Electronic Supplementary Material (ESI) for RSC Advances.

Pseudo Three-Component Approach to Coumarin-Annulated Azepines: Synthesis of Coumarin[3,4-*b*]azepines

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Supporting Information

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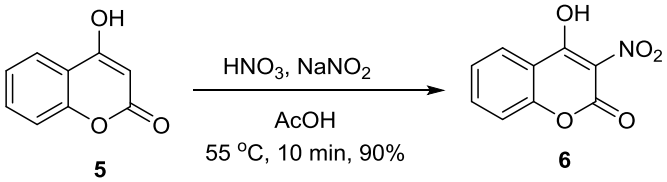
1. Instrumentation

Instrumentation. Melting points were determined on a Mel-Temp melting point apparatus in open capillaries and are uncorrected. Infrared (IR) spectra were recorded using 1725XFT-IR spectrophotometer. High resolution mass spectra (HRMS) were obtained on a Thermo Fisher Scientific Finnigan MAT95XL spectrometer using magnetic sector analyzer ^{1}H NMR (400 MHz) and ^{13}C NMR (100, or 150 MHz) spectra were recorded on a Varian VXR300 or Bruker 400/600 spectrometer. Chemical shifts were reported in parts per million on the δ scale relative to an internal standard (tetramethylsilane, or appropriate solvent peaks) with coupling constants given in hertz. ^{1}H NMR multiplicity data are denoted by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet. Analytical thin-layer chromatography (TLC) was carried out on Merck silica gel 60G-254 plates (25 mm) and developed with the solvents mentioned. Visualization was accomplished by using portable UV light, ninhydrin spray and iodine chamber. Flash chromatography was performed in columns of various diameters with Merck silica gel (230-400 mesh ASTM 9385 kieselgel 60H) by elution with the solvent systems. Solvents, unless otherwise specified, were reagent grade and distilled once prior to use. All new compounds exhibited satisfactory spectroscopic and analytical data.

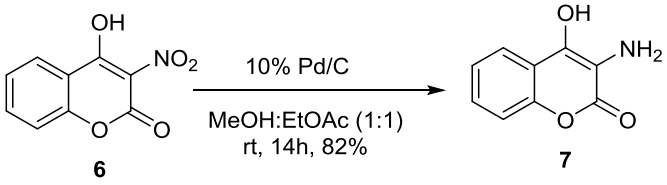
2. Synthesis of 4-hydroxy-3-nitrocoumarin (**6**)



5 **6**

To a stirred suspension of 4-hydroxycoumarin (**5**, 4.0 g, 24.67 mmol, 1.0 equiv.) in AcOH (50 mL) was added sodium nitrite (17 mg, 0.25 mmol, 0.01 equiv.) in one portion and HNO_3 (3.5 mL) dropwise. The resulting mixture was stirred at room temperature for 5 min and followed by heating at 70 °C for 30 min in an oil bath. As the brown solution attained to room temperature, the pure compound crystallized out from the solution. These crystals were filtered, washed with hexanes (6 x 50 mL), and dried *in vacuo* to afford 3-nitro-4-hydroxycoumarin (**6**) as off-yellow shiny crystals. 4.6 g; yield 90%; R_f = 0.15 (10% MeOH/DCM); mp 176–177 °C (lit.[1] 177 °C); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.41 (bs, 1H), 7.88 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.52 (td, *J* = 8.1, 1.8 Hz, 1H), 7.24–7.16 (m, 2H).

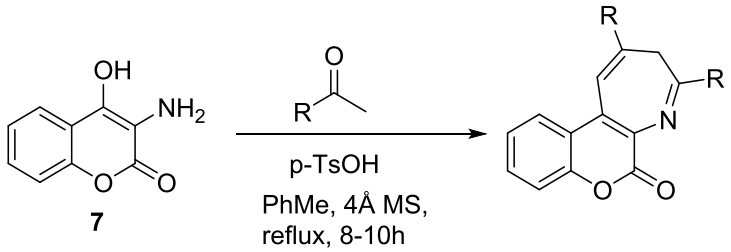
3. Synthesis of 3-amino-4-hydroxycoumarin (**7**)



6 **7**

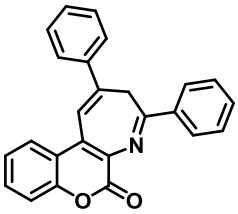
To a suspension of 4-hydroxy-3-nitrocoumarin (**6**, 4.0 g, 22.58 mmol) in ethanol (200 mL) was added 10% Pd/C (50 mg) at room temperature. The resulting solution was stirred under H_2 atmosphere for 6 hours at that temperature. After the completion of the reaction, the suspension was filtered through the celite pad, washed extensively with MeOH (3 x 30 mL each), acetone (3 x 30 mL), and concentrated to afford an off-white solid. 2.8 g; yield 82%; R_f= 0.16 (10% MeOH/DCM); mp 216 °C (lit.[2] 222–224 °C); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.19 (bs, 2H), 7.84 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.47 (td, *J* = 7.6, 1.6 Hz, 1H), 7.25–7.21 (m, 2H).

4. General procedure for the preparation of synthesis of coumarin-fused azepines (4)

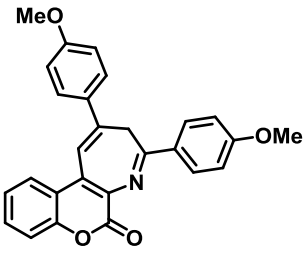


To a 100 mL round bottom flask was charged with 3-amino-4-hydroxycoumarin (1.0 equiv.), acetophenone derivative (2.1 equiv.), *p*-TsOH (0.2 equiv.), and 4 Å molecular sieves in anhydrous toluene (25 mL). The resulting mixture was refluxed for about 8-10 h. The dark solution was allowed to attain to the room temperature and the solvent was evaporated *in vacuo*. The residue was redissolved in DCM (50 mL), filtered, and washed with copious amounts of DCM. The resulting solvent was concentrated *in vacuo* and the product was subjected to flash column chromatography to afford the coumarin-fused azepine derivative.

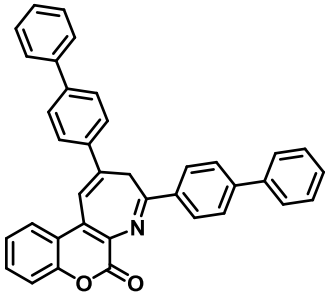
4a. off-yellow solid; yield 80%; R_f = 0.42 (30% EtOAc/hexanes); mp 220–221 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.89 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.83 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.62–7.60 (m, 2H), 7.50–7.40 (m, 5H), 7.38 (s, 1H), 7.39–7.35 (m, 1H), 7.33–7.29 (m, 3H), 5.01 (bs, 1H), 2.18 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.9, 152.0, 150.2, 138.4, 137.7, 136.0, 132.4, 131.1, 131.0, 129.8, 129.4, 129.1, 128.7, 128.5, 127.9, 124.4, 124.2, 119.7, 119.2, 117.2, 36.9; IR ν_{max} (ATR) 3427, 3062, 1721, 1602, 1446, 1280, 1186, 1067, 765, 690 cm^{-1}; HRMS (EI) calcd for $C_{25}H_{17}NO_2$ [M^+] 363.1259 found 363.1252.



4b. yellow solid; yield 63%; R_f = 0.34 (40% EtOAc/hexanes); mp 184–186 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.90 (d, *J* = 8.8 Hz, 2H), 7.84 (dd, *J* = 8.8, 1.2 Hz, 1H), 7.59 (d, *J* = 8.8 Hz, 2H), 7.48 (td, *J* =8.8, 1.2Hz, 1H), 7.42 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.34–7.29 (m, 1H), 7.36 (s, 1H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 9.2 Hz, 2H), 4.97 (bs, 1H), 3.89 (s, 3H), 3.81 (s, 3H), 2.09 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 161.8, 160.1, 160.2, 150.9, 150.0, 137.1, 132.0, 130.8, 130.1, 130.5, 129.4, 129.3, 128.6, 124.3, 124.0, 120.0, 117.5, 117.1, 114.3, 113.8, 55.3, 55.3, 36.6; IR ν_{max} (ATR) 3422, 3069, 1721, 1604, 1510, 1282, 1251, 1174, 1072, 832 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{21}NO_4$ [M^+] 423.1471 found 423.1468.

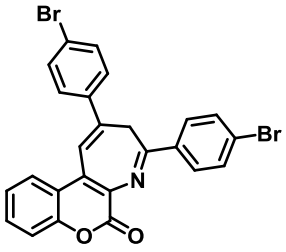


4c. light yellow solid; yield 71%; R_f = 0.34 (20% EtOAc/hexanes); mp 158–160 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.04 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.89 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.76 (d, *J* = 8.8 Hz, 1H), 7.73 (d, *J* = 8.8 Hz, 1H), 7.77–7.72 (m, 1H), 7.68 (d, *J* = 7.2 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 4H), 7.53 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.51 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.48 (s, 1H), 7.46–7.45 (m, 2H), 7.43 (d, *J* = 7.6 Hz, 2H) 7.38–7.32 (m, 2H), 5.11 (bs, 1H), 2.25 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.9, 151.5, 150.2, 143.5, 142.1, 139.8, 137.1, 137.1, 134.8, 132.4, 131.1, 129.8, 129.2, 128.9, 128.8, 128.4, 127.9, 127.6, 127.2, 127.02, 126.98, 124.4, 124.2,



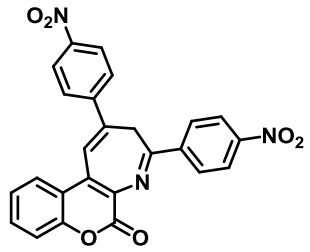
119.7, 119.1, 117.2, 36.6; IR ν_{max} (ATR) 3361, 3027, 1723, 1575, 1365, 1217, 1109, 934, 840 cm^{-1}; HRMS (EI) calcd for $C_{37}H_{25}NO_2$ [M^+] 515.1885 found 515.1882.

4d. white solid; yield 61%; R_f = 0.28 (20% EtOAc/hexanes); mp 256–258 ^oC; ^1H NMR



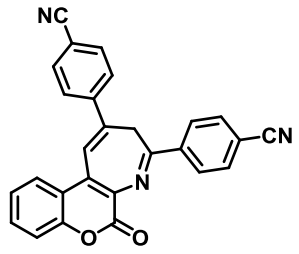
($CDCl_3$, 400 MHz) δ 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.62 (d, *J* = 8.8 Hz, 2H), 7.53 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.44 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.38 (s, 1H), 7.34 (td, *J* = 8.0, 1.2 Hz, 1H) 4.86 (bs, 1H), 2.19 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.6, 150.5, 150.3, 137.1, 136.1, 134.6, 132.4, 132.4, 131.9, 131.1, 130.1, 130.1, 129.4, 126.1, 124.6, 124.2, 124.1, 119.6, 119.4, 117.3, 36.5; IR ν_{max} (ATR) 3429, 3073, 1726, 1557, 1490, 1281, 1113, 1071, 1006, 748 cm^{-1}; HRMS (EI) calcd for $C_{25}H_{15}Br_2NO_2$ [M^+] 518.9470 found 518.9473.

4e. yellow solid; yield 62%; R_f = 0.32 (40% EtOAc/hexanes); mp 232–234 ^oC; ^1H NMR



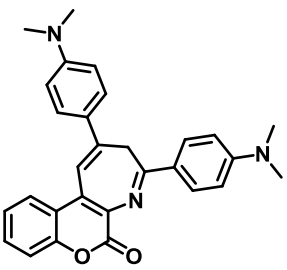
($CDCl_3$, 400 MHz) δ 8.37 (d, *J* = 8.8 Hz, 2H), 8.21 (d, *J* = 8.0 Hz, 2H), 8.04 (d, *J* = 8.8 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 8.8 Hz, 2H), 7.56 (td, *J* = 8.4, 1.2 Hz, 1H), 7.53 (s, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.38 (td, *J* = 8.0, 0.8 Hz, 1H) 4.94 (bs, 1H), 2.38 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.0, 150.5, 149.5, 149.2, 148.3, 143.9, 141.0, 134.6, 133.1, 131.5, 130.9, 129.5, 128.7, 124.9, 124.6, 124.3, 124.0, 122.2, 118.8, 117.6, 36.6; IR ν_{max} (ATR) 3339, 3081, 1730, 1594, 1513, 1342, 1282, 1108, 1074, 848 cm^{-1}; HRMS (EI) calcd for $C_{25}H_{15}N_3O_6$ [M^+] 453.0961 found 453.0965.

4f. brown solid; yield 69%; R_f = 0.42 (20% EtOAc/hexanes); mp 278–280 ^oC ; ^1H NMR



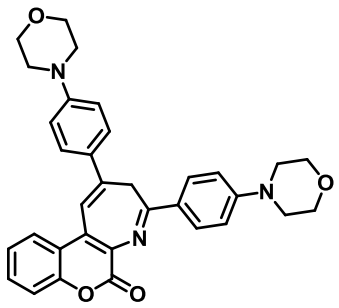
($CDCl_3$, 400 MHz) δ 7.97 (dd, *J* = 6.8, 2.0 Hz, 2H), 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.80 (dd, *J* = 8.8, 1.6 Hz, 2H), 7.72 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.55 (dd, *J* = 6.8, 1.6 Hz, 2H), 7.55 (td, *J* = 7.2, 0.8 Hz, 1H), 7.47 (s, 1H), 7.47 (dd, *J* = 7.2, 0.8 Hz, 1H), 7.37 (td, *J* = 8.0, 1.2 Hz, 1H), 4.89 (bs, 1H), 2.35 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.1, 150.4, 149.8, 142.1, 139.4, 135.0, 133.0, 132.9, 132.5, 131.4, 130.8, 129.0, 128.4, 124.8, 124.3, 121.7, 118.9, 118.0, 117.5, 114.6, 113.4, 36.2; IR ν_{max} (ATR) 3506, 3039, 2226, 1715, 1604, 1500, 1373, 1217, 1108, 1678, 837 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{15}N_3O_2$ [M^+] 413.1164 found 413.1169.

4g. orange solid; yield 28%; R_f = 0.22 (40% EtOAc/hexanes); mp 286–288 ^oC; ^1H NMR



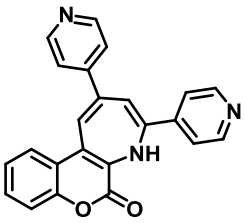
($CDCl_3$, 400 MHz) δ 7.90 (d, *J* = 9.2 Hz, 2H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.8 Hz, 2H), 7.44–7.39 (m, 2H), 7.31–7.30 (m, 2H), 6.75 (s, 2H), 6.58 (d, *J* = 9.2 Hz, 2H), 5.0 (bs, 1H), 3.06 (s, 6H), 2.99 (s, 6H), 2.03 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.8, 151.9, 151.4, 150.9, 150.0, 137.8, 131.7, 130.4, 129.2, 128.9, 126.0, 124.1, 123.9, 123.8, 120.6, 117.0, 115.7, 112.0, 111.2, 40.2, 40.0, 36.0; IR ν_{max} (ATR) 3388, 2923, 1724, 1603, 1524, 1366, 1137, 1074, 945, 895 cm^{-1}; HRMS (EI) calcd for $C_{29}H_{27}N_3O_2$ [M^+] 449.2103 found 449.2101.

4h. light green solid; yield 20%; R_f = 0.45 (30% EtOAc/hexanes); mp 148–150 °C; ^{1}H NMR



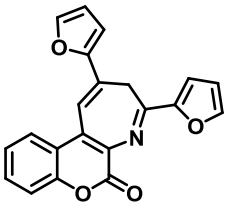
($CDCl_3$, 400 MHz) δ 7.90 (s, 1H), 7.89 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.60 (s, 1H), 7.58 (s, 1H), 7.46 (td, *J* = 8.0, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.32–7.30 (m, 2H), 6.97 (s, 1H), 6.94 (s, 1H), 6.80 (s, 1H), 6.77 (s, 1H), 4.98 (bs, 1H), 3.90 (t, *J* = 4.8 Hz, 4H), 3.84 (t, *J* = 4.8 Hz, 4H), 3.28 (t, *J* = 4.8 Hz, 4H), 3.23 (t, *J* = 4.8 Hz, 4H), 2.04 (bs, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.5, 159.9, 152.7, 151.7, 151.0, 150.1, 137.2, 132.0, 130.7, 130.3, 129.3, 129.2, 126.8, 124.2, 124.0, 120.3, 117.2, 117.0, 114.9, 113.9, 66.7, 66.6, 48.3, 47.8, 36.1; IR ν_{max} (ATR) 3073, 1726, 1579, 1557, 1491, 1281, 1111, 1071, 1006, 749 cm^{-1}; HRMS (EI) calcd for $C_{33}H_{31}N_3O_4$ [M^+] 533.2315 found 533.2310.

4i. brown solid; yield 43%; R_f = 0.15 (20% EtOAc/hexanes); mp 188–190 °C; ^{1}H NMR



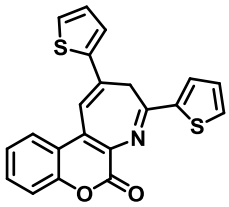
($CDCl_3$, 400 MHz) δ 12.14 (s, 1H), 8.86 (d, *J* = 6.0 Hz, 2H), 8.82 (d, *J* = 5.2 Hz, 2H), 8.55 (dd, *J* = 8.0, 1.6 Hz, 1H), 8.24 (d, *J* = 1.6 Hz, 1H), 8.22 (d, *J* = 1.6 Hz, 1H), 8.03 (dd, *J* = 4.4, 1.6 Hz, 2H), 7.69 (dd, *J* = 4.8, 1.6 Hz, 2H) 7.60 (td, *J* = 7.2, 1.2 Hz, 1H), 7.14 (d, *J* = 8.8 Hz, 1H), 6.98 (td, *J* = 8.4, 1.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 196.8, 164.1, 156.6, 154.4, 150.9, 150.7, 148.7, 144.9, 144.6, 137.1, 134.3, 122.3, 121.5, 121.1, 120.4, 118.9, 118.5; IR ν_{max} (ATR) 3049, 1628, 1587, 1482, 1352, 1238, 1151, 970, 807 cm^{-1}; HRMS (EI) calcd for $C_{23}H_{15}N_3O_2$ [M^+] 365.1164 found 365.1160.

4j. grey solid; yield 49%; R_f = 0.34 (20% EtOAc/hexanes); mp 243–245 °C; ^{1}H NMR



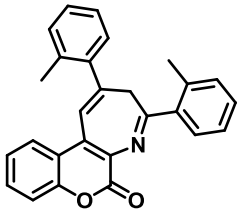
($CDCl_3$, 400 MHz) δ 7.89 (dd, *J* = 6.4, 1.6 Hz, 1H), 7.61 (s, 1H), 7.60 (d, *J* = 0.8 Hz, 1H), 7.52 (dd, *J* = 6.4, 1.6 Hz, 1H), 7.49 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.35 (td, *J* = 8.4, 1.6 Hz, 1H), 7.21 (d, *J* = 3.2 Hz, 1H), 6.93 (d, *J* = 3.2 Hz, 1H), 6.55 (ddd, *J* = 5.2, 3.5, 2.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.8, 152.2, 151.3, 150.2, 146.2, 144.6, 143.2, 132.4, 131.4, 129.9, 125.8, 124.4, 124.3, 119.7, 117.2, 115.7, 115.0, 112.7, 112.4, 111.4, 33.8; IR ν_{max} (ATR) 3423, 3132, 1718, 1596, 1474, 1786, 1172, 1109, 1023, 880 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{13}NO_4$ [M^+] 343.0845 found 343.0841.

4k. yellow solid; yield 54%; R_f = 0.40 (20% EtOAc/hexanes); mp 232–234 °C; ^{1}H NMR



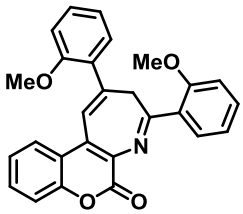
($CDCl_3$, 400 MHz) δ 7.78 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.47 (td, *J* = 8.4, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.29 (td, *J* = 8.0, 1.2 Hz, 1H), 7.26–7.23 (m, 2H), 7.22–7.18 (m, 3H), 7.15 (s, 1H), 7.16–7.12 (m, 1H), 7.07–7.04 (m, 2H), 4.46 (bs, 1H), 2.52 (s, 3H), 2.44 (bs, 1H), 2.35 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.6, 150.2, 146.7, 142.7, 142.6, 132.7, 132.1, 131.1, 130.3, 129.9, 129.8, 128.5, 128.2, 127.8, 127.3, 124.4, 124.2, 119.6, 117.2, 116.8, 37.7 ; IR ν_{max} (ATR) 3099, 1706, 1567, 1498, 1425, 1288, 1181, 1109, 1077, 852, 747 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{13}NO_2S_2$ [M^+] 375.0388 found 375.0385.

4l. off-white solid; yield 37%; R_f = 0.39 (20% EtOAc/hexanes); mp 216–218 °C; ^{1}H NMR



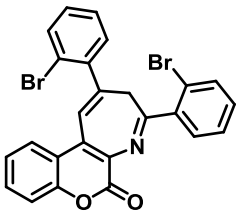
($CDCl_3$, 400 MHz) δ 7.78 (dd, *J* = 0.8, 8.0 Hz, 1H), 7.47 (td, *J* = 8.4, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.30 (td, *J* = 8.0, 1.2 Hz, 1H), 7.27–7.26 (m, 2H), 7.24–7.18 (m, 3H), 7.16–7.12 (m, 1H), 7.16 (s, 1H), 7.07–7.03 (m, 2H), 4.46 (br s, 1H), 2.52 (s, 3H), 2.44 (br s, 1H), 2.35 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 159.7, 154.4, 150.3, 140.3, 139.2, 138.3, 137.3, 135.9, 132.1, 131.3, 130.8, 130.1, 129.9, 129.7, 129.2, 128.9, 128.7, 126.2, 125.5, 124.4, 124.2, 121.4, 119.6, 117.2, 43.7, 21.6, 20.5; IR ν_{max} (ATR) 3061, 1723, 1378, 1174, 1069, 937, 766 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{21}NO_2$ [M^+] 391.1572 found 391.1577.

4m. yellow solid; yield 33%; R_f = 0.26 (30% EtOAc/hexanes); mp 239–241 °C; ^{1}H NMR



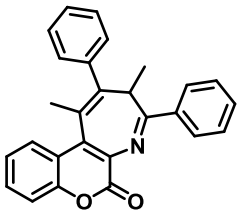
($CDCl_3$, 400 MHz) δ 7.83 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.75 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.48 (td, *J* = 8.4, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.36 (dd, *J* = 1.6, 8.8 Hz, 1H), 7.33 (dd, *J* = 1.6, 6.0 Hz, 1H), 7.31 (dd, *J* = 5.6, 2.0 Hz, 1H), 7.23 (s, 1H), 7.22 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.98–6.93 (m, 2H), 6.95 (s, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 4.88 (br s, 1H), 3.7 (s, 3H), 3.1 (s, 3H), 2.39 (br s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.1, 158.0, 157.6, 157.5, 150.4, 140.3, 132.7, 132.0, 131.5, 131.1, 130.9, 130.6, 129.9, 129.7, 127.9, 124.3, 124.2, 121.4, 120.6, 120.5, 119.8, 117.1, 111.0, 110.8, 55.3, 54.6, 41.3; IR ν_{max} (ATR) 3064, 2941, 1721, 1597, 1487, 1373, 1289, 1170, 1073, 749 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{21}NO_4$ [M^+] 423.1471 found 423.1474.

4n. off-white; yield 30%; R_f = 0.30 (40% EtOAc/hexanes); mp 180–182 °C; ^{1}H NMR ($CDCl_3$,



400 MHz) δ 7.84 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.64 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.58 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.52 (dd, *J* = 7.2, 1.6 Hz, 1H), 7.35 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.26 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.25 (s, 1H), 7.24–7.21 (m, 3H), 7.11 (dd, *J* = 7.2, 2.4 Hz, 1H), 4.66 (br s, 1H), 2.62 (br s, 1H); IR ν_{max} (ATR) 3064, 2941, 1721, 1597, 1487, 1373, 1289, 1170, 1073, 749 cm^{-1}; HRMS (EI) calcd for $C_{25}H_{15}Br_2NO_2$ [M^+] 518.9470 found 518.9473.

4o.off-white solid; yield 57%; R_f = 0.42 (30% EtOAc/hexanes); mp 155–157 °C; ^{1}H NMR



($CDCl_3$, 400 MHz) δ 7.95–7.93 (m, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.54–7.49 (m, 4H), 7.43 (d, *J* = 1.2 Hz, 1H), 7.41 (s, 1H), 7.39 (s, 1H), 7.38–7.36 (m, 1H), 7.28–7.26 (m, 1H), 7.25 (d, *J* = 6.8 Hz, 1H), 7.21 (dd, *J* = 8.0, 1.2 Hz, 1H), 4.29 (q, *J* = 7.2, 14.8 Hz, 1H), 1.76 (s, 3H), 1.23 (d, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.4, 159.4, 152.7, 150.6, 142.3, 139.7, 137.7, 130.7, 130.4, 130.4, 128.8, 128.7, 128.5, 127.8, 127.6, 125.6, 124.5, 124.0, 119.1, 117.3, 37.0, 19.8, 11.7 IR ν_{max} (ATR) 2926, 1721, 1605, 1448, 1302, 1180, 1061, 874, 753 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{21}NO_2$ [M^+] 391.1572 found 391.1576.

References

1. N. C. Ganguly, M. Datta, P. De and R. Chakravarty, *Synth. Commun.* 2003, **33**, 647.

2. I. Brady, D. Leane, H. P. Hughes, R. J. Forster and T. E. Keyes, *Dalton Trans.* 2004, **21**, 334.

DYYGR046

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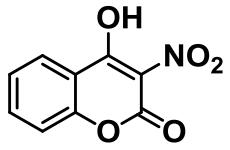

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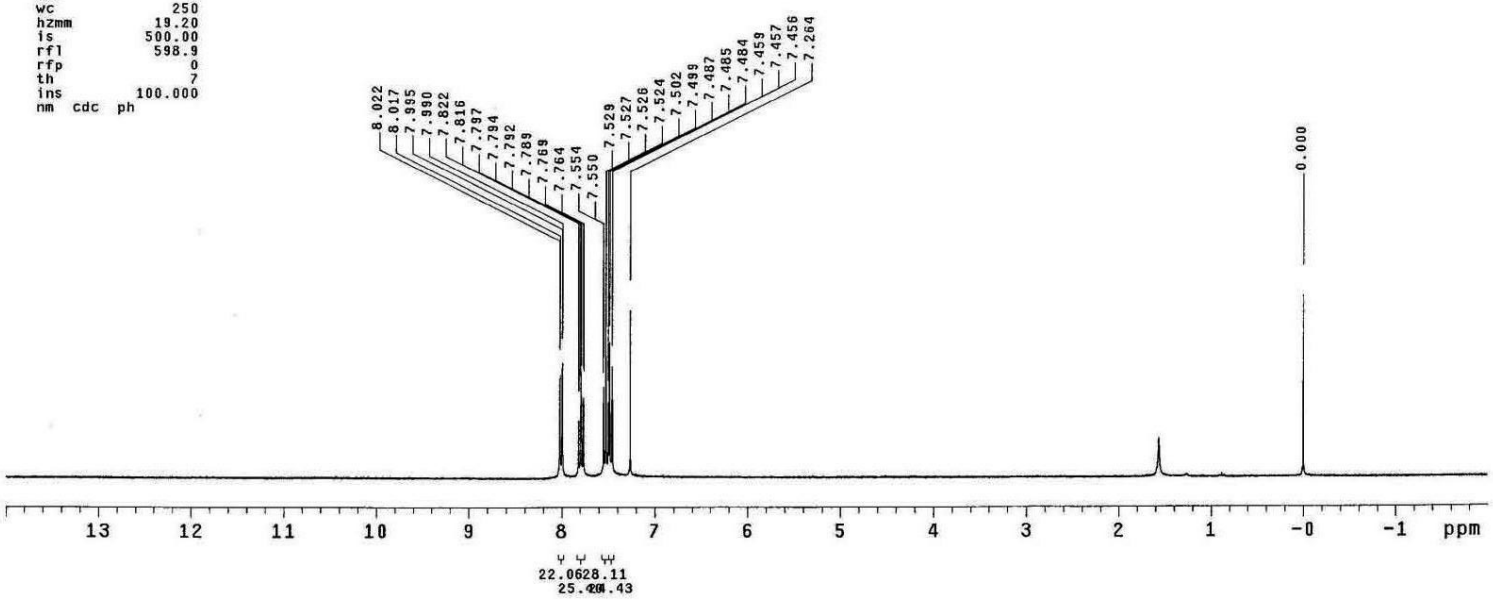

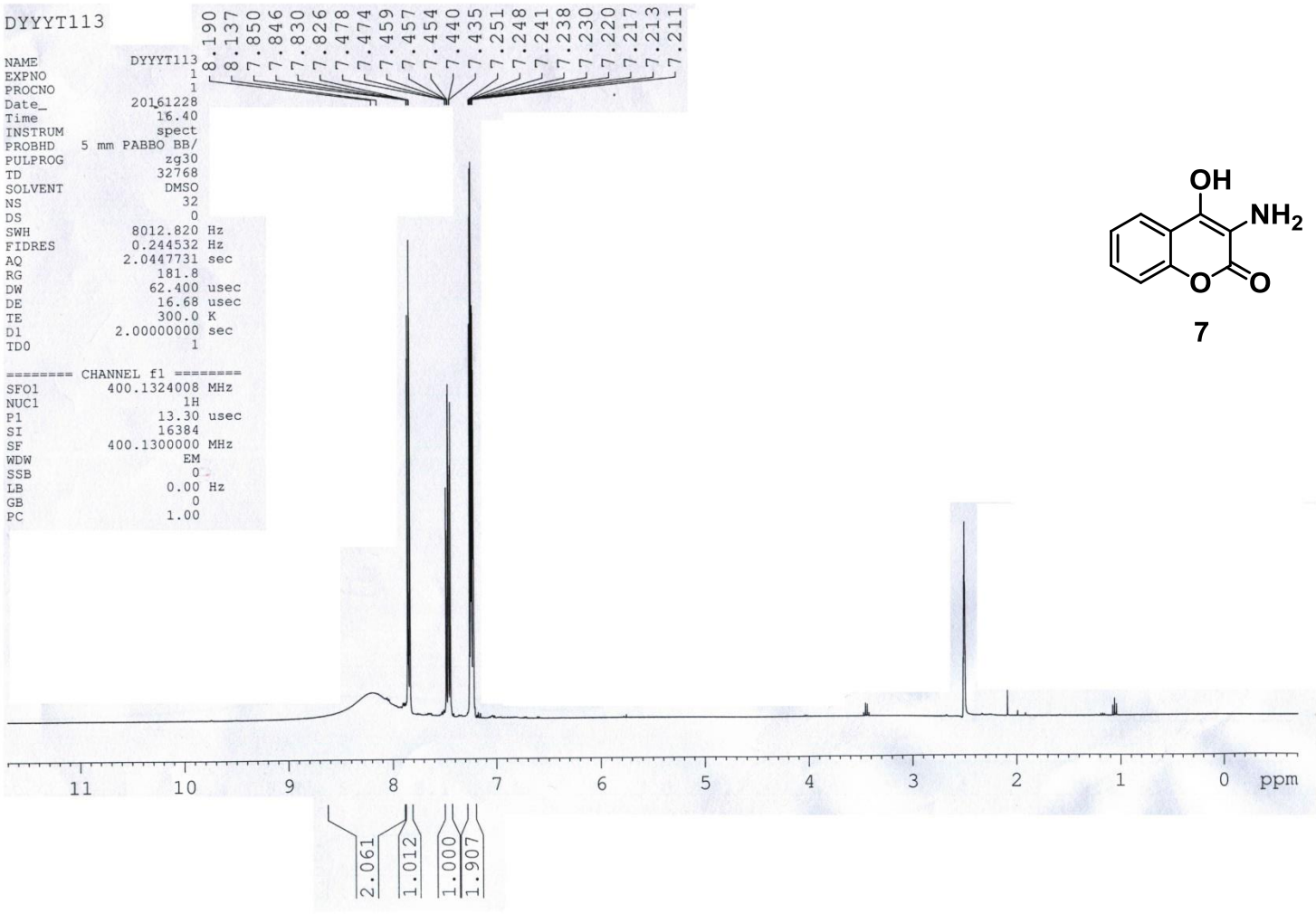
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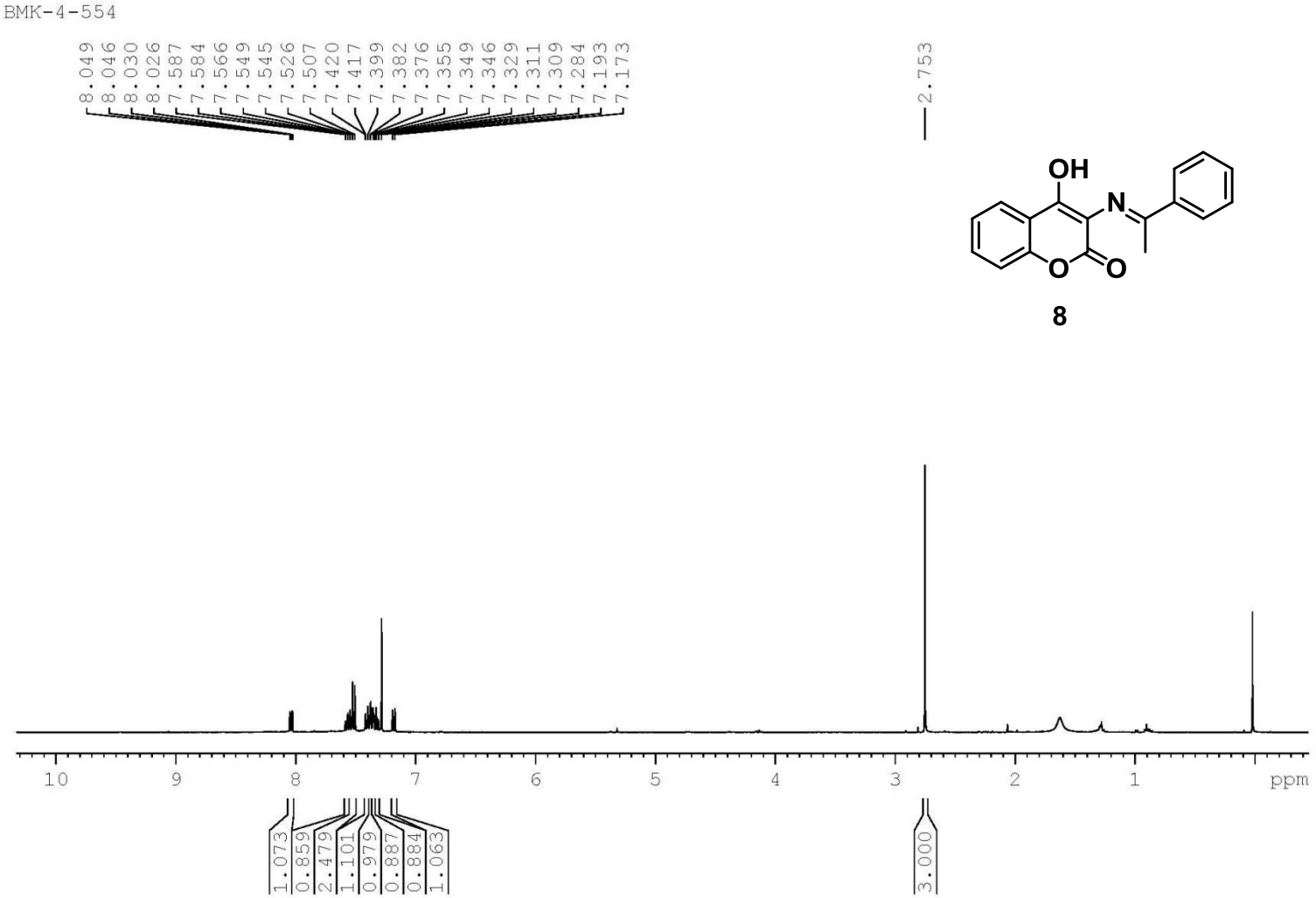
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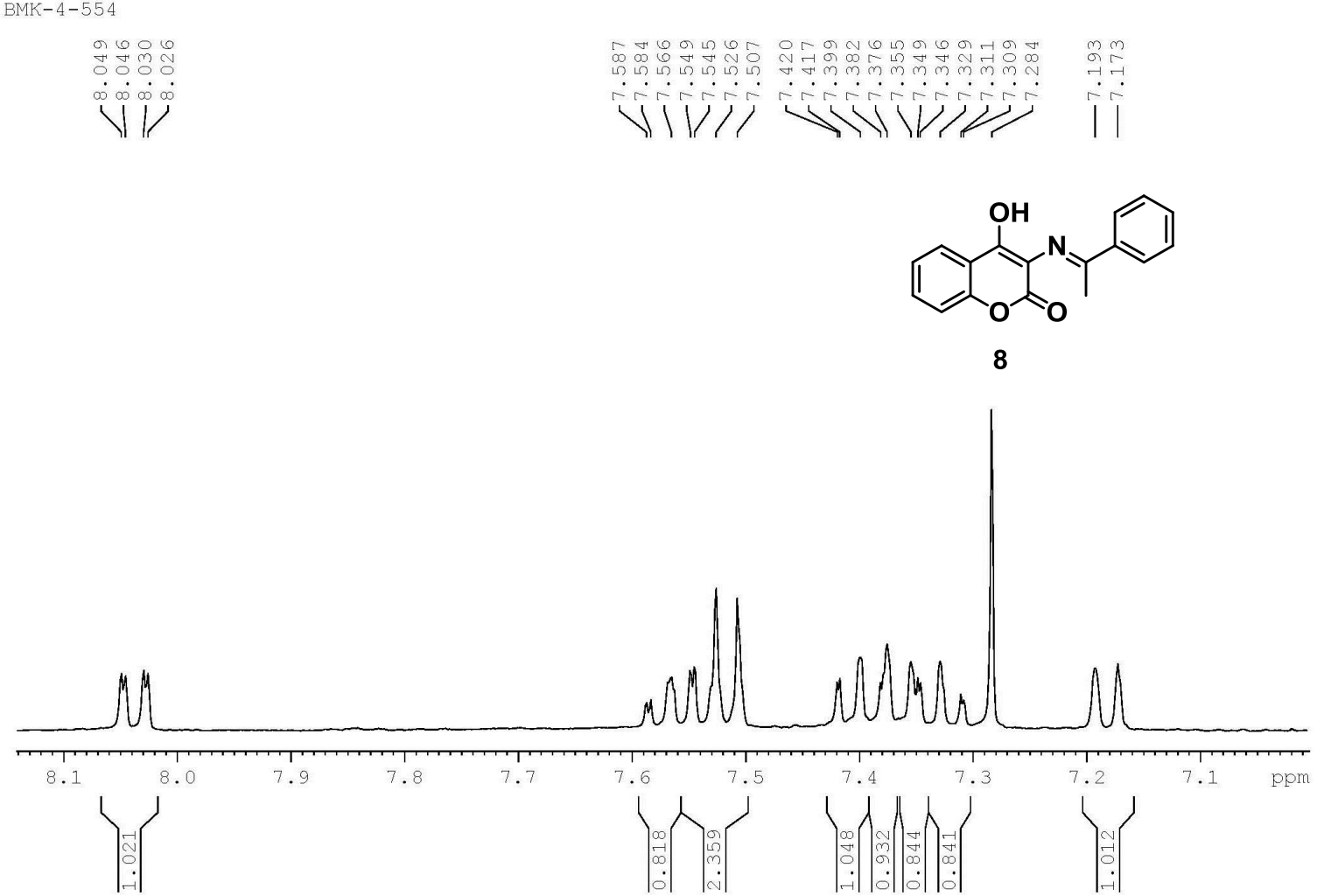
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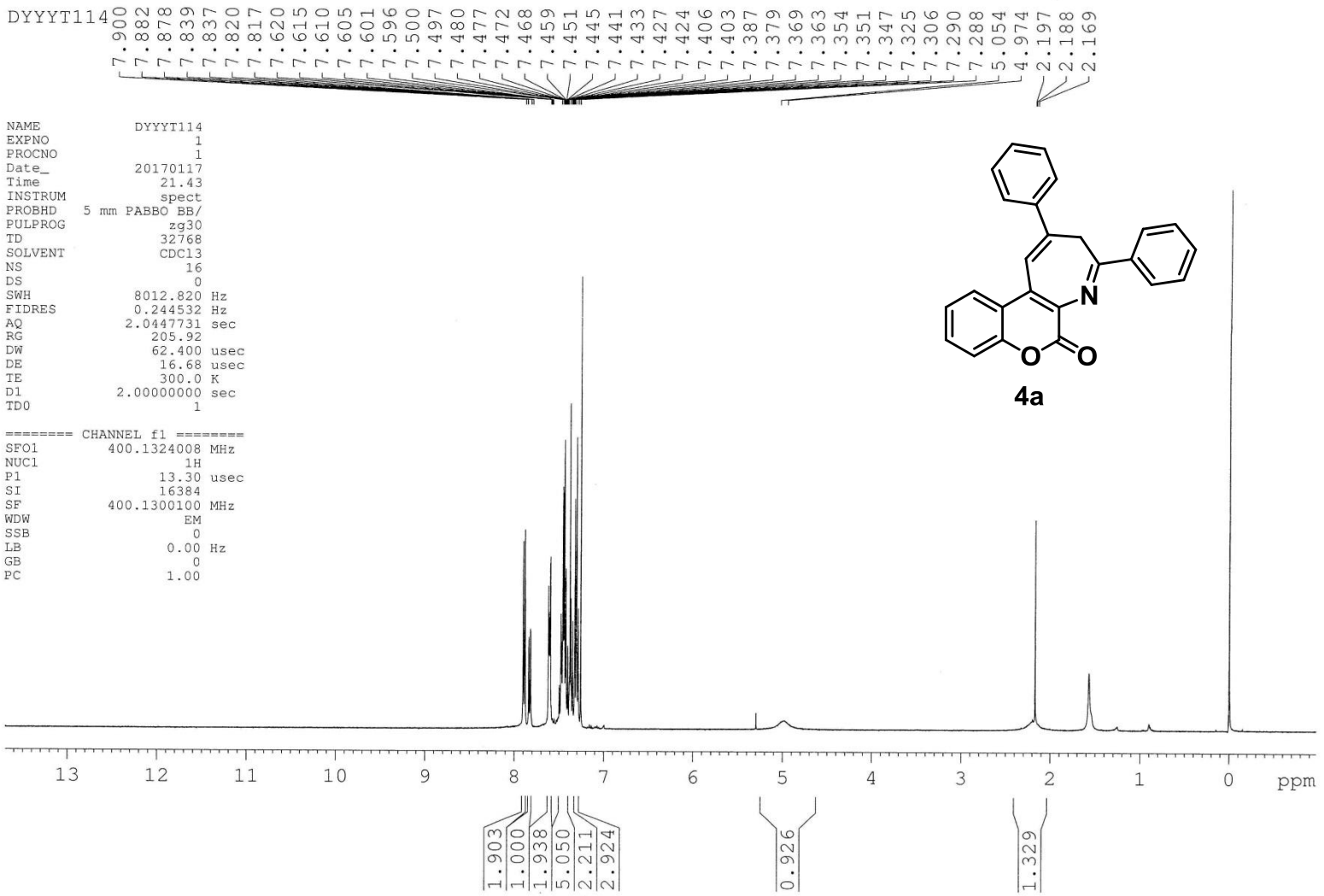
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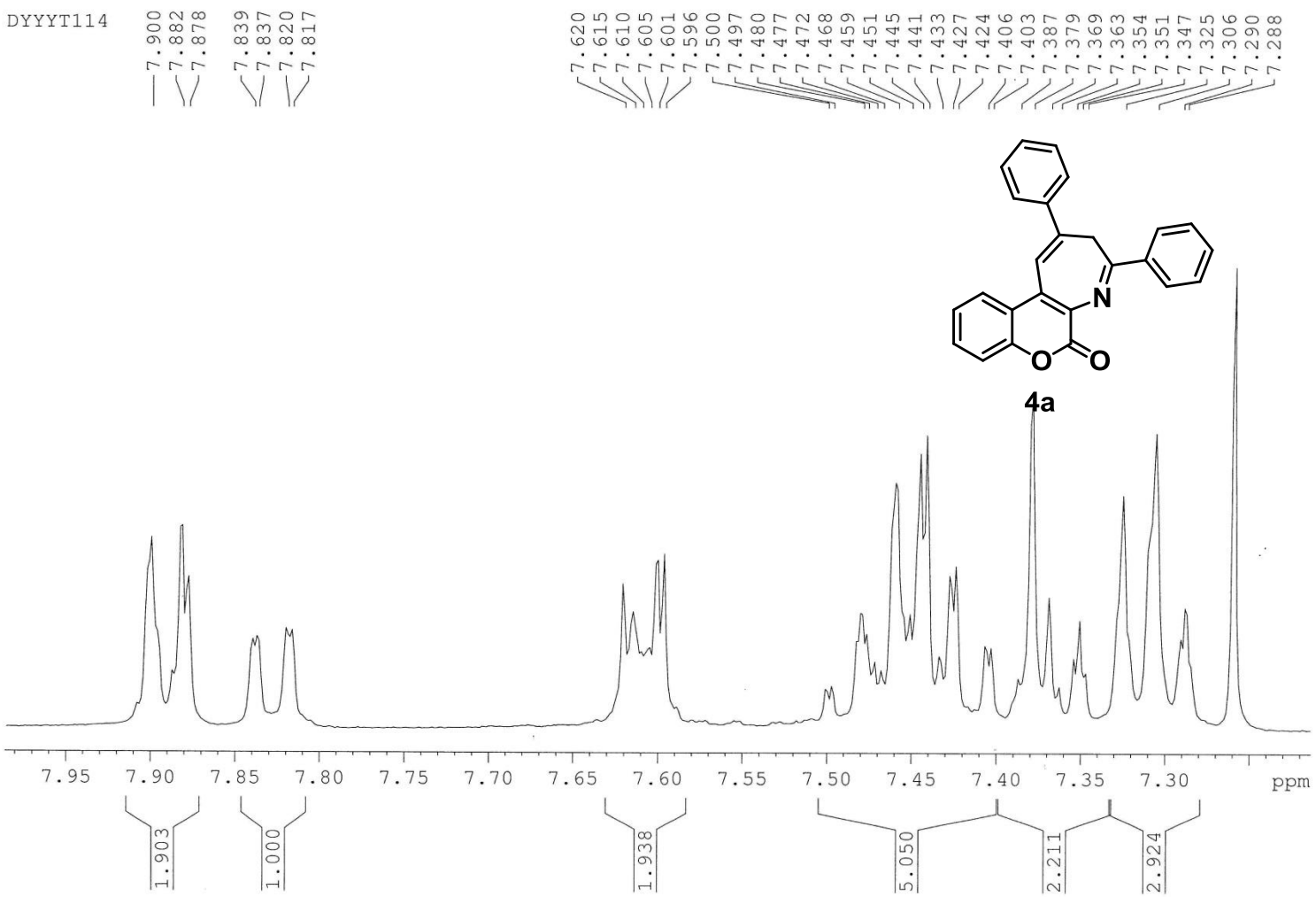
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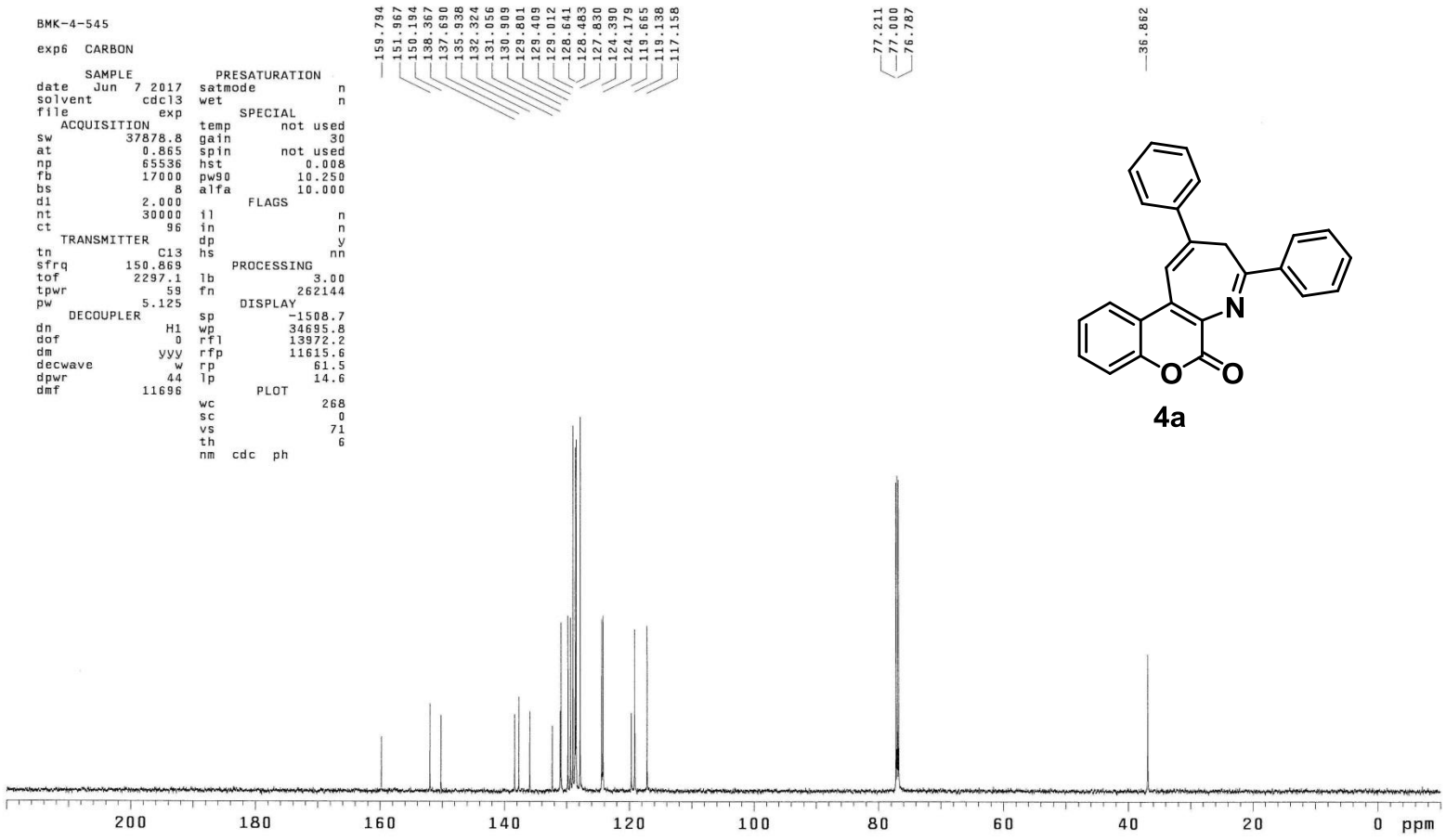
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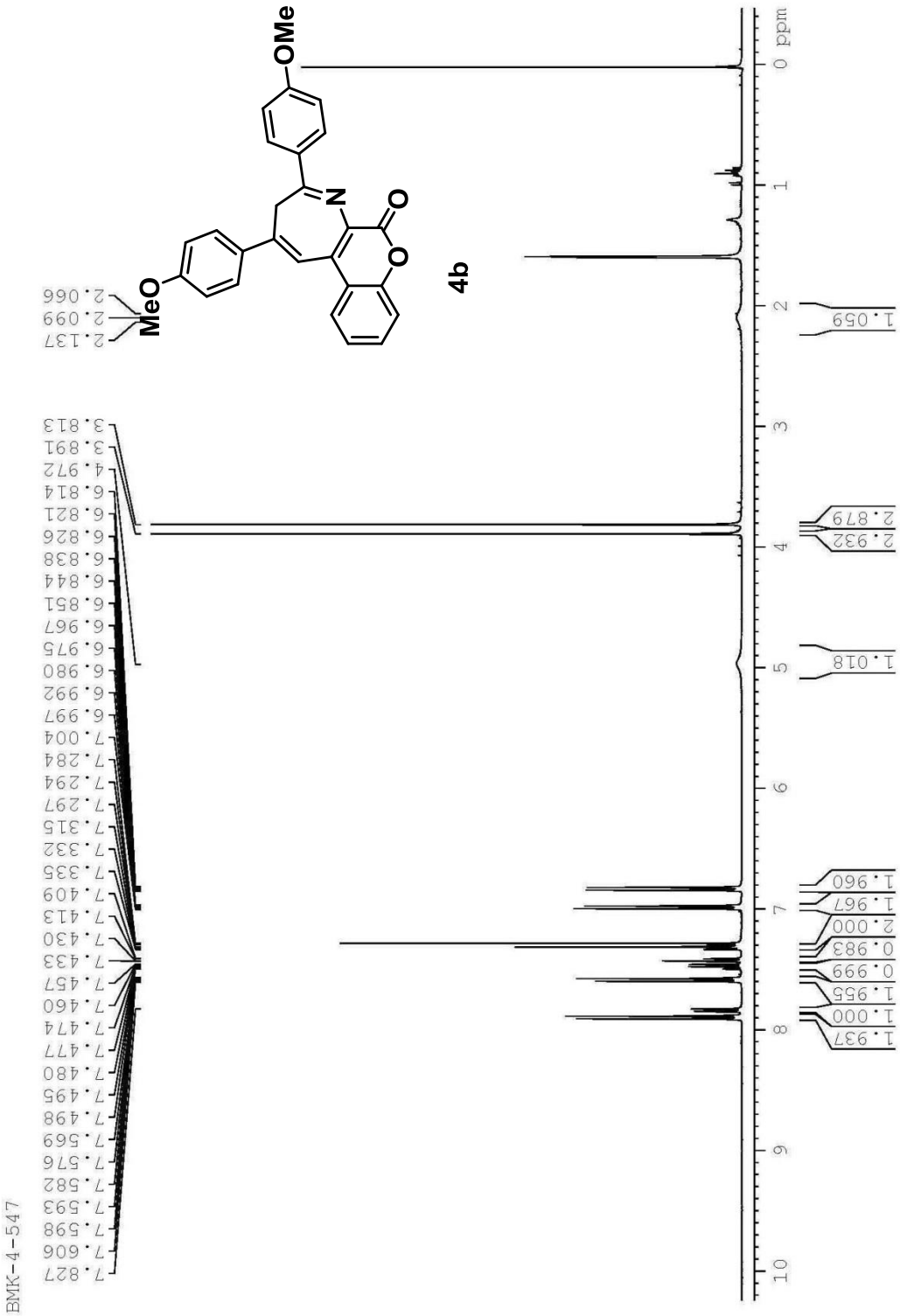
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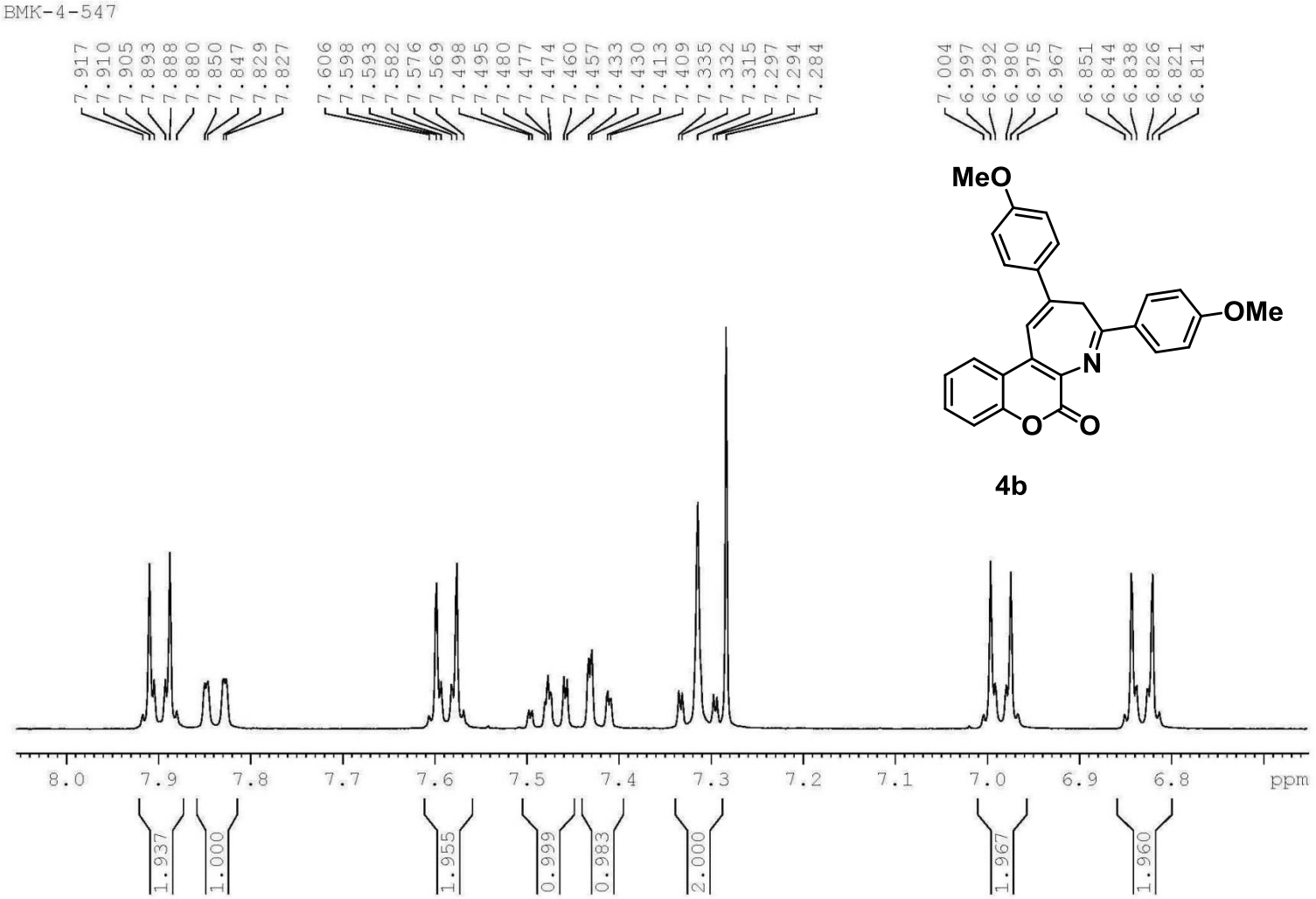
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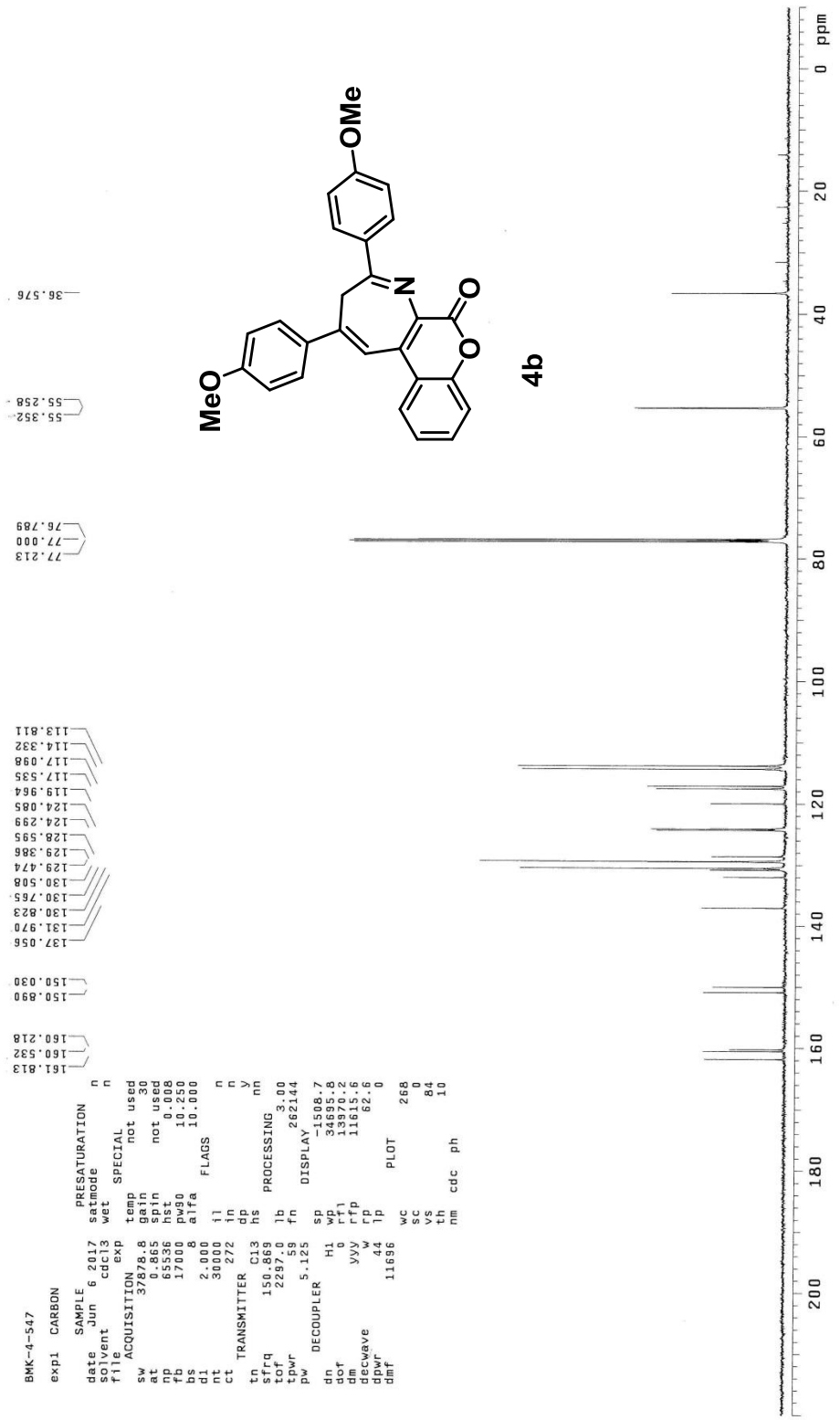
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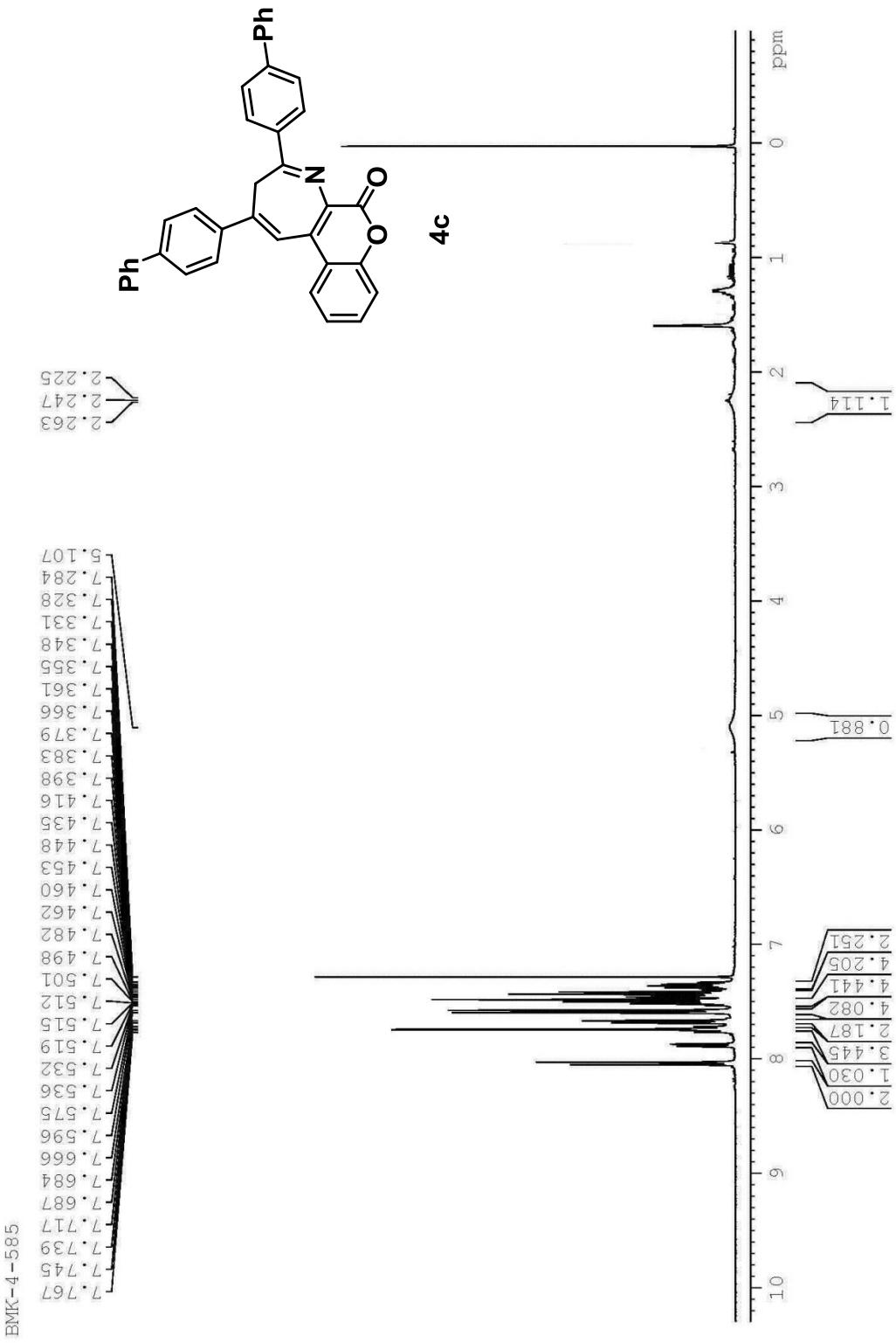
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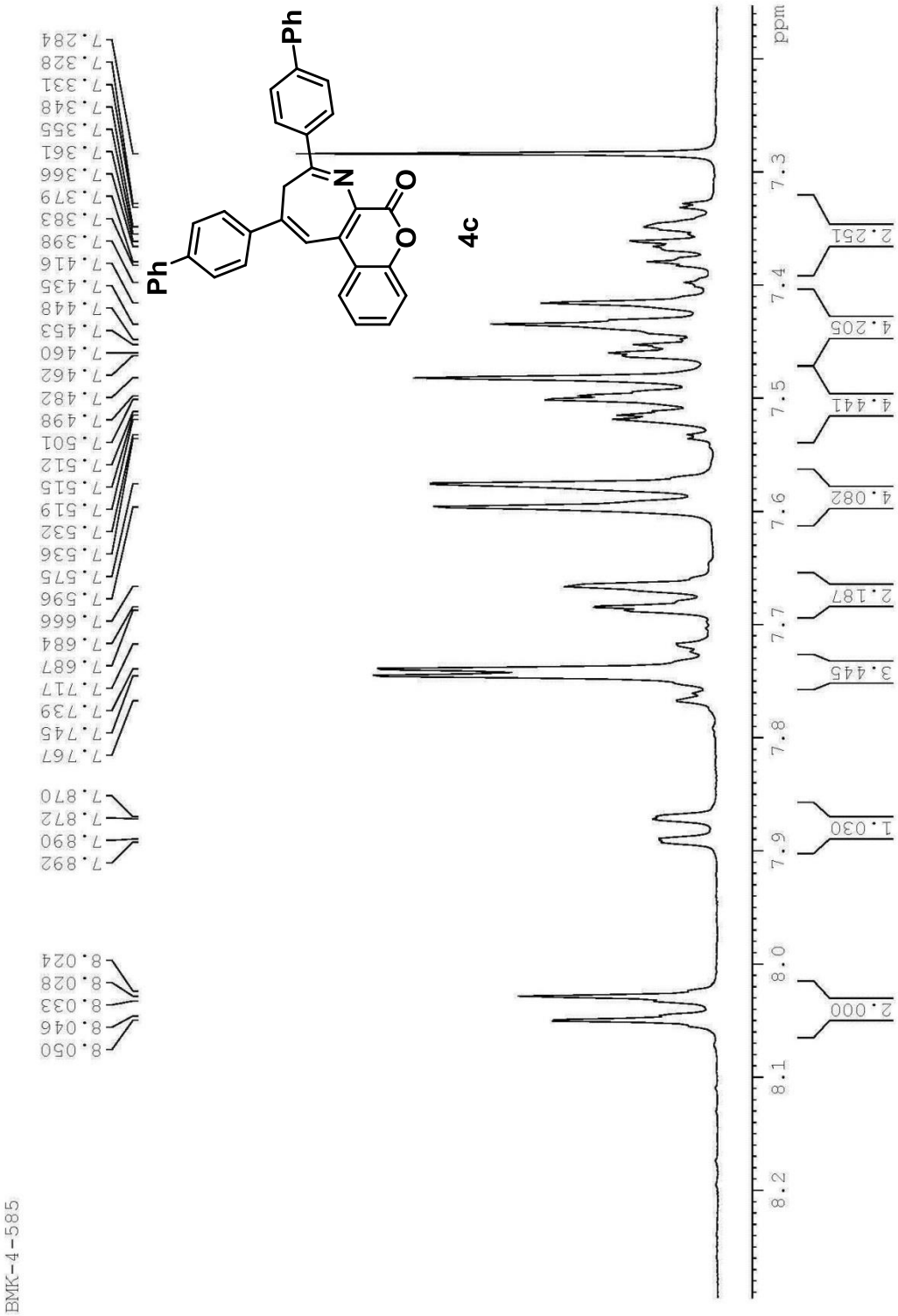
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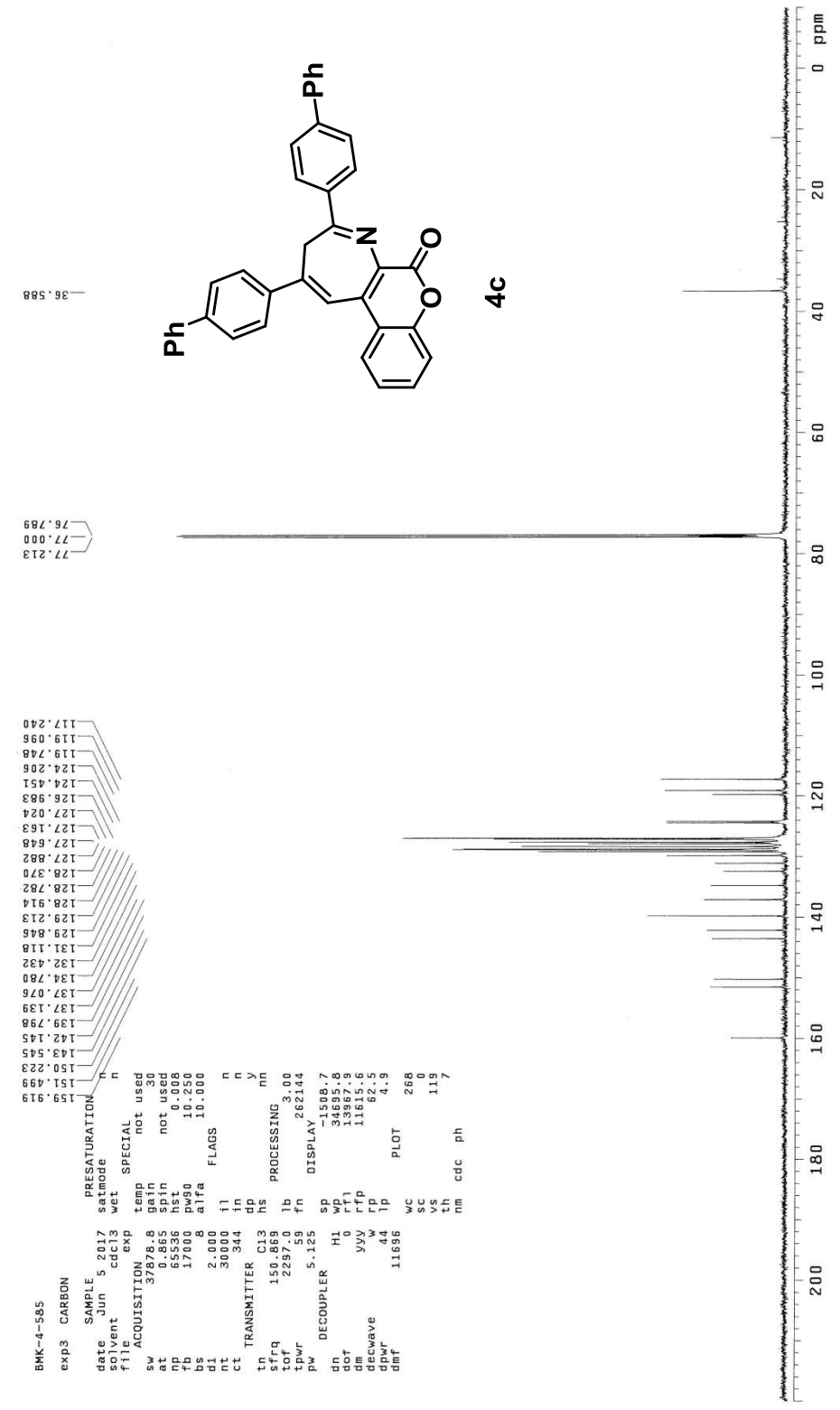
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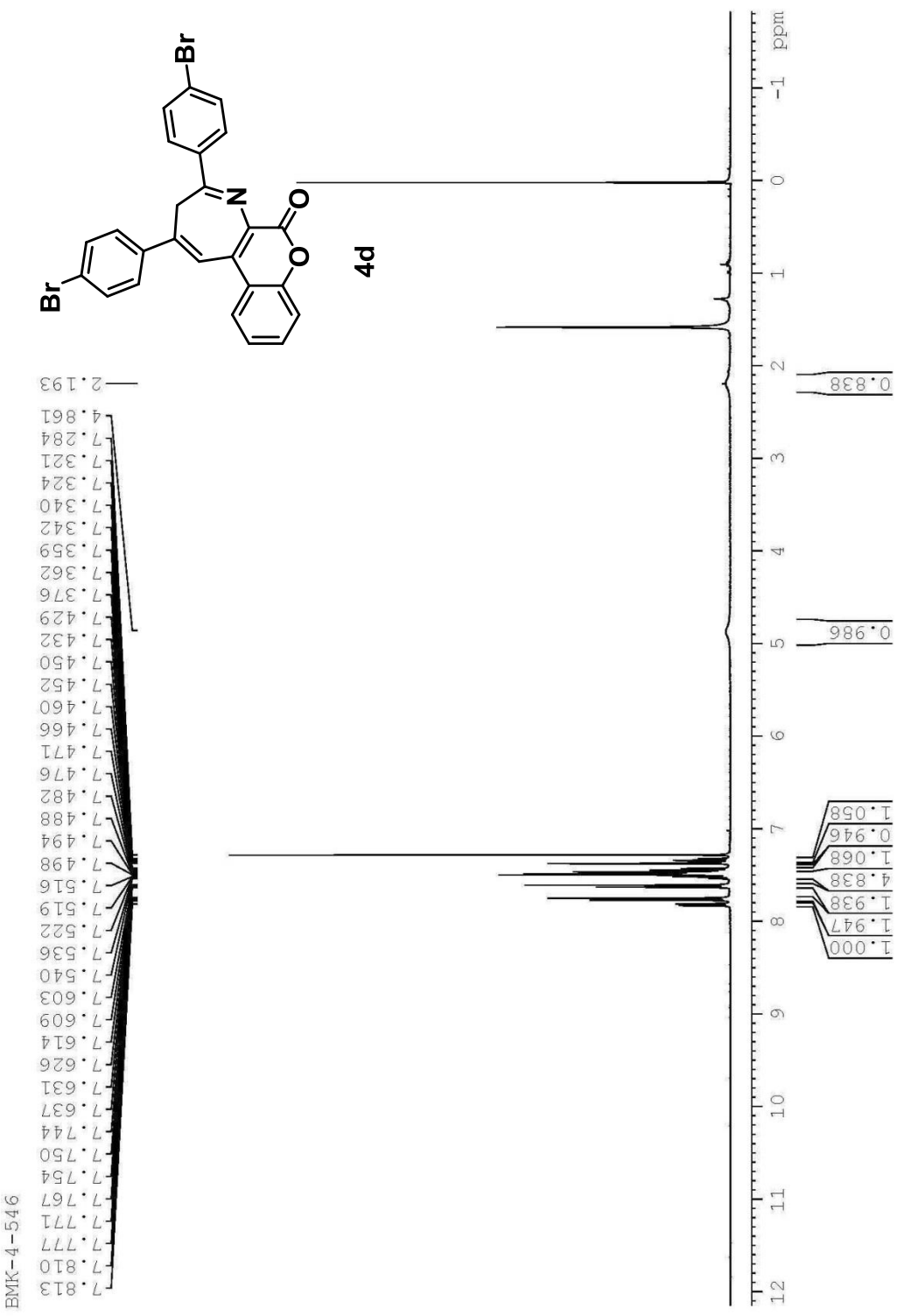
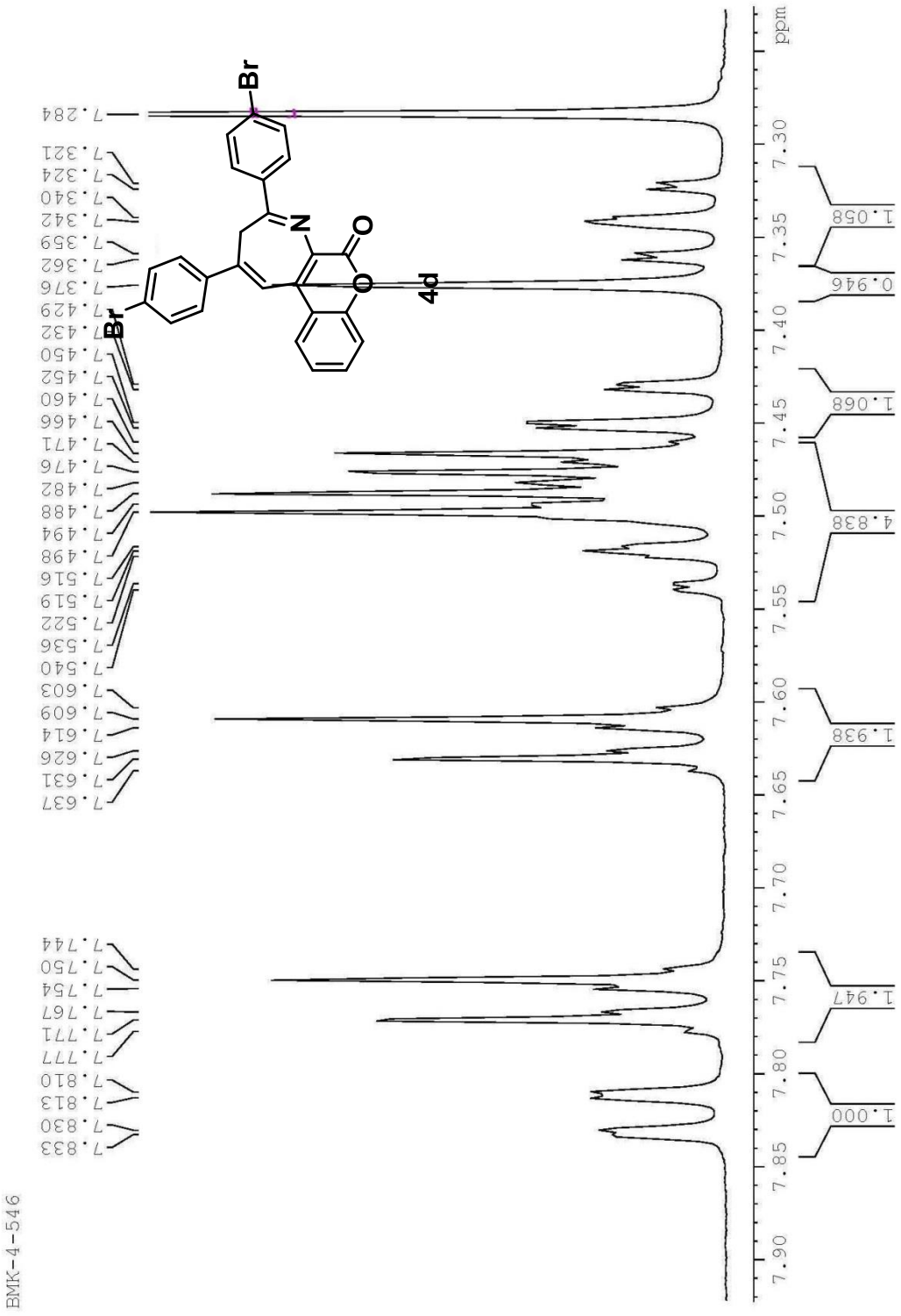
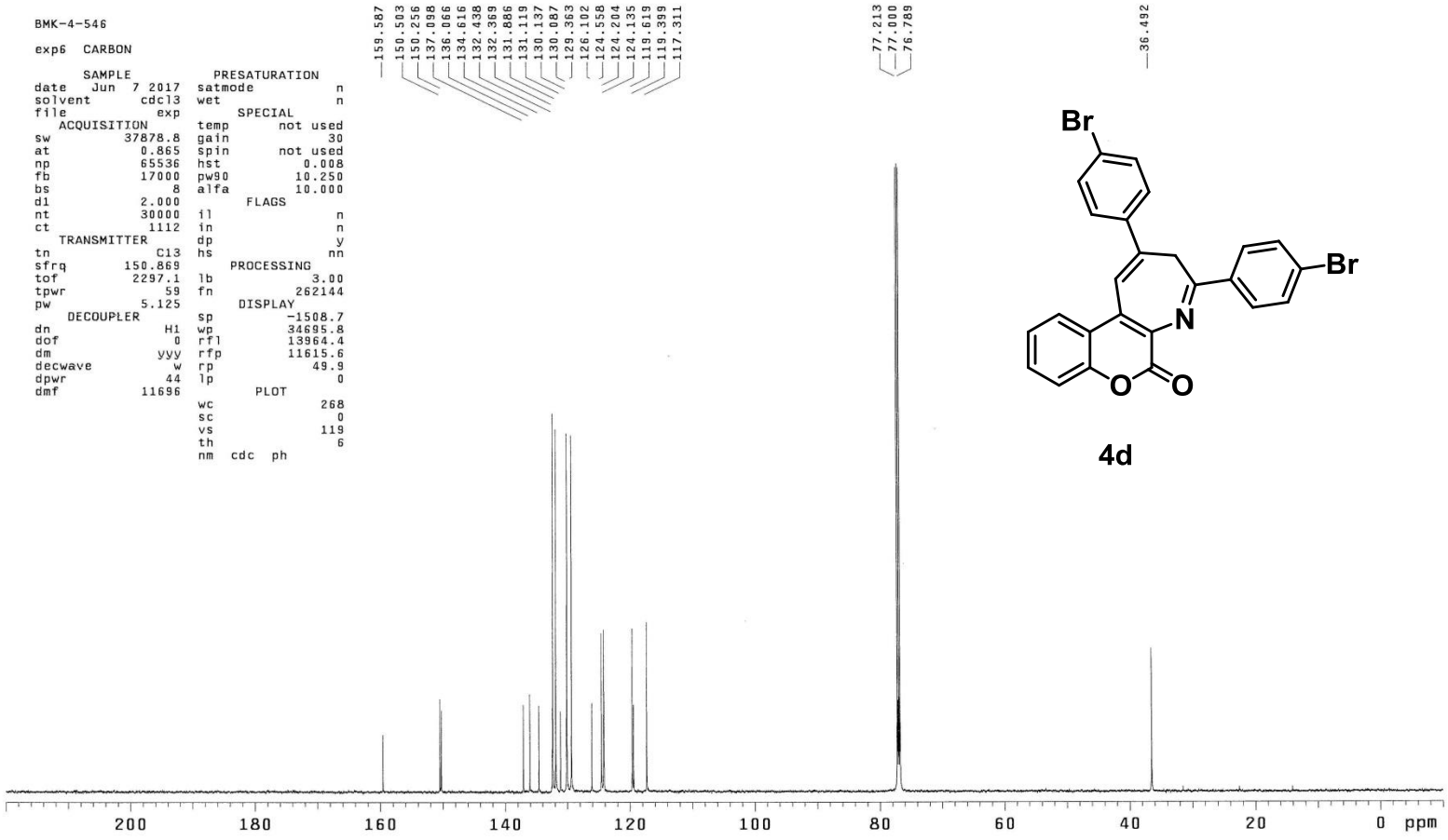
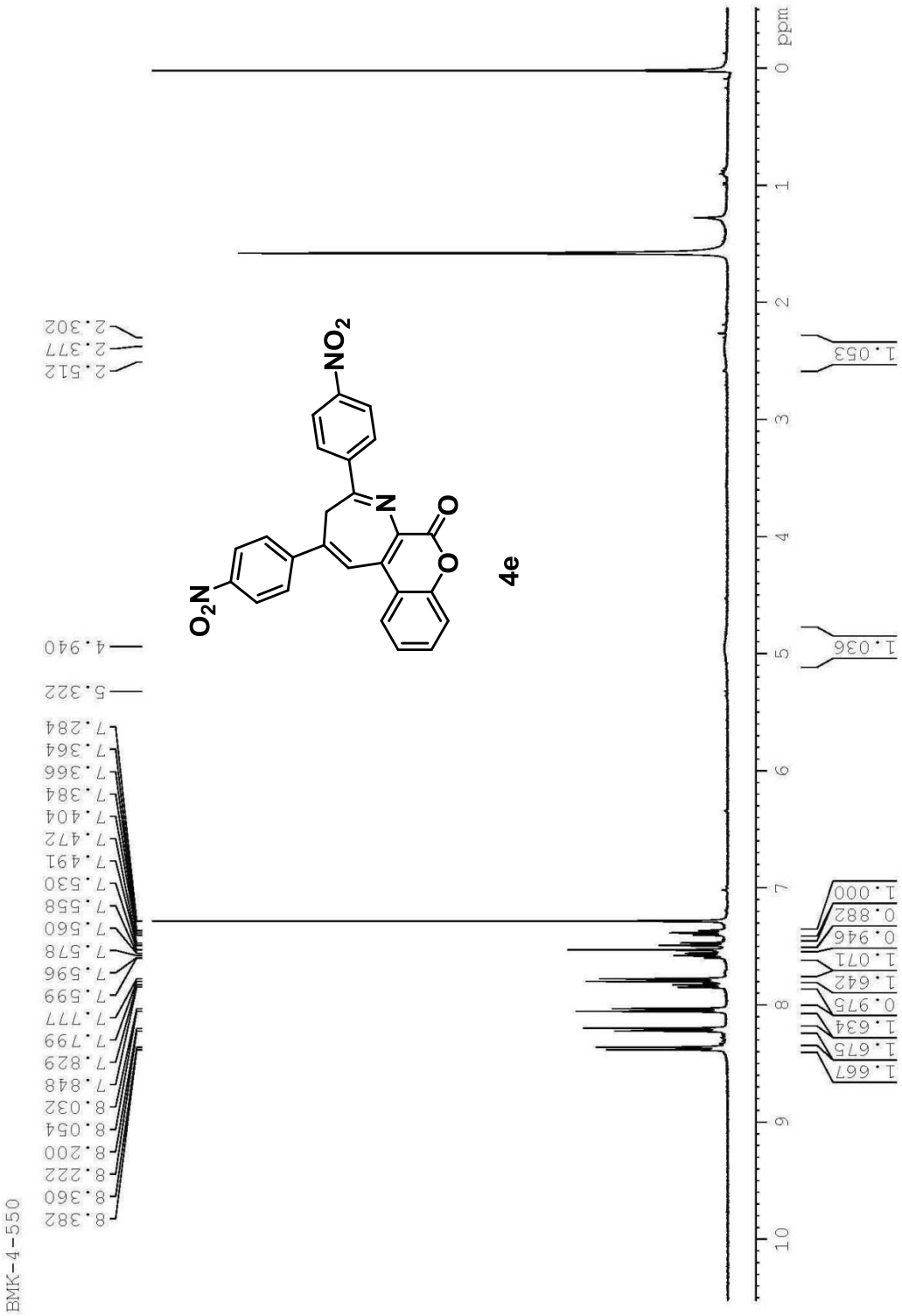
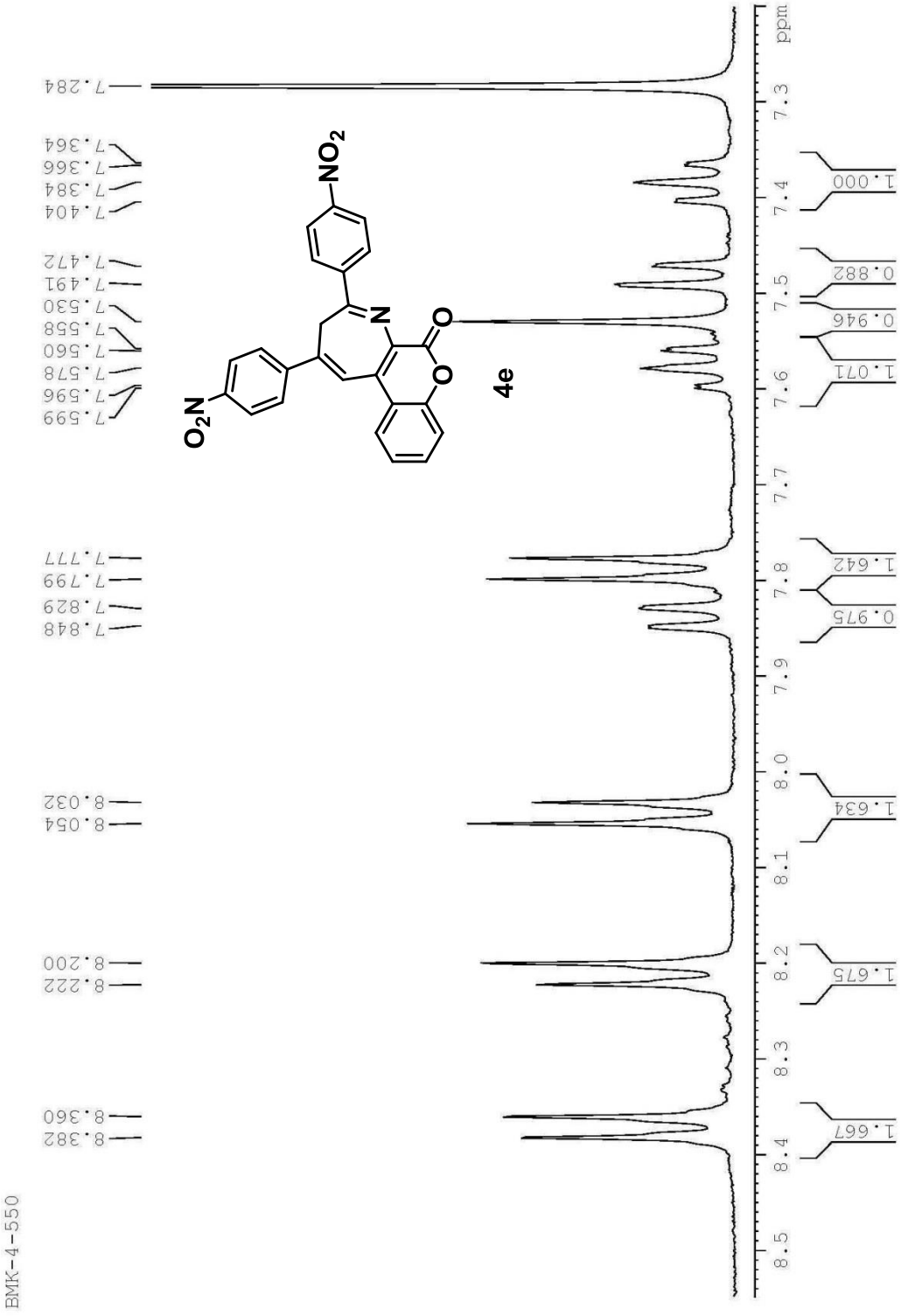
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13967.9
rfp 11615.6
rp 62.5
lp 4.9
PLOT
wc 268
sc 0
vs 119
th 7
nm cdc ph
200
180
160
140
120
100
80
60
40
20
0 ppm

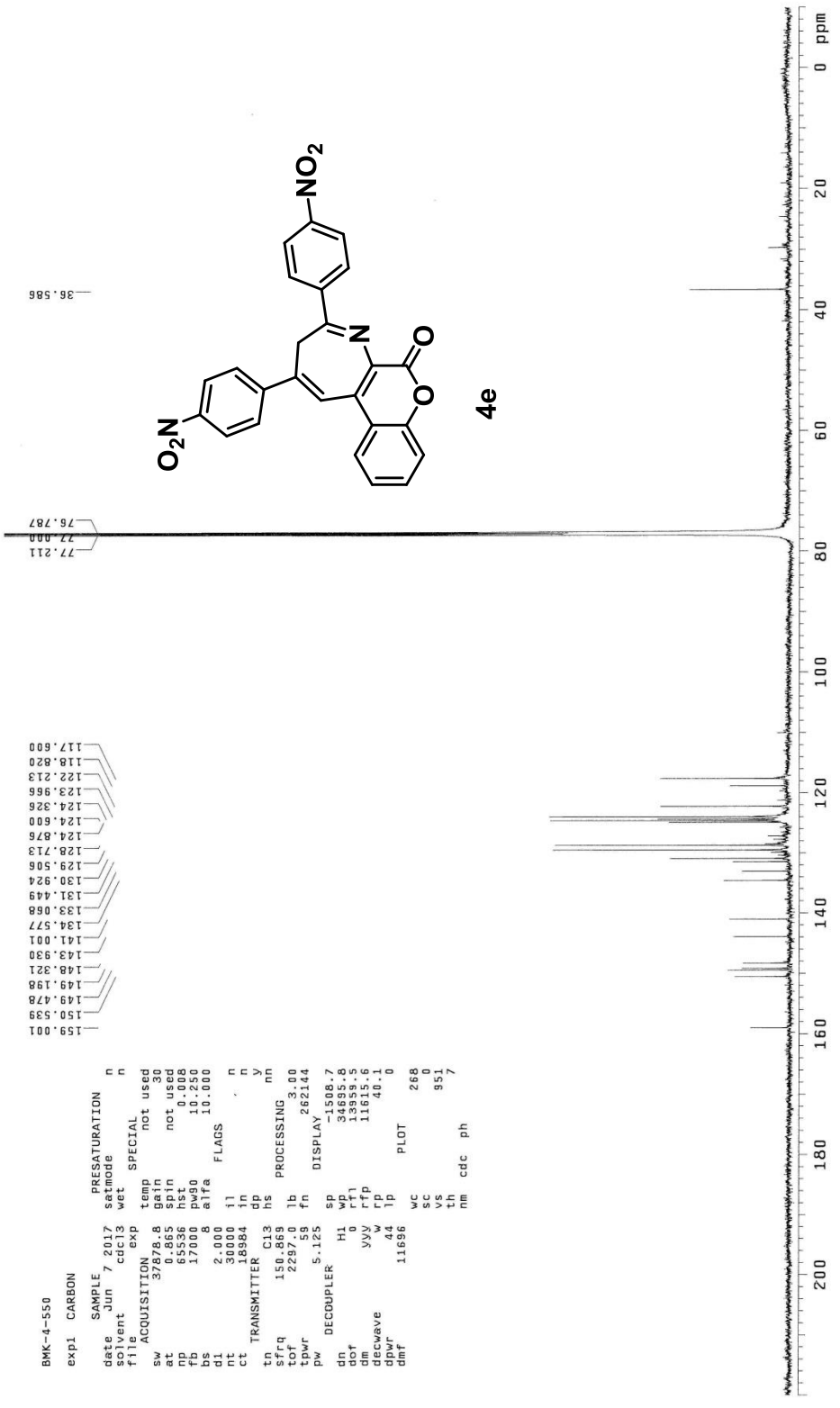

Br
N
O
O
Br
4d
BMK-4-546
ppm

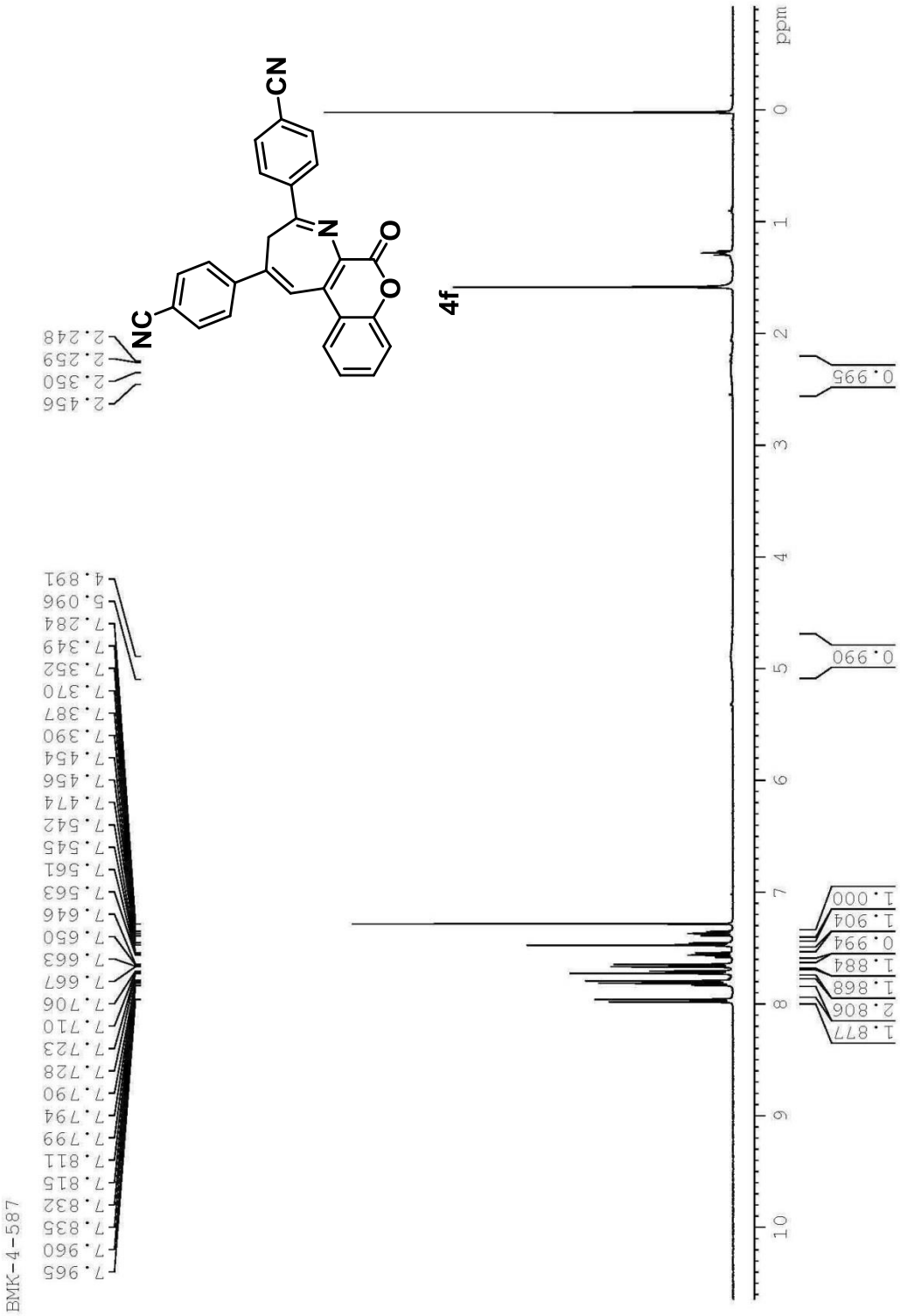

BMK-4-546
7.284
7.321
7.324
7.340
7.342
7.359
7.362
7.376
7.429
7.432
7.450
7.452
7.460
7.466
7.471
7.476
7.482
7.488
7.494
7.498
7.516
7.519
7.522
7.536
7.540
7.603
7.609
7.614
7.626
7.631
7.637
7.744
7.750
7.754
7.767
7.771
7.777
7.810
7.813
7.830
7.833
Br
N
O
O
Br
4d
ppm
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
1.000
1.947
1.938
4.838
1.068
0.946
1.058

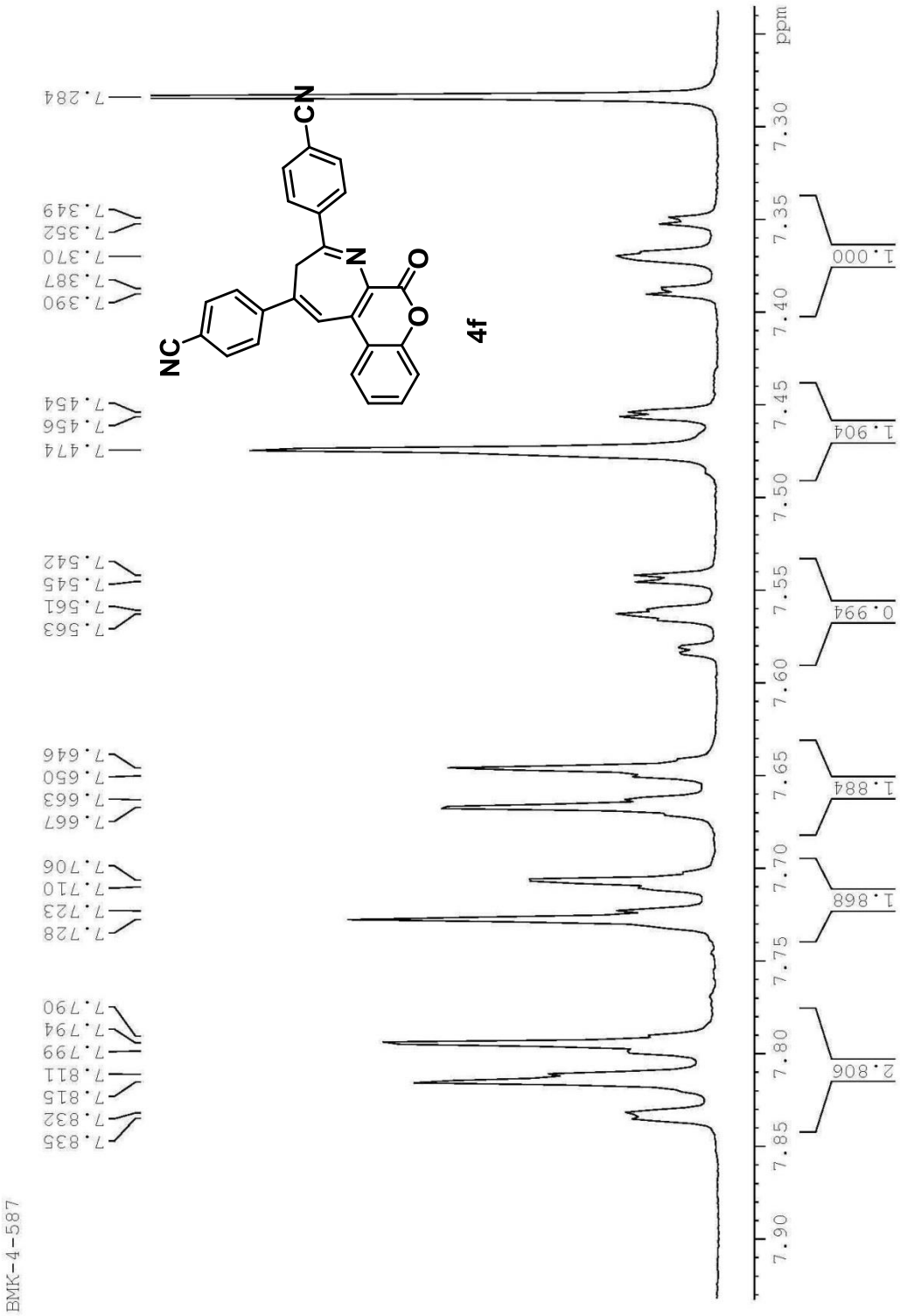

BMK-4-546
exp6 CARBON
SAMPLE
date Jun 7 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 1112
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.1
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13964.4
rfp 11615.6
rp 49.9
lp 0
PLOT
wc 268
sc 0
vs 119
th 6
nm cdc ph
159.587
150.503
150.256
137.098
136.066
134.616
132.438
132.369
131.886
131.119
130.137
130.087
129.363
126.102
124.558
124.204
124.135
119.619
119.399
117.311
77.213
77.000
76.789
36.492
Br
Br
N
O
O
4d
200
180
160
140
120
100
80
60
40
20
0 ppm

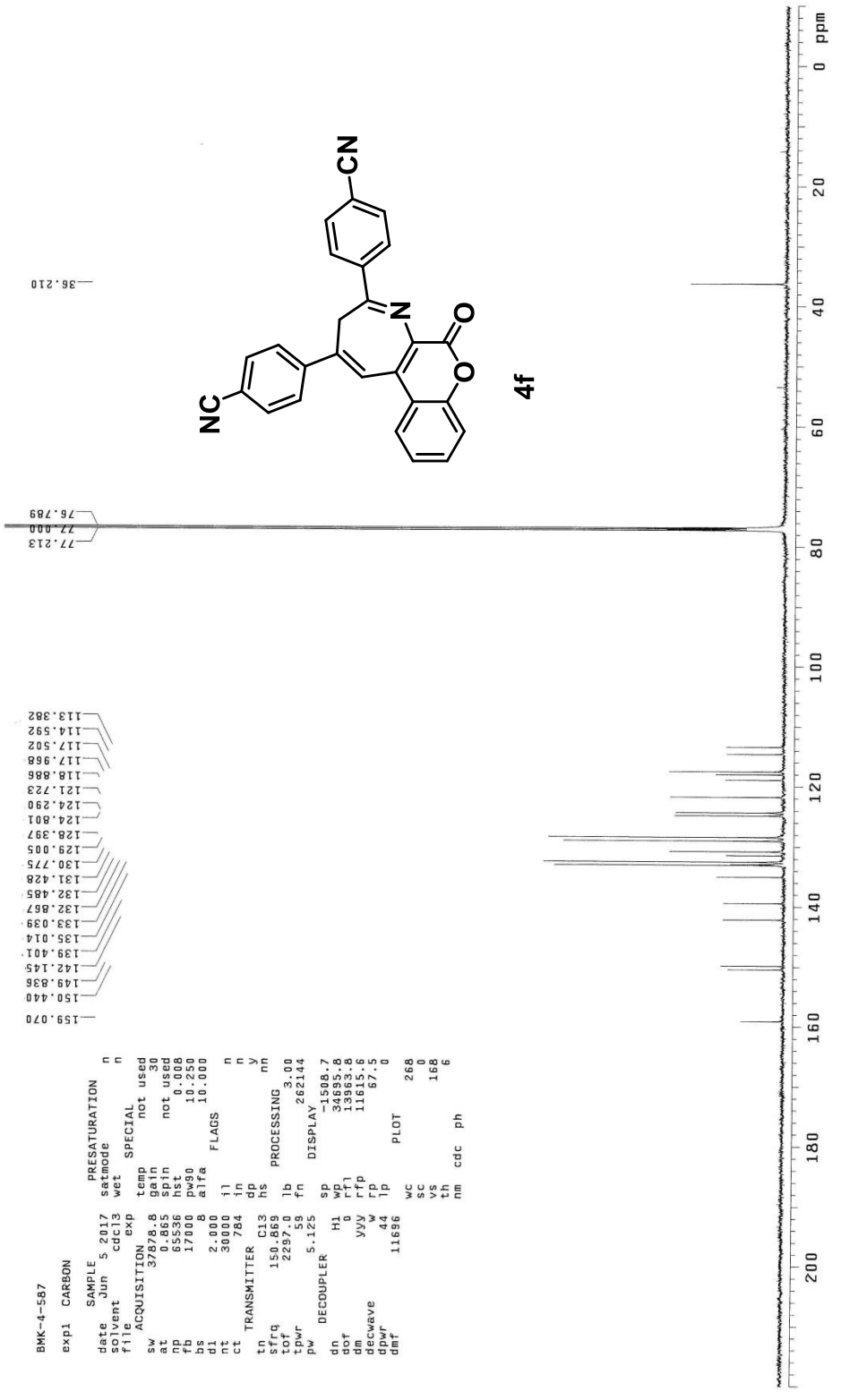

BMK-4-550
8.382
8.360
8.222
8.200
8.054
8.032
7.848
7.829
7.799
7.777
7.599
7.596
7.578
7.560
7.558
7.530
7.491
7.472
7.404
7.384
7.366
7.364
7.284
5.322
4.940
2.512
2.377
2.302
4e
1.053
1.036
1.667
1.675
1.634
0.975
1.642
1.071
0.946
0.882
1.000
ppm

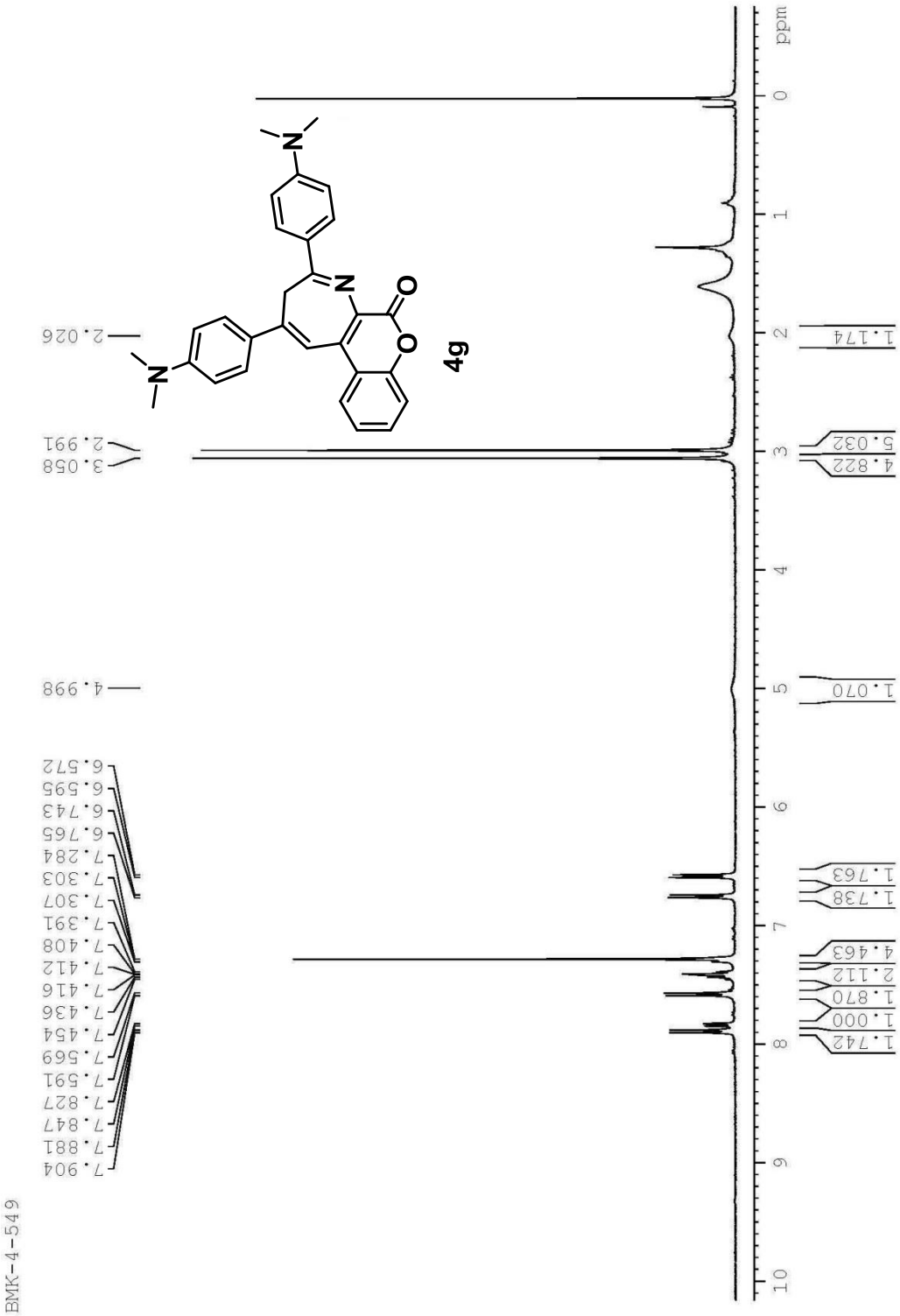

BMK-4-550
7.284
7.364
7.366
7.384
7.404
7.472
7.491
7.530
7.558
7.560
7.578
7.596
7.599
7.777
7.799
7.829
7.848
8.032
8.054
8.200
8.222
8.360
8.382
4e
ppm
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.3
8.4
8.5
1.000
0.882
0.946
1.071
1.642
0.975
1.634
1.675
1.667

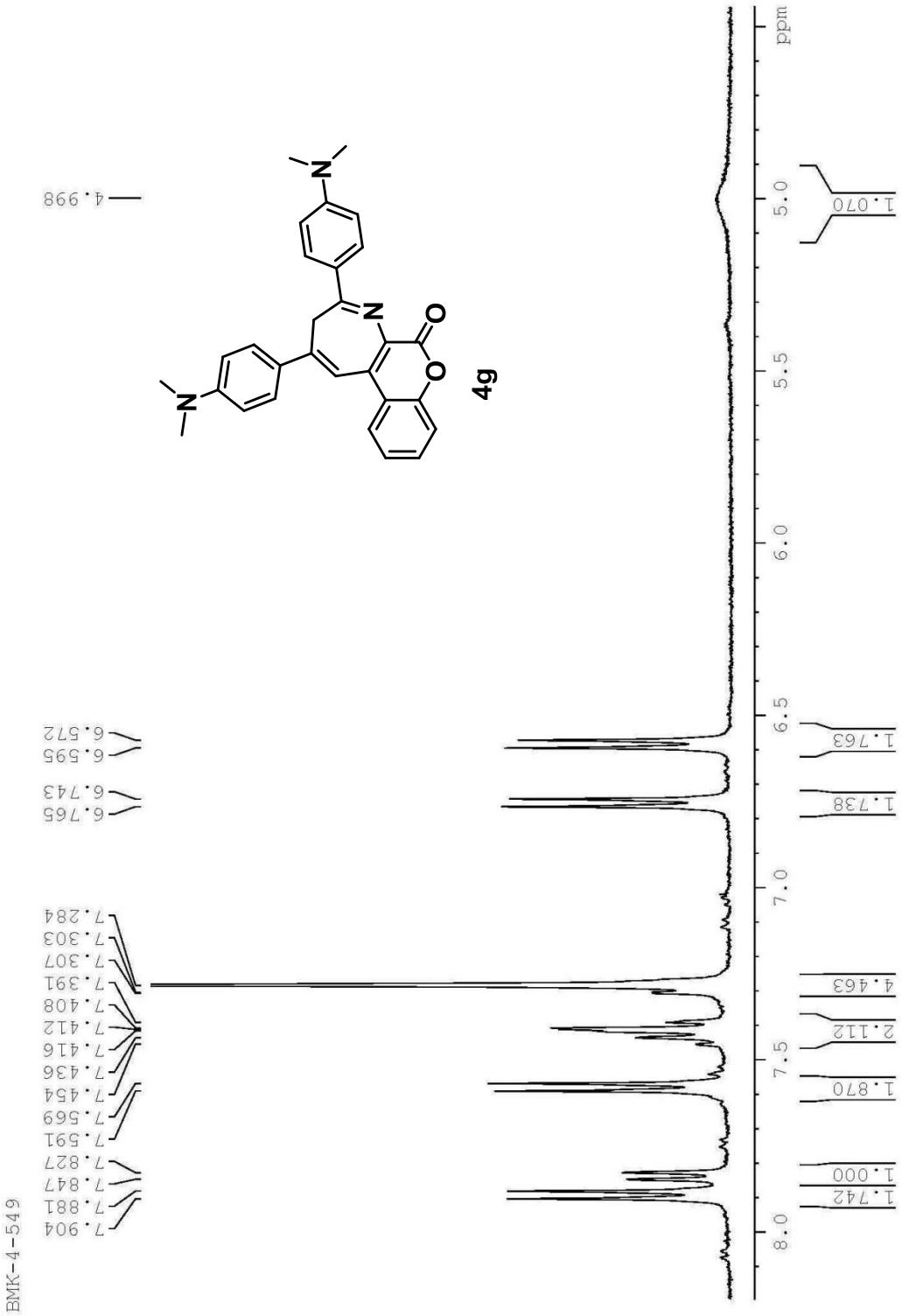

BMK-4-550
exp1 CARBON
SAMPLE
date Jun 7 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 18984
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode n
wet SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13959.5
rfp 11615.6
rp 40.1
lp 0
PLOT
wc 268
sc 0
vs 951
th 7
nm cdc ph
159.001
150.539
149.478
149.198
148.321
143.930
141.001
134.577
133.068
131.449
130.924
129.506
128.713
124.876
124.600
124.326
123.966
122.213
118.820
117.600
77.211
77.000
76.787
36.586
O2N
NO2
O
N
O
4e
200
180
160
140
120
100
80
60
40
20
0 ppm

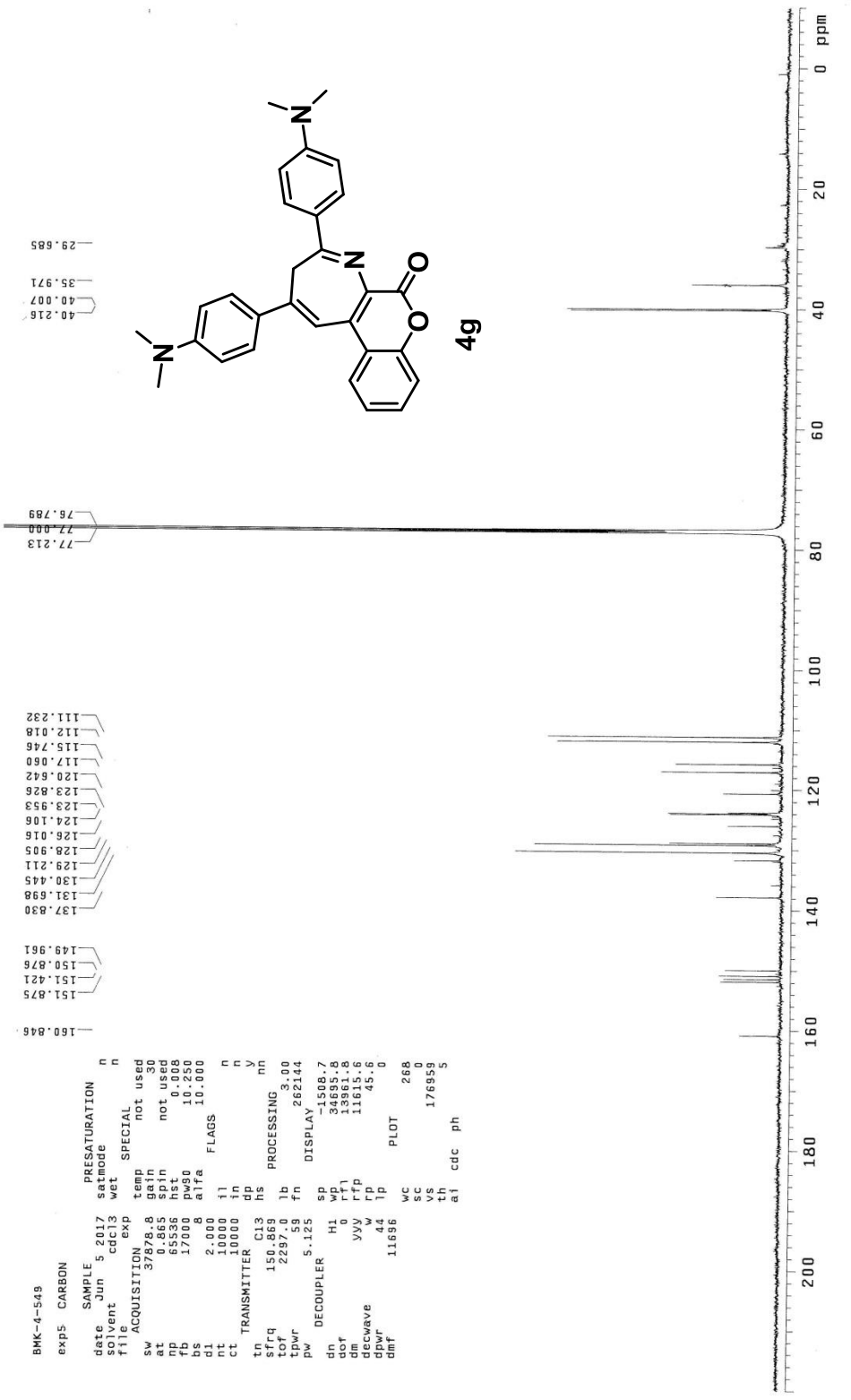

BMK-4-587
4f
CN
NC
N
O
O
ppm
0
1
2
3
4
5
6
7
8
9
10
0.995
0.990
1.877
2.806
1.868
1.884
0.994
1.904
1.000
2.248
2.259
2.350
2.456
4.891
5.096
7.284
7.349
7.352
7.370
7.387
7.390
7.454
7.456
7.474
7.542
7.545
7.561
7.563
7.646
7.650
7.663
7.667
7.706
7.710
7.723
7.728
7.790
7.794
7.799
7.811
7.815
7.832
7.835
7.960
7.965

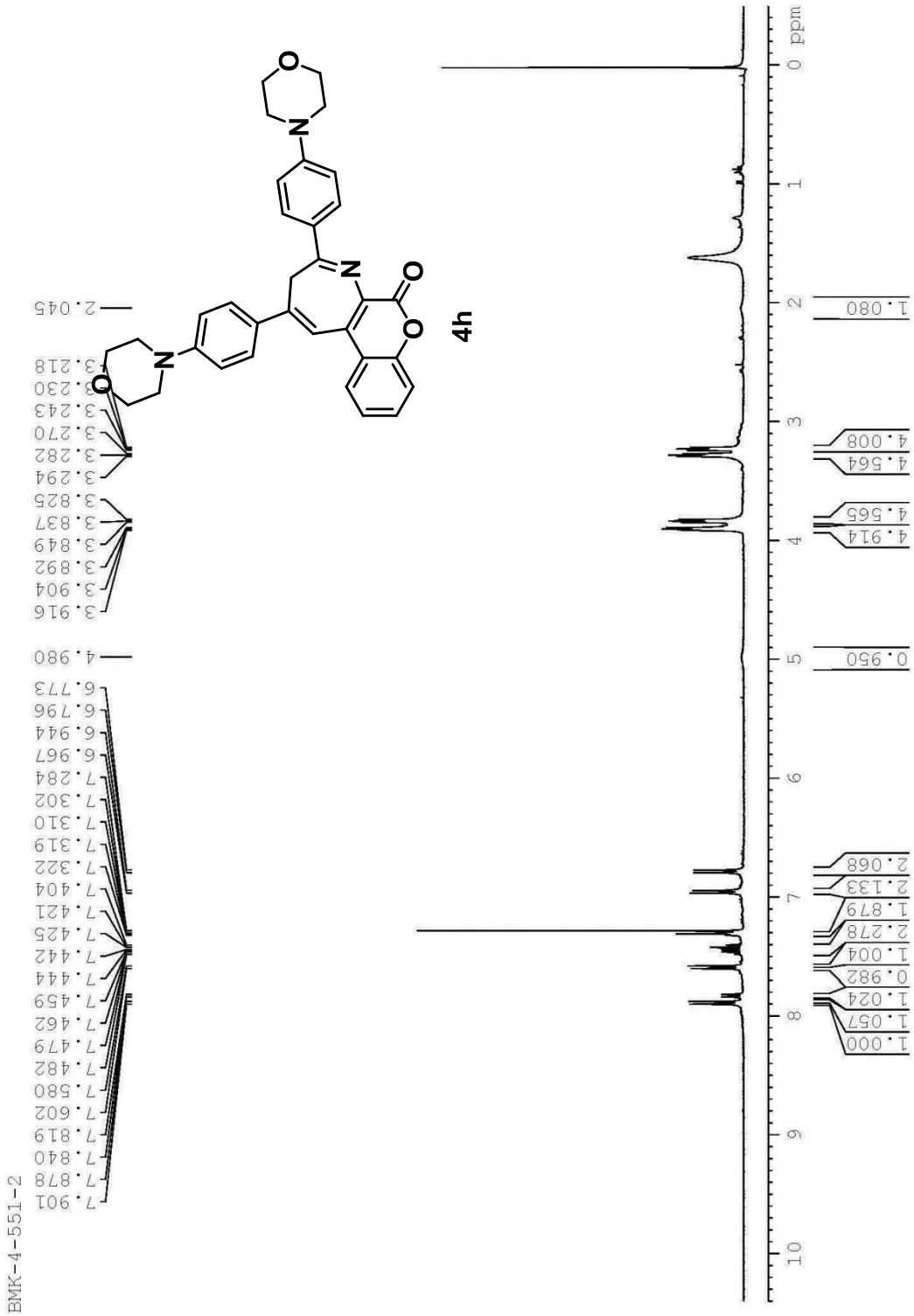

BMK-4-587
7.284
7.349
7.352
7.370
7.387
7.390
7.454
7.456
7.474
7.542
7.545
7.561
7.563
7.646
7.650
7.663
7.667
7.706
7.710
7.723
7.728
7.790
7.794
7.799
7.811
7.815
7.832
7.835
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
ppm
1.000
1.904
0.994
1.884
1.868
2.806
4f
CN
NC
N
O
O


4f
NC
CN
O
N
O
BMK-4-587
exp1 CARBON
SAMPLE
date Jun 5 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 784
TRANSMITTER C13
tn
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode n
wet SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rf1 13963.8
rfp 11615.6
rp 67.5
lp 0
PLOT
wc 268
sc 0
vs 168
th 6
nm cdc ph
159.070
150.440
149.836
142.145
139.401
135.014
133.039
132.867
132.485
131.428
130.775
129.005
128.397
124.801
124.290
121.723
118.886
117.968
117.502
114.592
113.382
77.213
77.000
76.789
36.210
200
180
160
140
120
100
80
60
40
20
0 ppm


BMK-4-549
7.904
7.881
7.847
7.827
7.591
7.569
7.454
7.436
7.416
7.412
7.408
7.391
7.307
7.303
7.284
6.765
6.743
6.595
6.572
4.998
3.058
2.991
2.026
4g
1.742
1.000
1.870
2.112
4.463
1.738
1.763
1.070
4.822
5.032
1.174
ppm


BMK-4-549
7.904
7.881
7.847
7.827
7.591
7.569
7.454
7.436
7.416
7.412
7.408
7.391
7.307
7.303
7.284
6.765
6.743
6.595
6.572
4.998
1.742
1.000
1.870
2.112
4.463
1.738
1.763
1.070
8.0
7.5
7.0
6.5
6.0
5.5
5.0
ppm
4g


4g
BMK-4-549
exp5 CARBON
SAMPLE
date Jun 5 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 10000
ct 10000
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave y
dpwr 44
dmf 11696
PRESATURATION n
satmode n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13961.8
rfp 11615.6
rp 45.6
lp 0
PLOT
wc 268
sc 0
vs 176959
th 5
al cdc ph
29.685
35.971
40.007
40.216
76.789
77.000
77.213
111.232
112.018
115.746
117.060
120.642
123.826
123.953
124.106
126.016
128.905
129.211
130.445
131.698
137.830
149.961
150.876
151.421
151.875
160.846
ppm


BMK-4-551-2
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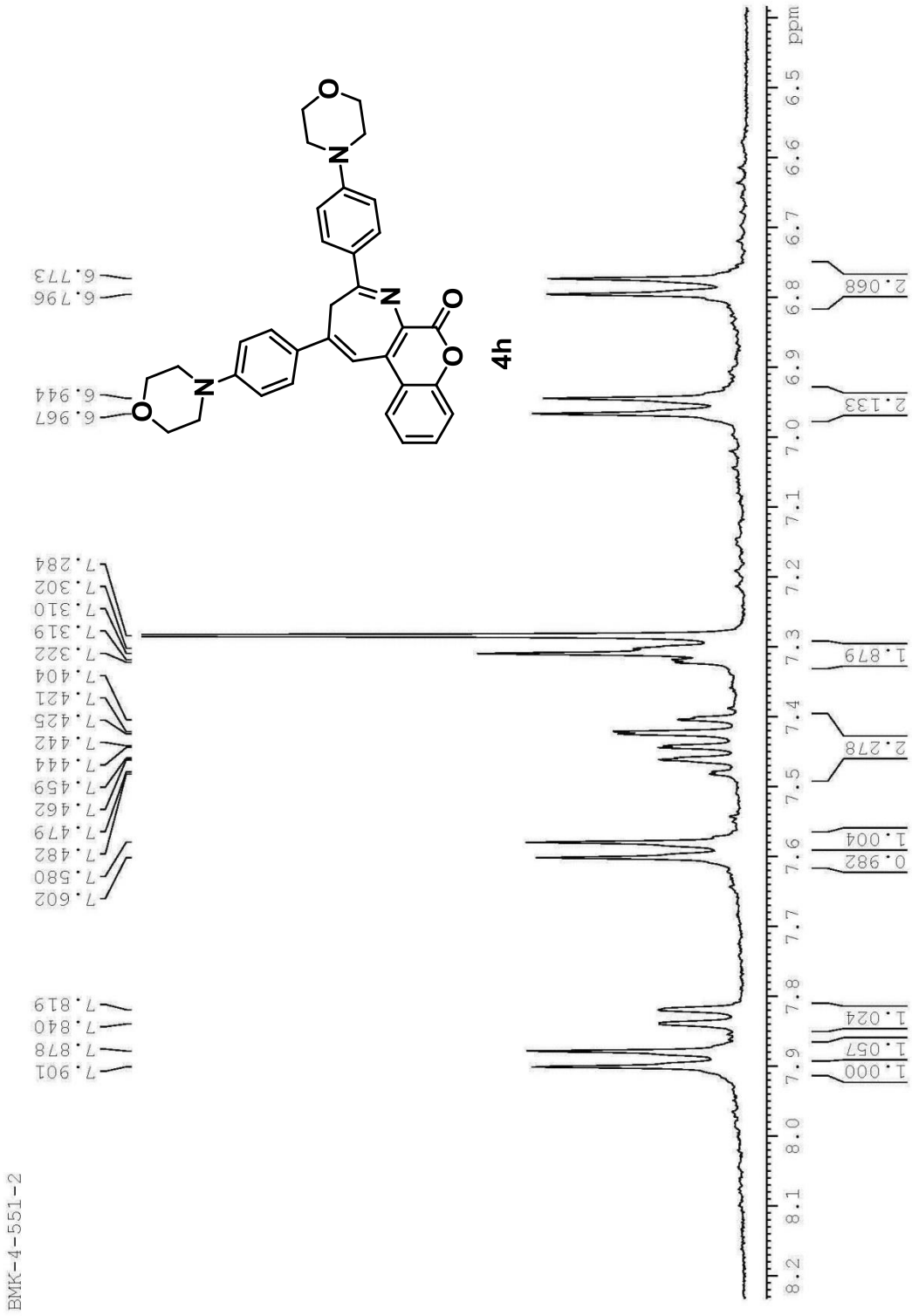
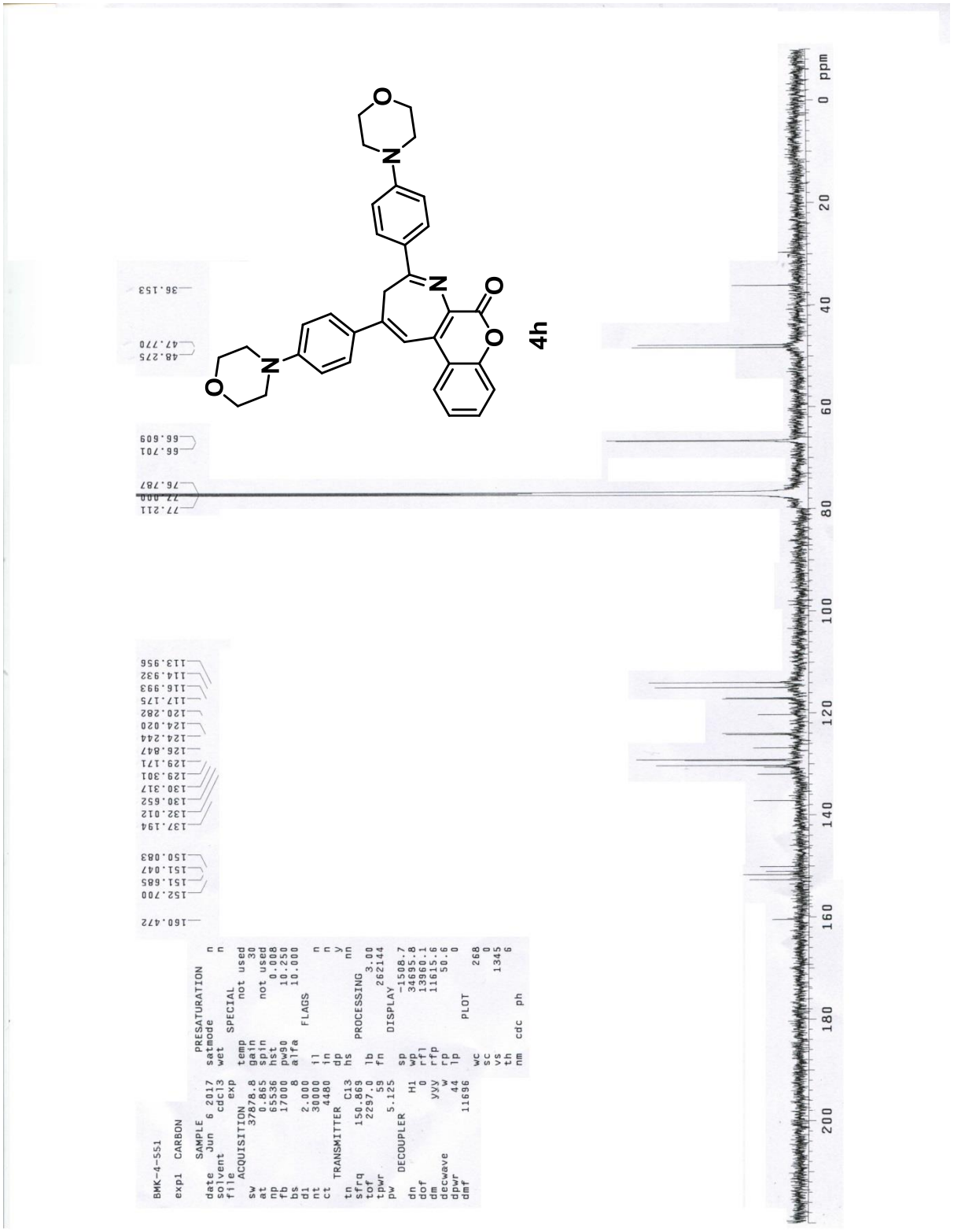
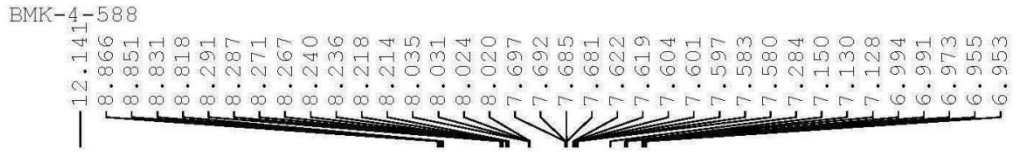
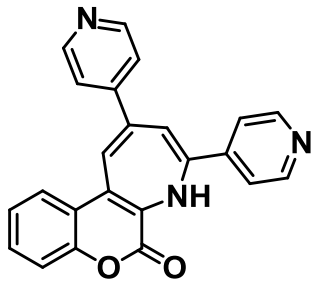
7.878
7.840
7.819
7.602
7.580
7.482
7.479
7.462
7.459
7.444
7.442
7.425
7.421
7.404
7.322
7.319
7.310
7.302
7.284
6.967
6.944
6.796
6.773
4.980
3.916
3.904
3.892
3.849
3.837
3.825
3.294
3.282
3.270
3.243
3.230
3.218
2.045
4h
0 ppm
1.080
4.564
4.008
4.565
4.914
0.950
2.068
2.133
1.879
2.278
1.004
0.982
1.024
1.057
1.000

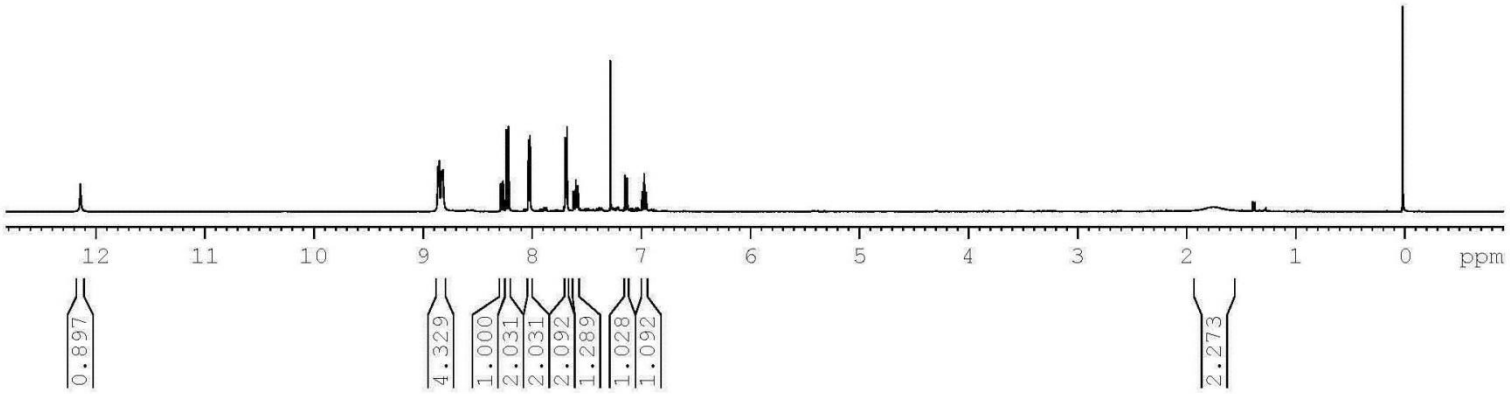

BMK-4-551-2
4h
ppm
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
7.901
7.878
7.840
7.819
7.602
7.580
7.482
7.479
7.462
7.459
7.444
7.442
7.425
7.421
7.404
7.322
7.319
7.310
7.302
7.284
6.967
6.944
6.796
6.773
1.000
1.057
1.024
0.982
1.004
2.278
1.879
2.133
2.068

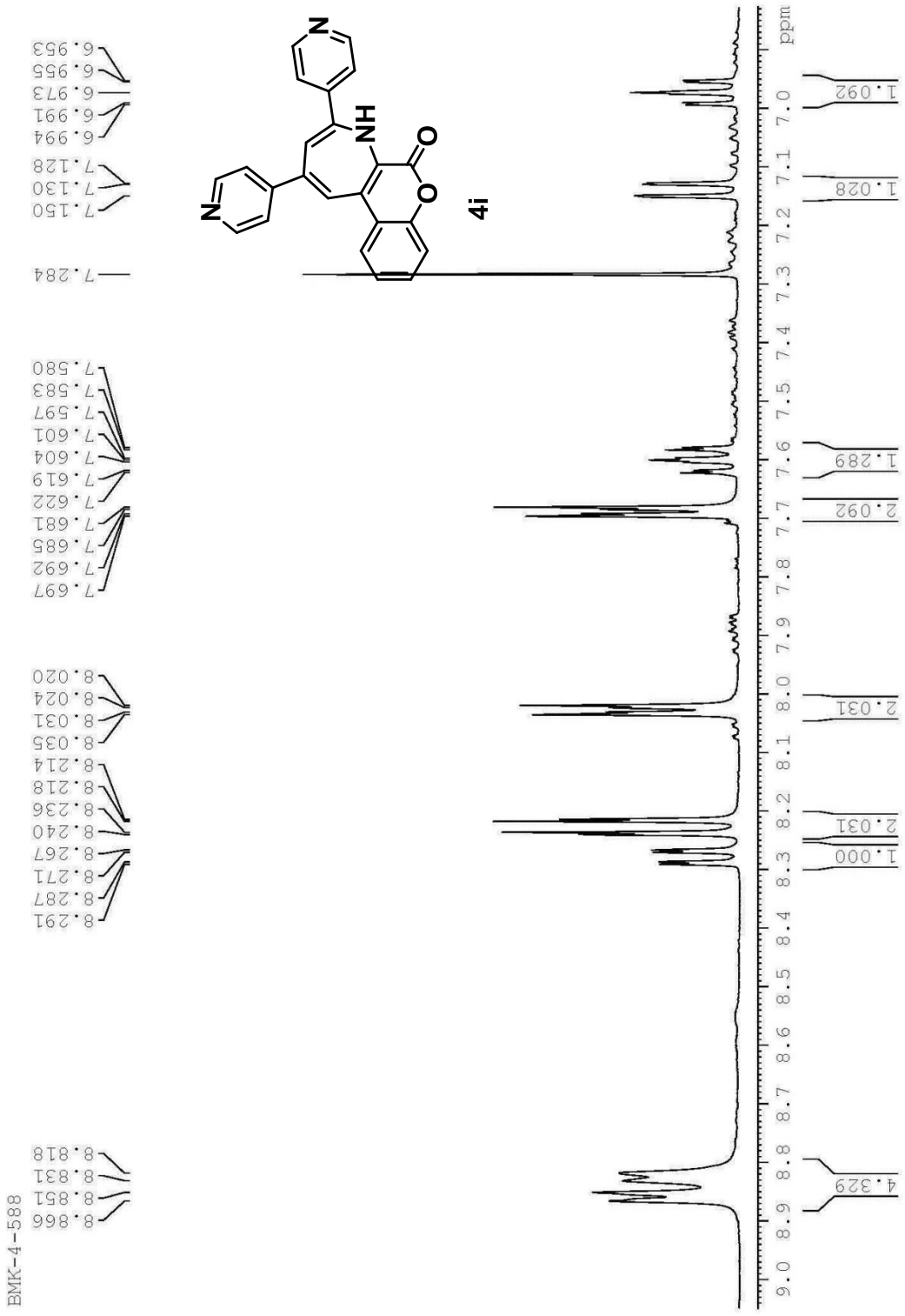

4h
BMK-4-551
exp1 CARBON
SAMPLE
date Jun 6 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 4480
TRANSMITTER
tn C13
sfrq 150.865
tof 2297.0
tpwr 53
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode n
wet
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13960.1
rfp 11615.6
rp 50.6
lp 0
PLOT
wc 268
sc 0
vs 1345
th 6
nm cdc ph
160.472
152.700
151.685
151.047
150.083
137.194
132.012
130.652
130.317
129.301
129.171
126.847
124.244
124.020
120.282
117.175
116.993
114.932
113.956
77.211
77.000
76.787
66.701
66.609
48.275
47.770
36.153
0 ppm

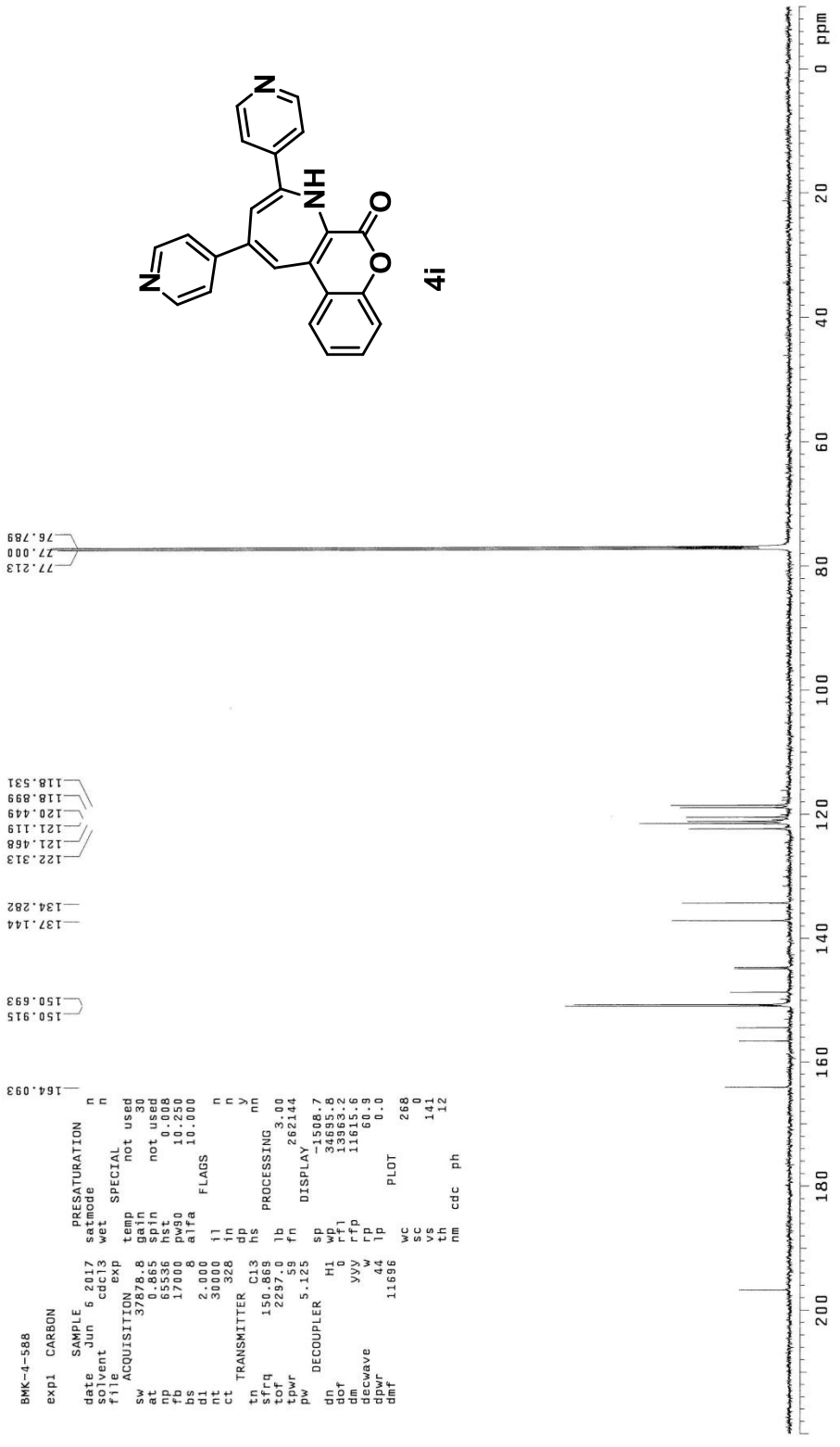

BMK-4-588
12.141
8.866
8.851
8.831
8.818
8.291
8.287
8.271
8.267
8.240
8.236
8.218
8.214
8.035
8.031
8.024
8.020
7.697
7.692
7.685
7.681
7.622
7.619
7.604
7.601
7.597
7.583
7.580
7.284
7.150
7.130
7.128
6.994
6.991
6.973
6.955
6.953
1.752

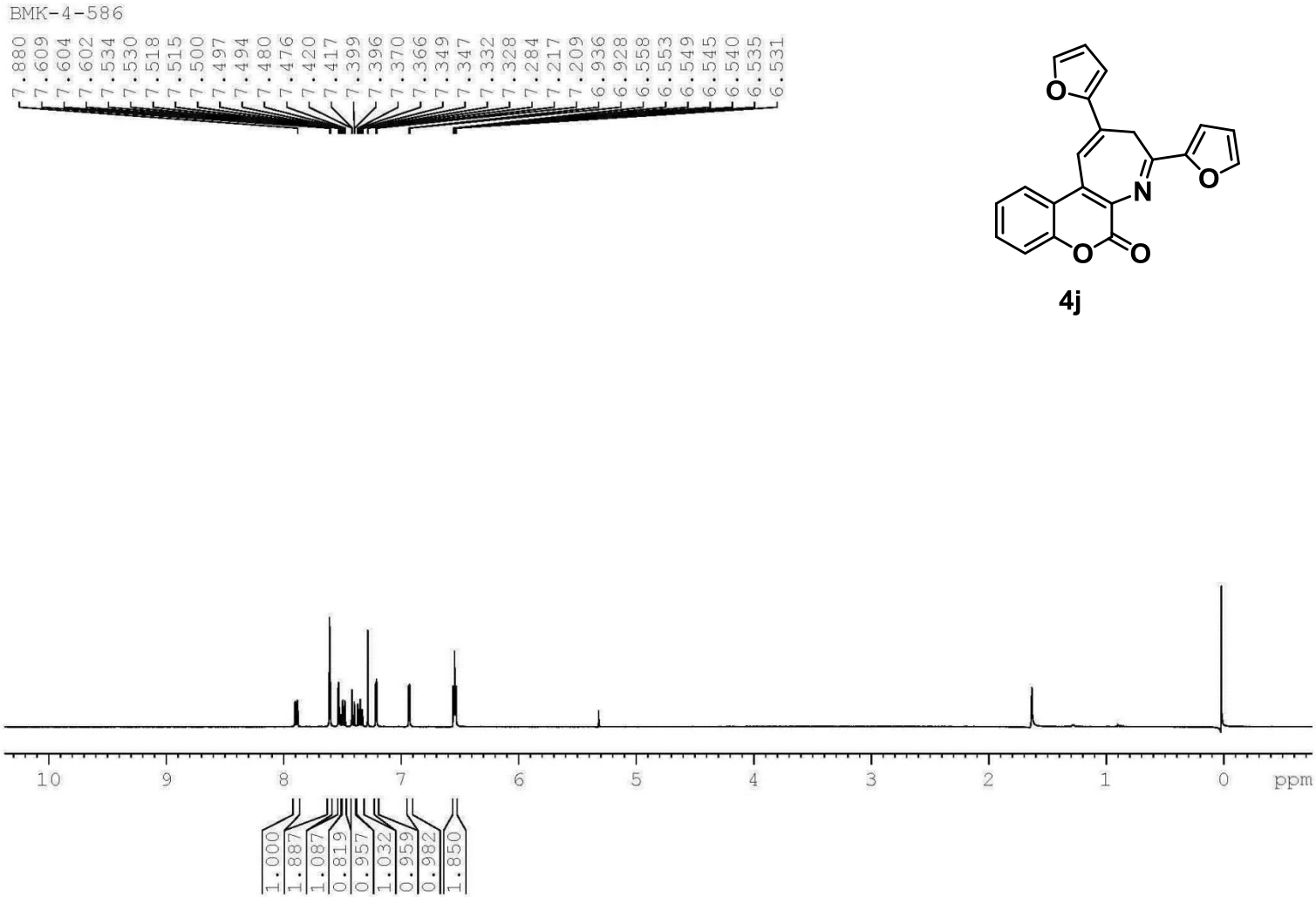


4i

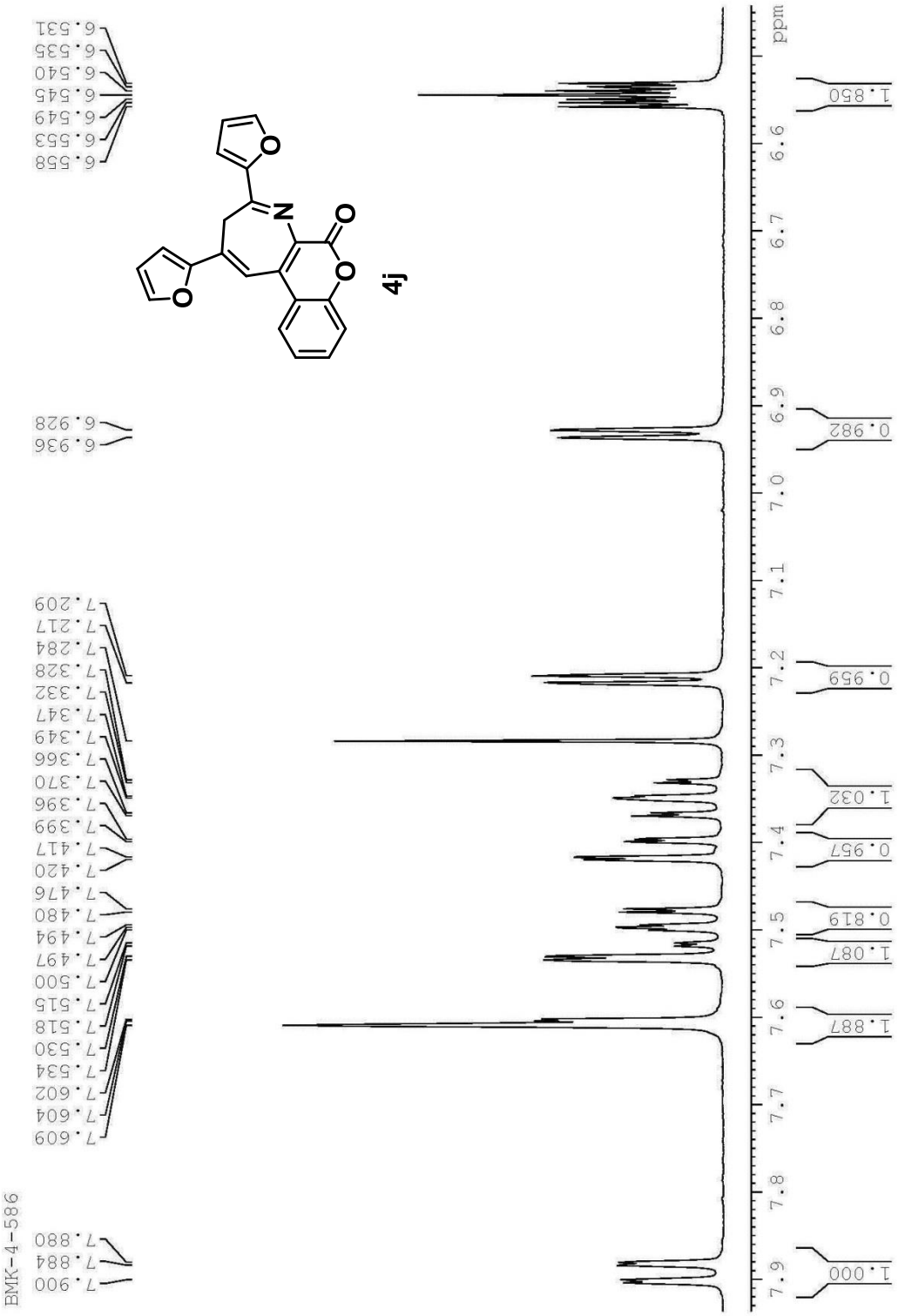

12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.897
4.329
1.000
2.031
2.031
2.092
1.289
1.028
1.092
2.273

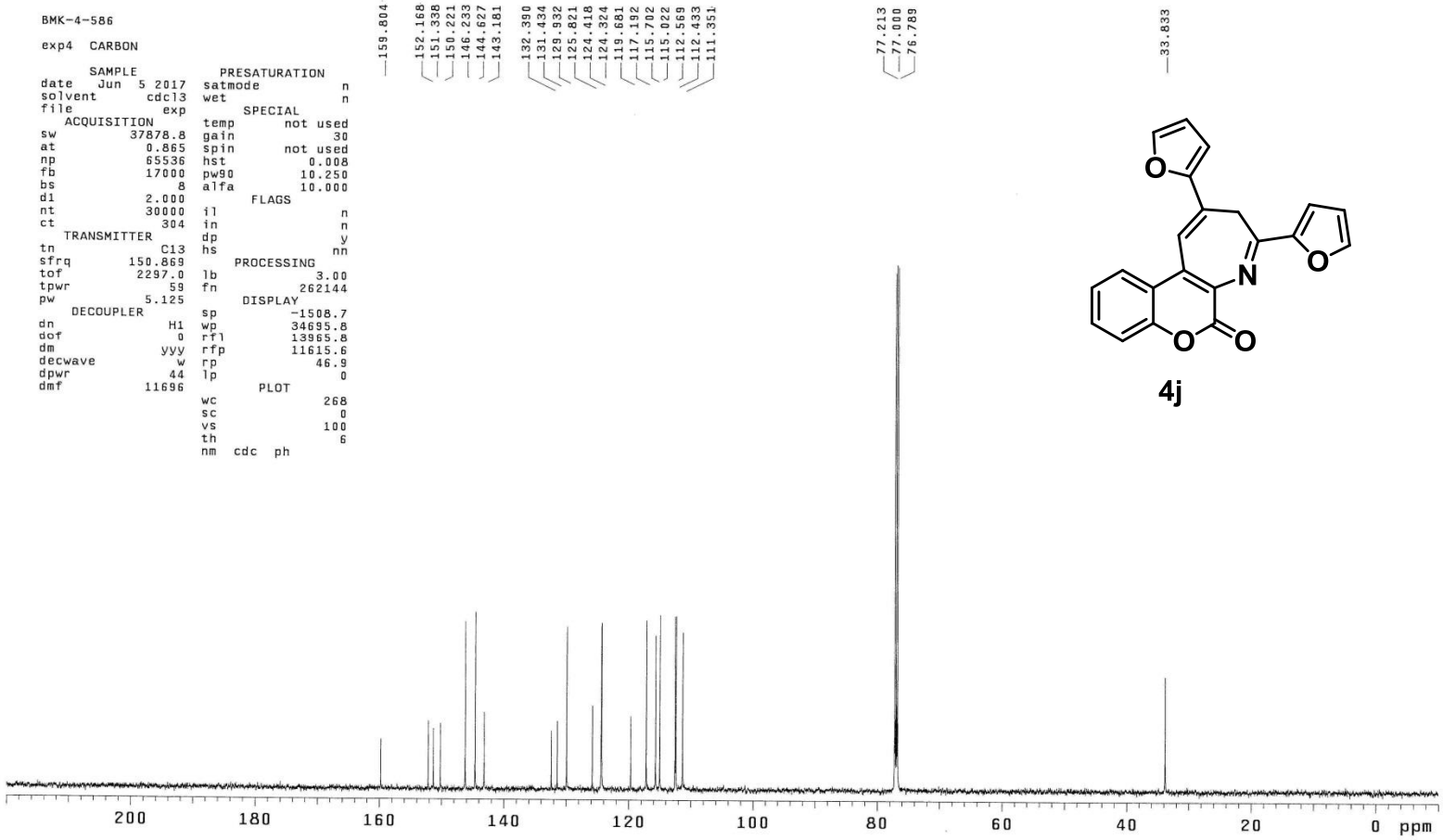

BMK-4-588
8.866
8.851
8.831
8.818
8.291
8.287
8.271
8.267
8.240
8.236
8.218
8.214
8.035
8.031
8.024
8.020
7.697
7.692
7.685
7.681
7.622
7.619
7.604
7.601
7.597
7.583
7.580
7.284
7.150
7.130
7.128
6.994
6.991
6.973
6.955
6.953
4i
ppm
9.0
8.9
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
4.329
1.000
2.031
2.031
1.289
2.092
1.028
1.092


4i
BMK-4-588
exp1 CARBON
SAMPLE
date Jun 6 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 328
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode n
wet SPECIAL
temp not used 30
gain not used
spin 0.008
hst 10.250
pw90 10.000
alfa
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13963.2
rfp 11615.6
rp 60.9
lp 0.0
PLOT
wc 268
sc 0
vs 141
th 12
nm cdc ph
164.093
150.915
150.693
137.144
134.282
122.313
121.468
121.119
120.449
118.899
118.531
77.213
77.000
76.789
200
180
160
140
120
100
80
60
40
20
0 ppm


BMK-4-586
7.880
7.609
7.604
7.602
7.534
7.530
7.518
7.515
7.500
7.497
7.494
7.480
7.476
7.420
7.417
7.399
7.396
7.370
7.366
7.349
7.347
7.332
7.328
7.284
7.217
7.209
6.936
6.928
6.558
6.553
6.549
6.545
6.540
6.535
6.531
O
O
N
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.887
1.087
0.819
0.957
1.032
0.959
0.982
1.850

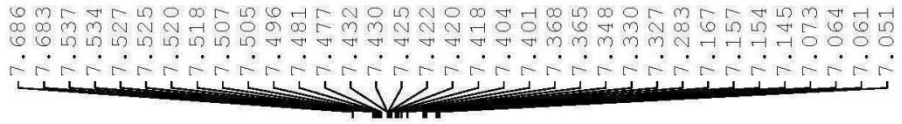
4j

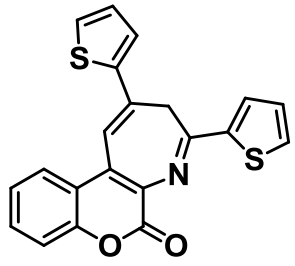

BMK-4-586
7.900
7.884
7.880
7.609
7.604
7.602
7.534
7.530
7.518
7.515
7.500
7.497
7.494
7.480
7.476
7.420
7.417
7.399
7.396
7.370
7.366
7.349
7.347
7.332
7.328
7.284
7.217
7.209
6.936
6.928
6.558
6.553
6.549
6.545
6.540
6.535
6.531
4j
ppm
1.000
0.959
1.032
0.957
0.819
1.087
1.887
0.982
1.850


BMK-4-586
exp4 CARBON
SAMPLE
date Jun 5 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 304
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13965.8
rfp 11615.6
rp 46.9
lp 0
PLOT
wc 268
sc 0
vs 100
th 6
nm cdc ph
159.804
152.168
151.338
150.221
146.233
144.627
143.181
132.390
131.434
129.932
125.821
124.418
124.324
119.681
117.192
115.702
115.022
112.569
112.433
111.351
77.213
77.000
76.789
33.833
O
O
N
O
O
200
180
160
140
120
100
80
60
40
20
0 ppm

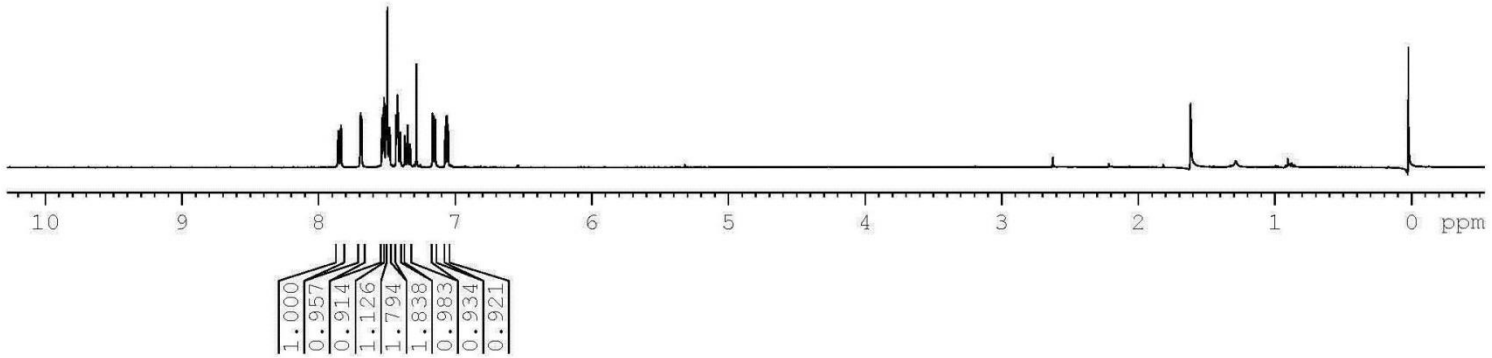
4j

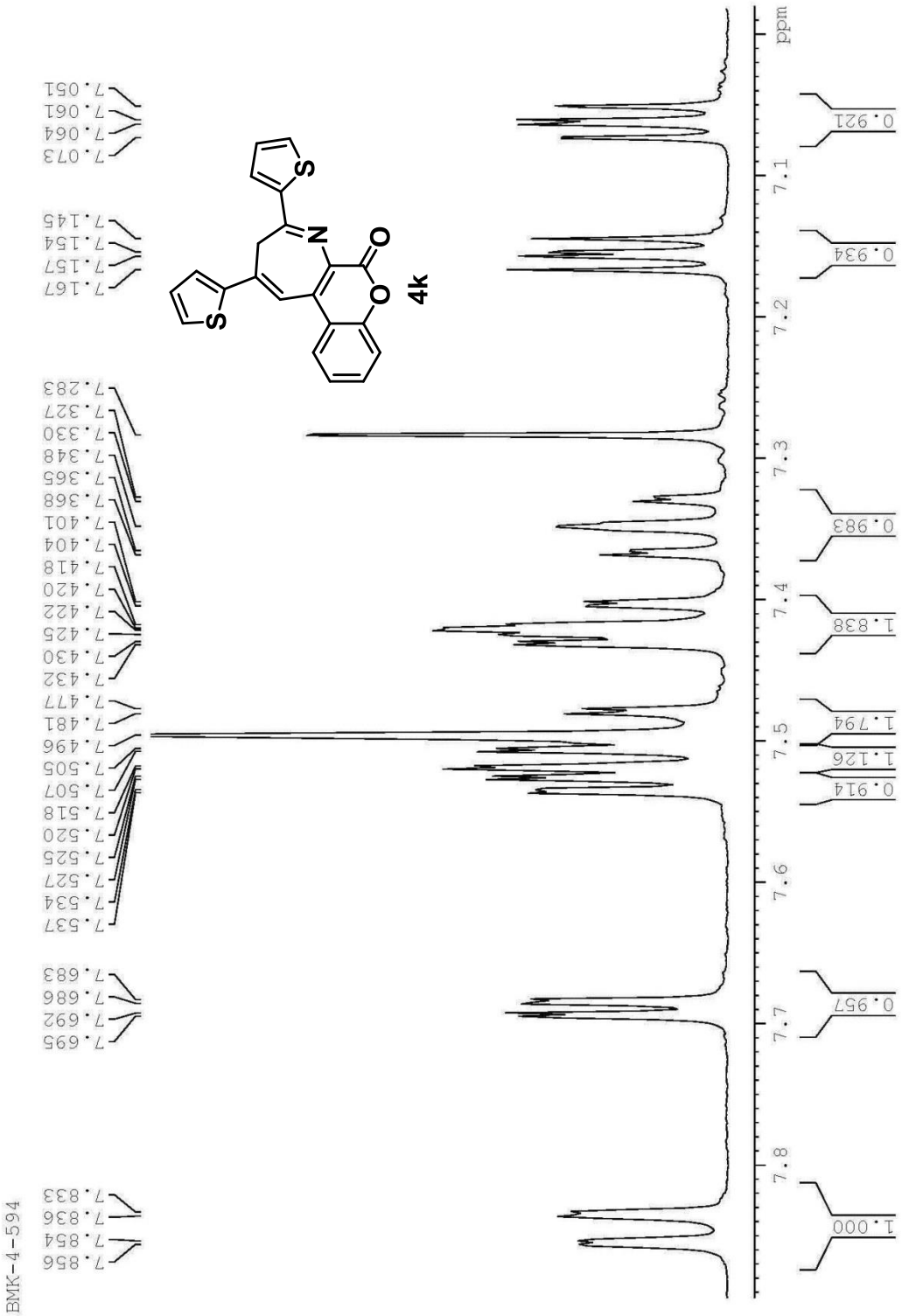
BMK-4-594

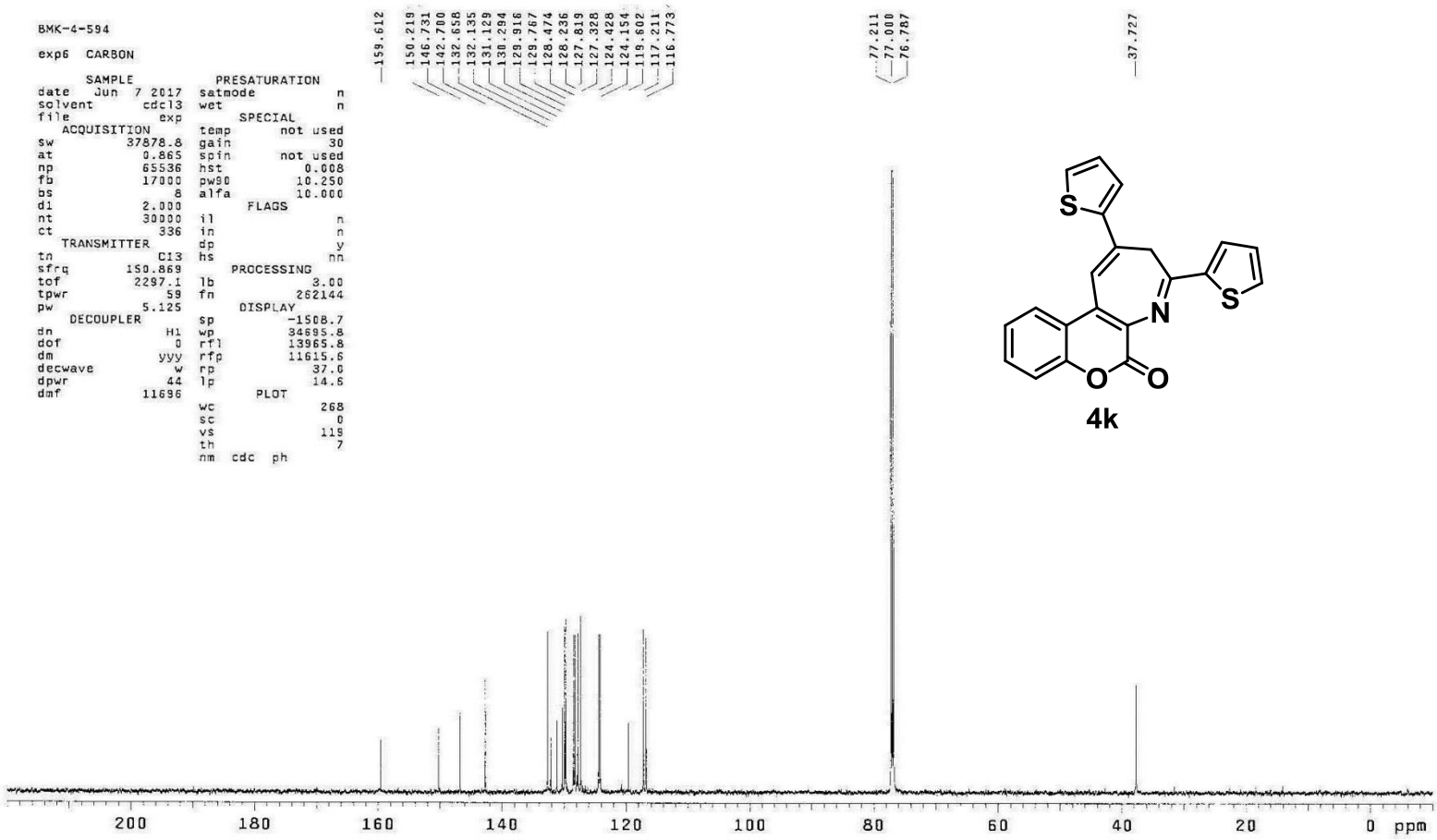

7.686
7.683
7.537
7.534
7.527
7.525
7.520
7.518
7.507
7.505
7.496
7.481
7.477
7.432
7.430
7.425
7.422
7.420
7.418
7.404
7.401
7.368
7.365
7.348
7.330
7.327
7.283
7.167
7.157
7.154
7.145
7.073
7.064
7.061
7.051

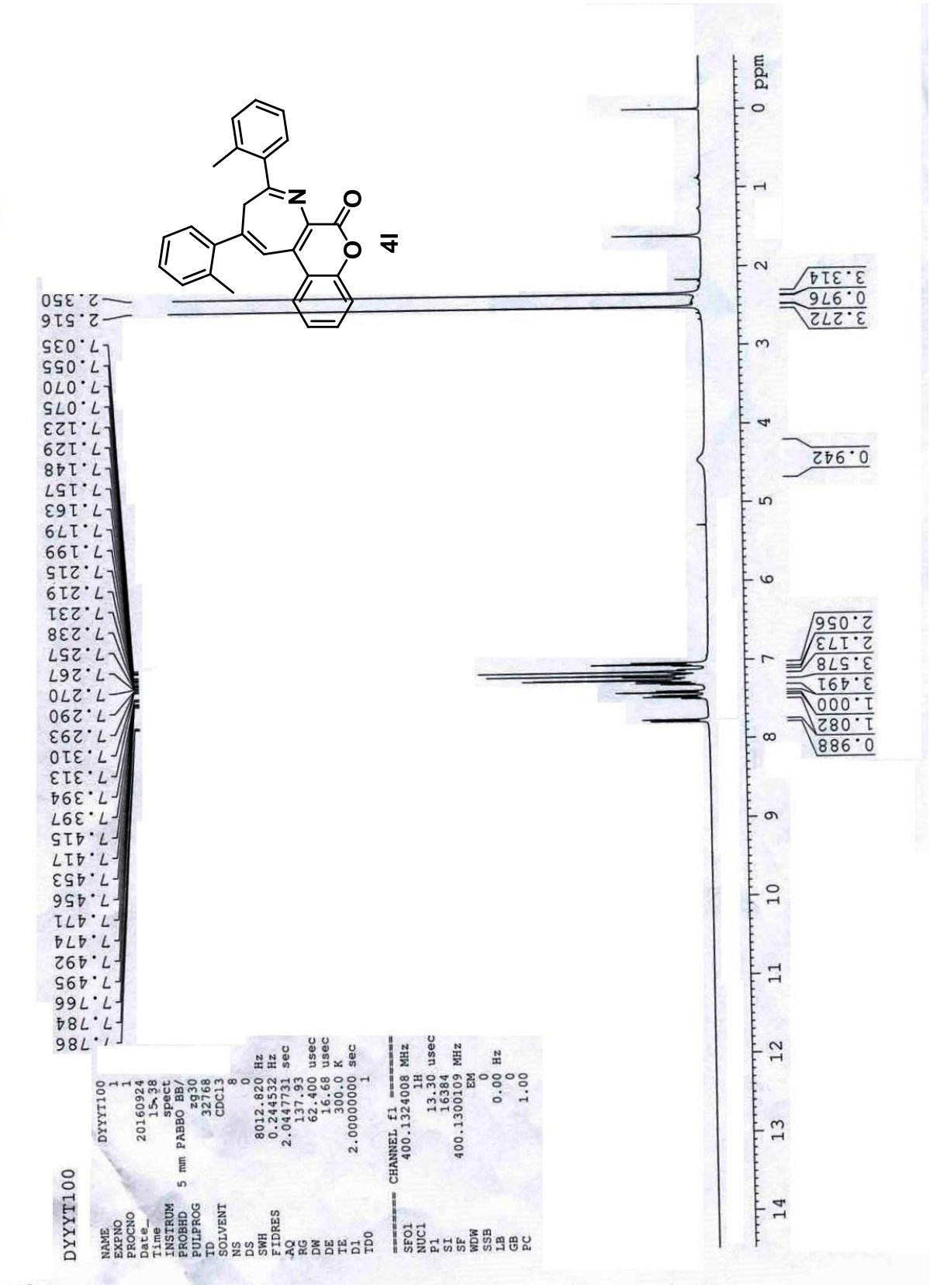

S
S
N
O
O

4k

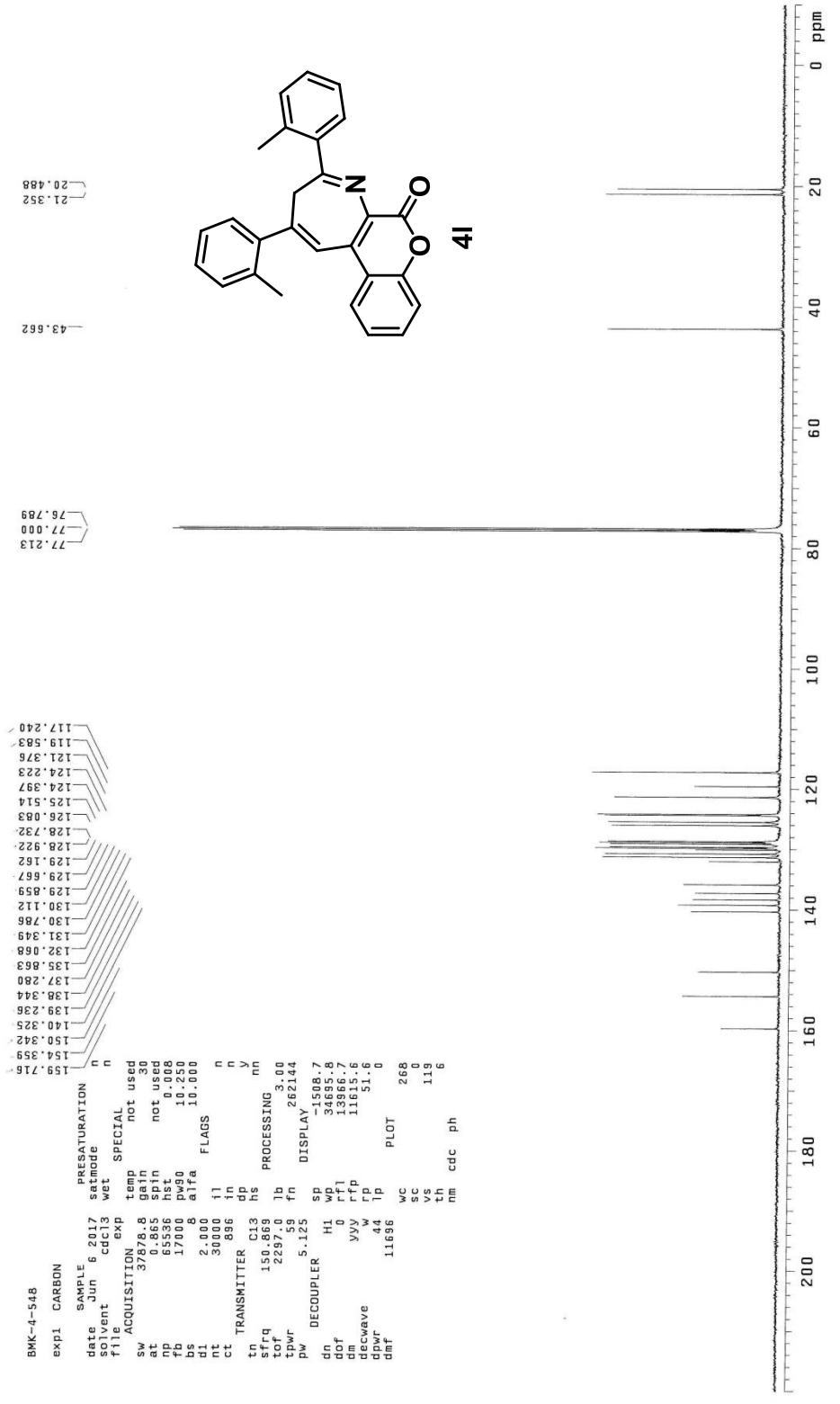

10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
0.957
0.914
1.126
1.794
1.838
0.983
0.934
0.921

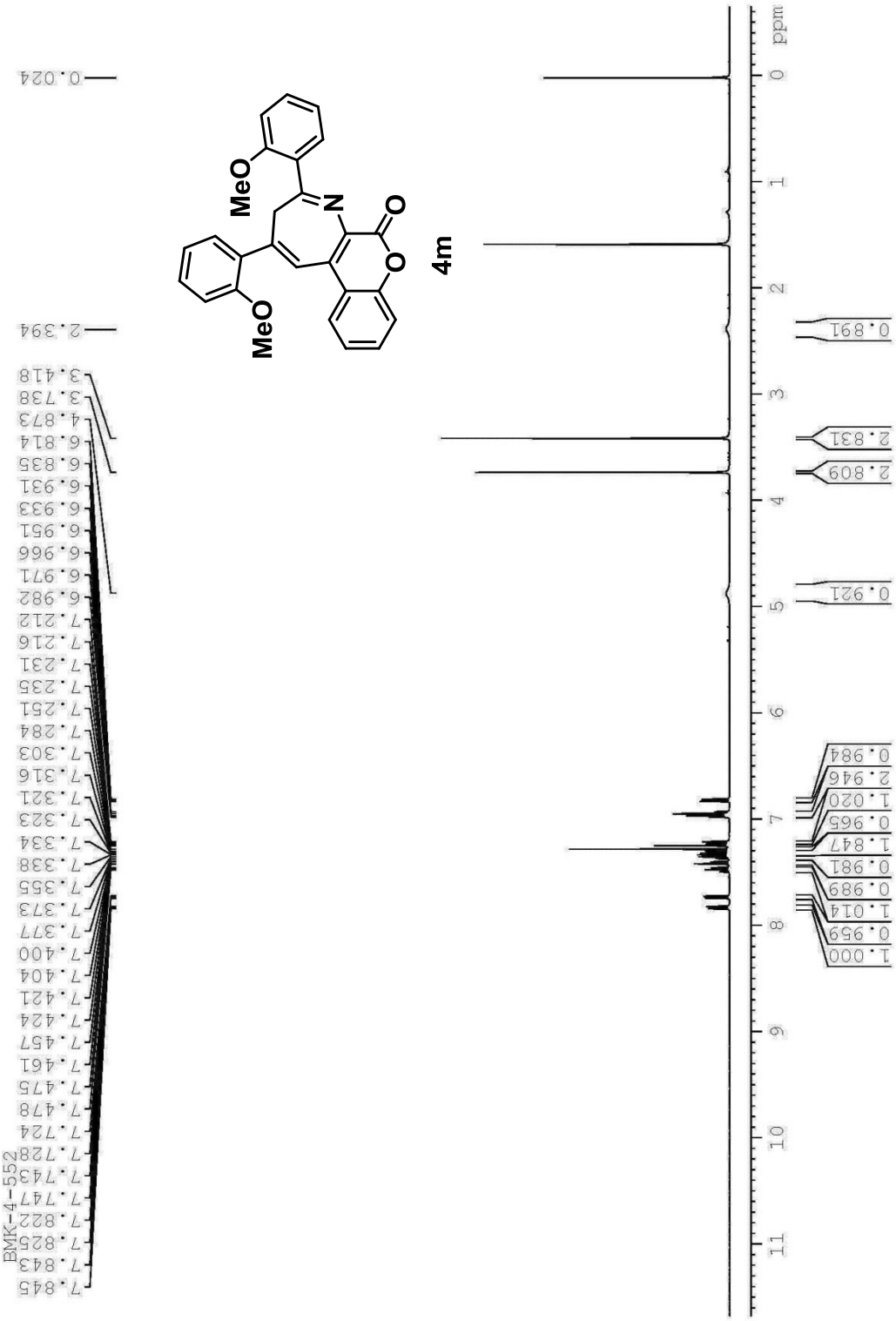

BMK-4-594
7.856
7.854
7.836
7.833
7.695
7.692
7.686
7.683
7.537
7.534
7.527
7.525
7.520
7.518
7.507
7.505
7.496
7.481
7.477
7.432
7.430
7.425
7.422
7.420
7.418
7.404
7.401
7.368
7.365
7.348
7.330
7.327
7.283
7.167
7.157
7.154
7.145
7.073
7.064
7.061
7.051
4k
ppm
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
1.000
0.957
0.914
1.126
1.794
1.838
0.983
0.934
0.921

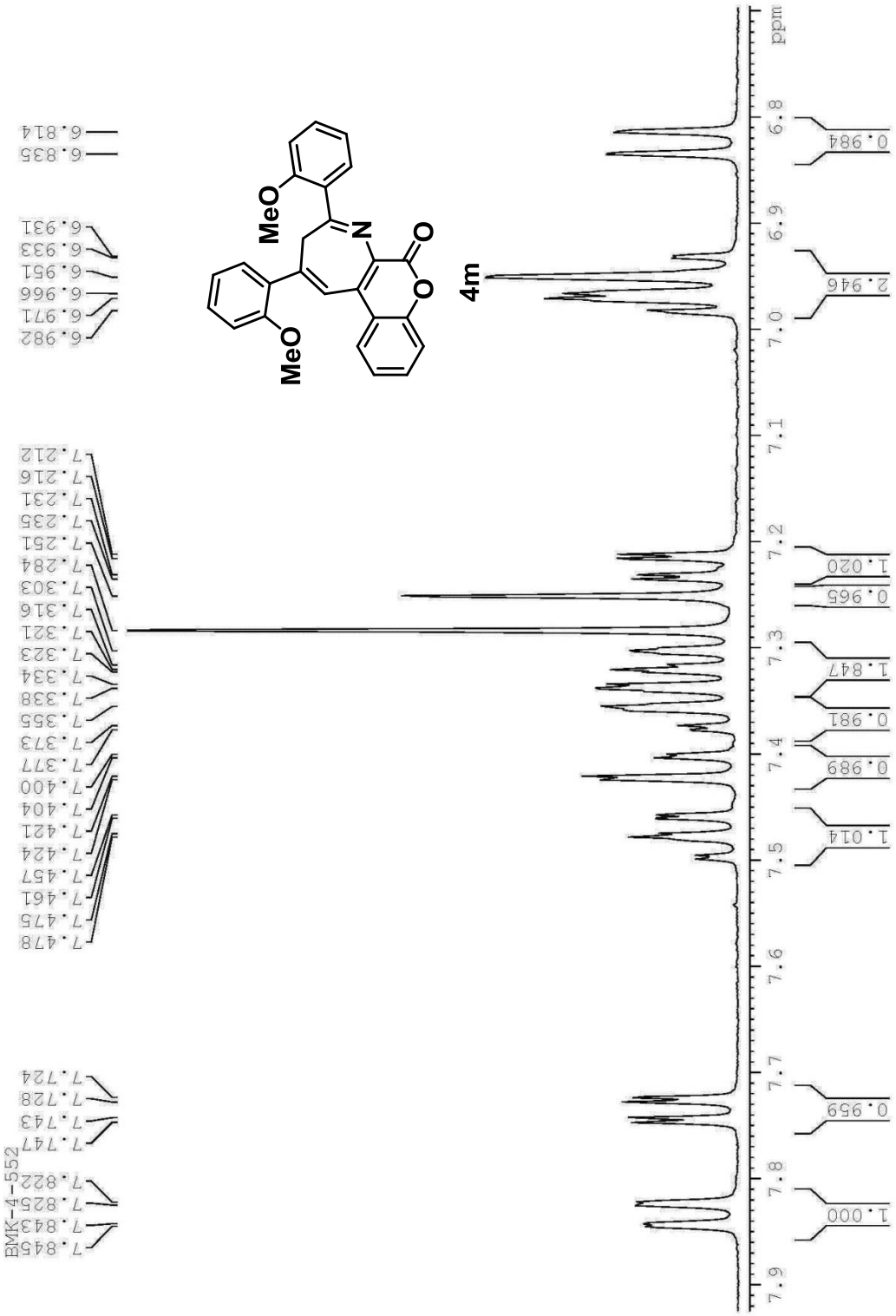

BMK-4-594
exp6 CARBON
SAMPLE
date Jun 7 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 336
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.1
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13965.8
rfp 11615.6
rp 37.0
lp 14.6
PLOT
wc 268
sc 0
vs 118
th 7
nm cdc ph
159.612
150.219
146.731
142.700
132.658
132.135
131.129
130.294
129.916
129.767
128.474
128.236
127.819
127.328
124.428
124.154
119.602
117.211
116.773
77.211
77.000
76.787
37.727
S
S
N
O
O
4k
200
180
160
140
120
100
80
60
40
20
0 ppm

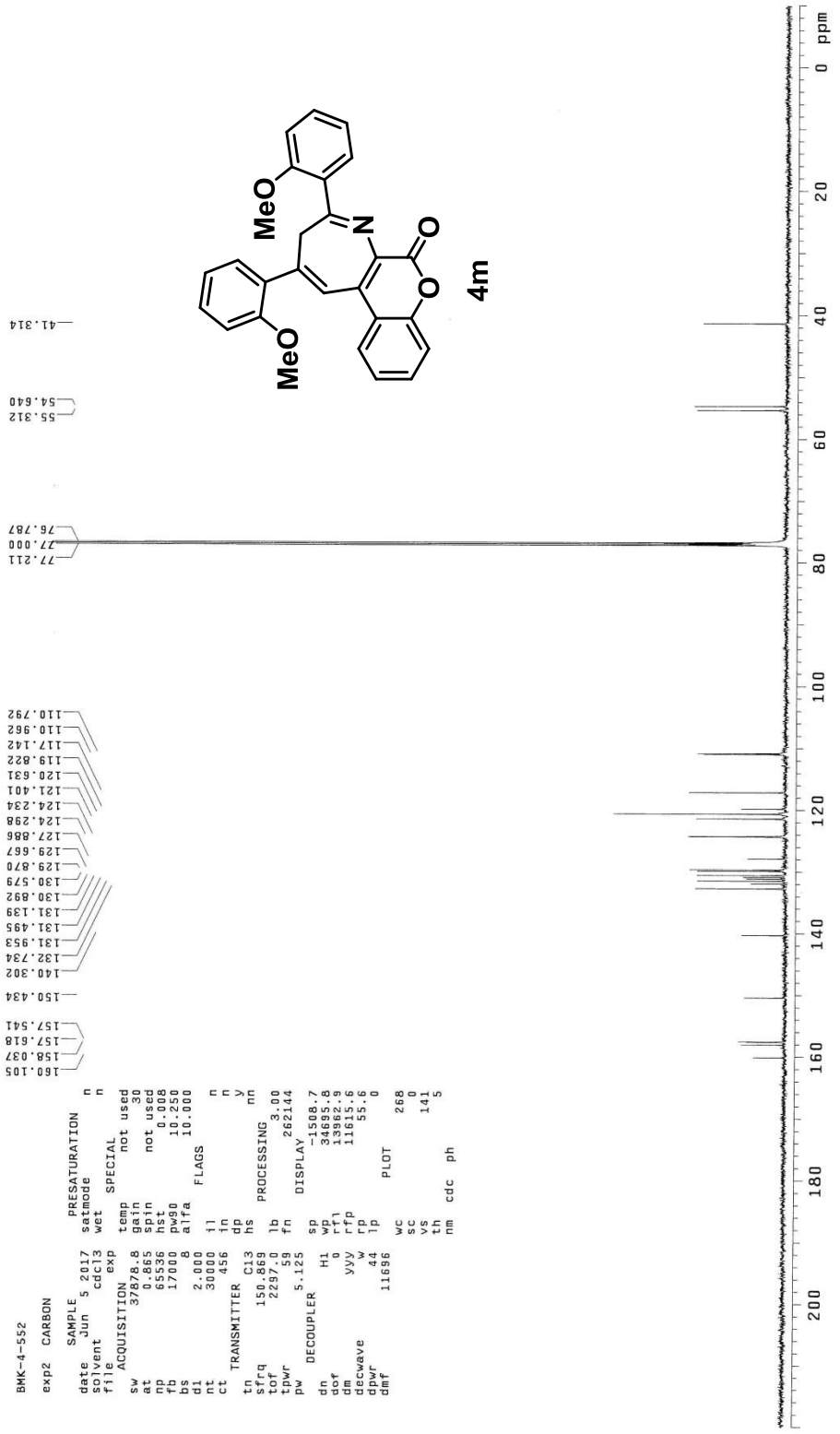

4l
DYYYT100
NAME DYYYT100
EXPNO 1
PROCNO 1
Date_ 20160924
Time 15.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 137.93
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.516
2.350
7.786
7.784
7.766
7.495
7.492
7.474
7.471
7.456
7.453
7.417
7.415
7.397
7.394
7.313
7.310
7.293
7.290
7.270
7.267
7.257
7.238
7.231
7.219
7.215
7.199
7.179
7.163
7.157
7.148
7.129
7.123
7.075
7.070
7.055
7.035
0.988
1.082
1.000
3.491
3.578
2.173
2.056
0.942
3.272
0.976
3.314
ppm

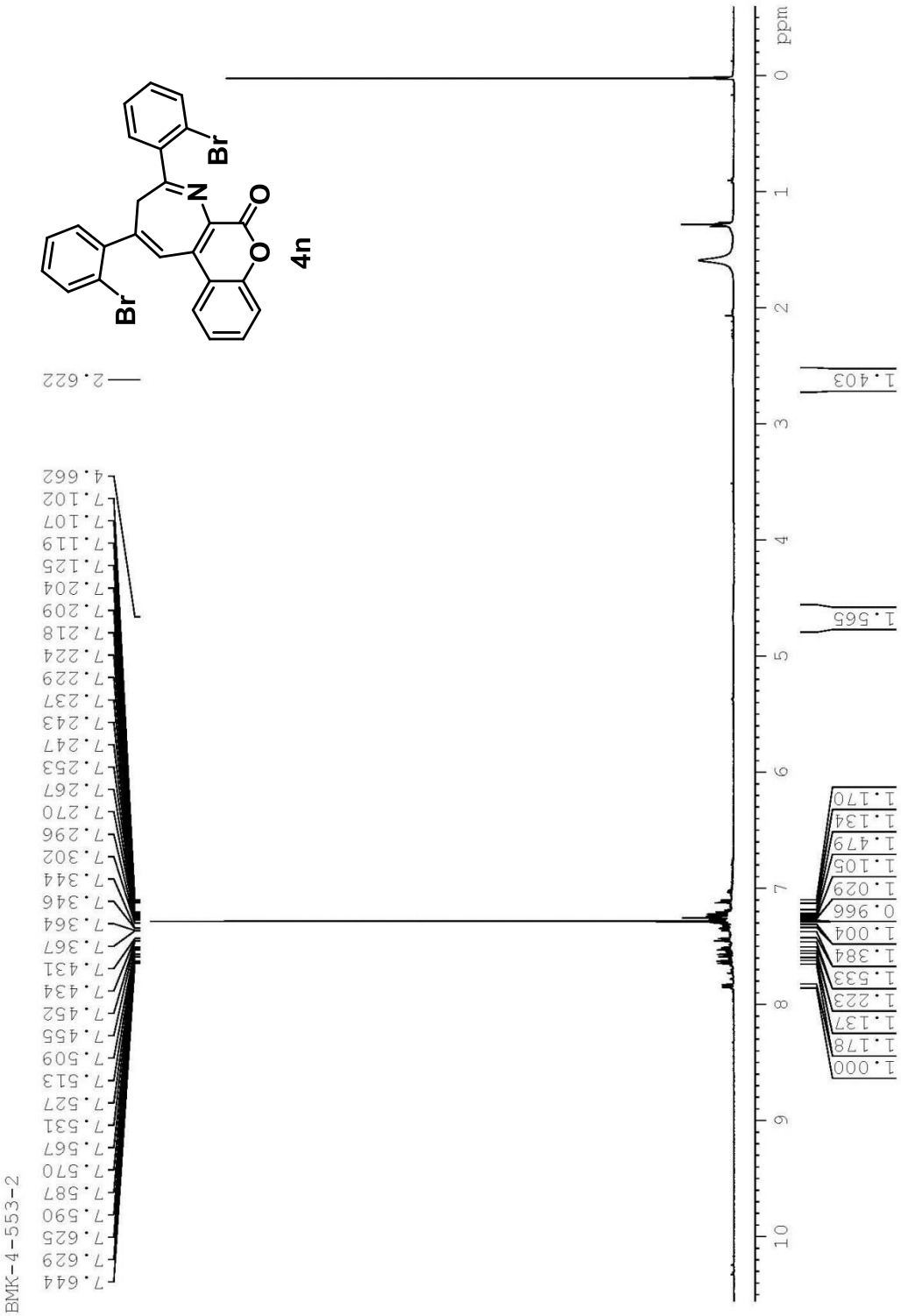
DYYYT100
7.035
7.055
7.070
7.075
7.123
7.129
7.148
7.157
7.163
7.179
7.199
7.215
7.219
7.231
7.238
7.257
7.267
7.270
7.290
7.293
7.310
7.313
7.394
7.397
7.415
7.417
7.453
7.456
7.471
7.474
7.492
7.495
7.766
7.784
7.786
4l
O
N
O
ppm
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
2.056
2.173
3.578
3.491
1.000
1.082
0.988


4l
BMK-4-548
exp1 CARBON
SAMPLE
date Jun 6 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 896
TRANSMITTER C13
tn
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER H1
dn
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp 1508.7
wp 3495.8
rfl 1396.7
rfp 11615.6
rp 51.6
lp 0
PLOT
wc 268
sc 0
vs 119
th 6
nm cdc ph
159.716
154.359
150.342
140.325
139.236
138.344
137.280
135.863
132.068
131.349
130.786
130.112
129.859
129.667
129.162
128.922
128.732
126.083
125.514
124.397
124.223
121.376
119.583
117.240
77.213
77.000
76.789
43.662
21.352
20.488
0 ppm


MeO
MeO
N
O
O
4m
BMK-4-552
ppm


BMK-4-552
MeO
MeO
N
O
O
4m
ppm


MeO
MeO
N
O
O
4m
ppm
0
20
40
60
80
100
120
140
160
180
200
41.314
54.640
55.312
76.787
77.000
77.211
110.792
110.962
117.142
119.822
120.631
121.401
124.234
124.298
127.886
129.667
129.870
130.579
130.892
131.139
131.495
131.953
132.734
140.302
150.434
157.541
157.618
158.037
160.105
BMK-4-552
exp2 CARBON
SAMPLE
date Jun 5 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 456
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
decwave yyy
dpwr 44
dmf 11696
PRESATURATION n
satmode n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13982.9
rfp 11615.6
rp 55.6
lp 0
PLOT
wc 268
sc 0
vs 141
th 5
nm cdc ph


BMK-4-553-2
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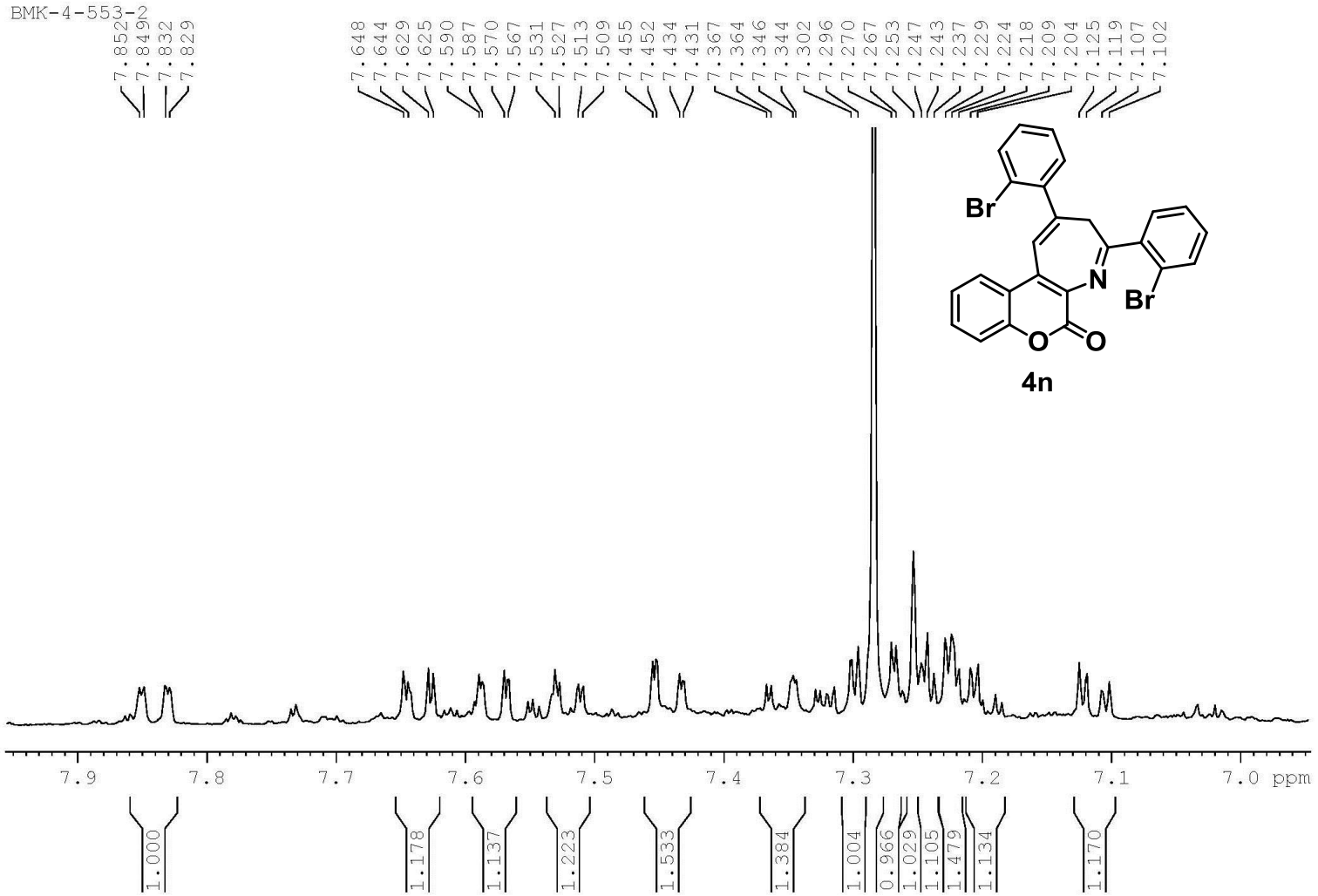
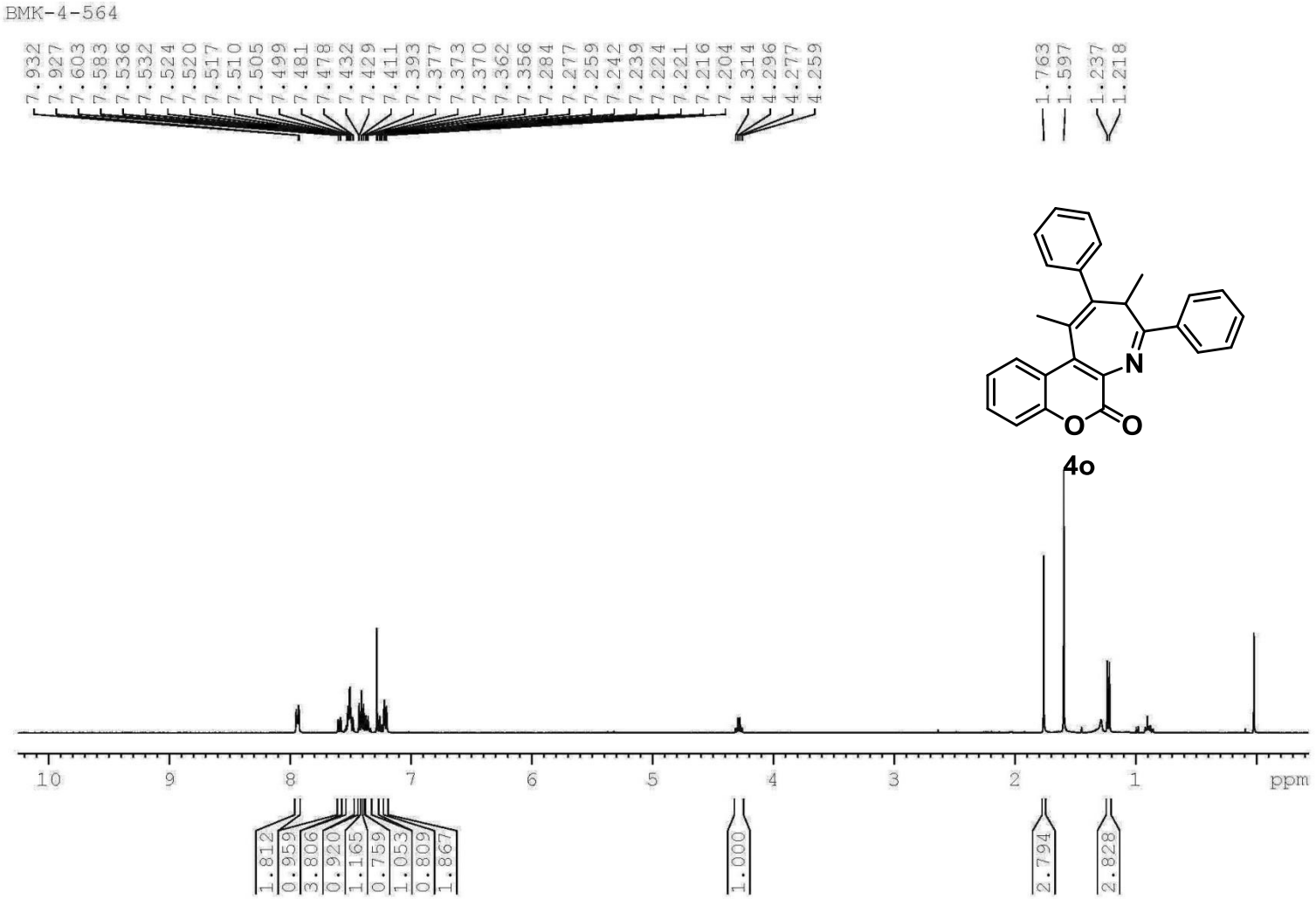
7.629
7.625
7.590
7.587
7.570
7.567
7.531
7.527
7.513
7.509
7.455
7.452
7.434
7.431
7.367
7.364
7.346
7.344
7.302
7.296
7.270
7.267
7.253
7.247
7.243
7.237
7.229
7.224
7.218
7.209
7.204
7.125
7.119
7.107
7.102
4.662
2.622
1.403
1.565
1.000
1.178
1.137
1.223
1.533
1.384
1.004
0.966
1.029
1.105
1.479
1.134
1.170
Br
Br
N
O
O
4n
ppm
0
1
2
3
4
5
6
7
8
9
10


BMK-4-553-2
7.852
7.849
7.832
7.829
7.648
7.644
7.629
7.625
7.590
7.587
7.570
7.567
7.531
7.527
7.513
7.509
7.455
7.452
7.434
7.431
7.367
7.364
7.346
7.344
7.302
7.296
7.270
7.267
7.253
7.247
7.243
7.237
7.229
7.224
7.218
7.209
7.204
7.125
7.119
7.107
7.102
Br
N
Br
O
O
4n
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0 ppm
1.000
1.178
1.137
1.223
1.533
1.384
1.004
0.966
1.029
1.105
1.479
1.134
1.170


BMK-4-564
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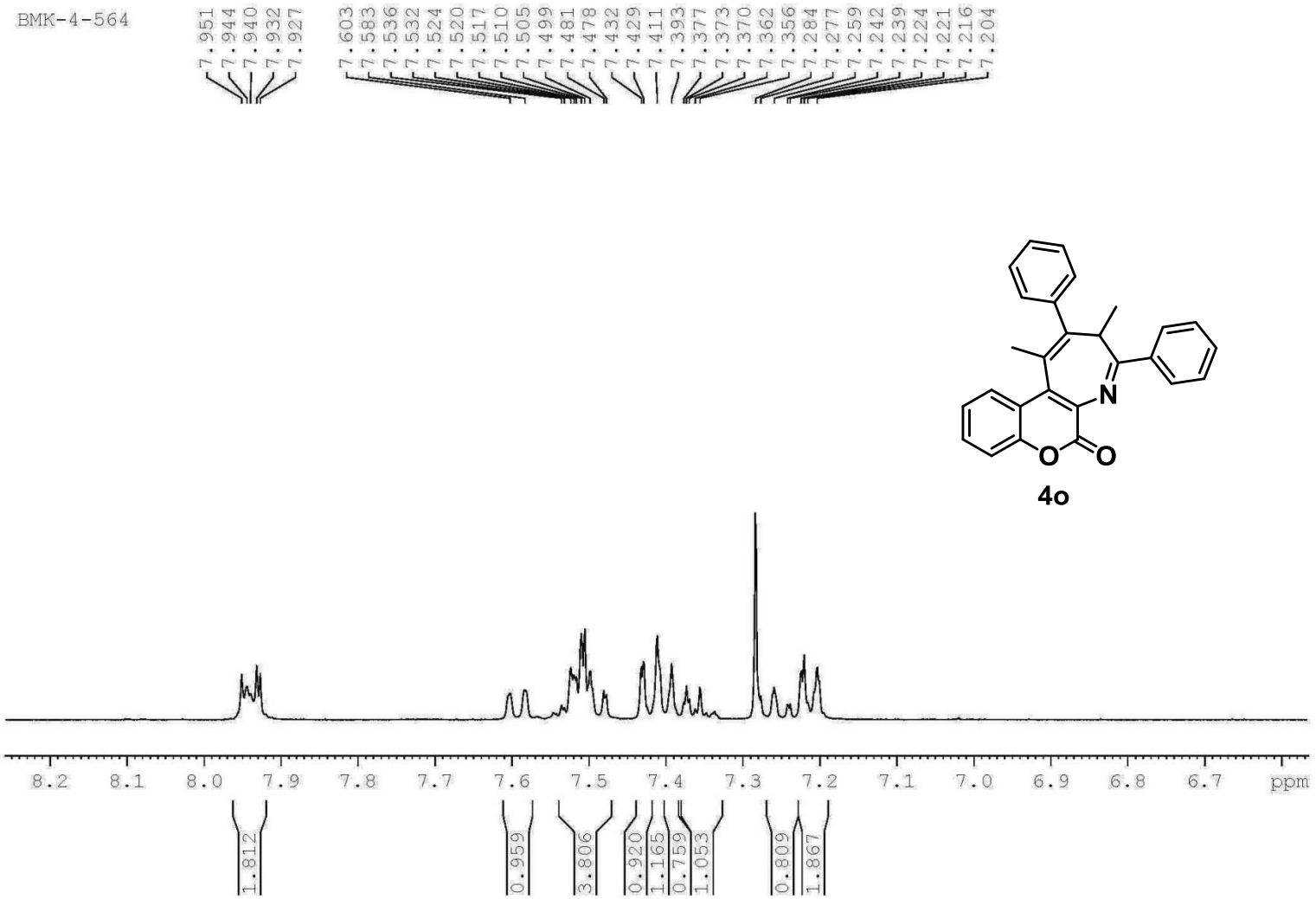
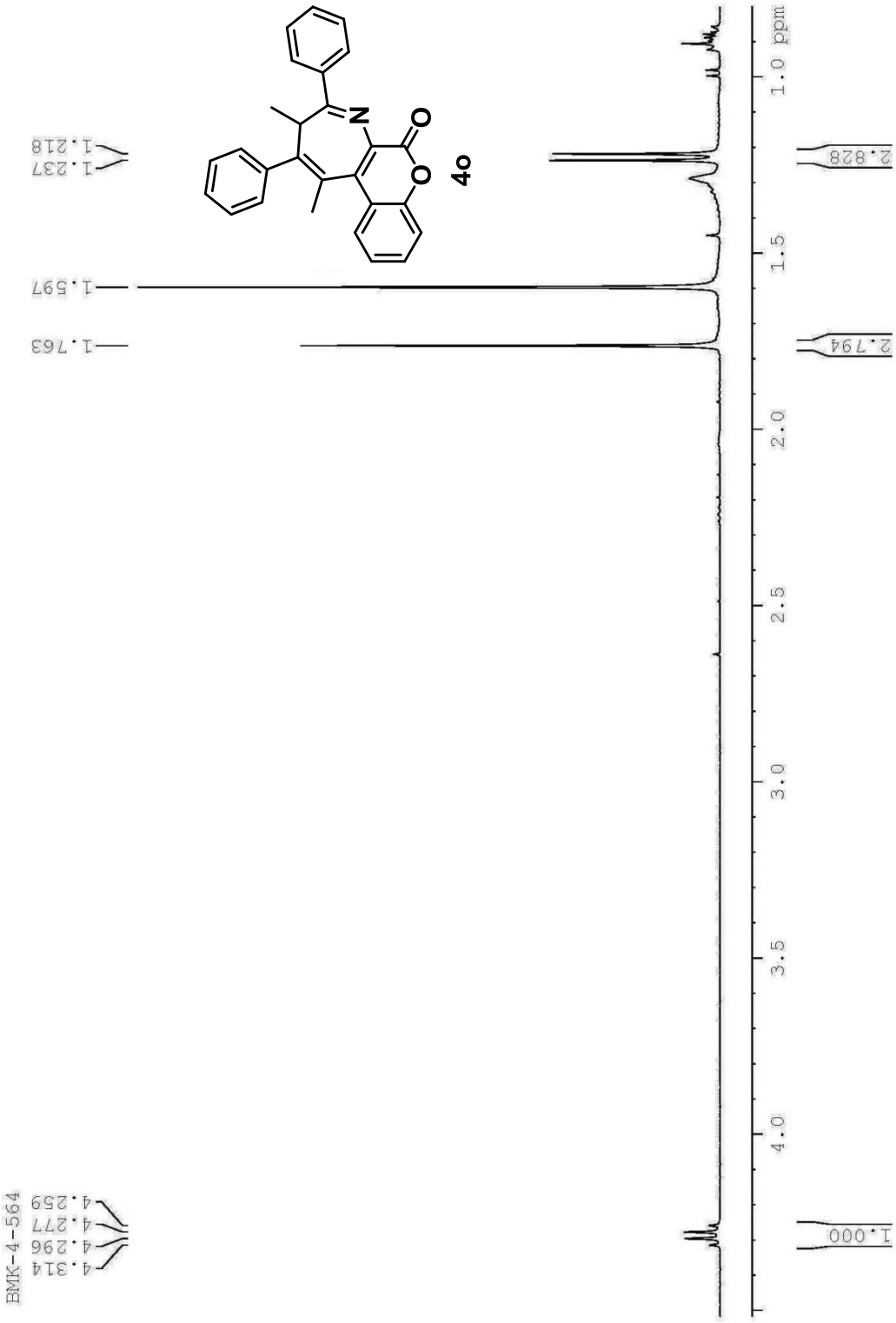
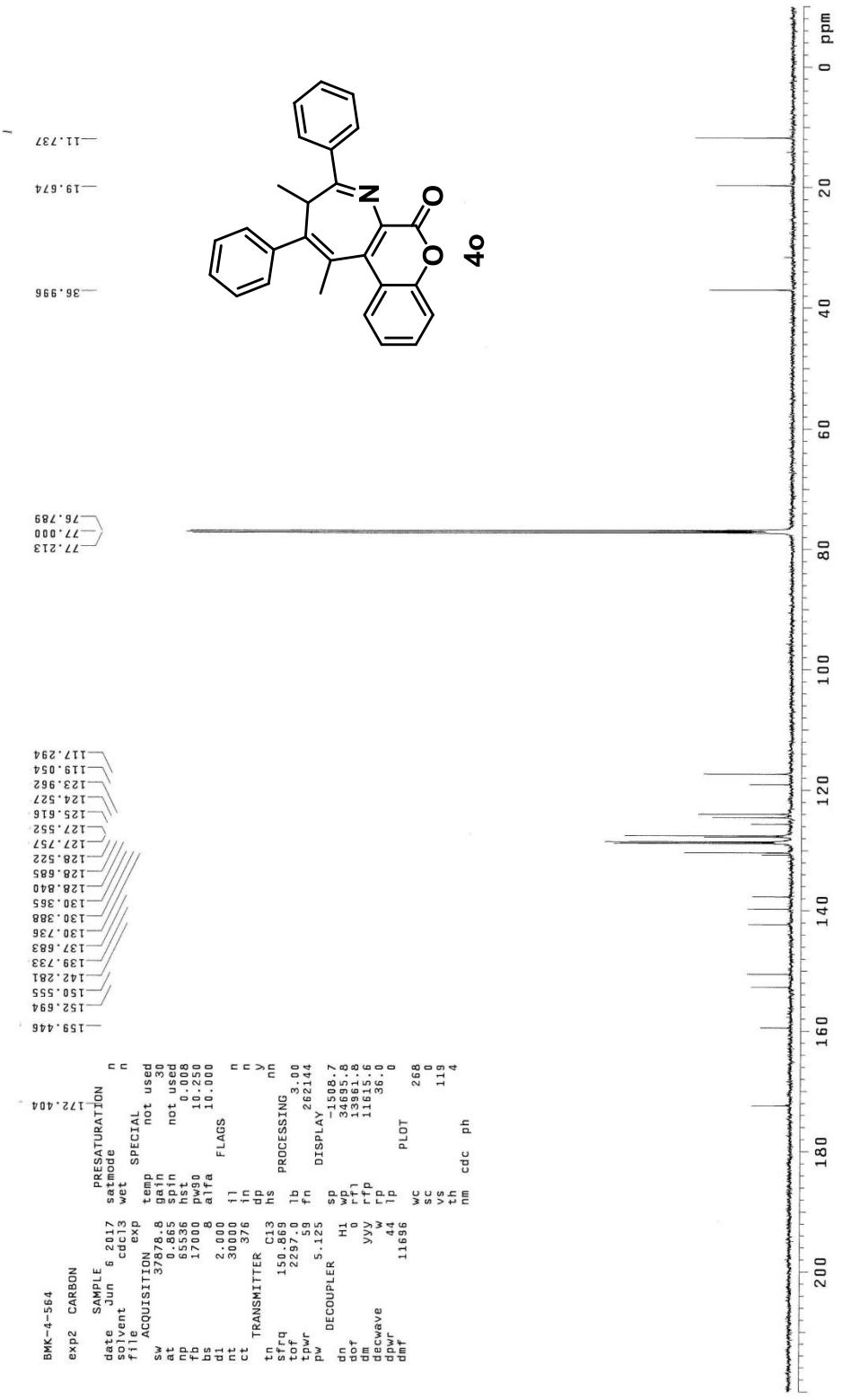
7.927
7.603
7.583
7.536
7.532
7.524
7.520
7.517
7.510
7.505
7.499
7.481
7.478
7.432
7.429
7.411
7.393
7.377
7.373
7.370
7.362
7.356
7.284
7.277
7.259
7.242
7.239
7.224
7.221
7.216
7.204
4.314
4.296
4.277
4.259
1.763
1.597
1.237
1.218
N
O
O
4o
10
9
8
7
6
5
4
3
2
1
ppm
1.812
0.959
3.806
0.920
1.165
0.759
1.053
0.809
1.867
1.000
2.794
2.828


BMK-4-564
7.951
7.944
7.940
7.932
7.927
7.603
7.583
7.536
7.532
7.524
7.520
7.517
7.510
7.505
7.499
7.481
7.478
7.432
7.429
7.411
7.393
7.377
7.373
7.370
7.362
7.356
7.284
7.277
7.259
7.242
7.239
7.224
7.221
7.216
7.204
N
O
O
4o
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
ppm
1.812
0.959
3.806
0.920
1.165
0.759
1.053
0.809
1.867


4o
BMK-4-564
4.259
4.277
4.296
4.314
1.218
1.237
1.597
1.763
1.000
2.794
2.828
4.0
3.5
3.0
2.5
2.0
1.5
1.0 ppm


BMK-4-564
exp2 CARBON
SAMPLE
date Jun 6 2017
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 376
TRANSMITTER
tn C13
sfrq 150.869
tof 2297.0
tpwr 59
pw 5.125
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 44
dmf 11696
PRESATURATION n
satmode n
wet SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.250
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.7
wp 34695.8
rfl 13981.8
rfp 11615.6
rp 36.0
lp 0
PLOT
wc 268
sc 0
vs 119
th 4
nm cdc ph
172.404
159.446
152.694
150.555
142.281
139.733
137.683
130.736
130.388
130.365
128.840
128.685
128.522
127.757
127.552
125.616
124.527
123.962
119.054
117.294
77.213
77.000
76.789
36.996
19.674
11.737
4o
0 ppm