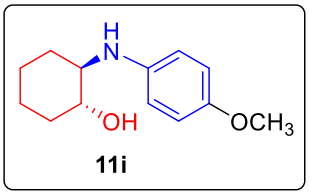
Hz, J_2 = 7.5 Hz, 1H), 3.84 (s, 1H), 3.46-3.40 (m, 1H), 3.18-3.13 (m, 1H), 2.17-2.07 (m, 2H), 1.83-1.69 (m, 2H), 1.47-1.38 (m, 1H), 1.38-1.26 (m, 1H), 1.14-1.04 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 147.5, 137.5, 121.3, 117.3, 111.5, 109.8, 74.4, 59.6, 55.4, 33.2, 31.5, 25.0, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 7.8 min, τ_R (major) = 9.3 min, 16% ee.

2-((3-methoxyphenyl)amino)cyclohexan-1-ol (11h): 82% yield, brown oil, **IR (ATR):** ν_{max} 3384, 2931, 2857, 1612, 1463, 1300, 1263, 1233, 1038, 829 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.07 (t, J = 8 Hz, 1H), 6.32-6.28 (m, 2H), 6.28-6.26 (m, 1H), 3.75 (s, 3H), 3.33-3.26 (m, 1H), 3.11-3.04 (m, 1H), 2.13-2.02 (m, 2H), 1.77-1.62 (m, 2H), 1.41-1.30 (m, 1H), 1.30-1.22 (m, 2H), 1.04-0.94 (m, 1H), ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 160.6, 149.2, 129.8, 107.0, 102.8, 100.0, 73.9, 59.6, 54.8, 33.2, 31.3, 24.6, 24.1 ppm. The ee was determined by HPLC using Chiralpak AS-H [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 13.9 min, τ_R (minor) = 16.2 min, 42% ee.



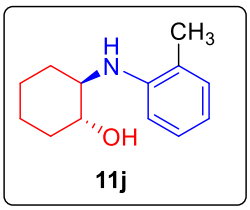
11h

2-((4-methoxyphenyl) amino) cyclohexan-1-ol (11i): 87% yield, brown oil, **IR (ATR):** ν_{max} 3336, 2929, 2857, 1509, 1451, 1235, 1234, 1067, 1034, 820 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 6.77 (dt, J_1 = 3.5 Hz, J_2 = 10 Hz, 2H), 6.68 (dt, J_1 = 3.5 Hz, J_2 = 10 Hz, 2H), 3.73 (s, 3H), 3.35-3.28 (m, 1H), 3.01-2.94 (m, 1H), 2.13-2.03 (m, 2H), 1.78-1.65 (m, 2H), 1.43-1.30 (m, 1H), 1.29-1.20 (m, 2H), 1.95-0.95 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 153.0, 141.5, 116.5, 114.9, 74.3, 61.7, 55.8, 33.3, 31.5, 25.1, 24.3 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 25.4 min, τ_R (minor) = 28.3 min, 4% ee.



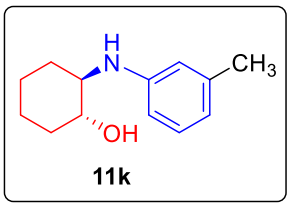
11i

2-((2-methylphenyl) amino) cyclohexan-1-ol (11j): 82% yield, blackish blue oil, **IR (ATR):** ν_{max} 3400, 3016, 2929, 2856, 1605, 1508, 1448, 1315, 1258, 744 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.16-7.11 (m, 1H), 7.09 (d, J = 7 Hz, 1H), 6.82 (d, J = 8 Hz, 1H), 6.72 (td, J_1 = 1 Hz, J_2 = 7.5 Hz, 1H), 3.45 (td, J_1 = 4 Hz, J_2 = 9.5 Hz, 1H), 3.25-3.18 (m, 1H), 2.20-2.11 (m, 5H), 1.83-1.77 (m, 1H), 1.76-1.69 (m, 1H), 1.48-1.38 (m, 1H), 1.37-1.27 (m, 2H), 1.15-1.05 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 145.5, 130.6, 127.2, 123.4, 118.2, 112.0, 74.6, 60.2, 33.4, 31.8, 25.1, 24.4,



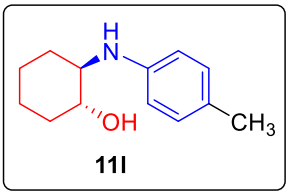
11j

17.8 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 10.6 min, τ_R (minor) = 11.1 min, 2% ee.



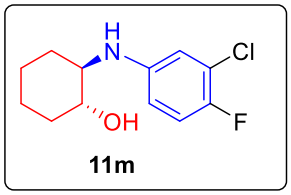
11k

2-(m-tolylamino)cyclohexan-1-ol (11k): 82% yield, light brown oil; **IR (ATR):** ν_{max} 3352, 3042, 2929, 2857, 1604, 1588, 1489, 1327, 767 ,692 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.08 (t, J_1 = 7.5 Hz, 1H), 6.63-6.56 (m, 3H), 3.38 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.17-3.10 (m, 1H), 2.28 (s, 3H), 2.16-2.03 (m, 2H), 1.82-1.67 (m, 2H), 1.44-1.34 (m, 1H), 1 .34-1.27 (m, 1H), 1.14-1.03 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 147.1, 139.4, 129.4, 120.1, 115.9, 112.3, 74.4, 60.9, 33.3, 31.5, 25.1, 24.4, 21.7 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 7.3 min, τ_R (major) = 7.7 min, 20% ee.



11l

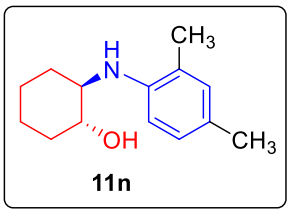
2-((4-methylphenyl) amino) cyclohexan-1-ol (11l): 78% yield, light brown oil, **IR (ATR):** ν_{max} 3383, 2929, 2858, 1617, 1518, 1450, 1302, 807 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.01 (d, J = 8 Hz, 2H), 6.69 (d, J = 8.5 Hz, 2H), 3.37 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.12-3.05 (m, 1H), 2.25 (s, 3H), 2.14-2.05 (m, 2H), 1.80-1.66 (m, 2H), 1.45-1.34 (m, 1H), 1.34-1.26 (m, 2H), 1.11-1.01 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 144.8, 130.0, 128.6, 115.4, 74.4, 61.3, 33.3, 31.4, 25.2, 24.4, 20.5 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 7.03 min, τ_R (minor) = 8.8 min, 10% ee.



11m

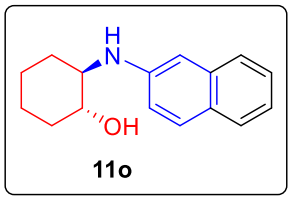
2-((3-chloro-4-fluorophenyl)amino)cyclohexan-1-ol (11m): 85% yield, greenish oil, **IR (ATR):** ν_{max} 3357, 2933, 2859, 1500, 1450, 1064, 1047, 804, 777, 732 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 6.88 (t, J = 9 Hz, 1H), 6.66 (dd, J_1 = 3 Hz, J_2 = 6 Hz, 1H), 6.50-6.45 (m, 1H), 3.32-3.25 (m, 1H), 2.97-2.90 (m, 1H), 2.06-1.98 (m, 2H), 1.75-1.61 (m, 2H), 1.38-1.27 (m, 1H), 1.27-1.19 (m, 2H), 1.04-0.92 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 151.2 (d, J = 237.0 Hz), 144.8 (d, J = 1.8 Hz), 120.9 (d, J = 18.1 Hz), 116.7 (d, J = 21.5 Hz), 115.2, 113.6 (d, J = 5.8 Hz), 74.2, 60.6, 33.4, 31.3, 24.7, 24.2 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 6.9 min, τ_R (minor) = 7.5 min, 22% ee.

2-((2,4-dimethylphenyl)amino)cyclohexan-1-ol (11n): 75% yield, light green oil, **IR (ATR):** ν_{max} 3398, 2929, 2856, 1619, 1510, 1266, 1039, 803, 731 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 6.99-6.92 (m, 2H), 6.74 (d, J = 8, 1H), 3.43 (td, J_1 = 4.5 Hz, J_2 = 9.5 Hz, 1H), 3.21-3.14 (m, 1H), 2.27 (s, 3H), 2.17-2.12 (m, 5H), 1.85-1.70 (m, 2H), 1.50-1.32 (m, 3H), 1.13-1.03 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 143.3, 131.3, 127.5, 127.3, 123.5, 112.3, 74.5, 60.4, 33.3, 31.8, 25.1, 24.4, 20.4, 17.7 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (95 : 5)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 8.7 min, τ_R (minor) = 9.4 min, 6% ee.



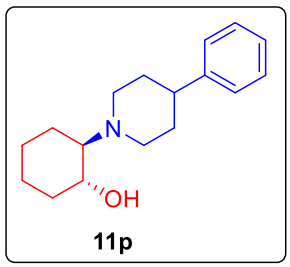
11n

2-(naphthalen-1-ylamino)cyclohexan-1-ol (11o): 80% yield, brown solid, mp: 102-105 ° C ;**IR (ATR):** ν_{max} 3469, 3327, 2927, 2858, 1582, 1528, 1449, 1274, 790, 768, cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.89-7.85 (m, 1H), 7.84-7.80 (m, 1H), 7.51-7.44 (m, 2H), 7.39-7.28 (m, 2H), 6.82 (d, J = 7.5, 1H), 3.58-3.50 (m, 1H), 3.41-3.34 (m, 1H), 2.30-2.22 (m, 1H), 2.21-2.12 (m, 1H), 1.86-1.70 (m, 2H), 1.53-1.30 (m, 3H), 1.18-1.08 (m, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 142.8, 134.6, 128.9, 126.5, 125.9, 125.0, 124.3, 120.1, 118.5, 106.6, 74.6, 60.0, 33.5, 31.3, 25.0, 24.4 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (95 : 5)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 28.3 min, τ_R (minor) = 39.9 min, 14% ee.



11o

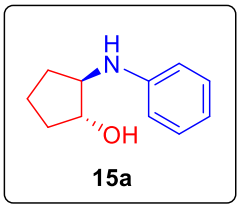
2-(4-phenylpiperidin-1-yl)cyclohexan-1-ol (11a): 80% yield, white solid; **IR (ATR):** ν_{max} 3345, 2959, 2923, 2853, 1600, 1468, 1275, 1269, 760, 750 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.33-7.27 (m, 2H), 7.25-7.17 (m, 3H), 3.41 (dt, J_1 = 4 Hz, J_2 = 9.5 Hz, 1H), 2.95 (d, J = 11 Hz, 1H), 2.80-2.70 (m, 2H), 2.50 (tt, J_1 = 4 Hz, J_2 = 12, 1H), 2.30-2.20 (m, 2H), 2.17- 2.12 (m, 1H), 1.92-1.76 (m, 5H), 1.75-1.61 (m, 2H), 1.25-1.16 (m, 4H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :** δ 146.4, 128.5, 127.0, 126.3, 70.8, 68.8, 53.6, 45.6, 43.1, 34.5, 34.1, 33.5, 25.8, 24.3, 22.4 ppm. The ee was determined by HPLC using Chiralpak AS-H [hexane/iPrOH (97 : 3)]; flow rate 0.5 mL min^{-1}; τ_R (major) = 19.1 min, τ_R (minor) = 20.8 min, 16% ee.



11p

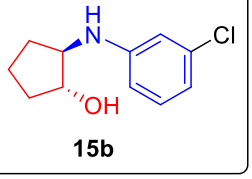
2-(phenylamino)cyclopentan-1-ol (15a): 85% yield, purple oil; **IR (ATR):** ν_{max} 3345, 2956, 2872, 1601, 1498, 1316, 978, 748, 693 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.21-7.16 (m, 2H), 6.77-6.68 (m, 3H), 4.10 (q, J = 4.5 Hz, 1H), 3.64-3.59 (m, 1H), 2.30-2.22 (m, 1H), 2.04-1.95 (m,

1H), 1.83-1.69 (m, 2H), 1.88-1.70 (m, 1H), 1.48-1.39 (m, 1H) ppm; **^{13}C NMR (125 MHz, CDCl$_3$) :**δ 147.3, 129.5, 118.2, 113.9, 76.9, 62.7, 33.0, 31.1, 21.1 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 9.8 min, τ_R (minor) = 11.3 min, 2% ee.



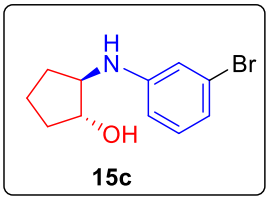
15a

2-((3-chlorophenyl) amino) cyclopentan-1-ol (15b): 82% yield, dark maroon oil, **IR (ATR):** ν_{max} 3347, 2929, 2874, 1596, 1500, 1481, 1326, 987, 762, 682 cm^{-1}; **^{1}H NMR (500 MHz, CDCl$_3$) :** δ 7.07 (t, J = 7.5 Hz, 1H), 6.69-6.64 (m, 2H), 6.53 (ddd, J_1 = 0.5, J_2 = 2, J_3 = 8 Hz, 1H), 4.05 (q, J = 4.5, 1H), 3.60-3.54 (m, 1H), 2.31-2.22 (m, 1H), 2.02-1.94 (m, 1H), 1.88-1.69 (m, 2H), 1.69-1.60 (m, 1H), 1.44-1.36 (m, 1H) ppm; **^{13}C NMR (125 MHz, CDCl$_3$) :**δ 148.8, 135.2, 130.4, 117.6, 113.2, 111.9, 76.9, 62.2, 33.1, 31.2, 21.1 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 10.4 min, τ_R (minor) = 11.4 min, 2% ee.



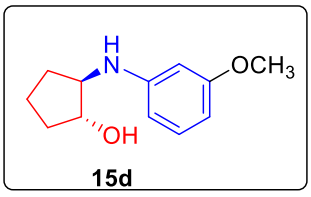
15b

2-((3-bromophenyl) amino) cyclopentan-1-ol (15c): 79% yield, dark red oil, **IR (ATR):** ν_{max} 3337, 2926, 2871, 1594, 1498, 1479, 1325, 984, 760, 682 cm^{-1}; **^{1}H NMR (500 MHz, CDCl$_3$) :** δ 7.01 (t, J = 8 Hz, 1H), 6.85-6.82 (m, 2H), 6.66 (ddd, J_1 = 1 Hz, J_2 = 2.5, J_3 = 7.5 Hz, 1H), 4.07 (q, J = 4.5 Hz, 1H), 3.59-3.54 (m, 1H), 2.30-2.21 (m, 1H), 2.03-1.94 (m, 1H), 1.88-1.70 (m, 2H), 1.69-1.60 (m, 1H), 1.46-1.36 (m, 1H) ppm; **^{13}C NMR (125 MHz, CDCl$_3$) :**δ 148.7, 130.7, 123.4, 120.9, 116.4, 112.5, 76.9, 62.5, 33.1, 31.1, 21.1 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 10.1 min, τ_R (minor) = 11.2 min, 2% ee.



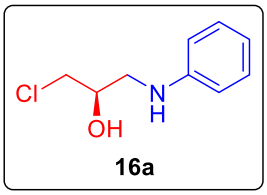
15c

2-((3-methoxyphenyl) amino) cyclopentan-1-ol (15d): 75% yield, dark brown oil, **IR (ATR):** ν_{max} 3353, 2955, 2835, 1600, 1495, 1261, 1209, 1160, 1038, 758 cm^{-1}; **^{1}H NMR (500 MHz, CDCl$_3$) :** δ 7.09 (t, J = 8 Hz, 1H), 6.34-6.28 (m, 3H), 4.10 (q, J = 4.5 Hz, 1H), 3.77 (s, 3H), 3.62-3. (m, 1H), 2.29-2.21 (m, 1H), 2.03-1.95 (m, 1H), 1.88-1.69 (m, 2H), 1.68-1.60 (m, 1H), 1.48-1.40 (m, 1H) ppm; **^{13}C NMR (125 MHz, CDCl$_3$) :**δ 161.0, 148.5, 130.2, 107.1, 103.6, 100.1, 76.9, 62.9, 55.3, 33.0, 31.1, 21.1 ppm. The ee was determined by HPLC using Chiralpak IB



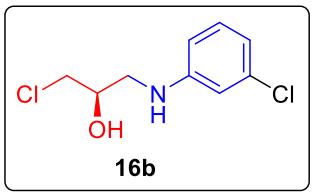
15d

[hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 14.6 min, τ_R (minor) = 17.8 min, 2% ee.



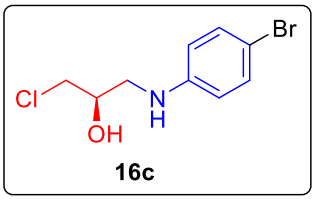
16a

1-chloro-3-(phenylamino)propan-2-ol (16a): 87% yield, dark purple oil, **IR (ATR):** ν_{max} 3309, 2920, 1599, 1584, 1504, 1324, 748, 692 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.24-7.19 (m, 2H), 6.84-6.80 (m, 1H), 6.77 (dd, J_1 = 1 Hz, J_2 = 8.5 Hz, 2H), 4.164.07 (m, 1H), 3.65 (qd, J_1 = 1 Hz, J_2 = 11.5 Hz, 2H), 3.40 (dd, J_1 = 4.5 Hz, J_2 = 13 Hz, 1H), 3.26 (dd, J_1 = 7.5 Hz, J_2 = 13 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 146.6, 129.6, 119.6, 114.5, 69.6, 48.3, 47.6 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 19.4 min, τ_R (major) = 20.4 min, 8% ee.



16b

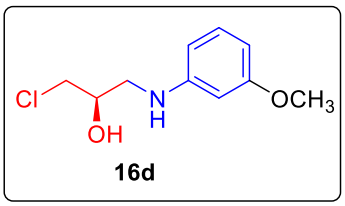
1-chloro-3-((3-chlorophenyl)amino)propan-2-ol (16b): 90% yield, greenish brown oil, **IR (ATR):** ν_{max} 3332, 2917, 1596, 1504, 1484, 1250, 841, 763, 681 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.09 (t, J = 8 Hz, 1H), 6.71 (dd, J_1 = 1 Hz, J_2 = 8 Hz, 1H), 6.63 (s, 1H), 6.52 (dd, J_1 = 1.5 Hz, J_2 = 8 Hz, 1H), 4.11-4.03 (m, 1H), 3.66 (ddd, J_1 = 4.5 Hz, J_2 = 11.5 Hz, J_3 = 28 Hz, 2H), 3.36 (dd, J_1 = 4 Hz, J_2 = 13 Hz, 1H), 3.22 (dd, J_1 = 7 Hz, J_2 = 13 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 149.0, 135.3, 130.5, 118.2, 113.1, 111.8, 69.9, 47.8, 47.0 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90: 10)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 36.8 min, τ_R (major) = 40.1 min, 28% ee.



16c

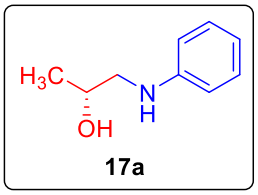
1-((4-bromophenyl)amino)-3-chloropropan-2-ol (16c): 88% yield, brown oil; **IR (ATR):** ν_{max} 2916, 2850, 1730, 1594, 1492, 1261, 1179, 812, 750 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.29 (dt, J_1 = 3 Hz, J_2 = 10 Hz, 2H), 6.63 (dt, J_1 = 3 Hz, J_2 = 9.5 Hz, 2H), 4.14-4.08 (m, 1H), 3.65 (qd, J_1 = 5 Hz, J_2 = 11.5 Hz, 2H), 3.36 (dd, J_1 = 4 Hz, J_2 = 13 Hz, 1H), 3.22 (dd, J_1 = 7.5 Hz, J_2 = 13.5 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 145.6, 132.2, 115.8, 111.2, 69.5, 48.0, 47.4 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 10.9 min, τ_R (minor) = 11.5 min, 26% ee.

1-((3-methoxyphenyl)amino)-3-chloropropan-2-ol (16d): 80% yield, green oil, **IR (ATR):** ν_{max} 3308, 2919, 1600, 1584, 1504, 1324, 1171, 1074, 748, 692 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.11 (t, J = 8 Hz, 1H), 6.40-6.27 (m, 3H), 4.16-4.08 (m, 1H), 3.77 (s, 3H), 3.65 (qd, J_1 = 5 Hz, J_2 = 11.5 Hz, 2H), 3.39 (dd, J_1 = 4.5 Hz, J_2 = 13.5 Hz, 1H), 3.25 (dd, J_1 = 7.5 Hz, J_2 = 13.5 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 161.0, 130.4, 107.2, 104.5, 100.3, 69.7, 55.3, 47.7, 29.8 ppm. The ee was determined by HPLC using Chiralpak IB [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min−1 ;τ_R (major)= 21.0 min, τ_R (minor) = 24.1 min, 18% ee.



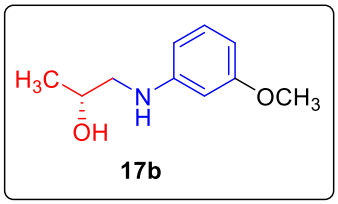
16d

1-(phenylamino)propan-2-ol (17a): 88% yield, reddish brown oil, **IR (ATR):** ν_{max} 3341, 3052, 2969, 2929, 1602, 1499, 1460, 1317, 1072, cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.25 (t, J = 7 Hz, 2H), 6.80 (t, J = 6.5 Hz, 1H), 6.70 (d, J = 8 Hz, 2H), 4.04 (s, 1H), 3.29-3.21 (m, 1H), 3.07-2.98 (m, 1H), 1.29 (d, J = 6 Hz, 1H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 148.2, 129.3, 117.9, 113.4, 66.4, 51.7, 20.9 ppm. The ee was determined by HPLC using phenomenex Amylose-1 [hexane/iPrOH (80 : 20)]; flow rate 1.0 mL min^{-1}; τ_R (minor) = 8.9 min, τ_R (major) = 9.9 min, 38% ee.



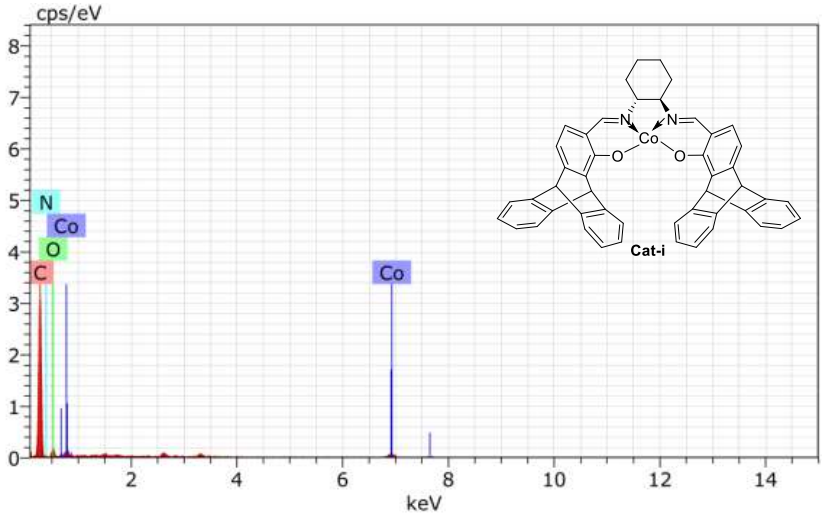
17a

1-((3-methoxyphenyl)amino)propan-2-ol (17b): 75% yield, dark brown solid; **IR (ATR):** ν_{max} 3341, 2958, 2923, 2853, 1611, 1461, 1278, 1261, 764, 750 cm^{-1}; **^{1}H NMR (500 MHz, $CDCl_3$) :** δ 7.08 (t, J = 8.5 Hz, 1H), 6.30 (dd, J_1 = 2 Hz, J_2 = 8 Hz, 1H), 6.25 (dd, J_1 = 1.5 Hz, J_2 = 8 Hz, 1H), 6.20 (t, J = 2 Hz, 1H), 4.03-3.94 (m, 1H), 3.75 (s, 3H), 3.17 (dd, J_1 = 3 Hz, J_2 = 13 Hz, 1H), 2.95 (dd, J_1 = 8.5 Hz, J_2 = 13 Hz, 1H), 1.22 (d, J = 6.5 Hz, 3H) ppm; **^{13}C NMR (125 MHz, $CDCl_3$) :**δ 160.8, 149.7, 130.1, 106.4, 103.4, 99.3, 66.3, 55.1, 51.6, 20.8 ppm. The ee was determined by HPLC using Chiralpak IA [hexane/iPrOH (90 : 10)]; flow rate 1.0 mL min^{-1}; τ_R (major) = 17.7 min, τ_R (minor) = 19.8 min, 46% ee.



17b

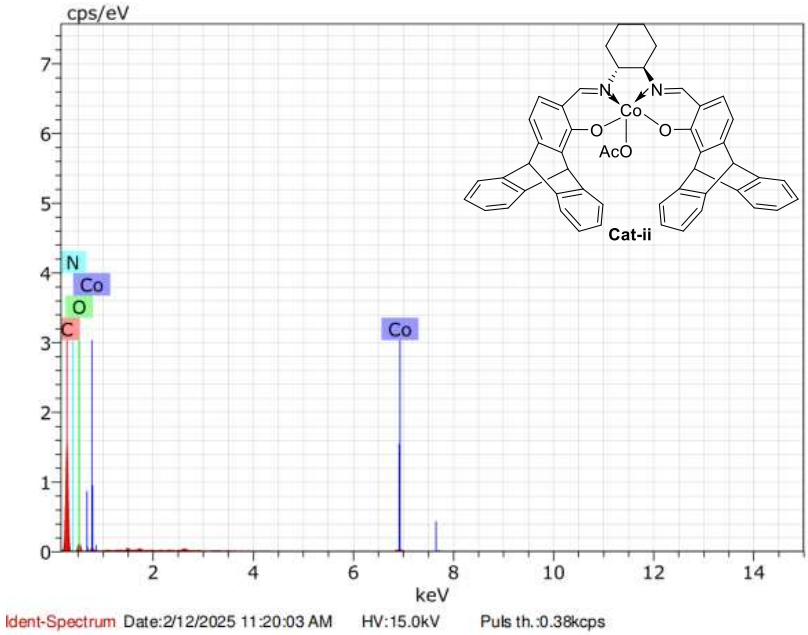
1.4. EDS Profile of Catalysts:



Spectrum: test 20980

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	74.18	74.18	82.14	33.07
Oxygen	K-series	11.43	11.43	9.50	10.39
Cobalt	K-series	7.32	7.32	1.65	1.61
Nitrogen	K-series	7.07	7.07	6.71	11.24
	Total:	100.00	100.00	100.00	

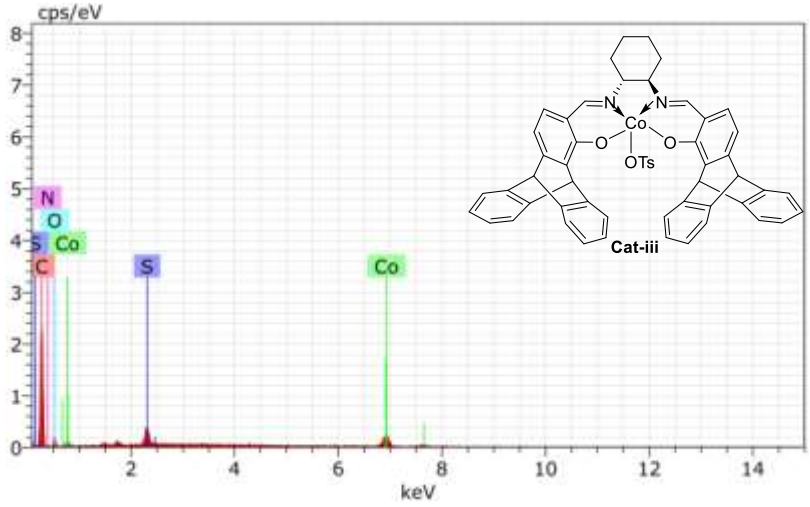
Figure S1: EDS spectrum of TpCo(II)(**Cat-i**).



Spectrum: test 20881

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	75.27	75.27	83.24	32.38
Oxygen	K-series	14.23	14.23	11.82	11.10
Cobalt	K-series	6.93	6.93	1.56	1.44
Nitrogen	K-series	3.57	3.57	3.38	6.96
	Total:	100.00	100.00	100.00	

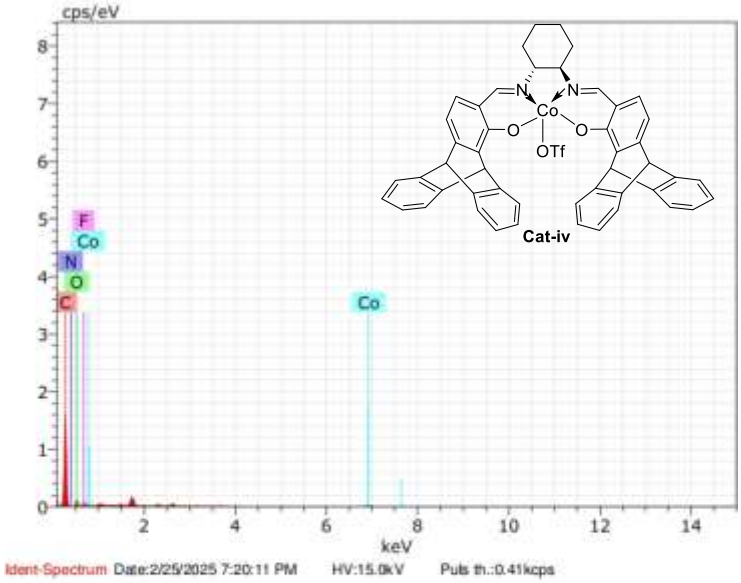
Figure S2: EDS spectrum of TpCo(III)-OAc (**Cat-ii**)



Spectrum: test 22138

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	37.70	65.35	84.09	17.00
Cobalt	K-series	13.63	23.62	6.19	1.76
Sulfur	K-series	1.52	2.63	1.27	0.29
Oxygen	K-series	3.49	6.05	5.85	3.16
Nitrogen	K-series	1.36	2.35	2.59	2.66
Total:		57.70	100.00	100.00	

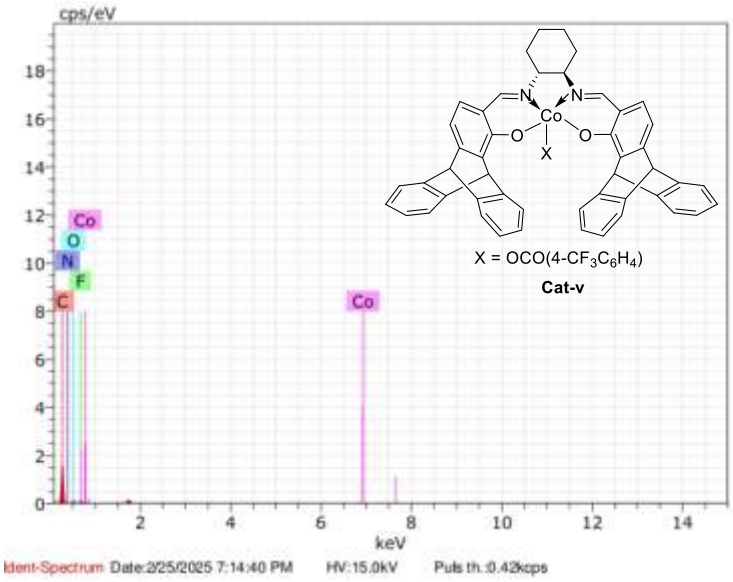
Figure S3: EDS spectrum of TpCo(III)-OTs (**Cat-iii**).



Spectrum: test 20994

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	72.04	72.04	79.88	30.82
Oxygen	K-series	14.25	14.25	11.86	10.67
Nitrogen	K-series	3.97	3.97	3.77	6.98
Cobalt	K-series	4.93	4.93	1.11	1.14
Fluorine	K-series	4.82	4.82	3.38	4.43
Total:		100.00	100.00	100.00	

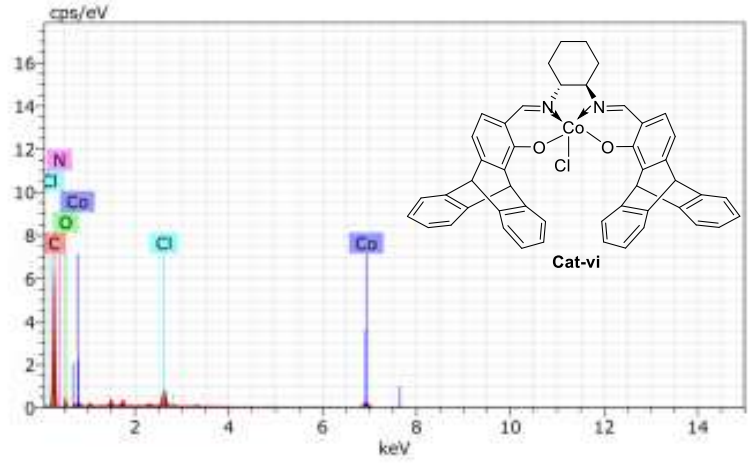
Figure S4: EDS spectrum of TpCo(III)-OTf (**Cat-iv**).



Spectrum: test 20992

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	69.49	69.49	78.74	29.57
Fluorine	K-series	6.68	6.68	4.79	5.23
Nitrogen	K-series	3.89	3.89	3.78	6.48
Oxygen	K-series	13.06	13.06	11.11	9.59
Cobalt	K-series	6.87	6.87	1.59	1.32
Total:		100.00	100.00	100.00	

Figure S5: EDS spectrum of TpCo(III)-OCO(4-$CF_3C_6H_4$) (**Cat-v**).



Spectrum: test 22641

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	57.18	73.08	84.56	23.28
Oxygen	K-series	7.08	9.05	7.86	5.00
Cobalt	K-series	8.29	10.60	2.50	1.19
Chlorine	K-series	2.78	3.56	1.39	0.41
Nitrogen	K-series	2.90	3.71	3.68	4.02
Total:		78.24	100.00	100.00	

Figure S6: EDS spectrum of TpCo(III)-Cl (**Cat-vi**).

1.5. PXRD graphs of ligand 8 and cobalt catalysts.

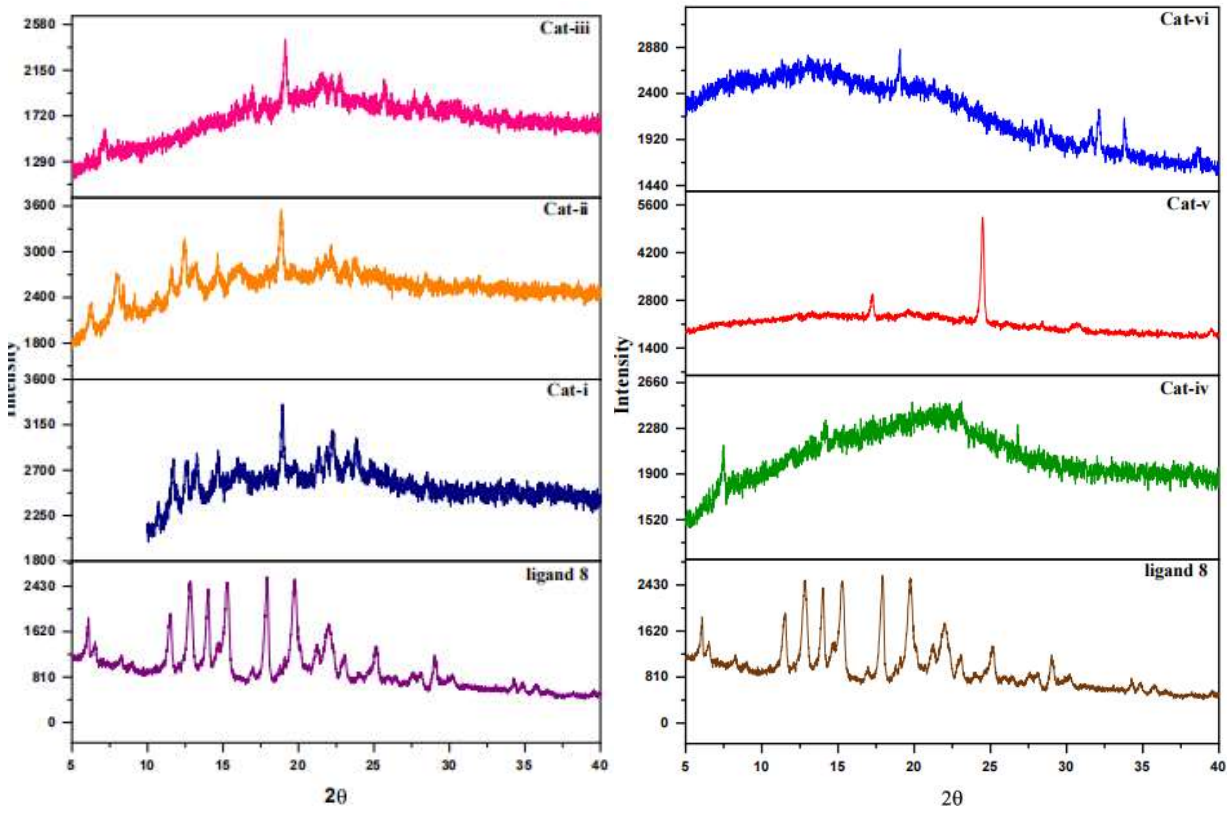
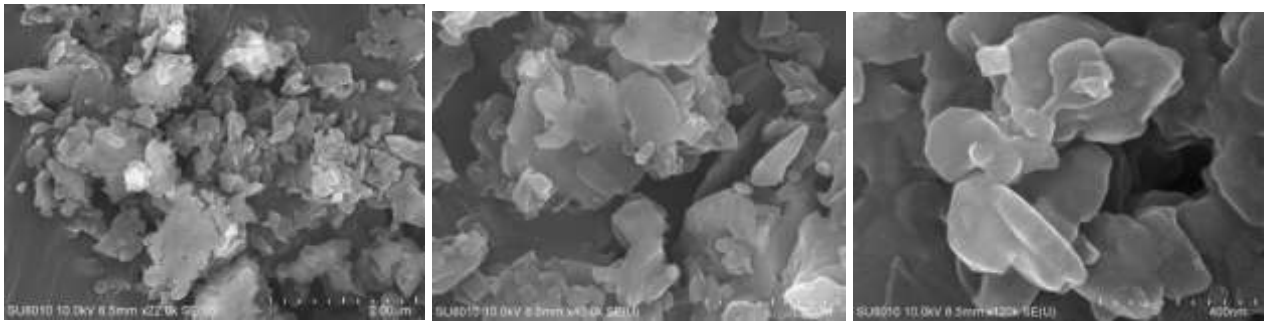


Figure S7: Comparison of PXRD graphs of ligand 8 **a)** with Cat-i - Cat-iii, **b)** Cat-iv - Cat-vi.

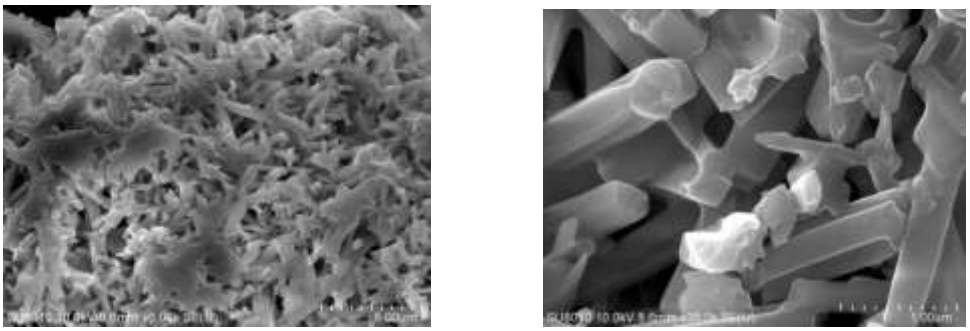
PXRD analysis was carried out for ligand 8 and all catalysts (Cat-i to Cat-Vi) to understand their structural characteristics. Ligand 8 exhibits a crystalline pattern, with several well-defined diffraction peaks in the 10-30° range. Prominent reflections at 11.5°, 12.9°, 13.9°, 15.3°, 17.8°, 19.7°, and 21.9° clearly indicate its crystalline nature. In Cat-i, these characteristic peaks become less intense and slightly shifted to 11.7°, 12.6°, 13.3°, 14.6°, and 18.9°, reflecting a transition to a semi-crystalline phase upon coordination with cobalt, thereby confirming successful complex formation. The PXRD profiles of Cat-ii to Cat-vi also display reduced peak intensity and partial broadening, suggesting semi-crystalline to moderately amorphous structural features across the series.

1.6. FESEM images of ligand 8, Cat-i and Cat-ii:



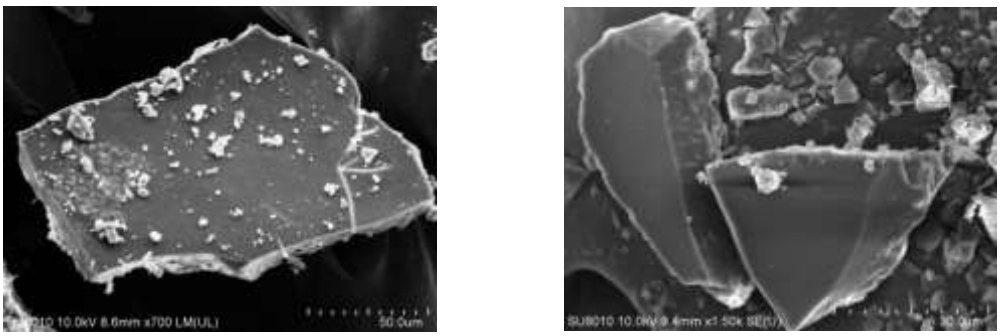
a) **b)** **c)**

Figure S8: FESEM images of triptycene ligand **8** at different scales **a)** at 2µm, **b)** at 1 µm, **c)** at 400nm.



a) **b)**

Figure S9: FESEM images of cat-i at different scales **a)** at 5µm **b)** at 1µm**.**



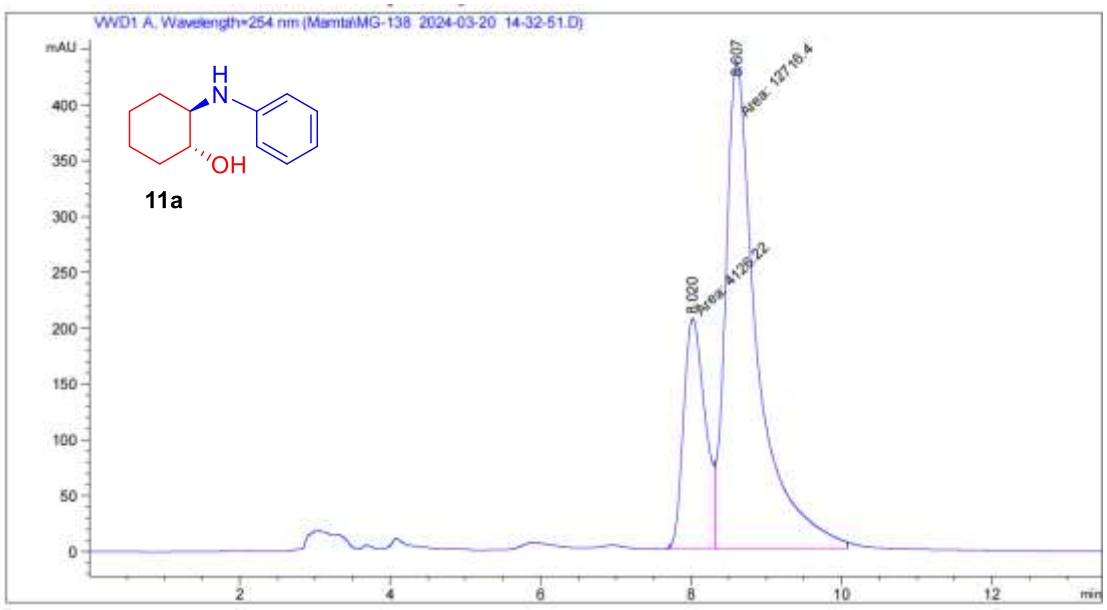
a) **b)**

Figure S10: FESEM images of cat-ii at different scales **a)** at 50µm **b)** at 30µm**.**

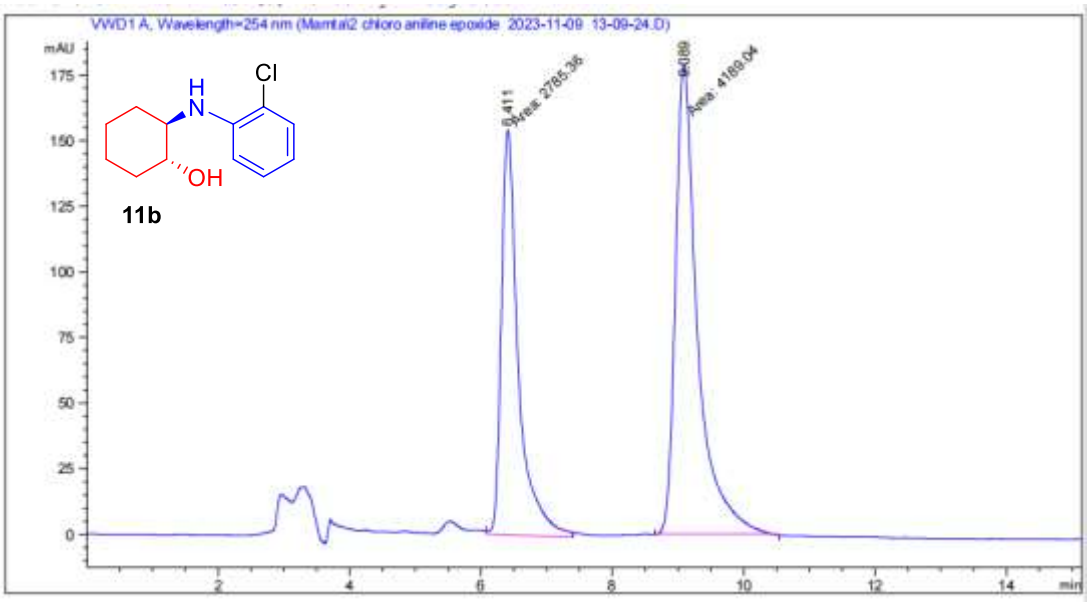
To study the structural morphology of the triptycene ligand **8** and the changes after coordination with the cobalt metal, SEM images of ligand **8, cat-i** and **cat-ii** were recorded and compared. The images of ligand 8 (figure S8) showed irregular, flaky particles that remain widely separated, indicating minimal interaction. In cat-i, the particles become closer and formed compact clusters (figure S9). The surface becomes denser, and the particles are no longer isolated, suggesting that cobalt coordination increased

the attraction between the molecules and lead to aggregated structures. After introducing the axial ligand to obtain cat-ii, material developed into thin, sheet-like layers (figure S10). These sheets are broad, slightly overlapping, and give a clear indication of a more organized and continuous structure.

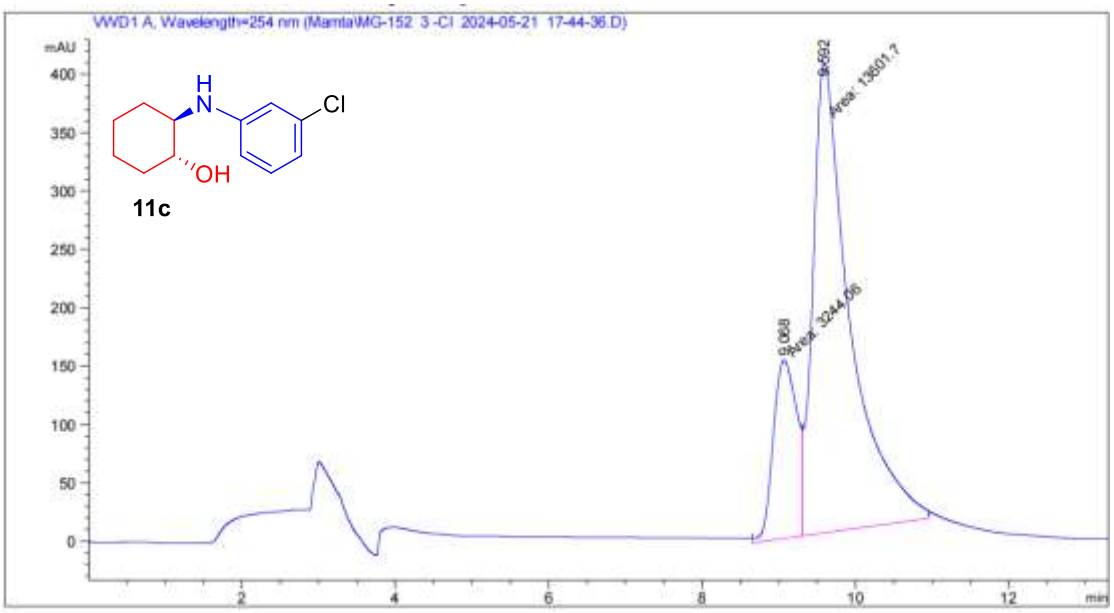
2. HPLC Chromatograms:



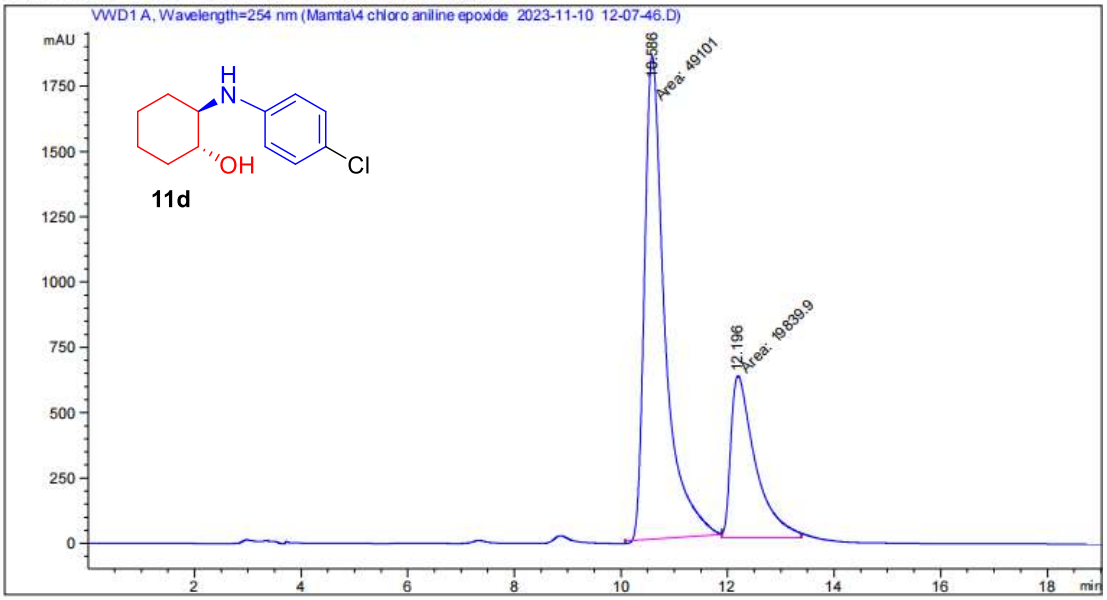
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.020	MF	0.3338	4126.21729	206.03537	24.4987
2	8.607	FM	0.4877	1.27164e4	434.57065	75.5013



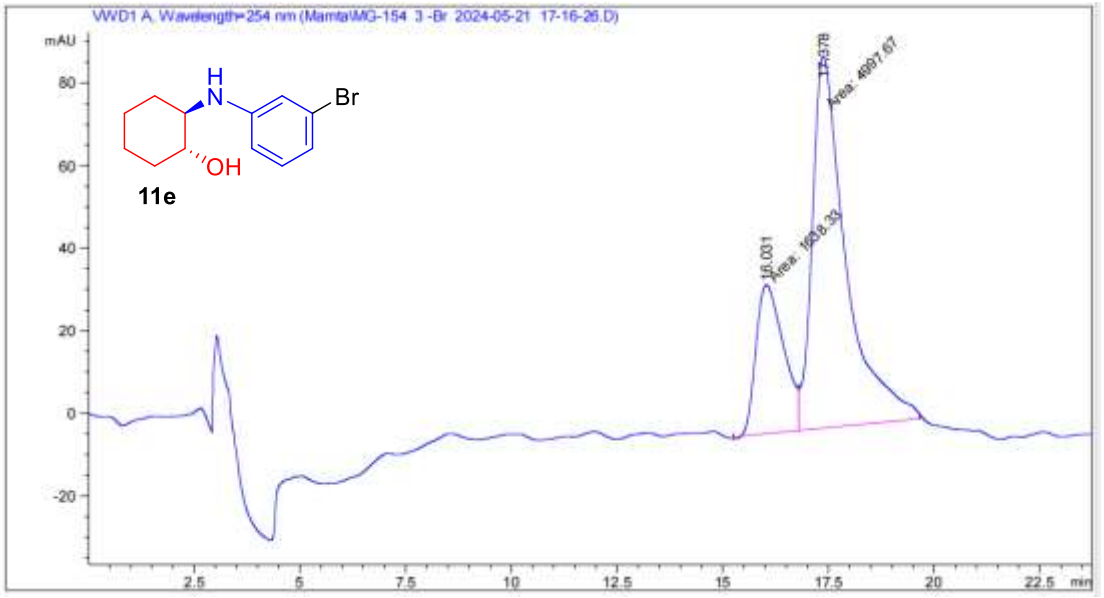
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.411	MM	0.3008	2785.35938	154.33278	39.9369
2	9.089	MM	0.3901	4189.03955	178.97623	60.0631



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.068	MF	0.3547	3244.05737	152.42877	19.2574
2	9.592	FM	0.5640	1.36017e4	401.97217	80.7426



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.586	MM	0.4427	4.91010e4	1848.70093	71.2219
2	12.196	MM	0.5334	1.98399e4	619.96796	28.7781



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
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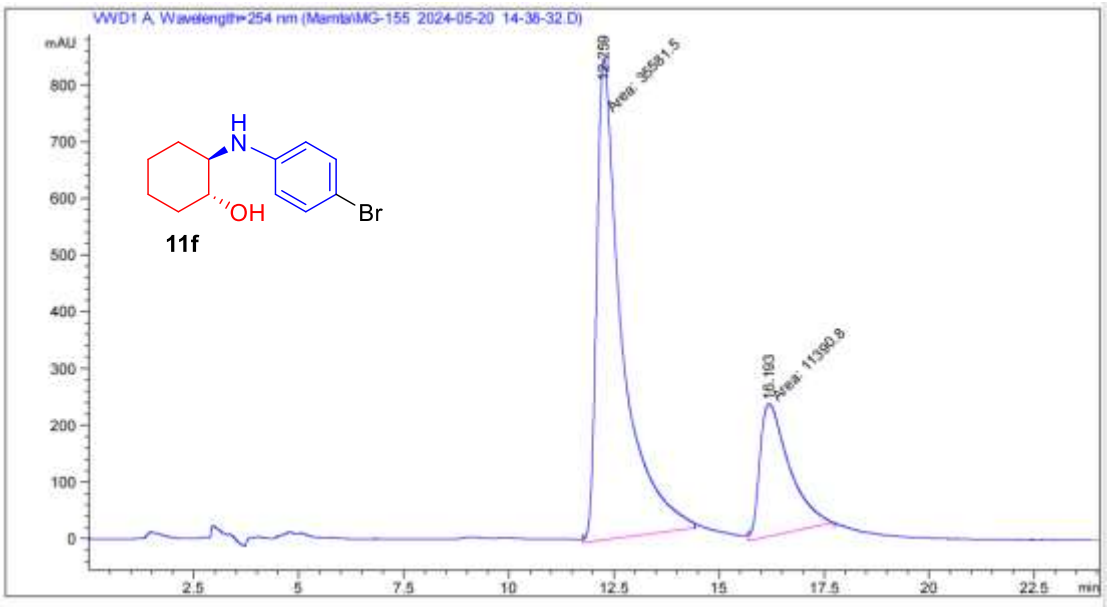
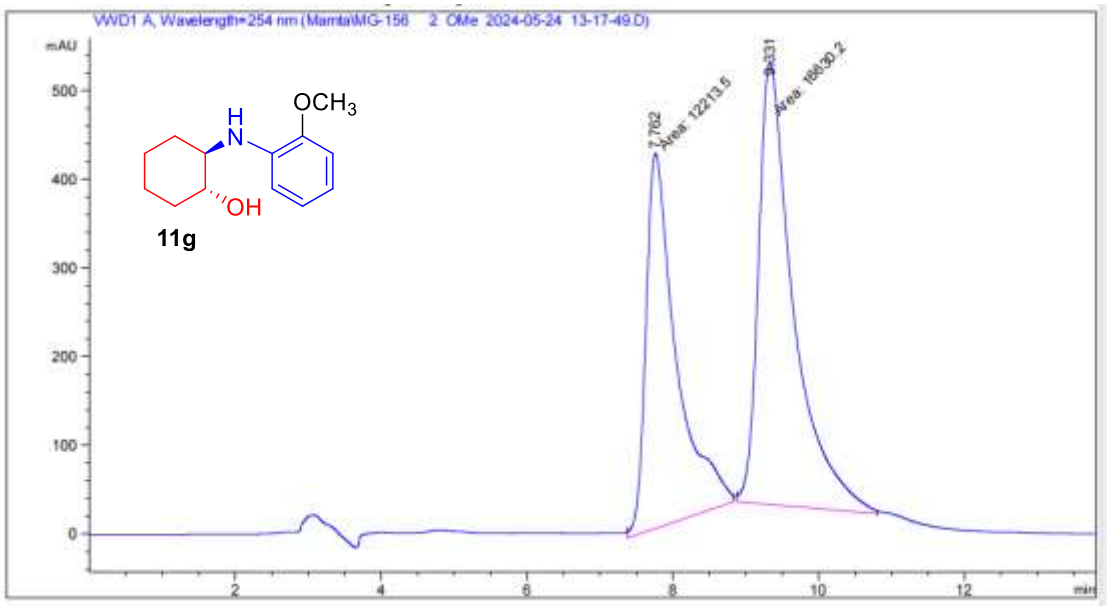
2	17.378	FM	0.9255	4997.66602	90.00153	75.3115



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.259	MM	0.6997	3.55815e4	847.57281	75.7499
2	16.193	MM	0.8150	1.13908e4	232.95294	24.2501



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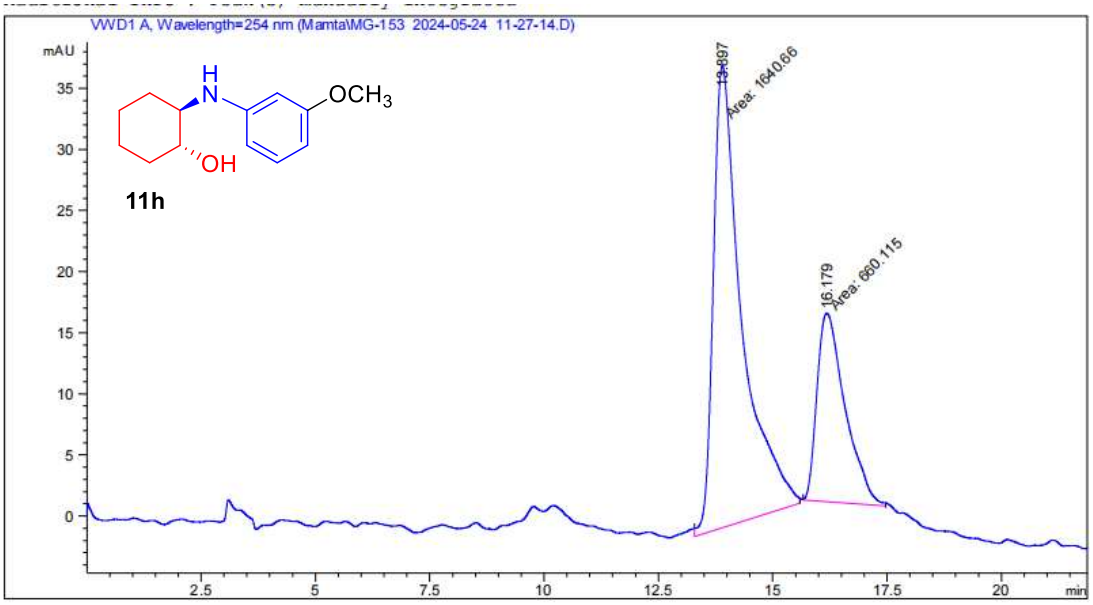
1	7.762	MM	0.4812	1.22135e4	423.02264	42.3439
2	9.331	MM	0.5558	1.66302e4	498.67950	57.6561



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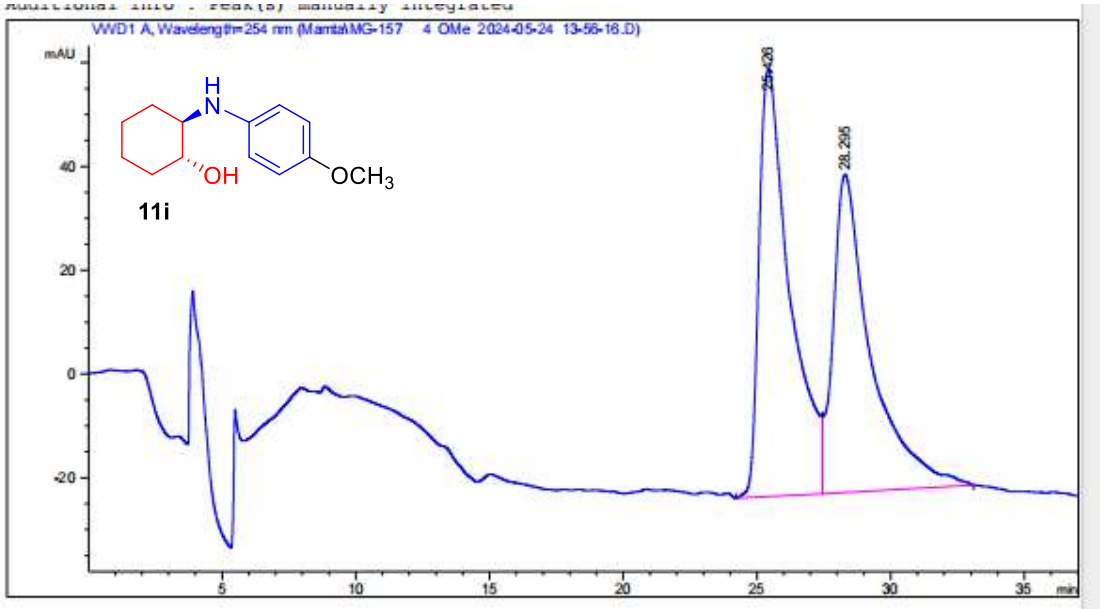
1	13.897	MM	0.7240	1640.65881	37.77045	71.3090
2	16.179	MM	0.7118	660.11511	15.45560	28.6910



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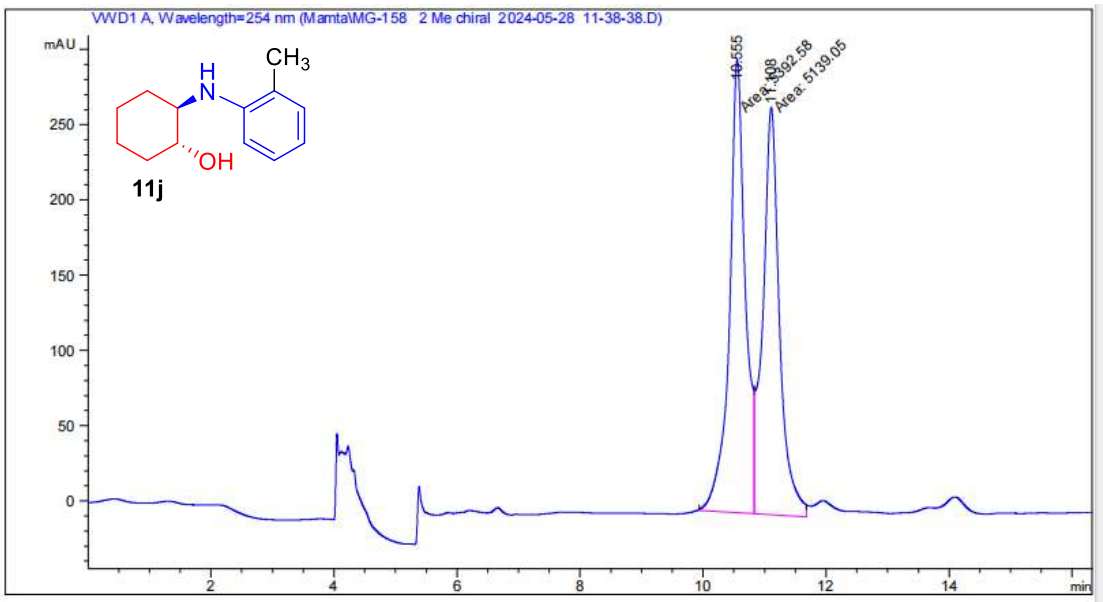
1	25.426	BV	0.9768	6614.34277	82.32774	52.3130
2	28.295	VV R	1.1520	6029.43848	61.27296	47.6870



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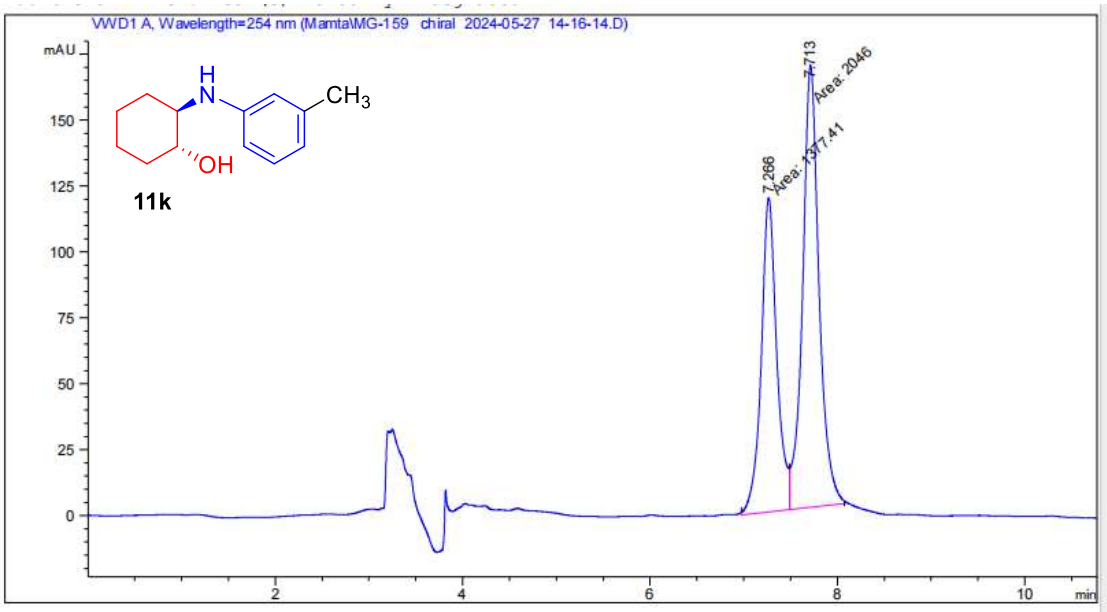
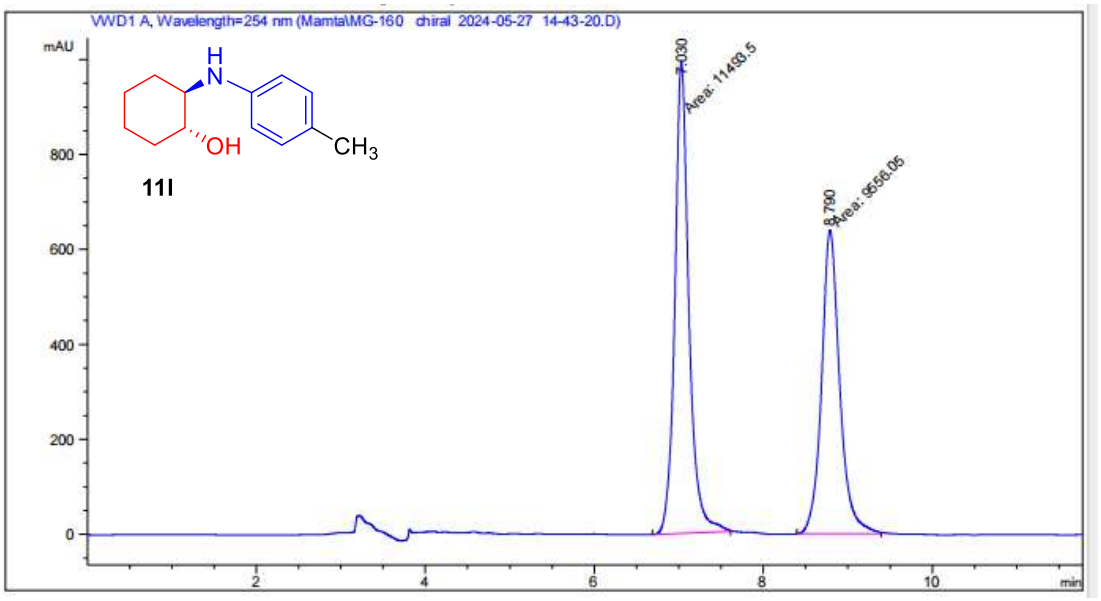
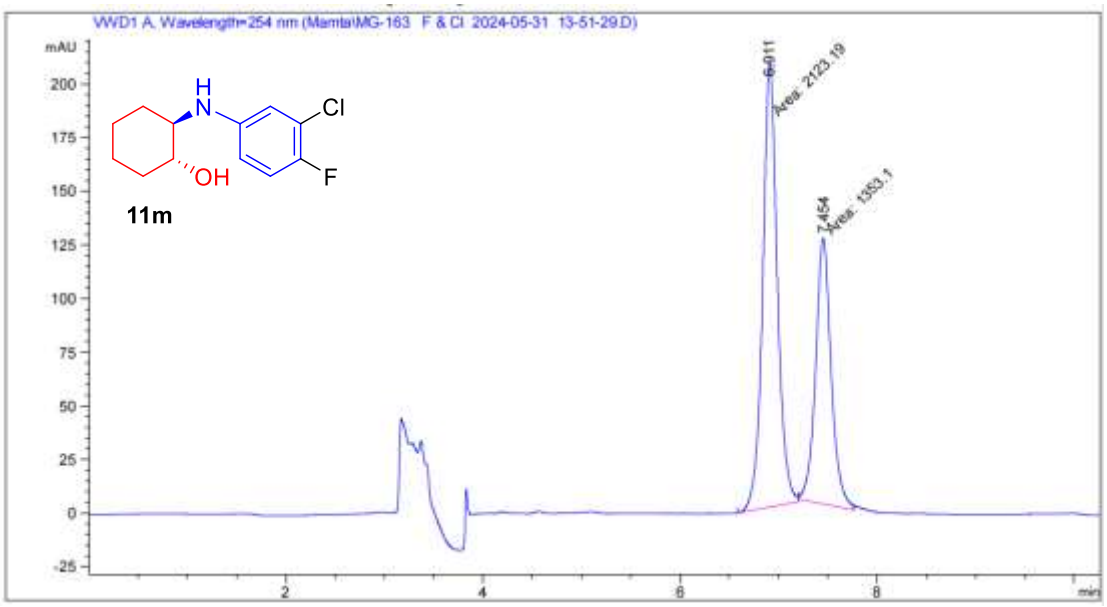
1	10.555	MF	0.2984	5392.57666	301.18539	51.2036
2	11.108	FM	0.3164	5139.05029	270.67279	48.7964



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.266	MF	0.1927	1377.40881	119.12554	40.2350
2	7.713	FM	0.2034	2045.99805	167.63611	59.7650



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.030	MM	0.1927	1.14935e4	993.99231	54.6021
2	8.790	MM	0.2490	9556.04590	639.69452	45.3979



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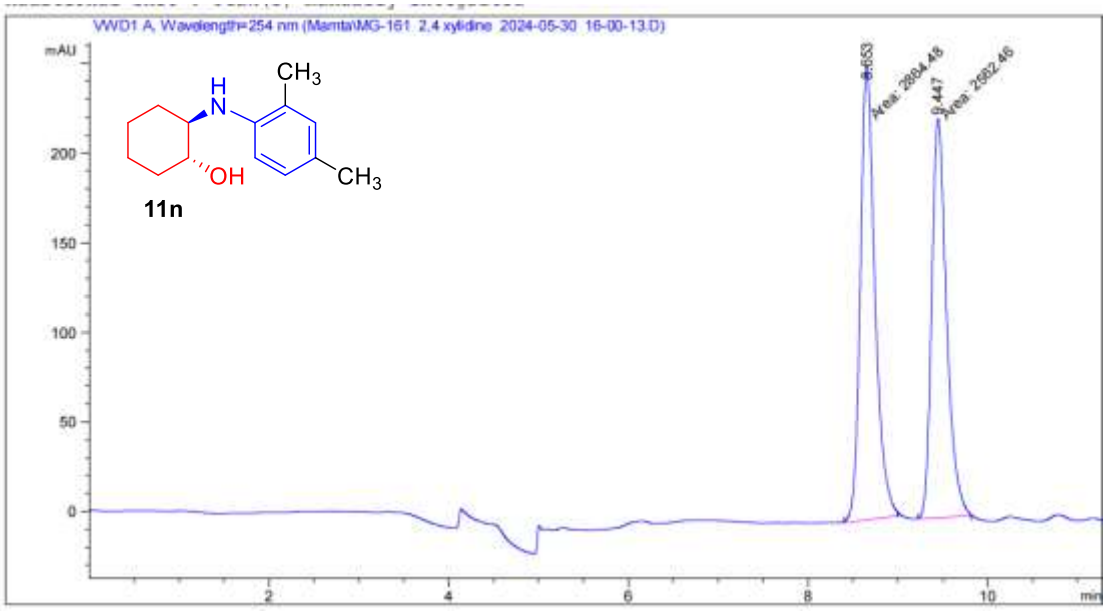
1	6.911	MM	0.1711	2123.18530	206.78302	61.0762
2	7.454	MM	0.1821	1353.10229	123.81221	38.9238



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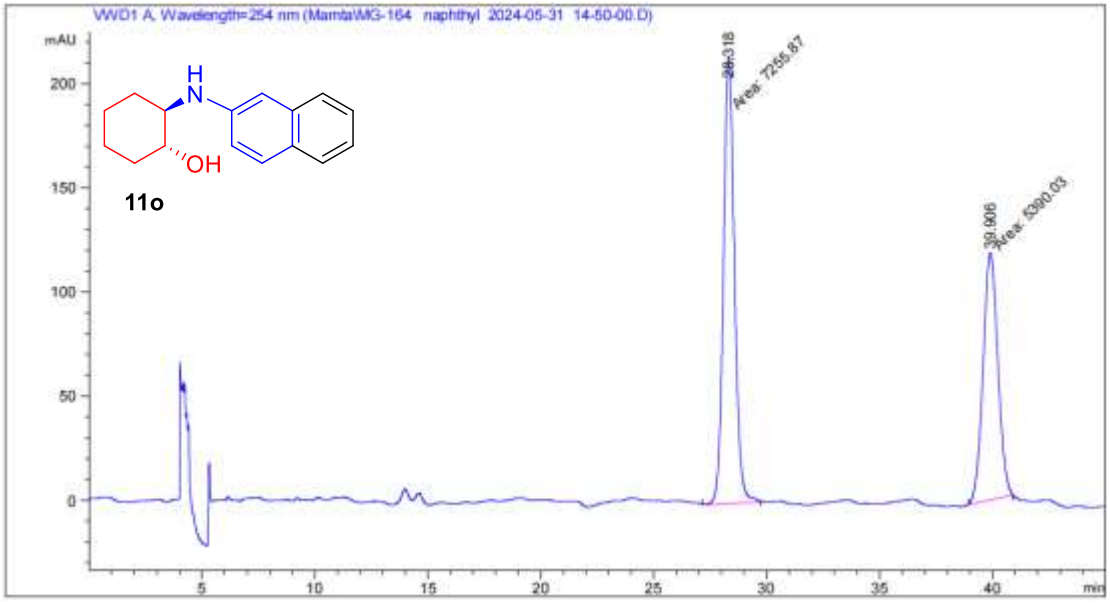
1	8.653	MM	0.1886	2864.48022	253.18053	52.7826
2	9.447	MM	0.1919	2562.45874	222.52878	47.2174



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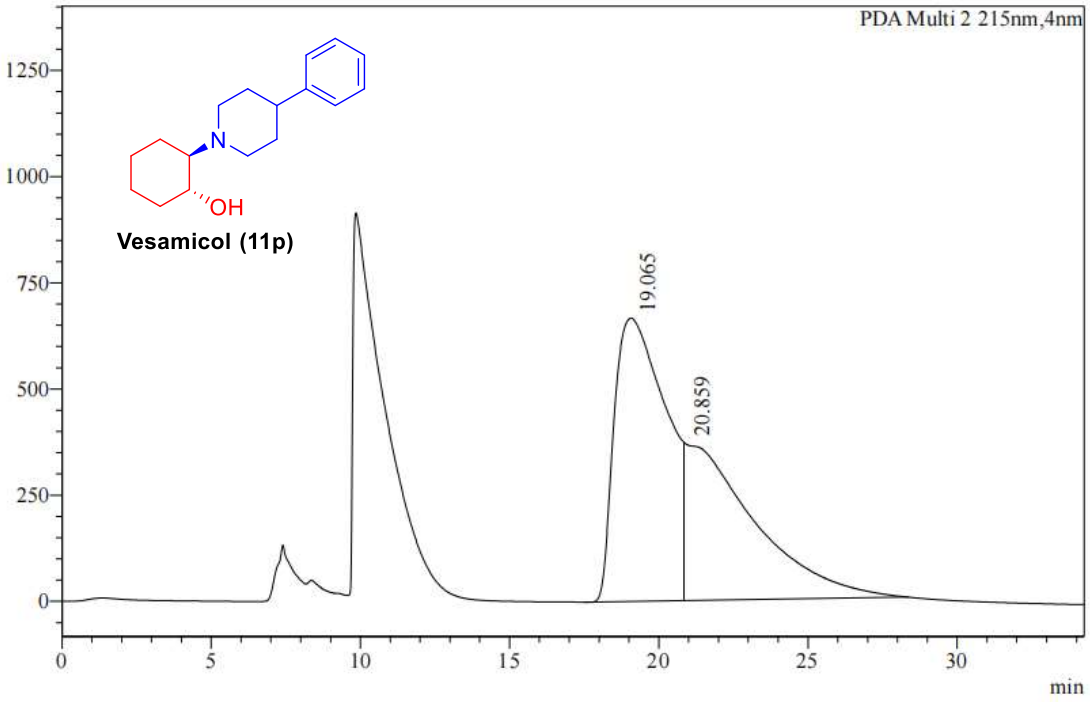
2	39.906	MM	0.7582	5390.02637	118.48366	42.6227



Peak Table

PDA Ch2 215nm

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1	19.065	80913137	666941	57.502
2	20.859	59799588	371948	42.498
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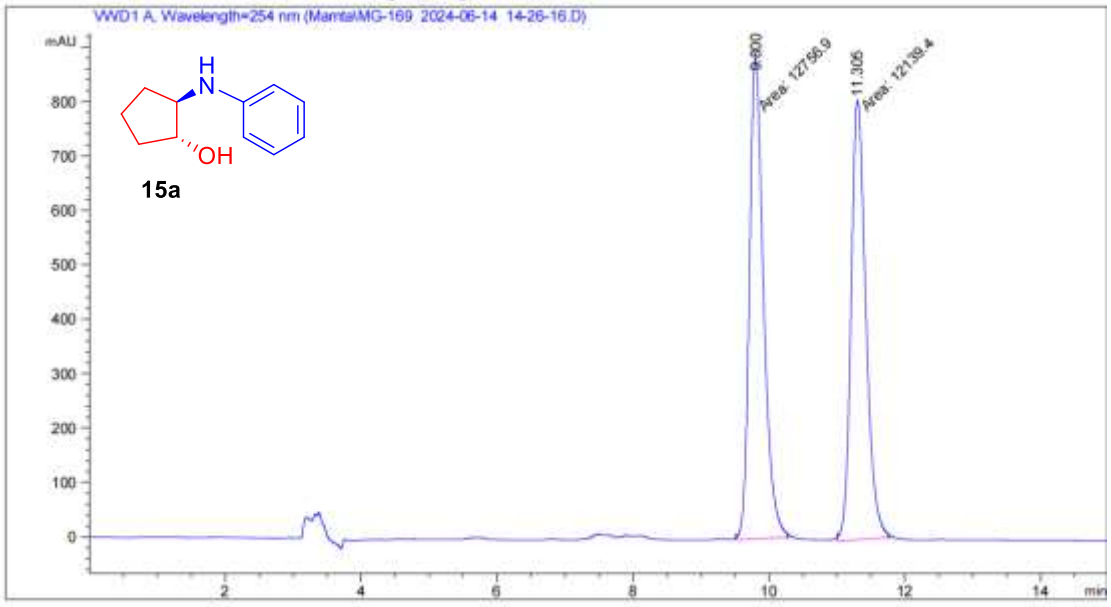
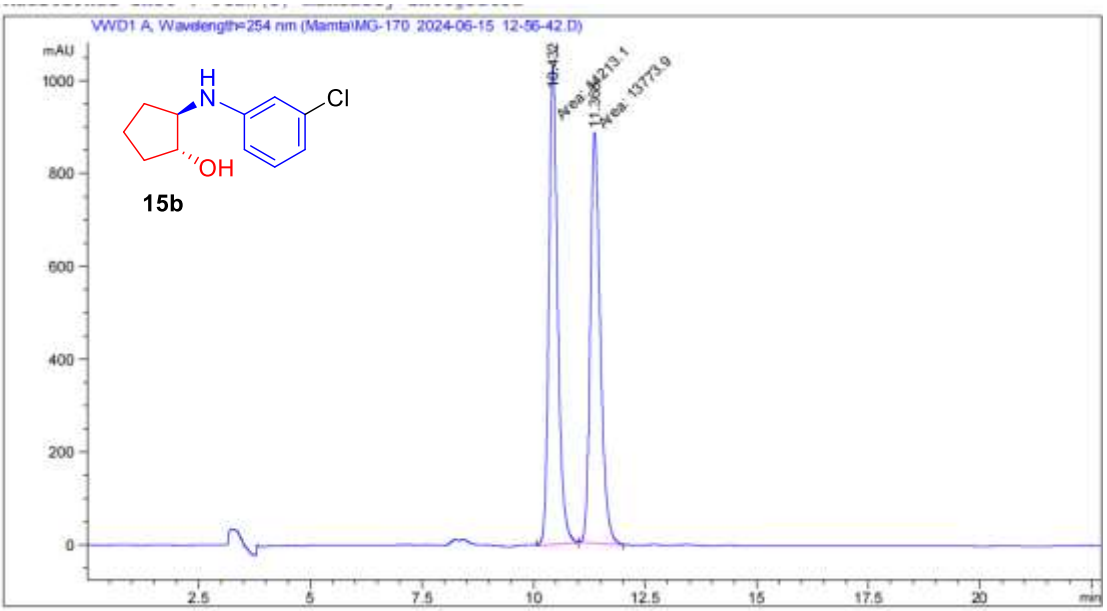
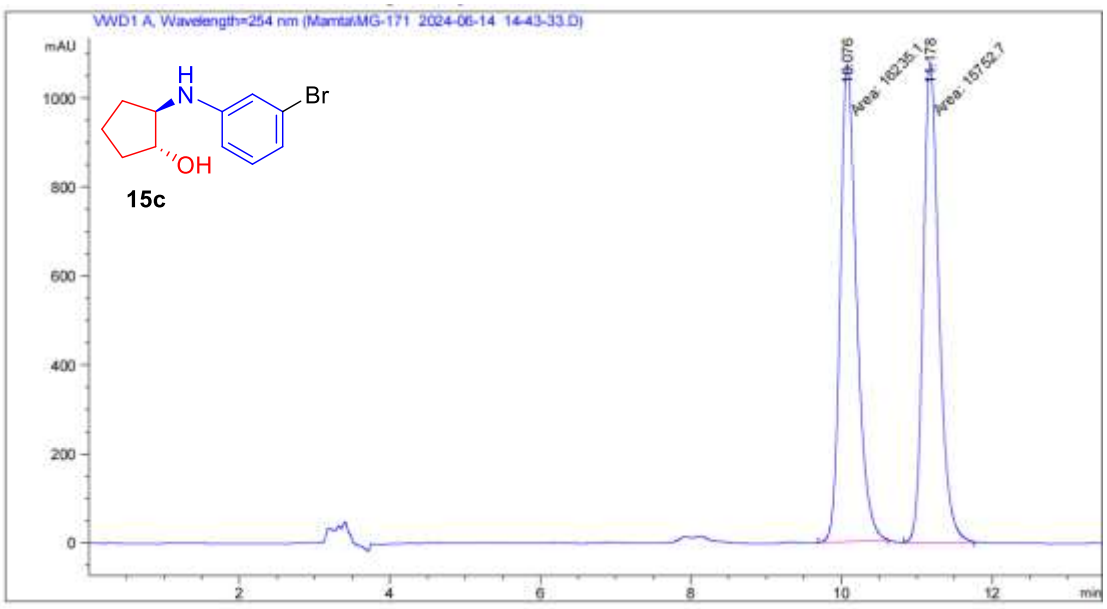


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.800	MM	0.2404	1.27569e4	884.34229	51.2402
2	11.305	MM	0.2507	1.21394e4	807.07159	48.7598



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.432	MM	0.2300	1.42131e4	1029.71936	50.7845
2	11.368	MM	0.2594	1.37739e4	885.14673	49.2155



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1	10.076	MM	0.2523	1.62351e4	1072.52429	50.7541
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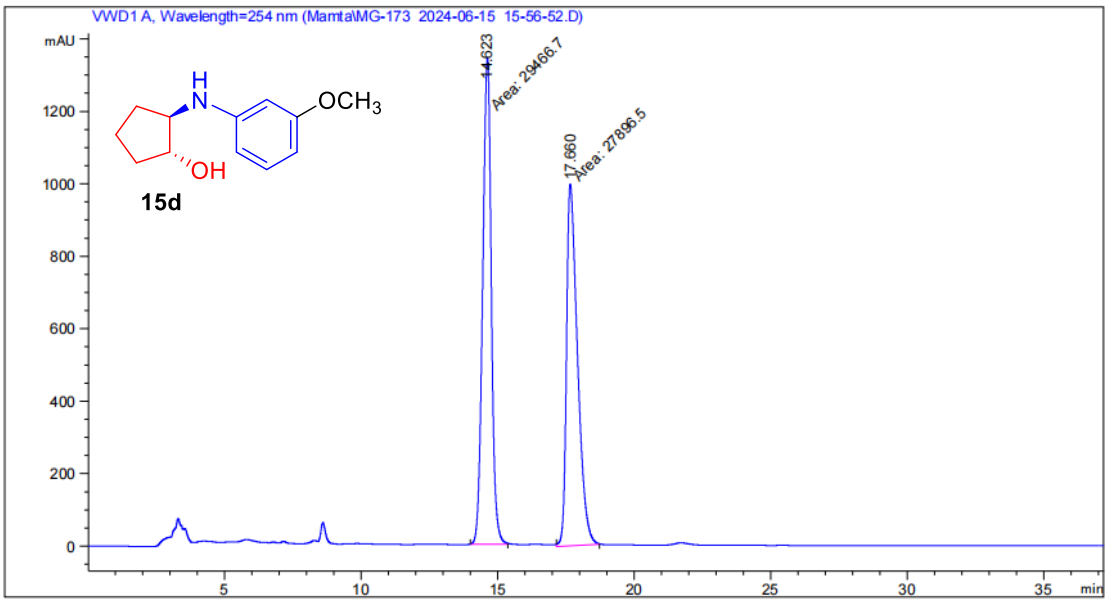
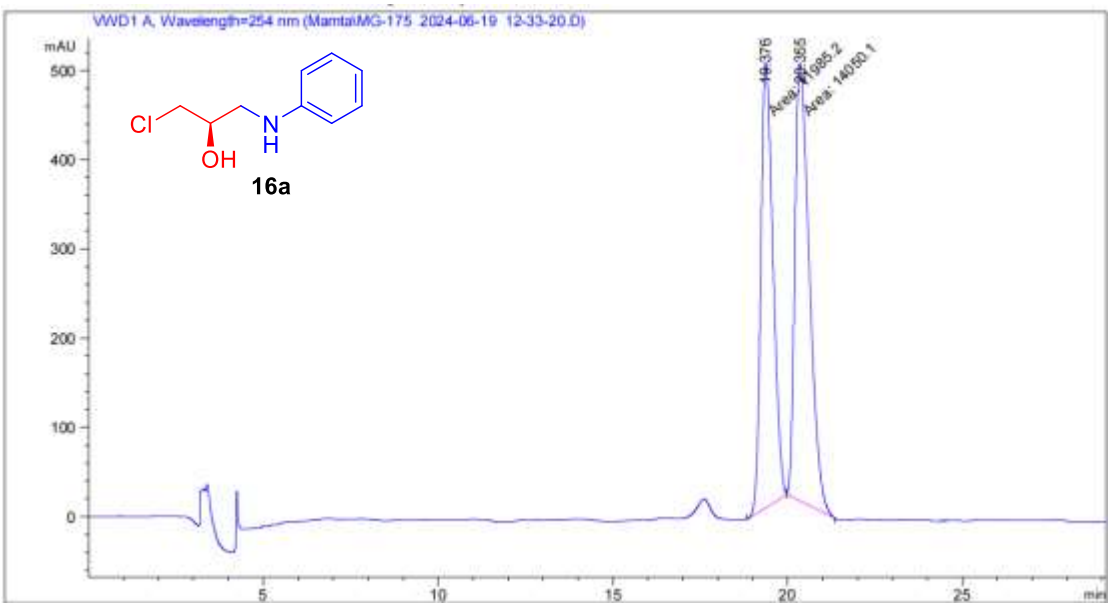


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.623	MM	0.3655	2.94667e4	1343.50720	51.3686
2	17.660	MM	0.4661	2.78965e4	997.56348	48.6314



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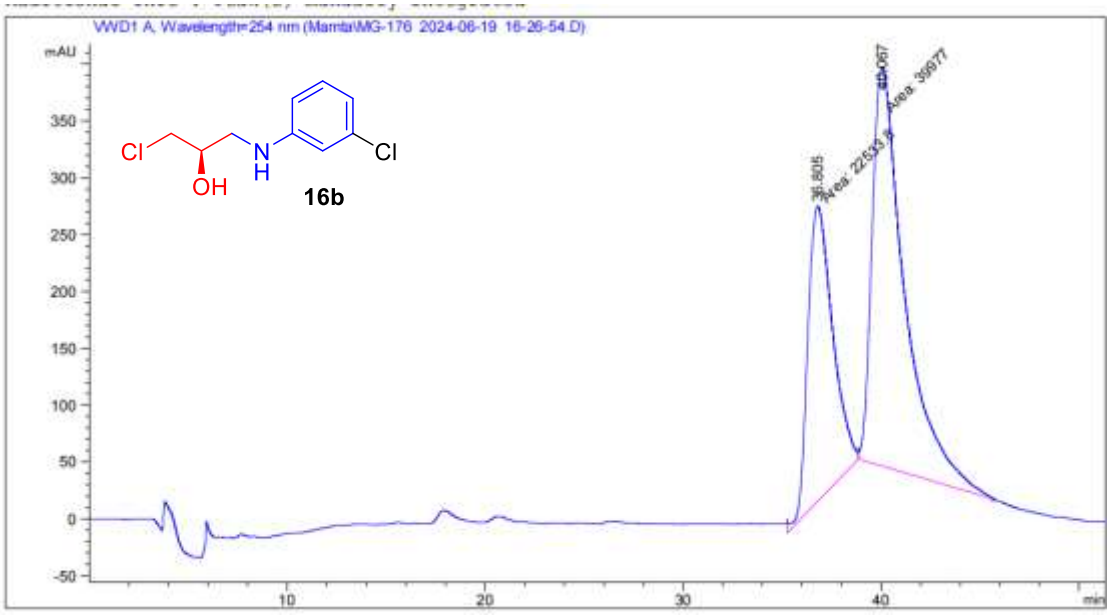
1	19.376	MM	0.4000	1.19852e4	499.35498	46.0343
2	20.355	MM	0.4771	1.40501e4	490.76544	53.9657



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.805	MM	1.4478	2.25338e4	259.40082	36.0478
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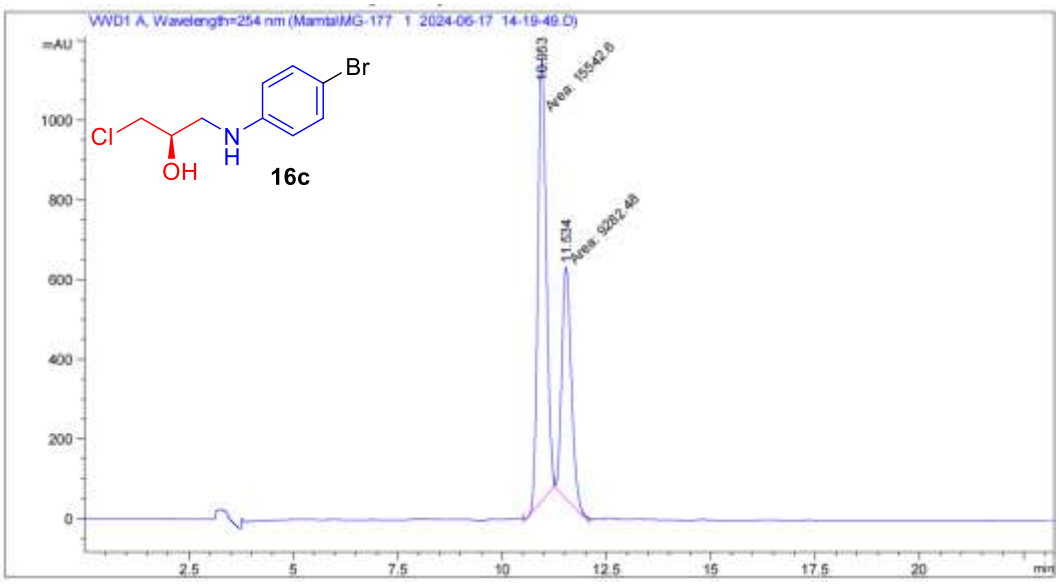

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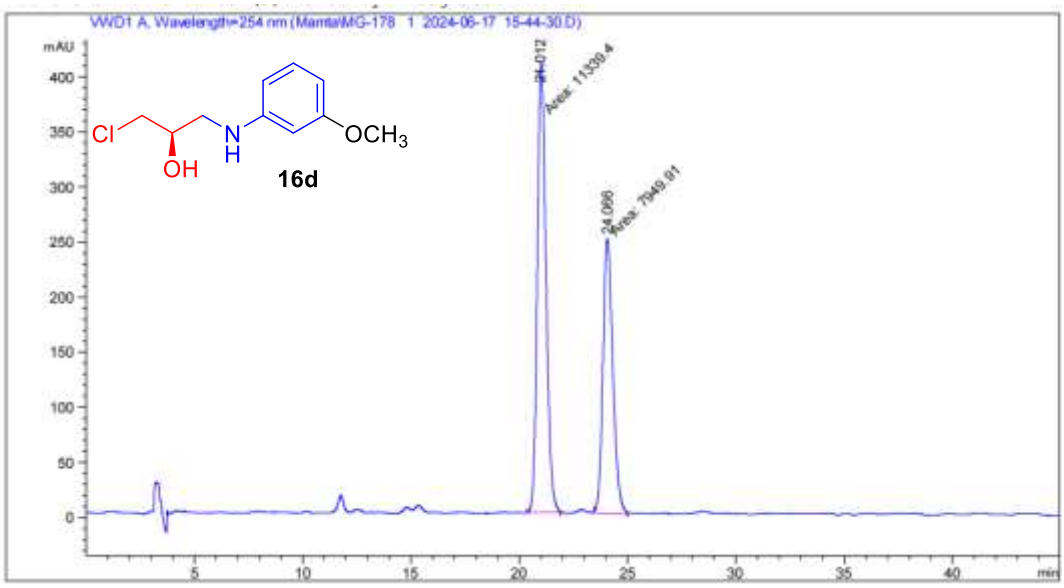
1	10.953	MM	0.2342	1.55426e4	1106.06543	62.6085
2	11.534	MM	0.2661	9282.48438	581.30933	37.3915



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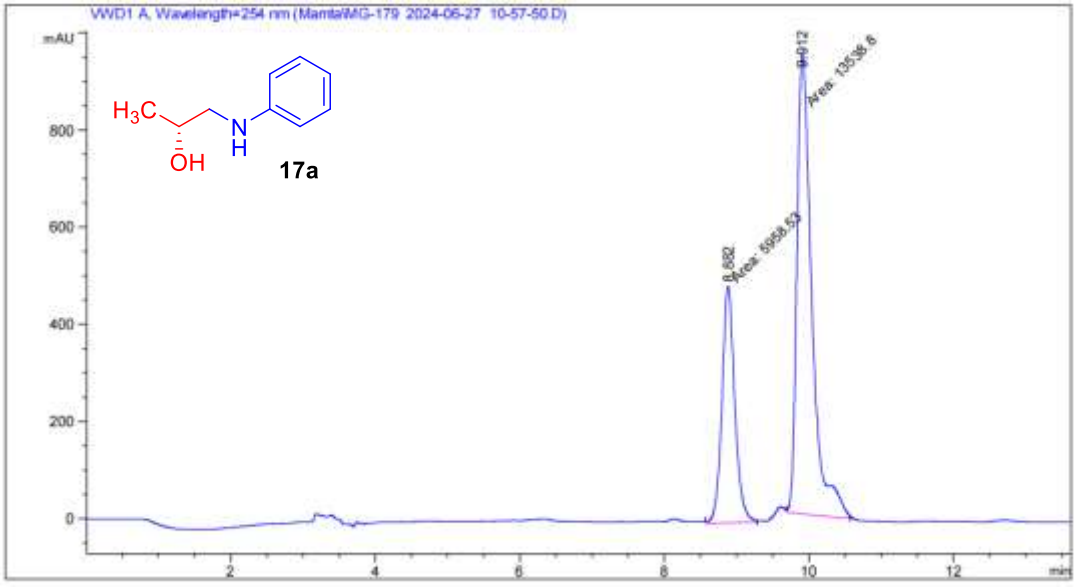
1	21.012	MM	0.4630	1.13394e4	408.20551	58.7859
2	24.066	MM	0.5301	7949.91357	249.93777	41.2141



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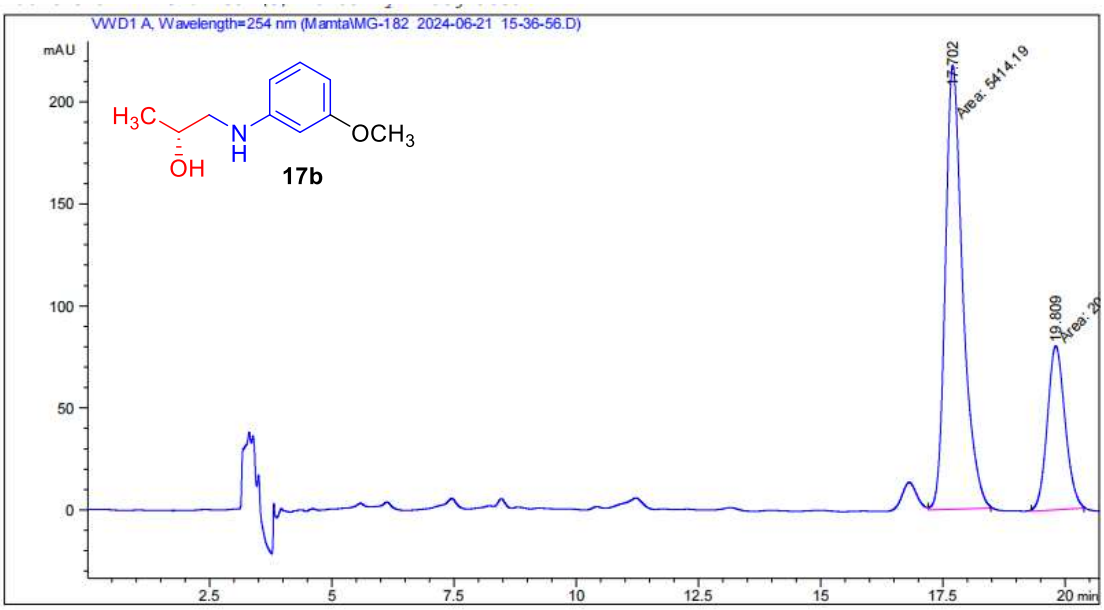
1	8.882	MM	0.2042	5958.52832	486.22195	30.5607
2	9.912	MM	0.2389	1.35388e4	944.72180	69.4393



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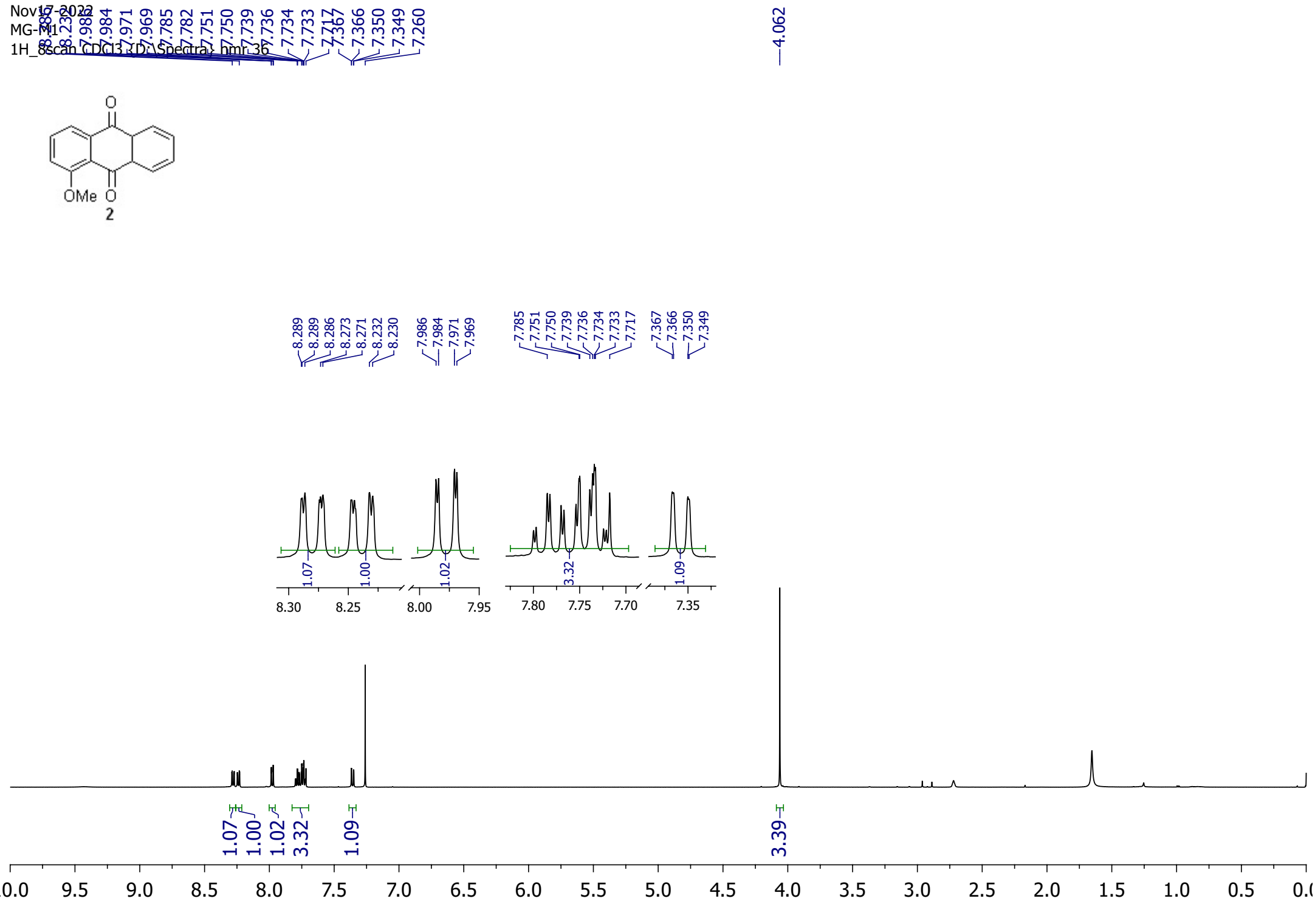
1	17.702	MM	0.4149	5414.18994	217.50145	72.6684
2	19.809	MM	0.4217	2036.35645	80.47579	27.3316

NMR Spectra of Triptycene Derivatives
&
Amino alcohol Products


Nov17-2022
MG-M1
1H_8scan CDCl3 2D:\Spectra\ hmr 36
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8.289
8.286
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8.232
8.230
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7.971
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7.260
4.062
OMe
O
O
2
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1.09
3.39
8.30
8.25
8.00
7.95
7.80
7.75
7.70
7.35
10.0
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9.0
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6.5
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Nov17-2022
MG-M1
C13CPD CDCl3 {D:\Spectra} nmr 36

183.661
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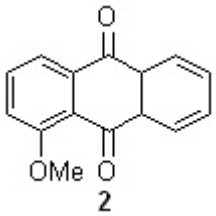

2

Nov17-2022
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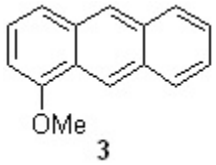

3

Nov17-2022
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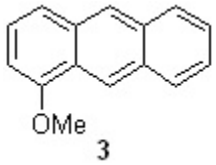
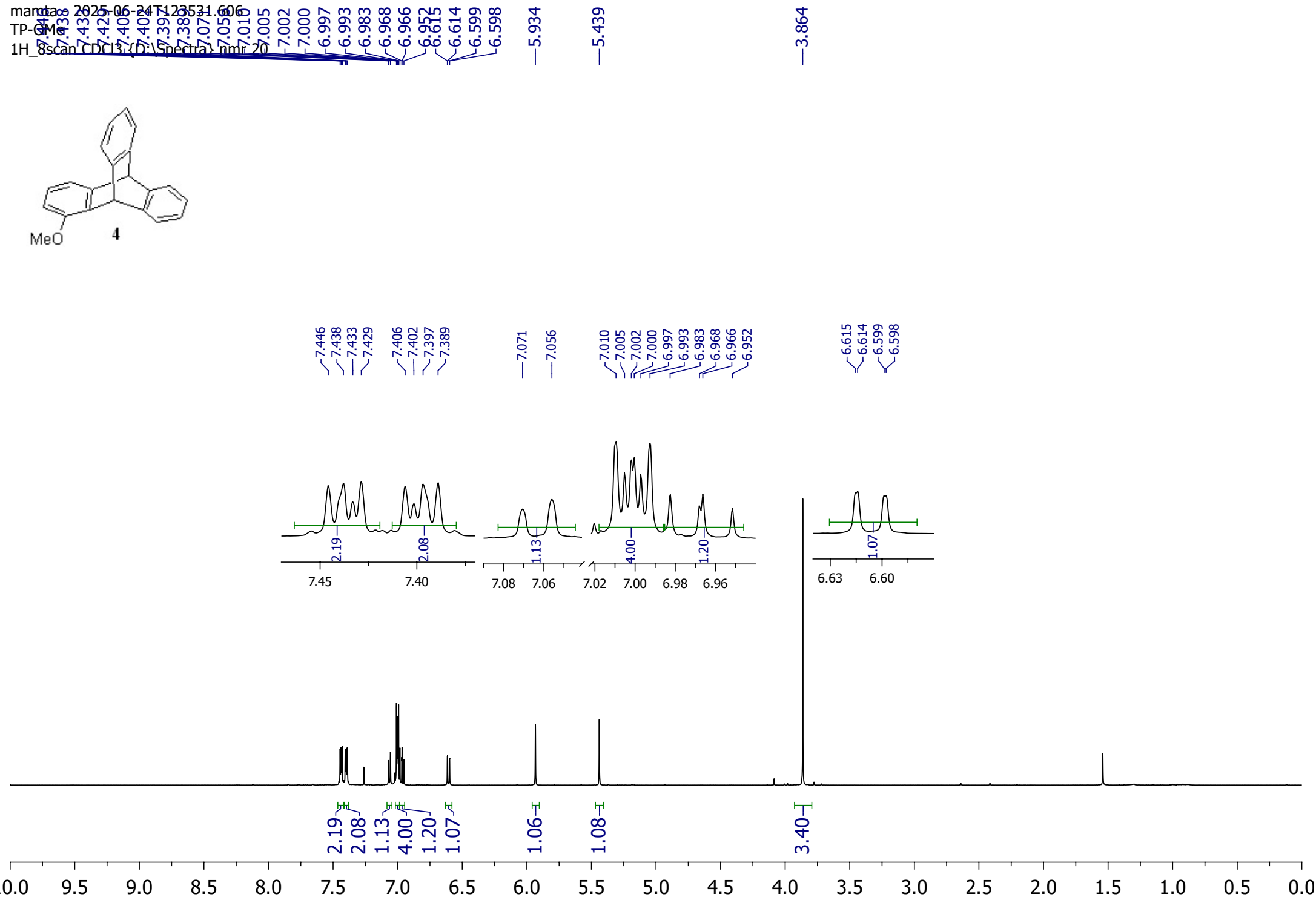
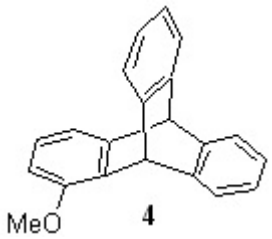


3

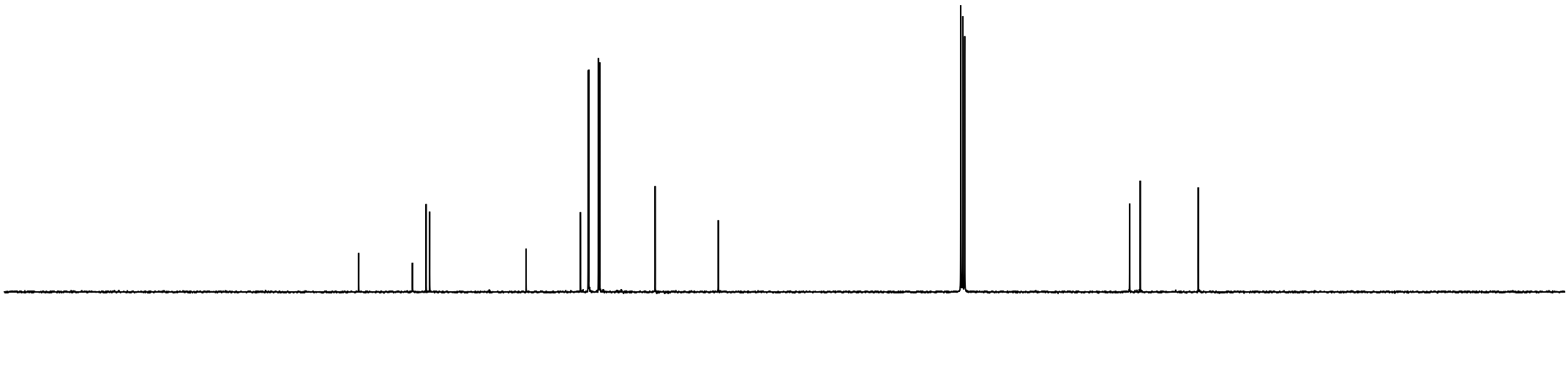

mamta - 2025-06-24T123531.606
TP-GMe
1H_8scan CDCl3 (D:\Spectra) nmr 20
7.446
7.438
7.433
7.429
7.406
7.402
7.397
7.389
7.071
7.056
7.010
7.005
7.002
7.000
6.997
6.993
6.983
6.968
6.966
6.952
6.615
6.614
6.599
6.598
5.934
5.439
3.864
MeO
4
2.19
2.08
1.13
4.00
1.20
1.07
1.06
1.08
3.40

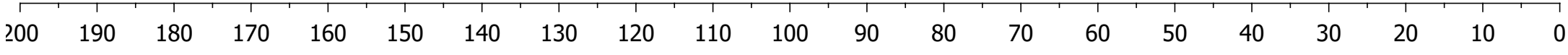
mamta - 2025-06-24T123531.606
TP-OMe
C13CPD CDCl3 {D:\Spectra} nmr 20

154.571
147.698
145.933
145.476
133.117
125.172
125.092
123.860
123.689
116.579
108.496
77.413
77.160
76.905
55.770
54.407
46.983



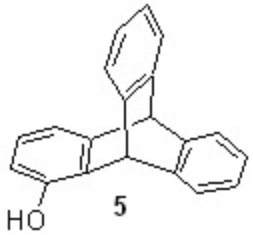
4

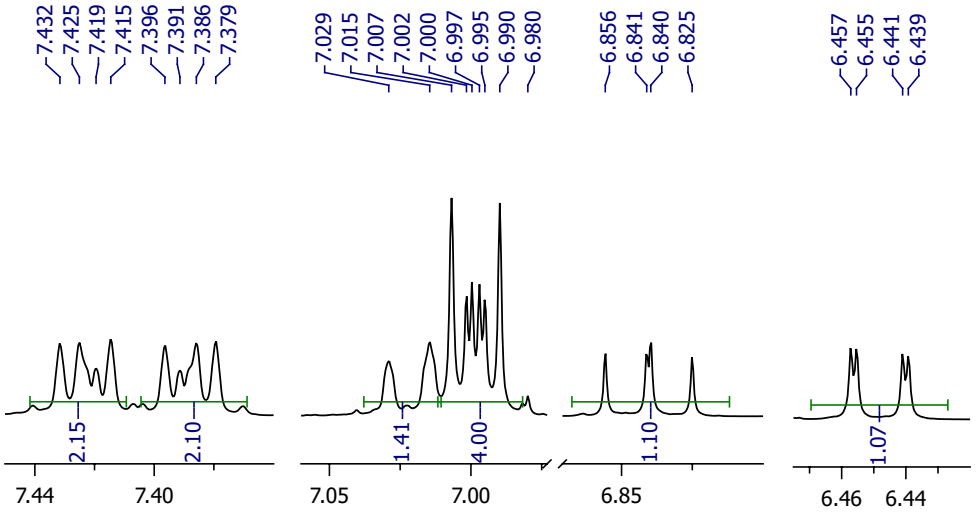


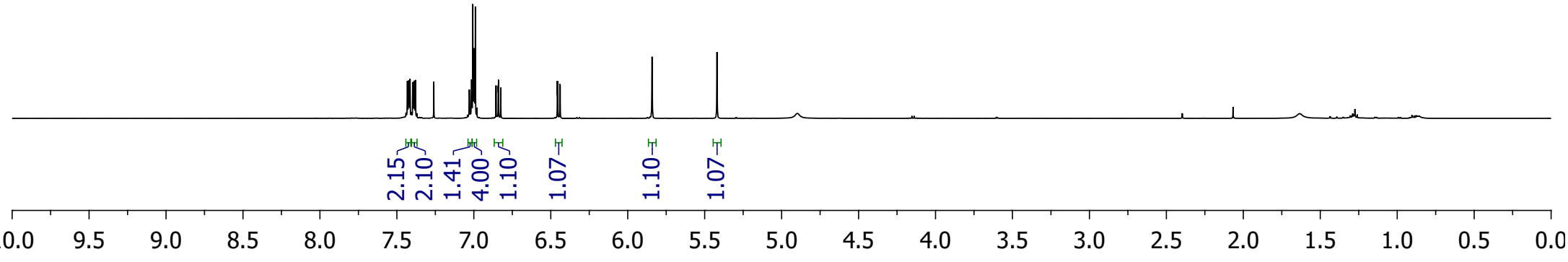


mamta, 2025-06-24T12:35:31.606
TP-OH
1H_8scan CDCl3 {D:\Spectra} nmr 21

7.432 7.425 7.419 7.415 7.396 7.391 7.386 7.379 7.029 7.015 7.007 7.002 7.000 6.997 6.995 6.990 6.856 6.841 6.840 6.825 6.457 6.455 6.441 6.439 5.842 5.420

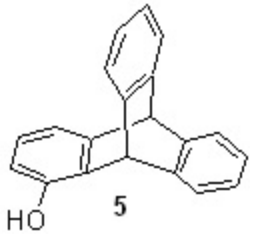


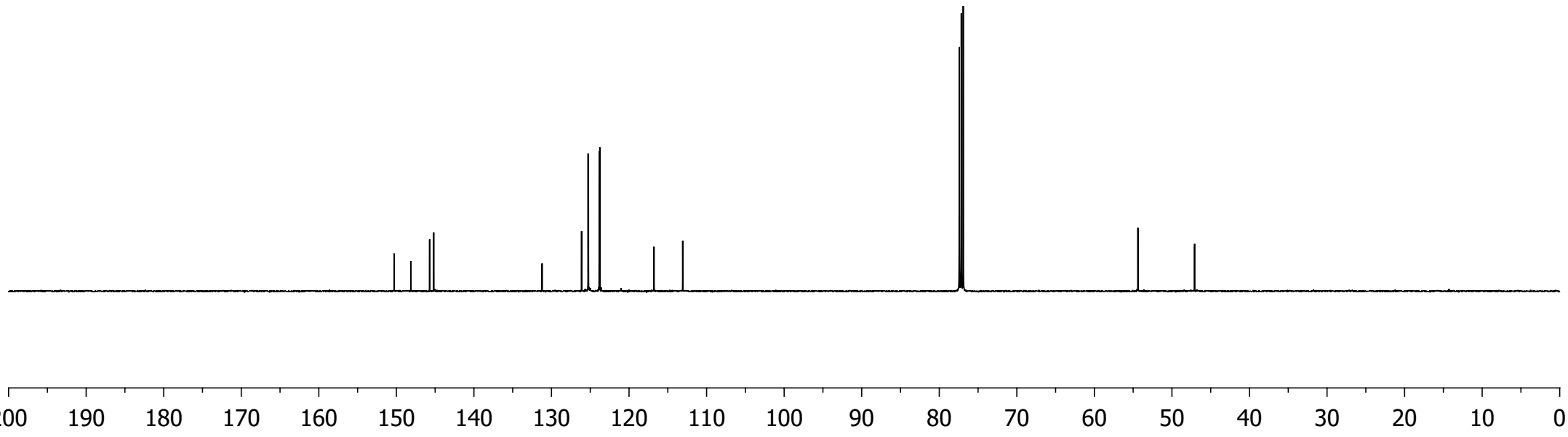




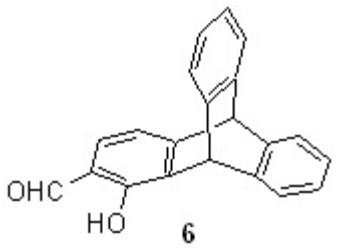
mamta - 2025-06-24T123531.606
TP-OH
C13CPD CDCl3 {D:\Spectra} nmr 21

150.289
148.123
145.715
145.184
131.209
126.123
125.277
125.234
123.848
123.767
116.779
113.079
77.415
77.160
76.907
54.385
47.082

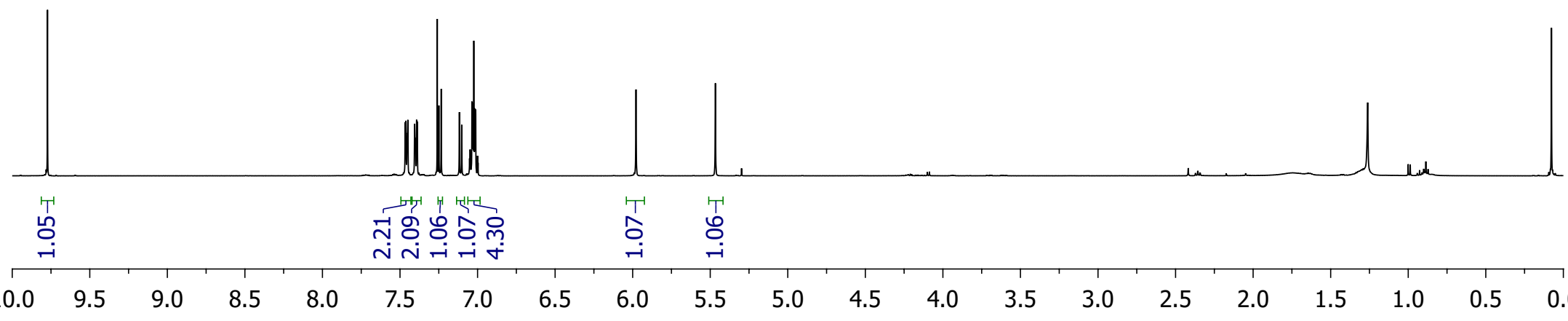




Mar01-2023
MG_16
1H_8scan CDCl3 {D:\Spectra} nmr 51

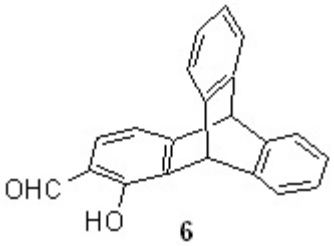


6

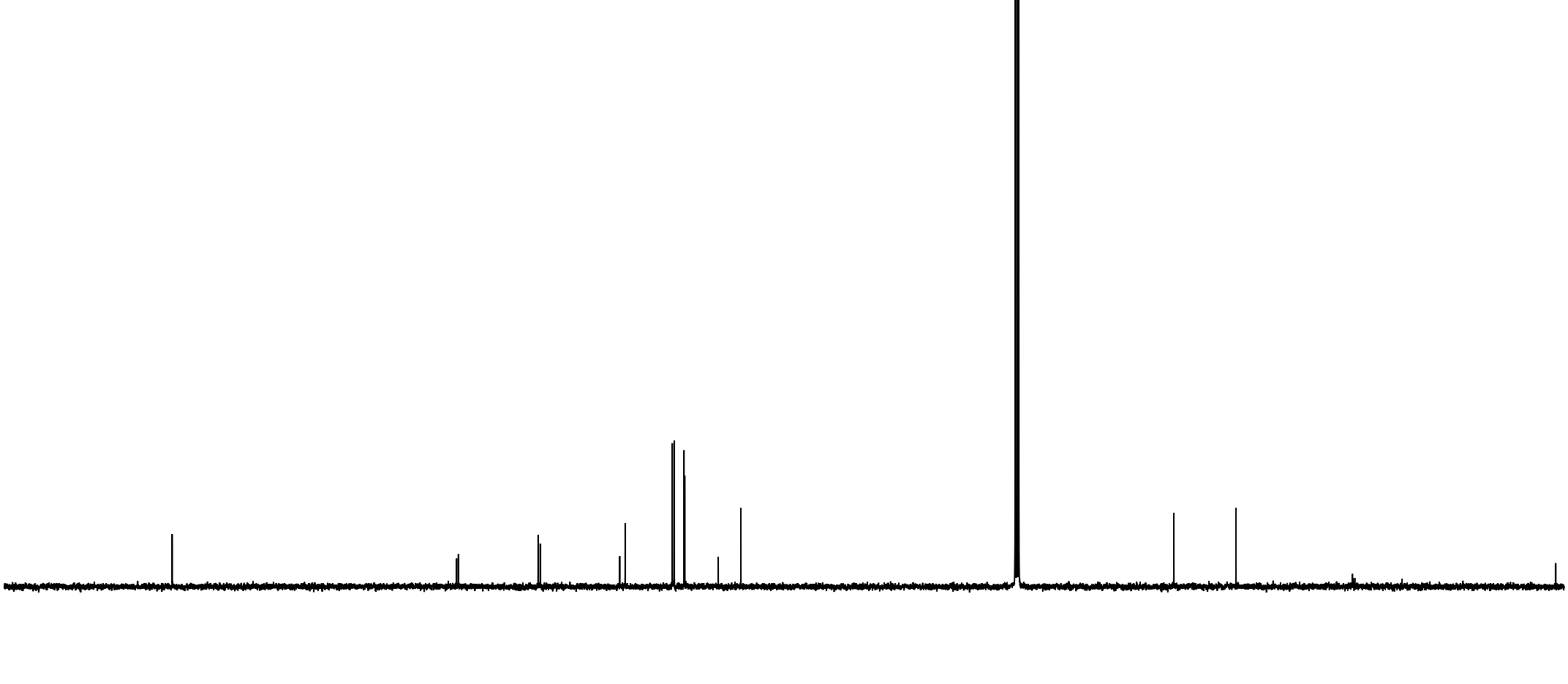


Mar01-2023
MG_16
C13CPD CDCl3 {D:\Spectra} nmr 51

196.318
156.203
155.951
144.699
144.396
133.186
132.421
125.771
125.501
124.127
124.017
119.293
116.101
77.414
77.160
76.907
55.033
46.270

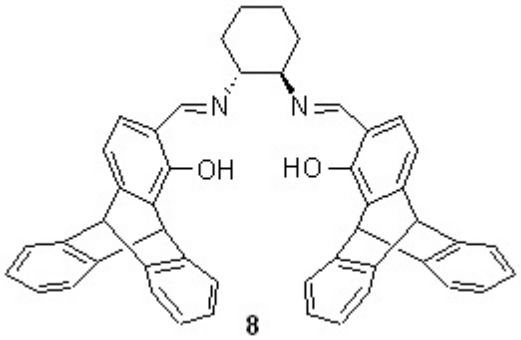


6



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

Jun21-2019
MB_150
1H_8scan DMSO {D:\Spectra} nmr 52



8

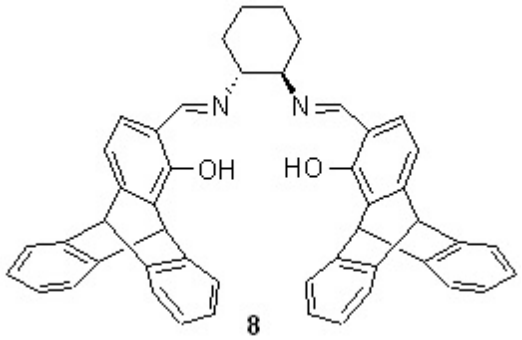
8.391
7.411
7.401
7.394
7.385
7.379
7.376
7.370
6.977
6.971
6.966
6.960
6.957
6.951
6.940
5.568
2.500
1.823
1.799
1.748
1.736
1.554
1.538
1.415
1.396
1.377

2.27
9.00
11.14
2.07
2.11
2.07
2.30
2.05
2.30
2.31

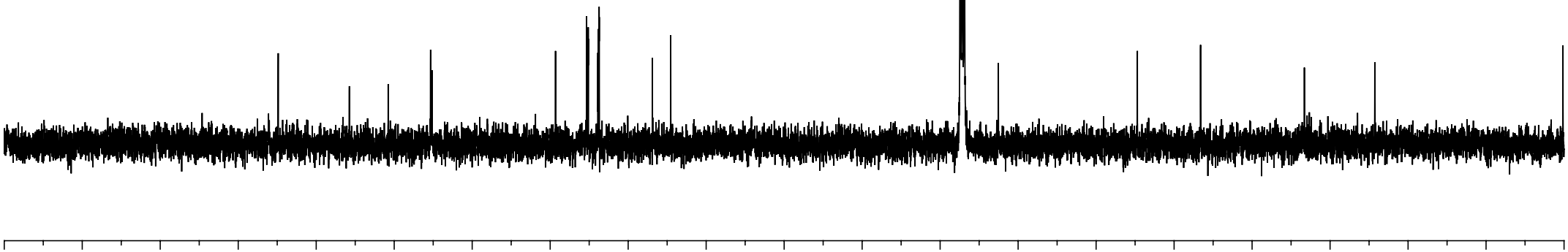
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

Jul11-2019
MB_150
C13CPD CDCl3 {D:\Spectra} nmr27

164.872
155.525
150.767
145.333
129.316
125.333
125.117
123.834
123.724
116.895
114.548
77.413
77.160
76.905
72.536
54.715
46.609
33.294
24.258

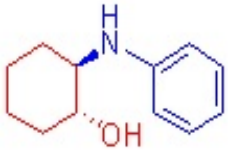


8

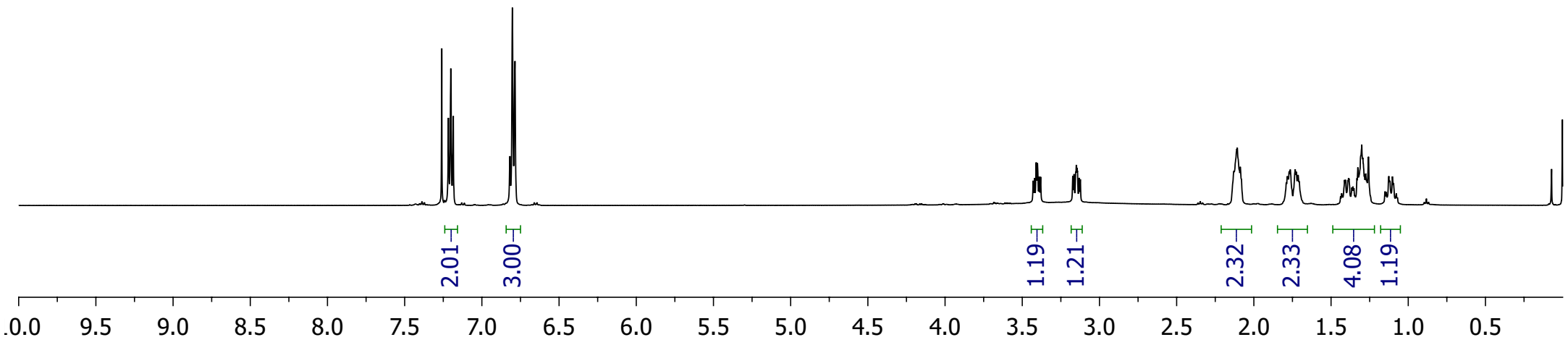


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

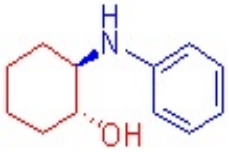
Jun26-2023
MG-64
1H_8scan CDCl3 {D:\Spectra} nmr 41



11a

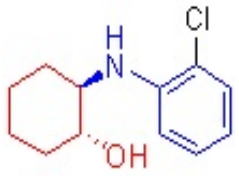


Jun26-2023
MG-64
C13CPD CDCl3 {D:\Spectra} nmr 41



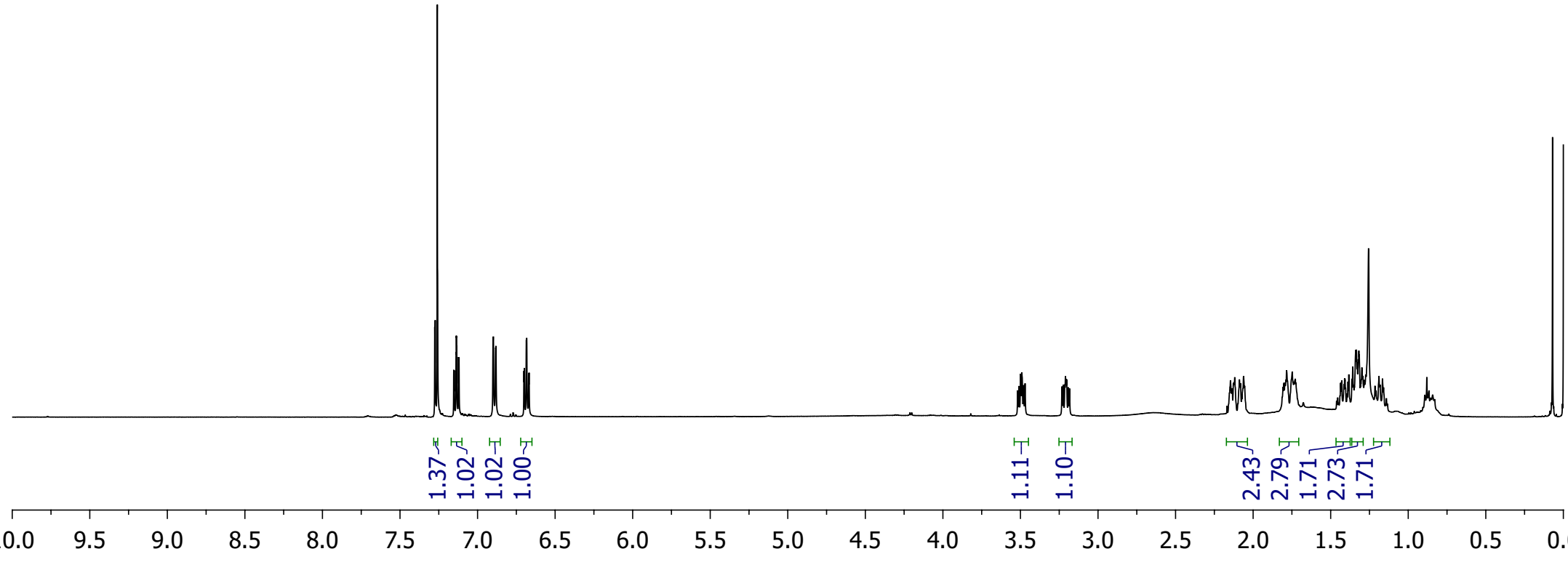
11a

Nov10-2023
MG-96
1H_8scan CDCl3 (D:\Spectra) nmr 56

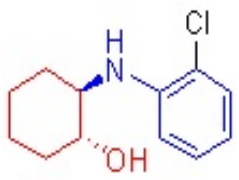


11b

7.270 7.263 7.158 7.151 7.139 7.137 7.136 7.134 7.122 7.119 6.898 6.882 6.701 6.698 6.685 6.683 6.670 6.668 3.499 3.491 3.470 3.210 3.206 3.202 2.146 2.127 2.123 2.118 2.088 2.081 2.062 1.801 1.795 1.789 1.784 1.748 1.735 1.728 1.724 1.436 1.429 1.410 1.407 1.388 1.383 1.363 1.357 1.339 1.334 1.329 1.322 1.318 1.316 1.305 1.303 1.299 1.292 1.284 1.281 1.274 1.256 1.189 1.166



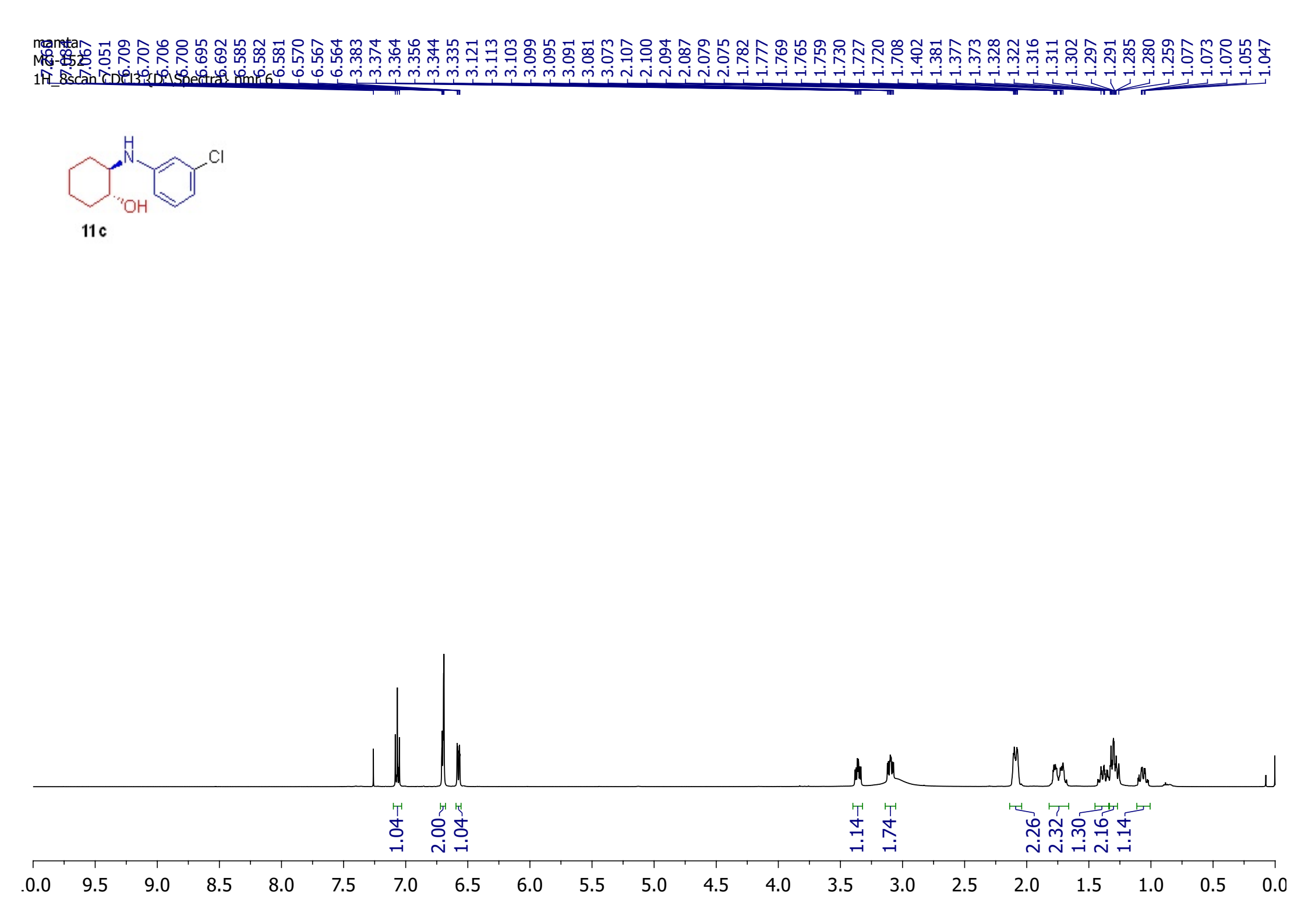
Nov10-2023
MG -96
C13CPD CDCl3 {D:\Spectra} nmr 56



11b

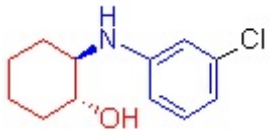
143.407
129.558
127.982
118.788
115.580
113.674
77.413
77.160
76.906
74.528
60.370
33.319
31.584
25.067
24.307

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

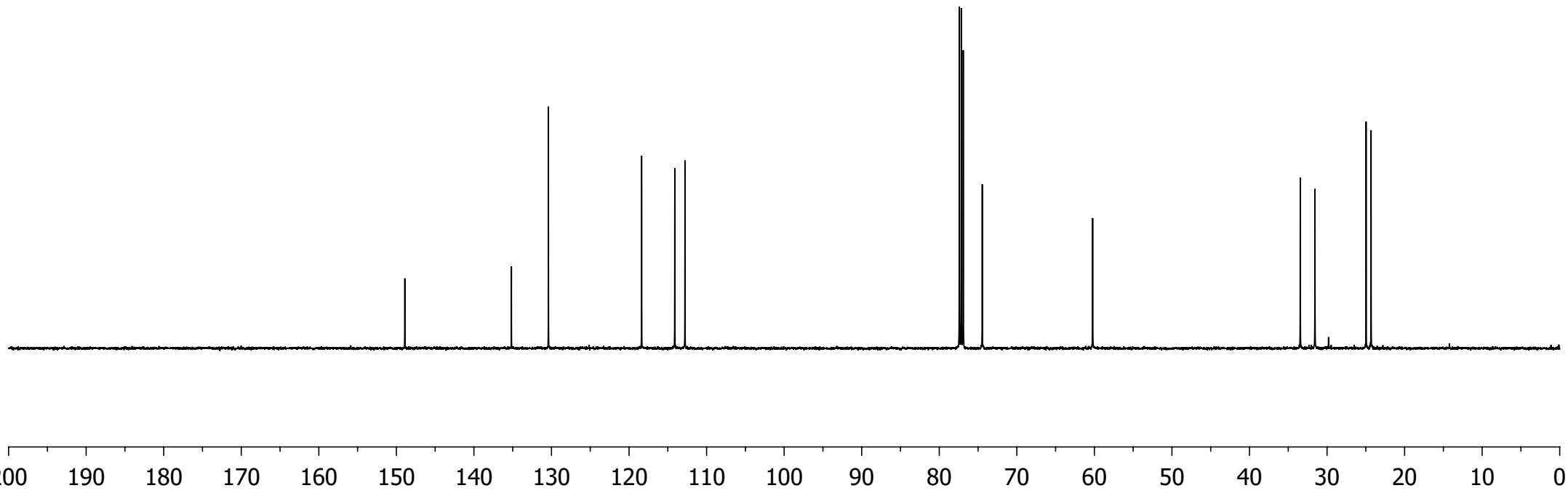

mamta
MG-152
1H_8scan CDCl3 (D:\Spectra: nmr6
7.088
7.067
7.051
6.709
6.707
6.706
6.700
6.695
6.692
6.585
6.582
6.581
6.570
6.567
6.564
3.383
3.374
3.364
3.356
3.344
3.335
3.121
3.113
3.103
3.099
3.095
3.091
3.081
3.073
2.107
2.100
2.094
2.087
2.079
2.075
1.782
1.777
1.769
1.765
1.759
1.730
1.727
1.720
1.708
1.402
1.381
1.377
1.373
1.328
1.322
1.316
1.311
1.302
1.297
1.291
1.285
1.280
1.259
1.077
1.073
1.070
1.055
1.047
NH
Cl
OH
11c
1.04
2.00
1.04
1.14
1.74
2.26
2.32
1.30
2.16
1.14
.0.0
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

mamta
MG-152
C13CPD CDCl3 {D:\Spectra} nmr 6

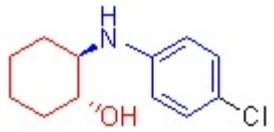
148.906
135.167
130.393
118.375
114.100
112.787
77.413
77.160
76.905
74.455
60.220
33.449
31.559
24.962
24.324



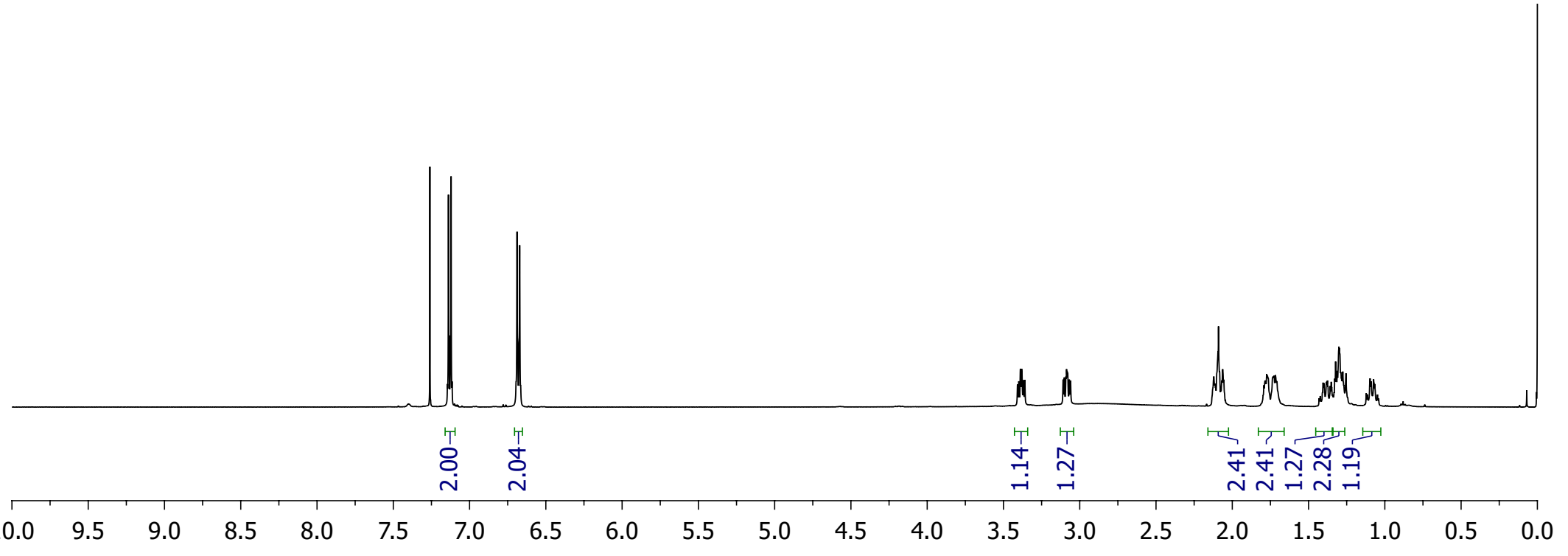
11c



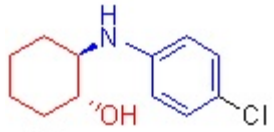
Nov23-2023
MG-99
1H_8scan CDCl3 2D\Spectra.nmr\27



11d

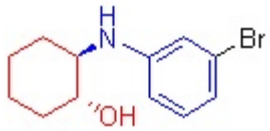


Nov22-2023
MG-99
C13CPD CDCl3 {D:\Spectra} nmr 27

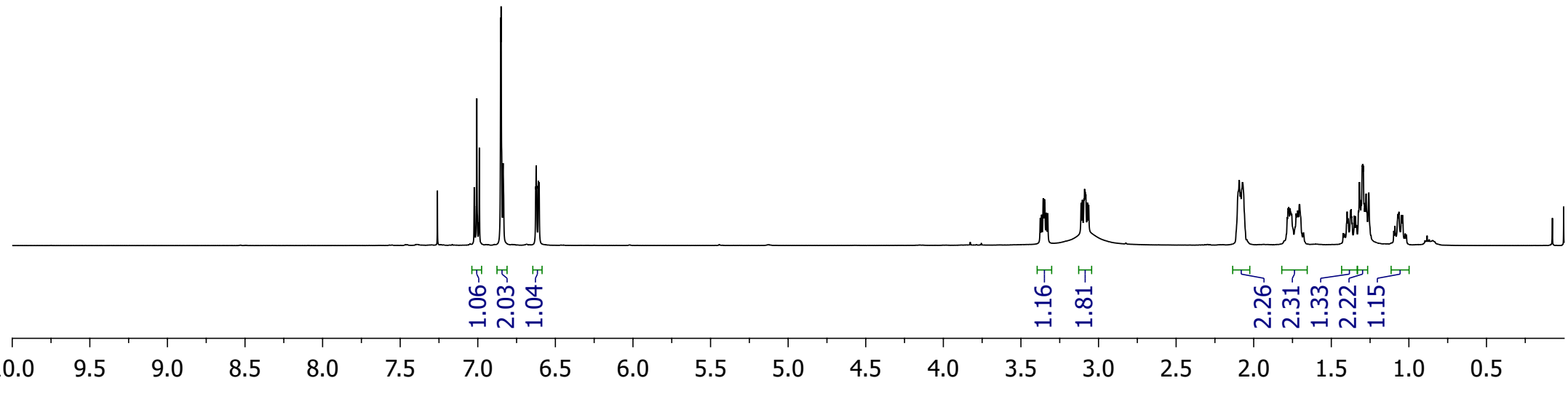


11d

mamta
MG-154
1H_8scan CDCl3 (D:\Spectra\ nmr) 7

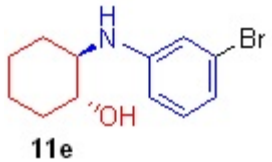


11e

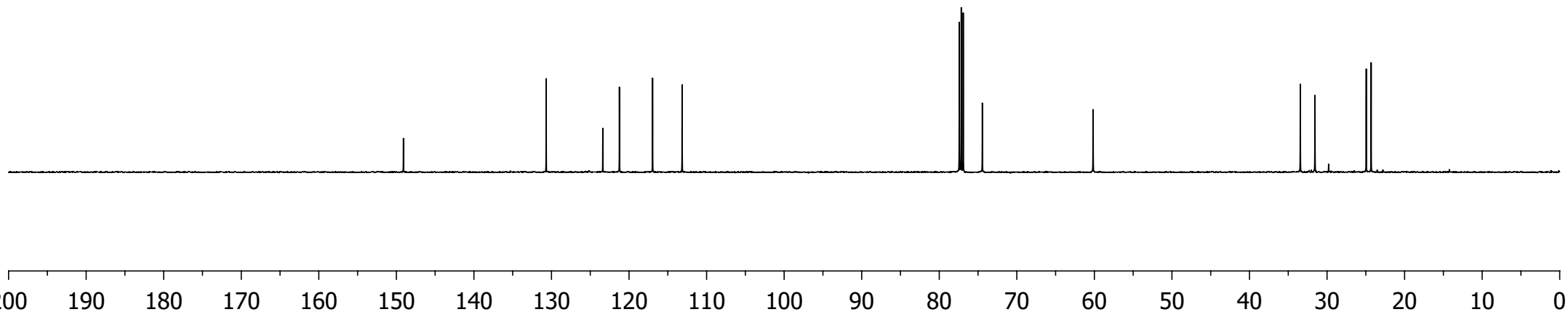


mamta
MG-154
C13CPD CDCl3 {D:\Spectra} nmr 7

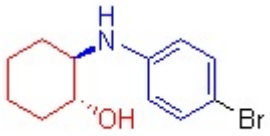
149.085
130.682
123.383
121.240
116.964
113.154
77.414
77.160
76.907
74.442
60.165
33.445
31.558
24.946
24.315



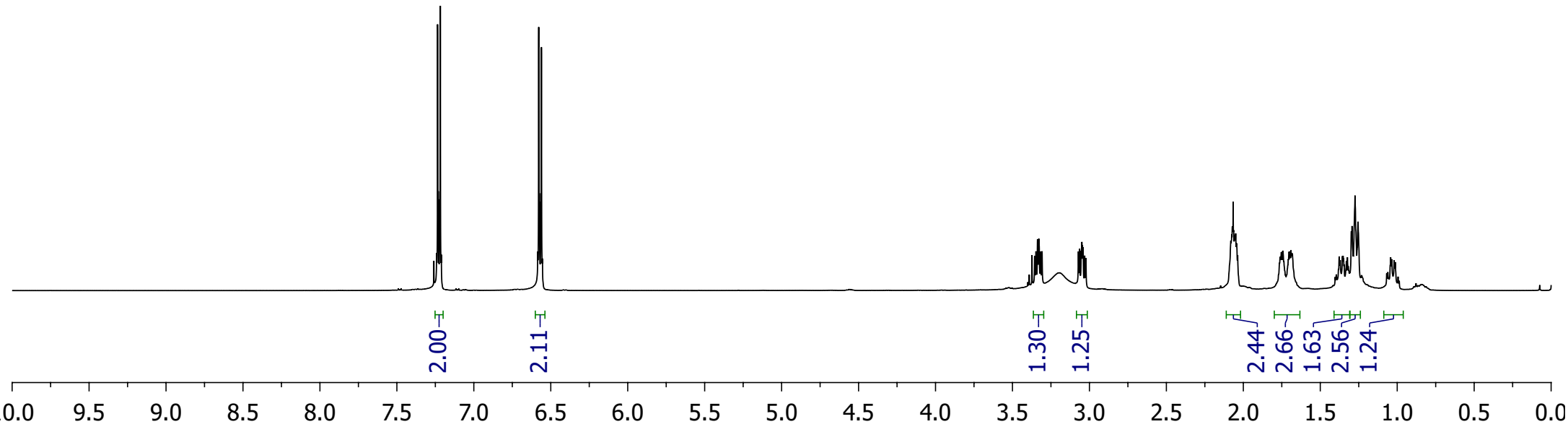
11e



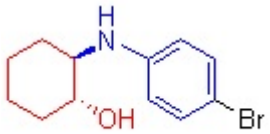
mamta
Mg-155NC
1H_8scan CDCl3 {D:\Spectra} nmr 43



11f

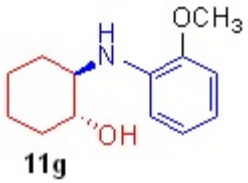


mamta
MG-155NC
C13CPD CDCl3 {D:\Spectra} nmr 43



11f

mamta
MG-156NC
1H_8scan CDCl3 (D:\Spectra: nmr 44



11g

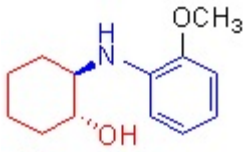
6.894 6.883 6.880 6.868 6.865 6.814 6.811 6.806 6.798 6.792 6.790 6.737 6.734 6.722 6.719 6.707 6.704 3.849 3.459 3.450 3.440 3.432 3.430 3.420 3.411 3.186 3.178 3.168 3.164 3.160 3.156 3.145 3.137 2.146 2.118 2.096 2.090 1.791 1.788 1.782 1.778 1.772 1.738 1.731 1.728 1.721 1.718 1.440 1.419 1.414 1.352 1.346 1.340 1.334 1.327 1.324 1.321 1.314 1.308 1.302 1.107 1.100 1.085

1.18 2.29 1.16 3.58 1.27 1.37 2.50 2.56 1.28 2.63 1.27

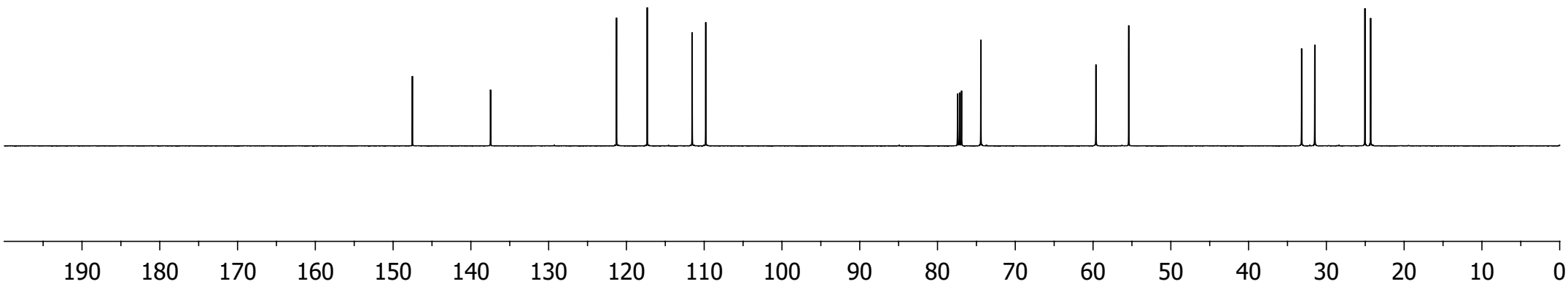
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

mamta
MG-156NC
C13CPD CDCl3 {D:\Spectra} nmr 44

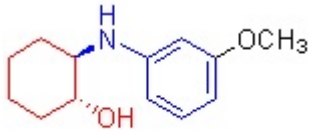
147.520
137.470
121.269
117.322
111.545
109.801
77.416
77.160
76.906
74.426
59.627
55.411
33.179
31.486
25.040
24.303



11g

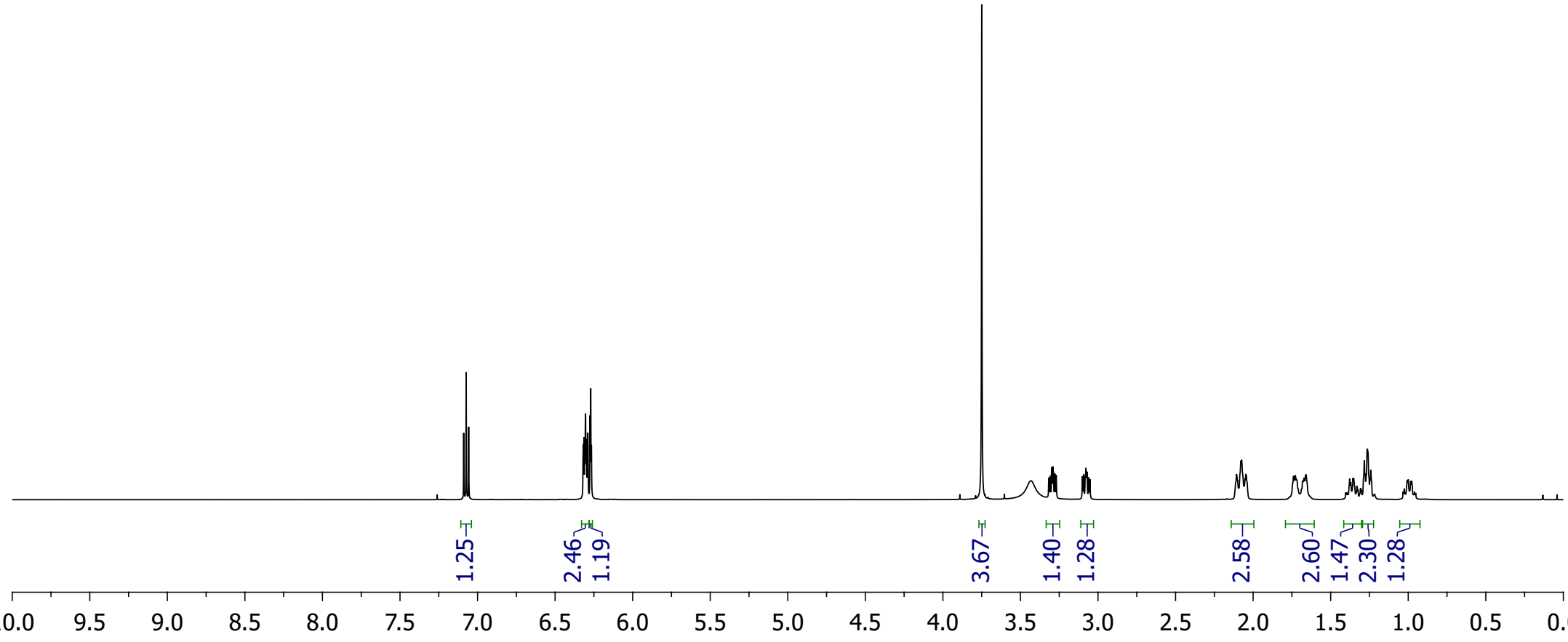


mamta
MG-153NC
1H_8scan CDCl3 (D:\Spectra: nmr 46

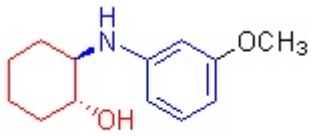


11h

7.277 7.257 6.318 6.314 6.310 6.309 6.304 6.299 6.298 6.294 6.292 6.289 6.275 6.270 6.266 3.750 3.317 3.309 3.299 3.297 3.290 3.288 3.278 3.269 3.101 3.093 3.082 3.078 3.074 3.071 3.060 3.052 2.107 2.100 2.078 2.074 2.053 2.046 2.040 1.739 1.735 1.728 1.724 1.718 1.681 1.679 1.671 1.661 1.659 1.378 1.370 1.356 1.352 1.283 1.278 1.264 1.246 1.242 1.007 1.003 1.000 0.985 0.977

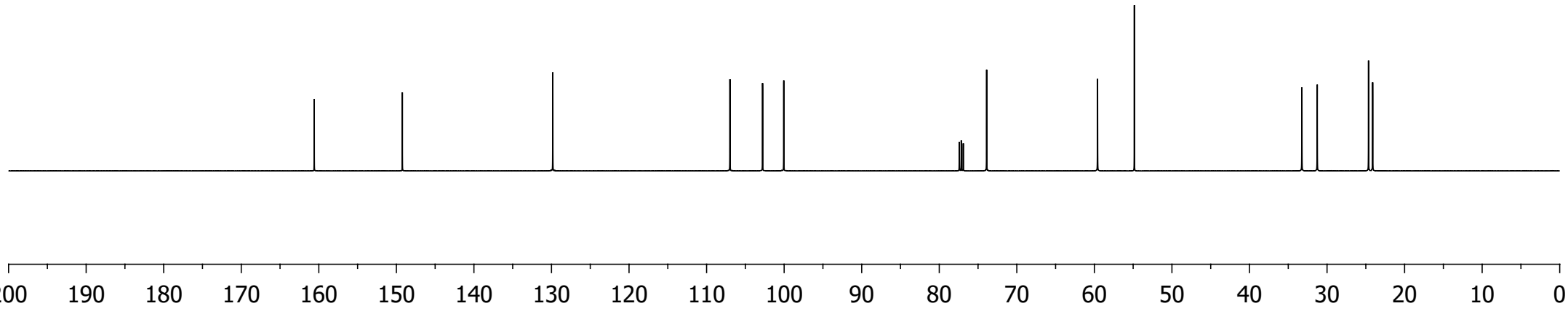


mamta
MG-153NC
C13CPD CDCl3 {D:\Spectra} nmr 46

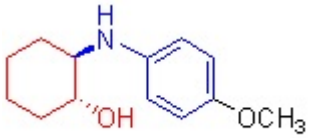


11h

160.591
149.231
129.834
106.987
102.781
100.047
77.414
77.160
76.903
73.890
59.591
54.846
33.246
31.265
24.649
24.116



mamta
MG-157NC
1H_8scan CDCl3 (D:\Spectra): nmr.45



11i

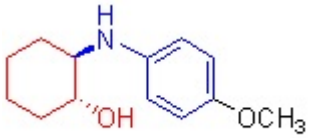
6.772 6.764 6.759 6.752 6.697 6.690 6.686 6.677 6.672 3.735 3.343 3.335 3.325 3.323 3.316 3.314 3.304 3.295 3.004 2.996 2.986 2.982 2.978 2.974 2.963 2.956 2.104 2.095 2.092 2.085 2.082 2.077 2.073 2.067 2.064 2.059 2.051 1.755 1.742 1.738 1.732 1.699 1.694 1.689 1.682 1.680 1.673 1.380 1.373 1.355 1.352 1.327 1.304 1.299 1.280 1.274 1.269 1.262 1.258 1.256 1.238 1.013 0.990

2.24 2.27 3.45 1.19 1.17 2.51 2.50 1.44 2.52 1.20

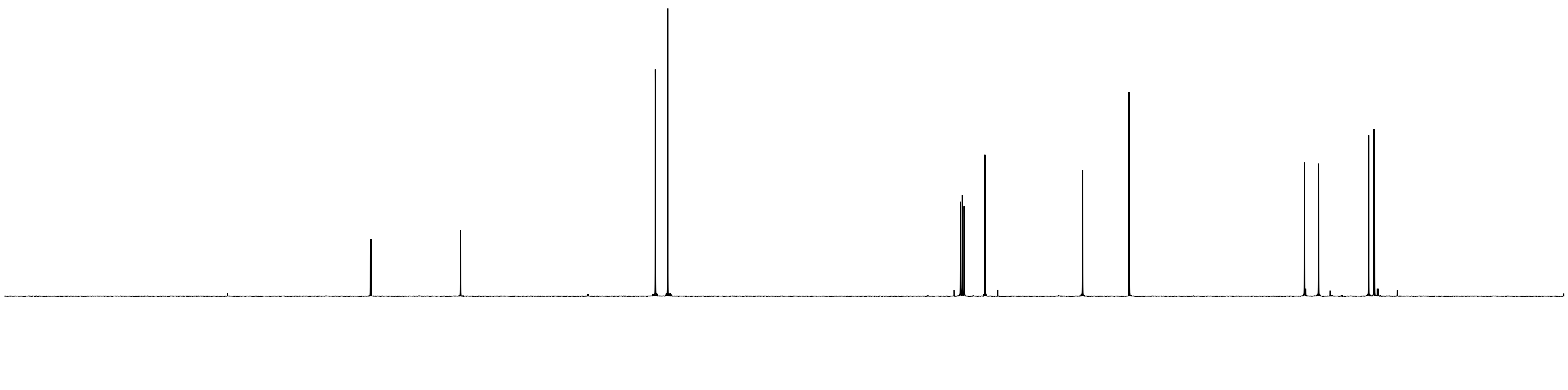
.0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

mamta
MG-157NC
C13CPD CDCl3 {D:\Spectra} nmr 45

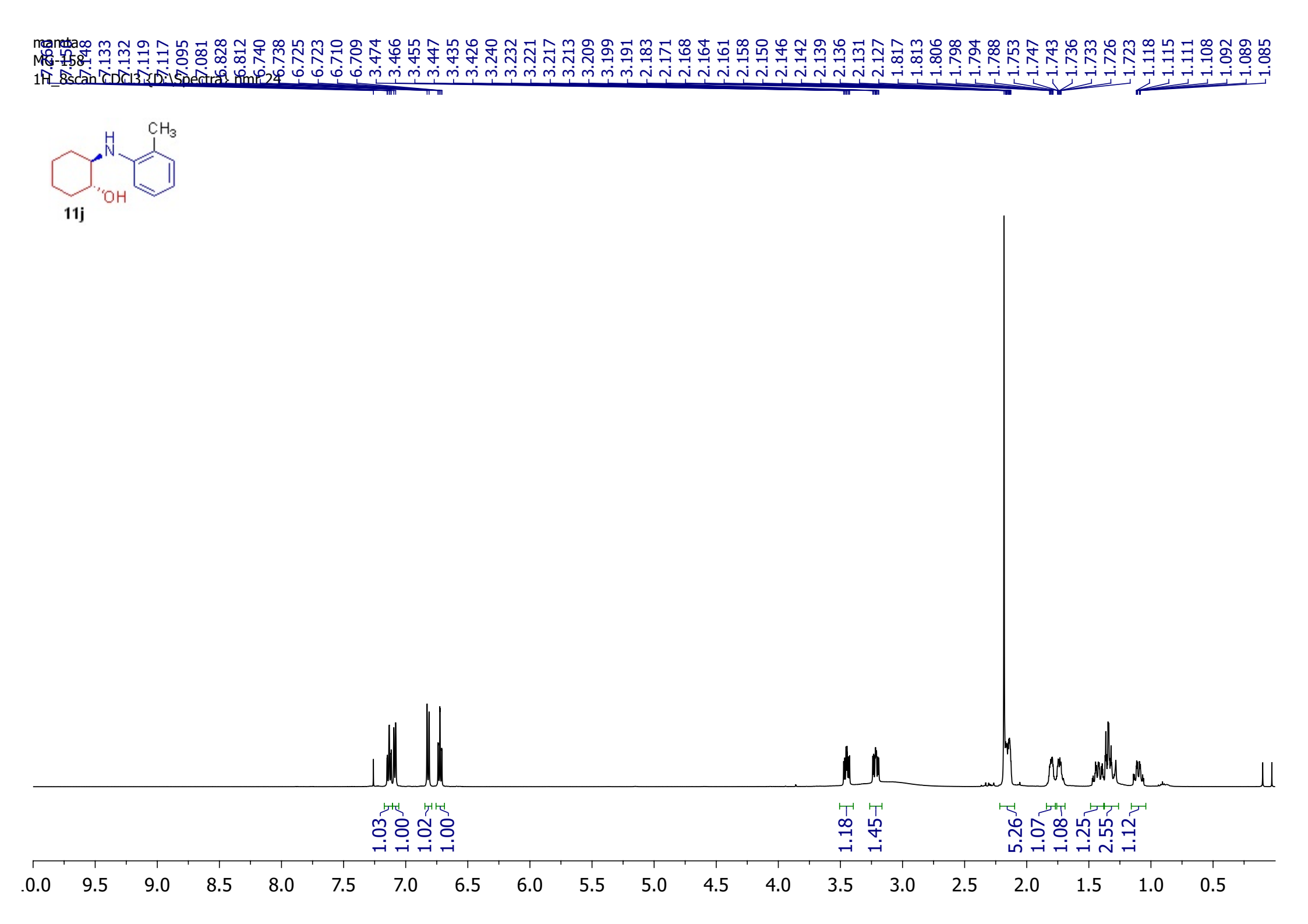
153.006
141.463
116.539
114.897
77.414
77.160
76.906
74.268
61.747
55.771
33.251
31.459
25.070
24.342



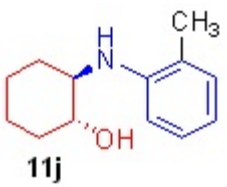
11i



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

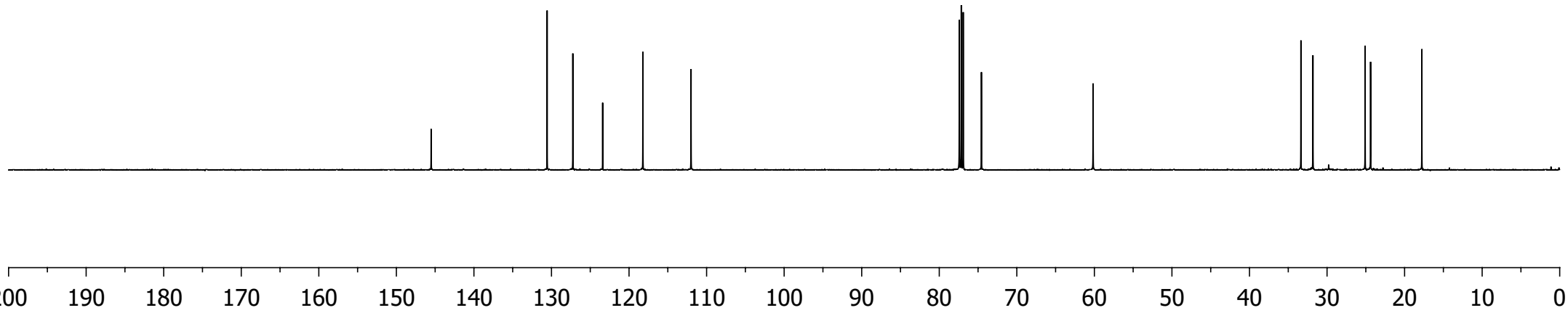

mamta
MG-158
1H_8scan CDCl3 (D:\Spectra\nmr\24
7.451
7.448
7.133
7.132
7.119
7.117
7.095
7.081
6.828
6.812
6.740
6.738
6.725
6.723
6.710
6.709
3.474
3.466
3.455
3.447
3.435
3.426
3.240
3.232
3.221
3.217
3.213
3.209
3.199
3.191
2.183
2.171
2.168
2.164
2.161
2.158
2.150
2.146
2.142
2.139
2.136
2.131
2.127
1.817
1.813
1.806
1.798
1.794
1.788
1.753
1.747
1.743
1.736
1.733
1.726
1.723
1.118
1.115
1.111
1.108
1.092
1.089
1.085
CH3
NH
OH
11j
1.03
1.00
1.02
1.00
1.18
1.45
5.26
1.07
1.08
1.25
2.55
1.12
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

mamta
MG-158
C13CPD CDCl3 {D:\Spectra} nmr 24

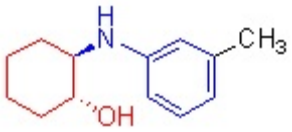


11j

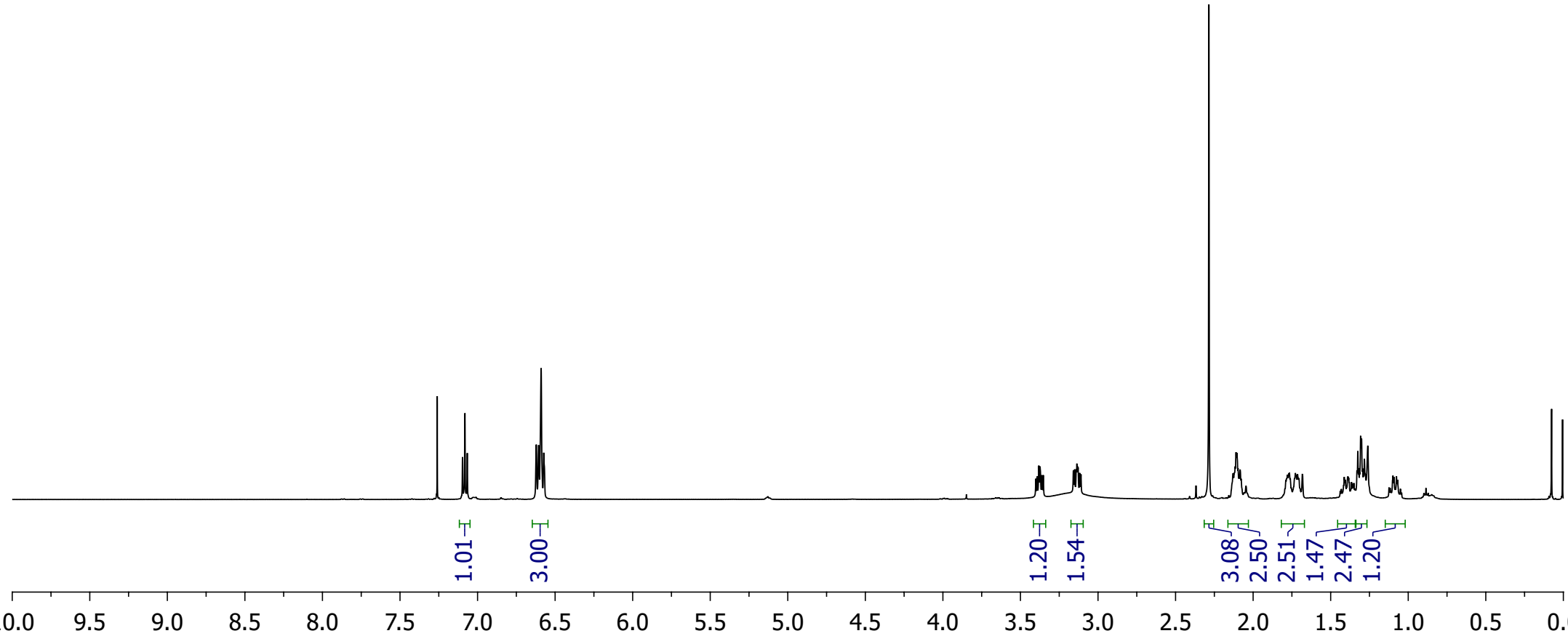
145.513
130.563
127.244
123.399
118.214
112.005
77.414
77.160
76.907
74.561
60.165
33.365
31.819
25.089
24.384
17.774



mamta
Mg-159
1H_8scan CDCl3 (D:\Spectra) nmr 41

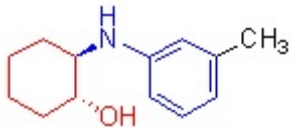


11k

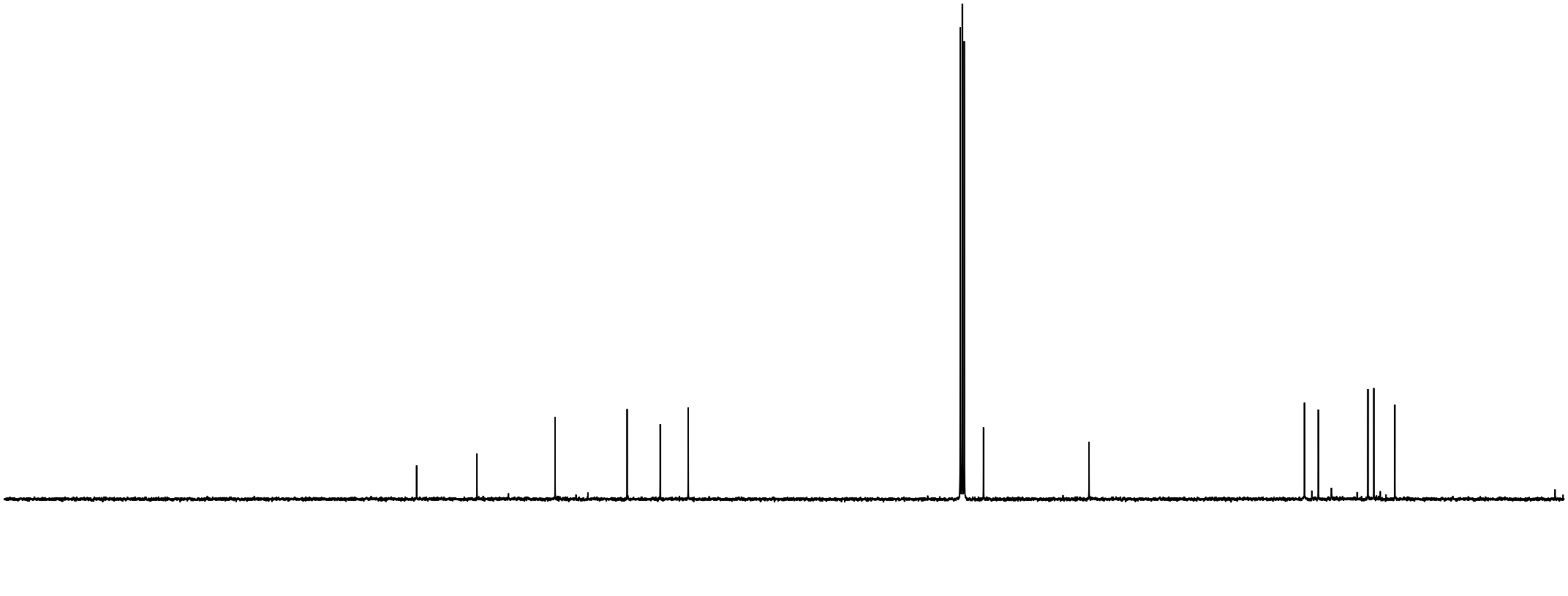


mamta
MG-159
C13CPD CDCl3 {D:\Spectra} nmr 41

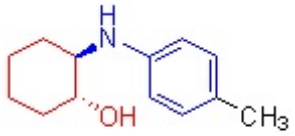
147.131
139.390
129.371
120.137
115.892
112.300
77.413
77.160
76.906
74.441
60.906
33.292
31.513
25.142
24.386
21.691



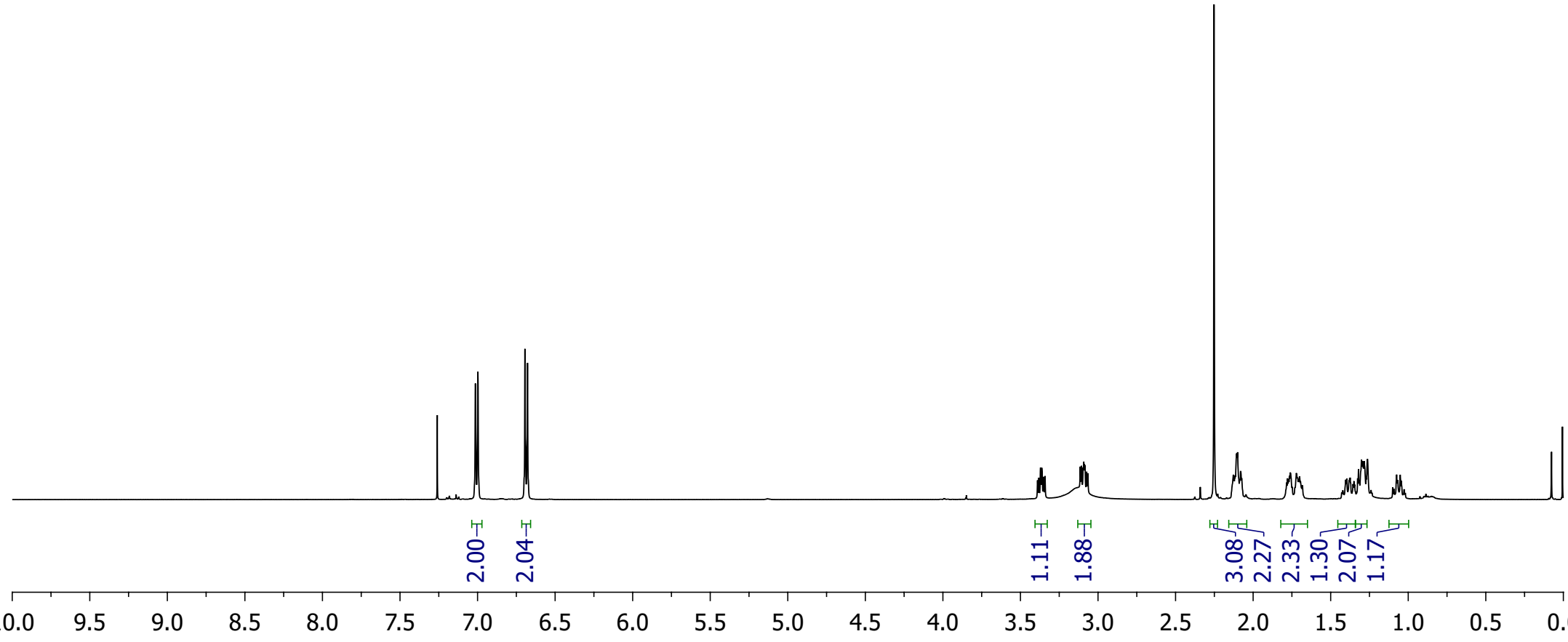
11k



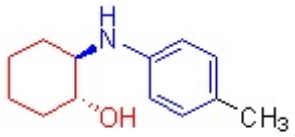
mamta
MG-160
1H_8scan CDCl3 (D:\Spectra) nmr 42



11l

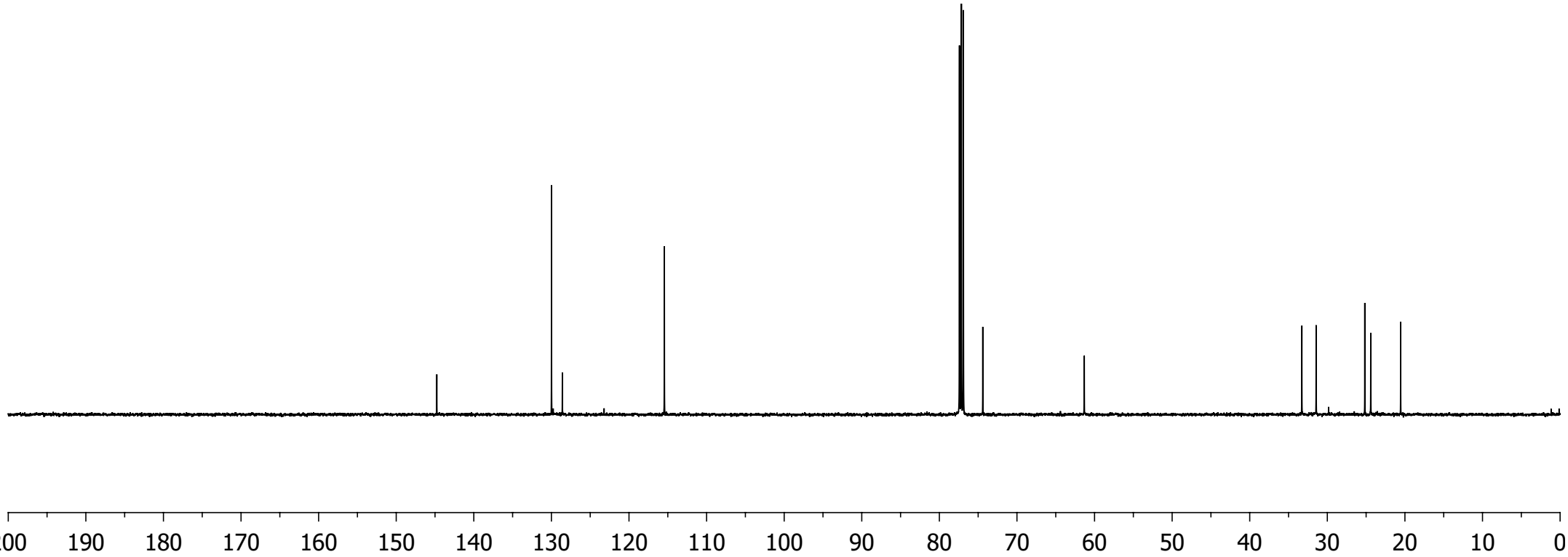


mamta
MG-160
C13CPD CDCl3 {D:\Spectra} nmr 42

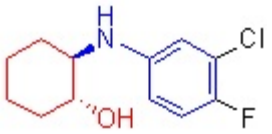


11l

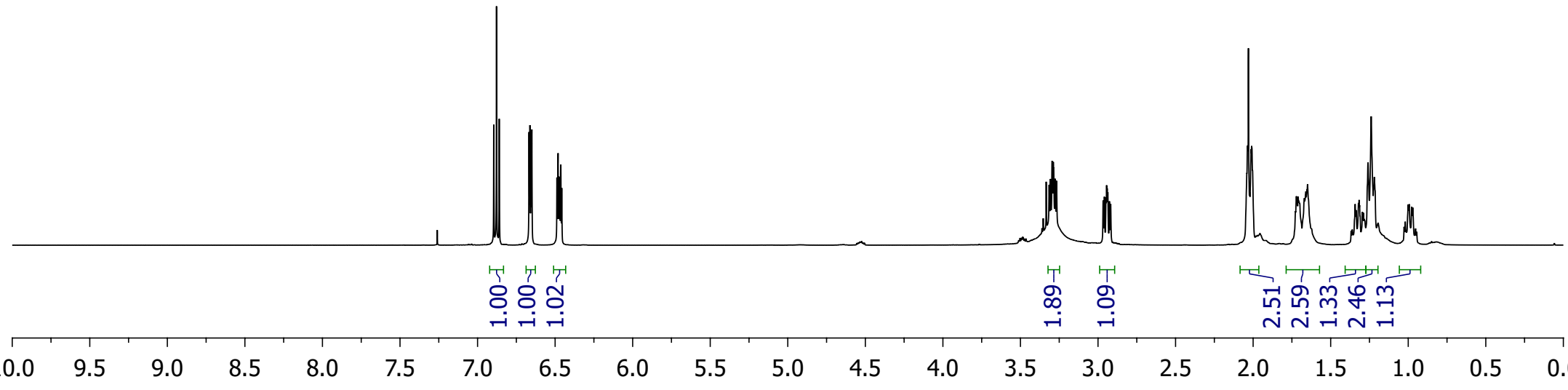
144.772
129.986
128.586
115.443
77.414
77.160
76.906
74.400
61.328
33.285
31.440
25.167
24.398
20.548



mamta
MG-163NC
1H_8scan CDCl3 (D:\Spectra: nmr 4)

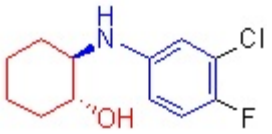


11m

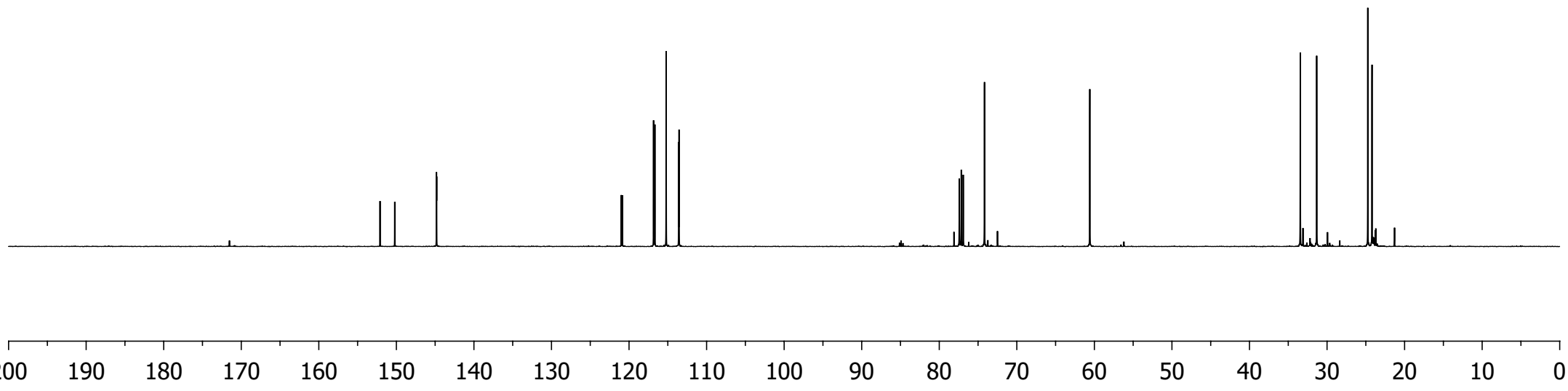


mamta
MG-163NC
C13CPD CDCl3 {D:\Spectra} nmr 47

152.105
150.209
144.824
144.809
121.000
120.855
116.839
116.667
115.214
113.596
113.549
77.414
77.160
76.906
74.171
60.590
33.447
31.332
24.746
24.201

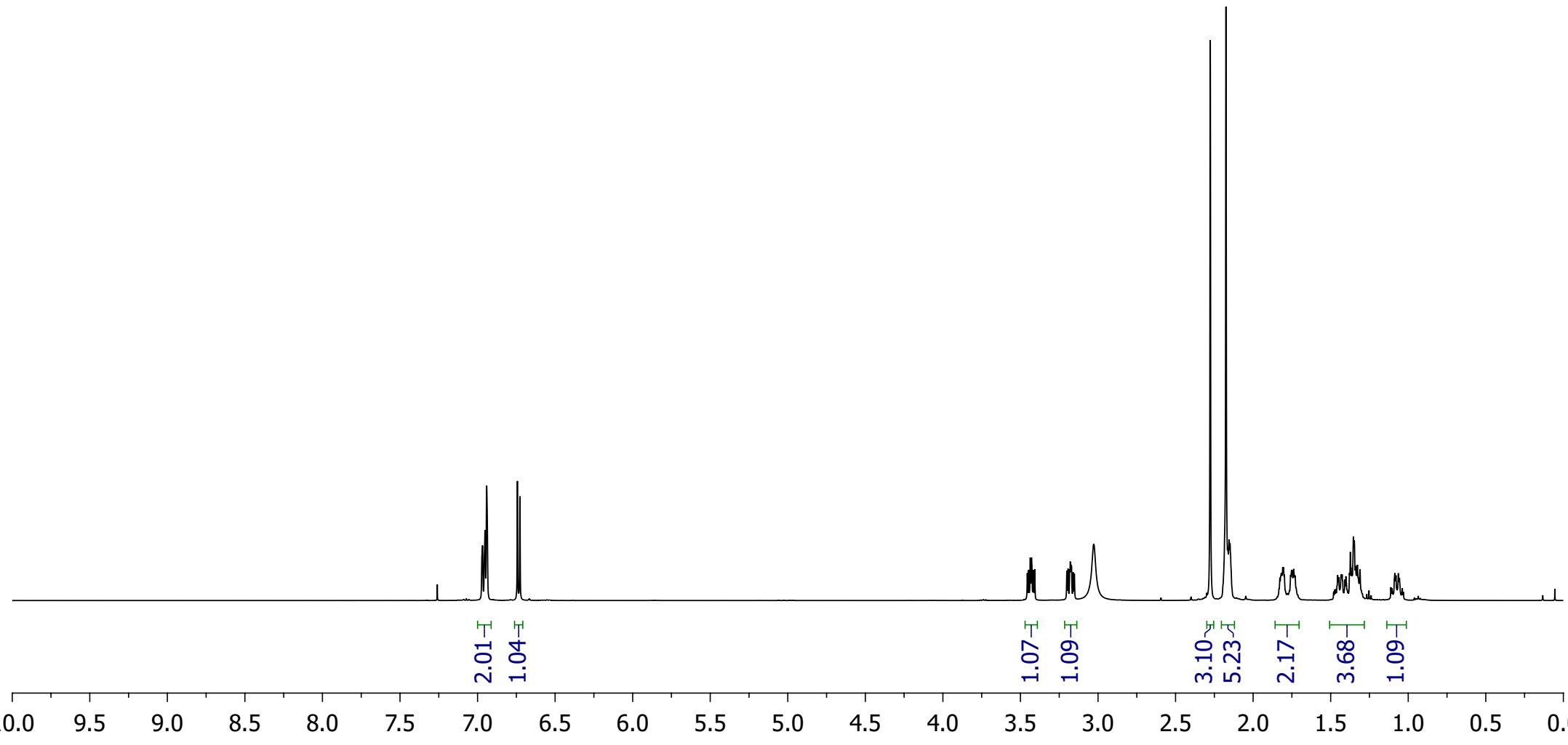


11m



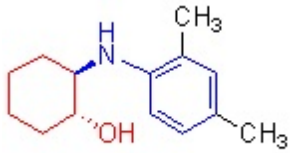
mamta
MG-261 (N)
1H_8scan CDCl3 (D:\Spectra) nmr 22

11n

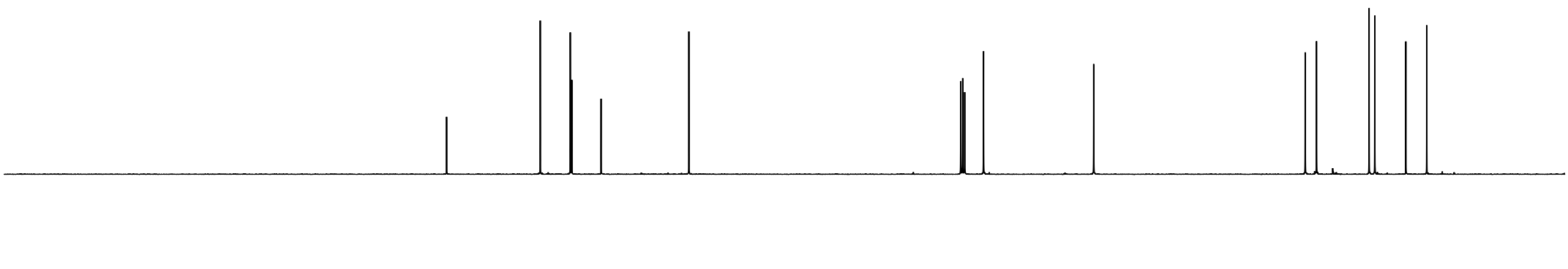


mamta
MG-161 (N)
C13CPD CDCl3 {D:\Spectra} nmr 22

143.303
131.299
127.452
127.256
123.510
112.255
77.413
77.160
76.905
74.493
60.372
33.263
31.819
25.079
24.351
20.371
17.690

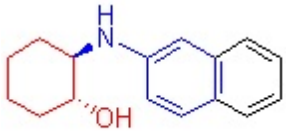


11n

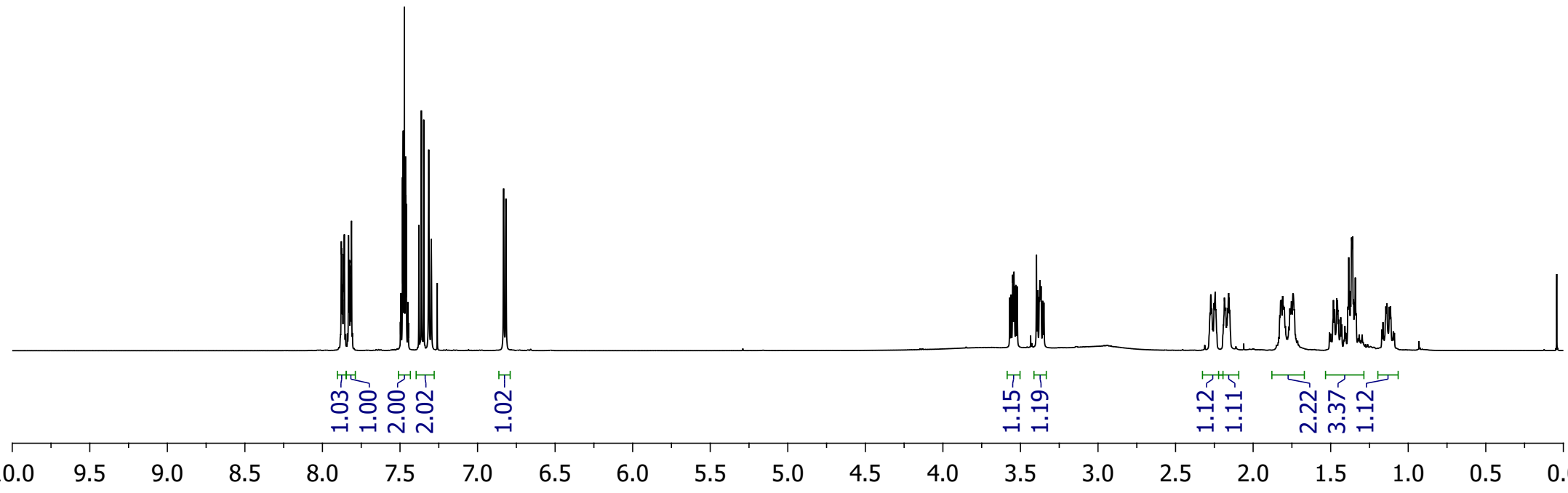


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

mamta
MG-264 (N)
1H_8scan CDCl3 (D:\Spectra) nmr 23

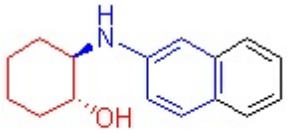


11o

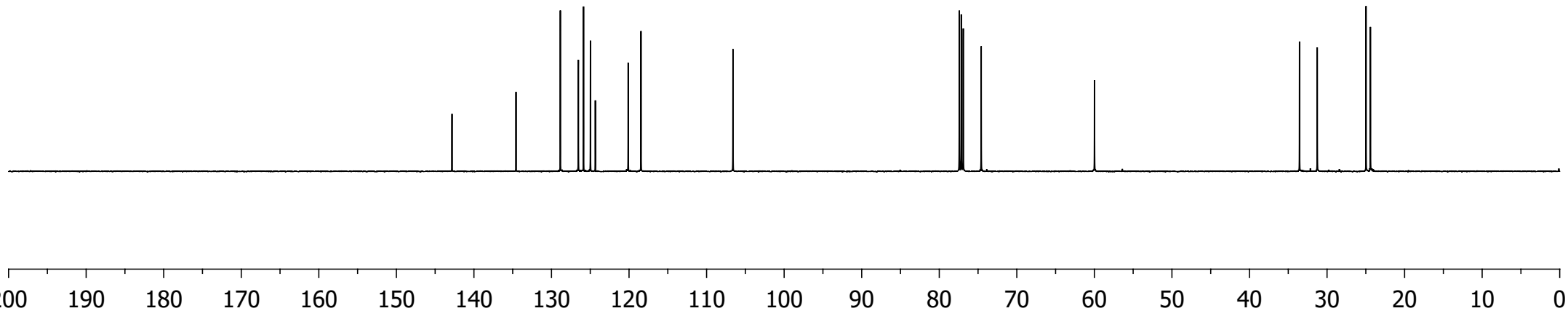


mamta
MG-164 (N)
C13CPD CDCl3 {D:\Spectra} nmr 23

142.825
134.573
128.857
126.533
125.864
124.976
124.345
120.108
118.458
106.596
77.413
77.160
76.905
74.591
59.983
33.548
31.262
24.972
24.405

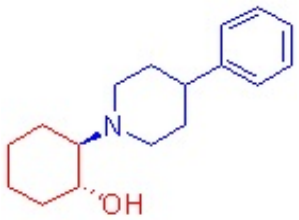


11o

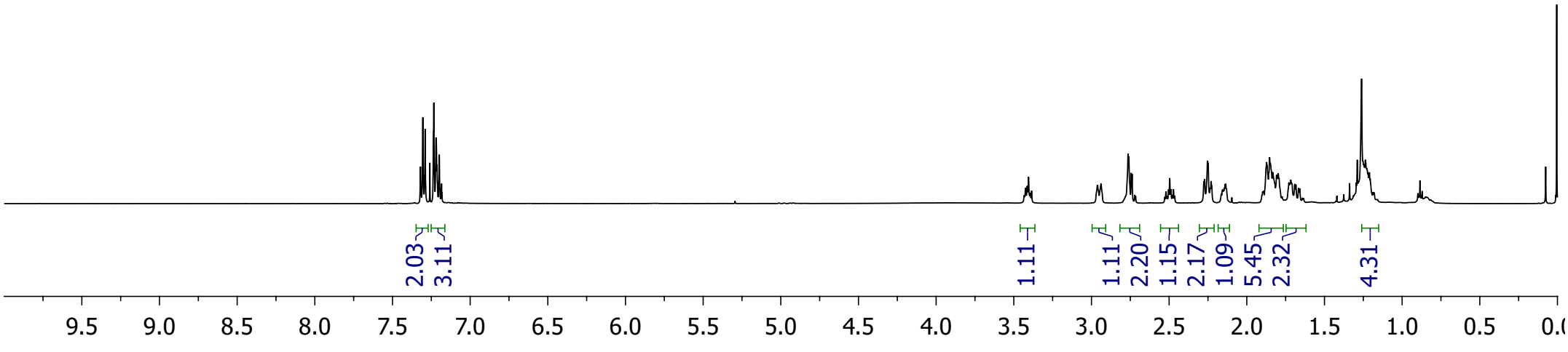


mamta
MG-185
1H_8scan CDCl3 D:\Spectra\ nmr 1

7.305 7.293 7.290 7.260 7.236 7.234 7.220 7.218 7.213 7.199 7.195 7.187 7.184 7.182 3.425 3.414 3.405 3.385 2.961 2.939 2.765 2.759 2.744 2.738 2.520 2.504 2.496 2.488 2.472 2.276 2.271 2.253 2.249 2.235 2.230 2.226 2.156 2.150 2.144 2.139 2.136 2.131 1.898 1.895 1.890 1.872 1.869 1.858 1.853 1.833 1.808 1.804 1.798 1.792 1.729 1.723 1.717 1.712 1.692 1.684 1.666 1.659

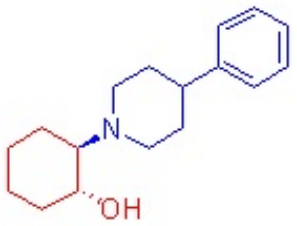


11p

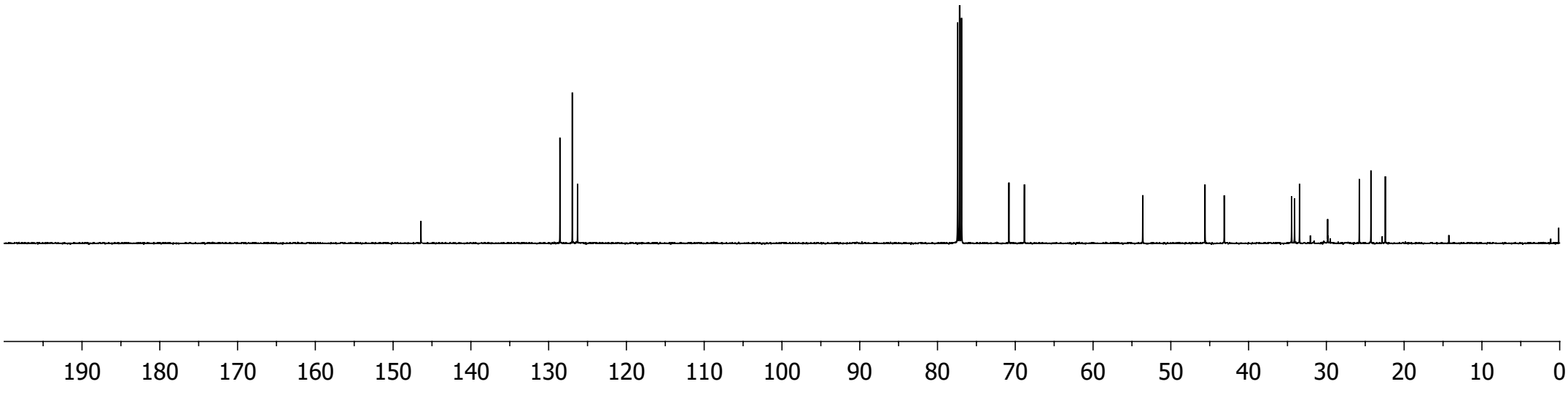


mamta
MG-185
C13CPD CDCl3 {D:\Spectra} nmr 1

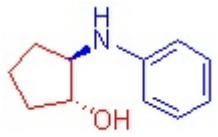
146.418
128.547
126.959
126.283
77.414
77.160
76.907
70.817
68.831
53.609
45.606
43.132
34.470
34.088
33.453
25.763
24.269
22.419



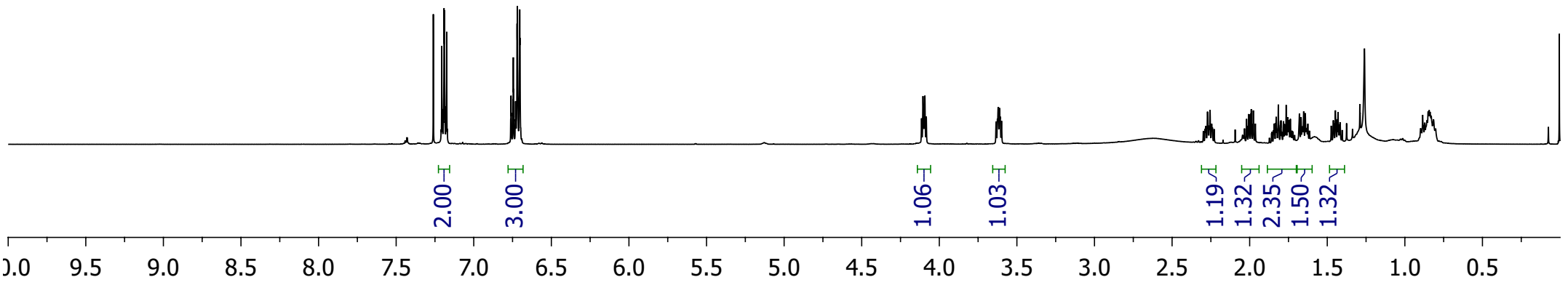
11p



MAMTA
MG-169
1H_8scan CDCl3 (D:\Spectra) nmr 1

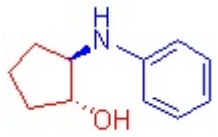


15a

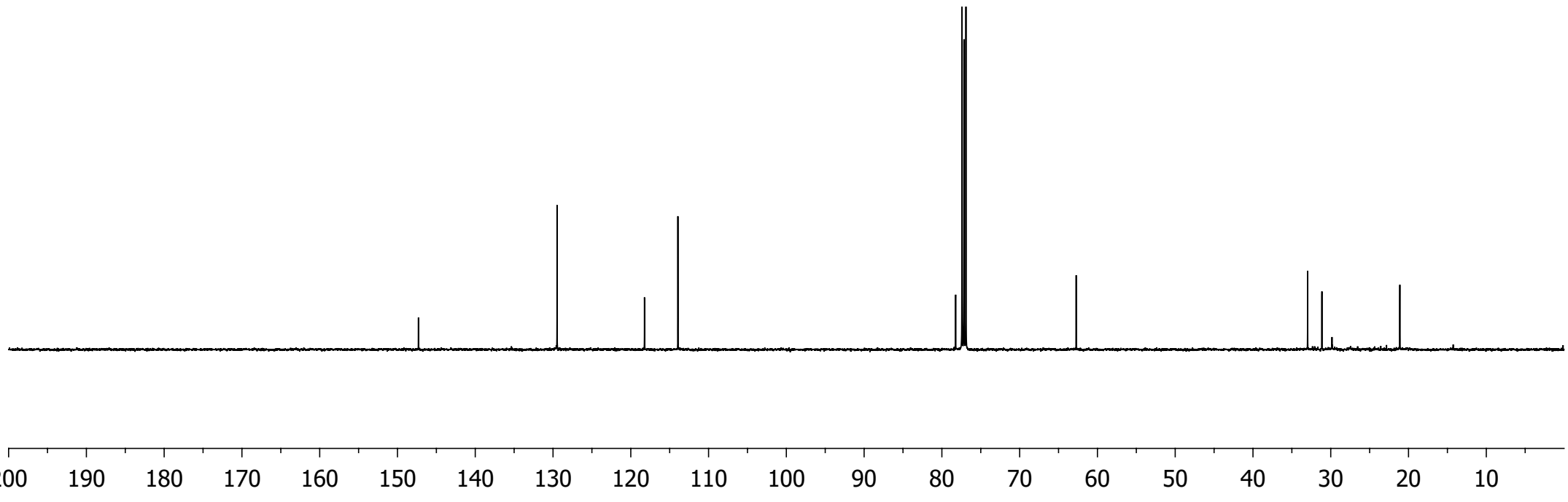


MAMTA
MG-169
C13CPD CDCl3 {D:\Spectra} nmr 1

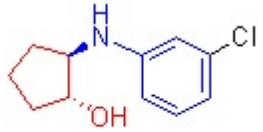
147.302
129.479
118.233
113.940
78.229
77.415
77.160
76.906
62.731
32.974
31.136
21.113



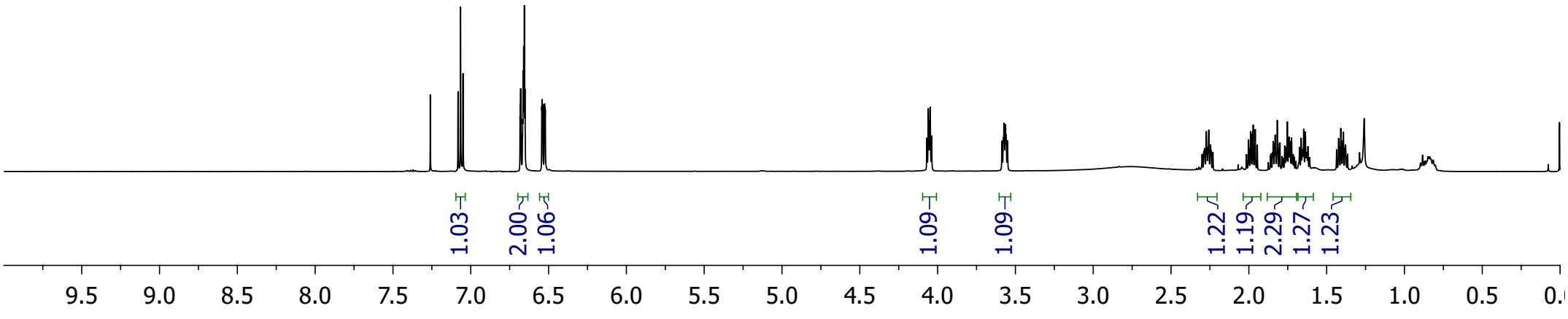
15a



MAMTA
MG-170
1H_8scan CDCl3 (D:\Spectra\ nmr) 2

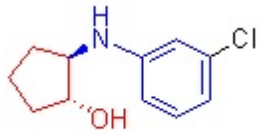


15b

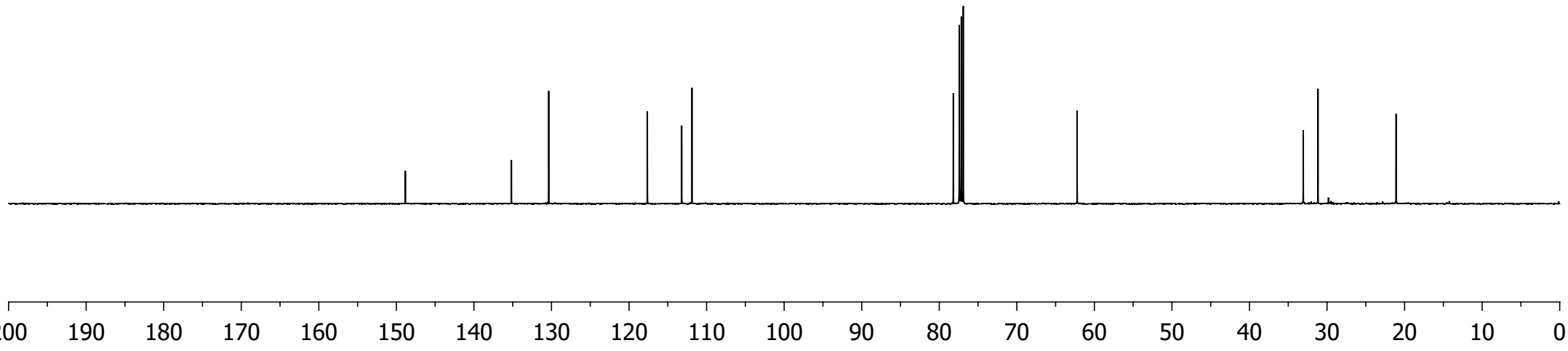


MAMTA
MG-170
C13CPD CDCl3 {D:\Spectra} nmr 2

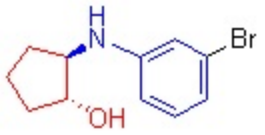
148.837
135.162
130.354
117.636
113.212
111.885
78.192
77.416
77.160
76.906
62.222
33.062
31.161
21.097



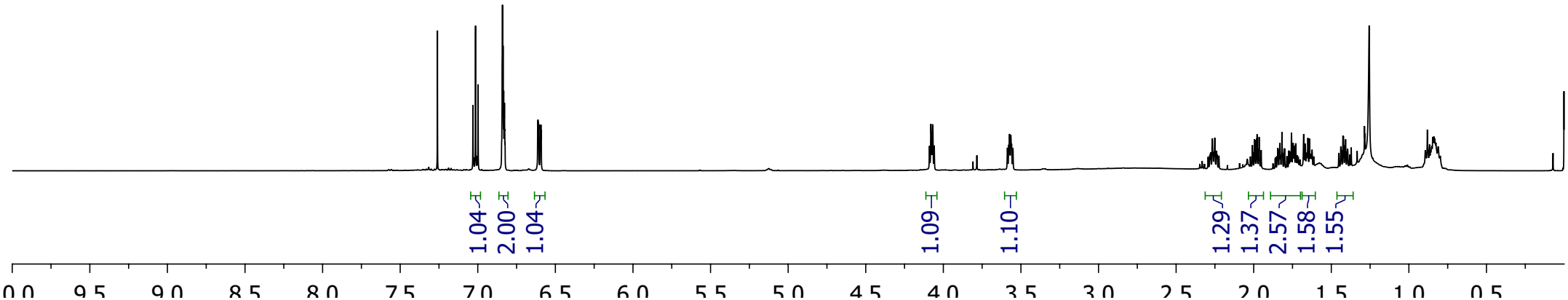
15b



MAMTA
Ma-171
1H_8scan CDCl3 D:\Spectra\ nmr 4

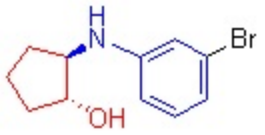


15c

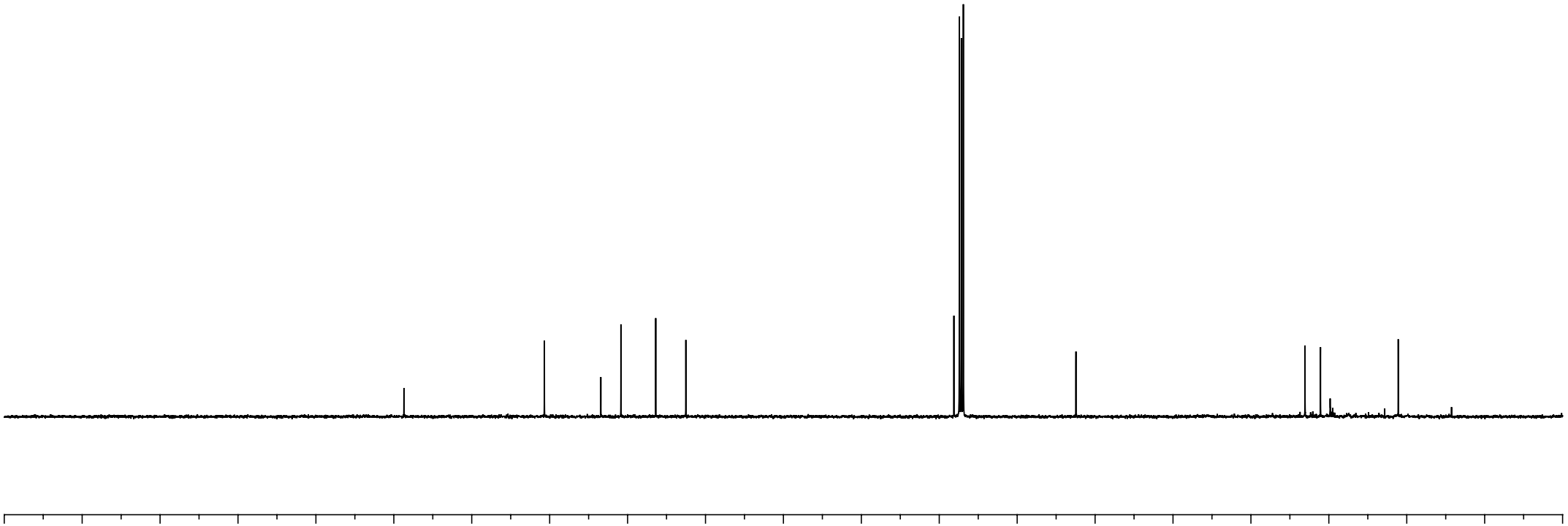


MAMTA
MG-171
C13CPD CDCl3 {D:\Spectra} nmr 4

148.699
130.678
123.433
120.853
116.387
112.521
78.118
77.416
77.160
76.906
62.456
33.059
31.073
21.092

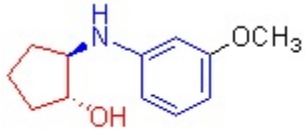


15c



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

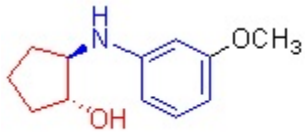
MAMTA
Mg-173
1H_8scan CDCl3 (D:\Spectra) nmr3



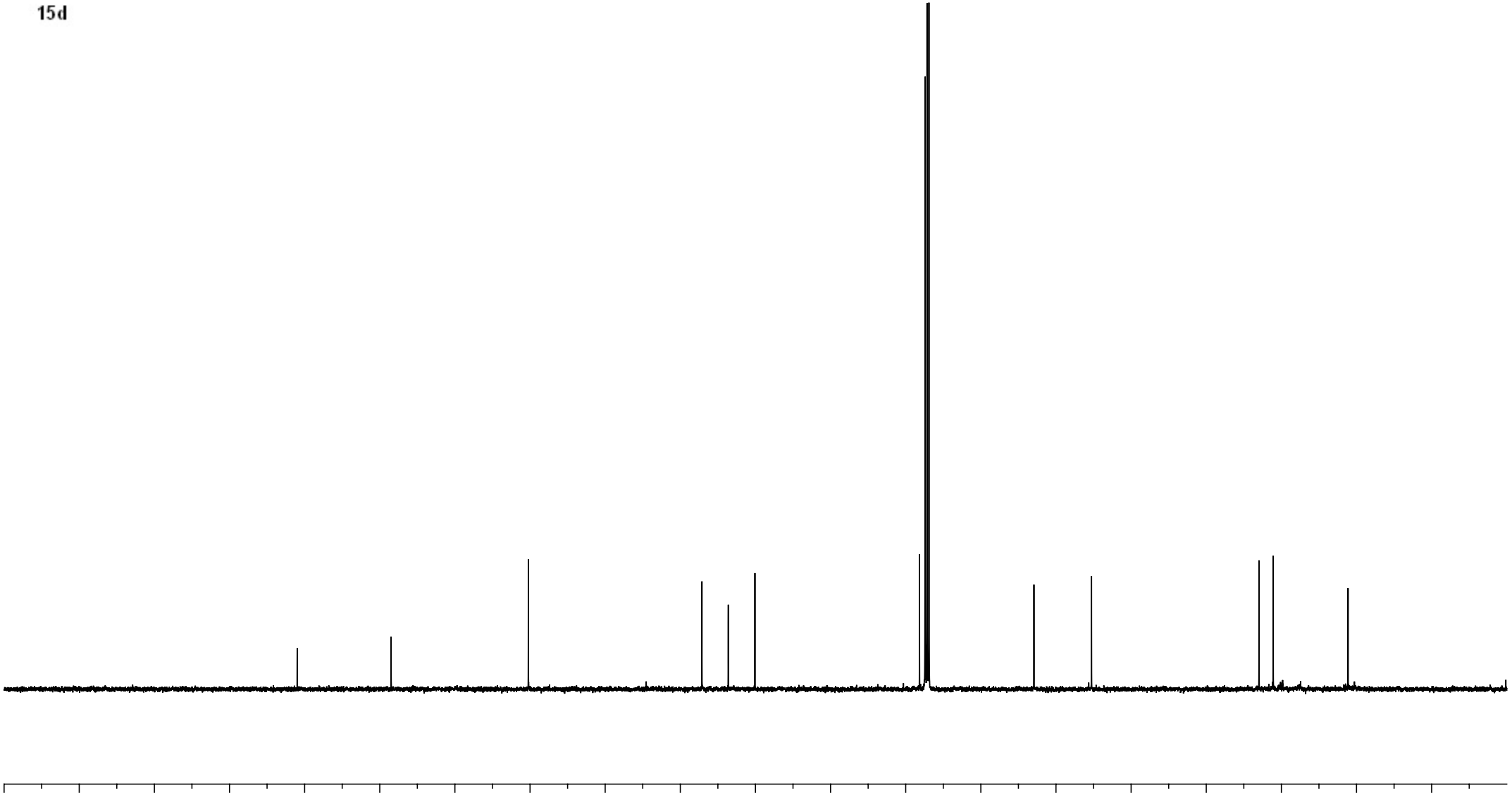
15d

MAMTA
MG-173
C13CPD CDCl3 {D:\Spectra} nmr 3

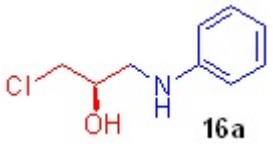
160.986
148.492
130.220
107.123
103.611
100.068
78.166
77.414
77.160
76.907
62.932
55.274
32.977
31.090
21.125



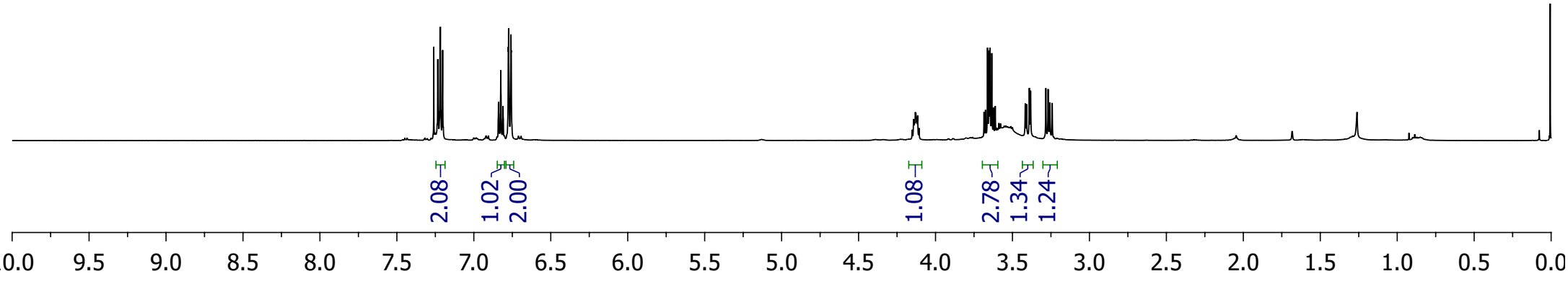
15d



mamta
MG-175
1H_8scan CDCl3 {D:\Spectra} nmr 13

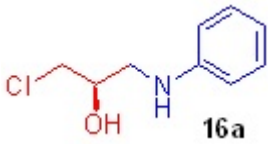


16a

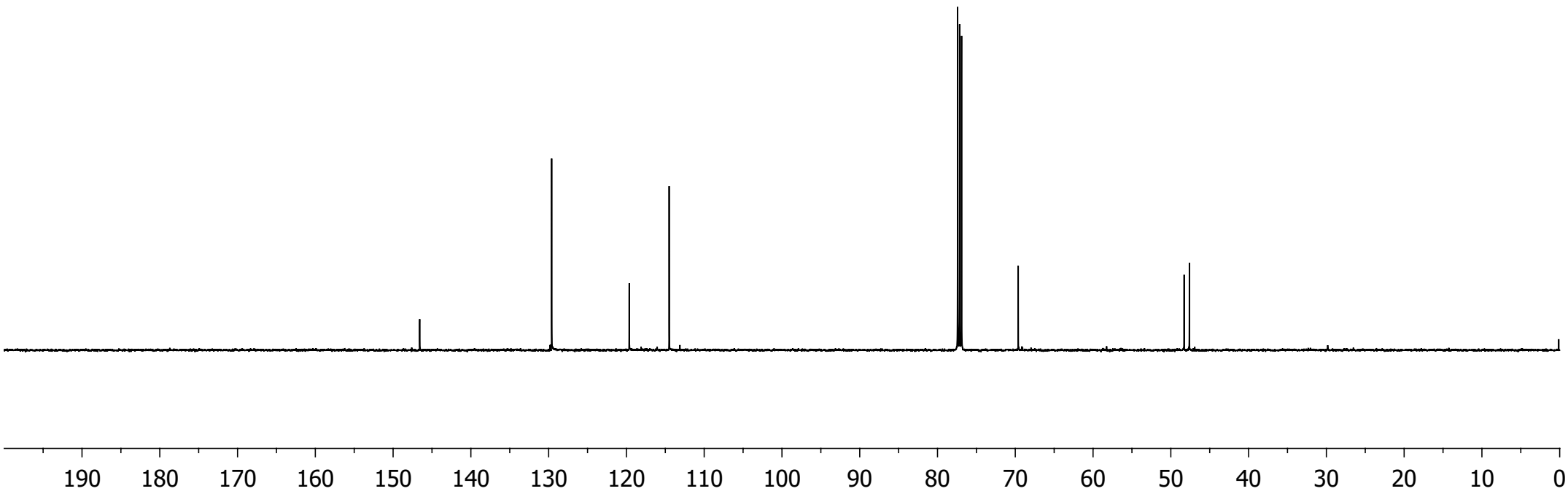


mamta
MG-175
C13CPD CDCl3 {D:\Spectra} nmr 13

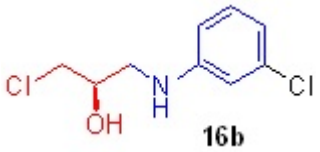
146.583
129.613
119.639
114.504
77.414
77.160
76.905
69.621
48.287
47.611



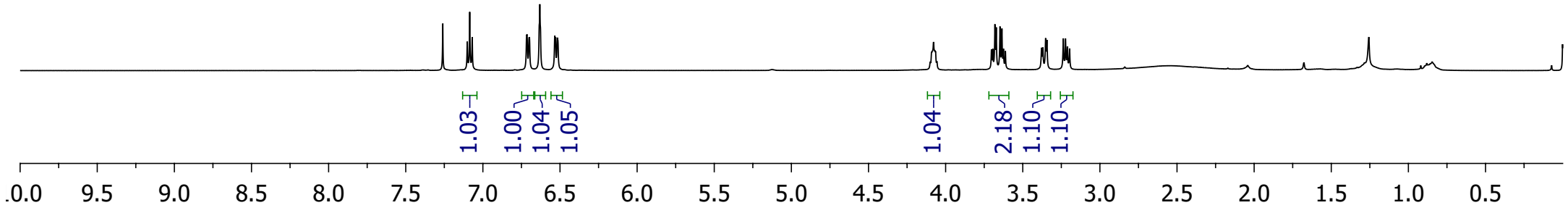
16a



mamta
MG-176
1H_8scan CDCl3 {D:\Spectra} nmr 9

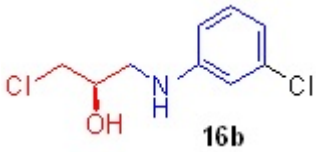


16b

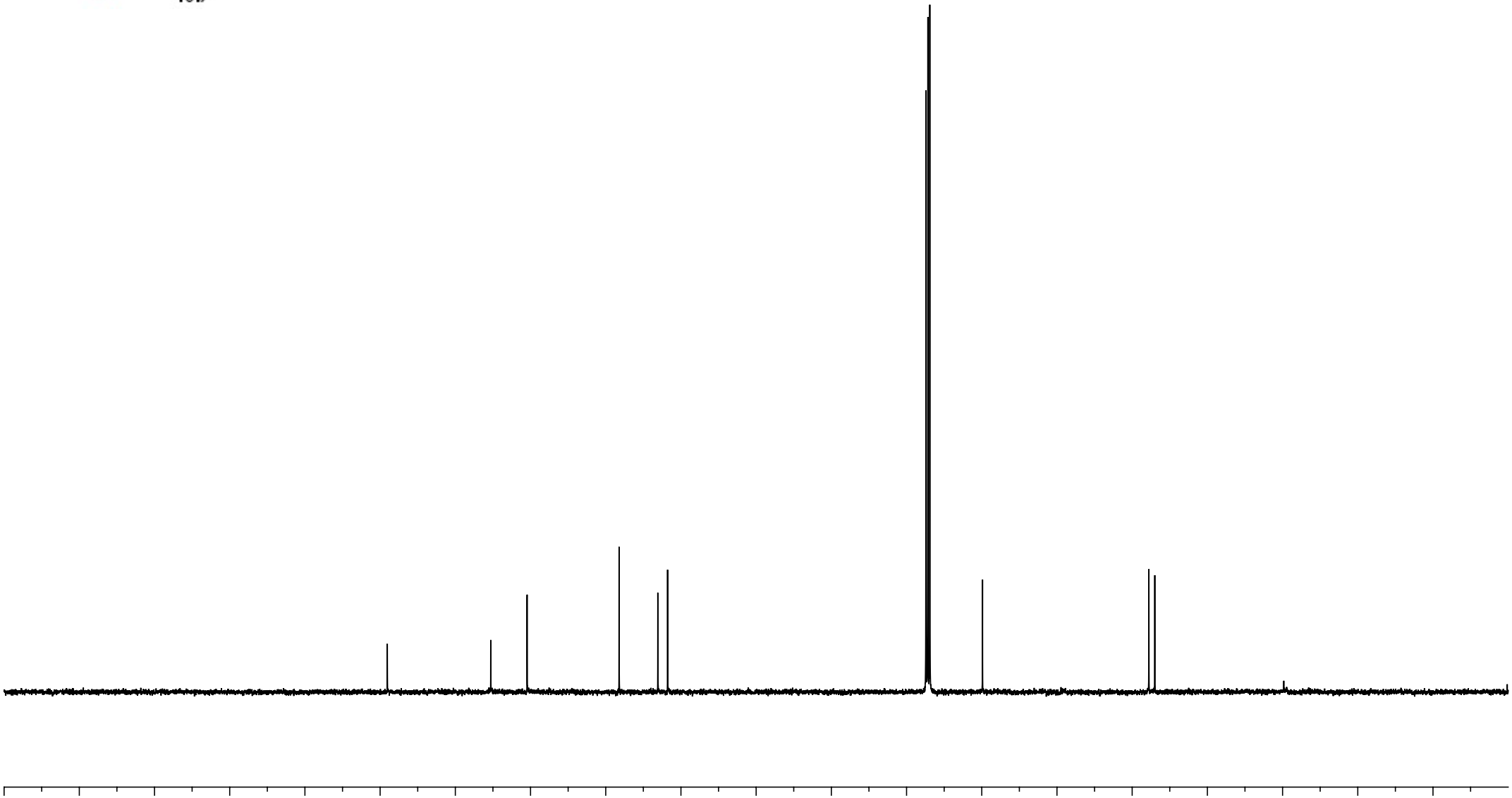


mamta
MG-176
C13CPD CDCl3 {D:\Spectra} nmr 9

149.047
135.288
130.458
118.222
113.069
111.777
77.415
77.160
76.907
69.899
47.779
46.988

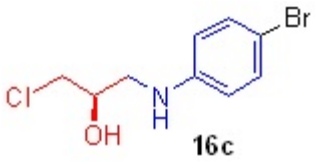


16b

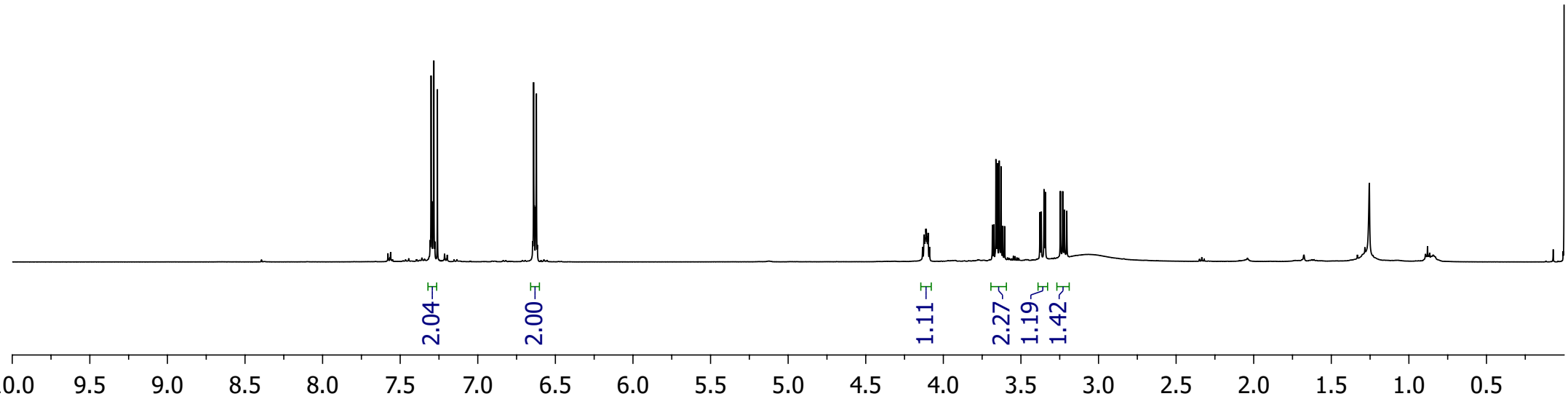


mamta
MG-177
1H_8scan CDCl3 {D:\Spectra} nmr 14

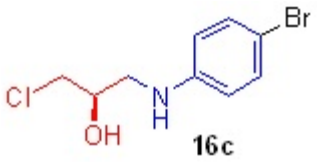
7.307 7.301 7.297 7.287 7.283 7.277 7.260 6.646 6.640 6.636 6.627 6.622 6.616
4.133 4.125 4.122 4.118 4.116 4.113 4.110 4.107 4.104 4.101 4.098 4.089
3.684 3.674 3.661 3.652 3.640 3.628 3.617 3.605 3.377 3.369 3.351 3.343 3.246 3.231 3.219 3.204



16c



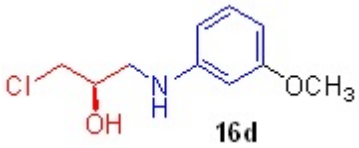
mamta
MG-177
C13CPD CDCl3 {D:\Spectra} nmr 14



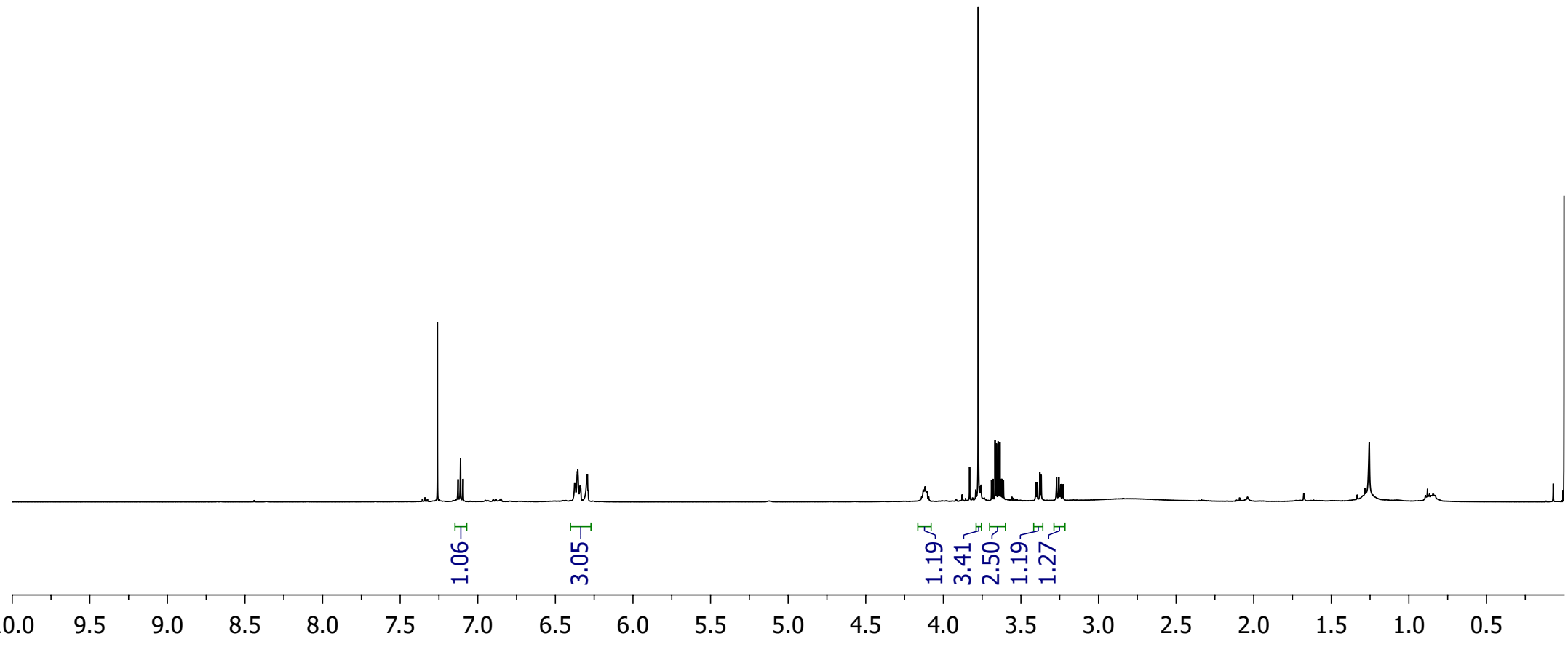
16c

145.595
132.212
115.837
111.245
77.275
77.022
76.768
69.457
47.993
47.437

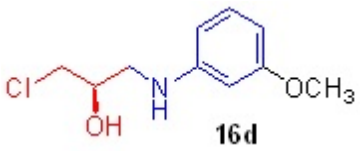
mamta
MG-178
1H_8scan CDCl3 {D:\Spectra} nmr 15



16d



mamta
MG-178
C13CPD CDCl3 {D:\Spectra} nmr 15

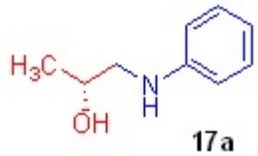


16d

161.035
130.378
107.204
104.516
100.309
77.414
77.160
76.905
69.703
55.332
47.676
29.842

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

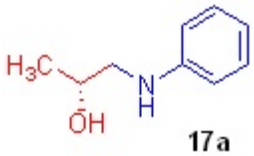
MAMTA
MG-179
1H_8scan CDCl3 {D:\Spectra} nmr 8



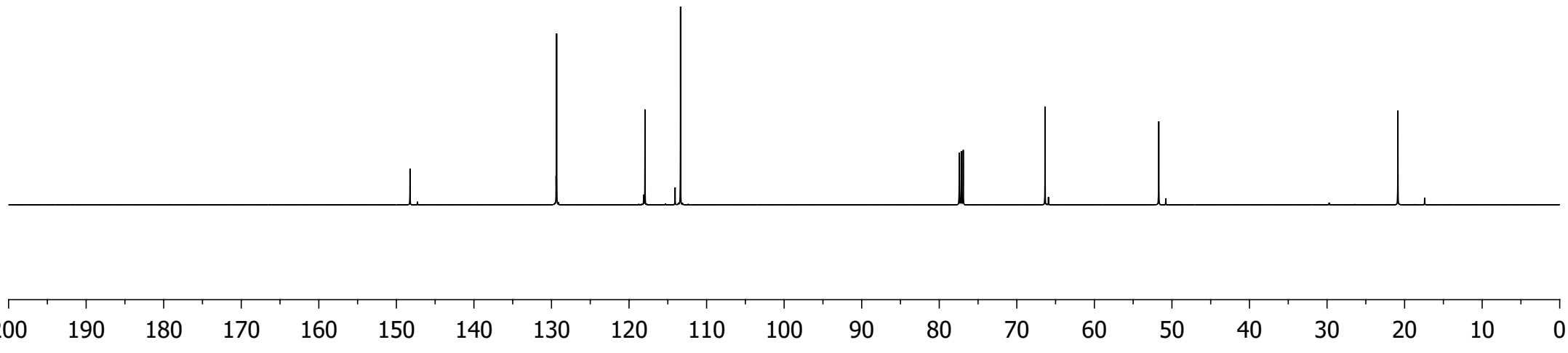
17a

MAMTA
MG-179
C13CPD CDCl3 {D:\Spectra} nmr 8

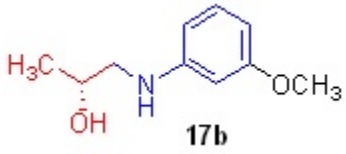
148.237
129.334
117.941
113.358
77.416
77.160
76.906
66.355
51.696
20.863



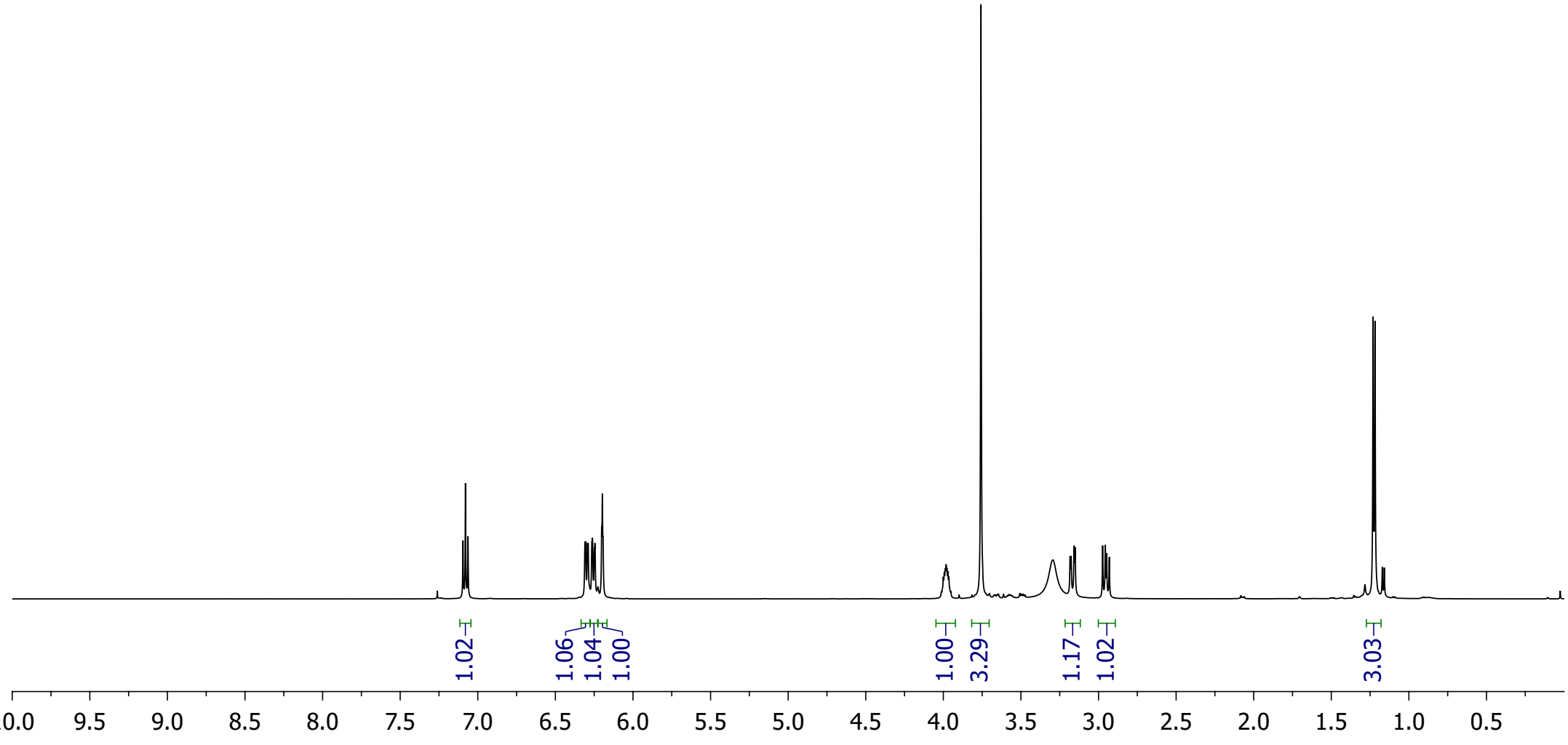
17a



MAMTA
MG-182
1H_8scan CDCl3 {D:\Spectra} nmr 3

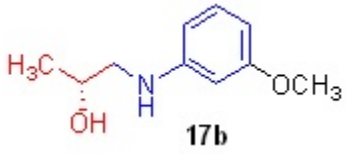


17b



mamta
MG-182
C13CPD CDCl3 {D:\Spectra} nmr 12

160.980
148.810
130.250
107.156
104.073
100.156
77.415
77.160
76.905
66.307
55.290
52.491
29.830
20.956



17b