

**Highly Selective Coupling of Alkenes and Aldehydes Catalyzed by
NHC–Ni–P(OPh)₃: Synergy Between a Strong σ -Donor and a Strong π -Acceptor**

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Supporting Information

Experimental Procedures, Analytical and Spectroscopic Data for Compounds

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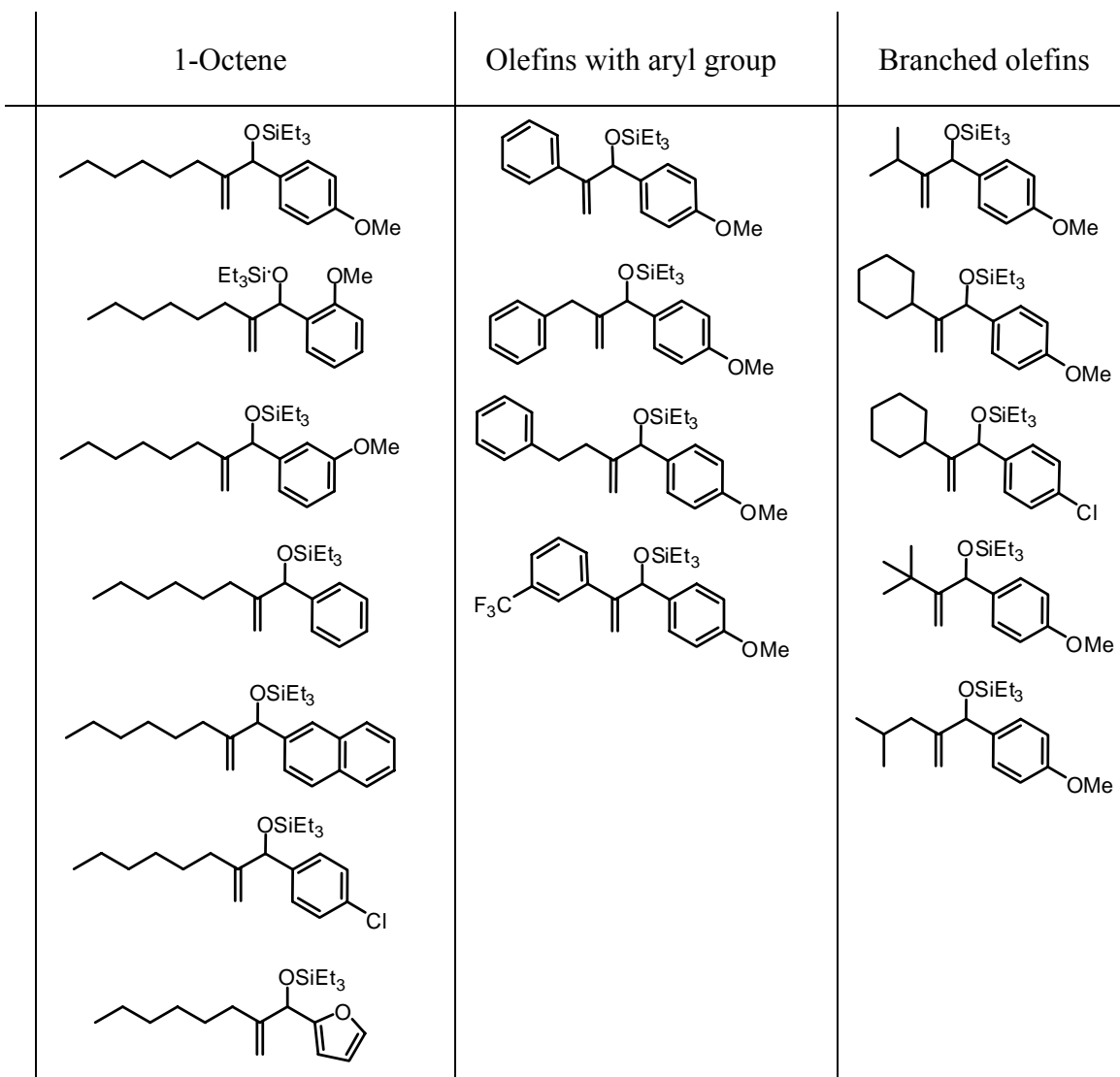
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General Experimental Procedures.

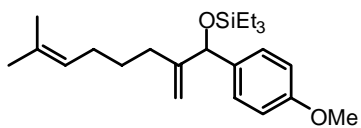
Unless otherwise indicated, all reactions were performed under an oxygen-free atmosphere of argon with rigid exclusion of moisture from reagents and glassware. All alkenes and aldehydes were used as received without further purification. Diphenylcyclohexylphosphine, ethyl diphenylphosphinite and diethyl phenylphosphonite were purchased from Aldrich Chemical Co. and were used as received. Other phosphorous ligands, 1,3-Bis(2,6-di-isopropylphenyl)imidazol-2-ylidene (IPr) and Bis(cyclooctadienyl) nickel(0) ($\text{Ni}(\text{cod})_2$) were purchased from Strem Chemicals, Inc., stored under nitrogen atmosphere and used without further purification. Triethylamine, toluene and Triethylsilyl trifluoromethanesulfonate (TESOTf) were distilled over calcium hydride before use.

Analytical thin layer chromatography (TLC) was performed using EM Science silica gel 60 F254 plates. The developed chromatogram was analyzed by UV lamp (254 nm), ethanolic phosphomolybdic acid (PMA) or potassium permanganate (KMnO_4). Liquid chromatography was performed using a forced flow (flash chromatography) of the indicated solvent system on Silicycle Silica Gel (230–400 mesh). ^1H and ^{13}C NMR spectra were recorded on Bruker 400 MHz spectrometers in CDCl_3 . Chemical shifts in ^1H NMR spectra are reported in ppm on the δ scale from residual chloroform (7.27 ppm). Chemical shifts of ^{13}C NMR spectra are reported in ppm from the central peak of CDCl_3 (77.23 ppm) on the δ scale. Infrared (IR) spectra were recorded on a Perkin–Elmer 2000 FT–IR. High resolution mass spectra (HRMS) were obtained on a Bruker Daltonics APEXII 3 Tesla Fourier Transform Mass Spectrometer by Dr. Li Li of the Massachusetts Institute of Technology, Department of Chemistry Instrument Facility.

Figure 1. Structures of the Coupling Products



With an additional double bond



Procedure for the Evaluation of Additives in Ni-IPr Mediated Alkene-Aldehyde Coupling Reactions (Table 1).

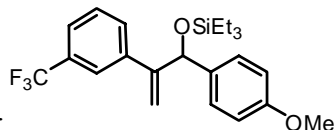
1,3-Bis(2,6-di-isopropylphenyl)imidazol-2-ylidene (IPr) (0.05 mmol, 20 mol%) and Ni(cod)₂ (0.05 mmol, 20 mol%) were added to an oven-dried test tube equipped with a stir bar in glove box. The tube was sealed with a septum, brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in degassed toluene (3.0 mL) under argon and stirred 1 h at room temperature. Vinylcyclohexene (0.25 mL), triethylamine (1.5 mmol, 600 mol%), *p*-anisaldehyde (0.25 mmol, 100 mol%), and the additive (0.05 mmol, 20 mol%) were added sequentially to the reaction mixture. Triethylsilyltriflate (0.44 mmol, 175 mol%) was added to the reaction mixture dropwise and the mixture was stirred 21 h at room temperature. The mixture was diluted with ether (5 mL) and was allowed to stir 30 mins in open air. The mixture was filtered through a short plug of silica gel and rinsed with 20% ethyl acetate/hexane (50 mL). The solvent was removed under reduced pressure, purification via flash chromatography on silica gel (1% ethyl acetate in hexane, unless otherwise indicated) afforded the coupling product.

General Procedure for IPr-Ni-P(OPh)₃ Catalyzed Alkene-Aldehyde Coupling Reactions (Table 2).

1,3-Bis(2,6-di-isopropylphenyl)imidazol-2-ylidene (IPr) (0.075 mmol, 30 mol%) and Ni(cod)₂ (0.075 mmol, 30 mol%) were added to an oven-dried test tube equipped with a stir bar in glove box. The tube was sealed with a septum, brought out of the glove box, and connected to an argon line. The catalyst mixture was dissolved in degassed toluene (3 mL) under argon and stirred at room temperature for 1 hour. The alkene (1.25 mmol, 500 mol% or indicated amount in Table 2), triethylamine (1.5 mmol, 600 mol%), aldehyde (0.25 mmol, 100 mol%), triphenylphosphite (0.11 mmol, 45 mol%) were then added sequentially to the reaction mixture. (When a solid aldehyde was used, the aldehyde was added as a stock solution in toluene.) Triethylsilyltriflate (0.44 mmol, 175 mol%) was added to the reaction mixture dropwise, and the mixture was stirred 48 h at 35 °C. After cooling to room temperature, the mixture was diluted with ether (5 mL) and was allowed to stir 30 mins in open air. The mixture was then filtered through a short plug of silica gel and rinsed with 20% ethyl acetate/hexane (50 mL). The solvent was removed under reduced pressure, and purification via flash chromatography on silica gel (1% ethyl acetate in hexane, unless otherwise indicated) afforded the coupling product.

In some cases, the coupling product coeluted with the starting materials, and characterization data of the corresponding alcohol was reported instead. The corresponding alcohol product was obtained by treating the mixture with 1 M TBAF in THF at 0 °C and allowed to stir 2 h at room temperature, followed by flash column chromatography on silica (15% ethyl acetate in hexane, or 80% DCM in hexane).

Compound Characterization Data



Side Product from *m*-CF₃ styrene as additive:

Table 1, entry 2. Yield: < 5%.

Characterization data of the corresponding alcohol:

¹H NMR (400 MHz, CDCl₃, δ): 7.59 (s, 1H), 7.50–7.46 (m, 2H), 7.39–7.36 (m, 1H), 7.31 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.68 (d, *J* = 3.7 Hz, 1H), 5.63 (t, *J* = 1.1 Hz, 1H), 5.67 (s, 1H), 3.81 (s, 3H), 2.07 (d, *J* = 3.7 Hz, 1H).

¹³C NMR (100 MHz, CDCl₃, δ): 159.6, 149.5, 140.4, 133.6, 130.6, 130.5, 128.9, 128.5, 124.5, 124.0, 115.3, 114.2, 75.6, 55.5.

IR (NaCl, thin film): 3398, 2925, 2852, 1669, 1611, 1587, 1513, 1490, 1465, 1442, 1332, 1251, 1167, 1125, 1074, 1034, 905, 832, 807, 701, 666.

HRMS–ESI (*m/z*): [M+Na]⁺ calcd for C₁₇H₁₅F₃O₂Na, 331.0916; found, 331.0924.

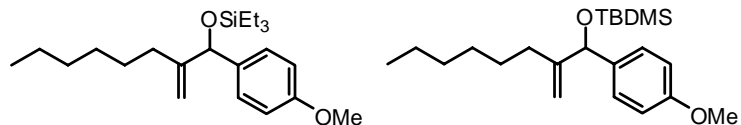


Table 2, entry 1 and 2:

The standard procedure was followed, except only 1.5 equiv of alkene was used. Yield: 91%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.28 (d, $J = 8.7$ Hz, 2H); 6.89 (d, $J = 8.7$ Hz, 2H); 5.27 (s, 1H); 5.09 (s, 1H); 4.97 (s, 1H); 3.81 (s, 3H); 2.06 (brs, 1H); 1.97–1.75 (m, 2H); 1.48–1.29 (m, 2H); 1.28–1.23 (m, 6H); 0.87 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.3, 151.5, 134.6, 128.2, 113.9, 109.2, 76.9, 55.4, 32.2, 31.9, 29.3, 27.9, 22.8, 14.3.

IR (NaCl, thin film): 3405, 2955, 2928, 2857, 1647, 1611, 1586, 1511, 1465, 1303, 1248, 1172, 1109, 1037, 903, 830, 779.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{24}\text{O}_2\text{Na}$, 271.1669; found, 271.1679.

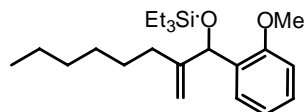


Table 2, entry 3:

The standard procedure was followed. Yield: 60%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.31–7.26 (m, 2H), 6.98 (dt, $J = 7.5, 1.0$ Hz, 1H); 6.92 (d, $J = 8.1$ Hz, 2H); 5.44 (d, $J = 5.9$ Hz, 1H); 5.16 (s, 1H); 4.99 (t, $J = 1.3$ Hz, 1H); 3.87 (s, 3H); 2.63 (d, $J = 6.0$ Hz, 1H); 2.20–1.90 (m, 2H); 1.50–1.42 (m, 2H); 1.32–1.24 (m, 6H); 0.89 (t, $J = 7.0$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 157.3, 150.9, 130.6, 128.9, 128.2, 121.0, 110.9, 109.5, 72.4, 55.6, 33.1, 31.9, 29.3, 28.1, 22.8, 14.3.

IR (NaCl, thin film): 3365, 2928, 2857, 1653, 1596, 1559, 1491, 1473, 1244, 1029, 904, 813, 753, 691.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{24}\text{O}_2\text{Na}$, 271.1669; found, 271.1672.

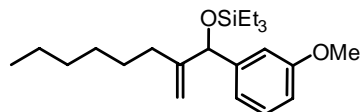


Table 2, entry 4:

The standard procedure was followed. Yield: 84%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.29–7.25 (m, 1H); 6.96–6.93 (m, 2H); 6.85–6.82 (m, 1H), 5.26 (s, 1H); 5.14 (s, 1H); 4.98 (s, 1H); 3.82 (s, 3H); 1.98–1.81 (m, 2H); 1.94 (d, J = 3.7 Hz, 1H); 1.44–1.37 (m, 2H); 1.29–1.20 (m, 6H); 0.86 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.9, 151.2, 144.1, 129.6, 119.3, 113.4, 112.3, 109.9, 77.5, 55.4, 32.0, 31.9, 29.3, 27.9, 22.8, 14.3.

IR (NaCl, thin film): 3403, 2927, 2857, 1684, 1636, 1653, 1602, 1559, 1540, 1507, 1489, 1457, 1437, 1260, 1156, 1042.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{24}\text{O}_2\text{Na}$, 271.1669; found, 271.1680.

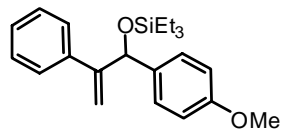


Table 2, entry 5:

The standard procedure was followed. Yield: 58%.

Characterization data of the corresponding alcohol:

¹H NMR (400 MHz, CDCl₃, δ): 7.35–7.22 (m, 7H); 6.86 (d, *J* = 8.7 Hz, 2H); 5.68 (s, 1H); 5.53 (brs, 2H); 3.80 (s, 3H); 2.04 (brs, 1H).

¹³C NMR (100 MHz, CDCl₃, δ): 159.4, 150.6, 139.6, 134.2, 128.6, 128.5, 127.8, 127.1, 114.1, 113.8, 75.6, 55.5.

IR (NaCl, thin film): 3406, 2922, 2852, 1611, 1552, 1512, 1462, 1248, 1173, 1027, 832, 666.

HRMS–ESI (*m/z*): [M+Na]⁺ calcd for C₁₆H₁₆O₂Na, 263.1043; found, 263.1053.

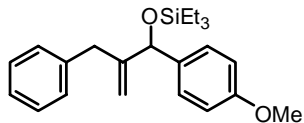


Table 2, entry 6:

The standard procedure was followed, except only 1.5 equiv of alkene was used. Yield: 74%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.31–7.27 (m, 4H); 7.23–7.22 (m, 1H), 7.13 (d, $J = 7.0$ Hz, 1H); 6.91 (d, $J = 8.6$ Hz, 2H); 5.37 (s, 1H); 5.06 (s, 1H); 4.91 (s, 1H); 3.84 (s, 3H); 3.36 (d, $J = 15.5$ Hz, 1H); 3.11 (d, $J = 15.5$ Hz, 1H); 1.93 (brs, 1H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.5, 150.8, 139.4, 134.2, 129.4, 128.5, 128.4, 126.4, 114.1, 111.9, 76.1, 55.5, 39.4.

IR (NaCl, thin film): 3406, 2959, 2923, 2852, 1642, 1511, 1463, 1378, 1250, 1159, 1071, 666.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{17}\text{H}_{18}\text{O}_2\text{Na}$, 277.1199; found, 277.1209.

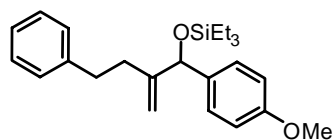


Table 2, entry 7:

The standard procedure was followed. Yield: 87%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.39–7.15 (m, 5H); 7.13 (d, $J = 1.3$ Hz, 2H), 6.88 (d, $J = 6.8$ Hz, 2H); 5.34 (s, 1H); 5.12 (s, 1H); 5.05 (s, 1H); 3.82 (s, 3H); 2.72 (t, $J = 8.1$ Hz, 1H); 2.31 (m, 2H), 1.85 (brs, 1H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.4, 150.7, 149.2, 142.1, 134.3, 128.5, 128.2, 126.0, 114.0, 110.1, 77.1, 55.5, 34.5, 33.8.

IR (NaCl, thin film): 3420, 2925, 1611, 1511, 1454, 1248, 1172, 1034, 831, 665.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{20}\text{O}_2\text{Na}$, 291.1356; found, 291.1358.

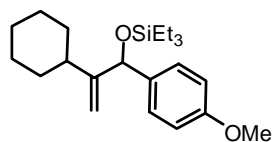


Table 2, entry 8:

The standard procedure was followed. Yield: 96%.

Characterization data of the corresponding alcohol:

¹H NMR (400 MHz, CDCl₃, δ): 7.24 (d, *J* = 8.7 Hz, 2H); 6.82 (d, *J* = 8.7 Hz, 2H); 5.21 (s, 1H); 5.08 (s, 1H); 4.88 (s, 1H); 3.80 (s, 3H); 1.74–1.53 (m, 6H); 1.22–1.02 (m, 2H); 0.90 (t, *J* = 8.0 Hz, 9H); 0.56 (dq, *J* = 8.0, 1.5 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃, δ): 158.8, 157.9, 136.0, 128.1, 113.4, 107.8, 77.1, 55.4, 39.7, 34.5, 33.5, 27.2, 27.0, 26.6, 7.1, 5.1.

IR (NaCl, thin film): 3425, 2925, 2852, 1643, 1511, 1462, 1247, 1171, 1073, 890, 855, 665.

HRMS–ESI (*m/z*): [M+Na]⁺ calcd for C₂₂H₃₆O₂SiNa, 383.2377; found, 383.2387.

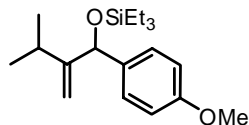


Table 2, entry 9:

The standard procedure was followed, except the reaction was carried out in sealed tube and using 1 mL of alkene. Yield: 93%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.30 (d, $J = 8.6$ Hz, 2H), 6.89 (d, $J = 8.6$ Hz, 2H), 5.29 (s, 1H), 5.17 (d, $J = 3.3$ Hz, 1H), 5.06 (s, 1H), 3.83 (s, 3H), 2.11 (m, 1H), 1.91 (brs, 1H), 1.01 (d, $J = 6.8$ Hz, 3H), 1.00 (d, $J = 6.8$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.4, 158.1, 134.8, 128.5, 114.0, 107.3, 76.3, 55.4, 30.4, 23.4, 22.5.

IR (NaCl, thin film): 3395, 2960, 2930, 2871, 1644, 1611, 1586, 1511, 1463, 1303, 1249, 1173, 1096, 1035, 904, 853, 831.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{13}\text{H}_{18}\text{O}_2\text{Na}$, 229.1199; found, 229.1206.

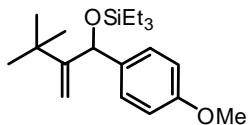


Table 2, entry 10:

The standard procedure was followed, except the reaction was carried out in sealed tube and using 40 mol% of Ni(cod)₂, IPr and 60 mol% of P(OPh)₃. Yield: 13%.

Characterization data of the corresponding alcohol:

¹H NMR (400 MHz, CDCl₃, δ): 7.34 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 5.39 (d, *J* = 4.1 Hz, 1H), 5.27 (s, 1H), 5.24 (d, *J* = 0.7 Hz, 1H), 3.83 (s, 3H), 1.73 (d, *J* = Hz, 1H), 1.10 (s, 9H).

¹³C NMR (100 MHz, CDCl₃, δ): 160.6, 159.3, 136.0, 128.5, 113.9, 110.0, 72.8, 55.5, 35.7, 30.1.

IR (NaCl, thin film): 3405, 2956, 2876, 1698, 1598, 1511, 1489, 1462, 1314, 1261, 1191, 1161, 1072, 1025, 9625, 861, 833, 727, 690, 666.

HRMS–ESI (m/z): [M+Na]⁺ calcd for C₁₄H₂₀O₂Na, 243.1356; found, 243.1367.

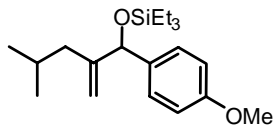


Table 2, entry 11:

The standard procedure was followed. Yield: 99%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.28 (d, $J = 8.7$ Hz, 2H); 6.89 (d, $J = 8.7$ Hz, 2H); 5.32, (s, 1H); 5.06 (s, 1H); 4.96 (s, 1H); 3.81 (s, 3H); 1.94 (d, $J = 3.6$ Hz, 1H); 1.83–1.71 (m, 3H); 0.85 (t, $J = 5.6$ Hz, 9H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.4, 150.0, 134.5, 128.4, 114.0, 110.5, 76.7, 55.5, 42.3, 26.5, 23.0, 22.5.

IR (NaCl, thin film): 3402, 2954, 2869, 2837, 1647, 1611, 1587, 1512, 1465, 1384, 1366, 1303, 1249, 1173, 1109, 1036, 906, 851, 829, 780.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{20}\text{H}_{34}\text{O}_2\text{SiNa}$, 357.2220; found, 357.2214.

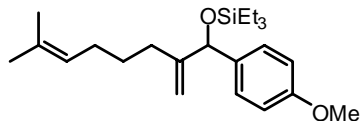


Table 2, entry 12:

The standard procedure was followed. Yield: 94%.

Characterization data of the corresponding alcohol:

^1H NMR (400 MHz, CDCl_3 , δ): 7.29 (d, $J = 8.7$ Hz, 2H); 6.88 (d, $J = 8.7$ Hz, 2H); 5.27 (s, 1H); 5.10 (d, $J = 3.1$ Hz, 1H); 5.06 (t, $J = 7.1$ Hz, 1H); 4.98 (s, 1), 3.82 (s, 3H); 1.96–1.84 (m, 5H); 1.67 (s, 3H); 1.56 (s, 3H); 1.48–1.42 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 159.3, 151.4, 134.6, 131.9, 128.2, 124.5, 114.0, 109.4, 77.0, 55.5, 31.9, 28.2, 27.9, 25.9, 17.9.

IR (NaCl, thin film): 3404, 2929, 1644, 1611, 1586, 1511, 1442, 1377, 1303, 1248, 1172, 1108, 1036, 902, 830.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{38}\text{O}_2\text{SiNa}$, 397.2533; found, 397.2544.

Table 2, entry 13 compound characterization has been reported in previous phosphine system, see: Ng, S.-S.; Jamison, T. F. *J. Am. Chem. Soc.* **2005**, *127*, 14194-14195.

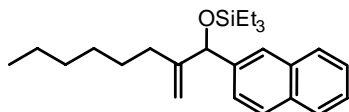


Table 2, entry 14:

The standard procedure was followed. Yield: 73%.

¹H NMR (400 MHz, CDCl₃, δ): 7.87–7.78 (m, 4H); 7.50–7.27 (m, 3H); 5.30 (s, 2H); 4.92 (s, 1H); 2.00–1.94 (m, 2H); 1.80–1.76 (m, 2H); 1.40–1.34 (m, 2H); 1.26–1.19 (m, 6H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.84 (t, *J* = 7.0 Hz, 3H), 0.63 (dq, *J* = 7.9, 1.9 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃, δ): 152.0, 141.2, 133.4, 133.0, 128.2, 127.9, 127.8, 126.0, 125.7, 125.1, 125.1, 109.7, 78.3, 31.9, 30.8, 29.3, 27.8, 22.8, 14.3, 7.1, 5.1.

IR (NaCl, thin film): 2955, 2929, 2875, 1652, 1559, 1507, 1458, 1239, 1122, 1080, 1006, 901, 857, 818, 743.

HRMS–ESI (m/z): [M+Na]⁺ calcd for C₂₅H₃₈OSiNa, 405.2590; found, 405.2583.

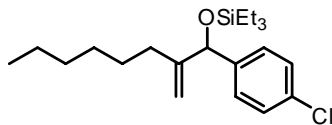


Table 2, entry 15:

The standard procedure was followed, except 40 mol% of Ni(cod)₂, IPr and 60 mol% of P(OPh)₃ was used, and the product was purified with hexane. Yield: 64%.

¹H NMR (400 MHz, CDCl₃, δ): 7.32–7.27 (m, 4H), 5.21 (s, 1H), 5.12 (s, 1H), 4.89 (s, 1H), 1.96–1.90 (m, 1H), 1.76–1.72 (m, 1H), 1.57–1.21 (m, 8H), 0.94 (t, *J* = 8.0 Hz, 9H), 0.88 (t, *J* = 7.1 Hz, 3H), 0.60 (q, *J* = 8.0 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃, δ): 151.9, 142.4, 132.8, 128.3, 127.9, 109.9, 77.7, 31.9, 30.6, 29.3, 27.9, 22.8, 14.3, 7.0, 5.0.

IR (NaCl, thin film): 2928, 2857, 1653, 1596, 1457, 1076, 1029, 904.

HRMS–ESI (*m/z*): [M+Na]⁺ calcd for C₂₁H₃₅OClSiNa, 389.2038; found, 389.2055.

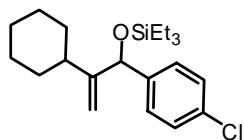


Table 2, entry 16:

The standard procedure was followed, except the product was purified with hexane only.

Yield: 21%.

^1H NMR (400 MHz, CDCl_3 , δ): 7.30–7.26 (m, 4H), 5.22 (t, $J = 1.3$ Hz, 1H), 5.12 (s, 1H), 4.92 (s, 1H), 1.73–1.56 (m, 6H); 1.16–1.11 (m, 2H); 0.92 (t, $J = 7.9$ Hz, 9H); 0.59 (dq, $J = 7.9, 0.4$ Hz, 6H).

^{13}C NMR (100 MHz, CDCl_3 , δ): 157.3, 142.5, 132.8, 128.3, 108.7, 78.2, 39.5, 34.5, 33.6, 27.1, 26.5, 7.0, 5.1.

IR (NaCl, thin film): 2926, 2876, 2852, 1644, 1596, 1489, 1448, 1408, 1239, 1130, 1080, 1015, 975, 890, 853, 808, 726, 666.

HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{21}\text{H}_{33}\text{OClSiNa}$, 387.1881; found, 387.1899.

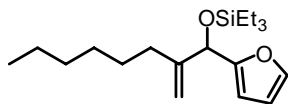


Table 2, entry 17:

The standard procedure was followed, except the reaction was carried out at room temperature, and with 40 mol% of Ni(cod)₂, IPr and 60 mol% of P(OPh)₃. Yield: 36%.

Characterization data of the corresponding alcohol:

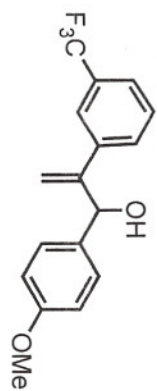
¹H NMR (400 MHz, CDCl₃, δ): 7.41 (m, 1H), 6.36 (m, 1H), 6.28 (m, 1H), 5.27 (s, 1H), 5.20 (d, *J* = 4.9 Hz, 1H), 5.06 (s, 1H), 2.08 (d, *J* = 4.9 Hz, 1H), 2.11–1.91 (m, 2H), 1.52–1.37 (m, 2H), 1.36–1.19 (m, 6H), 0.84 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, δ): 155.1, 149.0, 142.5, 110.7, 110.5, 107.4, 71.0, 32.5, 31.9, 29.3, 28.0, 22.8, 14.3.

IR (NaCl, thin film): 3367, 2957, 2929, 2858, 1651, 1503, 1466, 1378, 1223, 1143, 1039, 1010, 907, 798, 735, 598.

HRMS–ESI (m/z): [M+Na]⁺ calcd for C₁₃H₂₀O₂Na, 231.1356; found, 231.1358.

TES deprot m-CF3 Styrene Addt



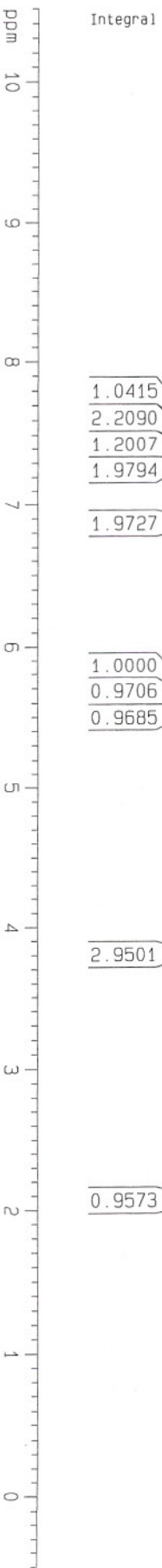
Current Data Parameters
 NAME NcyB-5546A
 EXPNO 1
 PROCNO 1

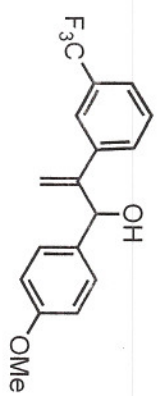
F2 - Acquisition Parameters
 Date_ 20050822
 Time 19.04
 INSTRUM spect
 PULPROG gm BB0 BB-1
 ID ZG30
 SOLVENT CUC12
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.135314 Hz
 AQ 3.9984243 sec
 RG 256
 DW 60.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 S1 32768
 SF 400.1300000 MHz
 MDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR Q10t Parameters
 CX 24.00 cm
 F1P 10.500 ppm
 F1 4201.35 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPM1CH 0.45833 ppm/cm
 MZCH 183.39291 Hz/cm





TES deprot m-CF3 styrene Apdt

Current Data Parameters
 NAME N79-25404
 EXPNO 2
 PRQNO 1

F2 - Acquisition Parameters
 DATE_ 20080822
 TIME 19.06
 INSTRUM spect
 PULPROG sm BRD BR-I
 PULPROG 299P30
 TO 65556
 SOLVENT CDCl3
 NS 14924
 DS 4
 SWH 24975.621 Hz
 FIDRES 0.319572 Hz
 AQ 1.3172656 sec
 AD 1.0000000
 DM 20.740 usec
 DE 6.100 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 15.29 usec
 PL1 3.00 dB
 SF01 100.6237569 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1360005 MHz

F2 - Processing parameters
 SI 32769
 SF 100.6127491 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

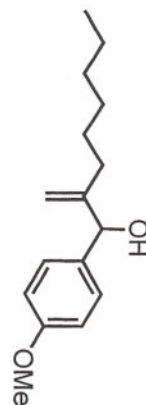
ID NMR plot parameters
 CX 24.00 cm
 F1P 226.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -98613 Hz
 PRQPCW 9.86133 ppm/cm
 HZCM 964.25944 Hz/cm

- 159.572
- 149.497
- 140.430
- 133.616
- 130.567
- 130.490
- 128.857
- 128.476
- 124.479
- 124.034
- 115.310
- 114.202
- 77.944
- 77.548
- 77.231
- 76.913
- 75.601
- 55.485

Chemical shift scale in ppm, ranging from 0 to 200.



TES deprotection, 1-octene



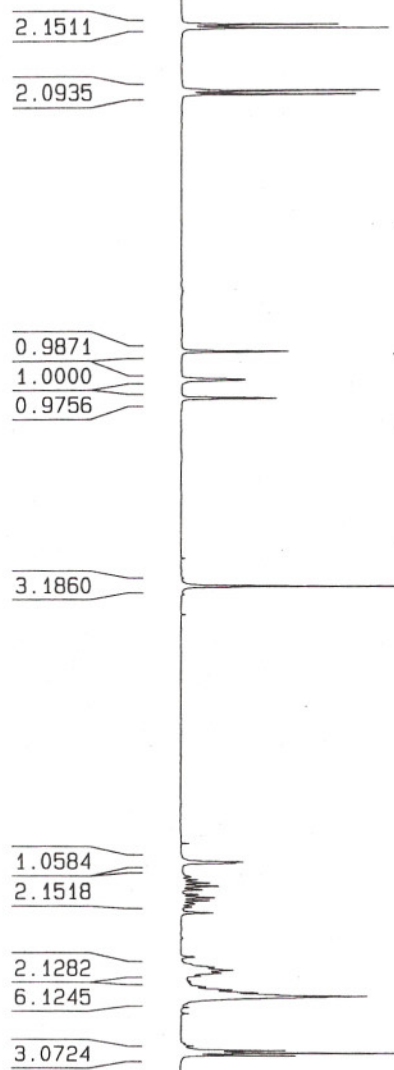
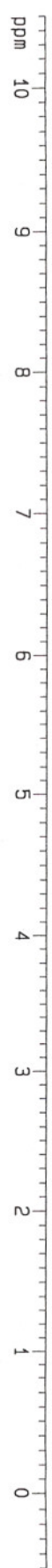
Current Data Parameters
 NAME hcyl-256-de-oct
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060722
 Time 11:35
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SFO1 400.132710 MHz
 SOLVENT CDCl3
 NS 1
 DS 1
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DM 60.400 usec
 DE 6.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCKR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 H
 P1 9.88 usec
 PL1 3.00 dB
 SFO1 400.132710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300054 MHz
 MW EM
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR Plot Parameters
 CX 24.00 cm
 CY 12.00 cm
 F1P 10.500 ppm
 F1 4201.35 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCH 0.45833 ppm/cm
 HZCM 183.38291 Hz/cm



Peak chemical shifts (ppm):
 2.1511
 2.0935
 0.9871
 1.0000
 0.9756
 3.1860
 1.0584
 2.1518
 2.1282
 6.1245
 3.0724

Current Data Parameters
 NAME hc98-226a-dr-act
 EXNO 2
 PROCNO 1

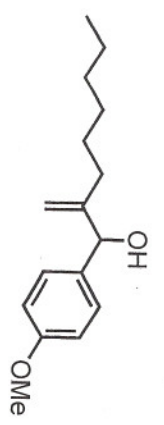
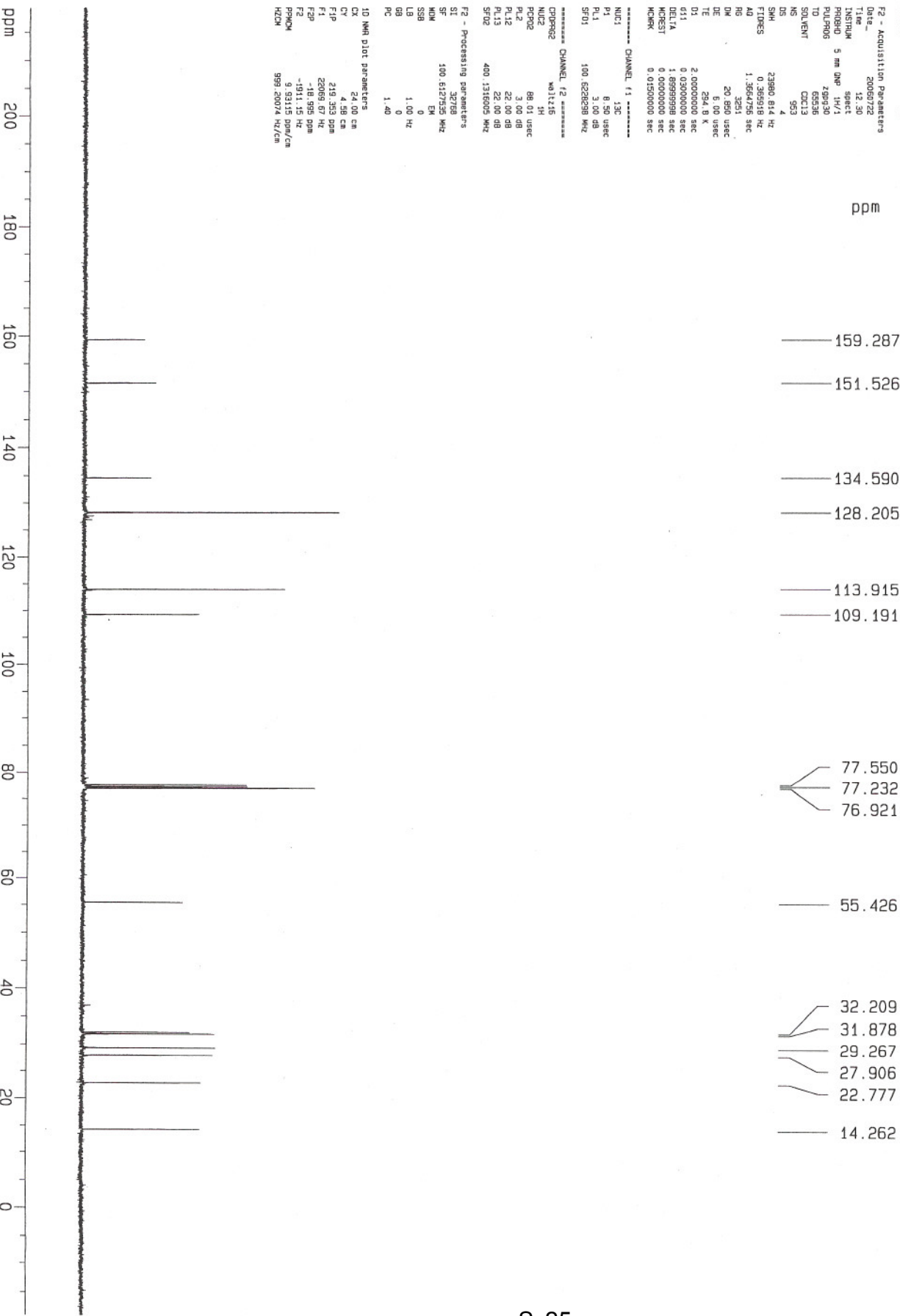
F2 - Acquisition Parameters
 Date_ 20060722
 Time 12.30
 INSTRUM spect
 PROBD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 529
 DS 2
 SMI 23980.814 Hz
 FIDRES 0.366318 Hz
 AQ 1.3664756 sec
 RG 3251
 DE 20.860 usec
 TE 294.4 K
 D1 2.00000000 sec
 DELTA 0.03000000 sec
 MCKEY 1.69999998 sec
 MCKEY 0.00000000 sec
 MCKEY 0.00000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.50 usec
 PL1 3.00 dB
 SFO1 100.6282398 MHz

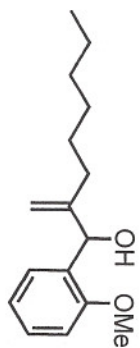
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 88.01 usec
 PL2 3.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127655 MHz
 MD 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR Dat Parameters
 CX 24.00 cm
 CY 4.58 cm
 F1P 219.353 DPM
 F1R 22069.87 Hz
 F2P -18.399 DPM
 F2R 913115 Hz/cm
 PSIGN 999.20074 Hz/cm
 HZCM



TES deprotected o-OMe Addt



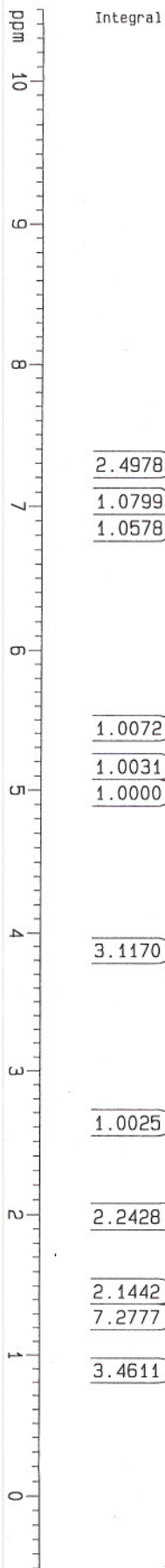
Current Data Parameters
 NAME hcp6-234cde-4
 EXNO 1
 PROCNO 1

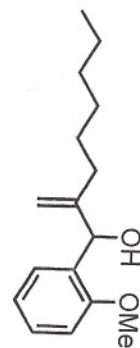
F2 - Acquisition Parameters
 Date_ 20060731
 Time 18.04
 INSTRUM spect
 PROBHD Sma BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 2
 DS 2
 SMI 8278.148 Hz
 FIDRES 0.126314 Hz
 AQ 3.996243 sec
 RG 161.3
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WHW 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 24.00 cm
 F1P 10.500 ppm
 F1 4201.36 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPM/CN 0.45833 ppm/cm
 HZ/CN 183.35291 Hz/cm





Current Data Parameters
 NAME hcvh-23acde-A
 EXPNO 2
 PROCNO 1

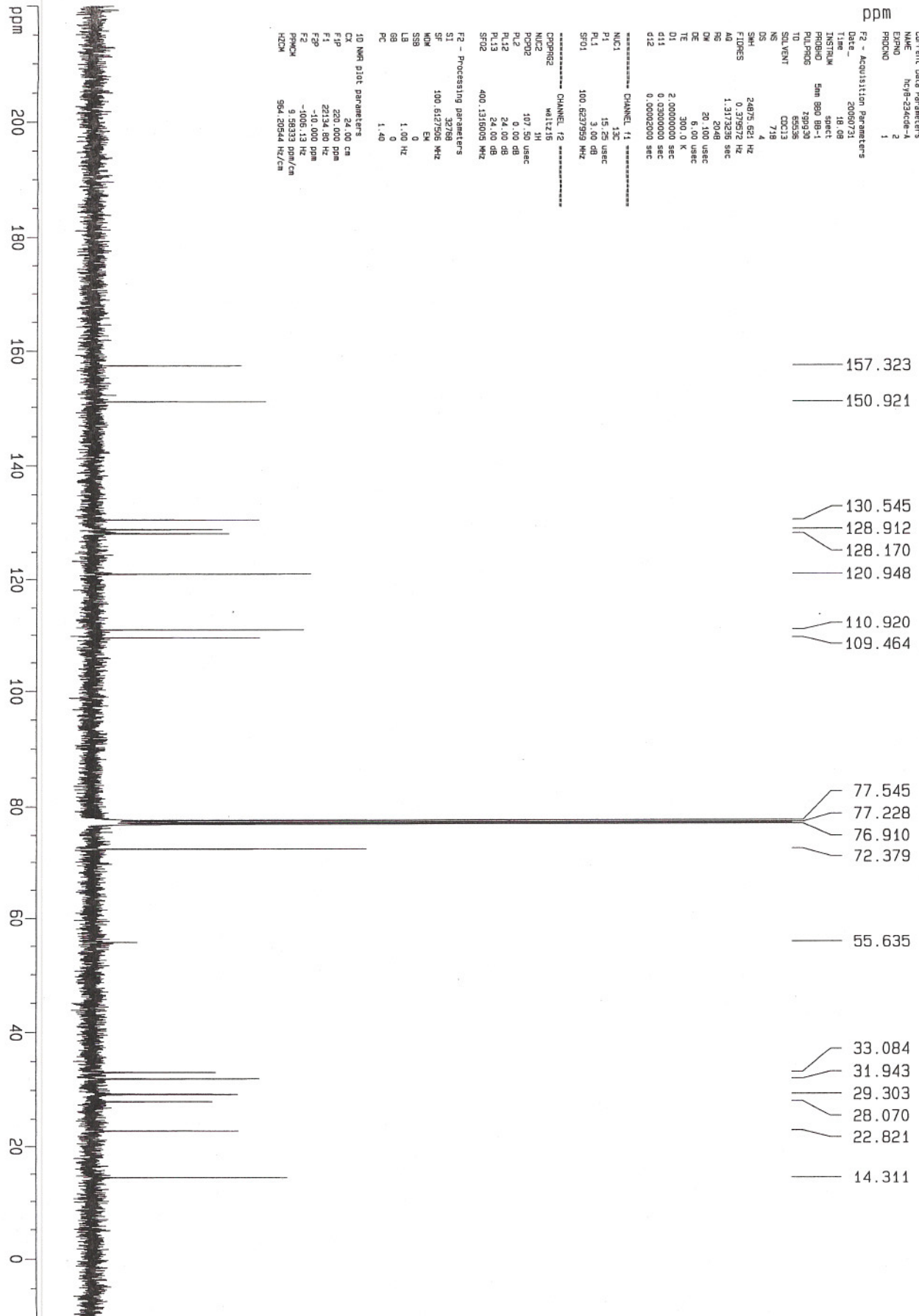
F2 - Acquisition Parameters
 Date_ 2008/07/31
 Time 18:08
 INSTRUM spect
 PROBHD 5mm BBO
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 718
 DS 4

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.00 usec
 PL1 0.00 dB
 SFO1 100.627399 MHz

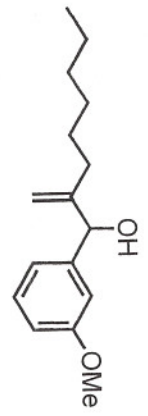
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SFO2 400.1315005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127505 MHz
 MDM EM
 SSB 0
 GB 1.00 Hz
 BR 0
 PC 1.40

1D NMR p10t parameters
 CX 24.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1005.13 Hz
 PRCMCH 9.98333 ppm/cm
 HZCM 964.20544 Hz/cm



m-OMe Addt



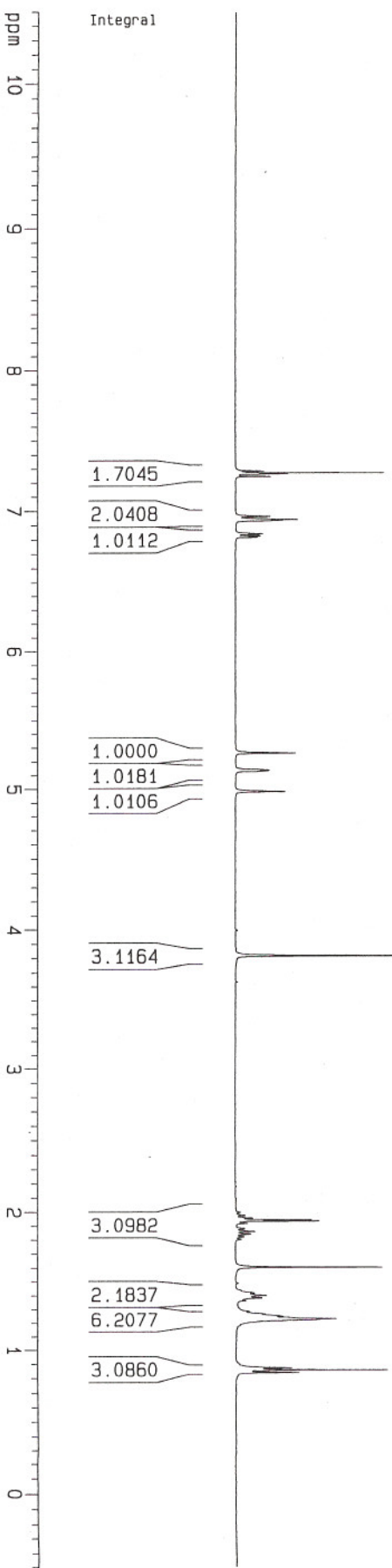
Current Data Parameters
 NAME hcyf-234de-mOme
 EXPNO 111
 PROCNO 1

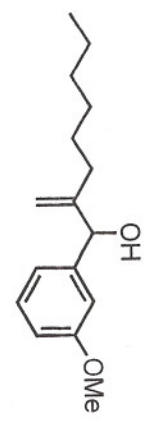
F2 - Acquisition Parameters
 Date_ 20060726
 Time 20.38
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zg30
 ID 65256
 SOLVENT CDCl3
 NS 8
 DS 1
 SWH 8378.146 Hz
 FIDRES 0.12514 Hz
 AQ 3.956424 sec
 SFO 562
 DM 60.400 usec
 DE 6.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCHNK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 9.89 usec
 PL1 3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32769
 SF 400.1300524 MHz
 WHW 64
 EQ 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR slot parameters
 CX 24.00 cm
 CY 7.79 cm
 FIP 10.500 ppm
 F1 4201.36 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCN 0.45933 ppm/cm
 MDCN 163.59291 Hz/cm





Current Data Parameters
 NAME nc98234de-8DME
 EXPNO 222
 PROCNO 1

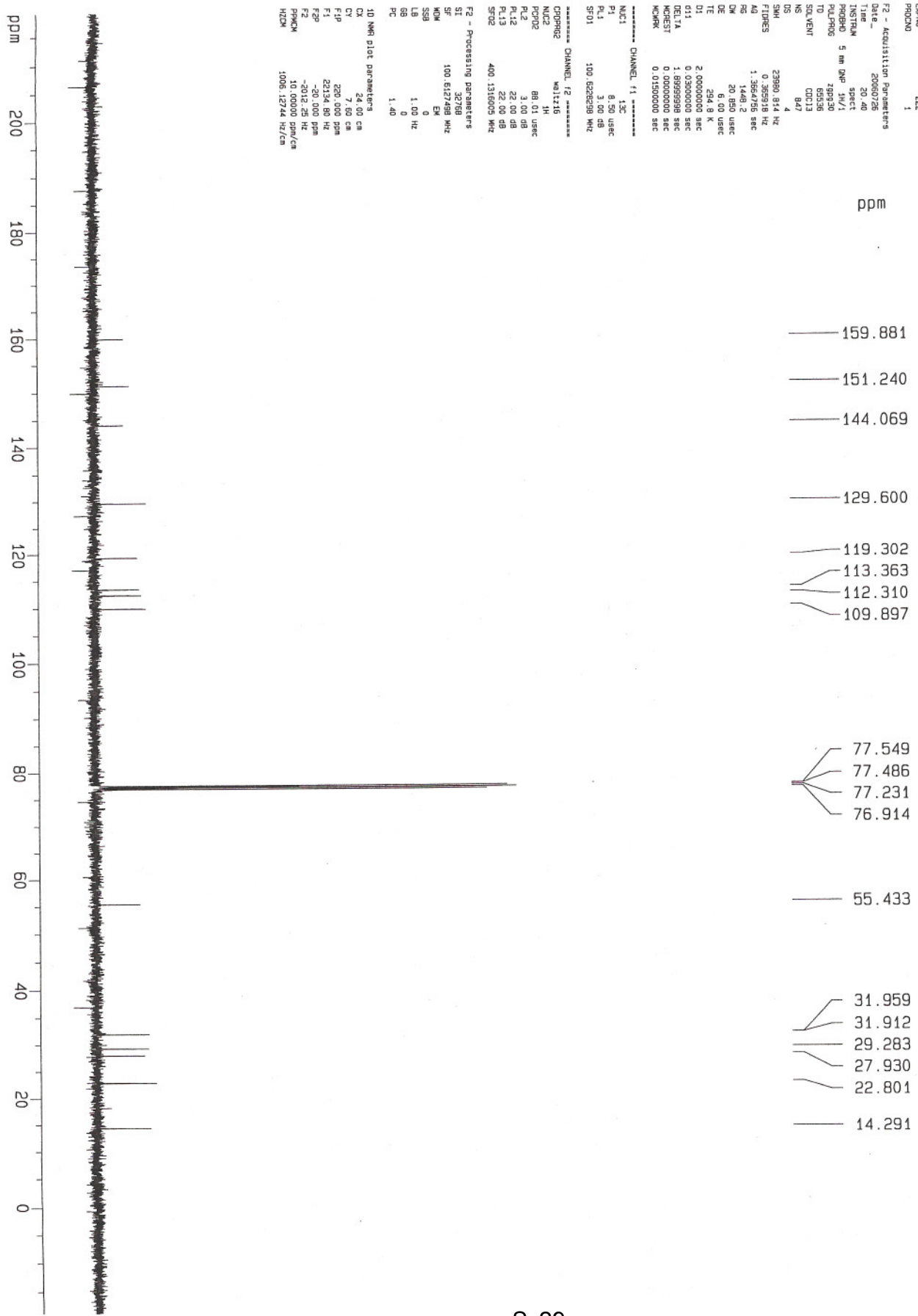
F2 - Acquisition Parameters
 Date_ 20060726
 Time 20.40
 INSTRUM spect
 PROCNO 5 nm DNP 1H/1
 PULPROG zgpg30
 TD 65536
 SFO1 100.626299 MHz
 SOLVENT CDCl3
 NS 847
 DS 4
 SWH 3390.844 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664766 sec
 RG 1448.2
 DW 20.850 usec
 DE 6.00 usec
 TE 294.8 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89959998 sec
 MCHRES 0.00000000 sec
 MCPR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 PL1 3.00 dB
 SFO1 100.626299 MHz

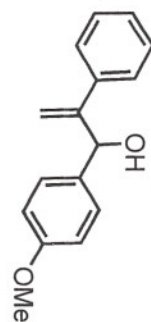
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPRG2 88.01 usec
 PUL2 3.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6124489 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 24.00 cm
 CY 7.60 cm
 F1P 220.000 ppm
 F1 62134.88 Hz
 F2P -20.000 ppm
 F2 -2015.23 Hz
 FREQM 10.00000 DPM/cm
 MICH 1000.12744 HZ/cm



Styrene, p-Anisaldendye, TES deprotection

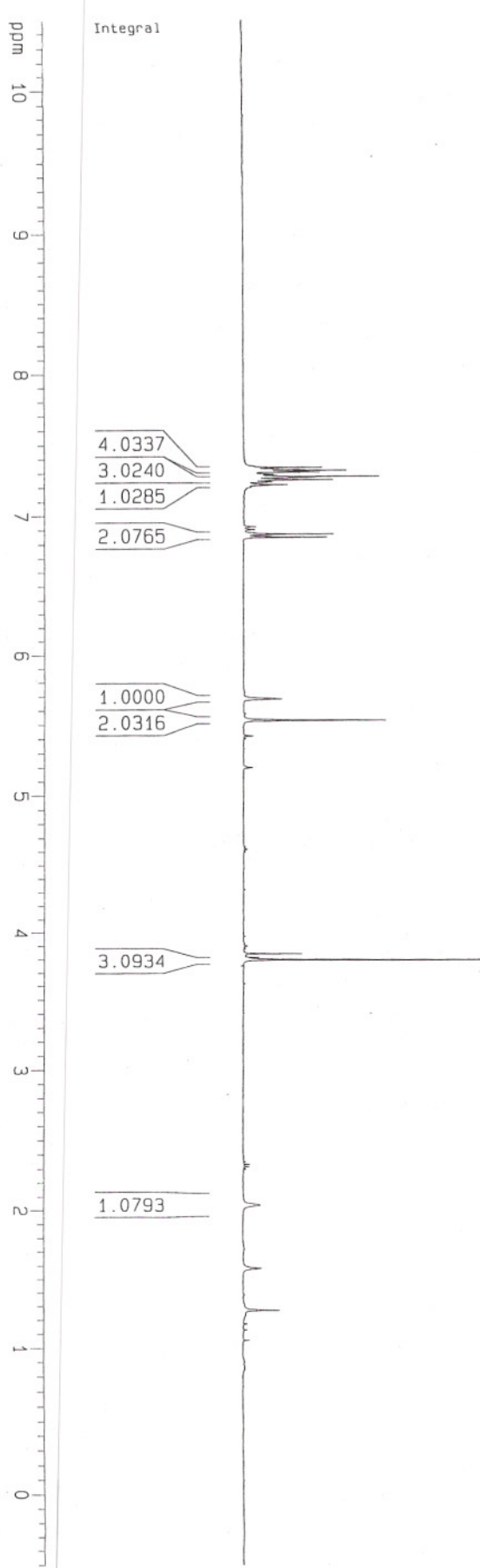


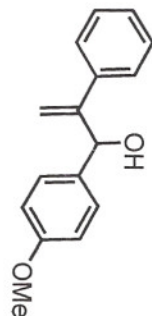
inv. R-122 to Ph CH₂CH₂OMe

Current Data Parameters
 NAME hcv18-17308
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060428
 Time 12:05
 INSTRUM spect
 PROBNM 5mm BBO BB-1
 PULPROG zg30
 TO 65536
 SOLVENT CDCl₃
 NS 8
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.166314 Hz
 AQ 3.1594443 sec
 RG 60
 FM 400
 DE 60.400 usec
 TE 300.0 K
 D1 1.00000000 sec
 ***** CHANNEL f1 *****

NUC1 ¹H
 P1 7.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz
 F2 - Processing Parameters
 SI 32768
 SF 400.1300018 MHz
 KW 64
 EN 16
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 ID WAT plot parameters
 CX 24.00 cm
 FIP 10.500 ppm
 F1 4201.36 Hz
 F2 -0.500 ppm
 F2 -200.07 Hz
 FREQHZ 0.45833 ppm/cm
 HZCM 183.39291 Hz/cm





Current Data Parameters
 NAME hcf-1736
 EXPNO 2
 PROCNO 1

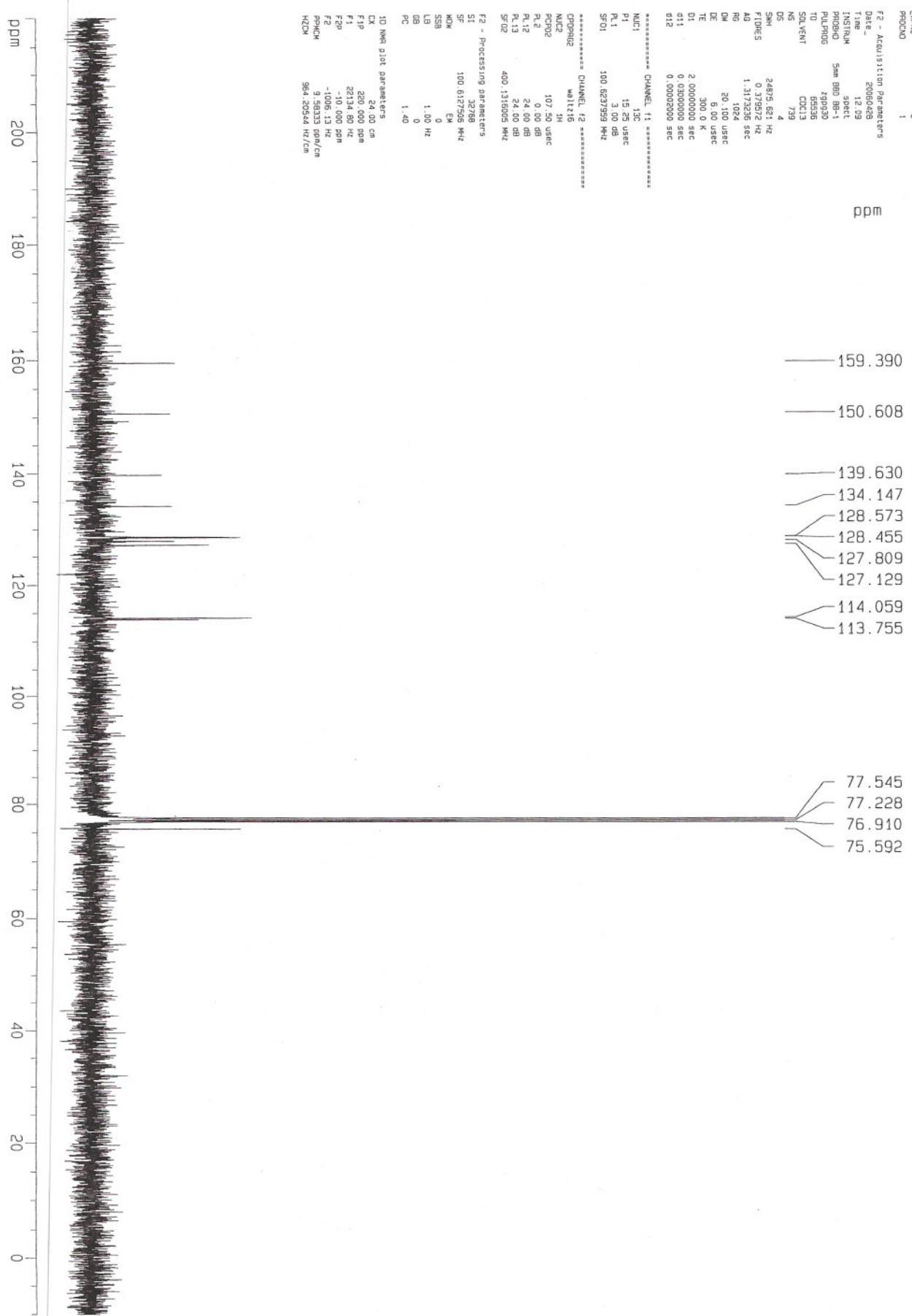
F2 - Acquisition Parameters
 Date_ 20060428
 Time 12.09
 INSTRUM spect
 PROBRD Sm B60 BB-1
 PULPROG zgpg30
 TD 89536
 SOLVENT CDCl3
 NS 759
 DS 2
 SFO1 24976.624 Hz
 P1 0.370924 sec
 A0 1.317326 sec
 RG 1024
 DM 20.100 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 15.25 usec
 PL 3.00 dB
 SFO1 100.6273959 MHz

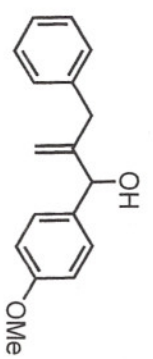
***** CHANNEL f2 *****
 PPGPRG2 waltz16
 NUC2 1H
 PCPD2 107.50 usec
 PL 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SFO2 400.1315005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127596 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 C1 20.000 cm
 F1p 230.000 ppm
 F1 22134.80 Hz
 F2p -10.000 ppm
 F2 -1006.13 Hz
 PPMICH 9.58333 ppm/cm
 HZCM 964.20544 Hz/cm



TES deprotected A11Y1Ph Apdt



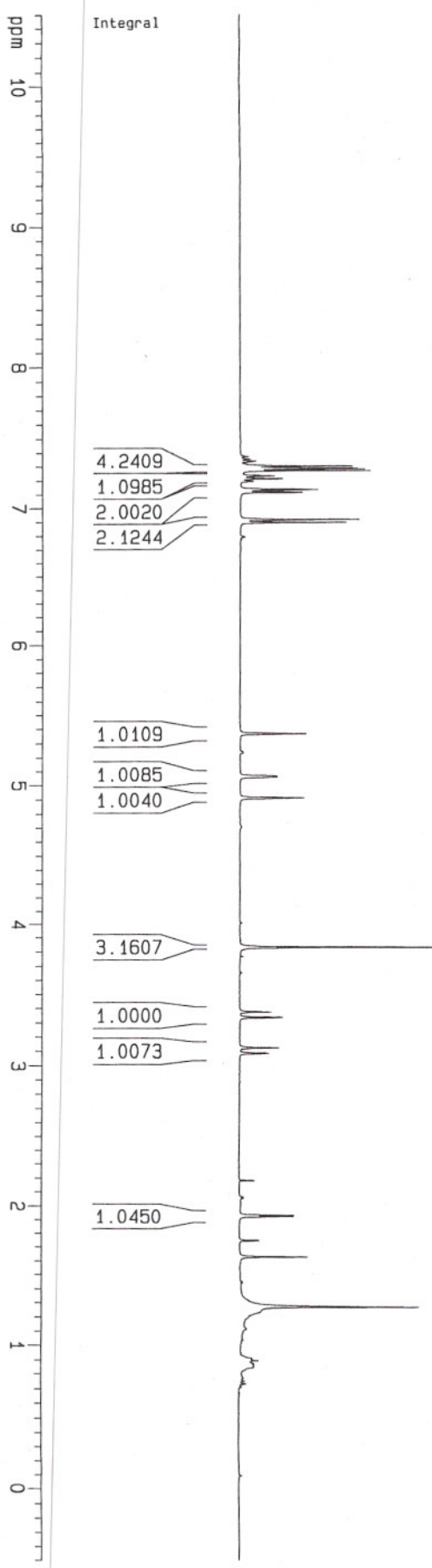
Current Data Parameters
 Name: 16319-269Hme 4
 ExpNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20060727
 Time: 12.26
 INSTRUM: spect
 PROBHD: 5 mm QNP 1H/1
 PULPROG: zg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 13
 DS: 1
 SWH: 8278.146 Hz
 FIDRES: 0.186314 Hz
 AQ: 3.3596241 sec
 RG: 655.36
 DM: 64.00 usec
 DE: 6.00 usec
 TE: 294.2 K
 O1: 1.00000000 sec
 MCHRG1: 0.00000000 sec
 MCHRG2: 0.01500000 sec

***** CHANNEL f1 *****
 NUC1: 1H
 P1: 9.88 usec
 PL1: 3.00 dB
 SF01: 400.1324710 MHz

F2 - Processing parameters
 SI: 32768
 SF: 400.1300524 MHz
 WHW: 0
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR list parameters
 CX: 24.00 cm
 CY: 7.79 cm
 F1P: 10.500 ppm
 F1: 4201.35 Hz
 F2P: -0.500 ppm
 F2: -200.07 Hz
 PPM0A: 0.45833 ppm/cm
 PPM0B: 163.39231 Hz/cm



Current Data Parameters
 Name: ncp-2207ms-2
 EXPNO: 1
 PROCNO: 1

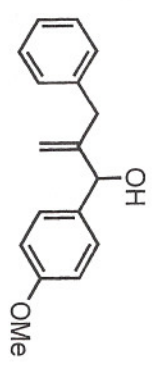
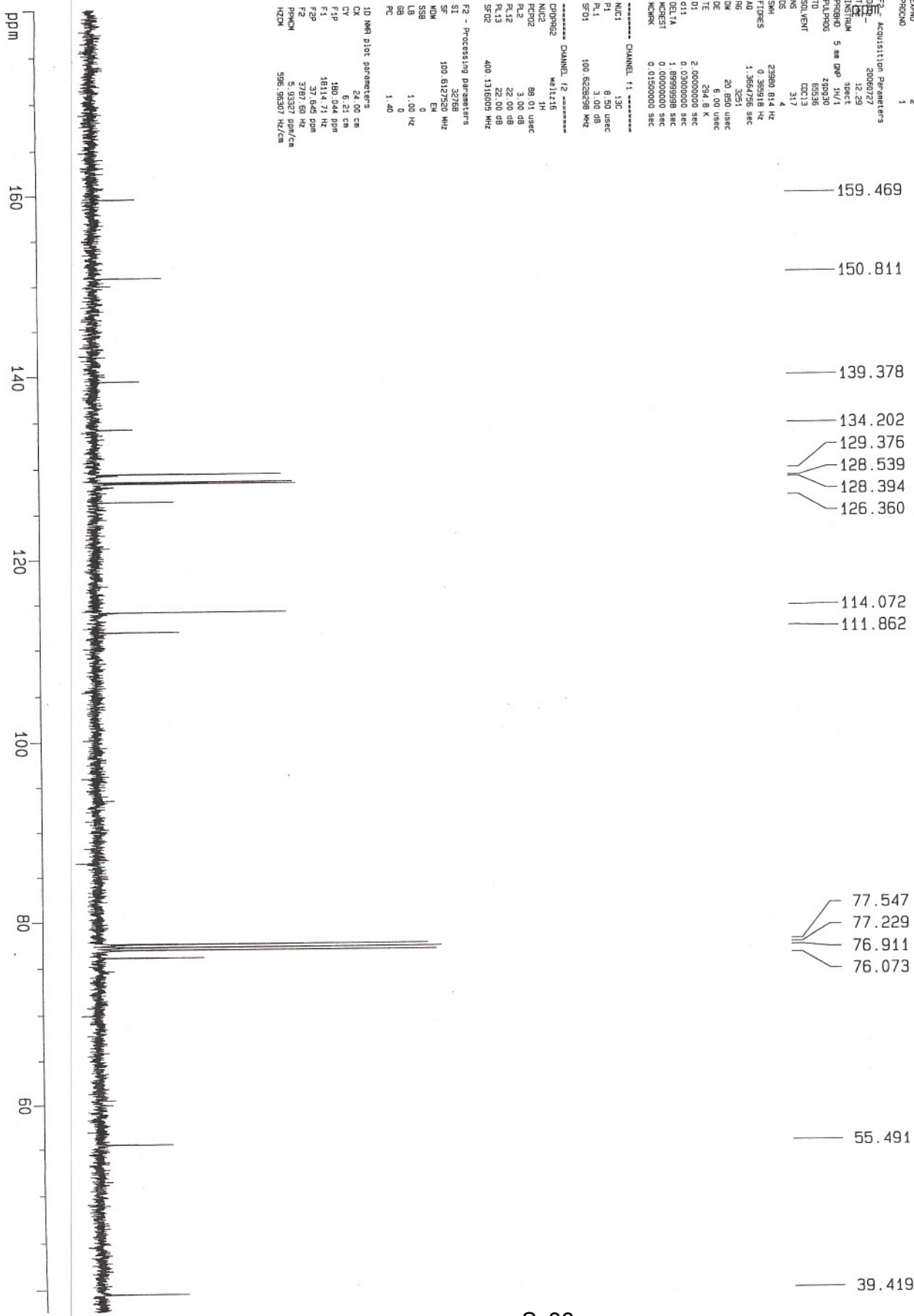
F2 - Acquisition Parameters
 Date_ : 20060727
 Time : 12.29
 INSTRUM : spect
 PRCBND : 5 mm QNP 1H/1
 PULPROG : zgpg30
 TO : 65936
 SOLVENT : CDCl3
 NS : 317
 DS : 4
 SWH : 2399.814 Hz
 FIDRES : 0.08241 Hz
 AQ : 1.366726 sec
 RG : 3251
 DW : 20.650 usec
 DE : 6.00 usec
 TE : 294.8 K
 D1 : 2.0000000 sec
 d11 : 0.0300000 sec
 DELTA : 1.8999998 sec
 MCKEY : 0.0000000 sec
 MCKK : 0.0150000 sec

***** CHANNEL f1 *****
 NUC1 : 13C
 P1 : 8.50 usec
 PL1 : 0.00 dB
 SF01 : 100.626268 MHz

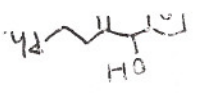
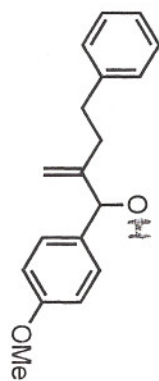
***** CHANNEL f2 *****
 CPDPRG2 : waltz16
 NUC2 : 1H
 P2 : 98.01 usec
 PL2 : 3.00 dB
 PL12 : 22.00 dB
 PL13 : 22.00 dB
 SF02 : 400.1316005 MHz

F2 - Processing Parameters
 SI : 32768
 SF : 100.6127261 MHz
 WDW : 0
 SSB : 0
 LB : 1.00 Hz
 GB : 0
 PC : 1.40

1D NMR plot parameters
 CX : 24.00 cm
 CY : 6.21 cm
 F1P : 180.044 ppm
 F1 : 18114.71 Hz
 F2P : 37.545 ppm
 F2 : 37871.69 Hz
 PPM0H : 2.33323 dip/cen
 N2DM : 598.58307 Hz/cen



TES depot Apdt: HomallylPh



Y. D. Miller, P.

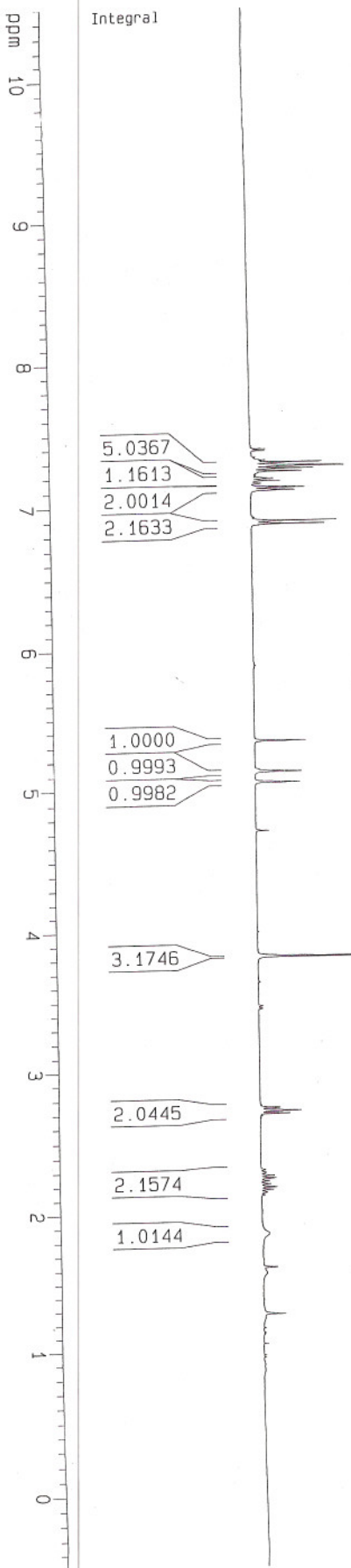
Current Data Parameters
 NAME hcy6-16detr2
 EXPNO 1
 PROCNO 1

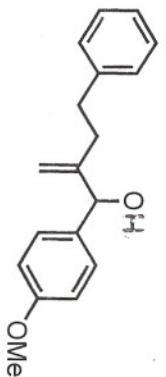
F2 - Acquisition Parameters
 Date_ 2006/02/1
 Time 15:41
 INSTRUM spect
 PULPROG zgpg30
 PCVPRG 5 mm QNP 1H/1
 TV 1030
 SOLVENT DMSO-d6
 NS 11
 DS 1
 SFO 8276.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9594243 sec
 RG 3282.5
 CW 60.400 usec
 DE 6.00 usec
 TE 299.1 K
 O1 1.00000000 sec
 MDEL 0.00000000 sec
 MCKEY 0.01500000 sec

***** CHANNEL f1 *****
 NUCL1 1H
 P1 9.00 usec
 PL1 3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300054 MHz
 KW 0
 EM 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 24.00 cm
 CY 5.95 cm
 F1 10.500 ppm
 F2 400.1300054 MHz
 F2P -300.07 Hz
 PR 0.45833 ppm/cm
 HDN 183.39291 Hz/cm





Current Data Parameters
 NAME hcq8-16dat121
 EXPNO 2
 PROCNO 1

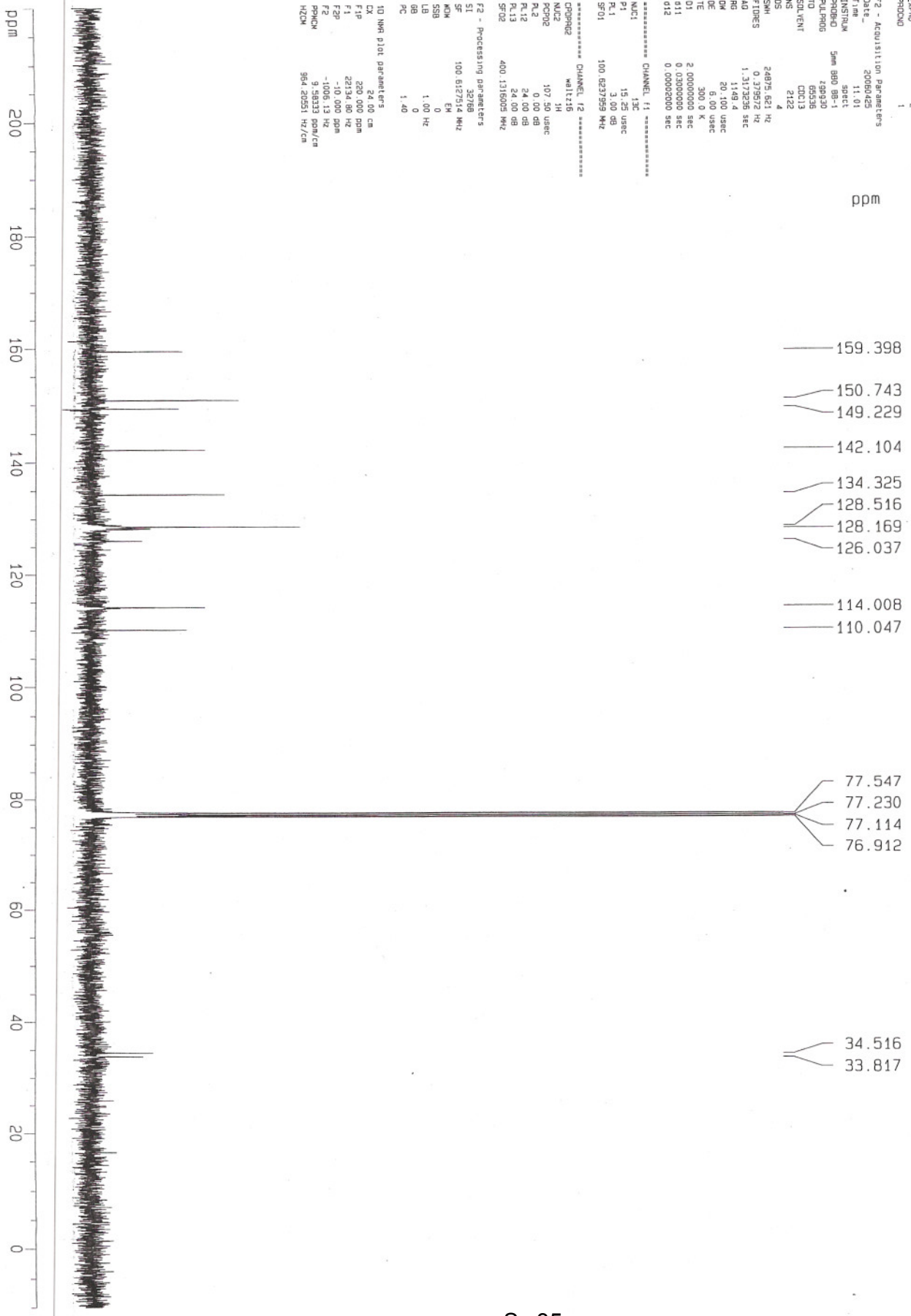
F2 - Acquisition Parameters
 Date_ 20060425
 Time 11:01
 INSTRUM spect
 PROBR0 5mm BBO BB-1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 2122
 DS 4
 SWH 24875.621 Hz
 FIDRES 0.375972 Hz
 AQ 1.317229 sec
 RG 3149
 DM 201400 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.00000000 sec
 O11 0.03000000 sec
 O12 0.00000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 15.25 usec
 PL1 3.00 dB
 SF01 100.627959 MHz

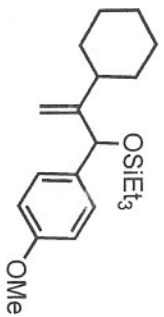
***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PCPRG2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.131802 MHz

F2 - Processing parameters
 SI 32768
 SF 100.617514 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 24.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPHC1H 9.58533 ppm/cm
 HZCM 964.20551 Hz/cm



Vinylcy, Apdt



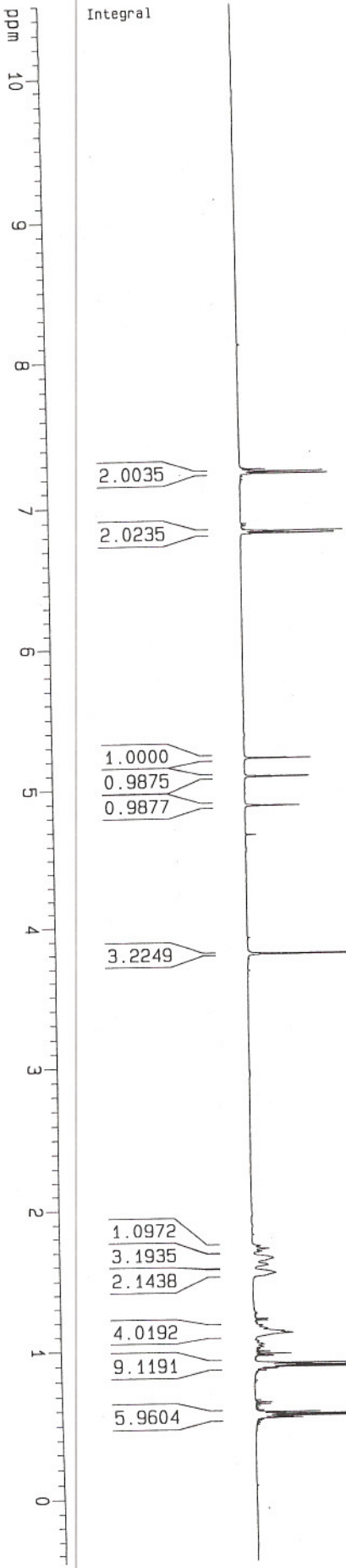
Current Data Parameters
 NAME ncyb-1520f2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060419
 Time 16.06
 INSTRUM spect
 PROBRD 5 mm QNP1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMS-D6
 NS 10
 DS 0
 SMH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4079740 sec
 RG 8
 DM 52.000 usec
 DE 5.00 usec
 TE 303.0 K
 D1 1.00000000 sec
 MCPRST 0.00000000 sec
 MCPRK 0.01500000 sec

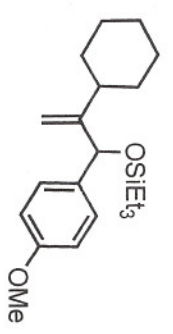
***** CHANNEL f1 *****
 NUC1 1H
 P1 10.00 usec
 PL1 0.80 dB
 SFO1 500.132834 MHz

F2 - Processing parameters
 SI 32768
 SF 600.1300170 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR 01st parameters
 CX 25.00 cm
 CY 8.49 cm
 F1P 10.500 ppm
 F1 6301.37 Hz
 F2P -0.500 ppm
 F2 -300.07 Hz
 PPM0M 0.44000 ppm/cm
 WZCM 264.05719 Hz/cm



Vinylcyclohexene, Apdt



Current Data Parameters
 NAME hcid-182d
 EXPNO 2
 PROCNO 1

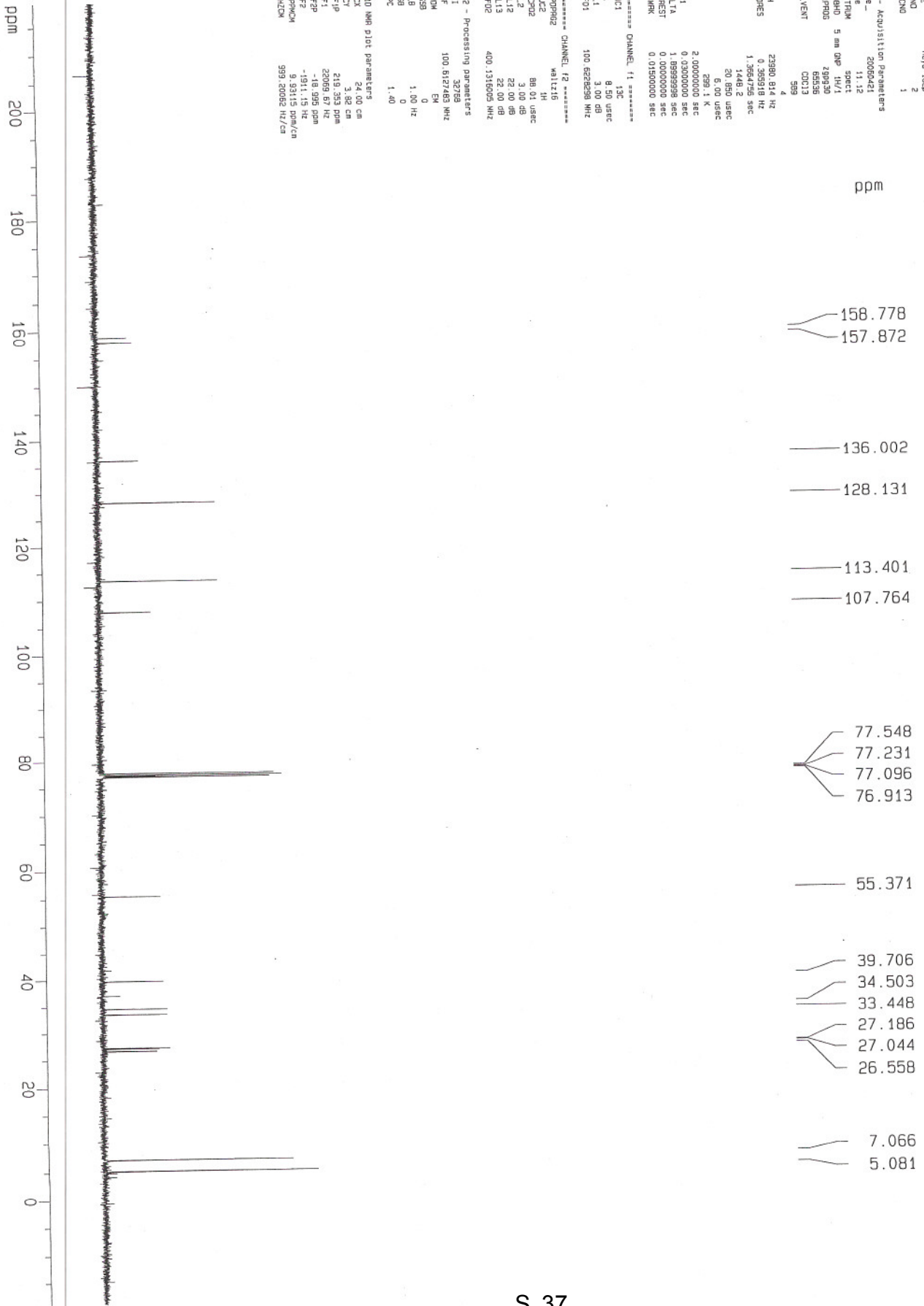
F2 - Acquisition Parameters
 Date_ 20060201
 Time 11:12
 INSTRUM spect
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 589
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.3659118 Hz
 AQ 1.3654756 sec
 RG 1448.2
 DM 20.850 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.0000000 sec
 d11 0.2000000 sec
 DELTA 1.8989998 sec
 WDELTA 0.0000000 sec
 MCHRG 0.0150000 sec

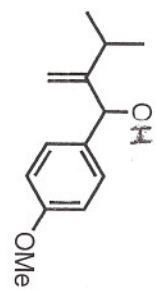
***** CHANNEL f1 *****
 NUC1 13C
 P1 8.50 usec
 PL1 3.00 dB
 SF01 100.6228298 MHz

***** CHANNEL f2 *****
 GRPFO2 waitf2
 NUC2 13C
 P2 8.50 usec
 PL2 3.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SF02 400.1316095 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127483 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CV 24.00 cm
 CY 3.82 cm
 FIP 219.353 ppm
 F1 22069.67 Hz
 F2P -18.995 ppm
 F2 -1911.15 Hz
 PPMCKM 9.93115 ppm/cm
 KZCM 999.20062 Hz/cm





Apdt

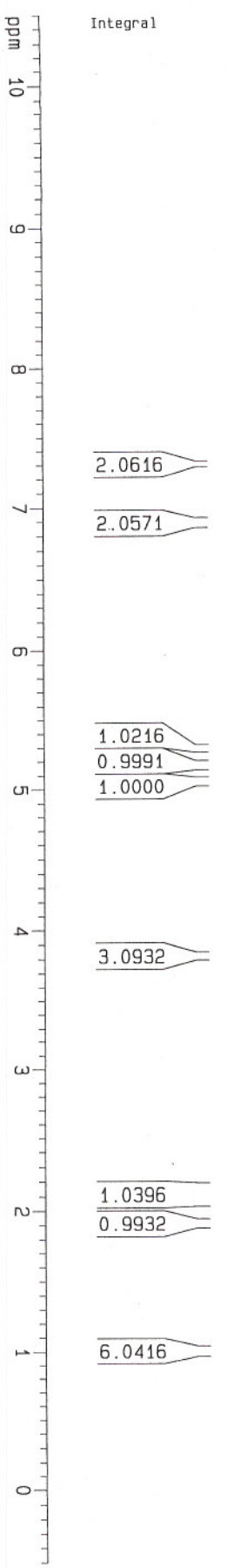
Current Data Parameters
 NAME Hcy9-244de-A
 EXPNO 1
 PROCNO 1

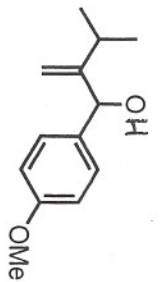
F2 - Acquisition Parameters
 Date_ 20060808
 Time 11:57
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TO 69598
 SOLVENT CDCl3
 NS 5
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.5964243 sec
 RG 287.4
 DE 80.400 usec
 QE 8.00 usec
 IE 300.0 K
 OT 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 KW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR list parameters
 CH 24.00 cm
 F1 14.500 ppm
 F2 4201.39 Hz
 F3 -90.07 Hz
 PPMCH 0.48693 ppm/cm
 HZCH 183.38281 Hz/cm





Current Data Parameters
 NAME hcy8-2440e-A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090809
 Time 12.02
 TMRNUC 13C
 PULPROG 5mm BIRD B1-1
 FIDRES 7000.90
 TD 65535
 SOLVENT CDCl3
 NS 74
 DS 4
 SFO1 100.6237989 MHz

159.436
 158.081
 134.775
 128.519
 113.998
 107.290

77.551
 77.234
 76.916
 76.281

55.431

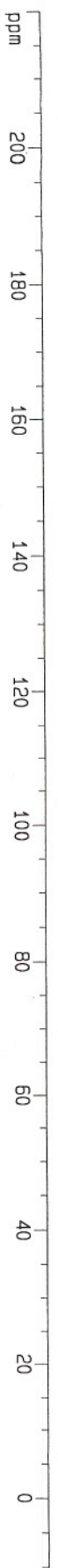
30.403
 23.371
 22.474

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.25 usec
 PL1 3.00 dB
 SFO1 100.6237989 MHz

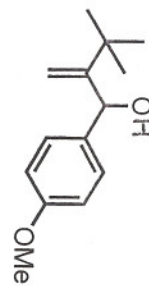
===== CHANNEL f2 =====
 CPDPRG2 MSLT216
 NUC2 1H
 P1 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.617484 MHz
 KHZ EN
 MVM EN
 SSB 0
 LB 1.00 Hz
 UB 0
 PC 1.40

10 NMR plot parameters
 CX 24.00 cm
 FIP 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 FZ -1006.13 Hz
 FREQM 9.58333 ppm/cm
 HDN 984.20544 Hz/cm



TES deprot Aplt



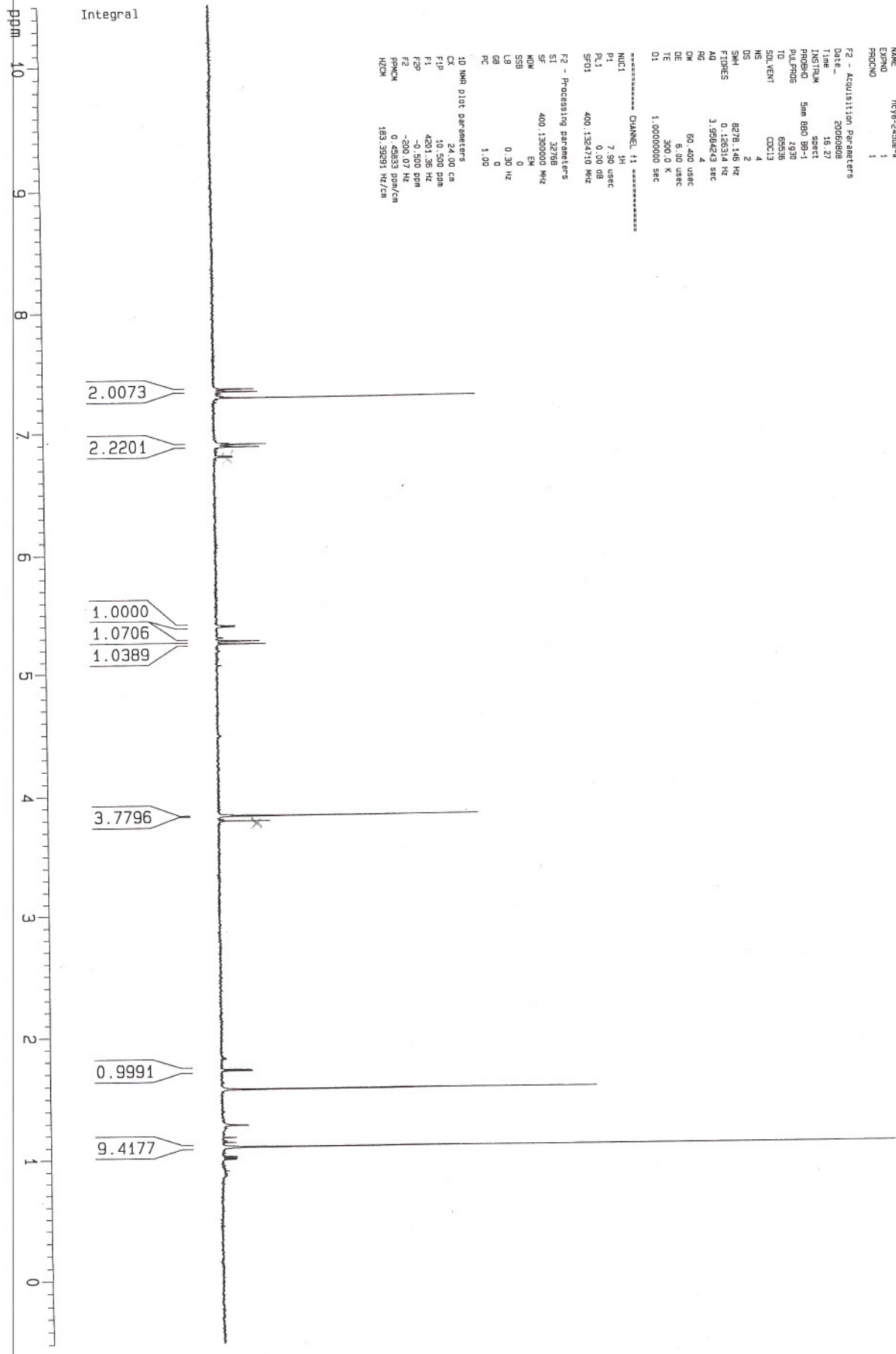
Current Data Parameters
 NAME: NCV6-245de-A
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20060808
 Time: 16:27
 INSTRUM: spect
 PROBRW: 5mm BBO 50mm
 PULPROG: zgpg30
 TO: 65536
 SOLVENT: CDCl3
 NS: 4
 DS: 2
 SFO1: 8276.146 Hz
 FIDRES: 0.126314 Hz
 AQ: 3.9584243 sec
 RG: 4
 DM: 60.400 usec
 DE: 6.00 usec
 TE: 300.0 K
 O1: 1.00000000 sec

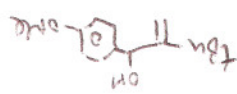
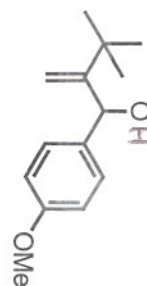
***** CHANNEL f1 *****
 NUC1: 1H
 P1: 7.50 usec
 PL1: 0.00 dB
 SFO1: 400.1324710 MHz

F2 - Processing parameters
 SI: 32768
 SF: 400.1300000 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 24.00 cm
 FIP: 10.500 ppm
 FI: 4204.36 Hz
 FQP: -0.500 ppm
 F2: -200.07 Hz
 PPMCK: 0.42653 ppm/cm
 HZCK: 153.32931 Hz/cm



TES depot Apdt



Current Data Parameters
 NAME: NoyB-265e-A
 EXPNO: 22
 PROCNO: 1

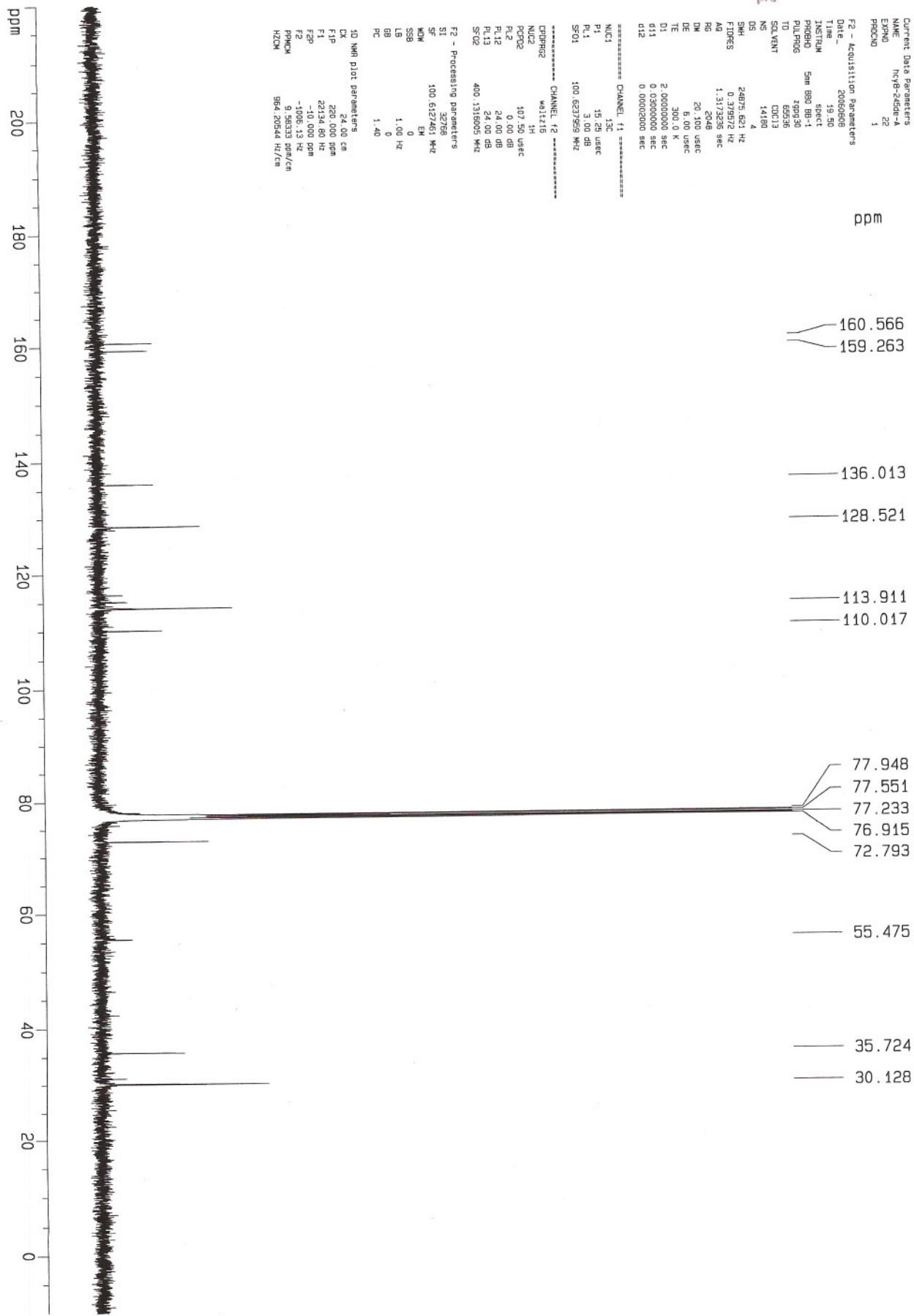
F2 - Acquisition Parameters
 Date_: 20060608
 Time: 19.50
 INSTRUM: spect
 PRRBHD: 5mm BB0 BB-1
 PULPROG: zgpg30
 TO: 65536
 SOLVENT: CDCl3
 NS: 14180
 DS: 4
 SMH: 24875.621 Hz
 FIDRES: 0.379392 Hz
 AQ: 1.317858 sec
 RG: 1.317858
 DW: 20.100 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 2.00000000 sec
 d11: 0.03000000 sec
 d12: 0.00002000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 15.29 usec
 PL1: 3.00 dB
 SF01: 100.627959 MHz

===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 P2: 107.50 usec
 PL2: 0.00 dB
 PL12: 24.00 dB
 PL13: 24.00 dB
 SF02: 400.1316005 MHz

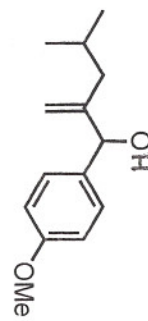
F2 - Processing parameters
 SI: 32768
 SF: 100.6127461 MHz
 MDW: EM
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

1D NMR plot parameters
 CK: 24.00 cm
 FK: 250.00 rpm
 F1P: 228.000 Hz
 F2P: 110.000 rpm
 F2: -1006.13 Hz
 RPWCM: 9.56333 rpm/cm
 NZCM: 964.20544 Hz/cm



- 160.566
- 159.263
- 136.013
- 128.521
- 113.911
- 110.017
- 77.948
- 77.551
- 77.233
- 76.915
- 72.793
- 55.475
- 35.724
- 30.128

db TES de A-pdt



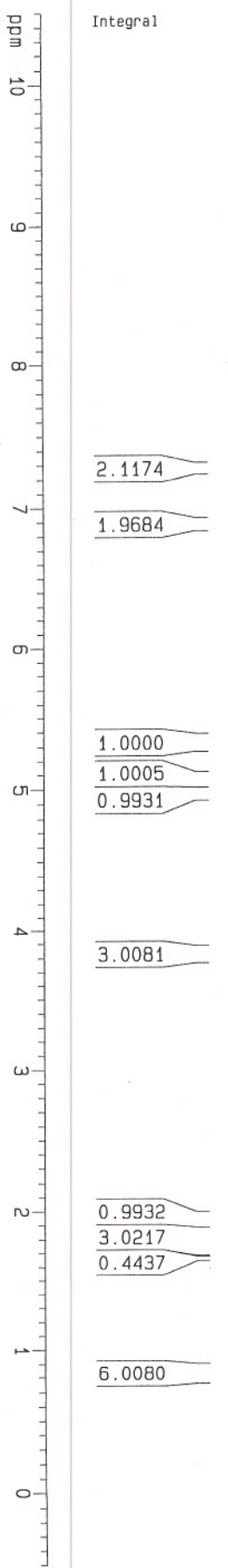
Current Data Parameters
 Name: h2y8230308-4
 Exp: 1
 PROCNO: 1

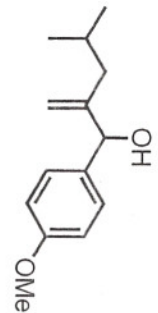
F2 - Acquisition Parameters
 Date_: 20060728
 Time: 17.54
 INSTRUM: spect
 PROBHD: 5 mm QNP 1H/1
 PULPROG: zg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 5
 DS: 1
 SWH: 8278.146 Hz
 FIDRES: 0.186314 Hz
 AQ: 3.594843 sec
 RG: 228.1
 DM: 80.400 usec
 DE: 254.9
 TE: 304.2
 D1: 1.00000000 sec
 MDC18: 0.00000000 sec
 MCK8K: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 9.88 usec
 PL1: 3.00 dB
 SF01: 400.1324710 MHz

F2 - Processing parameters
 SI: 32768
 SF: 400.1300054 MHz
 WM: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

10 NMR plot parameters
 CX: 24.00 cm
 CY: 6.88 cm
 FIP: 10.500 ppm
 F1: 4201.36 Hz
 F2: -200.07 Hz
 PPM1H: 0.45833 ppm/cm
 N2DM: 183.38231 Hz/cm





Current Data Parameters
 NAME ncv8-258bde-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 2006/07/28
 Time 17:39
 INSTRUM spect
 PROBR0 5 mm QNP 1H/1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 187
 DS 4

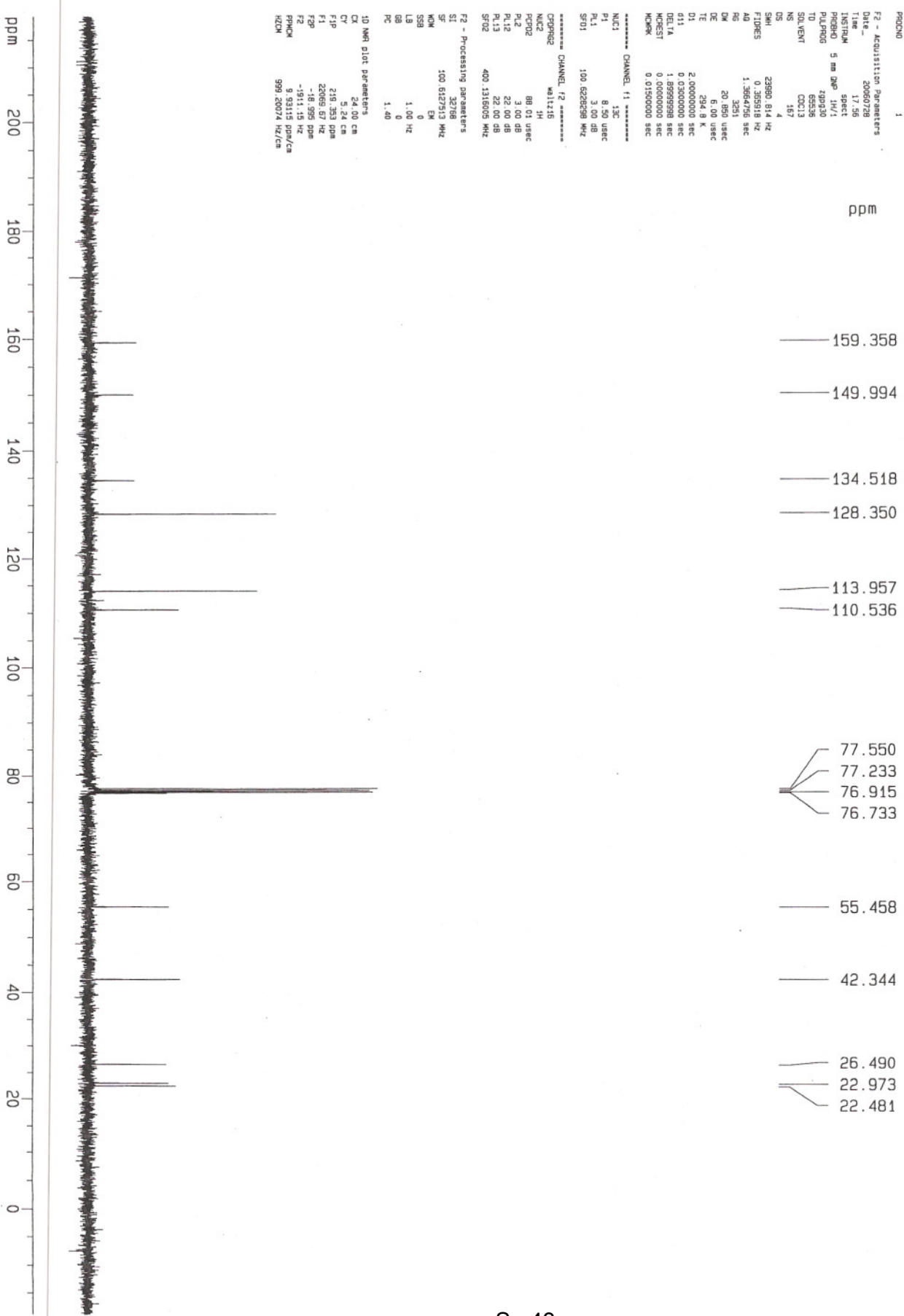
SMH 22980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 3251
 DW 20.950 usec
 DE 6.00 usec
 TE 294.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.2000000 sec
 MISCST 0.0000000 sec
 KICK 0.0150000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 8.50 usec
 PL1 3.00 dB
 SFO1 100.6282698 MHz

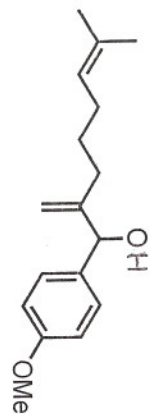
***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 88.01 usec
 PL2 3.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SFO2 400.1463003 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127513 MHz
 KW 64
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR1 plot parameters
 CX 24.00 cm
 CY 5.24 cm
 F1P 219.353 ppm
 F1 25089.57 Hz
 F2 -191.45 ppm
 F2 9.53115 ppm/cm
 PPMCK 999.20074 Hz/cm
 KZCM



diene TES deprotected A.pdt



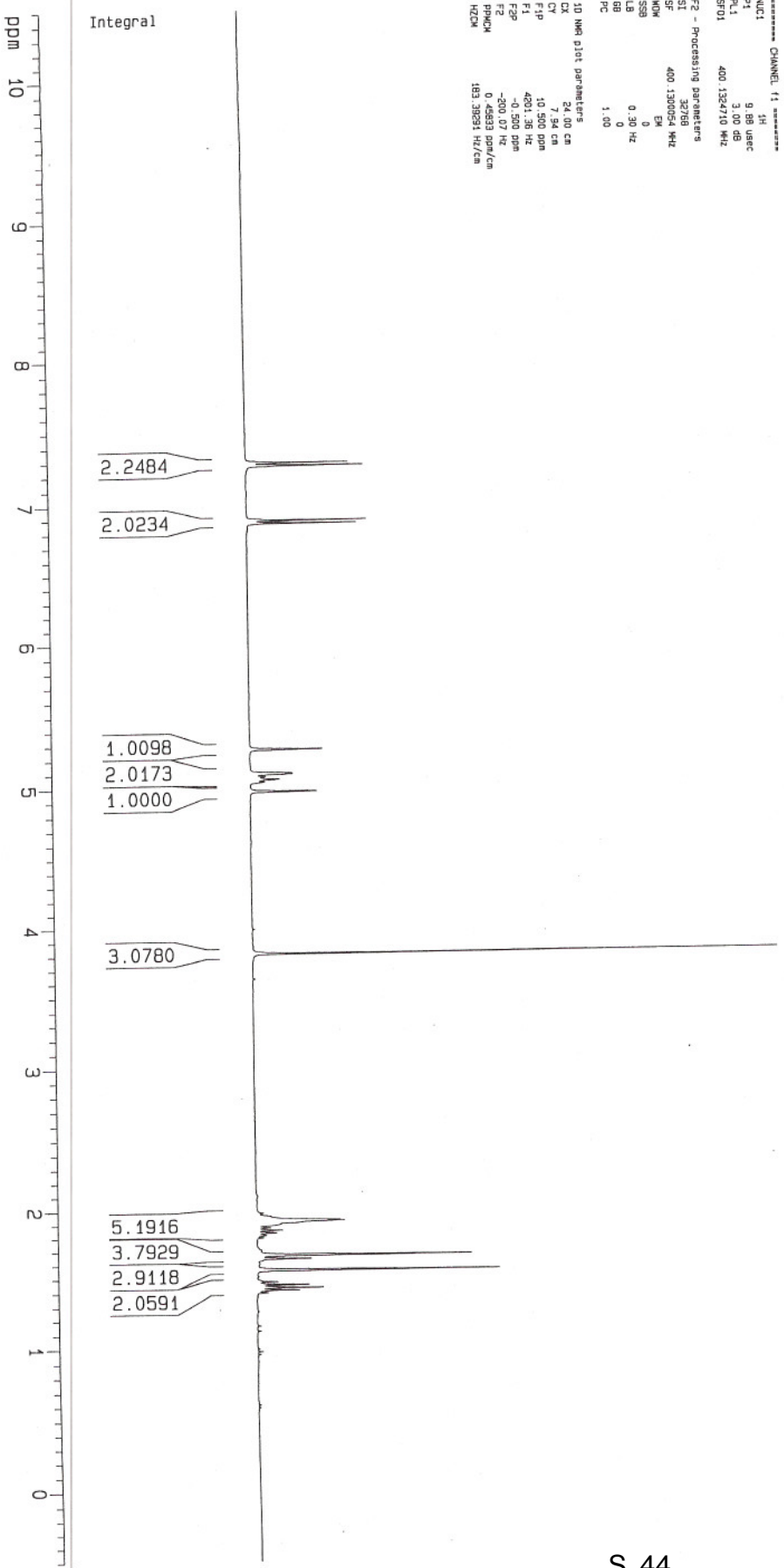
Current Data Parameters
 NAME hcv8-2500e-d1-10
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060727
 Time 18:47
 INSTRM spect
 PROBO 5 mm QNP 1H/1
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 4
 DS 1
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.3594643 sec
 RG 628
 HS 54.000 usec
 HM 5.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 D11 0.00000000 sec
 ACQRES 0.01500000 sec
 KICKER

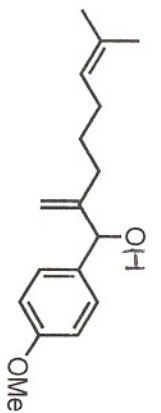
***** CHANNEL f1 *****
 NUC1 1H
 P1 9.88 usec
 PL1 3.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 400.130054 MHz
 SF 400.1324710 MHz
 WHW EM
 SSB 0
 LB 0.30 Hz
 BB 0
 PC 1.00

10 NMR list parameters
 CX 24.00 cm
 CY 7.94 cm
 F1P 10.500 ppm
 F1 4201.36 Hz
 F2P -0.500 ppm
 F2 -20.7 Hz
 PWDW 0.00000000 sec
 PWDW 149.38931 Hz/cm
 HZCM



diene TES deprotected A.pdt



Current Data Parameters
 NAME hc98-2566-d1-A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060727
 Time 18:51
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TO 65935
 SOLVENT CDCl3
 NS 123
 DS 4
 SM 23980.014 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664795 sec
 RG 3231
 DM 20.000 usec
 DE 28.00 usec
 TE 28.00 K
 TD 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999999 sec
 KCREST 0.0000000 sec
 MCPXK 0.0150000 sec

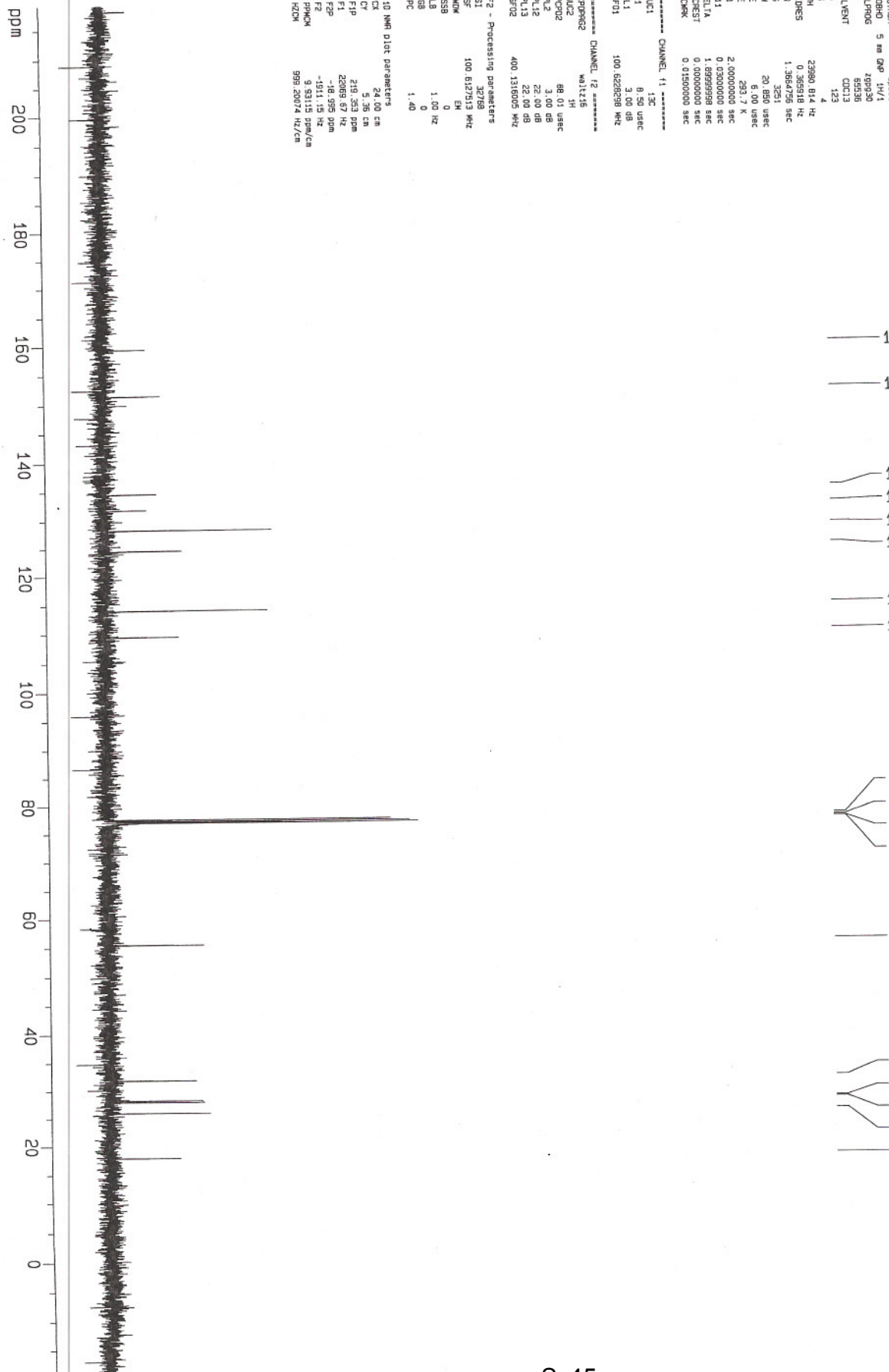
***** CHANNEL f1 *****
 NUC1 13C
 P1 6.50 usec
 PL1 3.00 dB
 SFO1 100.6262598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 88.01 usec
 PL2 3.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SFO2 400.1316005 MHz

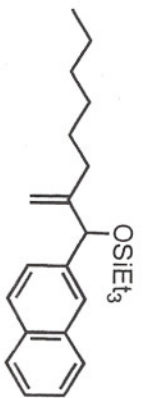
F2 - Processing parameters
 SI 32768
 SF 100.6127513 MHz
 MW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR Plot Parameters
 CX 24.00 cm
 CY 5.36 cm
 FIP 219.353 ppm
 FI 22089.87 Hz
 F2P -18.995 ppm
 F2 -1911.15 Hz
 PPMCM 9.53115 ppm/cm
 HZCM 999.20074 Hz/cm

- 159.334
- 151.385
- 134.548
- 131.854
- 128.211
- 124.530
- 113.946
- 109.356
- 77.546
- 77.229
- 76.983
- 76.911
- 55.465
- 31.850
- 28.183
- 27.943
- 25.906
- 17.891



2-NpCHO A-pdt



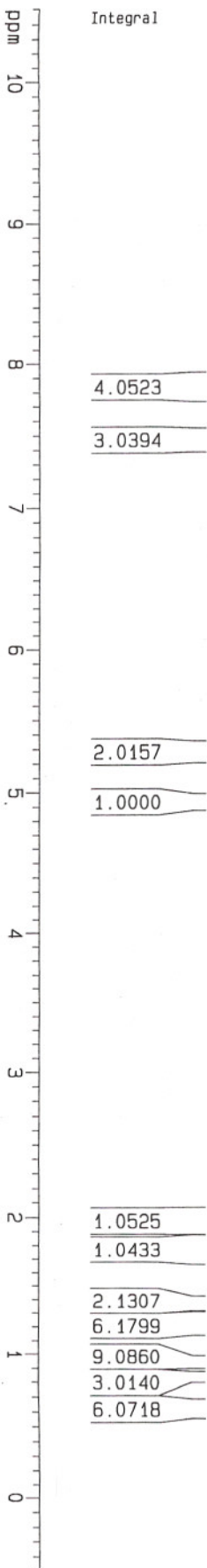
Current Data Parameters
 NAME hcr18-2340-Np
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060725
 Time 20.04
 INSTRUM spect
 PROBR0 5 mm QNP 1H/1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 1
 SWH 8278.146 Hz
 FIDRES 0.9534 Hz
 AQ 3.0964243 sec
 RG 143.7
 DM 50.400 usac
 DE 6.00 usac
 TE 293.7 K
 D1 1.00000000 sec
 MCPRST 0.00000000 sec
 MCNMR 0.01500000 sec

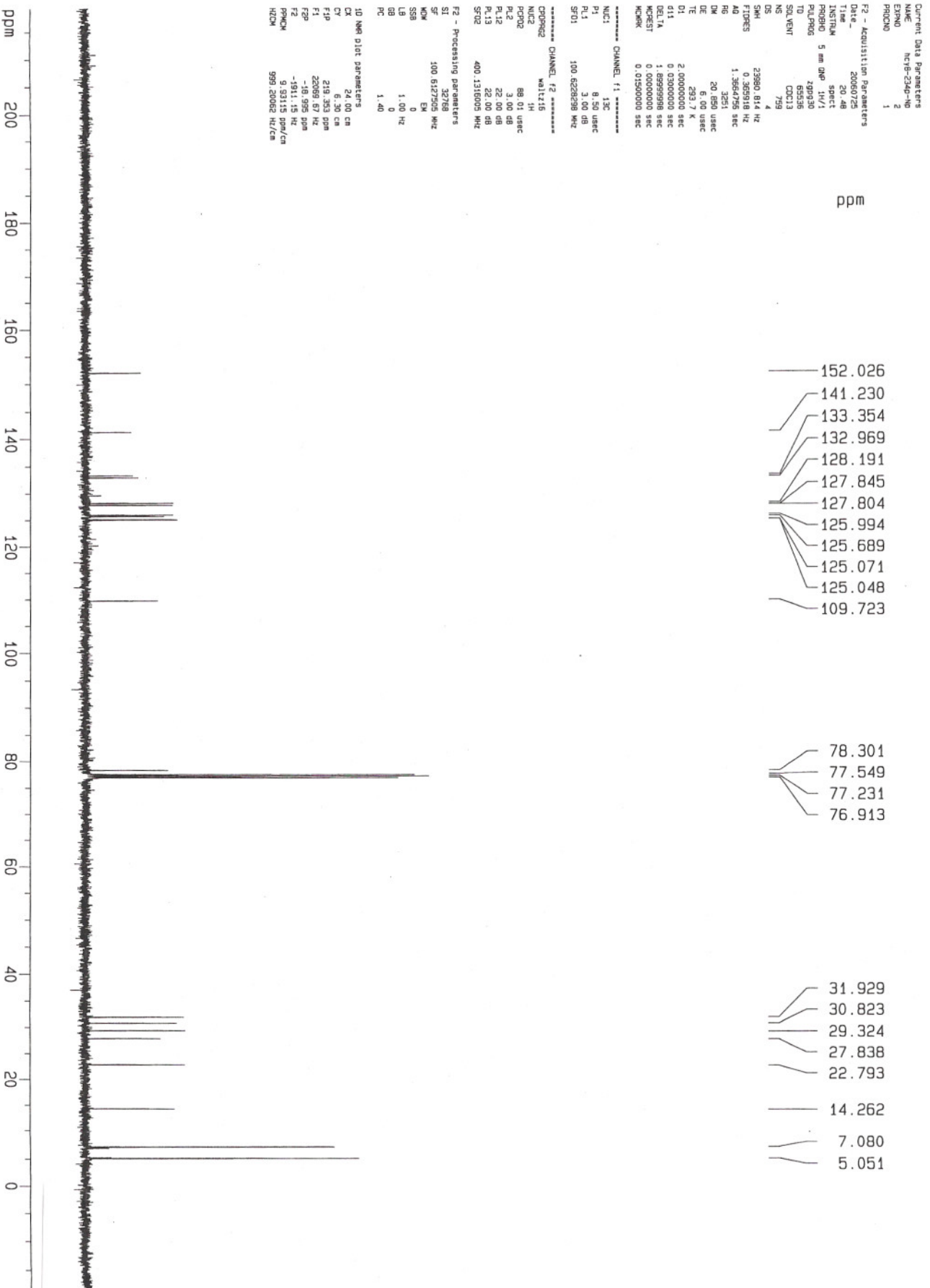
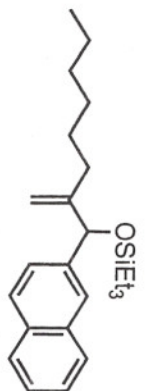
***** CHANNEL f1 *****
 NUC1 1H
 P1 9.88 usac
 PL1 3.00 dB
 SF01 400.1324710 MHz

F2 - Processing Parameters
 SI 32768
 SF 400.1300024 MHz
 WHW 64
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

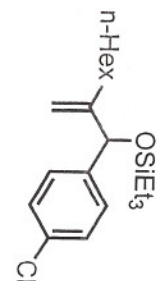
10 NMR plot parameters
 CX 24.00 cm
 CY 6.41 cm
 F1P 10.500 DPM
 F1 4201.36 Hz
 F2P -0.500 DPM
 F2 -200.07 Hz
 PPMCN 0.45833 DPM/cm
 HZCN 183.39291 HZ/cm



2-NpCHO A-Pdt



C1-Apdt

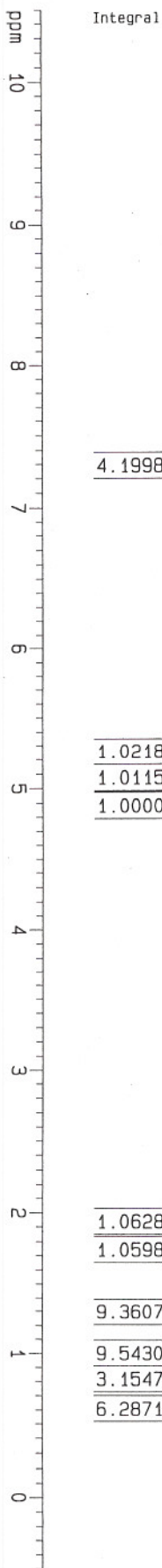


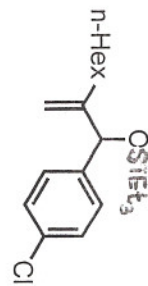
Current Data Parameters
NAME: NSP-2401-Apdt
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20060807
Time: 16.56
INSTRUM: spect
PROBHD: 5mm BBO BB-1
PULPROG: zg30
TO: 65536
SOLVENT: CDCl₃
NS: 6
DS: 2
SMH: 8278.146 Hz
FIDRES: 0.128314 Hz
AQ: 3.926243 sec
RG: 640
WDW: EM
SSB: 0
GB: 0
PC: 6.00 usec
TE: 300.0 K
D1: 1.00000000 sec

***** CHANNEL f1 *****
NUC1: 1H
P1: 7.50 usec
PL1: 0.00 dB
SFO1: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1300000 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
ID: NMR plot parameters
CY: 24.00 cm
FIP: 10.500 ppm
F1: 4201.36 Hz
F2: -0.500 ppm
F2: -200.07 Hz
PPMCK: 0.48833 ppm/cm
HZCM: 183.39291 Hz/cm



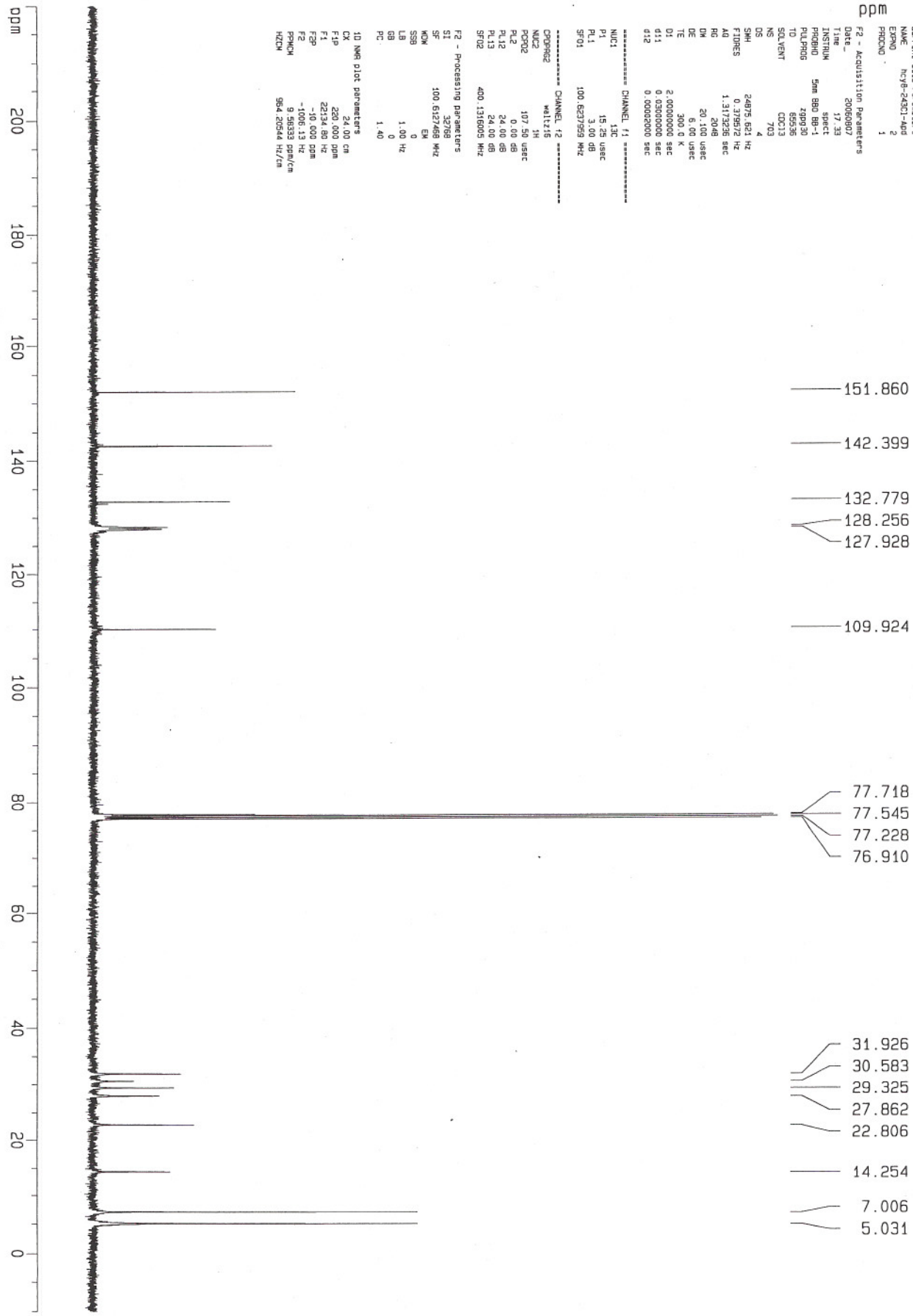


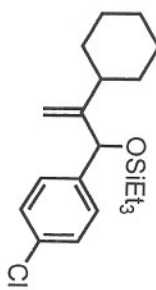
Current Data Parameters
 NAME NCVS-243C1-4ad
 EXPNO 2
 PRICHO 1
 F2 - Acquisition Parameters
 Date_ 20060907
 Time 17.33
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zgpg30
 TO 65535
 SOLVENT CDCl₃
 NS 762
 DS 4
 SWH 24875.621 Hz
 FIDRES 0.378572 Hz
 AQ 1.317235 sec
 RG 2048
 DM 20.100 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 15.25 usec
 PL1 3.00 dB
 SF01 100.627959 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127469 MHz
 KW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 ID NMR plot parameters
 SI 32768
 SF 100.6127469 MHz
 F1 23134.80 Hz
 F2 -10.000 ppm
 F2 -1006.13 Hz
 PSICH 9.8533 ppm/cm
 HZCM 564.20544 Hz/cm





Cl-4pdt

W14-R-2421-10 10 10 10 10

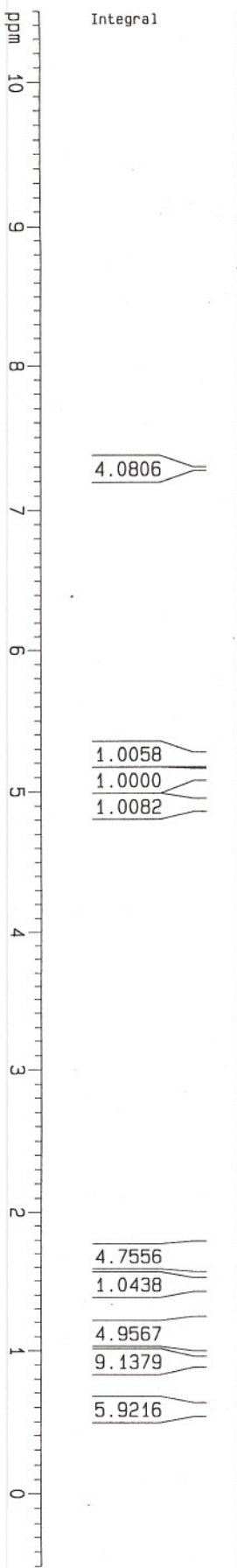
Current Data Parameters
 NAME hc19-2421-10d
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060907
 Time 10:55
 INSTRUM spect
 PROBRD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 5
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.594243 sec
 SFO1 161.3
 F2 60.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec

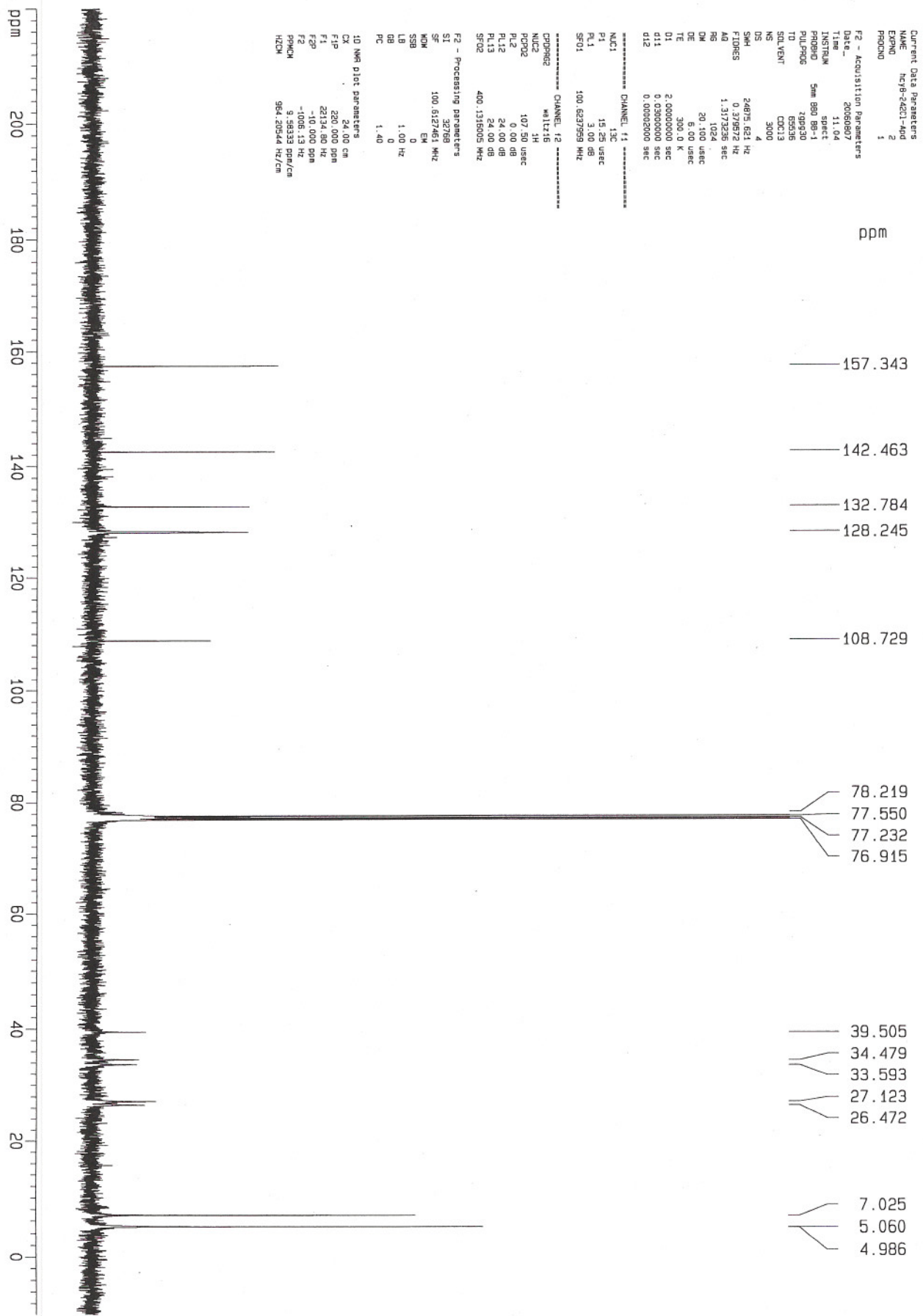
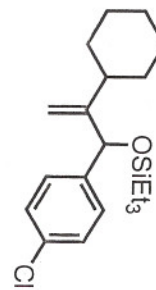
***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CK 24.00 cm
 F1P 10.500 ppm
 F2 4201.30 Hz
 F2P -300.07 Hz
 SFOCK 0.48833 ppm/cm
 HZCK 183.35261 Hz/cm



C1-Addt



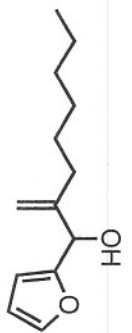
Current Data Parameters
 NAME hc19-24ct1-Add
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20060907
 Time 11.04
 INSTRUM spect
 PROBRF 5mm BBO BB-1
 PULPROG zgpg30
 PD 65235
 PC 3000
 SOLVENT CDCl3
 NS 4
 DS 4
 SFO1 24875.621 Hz
 FIDRES 0.376572 Hz
 AQ 1.317236 sec
 RG 1024
 DM 20.100 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.25 usec
 PL1 3.00 dB
 SF01 100.6273599 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127461 MHz
 MVM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

LD NMR Plot parameters
 CA 32768
 CB 32768
 FIP 24.000 cm
 FI 23134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 HPCMH 9.58333 ppm/cm
 HZCM 964.20544 Hz/cm



TES deprot 2-fur Apdt

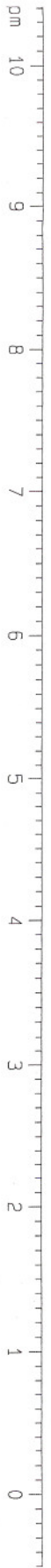
Current Data Parameters
 NAME hc98-5506a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080822
 Time 10.34
 INSTRUM spect
 PULPROG sm BRD BR-1
 PILEPUS 2930
 TO 65536
 SELVENT CUC13
 NS 2
 DS 2
 SFO1 400.1324710 MHz
 FIDRES 0.125314 Hz
 AQ 3.9584243 sec
 RG 181
 DW 50.400 usec
 DE 5.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 EN 64
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 24.00 cm
 F1P 10.500 ppm
 F1 4201.36 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PRGCM 0.45833 ppm/cm
 HZCM 83.39291 Hz/cm



0.9802

1.0000
0.9737

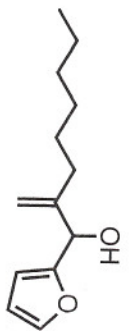
1.0100
0.9710
0.9912

3.0483

2.1242
6.2567

3.0689

TES deprot 2-fur Apdt



Current Data Parameters
 NAME ncy8-2506A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060822
 Time 10.48
 INSTRUM spect
 PROBRD 5mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 632
 DS 4
 SWH 24875.621 Hz
 FIDRES 0.370672 Hz
 AQ 1.317285 sec
 RG 1625.5
 DM 20.100 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 15.28 usec
 PL1 3.00 dB
 SF01 100.6273959 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 14.00 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 S1 32756
 SF 100.6127499 MHz
 WDM EH
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR tick parameters
 C1 24.00 cm
 F1 24.00 ppm
 F2 21831.74 Hz
 F2P -5.000 ppm
 F2 -503.06 Hz
 PPM2CM 9.16667 ppm/cm
 HQCN 922.2851 Hz/cm

