Electronic Supplementary Material (ESI) for RSC Advances

Supporting Information

Role of temperature in [3+2]-cycloaddition of isoselenocyanates with oxiranes using $BF_3 \cdot Et_2O$

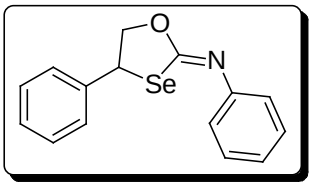
Mani Sengoden and Tharmalingam Punniyamurthy*

Department of Chemistry, Indian Institute of Technology Guwahati, Guwahati-781039, India.
E-mail: tpunni@iitg.ernet.in

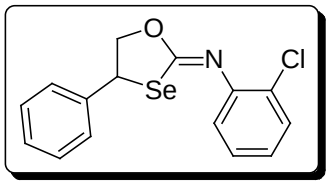
General Information. Anilines, oxiranes, alkenes, *m*-CPBA (77%), $BF_3 \cdot Et_2O$, $SnCl_4$ (1.0 M solution in DCM), $Cu(OTf)_2$ (98%) and $Sc(OTf)_3$ (99.99%) were purchased from Aldrich. $InCl_3$ (98%) was purchased from Lancaster. $FeCl_3$ (96%) was purchased from Rankem. Selenium (99.9%) was purchased from SRL. The column chromatography was performed with Rankem silica gel (60-120 mesh). NMR (^{1}H and ^{13}C) spectra were recorded with a Varian 400 spectrometer. Melting points were determined with a Büchi B-545 apparatus and are uncorrected. FT-IR spectra were recorded using Perkin Elmer IR spectrometer. Elemental analyses were recorded using Perkin Elmer CHNS analyzer. X-Ray data were collected on a Bruker SMART APEX equipped with a CCD area detector using Mo Kα radiation. The structures were solved by direct method using *SHELXL-97* (Göttingen, Germany).

General Procedure for Preparation of Isoselenocyanates 1a-l.[1] To a stirred solution of the isocyanide (2 mmol) in $CHCl_3$ (1 mL) were added selenium powder (3 mmol) and NEt_3 (2 mmol) at room temperature (26 °C) under nitrogen balloon and the mixture was stirred for 7 h. Progress of the reaction was monitored by TLC using hexane as eluent. The suspension was then passed through a celite bed and the solvent was removed by evaporation on a rotary evaporator. The residue was purified on a silica gel column chromatography using hexane as eluent.

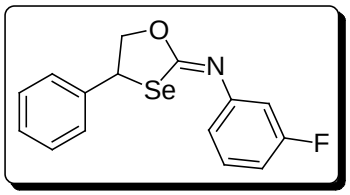
General Procedure for Synthesis of Substituted 2-Imino-1,3-oxaselenolanes 3a-p. A mixture of isoselenocyanate **1a-l** (0.5 mmol), oxirane **2a-f** (0.75 mmol) and $BF_3{\cdot}Et_2O$ (0.05 mmol) were stirred at -5 °C in CH_2Cl_2 under nitrogen balloon. The progress of reaction was monitored by TLC using ethyl acetate and hexane. The reaction mixture was diluted with CH_2Cl_2 (15 mL) and the solution was washed with 5% $NaHCO_3$ solution (5 mL), brine (1 x 3 mL) and water (3 x 5 mL) and. Drying (Na_2SO_4) and evaporation of the solvent gave a residue that was purified on silica gel column chromatography using hexane and ethyl acetate (8:2) as eluent.



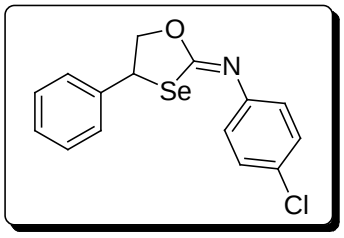
(*Z*)-*N*-(4-Phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3a: Yellow liquid; yield 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.6 Hz, 2H), 7.34-7.26 (m, 5H), 7.11 (t, *J* = 6.8 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 2H), 5.10 (t, *J* = 7.2 Hz, 1H), 4.67 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.51 (dd, *J* = 10.0, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 150.3, 137.8, 129.4, 129.2, 128.5, 127.8, 124.8, 121.0, 76.8, 48.8; FT-IR (neat) 3059, 3030, 2936, 2887, 1732, 1651, 1592, 1488, 1454, 1244, 1116, 1091, 1044, 1017 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{13}NOSe$: C 59.61, H 4.34, N 4.63, found: C 59.70, H 4.33, N 4.59.



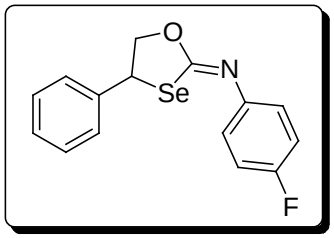
(*Z*)-2-Chloro-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3b: Yellow liquid; yield 42%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.37 (m, 2H), 7.36-7.26 (m, 3H), 7.19 (td, *J* = 7.6, 1.6 Hz, 2H), 7.04 (td, *J* = 8.0, 1.6 Hz, 1H), 6.96 (dd, *J* = 7.6, 1.2 Hz, 1H), 5.16 (t, *J* = 6.8 Hz, 1H), 4.73 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.58 (dd, *J* = 10.0, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 147.3, 137.7, 130.2, 129.5, 129.2, 128.6, 127.8, 126.7, 125.7, 121.9, 49.3. FT-IR (neat) 3029, 2924, 1651, 1585, 1494, 1471, 1451, 1280, 1245, 1101, 1059, 1042, 1017 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}ClNOSe$: C 53.51, H 3.59, N 4.16, found: C 53.59, H 3.57, N 4.12.



(*Z*)-3-Fluoro-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3c: Yellow liquid; yield 76%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.21 (m, 6H), 6.85-6.71 (m, 3H), 5.14 (t, *J* = 6.8 Hz, 1H), 4.70 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.54 (dd, *J* = 10.0, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 163.5, 162.0, 151.9, 151.8, 137.6, 130.6, 130.5, 129.2, 128.6, 127.7, 116.7, 111.7, 111.5, 108.6, 108.4, 77.0, 49.0; FT-IR (neat) 3063, 3030, 2928, 1652, 1604, 1582, 1480, 1454, 1262, 1092, 1044, 1022 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}FNOSe$: C 56.26, H 3.78, N 4.37, found: C 56.36, H 3.77, N 4.34.

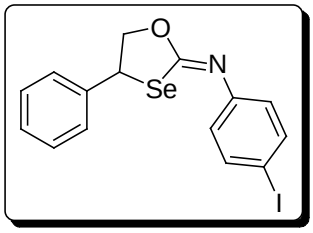


(*Z*)-4-Chloro-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3d: Yellow liquid; yield 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.28 (m, 5H), 7.26 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.12 (t, *J* = 7.2 Hz, 1H), 4.67 (dd, *J* = 9.6, 6.0 Hz, 1H), 4.51 (dd, *J* = 9.6, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 148.7, 137.6, 130.6, 130.0, 129.4, 129.2, 128.6, 127.7, 122.3, 76.9, 49.0; FT-IR (neat) 3061, 3030, 2928, 1651, 1590, 1485, 1455, 1245, 1091, 1044, 1020, 1010, 945 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}ClNOSe$: C 53.51, H 3.59, N 4.16, found: C 53.59, H 3.57, N 4.14.

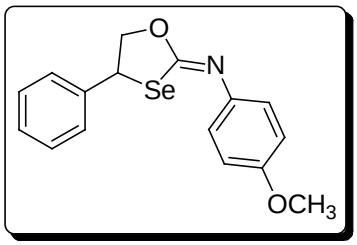


(*Z*)-4-Fluoro-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3e: Yellow liquid; yield 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.34-7.27 (m, 3H), 7.00-6.91 (m, 4H), 5.9 (t, *J* = 7.2 Hz, 1H), 4.66 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.19 (dd, *J* = 10.0, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 161.2, 158.8, 146.3, 137.7, 129.1, 128.5, 127.7, 122.3, 122.2, 116.1, 115.8, 76.8, 48.8; FT-IR (neat)

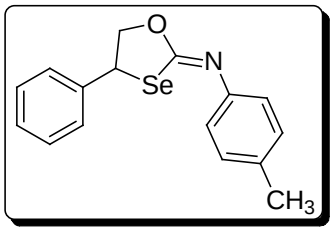
3062, 3031, 2928, 1658, 1601, 1501, 1454, 1214, 1091, 1044, 1020 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}FNOSe$: C 56.26, H 3.78, N 4.37, found: C 56.34, H 3.76, N 4.33.



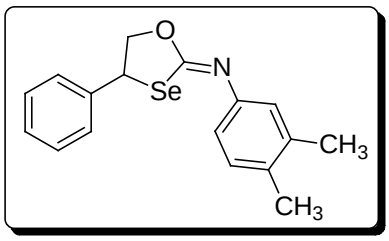
(*Z*)-4-Iodo-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3f: Colourless solid; yield 85%; mp 161-162 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.38-7.28 (m, 5H), 6.73 (d, *J* = 8.4 Hz, 2H), 5.11 (t, *J* = 6.8 Hz, 1H), 4.67 (dd, *J* = 10.0, 5.6 Hz, 1H), 4.50 (dd, *J* = 9.6, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1, 149.8, 138.4, 137.6, 129.3, 128.6, 127.8, 123.2, 88.7, 76.9, 49.1; FT-IR (KBr) 2943, 1652, 1576, 1473, 1462, 1244, 1202, 1115, 1093, 1079, 1041, 1003, 948 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}INOSe$: C 42.08, H 2.83, N 3.27, found: C, 42.15; H, 2.81; N, 3.24.



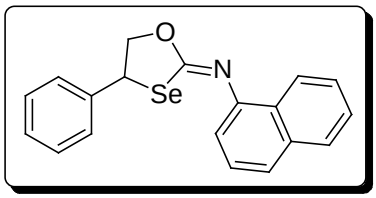
(*Z*)-4-Methoxy-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3g: Yellow liquid; yield 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 6.8 Hz, 2H), 7.34-7.25 (m, 3H), 6.91 (d, *J* = 6.8 Hz, 2H), 6.84 (t, *J* = 6.8 Hz, 2H), 5.90 (dd, *J* = 7.2, 6.0 Hz, 1H), 4.66 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.49 (dd, *J* = 10.0, 7.6 Hz, 1H), 3.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 156.8, 143.6, 137.9, 129.1, 128.4, 127.7, 121.9, 114.5, 76.6, 55.4, 48.6; FT-IR (neat) 3060, 3031, 2933, 2835, 1651, 1505, 1463, 1455, 1290, 1243, 1091, 1033, 945 cm^{-1}. Elemental analysis calcd (%) for $C_{16}H_{15}NO_2Se$: C 57.84, H 4.55, N 4.22, found: C 57.96, H 4.53, N 4.18.



(*Z*)-4-Methyl-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3h: Yellow liquid; yield 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.35 (m, 2H), 7.32-7.25 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 6.89 (d, *J* = 8.0 Hz, 2H), 5.06 (t, *J* = 6.4 Hz, 1H), 4.61 (dd, *J* = 10.0, 5.6 Hz, 1H), 4.46 (dd, *J* = 10.0, 7.2 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1, 147.7, 137.9, 134.1, 129.8, 129.0, 128.3, 127.6, 120.6, 76.5, 48.5, 20.9; FT-IR (neat) 3060, 3028, 2942, 2887, 1651, 1505, 1454, 1370, 1282, 1244, 1173, 1089, 1043, 1014, 945 cm^{-1}. Elemental analysis calcd (%) for $C_{16}H_{15}NOSe$: C 60.76, H 4.78, N 5.06, found: C 60.82, H 4.77, N 5.02.

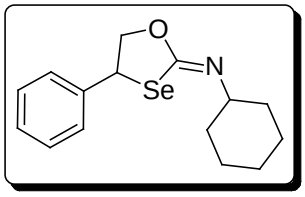


(*Z*)-3,4-Dimethyl-*N*-(4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3j: Yellow liquid; yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.36 (m, 2H), 7.35-7.25 (m, 4H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.80 (s, 1H), 6.75 (dd, *J* = 8.0, 2.4 Hz, 1H), 5.06 (dd, *J* = 7.2, 6.4 Hz, 1H), 4.63 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.48 (dd, *J* = 10.0, 7.2 Hz, 1H), 2.23 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.9, 148.0, 137.9, 137.5, 132.8, 130.3, 129.0, 128.3, 127.7, 122.1, 117.8, 76.5, 48.4, 19.8, 19.2; FT-IR (neat) 3060, 3027, 2938, 2885, 1654, 1603, 1496, 1453, 1371, 1281, 1244, 1120, 1089, 1044, 1025, 1000 cm^{-1}. Elemental analysis calcd (%) for $C_{17}H_{17}NOSe$: C 61.82, H 5.19, N 4.24, found: C 61.91, H 5.16, N 4.21.

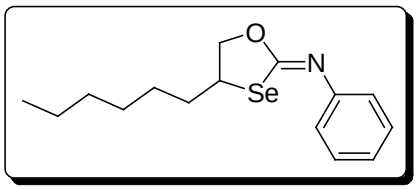


(*Z*)-*N*-(4-Phenyl-1,3-oxaselenolan-2-ylidene)naphthalen-1-amine 3k: Yellow liquid; yield 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (t, *J* = 4.8 Hz, 1H), 7.83 (t, *J* = 4.8 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 1H), 7.52-7.47 (m, 2H), 7.42-7.37 (m, 3H), 7.35-7.24 (m, 3H), 7.05 (d, *J* = 6.8 Hz, 1H), 5.07 (t, *J* = 6.8 Hz,

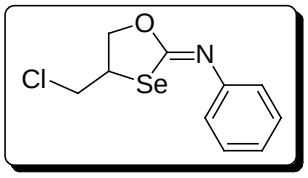
1H), 4.73 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.57 (dd, *J* = 10.0, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 147.2, 137.8, 134.3, 129.2, 128.5, 127.9, 127.8, 127.6, 126.5, 126.0, 125.8, 125.0, 123.7, 115.0, 77.1, 48.6; FT-IR (neat) 3059, 2927, 1651, 1574, 1494, 1454, 1393, 1270, 1245, 1113, 1073, 1045, 1013 cm^{-1}. Elemental analysis calcd (%) for $C_{19}H_{15}NOSe$: C 64.78, H 4.29, N 3.98, found: C 64.86, H 4.28, N 3.95.



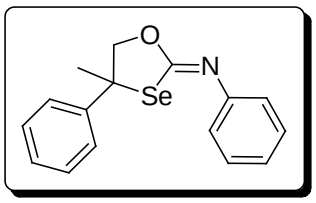
(*Z*)-*N*-(4-Phenyl-1,3-oxaselenolan-2-ylidene)cyclohexanamine 3l: Colourless liquid; yield 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.23 (m, 5H), 5.06 (dd, *J* = 6.8, 6.0 Hz, 1H), 4.45 (dd, *J* = 10.0, 6.0 Hz, 1H), 4.29 (dd, *J* = 10.0, 6.8 Hz, 1H), 2.53 (m, 1H), 1.78-1.75 (m, 3H), 1.61-1.58 (m, 1H), 1.48-1.40 (m, 2H), 1.35-1.19 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4, 138.2, 129.0, 128.4, 128.2, 127.7, 75.2, 48.5, 33.8, 33.7, 25.5, 24.6; FT-IR (neat) 3061, 3030, 2928, 2854, 1728, 1660, 1493, 1451, 1369, 1348, 1243, 1129, 1059, 1040, 1014 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{19}NOSe$: C 58.44, H 6.21, N 4.54, found: C 58.51, H 6.19, N 4.50.



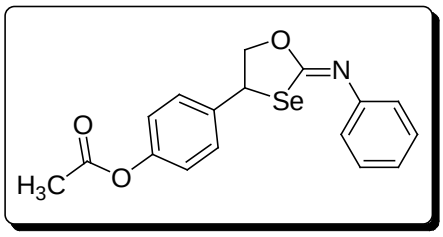
(*Z*)-*N*-(4-Hexyl-1,3-oxaselenolan-2-ylidene)benzenamine 3m: Yellow liquid; yield 79%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 7.6 Hz, 2H), 4.57-4.50 (m, 1H), 3.35 (dd, *J* = 10.0, 4.8 Hz, 1H), 3.13 (t, *J* = 10.0 Hz, 1H), 1.97-1.88 (m, 1H), 1.75-1.67 (m, 1H), 1.58-1.52 (m, 1H), 1.51-1.40 (m, 1H), 1.39-1.29 (m, 6H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 150.3, 129.3, 124.5, 121.0, 84.4, 34.1, 31.7, 31.2, 29.1, 26.0, 22.6, 14.1; FT-IR (neat) 3060, 3027, 2927, 2856, 1658, 1593, 1488, 1458, 1221, 1146, 1090, 1020 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{21}NOSe$: C 58.06, H 6.82, N 4.51, found: C 58.15, H 6.81, N 4.47.



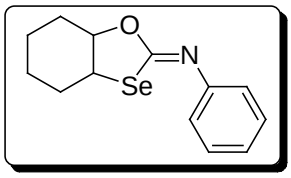
(*Z*)-*N*-(4-(Chloromethyl)-1,3-oxaselenolan-2-ylidene)benzenamine 3n: Colourless solid; yield 86%; mp 53-54 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 8.0 Hz, 2H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 7.2 Hz, 2H), 4.80-4.74 (m, 1H), 3.79 (qd, *J* = 11.2, 4.8 Hz, 2H), 3.47 (dd, *J* = 10.4, 5.6 Hz, 1H), 3.43 (dd, *J* = 10.4, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 149.9, 129.4, 124.9, 120.8, 81.5, 43.2, 28.5; FT-IR (KBr) 3062, 3031, 2928, 1637, 1551, 14841, 1454, 1214, 1095, 1024, 1020 cm^{-1}. Elemental analysis calcd (%) for $C_{10}H_{10}ClNOSe$: C 43.74, H 3.67, N 5.10, found: C 43.81, H 3.66, N 5.07.



(*Z*)-*N*-(4-Methyl-4-phenyl-1,3-oxaselenolan-2-ylidene)benzenamine 3o: Colourless liquid; yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.33 (t, *J* = 6.8 Hz, 3H), 7.29 (t, *J* = 7.6 Hz, 3H), 7.10 (t, *J* = 7.6 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 4.66 (d, *J* = 10.0 Hz, 1H), 4.41 (d, *J* = 9.6 Hz, 1H), 2.07 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.7, 150.1, 141.7, 129.4, 129.0, 127.9, 126.6, 124.8, 121.1, 81.5, 59.2, 29.4; FT-IR (neat) 2926, 1644, 1489, 1446, 1384, 1260, 1087, 1019, 912 cm^{-1}. Elemental analysis calcd (%) for $C_{16}H_{15}NOSe$: C 60.76, H 4.78, N 4.43, found: C 60.83, H 4.76, N 4.40.

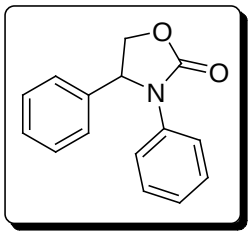


4-((*Z*)-2-(Phenylimino)-1,3-oxaselenolan-4-yl)phenyl acetate 3p: Yellow liquid; yield 65%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.25 (m, 4H), 7.11-6.92 (m, 4H), 5.05 (t, *J* = 7.2 Hz, 1H), 4.61 (dd, *J* = 10.4, 6.4 Hz, 1H), 4.45 (dd, *J* = 10.0, 7.2 Hz, 1H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 162.4, 150.6, 150.1, 135.5, 129.4, 128.9, 124.8, 122.4, 120.9, 76.7, 48.1, 21.1; FT-IR (neat) 3061, 3032, 3001, 2930, 2840, 1755, 1643, 1506, 1462, 1369, 1201, 1167, 1090, 1046, 1018 cm^{-1}. Elemental analysis calcd (%) for $C_{17}H_{15}NO_3Se$: C 56.68, H 4.20, N 3.89, found: C, 56.77, H 4.18, N 3.91.

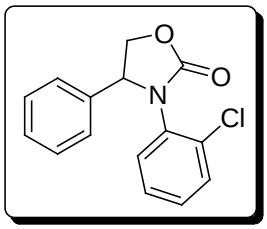


(*Z*)-*N*-(Hexahydrobenzo[d][1,3]oxaselenol-2-ylidene)benzenamine 3q: Colourless solid; yield 62%; mp 102-103 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 8.4 Hz, 2H), 7.09 (t, *J* = 8.8 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 2H), 4.00 (td, *J* = 11.2, 4.0 Hz, 1H), 3.56 (td, *J* = 10.8, 3.6 Hz, 1H), 2.38-2.34 (m, 1H), 2.17-2.13 (m, 1H), 1.95-1.82 (m, 1H), 1.80-1.78 (m, 1H), 1.68-1.58 (m, 2H), 1.48-1.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 150.1, 129.3, 124.6, 121.1, 88.5, 49.6, 30.4, 29.8, 26.1, 24.1; FT-IR (KBr) 3069, 2937, 2861, 1645, 1588, 1484, 1445, 1361, 1257, 1194, 1145, 1116, 1086, 1039 cm^{-1}. Elemental analysis calcd (%) for $C_{13}H_{15}NOSe$: C 55.72, H 5.40, N 5.00, found: C 55.78, H 5.39, N 4.98.

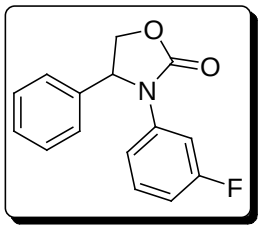
General Procedure for Synthesis of 1,3-Oxazolidinones 4a-p. A mixture of isoselenocyanate **1a-l** (0.5 mmol), oxirane **2a-f** (1 mmol) and $BF_3{\cdot}Et_2O$ (0.05 mmol) were stirred at 40 °C in CH_2Cl_2 under nitrogen balloon. The progress of reaction was monitored by TLC using ethyl acetate and hexane. The reaction mixture was filtered. The solution was diluted with CH_2Cl_2 (15 mL) and washed with 5% $NaHCO_3$ solution (5 mL), brine (1 x 3 mL) and water (3 x 5 mL). Drying (Na_2SO_4) and evaporation of the solvent gave a residue that was purified on silica gel column chromatography using hexane and ethyl acetate (8:2) as eluent.



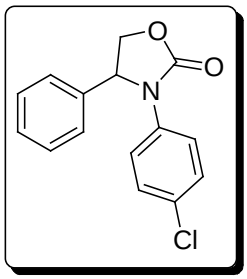
3,4-Diphenyloxazolidin-2-one 4a:[2a] Colourless solid; yield 73%; mp 119-120 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.0 Hz, 2H), 7.35-7.29 (m, 5H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.05 (t, *J* =7.2 Hz, 1H), 5.39 (dd, *J* = 8.4, 6.4 Hz, 1H), 4.77 (t, *J* = 8.0 Hz, 1H), 4.19 (dd, *J* = 8.0, 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 138.4, 137.2, 129.5, 129.1, 129.0, 126.4, 124.8, 121.0, 69.9, 60.8; FT-IR (KBr) 3062, 3001, 2971, 2911, 1754, 1597, 1499, 1401, 1367, 1222, 1202, 1127, 1042 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{13}NO_2$: C 75.30, H 5.48, N 5.85, found: C 75.39, H 5.45, N 5.81.



3-(2-Chlorophenyl)-4-phenyloxazolidin-2-one 4b: Yellow liquid; yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.32-7.25 (m, 5H), 7.15 (td, *J* = 7.2, 1.6 Hz, 1H), 7.10 (td, *J* = 7.2, 1.6 Hz, 1H), 7.03 (dd, *J* = 7.6, 1.6 Hz, 1H), 5.37 (dd, *J* = 8.8, 6.8 Hz, 1H), 4.84 (t, *J* = 8.8 Hz, 1H), 4.39 (dd, *J* = 8.8, 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 137.4, 133.5, 132.6, 130.6, 130.2, 129.7, 129.4, 129.2, 127.6, 127.5, 70.5, 62.0; FT-IR (neat) 3063, 3031, 2922, 1760, 1693, 1524, 1485, 1455, 1401, 1366, 1212, 1120, 1067, 1036 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}ClNO_2$: C 65.82, H 4.42, N 5.12, found: C 65.91, H 4.40, N 5.09.

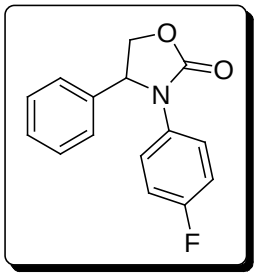


3-(3-Fluorophenyl)-4-phenyloxazolidin-2-one 4c: Yellow liquid; yield 61%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.05 (m, 7H), 6.74 (td, *J* = 8.4, 2.4 Hz, 1H), 5.34 (dd, *J* = 8.4, 5.6 Hz, 1H), 4.75 (t, *J* = 8.4 Hz, 1H), 4.17 (dd, *J* = 8.8, 5.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 161.7, 155.7, 138.8, 138.7, 138.0, 130.2, 130.1, 129.6, 129.1, 126.2, 115.8, 111.5, 111.3, 108.3, 108.0, 69.9, 60.6; FT-IR (neat) 3062, 3032, 2912, 1755, 1614, 1494, 1455, 1397, 1357, 1206, 1117, 1080, 1049 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}FNO_2$: C 70.03, H 4.70, N 5.44, found: C 70.10, H 4.67, N 5.41.

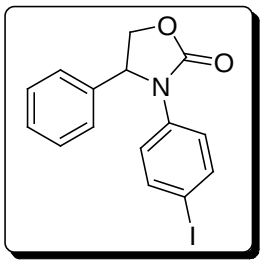


3-(4-Chlorophenyl)-4-phenyloxazolidin-2-one 4d: Yellow liquid; yield 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.27 (m, 5H), 7.26-7.16 (m, 4H), 5.34 (dd, *J* = 8.8, 6.0 Hz, 1H), 4.75 (t, *J* = 8.8 Hz, 1H), 4.16 (dd, *J* = 8.8, 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 137.8, 135.7, 130.0, 129.6, 129.1,

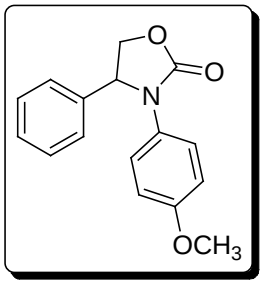
129.0, 126.3, 122.0, 69.9, 60.6; FT-IR (neat) 3027, 2961, 2919, 1755, 1747, 1637, 1495, 1455, 1396, 1354, 1292, 1211, 1129, 1096, 1047 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}ClNO_2$: C 65.82, H 4.42, N 5.12, found: C 65.90, H 4.40, N 5.08.



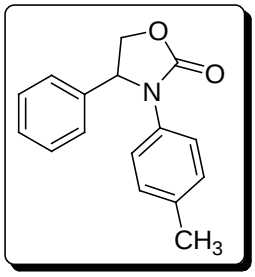
3-(4-Fluorophenyl)-4-phenyloxazolidin-2-one 4e: Yellow liquid; yield 65%; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.25 (m, 7H), 6.94-6.89 (m, 2H), 5.32 (dd, *J* = 8.8, 6.0 Hz, 1H), 4.75 (t, *J* = 8.8 Hz, 1H), 4.18 (dd, *J* = 8.8, 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 158.3, 156.1, 137.8, 133.0, 129.3, 128.9, 126.3, 123.0, 122.9, 115.7, 115.5, 69.7, 60.9; FT-IR (neat) 3062, 3030, 2919, 1754, 1510, 1455, 1400, 1358, 1228, 1159, 1126, 1081, 1047 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}FNO_2$: C 70.03, H 4.70, N 5.44, found: C 70.09, H 4.71, N 5.39.



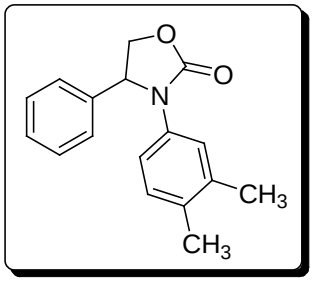
3-(4-Iodophenyl)-4-phenyloxazolidin-2-one 4f: Yellow liquid; yield 84%; ^1H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.8 Hz, 2H), 7.39-7.31 (m, 3H), 7.28 (d, *J* = 6.8 Hz, 2H), 7.19 (d, *J* = 9.2 Hz, 2H), 5.36 (dd, *J* = 8.8, 6.0 Hz, 1H), 4.77 (t, *J* = 8.8 Hz, 1H), 4.19 (dd, *J* = 8.4, 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 137.9, 137.0, 129.6, 129.1, 128.6, 126.2, 122.5, 88.5, 69.9, 60.4; FT-IR (neat) 3063, 3031, 2917, 1755, 1585, 1489, 1455, 1409, 1393, 1350, 1287, 1210, 1129, 1047 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{12}INO_2$: C 49.34, H 3.31, N 3.84, found: C 49.41, H 3.30, N 3.81.



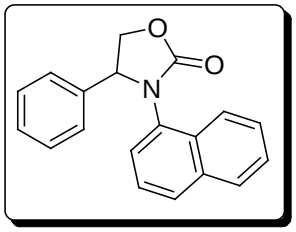
3-(4-Methoxyphenyl)-4-phenyloxazolidin-2-one 4g:[2c] Colourless solid; yield 68%; mp 136-137 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.22 (m, 7H), 6.77 (d, *J* = 7.2 Hz, 2H), 5.30 (dd, *J* = 8.8, 6.4 Hz, 1H), 4.75 (t, *J* = 8.8 Hz, 1H), 4.19 (dd, *J* = 8.4, 6.0 Hz, 1H), 3.70 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 156.4, 138.3, 129.9, 129.2, 128.7, 126.5, 123.3, 114.1, 69.7, 61.2, 55.2; FT-IR (KBr) 3065, 3032, 3003, 2912, 2836, 1751, 1611, 1514, 1457, 1427, 1400, 1297, 1249, 1181, 1126, 1081, 1034 cm^{-1}. Elemental analysis calcd (%) for $C_{16}H_{15}NO_3$: C 71.36, H 5.61, N 5.20, found: C 71.47, H 5.58, N 5.18.



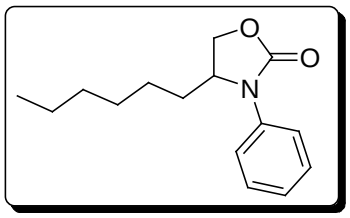
4-Phenyl-3-p-tolyloxazolidin-2-one 4h:[2b] Yellow solid; yield 70%; mp 105-106 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.23 (m, 7H), 7.03 (d, *J* = 8.4 Hz, 2H), 5.33 (dd, *J* = 8.8, 6.4 Hz, 1H), 4.72 (t, *J* = 8.8 Hz, 1H), 4.14 (dd, *J* = 8.8, 6.4 Hz, 1H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 138.5, 134.5, 129.5, 129.4, 128.8, 126.4, 121.2, 69.9, 60.9, 20.8; FT-IR (KBr) 3071, 3032, 2922, 2861, 1743, 1732, 1515, 1457, 1401, 1349, 1227, 1130, 1044 cm^{-1}. Elemental analysis calcd (%) for $C_{16}H_{15}NO_2$: C 75.87, H 5.97, N 5.53, found: C 75.96, H 5.95, N 5.50.



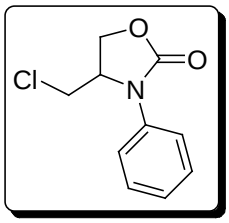
3-(3,4-Dimethylphenyl)-4-phenyloxazolidin-2-one 4j: Colourless solid; yield 78%; mp 130-131 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.26 (m, 5H), 7.22 (s, 1H), 6.96 (s, 2H), 5.33 (dd, *J* = 8.8, 6.4 Hz, 1H), 4.75 (t, *J* = 8.8 Hz, 1H), 4.16 (dd, *J* = 8.8, 6.4 Hz, 1H), 2.15 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 138.6, 137.2, 134.7, 133.3, 129.9, 129.3, 128.7, 126.4, 122.7, 118.6, 69.8, 60.8, 20.0, 19.1; FT-IR (KBr) 3062, 3032, 2970, 2938, 2920, 1746, 1615, 1506, 1455, 1398, 1356, 1222, 1211, 1135, 1112, 1050 cm^{-1}. Elemental analysis calcd (%) for $C_{17}H_{17}NO_2$: C 76.3, H 6.41, N 5.24, found: C 76.44, H 6.39, N 5.26.



3-(Naphthalen-1-yl)-4-phenyloxazolidin-2-one 4k: Colourless solid; yield 71%; mp 152-153 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 6.8 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 1H), 7.31-7.21 (m, 6H), 7.12 (s, 1H), 5.36 (s, 1H), 4.93 (t, *J* = 8.8 Hz, 1H), 4.49 (dd, *J* = 8.8, 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 137.7, 134.6, 132.5, 130.1, 129.1, 128.6, 127.3, 126.9, 126.4, 125.3, 122.6, 70.3, 63.5; FT-IR (KBr) 3062, 3031, 2927, 1741, 1601, 1501, 1459, 1411, 1376, 1300, 1256, 1215, 1135, 1089, 1052, 1026 cm^{-1}. Elemental analysis calcd (%) for $C_{19}H_{15}NO_2$: C 78.87, H 5.23, N 4.84, found: C 78.96, H 5.21, N 4.81.



4-Hexyl-3-phenyloxazolidin-2-one 4l: Pale yellow solid; yield 71%; mp 78-79 °C (from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.8 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.10 (t, *J* = 7.6 Hz, 1H), 4.60 (q, *J* = 6.0 Hz, 1H), 4.05 (t, *J* = 8.4 Hz, 1H), 3.63 (dd, *J* = 8.8, 7.6 Hz, 1H), 1.88-1.79 (m, 1H), 1.74-1.65 (m, 1H), 1.50-1.46 (m, 1H), 1.42-1.17 (m, 7H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 138.4, 128.9, 123.8, 118.1, 73.1, 50.4, 34.9, 31.6, 28.9, 24.4, 22.5, 22.4, 14.0; FT-IR (KBr) 3061, 2961, 2930, 2858, 1750, 1602, 1503, 1486, 1407, 1380, 1311, 1220, 1126, 1082 cm^{-1}. Elemental analysis calcd (%) for $C_{15}H_{21}NO_2$: C 72.84, H 8.56, N 5.66, found: C 72.97, H 8.55, N 5.63.



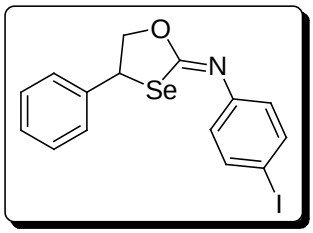
4-(Chloromethyl)-3-phenyloxazolidin-2-one 4m: Colourless solid; yield 73%; mp 98-99 °C(from hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.8 Hz, 2H), 7.36 (t, *J* = 8.0 Hz, 2H), 7.13 (t, *J* = 7.6 Hz, 1H), 4.85-4.80 (m, 1H), 4.13 (t, *J* = 9.2 Hz, 1H), 3.93 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.78-3.69 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.1, 137.9, 129.3, 124.5, 118.5, 71.0, 48.2, 44.7; FT-IR (KBr) 2947, 2862, 1747, 1611, 1504, 1466, 1385, 1065 cm^{-1}. Elemental analysis calcd (%) for $C_{10}H_{10}ClNO_2$: C 56.75, H 4.76, N 6.62, found: C 56.85, H 4.77, N 6.59.

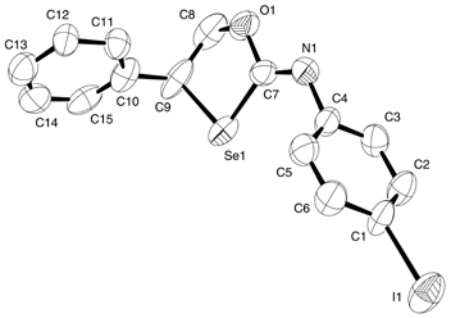
References

1 a) G. W. Gokel, R. P. Widera, W. P. Weber, *Org. Synth.*,1976**, 55**, 96.

2 a) I. Shibata, A. Baba, H. Iwasaki, H. Matsuda, *J. Org. Chem.* 1986, **51**, 2177; b) J. E. Herweh, T. A. Foglia, D. Swern, *J. Org. Chem.* 1968, **33**, 4029; c) B. Mallesham, B. M. Rajesh, P. R. Reddy, D. Srinivas, S. Trehan, *Org. Lett.*. 2003, **5**, 963.

Crystal Structure of 3f





Crystal number: Summary of Data CCDC 843128. Thermal ellipsoids are drawn at a 40% probability level. Hydrogen atoms have been omitted for clarity.

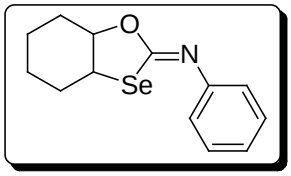
Formula: $C_{15}\,H_{12}\,I_1\,N_1\,O_1\,Se_1$

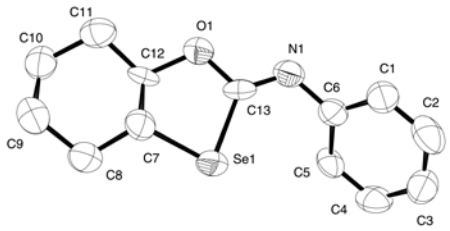
Unit cell parameters: a 12.7577(7) b 10.0993(6) c 11.6512(7) beta 94.400(3) space group P21/c

Datablock:

Bond precision:	C-C = 0.0146 A		Wavelength=0.71073
Cell:	a=12.7577(7)	b=10.0993(6)	c=11.6512(7)
	alpha=90	beta=94.400(3)	gamma=90
Temperature:	296 K		
	Calculated		Reported
Volume	1496.76(15)		1496.76(15)
Space group	P 21/c		P2(1)/c
Hall group	-P 2ybc		?
Moiety formula	$C_{15}\,H_{12}$ I N O Se		$C_{15}\,H_{12}$ I N O Se
Sum formula	$C_{15}\,H_{12}$ I N O Se		$C_{15}\,H_{12}$ I N O Se
Mr	428.12		428.12
Dx,g cm-3	1.900		1.904
Z	4		4
Mu (mm-1)	4.562		4.563
F000	816.0		816.0
F000'	814.04		
h,k,lmax	17,13,15		17,13,15
Nref	3809		3709
Tmin,Tmax	0.231,0.292		0.231,0.292
Tmin'	0.194		
Correction method	MULTI-SCAN		
Data completeness= 0.974		Theta(max)= 28.540	
R(reflections)= 0.0532(3585)		wR2(reflections)= 0.2460(3398)	
S = 1.420		Npar= 172	

Crystal structure of 3q





Crystal number: Summary of Data CCDC 843126. Thermal ellipsoids are drawn at a 85% probability level. Hydrogen atoms have been omitted for clarity.

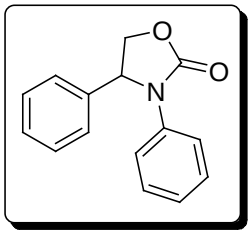
Formula: $C_{15}\,H_{13}\,N_1\,O_2$

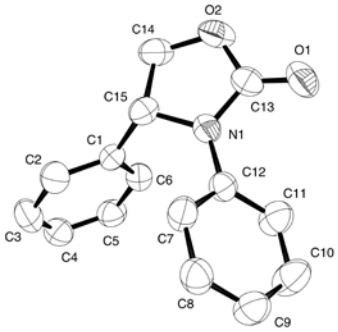
Unit cell parameters: a 6.7304(4) b 7.6804(4) c 23.0785(13) beta 92.209(4) space group P21/c

Datablock:

Bond precision:	C-C = 0.0079 A		Wavelength=0.71073
Cell:	a=6.7304(4)	b=7.6804(4)	c=23.0785(13)
	alpha=90	beta=92.209(4)	gamma=90
Temperature:	296 K		
	Calculated		Reported
Volume	1192.09(12)		1192.09(12)
Space group	P 21/c		P2(1)/c
Hall group	-P 2ybc		?
Moiety formula	$C_{13}\,H_{15}$ N O Se		$C_{13}\,H_{15}$ N O Se
Sum formula	$C_{13}\,H_{15}$ N O Se		$C_{13}\,H_{15}$ N O Se
Mr	280.22		280.22
Dx,g cm-3	1.561		1.561
Z	4		4
Mu (mm-1)	3.128		3.128
F000	568.0		568.0
F000'	567.82		
h,k,lmax	6,7,22		6,7,21
Nref	1115		1084
Tmin,Tmax	0.298,0.391		0.298,0.391
Tmin'	0.275		
Correction method	MULTI-SCAN		
Data completeness= 0.972		Theta(max)= 19.980	
R(reflections)= 0.0515(1022)		wR2(reflections)= 0.1223(1084)	
S = 1.030		Npar= 145	

Crystal structure of 4a





Crystal number: Summary of Data CCDC 843127. Thermal ellipsoids are drawn at a 40% probability level. Hydrogen atoms have been omitted for clarity.

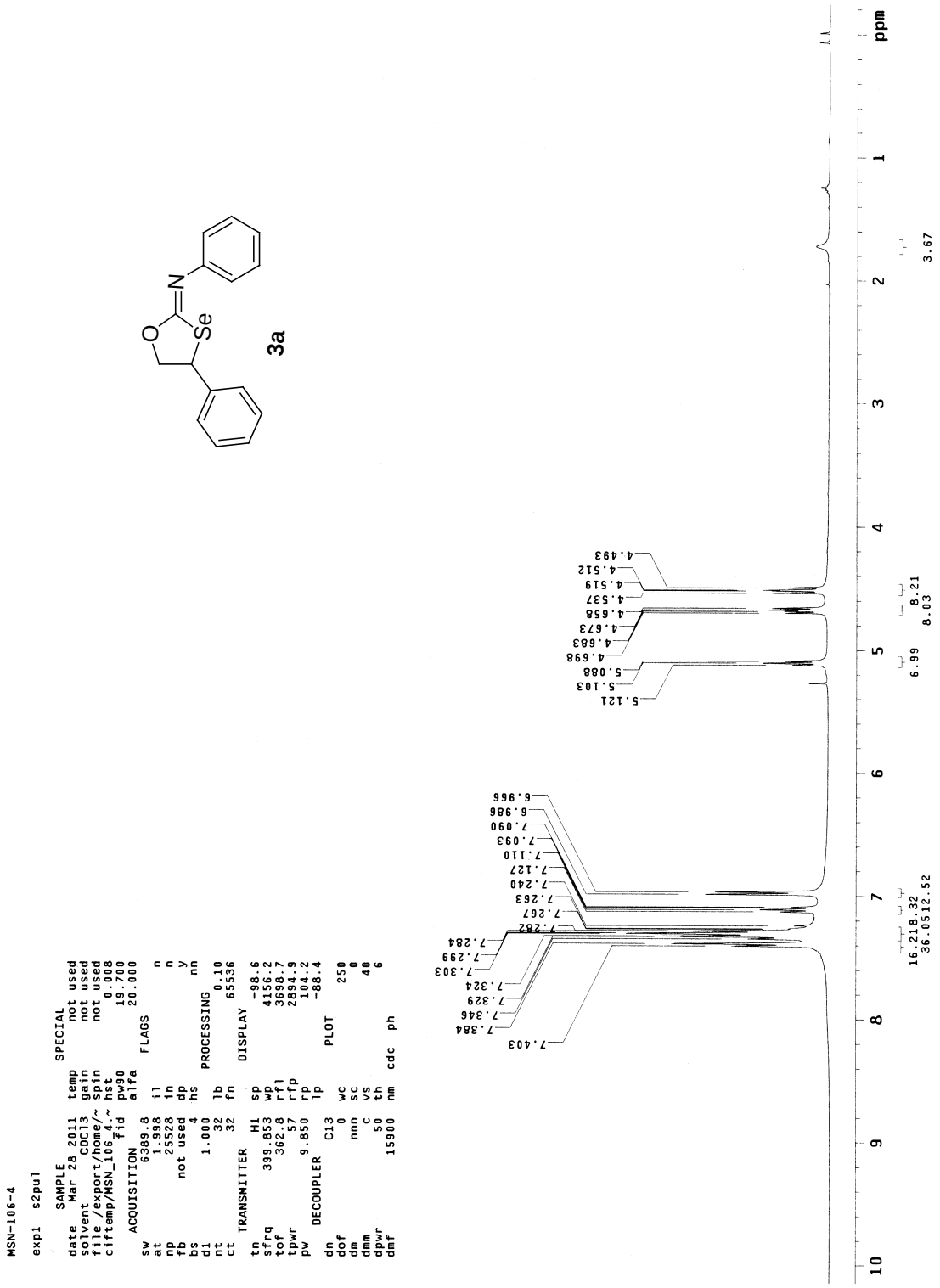
Formula: $C_{15}\,H_{13}\,N_1\,O_2$

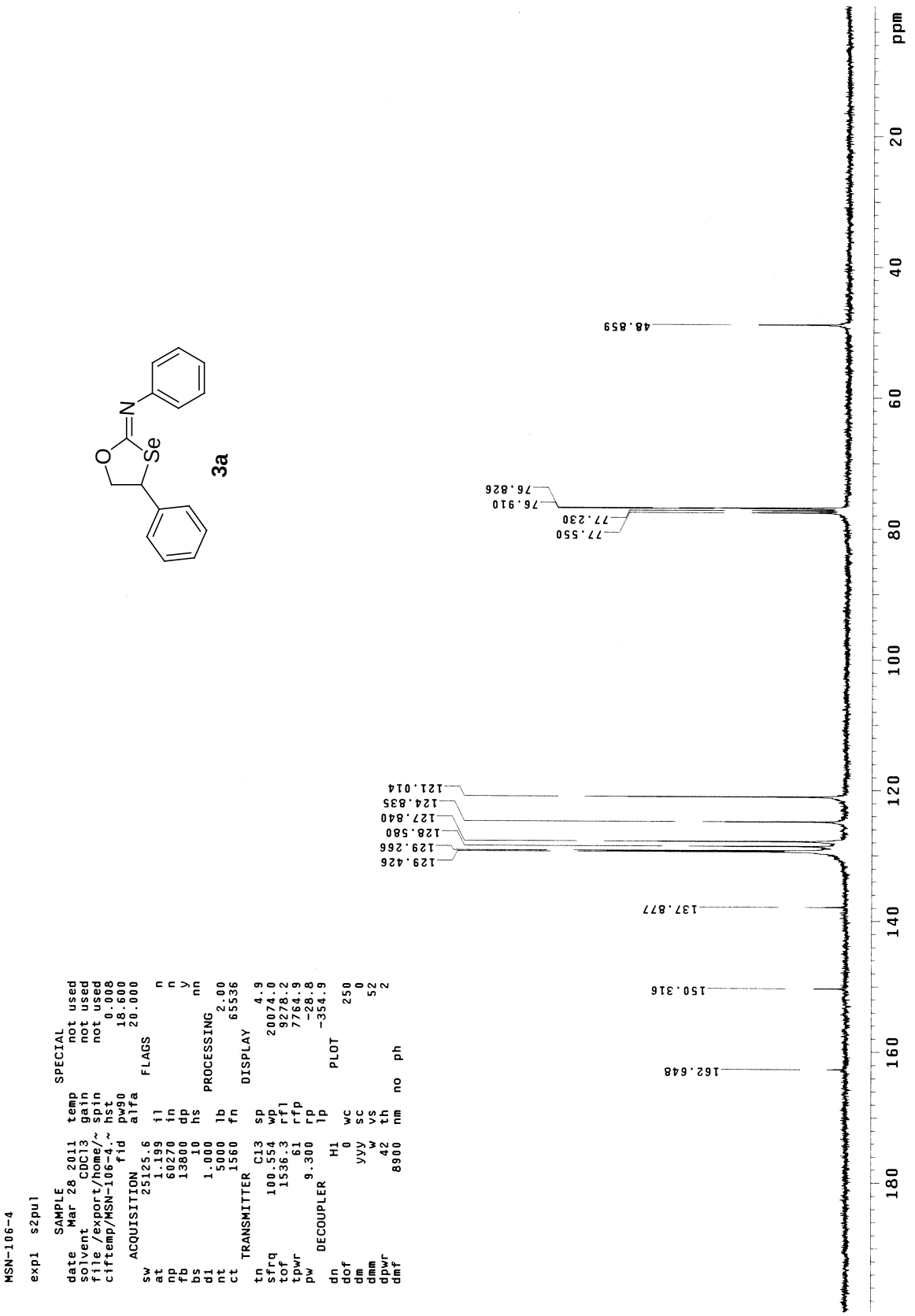
Unit cell parameters: a 10.4808(9) b 8.9744(8) c 13.0800(12) beta 91.143(5) space group P21/n

Data block:

Bond precision:	C-C = 0.0023 A		Wavelength=0.71073
Cell:	a=10.4808(9)	b=8.9744(8)	c=13.0800(12)
	alpha=90	beta=91.143(5)	gamma=90
Temperature:	296 K		
	Calculated		Reported
Volume	1230.05(19)		1230.05(19)
Space group	P 21/n		P2(1)/n
Hall group	-P 2yn		?
Moiety formula	$C_{15}\,H_{13}\,N\,O_2$		$C_{15}\,H_{13}\,N\,O_2$
Sum formula	$C_{15}\,H_{13}\,N\,O_2$		$C_{15}\,H_{13}\,N\,O_2$
Mr	239.26		239.26
Dx,g cm-3	1.292		1.292
Z	4		4
Mu (mm-1)	0.086		0.086
F000	504.0		504.0
F000'	504.23		
h,k,lmax	12,11,16		12,10,16
Nref	2453		2401
Tmin,Tmax	0.972,0.978		0.973,0.978
Tmin'	0.972		
Correction method	MULTI-SCAN		
Data completeness= 0.979		Theta(max)= 26.140	
R(reflections)= 0.0515(1779)		wR2(reflections)= 0.1508(2401)	
S = 1.085		Npar= 164	

^{1}H and ^{13}C NMR Spectra

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date Mar 28 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-106-4.~
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at 1.199
np 60270
fb 13800
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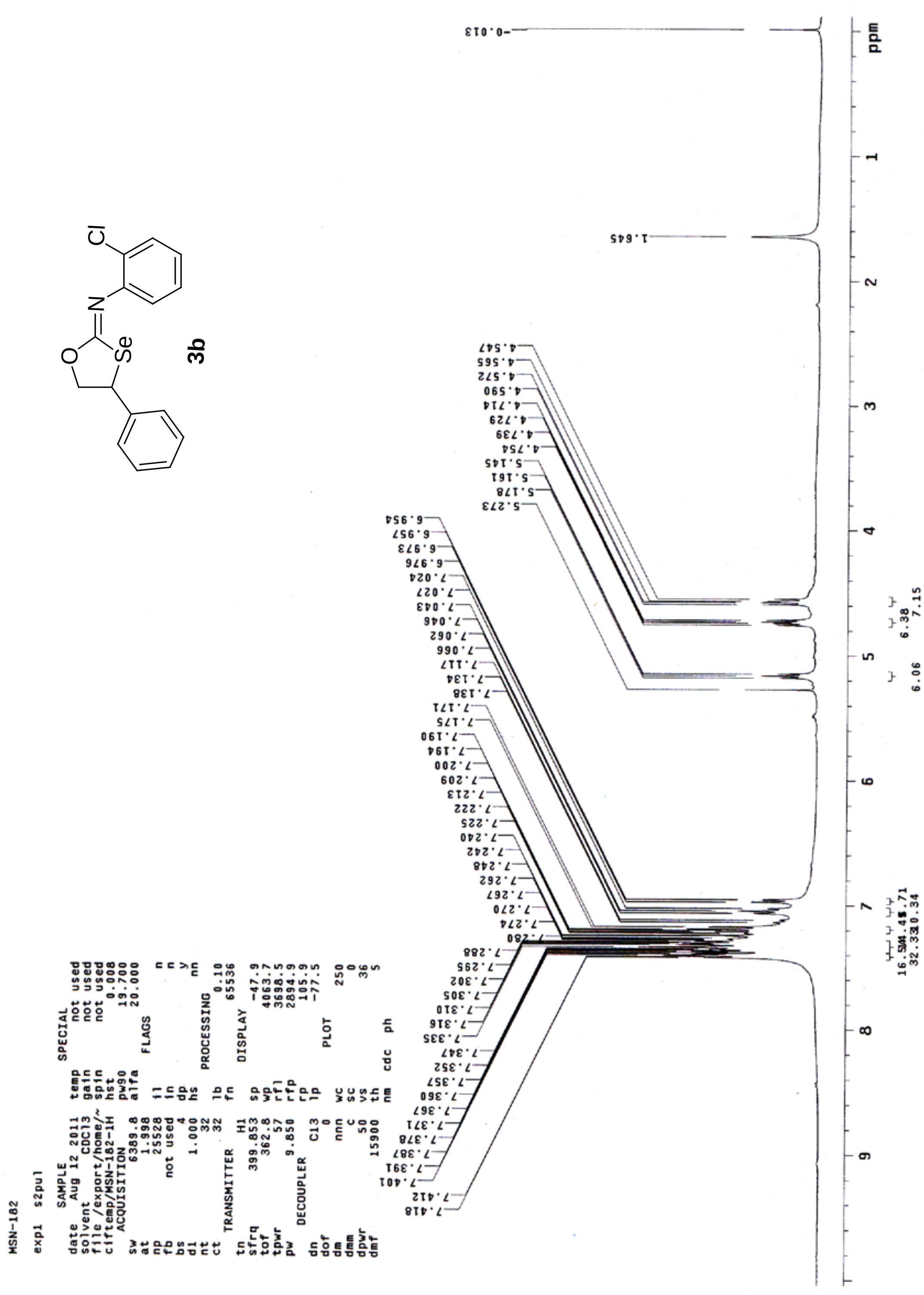
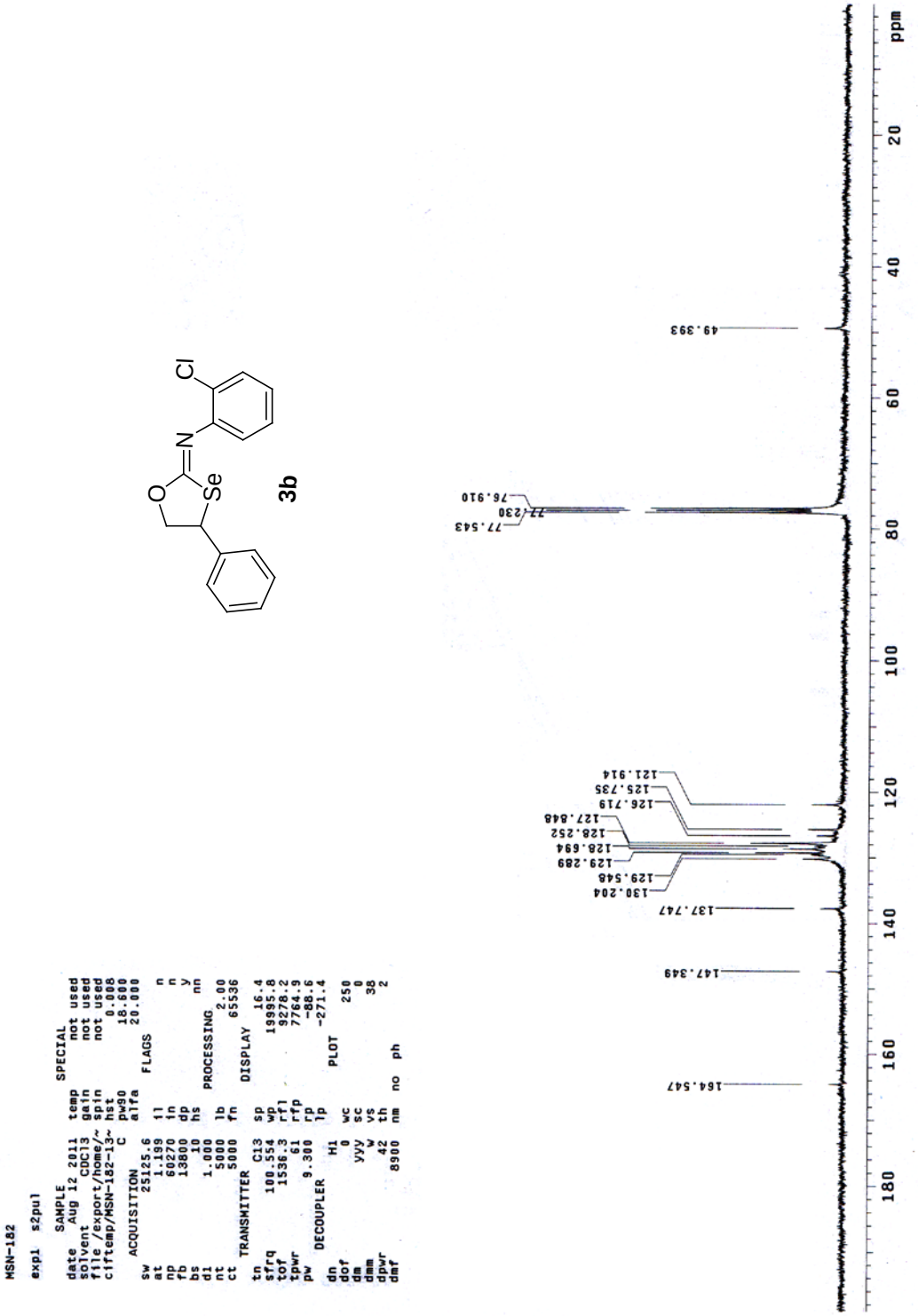
d1 1.000
nt 5000
ct 1560
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
temp
gain
spin
hst
pw90
alfa
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp 4.9
wp 20074.0
rfl 9278.2
rfp 7764.9
rp -28.8
lp -354.9
PLOT
wc 250
sc 0
vs 52
th 2
SPECIAL
not used
not used
not used
0.008
18.600
20.000
nm ph
no
3a
O
N
Se
48.859
76.826
76.910
77.230
77.550
121.014
124.835
127.840
128.580
129.266
129.426
137.877
150.316
162.648
ppm
180
160
140
120
100
80
60
40
20


MSN-182
3b
Se
O
N
Cl
ppm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expl s2pul
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date Aug 12 2011
solvent CDCl3
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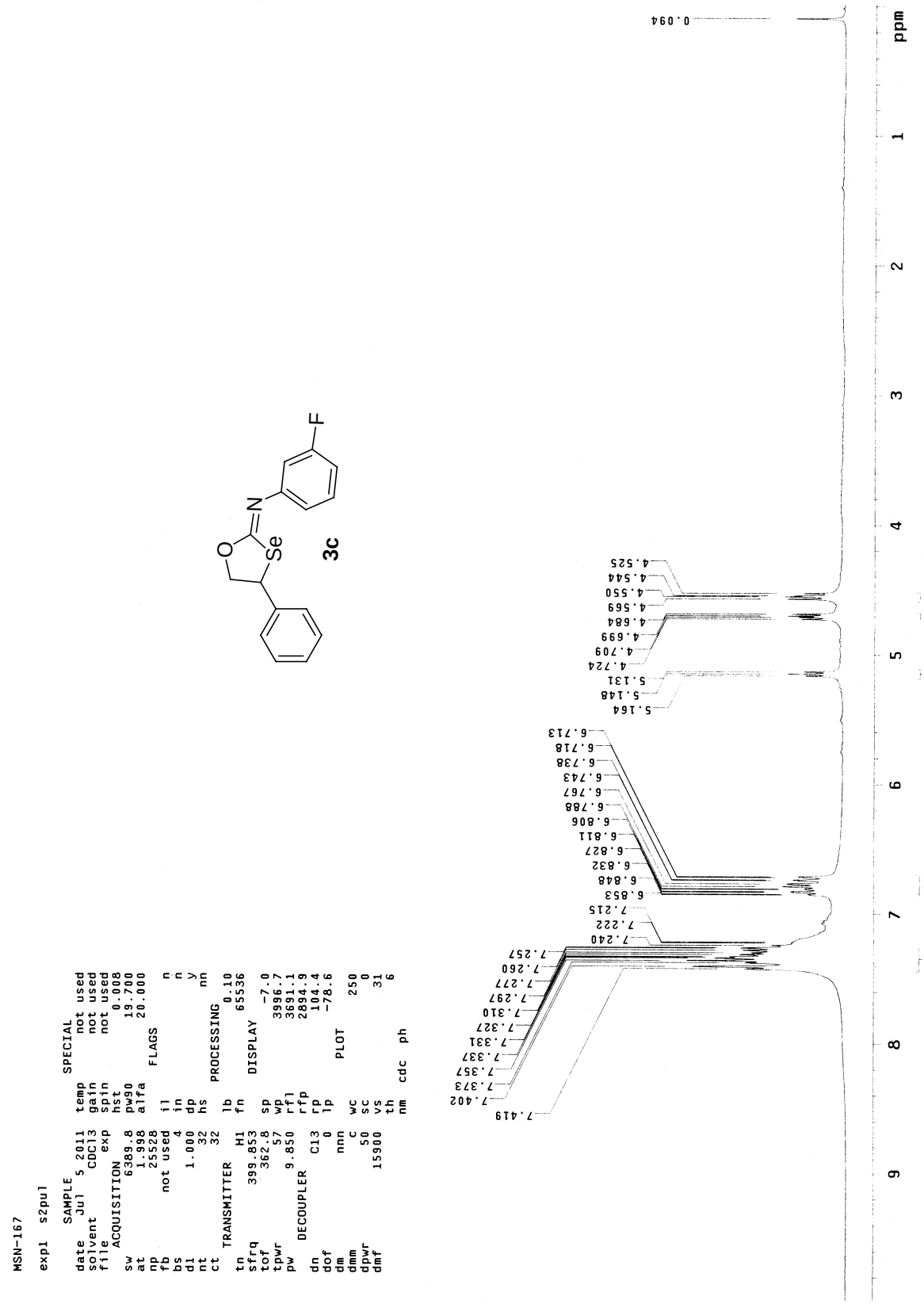
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 5000
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp 16.4
wp 19995.8
rfl 9278.2
rfp 7764.9
rp -88.6
lp -271.4
PLOT
wc 250
sc 0
vs 38
th 2
nm no ph
Cl
N
O
Se
3b
49.393
76.910
77.230
77.543
121.914
125.735
126.719
127.848
128.252
128.694
129.289
129.548
130.204
137.747
147.349
164.547
ppm
180
160
140
120
100
80
60
40
20


MSN-167
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SAMPLE
date Jul 5 2011
solvent CDCl3
file exp
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at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
at1a 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -7.0
wp 3996.7
rfl 3691.1
rfp 2894.9
rp 104.4
lp -78.6
PLOT
wc 250
sc 0
vs 31
th 6
cdc ph
nm
F
N
Se
O
3c
0.094
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4.544
4.550
4.569
4.684
4.699
4.709
4.724
5.131
5.148
5.164
6.713
6.718
6.738
6.743
6.767
6.788
6.806
6.811
6.827
6.832
6.848
6.853
7.215
7.222
7.240
7.257
7.260
7.277
7.297
7.310
7.327
7.331
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7.357
7.373
7.402
7.419
7.35
8.59
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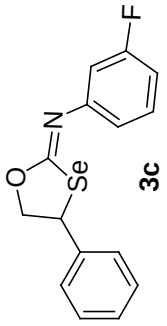
51.36
24.50
ppm


F
N
O
Se

3c

MSN-167
expl s2pul
SAMPLE
date Jul 5 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 2100
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -131.6
wp 20271.8
rfl 9282.8
rfp 7764.9
rp -17.2
lp -414.5
PLOT
wc 250
sc 0
vs 27
th 2
nm no ph
49.088
76.917
76.994
77.230
77.550
108.438
108.659
111.496
111.702
116.728
127.779
128.664
129.281
130.578
130.669
137.602
151.795
151.894
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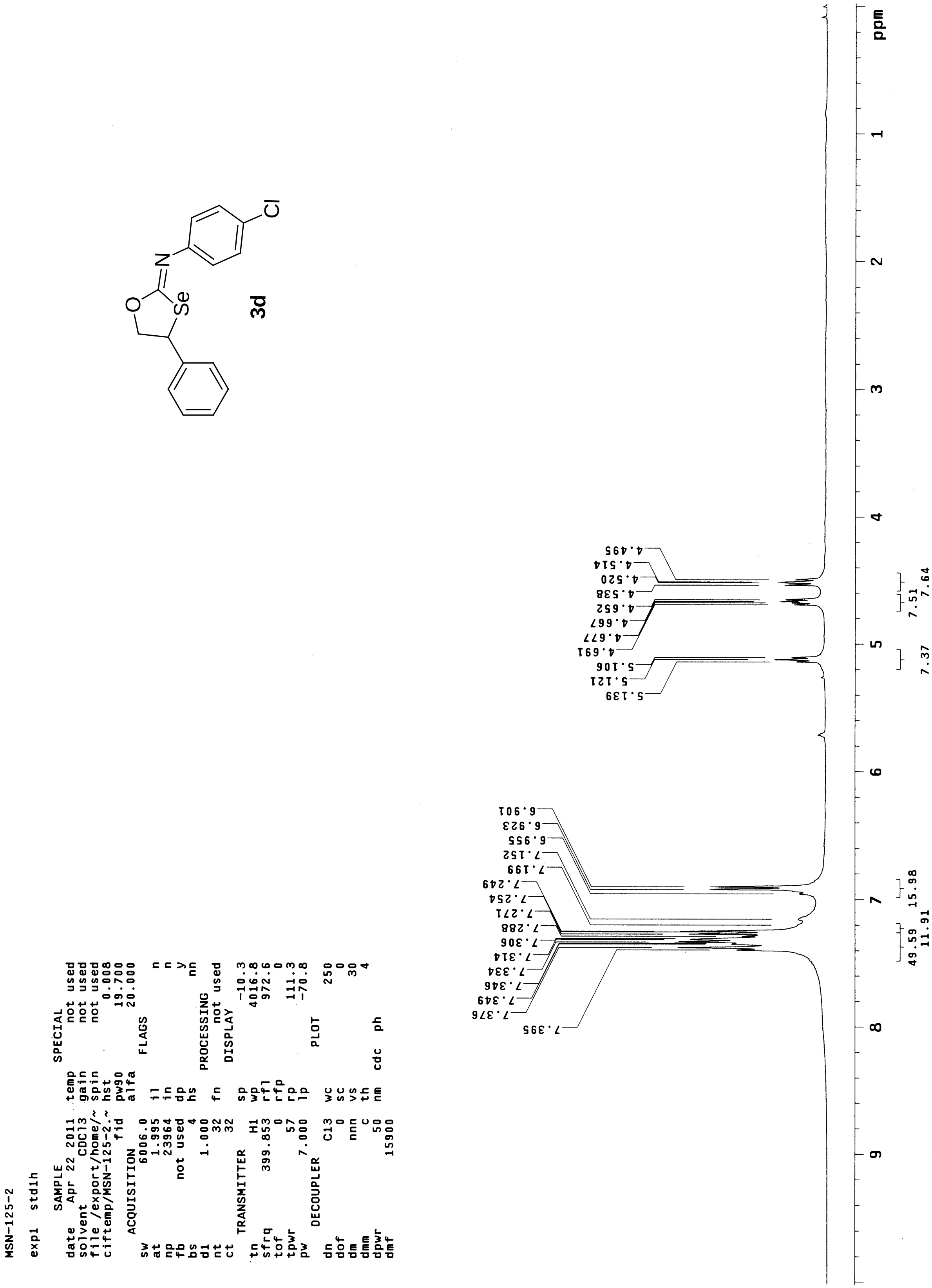
163.578
164.524
ppm


3d
MSN-125-2
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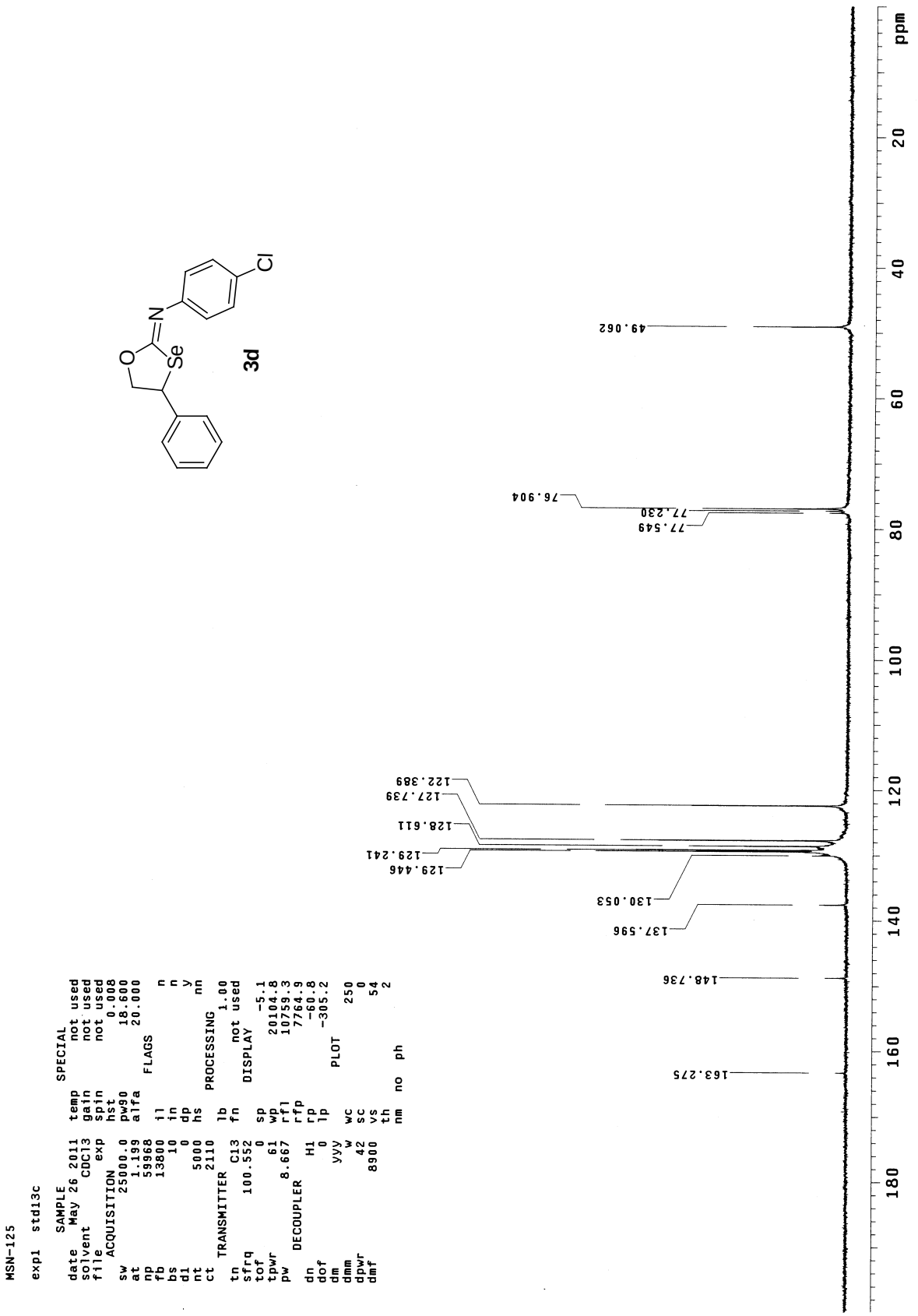
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solvent CDCl3
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np 59968
fb 13800
bs 10
di 0
nt 5000
ct 2110
TRANSMITTER
tn C13
sfrq 100.552
tof 0
tpwr 61
pw 8.667
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -5.1
wp 20104.8
rfl 10759.3
rfp 7764.9
rp -60.8
lp -305.2
PLOT
wc 250
sc 0
vs 54
th 2
nm no ph
3d
Cl
N
Se
O
49.062
76.904
77.230
77.549
122.389
127.739
128.611
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137.596
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163.275
ppm
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160
140
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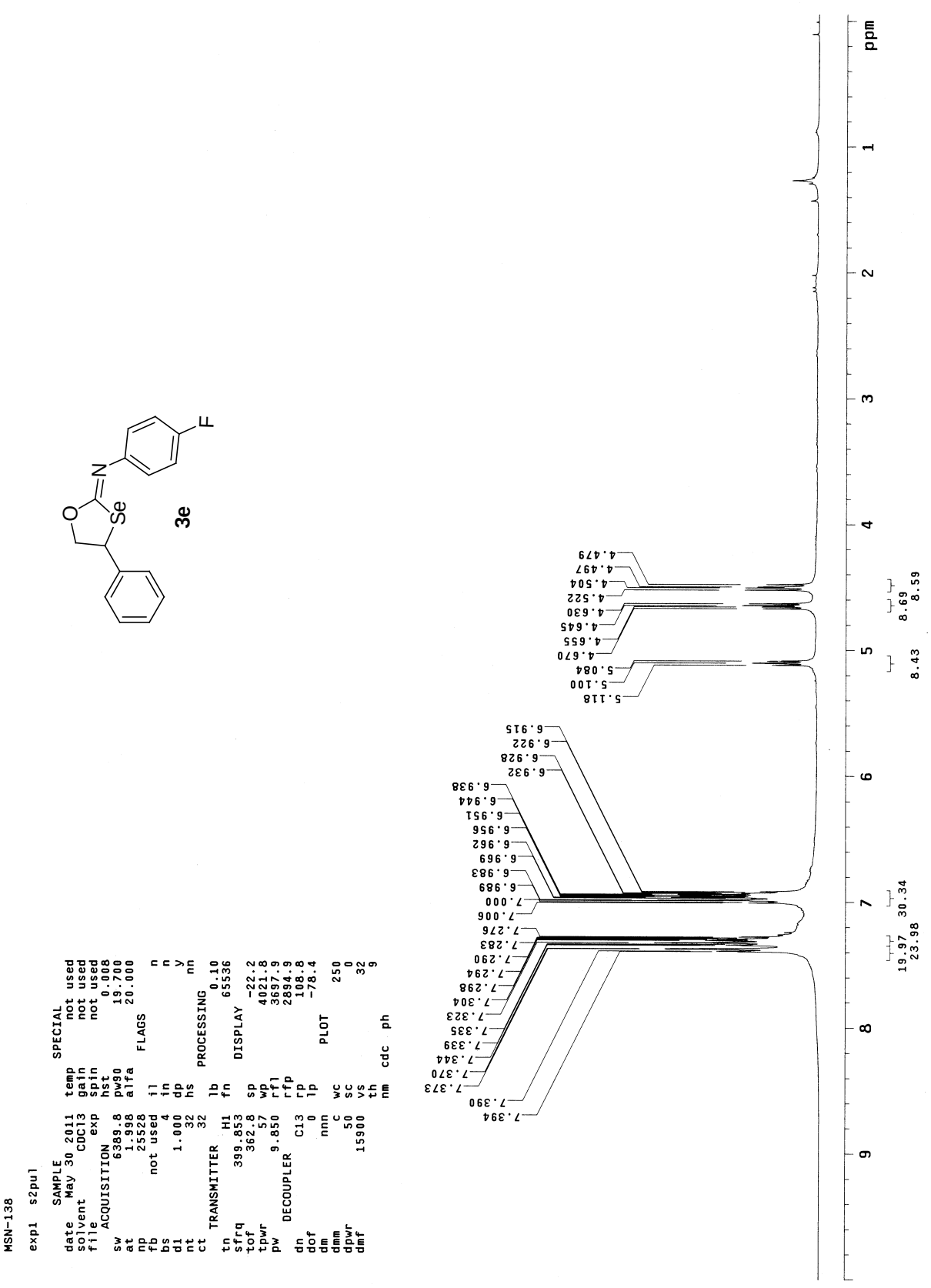
100
80
60
40
20


MSN-138
expl s2pul
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date May 30 2011
solvent CDCl3
file exp
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at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -22.2
wp 4021.8
rfl 3697.9
rfp 2894.9
rp 108.8
lp -78.4
PLOT
wc 250
sc 0
vs 32
th 9
nm cdc ph
3e
F
N
Se
O
ppm
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8.69
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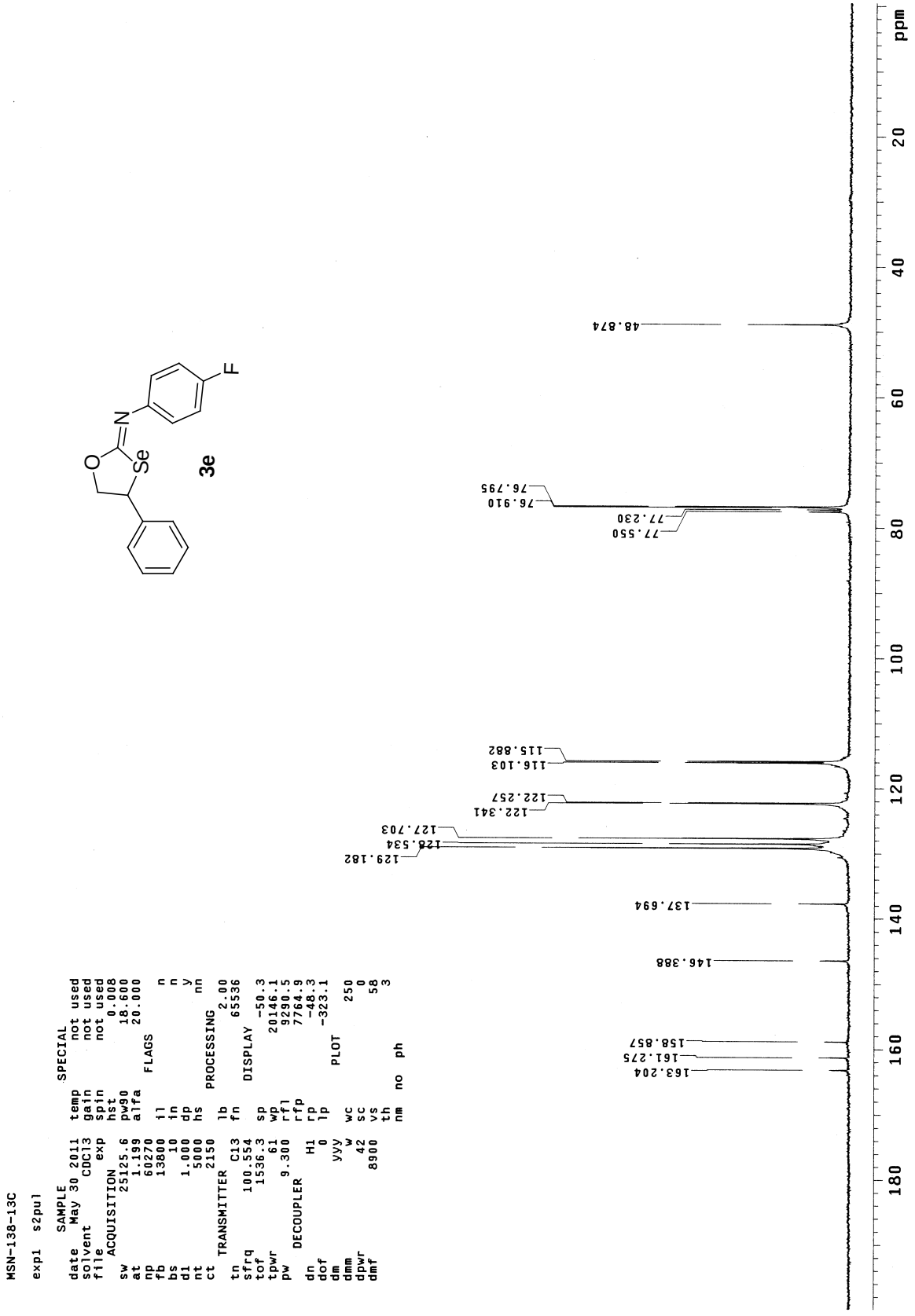
19.97
30.34
23.98


MSN-138-13C
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date May 30 2011
solvent CDCl3
file exp
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at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 2150
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -50.3
wp 20146.1
rfl 9290.5
rfp 7764.9
rp -48.3
lp -323.1
PLOT
wc 250
sc 0
vs 58
th 3
nm no ph
3e
48.874
76.795
76.910
77.230
77.550
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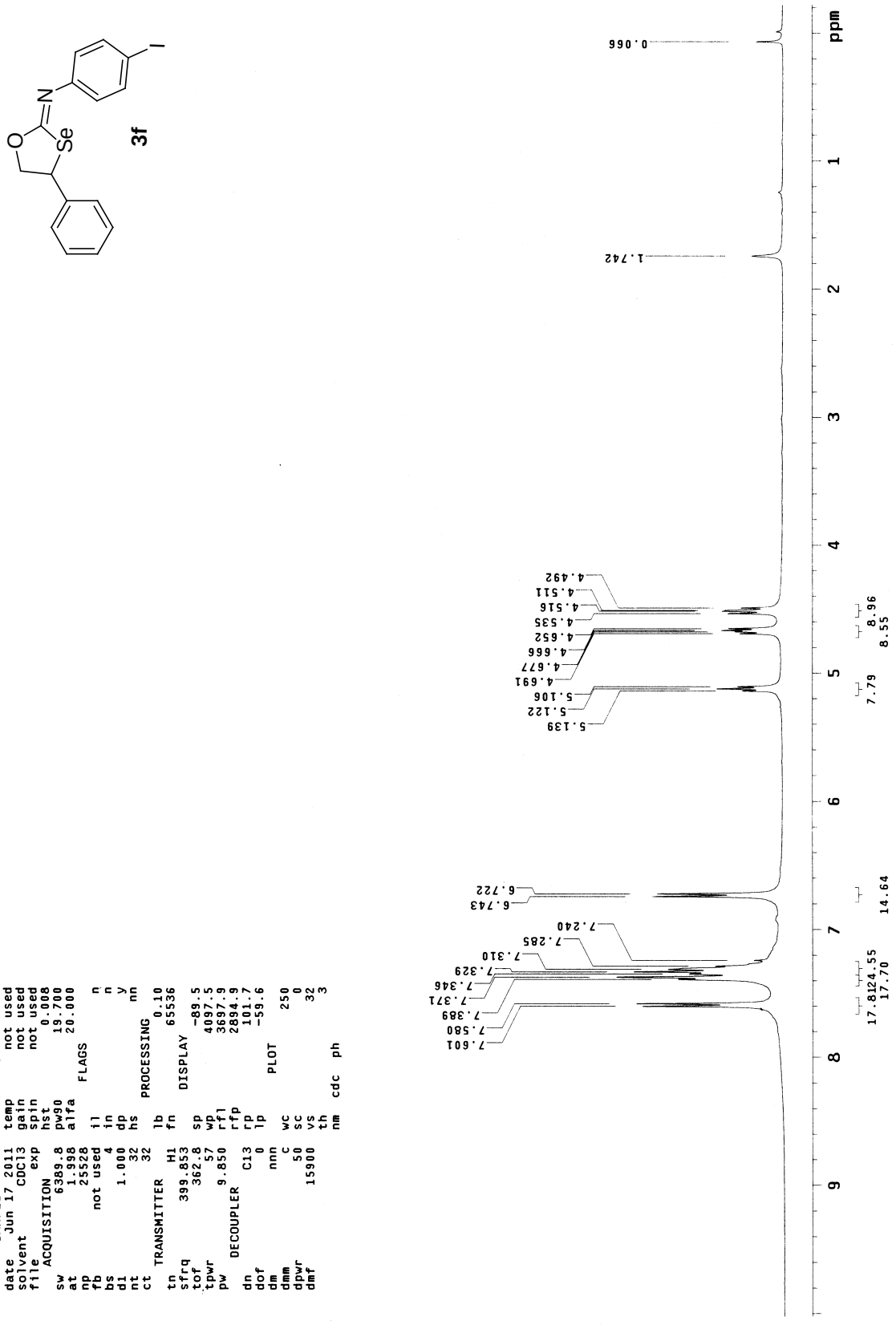
ppm


3f
MSN-147
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date Jun 17 2011
solvent CDCl3
file exp
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at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL not used
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -89.5
wp 4097.5
rfl 3697.9
rfp 2894.9
rp 101.7
lp -59.6
PLOT
wc 250
sc 0
vs 32
th 3
nm cdc ph
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1.742
4.492
4.511
4.516
4.535
4.652
4.666
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4.691
5.106
5.122
5.139
6.722
6.743
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7.310
7.329
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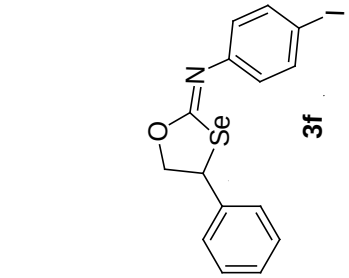
8.96
8.55



3f

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MSN-147

exp1  s2pul

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solvent CDCl3         gain          not used
file    exp           spin          0.008
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sw      25125.6       pw90          20.000
at      1.199         alfa
np      60270                 FLAGS
fb      13800         il            n
bs      10            in            n
d1      1.000         dp            y
nt      7000          hs            nn
ct      2190                PROCESSING
                                    2.00
  TRANSMITTER         lb
tn      C13           fn            65536
sfrq    100.554               DISPLAY
tof     1536.3        sp            -226.7
tpwr    61            wp            20370.7
pw      9.300         rfl           9279.0
  DECOUPLER           rfp           7764.9
dn      H1            rp            -12.5
dof     0             lp            -400.0
dm      yyy                   PLOT
dmm     w             wc            250
dpwr    42            sc            0
dmf     8900          vs            22
                      th            2
                      nm  no  ph
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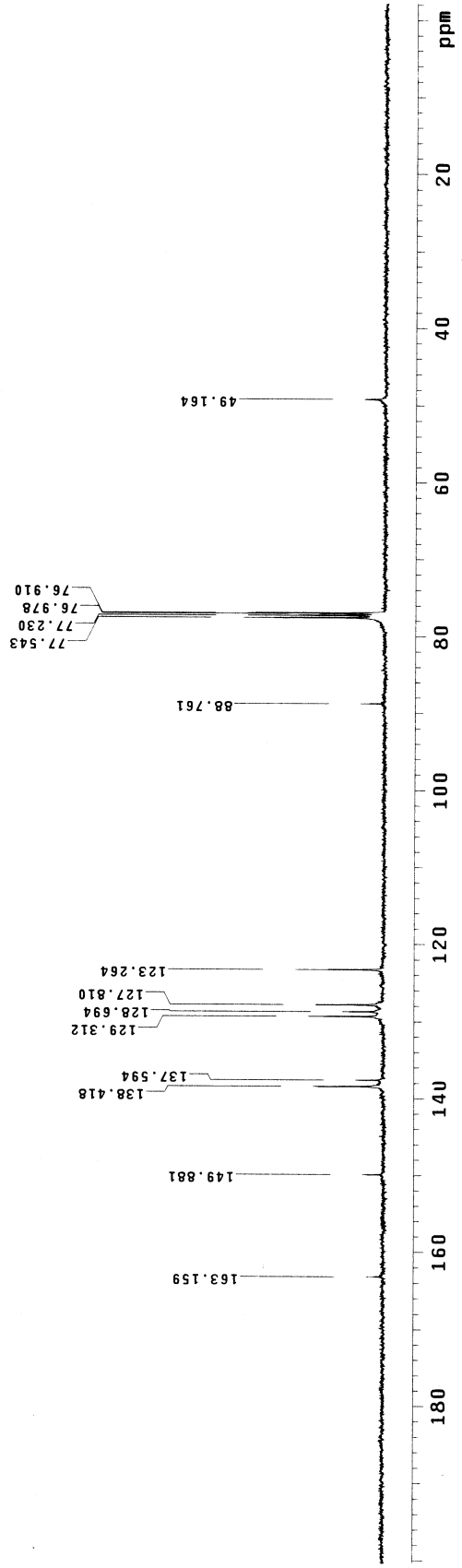

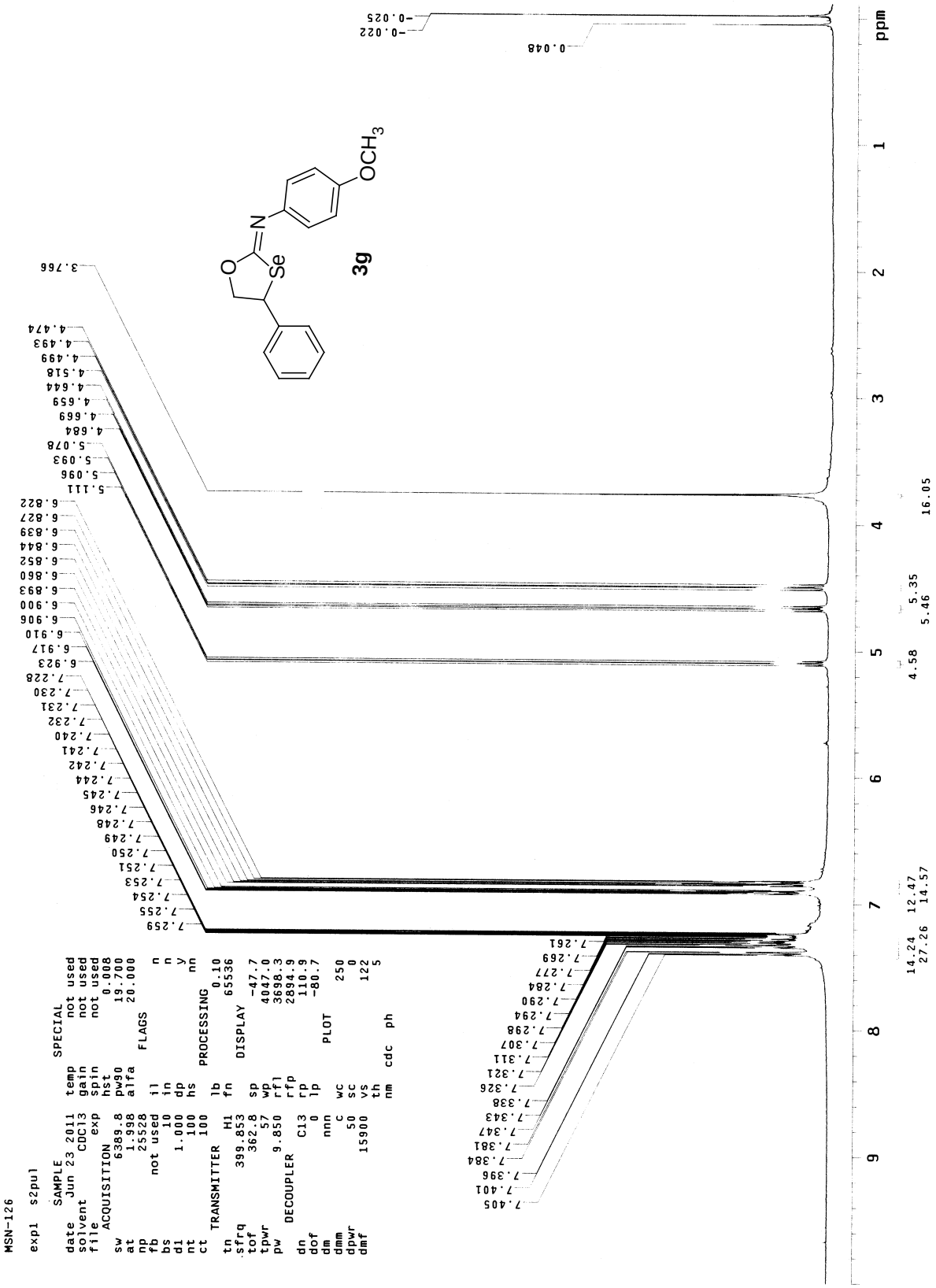
3g
OCH3
N
O
Se
ppm
MSN-126
expl s2pul
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date Jun 23 2011
solvent CDCl3
file exp
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at 1.998
np 25528
fb not used
bs 10
d1 1.000
nt 100
ct 100
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
Spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
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in n
dp y
hs nn
PROCESSING
lb 0.10
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wp 4047.0
rfl 3698.3
rfp 2894.9
rp 110.9
lp -80.7
PLOT
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sc 0
vs 122
th 5
cdc ph
nm
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-0.022
0.048
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4.493
4.499
4.518
4.644
4.659
4.669
4.684
5.078
5.093
5.096
5.111
6.822
6.827
6.839
6.844
6.852
6.860
6.893
6.900
6.906
6.910
6.917
6.923
7.228
7.230
7.231
7.232
7.240
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7.242
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7.245
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7.250
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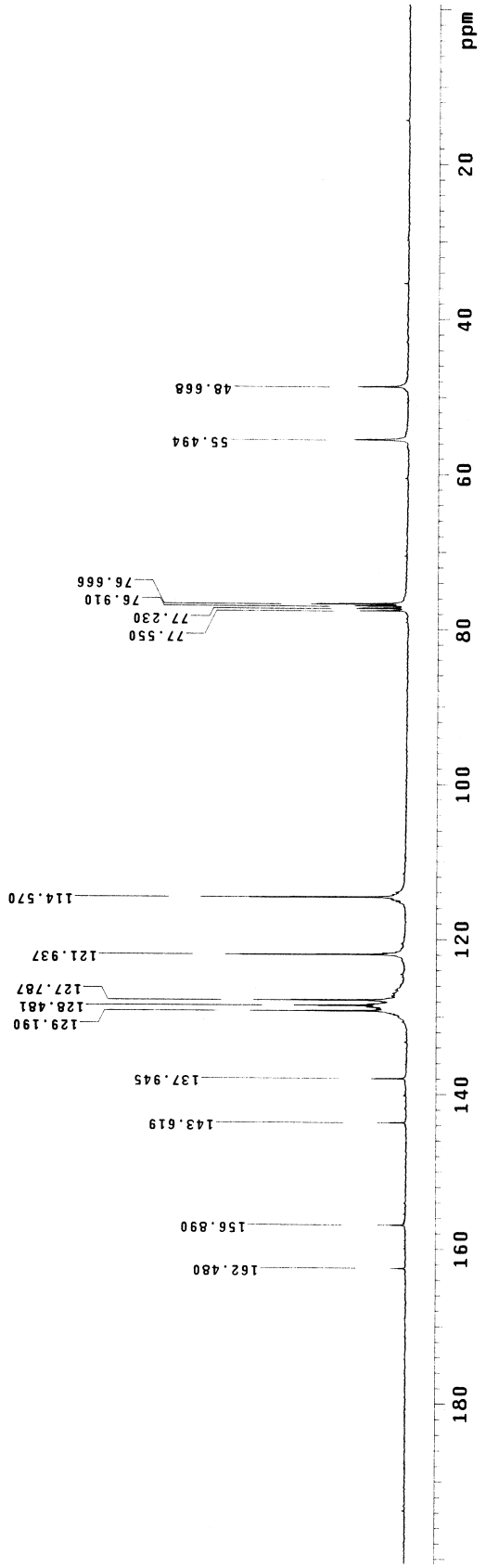
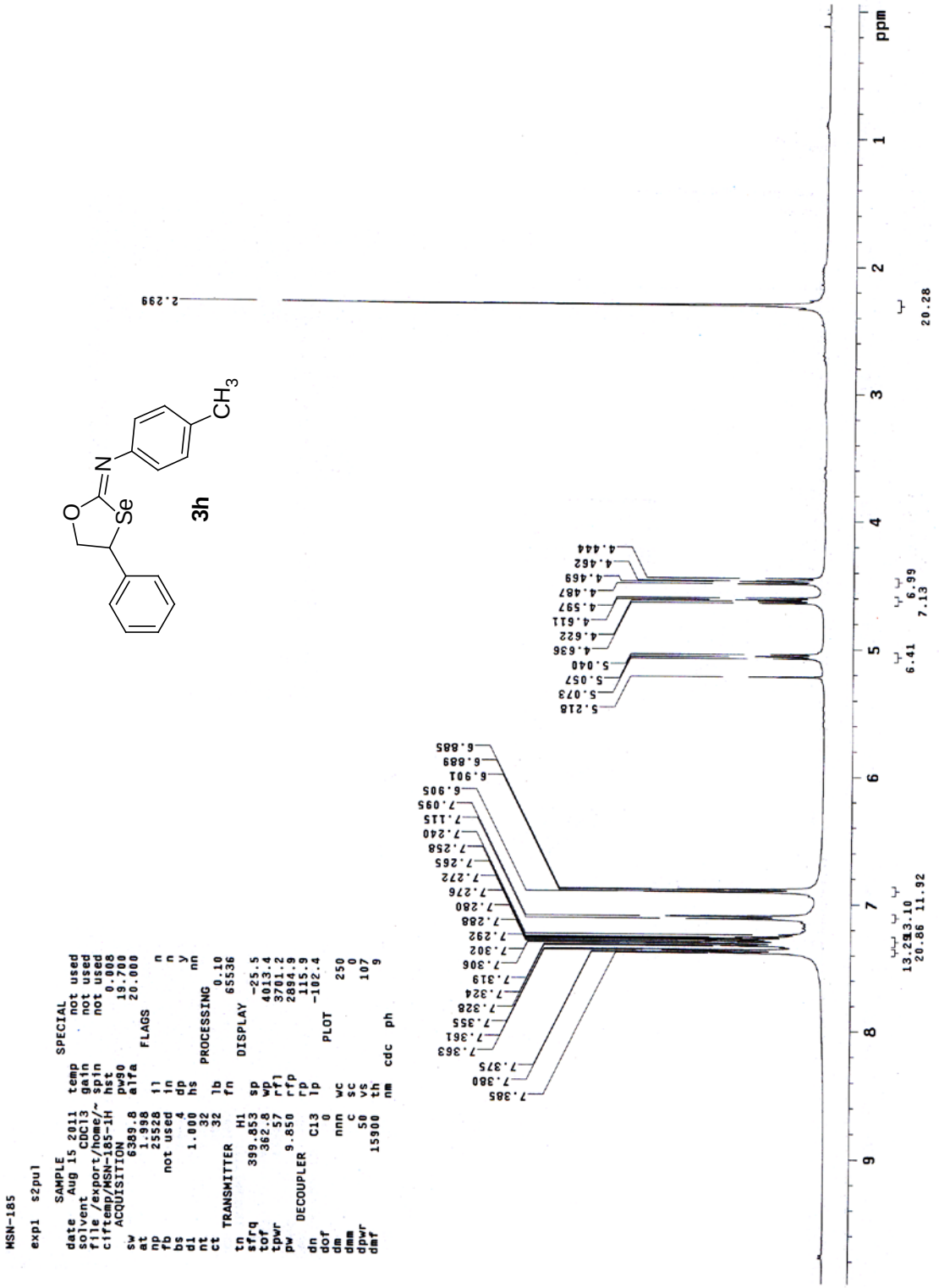
7.298
7.307
7.311
7.321
7.326
7.338
7.343
7.347
7.381
7.384
7.396
7.401
7.405
14.24
27.26
12.47
14.57
4.58
5.46
5.35
16.05


MSN-126-2
expl s2pul
SAMPLE
date May 4 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-126-2~~
13c.fid
ACQUISITION
25125.6
1.199
60270
13800
10
1.000
12000
12000
TRANSMITTER
C13
100.554
1536.3
61
9.300
DECOUPLER
H1
0
yyy
y
42
8900
SPECIAL
not used
not used
not used
0.008
18.600
20.000
FLAGS
n
n
y
nn
PROCESSING
2.00
65536
DISPLAY
-67.2
20238.8
9284.4
7764.9
-56.2
-332.5
PLOT
250
0
33
11
temp
gain
spin
hst
pw90
alfa
sw
at
np
fb
bs
d1
nt
ct
tn
sfrq
tof
tpwr
pw
dn
dof
dm
dmm
dpwr
dmf
il
in
dp
hs
lb
fn
sp
wp
rfl
rfp
rp
lp
wc
sc
vs
th
nm
no
ph

OCH3
N
Se
O

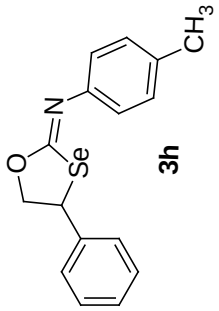
3g


48.668
55.494
76.666
76.910
77.230
77.550
114.570
121.937
127.787
128.481
129.190
137.945
143.619
156.890
162.480
ppm



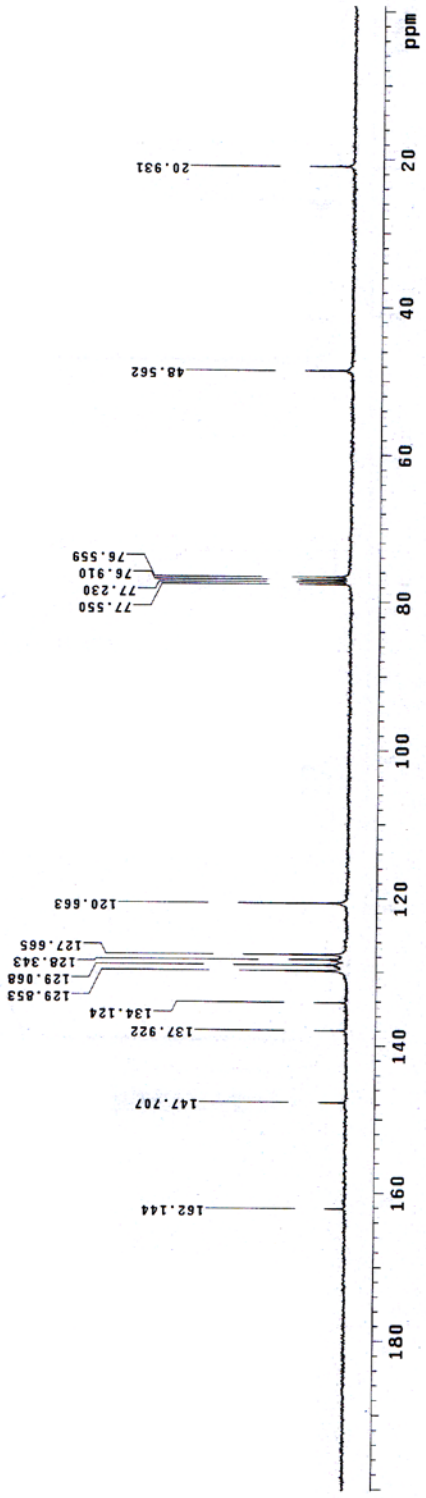
MSN-185

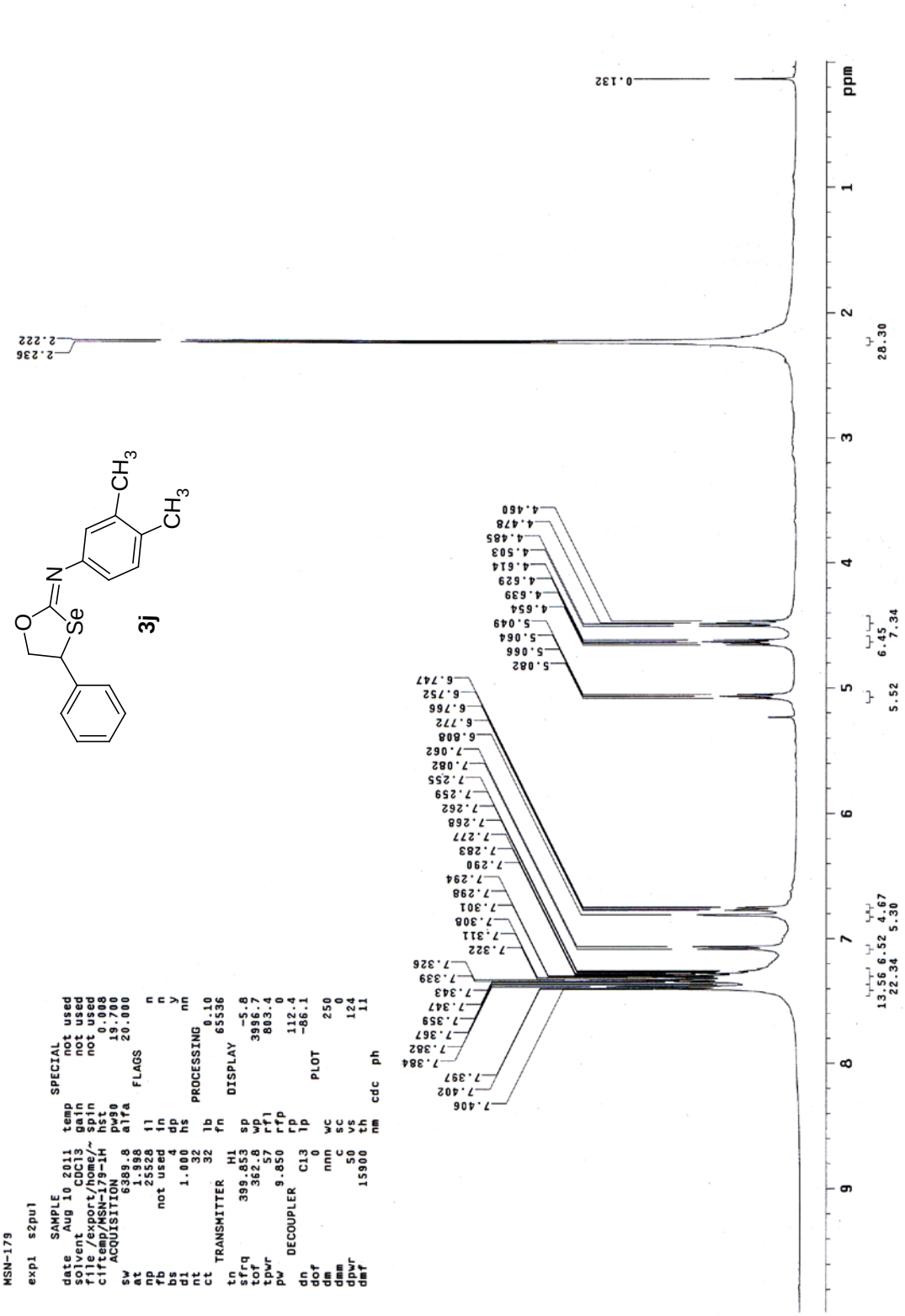
```
exp1  s2pul

   SAMPLE                  SPECIAL
date   Aug 15 2011  temp      not used
solvent     CDCl3   gain      not used
file /export/home/~ spin      not used
ciftemp/MSN-185-1H  hst         0.008
   ACQUISITION      pw90       19.700
sw          6389.8  alfa       20.000
at           1.998          FLAGS
np           25528  ll             n
fb       not used  in             n
bs              4  dp             y
d1          1.000  hs            nn
nt             32          PROCESSING
ct             32  lb          0.10
   TRANSMITTER     fn         65536
tn              H1          DISPLAY
sfrq       399.853  sp         -25.5
tof          362.8  wp        4013.4
tpwr            57  rfl       3701.2
pw           9.850  rfp       2894.9
   DECOUPLER       rp         115.9
dn             C13  lp        -102.4
dof              0          PLOT
dm             nnn  wc           250
dmm              c  sc             0
dpwr            50  vs          107
dmf          15900  th            9
                    nm   cdc  ph
```

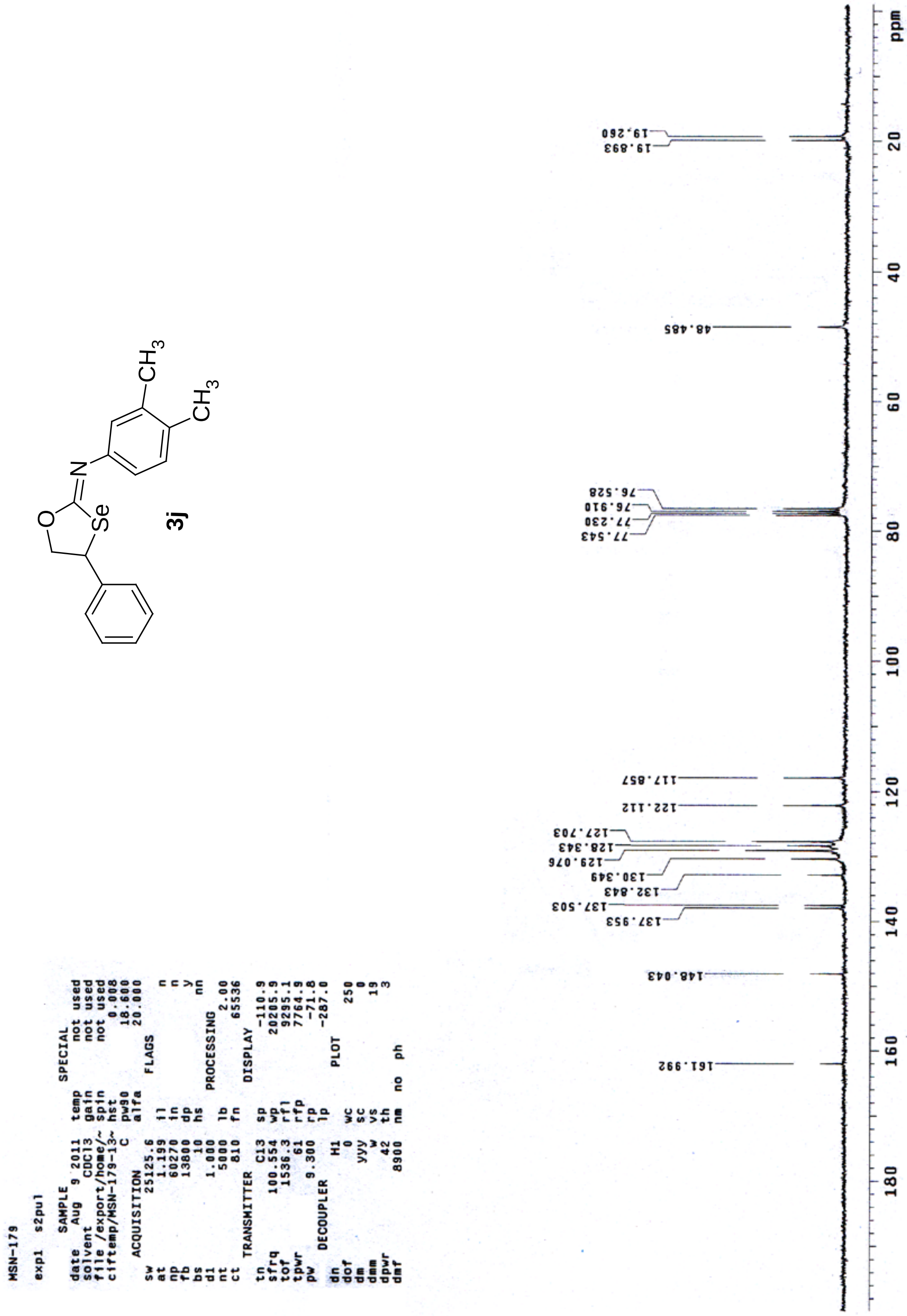


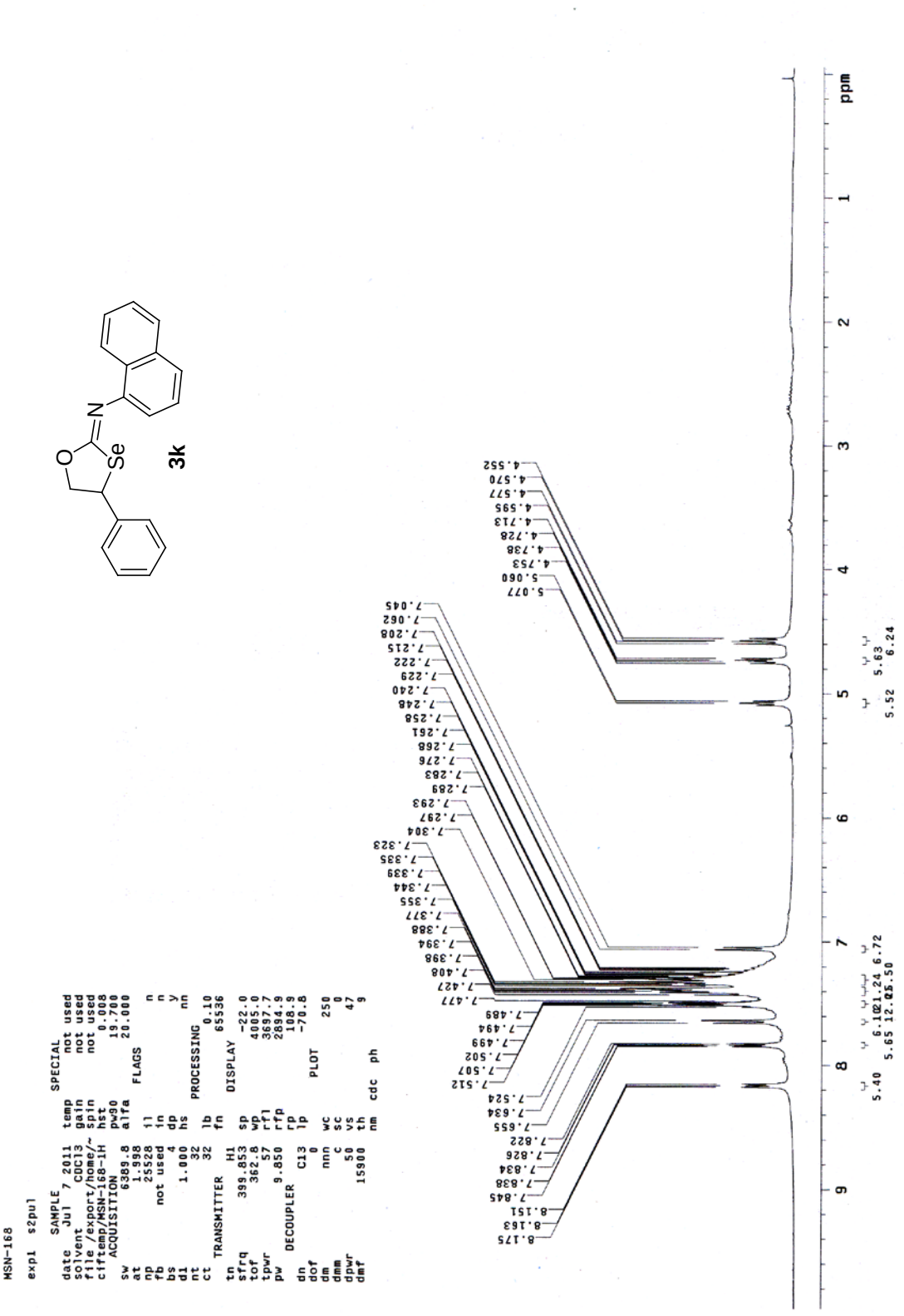
3h

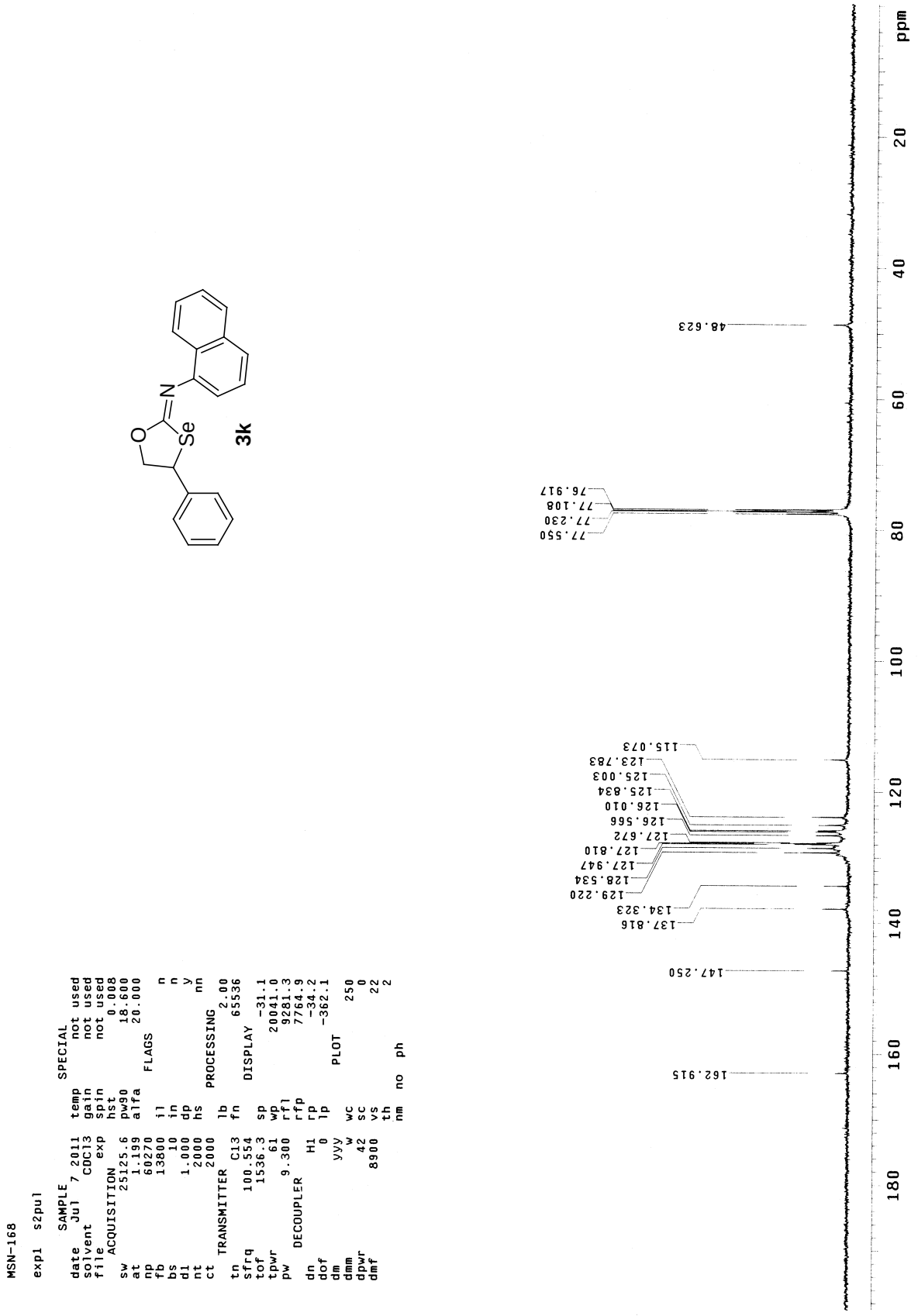
```
MSN-185

expl  s2pul

                                SPECIAL
   SAMPLE                         not used
date  Aug 15 2011  temp          not used
solvent     CDCl3  gain          not used
file /export/home/~  spin        0.008
ciftemp/MSN-185-13~  hst        18.600
         C  pw90  alfa  FLAGS   20.000
  ACQUISITION
sw      25125.6  fl                  n
at        1.199  in                  n
np        60270  dp                  y
fb        13800  hs                  nn
bs           10
d1        1.000  PROCESSING
nt        2000  lb      2.00
ct        1000  fn     65536
  TRANSMITTER  DISPLAY
tn          C13  sp     -78.7
sfrq     100.554  wp    20205.9
tof       1536.3  rfl    9295.9
tpwr          61  rfp    7764.9
pw         9.300  rp      -53.8
  DECOUPLER  lp      -324.4
dn          H1  PLOT
dof          0  wc       250
dm         yyy  sc         0
dmm          y  vs        19
dpwr        42  th         3
dmf       8900  nm   no  ph
```

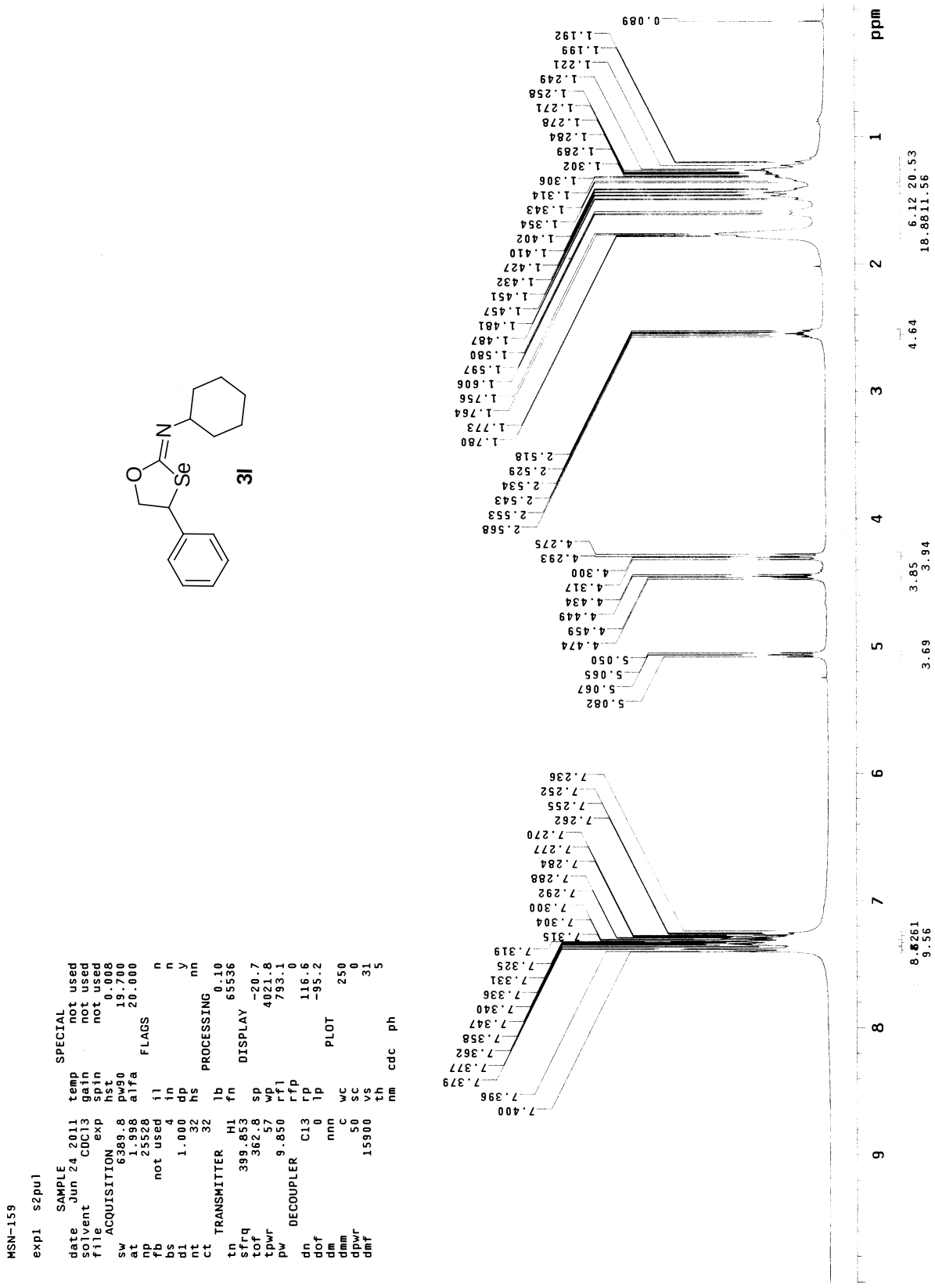


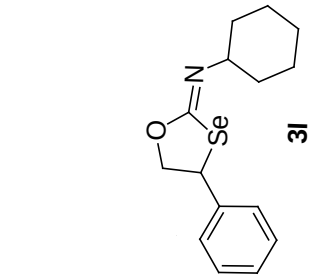

3j
CH3
CH3
N
O
Se
MSN-179
exp1 s2pul
SAMPLE
date Aug 10 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-179-1H
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL not used
temp not used
gain not used
spin 0.008
hst 19.700
pw90 20.000
alfa
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -5.8
wp 3996.7
rfl 803.4
rfp 0
rp 112.4
lp -86.1
PLOT
wc 250
sc 0
vs 124
th 11
cdc ph
nm
0.132
2.222
2.236
4.460
4.478
4.485
4.503
4.614
4.629
4.639
4.654
5.049
5.064
5.066
5.082
6.747
6.752
6.766
6.772
6.808
7.062
7.082
7.255
7.259
7.262
7.268
7.277
7.283
7.290
7.294
7.298
7.301
7.308
7.311
7.322
7.326
7.339
7.343
7.347
7.359
7.367
7.382
7.384
7.397
7.402
7.406
ppm
28.30
6.45
7.34
5.52
13.56
6.52
4.67
22.34
5.30


3j
MSN-179
ppm
19.260
19.893
48.485
76.528
76.910
77.230
77.543
117.857
122.112
127.703
128.343
129.076
130.349
132.843
137.503
137.953
148.043
161.992


3k
MSN-168
expl s2pul
SAMPLE
date Jul 7 2011
solvent CDCl3
file /export/home/~
cl#temp/MSN-168-1H
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL not used
temp not used
gain not used
spin 0.008
hst 19.700
pw90 20.000
alfa
FLAGS
il n
in n
dp y
hs nn
PROCESSING 0.10
lb 65536
fn
DISPLAY
sp -22.0
wp 4065.0
rfl 3697.7
rfp 2884.9
rp 108.9
lp -70.8
PLOT
wc 250
sc 0
vs 47
th 9
nm cdc ph
ppm

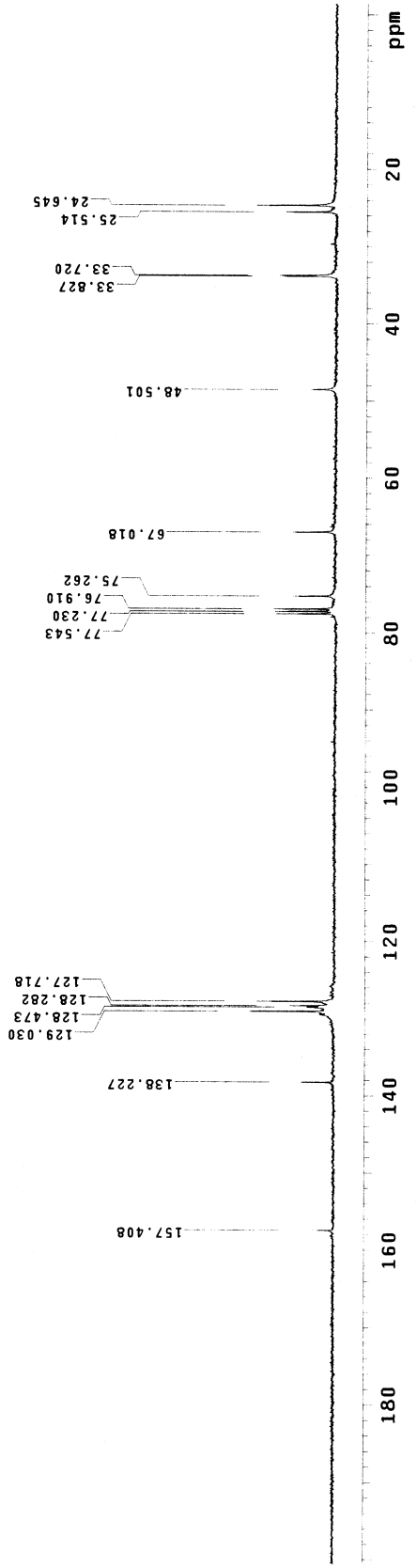

MSN-168
expl s2pul
SAMPLE
date Jul 7 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -31.1
wp 20041.0
rf1 9281.3
rfp 7764.9
rp -34.2
lp -362.1
PLOT
wc 250
sc 0
vs 22
th 2
nm no ph
3k
48.623
76.917
77.108
77.230
77.550
115.073
123.783
125.003
125.834
126.010
126.566
127.672
127.810
127.947
128.534
129.220
134.323
137.816
147.250
162.915
ppm

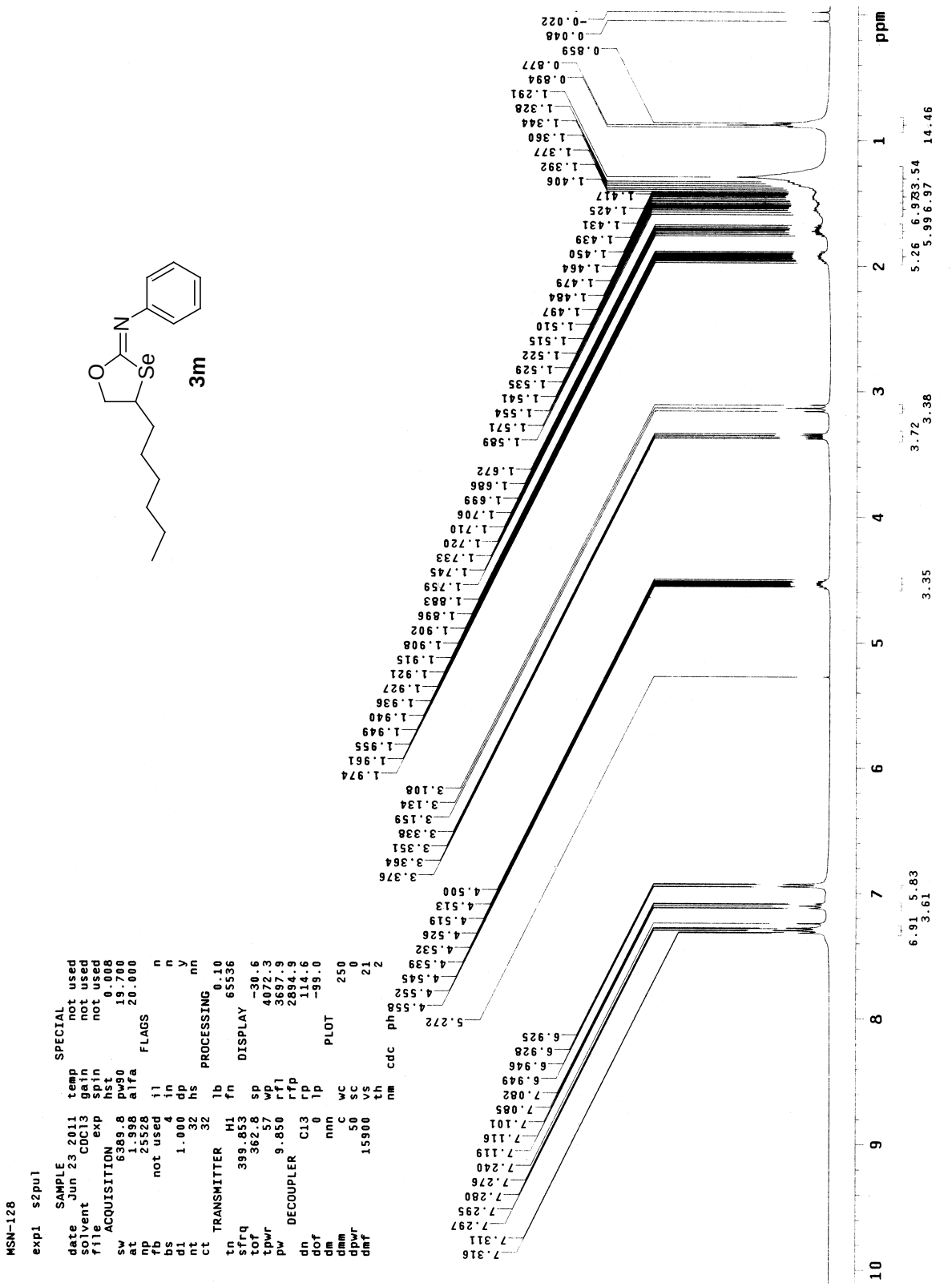

MSN-159
exp1 s2pul
SAMPLE
date Jun 24 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -20.7
wp 4021.8
rfl 793.1
rfp 0
rp 116.6
lp -95.2
PLOT
wc 250
sc 0
vs 31
th 5
cdc ph
nm
3l
ppm
0.089
1.192
1.199
1.221
1.249
1.258
1.271
1.278
1.284
1.289
1.302
1.306
1.314
1.343
1.354
1.402
1.410
1.427
1.432
1.451
1.457
1.481
1.487
1.580
1.597
1.606
1.756
1.764
1.773
1.780
2.518
2.529
2.534
2.543
2.553
2.568
4.275
4.293
4.300
4.317
4.434
4.449
4.459
4.474
5.050
5.065
5.067
5.082
7.236
7.252
7.255
7.262
7.270
7.277
7.284
7.288
7.292
7.300
7.304
7.315
7.319
7.325
7.331
7.336
7.340
7.347
7.358
7.362
7.377
7.379
7.396
7.400
6.12 20.53
18.8811.56
4.64
3.85 3.94
3.69
8.5261
9.56

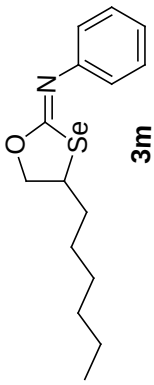


3l

```
MSN-158

expl  s2pul

                    SAMPLE                  SPECIAL
   date   Jun 24 2011   temp      not used
   solvent      CDCl3   gain      not used
   file           exp   spin      not used
      ACQUISITION       hst          0.008
   sw         25125.6   pw90        18.600
   at           1.199   alfa        20.000
   np           60270                FLAGS
   fb           13800   il                n
   bs              10   in                n
   d1           1.000   dp                y
   nt            5000   hs               nn
   ct            1110                PROCESSING
      TRANSMITTER       lb            2.00
   tn             C13   fn           65536
   sfrq        100.554                DISPLAY
   tof          1536.3   sp         -141.5
   tpwr             61   wp        20304.8
   pw            9.300   rfl        9292.8
      DECOUPLER         rfp        7764.9
   dn               H1   rp          -78.0
   dof               0   lp          -271.4
   dm              yyy                PLOT
   dmm               w   wc             250
   dpwr             42   sc               0
   dmf            8900   vs              13
                         th               3
                         nm   no  ph
```

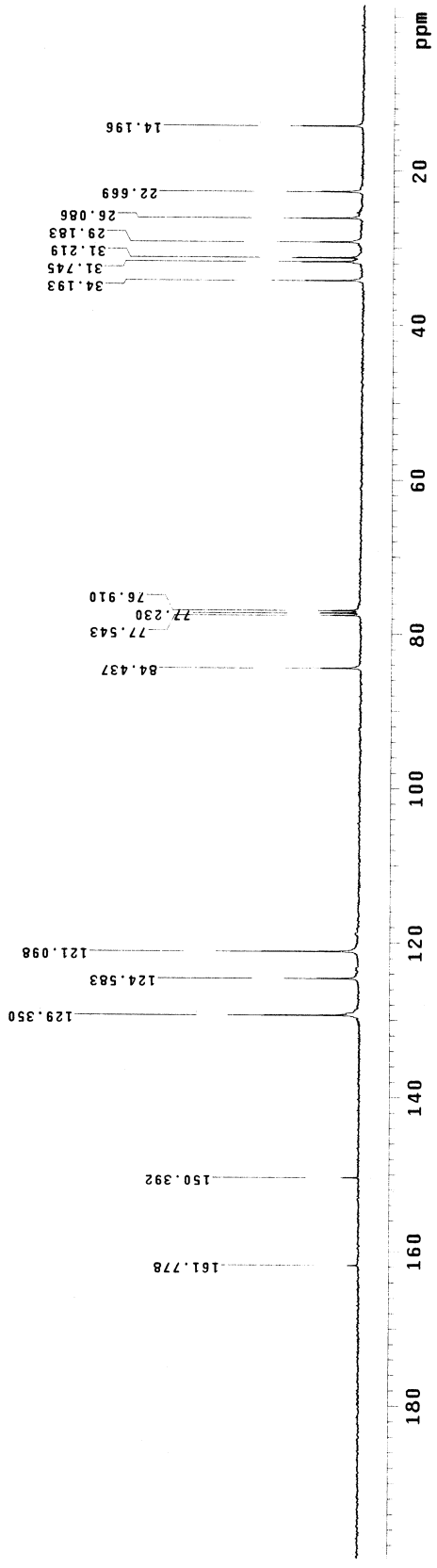


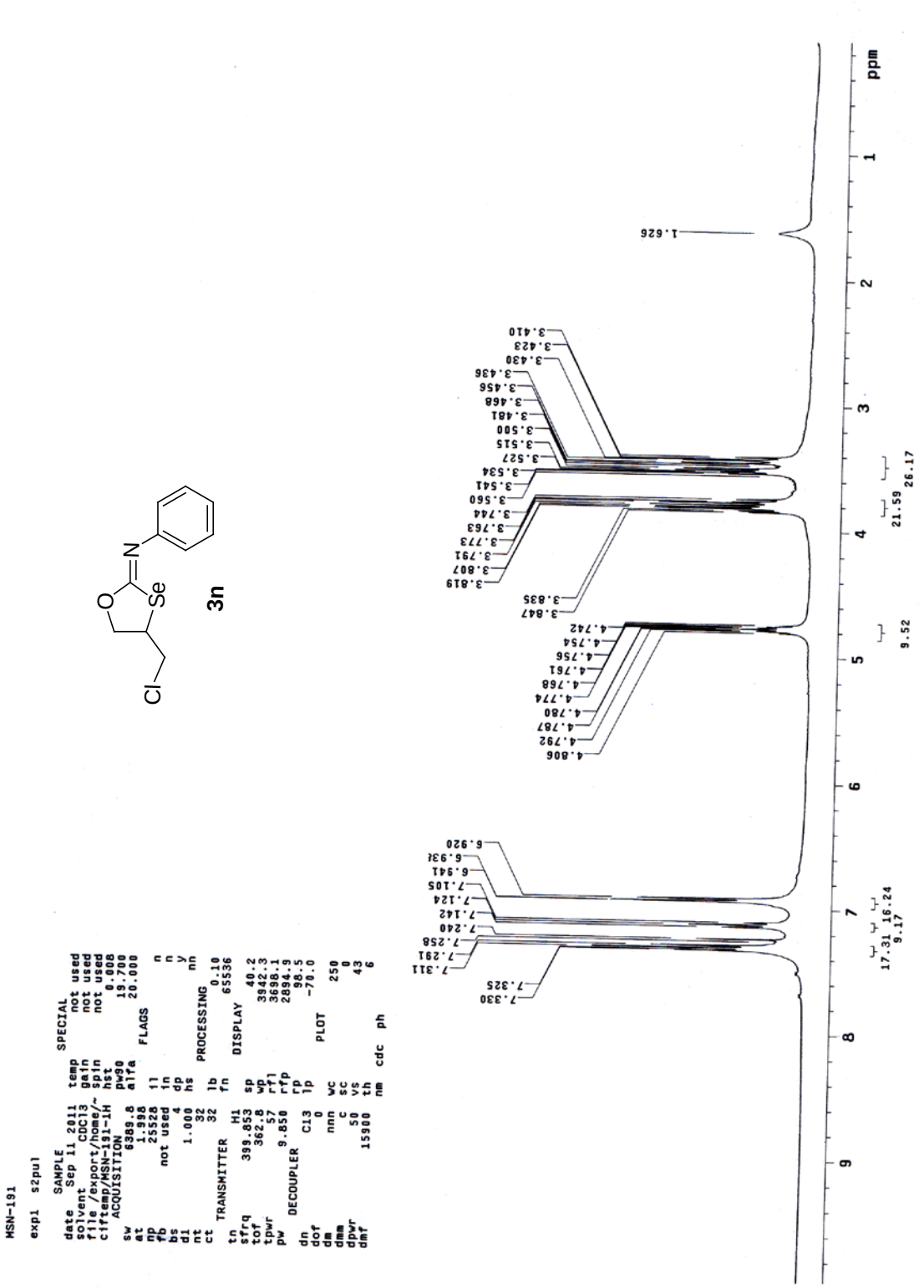

MSN-128
exp1 s2pul
SAMPLE
date Jun 23 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -30.6
wp 4072.3
rfl 3697.9
rfp 2894.9
rp 114.6
lp -99.0
PLOT
wc 250
sc 0
vs 21
th 2
nm
cdc
ph
3m
ppm
5.26 6.983.54
5.996.97
14.46
3.72
3.38
3.35
6.91 5.83
3.61

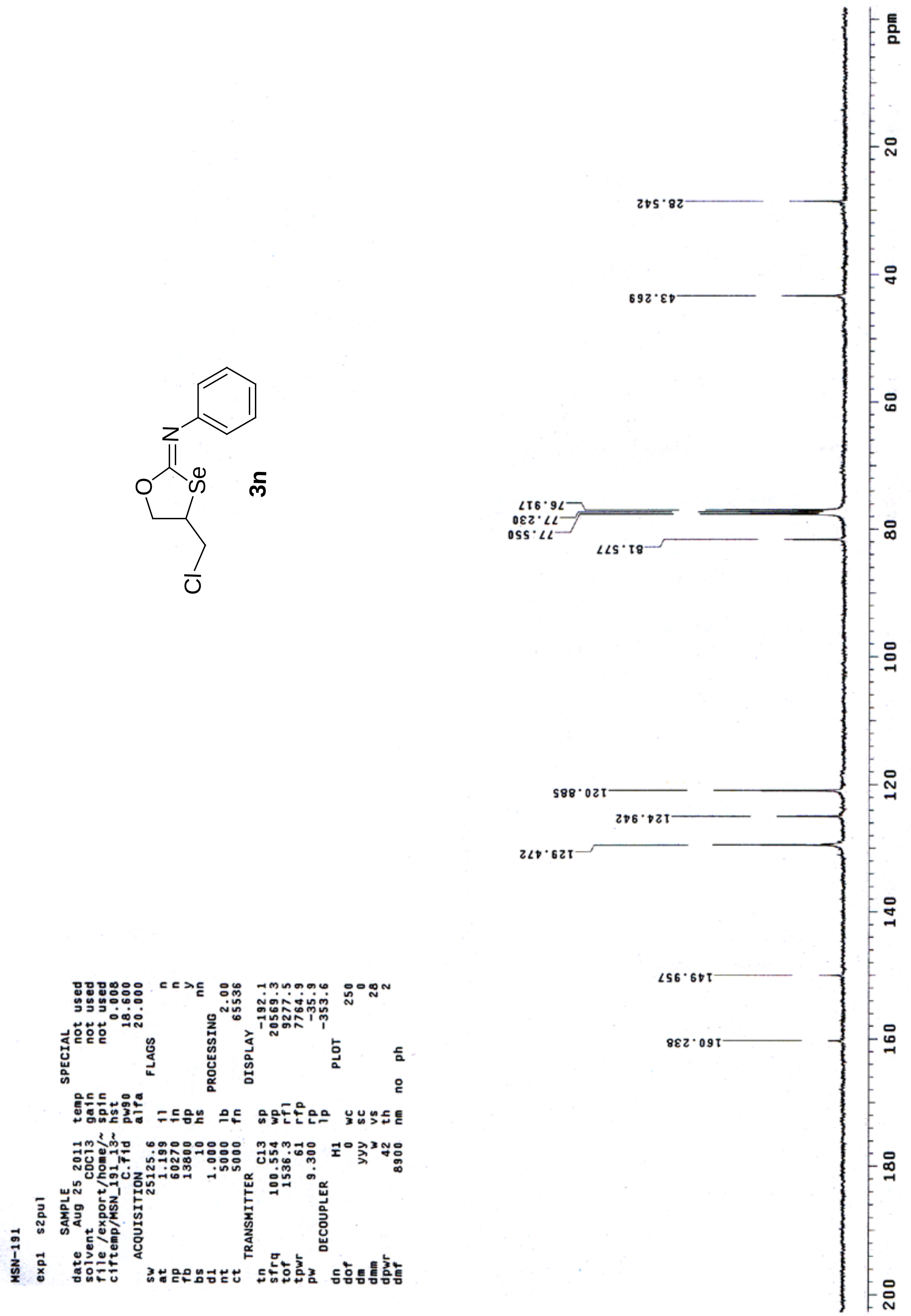


3m

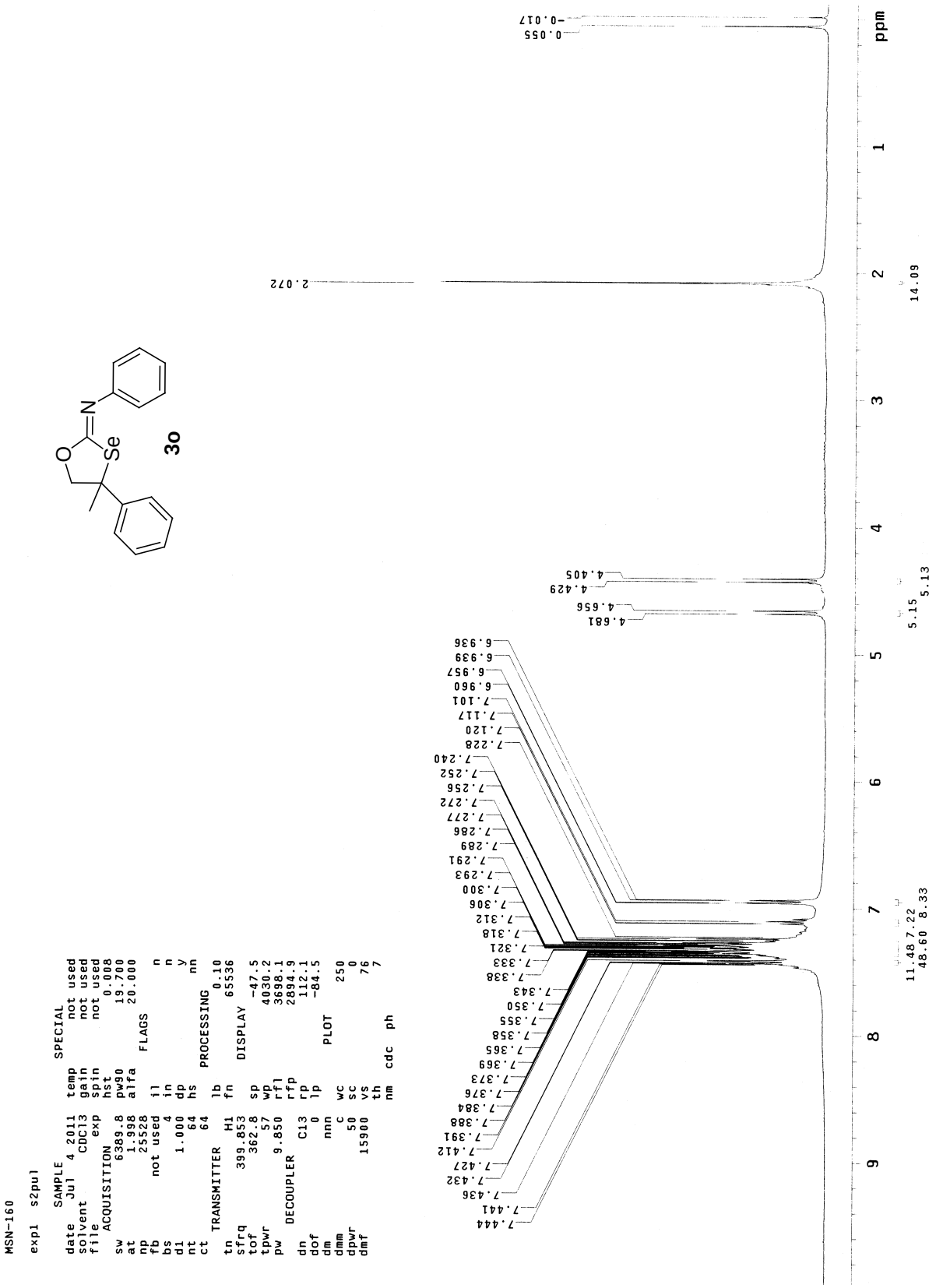
```
MSN-128-1

exp1  s2pul

                SAMPLE                  SPECIAL
date    May  2 2011   temp      not used
solvent      CDCl3    gain      not used
file /export/home/~   spin      not used
ciftemp/MSN_128-1~~   hst          0.008
          12C.fid     pw90        18.600
    ACQUISITION       alfa        20.000
sw        25125.6          FLAGS
at            1.199   il              n
np            60270   in              n
fb            13800   dp              y
bs               10   hs             nn
d1            1.000          PROCESSING
nt             5000   lb            2.00
ct             2070   fn           65536
   TRANSMITTER              DISPLAY
tn               C13   sp         -159.9
sfrq          100.554   wp        20238.8
tof            1536.3   rfl        9278.2
tpwr               61   rfp        7764.9
pw              9.300   rp          -58.3
    DECOUPLER          lp         -335.4
dn               H1                PLOT
dof               0   wc             250
dm              yyy   sc               0
dmm               w   vs              23
dpwr             42   th               2
dmf            8900   nm   no  ph
```

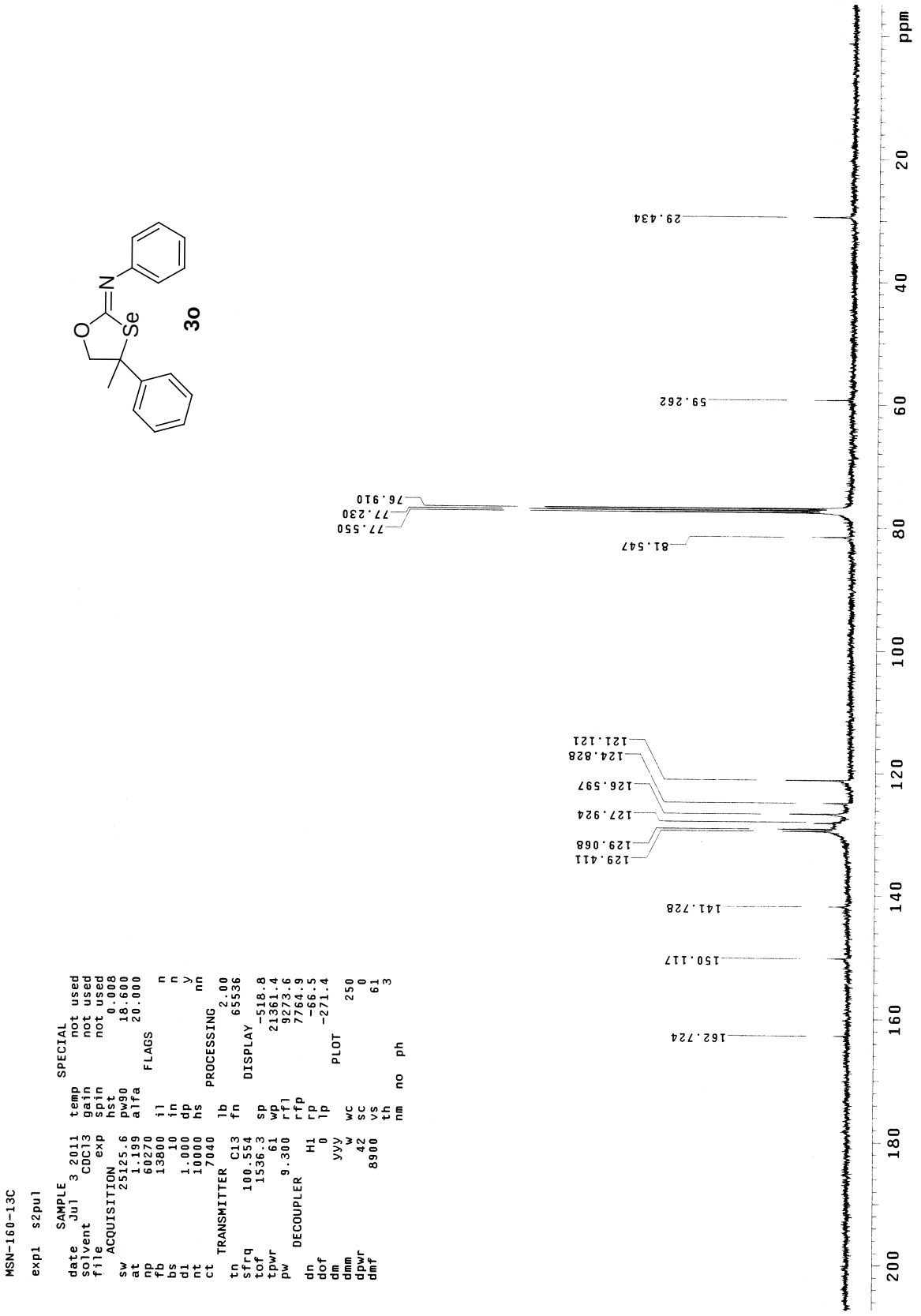


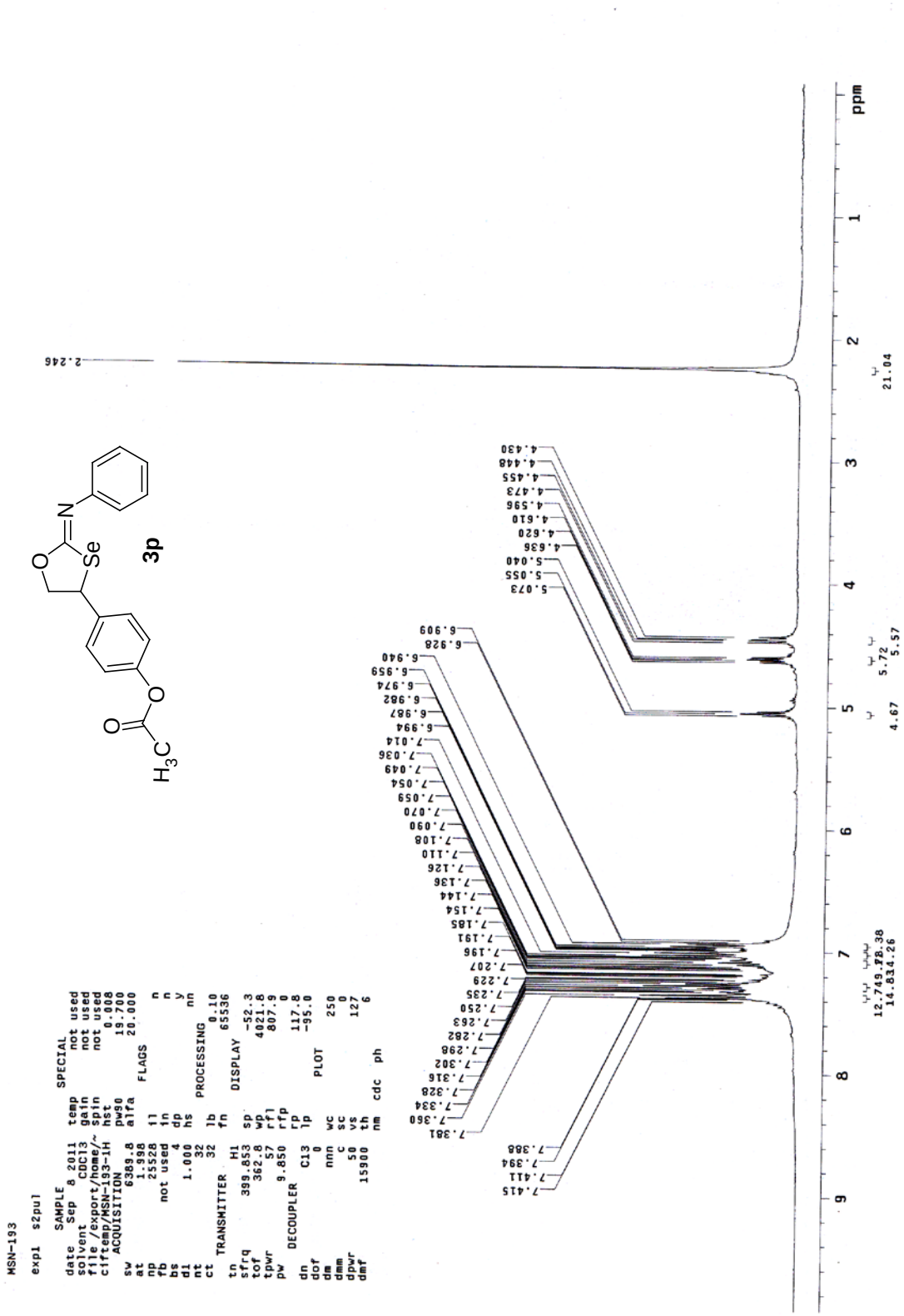

MSN-191
expl s2pul
SAMPLE
date Sep 11 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-191-1H
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hpt 0.008
pw90 19.700
alfa 20.000
FLAGS
n
y
nn
PROCESSING 0.10
lb 65536
fn
DISPLAY
sp 40.2
wp 3942.3
rfl 3698.1
rfp 2894.9
rp 98.5
lp -70.0
PLOT
wc 250
sc 0
vs 43
th 6
nm cdc ph
O
Se
N
Cl
3n
1.626
3.410
3.423
3.430
3.436
3.456
3.468
3.481
3.500
3.515
3.527
3.534
3.541
3.560
3.744
3.763
3.773
3.791
3.807
3.819
3.835
3.847
4.742
4.754
4.756
4.761
4.768
4.774
4.780
4.787
4.792
4.806
7.325
7.330
7.311
7.291
7.258
7.240
7.142
7.124
7.105
6.941
6.931
6.920
21.59
26.17
9.52
17.31
16.24
9.17
ppm

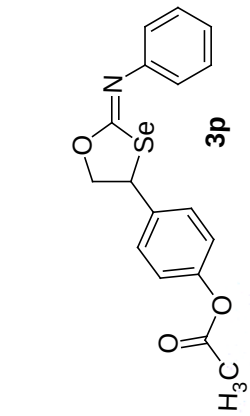

MSN-191
Se
O
N
Cl
28.542
43.269
76.917
77.230
77.550
81.577
120.885
124.942
129.472
149.957
160.238
ppm

3n

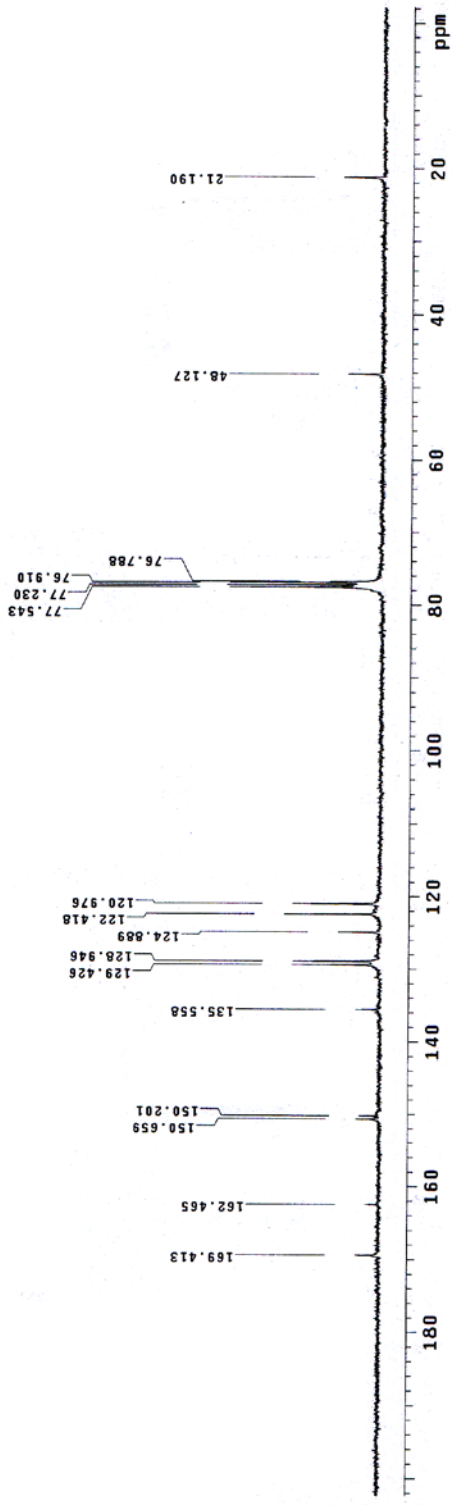

MSN-160
expl s2pul
SAMPLE
date Jul 4 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -47.5
wp 4030.2
rfl 3698.1
rfp 2894.9
rp 112.1
lp -84.5
PLOT
wc 250
sc 0
vs 76
th 7
nm cdc ph
3o
2.072
-0.017
0.055
4.405
4.429
4.656
4.681
5.15
5.13
14.09
11.48 7.22
48.60 8.33
ppm

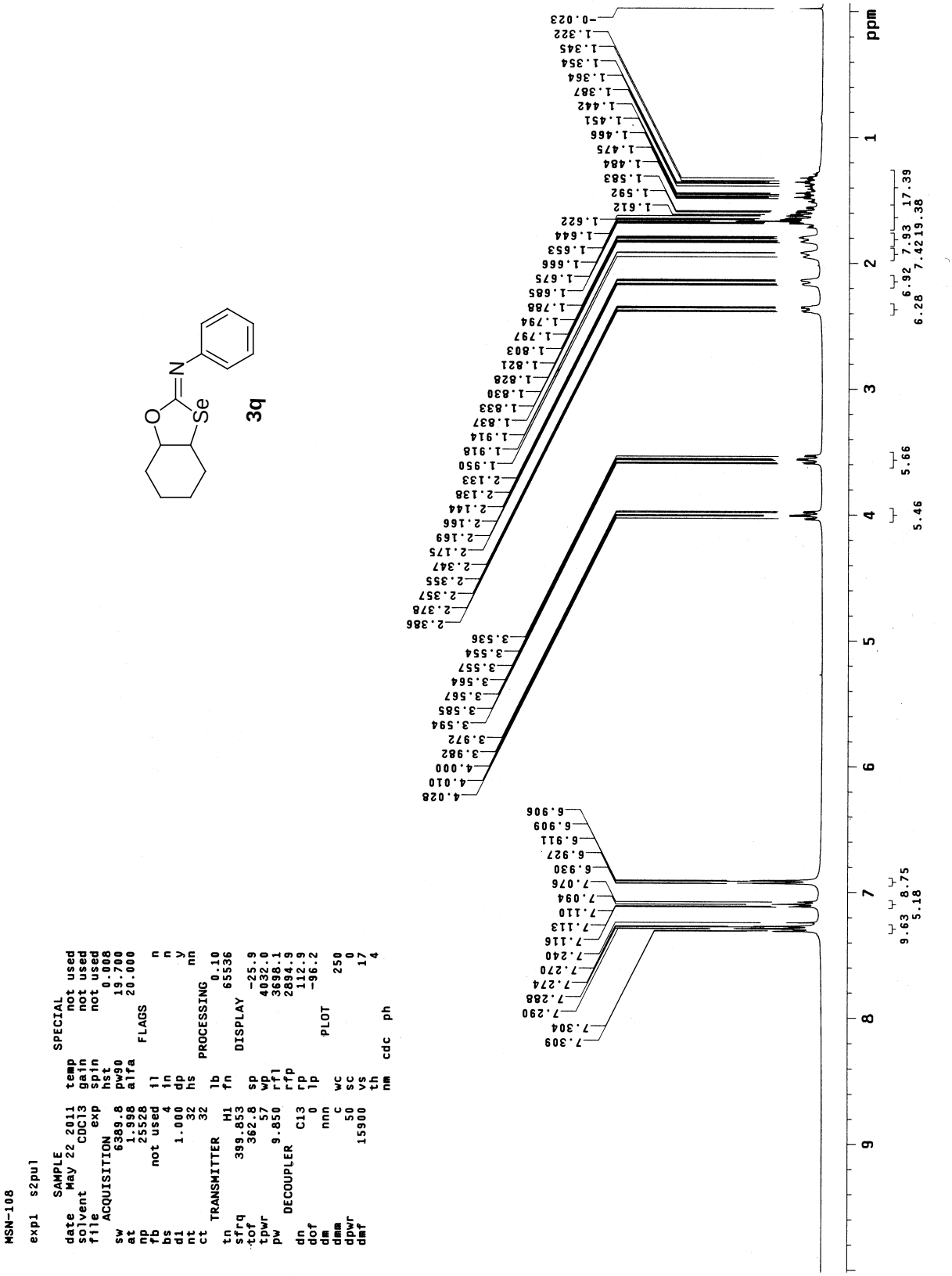

MSN-160-13C
exp1 s2pul
SAMPLE
date Jul 3 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 10000
ct 7040
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -518.8
wp 21361.4
rf1 9273.6
rfp 7764.9
rp -66.5
lp -271.4
PLOT
wc 250
sc 0
vs 61
th 3
nm
no ph
3o
Se
O
N
29.434
59.262
76.910
77.230
77.550
81.547
121.121
124.828
126.597
127.924
129.068
129.411
141.728
150.117
162.724
ppm
200
180
160
140
120
100
80
60
40
20

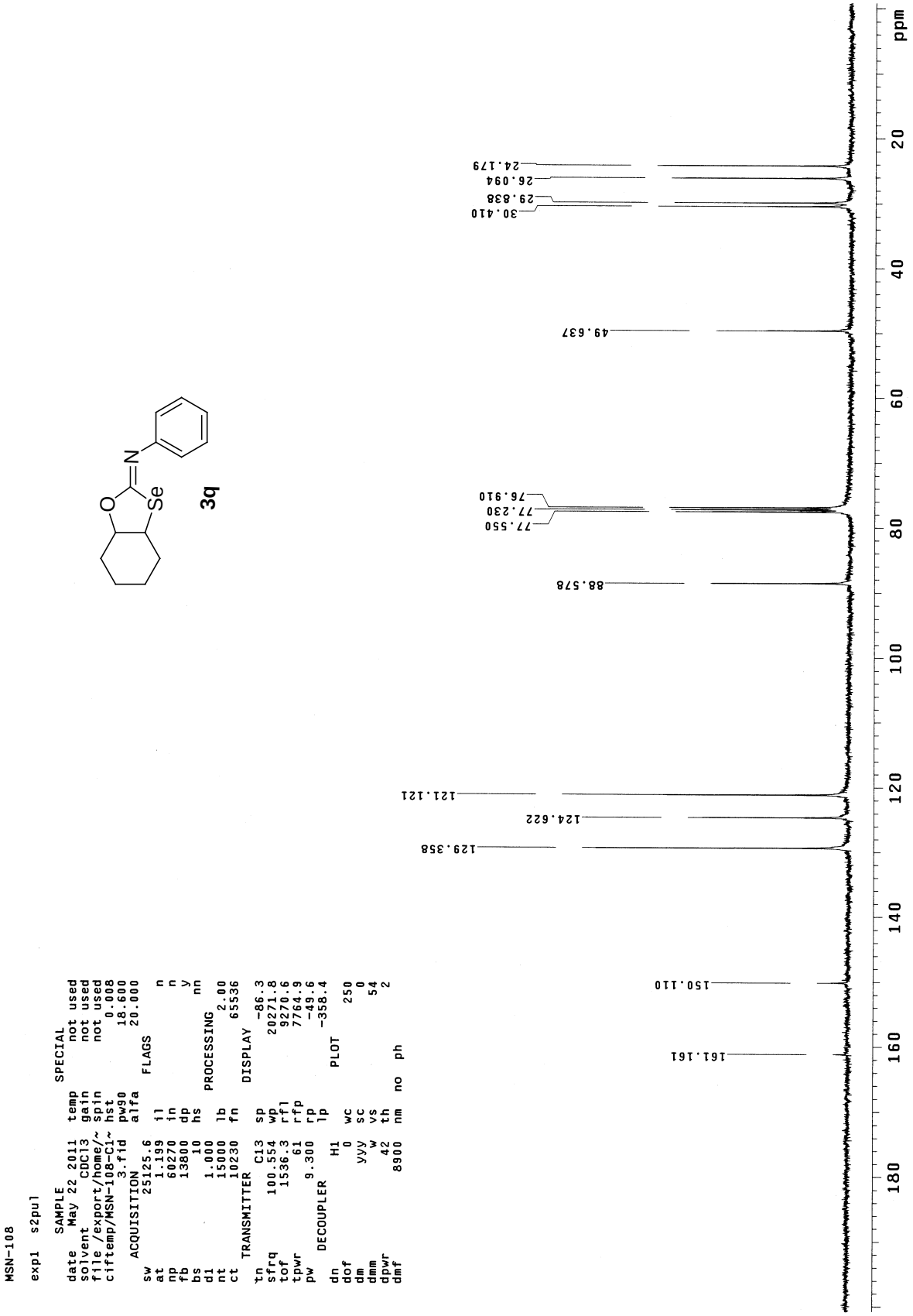

MSN-193
expl s2pul
SAMPLE
date Sep 8 2011
solvent CDCl3
file /export/home/~
cltemp/MSN-193-1H
ACQUISITION
sw 6389.8
at 1.398
np 25528
fb not used
bs 4
dl 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -52.3
wp 4021.8
rfl 807.9
rfp 0
rp 117.8
lp -95.0
PLOT
wc 250
sc 0
vs 127
th 6
nm cdc ph
3p
2.246
7.415
7.411
7.394
7.388
7.381
7.360
7.334
7.328
7.316
7.302
7.298
7.282
7.263
7.250
7.235
7.229
7.207
7.196
7.185
7.191
7.154
7.144
7.136
7.126
7.110
7.108
7.090
7.070
7.059
7.054
7.049
7.036
7.014
6.994
6.987
6.982
6.974
6.959
6.940
6.928
6.909
5.073
5.055
5.040
4.636
4.620
4.610
4.596
4.473
4.455
4.448
4.430
21.04
5.72
5.57
4.67
12.749
28.38
14.834
26.26
ppm

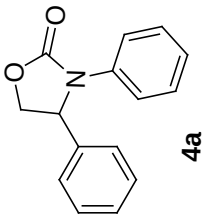



```
MSN-193

exp1  s2pul

    SAMPLE                  SPECIAL
  date  Aug 30 2011  temp      not used
solvent      CDCl3  gain      not used
file /export/home/~  spin      not used
ciftemp/MSN_193_13~  hst       0.000
         C.fid       pw90     18.800
   ACQUISITION       alfa     20.000
sw       25125.6     FLAGS
at          1.199    il           n
np        60270      in           n
fb        13800      dp           y
bs           10      hs          nn
d1            1.000  PROCESSING    2.00
nt         5000      lb
ct         5000      fn        65536
   TRANSMITTER  C13   DISPLAY
tn          C13     sp       -225.1
sfrq      100.554     wp      25569.3
tof        1536.3     rfl      9277.5
tpwr         61       rfp      7764.9
pw          9.300     rp       -336.7
   DECOUPLER          lp       -376.9
dn          H1      PLOT
dof          0      wc          250
dm          yyy     sc            0
dmm           w     vs           26
dpwr         42     th            2
dmf        8900     nm   no  ph
```

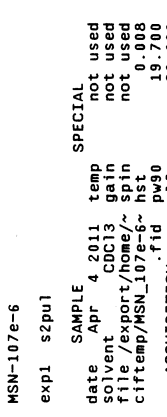


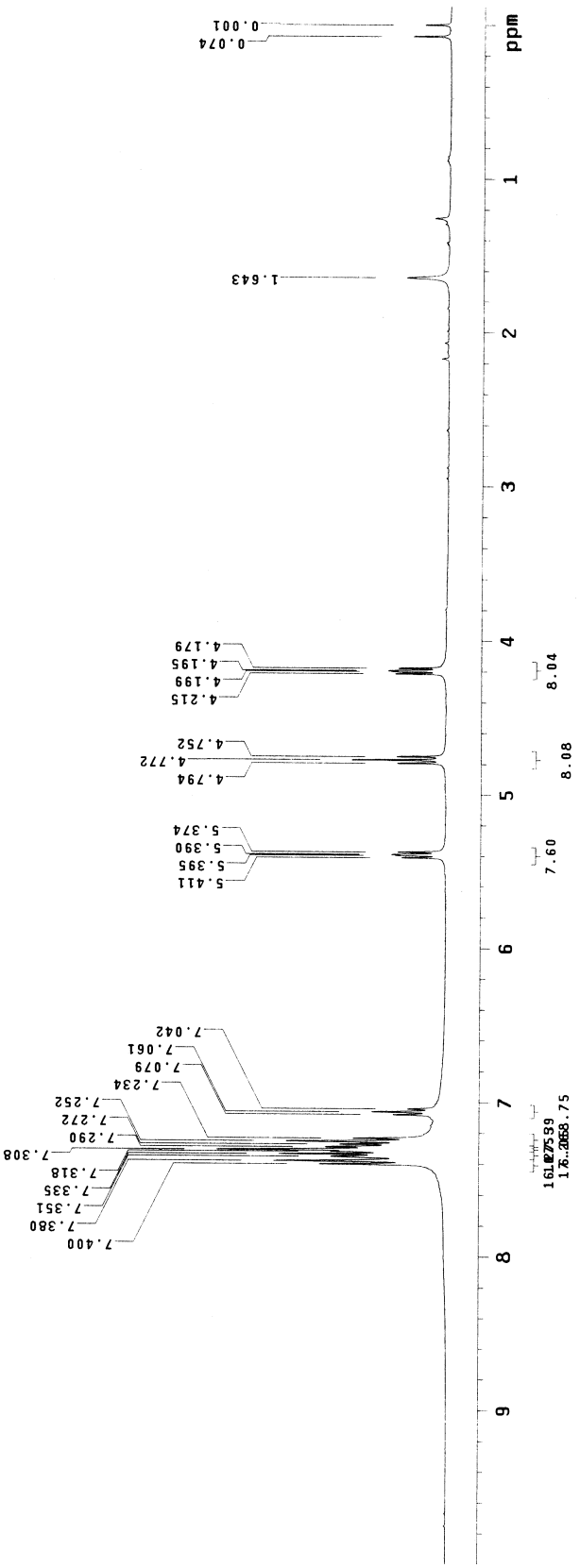

MSN-108
exp1 s2pul
SAMPLE
date May 22 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
di 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -25.9
wp 4032.0
rfl 3698.1
rfp 2894.9
rp 112.9
lp -96.2
PLOT
wc 250
sc 0
vs 17
th 4
nm cdc ph
3q
ppm
6.92 7.93 17.39
6.28 7.42 19.38
5.66
5.46
9.63 8.75
5.18

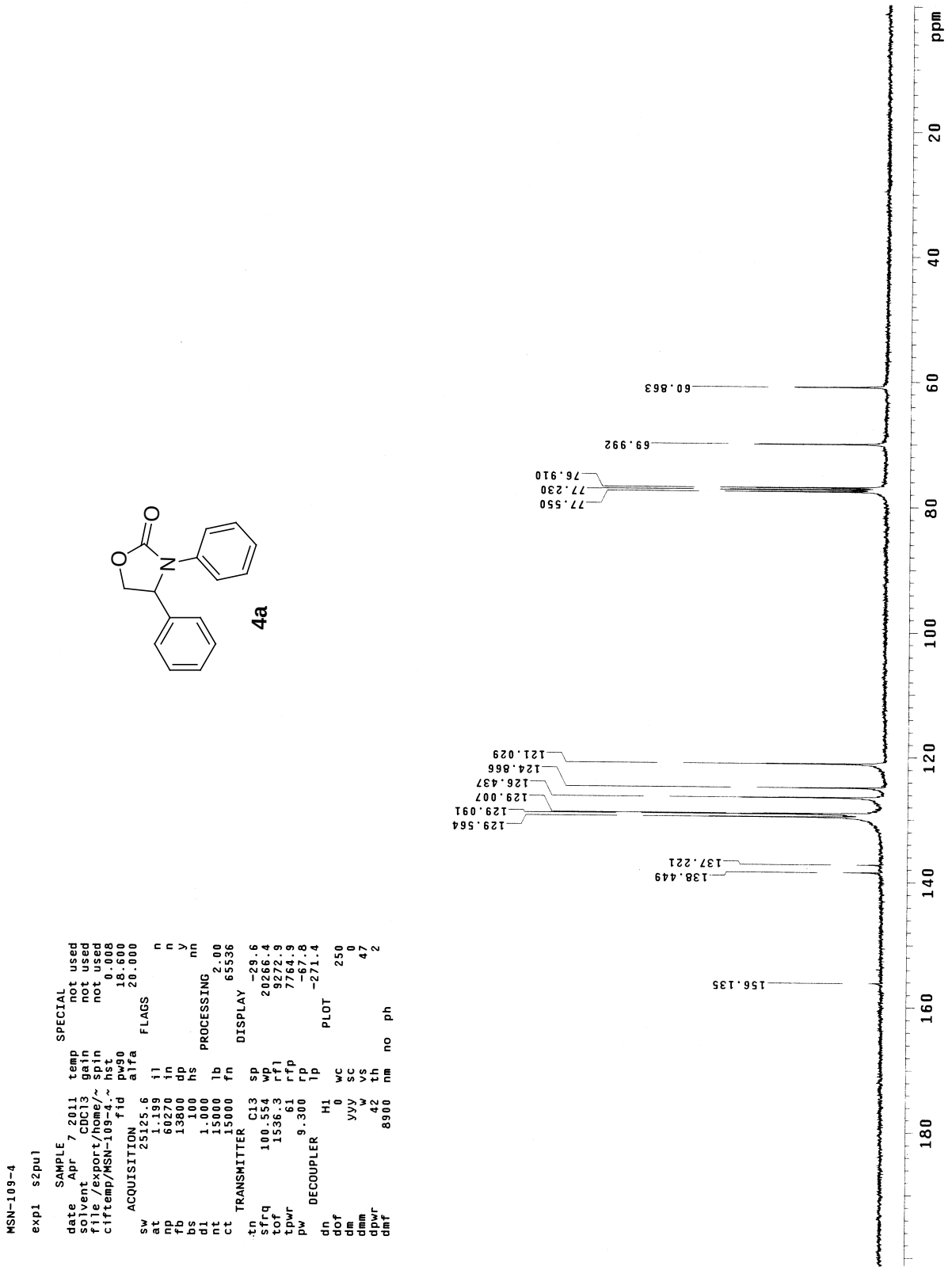

MSN-108
exp1 s2pul
SAMPLE
date May 22 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-108-C1~
3.fid
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 15000
ct 10230
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -86.3
wp 20271.8
rfl 9270.6
rfp 7764.9
rp -49.6
lp -358.4
PLOT
wc 250
sc 0
vs 54
th 2
nm no ph
O
N
Se
3q
24.179
26.094
29.838
30.410
49.637
76.910
77.230
77.550
88.578
121.121
124.622
129.358
150.110
161.161
ppm
180
160
140
120
100
80
60
40
20

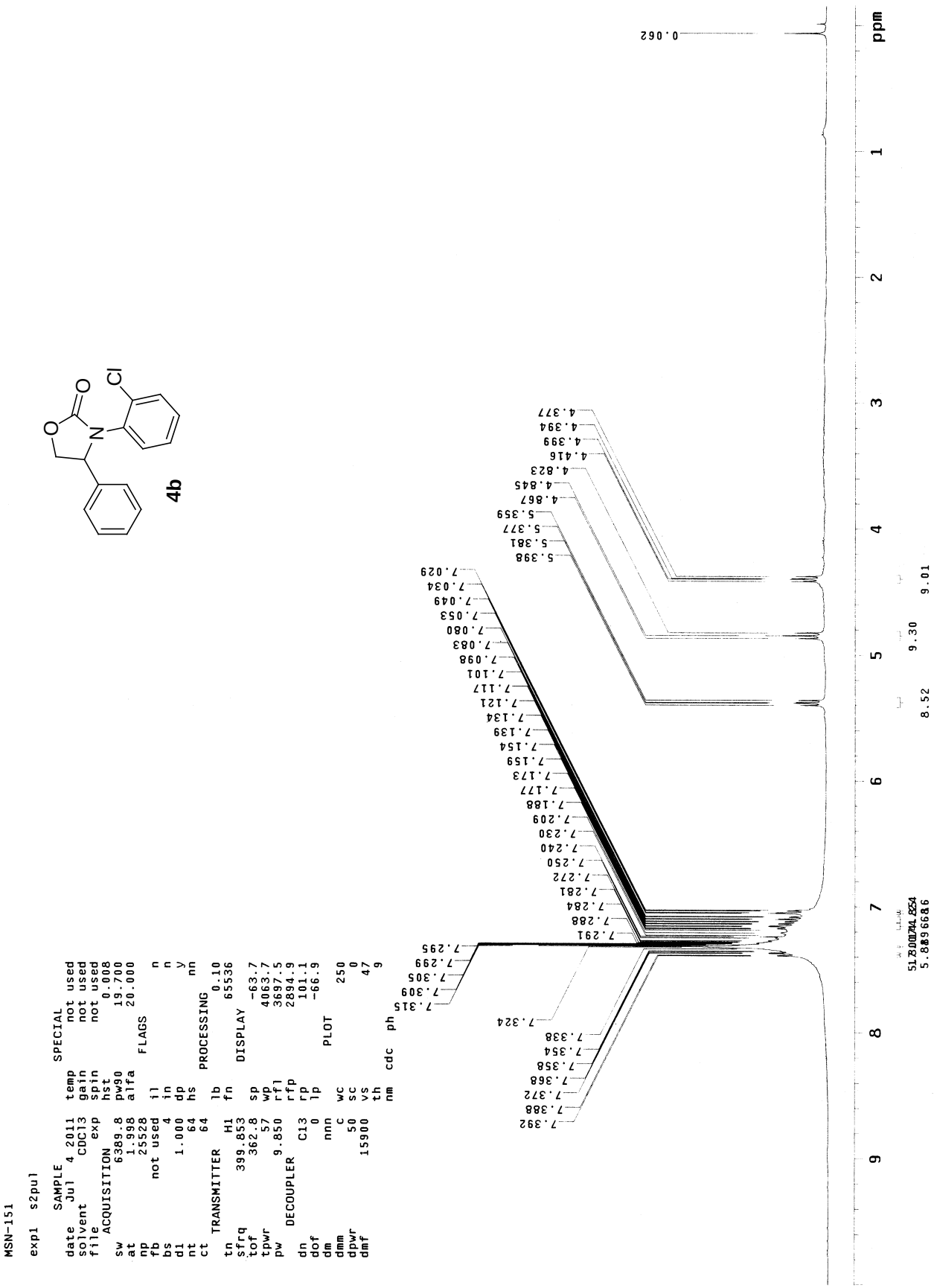


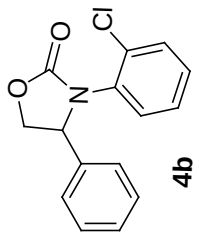
4a


MSN-107e-6
expl s2pul
SAMPLE
date Apr 4 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN_107e-6~
.fid
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
i1 n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -48.6
wp 4047.0
rfl 795.8
rfp 0
rp 117.4
lp -94.0
PLOT
wc 250
sc 0
vs 37
th 4
cdc ph
nm


0.001
0.074
1.643
4.179
4.195
4.199
4.215
4.752
4.772
4.794
5.374
5.390
5.395
5.411
7.042
7.061
7.079
7.234
7.252
7.272
7.290
7.308
7.318
7.335
7.351
7.380
7.400
ppm
1
2
3
4
5
6
7
8
9
7.60
8.04
8.08

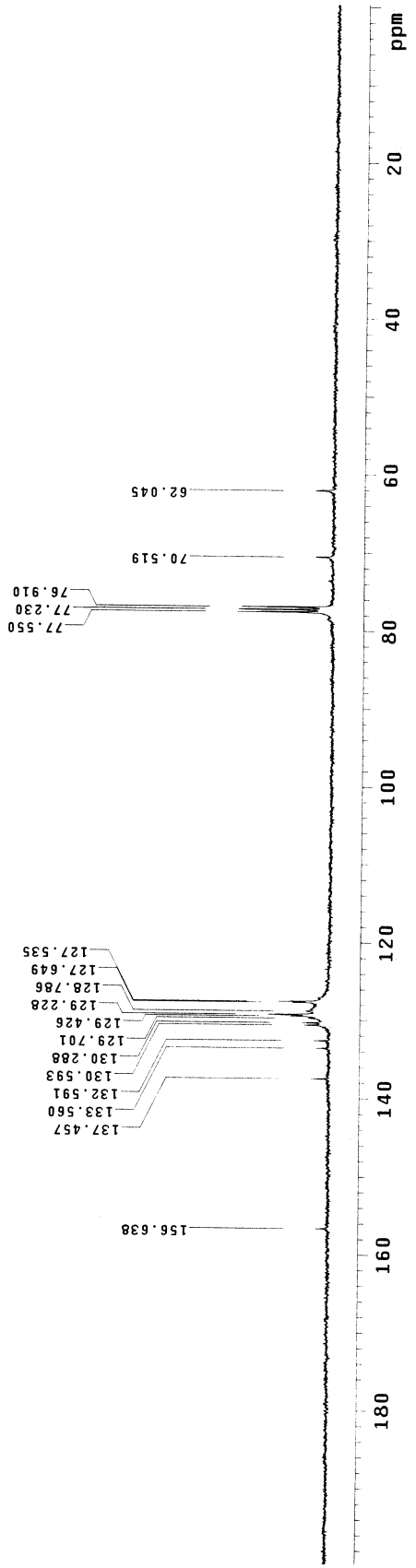

MSN-109-4
expl s2pul
SAMPLE
date Apr 7 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-109-4.~
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 100
d1 1.000
nt 15000
ct 15000
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
fid pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -29.6
wp 20266.4
rfl 9272.9
rfp 7764.9
rp -67.8
lp -271.4
PLOT
wc 250
sc 0
vs 47
th 2
nm no ph
4a
156.135
138.449
137.221
129.564
129.091
129.007
126.437
124.866
121.029
77.550
77.230
76.910
69.992
60.863
ppm
180
160
140
120
100
80
60
40
20

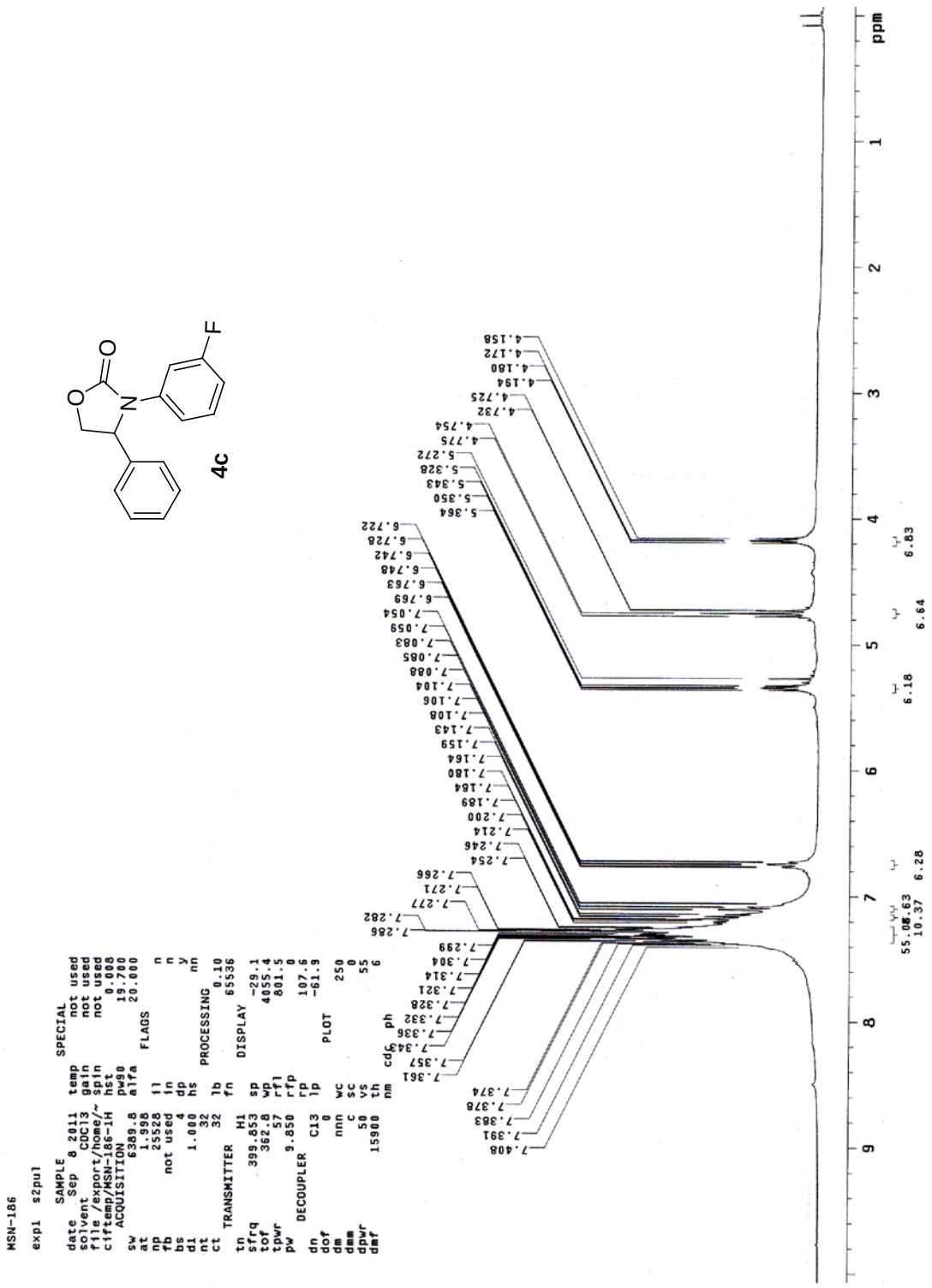

MSN-151
exp1 s2pul
SAMPLE
date Jul 4 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -63.7
wp 4063.7
rfl 3697.5
rfp 2884.9
rp 101.1
lp -66.9
PLOT
wc 250
sc 0
vs 47
th 9
nm cdc ph
4b
0.062
4.377
4.394
4.399
4.416
4.823
4.845
4.867
5.359
5.377
5.381
5.398
7.029
7.034
7.049
7.053
7.080
7.083
7.098
7.101
7.117
7.121
7.134
7.139
7.154
7.159
7.173
7.177
7.188
7.209
7.230
7.240
7.250
7.272
7.281
7.284
7.288
7.291
7.295
7.299
7.305
7.309
7.315
7.324
7.338
7.354
7.358
7.368
7.372
7.388
7.392
8.52
9.30
9.01
ppm

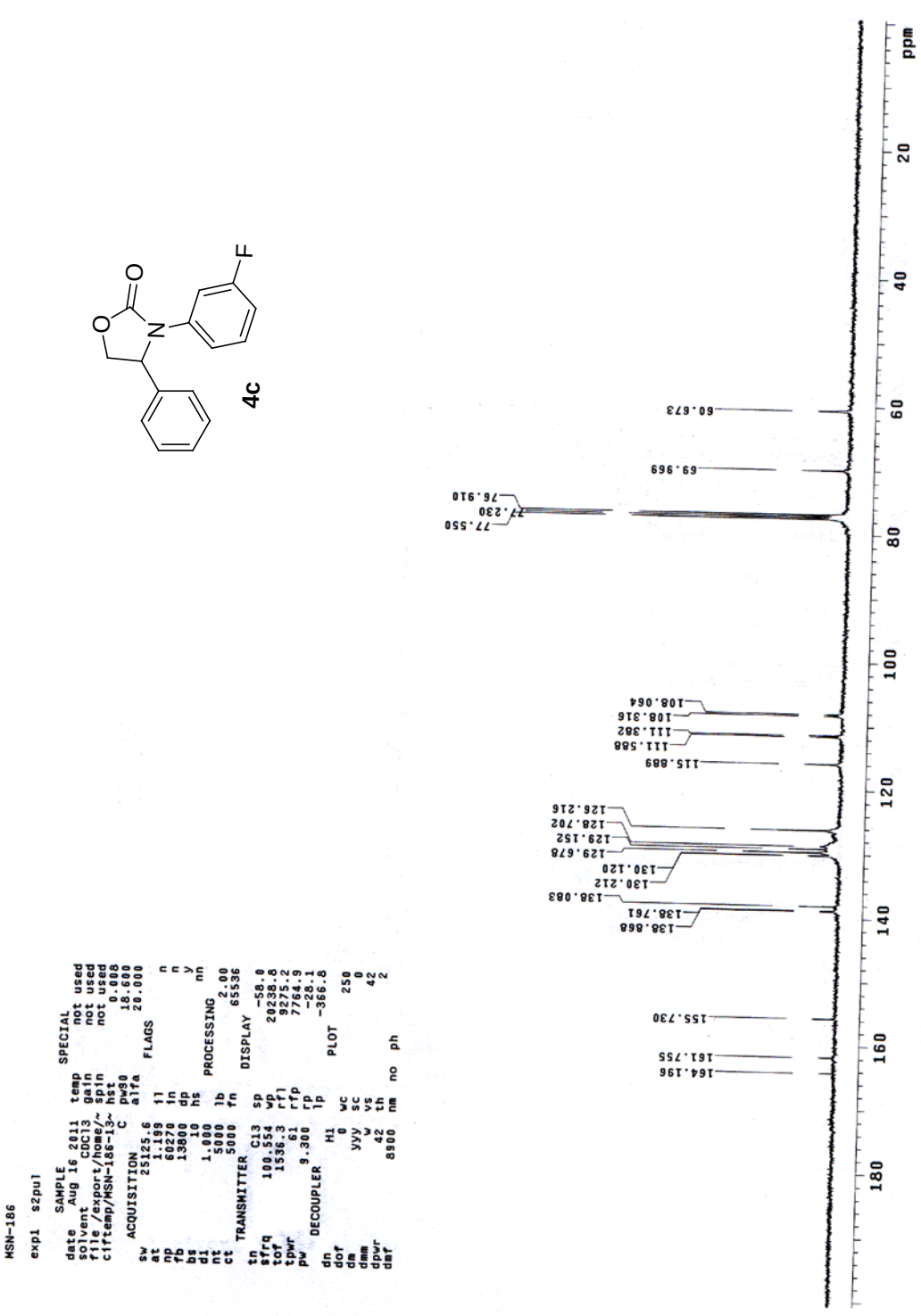


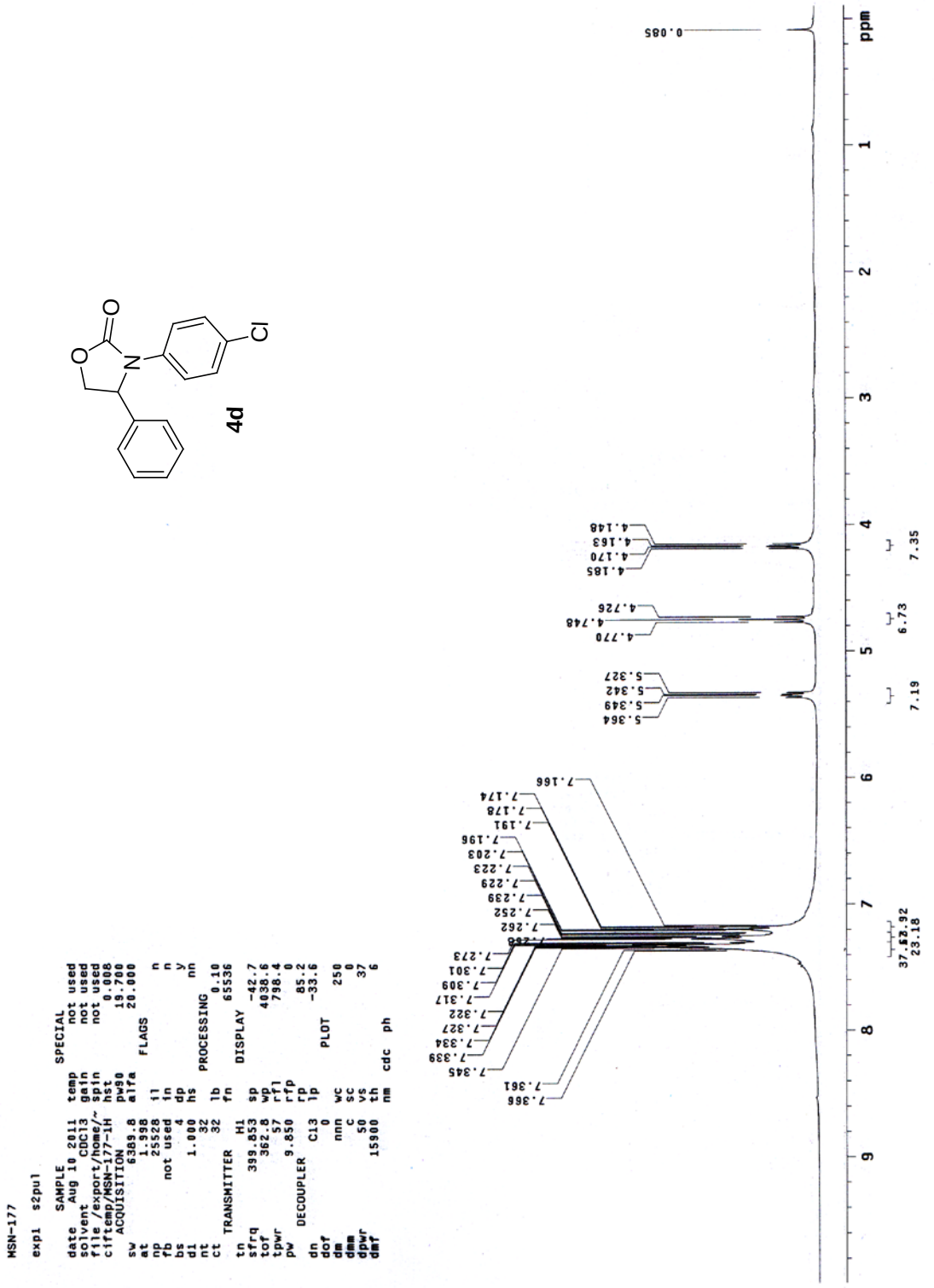
4b

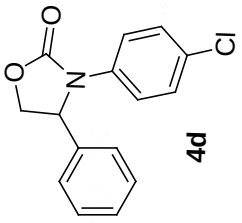
```
MSN-151

expl  s2pul

        SAMPLE                  SPECIAL
date    Jul  3 2011    temp        not used
solvent    CDCl3       gain        not used
file         exp       spin        not used
  ACQUISITION           hst           0.008
sw          25125.6     pw90         18.600
at            1.199     alfa         20.000
np            60270           FLAGS
fb            13800     il                n
bs               10     in                n
d1            1.000     dp                y
nt             7000     hs               nn
ct             3100   PROCESSING
  TRANSMITTER           lb            2.00
tn              C13     fn           65536
sfrq         100.554          DISPLAY
tof           1536.3     sp         -32.7
tpwr            61       wp         20106.9
pw            9.300      rfl         9282.8
  DECOUPLER             rfp         7764.9
dn              H1       rp          -21.2
dof              0       lp          -391.4
dm             yyy                   PLOT
dmm              w       wc             250
dpwr            42       sc               0
dmf           8900       vs              15
                         th               2
                         nm   no  ph
```



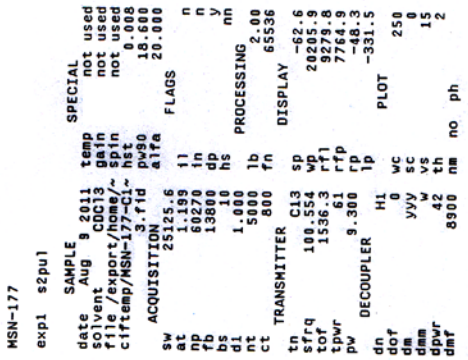

4c
F
O
O
N
ppm

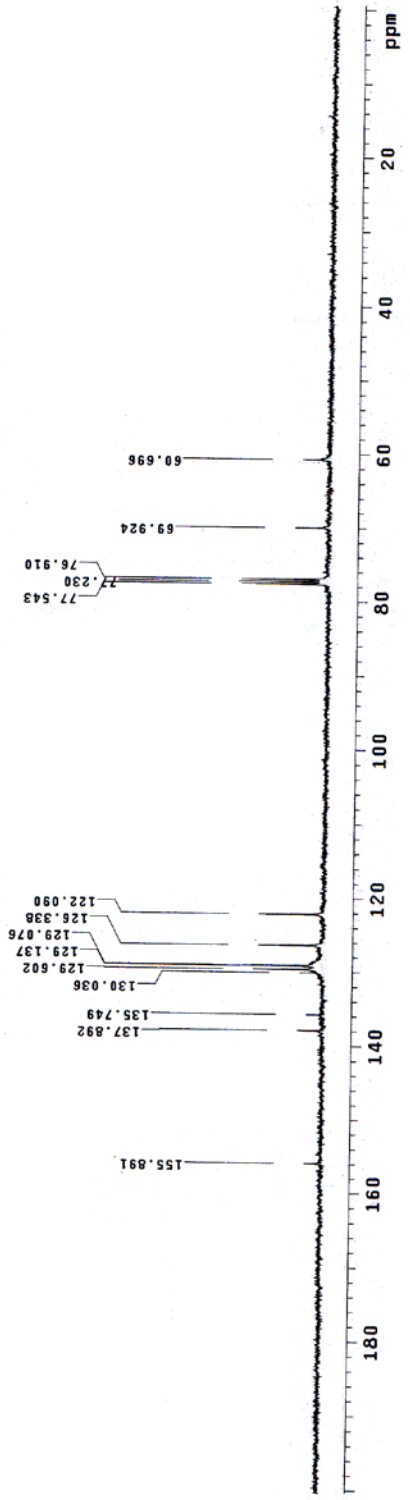

4c
MSN-186
expl s2pul
SAMPLE
date Aug 16 2011
solvent CDCl3
file /export/home/~
cifftemp/MSN-186-13~
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 5000
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -58.0
wp 20238.8
rfl 9275.2
rfp 7764.9
rp -28.1
lp -366.8
PLOT
wc 250
sc 0
vs 42
th 2
nm no ph
60.673
69.969
76.910
77.230
77.550
108.064
108.316
111.382
111.568
115.889
126.216
126.702
129.152
129.678
130.120
130.212
138.083
138.761
138.868
155.730
161.755
164.196
ppm

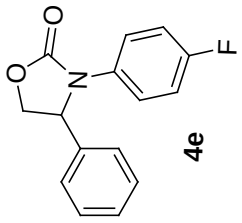

4d
Cl
O
O
N
MSN-177
expl s2pul
SAMPLE SPECIAL not used
date Aug 10 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/MSN-177-1H hst 0.008
ACQUISITION pw90 19.700
6389.8 alfa 20.000
sw 1.998 FLAGS n
at 25528 il n
np not used in y
fb 4 dp nn
bs 1.000 hs
d1 32 PROCESSING
nt 32 lb 0.10
ct TRANSMITTER fn 65536
H1 DISPLAY
tn 399.853 sp -42.7
sfrq 362.8 wp 4038.6
tof 57 rfl 798.4
tpwr 9.850 rfp 0
pw DECOUPLER rp 85.2
C13 lp -33.6
dn 0 PLOT
dof nnn wc 250
dm c sc 0
dmm 50 vs 37
dpwr 15900 th 6
dmf nm cdc ph
0.085
4.148
4.163
4.170
4.185
4.726
4.748
4.770
5.327
5.342
5.349
5.364
7.166
7.174
7.178
7.191
7.196
7.203
7.223
7.229
7.239
7.252
7.262
7.273
7.301
7.309
7.317
7.322
7.327
7.334
7.339
7.345
7.361
7.366
ppm
7.35
6.73
7.19
37.92
23.18



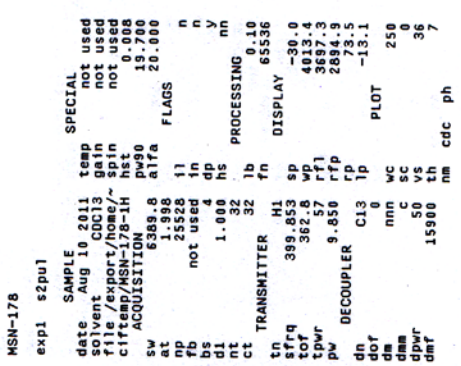
4d

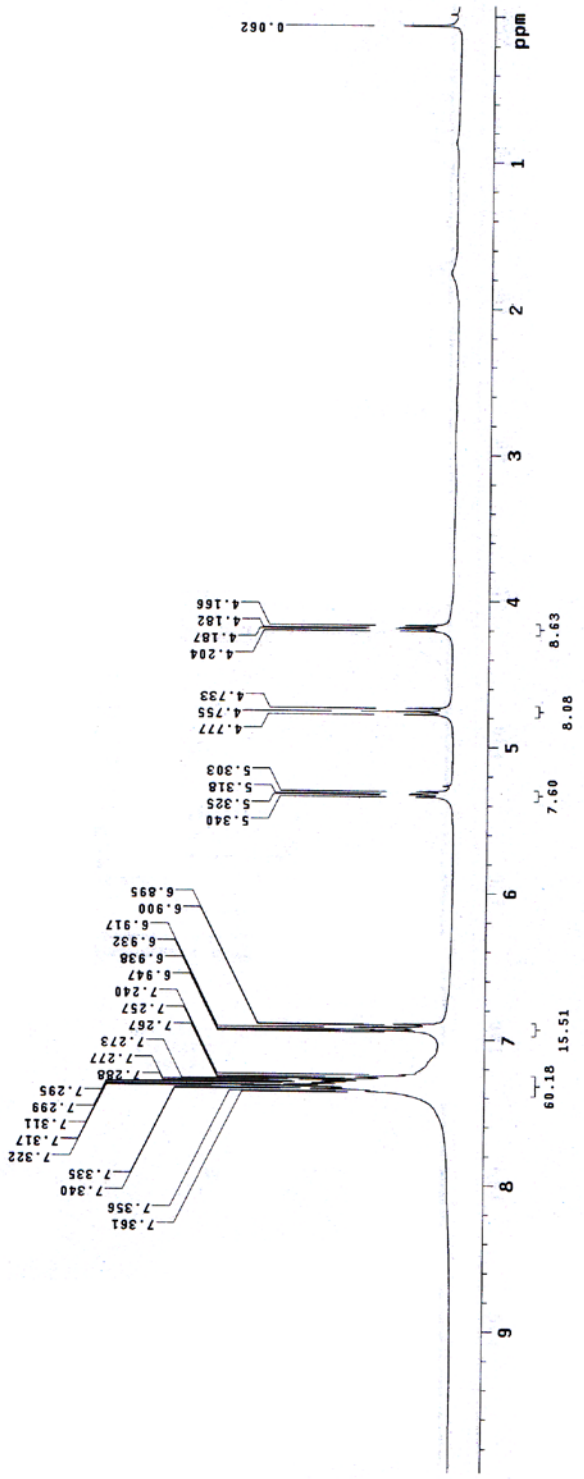

MSN-177
exp1 s2pul
SAMPLE
date Aug 9 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-177-C1~
3.fid
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 800
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -62.6
wp 2825.9
rfl 9279.8
rfp 7764.9
rp -148.3
lp -331.5
PLOT
wc 250
sc 0
vs 15
th 2
nm no ph

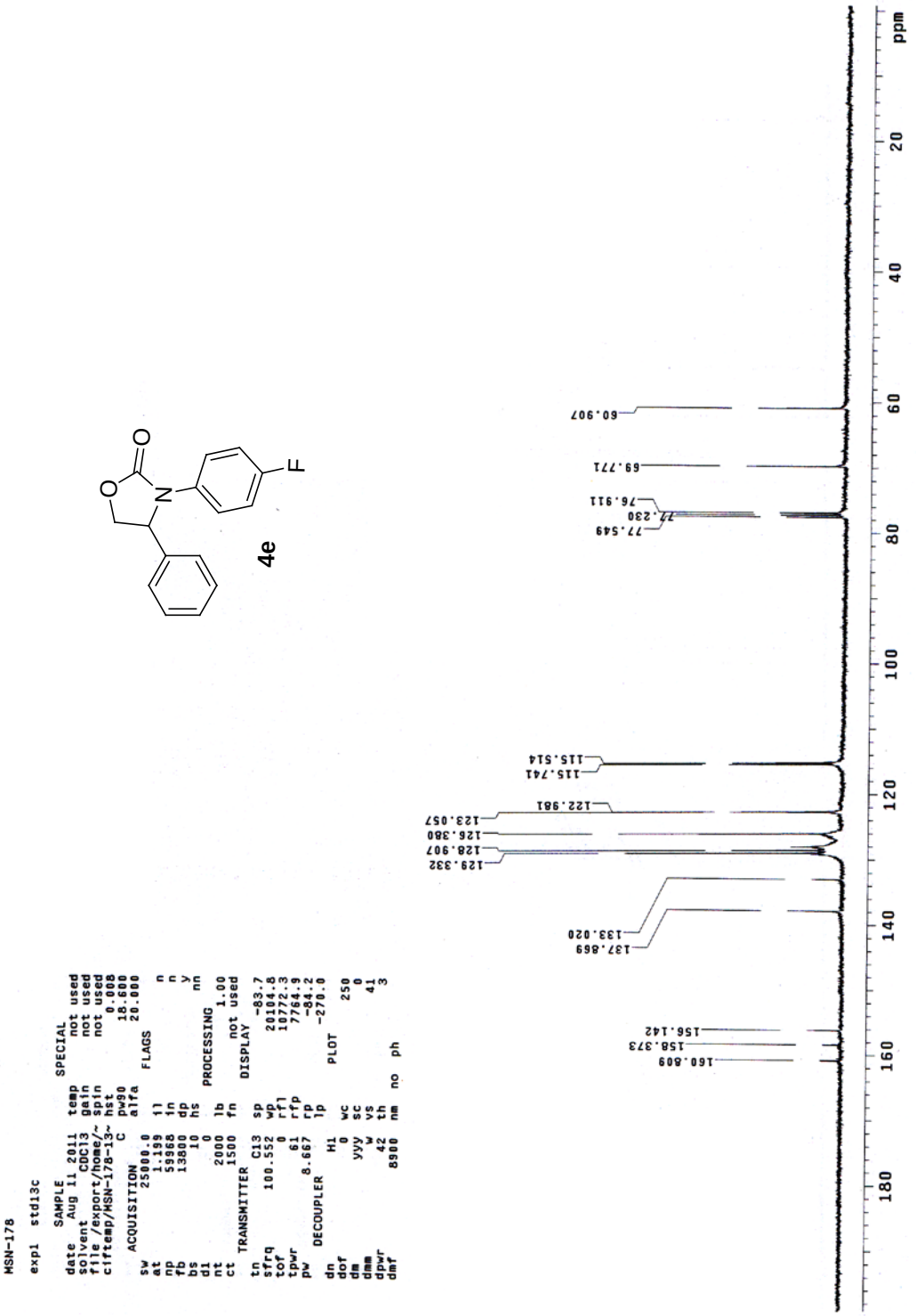

155.691
137.892
135.749
130.036
129.602
129.137
129.076
126.338
122.090
77.543
77.230
76.910
69.924
60.696
ppm

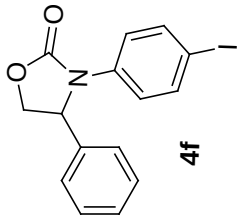

O
O
N
F

4e

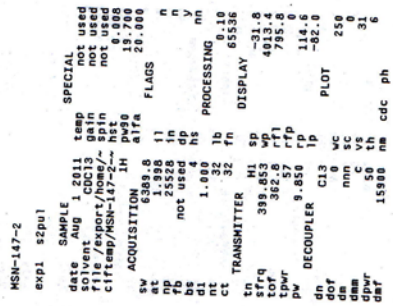

MSN-178
expl s2pul
SAMPLE
date Aug 10 2011
solvent CDCl3
file /export/home/~
curdir /MSN-178-1H
ACQUISITION
sw 5389.8
at 1.998
np 15528
fb not used
bs 4
nt 32
ct 32
TRANSMITTER H1
tn H1
sfrq 399.853
tof 362.0
tpwr 57
pw 9.850
DECOUPLER C13
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -30.0
wp 4013.4
rfl 3697.3
rfp 2894.9
rp 73.5
lp -13.1
PLOT
wc 250
sc 0
vs 36
th 7
cdc ph
nm

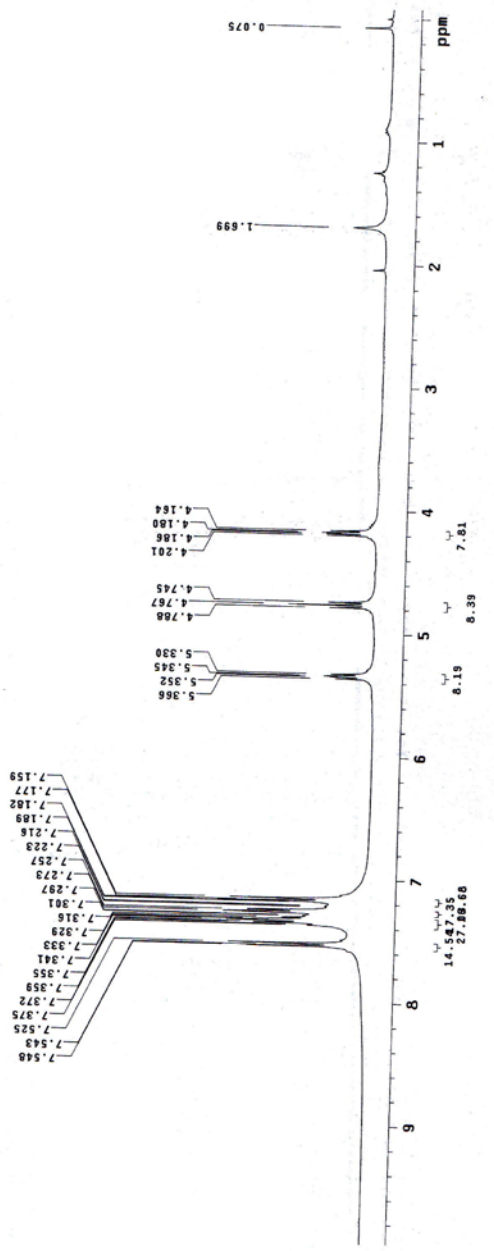

0.062
4.166
4.182
4.187
4.204
4.733
4.755
4.777
5.303
5.318
5.325
5.340
6.895
6.900
6.917
6.932
6.938
6.947
7.240
7.257
7.267
7.273
7.277
7.288
7.295
7.299
7.311
7.317
7.322
7.335
7.340
7.356
7.361
ppm
8.63
8.08
7.60
15.51
60.18

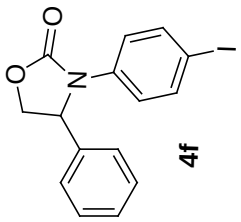

MSN-178
expl std13c
SAMPLE
date Aug 11 2011
solvent CDCl3
file /export/home/~
ciftemp/MSN-178-13~
C
ACQUISITION
sw 25000.0
at 1.199
np 59968
fb 13800
bs 10
d1 2000
nt 1500
ct
TRANSMITTER
tn C13
sfrq 100.552
tof 0
tpwr 61
pw 8.667
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -83.7
wp 20104.8
rfl 10772.3
rfp 7764.9
rp -84.2
lp -270.0
PLOT
wc 250
sc 0
vs 41
th 3
nm no ph
4e
F
60.907
69.771
76.911
77.230
77.549
115.514
115.741
122.981
123.057
126.380
128.907
129.332
133.020
137.869
156.142
158.373
160.809
ppm
20
40
60
80
100
120
140
160
180



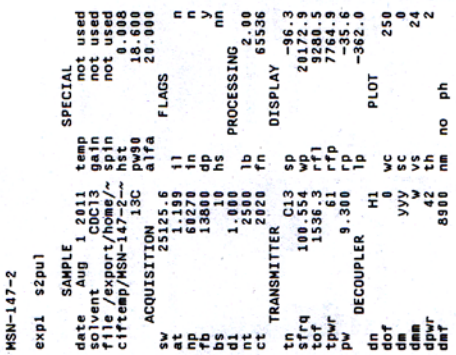
4f

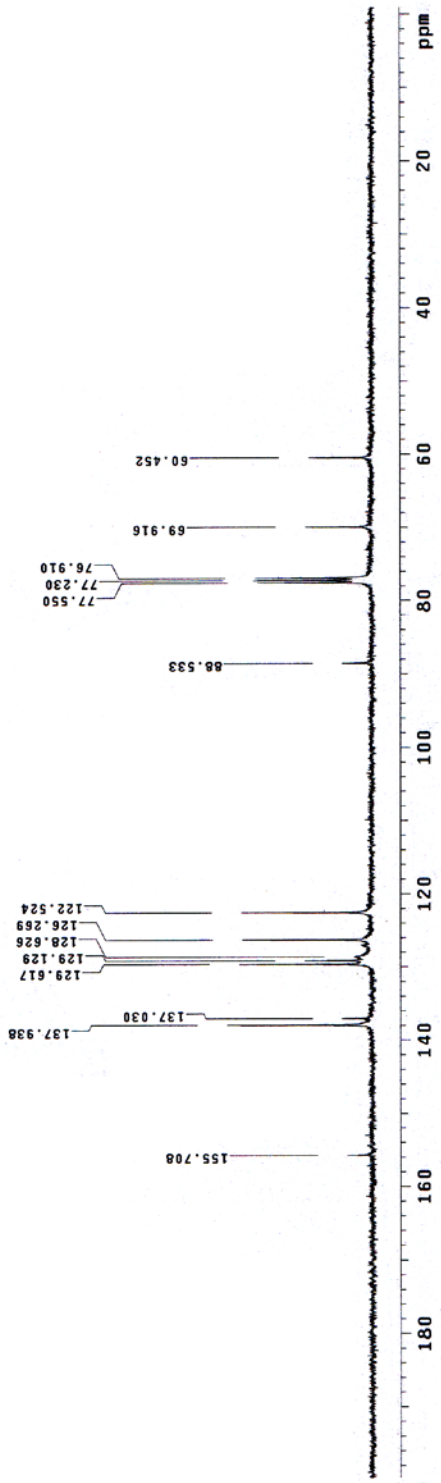


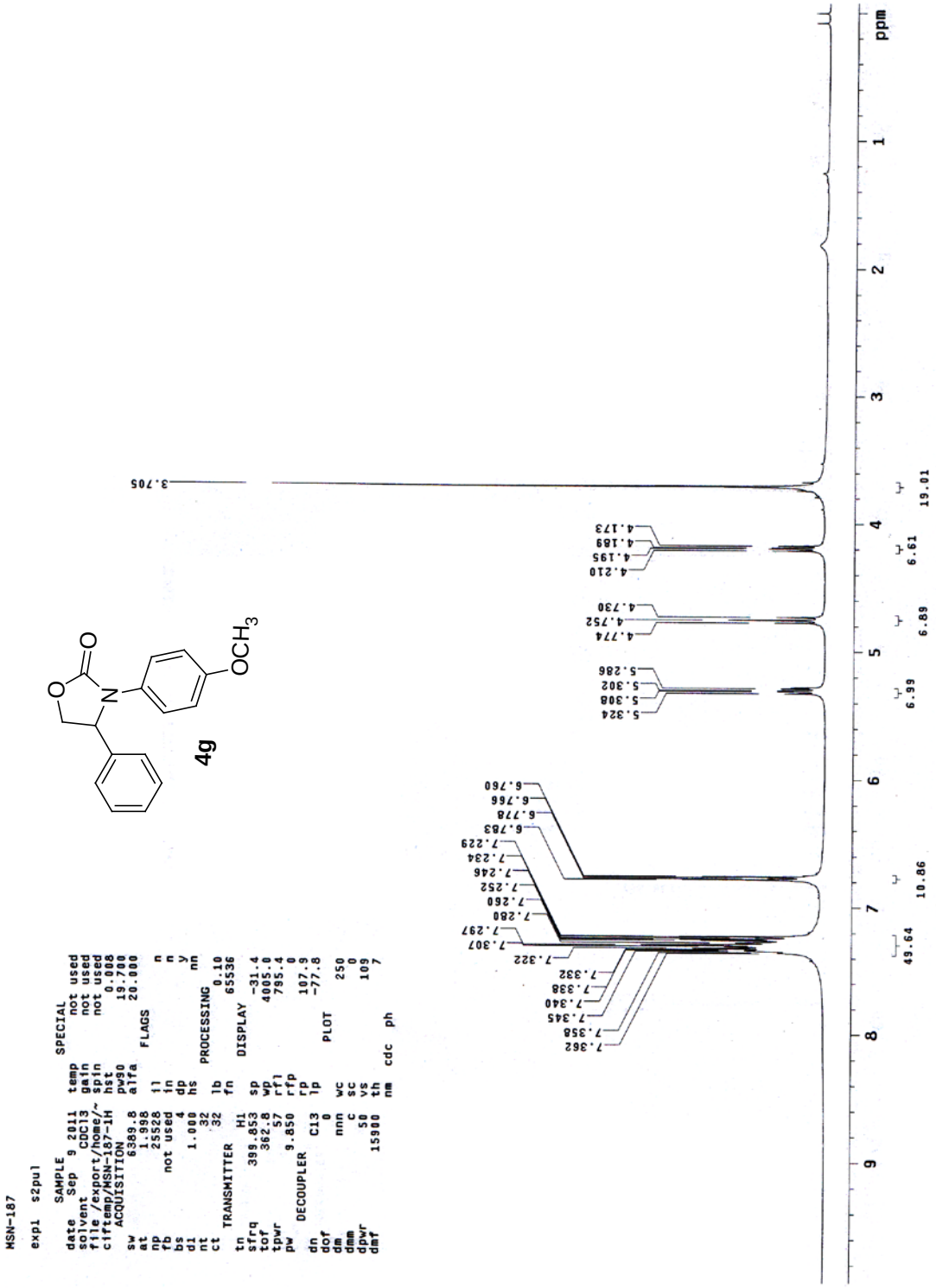


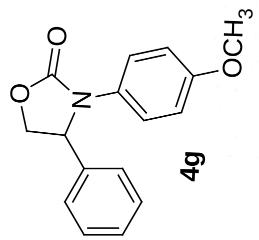


4f

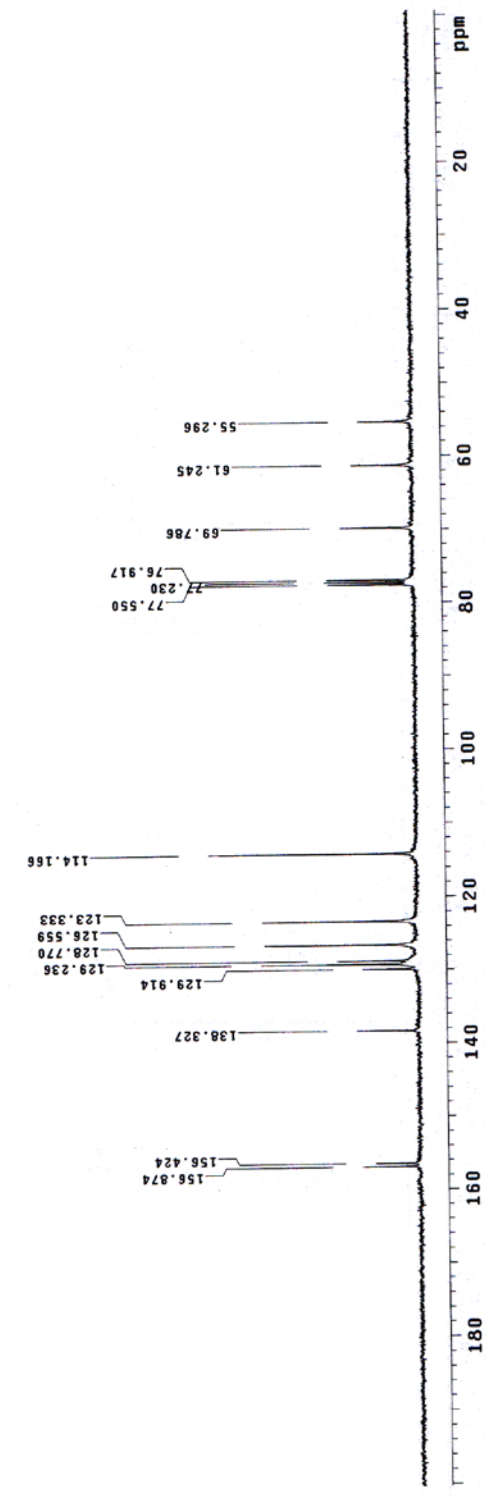

MSN-147-2
expl s2pul
SAMPLE
date Aug 1 2011
solvent CDCl3
file /export/home/~
cltemp/MSN-147-2~~
ACQUISITION 13C
sw 25125.6
at 1.139
np 68270
fb 13800
bs 10
d1 1.000
nt 2500
ct 2020
TRANSMITTER C13
tn
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER H1
dn
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
fl n
fn n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -96.3
wp 20172.9
rfl 3280.5
rfp 7764.9
rp -35.6
lp -362.0
PLOT
wc 250
sc 0
vs 24
th 2
nm no ph

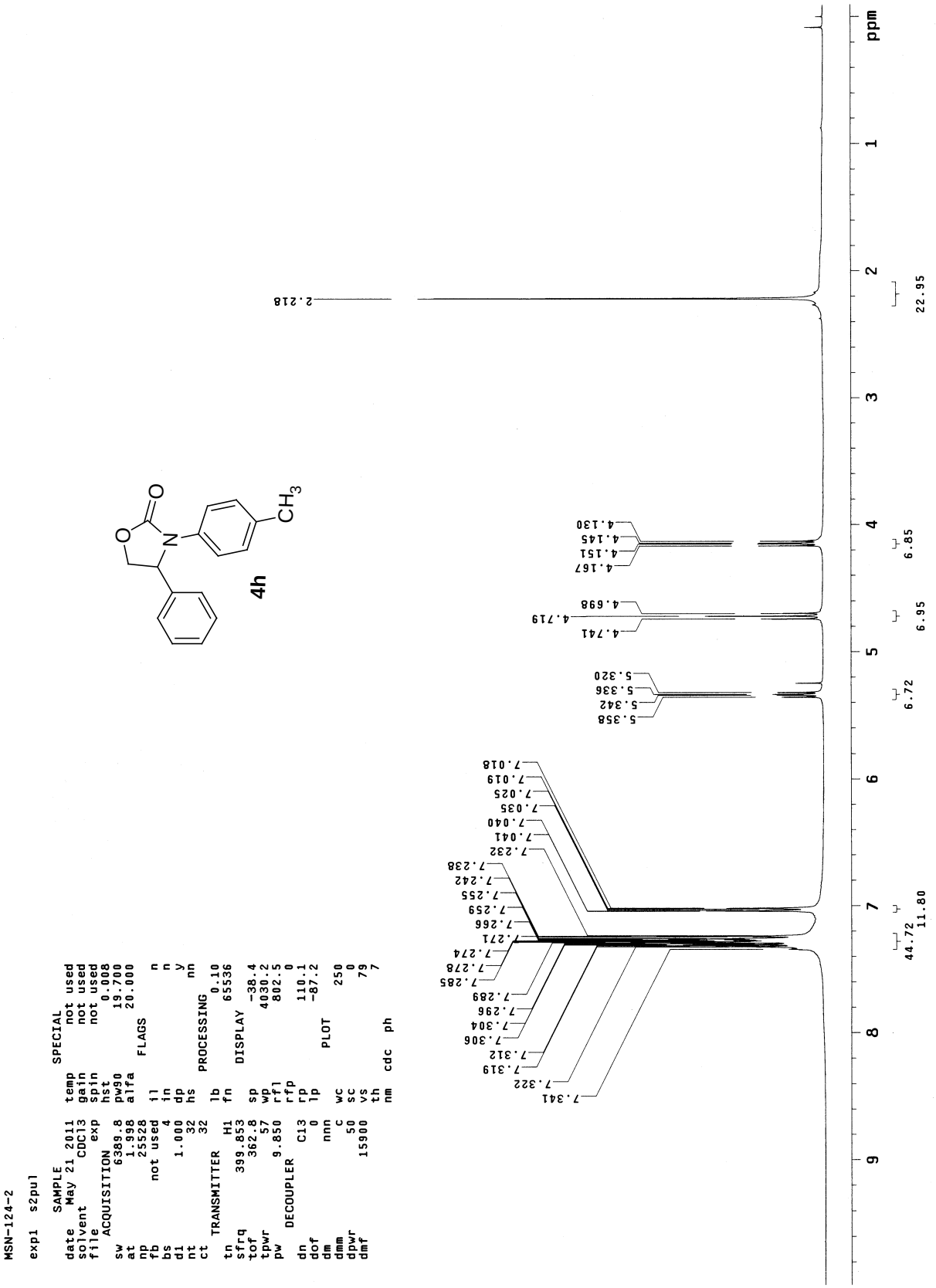

155.708
137.938
137.030
129.617
129.129
128.626
126.269
122.524
88.533
77.550
77.230
76.910
69.916
60.452
ppm

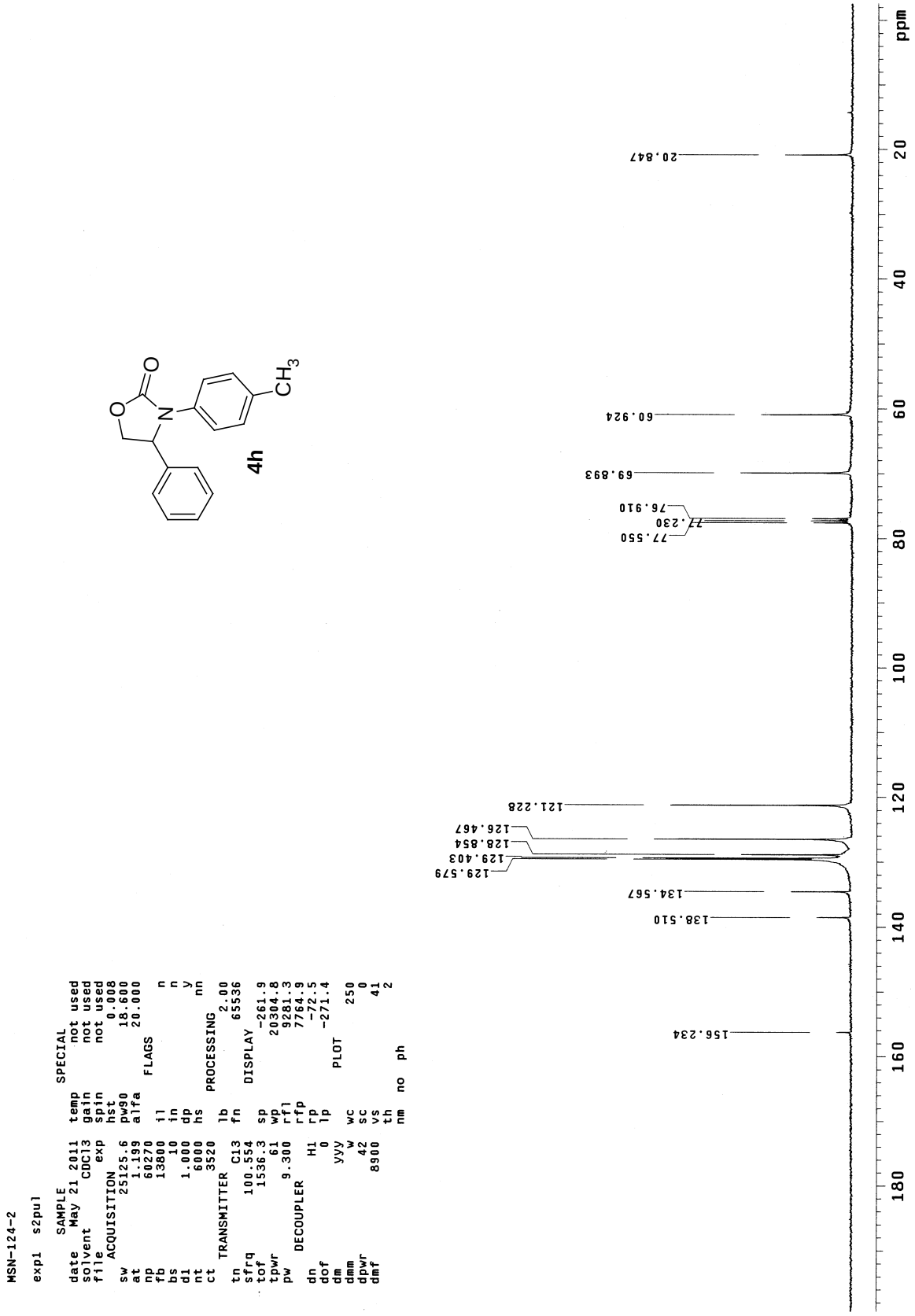


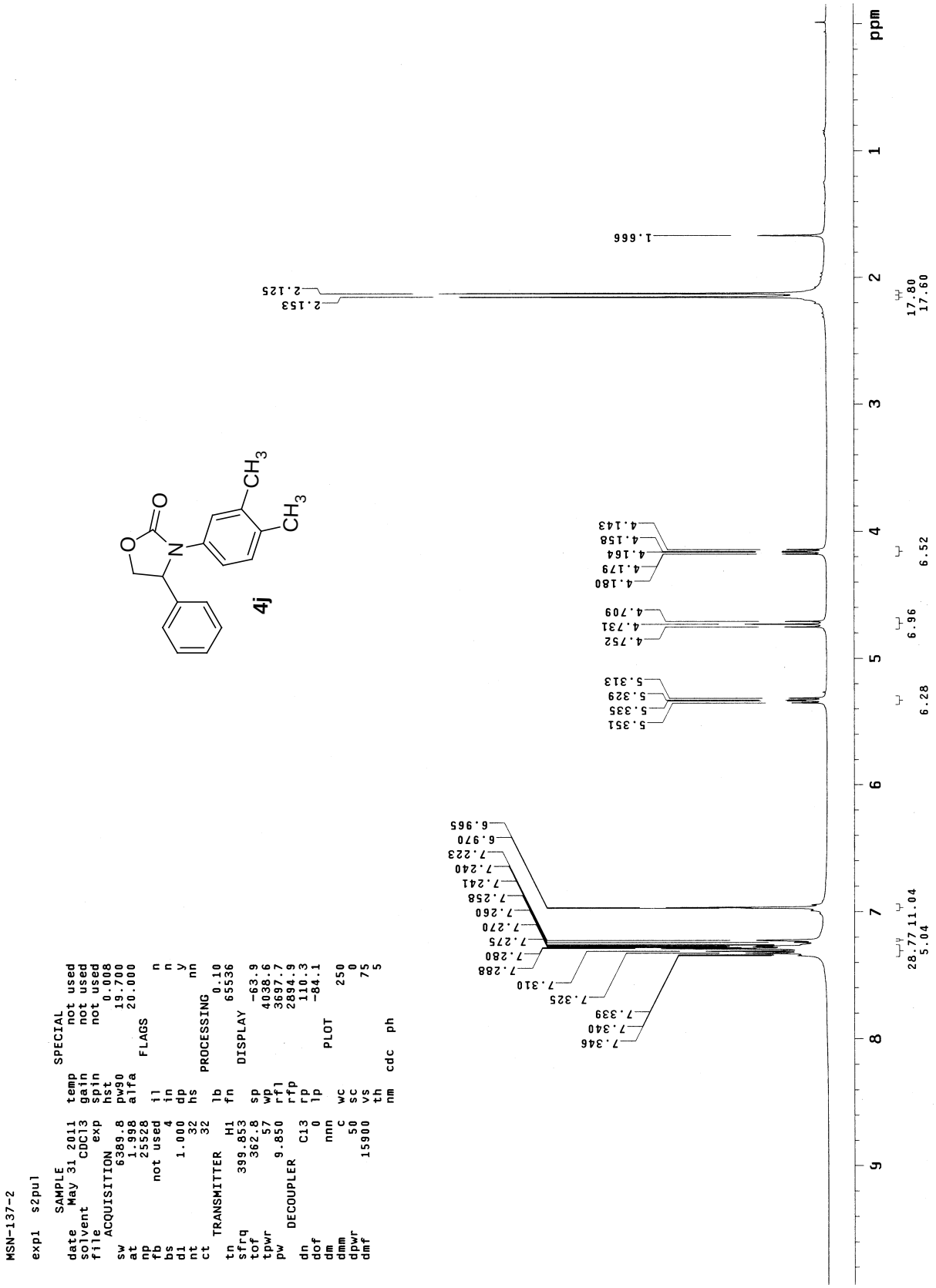


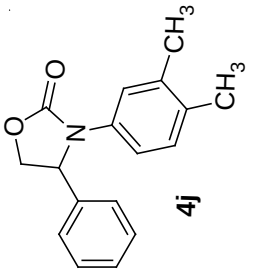
4g




MSN-124-2
exp1 s2pul
SAMPLE
date May 21 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -38.4
wp 4030.2
rfl 802.5
rfp 0
rp 110.1
lp -87.2
PLOT
wc 250
sc 0
vs 79
th 7
nm cdc ph
4h
2.218
4.130
4.145
4.151
4.167
4.698
4.719
4.741
5.320
5.336
5.342
5.358
7.018
7.019
7.025
7.035
7.040
7.041
7.232
7.238
7.242
7.255
7.259
7.266
7.271
7.274
7.278
7.285
7.289
7.296
7.304
7.306
7.312
7.319
7.322
7.341
ppm
22.95
6.85
6.95
6.72
44.72
11.80

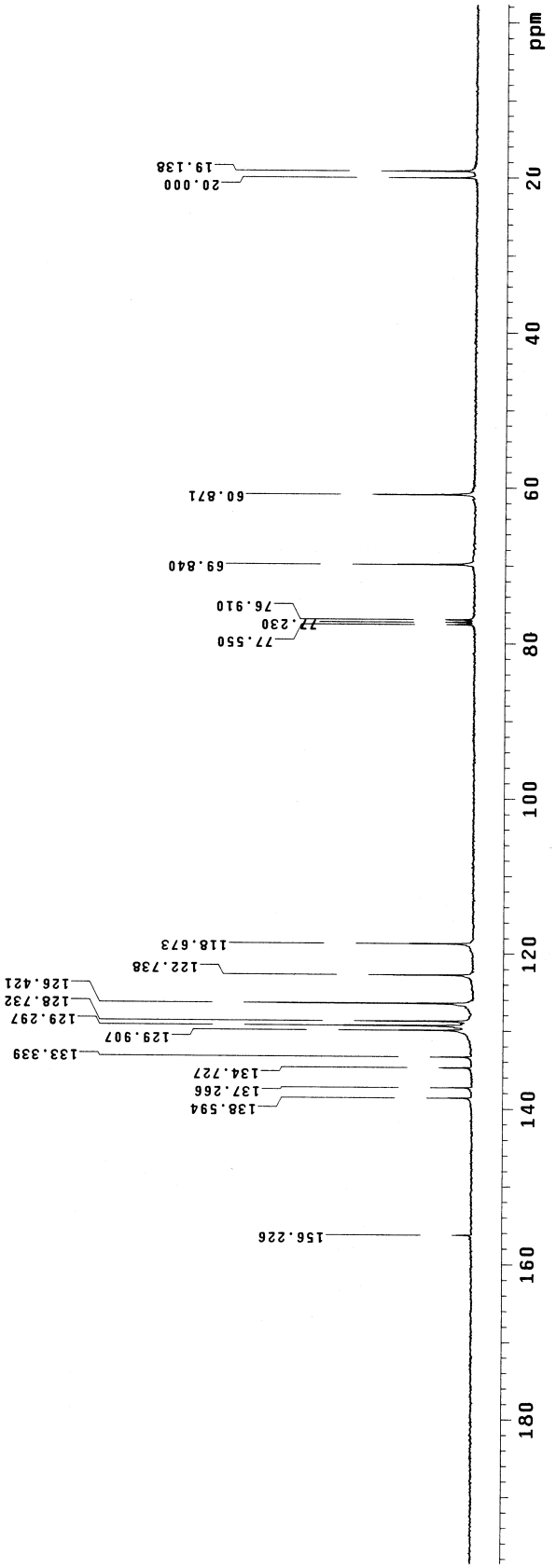

MSN-124-2
expl s2pul
SAMPLE
date May 21 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 6000
ct 3520
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -261.9
wp 20304.8
rfl 9281.3
rfp 7764.9
rp -72.5
lp -271.4
PLOT
wc 250
sc 0
vs 41
th 2
nm no ph
4h
20.847
60.924
69.893
76.910
77.230
77.550
121.228
126.467
128.854
129.403
129.579
134.567
138.510
156.234
ppm

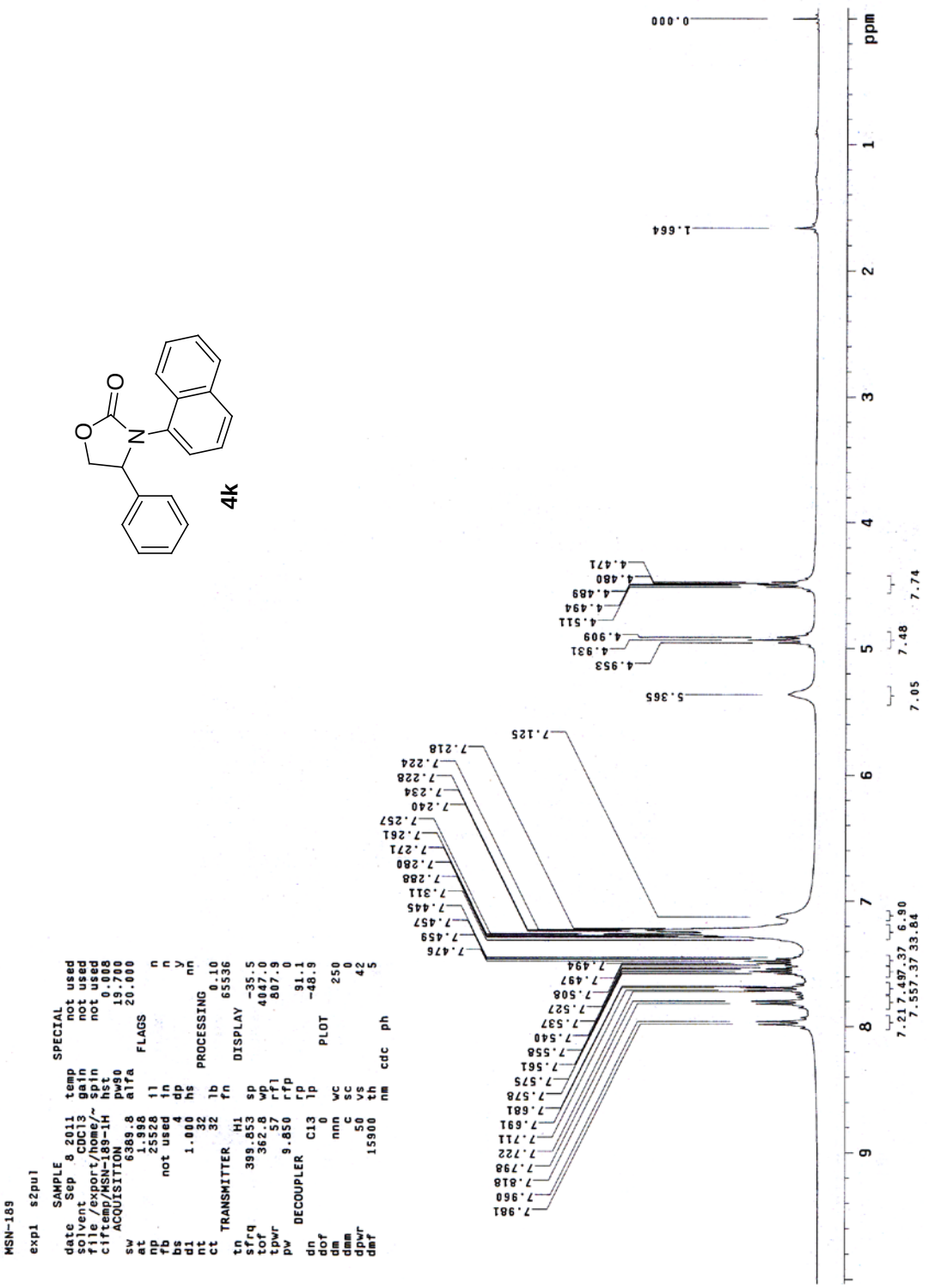

MSN-137-2
exp1 s2pul
SAMPLE
date May 31 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
not used
not used
not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -63.9
wp 4038.6
rfl 3697.7
rfp 2894.9
rp 110.3
lp -84.1
PLOT
wc 250
sc 0
vs 75
th 5
nm cdc ph
4j
1.666
2.125
2.153
4.143
4.158
4.164
4.179
4.180
4.709
4.731
4.752
5.313
5.329
5.335
5.351
6.965
6.970
7.223
7.240
7.241
7.258
7.260
7.270
7.275
7.280
7.288
7.310
7.325
7.339
7.340
7.346
28.77 11.04
5.04
17.80
17.60
6.52
6.96
6.28
ppm

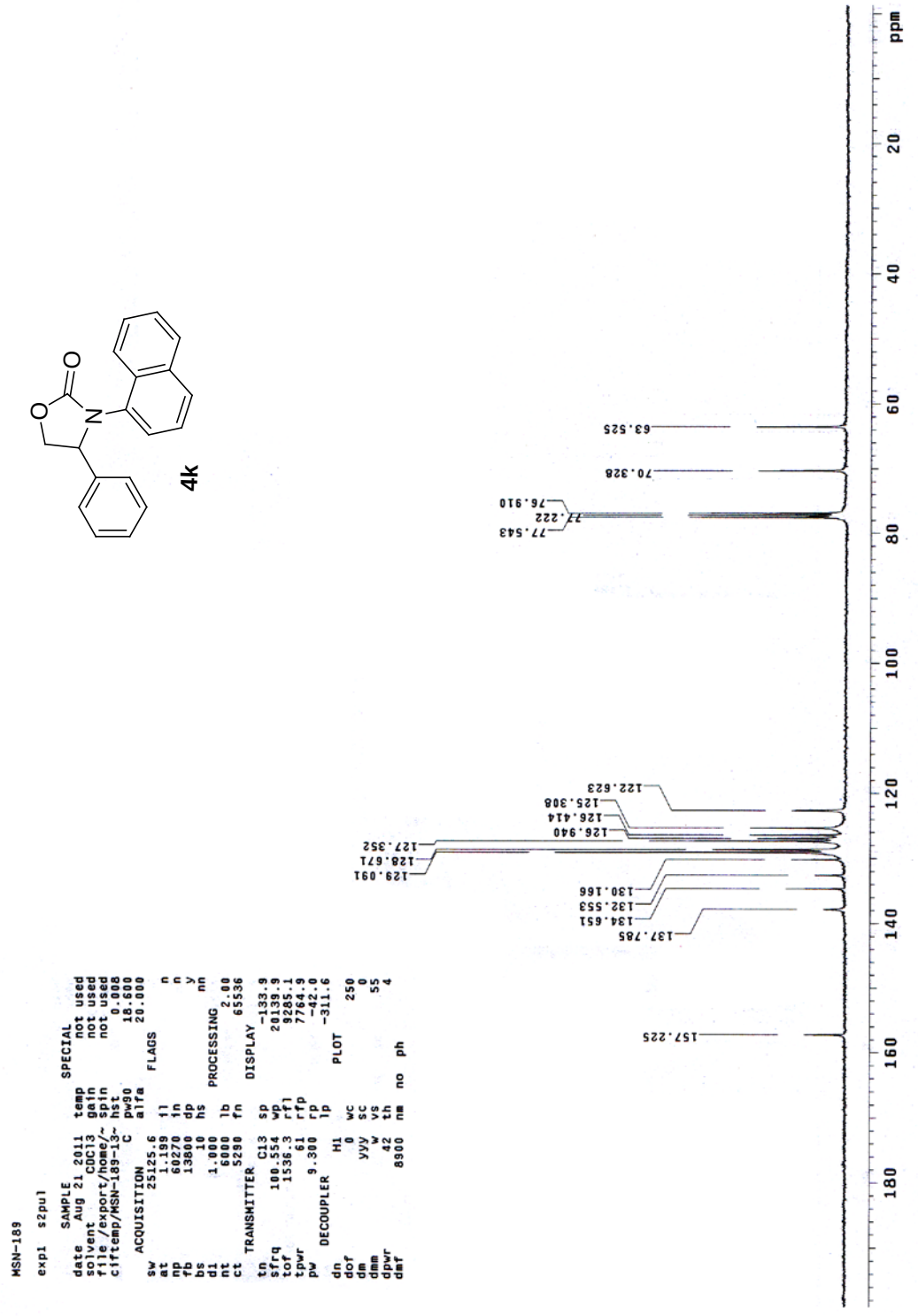


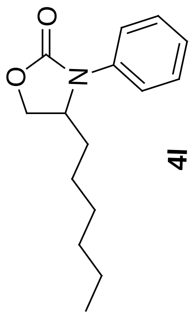
4j

```
MSN-137-2

exp1  s2pul

          SAMPLE                  SPECIAL
date   May 31 2011  temp          not used
solvent      CDCl3  gain          not used
file           exp  spin          not used
  ACQUISITION       hst            0.008
sw         25125.6  pw90          18.600
at               1  alfa          20.000
np           60270  FLAGS
fb           13800  il                 n
bs              10  in                 n
d1           1.000  dp                 y
nt            5000  hs                nn
ct            1480  PROCESSING         2.00
  TRANSMITTER       lb                65536
tn              C13  fn
sfrq        100.554  DISPLAY
tof          1536.3  sp             -236.6
tpwr             61  wp            20205.9
pw            9.300  rfl            9289.0
  DECOUPLER         rfp            7764.9
dn              H1   rp              -60.2
dof              0   lp             -288.6
dm               yyy  PLOT
dmm              w    wc              250
dpwr            42    sc                0
dmf           8900    vs               36
                      th                3
                      nm  no  ph
```

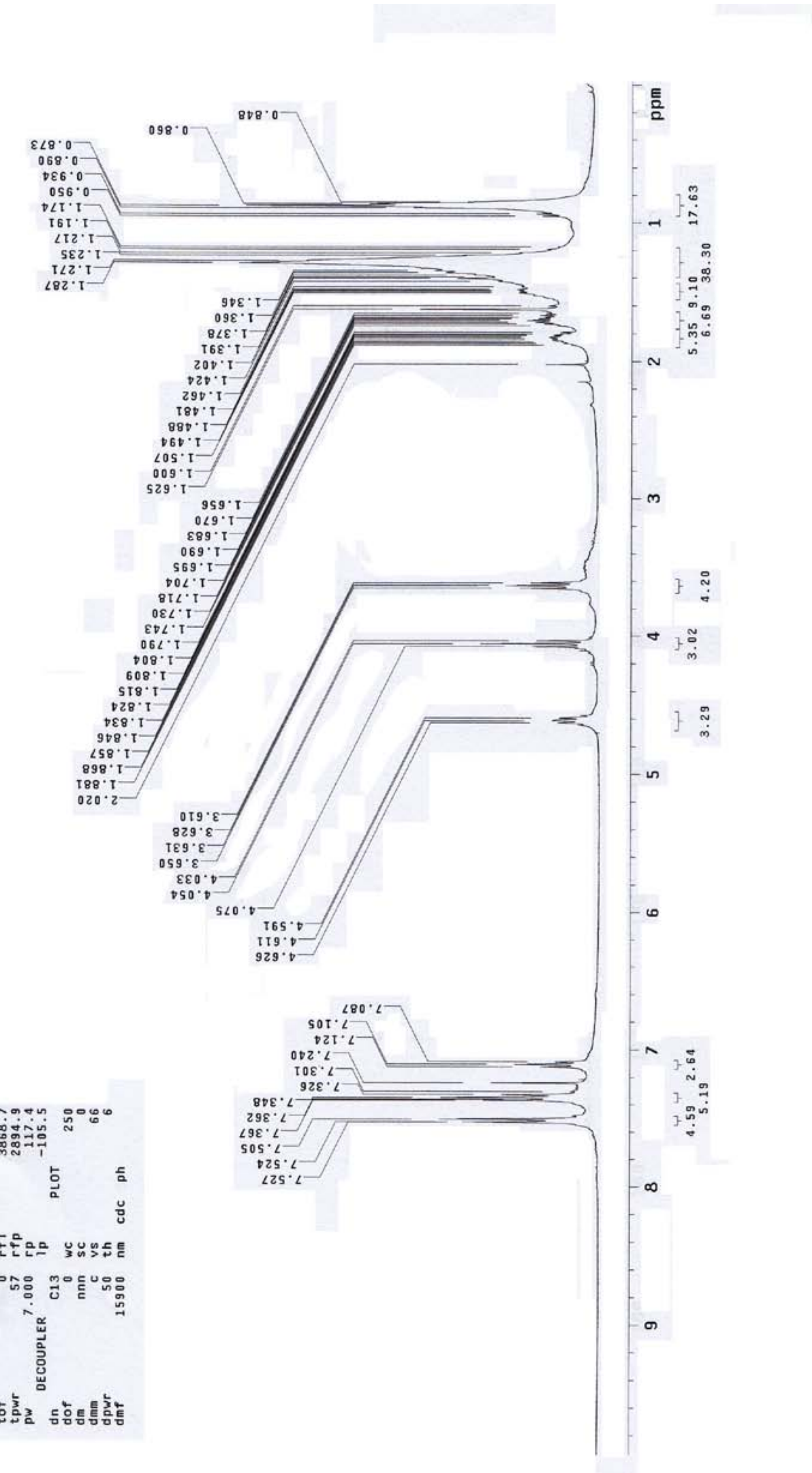


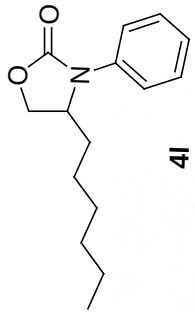

4k
MSN-189
expl s2pul
SAMPLE
date Sep 8 2011
solvent CDCl3
file /export/home/-
cltemp/MSN-189-1H
ACQUISITION
sw 6388.8
at 1.998
np 25528
fb not used
bs 4
bs 1.000
ds 32
nt 32
ct
TRANSMITTER H1
tn
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER C13
dn 0
dof nnn
dm c
dmm 50
dpwr 15900
dmf
SPECIAL not used
temp not used
gain not used
spin 0.008
hst 19.700
pw90 20.000
alfa
FLAGS
il n
in n
dp y
hs nn
PROCESSING 0.10
lb
fn 65536
DISPLAY -35.5
sp 4047.0
wp 807.9
rfl 0
rfp 91.1
rp -48.9
lp
PLOT
wc 250
sc 0
vs 42
th 5
nm cdc ph
0.000
1.664
4.471
4.480
4.489
4.494
4.511
4.909
4.931
4.953
5.365
7.125
7.218
7.224
7.228
7.234
7.240
7.257
7.261
7.271
7.280
7.288
7.311
7.445
7.457
7.459
7.476
7.494
7.497
7.508
7.527
7.537
7.540
7.558
7.561
7.575
7.578
7.681
7.691
7.711
7.722
7.798
7.818
7.960
7.981
7.21 7.49 7.37 6.90
7.55 7.37 33.84
7.05
7.48
7.74
ppm


4k
157.225
137.785
134.651
132.553
130.166
129.091
128.671
127.352
126.940
126.414
125.308
122.623
77.543
77.222
76.910
70.328
63.525
ppm
20
40
60
80
100
120
140
160
180

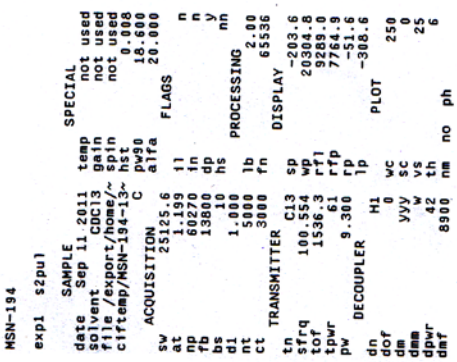


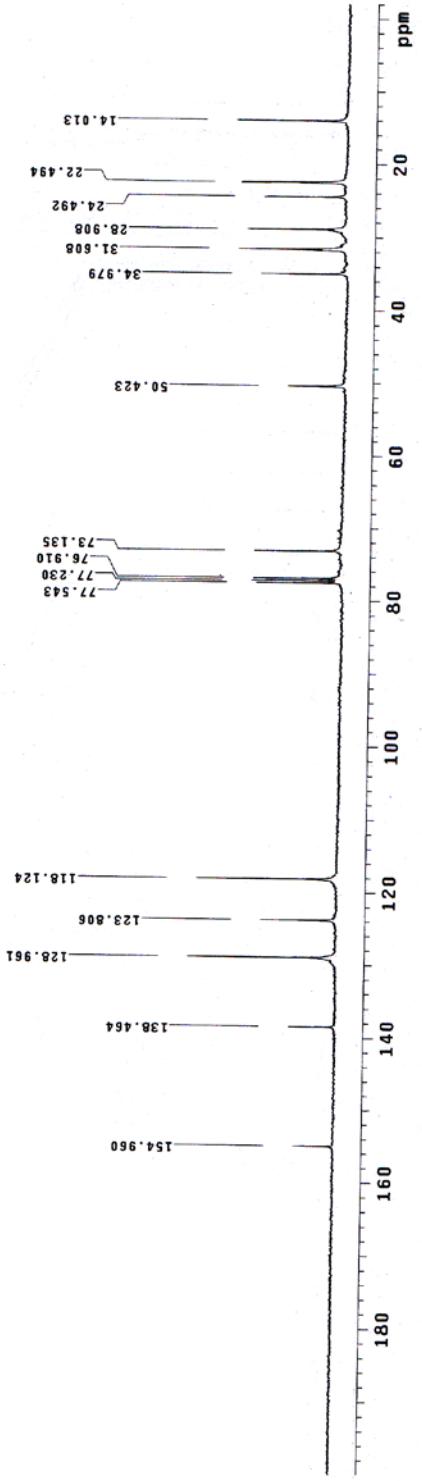
4l

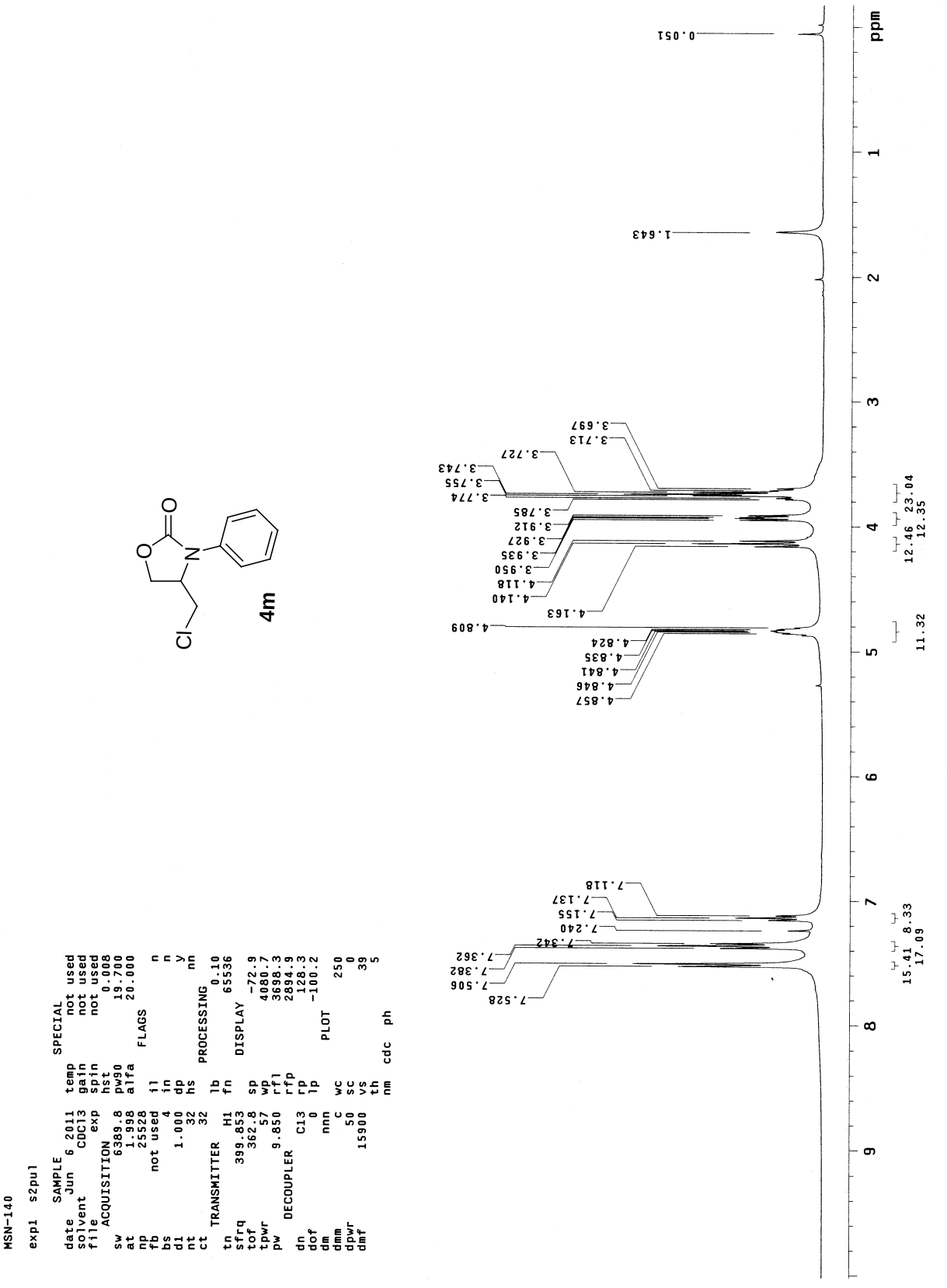


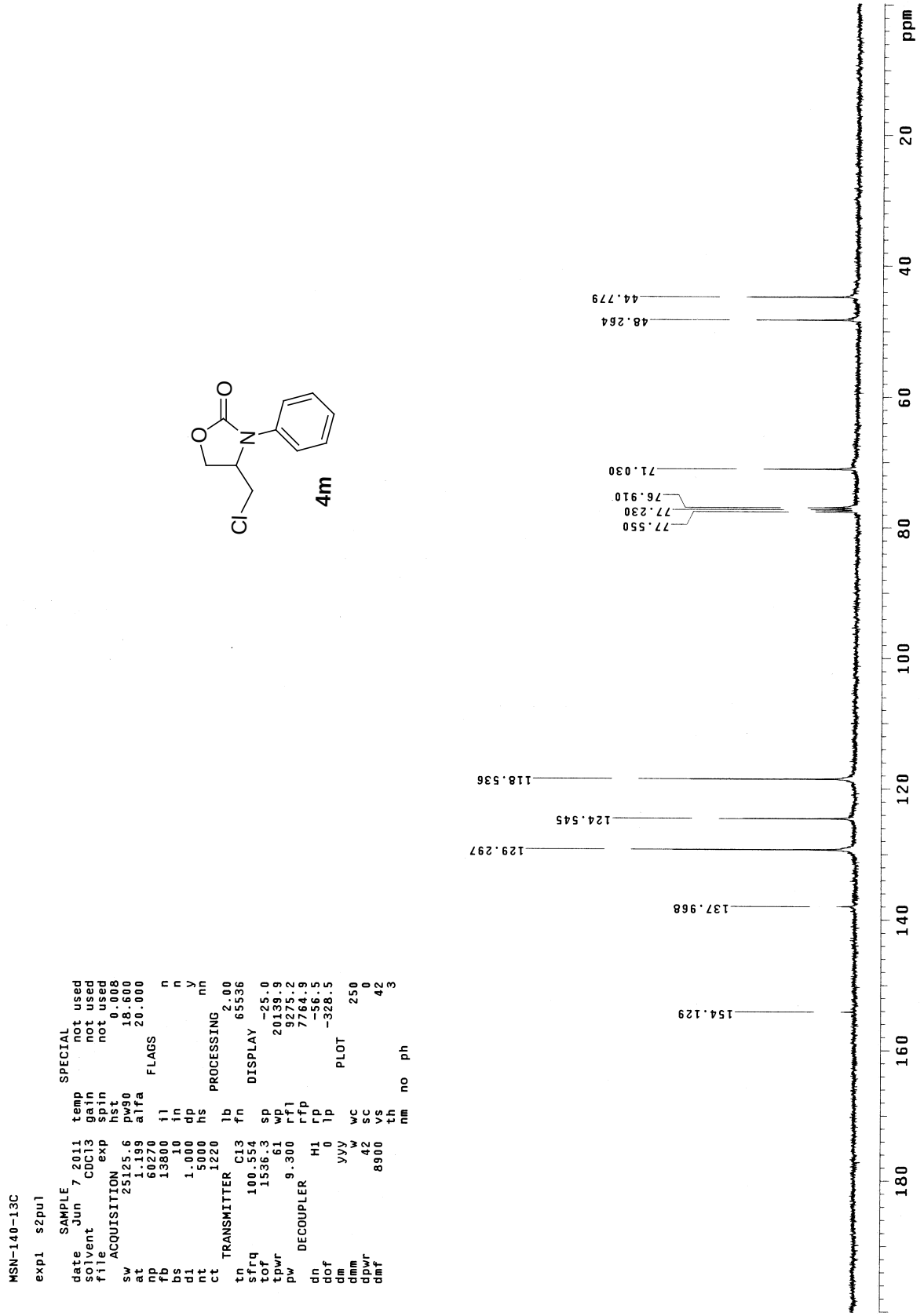


4l


MSN-194
expl s2pul
SAMPLE SPECIAL
date Sep 11 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/MSN-194-13~ hst 0.008
C pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 10 hs nn
d1 1.000 PROCESSING
nt 5000 lb 2.00
ct 3000 fn 65536
TRANSMITTER DISPLAY
tn C13 sp -203.6
sfrq 100.554 wp 20304.8
tof 1536.3 rfl 9289.0
tpwr 61 rfp 7764.9
pw 9.300 rp -51.6
DECOUPLER H1 lp -308.6
dn H1 PLOT
dof 0 wc 250
dm yyy sc 0
dmm w vs 25
dpwr 42 th 6
dmf 8900 nm no ph


14.013
22.494
24.492
28.908
31.608
34.979
50.423
73.135
76.910
77.230
77.543
118.124
123.806
128.961
138.464
154.960
ppm


MSN-140
exp1 s2pul
SAMPLE
date Jun 6 2011
solvent CDCl3
file exp
ACQUISITION
sw 6389.8
at 1.998
np 25528
fb not used
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 399.853
tof 362.8
tpwr 57
pw 9.850
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 50
dmf 15900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 19.700
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 65536
DISPLAY
sp -72.9
wp 4080.7
rfl 3698.3
rfp 2894.9
rp 128.3
lp -100.2
PLOT
wc 250
sc 0
vs 39
th 5
nm
cdc ph
4m
0.051
1.643
3.697
3.713
3.727
3.743
3.755
3.774
3.785
3.912
3.927
3.935
3.950
4.118
4.140
4.163
4.809
4.824
4.835
4.841
4.846
4.857
7.118
7.137
7.155
7.240
7.342
7.362
7.382
7.506
7.528
ppm
12.46 23.04
12.35
11.32
15.41 8.33
17.09


MSN-140-13C
expl s2pul
SAMPLE
date Jun 7 2011
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 10
d1 1.000
nt 5000
ct 1220
TRANSMITTER
tn C13
sfrq 100.554
tof 1536.3
tpwr 61
pw 9.300
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.600
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -25.0
wp 20139.9
rfl 9275.2
rfp 7764.9
rp -56.5
lp -328.5
PLOT
wc 250
sc 0
vs 42
th 3
nm no ph
4m
Cl
O
O
N
154.129
137.968
129.297
124.545
118.536
77.550
77.230
76.910
71.030
48.264
44.779
ppm
180
160
140
120
100
80
60
40
20