Direct generation of oxygen-stabilized radicals by H• transfer from transition metal hydrides

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Supporting Information

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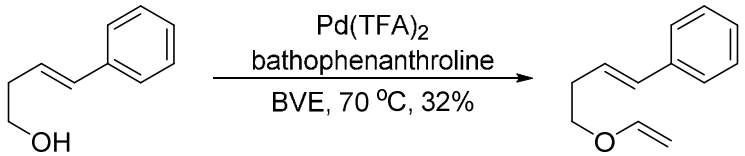
GENERAL METHODS

All reactions were carried out under an atmosphere of argon in glassware that had been either oven-dried or flame-dried under vacuum and backfilled with argon. Unless otherwise noted, all reagents were commercially obtained and, where appropriate, purified prior to use. Deuterated benzene (C_6D_6) and THF were purified over molten potassium & benzophenone ketyl. Et_2O, 1,4-dioxane, and benzene (C_6H_6) were distilled from sodium-benzophenone ketyl. Toluene (PhMe) and CH_2Cl_2 were dried by filtration through alumina. Triethylamine (Et_3N) was distilled from CaH_2. Butyl vinyl ether for kinetic runs was purified by vacuum transfer from CaH_2 onto activated 4Å molecular sieves. $CpCr(CO)_3D$, $CpCr(CO)_3H$,[1] and $HV(CO)_4(dppe)$[2] were synthesized according to the literature procedures and stored in an argon atmosphere glovebox (O_2 < 1 ppm). ^1H NMR and ^{13}C NMR spectra were recorded using a Bruker 500 Ascend, DRX 500, DRX 400, or DRX 300 spectrometer. The data are reported as follows: chemical shift in parts per million from internal tetramethylsilane on the δ scale, integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet), and coupling constants (Hz). High-resolution mass spectra were acquired on a JEOL JMS-HX110 HF mass spectrometer and were obtained by peak matching.

EXPERIMENTAL PROCEDURES AND CHARACTERIZATION DATA



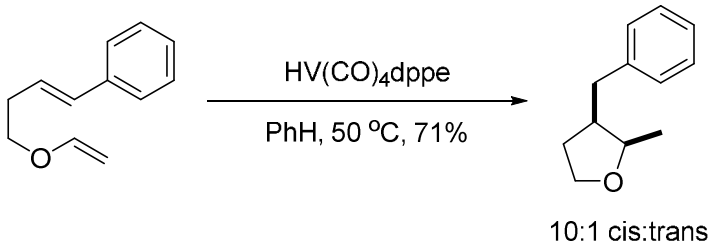
(E)-(4-(vinyloxy)but-1-en-1-yl)benzene. Bathophenanthroline (2.5 mg, 0.007 mmol) and $Pd(CF_3CO_2)_2$ (2.5 mg, 0.007 mmol) were added to butyl vinyl ether (4 mL, 29.8 mmol). (E)-4-phenylbut-3-en-1-ol (200 mg, 1.49 mmol) was added, and the mixture was stirred at 70°C overnight. The crude mixture was purified by flash column chromatography using 50/50 toluene/hexane as an eluent to afford 77 mg (32%) of the vinyl ether.

^1H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.23 – 7.13 (m, 1H), 6.52 – 6.45 (m, 2H), 6.23 (dt, *J* = 16.5 Hz, 6.5 Hz, 1H), 4.21 (dd, *J* = 14 Hz, 2 Hz, 1H), 4.01 (dd, *J* = 7 Hz, 2 Hz, 1H), 3.80 (t, *J* = 6.5 Hz, 2H), 2.58 (q, *J* = 7 Hz, 2H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 151.9, 137.5, 132.2, 128.6, 127.3, 126.2, 126.2, 86.6, 67.4, 32.8;

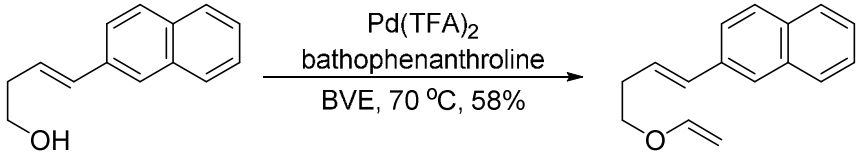
IR (ATR) 2924, 2870, 1612, 1194, 962, 813, 741, 692 cm^{-1};

HRMS (FAB) *m/z* calcd $C_{12}H_{14}O$: 174.1045, found: 174.1041.



10:1 cis:trans

3-benzyl-2-methyltetrahydrofuran. (E)-(4-(vinyloxy)but-1-en-1-yl)benzene (8.0 mg, 0.046 mmol) and $HV(CO)_4dppe$ (54 mg, 0.096 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 5.7 mg of the product (71%). The spectral data matches the data reported in the literature.[3]



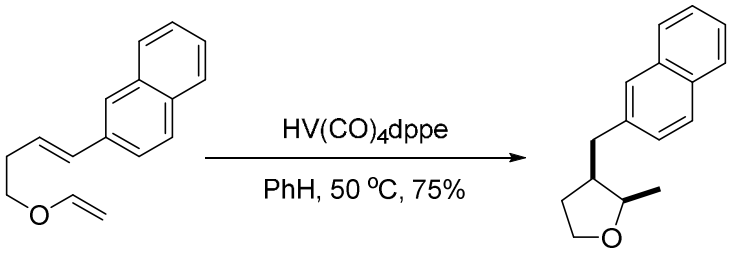
(E)-2-(4-(vinyloxy)but-1-en-1-yl)naphthalene. Bathophenanthroline (1.7 mg, 0.005 mmol) and $Pd(CF_3CO_2)_2$ (1.7 mg, 0.005 mmol) were added to butyl vinyl ether (2.6 mL, 20.2 mmol). (E)-4-(naphthalen-2-yl)but-3-en-1-ol (200 mg, 1.01 mmol) was added, and the mixture was stirred at 70°C overnight. The crude mixture was purified by flash column chromatography using 10/90 toluene/hexane as an eluent to afford 132 mg (58%) of the vinyl ether.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.77 (m, 3H), 7.75 (s, 1H), 7.61-7.56 (m, 1H), 7.46-7.38 (m, 2H), 6.64 (d, J = 16.0 Hz, 1H), 6.50 (dd, J = 14.8 Hz, 6.8 Hz, 1H), 6.35 (dt, J = 15.6 Hz, 6.8 Hz, 1H), 4.22 (dd, J = 14.8 Hz, 2.0 Hz, 1H), 4.02 (dd, J = 6.8 Hz, 2.0 Hz, 1H), 3.84 (t, J = 6.8 Hz, 2H), 2.63 (dq, J = 6.4 Hz, 1.2 Hz, 2H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 152.0, 135.0, 133.8, 133.0, 132.4, 128.3, 128.1, 127.8, 126.8, 126.4, 125.9, 125.8, 123.7, 86.8, 67.5, 33.1;

IR (ATR) 3053, 2926, 2870, 1613, 1194, 961, 810 cm^{-1};

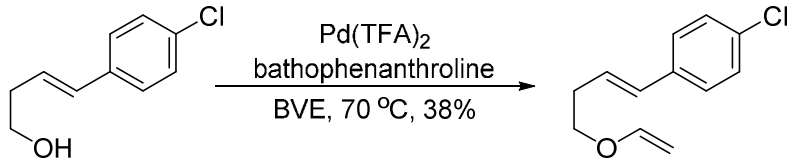
HRMS (FAB) *m/z* calcd $C_{16}H_{16}O$: 224.1201, found: 224.1193.



9:1 cis:trans

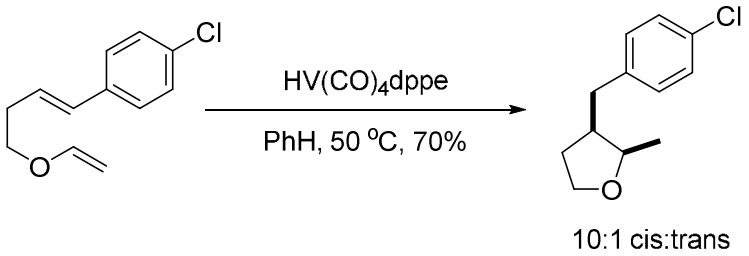
2-methyl-3-(naphthalen-2-ylmethyl)tetrahydrofuran. (E)-2-(4-(vinyloxy)but-1-en-1-yl)naphthalene (8.0 mg, 0.036 mmol) and $HV(CO)_4$dppe (42 mg, 0.075 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 6.0 mg of the product (75%) as a 9:1 ratio of cis:trans.

^{1}H NMR (500 MHz, $CDCl_3$) δ (Major Diastereomer) 7.83 – 7.74 (m, 3H), 7.62 (d, J = 1.5 Hz, 1H), 7.49 – 7.41 (m, 2H), 7.33 (dd, J = 8.5, 2.0 Hz, 1H), 4.14 (p, J = 6.0 Hz, 1H), 3.98 (td, J = 8.0, 4.5 Hz, 1H), 3.71 (q, J = 8.0 Hz, 1H), 3.01 – 2.92 (m, 1H), 2.66 – 2.52 (m, 2H), 1.92 – 1.84 (m, 1H), 1.76 – 1.69 (m, 1H), 1.23 (d, J = 6.5 Hz, 3H);
^{13}C NMR (126 MHz, $CDCl_3$) δ 138.6, 133.6, 132.0, 128.0, 127.6, 127.4, 126.9, 126.0, 125.2, 66.3, 43.3, 35.3, 30.8, 29.7, 16.3 (fully substituted sp_2 carbon missing);
IR (ATR) 2923, 2853, 1597, 1477, 1458, 1234, 747 cm^{-1};
HRMS (EI+) calcd for $C_{16}H_{18}O$ [M+]: 226.1358; found 226.1369.



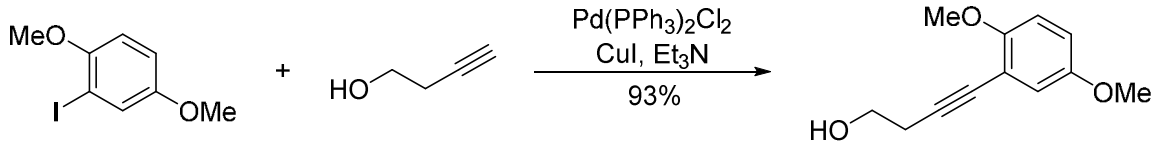
(E)-1-chloro-4-(4-(vinyloxy)but-1-en-1-yl)benzene. Bathophenanthroline (0.9 mg, 0.003 mmol) and $Pd(CF_3CO_2)_2$ (0.9 mg, 0.003 mmol) were added to butyl vinyl ether (1.4 mL, 10.9 mmol). (E)-4-(4-chlorophenyl)but-3-en-1-ol (100 mg, 0.547 mmol) was added, and the mixture was stirred at 70°C overnight. The crude mixture was purified by flash column chromatography using 20/80 toluene/hexane as an eluent to afford 43 mg (38%) of the vinyl ether.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.26-7.25 (m, 4H), 6.48 (dd, J = 14.0 Hz, 6.5 Hz, 1H), 6.43 (d, J = 16.0 Hz, 1H), 6.21 (dt, J = 15.5 Hz, 6.5 Hz, 1H), 4.20 (dd, 14.5 Hz, 2.0 Hz, 1H), 4.02 (dd, J = 7.0 Hz, 2.0 Hz, 1H), 3.80 (t, J = 7.0 Hz, 2H), 2.58 (dq, J = 6.5 Hz, 1.5 Hz, 2H);
^{13}C NMR ($CDCl_3$, 125 MHz) δ 151.8, 136.0, 132.8, 131.0, 128.7, 127.4, 127.1, 86.7, 67.3, 32.8;
IR (ATR) 2919, 1490, 1090, 1012, 804, 680 cm^{-1};
HRMS (FAB) *m/z* calcd $C_{12}H_{13}OCl$: 208.0655, found: 208.0647.

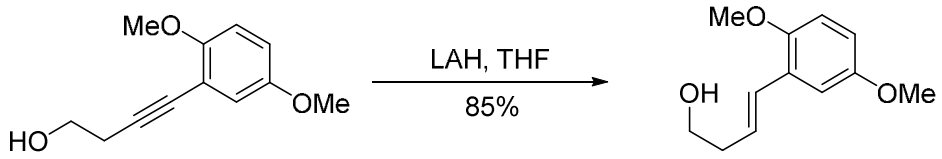


10:1 cis:trans

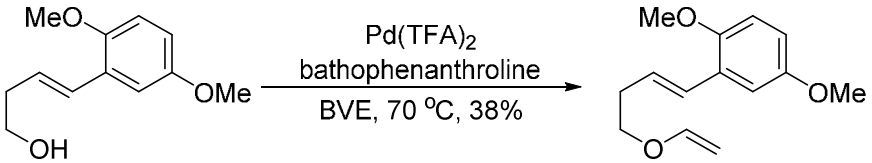
3-(4-chlorobenzyl)-2-methyltetrahydrofuran. (E)-1-chloro-4-(4-(vinyloxy)but-1-en-1-yl)benzene (8.0 mg, 0.036 mmol) and $HV(CO)_4dppe$ (46 mg, 0.081 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 5.6 mg of the product (70%) as a 10:1 ratio of cis:trans.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.23 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 4.09 (p, J = 6.0 Hz, 1H), 3.95 (td, J = 8.4, 4.4 Hz, 1H), 3.70 (q, J = 8.0 Hz, 1H), 3.48 (q, J = 7.2 Hz, 1H), 2.82 – 2.72 (m, 1H), 2.46 – 2.36 (m, 2H), 1.91 – 1.80 (m, 1H), 1.65 – 1.59 (m, 1H), 1.18 – 1.16 (d, J = 6.0 Hz, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 139.5, 131.7, 130.0, 128.5, 66.2, 43.3, 34.5, 30.6, 29.7, 16.2;
IR (ATR) 2962, 2926, 2855, 1726, 1492, 1088, 1016, 802, 696 cm^{-1}.
HRMS (EI+) calculated for $C_{12}H_{15}OCl$ [M+]: 210.0811; found 210.0809.



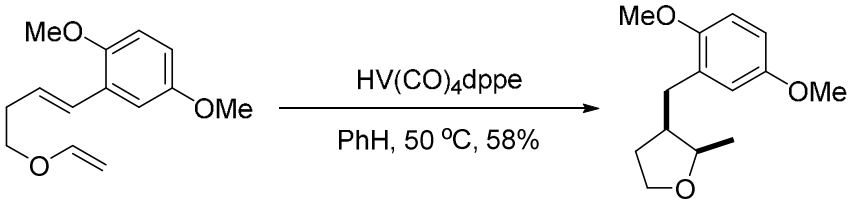
4-(2,5-dimethoxyphenyl)but-3-yn-1-ol. To a mixture of 2-iodo-1,4-dimethoxybenzene (600 mg, 2.27 mmol) in Et_3N (11 mL) was added 3-butyn-1-ol (0.21 mL, 2.73 mmol), $Pd(PPh_3)_2Cl_2$ (48 mg, 0.068 mmol). CuI was added (13 mg, 0.068 mmol) and the mixture was stirred overnight. The reaction was quenched with NH_4Cl, filtered through a pad of celite and was washed twice with Et_2O (~ 10 mL total). The reaction mixture was concentrated in vacuo and flash column chromatographed using 2/98 MeOH/DCM as the mobile phase to afford the 436 mg (93%) of product.
^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.93 (d, J = 3.5 Hz, 1H), 6.84-6.78 (m, 2H), 3.84 (s, 3H), 3.82 (q, J = 8.0 Hz, 2H), 3.76 (s, 3H), 2.74 (t, J = 7.5 Hz, 2H), 2.11 (t, J = 8.0 Hz, 1H);
^{13}C NMR ($CDCl_3$, 125 MHz) 154.7, 153.3, 118.2, 115.3, 113.0, 111.8, 90.9, 79.2, 61.1, 56.4, 55.9, 24.3;
IR (ATR) 2431, 2999, 2944, 2835, 1500, 1231, 1044 cm^{-1};
HRMS (FAB) *m/z* calcd $C_{12}H_{14}O_3$: 206.0943, found: 206.0940.



(E)-4-(2,5-dimethoxyphenyl)but-3-en-1-ol. Lithium aluminum hydride (88 mg, 2.33 mmol) was added to a pre-dried round bottom flask under argon. 8 mL of dry THF was added and 4-(2,5-dimethoxyphenyl)but-3-yn-1-ol was added in dropwise (200 mg, 0.97 mmol). The reaction was refluxed overnight. The reaction was cooled to 0°C and 0.1 mL of water was added, followed by 0.1 mL of 15% $NaOH/H_2O$, then 0.2 mL of H_2O. Magnesium sulfate was added to the mixture and allowed to stir. The solution was filtered through a fritted glass funnel, concentrated, and purified by flash column chromatography using 2/98 MeOH/DCM as the eluent to afford 171 mg (85%) of the reduced alcohol.
^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.98 (d, J = 3.5 Hz, 1H), 6.81-6.73 (m, 3H), 6.19 (dt, J = 16.0 Hz, 7.2 Hz, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 3.77 (q, J = 6.8 Hz, 2H), 2.51 (dq, J = 7.6 Hz, 1.6 Hz, 2H);
^{13}C NMR ($CDCl_3$, 125 MHz) δ 153.8, 151.0, 127.5, 127.4, 127.2, 113.3, 112.3, 112.1, 62.1, 56.3, 55.8, 36.9;
IR (ATR) 3370, 2997, 2938, 1493, 1217, 1044 cm^{-1};
HRMS (FAB) *m/z* calcd $C_{12}H_{16}O_3$: 208.1099, found: 208.1102.



(E)-1,4-dimethoxy-2-(4-(vinyloxy)but-1-en-1-yl)benzene. Bathophenanthroline (1 mg, 0.003 mmol) and $Pd(CF_3CO_2)_2$ (1 mg, 0.003 mmol) were added to butyl vinyl ether (1.1 mL, 8.54 mmol). (E)-4-(2,5-dimethoxyphenyl)but-3-en-1-ol (100 mg, 0.48 mmol) was added, and the mixture was stirred at 70°C overnight. The crude mixture was purified by flash column chromatography using toluene as an eluent to afford 43 mg (38%) of the vinyl ether.
^{1}H NMR ($CDCl_3$, 500 MHz) 6.98 (d, J = 2.5 Hz, 1H), 6.80-6.73 (m, 3H), 6.48 (dd, J = 14.5 Hz, 7.0 Hz, 1H), 6.21 (dt, J = 16.0 Hz, 7.0 Hz, 1H), 4.20 (d, J = 14.5 Hz, 1H), 4.00 (d, J = 6.5 Hz, 1H), 3.80 (t, J = 8.0 Hz, 2H), 3.80 (s, 3H), 3.78 (s, 3H), 2.60 (q, J = 7.0 Hz, 2H);
^{13}C NMR ($CDCl_3$, 125 MHz) δ 153.7, 151.8, 150.9, 127.3, 127.1, 126.7, 113.2, 112.2, 112.0, 86.5, 67.6, 56.2, 55.8, 33.1;
IR (ATR) 2938, 1615, 1496, 1221, 1048 cm^{-1} ;
HRMS (FAB) *m/z* calcd $C_{14}H_{18}O_3$: 234.1256, found: 234.1258.



7:1 cis:trans

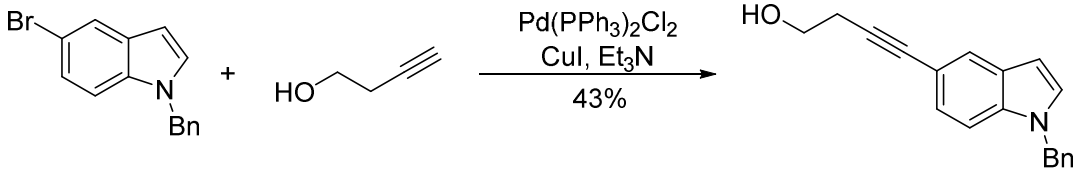
3-(2,5-dimethoxybenzyl)-2-methyltetrahydrofuran. (E)-1,4-dimethoxy-2-(4-(vinyloxy)but-1-en-1-yl)benzene (8.0 mg, 0.034 mmol) and $HV(CO)_4dppe$ (40 mg, 0.072 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 4.7 mg of the product (58%) as a 7:1 ratio of cis:trans.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.77 – 6.67 (m, 3H), 4.09 (p, J = 6.5 Hz, 1H), 3.94 (td, J = 8.0, 5.0 Hz, 1H), 3.77 (s, 3H), 3.76 (s, 3H), 3.68 (q, J = 8.0 Hz, 1H), 2.76 (dd, J = 13.0, 4.5 Hz, 1H), 2.51 – 2.36 (m, 2H), 1.86 – 1.78 (m, 1H), 1.70-1.64 (m, 1H), 1.19 (d, J = 6.5 Hz, 3H);

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.3, 151.9, 130.8, 116.9, 111.1, 110.8, 55.8, 55.7, 41.8, 30.7, 29.7, 29.3, 16.3;

IR (ATR) 2925, 2856, 1726, 1500, 1463, 1225, 1050, 800 cm^{-1}.

HRMS (FAB+) calculated for $C_{14}H_{20}O$ [M+]: 236.1412; found 236.1419.



4-(1-benzyl-1H-indol-5-yl)but-3-yn-1-ol. To a mixture of the known 1-benzyl-5-bromo-1H-indole (1.0 g, 3.91 mmol) in Et_3N (20 mL) was added 3-butyn-1-ol (0.35 mL, 4.68 mmol), $Pd(PPh_3)_2Cl_2$ (82 mg, 0.12 mmol). CuI was added (22 mg, 0.12 mmol) and the mixture was stirred overnight. The reaction was quenched with NH_4Cl, filtered through a pad of celite and was washed twice with Et_2O (~ 10 mL total). The reaction mixture was concentrated in vacuo and flash column chromatographed using 3/97 MeOH/DCM as the mobile phase to afford the 460 mg (43%) of product.

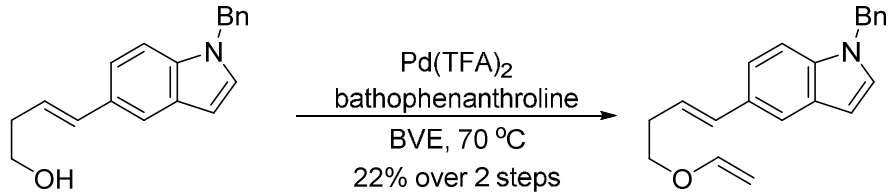
^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.31-7.16 (m, 5H), 7.12 (d, J = 3.0 Hz, 1H), 7.07 (d, J = 6.5 Hz, 1H), 6.50 (d, J = 3.0 Hz, 1H), 5.29 (s, 2H), 3.81 (q, J = 6.0 Hz, 2H), 2.70 (t, J = 6.0 Hz, 2H), 1.89 (s, 1H);

^{13}C NMR (126 MHz, CDCl3) δ 137.2, 135.8, 129.2, 128.8, 128.6, 127.7, 126.7, 125.4, 124.8, 114.2, 109.7, 101.9, 83.9, 83.4, 61.4, 50.2, 24.0.

IR (ATR) 3359, 2921, 1481, 1328, 1045, 728 cm^{-1};

HRMS (FAB) *m/z* calcd $C_{19}H_{17}ON$: 275.1310, found: 275.1316.

(E)-4-(1-benzyl-1H-indol-5-yl)but-3-en-1-ol. Lithium aluminum hydride (66 mg, 1.74 mmol) was added to a pre-dried round bottom flask under argon. 6 mL of dry THF was added and 4-(1-benzyl-1H-indol-5-yl)but-3-yn-1-ol was added in dropwise (200 mg, 0.73 mmol). The reaction was refluxed overnight. The reaction was cooled to 0°C and 0.1 mL of water was added, followed by 0.1 mL of 15% NaOH/H_2O, then 0.2 mL of H_2O. Magnesium sulfate was added to the mixture and allowed to stir. The solution was filtered through a fritted glass funnel, concentrated, and purified by flash column chromatography using 2/98 MeOH/DCM as the eluent to afford the crude product, which was used immediately in the next step.



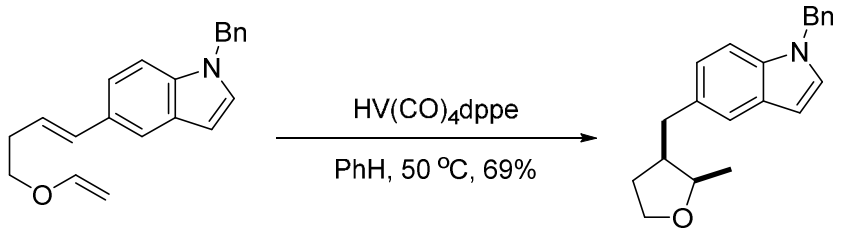
(E)-1-benzyl-5-(4-(vinyloxy)but-1-en-1-yl)-1H-indole. Bathophenanthroline (1 mg, 0.003 mmol) and $Pd(CF_3CO_2)_2$ (1 mg, 0.003 mmol) were added to butyl vinyl ether (1.4 mL, 10.8 mmol). The crude (E)-4-(1-benzyl-1H-indol-5-yl)but-3-en-1-ol (0.73 mmol) was added, and the mixture was stirred at 70°C overnight. The crude mixture was purified by flash column chromatography using 50/50 toluene/hexanes as an eluent to afford 48 mg (22% over two steps) of the vinyl ether.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, J = 1.6 Hz, 1H), 7.32 – 7.15 (m, 6H), 7.09 (m, 3H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.52-6.47 (m, 2H), 6.15 (dt, *J* = 16.0, 7.0 Hz, 1H), 5.29 (s, 2H), 4.21 (dd, *J* = 14.0, 2.0 Hz, 1H), 4.00 (dd, *J* = 7.0, 2.0 Hz, 1H), 3.80 (t, *J* = 7.0 Hz, 2H), 2.58 (qd, *J* = 7.0, 1.0 Hz, 2H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 151.9, 137.5, 135.9, 133.1, 129.3, 129.0, 128.8, 128.7, 127.6, 126.7, 123.1, 120.1, 119.0, 109.8, 102.0, 86.5, 67.8, 50.2, 32.9;

IR (ATR) 3028, 2921, 2870, 1614, 1483, 1195, 965, 718 cm^{-1}.

HRMS (FAB) *m/z* calcd $C_{21}H_{21}ON$: 303.1623, found: 303.1612.



10:1 cis:trans

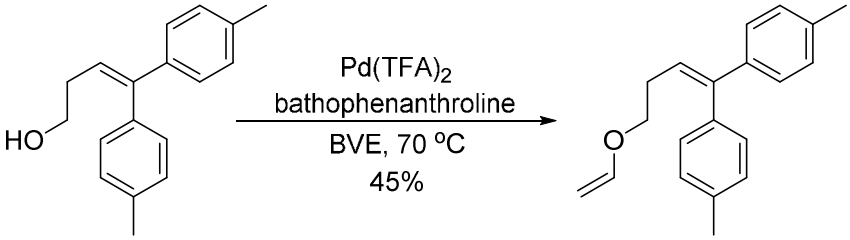
1-benzyl-5-((2-methyltetrahydrofuran-3-yl)methyl)-1H-indole. (E)-1-benzyl-5-(4-(vinyloxy)but-1-en-1-yl)-1H-indole (8.0 mg, 0.026 mmol) and $HV(CO)_4dppe$ (31 mg, 0.055 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 5.5 mg of the product (69%) as a 10:1 ratio of cis:trans.

^{1}H NMR (400 MHz, $CDCl_3$) δ (Major Diastereomer) 7.44 (s, 1H), 7.33 – 7.28 (m, 2H), 7.23 – 7.08 (m, 5H), 7.00 (dd, J = 8.0, 1.6 Hz, 1H), 6.51 – 6.46 (d, J = 3.2 Hz, 1H), 5.30 (s, 2H), 4.12 (p, J = 6.4 Hz, 1H), 3.96 (td, J = 8.0, 4.8 Hz, 1H), 3.69 (q, J = 8.0 Hz, 1H), 2.93 – 2.85 (m, 1H), 2.56 – 2.46 (m, 2H), 1.90 – 1.87 (m, 1H), 1.75 – 1.70 (m, 1H), 1.20 (d, J = 6.4 Hz, 3H);

^{13}C NMR (126 MHz, $CDCl_3$) δ 137.6, 135.0, 132.1, 128.9, 128.7, 128.4, 127.6, 126.8, 122.9, 120.5, 109.5, 101.2, 66.4, 50.2, 44.0, 35.0, 30.8, 29.7, 16.4;

IR (ATR) 2925, 2856, 1722, 1485, 1446, 1355, 1182, 1073, 795, 721 cm^{-1}.

HRMS (FAB+) calculated for $C_{21}H_{23}ON$ [M+]: 305.1780; found 305.1783.



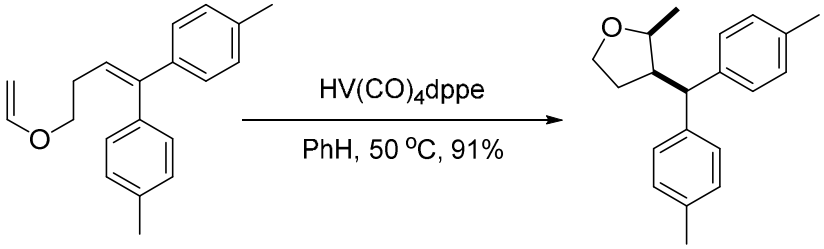
4,4'-(4-(vinyloxy)but-1-ene-1,1-diyl)bis(methylbenzene). Bathophenanthroline (1 mg, 0.003 mmol) and $Pd(CF_3CO_2)_2$ (1 mg, 0.003 mmol) were added to butyl vinyl ether (1.0 mL, 7.92 mmol). 4,4-di-p-tolylbut-3-en-1-ol (100 mg, 0.40 mmol) was added, and the mixture was stirred at 70°C for three days. The crude mixture was purified by flash column chromatography using 50/50 toluene/hexanes as an eluent to afford 50 mg (45%) of the vinyl ether.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (d, J = 8.0 Hz, 2H), 7.13 – 7.09 (m, 2H), 7.09 – 7.03 (m, 4H), 6.45 (dd, J = 14.0, 7.0 Hz, 1H), 6.05 (t, J = 7.0 Hz, 1H), 4.16 (dt, J = 14.0, 1.5 Hz, 1H), 3.97 (dt, J = 7.0, 1.5 Hz, 1H), 3.73 (t, J = 7.0 Hz, 2H), 2.47 (q, J = 7.0 Hz, 2H), 2.37 (s, 3H), 2.31 (s, 3H);

^{13}C NMR (126 MHz, $CDCl_3$) δ 151.8, 143.6, 139.8, 137.0, 136.8, 136.6, 129.7, 128.9, 128.8, 127.2, 123.8, 86.4, 67.8, 29.8, 21.2, 21.1;

IR (ATR) 3023, 2921, 1613, 1511, 1196, 618 cm^{-1};

HRMS (FAD) *m/z* calcd $C_{20}H_{22}O$: 278.1671 found: 278.1672.



12:1 cis:trans

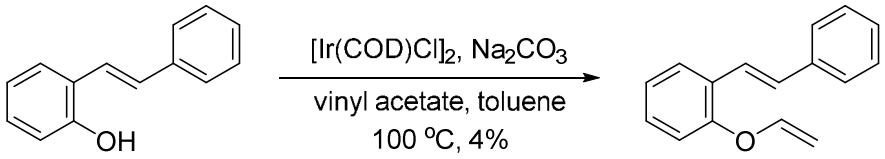
3-(di-p-tolylmethyl)-2-methyltetrahydrofuran. 4,4'-(4-(vinyloxy)but-1-ene-1,1-diyl)bis(methylbenzene) (8.0 mg, 0.028 mmol) and $HV(CO)_4$dppe (34 mg, 0.060 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 7.3 mg of the product (91%) as a 12:1 ratio of cis:trans.

^{1}H NMR ($CDCl_3$, 500 MHz) δ Major Diasteromer: 7.23 (d, J = 8.0 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 7.06 (dd, J = 15.0, 8.0 Hz, 4H), 4.19 (p, J = 6.5 Hz, 1H), 3.94 (dt, J = 8.0, 6.0 Hz, 1H), 3.73 (t, J = 8.0 Hz, 1H), 3.64 (m, 1H), 3.13 (m, 1H), 2.28 (s, 3H), 2.27 (s, 3H), 1.68 (qd, J = 6.5, 1.5 Hz, 2H), 0.94 (d, J = 6.5 Hz, 3H).

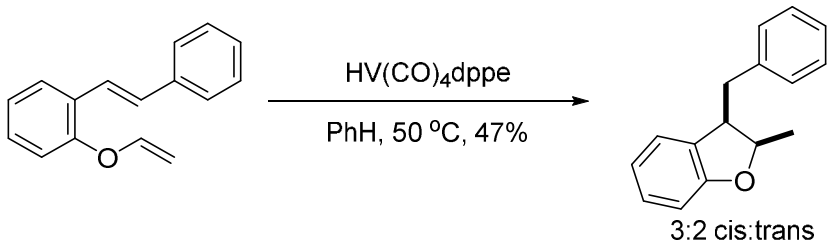
^{13}C NMR ($CDCl_3$, 126 MHz) δ 142.2, 140.7, 135.8, 135.7, 129.3, 129.2, 127.5, 127.3, 75.7, 66.4, 51.8, 46.6, 30.4, 21.0, 20.9, 16.1.

IR (ATR) 3020, 2968, 2925, 2869, 1727, 1511, 1454, 1376, 1110, 1061, 1034, 863, 805, 765 cm^{-1}.

HRMS (FAB+) calculated for $C_{20}H_{23}O$ [M+]: 279.1749; found 279.1741.

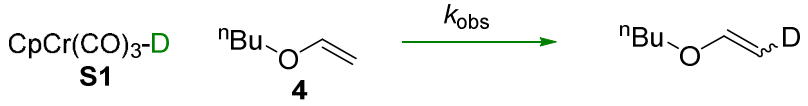


(E)-1-styryl-2-(vinyloxy)benzene. A solution of $[Ir(COD)Cl]_2$ (3 mg, 0.0051 mmol) and Na_2CO_3 (32 mg, 0.31 mmol) was suspended in toluene (1 mL). Vinyl acetate (93 µL, 1.02 mmol) and the (E)-2-styrylphenol (100 mg, 0.51 mmol) were added and the mixture was brought to 100 °C for 2 hours. The reaction was quenched with wet ether, concentrated, and subjected to column chromatography (50/50 toluene/hexanes) to afford 5 mg of the product (4%).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.55 – 7.49 (m, 2H), 7.40 (d, *J* = 16.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.25 – 7.16 (m, 2H), 7.15 – 7.07 (m, 2H), 6.98 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.63 (dd, *J* = 14.0, 6.0 Hz, 1H), 4.71 (dd, *J* = 14.0, 2.0 Hz, 1H), 4.42 (dd, *J* = 6.0, 2.0 Hz, 1H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 154.0, 149.1, 137.7, 130.0, 128.7, 128.7, 128.2, 127.7, 126.7, 126.5, 123.8, 122.7, 117.7, 94.8, 29.8;
IR (ATR) 3026, 2920, 2850, 1639, 1481, 1452, 1231, 961, 690 cm^{-1};
HRMS (FAB) *m/z* calcd $C_{16}H_{14}O$: 222.1045, found: 222.1037.



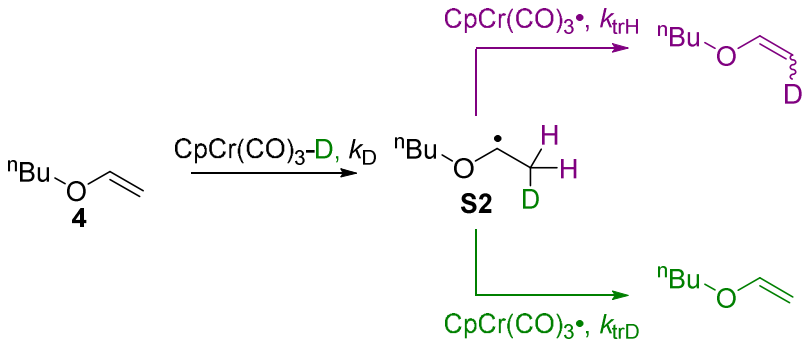
3-benzyl-2-methyl-2,3-dihydrobenzofuran. (E)-1-styryl-2-(vinyloxy)benzene (8.0 mg, 0.036 mmol) and $HV(CO)_4$dppe (43 mg, 0.076 mmol) were added to a J-Young tube and dissolved in benzene (1 mL). The resultant yellow solution was placed in a 50°C oil bath, and was left there for three days with occasional shaking. After three days the resultant brown solution was filted through a pad of silica and the tube was washed 3x with methylene chloride. The crude product was subjected to microscale flash column chromatography to afford 3.8 mg of the product (47%) as an inseparable mixture (~3:2 ratio of cis:trans).
^{1}H NMR (400 MHz, $CDCl_3$) δ (Both Diastereomers) 7.35 – 7.30 (m, 3H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.16 – 7.06 (m, 4H), 6.97 (dd, *J* = 18.4, 6.8 Hz, 2H), 6.84 – 6.74 (m, 2H), 6.67 (t, *J* = 7.2 Hz, 1H), 6.47 (d, *J* = 7.6 Hz, 1H), 3.12 – 3.00 (m, 2H),; (Major Diastereomer): 4.93 (p, *J* = 6.8 Hz, 1H), 3.61 (q, *J* = 8.0 Hz, 1H), 2.70 (dd, *J* = 13.6, 9.6 Hz, 1H), 1.21 (d, *J* = 6.0 Hz, 3H); (Minor Diastereomer): 4.55 (p, *J* = 6.0 Hz, 1H), 3.33 (q, *J* = 7.2 Hz, 1H), 2.83 (dd, *J* = 13.6, 8.8 Hz, 1H), 1.47 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ (Both Diastereomers)159.2, 159.1, 139.5, 139.0, 131.1, 130.4, 129.4, 129.2, 128.9, 128.7, 128.5, 128.3, 126.6, 126.4, 125.5, 124.7, 120.2, 120.0, 109.7, 109.6, 85.0, 82.6, 50.8, 46.0, 40.9, 35.7, 29.8, 15.9; IR (ATR) 2923, 2852, 1596, 1477, 1458, 1234, 1045, 748 cm^{-1};
HRMS (FAB) *m/z* calcd $C_{16}H_{16}O$: 222.1201, found: 222.1193.

NMR Kinetic Measurements



All experiments were performed at 500 MHz. A stock solution of $CpCr(CO)_3D$ (**S1**) was prepared with the internal standard (hexamethylcyclotrisiloxane), placed in a J-Young tube, and frozen under Ar. A solution of excess olefin (>10 equiv.) was then added and also frozen under Ar. The tube was allowed to thaw and its contents mixed before it was placed in the NMR probe (which was set at 323 K). Ethylene glycol was used to calibrate the temperature controller.

The height of the hydride peak (relative to the internal standard) was measured over time. Generally 4 pulses were used with 3 dummy pulses at each kinetic point, with 120 seconds between the 4 pulses. The kinetic data was fit to an exponential and the rate constant and infinity point were adjusted for a best fit to afford pseudo-first order rate constant $k_{\mathrm{obs}} = 0.0130(3)$.



The k_{obs} only accommodates a fraction of the outcomes from **S2**. Because D• abstraction is not observed, we estimate its contribution using the relation below.

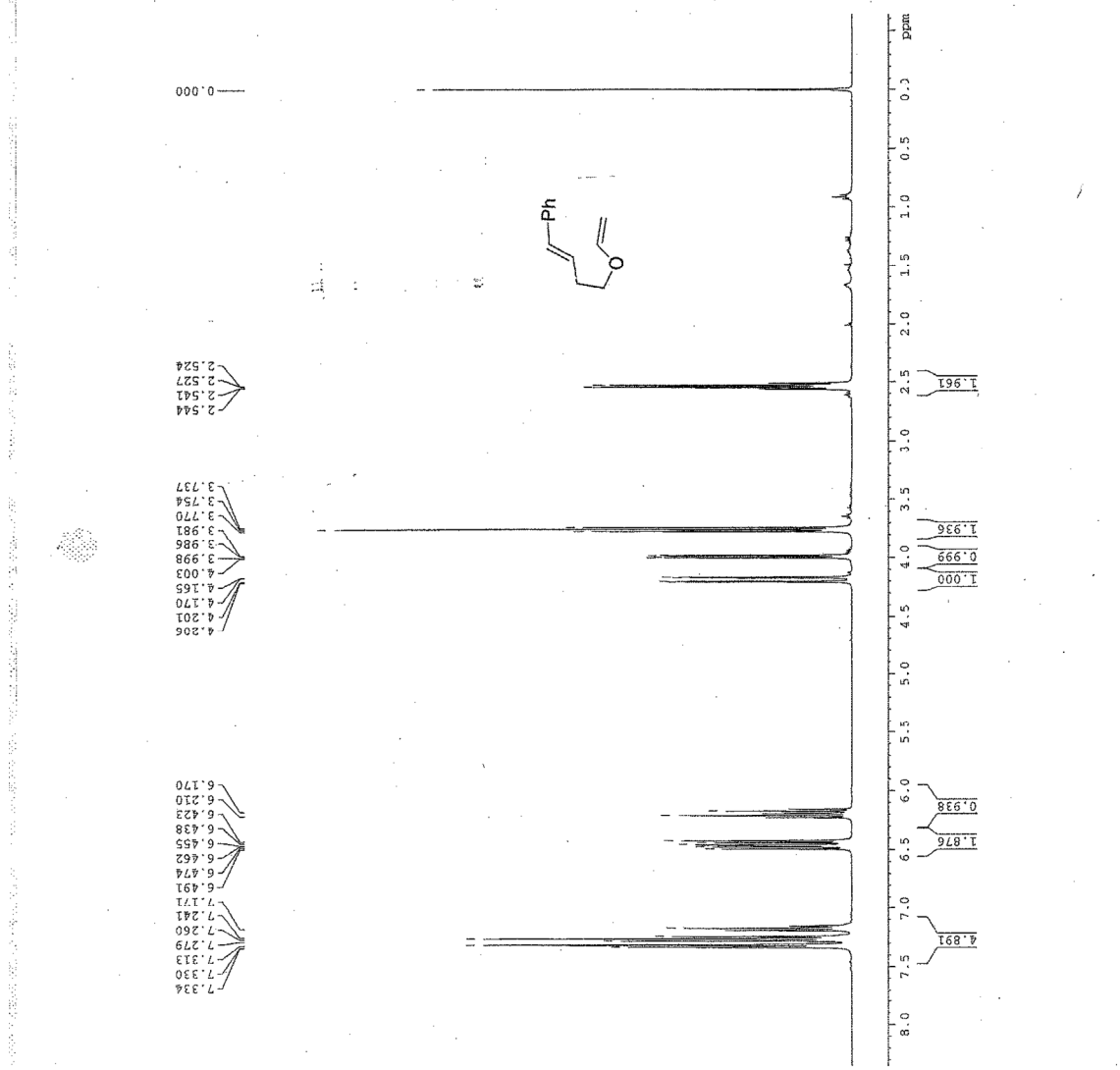
$$k_{\mathrm{obs}} = Sk_{\mathrm{D}}.$$

$$S = \frac{\left(\frac{2}{3}\right) k_{\mathrm{trH}}[\mathrm{Cr}\bullet]}{\left(\frac{2}{3}\right) k_{\mathrm{trH}}[\mathrm{Cr}\bullet] + \left(\frac{1}{3}\right) k_{\mathrm{trD}}[\mathrm{Cr}\bullet]} = \frac{2\frac{k_{\mathrm{trH}}}{k_{\mathrm{trD}}}}{2\frac{k_{\mathrm{trH}}}{k_{\mathrm{trD}}} + 1} \approx \frac{6}{7}$$

We can estimate $k_{\mathrm{trH}}/k_{\mathrm{trD}}$ as 3 as in our previous work.[4] This gives $k_{\mathrm{D}} = 0.0152(1)$. We use the $k_{\mathrm{H}}/k_{\mathrm{D}} = 0.45$ reported by Bullock and coworkers[5], resulting in $k_{\mathrm{H}} = 0.00684(5)$.

References

1. Smith, D. M.; Pulling, M. E.; Norton, J. R. *J. Am. Chem. Soc.* **2007,** *129*, 770.
2. Choi, J.; Pulling, M. E.; Smith, D. M.; Norton, J. R. *J. Am. Chem. Soc.* **2008,** *130*, 4250.
3. Sastry, M. N. V.; Mangelinckx, S.; Lucas, B.; De Kimpe, N. *Synlett.* **2011,** *2011*, 2852.
4. (a) Tang, L.; Papish, E. T.; Abramo, G. P.; Norton, J. R.; Baik, M.-H.; Friesner, R. A.; Rappé, A. *J. Am. Chem. Soc.* **2006,** *128*, 11314. ; (b) Tang, L.; Papish, E. T.; Abramo, G. P.; Norton, J. R.; Baik, M.-H.; Friesner, R. A.; Rappé, A. *J. Am. Chem. Soc.* **2003,** *125*, 10093.
5. Bullock, R. M.; Samsel, E. G. *J. Am. Chem. Soc.* **1990,** *112*, 6886.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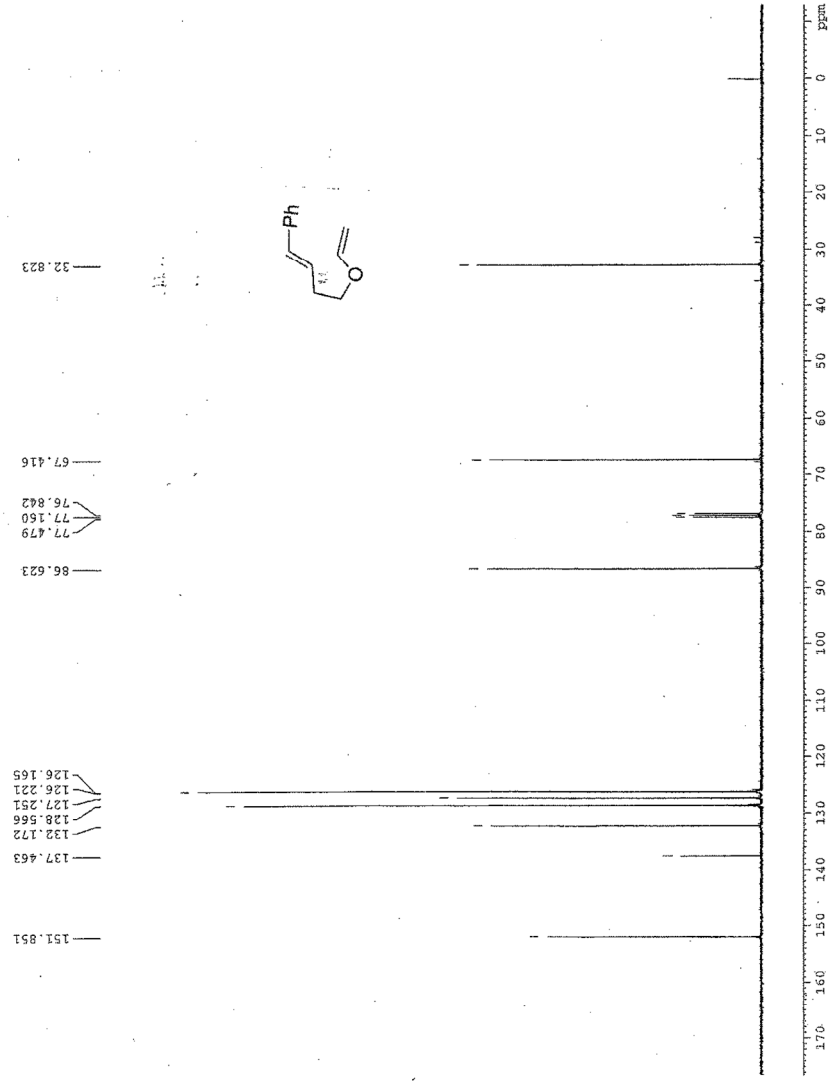
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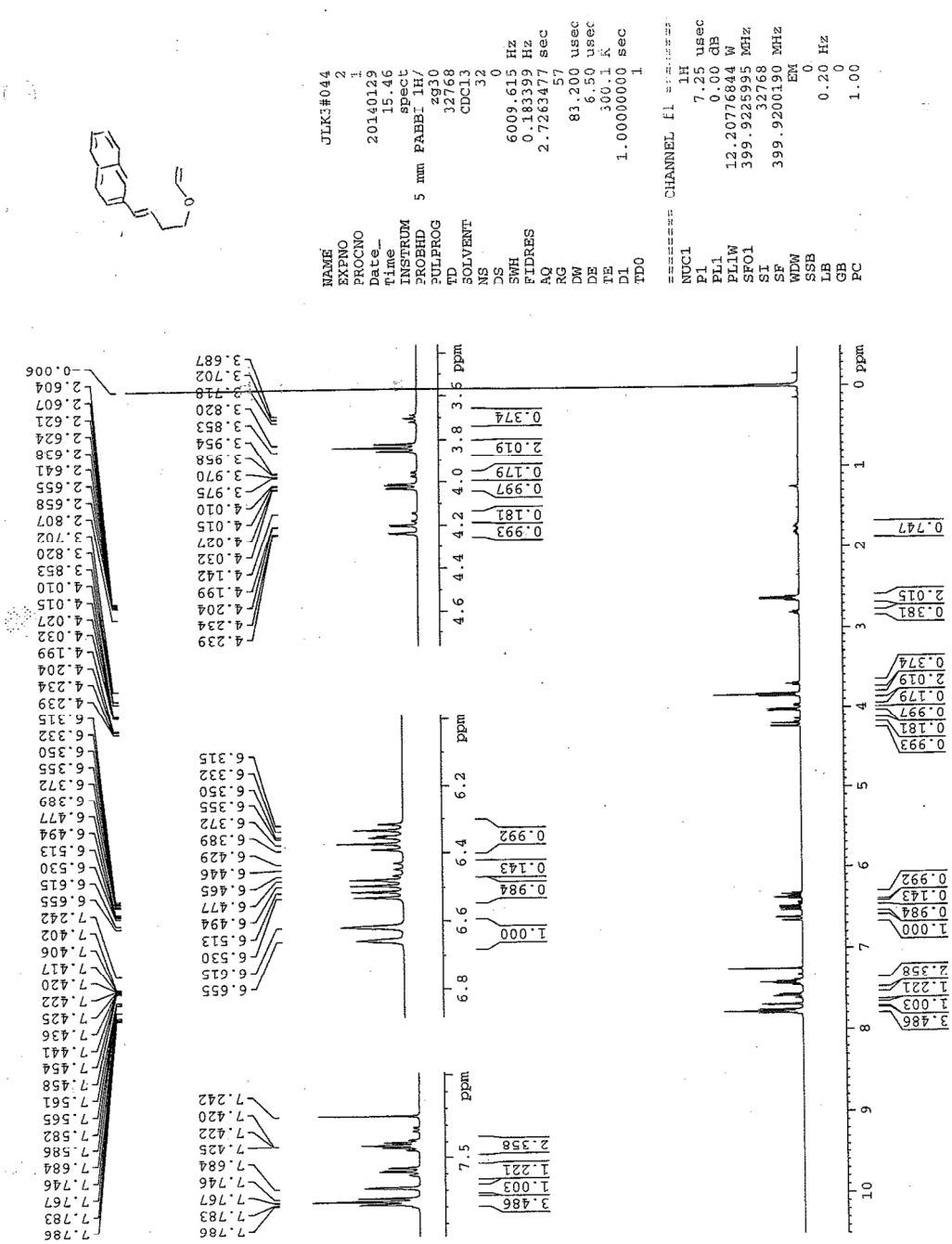

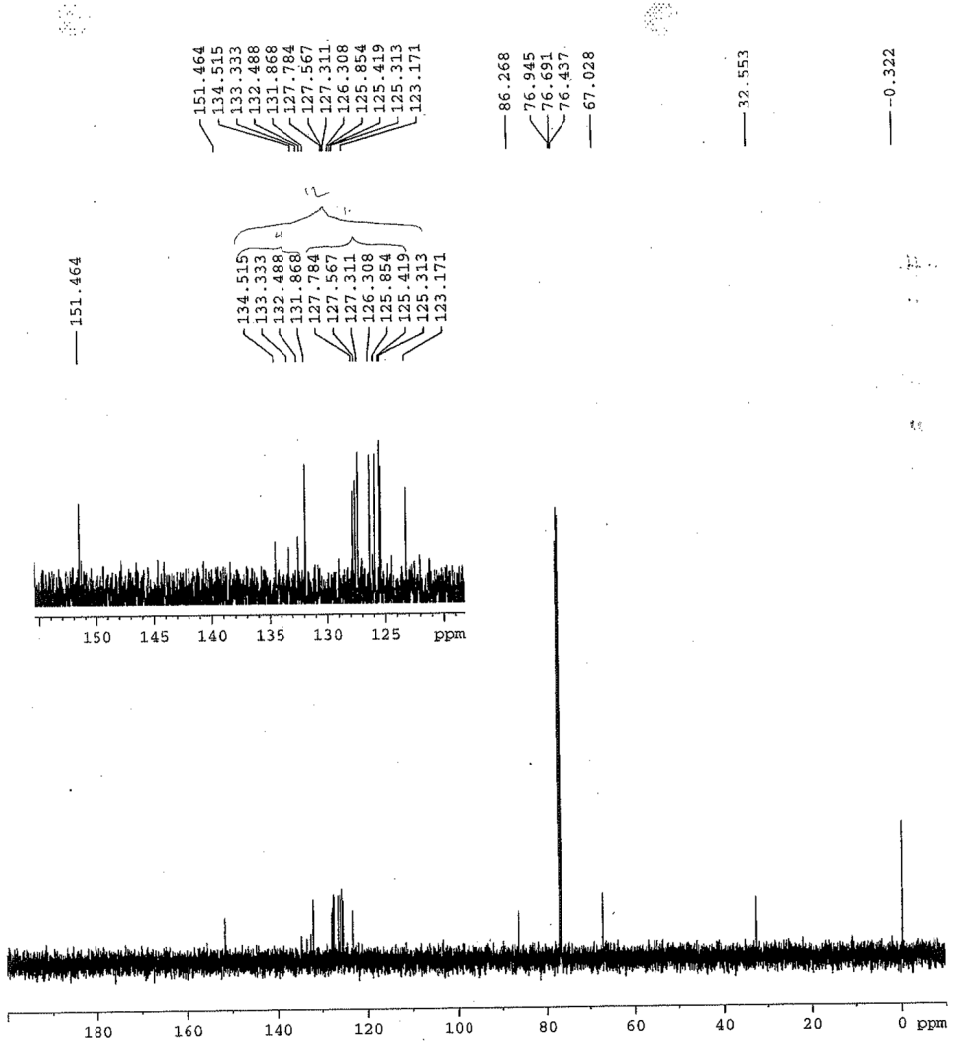

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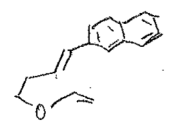

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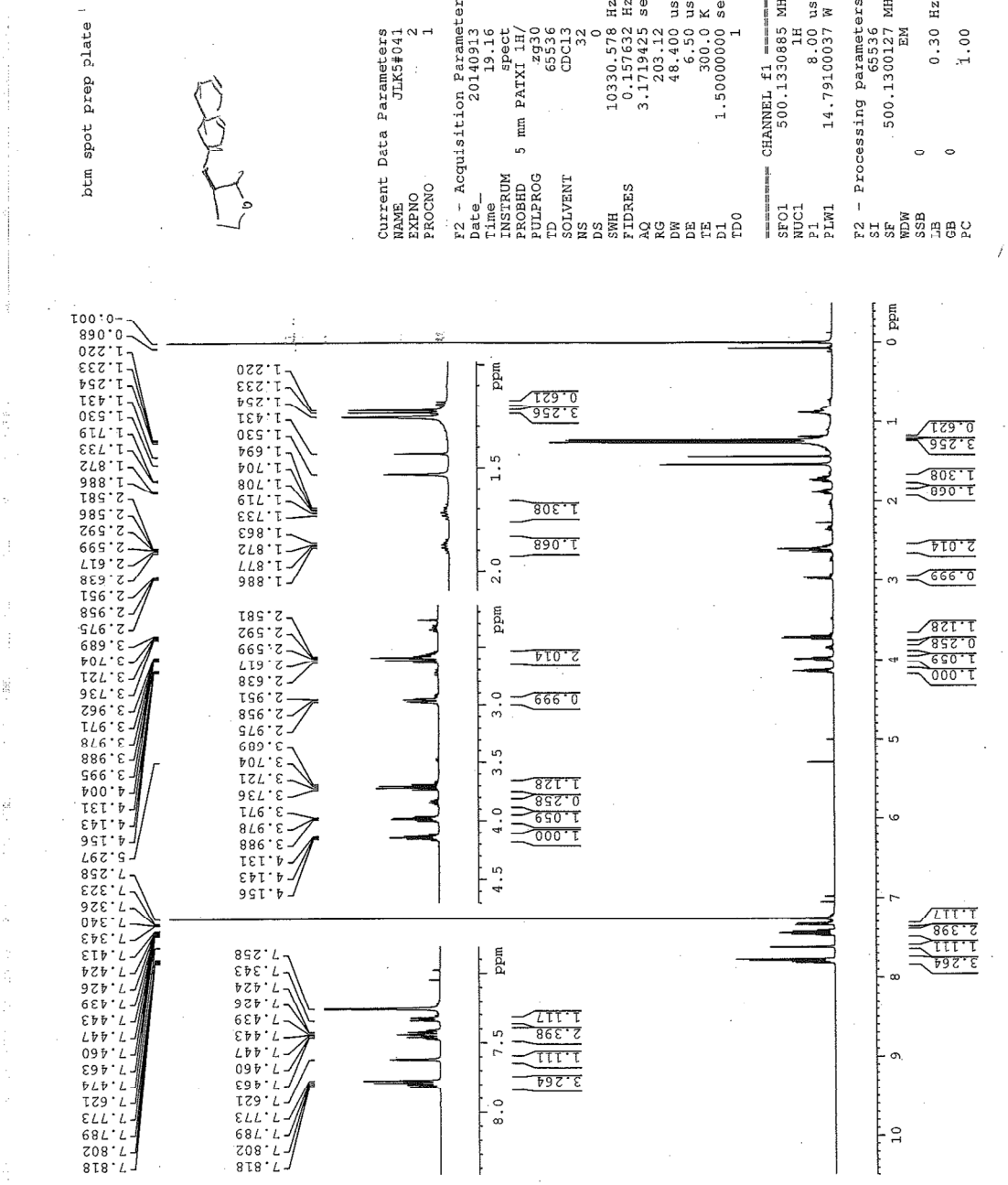
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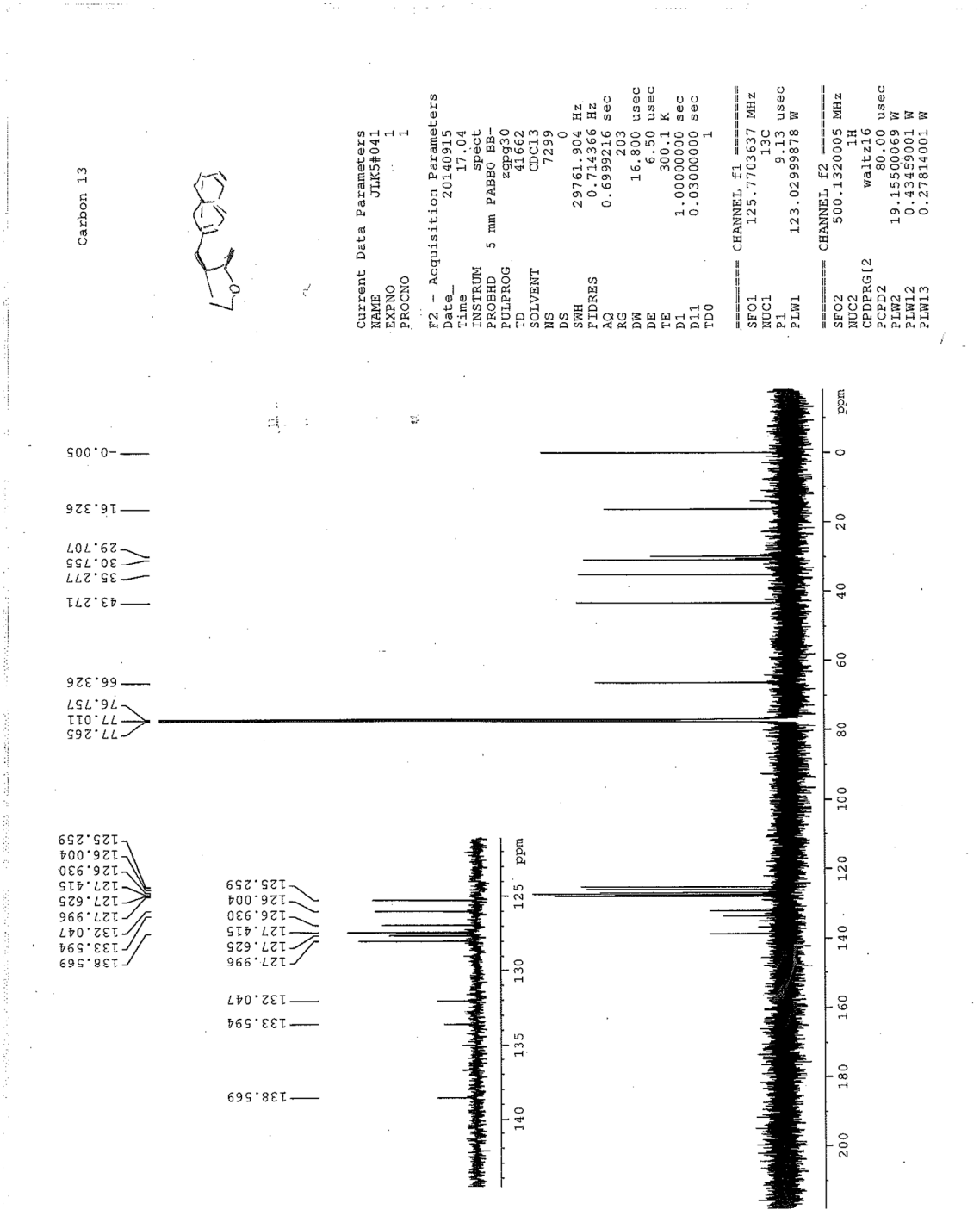
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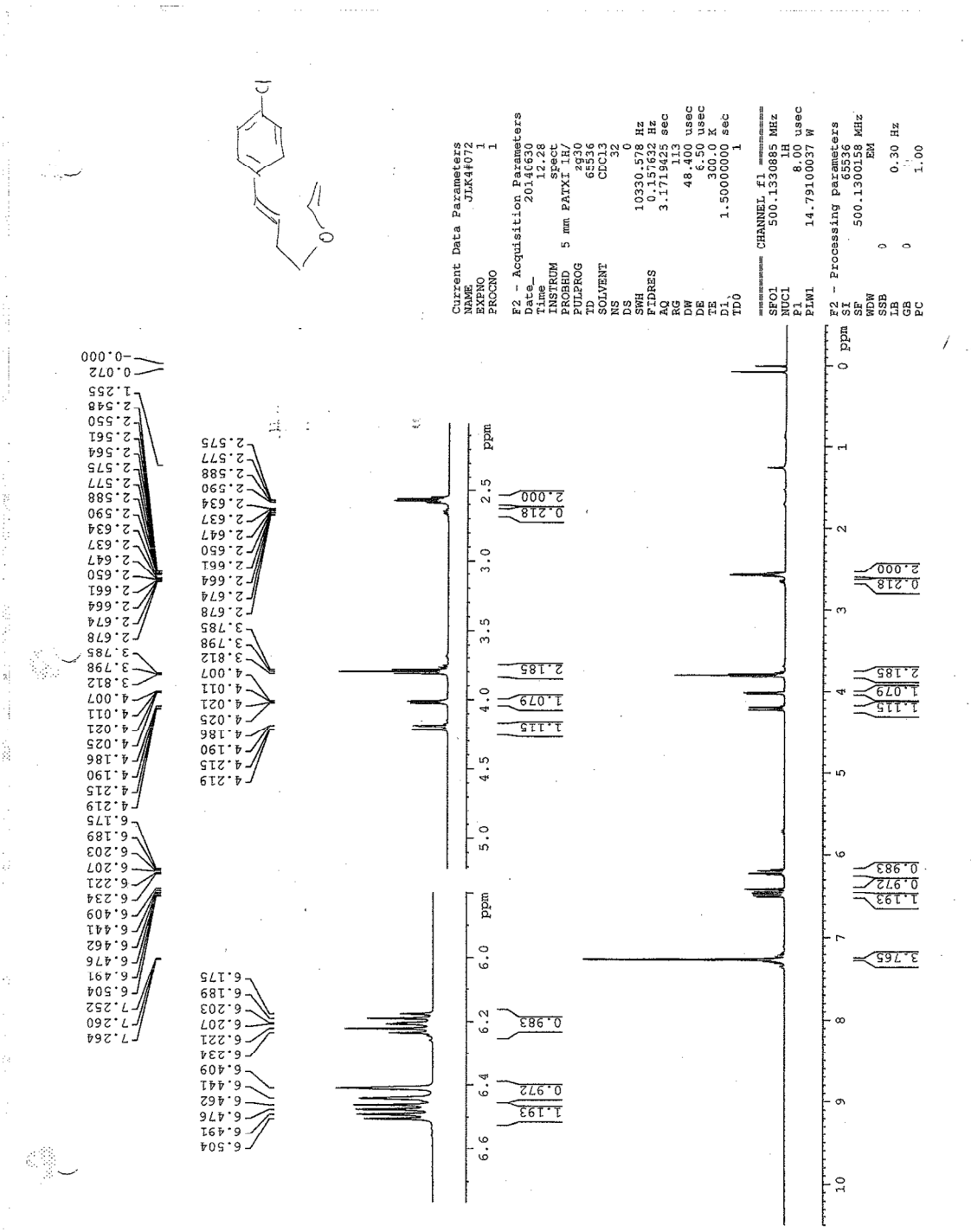

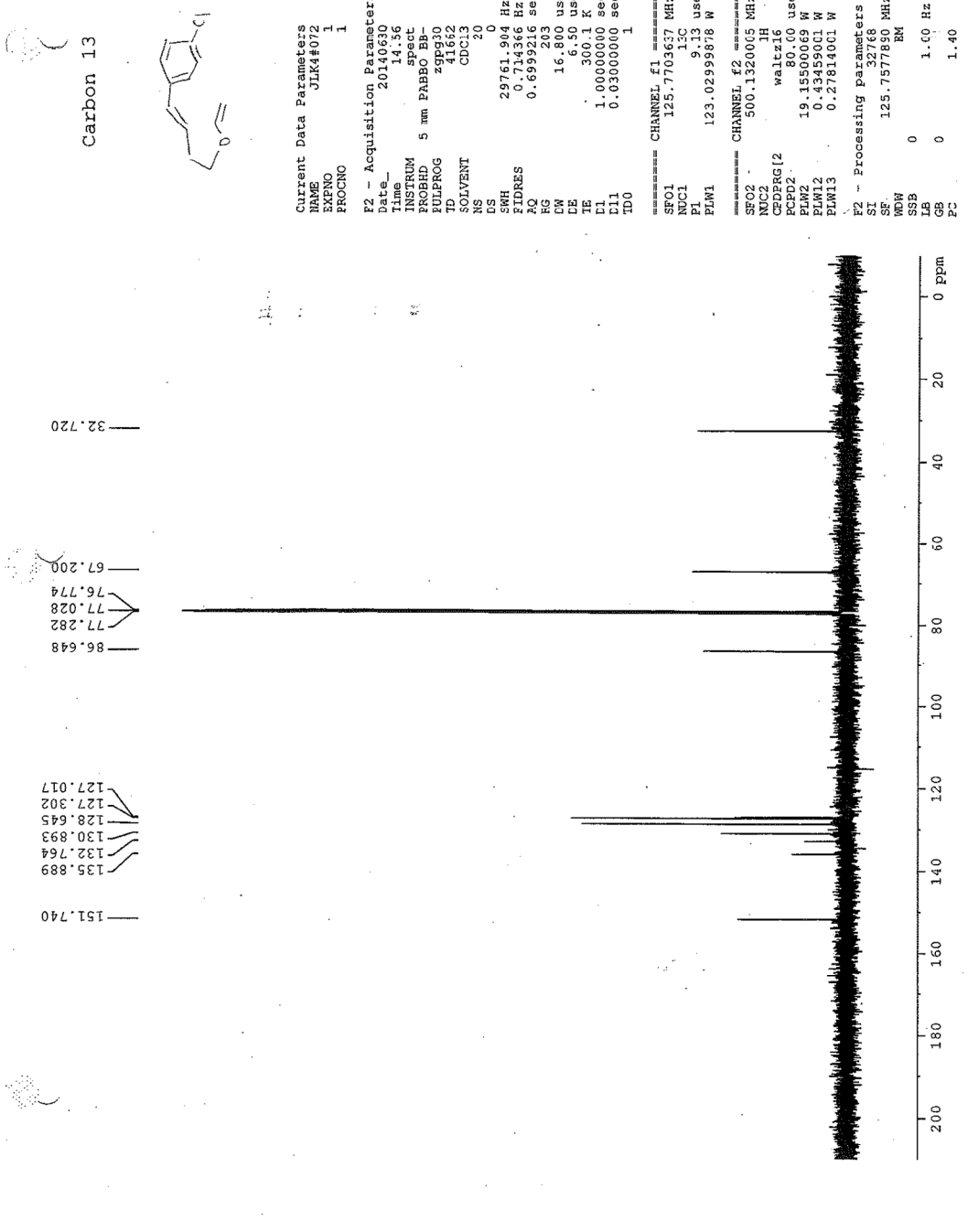
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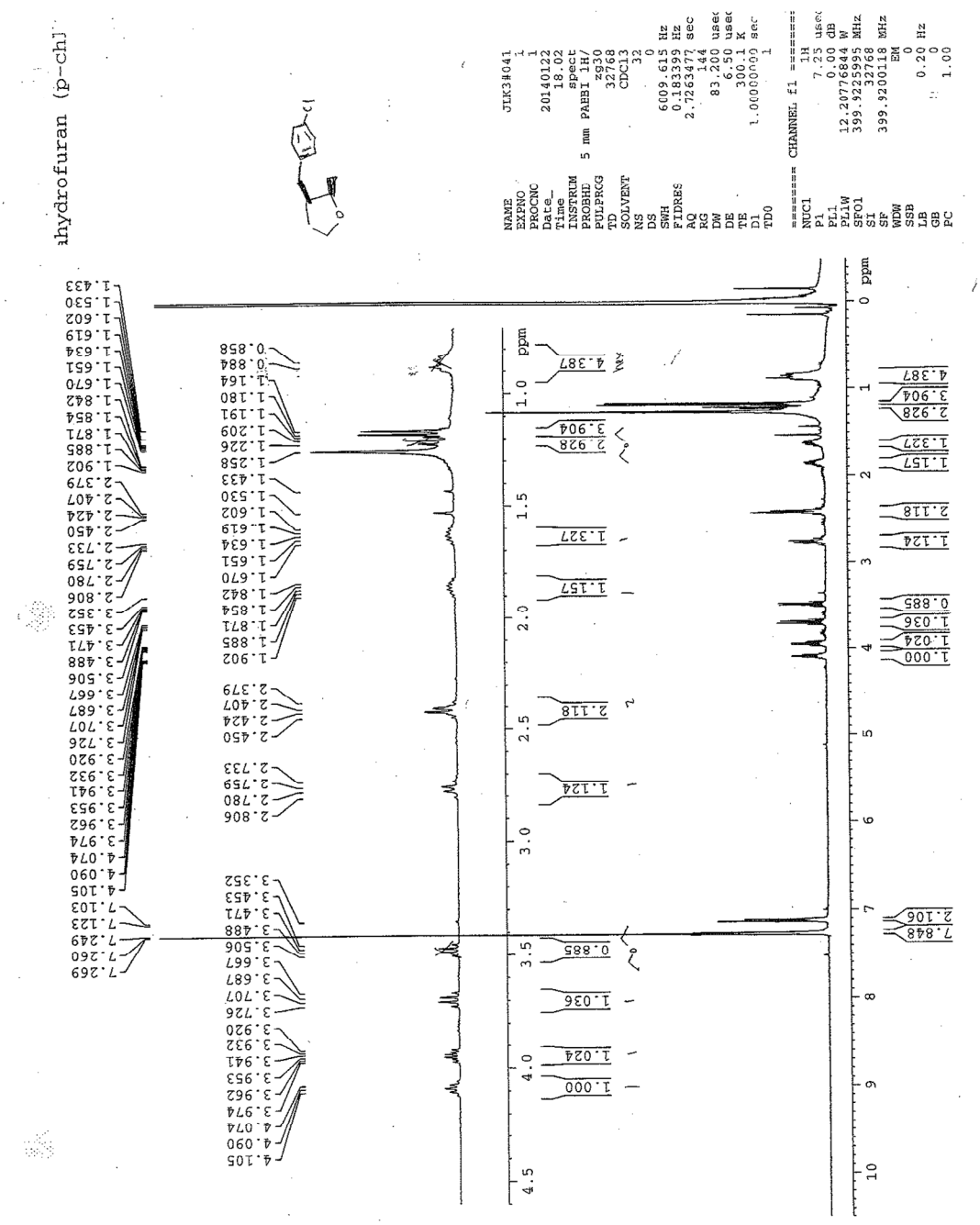
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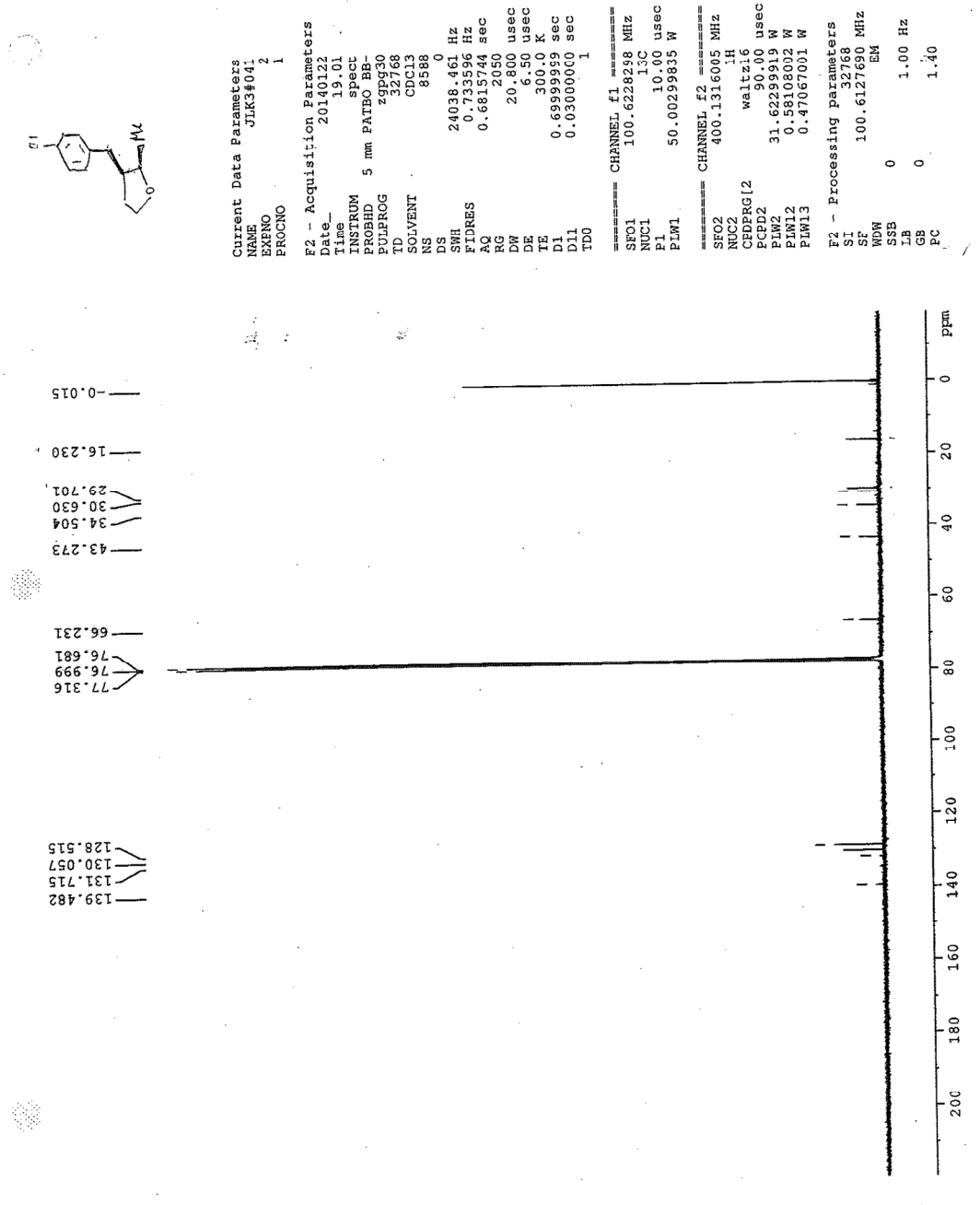
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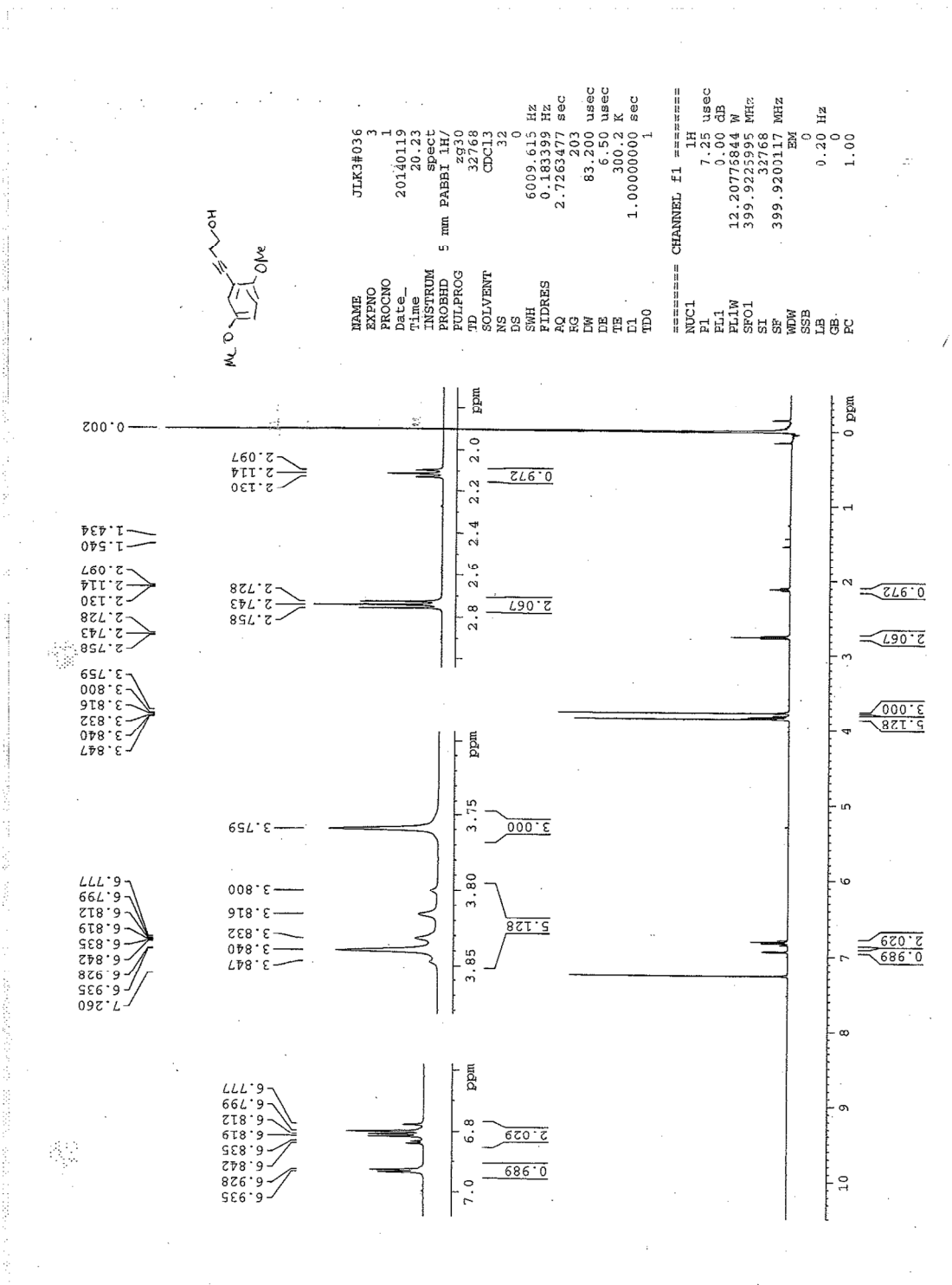
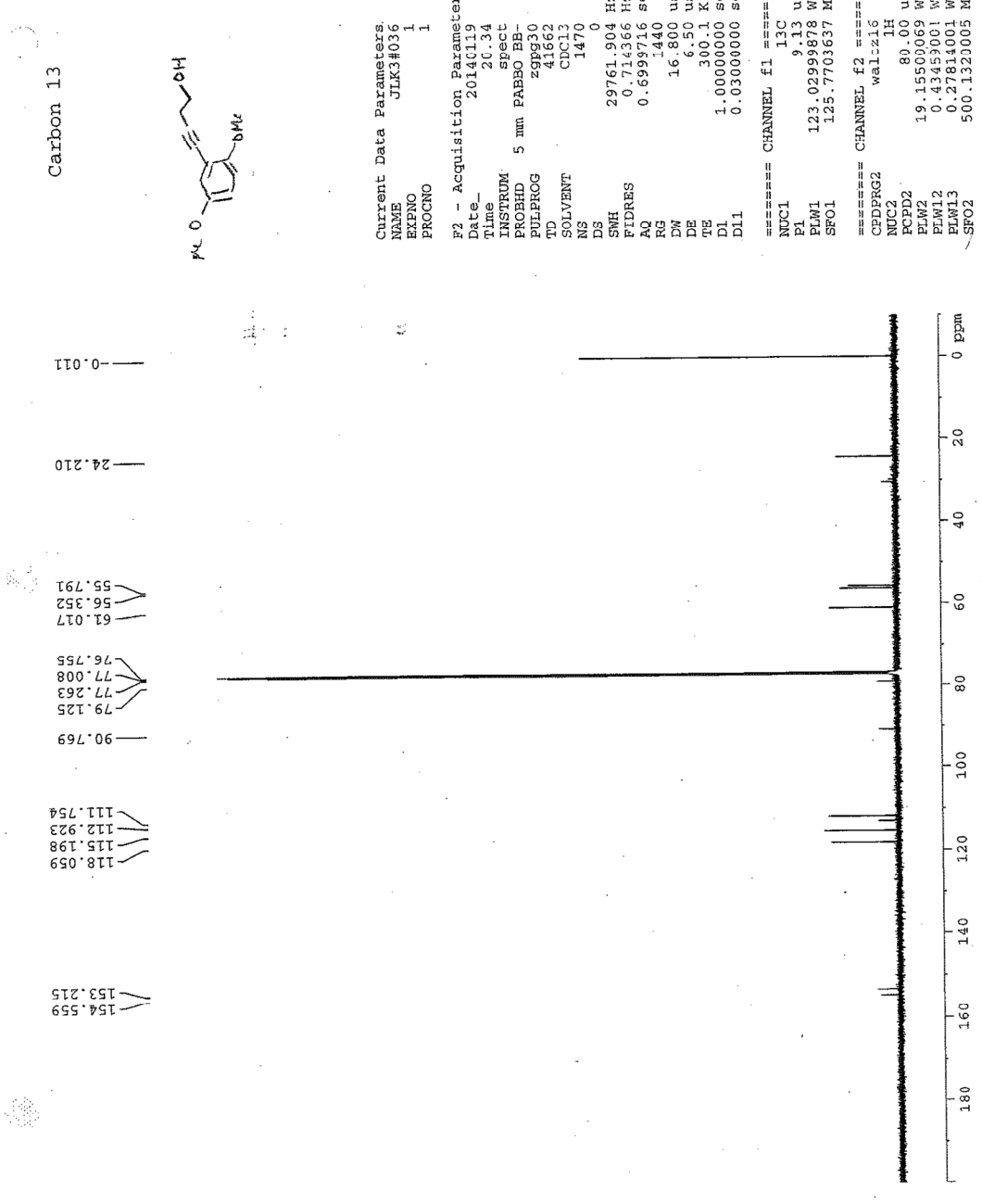
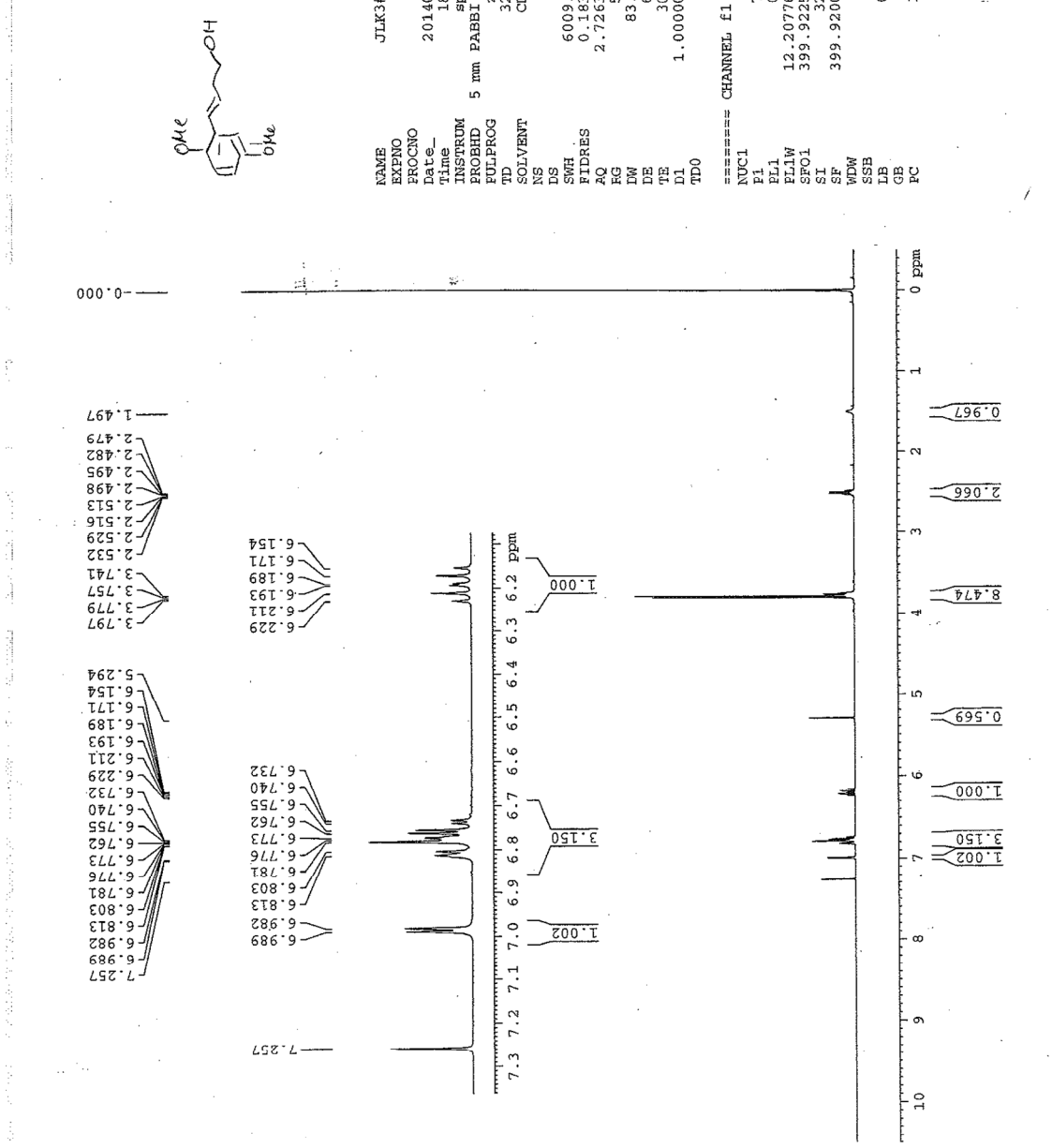
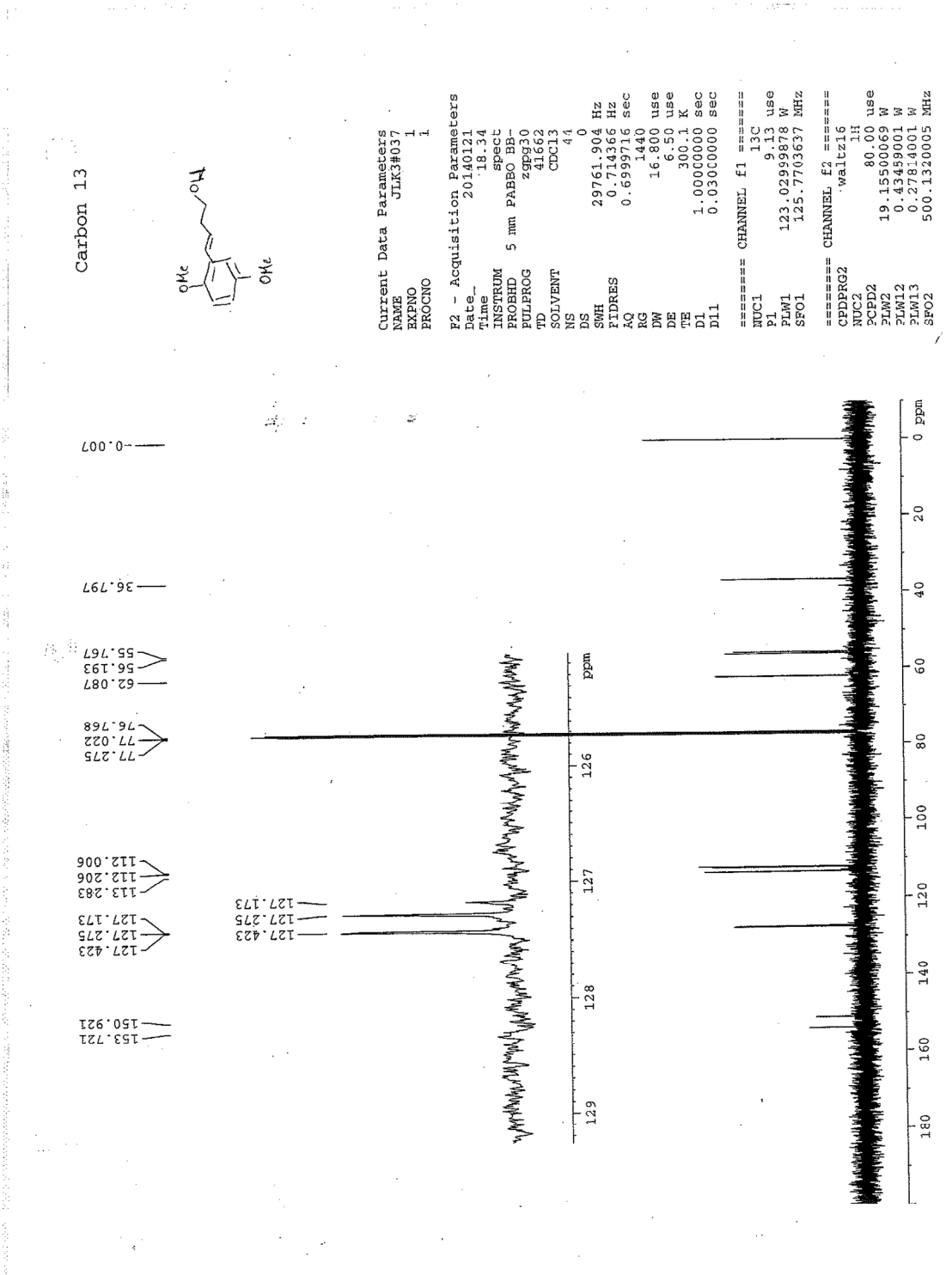
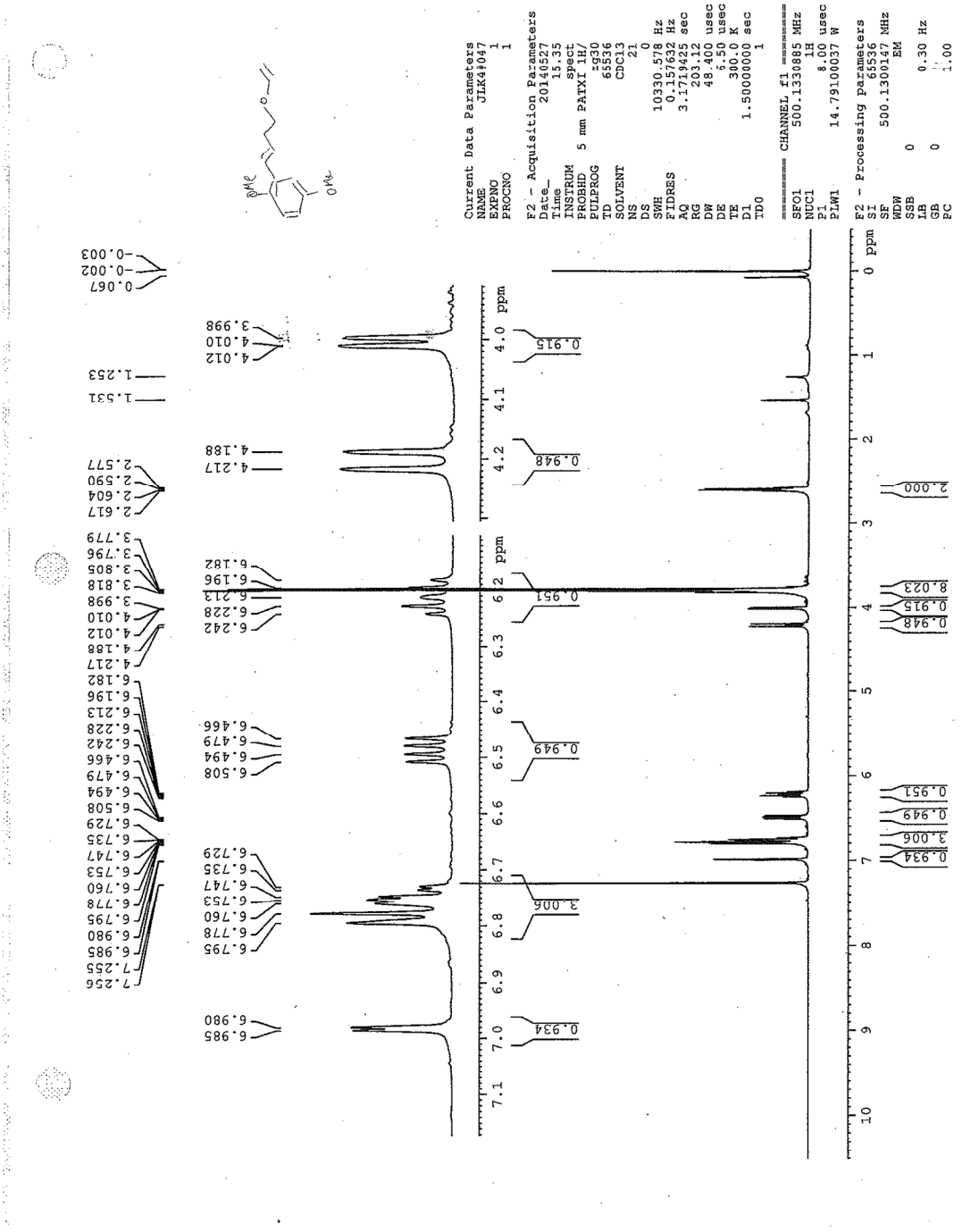
LB 1.00 Hz
GB 0
PC 1.40
-0.015
16.230
29.701
30.630
34.504
43.273
66.231
76.681
76.999
77.316
128.515
130.057
131.715
139.482
ppm

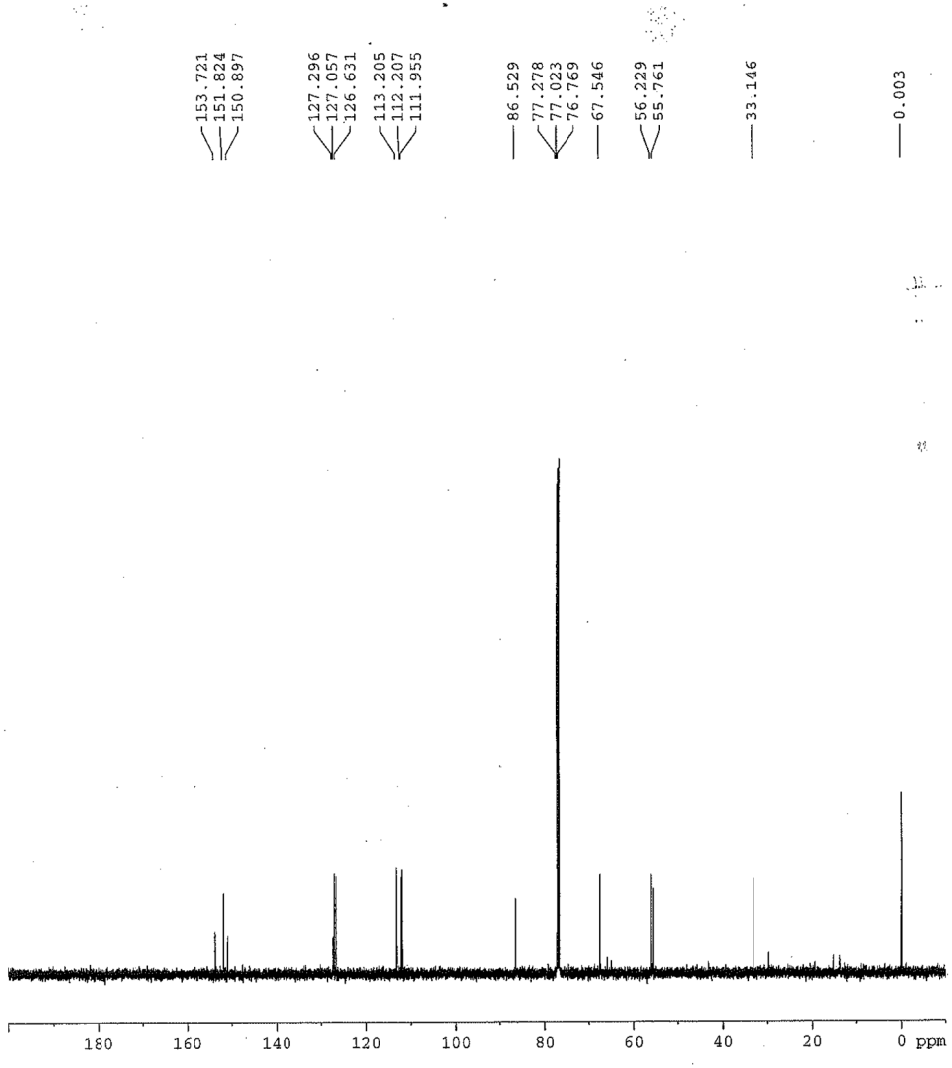



Carbon 13
Current Data Parameters
NAME JLK3#036
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140119
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 41662
SOLVENT CDCl3
NS 1470
DS 0
SWH 29761.904 Hz
FIDRES 0.714366 Hz
AQ 0.69997716 sec
RG 1440
DW 16.800 use
DE 6.50 use
TE 300.1 K
D1 1.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.13 use
PLW1 123.02998878 W
SFO1 125.7703637 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PLW2 19.15500069 W
PLW12 0.43453001 W
PLW13 0.27814001 W
SFO2 500.1320005 MHz
-0.011
24.210
55.791
56.352
61.017
76.755
77.008
77.263
79.125
90.769
111.754
112.923
115.198
118.059
153.215
154.559
0 ppm
20
40
60
80
100
120
140
160
180

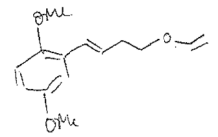

OMe
OMe
OH
NAME JLK3#037
EXPNO 1
PROCNO 1
Date_ 20140121
Time 18.25
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 50.8
DW 83.200 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.25 usec
PL1 0.00 dB
PL1W 12.20776844 W
SFO1 399.9225995 MHz
SI 32768
SF 399.9200128 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
ppm
0.967
2.066
8.474
0.569
1.000
3.150
1.002


Carbon 13
OMe
OH
OMe
Current Data Parameters
NAME JLK3#037
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140121
Time 18.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 41662
SOLVENT CDCl3
NS 44
DS 0
SWH 29761.904 Hz
FIDRES 0.714366 Hz
AQ 0.6999716 sec
RG 1440
DW 16.800 use
DE 6.50 use
TE 300.1 K
D1 1.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.13 use
PLW1 123.02999878 W
SFO1 125.7703637 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PLW2 19.15500069 W
PLW12 0.43459001 W
PLW13 0.27814001 W
SFO2 500.1320005 MHz
153.721
150.921
127.423
127.275
127.173
113.283
112.206
112.006
77.275
77.022
76.768
62.087
56.193
55.767
36.797
0.007
127.423
127.275
127.173
ppm
129
128
127
126
180
160
140
120
100
80
60
40
20
0 ppm

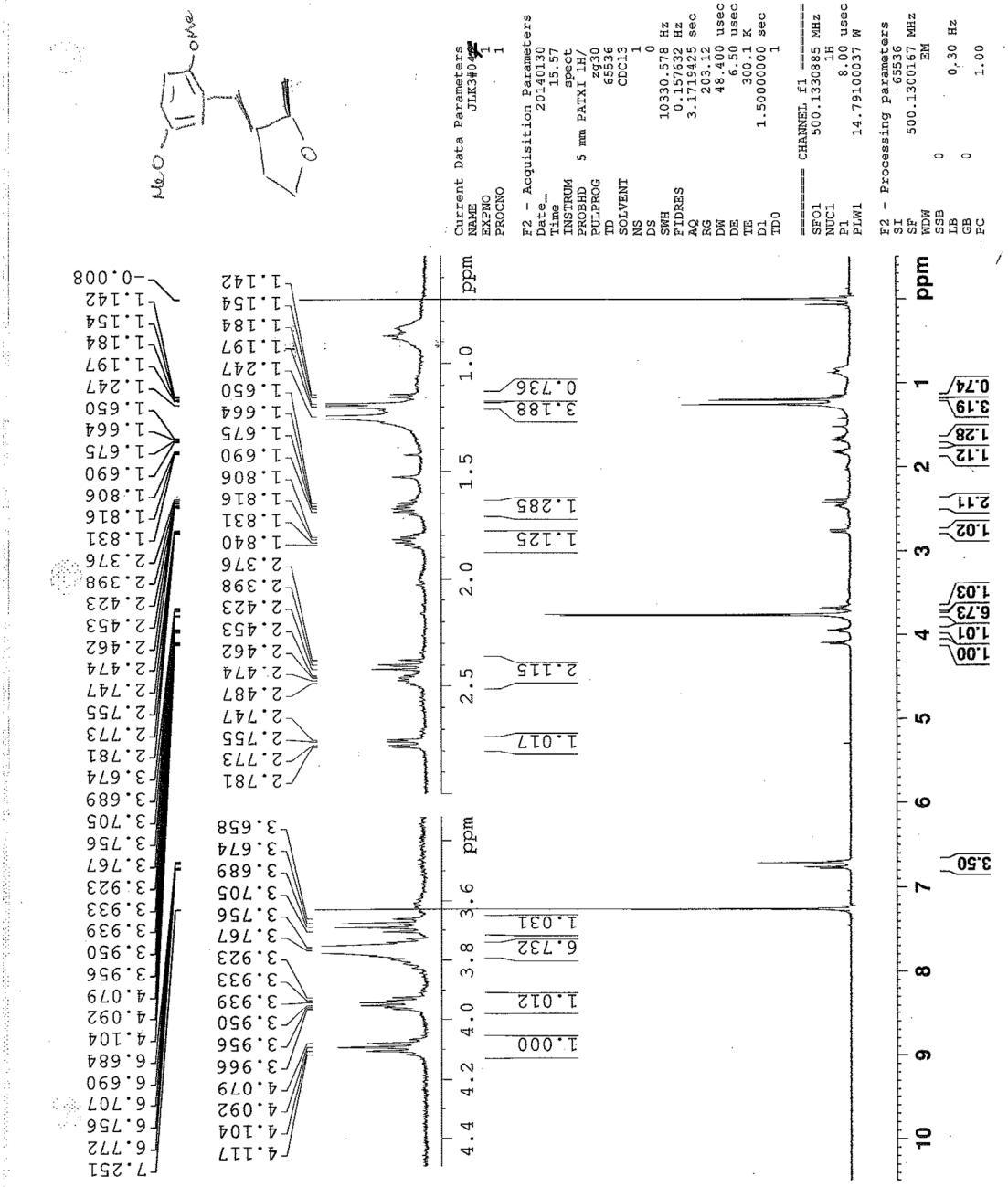


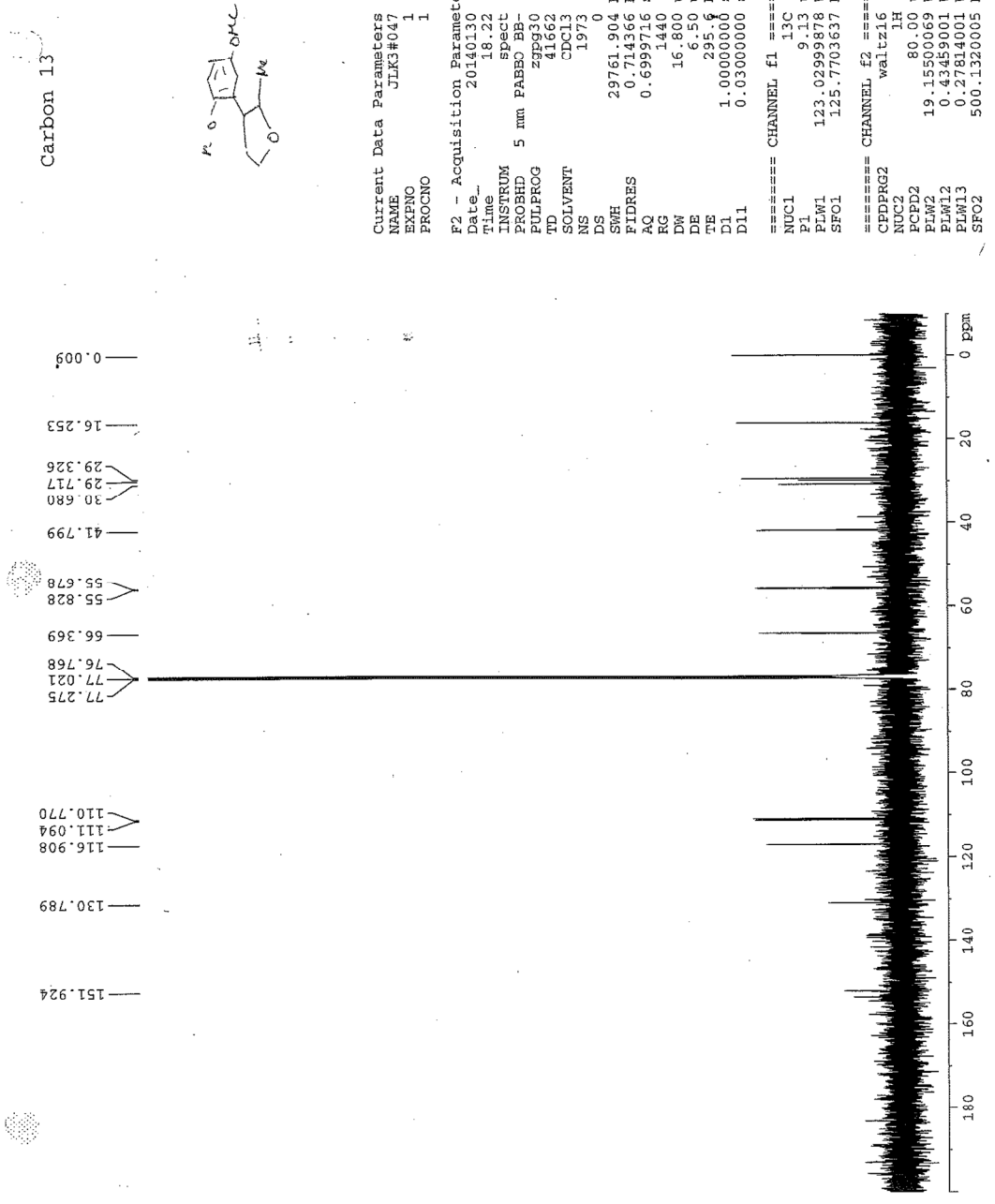


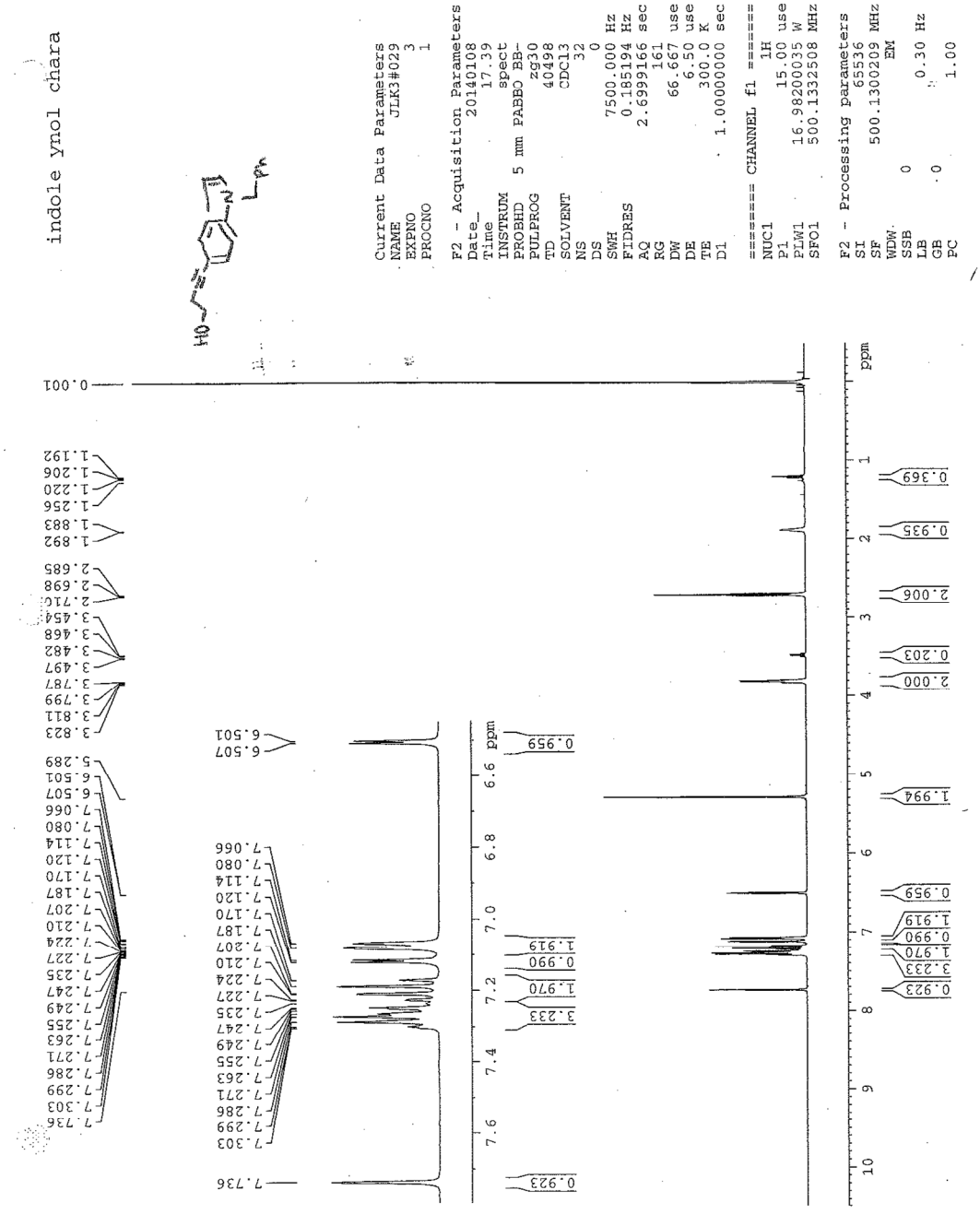
Carbon 13

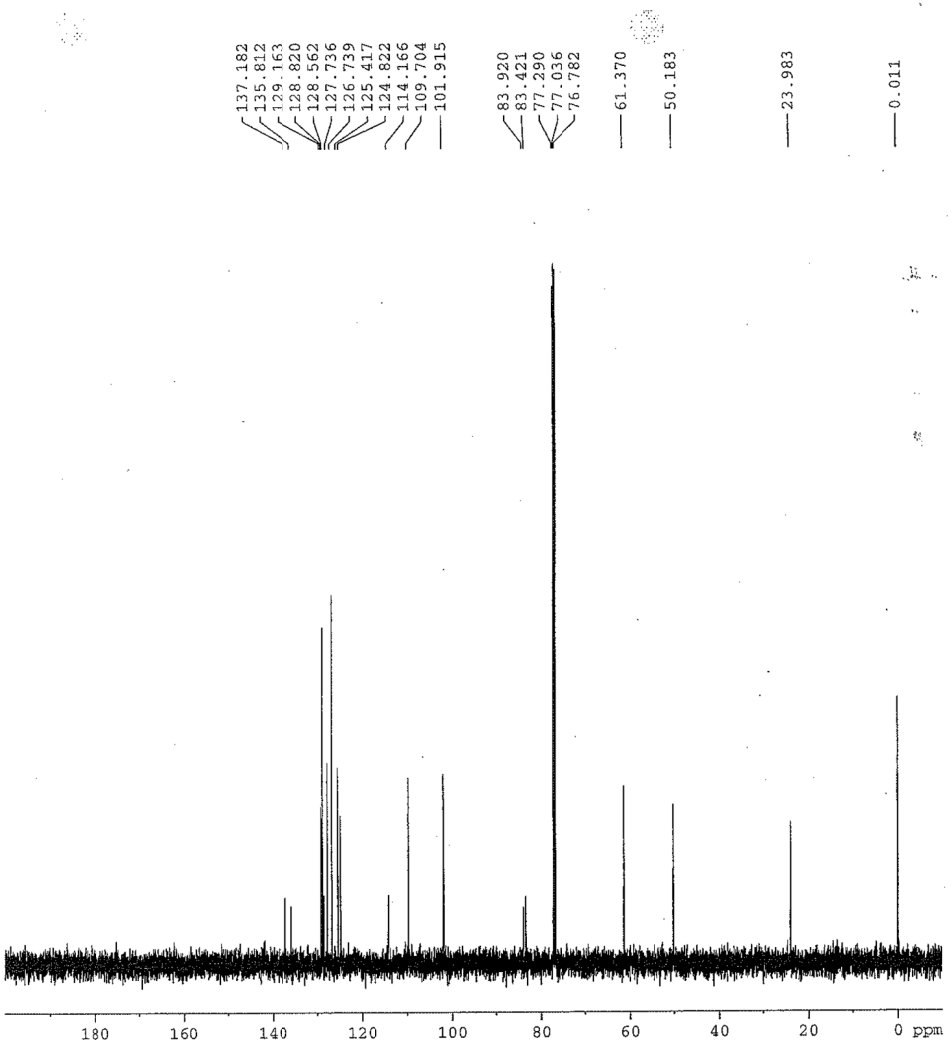



```
Current Data Parameters
NAME                  JLK3#053
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20140208
Time                 17.04
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   41662
SOLVENT              CDCl3
NS                     167
DS                       0
SWH              29761.904 Hz
FIDRES           0.714366 Hz
AQ               0.6999716 sec
RG                     203
DW                  15.800 use
DE                   6.50 use
TE                  300.0 K
D1             1.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 =======
NUC1                   13C
P1                    9.13 use
PLW1         123.02999878 W
SFO1          125.7703637 MHz

======== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 use
PLW2         19.15500069 W
PLW12         0.43459001 W
PLW13         0.27814001 W
SFO2          500.1320005 MHz
```

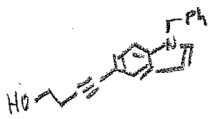

Current Data Parameters
NAME JLK3#0
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140130
Time_ 15.57
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 203.12
DW 48.400 usec
DE 6.50 usec
TE 300.1 K
D1 1.50000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 14.79100037 W
F2 - Processing parameters
SI 65536
SF 500.1300167 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


Carbon 13
Current Data Parameters
NAME JLK3#047
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140130
Time 18.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 41662
SOLVENT CDCl3
NS 1973
DS 0
SWH 29761.904 Hz
FIDRES 0.714366 Hz
AQ 0.6999716 sec
RG 1440
DW 16.800 use
DE 6.50 use
TE 295.6 K
D1 1.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 =======
NUC1 13C
P1 9.13 use
PLW1 123.02998878 W
SFO1 125.7703637 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PLW2 19.15500069 W
PLW12 0.43459001 W
PLW13 0.27814001 W
SFO2 500.1320005 MHz
0.009
16.253
29.326
29.717
30.680
41.799
55.678
55.828
66.369
76.768
77.021
77.275
110.770
111.094
116.908
130.789
151.924
0 ppm
20
40
60
80
100
120
140
160
180

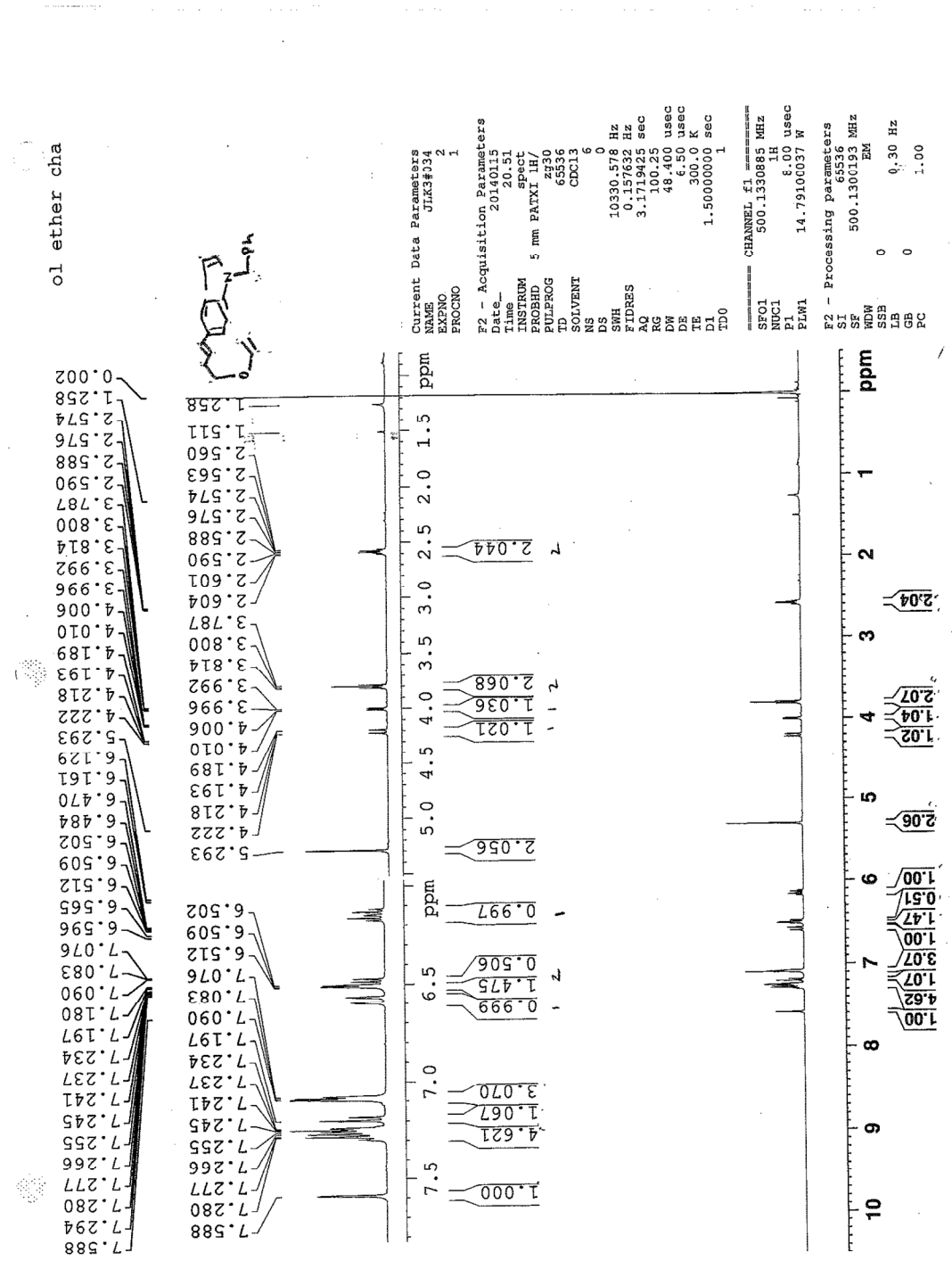

indole ynol chara
Current Data Parameters
NAME JLK3#029
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140108
Time 17.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 40498
SOLVENT CDC13
NS 32
DS 0
SWH 7500.000 Hz
FIDRES 0.185194 Hz
AQ 2.6999166 sec
RG 161
DW 66.667 use
DE 6.50 use
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 use
PLW1 16.98200035 W
SFO1 500.1332508 MHz
F2 - Processing parameters
SI 65536
SF 500.1300209 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

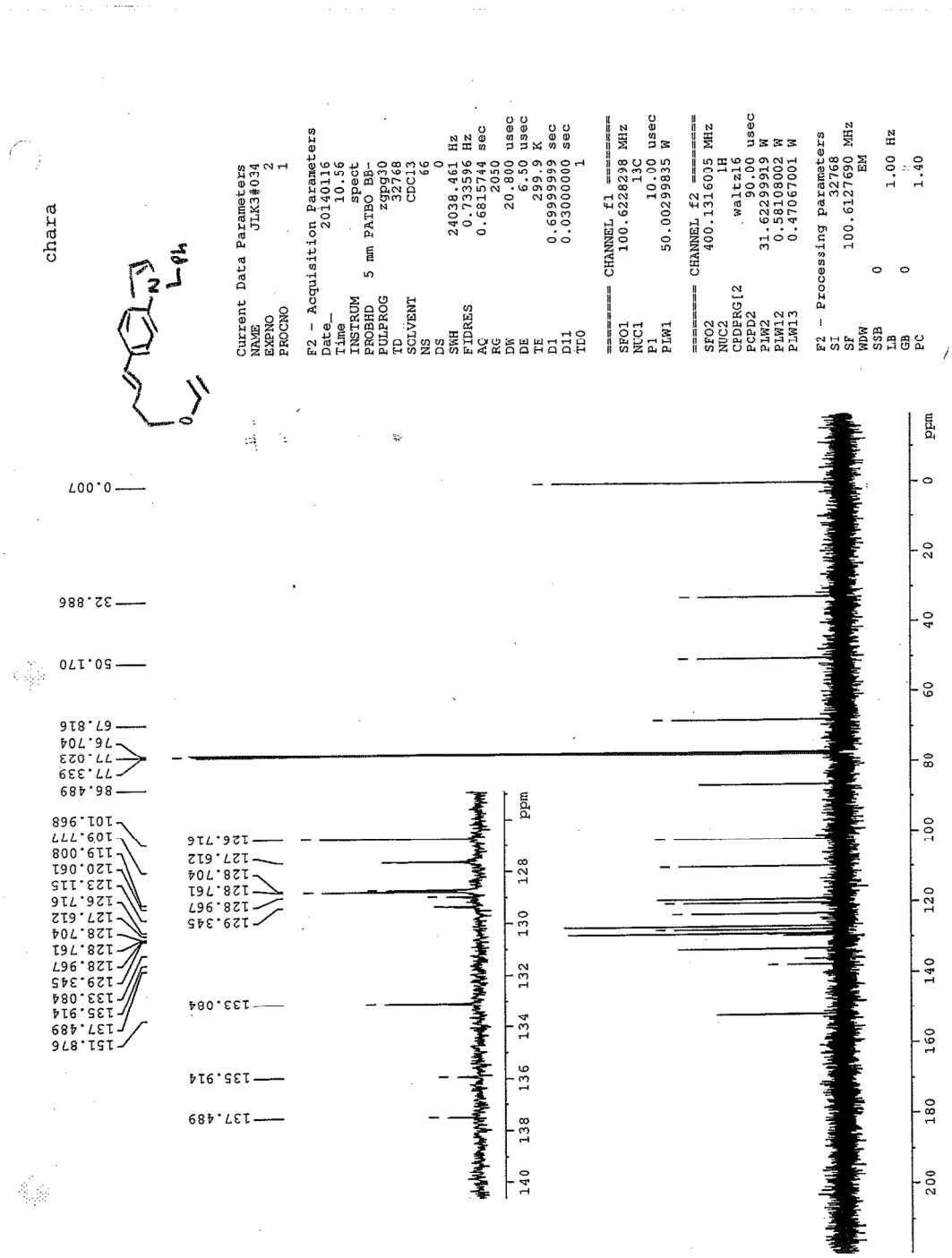


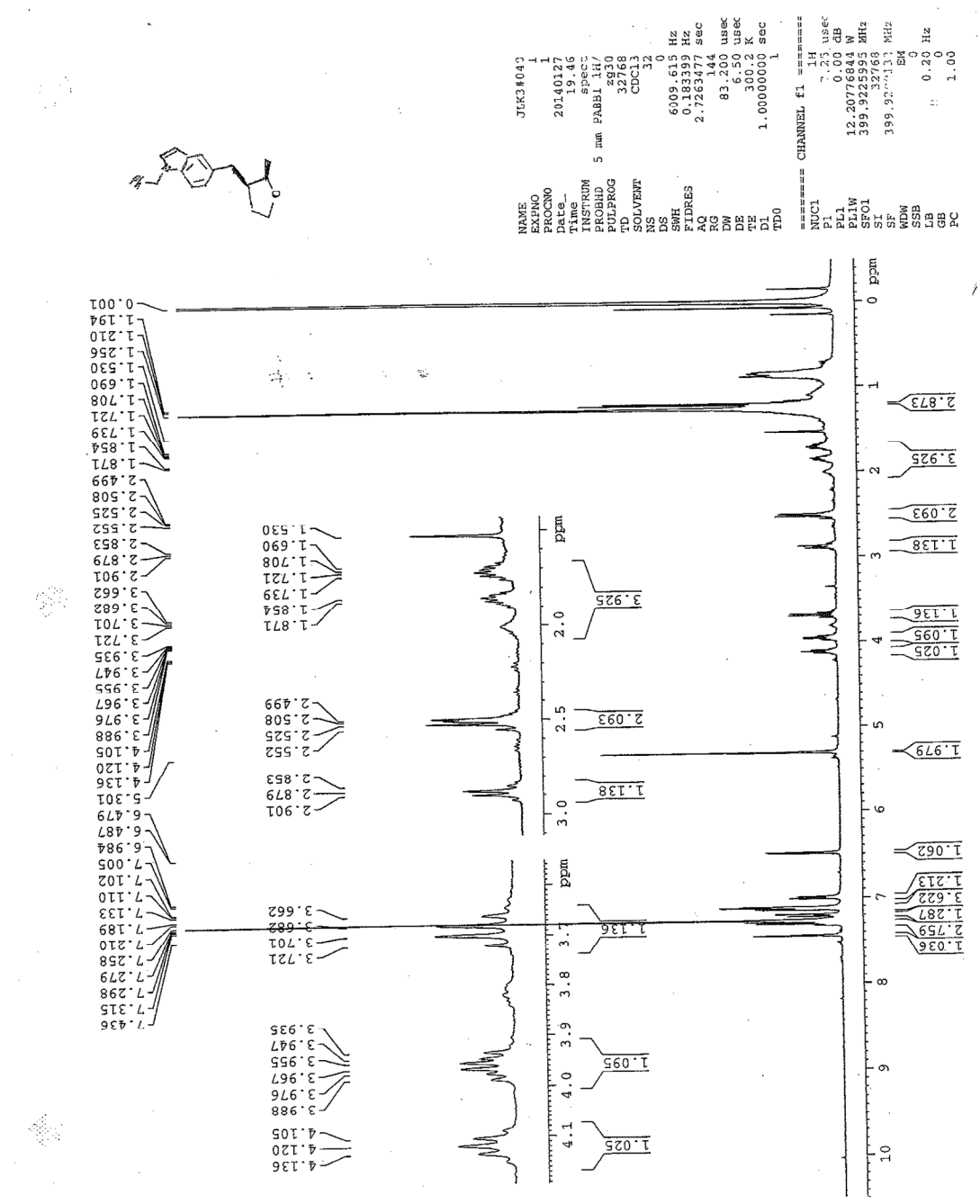
indole ynol chara

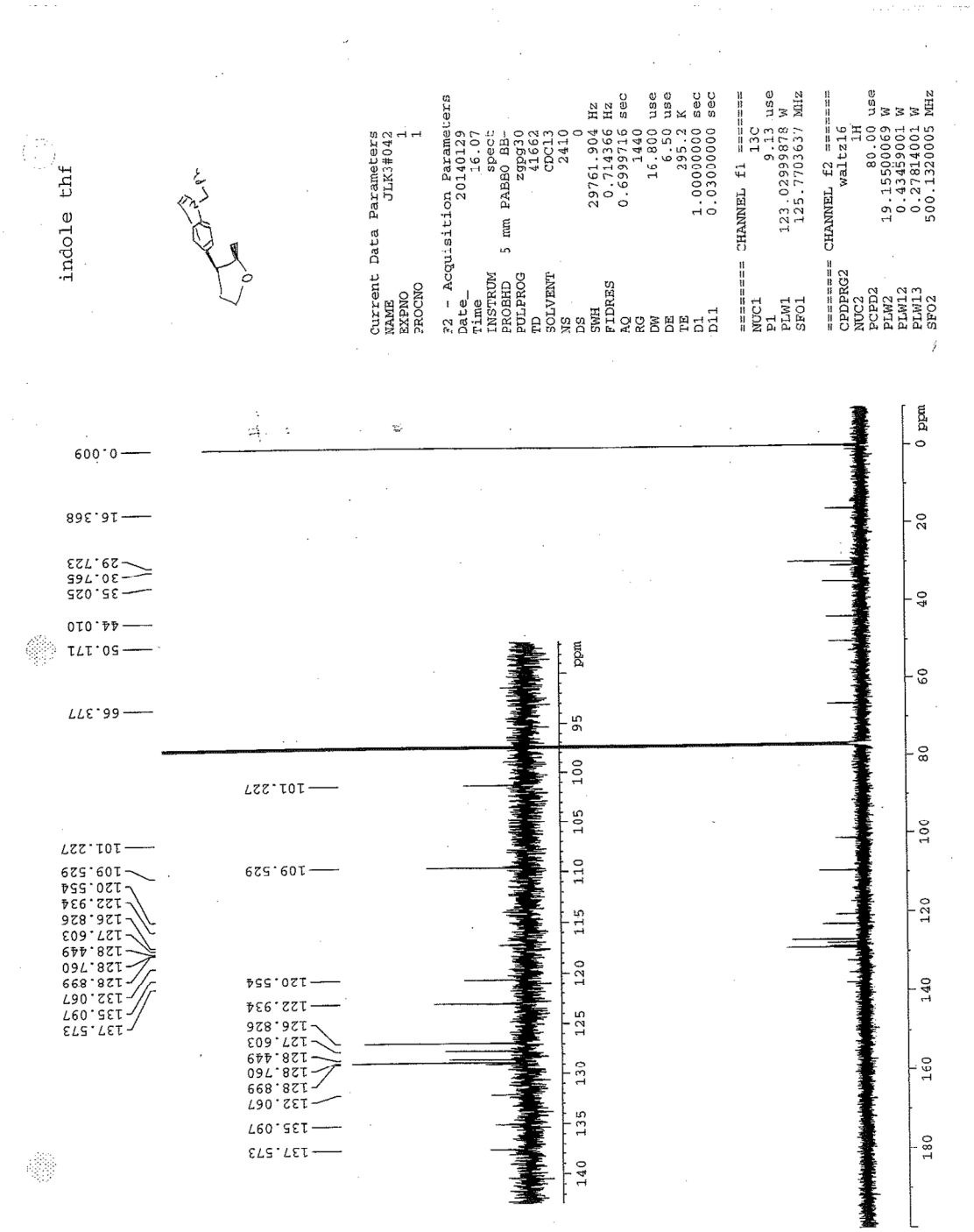


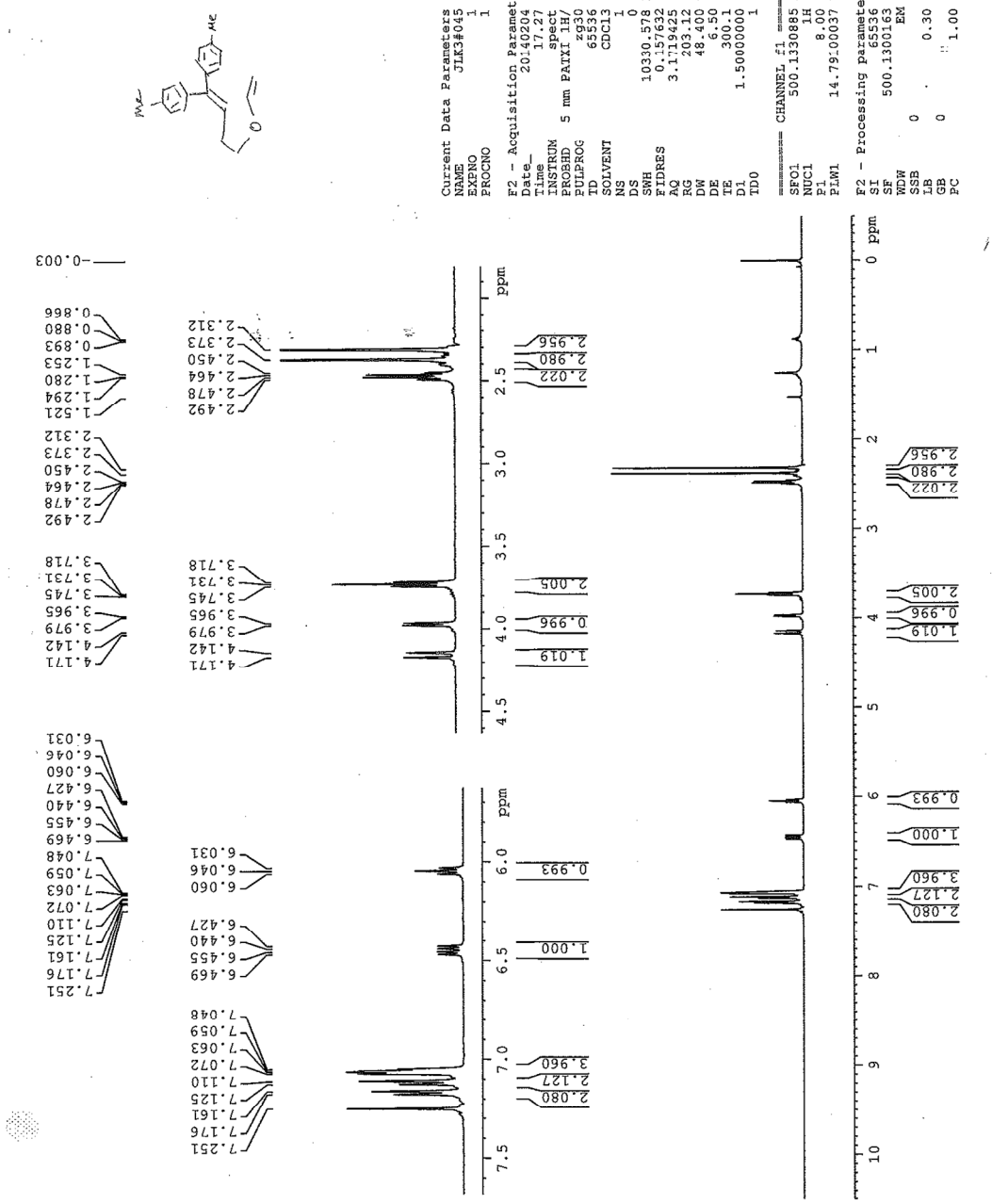

```
Current Data Parameters
NAME                  JLK3#029
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20140108
Time                     17.43
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      41662
SOLVENT                 CDCl3
NS                         37
DS                          0
SWH                 29761.904 Hz
FIDRES                0.714366 Hz
AQ                    0.6999716 sec
RG                       1290
DW                      16.800 use
DE                       6.50 use
TE                      300.1 K
D1                  1.00000000 sec
D11                 0.03000000 sec

======== CHANNEL f1 =======
NUC1                       13C
P1                        9.13 use
PLW1              123.02999878 W
SFO1               125.7703637 MHz

======== CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 use
PLW2              19.15500069 W
PLW12              0.43459001 W
PLW13              0.27814001 W
SFO2              500.1320005 MHz
```


ol ether cha
Current Data Parameters
NAME JLK3#034
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140115
Time 20.51
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 100.25
DW 48.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 14.79100037 W
F2 - Processing parameters
SI 65536
SF 500.1300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


chara
Current Data Parameters
NAME JLK3#034
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140116
Time 10.56
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 66
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.68151744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 0.69999999 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 50.00299835 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 31.62299919 W
PLW12 0.58108002 W
PLW13 0.47067001 W
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
0.007
32.886
50.170
67.816
76.704
77.023
77.339
86.489
101.968
109.777
119.008
120.061
123.115
126.716
127.612
128.704
128.761
128.967
129.345
133.084
135.914
137.489
151.876
126.716
127.612
128.704
128.761
128.967
129.345
133.084
135.914
137.489
ppm
ppm




indole thf
Current Data Parameters
NAME JLK3#042
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140129
Time 16.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 41662
SOLVENT CDCl3
NS 2410
DS 0
SWH 29761.904 Hz
FIDRES 0.714366 Hz
AQ 0.6999716 sec
RG 1440
DW 16.800 use
DE 6.50 use
TE 295.2 K
D1 1.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.13 use
PLW1 123.02999878 W
SFO1 125.7703637 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PLW2 19.15500069 W
PLW12 0.43459001 W
PLW13 0.27814001 W
SFO2 500.1320005 MHz
0.009
16.368
29.723
30.765
35.025
44.010
50.171
66.377
101.227
109.529
120.554
122.934
126.826
127.603
128.449
128.760
128.899
132.067
135.097
137.573
ppm


Current Data Parameters
NAME JLK3#045
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140204
Time 17.27
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 203.12
DW 48.400 usec
DE 6.50 usec
TE 300.1 K
D1 1.50000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 8.00 usec
PLW1 14.79500037 W
F2 - Processing parameters
SI 65536
SF 500.1300163 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
-0.003
0.866
0.880
0.893
1.253
1.280
1.294
1.521
2.312
2.373
2.450
2.464
2.478
2.492
3.718
3.731
3.745
3.965
3.979
4.142
4.171
6.031
6.046
6.060
6.427
6.440
6.455
6.469
7.048
7.059
7.063
7.072
7.110
7.125
7.161
7.176
7.251
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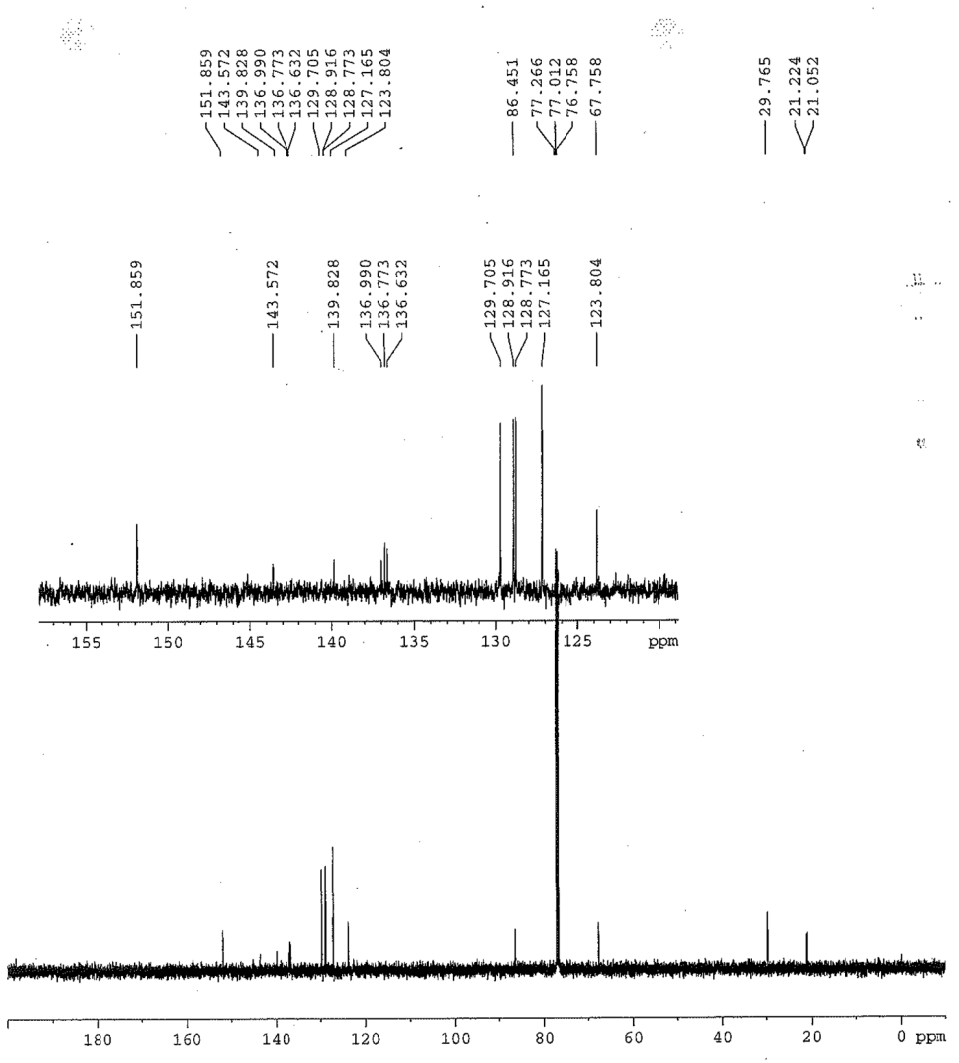
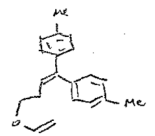
2.022
1.019
0.996
2.005
0.993
1.000
3.960
2.127
2.080



enol ether (diphenyl)

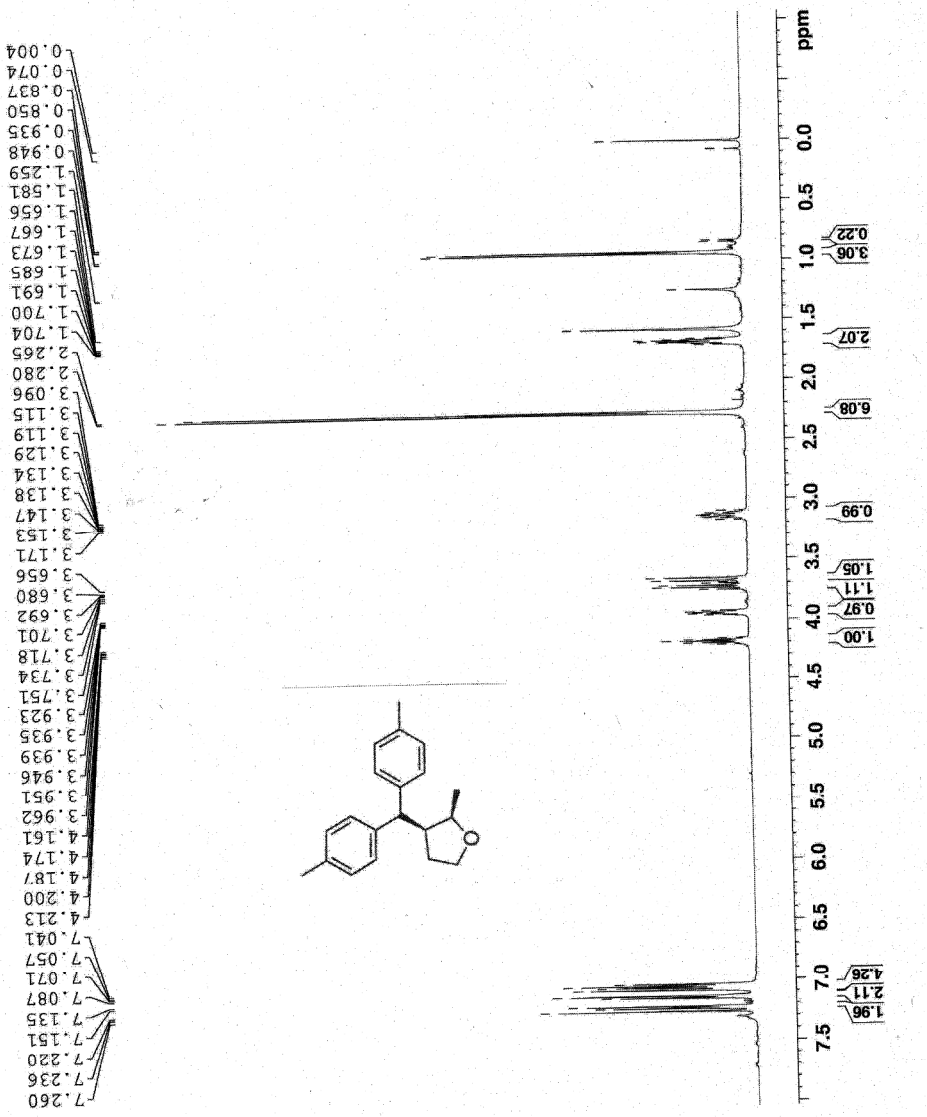



```
Current Data Parameters
NAME                  JLK3#045
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20140204
Time                   17.52
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    41662
SOLVENT               CDCl3
NS                      121
DS                        0
SWH               29761.904 Hz
FIDRES            0.714366 Hz
AQ                0.6999716 sec
RG                     1290
DW                   16.800 use
DE                    6.50 use
TE                   300.1 K
D1             1.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 =======
NUC1                    13C
P1                    9.13 use
PLW1         123.02999878 W
SFO1          125.7703637 MHz

======== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 use
PLW2         19.15500069 W
PLW12         0.43459001 W
PLW13         0.27814001 W
SFO2         500.1320005 MHz
```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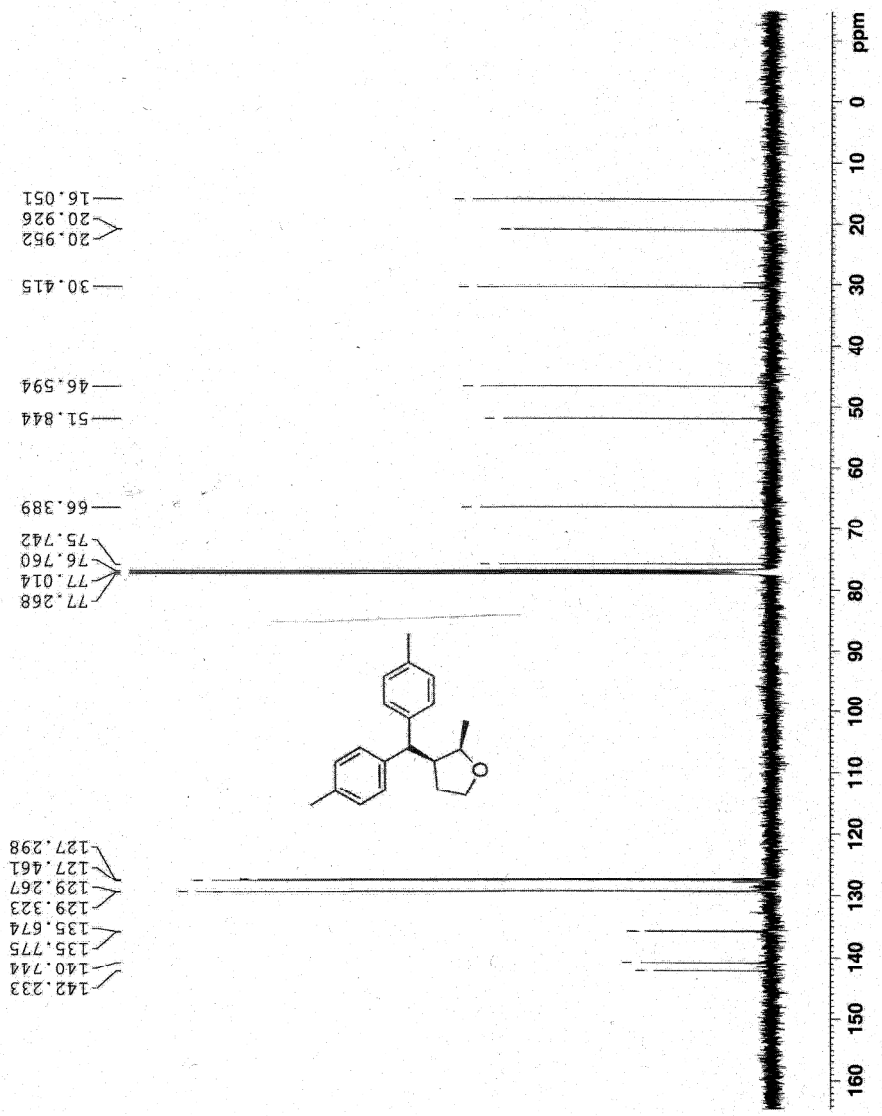
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
0.22
3.06
2.07
6.08
0.99
1.05
1.11
0.97
1.00
4.26
2.11
1.96
0.004
0.074
0.837
0.850
0.935
0.948
1.259
1.581
1.656
1.667
1.673
1.685
1.691
1.700
1.704
2.265
2.280
3.096
3.115
3.119
3.129
3.134
3.138
3.147
3.153
3.171
3.656
3.680
3.692
3.701
3.718
3.734
3.751
3.923
3.935
3.939
3.946
3.951
3.962
4.161
4.174
4.187
4.200
4.213
7.041
7.057
7.071
7.087
7.135
7.151
7.220
7.236
7.260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20.952
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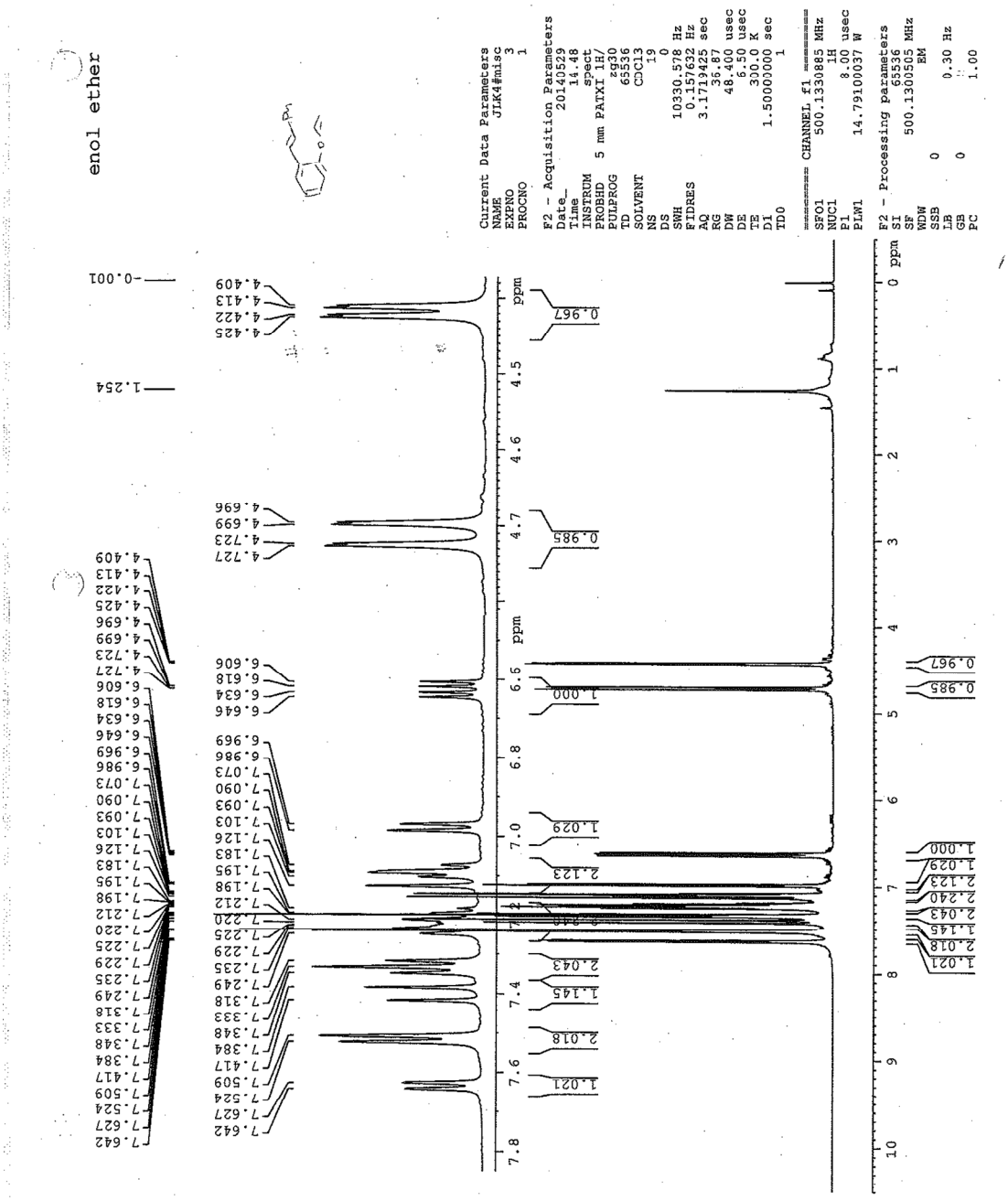
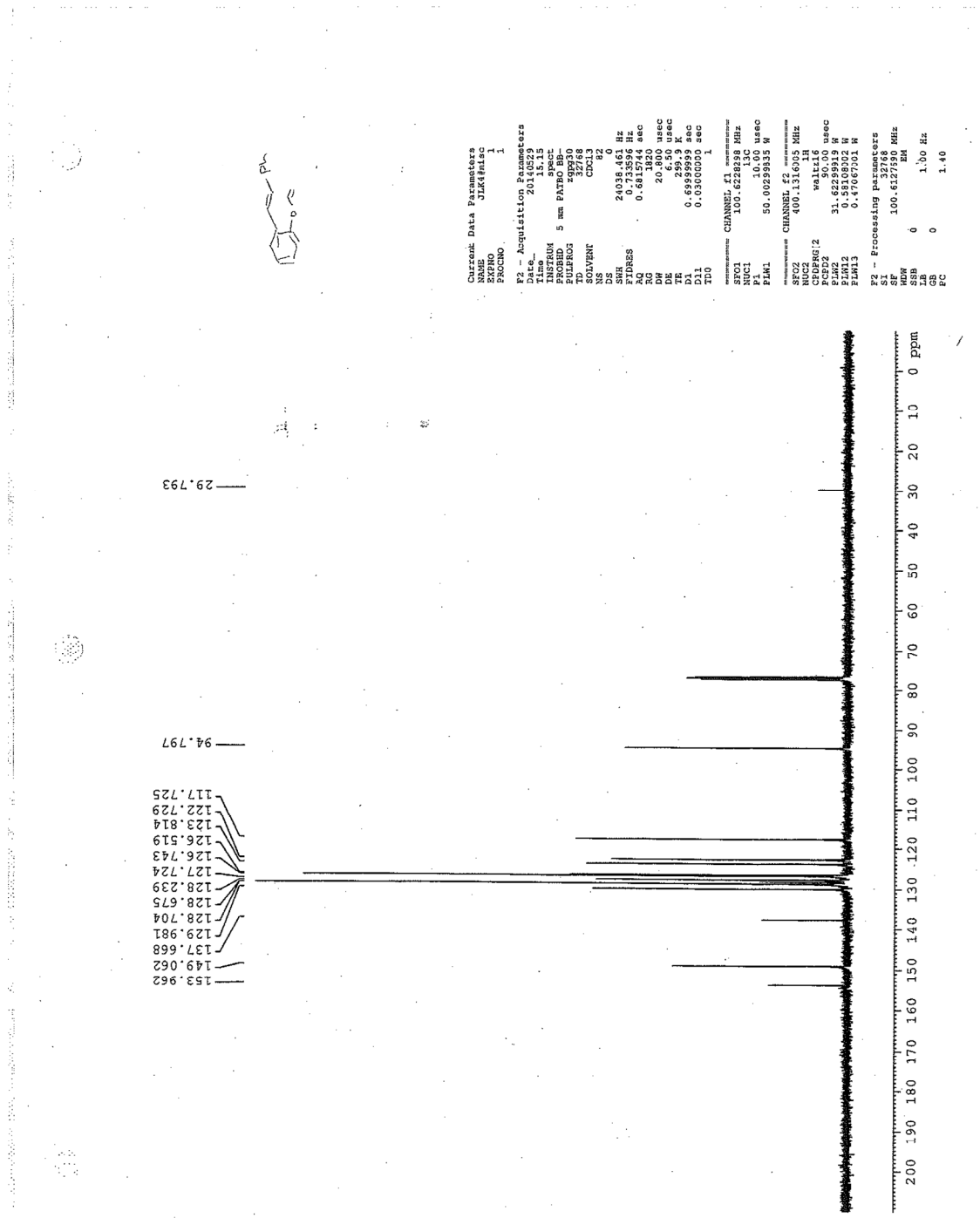
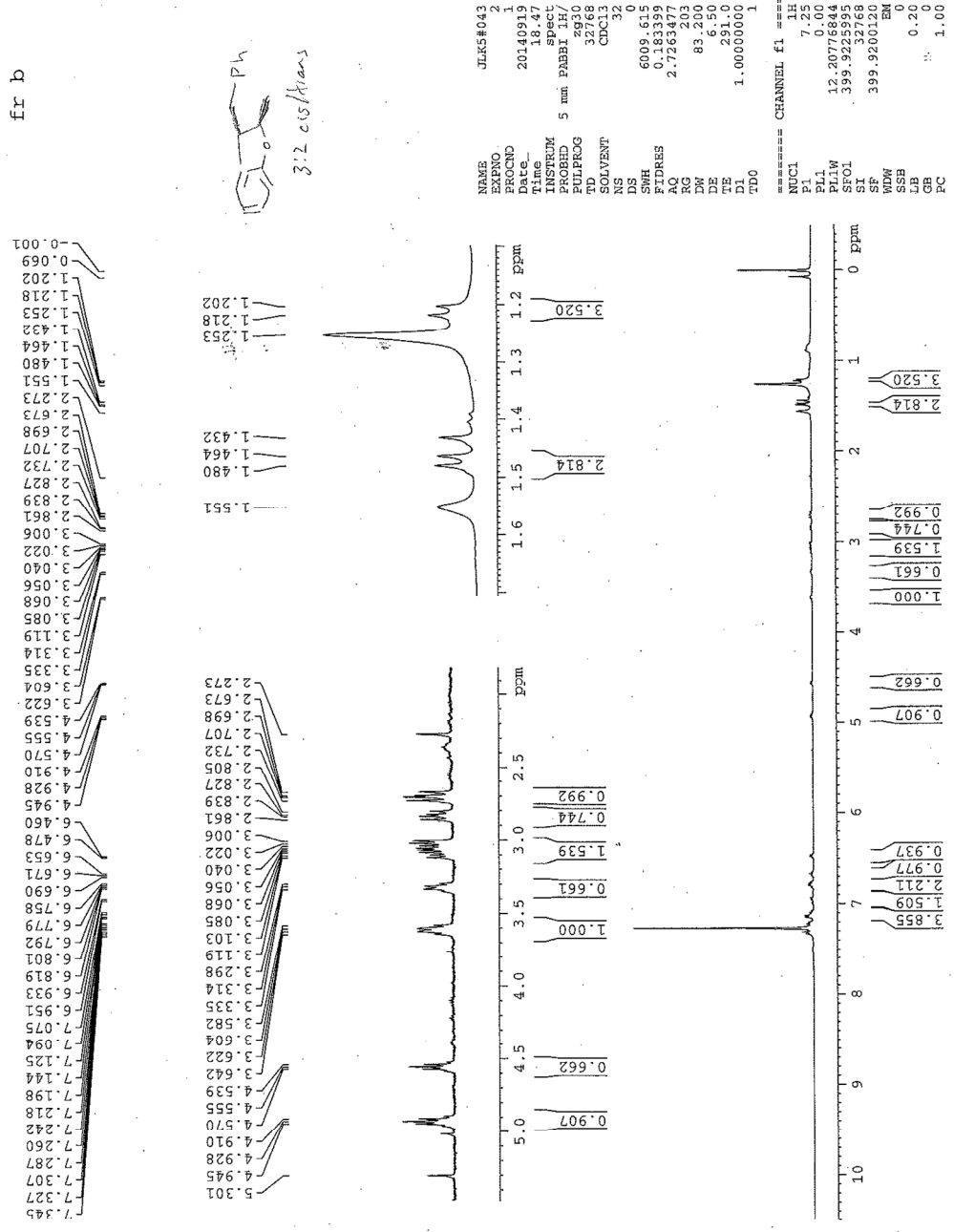
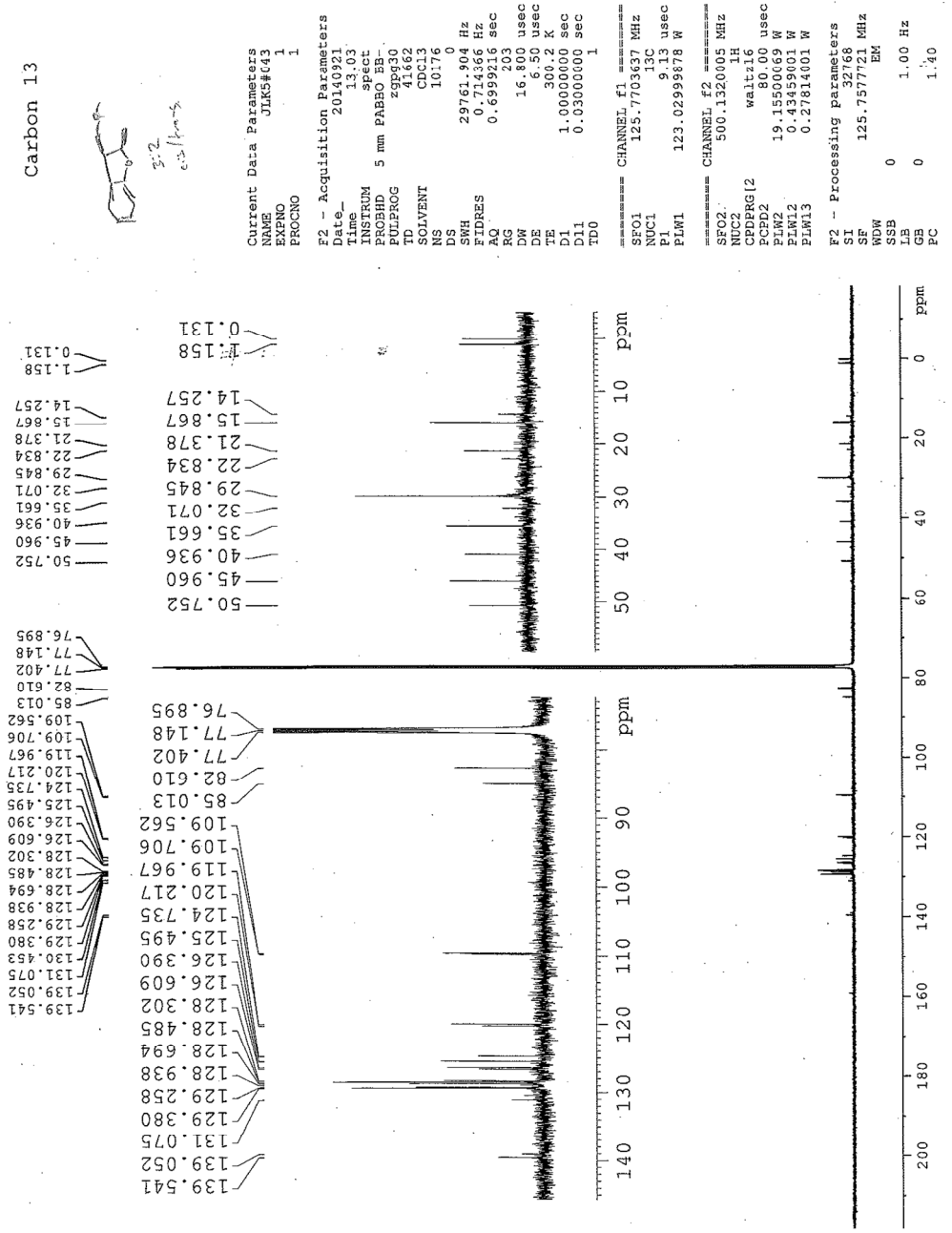
46.594
51.844
66.389
75.742
76.760
77.014
77.268
127.298
127.461
129.267
129.323
135.674
135.775
140.744
142.233
ppm


enol ether


Current Data Parameters
NAME JLK4#misc
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140529
Time 15.15
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 82
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 1820
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 0.69999999 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 50.00299835 W
CHANNEL f2
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 31.62299919 W
PLW12 0.58108002 W
PLW13 0.47067001 W
F2 - Processing parameters
SI 32768
SF 100.6127590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
29.793
94.797
117.725
122.729
123.814
126.519
126.743
127.724
128.239
128.675
128.704
129.981
137.668
149.062
153.962
ppm


fr b
3:2 cis/trans
Ph
NAME JLK5#043
EXPNO 2
PROCNO 1
Date_ 20140919
Time 18.47
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6009.615 Hz
FIDRES 0.1833399 Hz
AQ 2.7263477 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 291.0 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.25 usec
PL1 0.00 dB
PL1W 12.20776844 W
SFO1 399.9225995 MHz
SI 32768
SF 399.9200120 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
ppm


Carbon 13
Current Data Parameters
NAME JLK5#643
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140921
Time 13.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 41662
SOLVENT CDCl3
NS 10176
DS 0
SWH 29761.904 Hz
FIDRES 0.714366 Hz
AQ 0.6999216 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.13 usec
PLW1 123.02999678 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.15500069 W
PLW12 0.43459001 W
PLW13 0.27814001 W
F2 - Processing parameters
SI 32768
SF 125.7577721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm