Vinylation of Aryl Bromides Using an Inexpensive Vinylpolysiloxane

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SUPPORTING INFORMATION

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General Experimental

All reactions were performed in oven-dried (140 °C) or flame dried glassware under an atmosphere of dry nitrogen or argon, unless noted. Reaction solvents tetrahydrofuran (Fisher, HPLC grade), diethyl ether (Fisher, BHT stabilized ACS grade) and methylene chloride (Fisher, unstabilized HPLC grade) were dried by percolation through two columns packed with neutral alumina under a positive pressure of argon. Reaction solvents hexane (Fisher, OPTIMA grade) and toluene (Fisher, ACS grade) were dried by percolation through a column packed with neutral alumina and a column packed with Q5 reactant, a supported copper catalyst for scavenging oxygen, under a positive pressure of argon. Reaction solvent acetonitrile (Fisher, HPLC grade) was used without further purification. DME and dioxane were distilled from Na and benzophenone. Solvents for filtration and chromatography were certified ACS grade. Commercial reagents were purified by distillation or recrystallization prior to use. “Brine” refers to a saturated solution of sodium chloride. All reaction temperatures correspond to internal temperatures measured with Teflon coated thermocouples.

¹H NMR spectra and ¹³C NMR spectra were recorded on either a Varian Unity 400 (400 MHz, ¹H; 100 MHz, ¹³C) or a Varian Unity 500 (500 MHz, ¹H; 125 MHz, ¹³C) spectrometer.

Spectra were referenced to residual chloroform (7.26 ppm, ^1H ; 77.0 ppm, ^{13}C). Chemical shifts are reported in ppm, multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). The prefix nfo (non-first order) coupling when used, indicates coupling which is not first order. Coupling constants, J , are reported in Hertz. Analytical thin-layer chromatography was performed on Merck silica gel plates with QF-254 indicator. Visualization was accomplished with UV (254), potassium permanganate (KMnO_4) or iodine (I_2). Column chromatography was performed using 230-400 mesh silica gel purchased from EM Science.

Electron impact mass spectroscopy (EI) was performed at 70 eV. Data are reported in the form of m/z (intensity relative to base peak = 100). Infrared spectra (IR) peaks are reported in cm^{-1} with the indicated relative intensities: s (strong, 67-100 %); m (medium, 34-66%); w (weak, 0-33%). Analytical capillary gas chromatography (GC) was performed using a gas chromatograph fitted with a flame ionization detector (H_2 carrier gas, 1 mL/min). GC Method 1: Injections were made onto a Hewlett-Packard HP-5 50-m cross-linked 5%-phenyl methyl silicone gum phase column. The injector and detector temperature were 250 $^\circ\text{C}$. The column oven temperature was 225 $^\circ\text{C}$, isothermal. Total run time was 8 min. Retention times (t_R) and integrated ratios were obtained from reporting integrators. Melting points (mp) were determined on a Thomas-Hoover capillary melting point apparatus in sealed tubes and are corrected. Bulb-to-bulb distillations were performed on a Kugelrohr; boiling points (bp) corresponding to uncorrected air-bath temperatures (ABT).

Palladium acetate (Strem), palladium bromide (Strem), and 2-(di-*t*-butylphosphino)biphenyl (Strem), and 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (Gelest) were used as received. A 1.0 M solution of tetra-*n*-butylammonium fluoride (TBAF) was made from the trihydrate salt purchased from Fluka or Acros, dissolved in dry THF, and stored under argon.

Response Factors

Response factors (R_f) for quantitative GC analysis for GC Method 1 were obtained by the equation below:

Eq1: Response factor for A = (area A * mmol biphenyl)/ (mmol A * area biphenyl)

mmol biphenyl	Area biphenyl	mmol 4-bromoacetophenone	Area 4-bromoacetophenone	Response Factor
0.405	141445	0.377	78319	1.68
0.405	93881	0.377	50428	1.73
0.405	217973	0.377	116391	1.74
0.408	1167410	0.403	651991	1.77
0.408	447166	0.403	252412	1.75
0.408	83790	0.403	45036	1.84
0.406	91379	0.406	54336	1.68
0.406	51149	0.406	29321	1.74
0.406	34374	0.406	19516	1.76
Avg:				1.74

mmol biphenyl	Area biphenyl	mmol 3a	Area 3a	Response Factor
0.392	89274	0.403	51625	1.77
0.392	140561	0.403	77311	1.87
0.392	76845	0.403	42984	1.83
0.337	75137	0.406	49002	1.85
0.337	82876	0.406	53847	1.85
0.337	84735	0.406	57321	1.78
Avg:				1.83

Literature Preparations

Allylpalladium chloride dimer¹, palladium bis(dibenzylideneacetone)², and 4-bromoacetamide³ were prepared by the literature procedures.

General Procedure I: Cross-coupling of 4-Bromoacetophenone with 1,3,5,7-Tetramethyl 1,3,5,7-tetravinylcyclotetrasiloxane:

To an oven-dried, 5-mL, 2-necked, round-bottom flask equipped with a magnetic stir bar, septum and nitrogen inlet was added biphenyl and the ligand. The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (51.7 mg, 0.15 mmol, 0.3 equiv) in THF (0.2 mL), followed by TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 4-Bromoacetophenone (99.5 mg, 0.5 mmol) and the palladium source were then added, and the reaction was either allowed to stir at room temperature or was heated to 50 °C. The progress of the reaction was monitored by GC as follows: an aliquot of the reaction mixture was filtered through a small amount of silica gel with EtOAc and was analyzed by GC method 1.

Table 1, entry 1

Following General Procedure I, palladium dibenzylideneacetone (9.6 mg, 0.016 mmol, 0.05 equiv), 4-bromoacetophenone (49.8 mg, 0.25 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (35.4 mg, 0.102 mmol, 0.4 equiv), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol, 2 equiv) were stirred at room temperature for 18 h. Aliquots of the reaction mixture were analyzed by GC Method 1 at 1 h, 3 h, 6 h, 18 h. **3a**, t_R 5.39 min (was not detected).

Table 1, entry 2

Following General Procedure I, allyl palladium chloride dimer (4.5 mg, 0.012 mmol, 0.075 equiv), 4-bromoacetophenone (49.8 mg, 0.249 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (25.8 mg, 0.75 mmol, 0.3 equiv), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol, 2 equiv) were stirred at room temperature for 1 h. An aliquot of the reaction mixture was analyzed by GC Method 1. **3a**, t_R 5.39 min (was not detected).

General Procedure II: Vinylation of Aryl Bromides Using Solvents other than Tetrahydrofuran**Table 1, entry 3:**

To an oven-dried, 2-necked, 5-mL condenser flask equipped with a magnetic stir bar, condenser, and septum was added TBAF (161 mg, 0.51 mmol, 2.0 equiv) in the dry box. Upon removal from the dry box, dioxane (0.5 mL) was added *via* syringe, followed by a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (31 mg, 0.09 mmol, 0.32 equiv) in dioxane (0.1 mL), and this mixture was allowed to stir for 15 min at room temperature. 4-bromoacetophenone (49.2 mg, 0.25 mmol) and APC (4.6 mg, 0.012 mmol, 0.05 equiv) were then added, and the reaction was stirred at room temperature for 18 h. Aliquots of the reaction mixture was filtered through a small amount to silica gel with EtOAc and was analyzed by GC method 1 at 1 h, 3 h, and 18 h. **3a**, t_R 5.39 min (was not detected).

Table 1, entry 4:

Following General Procedure II, TBAF (157 mg, 0.5 mmol, 1.9 equiv), DMF (0.5 mL) 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (27 mg, 0.078 mmol, 0.3 equiv), 4-bromoacetophenone (52.1 mg, 0.26 mmol), and APC (4.5 mg, 0.012 mmol, 0.047 equiv) were stirred at room temperature for 18 h. An aliquot of the reaction mixture was analyzed by GC Method 1 at 1 h, 3 h, and 18 h. **3a**, t_R 5.39 min (was not detected).

Table 1, entry 5:

Following General Procedure I, APC (4.5 mg, 0.012 mmol, 0.05 equiv), bis(diphenylphosphino)butane (10.0 mg, 0.023 mmol, 0.1 equiv), 4-bromoacetophenone (51.1 mg, 0.26 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (26 mg, 0.075 mmol, 0.29 equiv), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol, 1.9 equiv) were stirred at room temperature for 18 h. An aliquot of the reaction mixture was analyzed by GC Method 1 at 1 h, 3 h, and 18 h: **3a** t_R 5.39 min (was not detected).

Table 1, entry 6:

Following General Procedure I, APC (4.4 mg, 0.012 mmol, 0.05 equiv), triphenylarsine (7.65 mg, 0.025 mmol, 0.1 equiv), 4-bromoacetophenone (51.5 mg, 0.26 mmol), 1,3,5,7-

tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (26.2 mg, 0.076 mmol, 0.29 equiv), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol, 1.9 equiv) were stirred at room temperature for 18 h. An aliquot of the reaction mixture was analyzed by GC Method 1 at 1 h, 3 h, and 18 h. **3a** t_R 5.39 min (was not detected).

Table 1, entry 7:

Following General Procedure I, APC (4.5 mg, 0.012 mmol, 0.05 equiv), 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.1 equiv), 4-bromoacetophenone (51.5 mg, 0.26 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (27 mg, 0.078 mmol, 0.3 equiv), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol, 2 equiv) were stirred at room temperature for 18 h. An aliquot of the reaction mixture was analyzed by GC Method 1. 18 h: **3a** t_R 5.39 min (54%).

Table 1, entry 8:

Following General Procedure I, palladium bromide (13 mg, 0.048 mmol, 0.1 equiv), 2-(di-*t*-butylphosphino)biphenyl (17.5 mg, 0.058 mmol, 0.11 equiv), 4-bromoacetophenone (101.1 mg, 0.5 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (54.6 mg, 0.16 mmol, 0.3 equiv), and TBAF (1 mL, 1 M in THF, 1 mmol, 2 equiv) were stirred at room temperature for 18 h. An aliquot of the reaction mixture was analyzed by GC Method 1. 3 h: **3a** t_R 5.39 min (41%), 18 h: **3a** t_R 5.39 min (100%).

Table 1, entry 9:

Following General Procedure I, palladium bromide (13.3 mg, 0.05 mmol, 0.1 equiv), 2-(di-cyclohexylphosphino)biphenyl (17.5 mg, 0.05 mmol, 0.1 equiv), 4-bromoacetophenone (101.1 mg, 0.5 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (55 mg, 0.16 mmol, 0.3 equiv), and TBAF (1 mL, 1 M in THF, 1 mmol, 2 equiv) were stirred at room temperature for 3 h. An aliquot of the reaction mixture was analyzed by GC Method 1. 1 h: **3a** t_R 5.39 min (was not detected), 3 h: **3a** t_R 5.39 min (was not detected).

Table 1, entry 10:

Following General Procedure I, palladium bromide (13.3 mg, 0.05 mmol, 0.1 equiv), 2-(di-*t*-butylphosphino)biphenyl (15.2 mg, 0.05 mmol, 0.1 equiv), 4-bromoacetophenone (101.1

mg, 0.5 mmol), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (54.6 mg, 0.16 mmol, 0.3 equiv), and TBAF (1 mL, 1 M in THF, 1 mmol, 2 equiv) were stirred at 50 °C for 3 h. An aliquot of the reaction mixture was analyzed by GC Method 1. 3 h: **3a** t_R 5.39 min (100%).

General Procedure III: Vinylation of Aryl Bromides with 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane at 50 °C

Table 2, entry 1: Preparation of 1-(4-ethynylphenyl)ethanone (3a**)**

To an oven-dried, 5-mL condenser flask equipped with a magnetic stir bar and nitrogen inlet was added 2-(di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.05 equiv). The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (101.2 mg, 0.3 mmol, 0.3 equiv) in THF (0.1 mL), followed by TBAF (2 mL, 1 M in THF, 2 mmol, 2.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 4-Bromoacetophenone (199.5 mg, 1.0 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were then added, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 3 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then was filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (25 mL), and the combined filtrates were concentrated *in vacuo* to afford 160 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (40:1), SiO₂, 125 mm x 40 mm) gave 125 mg (85%) of **3a** as a white solid.

Table 2, entry 2: Preparation of 2-Vinylnaphthalene (3b**)**

Following General Procedure III, 2-(di-*t*-butylphosphino)biphenyl (15.0 mg, 0.05 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (109 mg, 0.3 mmol, 0.3 equiv) in THF (0.1 mL), TBAF (2 mL, 1 M in THF, 2 mmol, 2.0 equiv), 2-bromonaphthalene (207 mg, 1.0 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were combined and warmed to 50 °C in an oil bath. GC analysis at 3 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (25 mL), and the combined filtrates were

concentrated *in vacuo* and immediately purified by column chromatography (hexane/ethyl acetate (40:1), SiO₂, 125 mm x 40 mm) gave 127 mg (82 %) of **3b** as a white solid.

Table 2, entry 3: Preparation of 1-Ethenyl-4-methoxybenzene (3c)

Following General Procedure III, 2-(di-*t*-butylphosphino)biphenyl (15.0 mg, 0.05 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (109 mg, 0.3 mmol, 0.3 equiv) in THF (0.1 mL), TBAF (2 mL, 1 M in THF, 2 mmol, 2.0 equiv), 4-bromoanisole (207 mg, 1.0 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 3h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. To the cooled mixture was added diethyl ether (10 mL) and allowed to stir for 10 min at room temperature, after which the mixture was filtered through a short pad of silica, washed with diethyl ether (25 mL), and concentrated *in vacuo* to afford 147 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (40:1), SiO₂, 150 mm x 40 mm) gave 43 mg (32 %) of **3c** as a clear oil.

Table 3, entry 1: (See Table 2, entry 3)

Table 3, entry 2:

Following General Procedure I, 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (51.1 mg, 0.15 mmol, 0.3 equiv) in THF (0.1 mL), TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv), 4-bromoanisole (93.5 mg, 0.5 mmol), hexamethylbenzene (4.9 mg, 0.03 mmol) and palladium bromide (6.5 mg, 0.025 mmol, 0.05 equiv) were combined, and the reaction was warmed to 70 °C in an oil bath. A 3 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **8** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 32.16, 67 %), stilbene (3.83 ppm, 6 H, 11.83, 12%) **3c**/stilbene (5.4:1).

Table 3, entry 3:

Following General Procedure II, TBAF (630 mg, 2 mmol, 2 equiv) dimethoxyethane (2 mL), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (111 mg, 0.32 mmol, 0.32 equiv) in DME (0.1 mL), 2-(di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.05 equiv), hexamethylbenzene (6.5 mg, 0.04 mmol), 4-bromoanisole (187 mg, 1.0 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were combined and the reaction was heated to reflux in an oil bath. A 3 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 47.66, 64 %), stilbene (3.83 ppm, 6 H, 19.18, 13%) **3c**/stilbene (5.0:1).

Table 3, entry 4:

Following General Procedure II, TBAF (630 mg, 2 mmol, 2 equiv), dioxane (2 mL) a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (111 mg, 0.32 mmol, 0.32 equiv) in dioxane (0.1 mL), 2-(Di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.05 equiv), hexamethylbenzene (8.7 mg, 0.053 mmol), 4-bromoanisole (187 mg, 1.0 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were combined, and the reaction was heated to reflux in an oil bath. A 3 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 39.62, 70 %), stilbene (3.83 ppm, 6 H, 13.35, 12 %), **3c**/stilbene (5.9:1).

Table 3, entry 5:

Following General Procedure I, 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (110 mg, 0.32 mmol, 0.6 equiv) in THF (0.1 mL), TBAF (2 mL, 1 M in THF, 2 mmol, 4.0 equiv), 4-bromoanisole (93.5 mg, 0.5 mmol), hexamethylbenzene (8.6 mg, 0.053 mmol) and palladium bromide (6.5 mg, 0.025 mmol, 0.05 equiv) were combined, and the reaction was warmed to 70 °C in an oil bath. A 1 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was

determined by relative integration. **3c** (3.81 ppm, 3 H, 20.71, 74 %), stilbene (3.83 ppm, 6 H, 1.53, 3 %) **3c**/stilbene (27:1).

Table 3, entry 6:

Following General Procedure I, 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (153 mg, 0.44 mmol, 0.88 equiv) in THF (0.1 mL), TBAF (3 mL, 1 M in THF, 3 mmol, 6.0 equiv), 4-bromoanisole (93.5 mg, 0.5 mmol), hexamethylbenzene (8.8 mg, 0.054 mmol) and palladium bromide (6.5 mg, 0.025 mmol, 0.05 equiv) were combined, and the reaction was warmed to 70 °C in an oil bath. A 1 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 25.77, 93 %), stilbene (3.83 ppm, 6 H, 0.06, 0.1 %) **3c**/stilbene (>99:1).

Table 3, entry 7:

Following General Procedure I, 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (154 mg, 0.45 mmol, 0.9 equiv) in THF (0.1 mL), TBAF (1.8 mL, 1 M in THF, 1.8 mmol, 3.6 equiv), 4-bromoanisole (93.5 mg, 0.5 mmol), hexamethylbenzene (4.7 mg, 0.029 mmol) and palladium bromide (6.5 mg, 0.025 mmol, 0.05 equiv) were combined, and the reaction was warmed to 70 °C in an oil bath. A 2 h aliquot was analyzed by ¹H-NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 48.73, 94 %), stilbene (3.83 ppm, 6 H, 2.56, 2 %) **3c**/stilbene (38:1).

Table 3, entry 8:

Following General Procedure II, 2-(di-*t*-butylphosphino)biphenyl (15 mg, 0.05 mmol, 0.05 equiv), 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (309 mg, 0.9 mmol, 0.9 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 4.0 equiv), 4-bromoanisole (174 mg, 1.0 mmol), hexamethylbenzene (13.2 mg, 0.081 mmol) and palladium bromide (13.3 mg, 0.05 mmol, 0.05 equiv) were combined, and the reaction was warmed to 70 °C in an oil bath. A

2 h aliquot was analyzed by ^1H -NMR spectroscopy. The yield was determined by the integration of **3c** versus the internal standard which was normalized to 18. The ratio of styrene/stilbene was determined by relative integration. **3c** (3.81 ppm, 3 H, 48.73, 94 %), stilbene (3.83 ppm, 6 H, was not observed, < 1 %) **3c**/stilbene (>99:1).

Scheme 2, entry 1:

To an oven-dried, 5-mL condenser flask equipped with a magnetic stir bar and nitrogen inlet was added 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), palladium bromide (6.6 mg, 0.025 mmol, 0.05 equiv). The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (154 mg, 0.45 mmol, 0.9 equiv) in THF (0.1 mL), followed by TBAF (2 mL, 1 M in THF, 2 mmol, 4.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 1-Bromonaphthalene (103.5 mg, 0.5 mmol) was then added *via* syringe, and the reaction was warmed to 70 °C in an oil bath. Two aliquots, 2.5 h and 5 h, were analyzed by ^1H -NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. 5 h: **2b** (8.21 ppm, 1 H, was not detected, 0 %), **3b** (8.15 ppm, 1 H, 6.29, 86 %), **7** (8.27 ppm, 2 H, 1.99, 14 %) **3b**/**7** (6.3:1).

General Procedure IV: Vinylation of Aryl Bromides using 2.0 Equivalents of 2-(di-*t*-butylphosphino)biphenyl.

Scheme 2, entry 2:

To an oven-dried, 10-mL, 2-necked, round-bottomed flask equipped with a magnetic stir bar, septum and nitrogen inlet was added 2-(di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.10 equiv), palladium bromide (6.6 mg, 0.025 mmol, 0.05 equiv). The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (51 mg, 0.15 mmol, 0.3 equiv) in THF (0.1 mL), followed by TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 1-Bromonaphthalene (103.4 mg, 0.5 mmol) was then added *via* syringe, and the reaction was stirred at room temperature for 1 h. A 1 h aliquot was analyzed by ^1H -NMR

spectroscopy, showing no product. The reaction was warmed to 50 °C in an oil bath. A 1.5 h (0.5 h after warming) aliquot was analyzed by ¹H-NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. **2b** (8.21 ppm, 1 H, was not detected, 0 %), **3b** (8.15 ppm, 1 H, 1.00, 87 %), **7** (8.27 ppm, 2 H, 0.31, 13 %) **3b/7** (6.4:1).

Table 4, entry 1:

To an oven-dried, 10-mL, 2-necked, round-bottomed flask equipped with a magnetic stir bar, septum and nitrogen inlet was added 2-(di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.10 equiv), palladium bromide (6.6 mg, 0.025 mmol, 0.05 equiv). The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (51 mg, 0.15 mmol, 0.3 equiv) in THF (0.1 mL), followed by TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 1-Bromonaphthalene (103.4 mg, 0.5 mmol) was then added *via* syringe, and the reaction was stirred at room temperature for 6 h. Two aliquots, 3 h and 6 h, were analyzed by ¹H-NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. 6 h: **2b** (8.21 ppm, 1 H, was not detected, 0 %), **3b** (8.15 ppm, 1 H, 1.00, 91 %), **7** (8.27 ppm, 2 H, 0.20, 9 %) **3b/7** (10:1).

Table 4, Entry 2, (See Scheme 2, Entry 2 above)

Table 4, Entry 3

Following General Procedure IV, 2-(di-*t*-butylphosphino)biphenyl (7.5 mg, 0.025 mmol, 0.05 equiv), palladium bis(dibenzylideneacetone) (14.1 mg, 0.025 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (51 mg, 0.15 mmol, 0.3 equiv) in THF (0.1 mL), TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) and 1-bromonaphthalene (103.4 mg, 0.5 mmol) were combined, and the mixture was warmed to 50 °C and stirred for 6 h. Three aliquots, 30 min, 2 h and 6 h, were analyzed by ¹H-NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. 6 h: **2b** (8.21 ppm, 1 H, 0.10, 9 %), **3b** (8.15 ppm, 1 H, 0.99, 87 %), **7** (8.27 ppm, 2 H, 0.10, 4 %) **3b/7** (19:1).

Table 4, Entry 4

Following General Procedure IV, 2-(di-*t*-butylphosphino)biphenyl (15 mg, 0.05 mmol, 0.10 equiv), palladium bromide (6.6 mg, 0.025 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (68.8 mg, 0.2 mmol, 0.4 equiv) in THF (0.1 mL), TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) and 1-bromonaphthalene (103.4 mg, 0.5 mmol) were combined, and the mixture was warmed to 50 °C and stirred for 1 h. A 1 h aliquot was analyzed by ¹H-NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. **2b** (8.21 ppm, 1 H, 1.00, 6 %), **3b** (8.15 ppm, 1 H, 16.13, 92 %), **7** (8.27 ppm, 2 H, 1.00, 3 %) **3b/7** (6.4:1).

Table 4, Entry 5

Following General Procedure IV, 2-(di-*t*-butylphosphino)biphenyl (15.1 mg, 0.05 mmol, 0.10 equiv), palladium bromide (6.6 mg, 0.025 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (86 mg, 0.25 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (1 mL, 1 M in THF, 1 mmol, 2.0 equiv) and 1-bromonaphthalene (103.4 mg, 0.5 mmol) were combined, and the mixture was warmed to 50 °C and stirred for 2.5 h. Two aliquots, 2 h and 2.5 h, were analyzed by ¹H-NMR spectroscopy. The conversion and styrene/stilbene ratio were determined by relative integration. 2.5 h: **2b** (8.21 ppm, 1 H, was not detected, 0 %), **3b** (8.15 ppm, 1 H, 32.01, 97 %), **7** (8.27 ppm, 2 H, 2.00, 3 %) **3b/7** (32:1).

General Procedure V: Preparative Vinylation of Aryl Bromides**Preparation of 1-(4-Ethenylphenyl)ethanone (**3a**)⁴**

To an oven-dried, 10-mL, 2-necked, round-bottomed flask equipped with a magnetic stir bar, septum and nitrogen inlet was added 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv). The flask was then evacuated and refilled with argon, and this evacuation/refill process was repeated two additional times. A solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), followed by TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) were then sequentially added to the flask, and the resulting mixture was allowed to stir for 10 min at room temperature. 4-Bromoacetophenone (398.0 mg, 2.0 mmol) was then added *via* syringe, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 4 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 645 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (50:1), SiO₂, 160 mm x 40 mm) gave 265 mg (91 %) of **3a** as a white solid.

Data for **3a:**

mp: 33-34 °C

¹H NMR: (500 MHz, CDCl₃)

7.92 (d, *J* = 8.6, 2 H, HC(3)), 7.48 (d, *J* = 8.4, 2 H, HC(4)), 6.76 (dd, *J* = 17.6 and 10.9, 1 H, HC(2)), 5.88 (dd, *J* = 17.6 and 0.6, 1 H, H_{trans}C(1)), 5.39 (dd, *J* = 10.9 and 0.6, 1 H, H_{cis}C(1)), 2.60 (s, 3 H, HC(8)).

¹³C NMR: (125 MHz, (CDCl₃))

197.9 (C(7)), 142.3 (C(3)), 136.5 (C(6)), 136.2 (C(2)), 128.9 (C(5)), 126.5 (C(4)), 117.0 (C(1)), 26.8 (C(8)).

IR: 3054 (m), 2987 (w), 2306 (w), 1681 (m), 1605 (w), 1422 (w), 1359 (w), 1265 (s), 1181 (w), 957 (w), 922 (w), 896 (w), 847 (w), 739 (s), 705 (s).

MS: (EI, 70eV)

146 (M^+ , 41), 132 (11), 131 (100), 103 (48), 77 (28).

R_f: 0.07 (hexane/ethyl acetate, 50:1)

Preparation of 1-Ethenynaphthalene (**3b**)⁵

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) and 1-bromonaphthalene (414 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 3 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Pentane (10 mL) was added and the mixture was stirred for 10 min at room temperature, then filtered twice through a short pad of silica (5 g). The silica pad was washed with pentane (50 mL) and the combined filtrates were concentrated *in vacuo* to afford 580 mg of crude material. Purification by column chromatography (RP C18, methanol/water (9:1), 24 mm x 115 mm) and bulb-to bulb distillation afforded 217 mg (71 %) of **3b** as a clear oil.

Data for 3b

bp: 65 °C (0.1 mm Hg, ABT)

¹H NMR: (500 MHz, CDCl₃)

8.15 (d, $J = 7.9$, 1 H, HC(8)), 7.87 (d, 1 H, HC(2)), 7.82 (d, $J = 8.4$, 1 H, HC(7)), 7.65 (dd, $J = 8.1$ and 1.3, 1 H, HC(4)), 7.56-7.44 (m, 4 H, HC(11), HC(6), HC(3), HC(5)), 5.83 (dd, $J = 17.5$ and 1.7, 1 H, H_{trans}C(12)), 5.51 (dd, $J = 10.9$ and 1.7, 1 H, H_{cis}C(12)).

¹³C NMR: (125 MHz, (CDCl₃))

135.9 (C(1)), 134.6 (C(11)), 133.8 (C(10)), 131.4 (C(9)), 128.8 (C(5), 128.4 (C(4)), 126.3 (C(7)), 126.0 (C(6)), 125.9 (3), 124.0 (C(8)), 123.9 (C(2)), 117.4 (C(12)).

MS: (EI, 70eV)
 154 (M^+ , 86), 153 (100), 152 (43), 76 (16).
 R_f : 0.50 (hexane)

Preparation of 1-Ethenyl-4-methoxybenzene (**3c**)⁴

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), 4-bromoanisole (374 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 10 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 370 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (20:1), SiO₂, 120 mm x 30 mm) and bulb-to-bulb distillation afforded 230 mg (86 %) of **3c** as a clear oil.

Data for **3c**

bp: 105 °C (20 mmHg, ABT)
 ^1H NMR: (500 MHz, CDCl₃)
 7.35 (d, J = 9.0, 2 H, HC(4)), 6.86 (d, J = 8.9, 2 H, HC(5)), 6.66 (dd, J = 17.6 and 10.9, 1 H, HC(2)), 5.62 (dd, J = 17.6 and 0.6, 1 H, H_{trans} C(1)), 5.12 (dd, J = 10.9 and 0.6, 1 H, H_{cis} C(1)), 3.81 (s, 3 H, HC(7)).
 ^{13}C NMR: (125 MHz, (CDCl₃)
 159.6 (C(6)), 136.4 (C(2)), 130.7 (C(3)), 127.6 (C(4)), 114.1 (C(5)), 111.8 (C(1)), 55.5 (C(7)).
IR: 3086 (w), 3004 (m), 2956 (m), 2908 (w), 2836 (m), 2551 (w) 2360 (w), 2062 (w), 1885 (w), 1812 (w), 1628 (s), 1607 (s), 1575 (m), 1511 (s), 1462 (m), 1442 (m),

1425 (w), 1408 (w), 1320 (w), 1301 (s), 1249 (s), 1174 (s), 1114 (m), 1040 (s), 1024 (m), 990 (m), 901 (m), 835 (s), 815 (m), 741 (w), 709 (w).

MS: (EI, 70eV)

135 ($M^+ + 1$, 12), 134 (100), 119 (40), 91 (65).

R_f: 0.2 (hexane/ethyl acetate, 100:1)

Preparation of 4-Ethenylbenzoic acid, Ethyl Ester (**3e**)⁶

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), ethyl 4-bromobenzoate (460 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 5 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 615 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (20:1), SiO₂, 120 mm x 30 mm) and bulb-to-bulb distillation afforded 290 mg (83 %) of **3e** as a clear oil.

Data for **3e**

bp: 135-140 °C (0.2 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

8.00 (d, $J = 8.3$, 2 H, HC(4)), 7.46 (d, $J = 8.3$, 2 H, HC(5)), 6.75 (dd, $J = 17.6$ and 10.9, 1 H, HC(2)), 5.86 (d, $J = 17.6$, 1 H, H_{trans}C(1)), 5.38 (d, $J = 10.9$, 1 H, H_{cis}C(1)), 4.38 (q, $J = 7.1$, 2 H, HC(8)), 1.40 (t, $J = 7.1$, 3 H, HC(9)).

¹³C NMR: (125 MHz, (CDCl₃))

166.6 (C(7)), 142.0 (C(3)), 136.3 (C(2)), 130.1 (C(5)), 129.9 (C(6)), 126.3 (C(4)), 116.6 (C(1)), 61.1 (C(8)), 14.5 (C(9)).

IR: 3067 (w), 2982 (m), 2936 (w), 2906 (w), 2873 (w), 2332 (w) 1945 (w), 1717 (s), 1600 (m), 1577 (w), 1568 (m), 1482 (m), 1464 (m), 1447 (w), 1390 (w), 1366 (m), 1225 (s), 1199 (w), 1171 (w), 1134 (s), 1076 (s), 917 (m), 855 (w), 771 (m), 757 (m), 715 (m).

MS: (EI, 70eV)
177 ($M^+ + 1$, 5), 176 (33), 148 (25), 132 (14), 131 (100), 103 (28), 77 (27)

***R_f*:** 0.14 (hexane/ethyl acetate, 20:1)

Preparation of 2-Ethenylbenzoic acid, Ethyl Ester (**3f**)⁷

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), ethyl 2-bromobenzoate (458 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 5 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of alumina (5 g). The alumina pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 615 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (25:1), SiO₂, 120 mm x 30 mm) and bulb-to-bulb distillation afforded 282 mg (86 %) of **3f** as a clear oil.

Data for **3f**

bp: 82 °C (0.1 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.87 (dd, $J = 7.9$ and 1.3 , 1 H, HC(7)), 7.58 (d, $J = 7.9$, 1 H, HC(4)), 7.48 (t, $J = 7.9$, 1 H, HC(5)), 7.46 (dd, $J = 17.6$ and 11.0 , 1 H, HC(2)), 7.32 (td, $J = 7.9$ and 1.2 , 1 H, HC(6)), 5.65 (dd, $J = 17.6$ and 1.2 , 1 H, H_{trans}C(1)), 5.35 (dd, $J = 10.9$ and 1.2 , 1 H, H_{cis}C(1)), 4.37 (q, $J = 7.2$, 2 H, HC(10)), 1.39 (t, $J = 7.1$, 3 H, HC(11)).

¹³C NMR: (125 MHz, (CDCl₃))
 167.5 (C(9)), 139.5 (C(3)), 135.9 (C(2)), 132.0 (C(7)), 130.2 (C(5)), 129.0 (C(8)),
 127.4 (C(4)), 127.2 (C(6)), 116.4 (C(1)), 61.0 (C(10)), 14.3 (C(11)).

IR: 3416 (w), 3089 (m), 3067 (m), 2982 (s), 2938 (m), 2905 (w), 2361 (w), 2339 (w),
 2256 (w), 1838 (w), 1717 (s), 1626 (w), 1599 (w), 1568 (m), 1482 (m), 1447 (w),
 1390 (m), 1366 (s), 1288 (s), 1254 (s), 1200 (m), 1171 (w), 1133 (s), 1076 (s),
 1022 (m), 990 (m), 917 (s), 855 (w), 771 (s), 716 (s).

MS: (EI, 70eV)
 177 (M⁺+1, 24), 176 (100), 149 (25), 148 (66), 147 (51), 132 (31), 131 (87), 130
 (12), 129 (11), 120 (21), 119 (41), 105 (30), 104 (12), 103 (58), 102 (36), 91 (34),
 77 (58), 76 (14), 75 (12), 74 (10), 63 (11).

R_f: 0.14 (hexane/ethyl acetate, 25:1)

Preparation of 2-Ethenylnaphthalene (**3g**)⁶

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) and 2-bromonaphthalene (414 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 3 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* and purified by column chromatography (pentane, SiO₂, 160 mm x 30 mm) gave 249 mg (81 %) of **3g** as a white solid.

Data for **3g**

mp: 64-66 °C

¹H NMR: (500 MHz, CDCl₃)
 7.89-7.92 (m, 3 H, HC(4), HC(5), HC(8)), 7.75 (bs, 1 H, HC(1)), 7.64 (dd, *J* = 8.6

and 2.0, 1 H, HC(3)), 7.46 (td, $J = 7.1$ and 1.8 , 1 H, HC(7)), 7.44 (td, $J = 7.1$ and 1.7 , 1 H, HC(6)), 6.88 (dd, $J = 17.6$ and 10.9 , 1 H, HC(11)), 5.88 (d, $J = 17.6$, 1 H, H_{trans}C(12)), 5.34 (dd, $J = 10.9$ and 1.4 , 1 H, H_{cis}C(12)).

¹³C NMR: (125 MHz, (CDCl₃))
136.9 (C(11)), 135.0 (C(2)), 133.5 (C(9)), 133.1 (C(10)), 128.2 (C(4)), 128.0 (C(8)), 127.7 (C(5)), 126.4 (C(1)), 126.2 (C(7)), 125.9 (C(6)), 123.2 (C(3)), 114.2 (C(12)).

IR: 3057 (m), 2922 (s), 2723 (s), 1702 (w), 1627 (w), 1591 (w), 1461 (s), 1376 (s), 1270 (w), 1168(w), 1124 (w), 986(m), 951 (w), 904 (m), 890 (w), 854 (m), 815 (m), 747 (m), 722 (m).

MS: (EI, 70eV)
154 (M⁺, 100), 153 (47), 152 (29), 128.1 (10), 76 (13).

R_f: 0.07 (hexane/ethyl acetate, 50:1)

Preparation of 1-Ethenyl-2-ethylbenzene (3h)⁸

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), 2-bromoethylbenzene (368 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 17 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 310 mg of crude material. Purification by column chromatography (pentane, SiO₂, 125 mm x 30 mm) and bulb-to-bulb distillation afforded 195 mg (75 %) of **3h** as a clear oil.

Data for **3h**

bp: 65 °C (10 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.49 (dd, *J* = 7.5 and 1.5, 1 H, HC(5)), 7.22-7.15 (m, 3 H, HC(4), HC(3), HC(2)), 7.00 (dd, *J* = 17.4 and 11.0, 1 H, HC(7)), 5.65 (dd, *J* = 17.3 and 1.5, 1 H, H_{trans}C(8)), 5.29 (dd, *J* = 11.0 and 1.5, 1 H, H_{cis}C(8)), 2.71 (q, *J* = 7.6, HC(9)), 1.20 (t, *J* = 7.6, 1 H, HC(10)).

¹³C NMR: (125 MHz, (CDCl₃))

141.8 (C(1)), 136.4 (C(6)), 134.8 (C(7)), 128.9 (C(3)), 128.1 (C(5)), 126.3 (C(4)), 125.9 (C(2)), 115.5 (C(8)), 26.5 (C(9)), 15.5 (C(10)).

IR: 3087 (w), 3061 (w), 3016 (w), 2966 (s), 2933 (m), 2874 (m), 2360 (w), 2341 (w), 1914 (w), 1813 (w), 1625 (w), 1599 (w), 1484 (m), 1467 (w), 1452 (m), 1408 (w), 1375 (w), 1270 (s), 1121 (s), 990 (m), 962 (w), 911 (m), 770 (s).

MS: (EI, 70eV)

133 (M⁺+1, 5), 132 (41), 131 (16), 129 (22), 128 (11), 118 (13), 117 (100), 116 (11), 115 (35), 91 (24).

R_f: 0.4 (pentane)

Preparation of 2-Ethenyl-1,3,5-trimethylbenzene (**3i**)⁹

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) and **82** (398 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. After 48 h, the reaction was allowed to cool to room temperature. Pentane (10 mL) was added and the mixture was stirred for 10 min at room temperature, then filtered twice through a short pad of silica (5 g). The silica pad was washed with pentane (50 mL) and the combined filtrates were concentrated *in vacuo* to afford 407 mg of crude material. Purification by column chromatography (pentane (20:1), SiO₂, 120 mm x 30 mm)

and bulb-to-bulb distillation afforded 211 mg (72 %) of **3i** as a clear oil, containing 5% of **2i** by ^1H -NMR analysis.

Data for 3i

bp: 60 °C (1 mmHg, ABT)

^1H NMR: (500 MHz, CDCl_3)

6.91 (s, 2 H, HC(5)), 6.71 (dd, $J = 18.0$ and 11.5 , 1 H, HC(2)), 5.54 (dd, $J = 11.5$ and 2.1 , 1 H, $\text{H}_{\text{cis}}\text{C}(1)$) 5.28 (dd, $J = 17.9$ and 2.1 , 1 H, $\text{H}_{\text{trans}}\text{C}(1)$), 2.32 (s, 6 H, HC(8)), 2.31 (s, 3 H, HC(7)).

^{13}C NMR: (125 MHz, CDCl_3)

136.4 (C(6)), 136.0 (C(4)), 135.2 (C(5)), 135.0 (C(3)), 128.7 (C(2)), 119.3 (C(1)), 21.1 (C(7)), 21.0 (C(8)).

IR: 3084 (m), 2999 (s), 2971 (s), 2948 (s), 2918 (s), 2859 (s), 2732 (w), 1842 (w), 1726 (w), 1630 (s), 1612 (s), 1567 (m), 1481 (s), 1442 (s), 1402 (m), 1376 (s), 1292 (s), 1206 (w), 1175 (w), 1147 (w), 1029 (s), 1015 (s), 994 (s), 919 (s), 850 (s), 754 (m), 713 (w), 697 (w), 687 (w), 664 (w).

MS: (EI, 70eV)

147 (M+1, 17), 146 (M+, 100), 145 (14), 131 (55), 129 (11), 115 (13), 91 (13).

R_f : 0.70 (pentane)

Preparation of 1-Ethenyl-2-methoxybenzene (3j)

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), 2-bromoanisole (374 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 10 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50

mL), and the combined filtrates were concentrated *in vacuo* to afford 297 mg of crude material. Purification by column chromatography (pentane/ether (100:1), SiO₂, 120 mm x 30 mm) and bulb-to-bulb distillation afforded 214 mg (80 %) of **3j** as a clear oil.

Data for 3j

bp: 80 °C (5 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.48 (dd, *J* = 7.7 and 1.7, 1 H, HC(3)), 7.27 (td, *J* = 8.4 and 1.7, 1 H, HC(5)), 7.06 (dd, *J* = 17.7 and 11.1, 1 H, HC(8)), 6.95 (td, *J* = 7.5 and 1.1, 1 H, HC(4)), 6.88 (d, *J* = 8.1, 1 H, HC(6)), 5.75 (dd, *J* = 17.6 and 1.5, H_{trans}C(9)), 5.28 (dd, *J* = 11.2 and 1.5, 1 H, H_{cis}C(9)), 3.86 (s, 3 H, HC(7)).

¹³C NMR: (125 MHz, CDCl₃)

156.7 (C(1)), 131.6 (C(8)), 128.8 (C(5)), 126.7 (C(2)), 126.5 (C(3)), 120.6 (C(4)), 114.1 (C(6)), 110.8 (C(9)), 55.4 (C(7)).

MS: (EI, 70eV)

135 (M⁺+1, 38), 134 (100), 121 (17), 119 (71), 118 (17), 105 (11), 97 (32), 92 (14), 91 (91), 90 (10), 85 (29), 78 (16), 77 (20), 73 (16), 65 (19), 63 (12), 59 (13).

***R*_f** 0.18 (hexane/ethyl acetate. 50:1)

Preparation of 4-Ethenylbenzenemethanol (3k)¹⁰

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), 4-bromobenzyl alcohol (374 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 14 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* and purified by column

chromatography (hexane/ethyl acetate (4:1), SiO₂, 120 mm x 30 mm) and bulb-to-bulb distillation afforded 145 mg (54 %) of **3k** as a clear oil.

Data for **3k**

bp.: 120 °C (0.1 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.40 (d, *J* = 7.9, 2 H, HC(4)), 7.31 (d, *J* = 7.9, 2 H, HC(5)), 6.72 (dd, *J* = 17.6 and 10.7, 1 H, HC(2)), 5.75 (d, *J* = 17.6, 1 H, H_{trans}C(1)), 5.25 (d, *J* = 10.9, 1 H, H_{cis}C(1)), 4.64 (s, 2 H, HC(7)), 1.94 (s, 1 H, -OH).

¹³C NMR: (125 MHz, (CDCl₃))

140.7 (C(6)), 137.2 (C(3)), 136.7 (C(2)), 127.4 (C(4)), 126.6 (C(5)), 114.1 (C(1)), 65.3 (C(7)).

IR: 3338 (bs), 3087 (m), 3051 (m), 3022 (m), 3006 (m), 2928 (w), 2873 (m), 2360 (w), 2342 (w), 1908 (w), 1818 (w), 1629 (s), 1569 (m), 1512 (s), 1454 (w), 1406 (s), 1367 (w), 1316 (w), 1288 (w), 1211 (m), 1115 (m), 1024 (s), 1013 (s), 991 (s), 947 (w), 908 (s), 847 (s), 824 (s), 718 (m).

MS: (EI, 70eV)

135 (M⁺+1, 11), 134 (97), 133 (37), 117 (19), 115 (17), 108 (13), 107 (26), 105 (100), 91 (36), 79 (74), 77 (65), 63 (17).

R_f: 0.20 (hexane/ethyl acetate, 4:1)

Preparation of 1-Ethenyl-2-nitrobenzene (3l**)**¹¹

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.6 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv), 2-bromonitrobenzene (404 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. GC analysis at 2 h indicated that no bromide remained, and the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered

through a short pad of alumina (5 g). The alumina pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 534 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (25:1), SiO₂, 140 mm x 30 mm) and Kugelrohr distillation afforded 270 mg (91 %) of **3I** as a yellow oil.

Data for 3I

bp: 65 °C (0.1 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.92 (dd, *J* = 8.3, and 1.2, 1 H, HC(7)), 7.62 (dd, *J* = 7.8, and 1.4, 1 H, HC(4)), 7.59 (td, *J* = 8.1 and 1.1, 1 H, HC(5)), 7.41 (td, *J* = 8.3 and 1.5, 1 H, HC(6)), 7.17 (dd, *J* = 17.4 and 10.9, 1 H, HC(2)), 5.74 (d, *J* = 17.4, 1 H, H_{trans}C(1)), 5.48 (d, *J* = 10.9, 1 H, H_{cis}C(1)).

¹³C NMR: (125 MHz, CDCl₃)

133.5 (C(3)), 133.4 (C(7)), 132.7 (C(2)), 128.7 (C(5)), 128.6 (C(4)), 127.3 (C(8)), 124.6 (C(6)), 119.2 (C(1)).

IR: 3094 (w), 3071 (m), 2958 (s), 2927 (s), 2871 (s), 2859 (s), 2375 (w), 1943 (w), 1855 (w), 1736 (w), 1608 (m), 1570 (s), 1526 (s), 1467 (m), 1444 (w), 1413 (w), 1349 (a), 1302 (m), 1269 (w), 1078 (bs), 982 (s), 857 (s), 783 (s), 760 (s), 709 (m), 668 (m).

MS: (EI, 70eV)

150 (M⁺+1, 7), 149 (M⁺, 4), 132 (50), 121 (16), 120 (59), 104 (21), 103 (14), 102 (23), 93 (21), 92 (35), 91 (100), 89 (11), 78 (11), 77 (66), 76 (18), 75 (15), 74 (14), 66 (18), 65 (57).

R_f: 0.17 (hexane/ethyl acetate, 25:1)

Preparation of N-(4-Ethenylphenyl)-acetamide (3m)¹²

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) and **2m** (428 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. At 12 h, the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature, after which the mixture was partitioned between dichloromethane (100 mL) and an aqueous solution of 2-(*N,N*-dimethylamino)ethanethiol (1 %, 100 mL), washed with water (100 mL), and back extracted with ethyl acetate. The combined organics were combined, dried over Na₂SO₄, filtered, and concentrated *in vacuo* to afford 654 mg of crude material. Purification by column chromatography (ethyl acetate/hexane (1:1), SiO₂, 120 mm x 30 mm) and recrystallization from isopropanol afforded 249 mg (77%) of **3m** as a white solid.

Data for **3m**

mp: 133-134 °C

¹H NMR: (500 MHz, CDCl₃)
 7.47 (d, *J* = 8.1, 2 H, HC(4)), 7.36 (d, *J* = 8.5, 1 H, HC(5)), 7.20 (bs, 1 H, -NH)
 6.67 (dd, *J* = 17.6 and 10.8, 1 H, HC(2)), 5.68 (d, *J* = 17.6, 1 H, HC_{trans}(1)), 5.19 (d, *J* = 10.8, 1 H, HC_{cis}(1)), 2.18 (s, 3 H, HC(8)).

¹³C NMR: (125 MHz, (CDCl₃)
 168.7 (C(7)), 137.7 (C(6)), 136.3 (C(2)), 133.9 (C(3)), 127.0 (C(4)), 120.1 (C(5)), 113.2 (C(1)), 24.7 (C(8)).

IR: 3288 (m), 3178 (w), 3108 (w), 1660 (m), 1626 (m), 1592 (w), 1513 (w), 1401 (m), 1323 (w), 1266 (w), 994 (w), 905 (w), 842 (w).

MS: (EI, 70eV)
 162(M⁺+1, 5), 161 (M⁺, 45), 120 (11), 119 (100), 118 (24), 91 (12).

R_f: 0.27 (hexane/ethyl acetate, 1:1)

Preparation of 2-Ethenyl-*N,N*-dimethylbenzenamine (3n)¹³

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.6 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.5 equiv) and *N,N*-dimethyl-2-bromoaniline (325 mg, 1.6 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. After 24 h, the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* affording 346 mg. Purification by column chromatography (hexane/ethyl acetate (100:1), SiO₂, 120 mm x 30 mm) followed by bulb-to-bulb distillation afforded 169 mg of a mixture of **3n** to the dimethylaniline (10:1 by ¹H-NMR) as a clear oil.

Data for 3n

bp: 65-70 °C (0.5 mm Hg, ABT)

¹H NMR: (500 MHz, CDCl₃)

7.49 (dd, *J* = 7.6 and 1.2, 1 H, HC(5)), 7.29-7.24 (m, 1 H, HC(3)), 7.11-7.01 (m, 3 H, HC(7), HC(2), HC(4)), 5.70 (dd, *J* = 17.8 and 1.4, 1 H, HC_{trans}(8)), 5.27 (dd, *J* = 11.0 and 1.5, 1 H, HC_{cis}(8)), 2.75 (s, 3 H, HC(9)).

¹³C NMR: (125 MHz, (CDCl₃))

135.1 (C(7)), 132.0 (C(1)), 129.3 (C(6)), 128.5 (C(3)), 127.1 (C(4)), 122.5 (C(5)), 118.2 (C(2)), 113.4 (C(8)), 44.8 (C(9)).

IR: 3440 (w), 3083 (w), 3064 (w), 3027 (w), 2980 (m), 2940 (m), 2863 (m), 2829 (m), 2781 (m), 1902 (w), 1821 (w), 1624 (m), 1596 (s), 1507 (m), 1487 (s), 1453 (s), 1431 (w), 1411 (w), 1316 (s), 1190 (m), 1156 (m), 1146 (m), 1097 (w), 1057 (w), 1022 (w), 1005 (m), 948 (s), 906 (s), 800 (w), 757 (s), 716 (w), 691 (w).

MS: (EI, 70eV)

148 (M⁺+1, 33), 147 (M⁺, 100), 146 (17), 133 (17), 132 (87), 131 (14), 130.1 (16), 118 (12), 117 (21).

R_f : 0.37 (hexane/ethyl acetate, 50:1)

Preparation of 3-Ethenylquinoline (**3o**)¹⁴

Following General Procedure V, 2-(di-*t*-butylphosphino)biphenyl (59.4 mg, 0.20 mmol, 0.10 equiv), palladium bromide (26.6 mg, 0.1 mmol, 0.05 equiv), a solution of 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (344 mg, 1.0 mmol, 0.5 equiv) in THF (0.1 mL), TBAF (4 mL, 1 M in THF, 4 mmol, 2.0 equiv) and 3-bromoquinoline (416 mg, 2.0 mmol) were combined, and the reaction was warmed to 50 °C in an oil bath. At 3 h, the reaction was allowed to cool to room temperature. Diethyl ether (10 mL) was added and the mixture was stirred for 10 min at room temperature then filtered through a short pad of silica (5 g). The silica pad was washed with diethyl ether (50 mL), and the combined filtrates were concentrated *in vacuo* to afford 542 mg of crude material. Purification by column chromatography (hexane/ethyl acetate (4:1), SiO₂, 140 mm x 30 mm) and bulb-to-bulb distillation afforded 278 mg (89 %) of **3o** as a clear oil.

Data for **3o**

bp: 120 °C (0.1 mmHg, ABT)

¹H NMR: (500 MHz, CDCl₃)

9.02 (d, $J = 1.9$, 1 H, HC(1)), 8.08 (d, $J = 8.5$, 1 H, HC(8)), 8.07 (d, $J = 2.5$, 1 H, HC(3)) 7.79 (d, $J = 8.3$, 1 H, HC(5)), 7.67 (t, $J = 7.6$, 1 H, HC(6)), 7.53 (t, $J = 7.8$, 1 H, HC(7)), 6.86 (dd, $J = 17.6$ and 11.0, 1 H, HC(10)), 5.98 (d, $J = 17.5$, 1 H, H_{trans}C(11)), 5.46 (d, $J = 11.0$, 1 H, H_{cis}C(11)).

¹³C NMR: (125 MHz, (CDCl₃))

149.0 (C(1)), 147.4 (C(9)), 133.7 (C(10)), 130.4 (C(3)), 129.4 (C(8)), 129.1 (C(7)), 128.0 (C(4)), 127.9 (C(5)), 127.0 (C(6)), 116.4 (C(11)).

IR: 3385 (m), 3088 (m), 3065 (m), 2291 (w), 1958 (w), 1839 (w), 1701 (w), 1633 (m), 1618 (m), 1570 (m), 1494 (s), 1462 (w), 1429 (w), 1413 (w), 1370 (s), 1328 (w), 1298 (w), 1233 (w), 1199 (w), 1126 (s), 1055 (w), 1024 (m), 988 (s), 976 (s), 957 (w), 910 (s), 861 (m), 787 (s), 753(s).

MS: (EI, 70eV)

156 ($M^+ + 1$, 15), 155 (100), 154 (44), 128 (12) 127(15).

R_f: 0.28 (hexane/ethyl acetate, 4:1)

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CRB-V-021C

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SAMPLE	DATE	MAY 23 2005	DIRECT	499.699
SOLVENT:	CHEM33	dm	HL	20
FILE	exp	dpr	dot	0
ACQUISITION	499.699	dm	num	c
IN	4.665	dm	c	200
AT	85336	deg	at	1.0
IDP	7024.5	area		n
AV	4000	homo		
FB				
BS				
TYP	6	FILES		
PW	5.8	proc	ft	
DL	0	zn	not used	f
TOF	2.0	match		
NT	32			
CT	24	wave		
ALOOK		used		
GAIN	not used	whs		
FUDGE		wtc		
IL	n			
LN	y			
DP	na			
BS				
DISPLAY	-750.0			
WD	5456.5			
SC	41			
ST	0			
WD	400			
NUM	21.99			
LS	1770.08			
KFL	10.6.4			
KFD	0			
LH	7			
LAS	1.000			
LI	ph

exp1 s2pul

NAME: CHIRV-0314 DEC. 6 VT
 Date: Jun 20 2005 dfrq: 500.075
 solvent: CDCl3 dm
 file: /export/home/- dfrq: 18
 data: /u15000b/butla- dfrq: 0
 r/CHIRV-0314.fid dm
 ACQUISITION: dm
 sfrq: 500.075 dmf 200
 ca: 1.0
 at: 4.089 dmf
 no: 65536 dmf
 av: 8000.0 PROCESSING: 0.30
 fd: 4000 lb
 be: 16 wfFile
 tpr: 55 proc ft
 pw: 7.6 ft not used
 dl: 0 match
 tof: 0
 nt: 4 wear
 ct: 4 wear
 alock: n who
 gain: not used wnt
 TRACE: not used wnt

11 n
 1n n
 4p y
 1s nm
 DISPLAY: -250.1
 wd: 5500.7
 vs: 57
 sc: 0
 wc: 400
 bnm: 13.75
 ls: 1432.27
 rfi: 5132.8
 rfp: 3630.5
 1n 7
 1n 1.000
 at ph

exp1 adjnt1

SAMPLE DRC. & VR
 date Oct 14 2005 drcq 500.075
 solvent CDCl3 dn H1
 file exp drcq 18
 ACQUISITION
 sfile 500.075 dm 0
 cu H1 dm c
 ac 20.000 dnt 200
 ap 32000 drcq
 av 8000.0 drcq 1.0
 eb 4000 homo n
 bs 15
 bps 35 drcq2 DRC2 0
 pw 7.8 dnt
 dl 23.000 drcq2 1
 tot 0 dnt2 0
 nt 1 dnt2 n
 ct 1 dnt2 c
 alock not used
 gain not used dnt2 200
 F1008
 1.1 n homo2 1.0
 1.1 n PROCESSING
 1.1 y lb 0.30
 bs mn vfile
 DISPLAY
 sp -450.1 kn not used
 wp 5500.8 mch f
 vs 38
 wt 0 WERT
 wc 400 wexp
 hwmn 13.75 wha
 ls 719.25 wnt
 rfi 5132.6
 rfo 3630.5
 ch 7
 fms 1.000
 al ph

96

INDEX	FREQUENCY(PPM)	FREQUENCY(Hz)	HEIGHT
1	7.497	3746.037	12.1
2	7.493	3744.322	12.1
3	7.481	3738.319	12.7
4	7.478	3736.604	12.7
5	7.270	3632.839	6.3
6	7.267	3631.125	10.0
7	7.259	3624.477	10.0
8	7.253	3623.834	9.7
9	7.252	3617.188	8.4
10	7.235	3615.473	8.0
11	7.098	3546.868	9.8
12	7.076	3535.720	10.1
13	7.062	3529.074	10.3
14	7.062	3517.925	10.4
15	7.040	3480.621	8.3
16	6.985	3480.192	8.3
17	6.985	3479.549	8.0
18	6.983	3479.120	14.5
19	6.950	3472.475	14.7
20	6.949	3465.614	7.2
21	6.935	3465.185	7.1
22	6.932	3464.542	6.6
23	6.932	3464.604	16.3
24	6.893	3436.457	16.6
25	6.877	3435.843	18.5
26	5.771	2882.043	11.0
27	5.768	2881.963	11.2
28	5.735	2864.446	18.7
29	5.732	2862.440	18.0
30	5.290	2632.482	17.9
31	5.287	2630.891	205.2
32	5.268	1928.862	5.3
33	3.860	1915.570	11.3
34	3.833	1915.570	11.3
35	1.555	777.155	7.2
36	0.901	448.995	7.2

exp1 #2pul

SAMPLE DMC. & VT
 date Sep 23 2005 dir 500.075
 solvent dm CDCl3 dm H1
 file exp dprc 18
 ACQUISITION
 #freq 500.075 dm 0
 nu H1 dm c
 at 4.096 dmf 200
 ng 65536 dseq
 sw 8000.0 dres 1.0
 fb 4000 homo n
 bs 16
 DMC2
 tpwr 55 dtrg2 0
 pw 7.6 dtr 1
 dl 0 dprc2 1
 tof 0 dtr2 0
 nt 4 dm2 n
 ct 4 dm2 c
 alock n dm2 200
 gain not used dtrg2
 YLOS not used dtrg2 1.0
 ll n homo2 n
 ln n PROCESSING
 dp y lb 0.30
 bs nm vefile
 DISPLAY
 sp -250.1 fm not used
 wp 5500.7 msh f
 vs 286
 ac 0 wear
 wc 400 wear
 hnm 13.75 whs
 ls 3205.94 wnt
 zfi 5123.6
 xfp 3630.5
 ca 7
 ins 1.000
 al ph

exp1 23jul

SAMPLE DEC. 2 VT
 Date Oct 12 2005 dly 500.075
 solvent CDCl3 dm K1
 file exp dly 18
 ACQUISITION 0
 dly 500.075 dm 0
 km 4.086 dm 200
 ac 65536 dm 1.0
 ap 8000.0 dm 25.0
 eb 4000 dm 0
 bs 15 temp 35
 type 7.6 dly2 0
 pw 1.000 dm 1
 dt 0 dly2 0
 nt 4 dly2 0
 ct 4 dm 2
 allock n dm 2
 gain not used dm 2 200
 PRMS n dm 2 1.0
 in n dm 2
 dp 7 PROCESSING 0.30
 bs 15
 DISPLAY
 -250.0 proc fit
 up 5500.7 fm not used
 vs 54 match 2
 ac 0
 wv 400 wvnt
 hnum 13.75 wvnt
 fa 1479.11 wvnt
 rfi 1499.5 wvnt
 rfo 0
 ch 7
 lsm 200.000
 at ph

SAMPLE DEC. 6 YR
 date Oct 6 2005 dfrq 500.075
 solvent CMC13 du H1
 file /export/home/-/dpr 18
 date/t1500h/Date-00f 0
 r/CBV-014C-fid dm mm
 ACQUISITION dm c
 freq 500.075 dmf 200
 nu H1 deeq 1.0
 at 4.096 dres n
 nu 65336 homo n
 sb 8000.0 lb PROCESSING 0.30
 bs 4000 1b wfile n
 uper 35 proc ft
 pw 7.6 En not used f
 dl 2.000 smth f
 tof 0
 nt 4 weat
 ct 4 weat
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exp1 #3pm1

SAMPLED DMC. & VP
 file May 26 2005 dfrq 500.075
 solvent CHCl3 dm H1
 exp dfrq 18
 ACQUISITION
 dfrq 500.075 dm 0
 nu 500.075 nm 200
 at 4.096 dmf c
 md 65536 dfrq 1.0
 sw 8000.0 dfrq 1.0
 fb 4000 homo n
 bs 15 DMC2
 bprz 35 dfrq3 0
 dl 7.6 dm1
 tod 0 dfrq2 1
 nt 0 dm2 0
 ct 4 dm1 n
 alock n dm2 200
 gain not used dfrq3 1.0
 r1a08 n homo2 n
 f1 n PROCESSING
 f1a n 0.30
 dfrq 7 lb
 bs nm write
 DISPLAY -1499.3 fm not used
 up 7999.8 match f
 vs 285
 ac 0 water
 wo 400 wrap
 ls 13.75 wte
 ls 3893.89 wte
 r1l 1499.5
 r1p 0
 h 7
 -ms 1.000
 al ph

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