Nickel-Catalyzed Coupling of Alkenes, Aldehydes, and Silyl Triflates

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Supporting Information

Experimental Procedures, Analytical and Spectroscopic Data for Compounds **1a** – **5b**.
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^{1}H and ^{13}C NMR spectra for compounds **1a** – **5b**.
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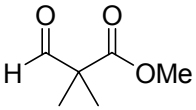
General Information.

Unless otherwise noted, all reactions were performed under an oxygen-free atmosphere of nitrogen or argon with rigid exclusion of moisture from reagents and glassware. Tetrahydrofuran was distilled from a blue solution of sodium benzophenone ketyl. Dichloromethane and toluene was distilled from calcium hydride. Aromatic aldehydes were purchased from Aldrich Chemical Co. and used as received. Other aldehydes were distilled and saturated with nitrogen before use. Bis(cyclooctadienyl)nickel(0) ($Ni(cod)_2$) and tris-(*o*-methoxyphenyl)-phosphine and triphenylphosphine were purchased from Strem Chemicals, Inc., stored under nitrogen atmosphere and used without further purification. Ethylene was purchased from BOC Gases and used as received. 1-octene was purchased from Alfa Aesar and used as received. All other alkenes were purchased from Aldrich Chemical Co. and used as received. Dicyclohexyl-phenylphosphine and ethyldiphenylphosphinite were purchased from Aldrich Chemical Co., stored under nitrogen atmosphere and used without further purification. Triethylsilyltrifluoro-methanesulfonate (TESOTf) and trimethylsilyltrifluoromethansulfonate (TMSOTf) were purchased from Aldrich Chemical Co. and were distilled over calcium hydride before use. Tert-butyldimethysilyltrifluoromethanesulfonate (TBSOTf) was purchased from Alfa Aesar and was distilled over calcium hydride before use.

Analytical thin layer chromatography (TLC) was performed using EM Science silica gel 60 F_{254} 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 Varian 300 MHz, Varian 500 MHz or Bruker 400 MHz spectrometers in $CDCl_3$ or C_6D_6, unless otherwise noted. Chemical shifts in ^1H NMR spectra are reported in parts per million (ppm) on the δ scale from an internal standard of residual chloroform (7.27 ppm) or residual benzene (7.16 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad), coupling constant in hertz (Hz), and integration. 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. Chiral GC analysis was performed on a Varian CP-3800 gas chromatograph fitted with Chiraldex B-PH, B-DA, and G-TA capillary columns. Chiral HPLC analysis was performed on a Hewlett-Packard 1100 chromatograph equipped with a variable wavelength detector and Chiralcel OD or OD-H columns.

Preparation of 2,2-dimethyl-3-oxo-propionic acid methyl ester



2,2-dimethyl-3-oxo-propionic acid methyl ester. 3-Hydroxy-2,2-dimethyl-propionic acid methyl ester (15 g, 113 mmol) in 200 mL dichloromethane was cooled to 0°C. Pyridinium chlorochromate (43 g, 200 mmol) was added. The mixture was slowly warmed to room temperature and stirred 24 h. The crude in dichloromethane was filtered through silica gel. Celite was added to the remaining black viscous oil from the reaction mixture until the viscous oil is all absorbed to the celite. Dichloromethane was added to this slurry and the dichloromethane solution was filtered through silica gel. Dichloromethane was removed at reduced pressure (80 Torr) to give a pale yellow crude oil. Fractional distillation removed residue dichloromethane and obtained 2,2-dimethyl-3-oxo-propionic acid methyl ester as a colorless oil (7 g, 48% yield).

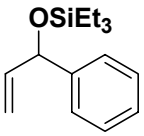
^{1}H NMR (300 MHz, $CDCl_3$, δ): 9.60 (s, 1H); 3.70 (s, 3H); 1.29 (s, 6H).
^{13}C NMR (75 MHz, $CDCl_3$, δ): 199.1, 173.2, 53.9, 52.6, 19.7.
IR (NaCl, thin film): 2988, 2958, 1726, 1468, 1278, 1151, 866.

Nickel-catalyzed couplings of ethylene and aldehydes (1a, 1b, 1c, 1d, 1i, 1j, 1l, 1m, 1n).

General procedure 1. A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.1 mmol, 20 mol%) and tris-*o*-methoxyphenylphosphine (70.5 mg, 0.2 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 15 min at room temperature. The reaction mixture was purged with ethylene for 1 min to remove argon, taken care not to introduce oxygen. The ethylene atmosphere was maintained with an ethylene balloon. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. Aldehyde (0.5 mmol, 100 mol%, as specified) was added. Silyltriflate (0.875 mmol, 175 mol%, as specified) was added. The mixture was stirred at room temperature for 3 – 18 h, as judged by the TLC of the reaction mixture. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and the crude mixture was diluted in hexane. Purification via flash chromatography on silica afforded the coupling product.

Nickel-catalyzed couplings of ethylene and aldehydes (1e, 1f, 1g, 1h, 1k).

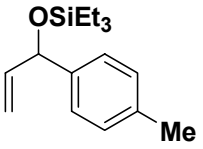
General procedure 2. A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.1 mmol, 20 mol%), tris-*o*-methoxyphenylphosphine (70.5 mg, 0.2 mmol, 40 mol%) and aldehyde (0.5 mmol, 100 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 15 min at room temperature. The reaction mixture was purged with ethylene for 1 min to remove argon, taken care not to introduce oxygen. The ethylene atmosphere was maintained with an ethylene balloon. Next triethylamine (418 μL, 3 mmol, 600 mol%) was added. Silyltriflate (0.875 mmol, 175 mol%, as specified) was added. The mixture was stirred at room temperature for 3 – 18 h, as judged by TLC of the reaction mixture. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and the crude mixture was diluted in hexane. Purification via flash chromatography on silica afforded the coupling product.



(1a)

The reaction of ethylene and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1a** in 82% isolated yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.32 – 7.45 (m, 4H); 7.29 (t, J = 7.0 Hz, 1H); 6.01 (ddd, J = 6.0, 10.2, 16.9 Hz, 1H); 5.34 (dt, J = 1.5, 16.9 Hz, 1H); 5.25 (d, J = 5.9 Hz, 1H); 5.13 (dt, J = 1.5, 10.2 Hz, 1H); 0.99 (t, J = 8.0 Hz, 9H); 0.66 (dq, J = 1.8, 7.8 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 143.9, 141.8, 128.4, 127.3, 126.2, 113.7, 75.9, 7.0, 5.1.
IR (NaCl, thin film): 2956, 2877, 1640, 1454, 1240, 1065, 744, 699.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{15}H_{24}OSiNa$, 271.149; found, 271.150.



(1b)

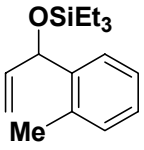
The reaction of ethylene and *p*-tolualdehyde (59 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1b** in 88% isolated yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.27 (d, *J* = 8.0, 2H); 7.16 (d, *J* = 8.0 Hz, 2H); 5.97 (ddd, *J* = 5.9, 10.2, 16.9 Hz, 1H); 5.30 (dt, *J* = 1.5, 17.0 Hz, 1H); 5.17 (d, *J* = 5.9 Hz, 1H); 5.09 (dt, *J* = 1.3, 10.2 Hz, 1H); 2.37 (s, 3H); 0.97 (t, *J* = 7.9 Hz, 9H); 0.65 (dq, *J* = 1.9, 7.5 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 142.1, 141.1, 136.8, 129.1, 126.2, 113.4, 75.8, 21.3, 7.0, 5.2.

IR (NaCl, thin film): 2955, 2877, 1640, 1513, 1458, 1415, 1079, 1007, 844.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{26}OSiNa$, 285.165; found, 285.165.



(1c)

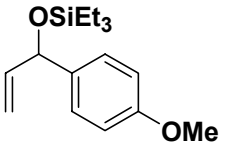
The reaction of ethylene and *o*-tolualdehyde (58 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1c** in 93% isolated yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.50 (d, *J* = 7.0, 1H); 7.11 – 7.24 (m, 3H); 5.93 (ddd, *J* = 5.7, 10.2, 17.0 Hz, 1H); 5.36 (d, *J* = 5.6 Hz, 1H); 5.22 (dt, *J* = 1.6, 17.1 Hz, 1H); 5.08 (dt, *J* = 1.5, 10.2 Hz, 1H); 2.34 (s, 3H); 0.95 (t, *J* = 8.0 Hz, 9H); 0.61 (dq, *J* = 2.8, 7.5 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 141.9, 140.7, 134.4, 130.3, 127.1, 126.5, 126.3, 113.7, 73.1, 19.4, 7.0, 5.1.

IR (NaCl, thin film): 2955, 2877, 1639, 1461, 1066, 1007, 744.

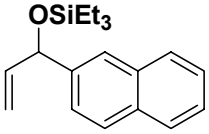
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{26}OSiNa$, 285.165; found, 285.165.



(1d)

The reaction of ethylene and *p*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1d** in 95% isolated yield as a colorless oil.

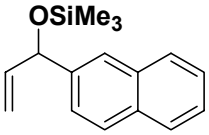
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30 (d, *J* = 8.7 Hz, 2H); 6.90 (d, *J* = 8.7 Hz, 2H); 5.97 (ddd, *J* = 5.9, 10.2, 16.9 Hz, 1H); 5.29 (dt, *J* = 1.4, 17.0 Hz, 1H); 5.16 (d, *J* = 5.9 Hz, 1H); 5.10 (dt, *J* = 1.4, 10.2 Hz, 1H); 3.83 (s, 3H); 0.96 (t, *J* = 7.9 Hz, 9H); 0.63 (dq, *J* = 1.8, 7.5 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 158.9, 142.0, 136.2, 127.4, 113.7, 113.4, 75.4, 55.4, 7.0, 5.1.
IR (NaCl, thin film): 2955, 2877, 1639, 1511, 1464, 1246, 1037, 744.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{26}O_2SiNa$, 301.159; found, 301.159.



(1e)

The reaction of ethylene and 2-naphthaldehyde (78.1 mg, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 2 above, afforded **1e** in 95% isolated yield as a colorless oil.

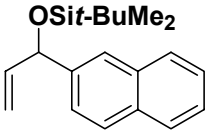
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.82 – 7.92 (m, 4H); 7.48 – 7.55 (m, 3H); 6.07 (ddd, *J* = 6.2, 10.2, 15.8 Hz, 1H); 5.35 – 5.45 (m, 2H); 5.17 (dt, *J* = 1.3, 10.1 Hz, 1H); 1.00 (t, *J* = 7.8 Hz, 9H); 0.68 (dq, *J* = 2.5, 7.5 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 141.7, 141.4, 133.5, 133.0, 128.2, 128.1, 127.7, 126.1, 125.8, 124.8, 124.6, 114.0, 76.0, 7.0, 5.1.
IR (NaCl, thin film): 2955, 2876, 1640, 1458, 1239, 1006, 743.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{26}OSiNa$, 321.165; found, 321.164.



(1f)

The reaction of ethylene and 2-naphthaldehyde (78.1 mg, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenyl- phosphine and TMSOTf (158 μL, 0.875 mmol), triethylamine in toluene following the general procedure 2 above, afforded **1f** in 60% isolated yield as a colorless oil.

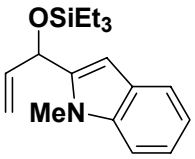
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.80 – 7.90 (m, 4H); 7.45 – 7.54 (m, 3H); 6.06 (ddd, *J* = 5.6, 10.2, 17.4 Hz, 1H); 5.30 (dt, *J* = 1.5, 17.3 Hz, 1H); 5.37 (bs, 1H); 5.17 (dt, *J* = 1.4, 10.2 Hz, 1H); 0.18 (s, 9H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 141.4, 141.0, 133.5, 133.0, 128.19, 128.18, 127.9, 126.2, 125.9, 124.9, 124.8, 114.4, 76.1, 0.4.
IR (NaCl, thin film): 2958, 1640, 1251, 1077, 841.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{20}OSiNa$, 279.118; found, 279.119.



(1g)

The reaction of ethylene and 2-naphthaldehyde (78.1 mg, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxy- phenylphosphine and TBSOTf (201 μL, 0.875 mmol), triethylamine in toluene following the general procedure 2 above, afforded **1g** in 67% isolated yield as a colorless oil.

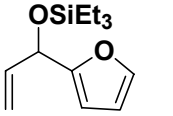
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.80 – 7.92 (m, 4H); 7.45 – 7.55 (m, 3H); 6.04 (ddd, *J* = 5.8, 10.2, 16.8 Hz, 1H); 5.39 (dt, *J* = 1.5, 17.0 Hz, 1H); 5.38 (s, 1H); 5.14 (dt, *J* = 1.5, 10.2 Hz, 1H); 0.99 (s, 9H); 0.16 (s, 3H); 0.06 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 141.8, 141.4, 133.5, 133.0, 128.2, 128.1, 127.9, 126.1, 125.8, 124.8, 124.6, 113.8, 76.2, 26.1, 18.6, -4.4, -4.6.
IR (NaCl, thin film): 2956, 2857, 1636, 1472, 1252, 1081, 837.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{26}OSiNa$, 321.165; found, 321.164.



(1h)

The reaction of ethylene and 1-methyl-2-indolecarboxaldehyde (79.6 mg, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxy- phenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 2 above, afforded **1h** in 67% isolated yield as a colorless oil.

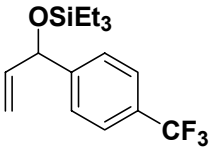
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.63 (d, *J* = 7.8 Hz, 1H); 7.36 (d, *J* = 8.2 Hz, 1H); 7.26 (t, *J* = 8.3 Hz, 1H); 7.14 (t, *J* = 7.9 Hz, 1H); 6.43 (s, 1H); 6.13 (ddd, *J* = 4.5, 10.3, 17.1 Hz, 1H); 5.52 (ddd, *J* = 1.7, 1.7, 4.5 Hz , 1H); 5.39 (ddd, *J* = 1.7, 1.7, 17.1 Hz, 1H); 5.25 (ddd, *J* = 1.7, 1.7, 10.4, 1H); 3.82 (s, 3H); 0.98 (t, *J* = 8.0 Hz, 9H); 0.66 (dq, *J* = 1.4, 8.0 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 140.6, 139.7, 138.5, 127.5, 121.5, 120.8, 119.4, 114.9, 109.1, 100.5, 70.4, 31.0, 7.0, 5.0.
IR (NaCl, thin film): 2955, 2911, 2876, 1911, 1758, 1641, 1469, 1238, 1009, 841, 731.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{18}H_{27}NOSiNa$, 324.178; found, 324.178.



(1i)

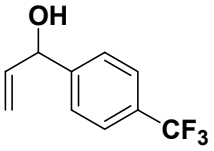
The reaction of ethylene and furan-2-carbaldehyde (41 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1i** in 38% isolated yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.37 (bs, 1H); 6.32 (dd, *J* = 1.9, 3.1 Hz, 1H); 6.22 (d, *J* = 3.2 Hz, 1H); 6.06 (m, 1H); 5.40 (d, *J* = 17.1 Hz, 1H); 5.21 (d, *J* = 7.9 Hz, 2H); 0.95 (t, *J* = 7.9 Hz, 9H); 0.63 (q, *J* = 7.9 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 156.0, 142.1, 138.1, 115.3, 110.4, 106.4. 69.3, 6.9, 4.9.
IR (NaCl, thin film): 2956, 2878, 1646, 1459, 1237, 1010, 733.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{13}H_{22}O_2SiNa$, 261.128; found, 261.129.



(1j)

The reaction of ethylene and 4-(trifluoromethyl)-benzaldehyde (70 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded a mixture of **1j** and triethylsilylethers of pinnacol coupling products. This mixture was subjected to TBAF to isolate 25% of the desilylated **1j** as a colorless oil.



(Desilylated 1j)

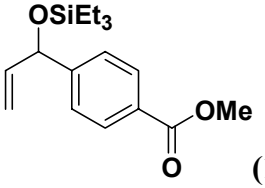
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.62 (d, *J* = 8.2 Hz, 2H); 7.50 (d, *J* = 8.4 Hz, 2H); 6.02 (ddd, *J* = 6.3, 10.3, 16.9 Hz, 1H); 5.38 (ddd, *J* = 1.2, 1.2, 17.0 Hz, 1H); 5.27 (bd, *J* = 7.0 Hz, 1H); 5.25 (ddd, *J* = 1.2, 1.2, 10.3 Hz, 1H); 2.10 (bs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 146.5, 139.8, 130.0 (*J* = 32.3 Hz), 126.7, 125.7, 123.0, 116.4, 75.1.

19 F NMR (376 MHz, $CDCl_3$, δ): -66.8 (s, 3F).

IR (NaCl, thin film): 3342, 1620, 1419, 1328, 1166, 1126, 1068, 931.

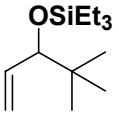
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{10}H_9OF_3Na$, 202.060; found, 202.059.



(1k)

The reaction of ethylene and methyl-4-formyl-benzoate (88 mg, 0.536 mmol) with $Ni(cod)_2$, tris-*o*-methoxy- phenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 2 above, afforded **1k** in 34% isolated yield as a colorless oil.

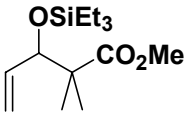
^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.01 (d, *J* = 8.4 Hz, 2H); 7.43 (d, *J* = 8.1 Hz, 2H); 5.92 (ddd, *J* = 6.0, 10.2, 16.9 Hz, 1H); 5.31 (ddd, *J* = 1.5, 1.5, 17.0 Hz, 1H); 5.21 (bd, *J* = 6.0 Hz, 1H); 5.11 (ddd, *J* = 1.4, 1.4, 10.2 Hz, 1H); 3.91 (s, 3H); 0.93 (t, *J* = 7.8 Hz, 9H); 0.61 (dq, *J* = 1.7, 7.5 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 167.2, 149.1, 141.1, 129.8, 129.1, 126.1, 114.5, 75.6, 52.2, 6.9, 5.0.
IR (NaCl, thin film): 2954, 2912, 2877, 1727, 1610, 1436, 1278, 1113, 1019, 842, 745.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{17}H_{26}O_3SiNa$, 329.154; found, 329.155.



(1l)

The reaction of ethylene and pivaldehyde (55 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1l** in 70% isolated yield as a colorless oil.

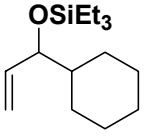
^{1}H NMR (400 MHz, $CDCl_3$, δ): 5.97 (ddd, *J* = 5.9, 10.2, 16.9 Hz, 1H); 5.11 (d, *J* = 8.5 Hz, 1H); 5.08 (bs, 1H); 3.67 (d, *J* = 7.5 Hz, 1H); 0.96 (t, *J* = 7.9 Hz, 9H); 0.86 (s, 9H); 0.63 (q, *J* = 7.7 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 139.4, 115.8, 82.4, 35.5, 26.0, 7.2, 5.3.
IR (NaCl, thin film): 2955, 2877, 1641, 1462, 1239, 1082, 835.



(1m)

The reaction of ethylene and 2,2-dimethyl-3-oxo-propionic acid methyl ester (70 mg, 0.54 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1m** in 81% (0.28 mmol) isolated yield as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 5.75 (ddd, *J* = 7.6, 10.4, 17.5 Hz, 1H); 5.17 (bd, *J* = 17.3 Hz, 1H); 5.15 (bd, *J* = 10.3 Hz, 1H); 4.31 (d, *J* = 7.6 Hz, 1H); 3.66 (s, 3H); 1.15 (s, 3H); 1.05 (s, 3H); 0.92 (t, *J* = 7.9 Hz, 9H); 0.55 (dq, *J* = 1.5, 7.6 Hz, 6H).
^{13}C NMR (100 MHz, $CDCl_3$, δ): 177.4, 137.8, 117.3, 79.2, 51.8, 48.3, 21.4, 19.9, 7.0, 5.2.
IR (NaCl, thin film): 2954, 2878, 1745, 1732, 1642, 1468, 1261, 1087, 834.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{14}H_{28}O_3SiNa$, 295.170; found, 295.171.



(1n)

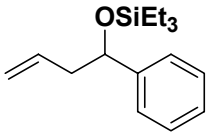
The reaction of ethylene and cyclohexanecarboxaldehyde (60 μL, 0.5 mmol) with $Ni(cod)_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 1 above, afforded **1n** in 25% yield as determined by ^{1}H NMR versus a standard. Another experiment was carried out under 2 atm of ethylene and yielded 34% **1n** and 66% silyl enol ether of cyclohexanecarboxaldehyde. Treatment of this mixture with a TBAF / THF / H_2O solution removed the silyl enol ether from the mixture and column chromatography isolated **1n** as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 5.78 (ddd, *J* = 7.0, 10.3, 17.3 Hz, 1H); 5.07 (m, 2H); 3.78 (t, *J* = 6.6 Hz, 1H); 1.40 – 0.90 (m, 11H); 0.95 (t, *J* = 8.0 Hz, 9H); 0.59 (q, *J* = 8.0 Hz, 6H).
^{13}C NMR (125 MHz, $CDCl_3$, δ): 140.7, 114.8, 78.9, 44.5, 29.0, 29.0, 26.9, 26.5, 26.5, 7.1, 5.2.
IR (NaCl, thin film): 2953, 2926, 2877, 1644, 1451, 1239, 1068, 743.
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{15}H_{30}OSiNa$, 277.196; found, 277.197.

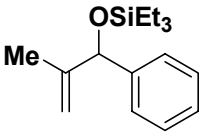
Nickel-catalyzed coupling of monosubstituted olefins and aldehydes (2a – 2p).

Nickel-catalyzed coupling of monosubstituted alkenes and aldehydes (homoallylic products)

General procedure 3. A 10 mL test tube and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.1 mmol, 20 mol%) and ligand (0.2 mmol, 40 mol% as specified) were added to the test tube, the test tube was sealed with a septum, and the sealed tube was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 5 min at room temperature. Alkene (0.5 mL), triethylamine (418 μL, 3 mmol, 600 mol%) and then aldehyde (0.5 mmol, 100 mol%) were added. TESOTf (197 μL, 0.875 mmol, 175 mol%) was added. The mixture was stirred at room temperature for 48 h. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and the crude mixture was diluted in hexane. Purification via flash chromatography on silica afforded the coupling product.

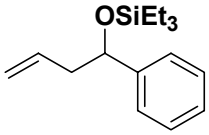


(2a)



(2a')

A 10 mL test tube and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.1 mmol, 20 mol%) and $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 5 min at room temperature. The reaction mixture was purged with propene for 1 min to remove argon, taken care not to introduce oxygen. The propene atmosphere was maintained with a propene balloon. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. benzaldehyde (51 μL, 0.5 mmol, 100 mol%) was added. Silyltriflate (0.875 mmol, 175 mol%, as specified) was added. The mixture was stirred at room temperature for 48 h. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and ^1H NMR of the crude mixture indicated the total yield of **2a** and **2a'** was 73% and the ratio of **2a**:**2a'** is 89:11. Purification via flash chromatography on silica afforded **2a** and **2a'** as colorless oils.



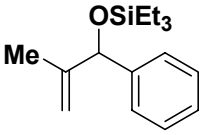
(2a)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.27-7.38 (m, 5H); 5.78-5.89 (m, 1H); 5.05-5.10 (m, 2H); 4.74 (dd, *J* = 7.2, 5.5 Hz, 1H); 2.42-2.59 (m, 2H); 0.94 (t, *J* = 7.9 Hz, 9H); 0.59 (dq, *J* = 2.6, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.3, 135.4, 128.2, 127.2, 126.1, 117.0, 75.1, 45.6, 7.0, 5.0.

IR (NaCl, thin film): 2954, 2927, 2876, 1644, 1493, 1449, 1239, 1090, 858, 699.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{26}OSiNa$, 285.165; found, 285.163.



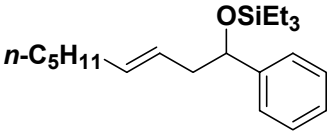
(2a')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.24-7.39 (m, 5H); 5.15 (m, 2H); 4.86 (s, 1H); 1.56 (s, 3H); 0.94 (t, *J* = 7.8 Hz, 9H); 0.61 (q, *J* = 7.8 Hz, 6H).

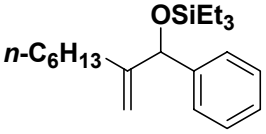
^{13}C NMR (100 MHz, $CDCl_3$, δ): 148.1, 143.5, 128.1, 127.0, 126.3, 111.0, 78.4, 17.4, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2913, 2877, 1451, 1237, 1091, 1066, 1005, 899, 853, 740, 698.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{26}OSiNa$, 285.165; found, 285.165.



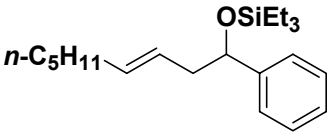
(2b)



(2b')

The reaction of 1-octene and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2b** and **2b'** in 85% total yield according to ^{1}H NMR of the crude mixture and the ratio of **2b**:**2b'** is 95:5. The *E* / *Z* ratio of **2b** is 75:25. Purification via flash chromatography on silica afforded **2b** and **2b'** as colorless oils.

In another experiment, the reaction of 1-octene (1 mL) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, Cy_2PhP (56 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2b'** and **2b** in 73% total yield according to ^{1}H NMR of the crude mixture and the ratio of **2b'**:**2b** is 71:29. Purification via flash chromatography on silica afforded **2b'** and **2b** in 70% isolated yield as a colorless oil.

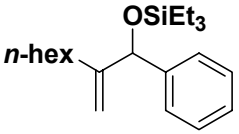


(2b)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.20–7.40 (m, 5H); 5.30–5.50 (m, 2H); 4.63 (dd, *J* = 5.6, 7.2 Hz, 1H); 2.45 (quintet, *J* = 6.1 Hz, 1H); 2.35 (quintet, *J* = 5.9 Hz, 1H); 1.33 (m, 2H); 0.92 (t, *J* = 7.8 Hz, 12H); 0.56 (dq, *J* = 2.4, 7.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.6, 133.3, 128.1, 127.1, 126.6, 126.2, 75.6, 44.5, 32.8, 31.6, 29.3, 22.8, 14.2, 7.0, 5.1.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{21}H_{36}OSiNa$, 355.243; found, 355.244.



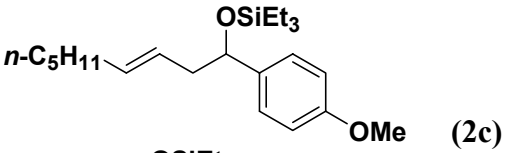
(2b')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.36 (d, *J* = 7.0 Hz, 2H); 7.31 (t, *J* = 7.1 Hz, 2H); 7.24 (t, *J* = 7.2, 1H); 5.22 (bs, 1H); 5.15 (bs, 1H); 4.87 (s, 1H); 1.96 (pentet, *J* = 7.8 Hz, 1H); 1.76 (pentet, *J* = 8.0 Hz, 1H); 1.15 – 1.40 (m, 8H); 0.93 (t, *J* = 8.0 Hz, 9H); 0.87 (t, *J* = 6.8 Hz, 3H); 0.60 (dq, *J* = 1.6, 7.9 Hz, 6H).

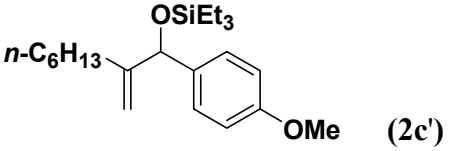
^{13}C NMR (100 MHz, $CDCl_3$, δ): 152.3, 143.8, 128.1, 127.1, 126.6, 109.5, 78.3, 32.0, 30.8, 29.4, 28.0, 22.8, 14.3, 7.0, 5.1.

IR (NaCl, thin film): 2956, 2876, 1647, 1456, 1089, 1066, 742.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{21}H_{36}OSiNa$, 355.243; found, 355.242.

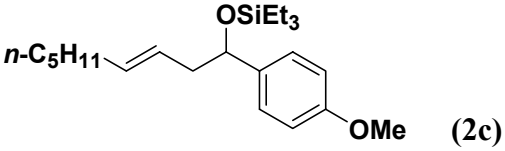


(2c)



(2c')

The reaction of 1-octene and 4-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2c** and **2c'** in 85% total yield according to ^1H NMR of the crude mixture and the ratio of **2c**:**2c'** is >95:5. The *E* / *Z* ratio of **2c** is 75:25. Purification via flash chromatography on silica afforded **2c** as a colorless oil. **2c'** was not detected.



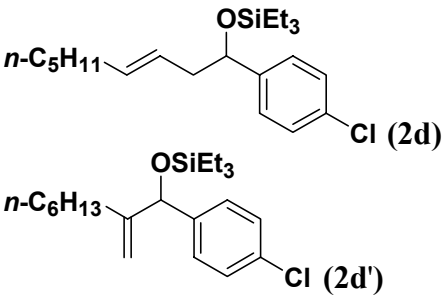
(2c)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.22 (d, *J* = 8.6 Hz, 2H); 6.84 (d, *J* = 8.6 Hz, 2H); 5.33-5.43 (m, 2H); 4.58 (dd, *J* = 6.1 Hz, 6.1 Hz, 1H); 3.81 (s, 3H); 2.27-2.42 (m, 2H); 1.93-1.98 (m, 2H); 1.22-1.60 (m, 6H); 0.95 (t, *J* = 8.0 Hz, 3H); 0.88 (t, *J* = 7.8 Hz, 9H); 0.53 (q, *J* = 7.8 Hz, 6H).

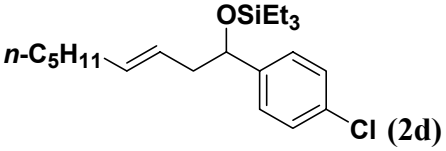
^{13}C NMR (100 MHz, $CDCl_3$, δ): 158.7, 137.9, 133.2, 127.3, 126.7, 113.4, 75.2, 55.4, 44.5, 32.8, 31.6, 29.3, 22.8, 14.3, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2876, 1613, 1512, 1459, 1302, 1247, 1172, 1078, 1005, 972, 830, 742.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{22}H_{38}O_2SiNa$, 385.2539; found, 385.2537.



The reaction of 1-octene and 4-chlorobenzaldehyde (70 mg, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2d** and **2d'** in 37% total yield according to ^1H NMR of the crude mixture and the ratio of **2d**:**2d'** is >95:5. The *E* / *Z* ratio of **2d** is 74:26. Purification via flash chromatography on silica afforded **2d** as a colorless oil. **2d'** was not detected.

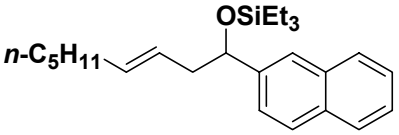


^1H NMR (400 MHz, $CDCl_3$, δ): 7.24 (m, 4H); 5.30-5.41 (m, 2H); 4.61 (dd, *J* = 6.1 Hz, 6.1 Hz, 1H); 2.26-2.40 (m, 2H); 1.89-1.97 (m, 2H); 1.21-1.59 (m, 6H); 0.94 (t, *J* = 8.0 Hz, 3H); 0.89 (t, *J* = 7.8 Hz, 9H); 0.54 (q, *J* = 7.8 Hz, 6H).

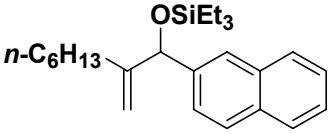
^{13}C NMR (100 MHz, $CDCl_3$, δ): 144.1, 133.7, 132.5, 128.2, 127.5, 126.0, 74.8, 44.4, 32.8, 31.5, 29.2, 22.7, 14.3, 7.0, 4.9.

IR (NaCl, thin film): 2956, 2876, 1647, 1456, 1089, 1066, 742.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{15}H_{20}Na$, 223.1463; found, 223.1305.

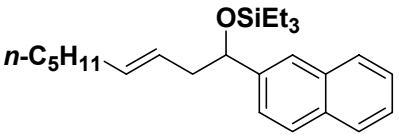


(2e)



(2e')

The reaction of 1-octene and 2-naphthaldehyde (78 mg, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2e** and **2e'** in 88% total yield according to ^1H NMR of the crude mixture and the ratio of **2e**:**2e'** is >95:5. The *E* / *Z* ratio of **2e** is 70:30. Purification via flash chromatography on silica afforded **2e** as a colorless oil. **2e'** was not detected.



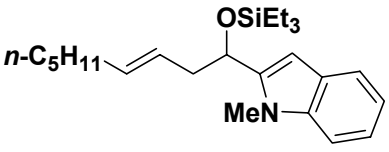
(2e)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.83-7.92 (m, 3H); 7.80 (s, 1H); 7.48-7.59 (m, 3H); 5.43-5.53 (m, 2H); 4.89 (dd, *J* = 6.9, 13.2 Hz, 1H); 2.45-2.68 (m, 2H); 1.98-2.05 (m, 2H); 1.26-1.39 (m, 6H); 0.97 (t, *J* = 8.0 Hz, 9H); 0.94 (t, *J* = 7.6 Hz, 3H); 0.63 (q, *J* = 4.1, 8.0 Hz 6H).

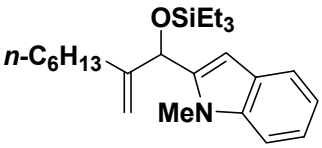
^{13}C NMR (100 MHz, $CDCl_3$, δ): 143.1, 133.4, 133.0, 132.3, 128.1, 127.9, 126.5, 126.0, 125.6, 125.6, 124.7, 124.7, 75.7, 44.4, 32.8, 31.7, 29.3, 22.8, 14.3, 7.0, 5.1.

IR (NaCl, thin film): 2956, 2929, 2875, 1458, 1414, 1377, 1239, 1086, 1005, 972, 744.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{25}H_{38}OSiNa$, 405.2590; found, 405.2584.

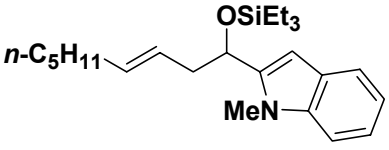


(2f)



(2f')

The reaction of 1-octene and 1-methyl-2-indolecarboxaldehyde (79.6 mg, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2f** and **2f'** in 56% total yield according to ^1H NMR of the crude mixture and the ratio of **2f**:**2f'** is >95:5. The *E* / *Z* ratio of **2f** is 83:17. Purification via flash chromatography on silica afforded **2f** as a colorless oil. **2f'** was not detected.



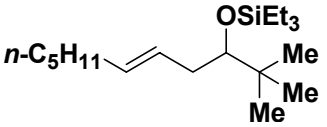
(2f)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.65 (d, *J* = 7.8 Hz, 1H); 7.37 (d, *J* = 8.2 Hz, 1H); 7.27 (t, *J* = 7.1 Hz, 1H); 7.17 (t, *J* = 7.1 Hz, 1H); 6.40 (s, 1H); 5.43-5.59 (m, 2H); 4.96 (dd, *J* = 6.5, 7.4 Hz, 1H); 3.92 (s, 3H); 2.56-2.71 (m, 2H); 2.01-2.07 (m, 2H); 1.29-1.42 (m, 6H); 0.97 (t, *J* = 8.0 Hz, 9H); 0.95 (t, *J* = 4.0 Hz, 3H); 0.63 (dq, *J* = 1.1, 8.0 Hz, 6H).

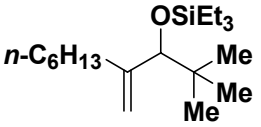
^{13}C NMR (100 MHz, $CDCl_3$, δ): 142.3, 138.4, 133.7, 127.7, 126.2, 121.3, 120.7, 119.4, 109.1, 100.2, 70.6, 42.2, 32.8, 31.6, 31.0, 29.3, 22.8, 14.3, 7.0, 5.0.

IR (NaCl, thin film): 2954, 2927, 2874, 1466, 1339, 1236, 1072, 1010, 731.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{24}H_{39}ONSiNa$, 408.2693; found, 408.2695.

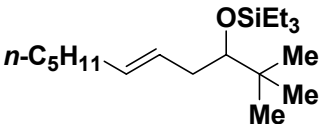


(2g)



(2g')

The reaction of 1-octene and pivaldehyde (55 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2g** and **2g'** in 64% total yield according to ^{1}H NMR of the crude mixture and the ratio of **2g**:**2g'** is >95:5. The *E* / *Z* ratio of **2g** is 78:22. Purification via flash chromatography on silica afforded **2g**. **2g'** was not detected.



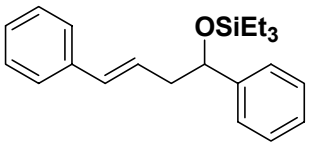
(2g)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 5.37-5.53 (m, 2H); 3.37 (dd, *J* = 3.8, 7.4 Hz, 1H); 2.30-2.36 (m, 1H); 1.99-2.12 (m, 3H); 1.27-1.42 (m, 6H); 0.99 (t, *J* = 8.0 Hz, 9H); 0.92 (t, *J* = 6.8 Hz, 3H); 0.90 (s, 9H); 0.63 (q, *J* = 8.0 Hz, 6H).

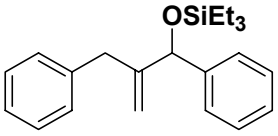
^{13}C NMR (100 MHz, $CDCl_3$, δ): 130.6, 128.5, 81.2, 36.2, 31.8, 31.4, 29.5, 27.6, 26.5, 22.8, 14.2, 7.3, 5.7.

IR (NaCl, thin film): 2956, 2876, 1466, 1238, 1096, 1009, 737.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{40}OSiNa$, 335.2746; found, 335.2741.

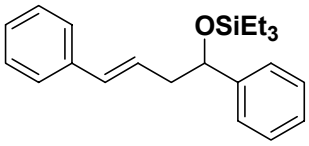


(2h)



(2h')

The reaction of allylbenzene and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2h** and **2h'** in 86% total yield according to ^{1}H NMR of the crude mixture and the ratio of **2h**:**2h'** is 92:8. The *E* / *Z* ratio of **2h** is >95:5. Purification via flash chromatography on silica afforded **2h** as a colorless oil. **2h'** was subjected to TBAF and the free alcohol was isolated by flash chromatography on silica as a colorless oil.



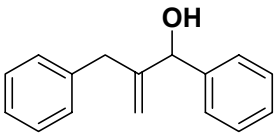
(2h)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30-7.50 (m, 10H); 6.51 (d, *J* = 15.9 Hz, 1H); 6.34 (dt, *J* = 7.2, 15.9 Hz, 1H); 4.89 (dd, *J* = 5.3, 7.2 Hz, 1H); 2.64-2.81 (m, 2H); 1.03 (t, *J* = 7.9 Hz, 9H); 0.68 (dq, *J* = 2.0, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.5, 138.0, 132.4, 128.8, 128.4, 127.4, 127.4, 127.2, 126.3, 126.2, 75.5, 45.0, 7.1, 5.2.

IR (NaCl, thin film): 3062, 3028, 2955, 2911, 2876, 1600, 1494, 1453, 1414, 1239, 1088, 1070, 1006, 965, 830, 742, 700.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for 361.1964; found, 361.1974.



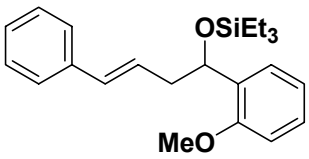
(Desilylated 2h')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.39 (m, 4H), 7.29-7.35 (m, 3H), 7.22-7.24 (m, 1H), 7.13-7.15 (m, 2H), 5.37 (s, 1H); 5.15 (s, 1H); 4.93 (s, 1H); 3.38 (d, *J* = 15.5 Hz, 1H); 3.13 (d, *J* = 15.5 Hz, 1H); 1.24 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 150.6, 142.0, 139.3, 129.4, 128.7, 128.5, 128.1, 127.0, 126.4, 112.4, 76.7, 39.2.

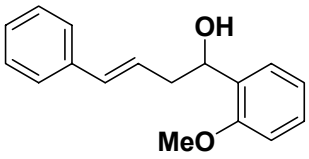
IR (NaCl, thin film): 3377, 3061, 3028, 2919, 1494, 1453, 1025, 909, 750, 699.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{16}H_{16}ONa$, 247.1099; found, 247.1101.



(2i-ortho)

The reaction of allylbenzene and *o*-anisaldehyde (60 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2i-ortho** and **2i'-ortho** in 78% total yield according to ^1H NMR of the crude mixture and the ratio of **2i-ortho**:**2i'-ortho** is 92:8. The *E* / *Z* ratio of **2i-ortho** is >95:5. **2i-ortho** was subjected to TBAF and the free alcohol was isolated as a colorless oil. Allylic alcohol **2i'-ortho** was not isolated.



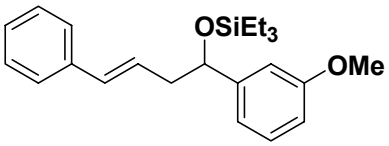
(**Desilylated 2i-ortho**)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.22–7.43 (m, 7H); 7.02 (t, *J* = 7.5 Hz, 1H); 6.93 (d, *J* = 8.1 Hz, 1H); 6,52 (d, *J* = 15.9 Hz, 1H); 6.31 (dt, *J* = 7.2, 15.9 Hz, 1H); 5.09 (dd, *J* = 5.1, 7.5 Hz, 1H); 3.89 (s, 3H); 2.69–2.81 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 156.5, 137.6, 132.8, 131.9, 128.6, 128.5, 127.3, 127.0, 126.9, 126.3, 120.9, 110.6, 70.2, 55.5, 41.3.

IR (NaCl, thin film): 3399, 3026, 2935, 2836, 1601, 1491, 1464, 1438, 1287, 1240, 1181, 1049, 1029, 966, 753, 694.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{23}H_{32}O_2SiNa$, 391.2069; found, 391.2053.



(2i-meta)

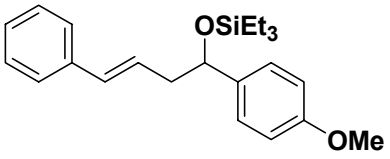
The reaction of allylbenzene and *m*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2i-meta** and **2i'-meta** in 96% total yield according to ^1H NMR of the crude mixture and the ratio of **2i-meta**:**2i'-meta** is 92:8. The *E* / *Z* ratio of **2i-meta** is >95:5. Purification via flash chromatography on silica afforded **2i-meta** as a colorless oil. Allylic alcohol **2i'-meta** was not isolated.

^1H NMR (400 MHz, $CDCl_3$, δ): 7.25–7.41 (m, 6H), 7.03 (m, 1H), 7.0 (d, J = 7.6 Hz, 1H); 6.87 (dd, J = 0.8, 2.7 Hz, 1H); 6.48 (d, J = 15.9 Hz, 1H); 6.30 (dt, J = 7.2, 15.9 Hz, 1H); 3.87 (s, 3H); 2.61–2.75 (m, 2H), 0.99 (t, J = 7.9 Hz, 9H); 0.64 (q, J = 7.9 Hz, 6H).

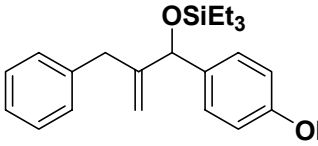
^{13}C NMR (100 MHz, $CDCl_3$, δ): 159.7, 147.1, 137.9, 134.0, 133.8, 132.3, 129.2, 128.9, 128.7, 128.6, 127.2, 127.1, 126.2, 118.4, 112.8, 111.3, 75.2, 55.3, 44.8, 7.0, 5.0.

IR (NaCl, thin film): 3027, 2954, 2910, 2876, 2835, 1601, 1587, 1488, 1456, 1435, 1359, 1320, 1284, 1263, 1153, 1083, 1050, 1006, 966, 943, 825, 779, 743, 699.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{23}H_{32}O_2SiNa$, 391.2069; found, 391.1750.



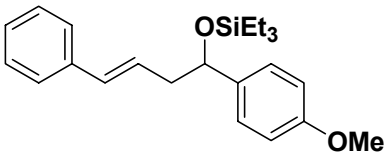
(2i-para)



(2i'-para)

The reaction of allylbenzene and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2i-para** and **2i'-para** in 99% total yield according to ^1H NMR of the crude mixture and the ratio of **2i-para**:**2i'-para** is 92:8. The *E* / *Z* ratio of **2i-para** is >95:5. Purification via flash chromatography on silica afforded **2i-para** as a colorless oil. **2i'-para** was not isolated.

In another experiment, general procedure 3 was followed, except that the reaction was carried out in five fold larger scale. The reaction was heated at 35 °C and 9 mL toluene was used as the solvent. This reaction afforded **2i-para** and **2i'-para** in 98% total yield according to ^1H NMR of the crude mixture and the ratio of **2i-para**:**2i'-para** is 92:8. The *E* / *Z* ratio of **2i-para** is >95:5. Purification via flash chromatography on silica afforded **2i-para** as a colorless oil. **2i'-para** was not isolated.



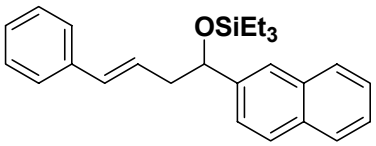
(2i-para)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.49 (m, 7H); 7.00 (d, *J* = 8.6 Hz, 2H); 6.52 (d, *J* = 15.9 Hz, 1H); 6.35 (dt, *J* = 7.2, 15.9 Hz, 1H); 4.85 (dd, *J* = 6.4, 6.4 Hz, 1H); 3.89 (s, 3H); 2.63-2.81 (m, 2H); 1.04 (t, *J* = 7.8 Hz, 9H); 0.70 (q, *J* = 7.8 Hz, 6H).

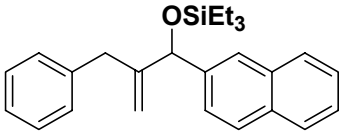
^{13}C NMR (100 MHz, $CDCl_3$, δ): 159.0, 138.1, 137.7, 132.3, 128.7, 127.5, 127.3 127.2, 126.3, 113.7, 75.0, 55.4, 45.1, 7.1, 5.2.

IR (NaCl, thin film): 3027, 2954, 2910, 2875, 1612, 1511, 1414, 1302, 1248, 1171, 1081, 1005, 966, 836, 743, 693.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{23}H_{32}O_2SiNa$, 391.2069; found, 391.2057.

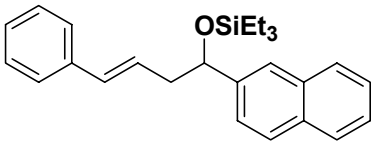


(2j)



(2j')

The reaction of allylbenzene and naphthaldehyde (78 mg, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2j** and **2j'** in 88% total yield according to ^1H NMR of the crude mixture and the ratio of **2j**:**2j'** is 95:5. The *E* / *Z* ratio of **2j** is >95:5. Purification via flash chromatography on silica afforded **2j** as a colorless oil. **2j'** was subjected to TBAF and the free alcohol was isolated by flash chromatography on silica as a colorless oil.



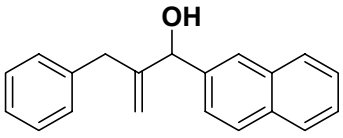
(2j)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.90-7.96 (m, 4H); 7.67 (d, *J* = 1.6 Hz, 1H); 7.60-7.65 (m, 2H); 7.30-7.59 (m, 5H); 6.54 (d, *J* = 15.9 Hz, 1H); 6.36 (dt, *J* = 7.2, 15.9 Hz, 1H); 5.05 (dd, *J* = 5.4, 7.2 Hz, 1H); 2.74-2.89 (m, 2H); 1.03 (t, *J* = 8.0 Hz, 9H); 0.70 (dq, *J* = 2.9, 8.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 142.9, 137.9, 133.4, 133.1, 132.4, 128.7, 128.1, 128.1, 127.9, 127.1, 126.2, 126.1, 125.7, 124.6, 75.5, 44.9, 7.0, 5.1.

IR (NaCl, thin film): 3026, 2954, 2910, 2875, 1507, 1496, 1457, 1239, 1123, 1083, 1005, 965, 819, 744.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{26}H_{32}OSiNa$, 411.2120; found, 411.2167.



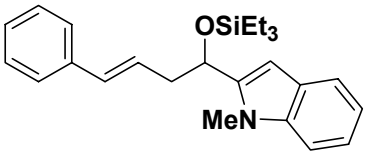
(Desilylated 2j')

^1H NMR (400 MHz, $CDCl_3$, δ): 7.86-7.88 (m, 4H); 7.48-7.55 (m, 3H); 7.20-7.36 (m, 3H); 7.13-7.16 (m, 2H); 5.43 (s, 1H); 5.32 (s, 1H); 4.97 (s, 1H); 3.41 (d, *J* = 15.6 Hz, 1H), 3.16 (d, *J* = 15.6 Hz, 1H), 2.02 (brs, 1H).

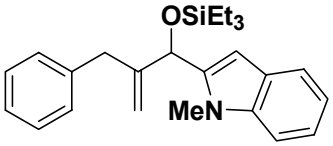
^{13}C NMR (100 MHz, $CDCl_3$, δ): 150.5, 149.2, 139.4, 139.3, 133.4, 133.3, 129.4, 128.6, 128.2, 127.9, 126.4, 126.4, 126.2, 126.0, 124.9, 112.8, 77.4, 39.2.

IR (NaCl, thin film): 3365, 3058, 2923, 1495, 1453, 1031, 908, 819, 745, 700.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{20}H_{18}ONa$, 297.1255; found, 297.1260.



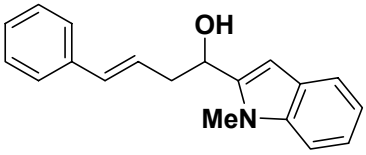
(2k)



(2k')

The reaction of allylbenzene and 1-methyl-2-indolecarboxaldehyde (79.6 mg, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2k** in 57% total yield according to ^1H NMR of the crude mixture and the ratio of **2k**:**2k'** is >95:5. The *E* / *Z* ratio of **2k** is >95:5. **2k'** was not detected. **2k** was subjected to TBAF and the free alcohols were isolated by flash chromatography on silica (buffered with Et_3N) as colorless oils.

In another experiment, the reaction of allylbenzene and 1-methyl-2-indolecarboxaldehyde (79.6 mg, 0.5 mmol) with $Ni(cod)_2$, Cy_2PhP (56mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2k'** and **2k** in 56% total yield according to ^1H NMR of the crude mixture and the ratio of **2k'**:**2k** is 80:20. The *E* / *Z* ratio of **2k** is >95:5. Both **2k'** and **2k** were subjected to TBAF and the free alcohols were isolated by flash chromatography on silica (buffered with Et_3N) as colorless oils.



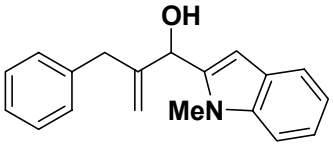
(Desilylated 2k)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.63 (d, *J* = 7.8 Hz, 1H); 7.20-7.41 (m, 7H); 7.14 (t, *J* = 7.8 Hz, 1H); 6.62 (d, *J* = 15.8 Hz, 1H); 6.55 (s, 1H); 6.34 (dt, *J* = 7.3, 15.8 Hz, 1H); 5.01 (m, 1H); 3.86 (s, 3H); 2.93-2.99 (m, 2H); 1.93 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 149.2, 141.3, 138.1, 137.2, 133.8, 128.7, 127.6, 126.4, 125.7, 122.1, 121.0, 119.8, 109.3, 99.4, 66.9, 40.2, 30.4.

IR (NaCl, thin film): 3640, 3026, 2953, 2910, 2875, 1467, 1339, 1237, 1073, 1006, 966, 744.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{19}ONNa$, 300.1364; found, 300.1365.



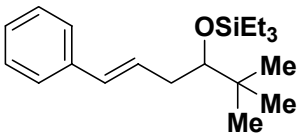
(Desilylated 2k')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.63 (d, 1H); 7.12-7.38 (m, 8H); 6.49 (s, 1H); 5.38 (s, 1H); 5.31 (s, 1H); 5.14 (s, 1H); 3.70 (s, 3H); 3.54 (d, *J* = 15.3 Hz, 1H); 3.33 (d, *J* = 15.3 Hz, 1H); 1.98 (d, *J* = 5.1Hz, 1H).

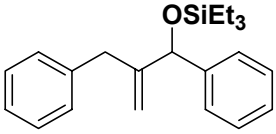
^{13}C NMR (100 MHz, $CDCl_3$, δ): 148.7, 139.6, 139.1, 138.4, 129.3, 128.6, 127.3, 126.6, 122.0, 121.0, 119.7, 113.2, 109.3, 101.5, 69.6, 40.2, 30.3.

IR (NaCl, thin film): 3349, 3059, 3027, 2923, 1649, 1601, 1494, 1468, 1453, 1318, 1234, 1030, 968, 907, 751, 737, 700.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{19}NONa$, 300.1364; found, 300.1369.

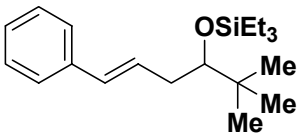


(2l)



(2l')

The reaction of allylbenzene and pivaldehyde (55 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2l** in 65% total yield according to ^1H NMR of the crude mixture and the ratio of **2l**:**2l'** is >95:5. The *E* / *Z* ratio of **2l** is 78:22. **2l'** was not detected. Purification via flash chromatography on silica afforded **2l** as a colorless oil.

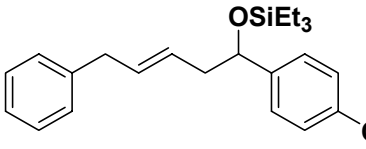


(2l)

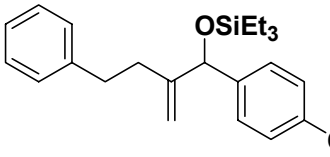
^1H NMR (400 MHz, $CDCl_3$, δ): 7.22–7.40 (m, 5H); 6.43 (d, *J* = 15.9 Hz, 1H); 6.32 (dt, *J* = 7.1, 15.9 Hz, 1H); 3.50 (dd, *J* = 3.4, 7.7 Hz, 1H); 2.49–2.55 (m, 1H), 2.28–2.35 (m, 1H), 1.00 (t, *J* = 8.0 Hz, 9H); 0.96 (s, 9H); 0.64 (q, *J* = 8.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 138.1, 131.3, 129.7, 128.7, 127.0, 126.1, 81.0, 37.4, 36.2, 26.6, 7.3, 5.7.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{20}H_{34}OSiNa$, 341.2277; found, 341.2263.

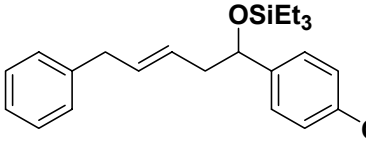


(2m)



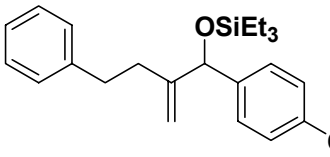
(2m')

The reaction of 4-phenyl-1-butene and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2m** and **2m'** in 91% total yield according to ^1H NMR of the crude mixture and the ratio of **2m**:**2m'** is 92:8. The *E* / *Z* ratio of **2m** is 68:32. Purification via flash chromatography on silica afforded **2m** and **2m'** as colorless oils.



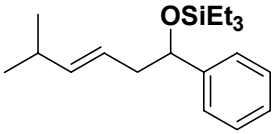
(2m)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.13–7.42 (m, 7H), 6.92 (d, *J* = 8.7 Hz, 2H), 5.49–5.69 (m, 2H), 4.76 (t, *J* = 6.3 Hz, 0.33 H), 4.70 (t, *J* = 6.4 Hz, 0.67 H), 3.87 (s, 3H), 3.37–3.39 (m, 2H), 2.35–2.81 (m, 2H), 0.96 (t, *J* = 7.9 Hz, 6H); 0.60 (q, *J* = 7.9 Hz, 9H).

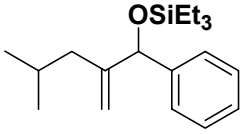


(2m')

^1H NMR (400 MHz, $CDCl_3$, δ): 7.13–7.42 (m, 7H), 6.93 (d, *J* = 8.7 Hz, 2H), 5.34 (s, 1H), 5.19 (s, 1H), 5.00 (s, 1H), 3.86 (s, 3H), 2.61–2.79 (m, 2H), 2.26–2.42 (m, 1H), 2.16–2.22 (m, 1H), 1.07 (t, *J* = 7.8 Hz, 9H); 0.74 (q, *J* = 7.9 Hz, 6H).



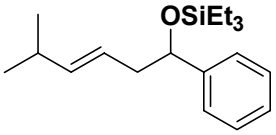
(2n)



(2n')

The reaction of 4-methyl-1-pentene and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2n** and **2n'** in 82% total yield according to ^1H NMR of the crude mixture and the ratio of **2n**:**2n'** is >95:5. The *E* / *Z* ratio of **2n** is 81:19. **2n'** was not detected. Purification via flash chromatography on silica afforded **2n** as a colorless oil.

In another experiment, a 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.2 mmol, 20 mol%) and dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 5 min at room temperature. 4-methyl-1-pentene (633 μL, 5 mmol, 1000 mol%) was added. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. Benzaldehyde (51 μL, 0.5 mmol, 100 mol%) was added to the reaction mixture, followed by TESOTf (197 μL, 0.875 mmol, 175 mol%). The mixture was stirred at room temperature for 14 h. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and NMR of the crude mixture indicated the ratio of **2n'**:**2n** is 75:25. Purification via flash chromatography on silica afforded **2n'** in 44% isolated yield as a colorless oil and **2n** in 10% isolated yield.
This reaction can be run according to general procedure 3, which also afforded **2n'** and **2n** in similar yield.

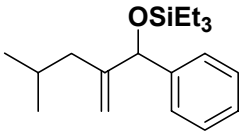


(2n)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30 (m, 5H); 5.40 (m, 2H); 4.63 (dd, *J* = 5.3, 7.3 Hz, 1H); 2.41 (quintet, *J* = 5.3 Hz, 1H); 2.30 (quintet, *J* = 5.5 Hz, 1H); 2.24 (septet, *J* = 6.7 Hz, 1H); 2.00 (m, 2H); 0.95 (dd, *J* = 6.7, 7.6 Hz, 6H); 0.89 (t, *J* = 7.9 Hz, 9H); 0.62 (q, *J* = 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.6, 140.2, 128.1, 127.0, 126.1, 123.7, 75.7, 44.5, 31.3, 22.6, 7.01, 5.0.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{32}OSiNa$, 327.212; found, 327.212.



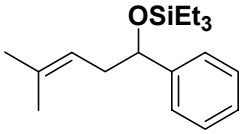
(2n')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.36 (d, *J* = 7.8 Hz, 2H); 7.32 (t, *J* = 7.1 Hz, 2H); 7.25 (t, *J* = 7.1, 1H); 5.30 (bs, 1H); 5.12 (bs, 1H); 4.87 (bs, 1H); 1.65 – 1.85 (m, 3H); 0.93 (t, *J* = 8.0 Hz, 9H); 0.84 (d, *J* = 6.4 Hz, 3H); 0.82 (d, *J* = 6.2 Hz, 3H); 0.60 (dq, *J* = 1.3, 8.3 Hz, 6H).

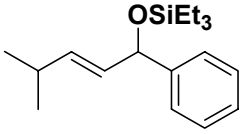
^{13}C NMR (100 MHz, $CDCl_3$, δ): 150.5, 143.7, 128.1, 127.1, 126.7, 110.7, 77.9, 41.1, 26.3, 23.0, 22.6, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2877, 1646, 1454, 1088, 1067, 743.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{19}H_{32}OSiNa$, 327.211; found, 327.212.

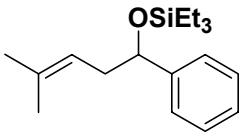


Triethyl-(4-methyl-1-phenyl-pent-3-enyloxy)-silane (2o).



Triethyl-(4-methyl-1-phenyl-pent-2-enyloxy)-silane (2o').

The reaction of 3-methyl-1-butene and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2o** and **2o'** in 95% total yield according to ^1H NMR of the crude mixture and the ratio of **2o**:**2o'** is 86:14. The *E* / *Z* ratio of **2o'** is >95:5. Purification via flash chromatography on silica afforded **2o**. **2o'** was not isolated.



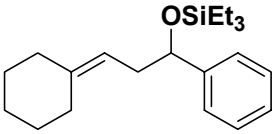
(2o)

^1H NMR (400 MHz, $CDCl_3$, δ): 7.27-7.43 (m, 5H); 5.19-5.24 (m, 1H); 4.68 (dd, *J* = 5.8, 7.2 Hz, 1H); 2.36-2.54 (m, 2H); 1.74 (d, *J* = 0.8 Hz, 3H); 1.58 (s, 3H); 0.95 (t, *J* = 7.8 Hz, 9H); 0.60 (dq, *J* = 3.4, 7.8 Hz, 6H).

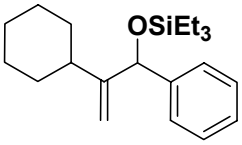
^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.8, 133.6, 128.1, 127.0, 126.1, 121.0, 75.4, 40.0, 26.0, 18.0, 7.0, 5.0.

IR (NaCl, thin film): 3028, 2956, 2877, 2912, 1454, 1414, 1377, 1239, 1089, 1069, 1005, 941, 744, 699.

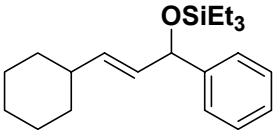
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{18}H_{30}OSiNa$, 313.1964; found, 313.1966.



(2p)



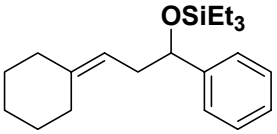
(2p')



(3p)

The reaction of vinylcyclohexane and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **2p** and **3p** in 99% total yield according to ^1H NMR of the crude mixture and the ratio of **2p**:**3p** is 75:25. The *E* / *Z* ratio of **3p** is >95:5. Purification via flash chromatography on silica afforded a mixture of **2p** and **3p**.

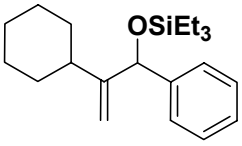
In another experiment, a 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.2 mmol, 20 mol%) and dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 5 min at room temperature. Vinylcyclohexane (856 μL, 6.25 mmol, 1250 mol%) was added. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. Benzaldehyde (51 μL, 0.5 mmol, 100 mol%) was added, followed by TESOTf (197 μL, 0.875 mmol, 175 mol%). The mixture was stirred at room temperature for 16 h. The mixture was filtered through a plug of silica gel. 1H NMR of the crude mixture indicated that **2p'** is the minor product, along with homoallylic product **2p** and 1,3-disubstituted allylic product **3p** as major products. Purification via flash chromatography on silica afforded **2p'** in 5% isolated yield as a colorless oil.



(2p)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.24-7.42 (m, 5H); 5.14 (t, *J* = 7.4 Hz, 1H); 4.66 (t, *J* = 6.4 Hz, 1H); 2.37-2.52 (m, 2H); 2.00-2.11 (m, 3H); 1.50-1.78 (m, 3H); 1.03-1.48 (m, 4H); 0.94 (t, *J* = 7.9 Hz, 9H); 0.59 (dq, *J* = 2.8, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.7, 141.6, 128.0, 127.0, 126.2, 117.5, 75.6, 39.0, 37.5, 29.0, 28.7, 27.8, 27.1, 7.0, 5.0.



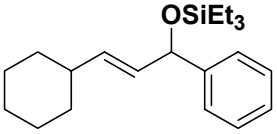
(2p')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.33 (d, *J* = 8.6 Hz, 2H); 7.29 (t, *J* = 7.9, 2H); 7.22 (t, *J* = 7.0 Hz, 1H); 5.23 (dd, *J* = 1.3, 1.3 Hz, 1H); 5.14 (s, 1H); 4.90 (s, 1H); 1.2 – 2.0 (m, 11H); 0.91 (t, *J* = 7.9 Hz, 9H); 0.58 (dq, J = 0.5, 7.8 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 157.7, 143.7, 128.0, 127.1, 126.9, 108.2, 77.6, 39.5, 34.5, 33.5, 27.1, 27.0, 26.5, 7.1, 5.0.

IR (NaCl, thin film): 2954, 2927, 2876, 1644, 1493, 1449, 1239, 1090, 858, 699.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{21}H_{34}OSiNa$, 353.227; found, 353.227.



(3p)

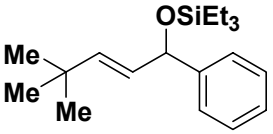
^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.24-7.42 (m, 5H); 5.69 (dd, *J* = 6.5, 15.4 Hz, 1H); 5.56 (dd, *J* = 7.0, 15.4 Hz, 1H); 5.18 (d, *J* = 7.0 Hz, 1H); 2.00-2.11 (m, 3H); 1.63-1.78 (m, 1H); 1.50-1.78 (m, 3H); 1.03-1.48 (m, 4H); 1.00 (t, *J* = 8.0 Hz, 9H); 0.67 (dq, *J* = 2.3, 8.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 144.8, 136.9, 131.2, 128.2, 126.9, 126.1, 75.9, 40.4, 33.0, 32.9, 26.4, 26.2, 7.1, 5.2.

The following IR and HRMS data is from a mixture of **2p** and **2p'**.

IR (NaCl, thin film): 2954, 2928, 2876, 2853, 1449, 1414, 1238, 1086, 1067, 1007, 969, 829, 744, 699.

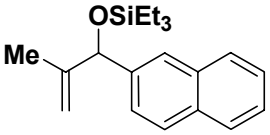
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{21}H_{34}OSiNa$, 353.2277; found, 353.2267.



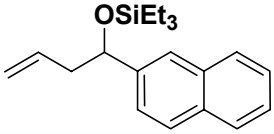
(**3q**)

The reaction of 3,3-dimethyl-1-butene and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **3q** only in 14% total yield according to ^1H NMR of the crude mixture. Purification via flash chromatography on silica afforded **3q** as a colorless oil.

^1H NMR (400 MHz, $CDCl_3$, δ): 7.30-7.41(m, 5H), 5.82 (d, J = 14.6 Hz, 1H), 5.59 (dd, J = 14.6, 7.0 Hz, 1H), 5.18 (m, 1H), 1.88 (brs, 1H), 1.05 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 149.2, 143.8, 128.6, 127.6, 127.3, 126.4, 75.6, 33.1, 29.6.
IR (NaCl, thin film): 3657, 2954, 2876, 1457, 1238, 966, 737, 691.
HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{19}H_{32}OSiNa$, 327.2115; found, 327.2105.

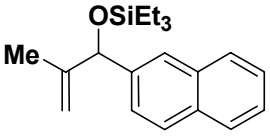


(2r')



(2r)

A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (28 mg, 0.1 mmol, 20 mol%), dicyclohexylphenylphosphine (56 mg, 0.2 mmol, 40 mol%) and 2-naphthaldehyde (78 mg, 0.5 mmol, 100 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (2.5 mL) under argon and stirred 5 min at room temperature. The system was purged with propene for 1 min. The propene atmosphere was maintained by a propene balloon. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. TESOTf (197 μL, 0.875 mmol, 175 mol%) was added. The mixture was stirred at room temperature for 6 h. The mixture was diluted with hexane and filtered through a plug of silica gel. Solvent was removed under reduced pressure. Purification via flash chromatography on silica afforded **2r'** in 73% isolated yield as a colorless oil and **2r** in 14% isolated yield as a colorless oil.



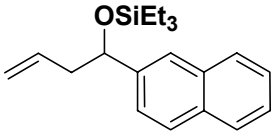
(2r')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.86 (m, 4H); 7.50 (m, 3H); 5.33 (s, 1H); 5.26 (s, 1H); 4.94 (s, 1H); 1.62 (s, 3H); 1.00 (t, J = 8.0 Hz, 9H); 0.67 (dq, J = 1.8, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 148.0, 141.0, 133.4, 133.0, 128.2, 127.8, 127.8, 126.0, 125.7, 124.9, 124.8, 78.6, 17.6, 7.1, 5.1.

IR (NaCl, thin film): 2955, 2912, 2876, 1652, 1508, 1457, 1238, 1084, 1005, 899, 742.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{20}H_{28}OSiNa$, 335.180; found, 335.181.



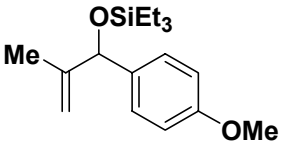
(2r)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.83 (t, *J* = 8.5 Hz, 3H); 7.75 (s, 1H); 7.48 (m, 3H); 5.81 (m, 1H); 5.05 (m, 1H); 5.02 (m, 1H); 4.86 (t, *J* = 5.9 Hz); 2.55 (m, 2H); 0.91 (t, *J* = 8.0 Hz, 9H); 0.57 (dq, *J* = 3.5, 7.5 Hz, 6H).

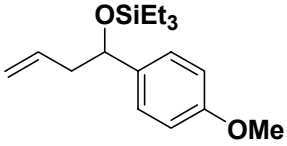
^{13}C NMR (100 MHz, $CDCl_3$, δ): 142.8, 135.3, 133.4, 133.0, 128.1, 127.9, 127.9, 126.1, 125.7, 124.6, 75.2, 45.6, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2876, 1458, 1239, 1084, 1005, 914, 817, 743.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{20}H_{28}OSiNa$, 335.180; found, 335.181.

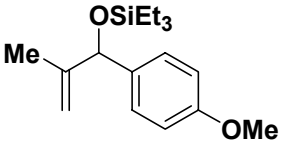


(2s')



(2s)

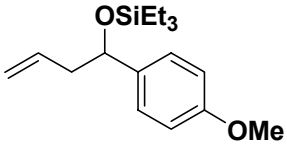
The reaction of propene (1atm, balloon) and *p*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, Cy_2PhP (56 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the procedure for **2r'** above afforded **2s'** and **2s** and the ratio of **2s'**:**2s** is 82:18. Purification via flash chromatography on silica afforded **2s'** and **2s** as a colorless mixture in 95% isolated yield.



(2s')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30 (d, *J* = 8.7 Hz, 2H); 6.90 (d, *J* = 8.7 Hz, 2H); 5.22 (s, 1H); 5.08 (s, 1H); 4.96 (s, 1H); 3.62 (s, 3H); 2.15 (s, 1H); 1.62 (s, 3H).

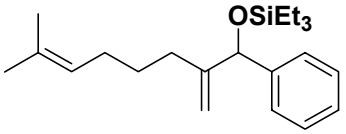
^{13}C NMR (100 MHz, $CDCl_3$, δ): 159.3, 147.2, 134.3, 127.9, 113.9, 110.8, 77.5, 55.4, 18.7.



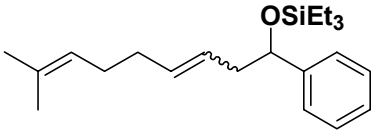
(2s)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30 (d, *J* = 8.7 Hz, 2H); 6.90 (d, *J* = 8.7 Hz, 2H); 5.82 (m, 1H); 5.15 (m, 2H); 4.69 (t, *J* = 6.5 Hz, 1H); 2.52 (d, *J* = 6.8 Hz, 2H), 2.15 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 159.1, 136.2, 134.8, 127.3, 118.4, 113.9, 73.1, 55.4, 43.9.



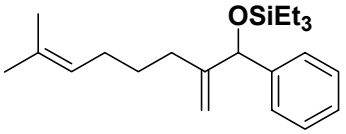
(2t')



(2t)

A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $Ni(cod)_2$ (27.5 mg, 0.2 mmol, 20 mol%) and dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (1.0 mL) under argon and stirred 5 min at room temperature. 7-methyl-1,7-octa- diene (825 μL, 5 mmol, 1000 mol%) was added. Triethylamine (418 μL, 3 mmol, 600 mol%) was added. TESOTf (197 μL, 0.875 mmol, 175 mol%) was added. Benzaldehyde (51 μL, 0.5 mmol, 100 mol%) in 1.5 mL toluene was added to the reaction mixture over 6 min. The mixture was stirred at room temperature for 18 h. The mixture was filtered through a plug of silica gel. Solvent was removed under reduced pressure and ^{1}H NMR of the crude mixture indicated the ratio of **2t'**:**2t** is 71:29. Purification via flash chromatography on silica afforded **2t'** in 50% isolated yield as a colorless oil and **2t** in 22% isolated yield as a colorless oil.

This reaction can be run according to general procedure 3, which also afforded **2t'** and **2t** in similar yield.



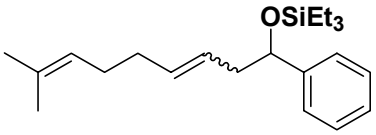
(2t')

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.40 (d, *J* = 7.0 Hz, 2H); 7.34 (t, *J* = 7.8 Hz, 2H); 7.27 (t, *J* = 7.2, 1H); 5.26 (bs, 1H); 5.18 (bs, 1H); 5.10 (t, *J* = 7.2 Hz, 1H); 4.81 (bs, 1H); 1.76 – 2.10 (m, 4H); 1.71 (s, 3H); 1.60 (s, 3H); 1.44 (quintet, *J* = 7.7 Hz, 2H); 0.97 (t, *J* = 7.9 Hz, 9H); 0.62 (dq, *J* = 1.5, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 152.1, 143.7, 131.6, 128.1, 127.1, 126.6, 124.8, 109.5, 78.2, 30.4, 28.2, 28.1, 25.9, 17.8, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2877, 1647, 1456, 1091, 1067, 743.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{22}H_{36}OSiNa$, 367.243; found, 367.243.



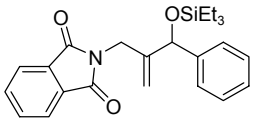
(2t)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.30 (m, 5H); 5.45 (m, 2H); 5.15 (t, *J* = 7.1 Hz, 1H); 4.64 (dd, *J* = 5.4, 7.3 Hz, 1H); 2.45 (quintet, *J* = 5.4 Hz, 1H); 2.35 (quintet, *J* = 5.9 Hz, 1H); 2.05 (m, 4H); 1.62 (s, 3H); 1.72 (s, 3H); 0.92 (t, *J* = 7.9 Hz, 9H); 0.55 (dq, *J* = 1.5, 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.6, 132.8, 131.7, 128.1, 127.1, 126.9, 126.1, 124.4, 75.6, 44.5, 33.1, 28.2, 25.9, 17.9, 7.0, 5.0.

IR (NaCl, thin film): 2955, 2914, 2876, 1454, 1089, 1005, 969, 699.

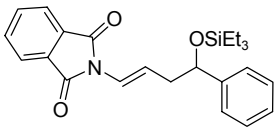
HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{22}H_{36}OSiNa$, 367.243; found, 367.243.



(4a)

The reaction of allylphthalimide (281 mg, 1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4a** and **4a'** in 67% total yield according to ^{1}H NMR of the crude mixture and the ratio of **4a**:**4a'** is 74:26. Purification via flash chromatography on silica afforded **4a** as a mixture of **4a** and the isomerized starting material.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.77 (dd, *J* = 3.0, 5.4 Hz, 2H); 7.73 (dd, *J* = 3.0, 5.4 Hz, 2H); 7.13–7.41 (m, 5H); 5.36 (s, 1H), 5.30 (s, 1H), 4.99 (s, 1H), 4.26 (d, *J* = 16 Hz, 1H), 4.08 (d, *J* = 16 Hz, 1H), 0.91 (t, *J* = 7.9 Hz, 9H); 0.59 (q, *J* = 7.9 Hz, 6H).



(4a').

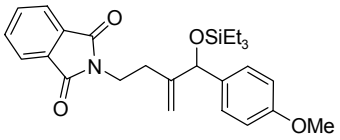
The reaction of allylphthalimide (1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4a** and **4a'** in 43% total yield according to ^{1}H NMR of the crude mixture and the ratio of **4a**:**4a'** is 12:88. The *E* / *Z* ratio of **4a'** is 60:40. Purification via flash chromatography on silica afforded **4a'** as a mixture with the isomerized starting material.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.86 (dd, *J* = 3.1, 5.4 Hz, 2H); 7.73 (dd, *J* = 3.1, 5.4 Hz, 2H); 7.25–7.37 (m, 5H); 6.62 (m, 2H); 4.76 (dd, *J* = 5.5, 6.9 Hz, 1H); 2.47–2.60 (m, 2H); 0.89 (t, *J* = 8.0 Hz, 9H); 0.56 (q, *J* = 2.8, 8.0 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 166.7, 149.2, 145.0, 134.5, 131.9, 128.2, 127.3, 126.1, 123.7, 119.5, 118.8, 75.0, 43.1, 7.0, 5.0.

IR (NaCl, thin film): 2954, 2876, 1781, 1721, 1384, 1088, 1069, 715, 701.

HRMS–ESI (m/z): $[M–OTES]^+$ calcd for $C_{18}H_{14}NO_2Na$, 276.1025; found, 276.1022.



(4b)

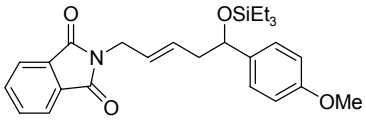
The reaction of homoallylphthalimide (1.5 mmol, 300 mol%) and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4b** and **4b'** in 54% total yield according to ^{1}H NMR of the crude mixture and the ratio of **4b**:**4b'** is 71:29. Purification via flash chromatography on silica afforded **4b** and **4b'.**

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.81 (dd, *J* = 3.0, 5.4, 2H); 7.70 (dd, *J* = 3.0, 5.4, 2H); 7.26 (d, *J* = 8.7 Hz, 2H); 6.79 (d, *J* = 8.7 Hz, 2H); 5.27 (s, 1H); 5.15 (s, 1H); 4.99 (s, 1H); 3.66−3.86 (m, 2H); 3.78 (s, 3H); 2.33−2.40 (m, 1H); 2.16−2.23 (m, 1H); 0.90 (t, *J* = 7.9 Hz, 9H); 0.57 (q, *J* = 7.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 168.4, 158.8, 148.6, 135.2, 134.0, 132.3, 127.7, 123.3, 113.5, 111.8, 77.6, 55.3, 37.2, 29.8, 7.0, 5.0.

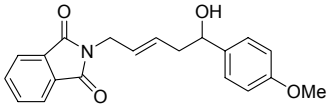
IR (NaCl, thin film): 2954, 2876, 1773, 1715, 1511, 1467, 1431, 1395, 1354, 1247, 1078, 952, 719.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{26}H_{33}O_4SiNa$, 474.2066; found, 474.2071.



(4b')

The reaction of homoallylphthalimide (1.5 mmol, 300 mol%) and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4b** and **4b'** in 76% total yield according to ^1H NMR of the crude mixture and the ratio of **4b**:**4b'** is <5:95. Treatment of 4b' with TBAF followed by flash chromatography on silica afforded a desilylated **4b'**.



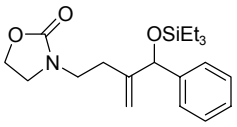
(Desilylated 4b')

^1H NMR (400 MHz, $CDCl_3$, δ): 7.86 (dd, *J* = 3.1, 5.4 Hz, 2H); 7.73 (dd, *J* = 3.1, 5.4 Hz, 2H); 7.25 (d, *J* = 8.7 Hz, 2H); 6.84 (d, *J* = 8.7 Hz, 2H); 5.73(dt, *J* = 6.0, 15.4 Hz, 1H); 5.62 (dt, *J* = 6.0, 15.4 Hz, 1H); 4.68 (dd, *J* = 6.4, 6.4 Hz, 1H); 4.25−4.33 (m, 2H); 3.78 (s, 3H); 2.46 (m, 2H), 2.09 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 168.2, 159.1, 136.1, 134.1, 132.3, 130.8, 127.2, 127.1, 123.5, 113.9, 73.1, 55.4, 42.3, 39.7.

IR (NaCl, thin film): 3466, 2929, 1770, 1711, 1611, 1512, 1395, 1249, 1174, 1034, 833, 720.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{20}H_{18}NO_3Na$, 320.129; found, 320.130.



(4c)

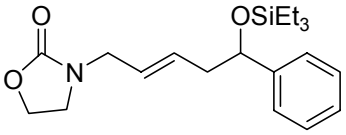
The reaction of homoallyloxazolidinone (1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at room temperature following the general procedure 3 above afforded **4c** and **4c'** in 60% total yield according to ^{1}H NMR of the crude mixture and the ratio of **4c**:**4c'** is 83:17. Purification via flash chromatography on silica afforded **4c** and **4c'** as colorless oils.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.23–7.38 (m, 5H); 5.31 (s, 1H); 5.20 (s, 1H); 5.00 (s, 1H); 4.16–4.21 (m, 2H); 3.19–3.36 (m, 4H); 2.02–2.26 (m, 2H); 0.93 (t, J = 7.9 Hz, 9H); 0.60 (q, J = 7.8 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 158.4, 148.1, 143.1, 128.2, 127.4, 126.3, 112.0, 78.1, 61.8, 44.3, 42.8, 27.9, 7.0, 4.9.

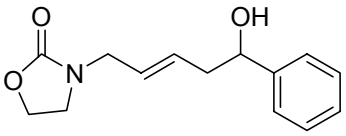
IR (NaCl, thin film): 2955, 2912, 2876, 1753, 1484, 1426, 1265, 1089, 1067, 1044, 1007, 861, 744, 701.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{20}H_{31}NO_3Na$, 384.1965; found, 384.1951.



(4c')

The reaction of homoallylphthalimide (1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at room temperature following the general procedure 3 above afforded **4c** and **4c'** in 28% total yield according to ^1H NMR of the crude mixture and the ratio of **4c**:**4c'** is 10:90. 4c' was subjected to TBAF and purification via flash chromatography on silica afforded a desilylated **4c'**.



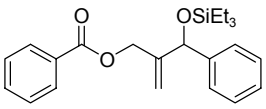
(Desilylated 4c')

^1H NMR (400 MHz, $CDCl_3$, δ): 7.28–7.42 (m, 5H), 5.68 (dt, *J* = 5.7, 7.1 Hz, 1H), 5.49 (dt, *J* = 5.7, 7.1 Hz, 1H), 4.77 (dd, *J* = 6.7, 6.8 Hz, 1H), 4.28 (t, *J* = 8.0 Hz, 2H), 3.80–3.82 (m, 2H), 3.38 (dt, *J* = 2.5, 8.0 Hz, 2H), 2.53–2.59 (m, 2H), 2.11–2.17 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 158.4, 143.9, 131.2, 128.7, 127.8, 127.3, 126.0, 73.8, 61.9, 46.4, 44.2, 42.1.

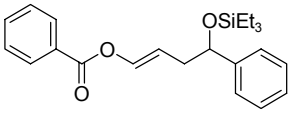
IR (NaCl, thin film): 3421, 2919, 2361, 1734, 1653, 1490, 1437, 1259, 1038, 762, 702.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{14}H_{17}NO_3Na$, 270.111; found, 270.110.



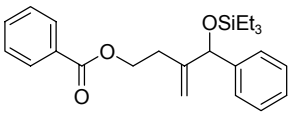
(4d)

The reaction of allylbenzoate (2.5 mmol, 500 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene following the general procedure 3 above afforded **4d** and **4d'** in <5% total yield according to ^{1}H NMR of the crude mixture. **4d** and **4d'** were not isolated from the reaction mixture.



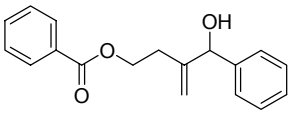
(4d')

The reaction of allylbenzoate (2.5 mmol, 500 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4d** and **4d'** in <5% total yield according to ^{1}H NMR of the crude mixture. **4d** and **4d'** were not isolated from the reaction mixture.



(4e)

The reaction of homoallylbenzoate (1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at room temperature following the general procedure 3 above afforded **4e** and **4e'** in 21% total yield according to ^{1}H NMR of the crude mixture. **4e** was subjected to TBAF and the free alcohol was isolated as a colorless oil.



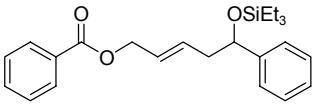
(Desilylated 4e)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.02 (d, J = 7.3 Hz, 2H); 7.58 (t, J = 7.3 Hz, 1H); 7.28 (m, 7H); 5.37 (s, 1H); 5.29 (s, 1H); 5.12 (s, 1H); 4.36–4.50 (m, 2H); 2.34–2.51 (m, 2H); 2.29 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 166.9, 147.0, 141.8, 133.1, 130.4, 129.7, 128.7, 128.5, 128.0, 126.7, 113.3, 77.6, 63.7, 31.3.

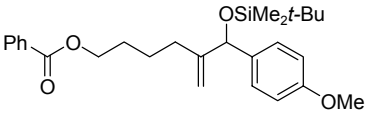
IR (NaCl, thin film): 3447, 3063, 3030, 2961, 1717, 1701, 1451, 1316, 1276, 1117, 1071, 1026, 912, 712, 701, 668.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{18}H_{18}O_3Na$, 305.1148; found, 305.1156.



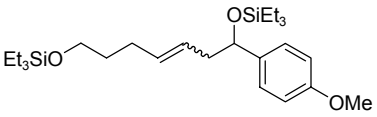
(4e')

The reaction of homoallylbenzoate (1.5 mmol, 300 mol%) and benzaldehyde (51 μL, 0.5 mmol) with $Ni(cod)_2$, Ph_3P (52 mg, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4e** and **4e'** in <5% total yield according to ^1H NMR of the crude mixture and. **4e'** was not isolated from the reaction mixture.

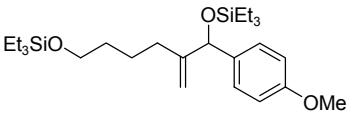


(4f).

The reaction of 1-hexen-6-benzoate (510.3 mg, 2.5 mmol, 500 mol%) and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol, 175 mol%), triethylamine in toluene following the general procedure 3 above afforded **4f** and **4f'** in 44% total isolated yield after flash chromatography on silica and according to ^1H NMR of the crude mixture the ratio of **4f**:**4f'** is 73:27. **4f** and **4f'** were isolated together as a mixture.

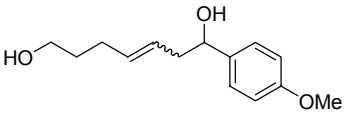


(4g)



(4g')

The reaction of triethyl-hex-5-enyloxy-silane (1.5 mmol, 300 mol%) and *o*-anisaldehyde (61 μL, 0.5 mmol) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol, 175 mol%), triethylamine in toluene at 35 °C following the general procedure 3 above afforded **4g** and **4g'** in 66% total yield according to ^{1}H NMR of the crude mixture and the ratio of **4g**:**4g'** is 92:8. The *E* / *Z* ratio of **4g** is 50:50. **4g'** was not isolated from the mixture. **4g** were subjected to TBAF and the free diols was isolated via flash chromatography on silica as a colorless oil.



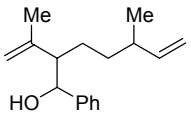
(Desilylated 4g)

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.26–7.28 (m, 2H); 6.89 (d, *J* = 8.6 Hz, 2H); 5.41–5.63 (m, 2H), 4.70 (dd, *J* = 4.8, 8.0 Hz, 0.5 H), 4.64 (dd, *J* = 7.2, 7.2 Hz, 0.5 H), 3.81 (s, 3H), 3.60–3.65 (m, 2H), 2.39–2.62 (m, 2H), 2.10–2.27 (m, 2H), 1.89 (brs, 2H), 1.58–1.67 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 159.2, 159.2, 136.5, 136.4, 134.2, 132.5, 127.2, 127.2, 126.6, 126.1, 114.0, 113.9, 73.7, 73.4, 62.7, 62.0, 55.5, 42.8, 37.3, 32.3, 32.1, 29.5, 23.7.

IR (NaCl, thin film): 3354, 2933, 1612, 1513, 1442, 1303, 1247, 1175, 1035, 832.

HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{26}H_{48}O_3Si_2Na$, 487.3040; found, 487.3017.



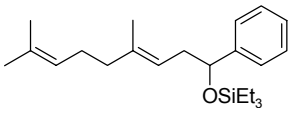
(5a)

To β-Citronellene (0.5 mmol) in anhydrous CH_2Cl_2 (5 mL) was added Me_2AlCl (1.0 M in hexane, 1.1 mL) at 0 °C. The mixture was stirred at room temperature for 24 h. The reaction was quenched by diluting the reaction mixture with diethylether, followed by slow addition of water until gas evolution ceased. The organic layer was separated, and the aqueous layer was extracted with ether twice. The combined organic layers were washed with brine, dried and evaporated in vacuo. Purification via flash chromatography on silica gel afforded the coupling product **5a** as a colorless oil. Homoallylic alcohol **5b** was not detected.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.28-7.39 (m, 5H); 5.45-5.61 (m, 1H); 5.09 (s, 1H); 5.00 (s, 1H); 4.79-4.94 (m, 2H); 4.38 (dd, J = 0.7, 8.5 Hz, 1H); 2.19-2.35 (m, 1H); 1.89-2.05 (m, 1H); 1.73, 1.75 (two s, 3H); 1.67 (brs, 1H); 0.91-1.27 (m, 4H); 0.87, 0.84 (two d, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.1, 144.3, 142.9, 128.5, 127.9, 127.4, 127.3, 126.0, 116.5, 116.4, 113.1, 112.4, 75.5, 75.4, 56.5, 56.4, 37.8, 37.5, 34.2, 34.2, 31.2, 26.3, 21.0, 19.5, 18.3, 18.1.

HRMS-EI (m / z): $[M]^{+\cdot}$ calcd for $C_{17}H_{24}ONa$, 244.182; found, 244.182.



(5b)

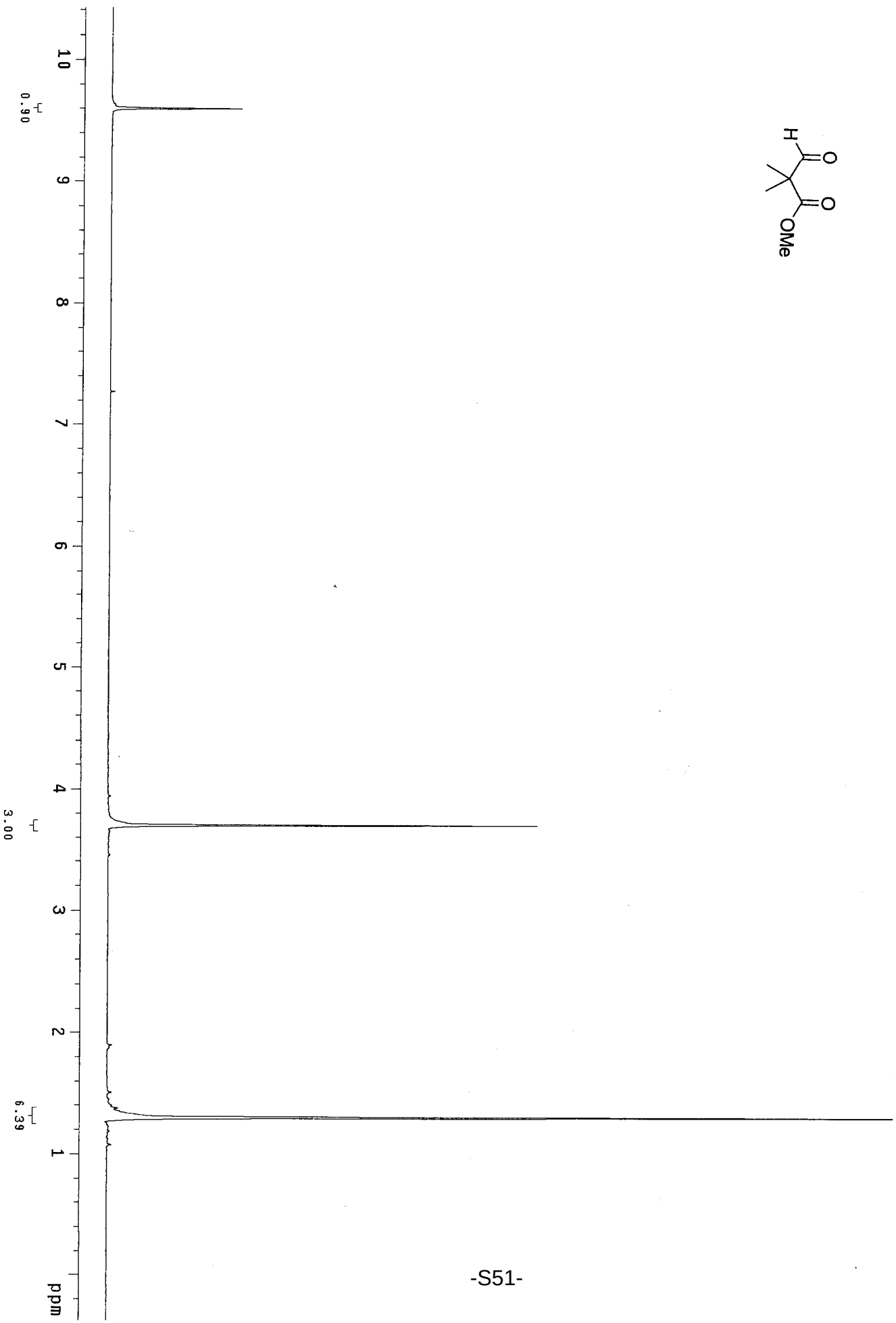
The reaction of β-Citronellene and benzaldehyde (51 μL, 0.5 mmol, 100 mol%) with $Ni(cod)_2$, $EtOPh_2P$ (43 μl, 0.2 mmol, 40 mol%) and TESOTf (197 μL, 0.875 mmol, 175 mol%), triethylamine in toluene following the general procedure 3 above afforded **5b** 75% total yield according to ^{1}H NMR of the crude mixture and the E/Z ratio of **5b** is 71:29. **5a** was not detected. Purification via flash chromatography on silica afforded **5b** as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.25-7.36 (m, 5H); 5.18-5.20 (m, 1H); 5.12-5.12 (m, 1H); 4.64-4.68 (m, 1H); 2.37-2.48 (m, 2H); 2.01-2.10 (m, 4H); 1.73 (m, 4H); 1.64 (m, 3H); 1.55 (s, 2H); 0.92 (t, J = 6.9 Hz, 9H); 0.57 (dq, J = 3.0, 6.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 145.8, 145.8, 137.2, 137.1, 131.7, 131.5, 128.1, 128.0, 127.0, 127.0, 126.1, 126.1, 124.6, 124.5, 121.8, 120.7, 75.5, 75.3, 40.0, 39.8, 39.7, 32.3, 26.8, 26.7, 25.9, 25.9, 23.6, 17.8, 17.8, 16.3, 7.0, 7.0, 5.0, 5.0.

IR (NaCl, thin film): 2955, 2876, 1454, 1376, 1239, 1088, 1068, 1006, 829, 743, 700.

HRMS-ESI (m / z): $[M + Na]^+$ calcd for $C_{23}H_{28}OSiNa$, 381.2590; found, 381.2583.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7
6
5
4
3
2
1
ppm
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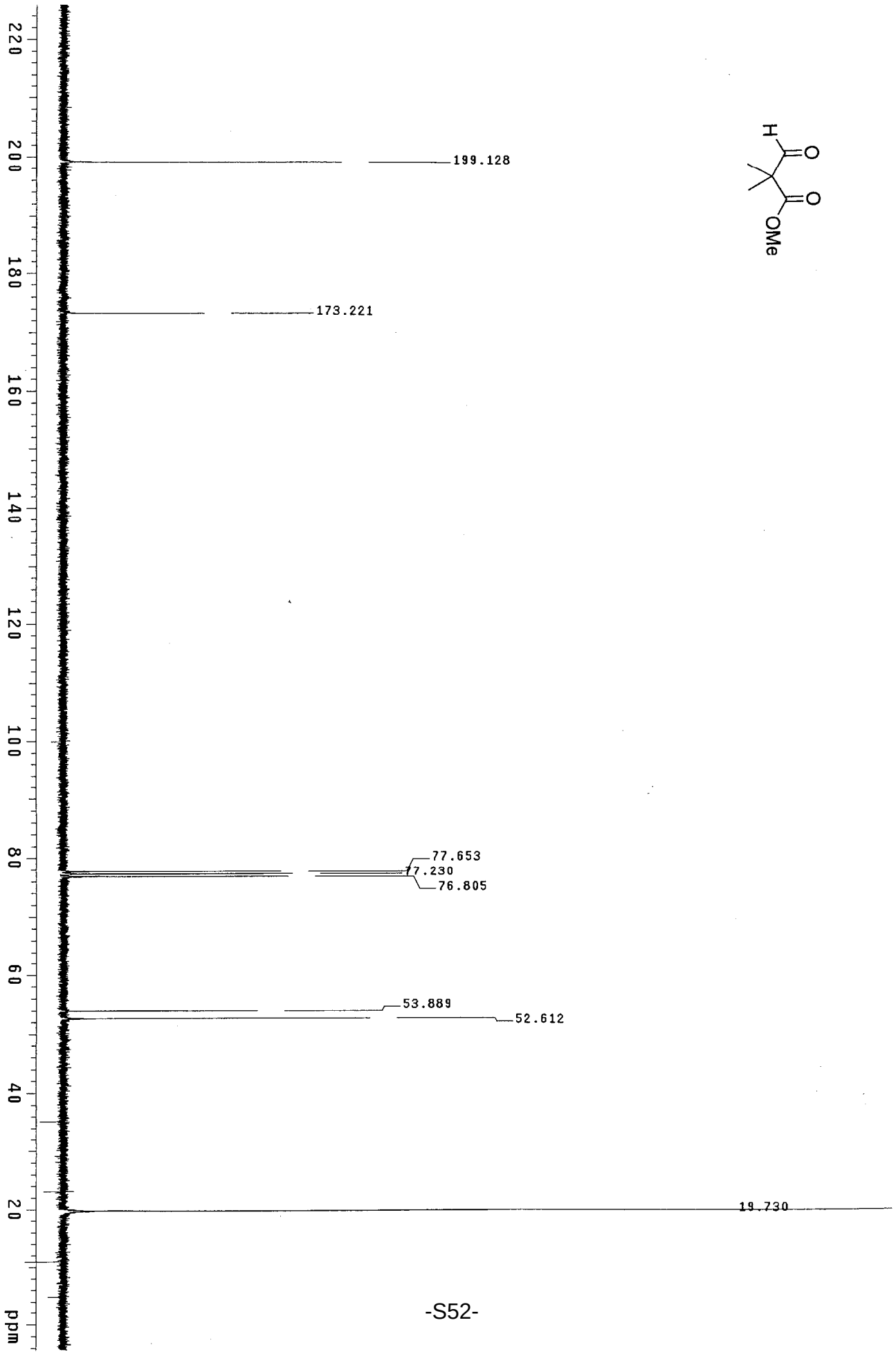
6.39
H
O
O
OMe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52.612
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220
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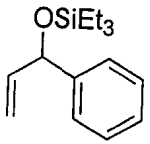
140
120
100
80
60
40
20
H
O
O
OMe

SN050690



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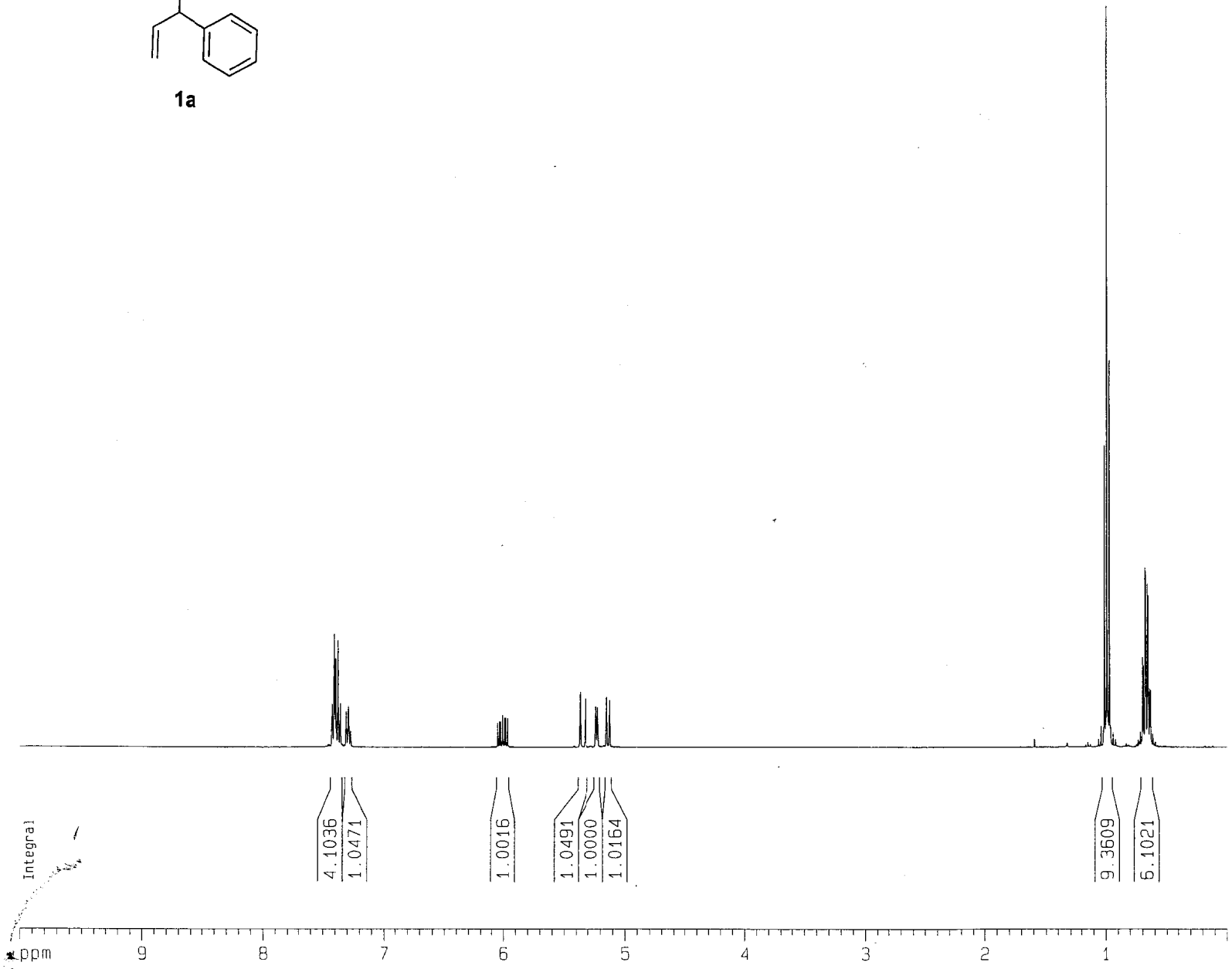

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 8278.146 Hz
FIDRES 0.126314 Hz
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RG 45.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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PL1 2.00 dB
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F2 - Processing parameters
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SSB 0
LB 0.30 Hz
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PC 1.00

1D NMR plot parameters
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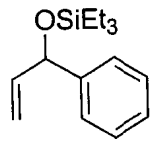
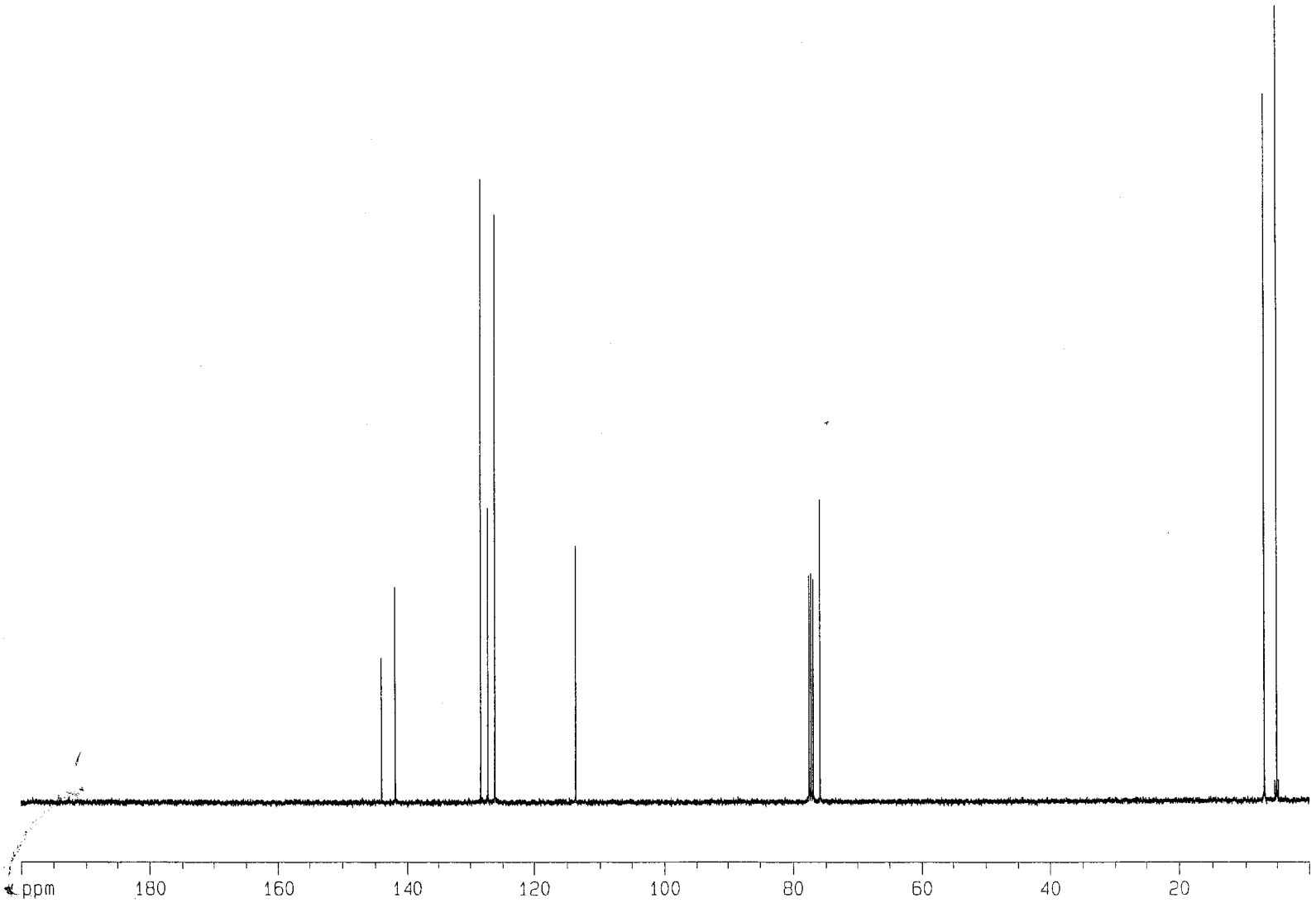
SN050690



1a

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EXPNO                      1
PROCNO                     1

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TD                   65536
SOLVENT              CDCl3
NS                     874
DS                       4
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FIDRES           0.371260 Hz
AQ               1.3468148 sec
RG                    1024
DW                 20.550 usec
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TE                 300.0 K
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============ CHANNEL f1 =============
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P1                  8.50 usec
PL1                 3.00 dB
SF01           100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 2.00 dB
PL12                22.00 dB
PL13                22.00 dB
SF02           400.1316005 MHz

F2 - Processing parameters
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SF             100.6127533 MHz
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SSB                     0
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GB                      0
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1D NMR plot parameters
CX                  20.00 cm
F1P                200.000 ppm
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HZCM           1006.12756 Hz/cm
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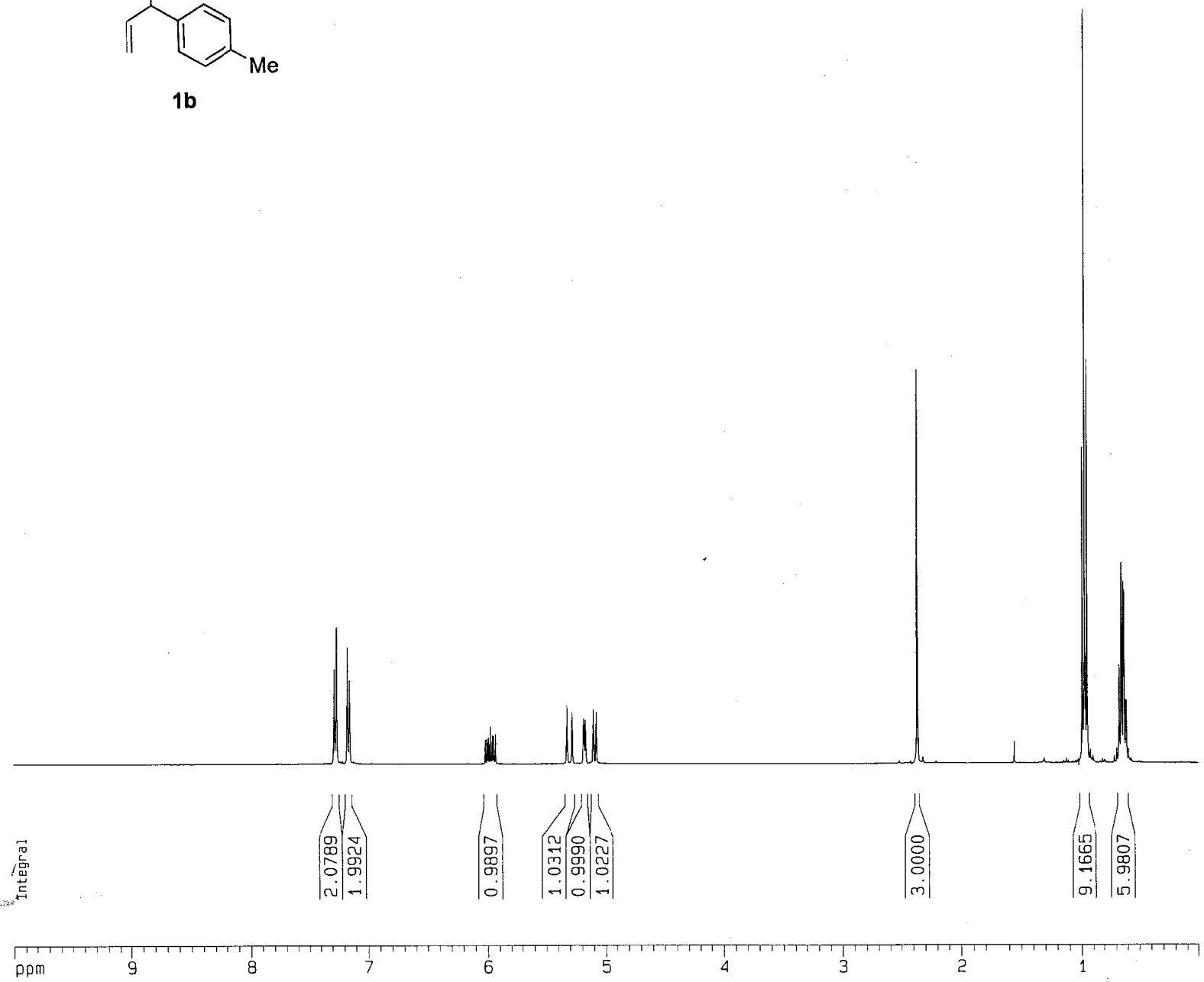
SN050714

1b



Current Data Parameters
NAME SN714-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050716
Time 21.05
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
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TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ========
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P1 7.90 usec
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F2 - Processing parameters
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GB 0
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1D NMR plot parameters
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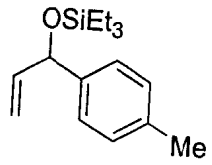
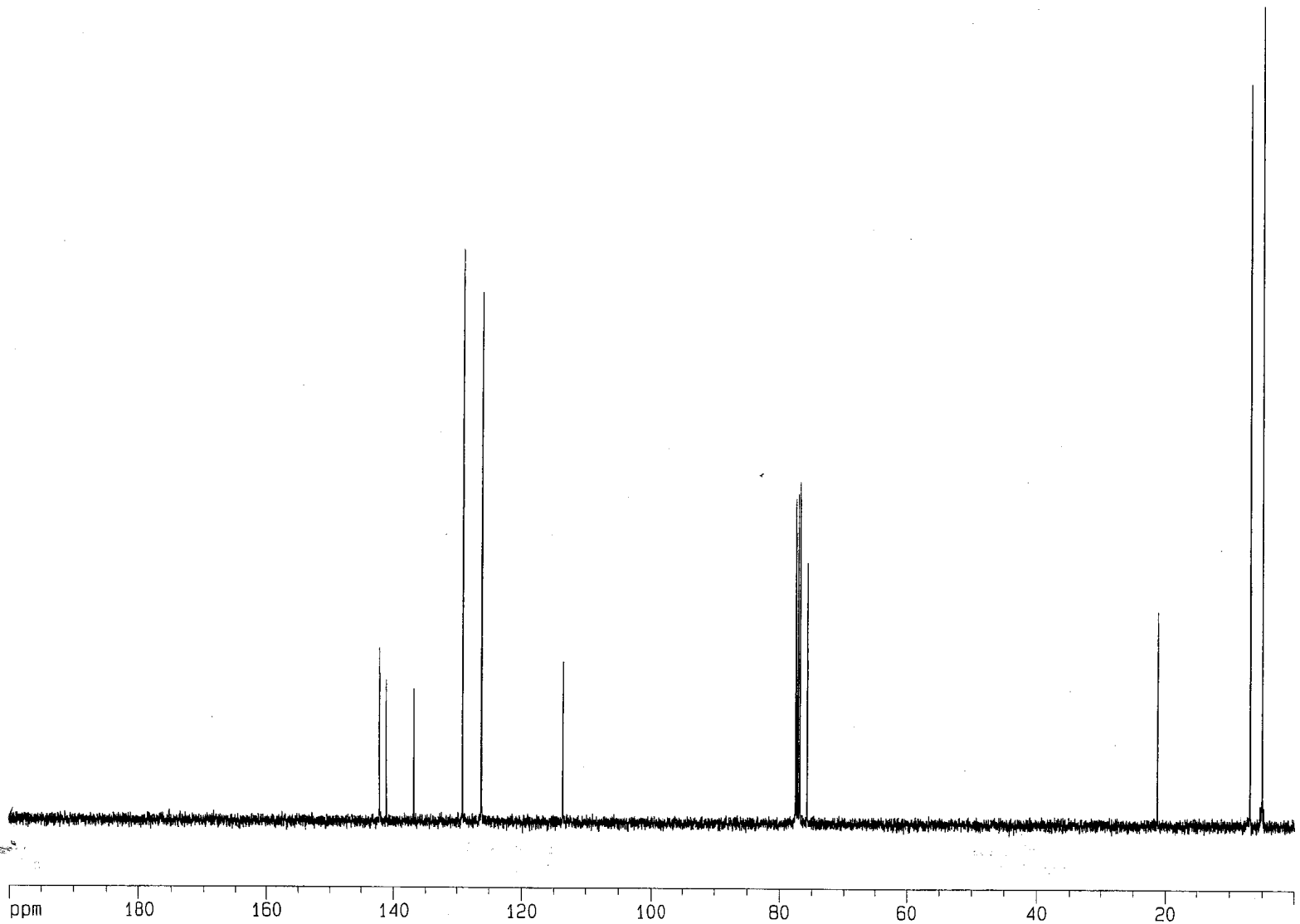
SN050714



1b



Current Data Parameters

NAME	SN714-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

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DS	4
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TE	300.0 K
D1	2.00000000 sec
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NUC1	13C
P1	15.25 usec
PL1	3.00 dB
SF01	100.6237959 MHz

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

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NUC2	1H
PCPD2	107.50 usec
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PL13	24.00 dB
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F2 - Processing parameters

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SF	100.6127492 MHz
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SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
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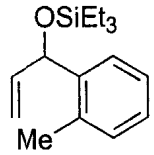

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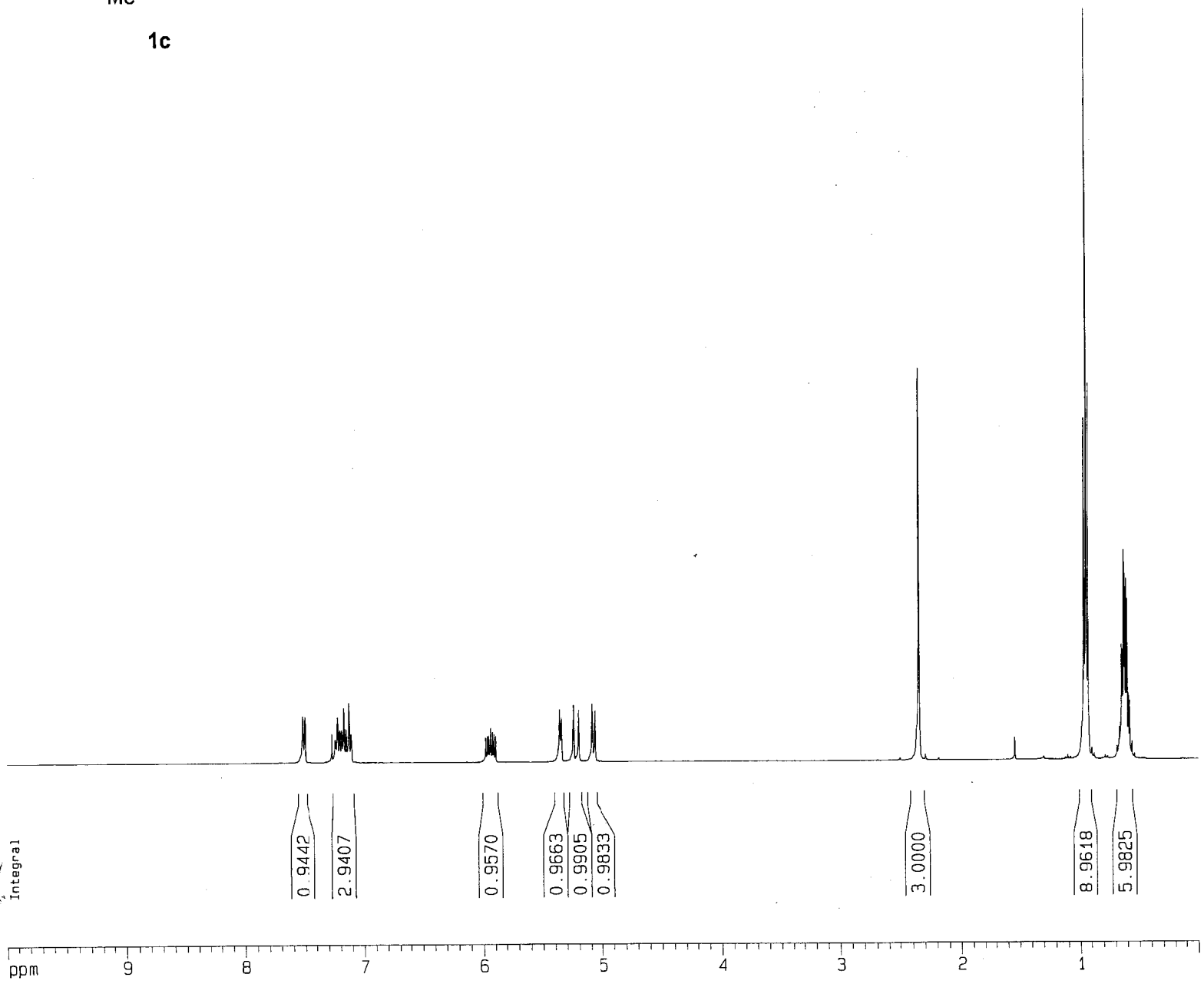
SN050715



Current Data Parameters
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EXPNO 1
PROCNO 1

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SOLVENT CDCl3
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DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
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DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ========
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P1 7.90 usec
PL1 0.00 dB
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F2 - Processing parameters
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
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1D NMR plot parameters
CX 20.00 cm
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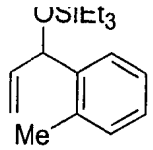

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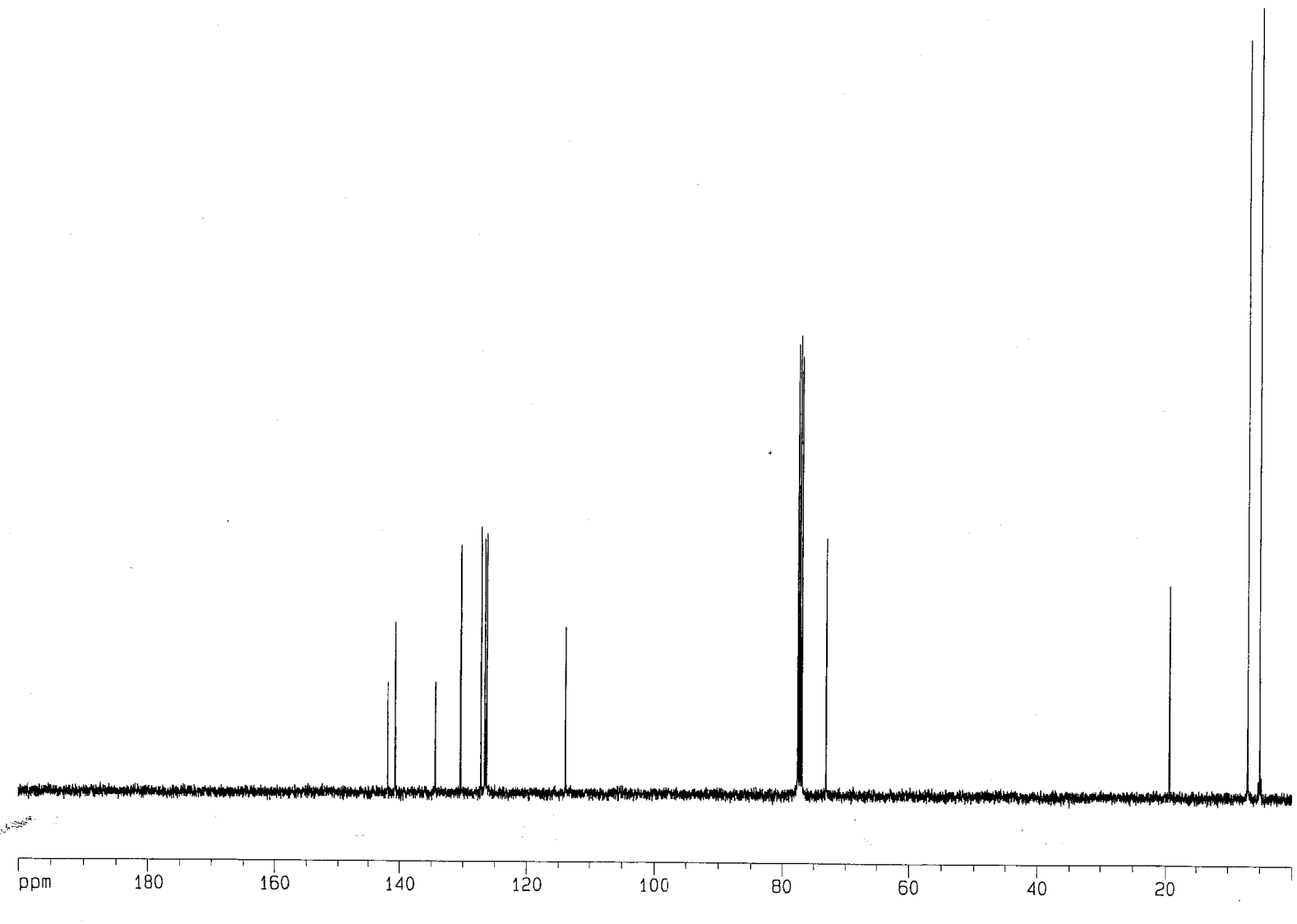
SN050715



Current Data Parameters

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PROCNO	1

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

NUC1	13C
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========== CHANNEL f2 ==========

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PL12	24.00 dB
PL13	24.00 dB
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F2 - Processing parameters

SI	32768
SF	100.6127476 MHz
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1D NMR plot parameters

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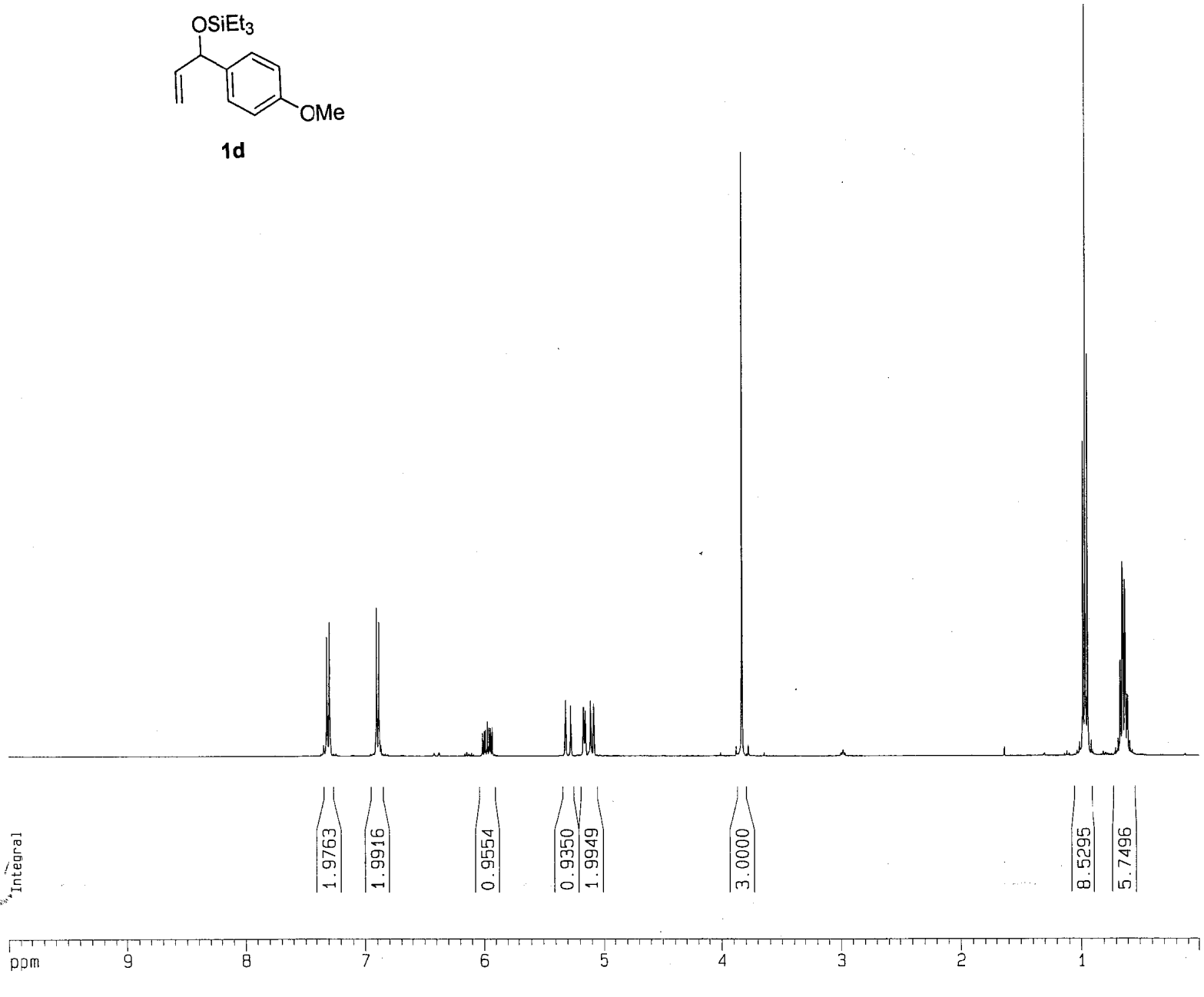
SN050729

1d



Current Data Parameters
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PROCNO 1

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PULPROG zg30
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SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ========
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GB 0
PC 1.00

1D NMR plot parameters
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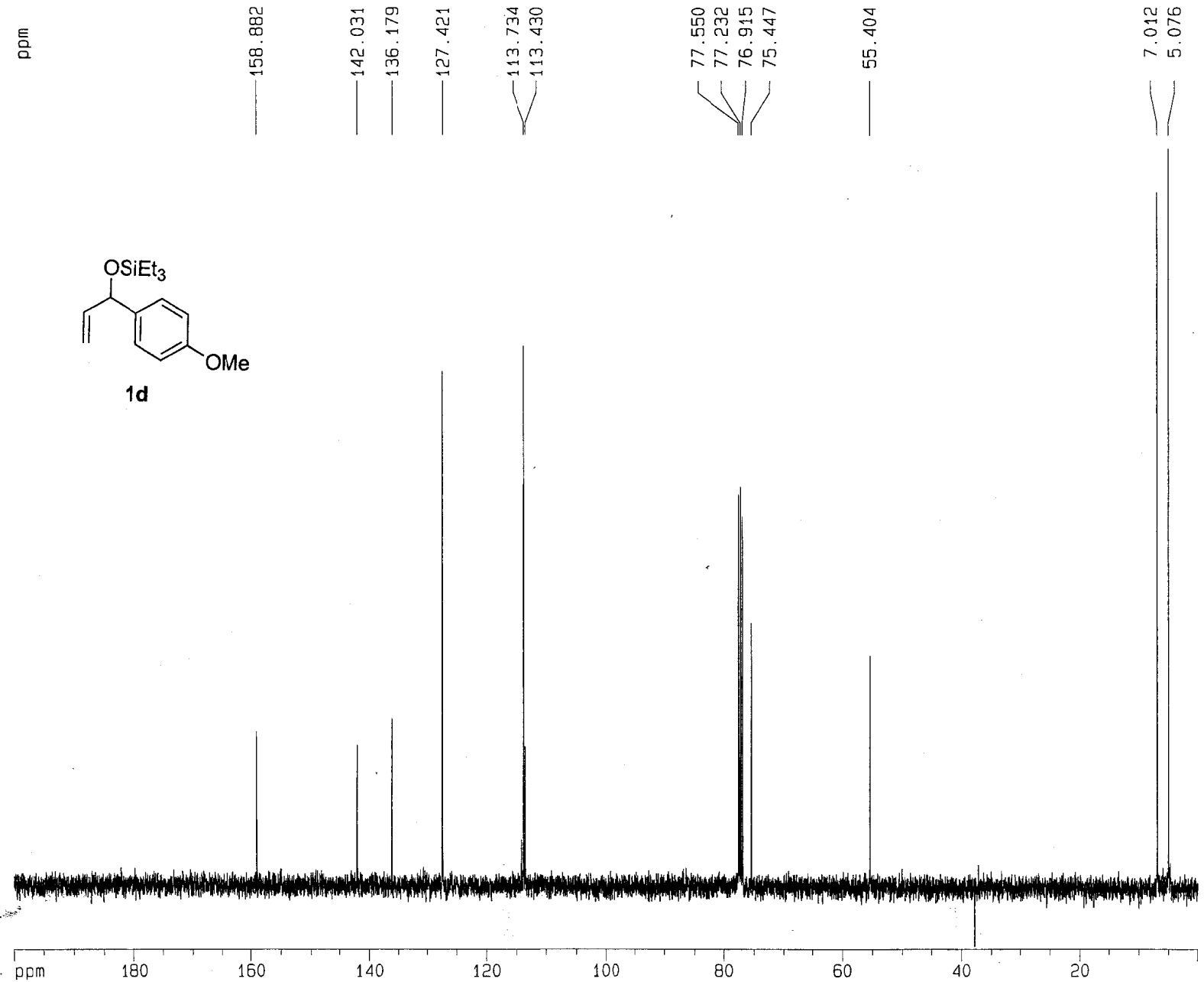
SN050729

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Current Data Parameters
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PROCNO                      1

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DS                        4
SWH               25125.629 Hz
FIDRES            0.383387 Hz
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RG                     2048
DW                   19.900 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                     15.25 usec
PL1                     3.00 dB
SFO1            100.6237959 MHz

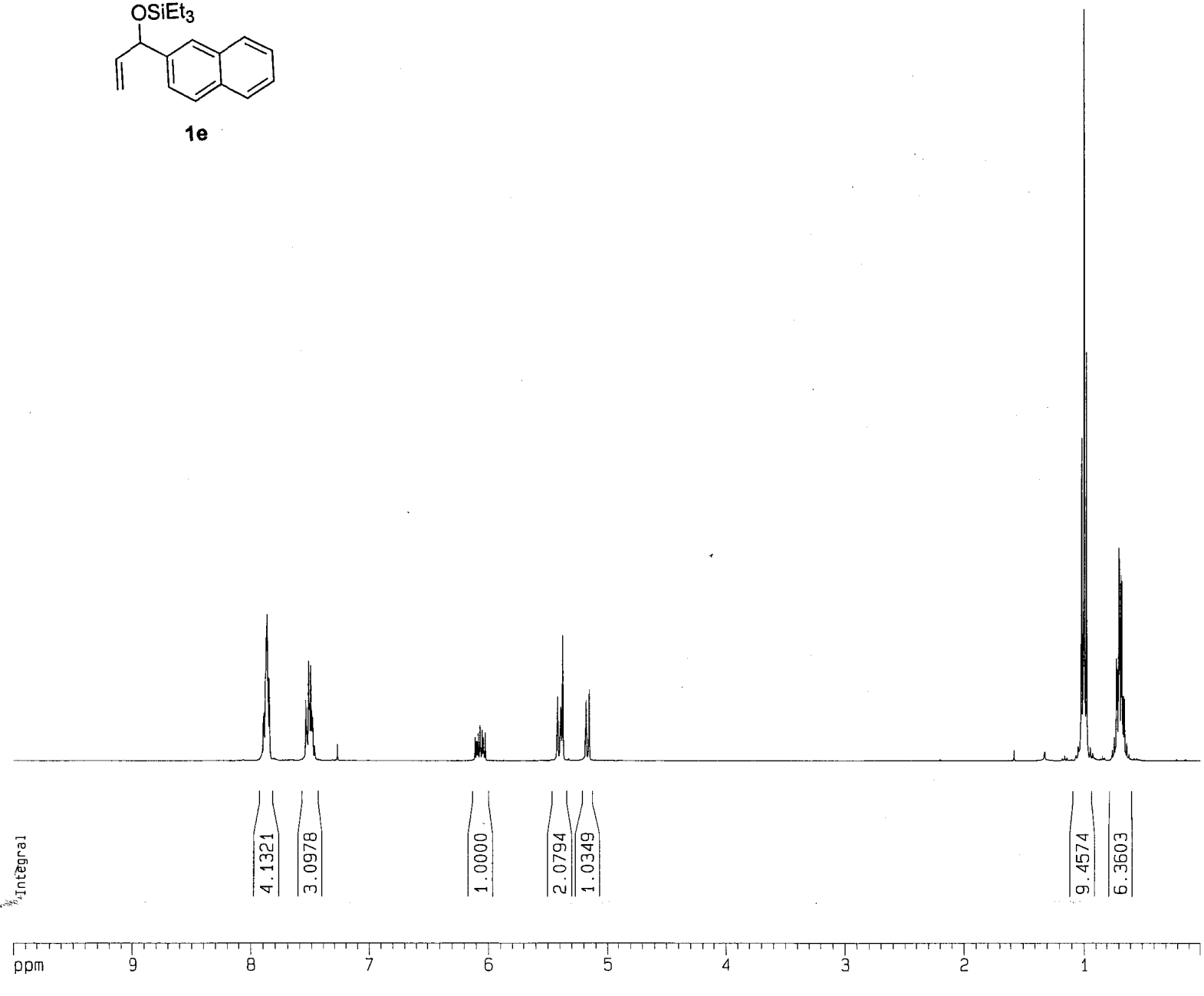
========== CHANNEL f2 ==========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                107.50 usec
PL2                     0.00 dB
PL12                   24.00 dB
PL13                   24.00 dB
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF              100.6127515 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1006.12756 Hz/cm
```

SN050723

1e



Current Data Parameters
NAME SN723-H
EXPNO 1
PROCNO 1

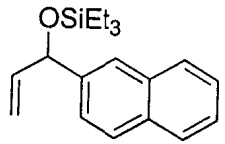
F2 - Acquisition Parameters
Date_ 20050721
Time 22.05
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

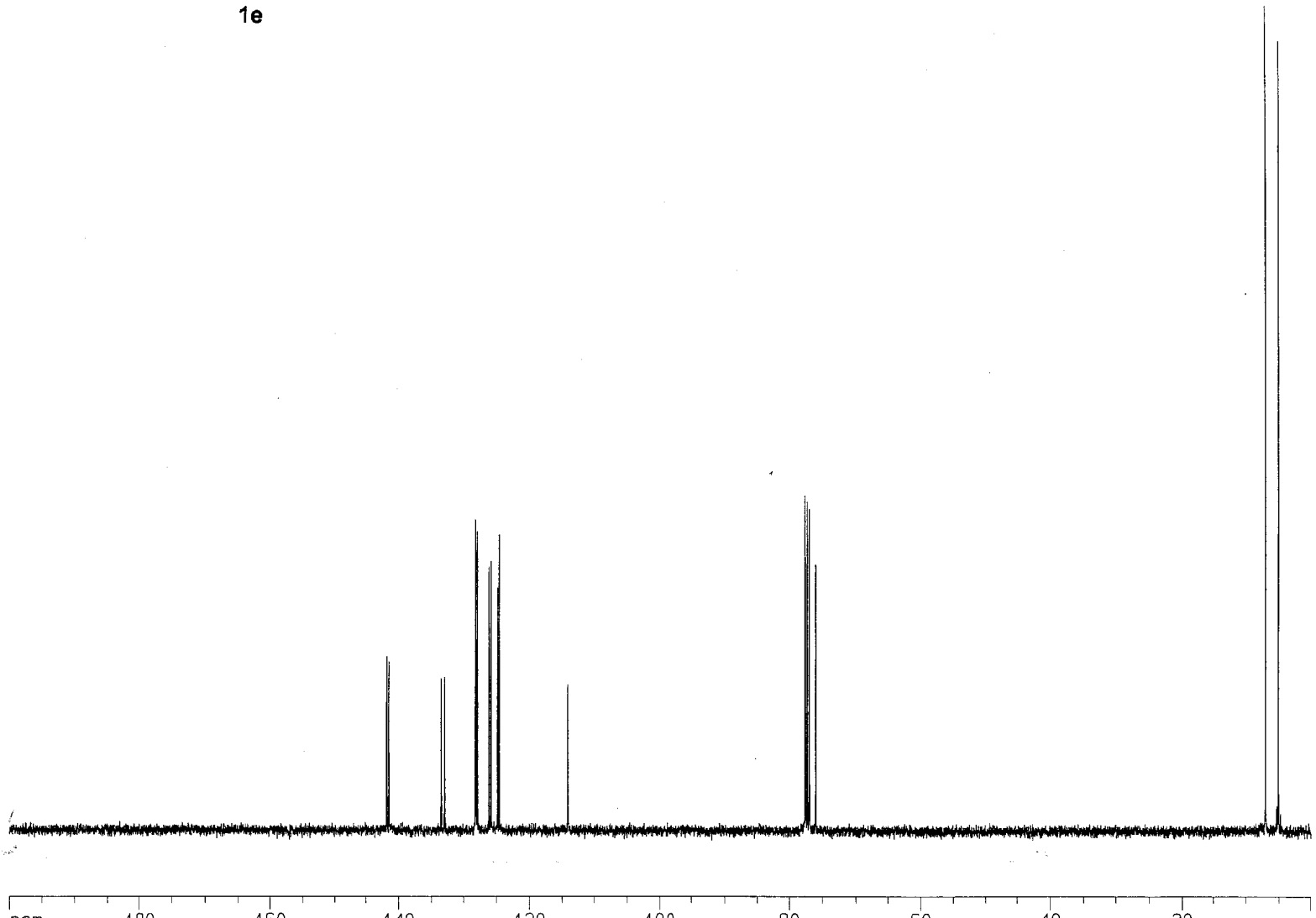
F2 - Processing parameters
SI 32768
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050723



1e



Current Data Parameters

NAME	SN723-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050721
Time	22.21
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	137
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	4096
DW	19.900 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	15.25 usec
PL1	3.00 dB
SFO1	100.6237959 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

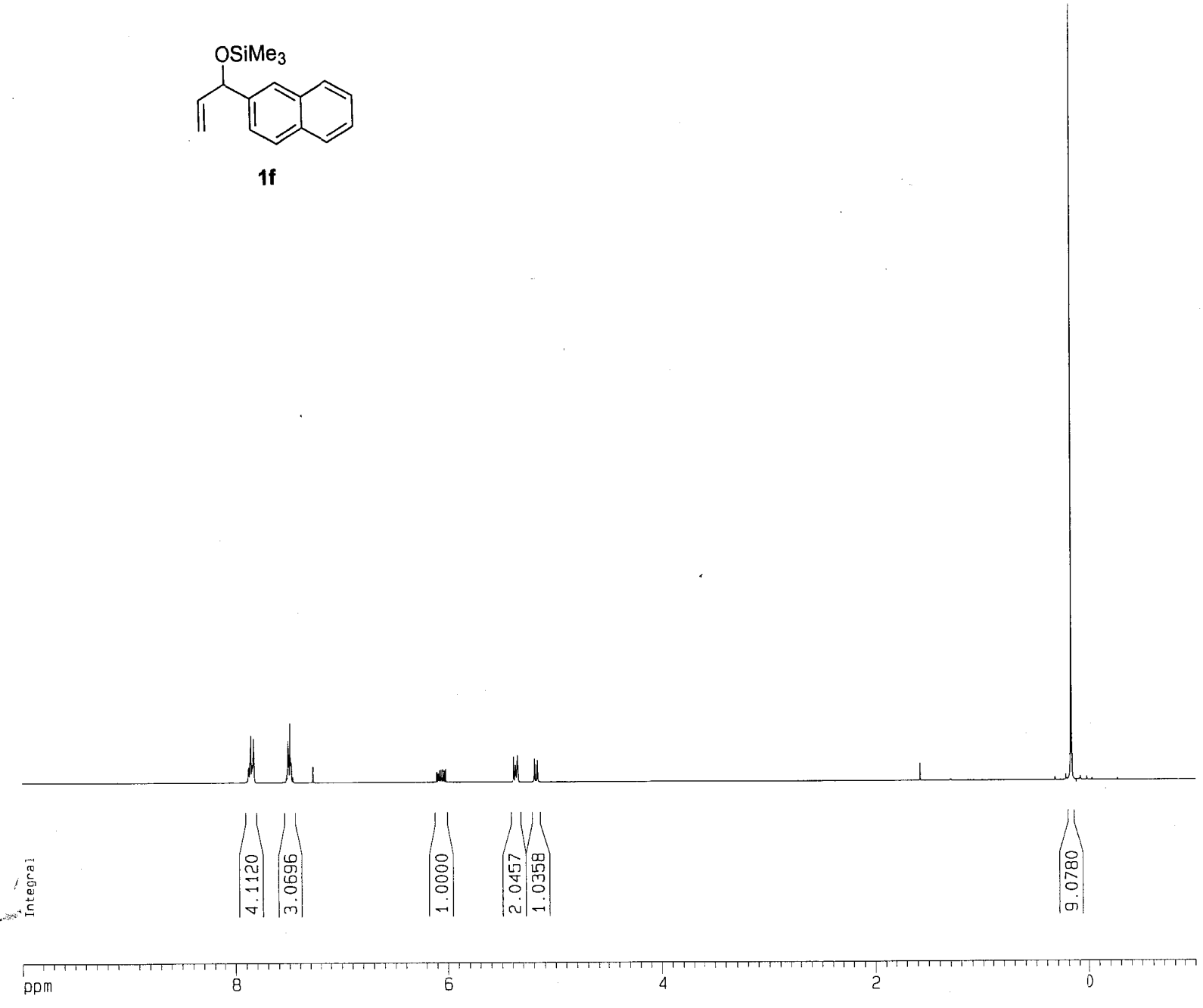
SI	32768
SF	100.6127538 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12756 Hz/cm

SN050740

1f



Current Data Parameters
NAME SN740-H
EXPNO 1
PROCNO 1

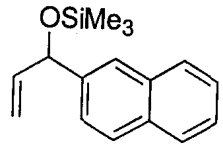
F2 - Acquisition Parameters
Date_ 20050729
Time 22.03
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

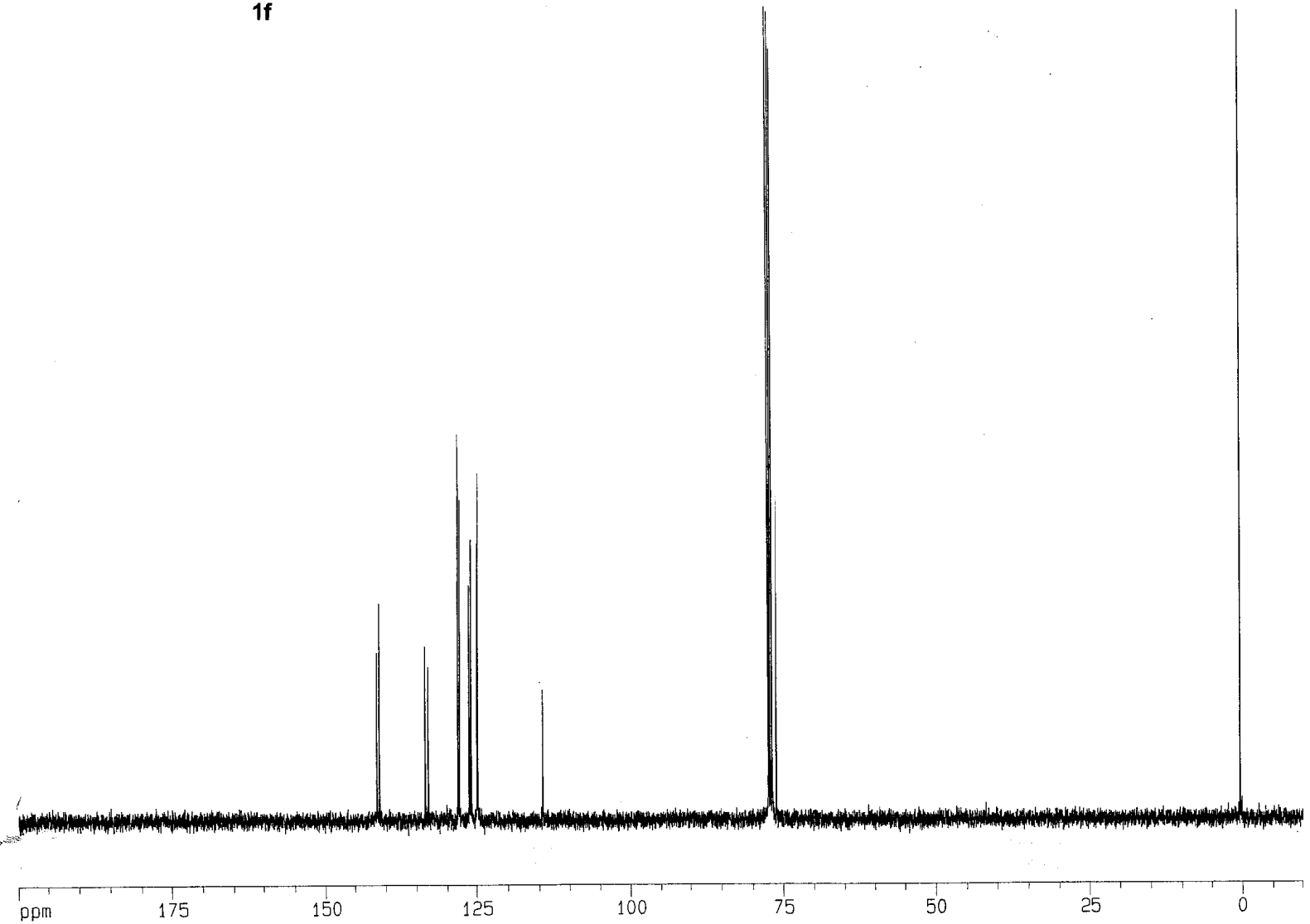
F2 - Processing parameters
SI 32768
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

SN050740



1f



Current Data Parameters

NAME	SN740-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050729
Time	22.05
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	201
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	16384
DW	19.900 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	15.25 usec
PL1	3.00 dB
SFO1	100.6237959 MHz

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

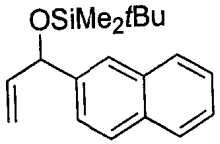
CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127522 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

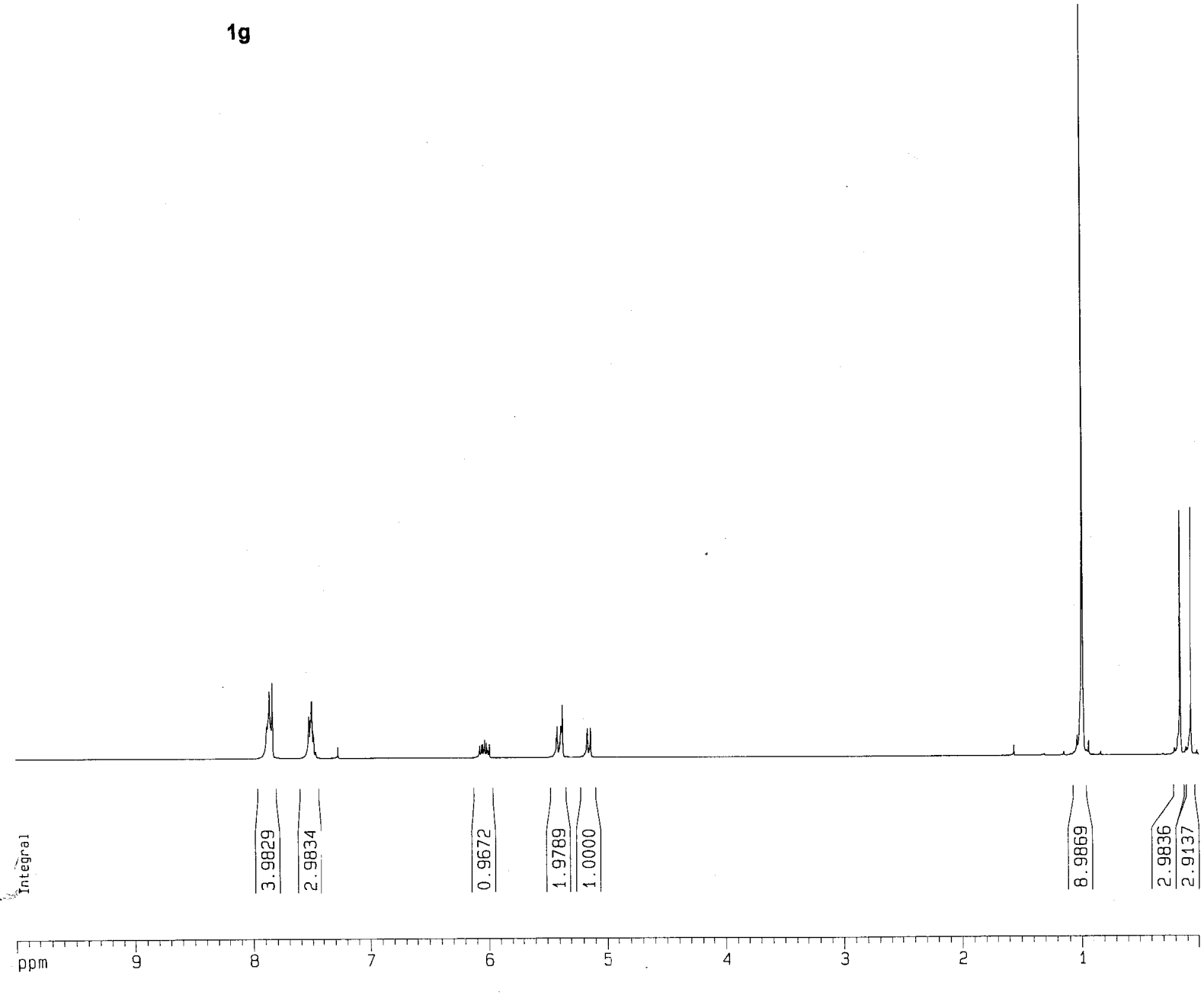
1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	10.50000 ppm/cm
HZCM	1056.43396 Hz/cm



1g

SN050737



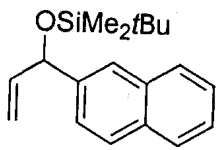
Current Data Parameters
NAME SN737-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050727
Time 22.18
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 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

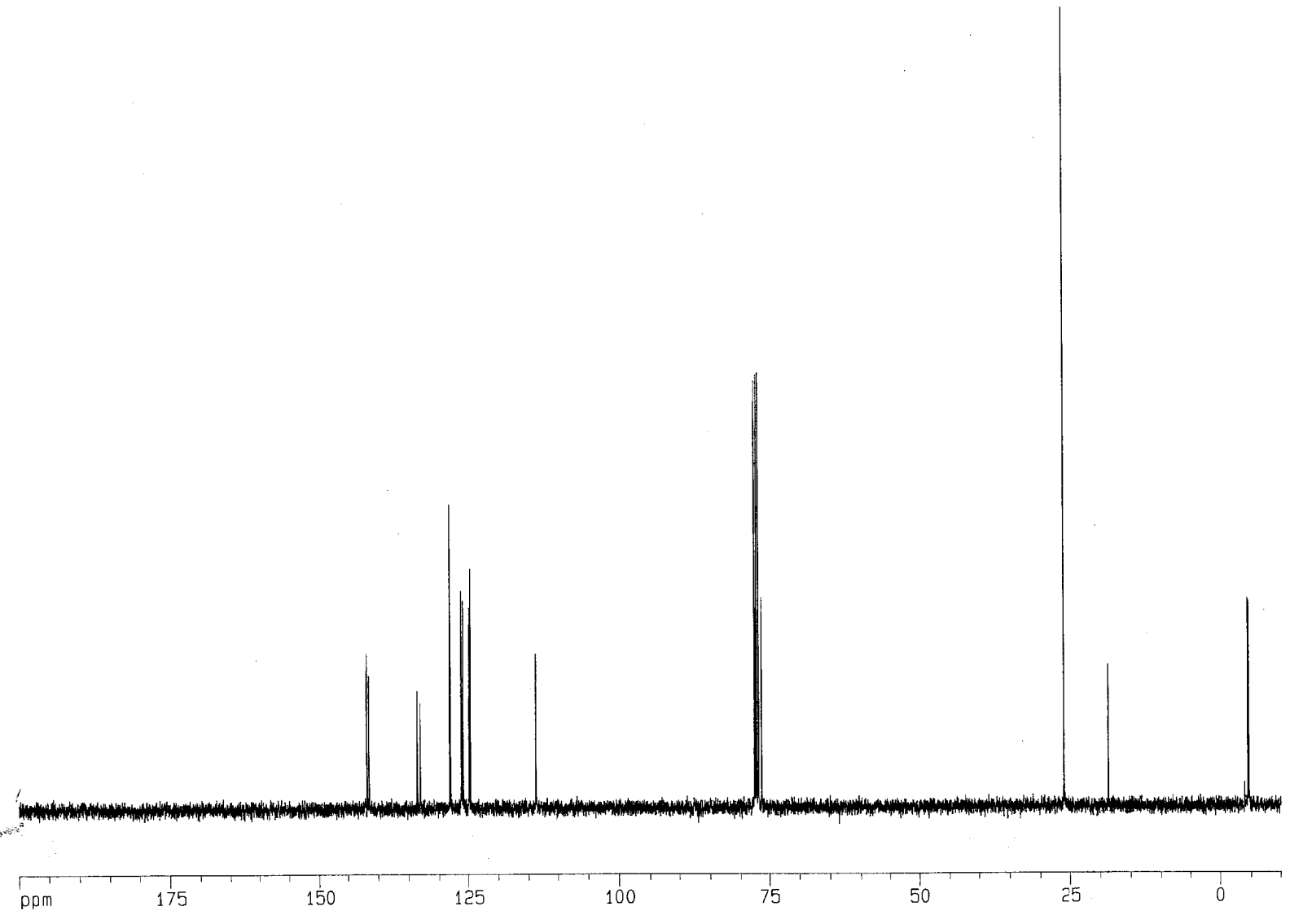
F2 - Processing parameters
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm



1g

SN050737




```
Current Data Parameters
NAME                  SN737-C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20050727
Time                  22.25
INSTRUM               spect
PROBHD      5mm BBO BB-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     151
DS                       4
SWH               25125.629 Hz
FIDRES            0.383387 Hz
AQ                1.3042164 sec
RG                    3251
DW                  19.900 usec
DE                   6.00 usec
TE                  300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

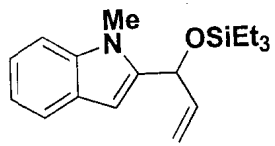
========== CHANNEL f1 ==========
NUC1                   13C
P1                   15.25 usec
PL1                   3.00 dB
SFO1          100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               107.50 usec
PL2                   0.00 dB
PL12                 24.00 dB
PL13                 24.00 dB
SFO2          400.1316005 MHz

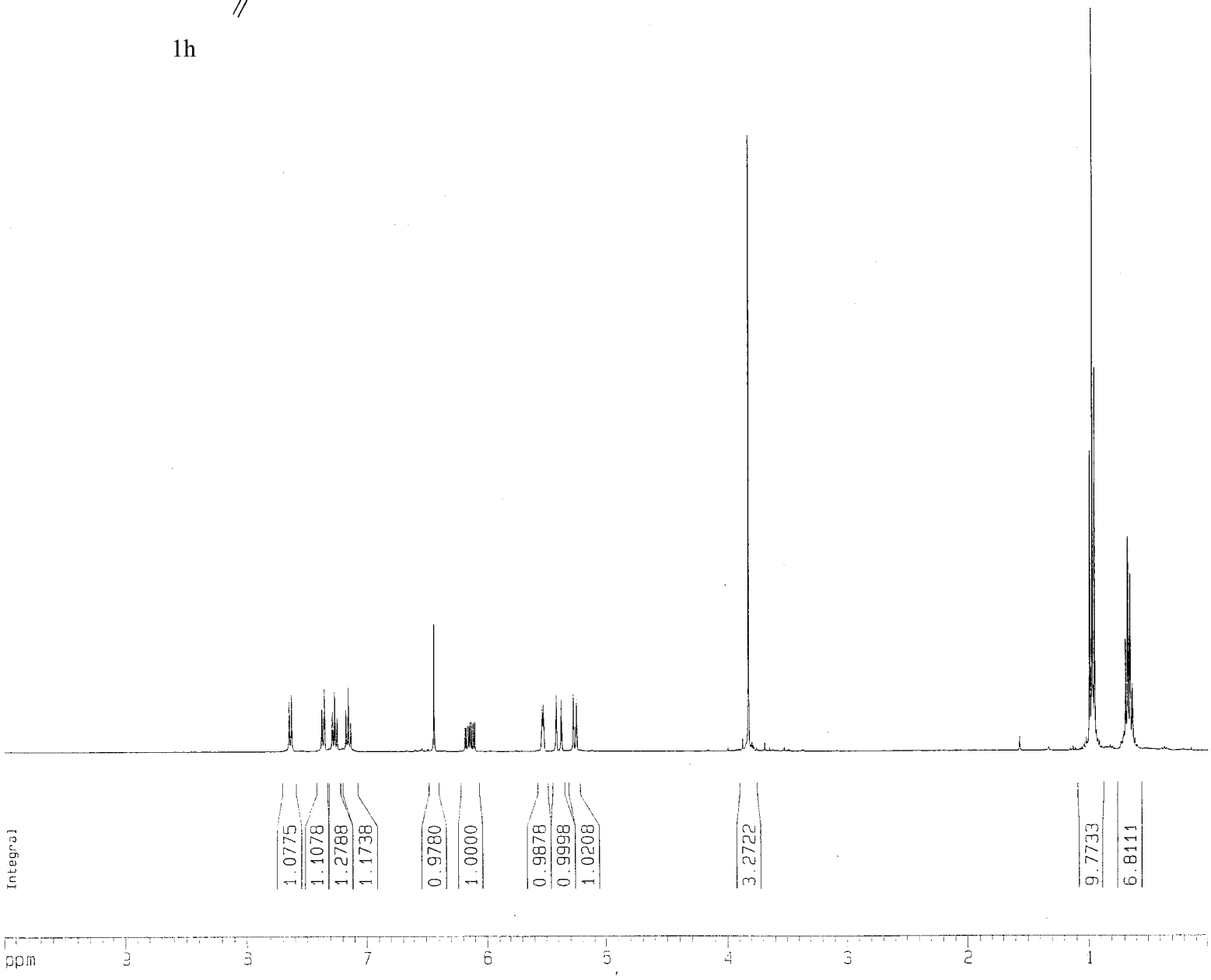
F2 - Processing parameters
SI                    32768
SF            100.6127530 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 200.000 ppm
F1                20122.55 Hz
F2P                -10.000 ppm
F2                -1006.13 Hz
PPMCM              10.50000 ppm/cm
HZCM            1056.43396 Hz/cm
```

SN061116 mixture



1h



Current Data Parameters
NAME SN1116-H3
EXPNO 1
PROCNO 1

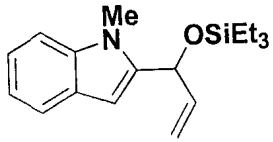
F2 - Acquisition Parameters
Date_ 20060331
Time 20.18
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

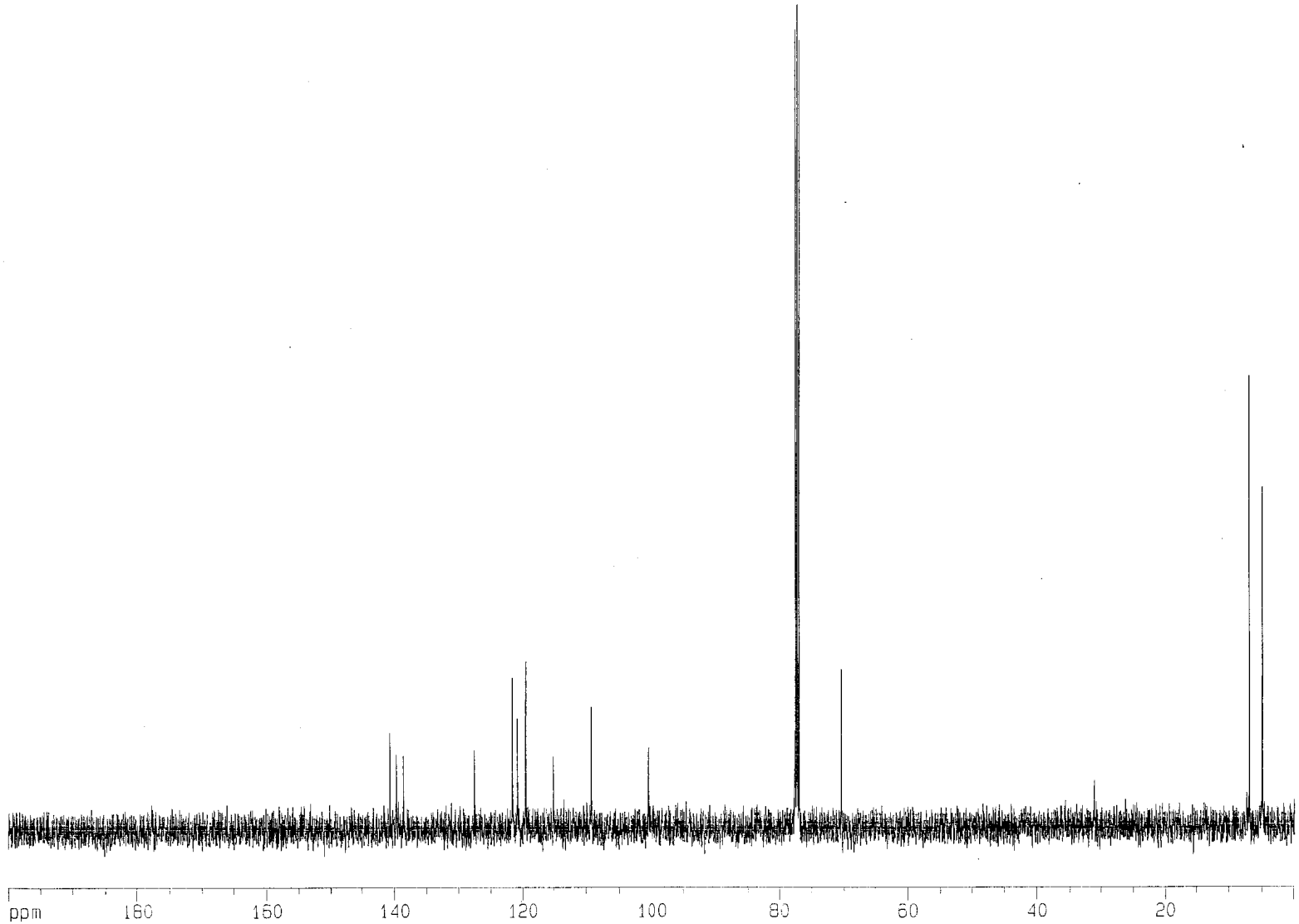
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN061116 crude



1h




```
Current Data Parameters
NAME                 SN1116-C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20060331
Time                   19.32
INSTRUM                spect
PROBHD      5mm BBO BB-1
PULPROG             zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        512
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       2048
DW                   19.900 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                      15.25 usec
PL1                      3.00 dB
SFO1             100.6237959 MHz

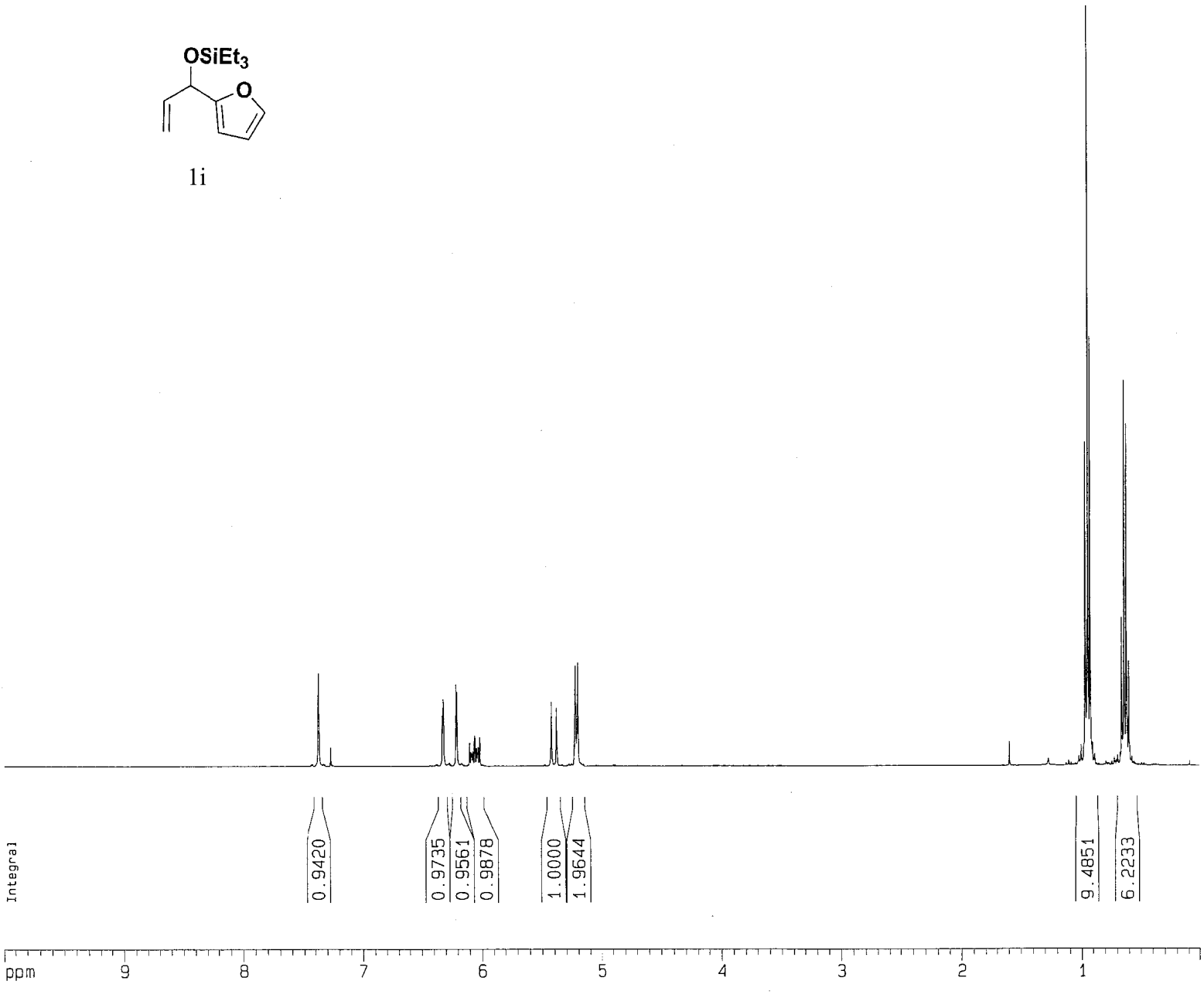
============ CHANNEL f2 ============
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 107.50 usec
PL2                      0.00 dB
PL12                    24.00 dB
PL13                    24.00 dB
SFO2             400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF               100.6127492 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                     0.000 ppm
F2                      0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1006.12744 Hz/cm
```

SN050721

1i



Current Data Parameters
NAME SN721-maj-H
EXPNO 1
PROCNO 1

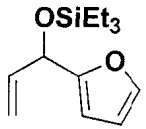
F2 - Acquisition Parameters
Date_ 20050720
Time 12.24
INSTRUM spect
PROBHD 5mm EBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

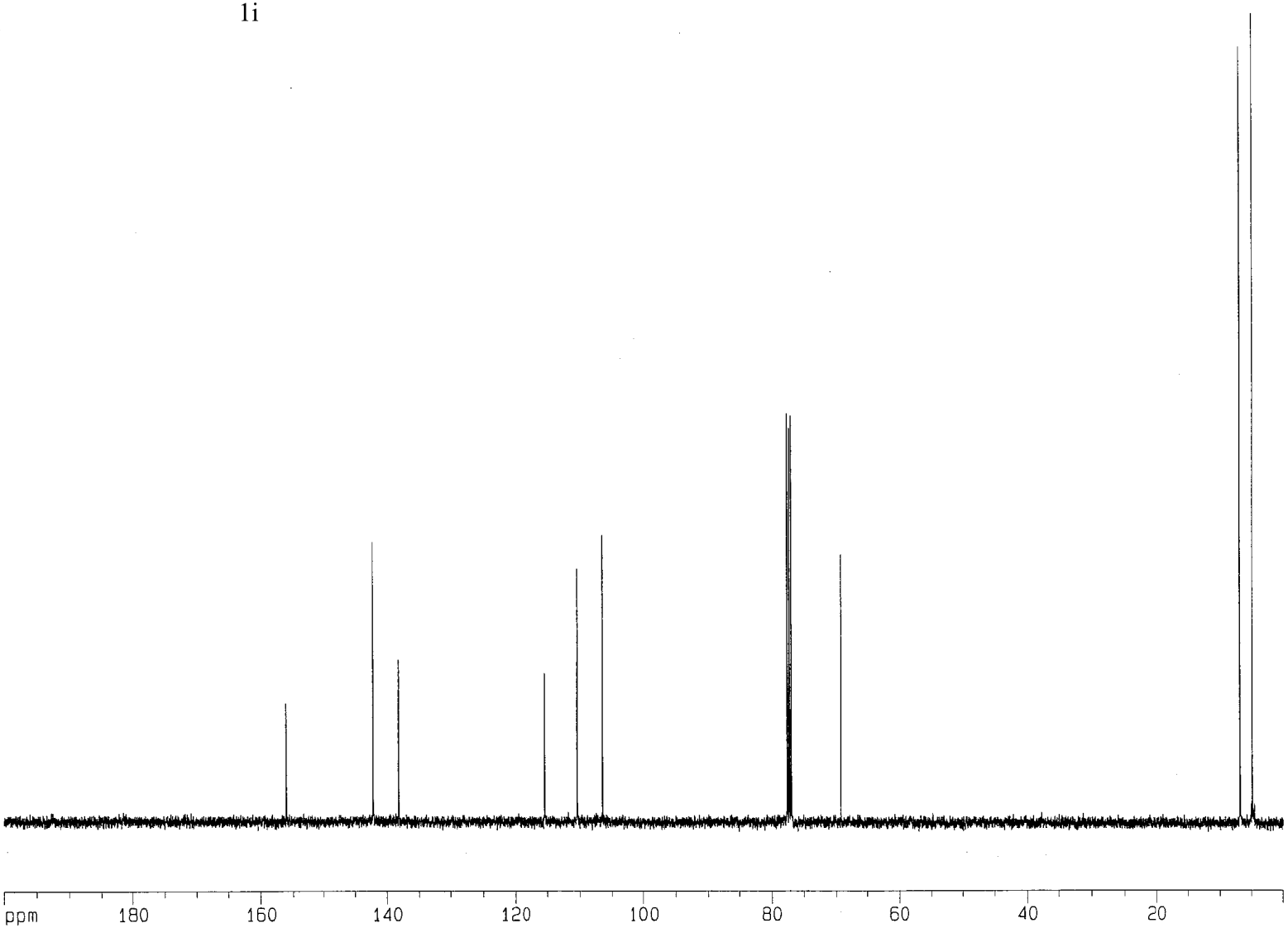
F2 - Processing parameters
SI 32768
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050721 major



1i



Current Data Parameters

NAME	SN721-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050720
Time	12.31
INSTRUM	spect
PROBHD	5mm EBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	140
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	2048
DW	19.900 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	15.25 usec
PL1	3.00 dB
SF01	100.6237959 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SF02	400.1316005 MHz

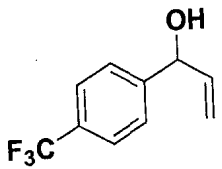
F2 - Processing parameters

SI	32768
SF	100.6127499 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

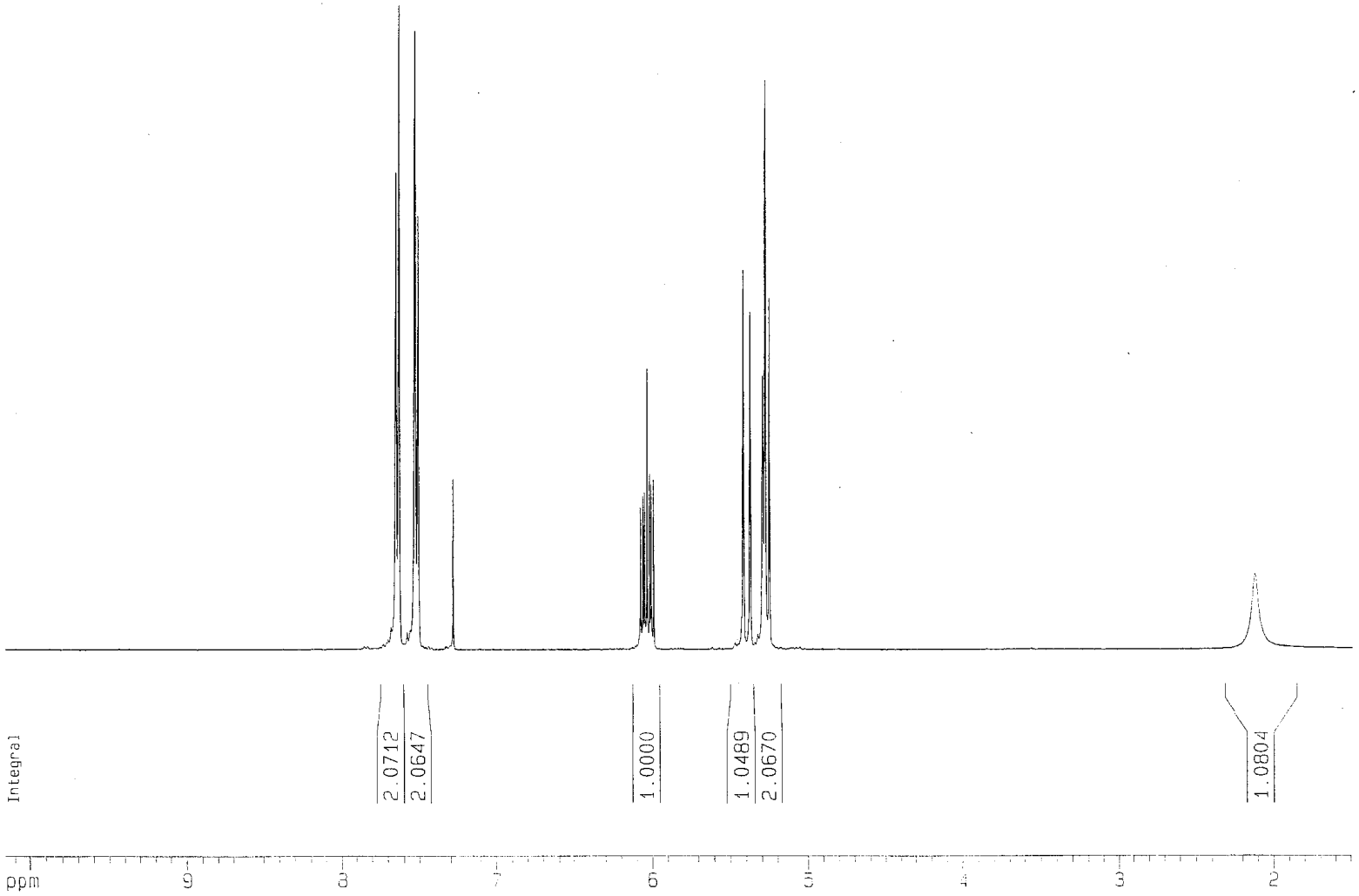
1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12744 Hz/cm

SN061118 OH



Desilylated 1j



Current Data Parameters

NAME	SN1118-OH-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060403
Time	20.32
INSTRUM	spect
PROBHD	5mm B30 BB-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	203.2
DW	60.400 usec
DE	6.00 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

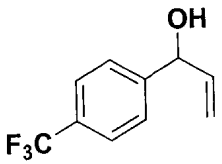
F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

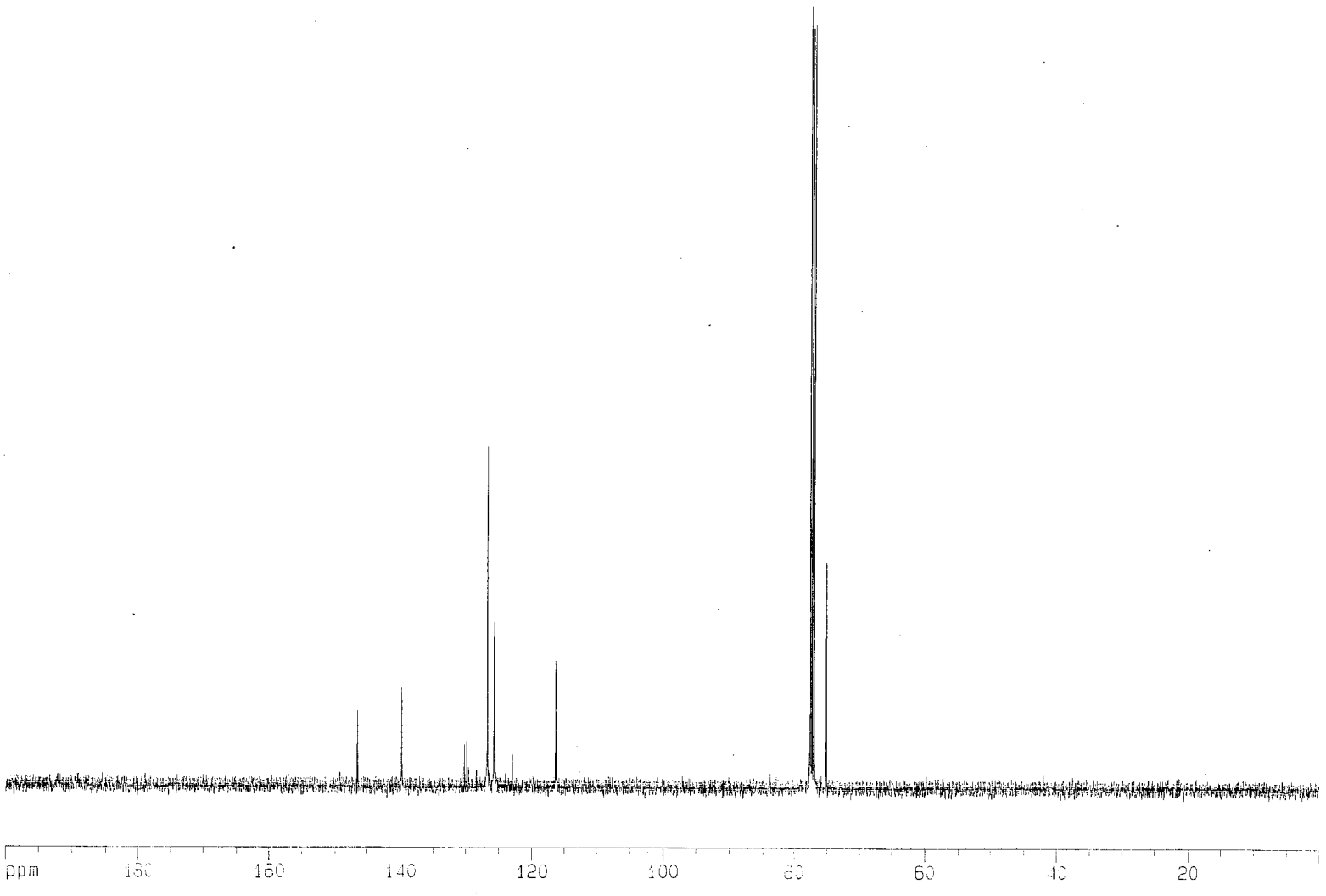
1D NMR plot parameters

CX	20.00 cm
F1P	10.165 ppm
F1	4067.20 Hz
F2P	1.490 ppm
F2	596.32 Hz
PPMCM	0.43372 ppm/cm
HZCM	173.54408 Hz/cm

SN061118 OH



Desilylated 1j




```
Current Data Parameters
NAME          SN1118-OH-C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20060403
Time               20.22
INSTRUM            spect
PROBHD      5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  946
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042154 sec
RG                1625.5
DW               19.900 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                15.25 usec
PL1                3.00 dB
SFO1         100.6237959 MHz

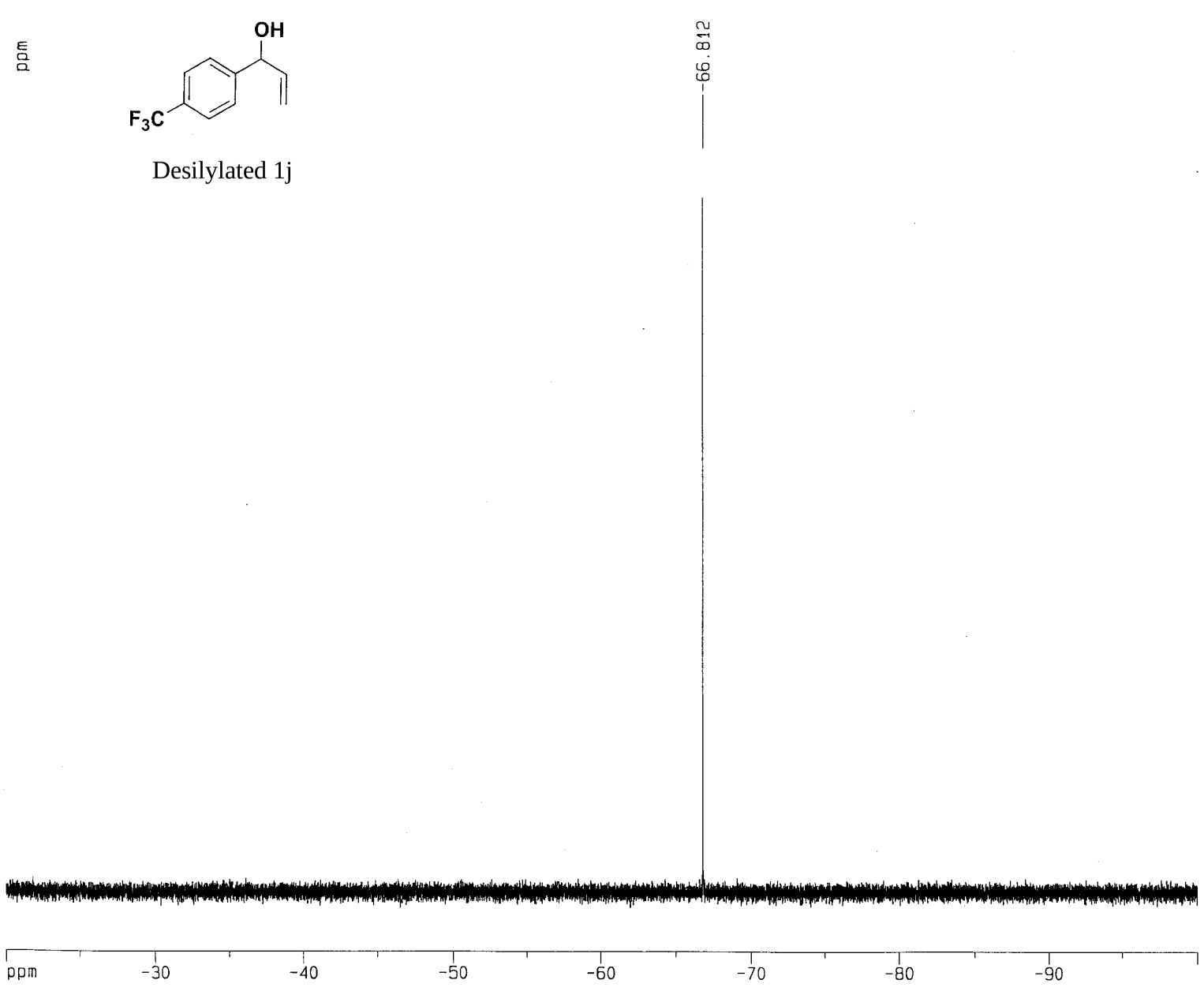
============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2           107.50 usec
PL2                0.00 dB
PL12              24.00 dB
PL13              24.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127492 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                 20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          15.00000 ppm/cm
HZCM         1006.12744 Hz/cm
```

SN061118 OH

Desilylated 1j



Current Data Parameters
NAME SN1118-OH-F
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060403
Time 18.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgflqn
TD 131072
SOLVENT C6D6
NS 28
DS 4
SWH 75187.969 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 6502
DW 6.650 usec
DE 6.00 usec
TE 291.5 K
D1 1.00000000 sec

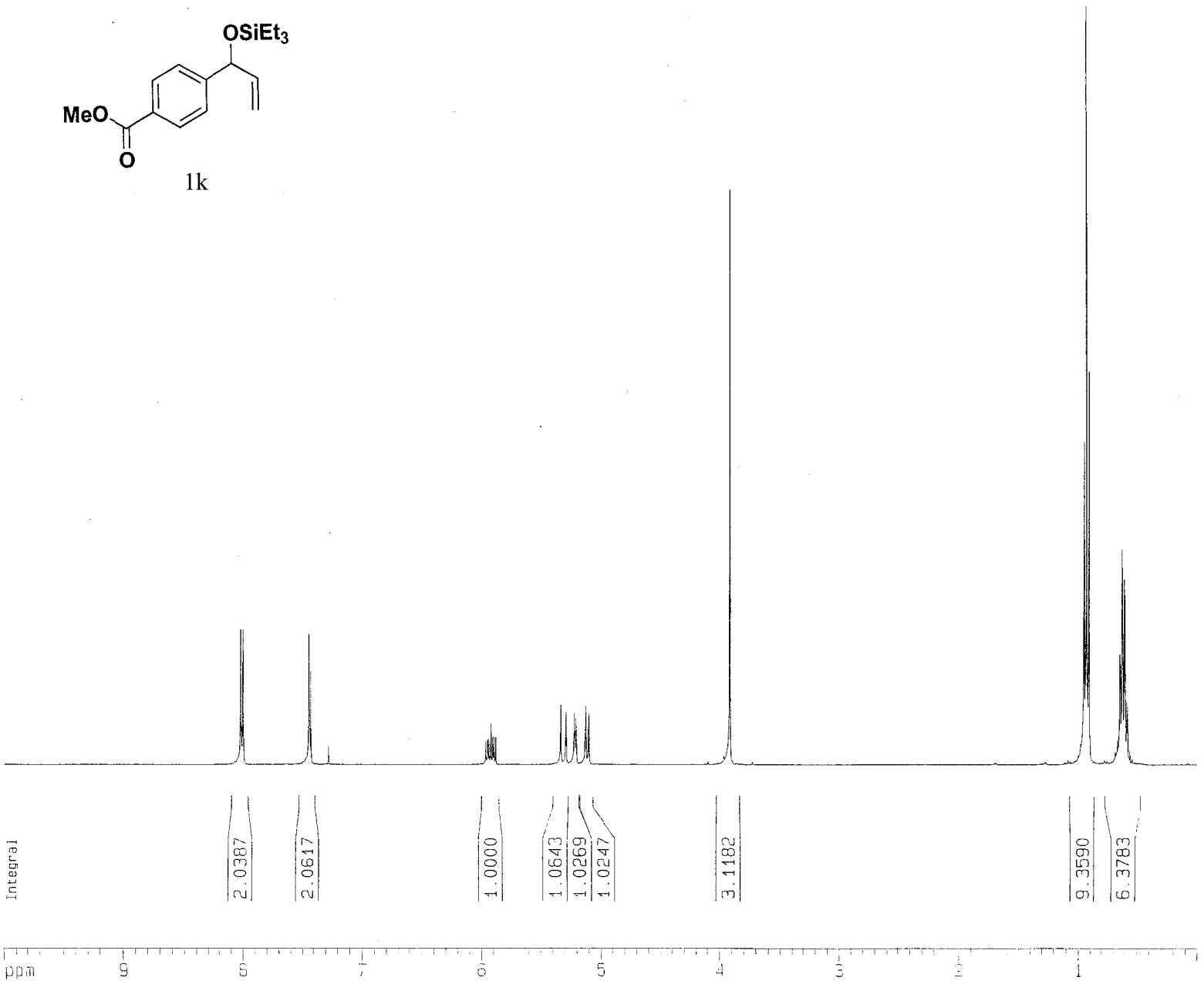
======== CHANNEL f1 ========
NUC1 19F
P1 19.50 usec
PL1 -4.00 dB
SFO1 376.4607044 MHz

F2 - Processing parameters
SI 65536
SF 376.5000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P -20.000 ppm
F1 -7530.00 Hz
F2P -100.000 ppm
F2 -37650.00 Hz
PPMCM 4.00000 ppm/cm
HZCM 1506.00000 Hz/cm

SN061117

1k



Current Data Parameters
NAME SN1117-H2
EXPNO 1
PROCNO 1

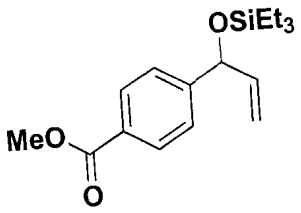
F2 - Acquisition Parameters
Date_ 20060403
Time 18.10
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

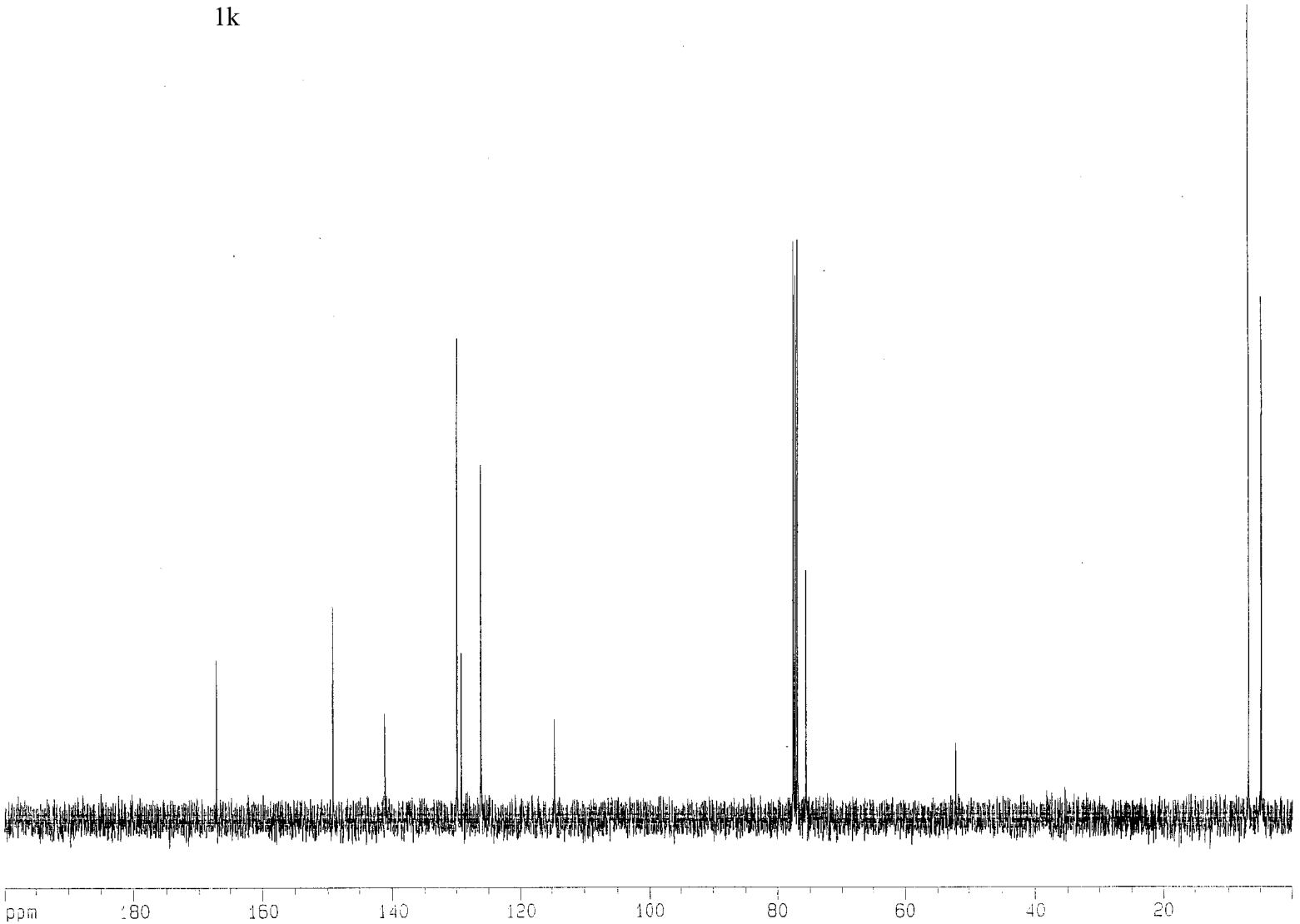
F2 - Processing parameters
SI 32768
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN061117



1k



Current Data Parameters
NAME SN1117-C2
EXPNO 1
PROCNO 1

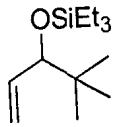
F2 - Acquisition Parameters
Date_ 20060403
Time 18.13
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2048
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

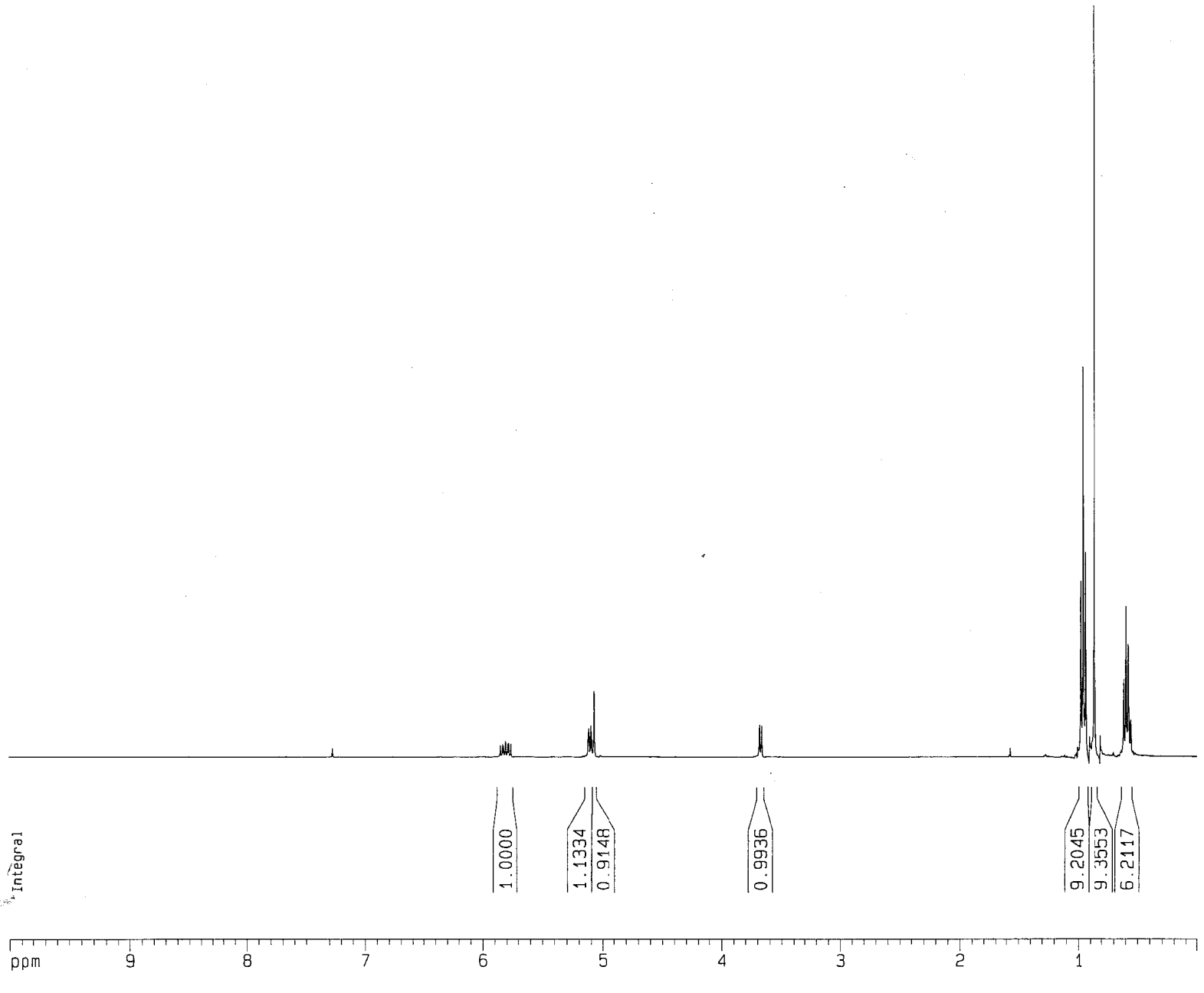
F2 - Processing parameters
SI 32768
SF 100.6127515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12756 Hz/cm



11

SN050733



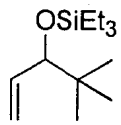
Current Data Parameters
NAME SN733-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050727
Time 22.00
INSTRUM spect
PROBHD 5mm B30 BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 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

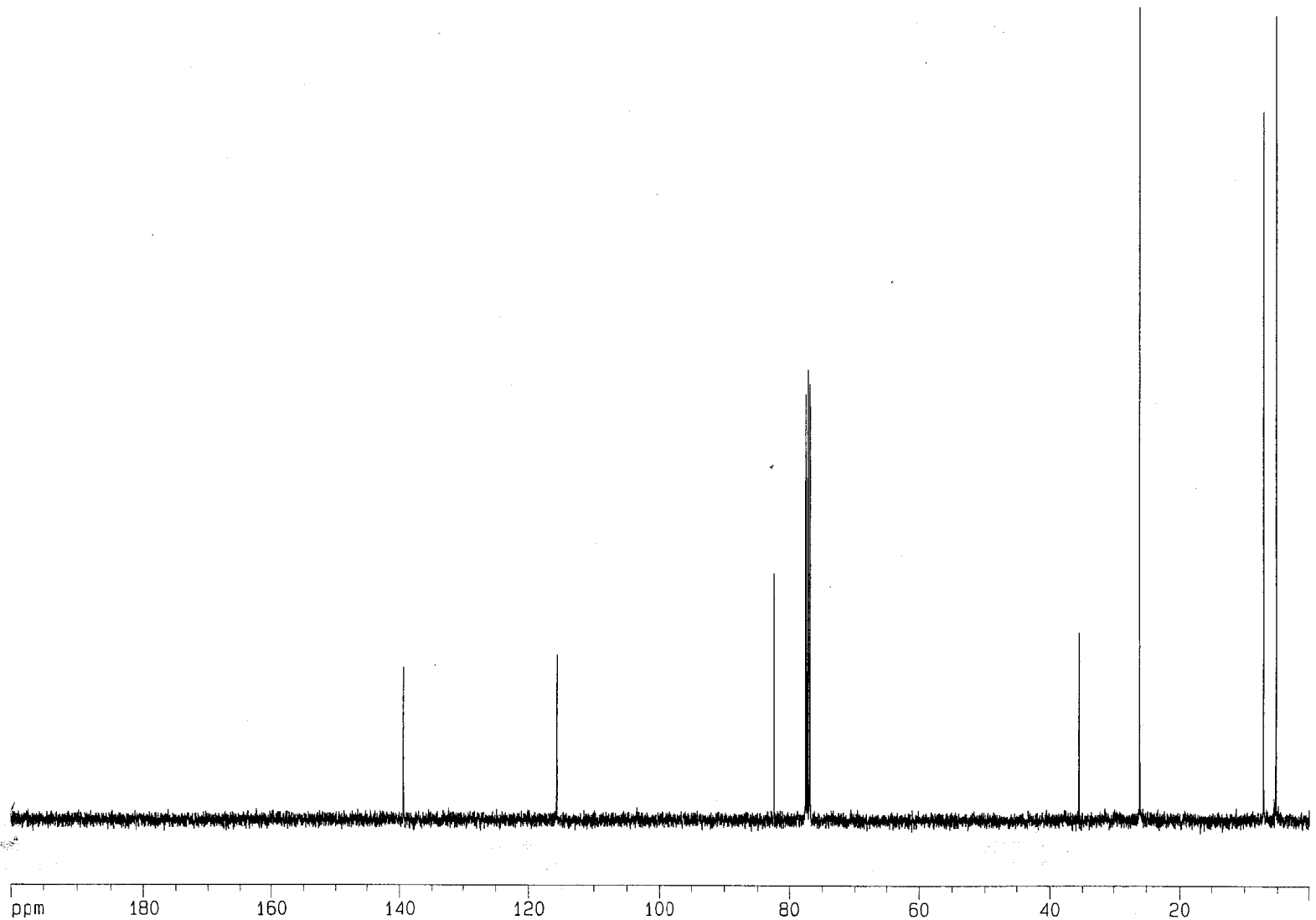
F2 - Processing parameters
SI 32768
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm



11

SN050733



Current Data Parameters
NAME SN733-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050727
Time 22.05
INSTRUM spect
PROBHD 5mm BB0 BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 132
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 4096
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

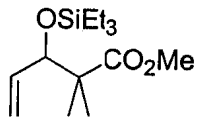
========== CHANNEL f1 ==========
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

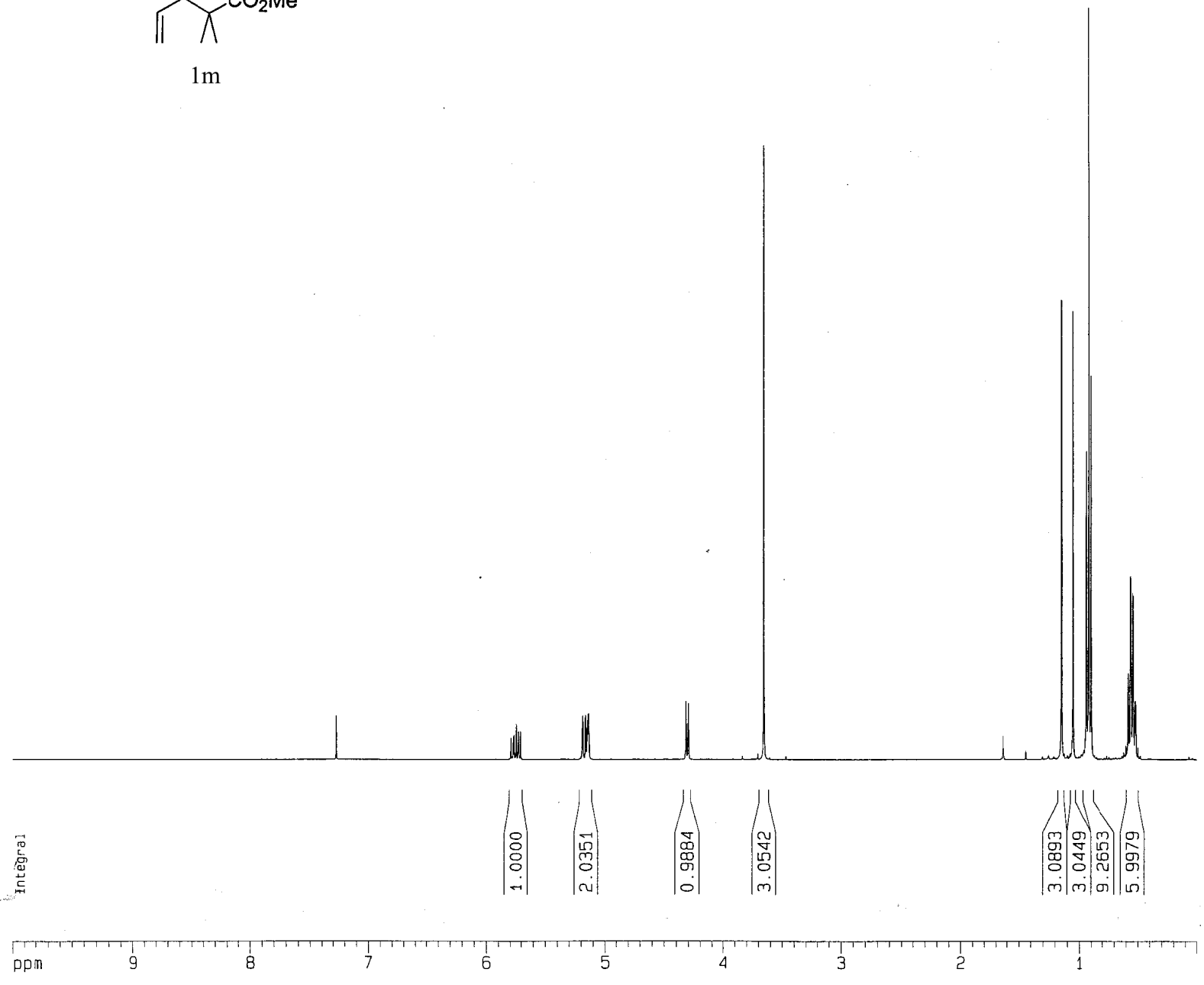
F2 - Processing parameters
SI 32768
SF 100.6127469 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm

SN050745



1m



Current Data Parameters
NAME SN745-H
EXPNO 1
PROCNO 1

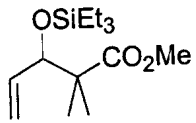
F2 - Acquisition Parameters
Date_ 20050801
Time 5.13
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

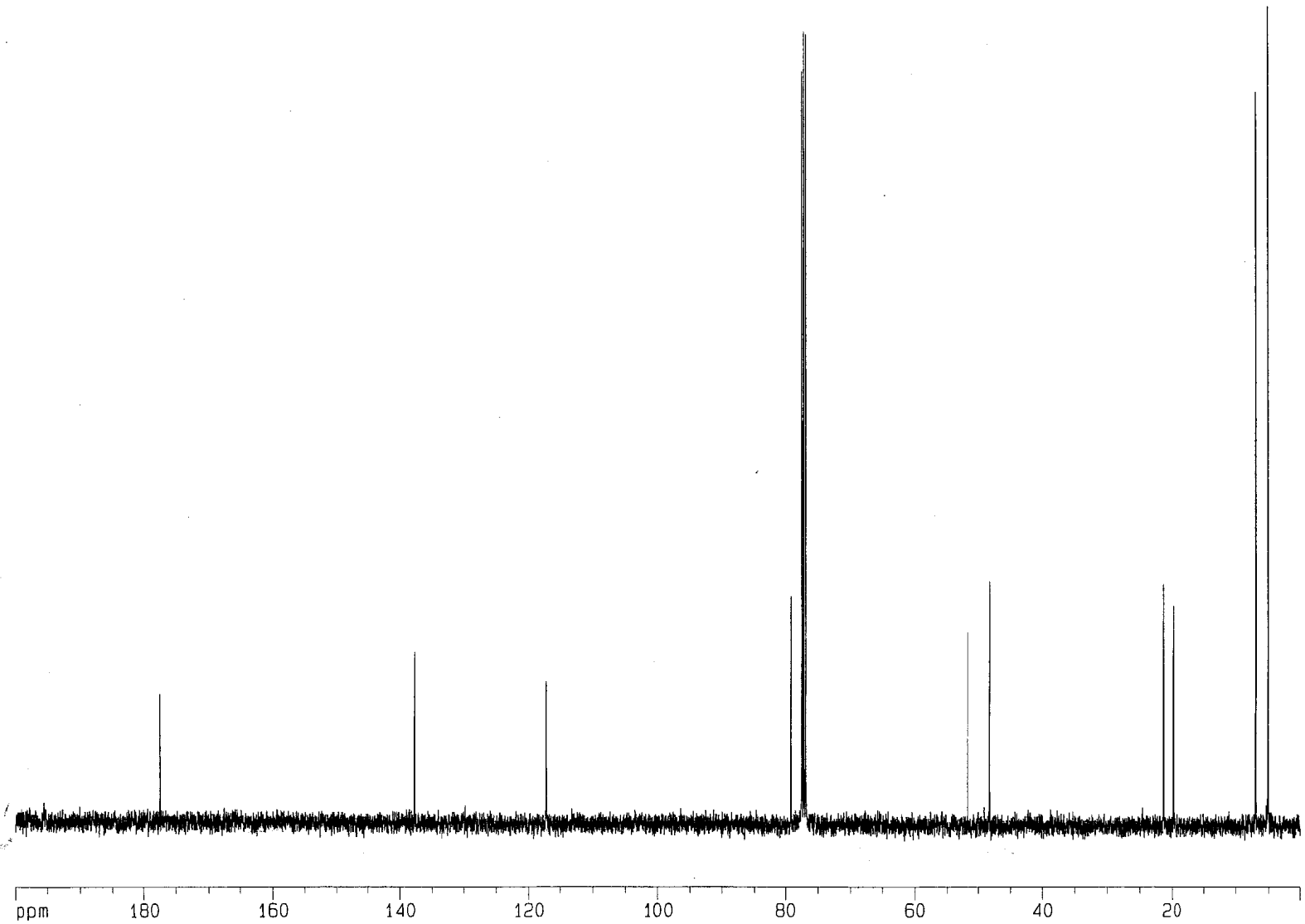
F2 - Processing parameters
SI 32768
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050745



1m



Current Data Parameters
NAME SN745-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050801
Time 5.22
INSTRUM spect
PROBHD 5mm B30 BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 150
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 4096
DW 19.900 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 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

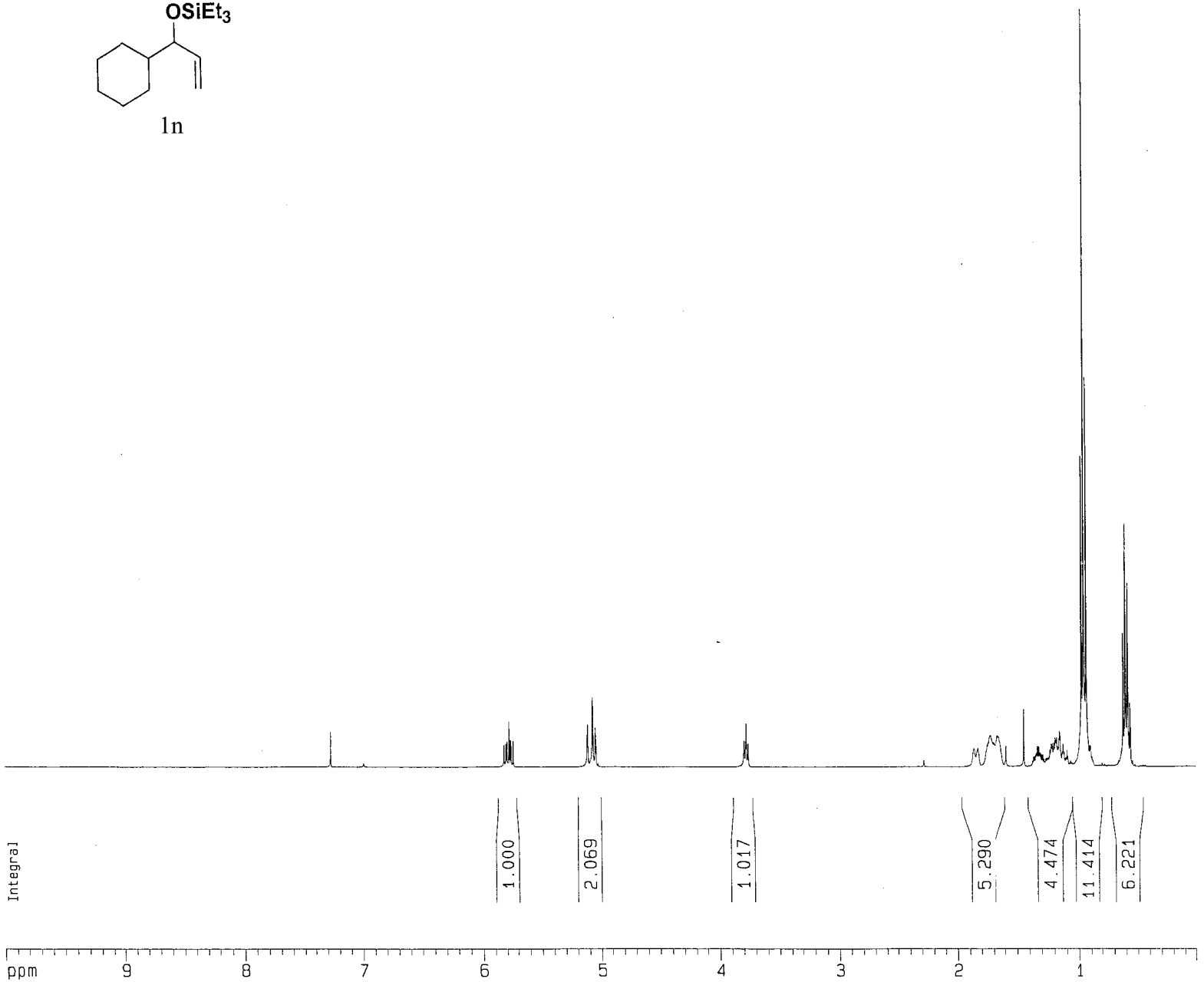
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.5127492 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm

SN061104

1n



Current Data Parameters
NAME SN1104-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060310
Time 12.00
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 292.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.88 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

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

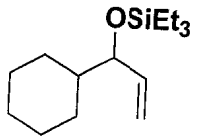
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN061104

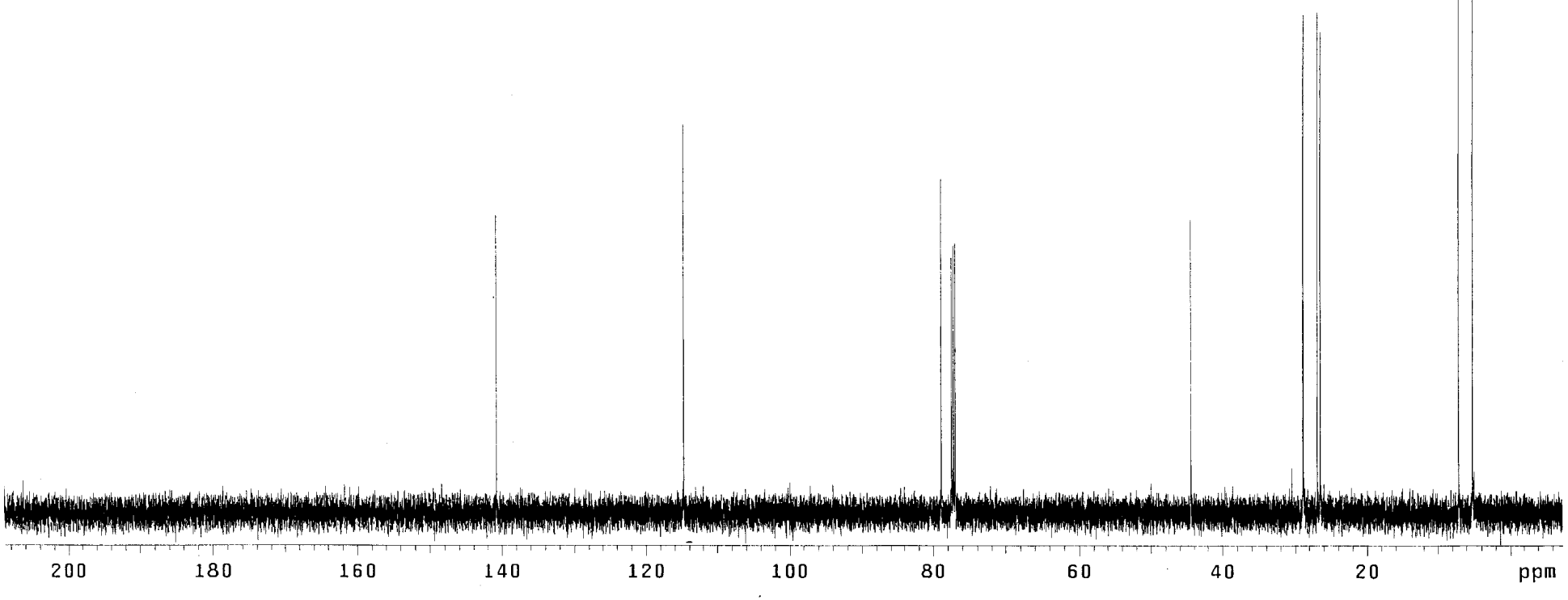
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "rocky"

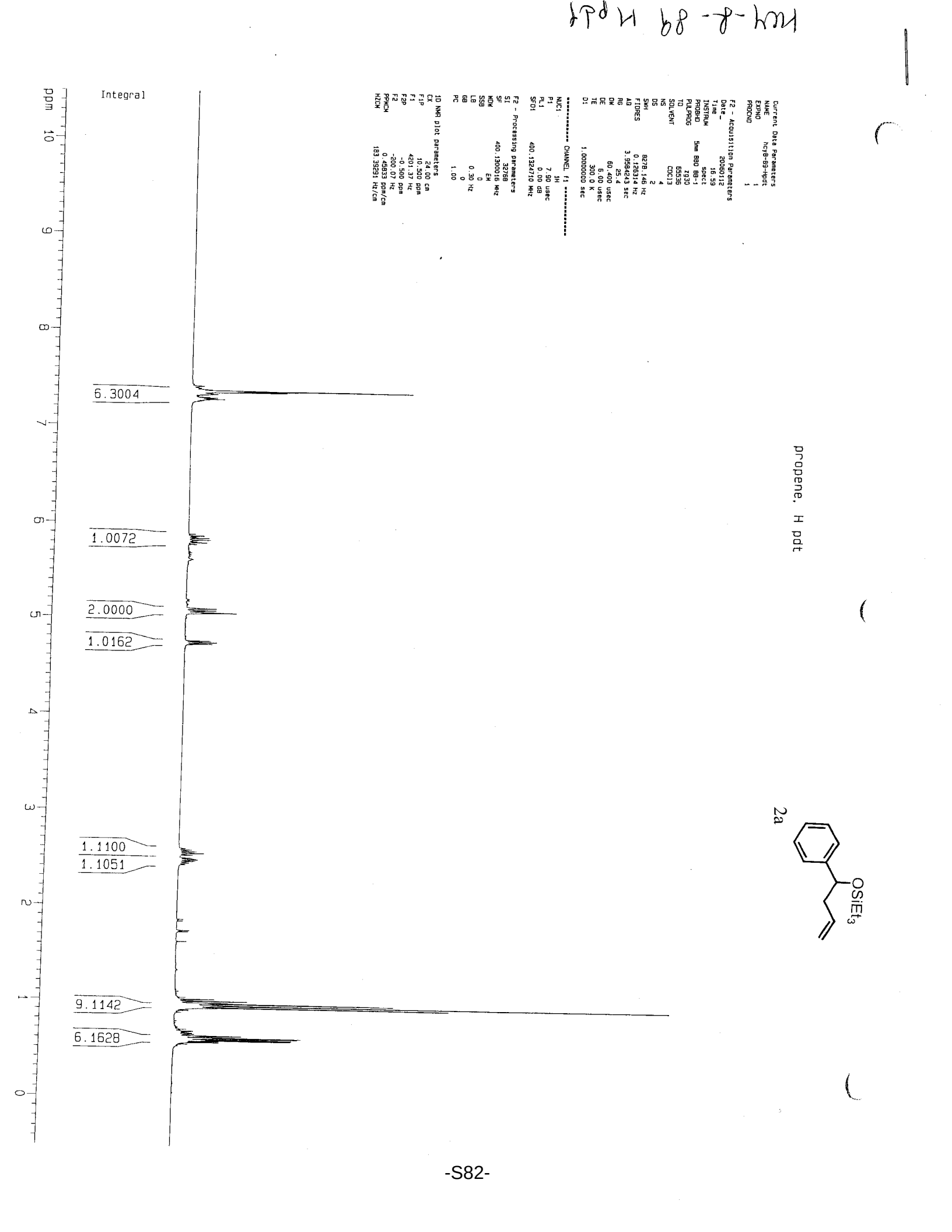
PULSE SEQUENCE
Relax. delay 0.763 sec
Pulse 65.4 degrees
Acq. time 1.736 sec
Width 37735.8 Hz
64 repetitions
OBSERVE C13, 125.7832251 MHz
DECOUPLE H1, 500.2332753 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 2 minutes

Fri Mar 10 18:11:58 EST 2006

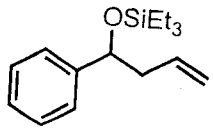


1n




Integral
ppm
6.3004
1.0072
2.0000
1.0162
1.1100
1.1051
9.1142
6.1628
propene, H pdt
2a
OSiEt3

propene, H-pdt



2a

```
Current Data Parameters
NAME        hcy8-89-Hpdt
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20060112
Time              17.03
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  146
DS                    4
SWH           24875.621 Hz
FIDRES        0.379572 Hz
AQ            1.3173236 sec
RG                 1024
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               15.25 usec
PL1               3.00 dB
SFO1        100.6237959 MHz

********** CHANNEL f2 **********
CPDPRG2         waltz16
NUC2               1H
PCPD2           107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127529 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX               24.00 cm
F1P             220.000 ppm
F1             22134.80 Hz
F2P             -10.000 ppm
F2             -1006.13 Hz
PPMCM           9.58333 ppm/cm
HZCM          964.20551 Hz/cm
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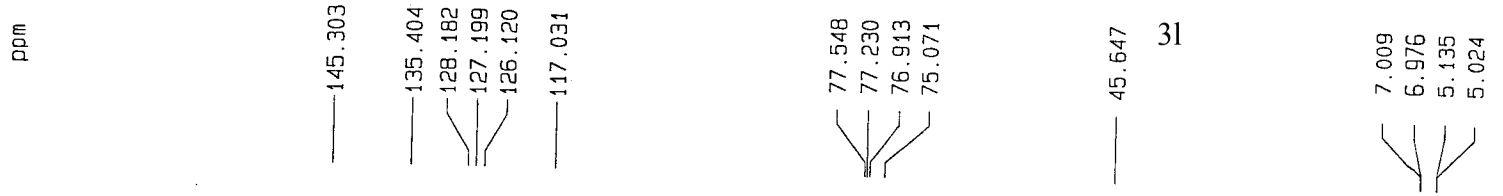
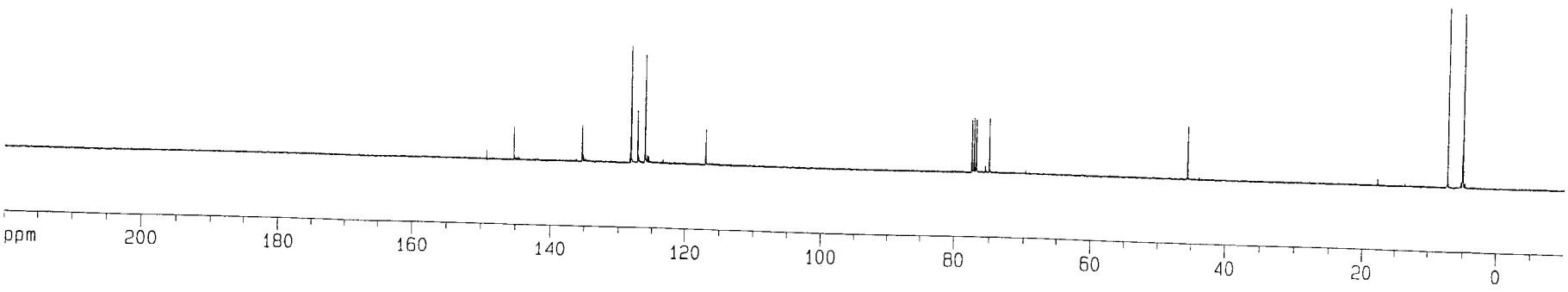
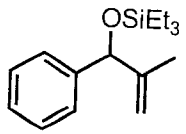




hcy-8-89 Apdt

propene, A-pdt



2a'

```
Current Data Parameters
NAME        hcy8-89-Apdt
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_         20060112
Time            16.54
INSTRUM         spect
PROBHD    5mm BBO BB-1
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  4
DS                  2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG              228.1
DW             60.400 usec
DE              6.00 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       400.1300016 MHz
WDW               EM
SSB               0
LB             0.30 Hz
GB                0
PC             1.00

1D NMR plot parameters
CX            24.00 cm
F1P           10.500 ppm
F1           4201.37 Hz
F2P           -0.500 ppm
F2           -200.07 Hz
PPMCM         0.45833 ppm/cm
HZCM        183.39291 Hz/cm
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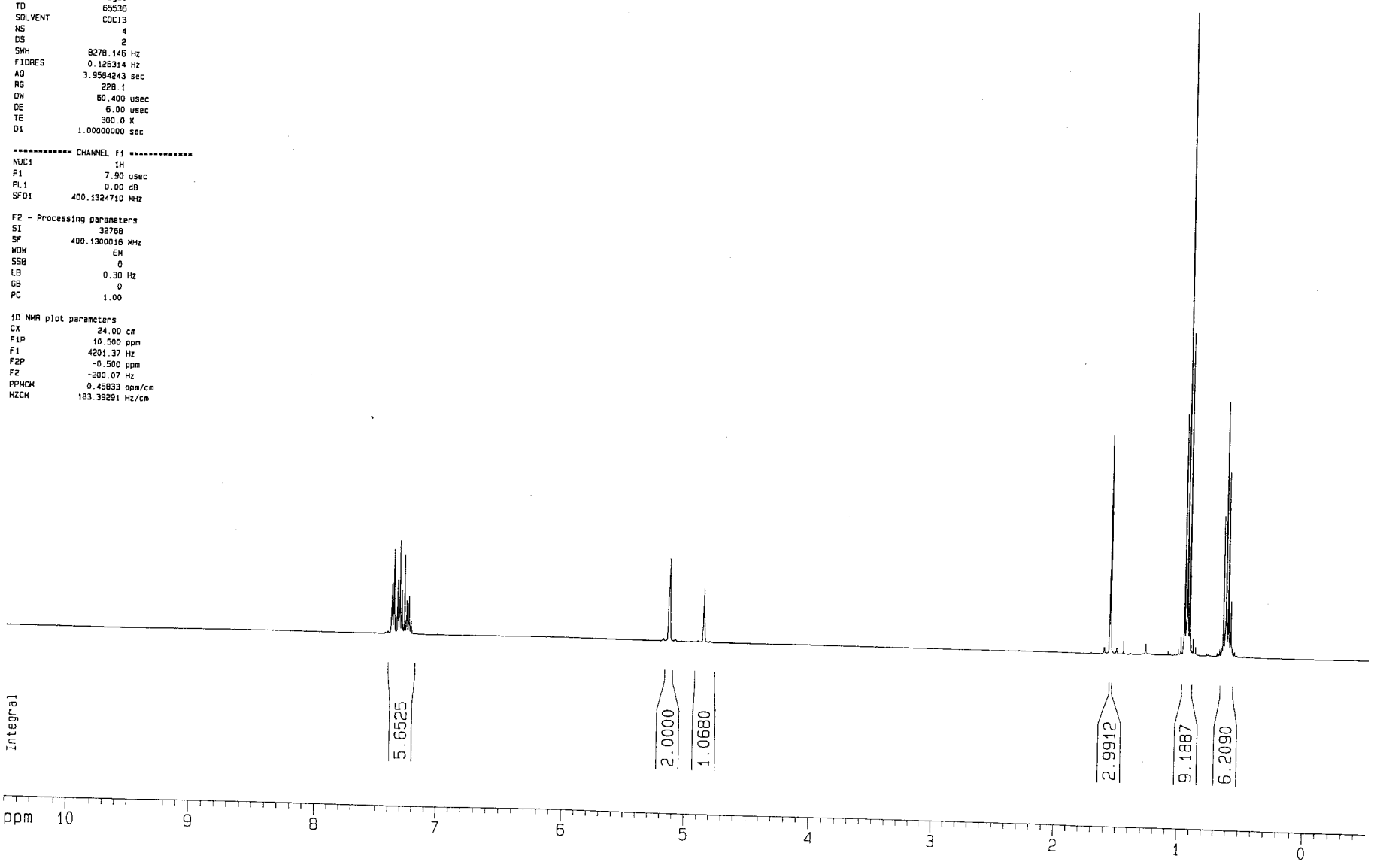

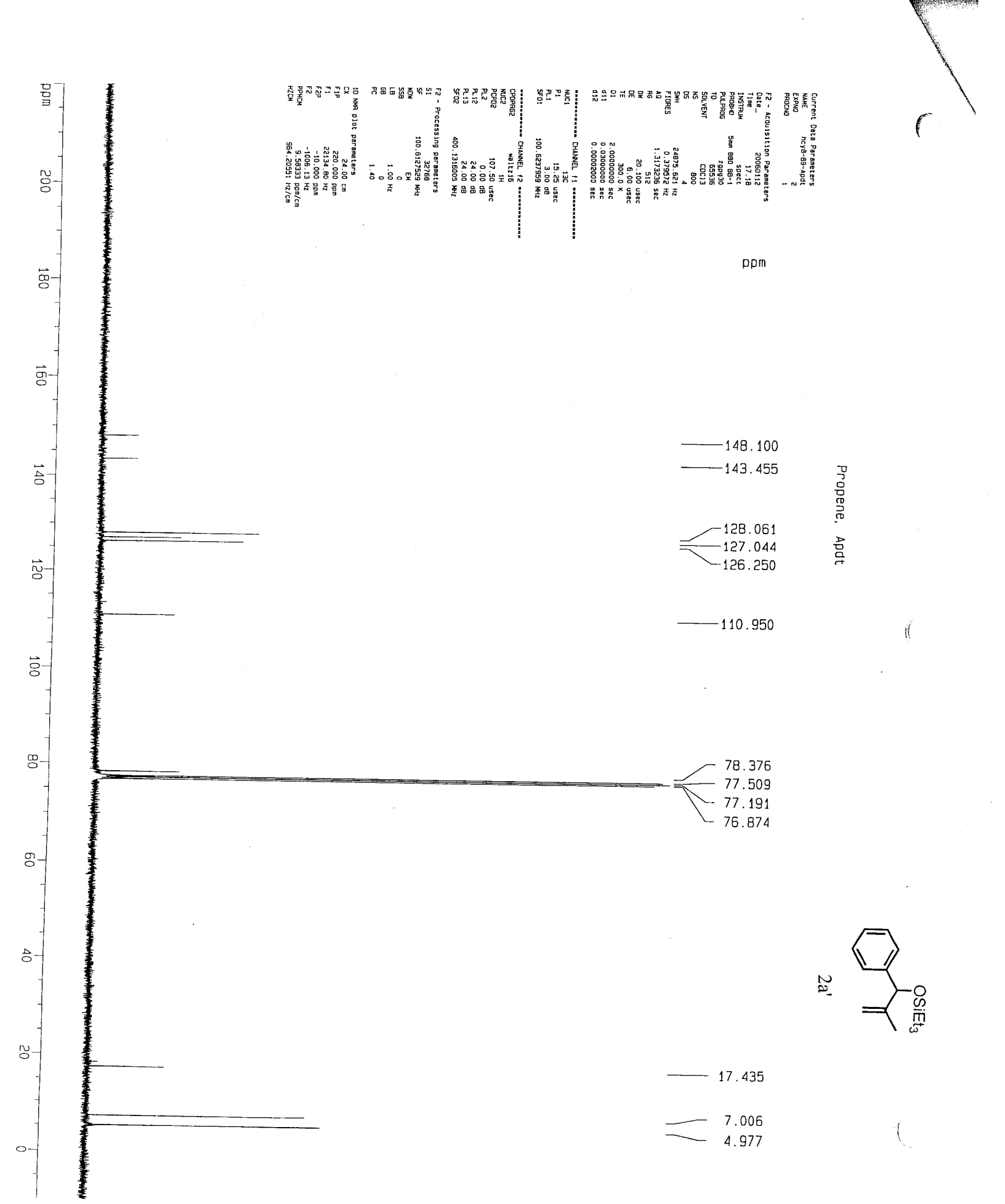
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127.044
126.250
110.950
78.376
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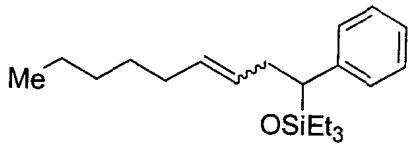
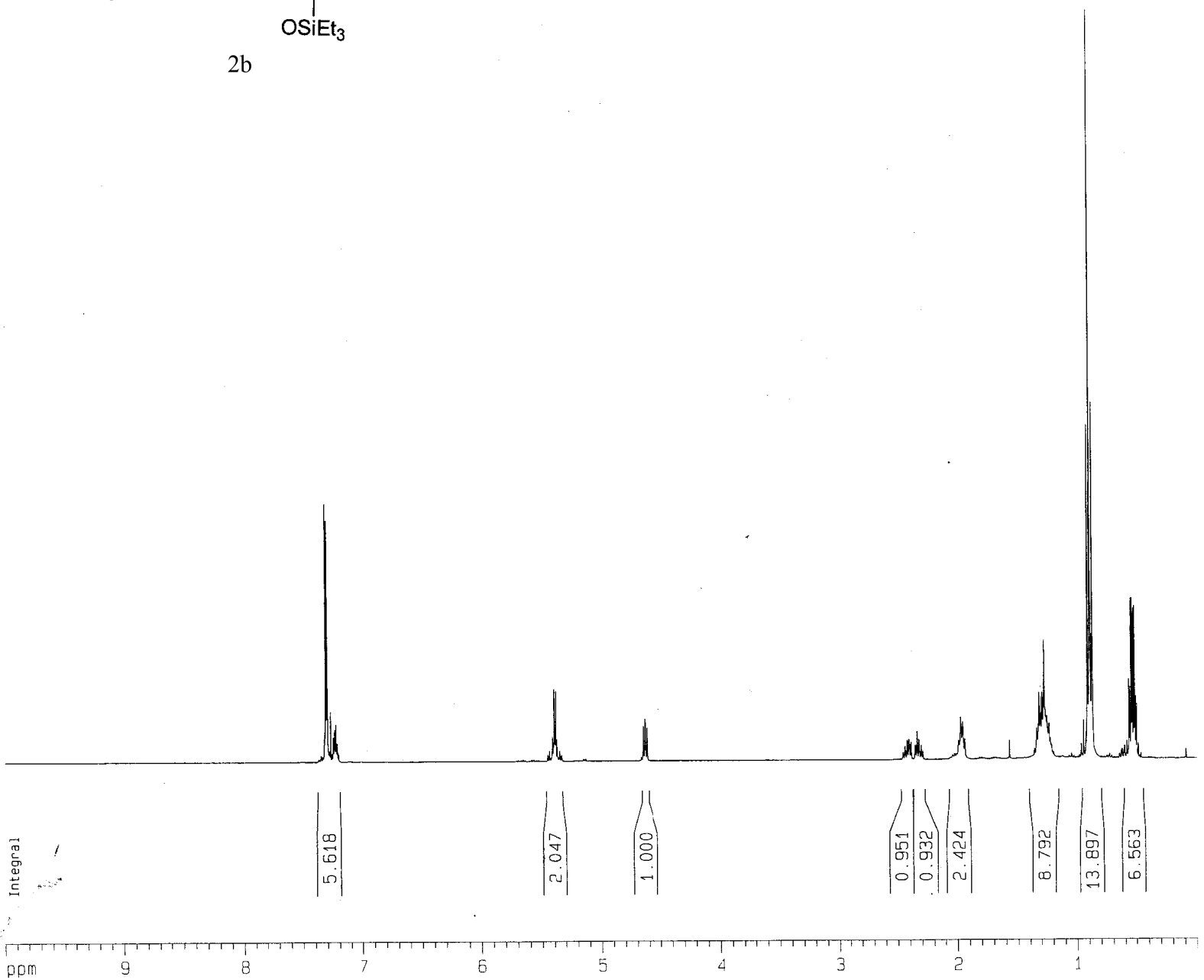
77.191
76.874
17.435
7.006
4.977
Propene, Apdt
2a'
OSiEt3
ppm

SN050658 ene product



2b



Current Data Parameters

NAME	SN568ene-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050614
Time	19.59
INSTRUM	spect
PROBHD	5 mm GNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	4
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	9.50 usec
PL1	2.00 dB
SFO1	400.1324710 MHz

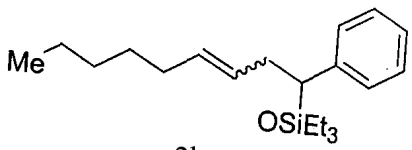
F2 - Processing parameters

SI	32768
SF	400.1300059 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

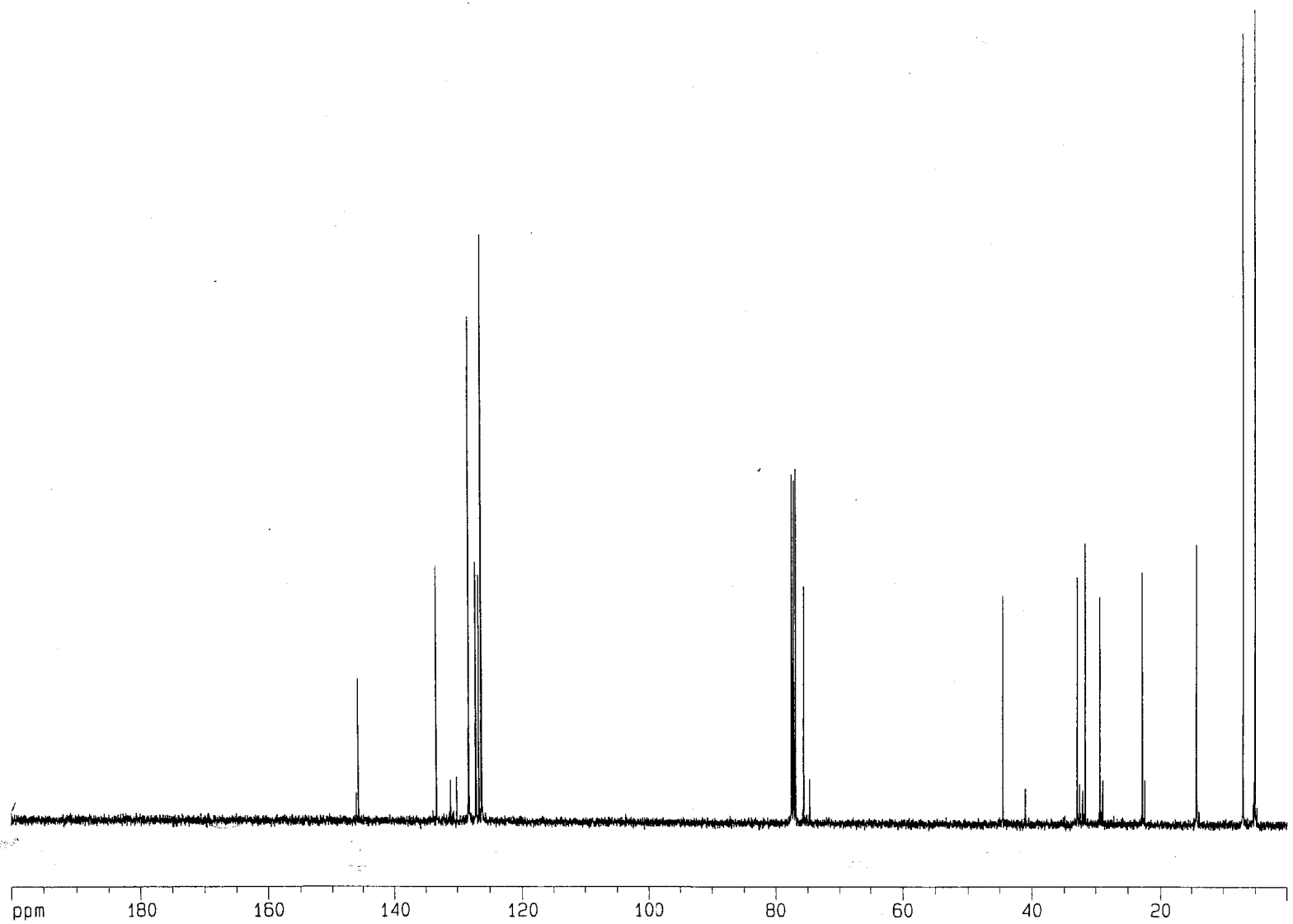
1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm

octene benzaldehyde TESOTf ene



2b



Current Data Parameters
NAME octene-ene-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050716
Time 22.11
INSTRUM spect
PROBHD 5mm BBC BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 213
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 us
TE 300.0 K
D1 2.00000000 se
d11 0.03000000 se
d12 0.00002000 se

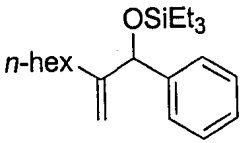
========== CHANNEL f1 ==========
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

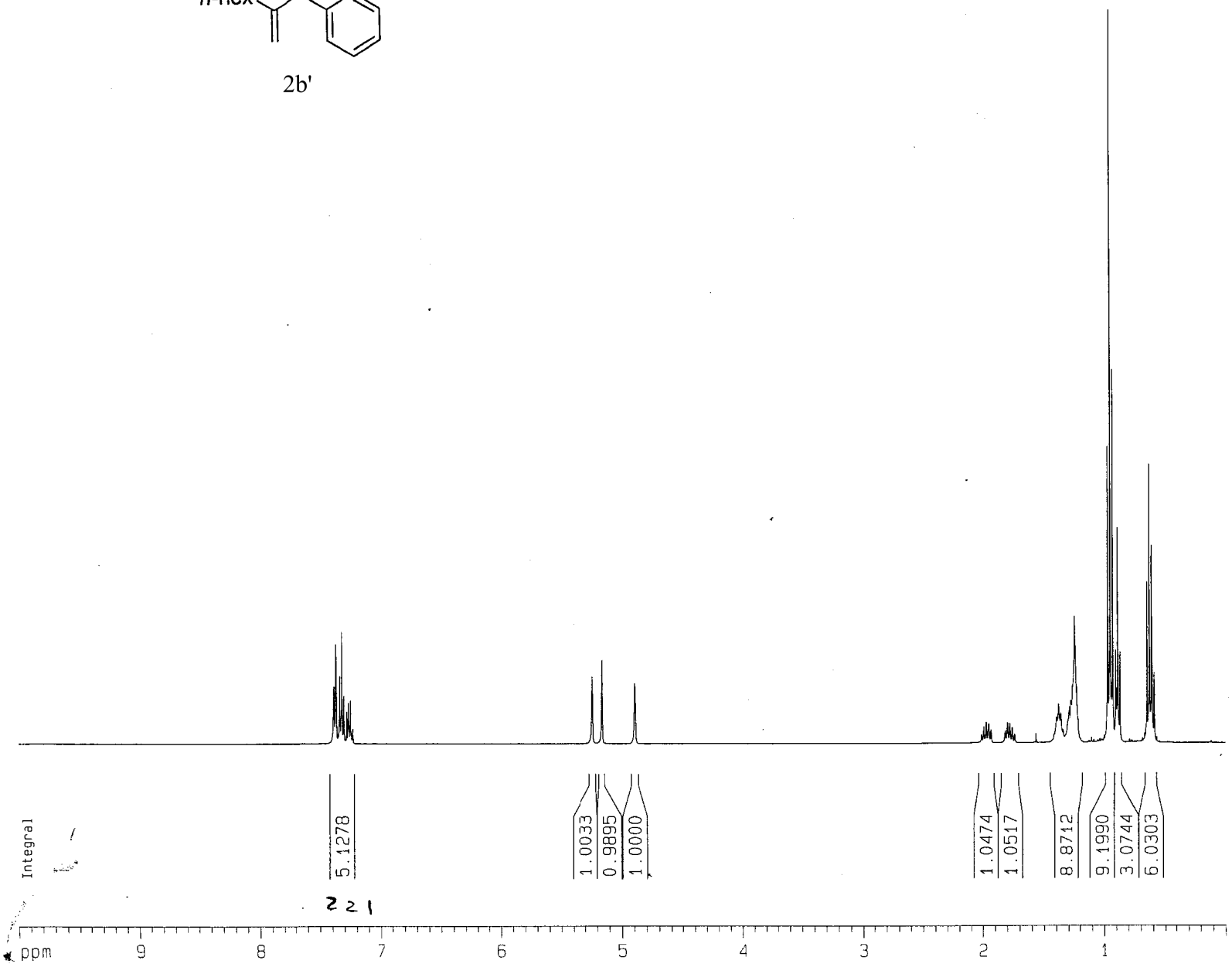
F2 - Processing parameters
SI 32768
SF 100.6127476 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm

SN050658 allylic alcohol



2b'



Current Data Parameters

NAME	SN658heck-c
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050614
Time	19.05
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	4
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	64
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

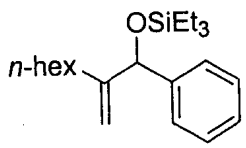
NUC1	1H
P1	9.50 usec
PL1	2.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

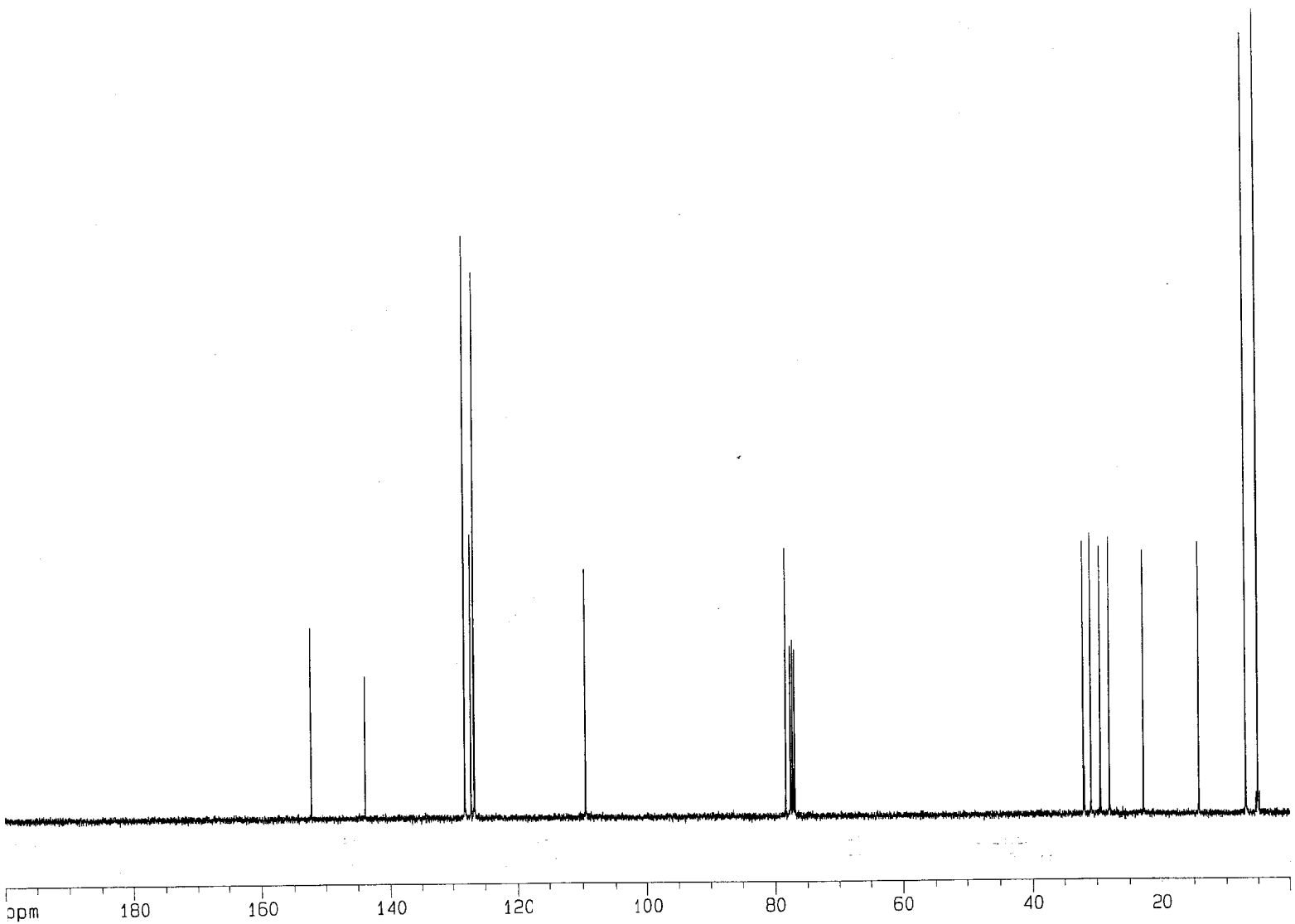
1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm



2b'

octene benzaldehyde TESOTf



Current Data Parameters

NAME	octene-al-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050716
Time	21.50
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	95
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	8192
DW	19.900 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	15.25 usec
PL1	3.00 dB
SFO1	100.6237959 MHz

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

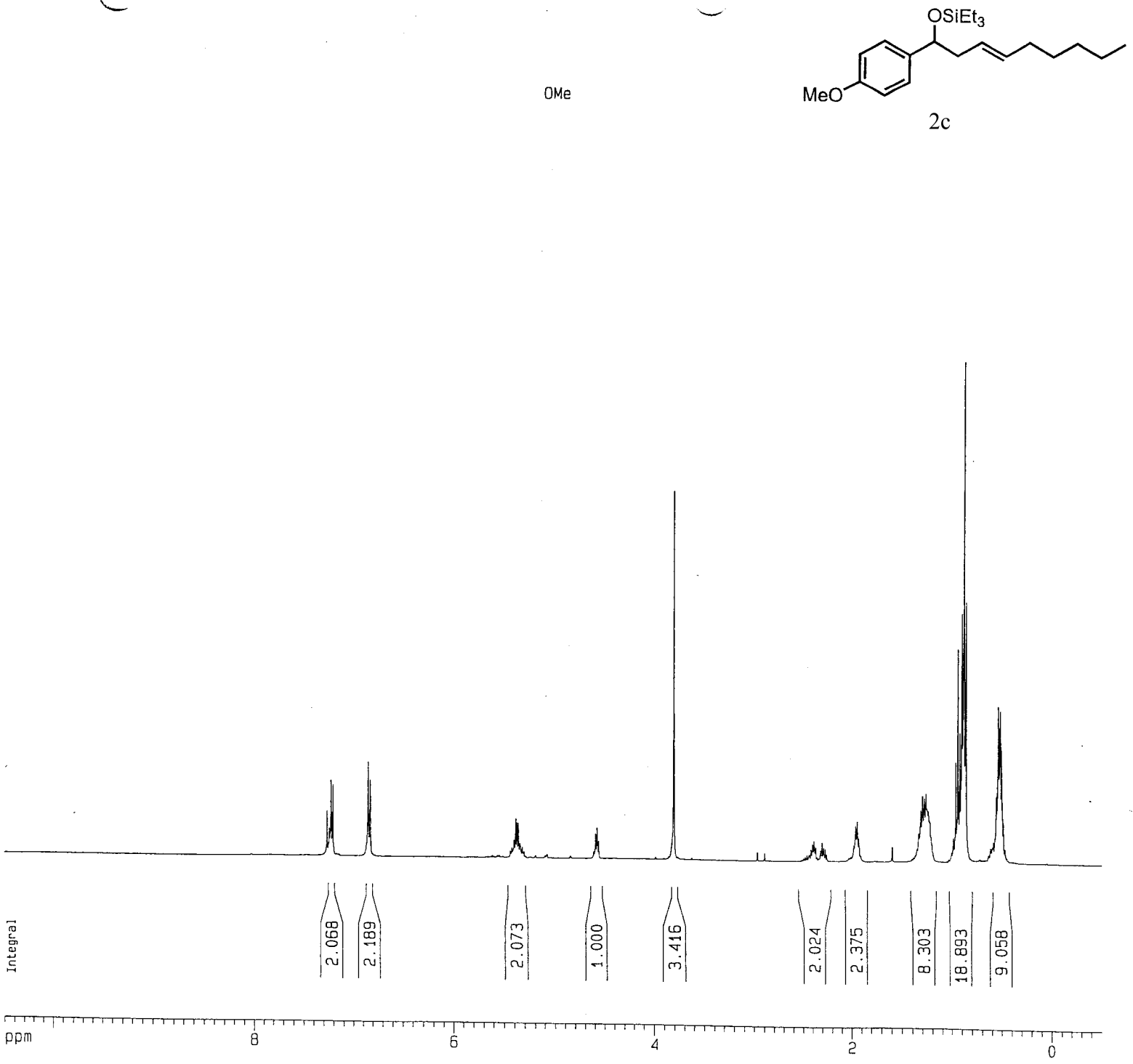
CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127484 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12744 Hz/cm



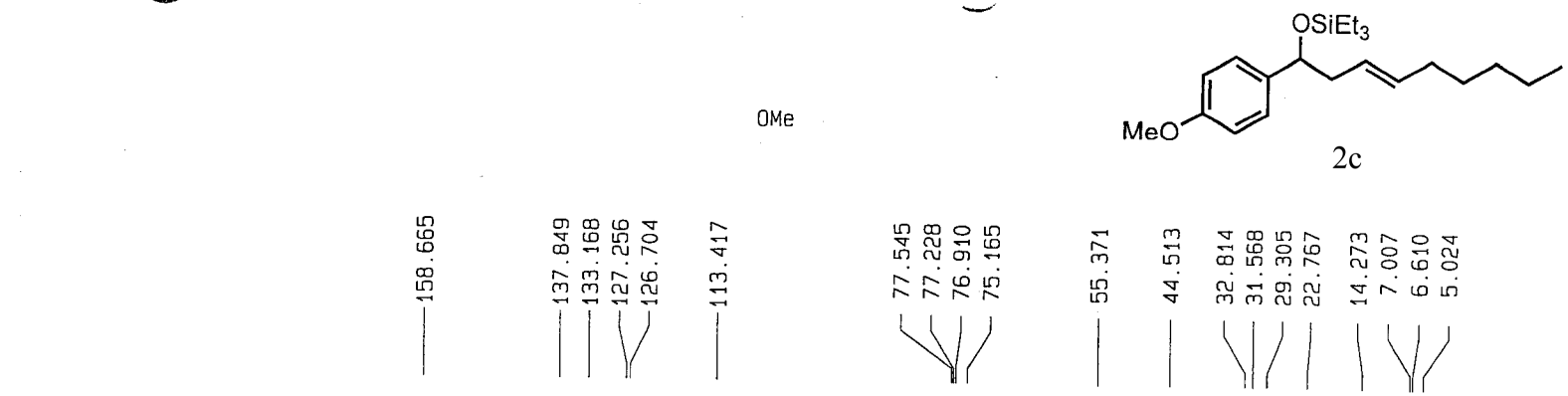
Current Data Parameters
NAME hcy-8-44p-OMe
EXPNO 1
PROCNO 1

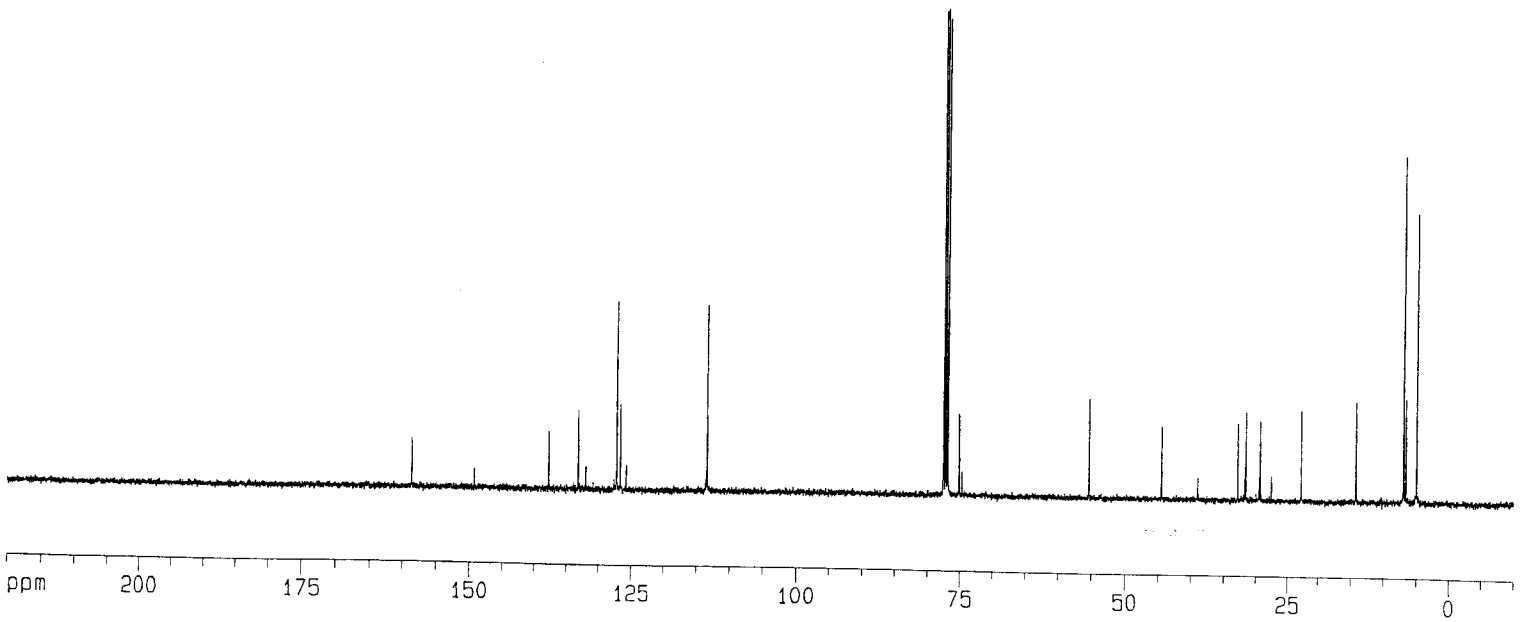
F2 - Acquisition Parameters
Date_ 20051028
Time 17.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 1
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.88 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 9.19 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm






```
Current Data Parameters
NAME                  hcy-8-44-OMe
EXPNO                            2
PROCNO                           1

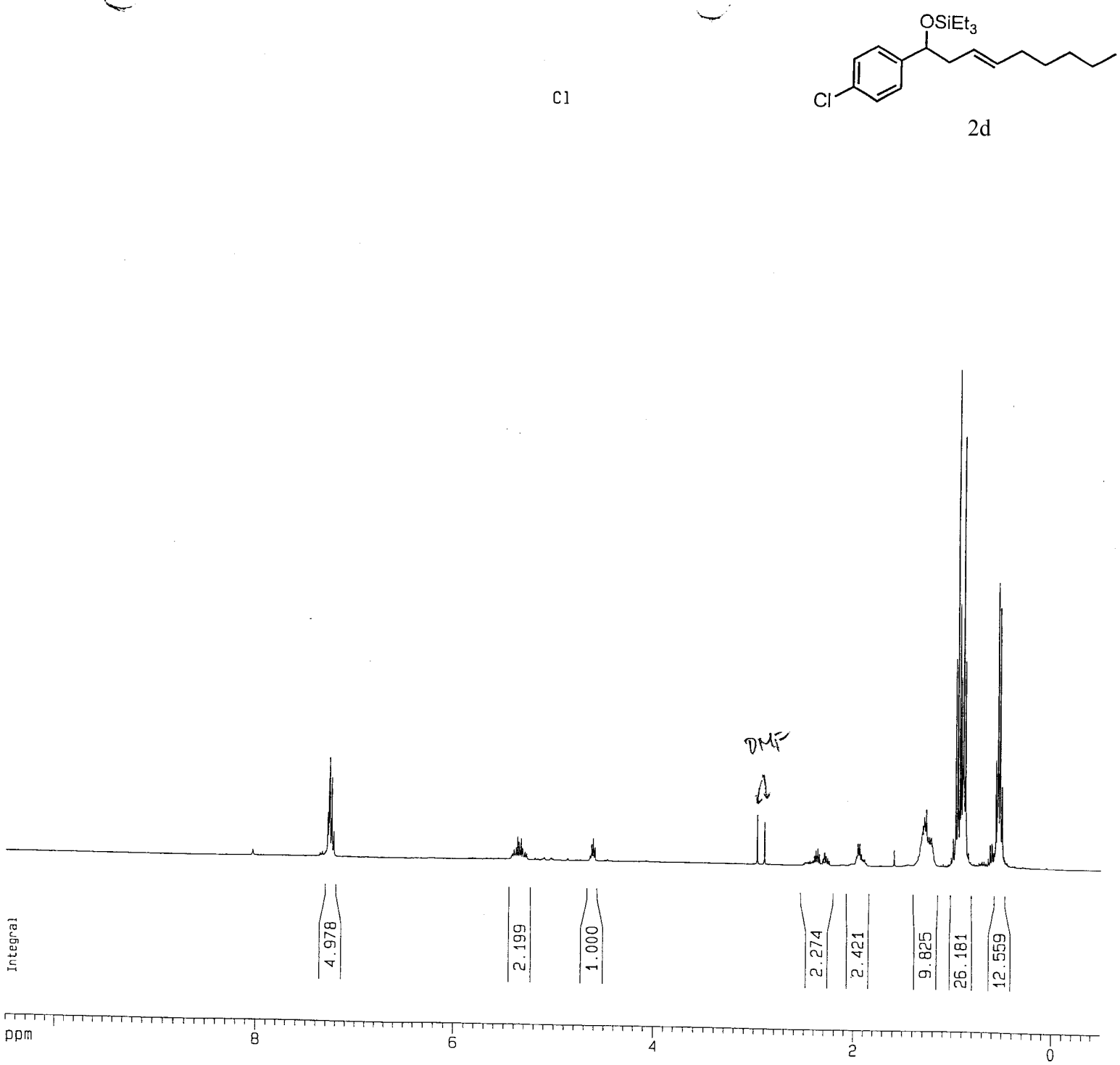
F2 - Acquisition Parameters
Date_                     20051028
Time                         16.05
INSTRUM                      spect
PROBHD             5mm BBO BB-1
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                           1024
DS                              4
SWH                  24875.621 Hz
FIDRES                0.379572 Hz
AQ                   1.3173236 sec
RG                           1024
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                     15.25 usec
PL1                      3.00 dB
SFO1              100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                  107.50 usec
PL2                      0.00 dB
PL12                    24.00 dB
PL13                    24.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                100.6127499 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                           1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                   220.000 ppm
F1                  22134.80 Hz
F2P                   -10.000 ppm
F2                  -1006.13 Hz
PPMCM                11.50000 ppm/cm
HZCM                1157.04663 Hz/cm
```



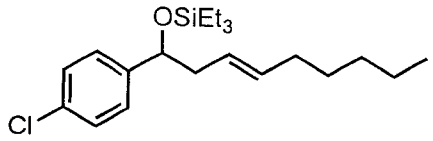
Current Data Parameters
NAME hcy-8-44p-Cl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051028
Time 17.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 1
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.88 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

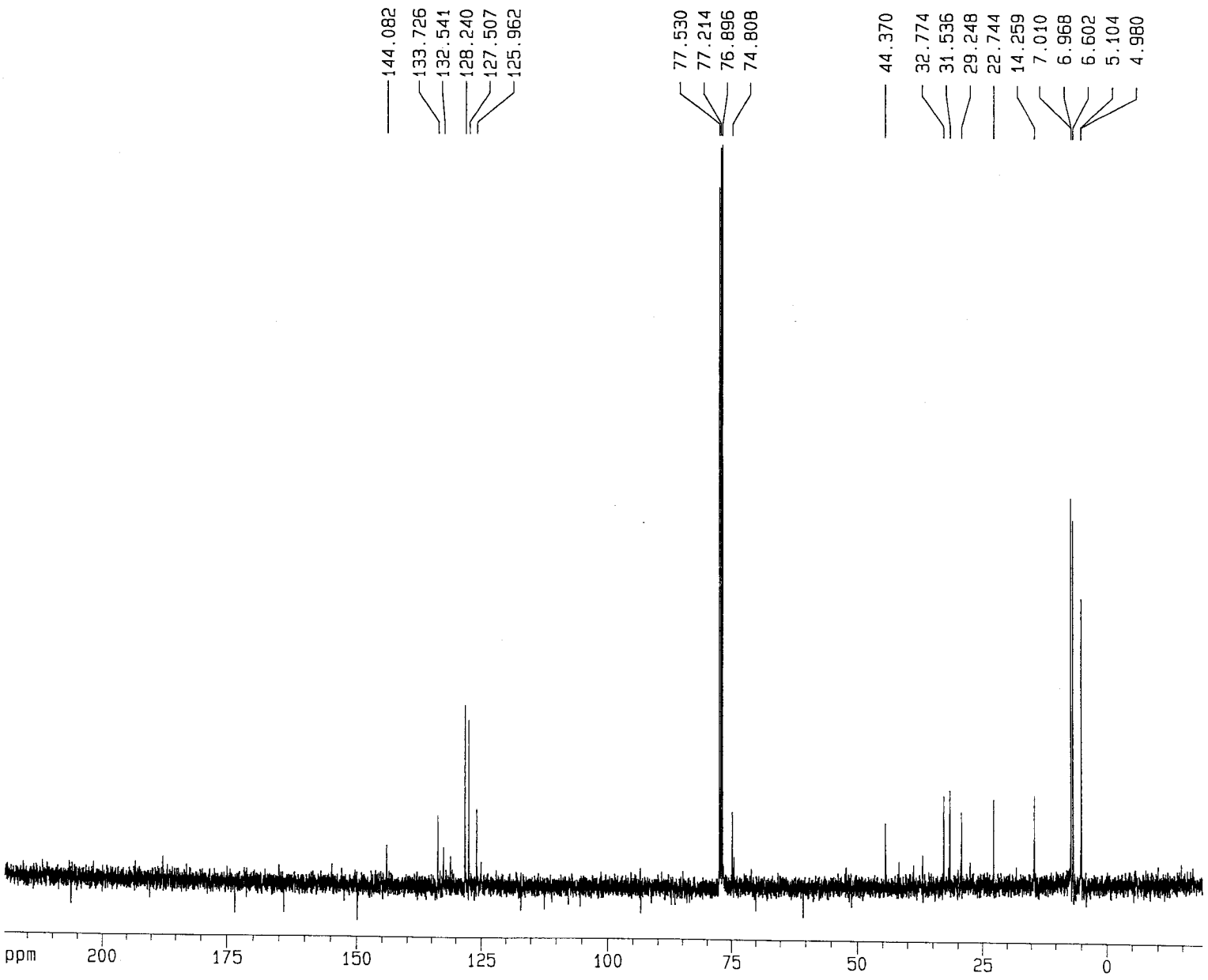
F2 - Processing parameters
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.19 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm



Cl

2d




```
Current Data Parameters
NAME          hcy-8-44-Cl
EXPNO                   2
PROCNO                  1

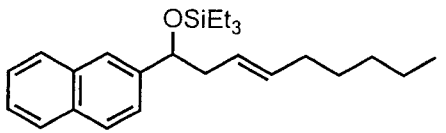
F2 - Acquisition Parameters
Date_           20051028
Time               12.15
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  800
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG               1824.6
DW               20.850 usec
DE                6.00 usec
TE               294.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             88.01 usec
PL2                3.00 dB
PL12              22.00 dB
PL13              22.00 dB
SF02         400.1316005 MHz

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

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P              219.353 ppm
F1               22069.67 Hz
F2P              -18.995 ppm
F2               -1911.15 Hz
PPMCM            11.91738 ppm/cm
HZCM          1199.04077 Hz/cm
```



2e

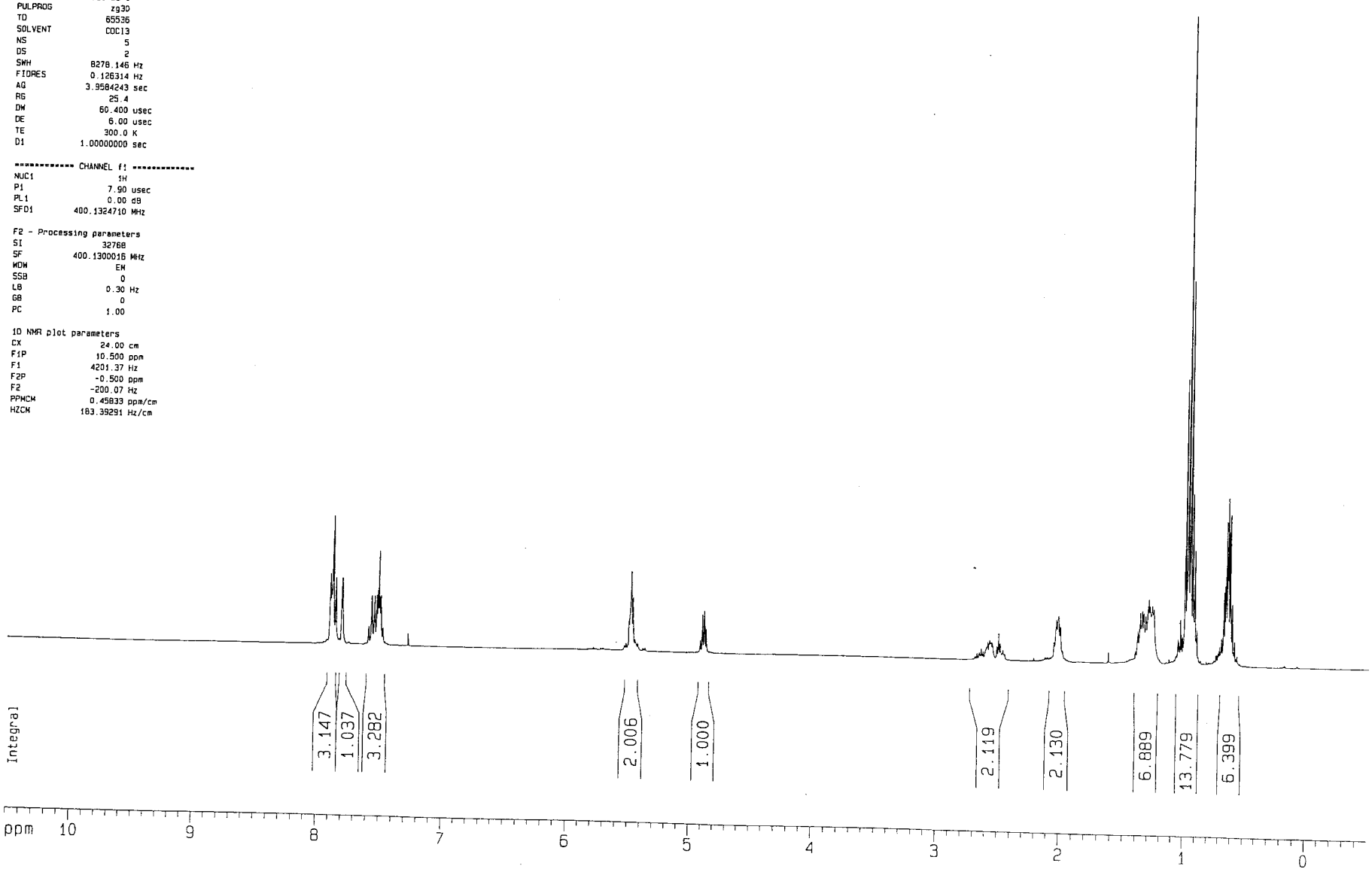
```
Current Data Parameters
NAME          hcy8-87ht-p
EXPNO                  11
PROCNO                  1

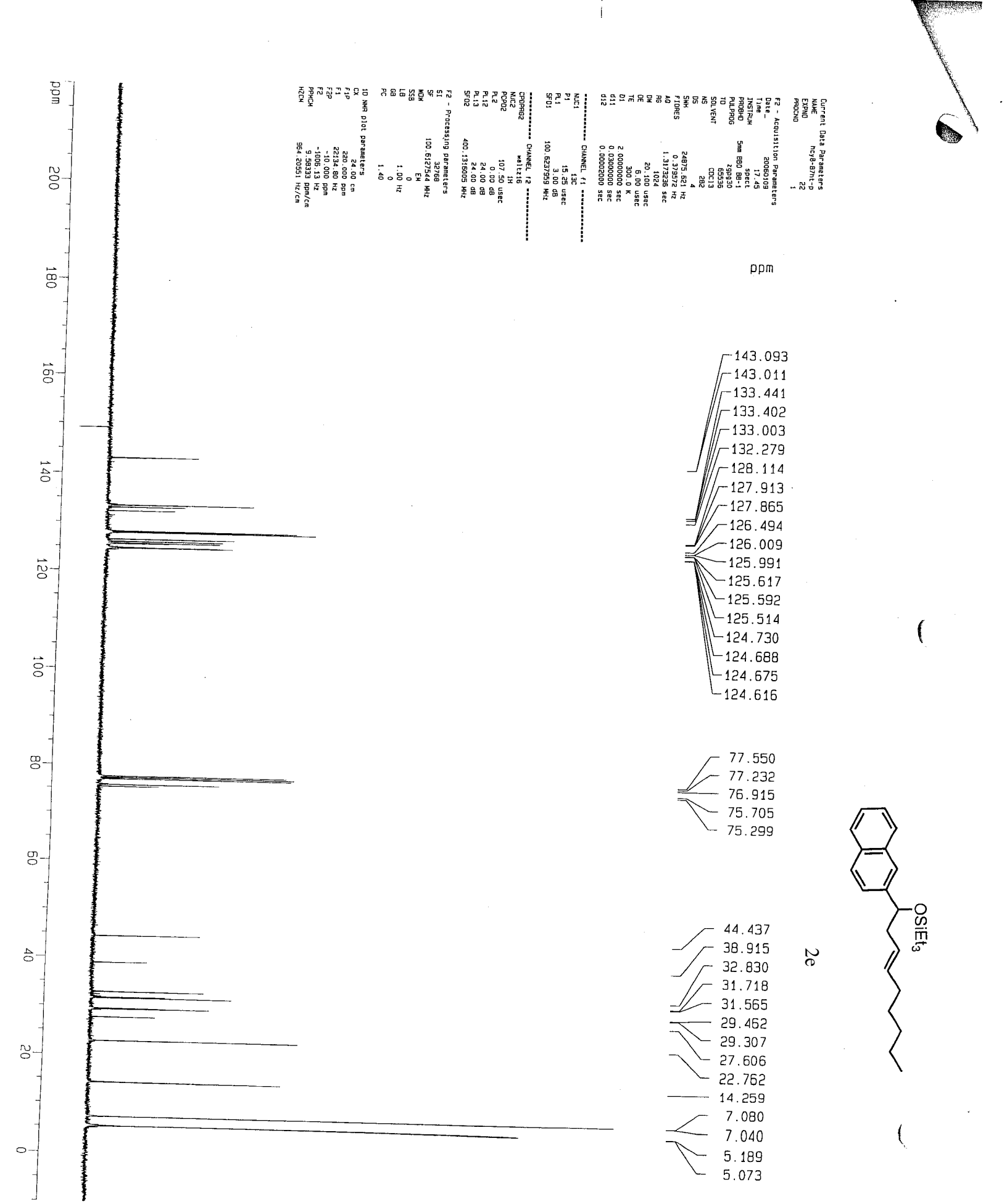
F2 - Acquisition Parameters
Date_            20060109
Time                17.29
INSTRUM             spect
PROBHD     5mm BBO BB-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                  25.4
DW               60.400 usec
DE                6.00 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

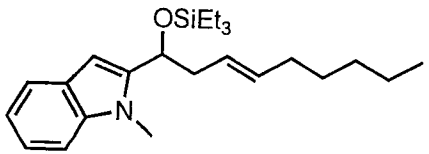
F2 - Processing parameters
SI                 32768
SF          400.1300016 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                24.00 cm
F1P               10.500 ppm
F1               4201.37 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM             0.45833 ppm/cm
HZCM            183.39291 Hz/cm
```

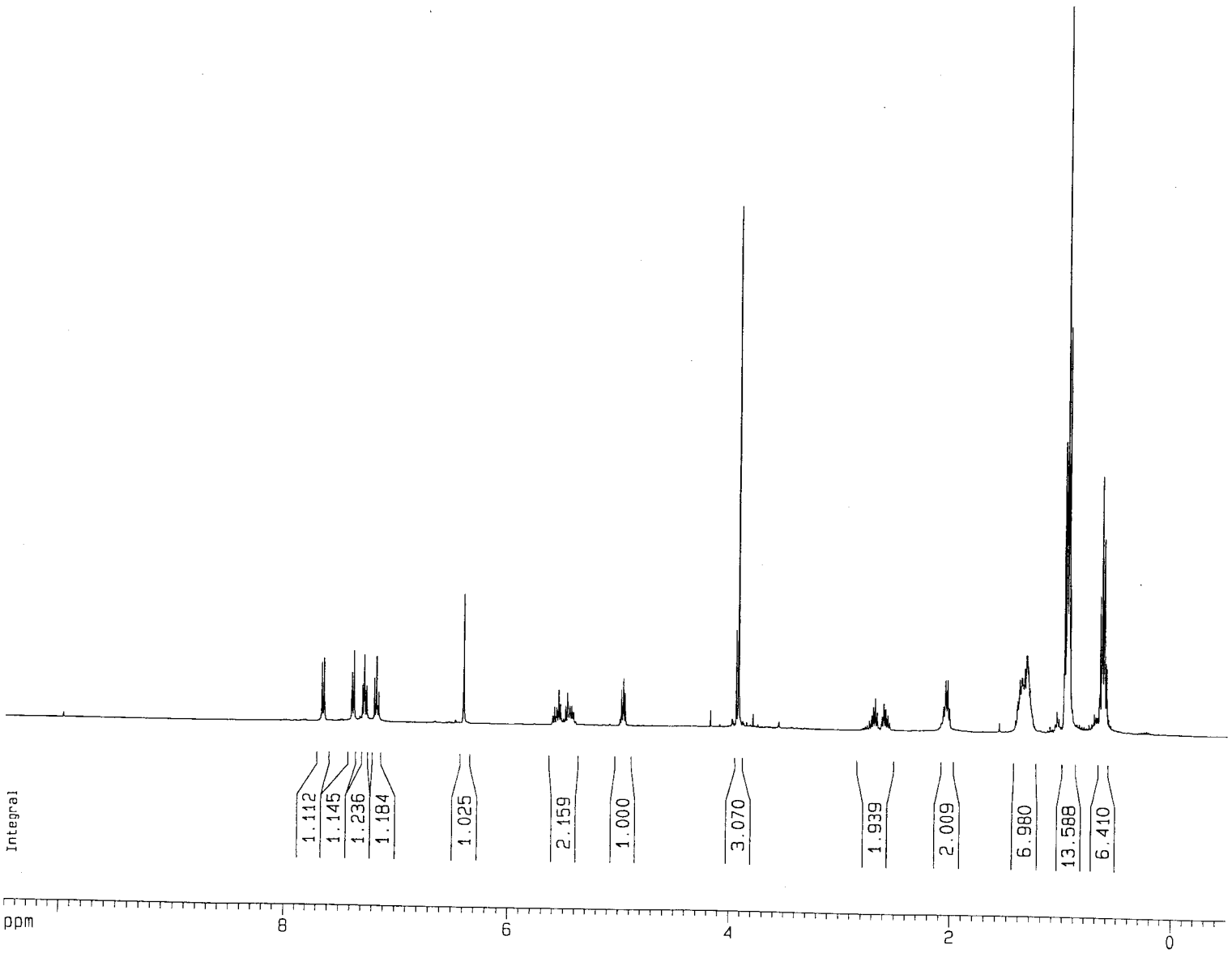



Current Data Parameters
NAME mcj8-8ht-p
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060109
Time 17.45
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 282
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3172336 sec
RG 1024
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
143.093
143.011
133.441
133.402
133.003
132.279
128.114
127.913
127.865
126.494
126.009
125.991
125.617
125.592
125.514
124.730
124.688
124.675
124.616
77.550
77.232
76.915
75.705
75.299
44.437
38.915
32.830
31.718
31.565
29.462
29.307
27.606
22.762
14.259
7.080
7.040
5.189
5.073
OSiEt3
2e
ppm

Me-indole-2-CHO, f1



2f



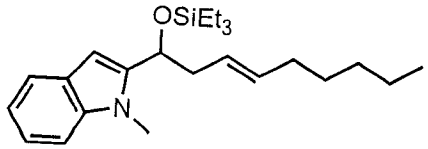
Current Data Parameters
NAME hcy-8-52-ind-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051110
Time 12.10
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 18
DW 60.400 usec
DE 6.00 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 400.1300016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

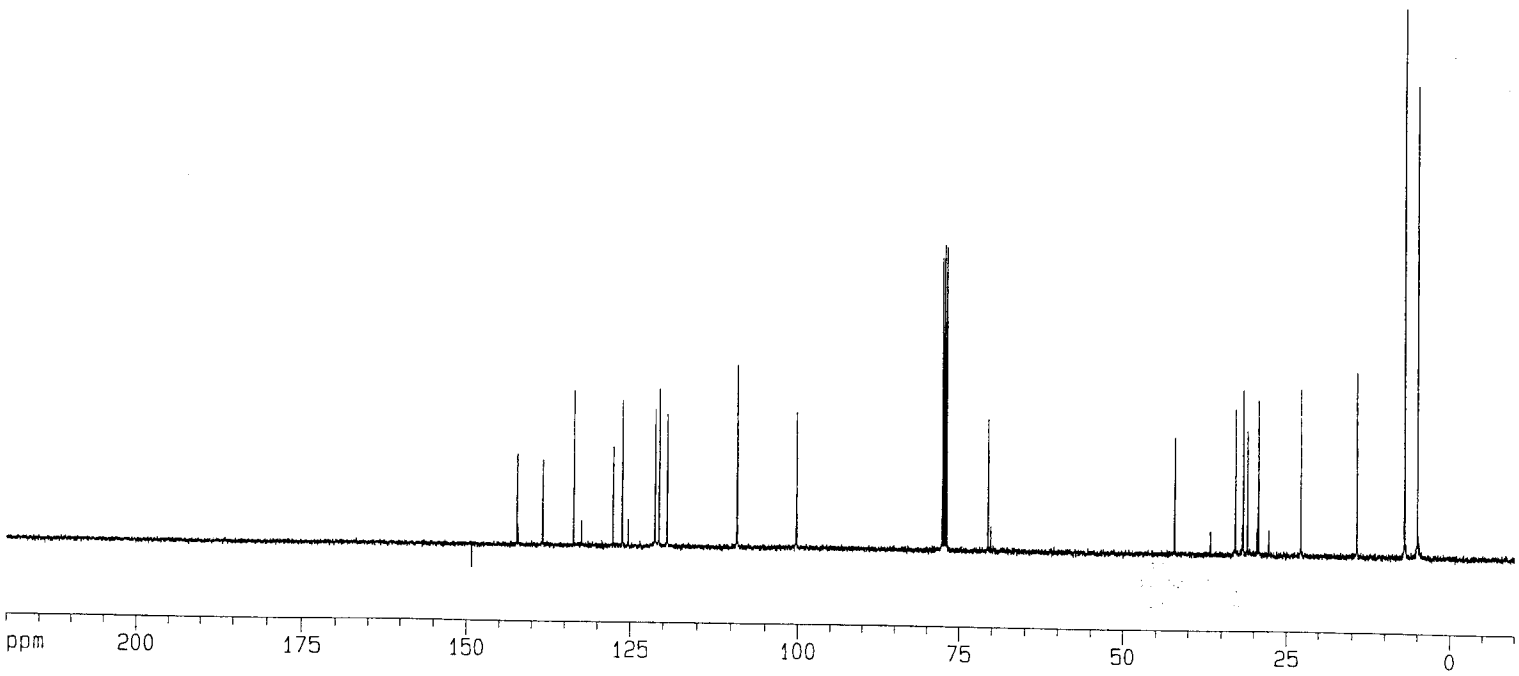
1D NMR plot parameters
CX 20.00 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm



2f

ppm

142.280
138.383
133.691
127.670
126.236
121.293
120.677
119.432
109.071
100.181
77.587
77.269
76.952
70.584
42.188
32.818
31.611
31.038
29.295
22.784
14.296
6.999
5.029



Current Data Parameters
NAME hcy-8-52-ind-2
EXPNO 2
PROCNO 1

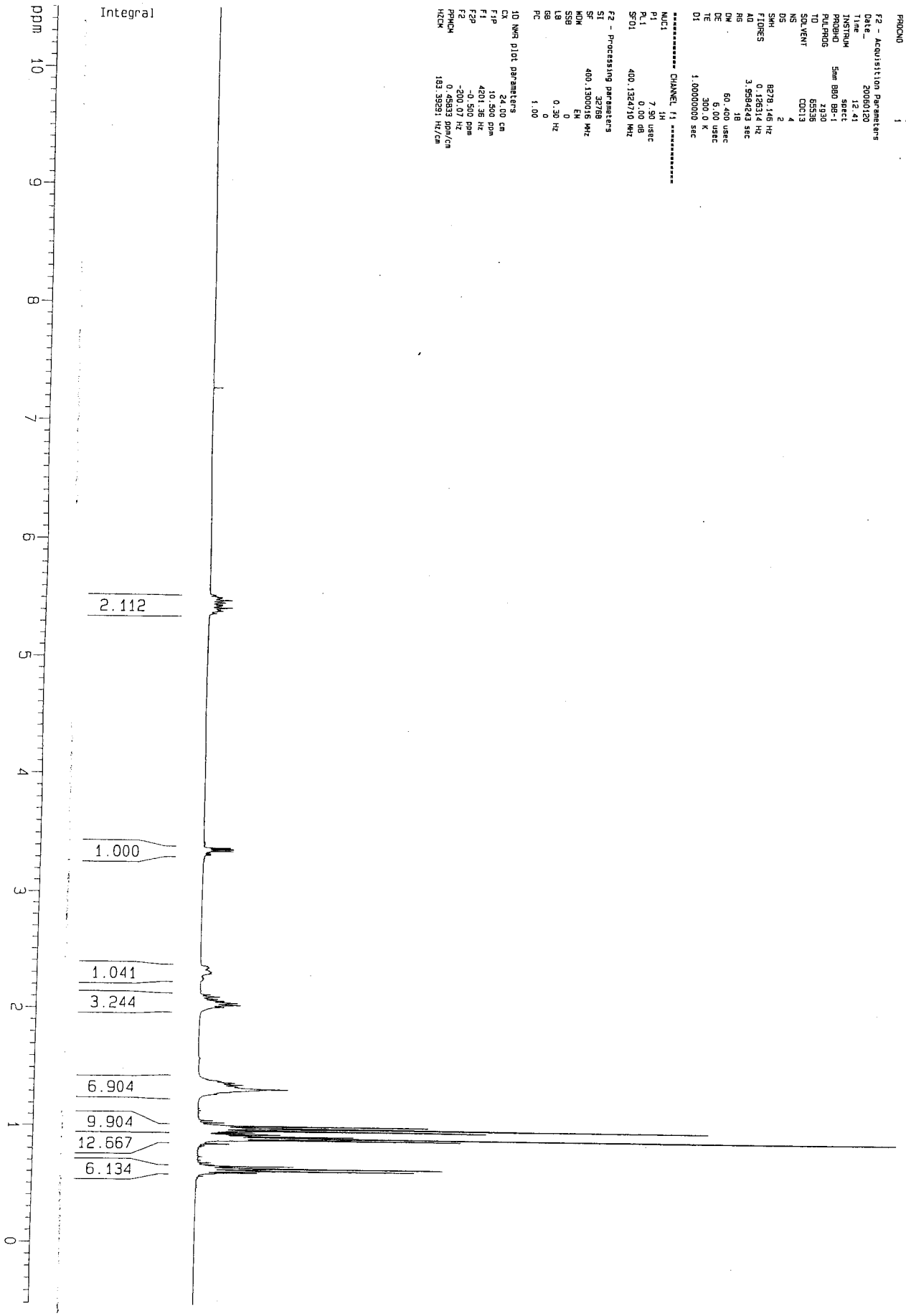
F2 - Acquisition Parameters
Date_ 20051110
Time 12.17
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 434
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04663 Hz/cm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ppm
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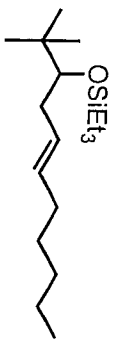
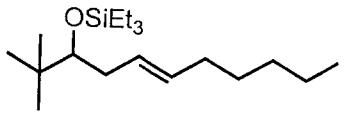
3.244
6.904
9.904
12.667
6.134
Current Data Parameters
NAME mcy8-95hpdt
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060120
Time 12.41
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.958243 sec
RG 18
DW 60.400 usec
DE 6.00 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 400.1300016 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
F1 4201.36 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.45833 ppm/cm
HZCM 183.39291 Hz/cm

H-pdt


OSiEt3

2g



2g

H-pdt

```
Current Data Parameters
NAME          hcy8-95Hpdt
EXPNO                   2
PROCNO                  1

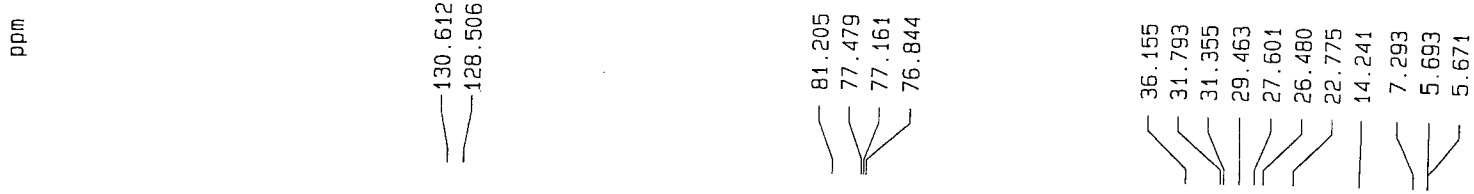
F2 - Acquisition Parameters
Date_          20060120
Time              12.55
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  238
DS                    4
SWH           24875.621 Hz
FIDRES         0.379572 Hz
AQ             1.3173236 sec
RG                1149.4
DW               20.100 usec
DE                6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

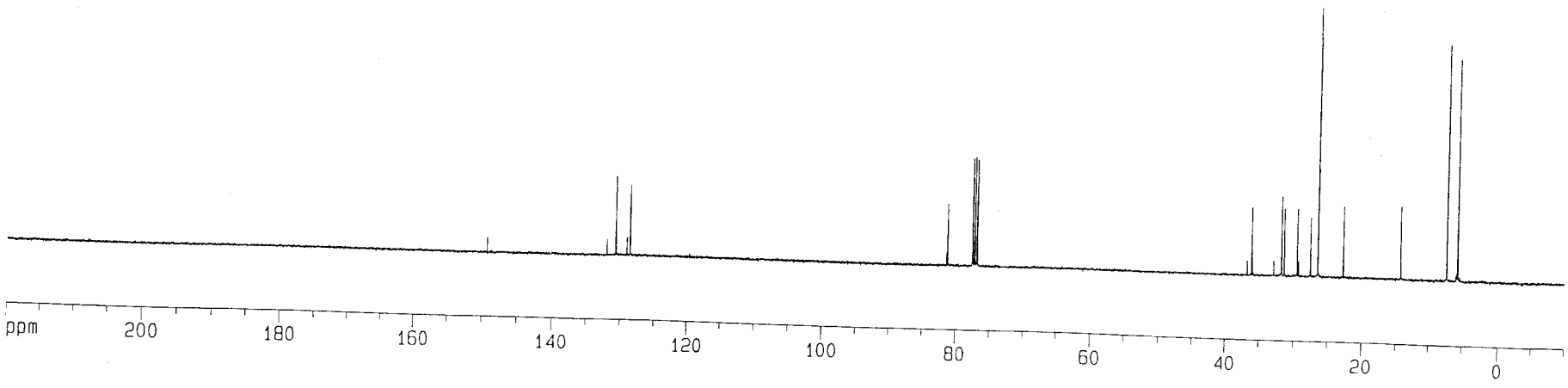
====== CHANNEL f1 ======
NUC1                13C
P1                15.25 usec
PL1               3.00 dB
SFO1         100.6237959 MHz

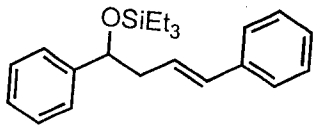
====== CHANNEL f2 ======
CPDPRG2         waltz16
NUC2                1H
PCPD2           107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127529 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                24.00 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM             9.58333 ppm/cm
HZCM           964.20551 Hz/cm
```







H-pdt

2h

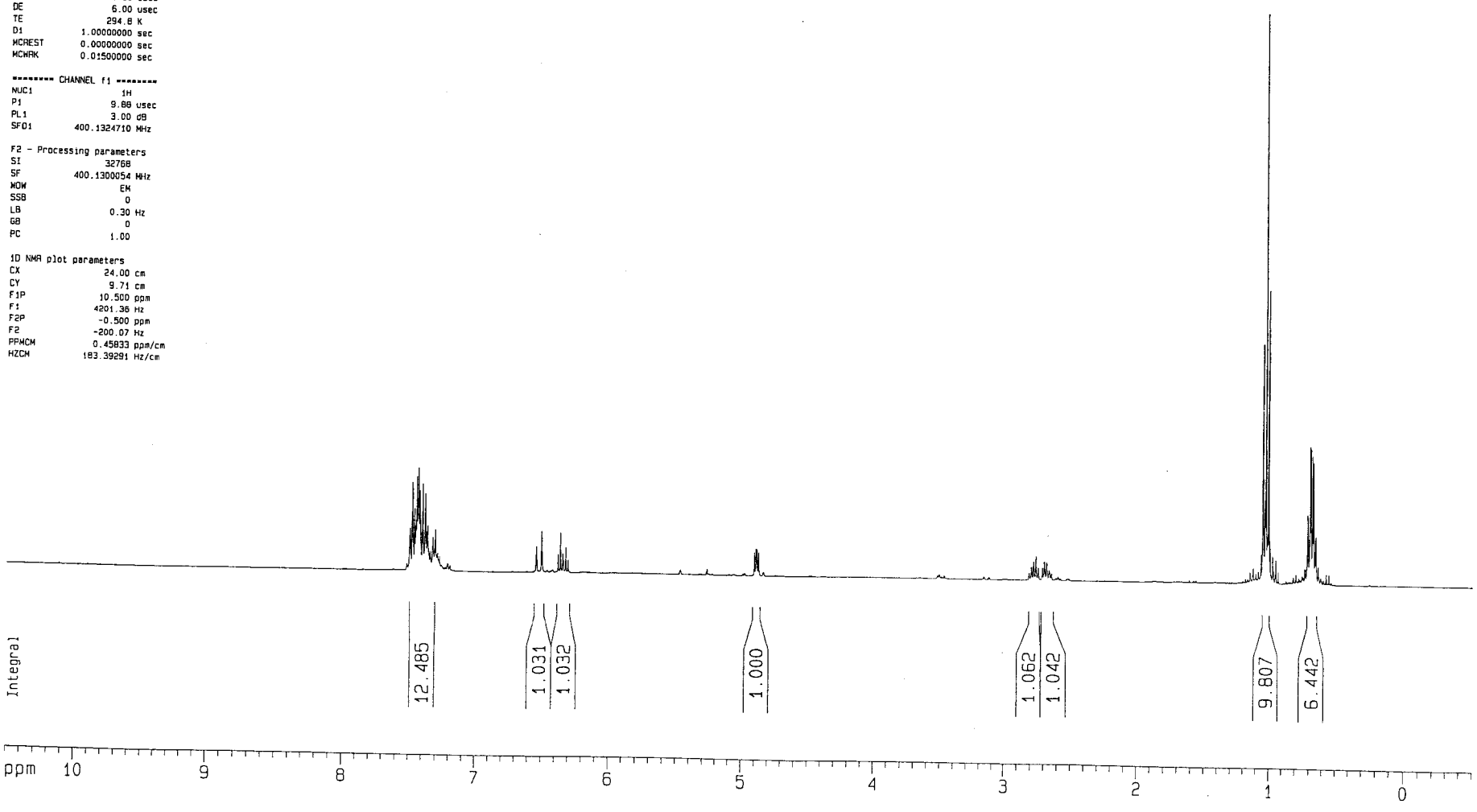
```
Current Data Parameters
NAME      allylcontrolf3
EXPNO                  1
PROCNO                 1

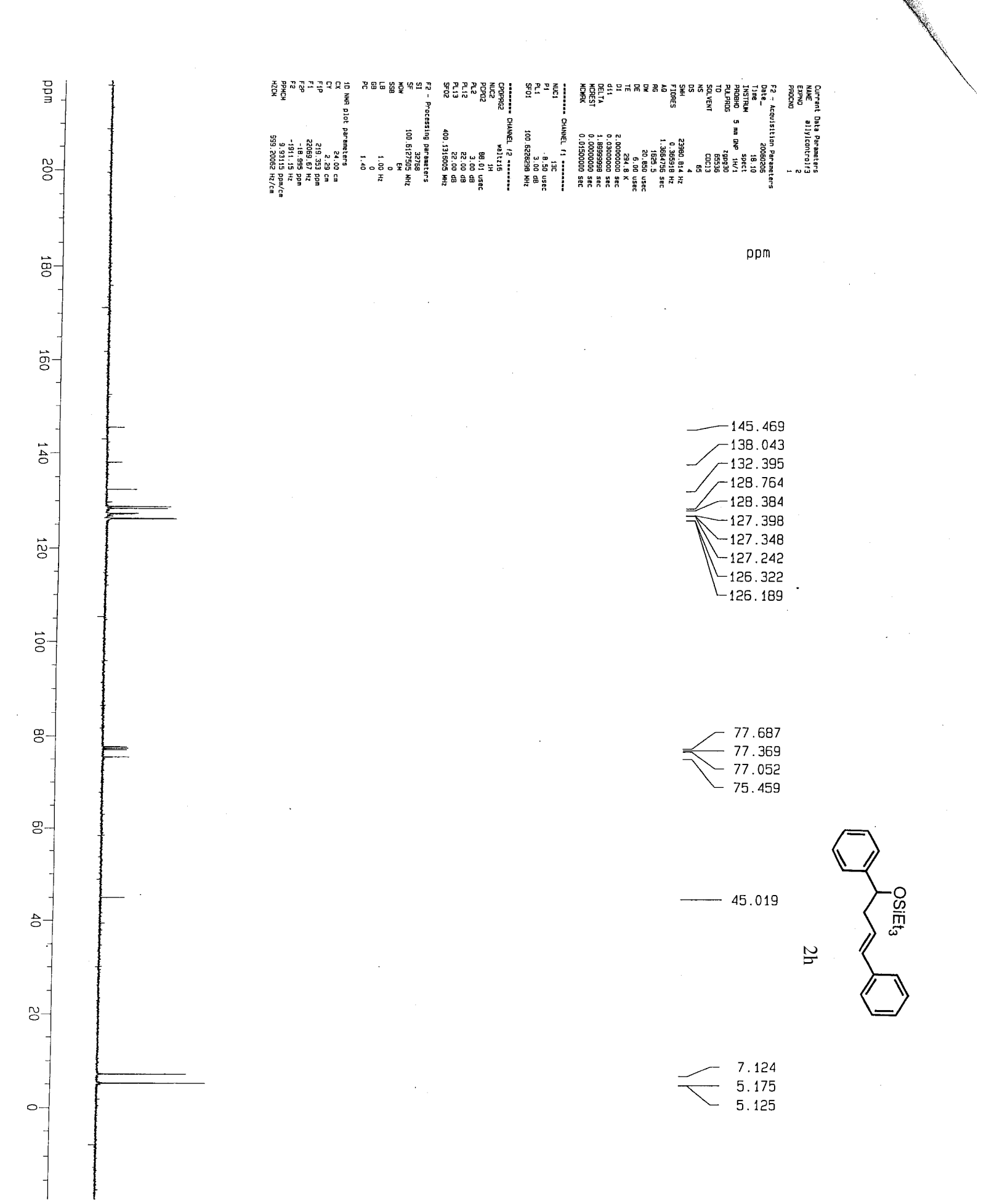
F2 - Acquisition Parameters
Date_           20060206
Time               18.07
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    1
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 35.9
DW               60.400 usec
DE                6.00 usec
TE                294.8 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

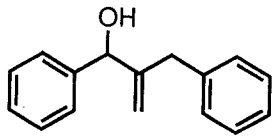
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.88 usec
PL1                3.00 dB
SFO1        400.1324710 MHz

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

1D NMR plot parameters
CX                24.00 cm
CY                 9.71 cm
F1P              10.500 ppm
F1               4201.36 Hz
F2P              -0.500 ppm
F2               -200.07 Hz
PPMCM            0.45833 ppm/cm
HZCM           183.39291 Hz/cm
```




Current Data Parameters
NAME allylcontrolf3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060206
Time 18.10
INSTRUM spect
PROBHD 5 mm DNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 65
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664795 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.01 usec
PL2 3.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127505 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
CY 2.29 cm
F1P 219.353 ppm
F1 22069.67 Hz
F2P -18.995 ppm
F2 -1911.15 Hz
PPMCM 9.93115 ppm/cm
HZCM 999.20062 Hz/cm
ppm
145.469
138.043
132.395
128.764
128.384
127.398
127.348
127.242
126.322
126.189
77.687
77.369
77.052
75.459
45.019
7.124
5.175
5.125
OSiEt3
2h



TES deprotection, Apdt

2h' (TES group deprotected)

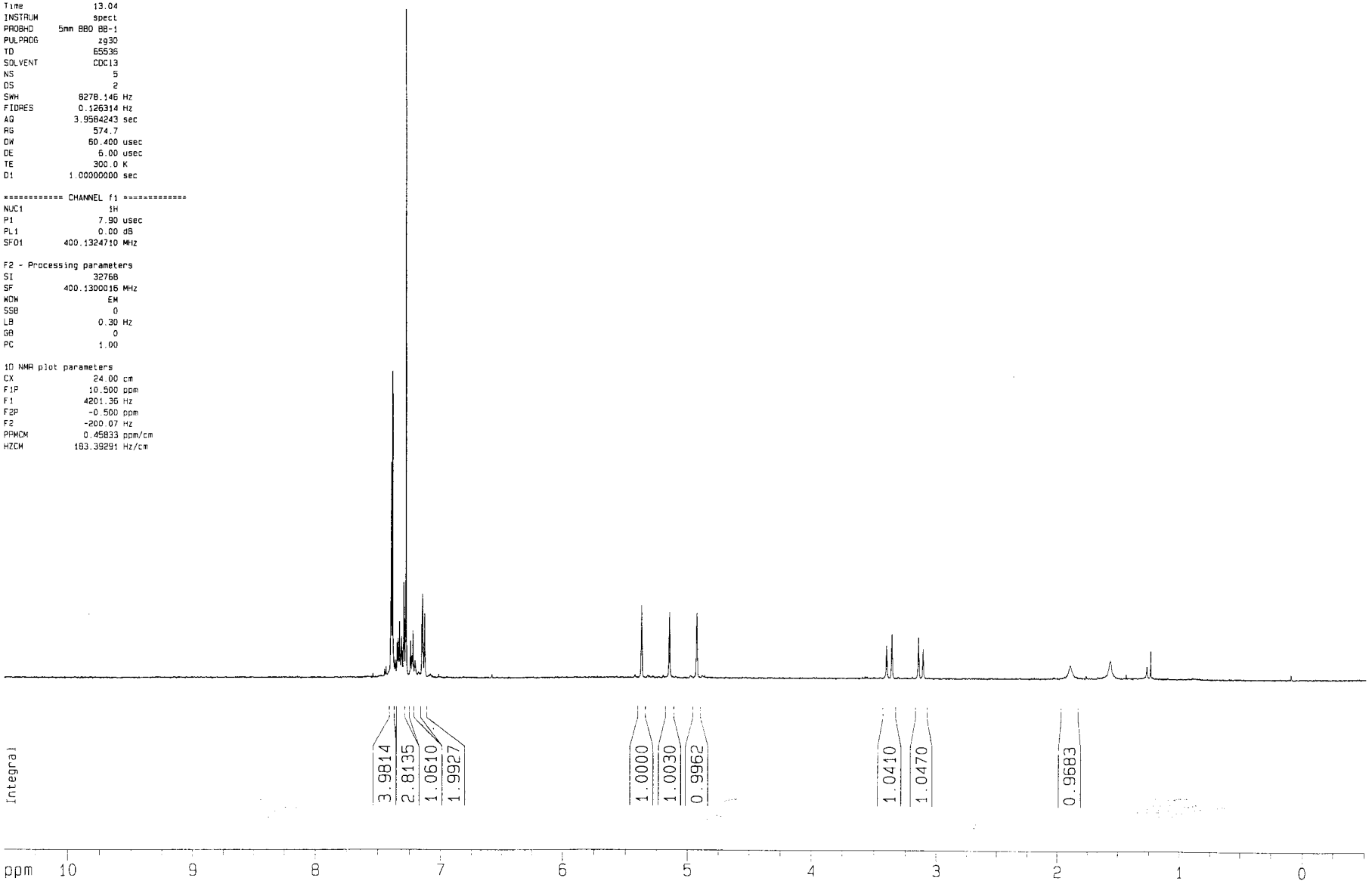
```
Current Data Parameters
NAME      hcy8-32phde-Ap
EXPNO                  1
PROCNO                 1

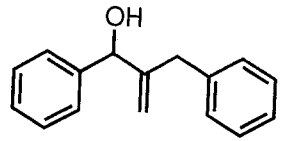
F2 - Acquisition Parameters
Date_           20060208
Time              13.04
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ             3.9584243 sec
RG                 574.7
DW                60.400 usec
DE                 6.00 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          400.1300016 MHz
WDW                   EM
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
PPMCM           0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```





2h' (TES group deprotected)

```
Current Data Parameters
NAME      hcy8-32phde-Ap
EXPNO                  2
PROCNO                 1

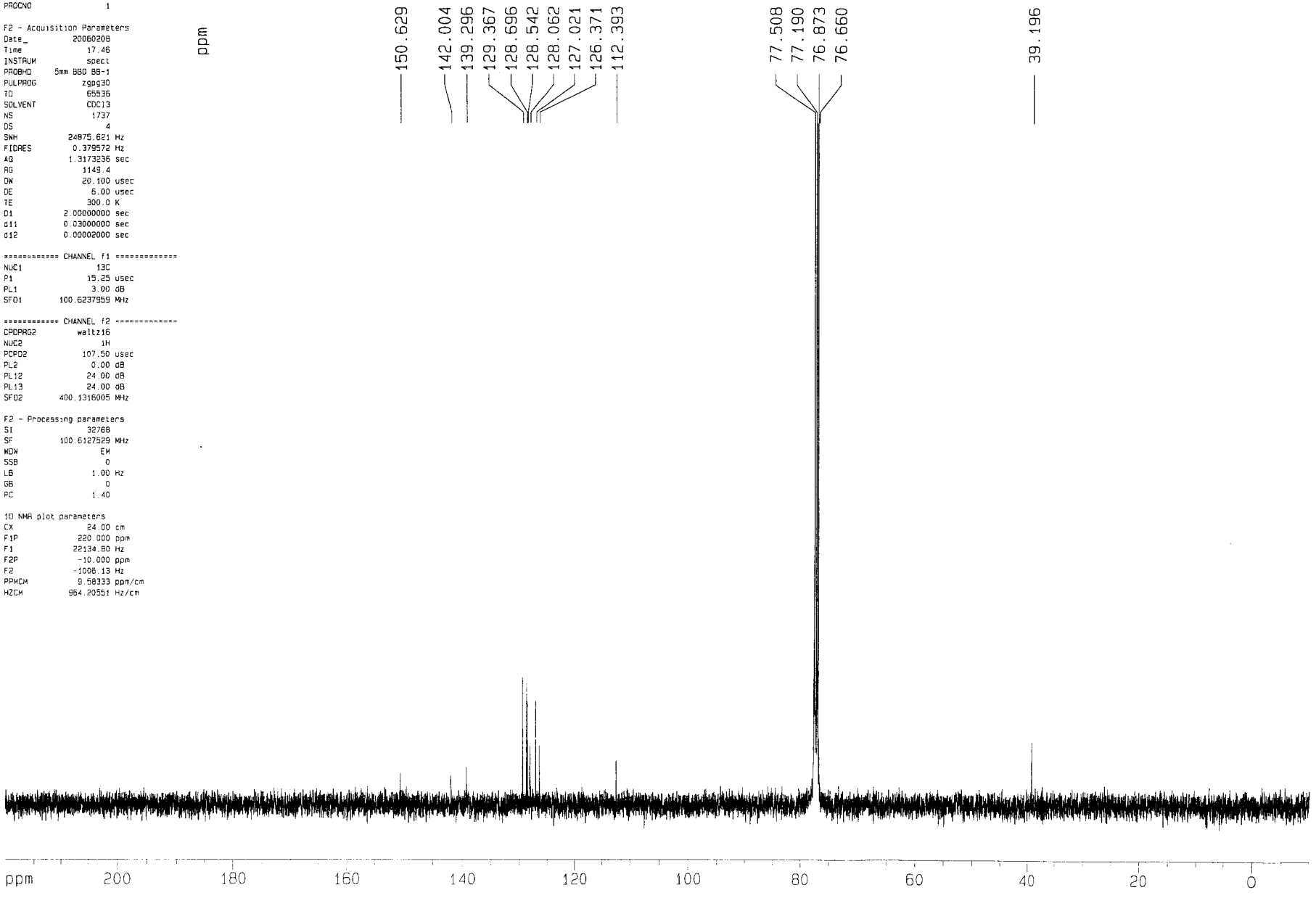
F2 - Acquisition Parameters
Date_           20060208
Time               17.46
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                  1737
DS                     4
SWH           24875.621 Hz
FIDRES         0.379572 Hz
AQ             1.3173236 sec
RG                1149.4
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                15.25 usec
PL1                3.00 dB
SFO1        100.6237959 MHz

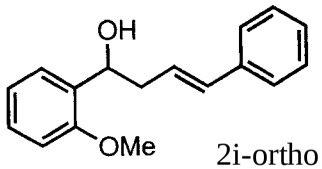
============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             107.50 usec
PL2                0.00 dB
PL12              24.00 dB
PL13              24.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127529 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 24.00 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM           9.58333 ppm/cm
HZCM          964.20551 Hz/cm
```



`o-OMe, TES deprot



2i-ortho

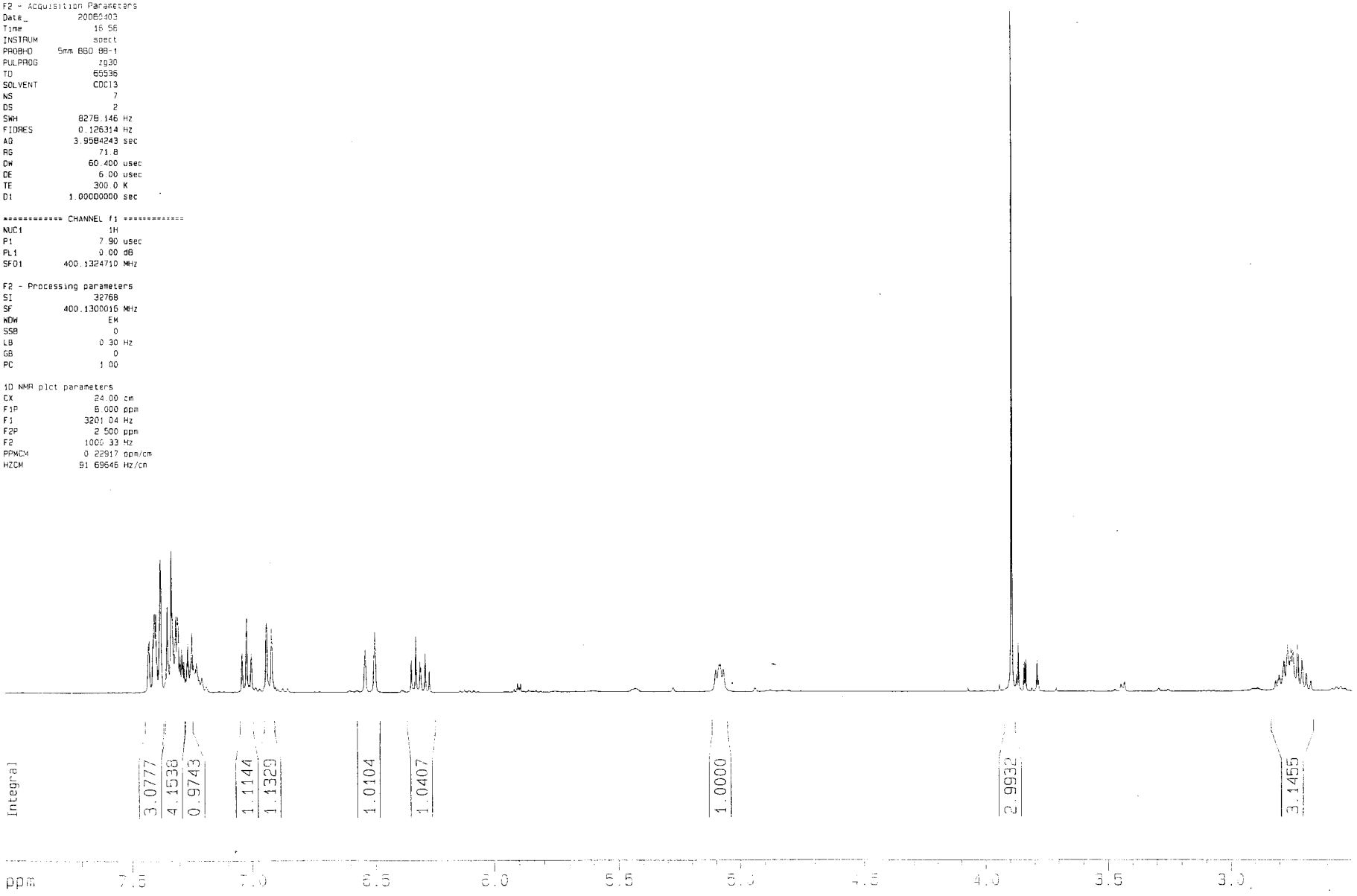
```
Current Data Parameters
NAME          pcyB-165pe
EXPNO                  1
PROCNO                 1

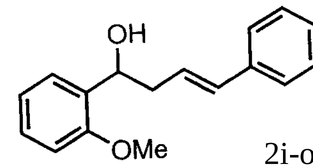
F2 - Acquisition Parameters
Date_          20060403
Time              16.56
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   7
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                71.8
DW               60.400 usec
DE                6.00 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          400.1300015 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               24.00 cm
F1P               6.000 ppm
F1             3201.04 Hz
F2P               2.500 ppm
F2             1000.33 Hz
PPMCM          0.22917 ppm/cm
HZCM          91.69646 Hz/cm
```





2i-ortho

Current Data Parameters
NAME ncy8-155pc
EXPNO 22
PROCNO 1

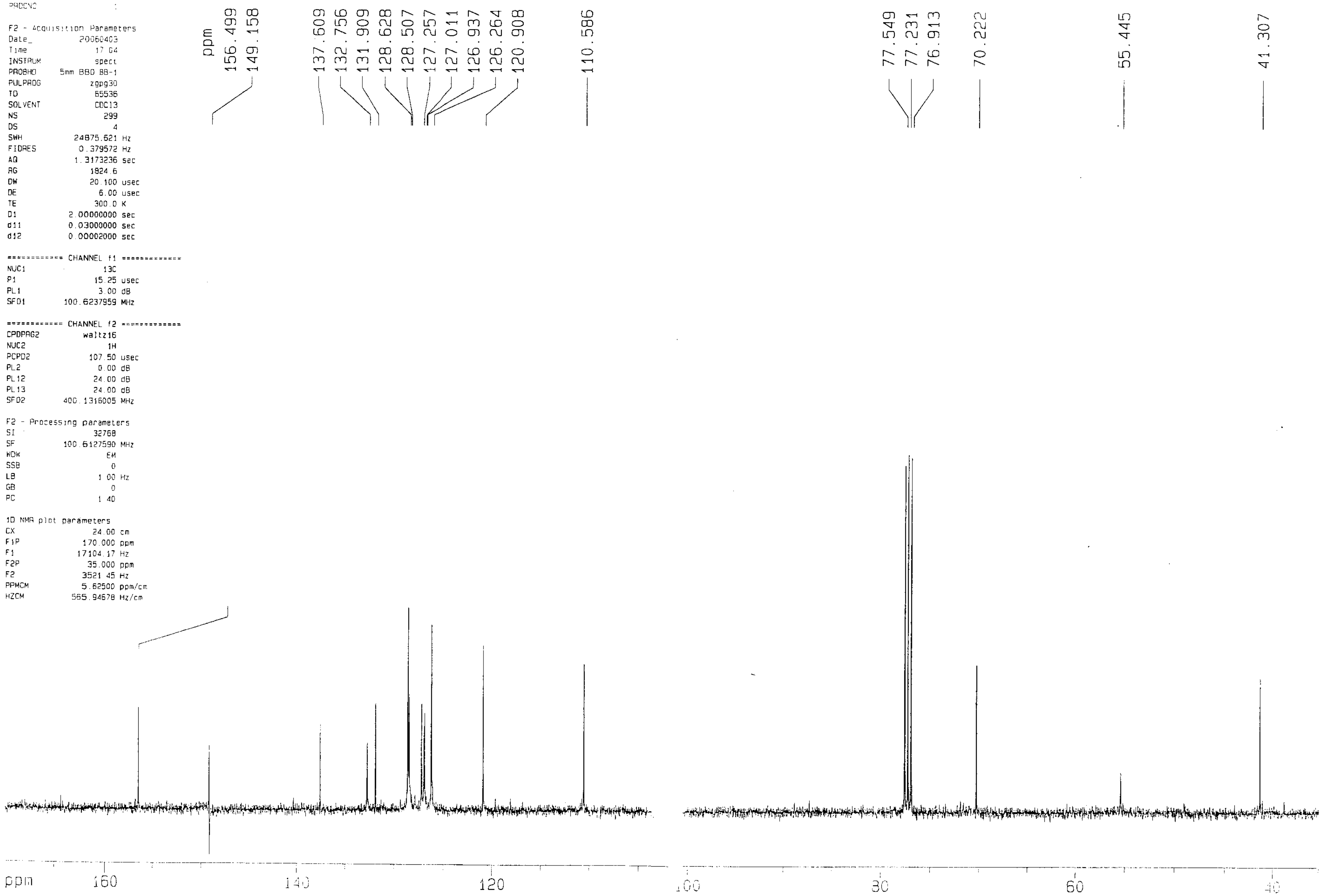
F2 - Acquisition Parameters
Date_ 20060403
Time 17.04
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 299
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1824.6
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

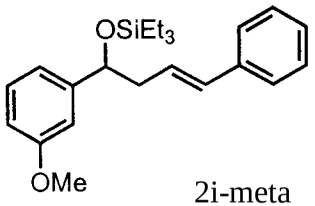
============ CHANNEL f1 ============
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 24.00 cm
F1P 170.000 ppm
F1 17104.17 Hz
F2P 35.000 ppm
F2 3521.45 Hz
PPMCM 5.62500 ppm/cm
HZCM 565.94678 Hz/cm





2i-meta

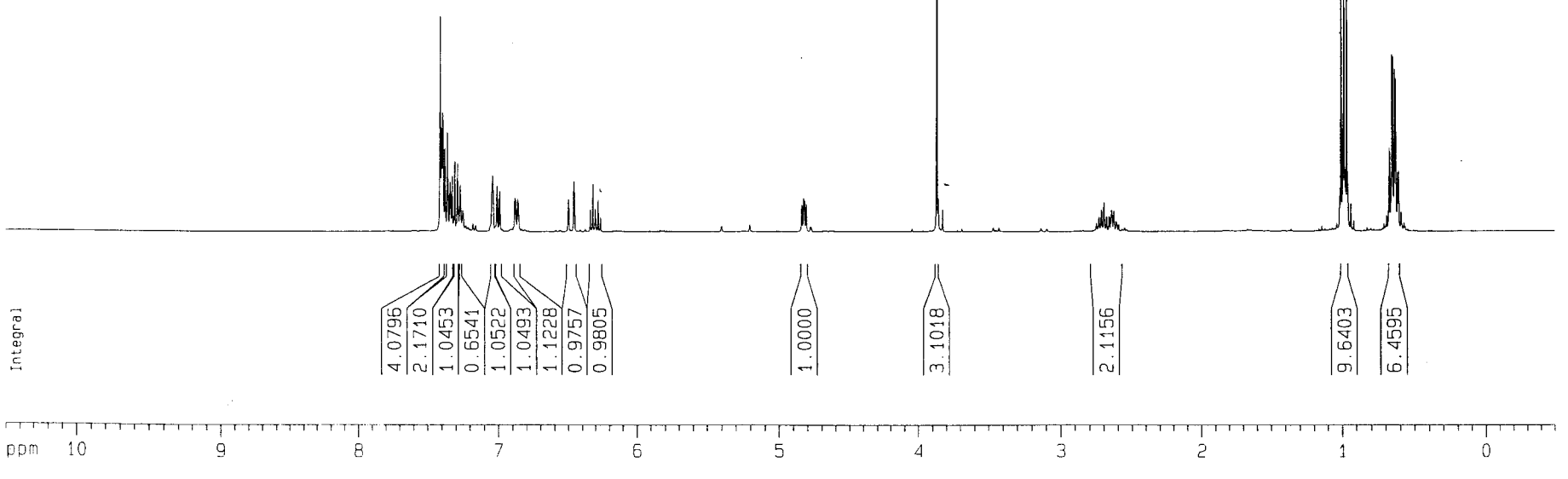
```
Current Data Parameters
NAME         hcy8-156pf2
EXPNO                  1
PROCNO                 1

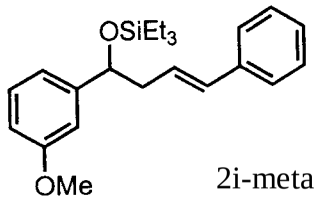
F2 - Acquisition Parameters
Date_           20060403
Time               12.45
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                     8
DS                     1
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                  28.5
DW             60.400 usec
DE              6.00 usec
TE              292.6 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

-------- CHANNEL f1 --------
NUC1                   1H
P1                  9.88 usec
PL1                 3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300054 MHz
WDW                   EM
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
PPMCM          0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```



2i-meta

```
Current Data Parameters
NAME          hcy6-156pf2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_          20060403
Time               12.51
INSTRUM            spect
PROBHD  5 mm QNP  1H/1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   92
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                 3251
DW               20.850 usec
DE                6.00 usec
TE               292.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              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           100.6127615 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  24.00 cm
CY                   6.93 cm
F1P               219.353 ppm
F1              22069.67 Hz
F2P               -18.995 ppm
F2              -1911.15 Hz
PPMCM             9.93115 ppm/cm
HZCM            999.20081 Hz/cm
```

ppm

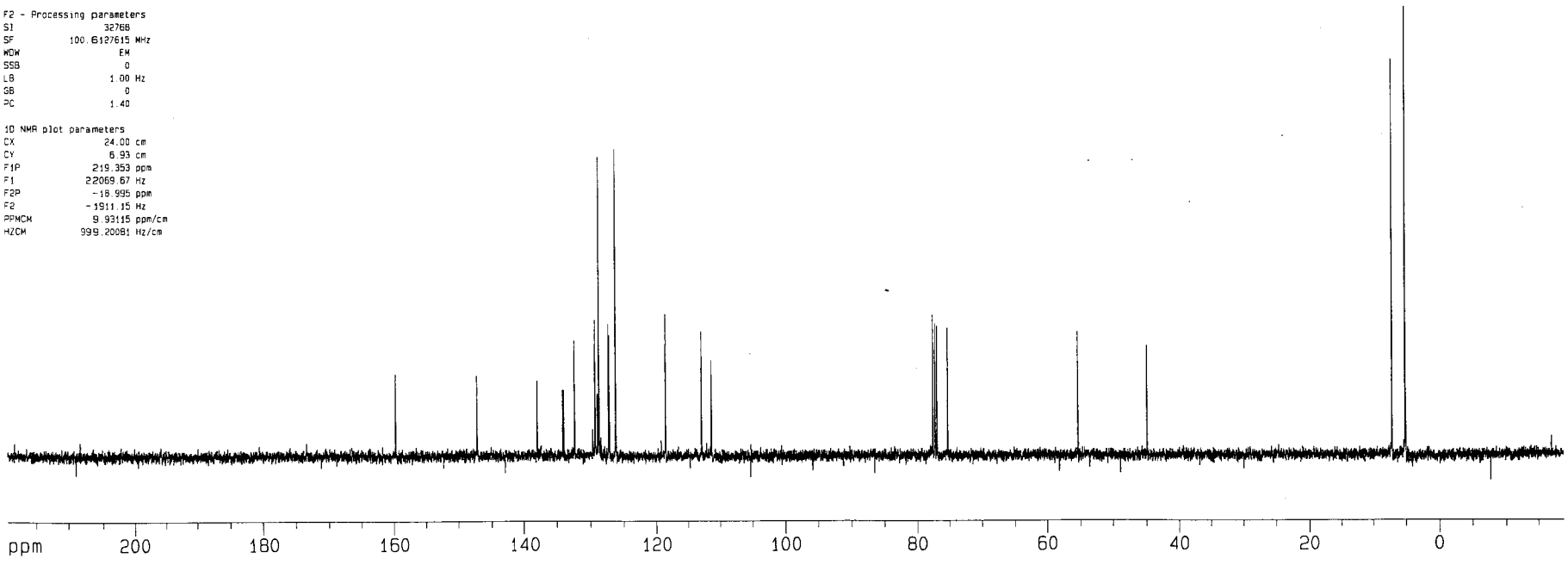
159.677 147.099 137.877 134.002 133.807 132.230 129.194 128.869 128.695 128.624 127.200 127.101 126.169 118.436 112.813 111.334

77.551 77.234 76.916 75.181

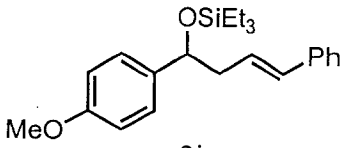
55.281

44.819

7.002 5.015



H-pdt



2i-para

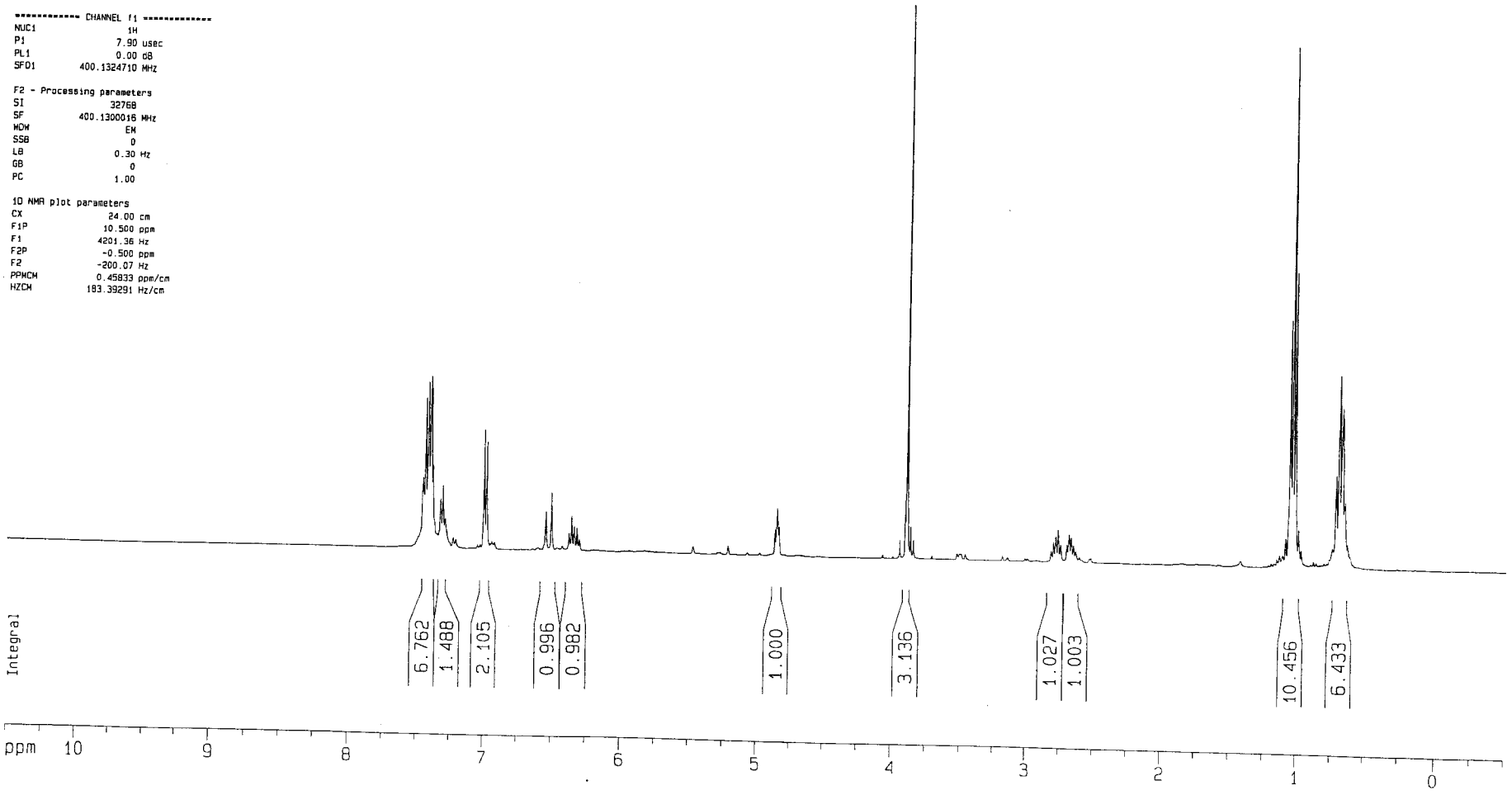
```
Current Data Parameters
NAME          ncy8-98Hpdt
EXPNO                    1
PROCNO                   1

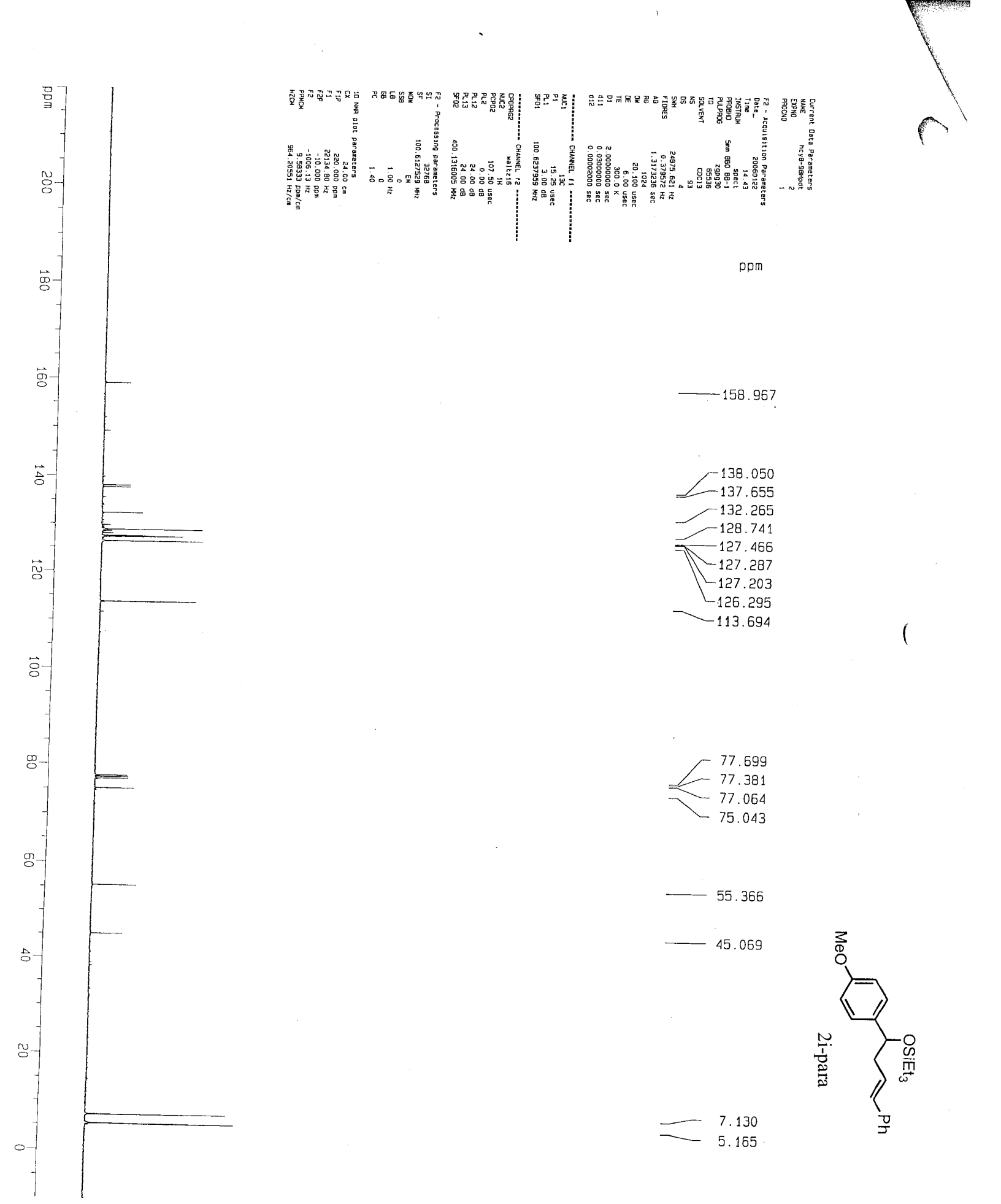
F2 - Acquisition Parameters
Date_          20060122
Time             14.39
INSTRUM          spect
PROBHD     5mm BBO BB-1
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                   5
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                20.2
DW               60.400 usec
DE                6.00 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          400.1300016 MHz
WDW                  EM
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
PPMCM          0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```




Current Data Parameters
NAME mcyB-98hpot
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060122
Time 14.43
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 93
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
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 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
158.967
138.050
137.655
132.265
128.741
127.466
127.287
127.203
126.295
113.694
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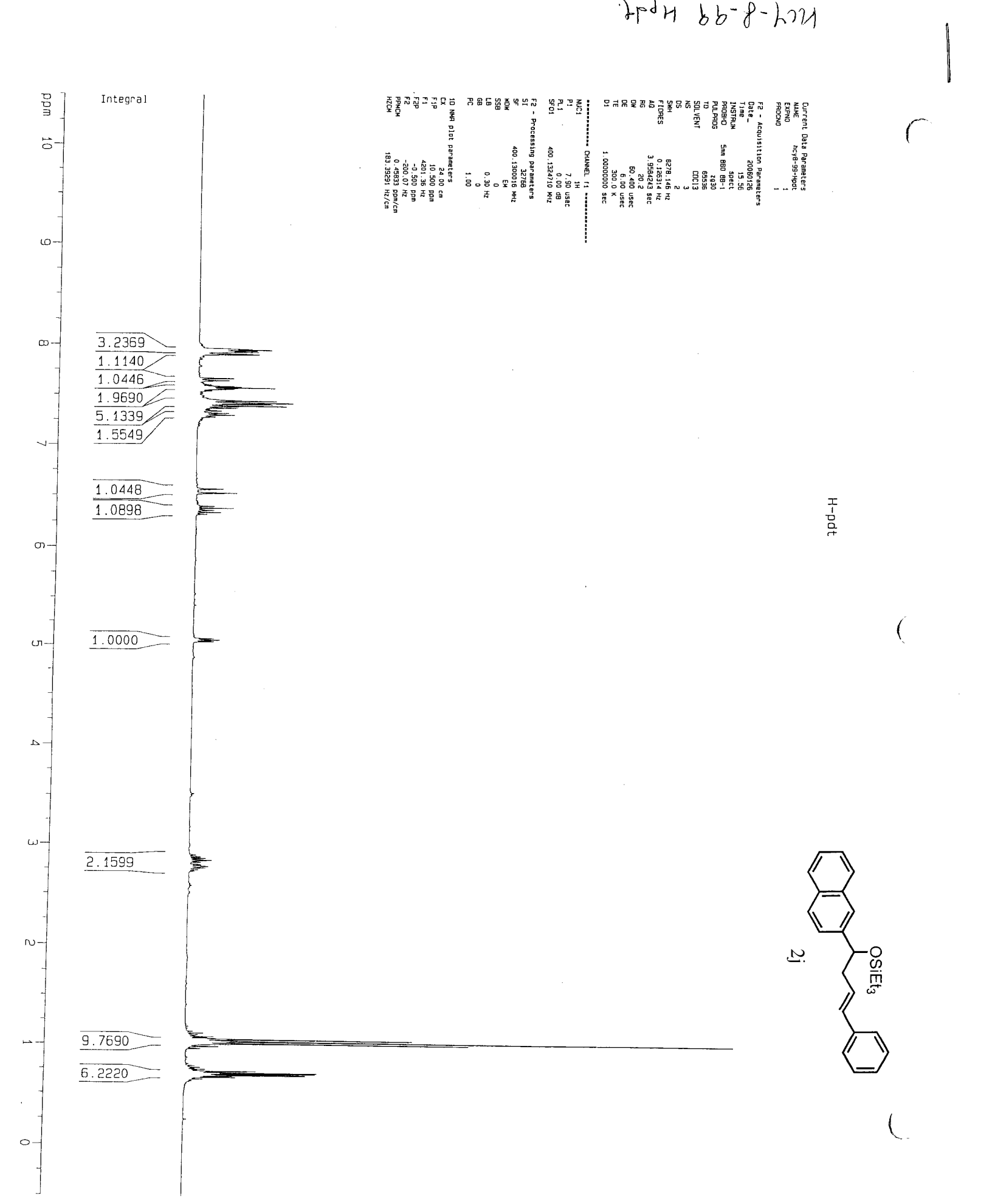
5.165
MeO
OSiEt3
Ph
2i-para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1.0446
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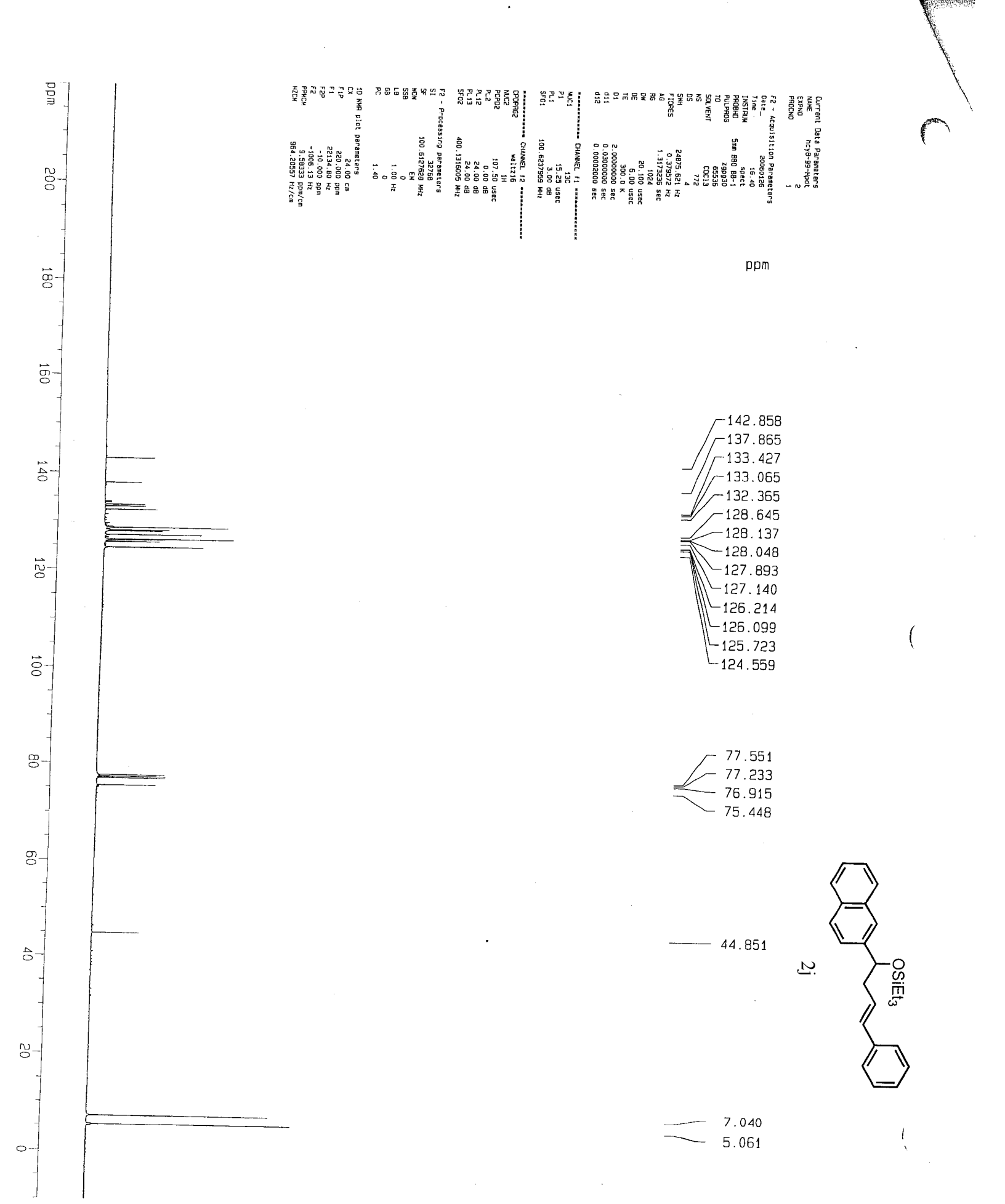
1.0448
1.0898
1.0000
2.1599
9.7690
6.2220
H-pdt
OSiEt3
2j


Current Data Parameters
NAME ncy8-99-Hpdt
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060126
Time 16.40
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 772
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
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 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127628 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.28557 Hz/cm
ppm
142.858
137.865
133.427
133.065
132.365
128.645
128.137
128.048
127.893
127.140
126.214
126.099
125.723
124.559
77.551
77.233
76.915
75.448
44.851
7.040
5.061
OSiEt3
2j
ppm
200
180
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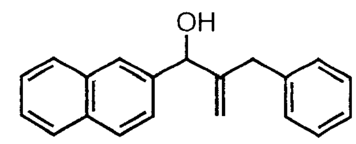
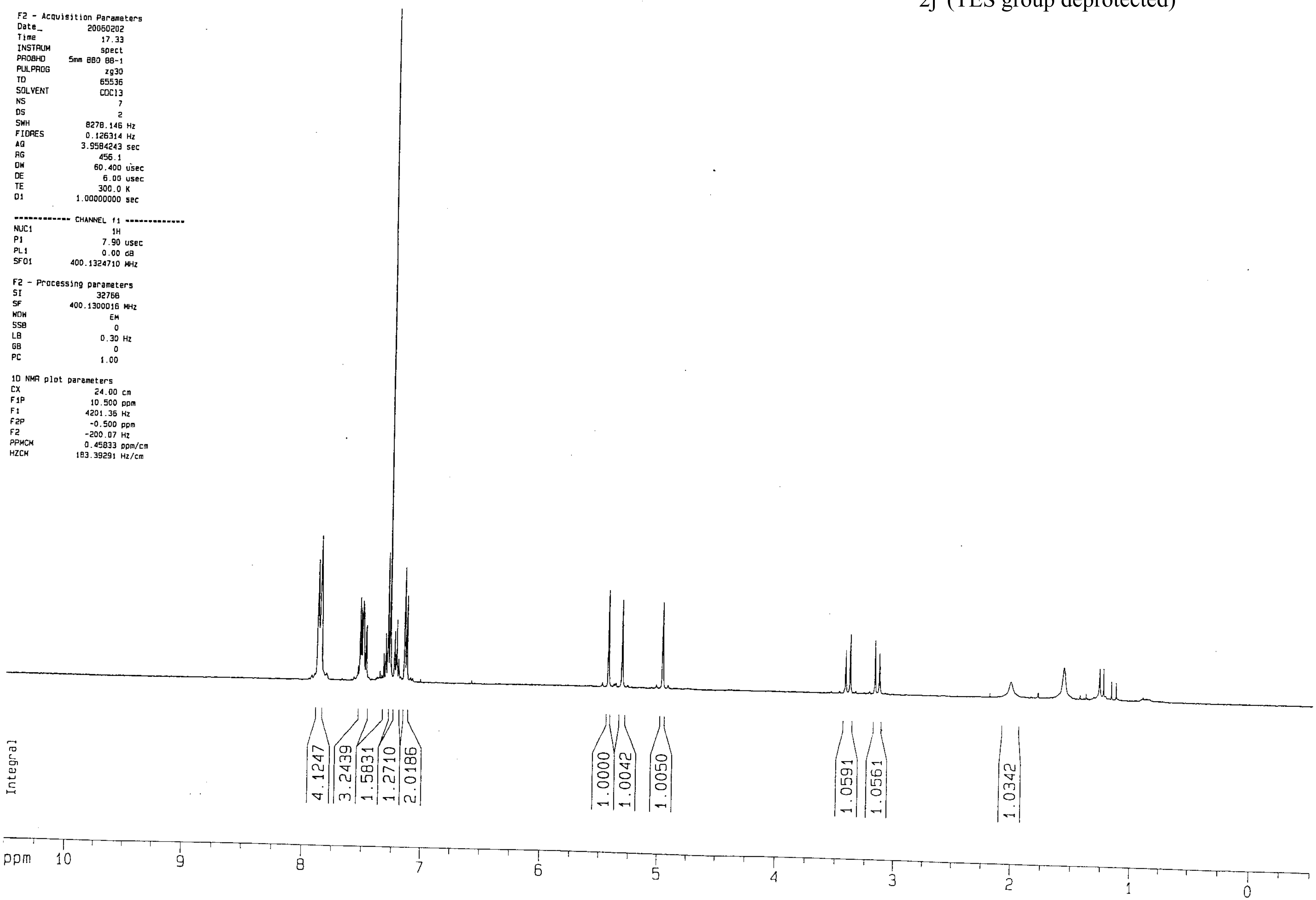
140
120
100
80
60
40
20
0

hcy-8-104 de - Apdt.



2j' (TES group deprotected)

TES deprotection, Apdt



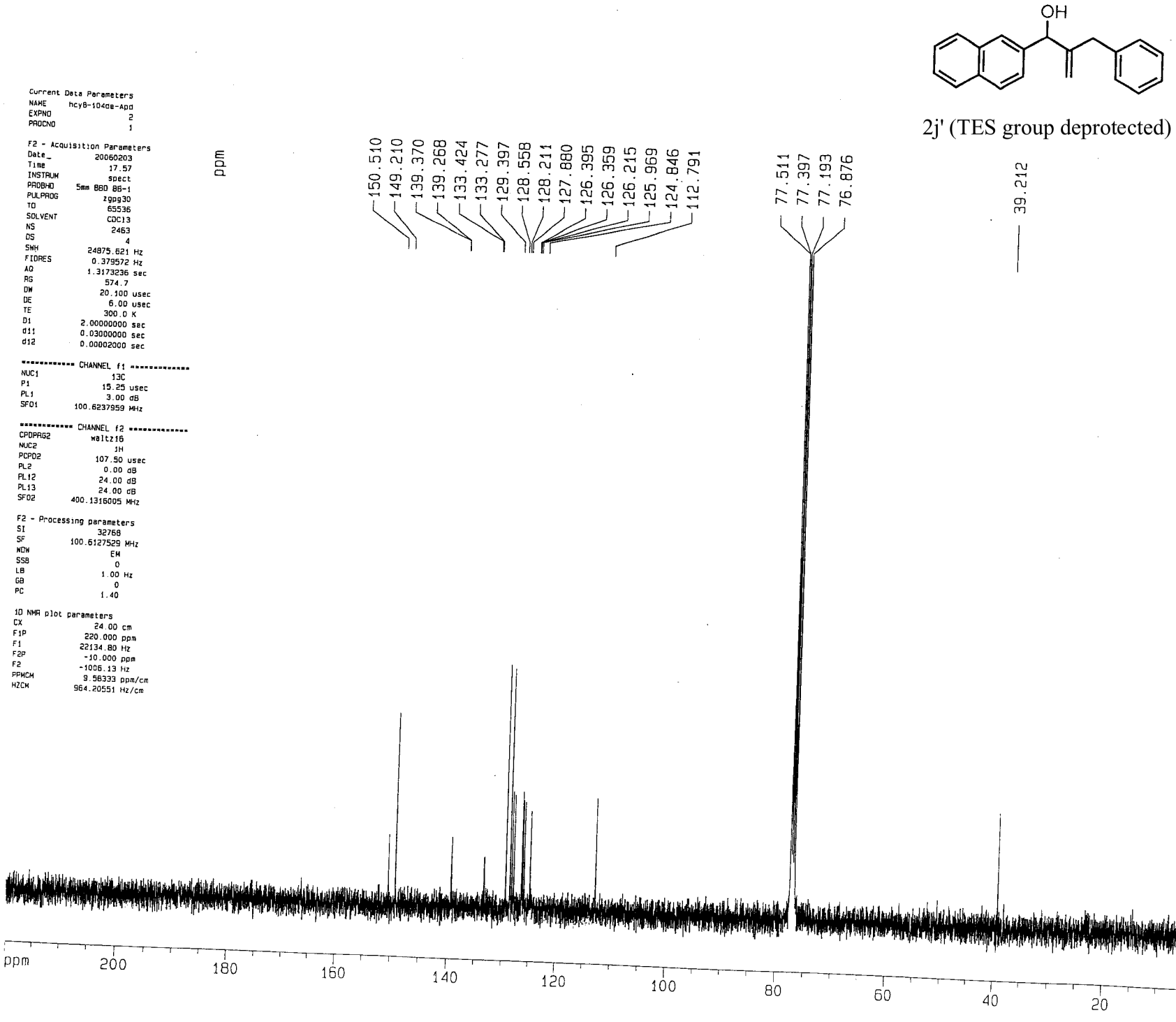
Current Data Parameters
NAME hcy8-104de-Apd
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060202
Time 17.33
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

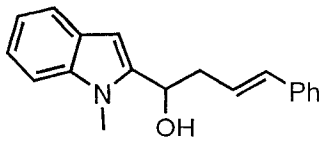
============ CHANNEL f1 ============
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300016 MHz
WDW EM
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
PPMCM 0.45833 ppm/cm
HZCM 183.39291 Hz/cm


OH
2j' (TES group deprotected)
Current Data Parameters
NAME hcy8-104de-Apd
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060203
Time 17.57
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2463
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 574.7
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 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
150.510
149.210
139.370
139.268
133.424
133.277
129.397
128.558
128.211
127.880
126.395
126.359
126.215
125.969
124.846
112.791
77.511
77.397
77.193
76.876
39.212
ppm
200
180
160
140
120
100
80
60
40
20
0

Me-indo-2-CHO AllylPh coupling, H pdt deprotection



2k (TES group deprotected)

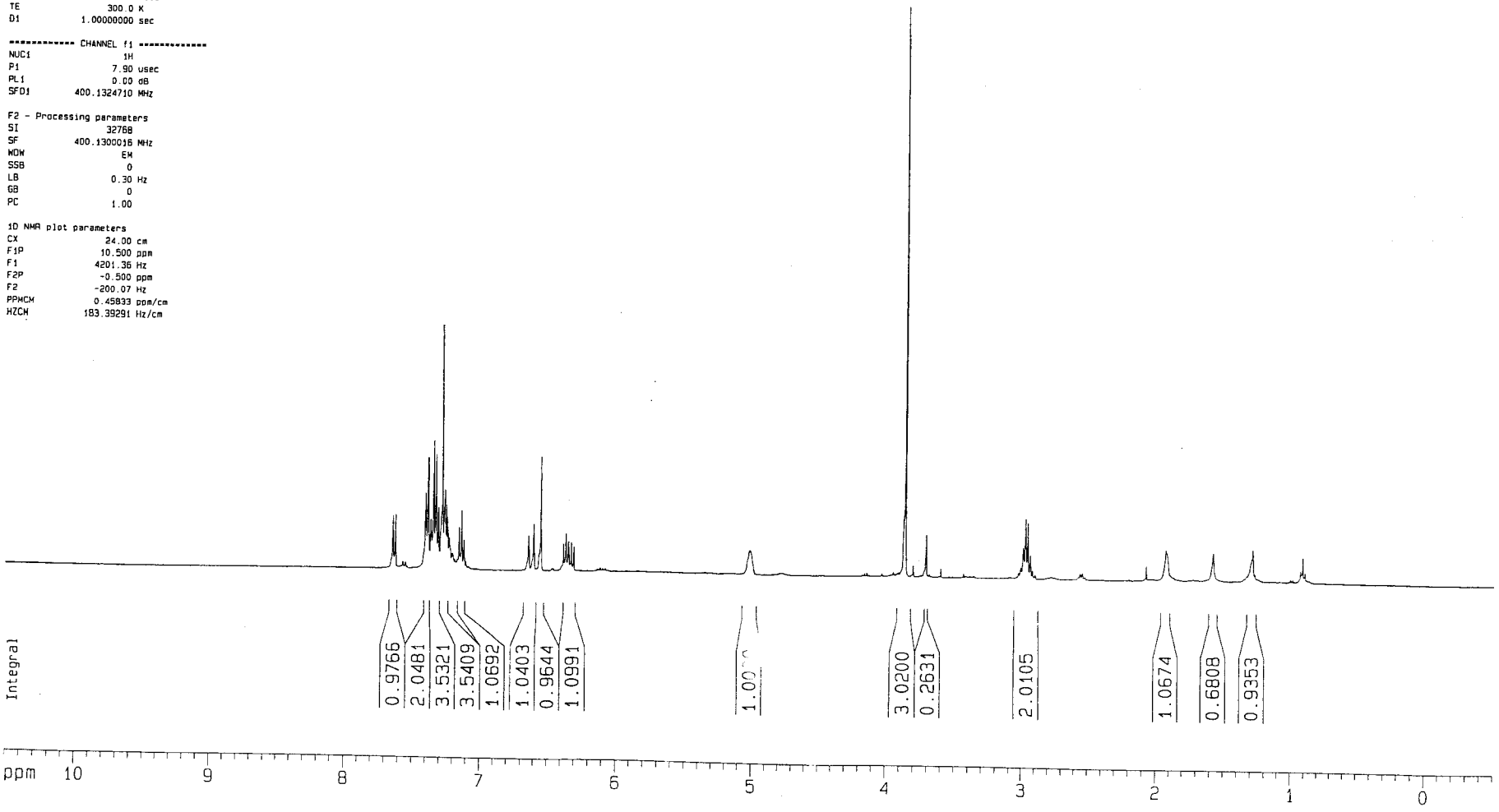
```
Current Data Parameters
NAME      hcyB-70de-Hpdt
EXPNO                  1
PROCNO                 1

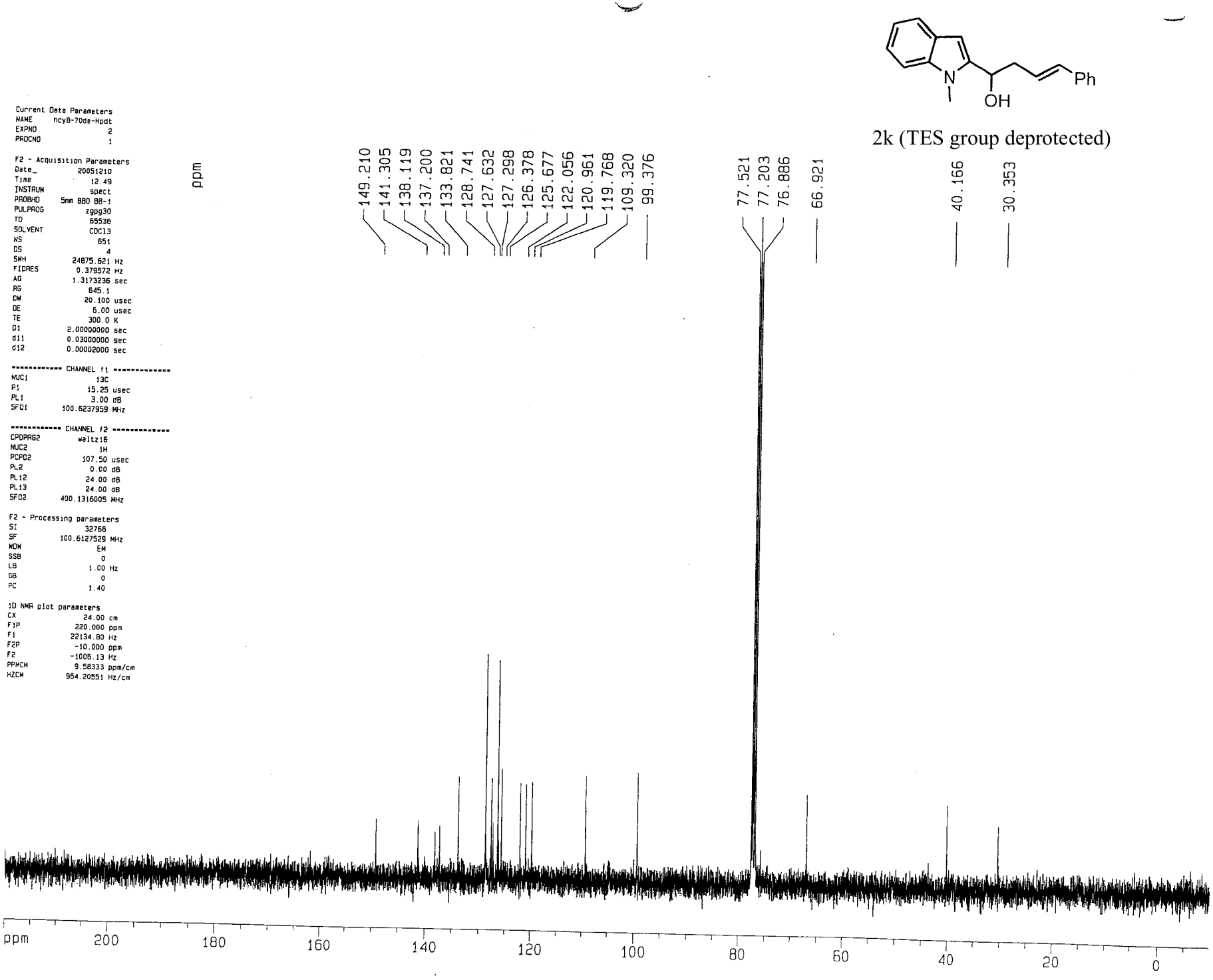
F2 - Acquisition Parameters
Date_           20051210
Time               12.45
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                287.4
DW               60.400 usec
DE                6.00 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          400.1300016 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

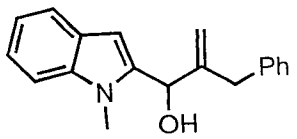
1D NMR plot parameters
CX                24.00 cm
F1P               10.500 ppm
F1               4201.36 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM             0.45833 ppm/cm
HZCM            183.39291 Hz/cm
```





2k (TES group deprotected)

Cy2PPh f9 deprotection, f6



2k' (TES group deprotected)

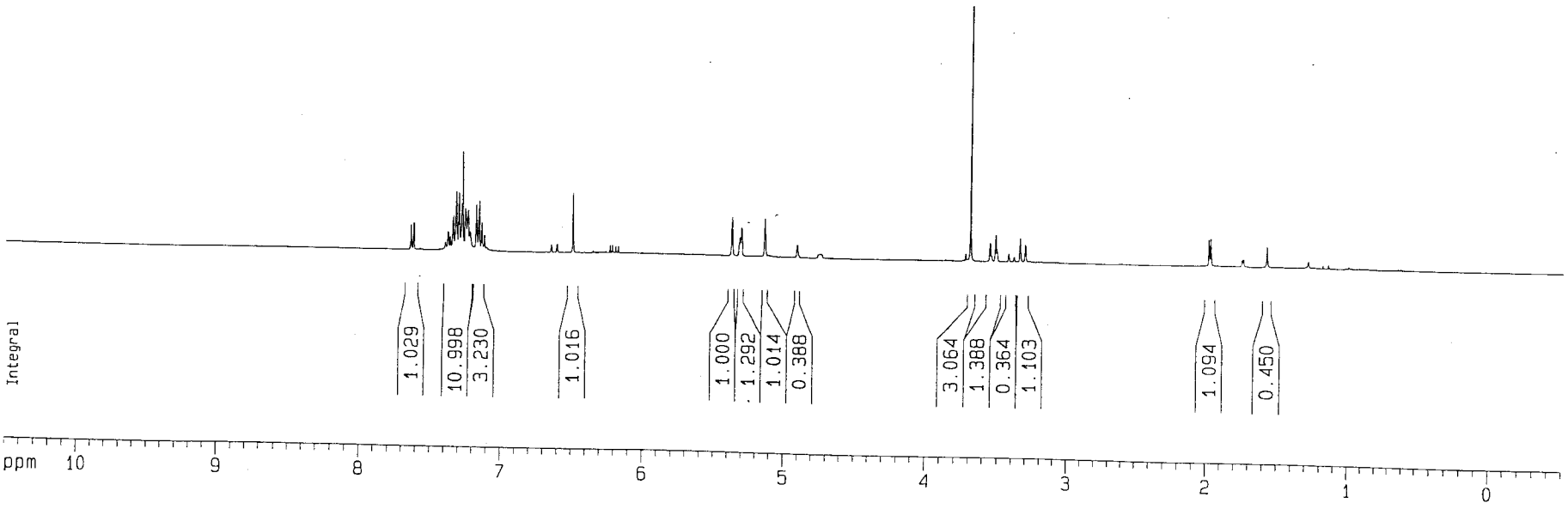
```
Current Data Parameters
NAME      hcy8-70dcyf9-6
EXPNO                1
PROCNO               1

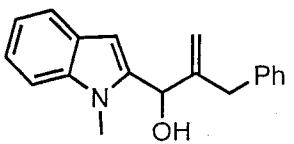
F2 - Acquisition Parameters
Date_         20051212
Time             14.52
INSTRUM          spect
PROBHD     5mm BBO BB-1
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  12
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 181
DW              60.400 usec
DE               6.00 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          400.1300016 MHz
WDW                  EM
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
PPMCM          0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```





2k' (TES group deprotected)

```
Current Data Parameters
NAME      hcy8-70dcyf9-6
EXPNO                  2
PROCNO                 1

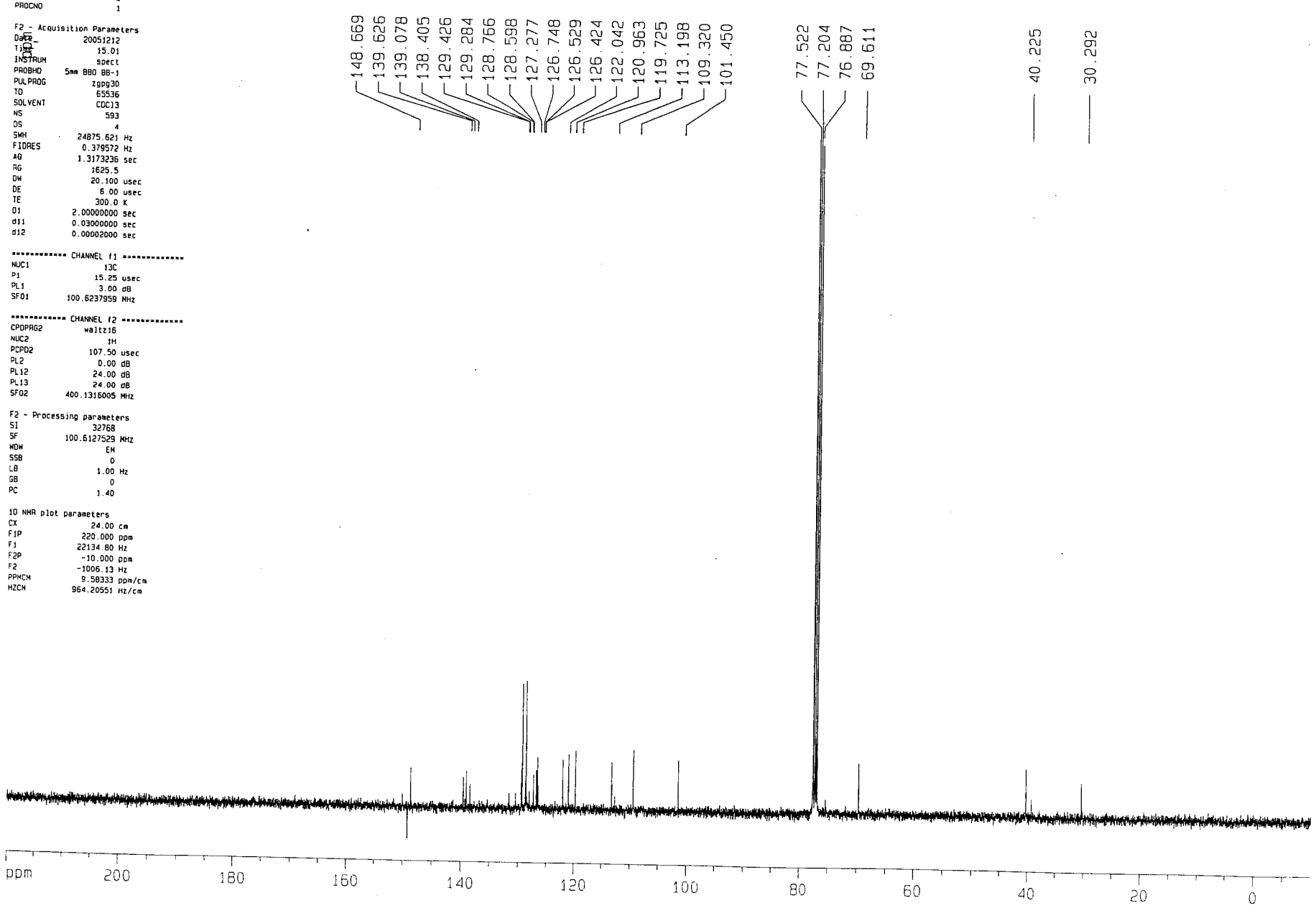
F2 - Acquisition Parameters
Date_          20051212
Time              15.01
INSTRUM             spect
PROBHD   5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  593
DS                    4
SWH         24875.621 Hz
FIDRES       0.379572 Hz
AQ           1.3173236 sec
RG              1625.5
DW             20.100 usec
DE              6.00 usec
TE             300.0 K
D1          2.00000000 sec
d11         0.03000000 sec
d12         0.00002000 sec

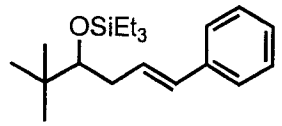
********** CHANNEL f1 **********
NUC1               13C
P1              15.25 usec
PL1              3.00 dB
SFO1       100.6237959 MHz

********** CHANNEL f2 **********
CPDPRG2         waltz16
NUC2                1H
PCPD2          107.50 usec
PL2              0.00 dB
PL12            24.00 dB
PL13            24.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127529 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                24.00 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM             9.58333 ppm/cm
HZCM            964.20551 Hz/cm
```





21

H-pdt

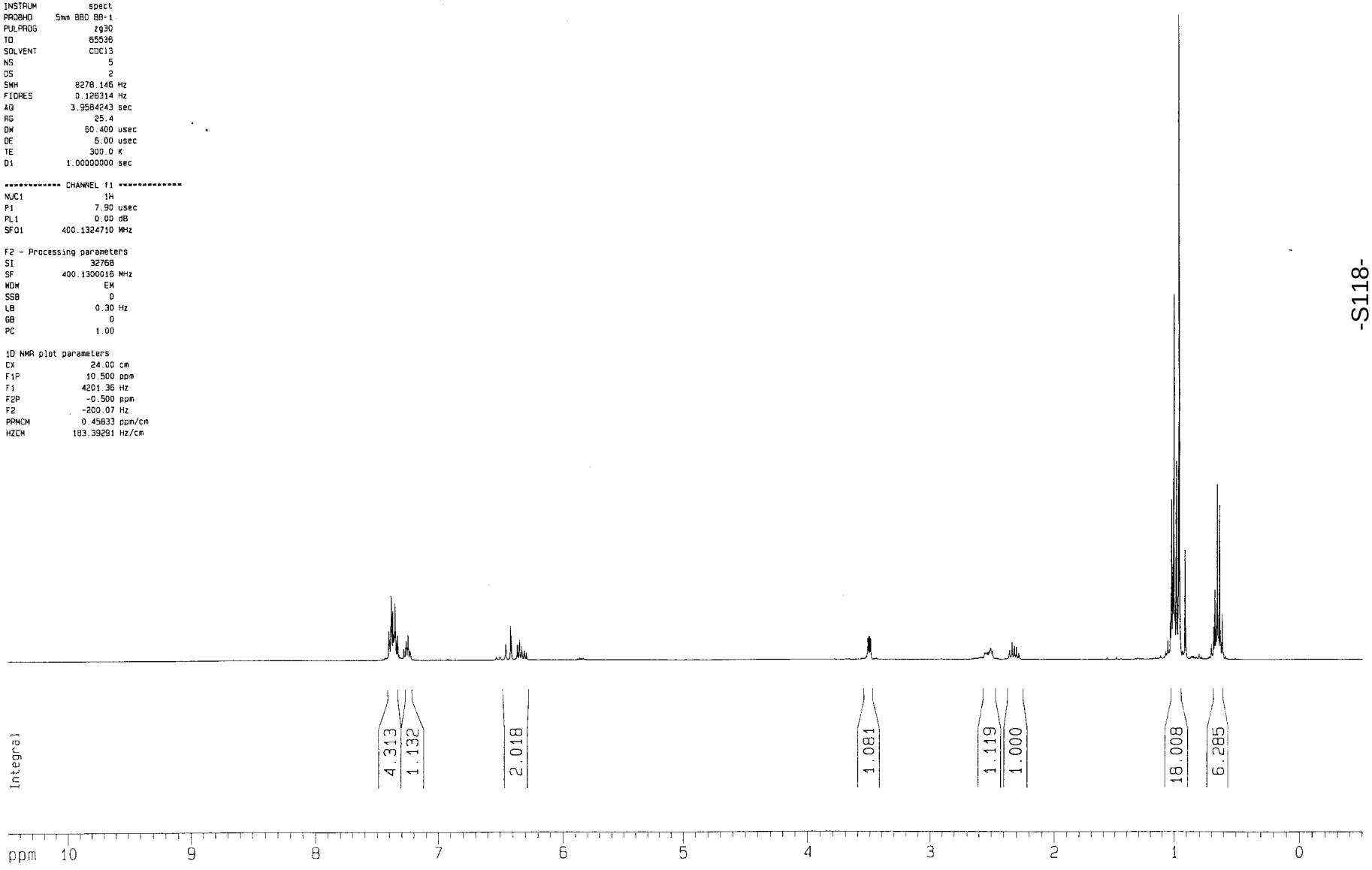
```
Current Data Parameters
NAME       hcy8-115-Hpdt
EXPNO                 1
PROCNO                1

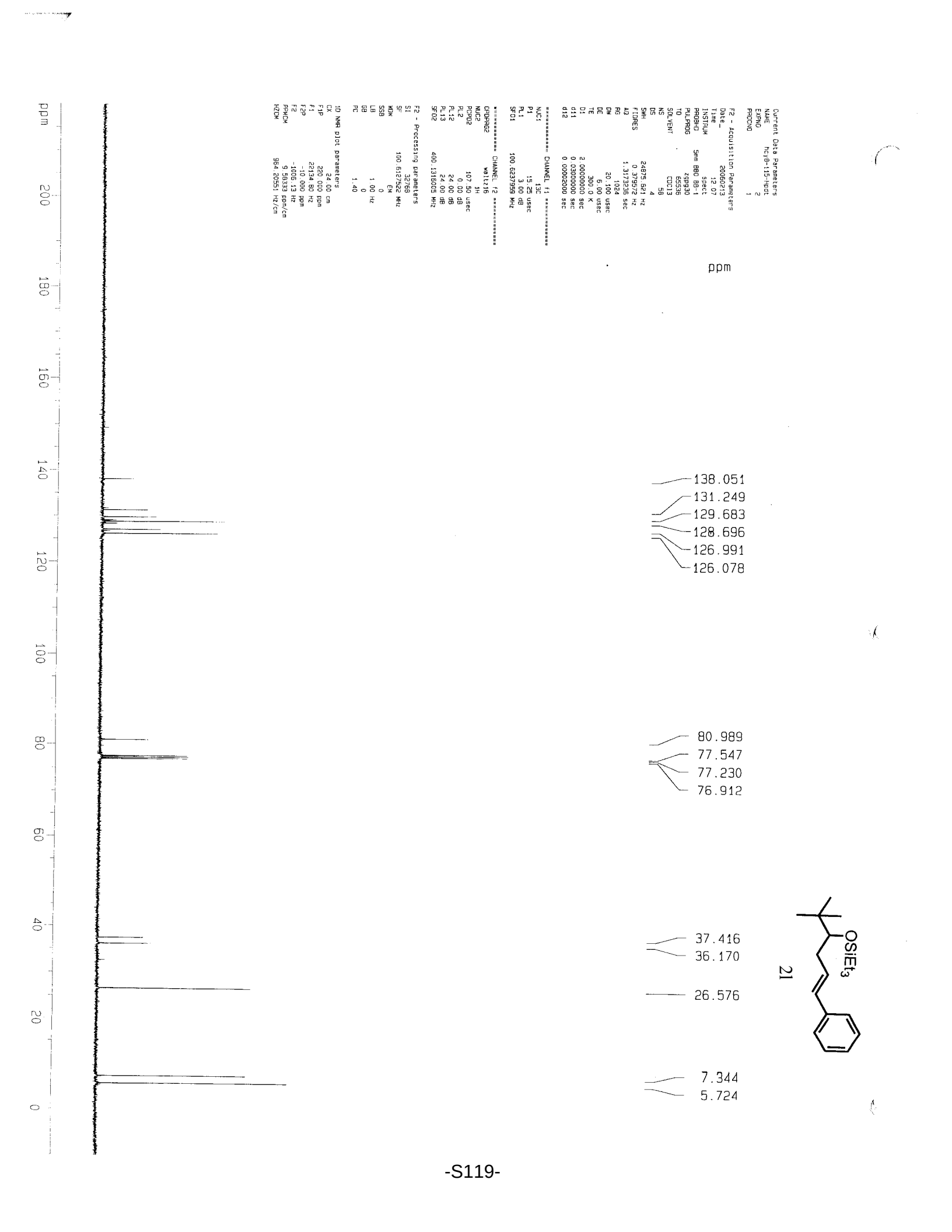
F2 - Acquisition Parameters
Date_          20060213
Time              12.03
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 25.4
DW               60.400 usec
DE                6.00 usec
TE                300.0 K
D1            1.00000000 sec

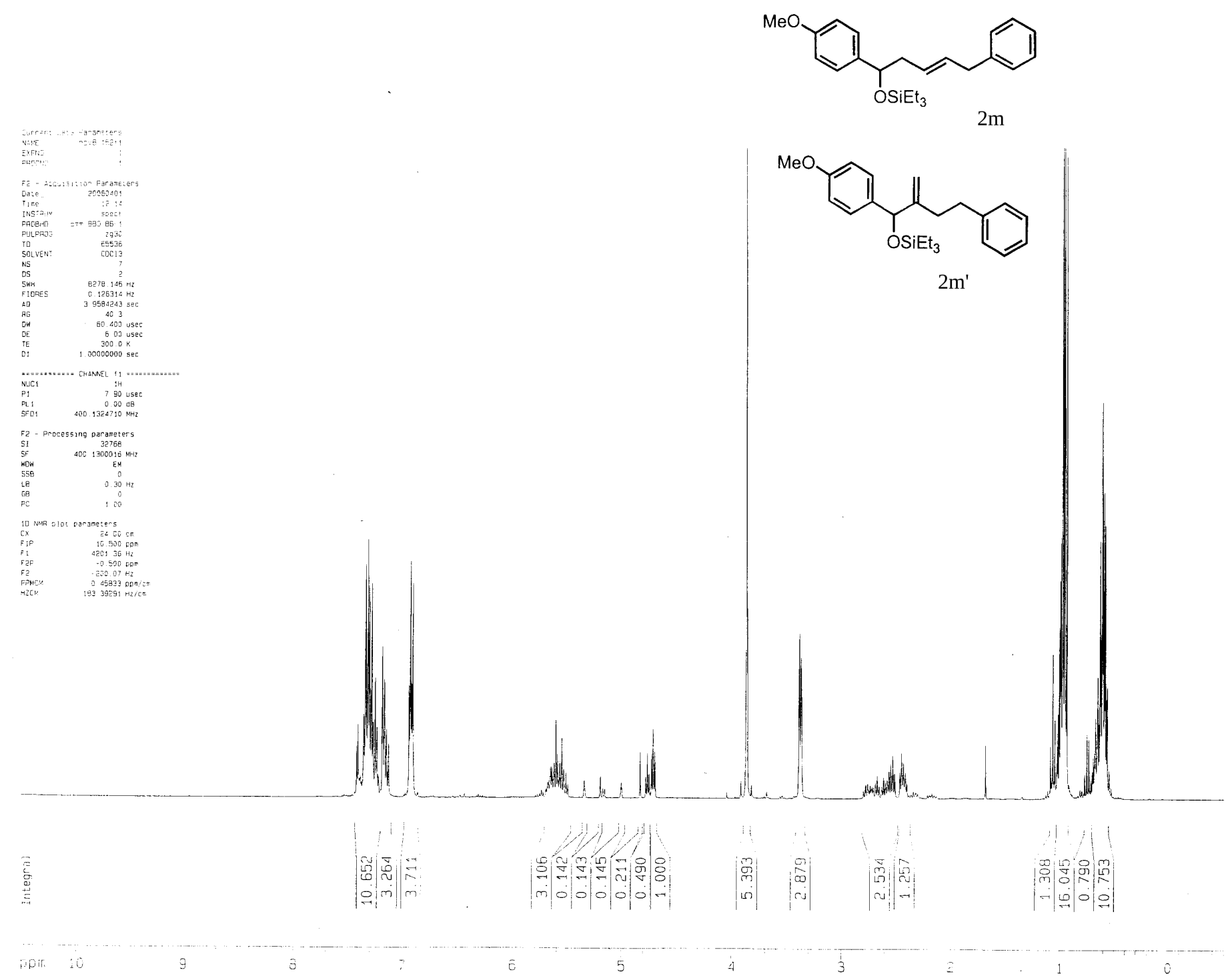
========== CHANNEL f1 ==========
NUC1                  1H
P1                 7.90 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

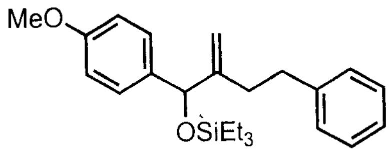
F2 - Processing parameters
SI                32768
SF          400.1300016 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                24.00 cm
F1P               10.500 ppm
F1              4201.36 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
PPMCM          0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```

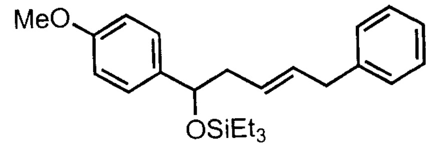



Current Data Parameters
NAME mcyb-115-Hpol
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060213
Time 12.07
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1 13C
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237956 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127522 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
138.051
131.249
129.683
128.696
126.991
126.078
80.989
77.547
77.230
76.912
37.416
36.170
26.576
7.344
5.724
OSiEt3
21


MeO
OSiEt3
2m
MeO
OSiEt3
2m'
Integral
10.652
3.264
3.711
3.106
0.142
0.143
0.145
0.211
0.490
1.000
5.393
2.879
2.534
1.257
1.308
16.045
0.790
10.753
ppm
10
9
8
7
6
5
4
3
2
1
0



2m'



2m

```
Current Data Parameters
NAME        ncl6 15241
EXPNO                2
PROCNO               1

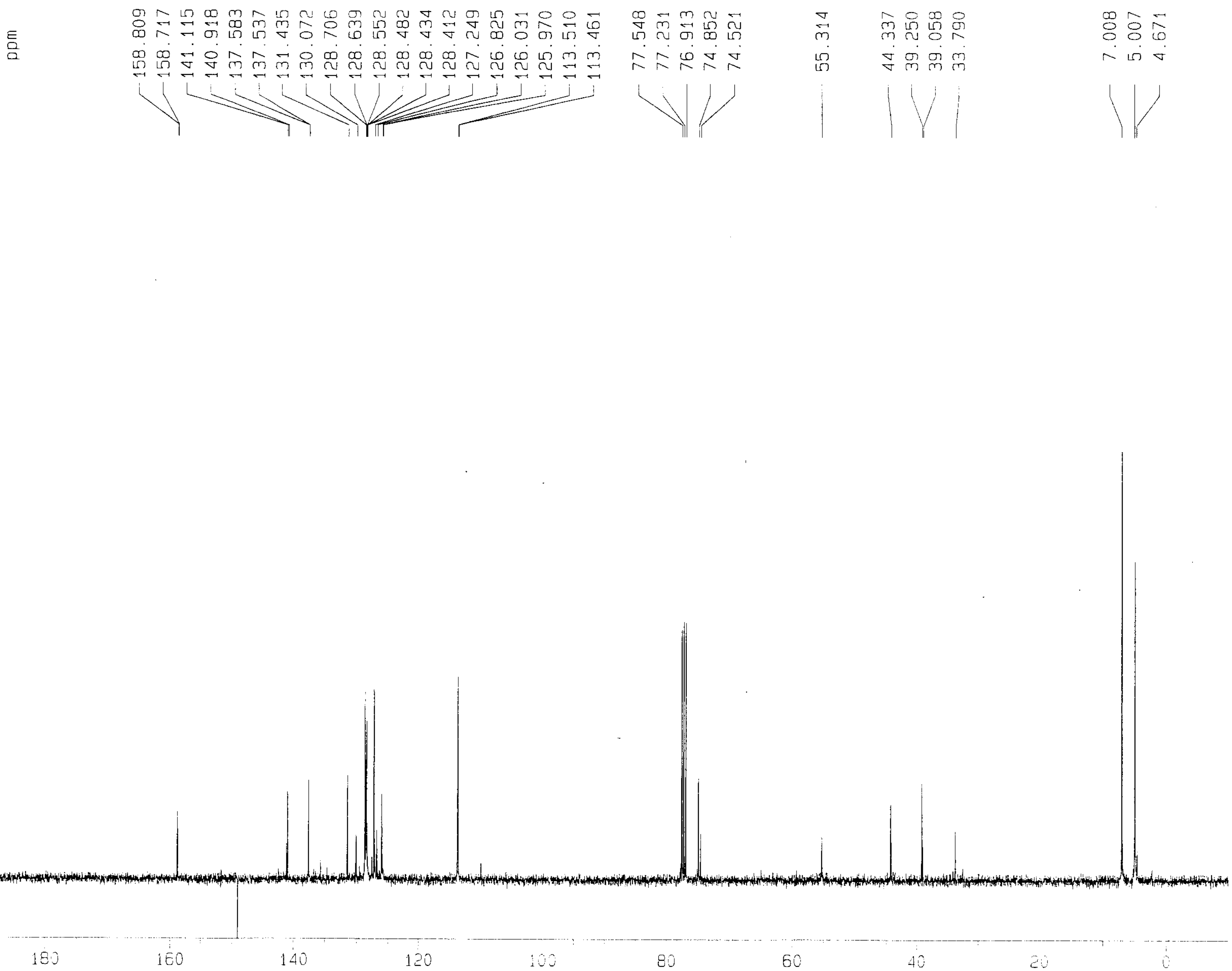
F2 - Acquisition Parameters
Date_         20060401
Time             12.16
INSTRUM           spect
PROBHD   5mm BBO BB-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 169
DS                   4
SWH         24875.621 Hz
FIDRES       0.379572 Hz
AQ          1.3173236 sec
RG                 512
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               15.25 usec
PL1               3.00 dB
SFO1      100.6237959 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2           107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2      400.1316005 MHz

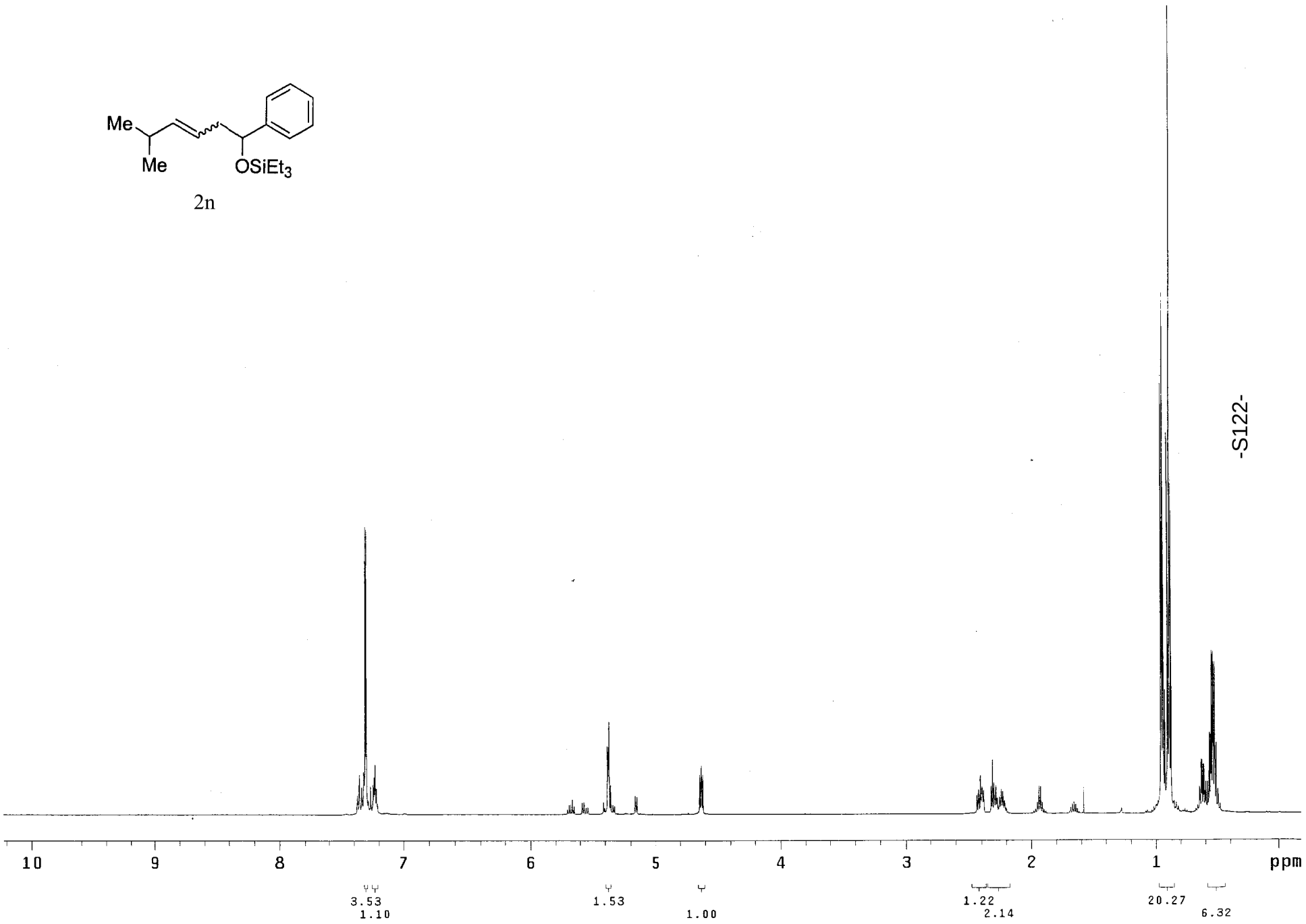
F2 - Processing parameters
SI                32768
SF          100.6127590 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

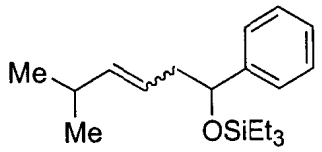
1D NMR plot parameters
CX                24.00 cm
F1P             220.000 ppm
F1             22134.80 Hz
F2P             -10.000 ppm
F2             -1006.13 Hz
PPMCM           9.58333 ppm/cm
HZCM          964.20557 Hz/cm
```



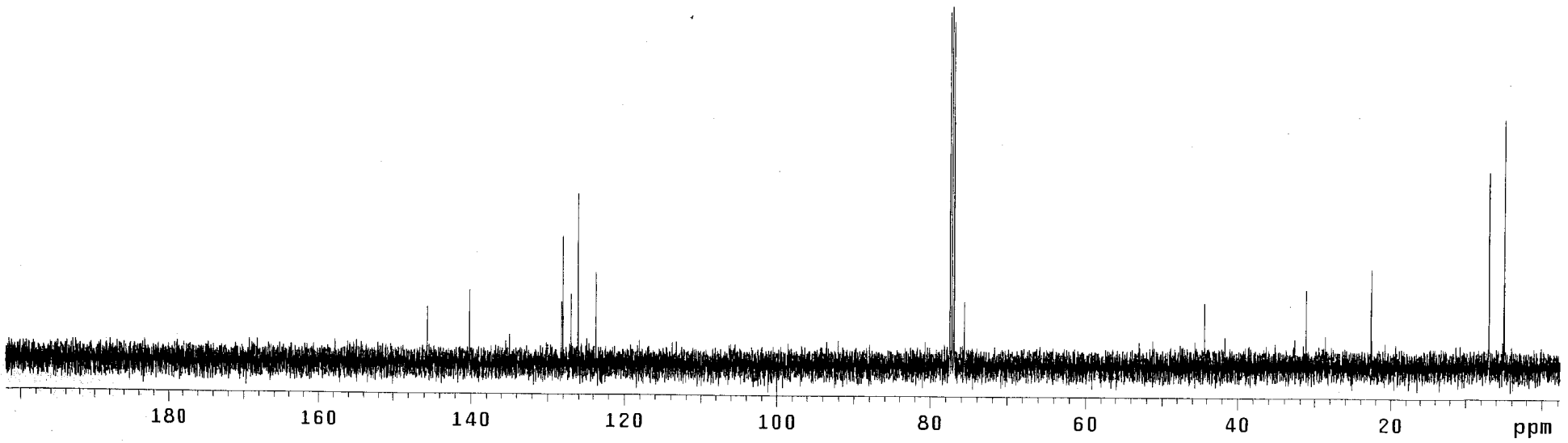
Me
Me
OSiEt3

2n

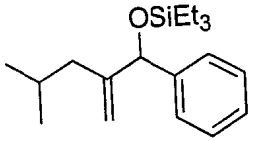

-S122-
10
9
8
7
6
5
4
3
2
1
ppm
3.53
1.10
1.53
1.00
1.22
2.14
20.27
6.32



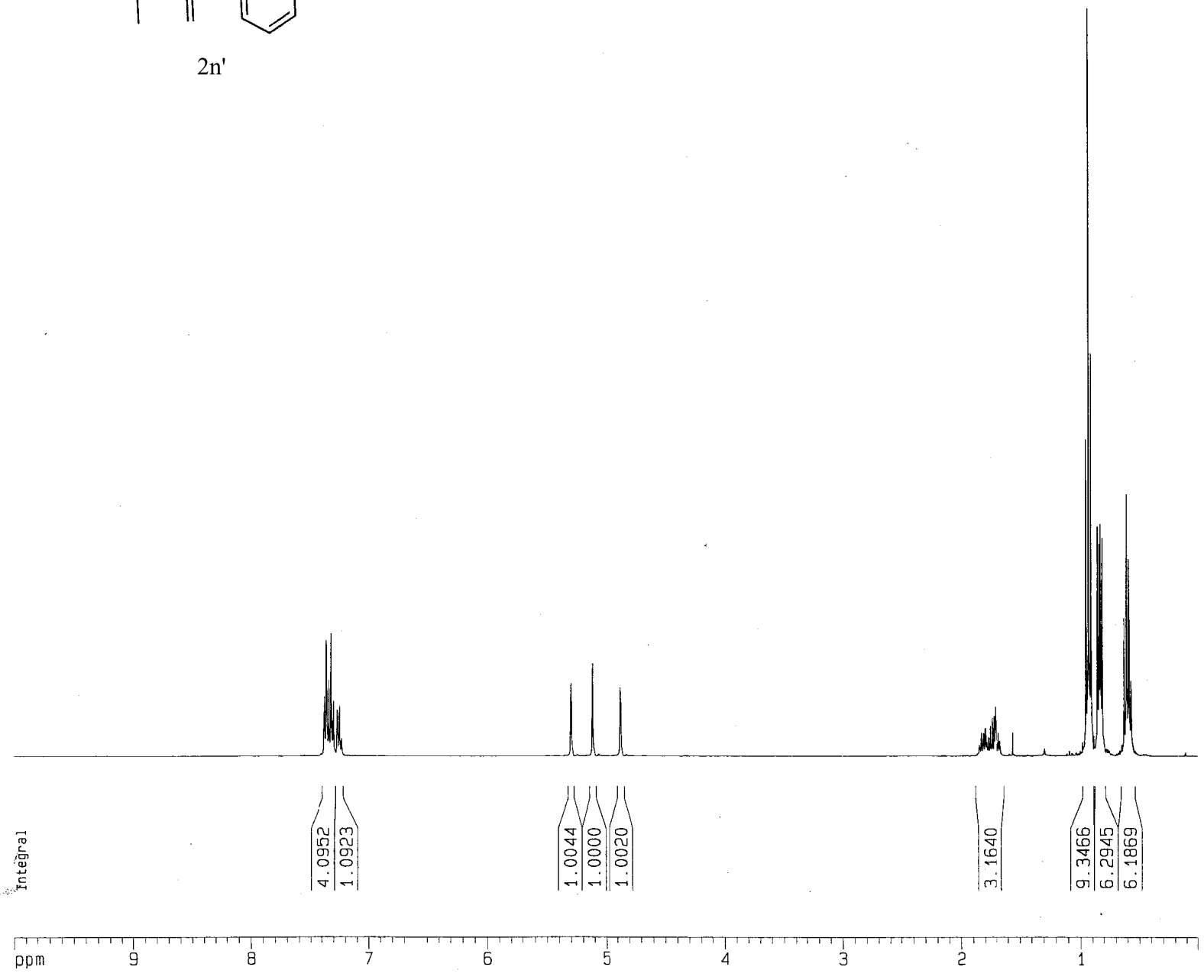
2n



SN050726 allylic alcohol



2n'



Current Data Parameters
NAME SN726-all-H
EXPNO 1
PROCNO 1

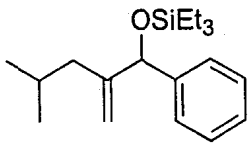
F2 - Acquisition Parameters
Date_ 20050722
Time 21.51
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 32
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

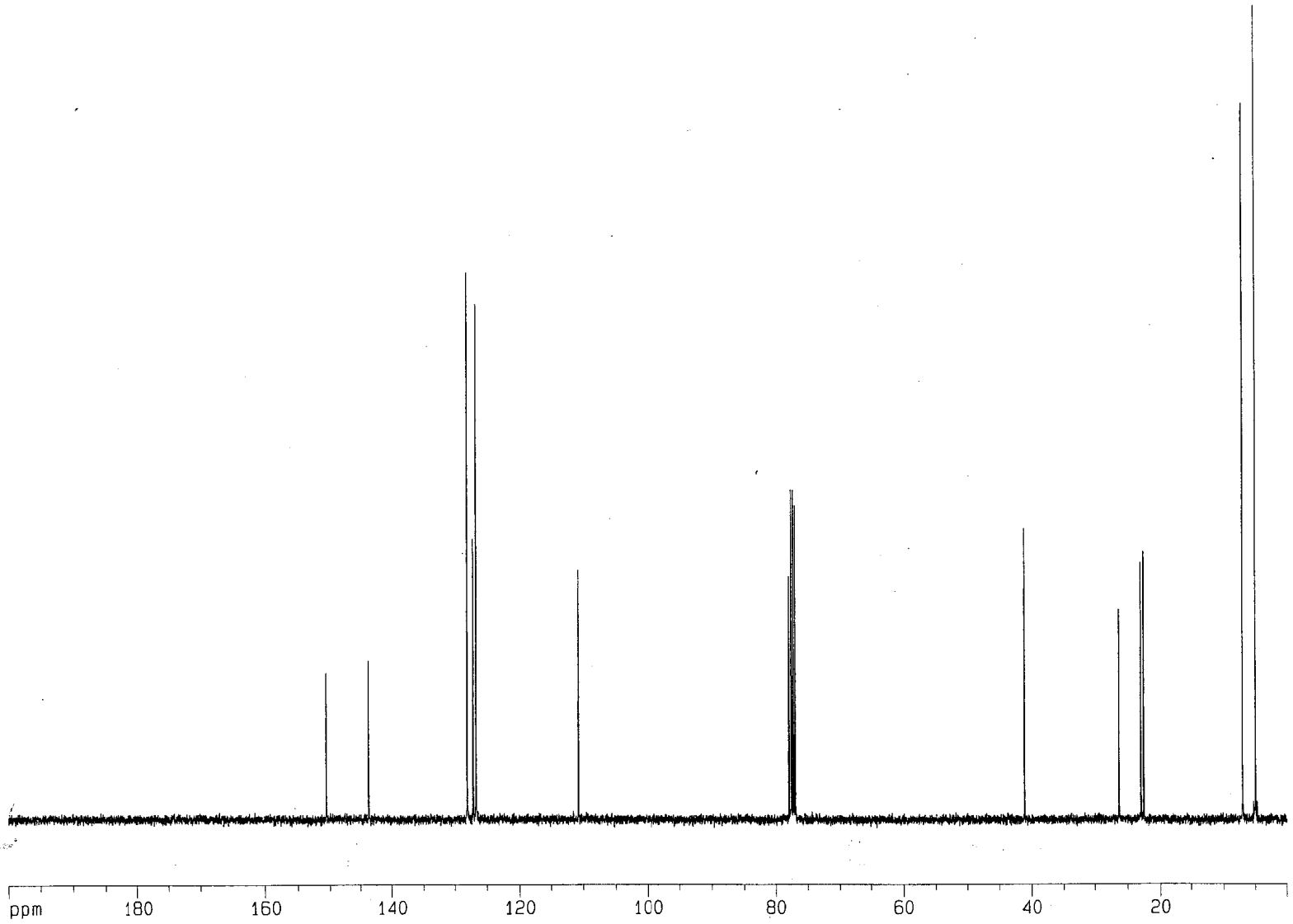
F2 - Processing parameters
SI 32768
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050726 allylic alcohol



2n'



Current Data Parameters
NAME SN726-all-C
EXPNO 1
PROCNO 1

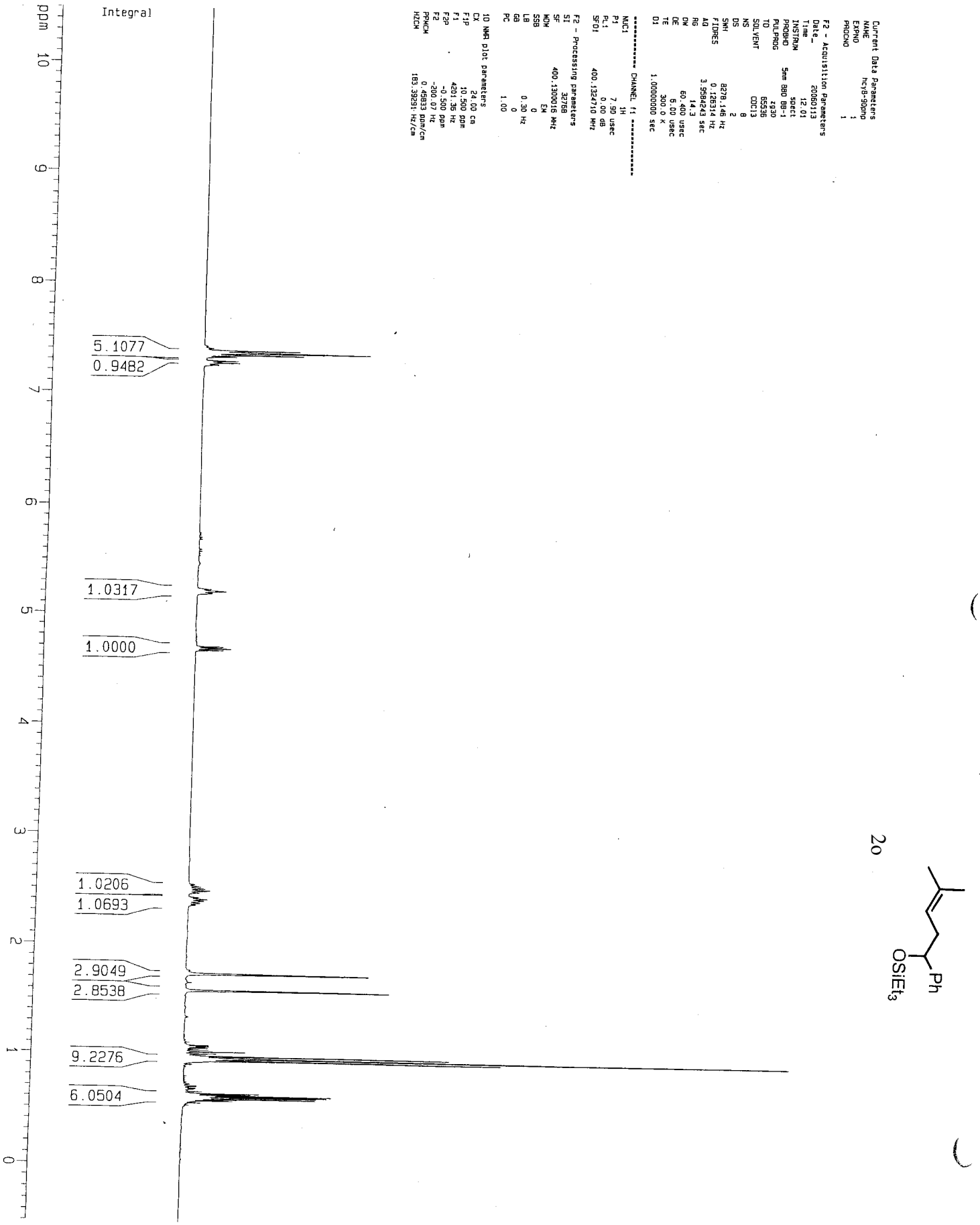
F2 - Acquisition Parameters
Date_ 20050722
Time 21.53
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 159
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

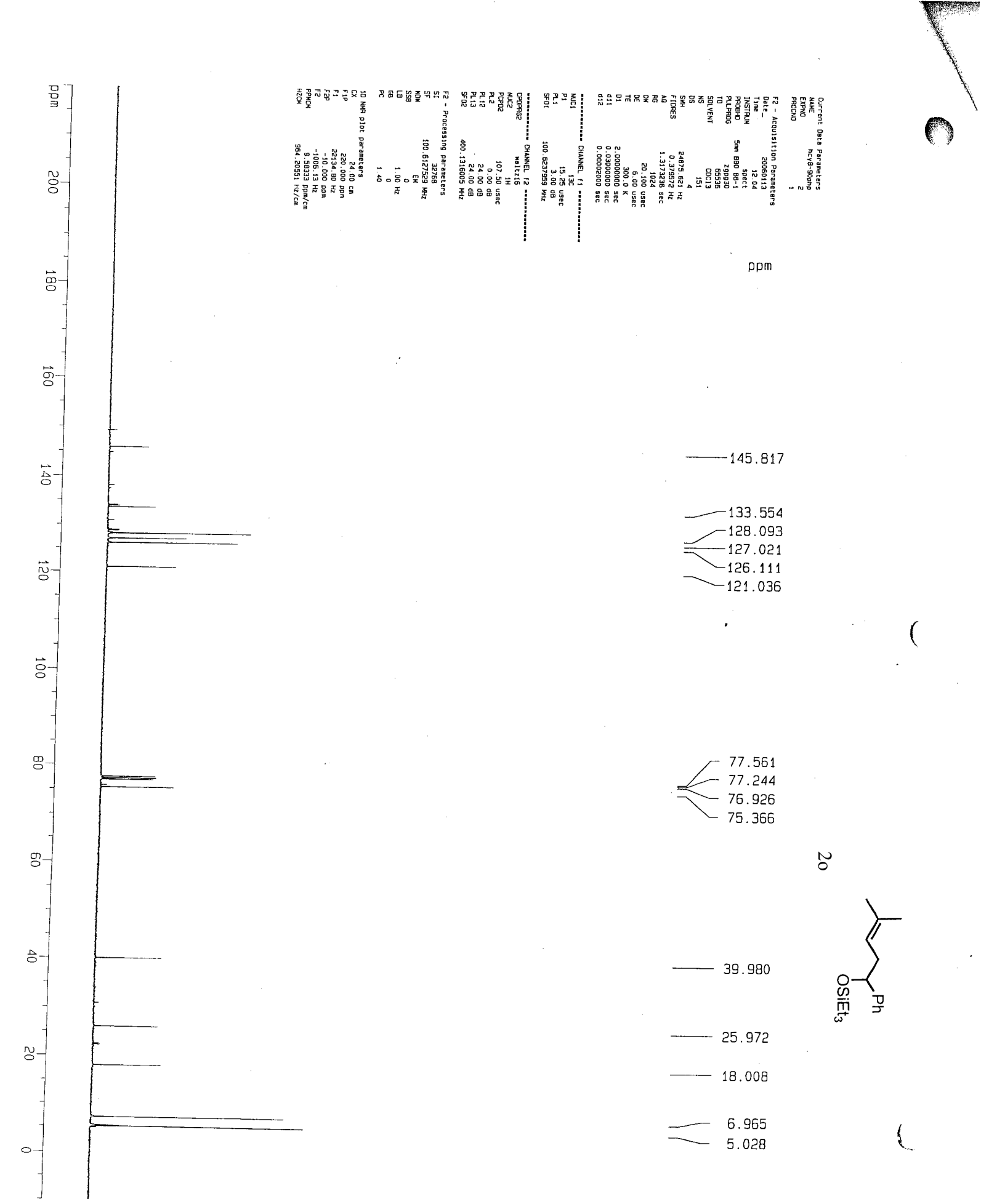
========== CHANNEL f1 ==========
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

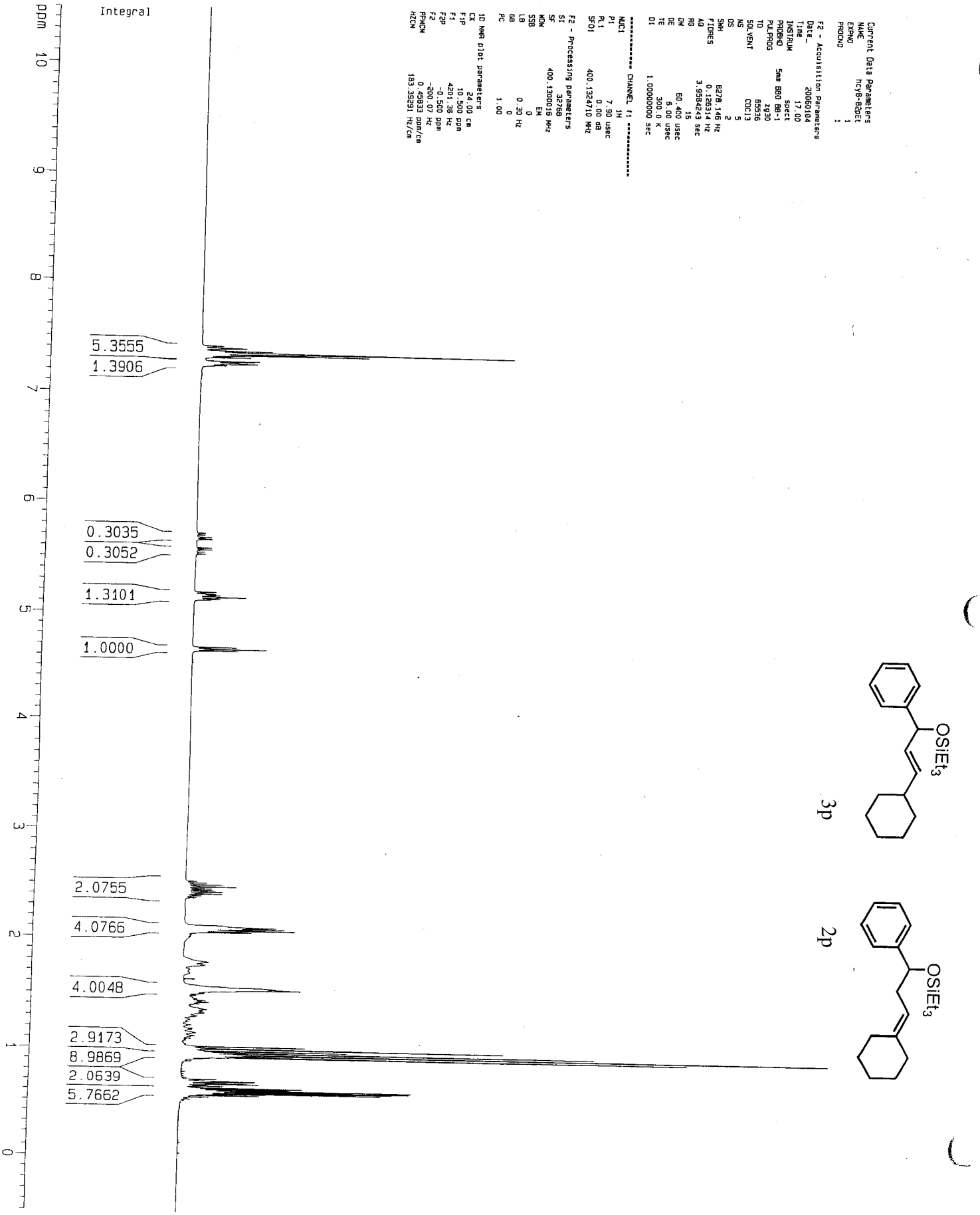
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127499 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm


Integral
ppm
10
9
8
7
6
5
4
3
2
1
0
5.1077
0.9482
1.0317
1.0000
1.0206
1.0693
2.9049
2.8538
9.2276
6.0504
Current Data Parameters
NAME hcy5-90dmp
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060113
Time 12.01
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 14.3
DW 60.400 usec
DE 6.00 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 400.1300016 MHz
WDW EM
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
PPMCM 0.45833 ppm/cm
HZCM 183.39231 Hz/cm
OSiEt3
Ph
20


Current Data Parameters
NAME hcy8-90mp
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060113
Time 12.04
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 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 100.6127529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
145.817
133.554
128.093
127.021
126.111
121.036
77.561
77.244
76.926
75.366
39.980
25.972
18.008
6.965
5.028
20
OSiEt3
Ph


Integral
ppm
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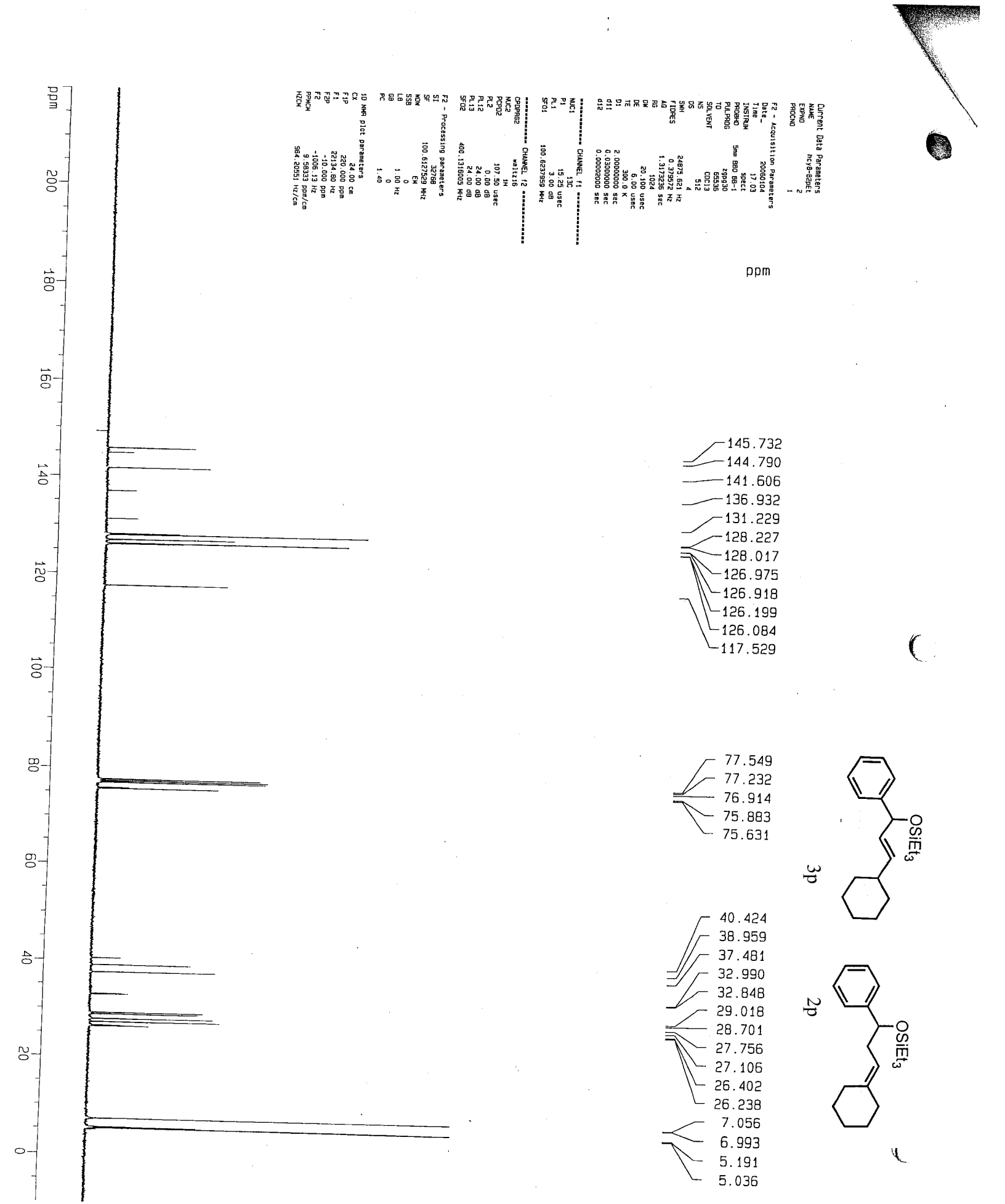
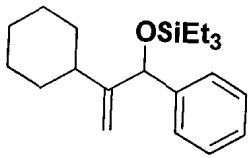
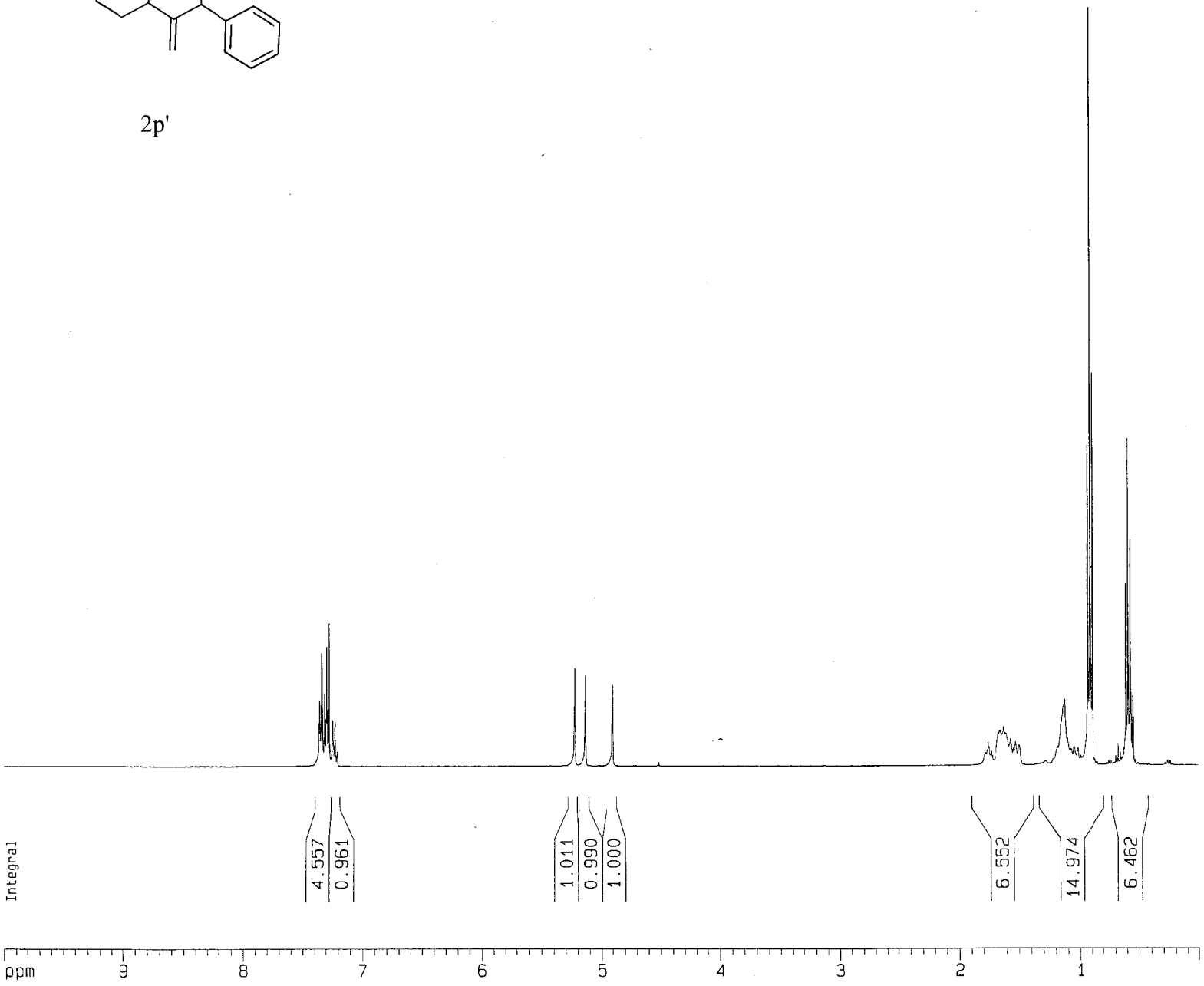
1.3906
0.3035
0.3052
1.3101
1.0000
2.0755
4.0766
4.0048
2.9173
8.9869
2.0639
5.7662
OSiEt3
3p
OSiEt3
2p


ppm
200
180
160
140
120
100
80
60
40
20
0
145.732
144.790
141.606
136.932
131.229
128.227
128.017
126.975
126.918
126.199
126.084
117.529
77.549
77.232
76.914
75.883
75.631
40.424
38.959
37.481
32.990
32.848
29.018
28.701
27.756
27.106
26.402
26.238
7.056
6.993
5.191
5.036
OSiEt3
3p
OSiEt3
2p

SN061114 allylic



2p'



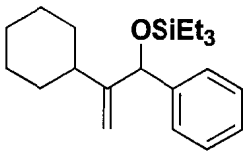
Current Data Parameters
NAME SN1114A-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060321
Time 16.57
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400
DE 6.00
TE 299.1
D1 1.00000000
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

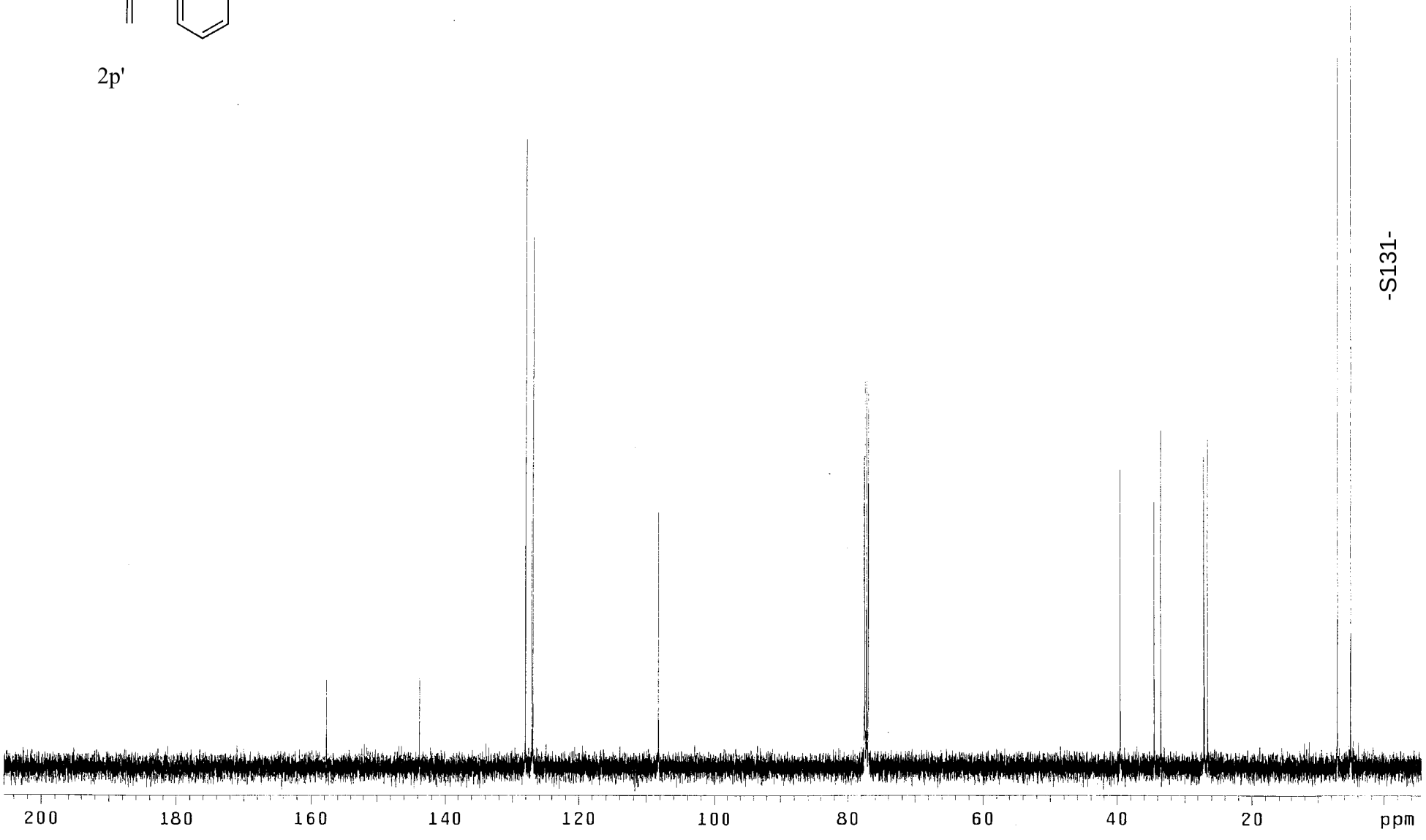
======== CHANNEL f1 ========
NUC1 1H
P1 9.88 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

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

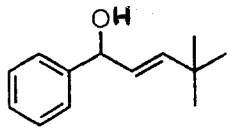
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm



2p'



tBu-vinyl, TES deprotection



3q (TES group deprotected)

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Current Data Parameters
NAME          hcy6-91ppe
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060117
Time               11.34
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                    3
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                143.7
DW               60.400 usec
DE                6.00 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          400.1300018 MHz
WDW                  EM
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
PPMCM             0.45633 ppm/cm
HZCM            183.39291 Hz/cm
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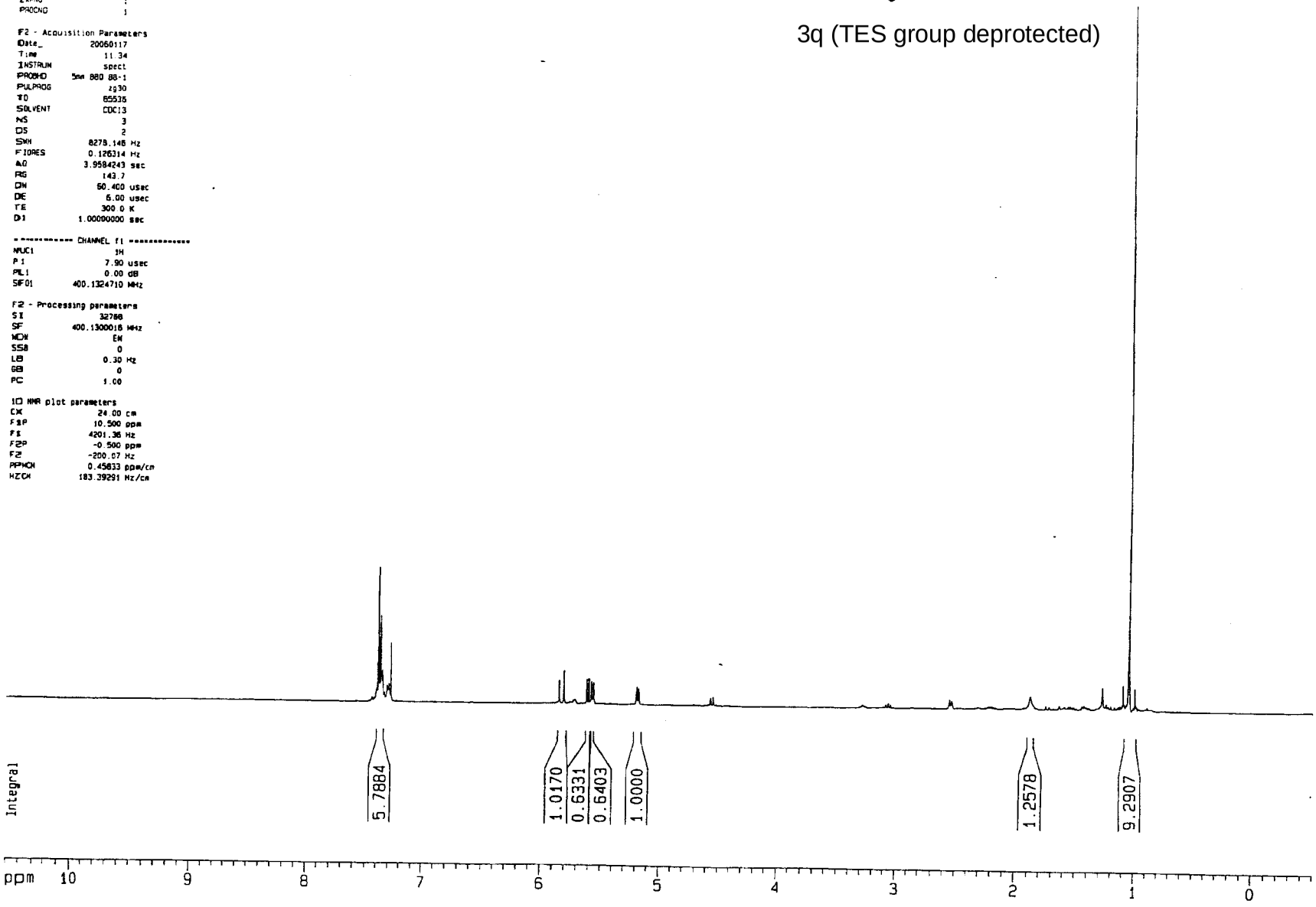
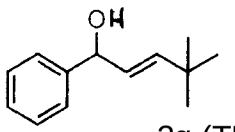
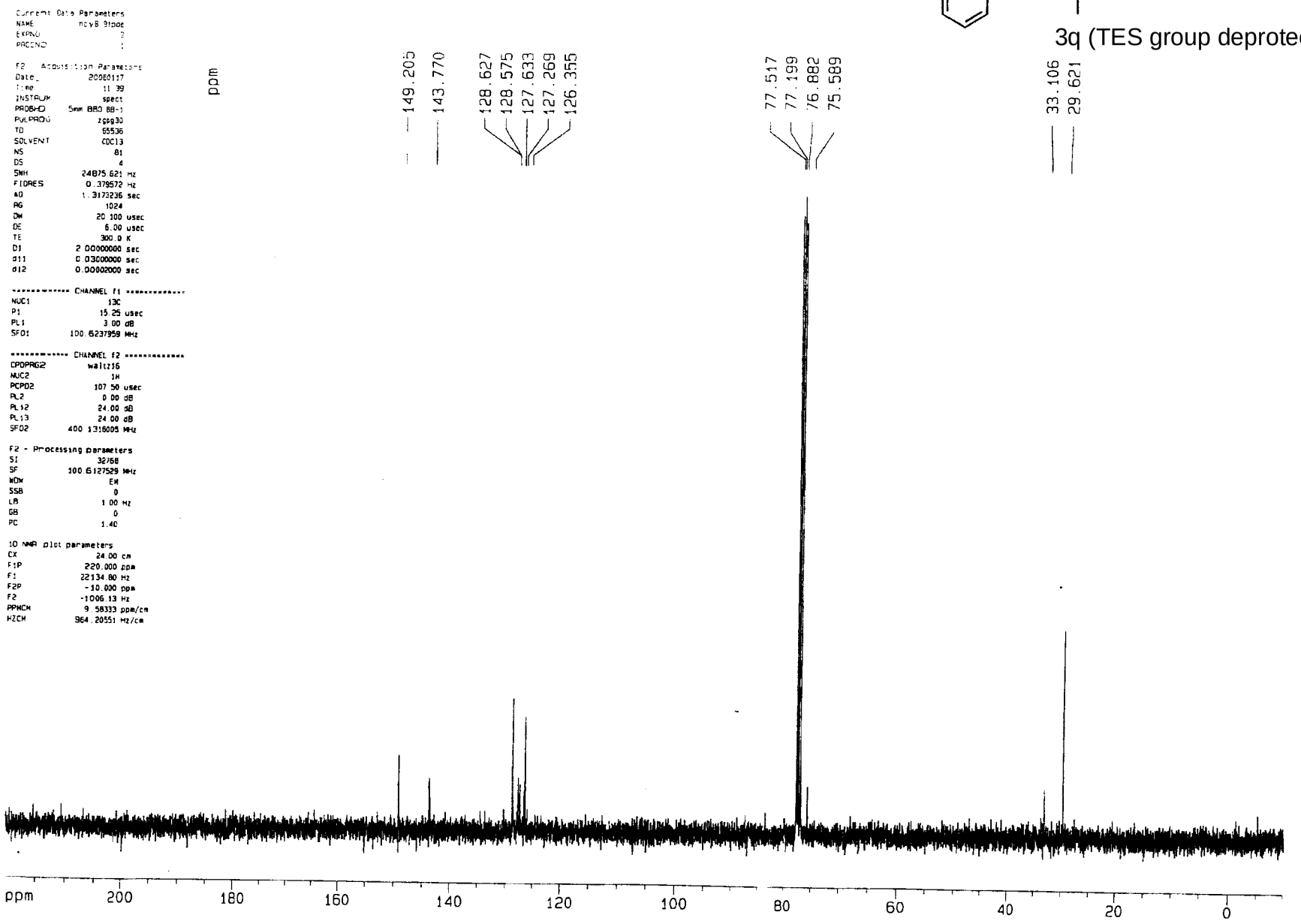


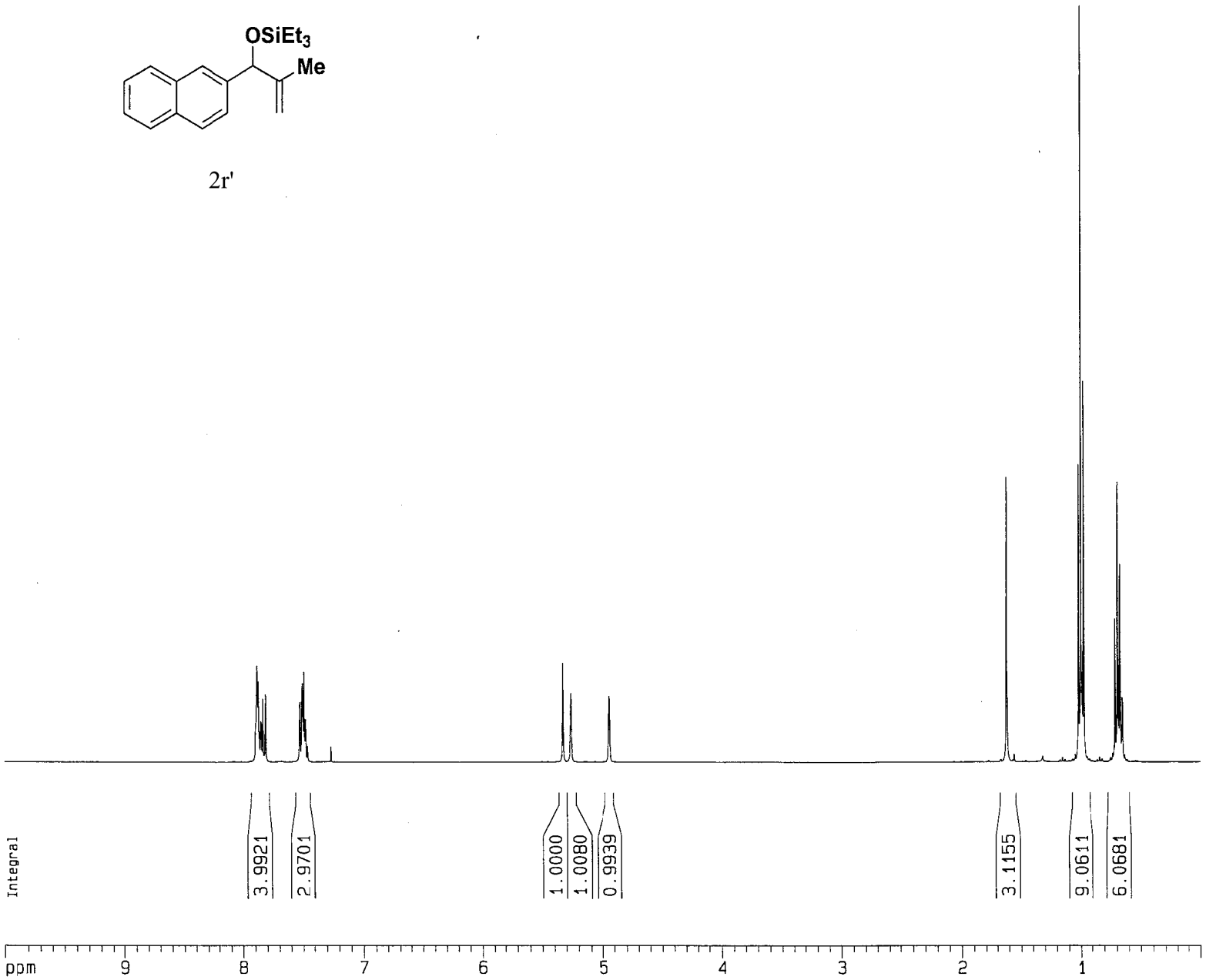


3q (TES group deprotected)



SN050835 allylic

2r'



Current Data Parameters
NAME SN835-all-H
EXPNO 1
PROCNO 1

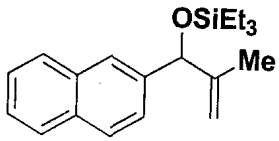
F2 - Acquisition Parameters
Date_ 20051014
Time 12.08
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400
DE 6.00
TE 294.8
D1 1.00000000
MCREST 0.00000000
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.88 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

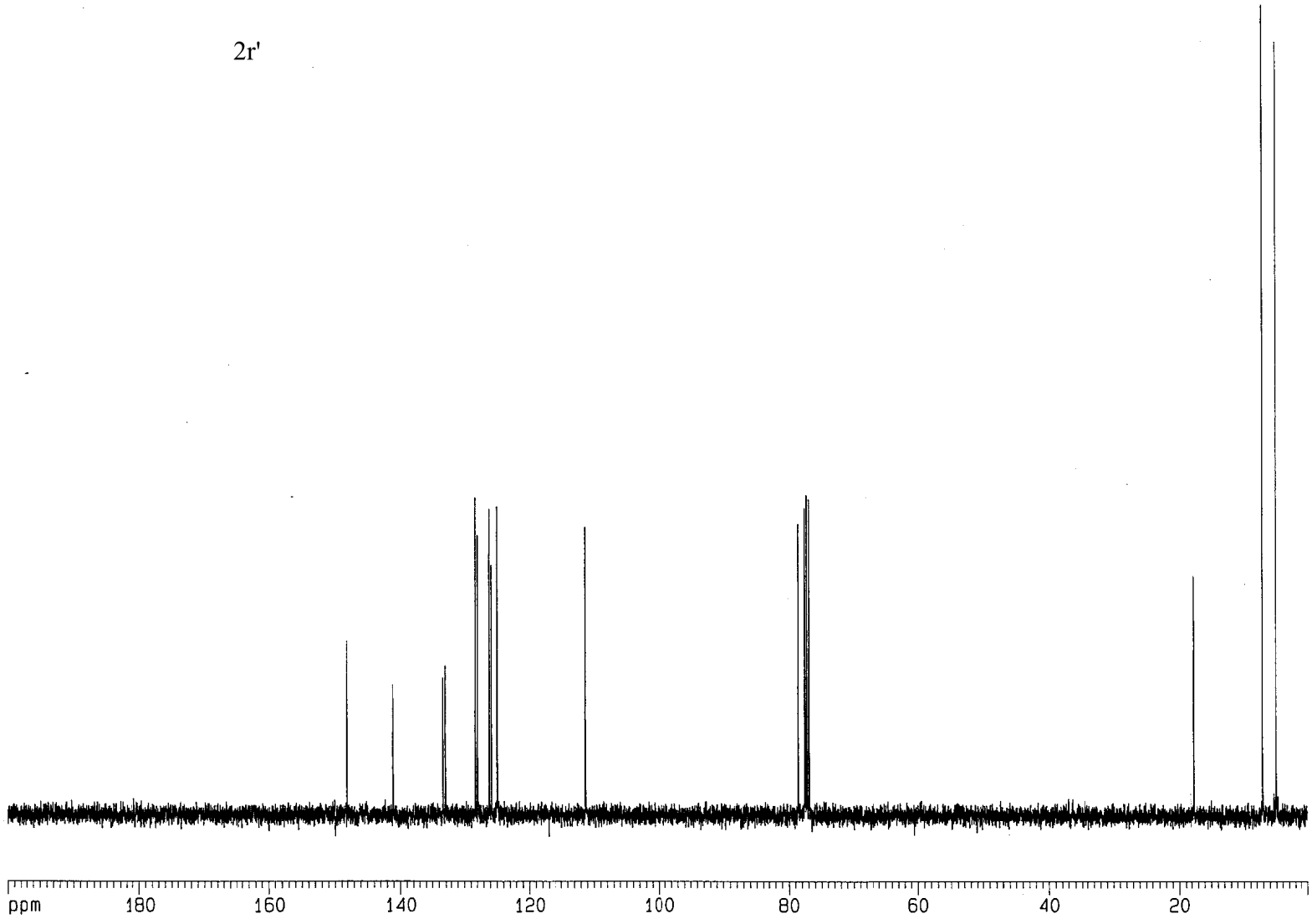
F2 - Processing parameters
SI 32768
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050835 allylic



2r'



Current Data Parameters
NAME SN835-all-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051014
Time 12.16
INSTRUM spect
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 193
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654756 sec
RG 2896.3
DW 20.850 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 3.00 dB
SFO1 100.6228298 MHz

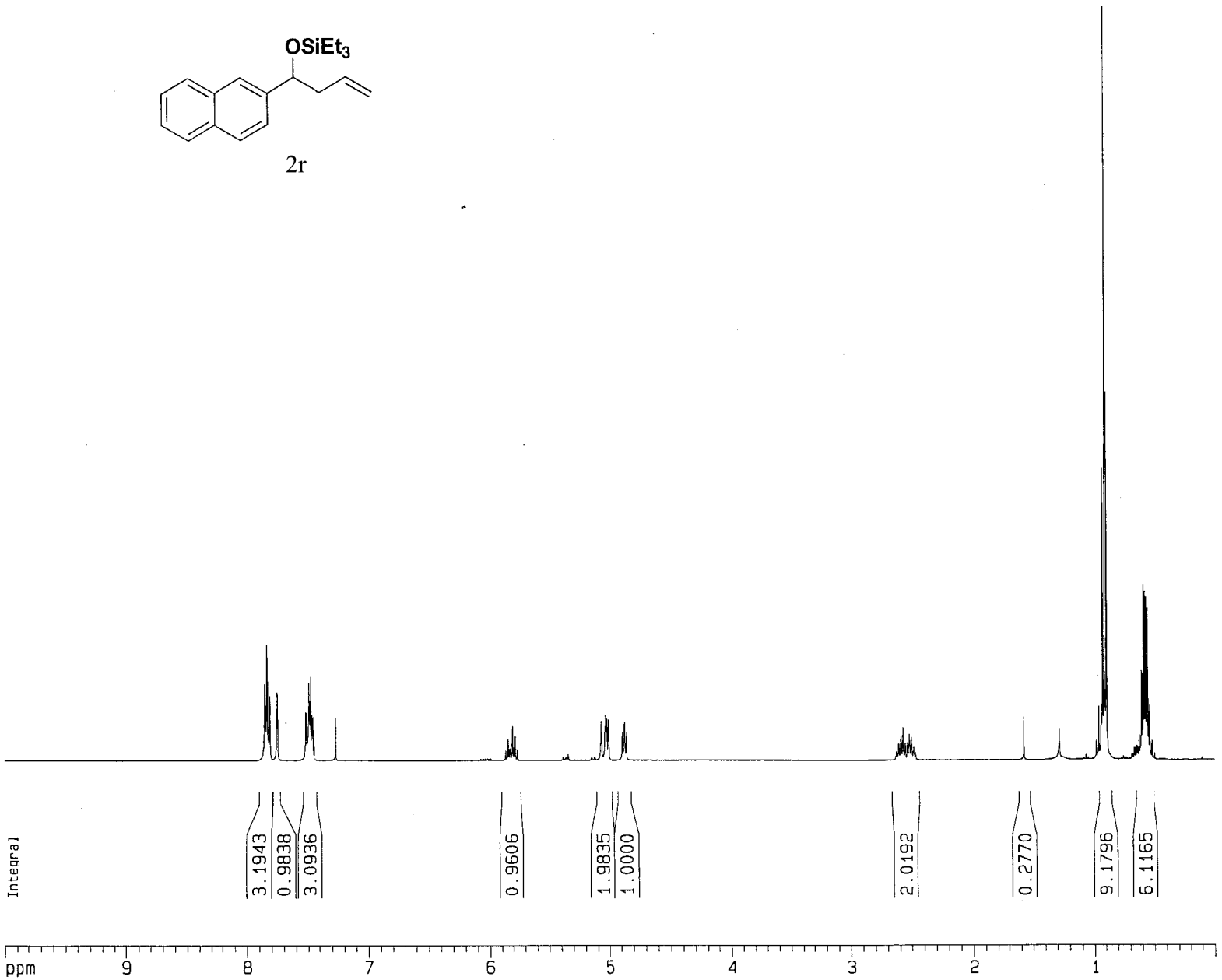
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 88.01 usec
PL2 3.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127535 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12756 Hz/cm

SN050835 homoallylic

2r



Current Data Parameters

NAME	SN835-ho-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20051014
Time	12.27
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	143.7
DW	60.400
DE	6.00
TE	294.8
D1	1.00000000
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	9.88 usec
PL1	3.00 dB
SFO1	400.1324710 MHz

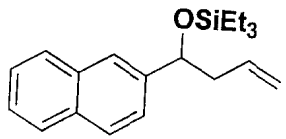
F2 - Processing parameters

SI	32768
SF	400.1300054 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

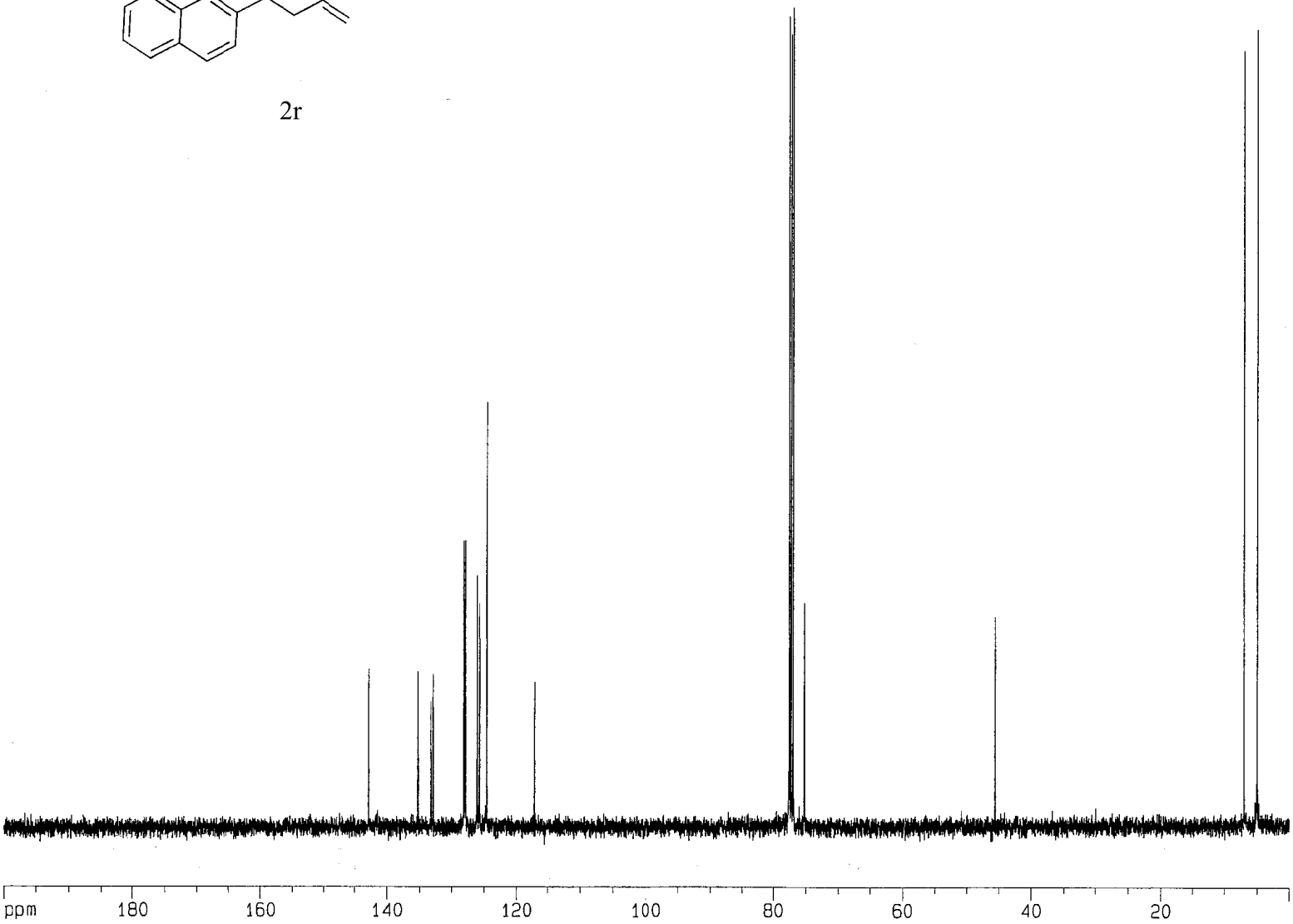
1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm

SN050835 homoallylic



2r



Current Data Parameters

NAME	SN835-ho-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20051015
Time	12.12
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	302
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	2048
DW	19.900 usec
DE	6.00 u
TE	300.0 K
D1	2.00000000 s
d11	0.03000000 s
d12	0.00002000 s

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

NUC1	13C
P1	15.25 usec
PL1	3.00 dB
SFO1	100.6237959 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SFO2	400.1316005 MHz

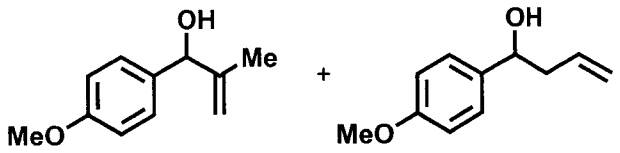
F2 - Processing parameters

SI	32768
SF	100.6127507 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

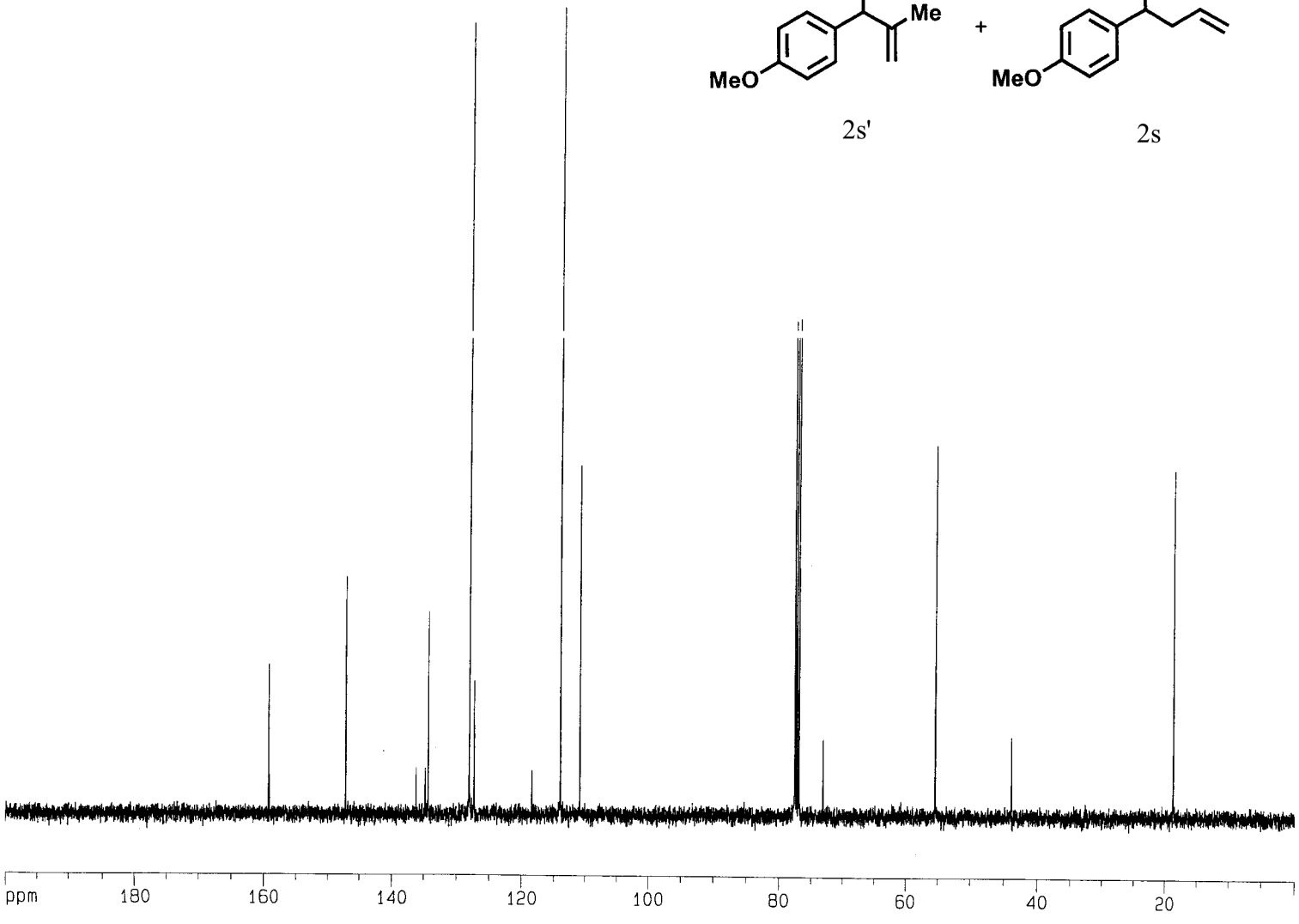
1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	10.00000 ppm/cm
HZCM	1006.12738 Hz/cm

SN061027 TBAF



2s'　　2s




```
Current Data Parameters
NAME        SN1027-TBAF-C
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20060131
Time              19.23
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   98
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG               3649.1
DW               19.900 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

NUC1                13C
P1               15.25 usec
PL1               3.00 dB
SFO1         100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2          107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127553 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40

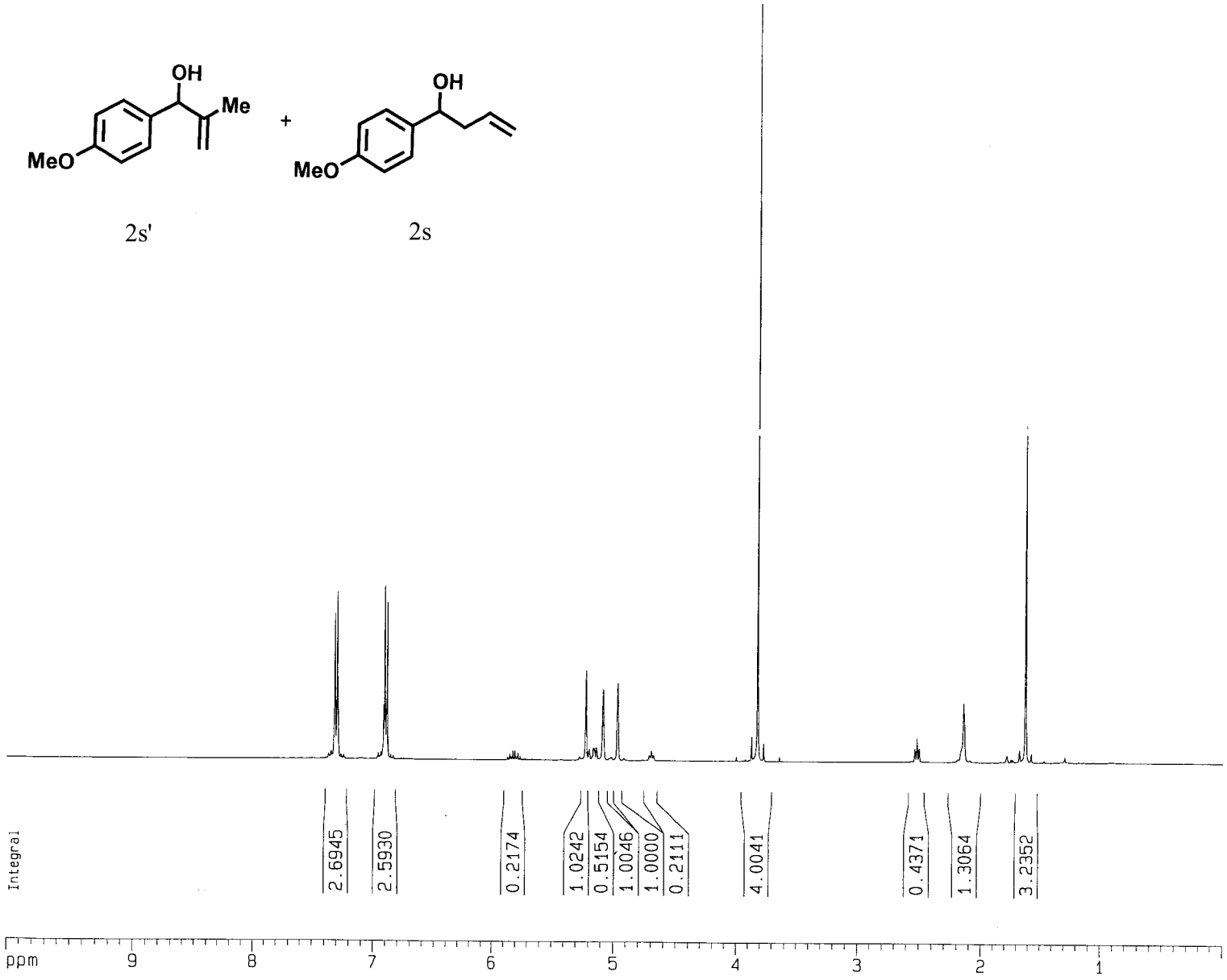
1D NMR plot parameters
CX               20.00 cm
F1P             200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         10.00000 ppm/cm
HZCM        1006.12756 Hz/cm
```

SN061027 TBAF

2s'

+

2s




```
Current Data Parameters
NAME        SN1027-TBAF-H
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060131
Time                19.16
INSTRUM             spect
PROBHD     5mm BBO BB-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                    57
DW              60.400 usec
DE               6.00 usec
TE               300.0 K
D1            1.00000000 sec

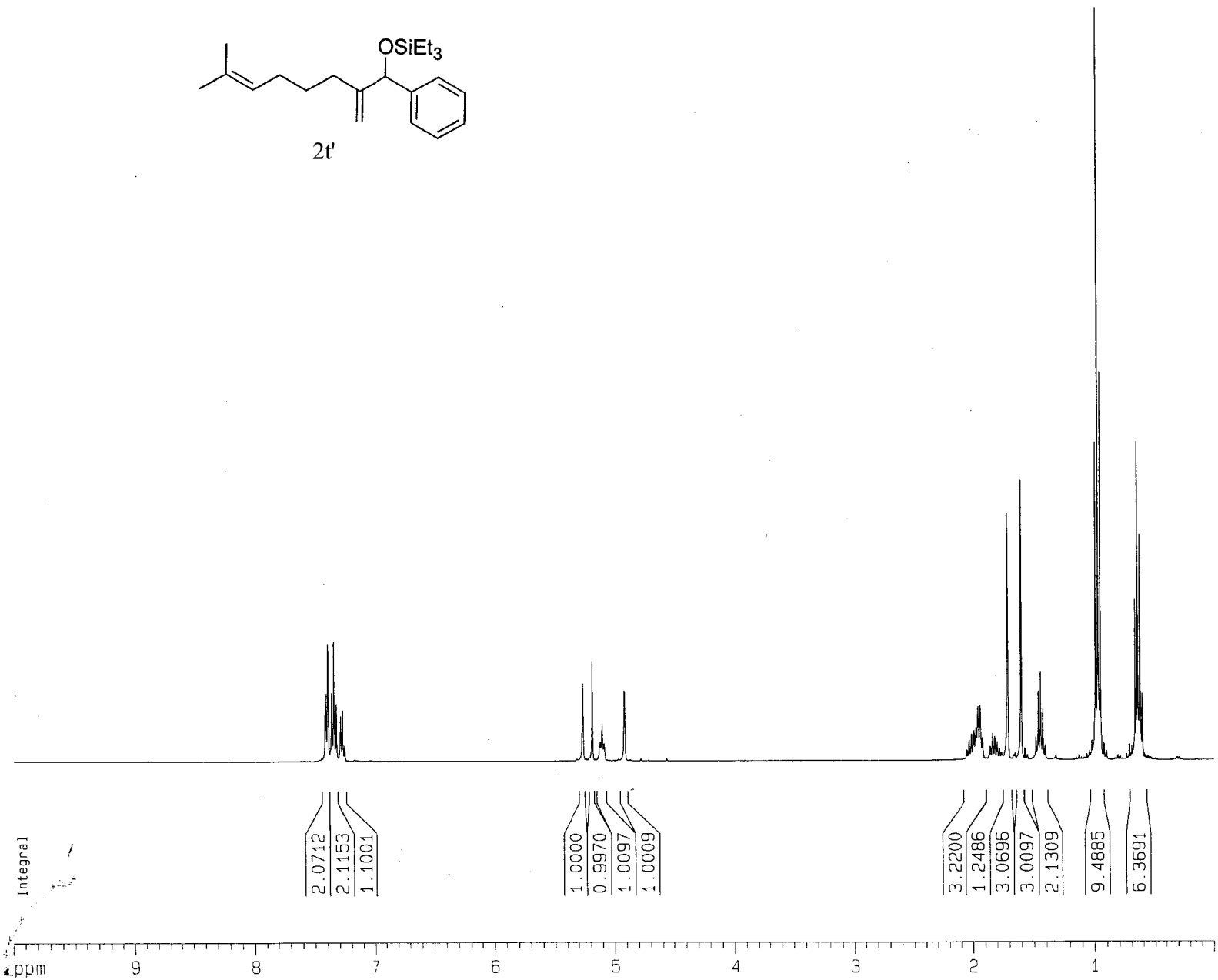
========== CHANNEL f1 ========
NUC1                   1H
P1                7.90 usec
PL1               0.00 dB
SFO1        400.1324710 MHz

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

1D NMR plot parameters
CX               20.00 cm
F1P             10.000 ppm
F1              4001.30 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         0.50000 ppm/cm
HZCM        200.06500 Hz/cm
```

SN050672 a

2t'



Current Data Parameters

NAME	SN672-a-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050625
Time	16.55
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	4
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	32
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	9.50 usec
PL1	2.00 dB
SFO1	400.1324710 MHz

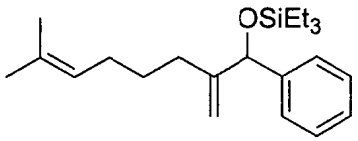
F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

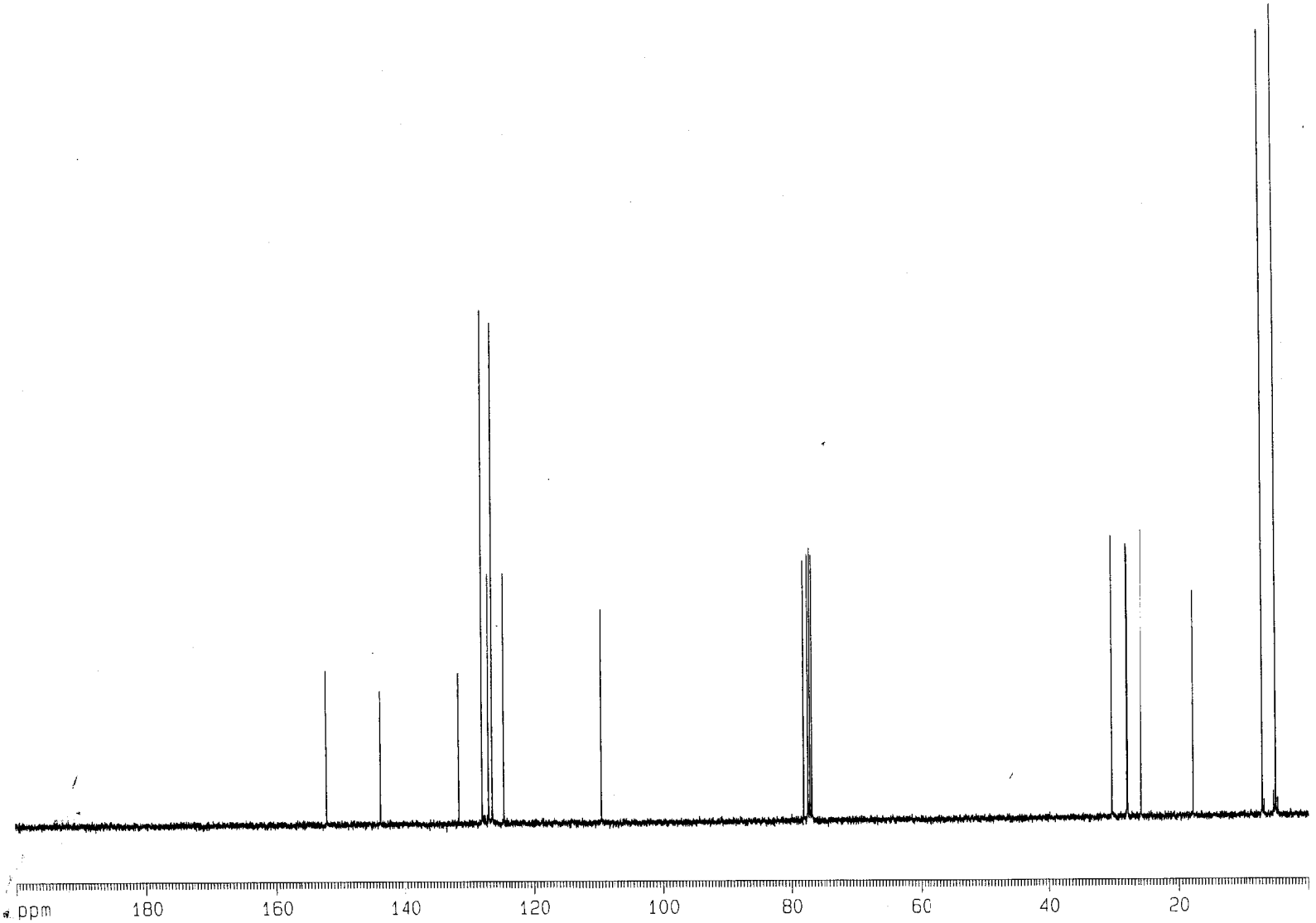
1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm

SN050672 a



2t'



Current Data Parameters
NAME SN672-a-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050625
Time 18.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 760
DS 4
SWH 24330.900 Hz
FIDRES 0.371260 Hz
AQ 1.3468148 sec
RG 1824.6
DW 20.550 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 8.50 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

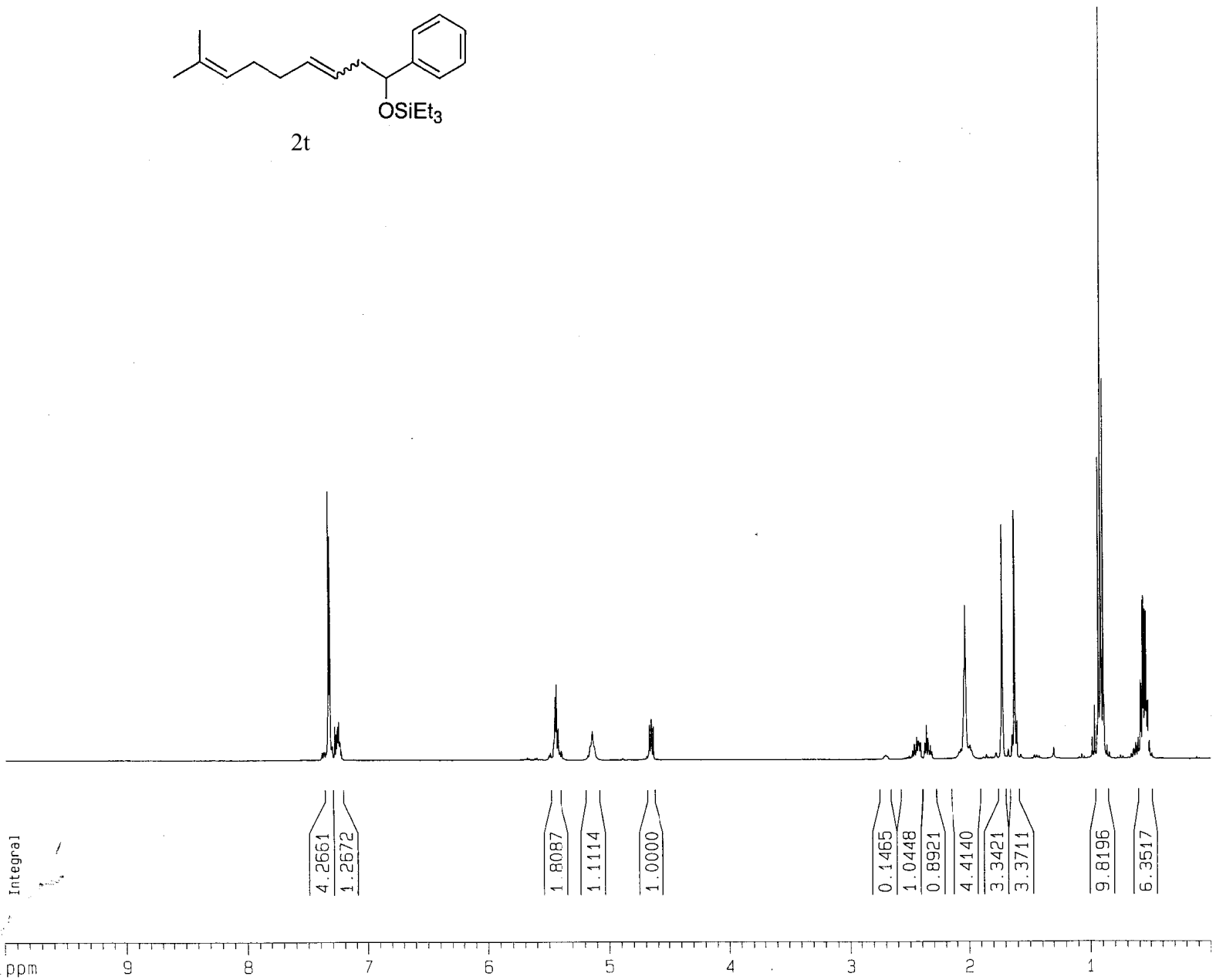
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127518 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12756 Hz/cm

SN050672-b

2t



Current Data Parameters
NAME SN672-b-H
EXPNO 1
PROCNO 1

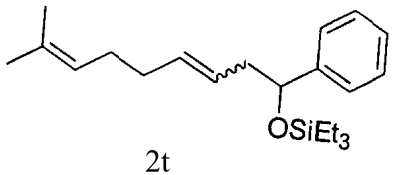
F2 - Acquisition Parameters
Date_ 20050625
Time 18.50
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 54
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 9.50 usec
PL1 2.00 dB
SFO1 400.1324710 MHz

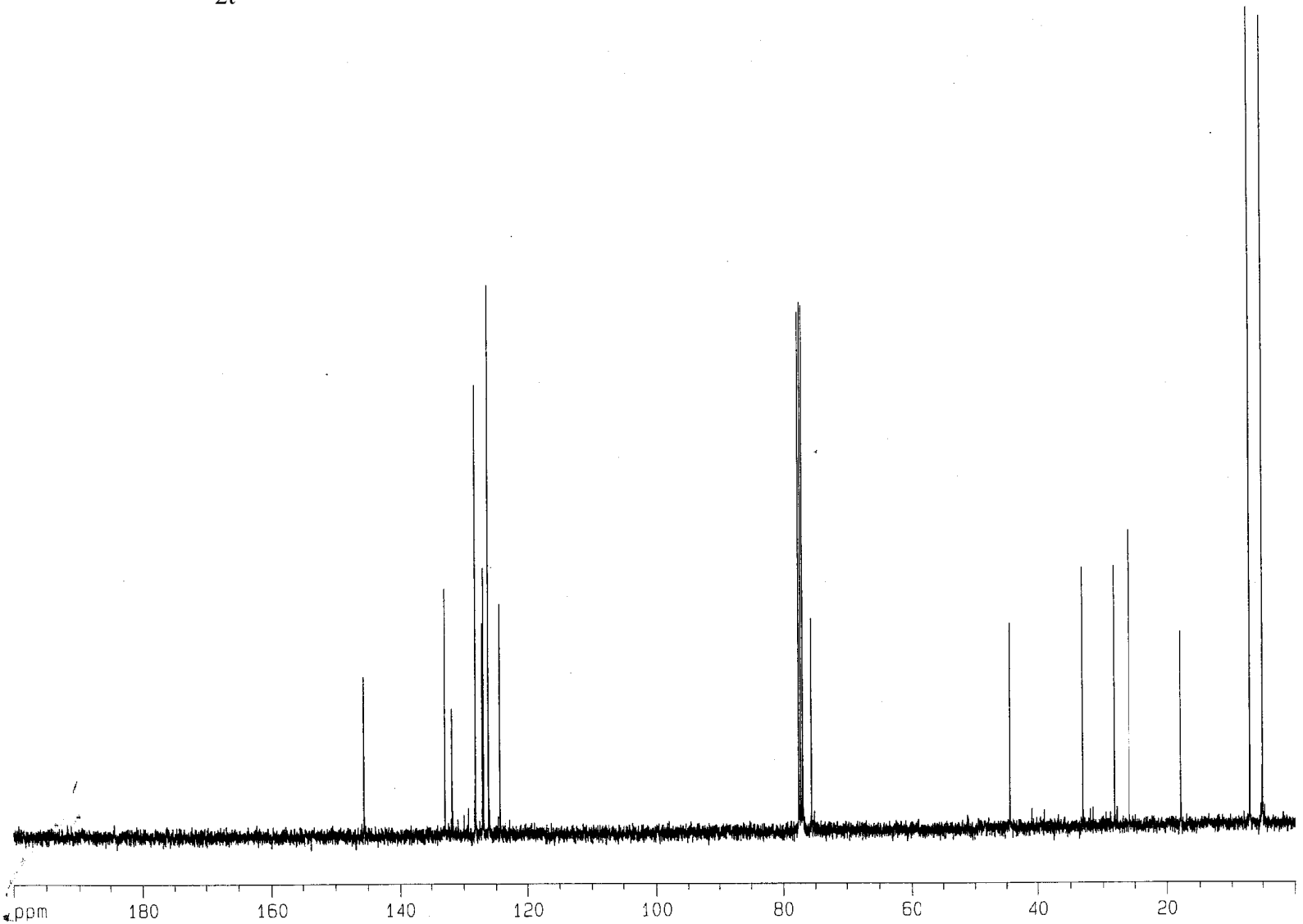
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm

SN050672-b



2t



Current Data Parameters
NAME SN672-b-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050625
Time 18.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 746
DS 4
SWH 24330.900 Hz
FIDRES 0.371260 Hz
AQ 1.3468148 sec
RG 1149.4
DW 20.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

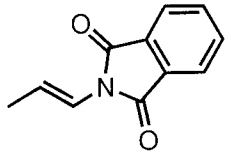
========== CHANNEL f1 ==========
NUC1 13C
P1 8.50 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

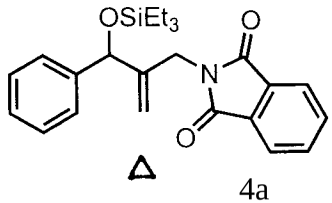
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127503 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm

CyPPh2, f1





4a

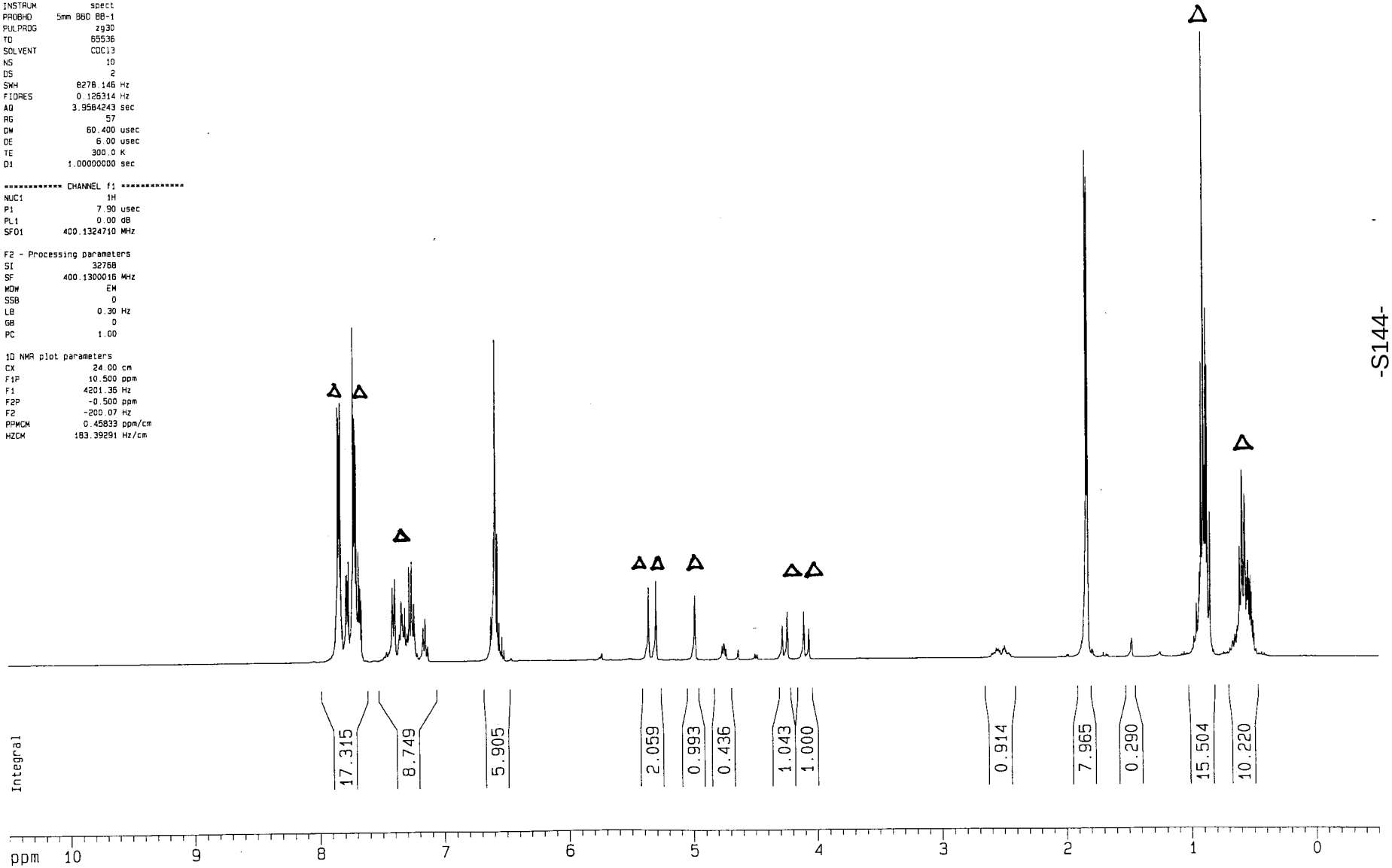
```
Current Data Parameters
NAME        hcyB-66pCy-f1
EXPNO                  1
PROCNO                 1

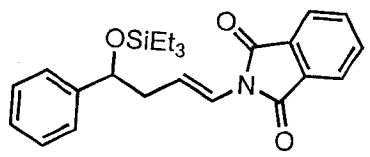
F2 - Acquisition Parameters
Date_           20051201
Time               16.12
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   10
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   57
DW              60.400 usec
DE               6.00 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          400.1300016 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                24.00 cm
F1P              10.500 ppm
F1              4201.35 Hz
F2P              -0.500 ppm
F2              -200.07 Hz
PPMCM            0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```





4a'

Current Data Parameters

NAME	hcy-8-51-pf1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20051108
Time	16.12
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	6
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

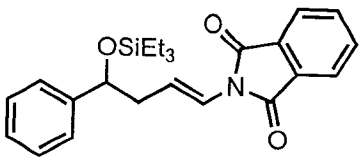
NUC1	1H
P1	7.90 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300016 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

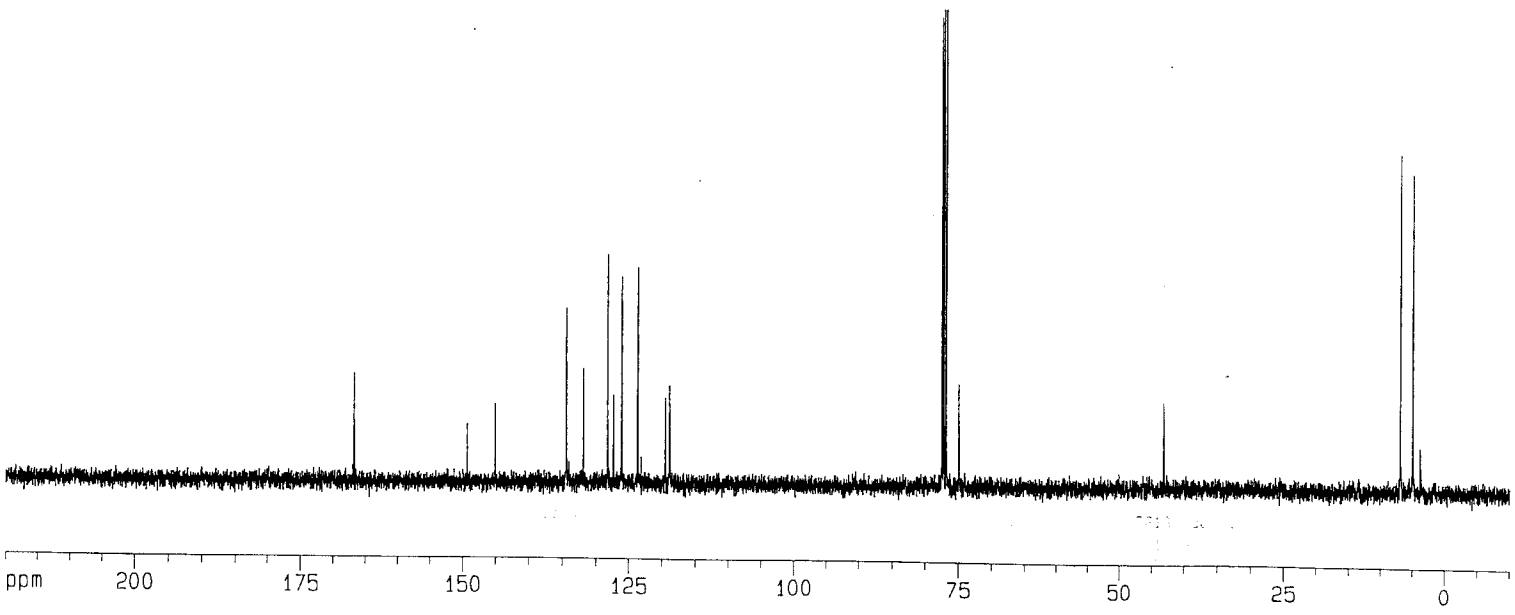
1D NMR plot parameters

CX	20.00 cm
F1P	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.55000 ppm/cm
HZCM	220.07150 Hz/cm



4a'

166.687
149.211
145.040
134.465
131.857
128.244
127.311
126.054
123.670
119.484
118.811
77.544
77.226
76.908
74.988
43.094
6.961
4.962



Current Data Parameters

NAME	hcy-8-51-pf1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20051108
Time	16.19
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	103
DS	4
SWH	24875.621 Hz
FIDRES	0.379572 Hz
AQ	1.3173236 sec
RG	1024
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	15.25 usec
PL1	3.00 dB
SFO1	100.6237959 MHz

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

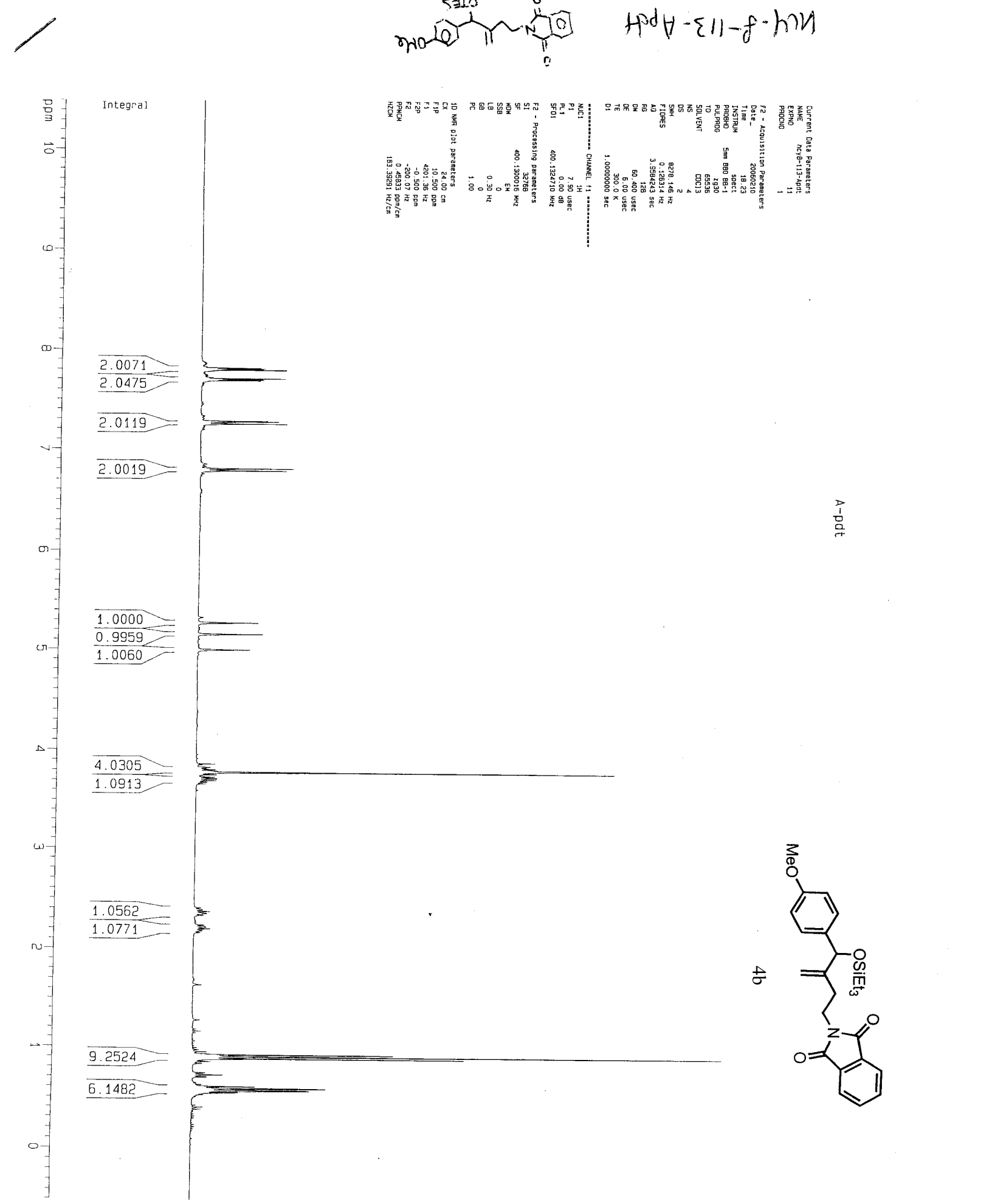
CPDPRG2	waltz16
NUC2	1H
PCPD2	107.50 usec
PL2	0.00 dB
PL12	24.00 dB
PL13	24.00 dB
SFO2	400.1316005 MHz

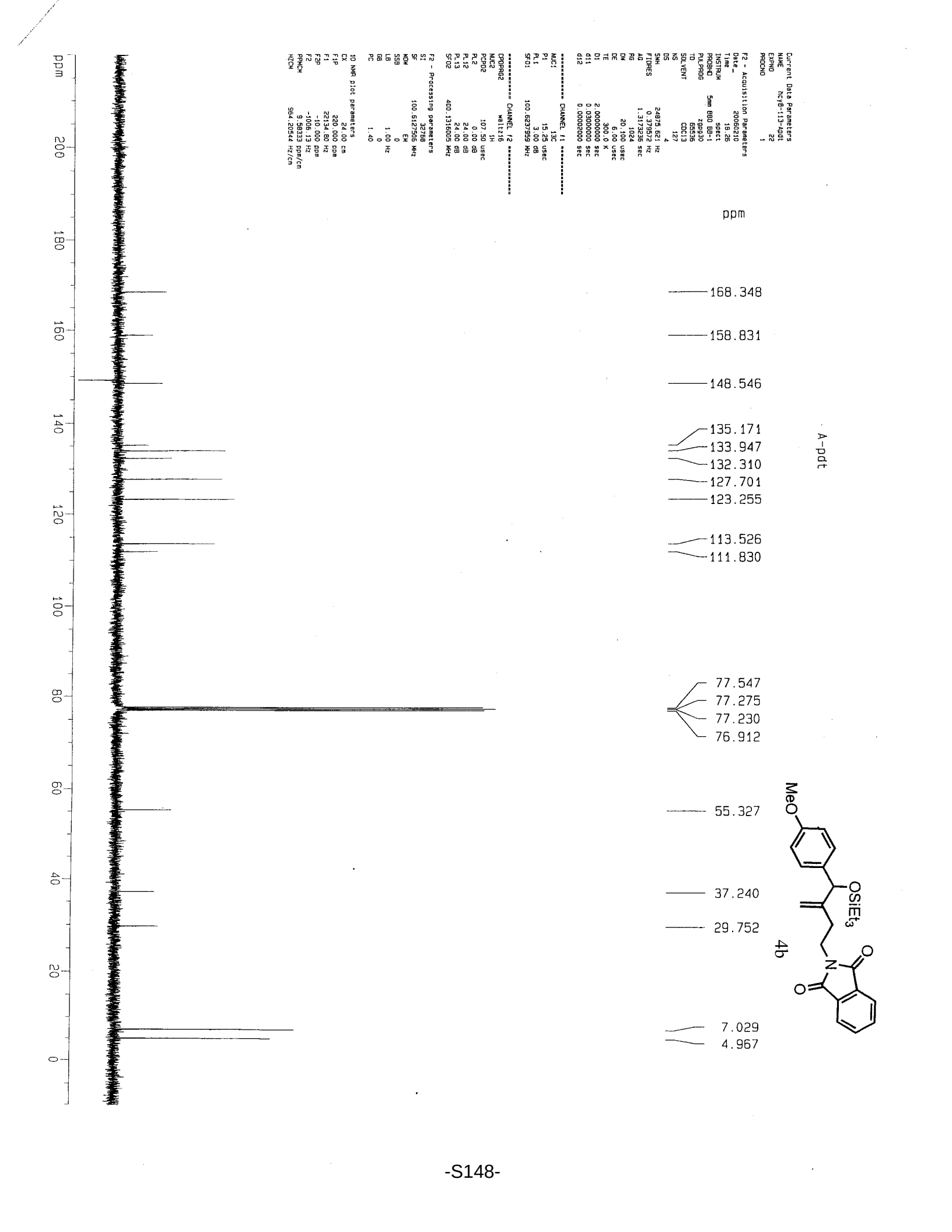
F2 - Processing parameters

SI	32768
SF	100.6127529 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

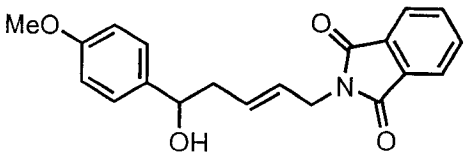
1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.80 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	11.50000 ppm/cm
HZCM	1157.04663 Hz/cm


Integral
ppm
2.0071
2.0475
2.0119
2.0019
1.0000
0.9959
1.0060
4.0305
1.0913
1.0562
1.0771
9.2524
6.1482
A-pdt
4b
MeO
OSiEt3
N
O
O


Current Data Parameters
NAME hcy8-113-Apdt
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060210
Time_ 15.26
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 127
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 1024
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 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127505 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.680 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20544 Hz/cm
ppm
168.348
158.831
148.546
135.171
133.947
132.310
127.701
123.255
113.526
111.830
77.547
77.275
77.230
76.912
55.327
37.240
29.752
7.029
4.967
A-pdt
MeO
OSiEt3
4b
O
N
O

H pdt TES deprotection



4b' (TES group deprotected)

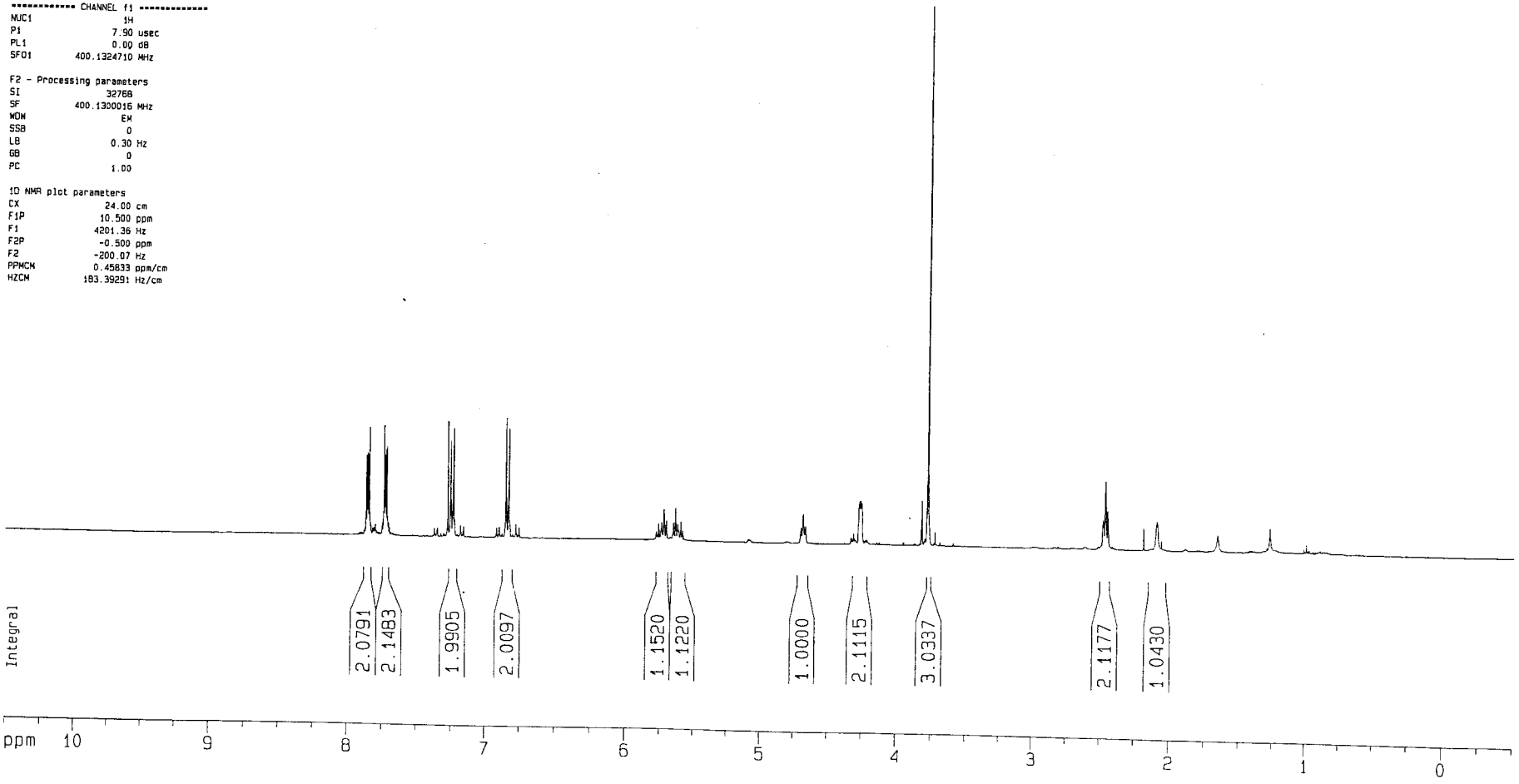
```
Current Data Parameters
NAME      hcy8-97de-Hpdt
EXPNO                  1
PROCNO                 1

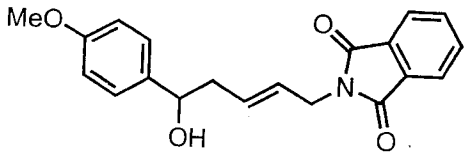
F2 - Acquisition Parameters
Date_           20060123
Time               15.38
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                  181
DW               60.400 usec
DE                6.00 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          400.1300016 MHz
WDW                   EM
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
PPMCM             0.45833 ppm/cm
HZCM            183.39291 Hz/cm
```





4b' (TES group deprotected)

```
Current Data Parameters
NAME      hcy8-97de-Hpdt
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060123
Time               15.41
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  179
DS                    4
SWH           24875.621 Hz
FIDRES         0.379572 Hz
AQ             1.3173236 sec
RG                 1024
DW               20.100 usec
DE                6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

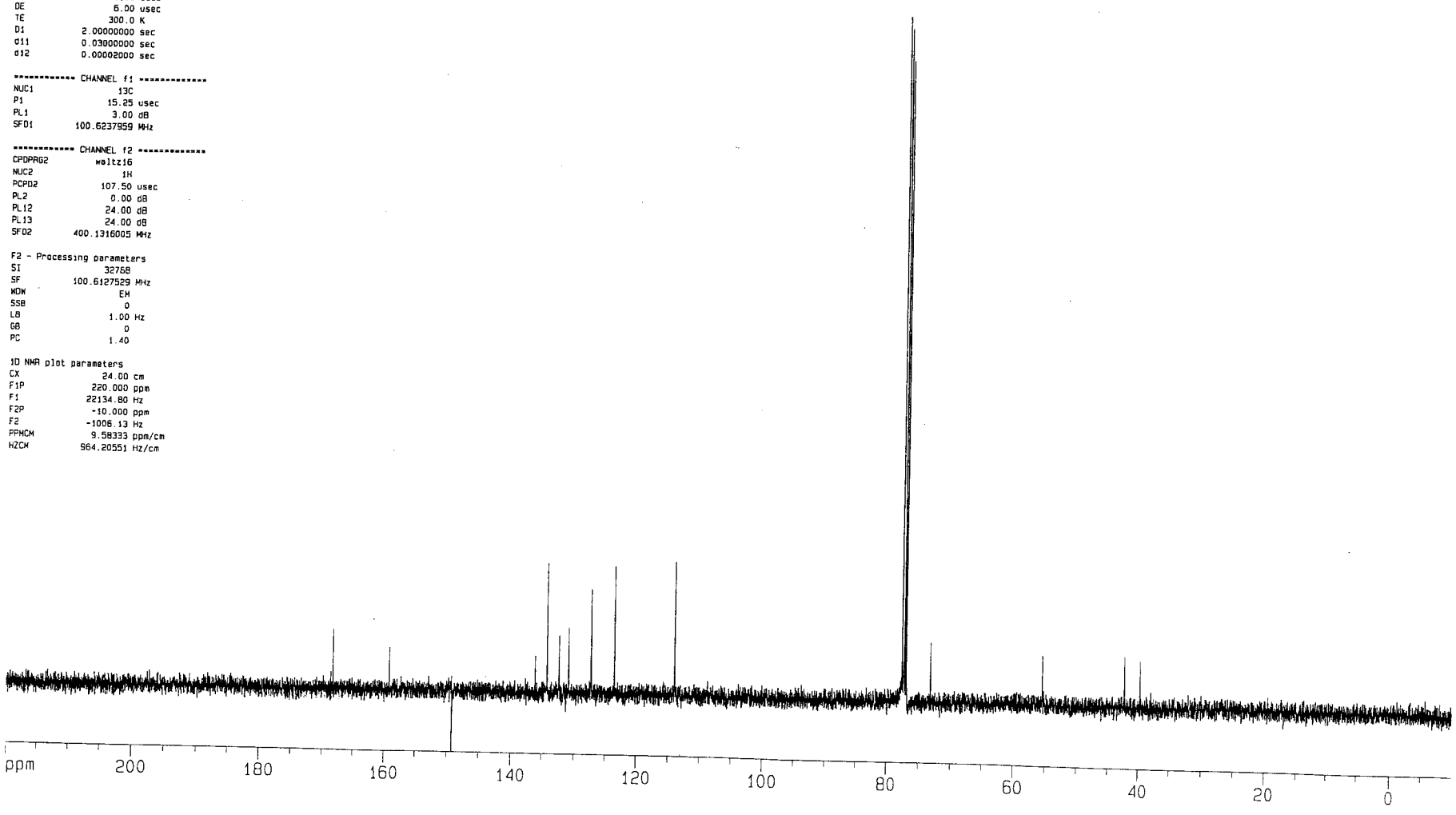
========== CHANNEL f1 ==========
NUC1                13C
P1                15.25 usec
PL1                3.00 dB
SFO1         100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2          waltz16
NUC2                1H
PCPD2           107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2         400.1316005 MHz

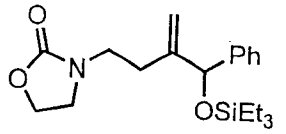
F2 - Processing parameters
SI                 32768
SF          100.6127529 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                24.00 cm
F1P              220.000 ppm
F1              22134.80 Hz
F2P              -10.000 ppm
F2              -1006.13 Hz
PPMCM            9.58333 ppm/cm
HZCM           964.20551 Hz/cm
```

ppm 168.173 159.126 136.046 134.137 132.282 130.787 127.192 127.139 123.459 113.885 77.523 77.410 77.205 76.888 73.124 55.410 42.328 39.724



A-pdt



4c

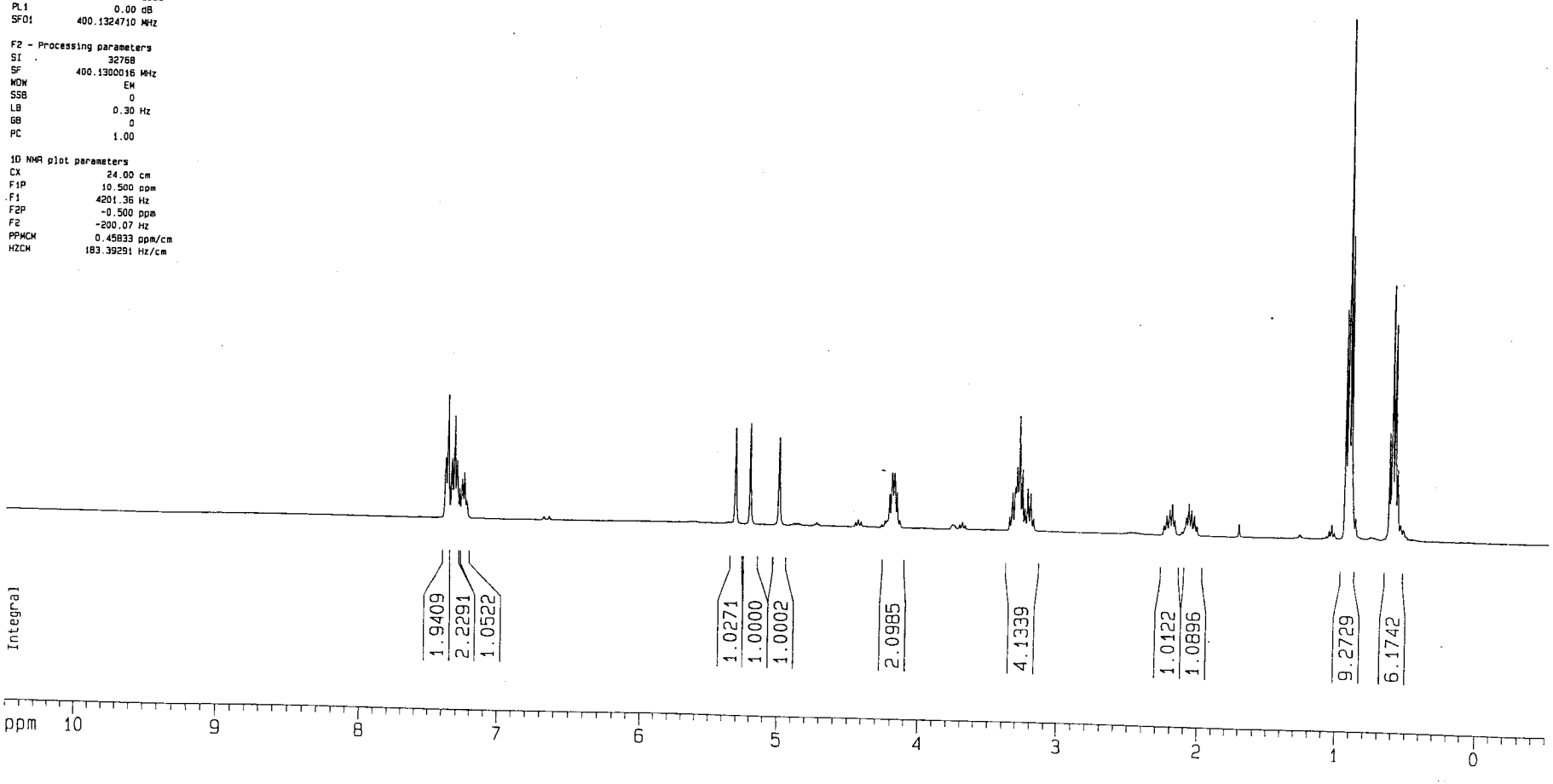
```
Current Data Parameters
NAME         hcy8-78cyf1
EXPNO                 1
PROCNO                1

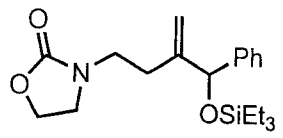
F2 - Acquisition Parameters
Date_          20051219
Time              13.21
INSTRUM           spect
PROBHD     5mm BBO BB-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                    5
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 90.5
DW               60.400 usec
DE                6.00 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          400.1300016 MHz
WDW                  EM
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
PPMCM          0.45833 ppm/cm
HZCM          183.39291 Hz/cm
```





4c

A-pdt

```
Current Data Parameters
NAME          hcy8-78cyf1
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_          20051219
Time              13.25
INSTRUM           spect
PROBHD     5mm BBO 88-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  101
DS                    4
SWH          24875.621 Hz
FIDRES        0.379572 Hz
AQ            1.3173236 sec
RG                 1024
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               15.25 usec
PL1               3.00 dB
SFO1        100.6237959 MHz

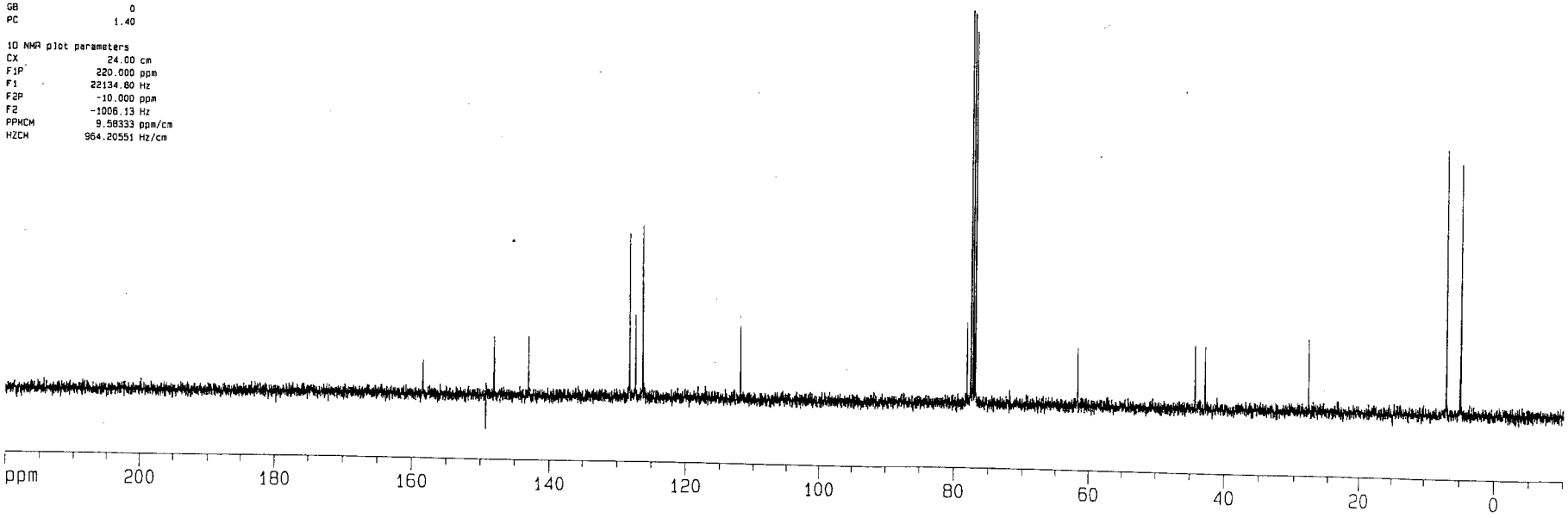
------------ CHANNEL f2 -------------
CPDPRG2         waltz16
NUC2                1H
PCPD2           107.50 usec
PL2              0.00 dB
PL12            24.00 dB
PL13            24.00 dB
SFO2        400.1316005 MHz

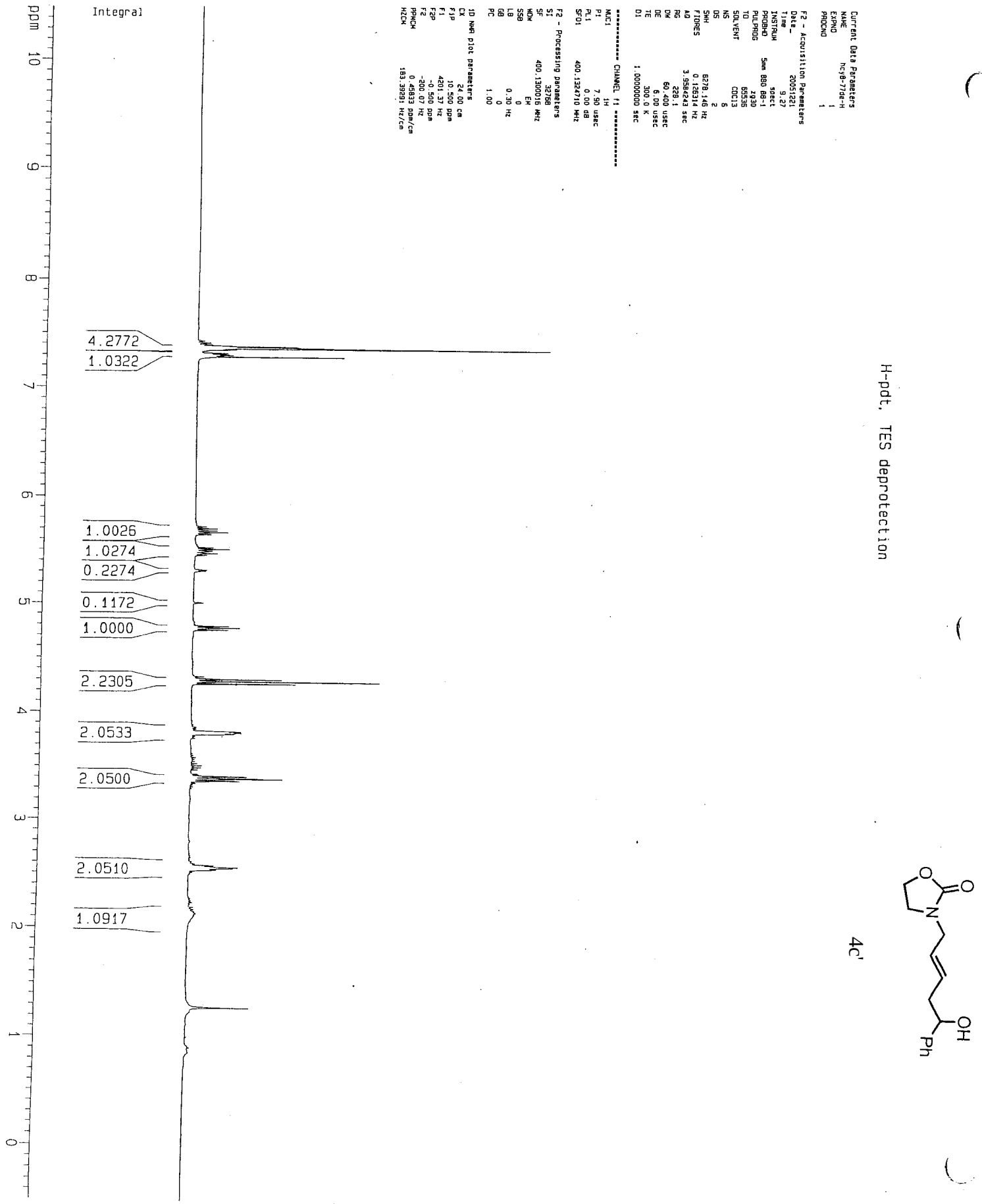
F2 - Processing parameters
SI                32768
SF          100.6127529 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                24.00 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM            9.58333 ppm/cm
HZCM           964.20551 Hz/cm
```

ppm

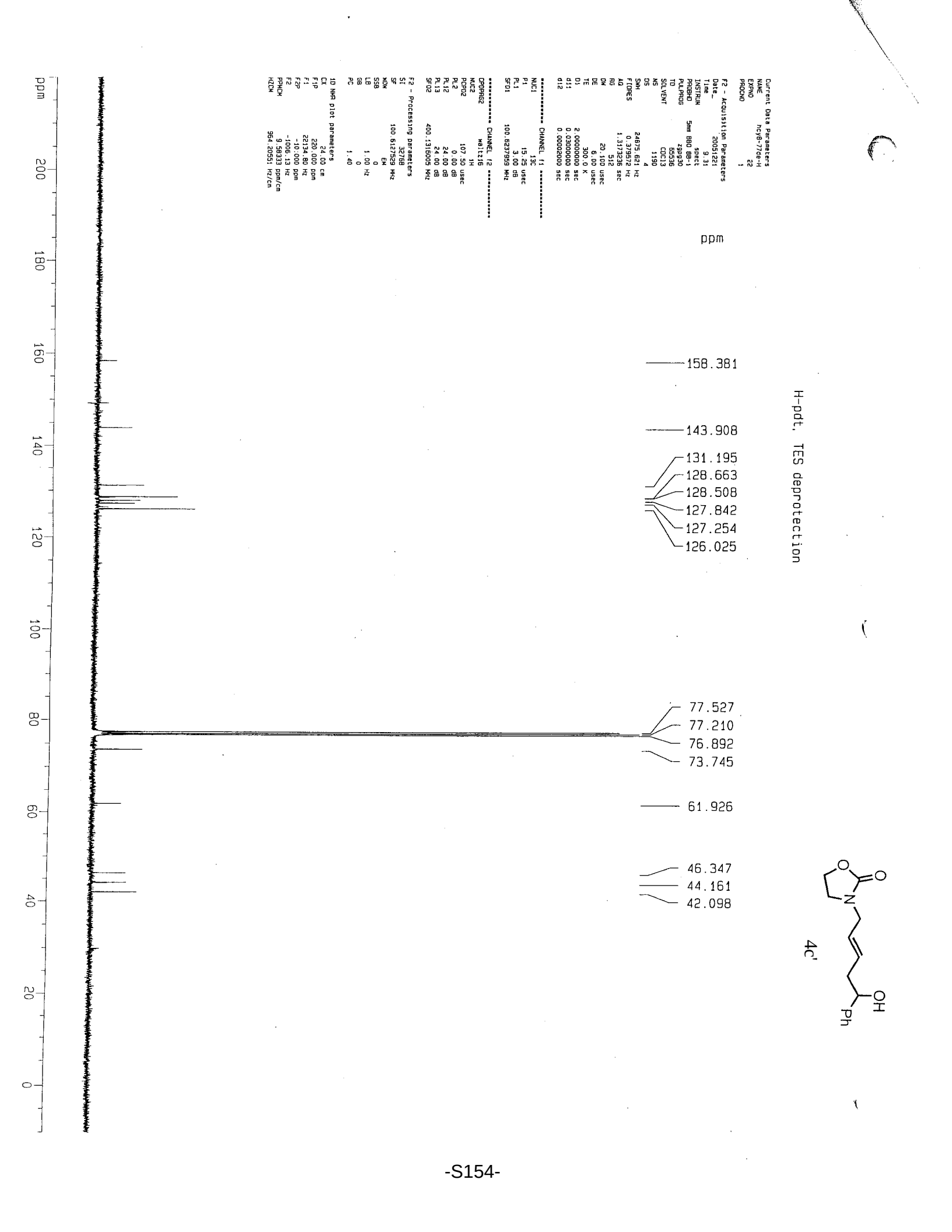
158.419 148.100 143.060 128.215 127.347 126.281 111.991 78.133 77.545 77.227 76.909 61.767 44.257 42.823 27.929 6.999 4.942

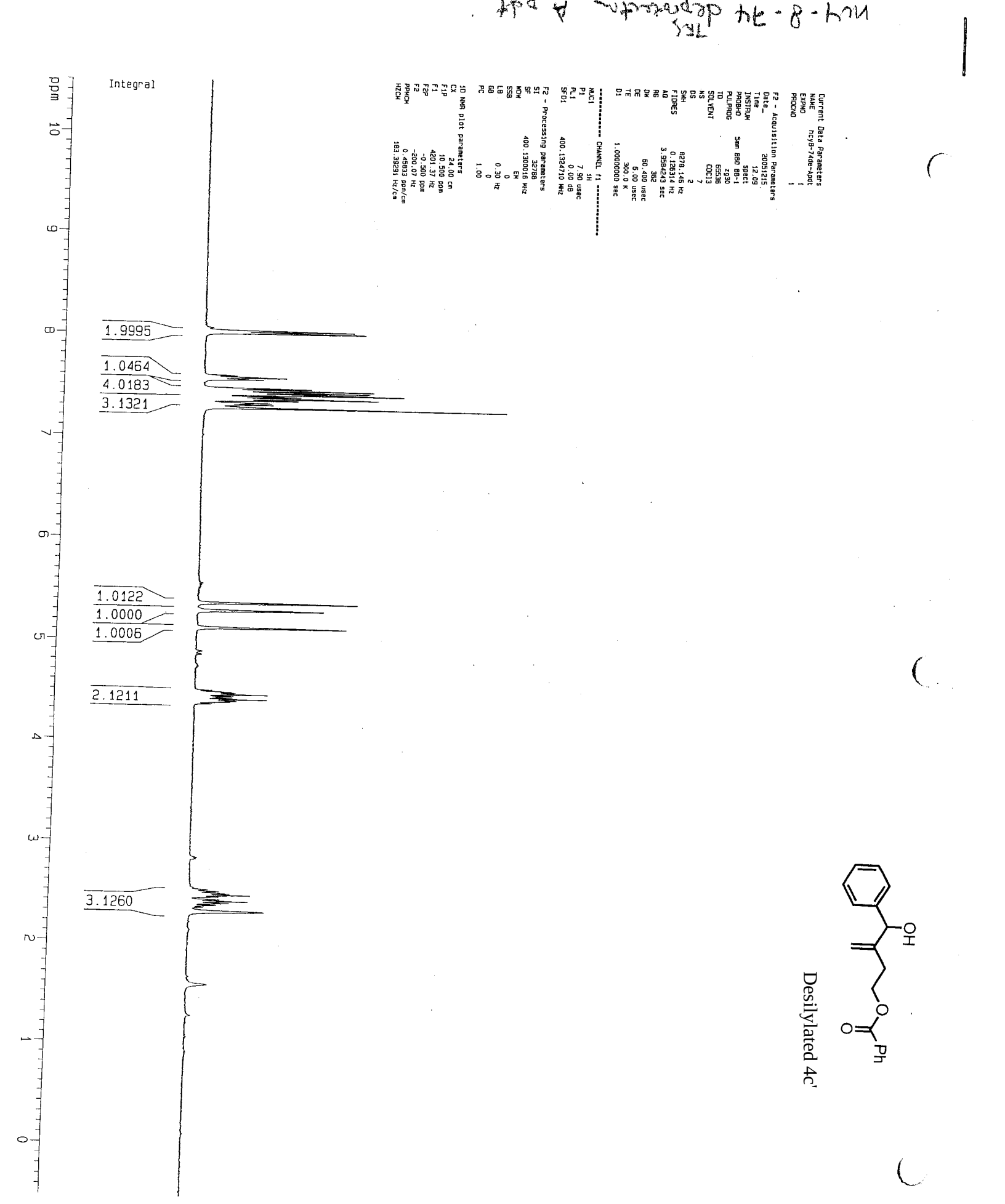




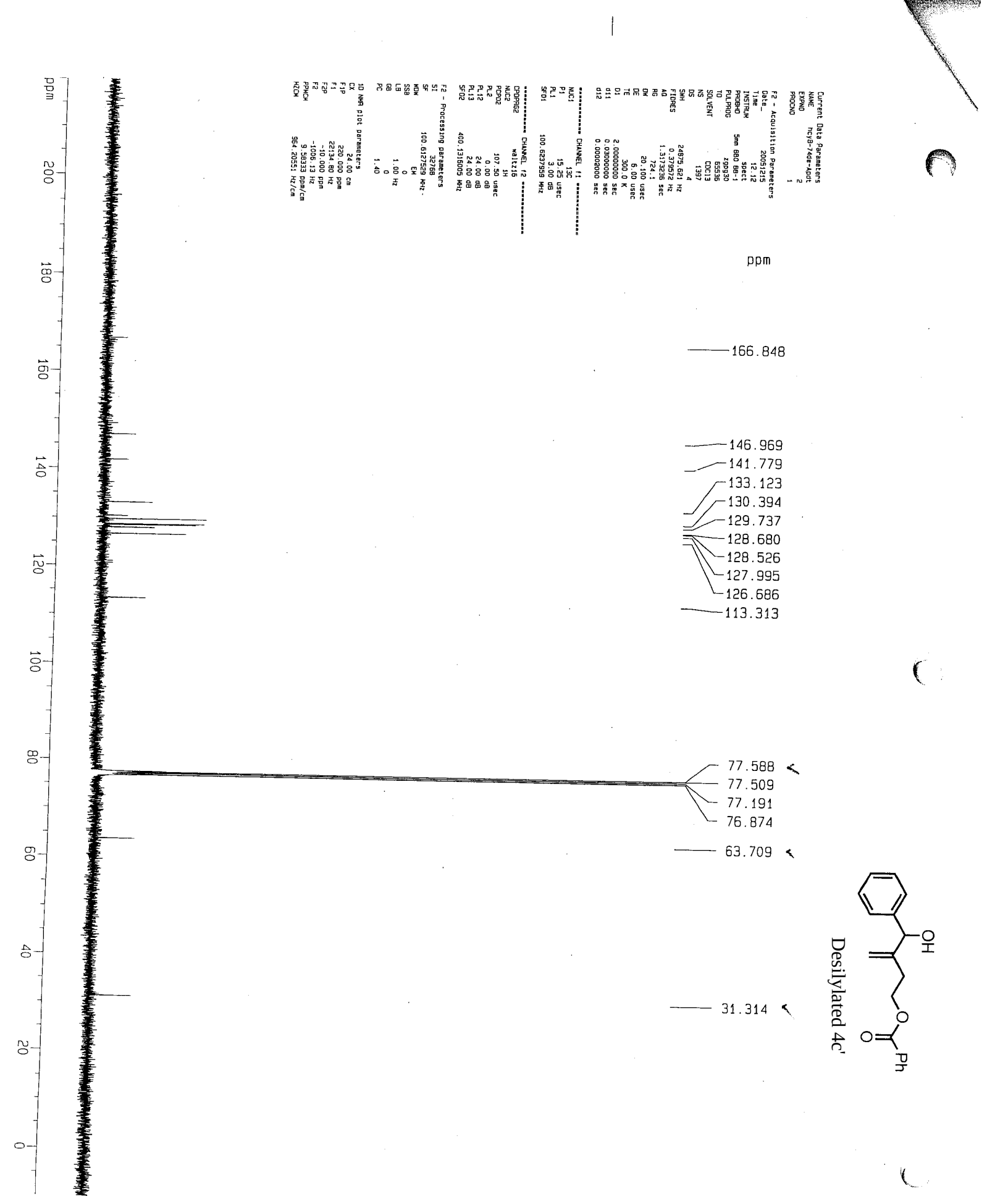
H-pdt, TES deprotection

4c'

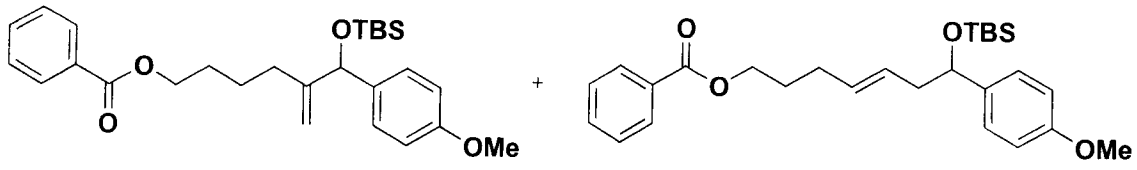

Current Data Parameters
NAME hcy8-77d-H
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051221
Time 9.31
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1190
DS 4
SWH 24675.621 Hz
FIDRES 0.375972 Hz
AQ 1.3173236 sec
RG 512
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 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 100.6127529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.16833 ppm/cm
HZCM 964.20551 Hz/cm
ppm
158.381
143.908
131.195
128.663
128.508
127.842
127.254
126.025
77.527
77.210
76.892
73.745
61.926
46.347
44.161
42.098
H-pdt, TES deprotection
4c'
OH
Ph
ppm
200
180
160
140
120
100
80
60
40
20
0



Desilylated 4c'


Current Data Parameters
NAME hcy8-74de-Apdt
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051215
Time 12.12
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1397
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 724.1
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 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 100.6127629 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1005.13 Hz
PPMCM 9.58333 ppm/cm
HZCM 964.20551 Hz/cm
ppm
166.848
146.969
141.779
133.123
130.394
129.737
128.680
128.526
127.995
126.686
113.313
77.588
77.509
77.191
76.874
63.709
31.314
OH
O
O
Ph
Desilylated 4c'

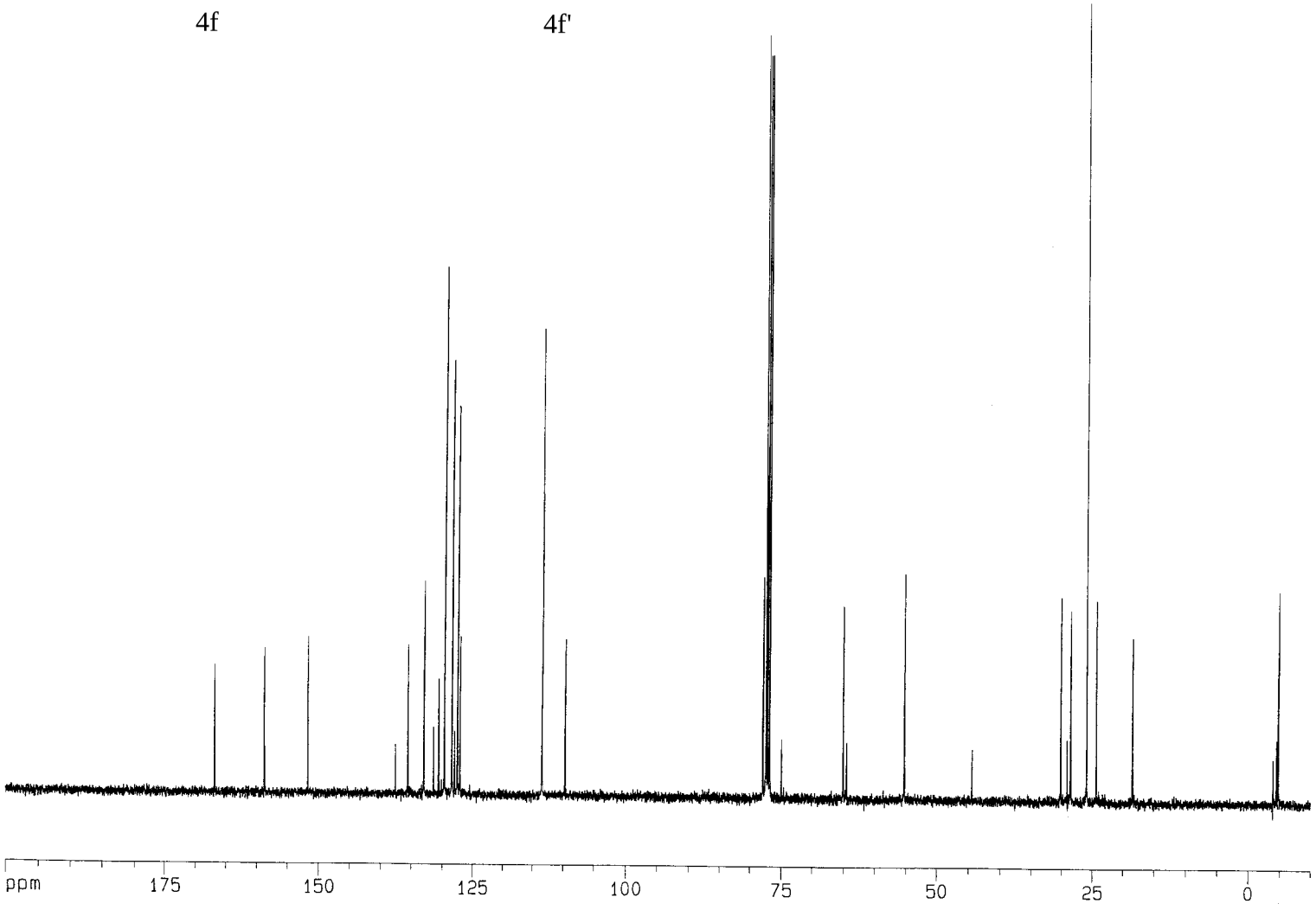
SN050737 allylic and ene



4f

+

4f'




```
Current Data Parameters
NAME          SN736-all-C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          20050728
Time             20.16
INSTRUM          spect
PROBHD     5mm BBO BB-1
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                 638
DS                   4
SWH          25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG               1625.5
DW              19.900 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               15.25 usec
PL1               3.00 dB
SFO1        100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2               1H
PCPD2           107.50 usec
PL2               0.00 dB
PL12             24.00 dB
PL13             24.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127522 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

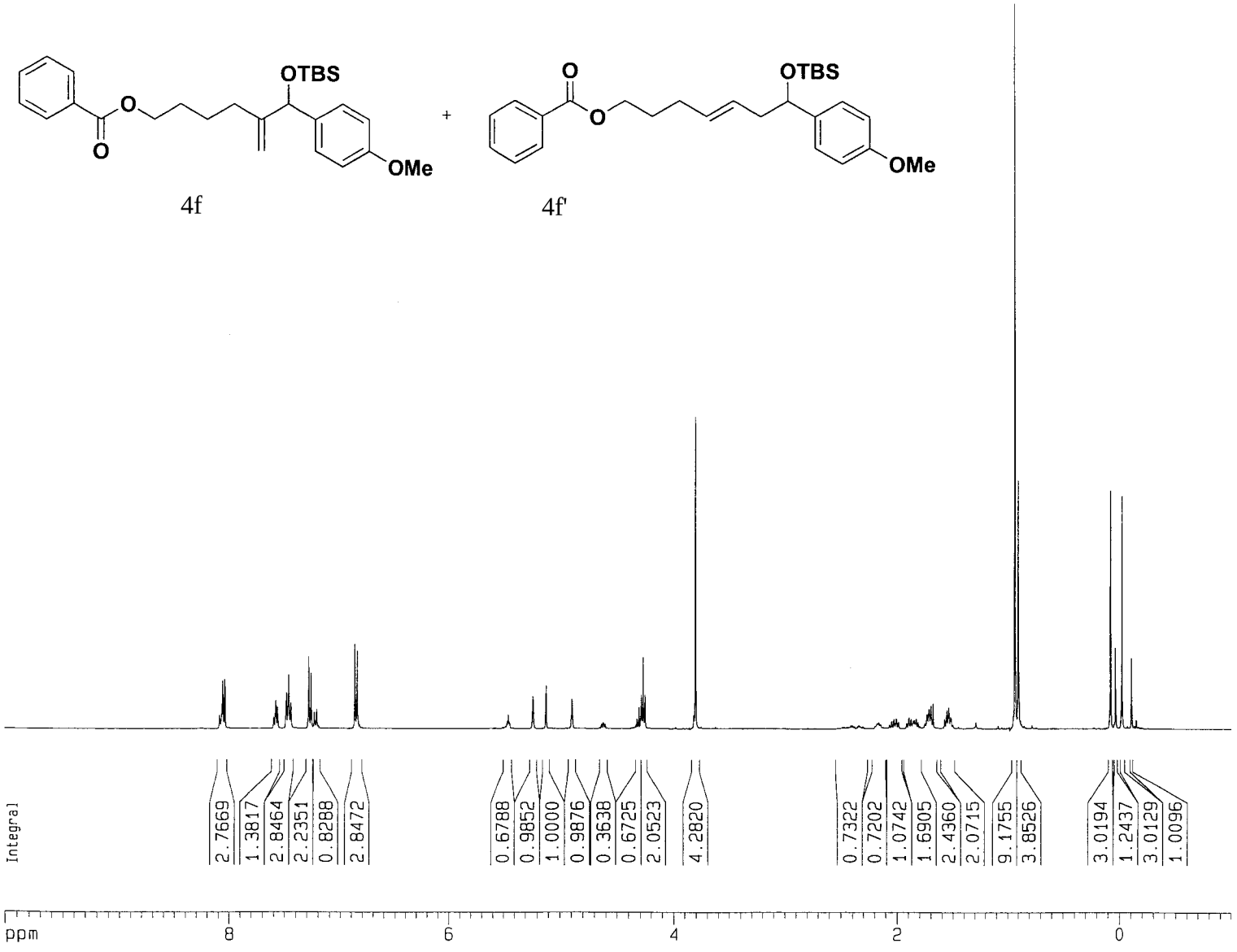
1D NMR plot parameters
CX              20.00 cm
F1P            200.000 ppm
F1            20122.55 Hz
F2P            -10.000 ppm
F2            -1006.13 Hz
PPMCM         10.50000 ppm/cm
HZCM        1056.43396 Hz/cm
```

SN050736 allylic and ene

4f

+

4f'



Current Data Parameters

NAME	SN736-all-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050728
Time	20.11
INSTRUM	spect
PROBHD	5mm BBO BB-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	45.3
DW	60.400 usec
DE	6.00 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

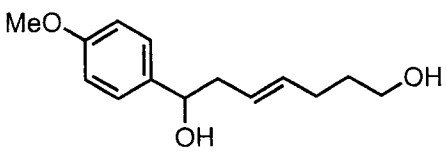
F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.55000 ppm/cm
HZCM	220.07150 Hz/cm

TES deprot H-pdt



Desilylated 4g

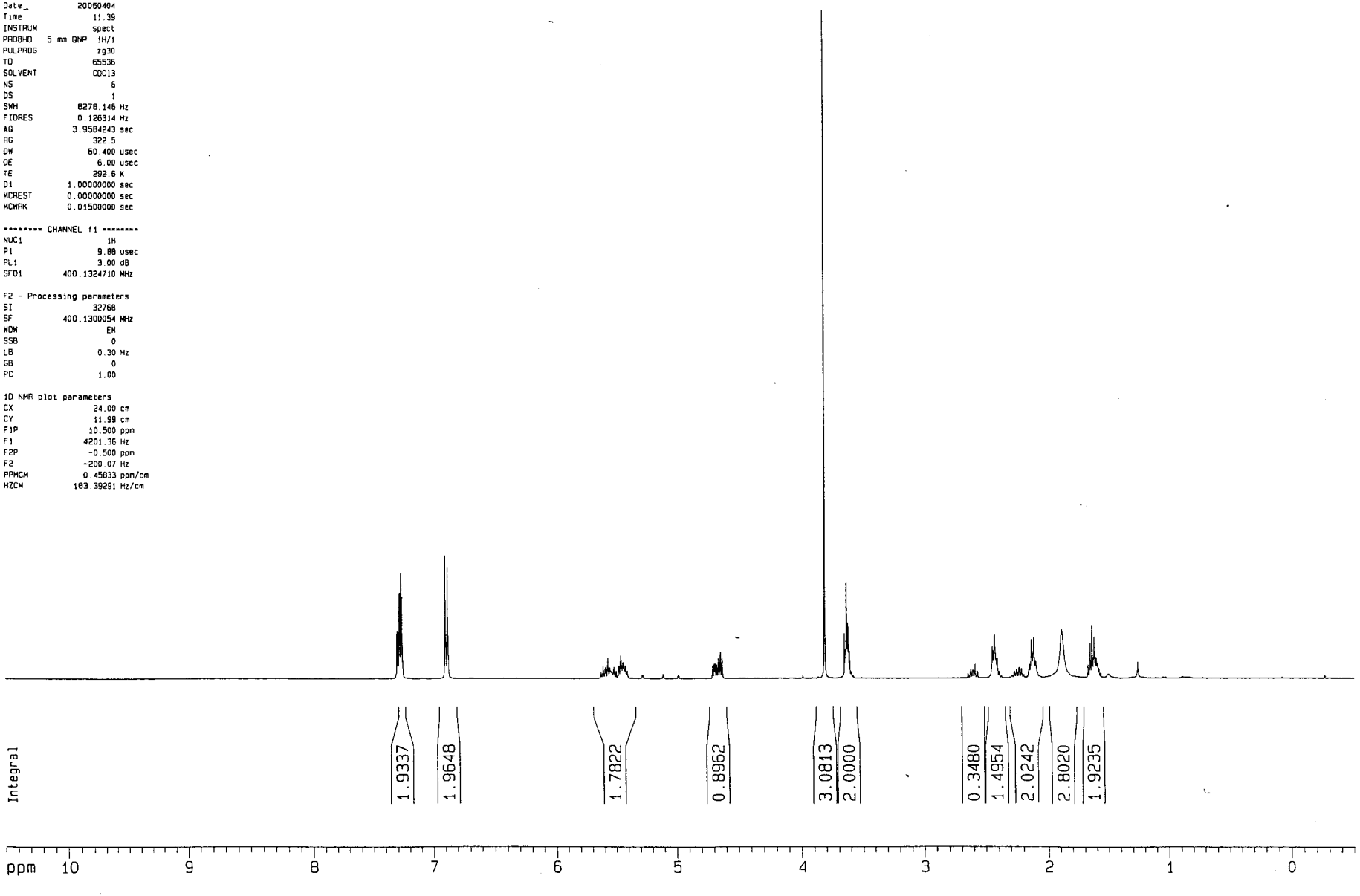
```
Current Data Parameters
NAME        hcy8-150de-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060404
Time               11.39
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                65536
SOLVENT            CDCl3
NS                     6
DS                     1
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 322.5
DW                60.400 usec
DE                 6.00 usec
TE                292.6 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.88 usec
PL1                 3.00 dB
SFO1          400.1324710 MHz

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

1D NMR plot parameters
CX                 24.00 cm
CY                 11.99 cm
F1P               10.500 ppm
F1               4201.36 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM            0.45833 ppm/cm
HZCM           183.39291 Hz/cm
```



Desilylated 4g

```
Current Data Parameters
NAME          hcy8-150de-H
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_          20060404
Time              11 42
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2499
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                 3251
DW               20.850 usec
DE                6.00 usec
TE                293.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              88.01 usec
PL2                 3.00 dB
PL12               22.00 dB
PL13               22.00 dB
SFO2         400.1316005 MHz

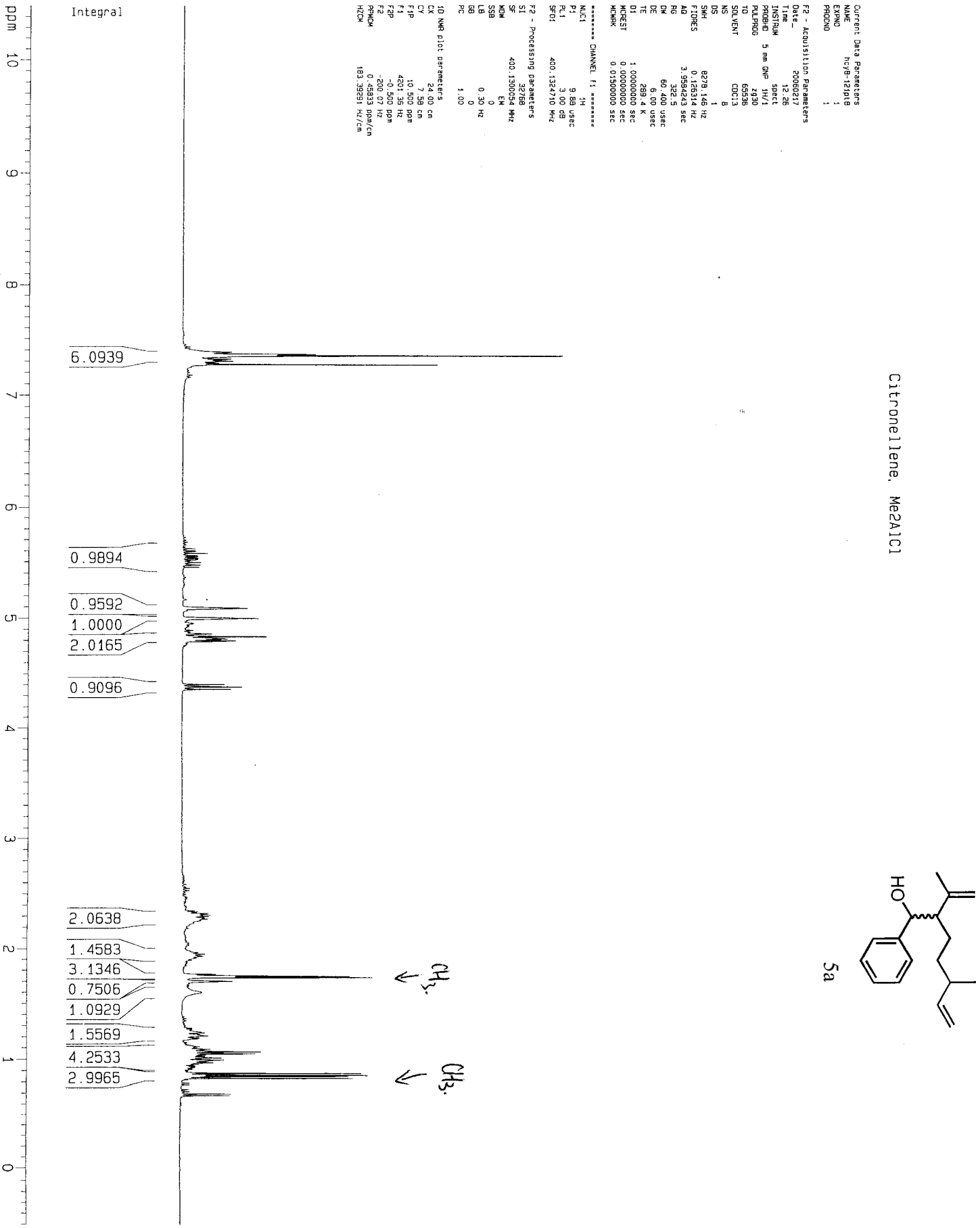
F2 - Processing parameters
SI                 32768
SF           100.6127505 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                24.00 cm
CY                25.48 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM            9.58333 ppm/cm
HZCM           964.20544 Hz/cm
```

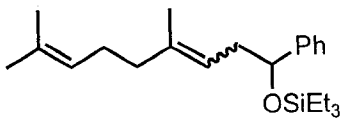
ppm

159.148
136.549
136.408
134.226
132.489
127.236
127.214
126.590
126.121
113.962
113.939
77.540
77.222
76.905
73.674
73.373
62.725
61.957
55.489
42.809
37.324
32.328
32.065
29.540
23.653

ppm 200 180 160 140 120 100 80 60 40 20 0


Citronellene, Me2AlCl
5a
HO
Integral
ppm
6.0939
0.9894
0.9592
1.0000
2.0165
0.9096
2.0638
1.4583
3.1346
0.7506
1.0929
1.5569
4.2533
2.9965
CH3.
CH3.
Current Data Parameters
NAME hcy8-12ipt8
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060217
Time 12.26
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 1
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 289.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 3.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 24.00 cm
CY 7.58 cm
F1P 10.500 ppm
F1 4201.35 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.45833 ppm/cm
HZCM 183.39231 Hz/cm

H-pdt



5b

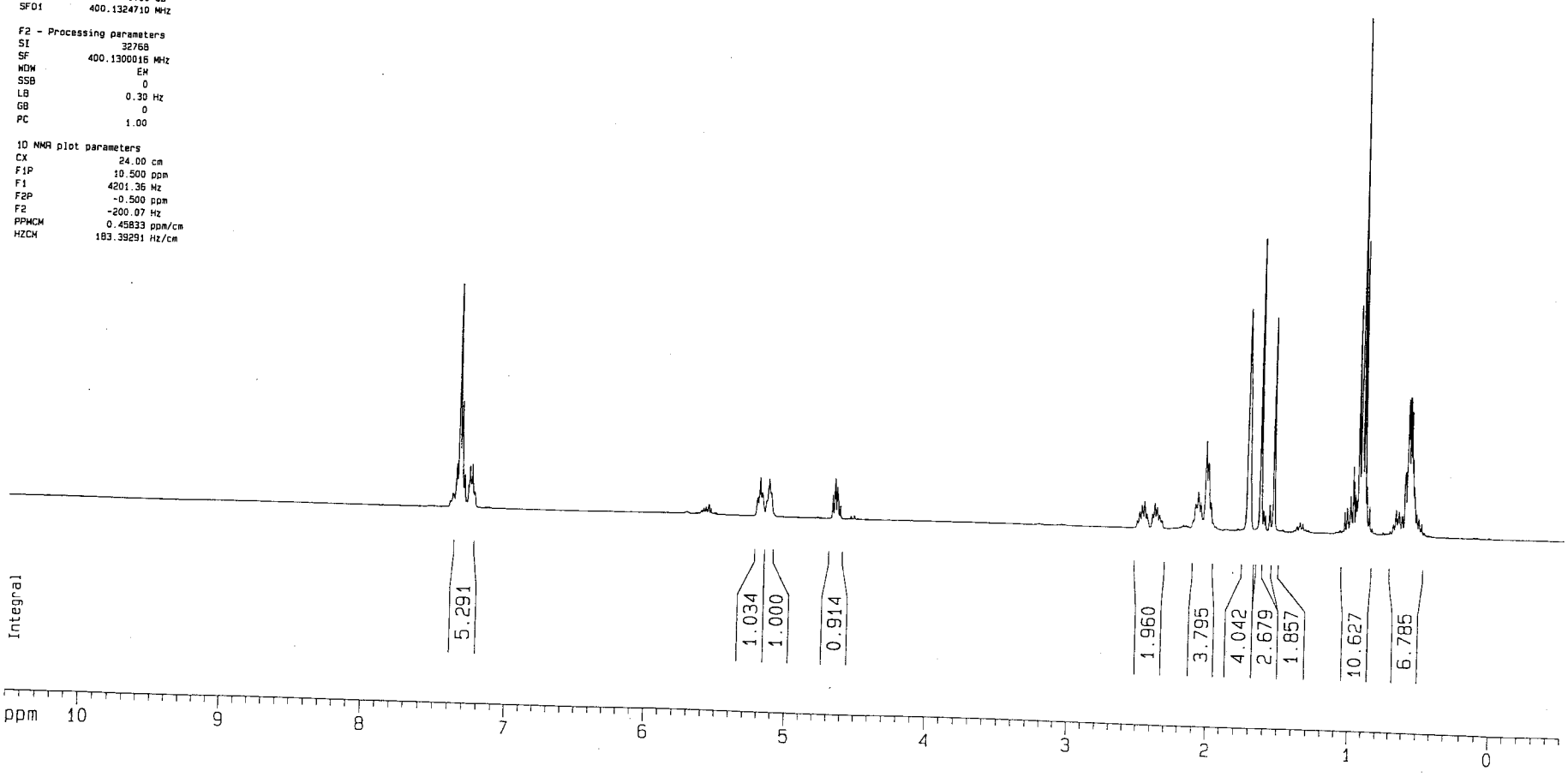
```
Current Data Parameters
NAME        hcy8-84H-pdt
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20060106
Time               12.0i
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                     4
DS                     2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG                    32
DW               60.400 usec
DE                6.00 usec
TE                300.0 K
D1           1.00000000 sec

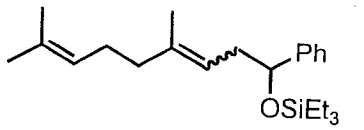
========== CHANNEL f1 ==========
NUC1                  1H
P1                 7.90 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300016 MHz
WDW                   EM
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
PPMCM             0.45833 ppm/cm
HZCM            183.39291 Hz/cm
```



-S162-



5b

```
Current Data Parameters
NAME        hcy8-84H-pdt
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060106
Time               12.05
INSTRUM            spect
PROBHD     5mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   59
DS                    4
SWH           24875.621 Hz
FIDRES        0.379572 Hz
AQ            1.3173236 sec
RG                 1024
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                15.25 usec
PL1                3.00 dB
SF01        100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2           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          100.6127529 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

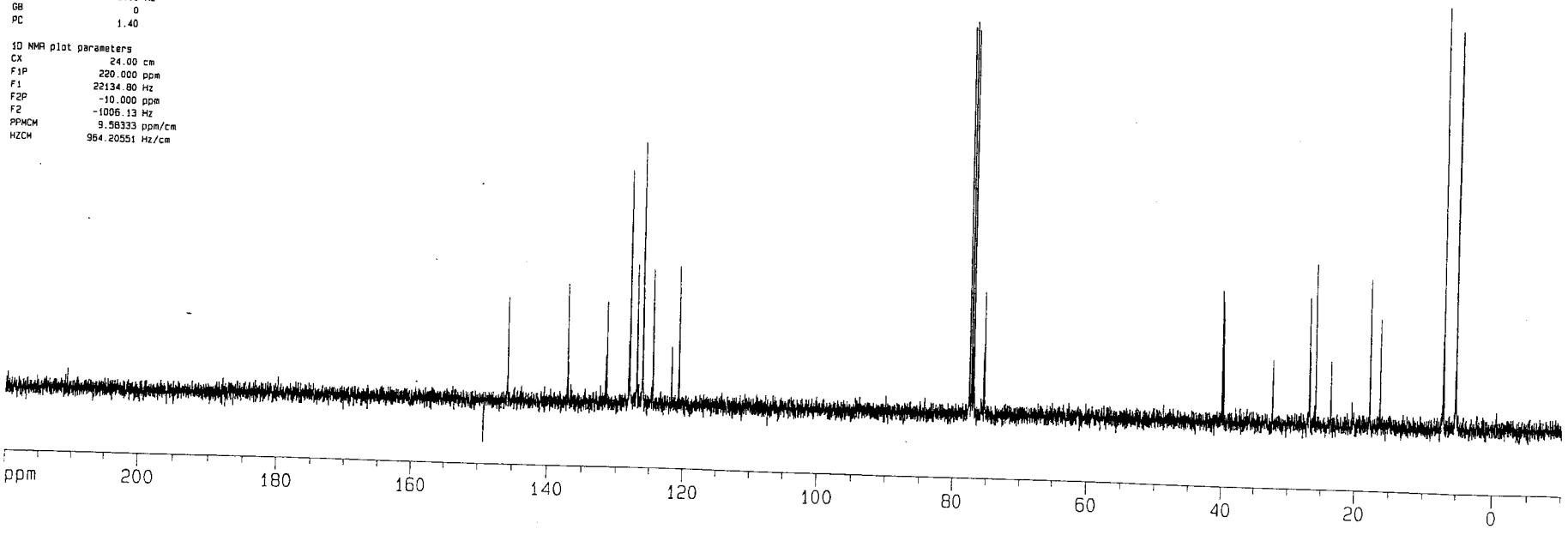
1D NMR plot parameters
CX                24.00 cm
F1P              220.000 ppm
F1             22134.80 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM           9.58333 ppm/cm
HZCM          964.20551 Hz/cm
```

ppm

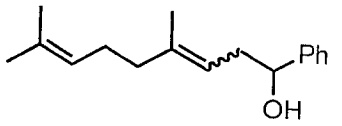
145.817 145.763 137.192 131.479 128.228 128.099 128.042 127.035 126.998 126.132 126.083 126.032 124.599 124.536 121.745 120.676

77.531 77.213 76.896 75.518 75.330

40.034 39.837 39.686 32.284 26.831 26.716 25.916 25.893 23.638 17.841 17.819 16.343 7.037 6.978 6.950 5.160 5.017 4.993



TES deprotection, H-pdt



5b (TES deprotected)

```
Current Data Parameters
NAME          hcy8-84de-p
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060107
Time                13.39
INSTRUM             spect
PROBHD     5mm BBO BB-1
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                      3
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   90.5
DW                60.400 usec
DE                 6.00 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           400.1300016 MHz
WDW                    EM
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
PPMCM             0.45833 ppm/cm
HZCM           183.39291 Hz/cm
```