Catalytic Conjugate Addition of Allyl Groups to Styryl-Activated Enones

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Supplementary Material

General. Melting points were determined using a Mel-Temp II melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded on Bruker DRX 300 or 400 MHz spectrometers or a Gemini-400 (400 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: 7.24 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, h = hextet, br = broad, m = multiplet), and coupling constants (Hz). ^{13}C NMR was recorded using a Bruker 400 MHz (100 MHz) instument, a Gemini-400 (100 MHz) instrument, or a Gemini-500 (125 MHz) instrument with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: 77.0 ppm). Low-resolution mass spectrometry was performed by the University of North Carolina, Department of Chemistry Mass Spectrometry Facility. Infrared (IR) spectra were obtained on a Nicolet 560 Magna-FTIR.

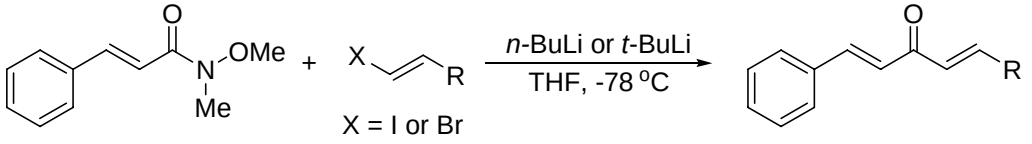
Liquid chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2, 40-63 μm) purchased from SiliCycle. Thin layer chromatography was performed on 250 μm silica gel plates from EMD Chemicals Inc. Visualization was achieved using UV light, phosphomolybdic acid in ethanol, or potassium permanganate in water, each followed by heating.

Analytical supercritical fluid chromatography (SFC) was performed on a Berger Instruments supercritical chromatograph equipped with an Alcott autosampler and a Knauer UV detector.

All reactions were conducted in oven or flame dried glassware under an inert atmosphere of nitrogen or argon. Anhydrous THF, used in reactions that were prepared in a dry-box, was purchased from Aldrich Chemical Company. For all other reactions prepared outside of a dry-box, THF that was freshly distilled from Na metal and benzophenone was used. Tris(dibenzylideneacetone)dipalladium, Bis(1,5-cyclooctadiene)nickel, and tricyclohexylphosphine were purchased from Strem Chemical Company. Allylboronic acid pinacol ester was purchased from Aldrich Chemical Company and distilled through a 6 inch Vigreux column (58-62 ^{o}C at 20 torr) and stored in the freezer under Ar. The styryl-activated substrates were synthesized by addition of the desired vinyl lithium reagent, prepared by Li-halogen exchange of the corresponding vinyl iodide or bromide, to *N*-methoxy-*N*-methylcinnamide at low temperature and is described below. *N*-Methoxy-*N*-methylcinnamide was synthesized according to the literature (Hiyama, T.; Reddy, G. B.; Minami, T.; Hanamoto, T. *Bull. Chem. Soc. Jpn.* **1995**, *68*, 350.). 1-Iododheptene was synthesized from 1-heptyne via hydroalumination with DIBAL followed by iodination (Stille, J. K.; Simpson, J. H. *J. Am. Chem. Soc.* **1987**, *109*, 2138. Trost, B. M.; Rudd, M. T. *Org. Lett.* **2003**, *5*, 4599.). 2-Iodovinylcyclohexane and 1-iodo-3,3-dimethyl-1-butene were synthesized via hydroboration of cyclohexylacetylene or *t*-butylacetylene, respectively, followed by iodination (Brown, H. C.; Hamaoka, T. Ravindran, N.; Subrahmanyam, C.; Somayaji, V.; Bhat, N. G. *J. Org. Chem.* **1989**, *54*, 6075. Gagnon, D.; Lauzon, S.; Godbout, C.; Spino, C. *Org. Lett.* **2005**, *7*, 4769.). Vinyl iodides bearing pendant TBS-protected alcohols were synthesized via hydrozirconation of the TBS-protected propargyl or homo propargyl alcohol with the Schwartz reagent, followed by iodination (Germain, J.; Deslongchamps, P. *J. Org. Chem.* **2002**, *67*, 5269.). Vinyl bromides were purchased from Aldrich Chemical Company and used without further

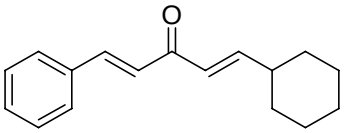
purification. All other reagents were purchased from either Fisher or Aldrich Chemical Companies and used directly.

Representative procedure for the synthesis of styryl-activated substrates.

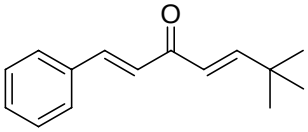


Method A, from the vinyl iodide: To 2.25 g (10.0 mmol) of 1-iodoheptene in 10 mL of THF at –78 °C was added 4.2 mL (10 mmol) of a 2.4 M solution of *n*-BuLi in hexane dropwise. This solution was stirred for 30 min at –78 °C and then transferred dropwise via canula to a solution of 0.965g (5.00 mmol) of *N*-methoxy-*N*-methylcinnamide in 50 mL of THF at –78 °C. After complete addition, TLC analysis showed complete consumption of the starting material after 15 min at –78 °C, so the reaction was subsequently quenched with satd. $NH_4Cl_{(aq)}$. The crude reaction was transferred to a separatory funnel with 1 M HCl and CH_2Cl_2. The organic layer was collected after shaking, and the aqueous layer was extracted with CH_2Cl_2 (1x). The combined organics were dried over Na_2SO_4 and concentrated using reduced pressure. Silica gel chromatography (hexanes/EtOAc) of the crude mixture afforded 1.0 g (91%) of (1*E*, 4*E*)-1-phenyldeca-1,4-dien-3-one as a yellow oil. Spectral data was consistent with the literature (Tsuge, O.; Kanemasa, S.; Nakagawa, N.; Suga, H. *Bull. Chem. Soc. Jpn.* **1987**, *69*, 4091.).

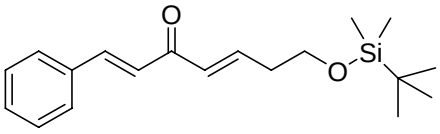
Method B, from the vinyl bromide: To 1.05 g of (*E*)-1-bromopropene in 16 mL of THF at –78 °C was added 10.8 mL (17 mmol) of a 1.6 M solution of *t*-BuLi in pentane dropwise. This solution was stirred at this temperature for 30 min and then transferred at ~1 drop/s via canula to a solution of 0.832 g of *N*-methoxy-*N*-methylcinnamide in 33 mL of THF at –78 °C (addition of the vinyllithium at faster rates gave lower yields). After complete addition, TLC analysis showed complete consumption of the starting material after 15 min at –78 °C, so the reaction was subsequently quenched with satd. $NH_4Cl_{(aq)}$. The crude reaction was transferred to a separatory funnel with 1 M HCl and Et_2O. The organic layer was collected after shaking, and the aqueous layer was extracted with Et_2O (1x). The combined organics were washed with H_2O then brine and finally dried over Na_2SO_4 and concentrated using reduced pressure. Silica gel chromatography (hexanes/EtOAc) of the crude mixture afforded 0.571 g (76%) of (1*E*, 4*E*)-1-phenylhexa-1,4-dien-3-one as a yellow oil.



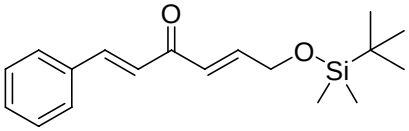
(1*E*, 4*E*)-1-cyclohexyl-5-phenylpenta-1,4-dien-3-one. Prepared using Method A in 96% yield. A yellow solid. mp 42-46 °C. R_f = 0.20 (12:1 Hexanes:EtOAc); IR (KBr): 2924 (m), 2847 (m), 1806 (w), 1670 (s), 1627 (s), 1592 (s), 1441 (s), 1332 (s), 1278 (m), 1184 (m) cm^{-1}; ^1H NMR: δ 7.62 (1H, d, *J* = 16 Hz), 7.55 (2H, m), 7.30-7.42 (3H, m), 6.97 (1H, d, *J* = 16 Hz), 6.93 (1H, dd, *J* = 16 Hz, *J* = 6.8 Hz), 6.37 (1H, dd, *J* = 14 Hz, *J* = 1.2 Hz), 2.19 (1H, m), 1.61-1.88 (5H, m) 1.10-1.40 (5H, m); ^{13}C NMR: δ 189.6, 153.2, 142.8, 134.8, 130.3, 128.9, 128.2, 126.8, 124.8, 40.87, 31.80, 25.90, 25.71. LRMS (ESI+) Calc'd for $C_{17}H_{20}O$ $(M + Na)^+$: 263.1 Found $(M + Na)^+$: 263.2.



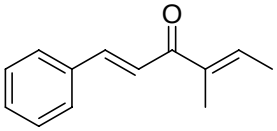
(1*E*, 4*E*)-6,6-dimethyl-1-phenylhepta-1,4-dien-3-one. Prepared using Method A in 86% yield. An off-white solid. mp 60-64 ^{o}C. R_f = 0.30 (12:1 Hexanes:EtOAc); IR (KBr): 3060 (w), 3017 (w), 2948 (m), 2862 (w), 1950 (w), 1880 (w), 1658 (s), 1588 (s), 1449 (m), 1324 (s), 1208 (s), 1165 (s) cm^{-1}; ^{1}H NMR: δ 7.63 (1H, d, *J* = 16 Hz), 7.56 (2H, m), 7.37 (3H, m), 6.98 (2H, d, *J* = 16 Hz), 6.32 (1H, d, *J* = 16 Hz), 1.12 (9H, s); ^{13}C NMR: δ 189.7, 157.9, 142.9, 134.8, 130.3, 128.9, 128.3, 124.9, 124.5, 33.97, 28.72. LRMS (ESI+) Calc'd for $C_{15}H_{18}O$ (M + Na)$^{+}$: 237.1 Found (M + Na)$^{+}$: 237.1.



(1*E*, 4*E*)-7-(*t*-butyldimethylsilyloxy)-1-phenylhepta-1,4-dien-3-one. Prepared using Method A in 87% yield. A yellow oil. R_f = 0.19 (12:1 Hexanes:EtOAc); IR (neat): 3029 (w), 2952 (s), 2854 (s), 1950 (w), 1802 (w), 1662 (s), 1631 (s), 1596 (s), 1472 (m), 1336 (m), 1254 (s), 1185 (m) cm^{-1}; ^{1}H NMR: δ 7.63 (1H, d, *J* = 16 Hz), 7.55 (2H, m), 7.38 (3H, m), 6.98 (1H, dt, *J* = 16 Hz, *J* = 8.6 Hz), 6.96 (1H, d, *J* = 16 Hz), 3.61 (1H, dt, *J* = 16 Hz, *J* = 1.2 Hz), 3.76 (2H, t, *J* = 6.4 Hz) 3.08 (2H, dq, *J* = 7.2 Hz, *J* = 1.6 Hz), 0.88 (9H, s), 0.050 (3H, s), 0.042 (3H, s); ^{13}C NMR: δ 189.1, 144.7, 143.1, 134.8, 130.8, 130.3, 128.9, 128.2, 124.6, 61.60, 36.15, 25.86, 18.28, -5.33. LRMS (ESI+) Calc'd for $C_{19}H_{28}O_2Si$ (M + Na)$^{+}$: 339.2 Found (M + Na)$^{+}$: 339.2.



(1*E*, 4*E*)-6-(*t*-butyldimethylsilyloxy)-1-phenylhexa-1,4-dien-3-one. Prepared using Method A in 60% yield. A yellow oil. R_f = 0.20 (12:1 Hexanes:EtOAc); IR (neat): 3060 (w), 2955 (s), 2932 (s), 2854 (s), 1953 (w), 1806 (w), 1666 (s), 1634 (s), 1592 (s), 1449 (s), 1332 (s), 1254 (s), 1138 (s) cm^{-1}; ^{1}H NMR: δ 7.64 (1H, d, *J* = 16 Hz), 7.56 (2H, m), 7.38 (3H, m), 7.02 (1H, dt, *J* = 15 Hz, *J* = 3.6 Hz), 6.95 (1H, d, *J* = 16 Hz), 6.73 (1H, dt, *J* = 15 Hz, *J* = 2.0 Hz), 4.41 (2H, m), 0.94 (9H, s), 0.10 (6H, s); ^{13}C NMR: δ 189.1, 146.1, 143.4, 134.7, 130.4, 128.9, 128.3, 126.5, 125.3, 62.52, 25.86, 18.37, -5.41. LRMS (ESI+) Calc'd for $C_{18}H_{26}O_2Si$ (M + H)$^{+}$: 303.2 Found (M + H)$^{+}$: 303.2.



(1*E*, 4*E*)-4-methyl-1-phenylhexa-1,4-dien-3-one. Prepared using Method B in 53% yield. A yellow oil. R_f = 0.19 (14:1 Hexanes:EtOAc); IR (neat): 3060 (m), 2920 (m), 1953 (w), 1887 (w), 1654 (s), 1600 (s), 1449 (s), 1328 (s), 1300 (s), 1223 (s) cm^{-1}; ^{1}H NMR: δ 7.59 (1H, d, *J* = 16 Hz), 7.55 (2H, m), 7.37 (3H, m), 7.28 (1H, d, *J* = 16 Hz), 6.82 (1H, q, *J* = 6.0 Hz), 1.89 (3H, d, *J* = 6.0 Hz), 1.88 (3H, s); ^{13}C NMR: δ 191.7, 142.5, 139.3, 137.4, 135.2, 129.9, 128.8, 128.1, 121.7, 14.80, 11.55. LRMS (ESI+) Calc'd for $C_{13}H_{14}O$ (M + Na)$^{+}$: 209.1 Found (M + Na)$^{+}$: 209.1.

Procedure for the catalytic conjugate allylation of dibenzylidene acetone, chalcone, and benzylidene acetone (Scheme 1).

The procedure for dibenzylidene acetone is representative. An oven-dried 20 mL vial equipped with a magnetic stir-bar was charged with 4.9 mg (0.0053 mmol) of tris(dibenzylideneacetone)dipalladium, 3.6 mg (0.013 mmol) of tricyclohexylphosphine, and 1.42 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 45 min. Next, 39.4 mg (0.234 mmol) of allylboronic acid pinacol ester was added followed by 50.0 mg (0.213 mmol) of dibenzylidene acetone. The vial was capped, taped with electrical

tape, removed from the dry-box, and allowed to stir at ambient temperature for the time indicated in Scheme 1. After this time period, water was added and the mixture transferred to a separatory funnel with CH_2Cl_2. After gently swirling the layers (to avoid formation of emulsions), the organic layer was collected and the aqueous layer washed with CH_2Cl_2 (1x). The combined organic layers were dried with Na_2SO_4, and volatiles were removed under reduced pressure. Silica gel chromatography (hexanes/EtOAc) afforded 50.4 mg (80%) of 1,5-diphenyl-1,7-octadien-3-one as a white solid whose spectral data was consistent with the literature (Mandal, S. K.; Amin, Sk. R.; Crowe, W. E. *J. Am. Chem. Soc.* **2001**, *123*, 6457.).

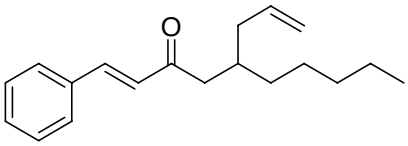
Procedure for the catalyst survey in the conjugate allylation (Table 1).

Procedure using $Pd_2(dba)_3$: An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 2.3 mg (0.0025 mmol) of tris(dibenzylideneacetone)dipalladium, 0.0060 mmol of phosphine ligand, and 0.20 mL of THF in a dry-box under an argon atmosphere (ligands that could not be weighed in directly were added as 0.20 mL (0.0060 mmol) of a 0.030 M stock solution in THF and no additional solvent was added). The vial was capped and stirred for 45 min. Next, 19 mg (0.11 mmol) of allylboronic acid pinacol ester was added followed by 0.31 mL (0.10 mmol) of a 0.325 M stock solution of (1*E*, 4*E*)-1-phenyldeca-1,4-dien-3-one in THF. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for the time indicated in Table 1. After this time period, water was added and the mixture transferred to a separatory funnel with CH_2Cl_2. After gently swirling the layers, the organic layer was collected and the aqueous layer washed with CH_2Cl_2 (1x). The combined organic layers were dried with Na_2SO_4, and volatiles were removed under reduced pressure. NMR analysis of the crude mixture was used to determine the regioselectivity of the reaction. Yields were determined after isolation of pure material, as a mixture of isomers, using silica gel chromatography (hexanes/EtOAc).

Procedure using $Ni(cod)_2$: An oven-dried 20 mL scintillation vial equipped with a magnetic stir-bar was charged with 6.0 mg (0.022 mmol) of bis(1,5-cyclooctadiene)nickel, 0.0438 mmol of phosphine ligand, and 1.46 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 45 min. Next, 44.2 mg (0.263 mmol) of allylboronic acid pinacol ester was added followed by 50.0 mg (0.219 mmol) of (1*E*, 4*E*)-1-phenyldeca-1,4-dien-3-one. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 4 h. After this time period, ~15 mL of water was added and the mixture transferred to a separatory funnel with CH_2Cl_2. After gently swirling the layers, the organic layer was collected and the aqueous layer washed with CH_2Cl_2 (1x). The combined organic layers were dried with Na_2SO_4, and volatiles were removed under reduced pressure. NMR analysis of the crude mixture was used to determine the regioselectivity of the reaction. Yields were determined after isolation of pure material, as a mixture of isomers, using silica gel chromatography (hexanes/EtOAc).

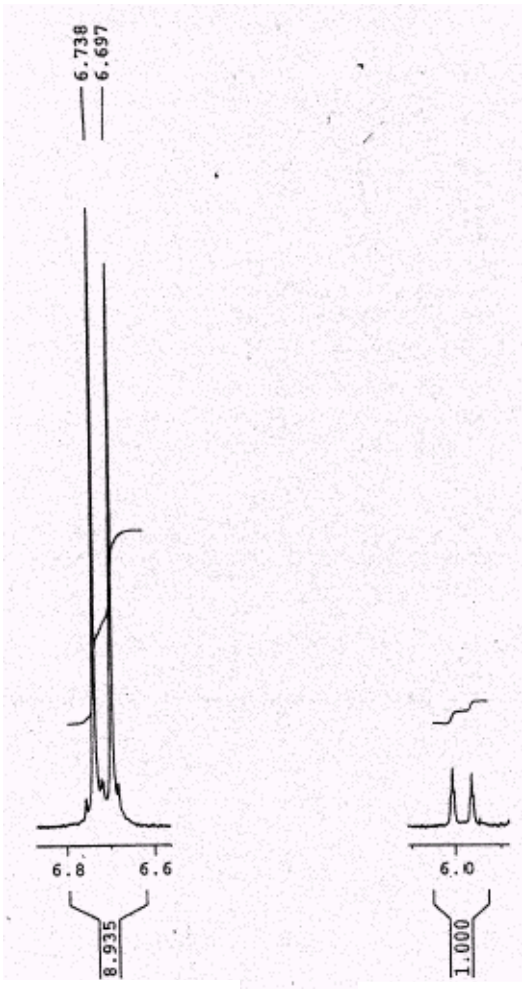
Procedure for the Ni-catalyzed conjugate allylation of styryl enones (Table 2).

The conjugate allylation of styryl-activated enones using $Ni(cod)_2$ was performed using the same procedure described above for the reaction in Table 1 using $Ni(cod)_2$, where tricyclohexylphosphine was the ligand used. Reactions were performed using 50.0 mg of substrate and run at the temperature and for the time indicated in Table 2. For the allylation reaction using (*E*)-5-methyl-1-phenylhexa-1,4-dien-3-one (entry 8) as the substrate, 2.0 equiv of allylboronic acid pinacol ester was used. For substrates bearing TBS-protected alcohols (entry 5, 6), buffer (pH=7) was used instead of H_2O in the extraction step. The reaction utilizing (1*E*, 4*E*)-4-methyl-1-phenylhexa-1,4-dien-3-one as the substrate (entry 9) was quenched with 2.2 equiv of 1.0 M HCl in Et_2O under N_2 before adding H_2O.



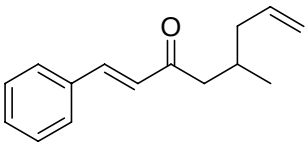
***E*)-5-allyl-1-phenyldec-1-en-3-one.** An oil. Regioisomers were separable using iterative column chromatography (SiO_2/hexanes:EtOAc). R_f(major) = 0.22 (18:1 Hexanes:EtOAc); R_f(minor) = 0.19 (18:1 Hexanes:EtOAc); IR (major, neat): 3064 (w), 2948 (s), 2917 (s), 2858 (s), 1942 (w), 1821 (w), 1689 (s), 1654 (s), 1608 (s), 1445 (s), 1320 (m), 1167 (m) cm^{-1}; ^1H NMR: δ (major) 7.48-7.59 (3H, m), 7.37 (3H, m), 6.72 (1H, d, *J* = 16 Hz), 5.76 (1H, m), 5.01 (2H, d, *J* = 12 Hz), 2.61 (1H, dd, *J* = 16 Hz, *J* = 6.0 Hz), 2.52 (1H, dd, *J* = 16 Hz, *J* = 6.0 Hz), 1.95-2.20 (3H, m), 1.15-1.40 (8H, m), 0.86 (3H, t, *J* = 7.2 Hz); δ (minor) 7.20-7.30 (2H, m), 7.10-7.20 (3H, m), 6.72 (1H, dt, *J* = 16 Hz, *J* = 7.2 Hz), 5.99 (1H, d, *J* = 16 Hz), 5.63 (1H, m), 4.94 (2H, m), 3.29 (1H, p, *J* = 7.2 Hz), 2.82 (2H, m), 2.37 (2H, t, *J* = 7.2 Hz), 2.13 (2H, q, *J* = 6.8 Hz), 1.38 (2H, p, *J* = 7.2 Hz), 1.17-1.32 (6H, m), 0.87 (3H, t, *J* = 6.8 Hz); ^{13}C NMR: δ (major) 200.4, 142.2, 136.6, 134.6, 130.3, 128.9, 128.2, 126.6, 116.6, 45.23, 38.26, 34.07, 33.80, 31.97, 26.40, 22.58, 14.03. LRMS (ESI+) Calc'd for $C_{19}H_{26}O$ $(M + Na)^+$: 293.2 Found $(M + Na)^+$: 293.2.

^1H NMR analysis of Crude Reaction Mixture (400 MHz, $CDCl_3$):



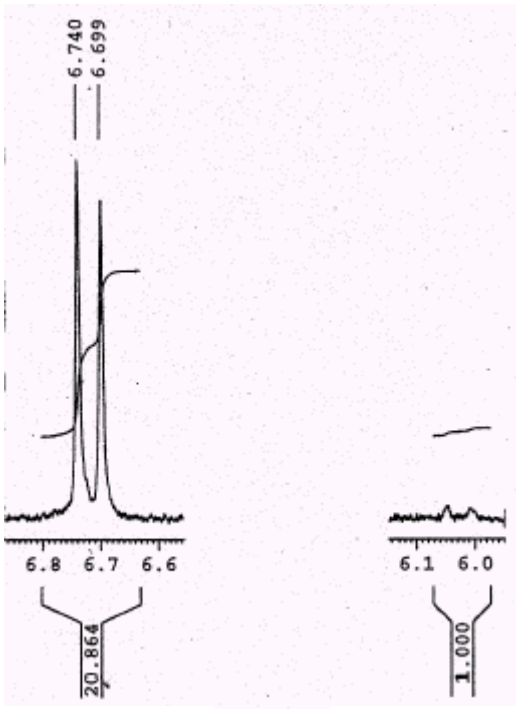
Vinyl CH (minor + major isomers)

Vinyl CH (minor isomer)



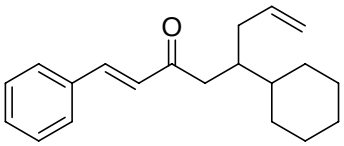
(*E*)-5-methyl-1-phenylocta-1,7-dien-3-one. An oil. R_f = 0.24 (12:1 Hexanes:EtOAc); IR (neat): 3064 (m), 2955 (s), 2917 (s), 1946 (w), 1829 (w), 1689 (s), 1666 (s), 1611 (s), 1452 (s), 1324 (s), 1173 (s) cm^{-1}; ^1H NMR: δ 7.45-7.57 (3H, m), 7.28-7.44 (3H, m), 6.72 (1H, d, *J* = 16 Hz), 5.77 (1H, m), 5.02 (2H, d, *J* = 14 Hz), 2.66 (1H, dd, *J* = 16 Hz, *J* = 6.0 Hz), 2.43 (1H, dd, *J* = 16 Hz, *J* = 8.0 Hz), 2.19 (1H, m), 1.95-2.12 (2H, m), 0.95 (3H, d, *J* = 6.4 Hz); ^{13}C NMR: δ 200.2, 142.4, 136.7, 134.5, 130.4, 128.9, 128.2, 126.5, 116.5, 47.43, 41.20, 29.52, 19.77. LRMS (ESI+) Calc'd for $C_{15}H_{18}O$ $(M + Na)^+$: 237.1 Found $(M + Na)^+$: 237.1.

^1H NMR analysis of Crude Reaction Mixture (400 MHz, $CDCl_3$):



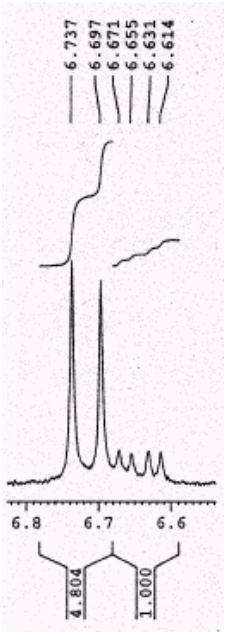
Vinyl CH (minor + major isomers)

Vinyl CH (minor isomer)

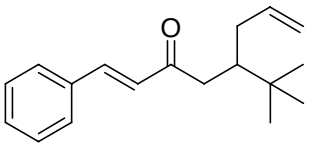


(*E*)-5-cyclohexyl-1-phenylocta-1,7-dien-3-one. An oil. Regioisomers were inseparable using column chromatography (SiO_2/hexanes:EtOAc). R_f = 0.29 (12:1 Hexanes:EtOAc); IR (neat): 3056 (m), 2913 (s), 2842 (s), 1953 (w), 1817 (w), 1685 (s), 1662 (s), 1603 (s), 1441 (s), 1320 (m), 1181 (m) cm^{-1}; ^1H NMR: δ 7.52 (3H, m, major), 7.37 (3H, m, major), 7.25 (2H, m, minor), 7.16 (3H, m, minor), 6.72 (1H, d, *J* = 16 Hz, major), 6.65 (1H, dd, *J* = 16 Hz, *J* = 6.8 Hz, minor), 5.93 (1H, d, *J* = 16 Hz, minor), 5.74 (1H, m, major), 5.63 (1H, m, minor), 4.90-5.05 (4H, m, major & minor), 3.28 (1H, p, *J* = 7.2 Hz, minor), 2.81 (2H, m, minor), 2.61 (1H, dd, *J* = 16 Hz, *J* = 6.0 Hz, major) 2.52 (1H, dd, *J* = 16 Hz, *J* = 6.8 Hz, major), 2.37 (2H, t, *J* = 7.2 Hz, minor), 2.16 (1H, m, major), 1.91-2.10 (3H, m, major & minor), 1.57-1.78 (10H, m, major & minor), 1.36 (1H, m, major), 0.94-1.29 (10H, m, major & minor); ^{13}C NMR: δ 200.4, 199.5, 152.3, 144.2, 142.0, 137.4, 136.2, 134.6, 130.2, 128.8, 128.3, 128.2, 128.0, 127.5, 126.5, 126.2, 116.6, 116.2, 46.10, 42.55, 41.05, 40.59, 40.54, 40.40, 39.20, 35.90, 31.73, 30.06, 29.68, 26.71, 25.90, 25.68. LRMS (ESI+) Calc'd for $C_{20}H_{26}O$ $(M + Na)^+$: 305.2 Found $(M + Na)^+$: 305.2.

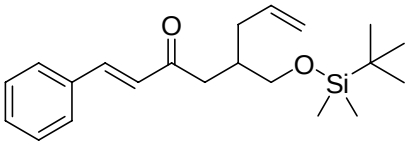
^1H NMR analysis of Crude Reaction Mixture (400 MHz, $CDCl_3$):



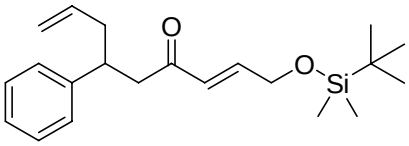
Vinyl CH
(minor + major
isomers)



(*E*)-5-*t*-butyl-1-phenylocta-1,7-dien-3-one. An oil. R_f = 0.24 (15:1 Hexanes:EtOAc); IR (neat): 3064 (m), 2959 (s), 2870 (m), 1953 (w), 1814 (w), 1689 (s), 1654 (s), 1608 (s), 1445 (s), 1363 (s), 1185 (m) cm^{-1}; ^1H NMR: δ 7.48-7.57 (3H, m), 7.37 (3H, m), 6.73 (1H, d, *J* = 16 Hz), 5.72 (1H, m), 4.97 (1H, d, *J* = 17 Hz), 4.91 (1H, d, *J* = 10 Hz) 2.66 (1H, dd, *J* = 17 Hz, *J* = 4.0 Hz), 2.46 (1H, dd, *J* = 17 Hz, *J* = 6.0 Hz), 2.32-2.41 (1H, m), 2.13 (1H, m), 1.78 (1H, m), 0.89 (9H, s); ^{13}C NMR: δ 200.1, 141.6, 138.4, 134.5, 130.1, 128.7, 128.0, 126.3, 115.8, 42.47, 41.99, 35.65, 33.28, 27.50. LRMS (ESI+) Calc'd for $C_{18}H_{24}O$ $(M + Na)^+$: 279.2 Found $(M + Na)^+$: 279.2.

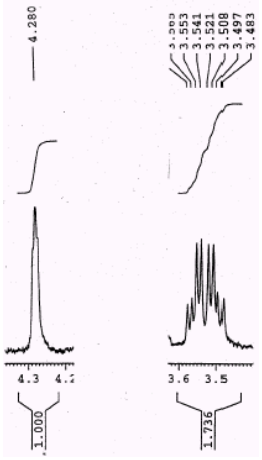


(*E*)-5-(*t*-butyldimethylsilyloxy)methyl)-1-phenylocta-1,7-dien-3-one. An oil. Regioisomers were separable using column chromatography (SiO_2/hexanes:EtOAc). R_f = 0.26 (14:1 Hexanes:EtOAc); IR (neat): 3076 (m), 2955 (s), 2924 (s), 2854 (s), 1689 (s), 1662 (s), 1611 (s), 1471 (m), 1363 (m), 1251 (s), 1103 (s) cm^{-1}; ^1H NMR: δ 7.49-7.59 (3H, m), 7.37 (3H, m), 6.72 (1H, d, *J* = 16 Hz), 5.77 (1H, m), 5.03 (2H, m), 3.56 (1H, dd, *J* = 10 Hz, *J* = 4.8 Hz), 3.51 (1H, dd, *J* = 10 Hz, *J* = 5.6 Hz), 2.77 (1H, dd, *J* = 16 Hz, *J* = 6.8 Hz), 2.53 (1H, dd, *J* = 16 Hz, *J* = 6.0 Hz), 2.11-2.31 (2H, m), 2.07 (1H, m), 0.87 (9H. s), 0.015 (3H, s), 0.0030 (3H, s); ^{13}C NMR: δ 200.2, 142.3, 136.5, 134.6, 130.3, 128.9, 128.2, 126.7, 116.6, 64.73, 41.91, 36.94, 35.63, 25.89, 18.26, -5.46. LRMS (ESI+) Calc'd for $C_{21}H_{32}O_2Si$ $(M + Na)^+$: 367.2 Found $(M + Na)^+$: 367.3.

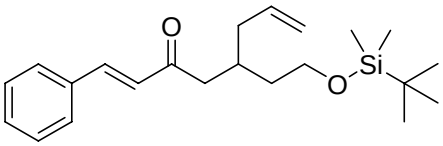


(*E*)-1-(*t*-butyldimethylsilyloxy)-6-phenylnona-2,8-dien-4-one. An oil. Regioisomers were separable using column chromatography (SiO_2/hexanes:EtOAc).R_f = 0.18 (14:1 Hexanes:EtOAc); IR (neat): 3056 (m), 3029 (m), 2948 (s), 2924 (s), 2858 (s), 1697 (s), 1670 (s), 1635 (s), 1468 (m), 1359 (m), 1251 (s), 1134 (s) cm^{-1}; ^1H NMR: δ 7.20-7.30 (2H, m), 7.10-7.20 (3H, m), 6.76 (1H, dt, *J* = 16 Hz, *J* = 3.2 Hz), 6.28 (1H, d, *J* = 16 Hz), 5.63 (1H, m), 4.96 (1H, d, *J* = 16 Hz), 4.93 (1H, d, *J* = 10 Hz), 4.28 (2H, m), 3.31 (1H, p, *J* = 7.2 Hz), 2.85 (2H, m), 2.37 (2H, t, *J* = 7.2 Hz), 0.90 (9H, s), 0.045 (6H, s); ^{13}C NMR: δ 198.9, 145.3, 144.2, 136.2, 128.4, 127.9, 127.5, 126.3, 116.7, 62.23, 46.70, 40.81, 40.60, 25.84, 18.35, -5.45. LRMS (ESI+) Calc'd for $C_{21}H_{32}O_2Si$ $(M + Na)^+$: 367.2 Found $(M + Na)^+$: 367.3.

^1H NMR analysis of Crude Reaction Mixture (400 MHz, $CDCl_3$):

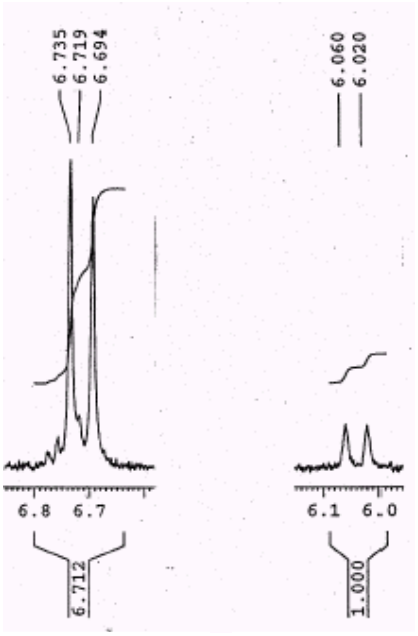


Carbinol (minor isomer) Carbinol (major isomer)



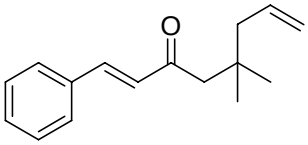
(*E*)-5-(2-*t*-butyldimethylsilyloxy)ethyl)-1-phenylocta-1,7-dien-3-one. An oil. Regioisomers were separable using iterative column chromatography (SiO_2/hexanes:EtOAc). R_f (major) = 0.25 (15:1 Hexanes:EtOAc); R_f (minor) = 0.18 (15:1 Hexanes:EtOAc); IR (major, neat): 3068 (m), 2948 (s), 2932 (s), 2847 (s), 1953 (w), 1817 (w), 1682 (s), 1658 (s), 1611 (s), 1468 (m), 1328 (m), 1254 (s) cm^{-1}; ^1H NMR: δ (major) 7.45-7.57 (3H, m), 7.37 (3H, m), 6.72 (1H, d, *J* = 16 Hz), 5.76 (1H, m), 5.02 (2H, m), 3.66 (2H, m) 2.63 (2H, d, *J* = 6.4 Hz), 2.27 (1H, heptet, *J* = 6.4 Hz), 2.01-2.20 (2H, m), 1.45-1.67 (2H, m), 0.86 (9H, s), 0.020 (6H, s); δ (minor) 7.21-7.30 (2H, m), 7.10-7.20 (3H, m), 6.74 (1H, dt, *J* = 16 Hz, *J* = 7.2 Hz), 6.04 (1H, d, *J* = 16 Hz), 5.62 (1H, m), 4.96 (1H, d, *J* = 15 Hz), 4.93 (1H, d, *J* = 10 Hz), 3.67 (2H, t, *J* = 6.4 Hz), 3.30 (1H, p, *J* = 7.2 Hz), 2.83 (2H, m), 2.35 (4H, m), 0.87 (9H, s), 0.023 (6H, s); ^{13}C NMR: δ (major) 200.1, 142.3, 136.4, 134.6, 130.3, 128.9, 128.2, 126.5, 116.9, 61.20, 45.29, 38.42, 36.61, 31.23, 25.92, 18.26, -5.34. LRMS (ESI+) Calc'd for $C_{22}H_{34}O_2Si$ $(M + Na)^+$: 381.2 Found $(M + Na)^+$: 381.3.

^1H NMR analysis of Crude Reaction Mixture (400 MHz, $CDCl_3$):

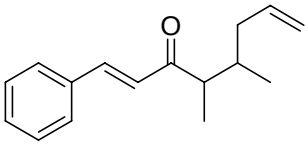


Vinyl CH (minor + major isomers)

Vinyl CH (minor isomer)



(*E*)-5,5-dimethyl-1-phenylocta-1,7-dien-3-one. An oil. R_f = 0.26 (14:1 Hexanes:EtOAc); IR (neat): 3072 (m), 2959 (s), 2862 (s), 1950 (w), 1814 (w), 1685 (s), 1651 (s), 1608 (s), 1440 (s), 1332 (s), 1200 (s) cm^{-1}; ^1H NMR: δ 7.52 (2H, m), 7.49 (1H, d, *J* = 16 Hz), 7.36 (3H, m), 6.72 (1H, d, *J* = 16 Hz), 5.84 (1H, m), 5.05 (2H, m), 2.52 (2H, s), 2.12 (2H, d, *J* = 7.6 Hz), 1.03 (6H, s); ^{13}C NMR: δ 200.0, 141.9, 135.0, 134.6, 130.3, 128.9, 128.3, 127.6, 117.7, 51.49, 46.86, 34.30, 27.40. LRMS (ESI+) Calc'd for $C_{16}H_{20}O$ $(M + Na)^+$: 251.1 Found $(M + Na)^+$: 251.2.



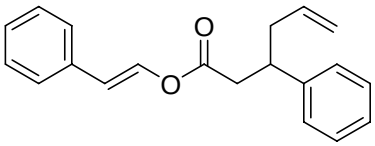
(*E*)-4,5-dimethyl-1-phenylocta-1,7-dien-3-one. An oil. Isolated as an inseparable mixture of diastereomers using column chromatography (SiO_2/hexanes:EtOAc). R_f = 0.26 (14:1 Hexanes:EtOAc); IR (neat): 3076 (m), 2967 (s), 2920 (s), 1942 (w), 1821 (w), 1685 (s), 1651 (s), 1608 (s), 1449 (s), 1324 (s), 1188 (m) cm^{-1}; ^1H NMR: δ 7.58 (2H, d, *J* = 16 Hz, major & minor), 7.54 (4H, m, major & minor), 7.37 (6H, major & minor), 6.80 (1H, d, *J* = 16 Hz, major), 6.79 (1H, d, *J* = 16 Hz, minor), 5.76 (2H, m, major & minor), 5.02 (4H, m, major & minor), 2.80 (1H, p, *J* = 6.4 Hz, minor), 2.73 (1H, p, *J* = 6.8 Hz, major), 1.85-2.28 (6H, m, major & minor), 1.13 (3H, d, *J* = 6.8 Hz, major), 1.06 (3H, d, *J* = 7.2 Hz, minor), 0.92 (3H, d, *J* = 6.4 Hz, major), 0.84 (3H, d, *J* = 6.4 Hz, minor); ^{13}C NMR: δ 203.6, 203.5, 142.3, 136.9, 136.65, 136.59, 134.6, 130.4, 130.3, 128.9, 128.3, 128.27, 125.3, 125.1, 116.5, 116.4, 49.60, 48.47, 39.67, 37.42, 35.31, 34.59, 17.72, 15.25, 13.65, 11.35. LRMS (ESI+) Calc'd for $C_{16}H_{20}O$ $(M + Na)^+$: 251.1 Found $(M + Na)^+$: 251.1.

Procedure for the ring closing metathesis in Scheme 2.

To 47.9 mg (0.173 mmol) of 1,5-diphenyl-1,7-octadien-3-one was added 2.2 mg (0.0035 mmol) of Hoveyda-Grubb's 2nd generation catalyst (Garber, S. B.; Kingbury, J. S.; Gray, B. L.; Hoveyda, A. H. *J. Am. Chem. Soc.* **2000**, *122*, 8168) in a dry-box under Ar. This was diluted with 5.7 mL of dry, degassed CH_2Cl_2. A magnetic stir bar was added, followed by a septum, and the reaction was removed from the dry-box and stirred at room temperature under N_2. After 1h, 3 drops (22 G needle) of *t*-butyl vinyl ether was added and volatiles removed under reduced pressure. Silica gel chromatography (pentane/Et_2O) of the crude mixture afforded 28.1 mg (94%) of 5-phenylcyclohex-2-enone whose spectral data was consistent with the literature (Rutherford, A. P.; Gibb, C. S.; Hartley, R. C.; Goodman, J. M. *J. Chem. Soc. Perkin Trans. 1* **2001**, 1051.).

Procedure for the Sn-catalyzed Baeyer-Villager oxidation in Scheme 2.

For lead reference, see: Göttlich, R.; Yamakoshi, K.; Sasai, H.; Shibasaki, M. *Synlett* **1997**, 971. In a 2-dram vial with magnetic stir bar in a dry-box under Ar was weighed ~30 mg of crushed 4 Å molecular sieves. Next, 33.8 μL (0.0338 mmol) of a 1 M solution of (±)-*trans*-1,2-diaminocyclohexane in CH_2Cl_2 was added by syringe followed by dilution with 0.32 mL of CH_2Cl_2. Next, 33.8 μL (0.0338 mmol) of a 1 M solution of $SnCl_4$ in CH_2Cl_2 was added and the vial was capped with a septum, removed from the dry-box and cooled to 0 °C (ice/brine). TMS_2O_2 was added dropwise as a 1 M solution in CH_2Cl_2 (0.27 mL, 0.27 mmol). After stirring for 10 min at this temperature, 37.3 mg (0.135 mmol) of 1,5-diphenyl-1,7-octadien-3-one was added in 0.59 mL CH_2Cl_2 via canula. The reaction became a blue-gray color and was subsequently warmed to room temperature and stirred for 15 h. Sodium sulfite (41 mg) was then added, and the reaction stirred for an additional 3 h. Finally, the reaction was filtered through a pad of silica gel using EtOAc and concentrated under reduced pressure. Silica gel chromatography (hexanes/EtOAc) of the crude material afforded 31.5 mg (80 %) of (*E*)-styryl-3-phenylhex-5-enoate as a white solid.



(*E*)-styryl-3-phenylhex-5-enoate. A white solid. mp = 66-70°C. R_f = 0.24 (30:1 Hexanes:EtOAc); IR (CH_2Cl_2 solution): 3087 (m), 3024 (m), 2917 (m), 1747 (s), 1652 (m), 1495 (m), 1212 (m), 1142 (s) cm^{-1}; ^1H NMR: δ 7.74 (1H, d, *J* = 13 Hz), 7.24-7.39 (6H, m), 7.10-7.24 (4H, m), 6.03 (1H, d, *J* = 13 Hz), 5.66 (1H, m), 5.02 (2H, m), 3.26 (1H, p, *J* = 7.2 Hz), 2.81 (1H, dd, *J* = 16 Hz, *J* = 6.4 Hz), 2.68 (1H, dd, *J* = 16 Hz, *J* = 8.4 Hz), 2.42 (2H, m); ^{13}C NMR: δ 169.3, 143.1, 136.1, 135.6, 134.0, 128.6, 128.5, 127.3, 126.7, 126.1, 117.1, 115.2, 41.56, 40.61, 40.26. Note that the peak at 127.3 ppm was 2 overlapping signals that could not be resolved at higher frequency. ^{13}C analysis in C_6D_6 (125 MHz) resolved these peaks. This data (C_6D_6 referenced at 128.39 relative to tetramethylsilane) was: 169.3, 143.9, 137.0, 136.4, 134.9, 129.2, 129.1, 128.1, 127.8, 127.3, 126.8, 117.4, 115.7, 42.22, 41.12, 40.63. LRMS (ESI+) Calc'd for $C_{20}H_{20}O_2$ $(M + Na)^+$: 315 Found $(M + Na)^+$: 315.

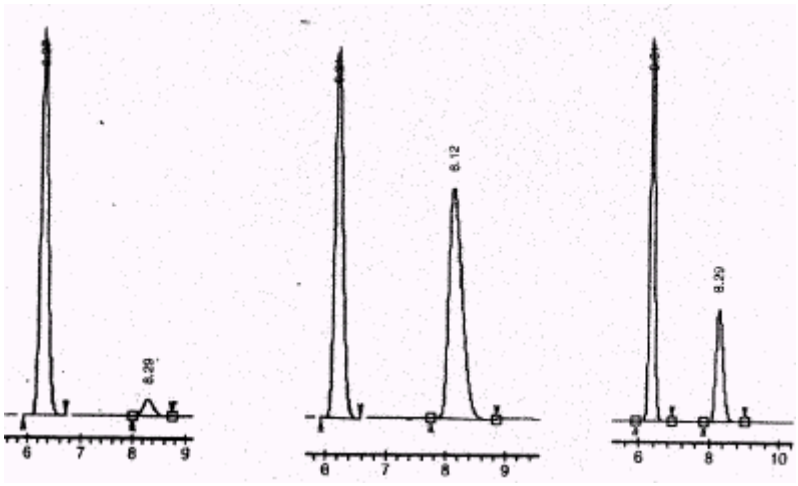
Procedure for the hydrolysis of (*E*)-styryl-3-phenylhex-5-enoate (Scheme 2).

To a solution of 26.3 mg (0.900 mmol) of (*E*)-styryl-3-phenylhex-5-enoate in 0.68 mL of THF was added 0.22 mL of water, and the reaction then subsequently cooled to 0 °C. $LiOH•H_2O$ (7.6 mg, 0.18 mmol) was then added, and the reaction was stirred at this temperature and monitored by TLC. After complete consumption of the starting material (2-2.5 h), the reaction was acidified with 1 M HCl and extracted with EtOAc (3x). The organic layers were combined, washed with brine, and dried over anhydrous Na_2SO_4. Volatiles were removed under reduced pressure, and the product was purified using silica gel chromatography (1% AcOH in CH_2Cl_2/Et_2O, R_f = 0.25 in 1% AcOH in 20:1 CH_2Cl_2:Et_2O) to give 16.5 mg (96%) of 3-phenyl-hex-5-enoic acid after removal of AcOH by azeotropic distillation with toluene using a rotary evaporator followed by removal of toluene via azeotropic distillation with CH_2Cl_2. Spectral data was consistent with the literature (Allin, S. M.; Essat, M.; Pita, C. H.; Baird, R. D.; McKee, V.; Elsegood, M.; Edgar, M.; Andrews, D. M.; Shah, P.; Aspinall, I. *Org. Biomol. Chem.* **2005**, *3*, 809.).

Procedure for the asymmetric conjugate allylation of dibenzylidene acetone (Scheme 4).

An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 2.5 mg (0.0027 mmol) of tris(dibenzylideneacetone)dipalladium, 6.3 mg (0.0064 mmol) of chiral ligand (for ligand synthesis see: Woodward, A. R.; Burks, H. E.; Chan, L. M.; Morken, J. P. *Org. Lett.* **2005**, *7*, 5505), and 0.71 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 45 min. Next, 19.8 mg (0.118 mmol) of allylboronic acid pinacol ester was added followed by 25.0 mg (0.107 mmol) of dibenzylidene acetone. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 24 h. After this time period, water was added and the mixture transferred to a separatory funnel with CH_2Cl_2. After gently swirling the layers, the organic layer was collected and the aqueous layer washed with CH_2Cl_2 (2x). The combined organic layers were dried with Na_2SO_4, and volatiles were removed under reduced pressure. Silica gel chromatography (hexanes/EtOAc) afforded 26.3 mg (83%) of 1,5-diphenyl-1,7-octadien-3-one whose spectral data has been reported previously (Mandal, S. K.; Amin, S. R.; Crowe, W. E. *J. Am. Chem. Soc.* **2001**, *123*, 6457.). The optical rotation was: $[\alpha]^{20}_D = +13^o$ (*c* = 1.0, $CHCl_3$). The absolute configuration was determined by performing the ring-closing metathesis shown in Scheme 2 on the chiral material and comparing the optical rotation ($[\alpha]^{20}_D = -42^o$ (*c* = 0.5, $CHCl_3$))with the known value (Hareau, G. P-J.; Koiwa, M.; Hikichi, S; Sato, F. *J. Am. Chem. Soc.* **1999**, *121*, 3640). The enantiomeric excess was determined using chiral SFC (data shown below).

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 3 mL/min, 4% MeOH) analysis of conjugate allylation product:

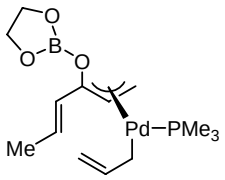


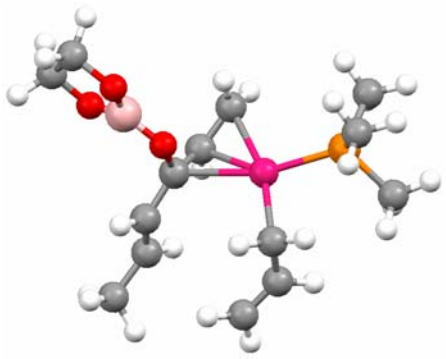
Allylation product | Racemic | Allylation product + racemic coinjection

#	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area [%]
1	UNKNOWN	5.91	6.27	6.72	0	94.41	24264.9	3924	94.412
2	UNKNOWN	8	8.29	8.75	0	5.59	1032.6	232.3	5.588
Total						100	25297.5	4156.2	100

Computational Methods: DFT calculations were performed using B3LYP[1,2] with the basis set of Stuttgart RSC 1997 ECP[3] for Pd, 6-311+G*[4] for others. All calculations were conducted using Gaussian 03 C02 package[5] with tight SCF convergence and ultra fine integration grids on the 128-CPU SGI Altix 3700 SMP machine[6] at University of North Carolina at Chapel Hill. In search for the transition state structure, a single-point frequency calculation has been performed to ensure that the final structure obtained (i) has only one imaginary frequency and (ii) the vibration mode of the negative frequency corresponds to the anticipated bond formation.

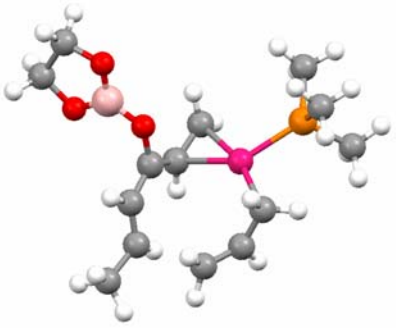
Reactant (3,3′-Elmination):

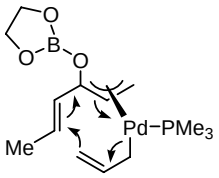




C	-2.21400	4.32800	0.38200
H	-1.45700	5.10300	0.21800
H	-2.84100	4.67800	1.21100
H	-2.84300	4.27600	-0.51100
B	-3.03100	-1.03000	-0.44800
C	-1.33600	0.53100	0.28900
C	3.16900	-2.19400	-1.51400
C	3.32900	-2.44700	1.34000
C	4.53300	-0.19900	0.04200
C	-0.74800	0.10100	1.49100
C	-5.12300	-1.49800	0.29100
C	-4.73400	-2.47100	-0.84800
C	-1.84100	1.87100	0.02100
C	-0.18400	-1.17200	1.69100
C	-1.58900	3.00300	0.69800
C	1.25700	3.71300	-0.75200
C	1.57700	1.30800	-1.42700
C	1.96800	2.57100	-0.79200
H	3.09000	-1.54300	-2.38600
H	4.13000	-2.71600	-1.54300
H	2.36300	-2.92800	-1.57300
H	2.52200	-3.18100	1.36900
H	4.27700	-2.96500	1.16700
H	3.36800	-1.95300	2.31200
H	4.60100	0.36200	0.97700
H	5.41800	-0.83600	-0.05300
H	4.52400	0.51800	-0.78000
H	-5.44200	-2.01000	1.20200
H	-5.90700	-0.79700	-0.00900
H	-4.59100	-3.49500	-0.48800
H	-5.45900	-2.48500	-1.66300
H	-2.49300	1.92600	-0.85000
H	-0.91100	2.98700	1.54800
H	2.38900	0.86900	-2.01100
H	0.69700	1.41000	-2.06700
H	2.93300	2.57100	-0.28000
H	0.24600	-1.39700	2.66000
H	1.62200	4.59000	-0.22600
H	0.30700	3.80900	-1.26600
H	-0.57000	0.87000	2.23700
H	-0.54600	-2.02900	1.13300
O	-1.81600	-0.44600	-0.60200
O	-3.48000	-1.97700	-1.34000
O	-3.92400	-0.75700	0.56300
P	2.98200	-1.19700	0.02600
Pd	1.00800	-0.03800	0.15400

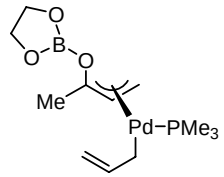
Transition State (3,3′-Elmination):

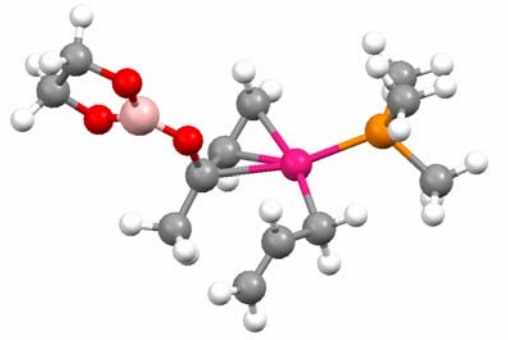




C	2.45700	4.27900	-0.05900
H	1.85300	5.18600	-0.13400
H	3.30900	4.40800	-0.73900
H	2.86000	4.22600	0.95700
B	3.04200	-1.20200	0.38000
C	1.51600	0.55600	-0.28200
C	-3.44400	-1.59400	1.83400
C	-2.87200	-2.95600	-0.61600
C	-4.61600	-0.68600	-0.62300
C	0.71200	0.27000	-1.40200
C	5.12200	-1.80300	-0.29500
C	4.57700	-2.83300	0.72500
C	2.01100	1.81300	0.11200
C	0.06800	-0.96100	-1.64700
C	1.68100	3.04000	-0.41200
C	-0.40700	3.57300	0.74500
C	-2.01800	1.66800	1.11000
C	-1.51300	2.81700	0.42300
H	-3.66000	-0.65700	2.35000
H	-4.31300	-2.25300	1.91200
H	-2.59600	-2.06300	2.33700
H	-2.00500	-3.45900	-0.18500
H	-3.76800	-3.54900	-0.40400
H	-2.72600	-2.90500	-1.69600
H	-4.52800	-0.57400	-1.70600
H	-5.41900	-1.39600	-0.40400
H	-4.88200	0.28700	-0.20700
H	5.44800	-2.25900	-1.23100
H	5.94600	-1.21000	0.11300
H	4.36000	-3.80000	0.26100
H	5.25000	-2.99400	1.56900
H	2.66000	1.79600	0.98600
H	1.10800	3.08700	-1.33200
H	-3.10400	1.57800	1.10000
H	-1.60100	1.47800	2.10000
H	-2.08800	3.14300	-0.44500
H	-0.36600	-1.12500	-2.62800
H	-0.27000	4.53500	0.26900
H	0.10700	3.42900	1.68900
H	0.57500	1.07900	-2.11200
H	0.40500	-1.85900	-1.14000
O	1.88000	-0.52200	0.52900
O	3.35000	-2.26400	1.20000
O	4.01300	-0.92900	-0.55600
P	-3.00000	-1.24800	0.07500
Pd	-1.22500	0.15700	-0.24700

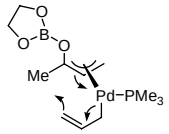
Reactant (1,2 addition to simple enone):

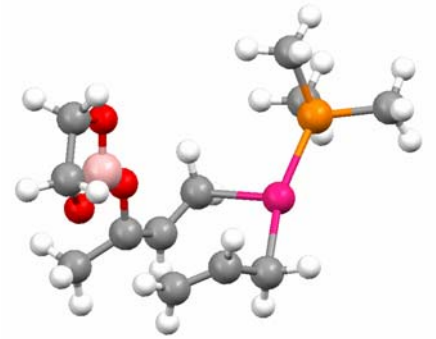




C	1.28100	0.44700	-0.93400
C	0.66100	-0.55700	-1.69600
C	0.11200	-1.73500	-1.15900
H	-0.39100	-2.42200	-1.82900
H	0.52800	-2.17900	-0.26000
B	3.14300	-0.35600	0.40500
O	3.68300	-0.66400	1.63300
O	4.01400	-0.55800	-0.64400
Pd	-0.97000	0.07900	-0.36400
P	-2.97800	-0.82600	0.30900
C	-3.19000	-0.93900	2.13900
H	-3.07700	0.05100	2.58400
H	-4.17200	-1.34100	2.40600
H	-2.41600	-1.58400	2.55700
C	-3.38400	-2.54600	-0.22700
H	-2.61500	-3.23200	0.13100
H	-4.35600	-2.87000	0.15600
H	-3.39700	-2.59600	-1.31700
C	-4.48700	0.09700	-0.21400
H	-4.55700	0.09600	-1.30300
H	-5.39400	-0.35300	0.20000
H	-4.42000	1.13400	0.11800
C	5.28300	-0.93200	-0.08600
H	5.66800	-1.80200	-0.62200
H	5.98200	-0.10100	-0.21700
C	4.98400	-1.22100	1.40500
H	4.95100	-2.29300	1.62100
H	5.70000	-0.75000	2.08200
C	-1.56200	2.01900	0.38300
H	-2.35600	1.84600	1.11800
H	-1.99700	2.55300	-0.47000
C	-0.47800	2.78800	1.01500
H	-0.10300	2.38900	1.95900
C	0.09500	3.91100	0.55200
H	-0.23400	4.38100	-0.37200
H	0.89800	4.40500	1.09000
O	1.87000	0.09500	0.29200
H	0.40600	-0.28300	-2.71600
C	1.78800	1.72400	-1.52100
H	2.86800	1.66700	-1.70000
H	1.29300	1.93700	-2.47100
H	1.59900	2.56200	-0.84300

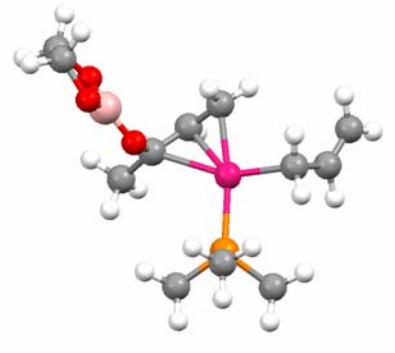
Transition State (1,2 addition to simple enone):





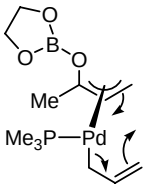
C	1.93000	-1.08900	1.16700
C	0.65200	-1.47000	1.56800
C	-0.49700	-0.67000	1.50400
H	-1.34600	-1.01400	2.09100
H	-0.33800	0.40500	1.51200
B	3.06400	0.92400	0.26600
O	3.01400	2.29500	0.12300
O	4.19600	0.36400	-0.29300
Pd	-1.58500	-0.69500	-0.44600
P	-3.22000	0.91600	0.10600
C	-2.68300	2.68800	0.11700
H	-2.30400	2.96200	-0.87000
H	-3.50200	3.36300	0.38400
H	-1.86700	2.82100	0.83100
C	-4.02600	0.78100	1.76700
H	-3.26700	0.84300	2.55000
H	-4.76300	1.57300	1.93100
H	-4.52100	-0.18800	1.85800
C	-4.70200	0.99900	-1.00200
H	-5.21000	0.03200	-1.00900
H	-5.41100	1.76800	-0.68300
H	-4.38400	1.21600	-2.02500
C	4.90700	1.41100	-0.97200
H	5.96800	1.34200	-0.72600
H	4.78700	1.27200	-2.05000
C	4.24900	2.71600	-0.46900
H	4.85300	3.21700	0.29400
H	4.04100	3.42500	-1.27200
C	-0.37400	-2.14000	-1.47000
H	-1.05600	-1.98900	-2.32100
H	-0.48700	-3.11700	-1.00000
C	0.94000	-1.65600	-1.62700
H	1.08200	-0.79600	-2.28000
C	2.02600	-2.09700	-0.88900
H	1.98900	-3.07800	-0.42800
H	3.01700	-1.71900	-1.10700
O	2.06700	0.27200	0.90400
H	0.54400	-2.51100	1.86100
C	3.15300	-1.77500	1.72600
H	3.43300	-1.31100	2.68000
H	2.94000	-2.82700	1.91900
H	4.01300	-1.71000	1.06000

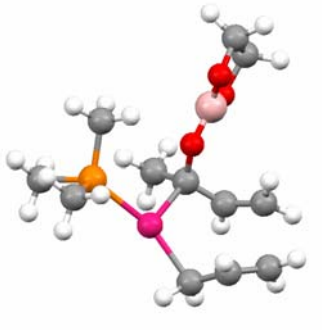
Reactant (1,4 addition to simple enone):



C	1.24400	-0.18500	1.10300
C	0.77800	1.11000	1.28600
C	0.48700	2.02000	0.23200
H	0.07900	2.98900	0.49400
H	1.07400	1.99600	-0.68200
B	3.22600	-0.25000	-0.28500
O	3.85300	-0.60000	-1.45800
O	4.05300	0.37900	0.61800
Pd	-0.92400	0.39700	0.05000
P	-2.15200	-1.57600	-0.16700
C	-3.95100	-1.47300	0.23400
H	-4.41800	-0.68400	-0.35700
H	-4.46400	-2.41800	0.02900
H	-4.08100	-1.22300	1.28900
C	-1.64900	-3.07900	0.78400
H	-1.70200	-2.87800	1.85600
H	-2.28800	-3.93600	0.55400
H	-0.61600	-3.33700	0.54000
C	-2.17500	-2.22800	-1.89300
H	-1.15700	-2.46400	-2.20800
H	-2.79200	-3.12700	-1.98000
H	-2.56300	-1.46500	-2.57000
C	5.37600	0.38400	0.05900
H	5.80000	1.38600	0.14900
H	5.99800	-0.31300	0.62800
C	5.18300	-0.06300	-1.41100
H	5.24700	0.77500	-2.11100
H	5.89200	-0.83300	-1.71900
C	-2.29100	1.56300	-1.09500
H	-2.89900	0.88200	-1.69800
H	-1.69800	2.18400	-1.77100
C	-3.12800	2.37500	-0.19100
H	-3.89900	1.83400	0.36200
C	-3.03500	3.69600	0.02700
H	-2.30000	4.30900	-0.48800
H	-3.68800	4.20600	0.72900
O	1.91400	-0.53100	-0.07900
H	0.40700	1.33900	2.28200
C	1.44000	-1.16900	2.21700
H	2.48700	-1.17500	2.54900
H	0.81400	-0.91800	3.07600
H	1.19600	-2.18400	1.89800

Transition State (1,4 addition to simple enone):





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C	1.42700	0.00000	0.00000
C	2.25600	1.09300	0.00000
H	3.30300	0.97200	-0.24400
H	1.85800	2.09500	-0.09800
B	-0.59700	1.67300	-1.70400
O	-1.09100	2.92900	-1.99700
O	-0.15600	0.99200	-2.81900
Pd	-0.59400	-0.02300	2.11800
P	-2.95300	0.30000	1.96800
C	-4.05700	-1.18600	1.88500
H	-3.82900	-1.86000	2.71400
H	-5.11500	-0.91100	1.93400
H	-3.88000	-1.73200	0.95600
C	-3.68700	1.37200	0.65500
H	-3.57500	0.89500	-0.32000
H	-4.75000	1.55700	0.83800
H	-3.15200	2.32100	0.61600
C	-3.59900	1.12300	3.49800
H	-3.12900	2.10100	3.61300
H	-4.68500	1.25500	3.46000
H	-3.34800	0.52500	4.37700
C	-0.21700	1.89800	-3.93000
H	0.80100	2.21000	-4.18400
H	-0.65100	1.38500	-4.79000
C	-1.08200	3.07400	-3.42300
H	-0.67000	4.05100	-3.68300
H	-2.11200	3.01900	-3.79100
C	1.29800	-0.13000	3.14900
H	1.89000	-1.01300	2.91000
H	0.69500	-0.26200	4.06100
C	1.93200	1.12600	2.99400
H	1.42200	1.99800	3.40000
C	3.05200	1.33500	2.22100
H	3.71700	0.50900	1.99800
H	3.47700	2.32700	2.11200
O	-0.60500	1.21300	-0.43100
H	1.87800	-0.98800	0.05700
C	-0.67700	-1.23100	-0.58500
H	-0.49900	-1.27600	-1.66600
H	-0.28100	-2.14100	-0.13100
H	-1.75500	-1.21300	-0.42300

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(4) R. Krishnan, J. S. Binkley, R. Seeger and J. A. Pople, *J. Chem. Phys.* **72**, 650 (1980).

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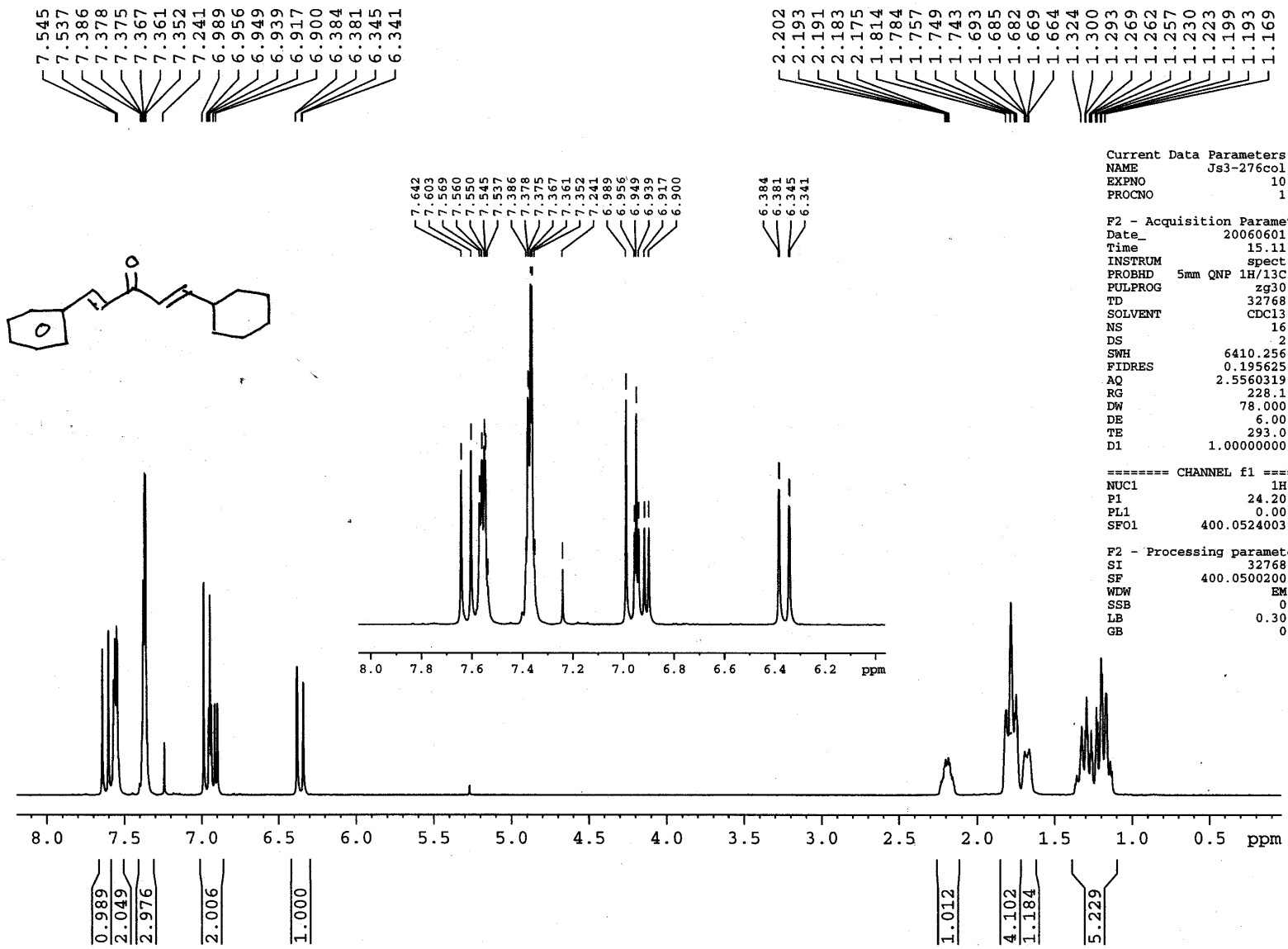
Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, and J. A. Pople, Gaussian, Inc., Wallingford CT, 2004.

(6) http://its.unc.edu/hpc/hardware/#Cedar/Cypress|outline

Js3-276 column



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EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

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INSTRUM	spect
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PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	228.1
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

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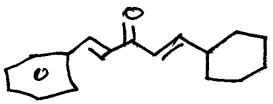
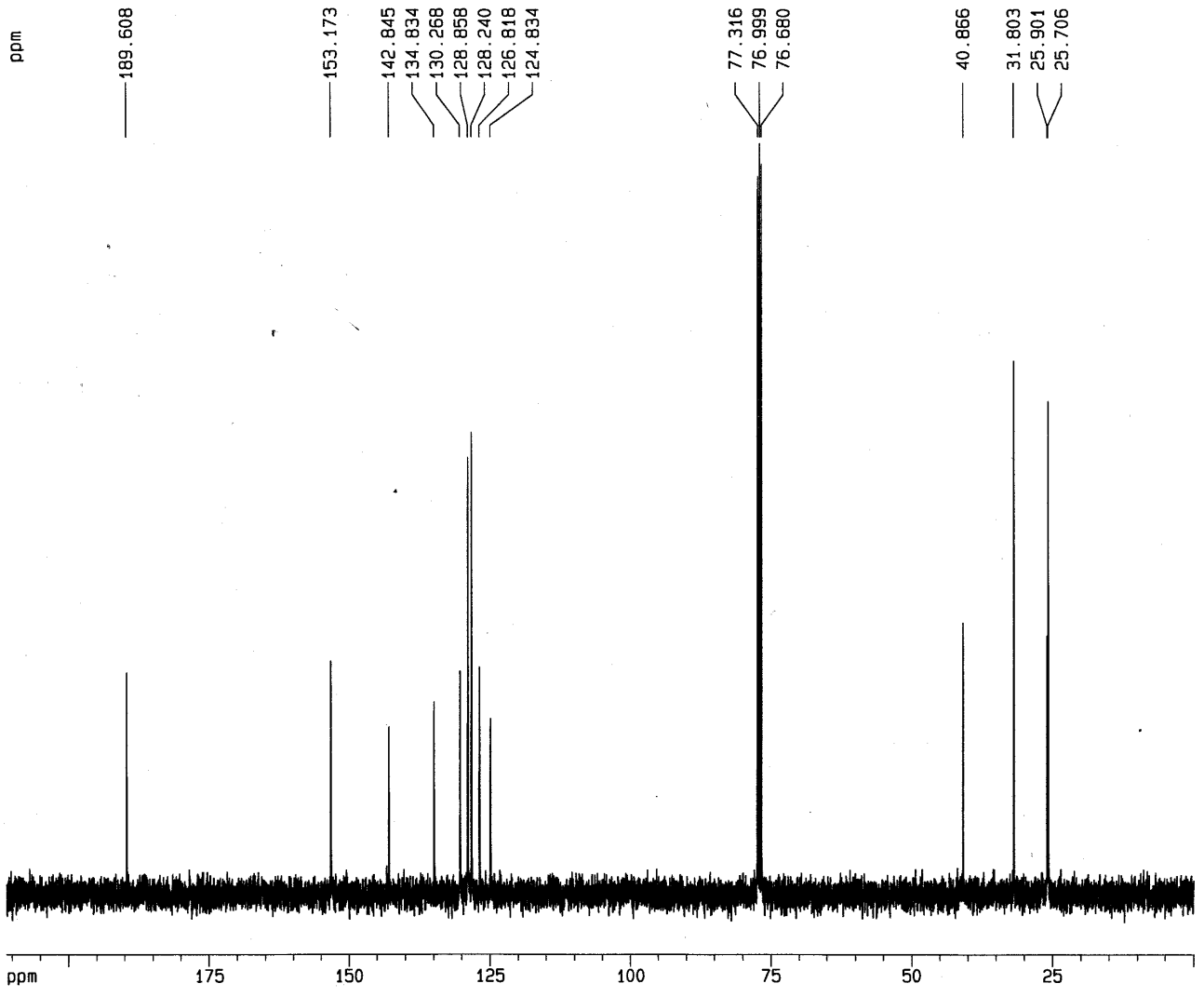


Js3-276 column



Current Data Parameters

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EXPNO	11
PROCNO	1

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TD	65536
SOLVENT	CDCl3
NS	81
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	3649.1
DW	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

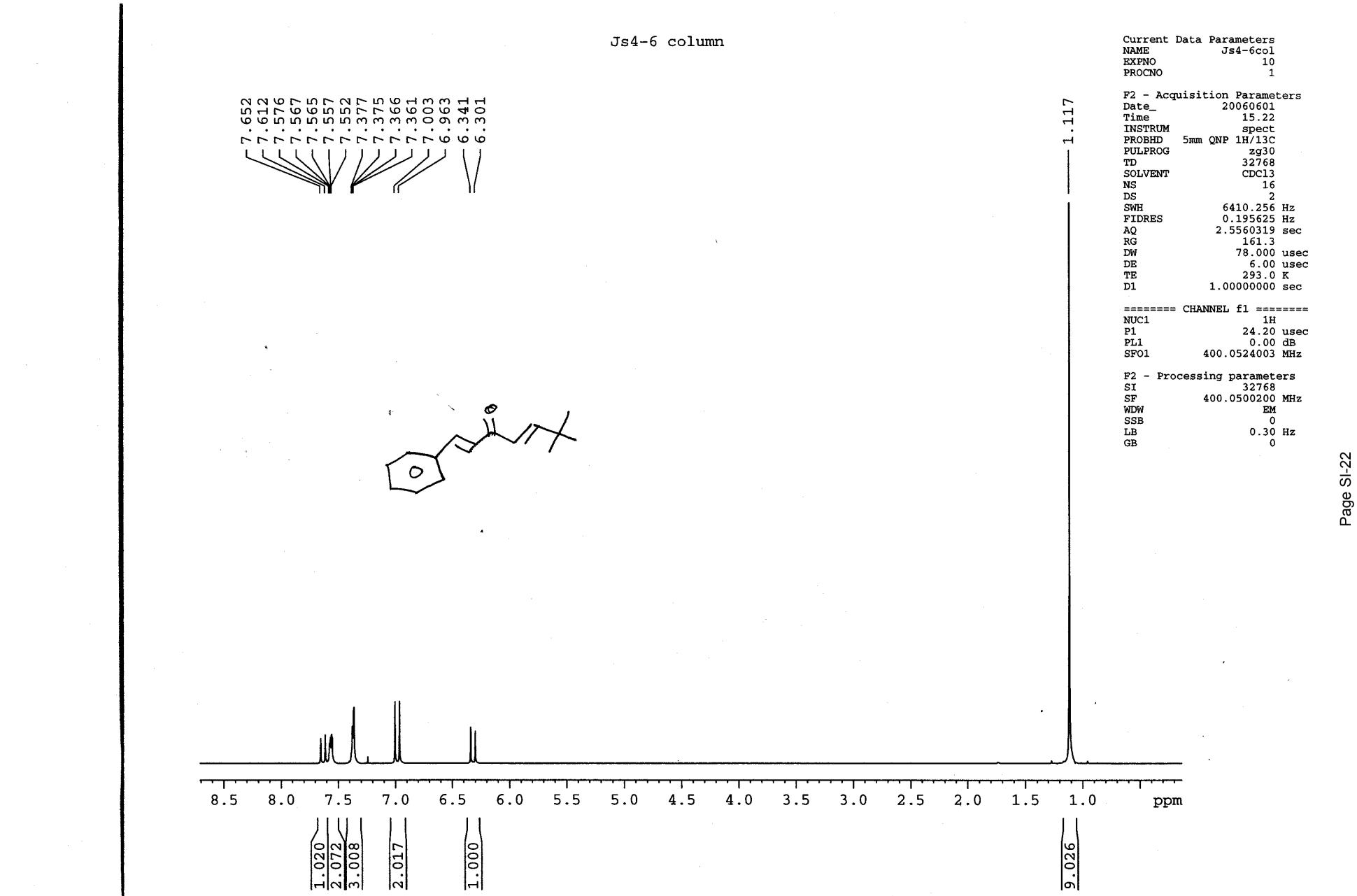
F2 - Processing parameters

SI	32768
SF	100.5926597 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	210.967 ppm
F1	21221.71 Hz
F2P	-0.092 ppm
F2	-9.28 Hz
PPMCM	10.55295 ppm/cm
HZCM	1061.54919 Hz/cm

Js4-6 column



Current Data Parameters

NAME	Js4-6col
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

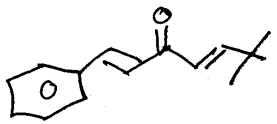
Date_	20060601
Time	15.22
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	161.3
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

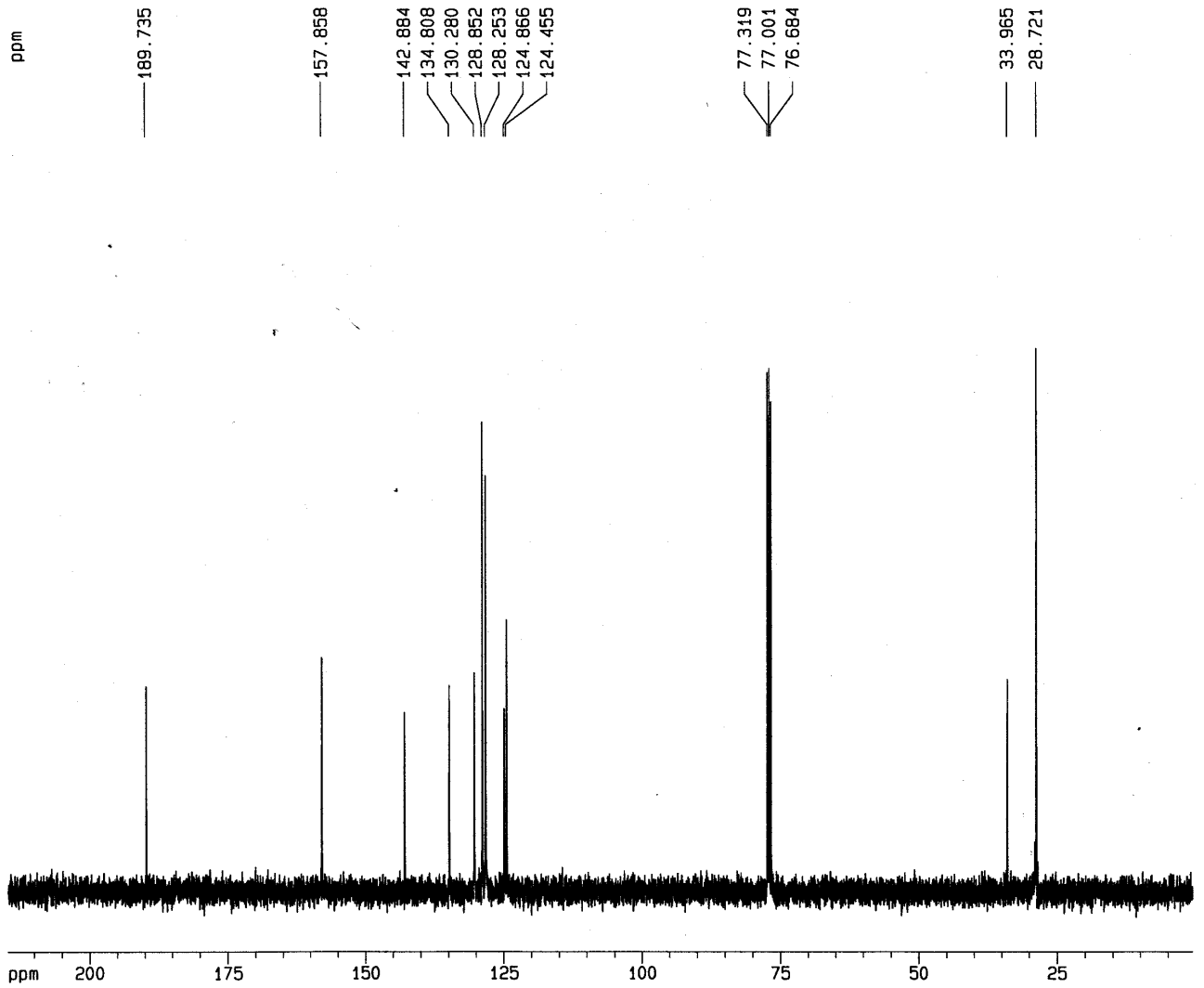
NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0



Js4-6 column



Current Data Parameters

NAME	Js4-6col
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060601
Time	15.24
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	51
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	6502
DW	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

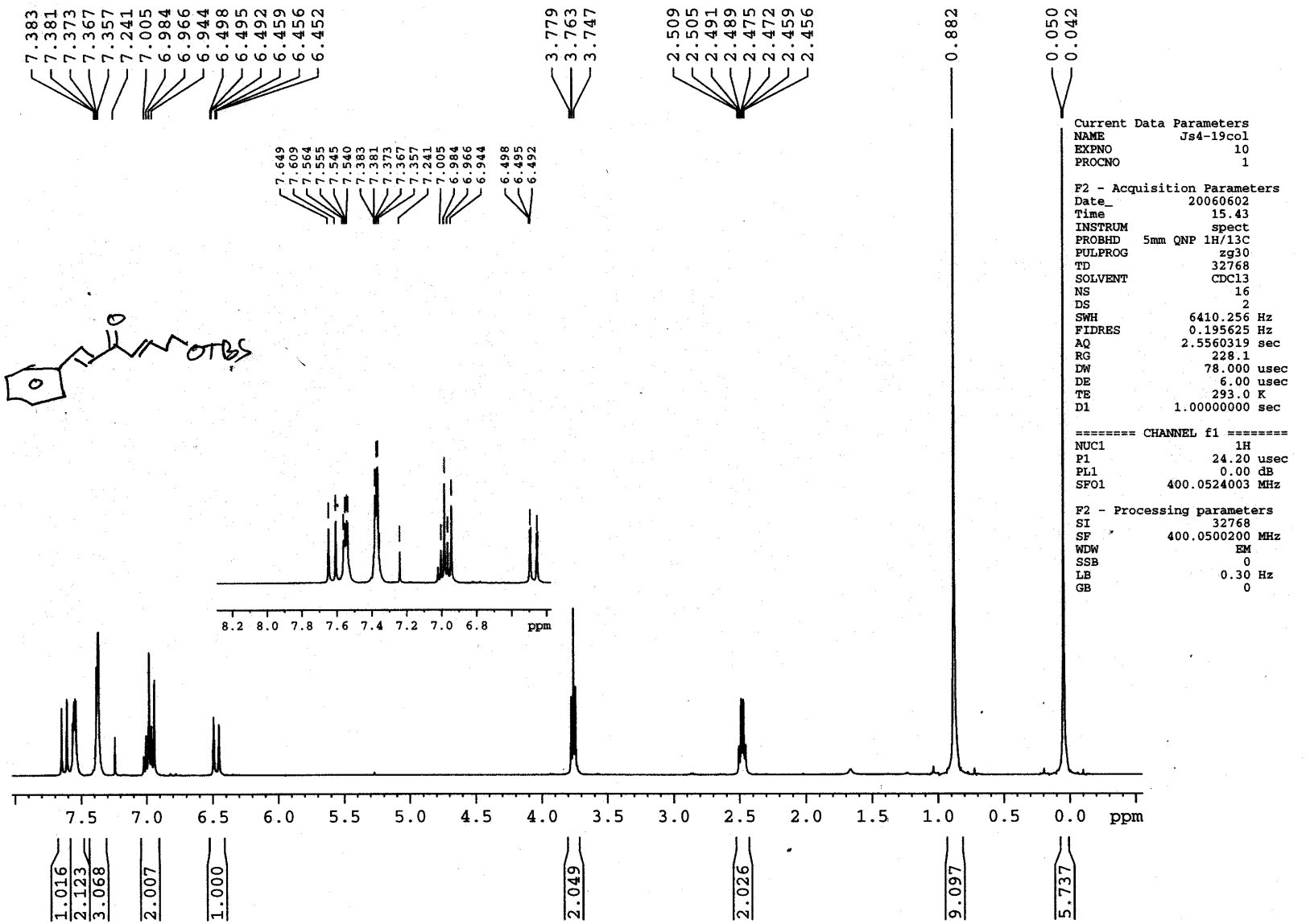
F2 - Processing parameters

SI	32768
SF	100.5926605 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	9.00 cm
F1P	214.715 ppm
F1	21598.80 Hz
F2P	0.583 ppm
F2	58.63 Hz
PPMCM	10.70663 ppm/cm
HZCM	1077.00867 Hz/cm

Js4-19 column



Current Data Parameters

NAME	Js4-19col
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

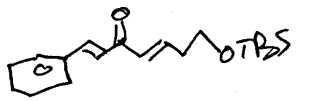
Date_	20060602
Time	15.43
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	228.1
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

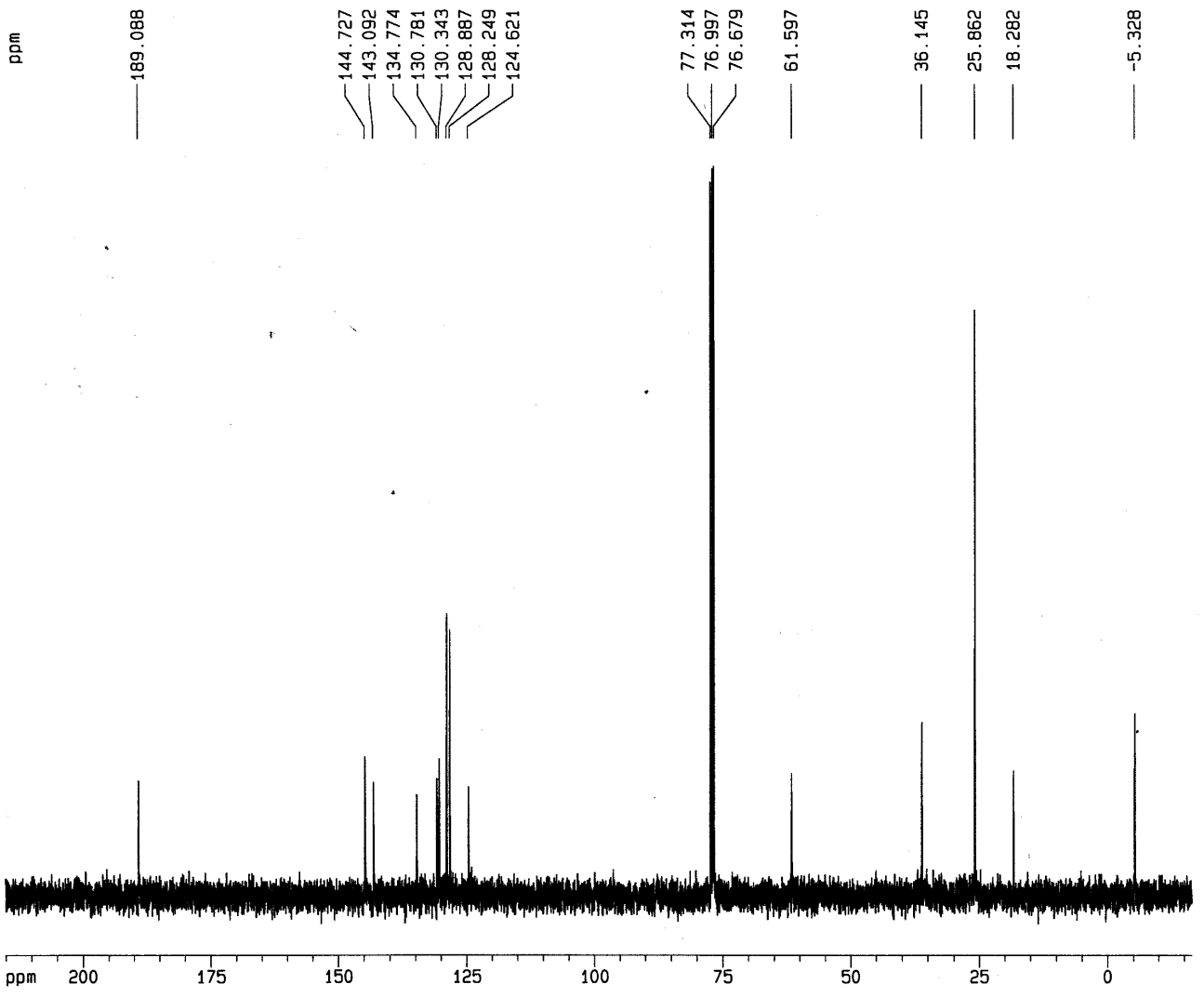
NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0

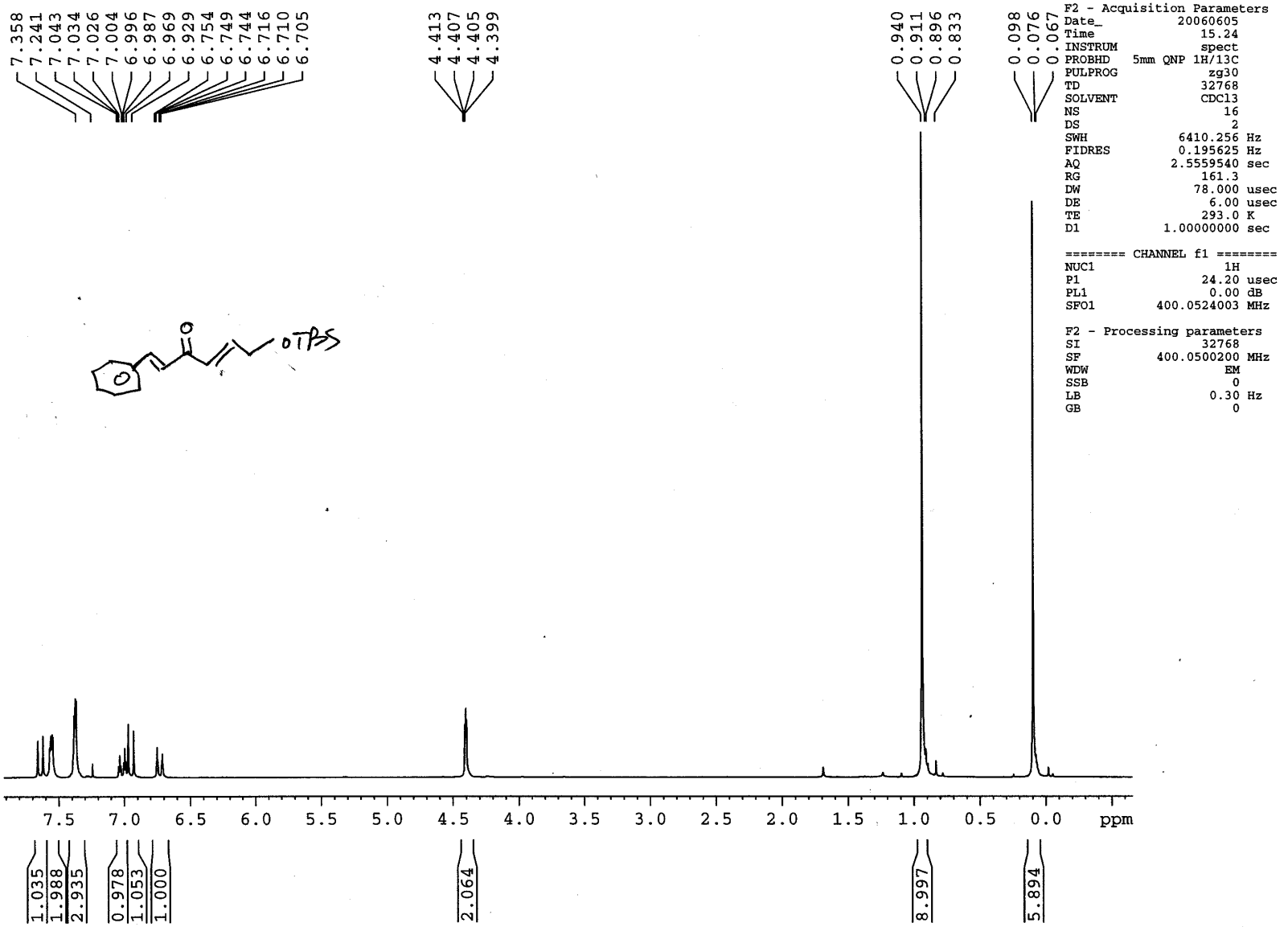


Js4-19 column




```
Current Data Parameters
NAME                  Js4-19col
EXPNO                        11
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20060602
Time                     15.45
INSTRUM                  spect
PROBHD     5mm QNP 1H/13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          70
DS                           2
SWH                26246.719 Hz
FIDRES             0.400493 Hz
AQ                 1.2485298 sec
RG                    7298.2
DW                   19.050 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.80000001 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    6.00 usec
PL1                   0.00 dB
SFO1              100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                  -6.00 dB
PL12                 13.80 dB
PL13                 14.50 dB
SFO2              400.0516002 MHz

F2 - Processing parameters
SI                        32768
SF                100.5926589 MHz
WDW                          EM
SSB                           0
LB                    1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                215.073 ppm
F1                 21634.76 Hz
F2P                -16.477 ppm
F2                 -1657.49 Hz
PPMCM              11.57751 ppm/cm
HZCM             1164.61243 Hz/cm
```

Js4-37 column



Current Data Parameters

NAME	Js4-37col
EXPNO	20
PROCNO	1

F2 - Acquisition Parameters

Date_	20060605
Time	15.24
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	161.3
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

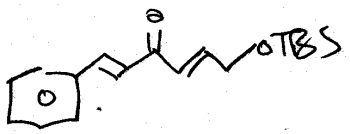
======== CHANNEL f1 ========

NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

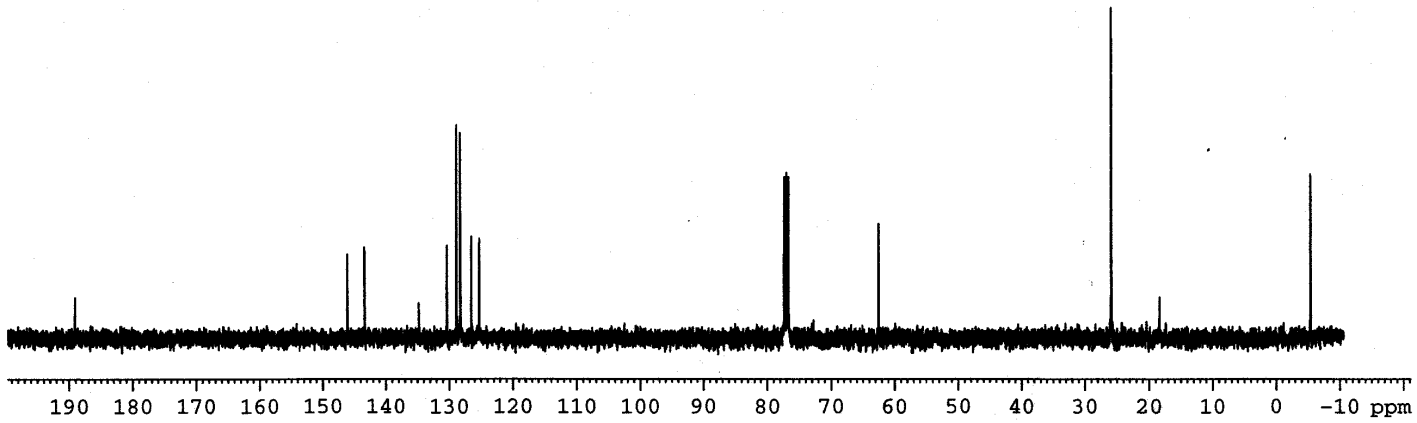
F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0

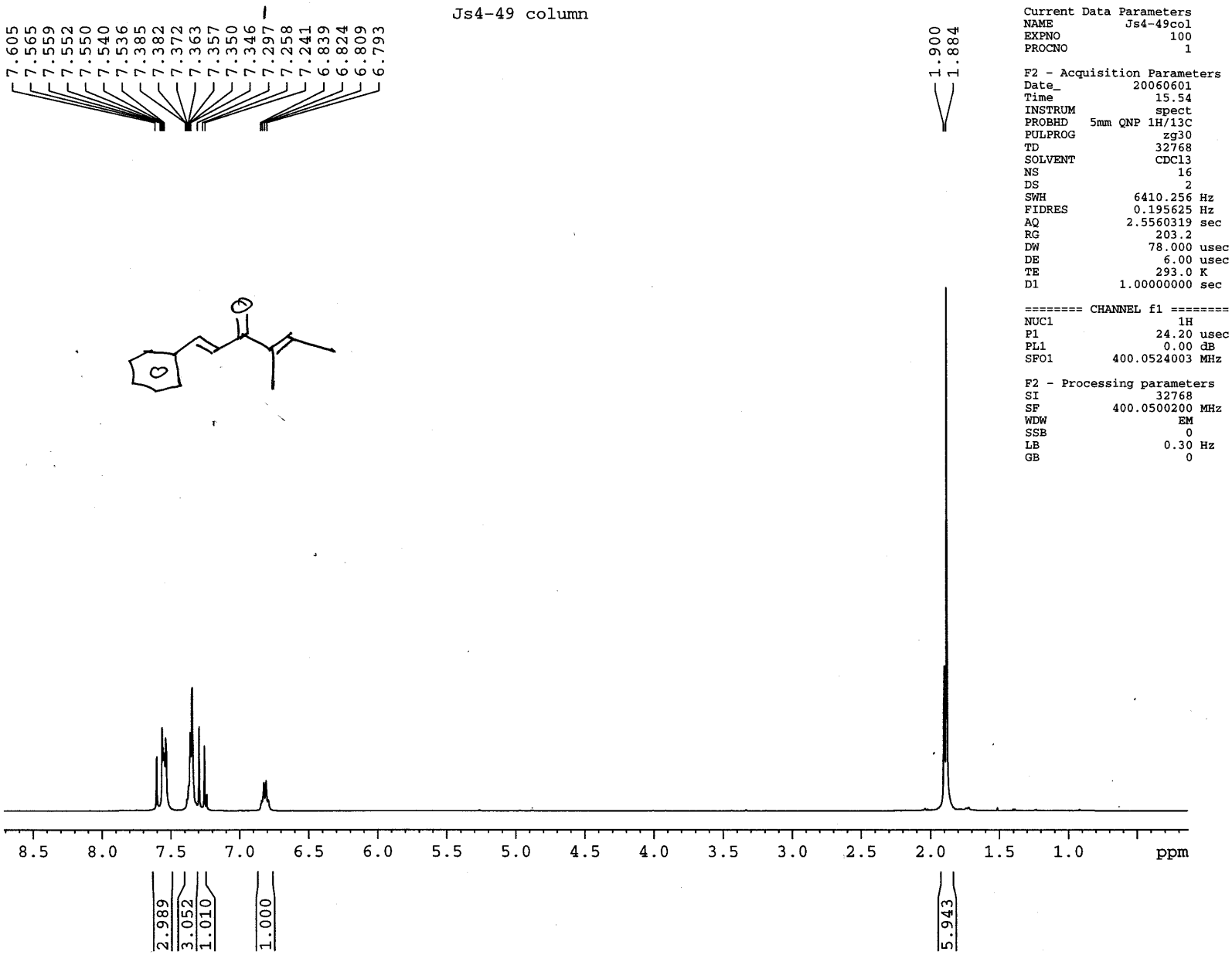
Js4-37 column

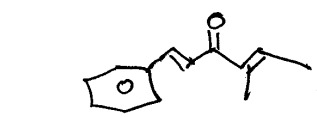


189.051
146.022
143.304
134.744
130.386
128.884
128.292
126.542
125.288
77.315
76.998
76.679
62.516
25.855
18.369
-5.407

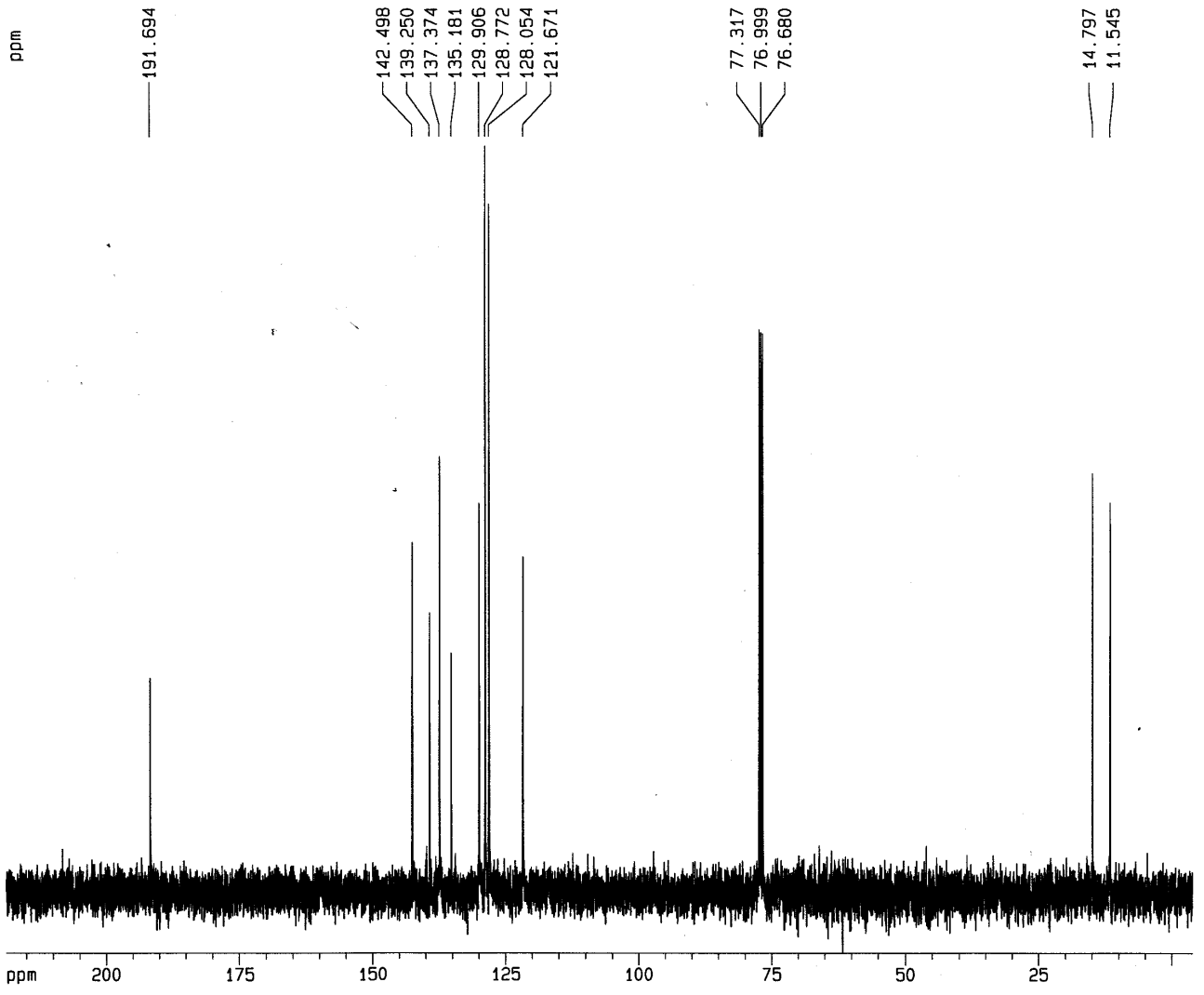



```
Current Data Parameters
NAME                  Js4-37col
EXPNO                       101
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20060605
Time                      17.58
INSTRUM                   spect
PROBHD    5 mm HR 13C/31
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                           114
DS                             2
SWH                    26178.010 Hz
FIDRES                 0.399445 Hz
AQ                     1.2517875 sec
RG                         13004
DW                        19.100 use
DE                        32.36 use
TE                        300.0 K
D1                     1.00000000 sec
d11                    0.03000000 sec
d12                    0.00002000 sec

======== CHANNEL f1 =======
NUC1                         13C
P1                          7.25 use
PL1                         0.00 dB
SFO1                100.5418136 MHz

======== CHANNEL f2 =======
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                      90.00 use
PL2                        -3.00 dB
PL12                       18.90 dB
PL13                       22.00 dB
SFO2                399.8015992 MHz

F2 - Processing parameters
SI                         65536
SF                  100.5297955 MHz
WDW                          EM
SSB                           0
```


Js4-49 column
7.605
7.565
7.559
7.552
7.550
7.540
7.536
7.385
7.382
7.372
7.363
7.357
7.350
7.346
7.297
7.258
7.241
6.839
6.824
6.809
6.793
1.900
1.884
Current Data Parameters
NAME Js4-49col
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060601
Time 15.54
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 203.2
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz
F2 - Processing parameters
SI 32768
SF 400.0500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.989
3.052
1.010
1.000
5.943



Js4-49 column



Current Data Parameters
NAME Js4-49col
EXPNO 101
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060601
Time 15.56
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 5792.6
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

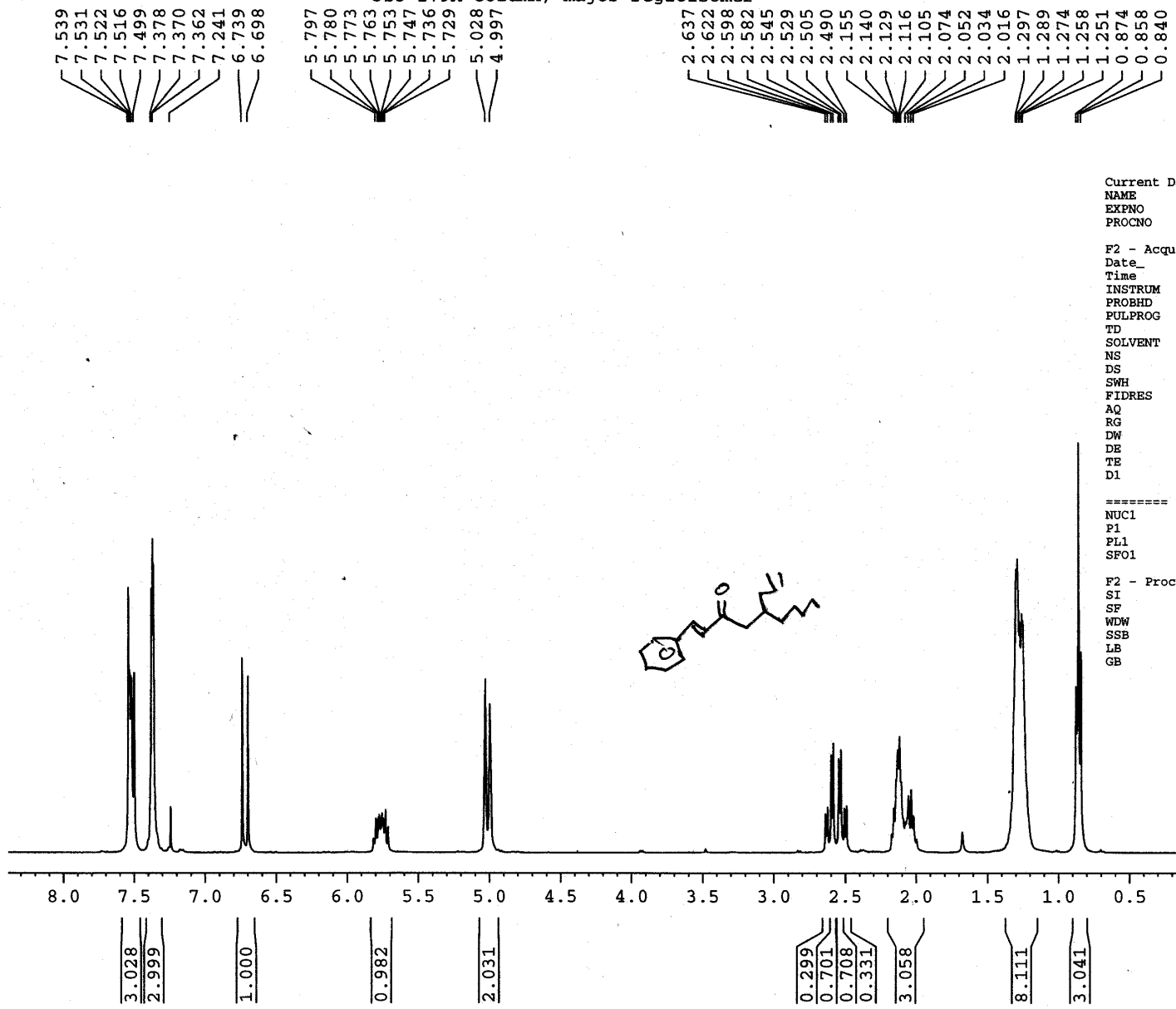
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 0.00 dB
SFO1 100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.0516002 MHz

F2 - Processing parameters
SI 32768
SF 100.5926613 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 218.806 ppm
F1 22010.25 Hz
F2P -3.865 ppm
F2 -388.78 Hz
PPMCM 11.13353 ppm/cm
HZCM 1119.95154 Hz/cm

Js3-279A column, major regioisomer



Current Data Parameters

NAME	Js3-279Acol
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

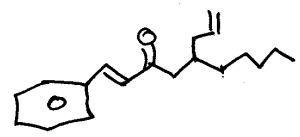
Date_	20060530
Time	15.17
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	143.7
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

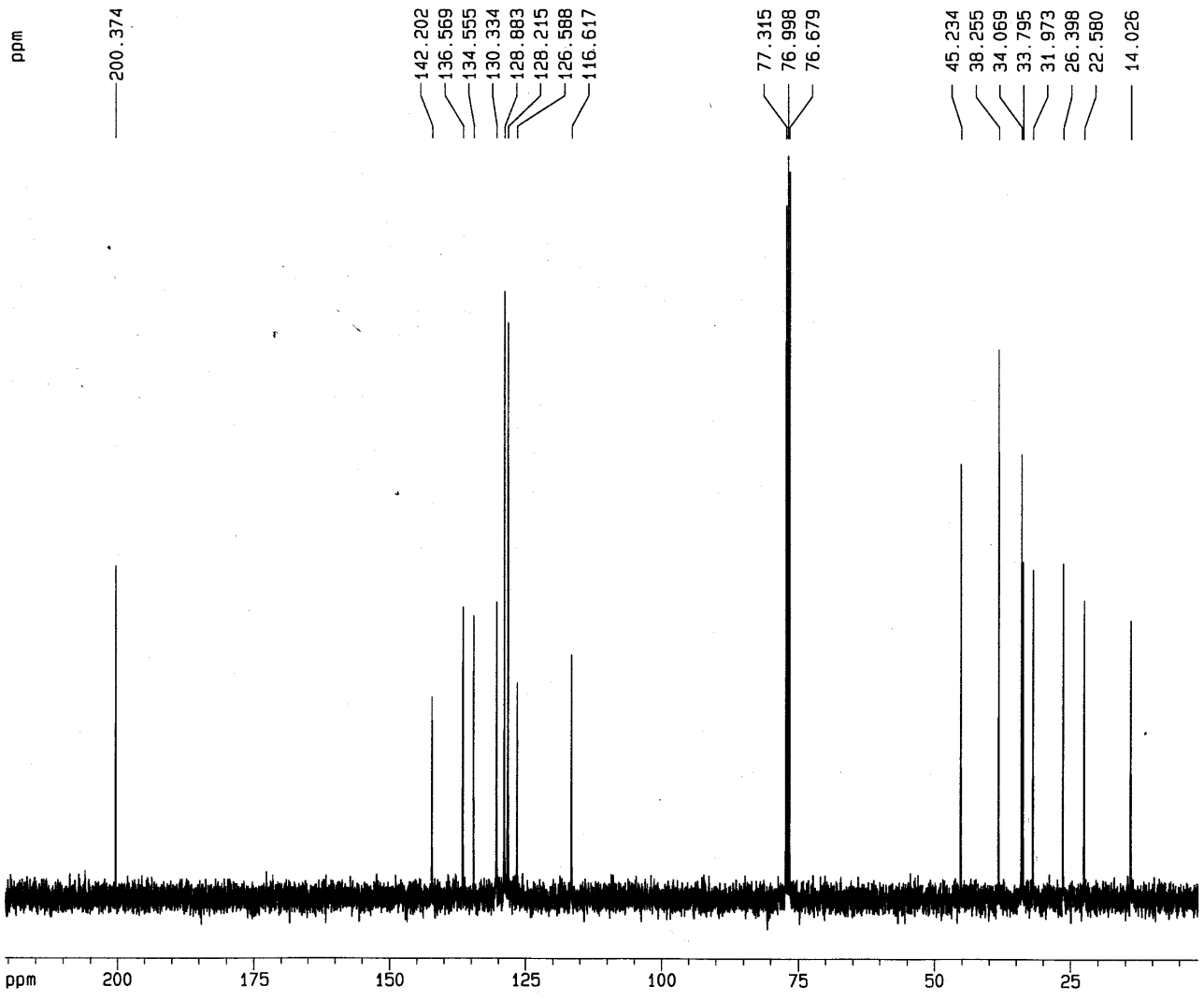
NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0



Js3-279A column, major regioisomer




```
Current Data Parameters
NAME          Js3-279Acol
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20060530
Time               15.22
INSTRUM            spect
PROBHD   5mm QNP 1H/13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  113
DS                    2
SWH           26246.719 Hz
FIDRES         0.400493 Hz
AQ             1.2485298 sec
RG                 2048
DW                19.050 usec
DE                 6.00 usec
TE                300.0 K
D1             0.80000001 sec
d11            0.03000000 sec
d12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 6.00 usec
PL1                0.00 dB
SFO1        100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               13.80 dB
PL13               14.50 dB
SFO2        400.0516002 MHz

F2 - Processing parameters
SI                 32768
SF          100.5926597 MHz
WDW                EM
SSB                 0
LB                 1.00 Hz
GB                  0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P              220.529 ppm
F1             22183.63 Hz
F2P              1.615 ppm
F2               162.49 Hz
PPMCM          10.94570 ppm/cm
HZCM         1101.05676 Hz/cm
```

Js3-279A column, minor regioisomer

7.275 7.255 7.241 7.176 7.157 7.138 6.758 6.740 6.719 6.701 6.684 6.008 5.968 5.665 5.640 5.623 5.598 4.981 4.939 4.915 3.306 3.288 3.271 2.832 2.816 2.813 2.389 2.372 2.354 2.152 2.134 2.117 2.100 1.556 1.407 1.389 1.371 1.353 1.307 1.291 1.274 1.254 1.245 1.219 0.882 0.865

Current Data Parameters

NAME	Js3-279Acol
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

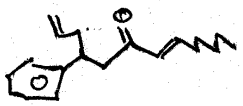
Date_	20060530
Time	15.42
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	21
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	456.1
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

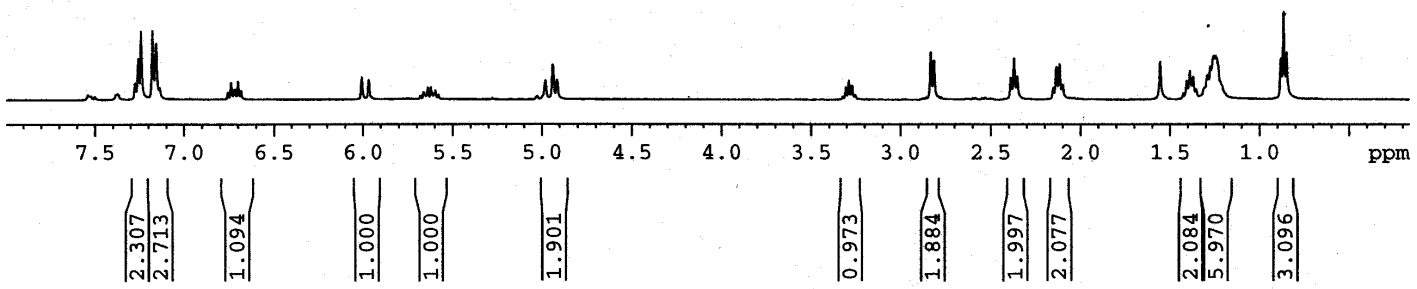
======== CHANNEL f1 ========

NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

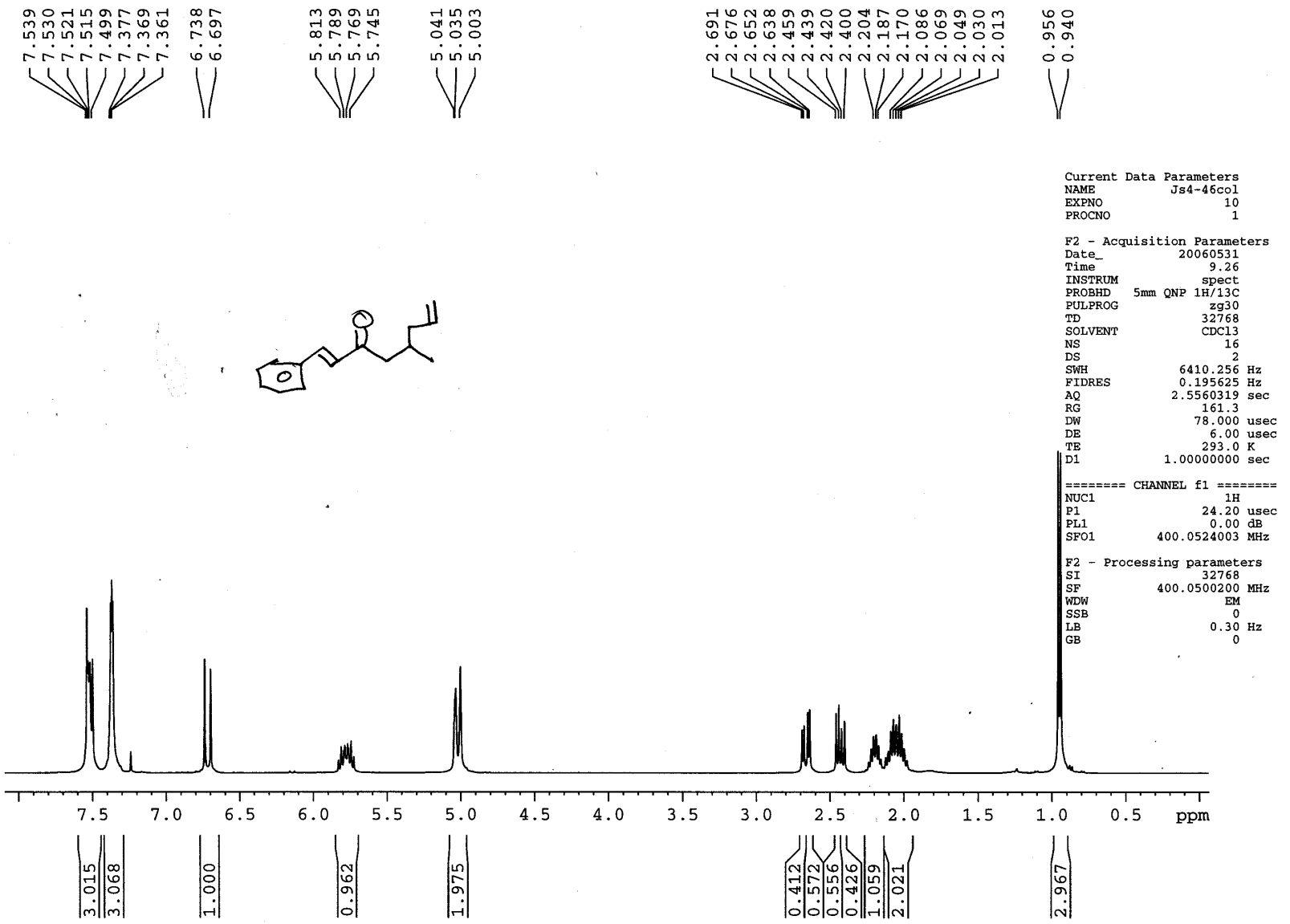
F2 - Processing parameters

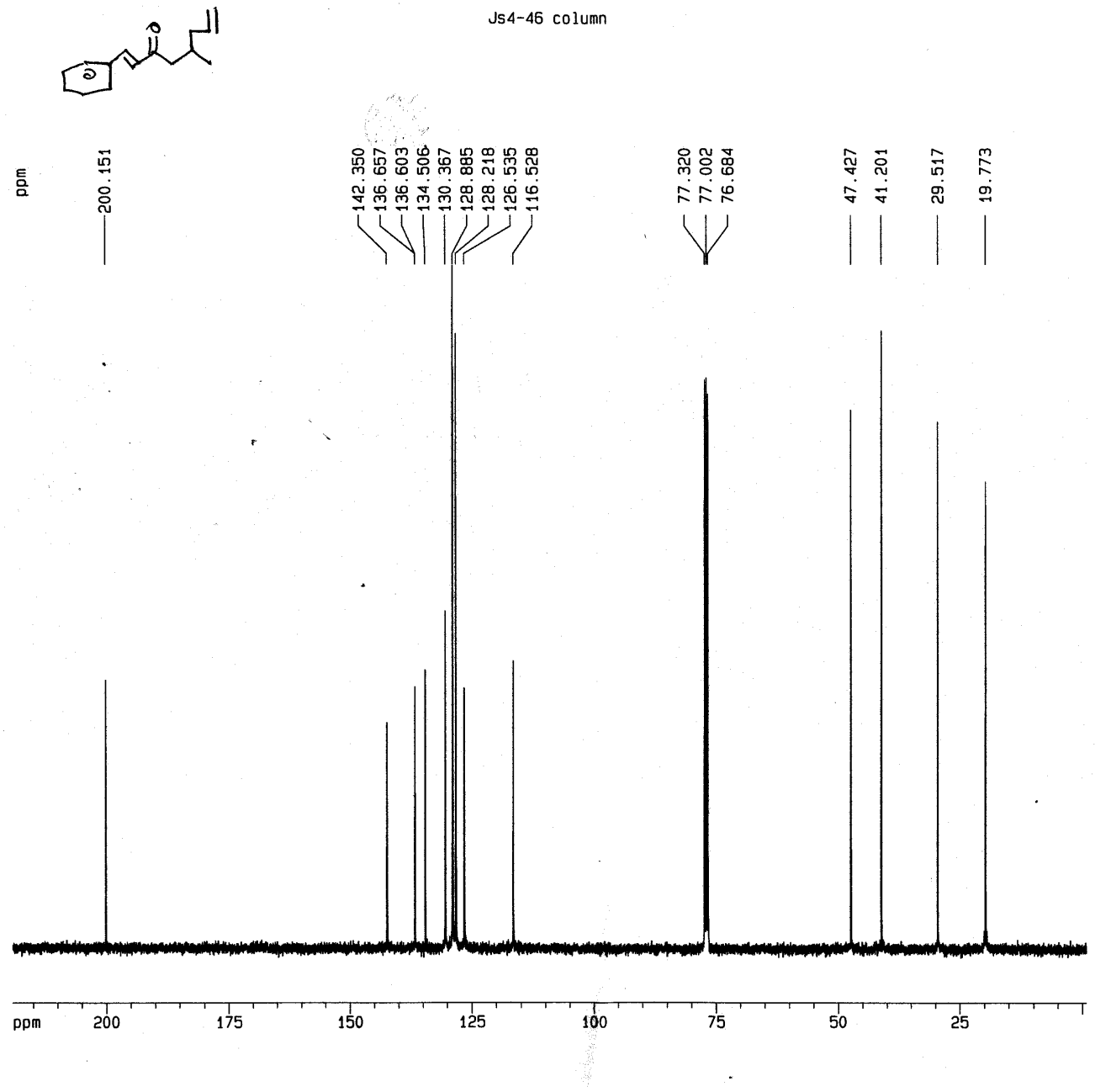
SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0





Js4-46 column





Current Data Parameters

NAME	Js4-46col
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060531
Time	9.58
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	910
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	2298.8
DW	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

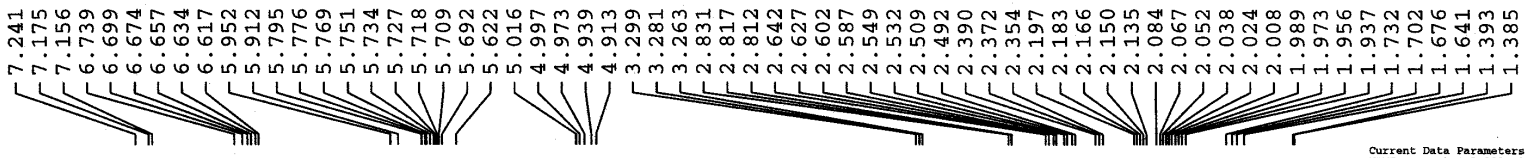
F2 - Processing parameters

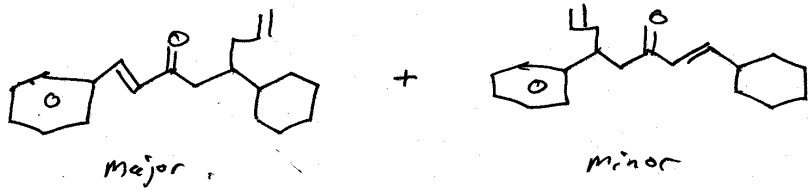
SI	32768
SF	100.5926605 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	219.155 ppm
F1	22045.41 Hz
F2P	-0.783 ppm
F2	-78.79 Hz
PPMCM	10.99692 ppm/cm
HZCM	1106.20984 Hz/cm

Js3-282 column



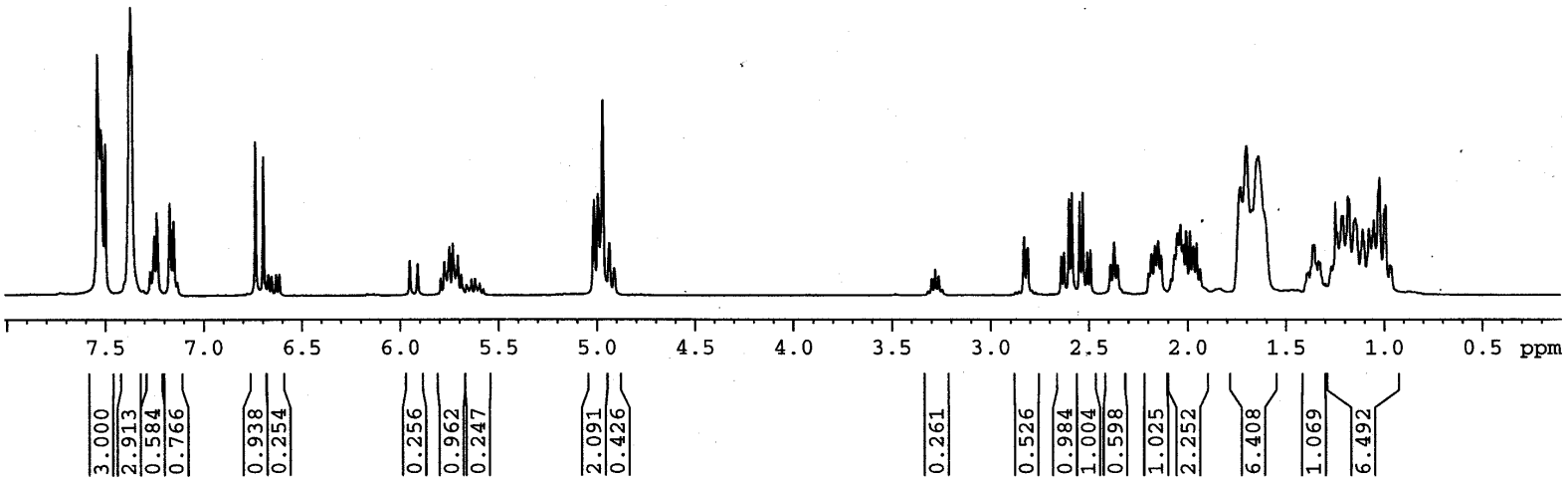


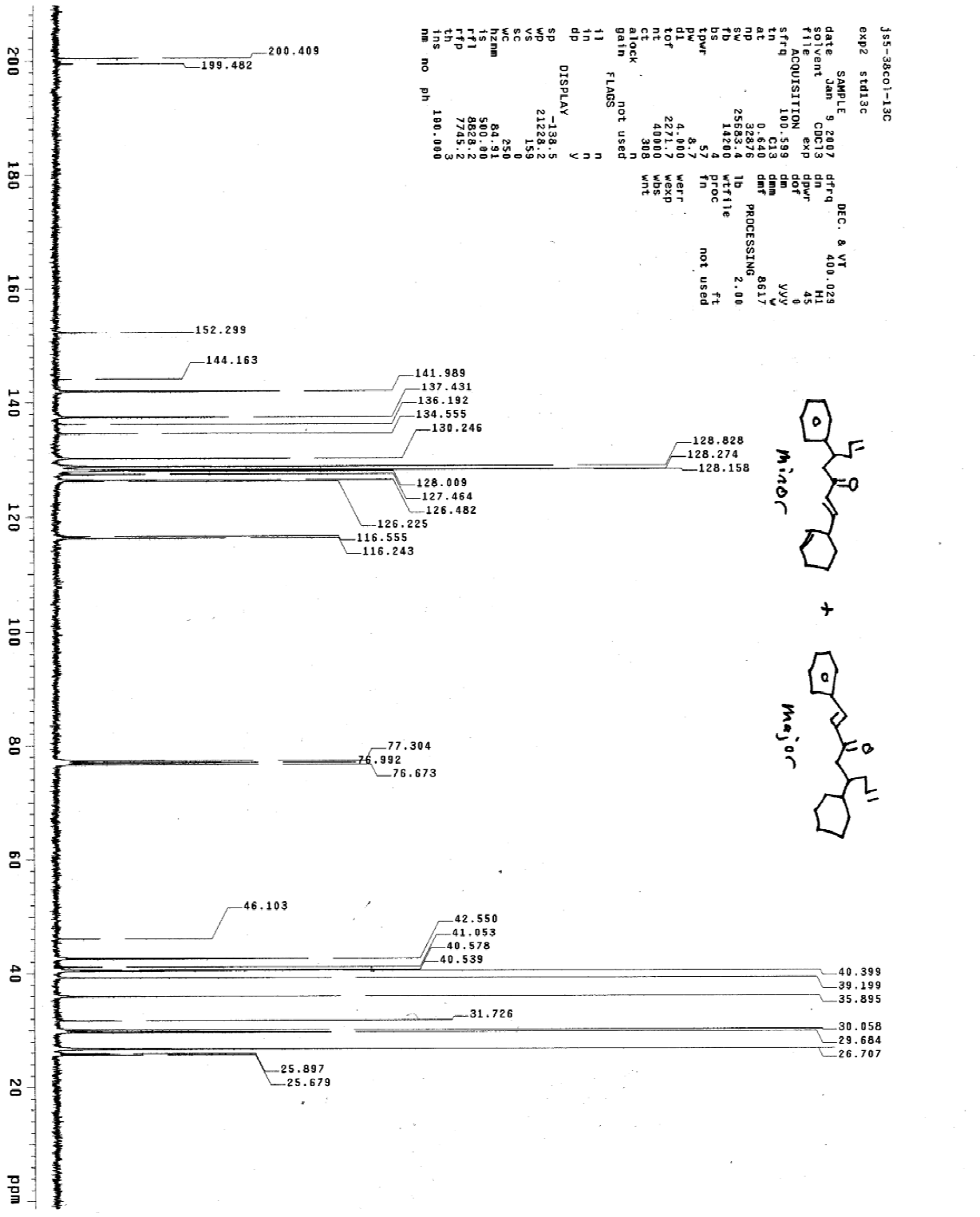
Current Data Parameters
NAME Js3-282col
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060607
Time 11.14
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 181
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec

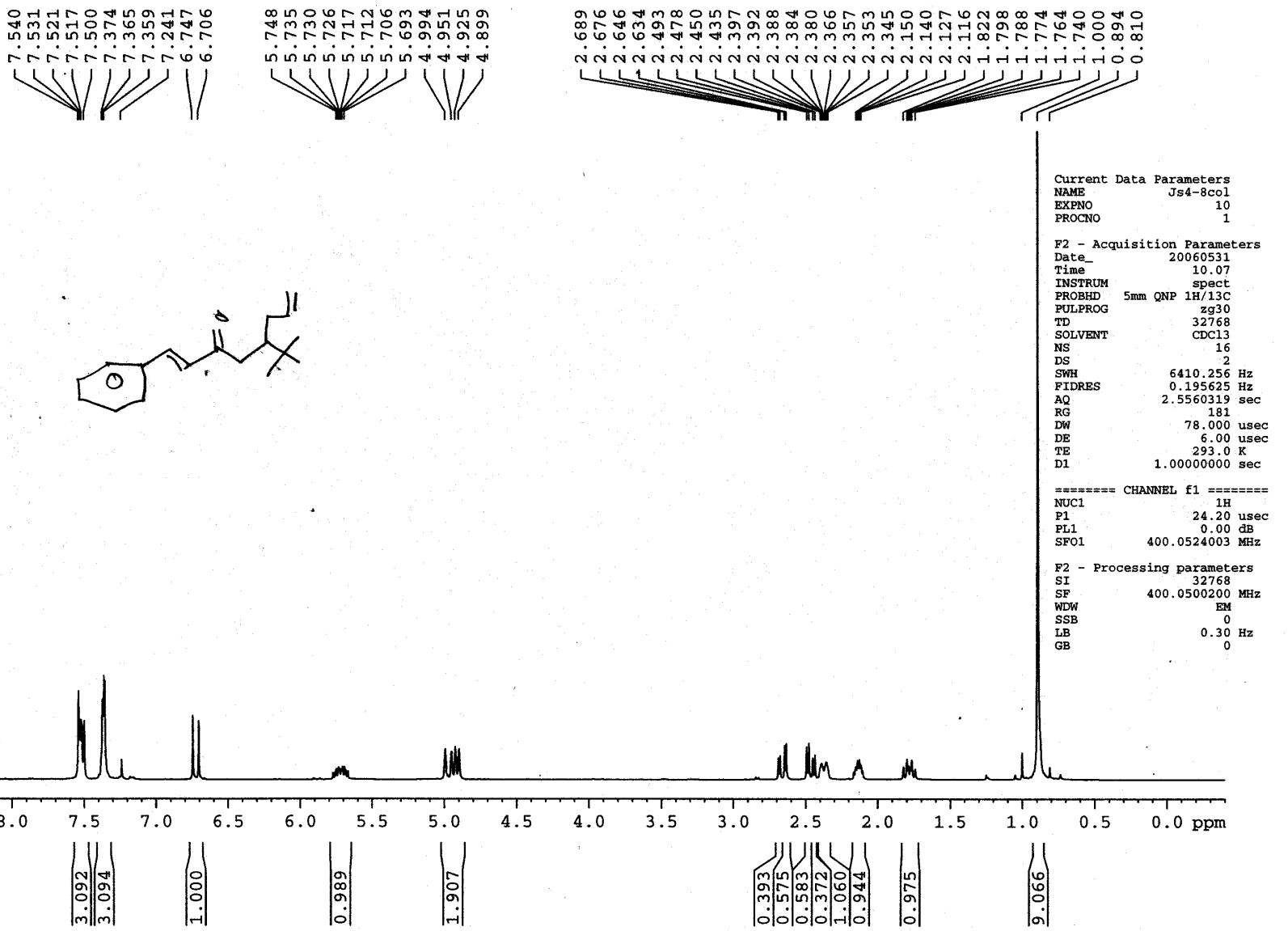
======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz

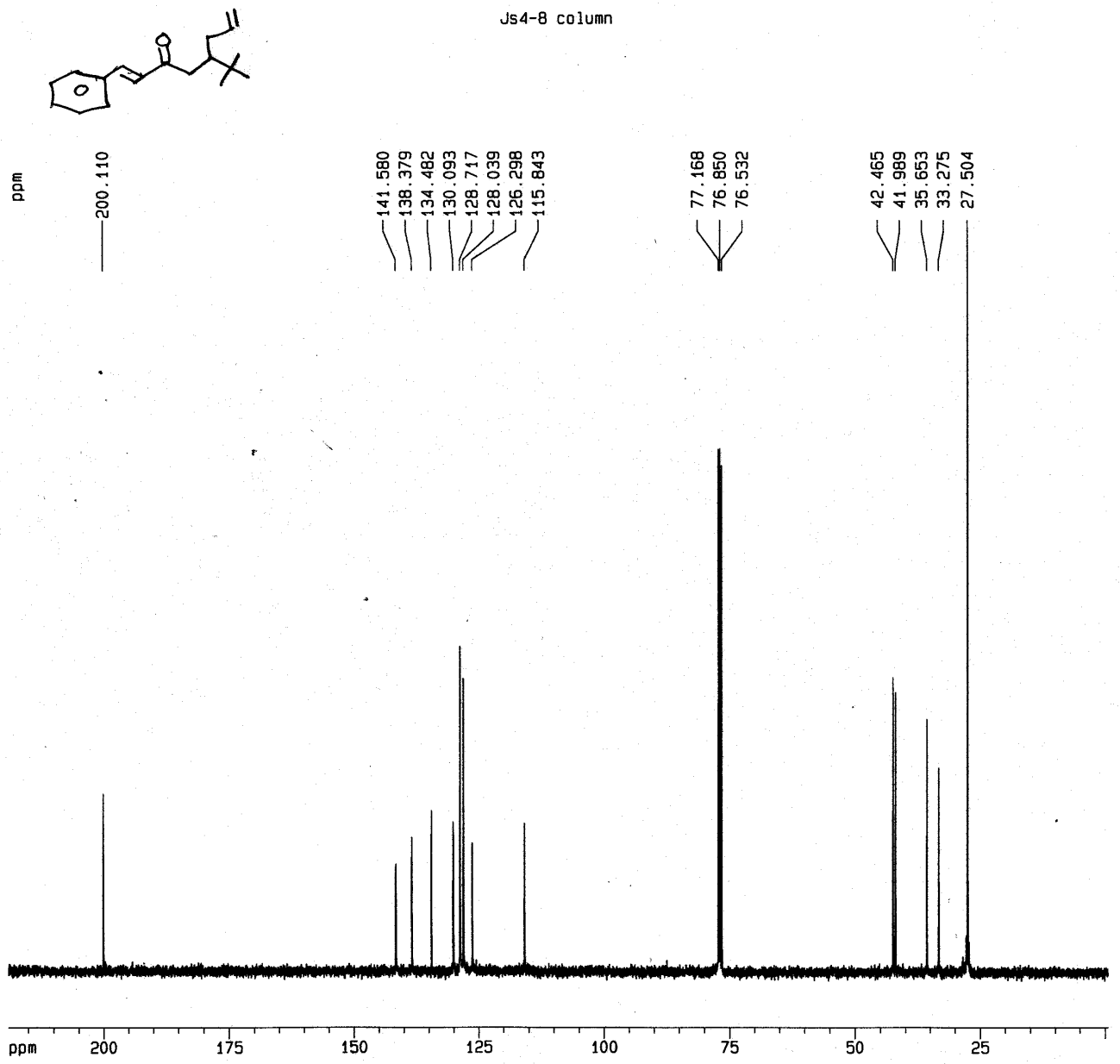
F2 - Processing parameters
SI 32768
SF 400.0500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




200.409
199.482
152.299
144.163
141.989
137.431
136.192
134.555
130.246
128.828
128.274
128.158
128.009
127.464
126.482
126.225
116.555
116.243
77.304
76.992
76.673
46.103
42.550
41.053
40.578
40.539
40.399
39.199
35.895
31.726
30.058
29.684
26.707
25.897
25.679
200
180
160
140
120
100
80
60
40
20
ppm
Minor
+
Major

Js4-8 column


7.540
7.531
7.521
7.517
7.500
7.374
7.365
7.359
7.241
6.747
6.706
5.748
5.735
5.730
5.726
5.717
5.712
5.706
5.693
4.994
4.951
4.925
4.899
2.689
2.676
2.646
2.634
2.493
2.478
2.450
2.435
2.397
2.392
2.388
2.384
2.380
2.366
2.357
2.353
2.345
2.150
2.140
2.127
2.116
1.822
1.798
1.788
1.774
1.764
1.740
1.000
0.894
0.810
Current Data Parameters
NAME Js4-8col
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060531
Time 10.07
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 181
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz
F2 - Processing parameters
SI 32768
SF 400.0500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.092
3.094
1.000
0.989
1.907
0.393
0.575
0.583
0.372
1.060
0.944
0.975
9.066



Current Data Parameters

NAME	Js4-8col
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060531
Time	10.24
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	462
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	3251
DW	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

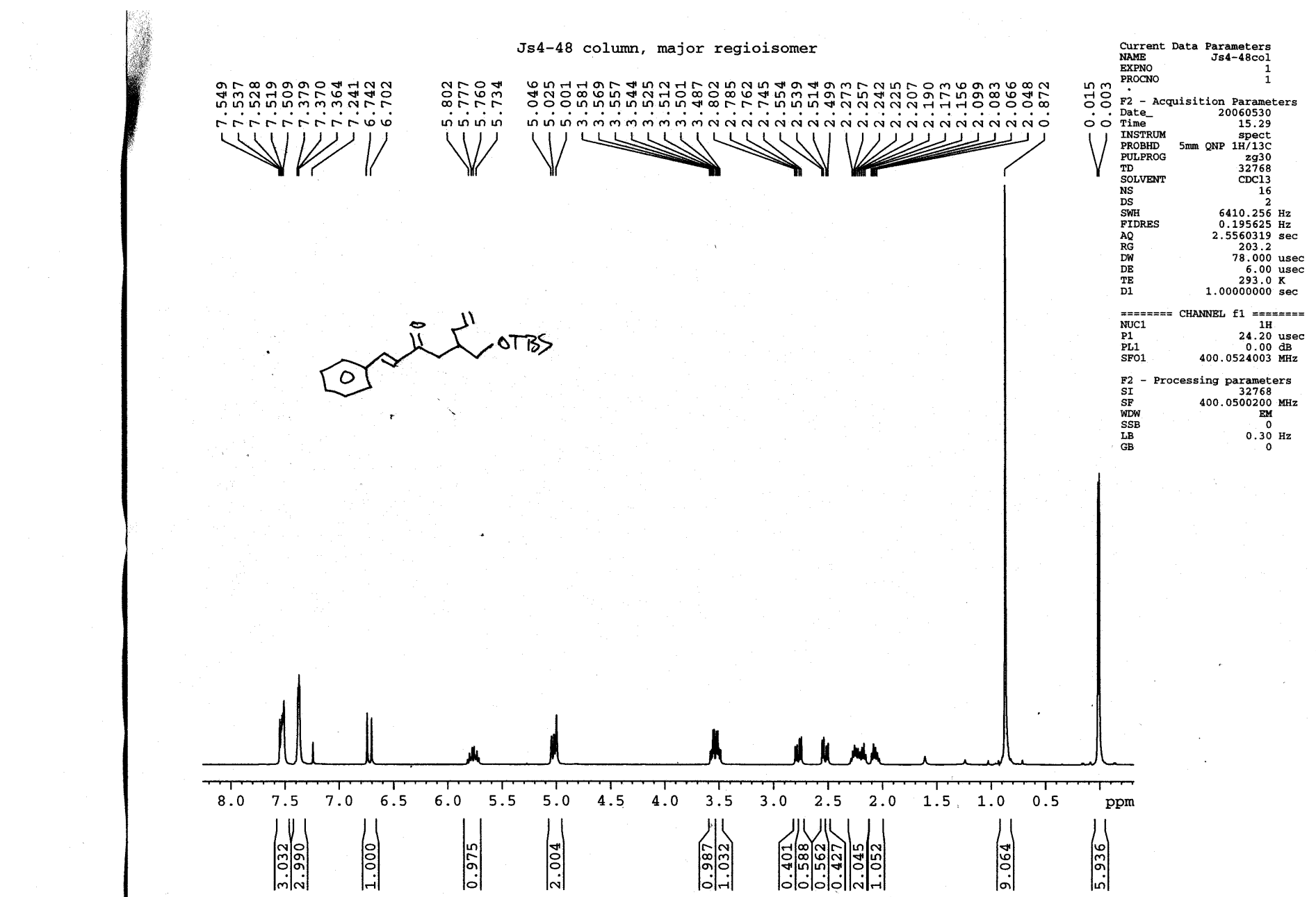
F2 - Processing parameters

SI	32768
SF	100.5926741 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	219.020 ppm
F1	22031.79 Hz
F2P	-0.577 ppm
F2	-58.06 Hz
PPMCM	10.97985 ppm/cm
HZCM	1104.49219 Hz/cm

Js4-48 column, major regioisomer



Current Data Parameters

NAME	Js4-48col
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

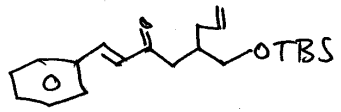
Date_	20060530
Time	15.29
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	203.2
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

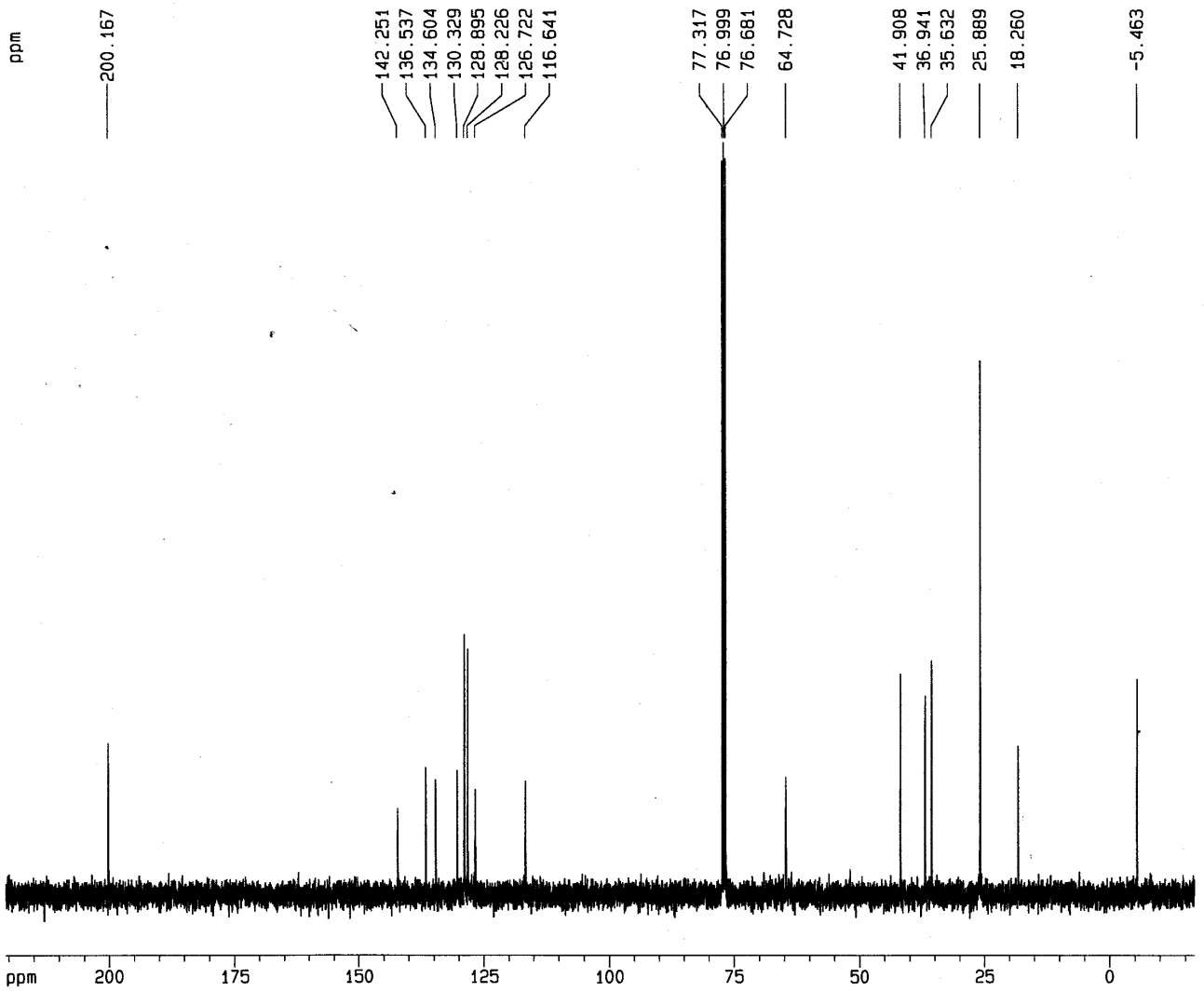
NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0

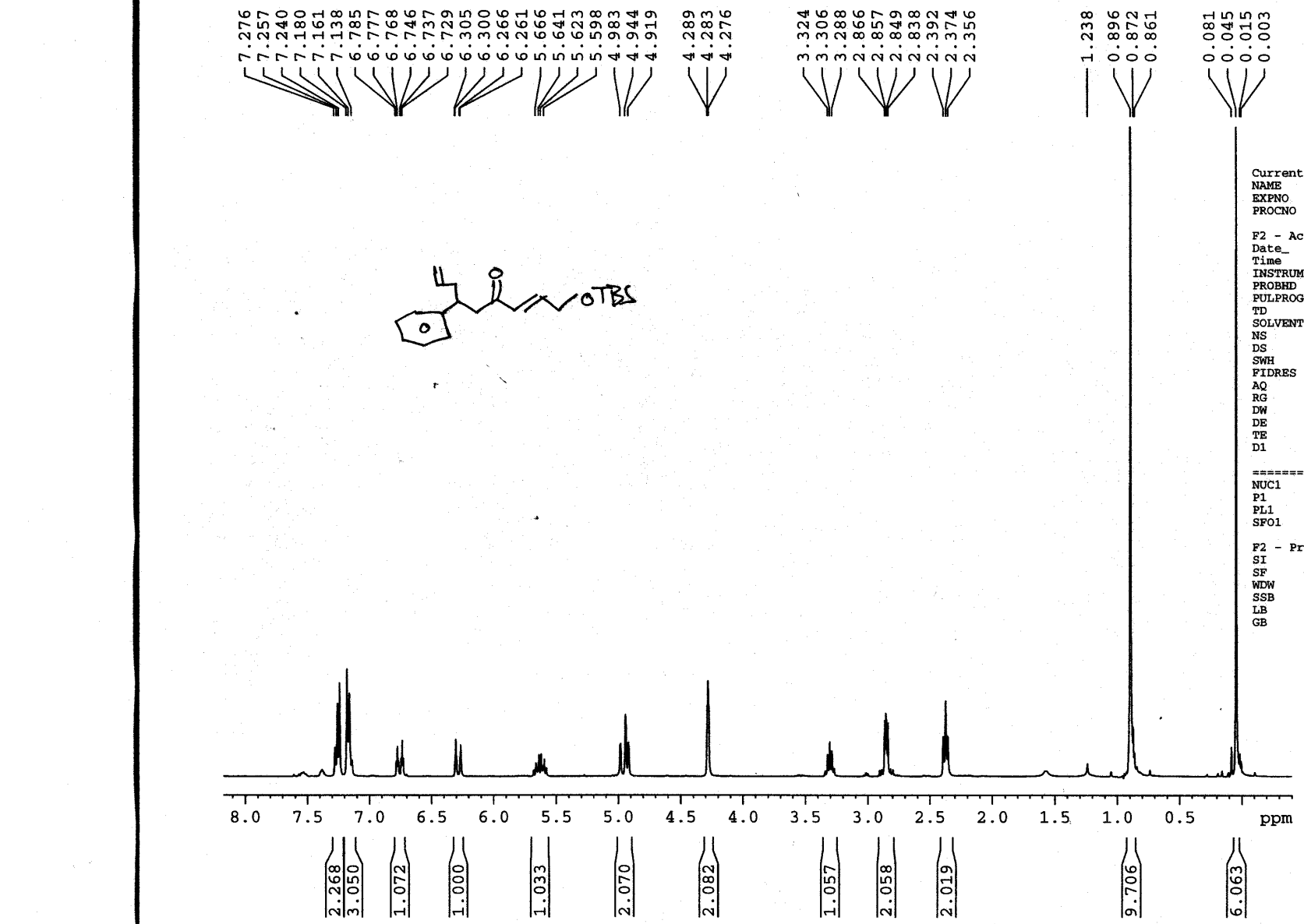


Js4-48 column, major regioisomer




```
Current Data Parameters
NAME                  Js4-48col
EXPNO                        11
PROCNO                        1

F2 - Acquisition Parameters
Date_              20060530
Time                    15.33
INSTRUM                 spect
PROBHD   5mm QNP 1H/13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         121
DS                           2
SWH                26246.719 Hz
FIDRES             0.400493 Hz
AQ                 1.2485298 sec
RG                        3251
DW                      19.050 usec
DE                       6.00 usec
TE                      300.0 K
D1                 0.80000001 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       6.00 usec
PL1                      0.00 dB
SFO1              100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -6.00 dB
PL12                    13.80 dB
PL13                    14.50 dB
SFO2              400.0516002 MHz

F2 - Processing parameters
SI                       32768
SF                100.5926573 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
CY                       12.50 cm
F1P                     220.553 ppm
F1                    22186.03 Hz
F2P                    -16.803 ppm
F2                    -1690.24 Hz
PPMCM                 11.86780 ppm/cm
HZCM                1193.81348 Hz/cm
```

Js4-48 column, minor regioisomer

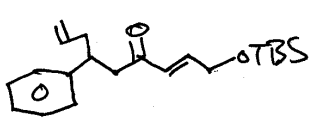


Current Data Parameters
NAME Js4-48col
EXPNO 2
PROCNO 1

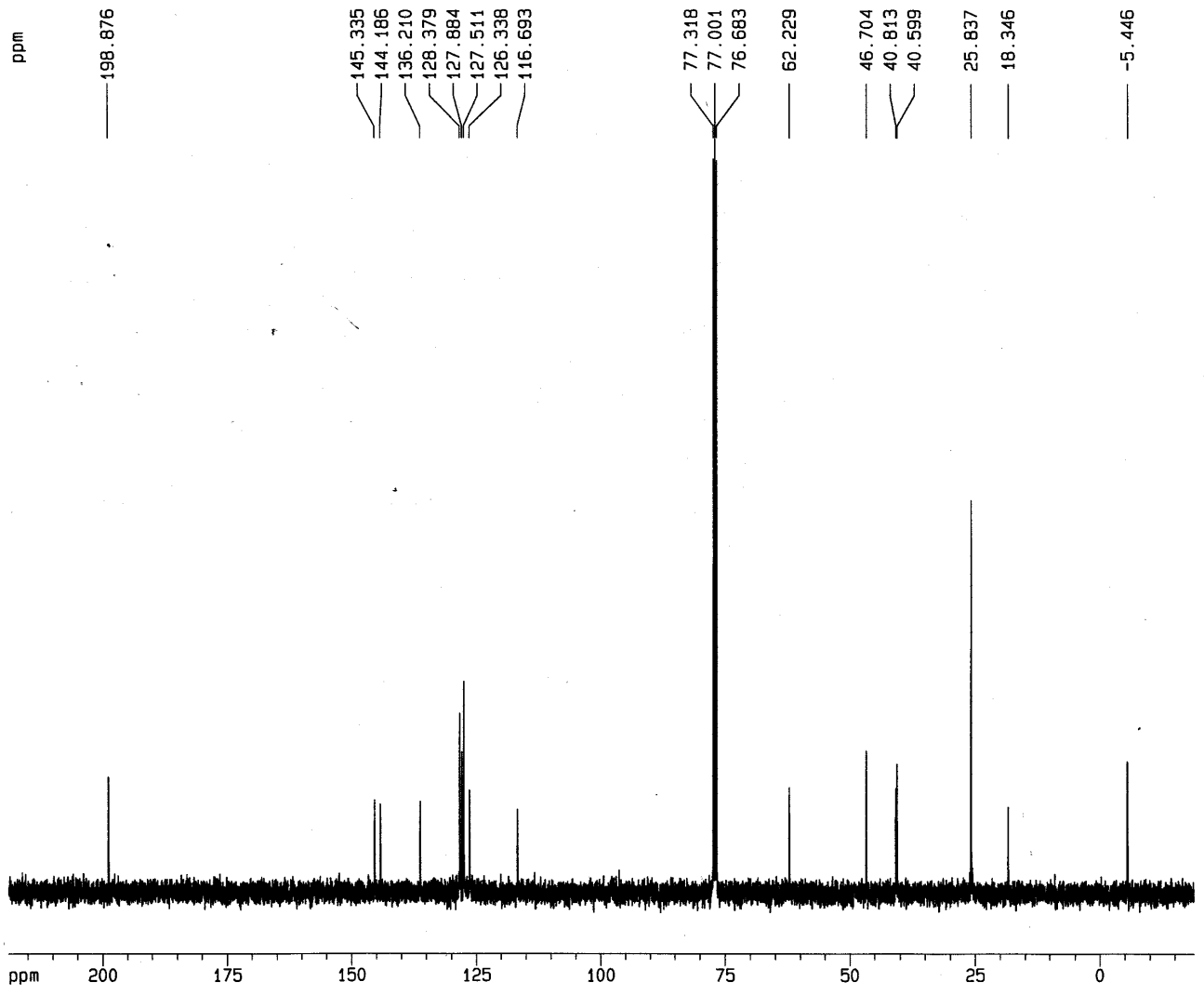
F2 - Acquisition Parameters
Date_ 20060530
Time 15.47
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 322.5
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz

F2 - Processing parameters
SI 32768
SF 400.0500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

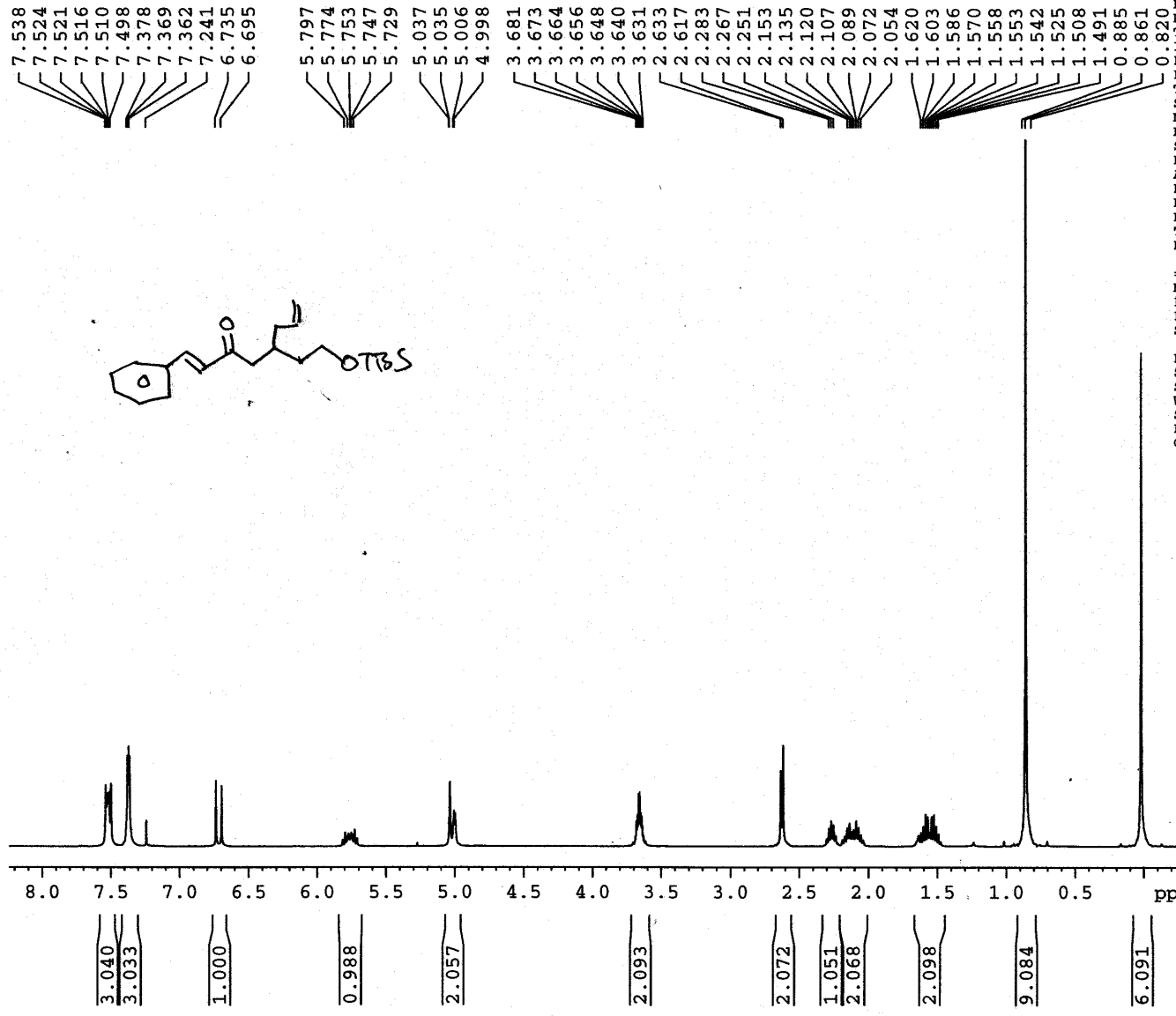


Js4-48 column, minor regioisomer



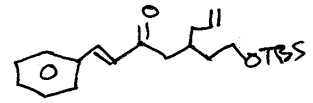

```
Current Data Parameters
NAME                  Js4-48col
EXPNO                        21
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20060530
Time                      15.55
INSTRUM                   spect
PROBHD     5mm QNP 1H/13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         245
DS                           2
SWH                26246.719 Hz
FIDRES             0.400493 Hz
AQ                 1.2485298 sec
RG                    3649.1
DW                   19.050 usec
DE                    6.00 usec
TE                    300.0 K
D1                 0.80000001 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    6.00 usec
PL1                   0.00 dB
SFO1              100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                80.00 usec
PL2                  -6.00 dB
PL12                 13.80 dB
PL13                 14.50 dB
SFO2              400.0516002 MHz

F2 - Processing parameters
SI                       32768
SF                100.5926565 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                218.854 ppm
F1                 22015.07 Hz
F2P                -18.844 ppm
F2                 -1895.56 Hz
PPMCM              11.88488 ppm/cm
HZCM            1195.53125 Hz/cm
```

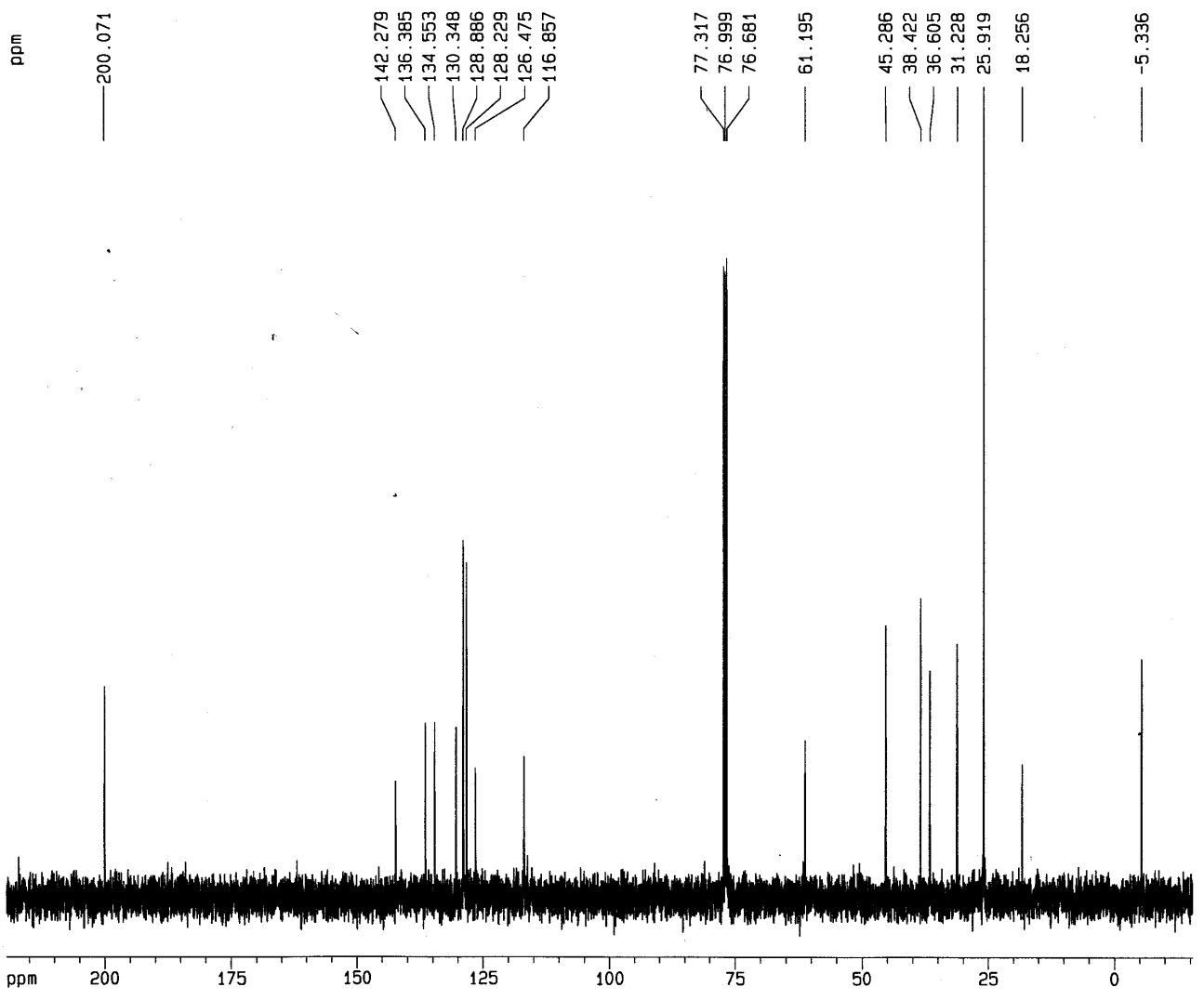
Js4-16 column, major regioisomer




```
Current Data Parameters
NAME                  Js4-16col
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20060602
Time                     15.51
INSTRUM                  spect
PROBHD    5mm QNP 1H/13C
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6410.256 Hz
FIDRES              0.195625 Hz
AQ                  2.5560319 sec
RG                         181
DW                    78.000 usec
DE                     6.00 usec
TE                     293.0 K
D1                 1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    24.20 usec
PL1                    0.00 dB
SFO1           400.0524003 MHz

F2 - Processing parameters
SI                       32768
SF             400.0500200 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
```

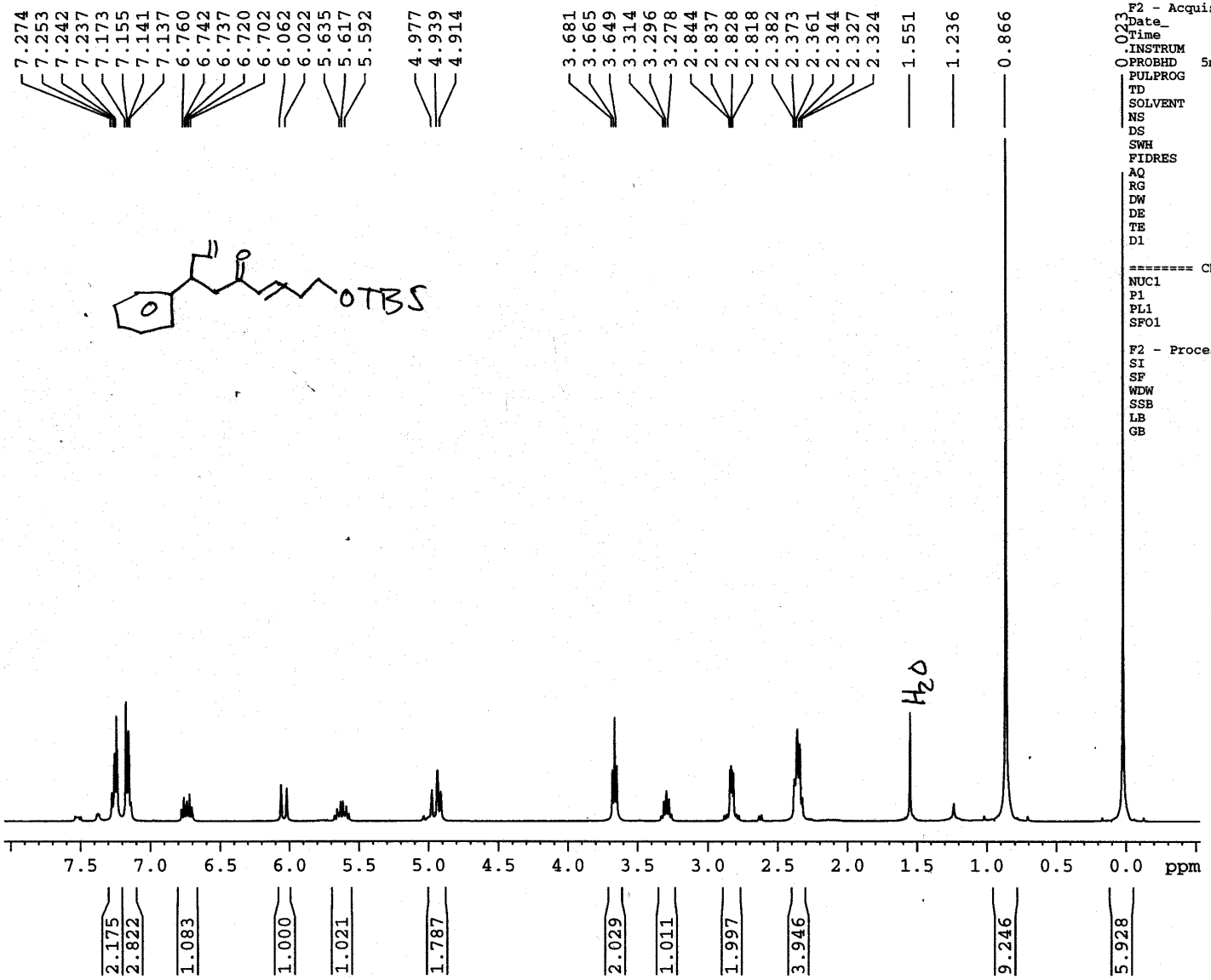
Js4-16 column, major regioisomer






```
Current Data Parameters
NAME                  Js4-16col
EXPNO                        11
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20060602
Time                     15.54
INSTRUM                  spect
PROBHD     5mm QNP 1H/13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          66
DS                           2
SWH                26246.719 Hz
FIDRES              0.400493 Hz
AQ                  1.2485298 sec
RG                       13004
DW                    19.050 usec
DE                     6.00 usec
TE                     300.0 K
D1                  0.80000001 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                     6.00 usec
PL1                    0.00 dB
SFO1              100.6036782 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                   -6.00 dB
PL12                  13.80 dB
PL13                  14.50 dB
SFO2              400.0516002 MHz

F2 - Processing parameters
SI                         32768
SF                100.5926581 MHz
WDW                           EM
SSB                            0
LB                     1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     12.50 cm
F1P                  219.521 ppm
F1                   22082.17 Hz
F2P                   -15.445 ppm
F2                   -1553.62 Hz
PPMCM                11.74827 ppm/cm
HZCM              1181.78943 Hz/cm
```

Js4-16 column, minor regioisomer



Current Data Parameters

NAME	Js4-16col
EXPNO	20
PROCNO	1

F2 - Acquisition Parameters

Date_	20060602
Time	16.36
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	574.7
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

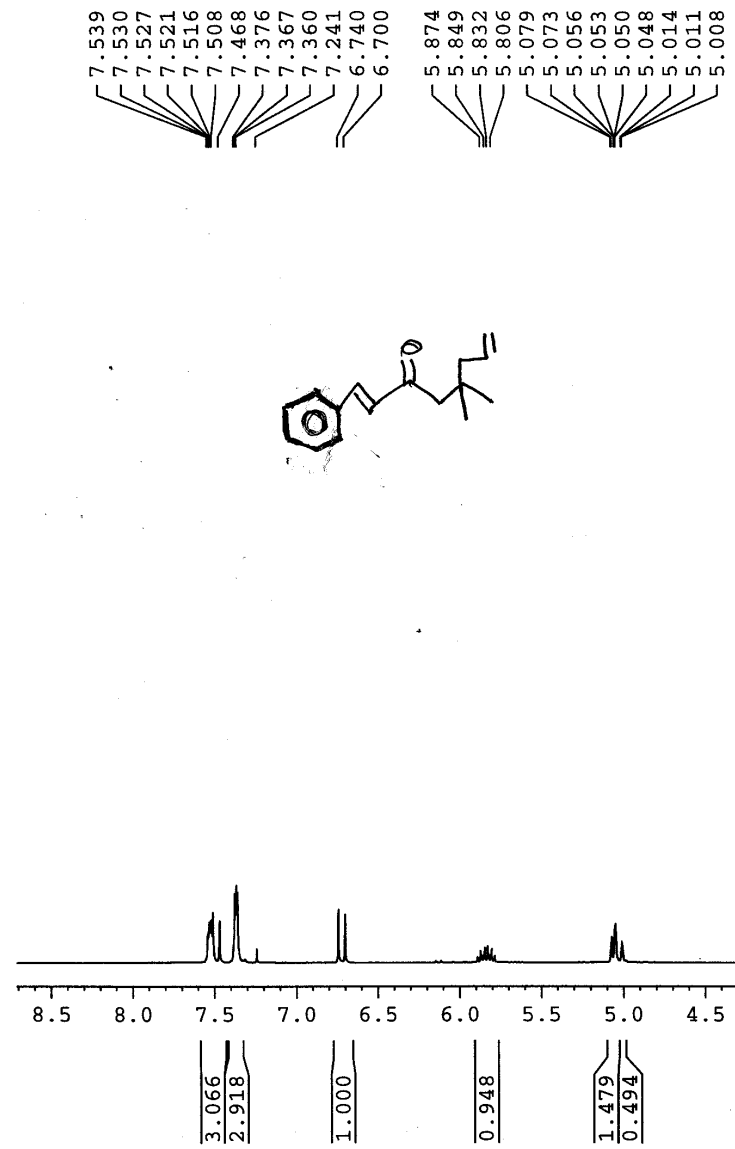
======== CHANNEL f1 ========

NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SFO1	400.0524003 MHz

F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0

Js4-34 column



Current Data Parameters

NAME	Js4-34col
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

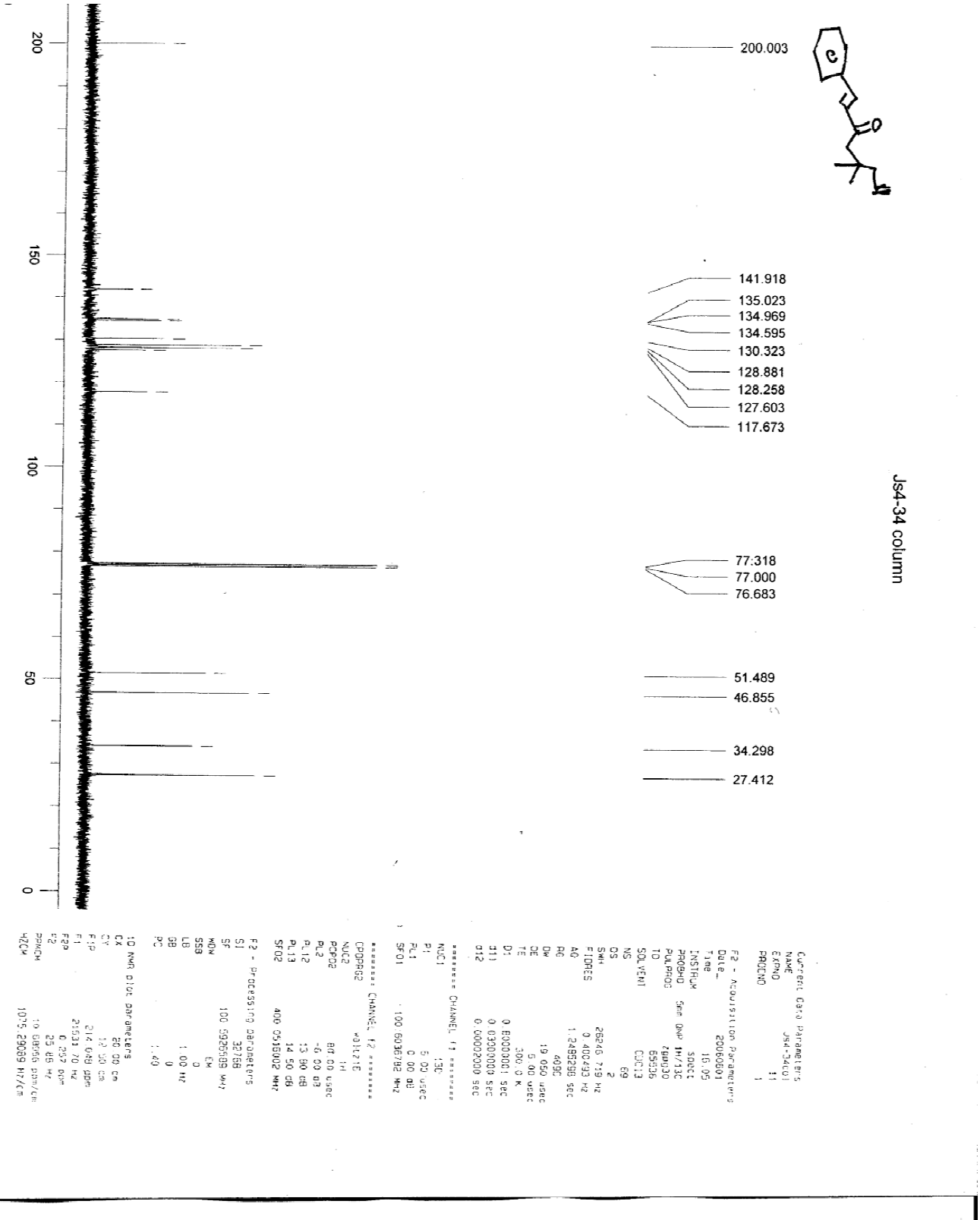
Date_	20060601
Time	16.02
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5560319 sec
RG	203.2
DW	78.000 usec
DE	6.00 usec
TE	293.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

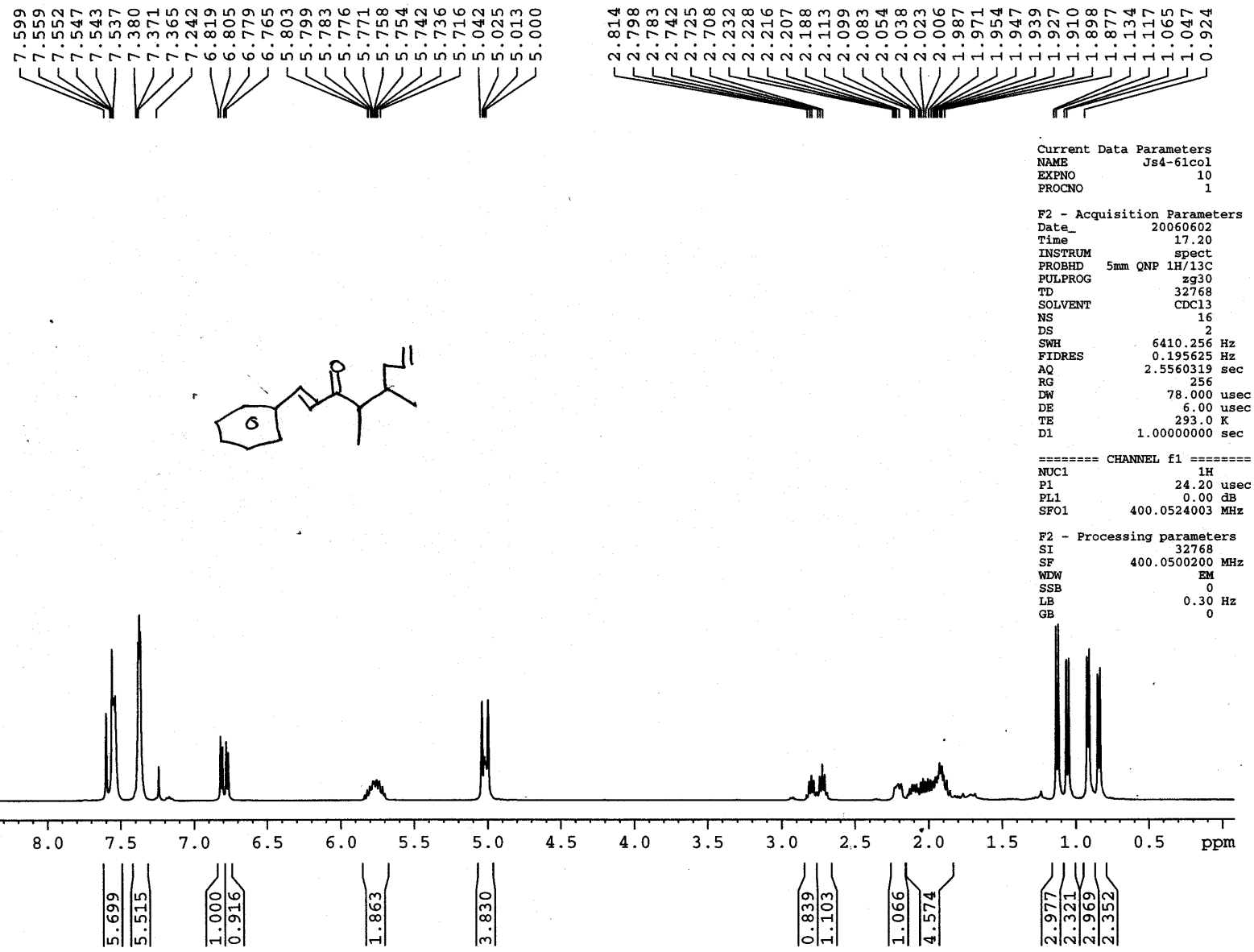
NUC1	1H
P1	24.20 usec
PL1	0.00 dB
SF01	400.0524003 MHz

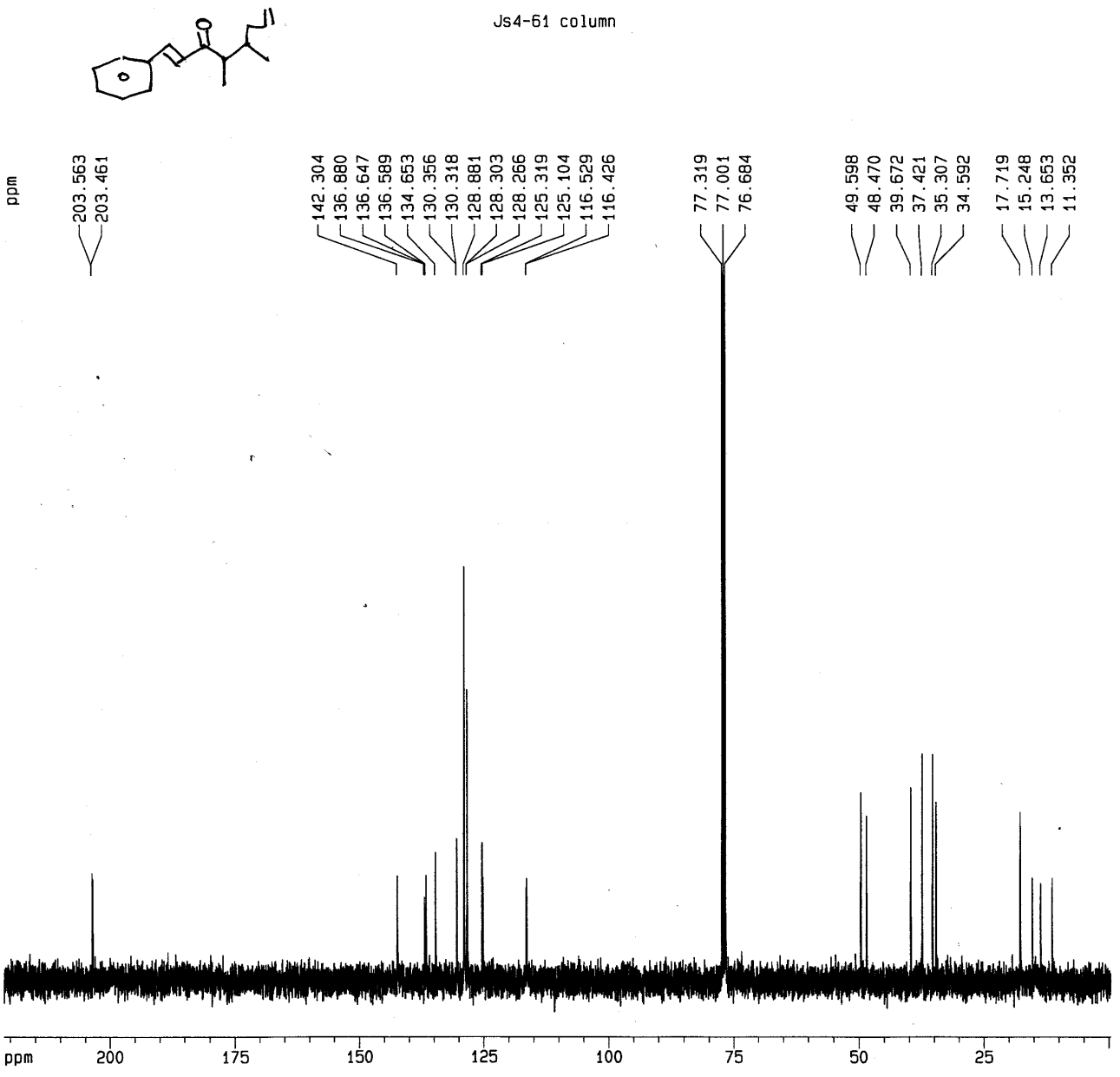
F2 - Processing parameters

SI	32768
SF	400.0500200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0


Js4-34 column
200.003
141.918
135.023
134.969
134.595
130.323
128.881
128.258
127.603
117.673
77.318
77.000
76.683
51.489
46.855
34.298
27.412
200
150
100
50
0

Js4-61 column





Current Data Parameters

NAME	Js4-61col
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060602
Time	17.23
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	3649.1
DW	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

F2 - Processing parameters

SI	32768
SF	100.5926581 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

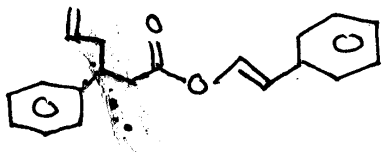
1D NMR plot parameters

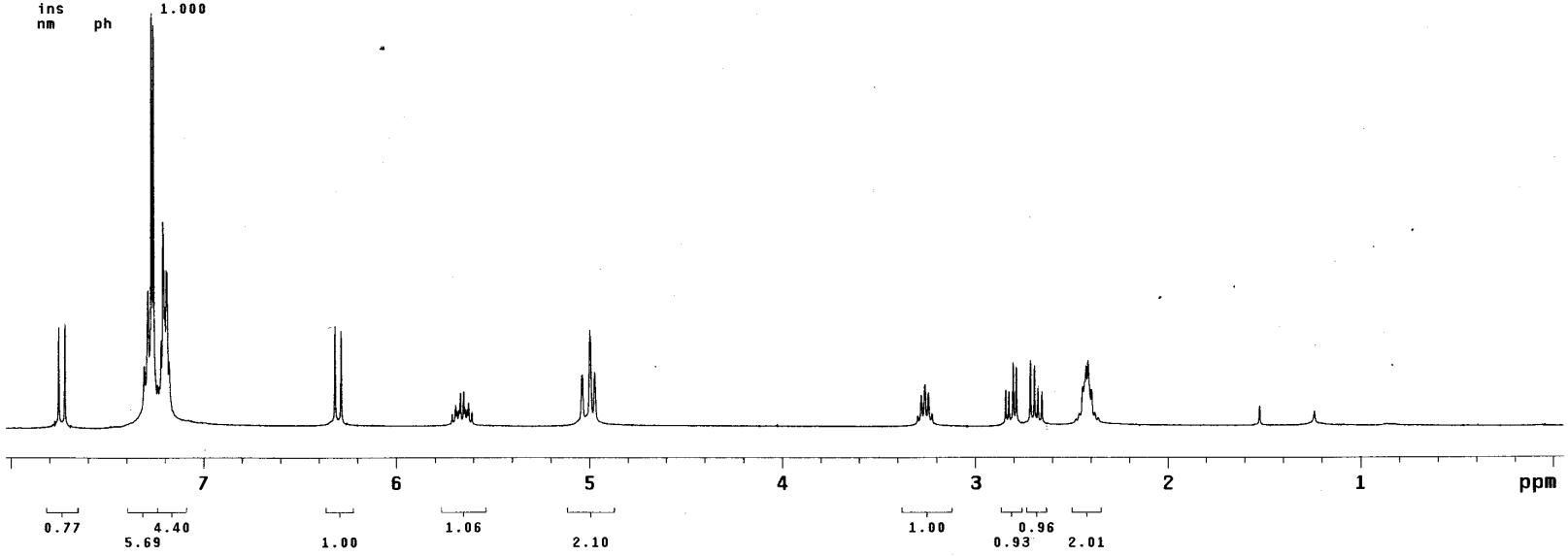
CX	20.00 cm
CY	18.00 cm
F1P	221.228 ppm
F1	22253.94 Hz
F2P	-0.418 ppm
F2	-42.03 Hz
PPMCM	11.08230 ppm/cm
HZCM	1114.79834 Hz/cm

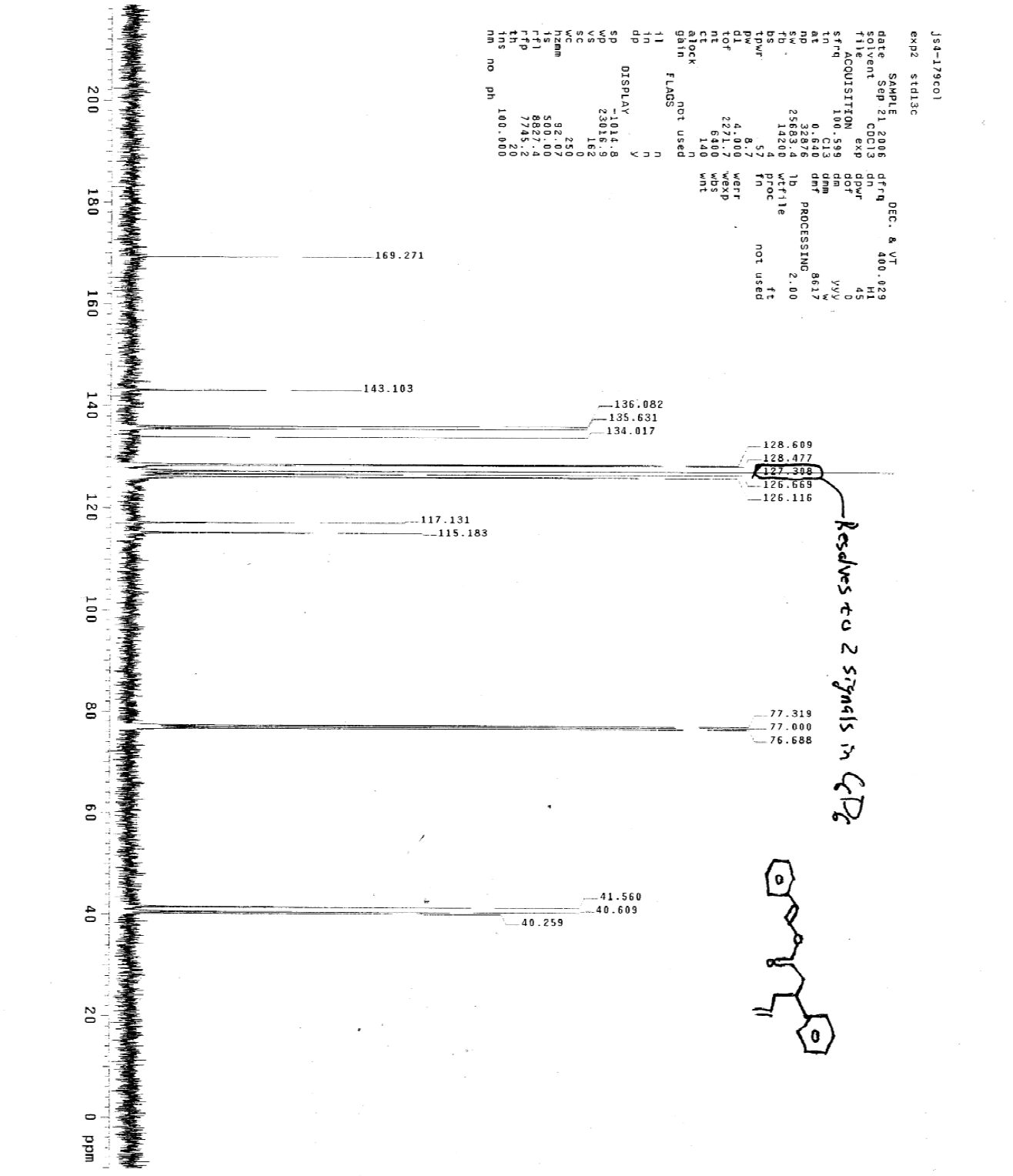
js4-179col

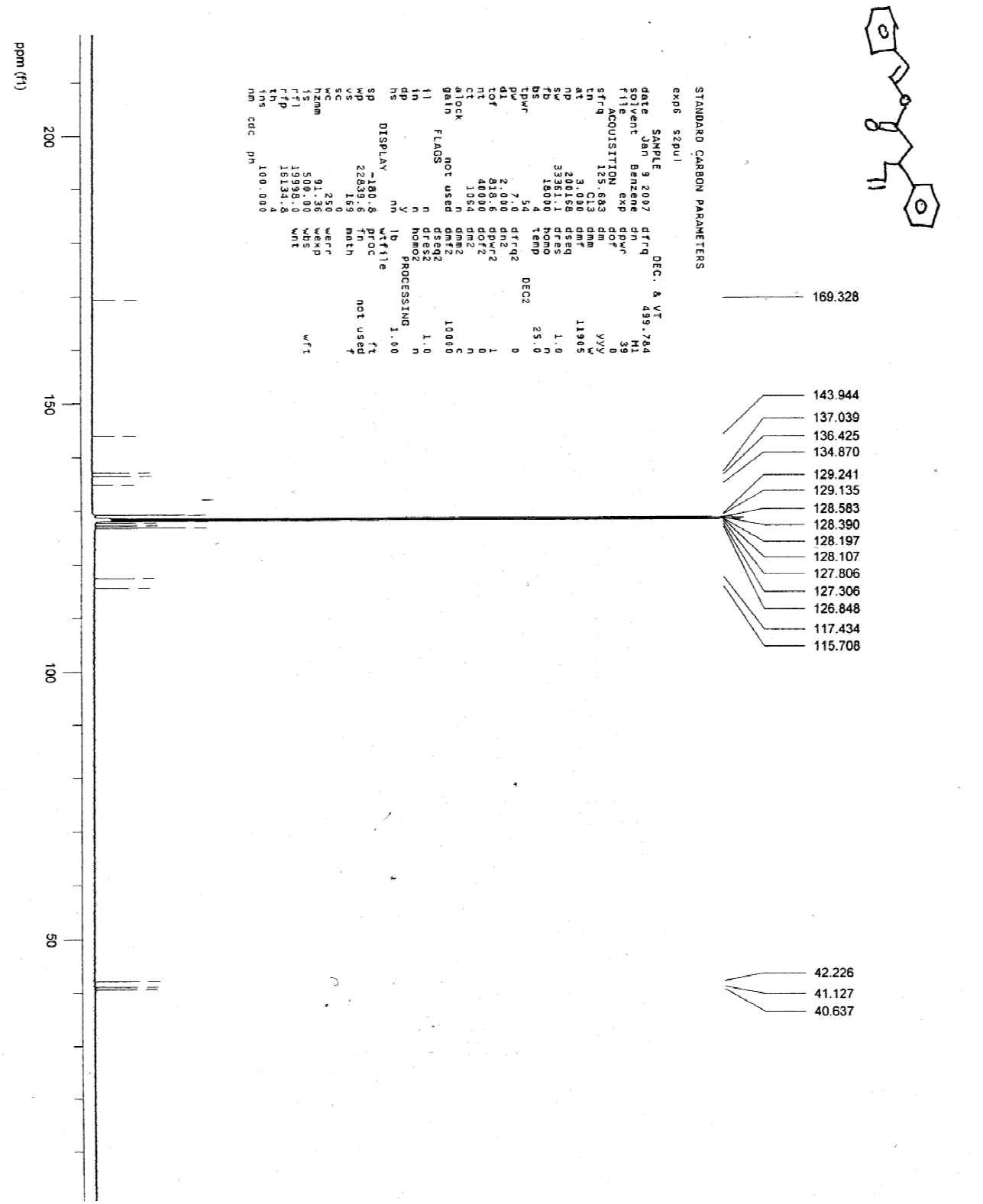
exp1 stdih

SAMPLE		DEC. & VT	
date	Sep 21 2006	dfrq	0
solvent	CDCl3	dn	
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.029	dm	nnn
tn	H1	dmm	c
at	3.000	dmf	200
np	35992	PROCESSING	
sw	5998.8	wtfile	
fb	3400	proc	ft
bs	4	fn	not used
tpwr	63		
pw	7.1	werr	
d1	4.000	wexp	
tof	0	wbs	
nt	16	wnt	
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-21.3		
wp	3231.8		
vs	66		
sc	0		
wc	250		
hzmm	12.93		
is	3747.97		
rfl	4102.7		
rfp	3088.2		
th	7		
ins	1.000		
nm	ph		






169.271
143.103
136.082
135.631
134.017
128.609
128.477
127.308
126.669
126.116
117.131
115.183
77.319
77.000
76.688
41.560
40.609
40.259
200
180
160
140
120
100
80
60
40
20
0 ppm
Resolves to 2 signals in CDCl3


STANDARD CARBON PARAMETERS
exp5 s2pul
SAMPLE
date Jan 9 2007
solvent Benzene
file
ACQUISITION
sfrq 125.683
tn C13
at 3.000
np 200168
sw 33351.1
fb 18000
bs 4
tpwr 54
pw 7.0
d1 2.000
tof 818.6
nt 40000
ct 1064
alock n
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -180.8
wp 22833.6
vs 169
sc 0
wc 250
hzmm 91.36
is 500.00
rfl 19998.0
rfp 16134.8
th 4
ins 100.000
nm cdc pn
DEC. & VT
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
temp
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
PROCESSING
lb
wtfile
proc
fn
math
not used
wft
wfr
exp
wbs
wnt
not used
ft
not used
f
499.784
H1
39
0
yyy
w
11905
1.0
n
n
25.0
0
1
0
n
c
10000
1.0
n
PROCESSING
1.00
ppm (f1)
200
150
100
50
169.328
143.944
137.039
136.425
134.870
129.241
129.135
128.583
128.390
128.197
128.107
127.806
127.306
126.848
117.434
115.708
42.226
41.127
40.637