SUPPORTING INFORMATION

Cyclopropanation of Protected Chiral, Acyclic Allylic Alcohols: an Expedient Access to the *anti*-Cyclopropylcarbinol Derivatives

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General experimental procedures. Characterization data for new compounds. GC traces for *syn* and *anti* diastereomers.

General: All non-aqueous reactions were run under an inert atmosphere (argon) with rigid exclusion of moisture from reagents and glassware using standard techniques for manipulating air-sensitive compounds.¹ All glassware was stored in the oven and/or was flame-dried prior to use under an inert atmosphere of gas. Anhydrous solvents were obtained either by filtration through drying columns (THF, ether, CH₂Cl₂, DMF) on a GlassContour system (Irvine, CA). Analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel (Merck 60 F₂₅₄). Visualization of the developed chromatogram was performed by UV absorbance, aqueous cerium molybdate or aqueous potassium permanganate. Flash column chromatography was performed using 230-400 mesh silica (EM Science or Silicycle) of the indicated solvent system according to standard technique.² Infrared spectra were taken on a Perkin Elmer Spectrum One FTIR and are reported in reciprocal centimeters (cm⁻¹). Nuclear magnetic resonance spectra (¹H, ¹³C, DEPT 135) were recorded either on a Bruker AV 300, AMX 300, AV 400 or ARX 400 spectrometer. Chemical shifts for ¹H NMR spectra are recorded in parts per million from tetramethylsilane with the solvent resonance as the internal standard (chloroform, δ 7.27 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, s= septuplet, o = octuplet, m = multiplet and br = broad), coupling constant in Hz and integration. Chemical shifts for ¹³C NMR spectra are recorded in parts per million from tetramethylsilane using the central peak of deuteriochloroform (77.00 ppm) as the internal standard. All spectra were obtained with complete proton decoupling. When ambiguous, proton and carbon assignments were established using DEPT experiments. High resolution mass spectra were performed by the Centre régional de spectroscopie de masse de l'Université de Montréal.

Analytical gas chromatography was carried out on a Hewlett Packard 5880A gas chromatograph equipped with a splitless mode capillary injector and a flame ionization detector. Unless otherwise noted, the injector and detector temperatures were set to 250 °C and hydrogen was used as the carrier gas (25 psi). Data are reported as follows: (column type, column length, initial temperature, initial time, rate, final temperature, final time: retention time (*t*)).

Reagents: Unless otherwise stated, commercial reagents were used without purification. Trifluoroacetic acid was distilled on phosphorus pentoxide and kept under argon. Diethylzinc was purchased neat from AkzoNobel and used without purification.

Preparation of starting allylic ethers: All ethers were prepared from the corresponding racemic allylic alcohols according to the protocols below, except **5**.³ (3*E*)-4-phenylbut-3-en-2-ol,⁴ (1*E*)-1-phenylpent-1-en-3-ol,⁵ (1*E*)-4-methyl-1-phenylpent-1-en-3-ol,⁶ (3*E*)-3-methyl-4-phenylbut-3-en-2-ol,⁷ (1*E*)-2-methyl-1-phenylpent-1-en-3-ol⁶ and (3*Z*)-6-phenylhex-3-en-2-ol³ are all known compounds and were prepared according to a literature procedure. (3*Z*)-4-phenylbut-3-en-2-ol and (3*E*)-6-phenylhex-3-en-2-ol were prepared by the same procedure as (3*Z*)-6-phenylhex-3-en-2-ol³, except that the last intermediate for the former was reduced according to a protocol by Denmark⁸ and the latter was prepared starting from phenylacetylene (Aldrich). The benzyl ether **5** is a known product prepared according to a literature procedure.³

General procedure for the preparation of allylic *tert*-butyldimethylsilyl ethers. Compounds **1**, **6**, and **11** were prepared according to the following general procedure. To a solution of the allylic alcohol (7.4 mmol, 1.0 equiv) in 60 mL of dry DMF at room temperature was added subsequently imidazole (1.0 g, 14.8 mmol, 2.0 equiv) and *tert*-butyldimethylsilyl chloride (1.70 g, 11.1 mmol, 1.5 equiv). The solution was stirred for 3 h and was quenched with saturated aqueous NH₄Cl. The layers were separated and the aqueous layer was washed with ethyl acetate (2 x 30

¹ Shriver, D. F.; Drezdson, M. A. *The manipulation of air-sensitive compounds*; 2nd Edition ed.; Wiley: New York, 1986.

² Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

³ Charette, A. B.; Lebel, H. *J. Org. Chem.* **1995**, *60*, 2966.

⁴ Burgess, K.; Jennings, L. D. *J. Am. Chem. Soc.* **1991**, *113*, 6129.

⁵ Marples, B. A.; Rogers-Evans, M. *Tetrahedron Lett.* **1989**, *30*, 261.

⁶ Atkinson, R. S.; Ulukanli, S.; Williams, P. J. *J. Chem. Soc., Perkin Trans. 1* **1999**, 2121.

⁷ Alder, V. K.; Haydn, J.; Heimbach, K.; Neufang, K. *Justus Liebigs Ann. Chem.* **1954**, *586*, 110.

⁸ Denmark, S. E.; Jones, T. K. *J. Org. Chem.* **1982**, *47*, 4595.

mL). The combined organic layers were then washed with saturated aqueous NaHCO_3 (2 x 30 mL) and saturated aqueous NaCl (2 x 30 mL). The organic solution was dried over MgSO_4 , filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography to give the pure allylic *tert*-butyldimethylsilyl ether.

General procedure for the preparation of allylic triethylsilyl ethers. Compounds **2**, **7**, **8**, **10** and **13** were prepared according to the following general procedure. To a solution of the allylic alcohol (6.7 mmol, 1.0 equiv) in 50 mL of dry DMF at room temperature was added subsequently imidazole (0.92 g, 13.5 mmol, 2.0 equiv) and triethylsilyl chloride (2.30 mL, 13.5 mmol, 2.0 equiv). The solution was stirred for 6 h and was quenched with saturated aqueous NH_4Cl . The layers were separated and the aqueous layer was washed with ethyl acetate (2 x 30 mL). The combined organic layers were then washed with saturated aqueous NaHCO_3 (2 x 25 mL) and saturated aqueous NaCl (2 x 25 mL). The organic solution was dried over MgSO_4 , filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography to give the pure allylic triethylsilyl ether.

General procedure for the preparation of allylic triisopropylsilyl ethers. Compounds **3**, **4**, **9** and **12** were prepared according to the following general procedure. To a solution of the allylic alcohol (11.3 mmol, 1.0 equiv) in 100 mL of dry CH_2Cl_2 at $-10\text{ }^\circ\text{C}$ (acetone/ice bath) was added subsequently 2,6-lutidine (2.20 mL, 19.2 mmol, 1.7 equiv) and triisopropylsilyl triflate (4.60 mL, 17.0 mmol, 1.5 equiv). The solution was stirred for 3.5 h at $-10\text{ }^\circ\text{C}$ and quenched with saturated aqueous NH_4Cl . The solution was diluted with ether (100 mL), the layers were separated and the organic layer was washed with saturated aqueous NaHCO_3 (2 x 50 mL) and saturated aqueous NaCl (2 x 50 mL). The organic solution was dried over MgSO_4 , filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography to give the pure allylic triisopropylsilyl ether.

***tert*-Butyl(dimethyl){[(2*E*)-1-methyl-3-phenylprop-2-enyl]oxy}silane (**1**).** The general procedure for the preparation of allylic *tert*-butyldimethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **1** as a colorless oil (1.24 g, 92% from (3*E*)-4-phenylbut-3-en-2-ol): R_f 0.40 (2.5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.38 (d, J = 7.1 Hz, 2H), 7.31 (t, J = 7.5 Hz, 2H), 7.22 (m, 1H), 6.52 (d, J = 15.9 Hz, 1H), 6.22 (dd, J = 5.6, 15.8 Hz, 1H), 4.48 (qn, J = 6.2 Hz, 1H), 1.31 (d, J = 6.3 Hz, 3H), 0.94 (s, 9H), 0.11 (s, 3H), 0.10 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.1, 134.4, 128.4 (2), 127.8, 127.1, 126.2 (2), 69.3, 25.8 (3), 24.5, 18.2, -4.6, -4.8; IR (neat) 3026 (w), 2954(s), 2928(s), 2861(s), 1470(m), 1370(m), 1367(m), 1253(s), 1145(s), 1078(s), 964(s), 836(vs), 774(s), 692(m); HRMS (MAB) calcd for $\text{C}_{16}\text{H}_{26}\text{OSi}$ $[M]^+$: 262.1753, found 262.1754.

Triethyl{[(2*E*)-1-methyl-3-phenylprop-2-enyl]oxy}silane (2**).** The general procedure for the preparation of allylic triethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **2** as a colorless oil (1.56 g, 88% from (3*E*)-4-phenylbut-3-en-2-ol): R_f 0.38 (2.5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.40 (d, J = 7.6 Hz, 2H), 7.34 (t, J = 7.6 Hz, 2H), 7.25 (t, J = 7.6 Hz, 1H), 6.55 (d, J = 16.0 Hz, 1H), 6.27 (dd, J = 5.9, 16.0 Hz, 1H), 4.51 (qn, J = 5.9 Hz, 1H), 1.36 (d, J = 6.0 Hz, 3H), 1.02 (t, J = 8.0 Hz, 9H), 0.68 (q, J = 8.0 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.0, 134.3, 128.4 (2), 127.9, 127.1, 126.2 (2), 69.0, 24.6, 6.7 (3), 4.8 (3); IR (neat) 3031(w), 2959(vs), 2877(vs), 1496(m), 1460(m), 1367(m),

1146(s), 1084(vs), 1002(vs), 966(vs), 770(s), 749(vs), 692(s); HRMS (MAB) calcd for $C_{16}H_{26}OSi$ $[M]^+$: 262.1753, found 26.1747.

Triisopropyl[(2E)-1-methyl-3-phenylprop-2-enyl]oxy silane (3). The general procedure for the preparation of allylic triisopropylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **3** as a colorless oil (2.65 g, 86% from (3E)-4-phenylbut-3-en-2-ol): R_f 0.50 (2.5% EtOAc/hexanes); 1H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, J = 8.1 Hz, 2H), 7.35 (t, J = 7.5 Hz, 2H), 7.25 (m, 1H), 6.57 (d, J = 16.0 Hz, 1H), 6.28 (dd, J = 5.9, 16.0 Hz, 1H), 4.61 (qn, J = 5.9 Hz, 1H), 1.38 (d, J = 6.0 Hz, 3H), 1.14 (m, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.1, 134.9, 128.4 (2), 127.7, 127.1, 126.2 (2), 69.3, 24.9, 18.0 (6), 12.2 (3); IR (neat) 3026(w), 2944(vs), 2866(vs), 1491(w), 1460(m), 1367(m), 1146(s), 1084(s), 960(s), 883(s), 739(s), 688(s); HRMS (MAB) calcd for $C_{19}H_{32}OSi$ $[M]^+$: 304.2222, found 304.2237.

Triisopropyl[(2E)-1-methyl-5-phenylpent-2-enyl]oxy silane (4). The general procedure for the preparation of allylic triisopropylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **4** as a colorless oil (1.29 g, 98% from (3E)-6-phenylhex-3-en-2-ol): R_f 0.69 (5% EtOAc/hexanes); 1H NMR (400 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.20 (m, 3H), 5.61 (dt, J = 5.8, 15.4 Hz, 1H), 5.53 (dd, J = 5.8, 15.4 Hz, 1H), 4.35 (qn, J = 6.3 Hz, 1H), 2.70 (m, 2H), 2.35 (m, 2H), 1.23 (d, J = 6.3 Hz, 3H), 1.08 (m, 21H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.4, 136.2, 128.8 (2), 128.7 (2), 128.3, 126.2, 69.7, 36.2, 34.3, 25.5, 18.5 (3), 12.7 (6); IR (neat) 3024(w), 2943(vs), 2866(vs), 1496(w), 1464(s), 1365(m), 1088(s), 966(m), 882(m), 745(m), 680(m); HRMS (MAB) calcd for $C_{21}H_{36}OSi$ $[M-NH_4]^+$: 350.2879, found 350.2874.

tert-Butyl(dimethyl)[(2E)-1-ethyl-3-phenylprop-2-enyl]oxy silane (6). The general procedure for the preparation of allylic *tert*-butyldimethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **6** as a colorless oil (1.52 g, 89% from (1E)-1-phenylpent-1-en-3-ol): R_f 0.58 (5% EtOAc/hexanes); 1H NMR (300 MHz, $CDCl_3$) δ 7.37-7.19 (m, 5H), 6.47 (d, J = 15.9 Hz, 1H), 6.15 (dd, J = 6.3, 15.9 Hz, 1H), 4.17 (q, J = 6.2 Hz, 1H), 1.57 (m, 2H), 0.90 (t, J = 7.4 Hz, 3H), 0.90 (s, 9H), 0.06 (s, 3H), 0.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 133.8, 129.4, 129.0 (2), 127.7, 126.8 (2), 75.2, 31.7, 26.4 (3), 18.8, 10.2, -3.8, -4.3; IR (neat) 3027(w), 2957(s), 2929(s), 2857(s), 1494(w), 1472(m), 1463(m), 1361(m), 1255(s), 1068(m), 1005(w), 966(m), 835(s), 744(m), 692(m); HRMS (MAB) calcd for $C_{17}H_{28}OSi$ $[M]^+$: 276.1909, found 276.1904.

Triethyl[[(2E)-1-ethyl-3-phenylprop-2-enyl]oxy]silane (7). The general procedure for the preparation of allylic triethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **7** as a colorless oil (1.59 g, 93% from (1E)-1-phenylpent-1-en-3-ol): R_f 0.49 (5% EtOAc/hexanes); ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.17 (m, 5H), 6.46 (d, J = 15.9 Hz, 1H), 6.14 (dd, J = 6.7, 15.9 Hz, 1H), 4.15 (q, J = 6.7 Hz, 1H), 1.56 (m, 2H), 0.92 (m, 12H), 0.59 (q, J = 7.8 Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.6, 133.7, 129.6, 129.0 (2), 127.7, 126.8 (2), 75.3, 31.8, 10.2, 7.3 (3), 5.4 (3); IR (neat) 3026(w), 2955(s), 2875(s), 1600(w), 1494(w), 1457(m), 1362(w), 1238(m), 1063(s), 1005(vs), 965(s), 743(s), 692(m); HRMS (MAB) calcd for $\text{C}_{17}\text{H}_{28}\text{OSi}$ $[\text{M}]^+$: 276.1909, found 276.1910.

Triethyl[[(2E)-1-isopropyl-3-phenylprop-2-enyl]oxy]silane (8). The general procedure for the preparation of allylic triethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **8** as a colorless oil (1.46 g, 95% from (1E)-4-methyl-1-phenylpent-1-en-3-ol): R_f 0.57 (5% EtOAc/hexanes); ^1H NMR (300 MHz, CDCl_3) δ 7.37-7.18 (m, 5H), 6.45 (d, J = 15.9 Hz, 1H), 6.16 (dd, J = 7.1, 15.9 Hz, 1H), 3.96 (t, J = 6.8 Hz, 1H), 1.74 (o, J = 6.6 Hz, 1H), 0.92 (m, 15H), 0.59 (q, J = 7.8 Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.7, 132.3, 130.5, 129.0 (2), 127.7, 126.8 (2), 79.2, 35.5, 18.8, 7.4 (3), 5.5 (3); IR (neat) 3031(w), 2959(vs), 2908(s), 2877(vs), 1496(w), 1465(m), 1383(m), 1367(m), 1239(m), 1069(vs), 1007(s), 971(s), 806(m), 744(vs), 692(s); HRMS (MAB) calcd for $\text{C}_{18}\text{H}_{30}\text{OSi}$ $[\text{M}]^+$: 290.2066, found 290.2068.

Triisopropyl[[(2E)-1-ethyl-3-phenylprop-2-enyl]oxy]silane (9). The general procedure for the preparation of allylic triisopropylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **9** as a colorless oil (2.40 g, 81% from (1E)-1-phenylpent-1-en-3-ol): R_f 0.64 (5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.38 (d, J = 7.8 Hz, 2H), 7.32 (t, J = 7.8 Hz, 2H), 7.25 (m, 1H), 6.50 (d, J = 15.9 Hz, 1H), 6.18 (dd, J = 6.8, 15.9 Hz, 1H), 4.34 (q, J = 6.3 Hz, 1H), 1.70-1.63 (m, 2H), 1.09 (m, 21H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.7, 133.8, 129.6, 128.9 (2), 127.6, 126.8 (2), 75.2, 31.9, 18.5 (6), 12.8 (3), 9.6; IR (neat) 3026(w), 2942(vs), 2866(vs), 1493(w), 1464(s), 1364(m), 1065(m), 966(m), 882(s), 745(m), 691(s); HRMS (MAB) calcd for $\text{C}_{20}\text{H}_{34}\text{OSi}$ $[\text{M}]^+$: 318.2379, found 318.2387.

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Triethyl[(2E)-1,2-dimethyl-3-phenylprop-2-enyl]oxy silane (10). The general procedure for the preparation of allylic triethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **10** as a colorless oil (2.44 g, 95% from (3E)-3-methyl-4-phenylbut-3-en-2-ol): R_f 0.51 (5% EtOAc/hexanes); ^1H NMR (300 MHz, CDCl_3) δ 7.34-7.16 (m, 5H), 6.46 (s, 1H), 4.31 (q, $J = 6.0$ Hz, 1H), 1.83 (s, 3H), 1.30 (d, $J = 6.0$ Hz, 3H), 0.96 (t, $J = 8.0$ Hz, 9H), 0.61 (q, $J = 8.0$ Hz, 6H); ^{13}C (75 MHz, CDCl_3) δ 142.6, 138.6, 129.4 (2), 128.5 (2), 126.5, 124.0, 74.3, 23.8, 13.8, 7.3 (3), 5.3 (3); IR (neat) 2954(s), 2876(s), 1458(m), 1414(w), 1368(w), 1238(m), 1109(m), 1084(vs), 1053(m), 1004(s), 986(m), 743(m), 697(s), 631(s); HRMS (MAB) calcd for $\text{C}_{17}\text{H}_{28}\text{OSi}$ $[\text{M}]^+$: 276.1919, found 276.1913.

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tert-Butyldimethyl[(2E)-1-ethyl-2-methyl-3-phenylprop-2-enyl]oxy silane (11). The general procedure for the preparation of allylic *tert*-butyldimethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **11** as a colorless oil (2.21 g, 89% from (1E)-2-methyl-1-phenylpent-1-en-3-ol): R_f 0.64 (5% EtOAc/hexanes), ^1H (300 MHz, CDCl_3) δ 7.34-7.16 (m, 5H), 6.40 (s, 1H), 4.02 (t, $J = 6.0$ Hz, 1H), 1.80 (s, 3H), 1.58 (m, 2H), 0.90 (s, 9H), 0.88 (t, $J = 7.0$ Hz, 3H), 0.07 (s, 3H), 0.03 (s, 3H); ^{13}C (75 MHz, CDCl_3) δ 141.3, 138.5, 129.3 (2), 128.5 (2), 126.6, 125.5, 80.5, 29.7, 26.3 (3), 18.7, 13.6, 10.6, -4.1, -4.5; IR (neat) 2957(s), 2929(s), 2857(s), 1472(m), 1463(m), 1361(w), 1251(s), 1096(m), 1064(s), 1014(m), 877(m), 835(s), 775(m), 698(m); HRMS (MAB) calcd for $\text{C}_{18}\text{H}_{30}\text{OSi}$ $[\text{M}]^+$: 290.2066, found 290.2072.

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Triisopropyl[(2Z)-1-methyl-5-phenylpent-2-enyl]oxy silane (12). The general procedure for the preparation of allylic triisopropylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **12** as a colorless oil (1.36 g, 96% from (3Z)-6-phenylhex-3-en-2-ol): R_f 0.74 (5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.20 (m, 5H), 5.49 (m, 1H), 5.32 (m, 1H), 4.68 (qn, $J = 6.8$ Hz, 1H), 2.68 (m, 2H), 2.37 (m, 2H), 1.15 (d, $J = 6.2$ Hz, 3H), 1.08 (m, 21H); ^{13}C NMR (75 MHz, CDCl_3) δ 142.2, 136.8, 128.9 (2), 128.8 (2), 126.7, 126.3, 65.5, 36.3, 30.2, 25.4, 18.5 (3), 12.7 (6); IR (neat) 3024(w), 2943(vs), 2866(vs), 1494(w), 1464(s), 1366(m), 1126 (s), 1090(s), 882(m), 882(m), 726(m), 683(m); HRMS (MAB) calcd for $\text{C}_{21}\text{H}_{36}\text{OSi}$ $[\text{M}-\text{NH}_4]^+$: 350.2879, found 350.2884.

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Triethyl[[(2Z)-1-methyl-3-phenylprop-2-enyl]oxy]silane (13). The general procedure for the preparation of allylic triethylsilyl ethers was followed. The crude product was purified on silica gel (5% EtOAc/hexanes) to afford the desired allylic ether **13** as a colorless oil (1.13 g, 85% from (3Z)-4-phenylbut-3-en-2-ol): R_f 0.60 (5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.35 (m, 2H), 7.26 (m, 1H), 7.21 (m, 2H), 6.40 (d, $J = 11.7$ Hz, 1H), 5.72 (dd, $J = 8.9, 11.7$ Hz, 1H), 4.79 (m, 1H), 1.38 (d, $J = 6.2$ Hz, 3H), 0.87 (t, $J = 7.9$ Hz, 9H), 0.48 (q, $J = 7.9$ Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.8, 137.5, 129.0 (2), 128.6 (2), 127.8, 127.3, 65.1, 24.9, 7.1 (3), 5.3 (3); IR (neat) 3014(w), 2954(s), 2876(s), 1493(m), 1458(m), 1368(m), 1238(m), 1116(s), 1080(s), 1066(s), 1003(s), 744(m), 699(m); HRMS (MAB) calcd for $\text{C}_{16}\text{H}_{26}\text{OSi}$ $[M]^+$: 262.1753, found 262.1754.

General procedure for the synthesis of cyclopropylcarbinyl ethers. Compounds **14**, **15**, **16**, **17**, **20**, **26**, **27** and **28** were prepared according to the following general procedure.⁹ To a vigorously stirred solution of diethylzinc (0.20 mL, 2.0 mmol, 2.0 equiv) in 4.0 mL of dry CH_2Cl_2 at 0 °C (ice bath) was added over 10 min. a solution of trifluoroacetic acid (0.15 mL, 2.0 mmol, 2.0 equiv) in 1.0 mL of CH_2Cl_2 . The resulting slurry was stirred for 20 min. after which a solution of diiodomethane (0.16 mL, 2.0 mmol, 2.0 equiv) in 1.0 mL of CH_2Cl_2 was added over 6-8 min. After 20 min. of stirring, a white precipitate was usually observed and a solution of the allylic ether (1.0 mmol, 1.0 equiv) in 1.0 mL of CH_2Cl_2 was added over 10 min. The mixture was stirred at 0 °C until the reaction was completed and was quenched with saturated aqueous NaHCO_3 . The combined layers were diluted with ether (15 mL) and washed with saturated aqueous NH_4Cl (1 x 10 mL), saturated aqueous Na_2SO_3 (2 x 5 mL), saturated aqueous NaHCO_3 (2 x 5 mL) and saturated aqueous NaCl (2 x 5 mL). The organic solution was dried over MgSO_4 , filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography to give the pure cyclopropylcarbinyl ether. The diastereomeric ratios of the cyclopropylcarbinyl silyl ethers were determined by capillary GC analysis of the corresponding acetate derivatives that were prepared by deprotection of the silyl ether (TBAF 1.0 M in THF (1.5 equiv), 50 °C, 12h) and subsequent acetylation of the resulting alcohol (pyridine : acetic anhydride 2 : 1, 25 °C, 30 min.) using the corresponding *syn* (or mixture of *syn* and *anti*) cyclopropylcarbinyl acetates as standards.³ The diastereomeric ratio of product **18** was directly determined by capillary GC analysis and was compared to a sample of the known benzyl cyclopropylcarbinyl ether.¹⁰

14

tert-Butyl(dimethyl)((1S*)-1-[(1R*,2R*)-2-phenylcyclopropyl]ethyl)oxy)silane (14). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **14** (238 mg, 86%) as a colorless oil: R_f 0.41 (2.5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.33-7.28 (m, 2H), 7.22-7.14 (m, 3H), 3.69 (qn, $J = 6.0$ Hz, 1H), 1.98 (m, 1H), 1.33 (d, $J = 6.1$ Hz, 3H), 1.28 (m, 1H), 0.97 (s, 9H), 0.91 (m, 1H), 0.14 (s, 3H), 0.13 (s, 3H); ^{13}C (100 MHz, CDCl_3) δ 143.4, 128.0 (2), 125.9 (2), 125.1, 70.0, 30.6, 25.8 (3), 23.8, 20.1, 18.0, 12.4, -4.5, -4.7; IR (neat) 2957(s), 2930(s), 2858(s), 1606(w), 1499(w), 1463(m), 1370(m), 1255(s), 1116(m), 835(vs), 775(s), 697(m), 559(vs); HRMS (MAB) calcd for $\text{C}_{17}\text{H}_{28}\text{OSi}$ $[M]^+$: 276.1909, found 276.1913; GC (DB-1701, 30 m, 130 °C) t_r (major) 3.5 min., t_r (minor) 3.8 min.

⁹ Yang, Z.; Lorenz, J. C.; Shi, Y. *Tetrahedron Lett.* **1998**, 39, 8621.

¹⁰ Charette, A. B.; Lebel, H.; Gagnon, A. *Tetrahedron* **1999**, 55, 8845.

Compound 14

Standard

15

Triethyl((1S*)-1-[(1R*,2R*)-2-phenylcyclopropyl]ethyl)oxy)silane (15). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **15** (240 mg, 87%) as a colorless oil: R_f 0.32 (2.5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.28-7.24 (m, 2H), 7.16-7.08 (m, 3H), 3.60 (q, J = 6.0 Hz, 1H), 1.90 (dt, J = 5.0, 8.7 Hz, 1H), 1.26 (m, 1H), 1.28 (d, J = 6.1 Hz, 3H), 0.97 (t, J = 7.9 Hz, 9H), 0.95-0.84 (m, 2H), 0.61 (q, J = 7.9, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 143.9, 128.6 (2), 126.3 (2), 125.6, 70.8, 31.2, 24.3, 21.1, 13.3, 7.3 (3), 5.4 (3); IR (neat) 2955(vs), 2877(vs), 1498(w), 1459(m), 1368(m), 1239(m), 1115(m), 1008(s), 746(s), 697(m), 574(s); HRMS (MAB) calcd for $\text{C}_{18}\text{H}_{28}\text{OSi}$ $[\text{M}]^+$: 276.1909, found 276.1919; GC (DB-1701, 30 m, 130 °C) t_r (major) 3.5 min., t_r (minor) 3.8 min.

Compound 15

Standard

16

Triisopropyl((1*S*^{*})-1-[(1*R*^{*},2*R*^{*})-2-phenylcyclopropyl]ethyl)oxy)silane (16**).** The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **16** (280 mg, 88%) as a colorless oil: *R*_f 0.49 (5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.28-7.24 (m, 2H), 7.14 (m, 1H), 7.09 (m, 2H), 3.83 (qn, *J* = 5.9 Hz, 1H), 1.94 (m, 1H), 1.31 (d, *J* = 6.1 Hz, 3H), 1.27 (m, 1H), 1.15-0.92 (m, 21H), 0.85 (m, 2H); ¹³C (100 MHz, CDCl₃) δ 143.5, 128.0 (2), 125.8 (2), 125.0, 69.7, 31.0, 23.9, 20.1, 18.1 (6), 12.6, 12.4 (3); IR (neat) 2962(s), 2944(vs), 2867(vs), 1498(w), 1464(m), 1370(w), 1118(m), 1013(w), 882(m), 751(m), 696(m), 678(w), 556(vs); HRMS (MAB) calcd for C₂₀H₃₄OSi: 318.2379, found 318.2382; GC (DB-1701, 30 m, 130 °C) *t*_r (major) 3.5 min., *t*_r (minor) 3.8 min.

Compound **16**

Standard

Triisopropyl((1S*)-1-[(1R*,2R*)-2-(2-phenylethyl)cyclopropyl]ethyl)oxy)silane (17). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **17** (291 mg, 84%) as a colorless oil: R_f 0.70 (5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.27 (m, 2H), 7.18 (m, 3H), 3.54 (dq, $J = 6.1, 6.1$ Hz, 1H), 2.70 (dd, $J = 7.6, 7.9$ Hz, 2H), 1.64 (m, 1H), 1.45 (m, 1H), 1.21 (d, $J = 6.1$ Hz, 3H), 1.05 (m, 21H), 0.77 (m, 1H), 0.70 (m, 1H), 0.40 (m, 1H), 0.21 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 142.5, 128.3 (2), 128.1(2), 125.5, 36.0, 35.9, 27.0, 23.8, 18.1 (7), 16.4, 12.5 (3), 8.8; IR (neat) 2943(s), 2866(s), 1464(m), 1369(w), 1150(w), 1115(m), 1014(m), 883(s), 745(s), 698(m), 632(s); HRMS (MAB) calcd for $\text{C}_{22}\text{H}_{38}\text{OSi}$ [M^+]: 346.2692, found 346.2688; GC (DB-1701, 30 m, 130 $^\circ\text{C}$) t_r (major) 6.6 min., t_r (minor) 7.3 min.

Compound 17

Standard

Benzyl(1S*)-1-[(1R*,2R*)-2-phenylcyclopropyl]ethyl ether (18). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C , thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (10% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl benzyl ether **18** (202 mg, 80%) as a colorless oil: R_f 0.47 (15% EtOAc/hexanes). Spectral data was identical to the reported literature data.¹⁰ ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.25 (m, 7H), 7.17 (t, $J = 7.3$ Hz, 1H), 7.09 (d, $J = 7.0$ Hz, 2H), 4.69 (dd, $J = 12.0, 36.0$ Hz, 2H), 3.15 (qn, $J = 6.4$ Hz, 1H), 1.90 (q, $J = 4.5$ Hz, 1H), 1.34 (d, $J = 6.2$ Hz, 3H), 1.25 (m, 1H), 0.92 (m, 1H), 0.81 (m, 1H); GC (DB-1701, 30 m, 170 $^\circ\text{C}$) t_r (major) 5.9 min., t_r (minor) 6.6 min.

Compound 18

Standard

19

***tert*-Butyl(dimethyl)((*1S*^{*})-1-[(*1R*^{*},*2R*^{*})-2-phenylcyclopropyl]propyl)oxy)silane (19).** The general procedure for the synthesis of cyclopropylcarbiny ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbiny silyl ether **19** (247 mg, 85%) as a colorless oil: *R*_f 0.60 (5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.25 (m, 2H), 7.16-7.06 (m, 3H), 3.38 (q, *J* = 5.8 Hz, 1H), 1.87 (dt, *J* = 4.8, 8.6 Hz, 1H), 1.62 (m, 2H), 1.23 (m, 1H), 0.95 (t, *J* = 7.4 Hz, 3H), 0.89 (s, 9H), 0.88 (m, 2H), 0.06 (s, 3H), 0.04 (s, 3H); ¹³C (75 MHz, CDCl₃) δ 143.9; 128.6 (2), 126.3 (2), 125.6, 75.6, 31.3, 29.1, 26.3 (3), 20.8, 18.6, 13.3, 10.0, -3.8, -4.0; IR (neat) 2958(s), 2929(s), 2857(s), 1605(w), 1498(w), 1463(m), 1361(w), 1255(m), 1054(w), 1015(w), 835(s), 774(m), 696(m), 518(m); HRMS (MAB) calcd for C₁₈H₃₀OSi [M]⁺: 290.2066, found 290.2063; GC (DB-1701, 30 m, 160 °C) *t*_r (major) 2.1 min., *t*_r (minor) 2.3 min.

Compound 19

Standard

20

Triethyl((1*S*^{*})-1-[(1*R*^{*},2*R*^{*})-2-phenylcyclopropyl]propyl)oxy)silane (20**).** The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C, thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **20** (253 mg, 87%) as a colorless oil: *R*_f 0.61 (5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.26-7.21 (m, 2H), 7.14 (m, 1H), 7.07 (m, 2H), 3.34 (q, *J* = 6.1 Hz, 1H), 1.86 (dt, *J* = 4.9, 8.8 Hz, 1H), 1.25 (m, 1H), 0.96 (t, *J* = 4.9, 3H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.88 (m, 2H), 0.59 (q, *J* = 8.0 Hz, 6H); ¹³C (75 MHz, CDCl₃) δ 143.9, 128.6 (2), 126.2 (2), 125.6, 76.0, 31.4, 29.2, 21.0, 13.7, 10.2, 7.4 (3), 5.6 (3); IR (neat) 2657(s), 2876(s), 1605(w), 1498(w), 1460(m), 1412(w), 1238(w), 1115(m), 1055(w), 1011(m), 840(w), 745(m), 697(w); HRMS (MAB) calcd for C₁₈H₃₀OSi [M⁺]: 290.2066, found 290.2067; GC (DB-1701, 30 m, 160 °C) *t*_r (major) 2.1 min., *t*_r (minor) 2.3 min.

Compound **20**

Standard

Triethyl((1*S*^{*})-2-methyl-1-[(1*R*^{*},2*R*^{*})-2-phenylcyclopropyl]propyl)oxy)silane (21, major) and triethyl((1*S*^{*})-2-methyl-1-[(1*S*^{*},2*S*^{*})-2-phenylcyclopropyl]propyl)oxy)silane (22, minor). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C, thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ethers **21** and **22** (238 mg, 78% combined yield) as a colorless oil: *R*_f 0.60 (5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.22-7.27 (m, 2H, **21** and **22**), 7.13 (m, 1H, **21** and **22**), 7.05 (m, 2H, **21** and **22**), 3.23 (dd, *J* = 4.0, 6.6 Hz, 1H, **21**), 3.11 (dd, *J* = 4.4, 7.1 Hz, 1H, **22**), 1.84 (m, 2H, **21**), 1.74 (m, 2H, **22**), 1.23 (m, 1H, **21** and **22**), 0.99 (t, *J* = 7.9 Hz, 9H, **22**), 0.99-0.82 (m, 8H, **21** and **22**), 0.93 (t, *J* = 7.9 Hz, 9H, **21**), 0.63 (q, *J* = 8.2 Hz, 6H, **22**), 0.58 (q, *J* = 8.1 Hz, 6H, **21**); ¹³C NMR (75 MHz, CDCl₃) δ 143.9, 143.5, 128.7, 128.5, 126.1, 125.8, 125.6, 80.5, 79.5, 35.7, 35.5, 27.2, 26.7, 21.5, 18.6, 18.5, 14.7, 13.9, 7.4, 5.8, 5.7, 5.4; IR (neat) 2957(vs), 2876(s), 1605(w), 1498(w), 1460(m), 1365(w), 1238(m), 1058(m), 1009(m), 825(w), 743(s), 696(m), 562(s); HRMS (MAB) calcd for C₁₉H₃₂OSi [M⁺]: 304.2222, found 304.2220; GC (DB-1701, 30 m, 130 °C) *t*_r (major) 5.7 min., *t*_r (minor) 6.3 min.

Compounds **21** and **22**

Standard

Triisopropyl((1*S*^{*})-1-[(1*R*^{*},2*R*^{*})-2-phenylcyclopropyl]propyl)oxy)silane (23). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C, thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **23** (293 mg, 88%) as a colorless oil: *R*_f 0.65 (5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.26 (m, 2H), 7.14 (m, 1H), 7.07 (d, *J* = 7.2 Hz, 2H), 3.62 (q, *J* = 5.5 Hz, 1H), 1.94 (m, 1H), 1.68 (m, 2H), 1.29 (m, 1H), 1.08 (m, 24H), 0.98 (t, *J* = 7.4 Hz, 3H), 0.87 (m, 2H); ¹³C (75 MHz, CDCl₃) δ 144.0, 128.6 (2), 126.1 (2), 125.6, 75.3, 31.5, 29.0, 20.7, 18.7 (6), 13.6, 13.2 (3), 9.7; IR (neat) 2960(s), 2943(vs), 2867(vs), 1605(w), 1498(w), 1463(m), 1378(w), 1117(m), 1062(m), 1011(w), 883(m), 827(w), 750(w), 696(m), 566(s); HRMS (MAB) calcd for C₂₁H₃₆OSi [M⁺]: 332.2535, found 332.2535; GC (DB-1701, 30 m, 130 °C) *t*_r (major) 4.6 min., *t*_r (minor) 5.2 min.

Compound **23**

Standard

Triethyl((1*S*^{*})-1-[(1*R*^{*},2*S*^{*})-1-methyl-2-phenylcyclopropyl]ethyl)oxy)silane (24). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C, thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (2%

EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ether **24** (256 mg, 88%) as a colorless oil: R_f 0.52 (5% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.15 (m, 5H), 3.50 (q, J = 6.1 Hz, 1H), 2.08 (dd, J = 6.1, 8.6 Hz, 1H), 1.24 (d, J = 6.1 Hz, 3H), 0.99 (t, J = 7.7 Hz, 9H), 0.96 (m, 1H), 0.74 (s, 3H), 0.71 (m, 1H), 0.63 (q, J = 7.6 Hz, 6H); ^{13}C (75 MHz, CDCl_3) δ 140.4, 129.6 (2), 128.2 (2), 125.9, 73.8, 29.0, 26.8, 21.6, 15.3, 14.8, 7.4 (3), 5.5 (3); IR (neat) 2956(vs), 2912(m), 2876(s), 1605(w), 1498(w), 1457(m), 1370(w), 1239(m), 1120(m), 1100(m), 1080(m), 1011(m), 967(w), 786(m), 7439(m), 699(m), 562(m), 554(s); HRMS (MAB) calcd for $\text{C}_{18}\text{H}_{30}\text{OSi}$ [M^+]: 290.2066, found 290.2065; GC (DB-1701, 30 m, 160 °C) t_r (major) 1.8 min. t_r (minor) 1.9 min.

Compound 24

Standard

25

(1S*)-1-[(1R*,2S*)-1-methyl-2-phenylcyclopropyl]propan-1-ol (25). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed, except that the reaction was performed at -20°C, thus 40 min. (instead of 20 min.) of stirring were required before adding the substrate to the carbenoid. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the corresponding cyclopropylcarbinyl silyl ether (268 mg, 88%) which was subsequently deprotected (TBAF 1.0 M in THF, 1.5 equiv. 50 °C, 12h), yielding the desired cyclopropyl alcohol **25**, purified on silica gel (25% EtOAc/hexanes), as a colorless oil: R_f 0.35 (25% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.27 (m, 2H), 7.20 (m, 3H), 2.99 (dd, J = 5.1, 8.2 Hz, 1H), 2.04 (dd, J = 6.1, 8.7 Hz, 1H), 1.66 (m, 2H), 1.48 (s (br), 1H), 1.02 (t, J = 7.4 Hz, 3H), 0.94 (dd, J = 5.1, 8.8 Hz, 1H), 0.87 (dd, J = 5.2, 6.0 Hz, 1H), 0.76 (s, 3H); ^{13}C (100 MHz, CDCl_3) δ 138.8, 129.0 (2), 127.9 (2), 125.7, 80.9, 27.3, 27.0, 15.4, 12.4, 10.8; IR (neat) 3393(s, br), 3055(w), 2963(s), 2929(s), 2876(m), 1604(m), 1498(s), 1451(s), 1383(m), 1103(m), 968(m), 779(m), 732(m), 700(s); HRMS (MAB) calcd for $\text{C}_{13}\text{H}_{18}\text{O}$ [M^+]: 190.1358, found 190.1358; GC (DB-1701, 30 m, 130 °C) t_r (major) 5.0 min. t_r (minor) 5.5 min.

Compound 25

Standard

(1S*)-1-[(1R*,2S*)-2-(2-phenethyl)cyclopropyl]ethanol (26). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2% EtOAc/hexanes) to afford the corresponding cyclopropylcarbinyl silyl ether (180 mg, 52%) which was subsequently deprotected (TBAF 1.0 M in THF, 1.5 equiv. 50°C, 12h), yielding the cyclopropyl alcohol **26**, purified on silica gel (25% EtOAc/hexanes), as a light yellow oil: R_f 0.28 (25% EtOAc/hexanes); 1H NMR (400 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.20 (m, 3H), 3.36 (dq, $J = 6.2$, 9.0 Hz, 1H), 2.78 (m, 2H), 1.88 (m, 1H), 1.61 (m, 1H), 1.30 (d, $J = 6.2$ Hz, 3H), 1.28 (s, 1H), 0.89 (m, 2H), 0.72 (m, 1H), -0.11 (q, $J = 5.2$ Hz, 1H); ^{13}C (100 MHz, $CDCl_3$) δ 142.8, 128.9 (2), 128.8 (2), 126.3, 69.4, 36.8, 31.1, 24.9, 23.8, 16.6, 9.6; IR (neat) 3367(s, br), 3055(m), 3024(m), 2992(s), 2967(s), 2930(s), 1602(w), 1494(m), 1452(s), 1367(m), 1162(m), 1074(m), 964(m), 795(m); HRMS (MAB) calcd for $C_{13}H_{18}O$ [M^+]: 190.1358, found 190.1361; GC (DB-1701, 30 m, 130°C) t_r (minor) 1.9 min. t_r (major) 3.0 min.

Compound 26

Standard

^1H NMR compound **26** ^1H NMR standard

Triethyl((1*S*^{*})-1-[(1*R*^{*},2*S*^{*})-2-phenylcyclopropyl]ethyl)oxy)silane (27, minor) and triethyl((1*S*^{*})-1-[(1*S*^{*},2*R*^{*})-2-phenylcyclopropyl]ethyl)oxy)silane (28, major). The general procedure for the synthesis of cyclopropylcarbinyl ethers was followed. The crude product was purified on silica gel (2/2/96 Et₃N/EtOAc/hexanes) to afford the desired cyclopropylcarbinyl silyl ethers **27** and **28** (246 mg, 89% combined yield) as a light yellow oil: *R*_f 0.60 (2/2/96 Et₃N/EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.27 (m, 2H, **27** and **28**), 7.18 (m, 3H, **27** and **28**), 3.17 (dq, *J* = 6.4, 8.8 Hz, 1H, **27**), 2.96 (dq, *J* = 6.0, 9.5 Hz, 1H, **28**), 2.21 (m, 1H, **27** and **28**), 1.33 (m, 1H, **27**), 1.26 (d, *J* = 3H, **27**), 1.23 (m, 1H, **28**), 1.07 (m, 2H, **27** and **28**), 0.99 (d, *J* = 6.2 Hz, 3H, **28**), 0.95 (t, *J* = 7.9 Hz, 9H, **28**), 0.78 (t, *J* = 7.9 Hz, 9H, **27**), 0.56 (q, *J* = 8.0 Hz, 6H, **28**), 0.25 (m, 6H, **27**); ¹³C (75 MHz, CDCl₃) δ 138.6 (**27**), 138.4 (**28**), 128.8 (2, **27**), 128.3 (2, **28**), 127.9 (2, **28**), 127.7 (2, **27**), 125.6 (**27** and **28**), 69.5 (**28**), 67.7 (**27**), 28.2 (**27**), 27.4 (**28**), 24.1 (**27**), 23.6 (**28**), 21.6 (**27**), 20.9 (**28**), 8.8 (**28**), 7.1 (**27**), 6.7 (3, **27** and **28**), 4.9 (3, **28**), 4.7 (3, **27**); IR (neat) 3004(w), 2954(s), 2911(m), 2875(s), 1604(w), 1498(m), 1458(s), 1370(m), 1237(m), 1111(s), 1092(s), 1004(vs), 970(m), 763(m), 741(s), 726(vs), 698(vs), 669(m); HRMS (MAB) calcd for C₁₇H₂₈OSi [M⁺]: 276.1909, found 276.1906; GC (DB-1701, 30 m, 130 °C) *t*_r (minor) 2.2 min. *t*_r (major) 3.0 min.

Compounds **27** and **28**

Standard