Supporting Information

An Anionic Strategy for Three and Four Component Coupling Reactions Leading to Ketone Frameworks Based on Vinylogous Conversion of Dilithio Ketone α,β -Dianions

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General Methods. All nonaqueous operations were carried out in flame-dried glassware and under a dry nitrogen atmosphere. Tetrahydrofuran was distilled from sodium benzophenone ketyl. Phenyl vinyl sulfide, 1,1-diphenylethene and other reagents were distilled prior to use. β -Dichlorobutylstannyl ketones **1a-1e** were prepared from siloxycyclopropanes and butyltin trichloride according to the procedure reported previously.¹ ¹H NMR was recorded on a JEOL JNM-EX270 (270 MHz) spectrometer. Chemical shifts are reported in parts per million δ downfield from internal TMS. ¹³C NMR spectra were recorded on a JEOL JNM-EX270 (68 MHz) spectrometer. IR spectra were obtained from a Hitachi 270-30 Infrared Spectrophotometer. Mass spectra (EIMS) were recorded on a Shimadzu GCMS-QP 5000 Mass Spectrometer. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-DX303 HF spectrometer using electronical ionization (EI) mass spectrometry. The products were isolated by flash silica gel chromatography (Fuji Silysia, BW-300) and, if necessary, were further purified by recycling preparative HPLC (JAI, JAIGER-1H) equipped with a GPC column. Elemental analyses were performed at the Analytical Center, Faculty of Engineering, Osaka University.

(Z)-2,2-Dimethyl-3-(trimethylsiloxy)-7-(trimethylsilyl)-7-(triphenylsilyl)-3-heptene (**4**)

To a solution of β -dichlorobutylstannyl ketone **1a** (360 mg, 1.0 mmol) in THF (10 mL) was added *n*-BuLi (2.59 mL, 1.54 M solution in hexane, 4.0 mmol) dropwise over a period of 5 min at -78 °C. This solution was allowed to warm to 0 °C and stirred for 30 min. To the resulting

solution of α,β -dianion **2a** was added a solution of triphenylvinylsilane (315 mg, 1.1 mmol) in THF (3 mL) transferred via cannula at 0 °C, followed by stirring for 1 h at the same temperature. After cooling to -78 °C, the resulting brown solution was quenched with chlorotrimethylsilane (279 μ L, 2.2 mmol). The mixture was stirred for 30 min at 0 °C and poured into saturated aqueous NH_4Cl (40 mL). The aqueous layer was separated and extracted with pentane (40 mL x 3). The combined organic solution was dried over MgSO_4 and concentrated under reduced pressure. Purification by flash chromatography (elution with hexane/ Et_2O , 100:1) provided the desired δ -silyl enol silyl ether **4** (382 mg, 70%) as a white solid. R_f = 0.2 (hexane/ Et_2O , 100:1); m.p. 56-58 °C; ^1H NMR (270 MHz, CDCl_3) δ -0.14 (s, 9H), 0.17 (s, 9H), 1.01 (s, 9H), 0.87 (dd, 1H, J = 2.6, 6.3 Hz), 1.60-1.90 (m, 2H), 1.95-2.10 (m, 2H), 4.30 (t, 1H, J = 6.3 Hz), 7.34-7.62 (m, 15H); ^{13}C NMR (68 MHz, CDCl_3) δ 1.10, 1.13, 10.23, 27.12, 28.59, 29.65, 36.19, 103.68, 127.65, 129.04, 135.86, 136.15, 158.20; EIMS m/z (relative intensity) 529 ($\text{M}^+ - \text{CH}_3$, 0.3), 259 (60), 73 (100); IR (KBr, cm^{-1}) 1654, 1250; HRMS calcd for $\text{C}_{32}\text{H}_{45}\text{OSi}_3$ ($\text{M}^+ - \text{CH}_3$): 529.2778. Found: 529.2803; Anal. Calcd for $\text{C}_{33}\text{H}_{48}\text{OSi}_3$: C, 72.73; H, 8.88. Found: C, 72.70; H, 8.64.

(Z)-6-Methyl-5-(trimethylsiloxy)-1-(trimethylsilyl)-1-(phenylthio)-4-octene (5)

Purification by flash chromatography (elution with hexane/ Et_2O , 100:1) provided a disilylated product **5** (241 mg, 61%) as a colorless oil. R_f = 0.2 (hexane/ Et_2O , 100:1); ^1H NMR (270 MHz, CDCl_3) δ 0.09 (s, 9H), 0.11 (s, 9H), 0.81 (t, 3H, J = 7.6 Hz), 0.93 (d, 2H, J = 6.9 Hz), 1.18-1.73 (m, 4H), 1.84-1.87 (m, 1H), 2.10-2.20 (m, 2H), 2.43 (dd, 1H, J = 6.0, 2.0 Hz), 4.27 (t, 1H, J = 6.9 Hz), 7.05-7.35 (m, 5H); ^{13}C NMR (68 MHz, CDCl_3) δ -2.15, 0.76, 11.61, 18.04, 24.60, 27.12, 32.20, 34.22, 41.38, 105.97, 125.45, 128.64, 129.24, 138.65, 154.61; EIMS m/z (relative intensity) 394 (M^+ , 2), 209 (39), 73 (100); IR (neat, cm^{-1}) 1656, 1248; HRMS calcd for $\text{C}_{21}\text{H}_{38}\text{OSSi}_2$: 394.2182. Found: 394.2172; Anal. Calcd for $\text{C}_{21}\text{H}_{38}\text{OSSi}_2$: C, 63.89; H, 9.70. Found: C, 63.86; H, 9.69.

(Z)-1-Phenyl-1-(trimethylsiloxy)-5-(trimethylsilyl)-5-(phenylthio)-1-pentene (6)

Purification by flash chromatography (elution with hexane/ Et_2O , 20:1) provided a disilylated product **6** (244 mg, 59%). R_f = 0.35 (hexane/ Et_2O , 50:1); ^1H NMR (270 MHz, CDCl_3) δ 0.09 (s, 9H), 0.18 (s, 9H), 1.8-2.0 (m, 2H), 2.3-2.5 (m, 2H), 2.55 (dd, 1H, J = 4.9 Hz, 3.0 Hz), 5.05 (t, 1H, J = 7.1 Hz), 7.15-7.45 (m, 10H); ^{13}C NMR (68 MHz, CDCl_3) δ -2.66, 0.04, 24.78, 31.41, 33.80, 110.31, 124.80, 125.08, 126.86, 127.45, 128.19, 128.86, 137.97, 138.58, 148.71; EIMS m/z (relative intensity) 414 (M^+ , 2), 209 (11), 73 (100); IR (neat, cm^{-1}) 1636, 1250; HRMS calcd for

C₂₃H₃₄OSi₂S: 414.1869. Found 414.1866.

1-(Phenylsulfonyl)-5-undecanone (7)

Purification by flash chromatography (elution with hexane/Et₂O, 10:1) provided a ketone **7** (180 mg, 65%). R_f = 0.1 (hexane/Et₂O, 20:1); white solid; mp. 32-34 °C; ¹H NMR (270 MHz, CDCl₃) δ 0.88 (t, 3H, J = 6.7 Hz), 1.10-1.30 (m, 6H), 1.54-1.80 (m, 6H), 2.34-2.43 (m, 4H), 2.91 (t, 2H, J = 6.9 Hz), 7.16-7.34 (m, 5H); ¹³C NMR (68 MHz, CDCl₃) δ 13.98, 22.45, 22.84, 23.76, 28.64, 28.86, 31.56, 33.33, 42.03, 42.79, 125.77, 128.81, 129.00, 136.57, 210.87; EIMS m/z (relative intensity) 278 (M⁺, 16), 169 (80), 55 (100); IR (KBr, cm⁻¹) 1698; HRMS calcd for C₁₇H₂₆OS 278.1704. Found 278.1696. Anal. Calcd for C₁₇H₂₆OS: C, 73.33; H, 9.41. Found: C, 73.25; H, 9.40.

2,2-Dimethyl-7,7-diphenyl-3-heptanone (8)

The residue was purified by flash column chromatography on silica gel (elution with hexane/Et₂O, 20:1) to furnish **8** (180 mg) in 61% yield. R_f = 0.1 (hexane/Et₂O, 10:1); ¹H NMR (270 MHz, CDCl₃) δ 1.03 (s, 9H), 1.50-1.56 (m, 2H), 1.97-2.06 (m, 2H), 2.48 (t, 2H, J = 6.3 Hz), 3.90 (t, 1H, J = 7.7 Hz), 7.12-7.29 (m, 10H); ¹³C-NMR (68 MHz, CDCl₃) δ 22.42, 26.39, 35.22, 36.25, 44.01, 51.45, 126.07, 127.80, 128.39, 144.89, 215.65; EIMS m/z (relative intensity) 294 (M⁺, 4), 167 (100); IR (neat, cm⁻¹) 1704; HRMS calcd for C₂₁H₂₆O 294.1984. Found 294.1971.

2,2-Dimethyl-6-hydroxymethyl-7-phenyl-3-heptanone (9)

Purification by flash chromatography (elution with hexane/Et₂O, 1:1) provided a desired product **9** (173 mg, 70%) as a colorless oil. R_f = 0.1 (hexane/Et₂O, 3:1); ¹H NMR (270 MHz, CDCl₃) δ 1.11 (s, 9H), 1.59-1.72 (m, 3H), 2.49-2.68 (m, 4H), 3.45 (m, 2H), 7.16-7.27 (m, 5H); ¹³C NMR (68 MHz, CDCl₃) δ 24.34, 26.58, 34.12, 38.08, 42.54, 44.18, 64.08, 125.82, 128.20, 128.97, 140.35, 216.62; EIMS m/z (relative intensity) 248 (M⁺, 1), 230 (61), 130 (100), 91 (66), 57 (45); IR (neat, cm⁻¹) 3440, 1701; HRMS (EI) calcd for C₁₆H₂₄O₂: 248.1776. Found. 248.1795.

2,2-Dimethyl-7-phenyl-6-(trimethylsilyl)-3-heptanone (10) and 2,2-dimethyl-7-(trimethylsilyl)-6-phenyl-3-heptanone (10')

Purification by flash chromatography (elution with hexane) provided a ketone **10** and **10'** (130 mg, 90%). R_f = 0.15 (hexane/Et₂O, 100:1); ¹H NMR (270 MHz, CDCl₃) major isomer **10**: δ 0.00 (s, 9H), 0.92-0.96 (m, 1H), 1.00 (s, 9H), 1.45-1.51 (m, 1H), 1.71-1.77 (m, 1H), 2.25-2.31 (m,

2H), 2.44 (dd, 1H, $J = 9.7, 13.7$ Hz), 2.80 (dd, 1H, $J = 5.1, 13.7$ Hz), 7.12-7.28 (m, 5H); minor isomer **10'**: not determined; ^{13}C NMR (68 MHz, CDCl_3) major isomer **10**: δ -2.41, 24.17, 26.40, 27.52, 36.44, 36.54, 43.93, 125.58, 128.15, 129.00, 142.80, 215.70; minor isomer **10'**: δ -1.02, 25.78, 26.44, 34.52, 34.57, 41.40, 44.07, 125.95, 127.38, 128.10, 146.42, 215.59; EIMS m/z (relative intensity) major isomer **10**: 290 (M^+ , 1), 190 (70), 73 (100); CIMS m/z (relative intensity) minor isomer **10'** 291 (M^++1 , 100), 275 (26), 172 (24), 105 (28), 73 (8); IR (neat, cm^{-1}) 1704; HRMS (EI) major isomer **10**: calcd for $\text{C}_{18}\text{H}_{30}\text{OSi}$ 290.2066. Found 290.2061. HRMS (CI) minor isomer **10'**: calcd for $\text{C}_{18}\text{H}_{31}\text{OSi}$ (M^++H): 291.2144. Found 291.2148.

(Z)-9,9-Dimethyl-8-(trimethylsiloxy)-4-(triphenylsilyl)-1,7-decadiene (11)

Purification by flash chromatography (elution with hexane/ Et_2O , 100:1) provided silyl enol ether **11** (161 mg, 63%). $R_f = 0.2$ (hexane/ Et_2O , 100:1); ^1H NMR (270 MHz, CDCl_3) δ 0.19 (s, 9H), 1.04 (s, 9H), 1.5-1.6 (m, 1H), 1.7-1.9 (m, 2H), 1.9-2.3 (m, 3H), 2.5-2.6 (m, 1H), 4.40 (t, 1H, $J = 6.3$ Hz), 4.91 (m, 2H), 5.79 (m, 1H), 7.33-7.70 (m, 15H); ^{13}C NMR (68 MHz, CDCl_3) δ 1.12, 23.02, 26.24, 28.63, 30.28, 34.67, 36.41, 103.68, 115.08, 128.09, 129.25, 134.81, 135.78, 139.28, 158.17; EIMS m/z (relative intensity) 512 (M^+ , 0.5), 259 (100), 185 (35), 73 (88); IR (neat, cm^{-1}) 1656, 1637, 1251; HRMS calcd for $\text{C}_{33}\text{H}_{44}\text{OSi}_2$: 512.2930. Found 512.2949.

2,2-Dimethyl-7-(triphenylsilyl)-3-tridecanone (12)

Purification by flash chromatography (elution with hexane/ Et_2O , 20:1) provided a δ -silylketone **12** (152 mg, 63%). $R_f = 0.1$ (hexane/ Et_2O , 100:1); ^1H NMR (270 MHz, CDCl_3) δ 0.82 (t, 3H, $J = 6.4$ Hz), 1.06 (s, 9H), 1.1-1.2 (m, 6H), 1.4-1.6 (m, 6H), 1.6-1.7 (m, 3H), 2.30-2.35 (m, 2H), 7.26-7.57 (m, 15H); ^{13}C NMR (68 MHz, CDCl_3) δ 14.16, 22.67, 23.91, 24.80, 26.43, 29.64, 30.31, 30.66, 30.78, 31.71, 36.91, 44.05, 127.63, 129.07, 134.96, 135.85, 215.59; EIMS m/z (relative intensity) 484 (M^+ , 2), 427 (10), 259 (100); IR (neat, cm^{-1}) 1706; HRMS calcd for $\text{C}_{33}\text{H}_{44}\text{OSi}$: 484.3161. Found: 484.3150.

2,2-Dimethyl-7-hydroxy-3-tridecanone (13)

Purification by flash chromatography (elution with hexane/ Et_2O , 3:1) provided a δ -hydroxyketone **13** (21 mg, 88%). $R_f = 0.1$ (hexane/ Et_2O , 3:1); ^1H NMR (270 MHz, CDCl_3) δ 0.81 (t, 3H, $J = 7.0$ Hz), 1.07 (s, 9H), 1.1-1.4 (m, 12H), 1.55-1.71 (m, 2H), 2.45 (t, 2H, $J = 6.9$ Hz), 3.44-3.52 (m, 1H); ^{13}C NMR (68 MHz, CDCl_3) δ 14.17, 19.77, 22.69, 25.70, 26.52, 29.41, 31.89, 36.32, 37.00, 37.47, 44.11, 71.50, 215.97; EIMS m/z (relative intensity) 242 (M^+ , 2), 224 (39),

167 (100), 57 (81); IR (neat, cm^{-1}) 3431, 1706; HRMS calcd for $\text{C}_{15}\text{H}_{30}\text{O}_2$: 242.2246. Found: 242.2245.

2,2-Dimethyl-4-(1-propenyl)-7-(triphenylsilyl)-3-dodecanone (14)

Purification by flash chromatography (elution with hexane/ Et_2O , 20:1) provided a δ -silylketone **13** (162 mg, 62%, diastereomer ratio 60:40 (determined by ^{13}C NMR)). R_f = 0.5 (hexane/ Et_2O , 5:1); ^1H NMR (270 MHz) (signals for minor isomer are in square brackets) δ 0.83 (t, 3H, J = 6.7 Hz), 0.99 (s, 9H) [1.01 (s)], 1.1-1.7 (m, 15H), 1.94-2.15 (m, 1H), 2.14-2.22 (m, 1H), 2.71-2.77 (m, 1H), 4.88-4.95 (m, 2H), 5.49-5.62 (m, 1H), 7.2-7.5 (m, 15H); ^{13}C NMR (68 MHz, CDCl_3) (signals for minor isomer are in square brackets) δ 14.16, 22.64, 24.06 [24.12], 26.29 [26.31], 28.97 [29.06], [29.49] 29.53, 30.05, 30.57, 31.70, [33.07] 33.25, 36.72 [36.80], 44.30, 45.72 [45.86], [116.40] 116.43, 127.64, 129.09, 134.82, 135.82, [135.98] 136.02, 217.62 [217.68]; EIMS m/z (relative intensity) 524 (M^+ , 9), 467 (21), 259 (100); IR (neat, cm^{-1}) 1701, 1639; HRMS calcd for $\text{C}_{36}\text{H}_{48}\text{OSi}$: 524.3474. Found: 524.3464; Anal. Calcd for $\text{C}_{36}\text{H}_{48}\text{OSi}$: C, 82.38; H, 9.22. Found: C, 82.16; H, 9.08.

2,2-Dimethyl-4-(1-propenyl)-7-hydroxy-3-dodecanone (15)

Purification by flash chromatography (elution with hexane/ Et_2O , 3:1) provided a δ -hydroxyketone **18** (33 mg, 70%, diastereomer ratio 60:40 (determined by ^{13}C NMR)). R_f = 0.1 (hexane/ Et_2O , 3:1); ^1H -NMR (270 MHz, CDCl_3) δ 0.88 (t, 3H, J = 7.0 Hz), 1.14 (s, 9H), 1.2-1.7 (m, 14H), 2.05-2.12 (m, 1H), 2.26-2.35 (m, 1H), 2.93-3.02 (m, 1H), 3.49-3.55 (m, 1H), 4.98-5.06 (m, 2H), 5.61-5.77 (m, 1H); ^{13}C -NMR (68 MHz, CDCl_3) (signals for minor isomer are in square brackets) δ 14.18, 22.69, 25.64, 26.43 [26.47], 28.05 [28.12], 29.38, 31.86, 35.21 [35.25], 36.95 [37.02], 37.39 [37.52], 44.48, 45.21, [71.84] 71.94, 116.72, [135.82] 135.85, 217.86, [218.04]; EIMS m/z (relative intensity) 282 (M^+ , 8), 225 (100), 179 (43), 95 (48), 57 (56); IR (neat, cm^{-1}) 3402, 1707; HRMS calcd for $\text{C}_{18}\text{H}_{34}\text{O}_2$: 282.2559. Found: 282.2558.

(1-E,5-Z)-7-Methyl-1,6-bis(trimethylsiloxy)-1-(triphenylsilyl)-1,5-octadiene (16)

The preparation of α,δ -dianion was performed as same as above. The three-way stopcock was then connected to a vacuum line and also to a balloon filled with carbon monoxide through a glass column packed with anhydrous calcium chloride. After two cycles of evacuation of nitrogen and filling with carbon monoxide, the reaction mixture was stirred under the atmosphere of carbon monoxide for 2 h at room temperature. After having been cooled to $-78\text{ }^\circ\text{C}$, the

resulting brown solution was quenched with chlorotrimethylsilane. The quenched mixture was diluted with sat. NH_4Cl aq. and extracted with pentane, dried (MgSO_4) and concentrated *in vacuo*. The residue was purified by flash column chromatography on flash silica gel (elution with hexane/ Et_2O , 100:1) to furnish **16** (294 mg) in 53% yield. $R_f = 0.15$ (hexane/ Et_2O , 100:1); ^1H -NMR (270 MHz, CDCl_3) δ -0.17 (s, 9H), 0.11 (s, 9H), 1.03 (d, 6H, $J = 6.6$ Hz), 2.1-2.2 (m, 1H), 2.07-2.26 (m, 4H), 4.49 (t, 1H, $J = 6.3$ Hz), 5.29 (t, 1H, $J = 6.6$ Hz), 7.26-7.56 (m, 15H); ^{13}C -NMR (68 MHz, CDCl_3) δ 1.39, 1.47, 21.38, 25.90, 27.14, 34.99, 105.40, 128.12, 130.16, 131.45, 134.62, 137.19, 152.80, 156.82; EIMS m/z (relative intensity) 558 (M^+ , 47), 259 (50), 73 (100); IR (neat, cm^{-1}) 1666, 1610, 1252; HRMS calcd for $\text{C}_{33}\text{H}_{46}\text{O}_2\text{Si}_3$: 558.2805. Found 558.2826.

7,7-Dimethyl-2-(triphenylsilyl)-6-oxooctanal (**17**)

Purification by flash chromatography (elution with hexane/ Et_2O , 5:1) provided a keto aldehyde **17** (261 mg, 61%). $R_f = 0.12$ (hexane/ Et_2O , 5:1); ^1H -NMR (500 MHz, CDCl_3) δ -1.06 (s, 9H), 1.50-1.63 (m, 3H), 2.15-2.17 (m, 1H), 3.21-3.23 (m, 1H), 7.23-7.62 (m, 15H), 9.71 (s, 1H); ^{13}C -NMR (125 MHz, CDCl_3) δ -24.60, 25.09, 26.31, 36.14, 43.89, 47.68, 128.11, 130.07, 131.99, 135.77, 202.70, 215.22; EIMS m/z (relative intensity) 428 (M^+ , 2), 259 (100), 57 (33); IR (neat, cm^{-1}) 1701; HRMS calcd for $\text{C}_{28}\text{H}_{32}\text{O}_2\text{Si}$: 428.2172, Found 428.2165.

- 1) Nakahira, H.; Ryu, I.; Ikebe, M.; Oku, Y.; Ogawa, A.; Kambe, N.; Sonoda, N.; Murai, S. *J. Org. Chem.* **1992**, *57*, 17.

U4-MAH-98 22.03.03

YT-072, PRO

DFILE SAVING

OBNUC 1H

EXMOD NON

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OBSET 112.00 kHz

OBFIN 5800.0 Hz

POINT 32768

FREQU 5405.4 Hz

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PW1 4.7 us

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CTEMP 22.5 c

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EXREF 7.26 ppm

BF 0.10 Hz

RGAIN 22

OPERATOR :

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15-JUN-98 16:53:40

YT-089, CRUDE

DEFILE SAVING

OBNUC 1H

EXMOD NON

OFR 270.05 MHz

OBSET 112.00 kHz

OBFIN 5800.0 Hz

POINT 32768

FREQ 5405.4 Hz

SCANS 16

ACQTM 3.031 sec

PD 3.969 sec

PW1 4.7 US

IRNUC 1H

CTEMP 24.3 C

SLVNT CDCL3

EXREF 0.00 ppm

BF 0.10 Hz

RGAIN 17

OPERATOR :

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2.000

5.115

18-NOV-97 19:24:53

DFILE SAVING

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EXMOD NON

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OBSET 112.00 KHz

OBFIN 5800.0 Hz

POINT 32768

FREQ 5405.4 Hz

SCANS 16

ACQTM 3.031 sec

PD 3.969 sec

PW1 4.7 us

IRNUC 1H

CTEMP 21.4 c

SLVNT CDCL3

EXREF 0.00 ppm

BF 0.10 Hz

RGAIN 16

OPERATOR :

YT-040, PRODUCT

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1.000 →
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13

13.7.0 Hz

YT-074, PRO

10-MAR-98 13:58:17
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 POINT 32768
 FREQ 5405.4 Hz
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 OPERATOR :

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17

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