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Supporting Information
for DOI: 10.1055/s-0036-1588559

Thieme

Supporting Information

General Information

Commercial reagents were purchased from Fisher Chemicals, Sigma-Aldrich, J.T. Baker Chemical Company, and Acros Organics and used without purification. Acetonitrile (MeCN) and dichloromethane (DCM) solvents were purified using a J. C. Meyer Designed Solvent Dispensing System coupled with J-Kem Scientific Model 280 Digital Vacuum Regulator. All reactions were performed in the fume hood under atmospheric pressure, unless otherwise noted, and reaction products were stored in vials at ambient temperature.

Reactions were monitored by thin-layer chromatography (TLC) on EMD Silica Gel 60 F254 plates under UV light (254 nm) or visualized with $KMnO_4$ or anisaldehyde. Flash chromatography was performed using silica gel 60 (230-400 mesh) from EM Science on a Biotage SP4 system. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator R-200. Proton nuclear magnetic resonance (^1H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker Magnet System 400MHz. All chemical shifts are reported in parts per million (ppm) downfield from tetramethylsilane. Proton resonances are referenced to residual protium in the NMR solvent (7.26 ppm for $CHCl_3$, 2.50 ppm for d_6-DMSO, 3.31 ppm for d_4-methanol, and 7.16 ppm for d_6-benzene). Carbon resonances are referenced to the carbon resonances of the NMR solvent (77.00 ppm for $CHCl_3$, 39.52 ppm for d_6-DMSO, 49.00 ppm for d_4-methanol, and 128.06 ppm for d_6-benzene). Data are represented as follows: chemical shift, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. Infrared (IR) spectra were obtained using a Bruker Alpha-P spectrometer equipped with an attenuated total reflectance (ATR) single reflection unit. Mass spectral (MS) data were obtained on a JEOL AccuTOF T100LP Mass Spectrometer equipped with a DART (Direct Analysis in Real Time) ionization module.

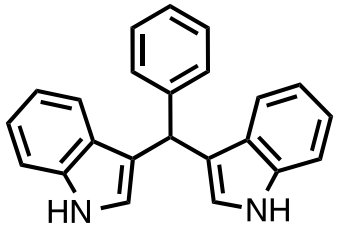
General procedure for liquid aldehydes with malachite green as catalyst:
Indole (23.4 mg, 0.20 mmol), and malachite green (1.8 mg, 0.005 mmol) were charged to a 1 dram vial equipped with a stir bar. Dichloromethane (0.5 mL, 0.2M) was added, followed by the aldehyde (0.11 mmol) and the reaction was then heated to 35 ºC. The reaction was stirred and the progress of the reaction was followed by TLC. The crude reaction was then chromatographed to isolate the product.

General procedure for solid aldehydes with malachite green as catalyst:
Indole (23.4 mg, 0.20 mmol), aldehyde (0.11 mmol), and malachite green (1.8 mg, 0.005 mmol) were charged to a 1 dram vial equipped with a stir bar. Dichloromethane (0.5 mL, 0.2M) was added and the reaction was then heated to 35 ºC. The reaction was stirred and the progress of the reaction was followed by TLC. The crude reaction was then chromatographed to isolate the product.

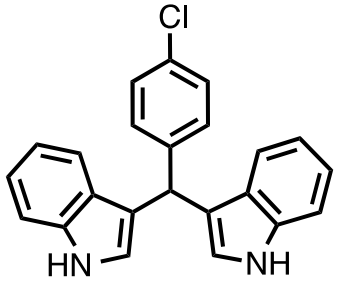
General procedure for liquid aldehydes with other triaryl catalysts:
Triaryl alcohol (0.006 mmol) was added to a 1 dram vial equipped with a stir bar and a cap fitted with a septum. Dichloromethane (0.2 mL) was added, followed by tetrafluoroboric acid diethyl etherate complex (0.7 μL, 0.005 mmol) to give a bright color, which depended on the identity of

the catalyst. Aldehyde (0.11 mmol) was then added via syringe, followed by indole (23.4 mg, 0.20 mmol) as a solution in dichloromethane (0.2 mL, and a 0.1 mL rinse). The reaction was heated to 35 ºC. The reaction was stirred and the progress of the reaction was followed by TLC. The crude reaction was then chromatographed to isolate the product.

General procedure for solid aldehydes with other triaryl catalysts:
Triaryl alcohol (0.006 mmol) was added to a 1 dram vial equipped with a stir bar and a cap fitted with a septum. Dichloromethane (0.2 mL) was added, followed by tetrafluoroboric acid diethyl etherate complex (0.7 μL, 0.005 mmol) to give a bright color, which depended on the identity of the catalyst. Aldehyde (0.11 mmol) and indole (23.4 mg, 0.20 mmol) as a solution in dichloromethane (0.2 mL, and a 0.1 mL rinse). The reaction was heated to 35 ºC. The reaction was stirred and the progress of the reaction was followed by TLC. The crude reaction was then chromatographed to isolate the product.

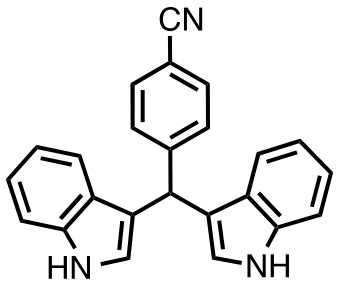


3,3'-(phenylmethylene)bis(1*H*-indole) 8a: The reaction time was 5 hours and a 15-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red foam (29.5 mg, 99% yield). The spectral values were in accord with literature values.[1] IR (thin film) 3389, 3051, 3020, 1597, 1490, 1455, 1419, 1352, 1336, 1260, 1218, 1176, 1123, 1090, 1057, 1041, 1006, 927, 828, 798, 763, 746, 729, 699, 620, 601, 593, 582, 495, 463, 441, 421 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.86 (br s, 2H), 7.40 (d, *J* = 7.9 Hz, 2H), 7.33-7.38 (m, 4H), 7.21-7.32 (m, 3 H), 7.18 (dt, *J* = 1.0, 7.1 Hz, 2H), 7.02 (dt, *J* = 0.7, 7.9 Hz, 2H), 6.63 (s, 2H), 5.90 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 144.0, 136.6, 128.7, 128.2, 127.0, 126.1, 123.6, 121.9, 119.9, 119.7, 119.2, 111.0, 40.1 ppm. DART-MS-TOF m/z calculated for $C_{23}H_{18}N_2$ (M+) = 322.1470, found 322.1468.

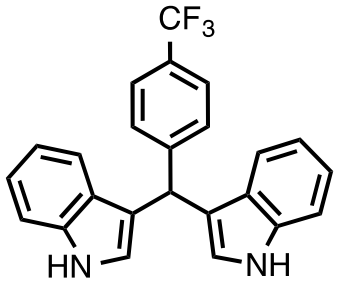


3,3'-((4-chlorophenyl)methylene)bis(1H-indole) 8b: The reaction time was 2 hours and a 15-35% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red foam (33.8 mg, 95% yield). The spectral values were in accord with literature values.[5] IR IR (thin film) 3408, 3056, 1617, 1487, 1455, 1416, 1336, 1214, 1150, 1124, 1088, 1038, 1012, 908, 857, 786, 738, 666, 597, 579, 559, 480, 453, 421 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.92 (br s, 2H), 7.35-7.42 (m, 4H), 7.24-7.31 (m, 4H), 7.22 (t, *J* = 7.9 Hz, 2H), 7.05 (t, *J* = 7.6 Hz, 2H), 6.63 (s, 2H), 5.89 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 142.5, 136.5,

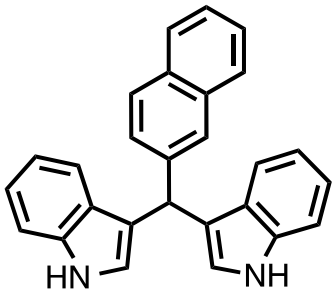
131.6, 130.0, 128.3, 126.7, 123.6, 122.0, 119.7, 119.2, 118.9, 111.1, 39.5 ppm. DART-MS-TOF m/z calculated for $C_{23}H_{17}ClN_2$ (M+) = 356.1080, found 356.1079.



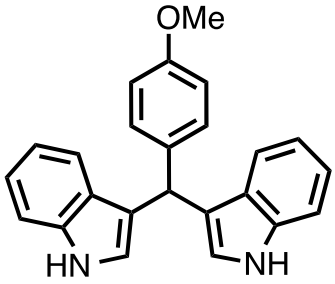
4-(di(1*H*-indol-3-yl)methyl)benzonitrile 8c: The reaction time was 30 minutes and a 15-45% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a pink foam (34.5 mg, 99.9% yield). The spectral values were in accord with literature values.[6–8] IR (thin film) 3409, 3056, 2227, 1605, 1499, 1487, 1456, 1418, 1338, 1241, 1217, 1124, 1095, 1038, 1010, 866, 791, 744, 667, 601, 582, 552, 506, 424 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$), δ 8.02 (br s, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.20 (dt, *J* = 1.1, 7.0 Hz, 2H), 7.02 (dt, *J* = 0.9, 7.1 Hz, 2H), 6.67 (s, 2H), 5.94 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 149.7, 136.6, 132.2, 129.5, 126.7, 123.6, 122.3, 119.6, 119.5, 119.2, 118.2, 111.2, 110.0, 40.3 ppm. DART-MS-TOF m/z calculated for $C_{24}H_{17}N_3$ (M+) = 347.1422, found 347.1423.



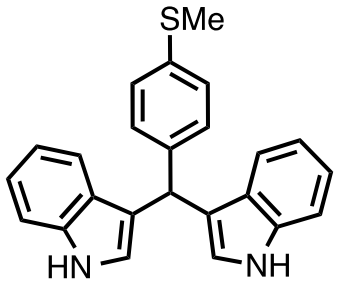
3,3'-((4-(trifluoromethyl)phenyl)methylene)bis(1*H*-indole) 8d: The reaction time was 30 minutes and a 15-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red foam (34.6 mg, 91% yield). The spectral values were in accord with literature values.[9] IR (thin film) 3409, 3056, 1616, 1487, 1456, 1417, 1321, 1241, 1216, 1162, 1118, 1106, 1065, 1038, 1017, 930, 866, 795, 740, 669, 626, 601, 579, 470, 424 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$), δ 7.95 (br s, 2H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.35-7.39 (m, 4H), 7.20 (t, *J* = 7.0 Hz, 2H), 7.03 (t, *J* = 7.7 Hz, 2H), 6.64 (s, 2H), 5.95 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 148.1, 136.6, 128.9, 128.4 (q, *J* = 32.1 Hz), 126.7, 125.2 (q, *J* = 2.8 Hz), 124.4 (q, *J* = 271.9 Hz), 123.7, 122.1, 119.6, 119.4, 118.6, 111.2, 40.0 ppm. DART-MS-TOF m/z calculated for $C_{24}H_{17}F_3N_2$ (M+) = 390.1344, found 390.1343.



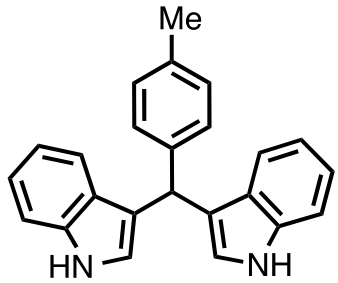
3,3'-(naphthalen-2-ylmethylene)bis(1*H*-indole) 8e: The reaction time was 1 hour and a 2-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red foam (34.7 mg, 94% yield). The spectral values were in accord with literature values.[4] IR (thin film) 3411, 3053, 2972, 2917, 2849, 1617, 1599, 1507, 1484, 1456, 1413, 1337, 1242, 1202, 1124, 1093, 1043, 1011, 800, 743, 478, 423 cm^{-1}. ^{1}H NMR (400 MHz, d_6-DMSO), δ 10.88 (s, 2H), 7.74-7.87 (m, 4H), 7.57 (d, *J* = 8.5 Hz, 1H), 7.40-7.46 (m, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 2H), 6.90 (s, 2H), 6.86 (t, *J* = 7.9 Hz, 2H), 6.03 (s, 1H) ppm. ^{13}C NMR (126 MHz, d_6-DMSO), δ 142.7, 136.6, 133.1, 131.7, 127.6, 127.6, 127.5, 127.5, 126.7, 125.9, 125.3, 123.8, 121.0, 119.1, 118.3 (2), 117.9, 111.5, 39.8 ppm. DART-MS-TOF m/z calculated for $C_{27}H_{20}N_2$ (M+) = 372.1626, found 372.1622.



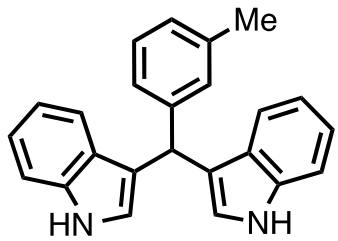
3,3'-((4-methoxyphenyl)methylene)bis(1H-indole) 8f: The reaction time was 3 hours and a 15-40% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as an orange foam (33.7 mg, 96% yield). The spectral values were in accord with literature values.[2] IR (thin film): 3401, 3054, 2932, 2833, 1609, 1507, 1484, 1455, 1414, 1336, 1300, 1242, 1218, 1172, 1149, 1123, 1092, 1029, 1010, 907, 849, 815, 791, 737, 647, 583, 541, 490, 423 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.91 (br s, 2H), 7.39 (d, *J* = 7.9 Hz, 2H), 7.35 (d, *J* = 8.2 Hz, 2H), 7.23-7.27 (m, 2H), 7.17 (dt, *J* = 1.0, 7.1 Hz, 2H), 7.00 (dt, *J* = 0.9, 8.0 Hz, 2H), 6.82 (d, *J* =8.7 Hz, 2H), 6.65 (s, 2H), 5.84 (s, 1H), 3.78 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 157.9, 136.7, 136.2, 129.6, 127.0, 123.5, 121.9, 120.0, 120.0, 119.2, 113.5, 111.0, 55.2, 39.3 ppm. DART-MS-TOF m/z calculated for $C_{24}H_{20}N_2O$ (M+) = 352.1576, found 352.1574.



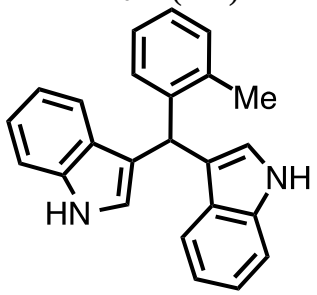
3,3'-((4-(methylthio)phenyl)methylene)bis(1*H*-indole) 8g: The reaction time was 5 hours and a 10-35% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red oil (32.5 mg, 90% yield). The spectral values were in accord with literature values.[3] IR (thin film) 3411, 3054, 2972, 2919, 1618, 1587, 1490, 1456, 1416, 1337, 1238, 1216, 1150, 1124, 1092, 1040, 1011, 967, 876, 857, 785, 743, 599, 580, 484, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.86 (br s, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.15-7.21 (m, 4H), 7.02 (t, *J* = 7.1 Hz, 2H), 6.60 (s, 2H), 5.85 (s, 1H), 2.46 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 141.1, 136.6, 135.5, 129.2, 126.9, 126.6, 123.6, 121.9, 119.8, 119.4, 119.2, 111.1, 39.6, 15.9 ppm. DART-MS-TOF m/z calculated for $C_{24}H_{20}N_2S$ (M+) = 368.1347, found 368.1345.



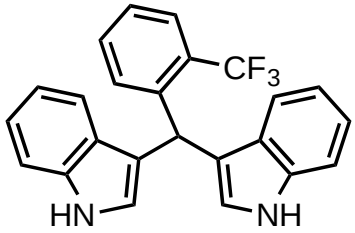
3,3'-(*p*-tolylmethylene)bis(1H-indole) 8h: The reaction time was 1 hour and a 15-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red foam (30.3 mg, 91% yield). The spectral values were in accord with literature values.[2] IR (thin film): 3414, 3053, 2919, 2853, 1678, 1606, 1510, 1483, 1456, 1413, 1337, 1238, 1203, 1125, 1092, 1038, 1010, 908, 859, 777, 737, 648, 598, 582, 531, 489, 458, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.82 (s, 2H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 6.2 Hz, 2H), 7.18 (t, *J* = 8.2 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 2H), 7.02 (t, *J* = 7.0 Hz, 2H), 6.62 (d, *J* = 1.4 Hz, 2H), 5.87 (s, 1H), 2.34 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$), δ 140.9, 136.6, 135.4, 128.9, 128.5, 127.1, 123.5, 121.8, 119.9, 119.8, 119.1, 111.0, 39.7, 21.1 ppm. DART-MS-TOF m/z calculated for $C_{24}H_{20}N_2$ (M+) = 336.1626, found 336.1630.



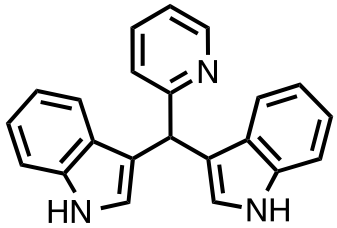
3,3'-(*m*-tolylmethylene)bis(1H-indole) 8i: The reaction time was 2 hours and a 15-35% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a peach foam (33.5 mg, 99.7% yield). The spectral values were in accord with literature values.[12] IR (thin film): 3410, 3054, 2918, 1686, 1605, 1518, 1485, 1455, 1412, 1337, 1271, 1239, 1201, 1124, 1093, 1041, 1010, 908, 770, 734, 701, 648, 624, 600, 579, 461, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.84 (br s, 2H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 7.1 Hz, 2H), 7.11-7.18 (m, 3H), 6.98-7.06 (m, 3H), 6.64 (s, 2H), 5.85 (s, 1H), 2.30 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 143.9, 137.6, 136.6, 129.4, 128.0, 127.1, 126.9, 125.7, 123.6, 121.8, 119.9, 119.8, 119.2, 111.0, 40.1, 21.5 ppm. DART-MS-TOF m/z calculated for $C_{27}H_{26}N_2$ (M+) = 336.1626, found 336.1626.



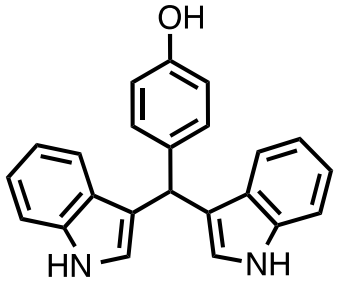
3,3'-(*o*-tolylmethylene)bis(1H-indole) 8j: The reaction time was 21 hours and a 15-30% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a white foam (34.1 mg, 99.4% yield). The spectral values were in accord with literature values.[13] IR (thin film): 3412, 3056, 2916, 2849, 1699, 1601, 1519, 1481, 1456, 1408, 1335, 1199, 1123, 1093, 1037, 1010, 907, 834, 785, 733, 648, 620, 600, 580, 449, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.89 (br s, 2H), 7.36 (d, *J* = 8.8 Hz, 4H), 7.03-7.23 (m, 6H), 7.01 (dt, *J* = 1.0, 7.8 Hz, 2H), 6.58 (s, 2H), 6.03 (s, 1H), 2.39 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 142.0, 136.7, 136.0, 130.1, 128.4, 127.2, 126.0, 125.8, 123.8, 121.9, 119.8, 119.2, 119.2, 111.0, 36.2, 19.5 ppm. DART-MS-TOF m/z calculated for $C_{27}H_{26}N_2$ (M+) = 336.1626, found 336.1629.



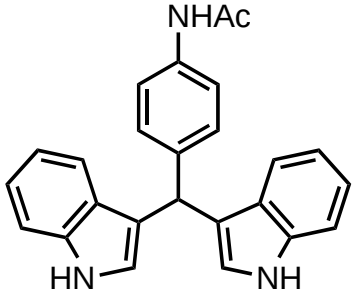
3,3'-((2-(trifluoromethyl)phenyl)methylene)bis(1*H*-indole) 8k: The reaction time was 24 hours and a 20-40% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the known title compound[10] as a white foam (36.7 mg, 95% yield). IR (thin film) 3460, 3396, 1456, 1419, 1337, 1307, 1253, 1159, 1117, 1093, 1060, 1037, 1008, 904, 790, 773, 737, 724, 668, 648, 641, 626, 599, 581, 502, 459, 425 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.73-7.81 (m, 3H), 7.30-7.50 (m, 7H), 7.19 (dt, *J* = 1.0, 7.1 Hz, 2H)), 7.03 (dt, *J* = 0.9, 8.0 Hz, 2H), 6.51 (d, *J* = 1.4 Hz, 2H), 6.32 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 142.6, 136.6, 131.6, 130.9, 128.1 (q, *J* = 29.6 Hz), 126.7, 126.3, 125.9 (q, *J* = 5.9 Hz), 124.8 (q, *J* = 274.5 Hz), 123.8, 122.0, 119.7, 119.3, 119.2, 111.0, 35.8 (q, *J* =2.1 Hz) ppm. DART-MS-TOF m/z calculated for $C_{24}H_{17}F_3N_2$ (M+) = 390.1344, found 390.1342.



3,3'-(2-pyridinylmethylene)bis-(1*H*-indole) 8l: The reaction time was 16 hours and a 40-70% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a white solid (30.5 mg, 95% yield). The spectral values were in accord with literature values.[16] IR (thin film) 3447, 3412, 3137, 3054, 2919, 2868, 1617, 1587, 1566, 1491, 1470, 1455, 1434, 1415, 1354, 1338, 1269, 1255, 1242, 1214, 1193, 1150, 1123, 1090, 1065, 1040, 1002, 926, 864, 843, 818, 796, 783, 765, 747, 736, 669, 626, 616, 603, 592, 551, 453, 425, 408 cm^{-1}. ^{1}H NMR (400 MHz, CD_3OD), δ 8.46 (d, *J* = 5.0 Hz, 1H), 7.70 (dt, *J* = 1.6, 7.6 Hz, 1H), 7.30-7.38 (m, 4H), 7.21-7.30 (m, 4H), 7.06 (t, *J* = 7.6 Hz, 2H), 6.88 (t, *J* = 7.1 Hz, 2H), 6.71 (s, 2H), 6.00 (s, 1H) ppm. ^{13}C NMR (126 MHz, CD_3OD), δ 165.5, 149.2, 138.6, 138.5, 128.2, 124.9, 124.8, 122.9, 122.4, 120.1, 119.6, 118.2, 112.3, 44.2 ppm. DART-MS-TOF m/z calculated for $C_{22}H_{17}N_3$ (M+) = 323.1422, found 323.1420.

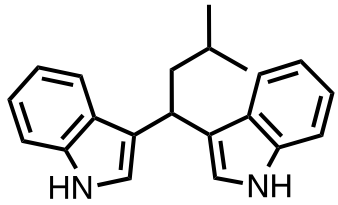


4-(di(1*H*-indol-3-yl)methyl)phenol 8m: The reaction time was 3 hours and a 25-80% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as an orange foam (30.2 mg, 91% yield). The spectral values were in accord with literature values.[2] IR (thin film): 3408, 3054, 2969, 2852, 1611, 1596, 1509, 1488, 1455, 1417, 1337, 1218, 1170, 1124, 1092, 1038, 1010, 786, 743, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.31 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 8.5 Hz, 2H), 7.03 (t, *J* = 7.1 Hz, 2H), 6.86 (t, *J* = 7.1 Hz, 2H), 6.69 (d, *J* = 8.5 Hz, 2H), 6.61 (s, 2H), 5.74 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 156.4, 138.5, 137.3, 130.7, 128.4, 124.6, 122.1, 120.6, 120.5, 119.3, 115.7, 112.0, 40.8 ppm. DART-MS-TOF m/z calculated for $C_{23}H_{18}N_2O$ (M+) = 338.1419, found 338.1422.

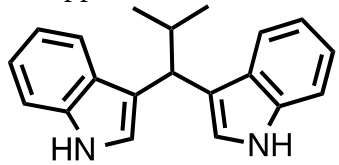


***N*-(4-(di(1*H*-indol-3-yl)methyl)phenyl)acetamide 8n:** The reaction time was 1 hour and a 20-100% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red-orange foam (36.6 mg, 96% yield). The spectral values were in accord

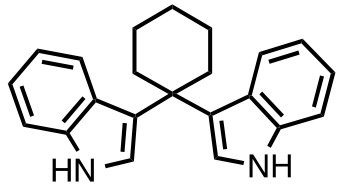
with literature values.[11] IR (thin film): 3410, 3301, 3123, 3056, 2974, 2929, 2856, 1734, 1717, 1670, 1636, 1601, 1576, 1540, 1510, 1489, 1473, 1456, 1436, 1408, 1371, 1339, 1316, 1268, 1245, 1217, 1180, 1150, 1097, 1038, 1010, 965, 929, 857,789, 742, 701, 670, 623, 604, 535, 519, 484, 466, 425 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.92 (br s, 2H), 7.32-7.41 (m, 6H), 7.26-7.32 (m, 2H), 7.16 (dt, *J* = 1.0, 8.1 Hz, 2H), 7.12 (br s, 1H), 7.00 (dt, *J* = 0.9, 8.0 Hz, 2H), 6.64 (d, *J* = 0.9 Hz, 2H), 5.85 (s, 1H), 2.16 (s, 3H) ppm. ^{13}C NMR (126 MHz, d_6-DMSO), δ 168.0, 139.6, 137.1, 136.6, 128.4, 126.6, 123.5, 120.8, 119.1, 118.8, 118.2, 118.1, 111.4, 39.1, 23.9 ppm.



3,3'-(3-methylbutane-1,1-diyl)bis(1*H*-indole) 8o: The reaction time was 2 hours and a 15-35% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the known title compound[14] as a light brown foam (27.2 mg, 90% yield). IR (thin film): 3404, 3056, 2954, 2928, 2866, 1684, 1653, 1636, 1617, 1559, 1541, 1520, 1507, 1488, 1456, 1419, 1384, 1337, 1308, 1239, 1218, 1155, 1098, 1011, 806, 739, 701, 667, 640, 582, 459, 424 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.87 (br s, 2H), 7.64 (d, *J* = 7.9 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.16 (dt, *J* = 1.0, 7.1 Hz, 2H), 7.06 (dt, *J* = 1.0, 8.0 Hz, 2H), 6.98 (d, *J* = 2.0 Hz, 2H), 4.62 (t, *J* = 7.7 Hz, 1H), 2.11 (t, *J* = 7.1 Hz, 2H), 1.65 (nonet, *J* = 6.6 Hz, 1H), 1.00 (d, *J* = 6.6 Hz, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 136.6, 127.1, 121.7, 121.4, 120.5, 119.6, 119.0, 111.0, 45.1, 31.6, 25.9, 22.8 ppm. DART-MS-TOF m/z calculated for $C_{21}H_{23}N_2$ (M+H) = 303.1856, found 303.1827.

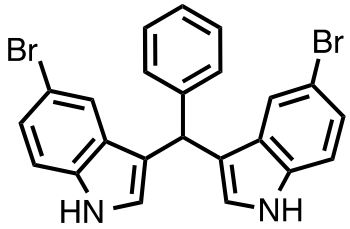


3,3'-(2-Methylpropylidene)bis(1H-indole) 8p: The reaction time was 16 hours and a 10-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a white foam (28.9 mg, 99% yield). The spectral values were in accord with literature values.[17] IR (thin film): 3409, 3054, 2955, 2867, 1617, 1487, 1455, 1417, 1383, 1336, 1244, 1220, 1093, 1038, 1010, 906, 846, 782, 733, 648, 599, 580, 489, 462, 423 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.83 (br s, 2H), 7.73 (d, *J* = 7.7 Hz, 2H), 7.11-7.25 (m, 6H), 6.92 (s, 2H), 4.29 (d, *J* = 8.4 Hz, 1H), 2.68 (m, 1H), 1.07 (d, *J* = 6.6 Hz, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 136.0, 127.5, 121.7, 121.3, 119.4, 119.3, 118.7, 111.0, 40.9, 32.8, 21.7 ppm. DART-MS-TOF m/z calculated for $C_{20}H_{20}N_2$ (M+) =288.1626, found 288.1627.

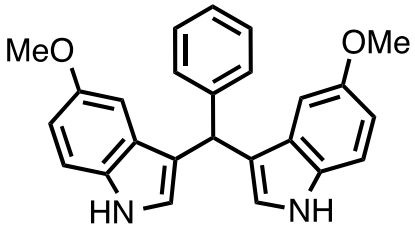


3,3'-(cyclohexane-1,1-diyl)bis(1*H*-indole) 8q: The reaction time was 16 hours and a 10-25% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a white foam (30.8 mg, 98% yield). The spectral values were in accord with literature values.[15] IR (thin film): 3410, 3053, 2929, 2853, 1616, 1486, 1455, 1415, 1335, 1243,

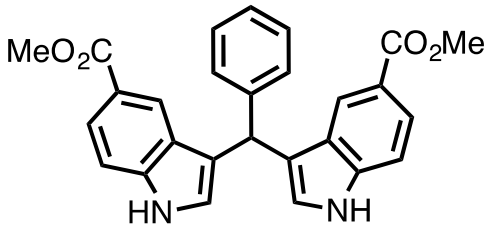
1121, 1102, 1014, 993, 907, 814, 765, 740, 613, 582, 504, 481, 425 cm^{-1}. ^{1}H NMR (400 MHz, d_6-benzene), δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.10-7.20 (m, 2H), 7.00-7.05 (m, 4H), 6.64 (br s, 2H), 6.59 (d, *J* = 2.4 Hz, 2H), 2.60-2.64 (m, 4H), 1.60-1.70 (m, 4H), 1.50-1.60 (m, 2H) ppm. ^{13}C NMR (126 MHz, d_6-benzene), δ 137.6, 126.8, 123.7, 122.3, 122.0, 121.6, 119.1, 111.5, 40.1, 37.2, 27.2, 23.4 ppm. DART-MS-TOF m/z calculated for $C_{22}H_{22}N_2$ (M+) =314.1783, found 314.1780.



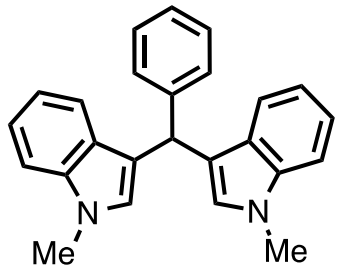
3,3'-(phenylmethylene)bis(5-bromo-1*H*-indole) 8r: The reaction time was 45 minutes and a 5-45% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red-orange foam (82.0 mg, 87% yield). The spectral values were in accord with literature values.[2] IR (thin film) 3423, 3062, 3025, 2958, 2928, 2871, 1733, 1699, 1601, 1569, 1542, 1509, 1493, 1457, 1418, 1399, 1362, 1318, 1262, 1205, 1129, 1096, 907, 883, 843, 794, 764, 742, 702, 657, 582, 481, 421 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.99 (br s, 2H), 7.47(d, *J* = 1.4 Hz, 2H), 7.27-7.31 (m, 4H), 7.23-7.26 (m, 4H), 6.64 (d, *J* = 1.4 Hz, 2H), 5.75 (s, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 143.0, 135.3, 128.6, 128.5, 128.4, 126.5, 124.9, 124.7, 122.2, 119.0, 112.6, 112.6, 39.9 ppm. DART-MS-TOF m/z calculated for $C_{23}H_{16}Br_2N_2$ (M+) = 477.9680, found 477.9679.



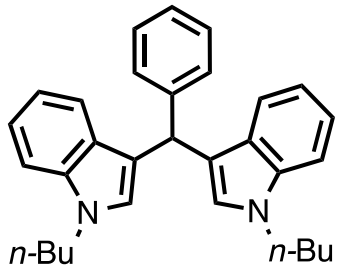
3,3'-(phenylmethylene)bis(5-methoxy-1*H*-indole) 8s: The reaction time was 15 minutes and a 5-30% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a white solid (70.1 mg, 93% yield). The spectral values were in accord with literature values.[2] IR (thin film) 3412, 3056, 2973, 2937, 2830, 1717, 1699, 1684, 1622, 1584, 1541, 1518, 1483, 1455, 1439, 1408, 1355, 1290, 1270, 1210, 1171, 1126, 1090, 1043, 1029, 926, 909, 832, 797, 772, 738, 716, 646, 610, 599, 484, 431 cm^{-1}. ^{1}H NMR (400 MHz, d_6-DMSO), δ 10.66 (d, *J* = 2.0 Hz, 2H), 7.36 (d, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 2H), 7.23 (d, *J* = 8.7 Hz, 2H), 7.17 (tt, *J* = 1.2, 7.3 Hz, 1H), 6.81 (d, *J* = 2.2 Hz, 2H), 6.72 (d, *J* = 2.4 Hz, 2H), 6.69 (dd, *J* = 2.4, 8.6 Hz, 2H), 5.73 (s, 1H), 3.58 (s, 3H) ppm. ^{13}C NMR (126 MHz, d_6-DMSO), δ 152.6, 145.0, 131.8, 128.3, 128.0, 127.0, 125.7, 124.3, 117.7, 112.0, 110.5, 101.4, 55.2, 39.6 ppm. DART-MS-TOF m/z calculated for $C_{25}H_{22}N_2O_2$ (M+) =382.1681, found 382.1668.



dimethyl 3,3'-(phenylmethylene)bis(1*H*-indole-5-carboxylate) 8t: The reaction time was 1 hour and a 18-45% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the known title compound[18] as a light pink solid (74.0 mg, 87% yield). IR (thin film) 3322, 1716, 1694, 1654, 1636, 1618, 1583, 1559, 1541, 1520, 1508, 1491, 1474, 1436, 1399, 1312, 1282, 1264, 1238, 1216, 1102, 1028, 969, 911, 893, 822, 808, 762, 747, 701, 665, 641, 622, 588, 577, 555, 506, 484, 447, 420 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 8.19 (br s, 2H), 8.14 (d, *J* = 0.6 Hz), 7.88 (dd, *J* = 1.6, 8.6 Hz, 2H), 7.35 (d, *J* = 8.6 Hz, 2H), 7.19-7.35 (m, 5H), 6.71 (d, *J* = 1.2 Hz, 2H), 5.96 (s, 1H), 3.85 (s, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 168.2, 143.3, 139.3, 128.5, 128.4, 126.5, 126.5, 124.8, 123.5, 122.6, 121.5, 120.9, 110.8, 51.8, 39.8 ppm.

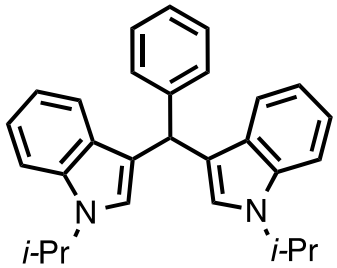


3,3'-(phenylmethylene)bis(1-methyl-1*H*-indole) 8u: This reaction was done on a 0.4 mmol scale. The reaction time was 1 hour and a 2-10% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a pink foam (133.6 mg, 95% yield). The spectral values were in accord with literature values.[2] IR (thin film) 3053, 3020, 2927, 1719, 1613, 1597, 1550, 1472, 1446, 1425, 1368, 1345, 1327, 1238, 1224, 1199, 1174, 1151, 1129, 1116, 1054, 1027, 1011, 924, 852, 800, 768, 740, 701, 679, 600, 572, 539, 517, 453, 427 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.36 (d, *J* = 7.9 Hz, 2H), 7.33 (d, *J* = 7.1 Hz, 2H), 7.15-7.30 (m, 7H), 6.97 (t, *J* = 7.6 Hz, 2H), 6.51 (s, 2H), 5.86 (s, 1H), 3.65 (s, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 144.4, 137.4, 128.7, 128.2, 128.2, 127.4, 126.0, 121.4, 120.0, 118.6, 118.2, 109.0, 40.0, 32.6 ppm. DART-MS-TOF m/z calculated for $C_{25}H_{22}N_2$ (M+) = 350.1783, found 350.1780.

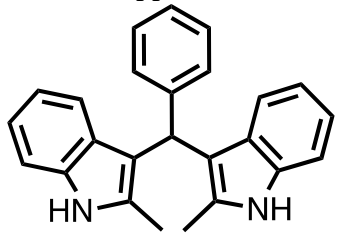


3,3'-(phenylmethylene)bis(1-butyl-1*H*-indole) 8v: The reaction time was 15 minutes and a 5-20% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the known title compound[19] as a red foam (38.6 mg, 90% yield). IR (thin film) 3054, 2956, 2929, 2871, 1726, 1611, 1546, 1492, 1479, 1465, 1393, 1363, 1333, 1234, 1190, 1154, 1137, 1121,

1074, 1030, 1014, 910, 793, 734, 701, 649, 614, 571, 518, 490, 427 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.36-7.51 (m, 8H), 7.25-7.35 (m, 3H), 7.08 (dt, *J* = 1.0, 7.0 Hz, 2H), 6.68 (d, *J* = 0.8 Hz, 2H), 6.00 (s, 1H), 4.10 (t, *J* = 7.1 Hz, 4H), 1.85 (pentet, *J* = 7.3 Hz, 4H), 1.39 (sextet, *J* = 7.4 Hz, 4H), 1.01 (t, *J* = 7.3 Hz, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 144.4, 136.6, 128.7, 128.1, 127.5, 127.3, 125.9, 121.1, 120.2, 118.4, 118.0, 109.2, 45.9, 40.2, 32.3, 20.1, 13.7 ppm. DART-MS-TOF m/z calculated for $C_{31}H_{34}N_2$ (M+) = 434.2722, found 434.2718.



3,3'-(phenylmethylene)bis(1-isopropyl-1*H*-indole) 8w: The reaction time was 45 minutes and a 2-12% ethyl acetate/hexanes gradient was used to purify the crude reaction mixture to give the title compound as a red film (69.0 mg, 86% yield). IR (thin film) 3054, 2973, 2931, 1609, 1546, 1492, 1460, 1404, 1355, 1304, 1216, 1195, 1152, 1129, 1071, 1015, 906, 844, 792, 771, 729, 700, 666, 647, 582, 564, 522, 489, 426 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.50-7.61 (m, 6H), 7.46 (t, *J* = 7.2 Hz, 2H), 7.31-7.40 (m, 3H), 7.16 (t, *J* = 7.6 Hz, 2H), 6.93 (s, 2H), 6.11 (s, 1H), 4.77 (sextet, *J* = 6.7 Hz, 2H), 1.59 (d, *J* = 6.7 Hz, 12H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 144.5, 136.2, 128.7, 128.0, 127.6, 125.8, 123.1, 121.0, 120.2, 118.5, 118.2, 109.3, 46.9, 40.5, 22.6, 22.6 ppm. DART-MS-TOF m/z calculated for $C_{29}H_{30}N_2$ (M+) = 406.2409, found 406.2408.



3,3'-(phenylmethylene)bis(2-methyl-1*H*-indole) 8x: The reaction time was 30 minutes and then diluted with methanol and washed with sodium bisulfite to remove excess benzaldehyde.[20] Ethyl acetate: hexanes (1:1) was then added to extract the organic layer, which was then dried ($MgSO_4$), filtered, and conc. *in vacuo* to give the title compound as a pink foam (35.0 mg, 99% yield). The material was pure by ^{1}H NMR. The spectral values were in accord with literature values.[21] IR (thin film) 3395, 1459, 1295, 1219, 1009, 743, 726, 700, 596, 496 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.72 (br s, 2H), 7.18-7.30 (m, H), 7.04 (dt, *J* = 1.2, 8.1 Hz, 2H), 6.98 (d, *J* = 7.9 Hz, 2H), 6.85 (dt, *J* = 1.0, 7.0 Hz, 2H), 6.01 (s, 1H), 2.06 (s, 6H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$), δ 143.7, 135.0, 131.8, 129.1, 128.9, 128.1, 125.9, 120.6, 119.3, 119.0, 113.4, 109.9, 39.2, 12.4 ppm. DART-MS-TOF m/z calculated for $C_{25}H_{21}N_2$ $(M-H)^+$ = 349.1699, found 349.1671.

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1H turbo precursor **8a**
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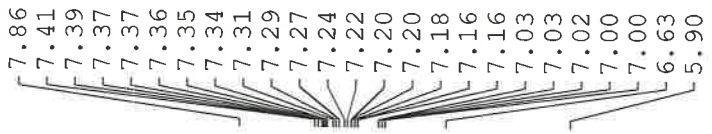

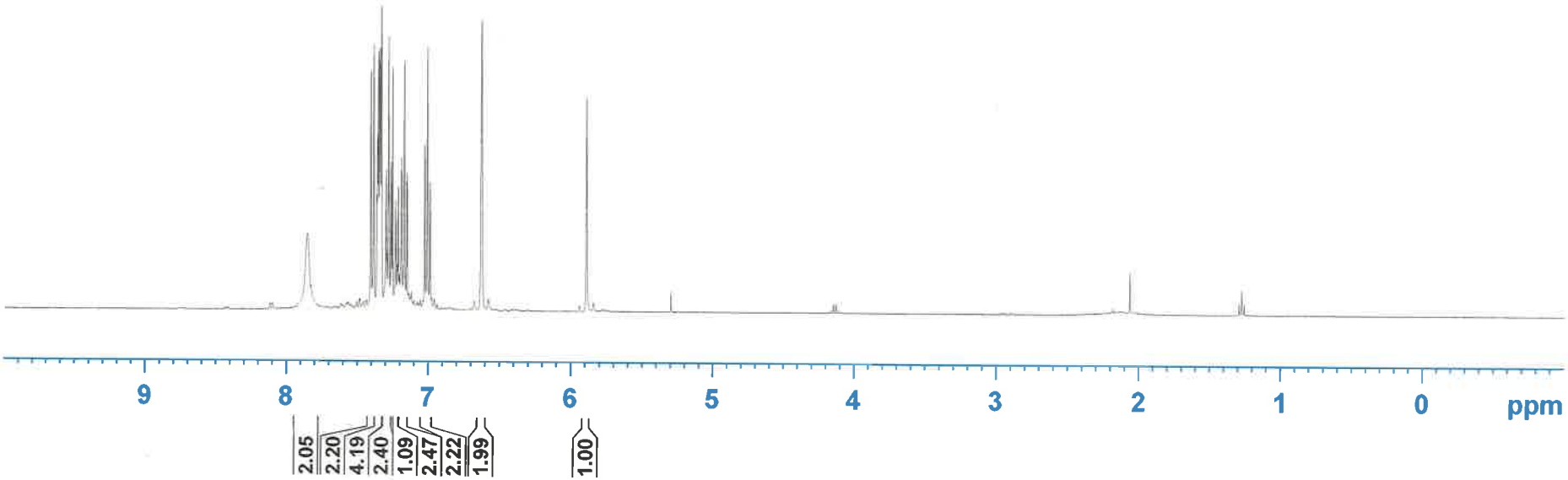

13C turbo precursor **8a**
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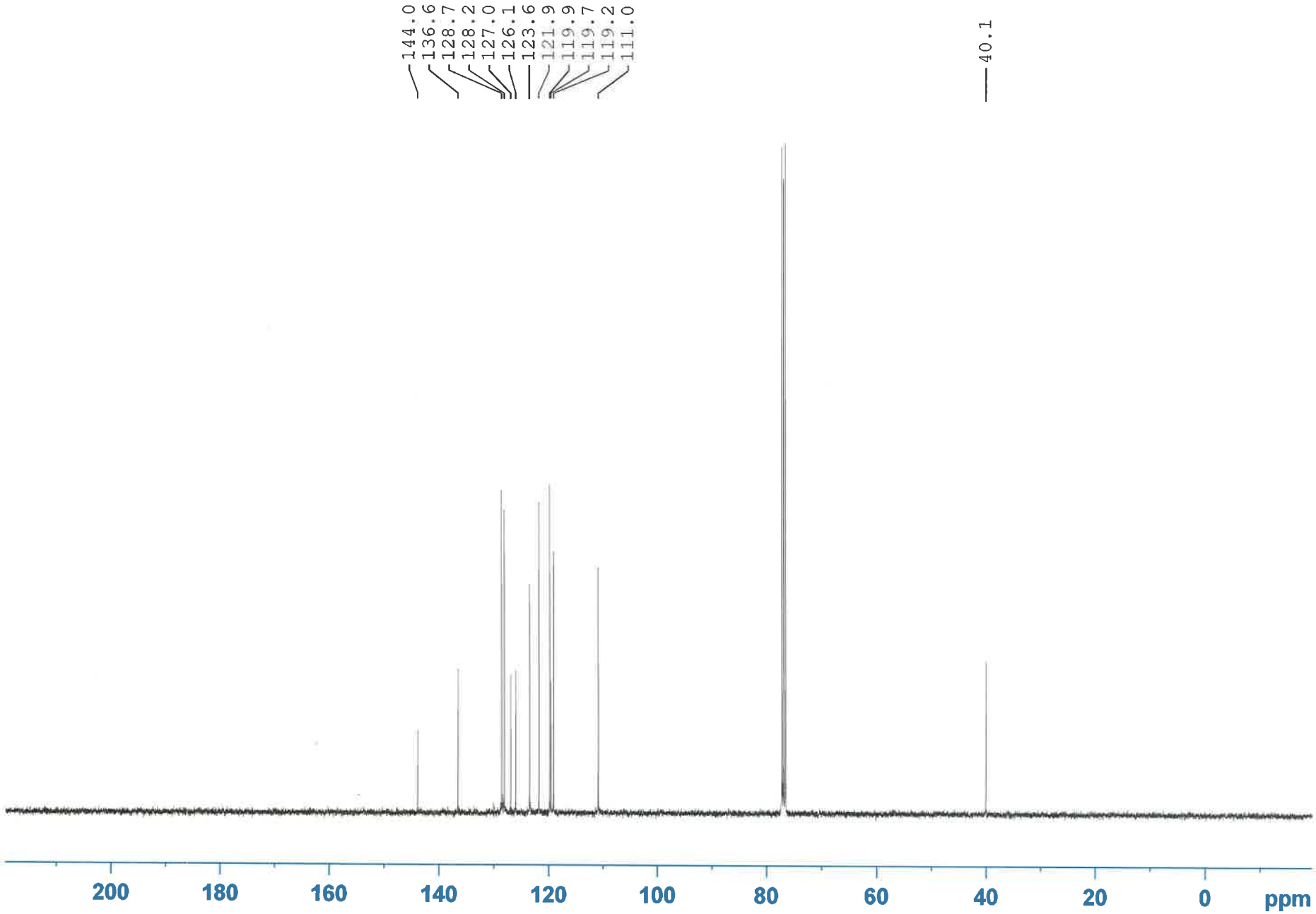

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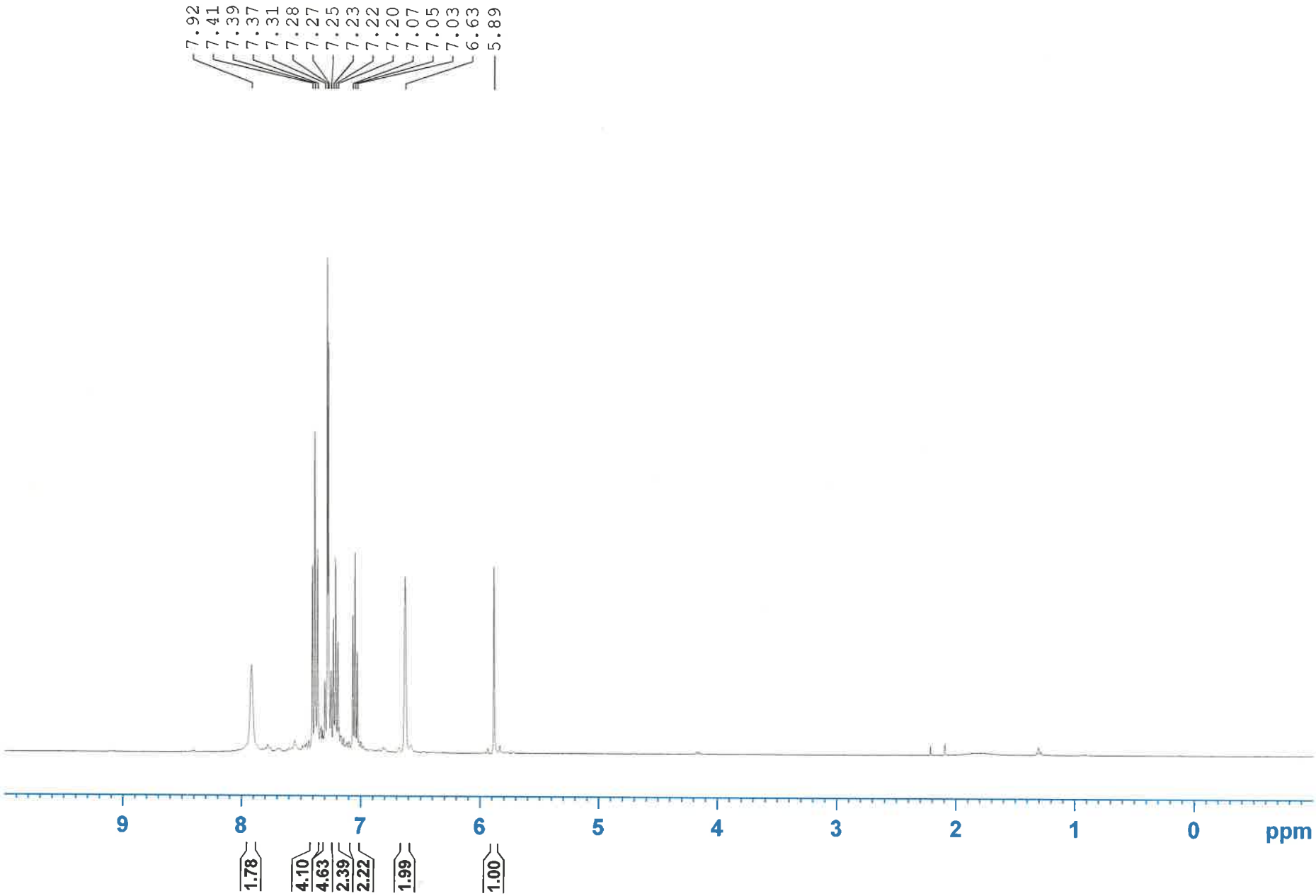

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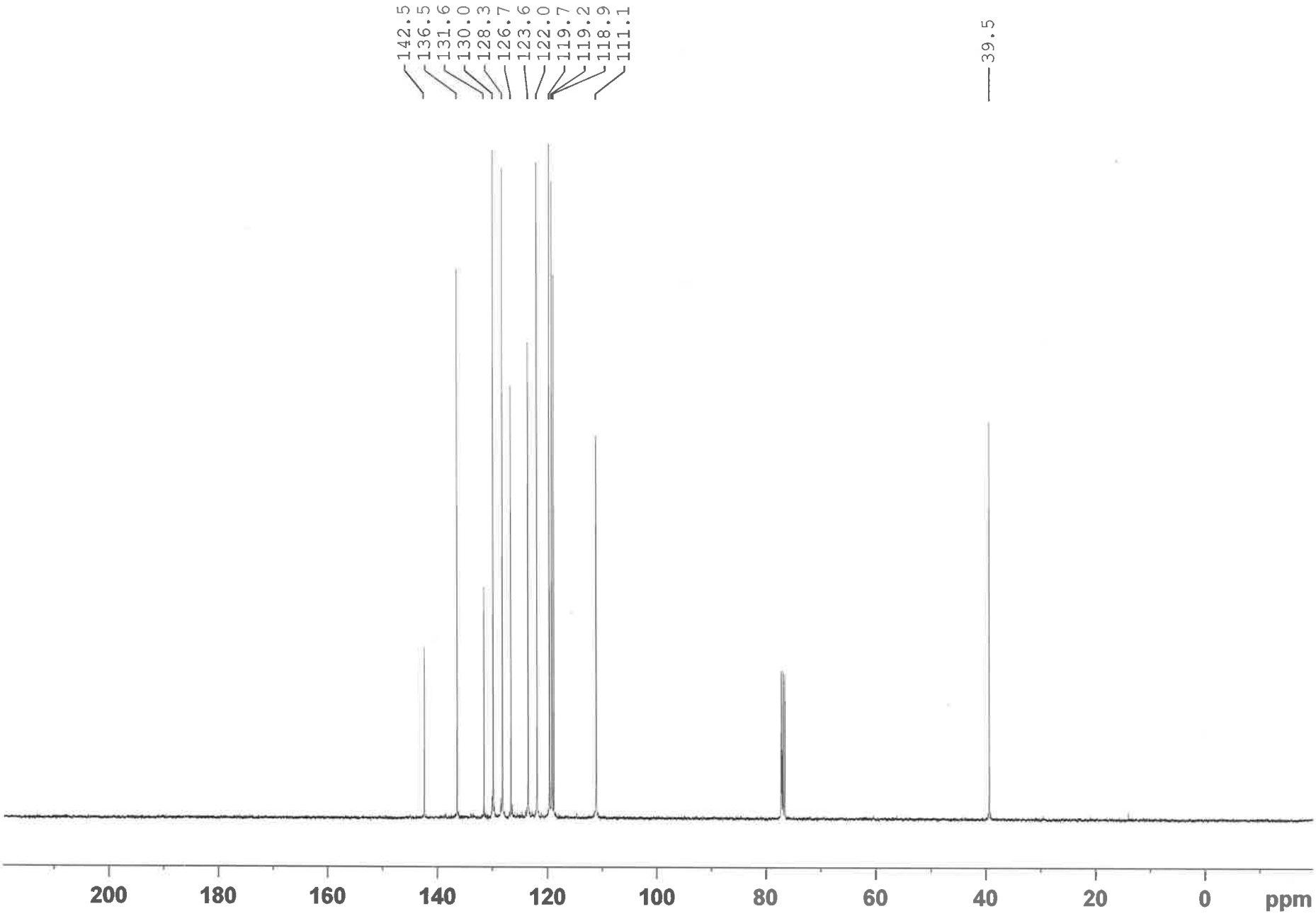

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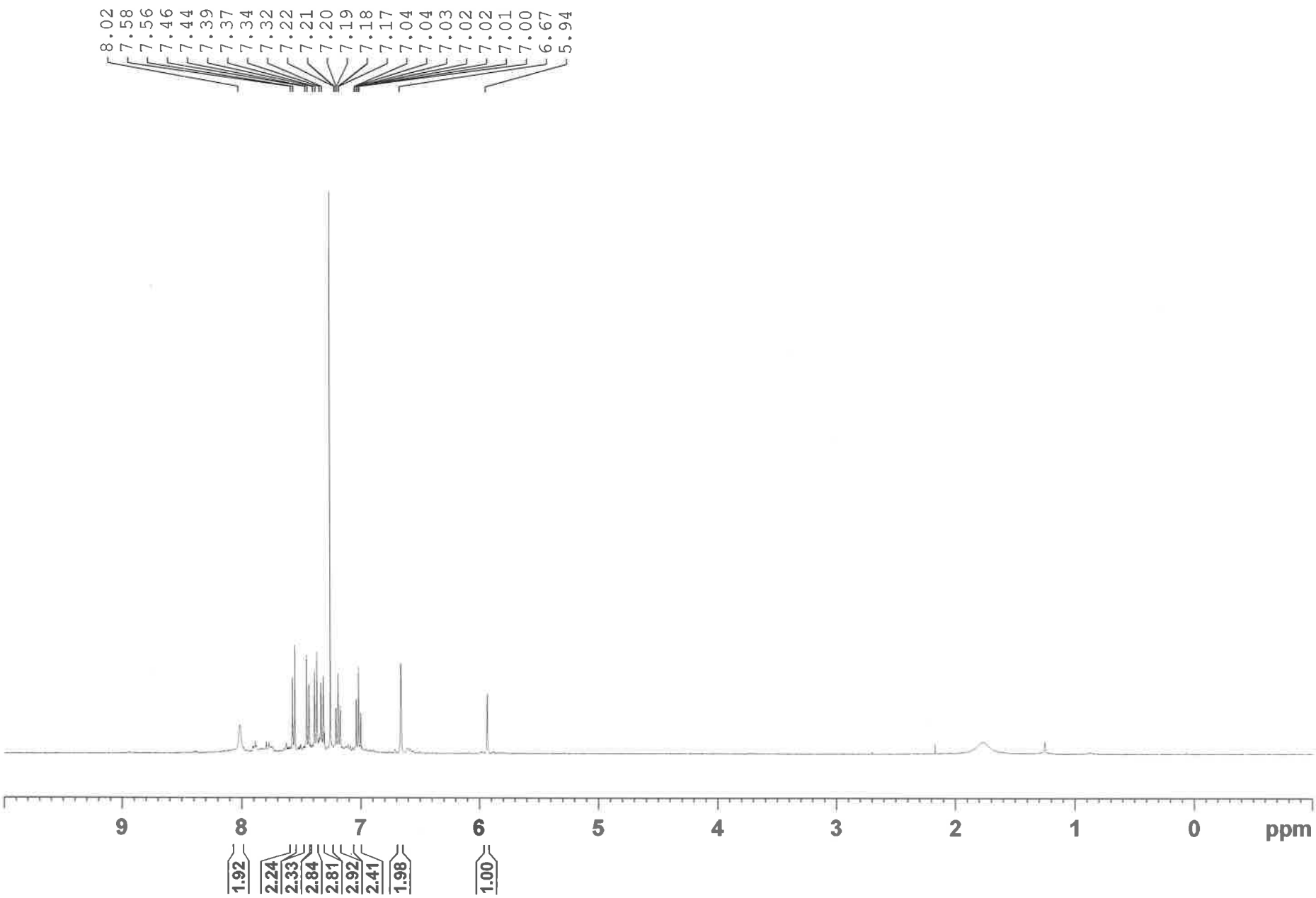

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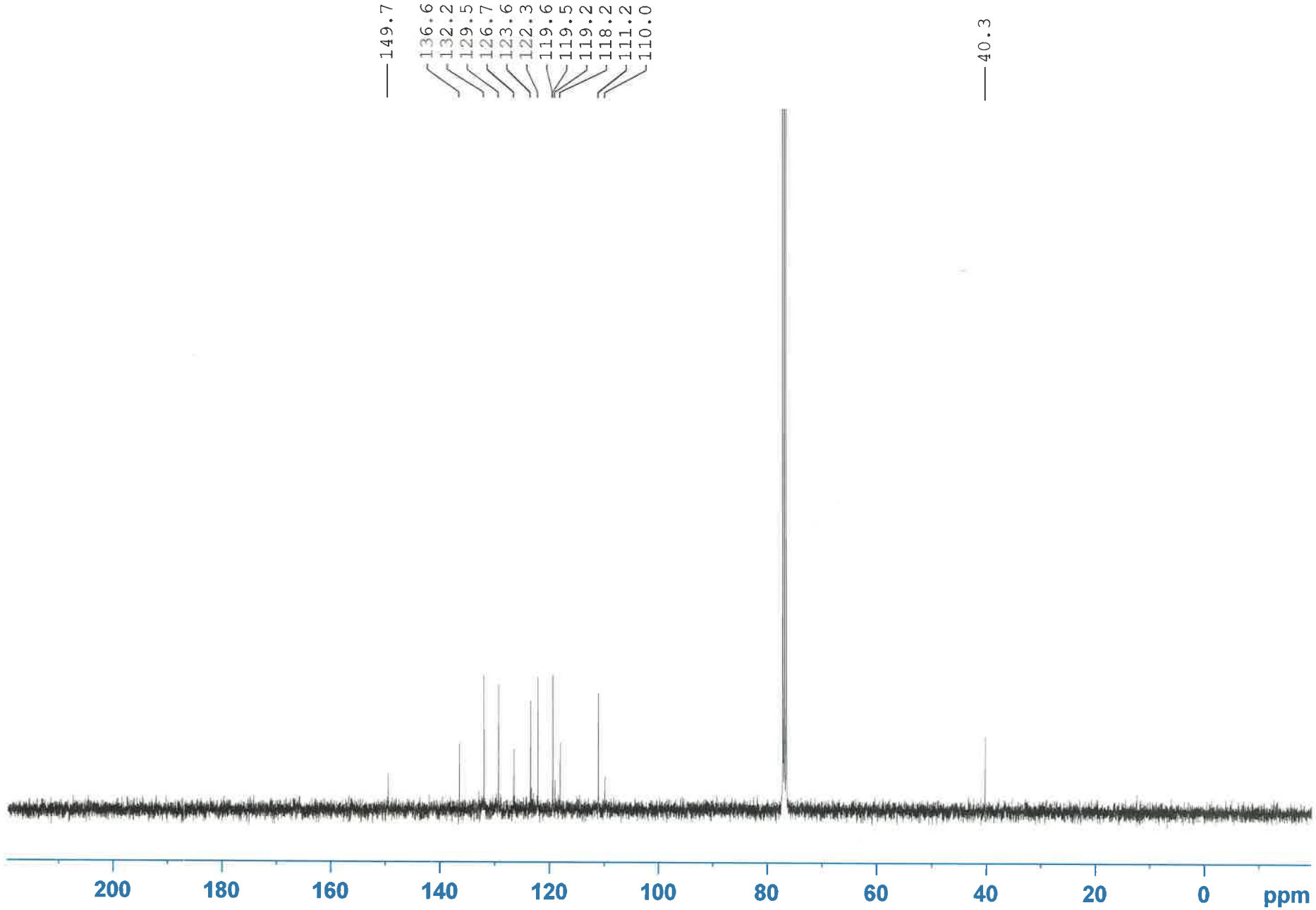
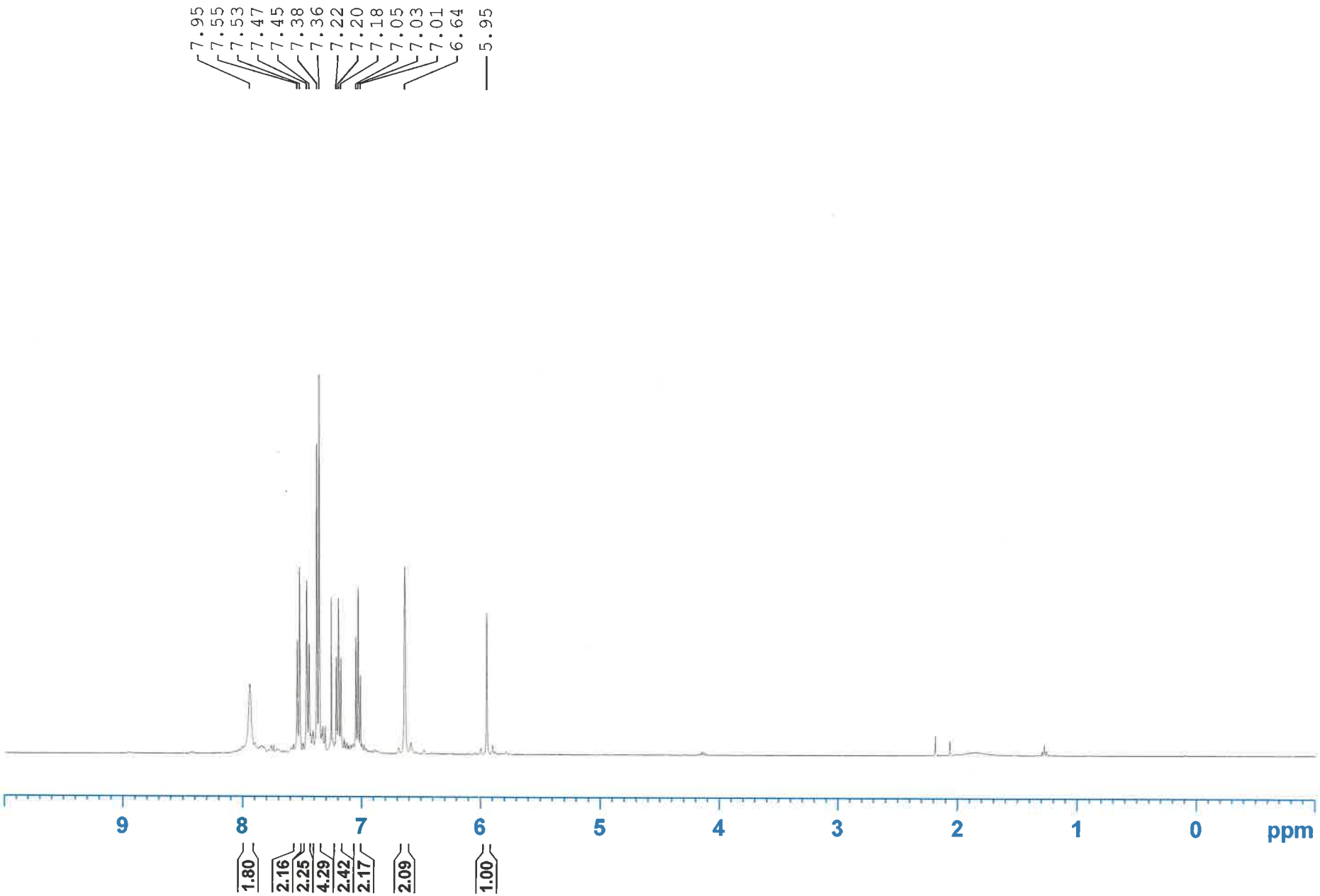
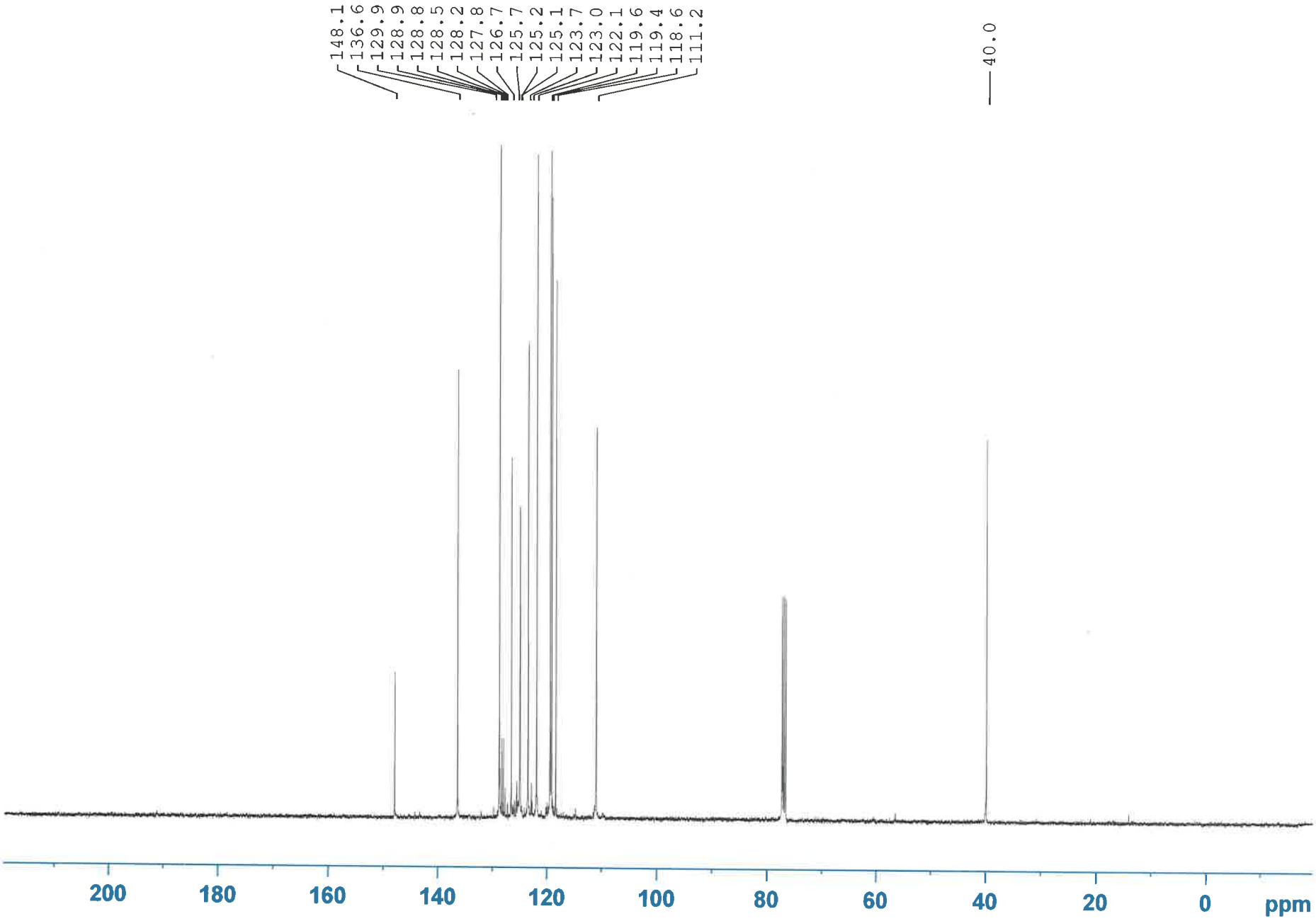


1H pCF3 **8d**
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\CBrindle\07172015} OrgoLab 2



8d

13C pCF3
C13CPD CDCl3 {C:\Bruker\TOPSPIN\CBrindle\07162015} OrgoLab 4



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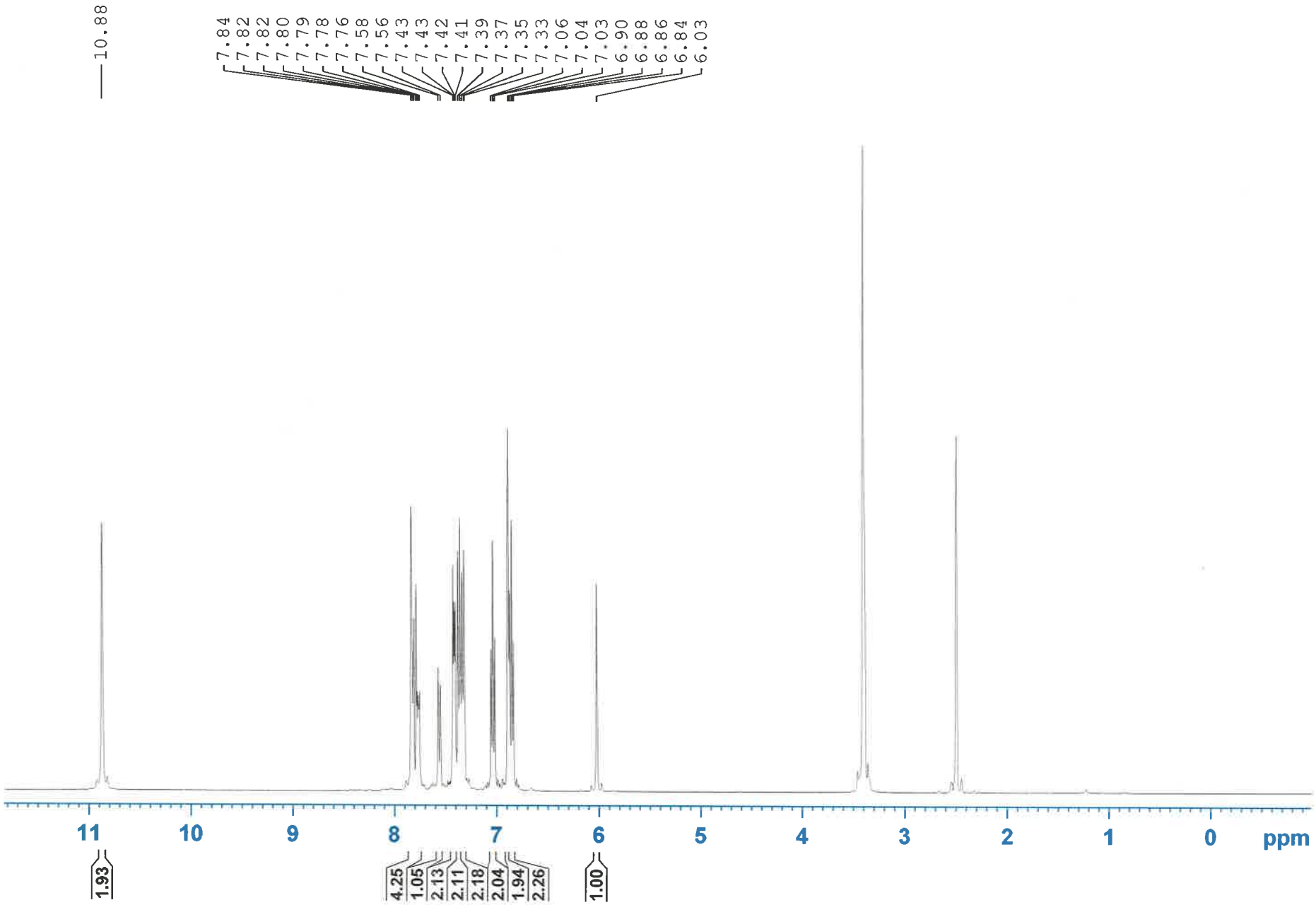
TrinPROTON DMSO {C:\Bruker\TOPSPIN\CBrindle\Aug142015} OrgoLab 5



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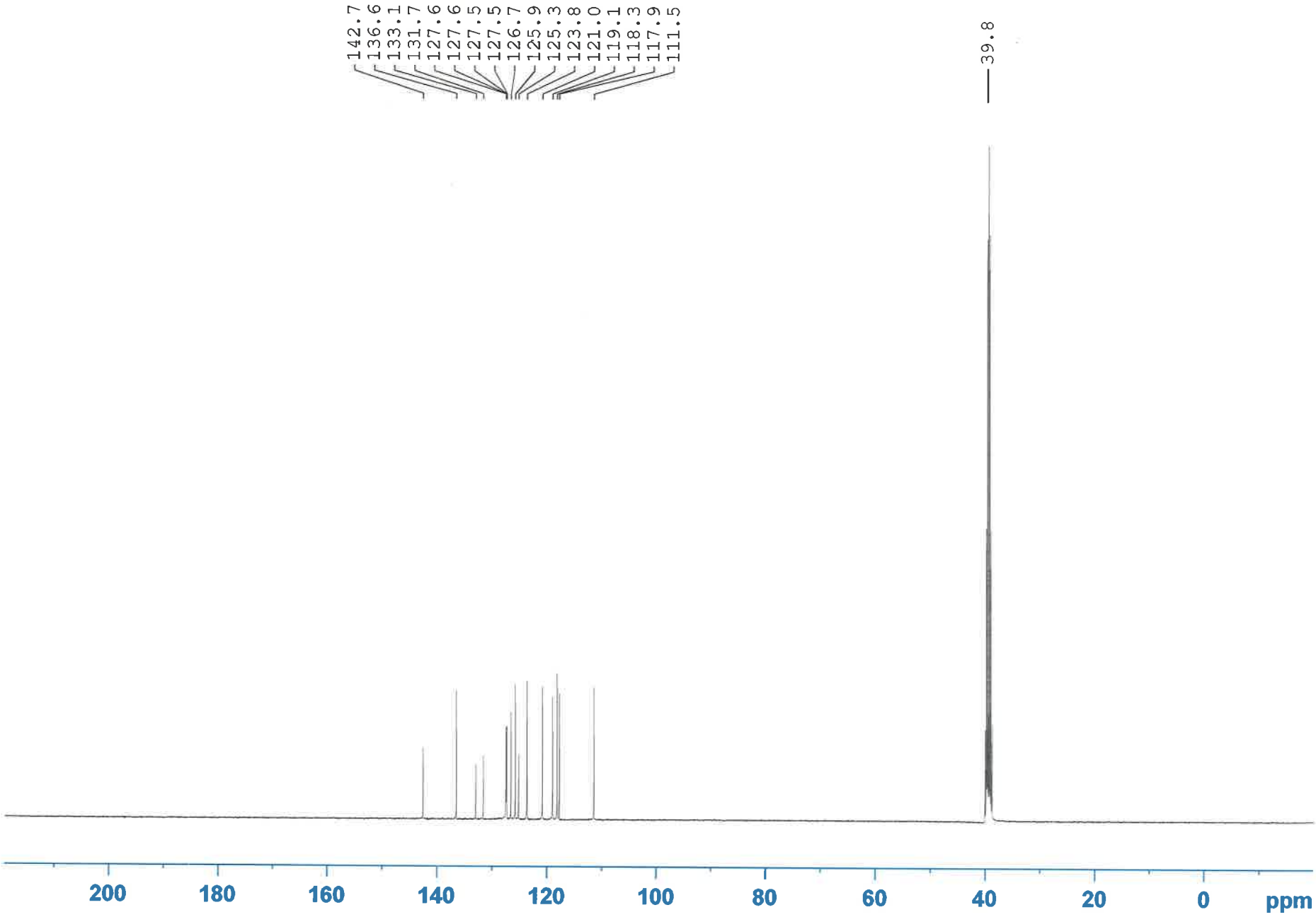
C13CPD DMSO {C:\Bruker\TOPSPIN\CBrindle\Aug142015} OrgoLab 5



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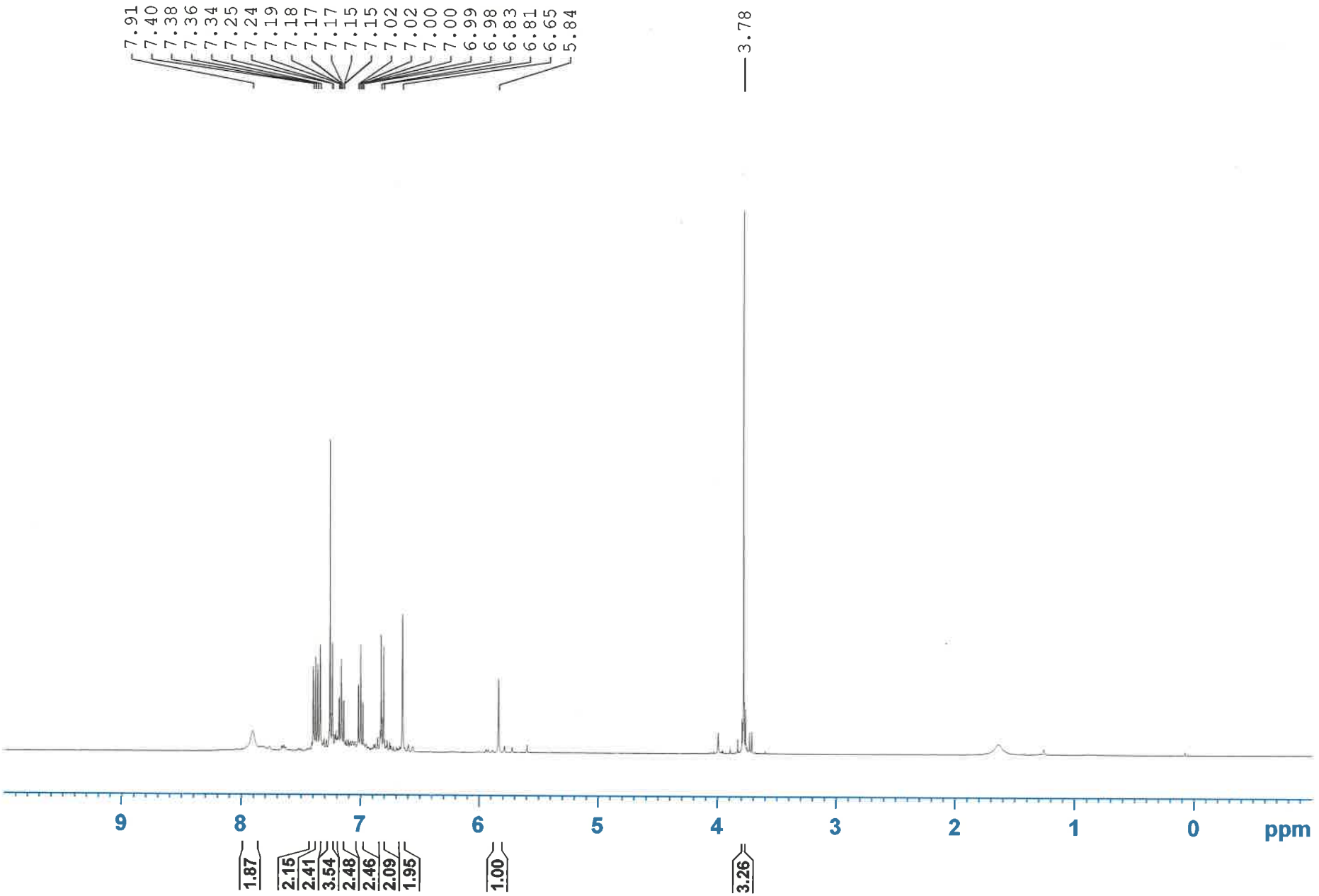

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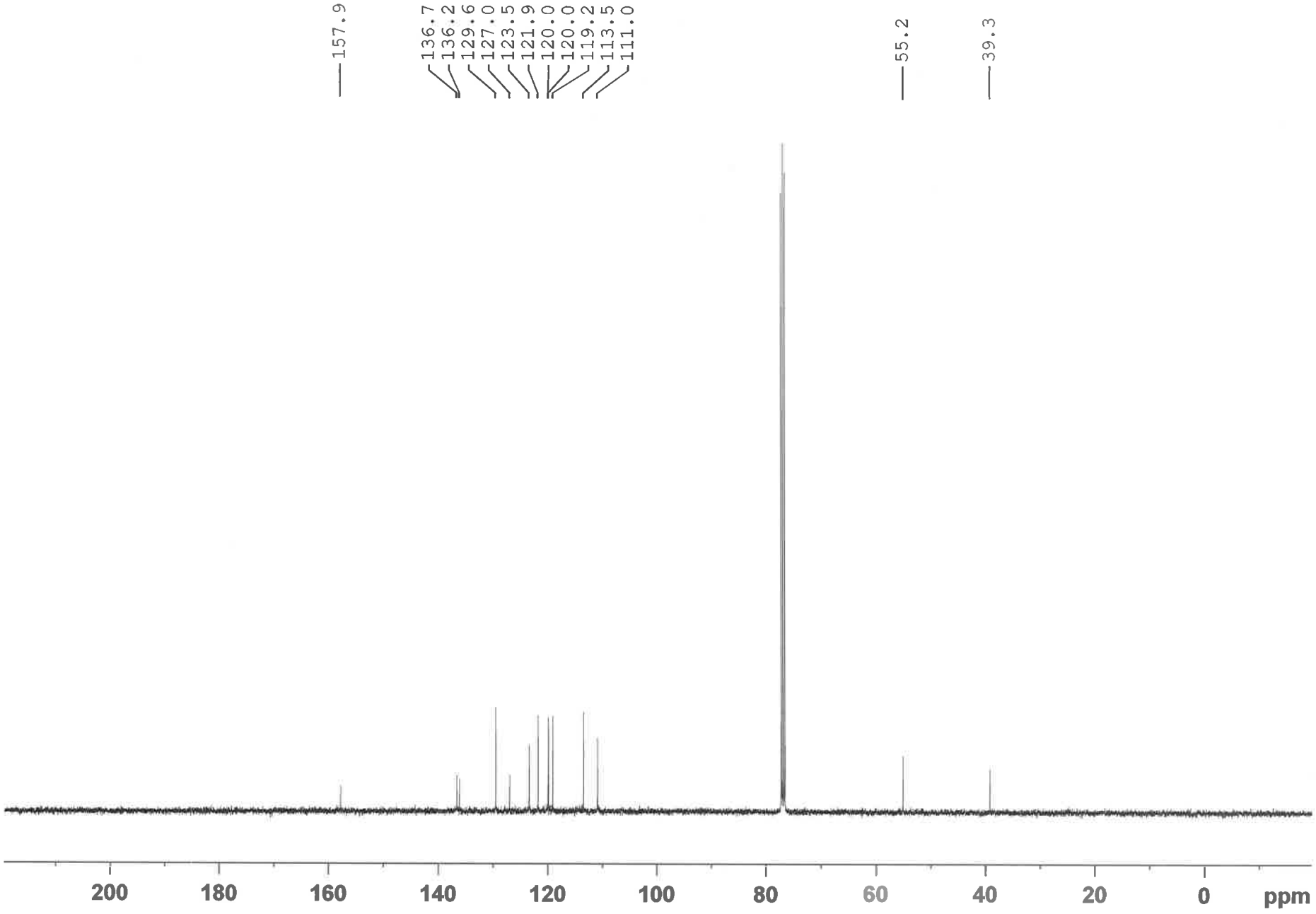

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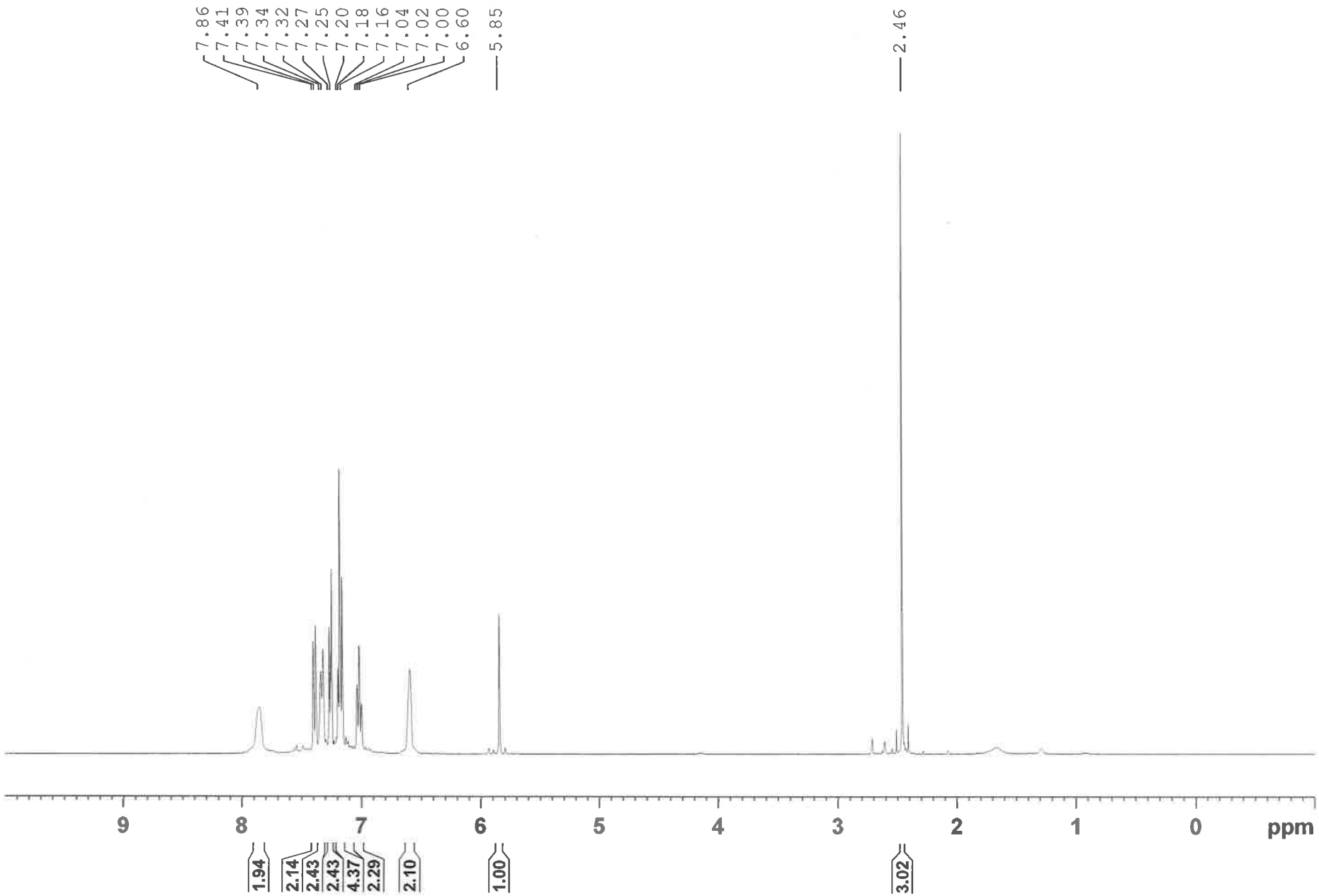

13C pSMe **8g**
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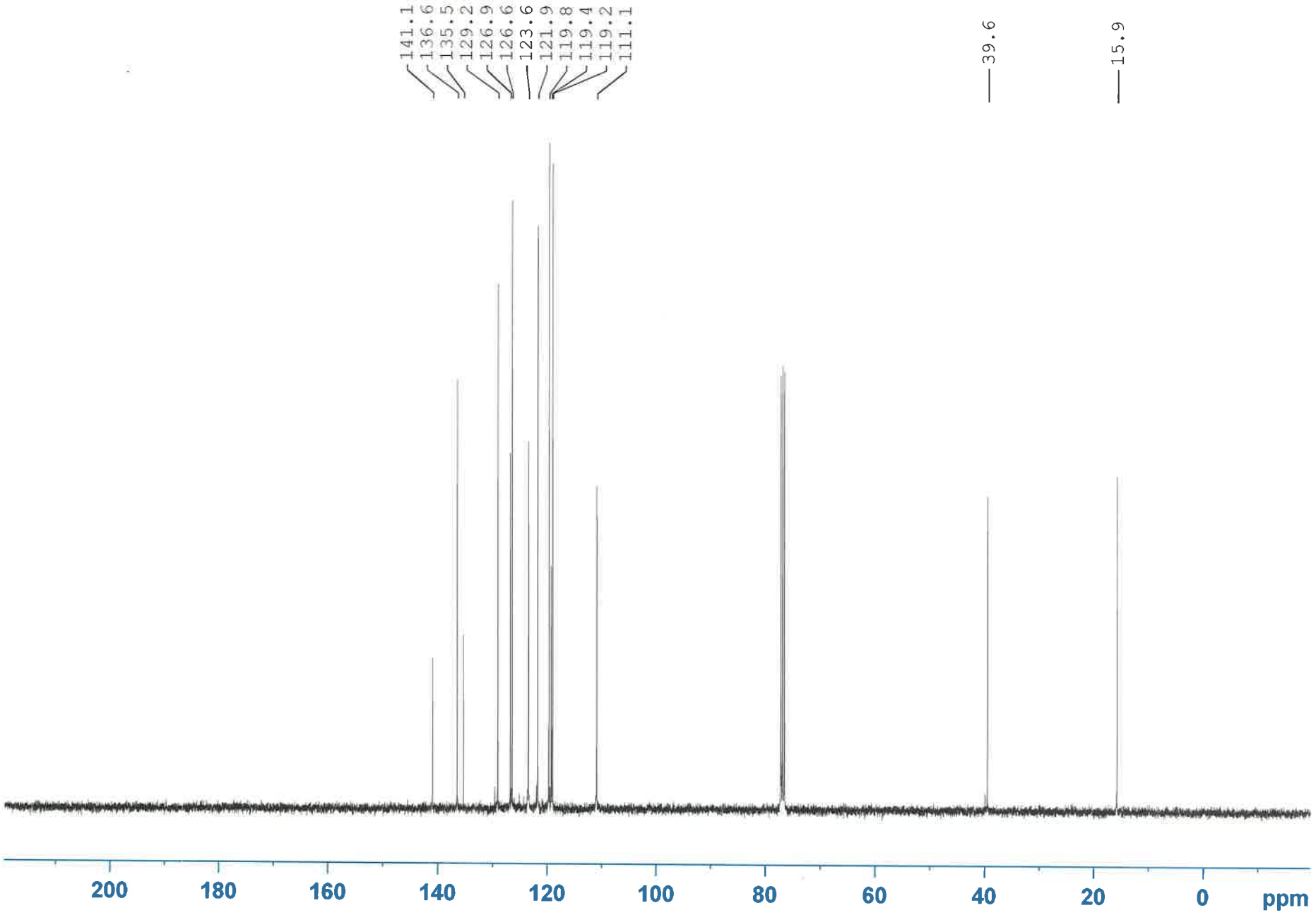
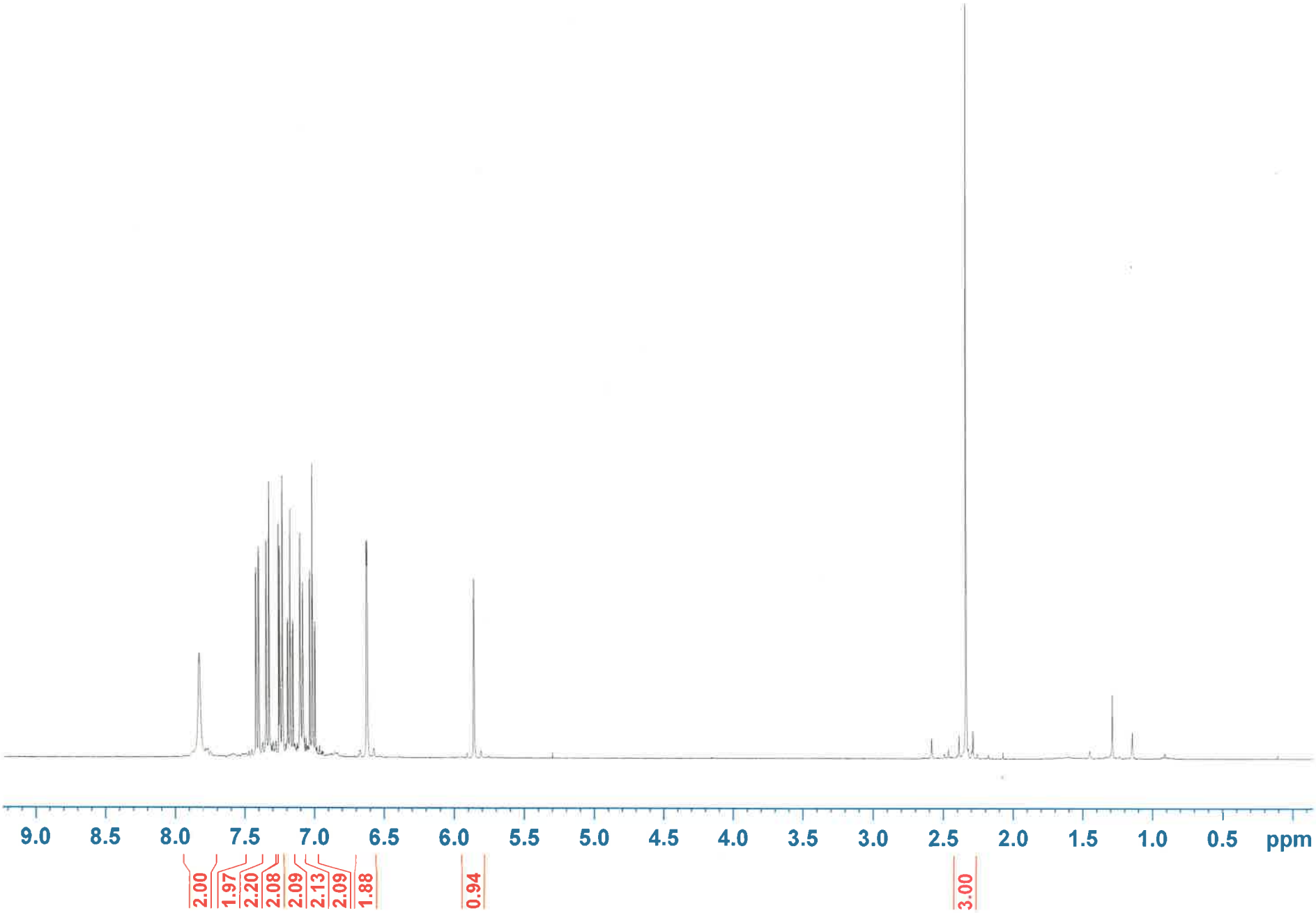


8h

cbip29 para 1H



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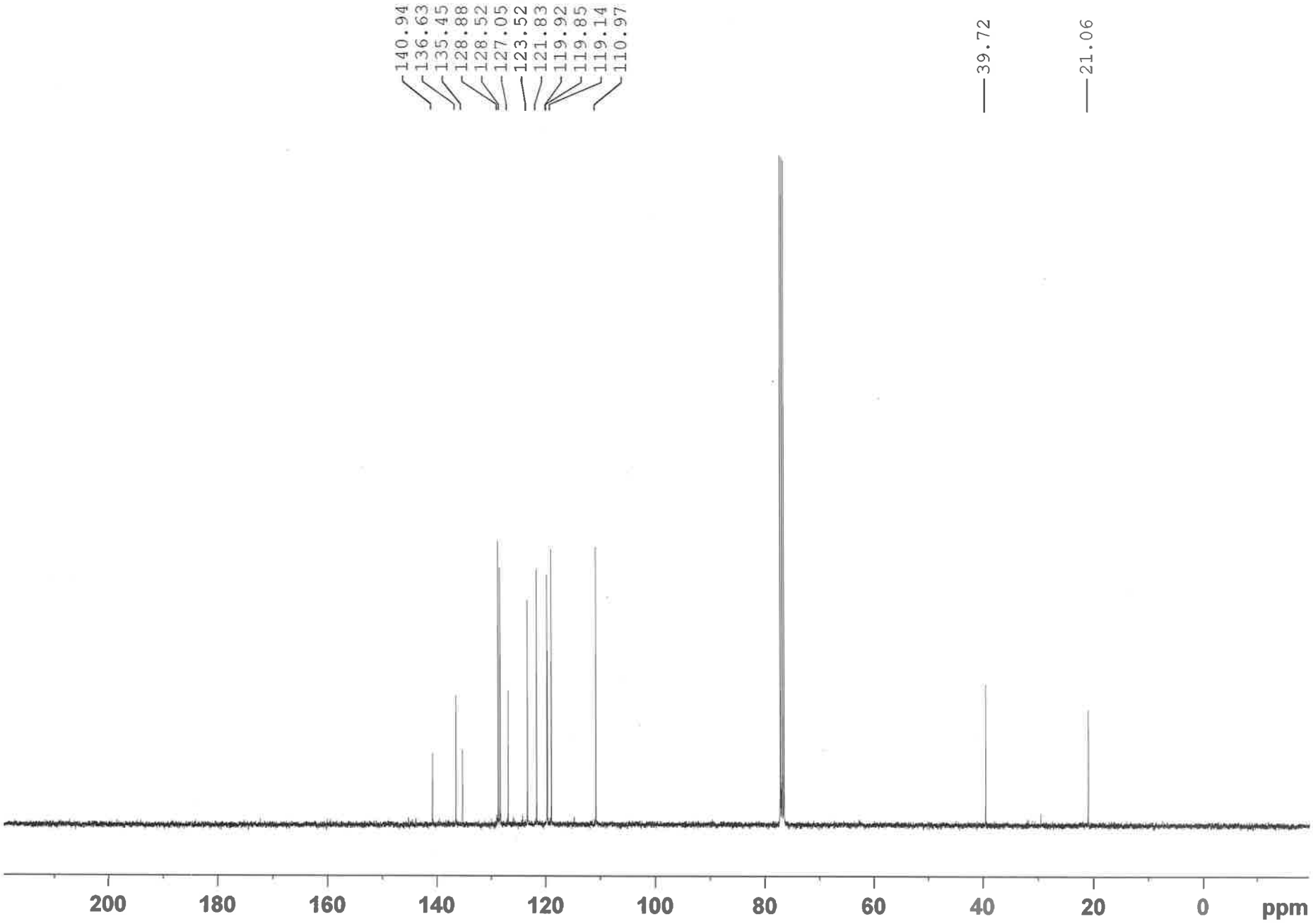
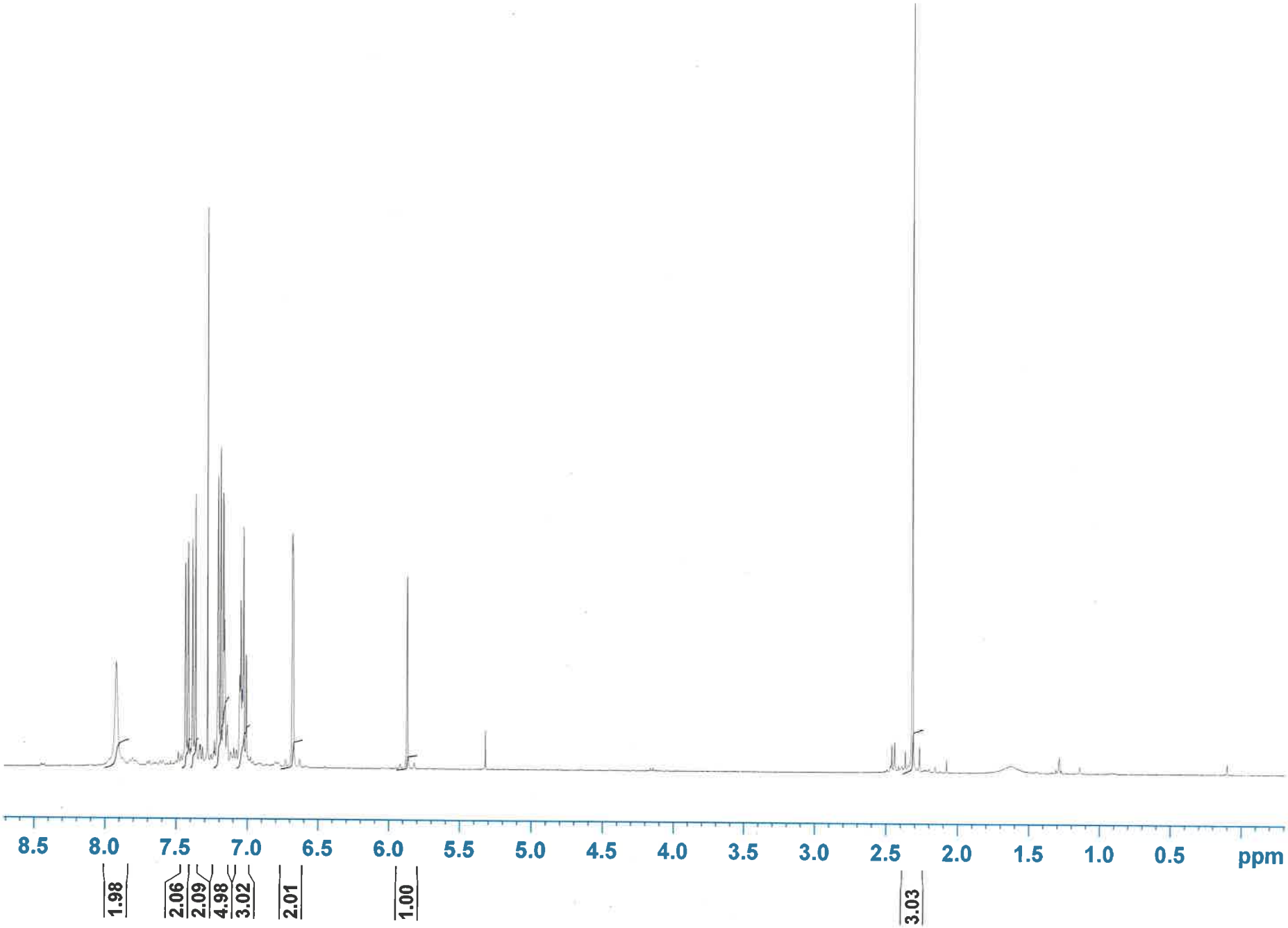


cbip29 meta 1H

8i



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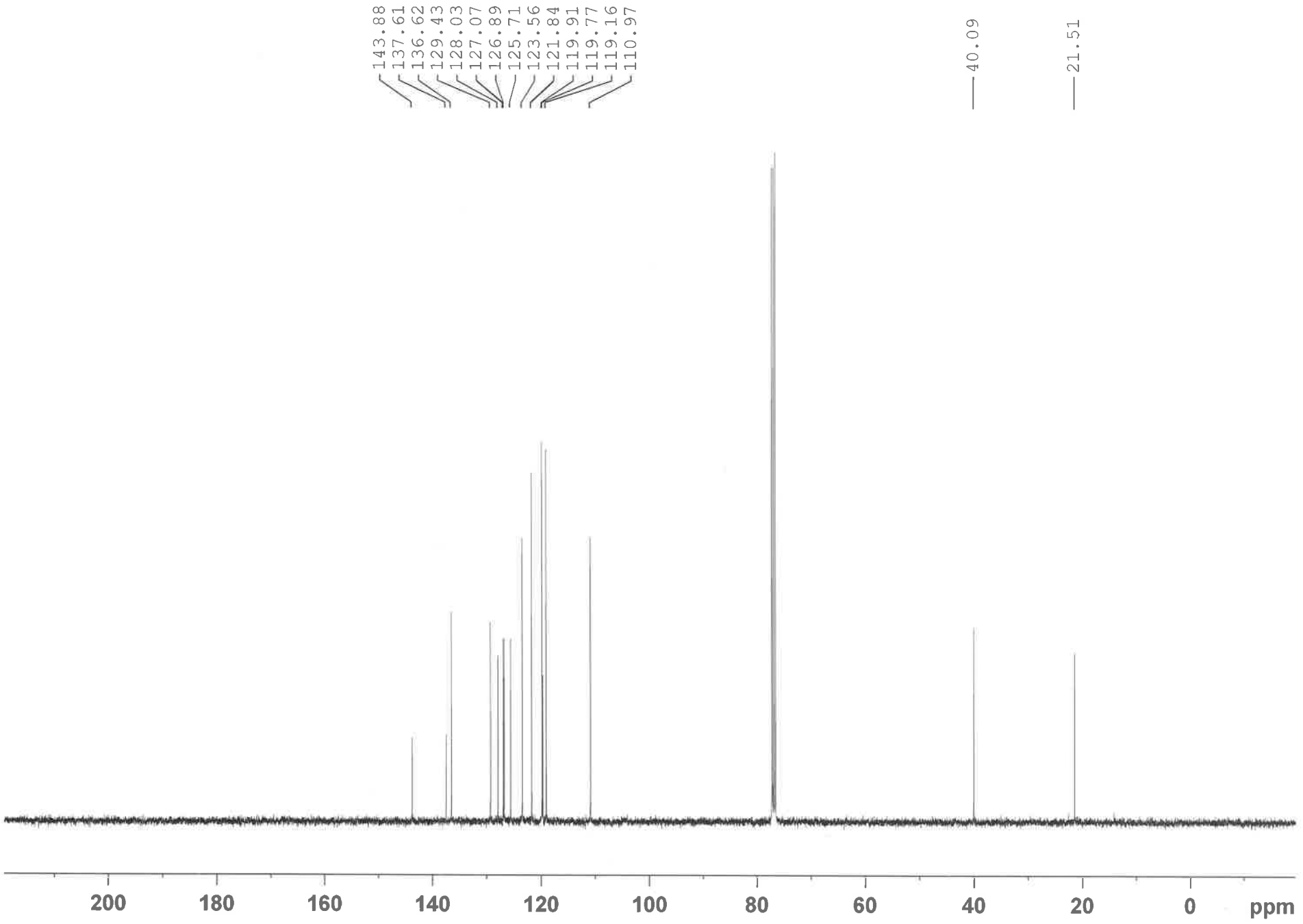

8j

1H o-tolyl
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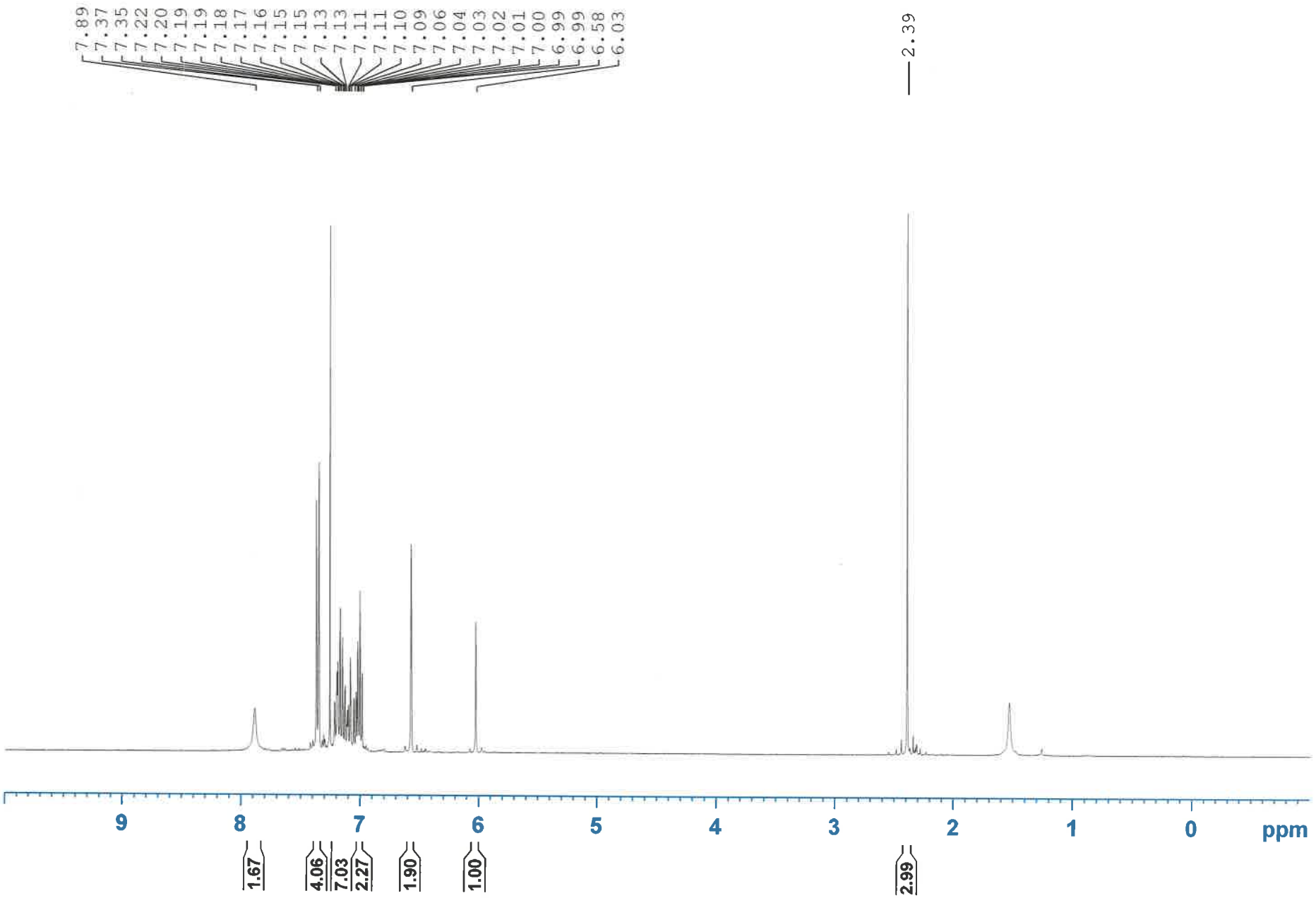

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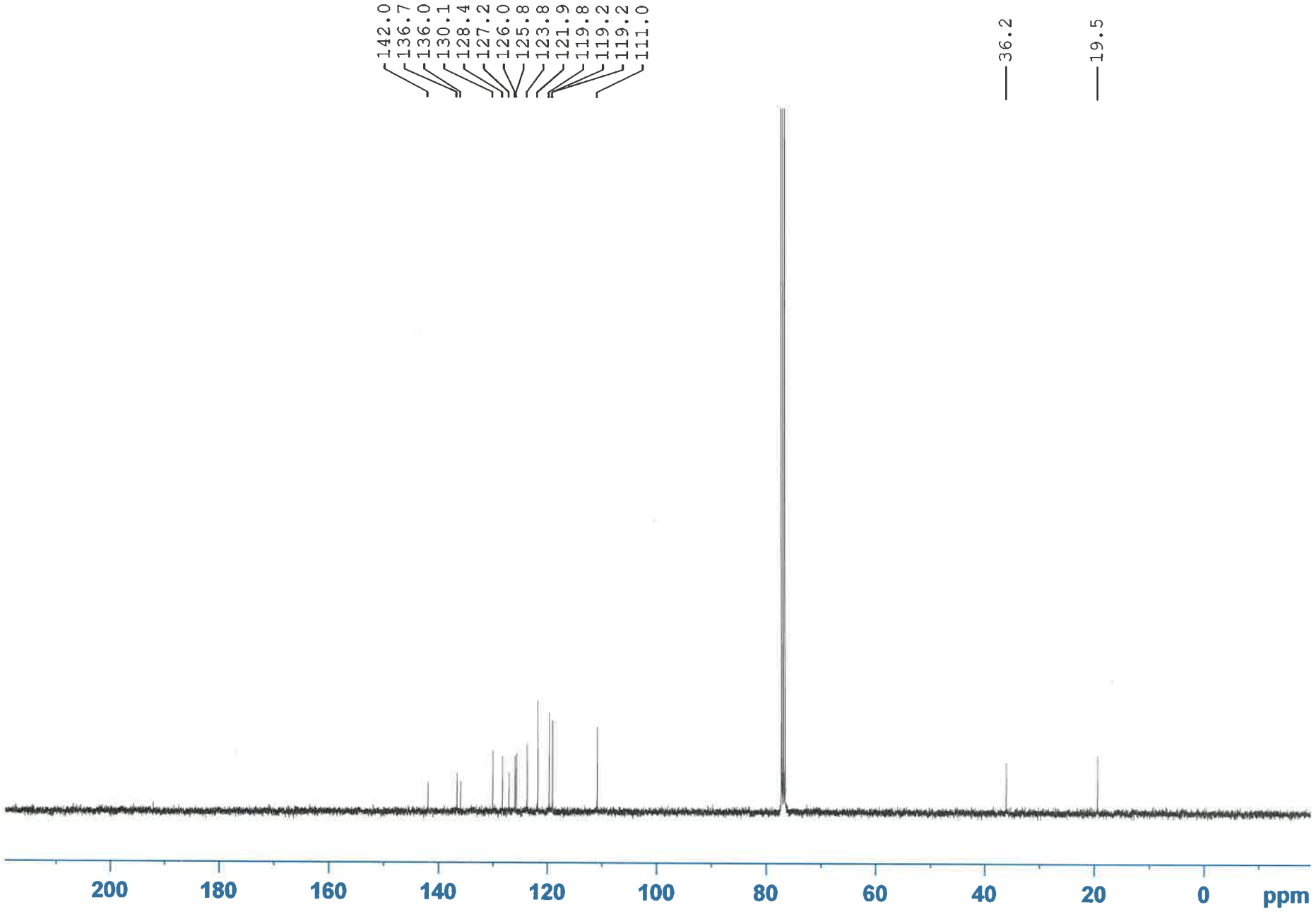
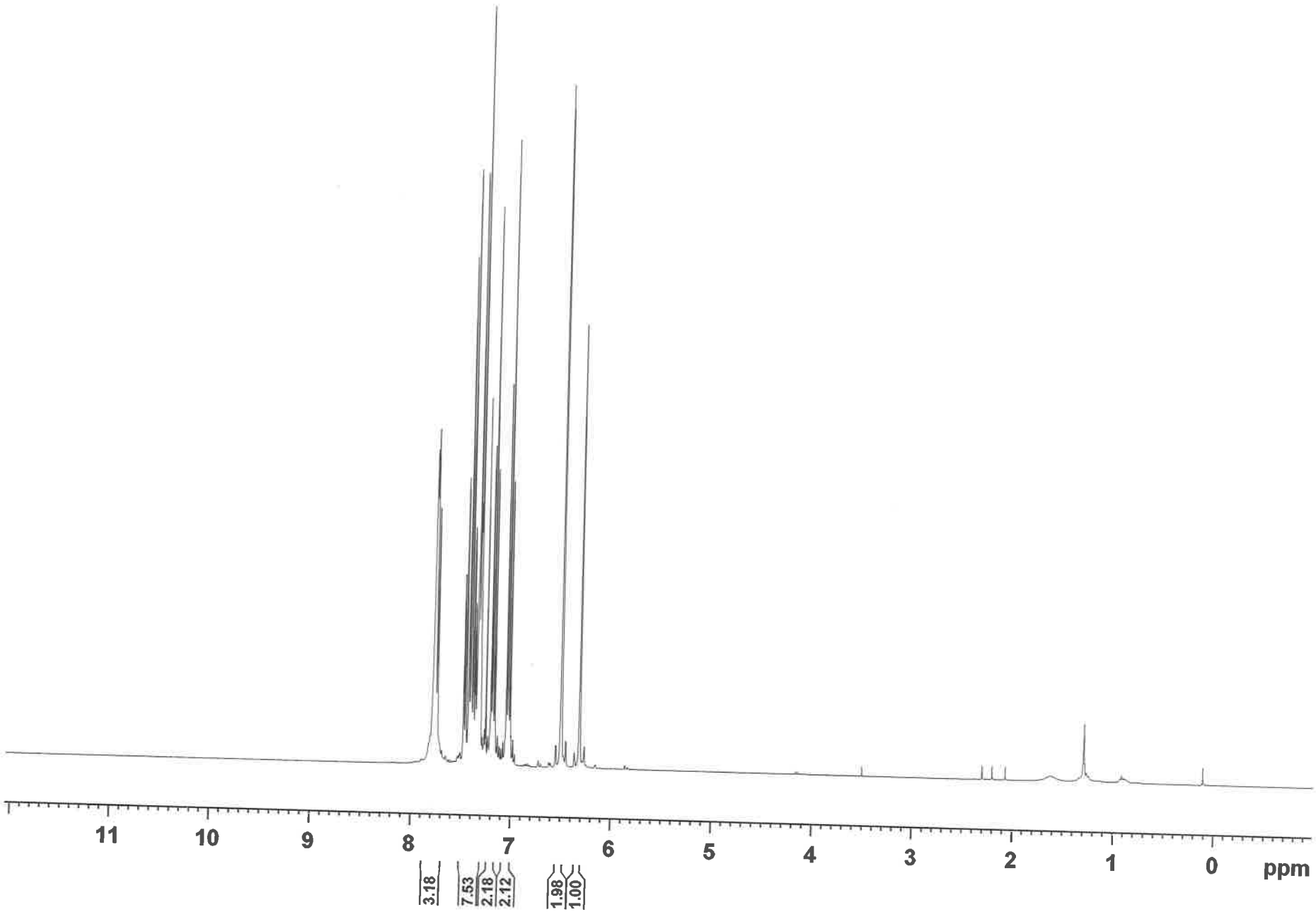
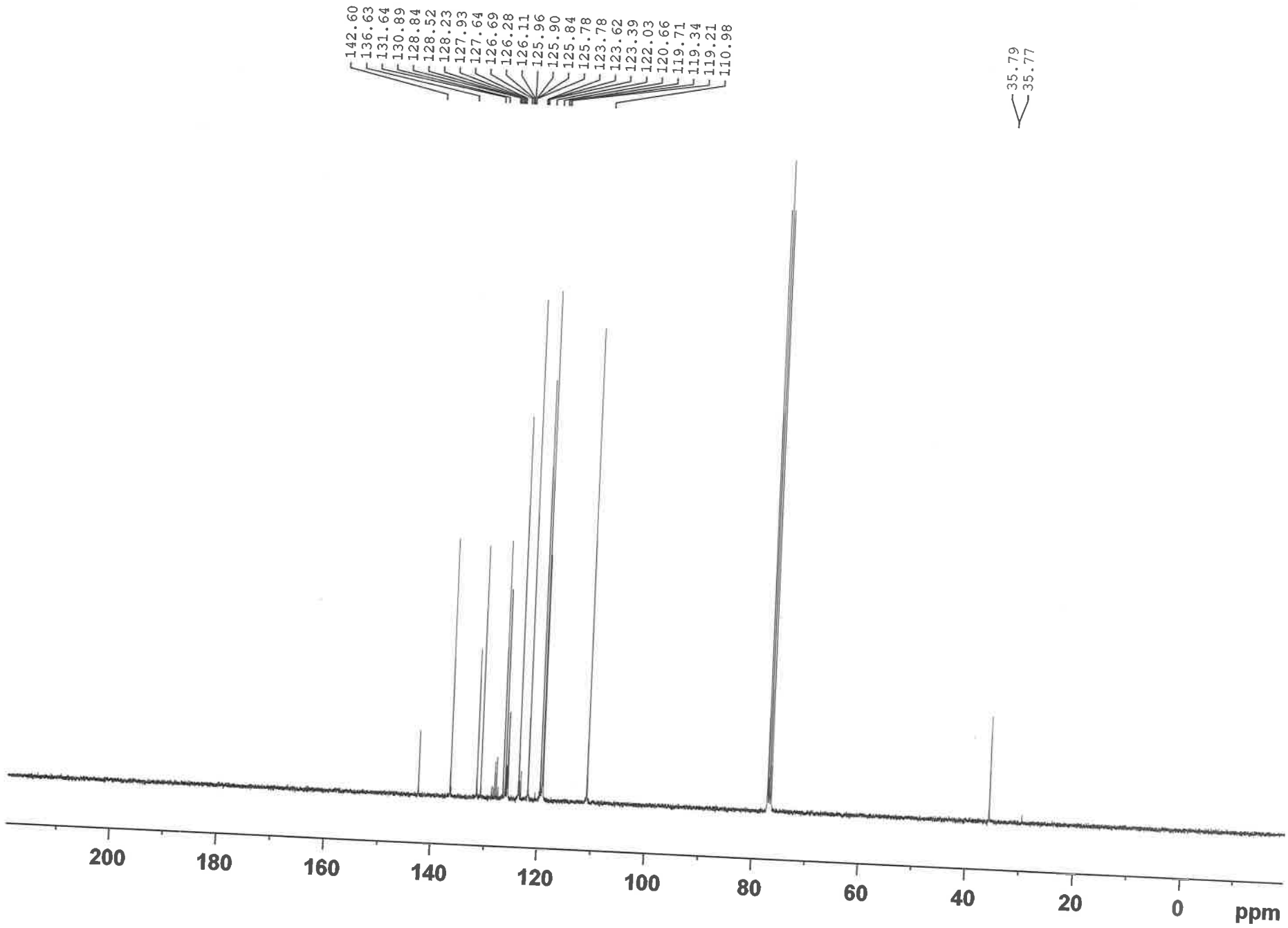


1H cbvip70 o-CF3 **8k**



13C cbvip70 o-CF3 **8k**



1H o-pyr **8l**

TrinPROTON ~~CD2Cl2~~ MeOH {C:\Bruker\TOPSPIN\CBrindle\Aug102015} OrgoLab 2

8.47
8.47
8.46
8.46
8.45
7.72
7.72
7.70
7.70
7.68
7.68
7.37
7.35
7.35
7.33
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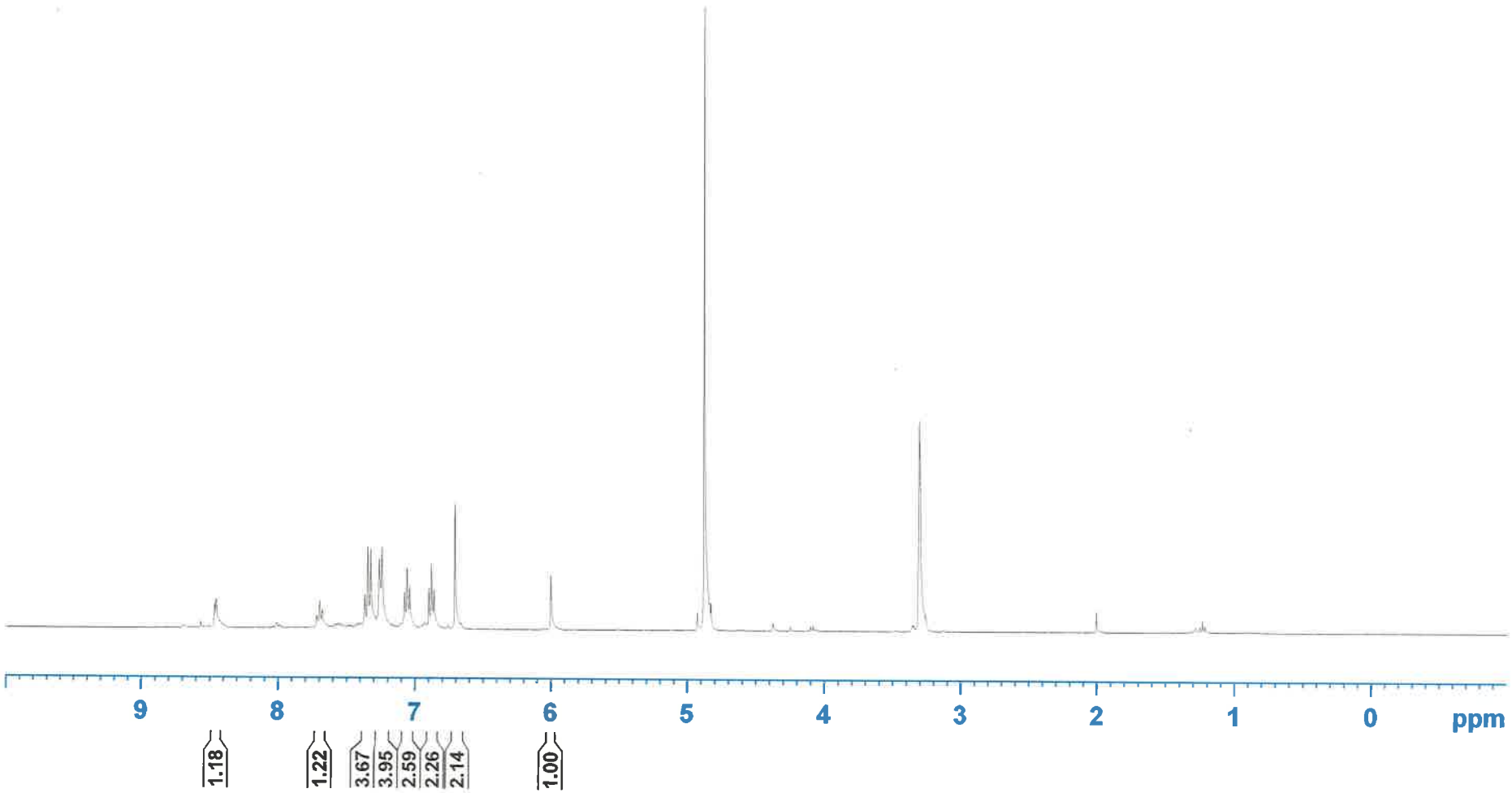
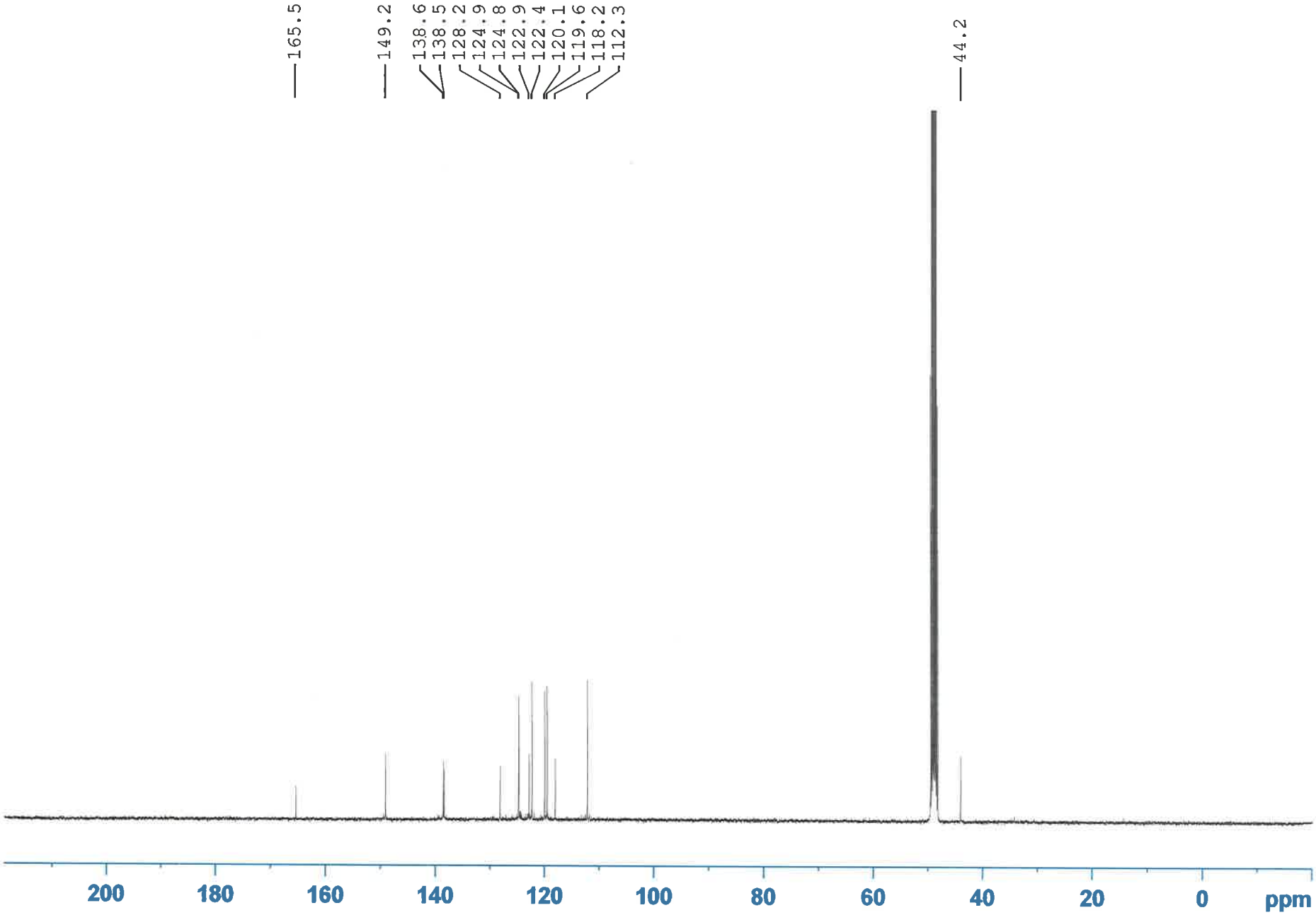


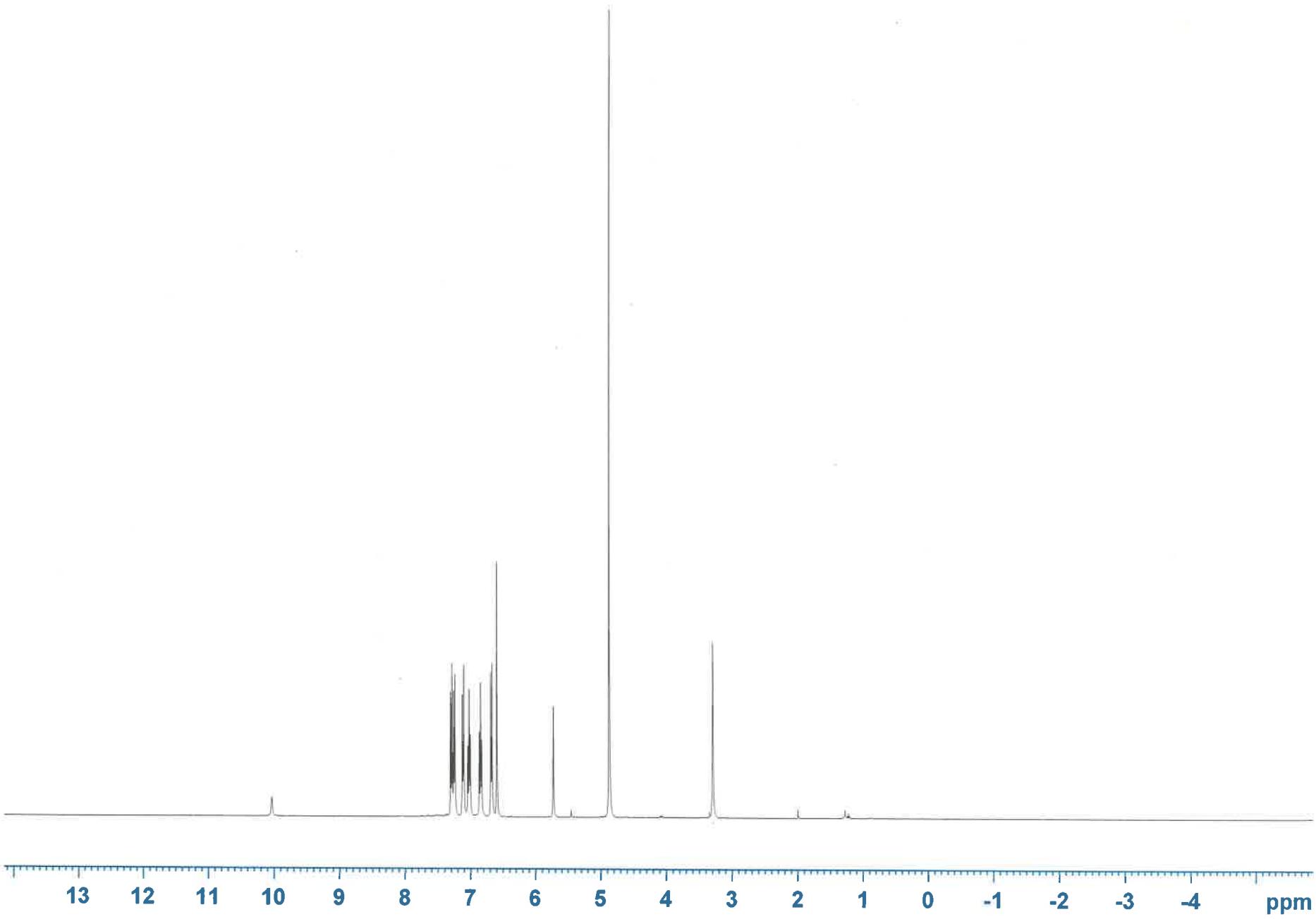
8l

13C o-pyr
C13CPD ~~CD2Cl2~~ MeOH {C:\Bruker\TOPSPIN\CBrindle\Aug102015} OrgoLab 2



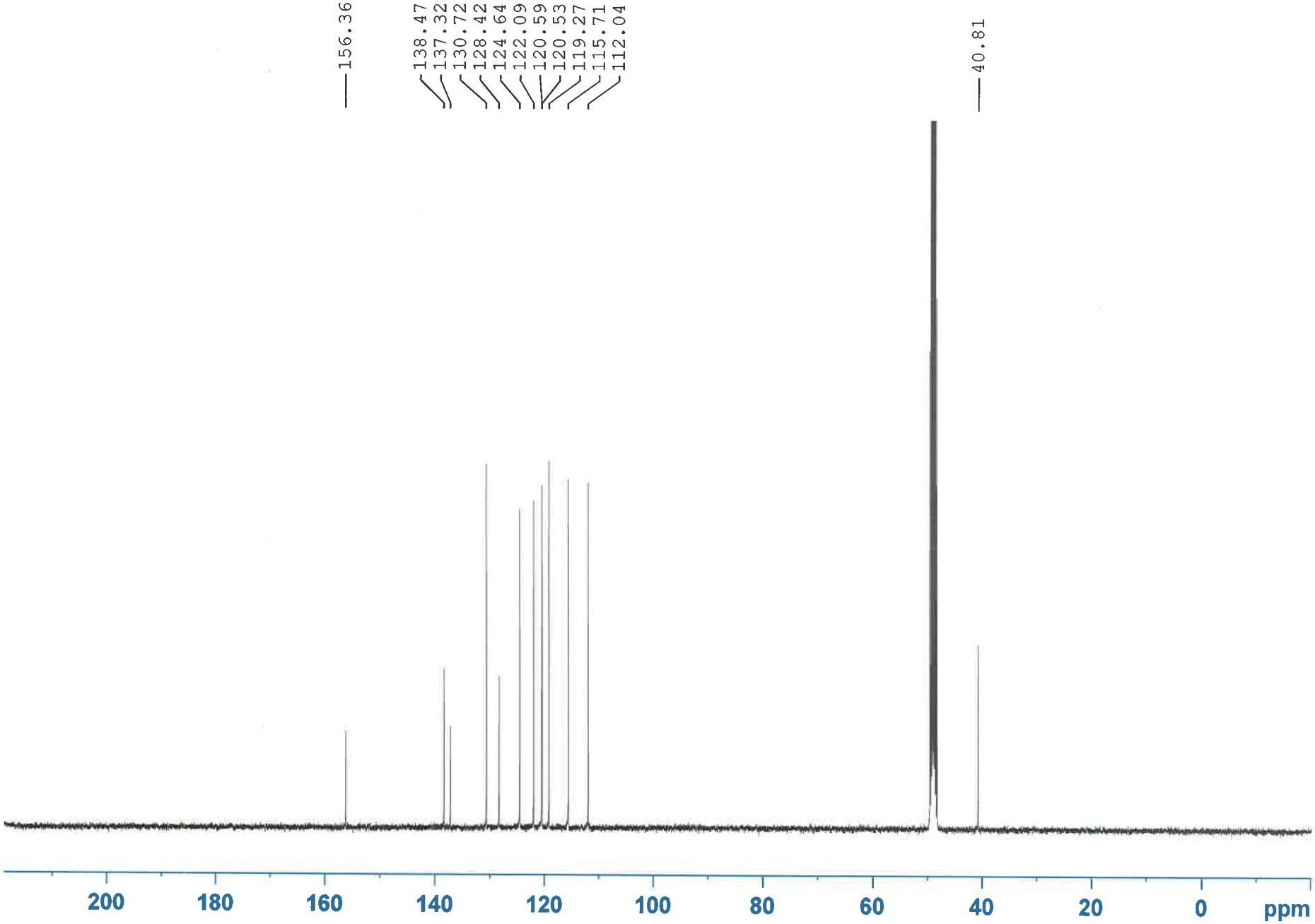
8m

BACp1 REDO Repurified High Vac.

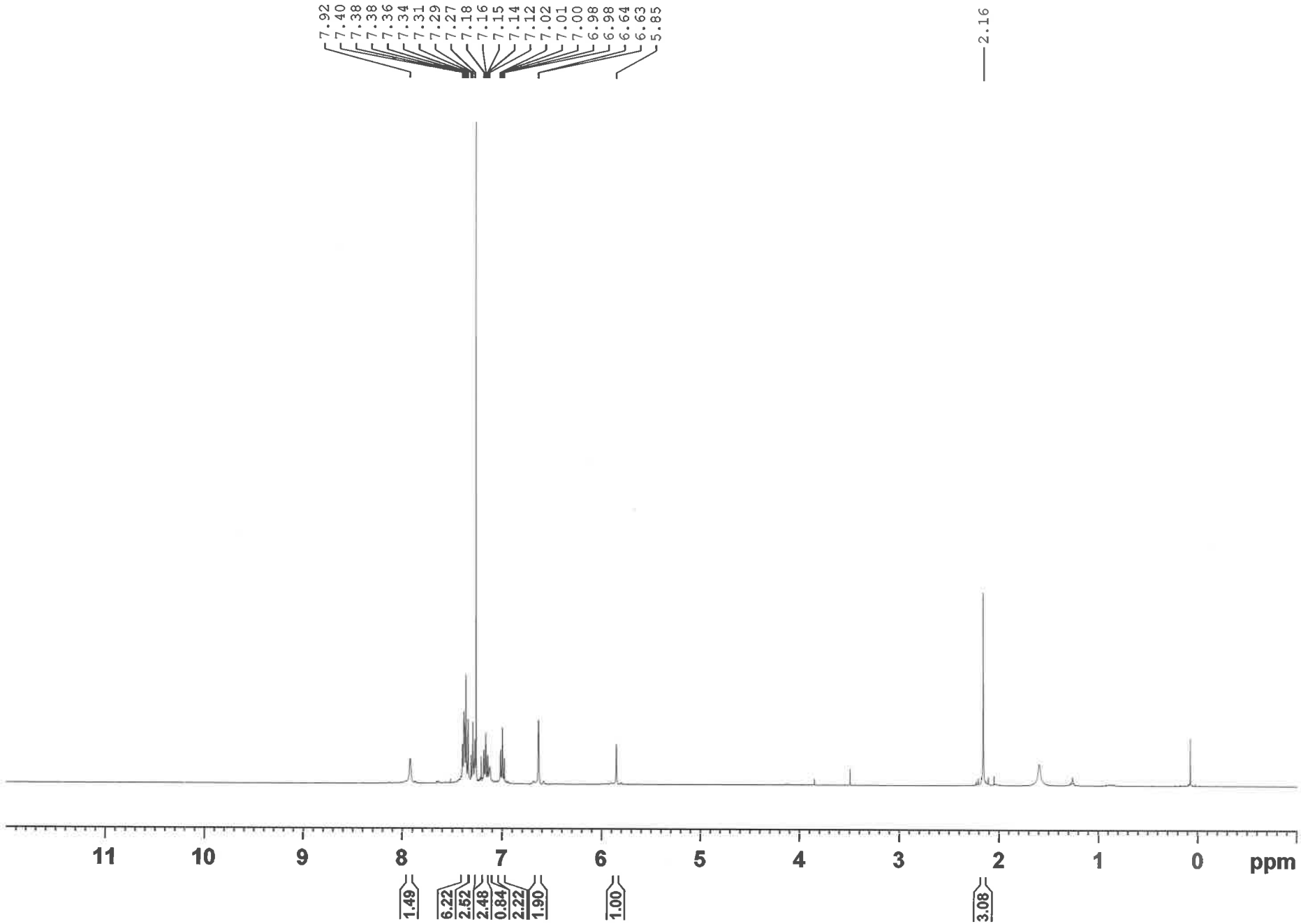


8m

briana chang hydroxy 13C
C13CPD CD2Cl2 {C:\Bruker\TOPSPIN} OrgoLab 3

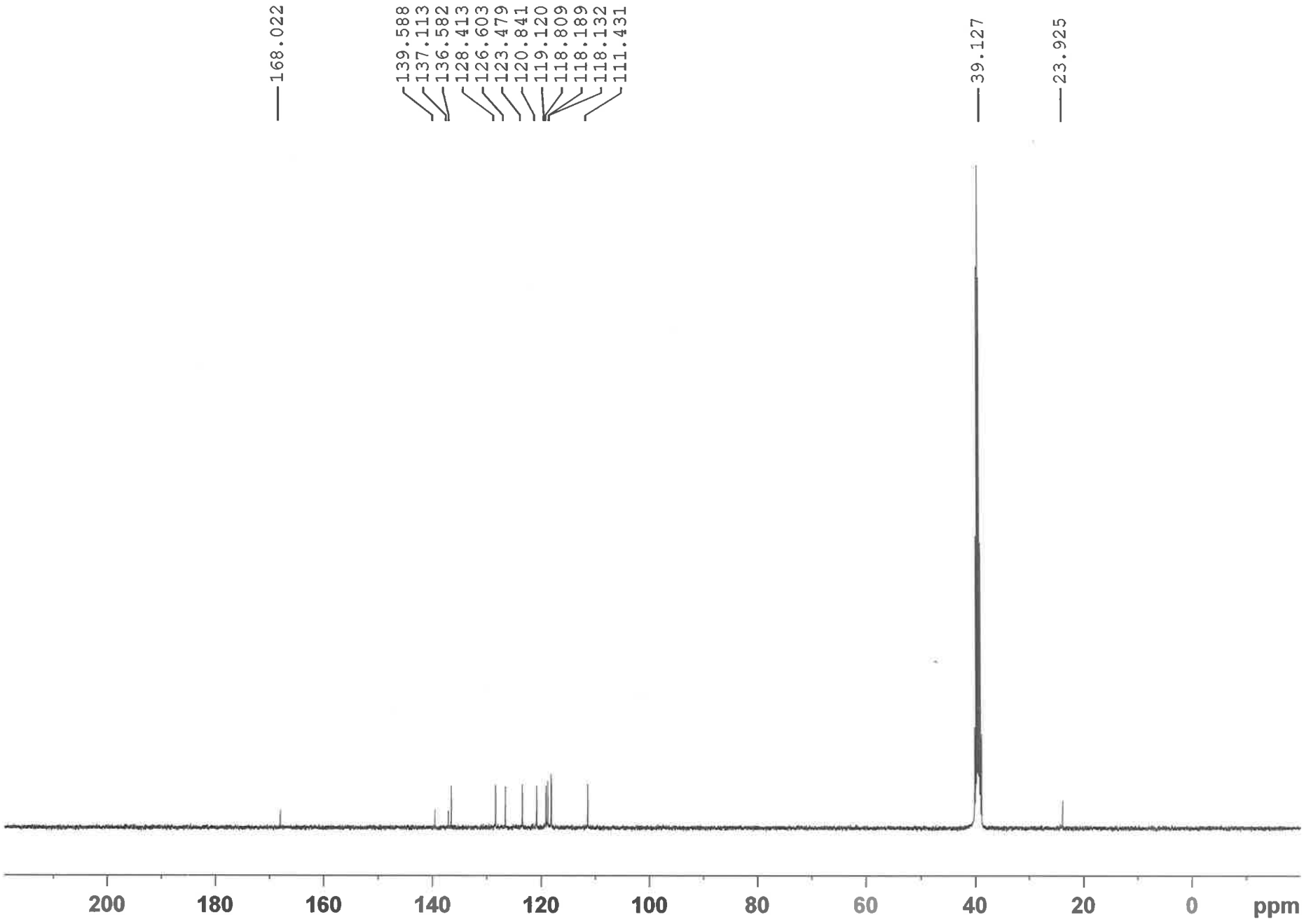


1H cbvip78 pNHAc **8n**



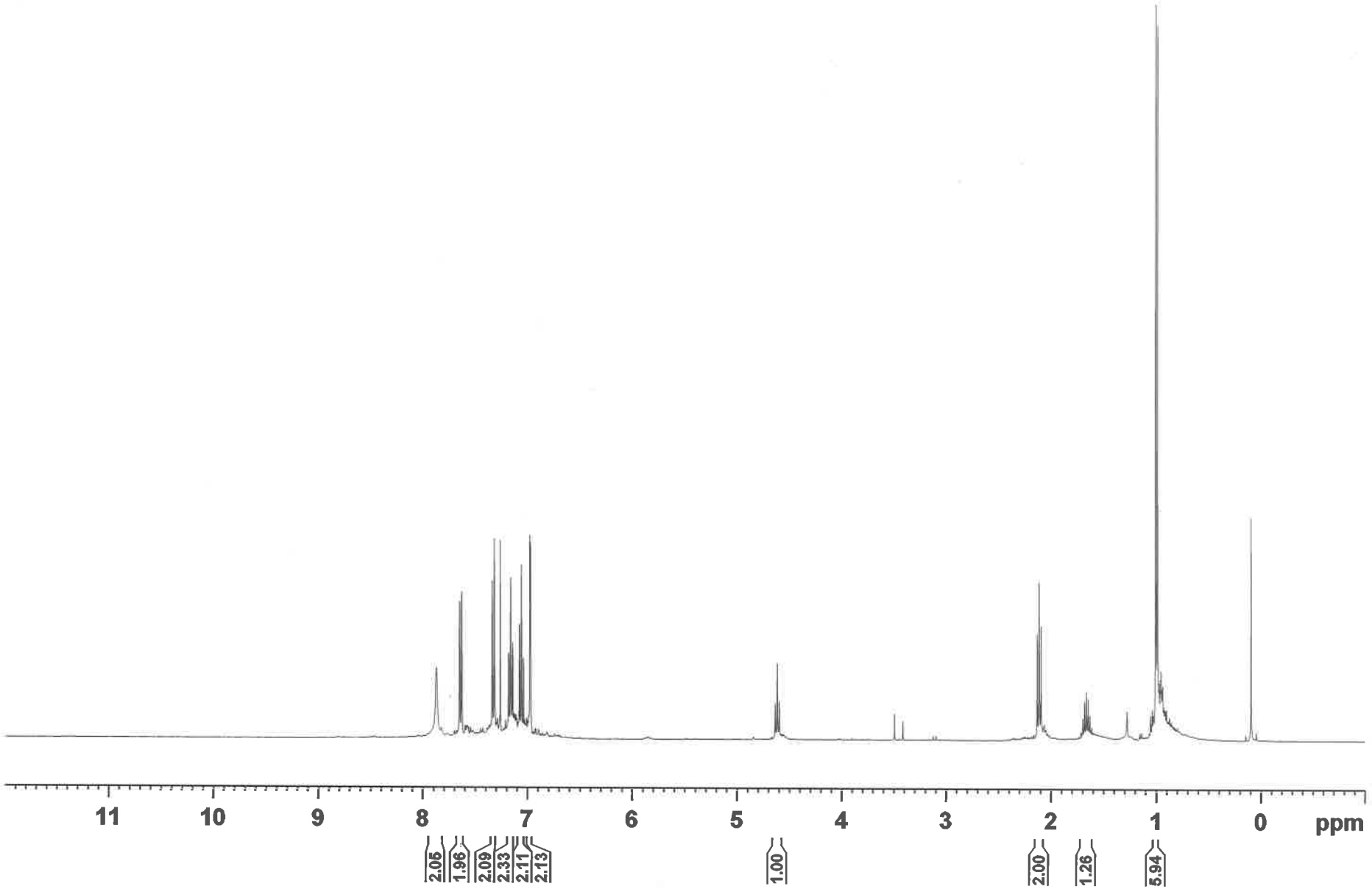
8n

CBVI p78
C13CPD DMSO {C:\Bruker\TOPSPIN\CBrindle\02042017}



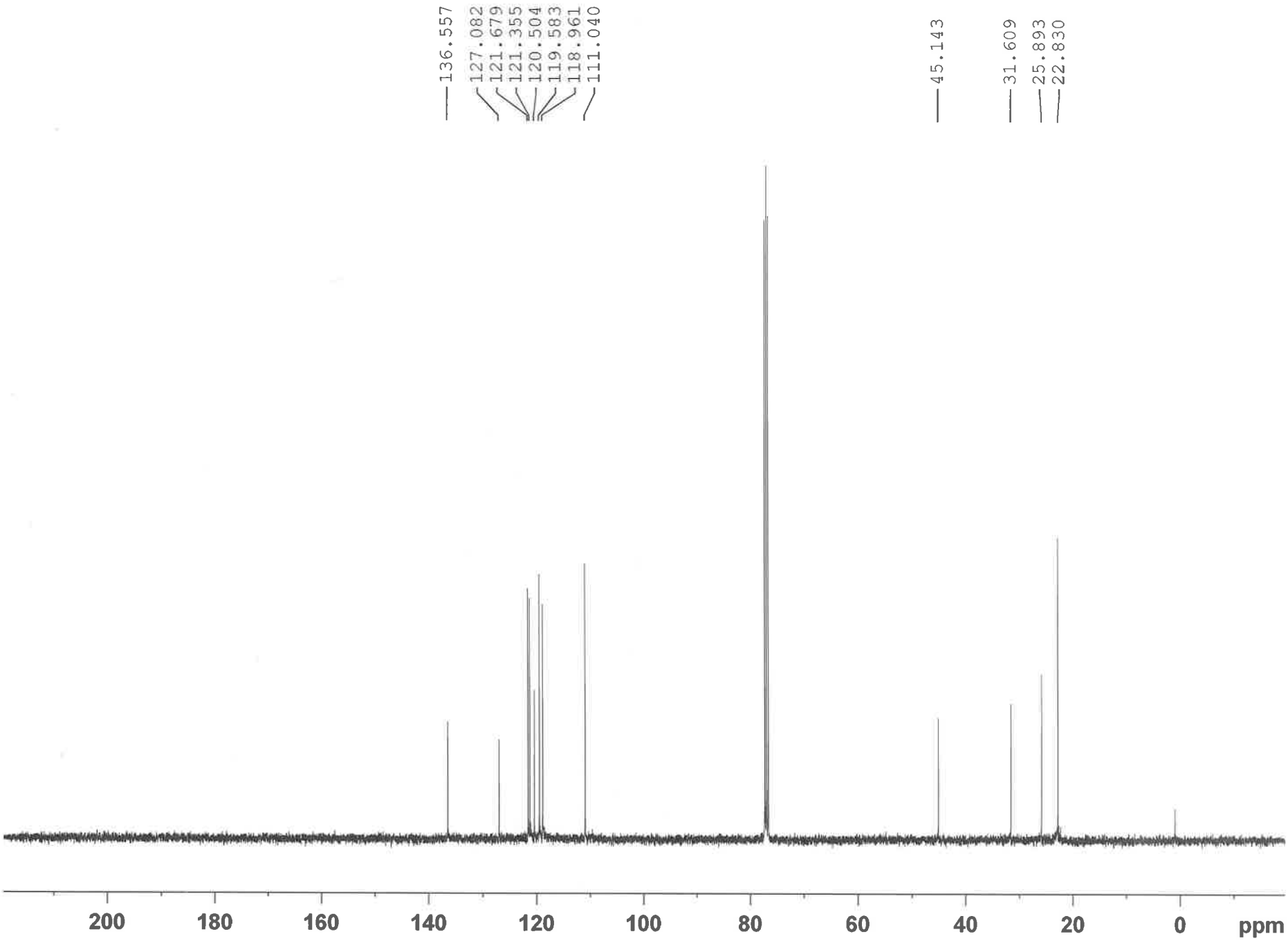
8o

1H cbvip71 isovaleraldehyde derived



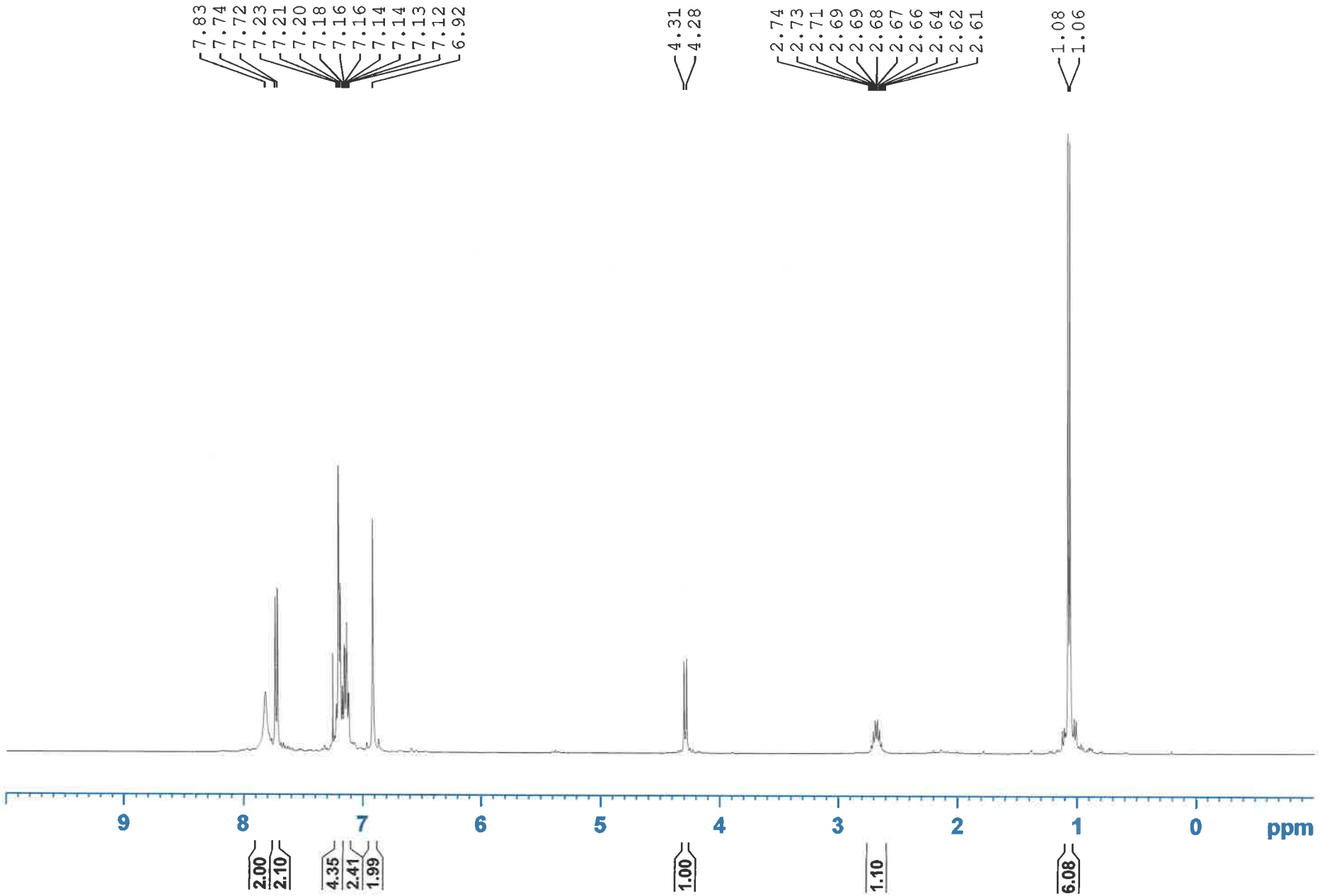
8o

13C cbvip71 isovaleraldehyde derived



8p

1H i-Pr
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\CBrindle\07132015} OrgoLab 4



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13C i-Pr
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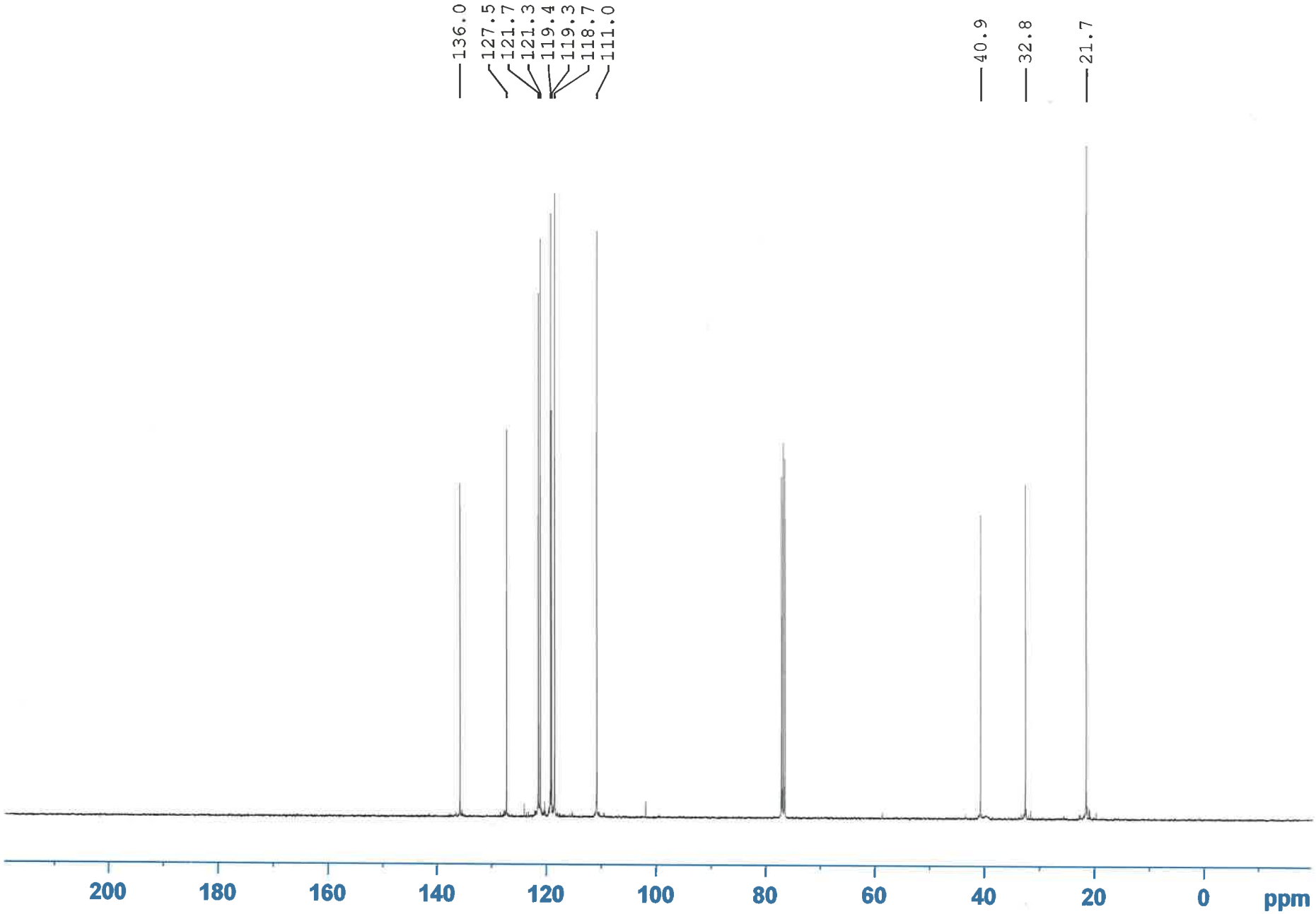
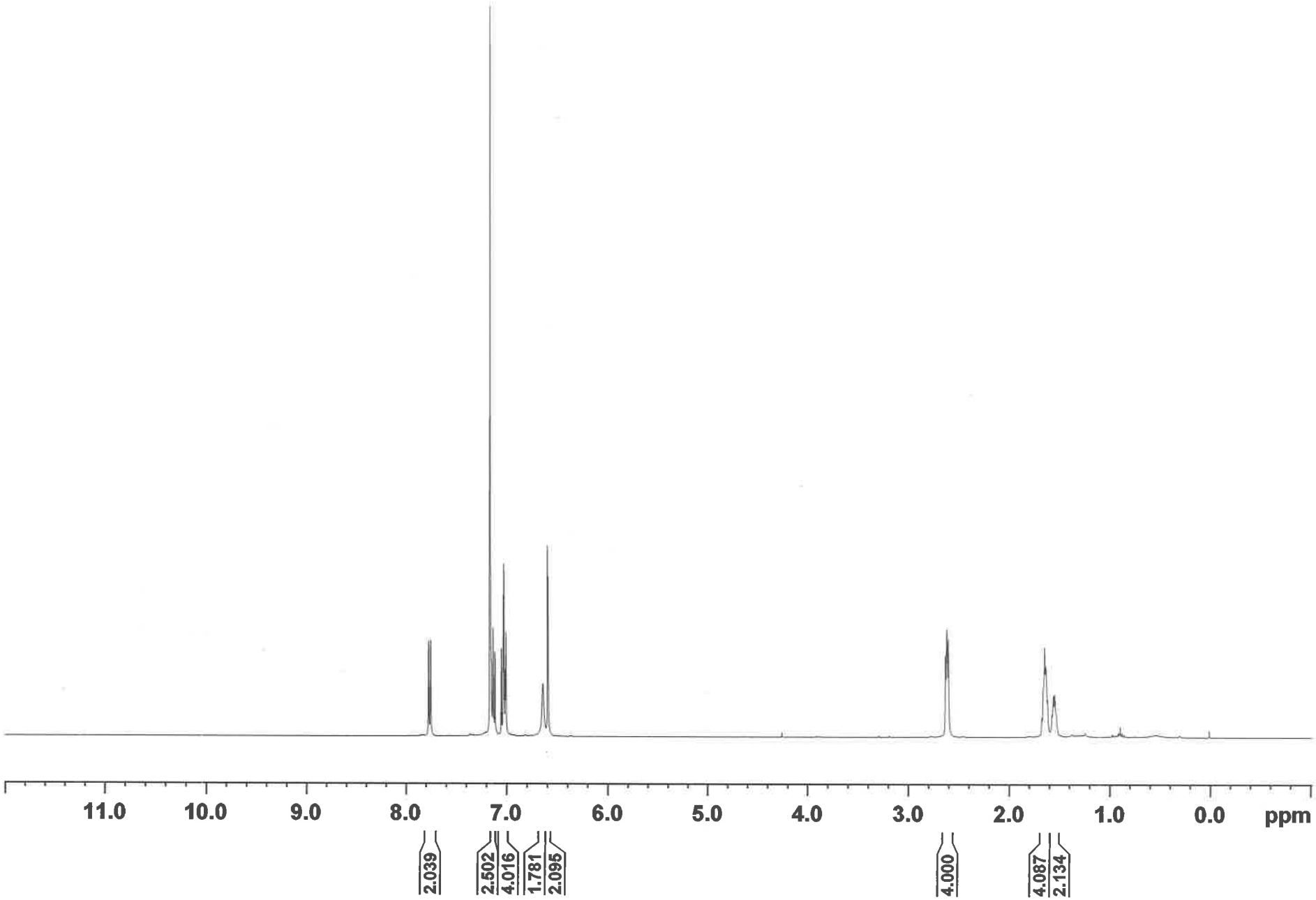
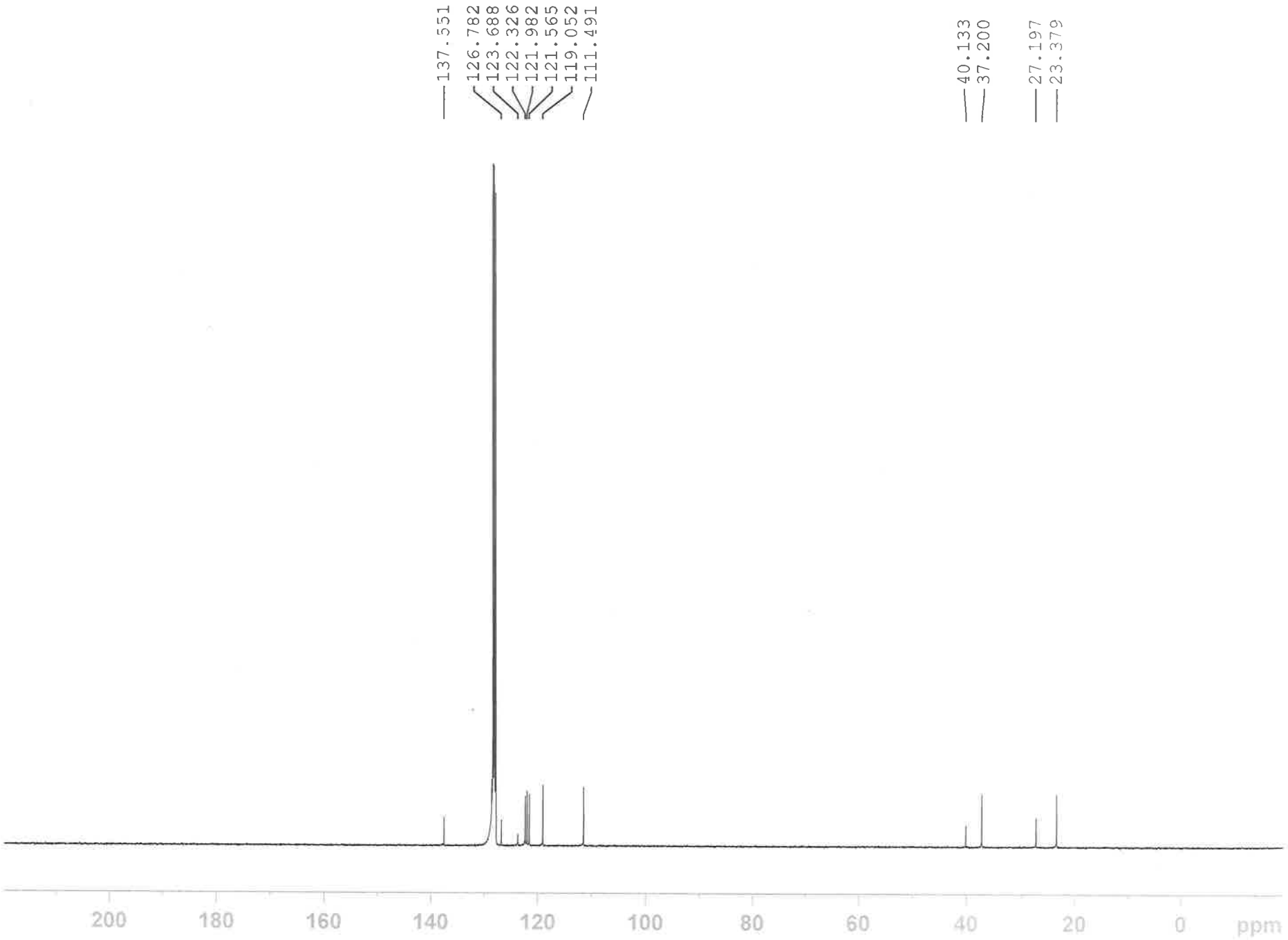


cbviip94C d6-benzene 8q

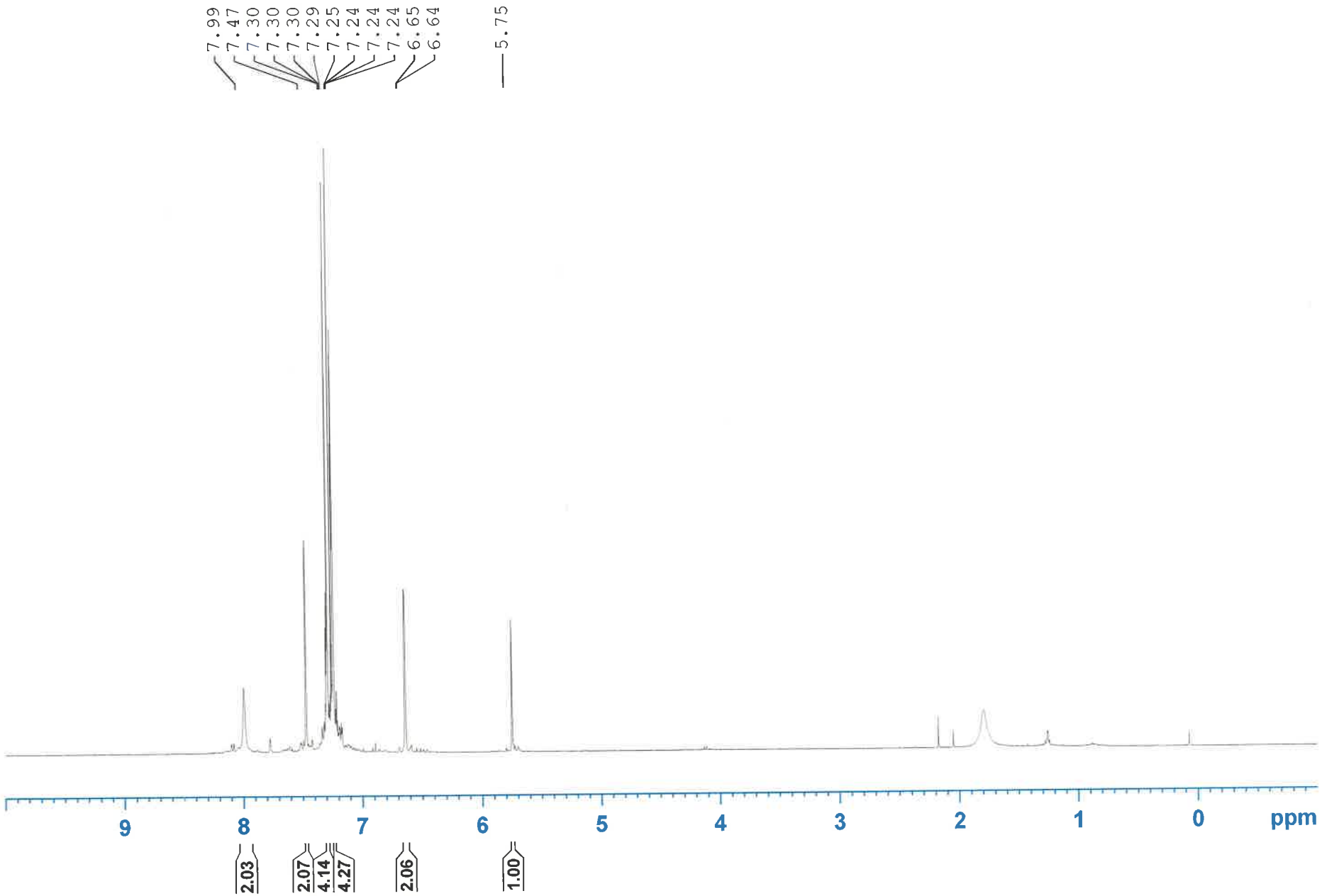


cbviip94C d6-benzene 13C 8q



8r

1H 5-Br
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\CBrindle\Aug27.2015} OrgoLab 1



8r

13C 5Br
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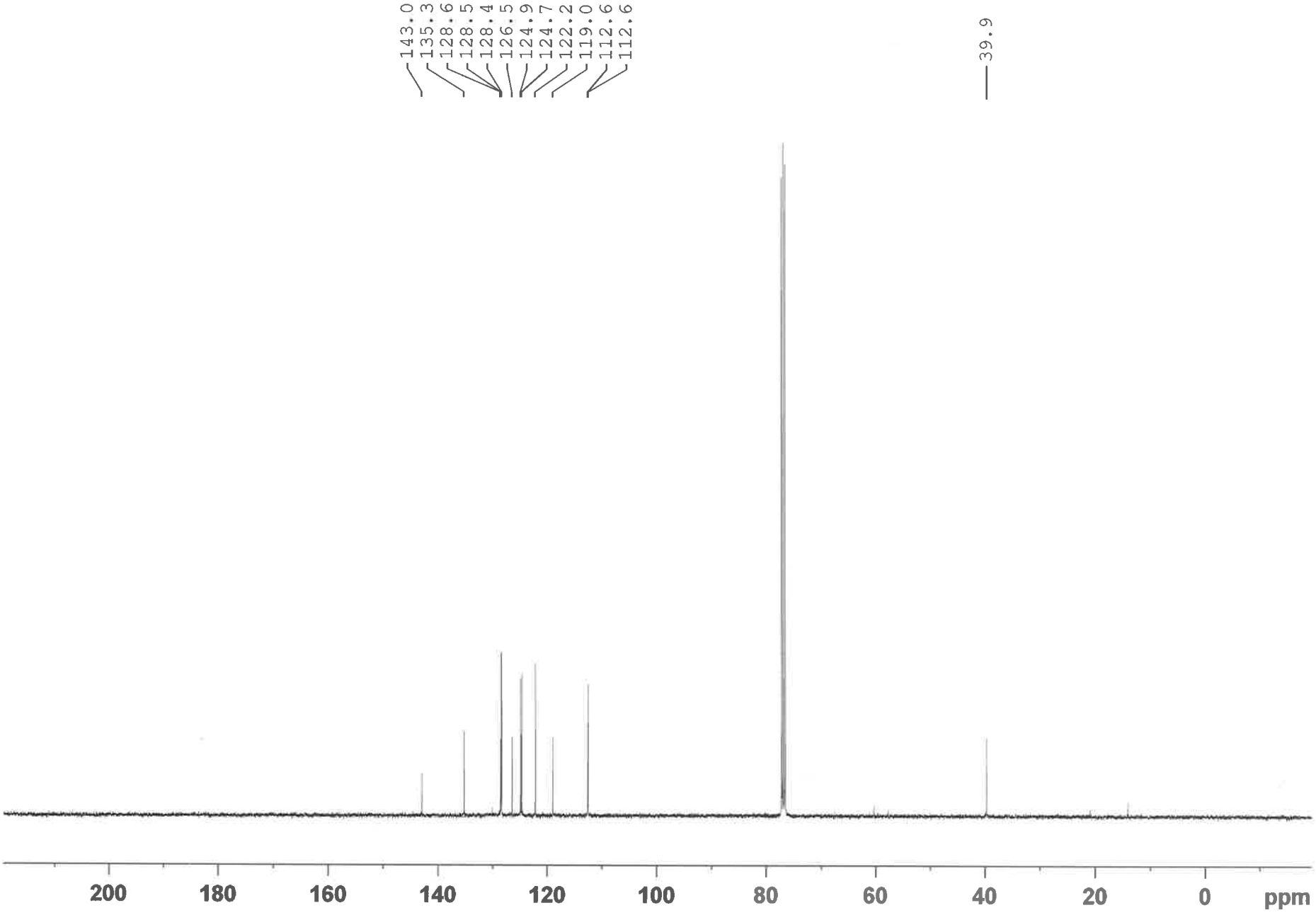
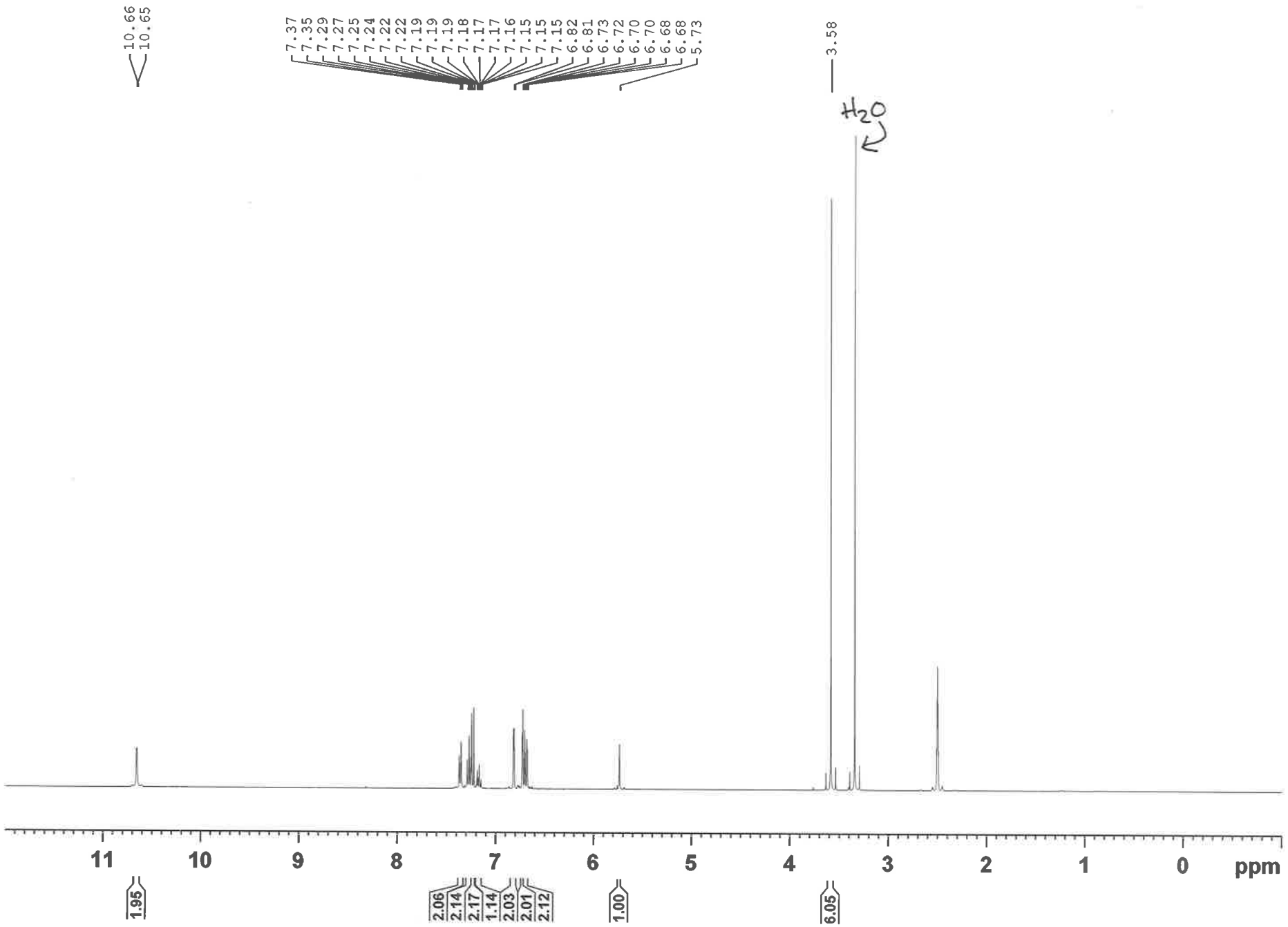
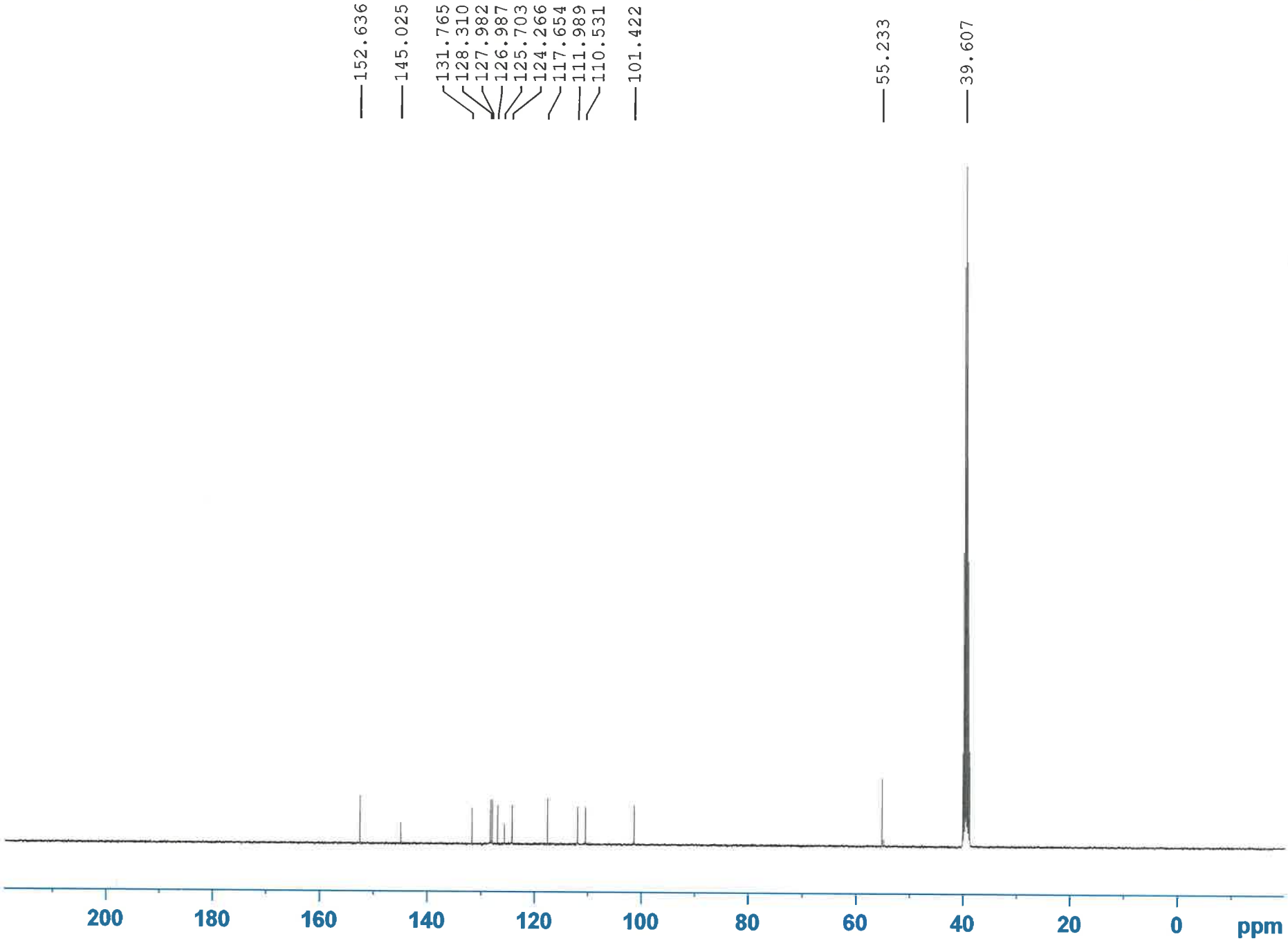


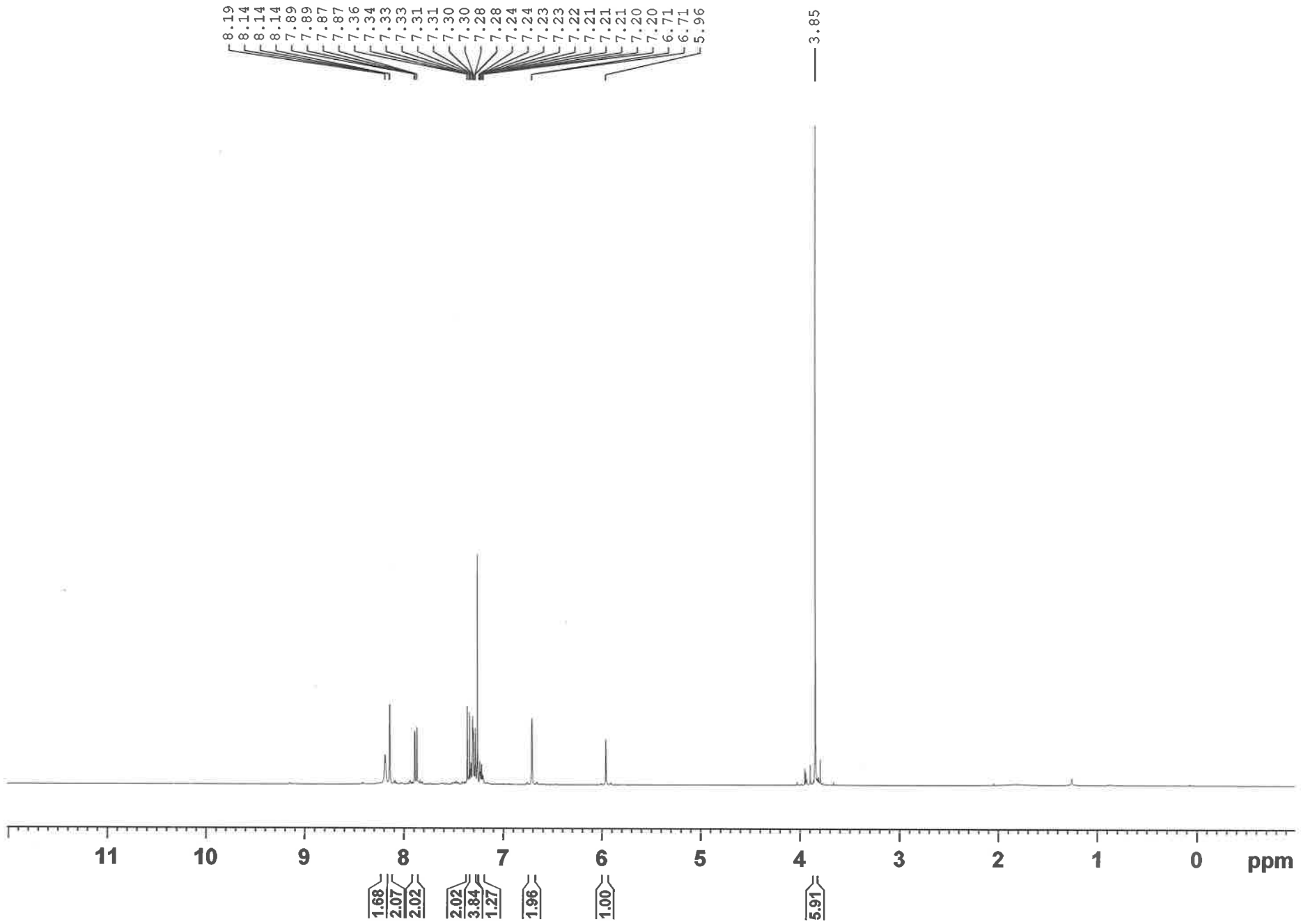
1H cbvip79 dmso 5OMe



13C cbvip79 dmso 5OMe **8s**

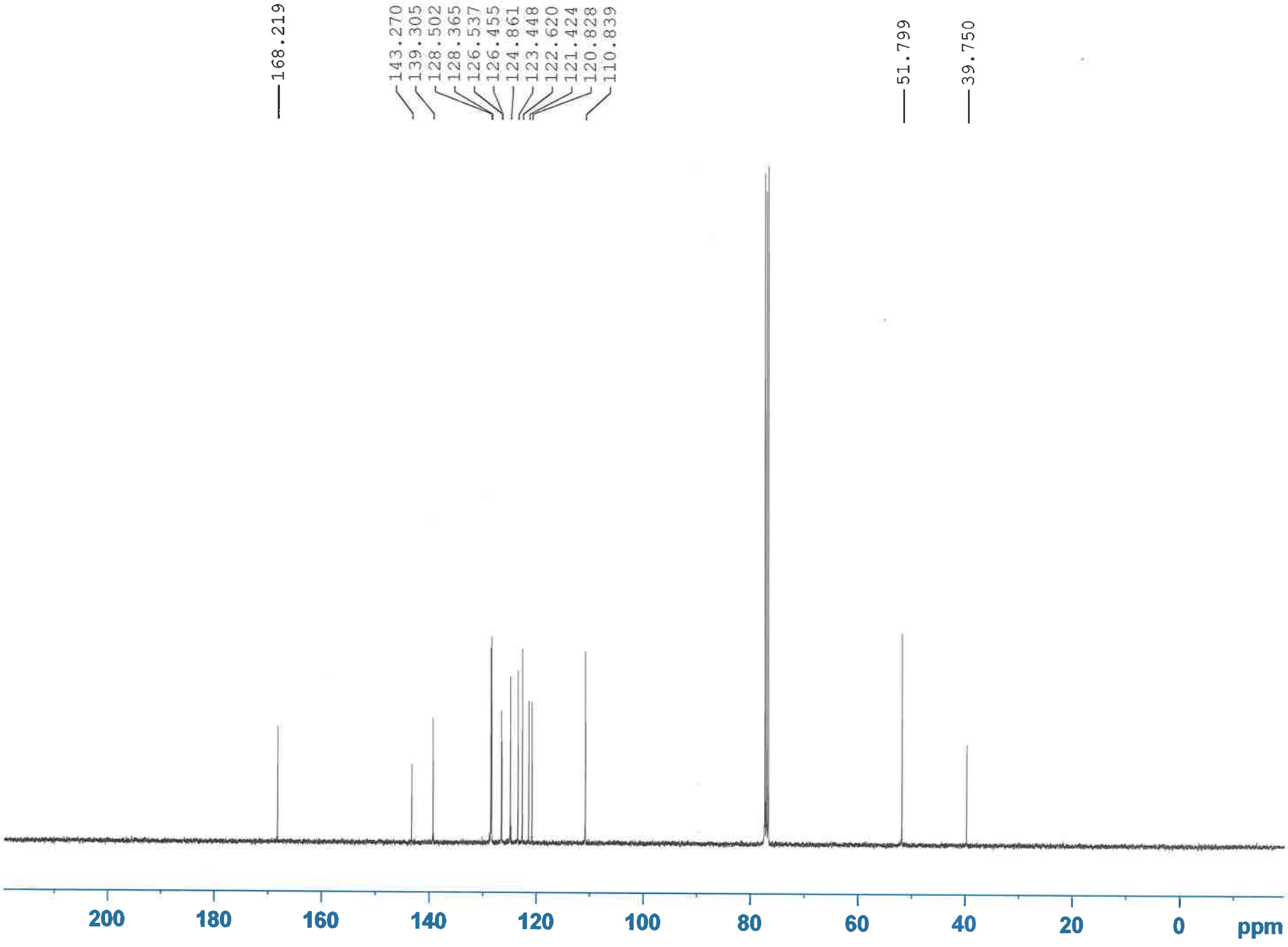


1H dcip31 5-CO2Me **8t**



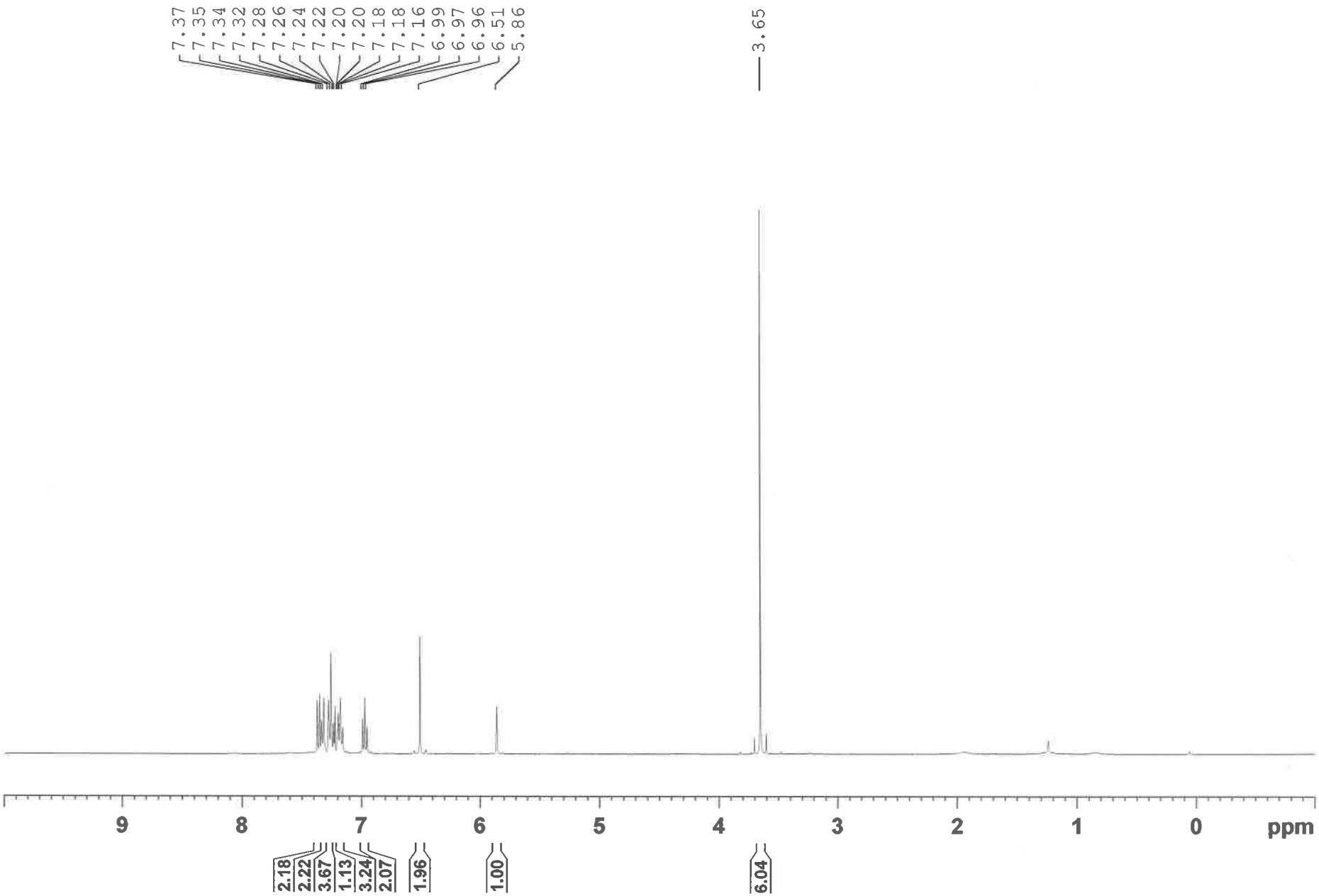
13C dcip31 5-CO2Me

8t



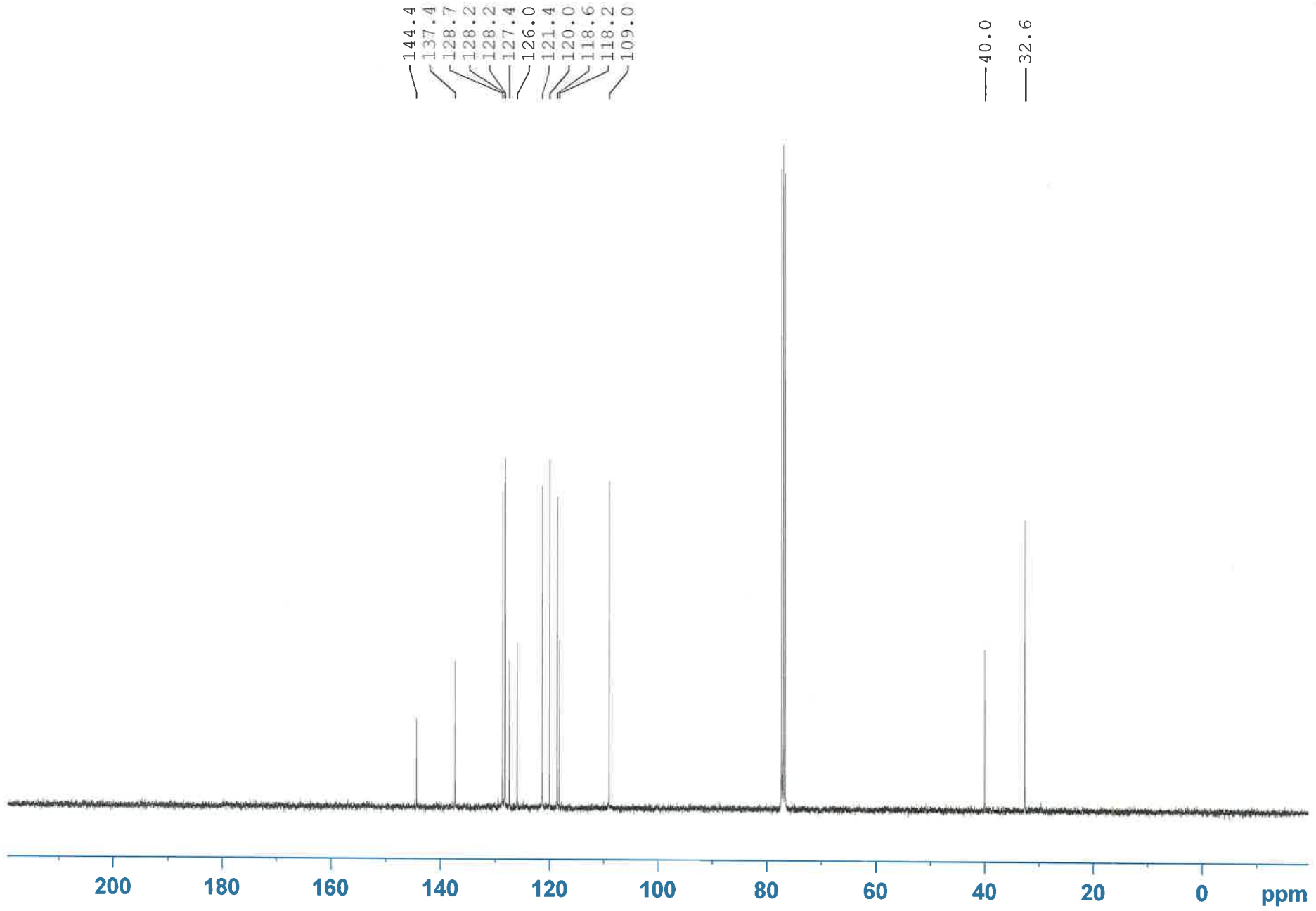
8u

Methyl Indole
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\PQuach\Jul152015} OrgoLab 15



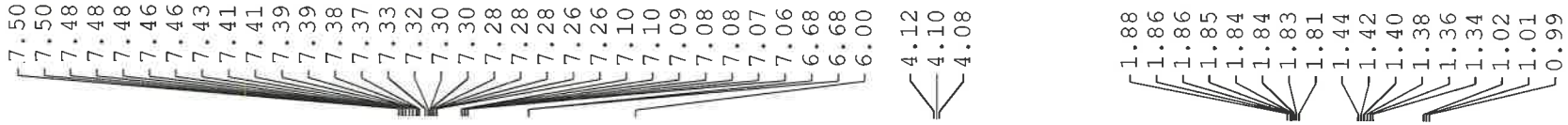
8u

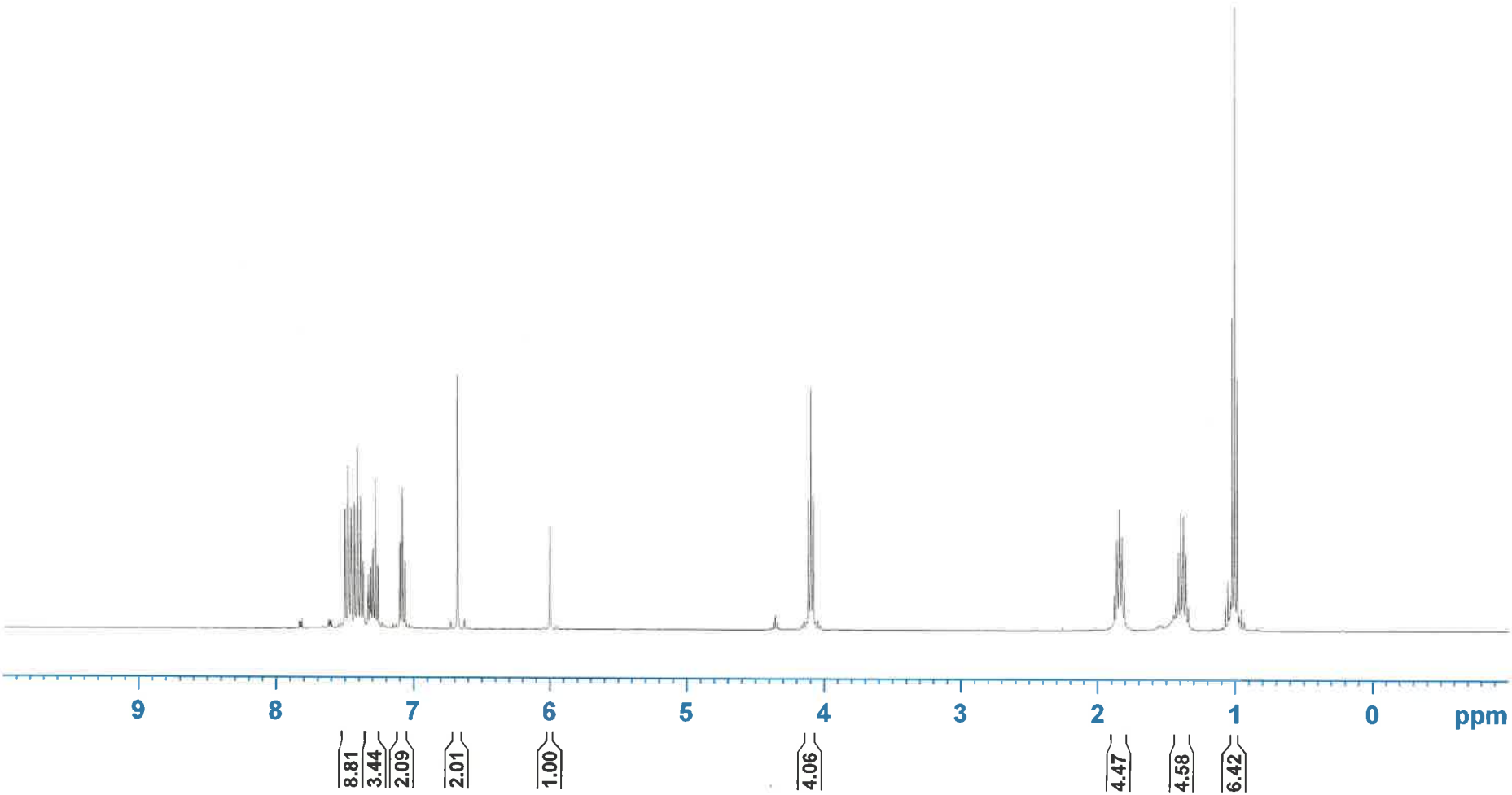
1 ~~Nalph~~ 33-40
C13CPD CDCl3 {C:\Bruker\TOPSPIN\PQuach\Jul152015} OrgoLab 15



8v

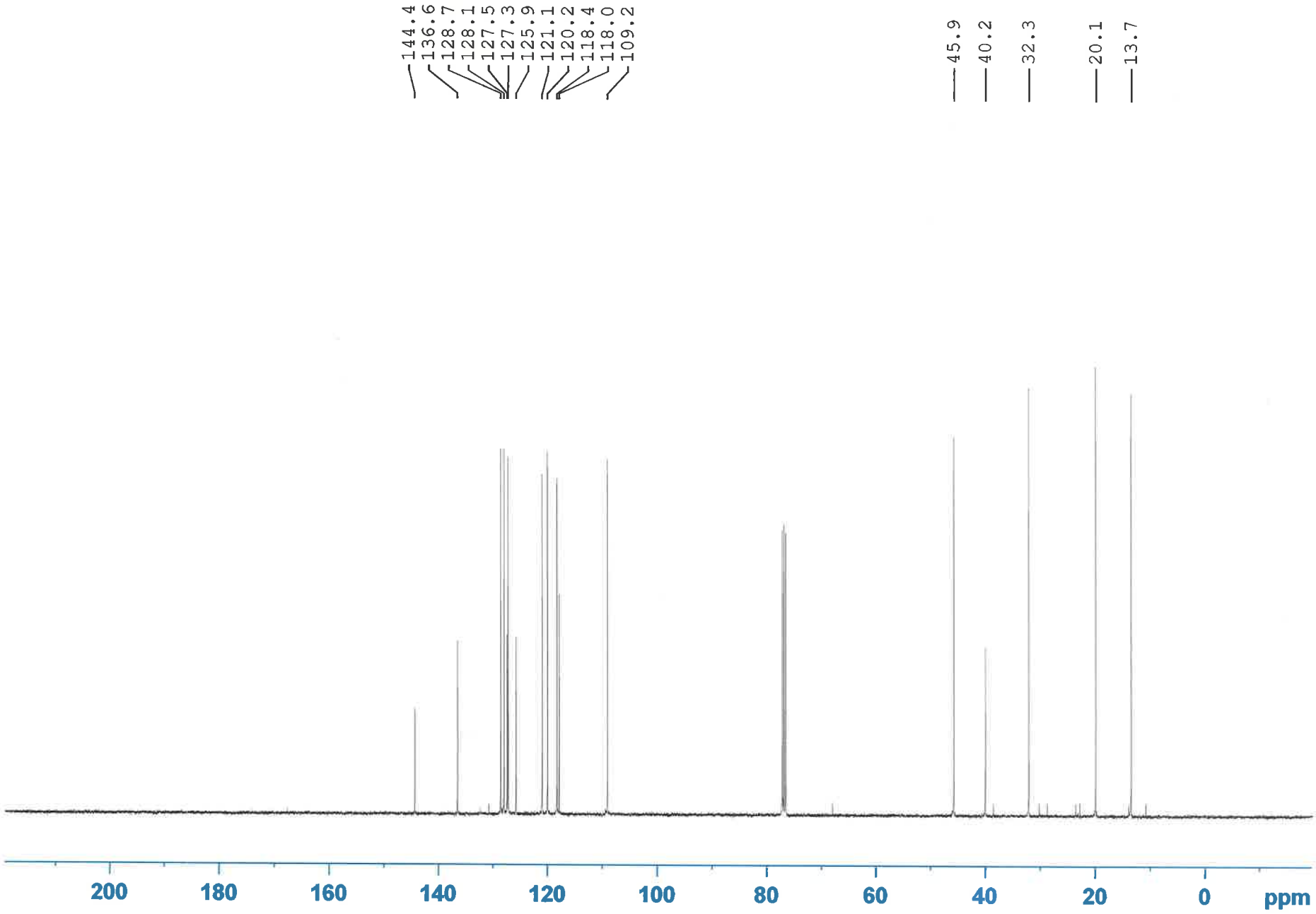
1H NnBu
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\CBrindle\Jul222015} OrgoLab 7





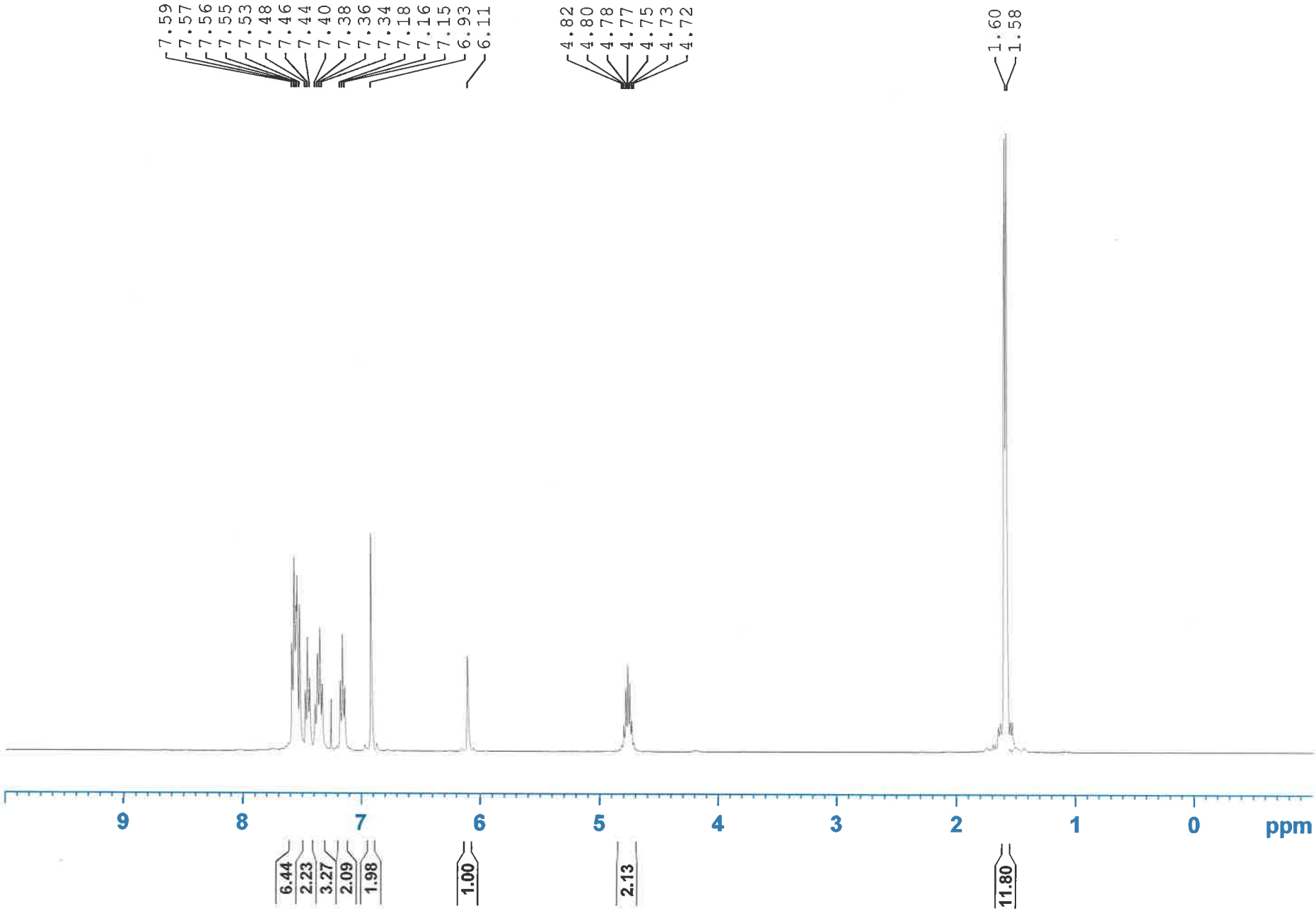
8v

13C NnBu
C13CPD CDCl3 {C:\Bruker\TOPSPIN\CBrindle\Aug142015} OrgoLab 3



8w

1H NiPr
TrinPROTON CDCl3 {C:\Bruker\TOPSPIN\CBrindle\Jul222015} OrgoLab 6



8w

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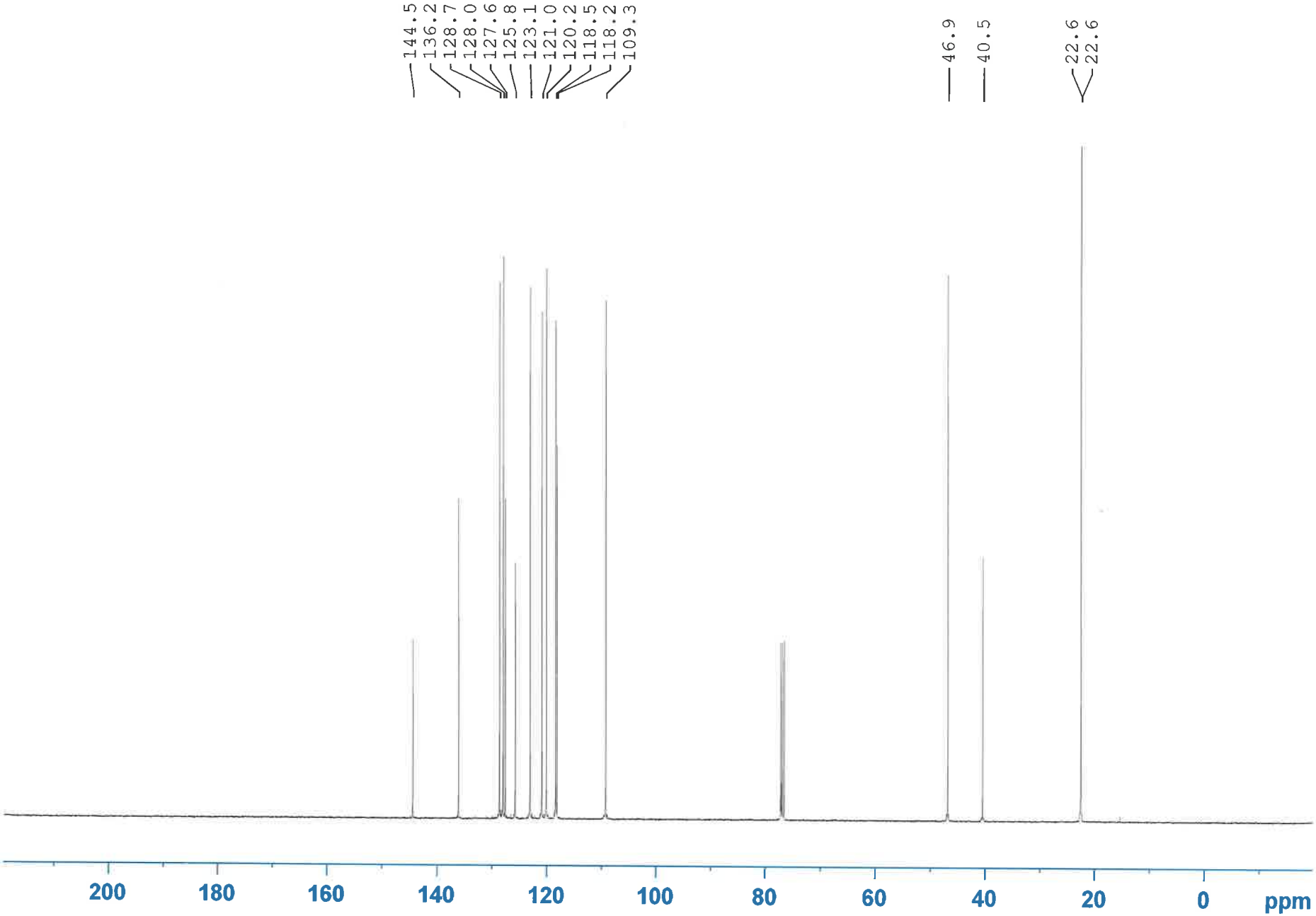
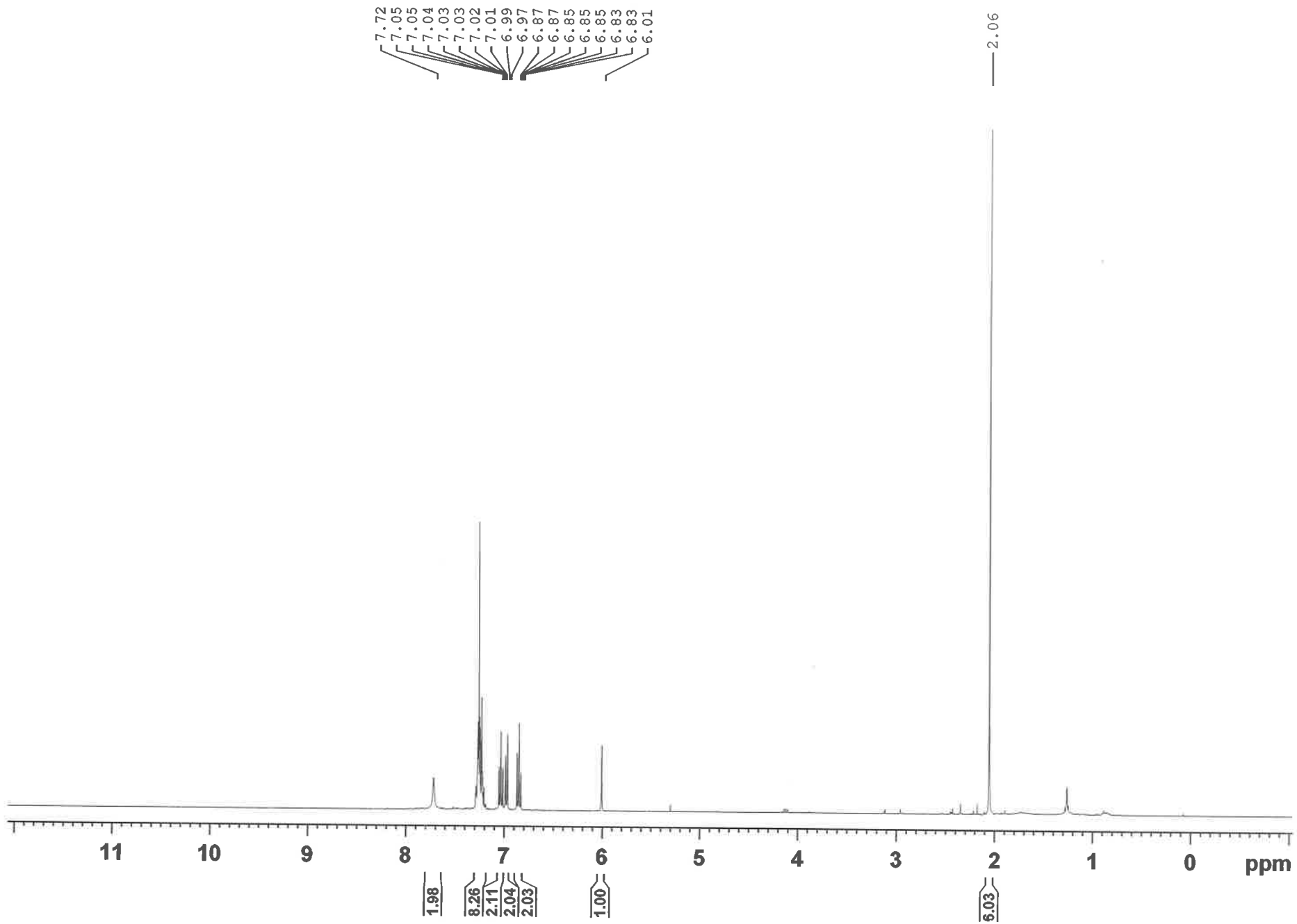
C13CPD CDCl3 {C:\Bruker\TOPSPIN\CBrindle\Jul222015} OrgoLab 1



WNTip71-Workup-2-Me-Indole **8x**



13C wntip71 2-Me indole adduct **8x**