

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

Ng, S.-S.; Jamison, T. F. *

Massachusetts Institute of Technology, Department of Chemistry, Cambridge, MA 02139

Supporting Information

Experimental Procedures, Analytical and Spectroscopic Data for Compounds **1a** – **2l**.

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¹H and ¹³C NMRs for compounds **1a** – **2l**.

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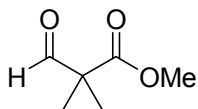
General Information.

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. All aldehydes were distilled and saturated with nitrogen before use. Bis(cyclooctadienyl)nickel(0) ($\text{Ni}(\text{cod})_2$) and tris-(*o*-methoxyphenyl)-phosphine was 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, 4-methyl-1-pentene, 7-methyl-1,6-octadiene were purchased from Aldrich Chemical Co. and used as received. Dicyclohexylphenylphosphine was purchased from Aldrich Chemical Co., stored under nitrogen atmosphere and used without further purification. Triethylsilyltrifluoromethanesulfonate (TESOTf) and trimethylsilyl-trifluoromethanesulfonate (TMSOTf) were purchased from Aldrich Chemical Co. and were distilled over calcium hydride before use. Tert-butyldimethylsilyl-trifluoromethanesulfonate (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 F₂₅₄ plates. The developed chromatogram was analyzed by UV lamp (254 nm), ethanolic phosphomolybdic acid (PMA) or potassium permanganate (KMnO_4). Liquid chromatography was performed using a forced flow (flash chromatography) of the indicated solvent system on Silicycle Silica Gel (230 – 400 mesh). ^1H and ^{13}C NMR spectra were recorded on 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. Specific Rotations ($[\alpha]_D$) were measured on a Perkin-Elmer 241

polarimeter at 589 nm.

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. Distillation removed residue dichloromethane and obtained 2,2-dimethyl-3-oxo-propionic acid methyl ester as a colorless oil (7 g, 48% yield).

^1H NMR (300 MHz, CDCl_3 , δ): 9.60 (s, 1H); 3.70 (s, 3H); 1.29 (s, 6H).

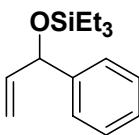
^{13}C NMR (75 MHz, CDCl_3 , δ): 199.1, 173.2, 53.9, 52.6, 19.7.

IR (NaCl, thin film): 2988, 2958, 1726, 1468, 1278, 1151, 866.

Nickel-catalyzed couplings of ethylene and liquid aldehydes (1a, 1b, 1c, 1d, 1h, 1i).

General procedure. A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $\text{Ni}(\text{cod})_2$ (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. Next triethylamine (418 μL , 3 mmol, 600 mol%) was added. Silyltriflate (0.875 mmol, 175 mol%, as specified) was added. Aldehyde (0.5 mmol, 100 mol%, as specified) was added. The mixture was stirred at room temperature for 3 – 18 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.



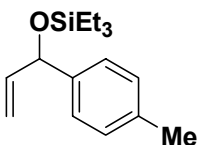
Triethyl-(1-phenyl-allyloxy)-silane (1a). The reaction of ethylene and benzaldehyde (51 μ L, 0.5 mmol) with $\text{Ni}(\text{cod})_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1a** in 82% isolated yield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

^1H 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{24}\text{OSi}$, 271.149; found, 271.150.



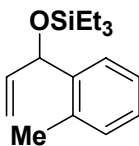
Triethyl-(1-p-tolyl-allyloxy)-silane (1b). The reaction of ethylene and *p*-tolualdehyde (59 μ L, 0.5 mmol) with $\text{Ni}(\text{cod})_2$, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1b** in 88% isolated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

^1H 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{26}\text{OSi}$, 285.165; found, 285.165.



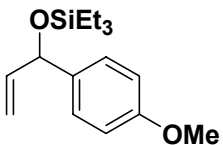
Triethyl-(1-*o*-tolyl-allyloxy)-silane (1c). The reaction of ethylene and *o*-tolualdehyde (58 μ L, 0.5 mmol) with Ni(cod)₂, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1c** in 93% isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, δ): 7.50 (d, J = 7.0, 1H); 7.11 – 7.24 (m, 4H); 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).

¹³C NMR (100 MHz, CDCl₃, 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 C₁₆H₂₆OSi, 285.165; found, 285.165.



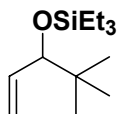
Triethyl-[1-(4-methoxy-phenyl)-allyloxy]-silane (1d). The reaction of ethylene and *p*-anisaldehyde (61 μ L, 0.5 mmol) with Ni(cod)₂, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1d** in 95% isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, 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).

¹³C NMR (100 MHz, CDCl₃, 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 C₁₆H₂₆O₂Si, 301.159; found, 301.159.

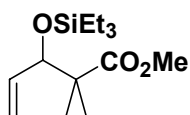


(1-tert-Butyl-allyloxy)-triethyl-silane (1h). The reaction of ethylene and pivaldehyde (55 μ L, 0.5 mmol) with Ni(cod)₂, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1h** in 70% isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, δ): 5.97 (ddd, J = 5.9, 10.2, 16.9 Hz, 1H); 5.12 (bs, 1H); 5.10 (bs, 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).

¹³C NMR (100 MHz, CDCl₃, 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.



2,2-Dimethyl-3-triethylsilyloxy-pent-4-enoic acid methyl ester

(1i). 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 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1i** in 81% (0.28 mmol) isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, 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); 5.10 (dt, J = 1.4, 10.2 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).

¹³C NMR (100 MHz, CDCl₃, δ): 177.4, 137.8, 117.3, 79.2, 51.8, 48.3, 21.4, 19.9, 7.0, 5.2.

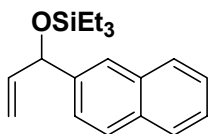
IR (NaCl, thin film): 2954, 2878, 1745, 1732, 1642, 1468, 1261, 1087, 834.

HRMS-ESI (m/z): [M + Na]⁺ calcd for C₁₄H₂₈O₃Si, 295.170; found, 295.171.

Nickel-catalyzed couplings of ethylene and 2-naphthaldehyde (1e, 1f, 1j).

General procedure. 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 2-naphthaldehyde (78 mg, 0.5 mmol) 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 μ L, 3 mmol, 600 mol%) was added. Silyltriflate (0.875 mmol, 175 mol%, as specified) was added. The mixture was stirred at room temperature for 5 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.



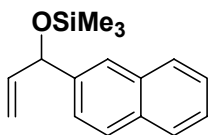
Triethyl-(1-naphthalen-2-yl-allyloxy)-silane (1e). The reaction of ethylene and 2-naphthaldehyde with Ni(cod)₂, tris-*o*-methoxyphenylphosphine and TESOTf (197 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1e** in 95% isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, 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).

¹³C NMR (100 MHz, CDCl₃, 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 C₁₉H₂₆OSi, 321.165; found, 321.164.



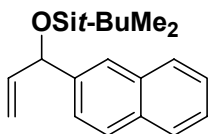
Trimethyl-(1-naphthalen-2-yl-allyloxy)-silane (1f). The reaction of ethylene and 2-naphthaldehyde with Ni(cod)₂, tris-*o*-methoxyphenylphosphine and TMSOTf (158 μ L, 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1f** in 60% isolated yield as a colorless oil.

^1H 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{20}\text{OSi}$, 279.118; found, 279.119.



Tert-Butyl-dimethyl-(1-naphthalen-2-yl-allyloxy)-silane (1g). The reaction of ethylene and 2-naphthaldehyde with $\text{Ni}(\text{cod})_2$, tris-*o*-methoxyphenylphosphine and TBSOTf (201 μL , 0.875 mmol), triethylamine in toluene following the general procedure above, afforded **1g** in 67% isolated yield as a colorless oil.

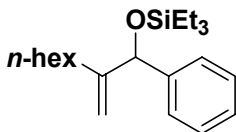
^1H NMR (400 MHz, CDCl_3 , δ): 7.80 – 7.92 (m, 4H); 7.45 – 7.55 (m, 3H); 6.04 (ddd, J = 5.8, 10.2, 16.8 Hz, 1H); 5.39 (dt, J = 1.5, 17.0, 1H); 5.38 (s, 1H); 5.14 (dt, J = 1.5, 10.2 Hz, 1H); 0.99 (s, 9H); 0.16 (s, 3H); 0.06 (s, 3H).

^{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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{26}\text{OSi}$, 321.165; found, 321.164.

Nickel-catalyzed couplings of α -olefin and benzaldehyde (**1j**, **1k**, **1l**).



Triethyl-(2-methylene-1-phenyl-octyloxy)-silane (1j). A 25 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $\text{Ni}(\text{cod})_2$ (110 mg, 0.4 mmol, 20 mol%) and dicyclohexylphenylphosphine (220 mg, 0.8 mmol,

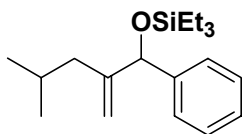
40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was brought out of the glove box and connected to an argon line. The catalyst mixture was dissolved in toluene (5.0 mL) under argon and stirred 5 min at room temperature. 1-octene (1.57 mL, 10 mmol, 500 mol%) was added. Triethylamine (1.7 mL, 12 mmol, 600 mol%) was added. TESOTf (0.8 mL, 3.5 mmol, 175 mol%) was added. Benzaldehyde (200 μ L, 2 mmol, 100 mol%) in 5 mL toluene was added to the reaction mixture over 10 min. The mixture was stirred at room temperature for 15 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 **1j** in 44% isolated yield as a colorless oil.

^1H 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); 5.25 (d, J = 5.9 Hz, 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{21}\text{H}_{36}\text{OSi}$, 355.243; found, 355.242.



Triethyl-(4-methyl-2-methylene-1-phenyl-pentyloxy)-silane (1k).

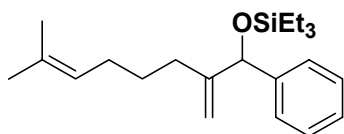
A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $\text{Ni}(\text{cod})_2$ (27.5 mg, 0.2 mmol, 20 mol%) and dicyclohexylphenylphosphine (55 mg, 0.4 mmol, 40 mol%) were added to the round bottom flask, the flask was sealed with a septum, and the sealed flask was 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 μ L, 5 mmol, 1000 mol%) was added. Triethylamine (418 μ L, 3 mmol, 600 mol%) was added. TESOTf (197 μ L, 0.875 mmol, 175 mol%) was added. Benzaldehyde (51 μ L, 0.5 mmol, 100 mol%) was added to the reaction mixture. 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 the crude mixture was diluted in hexane. Purification via flash chromatography on silica afforded **1k** in 44% isolated yield as a colorless oil.

^1H 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{32}\text{OSi}$, 327.211; found, 327.212.



Triethyl-(7-methyl-2-methylene-1-phenyl-oct-6-

enyloxy)-silane (11). A 10 mL round bottom flask and a stir bar were oven-dried and brought into a glove box. $\text{Ni}(\text{cod})_2$ (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-octadiene (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 the crude mixture was diluted in hexane. Purification via flash chromatography on silica afforded **11** in 50% isolated yield as a colorless oil.

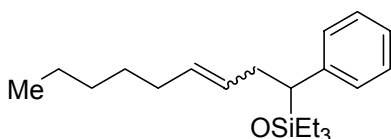
^1H NMR (400 MHz, CDCl_3 , δ): 7.40 (d, $J = 7.0$ Hz, 2H); 7.34 (t, $J = 7.8$ Hz, 2H); 7.27 (t, $J = 7.2$, 1H); 5.26 (bs, 1H); 5.18 (bs, 1H); 5.10 (t, $J = 7.2$ Hz, 1H); 4.81 (bs, 1H); 1.76 – 2.10 (m, 4H); 1.71 (s, 3H); 1.60 (s, 3H); 1.44 (quintet, $J = 7.7$ Hz); 0.97 (t, $J = 7.9$ Hz, 9H); 0.62 (dq, $J = 1.5$, 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): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{36}\text{OSi}$, 367.243; found, 367.243.

Nickel-catalyzed couplings of α -olefin and benzaldehyde (2j, 2k, 2l). For entries 10, 11, and 12, in addition to the major products isolated (i.e. **1j**, **1k** and **1l**), homoallylic minor products were also isolated (**2j**, **2k**, **2l**). NMR of the crude mixtures of all three entries suggested the ratio of the major product to the minor product was approximately 2.5 : 1. Trace amount of other regioisomers could be observed by NMR (<5%).

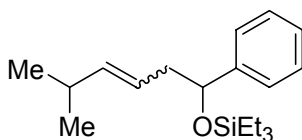


Triethyl-(1-phenyl-non-3-enyloxy)-silane (2j). The reaction of 1-octene and benzaldehyde with Ni(cod)₂, dicyclohexylphenylphosphine, TESOTf and triethylamine in toluene (see **1j** for experimental procedure) afforded **1j** (44%) and **2j** in 19% isolated yield as a colorless oil.

¹H NMR (400 MHz, CDCl₃, 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).

¹³C NMR (100 MHz, CDCl₃, 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 C₂₁H₃₆OSi, 355.243; found, 355.244.

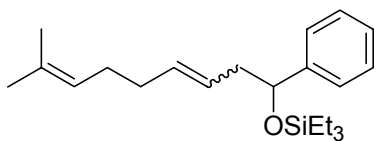


Triethyl-(5-methyl-1-phenyl-hex-3-enyloxy)-silane (2k). The reaction of 4-methyl-1-pentene and benzaldehyde with Ni(cod)₂, dicyclohexylphenylphosphine, TESOTf and triethylamine in toluene (see **1k** for experimental procedure) afforded **1k** (44%) and **2k** in ~10% isolated yield as a colorless oil. **2k** was isolated together with a small amount of a third regioisomer.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 $C_{19}H_{32}OSi$, 327.212; found, 327.212.



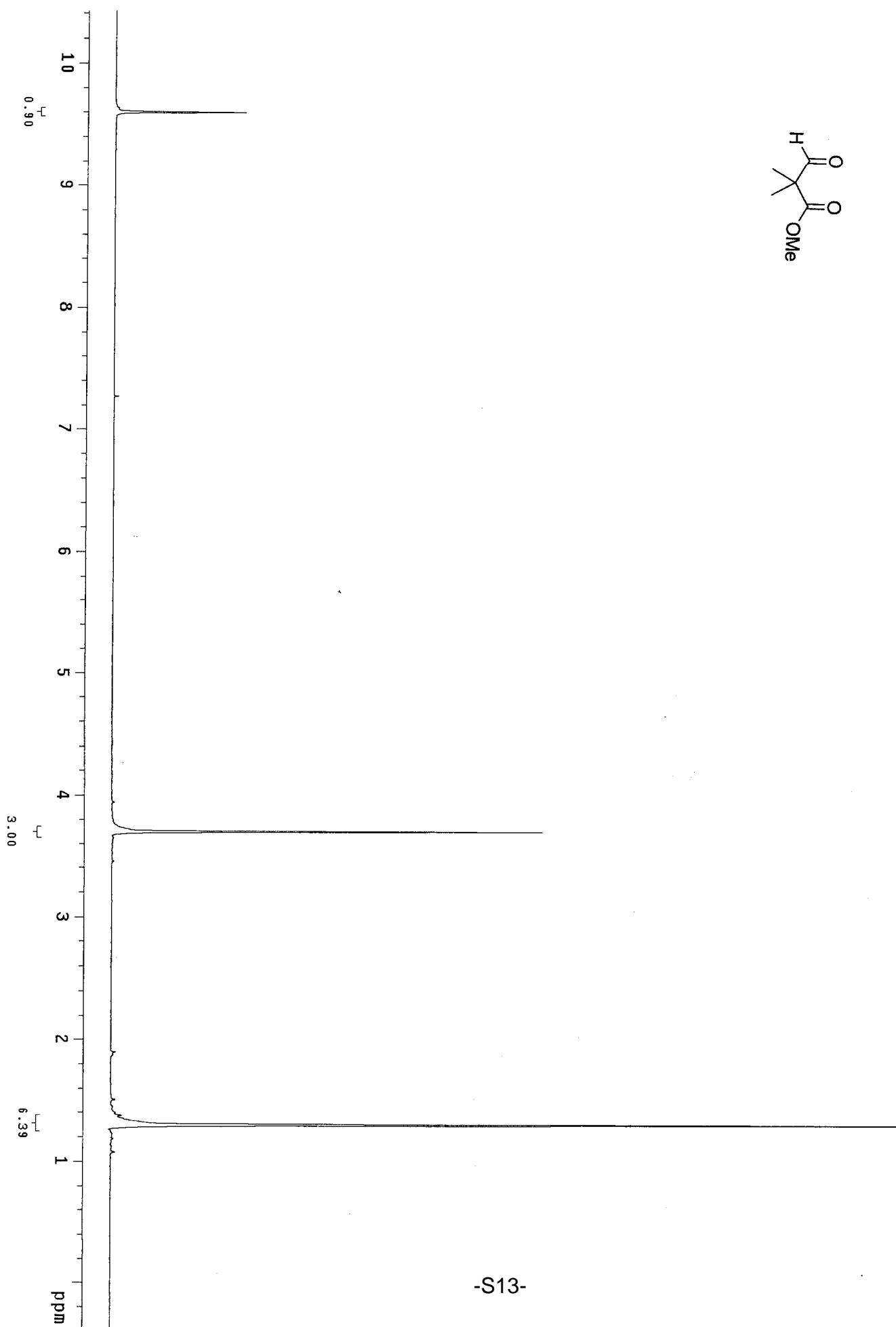
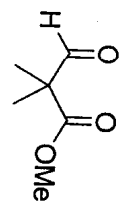
Triethyl-(8-methyl-1-phenyl-nona-3,7-dienyloxy)-silane (11). The reaction of 7-methyl-1,7-octadiene and benzaldehyde with $Ni(cod)_2$, dicyclohexylphenylphosphine, TESOTf and triethylamine in toluene (see **1k** for experimental procedure) afforded **11** (50%) and **2l** in 22% isolated yield.

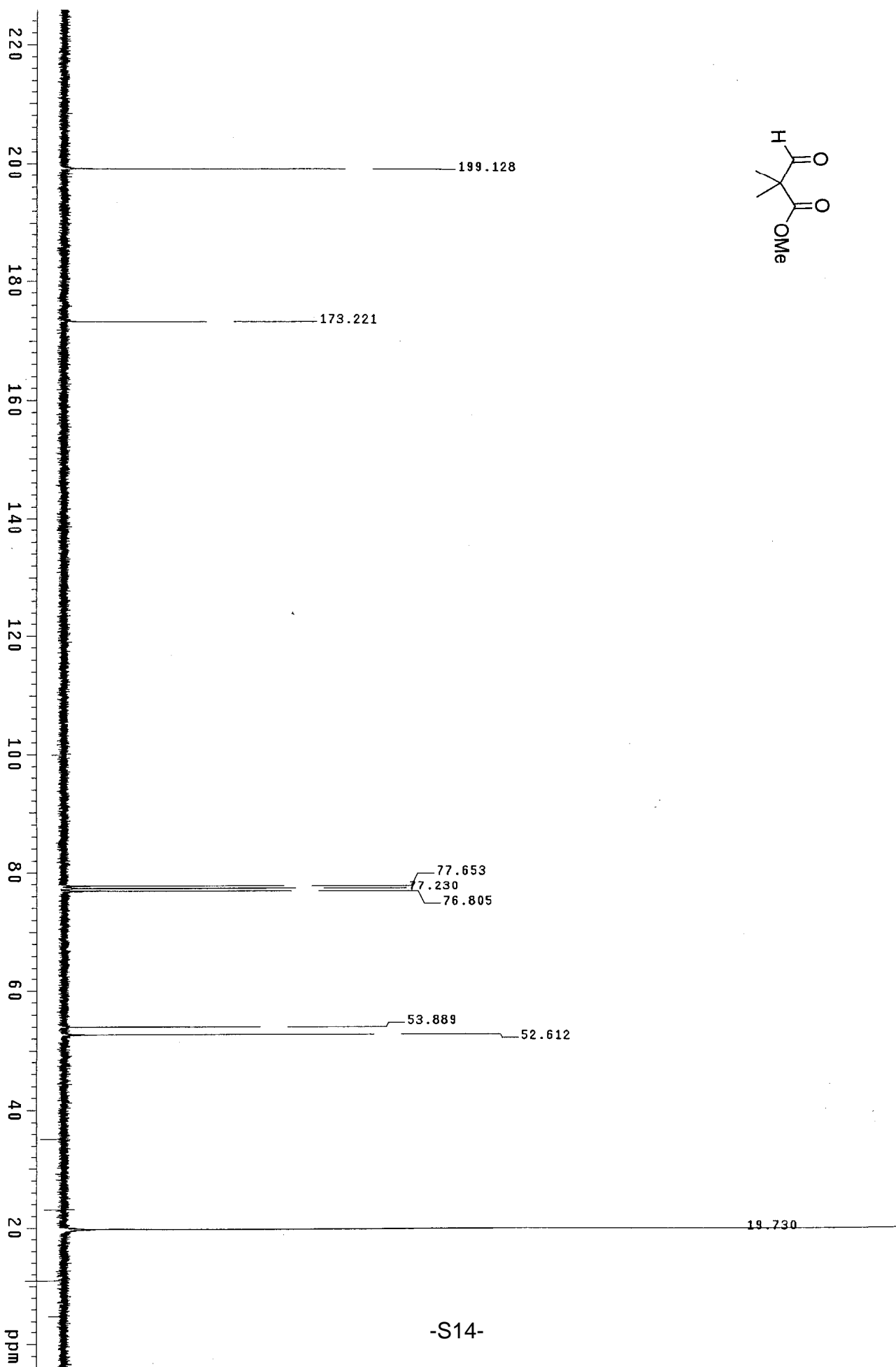
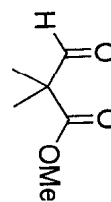
1H NMR (400 MHz, $CDCl_3$, δ): 7.30 (m, 5H); 5.45 (m, 2H); 5.15 (t, $J = 7.1$ Hz, 1H); 4.64 (dd, $J = 5.4, 7.3$ Hz, 1H); 2.45 (quintet, $J = 5.4$ Hz, 1H); 2.35 (quintet, $J = 5.9$ Hz, 1H); 2.05 (m, 4H); 1.62 (s, 3H); 1.72 (s, 3H); 1.44 (quintet, $J = 7.7$ Hz); 0.92 (t, $J = 7.9$ Hz, 9H); 0.55 (dq, $J = 1.5, 7.9$ 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.

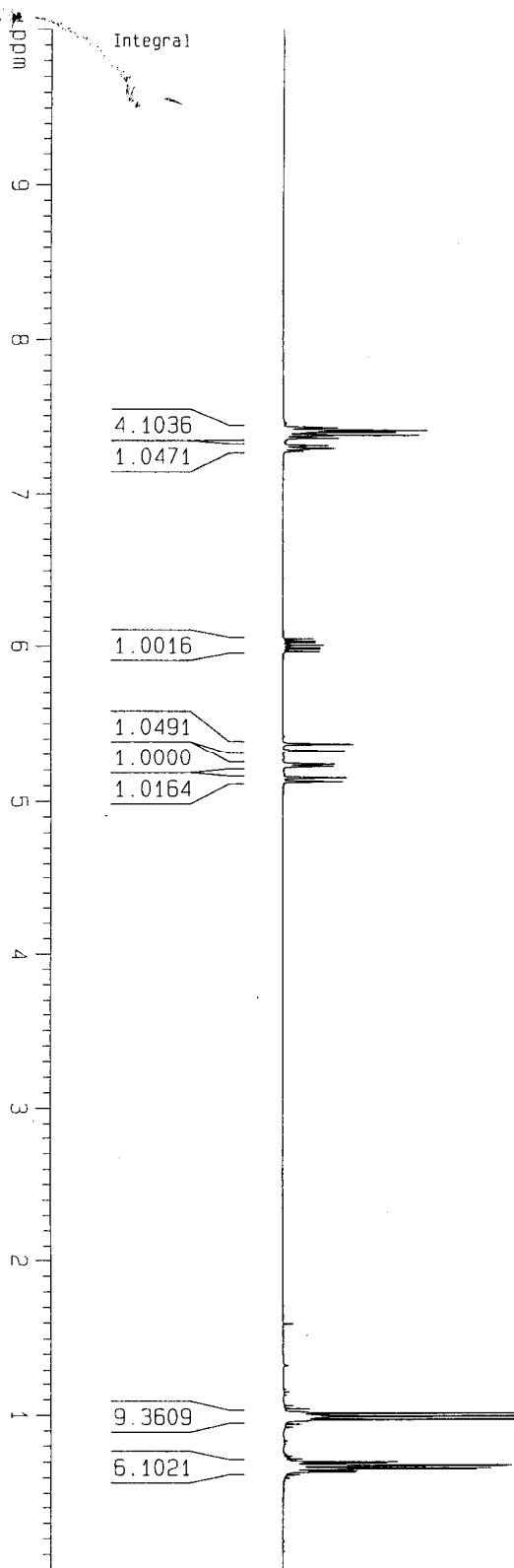
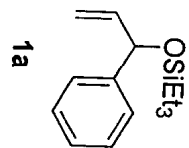
IR (NaCl, thin film): 2955, 2876, 1454, 1089, 1005, 742.

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





SN050690



Current Data Parameters
 NAME SN690-H
 EXPNO 1
 PROCNO 1

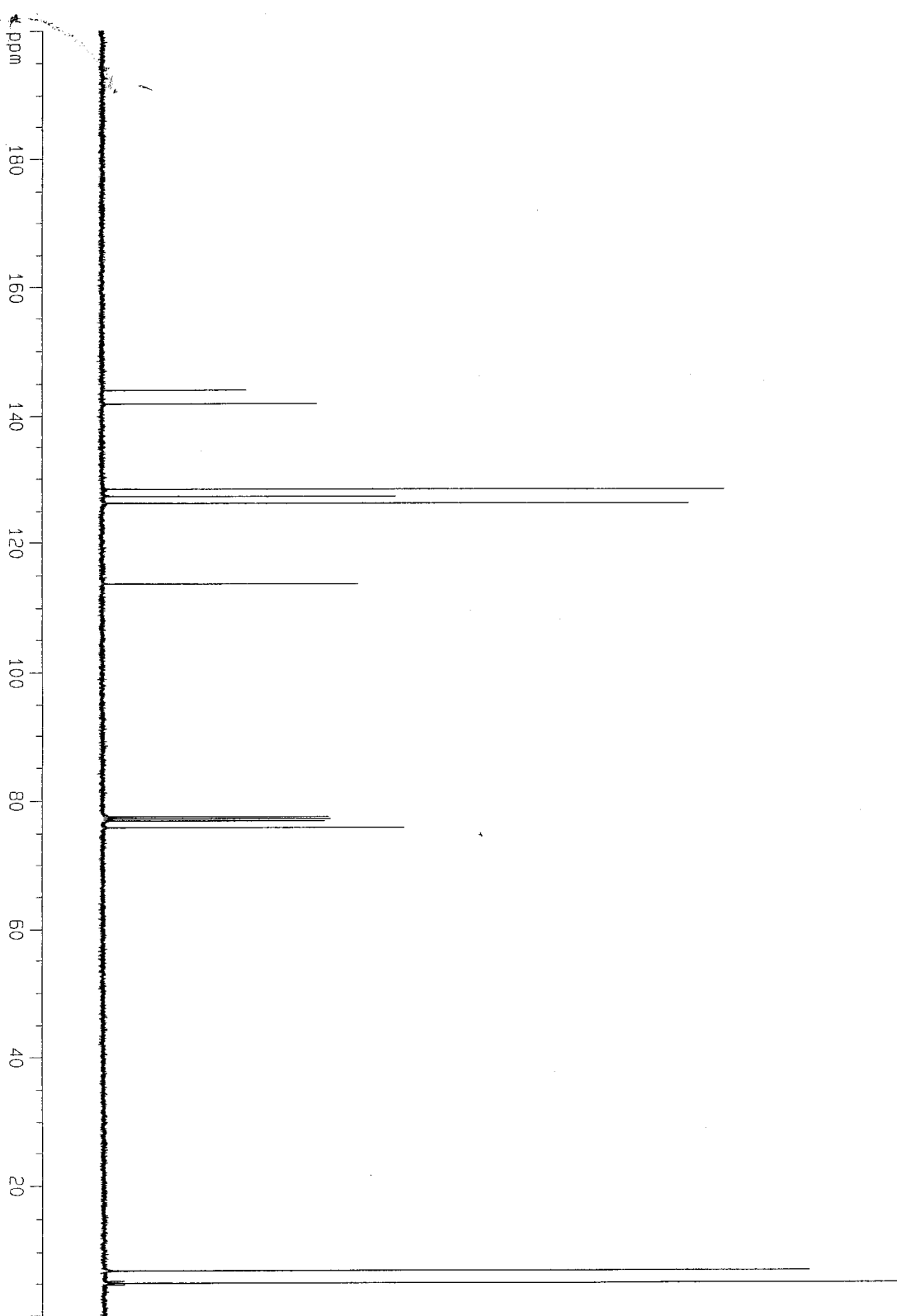
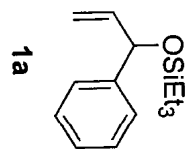
F2 - Acquisition Parameters
 Date_ 20050707
 Time 19.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 4
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 45.3
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 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

SN050690



Current Data Parameters
NAME SN690-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050707
Time 20.02

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30

TD 65536
SOLVENT CDCl3

NS 874
DS 4
SWH 24330.900 Hz
FIDRES 0.371260 Hz

AQ 1.3468148 sec
RG 1024
DW 20.550 usec
DE 6.00 usec

TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

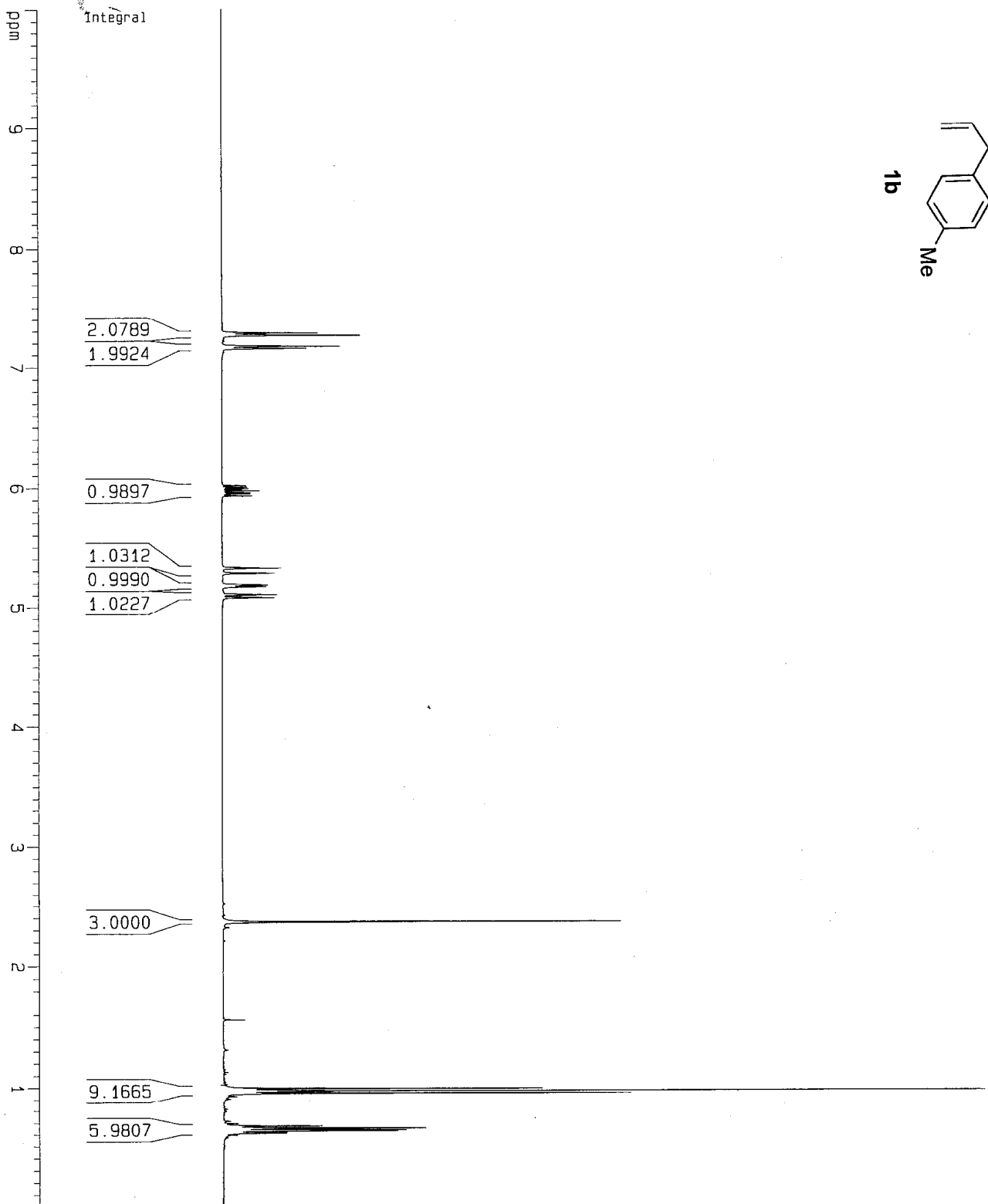
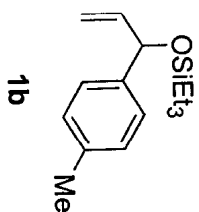
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NUC1 13C
P1 8.50 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== 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
SI 32768
SF 100.6127533 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
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HZCM 1006.12756 Hz/cm

SN050714



Current Data Parameters
NAME SN714-H
EXPNO 1
PROCNO 1

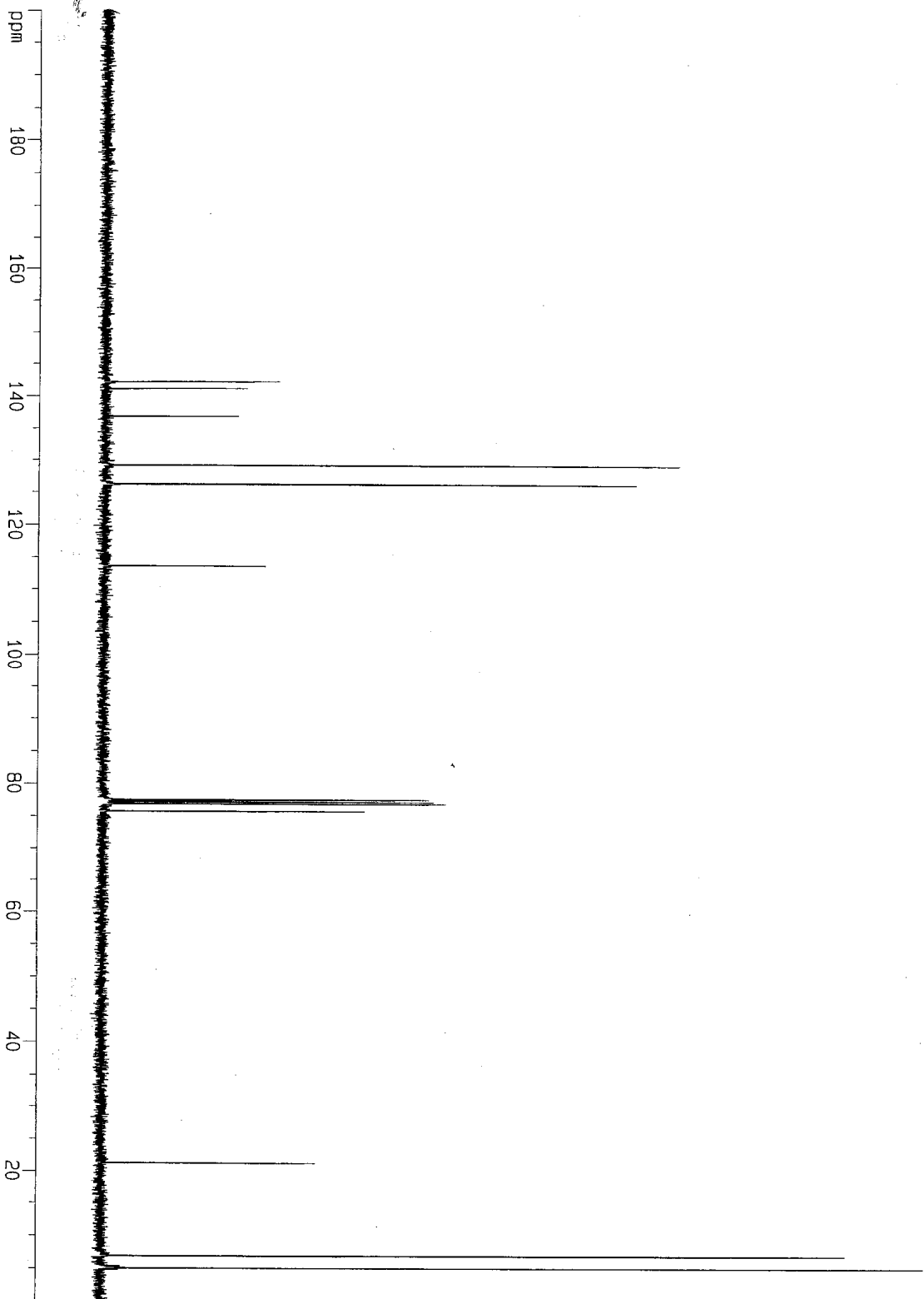
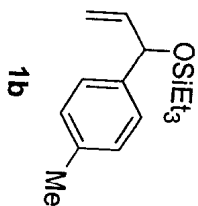
F2 - Acquisition Parameters
Date_ 20050716
Time 21.05
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 32768
SF 400.130051 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

SN050714



Current Data Parameters
NAME SN714-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050716
Time 20.58

INSTRUM spect
PROBHD 5mm BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 109
DS 4

SWH 25125.629 Hz
FIDRES 0.38387 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 0.0002000 sec

===== CHANNEL f1 =====
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P1 15.25 usec
PL1 3.00 dB
SF01 100.623959 MHz

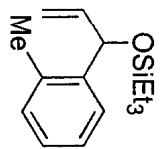
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CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB

PL13 24.00 dB
SF02 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 1006.2744 Hz/cm



SN050715

Current Data Parameters

NAME SN715-H

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050716

Time 21.18

INSTRUM spect

PROBHD 5mm BB0 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 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

SF 400.130054 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

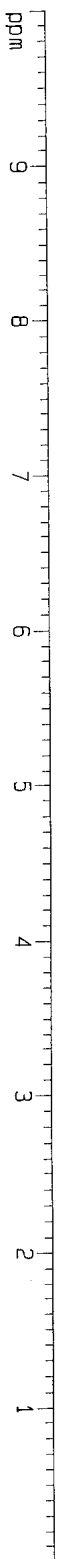
F1 4001.30 Hz

F2P 0.000 ppm

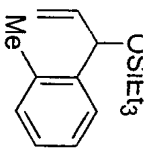
F2 0.00 Hz

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HZCM 200.06500 Hz/cm



Integral



SN050715

Current Data Parameters
 NAME SN715-C
 EXPNO 1
 PROCNO 1

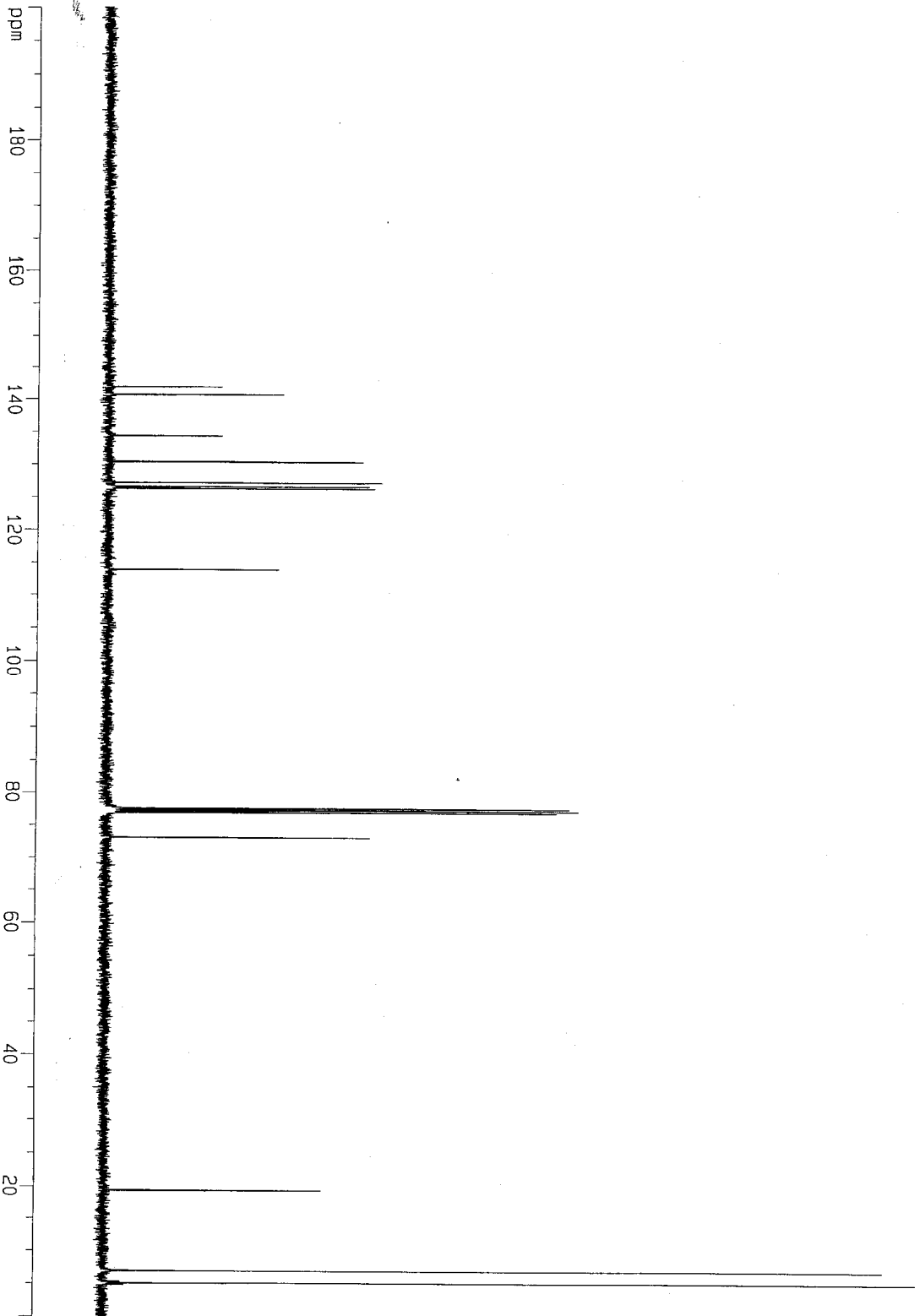
F2 - Acquisition Parameters
 Date_ 20050716
 Time 21.28
 INSTRUM spect
 PROBHD 5mm BB0 BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 297
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.38387 Hz
 AQ 1.3042164 sec
 RG 1290.2
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

===== CHANNEL f1 =====
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 P1 15.25 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

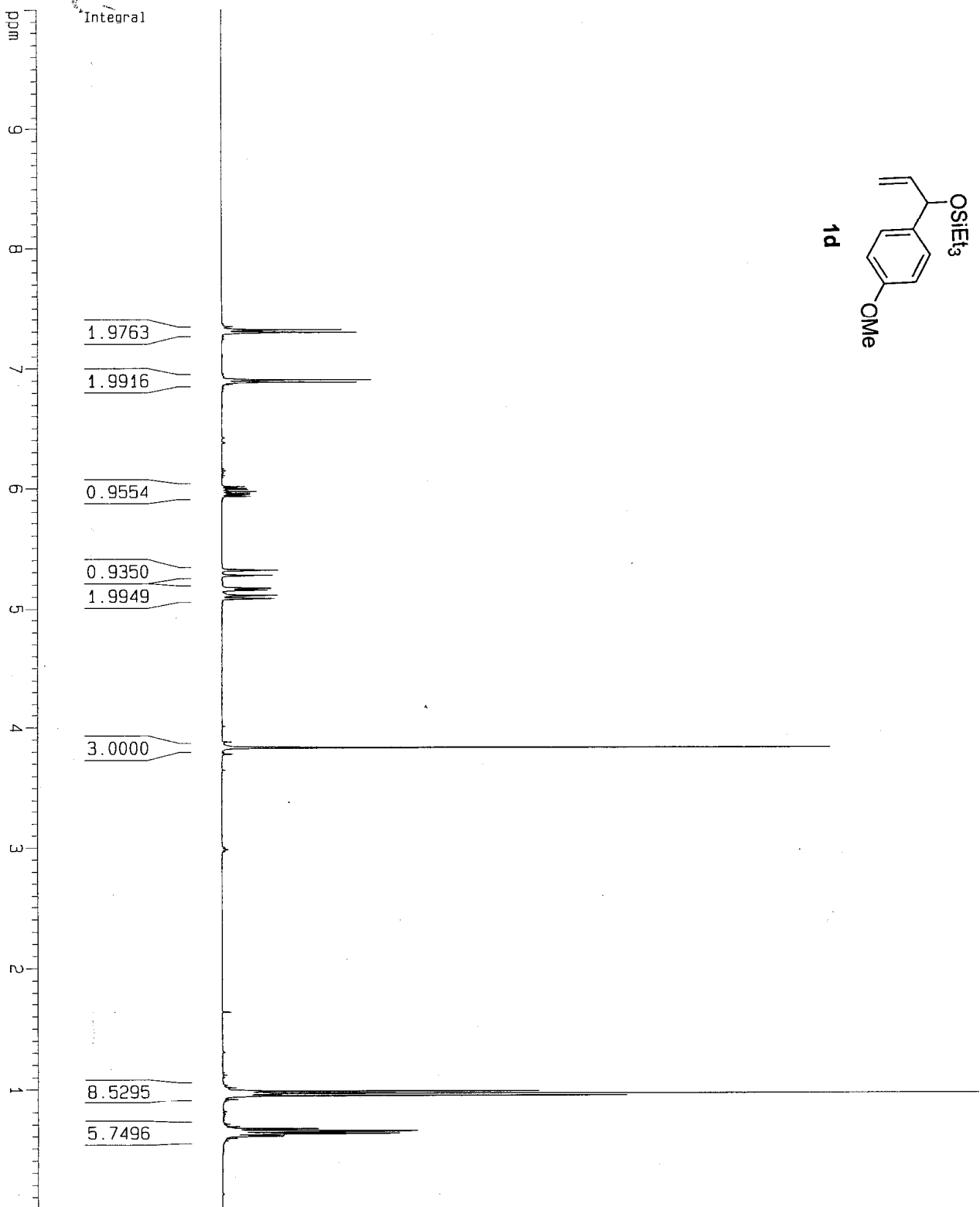
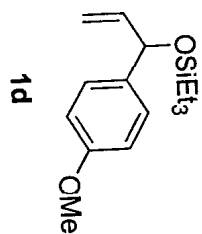
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 NUC2 1H
 PCPD2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing Parameters
 SI 32768
 SF 100.6127476 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
 PPMCK 10.0000 ppm/cm
 HZCM 1006.12744 Hz/cm



SN050729



Current Data Parameters

NAME SN729-H

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050724

Time 19.06

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 5.00 usec

TE 300.0 K

D1 1.00000000 sec

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

NUC1 1H

P1 7.90 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300000 MHz

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

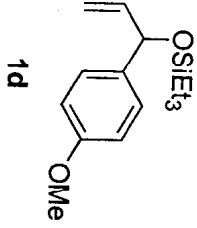
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HZCM 200.06500 Hz/cm

SN050729

ppm

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77.232
76.915
75.447
55.404
7.012
5.076



180
160
140
120
100
80
60
40
20

Current Data Parameters
NAME SN729-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050724
Time 19.26

INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 28
DS 4

SWH 25125.629 Hz
FIDRES 0.38387 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 0.0002000 sec

===== CHANNEL f1 =====
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PL1 3.00 dB
SF01 100.6237959 MHz

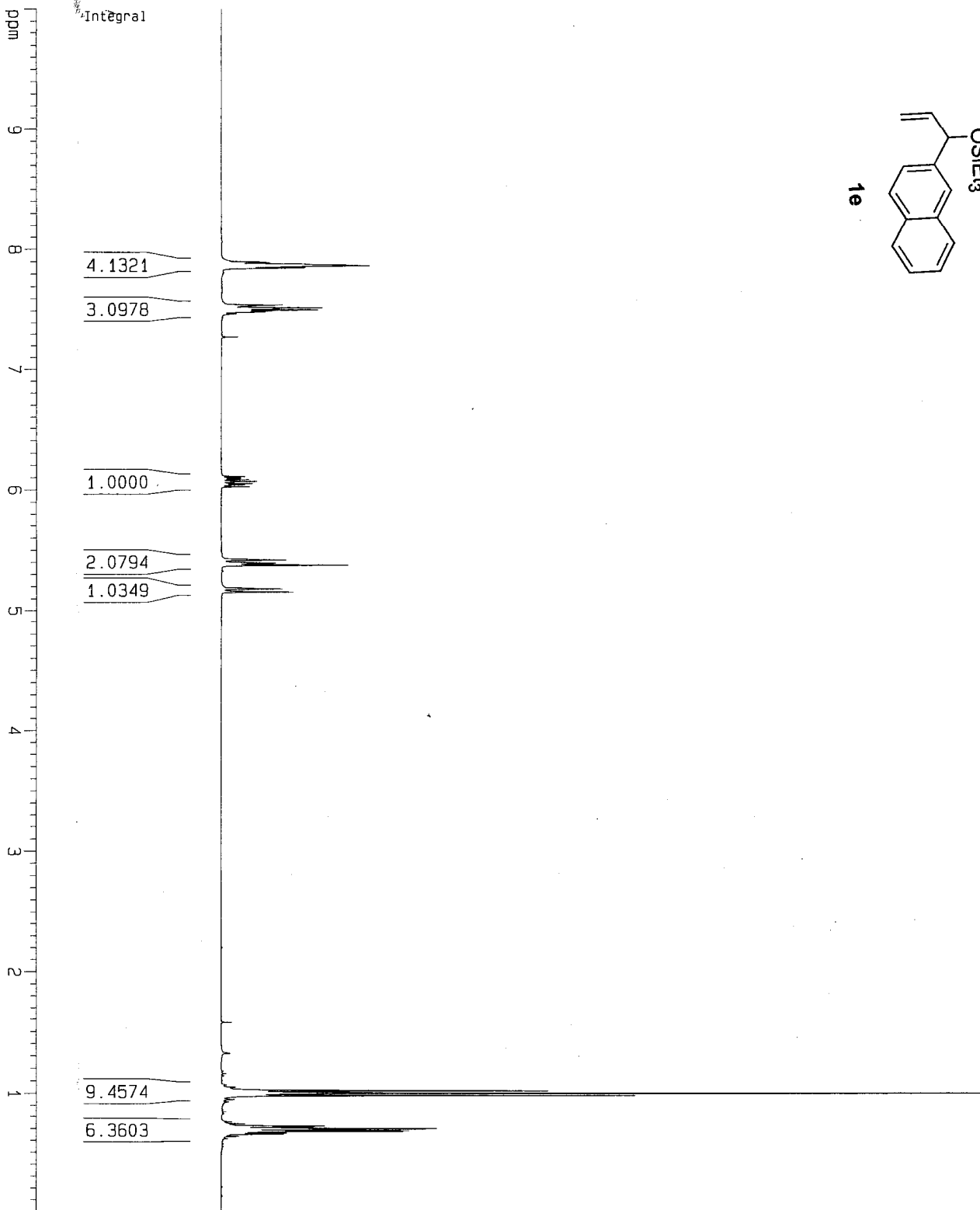
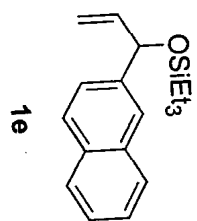
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NUC2 ¹H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing Parameters
SI 32768
SF 100.6127515 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
PPMCH 10.00000 ppm/cm
HZCM 1006.12756 Hz/cm

SN050723



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
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.130056 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

Current Data Parameters
NAME SN723-C

NAME	SN723-C
EXPND	1
PROCND	1

Date_ 20050721

Time	22.21
INSTRUM	spect
PROBD	5mm B80 BB-1
PULPROG	zj9j30
TD	65536
SOLVENT	CDCl3
NS	137

SWH	25,125, 629	Hz
F1DRES	0.393387	Hz
AQ	1.3042164	sec
R6	4096	
DW	19,900	usec
DE	6.00	usec
TE	300.0	K
D1	2.0000000	sec
d11	0.0300000	sec
d12	0.0000200	sec

```
===== CHANNEL f1 =====
NUC1      13C
P1         15.25 usec
PL1        3.00 dB
SF01      100.6237959 MHz
```

```
===== CHANNEL f2 =====
CPDPCG2      waltz16
NUC2          1H
PCPDc        107.50 usec
PL2           0.00 dB
PL12          24.00 dB
PL13          24.00 dB
SF02         400.1316005 MHz
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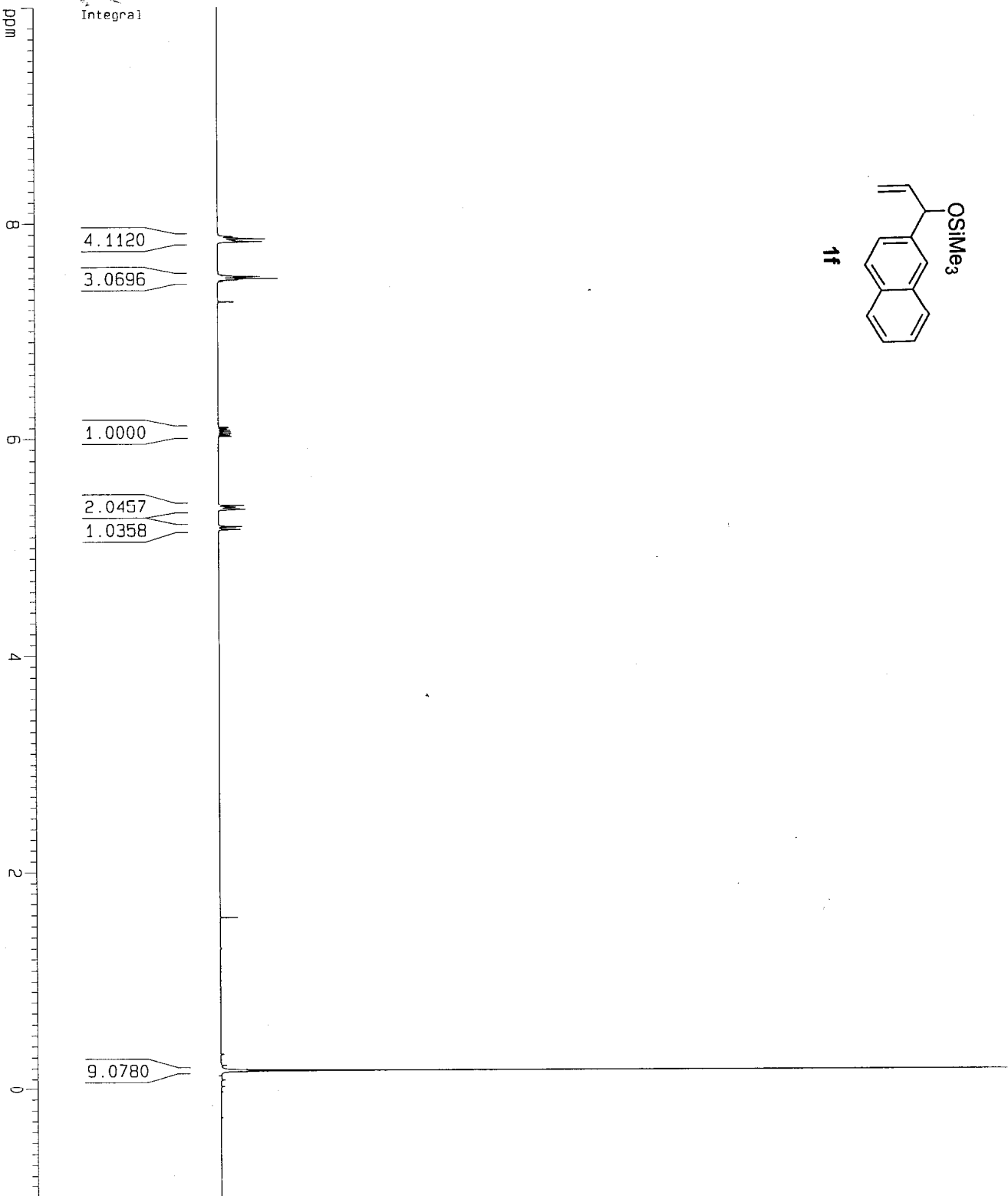
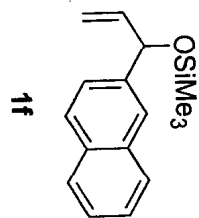
F2 - Processing parameters	
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SF	100.6127538 MHz
MDM	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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



Current Data Parameters
NAME SN740-H
EXPNO 1
PROCNO 1

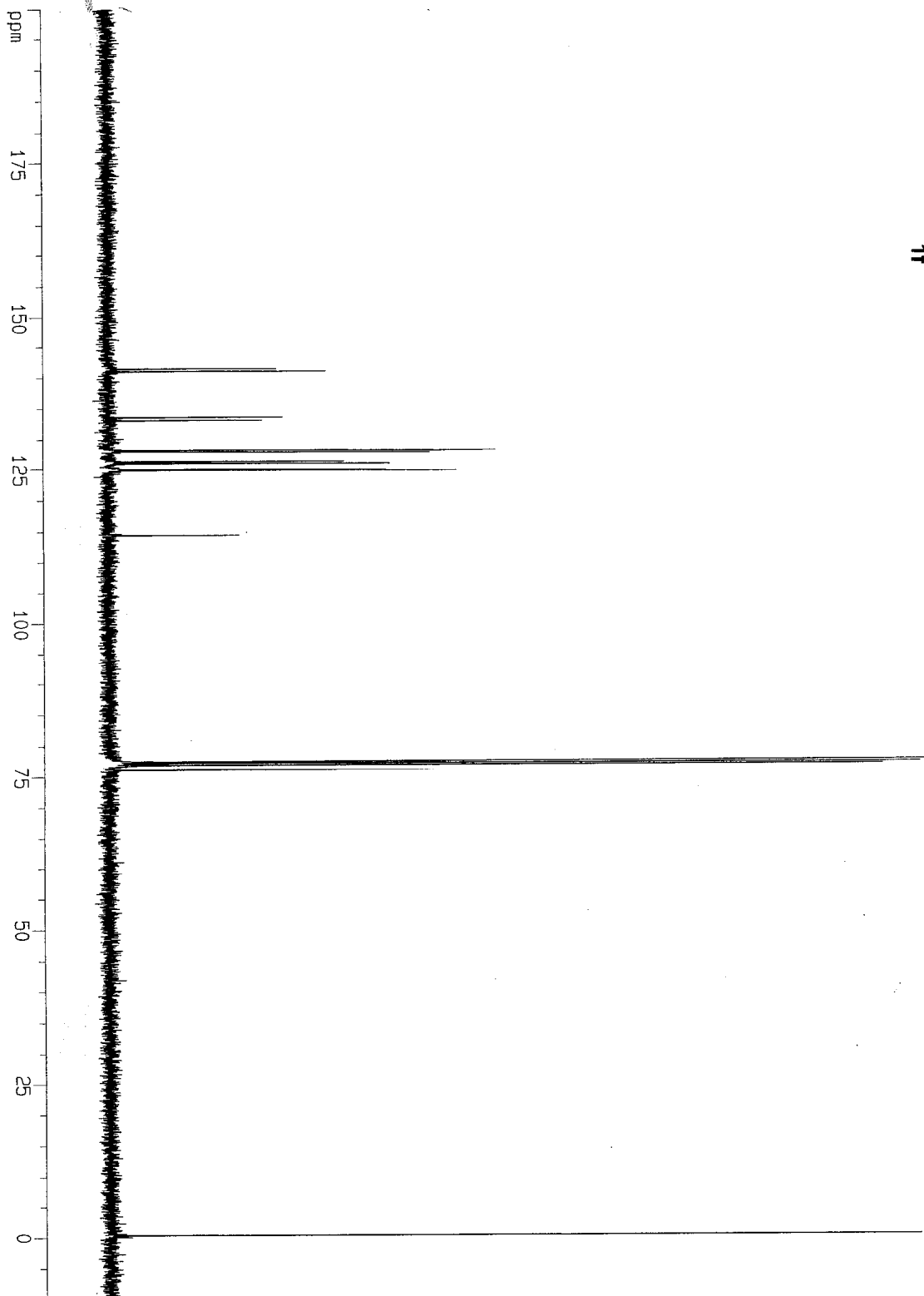
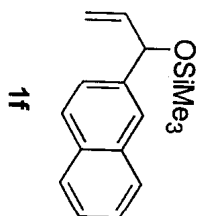
F2 - Acquisition Parameters
Date_ 20050729
Time 22.03
INSTRUM spect
PROBHD 5mm BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 32768
SF 400.130059 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



Current Data Parameters
NAME SN740-C
EXPNO 1
PROCNO 1

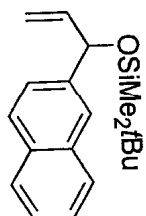
F2 - Acquisition Parameters
Date_ 20050729
Time 22.05
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 201
DS 4
SWH 25125.629 Hz
FIDRES 0.33387 Hz
AQ 1.3042164 sec
RG 16384
DM 13.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== 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 100.6127522 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 1056.4396 Hz/cm



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SN050737

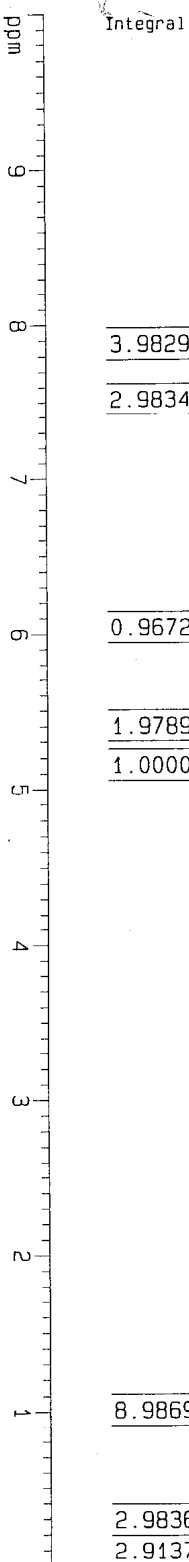
Current Data Parameters
NAME SN737-H
EXPNO 1
PROCNO 1

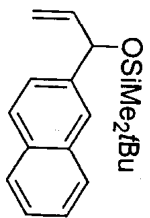
F2 - Acquisition Parameters
Date_ 20050727
Time 22.18
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 0.00 dB
SF01 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





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
 SMH 25125.629 Hz
 FIDRES 0.38387 Hz
 AQ 1.3042164 sec
 RG 3251
 DM 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

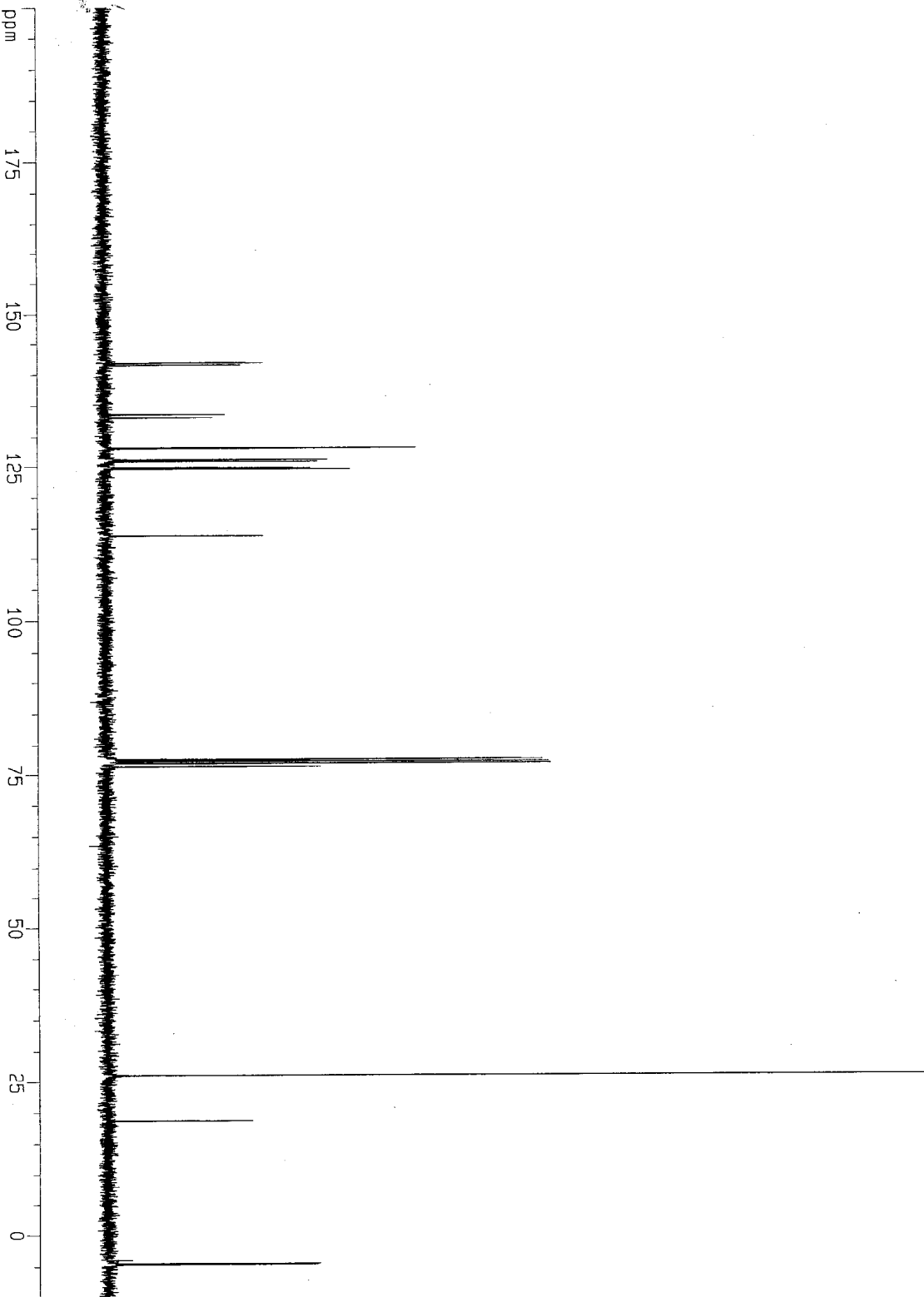
===== CHANNEL f1 =====
 NUC1 13C
 P1 15.25 usec
 PL1 3.00 dB
 SF01 100.6237959 MHz

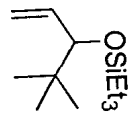
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 107.50 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1316005 MHz

F2 - Processing Parameters
 SI 32768
 SF 100.6127530 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 1056.43395 Hz/cm





1h

SN050733

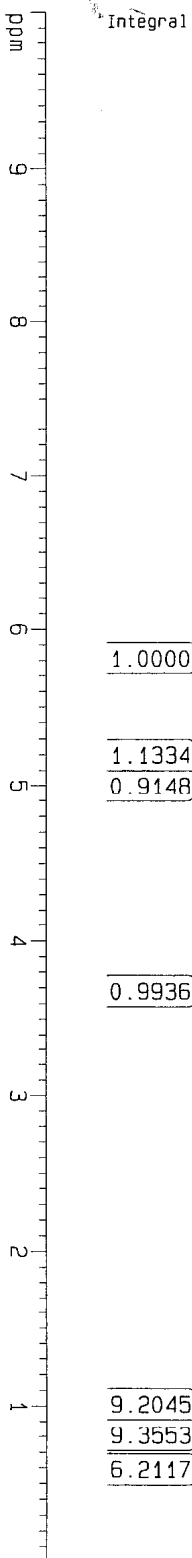
Current Data Parameters
NAME SN733-H
EXPNO 1
PROCNO 1

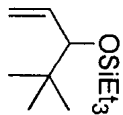
F2 - Acquisition Parameters
Date_ 20050727
Time 22.00
INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 32768
SF 400.130056 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
PPMCW 0.50000 ppm/cm
HZCM 200.06500 Hz/cm





SN050733

Current Data Parameters
NAME SN733-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050727
Time 22.05

INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 132
DS 4
SWH 25125.629 Hz
FIDRES 0.38387 Hz
AQ 1.3642164 sec
RG 4096
DM 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03600000 sec
d12 0.0002000 sec

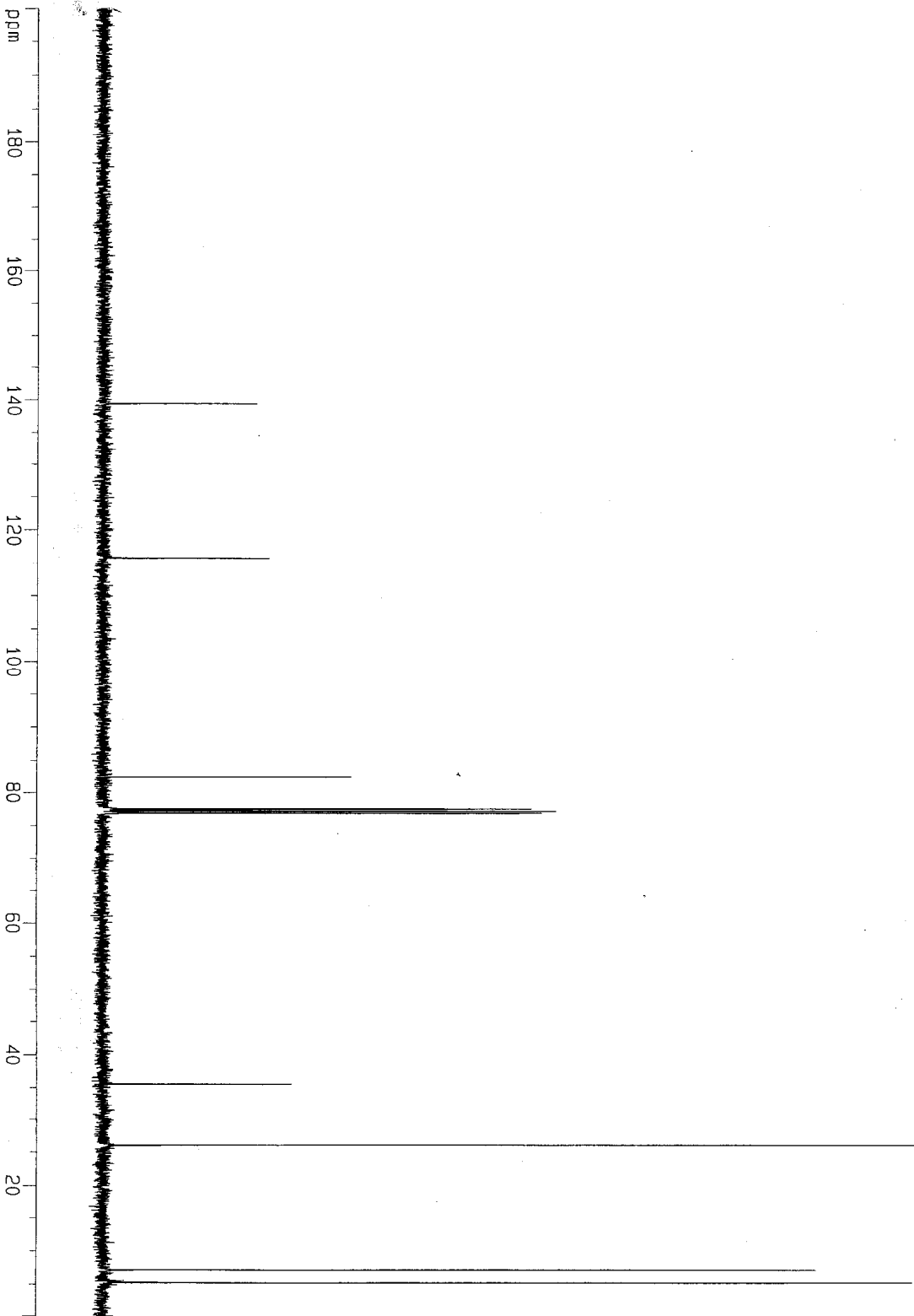
===== CHANNEL f1 =====
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

===== 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

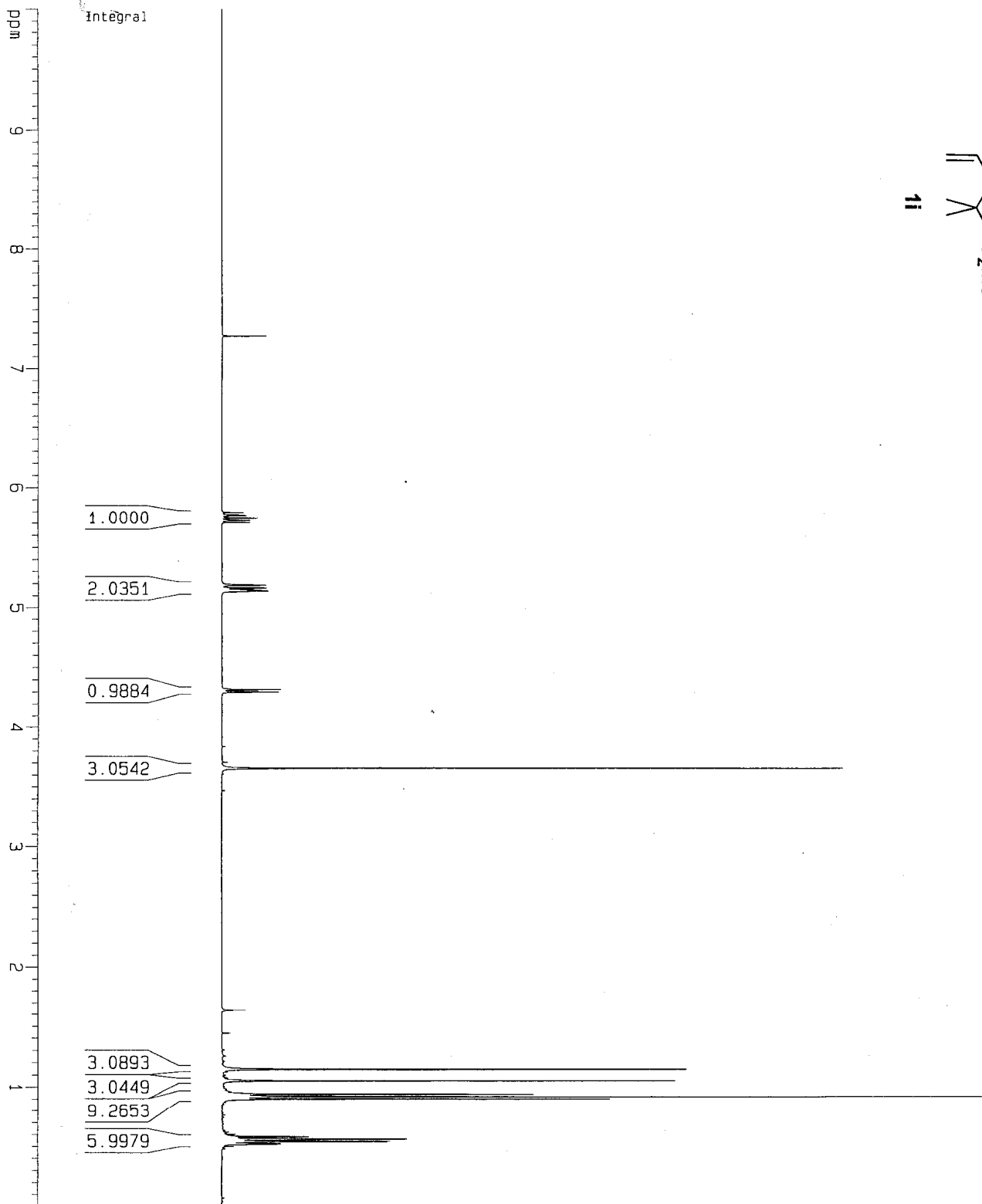
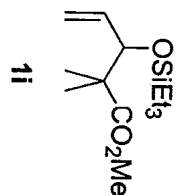
F2 - Processing parameters
SI 32768
SF 100.6127469 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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



Current Data Parameters
 NAME SN745-H
 EXPNO 1
 PROCNO 1

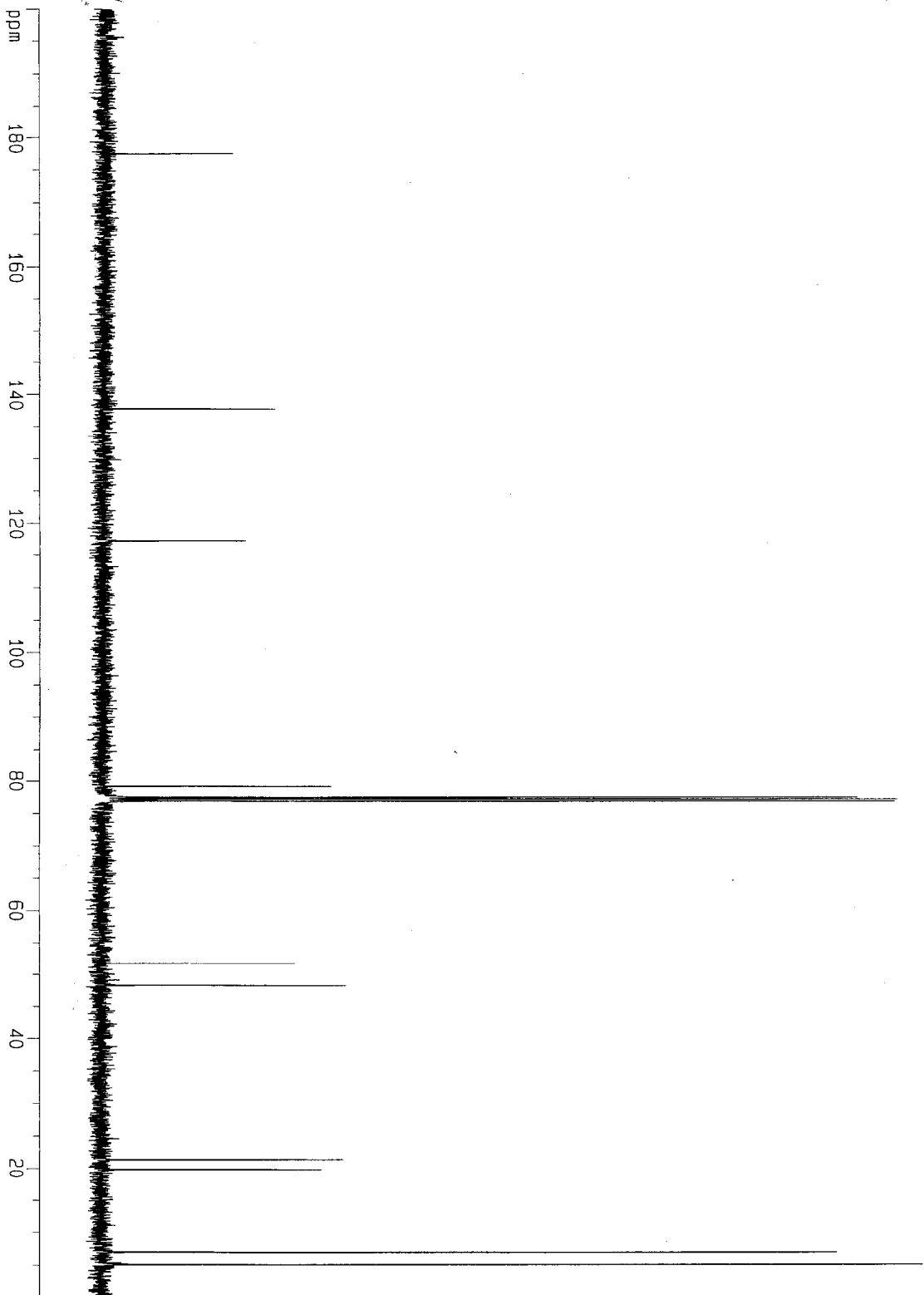
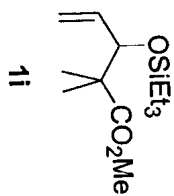
F2 - Acquisition Parameters
 Date_ 20050801
 Time 5.13
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 64
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

F2 - Processing parameters
 SI 32768
 SF 400.130059 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



Current Data Parameters
NAME SN745-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050801
Time 5.22

INSTRUM spect
PROBHD 5mm BBO 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 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SF01 100.6237959 MHz

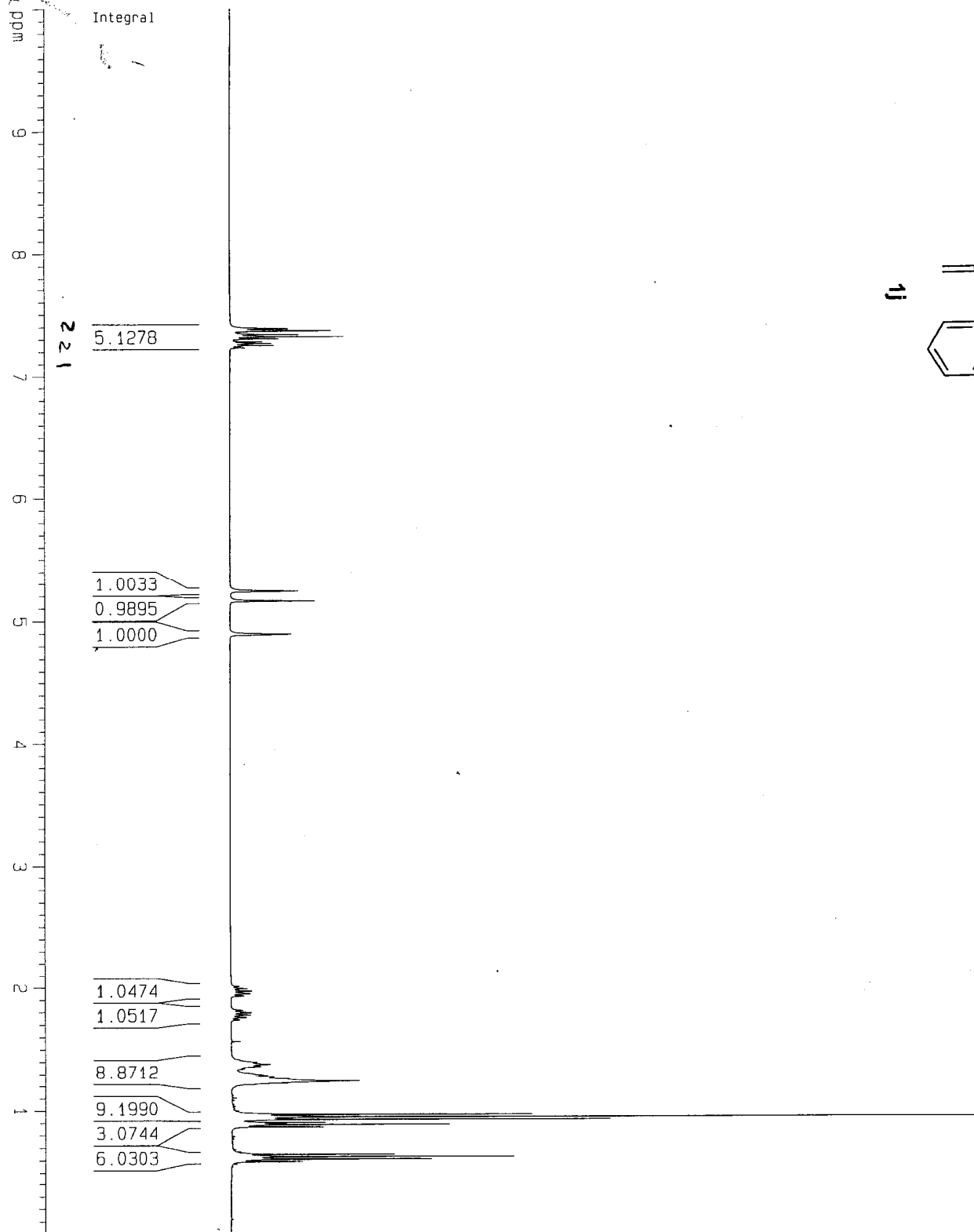
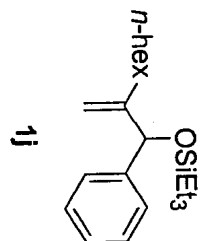
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.5127492 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

SN050658 allylic alcohol



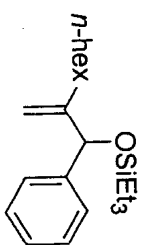
Current Data Parameters
 NAME SN658neck-c
 EXPNO 1
 PROCNO 1

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
 DM 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
 SF01 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



octene benzaldehyde TESOTf

Current Data Parameters

NAME octene-a1-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

SMH 25125.629 Hz

FIDRES 0.38387 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 0.0002000 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

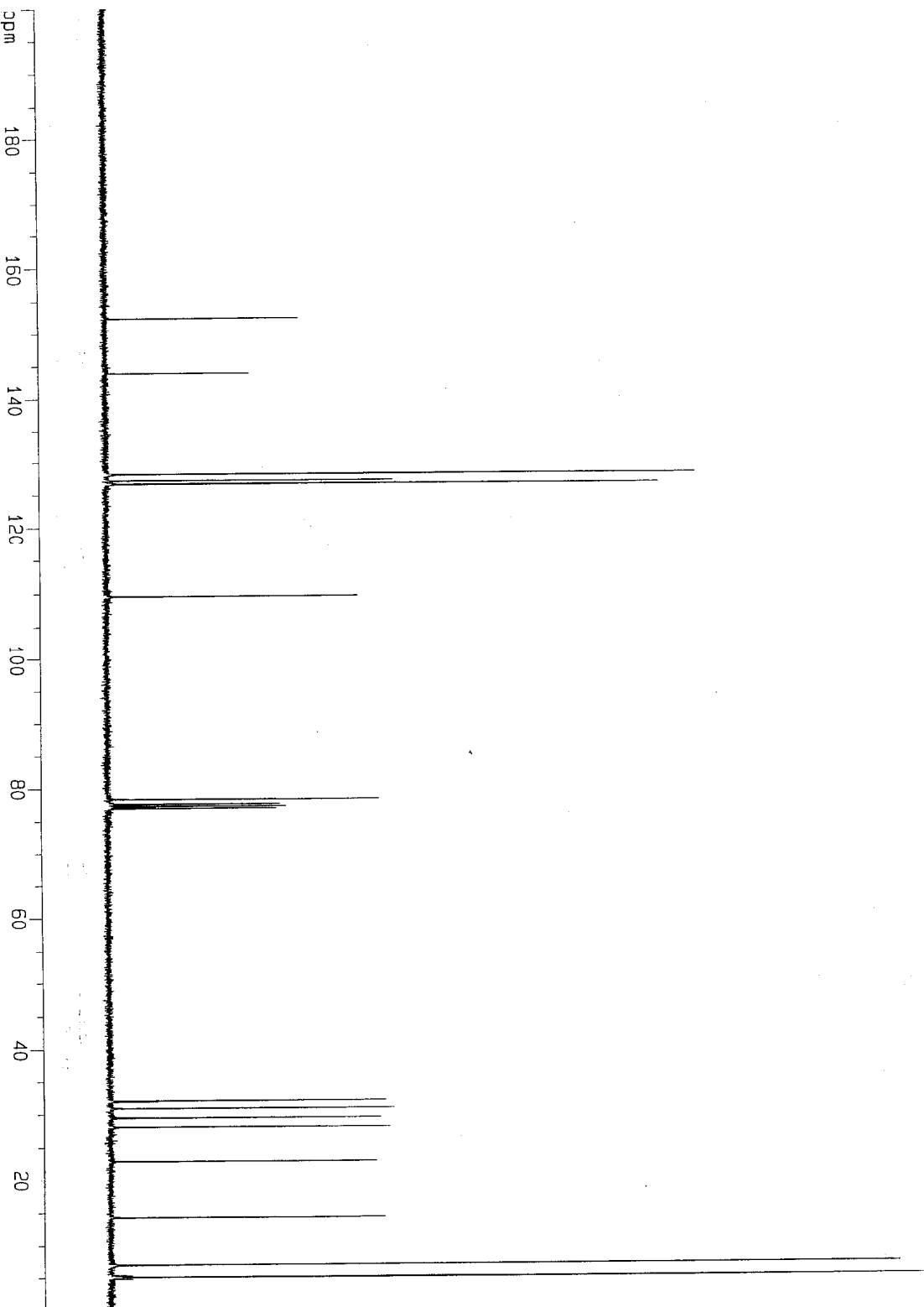
F1 20122.55 Hz

F2P 0.000 ppm

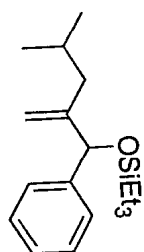
F2 0.00 Hz

PPMCM :0.00000 ppm/cm

HZCM 1006.12744 Hz/cm



SN050726 allylic alcohol



1k

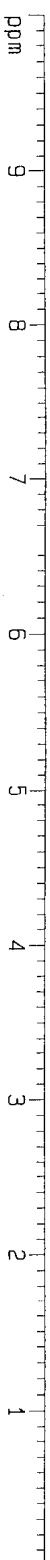
Current Data Parameters
NAME SN726-allyl-H
EXPNO 1
PROCNO 1

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
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

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

F2 - Processing parameters
SI 32768
SF 400.130056 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



Integral

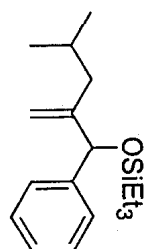
4.0952
1.0923

1.0044
1.0000
1.0020

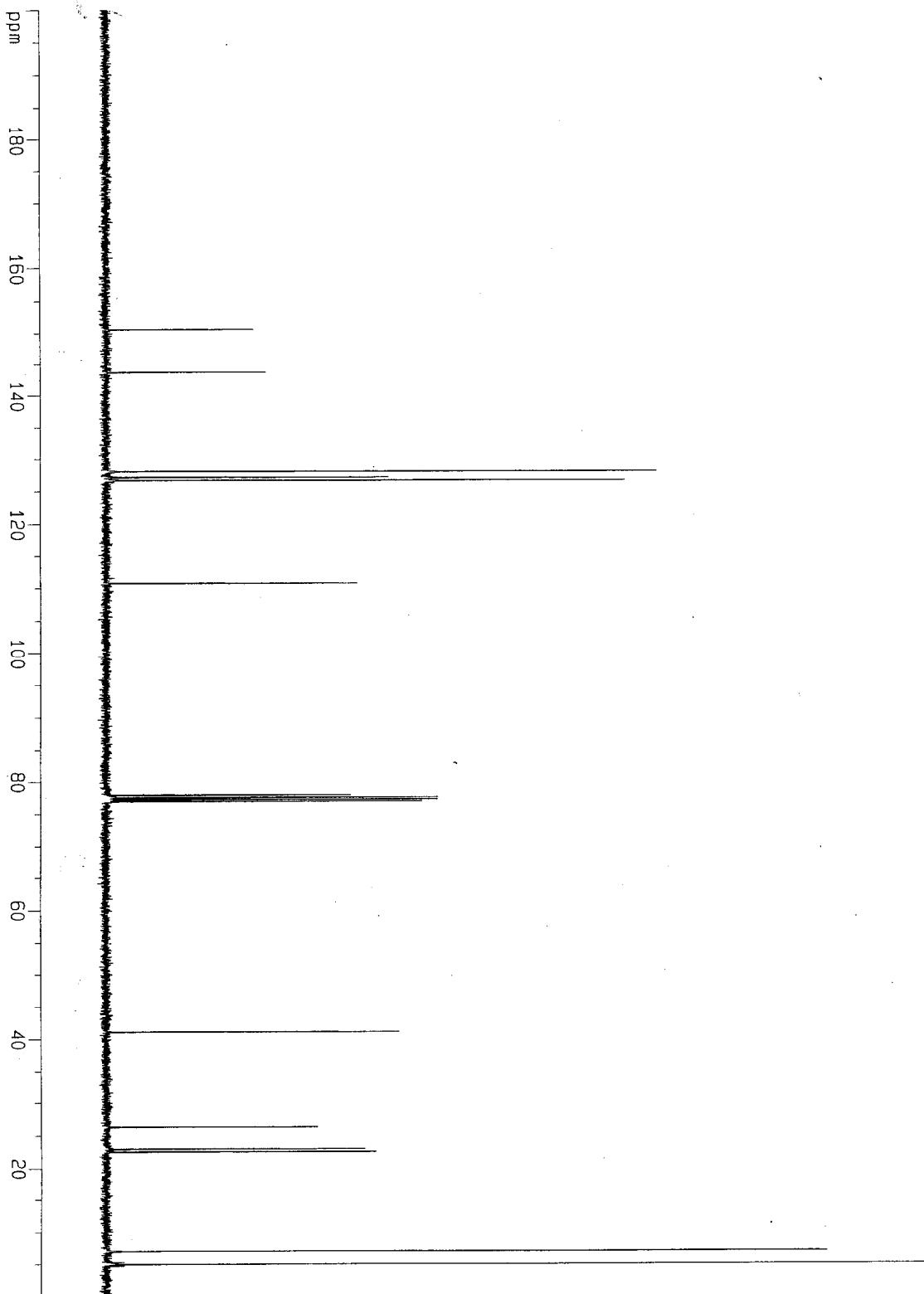
3.1640

9.3466
6.2945
6.1869

SN050726 allylic alcohol



1k



Current Data Parameters
NAME SN726-ajj-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 0.38387 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

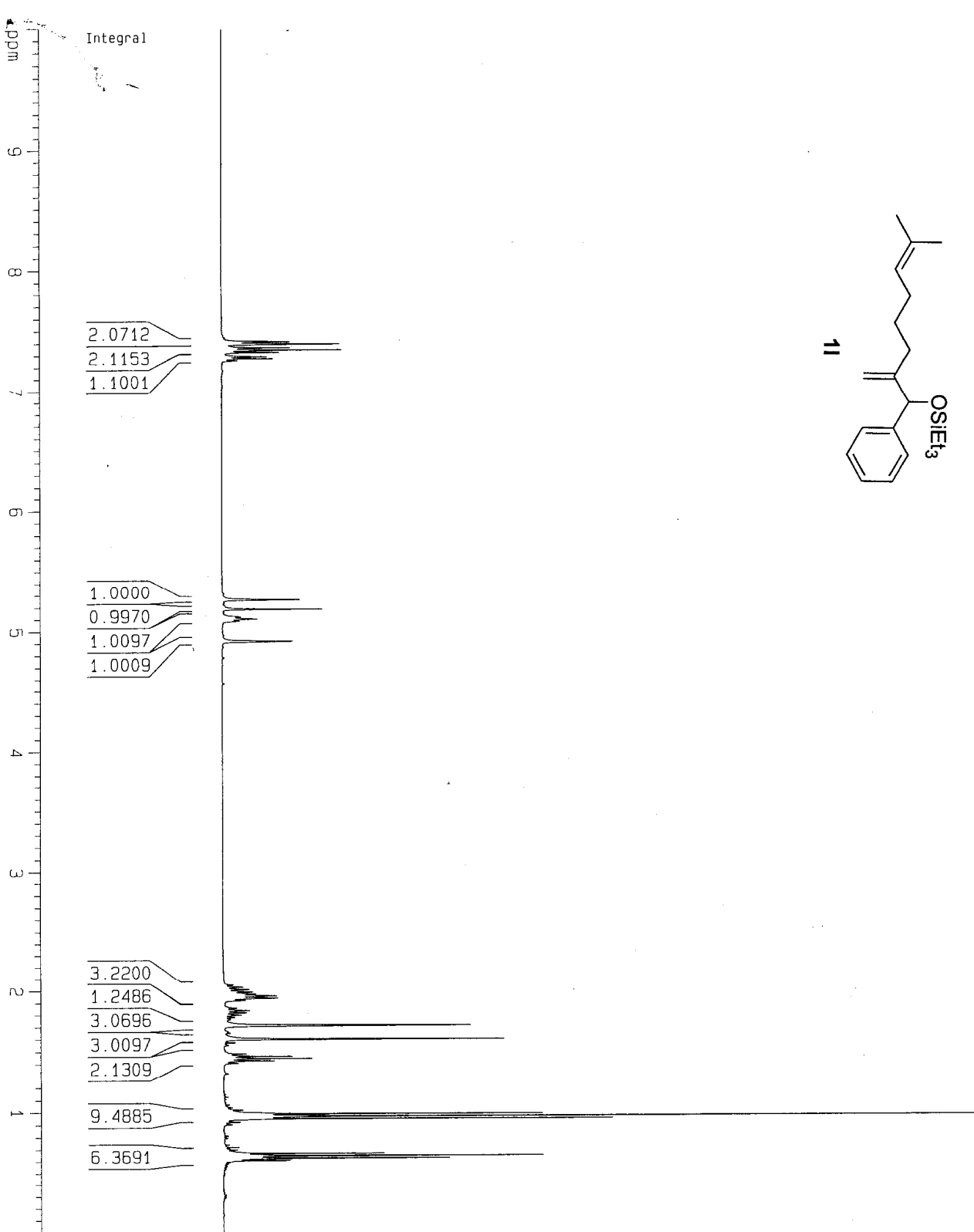
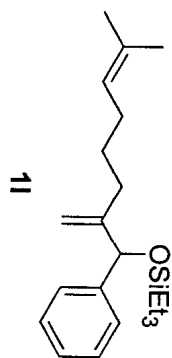
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.50 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz

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
PPKCM 10.00000 ppm/cm
HZCM 1006.12744 Hz/cm

SN050672 a



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

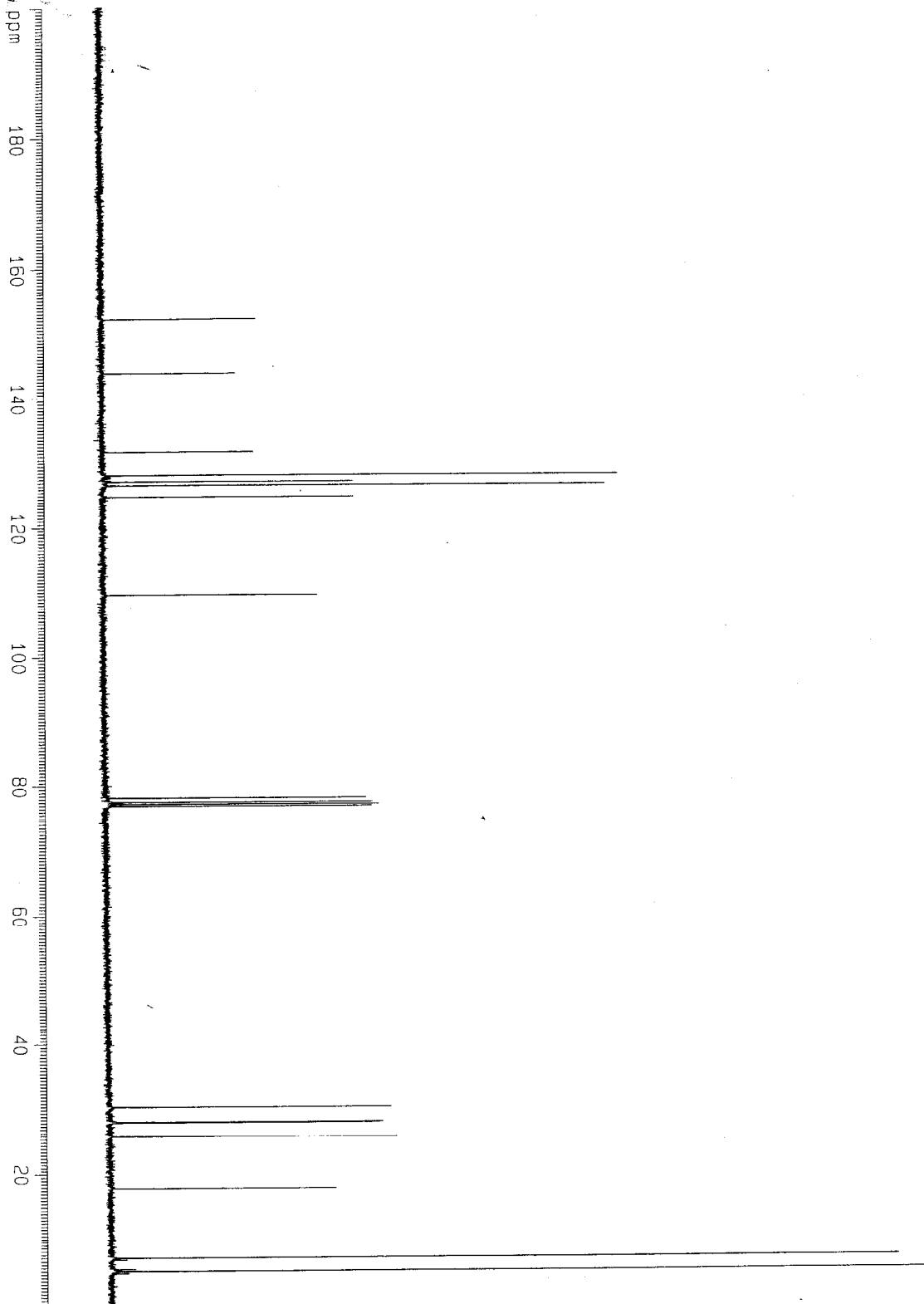
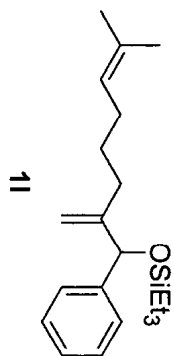
F2 - Acquisition Parameters
 Date_ 20050625
 Time 16.55
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 32
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 2.00 dB
 SF01 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



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

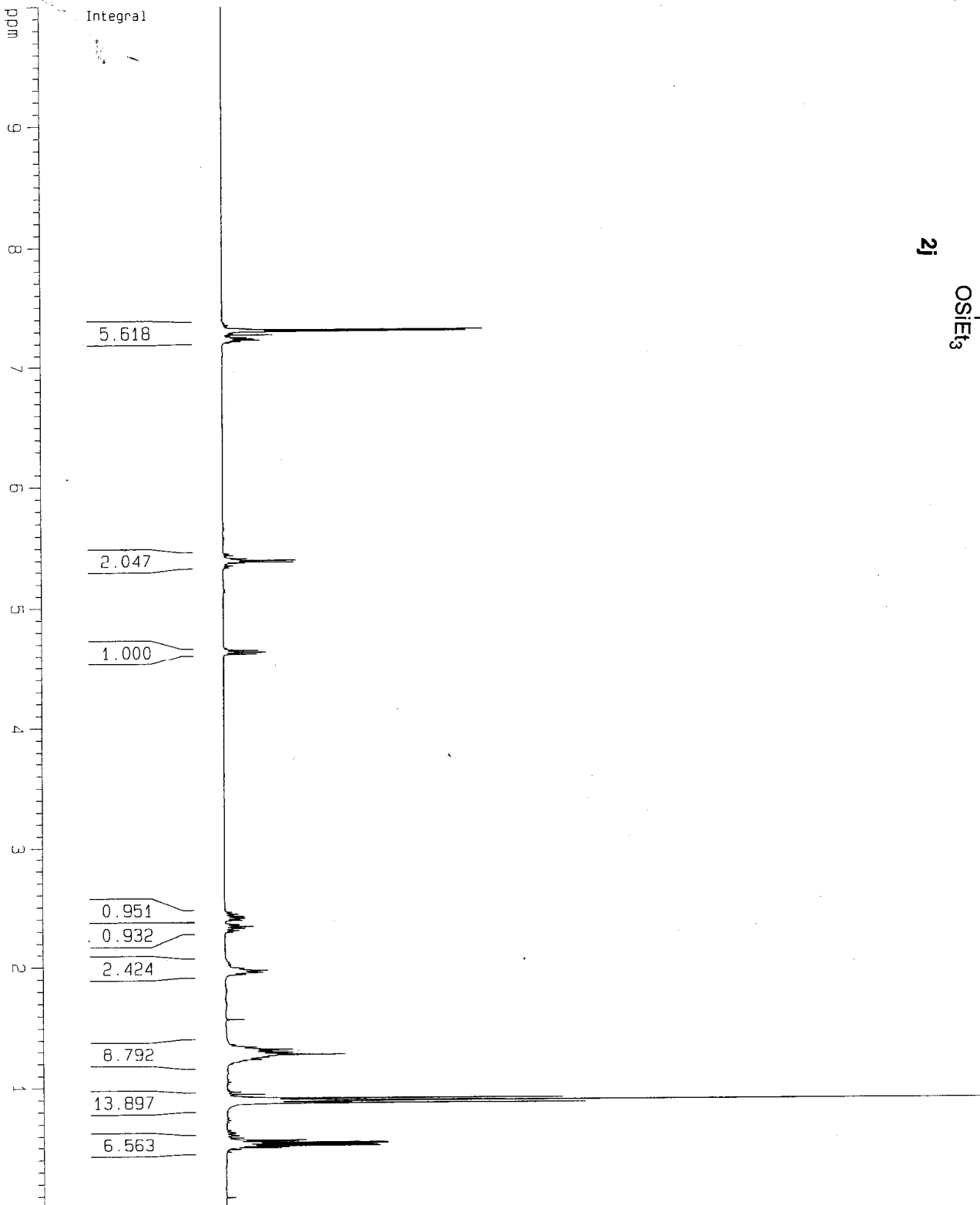
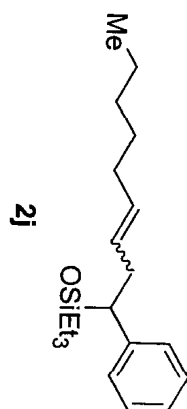
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 3.00 dB
SF01 100.6237959 MHz

===== 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
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

SN050658 ene product



Current Data Parameters

NAME	SN568ene-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050614
Time	19.59
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	128
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

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

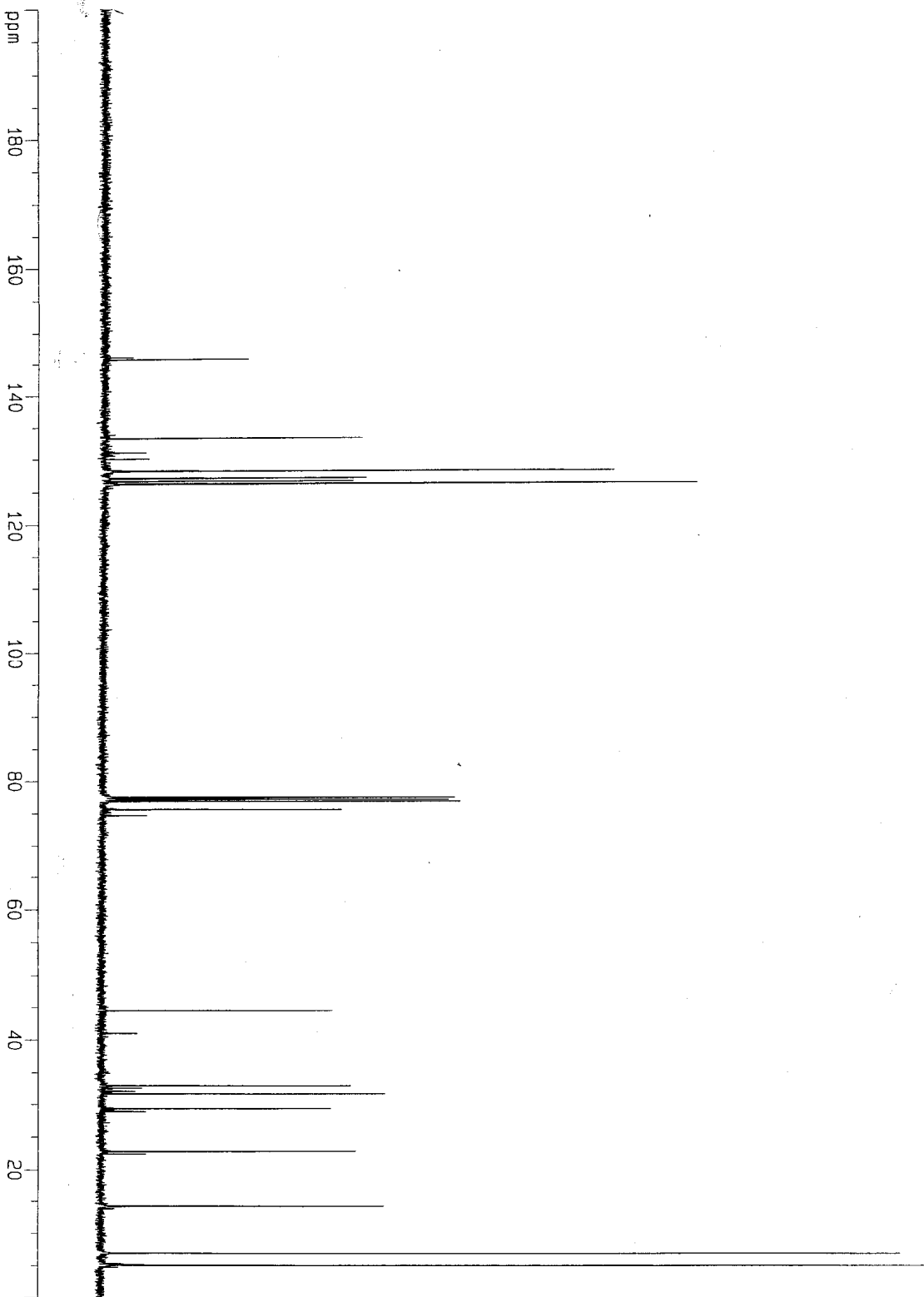
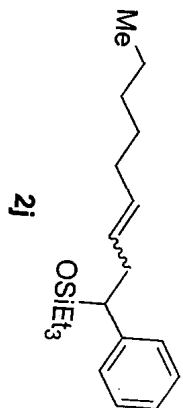
F2 - Processing parameters

SI	32768
SF	400.130059 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



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

F2 - Acquisition Parameters
Date_ 20050716
Time 22.11

INSTRUM spect
PROBHD 5mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 213
DS 4

SWH 25125.629 Hz
FIDRES 0.38387 Hz
AQ 1.3042164 sec

RG 1149.4
DM 19.900 usec
DE 6.00 usec

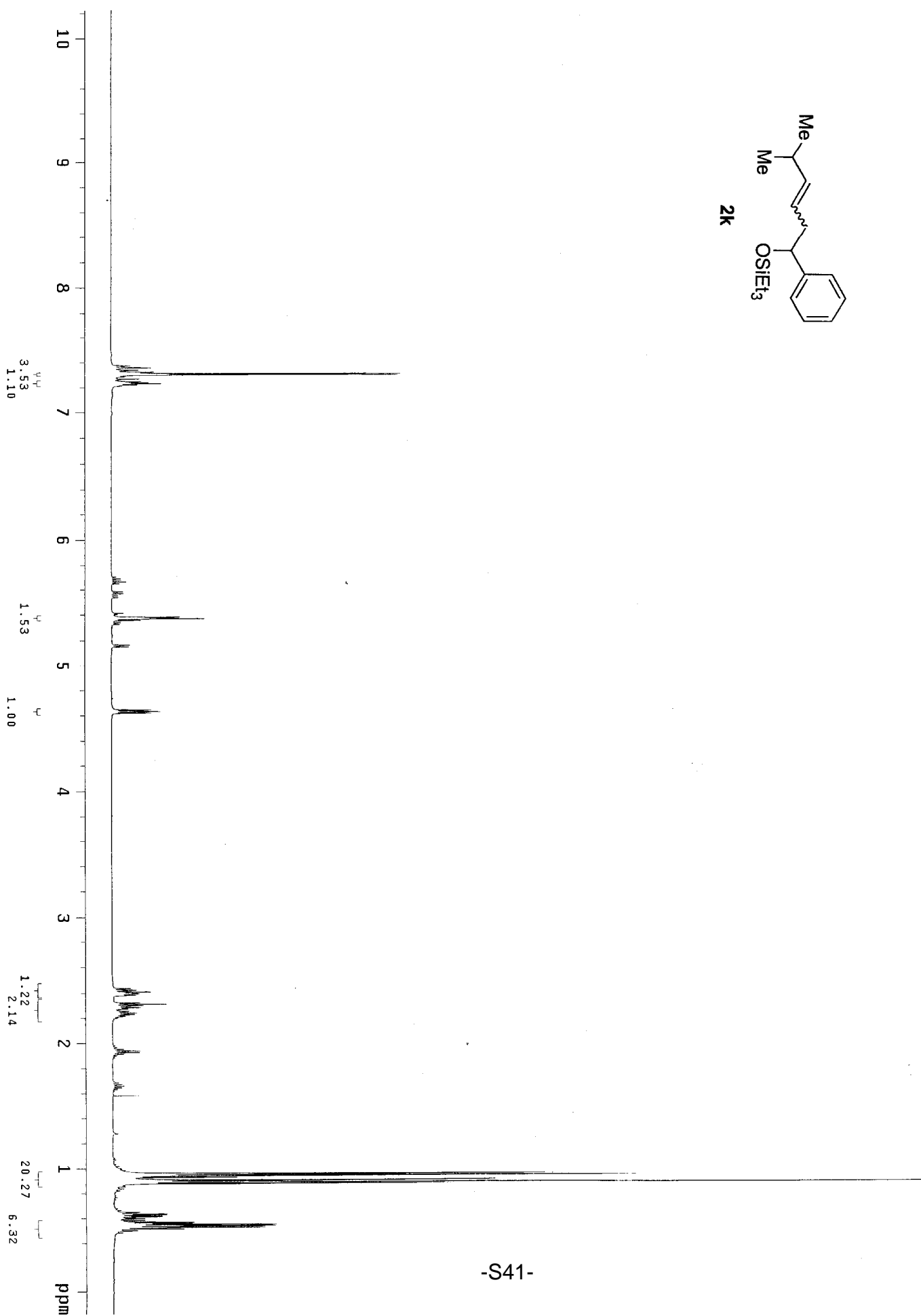
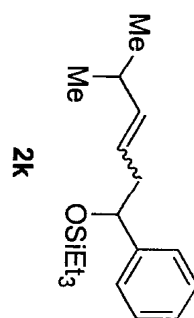
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

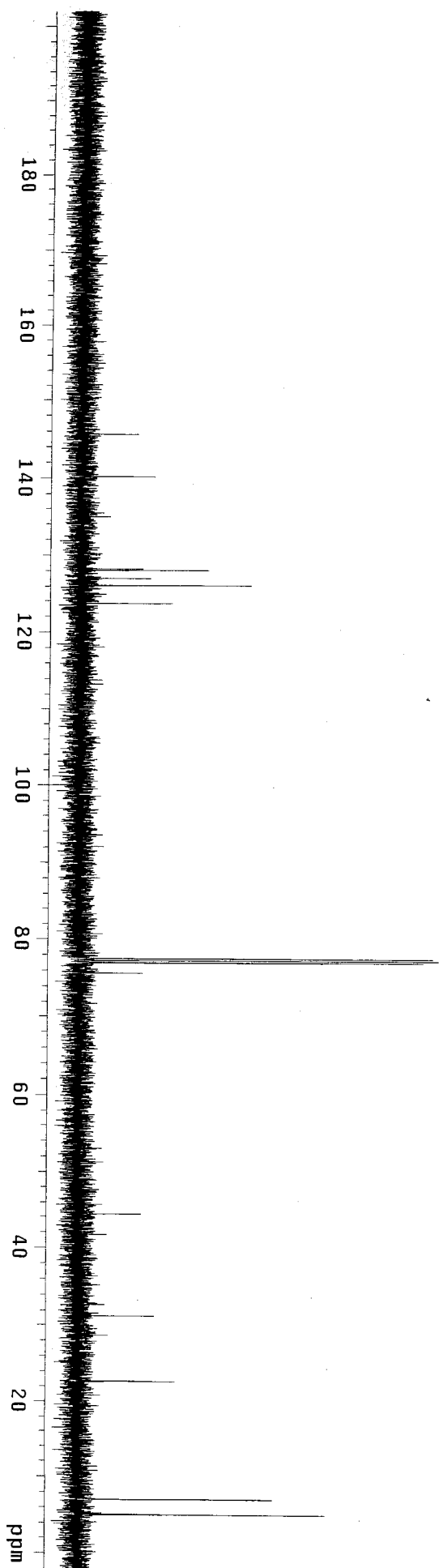
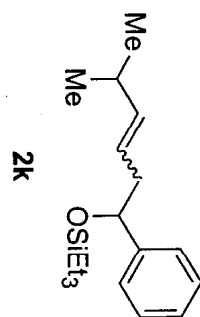
===== CHANNEL f1 =====
NUC1 13C
P1 15.25 usec
PL1 3.00 dB
SFO1 100.6237959 MHz

===== 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

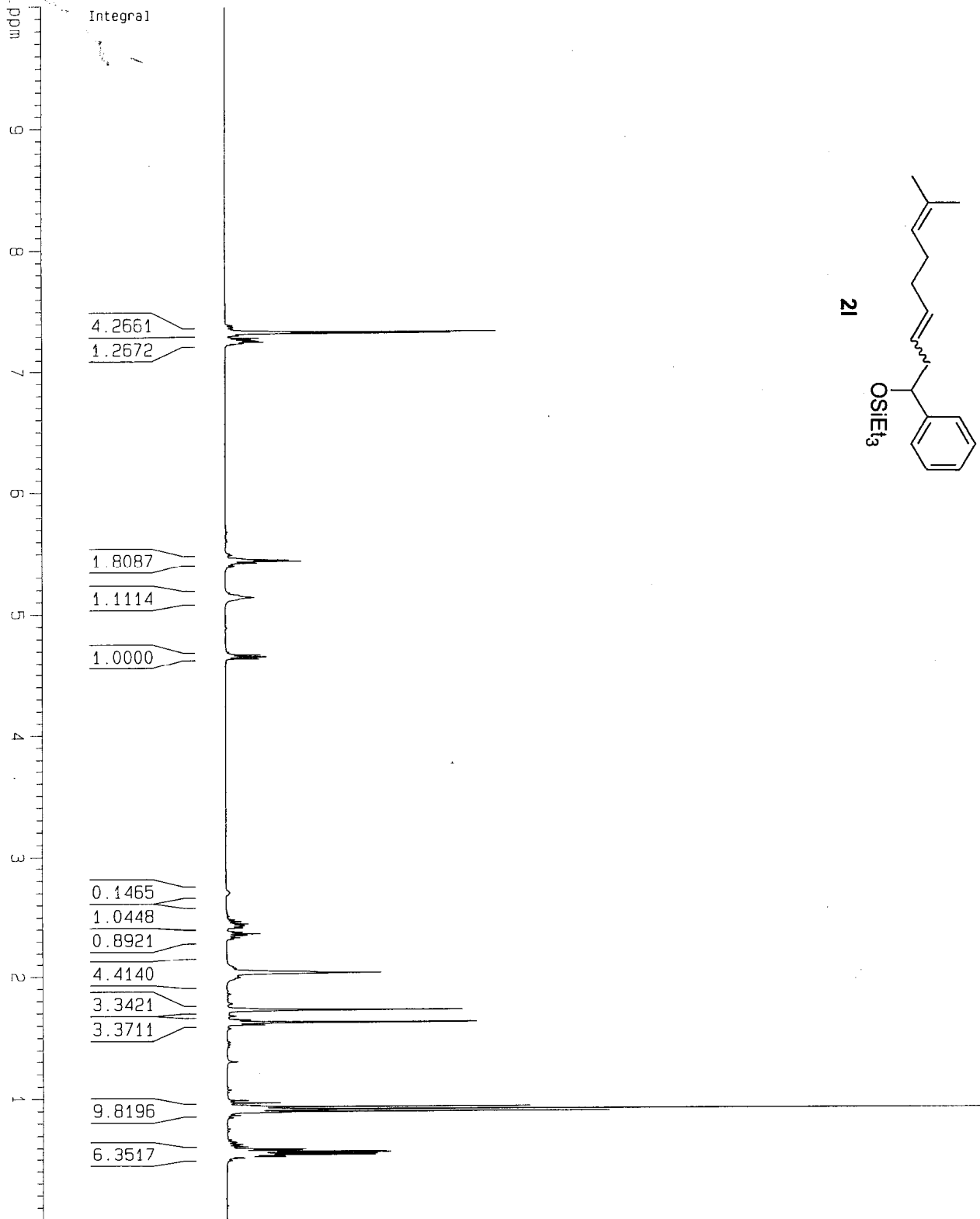
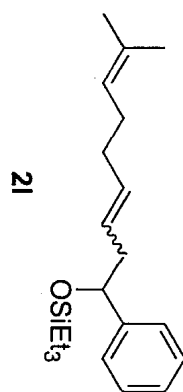
F2 - Processing parameters
SI 32768
SF 100.6127476 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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





SN050672-b



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

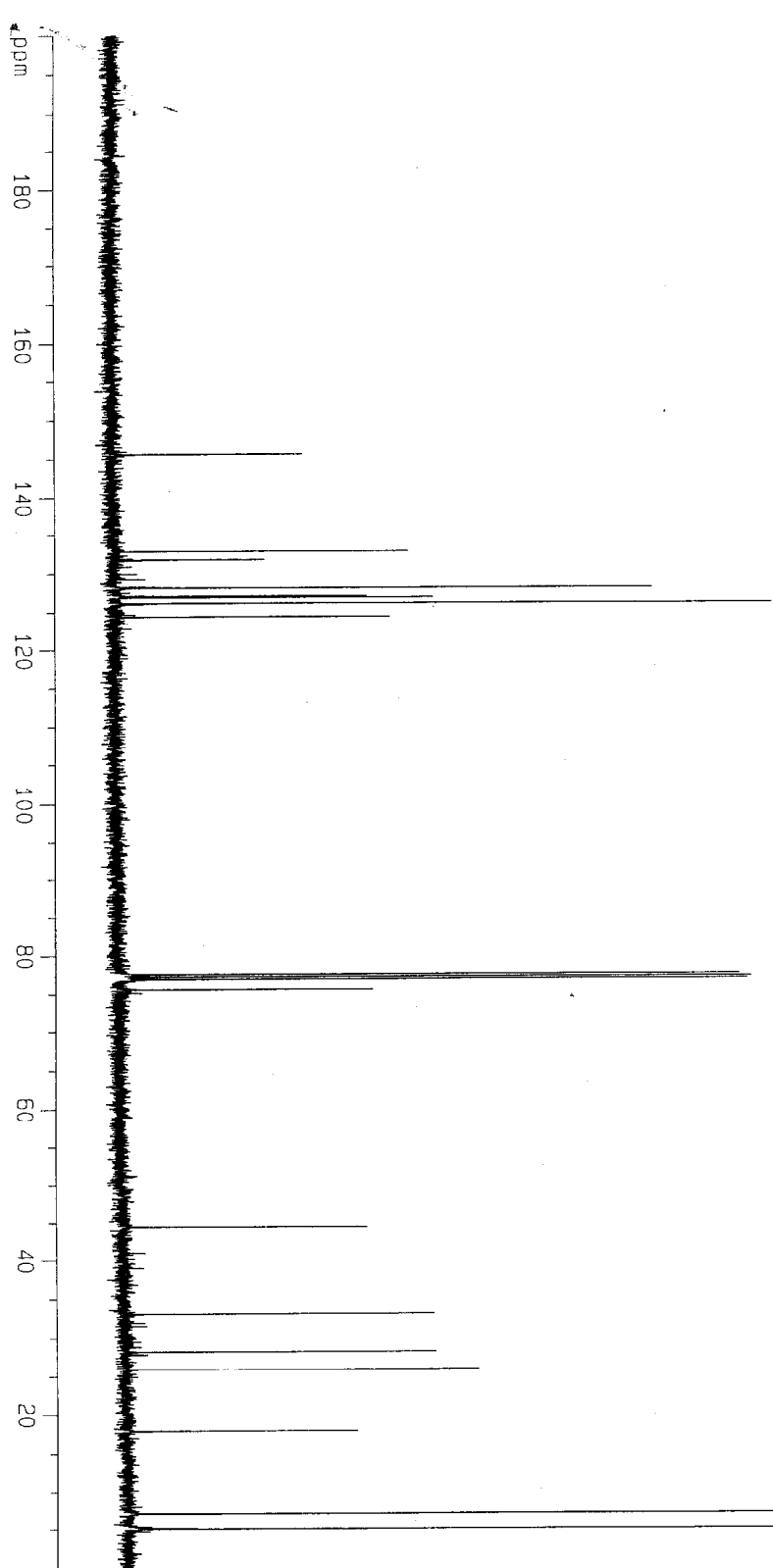
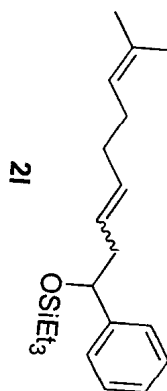
F2 - Acquisition Parameters
Date_ 20050625
Time 18.50
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 34
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 32768
SF 400.130000 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



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
SMH 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 0.0002000 sec

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

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

SI 32768
SF 100.6127503 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR p10: parameters

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