

**Supporting Information for *Org. Lett.*****Stereospecific Synthesis of Highly Substituted Skipped Dienes  
through Multifunctional Palladium Catalysis**

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5735 South Ellis Avenue, Chicago, IL 60637***EXPERIMENTAL PROCEDURES and CHARACTERIZATION DATA**

**General.** All alkynes, allyl and methallylbromide and allyl chloride were distilled prior to use. Dimethoxyethane and THF were distilled over CaH<sub>2</sub> and sodium/benzophenone respectively. All other reagents were used as received (Aldrich, Acros, Strem). Melting points are uncorrected and were measured on a Fisher-Johns melting point apparatus. <sup>1</sup>H and <sup>13</sup>C NMR were recorded at 400 or 500 MHz and 100 or 125 MHz respectively on a Bruker DRX-400 or 500 spectrometer. Proton chemical shifts were internally referenced to the residual proton resonance in CDCl<sub>3</sub> (δ 7.26). Carbon chemical shifts were internally referenced to the deuterated solvent signals in CDCl<sub>3</sub> (δ 77.00). Infrared spectra were obtained on a Nicolet 20 SXB FT-IR spectrometer. Silica gel (60 Å, 230-400 mesh) was obtained from Silicycle and used as received.

**General Procedure for Bromoallylation of Alkynes (Table 1)**

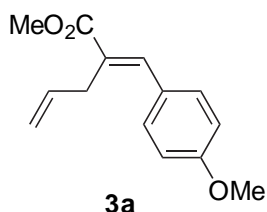
To a solution of allylbromide (173 μL, 2.00 mmol) in anhydrous DME (5.0 mL) cooled in an ice-bath was added dibromo(bisbenzonitrile)palladium(II) (28.3 mg, 0.06 mmol). A solution of the alkyne (2.00 mmol) in anhydrous DME (5.0 mL) was then added dropwise over *ca.* 30 min. The reaction mixture was stirred for another 5.5 h at rt. The solvent was removed *in vacuo* and the residue subjected to silica gel chromatography (EtOAc/hexanes). Compounds **1a-d** have been previously described in the literature.<sup>1,2</sup>

**General Procedure for One-pot Tandem Bromoallylation/Suzuki Cross-Coupling of Alkynes (Table 2)**

To a solution of allylbromide (173 μL, 2.00 mmol) in anhydrous DME (5.0 mL) cooled in an ice-bath was added dibromo(bisbenzonitrile)palladium(II) (28.3 mg, 0.06 mmol). A solution of the

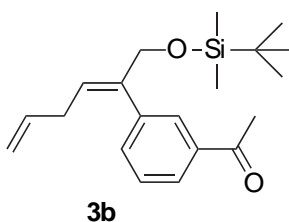
alkyne (2.00 mmol) in anhydrous DME (5.0 mL) was then added dropwise over *ca.* 30 min. The reaction mixture was stirred for another 5.5 h at rt. Cesium carbonate (1.30 g, 4.00 mmol) and the boronic acid (4.00 mmol) were then added and the reaction mixture vigorously stirred at 75 – 80°C (bath temperature) for 16 h. The solvents were subsequently removed *in vacuo* and the residue diluted with EtOAc/hexanes (1:4) (100 ml). The solids were filtered off and the filtrate concentrated *in vacuo*. The resulting residue was then subjected to flash silica gel chromatography using EtOAc/hexanes.

### Methyl 2-(4-Methoxybenzylidene)pent-4-enoate (**3a**)

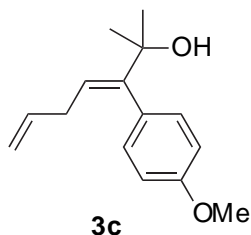


**3a** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.19 (2H, d,  $J$  = 8.5 Hz), 6.81 (2H, d,  $J$  = 8.5 Hz), 6.58 (1H, s), 5.86 (1H, ddt,  $J$  = 17.0, 10.0, 6.5 Hz), 5.17 – 5.06 (2H, m), 3.74 (3H, s), 3.64 (3H, s), 3.13 (2H, dq,  $J$  = 6.5, 1.0 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  169.60, 159.18, 134.67, 133.98, 130.20, 129.53, 128.18, 116.85, 113.33, 54.89, 51.23, 39.14; IR (film)  $\nu$  3072, 3005, 2951, 2837, 1718, 1675, 1607, 1512, 1436, 1302, 1255, 1214, 1178, 1125, 1033, 912, 835  $\text{cm}^{-1}$ .

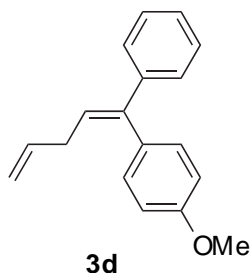
### 1-{3-[1-*tert*-Butyldimethylsilyloxymethyl]penta-1,4-dienyl}phenyl}ethanone (**3b**)



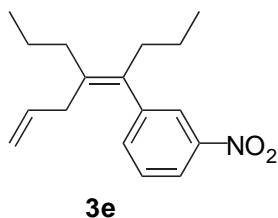
**3b** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.86 (1H, dt,  $J$  = 7.5, 1.5 Hz), 7.78 (1H, t,  $J$  = 1.5 Hz), 7.42 (1H, t,  $J$  = 7.5 Hz), 7.39 (1H, dt,  $J$  = 7.5, 1.5 Hz), 5.86 – 5.74 (2H, m), 5.05 – 4.95 (2H, m), 4.31 (2H, s), 2.73 – 2.67 (2H, m), 2.58 (3H, s), 0.87 (9H, s), 0.03 (6H, s);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  197.98, 140.30, 139.13, 136.91, 136.72, 133.37, 128.49, 128.31, 126.91, 124.69, 114.90, 67.23, 32.63, 26.61, 25.81, 18.29, -5.41; IR (film)  $\nu$  3062, 2954, 2929, 2885, 2857, 1728, 1687, 1472, 1467, 1428, 1360, 1258, 1114, 838, 779, 699  $\text{cm}^{-1}$ .

**3-(4-Methoxyphenyl)-2-methylhepta-3,6-dien-2-ol (3c)**

**3c** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.02 (2H, d,  $J = 8.5$  Hz), 6.87 (2H, d,  $J = 8.5$  Hz), 5.84 (1H, t,  $J = 7.5$  Hz), 5.72 (1H, ddt,  $J = 17.0, 10.5, 6.0$  Hz), 4.98 – 4.87 (2H, m), 3.80 (3H, s), 2.50 – 2.44 (2H, m), 1.68 (1H, br s), 1.34 (6H, s);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  158.43, 148.78, 137.12, 130.84, 130.66, 122.69, 114.50, 113.37, 72.96, 55.09, 33.24, 29.49; IR (film)  $\nu$  3385, 3033, 2975, 2835, 1636, 1607, 1511, 1465, 1441, 1359, 1285, 1235, 1176, 1176, 1102, 1036, 914, 828, 734  $\text{cm}^{-1}$ .

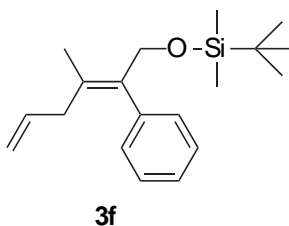
**1-Methoxy-4-[(1E)-1-phenyl-1,4-pentadienyl]benzene (3d)**

**3d** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.25 – 7.14 (5H, m), 7.09 (2H, d,  $J = 8.5$  Hz), 6.86 (2H, d,  $J = 8.5$  Hz), 6.03 (1H, t,  $J = 7.5$  Hz), 5.87 (1H, ddt,  $J = 17.0, 10.0, 6.0$  Hz), 5.07 (1H, dq,  $J = 17.0, 1.5$  Hz), 5.00 (1H, dq,  $J = 10.0, 1.5$  Hz), 3.76 (3H, s), 2.90 – 2.84 (2H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  158.62, 142.96, 142.28, 137.04, 132.06, 130.86, 127.98, 127.35, 126.88, 126.24, 114.96, 113.50, 55.05, 34.06; IR (film)  $\nu$  3058, 3001, 2956, 2933, 2836, 1725, 1653, 1639, 1607, 1576, 1511, 1493, 1463, 1445, 1361, 1294, 1248, 1177, 1108, 1033, 994, 915, 833, 764, 700  $\text{cm}^{-1}$ .

**1-(1,2-Dipropylpenta-1,4-dienyl)-3-nitrobenzene (3e)**

**3e** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.08 (1H, dt,  $J = 7.5, 2.0$  Hz), 7.97 (1H, t,  $J = 1.5$  Hz), 7.49 – 7.41 (2H, m), 5.68 (1H, ddt,  $J = 17.0, 10.0, 6.0$  Hz), 4.97 (1H, dq,  $J = 10.0, 1.5$  Hz), 4.91 (1H, dq,  $J = 17.0, 1.5$  Hz), 2.55 (2H, d,  $J = 6.5$  Hz), 2.37 (2H, dd,  $J = 8.0, 7.5$  Hz), 2.22 – 2.15 (2H, m), 1.50 (2H, sextet,  $J = 7.5$  Hz), 1.25 (2H, sextet,  $J = 7.5$  Hz), 0.98 (3H, t,  $J = 7.5$  Hz), 0.87 (3H, t,  $J = 7.5$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  148.07, 145.28, 136.58, 135.59, 135.09, 128.74, 123.58, 121.16, 115.52, 37.46, 35.85, 33.09, 21.86, 21.35, 14.25, 13.84 (the nitro substituted carbon was not detected); IR (film)  $\nu$  3077, 2960, 2931, 2871, 1635, 1529, 1465, 1350, 1080, 993, 911, 808, 739, 698  $\text{cm}^{-1}$ .

***tert*-Butyldimethyl-(3-methyl-2-phenylhexa-2,5-dienyloxy)silane (**3f**)**



**3f** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.35 – 7.28 (2H, m), 7.27 – 7.19 (1H, m), 7.18 – 7.13 (2H, m), 5.75 (1H, ddt,  $J = 17.0, 10.5, 6.5$  Hz), 4.98 – 4.89 (2H, m), 4.32 (2H, s), 2.77 (2H, d,  $J = 6.5$  Hz), 2.02 (3H, s), 0.94 (9H, s), 0.12 (6H, s);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  144.47, 137.24, 134.41, 132.62, 128.03, 127.77, 126.26, 115.06, 61.14, 35.06, 25.99, 20.79, 18.41, -5.20; IR (film)  $\nu$  3061, 2951, 2930, 2885, 2857, 1729, 1686, 1471, 1464, 1446, 1362, 1256, 1100, 837, 776, 702  $\text{cm}^{-1}$ .

**Procedure for the One-pot Tandem Bromoallylation/Suzuki Cross-Coupling of Alkynes with Carbene Additives (Eq. 1)**

The bromoallylation of methylpropiolate was carried out as described above (Table 1, entry 1). To the resulting crude mixture was added cesium carbonate (1.30 g, 4.00 mmol), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (34mg, 0.10 mmol) and 4-methoxyphenylboronic acid (4.00 mmol). The reaction mixture was then heated at 45 °C for 12 h. The work-up procedure is as described in Table 2. **3a** was obtained as a clear, colorless liquid in 85% yield.

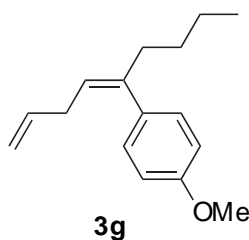
**General Procedure for the One-pot Tandem Bromoallylation/Suzuki Cross-Coupling of Alkynes at Room Temperature (Table 3).**

To a solution of allylbromide (173  $\mu\text{L}$ , 2.00 mmol) in anhydrous THF (5.0 mL) cooled in an ice-bath was added dibromo(bisbenzotrile)palladium(II) (28.3 mg, 0.06 mmol). A solution of the alkyne (2.00 mmol) in anhydrous THF (5.0 mL) was then added dropwise over *ca.* 30 min. The



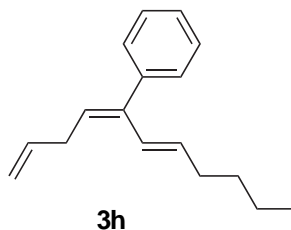
reaction mixture was stirred for another 5.5 h at rt. Tri-*t*-butylphosphine (1M in THF, 120  $\mu$ L, 0.12 mmol) was then added and the reaction mixture stirred for 15 min at rt. Cesium carbonate (1.30 g, 4.00 mmol) was subsequently added followed by the boronic acid (4.00 mmol). The reaction mixture was stirred for 16 h at rt and then concentrated *in vacuo*. The mixture was diluted with Et<sub>2</sub>O (100 mL) and the insoluble material filtered off. The filtrate was concentrated *in vacuo* and the residue chromatographed on silica gel (EtOAc/hexanes).

### 1-(1-Butylpenta-1,4-dienyl)-4-methoxybenzene (**3g**)

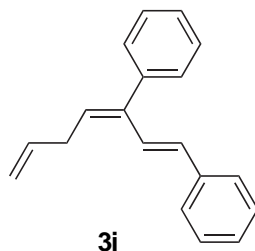


**3g** isolated as a clear, colorless oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  7.08 (2H, d,  $J$  = 8.5 Hz), 6.87 (2H, d,  $J$  = 8.5 Hz), 5.82 (1H, ddt,  $J$  = 17.0, 10.0, 6.0 Hz), 5.44 (1H, t,  $J$  = 7.5 Hz), 5.02 (1H, dq,  $J$  = 17.0, 2.0 Hz), 4.97 (1H, dq,  $J$  = 10.0, 1.5 Hz), 3.82 (3H, s), 2.70 (2H, t,  $J$  = 6.5 Hz), 2.34 (2H, t,  $J$  = 6.5 Hz), 1.35 – 1.25 (4H, m), 0.86 (3H, t,  $J$  = 7.0 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  158.20, 141.96, 137.88, 133.27, 129.28, 123.51, 114.32, 113.38, 55.16, 39.02, 33.25, 30.36, 22.22, 13.90; IR (film)  $\nu$  3030, 3000, 2956, 2932, 2871, 2837, 1727, 1676, 1608, 1511, 1465, 1378, 1290, 1248, 1179, 1110, 1035, 912, 834 cm<sup>-1</sup>.

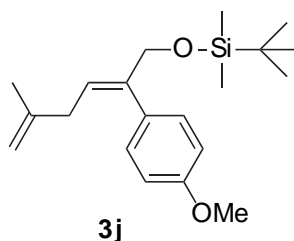
### (1-Hex-1-enylpenta-1,4-dienyl)benzene (**3h**)



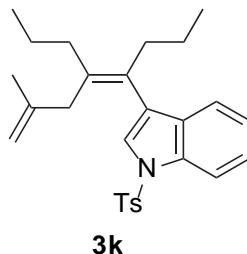
**3h** isolated as a clear, colorless oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  7.34 – 7.21 (5H, m), 6.46 (1H, dq,  $J$  = 15.5, 1.0 Hz), 5.89 (1H, ddt,  $J$  = 17.0, 10.0, 6.0 Hz), 5.56 (1H, dt,  $J$  = 15.5, 7.0 Hz), 5.44 (1H, t,  $J$  = 7.5 Hz), 5.11 (1H, dq,  $J$  = 17.0, 1.5 Hz), 5.02 (1H, dq,  $J$  = 10.0, 1.5 Hz), 3.08 – 3.01 (2H, m), 2.13 (2H, q,  $J$  = 6.5 Hz), 1.41 – 1.24 (4H, m), 0.89 (3H, t,  $J$  = 7.0 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  142.45, 140.53, 136.66, 135.50, 128.74, 127.88, 126.85, 126.74, 126.31, 115.04, 32.99, 32.38, 31.47, 22.31, 13.95; IR (film)  $\nu$  3079, 3057, 3022, 2956, 2931, 2861, 1725, 1684, 1641, 1600, 1575, 1493, 1466, 1443, 1379, 1319, 1074, 988, 916, 767, 703 cm<sup>-1</sup>.

**(1-vinylphenylpenta-1,4-dienyl)benzene (3i)**

**3i** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.43 – 7.17 (11H, m), 6.42 (1H, d,  $J = 16.0$  Hz), 5.93 (1H, ddt,  $J = 17.0, 10.0, 6.0$  Hz), 5.62 (1H, t,  $J = 7.5$  Hz), 5.16 (1H, dq,  $J = 17.0, 1.5$  Hz), 5.07 (1H, dq,  $J = 10.0, 1.5$  Hz), 3.20 – 3.14 (2H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  141.82, 140.64, 137.54, 136.31, 132.49, 129.87, 128.95, 128.56, 128.08, 127.54, 127.04, 126.47, 125.25, 115.44, 32.48; IR (film)  $\nu$  3093, 3078, 3027, 2976, 1638, 1597, 1574, 1491, 1446, 1300, 1073, 1029, 991, 964, 911, 749, 701  $\text{cm}^{-1}$ .

***tert*-Butyl-[2-(4-methoxyphenyl)-5-methylhexa-2,5-dienyloxy]dimethylsilane (3j)**

**3j** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.13 (2H, d,  $J = 8.5$  Hz), 6.89 (2H, d,  $J = 8.5$  Hz), 5.82 (1H, tt,  $J = 7.5, 1.5$  Hz), 4.77 – 4.72 (2H, m), 4.33 (2H, d,  $J = 1.5$  Hz), 3.82 (3H, s), 2.70 (2H, d,  $J = 7.5$  Hz), 1.71 (3H, s), 0.93 (9H, s), 0.08 (6H, s);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  158.51, 145.29, 140.64, 130.96, 129.65, 123.21, 113.42, 110.01, 67.30, 55.12, 36.60, 25.90, 22.88, 18.41, -5.37; IR (film)  $\nu$  3031, 2955, 2930, 2856, 1728, 1690, 1608, 1512, 1463, 1289, 1250, 1177, 1121, 1035, 836, 778, 666  $\text{cm}^{-1}$ .

**3-(4-Methyl-1,2-dipropylpenta-1,4-dienyl)-1-(toluene-4-sulfonyl)-1H-indole (3k)**

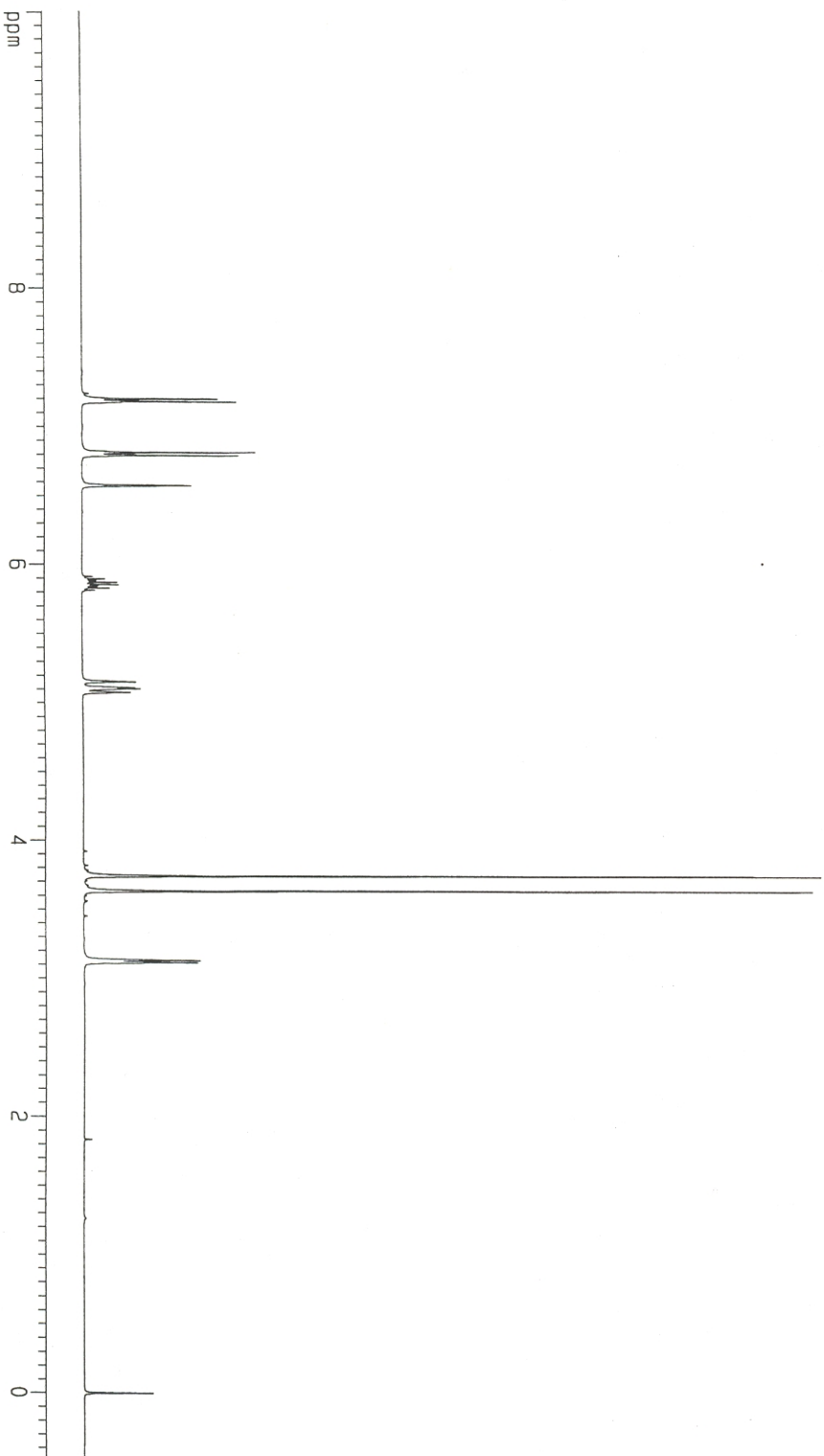
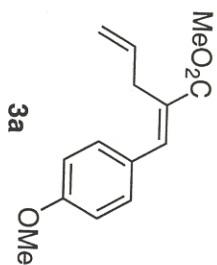
**3k** isolated as a clear, colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.99 (1H, d,  $J$  = 8.0 Hz), 7.69 (2H, d,  $J$  = 8.5 Hz), 7.32 – 7.24 (3H, m), 7.22 – 7.14 (3H, m), 4.72 (1H, s), 4.63 (1H, s), 2.45 (2H, s), 2.35 – 2.27 (5H, m), 2.19 – 2.13 (2H, m), 1.54 – 1.44 (5H, m), 1.23 (2H, sextet,  $J$  = 7.5 Hz), 0.98 (3H, t,  $J$  = 7.5 Hz), 0.83 (3H, t,  $J$  = 7.5 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  144.59, 144.39, 137.55, 135.05, 135.01, 131.16, 129.59, 128.08, 126.53, 124.86, 124.43, 123.13, 123.08, 120.31, 113.79, 110.87, 41.45, 34.83, 32.66, 22.51, 22.08, 21.81, 21.37, 14.23, 13.83; IR (film)  $\nu$  3069, 2959, 2930, 2870, 1647, 1698, 1446, 1372, 1306, 1175, 1126, 1093, 1018, 974, 889, 811, 747, 666  $\text{cm}^{-1}$ .

#### General Procedure for the One-pot Tandem Chloroallylation/Suzuki Cross-Coupling of Alkynes (Table 4).

To a solution of allylchloride (163  $\mu\text{L}$ , 2.00 mmol) in anhydrous THF (5.0 mL) cooled in an ice-bath was added dibromo(bisbenzonitrile)palladium(II) (28.3 mg, 0.06 mmol). A solution of the alkyne (2.00 mmol) in anhydrous THF (5.0 mL) was then added dropwise over *ca.* 30 min. The reaction mixture was stirred for another 5.5 h at rt (12 h for disubstituted alkynes). Tri-*t*-butylphosphine (1M in THF, 120  $\mu\text{L}$ , 0.12 mmol) was then added and the reaction mixture stirred for 15 min at rt. Cesium carbonate (1.30 g, 4.00 mmol) was subsequently added followed by the boronic acid (4.00 mmol). The reaction mixture was stirred for 16 h at 45  $^\circ\text{C}$  and then concentrated *in vacuo*. The mixture was diluted with  $\text{Et}_2\text{O}$  (100 mL) and the insoluble material filtered off. The filtrate was concentrated *in vacuo* and the residue subjected to flash silica gel chromatography ( $\text{EtOAc}$ /hexanes).

#### References

- 1) Kaneda, K.; Uchiyama, T.; Fujiwara, Y.; Imanaka, T.; Teranishi, S. *J. Org. Chem.* **1979**, *44*, 55-63.
- 2) Llebaria, A.; Camps, F.; Moretó, J. M. *Tetrahedron* **1993**, *49*, 1283-1296.



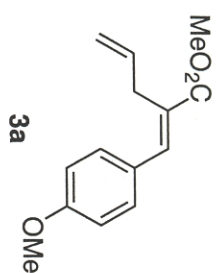
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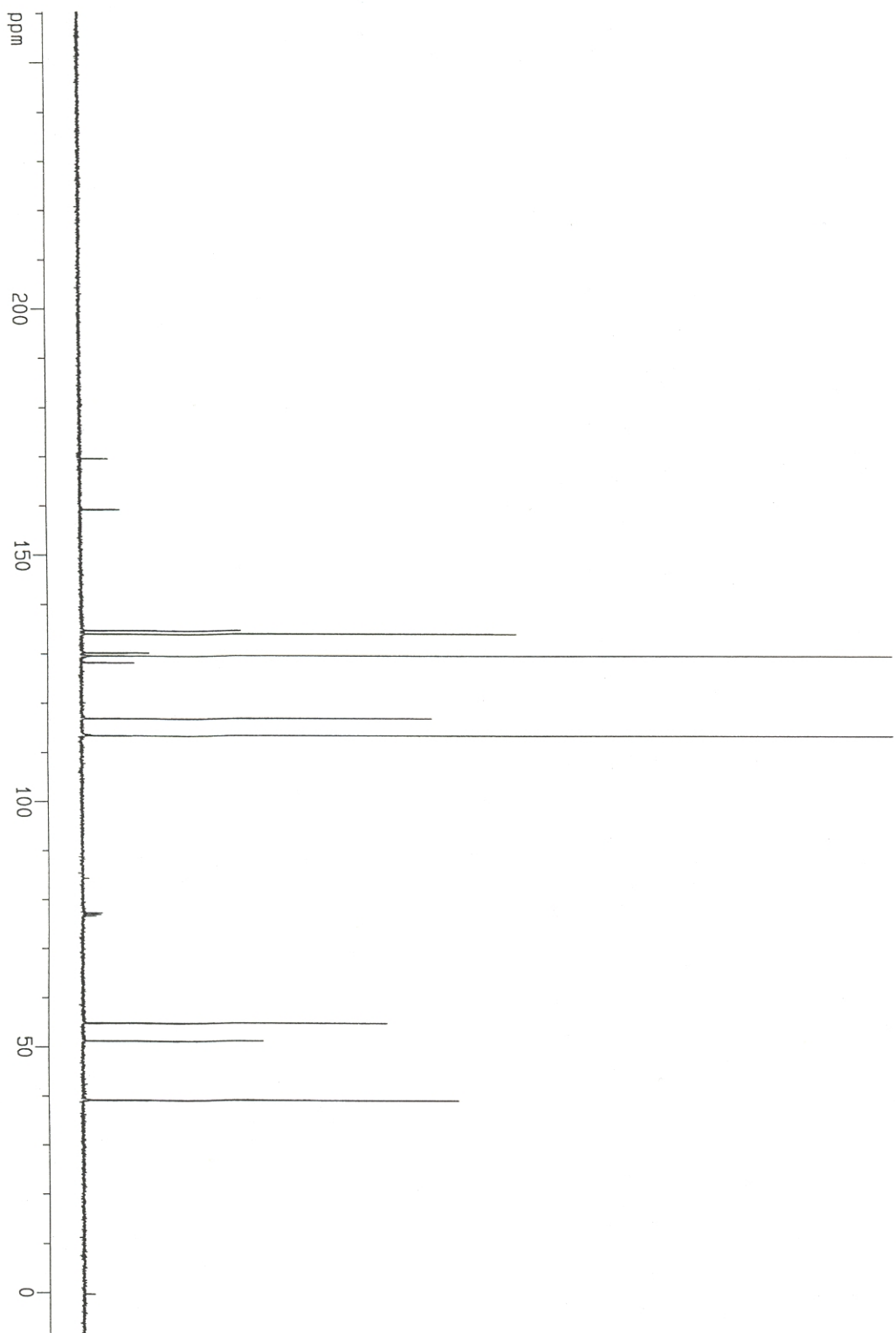
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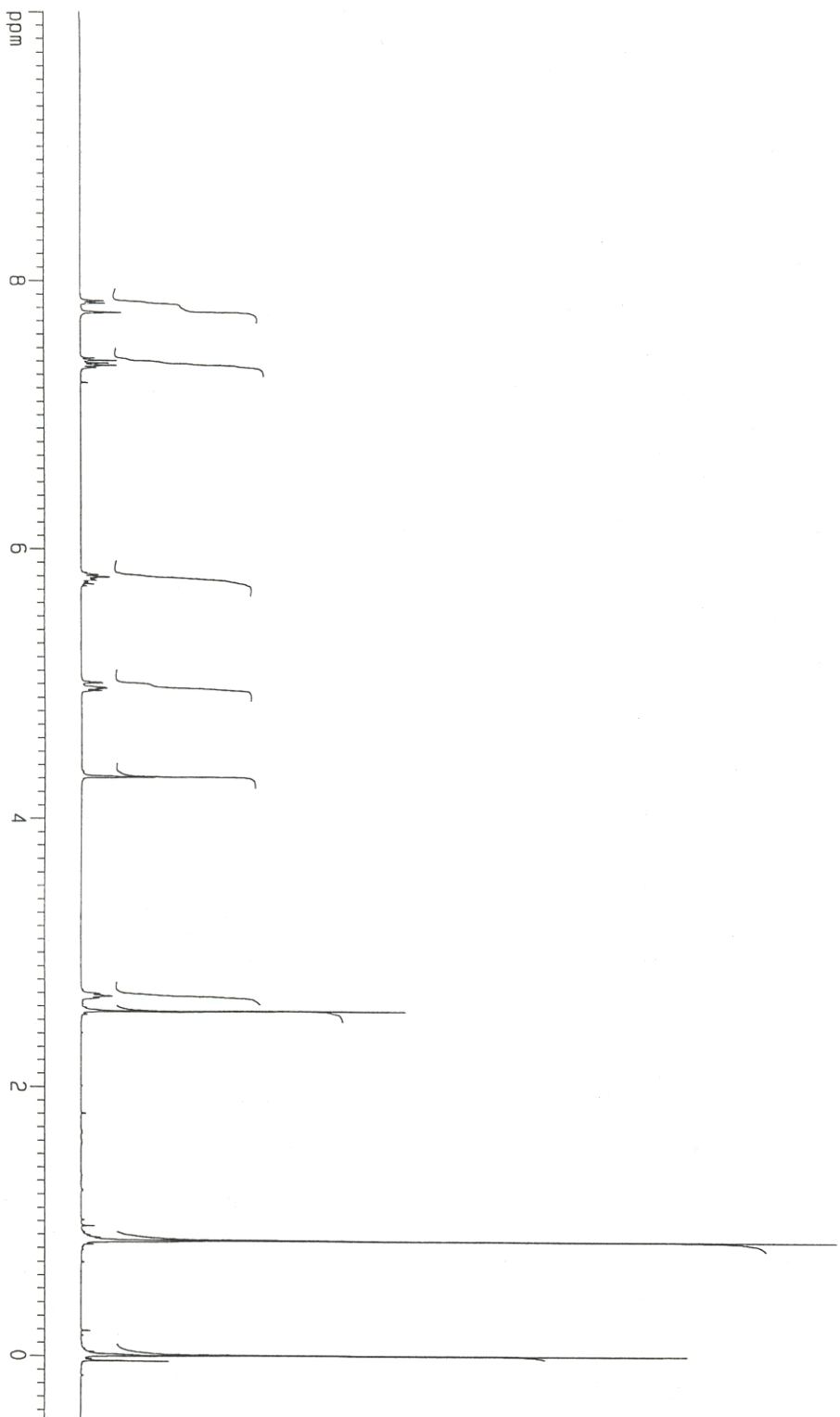
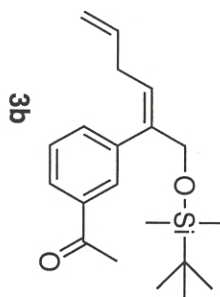
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127942 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.322 ppm  
 F1 26191.71 Hz  
 F2P -9.030 ppm  
 F2 -908.56 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01355 Hz/cm





Current Data Parameters  
NAME naphthoindole  
EXPNO 159  
PROCNO 1

# F2 - Acquisition Parameters

Date\_ 20020112  
Time 18.15  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zg  
TD 32768  
SOLVENT CDCl<sub>3</sub>  
NS 4  
DS 0  
SWH 4194.631 Hz  
FIDRES 0.128010 Hz  
AQ 3.905956 sec  
RG 35.9  
DW 119.200 usec  
DE 4.50 usec  
TE 300.0 K  
D1 2.00000000 sec

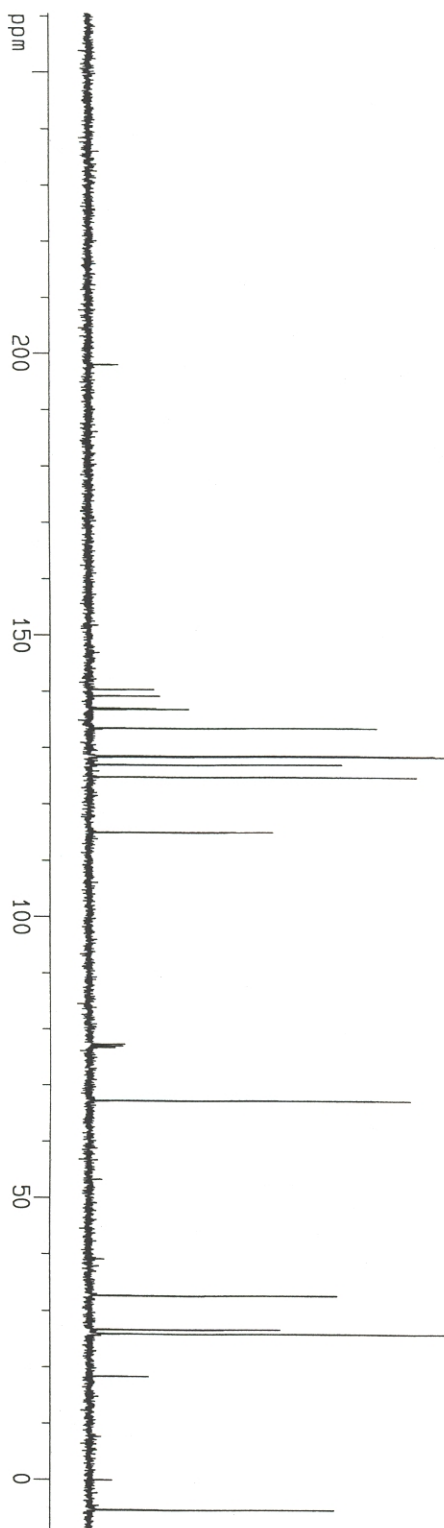
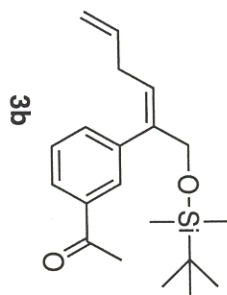
===== CHANNEL f1 =====  
NUC1 1H  
P1 10.50 usec  
PL1 0.00 dB  
SFO1 400.1319246 MHz

# F2 - Processing parameters

SI 16384  
SF 400.1300177 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

# 1D NMR plot parameters

CX 20.00 cm  
F1P 10.007 ppm  
F1 4004.18 Hz  
F2P -0.476 ppm  
F2 -190.45 Hz  
PPMCM 0.52416 ppm/cm  
HZCM 209.73154 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 172  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020119  
 Time 14.45

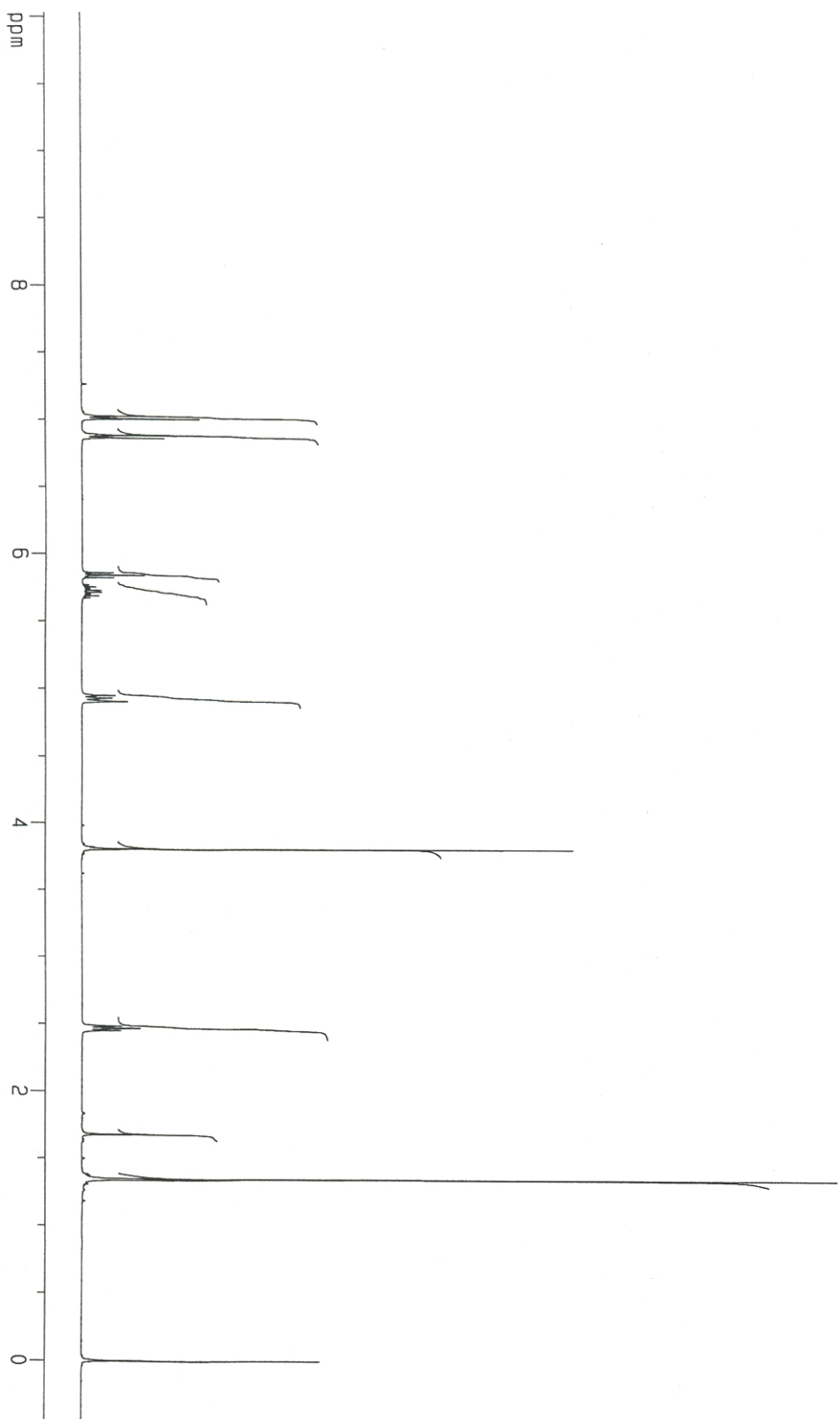
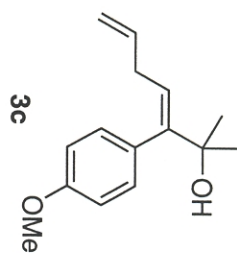
INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg  
 TD 32768  
 SOLVENT CDCl<sub>3</sub>  
 NS 37  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 1149.4  
 DM 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 <sup>1</sup>H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.612777 MHz  
 MDM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 250.486 ppm  
 F1 26208.25 Hz  
 F2P -8.866 ppm  
 F2 -892.02 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01355 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 130  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20011208  
 Time 18.30

INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0

SWH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.905956 sec

RG 14.3  
 DW 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB

SFO1 400.1319246 MHz

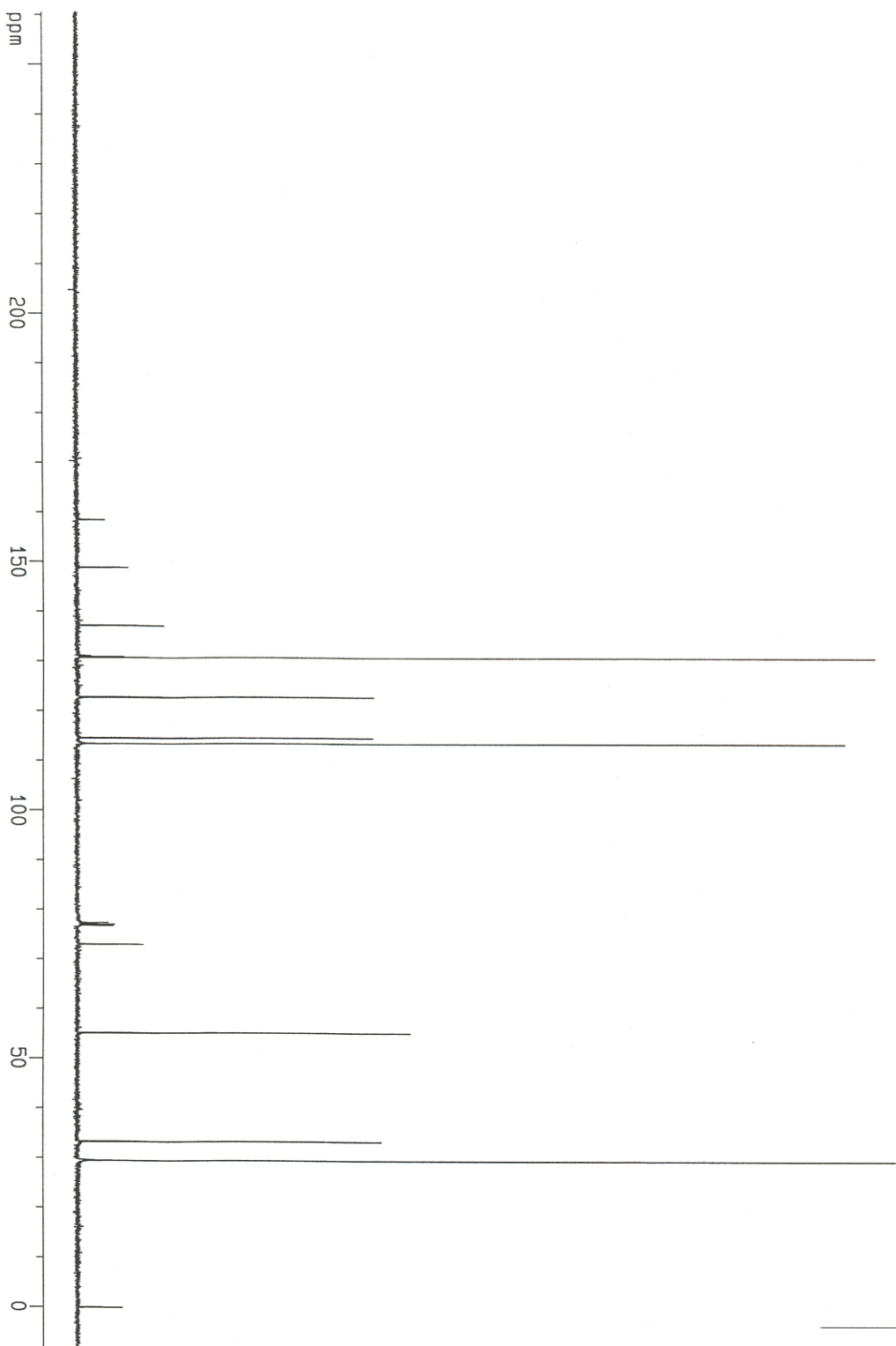
F2 - Processing parameters

SI 16384  
 SF 400.1300089 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters

CX 20.00 cm  
 F1P 10.029 ppm  
 F1 4013.00 Hz  
 F2P -0.454 ppm  
 F2 -181.63 Hz  
 PPMCM 0.52416 ppm/cm  
 HZCM 209.73154 Hz/cm





NAME	haploindole
EXPNO	131
PROCNO	1

Date_	20011208
Time	18.33

INSTRUM	spect	
PROBHD	5 mm QNP 1H	
PULPROG	zgpg	
TD	32768	
SOLVENT	CDCl3	
NS	192	
DS	0	
SMH	27100.271 Hz	
FIDRES	0.827035 Hz	
AQ	0.6046196 sec	
RG	2298.8	
DM	18.450 usec	
DE	7.50 usec	
TE	300.0 K	
D1	3.00000000 sec	
d11	0.03000000 sec	

```
===== CHANNEL f1 =====
```

NUC1	13C
P1	11.00 usec
PL1	-2.00 dB
SFO1	100.6254358 MHz

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

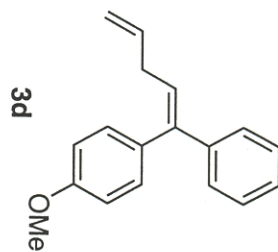
CPDP662	waitz16
NUC2	1H
PCPD2	101.00 usec
PL2	120.00 dB
PL12	19.00 dB
SF02	400.1324710 MHz

## F2 - Processing parameters

SI	16384	
SF	100.612760	MHz
MDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.00	

## 1D NMR plot parameters

CX	20.00 cm
F1P	260.503 ppm
F1	26209.90 Hz
F2P	-8.849 ppm
F2	-890.37 Hz
PPMCM	13.46761 ppm/cm
HZCM	1355.01367 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 138  
 PROCNO 1

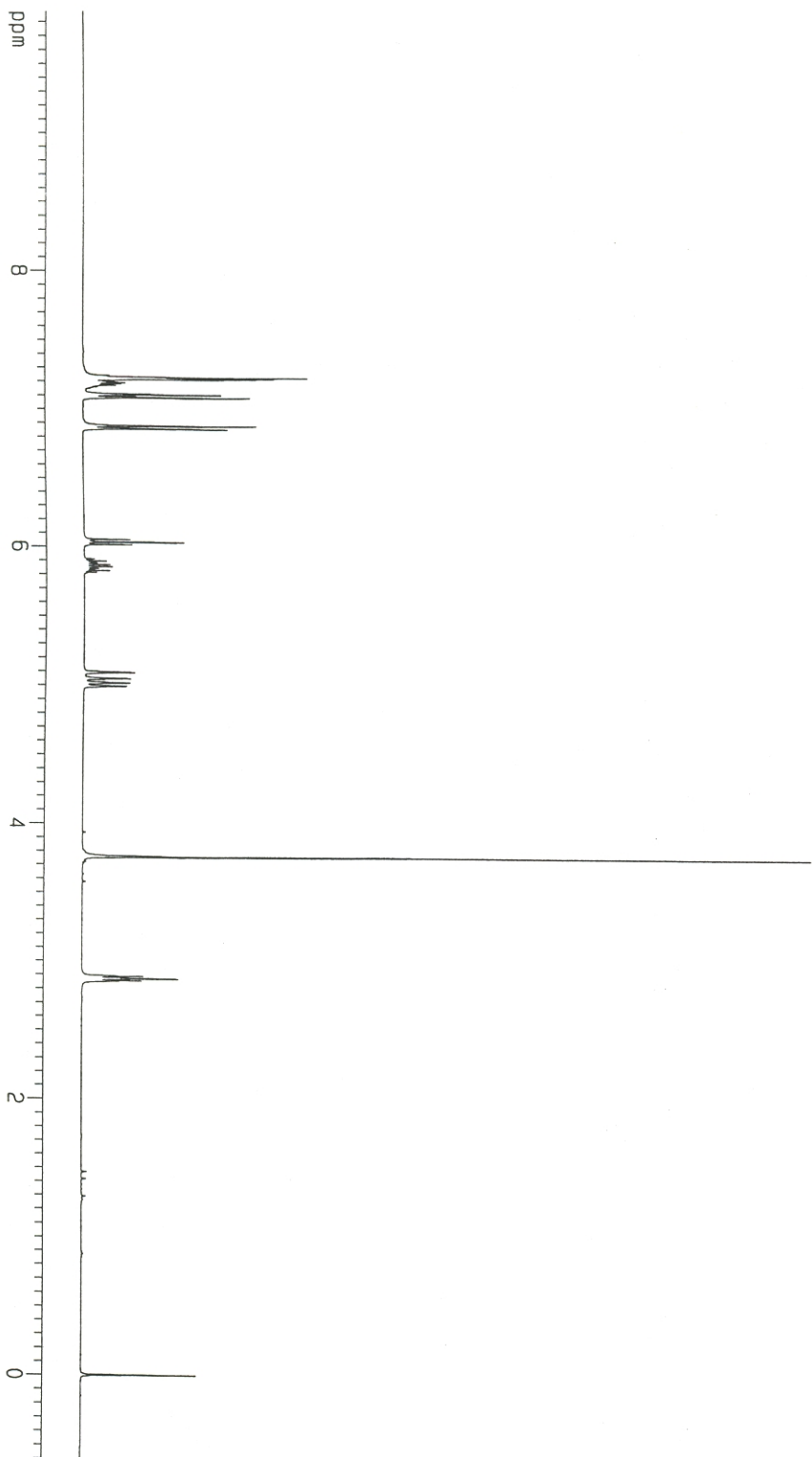
## F2 - Acquisition Parameters

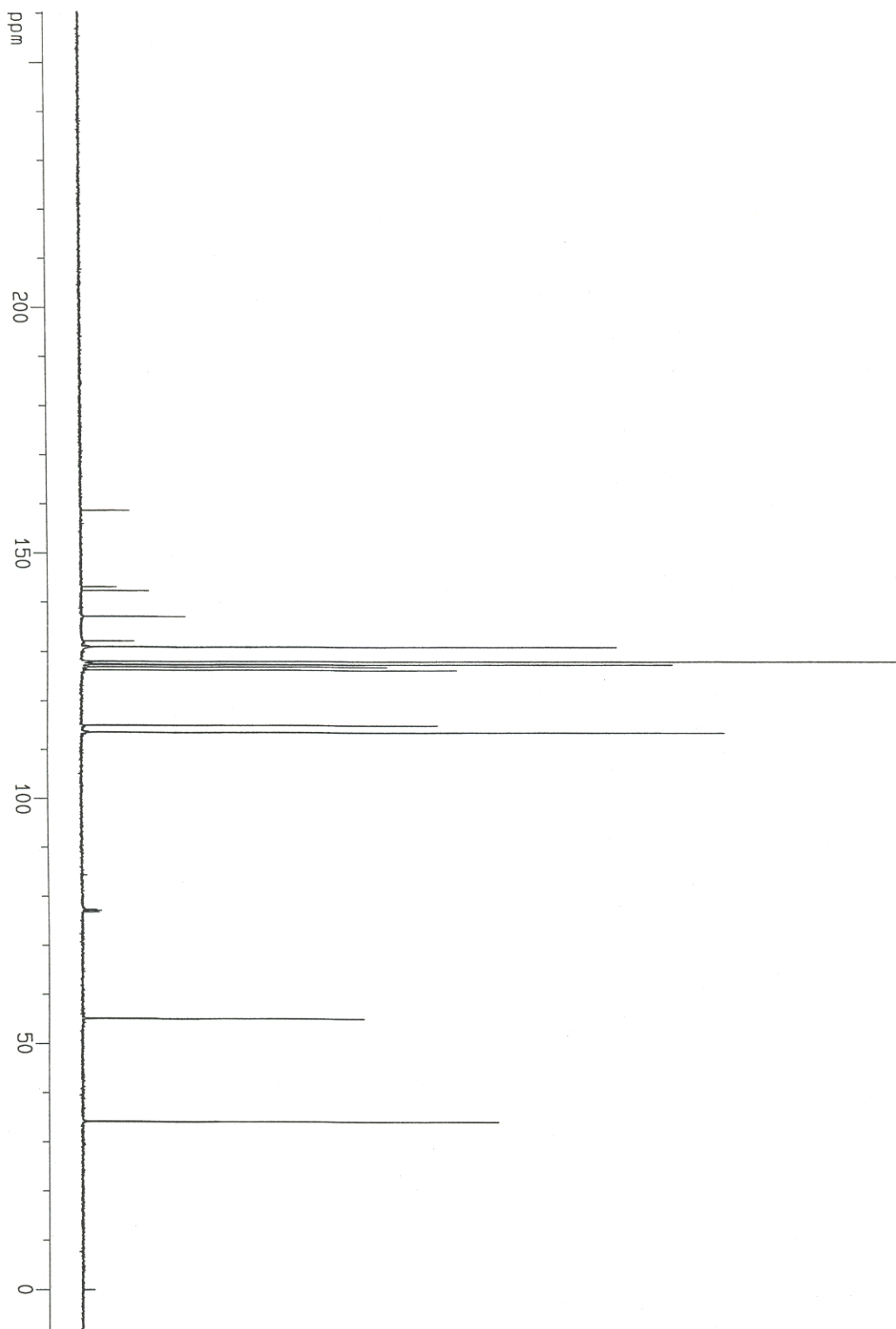
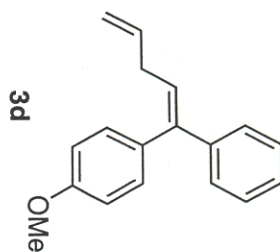
Date\_ 20011217  
 Time 11.53  
 INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0  
 SMH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.905956 sec  
 RG 8  
 DW 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB  
 SF01 400.1319246 MHz

F2 - Processing parameters  
 SI 16384  
 SF 400.1300689 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 9.879 ppm  
 F1 3952.95 Hz  
 F2P -0.604 ppm  
 F2 -241.68 Hz  
 PPMCM 0.52416 ppm/cm  
 HZCM 209.73152 Hz/cm





Current Data Parameters  
 NAME haploindole  
 EXPNO 139  
 PROCNO 1

# F2 - Acquisition Parameters

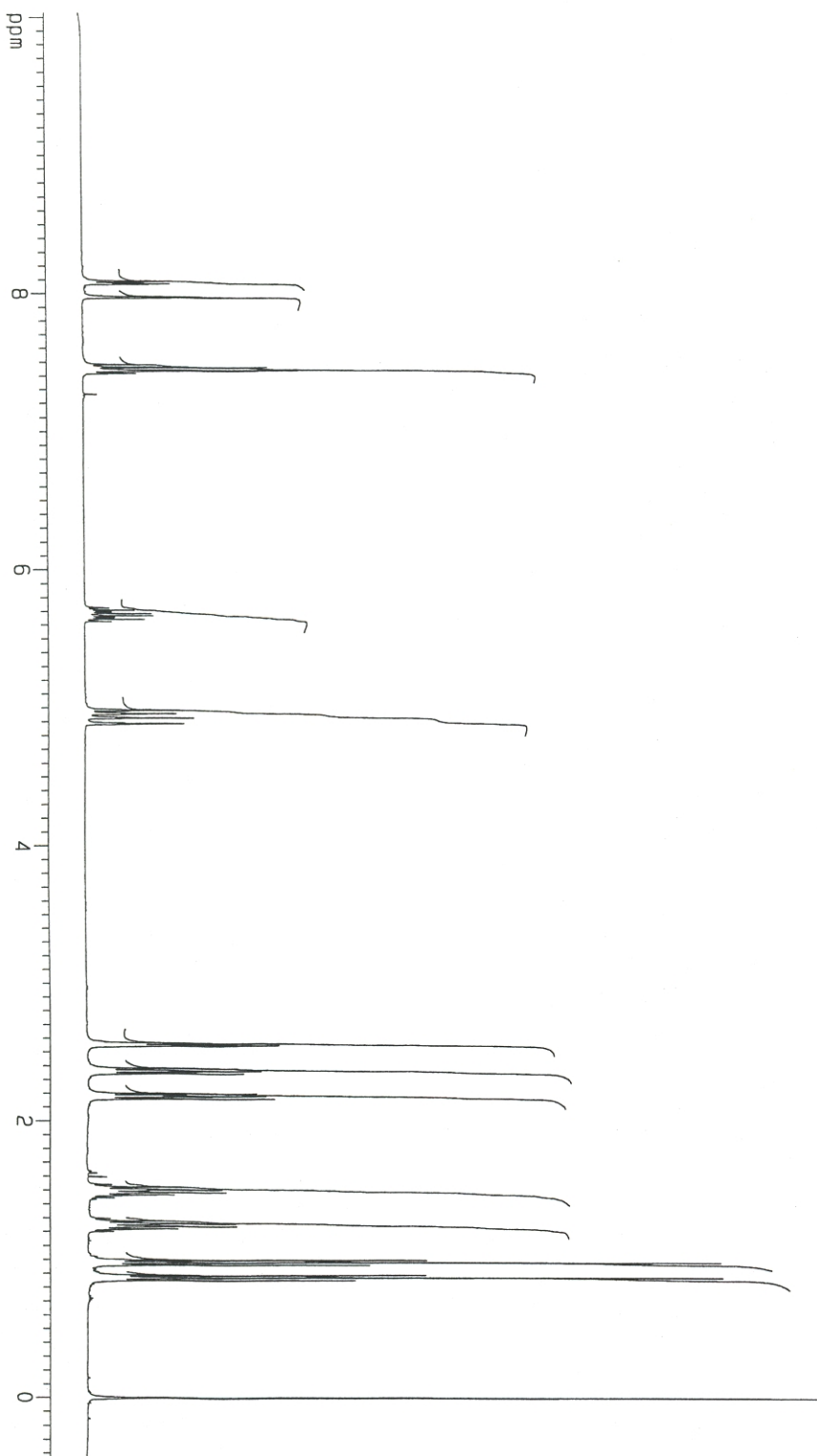
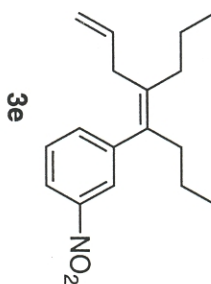
Date\_ 20011217  
 Time 11.56  
 INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg  
 TO 32768  
 SOLVENT CDCl3  
 NS 136  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 574.7  
 DW 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing Parameters  
 SI 16384  
 SF 100.6127909 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.355 ppm  
 F1 26195.02 Hz  
 F2P -8.997 ppm  
 F2 -905.25 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01365 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 132  
 PROCNO 1

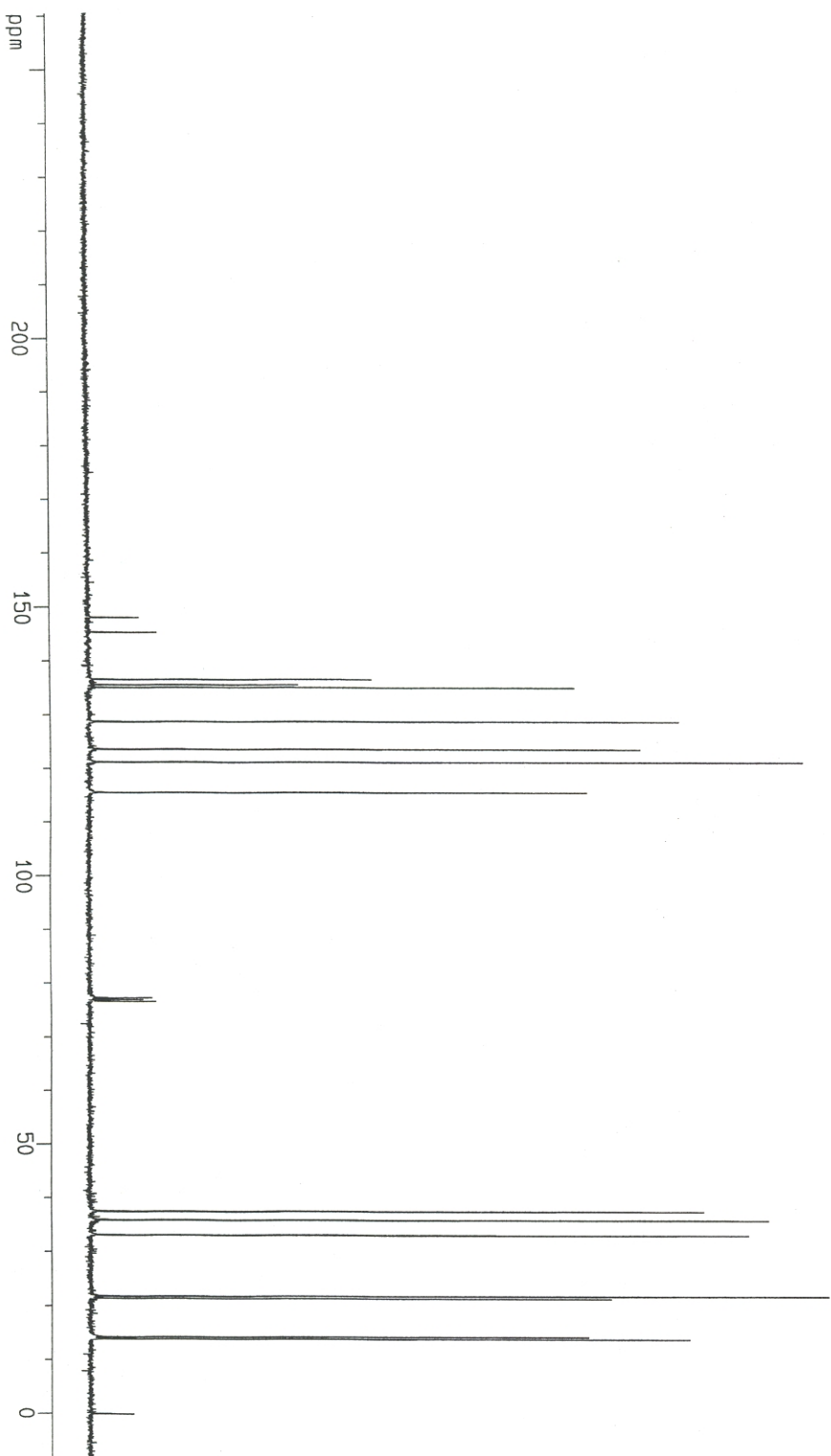
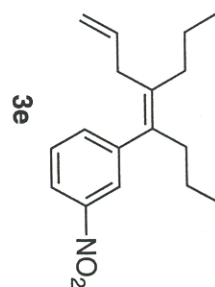
F2 - Acquisition Parameters  
 Date\_ 20011211

Time 14.28  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0  
 SWH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.905956 sec  
 RG 12.7  
 DW 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB  
 SF01 400.1319246 MHz

F2 - Processing parameters  
 SI 16384  
 SF 400.130067 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.035 ppm  
 F1 4015.16 Hz  
 F2P -0.449 ppm  
 F2 -179.47 Hz  
 PPMCM 0.52416 ppm/cm  
 HZCM 209.73154 Hz/cm



Current Data Parameters  
 NAME hap10indole  
 EXPNO 133  
 PROCNO 1

# F2 - Acquisition Parameters

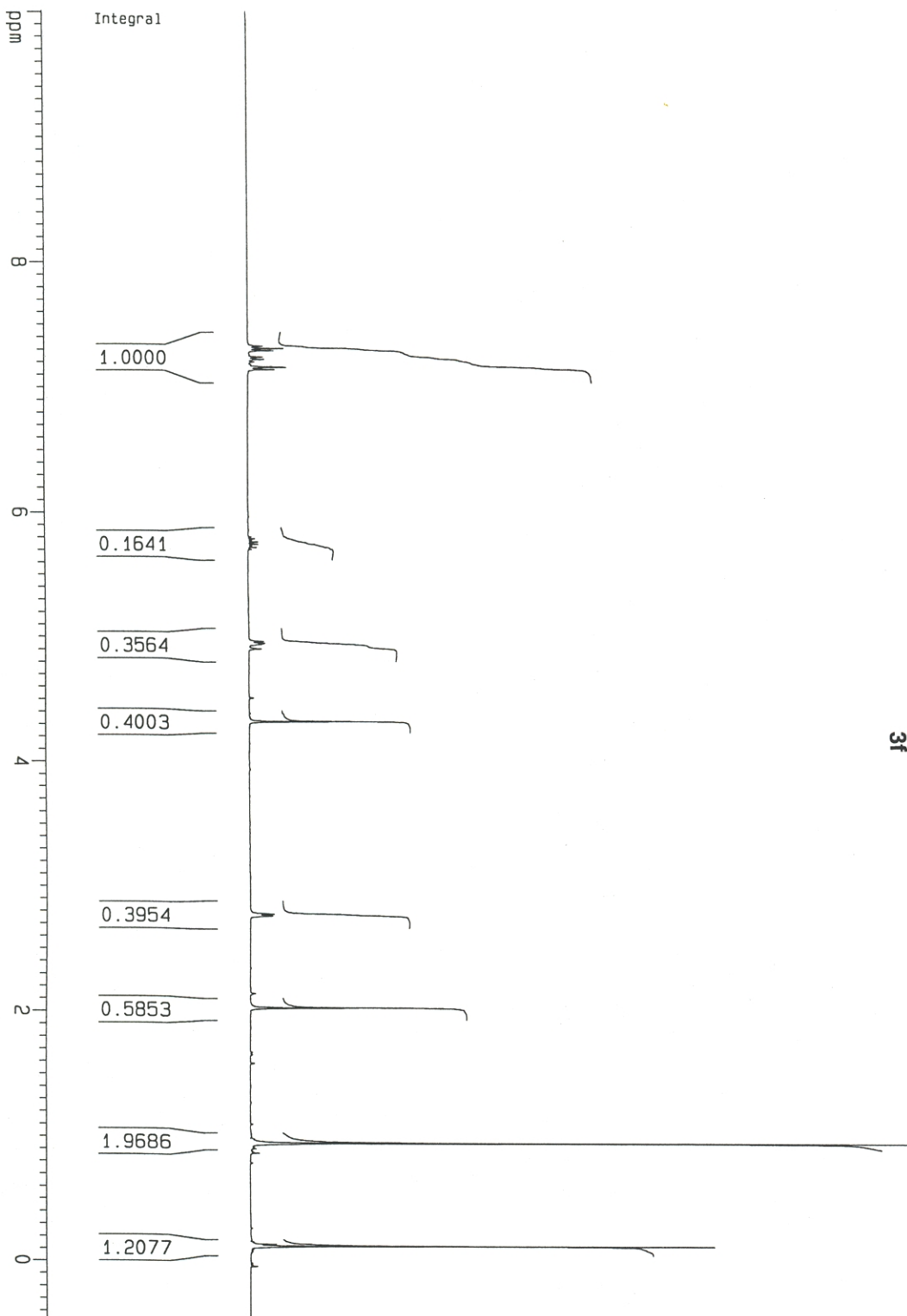
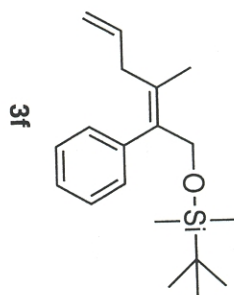
Date\_ 20011211  
 Time 14.31  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpg  
 TD 32768  
 SOLVENT CDCl3  
 NS 445  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 812.7  
 DM 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SFO1 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 POPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SFO2 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127727 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.536 ppm  
 F1 26213.21 Hz  
 F2P -8.817 ppm  
 F2 -887.06 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01355 Hz/cm



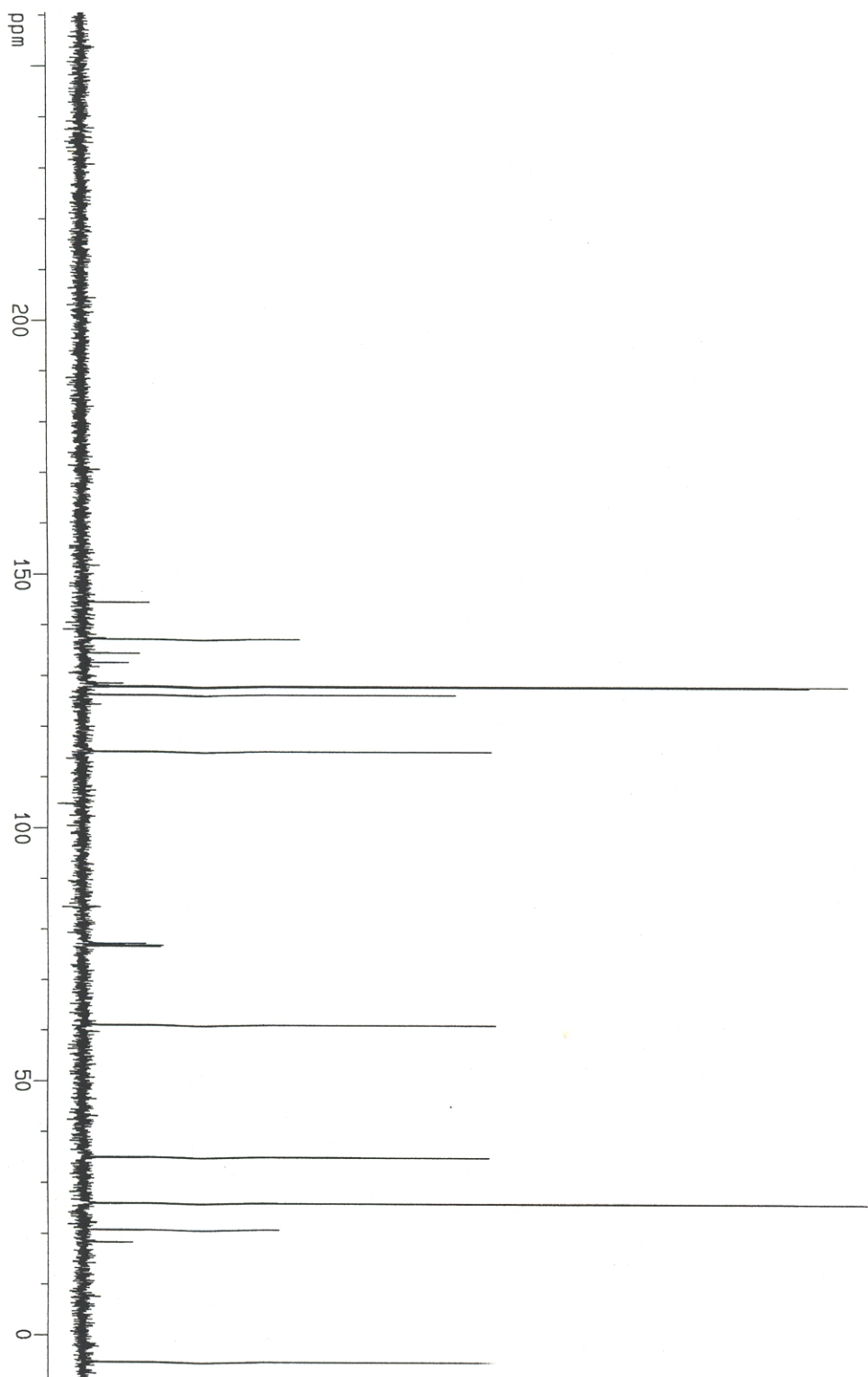
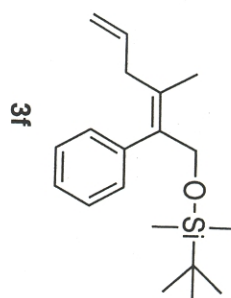
Current Data Parameters  
 NAME haploindole  
 EXPNO 110  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20011129  
 Time 17.13  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0  
 SMH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.9059956 sec  
 RG 16  
 DM 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB  
 SF01 400.1319246 MHz

F2 - Processing parameters  
 SI 16384  
 SF 400.1300177 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.007 ppm  
 F1 4004.18 Hz  
 F2P -0.476 ppm  
 F2 -190.45 Hz  
 PPMCM 0.52416 ppm/cm  
 HZCM 209.73154 Hz/cm



Current Data Parameters  
NAME haploindole  
EXPNO 111  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20011129

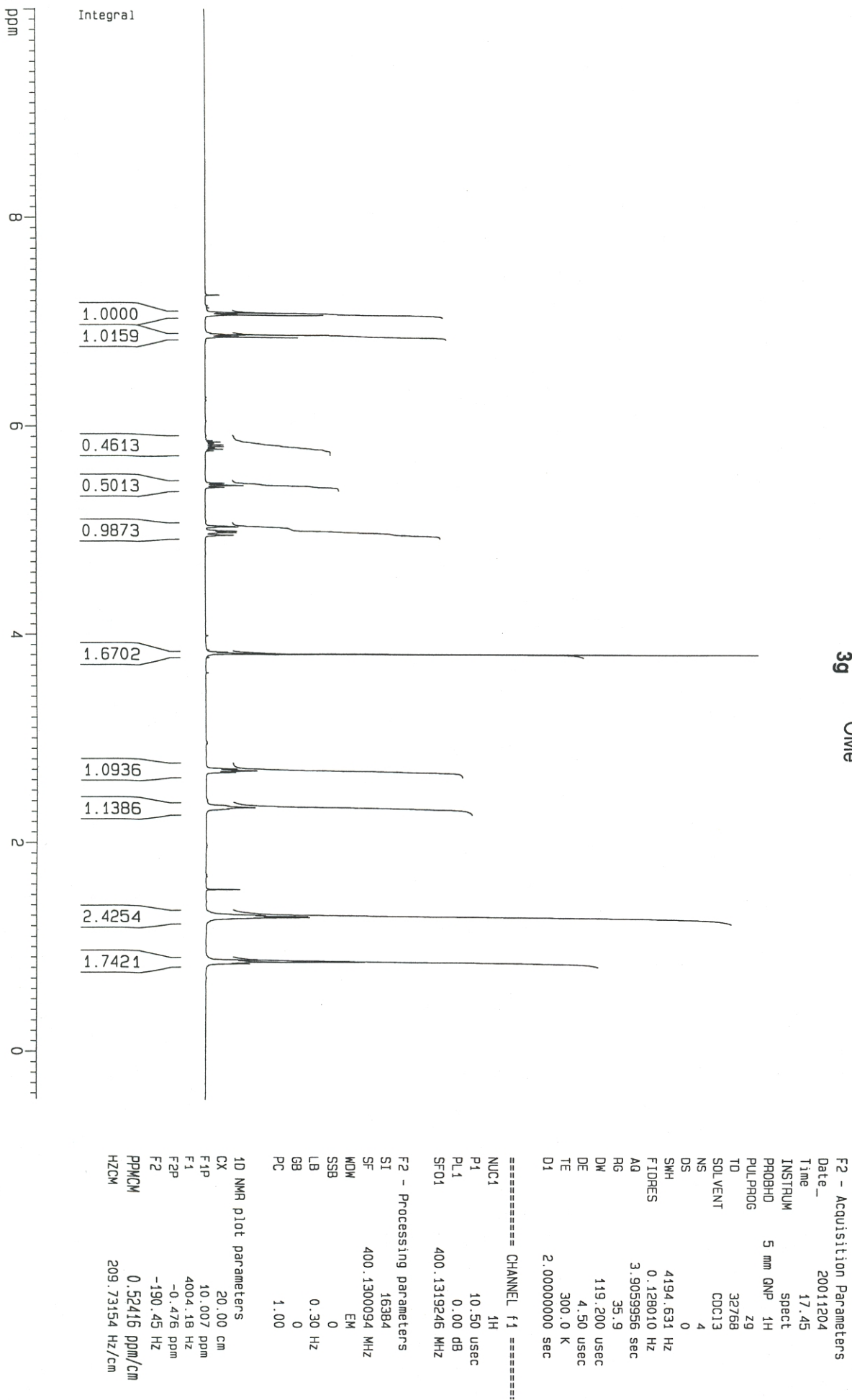
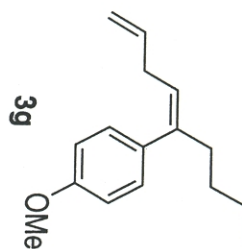
Time 17.24  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg  
TD 32768  
SOLVENT CDCl3  
NS 49  
DS 0  
SWH 27100.271 Hz  
FIDRES 0.827035 Hz  
AQ 0.6046196 sec  
RG 2298.8  
DM 18.450 usec  
DE 7.50 usec  
TE 300.0 K  
D1 3.00000000 sec  
d11 0.03000000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.00 usec  
PL1 -2.00 dB  
SF01 100.6254358 MHz

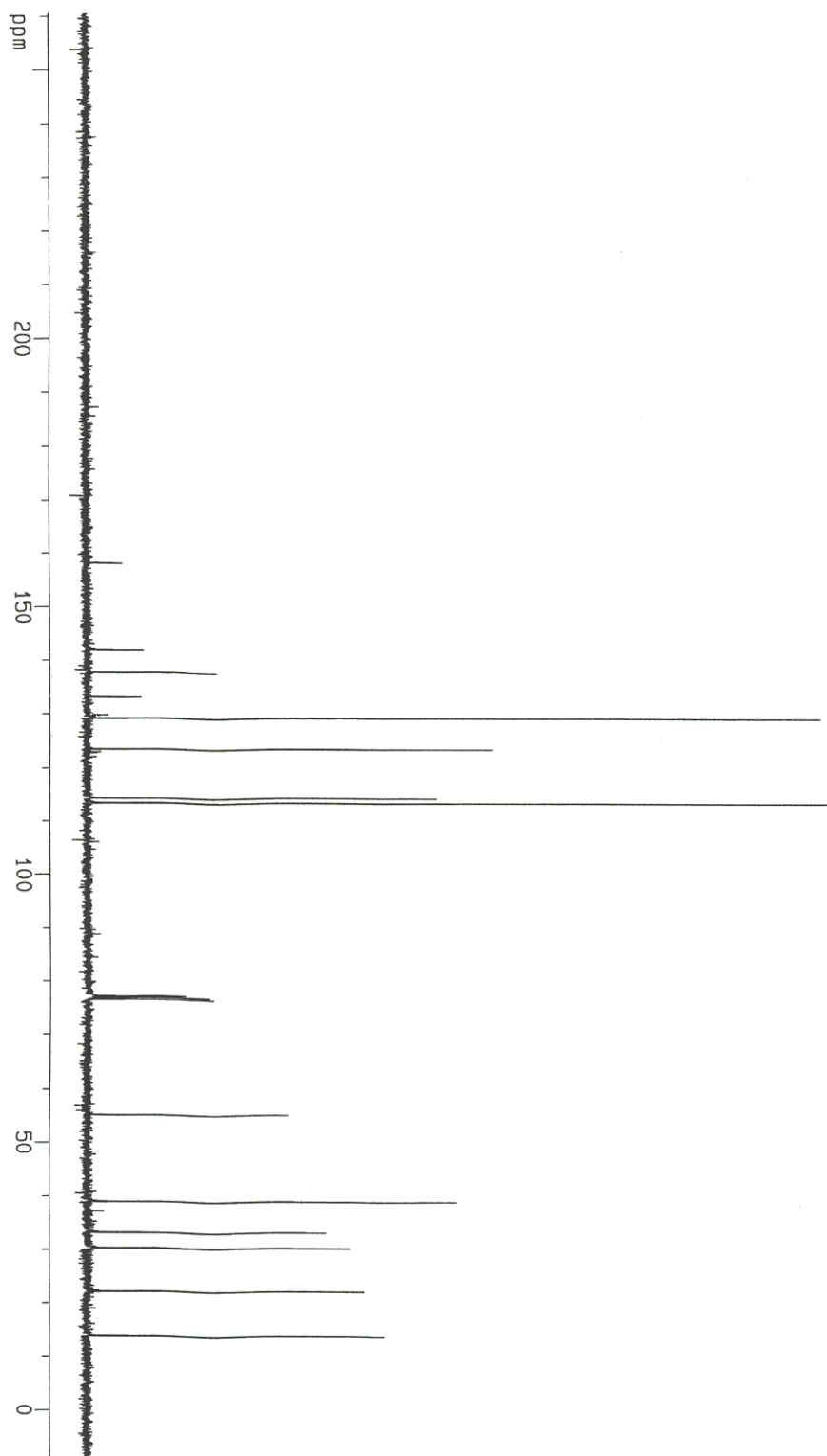
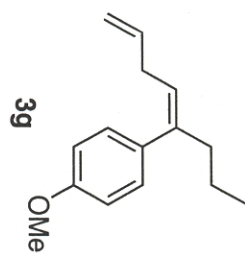
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 101.00 usec  
PL2 120.00 dB  
PL12 19.00 dB  
SF02 400.1324710 MHz

F2 - Processing parameters  
SI 16384  
SF 100.6127743 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 260.519 ppm  
F1 26211.56 Hz  
F2P -8.833 ppm  
F2 -888.71 Hz  
PNUCM 13.46761 ppm/cm  
HZCM 1355.01367 Hz/cm







Current Data Parameters  
 NAME haploindole  
 EXPNO 124  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20011204

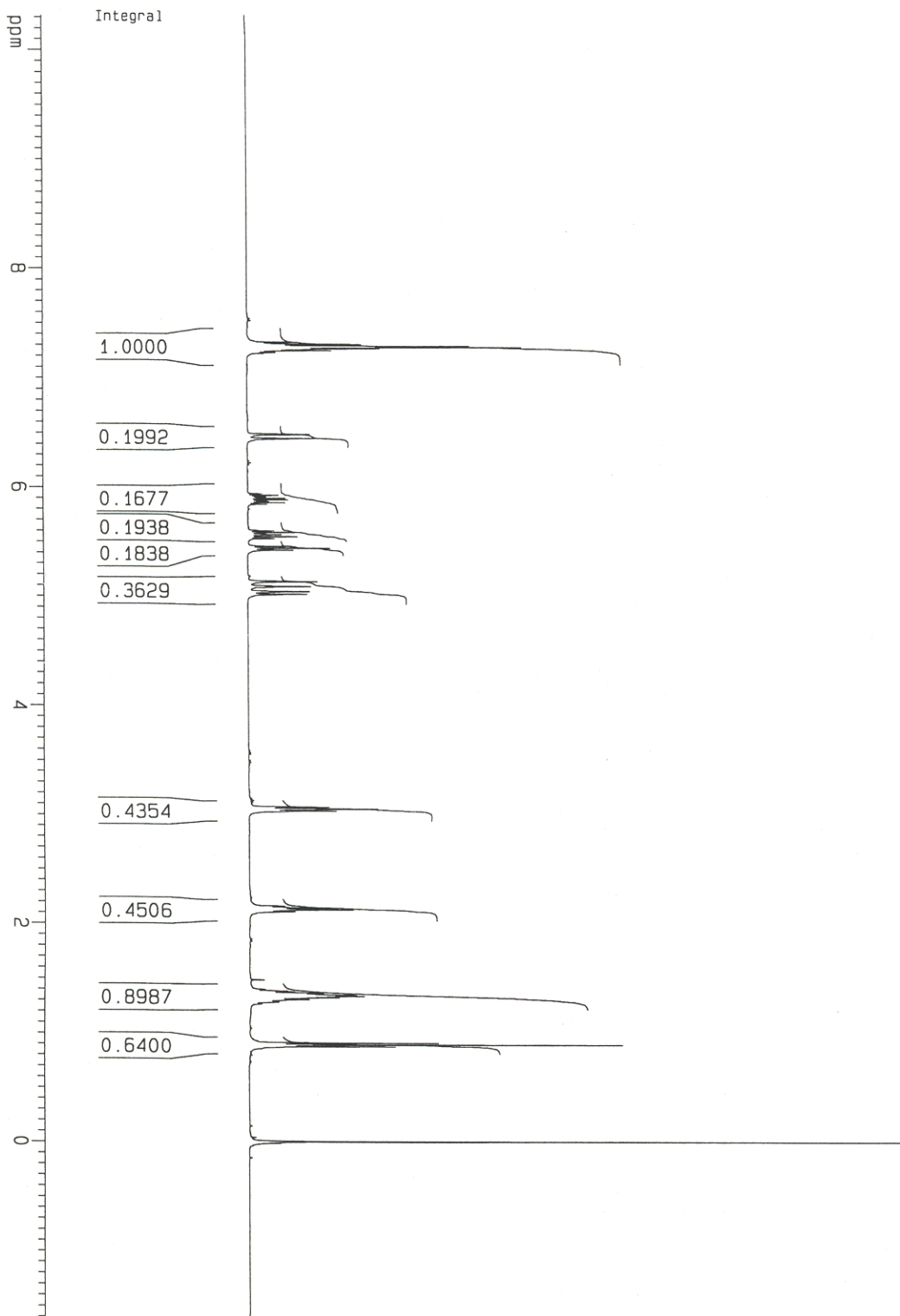
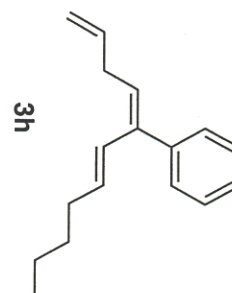
Time 19.05  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H  
 PULPROG zgpg  
 TD 32768  
 SOLVENT CDCl3  
 NS 389  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 1149.4  
 DM 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127710 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.552 ppm  
 F1 26214.86 Hz  
 F2P -8.800 ppm  
 F2 -885.41 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01355 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 21  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020212  
 Time 10.17

INSTRUM spect  
 PROBD 5 mm Multinu  
 PULPROG zg  
 TD 39046  
 SOLVENT CDCl3

NS 4  
 DS 0  
 SWH 4807.692 Hz  
 FIDRES 0.123129 Hz  
 AQ 4.0508339 sec

RG 18  
 DW 104.000 usec  
 DE 7.00 usec  
 TE 300.0 K

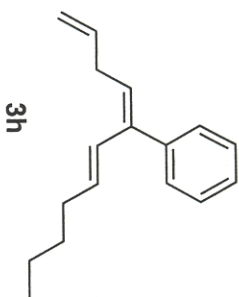
D1 2.0000000 sec  
 P1 7.70 usec  
 SF01 400.1317512 MHz  
 FIDC1 1H  
 PL1 -6.00 dB

F2 - Processing parameters  
 SI 32768  
 SF 400.1300267 MHz  
 MDM EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.384 ppm  
 F1 4155.07 Hz  
 F2P -1.631 ppm  
 F2 -652.63 Hz  
 PPMCM 0.60077 ppm/cm  
 HZCM 240.38463 Hz/cm

Current Data Parameters  
 NAME haploindole  
 EXPNO 22  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020212

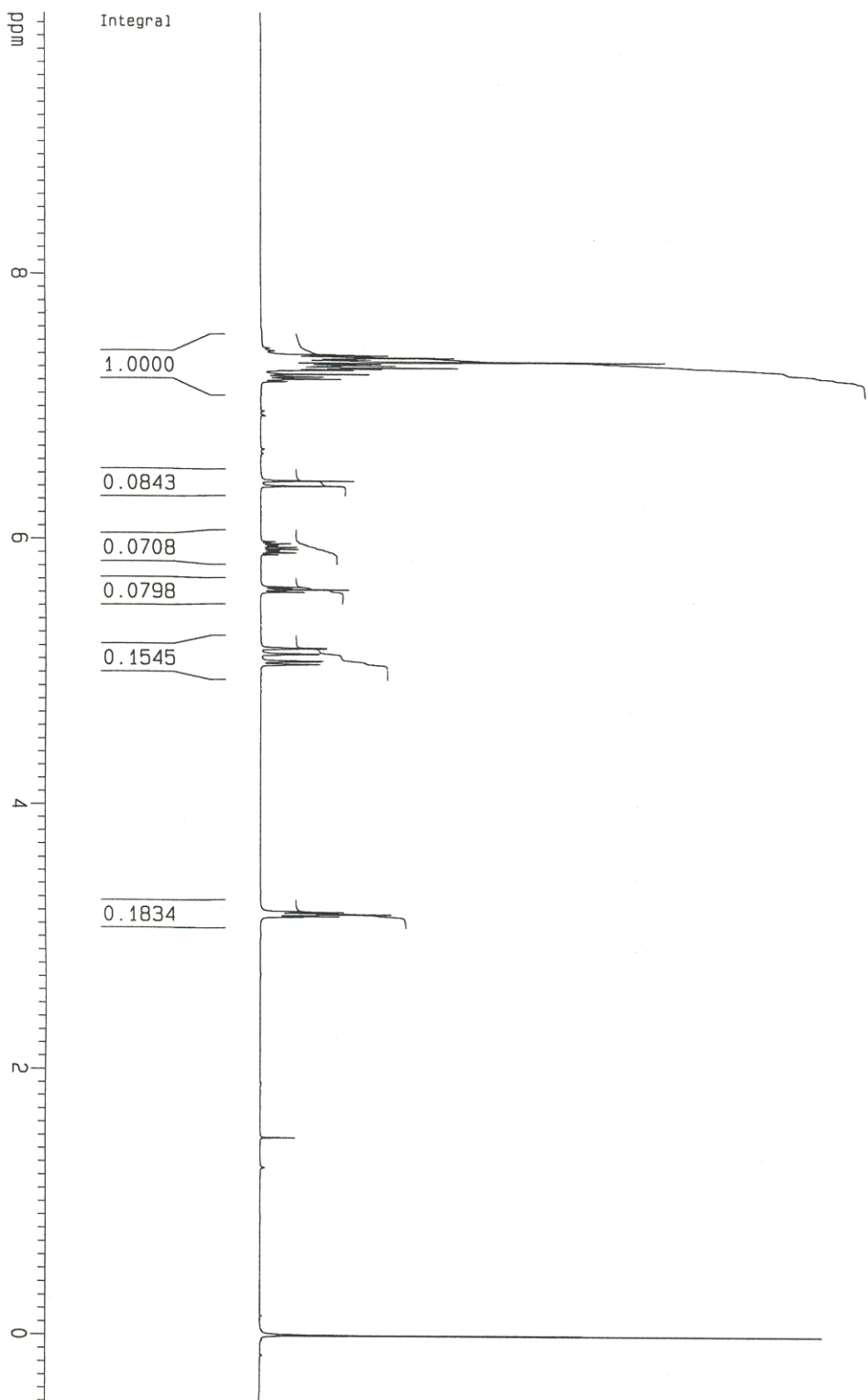
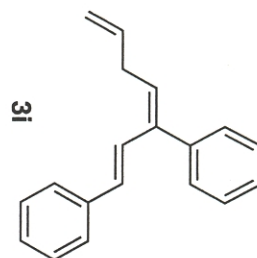


Time 10.23  
 INSTRUM spect  
 PROBD 5 mm Multinu  
 PULPROG zgpg  
 TD 32768  
 SOLVENT C6D6  
 NS 294  
 DS 0  
 SWH 30303.031 Hz  
 FIDRES 0.924775 Hz  
 AQ 0.5407220 sec  
 RG 574.7  
 DW 16.500 usec  
 DE 7.50 usec  
 TE 300.0 K  
 d11 0.03000000 sec  
 PL12 20.00 dB  
 CPDPRG2 waltz16  
 PCPD2 105.00 usec  
 SFO2 400.1329209 MHz  
 NUC2 1H  
 PL2 120.00 dB  
 D1 1.50000000 sec  
 P1 6.80 usec  
 SFO1 100.6223610 MHz  
 NUC1 13C  
 PL1 -6.00 dB

F2 - Processing parameters  
 SI 16384  
 SF 100.6127735 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 245.883 ppm  
 F1 24739.00 Hz  
 F2P -55.301 ppm  
 F2 -5564.03 Hz  
 PPMCM 15.05924 ppm/cm  
 HZCM 1515.15149 Hz/cm





Current Data Parameters  
 NAME haploindole  
 EXPNO 206  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020219  
 Time 15.05

INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0

SWH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.9059956 sec

RG 28.5  
 DW 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB

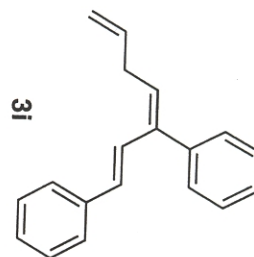
SFO1 400.1319246 MHz

F2 - Processing parameters

SI 16384  
 SF 400.1300303 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

10 NMR plot parameters

CX 20.00 cm  
 F1P 9.976 ppm  
 F1 3991.61 Hz  
 F2P -0.507 ppm  
 F2 -203.02 Hz  
 PPMCM 0.52416 ppm/cm  
 HZCM 209.73155 Hz/cm



Current Data Parameters  
 NAME haploindole  
 EXPNO 207  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020219

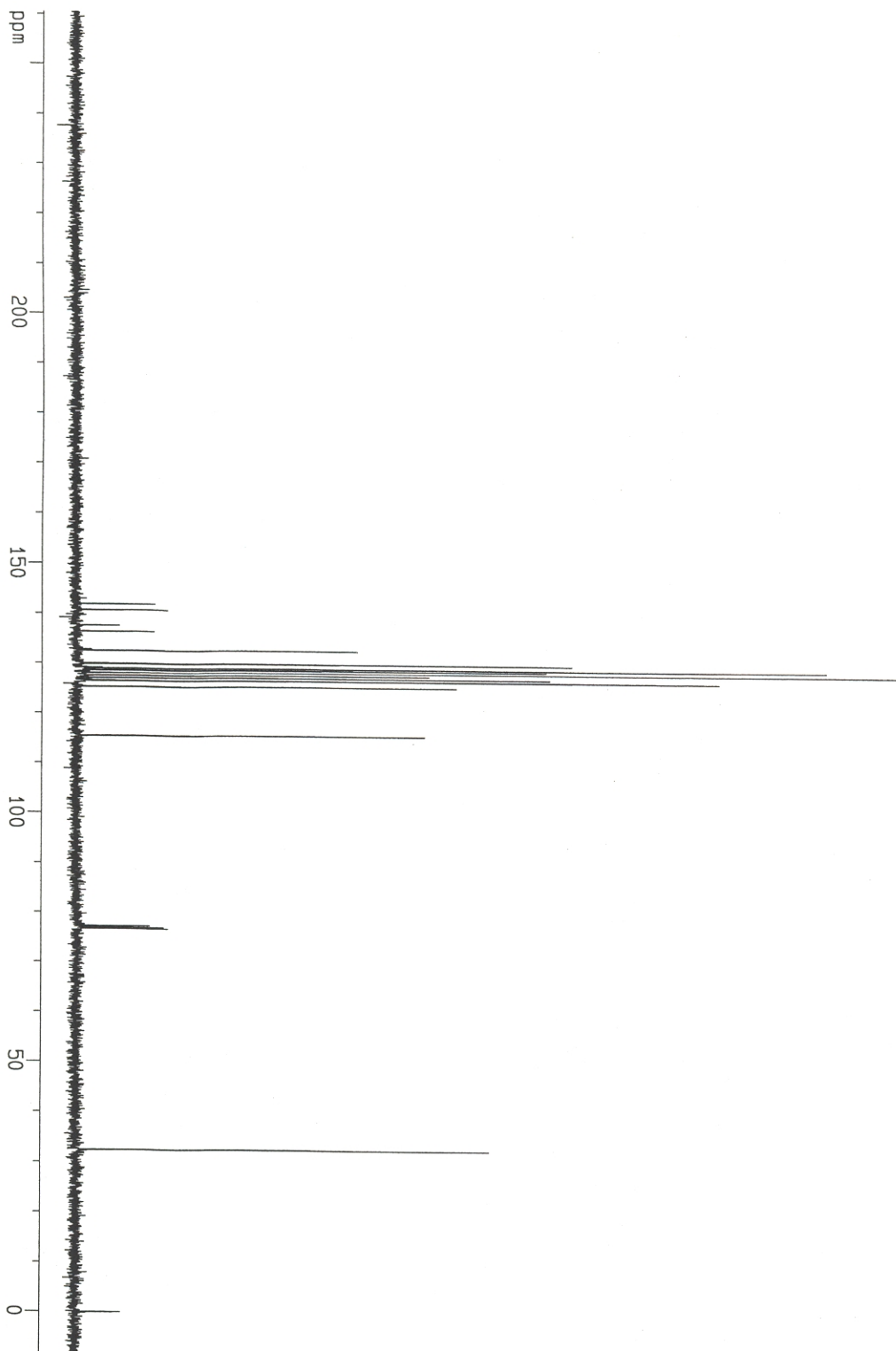
Time 15.11  
 INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg  
 TO 32768  
 SOLVENT CDCl3  
 NS 135  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 2298.8  
 DW 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

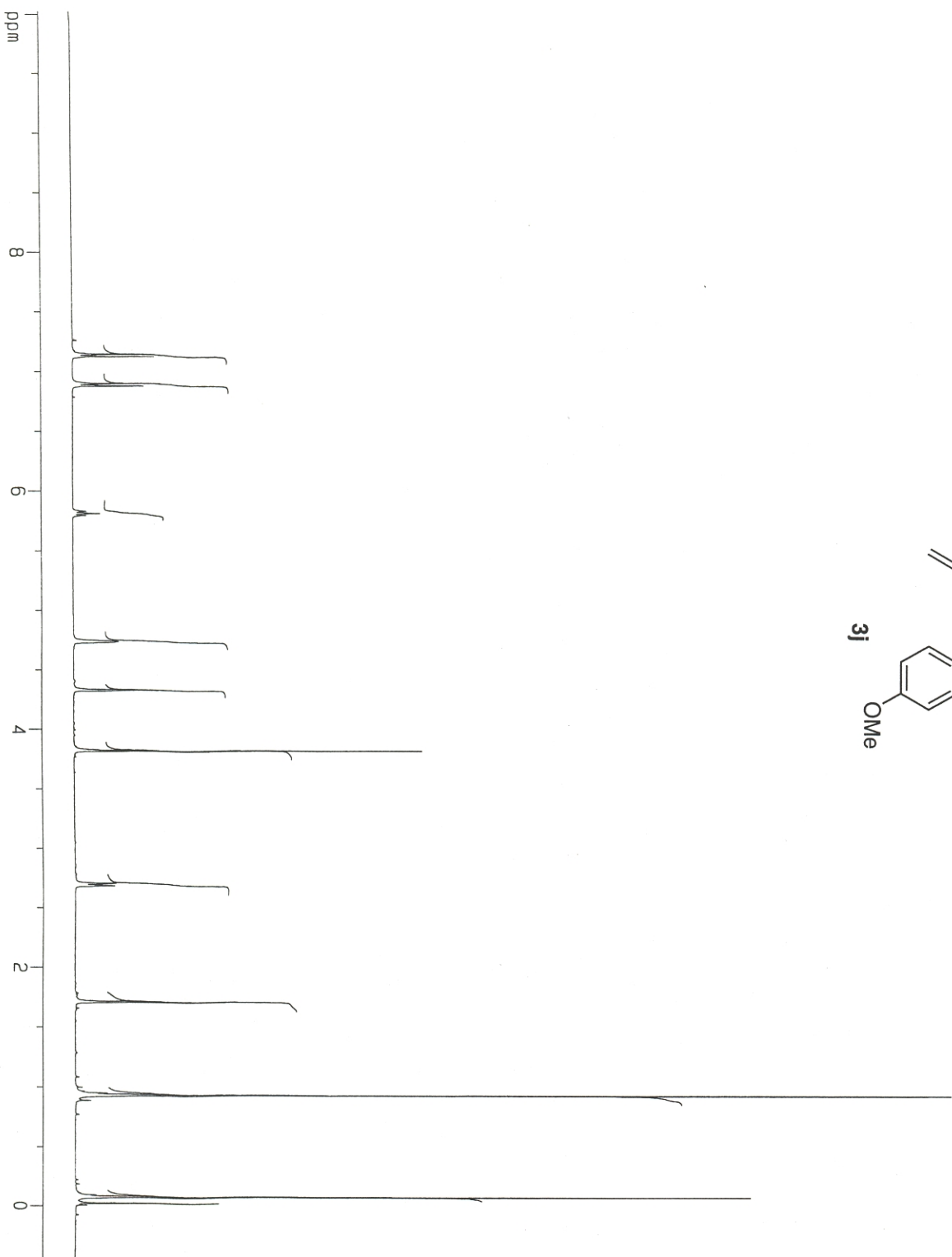
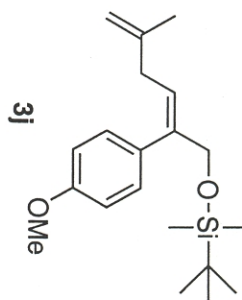
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127777 MHz  
 WDM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.486 ppm  
 F1 26208.25 Hz  
 F2P -8.866 ppm  
 F2 -892.02 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01367 Hz/cm





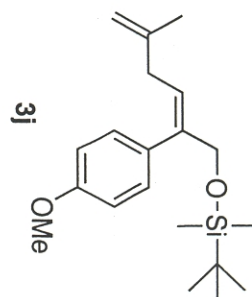
Current Data Parameters  
 NAME haploindole  
 EXPNO 148  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020103  
 Time 12.15  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 4  
 DS 0  
 SMH 4194.631 Hz  
 FIDRES 0.128010 Hz  
 AQ 3.905956 sec  
 RG 16  
 DM 119.200 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 10.50 usec  
 PL1 0.00 dB  
 SFO1 400.1319246 MHz

F2 - Processing parameters  
 SI 16384  
 SF 400.1300094 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.028 ppm  
 F1 4012.49 Hz  
 F2P -0.455 ppm  
 F2 -182.14 Hz  
 PPMCH 0.52416 ppm/cm  
 HZCM 209.73155 Hz/cm



Current Data Parameters  
 NAME hapi0indole  
 EXPNO 149  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020103  
 Time 12.19

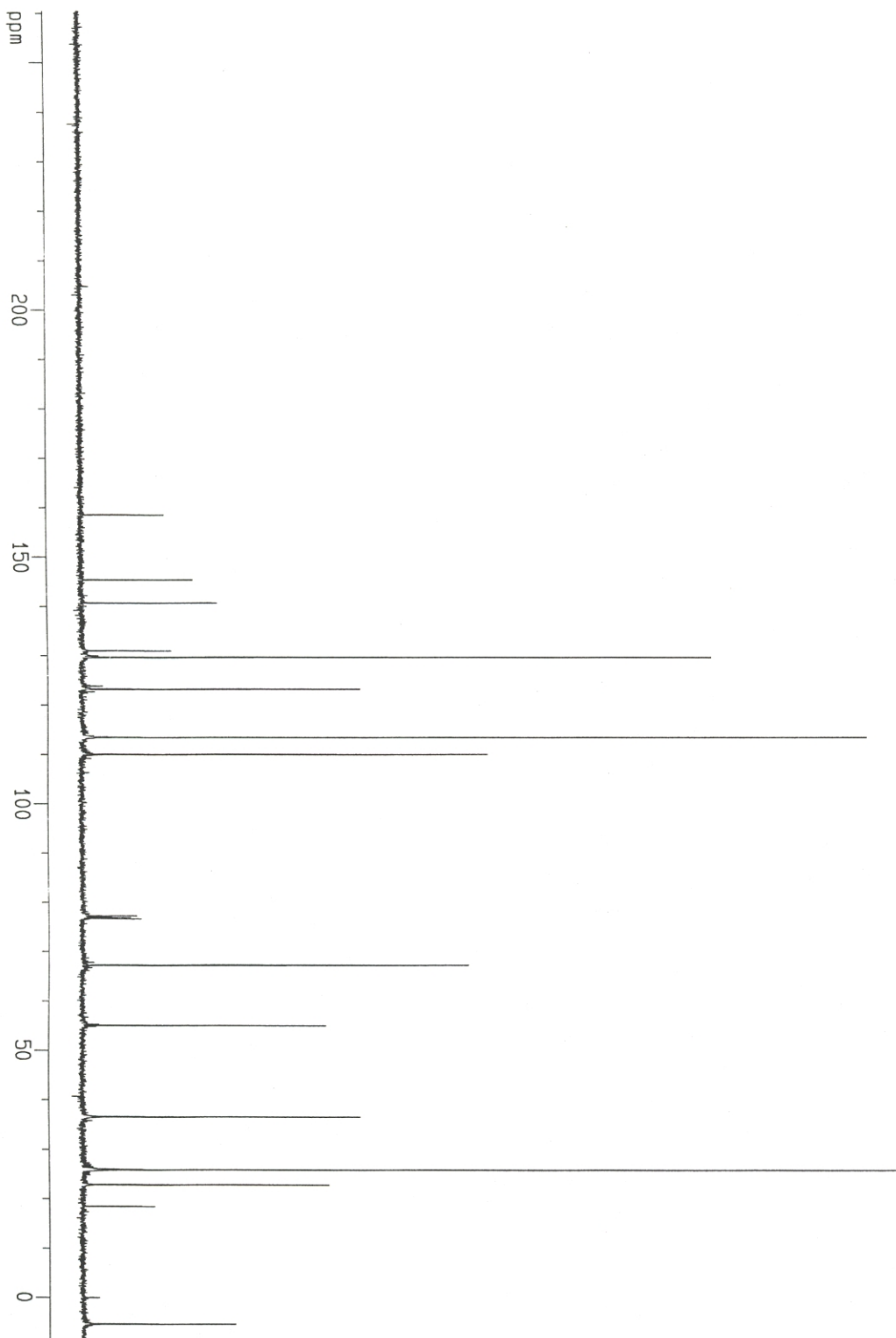
INSTRUM spect  
 PROBD 5 mm QNP 1H  
 PULPROG zgpg  
 TO 32768  
 SOLVENT CDCl3  
 NS 322  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 3251  
 DM 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

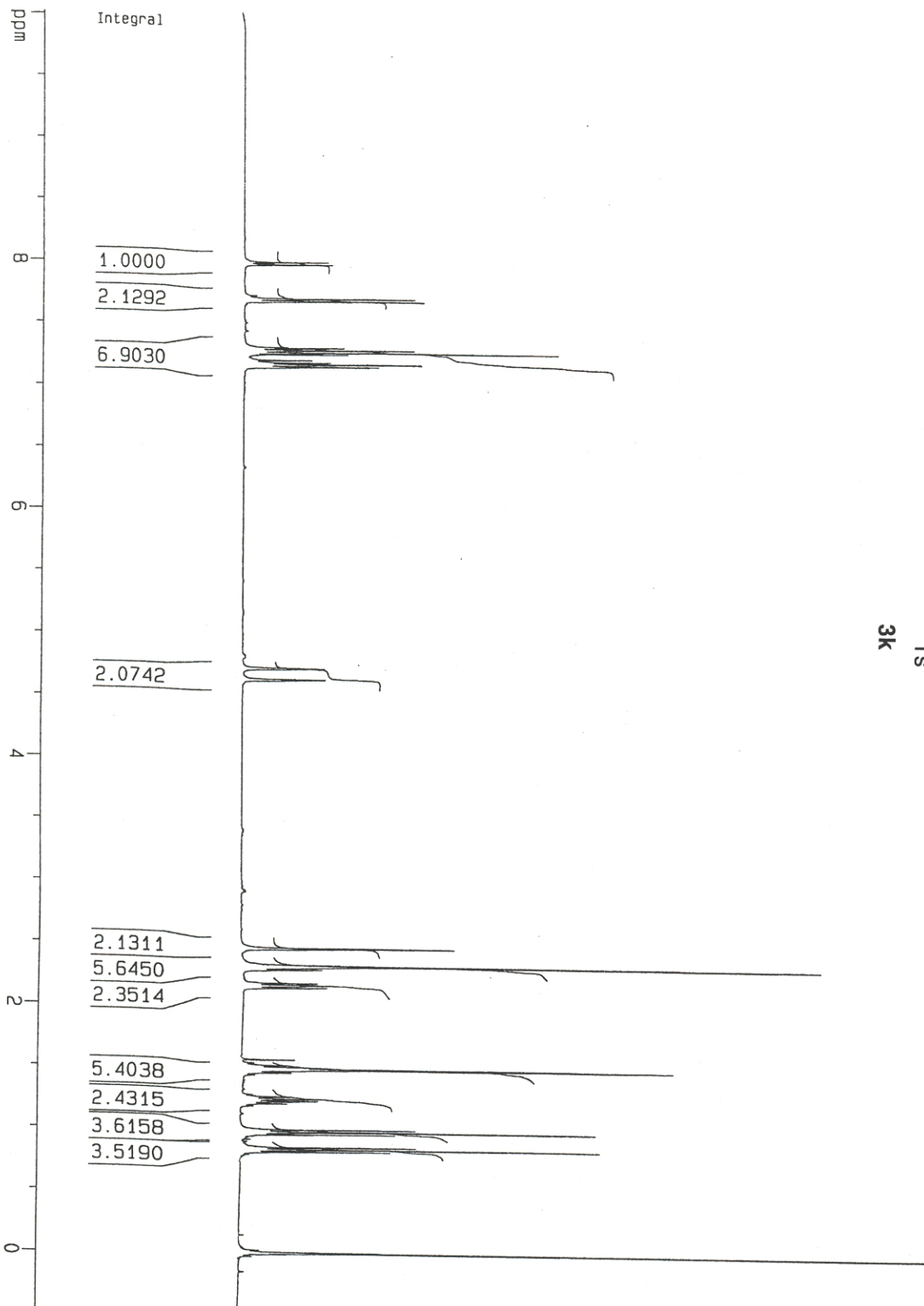
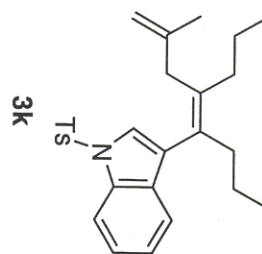
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127760 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.503 ppm  
 F1 26209.90 Hz  
 F2P -8.849 ppm  
 F2 -890.37 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01355 Hz/cm





Current Data Parameters  
NAME haploindole  
EXPNO 176  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20020128

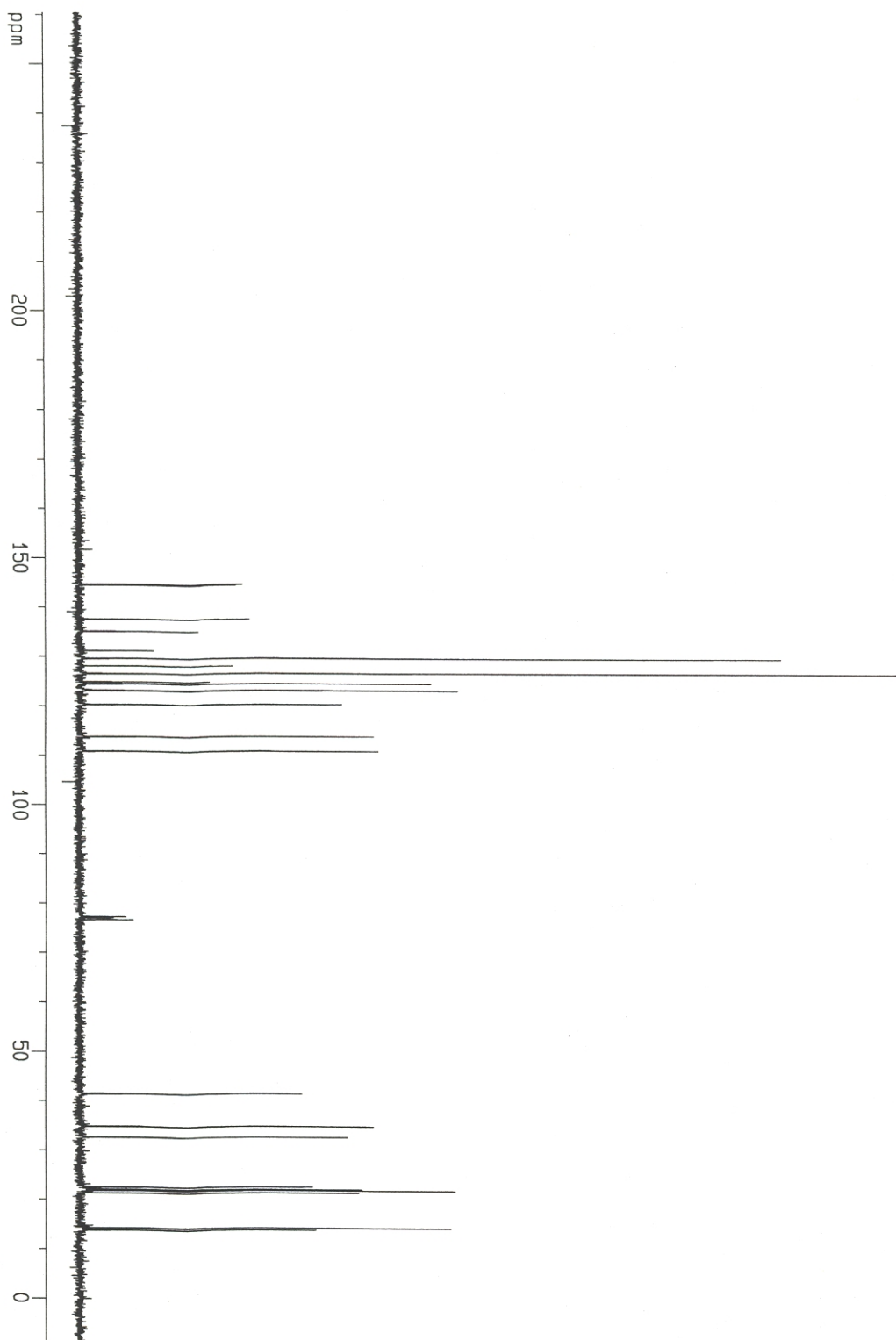
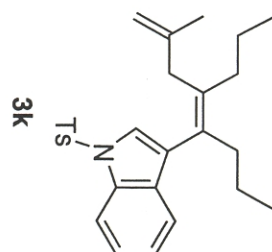
Time 15.35  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zg  
TD 32768  
SOLVENT CDCl3  
NS 4  
DS 0  
SMH 4194.631 Hz  
FIDRES 0.128010 Hz  
AQ 3.905956 sec  
RG 40.3  
DW 119.200 usec  
DE 4.50 usec  
TE 300.0 K  
D1 2.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.50 usec  
PL1 0.00 dB  
SF01 400.1319246 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300121 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.021 ppm  
F1 4009.78 Hz  
F2P -0.462 ppm  
F2 -184.85 Hz  
PPMCM 0.52416 ppm/cm  
HZCM 209.73155 Hz/cm





Current Data Parameters  
 NAME naploindo1e  
 EXPNO 177  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20020128  
 Time 15.04  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H  
 PULPROG zgpg  
 TD 32768  
 SOLVENT CDCl3  
 NS 38  
 DS 0  
 SMH 27100.271 Hz  
 FIDRES 0.827035 Hz  
 AQ 0.6046196 sec  
 RG 4096  
 DW 18.450 usec  
 DE 7.50 usec  
 TE 300.0 K  
 D1 3.00000000 sec  
 d11 0.03000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.00 usec  
 PL1 -2.00 dB  
 SF01 100.6254358 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 101.00 usec  
 PL2 120.00 dB  
 PL12 19.00 dB  
 SF02 400.1324710 MHz

F2 - Processing parameters  
 SI 16384  
 SF 100.6127843 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 260.421 ppm  
 F1 26201.63 Hz  
 F2P -8.932 ppm  
 F2 -898.63 Hz  
 PPMCM 13.46761 ppm/cm  
 HZCM 1355.01343 Hz/cm