Microwave-Promoted, Metal- and Catalyst-Free Decarboxylative α,β-Difunctionlization of Secondary α-Amino Acids via Pseudo Four-Component Reactions

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Supporting Information

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1. Instrumentation

Melting points were determined on a Mel-Temp melting point apparatus in open capillaries and are uncorrected. MS were performed with a JEOL JMS-SX/SX 102A spectrometer. Single-crystal structures were determined with a Bruker AXS SMART-1000 X-ray single-crystal diffractometer. Microwave reactions were performed using a CEM Discover unit (operating at 110 V, microwave irradiation of 2.45 GHz, maximum microwave output of 300 W) in 50 mL capacity open round-bottom flasks. ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75 MHz on a Varian VXR300 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), and 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. 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.

2.1. Synthesis of compound 1a by classical heating method

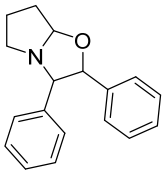
To a mixture of proline (100 mg, 0.87 mmol, 1.0 equiv), 4-hydroxycoumarin (140 mg, 0.87 mmol, 1.0 equiv) in toluene (25 mL) was added freshly distilled benzaldehyde (202 mg, 191 mmol, 2.2 equiv) at room temperature. The resulting mixture was refluxed for 1 h. After completion of the reaction, the solution was concentrated *in vacuo* and the crude product was purified by column chromatography (30% EtOAc/hexanes) to afford the desired colorless solid **1a** (126 mg, yield 35%), along with compound **2**[1] (yellow oil; 35 mg, yield 15%; R_f = 0.75 in 30% EtOAc/hexanes) and compound **3**[2] (white solid; 97 mg, yield 28%; R_f= 0.30 in 40% EtOAc/hexanes).

2.2. Synthesis of compound 1a by microwave irradiation

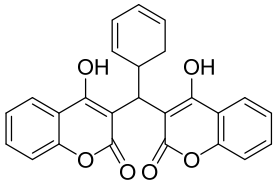
To a round bottom flask (50 mL) was charged with proline (100 mg, 0.87 mmol, 1.0 equiv), 4-hydroxycoumarin (140 mg, 0.87 mmol, 1.0 equiv) and freshly distilled benzaldehyde (0.210 mL, 191 mmol, 2.2 equiv) in toluene (25 mL) at room temperature. The reaction mixture was irradiated in a microwave reactor at 150 ^{o}C (150 W, open vessel conditions) for 10 minutes. After the heat was subsided by

compressed air, the reaction mixture was concentrated *in vacuo*. The crude residue was then purified by column chromatography to afford desired product **1a** as off-white solid (165 mg, yield 45%) along with trace amounts of **2**[1] and **3**.[2]

2. R_f = 0.75 (15% EtOAc/hexanes); yellow oil; 35 mg; yield 15%; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.27–7.25 (m, 10H), 5.30–5.14 (m, 1H), 4.67 (d, *J* = 9.0 Hz, 1H), 3.80 (d, *J* = 8.1 Hz, 1H), 3.15–2.90 (m, 2H), 2.16–2.05 (m, 2H), 1.88–1.82 (m, 2H).



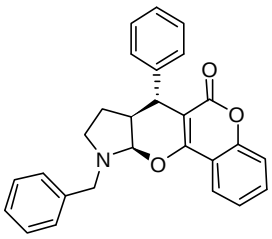
3. R_f = 0.30 (40% EtOAc/hexanes); white solid; 97 mg; yield 28%; ^{1}H NMR (CD_3OD, 300 MHz) δ 7.86 (dd, *J* = 8.4, 1.5 Hz, 2H), 7.45 (td, *J* = 8.4, 1.5 Hz, 2H), 7.21–7.01 (m, 9H), 6.39 (s, 1H).



3. General procedure for the preparations of pyrano[2,3-*b*]pyrrole derivatives 1a–x

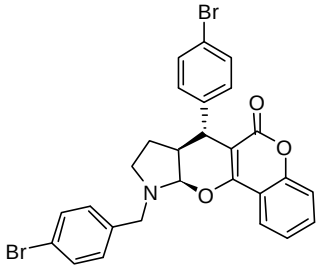
To a 50 mL round bottom flask was charged with proline (0.87 mmol, 1.0 equiv), 1,3-diketone (0.87 mmol, 1.0 equiv) and appropriate benzaldehyde (1.72 mmol, 2.0 equiv) in 1,4-dioxane (15 mL) at room temperature. The reaction mixture was irradiated in a microwave reactor at 200 ^{o}C (200 W, open vessel conditions) for 15 minutes. After the heat was subsided by compressed air, the reaction mixture was concentrated *in vacuo*. The crude residue was purified by column chromatography to afford the desired product.

1a. R_f = 0.65 (30% EtOAc/hexanes); off–white solid; 188 mg; yield 53%; mp 152–154 ^{o}C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.90 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.56 (td, *J* = 8.4, 1.8 Hz, 1H), 7.37–7.28 (m, 8H), 7.22–7.18 (m, 4H), 5.33 (d, *J* = 3.3 Hz, 1H), 4.37, 4.00 (ABq, *J* = 13.8 Hz, 1H each), 4.11 (d, *J* = 1.5 Hz, 1H), 3.15–3.07 (m, 1H), 2.89–2.78 (m, 2H), 2.34–2.25 (m, 1H), 1.83–1.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.0, 160.3, 153.1, 143.5, 138.0, 131.6, 128.8, 128.4, 127.24, 127.18, 126.8, 123.7, 122.8, 116.7, 115.8, 99.3, 94.1, 53.4, 49.0, 45.3,



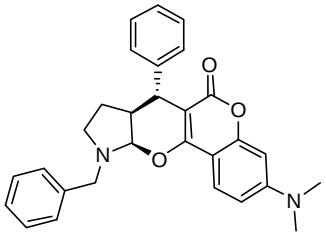
39.4, 27.4; IR ν_{max} (KBr) 3687, 2929, 1707, 1621.5, 1488, 1297, 1153, 1090, 897, 749 cm^{-1}; HRMS (EI) m/z calcd for $C_{27}H_{23}NO_3$ [M^+] 409.1678, found 409.1685.

1b. R_f = 0.65 (30% EtOAc/hexanes); white solid; 245 mg; yield 50%; mp 148–150 ^oC;



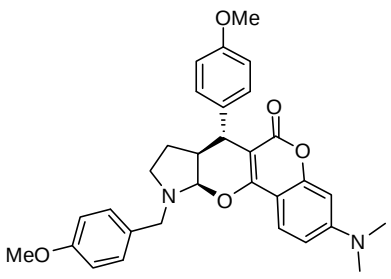
^1H NMR ($CDCl_3$, 300 MHz) δ 7.85 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.58 (td, *J* = 8.1, 1.5 Hz, 1H), 7.48–7.33 (m, 6H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 5.24 (d, *J* = 3.3 Hz, 1H), 4.30, 3.95 (ABq, *J* = 13.8 Hz, 1H each), 4.05 (s, 1H), 3.13–3.05 (m, 1H), 2.84–2.77 (m, 2H), 2.33–2.21 (m, 1H), 1.82–1.60 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.8, 160.2, 153.0, 142.4, 136.9, 131.9, 131.86, 131.6, 130.1, 128.9, 123.8, 122.7, 121.1, 120.6, 116.8, 115.6, 98.9, 93.7, 52.8, 48.9, 45.1, 38.9, 27.3; IR ν_{max} (KBr) 3695, 2914, 1707, 1621, 1487, 1380, 1150, 1011, 897, 763 cm^{-1}; HRMS (EI) m/z calcd for $C_{27}H_{21}Br_2NO_3$ [M^+] 564.9888, found 564.9892.

1c. R_f = 0.45 (30% EtOAc/hexanes); off–white solid; 220 mg; yield 56%; mp 178–180



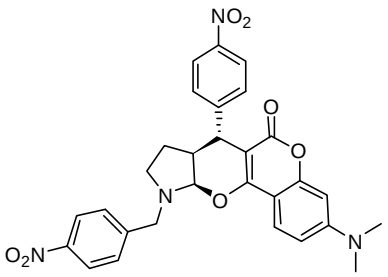
^oC; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.20 (d, *J* = 8.7 Hz, 1H), 7.36–7.16 (m, 10H), 6.78 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.54 (d, *J* = 2.4 Hz, 1H), 5.23 (d, *J* = 3.6 Hz, 1H), 4.34, 3.96 (ABq, *J* = 13.8 Hz, 1H each), 4.05 (s, 1H), 3.13–3.00 (m, 7H), 2.83–2.80 (m, 2H), 2.30–2.10 (m, 1H), 1.84–1.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.9, 161.3, 155.1, 152.9, 144.2, 138.2, 128.6, 128.45, 128.36, 127.2, 127.1, 126.5, 123.4, 108.6, 104.6, 98.0, 94.7, 93.3, 53.4, 49.1, 45.4, 40.2, 39.2, 27.4; IR ν_{max} (KBr) 3696, 2835, 1703, 1611, 1527, 1411, 1367, 1092, 916, 745 cm^{-1}; HRMS (EI) m/z calcd for $C_{29}H_{28}N_2O_3$ [M^+] 452.2100, found 452.2108.

1d. R_f = 0.67 (30% EtOAc/hexanes); pink solid; 282 mg; yield 64%; mp 150–152 ^oC;

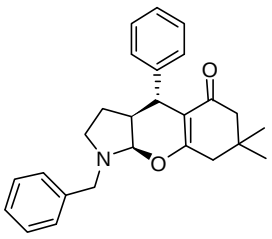


^1H NMR ($CDCl_3$, 300 MHz) δ 7.70 (d, *J* = 9.0 Hz, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 6.68 (dd, *J* = 8.7, 2.7 Hz, 1H), 6.53 (d, *J* = 2.1 Hz, 1H), 5.20 (d, *J* = 3.6 Hz, 1H), 4.25, 3.89 (ABq, *J* = 13.2 Hz, 1H each), 4.00 (s, 1H), 3.79 (s, 3H), 3.74 (s, 3H), 3.06–2.99 (m, 1H), 3.05 (s, 6H), 2.77–2.74 (m, 2H), 2.28–2.20 (m, 1H), 1.80–1.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.9, 161.1, 158.7, 158.1, 155.0, 152.9, 136.3, 130.3, 129.6, 128.2, 123.3, 114.0, 113.7, 108.5, 104.7, 98.0, 95.0, 93.0, 55.18, 55.16, 52.8, 49.1, 45.5, 40.1, 38.4, 27.2; IR ν_{max}

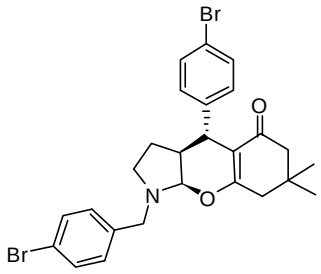
(KBr) 3695, 2897, 1703, 1605, 1512, 1409, 1242, 1124, 1032, 817, 750 cm^{-1}; HRMS (EI) m/z calcd for $C_{31}H_{32}N_2O_5$ [M^+] 512.2311, found 512.2319.

1e. R_f = 0.35 (30% EtOAc/hexanes); brown solid; 280 mg; yield 60%; mp 136–138 

°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.19 (d, *J* = 8.7 Hz, 2H), 8.14 (d, *J* = 9.0 Hz, 2H), 7.65 (d, *J* = 9.0 Hz, 1H), 7.53 (d, *J* = 9.0 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 2H), 7.69 (dd, *J* = 9.0, 2.7 Hz, 1H), 6.53 (d, *J* = 2.4 Hz, 1H), 5.18 (d, *J* = 3.6 Hz, 1H), 4.47, 4.10 (ABq, *J* = 14.7 Hz, 1H each), 4,18 (s, 1H), 3.08 (s, 6H), 2.88–2.80 (m, 2H), 2.34–2.31 (m, 1H), 1.90–1.87 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.6, 161.3, 155.2, 153.2, 151.7, 147.3, 146.8, 145.7, 128.9, 128.2, 127.9, 124.0, 123.7, 123.3, 108.7, 103.9, 97.9, 93.4, 92.6, 52.9, 49.0, 44.8, 40.1, 39.1, 31.5, 29.0, 27.5, 22.6, 14.1; IR ν_{max} (KBr) 3696, 2858, 1703, 1605, 1516, 1343, 1094, 913, 754 cm^{-1}; HRMS (EI) m/z calcd for $C_{29}H_{26}N_4O_7$ [M^+] 542.1801, found 542.1805.

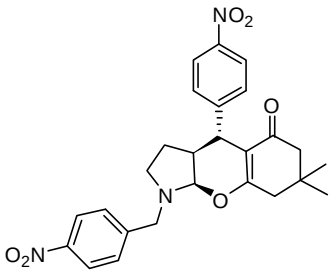
1f. R_f = 0.65 (30% EtOAc/hexanes); colorless; 145 mg; yield 43%; mp 80–82 °C; ^{1}H 

NMR ($CDCl_3$, 300 MHz) δ 8.08 (d, *J* = 8.4 Hz, 1H), 7.43 (t, *J* = 8.1 Hz, 2H), 7.24–7.22 (m, 5H), 7.15–7.13 (m, 3H), 5.35 (d, *J* = 3.6 Hz, 1H), 4.17, 3.80 (ABq, *J* = 13.8 Hz, 1H each), 3.90 (s, 1H), 3.03–2.99 (m, 1H), 2.71–2.61 (m, 1H), 2.43 (s, 2H), 2.27 (s, 2H), 2.19–2.13 (m, 1H), 1.63–1.57 (m, 1H), 1.12 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 197.3, 170.6, 144.8, 138.2, 128.4, 128.3, 128.23, 128.18, 128.1, 127.15, 127.02, 126.9, 126.2, 108.4, 92.8, 53.3, 53.2, 48.9, 45.0, 37.2, 32.1, 27.4; IR ν_{max} (KBr) 2957, 1714, 1651, 1628, 1396, 1218, 1083, 930, 762 cm^{-1}; HRMS (EI) m/z calcd for $C_{26}H_{29}NO_2$ [M^+] 387.2198, found 387.2193.

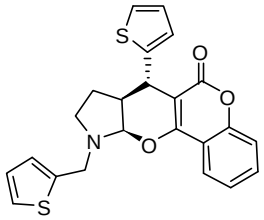
1g. R_f = 0.50 (30% EtOAc/hexanes); brown solid; 210 mg; yield 45%; mp 103–105 

°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.42–7.35 (m, 4H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 4.94 (d, *J* = 3.6 Hz, 1H), 4.10, 3.75 (ABq, *J* = 14.1 Hz, 1H each), 3.84 (s, 1H), 3.02–2.95 (m, 1H), 2.71–2.54 (m, 2H), 2.42–2.35 (m, 2H), 2.49–2.13 (m, 2H), 1.62–1.55 (m, 2H), 1.11 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 196.7, 170.3, 143.9, 137.2, 131.4, 131.3, 131.1, 129.9, 128.8, 128.4, 108.0, 92.3, 52.5, 50.5, 48.9, 44.8, 42.4, 36.7, 32.0, 28.6, 28.1, 27.3; IR ν_{max} (KBr) 2958,

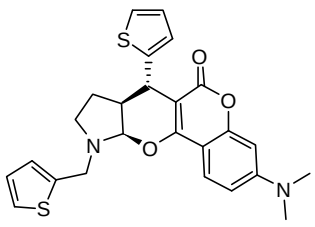
1709, 1620, 1487, 1390, 1144, 1078, 1010, 909, 732 cm^{-1}; HRMS (EI) m/z calcd for $C_{26}H_{27}Br_2NO_2$ [M^+] 543.0409, found 543.0400.

1h. R_f = 0.45 (30% EtOAc/hexanes); pale yellow solid; 190 mg; yield 46%; mp 78–80 

°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.16 (d, *J* = 8.7 Hz, 2H), 8.12 (d, *J* = 8.7 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2 H), 7.31 (d, *J* = 8.7 Hz, 2H), 4.97 (d, *J* = 3.3 Hz, 1H), 4.30, 3.93 (ABq, *J* = 14.7 Hz, 1H each), 4.00 (s, 1H), 3.10–3.01 (m, 1H), 2.80–2.60 (m, 2H), 2.50–2.40 (m, 2H), 2.27 (s, 2H), 1.74–1.63 (m, 2H), 1.13 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 196.9, 170.5, 152.5, 147.1, 146.5, 145.7, 128.8, 128.0, 123.9, 123.6, 107.6, 92.1, 52.7, 50.5, 49.0, 44.4, 42.5, 37.3, 32.2, 28.7, 28.0, 27.5; IR ν_{max} (KBr) 3695, 2927, 1738, 1595, 1515, 1342, 1216, 1079, 929, 739 cm^{-1}; HRMS (EI) m/z calcd for $C_{26}H_{27}N_3O_6$ [M^+] 477.1900, found 477.1904.

1i. R_f = 0.56 (30% EtOAc/hexanes); off–white solid; 245 mg; yield 67%; mp 178–180 

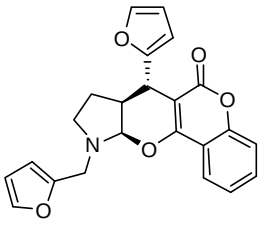
°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.87–7.83 (m, 1H), 7.58–7.52 (m, 1H), 7.35–7.29 (m, 2H), 7.24 (dd, *J* = 5.4, 0.9 Hz, 1H), 7.12 (dd, *J* = 5.4, 1.5 Hz, 1H), 7.02–7.00 (m, 1H), 6.98–6.95 (m, 1H), 6.91–6.86 (m, 2H), 5.50 (d, *J* = 3.3 Hz, 1H), 4.55, 4.22 (ABq, *J* = 14.4 Hz, 1H each), 4.38 (d, *J* = 1.5 Hz, 1H), 3.25–3.17 (m, 1H), 3.06–2.97 (m, 1H), 2.88–2.80 (m, 1H), 2.33–2.21 (m, 1H), 1.78–1.65 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.1, 159.8, 153.3, 147.2, 141.9, 132.1, 127.2, 126.9, 126.1, 125.4, 124.7, 124.0, 124.0, 123.2, 117.0, 116.0, 100.2, 94.1, 49.3, 48.3, 45.4, 34.5, 27.0; IR ν_{max} (KBr) 3039, 1692, 1619, 1571, 1382, 1298, 1152, 1090, 893, 724 cm^{-1}; HRMS (EI) m/z calcd for $C_{23}H_{19}NO_3S_2$ [M^+] 421.0806, found 421.0811.

1j. R_f = 0.45 (30% EtOAc/hexanes); white solid; 305 mg; yield 76%; mp 160–162 °C; 

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.65 (d, *J* = 9.0 Hz, 1H), 7.39 (dd, *J* = 5.1, 1.5 Hz, 1H), 7.10 (dd, *J* = 5.1, 1.5 Hz, 1H), 7.00–6.86 (m, 4H), 6.65 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.51 (d, *J* = 2.4 Hz, 1H), 5.41 (d, *J* = 3.6 Hz, 1H), 4.52, 4.18 (ABq, *J* = 14.1 Hz, 1H each), 4.32 (s, 1H), 3.22–3.14 (m, 1H), 3.06 (s, 6H), 2.99–2.95 (m, 1H), 2.84–2.77 (m, 1H), 2.27–2.18 (m, 1H), 1.80–1.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.8, 160.5, 155.0, 153.0, 148.0, 141.8, 126.8, 126.5, 125.6, 124.9, 124.0, 123.4, 123.3, 108.5, 104.3, 97.8, 95.3, 92.9, 49.0, 48.0, 45.2, 40.1, 33.9, 26.6 ; IR ν_{max} (KBr) 3080, 2377, 1697, 1528, 1413, 1348,

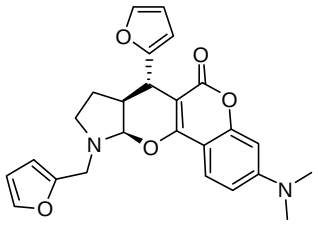
1227, 1092, 910, 820 cm^{-1}; HRMS (EI) m/z calcd for $C_{25}H_{24}N_2O_3S_2$ [M^+] 464.1228, found 464.1223.

1k. R_f = 0.60 (30% EtOAc/hexanes); light brown solid; 207 mg; yield 62%; mp



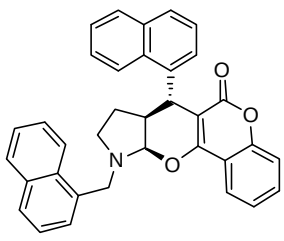
146–148 ^oC; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.81 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.53 (td, *J* = 6.9, 1.5 Hz, 1H), 7.40 (dd, *J* = 2.7, 1.8 Hz, 1H), 7.33–7.26 (m, 3H), 6.37–6.35 (m, 1H), 6.30–6.29 (m, 1H), 6.26–6.24 (m, 1H), 6.88 (d, *J* = 3.3 Hz, 1H), 5.49 (d, *J* = 3.6 Hz, 1H), 4.36, 4.04 (ABq, *J* = 14.4 Hz, 1H each), 4.17 (d, *J* = 1.2 Hz, 1H), 3.20–3.02 (m, 2H), 2.91–2.83 (m, 1H), 2.29–2.17 (m, 1H), 1.76–1.63 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.8, 159.9, 155.0, 152.9, 151.8, 142.2, 141.5, 131.7, 123.6, 122.8, 116.6, 115.8, 110.3, 110.2, 108.0, 105.9, 97.4, 94.4, 48.9, 45.8, 41.6, 32.6, 26.2 ; IR ν_{max} (KBr) 2877, 1698, 1625, 1490, 1401, 1367, 1222, 1092, 889, 728 cm^{-1}; HRMS (EI) m/z calcd for $C_{23}H_{19}NO_5$ [M^+] 389.1263, found 389.1269.

1l. R_f = 0.58 (30% EtOAc/hexanes); brown solid; 252 mg; yield 67%; mp 152–154



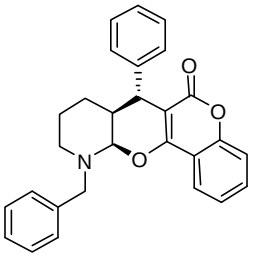
^oC; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.61 (d, *J* = 9.0 Hz, 1H), 7.40–7.39 (m, 1H), 7.28–7.27 (m, 1H), 6.63 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.50 (d, *J* = 2.4 Hz, 1H), 6.35–6.33 (m, 1H), 6.28–6.22 (m, 2H), 6.05–6.03 (m, 1H), 5.39 (d, *J* = 3.9 Hz, 1H), 4.33, 3.99 (ABq, *J* = 14.4 Hz, 1H each), 4.12 (s, 1H), 3.18–3.09 (m, 2H), 3.04 (s, 6H), 2.87–2.79 (m, 1H), 2.26–2.14 (m, 1H), 1.77–1.66 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.7, 160.9, 155.8, 155.0, 153.0, 152.0, 142.1, 141.3, 123.4, 110.2, 108.4, 108.0, 105.6, 105.4, 104.5, 97.8, 97.4, 93.5, 92.8, 49.0, 45.8, 41.5, 40.1, 32.4, 26.2; IR ν_{max} (KBr) 2935, 1698, 1609, 1524, 1411, 1365, 1276, 1006, 904, 816 cm^{-1}; HRMS (EI) m/z calcd for $C_{25}H_{24}N_2O_5$ [M^+] 432.1685, found 432.1688.

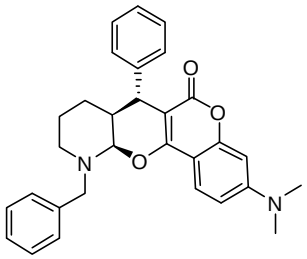
1m. R_f = 0.73 (30% EtOAc/hexanes); white solid; 309 mg; yield 70%; mp 174–176

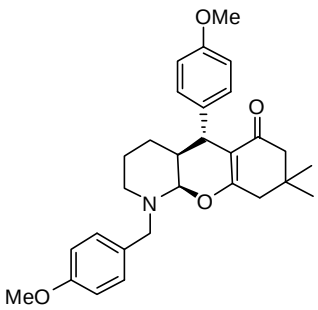


^oC; ^1H NMR ($CDCl_3$, 300 MHz) δ 8.13 (d, *J* = 8.1 Hz, 1H), 8.12–8.04 (m, 1H), 7.98 (dd, *J* = 8.4, 1.8 Hz, 1H),7.87 (d, *J* = 7.8 Hz, 1H), 7.82 (dd, *J* = 7.2, 3 Hz, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.64–7.57 (m, 2H), 7.55–7.48 (m, 2H), 7.45–7.36 (m, 5H), 7.30–7.25 (m, 2H), 7.05 (d, *J* = 7.2 Hz, 1H), 5.16 (d, *J* = 3.6 Hz, 1H), 4.91 (s, 1H), 4.73, 4.41 (ABq, *J* = 13.8 Hz, 1H each), 3.18–3.10 (m, 1H), 3.00–2.93 (m, 1H), 2.43–2.40 (m, 1H), 2.00–1.90 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.8, 160.8, 153.1, 138.5,

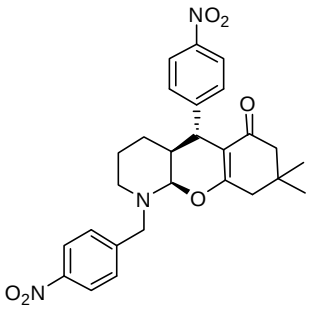
134.3, 133.7, 133.3, 131.9, 131.2, 130.8, 129.1, 128.4, 128.4, 127.6, 127.0, 126.4, 125.9, 125.8, 126.6, 125.4, 125.1, 123.9, 123.7, 123.3, 122.7, 122.6, 116.7, 115.8, 99.0, 93.6, 51.2, 49.4, 43.9, 35.0, 27.1; IR ν_{max} (KBr) 2951, 1699, 1628, 1492, 1364, 1236, 1094, 1018, 804, 777cm^{-1}; HRMS (EI) m/z calcd for $C_{35}H_{27}NO_3$ [M^+] 509.1991, found 509.1997.

1n. R_f = 0.65 (30% EtOAc/hexanes); white solid; 164 mg; yield 45%; mp 136–138  °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.90 (dd, *J* = 8.7, 1.5 Hz, 1H), 7.57 (td, *J* = 7.8, 1.8 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.33–7.26 (m, 8H), 7.24–7.22 (m, 1H), 7.17–7.15 (m, 2H), 5.08 (d, *J* = 2.1 Hz, 1H), 4.13, 4.00 (ABq, *J* = 13.8 Hz, 1H each), 4.00 (s, 1H), 2.76–2.60 (m, 2H), 2.28–2.21 (m, 1H), 1.86-1.79 (m, 1H), 1.72-1.65 (m, 2H), 1.49–1.37 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.7, 160.4, 153.1, 143.2, 137.9, 131.6, 128.6, 128.4, 128.3, 127.5, 127.1, 126.7, 123.6, 122.6, 116.7, 115.8, 99.6, 90.3, 59.0, 44.2, 43.3, 41.0, 25.4, 24.5; IR ν_{max} (KBr) 3695, 2937, 1710, 1629, 1366, 1217, 1105, 1033, 990, 759 cm^{-1}; HRMS (EI) m/z calcd for $C_{28}H_{25}NO_3$ [M^+] 423.1834, found 423.1835.

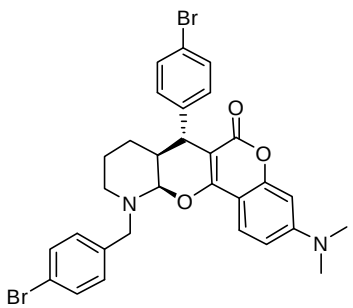
1o. R_f = 0.47 (30% EtOAc/hexanes); white solid; 230 mg; yield 57%; mp 206–208  °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.70 (d, *J* = 9.0 Hz, 1H), 7.30–7.16 (m, 10H), 6.69 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.55 (d, *J* = 2.4 Hz, 1H), 5.01 (d, *J* = 3.0 Hz, 1H), 4.09, 3.97 (ABq, *J* = 14.1 Hz, 1H each), 3.95 (s, 1H), 3.07 (s, 6H), 2.73–2.65 (m, 1H), 2.61–2.56 (m, 1H), 2.23–2.16 (m, 1H), 1.84–1.76 (m, 1H), 1.70–1.64 (m, 2H), 1.47–1.40 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.6, 161.3, 155.1, 152.9, 143.9, 138.2, 128.43, 128.40, 128.2, 127.5, 127.0, 126.5, 123.3, 108.5, 104.6, 97.9, 95.0, 89.5, 59.0, 44.2, 43.1, 41.2, 40.1, 25.4, 24.6; IR ν_{max} (KBr) 3691, 2929, 1739, 1690, 1602, 1404, 1217, 1037, 912, 815 cm^{-1}; HRMS (EI) m/z calcd for $C_{30}H_{30}N_2O_3$ [M^+] 466.2256, found 466.2259.

1p. R_f = 0.43 (30% EtOAc/hexanes); white solid; 180 mg; yield 45%; mp 153–155 

°C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.15 (d, *J* = 8.4 Hz, 2H), 7.02 (d, *J* = 9.0 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 4.77 (d, *J* = 2.4 Hz, 1H), 3.85, 3.73 (ABq, *J* = 13.5 Hz, 1H each), 3.78 (s, 3H), 3.76 (s, 3H), 3.72 (s, 1H), 2.52–2.46 (m, 2H), 2.31, 2.22 (ABq, *J* = 16.2 Hz, 1H each), 2.40 (s, 1H), 2.34 (s, 1H), 2.02–1.95 (m, 1H), 1.72–1.64 (m, 2H), 1.30–1.20 (m, 2H), 1.17 (s, 3H), 1.11 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 197.1, 170.1, 158.6, 157.9, 136.9, 130.5, 129.5, 128.4, 113.7, 113.5, 109.1, 89.2, 58.2, 55.17, 55.15, 50.8, 43.7, 42.4, 41.1, 40.3, 32.3, 29.3, 27.8, 25.4, 24.7; IR ν_{max} (KBr) 3695, 2928, 1739, 1626, 1510, 1381, 1146, 1033, 841, 788 cm^{-1}; HRMS (EI) m/z calcd for $C_{29}H_{35}NO_4$ [M^+] 461.2566, found 461.2557.

1q. R_f = 0.23 (30% EtOAc/hexanes); off–white solid; 200 mg; yield 47%; mp 

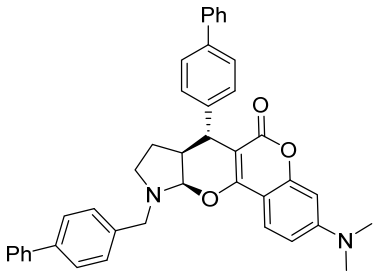
110–112 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.28–7.10 (m, 8H), 4.79 (d, *J* = 2.7 Hz, 1H), 3.92, 3.79 (ABq, *J* = 13.8 Hz, 1H each), 3.82 (s, 1H), 2.54–2.49 (m, 2H), 2.41 (s, 1H), 2.36 (s, 1H), 2.32, 2.23 (ABq, *J* = 16.2 Hz, 1H each), 2.08–2.00 (m, 1H), 1.76–1.68 (m, 2H), 1.34–1.24 (m, 2H), 1.18 (s, 3H), 1.11 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 197.2, 170.3, 144.6, 138.4, 128.31, 128.29, 128.2, 127.5, 126.9, 126.2, 108.8, 89.3, 58.8, 50.8, 43.9, 42.4, 41.2, 40.9, 32.3, 29.3, 27.8, 25.5, 24.6; IR ν_{max} (KBr) 3623, 2943, 1684, 1604, 1508, 1342, 1144, 979, 856, 740 cm^{-1}; HRMS (EI) m/z calcd for $C_{27}H_{29}N_3O_6$ [M^+] 491.2056, found 491.2050.

1r. R_f = 0.45 (30% EtOAc/hexanes); pink solid; 235 mg; yield 44%; mp 218–220 °C; 

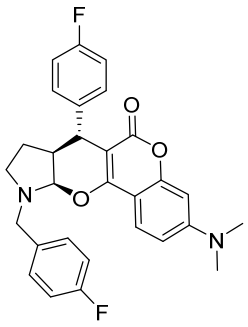
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.63 (d, *J* = 9.0 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 2H), 7.41 (d, *J* = 8.7 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.68 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.54 (d, *J* = 2.4 Hz, 1H), 4.91 (d, *J* = 3.3 Hz, 1H), 4.02, 3.92 (ABq, *J* = 14.1 Hz, 1H each), 3.90 (s, 1H), 3.07 (s, 6H), 2.72–2.63 (m, 1H), 2.58–2.51 (m, 1H), 2.17–2.10 (m, 1H), 1.82–1.74 (m, 1H), 1.70–1.66 (m, 2H), 1.51–1.39 (m,

1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.5, 161.4, 155.1, 153.0, 143.0, 137.2, 131.6, 131.4, 130.1, 129.3, 123.3, 120.9, 120.4, 108.6, 104.3, 97.9, 94.4, 89.3, 58.5, 44.1, 42.6, 41.2, 40.2, 25.2, 24.5; IR ν_{max} (KBr) 3696, 2922, 1705, 1611, 1486, 1208, 1167, 1011, 910, 824 cm^{-1}; HRMS (EI) m/z calcd for $C_{30}H_{28}Br_2N_2O_3$ [M^+] 622.0467, found 622.0464.

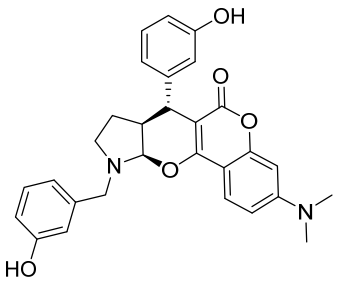
1s. R_f = 0.55 (30% EtOAc/hexanes); pale pink solid; 273 mg; yield 52%; mp 140–142 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.72 (d, *J* = 9.0 Hz, 1H), 7.57–7.48 (m, 8H), 7.45–7.40 (m, 5H), 7.39–7.30 (m, 5H), 6.69 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.54 (d, *J* = 2.4 Hz, 1H), 5.31 (d, *J* = 3.6 Hz, 1H), 4.40, 4.02 (ABq, *J* = 13.8 Hz, 1H each), 4.12 (s, 1H), 3.18–3.06 (m, 1H), 3.07 (s, 6H), 2.90–2.80 (m, 2H), 2.36–2.31 (m, 1H), 1.91–1.76 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 164.0, 161.3, 161.2, 155.1, 153.0, 143.9, 141.0, 140.8, 140.3, 139.6, 137.3, 137.2, 129.0, 128.7, 128.6, 127.7, 127.5, 127.2, 127.0, 123.4, 108.6, 104.6, 98.1, 94.8, 93.2, 93.1, 53.2, 49.3, 45.3, 40.2, 38.8, 27.4; IR ν_{max} (KBr) 3302, 2844, 1702, 1605, 1411, 1369, 1229, 945, 761, 696 cm^{-1}; HRMS (EI) m/z calcd for $C_{41}H_{36}N_2O_3$ [M^+] 604.2726, found 604.2722.



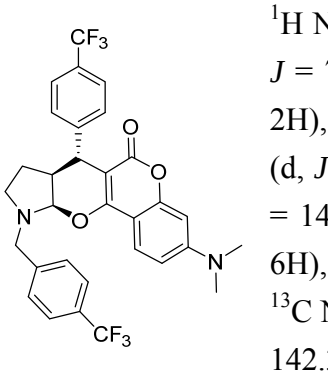
1t. R_f = 0.50 (30% EtOAc/hexanes); off–white solid; 234 mg; yield 55%; mp 130–132 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.68 (d, *J* = 9.0 Hz, 1H), 7.32 (td, *J* = 8.7, 5.7 Hz, 2H), 7.16 (td, *J* = 6.7, 5.4 Hz, 2H), 7.04–6.92 (m, 4H), 6.68 (dd, *J* = 6.3, 2.4 Hz, 1H), 6.56 (d, *J* = 2.1 Hz, 1H), 5.18 (d, *J* = 3.6 Hz, 1H), 4.30, 3.94 (ABq, *J* = 13.5 Hz, 1H each), 4.04 (s, 1H), 3.05 (s, 6H), 3.04–3.00 (m, 1H), 2.81–2.70 (m, 2H), 2.31–2.18 (m, 1H), 1.83–1.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.8, 162.2 (d, $^1J_{CF}$ = 244.1 Hz), 161.6 (d, $^1J_{CF}$ = 243.4 Hz), 161.1, 155.1, 153.0, 139.8, 133.7, 130.1 (d, $^3J_{CF}$ = 7.8 Hz), 128.7 (d, $^3J_{CF}$ = 7.8 Hz), 123.3, 115.4 (d, $^2J_{CF}$ = 21.1 Hz), 115.2 (d, $^2J_{CF}$ = 21.3 Hz), 108.6, 104.4, 98.0, 94.7, 92.8, 52.7, 49.1, 45.4, 40.2, 38.4, 27.2; IR ν_{max} (KBr) 2961, 2918, 1702, 1604, 1506, 1407, 1214, 1011, 835, 753 cm^{-1}; HRMS (EI) m/z calcd for $C_{29}H_{26}F_2N_2O_3$ [M^+] 488.1911, found 488.1915.



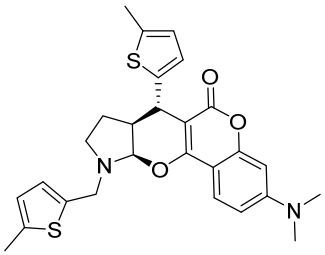
1u. R_f = 0.35 (40% EtOAc/hexanes); off–white solid; 190 mg; yield 45%; mp 140–142 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.68 (d, *J* = 9.0 Hz, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 7.10 (t, *J* = 7.8 Hz, 1H), 6.91–6.85 (m, 2H), 6.74–6.62 (m, 5H), 6.51 (d, *J* = 2.7 Hz, 1H), 5.24 (d, *J* = 3.3 Hz, 1H), 4.28, 3.90 (ABq, *J* = 13.8 Hz, 1H each), 3.99 (s, 1H), 3.06 (s, 6H), 3.04–2.96 (m, 1H), 2.80–2.73 (m, 2H), 2.26–2.17 (m, 1H), 1.83–1.71 (m, 1H); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 163.6, 160.7, 155.1, 153.1, 149.4, 143.7, 129.9, 129.7, 129.4, 129.1, 125.8, 124.5, 123.5, 111.2, 109.8, 108.8, 108.6, 104.1, 97.9, 97.2, 94.5, 92.7, 49.0, 48.4, 45.0, 40.1, 34.3, 26.6; IR ν_{max} (KBr) 2947, 1736, 1597, 1416, 1373, 1275, 1123, 1056, 910, 782 cm^{-1}; HRMS (EI) m/z calcd for $C_{29}H_{28}N_2O_5$ [M^+] 484.1998, found 484.1995.



1v. R_f = 0.35 (40% EtOAc/hexanes); pink solid; 368 mg; yield 72%; mp 146–148 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.65 (d, *J* = 9.0 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 2H), 7.53 (d, *J* = 8.1 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 6.69 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.53 (d, *J* = 2.4 Hz, 1H), 5.20 (d, *J* = 3.6 Hz, 1H), 4.41, 4.04 (ABq, *J* = 14.1 Hz, 1H each), 4.13 (s, 1H), 3.13–3.05 (m, 1H), 3.08 (s, 6H), 2.85–2.75 (m, 2H), 2.36–2.23 (m, 1H), 1.89–1.76 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.8, 161.4, 155.2, 153.1, 148.2, 142.3, 129.4 (q, $^2J_{CF}$ = 32.5 Hz), 128.9 (d, $^2J_{CF}$ = 32.5 Hz), 128.6, 127.6, 125.7 (q, $^1J_{CF}$ = 270.5 Hz), 125.4 (q, $^1J_{CF}$ = 3.5 Hz), 125.2 (q, $^3J_{CF}$ = 3.5 Hz), 124.1 (q, $^1J_{CF}$ = 270.4 Hz), 123.4, 108.7, 104.1, 97.9, 93.8, 92.9, 53.1, 49.1, 45.0, 40.2, 39.0, 27.4; IR ν_{max} (KBr) 2849, 1701, 1602, 1527, 1321, 1120, 1019, 963, 821, 729 cm^{-1}; HRMS (EI) m/z calcd for $C_{31}H_{26}F_6N_2O_3$ [M^+] 588.1848, found 588.1843.

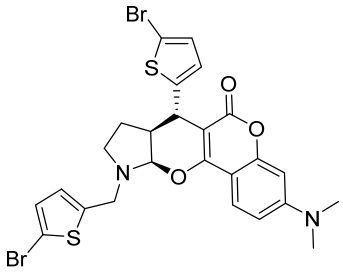


1w. R_f = 0.45 (30% EtOAc/hexanes); off white solid; 283 mg; yield 66%; mp 144–146 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.64 (d, *J* = 9.0 Hz, 1H), 6.77 (d, *J* = 3.6 Hz, 1H), 6.65 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.62 (d, *J* = 4.5 Hz, 1H), 6.59–6.58 (m, 1H), 6.52 (d, *J* = 2.4 Hz, 2H), 5.41 (d, *J* = 3.6 Hz, 1H), 4.43, 4.08 (ABq, *J* = 14.1 Hz, 1H each), 4.22 (s, 1H), 3.20–3.13 (m, 1H), 3.05 (s, 6H), 2.99–2.90 (m, 1H), 2.82–2.73 (m, 1H), 2.46 (s, 3H), 2.38 (s, 3H), 2.28–2.16 (m, 1H), 1.76–1.62 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.8, 160.5, 155.1, 153.0, 145.0, 140.0, 139.3, 137.8, 125.7, 124.8, 124.5, 123.8, 123.5, 108.5, 104.5, 97.9, 95.4, 92.9, 49.1. 48.2, 45.1, 40.2, 34.1, 26.0,



15.4, 15.3; IR ν_{max} (KBr) 2833, 1709, 1614, 1527, 1411, 1356, 1279, 1132, 915, 794 cm^{-1}; HRMS (EI) m/z calcd for $C_{27}H_{28}N_2O_3S_2$ [M^+] 492.6520, found 492.6523.

1x. R_f = 0.40 (40% EtOAc/hexanes); brown solid; 334 mg; yield 62%; mp 82–84 °C;

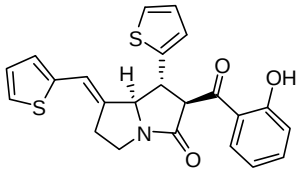


^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.59 (d, *J* = 8.7 Hz, 1H), 6.87 (d, *J* = 3.6 Hz, 1H), 6.84 (d, *J* = 3.9 Hz, 1H), 6.76 (d, *J* = 3.9 Hz, 1H), 6.66 (d, *J* = 2.7 Hz, 1H), 6.40–6.23 (m, 1H), 6.51 (d, *J* = 2.4 Hz, 1H), 5.36 (d, *J* = 3.9 Hz, 1H), 4.34, 4.10 (ABq, *J* = 14.4 Hz, 1H each), 4.23 (s, 1H), 3.22–3.14 (m, 1H), 3.06 (s, 6H), 2.98–2.88 (m, 1H), 2.83–2.74 (m, 1H), 2.30–2.17 (m, 1H), 1.76–1.68 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 163.7, 160.7, 155.4, 153.2, 149.5, 143.7, 129.7, 129.3, 125.9, 124.5, 123.4, 111.6, 109.8, 108.6, 104.2, 97.9, 94.5, 92.6, 49.0, 48.4, 45.0, 40.2, 34.3, 26.6; IR ν_{max} (KBr) 2920, 1702, 1604, 1525, 1411, 1276, 1154, 964, 890, 793 cm^{-1}; HRMS (EI) m/z calcd for $C_{25}H_{22}Br_2N_2O_3S_2$ [M^+] 619.9439, found 619.9435.

4. General procedure for the preparations of pyrrolizinones derivatives 4a–e

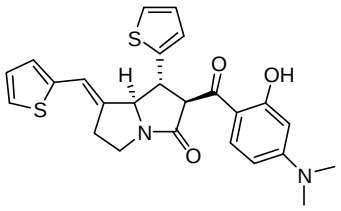
To a mixture of proline (0.87 mmol, 1.0 equiv), 1,3-diketone (0.87 mmol, 1.0 equiv) in 1,4-dioxane (10 mL) was added carbaldehyde (3.47 mmol, 4.0 equiv, thiophene or furan carbaldehyde) at room temperature. The reaction mixture was heated in a microwave reactor at 200 °C (250 W, open vessel conditions) for 15 minutes. After the heat was subsided by compressed air, the reaction mixture was concentrated *in vacuo*. The crude residue was purified by column chromatography to afford pyrrolizinone derivative as a major product, along with pyrano[2,3-*b*]pyrrole derivative as a minor (10–15% yield) product.

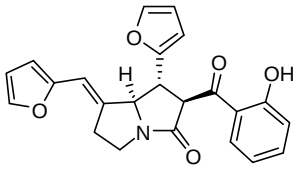
4a. R_f = 0.30 (40% EtOAc/hexanes); white solid; 237 mg; yield 65%; mp 178–180 °C;

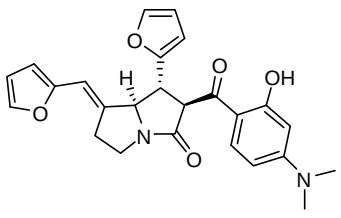


^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.99 (s, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.47 (td, *J* = 6.9, 1.5 Hz, 1H), 7.32–7.25 (m, 2H), 7.05–6.88 (m, 6H), 6.55 (d, *J* = 2.1 Hz, 1H), 4.94 (d, *J* = 11.1 Hz, 1H), 4.79 (d, *J* = 8.7 Hz, 1H), 4.51 (dd, *J* = 11.1, 8.7 Hz, 1H), 4.21–4.13 (m, 1H), 3.28–3.18 (m, 1H), 3.01–2.96 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 199.1, 167.9, 162.8, 141.6, 140.3, 137.8, 137.0, 131.6, 127.4, 127.2, 127.1, 126.0, 125.8, 124.9, 120.0, 119.0, 118.1, 116.9, 68.1, 61.6, 46.6, 42.0,

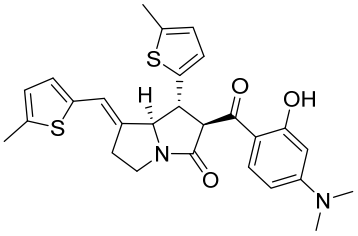
31.8; IR ν_{max} (KBr) 3076, 1698, 1626, 1414, 1299, 1216, 984, 850, 751, 726 cm^{-1}; HRMS (EI) m/z calcd for $C_{23}H_{19}NO_3S_2$ [M^+] 421.0806, found 421.0801.

4b. R_f = 0.35 (40% EtOAc/hexanes); brown solid; 210 mg; yield 52%; mp 242–244  °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 12.69 (s, 1H), 7.49 (d, *J* = 9.6 Hz, 1H), 7.29 (d, *J* = 5.4 Hz, 1H), 7.24 (dd, *J* = 5.4, 1.2 Hz, 1H), 7.03–6.91 (m, 4H), 6.55 (q, *J* = 1.8 Hz, 1H), 6.19 (dd, *J* = 9.3, 2.7 Hz, 1H), 6.04 (d, *J* = 2.4 Hz, 1H), 4.80–4.75 (m, 2H), 4.48 (dd, *J* = 11.1, 8.4 Hz, 1H), 4.22–4.14 (m, 1H), 3.26–3.16 (m, 1H), 3.03 (s, 6H), 3.00–2.96 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 194.4, 169.0, 165.4, 156.2, 142.3, 142.5, 138.4, 138.5, 132.9, 127.4, 127.1, 127.1, 125.8, 125.7, 124.7, 116.7, 110.8, 104.3, 97.4, 68.3, 60.7, 46.8, 42.0, 39.9, 31.9; IR ν_{max} (KBr) 3090, 1695, 1628, 1544, 1363, 1250, 1148, 1011, 876, 740 cm^{-1}; HRMS (EI) m/z calcd for $C_{25}H_{24}N_2O_3S_2$ [M^+] 464.1228, found 464.1225.

4c. R_f = 0.35 (30% EtOAc/hexanes); brown solid; 169 mg; yield 50%; mp 157–159  °C; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 11.97 (s, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.41 (s, 1H), 6.96 (d, *J* = 7.8 Hz, 1H), 6.92 (t, *J* = 8.4 Hz, 1H), 6.43–6.42 (m, 1H), 6.34–6.33 (m, 1H), 6.28–6.27 (m, 1H), 6.25–6.24 (m, 3H), 5.10 (d, *J* = 10.8 Hz, 1H), 4.76 (d, *J* = 7.8 Hz, 1H), 4.27 (dd, *J* = 10.8, 8.4 Hz, 1H), 4.15–4.11 (m, 1H), 3.19–3.16 (m, 1H), 3.08–3.04 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 199.3, 168.8, 162.9, 152.4, 151.3, 142.7, 142.1, 138.3, 137.0, 131.6, 119.9, 119.1, 118.3, 112.1, 111.4, 110.6, 109.3, 107.8, 65.0, 57.6, 44.5, 42.0, 31.9; IR ν_{max} (KBr) 3087, 2929, 1707, 1621, 1488, 1297, 1153, 1090, 897, 749 cm^{-1}; HRMS (EI) m/z calcd for $C_{23}H_{19}NO_5$ [M^+] 389.1263, found 389.1258.

4d. R_f = 0.28 (30% EtOAc/hexanes); brown solid; 215 mg; yield 58%; mp 176–178  °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 12.68 (s, 1H), 7.52 (d, *J* = 9.0 Hz, 1H), 7.40 (s, 1H), 6.42–6.40 (m, 1H), 6.31–6.28 (m, 2H), 6.22–6.19 (m, 4H), 6.04 (d, *J* = 2.7 Hz, 1H), 4.94 (d, *J* = 11.1 Hz, 1H), 4.73 (d, *J* = 8.4 Hz, 1H), 4.28 (dd, *J* = 11.1, 8.4 Hz, 1H), 4.12–4.10 (m, 1H), 3.20–2.96 (m, 3H), 3.02 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 194.6, 169.2, 165.4, 156.2, 152.4, 151.8, 142.4, 141.9, 138.8, 132.8, 111.8, 111.3, 111.0, 110.5, 109.0, 107.5, 104.3, 97.4, 65.2, 56.6, 44.6, 41.9, 39.9, 32.0; IR ν_{max} (KBr) 2902, 1699, 1627, 1549, 1530, 1383, 1149, 1014, 824, 759 cm^{-1}; HRMS (EI) m/z calcd for $C_{25}H_{24}N_2O_5$ [M^+] 432.1685, found 432.1690.

4e. R_f = 0.35 (40% EtOAc/hexanes); light yellow solid; 226 mg; yield 53%; mp 204–206 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 12.71 (s, 1H), 7.51 (d, *J* = 9.3 Hz, 1H), 6.77 (d, *J* = 3.3 Hz, 1H), 6.71 (d, *J* = 3.6 Hz, 1H), 6.66 (d, *J* = 3.6 Hz, 1H), 6.48 (d, *J* = 1.5 Hz, 1H), 6.58 (d, *J* = 3.3 Hz, 1H), 6.21 (dd, *J* = 6.6, 2.4 Hz, 1H), 6.05 (d, *J* = 2.4 Hz, 1H), 4.76–4.67 (m, 2H), 4.46 (dd, *J* = 10.8, 8.1 Hz, 1H), 4.18–4.11 (m, 1H), 3.30–3.13 (m, 1H), 3.03 (s, 6H), 2.95–2.93 (m, 2H), 2.48 (s, 3H), 2.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 194.6, 169.1, 165.4, 156.1, 140.5, 139.9, 139.2, 138.6, 136.9, 132.9, 127.1, 125.7, 125.31, 125.26, 116.9, 110.9, 104.3, 97.5, 68.1, 60.5, 47.1, 41.9, 39.9, 31.8, 15.4, 15.3; IR ν_{max} (KBr) 2918, 1698, 1599, 1362, 1240, 1151, 1046, 791, 715, 727 cm^{-1}; HRMS (EI) m/z calcd for $C_{27}H_{28}N_2O_3S_2$ [M^+] 492.1541, found 492.1544.

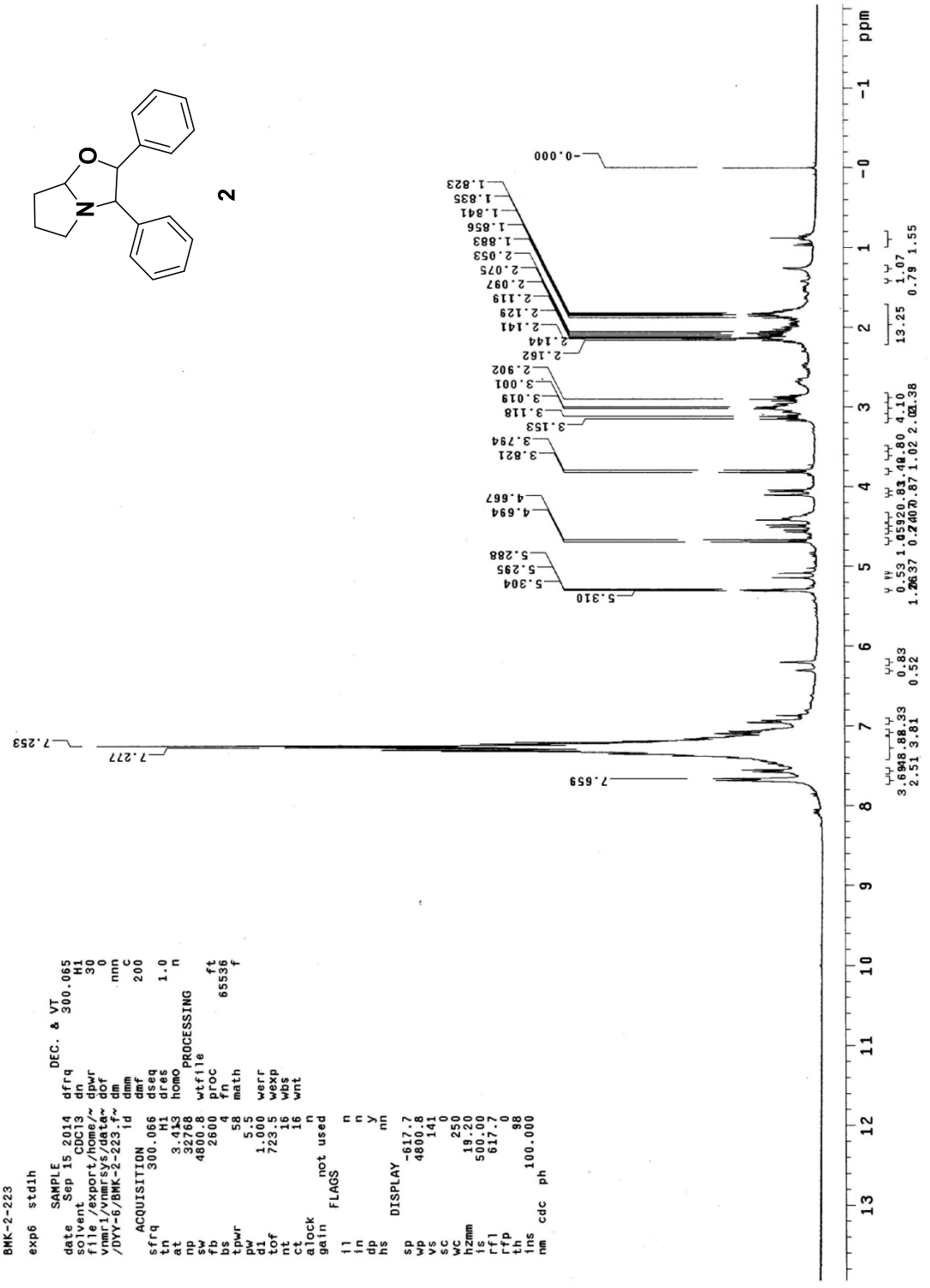


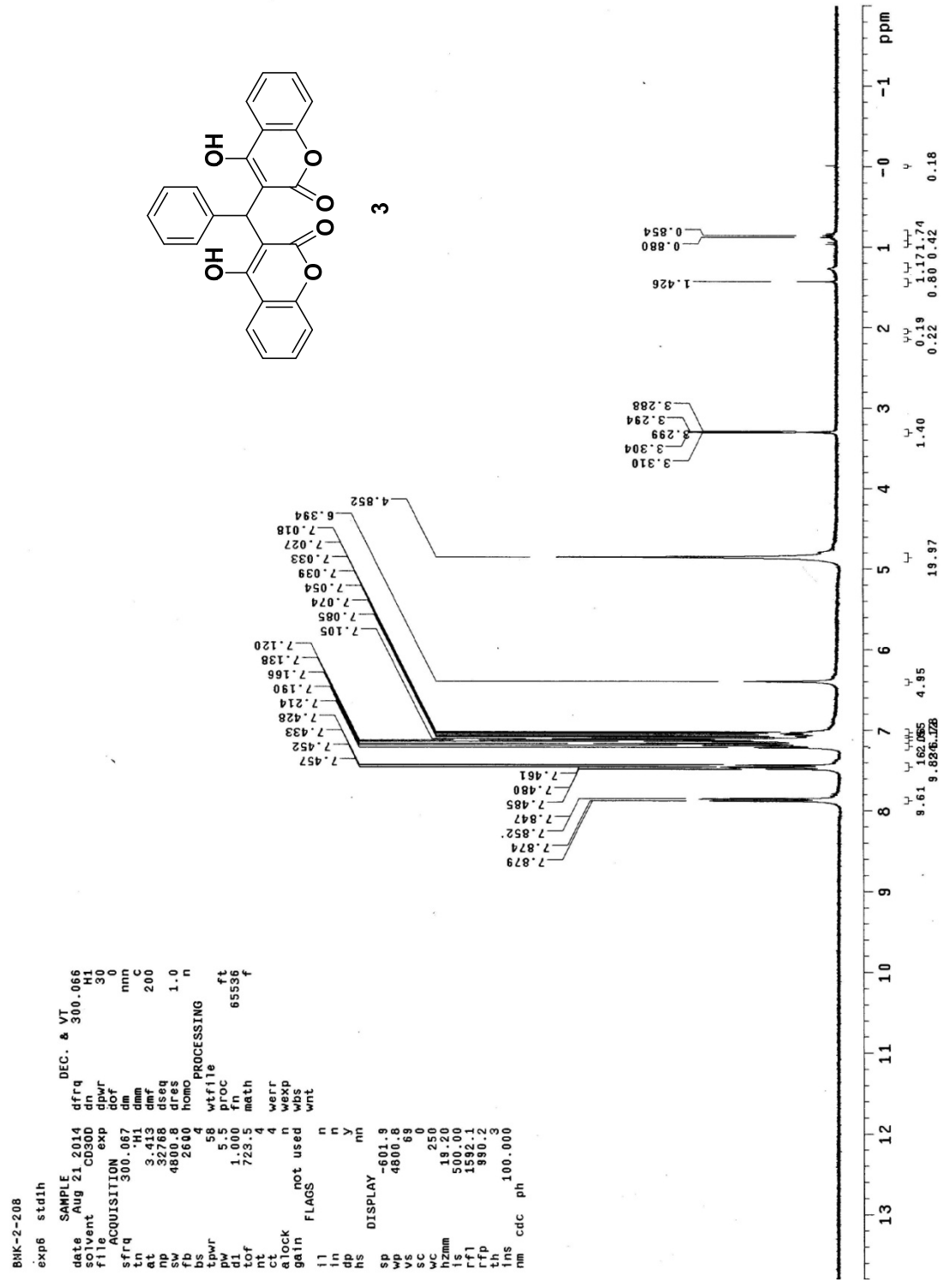
Crystallographic data (excluding structure factors) for **1a**, **1p**, and **4a** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-1018971, -1018970, and -1019925, respectively. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

4. References

1. (a) Orsini, F.; Pelizzoni, F.; Forte, M.; Destro, R.; Gariboldi, P. *Tetrahedron* **1988**, *44*, 519. (b) Zhang, C.; Seidel, D. *J. Am. Chem. Soc.* **2010**, *132*, 1798.
2. (a) Manoj, R. Z.; Mustafa, J. R.; Sachin,D. G.; Veerababurao, K.; Donala, J.; Kuo, C-W.; Ram, A.; Yao, C-F. *J. Org. Chem.*, **2012**, *77*, 6495. (b) Atul, K.; Maneesh, K. G.; Mukesh, K. *Tetrahedron Lett.* **2011**, *52*, 4521.

5. Copies of ^{1}H and ^{13}C NMR spectra of synthesized compounds

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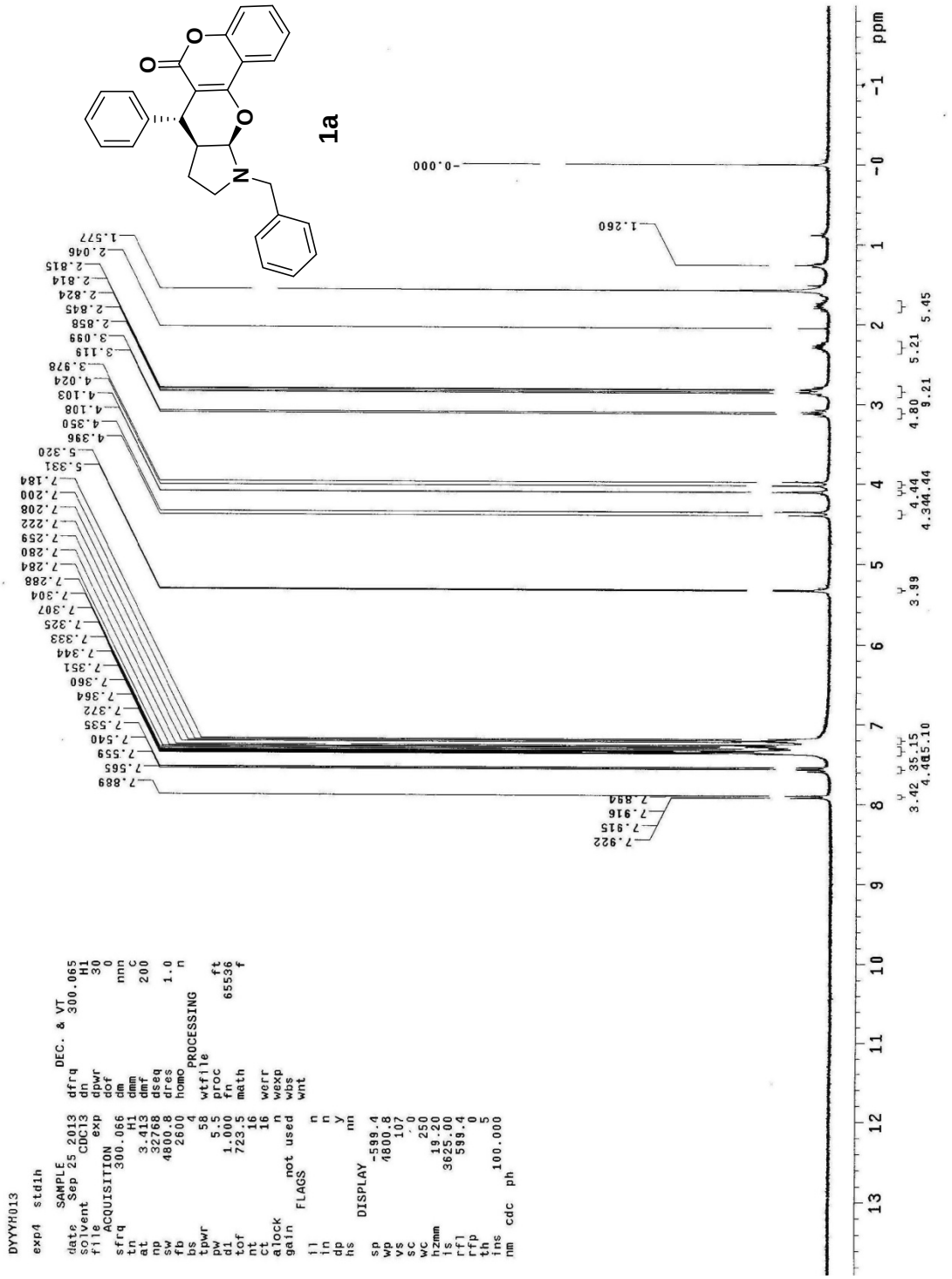
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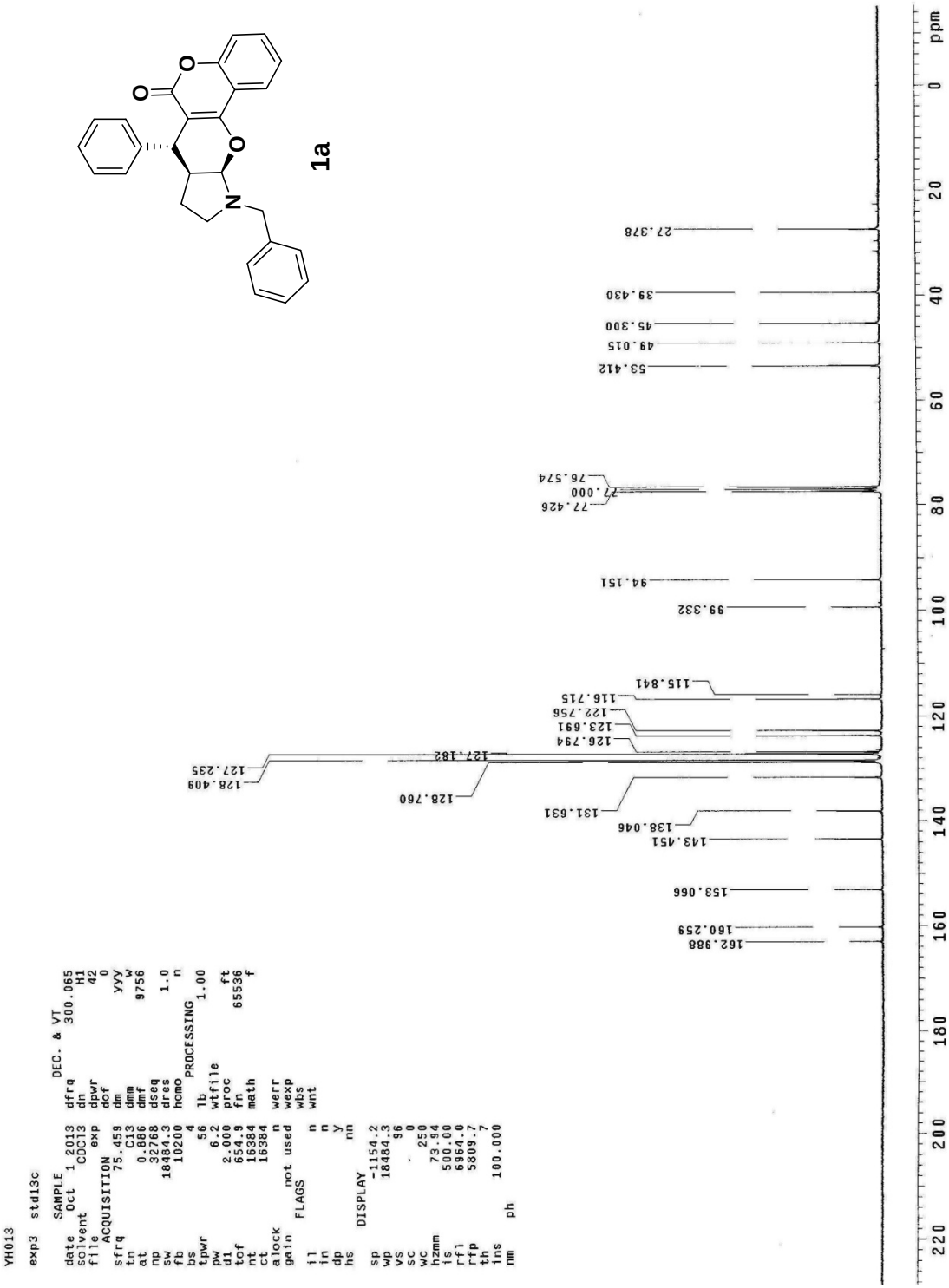
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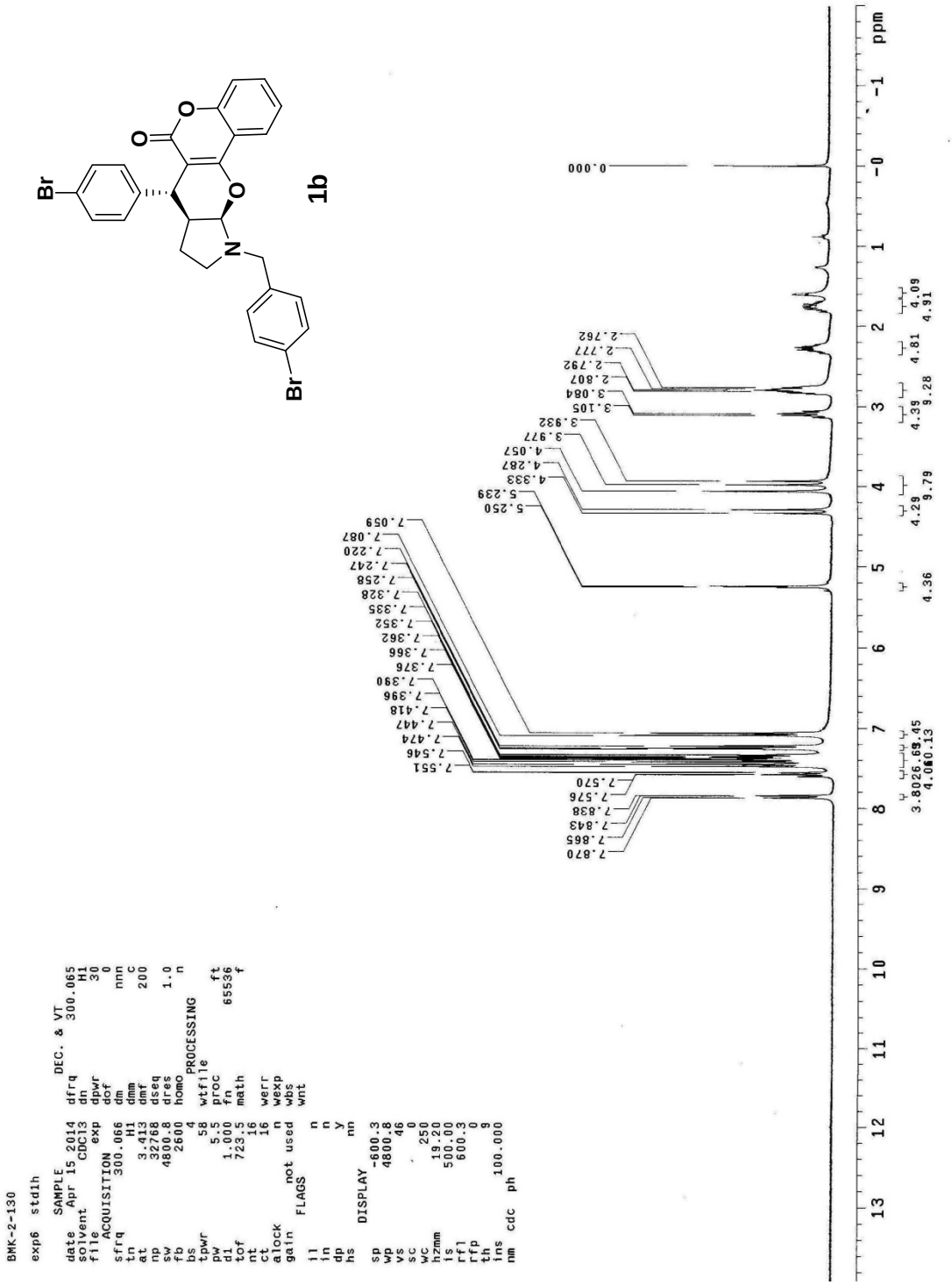
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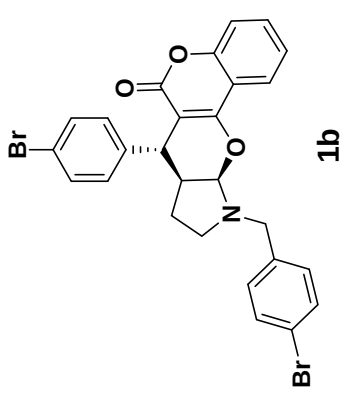
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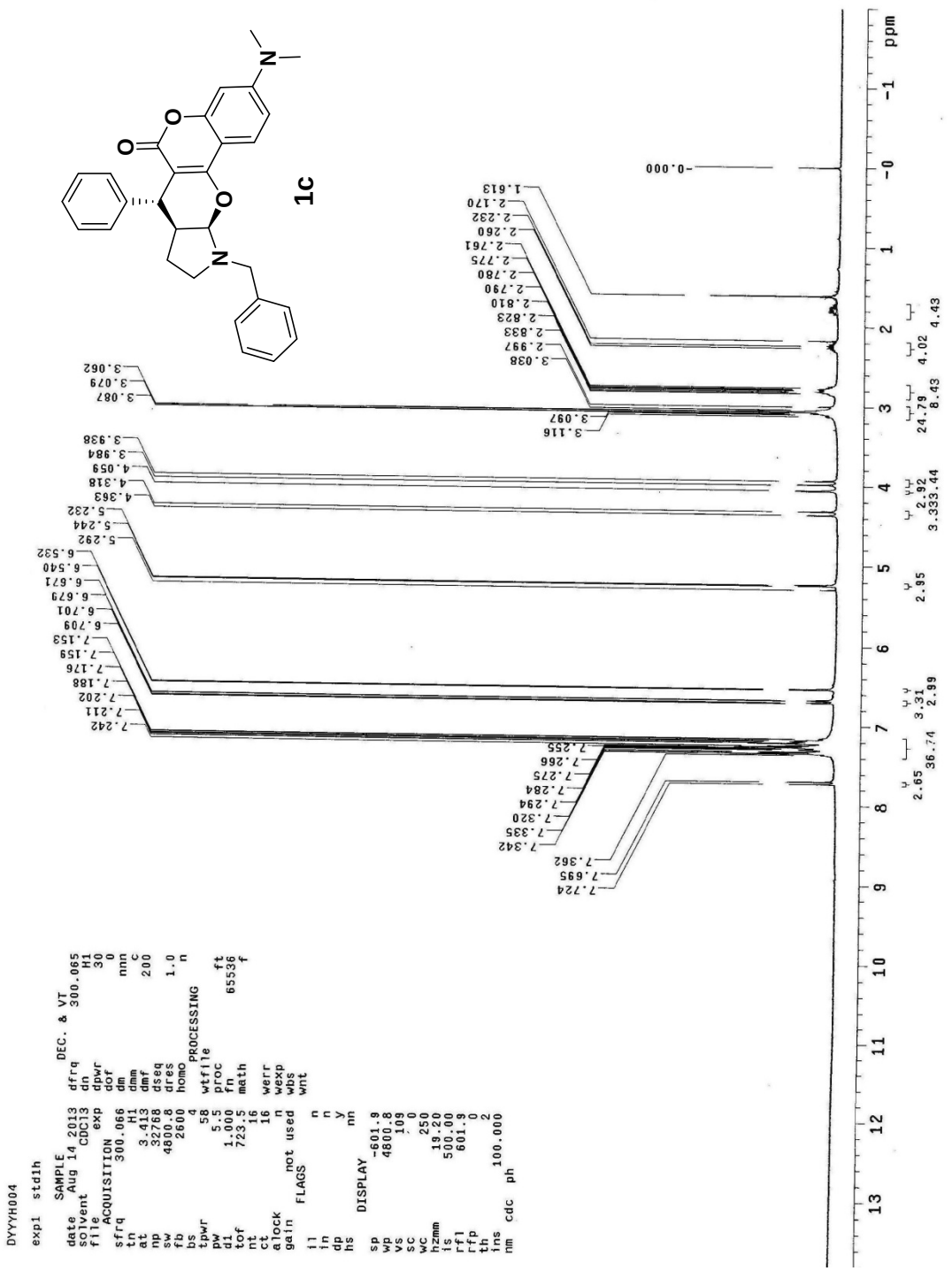
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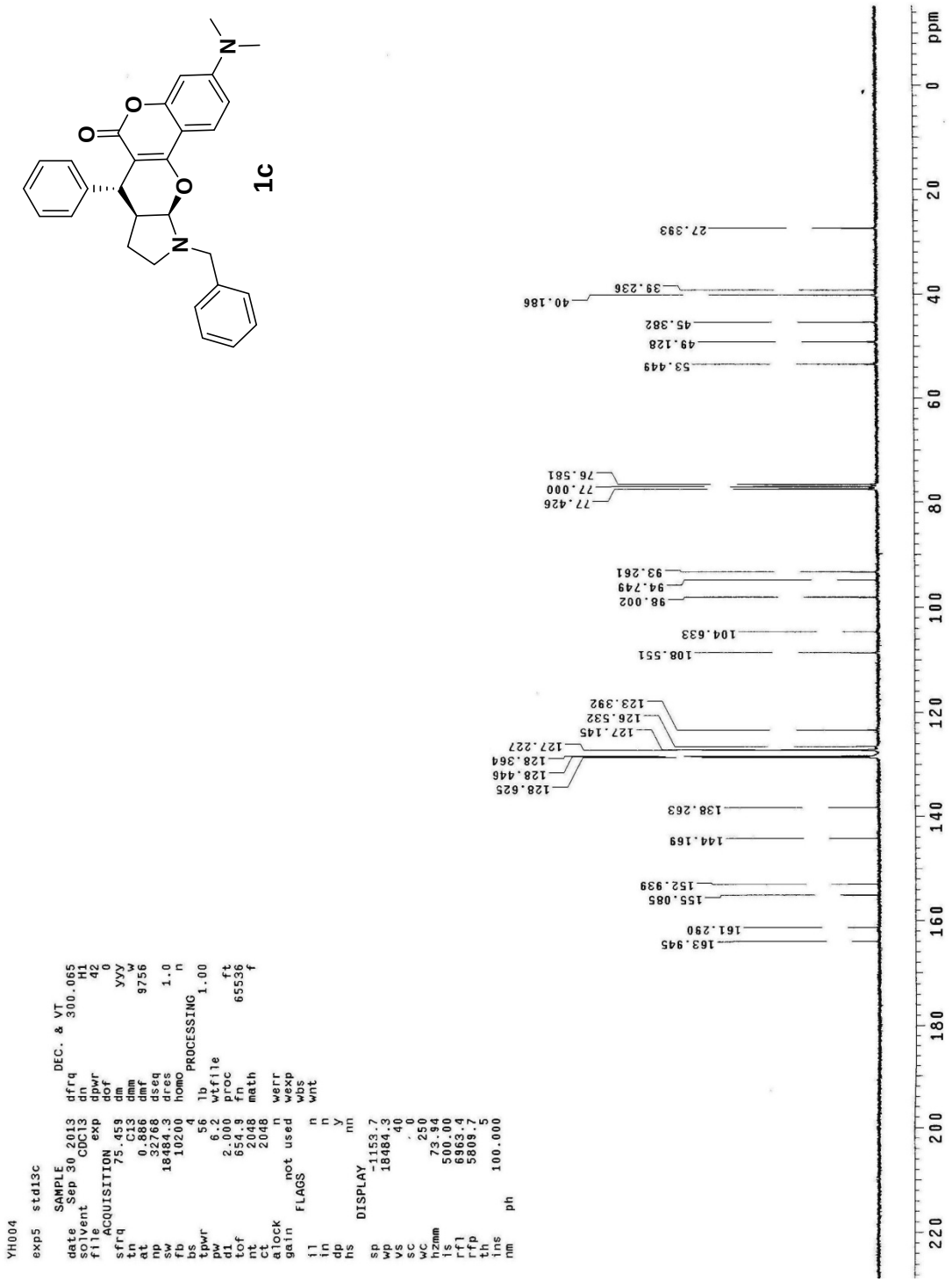
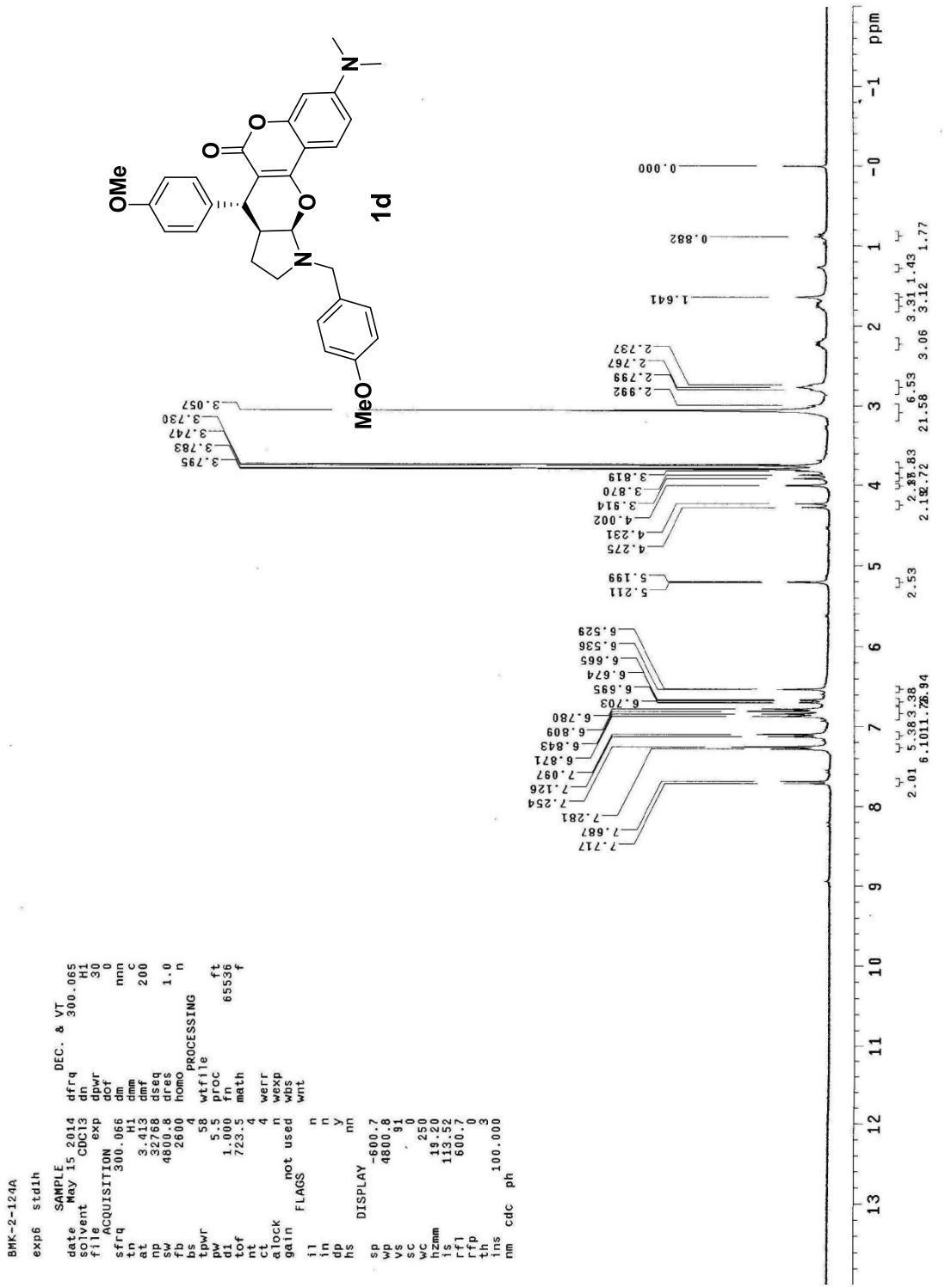
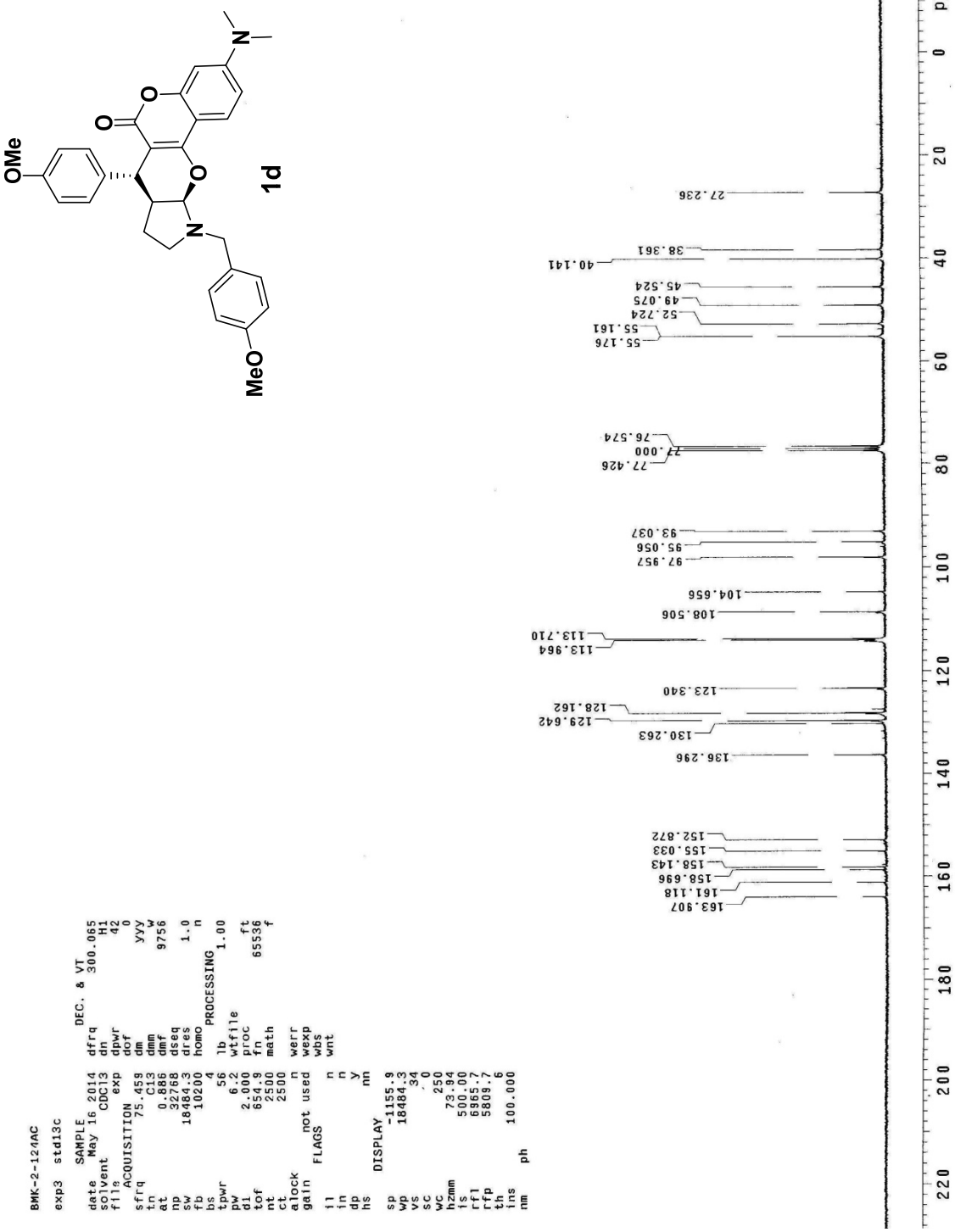
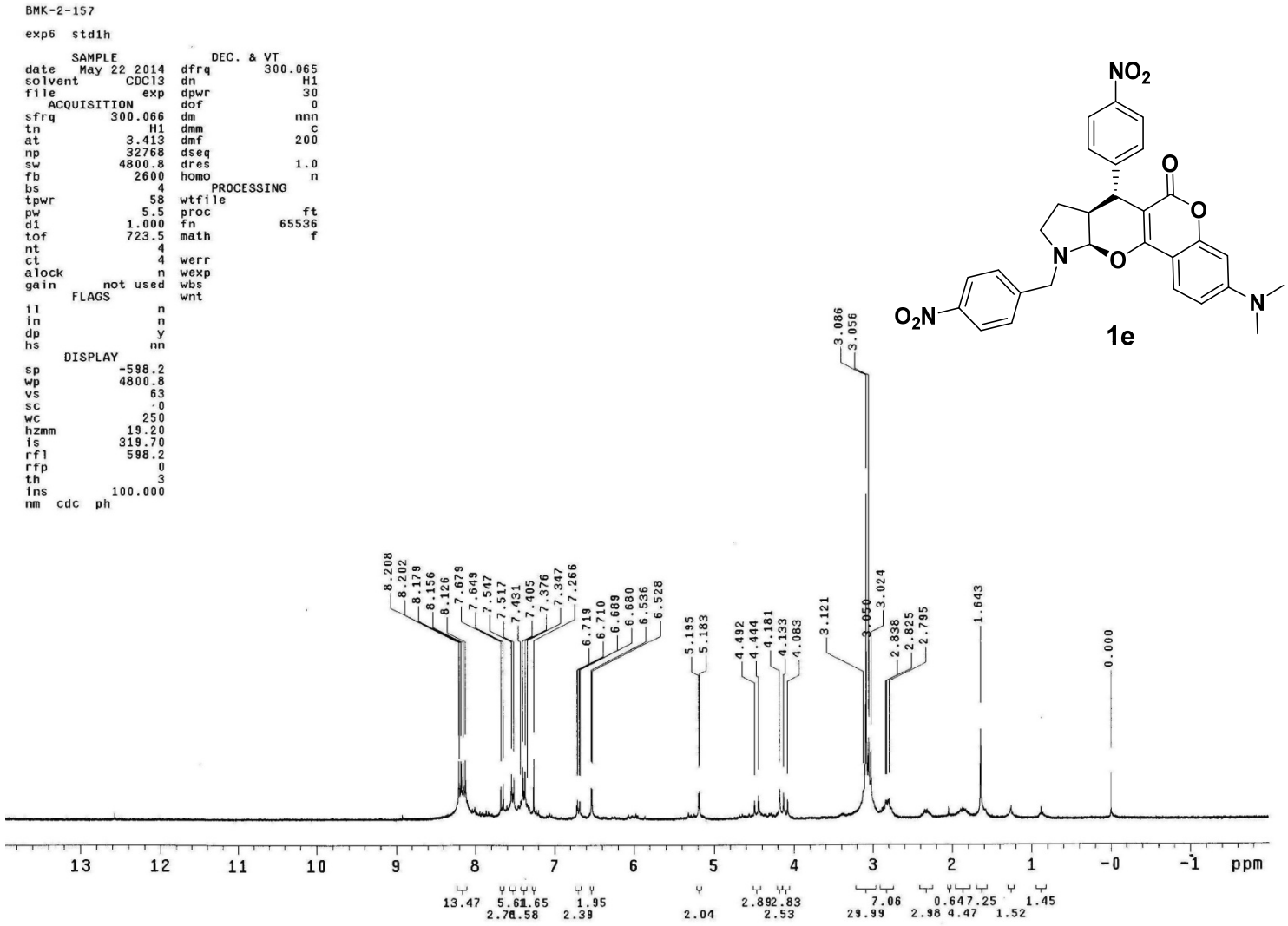
werr
wexp
wbs
wnt
ppm
2.65
36.74
3.31
2.99
2.95
2.92
3.333.44
24.79
8.43
4.02
4.43

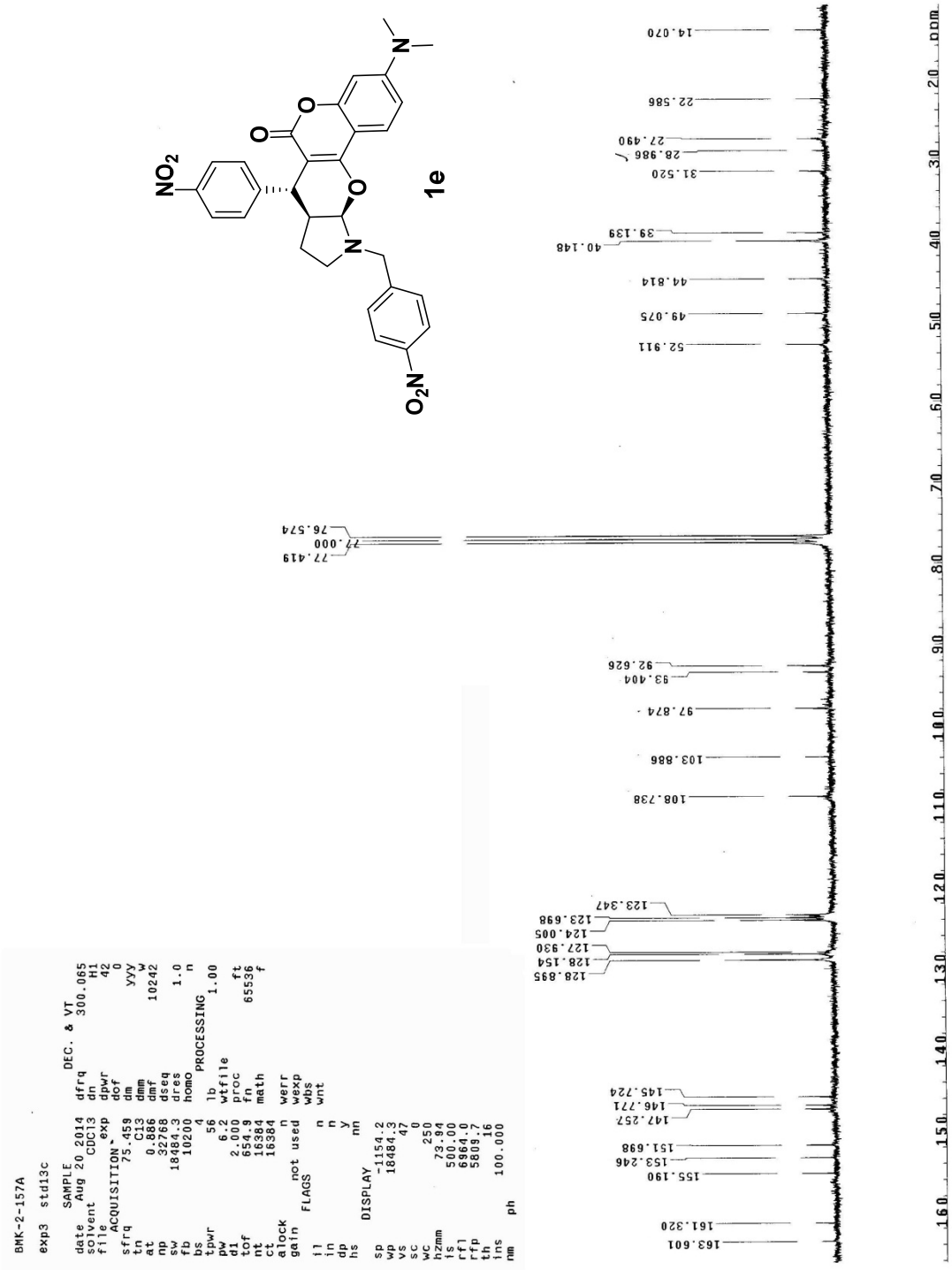



```
YH004
exp5  std13c

      SAMPLE                  DEC. & VT
date  Sep 30 2013  dfrq  300.065
solvent     CDCl3  dn          H1
file         exp   dpwr        42
  ACQUISITION      dof          0
sfrq   75.459      dm          yyy
tn          C13    dmm          w
at        0.886    dmf       9756
np        32768    dseq         n
sw     18484.3     dres       1.0
fb       10200     homo         n
bs           4       PROCESSING
tpwr                  lb     1.00
pw          6.2       wtfile
d1        2.000       proc      ft
tof       654.9       fn     65536
nt         2048       math       f
ct         2048
alock         n       werr
gain   not used       wexp
  FLAGS               wbs
il            n       wnt
in            n
dp            y
hs           nn
  DISPLAY
sp        -1153.7
wp        18484.3
vs             40
sc              0
wc            250
hzmm        73.94
is         500.00
rfl        6963.4
rfp        5809.7
th              5
ins   100.000
nm    ph
```


BMK-2-124A
exp6 stdih
SAMPLE
date May 15 2014
solvent CDCl3
file ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -600.7
wp 4800.8
vs 91
sc 0
wc 250
hzmm 19.20
is 113.52
rfl 600.7
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
diseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
ft 65536
math f
werr
wexp
wbs
wnt
OMe
MeO
1d
0.000
0.882
1.641
2.737
2.767
2.799
2.992
3.057
3.730
3.747
3.783
3.795
3.819
3.870
3.914
4.002
4.231
4.275
5.199
5.211
6.529
6.536
6.665
6.674
6.695
6.703
6.780
6.809
6.843
6.871
7.097
7.126
7.254
7.281
7.687
7.717
ppm


OMe
MeO
1d
BMK-2-124AC
exp3 std13c
SAMPLE
date May 16 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1155.9
wp 18484.3
vs 34
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6965.7
rfp 5809.7
th 6
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 9756
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
27.236
38.361
40.141
45.524
49.075
52.724
55.161
55.176
76.574
77.000
77.426
93.037
95.056
97.957
104.656
108.506
113.710
113.964
123.340
128.162
129.642
130.263
136.296
152.872
155.033
158.143
158.696
161.118
163.907
220
200
180
160
140
120
100
80
60
40
20
0
ppm

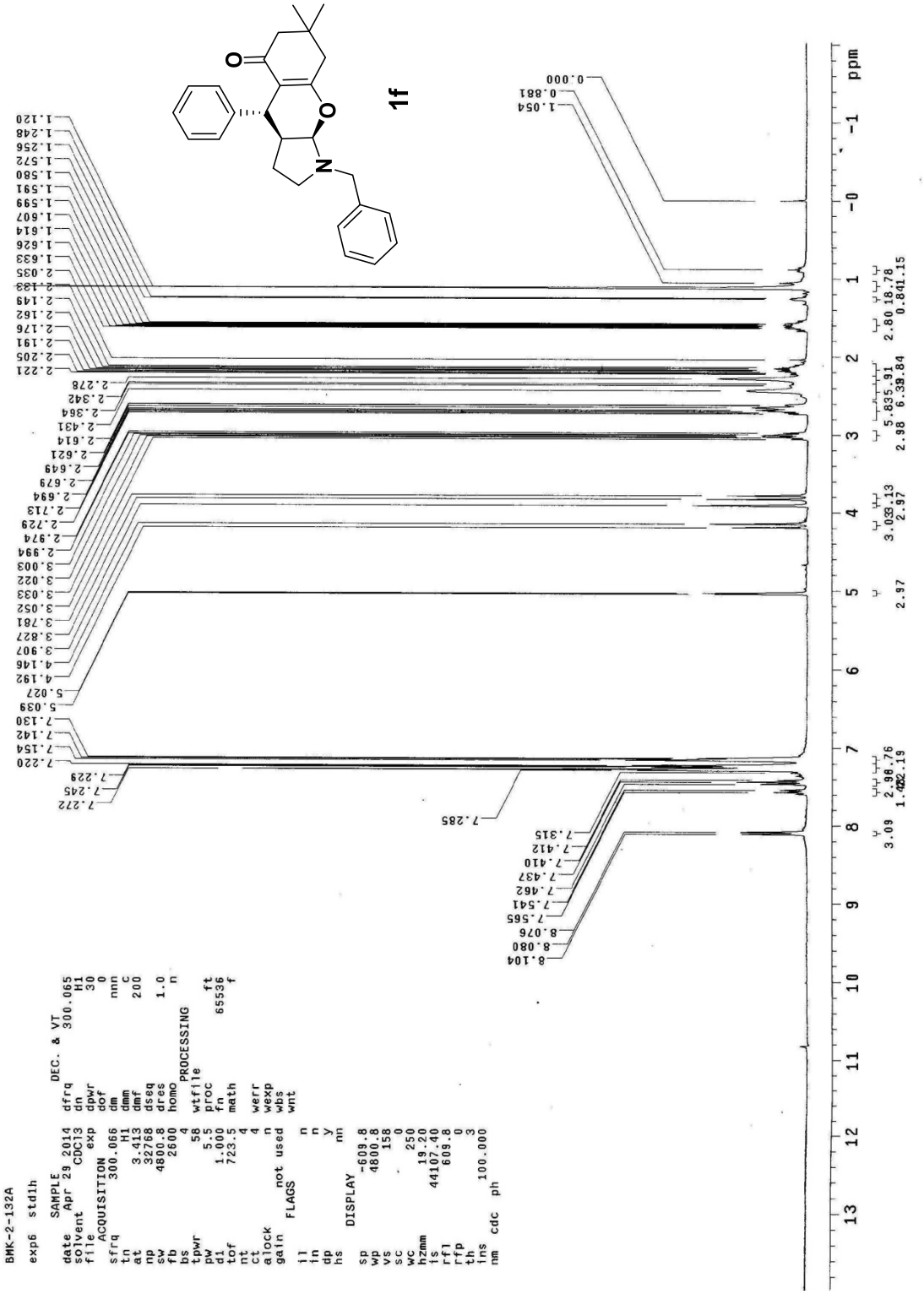

BMK-2-157
exp6 std1h
SAMPLE
date May 22 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -598.2
wp 4800.8
vs 63
sc -0
wc 250
hzmm 19.20
is 319.70
rfl 598.2
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
NO2
O2N
O
N
1e
8.208
8.202
8.179
8.156
8.126
7.679
7.649
7.547
7.517
7.431
7.405
7.376
7.347
7.266
6.719
6.710
6.689
6.680
6.536
6.528
5.195
5.183
4.492
4.444
4.181
4.133
4.083
3.121
3.086
3.056
3.050
3.024
2.838
2.825
2.795
1.643
0.000
13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
13.47 5.61 1.65 1.95 2.04 2.89 2.83 7.06 0.64 7.25 1.45
2.76 1.58 2.39 2.53 29.99 2.98 4.47 1.52

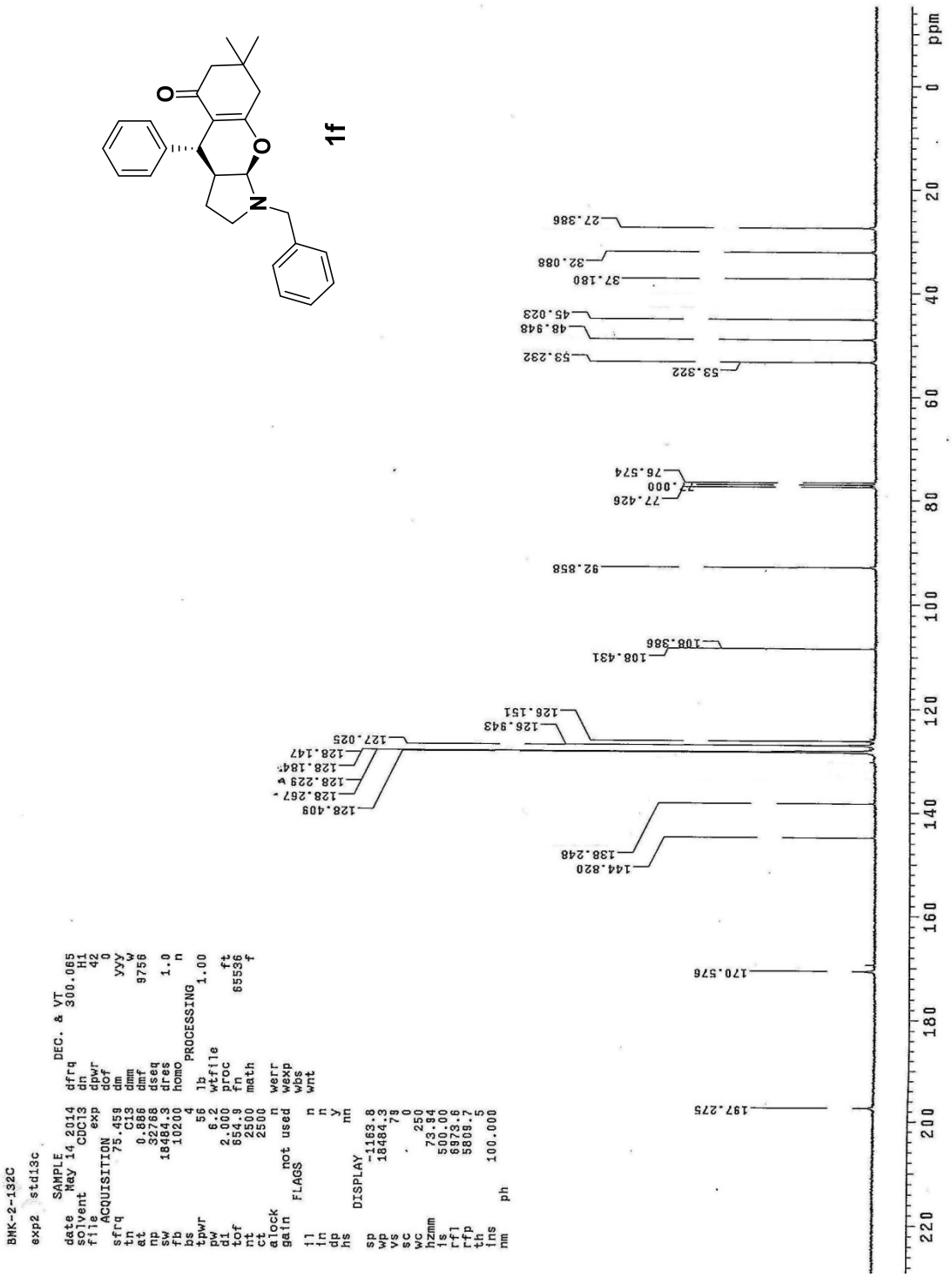


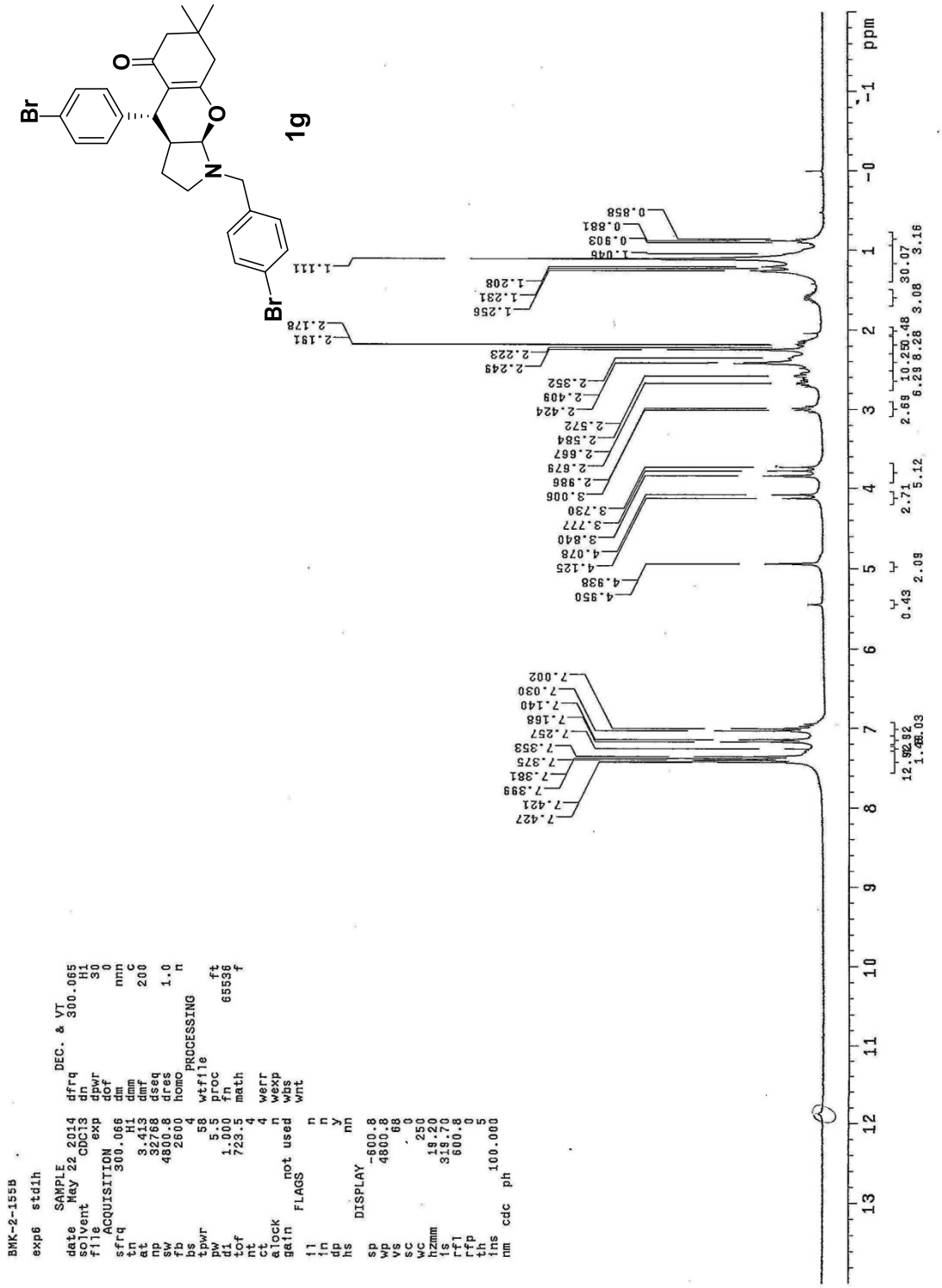
BMK-2-157A

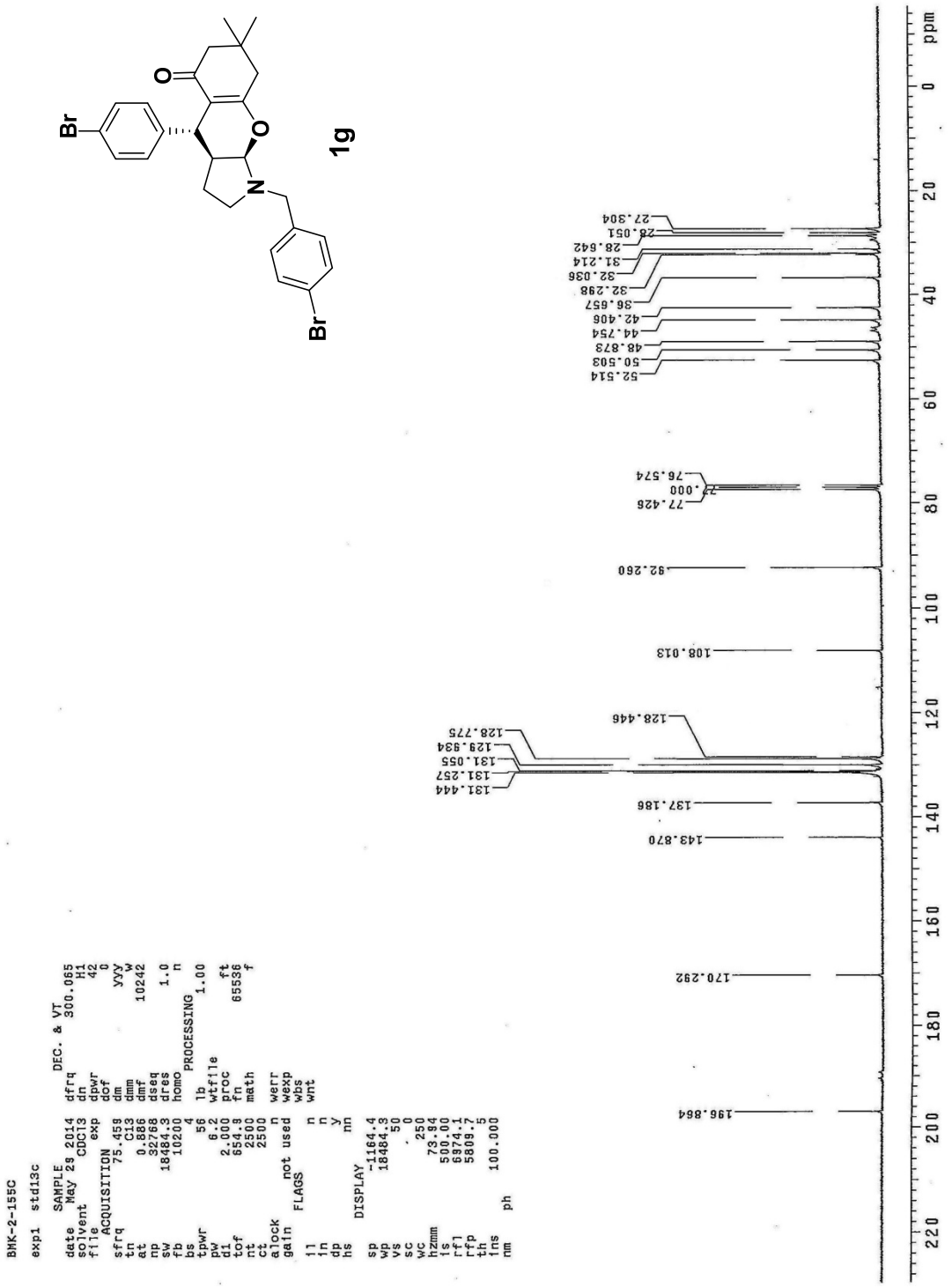
exp3 std13c

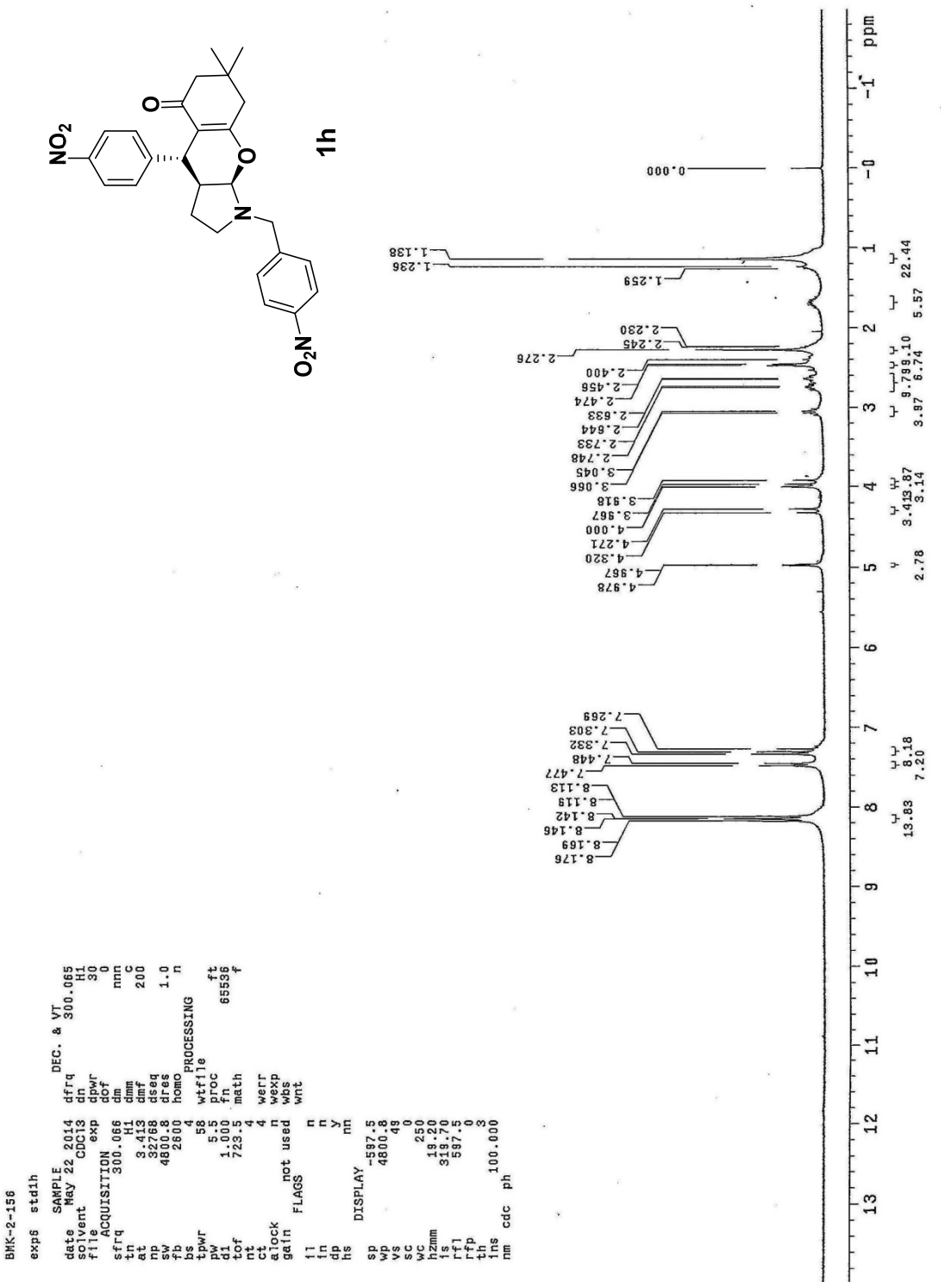
SAMPLE DEC. & VT
date Aug 20 2014 dfrq 300.065
solvent CDCl3 dn H1
file exp dpwr 42
ACQUISITION dof 0
sfrq 75.459 dm yyy
tn C13 dmm w
at 0.886 dmf 10242
np 32768 dseq
sw 18484.3 dres 1.0
fb 10200 homo n
bs 4 PROCESSING
tpwr 56 lb 1.00
pw 6.2 wtfile
d1 2.000 proc ft
tof 654.9 fn 65536
nt 16384 math f
ct 16384
dlock n werr
gain not used wexp
FLAGS wbs
il n wnt
in n
dp y
hs nn
DISPLAY
sp -1154.2
wp 18484.3
vs 47
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6964.0
rfp 5809.7
th 16
ins 100.000
nm ph

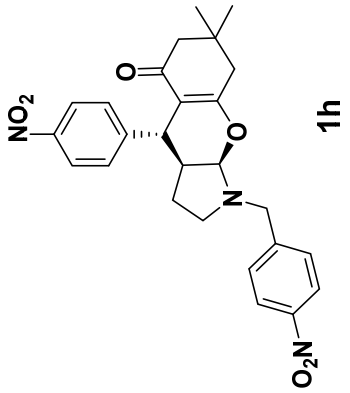

1f
BMK-2-132A
expb stdih
SAMPLE
date Apr 29 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -609.8
wp 4800.8
vs 158
sc 0
wc 250
hzmm 19.20
is 44107.40
rfl 609.8
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
ppm


1f
BMK-2-132C
exp2 std13c
SAMPLE
DEC. & VT
date May 14 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1163.8
wp 18484.3
vs 73
sc 0
wc 250
hzmm 73.94
is 500.00
rf1 6973.6
rfp 5809.7
th 5
ins 100.000
nm ph
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 9756
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
197.275
170.576
144.820
138.248
128.408
128.267
128.229
128.184
128.147
127.025
126.943
126.151
108.586
108.431
92.858
77.426
77.000
76.574
53.322
53.232
48.948
45.023
37.180
32.088
27.386
220
200
180
160
140
120
100
80
60
40
20
0
ppm

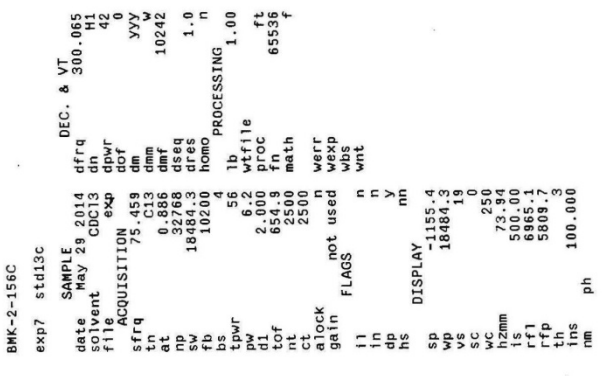

1g
Br
O
O
N
Br
BMK-2-155B
expb stdih
SAMPLE
date May 22 2014
solvent CDCl3
file ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4807.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
fl n
fn n
dp y
hs nn
DISPLAY
sp 600.8
wp 4800.8
vs 68
sc 0
wc 250
hzmm 19.20
fs 319.70
rfl 600.8
rfp 0
th 5
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.085
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
7.427
7.421
7.399
7.381
7.375
7.353
7.257
7.168
7.140
7.030
7.002
4.950
4.938
4.125
4.078
3.840
3.777
3.730
3.006
2.986
2.679
2.667
2.584
2.572
2.424
2.409
2.352
2.249
2.223
2.181
2.178
1.256
1.231
1.208
1.111
1.046
0.903
0.881
0.858
ppm

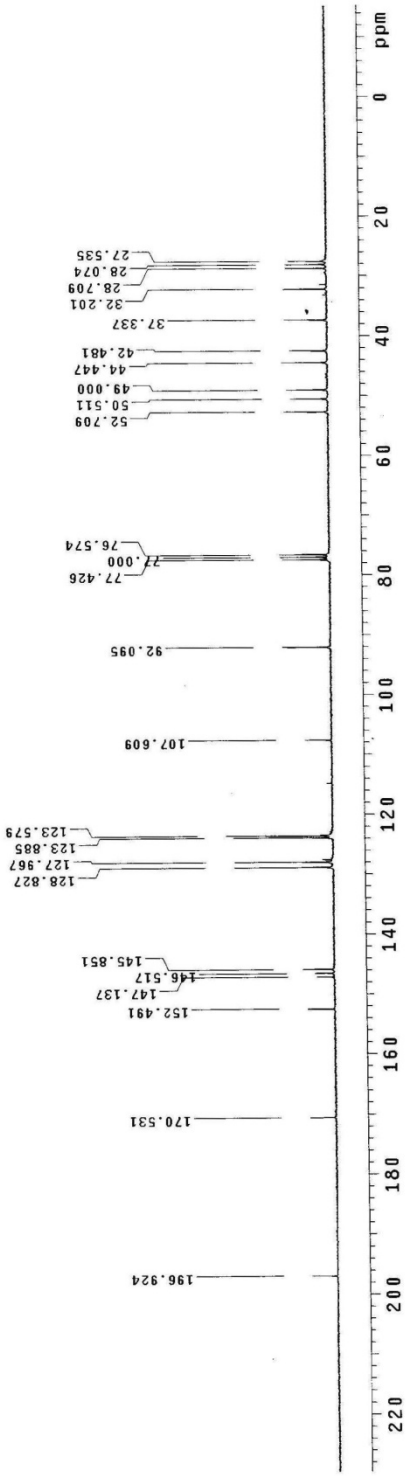

Br
O
O
N
Br
1g
BMK-2-155C
exp1 std13c
SAMPLE
date May 29 2014
solvent CDCl3
file ACQUISITION
sfrq 75.459
tn C13
at 0.886
nt 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.8
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1164.4
wp 18484.3
vs 50
sc 0
wc 250
hzmm 73.84
is 500.00
rfl 6974.1
rfp 5809.7
th 5
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 40
dof 0
dm yyy
dmm y
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
196.864
170.292
143.870
137.186
131.444
131.257
131.055
129.934
128.775
128.446
108.013
92.260
77.425
77.000
76.574
52.514
50.503
48.873
44.754
42.406
36.657
32.298
32.036
31.214
28.642
28.051
27.304
220
200
180
160
140
120
100
80
60
40
20
0
ppm


1h
O2N
NO2
O
N
O
BMK-2-156
exps stdih
SAMPLE
date May 22 2014
solvent CDCl3
file
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
ll n
ln n
dp y
hs nn
DISPLAY
sp -597.5
wp 4800.8
vs 49
sc 0
wc 250
hzmm 19.20
is 319.70
rfl 597.5
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
0.000
1.138
1.236
1.259
2.230
2.245
2.276
2.400
2.456
2.474
2.633
2.644
2.733
2.748
3.045
3.066
3.918
3.967
4.000
4.271
4.320
4.967
4.978
7.169
7.176
8.142
8.146
8.113
8.119
7.448
7.477
7.303
7.332
7.269
ppm


O
NO2
O
N
O2N

1h

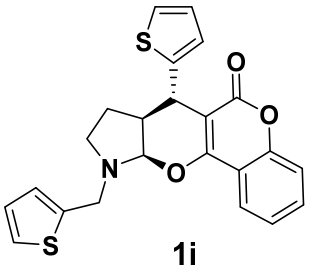

BMK-2-156C
exp7 std13c
SAMPLE
date May 29 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1155.4
wp 18484.3
vs 19
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6965.1
rfp 5809.7
th 3
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt


196.924
170.531
152.491
147.137
146.517
145.851
128.827
127.967
123.885
123.579
107.609
92.095
77.426
77.000
76.574
52.709
50.511
49.000
44.447
42.481
37.337
32.201
28.709
28.074
27.535
ppm

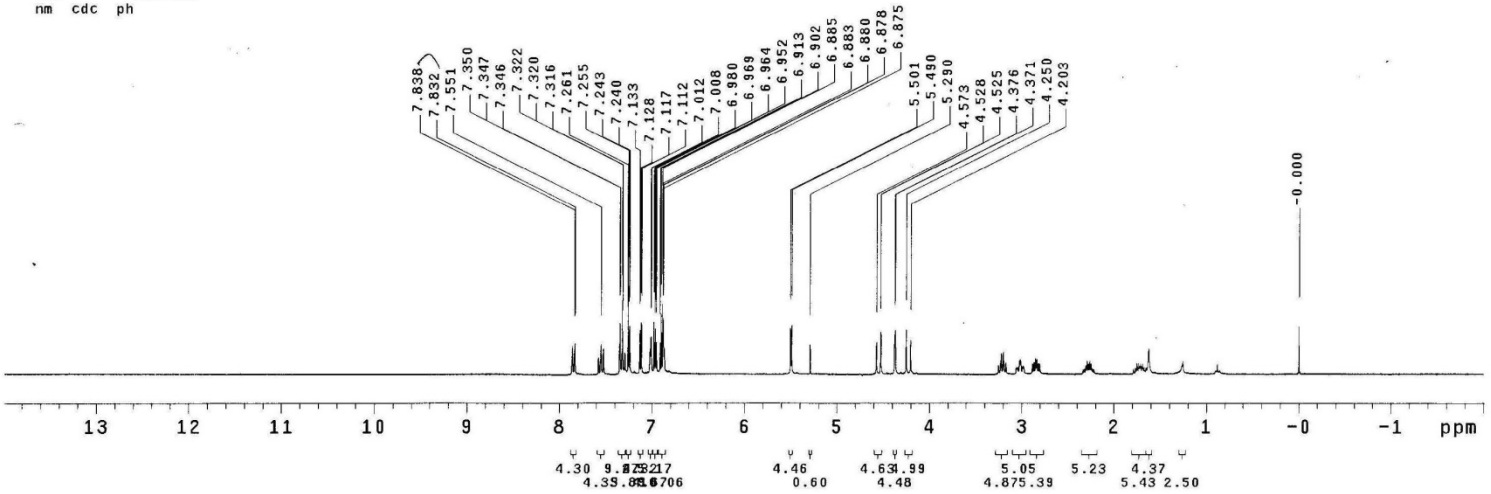
BMK-2-149B

exp6 std1h

SAMPLE		DEC. & VT	
date	Jun 17 2014	dfrq	300.065
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.066	dm	nnn
tn	H1	dmm	c
at	3.413	dmf	200
np	32768	dseq	
sw	4800.8	dres	1.0
fb	2600	homo	n
bs	4	PROCESSING	
tpwr	58	wtfile	
pw	5.5	proc	ft
d1	1.000	fn	65536
tof	723.5	math	f
nt	4		
ct	4	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-602.3		
wp	4800.8		
vs	15		
sc	0		
wc	250		
hzmm	19.20		
is	13.71		
rfl	602.3		
rfp	0		
th	5		
ins	100.000		
nm cdc ph			



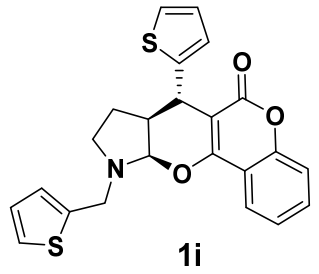
1i



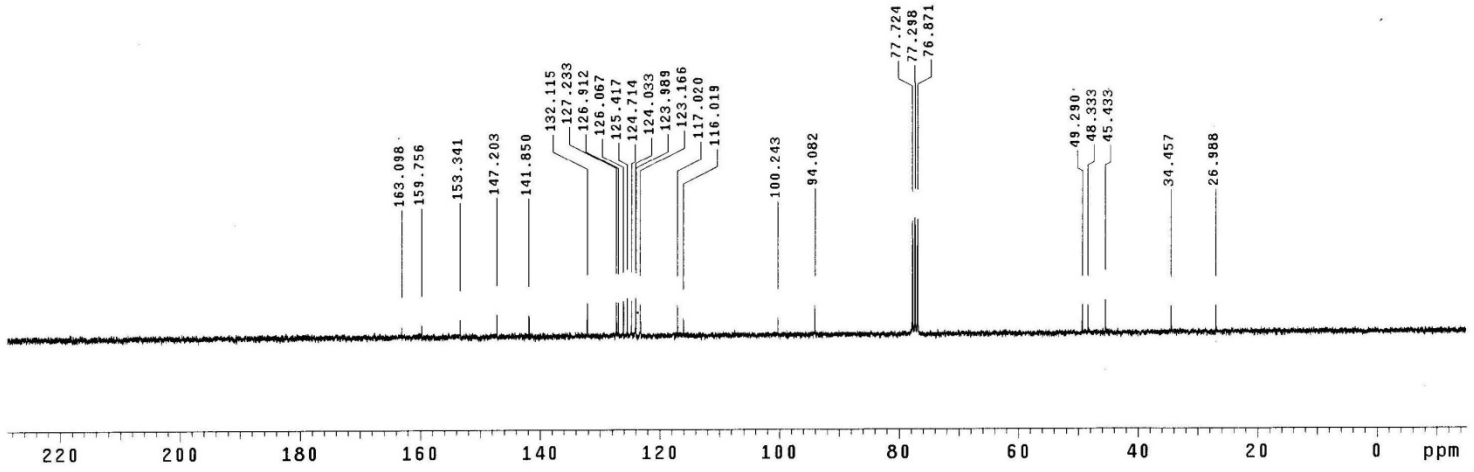
BMK-2-149BC

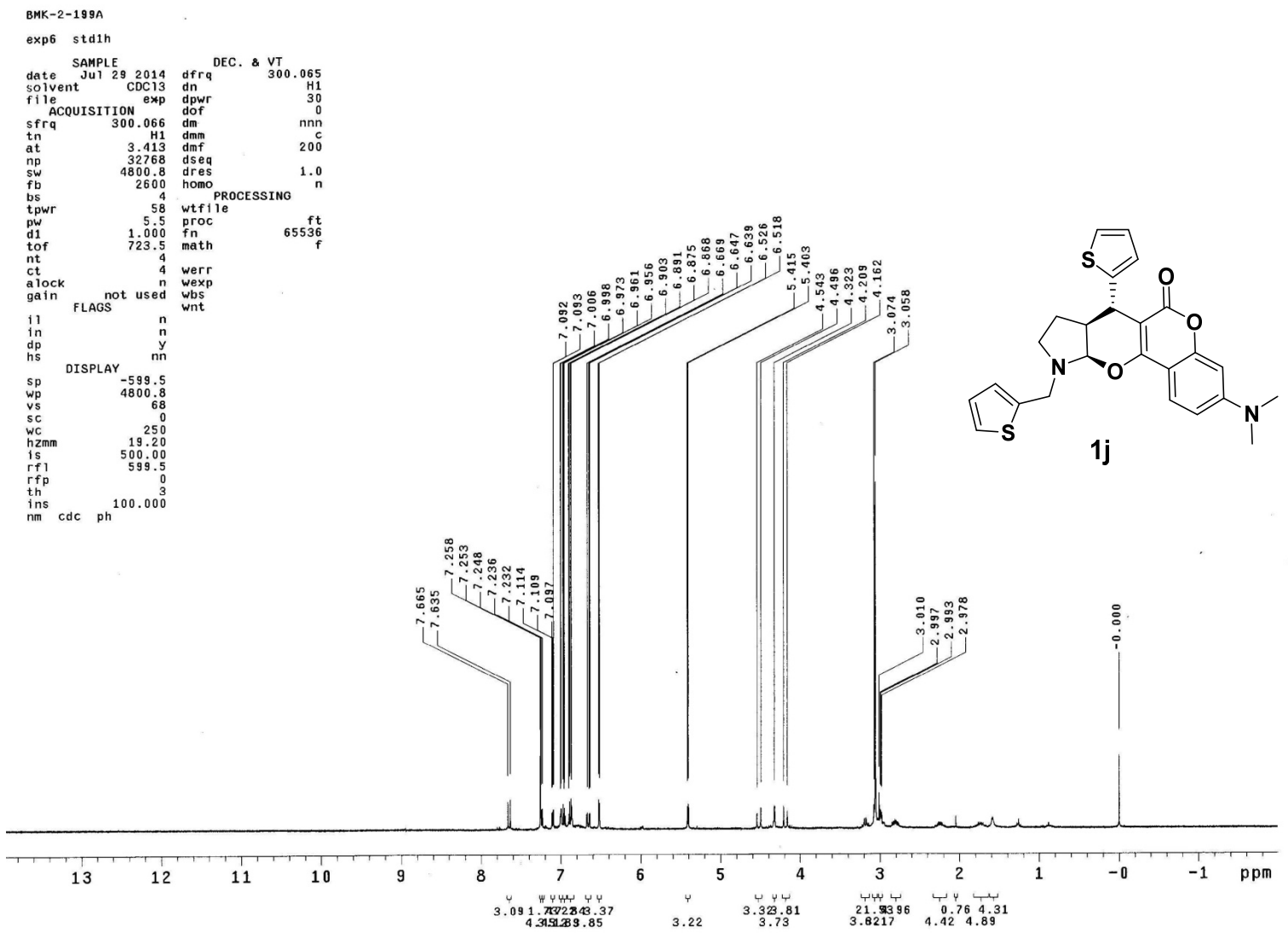
exp3 std13c

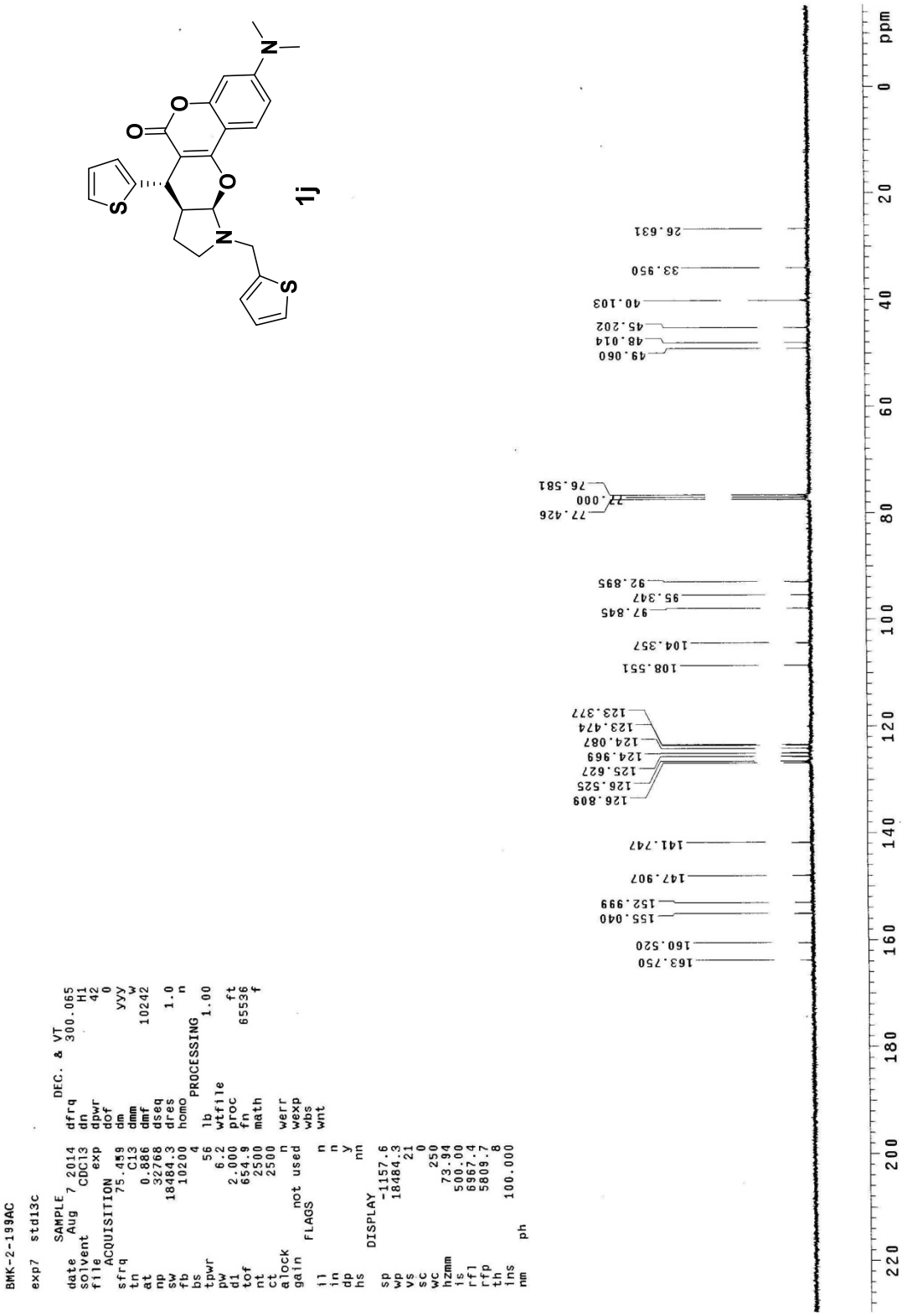
SAMPLE		DEC. & VT	
date	Aug 13 2014	dfrq	300.065
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	75.459	dm	yyy
tn	C13	dmm	w
at	0.886	dmf	10242
np	32768	dseq	
sw	18484.3	dres	1.0
fb	10200	homo	n
bs	4	PROCESSING	
tpwr	56	lb	1.00
pw	6.2	wtfile	
d1	2.000	proc	ft
tof	654.9	fn	65536
nt	2500	math	f
ct	2500		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1131.2		
wp	18484.3		
vs	31		
sc	0		
wc	250		
hzmm	73.94		
is	500.00		
rfl	1131.2		
rfp	0		
th	12		
ins	100.000		
nm	ph		

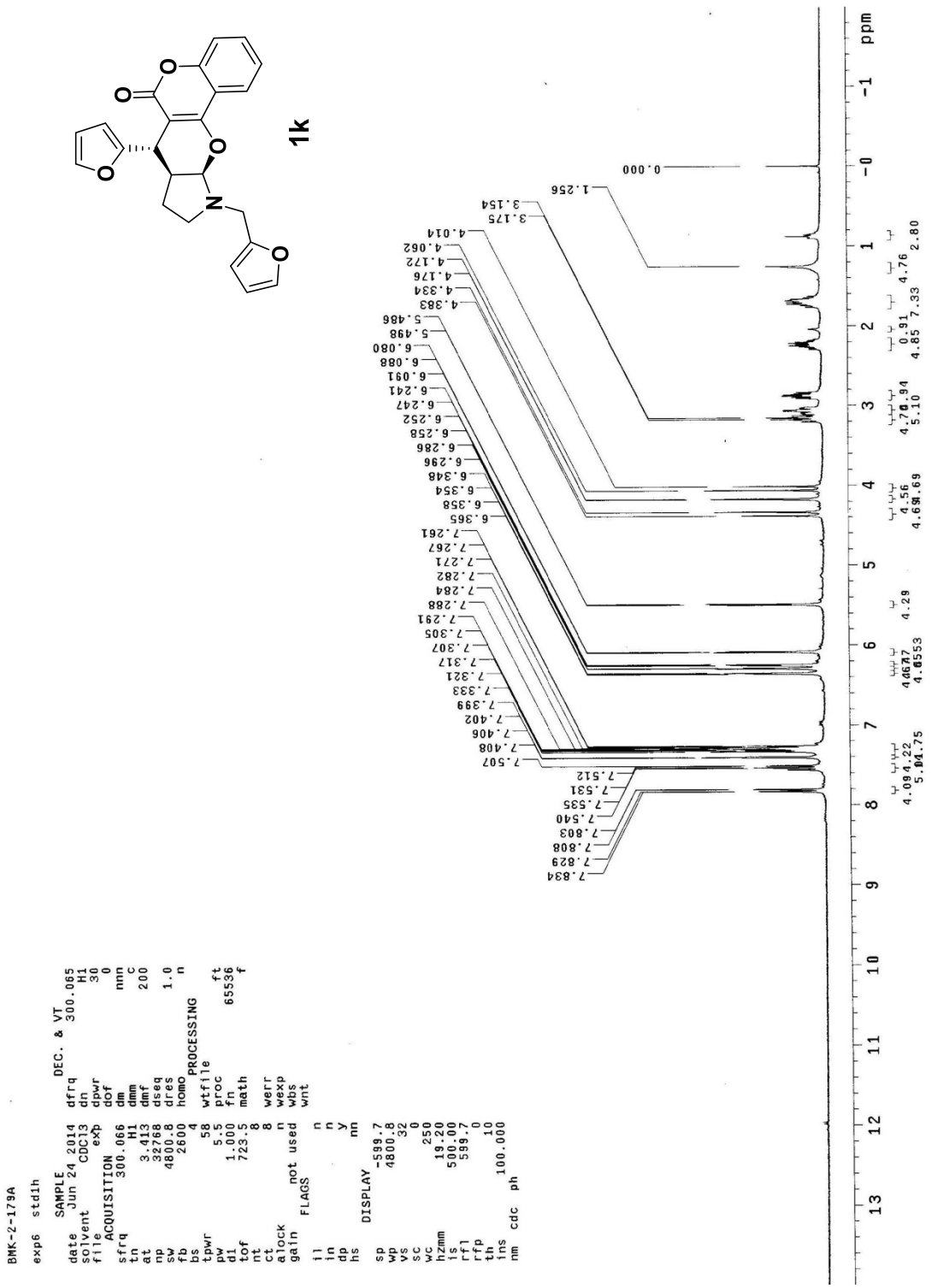


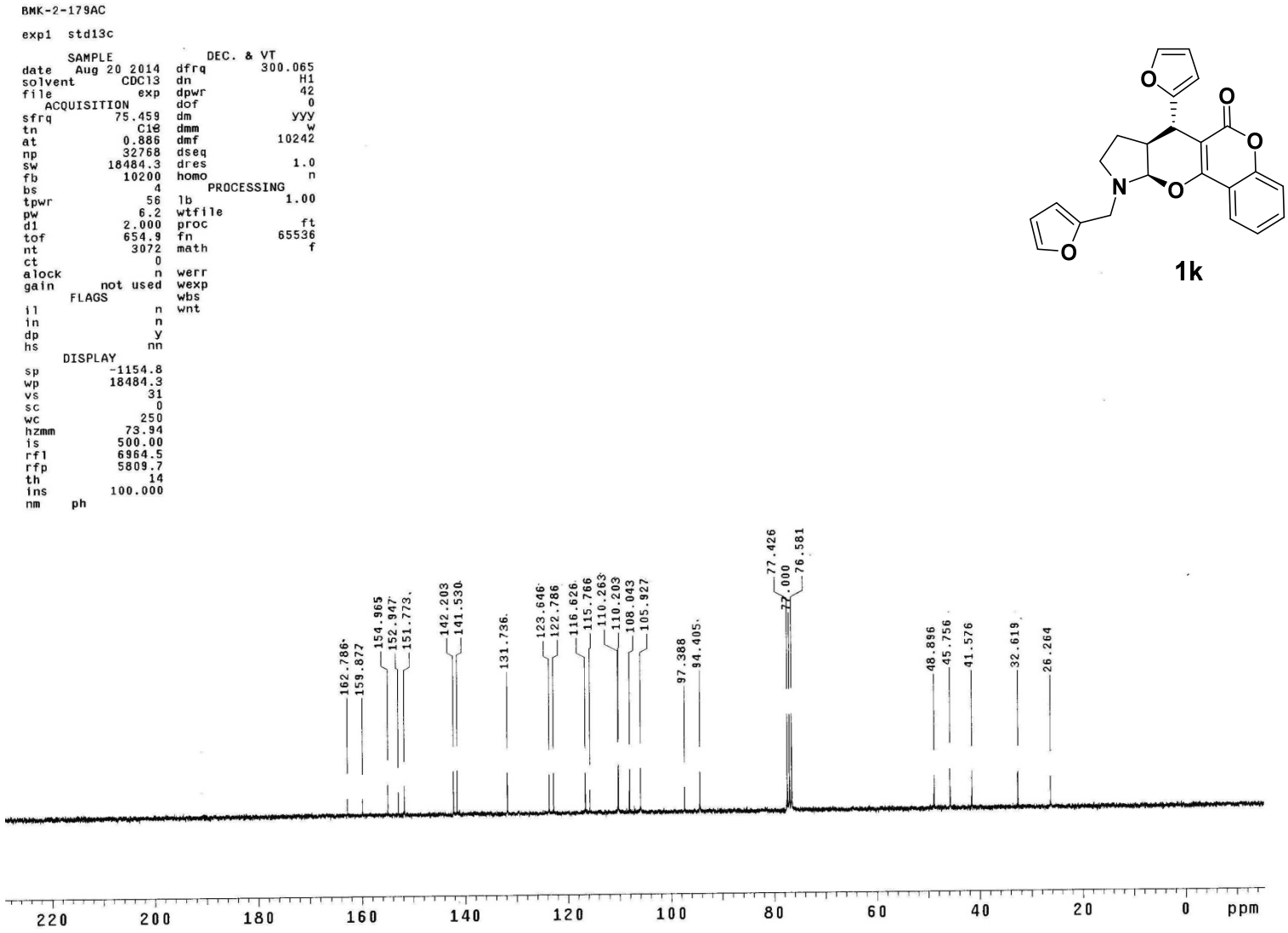
1i

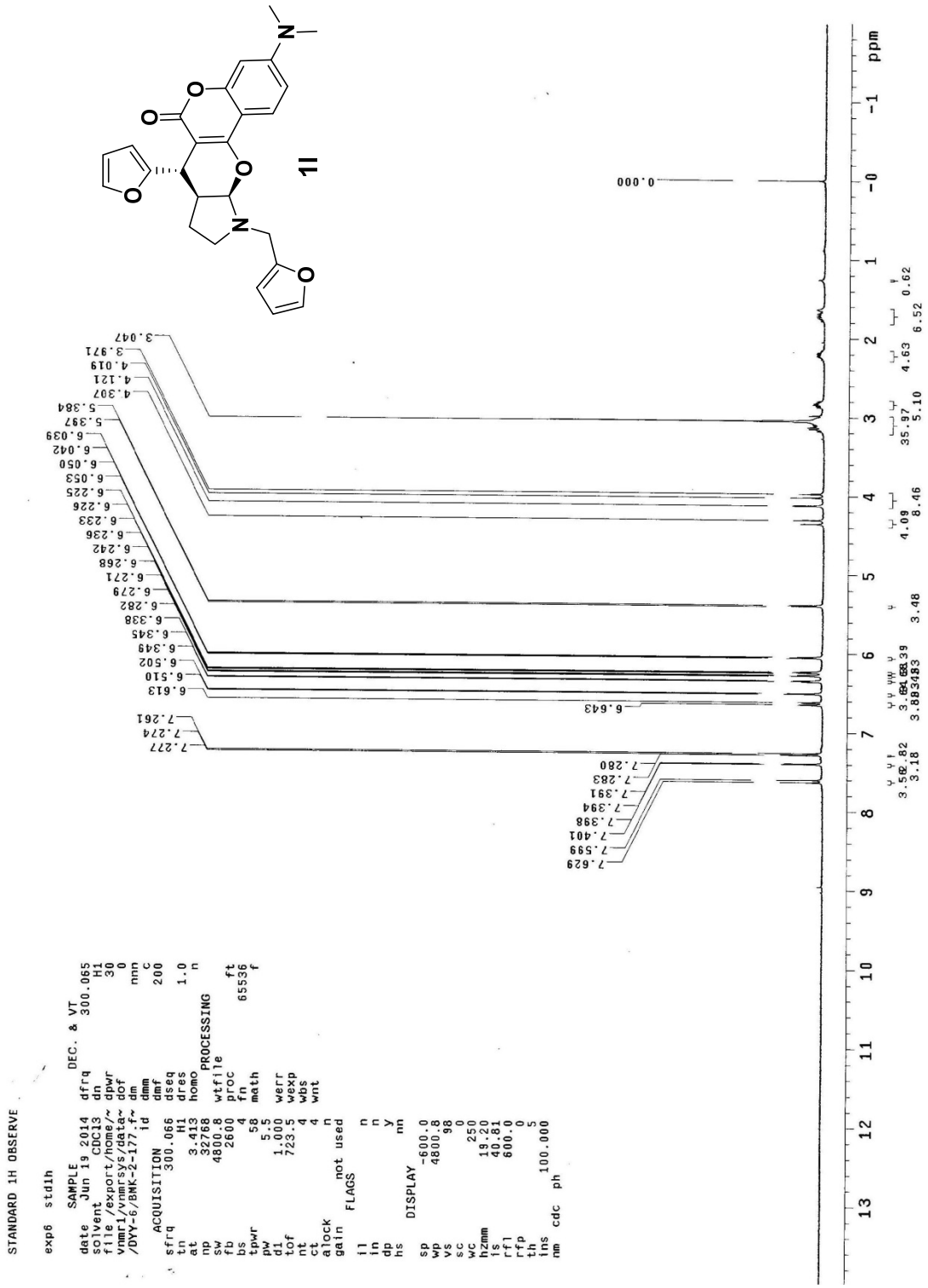



BMK-2-199A
exp6 stdlh
SAMPLE
date Jul 29 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -599.5
wp 4800.8
vs 68
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 599.5
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
7.665
7.635
7.258
7.253
7.248
7.236
7.232
7.114
7.109
7.097
7.092
7.053
7.006
6.998
6.973
6.961
6.956
6.903
6.891
6.875
6.868
6.669
6.647
6.639
6.526
6.518
5.415
5.403
4.543
4.496
4.323
4.209
4.162
3.074
3.058
3.010
2.997
2.993
2.978
-0.000
S
O
O
N
O
N
S
1j
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
3.09
3.22
3.73
3.81
4.42
4.89
0.76
4.31


1j
BMK-2-199AC
exp7 std13c
SAMPLE
date Aug 7 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1157.6
wp 18484.3
vs 21
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6867.4
rfp 5809.7
th 8
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm y
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
163.750
160.520
155.040
152.999
147.907
141.747
126.809
126.525
125.627
124.969
124.087
123.474
123.377
108.551
104.357
97.845
95.347
92.895
77.426
77.000
76.581
49.060
48.014
45.202
40.103
33.950
26.631
220
200
180
160
140
120
100
80
60
40
20
0
ppm


1k
BMK-2-179A
exp6 std1h
SAMPLE
date Jun 24 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -599.7
wp 4800.8
vs 32
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 599.7
rfp 0
th 10
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
0.000
1.256
3.154
3.175
4.014
4.062
4.172
4.176
4.334
4.383
5.486
5.498
6.080
6.088
6.091
6.241
6.247
6.252
6.258
6.286
6.296
6.348
6.354
6.358
6.365
7.261
7.267
7.271
7.282
7.284
7.288
7.291
7.305
7.307
7.317
7.321
7.333
7.399
7.402
7.406
7.408
7.507
7.512
7.531
7.535
7.540
7.803
7.808
7.829
7.834
ppm

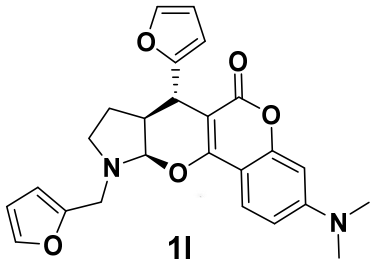

BMK-2-179AC
exp1 std13c
SAMPLE
date Aug 20 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 3072
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1154.8
wp 18484.3
vs 31
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6964.5
rfp 5809.7
th 14
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
O
O
O
N
O
O
1k
162.786
159.877
154.965
152.947
151.773
142.203
141.530
131.736
123.646
122.786
116.626
115.766
110.263
110.203
108.043
105.927
97.388
94.405
77.426
77.000
76.581
48.896
45.756
41.576
32.619
26.264
220
200
180
160
140
120
100
80
60
40
20
0
ppm


11
STANDARD 1H OBSERVE
exp6 std1h
SAMPLE
Jun 19 2014
CDCl3
/export/home/~
/vnmrsys/data~
/DYY-6/BMK-2-177
DEC. & VT
300.065
H1
30
0
nnn
c
200
1.0
n
PROCESSING
ft
65536
f
n
n
y
nn
-600.0
4800.8
98
0
250
19.20
40.81
600.0
0
5
100.000
0.000
7.629
7.599
7.401
7.398
7.394
7.391
7.283
7.280
7.277
7.274
7.261
6.643
6.613
6.510
6.502
6.349
6.345
6.338
6.282
6.279
6.271
6.268
6.242
6.236
6.233
6.226
6.225
6.053
6.050
6.042
6.039
5.397
5.384
4.307
4.121
4.019
3.971
3.047
3.582
3.180
3.884
3.883
3.480
4.09
8.46
35.97
5.10
4.63
6.52
0.62
ppm

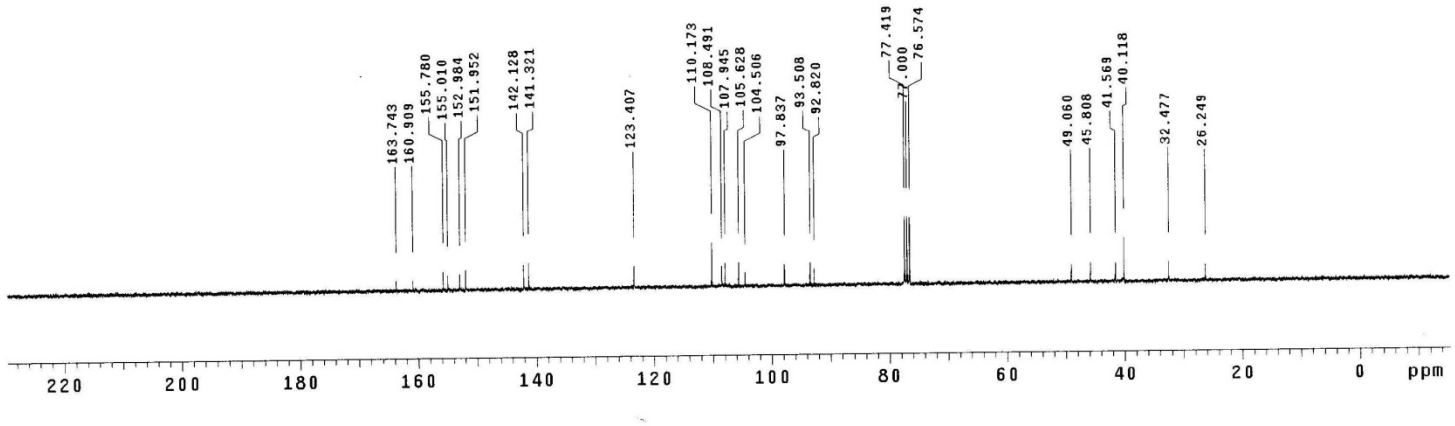
BMK-2-177C

exp1 std13c

SAMPLE		DEC. & VT	
date	Aug 12 2014	dfrq	300.065
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	75.459	dm	yyy
tn	C13	dmm	w
at	0.886	dmf	10242
np	32768	dseq	
sw	18484.3	dres	1.0
fb	10200	homo	n
bs	4	PROCESSING	
tpwr	56	lb	1.00
pw	6.2	wtfile	
d1	2.000	proc	ft
tof	654.9	fn	65536
nt	2500	math	f
ct	2500		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1154.8		
wp	18484.3		
vs	19		
sc	0		
wc	250		
hzmm	44.91		
is	500.00		
rfl	6964.5		
rfp	5809.7		
th	8		
ins	100.000		
nm	ph		



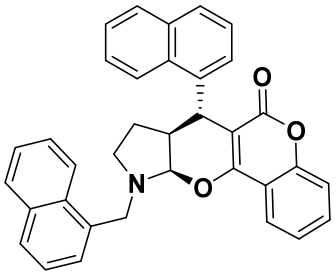
1l



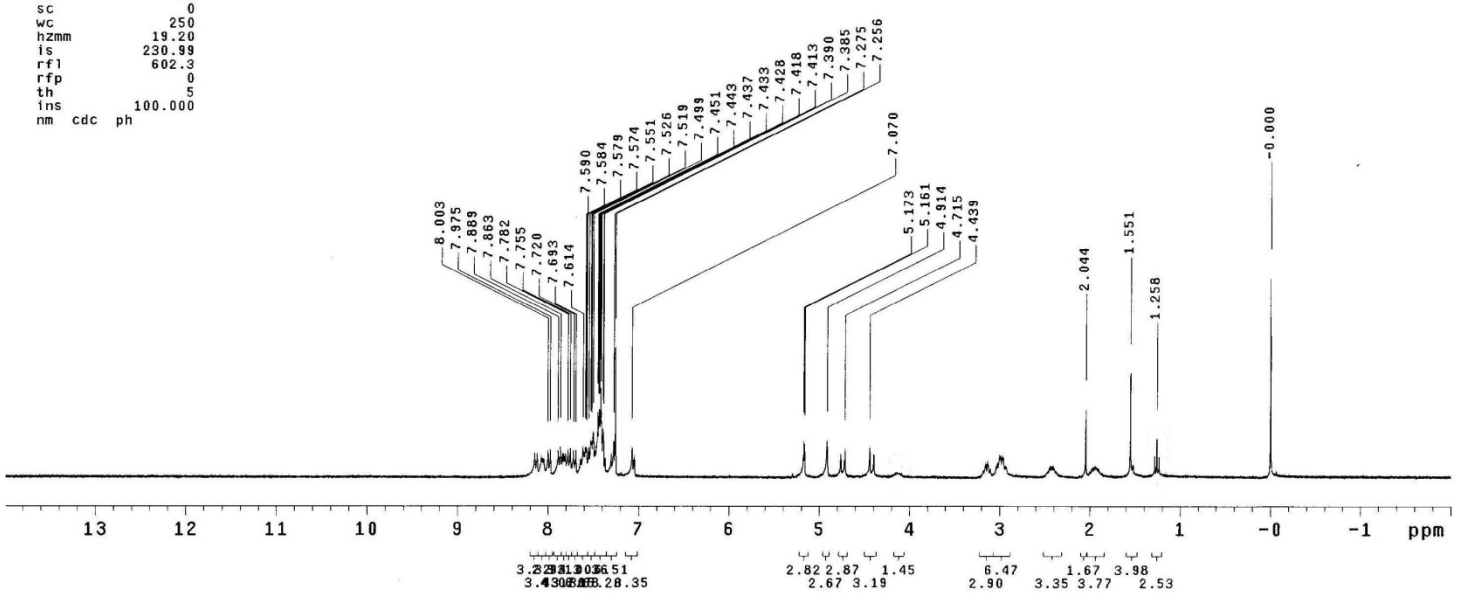
BMK-2-183B

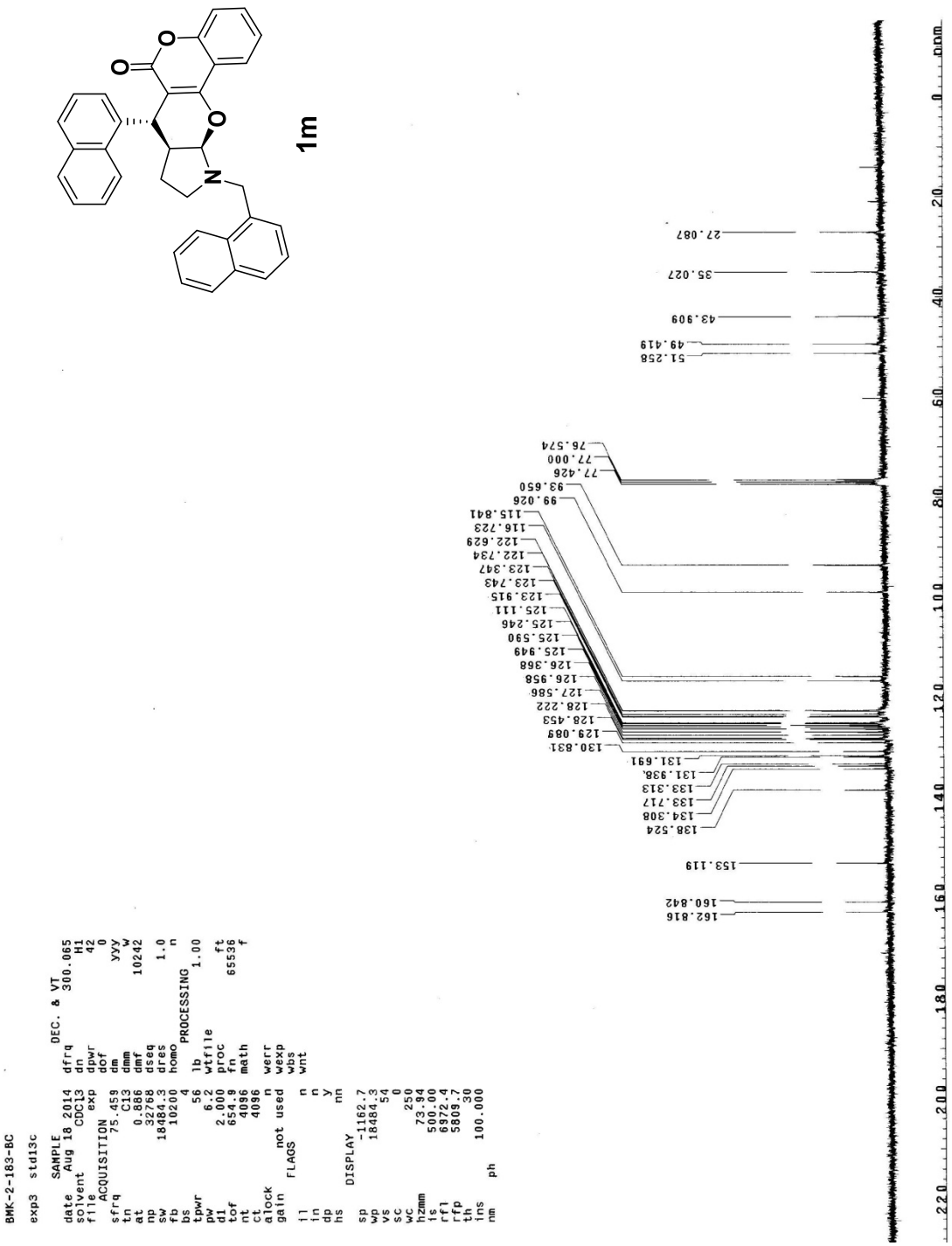
exp6 std1h

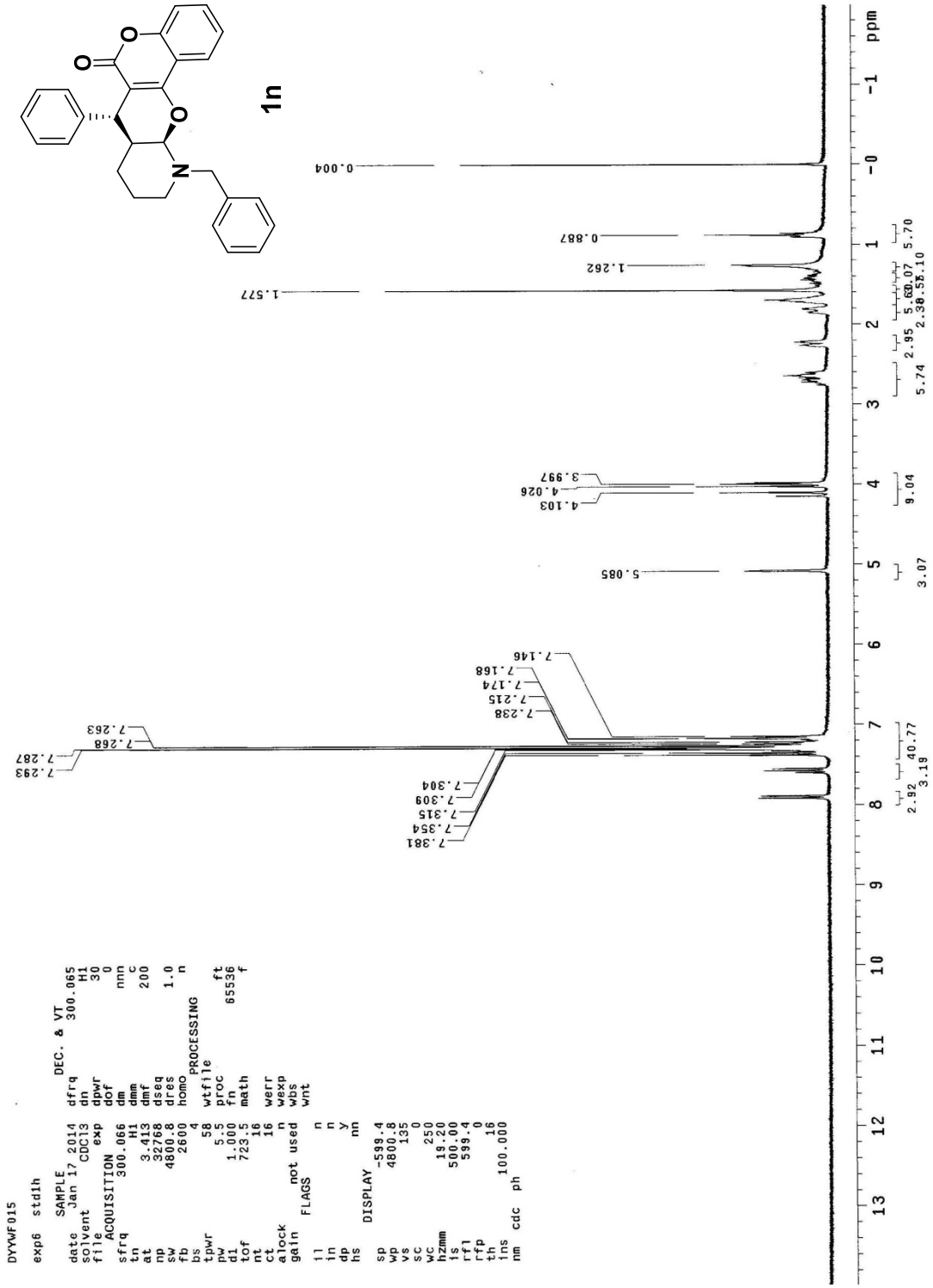
SAMPLE		DEC. & VT	
date	Aug 12 2014	dfrq	300.065
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.066	dm	nnn
tn	H1	dmm	c
at	3.413	dmf	200
np	32768	dseq	
sw	4800.8	dres	1.0
fb	2600	homo	n
bs	4	PROCESSING	
tpwr	58	wtfile	
pw	5.5	proc	ft
d1	1.000	fn	65536
tof	723.5	math	f
nt	16		
ct	16	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-602.3		
wp	4800.8		
vs	35		
sc	0		
wc	250		
hzmm	19.20		
is	230.99		
rfl	602.3		
rfp	0		
th	5		
ins	100.000		
nm cdc ph			

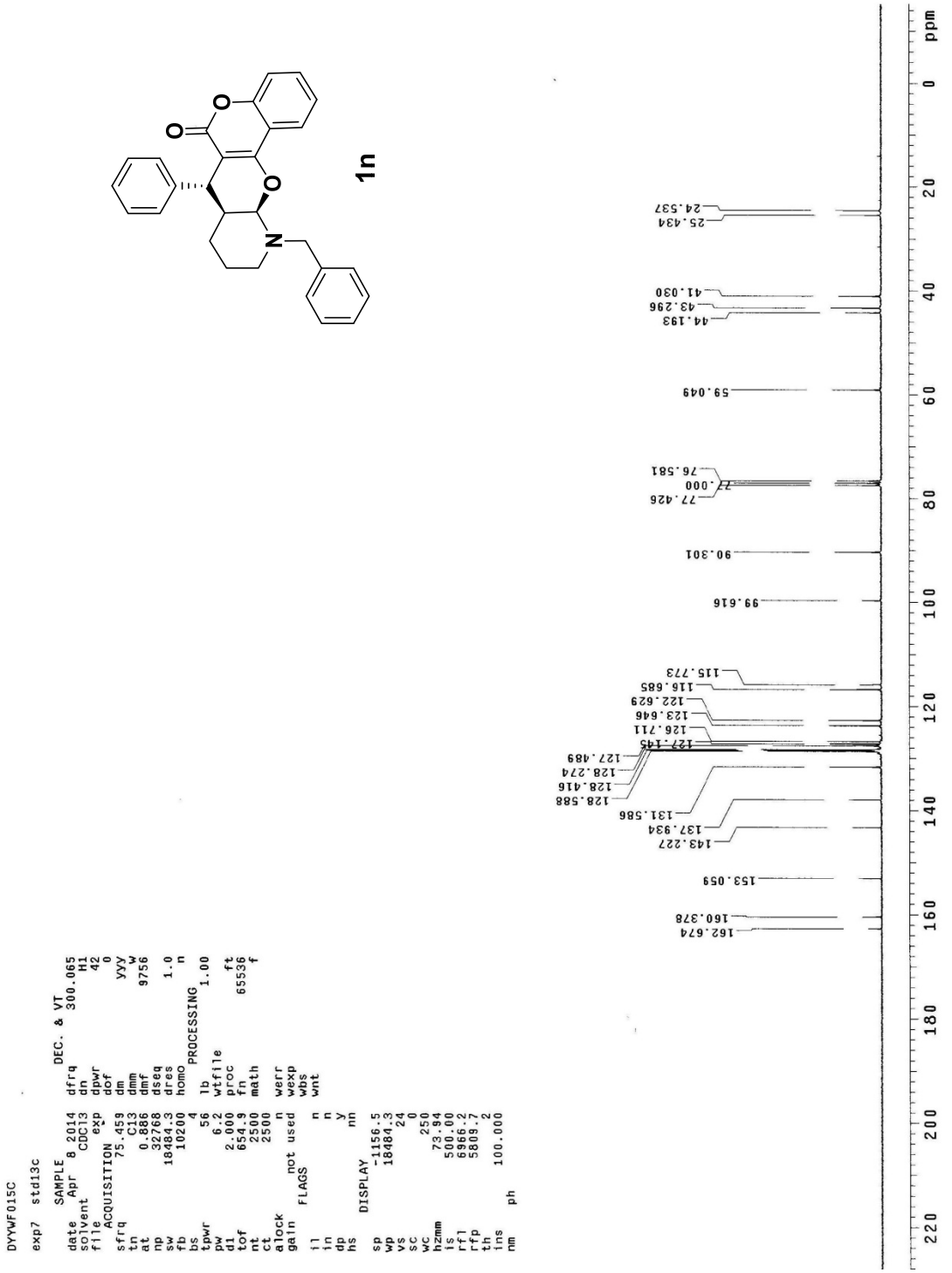


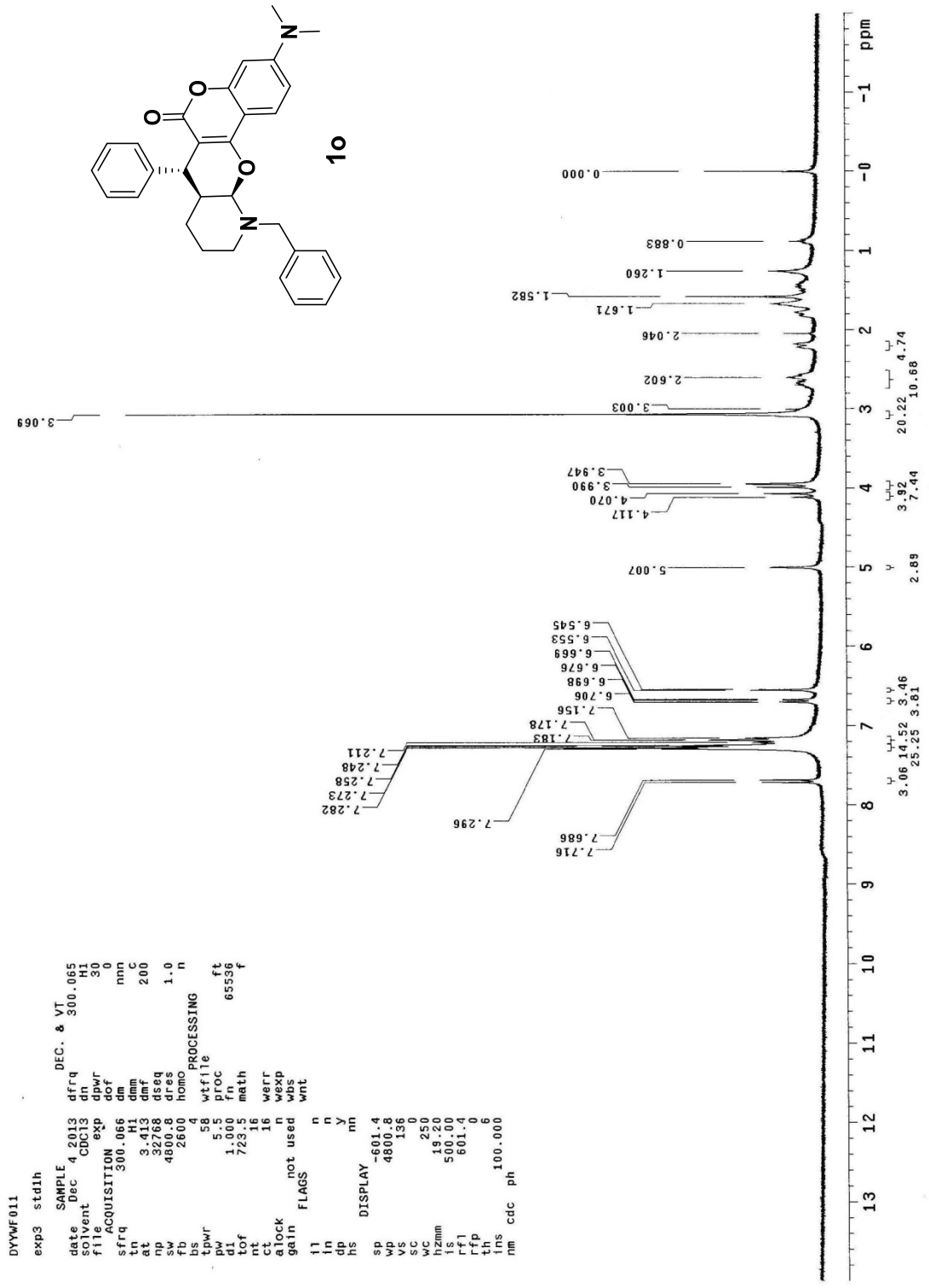
1m

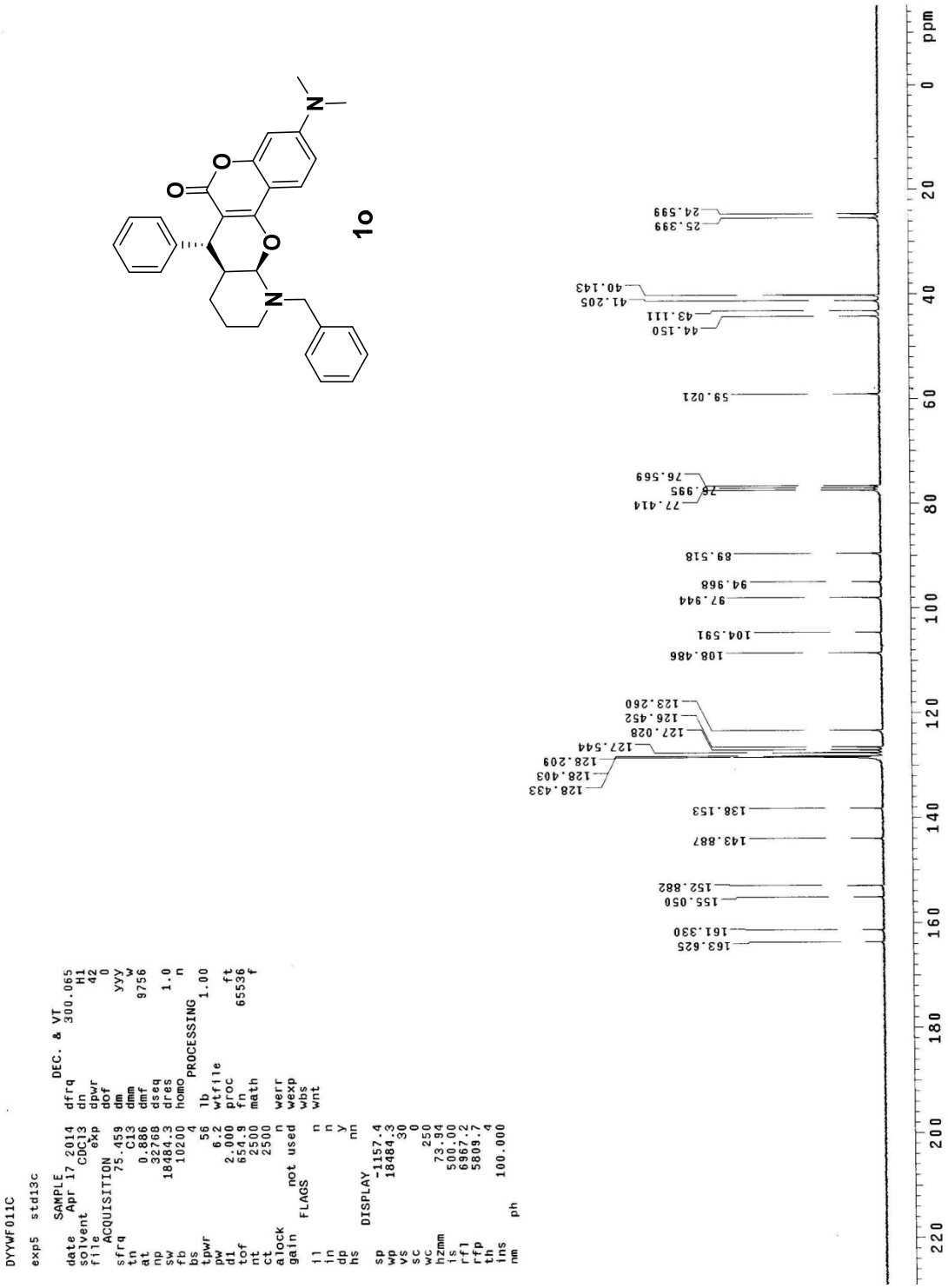


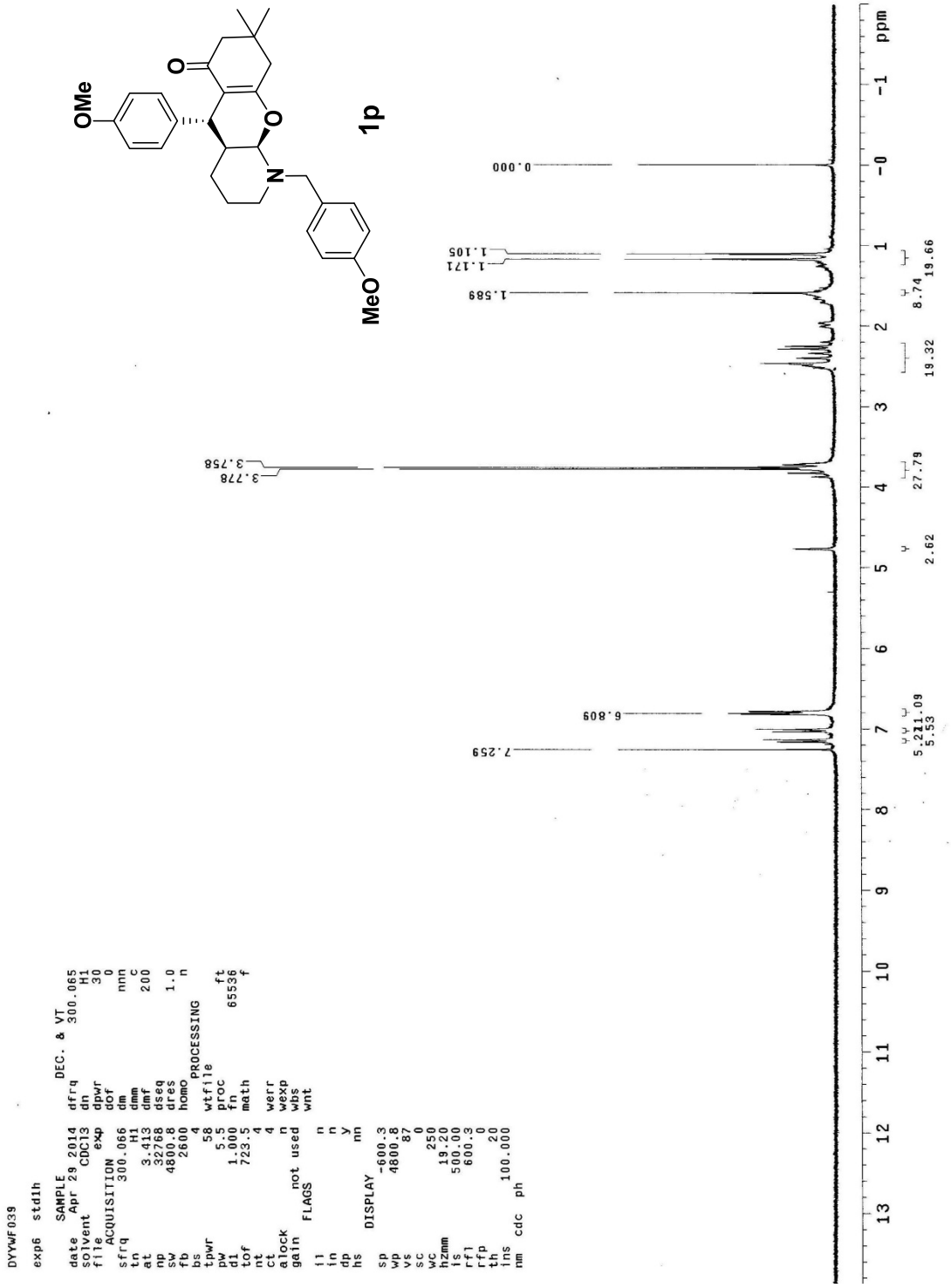

1m
BMK-2-183-BC
exp3 std13c
SAMPLE DEC. & VT
date Aug 18 2014 dfrq 300.065
solvent CDCl3 dn H1
file exp dpwr 42
ACQUISITION dof 0
sfrq 75.458 dm yyy
tn C13 dmm y
at 0.886 dmf 10242
np 32768 dseq
sw 18484.3 dres 1.0
fb 10200 homo n
bs 4 PROCESSING
tpwr 56 lb 1.00
pw 6.2 wtfile
d1 2.000 proc ft
tof 654.9 fn 65536
nt 4096 math f
ct 4096
alock n werr
gain not used wexp
FLAGS wbs
il n wnt
in n
dp y
hs nn
DISPLAY
sp -1162.7
wp 18484.3
vs 54
sc 0
wc 250
hzmm 73.94
fs 500.00
rfl 6972.4
rfp 5805.7
th 30
ins 100.000
nm ph
27.087
35.027
43.909
49.419
51.258
76.574
77.000
77.426
93.650
99.026
115.841
116.723
122.629
122.734
123.347
123.743
123.915
125.111
125.246
125.590
125.949
126.368
126.958
127.586
128.222
128.453
129.089
130.831
131.691
131.938
133.313
133.717
134.308
138.524
153.119
160.842
162.816
220
200
180
160
140
120
100
80
60
40
20
0
ppm

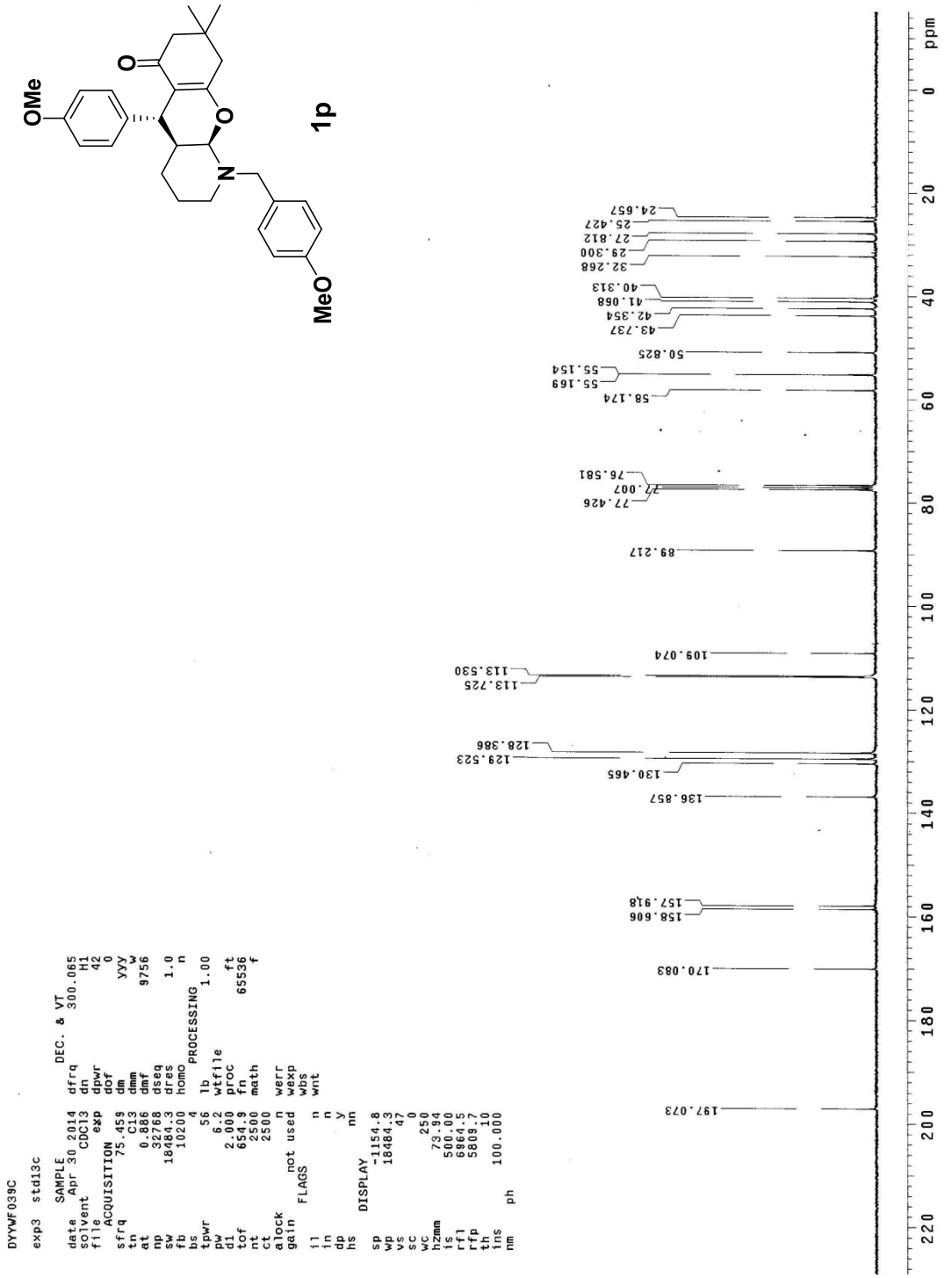

1n
0.004
0.887
1.262
1.577
3.997
4.026
4.103
5.085
7.146
7.168
7.174
7.215
7.238
7.263
7.268
7.287
7.293
7.304
7.309
7.315
7.354
7.381
ppm

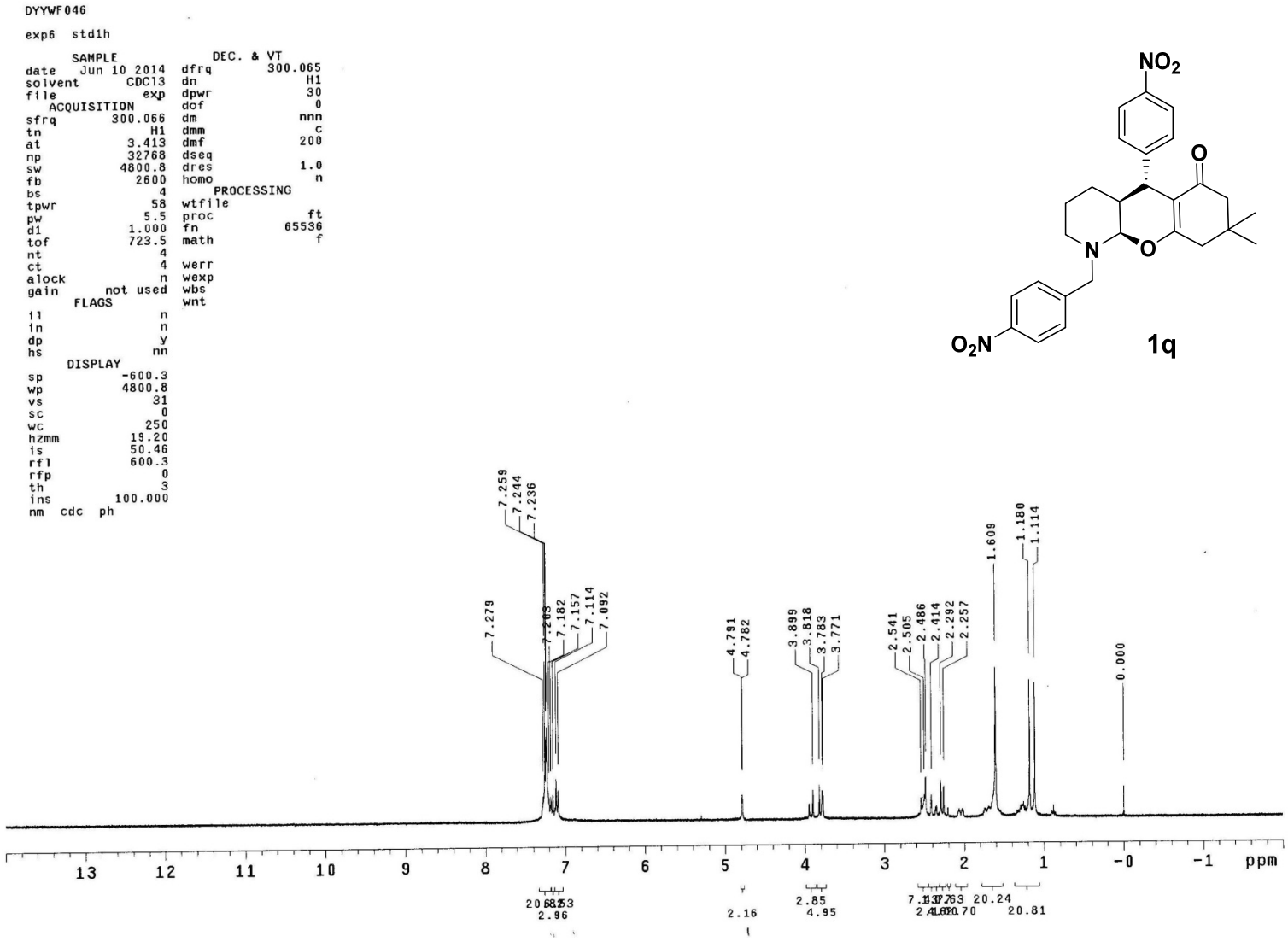

1n
DYYWF015C
exp7 std13c
SAMPLE
date Apr 8 2014
solvent CDCl3
file ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nn
DISPLAY
sp -1156.5
wp 18484.3
vs 24
sc 0
wc 250
hzmm 73.84
is 500.00
rfl 6866.2
rfp 5809.7
th 2
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 9756
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
ft 65536
math f
werr
wexp
wbs
wnt
ppm
162.674
160.378
153.059
143.227
137.934
131.586
128.588
128.416
128.274
127.489
127.145
126.711
123.646
122.629
116.685
115.773
99.616
90.301
77.426
77.000
76.581
59.049
44.193
43.296
41.030
25.434
24.537

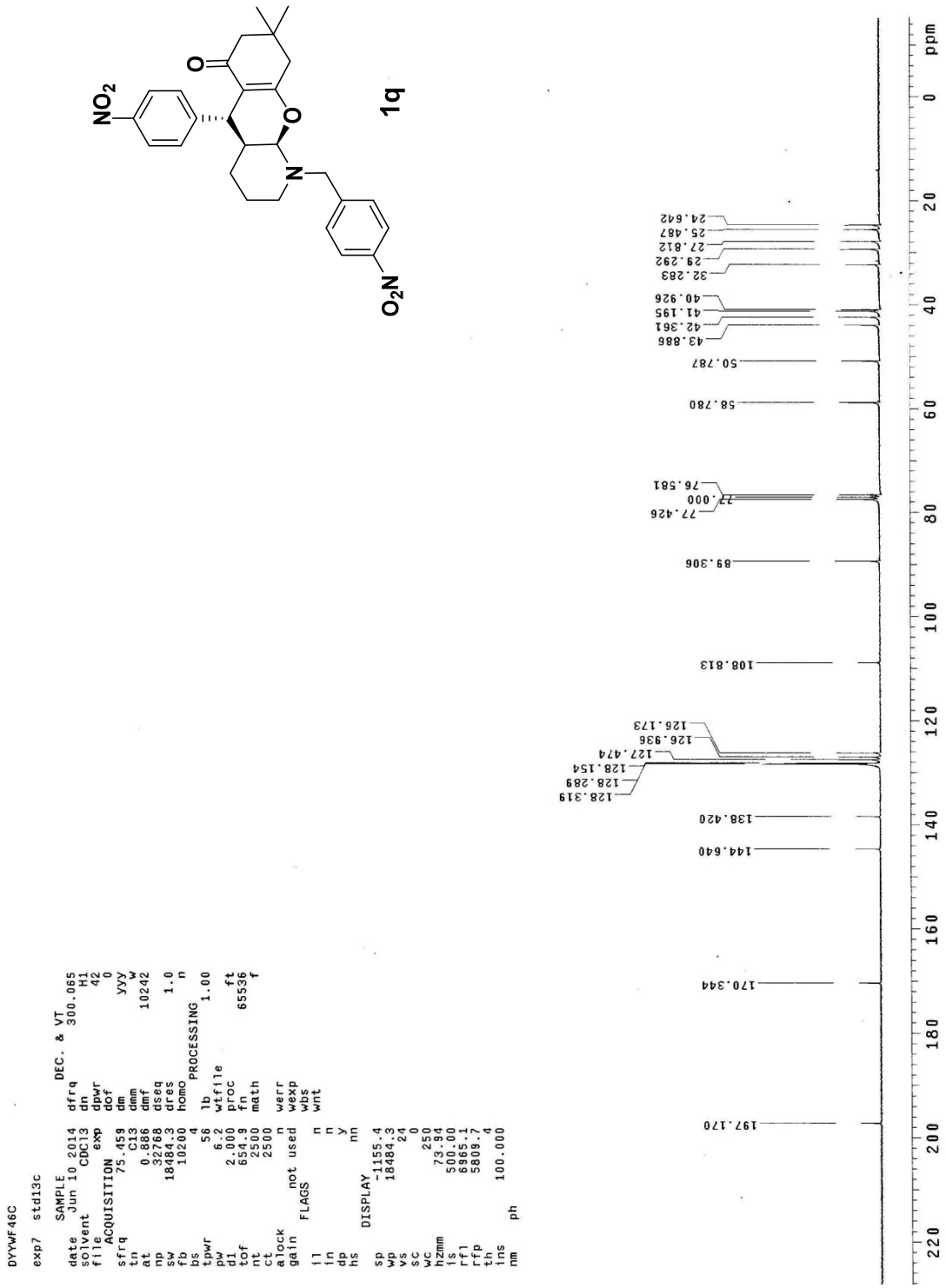

1o
3.069
3.003
2.602
2.046
1.671
1.582
1.260
0.883
0.000
5.007
4.117
4.070
3.990
3.947
6.545
6.553
6.669
6.676
6.698
6.706
7.156
7.178
7.183
7.211
7.248
7.258
7.273
7.282
7.296
7.686
7.716
ppm

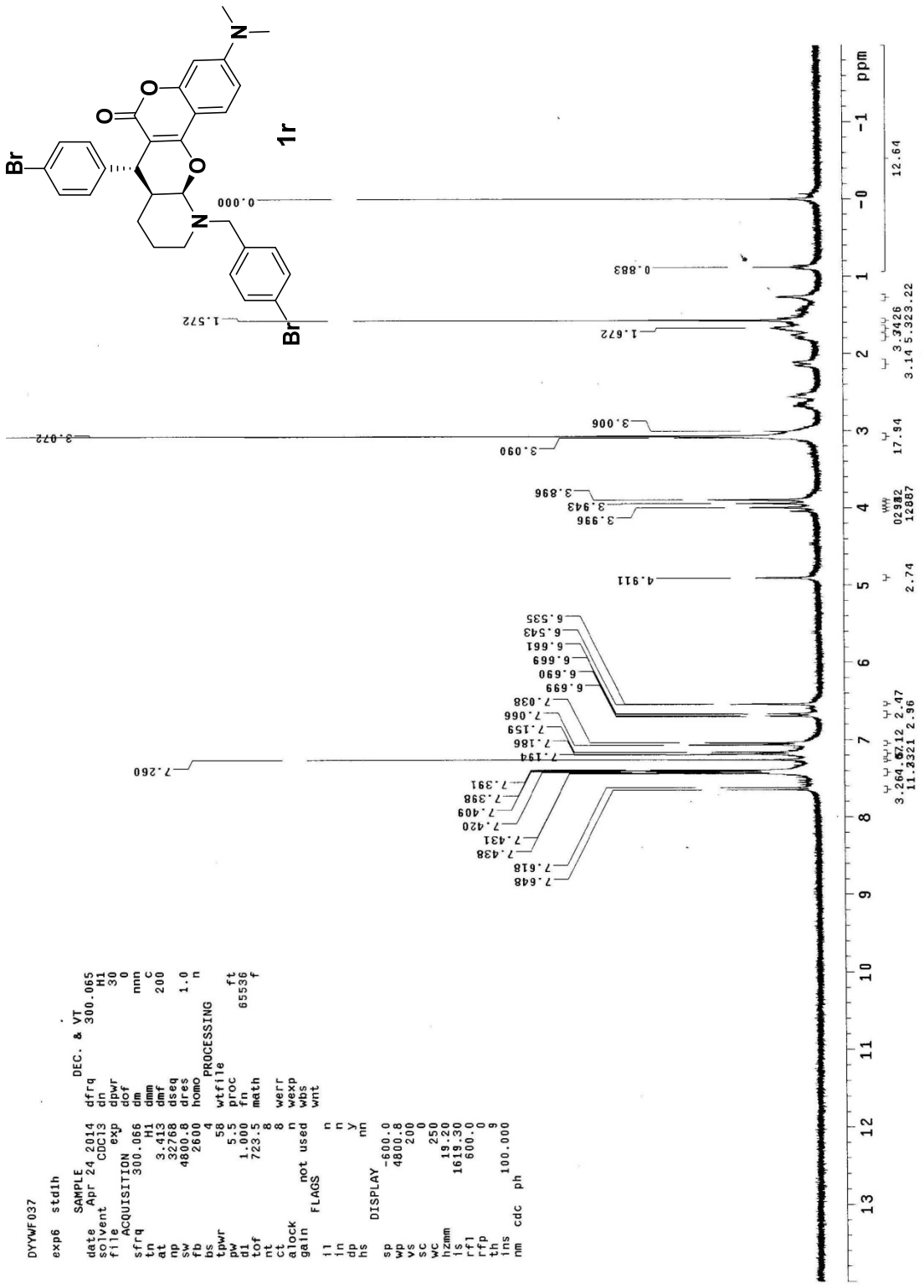

DYYWF011C
exp5 std13c
SAMPLE
date Apr 17 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1157.4
wp 18484.3
vs 30
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6967.2
rfp 5809.7
th 4
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 9756
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
1o
163.625
161.330
155.050
152.882
143.887
138.153
128.433
128.403
128.209
127.544
127.028
126.452
123.260
108.486
104.591
97.944
94.968
89.518
77.414
76.995
76.569
59.021
44.150
43.111
41.205
40.143
25.399
24.599
220
200
180
160
140
120
100
80
60
40
20
0
ppm

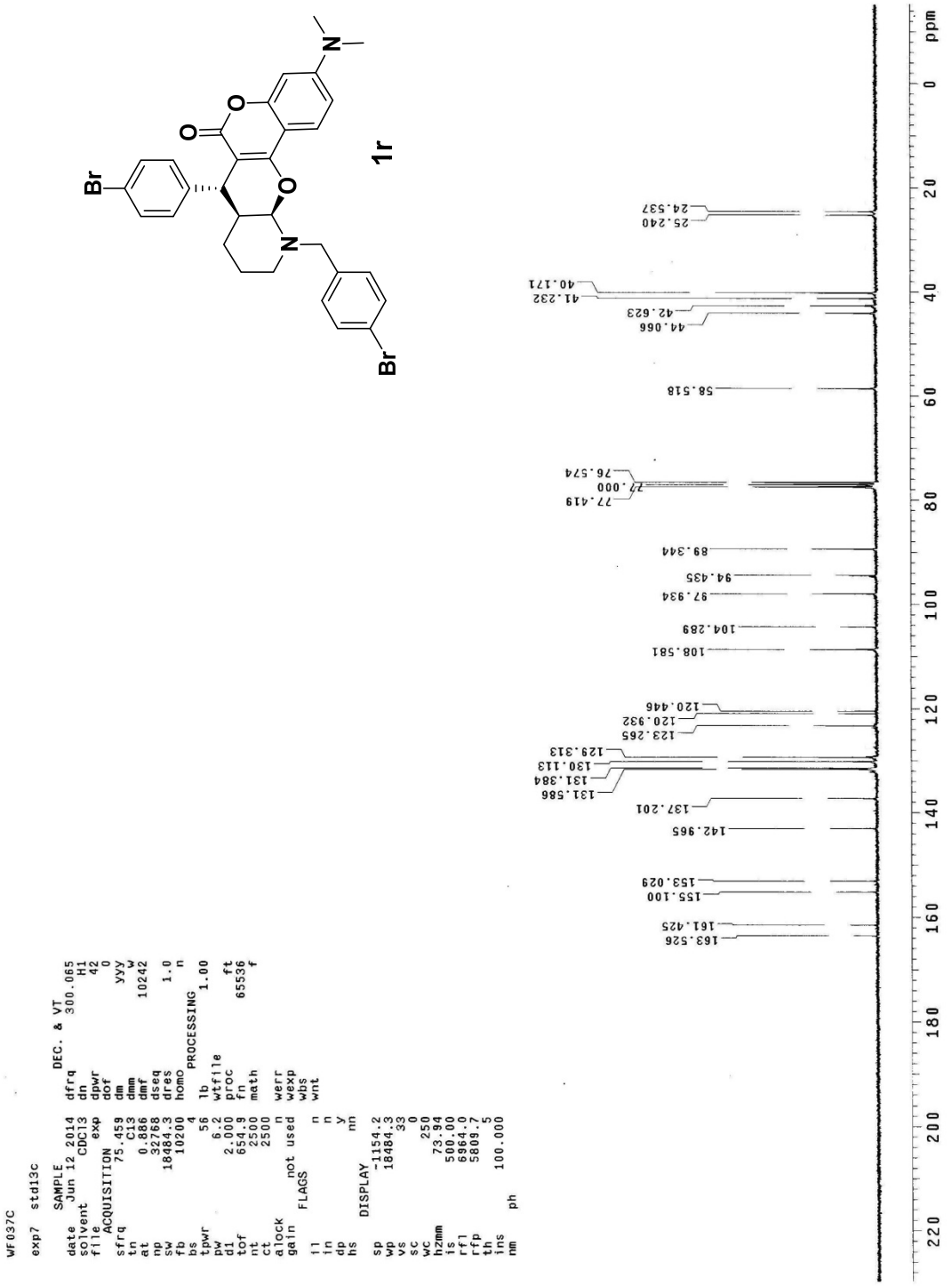

DYYWF039
exp6 std1h
SAMPLE
date Apr 29 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -600.3
wp 4800.8
vs 87
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 600.3
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
OMe
MeO
1p
7.259
6.809
3.778
3.758
1.589
1.171
1.105
0.000
5.21
1.09
5.53
2.62
27.79
19.32
8.74
19.66
ppm


OMe
O
O
N
MeO
1p
DYYWF039C
exp3 std13c
SAMPLE
date Apr 30 2014
solvent CDCl3
file ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 9756
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1154.8
wp 18484.3
vs 47
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6964.5
rfp 5809.7
th 10
ins 100.000
nm ph
197.073
170.083
158.606
157.918
136.857
130.465
129.523
128.386
113.725
113.530
109.074
89.217
77.426
77.007
76.581
58.174
55.169
55.154
50.825
43.737
42.354
41.068
40.313
32.268
29.300
27.812
25.427
24.657
ppm

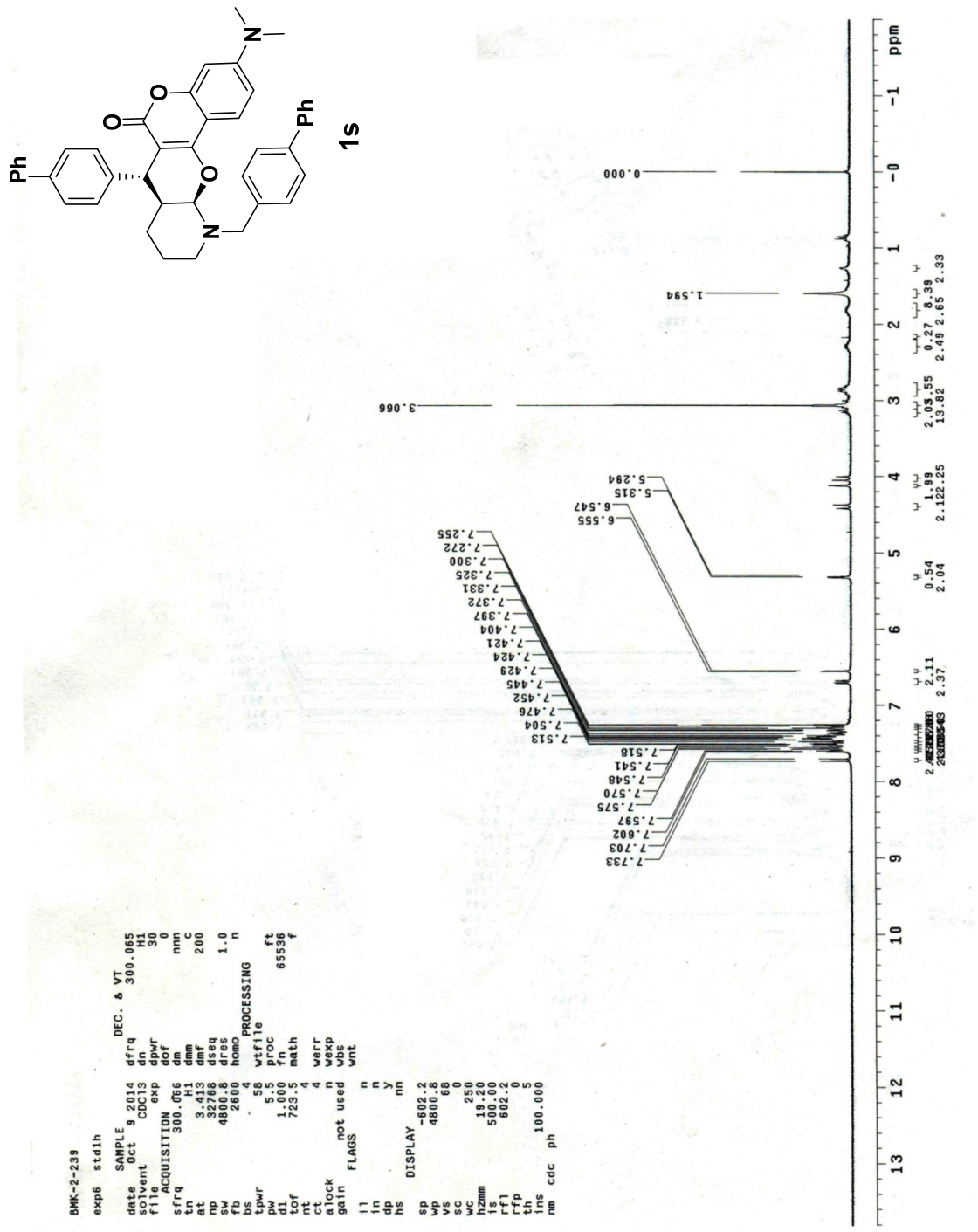

DYYWF046
exp6 std1h
SAMPLE
date Jun 10 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -600.3
wp 4800.8
vs 31
sc 0
wc 250
hzmm 19.20
is 50.46
rfl 600.3
rfp 0
th 3
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
NO2
O
N
O
O2N
1q
7.279
7.259
7.244
7.236
7.203
7.182
7.157
7.114
7.092
4.791
4.782
3.899
3.818
3.783
3.771
2.541
2.505
2.486
2.414
2.292
2.257
1.609
1.180
1.114
0.000
13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
2.96
2.16
4.95
20.24
20.81

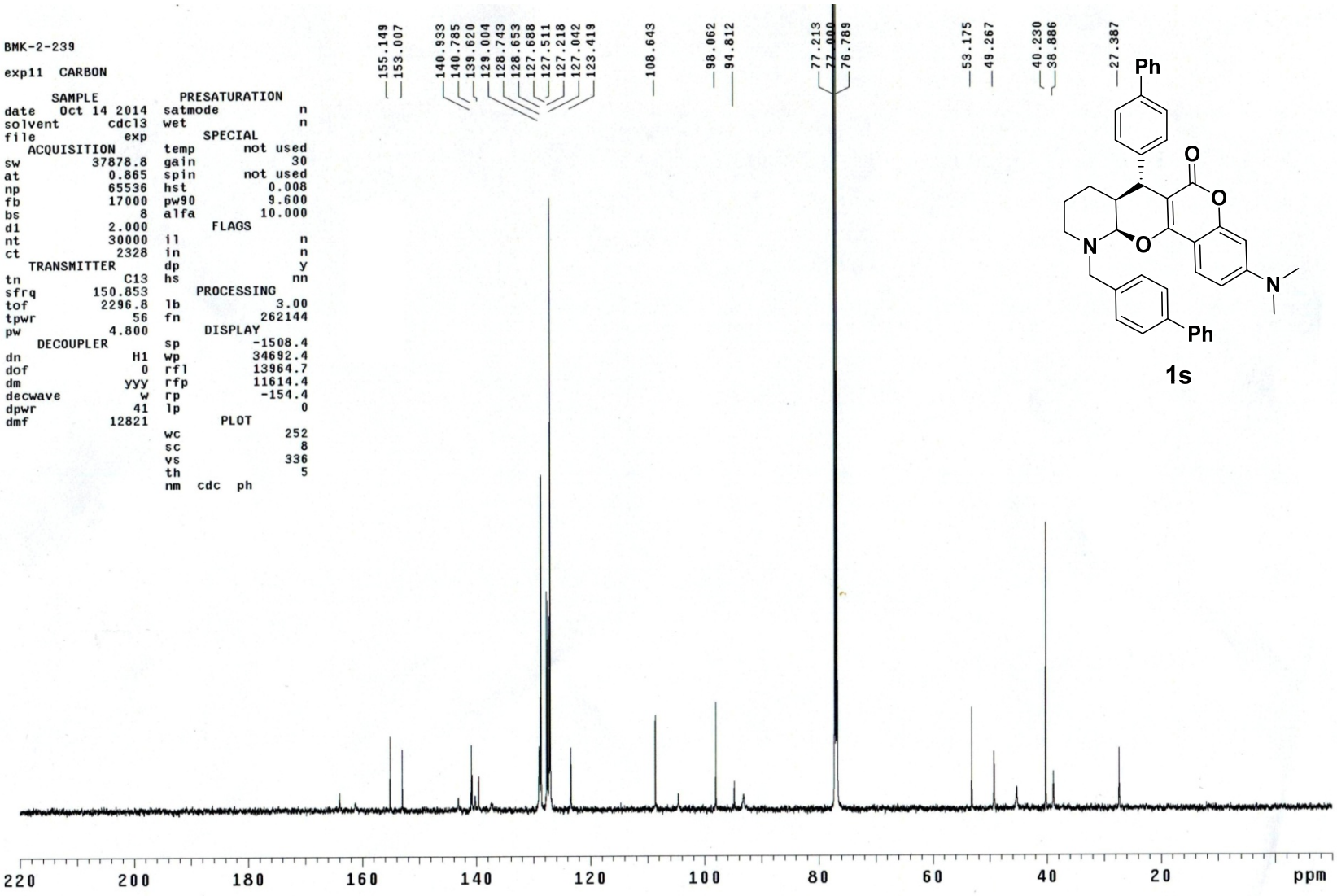

DYYWF46C
exp7 std13c
DEC. & VT
SAMPLE
date Jun 10 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nn
DISPLAY
sp -1155.4
wp 18484.3
vs 24
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6985.1
rfp 5809.7
th 4
ins 100.000
nm ph
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm y
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
O
NO2
O
N
O2N
1q
197.170
170.344
144.640
138.420
128.319
128.289
128.154
127.474
126.936
126.173
108.813
89.306
77.426
77.000
76.581
58.780
50.787
43.886
42.361
41.195
40.926
32.283
29.292
27.812
25.487
24.642
220
200
180
160
140
120
100
80
60
40
20
0
ppm

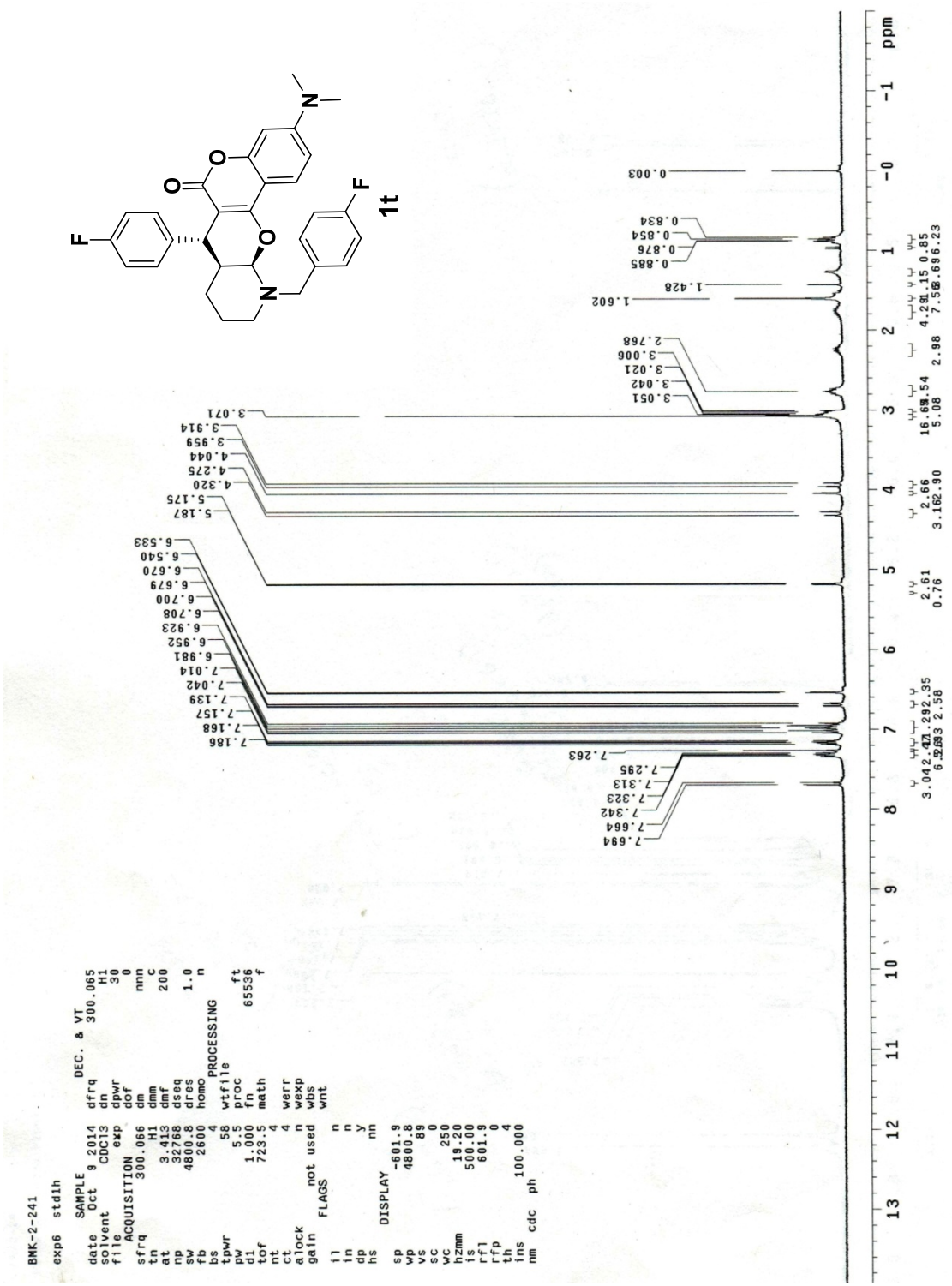

1r
Br
Br
DYYWF037
exp6 stdih
SAMPLE
date Apr 24 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
dl 1.000
tof 723.5
nt 8
ct 8
alock n
gain not used
FLAGS
DISPLAY
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
sp -600.0
wp 4800.8
vs 200
sc 0
wc 250
hzmm 19.20
is 1619.30
rfl 600.0
rfp 0
th 9
ins 100.000
nm cdc ph
0.000
0.883
1.572
1.672
3.006
3.090
3.072
3.896
3.943
3.996
4.911
6.535
6.543
6.661
6.669
6.690
6.699
7.038
7.066
7.159
7.186
7.194
7.260
7.391
7.398
7.409
7.420
7.431
7.438
7.618
7.648
ppm
12.64
3.3426
3.14 5.323.22
17.94
02982
12887
2.74
3.264 67.12 2.47
11.8321 2.96

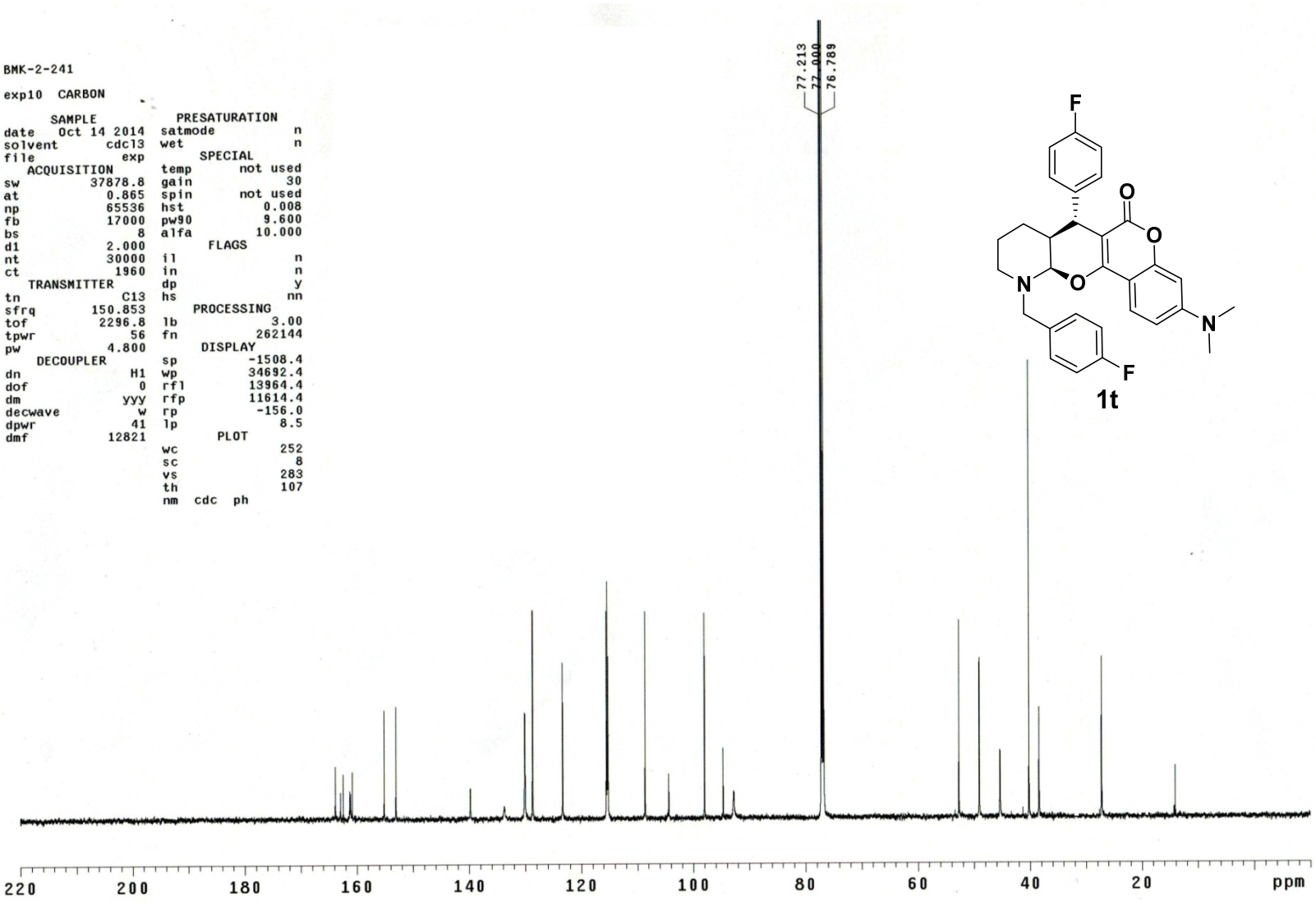


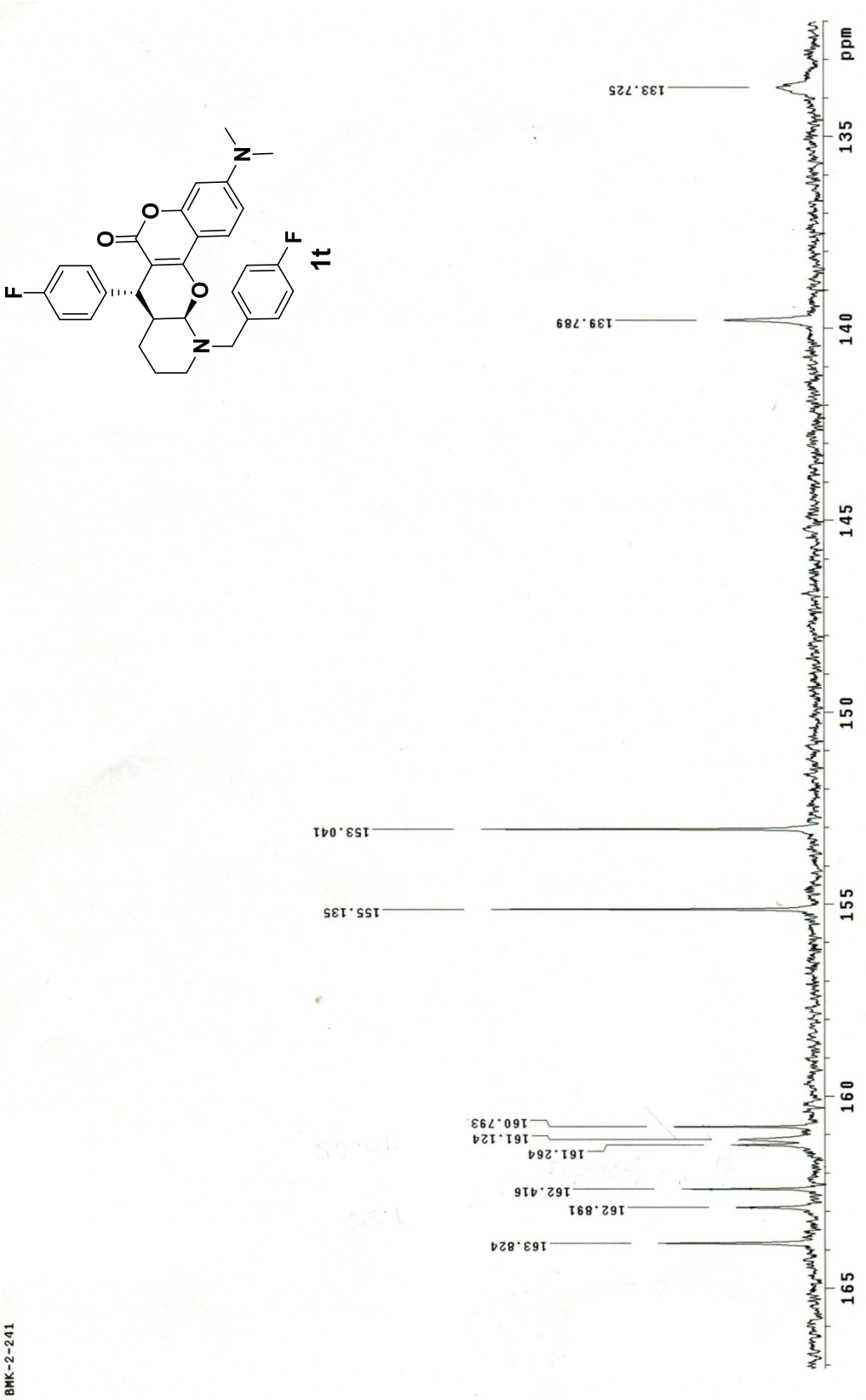

```
wf037C
exp7  std13c
                                DEC. & VT
      SAMPLE                    dfrq      300.065
date  Jun 12 2014               dn             H1
solvent      CDCl3              dpwr           42
file      exp                   dof             0
ACQUISITION                     dm            yyy
sfrq      75.459                dmm             w
tn            C13               dmf         10242
at          0.886               dseq
np          32768               dres          1.0
sw       18484.3                homo            n
fb         10200                PROCESSING
bs             4                lb             1.00
tpwr          56                wtfile
pw           6.2                proc            ft
d1         2.000                fn          65536
tof        654.9                math            f
nt          2500
ct          2500
alock          n                werr
gain       not used             wexp
           FLAGS                wbs
il                n             wnt
in                n
dp                y
hs               nn
                DISPLAY
sp          -1154.2
wp          18484.3
vs               33
sc                0
wc              250
hzmm          73.94
is           500.00
rfl         6964.0
rfp         5809.7
th                5
ins          100.000
nm    ph
```

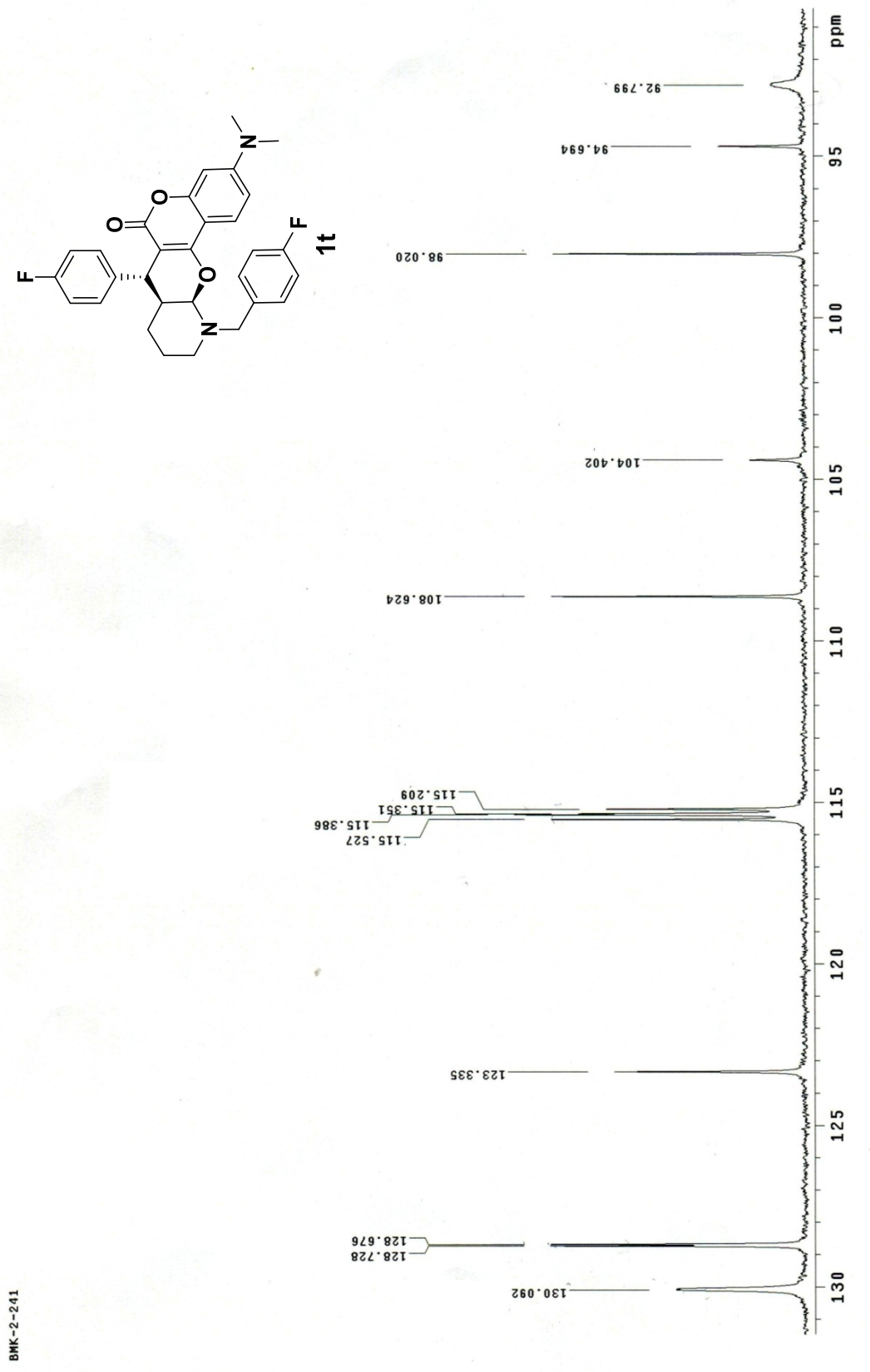

1s
Ph
Ph
O
O
O
N
N
BMK-2-239
exp6 std1h
SAMPLE
date Oct 9 2014
solvent CDCl3
file AQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
ll n
ln n
dp y
hs nn
DISPLAY
sp -602.2
wp 4800.8
vs 68
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 602.2
rfp 0
th 5
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
0.000
1.594
3.066
5.294
5.315
6.547
6.555
7.255
7.272
7.300
7.325
7.331
7.372
7.397
7.404
7.421
7.424
7.429
7.445
7.452
7.476
7.504
7.513
7.518
7.541
7.548
7.570
7.575
7.597
7.602
7.703
7.733
ppm

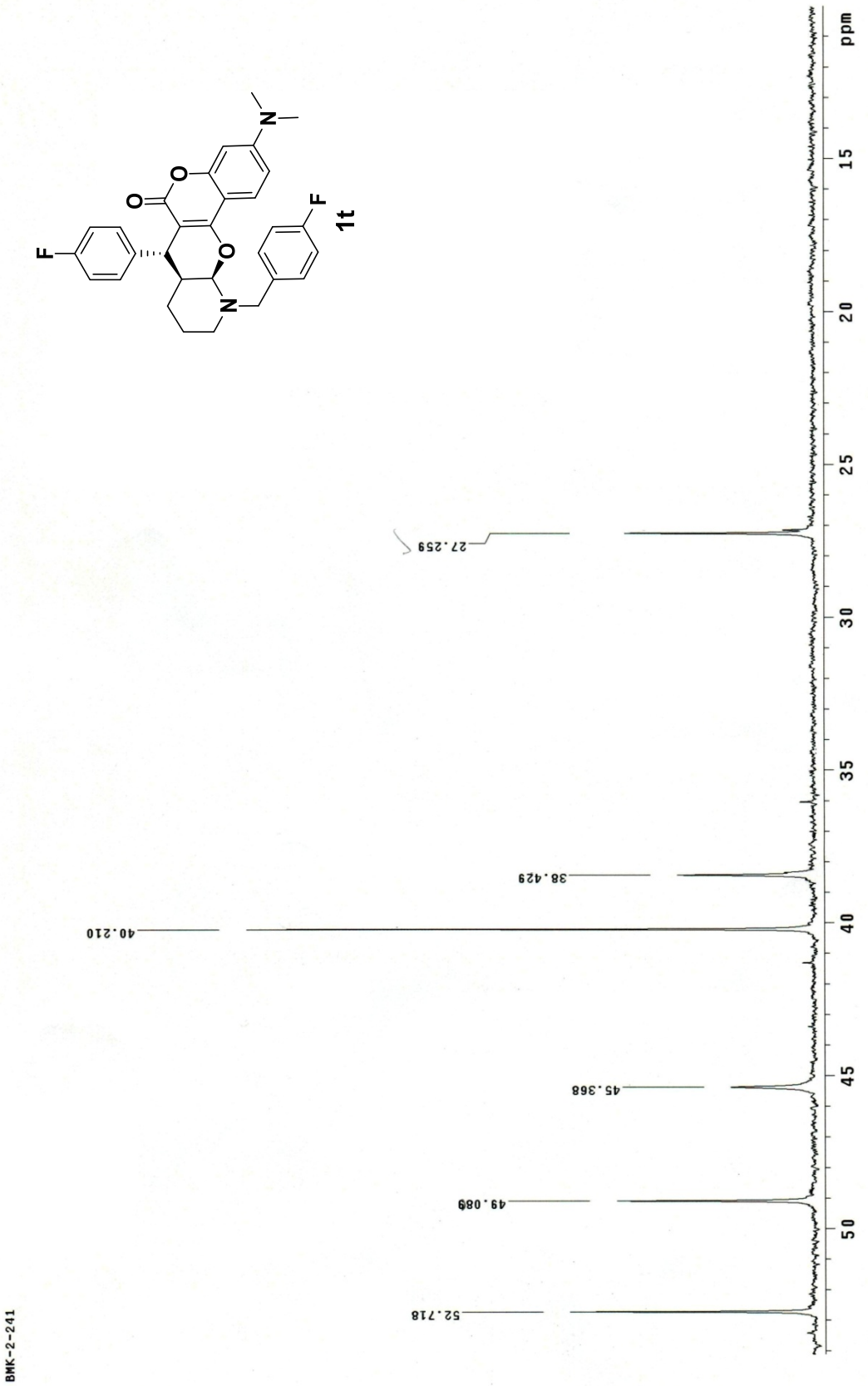

BMK-2-239
exp11 CARBON
SAMPLE
date Oct 14 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 2328
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.8
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13964.7
rfp 11614.4
rp -154.4
lp 0
PLOT
wc 252
sc 8
vs 336
th 5
nm cdc ph
155.149
153.007
140.933
140.785
139.620
129.004
128.743
128.653
127.688
127.511
127.218
127.042
123.419
108.643
98.062
94.812
77.213
77.000
76.789
53.175
49.267
40.230
38.886
27.387
Ph
O
O
N
O
N
Ph
1s
220
200
180
160
140
120
100
80
60
40
20
ppm

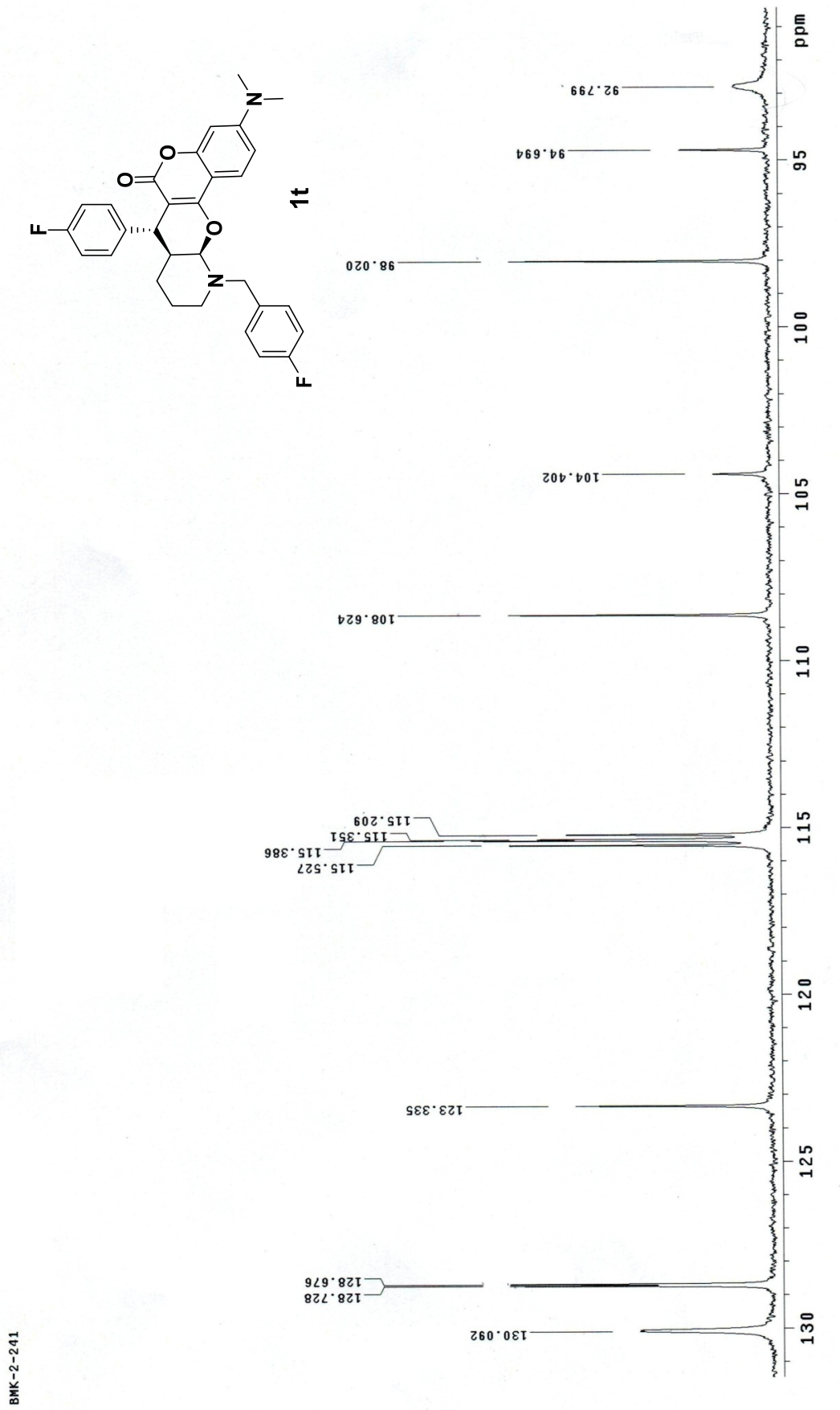

1t
F
F
O
O
O
N
N
BMK-2-241
exp6 stdih
SAMPLE
date Oct 9 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -601.9
wp 4800.8
vs 89
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 601.9
rfp 0
th 4
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
0.003
0.834
0.854
0.876
0.885
1.428
1.602
2.768
3.006
3.021
3.042
3.051
3.071
3.914
3.959
4.044
4.275
4.320
5.175
5.187
6.533
6.540
6.670
6.679
6.700
6.708
6.923
6.952
6.981
7.014
7.042
7.139
7.157
7.168
7.186
7.263
7.295
7.313
7.323
7.342
7.664
7.694
ppm

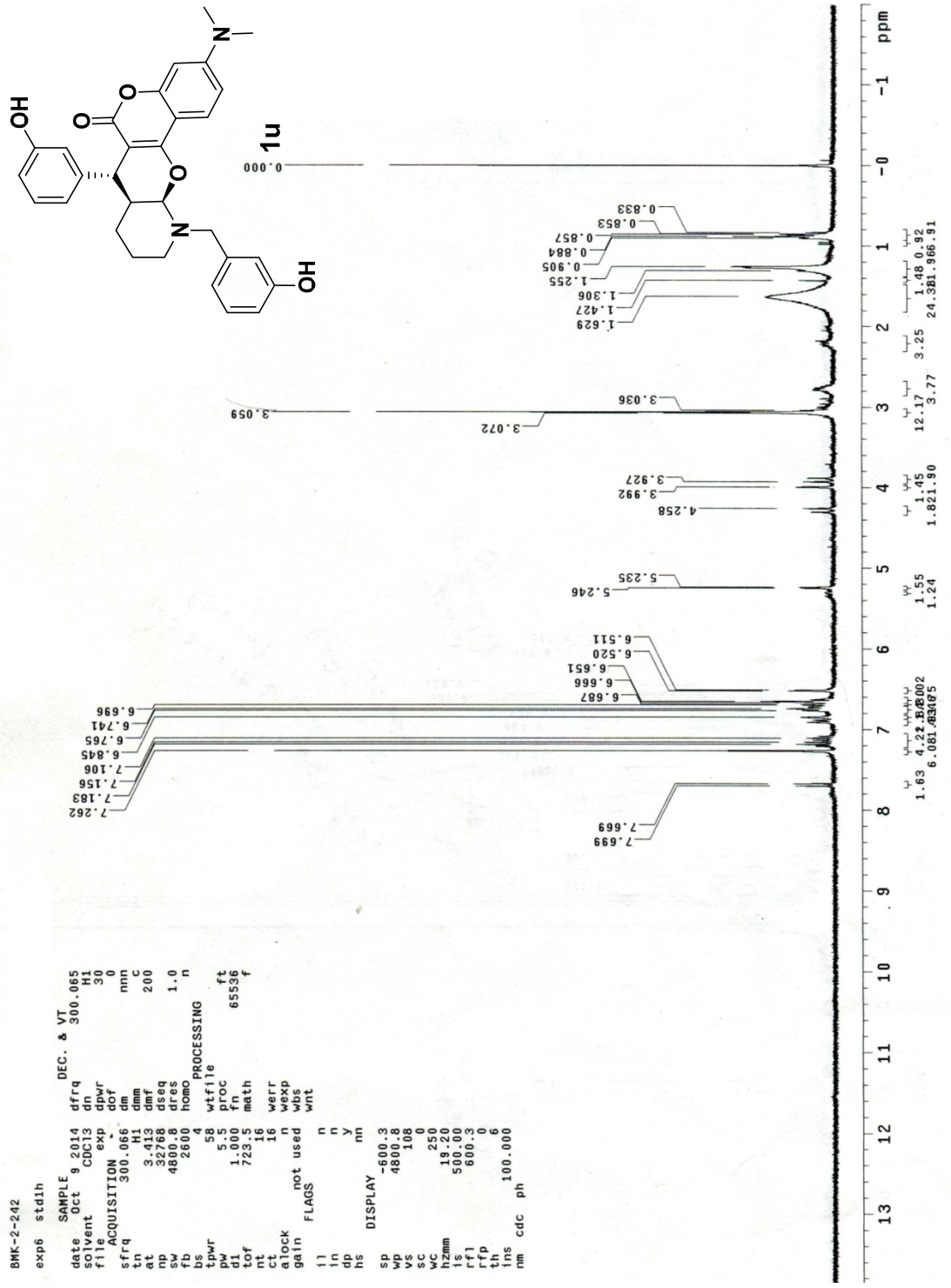

BMK-2-241
exp10 CARBON
SAMPLE
date Oct 14 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 1960
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.8
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13964.4
rfp 11614.4
rp -156.0
lp 8.5
PLOT
wc 252
sc 8
vs 283
th 107
nm cdc ph
77.213
77.000
76.789
F
O
O
N
O
N
F
1t
220
200
180
160
140
120
100
80
60
40
20
ppm

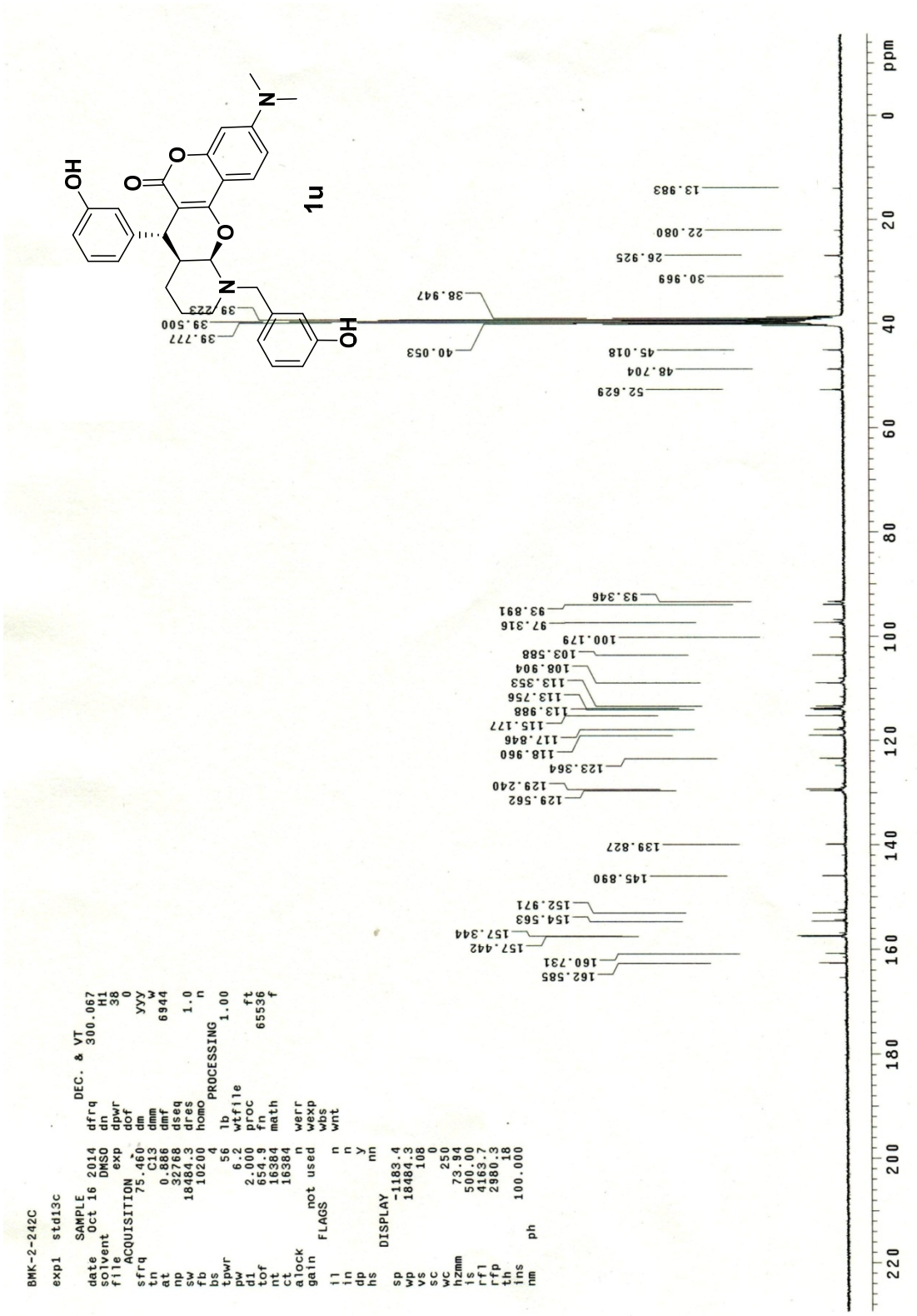

1t
F
F
O
O
O
N
N
BMK-2-241
ppm
135
140
145
150
155
160
165
133.725
139.789
153.041
155.135
160.793
161.124
161.264
162.416
162.891
163.824


1t
92.799
94.694
98.020
104.402
108.624
115.209
115.351
115.386
115.527
123.335
128.676
128.728
130.092
ppm
95
100
105
110
115
120
125
130
BMK-2-241

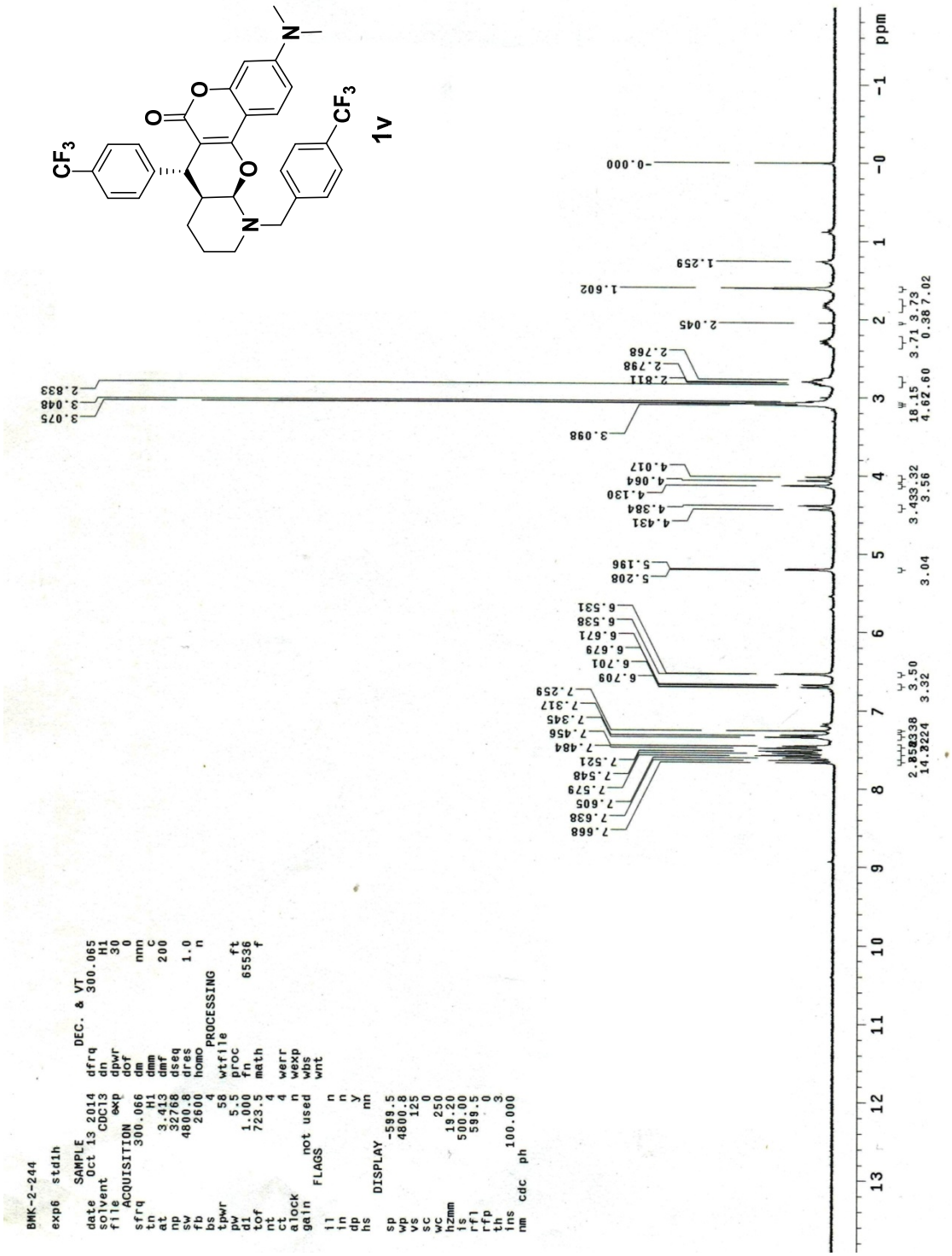

BMK-2-241
52.718
49.089
45.368
40.210
38.429
27.259
ppm
1t

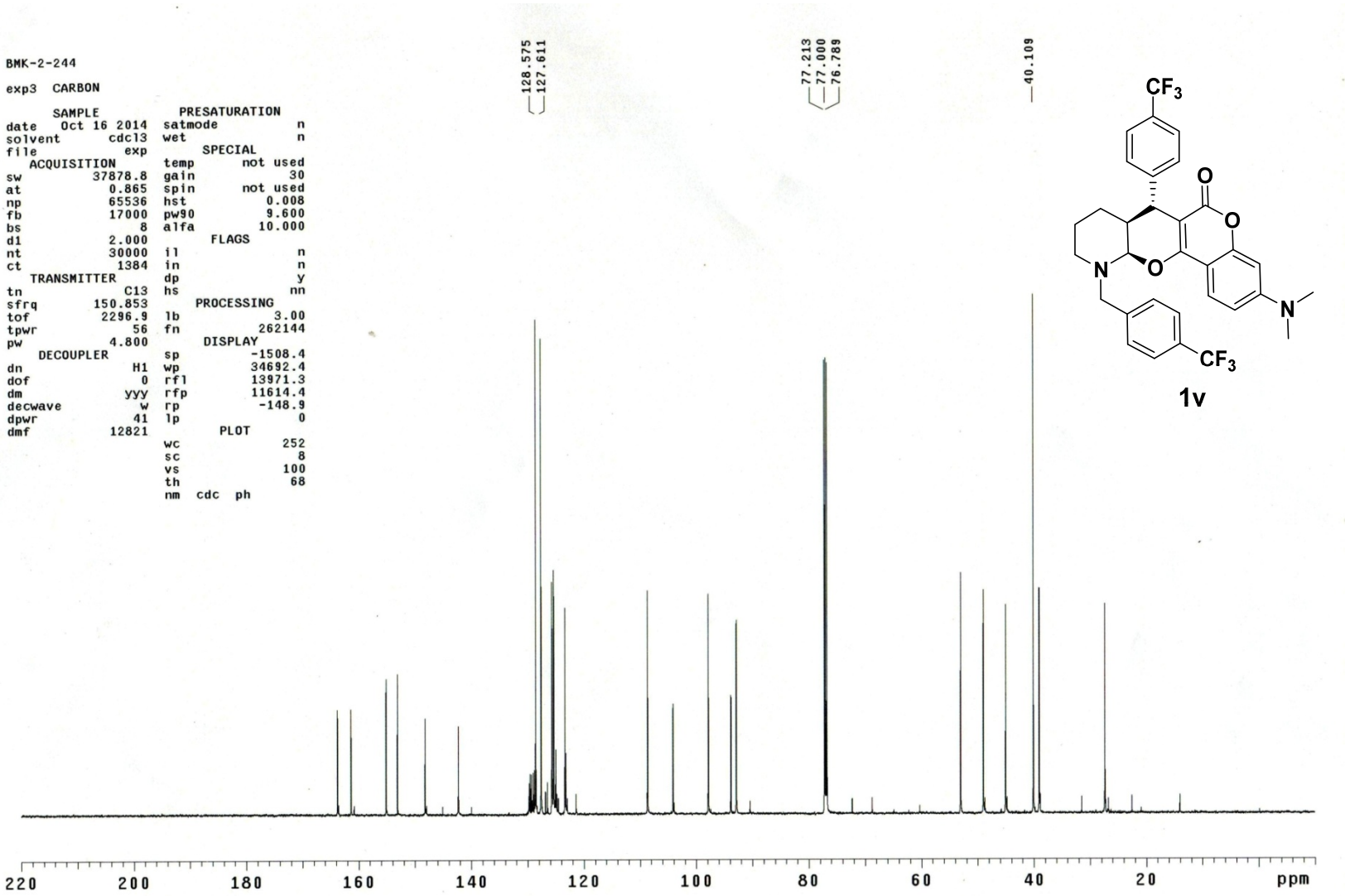

1t
BMK-2-241
ppm
92.799
94.694
98.020
104.402
108.624
115.209
115.351
115.386
115.527
123.335
128.676
128.728
130.092

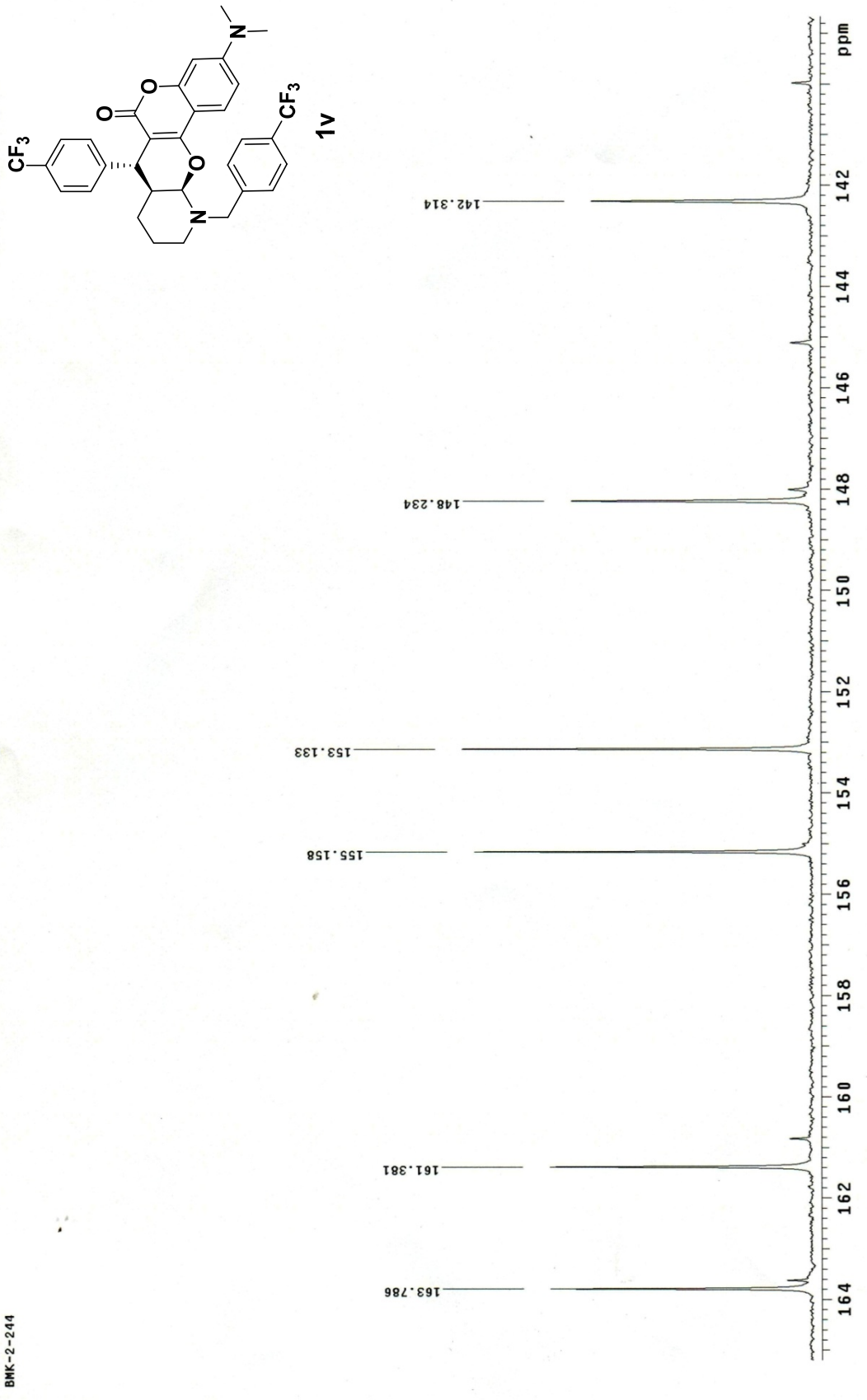

1u
OH
O
O
N
OH
0.000
0.833
0.853
0.857
0.884
0.905
1.255
1.306
1.427
1.629
3.036
3.072
3.059
3.927
3.992
4.258
5.235
5.246
6.511
6.520
6.651
6.666
6.687
7.669
7.699
6.696
6.741
6.765
6.845
7.106
7.156
7.183
7.262
ppm

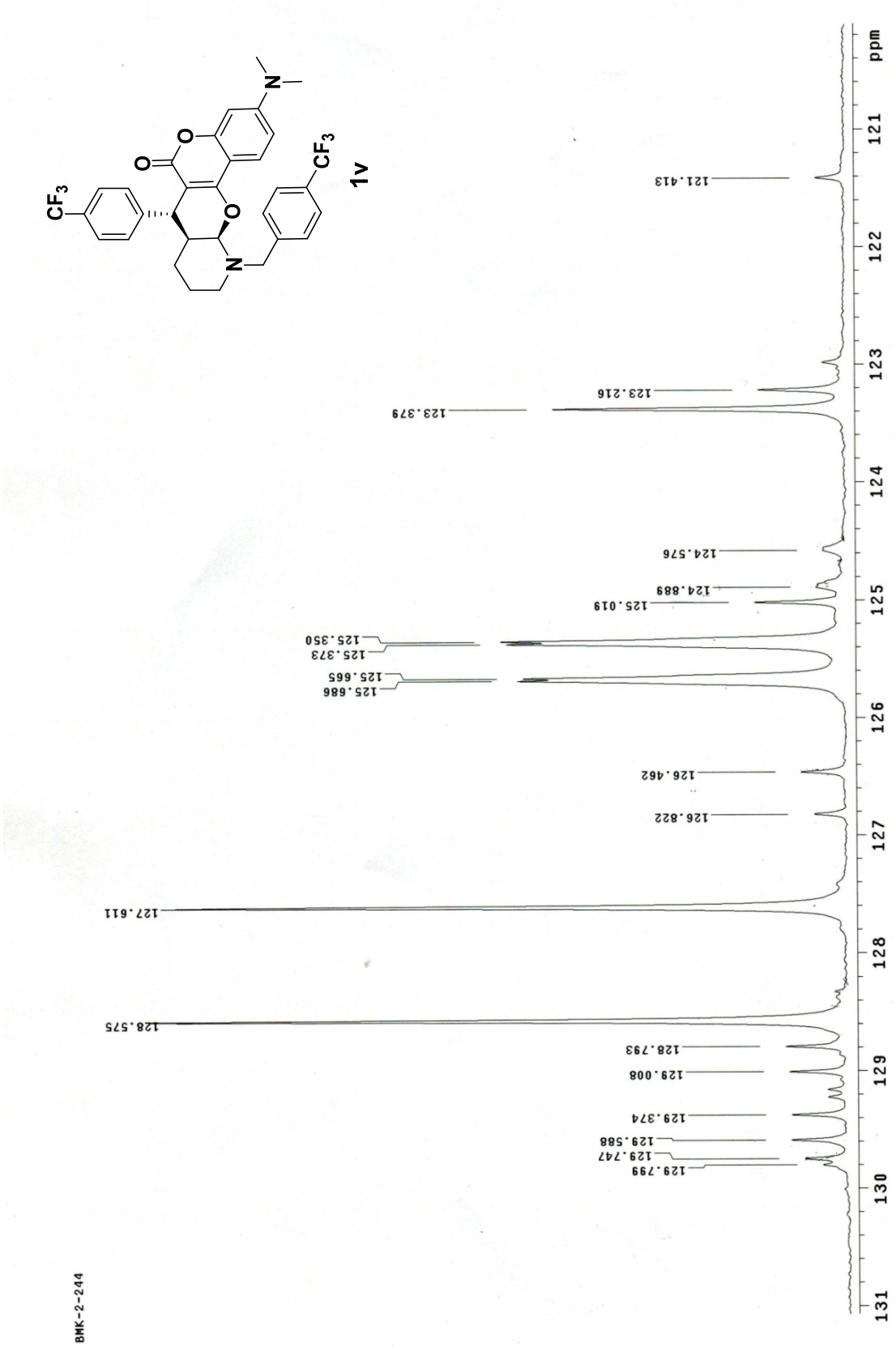


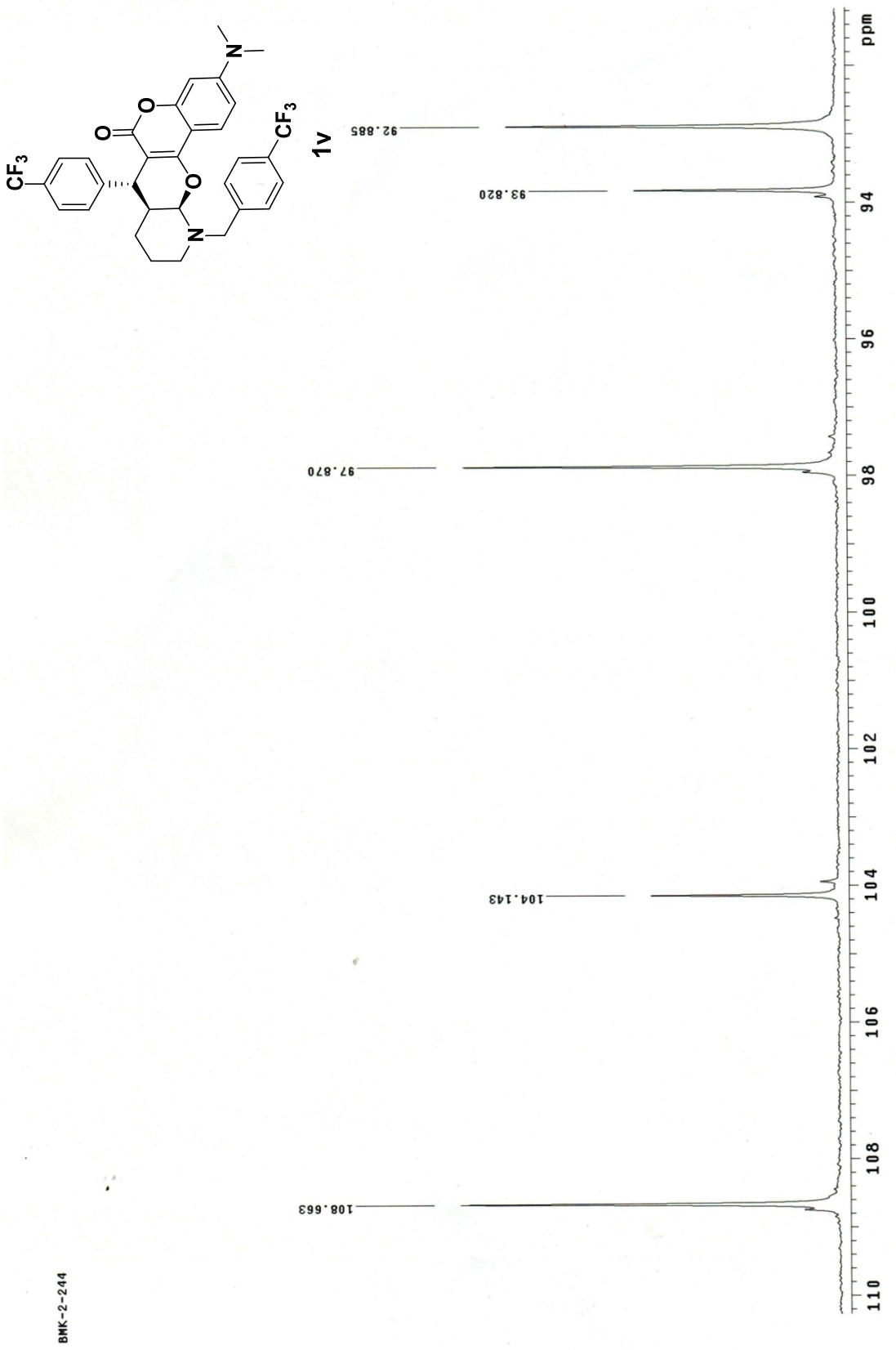

```
BMK-2-242C

exp1  std13c

      SAMPLE                DEC. & VT
date  Oct 16 2014  dfrq    300.067
solvent       DMSO  dn          H1
file          exp   dpwr        38
ACQUISITION         dof         0
sfrq      75.460    dm          yyy
tn            C13   dmm         w
at          0.886   dmf         6944
np          32768   dseq
sw        18484.3   dres        1.0
fb          10200   homo        n
bs              4         PROCESSING
tpwr           56   lb          1.00
pw            6.2   wtfile
d1          2.000   proc        ft
tof         654.9   fn          65536
nt          16384   math        f
ct          16384
alock           n   werr
gain      not used  wexp
                    wbs
FLAGS               wnt
il              n
in              n
dp              y
hs             nn
DISPLAY
sp        -1183.4
wp        18484.3
vs            108
sc              0
wc            250
hzmm        73.94
is          500.00
rfl        4163.7
rfp        2980.3
th              18
ins       100.000
nm    ph
```

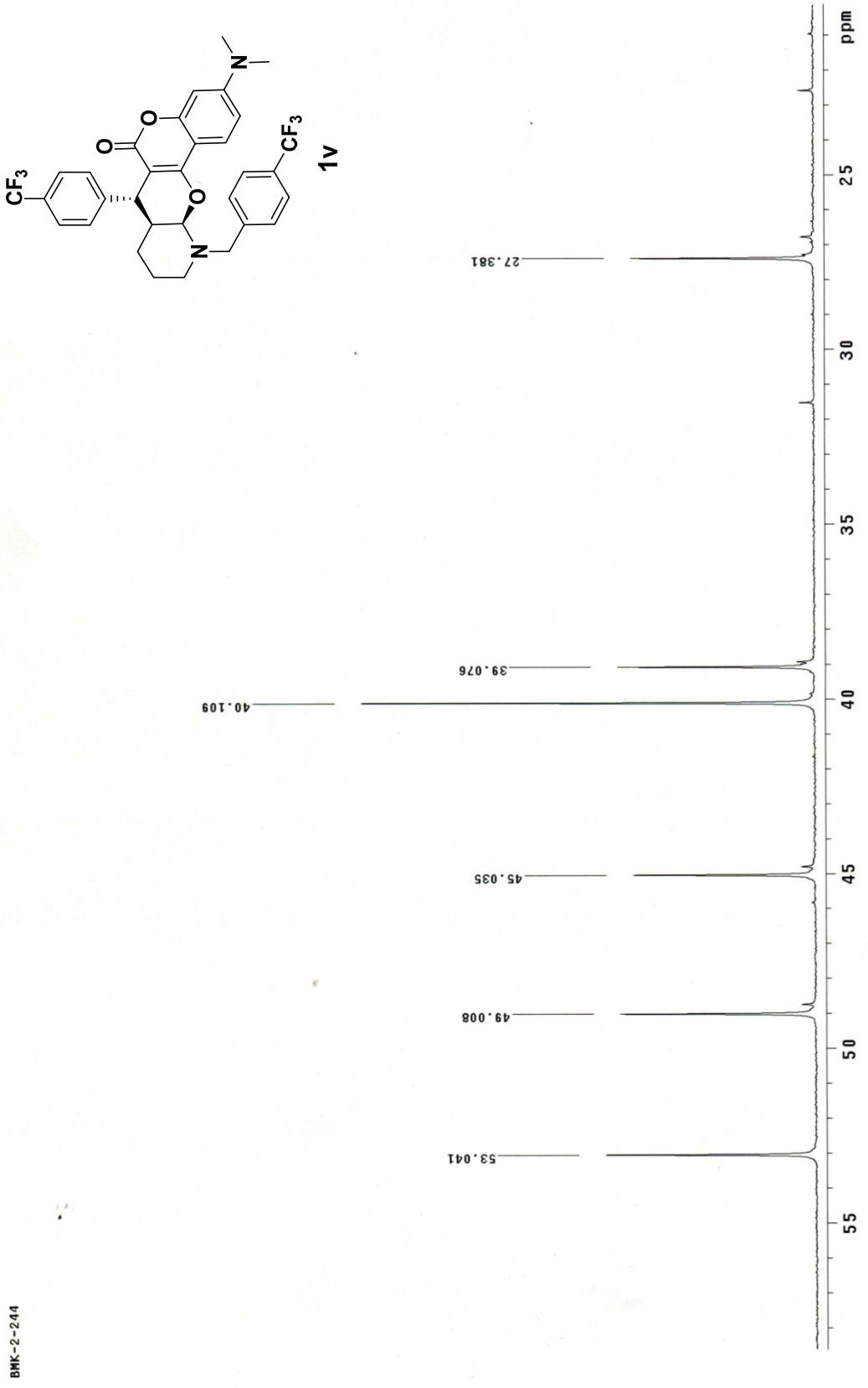

1v
ppm

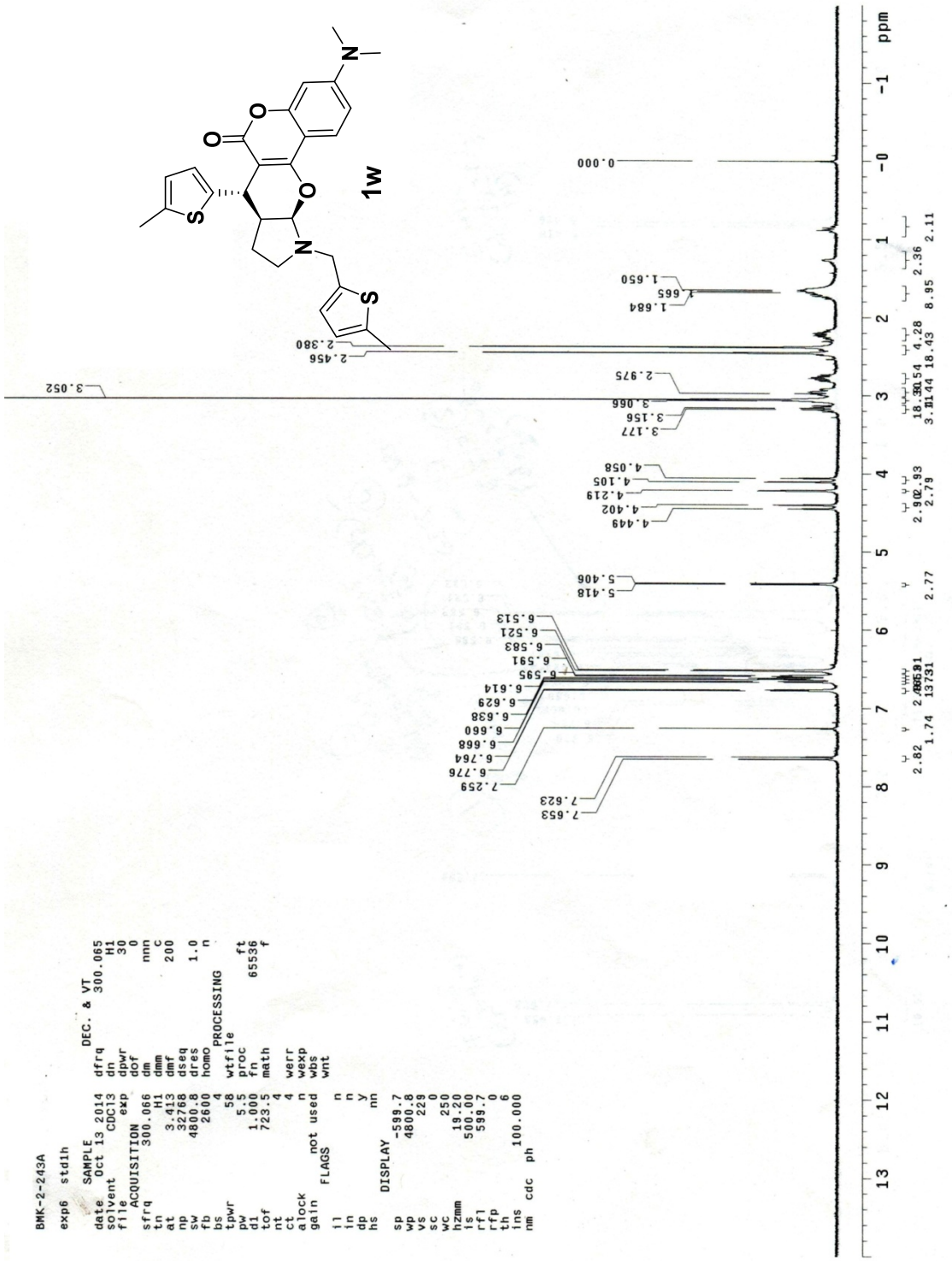

BMK-2-244
exp3 CARBON
SAMPLE
date Oct 16 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 1384
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.9
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13971.3
rfp 11614.4
rp -148.9
lp 0
PLOT
wc 252
sc 8
vs 100
th 68
nm cdc ph
128.575
127.611
77.213
77.000
76.789
40.109
CF3
O
O
N
O
N
CF3
1v
220
200
180
160
140
120
100
80
60
40
20
ppm

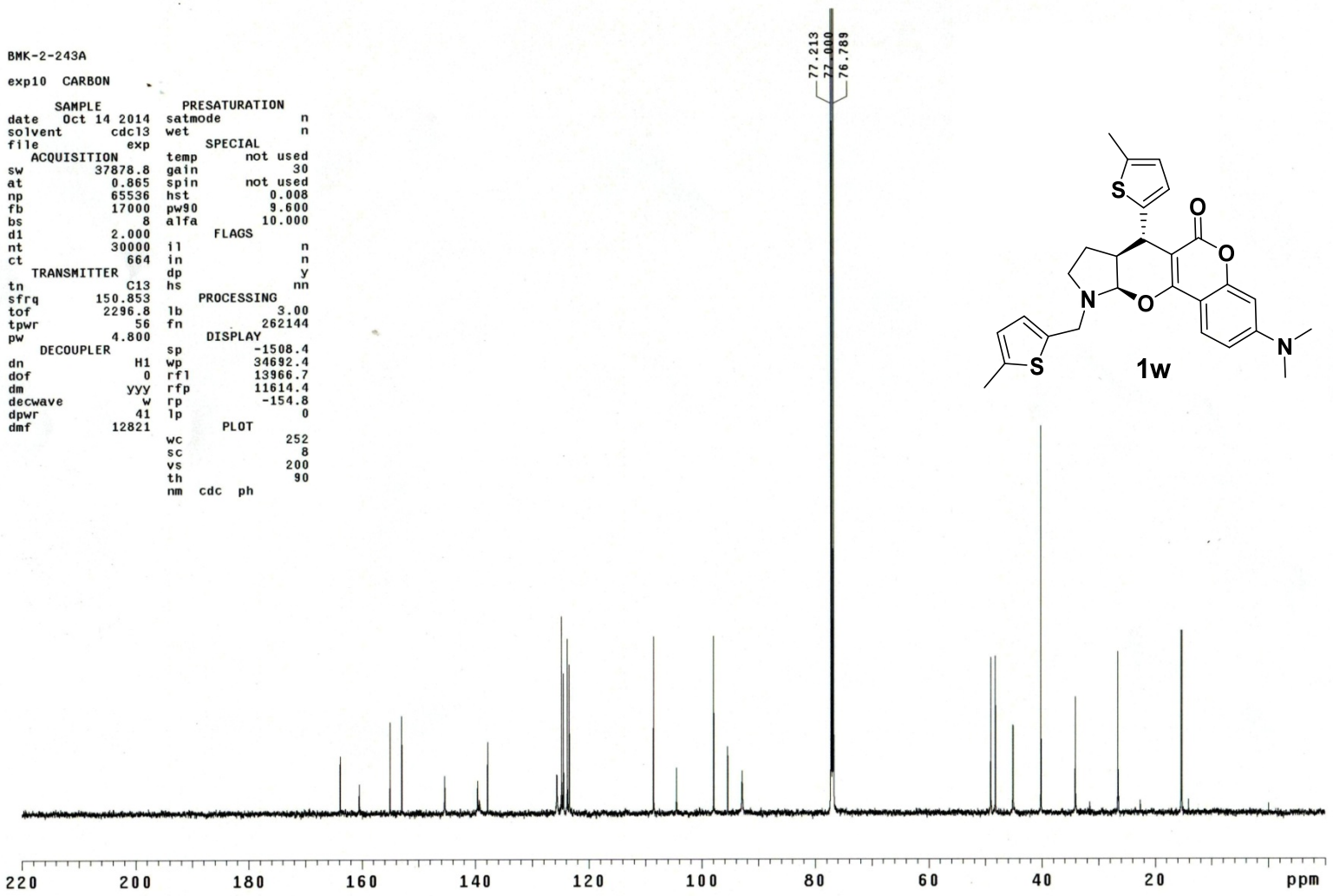

1v
142.314
148.234
153.133
155.158
161.381
163.786
ppm
BMK-2-244

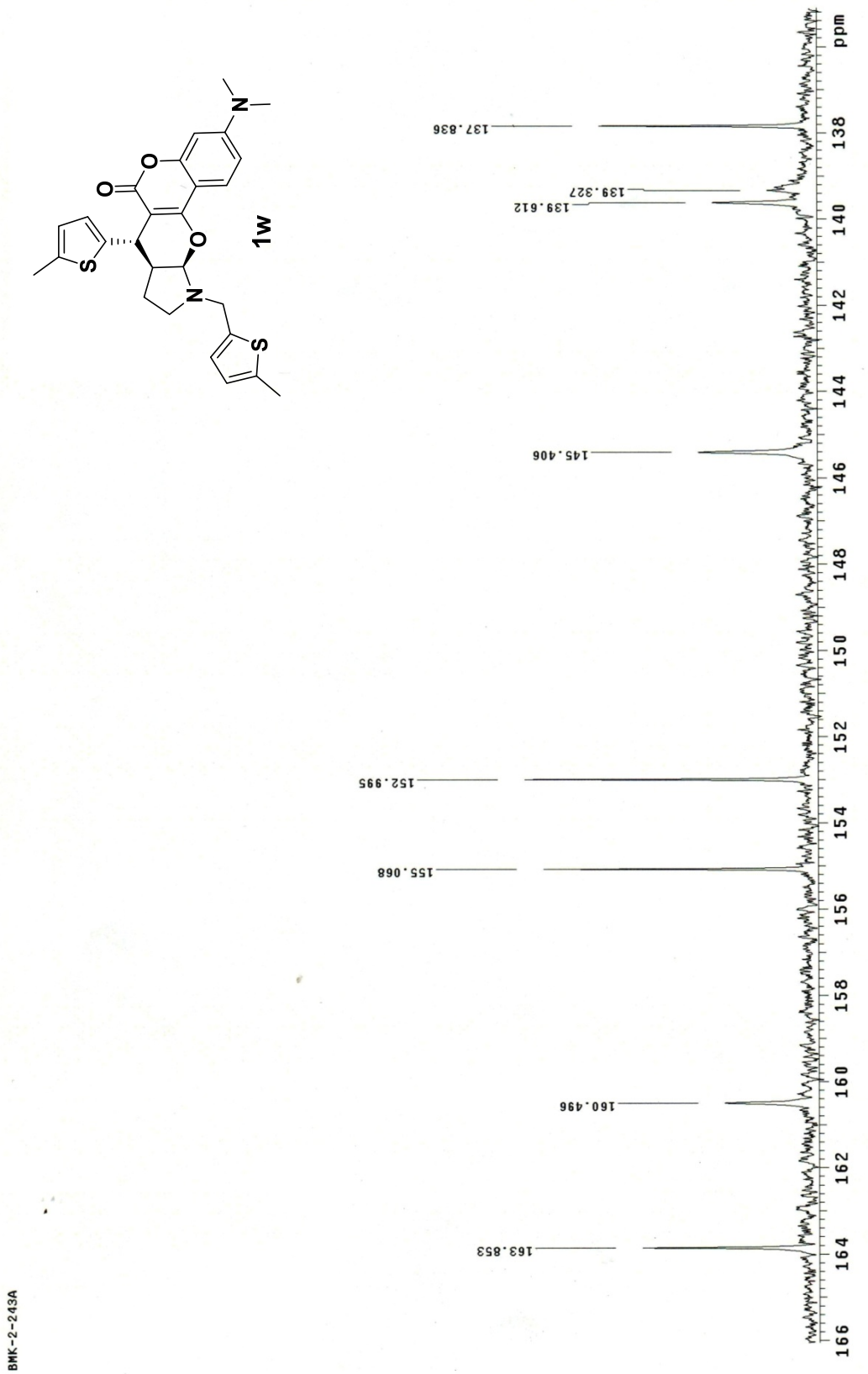

1v
BMK-2-244
121.413
123.379
123.216
124.576
124.889
125.019
125.373
125.350
125.686
125.665
126.462
126.822
127.611
128.575
128.793
129.008
129.374
129.588
129.747
129.799
ppm

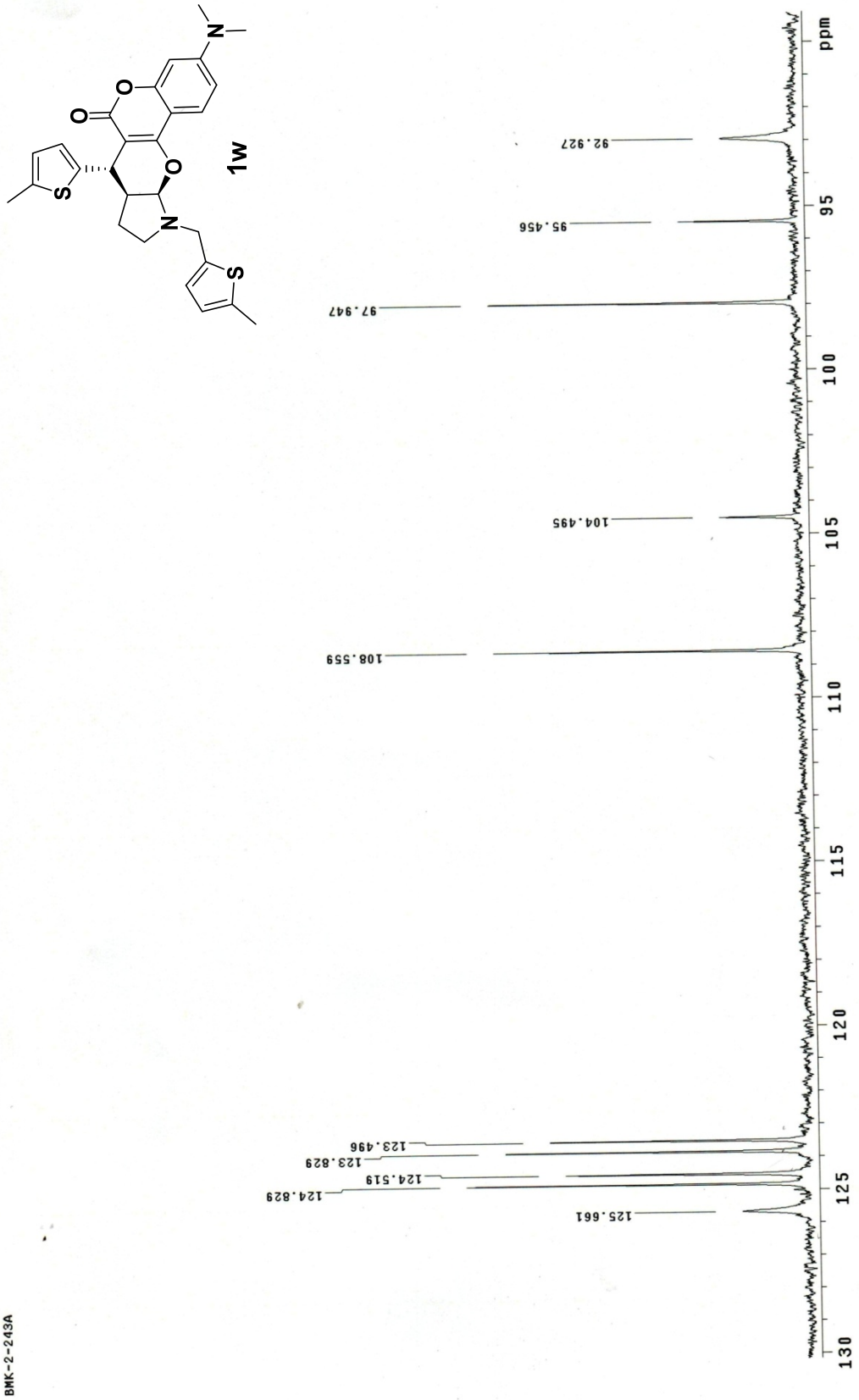

1v
92.885
93.820
97.870
104.143
108.663
ppm
94
96
98
100
102
104
106
108
110
BMK-2-244

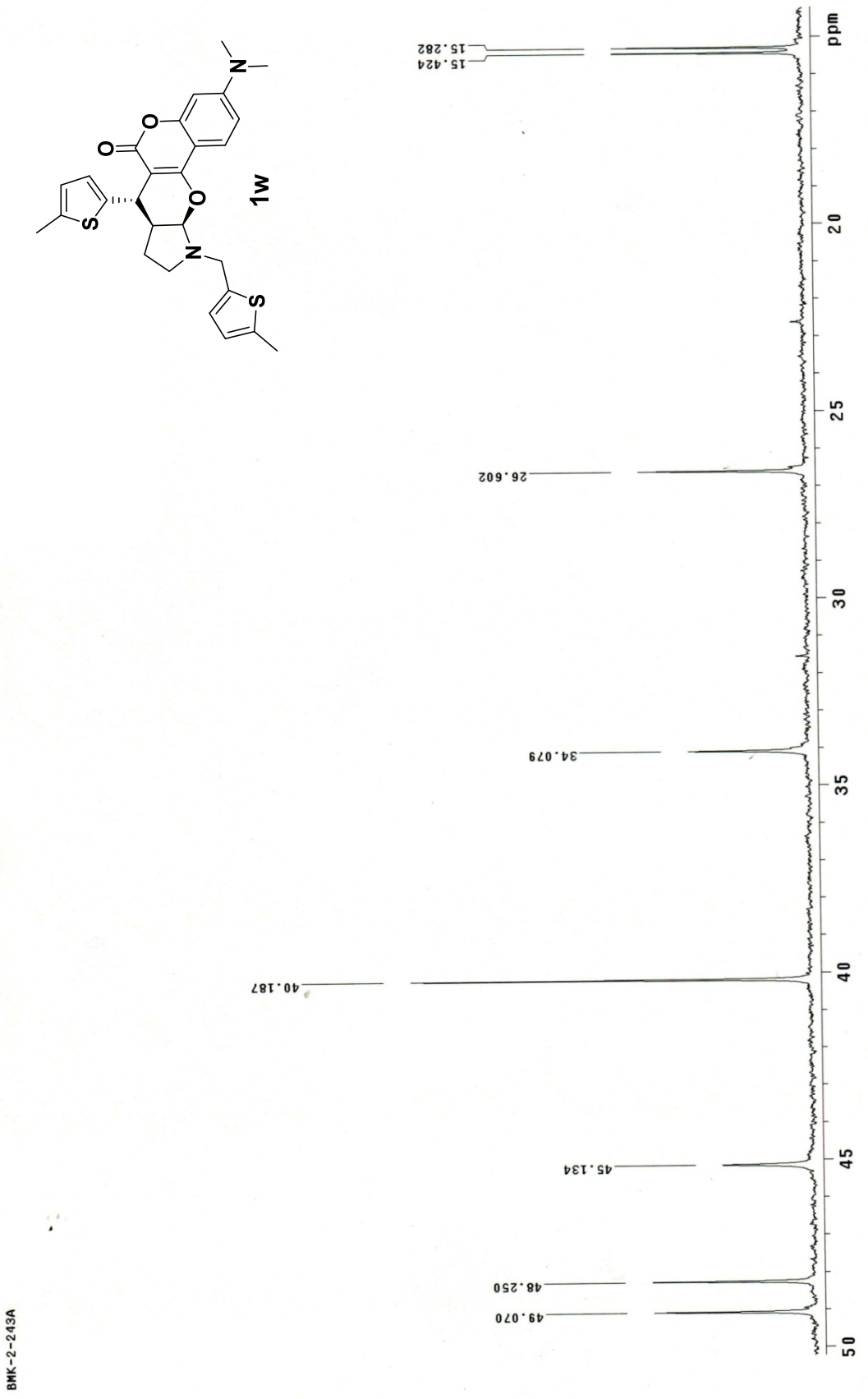

1v
27.381
39.076
40.109
45.035
49.008
53.041
ppm
25
30
35
40
45
50
55
BMK-2-244

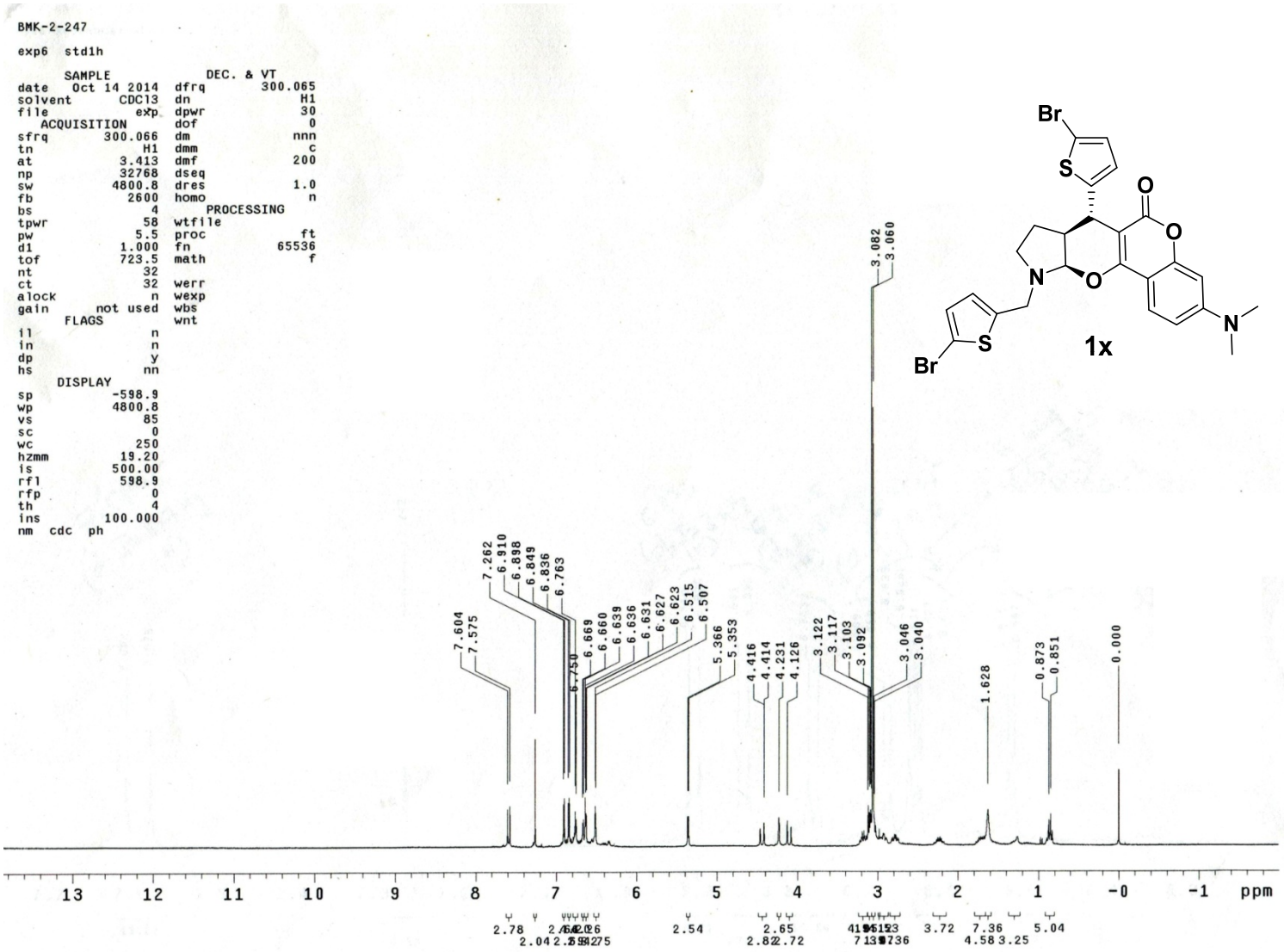

1w
BMK-2-243A
exp6 stdih
DEC. & VT
SAMPLE
date Oct 13 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -599.7
wp 4800.8
vs 229
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 599.7
rfp 0
th 6
ins 100.000
nm cdc ph
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
0.000
1.650
1.665
1.684
2.380
2.456
2.975
3.052
3.066
3.156
3.177
4.058
4.105
4.219
4.402
4.449
5.406
5.418
6.513
6.521
6.583
6.591
6.595
6.614
6.629
6.638
6.660
6.668
6.764
6.776
7.259
7.623
7.653
ppm

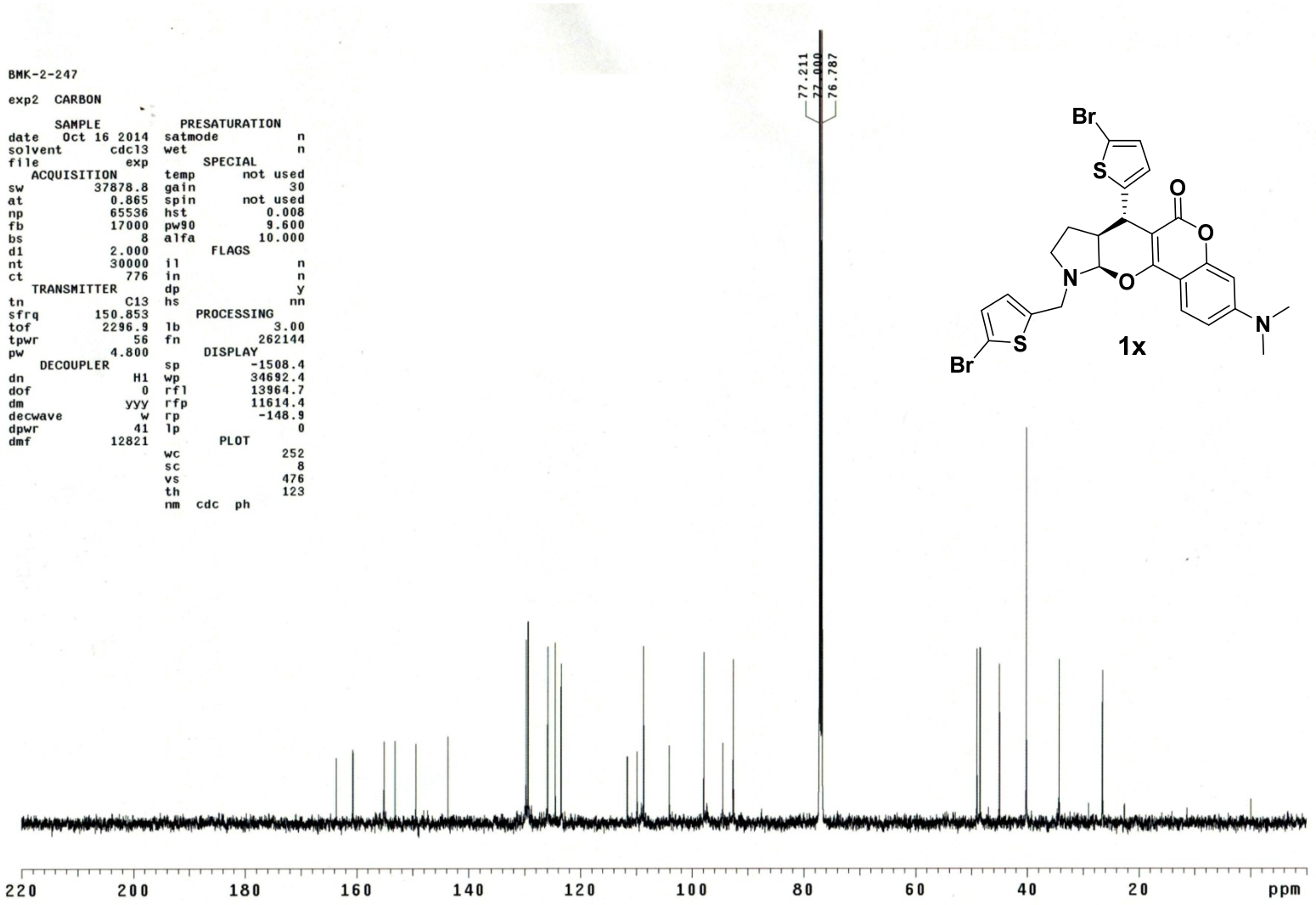

BMK-2-243A
exp10 CARBON
SAMPLE
date Oct 14 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 664
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.8
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13966.7
rfp 11614.4
rp -154.8
lp 0
PLOT
wc 252
sc 8
vs 200
th 90
nm cdc ph
77.213
77.000
76.789
1w
220
200
180
160
140
120
100
80
60
40
20
ppm

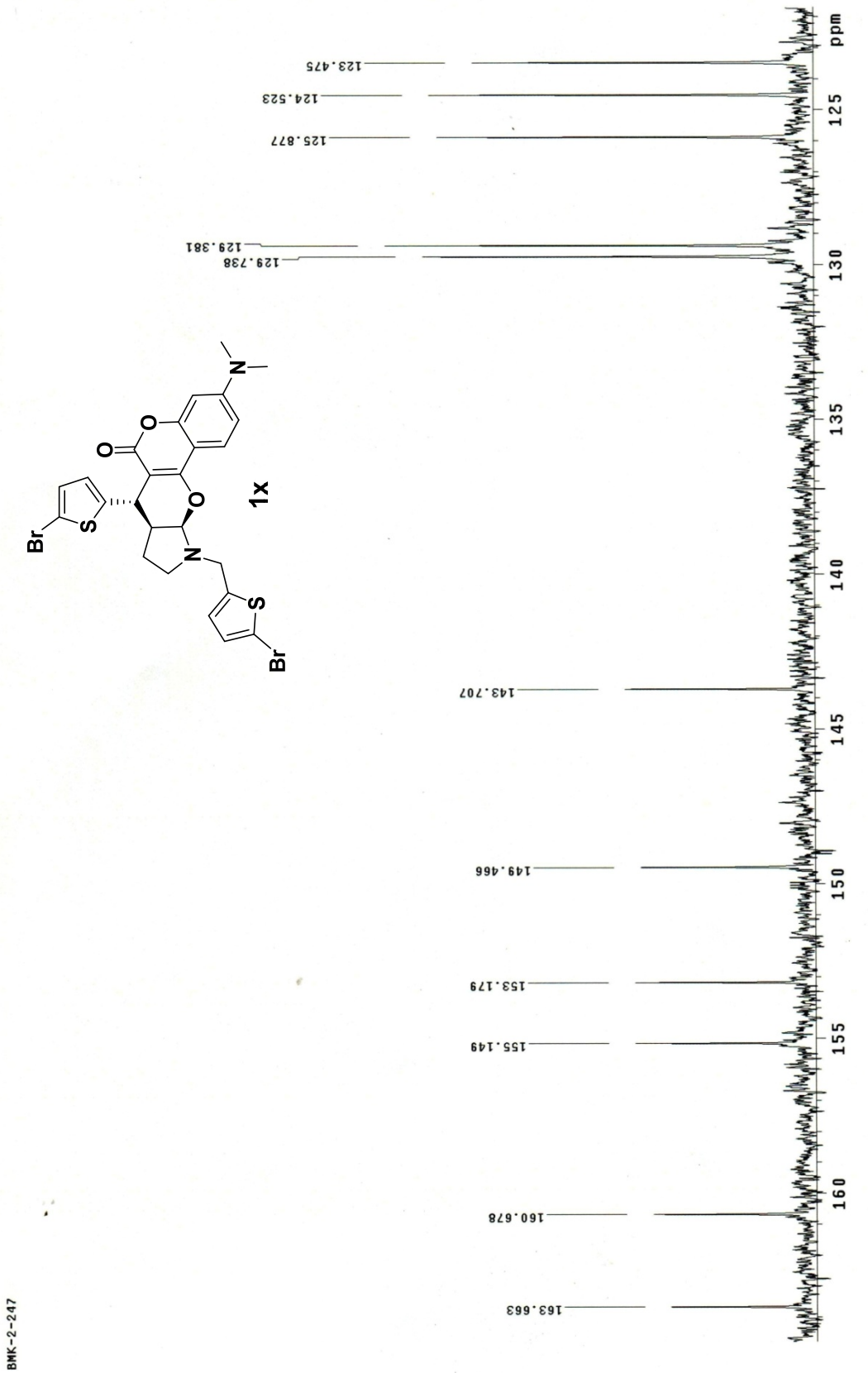

1w
137.836
139.327
139.612
145.406
152.995
155.068
160.496
163.853
ppm
138
140
142
144
146
148
150
152
154
156
158
160
162
164
166
BMK-2-243A

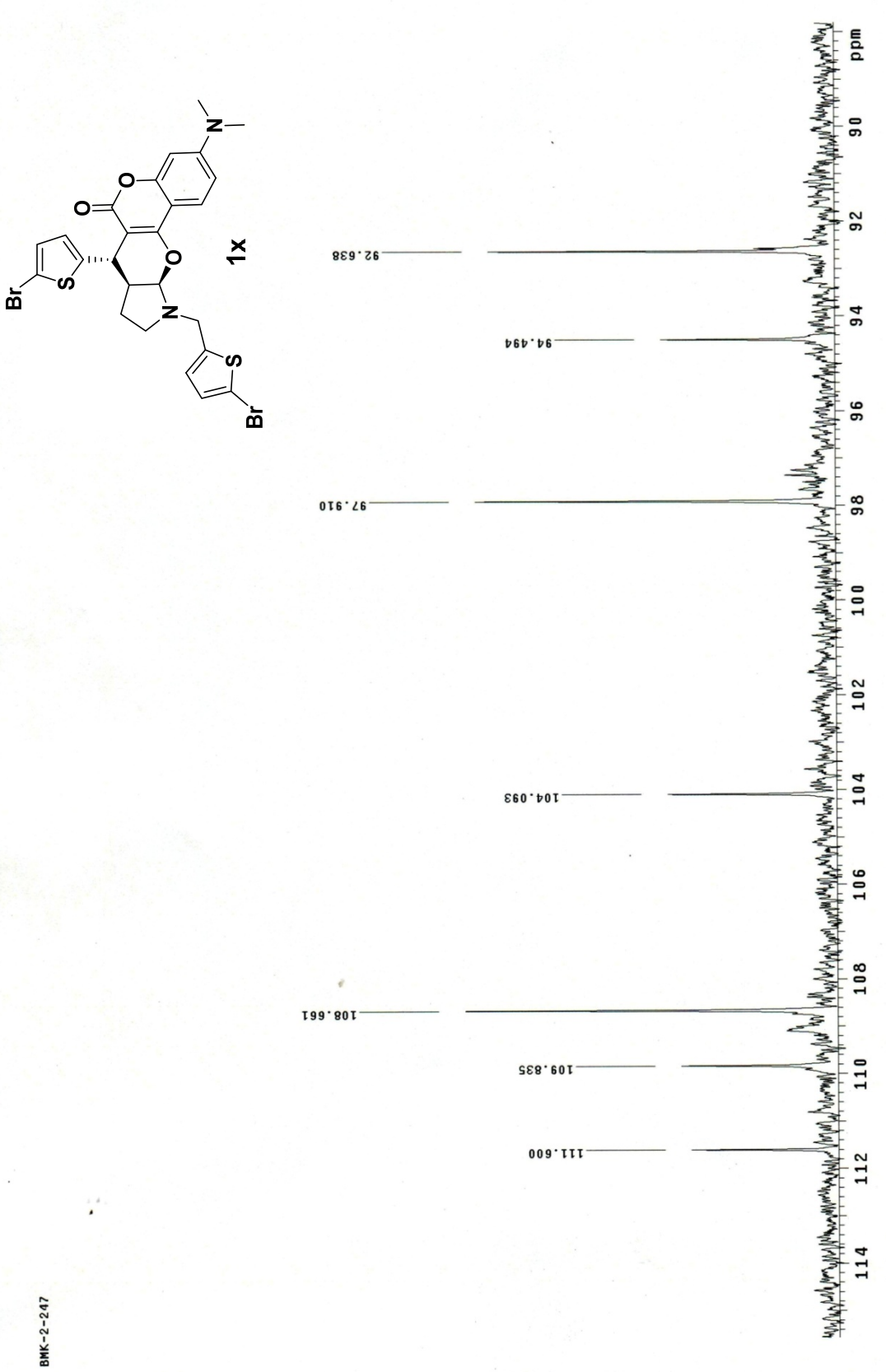

1w
92.927
95.456
97.947
104.495
108.559
123.496
123.829
124.519
124.829
125.661
ppm
95
100
105
110
115
120
125
130
BMK-2-243A

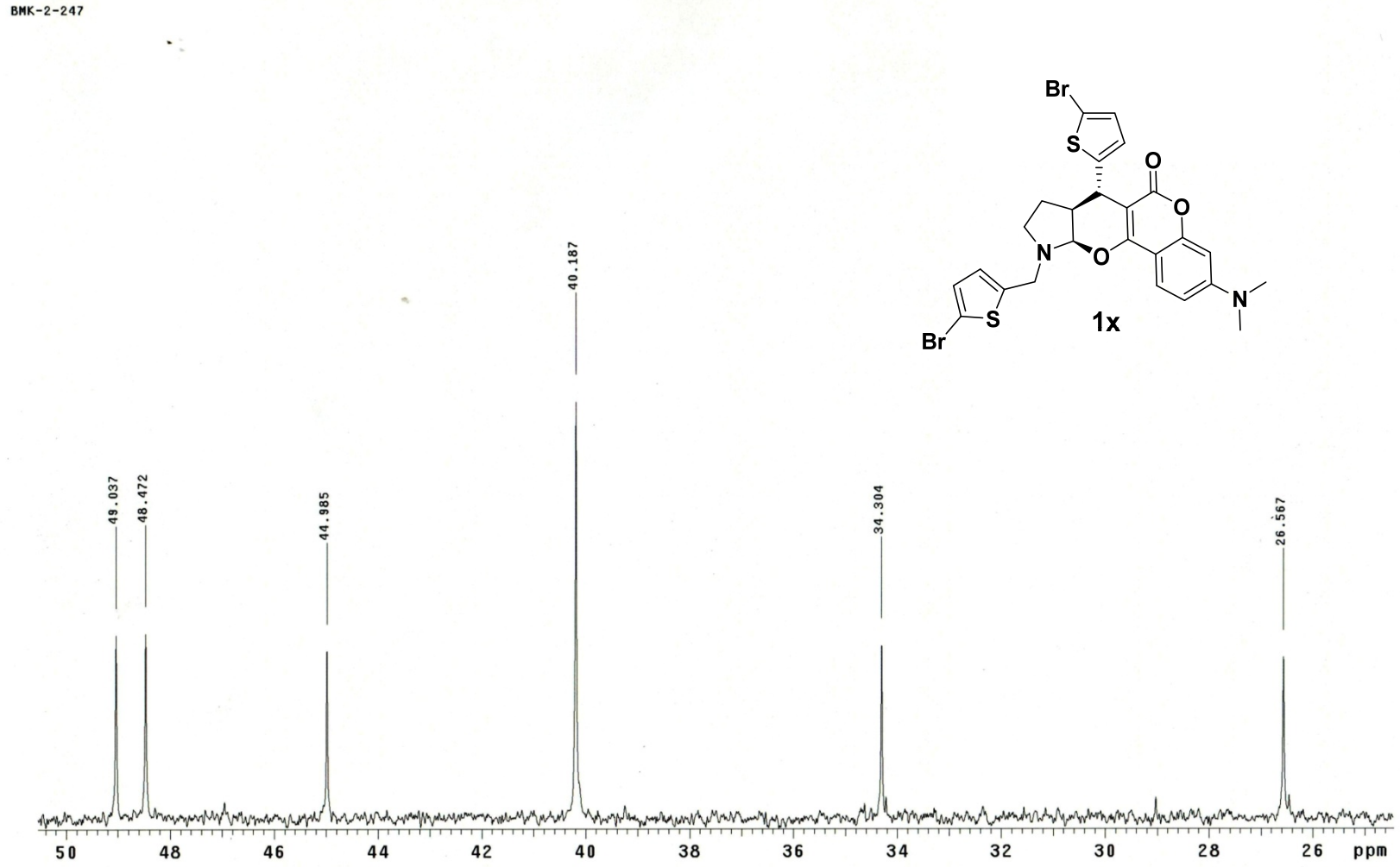

1w
BMK-2-243A
15.282
15.424
26.602
34.079
40.187
45.134
48.250
49.070
ppm
50
45
40
35
30
25
20

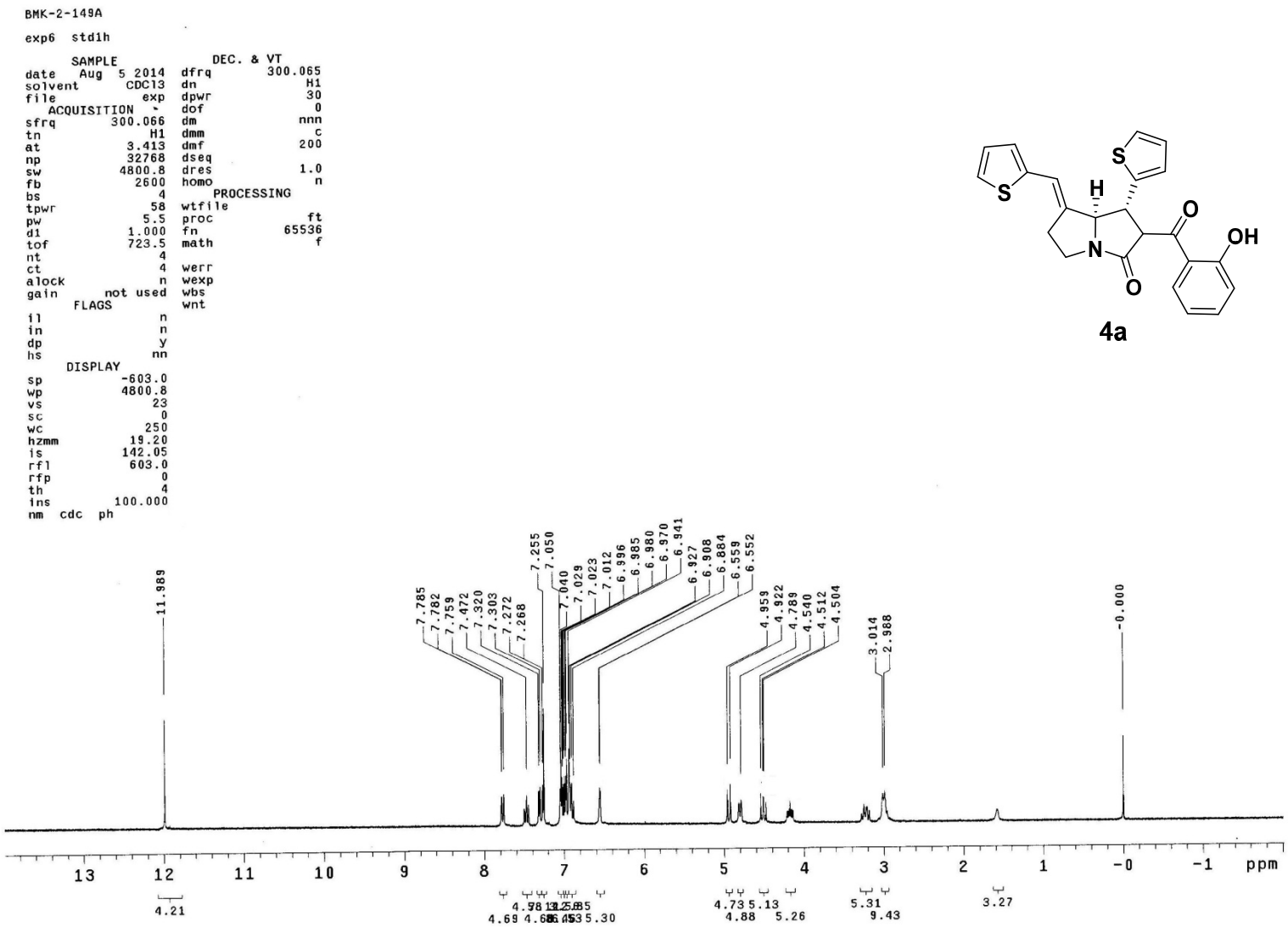

BMK-2-247
exp6 std1h
SAMPLE
date Oct 14 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -598.9
wp 4800.8
vs 85
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 598.9
rfp 0
th 4
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
Br
S
O
O
N
O
N
S
Br
1x
7.604
7.575
7.262
6.910
6.898
6.849
6.836
6.763
6.750
6.669
6.660
6.639
6.636
6.631
6.627
6.623
6.515
6.507
5.366
5.353
4.416
4.414
4.231
4.126
3.122
3.117
3.103
3.092
3.082
3.060
3.046
3.040
1.628
0.873
0.851
0.000
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.78
2.04
2.54
2.65
2.82
2.72
3.72
7.36
4.58
3.25
5.04


BMK-2-247
exp2 CARBON
SAMPLE
date Oct 16 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 776
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.9
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13964.7
rfp 11614.4
rp -148.9
lp 0
PLOT
wc 252
sc 8
vs 476
th 123
nm cdc ph
77.211
77.000
76.787
Br
S
O
O
N
O
N
S
Br
1x
220
200
180
160
140
120
100
80
60
40
20
ppm

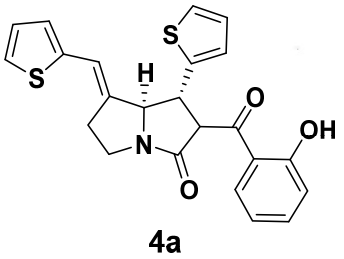

BMK-2-247
1x
Br
S
O
O
N
O
N
S
Br
163.663
160.678
155.149
153.179
149.466
143.707
129.738
129.381
125.877
124.523
123.475
ppm
160
155
150
145
140
135
130
125


1x
92.638
94.494
97.910
104.093
108.661
109.835
111.600
ppm
90
92
94
96
98
100
102
104
106
108
110
112
114
BMK-2-247

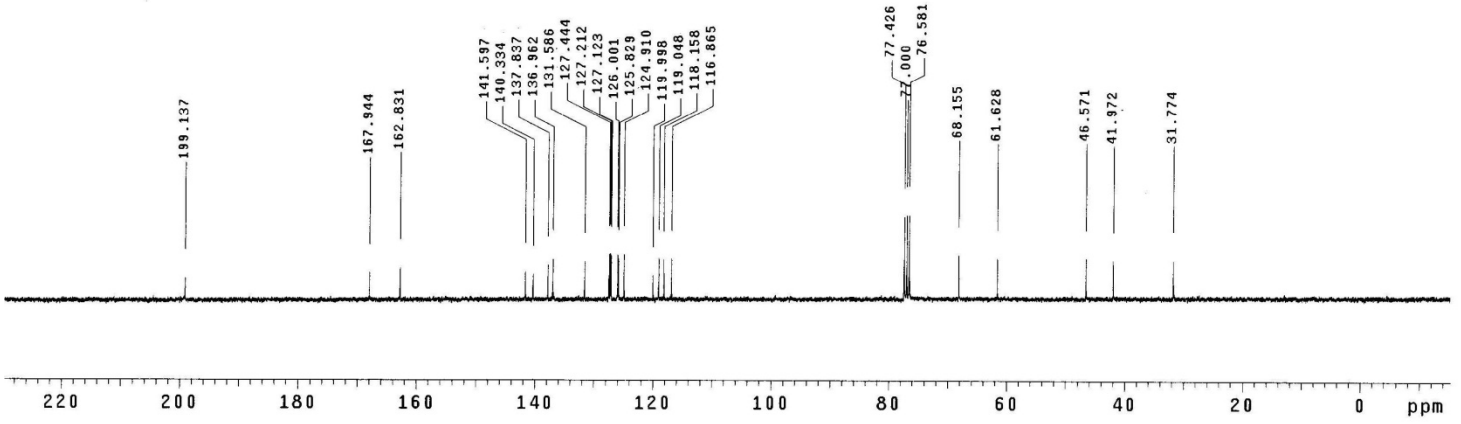

BMK-2-247
Br
S
O
O
N
O
N
S
Br
1x
49.037
48.472
44.985
40.187
34.304
26.567
50
48
46
44
42
40
38
36
34
32
30
28
26
ppm

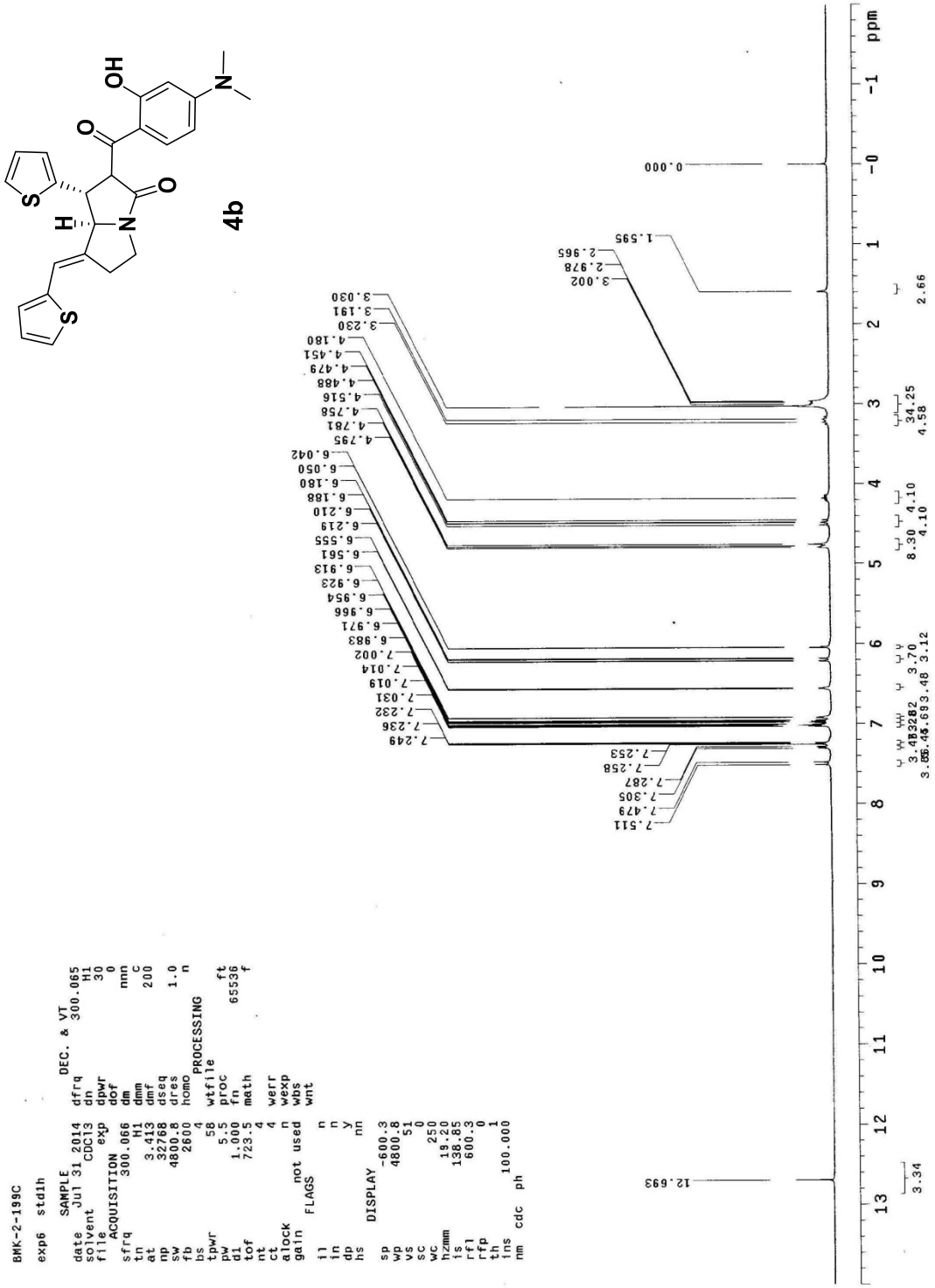

BMK-2-149A
exp6 std1h
SAMPLE
date Aug 5 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -603.0
wp 4800.8
vs 23
sc 0
wc 250
hzmm 19.20
is 142.05
rfl 603.0
rfp 0
th 4
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
11.989
7.785
7.782
7.759
7.472
7.320
7.303
7.272
7.268
7.255
7.050
7.040
7.029
7.023
7.012
6.996
6.985
6.980
6.970
6.941
6.927
6.908
6.884
6.559
6.552
4.959
4.922
4.789
4.540
4.512
4.504
3.014
2.988
-0.000
ppm
4.21
4.69
5.30
4.73
4.88
5.13
5.26
5.31
9.43
3.27
S
S
H
N
O
O
OH
4a

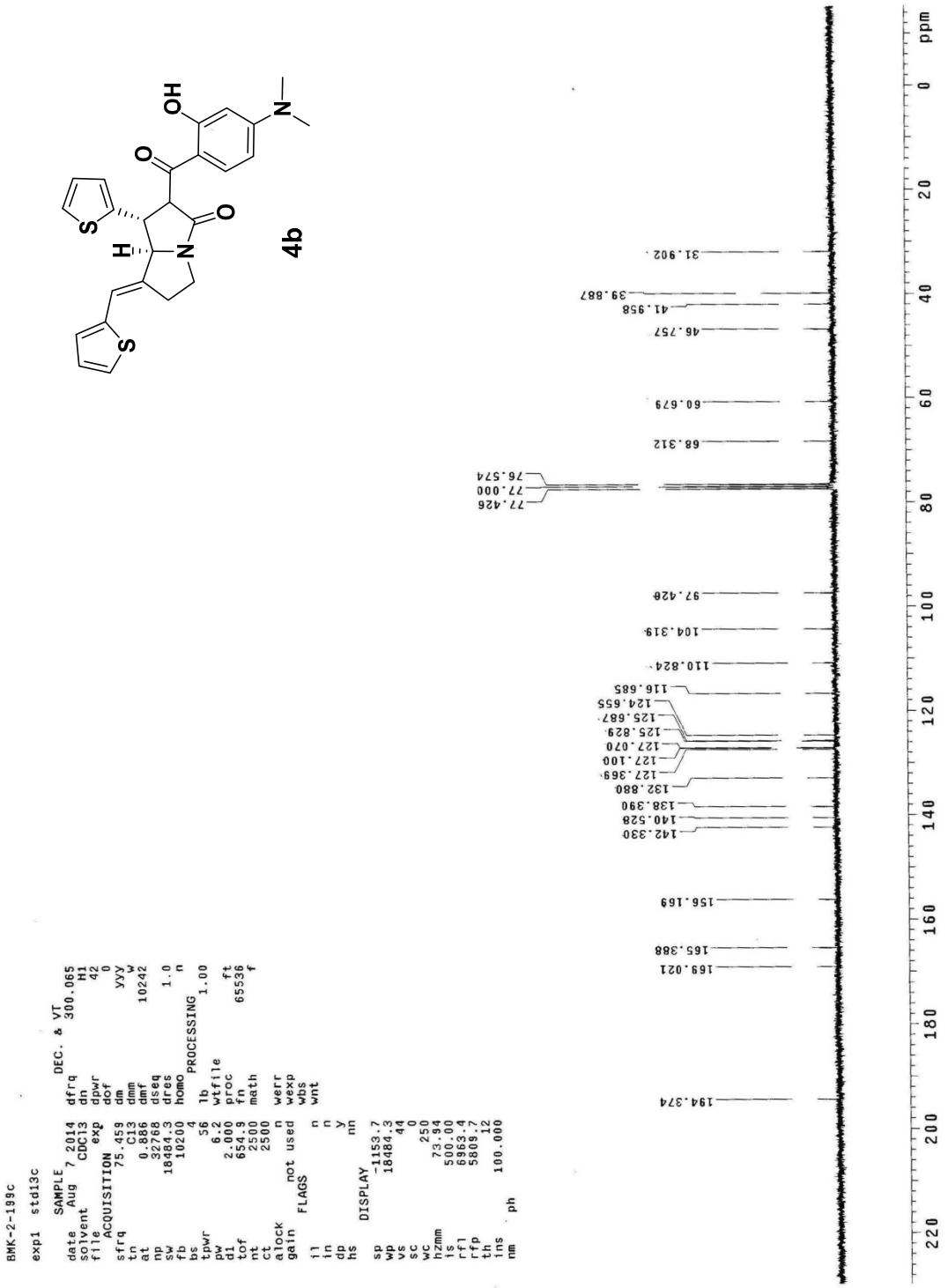
```
BMK-2-149AC

exp1  std13c

      SAMPLE
date    Aug  6 2014
solvent       CDCl3
file             exp
   ACQUISITION
sfrq          75.459
tn               C13
at             0.886
np             32768
sw          18484.3
fb             10200
bs                 4
tpwr              56
pw               6.2
d1             2.000
tof            654.9
nt              2500
ct              2500
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp          -1157.6
wp          18484.3
vs               24
sc                0
wc              250
hzmm          73.94
is           500.00
rfl         6967.4
rfp         5809.7
th                11
ins          100.000
nm    ph

     DEC. & VT
dfrq         300.065
dn                H1
dpwr              42
dof                0
dm               yyy
dmm                w
dmf            10242
dseq
dres             1.0
homo               n
     PROCESSING
lb               1.00
wtfile
proc              ft
fn             65536
math               f

werr
wexp
wbs
wnt
```

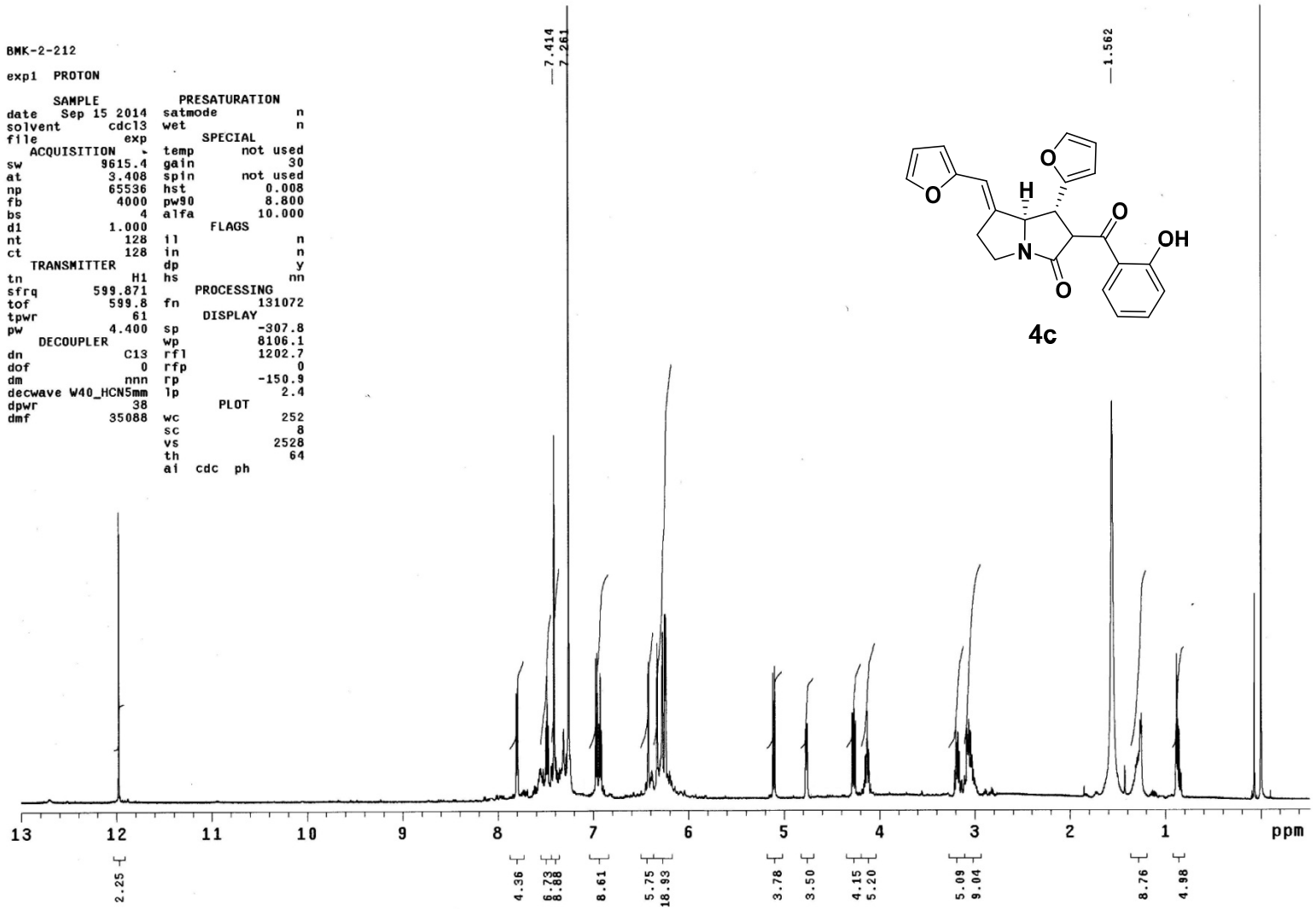


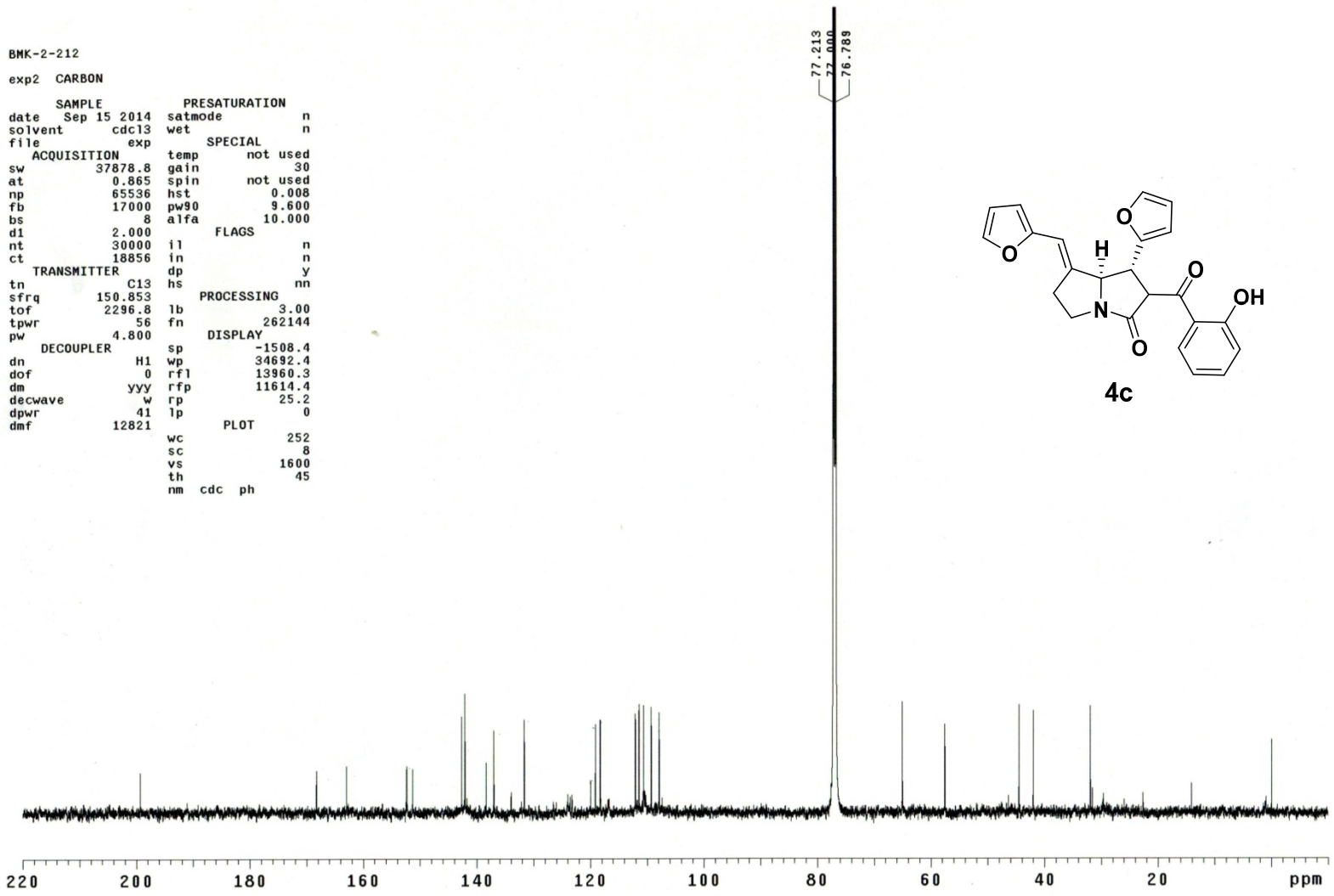
4a



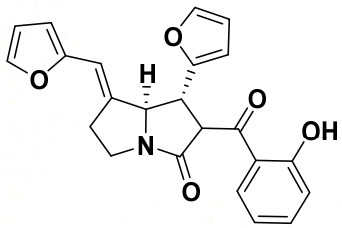

4b
BMK-2-199C
exp6 stdih
SAMPLE
date Jul 31 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -600.3
wp 4800.8
vs 51
sc 0
wc 250
hzmm 19.20
is 138.85
rfl 600.3
rfp 0
th 1
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
12.693
7.511
7.479
7.305
7.287
7.258
7.253
7.249
7.236
7.232
7.031
7.019
7.014
7.002
6.983
6.971
6.966
6.954
6.923
6.913
6.561
6.555
6.219
6.210
6.188
6.180
6.050
6.042
4.795
4.781
4.758
4.516
4.488
4.479
4.451
4.180
3.230
3.191
3.030
3.002
2.978
2.965
1.595
0.000
ppm


4b
BMK-2-199c
exp1 std13c
SAMPLE
date Aug 7 2014
solvent CDCl3
file ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
d1 2.000
tof 654.9
nt 2500
ct 2500
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1153.7
wp 18484.3
vs 44
sc 0
wc 250
hzmm 73.94
is 500.00
rfl 6963.4
rfp 5609.7
th 12
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
194.374
169.021
165.388
156.169
142.330
140.528
138.390
132.880
127.369
127.100
127.070
125.829
125.687
124.655
116.685
110.824
104.319
97.428
77.426
77.000
76.574
68.312
60.679
46.757
41.958
39.887
31.902
ppm

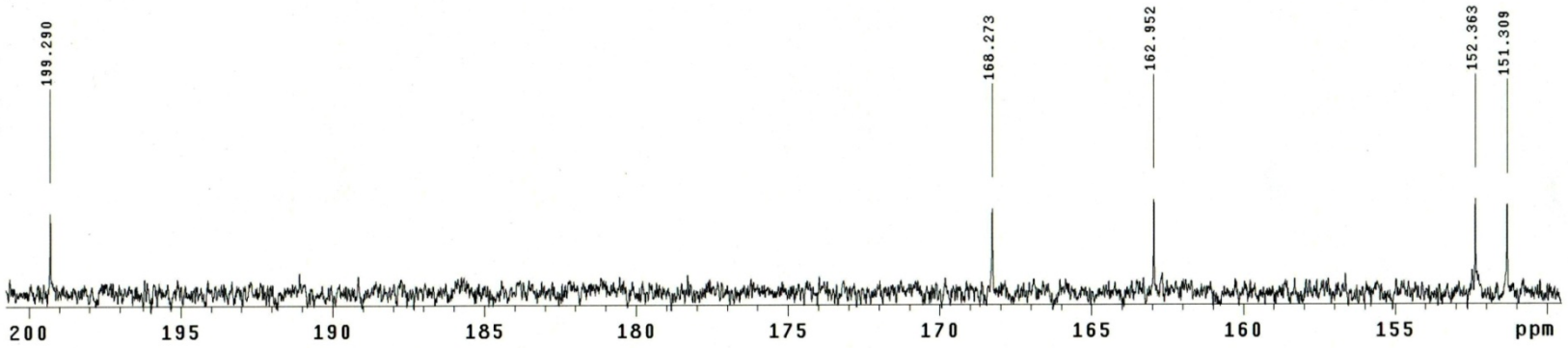

BMK-2-212
exp1 PROTON
SAMPLE
date Sep 15 2014
solvent cdcl3
file exp
ACQUISITION
sw 9615.4
at 3.408
np 65536
fb 4000
bs 4
d1 1.000
nt 128
ct 128
TRANSMITTER
tn H1
sfrq 599.871
tof 599.8
tpwr 61
pw 4.400
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_HCN5mm
dpwr 38
dmf 35088
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 8.800
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 131072
DISPLAY
sp -307.8
wp 8106.1
rfl 1202.7
rfp 0
rp -150.9
lp 2.4
PLOT
wc 252
sc 8
vs 2528
th 64
ai cdc ph
7.414
7.261
1.562
4c
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
2.25
4.36
6.73
8.88
8.61
5.75
18.93
3.78
3.50
4.15
5.20
5.09
9.04
8.76
4.98

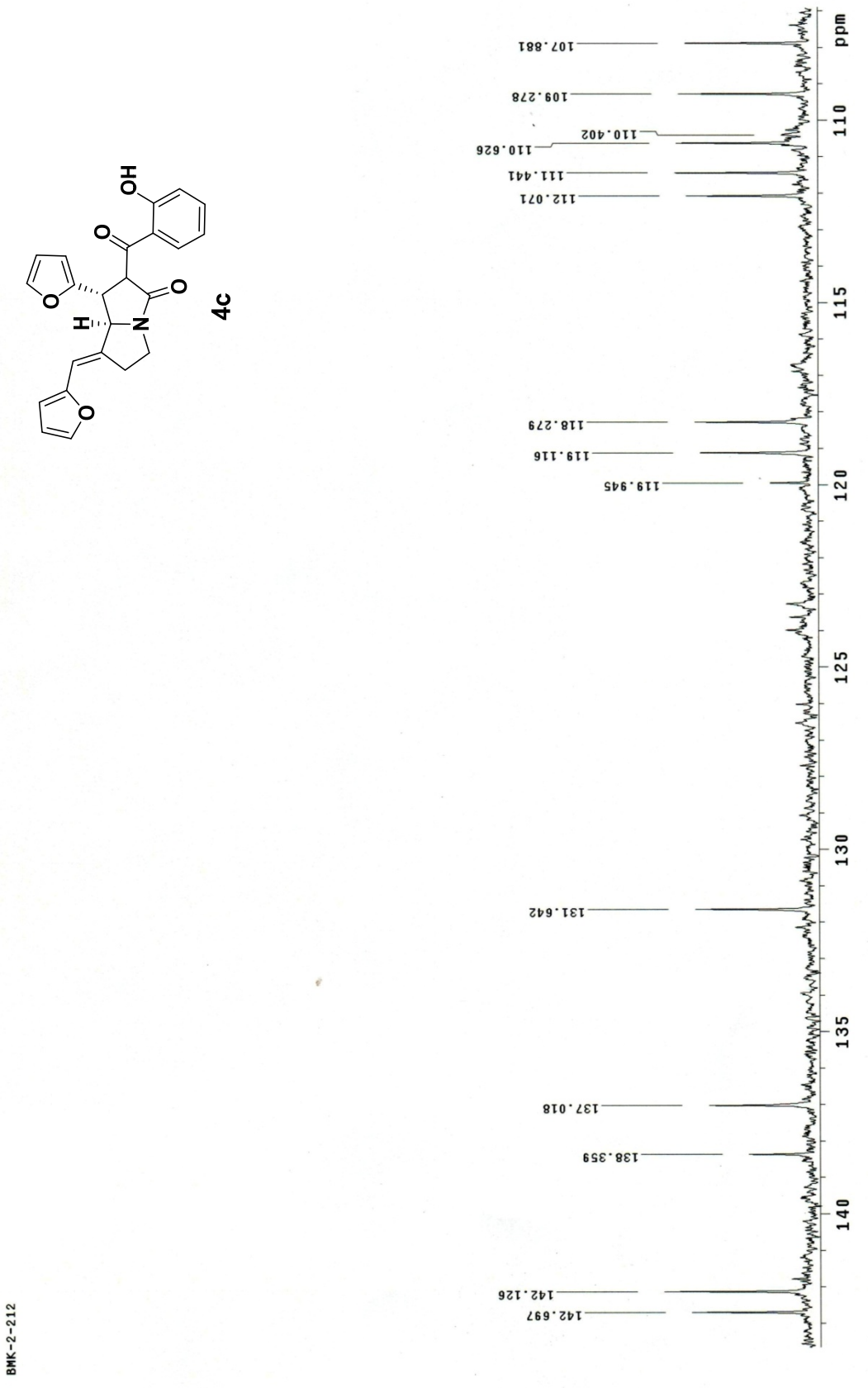

BMK-2-212
exp2 CARBON
SAMPLE
date Sep 15 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 18856
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.8
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13960.3
rfp 11614.4
rp 25.2
lp 0
PLOT
wc 252
sc 8
vs 1600
th 45
nm cdc ph
77.213
77.000
76.789
O
O
H
O
N
O
OH
4c
220
200
180
160
140
120
100
80
60
40
20
ppm

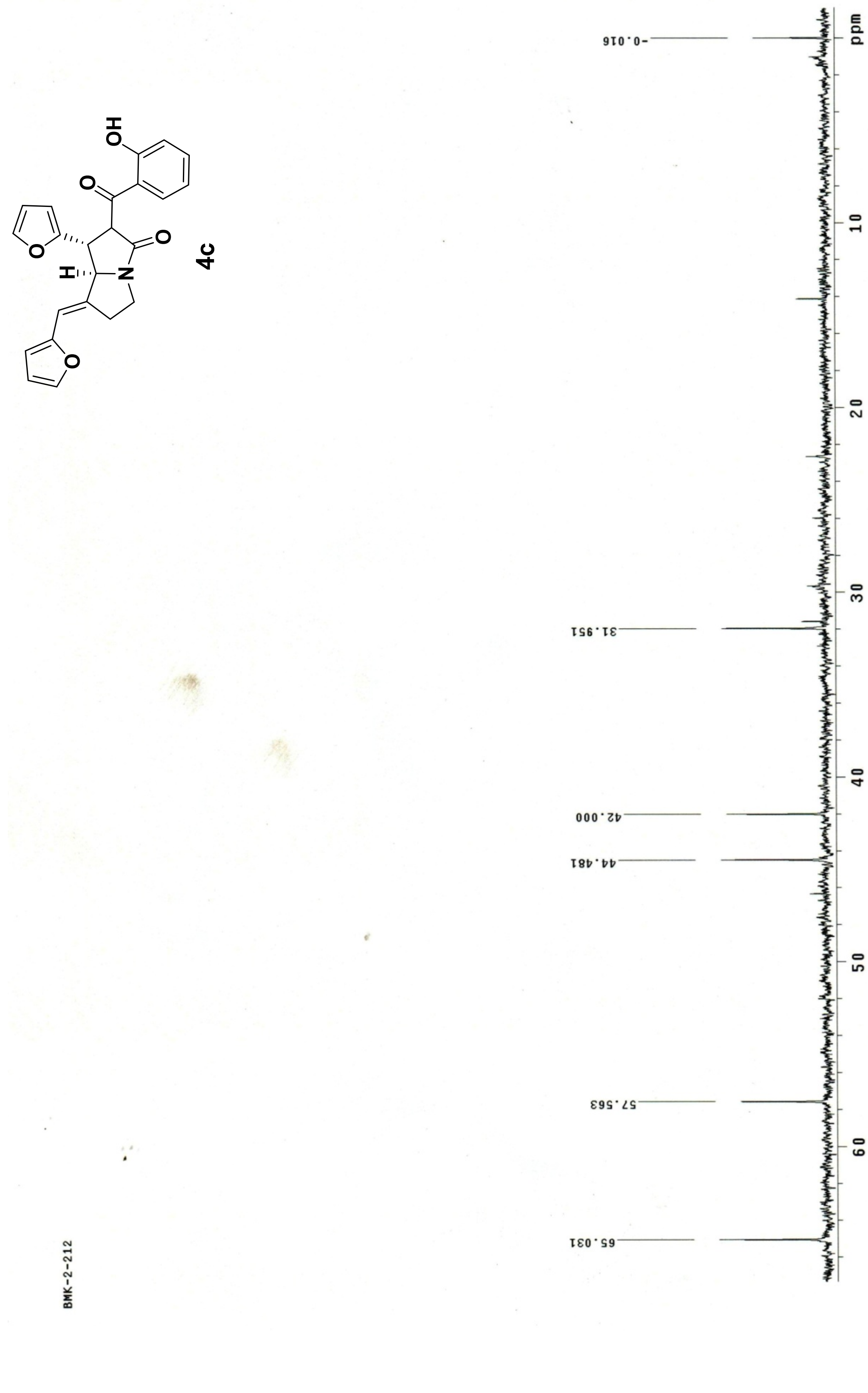
BMK-2-212


O
O
H
O
OH
N
O

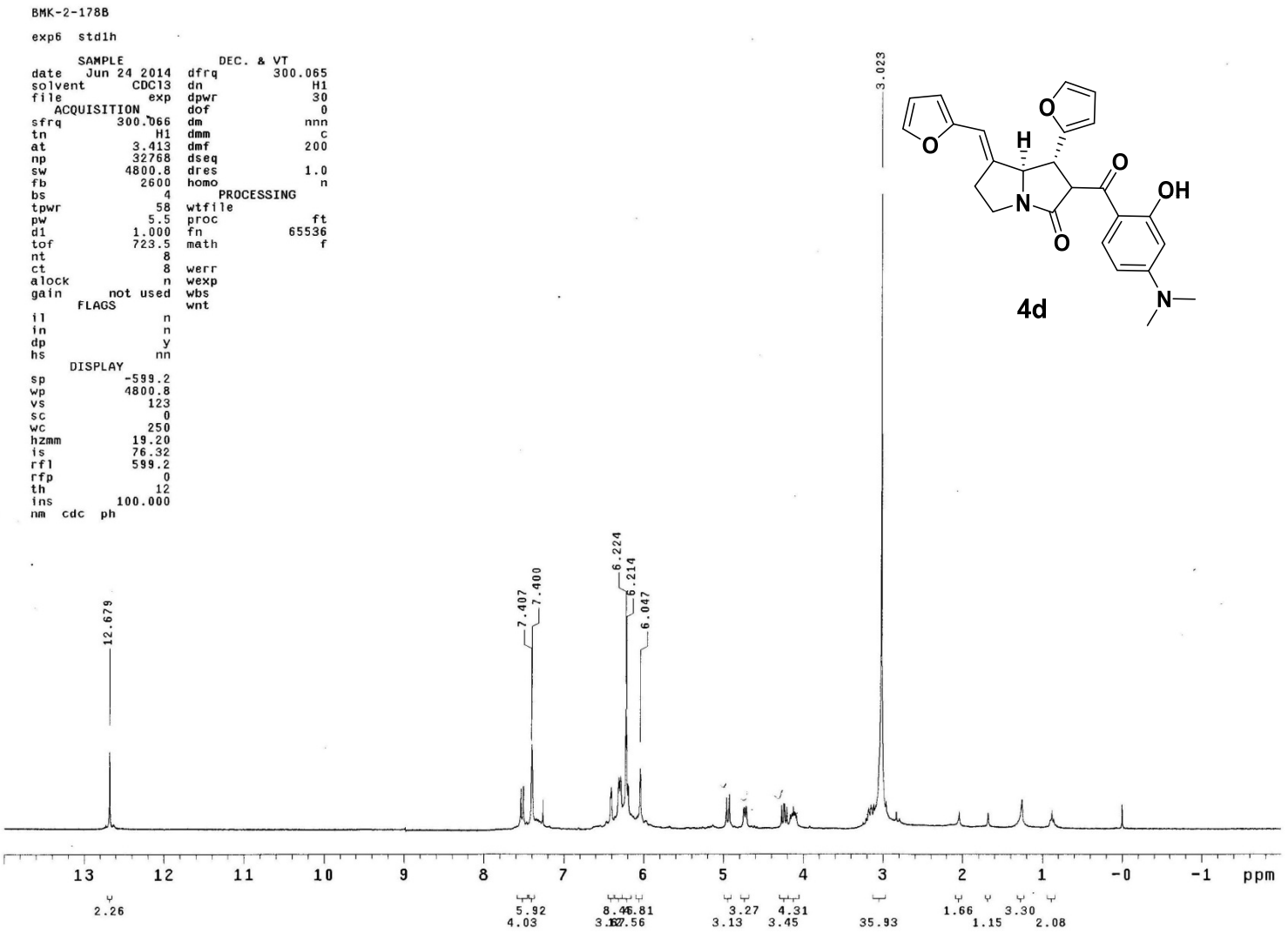
4c

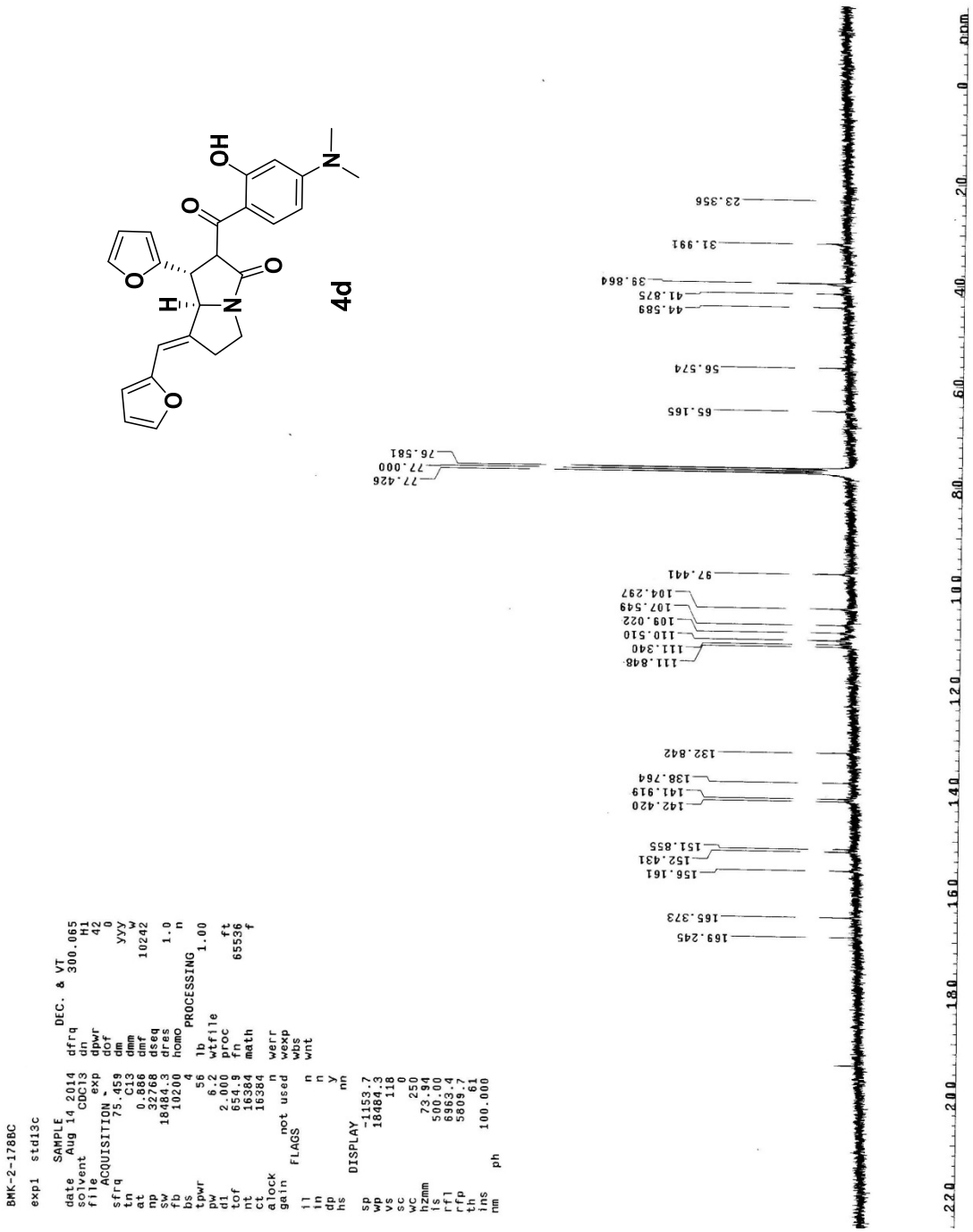

199.290
168.273
162.952
152.363
151.309
200
195
190
185
180
175
170
165
160
155
ppm

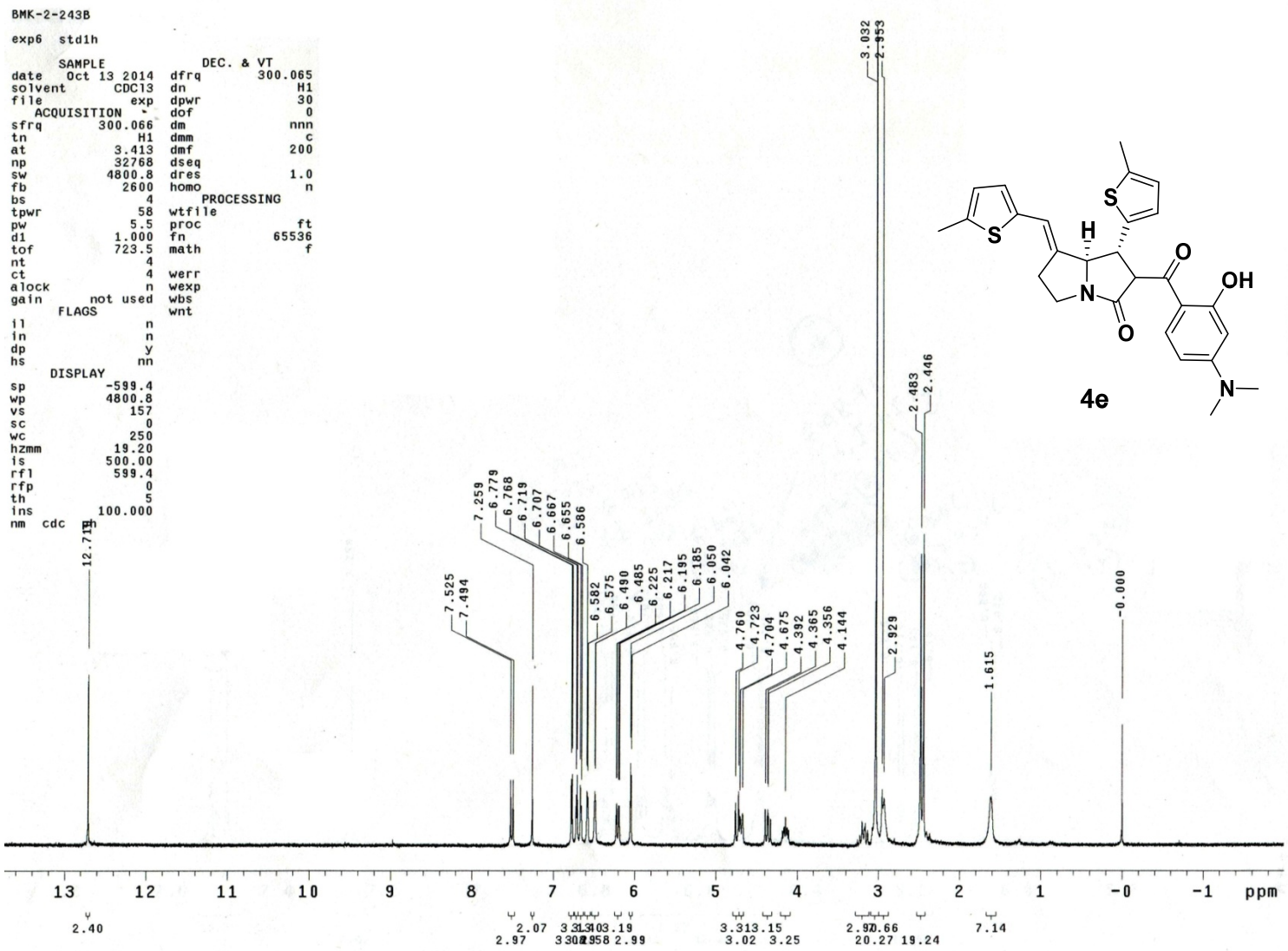

OH
O
O
O
H
N
O
4c
BMK-2-212
142.697
142.126
138.359
137.018
131.642
119.945
119.116
118.279
112.071
111.441
110.626
110.402
109.278
107.881
ppm
140
135
130
125
120
115
110

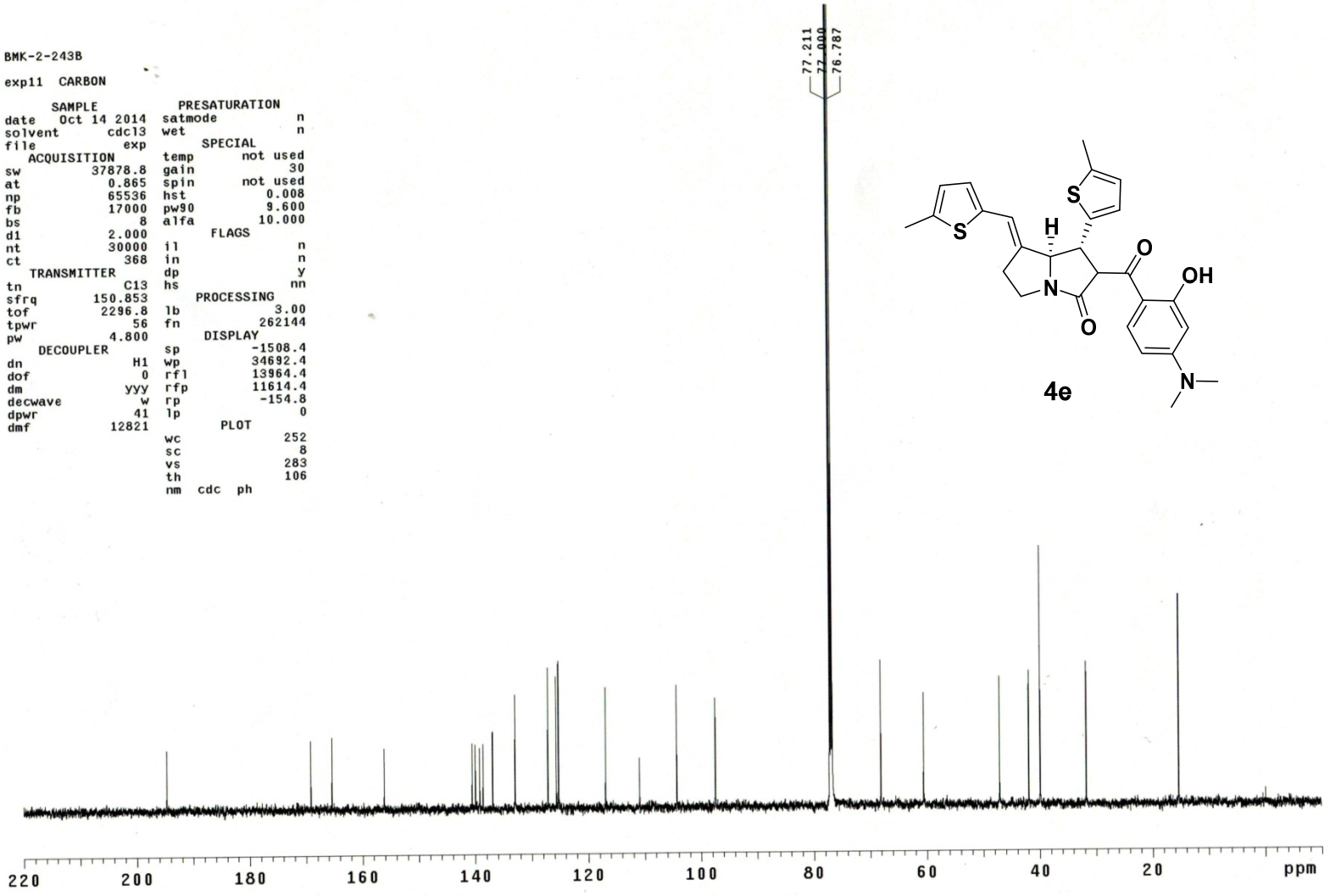


BMK-2-212
4c
OH
O
N
H
-0.016
31.951
42.000
44.481
57.563
65.031
ppm
0
10
20
30
40
50
60

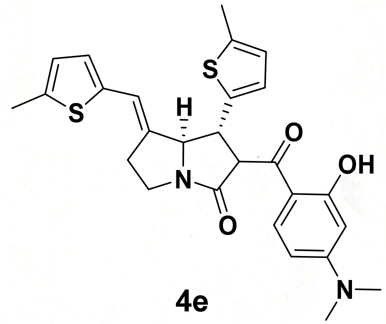

BMK-2-178B
exp6 stdlh
SAMPLE
date Jun 24 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -599.2
wp 4800.8
vs 123
sc 0
wc 250
hzmm 19.20
is 76.32
rfl 599.2
rfp 0
th 12
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
12.679
7.407
7.400
6.224
6.214
6.047
3.023
4d
13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
2.26
5.92
4.03
8.46
6.81
3.27
3.13
4.31
3.45
35.93
1.66
1.15
3.30
2.08


BMK-2-178BC
exp1 std13c
SAMPLE
date Aug 14 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 75.459
tn C13
at 0.886
np 32768
sw 18484.3
fb 10200
bs 4
tpwr 56
pw 6.2
dl 2.000
tof 654.3
nt 16384
ct 16384
alock n
gain n
not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1153.7
wp 18484.3
vs 118
sc 0
wc 250
hzmm 73.94
fs 500.00
rfl 6963.4
rfp 5603.7
th 61
ins 100.000
nm ph
DEC. & VT
dfrq 300.065
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 10242
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
4d
23.356
31.991
39.864
41.875
44.589
56.574
65.165
76.581
77.000
77.426
97.441
104.297
107.549
109.022
110.510
111.340
111.848
132.842
138.764
141.919
142.420
151.855
152.431
156.161
165.373
169.245

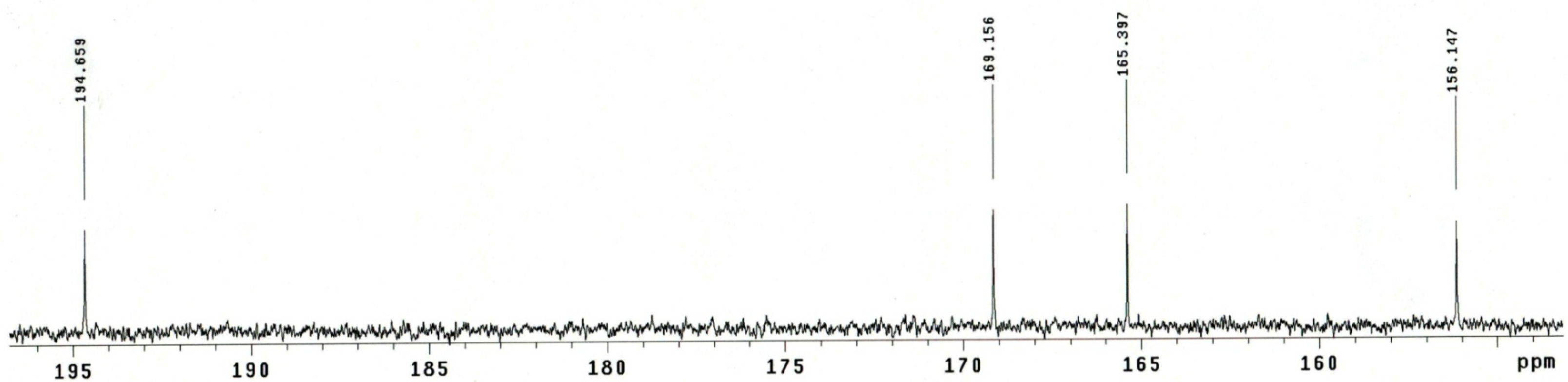

BMK-2-243B
exp6 std1h
SAMPLE
date Oct 13 2014
solvent CDCl3
file exp
ACQUISITION
sfrq 300.066
tn H1
at 3.413
np 32768
sw 4800.8
fb 2600
bs 4
tpwr 58
pw 5.5
d1 1.000
tof 723.5
nt 4
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -599.4
wp 4800.8
vs 157
sc 0
wc 250
hzmm 19.20
is 500.00
rfl 599.4
rfp 0
th 5
ins 100.000
nm cdc h
DEC. & VT
dfrq 300.065
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
12.719
7.525
7.494
7.259
6.779
6.768
6.719
6.707
6.667
6.655
6.586
6.582
6.575
6.490
6.485
6.225
6.217
6.195
6.185
6.050
6.042
4.760
4.723
4.704
4.675
4.392
4.365
4.356
4.144
3.032
2.929
2.483
2.446
1.615
-0.000
13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
2.40
2.07
2.97
3.19
2.99
3.31
3.02
3.15
3.25
2.97
20.27
19.24
7.14
S
S
H
N
O
O
OH
N
4e

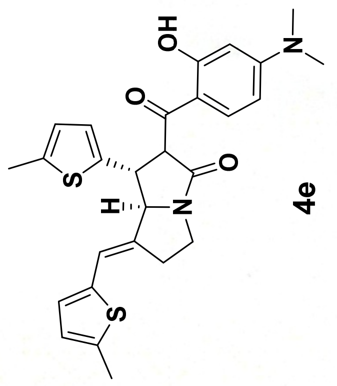

BMK-2-243B
exp11 CARBON
SAMPLE
date Oct 14 2014
solvent cdcl3
file exp
ACQUISITION
sw 37878.8
at 0.865
np 65536
fb 17000
bs 8
d1 2.000
nt 30000
ct 368
TRANSMITTER
tn C13
sfrq 150.853
tof 2296.8
tpwr 56
pw 4.800
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 41
dmf 12821
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 9.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn 262144
DISPLAY
sp -1508.4
wp 34692.4
rfl 13964.4
rfp 11614.4
rp -154.8
lp 0
PLOT
wc 252
sc 8
vs 283
th 106
nm cdc ph
77.211
77.000
76.787
S
S
H
N
O
O
OH
N
4e
220
200
180
160
140
120
100
80
60
40
20
ppm

BMK-2-243B

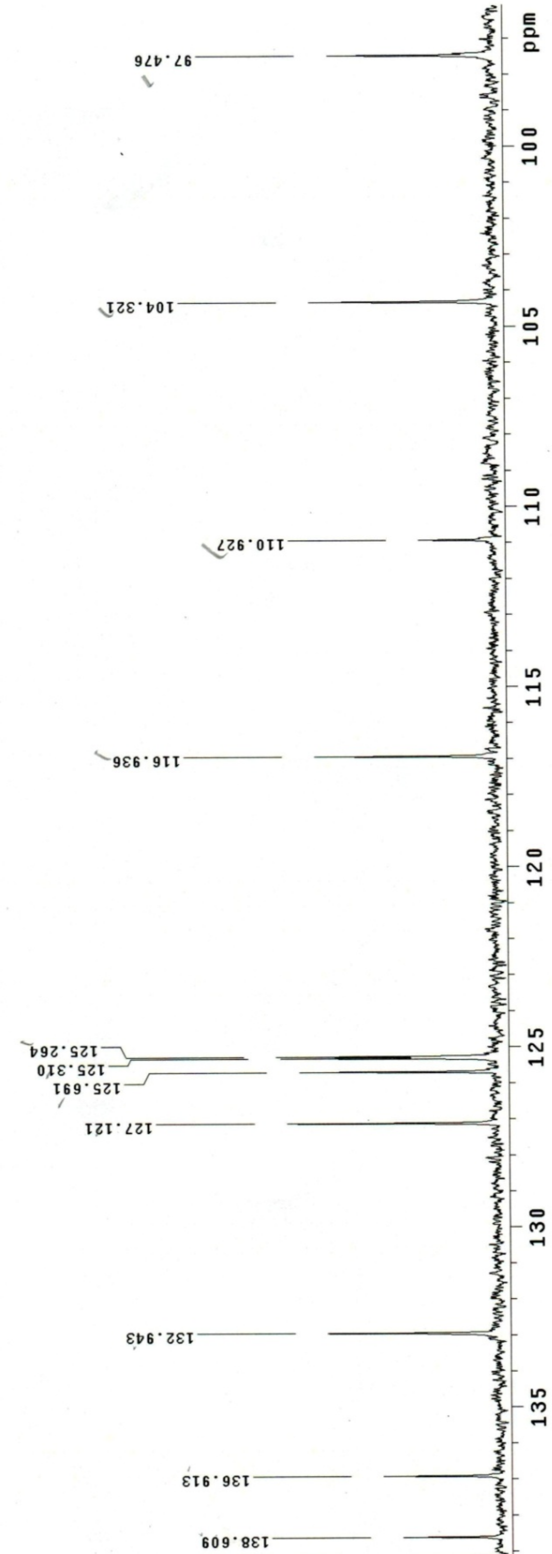


4e

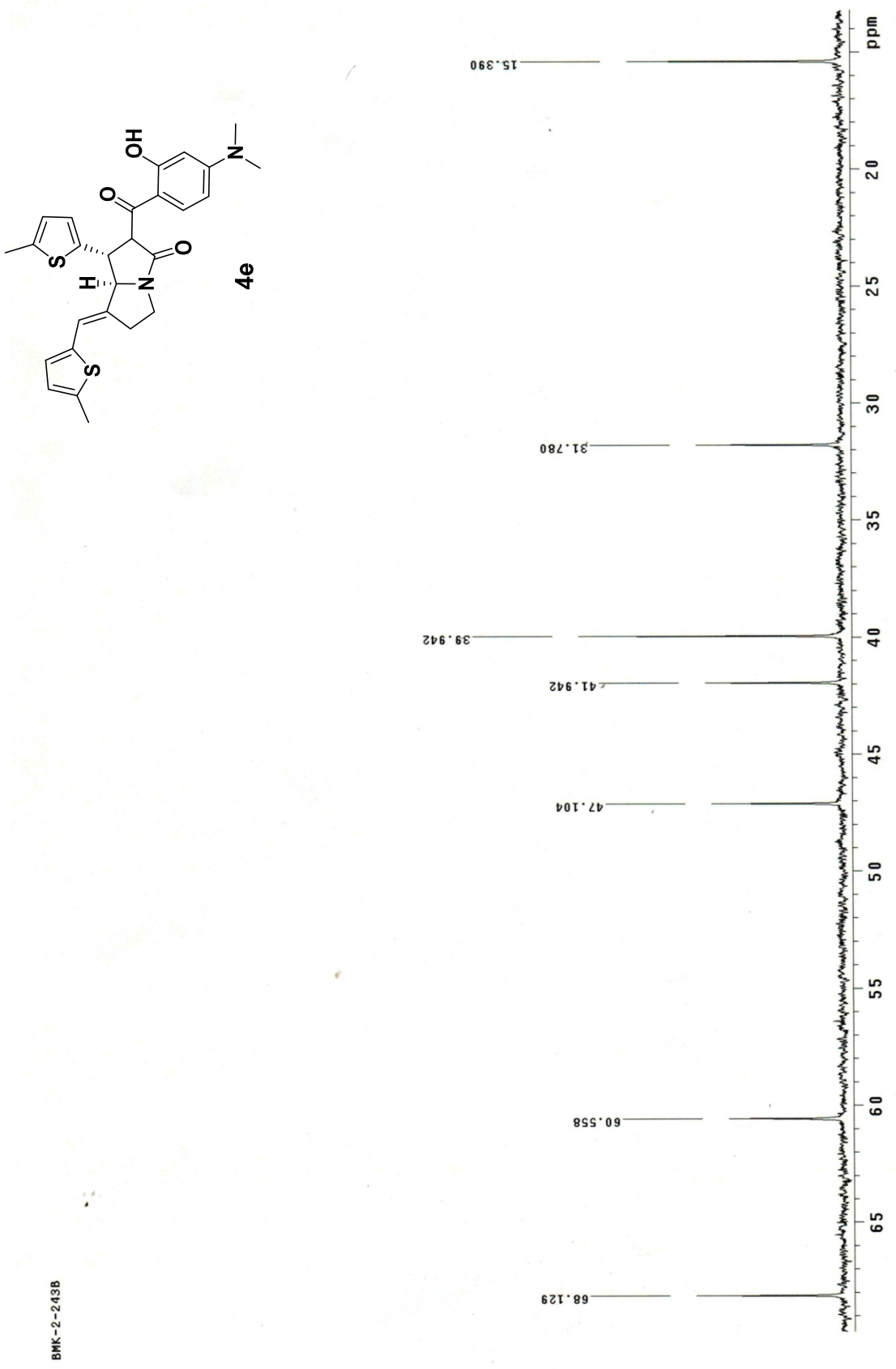

194.659
169.156
165.397
156.147
195
190
185
180
175
170
165
160
ppm



4e


97.476
104.321
110.927
116.936
125.264
125.310
125.691
127.121
132.943
136.913
138.609
139.185
139.952
140.549
ppm
100
105
110
115
120
125
130
135
140

BMK-2-243B


OH
O
N
O
S
H
N
S
4e
15.390
31.780
39.942
41.942
47.104
60.558
68.129
ppm
20
25
30
35
40
45
50
55
60
65
BMK-2-243B