

STUDIES IN THE SYNTHESIS OF THE INOSTAMYCIN NATURAL PRODUCTS: A REDUCTIVE ALDOL/REDUCTIVE CLAISEN APPROACH TO THE C₁₀-C₂₄ KETONE FRAGMENT

Nathan O. Fuller and James P. Morken*

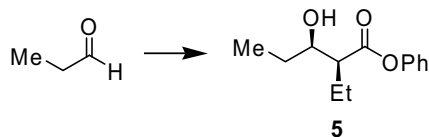
*Department of Chemistry, Venable and Kenan Laboratories
University of North Carolina, Chapel Hill, NC 27599-3290*

Supplementary Material

General. Infrared spectra were recorded on a Nicolet Magna 560 spectrometer, ν_{\max} in cm^{-1} . Bands are characterized as broad (br), strong (s), medium (m), and weak (w). ^1H NMR spectra were recorded on Varian Gemini (300 MHz) and Bruker (400 MHz) spectrometers. 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, br = broad, m = multiplet), coupling constants (Hz), and assignment. ^{13}C NMR were recorded on a Bruker 400 (75 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard (CDCl_3 : 77.0 ppm).

Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on Sigma silica gel 60 (SiO_2 , 230-400 mesh). Thin layer chromatography was performed on EM Science 0.25 mm silica gel 60 plates. Visualization was achieved with phosphomolybdic acid in ethanol followed by heating, potassium permanganate in ethanol/water, followed by heating, or by UV light (254 nm). Analytical gas-liquid chromatography (GLC) was performed on a Hewlett-Packard 6890 Series chromatograph equipped with a split mode capillary injection system, the indicated chiral GLC column, a flame ionization detector and using helium as the carrier gas. Analytical supercritical fluid chromatography (SFC) was performed on a Berger SFC using methanol as the modifier with the indicated chiral column.

All reactions were conducted in oven or flame-dried glassware under an inert atmosphere of dry nitrogen or argon. 1,2-Dichloroethane was sequentially washed with concentrated H_2SO_4 , water, aqueous NaHCO_3 , and water, then dried with MgSO_4 and fractionally distilled from CaH_2 . Chloro(1,5-cyclooctadiene)rhodium (I) dimer and (*R*)-BINAP were purchased from Strem Chemical Company. All other reagents were purchased from either Lancaster or Aldrich Chemical Companies. $[(\text{cod})\text{RhBINAP}]\text{BF}_4$ was synthesized according to the procedure by: Miyshita, A.; Takaya, H.; Souchi, T.; Noyori, R. *Tetrahedron* **1984**, 40, 1245-1253.

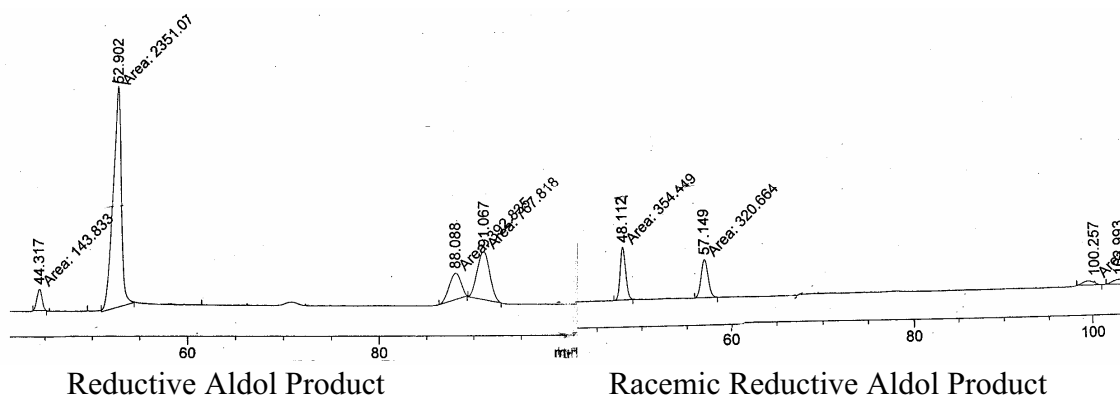


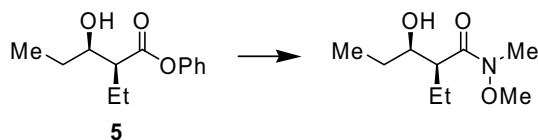
(2*S*,3*R*)-2-Ethyl-3-hydroxy-pentanoic acid phenyl ester (5). An oven-dried 250 mL flask equipped with a stir bar was charged with 0.3696 g (0.76 mmol) chloro(1,5-cyclooctadiene)rhodium (I) dimer, 1.213 g (1.98 mmol) (*S*)-BINAP and 30 mL of dry dichloroethane under an argon atmosphere. After stirring for one hour, diethylmethylsilane (21.7 mL, 5.0 mmol) was added, and the reaction mixture was stirred for 30 minutes. Propionaldehyde (2.2 mL, 30.4 mmol), phenyl crotonate (5.5 mL, 36.6 mmol), and 30 mL of dry dichloroethane were then added, and the reaction was stirred at ambient temperature for 48 hours. The solvent was then removed by rotary evaporation, and the crude mixture was stirred with a 1:1:1 THF:MeOH:4 N HCl mixture (39 mL) for 1 hour. Water was then added (30 mL) and the layers were separated. The aqueous layer was extracted with ethyl acetate three times, and then the combined organic layers were washed twice with aqueous saturated NaHCO₃, dried over Na₂SO₄, filtered, and concentrated. Flash column chromatography in 4:1 hexanes:ethyl acetate (*R_f* = 0.12, 7:1 hexanes:ethyl acetate) provided 5.13 g (76%) of **5** in a 4.3:1 ratio of separable *syn:anti* diastereomers (88% *ee* for the *syn* diastereomer). IR (neat): 3446 (br, s), 1756 (s), 1646 (s), 1594 (s), 1495 (s), 1383 (s), 1349 (s) cm⁻¹; ¹H NMR: δ 7.53 (2 H, t, *J* = 7.8 Hz), 7.38 (1 H, t, *J* = 7.4 Hz), 7.22 (2 H, d, *J* = 7.0 Hz), 4.13 (1 H, m), 2.79 (1 H, m), 2.33 (1 H, d, *J* = 5.0 Hz), 2.06 – 1.9 (2 H, m), 1.8 – 1.7 (2 H, m), 1.23 (3 H, t, *J* = 7.5 Hz), 1.18 (3 H, t, *J* = 7.4 Hz); ¹³C NMR: δ 173.7, 150.4, 129.4, 125.9, 121.5, 73.5, 52.7, 27.5, 20.6, 12.1, 10.2. HRMS (FAB) Calc'd for C₁₃H₁₈O₃ (M + Na)⁺: 245.1148. Found (M + Na)⁺: 245.1152.

¹H NMR of the *anti* diastereomer: δ 7.53 (2 H, t, *J* = 7.8 Hz), 7.38 (1 H, t, *J* = 7.4 Hz), 7.22 (2 H, d, *J* = 7.0 Hz), 3.90 (1 H, m), 2.79 (1 H, m), 2.46 (1 H, d), 2.06 – 1.9 (2 H, m), 1.8 – 1.7 (2 H, m), 1.23 (3 H, t, *J* = 7.5 Hz), 1.18 (3 H, t, *J* = 7.4 Hz).

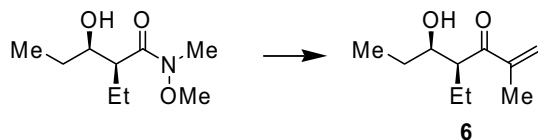
Proof of Stereochemistry: Stereochemical ratios were determined in comparison to racemic materials prepared via the reductive aldol reaction using *racemic*-BINAP as ligand. Absolute stereochemistry established in comparison to authentic 2(*S*),3(*R*) isomer prepared via a Crimmins-type aldol reaction using the thiazolidinethione butyrate to provide the appropriate Evans-*syn* aldol product, followed by conversion to the phenyl ester (Chaudhary, K and Crimmins, M. T. *Org. Lett.* **2000**, 2, 775).

Chiral CE (5% γ-cyclodextrin, -15 kV, 20°C) analysis of reductive aldol product:

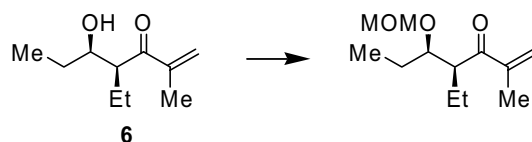




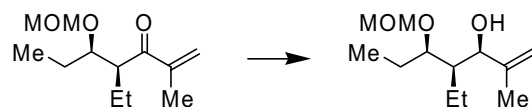
(2*S*,3*R*)-2-ethyl-3-hydroxy-N-methoxy-N-methyl pentanamide. To a flame-dried, 250 mL flask was added 3.4680 g (35.56 mmol) *N,O*-dimethylhydroxylamine hydrochloride under N₂. To the flask was then added 13.9 mL dry THF and the solution cooled to -10°C. Subsequently, a solution of trimethylaluminum (17.8 mL, 2.0 M in toluene, 35.5 mmol) was added dropwise via syringe over 30 minutes. When the addition was complete, the reaction mixture was warmed to 0°C and stirred for 10 minutes, then warmed to ambient temperature and stirred for 15 minutes. The reaction mixture was then returned to a -10°C bath, and a flame-dried addition funnel was fitted to the flask. A solution of β-hydroxyester **5** (2.6311 g, 11.85 mmol) in 12 mL dry THF/15 mL dry CH₂Cl₂ was added dropwise via addition funnel at -10°C. Once the addition was complete, the reaction flask was warmed to 0 °C over 10 min. The reaction was stirred at 0 °C for 3 hours, and was then transferred to a rapidly stirring 0°C 1:1 mixture of CH₂Cl₂:0.5 M HCl. This was stirred at 0°C for 1 h, then allowed to warm to ambient temperature and stirred overnight. The layers were separated, the aqueous layer was extracted with twice with CH₂Cl₂, and the combined organic layers were washed with brine, dried over Na₂SO₄, filtered, and concentrated. The product was purified via flash column chromatography with 2:1 hexanes:ethylacetate (*R_f* = 0.22), providing 2.1731 g (97%) of product. ¹H NMR: δ 3.70 (3 H, s), 3.68 (1 H, m), 3.27 (1 H, s), 3.21 (3 H, s), 2.91 (1 H, m), 1.9 – 1.4 (4 H, m), 0.94 (3 H, t, *J* = 7.4 Hz), 0.88 (3 H, t, *J* = 7.5 Hz); ¹³C NMR: δ 177.8, 74.2, 61.9, 46.3, 32.3, 27.6, 19.5, 12.7, 10.9.



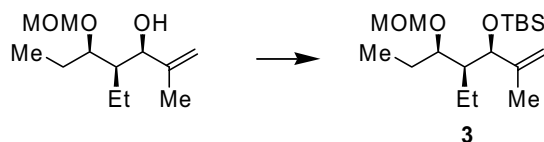
(4*S*,5*R*)-4-ethyl-5-hydroxy-2-methylhept-1-en-3-one (6). A 100 mL flask was filled with 20 mL dry ether and 3.6 mL of 2-bromopropene and cooled to -78°C under N₂. Next, a solution of *t*-BuLi (51 mL, 1.59 M in pentane) was added dropwise via addition funnel over 40 minutes. The reaction was stirred 1 hour at -78°C and then allowed to warm to room temperature. Over the course of 20 minutes, this mixture was then transferred by cannula to a solution of the Weinreb amide (3.0785 g, 16.29 mmol) in 108 mL of dry THF at -78°C. Stirring was continued at -78°C for 2 hours and then the reaction was transferred via cannula to a rapidly stirred 2:1 mixture of saturated aqueous NH₄Cl:THF at 0°C. After stirring at 0°C for 20 min the mixture was warmed to room temperature and stirred overnight. The reaction mixture was then partitioned between 450 mL brine and 750 mL ether. The layers were separated the aqueous layer was extracted with ether (3X). The organic layer was dried over Na₂SO₄, filtered, and concentrated. Purification was carried out via flash column chromatography in 2:1 hexanes:ethyl acetate (*R_f* = 0.58), yielding 2.4571 g (88%) of compound **6**. ¹H NMR: δ 5.97 (1 H, s), 5.83 (1 H, s), 3.64 (1 H, dddd, *J* = 4.6 Hz), 3.20 (1 H, dt, *J* = 8.9, 4.6 Hz), 2.52 (1 H, s), 1.87 (3 H, s), 1.70 (2 H, m), 1.41 (2 H, m), 0.93 (3 H, t, *J* = 7.4 Hz), 0.82 (3 H, t, *J* = 7.5 Hz); ¹³C NMR: δ 207.6, 146.1, 74.1, 51.1, 28.2, 20.9, 18.0, 12.7, 10.8. MS (ESI) Calc'd for C₁₀H₁₈O₂ (M + Na)⁺: 193.12. Found (M + Na)⁺: 193.1.



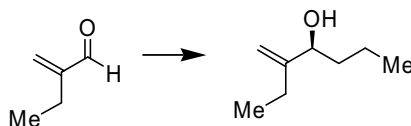
(4*S*,5*R*)-4-ethyl-5-(methoxymethoxy)-2-methylhept-1-en-3-one. A solution of β -hydroxy ketone **6** (1.3667 g, 8.04 mmol) in dry CH_2Cl_2 (16.1 mL) was cooled to 0°C . Subsequently, diisopropylethylamine (4.2 mL, 24.12 mmol) and chloromethyl methyl ether (1.83 mL, 24.12 mmol) were added and the mixture was stirred for 30 minutes at 0°C , then allowed to slowly warm to RT and stir for 12 h. The reaction was quenched by addition of water and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2X) and the combined organic layers were washed with brine, dried over Na_2SO_4 , filtered, and concentrated. The unpurified material (1.6789 g, 97%) was clean by both NMR and TLC analysis and was used directly without further purification. ^1H NMR: δ 5.93 (1 H, s), 5.79 (1 H, s), 4.62 (2 H, s), 3.67 (1 H, ddd, $J = 9.1, 6.7, 4.4$ Hz), 3.36 (3 H, s), 3.35 (1 H, m), 1.87 (3 H, s), 1.69 (2 H, m), 1.38 (2 H, dq, $J = 21.7, 7.3$ Hz), 0.88 (3 H, t, $J = 7.4$ Hz), 0.80 (3 H, t, $J = 7.5$ Hz); ^{13}C NMR: δ 205.0, 146.5, 125.1, 96.9, 80.9, 56.4, 50.8, 25.9, 22.6, 18.2, 12.4, 10.0.



(3*R*,4*R*,5*R*)-4-ethyl-5-(methoxymethoxy)-2-methylhept-1-en-3-ol. A solution of α,β -unsaturated ketone **7a** (2.2176 g, 10.36 mmol) in dry CH_2Cl_2 (20.7 mL) was cooled to -78°C and diisobutylaluminum hydride (18.7 mL, 1.0 M in hexanes, 18.65 mmol) was added dropwise via syringe. Stirring was continued at -78°C for 2 h, then the reaction was quenched by cannula transfer to a rapidly stirring 5:2 mixture of saturated sodium potassium tartrate (aq): CH_2Cl_2 (217 mL:87mL) and stirred overnight. After addition of water, the layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2X). The combined organic layers were dried over Na_2SO_4 , filtered, and concentrated. Purification and separation of the diastereomers was carried out using flash column chromatography in 4:1 hexanes:ethyl acetate ($R_f = 0.38$), yielding 1.91 g (85%) of allylic alcohol (10:1 ratio of diastereomers by analysis of crude NMR). $[\alpha]_D = +2.5^\circ$ (c 0.82, CHCl_3); IR (THF) 3514 (br), 3704, 2360, 2344, 2248 (s), 1654 (s), 1559, 1542, 1382, 1289, 1027, 991; ^1H NMR: δ 5.15 (1 H, s), 5.07 (1 H, s), 4.87 (1 H, d, $J = 6.8$ Hz), 4.81 (1 H, d, $J = 6.8$ Hz), 4.26 (1 H, dd, $J = 7.7, 4.3$ Hz), 4.09 (1 H, d, $J = 4.3$ Hz), 3.87 (1 H, ddd, $J = 8.9, 5.5, 2.4$ Hz), 3.57 (3 H, s), 1.85 (3 H, s), 1.84 (2 H, m), 1.71 (1 H, m), 1.53 (1 H, m), 1.37 (1 H, m), 1.09 (3 H, t, $J = 7.4$ Hz), 1.08 (3 H, t, $J = 7.5$ Hz); ^{13}C NMR: δ 146.4, 113.3, 96.9, 82.1, 76.9, 56.4, 44.1, 23.9, 19.7, 17.9, 12.9, 11.3; MS (ESI) Calc'd for $\text{C}_{12}\text{H}_{24}\text{O}_3$ ($\text{M} + \text{Na}$) $^+$: 239.16. Found ($\text{M} + \text{Na}$) $^+$: 239.0.



(5R,6S,7R)-5,6-diethyl-9,9,10,10-tetramethyl-7-(prop-1-en-2-yl)-2,4,8-trioxa-9-silaundecane (3). Alcohol **7b** (1.1160 g, 5.17 mmol) and 2,6-lutidine (2.1 mL, 18.08 mmol) were dissolved in CH_2Cl_2 (43 mL) and the solution cooled to 0°C . A solution of TBSOTf (1.8 mL, 7.75 mmol) in 8 mL CH_2Cl_2 was then added dropwise by cannula. After stirring at 0°C for 30 minutes, the solution was allowed to warm to room temperature and stir for an additional 2.5 hours. The reaction mixture was then transferred to a solution of saturated aqueous NaHCO_3 allowed to stir for 30 minutes. The layers were separated and the organic layer was washed with 1 M HCl, brine, and saturated NaHCO_3 . The organic layer was then dried over Na_2SO_4 , filtered, and concentrated. Purification via flash column chromatography in 15:1 hexanes:ethyl acetate ($R_f = 0.31$) provided 1.1865 g (70%) of silyl ether **3**. $[\alpha]_D = +31^\circ$ (c 0.14, CHCl_3); IR (neat): 3445 (br), 2957, 2929, 1718, 1461, 1225, 1112 cm^{-1} ; IR (neat): 2957, 2930, 2882, 2359, 1472, 1250, 1042, 835 cm^{-1} ; ^1H NMR: δ 4.92 (1 H, s), 4.86 (1 H, s), 4.70 (1 H, d, $J = 6.6$ Hz), 4.59 (1 H, d, $J = 6.5$ Hz), 3.99 (1 H, d, $J = 7.6$ Hz), 3.73 (1 H, ddd, $J = 7.7, 6.2, 1.8$ Hz), 3.36 (3 H, s), 3.36 (1 H, m), 1.70 (1 H, m), 1.63 (3 H, s), 1.55 – 1.35 (2 H, m), 1.17 (1 H, m), 0.92 (3 H, t, $J = 8.1$ Hz), 0.89 (3 H, t, $J = 7.4$ Hz), 0.88 (9 H, s), 0.02 (3 H, s), -0.02 (3 H, s); ^{13}C NMR: δ 147.0, 113.6, 97.7, 80.4, 78.0, 55.7, 46.7, 28.0, 26.4, 19.1, 18.7, 17.9, 14.4, 11.1, -3.9, -5.0. MS (ESI) Calc'd for $\text{C}_{18}\text{H}_{38}\text{O}_3\text{Si}$ ($\text{M} + \text{Na}$) $^+$: 353.25. Found ($\text{M} + \text{Na}$) $^+$: 353.2.

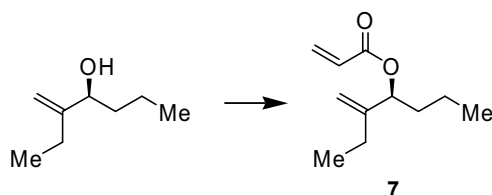
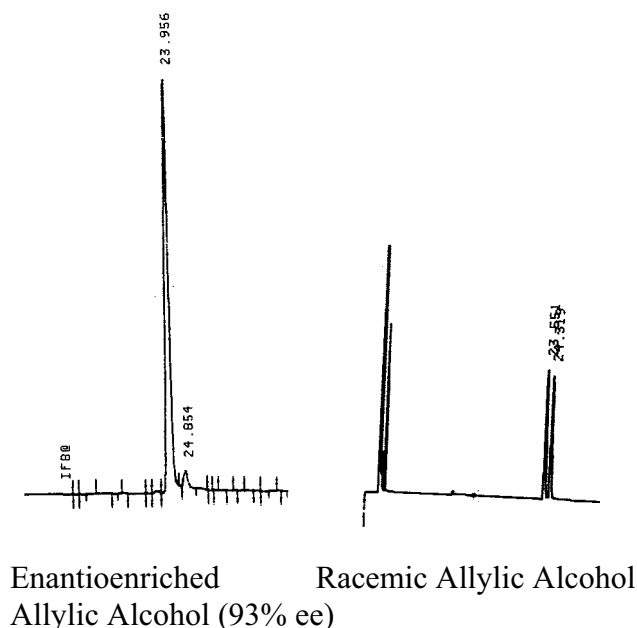


(S)-2-ethyl-1-hexen-3-ol. A solution of propyl magnesium bromide (172 mL, 1.76 M in ether, 303 mmol) was added dropwise via addition funnel to a -78°C solution of 2-ethylacrolein (17.0 g, 202 mmol) in 404 mL of dry THF. The reaction was stirred at -78°C for 30 minutes, warmed to 0°C and then stirred for an additional 5 hours. The reaction was then quenched by addition of saturated NH_4Cl (aq). After separating the layers, the aqueous layer was extracted with ether three times, and the combined organic layers were then washed with brine and water, sequentially. The organic layer was then dried over Na_2SO_4 , filtered, and concentrated. The crude product was purified using flash column chromatography in 4:1 hexanes:ethyl acetate ($R_f = 0.53$), providing 18.5838 g (72%) of the racemic allylic alcohol. ^1H NMR: δ 5.01 (1 H, s), 4.85 (1 H, s), 4.11 (1 H, dd, $J = 6.3$ Hz), 2.08 (2 H, m), 1.6 – 1.2 (4 H, m), 1.08 (3 H, t, $J = 7.4$ Hz), 0.95 (3 H, t, $J = 7.3$ Hz); ^{13}C NMR: δ 136.2, 108.7, 76.9, 75.9, 66.3, 38.1, 20.6, 19.3.

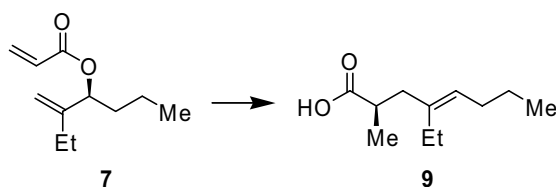
The racemic allylic alcohol prepared above (5.6197 g, 43.9 mmol) was dissolved in 175 mL of dry CH_2Cl_2 and added to a 500 mL flask containing 1.4 g of activated 3 Å molecular sieve powder. (*D*)-Diisopropyl tartrate (1.4 mL, 6.59 mmol) was added and the reaction mixture was cooled to -20°C . After addition of freshly distilled $\text{Ti}(\text{O}i\text{Pr})_4$ (1.3 mL, 4.39 mmol), the reaction was stirred at -20°C for 30 minutes. Subsequently, *t*-butyl hydroperoxide (10.2 mL, 3 M in toluene, 30.7 mmol, dried for 30 min over activated 4 Å molecular sieves) was added by cannula and the reaction was stirred at -20°C . Reaction progress was monitored by chiral GC analysis of the remaining starting material. After 34 h, the reaction was quenched by additional of 18 g FeSO_4 , 6 g citric acid, and 55 mL water. The mixture was allowed to warm to ambient temperature and stir for 30 minutes. The layers were then separated and the aqueous layer was extracted with CH_2Cl_2 , dried over Na_2SO_4 , filtered, and concentrated to ca. 175 mL. At this time, 50 mL of 30% NaOH in brine was added and the mixture allowed to stir for 30

minutes. The layers were then separated and the aqueous layer was extracted with CH_2Cl_2 . The combined organic layers were then washed with brine, dried over Na_2SO_4 , filtered, and concentrated. NMR analysis of the unpurified mixture showed 61% conversion, and GC analysis (*Supelco*, β -dex, 80°C , 20 psi) showed the recovered allylic alcohol to have an enantiomeric purity of 93% ee. Purification was facilitated after carrying out acylation and the material was therefore used directly in the next step.

Chiral GLC (β -dex, Supelco, 80°C , 20 psi) analysis of kinetic resolution of allylic alcohol:



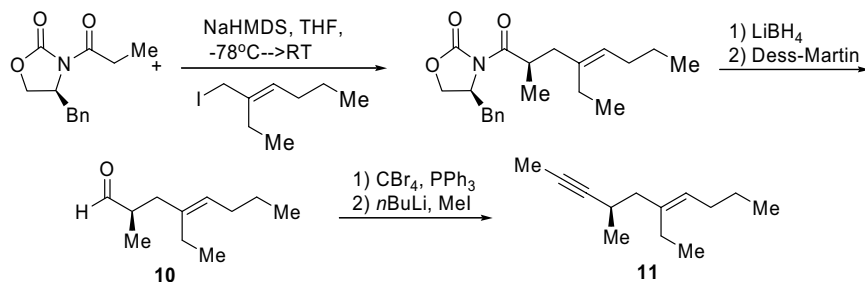
(*S*)-2-ethyl-1-hexen-3-yl acrylate (7). Triethylamine (12.7 mL, 91.1 mmol) was added to a solution of the unpurified reaction mixture obtained above (7.7565 g in 201 mL dry CH_2Cl_2) and the mixture stirred at room temperature for 45 minutes. The reaction mixture was then cooled to 0°C and acryloyl chloride (5.4 mL, 66 mmol) was added dropwise. The mixture was allowed to slowly warm to ambient temperature and then stir for 16 hours. The reaction was then quenched with 1 M HCl and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 , and the combined organic layers were then washed with saturated NaHCO_3 (aq) and brine, dried over Na_2SO_4 , filtered, and concentrated. The crude mixture was purified on a silica gel column in 10:1 hexanes:ethyl acetate (R_f allylic acrylate = 0.71, R_f epoxy acrylate = 0.31), furnishing 2.5852 g of chiral allylic acrylate **7** (83% yield based on 61 % conversion in the kinetic resolution). $[\alpha]_D = -17^\circ$ (c 0.54, CHCl_3); IR (neat): 2959, 2360, 1732, 1558, 1540, 1183 cm^{-1} ; ^1H NMR: δ 6.40 (1 H, d, $J = 24.0$ Hz), 6.15 (1 H, dd, $J = 13.6, 9.6$ Hz), 5.82 (1 H, d, $J = 12.0$ Hz), 5.29 (1 H, dd, $J = 8.8$ Hz), 5.02 (1 H, s), 4.89 (1 H, s), 2.07 (2 H, q, $J = 8.4$ Hz), 1.65 (2 H, m), 1.32 (2 H, m), 1.09 (3 H, t, $J = 10.0$ Hz), 0.92 (3 H, t, $J = 9.6$ Hz); ^{13}C NMR: δ 165.5, 149.2, 130.2, 128.9, 109.9, 77.1, 35.3, 24.4, 18.6, 13.7, 12.0.

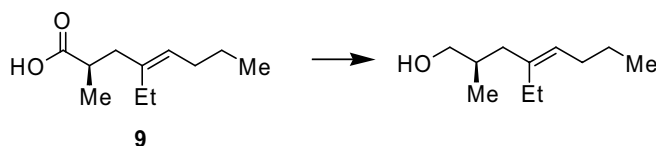


(*R,E*)-4-ethyl-2-methyloct-4-enoic acid (9). In glove box under an argon atmosphere, [Rh(cod)Cl]₂ (0.0845 g, 0.171 mmol, 1.25 mol%) and (*S,S*)-Me-DuPhos (0.1050 g, 0.342 mmol, 2.5 mol%) were weighed into an oven-dried flask, which was capped with a rubber septum and removed from the glove box. The catalyst-ligand combination was dissolved in benzene (40 mL) and stirred for 30 minutes. Chlorodimethylsilane (1.8 mL, 17.12 mmol) was added and the mixture was stirred for 5 min before addition of allylic acrylate **7** (2.4956 g, 13.7 mmol) in benzene (5.7 mL). The mixture was stirred at room temperature for 4 h, then concentrated in vacuo. The remaining material was purified by silica gel chromatography using a gradient elution with 2:1 to 1:1 hexanes:ethyl acetate (*R_f* = 0.69, 2:1 hexanes:ethyl acetate). The procedure provided 2.2178 g (88%) of the rearrangement product **9**. ¹H NMR: δ 11.09 (1 H, br s), 5.15 (1 H, t, *J* = 7.2 Hz), 2.61 (1 H, hex, *J* = 6.9 Hz), 2.45 (1 H, dd, *J* = 7.8 Hz), 2.0 (5 H, m), 1.35 (2 H, hex, *J* = 7.4 Hz), 1.14 (3 H, d, *J* = 6.8 Hz), 0.96 (3 H, t, *J* = 7.6 Hz), 0.88 (3 H, t, *J* = 7.4 Hz); ¹³C NMR: δ 182.0, 137.6, 127.0, 40.3, 37.8, 29.6, 23.0, 22.6, 16.4, 13.6, 12.9.

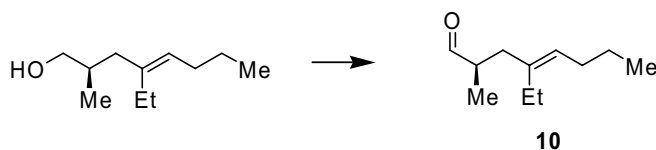
Proof of Stereochemistry: The stereochemistry of the methyl stereocenter was proven by comparison to an authentic sample prepared via Evans' alkylation protocol using (*S*)-(+)-4-benzyl-3-propionyl-2-oxazolidinone (Evans, D. A.; Ennis, M. D.; Mathre, D. J. *J. Am. Chem. Soc.* **1982**, *104*, 1737) as describe below. Both the Evans alkylation product and the reductive Claisen rearrangement product were reduced to the alcohol, oxidized to aldehyde **10**, and then converted to methyl alkyne **11**. Chiral GC analysis of **11** showed both products to possess the same absolute configuration (see below for chromatograms).

Structure Proof:

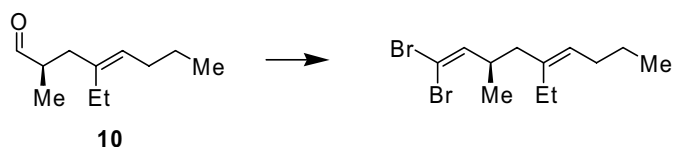




(R,E)-4-Ethyl-2-methyloct-4-en-1-ol. In glove box, lithium aluminum hydride (0.6836 g, 18.0 mmol) was weighed into an oven-dried flask, capped with a septum, and removed from box. To this was added 12 mL of dry THF and the suspension cooled to 0°C. A solution of carboxylic acid **9** (2.2109 g, 12.0 mmol in 12 mL THF) was then added with stirring. The mixture was stirred at 0°C for an additional hour and then allowed to slowly warm to room temperature. After 18 h, the reaction was quenched by slow dropwise addition of water. Once all bubbling had ceased, 10% aq H₂SO₄ was added. The layers were separated and the aqueous layer extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na₂SO₄, filtered, and concentrated. This procedure provided 1.8175 g (89%) of the desired alcohol, which was used without further purification. IR (neat): 3421, 1729, 1465, 1154 cm⁻¹; ¹H NMR: δ 5.12 (1 H, t, J = 7.2 Hz), 3.45 (1 H, dd, J = 23.0, 5.4 Hz), 3.45 (1 H, ddd, J = 33.6, 10.7, 5.4 Hz), 2.05 (5 H, m), 1.81 (2 H, q, J = 17.0 Hz), 1.34 (2 H, hex, J = 7.4 Hz), 0.94 (3 H, t, J = 7.6 Hz), 0.88 (3 H, t, 7.3 Hz), 0.86 (3 H, d, J = 6.4 Hz); ¹³C NMR: δ 139.2, 126.6, 69.0, 41.5, 34.2, 30.0, 23.5, 23.1, 17.2, 14.2, 13.5. HRMS (ESI) Calc'd for C₁₁H₂₂O (M + H)⁺: 171.1779. Found (M + H)⁺: 171.1760.

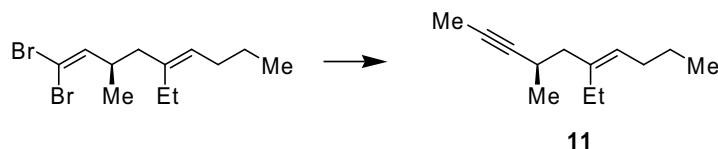


(R,E)-4-ethyl-2-methyloct-4-enal (10). To a solution of Dess-Martin periodinane (4.9460 g, 11.6 mmol) in CH₂Cl₂ (57 mL) was added a solution of the alcohol prepared above (1.3158 g, 7.73 mmol in 20 mL CH₂Cl₂). The reaction was stirred at room temperature for 6 hours, then treated with 120 mL of a 1:1 mixture of saturated NaHCO₃ (aq):saturated Na₂S₂O₃ (aq) for 30 minutes. After diluting with ether and separating the layers, the organic layer was washed once with water, and the combined aqueous layers were extracted with ether three times. The combined organic layers were dried over Na₂SO₄, filtered, and concentrated. Flash column chromatography in 4:1 hexanes:ethyl acetate (*R_f* = 0.75) provided 1.1213 g of aldehyde **10** (86%). ¹H NMR: δ 9.60 (1 H, d, J = 2.0 Hz), 5.11 (1 H, t, J = 7.2 Hz), 2.45 (2 H, m), 2.08 – 1.9 (5 H, m), 1.34 (2 H, hex, J = 7.4 Hz), 1.04 (3 H, d, J = 6.9 Hz), 0.95 (3 H, t, J = 7.6 Hz), 0.85 (3 H, t, J = 7.4 Hz); ¹³C NMR: δ 205.8, 137.16, 127.7, 44.9, 38.0, 30.0, 23.4, 23.2, 14.2, 13.7, 13.4.



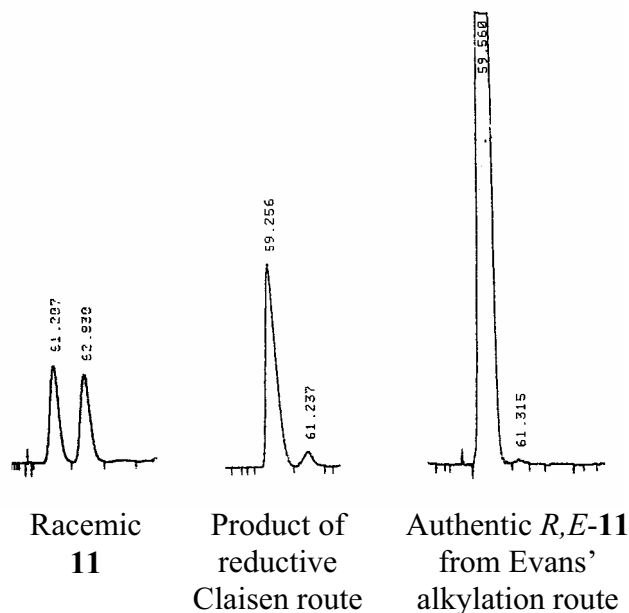
(R,E)-1,1-Dibromo-5-ethyl-3-methylnona-1,5-diene. Freshly-sublimed carbon tetrabromide (0.3630 g, 1.09 mmol) was dissolved in CH₂Cl₂ (1.1 mL) and cooled to 0°C. A solution of triphenylphosphine (0.5741 g, 2.19 mmol) in CH₂Cl₂ (1.5 mL) was then added dropwise. After the addition was complete, the mixture was cooled to -78°C and a solution of **10** (0.0920 g, 0.547 mmol) in CH₂Cl₂ (0.55 mL) was added in one portion. The reaction was stirred at -78°C for 30 minutes, then at 0°C for 20 min. After diluting with ether, the mixture was poured into brine, and the layers were separated. The aqueous layer was extracted with ether, and the combined organic layers were dried over Na₂SO₄, filtered, and

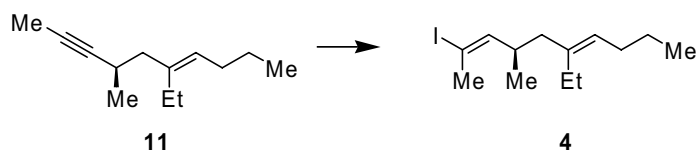
concentrated. Purification by flash column chromatography in 20:1 hexanes:ethyl acetate ($R_f = 0.38$) provided 0.1465 g (83%) of vinyl dibromide. ^1H NMR: δ 6.11 (1 H, d, $J = 9.6$ Hz), 5.05 (1 H, t, $J = 7.2$ Hz), 2.57 (1 H, m), 1.98 (6 H, m), 1.34 (2 H, hex, $J = 7.3$ Hz), 0.98 (3 H, d, $J = 6.7$ Hz), 0.96 (3 H, t, $J = 7.6$ Hz), 0.88 (3 H, t, $J = 7.5$ Hz); ^{13}C NMR: δ 144.7, 138.5, 127.4, 87.4, 43.3, 37.1, 30.0, 23.5, 23.1, 19.2, 14.2, 13.5.



(*R,E*)-6-ethyl-4-methyldec-6-en-2-yne (11). The vinyl dibromide (0.5977 g, 1.85 mmol) prepared above was dissolved in THF (18.5 mL) and cooled to -78°C . To this was added $n\text{BuLi}$ (2.2 mL, 1.69 M in hexanes, 3.70 mmol) dropwise with stirring. The reaction was then stirred for 5 min at -78°C , then 1 hour at -25°C . The reaction was then cooled to -78°C , and a solution of iodomethane (0.11 mL, 1.85 mmol) in THF (1.4 mL) was added dropwise. The reaction was then allowed to slowly warm to room temperature and stir for 1 hour. The mixture was treated with saturated NH_4Cl (aq) and diluted with ether. The layers were separated and the aqueous layer was extracted with ether. The combined organic layers were dried over Na_2SO_4 , filtered, and concentrated. Purification by flash column chromatography in neat hexanes ($R_f = 0.83$) afforded 0.3048 g (93%) of methyl alkyne **11**. Analysis of **11** by GC (Supelco, β -dex, 70°C , 20 psi) showed the enantiomeric purity to be 86% ee. $[\alpha]_D = -3^\circ$ (c 0.18, CHCl_3); IR (neat): 2963, 1716, 1652, 1540, 1005 cm^{-1} ; ^1H NMR: δ 5.02 (1 H, t, $J = 7.2$ Hz), 2.47 (1 H, m), 2.19 (1 H, dd, $J = 12.9, 7.6$ Hz), 1.98 (5 H, m), 1.76 (3 H, s), 1.35 (2 H, hex, $J = 7.3$ Hz), 1.07 (3 H, d, $J = 6.8$ Hz), 0.93 (3 H, t, $J = 7.6$ Hz), 0.88 (3 H, t, $J = 7.4$ Hz); ^{13}C NMR: δ 138.5, 126.7, 84.1, 75.4, 44.3, 29.6, 24.5, 23.1, 22.7, 20.8, 13.7, 13.1, 3.4. HRMS (HRCI) Calc'd for $\text{C}_{13}\text{H}_{22}$ ($\text{M} + \text{H}$) $^+$: 179.1779. Found ($\text{M} + \text{H}$) $^+$: 179.1804.

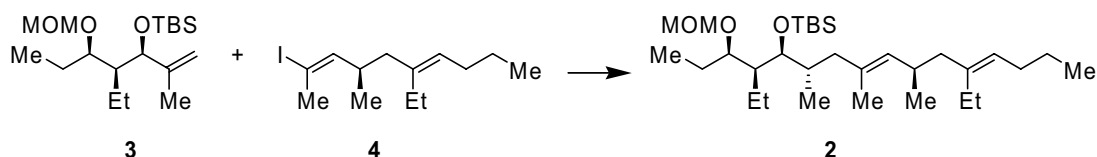
Chiral GLC (β -dex, Supelco, 75°C , 20 psi) analysis of methyl alkyne 11:





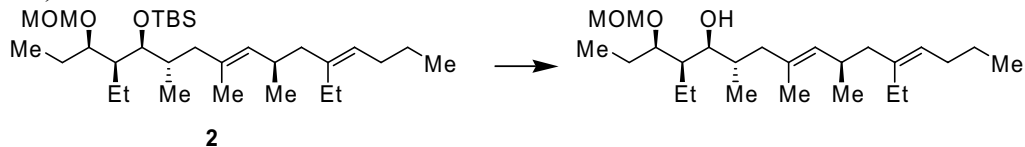
(*R,2E,6E*)-6-ethyl-2-iodo-4-methyldeca-2,6-diene (4). In glove box, bis(cyclopentadienyl)zirconium chloro hydride (Schwartz' reagent, 0.840 g, 3.26 mmol) was weighed into an oven-dried flask, which was then capped with a septum and removed from the box. Upon removal from glove box, the flask was immediately wrapped in aluminum foil. A solution of alkyne **11** (0.2321 g, 1.30 mmol) in CH₂Cl₂ (8.7 mL) was added and the reaction was stirred in the dark at room temperature for 2 hours. After this time, an additional 6 mL of CH₂Cl₂ was added and the reaction was cooled to 0°C. A saturated solution of I₂ in CCl₄ was added dropwise at 0°C until the reaction mixture became dark purple. This mixture was quenched by addition of saturated Na₂S₂O₃ (aq.), then CH₂Cl₂ was added and the layers separated. The aqueous layer was extracted with CH₂Cl₂ three times, then the combined organic layers were washed with water and brine. The organic solution was then dried over Na₂SO₄, filtered, and concentrated. Flash column chromatography in neat hexanes (*R_f* = 0.62) provided vinyl iodide **4** in a 4:1 ratio of separable regioisomers (0.3312 g overall yield, 84%; 68% yield of desired vinyl iodide **4**). ¹H NMR: δ 6.10 (1 H, d, *J* = 7.7 Hz), 5.03 (1 H, t, *J* = 6.4 Hz), 2.51 (1 H, m), 2.31 (3 H, s), 2.01 – 1.85 (6 H, m), 1.30 (2 H, hex, *J* = 9.4 Hz), 1.00 – 0.79 (9 H, m); ¹³C NMR: δ 147.7, 127.1, 92.5, 77.6, 44.1, 34.7, 30.1, 28.0, 23.5, 23.3, 20.4, 14.2, 13.5.

¹H NMR of the minor regioisomer: δ 6.20 (1 H, q, *J* = 4.3 Hz), 5.15 (1 H, t, *J* = 6.4 Hz), 2.15 (1 H, m), 2.01 – 1.85 (6 H, m), 1.61 (3 H, d, *J* = 8.6 Hz), 1.30 – 1.15 (2 H, m), 1.00 – 0.79 (9 H, m).

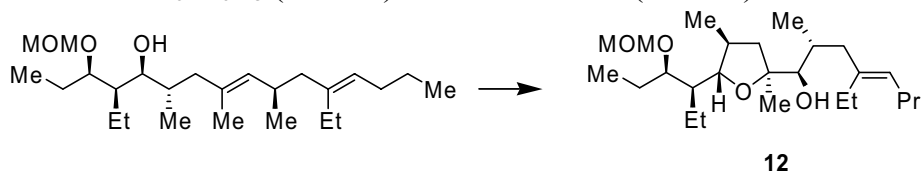


(*5R,6S,7S*)-5,6-diethyl-7-((*2S,4E,6R,8E*)-8-ethyl-4,6-dimethyldodeca-4,8-dien-2-yl)-9,9,10,10-tetramethyl-2,4,8-trioxa-9-silaundecane (2). A solution of alkene **3** (0.1598 g, 0.48 mmol) in THF (0.97 mL) was added to a -15°C stirred solution of 9-BBN (1.8 mL, 0.5 M in THF, 0.88 mmol). The mixture was allowed to slowly warm to RT and stir for 11 hours. To a separate flask containing vinyl iodide **4** (0.1347 g, 0.44 mmol) in DMF (1.5 mL), was sequentially added Cs₂CO₃ (0.2151 g, 0.66 mmol), PdCl₂(dppf) (0.0359 g, 0.044 mmol), and triphenylarsine (0.0135 g, 0.044 mmol). This was allowed to stir 30 minutes, before a solution of water (0.12 mmol, 6.6 mmol) was added. After addition of water, the the vinyl iodide solution was stirred for 5 minutes before the alkyl borane solution was added via cannula (0.5 mL THF rinse). The reaction mixture was then heated at 65°C for 8 hours. The mixture was cooled to room temperature and poured into water, diluted with ether and the layers separated. The aqueous layer was extracted with ether, then the combined organic layers were washed with water and brine, dried over Na₂SO₄, filtered, and concentrated. The isolated material was purified and diastereomers separated by flash column chromatography in 25:1 hexanes:ethyl acetate (*R_f* = 0.27), providing compound **2** (0.2062 g, 91 %). ¹H NMR: δ 5.02 (1 H, t, *J* = 7.2 Hz), 4.85 (1 H, d, *J* = 9.6 Hz), 4.72 (1 H, d, *J* = 6.8 Hz), 4.58 (1 H, d, *J* = 6.8 Hz), 3.36 (3 H, s), 3.35 (1 H, m), 2.46 (1 H, m), 2.26 (1 H, d, *J* = 13.3 Hz), 2.05 – 1.70 (8 H, m), 1.65 – 1.40 (6 H, m), 1.52 (3 H, s), 1.32 (2 H, q, *J* = 7.4 Hz), 0.98 – 0.80 (24 H, m), 0.75 (3 H, d, *J* = 6.8 Hz), 0.03 (6 H, s); ¹³C NMR: δ 139.9, 134.1, 133.6, 129.0, 97.2, 80.7, 77.9, 77.6, 56.0, 47.5, 44.9, 43.4, 35.4, 31.9, 31.1, 30.1, 27.4, 26.6, 23.6, 23.3, 21.1, 20.7, 18.9,

17.0, 16.4, 14.3, 14.2, 13.5, 10.4, -3.0, -3.9. MS (ESI) Calc'd for $C_{31}H_{62}O_3Si$ ($M + Na$)⁺: 533.44. Found ($M + Na$)⁺: 533.5.

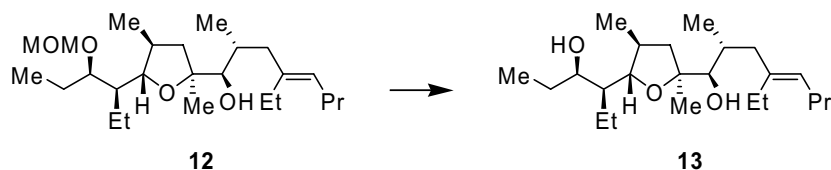


(3*R*,4*R*,5*S*,6*S*,8*E*,10*R*,12*E*)-4,12-diethyl-3-(methoxymethoxy)-6,8,10-trimethylhexadeca-8,12-dien-5-ol. Tetrabutylammonium fluoride (0.4 mL, 1.0 M in THF, 0.392 mmol) was added to a solution of silyl ether **2** (0.0500 g, 0.098 mmol) in dry THF (0.65 mL). After 2 hours, the reaction was quenched by addition of water, the layers separated, and the aqueous layer extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4 , filtered, and concentrated. The product was purified via flash column chromatography in 10:1 hexanes:ethyl acetate (R_f = 0.30) and the alcohol isolated (0.0281 g, 72%). [α]_D = +2° (*c* 0.34, $CHCl_3$); IR (neat): 3503 (br), 2961, 2929, 1716, 1652, 1457, 1092 cm^{-1} ; 1H NMR: δ 5.02 (1 H, t, *J* = 7.6 Hz), 4.91 (1 H, d, *J* = 9.1 Hz), 4.67 (1 H, d, *J* = 6.8 Hz), 4.65 (1 H, d, *J* = 6.8 Hz), 3.82 (1 H, ddd, *J* = 8.0, 7.2, 2.2 Hz), 3.55 (1 H, d, *J* = 5.6 Hz), 3.41 (1 H, m), 3.40 (3 H, s), 2.49 (1 H, m), 2.38 (1 H, d, *J* = 14.7), 2.04 – 1.46 (13 H, m), 1.52 (3 H, s), 1.32 (2 H, hex, *J* = 8.2 Hz), 0.94 (3 H, t, *J* = 7.6 Hz), 0.93 (3 H, t, *J* = 7.6 Hz), 0.91 (3 H, t, *J* = 8.6 Hz), 0.86 (3 H, t, *J* = 7.4 Hz), 0.86 (3 H, d, *J* = 6.6 Hz), 0.78 (3 H, d, *J* = 6.9 Hz); ^{13}C NMR: δ 139.5, 133.2, 131.6, 125.6, 95.9, 80.8, 77.1, 76.3, 56.1, 44.5, 42.7, 41.0, 33.5, 30.8, 29.6, 23.5, 23.1, 22.9, 20.8, 18.5, 16.1, 13.7, 13.1, 12.5, 10.7. MS (ESI) Calc'd for $C_{25}H_{48}O_3$ ($M + Na$)⁺: 419.35. Found ($M + Na$)⁺: 419.3.



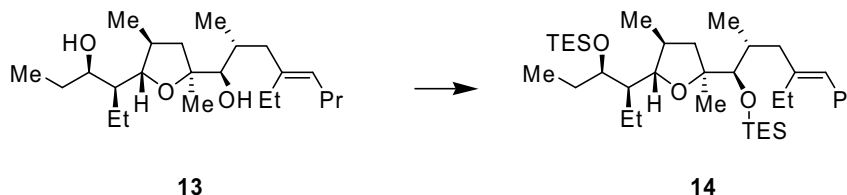
(3*R*,4*R*,5*S*,6*S*,8*S*,9*R*,10*R*,12*E*)-4,12-diethyl-5-furan-3-(methoxymethoxy)-6,8,10-trimethylhexadeca-12-ene-9-ol (12). The bis-homoallylic alcohol prepared above (0.0508 g, 0.128 mmol) was dissolved in dry CH_2Cl_2 (0.65 mL), then $VO(acac)_2$ (0.0023 g, 0.07 eq) was added, followed by sodium acetate (0.001 g, 0.1 eq). The flask was cooled to 0°C and *t*-butyl hydrogen peroxide (40 μ L, 5 M in nonane, 0.23 mmol) was added (the TBHP solution was dried over activated molecular sieves for 30 min prior to addition). This mixture was stirred at 0°C for 1.5 hours, then gradually warmed to room temperature. After 2 hours, the reaction had turned yellow, so added an additional 0.07 eq of $VO(acac)_2$ was added which immediately returned the reaction mixture to the original deep red color. This process was repeated after 4 hours. After 6 hours the reaction was treated with addition of satd. $Na_2S_2O_3$ (aq.). After stirring for 10 min, the mixture was partitioned between 1/3 CH_2Cl_2 /hexanes and 1% aqueous $NaHCO_3$. The layers were separated and the organic layer washed with 0.2 N aq. $NaHSO_4$, then with pH 7 phosphate buffer. The organic layers was then dried over Na_2SO_4 , filtered, and concentrated. The concoction was eluted on a silica gel column with 10:1 hexanes:ethyl acetate (R_f = 0.4), which provided 0.0225 g (42%) of the epoxidation/cyclization product **12** in a 6:1 ratio of separable diastereomers. [α]_D = -4° (*c* 0.32, $CHCl_3$); IR (neat): 3445 (br), 2957, 2929, 1718, 1461, 1225, 1112 cm^{-1} ; 1H NMR: δ 5.09 (1 H, t, *J* = 7.1 Hz), 4.69 (1 H, d, *J* = 6.6 Hz), 4.58 (1 H, d, *J* = 6.6 Hz), 3.74 (1 H, ddd, *J* = 8.8, 6.7, 1.8 Hz), 3.45 (1 H, m), 3.42 (1 H, dd, *J* = 12.0, 7.1 Hz), 3.35 (3 H, s), 2.69 (1 H, s), 2.15 (2 H, m), 1.95 (6 H, m), 1.85 – 1.15 (9 H, m), 1.08 (3 H, s), 1.05 (3 H, d, *J* = 6.3 Hz), 0.94 (3 H, t, *J* = 7.1 Hz), 0.92 (3 H, t, *J* = 5.6 Hz), 0.89 (3 H, t, *J* = 6.9 Hz), 0.87 (3 H, t, *J* = 7.4 Hz), 0.80 (3 H, d, *J* = 6.8 Hz).

^1H NMR of the minor diastereomer: δ 5.05 (1 H, t, J = 7.1 Hz), 4.78 (1 H, d, J = 6.4 Hz), 4.59 (1 H, d, J = 6.5 Hz), 3.83 (1 H, ddd, J = 9.0, 6.0, 2.5 Hz), 3.59 (1 H, m), 3.38 (3 H, s), 3.18 (1 H, dd, J = 13.4, 6.7 Hz), 3.06 (1 H, s), 2.15 (2 H, m), 1.95 (6 H, m), 1.85 – 1.15 (9 H, m), 1.08 (3 H, s), 1.05 (3 H, d, J = 6.3 Hz), 0.94 (3 H, t, J = 7.1 Hz), 0.92 (3 H, t, J = 5.6 Hz), 0.89 (3 H, t, J = 6.9 Hz), 0.87 (3 H, t, J = 7.4 Hz), 0.80 (3 H, d, J = 6.8 Hz).



(3R,4R,5S,6S,8S,9R,10R,12E)-4,12-diethyl-5-furan-6,8,10-trimethylhexadeca-12-ene-3,9-diol (13).

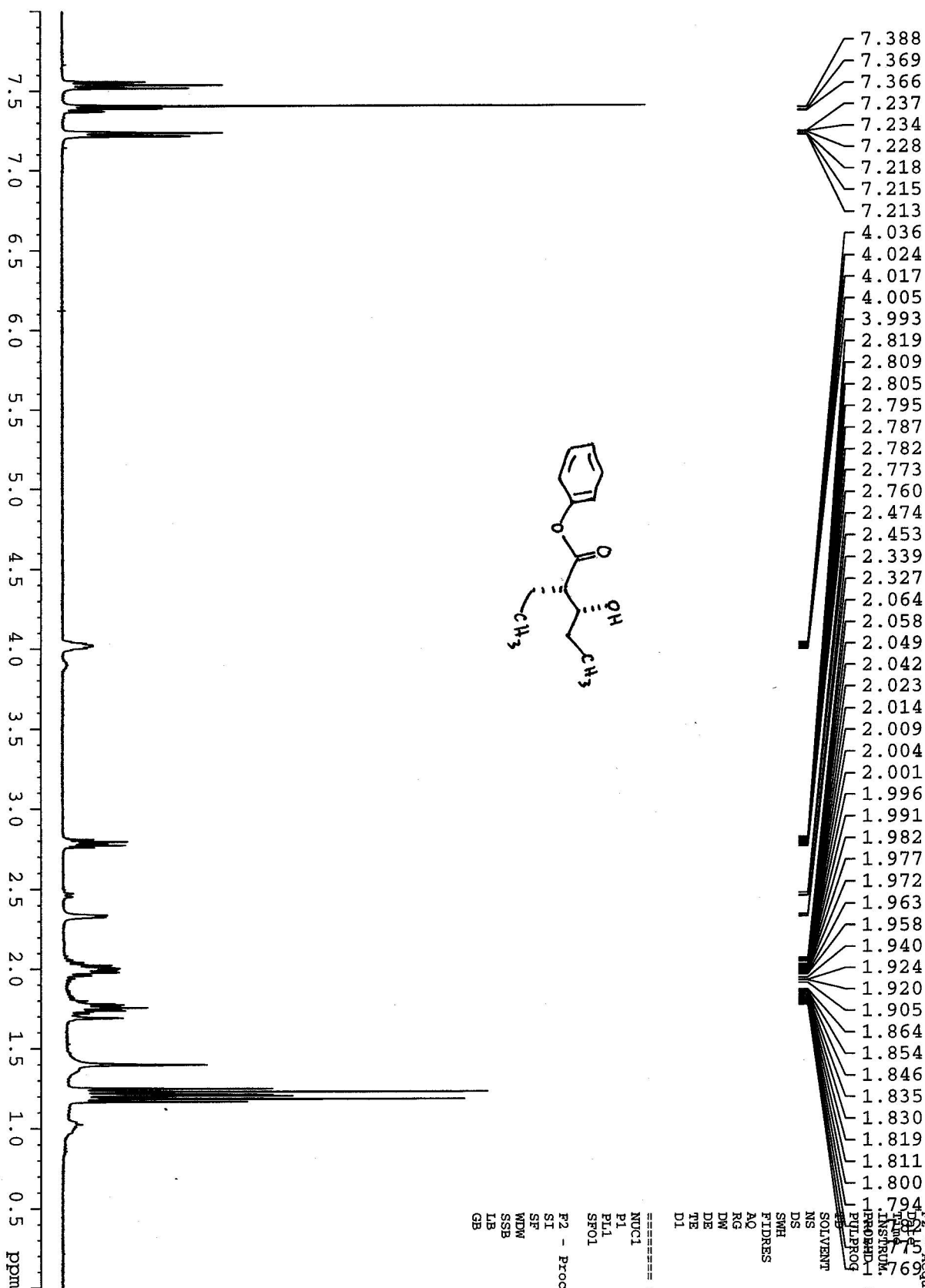
The MOM ether **12** prepared above (0.0293 g, 0.071 mmol) was dissolved in anhydrous methanol (3.6 mL) and 2 drops of conc. HCl were added. The mixture was then stirred at room temperature and monitored by TLC until complete disappearance of starting material was observed (5 hours), at which point the reaction was quenched by addition of satd. NaHCO_3 (aq). The mixture was diluted with ether and the layers were separated. The aqueous layer was extracted with ether and then the combined organic layers were washed with brine, dried over Na_2SO_4 , filtered, and concentrated. The unpurified product was sufficiently pure to characterize and use in the next step with a quantitative crude yield of **13** (0.0293g). $[\alpha]_D^{25} = +28^\circ$ (c 0.085, CHCl_3); IR (neat): 3445 (br), 2961, 2925, 2872, 1651, 1457, 1377, 1119 cm^{-1} ; ^1H NMR: δ 5.15 (1 H, t, J = 7.2 Hz), 3.84 (1 H, dd, 7.2 Hz), 3.69 (1 H, d, J = 7.5 Hz), 3.68 (1 H, m), 3.63 (1 H, dd, J = 10.2, 2.4 Hz), 3.53 (1 H, s), 2.30 (2 H, m), 2.17 (1 H, dd, J = 13.4, 7.1 Hz), 2.01 (6 H, m), 1.80 (2 H, m), 1.70 – 1.20 (6 H, m), 1.30 (3 H, s), 1.00 (3 H, d, J = 6.4 Hz), 0.98 (3 H, t, J = 7.6 Hz), 0.97 (3 H, t, J = 7.2 Hz), 0.95 (3 H, t, J = 7.4 Hz), 0.92 (3 H, t, J = 7.3 Hz), 0.87 (3 H, d, J = 6.8 Hz); ^{13}C NMR: δ 138.9, 126.6, 87.8, 85.6, 78.0, 72.6, 44.3, 42.7, 40.6, 36.0, 31.5, 29.5, 26.6, 24.9, 23.1, 22.4, 18.4, 15.6, 13.3, 13.0, 12.5, 10.7, 0.9. MS (ESI) Calc'd for $\text{C}_{23}\text{H}_{44}\text{O}_3$ ($\text{M} + \text{Na}$) $^+$: 391.32. Found ($\text{M} + \text{Na}$) $^+$: 391.3.



(3R,4R,5S,6S,8S,9R,10R,12E)-4,12-diethyl-5-furan-3,9-triethylsilyloxy-6,8,10-trimethylhexadeca-12-ene (14).

Diol **13** (0.0262 g, 0.071 mmol) was dissolved in dry CH_2Cl_2 and 2,6-lutidine (60 μL , 0.515 mmol) was added. The solution was cooled to 0°C and triethylsilyltriflate (50 μL , 0.221 mmol) was then added. After stirring at 0°C for 30 minutes, the mixture was warmed to room temperature, stirred 5 hours, and quenched by addition of satd. NaHCO_3 (aq). The aqueous layer was extracted with CH_2Cl_2 three times, then the combined organic layers were washed sequentially with 1 M HCl, brine, and satd. NaHCO_3 (aq). The organic solution was then dried over Na_2SO_4 , filtered, and concentrated. Purification was accomplished by flash column chromatography in neat hexanes (R_f = 0.35) and provided 0.0317 g (75%) of bis-triethylsilyl ether **14**. $[\alpha]_D^{25} = -5^\circ$ (c 0.05, CHCl_3); ^1H NMR: δ 5.05 (1 H, t, J = 6.9 Hz), 3.85 (1 H, ddd, J = 8.8, 2.8, 2.9 Hz), 3.45 (1 H, d, J = 1.9 Hz), 3.34 (1 H, dd, J = 8.8, 6.6 Hz), 2.23 – 1.62 (12 H, m), 1.51 (2 H, m), 1.34 (2 H, q, J = 7.3 Hz), 1.25 (1 H, m), 1.00 (3 H, d, J = 6.4 Hz), 0.96 (9H, t, J = 8.1 Hz), 0.94 (3 H, t, J = 7.3 Hz), 0.93 (9 H, t, J = 7.8 Hz), 0.92 (3 H, t, J = 7.2 Hz), 0.88 (3 H, t, J = 7.4 Hz), 0.83 (3 H, t, J = 7.4 Hz), 0.73 (3 H, d, J = 6.5 Hz), 0.62 (6 H, q, J = 8.0 Hz), 0.57 (6 H, q, J = 8.0 Hz); ^{13}C NMR: δ 140.1, 126.3, 86.4, 84.9, 81.8, 74.4, 49.3, 45.4, 44.0, 37.1,

32.8, 30.1, 30.0, 24.6, 23.6, 23.0, 20.2, 18.8, 14.7, 14.5, 14.0, 13.5, 10.6, 7.1, 7.0, 5.5, 5.1. MS (ESI)
Calc'd for $C_{35}H_{72}O_3Si_2$ ($M + H$)⁺: 597.50. Found ($M + H$)⁺: 597.5.

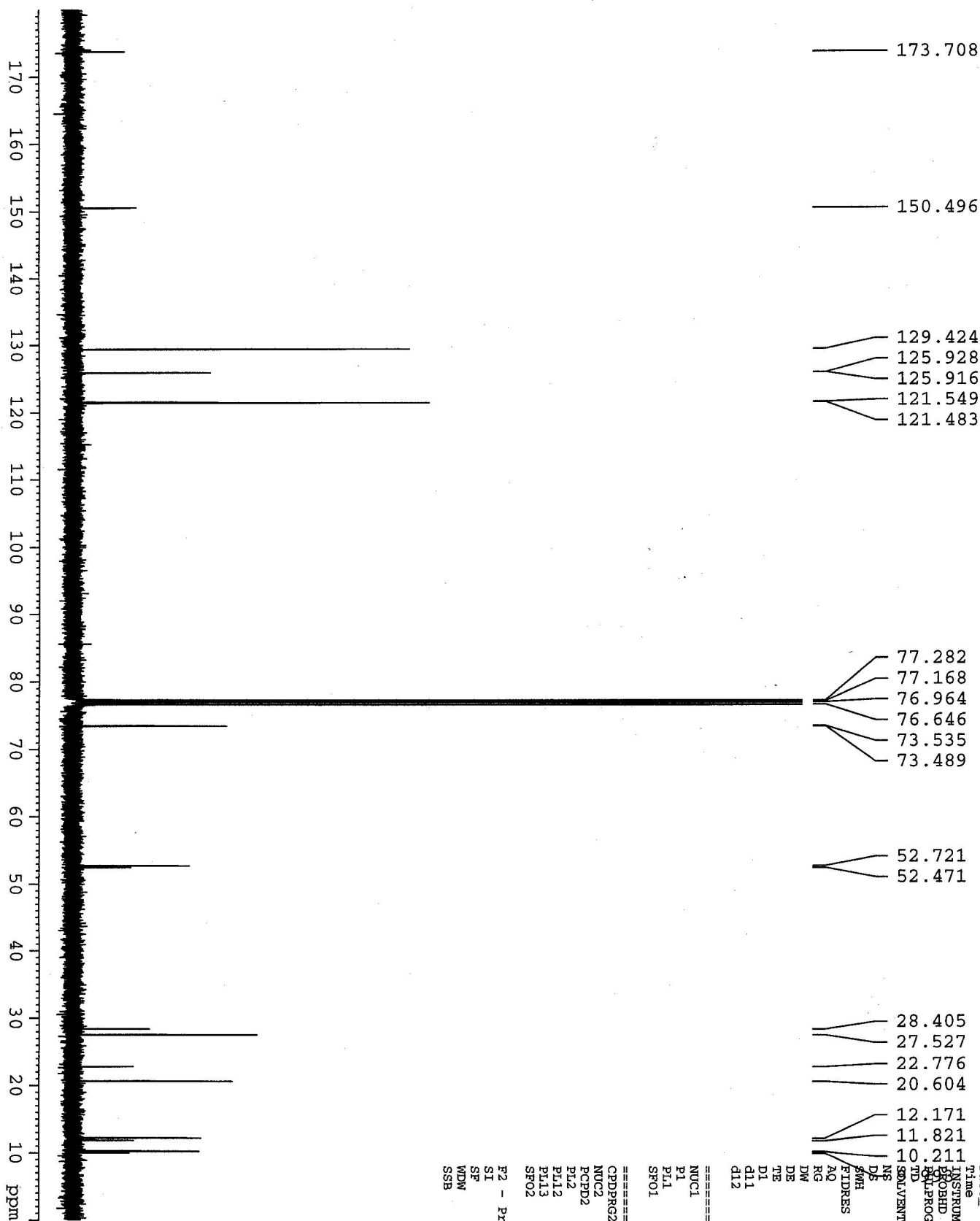


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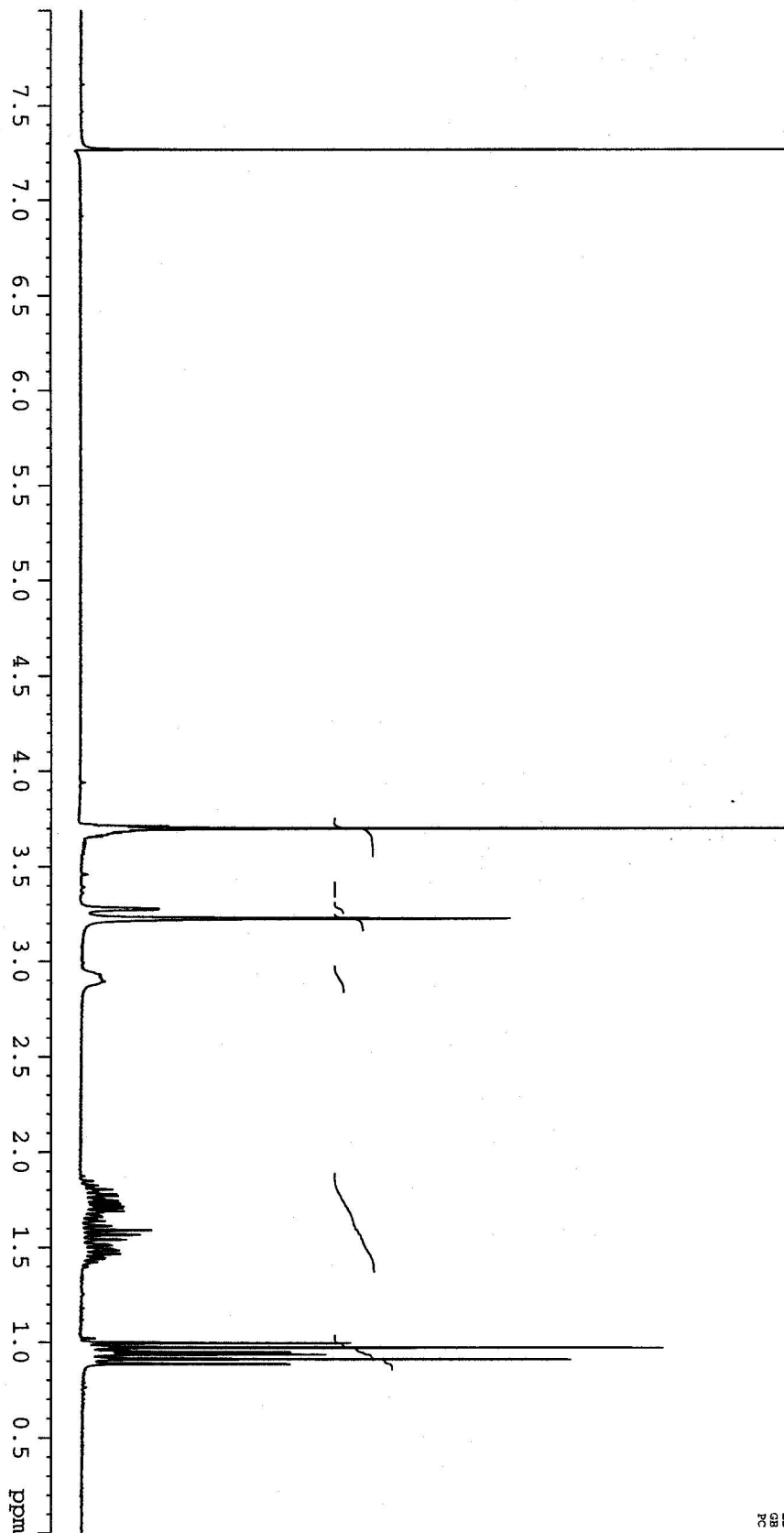
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DE 32.36 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

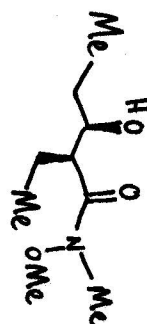
===== CHANNEL f1 =====
NUC1 13C
P1 7.05 usec
PL1 0.00 dB
SFO1 100.5413136 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 13.90 dB
PL13 22.00 dB
SFO2 399.8015992 MHz

F2 - Processing parameters
SI 65536
SF 100.5297927 MHz
WDW no
SSB 0



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180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

77.731
77.617
77.413
77.096
76.859
74.180
61.864

46.299

32.310
27.683
23.589
19.506

12.686
10.916

Current Data Parameters
NAME NF-111-41pC13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040130
Time 5.15

INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 20000
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 11585.2
DM 19.050 use
DE 6.00 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW no
SSB 0

Current Data Parameters
 NAME NF-IT-52-H
 EXTNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040618
 Time 0.10

INSTRUM spect
 P1 0.10

PROBHD 5mm QNP 1H/13C
 PULPROG zg30

TD 32768
 SOLVENT CDCl3

DS 64
 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 22.50 usec
 PL1 0.00 dB

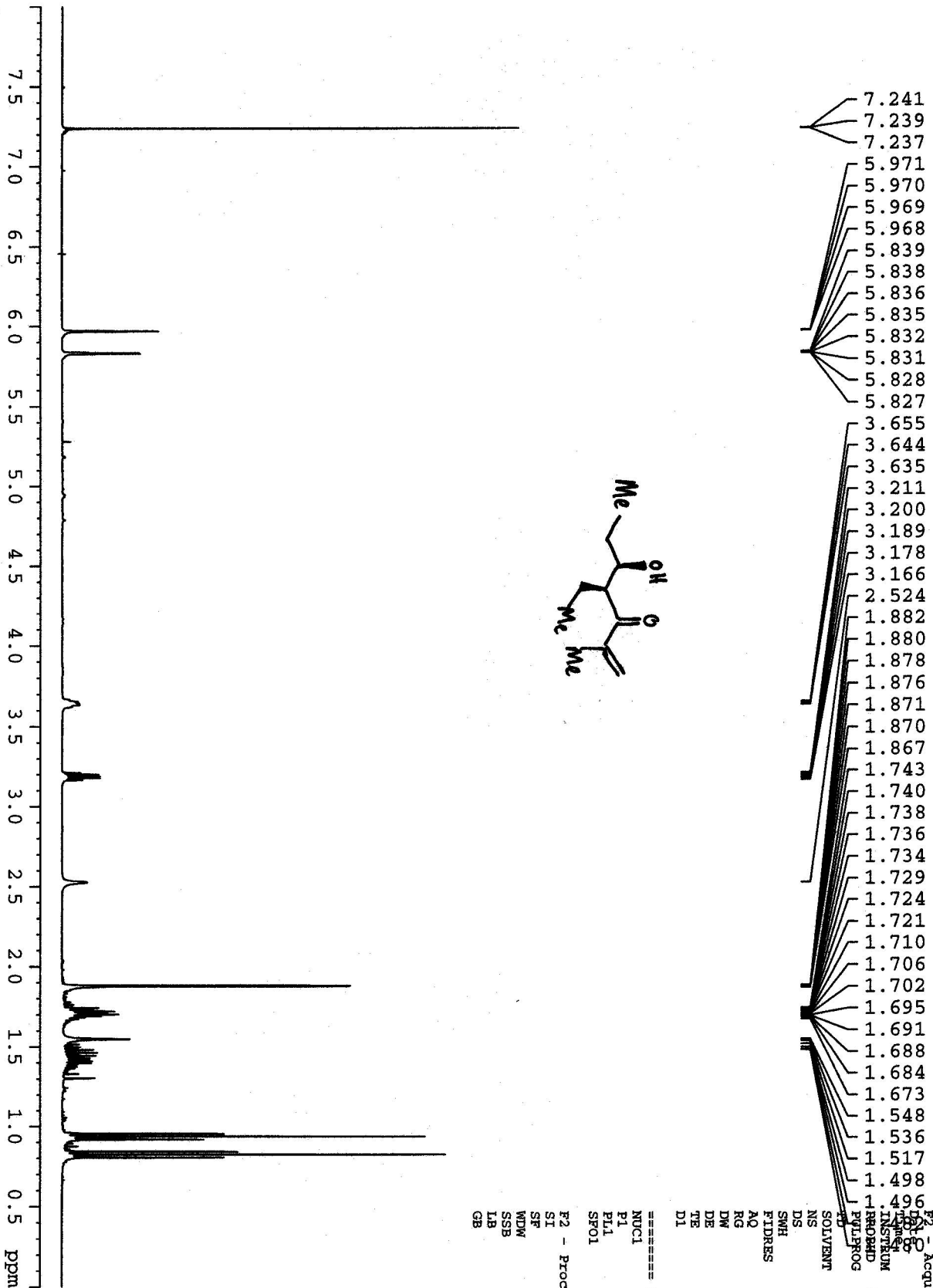
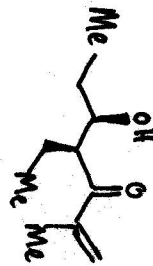
SFO1 400.132608 MHz

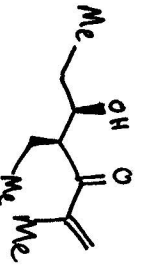
F2 - Processing parameters
 SI 32768

SF 400.1300172 MHz
 MDW no

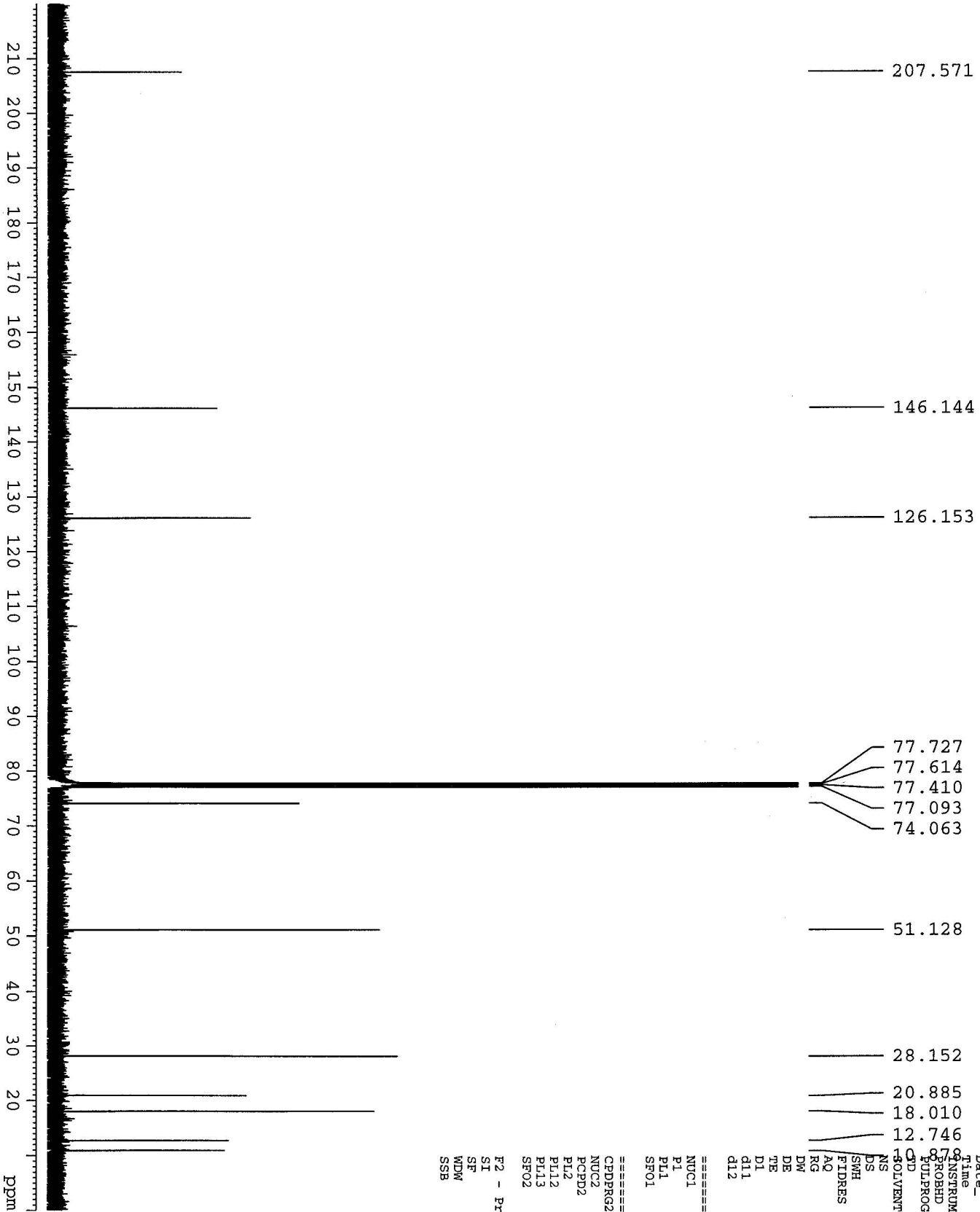
SSB 0
 LB 0.00 Hz

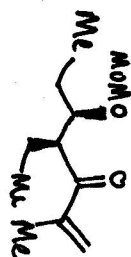
GB 0





Current Data Parameters	
NAME	NF-III-52-C13
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20040618
Time	7.06
QNTSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
CD	65336
TD	12000
NS	2
DS	2
SMH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	2580.3
DW	19.050 use
DE	6.00 use
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec
===== CHANNEL f1 =====	
NUC1	13C
PI	5.70 use
PL1	0.00 dB
SFO1	100.6237964 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 use
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.1322200 MHz
F2 - Processing parameters	
SI	32768
SF	100.6127290 MHz
WDW	no
SSB	0

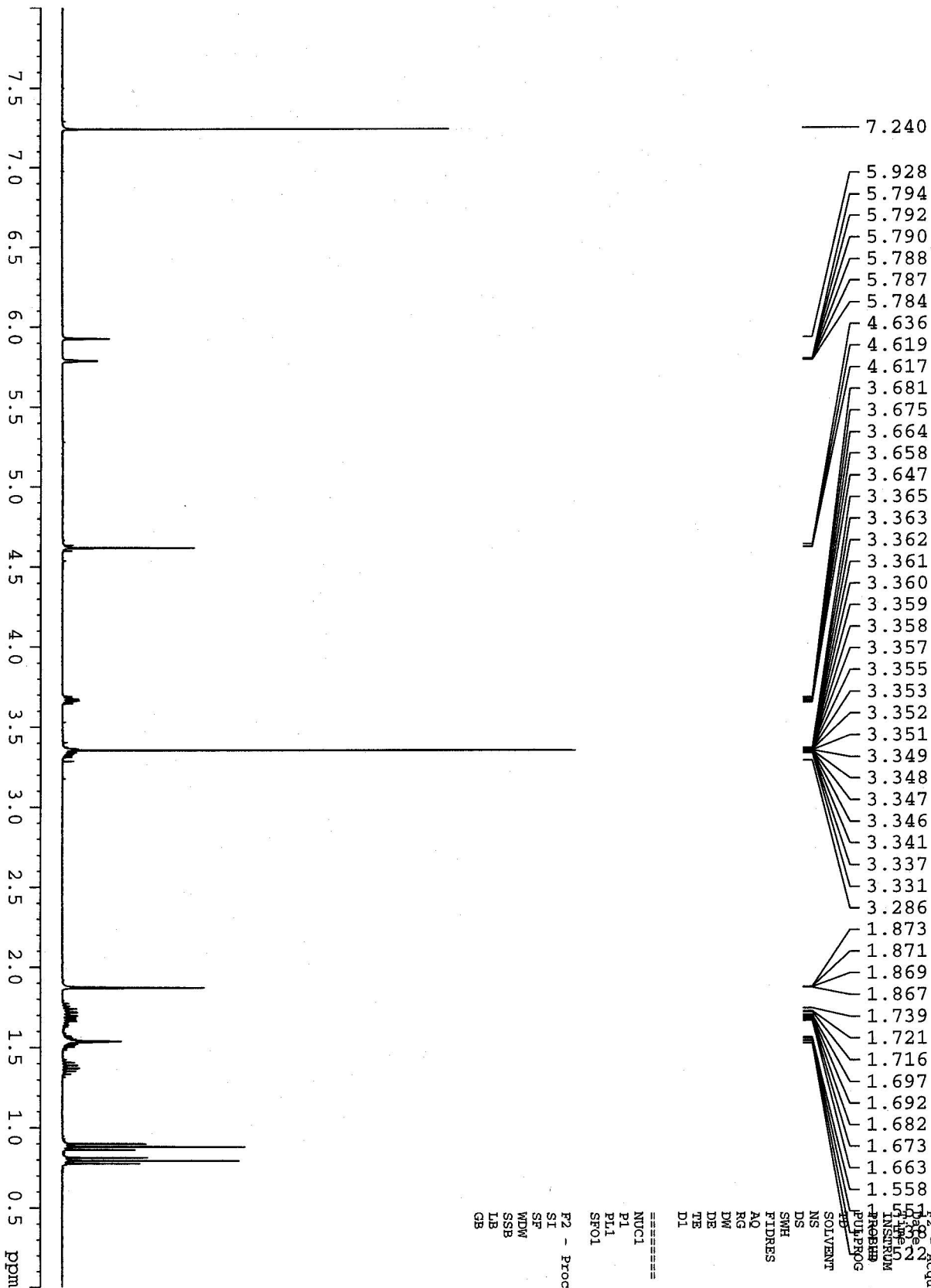


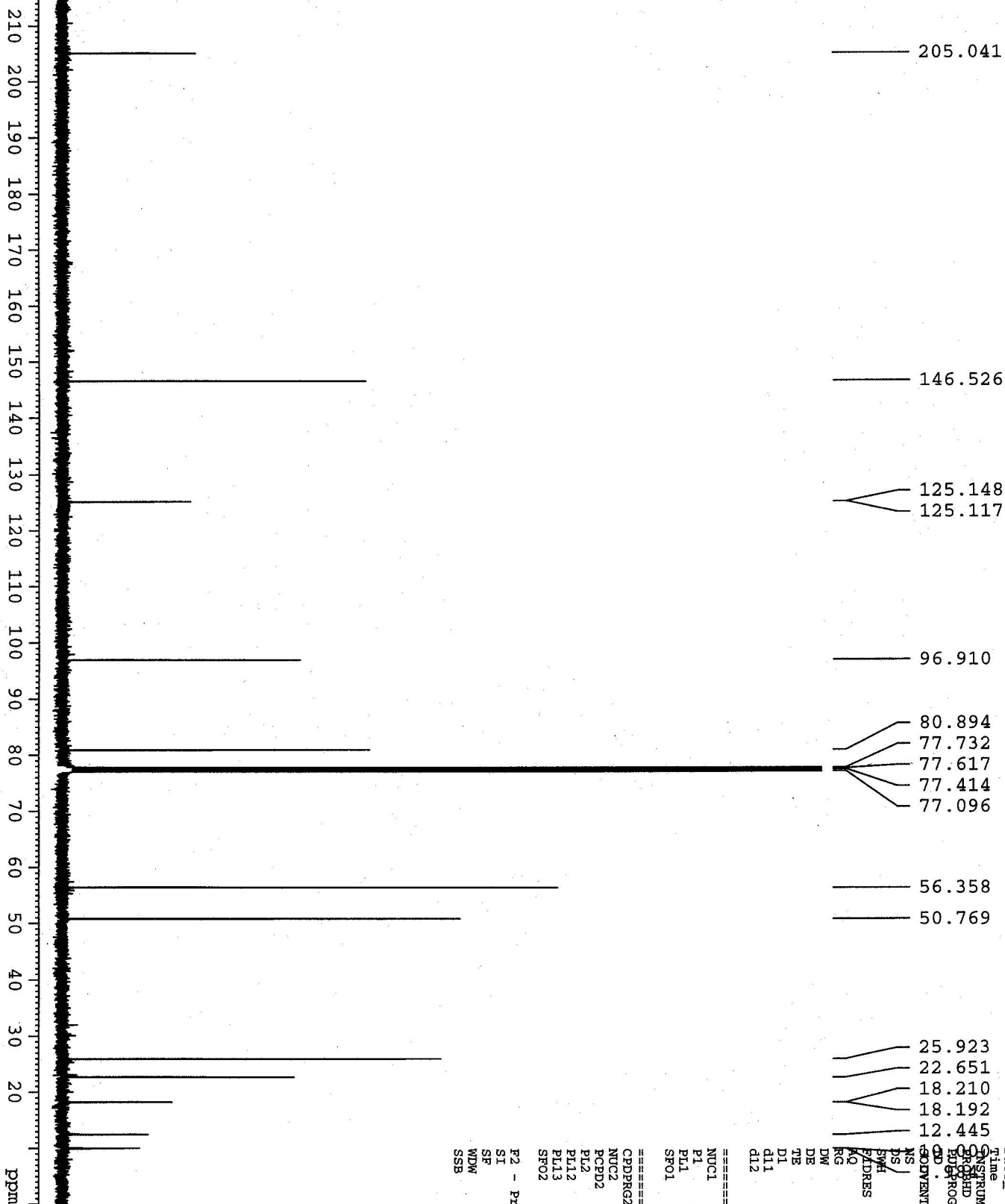
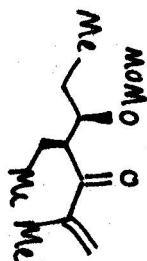


Current Data Parameters
 NAME NF-IV-166(22-34)
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050416
 Time 12.01
 INSTRUM spect
 PROBHD 5 mm HR 13C/31
 PULPROG zg30
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 161.3
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.30 usec
 PL1 -3.00 dB
 SFO1 399.8023988 MHz
 F2 - Processing parameters
 SI 32768
 SF 399.8000174 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0





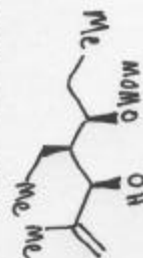
Current Data Parameters
 NAME NF-IV-16pC13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050417
 Time 7.27
 SPECTRUM spect
 PULPROG zgpg30
 FIDRES 5mm QNP 1H/13C
 AQ 1.2485298 sec
 RG 2580.3
 DW 19.050 use
 DE 5.00 use
 TE 300.0 K
 D1 0.80000001 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

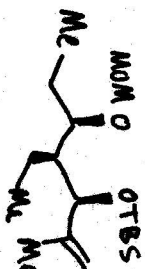
===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 use
 PL1 0.00 dB
 SFO1 100.6237964 MHz

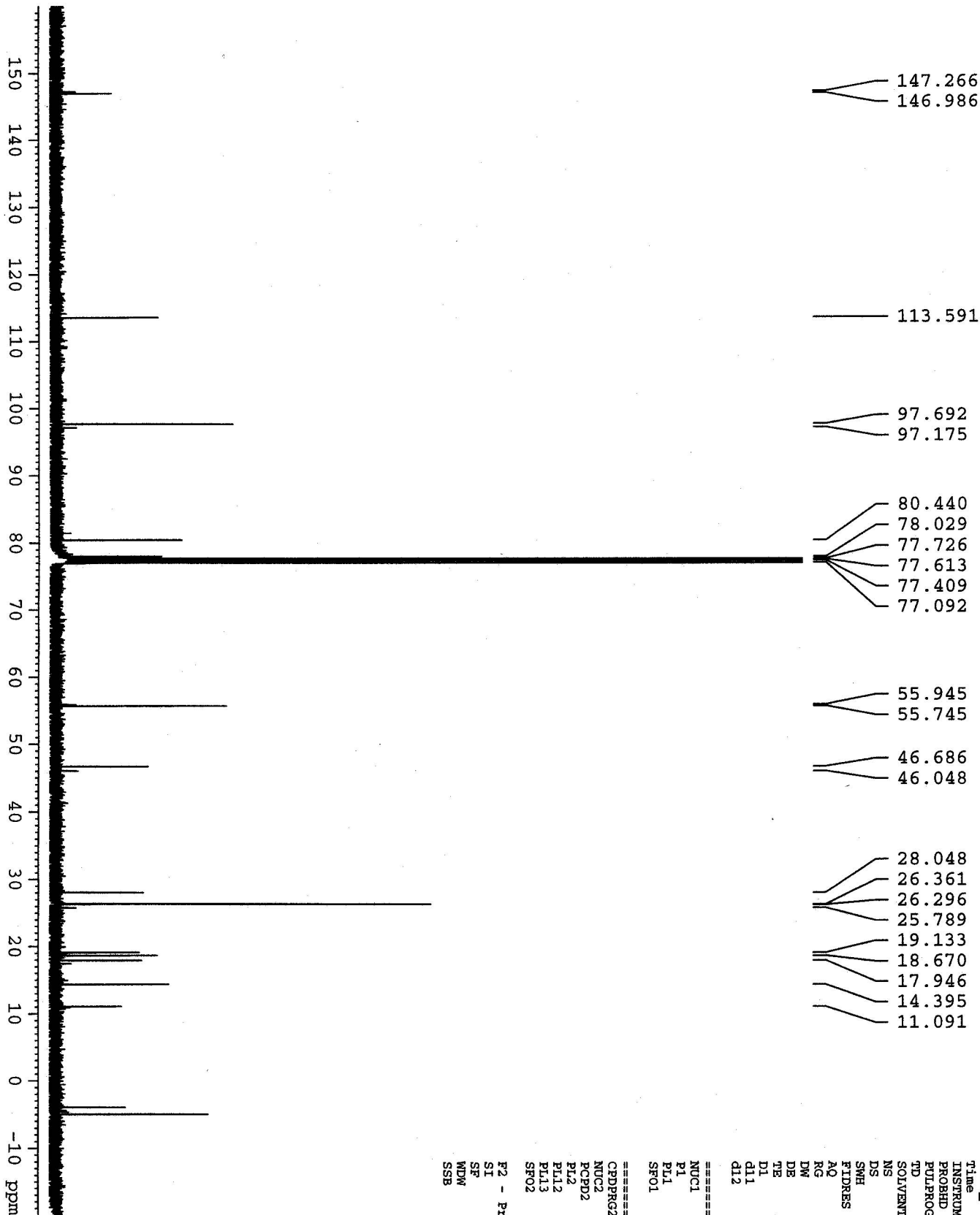
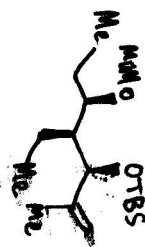
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 use
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SFO2 400.1322200 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW no
 SSB 0



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Current Data Parameters
 NAME NF-III-57-C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040617
 Time 7.05

INSTRUM spect
 PROBD 5mm QNP 1H/13C
 PULPROG zgpg30

TD 65536
 SOLVENT CDCl3
 NS 13000
 DS 2

SWH 26246.719 Hz
 FIDRES 0.400493 Hz

AQ 1.2485298 sec
 RG 912.3

DW 19.050 use
 DE 6.00 use

TE 300.0 K
 DI 0.80000001 sec

dI1 0.03000000 sec
 dI2 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C

P1 5.70 use
 PL1 0.00 dB

SFO1 100.6237964 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16

NUC2 1H

PCPD2 80.00 use

PL2 -6.00 dB

PL12 13.80 dB

PL13 14.50 dB

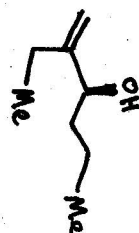
SFO2 400.1322200 MHz

F2 - Processing parameters
 SI 32768

SF 100.6127290 MHz

WDW no

SSB 0



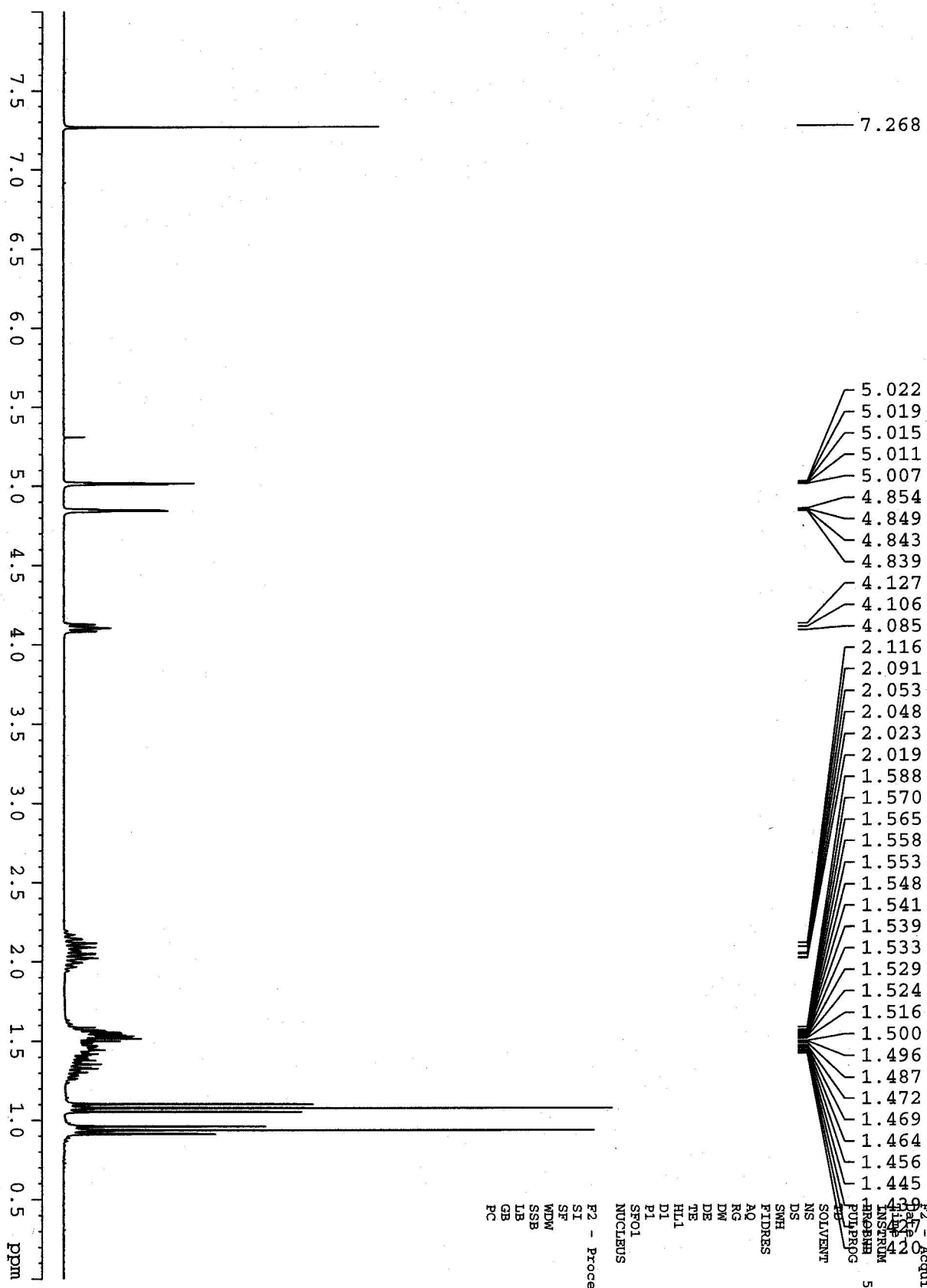
Current Data Parameters
 NAME NF-IV-44(6-11)
 EXPNO 1
 PROCNO 1

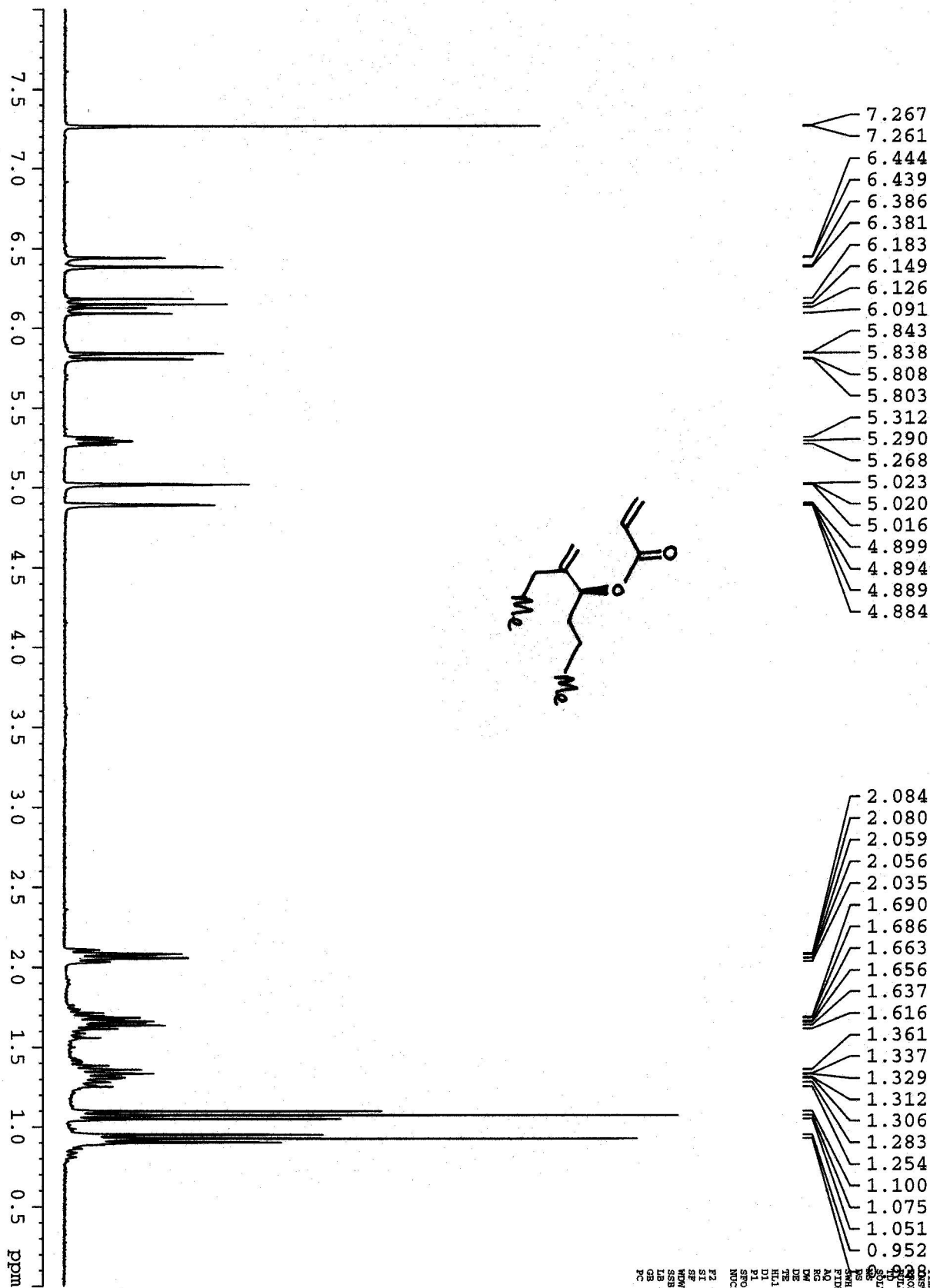
F2 - Acquisition Parameters

Date_ 20050120
 Time 11.33
 Instrument spect
 PULPROG zgpg30
 INVERT 0
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 4237.288 Hz
 FIDRES 0.129312 Hz
 AQ 3.8666739 sec
 RG 4096
 DW 118.000 usec
 DE 168.57 usec
 TE 293.0 K
 HL1 1 dB
 D1 1.0000000 sec
 P1 11.50 usec
 SFO1 300.1319000 MHz
 NUCLEUS 1H

F2 - Processing parameters

SI 16384
 SF 300.1300041 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

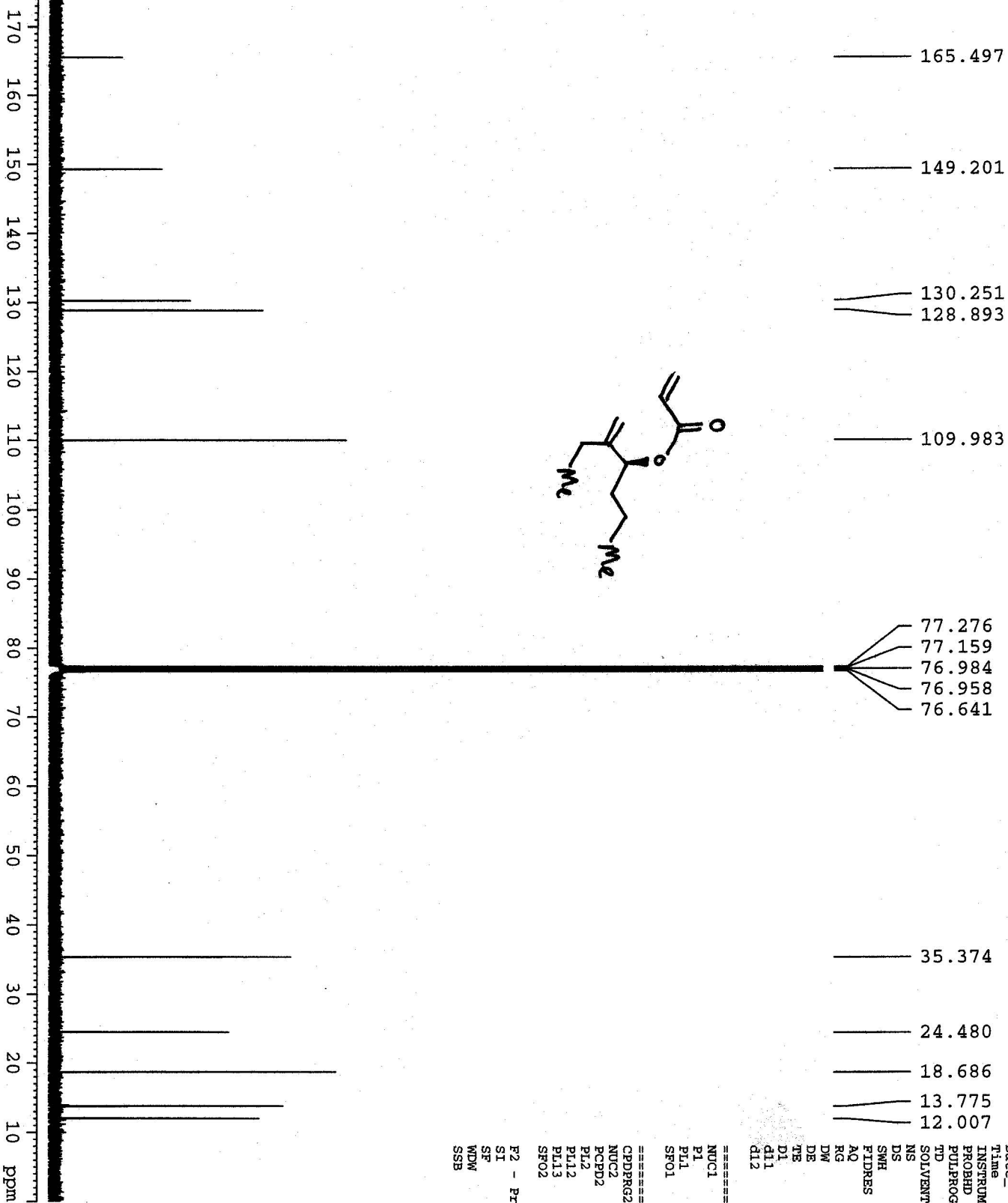
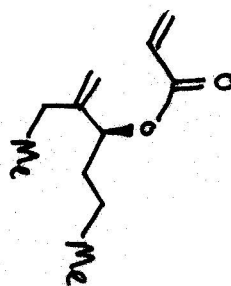




Current Data Parameters
NAME NP-111NF-III-32AA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040315
Time 16.08
INSTRUM spect
PROBHD 5 mm TBO 13C/3
PULPROG zgpg30
SOLVENT CDCl3
NS 64
DS 2
SWH 4237.268 Hz
FIDRES 0.129112 Hz
AQ 3.86672 sec
RG 320
DE 118.000 usec
TE 293.0 K
D1 1.0000000 sec
D2 0.0000000 sec
SFO1 300.131900 MHz
NUC1 1H

F2 - Processing parameters
SI 16384
SF 300.130041 MHz
WDW NO
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



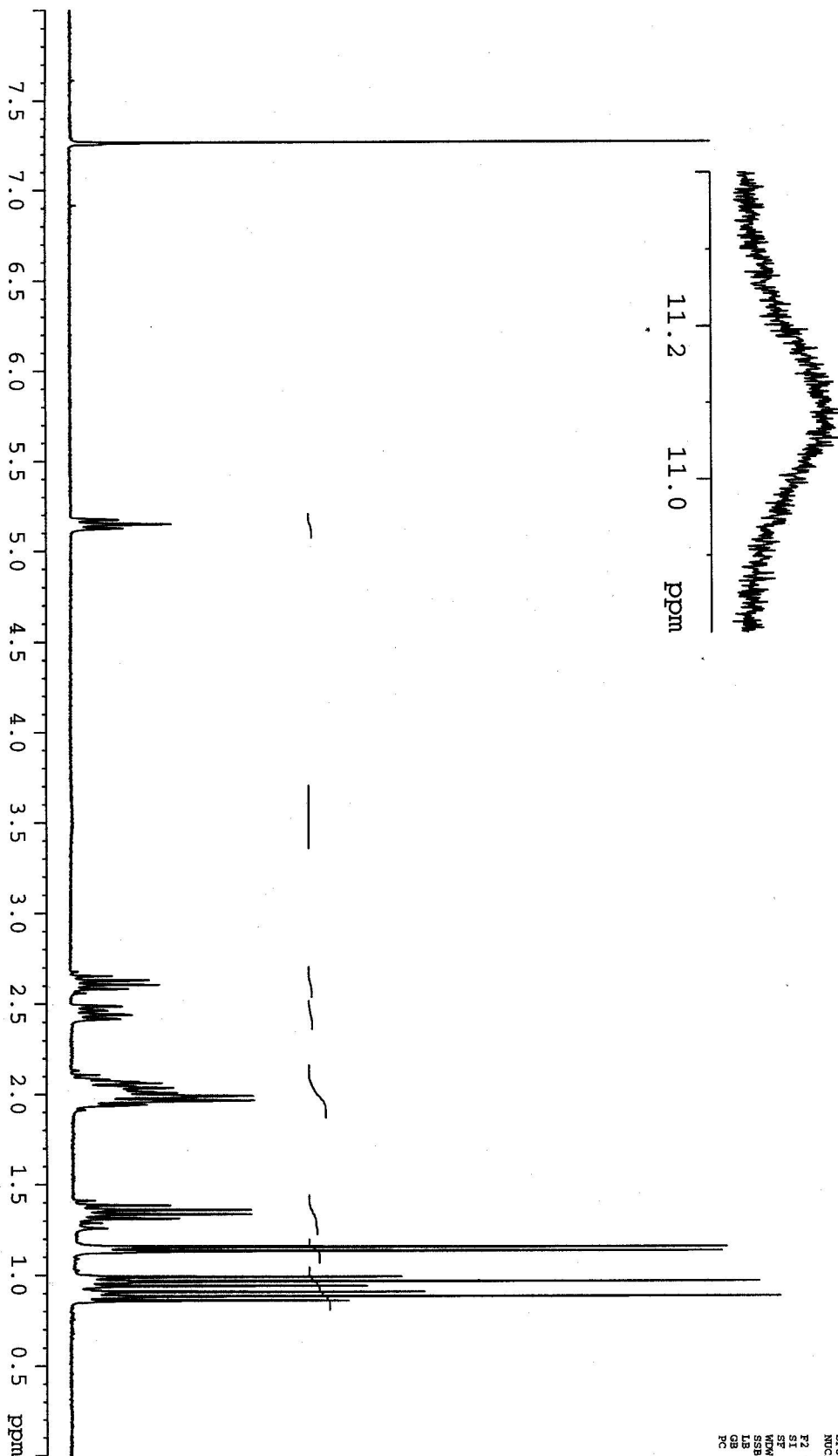
Current Data Parameters
 NAME NF-III-32PC13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040116
 Time 3.04
 INSTRUM spect
 PROBD 5 mm HR 13C/31
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 9000
 DS 2
 SMH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 16384
 DW 19.100 use
 DE 32.36 use
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

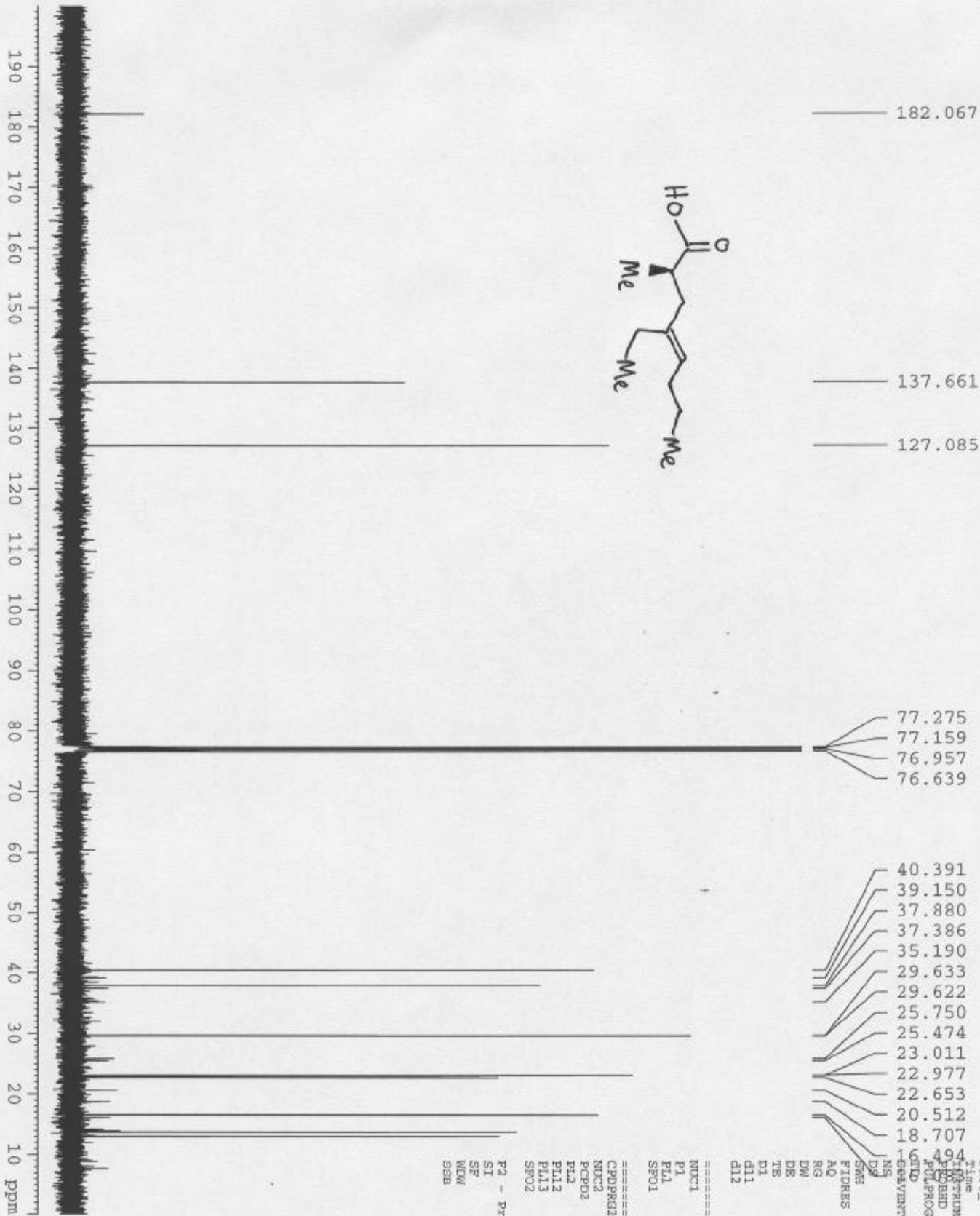
===== CHANNEL f1 =====
 NUC1 13C
 P1 7.05 use
 PL1 0.00 dB
 SFO1 100.5418136 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 use
 PL2 -3.00 dB
 PL12 18.90 dB
 PL13 22.00 dB
 SFO2 399.8015992 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5297927 MHz
 WDW no
 SSB 0



Current Data Parameters
 NAME NF11286p
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20031105
 Time 11:29
 INSTRUM spect
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 4317.388 Hz
 FWHM 0.113911 Hz
 AQ 3.866673 sec
 RG 4096
 DW 118.000 usec
 DE 168.57 usec
 TE 293.0 K
 D1 1.00000000 sec
 P1 11.50 usec
 SFO1 300.1319000 MHz
 NUC1 1H
 F2 - Processing parameters
 SI 32768
 SF 300.130041 MHz
 WTW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



Current Data Parameters

NAME NF-III-36PC13

EXPNO 1

PROCNO 1

P2 - Acquisition Parameters

Date_ 20040121

Time 6.38

INSTRUM spect

PROBHD 5 mm HR 13C/31

PULPROG zgpg30

TD 65536

RG 65536

CD 13

NS 10000

DS 2

SWH 26178.010 Hz

FIDRES 0.399445 Hz

AQ 1.2517875 sec

RG 16384

DM 19.100 use

DE 32.36 use

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

d12 0.0002000 sec

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

NUC1 13C

P1 7.05 use

PL1 0.00 dB

SFO1 100.5418136 MHz

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

CPDPRG2 waltz16

NUC2 1H

PCPD2 100.00 use

PL2 -3.00 dB

PL12 18.90 dB

PL13 22.00 dB

SFO2 399.8015992 MHz

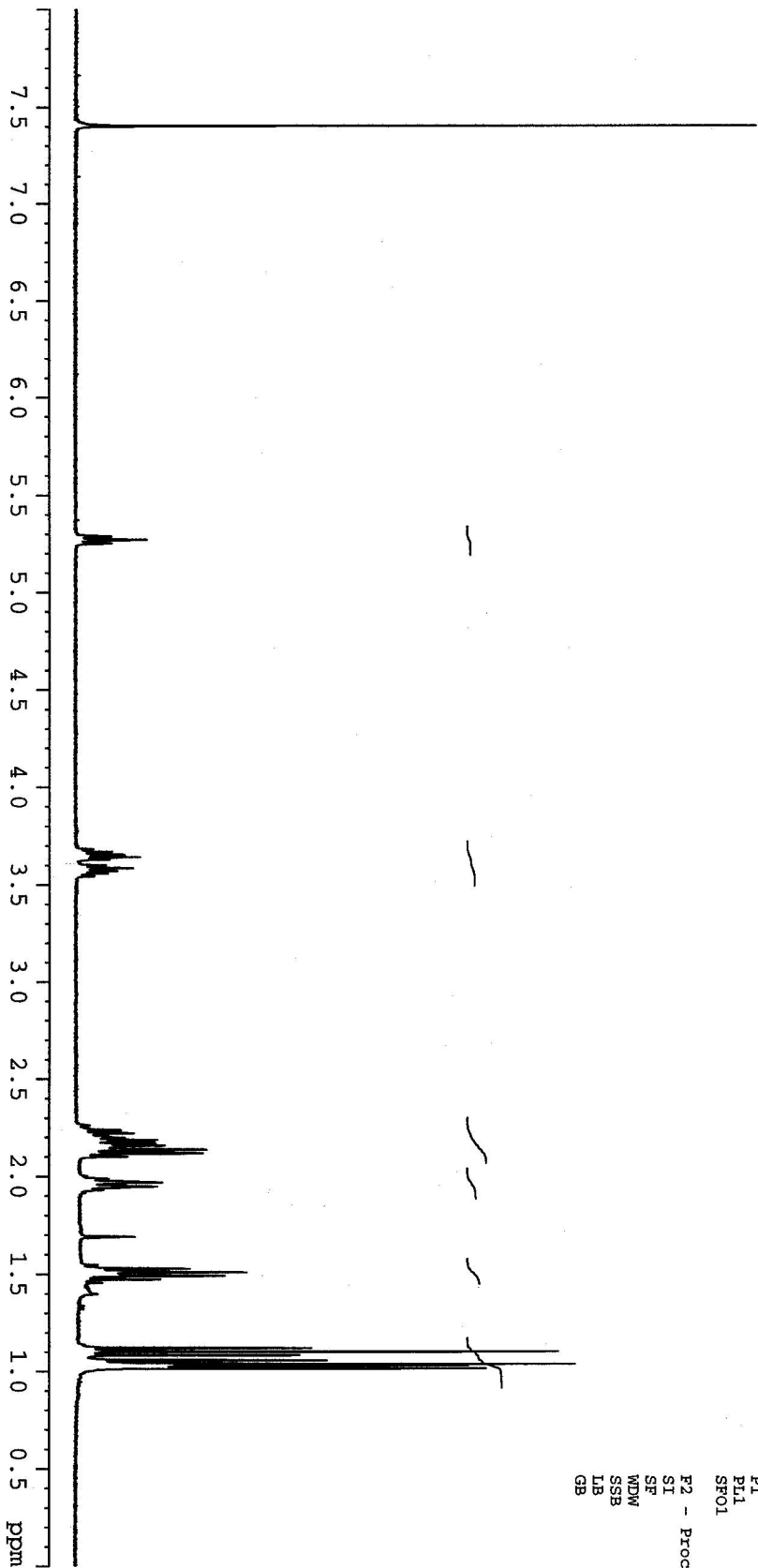
P2 - Processing Parameters

SI 65536

SF 100.5297927 MHz

WDW no

SSB 0

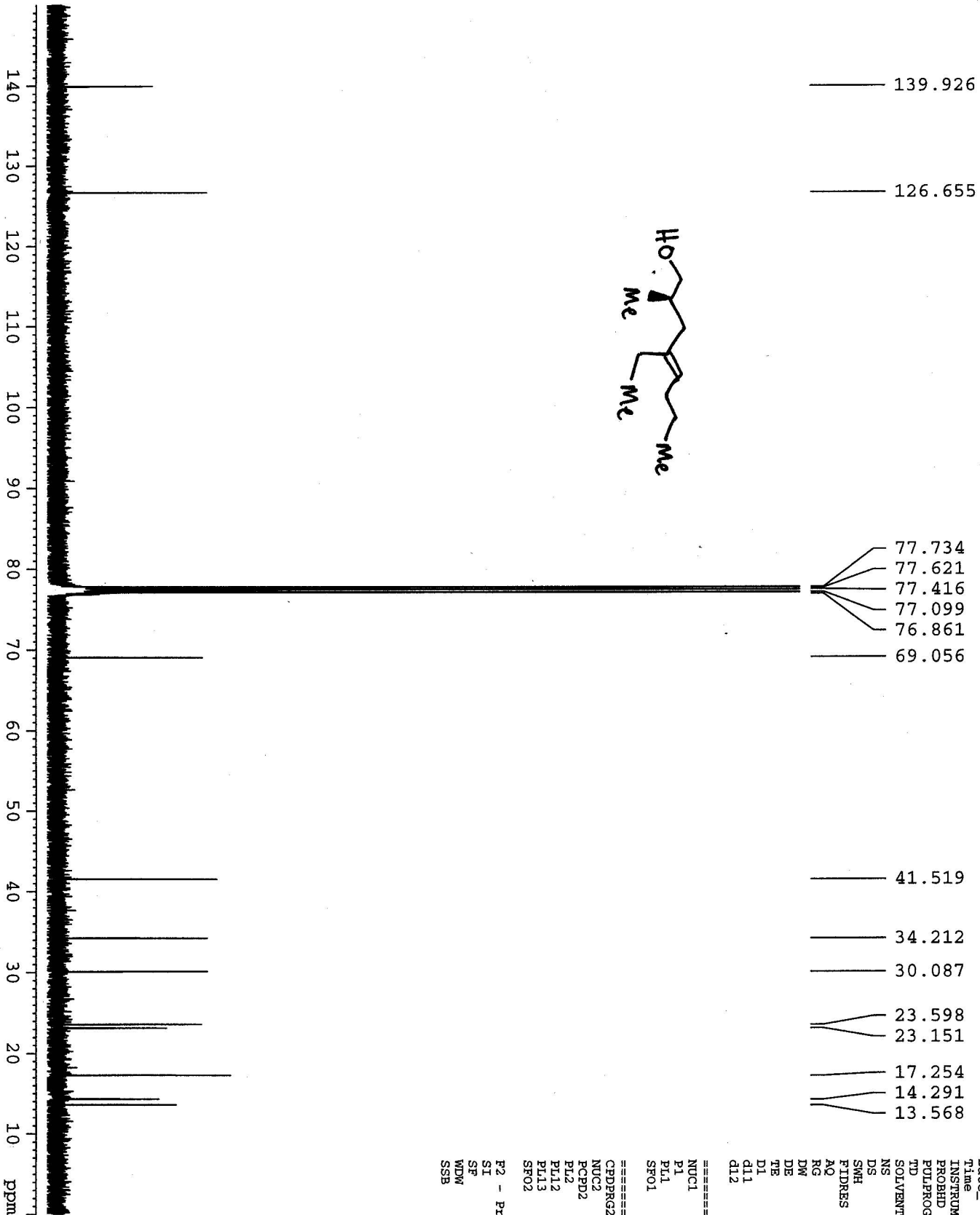


Current Data Parameters
 NAME NF-IT-62p
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040315
 Time 13.30
 INSTRUM spect
 PROBHD 5 mm HR 13C/31
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 161.3
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUCl 1H
 P1 10.10 usec
 PL -3.00 dB
 SFO1 399.802398 MHz

F2 - Processing parameters
 SI 32768
 SF 399.799519 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0



Current Data Parameters
 NAME NF-111-38_C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040121
 Time 21.11
 INSTRUM spect
 PROBD 5mm QNP 1H/13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5500
 DS 2
 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2485298 sec
 RG 8192
 DW 19.050 use
 DE 6.00 use
 TE 300.0 K
 D1 0.8000001 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 use
 PL1 0.00 dB
 SFO1 100.6237964 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 use
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SFO2 400.1322200 MHz

F2 - Processing Parameters
 SI 32768
 SF 100.6127290 MHz
 WDW no
 SSB 0

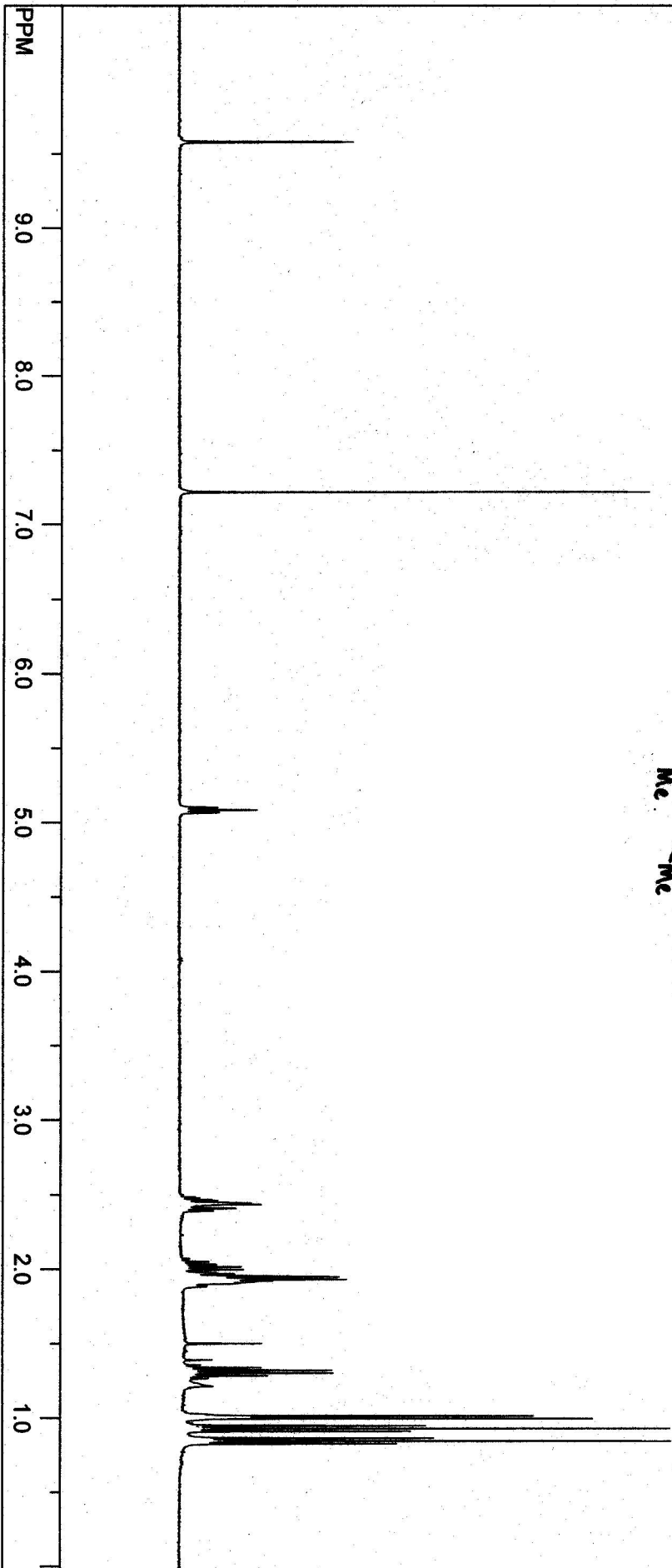
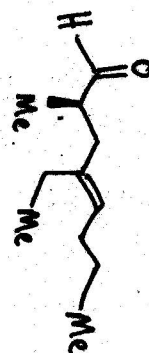
SpinWorks 2.4:

9.5846
9.5831
9.5796

7.2208
7.2194

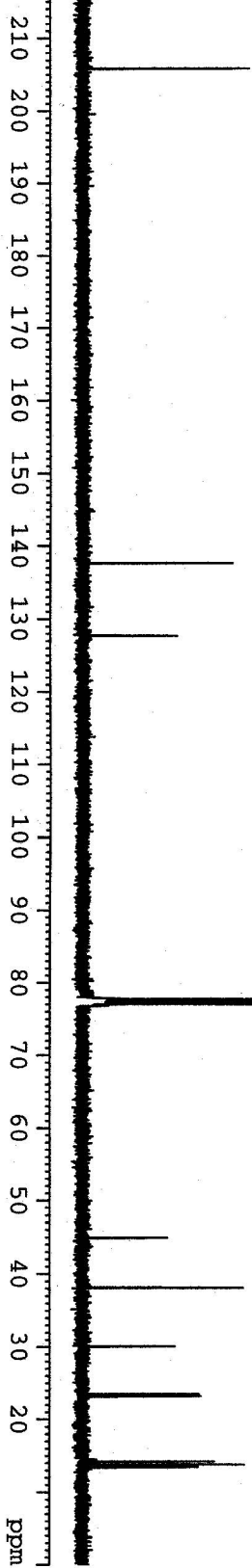
5.0878

2.4460
2.4360
2.4093
2.0186
1.9997
1.9717
1.9536
1.9356
1.9249
1.9174
1.9122
1.9101
1.9077
1.5037
1.3404
1.3219
1.3034
1.2852
1.0173
1.0003
0.9512
0.9323
0.9132
0.8668
0.8486



file: D:\dsk345\mofuller\mnmf-lll-64p\11fd exp: <zg30>
transmitter freq.: 399.802399 MHz
time domain size: 32768 points
width: 5895.20 Hz = 14.995417 ppm = 0.182959 Hz/pt
number of scans: 16

freq. of 0 ppm: 399.800025 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000
Hz/cm: 168.615 ppm/cm: 0.42175



137.665
127.746
77.732
77.616
77.414
77.096
76.840

44.914
38.095
30.081
23.470
23.201
14.228
13.782

Current Data Parameters
NAME NF-III-64PC13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040317
Time 7.22
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
CTD 65536
SOLVENT CDCl3
NS 17000
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 5160.6
DM 19.050 use
DE 6.00 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW no
SSB 0

Current Data Parameters
 NAME NF-III-47(18-23)
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

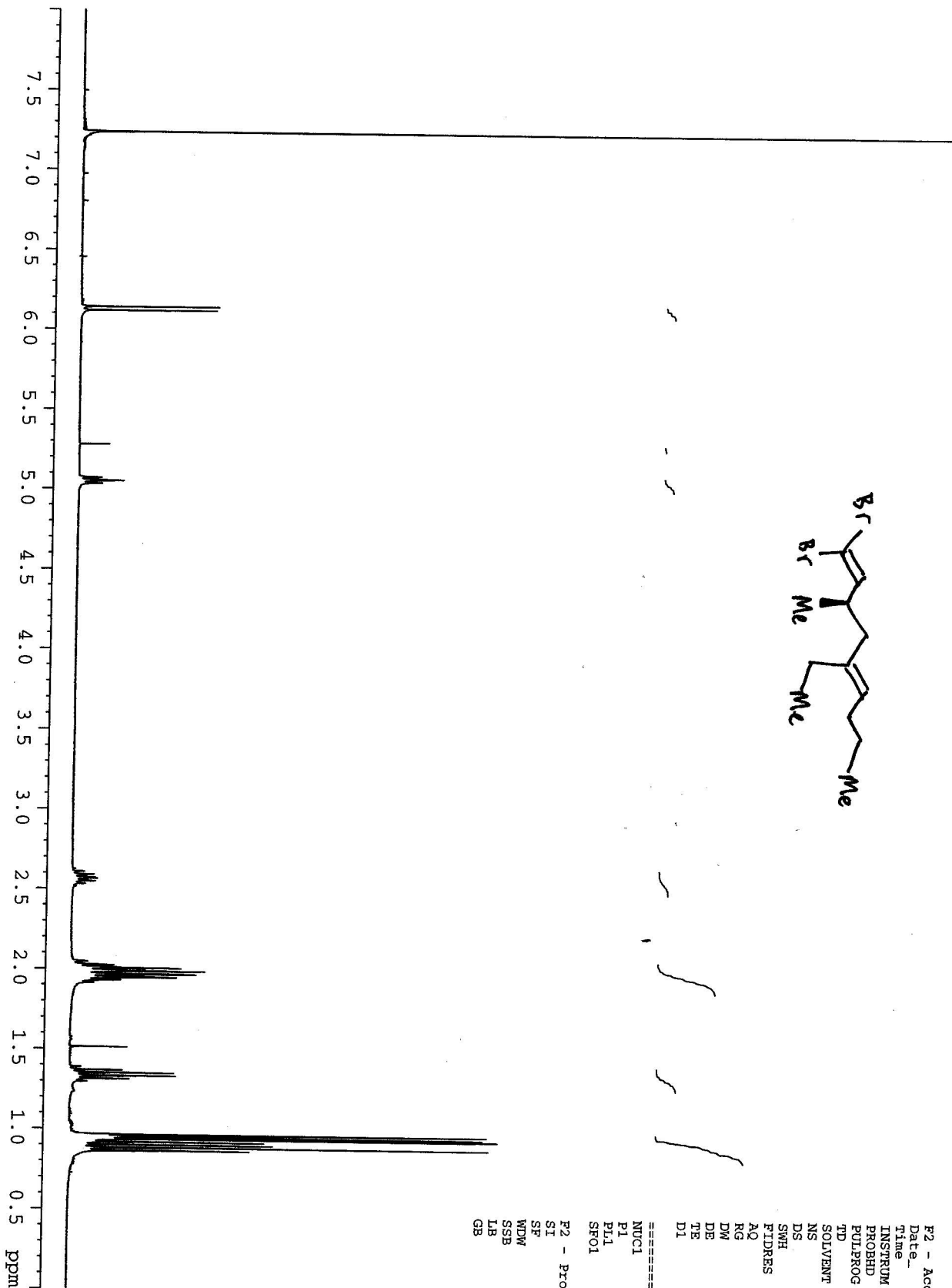
Date_ 20040204
 Time 22.16
 INSTRUM spect
 PROBHD 5mm QNP 1H/13C
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 2.5560319 sec
 RG 912.3
 DW 78.000 usec
 DE 6.00 usec
 TE 293.0 K
 D1 1.00000000 sec

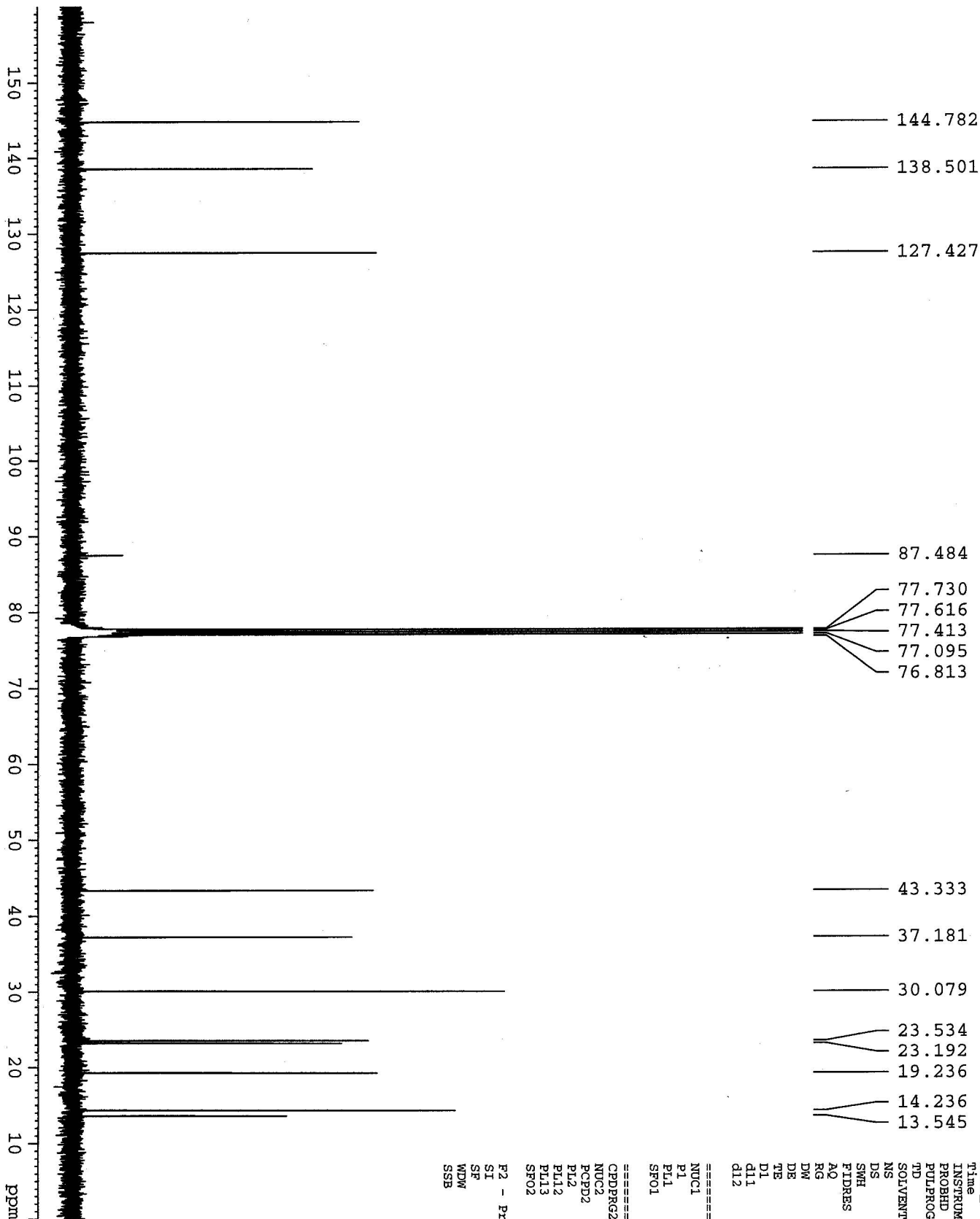
===== CHANNEL f1 =====

NUC1 1H
 P1 22.50 usec
 PL1 0.00 dB
 SFO1 400.1326008 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300172 MHz
 WDW no
 SSB 0
 LB 0
 GB 0





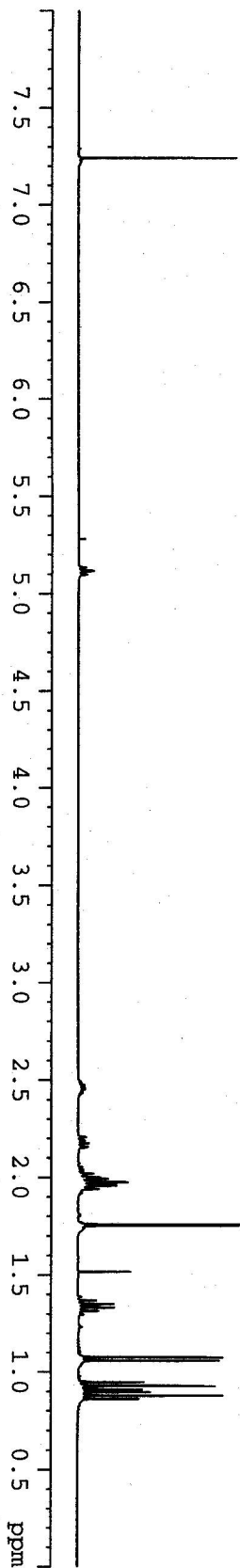
Current Data Parameters
 NAME NF-111-47PC13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040205
 Time 7.27
 INSTRUM spect
 PROBD 5mm QNP 1H/13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16000
 DS 2
 SWH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2485298 sec
 RG 9195.2
 DW 19.050 use
 DE 6.00 use
 TE 300.0 K
 D1 0.80000001 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 use
 PL1 0.00 dB
 SFO1 100.6237964 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 use
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SFO2 400.1322200 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 WDW no
 SSB 0



7.240
7.237

5.135
5.117
5.100

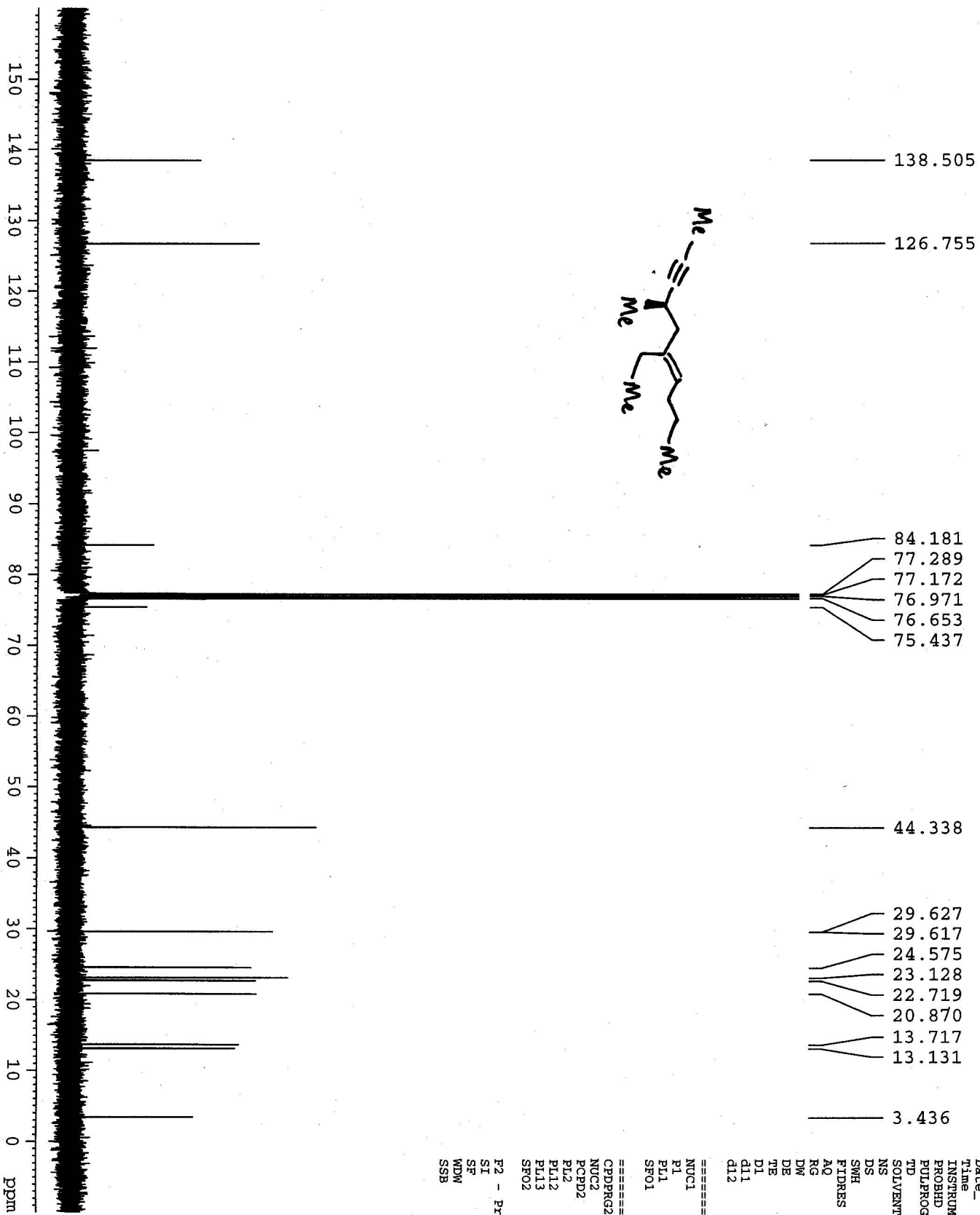
2.458
2.452
2.209
2.207
2.189
2.175
2.173
2.157
2.155
2.152
2.019
2.011
2.000
1.994
1.976
1.958
1.940
1.759
1.753
1.748
1.747
1.518
1.370
1.352
1.333

Current Data Parameters
NAME NF-III-222ck
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050524
Time 22.27
INSTRUM spect
PROBHD 5 mm HR 13C/31
PULPROG zg30
SOLVENT CDCl3
DS 48
NS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 161.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.30 usec
PL1 -3.00 dB
SFO1 399.8023968 MHz

F2 - Processing parameters
SI 32768
SF 399.8000176 MHz
WDW ho
SSB 0
LB 0.00 Hz
GB 0



Current Data Parameters
 NAME NF-III-222-C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050525
 Time 2.55
 INSTRUM spect
 PROBD 5 mm HR 13C/31
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6531
 DS 2
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 18390.4
 DW 19.100 use
 DE 32.36 use
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 d12 0.0002000 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 100.5297927 MHz
 WDW no
 SSB 0

Current Data Parameters
 NAME NF-117-159(6-8)
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040623
 Time 21.21

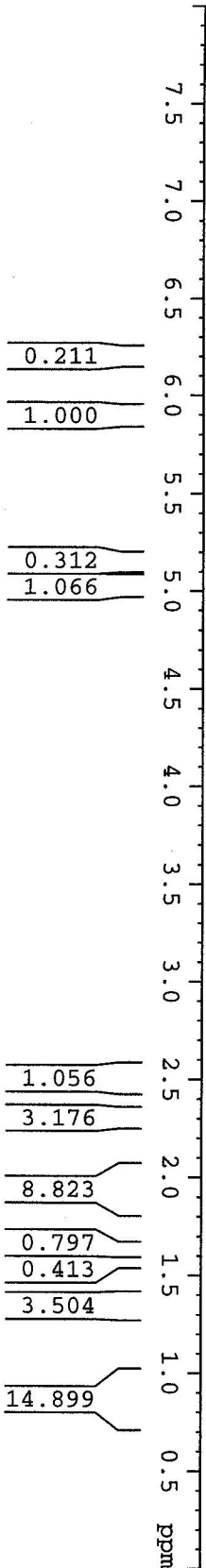
INSTRUM spect
 PROBHD 5mm QNP 1H/13C
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2

SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 2.5560319 sec

RG 1024
 DW 78.000 usec
 DE 6.00 usec
 TE 293.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 22.50 usec
 PL1 0.00 dB
 SFO1 400.1326008 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300172 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0



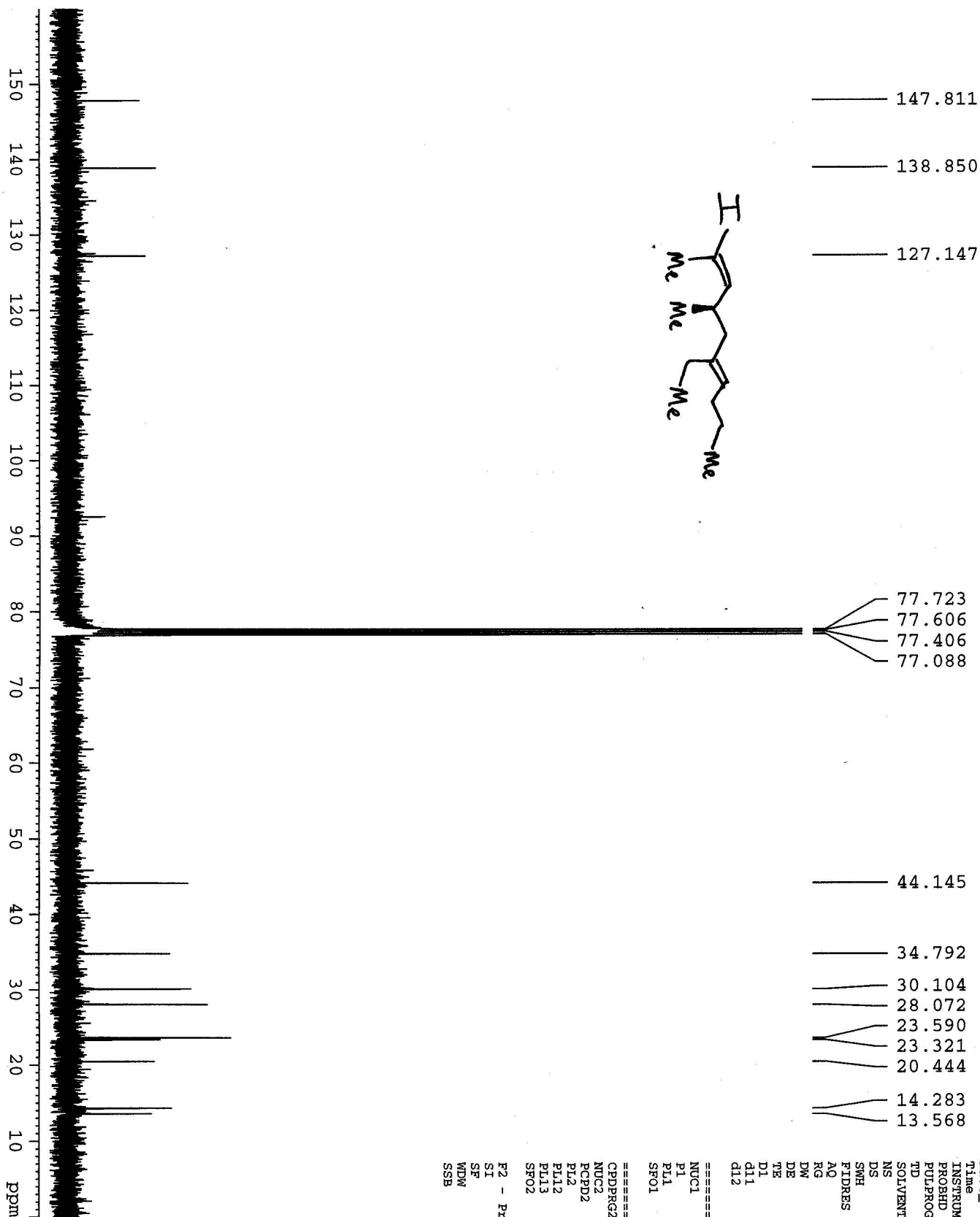
Current Data Parameters
NAME NF-111-159C13
EXPNO 1
PROCNO 1

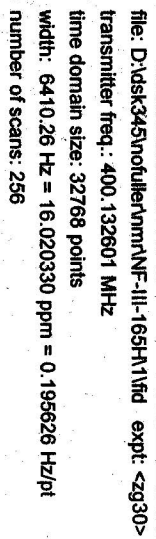
F2 - Acquisition Parameters
Date_ 20040624
Time 5.35
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 14000
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 1824.6
DE 19.050 use
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 use
PL1 0.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW no
SSB 0





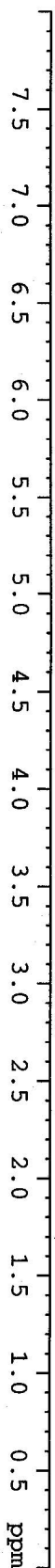
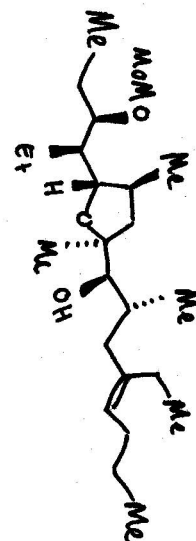
Current Data Parameters
 NAME NF-III-191(115-136)
 EXPNO 1
 PROCNO 1

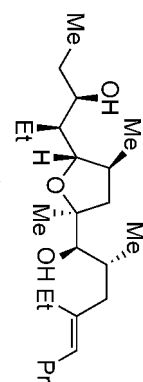
F2 - Acquisition Parameters

Date_ 20040730
 Time 7.36
 INSTRUM spect
 PROBD 5mm QNP 1H/13C
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 2.5560319 sec
 RG 128
 DW 78.000 usec
 DE 6.00 usec
 TE 293.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 22.50 usec
 PL1 0.00 dB
 SFO1 400.1326008 MHz

F2 - Processing Parameters
 SI 32768
 SF 400.1300172 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0





Current Data Parameters
 NAME NF-IV-271p
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050824
 Time 2.10

INSTRUM spect
 PROBD 3mm Micro In
 PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 128

DS 2

SWH 8012.820 Hz

FIDRES 0.244532 Hz

AO 2.0447731 sec

RG 362

DW 62.400 usec

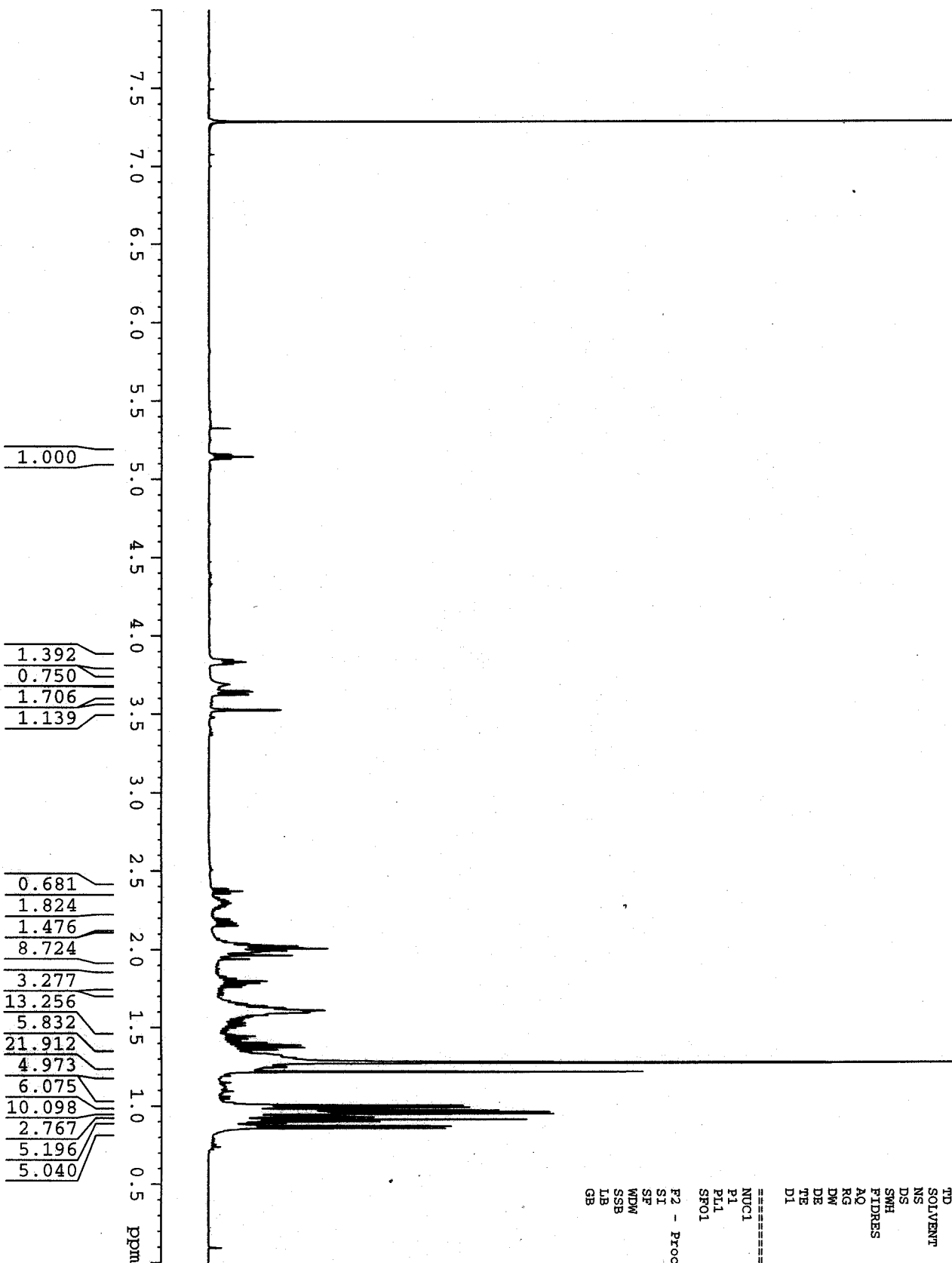
DE 6.00 usec

TE 293.0 K

D1 1.00000000 sec

===== CHANNEL f1 =====
 NUCL1 1H
 P1 6.10 usec
 PL1 0.00 dB
 SFO1 500.1333807 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0



Current Data Parameters
 NAME nf-111-75C13
 EXPNO 2
 PROCNO 1

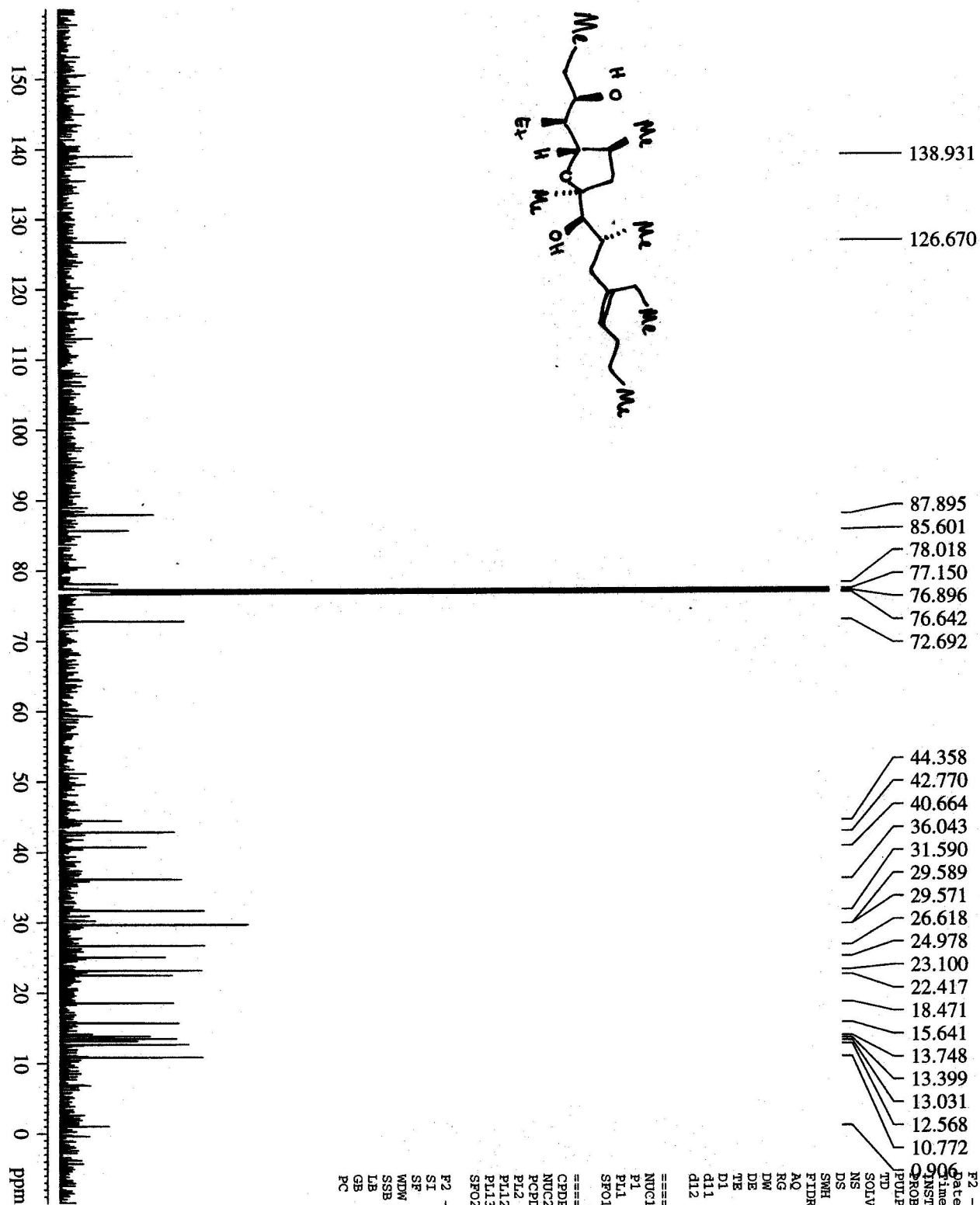
F2 - Acquisition Parameters

Date_ 20050615
 Time 21.21
 INSTRUM spect
 PROBHD 3mm Micro Du
 PULPROG zgpg30
 TD 65536
 CDC13
 SOLVENT 17372
 NS 2
 DS 2
 SWH 30581.039 Hz
 FIDRES 0.466630 Hz
 AQ 1.0715636 sec
 RG 8192
 DW 16.350 use
 DE 6.00 use
 TE 293.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

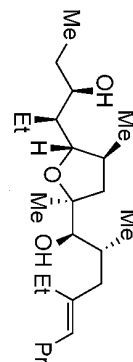
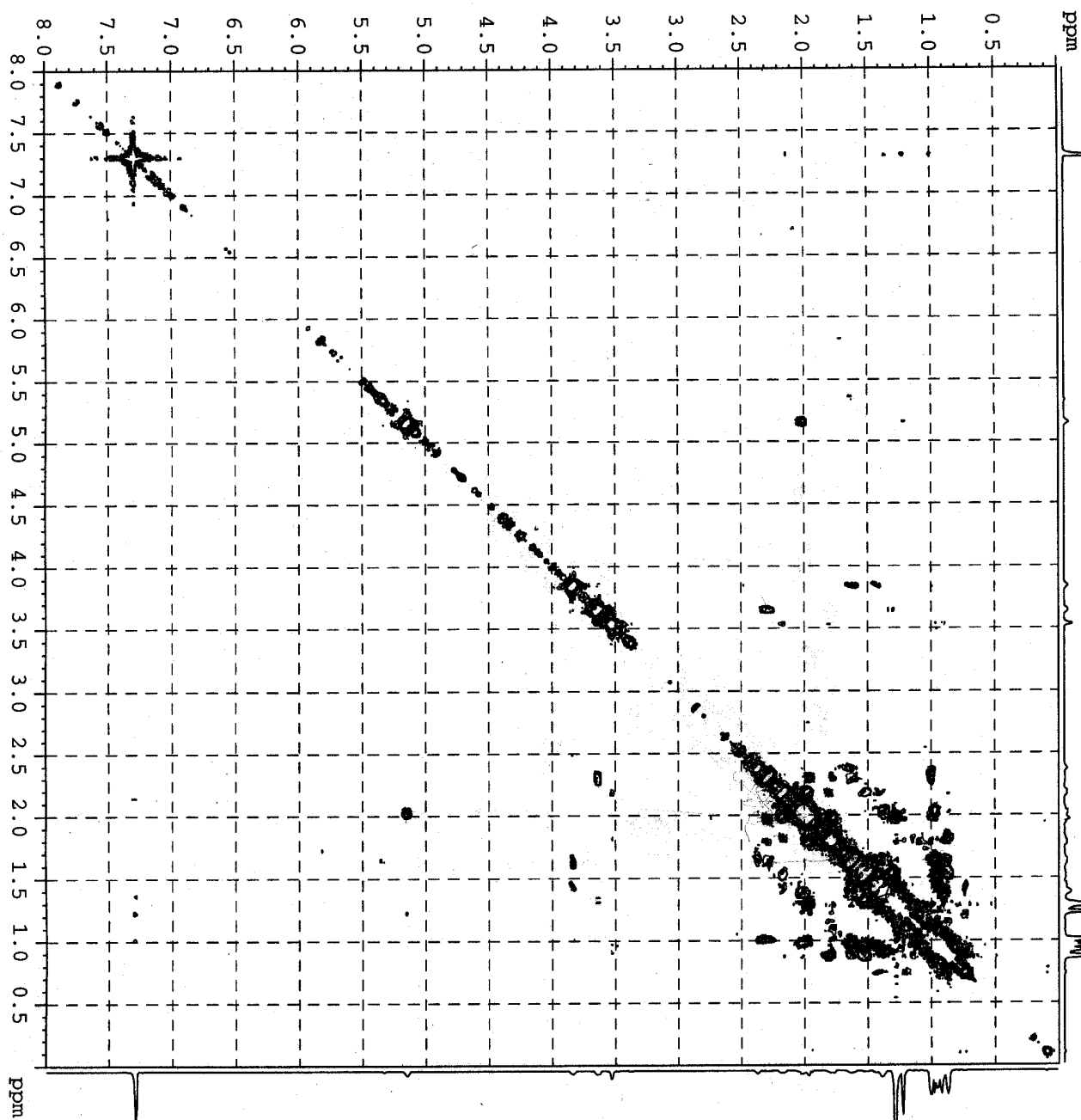
===== CHANNEL f1 ===
 NUC1 13C
 P1 6.80 use
 PL1 0.00 dB
 SFO1 125.7705006 MHz

===== CHANNEL f2 ===
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 89.00 use
 PL2 -3.00 dB
 PL12 14.00 dB
 PL13 14.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 65536
 SF 125.7578051 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.40



g-COSY



COSY

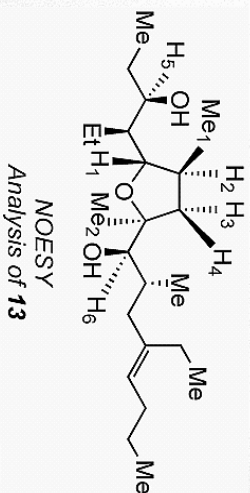
Current Data Parameters
 NAME: ME-1V-2711COSY
 EXPRNO: 2
 PROCNO: 1

F2 - Acquisition Parameters
 Date_ : 20080414
 Time : 9.41
 INSTRUM : spect
 PULPROG : zgpg30
 PROBHD : 3mm Micro In
 PULPROG : zgpg30
 F2 - Processing parameters
 SI : 1024
 SF : 500.130000 MHz
 WDW : SINE
 GB : 0.00 Hz
 PC : 1.40

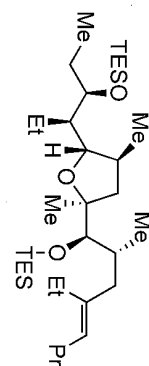
F1 - Processing parameters
 SI : 1024
 SF : 500.130000 MHz
 WDW : SINE
 GB : 0.00 Hz
 PC : 1.40

===== CHANNEL f1 =====
 NUC1 : 1H
 P1 : 8.35 usec
 PL1 : -1.00 dB
 SFO1 : 500.132506 MHz
 GRADIENT CHANNEL
 P16 : 1000.00 usec

F1 - Acquisition parameters
 NUC1 : 1H
 TD : 229
 SFO1 : 500.1323 MHz
 FIDRES : 21.834061 Hz
 SW : 9.997 ppm



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Current Data Parameters
 NAME NP-3-212-4
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20050706
 Time 13.56
 INSTRUM spect
 PROBHD 3mm Micro In
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 362
 DW 62.400 usec
 DE 6.00 usec
 TE 293.0 K
 D1 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 6.10 usec
 PL1 0.00 dB
 SFO1 500.1333807 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0

