Supporting Information
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Supporting Information

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General experimental details

1H NMR spectra were recorded on Bruker DPX 400 or 500 spectrometers operating at 400 and 500 MHz respectively. 13C NMR spectra were recorded on Bruker 400 or 500 spectrometers operating at 100 and 125 MHz respectively. Chemical shifts are quoted relative to residual solvent (7.26 ppm for CHCl3 and 77.0 ppm for 13C of CDCl3), and coupling constants (J) are given in Hz, to the nearest 0.1 Hz. The following abbreviations are used singularly or in combination to indicate the multiplicity of signals: s singlet, d doublet, t triplet, q quartet, sept septet, m multiplet and b broad. NMR spectra were acquired at 300 K unless otherwise indicated. High resolution mass spectroscopic (HRMS) analyses were measured on a Micromass Q-TOF or a Micromass LCT Premier spectrometer at the Department of Chemistry, University of Cambridge or on a Finnigan MAT 900 XLT or a Finnigan MAT 95XP spectrometer at the EPSRC National Mass Spectrometry Service Centre, Swansea. Infrared spectra were recorded on a Perkin Elmer 1 FT-IR Spectrometer fitted with an Attenuated Total Reflectance (ATR) sampling accessory as neat films. Selected absorption maxima (υmax) are reported in wavenumbers (cm⁻¹). Melting points were obtained using a Reichert hot plate microscope with a digital thermometer attachment and are uncorrected. Reactions were carried out in oven-dried glassware under an atmosphere
of nitrogen with dry, freshly distilled solvents. Tetrahydrofuran was distilled from LiAlH₄ and CaH₂ with triphenylmethane as indicator. All chemicals were purchased from Sigma-Aldrich or Strem. Grignard reagent and alkyllithium solutions were titrated with 1,10-phenanthroline and menthol before use. All flash chromatography was carried out using dry-packed Merck 9385 Kieselgel 60 silica gel. Temperatures of ~ 5°C were maintained with an ice-water bath; temperatures of -78 °C were maintained with a dry ice/acetone bath. Unless otherwise stated, 'pet. ether' refers to the fraction boiling between 40 °C and 60 °C.

Experimental details

1.1.1. Dimethyl(methylthiomethyl)silane (12)

\[
\begin{array}{c}
\text{S} \\
\text{Si} \\
\text{H} \\
\end{array}
\]

Freshly-distilled TMEDA (7.5 mL, 50.0 mmol) was added, dropwise, to a stirred solution of n-butyllithium (2.00 M, 25 mL in hexanes, 50.0 mmol) cooled to ~ 5 °C (ice bath). On completion of this addition, the reaction was stirred for 30 minutes, then anhydrous dimethyl sulfide (3.65 mL, 50.0 mmol) was added and the reaction stirred for four hours at room temperature, during which time a white precipitate was observed to form. Ether (40 mL) was added, the reaction was cooled to -78°C and dimethylchlorosilane (6.5 mL, 60.0 mmol) was added; on completion of this addition, the reaction was slowly warmed to room temperature and stirred at room temperature for 12 hours. Saturated aqueous ammonium chloride (50 mL) was then added, the resulting suspension was filtered, and the organic layer was separated and dried over MgSO₄. The solvent was removed by atmospheric pressure distillation (bath temperature of 60 °C, and the residual material was then fractionated using a 10 cm Vigreux column to give a clear 1.56 g fraction boiling at (62 °C, 294 mmHg) which, on NMR analysis, was found to be the title compound (26 % yield).

\[\delta_\text{H} \ (400 \text{ MHz; CDCl}_3): \ 3.96 \ (1\text{H, m, 4}), \ 2.16 \ (3\text{H, s, 1}), \ 1.82 \ (2\text{H, d, 3.2 Hz, 2}), \ 0.16 \ (6\text{H, d, 3.6 Hz, 3})\]

\[\delta_\text{C} \ (100 \text{ MHz; CDCl}_3): \ 19.20, \ 17.87, \ -5.19\]

\[\nu_{\text{max}}/\text{cm}^{-1}: \ 2124.9 \ (s) \ (\text{Si-H})\]

Owing to the low molecular weight of this compound, it was not possible to obtain an accurate high resolution mass spectrum. However, the spectroscopic data above together with its successful use in the hydrosilylation of phenylacetylene to form 16 strongly suggest that the species isolated is indeed the title compound.
1.1.2. Dimethylbis(methylthiomethyl)silane (14)

Freshly-distilled TMEDA (7.5 mL, 50.0 mmol) was added, dropwise, to a stirred solution of n-butyllithium (1.60 M, 31.25 mL in hexanes, 50.0 mmol) cooled to ~5 °C (ice bath). On completion of this addition, the reaction was stirred for 30 minutes, then anhydrous dimethyl sulfide (3.65 mL, 50.0 mmol) was added and the reaction stirred for four hours at room temperature, during which time a white precipitate was observed to form. The reaction was then cooled to -78 °C and dimethylchlorosilane (6.5 mL, 60.0 mmol) was added; on completion of this addition, the reaction was slowly warmed to room temperature and stirred at room temperature for 12 hours. Saturated aqueous ammonium chloride (50 mL) was then added, the resulting suspension was filtered, and the organic layer was separated and dried over MgSO₄. The solvent was removed by atmospheric pressure distillation; the residual material, when analysed by NMR, proved to be the title compound (2.62 g, 58 % of theoretical maximum).

δ_H (400 MHz; CDCl₃): 2.16 (6H, s, 1), 1.87 (4H, s, 2), 0.17 (6H, s, 3)
δ_C (100 MHz; CDCl₃): 20.7 (1), 20.1 (2), -3.0 (3)

υ_max/cm⁻¹: no significant absorbances >1500 cm⁻¹

m/z (ESI⁺): Found [M+Na]⁺, 203.0367 (C₆H₁₆NaS₂Si requires 203.0360, Δppm = 3.45)

1.1.3. (E)-(methylthiomethyl)dimethyl(styryl)silane (16)

A two-necked flask fitted with a reflux condenser was charged with toluene (5 mL), Pt(DVDS) (0.5 mL of a 0.1 M solution in poly(dimethyl)siloxane, 0.05 mmol) and P'Bu₃ (0.05 mL of a 1.0 M solution in toluene, 0.05 mmol), and the resulting solution was stirred at room temperature for five minutes. The silane 12 (1.25 g of a solution in THF/ether, approx. 64 % by mass, approx. 6.0 mmol) was then added, followed by phenylacetylene 15 (670 μL, 6.0 mmol), and the reaction was stirred at 60 °C for 12 hours. It was then concentrated under reduced pressure and purified by flash chromatography (4:1 hexanes:DCM, R_f=0.34) to give the title compound as a yellow oil (902 mg, 65 %).
δ_1H (400 MHz; CD_2Cl_2): 7.47 (2H, d, 7.0 Hz, 3), 7.36 (2H, t, 7.2 Hz, 2), 7.28 (1H, tt, 7.2 Hz, 1.2 Hz, 1), 6.98 (1H, d, 19.2 Hz, 4), 6.50 (1H, d, 19.2 Hz, 5), 2.18 (3H, s, 8), 1.93 (2H, s, 7), 0.27 (6H, s, 6)

δ_C (100 MHz; CDCl_3): 145.07, 138.01, 128.48, 128.17, 126.51, 126.47, 20.65, 20.33, -3.10

υ_{max}/cm^{-1}: 1686.6 (w), 1603.5 (s), 1573.9 (s)

m/z (ESI^+): Found [M+H]^+, 223.0970 (C_{12}H_{18}SSi requires 223.0971, Δppm = +0.6)

1.1.4. (Chloromethyl)dimethyl(phenylethynyl)silane (20)

\[ \text{\begin{tikzpicture}
\draw (0,0) -- (1,1) -- (2,0) -- (1,-1) -- cycle;
\draw (1,1) -- (2,2) -- (3,1) -- cycle;
\draw (2,0) -- (3,0);
\end{tikzpicture}} \]

n-Butyllithium (6.5 mL of a 1.55 M solution in hexanes, 10.0 mmol) was added slowly to a stirred solution of phenylacetylene 15 (1.2 mL, 11.0 mmol) in ether (20 mL) at ~5 °C (ice bath). On completion of the addition, the solution was stirred for 30 minutes, then chloromethyldimethylchlorosilane (1.33 mL, 10.0 mmol) was added and the reaction was warmed to room temperature. After stirring for one hour, saturated aqueous ammonium chloride (20 mL) was added and the precipitated salts were removed by filtration. The filtrate was washed with water (20 mL) and brine (20 mL), then dried over MgSO_4, concentrated under reduced pressure, and purified by distillation (66 °C, 0.3 mmHg) to furnish the title compound as a clear oil (1.93 g, 93 %).

δ_1H (400 MHz; CDCl_3): 7.48 (2H, dd, 8 Hz, 1.2 Hz, 4), 7.36 - 7.28 (3H, m, 5 and 3), 2.94 (2H, s, 1), 0.38 (6H, s, 2)

δ_C (100 MHz; CDCl_3): 133.41, 130.28, 129.59, 123.79, 108.27, 91.53, 31.40, -1.86

υ_{max}/cm^{-1}: 2158.9 (s) (alkyne)

m/z (ESI^+): Found [M+Na]^+, 231.065 (C_{11}H_{13}^{35}ClNaSi requires 231.0373, Δppm = -3.46)

1.1.5. (Z)-(chloromethyl)dimethyl(styryl)silane (29)

\[ \text{\begin{tikzpicture}
\draw (0,0) -- (1,1) -- (2,0) -- (1,-1) -- cycle;
\draw (1,1) -- (2,2) -- (3,1) -- cycle;
\draw (2,0) -- (3,0);
\end{tikzpicture}} \]

A stirred solution of borane-THF complex (1.0 M in THF, 5.5 mL, 5.5 mmol) was cooled to ~5 °C (ice bath), and cyclohexene (1.11 mL, 11.0 mmol) was added dropwise; on completion of this addition, the cooled reaction was stirred for 15
minutes, during which time a white precipitate formed. The reaction was then warmed to room temperature and stirred for one hour, then cooled to ~ 5 °C. Silylalkyne 20 (1.04 g, 5.0 mmol) was then added dropwise, and the cooled reaction was stirred for 30 minutes, then warmed to room temperature and stirred for one hour. It was then cooled to ~5 °C again, and glacial acetic acid (2.5 mL) was added; on completion of this addition, the reaction was warmed to room temperature and stirred for two hours. The reaction mixture was then poured into ether (30 mL) and washed with water (4 x 30 mL) and brine (1 x 30 mL), then dried over MgSO₄ and concentrated under reduced pressure. The crude product so obtained was purified by flash chromatography (pet. ether, Rf= 0.52) to furnish the title compound as a clear oil (610 mg, 58 %).

δ_H (400 MHz; CDCl₃): 7.49 (1H, d, 16.0 Hz, 4), 7.30 (5H, m, Ph), 5.83 (1H, d, 16.0 Hz, 3), 2.74 (2H, s, 1), 0.14 (6H, s, 2)
δ_C (100 MHz; CDCl₃): 149.45, 140.31, 128.84, 128.51, 128.27, 128.18, 31.52, -2.43
υ_max/cm⁻¹: 1591.7, 1571.2
m/z (ESI⁺): Found [M+Na]⁺, 233.0525 (C₁₁H₁₅ClNaSi requires 233.0529, Δppm = -1.71)

1.1.6. ((pent-4-yn-1-yloxy)methyl)benzene (22)

4-pentyn-1-ol (2.31 mL, 27.5 mmol) was added dropwise to a stirred suspension of sodium hydride (1.3 g of a 60 % dispersion in mineral oil, 33.0 mmol) in THF (25 mL) and stirred for 15 minutes. Benzyl bromide (3.6 mL, 30.0 mmol) was then added, and the reaction mixture was heated at reflux for 14 hours. It was then cooled to room temperature and quenched by the addition of saturated aqueous ammonium chloride. The organic layer was separated, washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure to afford the crude product, which was purified by flash chromatography (pet. ether; Rf = 0.41) to afford the title compound as a colourless oil (2.92 g, 61 %).

δ_H (400 MHz; CDCl₃): 7.38 - 7.25 (5H, m, Ph), 4.52 (2H, s, 5), 3.58 (2H, t, 6.4 Hz, 4), 2.32 (2H, td, 7.2 Hz, 2.8 Hz, 2), 1.95 (1H, t, 2.8 Hz, 1), 1.84 (2H, app. quintet, 6.4 Hz, 3)
δ_C (100 MHz; CDCl₃): 138.9, 128.8, 128.1, 128.0, 84.4, 73.4, 69.1, 68.9, 29.1, 15.7

Spectroscopic data match literature values.¹
1.1.7. (5-(benzyloxy)pent-1-en-2-yl)(chloromethyl)dimethylsilane (23)

A solution of alkyne 22 (2.71 g, 15.6 mmol) and Cp*Ru(MeCN)₃.PF₆ (81 mg, 0.16 mmol) in acetone (16 mL) was cooled to ~5 °C. Chloromethyl(dimethyl)silane (2.0 mL, 16.4 mmol) was added dropwise, and the reaction mixture was stirred with cooling for two hours. It was then concentrated under reduced pressure and purified directly by flash chromatography (29:1 pet. ether:ethyl acetate, Rf = 0.34) to afford the title compound as a colourless oil (4.13 g, 94%).

δ_H (400 MHz; CDCl₃): 7.37 - 7.27 (5H, m, Ph), 5.69 - 5.67 (1H, m, 5), 5.42 - 5.41 (1H, m, 6), 4.50 (2H, s, 1), 3.48 (2H, t, 6.4 Hz, 2), 2.83 (2H, s, 8), 2.22 (2H, t, 7.6 Hz, 4), 1.74 (2H, m, 3), 0.22 (6H, s, 7)

δ_C (100 MHz; CDCl₃): 148.3, 138.9, 128.8, 128.1, 128.0, 127.0, 73.3, 70.2, 32.7, 30.6, 29.3, -4.2

υ_{max}/cm⁻¹: 1603.0 (w)

m/z (ESI⁺): Found [M+Na]⁺, 305.1109 (C₁₅H₂₃SiClNa requires 305.1104, Δppm = 1.64)

1.1.8. General procedure 1: S_N2 displacement with potassium ethanethiolate

The alkenyl(chloromethyl)silane was dissolved in methanol to give a 0.2 M solution. Potassium carbonate (1.5 equivalents) was then added, and the reaction vessel was sparged with nitrogen for 15 minutes. Ethanethiol (1.1 equivalents) was then added, and the reaction was stirred at room temperature for 16 hours. The reaction mixture was then concentrated under reduced pressure, and the residue was partitioned between water and ether. The organic layer was separated, and the aqueous was extracted twice with ether. The combined organic extracts were washed with brine, dried (MgSO₄), filtered, and concentrated under reduced pressure to furnish the crude product, which was purified by flash chromatography.

1.1.9. (Z)-(ethylthiomethyl)dimethyl(styryl)silane (18)

According to general procedure 1, (Z)-(chloromethyl)dimethyl(styryl)silane 29 (400 mg, 1.90 mmol) in methanol (4.8 mL) was treated with pulverised potassium carbonate (393 mg, 2.85 mmol) and ethanethiol (365 µL, 4.80 mmol). Purification by
flash chromatography (19:1 pet. ether: ethyl acetate, Rf = 0.35) furnished the title compound as a colourless oil (381 mg, 85 %).

δH (400 MHz; CDCl3): 7.46 (1H, d, 14.8 Hz, 6), 7.34 - 7.28 (5H, m, Ph), 5.87 (1H, d, 14.8 Hz, 5), 2.49 (2H, q, 7.2 Hz, 2), 1.81 (2H, s, 4), 1.22 (3H, t, 7.2 Hz, 1), 0.12 (6H, s, 4)

δC (100 MHz; CDCl3): 148.04, 140.00, 130.22, 128.03, 127.97, 127.54, 29.95, 17.85, 14.18, -1.58

υmax/cm⁻¹: 1592.4, 1580.6

m/z (ESI⁺) Found [M+H]⁺, 237.1126 (C13H20SSi requires 237.1128, Δppm = +0.9)

1.1.10. (5-(benzyl oxy)pent-1-en-2-yl)((ethylthio)methyl)dimethylsilane (21)

According to general procedure 1, α-(chloromethyl)dimethyl(styryl)silane 23 (1.00 g, 3.54 mmol) in methanol (7 mL) was treated with pulverised potassium carbonate (1.46 g, 10.6 mmol) and ethanethiol (511 µL, 3.54 mmol). Purification by flash chromatography (39:1 pet. ether: ethyl acetate, Rf = 0.35) furnished the title compound as a colourless oil (1.03 g, 94 %).

δH (400 MHz; CDCl3): 7.33 - 7.25 (5H, m, Ph), 5.64 (1H, m, 5), 5.40 (1H, m, 6), 4.50 (2H, s, 1), 3.48 (2H, t, 6.8 Hz, 2), 2.52 (2H, q, 7.6 Hz, 9), 2.22 (2H, t, 7.6 Hz, 4), 1.85 (2H, s, 8), 1.74 (2H, tt, 7.6 Hz, 6.8 Hz, 3), 1.24 (3H, t, 7.6 Hz, 10), 0.17 (6H, s, 7)

δC (100 MHz; CDCl3): 149.8, 139.0, 128.8, 128.1, 127.9, 126.0, 73.3, 70.4, 32.6, 30.5, 29.3, 17.0, 14.6, -2.9

υmax/cm⁻¹: 1496.0, 1453.7

m/z (ESI⁺) Found [M+Na]⁺, 331.1532 (C17H28NaOSSi requires 331.1528, Δppm = 1.21)

1.1.11. General procedure 2: S_n2 displacement with pyrrolidine

The alkenylchloromethylsilane was dissolved in acetonitrile to give a 0.2 M solution. Pyrrolidine (4 equivalents) was then added, and the resulting solution was heated to reflux and stirred for two hours. The reaction mixture was then cooled to room temperature and partitioned between ethyl acetate and saturated aqueous sodium bicarbonate. The organic layer was separated, and the aqueous layer was extracted twice with ethyl acetate. The combined organic layers were washed with water and then with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure to afford the crude product, which was further purified by flash chromatography.
1.111.1. (E)-1-((dimethyl(styryl)silyl)methyl)pyrrolidine (26)

δ_H (400 MHz; CDCl3): 7.47 (2H, d, 7.2 Hz, 7), 7.35 (2H, t, 7.6 Hz, 8), 7.27 (1H, t, 7.2 Hz, 9), 6.96 (1H, d, 19.2 Hz, 6), 6.52 (1H, d, 19.2 Hz, 5), 2.52 (4H, m, 2), 2.18 (2H, s, 3), 1.78 (4H, m, 1), 0.26 (6H, s, 4)
δ_C (100 MHz; CDCl3): 147.24, 141.22, 131.30, 130.82, 130.77, 129.24, 61.05, 49.84, 26.76, 0.02
υ_max/cm⁻¹: 1587.2, 1561.4
m/z (ESI⁺): Found [M+H]⁺, 246.1685 (C_{15}H_{24}NSi requires 246.1678, Δppm = 2.84)

1.111.2. (Z)-1-((dimethyl(styryl)silyl)methyl)pyrrolidine (27)

According to general procedure 2, (Z)-(chloromethyl)dimethyl(styryl)silane 29 (1.50 g, 7.12 mmol) in acetonitrile (35 mL) was treated with pyrrolidine (2.33 mL, 28.5 mmol). Purification by flash chromatography (9:1 CHCl₃:10 % NH₄OH in MeOH, R_f = 0.39) furnished the title compound as a colourless oil (1.45 g, 83 %).

δ_H (400 MHz; CDCl3): 7.34 (1H, d, 15.2 Hz, 6), 7.22 (5H, m, Ph), 5.80 (1H, d, 15.2 Hz, 5), 2.38 (4H, m, 2), 2.01 (2H, s, 3), 1.66 (4H, m, 1), 0.00 (6H, s, 4)
δ_C (100 MHz; CDCl3): 148.71, 141.42, 132.47, 129.47, 129.27, 128.76, 59.48, 49.11, 25.30, 0.03
υ_max/cm⁻¹: 1591.5, 1570.8
m/z (ESI⁺): Found [M+H]⁺, 246.1688 (C_{15}H_{24}NSi requires 246.1678, Δppm = 4.06)

1.111.3. 1-(((5-(benzyloxy)pent-1-en-2-yl)dimethylsilyl)methyl)pyrrolidine (28)

According to general procedure 2, (5-(benzyloxy)pent-1-en-2-yl)(chloromethyl)dimethylsilane (23) 150 (1.00 g, 3.54 mmol) in acetonitrile (15 mL) was treated with pyrrolidine (2.33 mL, 28.5 mmol). Purification by flash
chromatography (19:1 CHCl₃: 10 % NH₄OH in MeOH, Rᵢ = 0.31) furnished the title compound as a colourless oil (0.97 g, 87 %).

δₜ (400 MHz; CDCl₃): 7.34 - 7.26 (5H, m, Ph), 5.60 (1H, m, 5), 5.39 (1H, m, 6), 4.49 (2H, s, 1), 3.48 (2H, t, 6.8 Hz, 2), 2.43 (4H, m, 9), 2.22 (2H, t, 7.6 Hz, 4), 2.08 (2H, s, 8), 1.79 - 1.69 (6H, m, 10 and 3), 0.15 (6H, s, 7)

δₜ (100 MHz; CDCl₃): 150.8, 139.0, 128.8, 128.0, 127.9, 125.4, 73.3, 70.5, 58.5, 46.7, 32.6, 29.3, 24.3, -2.5

υₘₐₓ/cm⁻¹: 1495.9, 1454.2

m/z (ESI⁺): Found [M+H]⁺, 318.2248 (C₁₉H₃₂NOSi requires 318.2253, Δppm = -1.57)

1.1.12. General procedure 3: Homocoupling and desilylation of alkenylsilanes bearing coordinating functionality

The alkenylsilane was dissolved in acetonitrile. Copper iodide was then added and the reaction was stirred for 10 minutes at room temperature until homogeneous. Caesium fluoride was then added, and the reaction was heated to reflux and stirred for two hours or until TLC analysis indicated complete consumption of the starting material; during this time, a dark metallic precipitate formed. The reaction mixture was then cooled to room temperature and concentrated under reduced pressure. The dark residue was partitioned between diethyl ether (50 mL per mmol of substrate) and ammonium hydroxide (20 mL per mmol of substrate) and stirred at room temperature until the aqueous layer was deep blue and all the solid residue had disappeared. The organic layer was then separated, washed with water and then with brine, dried over MgSO₄, filtered, concentrated under reduced pressure, and purified by flash chromatography or recrystallisation.

1.1.12.1. (1E, 3E)-1,4-diphenylbuta-1,3-diene (17)

![Diagram of (1E, 3E)-1,4-diphenylbuta-1,3-diene](image)

According to general procedure 3, alkenylsilane 26 (400 mg, 1.60 mmol) in acetonitrile (8.0 mL) was treated with copper iodide (309 mg, 1.60 mmol) and caesium fluoride (365 mg, 2.40 mmol) to furnish, after recrystallisation (ethyl acetate), the title compound as a white solid (142 mg, 86 %).

δₜ (400 MHz; CDCl₃): 7.47 (4H, d, 7.2 Hz, 3), 7.36 (4H, app. t, 7.2 Hz, 2), 7.25 (2H, t, 7.2 Hz, 1), 6.98 (2H, m, 4), 6.69 (2H, m, 5)

δₜ (100 MHz; CDCl₃): 137.4, 132.9, 129.3, 128.7, 127.6, 126.4

Spectroscopic data obtained are consistent with literature values.²
1.1.12.2.  (1Z, 3Z)-1,4-diphenylbuta-1,3-diene (24)

According to general procedure 3, alkenylsilane 27 (400 mg, 1.60 mmol) in acetonitrile (8.0 mL) was treated with copper iodide (309 mg) and caesium fluoride (365 mg, 2.40 mmol) to furnish, after ash chromatography (pet. ether, \( R_f = 0.28 \)), the title compound as a white solid (147 mg, 89 %).

\[ \delta_H (400 \text{ MHz}; \text{CDCl}_3): \] 7.39 (4H, d, 7.5 Hz), 7.28 (4H, app. t, 7.5 Hz), 7.17 (2H, app. tt, 7.5 Hz, 2.0 Hz), 6.92 (2H, m), 6.63 (1H, m)

\[ \delta_C (100 \text{ MHz}; \text{CDCl}_3): \] 137.7, 133.1, 129.5, 128.9, 127.8, 126.6

Melting point: 66-67 °C (pet. ether)

Analytical data obtained are consistent with literature values.\(^3\)

1.1.12.3.  (((4,5-dimethyleneoctane-1,8-diyl)bis(oxy))bis(methylene))dibenzene (25)

According to general procedure 1, pyrrolidinyldimethylsilylalkene 28 (400 mg, 1.26 mmol) in acetonitrile (6 mL) was treated with copper iodide (240 mg, 1.26 mmol) and caesium fluoride (289 mg, 1.90 mmol). Purification by flash chromatography (39:1 pet. ether: ethyl acetate, \( R_f = 0.33 \)) furnished the title compound as a colourless oil (203 mg, 92 %).

\[ \delta_H (400 \text{ MHz}; \text{CDCl}_3): \] 7.35 - 7.25 (10H, m, Ph), 5.08 (2H, m, 5), 4.94 (2H, m, 5), 4.50 (4H, s, 1), 3.47 (4H, t, 6.4 Hz, 2), 2.33 (4H, t, 7.2 Hz, 4), 1.76 (4H, tt, 7.2 Hz, 6.4 Hz, 3)

\[ \delta_C (100 \text{ MHz}; \text{CDCl}_3): \] 147.2, 139.0, 128.8, 128.1, 127.9, 112.5, 73.3, 70.3, 31.0, 29.1

\[ \nu_{max}/\text{cm}^{-1}: \] 1594.8, 1495.6

m/z (ESI\(^+\)): Found [M+Na]\(^+\), 373.2146 (C\(_{24}\)H\(_{30}\)NaO\(_2\) requires 373.2143, \( \Delta \text{ppm} = 0.80 \))

References:


Selected NMR spectra