

Iterative Approach to Oligo(arylenevinylene)s Containing Tetrasubstituted Vinylene Units

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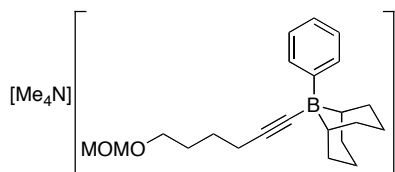
General. NMR spectra were recorded on a Varian Gemini 2000 (^1H at 300 MHz and ^{13}C at 75 MHz), JEOL JNM-A500 (^1H at 500 MHz and ^{13}C at 150 MHz), or Varian 400-MR Auto Tune X5 (^{11}B at 128 MHz) spectrometers. Unless otherwise noted, CDCl_3 was used as a solvent. Chemical Shifts are recorded in δ ppm referenced to a residual CDCl_3 ($\delta = 7.26$ for ^1H , $\delta 77.0$ for ^{13}C), CD_3CN ($\delta = 1.94$ for ^1H , $\delta = 1.32$ for ^{13}C), and $\text{BF}_3 \cdot \text{OEt}_2$ ($\delta = 0.00$ for ^{11}B). High-resolution mass spectra were recorded on Applied Biosystems Voyager Elite or JEOL JMS-HX110A spectrometer. Infrared spectra were recorded on a SHIMADZU FT-IR 8100. Column chromatography was performed with silica gel 60 N (Kanto). Preparative thin-layer chromatography was performed with Silica gel 60 PF₂₅₄ (Merck). Gel permeation chromatography (GPC) was carried out with Japan Analytical Industry LC-908 or LC-9204.

Materials. Unless otherwise noted, all chemicals and anhydrous solvents were obtained from commercial suppliers. Toluene was dried over Na-benzophenone ketyl. (XANTPhos)Pd(π -allyl)Cl,¹ Ar-9-BBN,² and 5-Hexyn-1-yl(methoxymethyl)ether³ were prepared according to the reported procedure.

Preparation of alkynylborates 1a: A typical procedure for the preparation of alkynylborates 1a, 1b

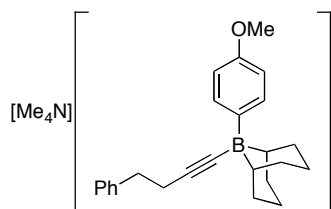
To a stirred solution of 5-hexyn-1-yl(methoxymethyl)ether (2.04 g, 14.4 mmol) in THF (20.0 ml) at -78°C was added *n*-BuLi (1.6 M in hexane, 9.0 ml, 14.4 mmol). After 30 minutes at this temperature, phenyl-9-BBN (2.60 g, 13.0 mmol) was added and the cooling bath was removed. After being stirred for 1 h at room temperature, the reaction was quenched by adding a small amount of methanol. Then, volatile materials were removed under reduced pressure. The residue was dissolved in methanol and tetramethylammonium chloride (1.60 g, 14.6 mmol) was added with stirring at -78°C , resulting in white solid. It was collected by filtration and was washed with cold methanol to give alkynylborate **1a** (3.20 g, 7.7 mmol, 60% yield).

1a



^1H NMR (CD_3CN , 300 MHz): $\delta = 0.91$ (bs, 2H), 1.10-1.19 (m, 1H), 1.32-1.96 (m, 15H), 2.02 (t, $J = 6.6$ Hz, 2H), 2.36-2.51 (m, 2H), 3.04 (s, 12H), 3.28 (s, 3H), 3.46 (t, $J = 6.9$ Hz, 2H), 4.54 (s, 2H), 6.78-6.85 (m, 1H), 6.98-7.05 (m, 2H), 7.34 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (CD_3CN , 75 MHz): $\delta = 21.0, 26.7$ (br), 27.1, 27.5, 28.2, 29.7, 32.4, 34.9, 55.1, 56.1, 68.1, 92.8, 96.8, 122.1, 126.9, 133.8; ^{11}B NMR (128 MHz): $\delta = -18.0$; HRMS (FAB) Calcd for $\text{C}_{22}\text{H}_{32}\text{BO}_2$ [$\text{M}-(\text{NMe}_4)^+$] 339.2495. Found 339.2493.

1b

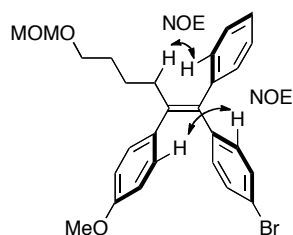


^1H NMR (CD_3CN , 300 MHz): δ = 0.88 (bs, 2H), 1.10-1.20 (m, 1H), 1.34-1.96 (m, 9H), 2.25 (t, J = 7.2 Hz, 2H), 2.34-2.49 (m, 2H), 2.63 (t, J = 7.2 Hz, 2H), 3.03 (s, 12H), 3.72 (s, 3H), 6.63-6.69 (m, 2H), 7.11-7.28 (m, 7H); ^{13}C NMR (CD_3CN , 100 MHz) δ = 24.2, 27.2, 27.5, 32.4, 34.9, 38.1, 55.4, 56.0, 112.9, 126.6, 128.9, 129.8, 134.6, 143.4, 156.3; ^{11}B NMR (128 MHz): δ = -18.2; HRMS (FAB) Calcd for $\text{C}_{25}\text{H}_{30}\text{BO}$ $[\text{M}-(\text{NMe}_4)]^-$ 357.2390. Found 357.2383.

One-pot synthesis of Tetrasubstituted Olefin 3. A typical procedure for the synthesis of tetrasubstituted oligo(arylenevinylene)s 3-12.

Under an argon atmosphere, a mixture of alkynylborate **1**, (XANTPhos)Pd(π -allyl)Cl (X mg, Y mmol), and aryl halide **A** in toluene was stirred for time t_1 at 50 °C. Then, aryl halide **B** and base **C** were added to the reaction mixture, which was stirred for time t_2 and water was added. After addition of water, the aqueous layer was extracted with AcOEt (3 times), washed with water (once), brine (once), dried over Na_2SO_4 and concentrated. The residue was purified by GPC to afford the tetrasubstituted oligo(arylenevinylene)s

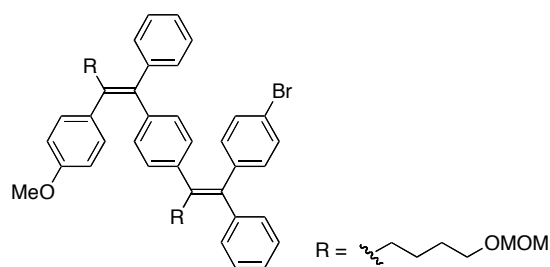
3



alkynylborate **1**: **1a** (827 mg, 2.0 mmol), (XANTPhos)Pd(π -allyl)Cl (15 mg, 20 μmol), aryl halide **A**: 4-bromoanisole (393 mg, 2.1 mmol), aryl halide **B**: 4-bromiodobenzene (566 mg, 2.0 mmol), base **C**: NaOH (240 mg, 6.0 mmol), t_1 : 0.5 h, t_2 : 6 h

^1H NMR (300 MHz): δ = 1.30-1.58 (m, 4H), 2.41 (t, J = 7.8 Hz, 2H), 3.27 (s, 3H), 3.37 (t, J = 6.6 Hz, 2H), 3.76 (s, 3H), 4.52 (s, 2H), 6.67-6.75 (m, 4H), 6.97-7.02 (m, 2H), 7.09-7.36 (m, 7H); ^{13}C NMR (75 MHz): δ = 25.5, 29.8, 35.6, 55.10, 55.14, 67.6, 96.4, 113.4, 119.6, 126.7, 128.2, 129.4, 130.46, 130.49, 132.3, 133.9, 137.6, 140.8, 142.0, 143.0, 158.0; HRMS (MALDI-TOF-MS (DCTB) calcd for $\text{C}_{27}\text{H}_{29}\text{BrO}_3$ $[\text{M}]^+$ 480.1300. Found 480.1269. The (*Z*)-stereochemistry was determined by the 2D NOESY spectroscopy.

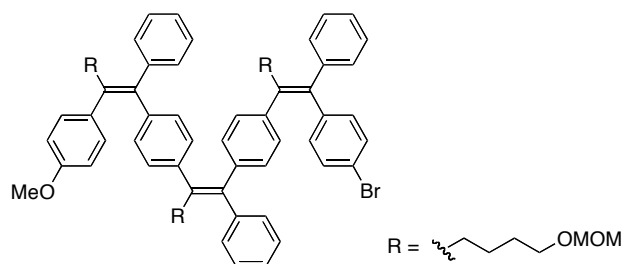
4



alkynylborate **1**: **1a** (863 mg, 2.1 mmol), (XANTPhos)Pd(π -allyl)Cl (15 mg, 20 μ mol), aryl halide **A**: **3** (963 mg, 2.0 mmol), aryl halide **B**: 4-bromiodobenzene (622 mg, 2.2 mmol), base **C**: NaOH (240 mg, 6.0 mmol), t_1 : 0.5 h, t_2 : 6 h

^1H NMR (300 MHz): δ = 1.24-1.54 (m, 8H), 2.33 (t, J = 7.8 Hz, 2H), 2.42 (t, J = 7.5 Hz, 2H), 3.25 (s, 3H), 3.29 (s, 3H), 3.31-3.41 (m, 4H), 3.80 (s, 3H), 4.51 (s, 2H), 4.53 (s, 2H), 6.62-6.78 (m, 8H), 6.97-7.03 (m, 2H), 7.06-7.38 (m, 12H); ^{13}C NMR (150 MHz): δ = 25.2, 25.4, 29.5, 29.6, 35.3, 35.4, 55.02, 55.04, 55.2, 67.5, 67.6, 96.29, 96.32, 113.1, 119.6, 126.5, 126.7, 128.1, 128.2, 128.5, 129.41, 129.42, 130.3, 130.4, 130.6, 132.4, 134.3, 137.7, 138.5, 139.0, 140.0, 141.3, 141.4, 141.8, 142.9, 143.5, 157.9; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{47}\text{H}_{51}\text{BrO}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 797.2818. Found 797.2849.

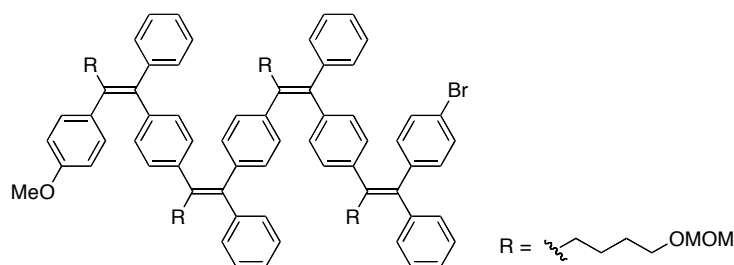
5



alkynylborate **1**: **1a** (434 mg, 1.05 mmol), (XANTPhos)Pd(π -allyl)Cl (7.6 mg, 10 μ mol), aryl halide **A**: **4** (775 mg, 1.0 mmol), aryl halide **B**: 4-bromiodobenzene (310 mg, 1.1 mmol), base **C**: NaOH (120 mg, 3.0 mmol), t_1 : 1 h, t_2 : 12 h

^1H NMR (300 MHz): δ = 1.22-1.55 (m, 12 H), 2.28-2.39 (m, 4H), 2.46 (t, J = 7.5, 2H), 3.25 (s, 3H), 3.27 (s, 3H), 3.28 (s, 3H), 3.30-3.42 (m, 6H), 3.74 (s, 3H), 4.50 (s, 2H), 4.52 (s, 2H), 4.54 (s, 2H), 6.54-6.62 (m, 4H), 6.66-6.78 (m, 8H), 6.96-7.04 (m, 4H), 7.12-7.37 (m, 15H); ^{13}C NMR (75 MHz): δ = 25.4, 25.6, 29.6, 29.7, 29.8, 35.26, 35.34, 35.5, 55.1, 67.5, 67.6, 96.3, 113.0, 119.6, 126.4, 126.7, 128.0, 128.1, 128.6, 129.4, 130.1, 130.2, 130.4, 130.6, 132.4, 134.4, 137.7, 138.4, 138.7, 139.2, 139.3, 139.8, 140.2, 140.9, 141.4, 141.5, 141.7, 142.8, 143.3, 143.5, 157.7; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{67}\text{H}_{73}\text{BrO}_7\text{Na}$ $[\text{M}+\text{Na}]^+$ 1091.4437. Found 1091.4482.

6

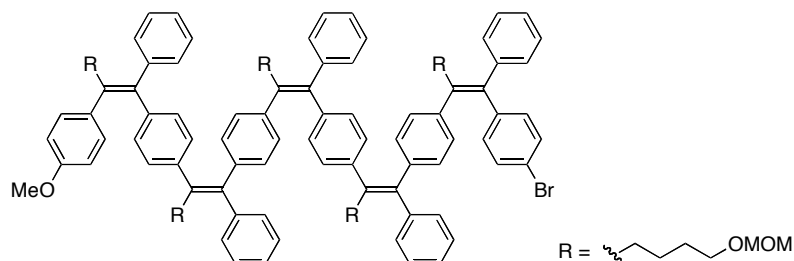


alkynylborate **1**: **1a** (434 mg, 1.05 mmol), (XANTPhos)Pd(π -allyl)Cl (7.6 mg, 10 μ mol), aryl halide **A**: **5** (1.07 mg, 1.0 mmol), aryl halide **B**: 4-bromiodobenzene (310 mg, 1.1 mmol), base **C**: NaOH (120 mg, 3.0 mmol), t_1 : 1.5 h, t_2 : 12 h

^1H NMR (300 MHz): δ = 1.20-1.56 (m, 16H), 2.26-2.48 (m, 8H), 3.22-3.28 (m, 12H), 3.30-3.41 (m, 8H), 3.71 (s, 3H),

4.48-4.54 (m, 8H), 6.48-6.75 (m, 16H), 6.95-7.03 (m, 4H), 7.14-7.37 (m, 20H); ^{13}C NMR (75 MHz): δ = 25.3, 25.4, 25.6, 25.7, 29.69, 29.74, 29.8, 35.0, 35.4, 35.6, 55.0, 55.1, 67.5, 67.6, 96.3, 113.1, 119.6, 126.4, 126.7, 127.9, 128.0, 128.1, 128.3, 128.8, 129.3, 129.4, 130.0, 130.1, 130.2, 130.5, 130.6, 132.3, 134.2, 137.7, 138.5, 138.7, 138.9, 139.1, 139.4, 139.6, 139.7, 140.1, 140.5, 140.9, 141.1, 141.3, 141.4, 141.7, 142.7, 143.2, 143.3, 143.5, 157.7; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{87}\text{H}_{95}\text{BrO}_9\text{Na}$ $[\text{M}+\text{Na}]^+$ 1385.6057. Found 1385.6062.

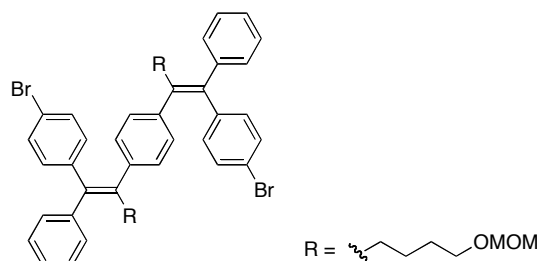
7



alkynylborate **1**: **1a** (227 mg, 0.55 mmol), (XANTPhos)Pd(π -allyl)Cl (3.8 mg, 5.0 μmol), aryl halide **A**: **6** (682 mg, 0.50 mmol), aryl halide **B**: 4-bromoiodobenzene (170 mg, 0.60 mmol), base **C**: NaOH (60 mg, 1.5 mmol), t_1 : 1.5 h, t_2 : 12 h

^1H NMR (300 MHz): δ = 1.18-1.55 (m, 20H), 2.24-2.48 (m, 10H), 3.22-3.40 (m, 25H), 3.74 (s, 3H), 4.47-4.52 (m, 10 H), 6.49-6.79 (m, 20 H), 6.90-6.95 (m, 2H), 7.01-7.06 (m, 2H), 7.10-7.36 (m, 25H); ^{13}C NMR (75 MHz): δ = 25.29, 25.35, 25.6, 25.7, 29.68, 29.71, 29.8, 35.1, 35.4, 35.5, 35.6, 55.0, 67.46, 67.55, 67.6, 96.3, 113.1, 119.5, 126.4, 126.7, 127.95, 128.0, 128.1, 128.3, 128.5, 128.7, 129.3, 129.37, 129.44, 130.1, 130.2, 130.4, 130.6, 132.2, 134.3, 137.7, 138.4, 138.7, 138.9, 139.0, 139.1, 139.4, 139.76, 139.79, 140.1, 140.4, 140.8, 140.9, 141.2, 141.3, 141.6, 142.7, 143.18, 143.21, 143.4, 143.5, 157.8; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{107}\text{H}_{117}\text{BrO}_{11}\text{Na}$ $[\text{M}+\text{Na}]^+$ 1679.7677. Found 1679.7694.

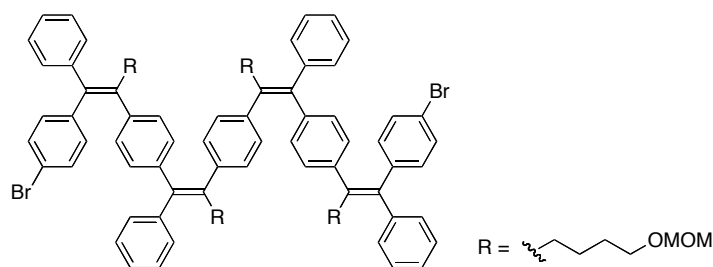
8



alkynylborate **1**: **1a** (868 mg, 2.1 mmol), (XANTPhos)Pd(π -allyl)Cl (7.6 mg, 10 μmol), aryl halide **A**: 1,4-dibromobenzene (236 mg, 1.0 mmol), aryl halide **B**: 4-bromoiodobenzene (622 mg, 2.2 mmol), base **C**: NaOH (240 mg, 6.0 mmol), t_1 : 1.0 h, t_2 : 24 h

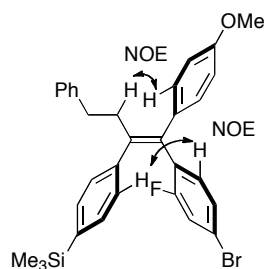
^1H NMR (300 MHz): δ = 1.28-1.54 (m, 8H), 2.40 (t, J = 7.8 Hz, 4H), 3.30 (s, 6H), 3.39 (t, J = 6.6 Hz, 4H), 4.56 (s, 4H), 6.68-6.73 (m, 4H), 6.91 (s, 4H), 7.10-7.21 (m, 8H), 7.22-7.37 (m, 6H); ^{13}C NMR (75 MHz): δ = 25.4, 29.7, 35.4, 55.1, 67.6, 96.3, 119.8, 126.8, 128.2, 129.1, 129.3, 130.3, 132.3, 138.0, 140.0, 141.1, 141.7, 142.7; HRMS (MALDI-TOF-MS (DCTB)) calcd for $\text{C}_{46}\text{H}_{48}\text{Br}_2\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 822.1919. Found 822.1905.

9



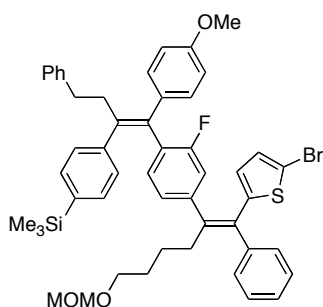
alkynylborate **1**: **1a** (62.0 mg, 0.15 mmol), (XANTPhos)Pd(π -allyl)Cl (0.80 mg, 1.0 μ mol), aryl halide **A**: **8** (41.2 mg, 0.050 mmol), aryl halide **B**: 4-bromiodobenzene (56.6 mg, 0.20 mmol), base **C**: NaOH (18.0 mg, 4.5 mmol), t_1 : 2.0 h, t_2 : 24 h
 ^1H NMR (300 MHz): δ = 1.22-1.54 (m, 16H), 2.29-2.45 (m, 8H), 3.22 (s, 6H), 3.29 (s, 6H), 3.30-3.40 (m, 8H), 4.46 (s, 4H), 4.53 (s, 4H), 6.56-6.72 (m, 12H), 6.88 (s, 4H), 7.04-7.10 (m, 4H), 7.14-7.18 (m, 4H), 7.22-7.40 (m, 16H); ^{13}C NMR (150 MHz): δ = 25.4, 29.62, 29.63, 35.6, 35.7, 55.01, 55.03, 67.4, 67.6, 96.29, 96.31, 119.7, 126.6, 126.8, 128.1, 128.18, 128.22, 129.0, 129.5, 129.6, 130.4, 130.8, 132.4, 137.9, 138.5, 139.4, 140.3, 140.5, 140.9, 141.6, 141.7, 142.7, 143.4; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{86}\text{H}_{92}\text{Br}_2\text{O}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 1433.5057. Found 1433.5040.

10



alkynylborate **1**: **1b** (432 mg, 1.0 mmol), (XANTPhos)Pd(π -allyl)Cl (7.6 mg, 10 μ mol), aryl halide **A**: 4-bromotrimethylsilylbenzene (241 mg, 1.05 mmol), aryl halide **B**: 4-bromo-2-fluoriodobenzene (301 mg, 1.0 mmol), base **C**: NaOH (120 mg, 3.0 mmol), t_1 : 0.5 h, t_2 : 12 h
 ^1H NMR (300 MHz): δ = 0.24 (s, 9H), 2.60-2.67 (m, 2H), 2.76-2.83 (m, 2H), 3.80 (s, 3H), 6.76-6.86 (m, 3H), 6.94-7.07 (m, 6H), 7.09-7.28 (m, 5H), 7.34 (d, J = 8.4 Hz, 2H); ^{13}C NMR (150 MHz): δ = -1.1, 34.9, 37.2, 55.2, 113.5, 118.9 (d, $J_{\text{C-F}}$ = 25.5 Hz), 120.2 (d, $J_{\text{C-F}}$ = 9.2 Hz), 125.8, 126.8 (d, $J_{\text{C-F}}$ = 3.5 Hz), 128.0, 128.3, 128.4, 130.0, 130.4 (d, $J_{\text{C-F}}$ = 15.9 Hz), 132.6, 132.9, 133.2 (d, $J_{\text{C-F}}$ = 4.5 Hz), 133.9, 138.7, 141.7, 141.8, 143.0, 158.5, 159.6 (d, $J_{\text{C-F}}$ = 249.3 Hz); HRMS (MALDI-TOF-MS (DCTB)) calcd for $\text{C}_{32}\text{H}_{32}\text{BrFOSi}$ $[\text{M}]^+$ 558.1390. Found 558.1392. The (*Z*)-stereochemistry was determined by the 2D NOESY spectroscopy.

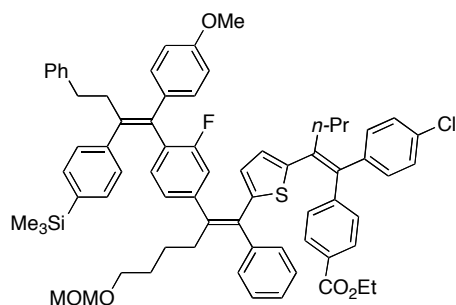
11



alkynylborate **1**: **1a** (219 mg, 0.53 mmol), (XANTPhos)Pd(π -allyl)Cl (3.8 mg, 5.0 μ mol), aryl halide **A**: **10** (279 mg, 0.50

mmol), aryl halide **B**: 2,5-dibromothiophene (406 mg, 1.5 mmol), base **C**: NaOH (60 mg, 1.5 mmol), t_1 : 0.5 h, t_2 : 12 h
 ^1H NMR (300 MHz): δ = 0.24 (s, 9H), 1.16-1.26 (m, 2H), 1.30-1.42 (m, 2H), 2.11 (t, J = 7.8, 2H), 2.64-2.72 (m, 2H), 2.82-2.89 (m, 2H), 3.25 (s, 3H), 3.28 (t, J = 6.6 Hz, 2H), 3.82 (s, 3H), 4.48 (s, 2H), 5.87 (d, J = 3.9 Hz, 1H), 6.60 (d, J = 3.9 Hz, 1H), 6.69-6.76 (m, 2H), 6.84-6.96 (m, 3H), 7.05-7.42 (m, 16H); ^{13}C NMR (150 MHz): δ = -1.0, 24.4, 29.3, 35.1, 36.2, 37.2, 55.0, 55.2, 67.3, 96.3, 112.7, 113.5, 116.1 (d, $J_{\text{C-F}}$ = 22.5 Hz), 124.4 (d, $J_{\text{C-F}}$ = 3.2 Hz), 125.8, 127.4, 128.27, 128.31, 128.37, 128.40, 128.56, 128.61, 129.5, 130.2, 130.7 (d, $J_{\text{C-F}}$ = 15.5 Hz), 132.4, 132.9, 133.0 (d, $J_{\text{C-F}}$ = 4.5 Hz), 133.3, 134.5, 138.5, 139.5 (d, $J_{\text{C-F}}$ = 1.2 Hz), 141.2, 141.6 (d, $J_{\text{C-F}}$ = 8.0 Hz), 141.9, 142.2, 142.3, 146.2, 158.4, 160.2 (d, $J_{\text{C-F}}$ = 246.5 Hz); HRMS (MALDI-TOF-MS (DCTB)) calcd for $\text{C}_{50}\text{H}_{52}\text{BrFO}_3\text{SSiNa}$ $[\text{M}+\text{Na}]^+$ 858.2574. Found 858.2600.

12



alkynylborate **1**: **1c** (39.3 mg, 0.105 mmol), (XANTPhos)Pd(π -allyl)Cl (0.80 mg, 1.0 μmol), aryl halide **A**: **11** (86.0 mg, 0.10 mmol), aryl halide **B**: 4-(ethoxycarbonyl)iodobenzene (30.4 mg, 0.11 mmol), base **C**: Cs_2CO_3 (97.7 mg, 0.30 mmol), t_1 : 1.0 h, t_2 : 12 h

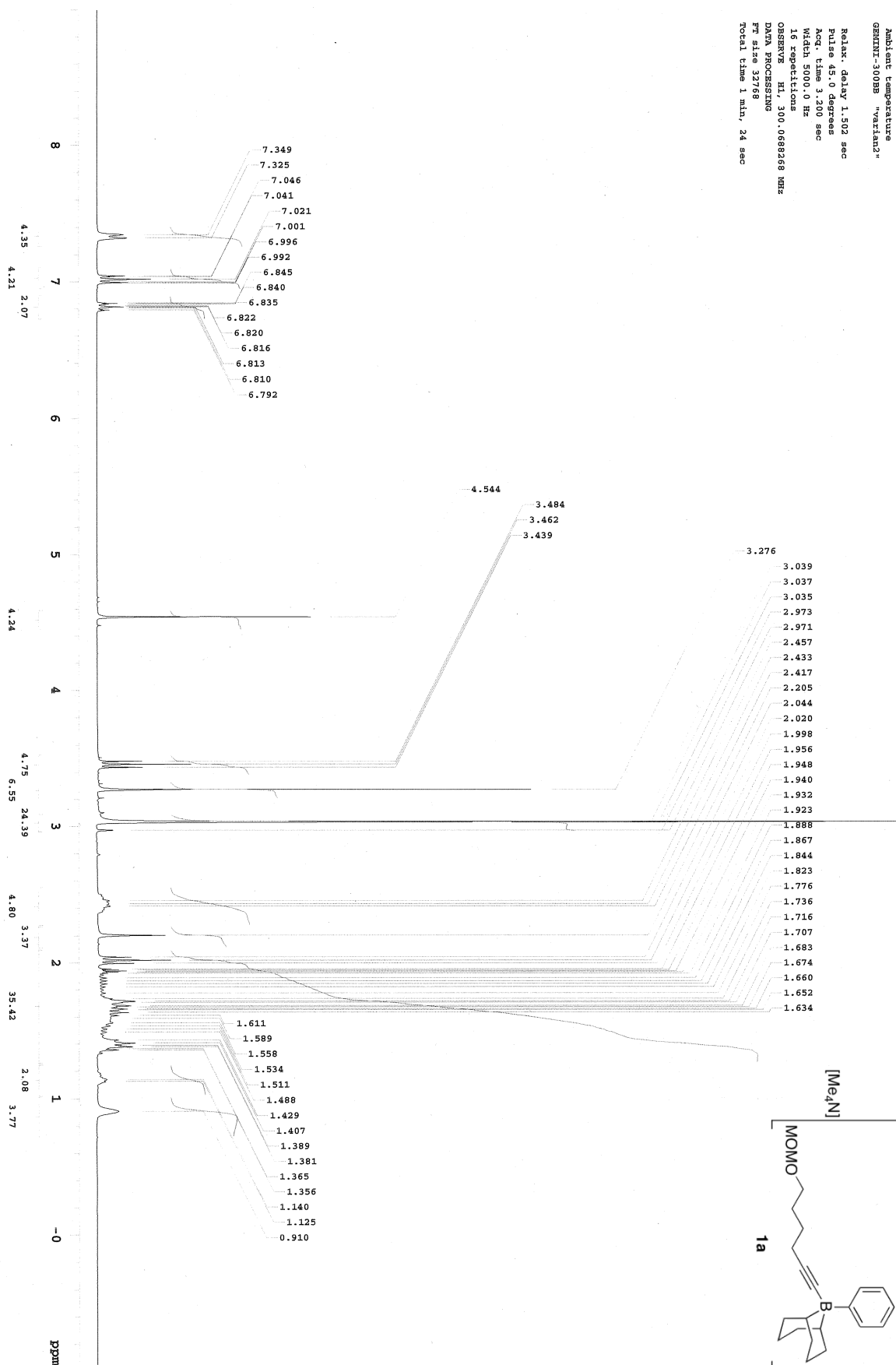
^1H NMR (300 MHz): δ = 0.21 (s, 9H), 0.77 (t, J = 7.2 Hz, 3H), 1.12-1.43 (m, 9H), 2.10-2.24 (m, 4H), 2.64-2.72 (m, 2H), 2.79-2.87 (m, 2H), 3.23-3.33 (m, 5H), 3.73 (s, 3H), 4.34 (q, J = 6.9 Hz, 2H), 4.51 (s, 2H), 5.80 (d, J = 3.6 Hz, 1H), 6.13 (d, J = 3.9 Hz, 1H), 6.66-6.72 (m, 2H), 6.79-6.94 (m, 5H), 7.03-7.37 (m, 20H), 7.73 (d, J = 8.4 Hz, 2H); ^{13}C NMR (150 MHz): δ = -1.1, 14.1, 14.3, 22.5, 24.4, 29.3, 35.0, 35.9, 37.0, 38.5, 55.0, 55.1, 60.7, 67.4, 96.3, 113.4, 115.9 (d, $J_{\text{C-F}}$ = 22.4 Hz), 124.3 (d, $J_{\text{C-F}}$ = 2.9 Hz), 125.8, 126.9, 127.1, 128.08, 128.11, 128.15, 128.20, 128.23, 128.38, 128.44, 129.0, 129.3, 130.0, 130.1 (d, $J_{\text{C-F}}$ = 15.8 Hz), 130.2, 130.7, 132.3 (d, $J_{\text{C-F}}$ = 4.7 Hz), 132.58, 132.61, 132.8, 133.6, 134.4, 135.6, 137.9, 138.2, 139.0 (d, $J_{\text{C-F}}$ = 1.2 Hz), 140.9, 141.90, 141.91, 142.16 (d, $J_{\text{C-F}}$ = 8.0 Hz), 142.19, 142.22, 143.8, 144.6, 147.6, 158.3, 160.0 (d, $J_{\text{C-F}}$ = 244.8 Hz), 166.4; IR (KBr) = 2955, 1717, 1509, 1273, 1248, 1111, 837 cm^{-1} ; HRMS (MALDI-TOF-MS (DIT)) calcd for $\text{C}_{70}\text{H}_{72}\text{ClFO}_5\text{SSiNa}$ $[\text{M}+\text{Na}]^+$ 1129.4440. Found 1129.4465.

1. Johns, A. M.; Utsunomiya, M.; Incarvito, C. D.; Hartwig, J. F. *J. Am. Chem. Soc.* **2006**, *128*, 1828.
2. Kramer, G. W.; Brown, H. C.; *J. Organomet. Chem.* **1974**, *73*, 1.
3. Sun, W. C.; Ng, C. S.; Prestwich, G. D. *J. Org. Chem.* **1992**, *57*, 132.

STANDARD 1H OBSERVE

Pulse Sequence: zgpg30
Solvent: cdcl3
Ambient Temperature
GEMINI-300SB "varian2"

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.068268 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec



13C OBSERVE

Pulse Sequence: zgpg30

Solvent: cdcl₃

Ambient Temperature
GEMINI-300MHz "Varian2"

Relax. delay 1.198 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

14848 repetitions

OBSERVE C13, 75.453021 MHz

DECOUPLE H1, 300.070309 MHz

Power 37 dB

continuously on

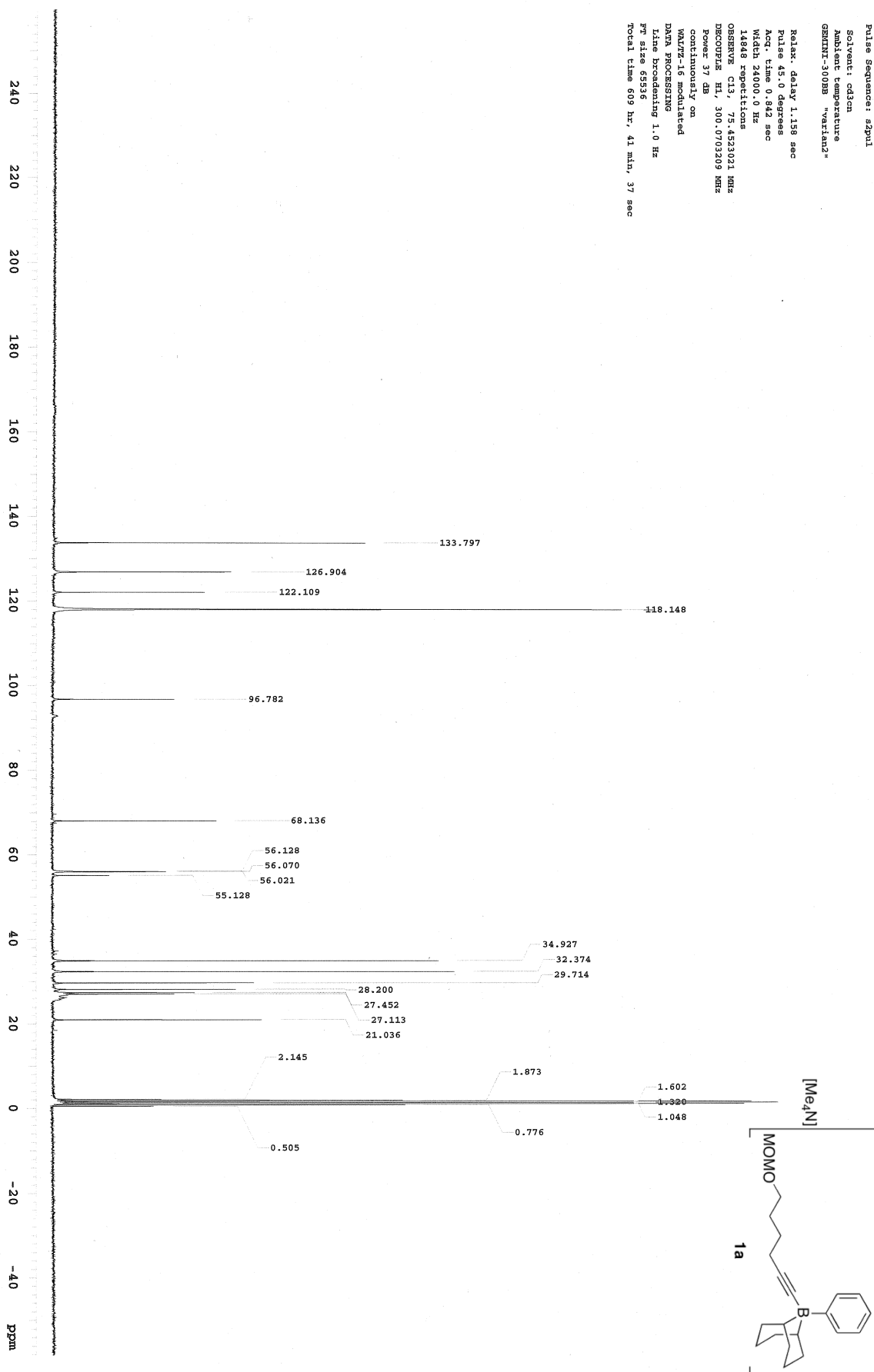
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

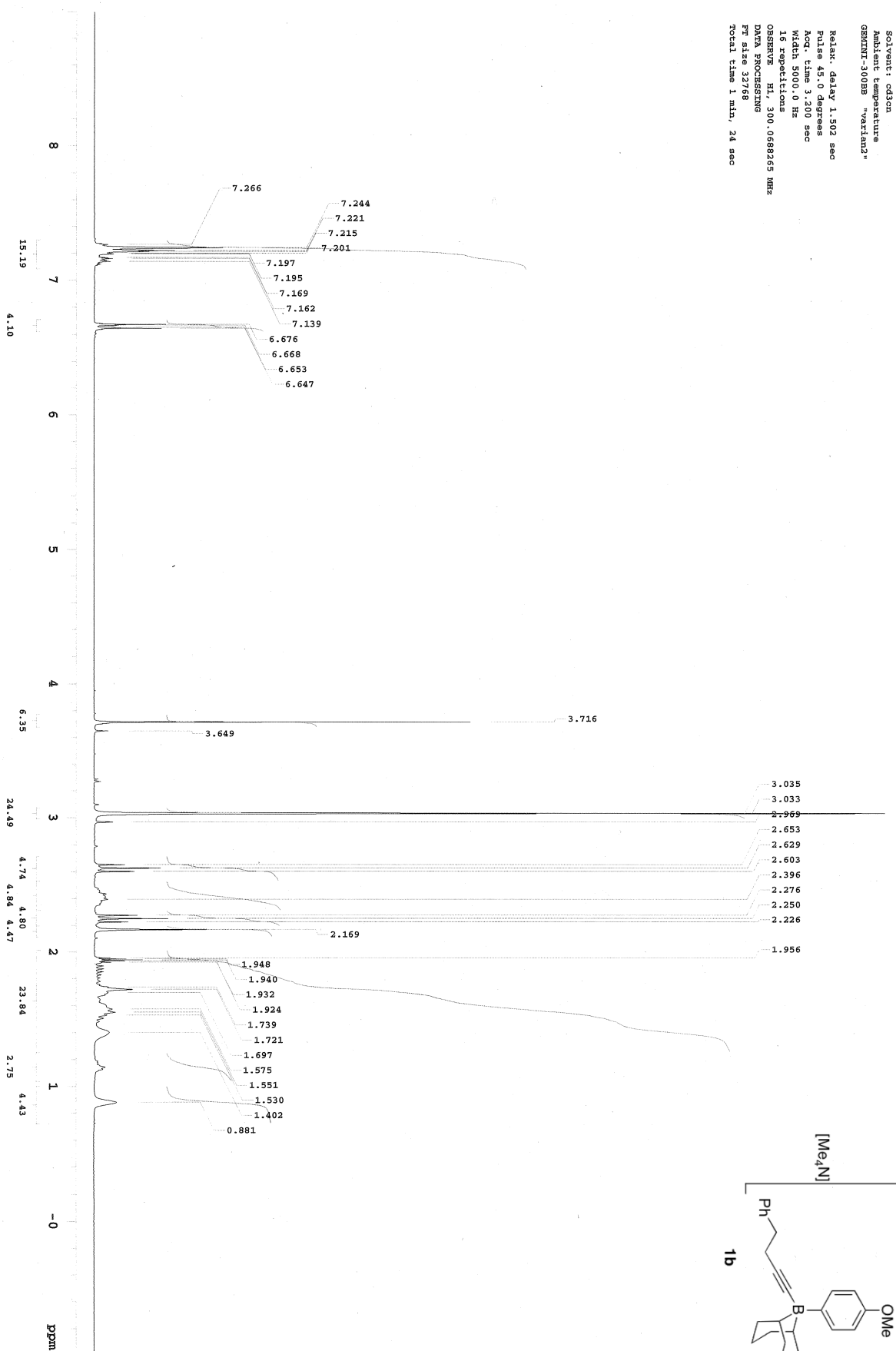
Total time 609 hr, 41 min, 37 sec



STANDARD 1H OBSERVE

Pulse Sequence: zgpg30
Solvent: cd3cn
Ambient temperature
GEMINI-300B8 "varian2"

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.068265 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec



bortate

File: xp

Pulse Sequence: szpul

Solvent: cd3cn

Ambient temperature

Operator: ymm1

Mercury-400HB "Varian-NMR"

Relax. delay 0.700 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 2390.8 Hz

31616 repetitions

OBSERVE C13, 100.621109 MHz

DECOUPLE H1, 400.4479242 MHz

Power 40 dB

continuously on

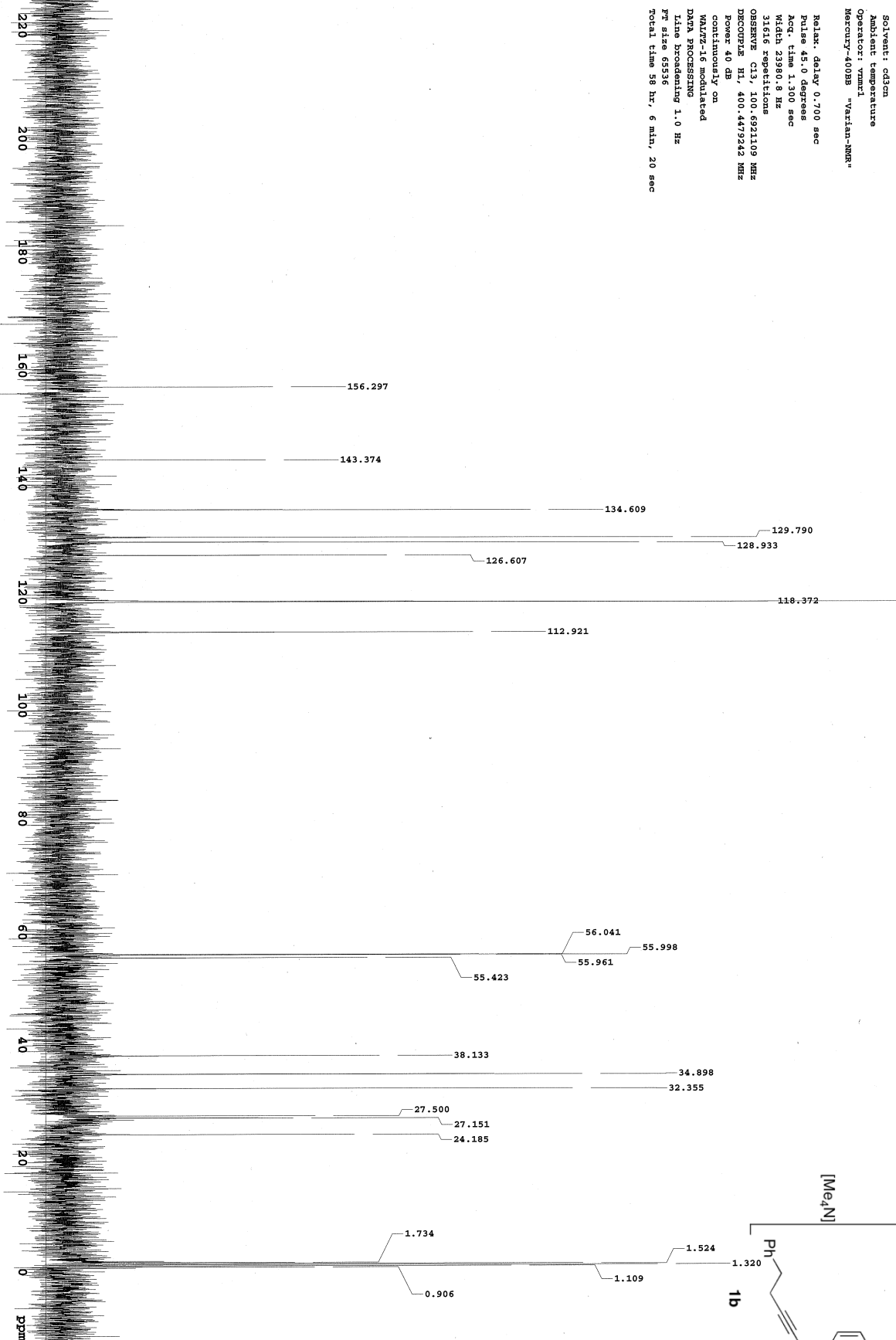
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

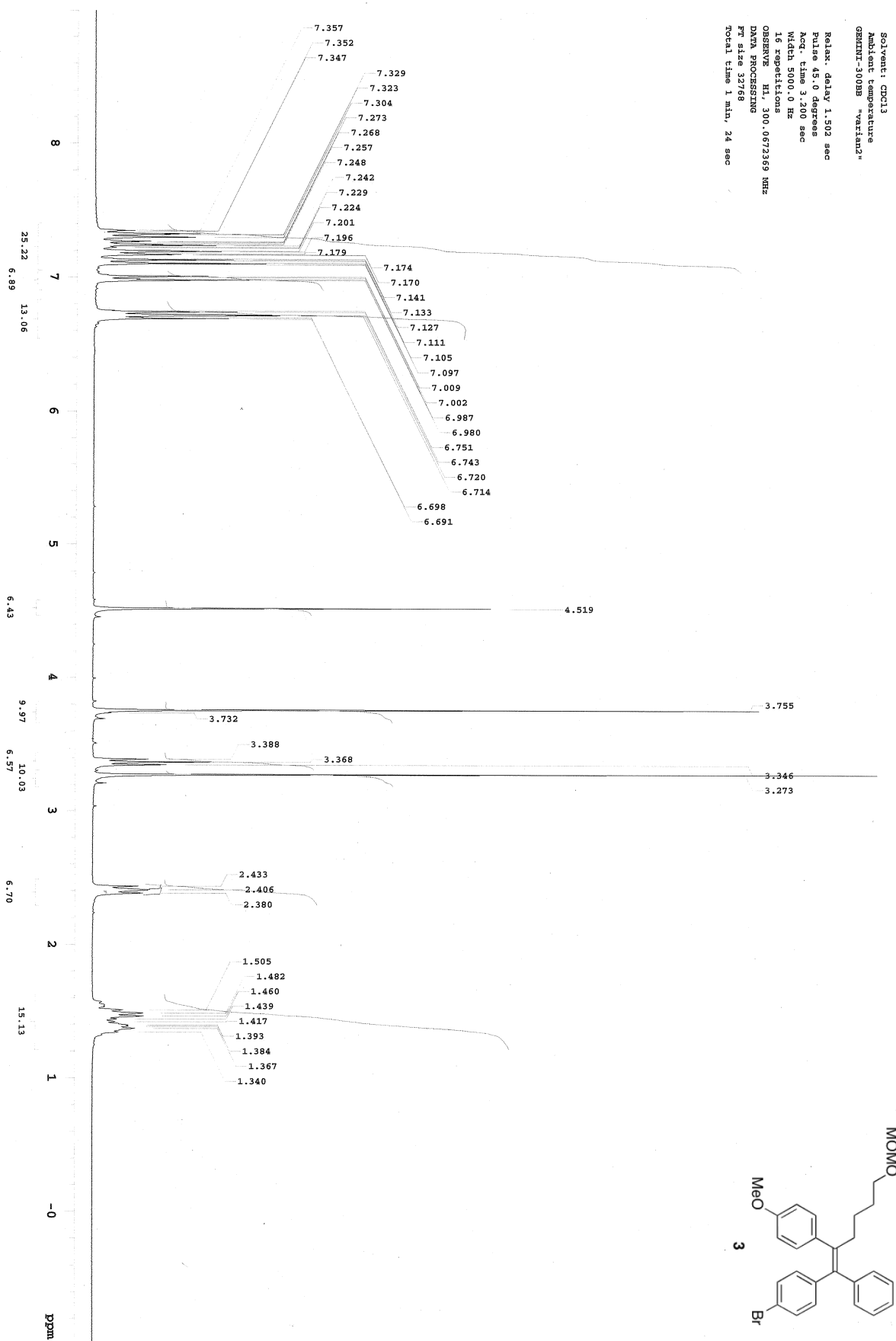
Total time 58 hr, 6 min, 20 sec



STANDARD 1H OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient Temperature
 GEMINT-300MHz "Varian2"

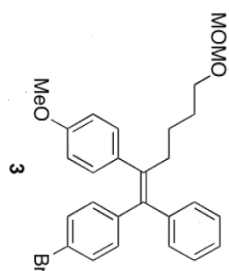
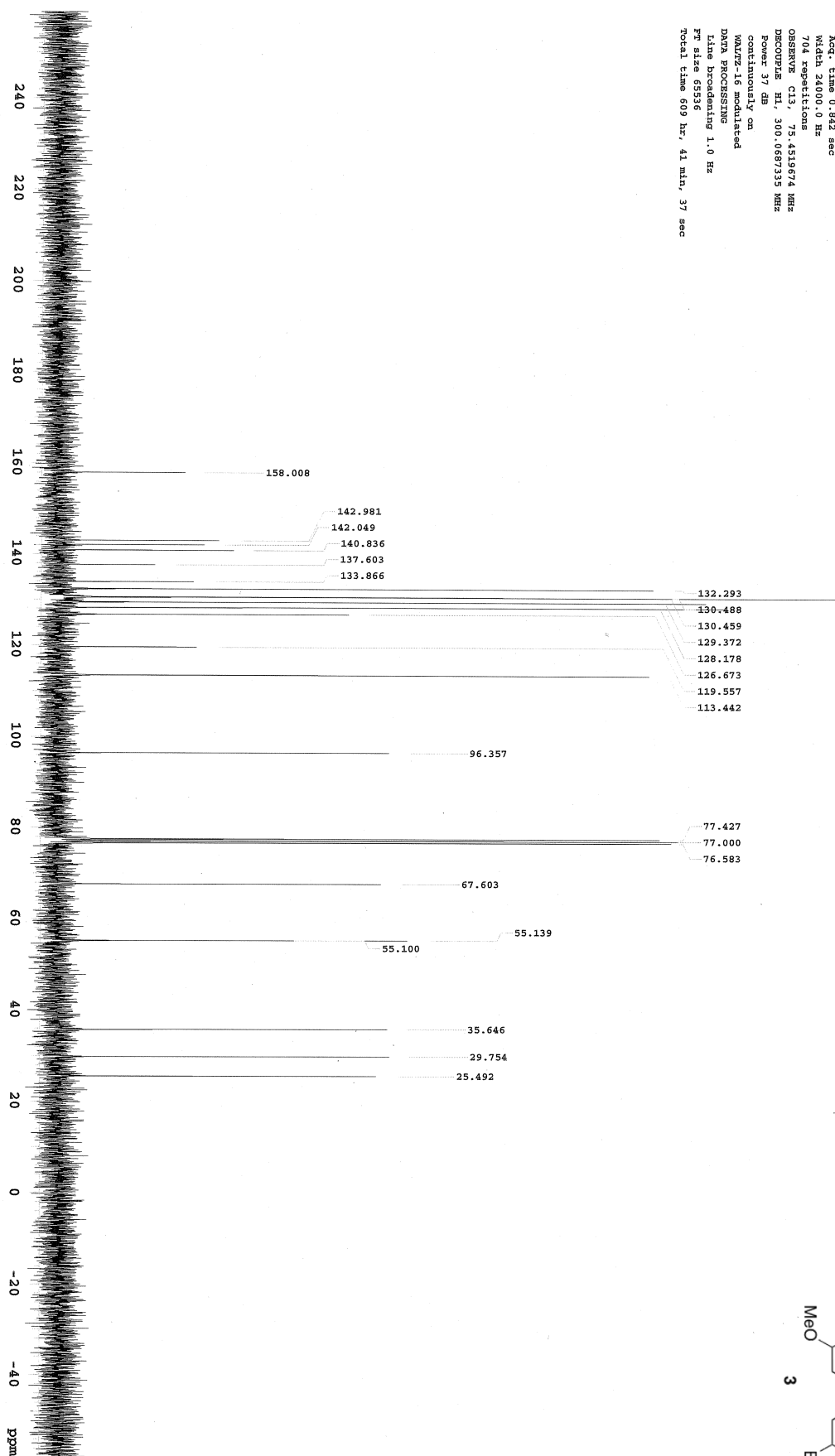
Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE H1, 300.0672369 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec



13C OBSERVE

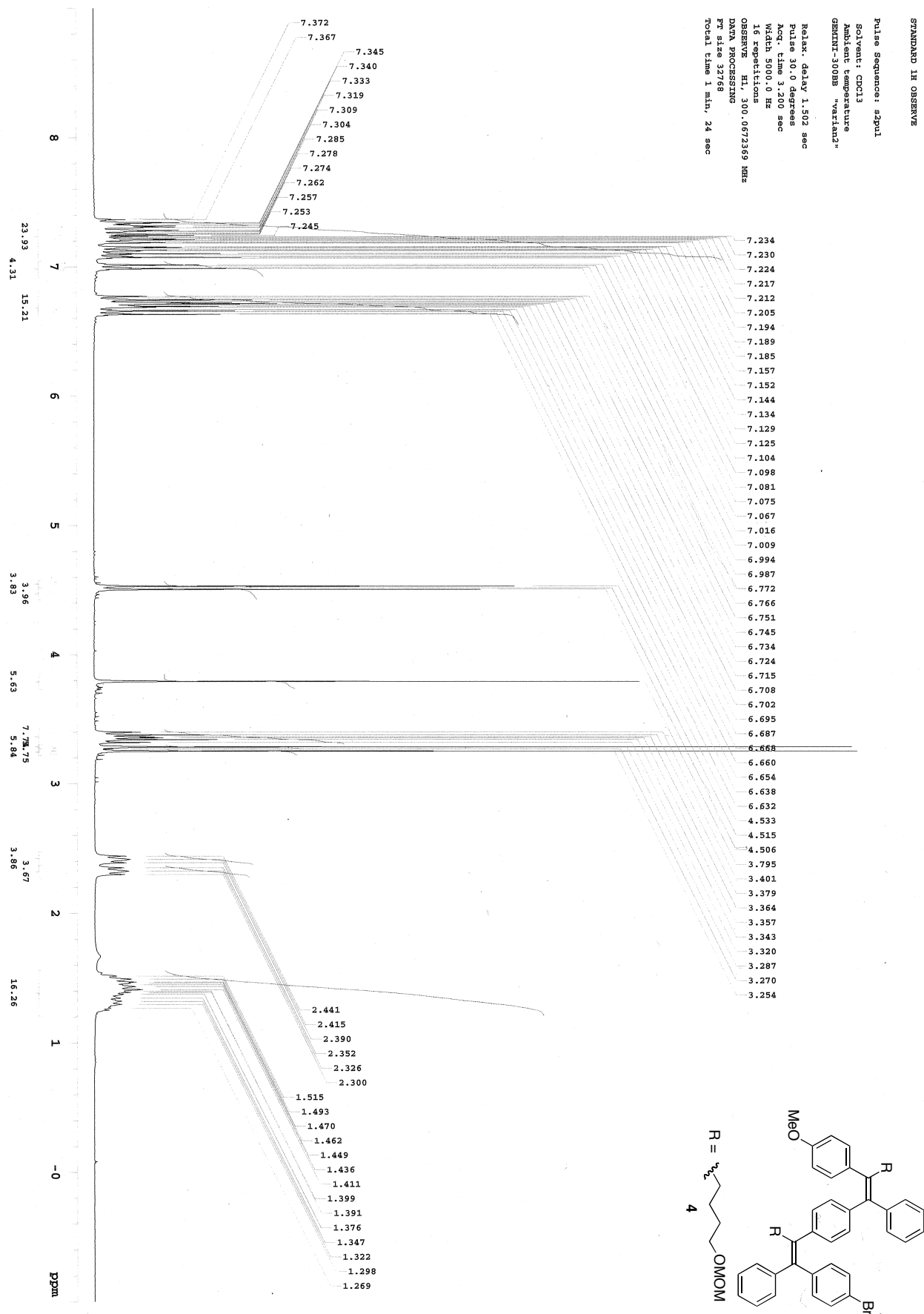
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
GEMINI-300MHz "varian2"

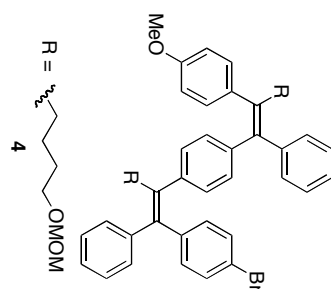
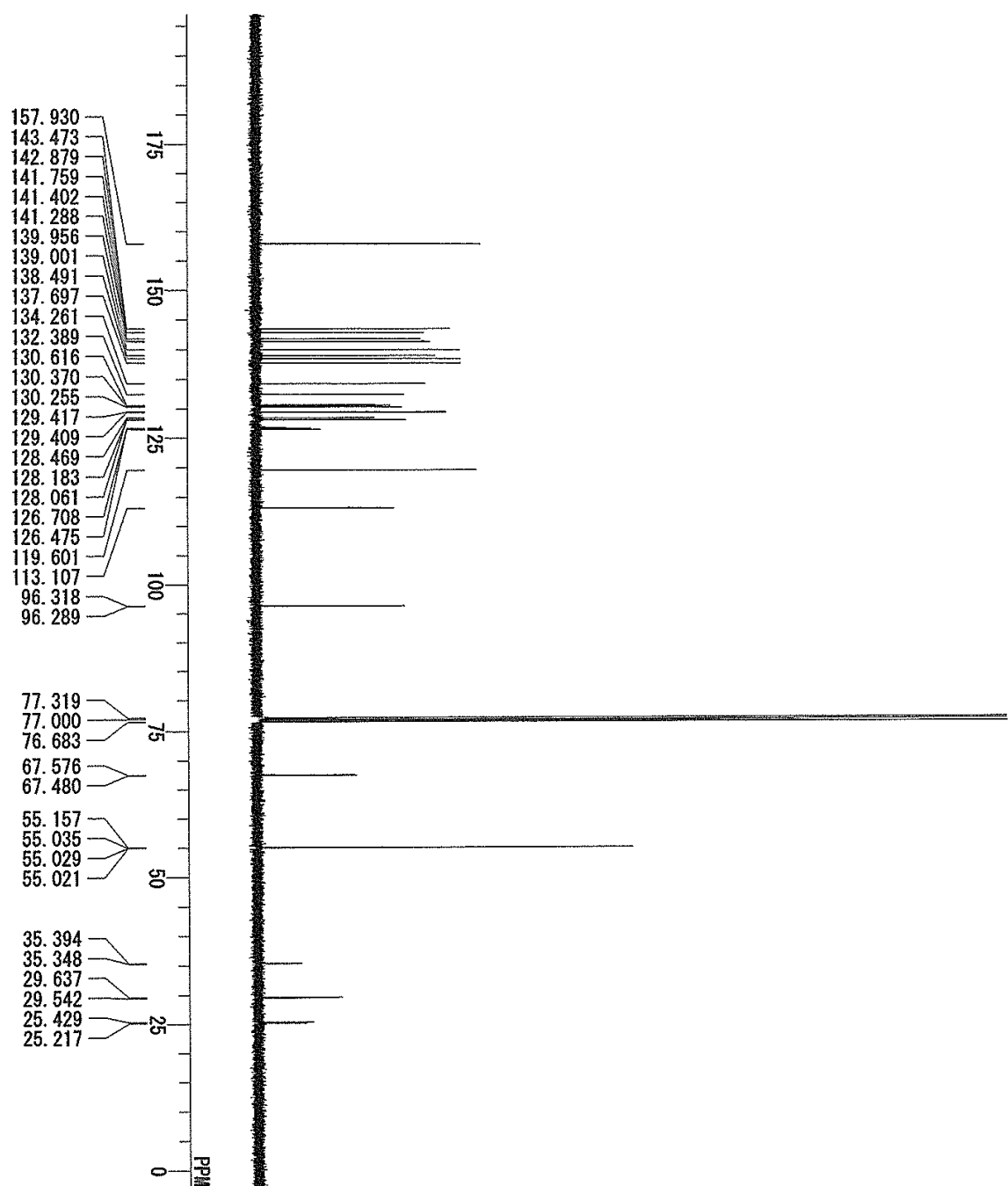
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 24000.0 Hz
704 repetitions
OBSERVE C13, 75.4519674 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 609 hr, 41 min, 37 sec



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300MHz "varian2"
 Relax. delay 1.502 sec
 Pulse 30.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE H1 300.067369 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec





STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CHCl3

Probe: 300MR "Varian2"

Relax. delay 1.502 sec

Pulse 30.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

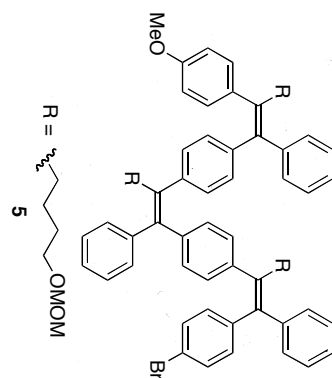
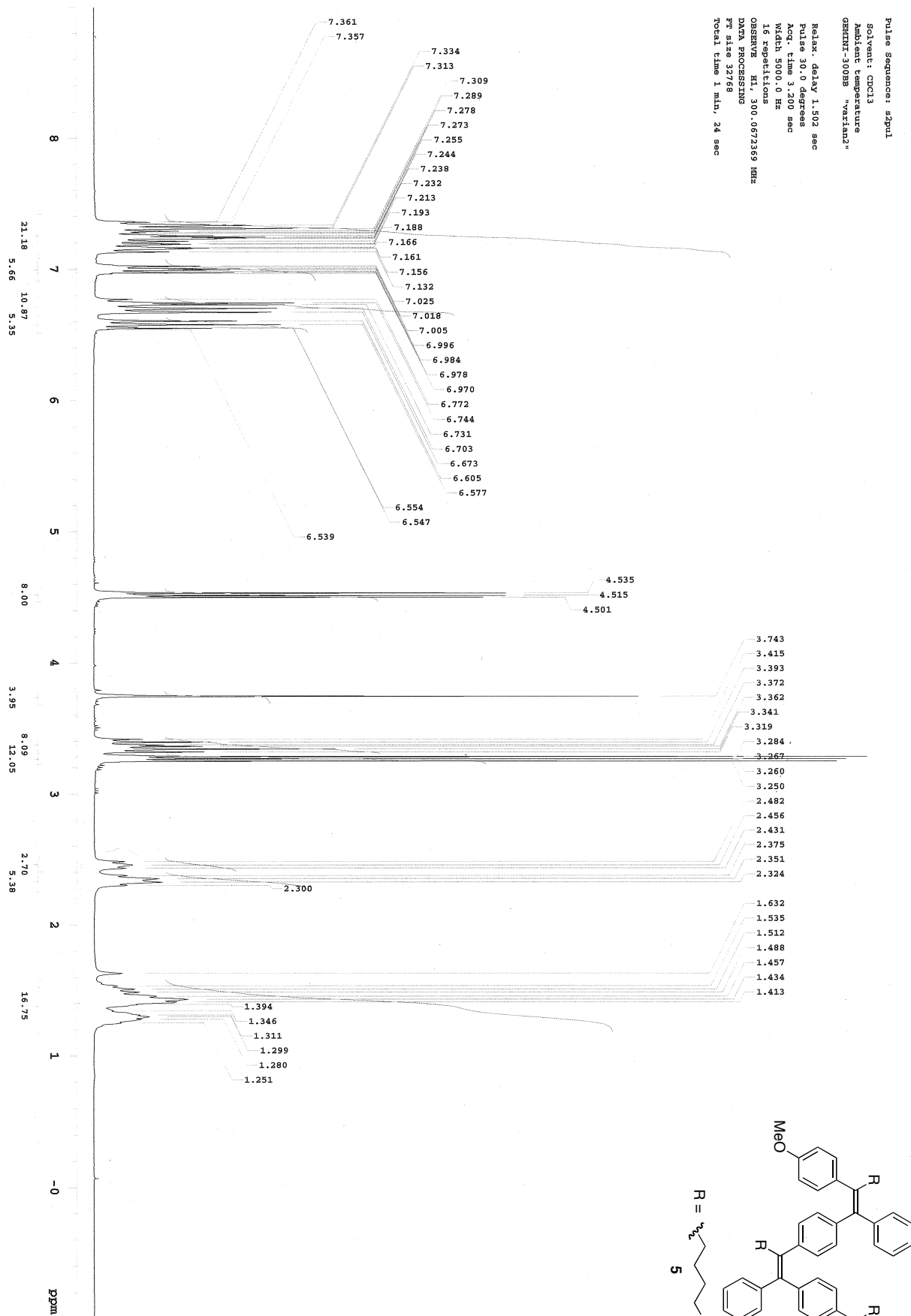
16 repetitions

OBSERVE H1, 300.067369 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 24 sec



13C OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3
Ambient Temperature
GEMINI-300SB "Varian2"

Relax. delay 1.156 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

1056 repetitions

OBSERVE C13 75.4519710 MHz

DECOUPLE H1 300.067335 MHz

Power 37 dB

continuously on

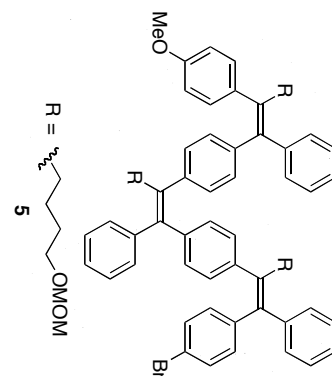
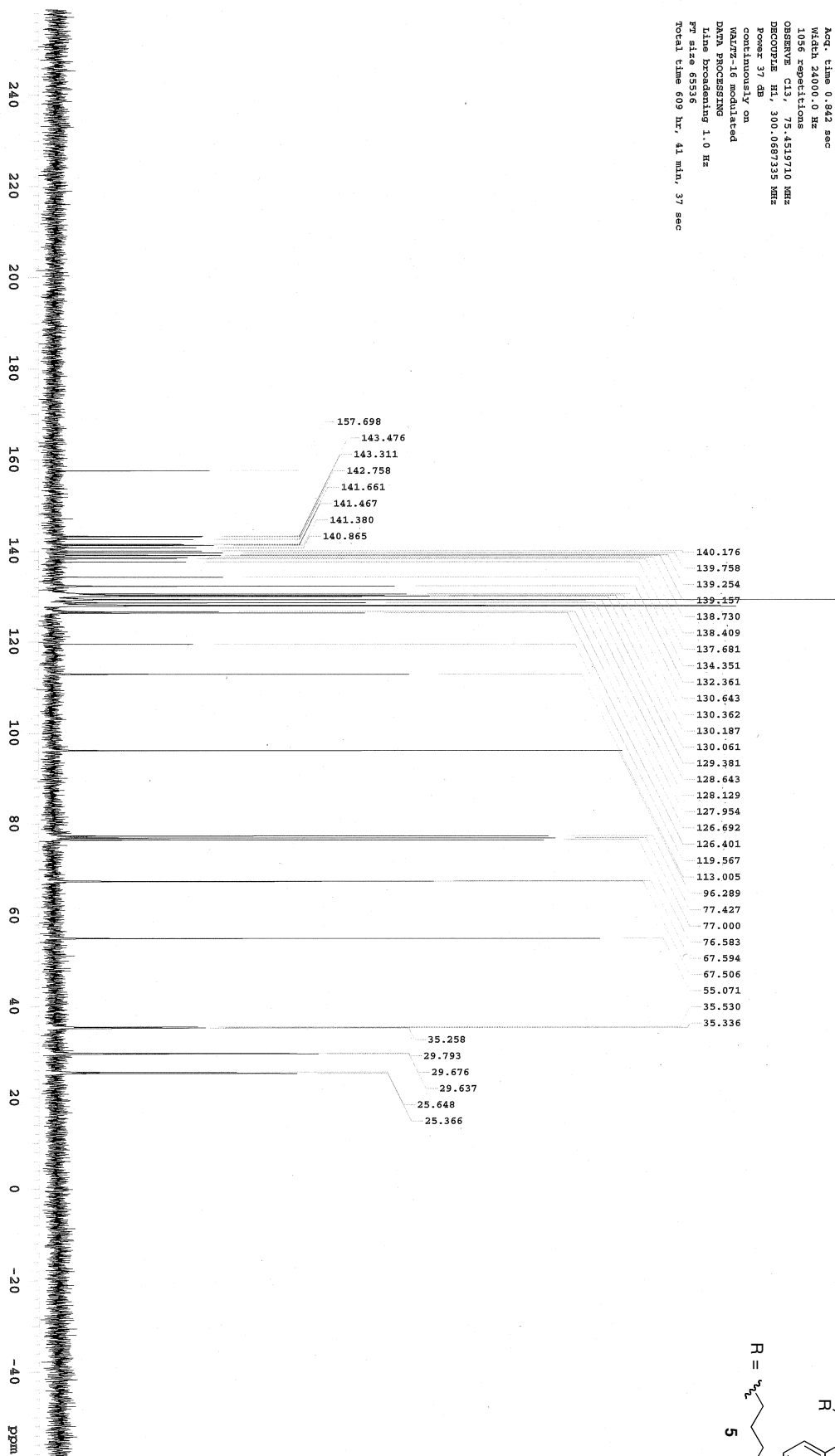
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

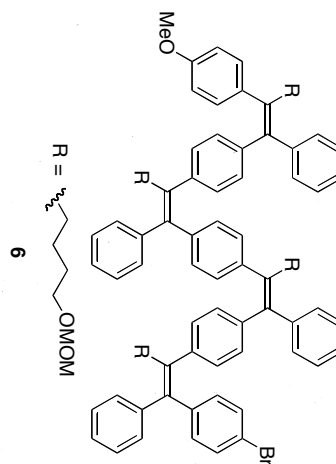
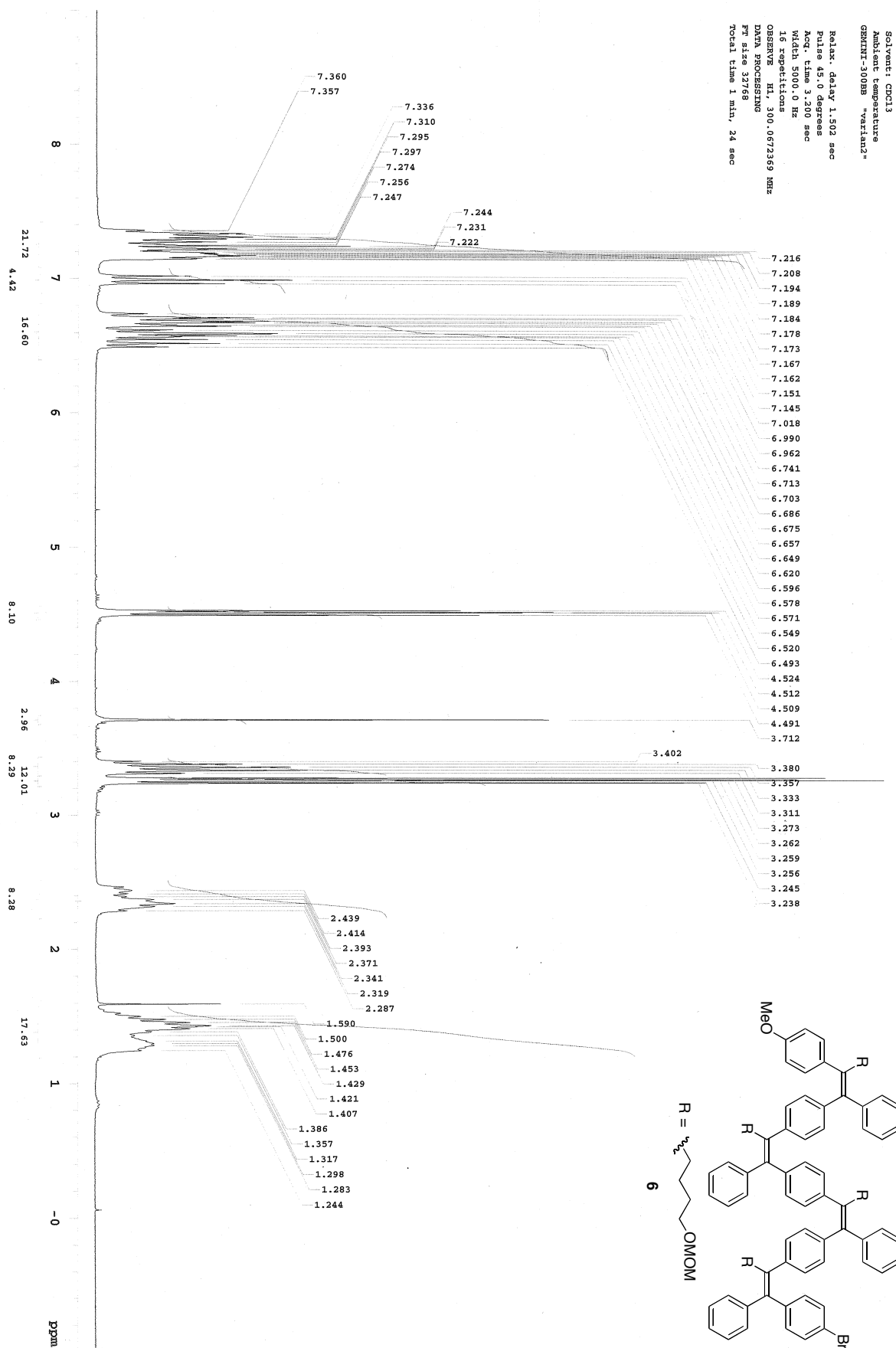
Total time 609 hr, 41 min, 37 sec



Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
GEMINI-300BB "varian2"

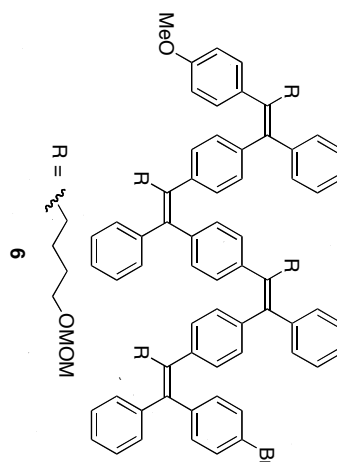
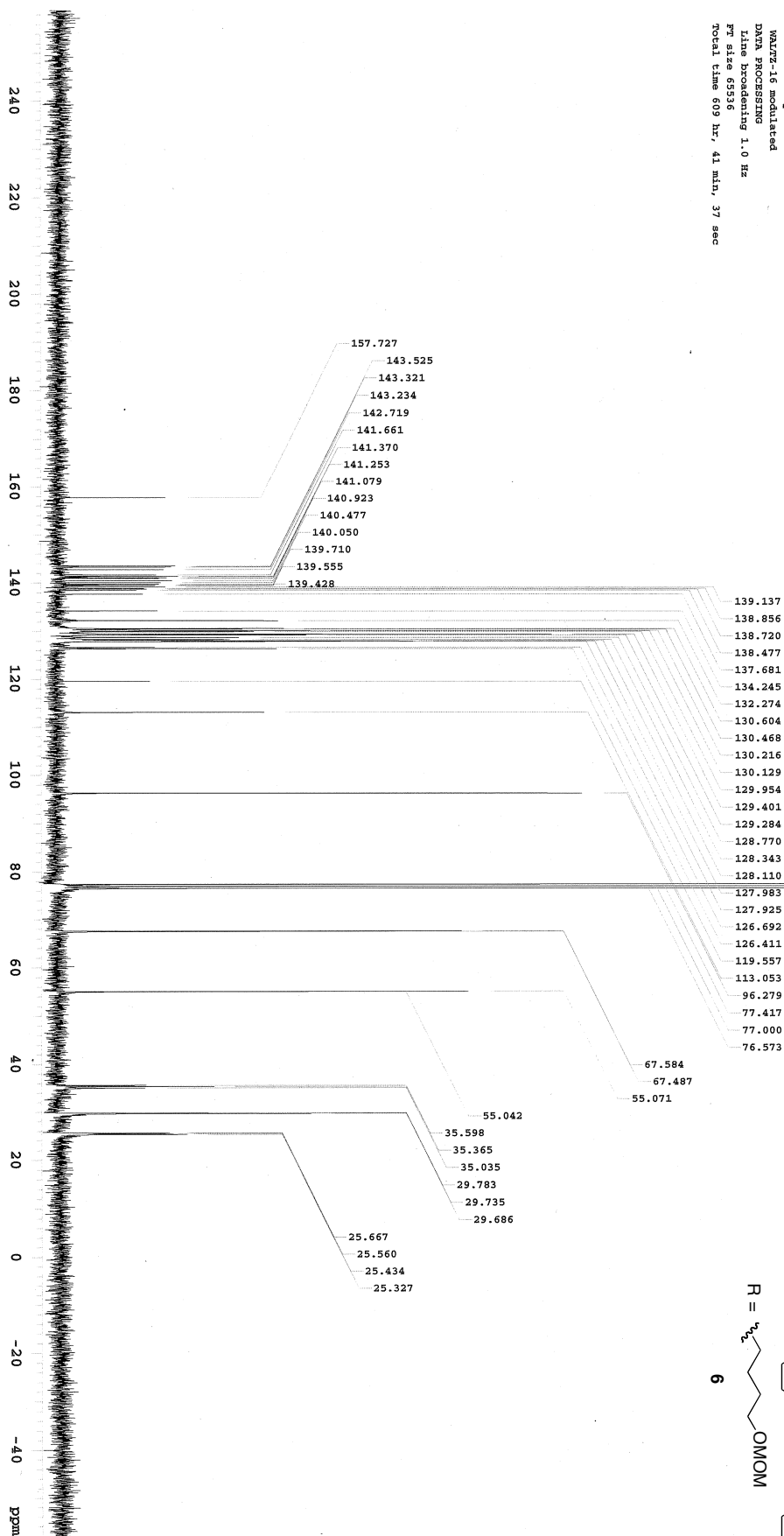
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672369 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec



¹³C OBSERVE

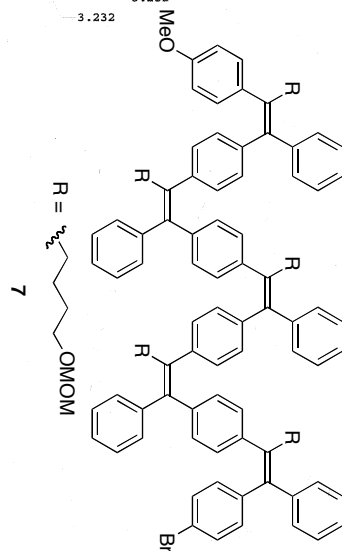
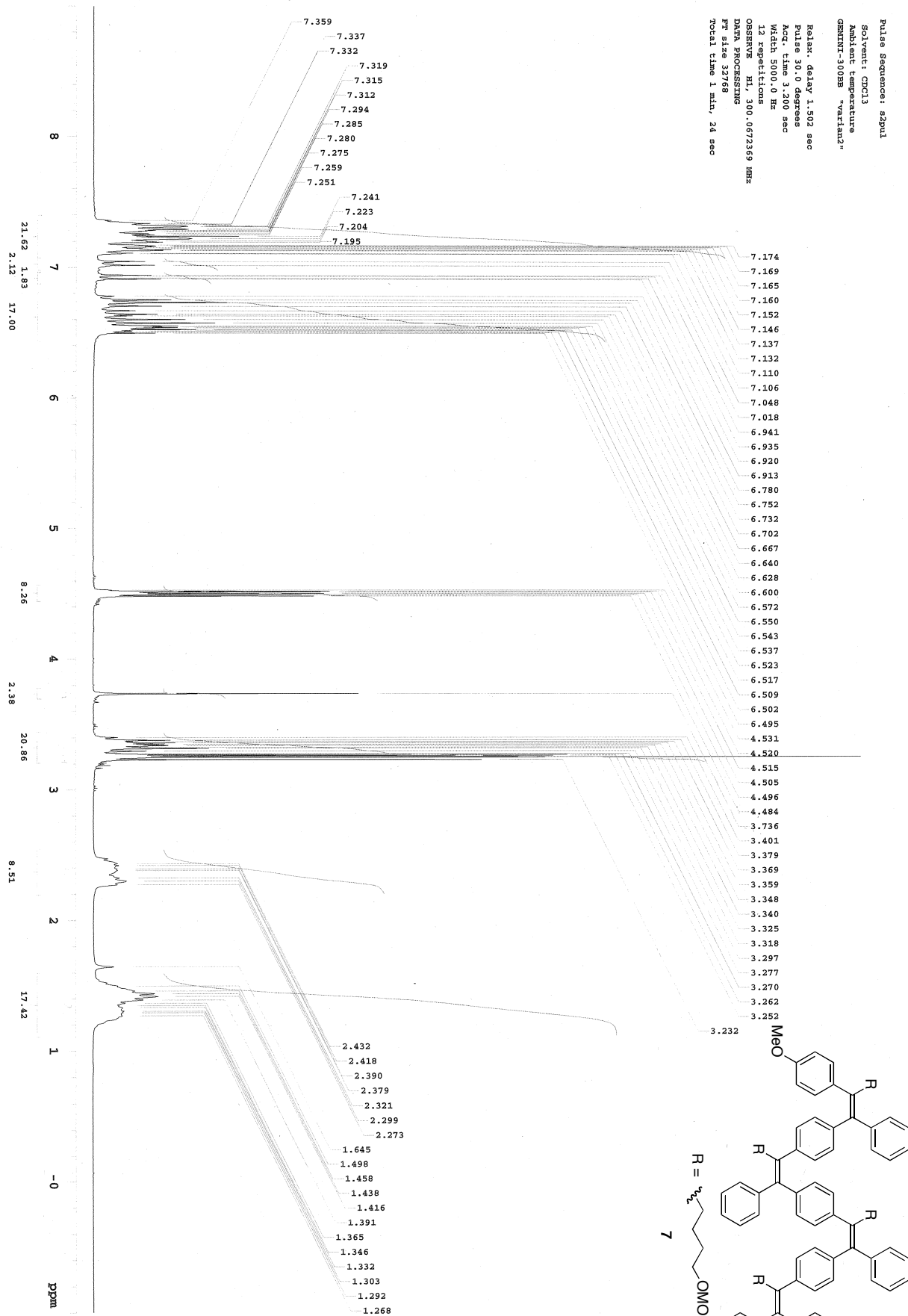
Pulse Sequence: zgpg30
 Solvent: CDCl₃
 Ambient temperature
 GPC-3000B "varian2"

Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 2336 repetitions
 OBSERVE C13, 75.451703 MHz
 DECOUPLE H1, 300.067335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 609 hr, 41 min, 37 sec



STANDARD 1H OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient Temperature
 GEMINI-300MHz "Varian2"
 Relax. delay 1.502 sec
 Pulse 30.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 12 repetitions
 OBSERVE F1, 300.067369 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec



13C OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient Temperature

OSIRIS-300MR "varian2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

816 repetitions

OBSERVE C13 75.4519710 MHz

DECOUPLE H1 100.6267335 MHz

Power 37 dB

continuously on

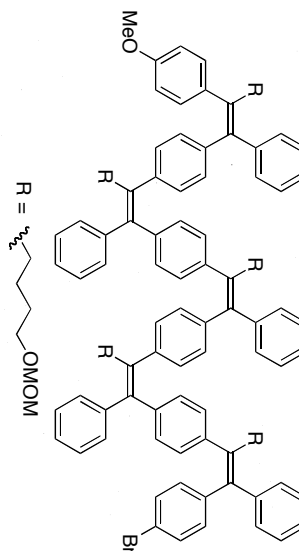
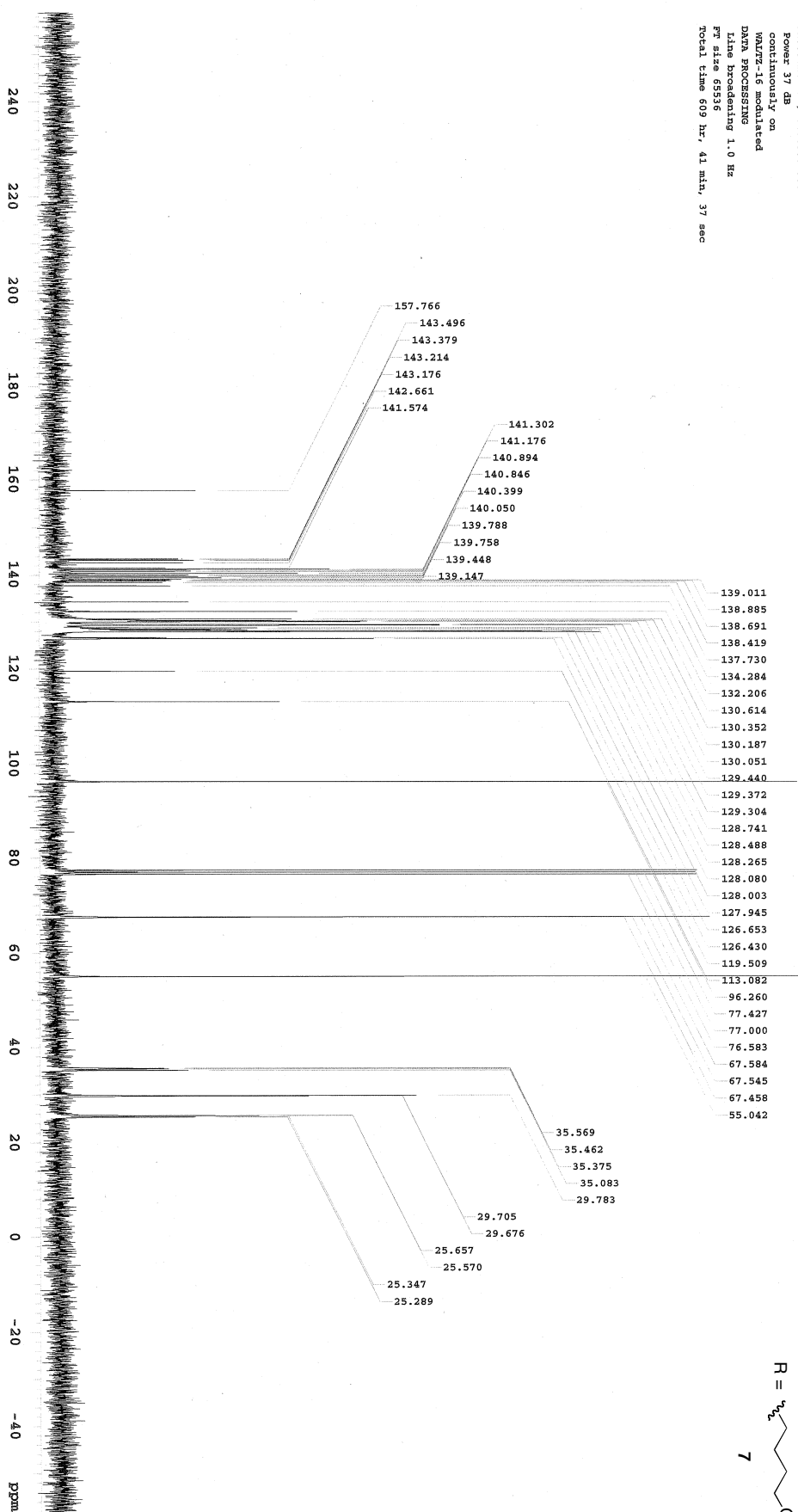
WALTZ-16 modulated

DATA PROCESSING

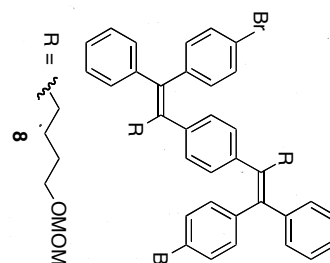
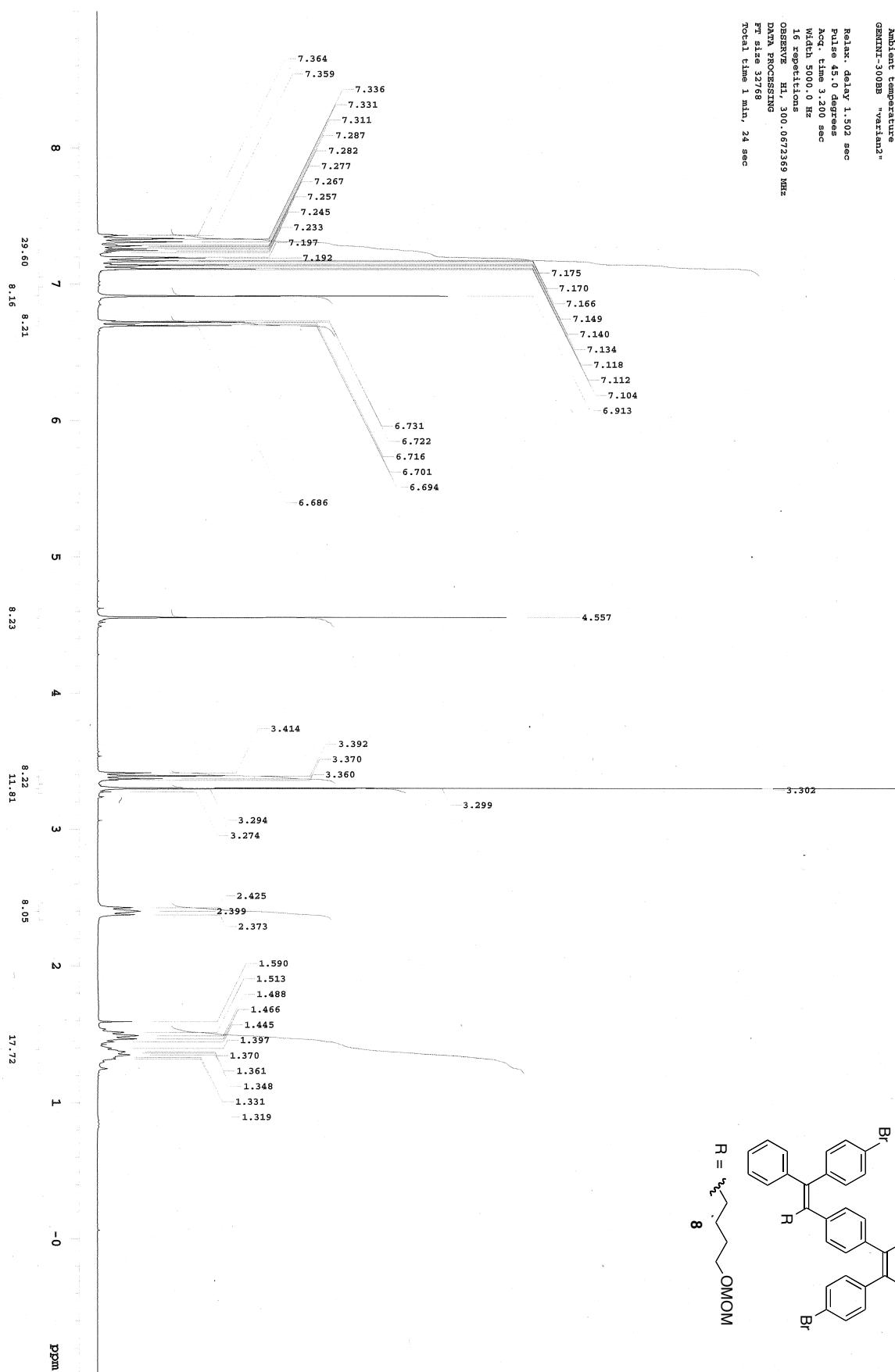
Line broadening 1.0 Hz

FT size 65536

Total time 609 hr, 41 min, 37 sec



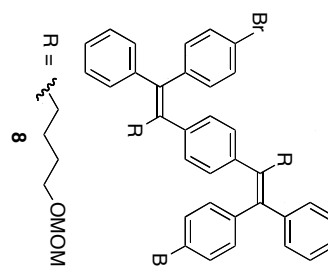
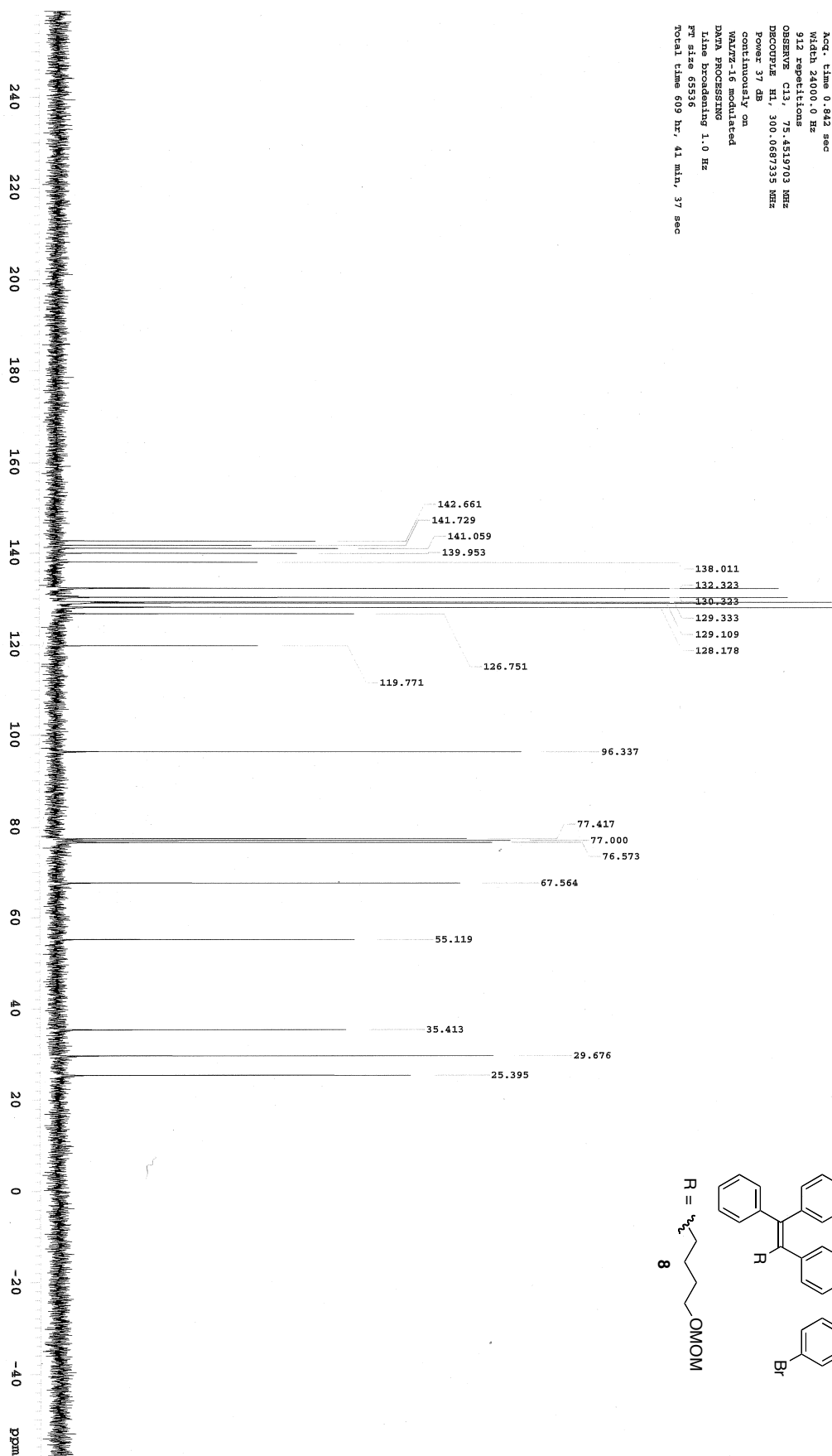
Total time 1 min, 24 sec

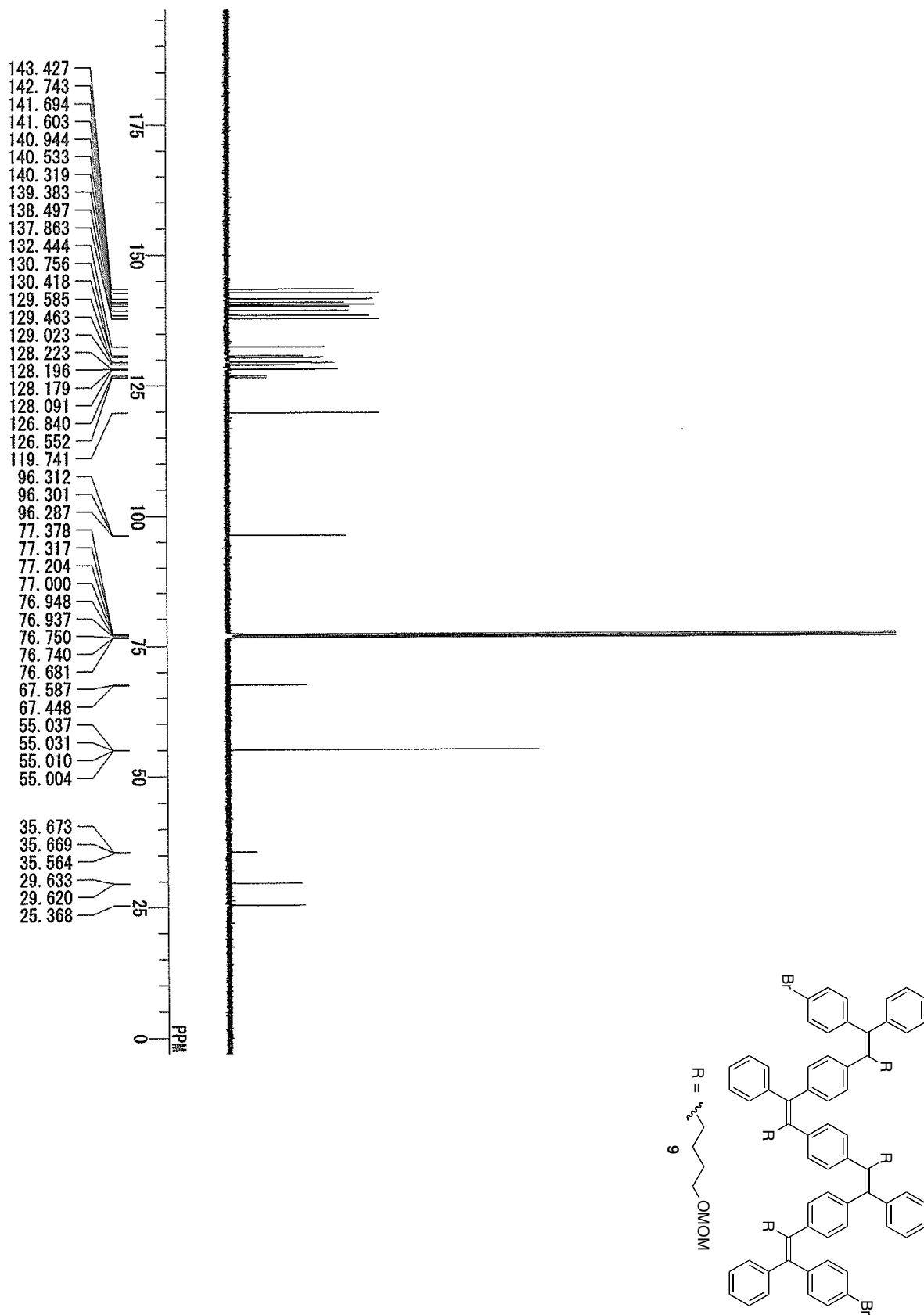


13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300MB "variant2"

Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 912 repetitions
 OBSERVE C13, 75.451903 MHz
 DECOUPLE H1, 300.068735 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 609 hr, 41 min, 37 sec





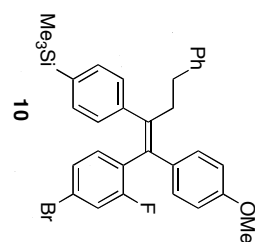
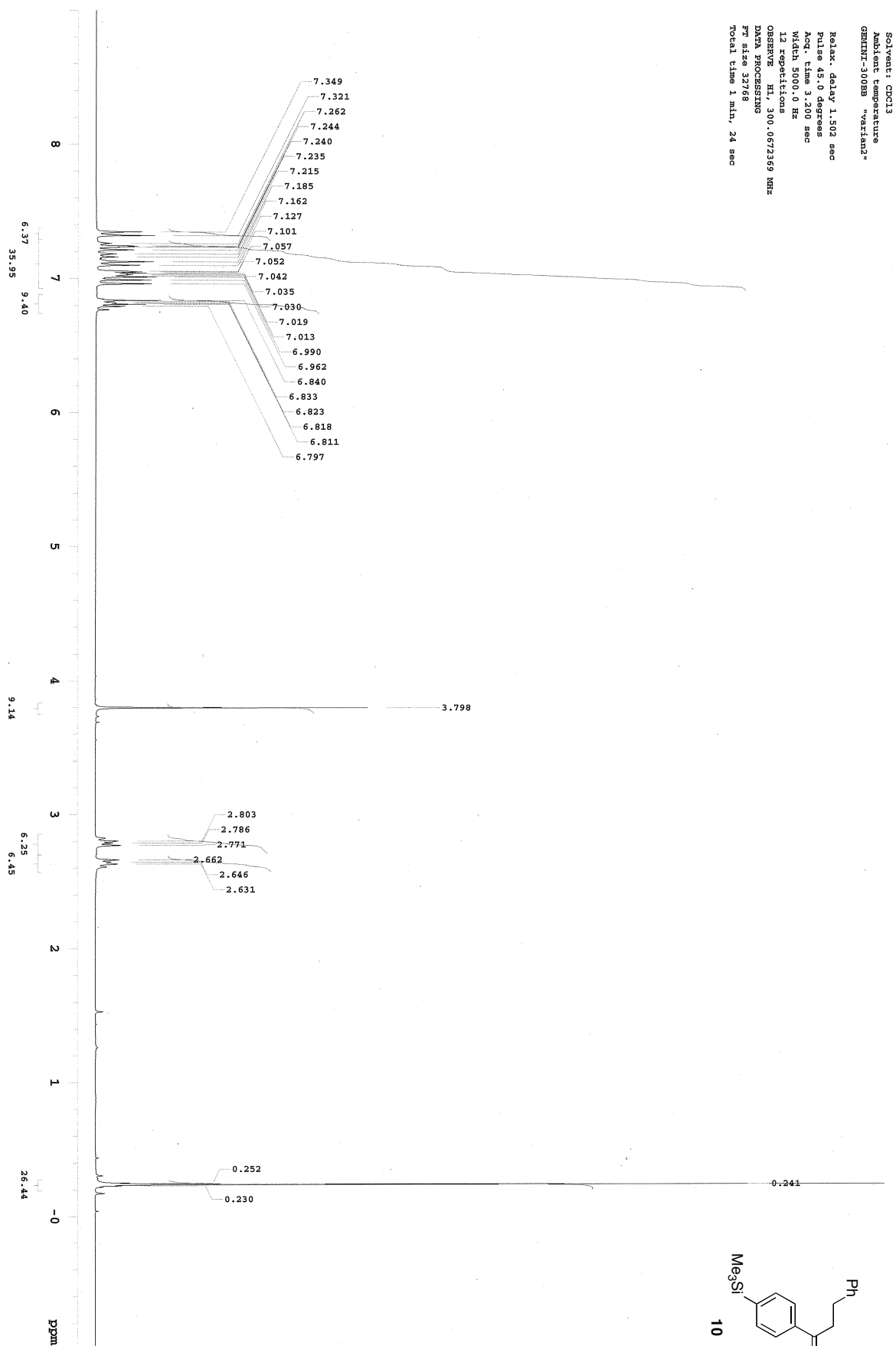
Pulse Sequence: s2pu1

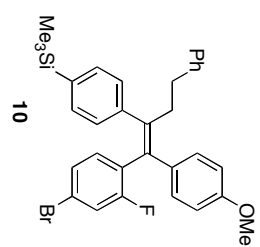
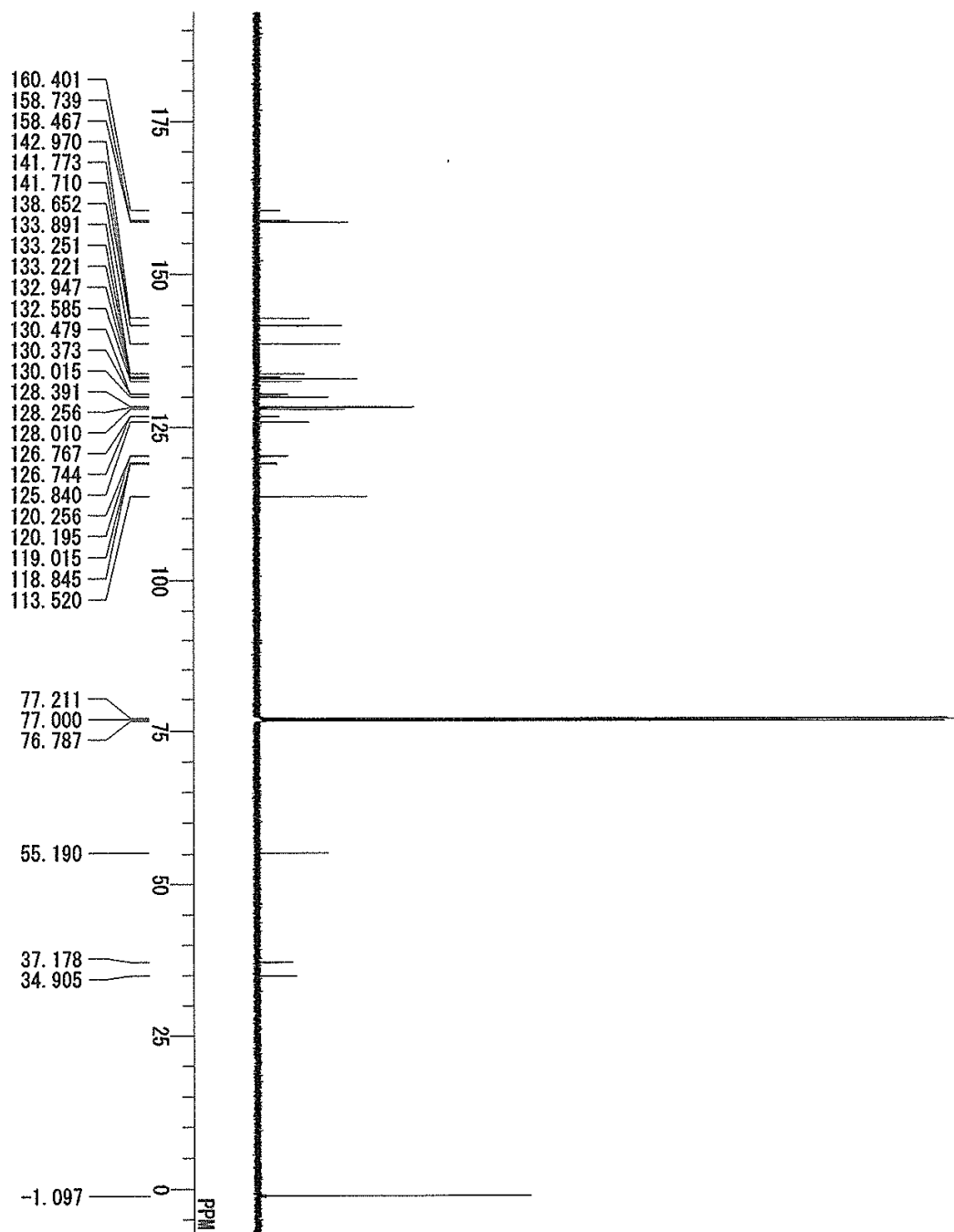
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

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Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
12 repetitions
OBSERVE H1, 300.0672369 MHz
DATA PROCESSING
F1 size 32768
Total time 1 min, 24 sec

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STANDARD 1H OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient temperature

GRUNT-300MHz "varian2"

Relax. delay 1.502 sec

Acq. time 3.200 sec

Width 5000.0 Hz

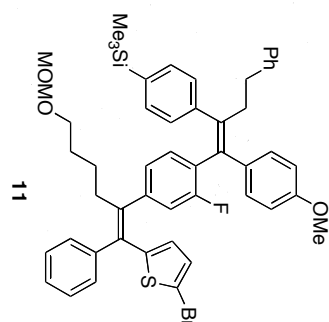
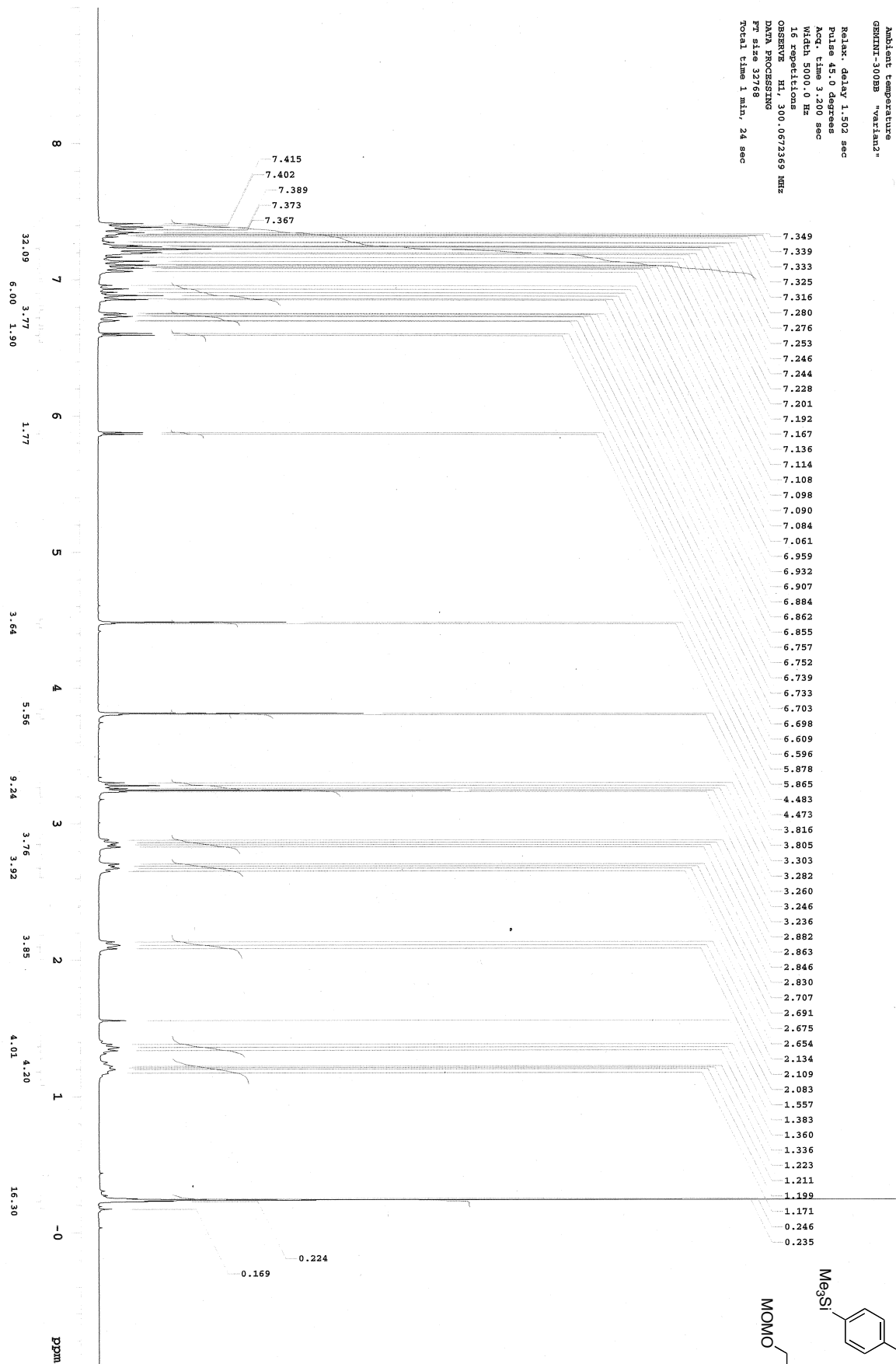
16 repetitions

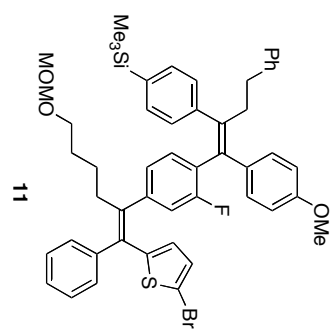
OBSERVE H1 300.0672369 MHz

DATA PROCESSING

PR size 32768

Total time 1 min, 24 sec





STANDARD 1H OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
GEMINI-300MHz "varian2"

Relax. delay 1.502 sec
Pulse 30.0 degrees
Acq. time 3.200 sec
Nuclei 5000.0 Hz
16 repetitions
OBSERVE: H1, 300.0672369 MHz
DATA PROCESSING
F1 size 32768
Total time 1 min, 24 sec

