Supporting Information for

Bis(2,2,2-trifluoroethyl)bromophosphonoacetate, a Novel HWE Reagent for the Preparation of (*E*)-α-Bromoacrylates: A General and Stereoselective Method for the Synthesis of Trisubstituted Alkenes.

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General

Unless otherwise noted, all reactions were carried out in oven-dried glassware under a nitrogen atmosphere. Tetrahydrofuran (THF) was distilled from sodium metal/benzophenone ketyl. Dichloromethane (CH_2Cl_2) was distilled from calcium hydride. All other dry solvents were purchased from Aldrich in SureSeal™ containers. All other commercially obtained reagents were used as received. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian 400 spectrometer. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Infrared spectra were recorded on a JASCO FT-IR-8900 spectrometer. Mass spectra were obtained on a JEOL HX-100, an SX-102A or a JMS-AX-505H mass spectrometer. Analytical TLC was performed on 0.25 mm pre-coated Merck silica gel 60 F_{254} plates. Flash column chromatography was performed on Merck silica gel 60 (230–400 mesh).

Experimental

Ethyl bis(trifluoroethyl)bromophosphonoacetate 3a

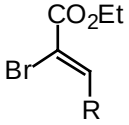
A solution of sodium hydroxide (40.0 g, 1.0 mol) in H_2O (120 ml) was cooled to 0 °C in an ice-salt bath, and bromine (25.5 ml, 0.50 mol) was slowly added stirring over 30 min such that the temperature of the mixture did not exceed 10 °C. Methyl bis(2,2,2-trifluoroethyl)phosphonoacetate (**4**) (50.0 g, 0.13 mol) was added to the solution for 5 min, and the resulting mixture was added H_2O (200ml) and extracted with chloroform ($CHCl_3$) (200 ml x 1, 100 ml x 2). The combined organic extracts were washed with H_2O (100 ml x 4), dried over $MgSO_4$, and concentrated in vacuo after filtration. The product was distilled under reduced pressure (bp 100–102 °C, 1 mmHg) to obtain methyl bis(2,2,2-trifluoroethyl)dibromophosphonoacetate (41.8 g, 85% yield) as a colorless oil. This dibromide (21.3 g, 44.2 mmol) was dissolved in EtOH (40 ml), and the solution was cooled to –30 °C. A solution of $SnCl_2$•$2H_2O$ (12.9 g, 56.0 mmol) in H_2O (100 ml) was added to the reaction mixture for 40 min such that the temperature did not exceed –25 °C. After addition was completed, the reaction mixture was extracted with $CHCl_3$ (200 ml x 1, 100 ml x 1, 50 ml x 2). The combined extracts were washed with H_2O (100 ml x 4), dried over $MgSO_4$, and concentrated in vacuo after filtration. In order to

remove dibromide and **4**, the residue was purified by flash column chromatography. The column was packed with silica gel (290 g) using a mixture of CH_2Cl_2 (750 ml) and 4N HCl in ethyl acetate (30 ml). After the solvent (CH_2Cl_2:acetone = 50:1, 500 ml) was flowed through the column, the crude product was applied and eluted (one fraction; 65 ml). Combined fractions (from No. 7 to 20), which contained only **3a**, were concentrated in vacuo. The residue was dissolved in $CHCl_3$ (300 ml) and washed with H_2O (50 ml x 4), dried over $MgSO_4$, and concentrated in vacuo after filtration. The residue was distilled under reduced pressure (bp 85–87 °C, 0.4 mmHg) to obtain **3a** (12.2 g, 70% yield) as a colorless oil: IR (film) υ_{max} 3029, 2966, 1745, 1455, 1440, 1421, 1375, 1301, 1268, 1175, 1102, 1072, 1012, 964, 903, 886, 846, 808, 726, 659, 556, 232, 478, 448 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ ppm: 3.87 (s, 3 H), 4.45–4.60 (m, 5 H); HRMS (FAB) calcd for $C_7H_{10}O_5BrF_6P$ $(M+H)^+$ 396.9275, obsd 396.9271; Anal. Calcd for $C_7H_9O_5BrF_6P$: C, 21.18; H, 2.03. Found: C, 20.87; H, 2.14.

General procedure of HWE reaction

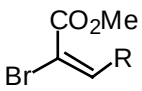
A solution of **1a** or **3a** (1.1 mmol) and 18-C-6/CH_3CN (397 mg, 1.2 mmol) in THF (8 ml) was cooled to –78 °C, then 1.0 M potassium *tert*-butoxide solution in THF (1.05 ml, 1.05 mmol) was added to the solution. After stirring for 30 min at –78 °C, aldehyde (1.0 mmol) was added to the reaction mixture and the stirring was continued. When the reaction was completed, a saturated aqueous NH_4Cl was added to the solution and the organic material was extracted with AcOEt. The combined organic extracts were washed with H_2O and brine, dried over Na_2SO_4, and concentrated in vacuo after filtration. The residue was purified by silica gel flash chromatography to afford methyl α-bromo-acrylate.

The spectrum and analytical data of methyl *(Z)*-α-bromo-acrylate



R = Ph[1]: IR ($CHCl_3$ soln.) υ_{max} 2985, 2940, 2907, 1719, 1612, 1577, 1492, 1475, 1466, 1447, 1392, 1368, 1340, 1263, 1184, 1095, 1077, 1039, 1000, 989, 969, 928, 882, 866, 850, 808 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.39 (3 H, t, *J* 7.1 Hz), 4.36 (2 H, q, *J* 7.1 Hz), 7.40–7.46 (3 H, m), 7.84–7.87 (2 H, m), 8.22 (1 H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 62.8, 113.0, 128.3, 130.0, 130.1, 133.6, 140.6, 163.1.

The spectrum and analytical data of methyl *(E)*-α-bromo-acrylate



R = Ph[2]: IR ($CHCl_3$ soln.) υ_{max} 2954, 1728, 1611, 1576, 1496, 1447, 1435, 1346, 1316, 1289, 1244, 1210, 1183, 1077, 1031, 1018, 1007, 928, 902, 878, 831 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.76 (3 H, s), 7.26–7.38 (6 H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.0, 111.1, 128.1, 128.5, 129.0, 134.8, 140.1, 164.8; HRMS (EI) calcd for $C_{10}H_9O_2Br$ $(M)^+$ 239.9786, obsd 239.9788.

R = 4-OMe-Ph: IR ($CHCl_3$ soln.) υ_{max} 2954, 2937, 2911, 2841, 1724, 1605, 1575, 1511, 1464, 1437, 1422, 1349, 1300, 1257, 1223, 1206, 1176, 1115, 1033, 1004, 941, 908, 887, 831 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.79 (3 H, s), 3.82 (3 H, s), 6.81 (2 H, d, *J* 9.5 Hz), 7.28 (2 H, d, *J* 9.5 Hz), 7.32 (1 H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.0, 55.3, 108.7, 113.9, 127.2, 130.2, 132.5, 140.4, 160.3; HRMS (EI) calcd for $C_{11}H_{11}O_3Br$ $(M)^+$ 269.9892, obsd 269.9892.

R = 3-OMe-Ph: IR ($CHCl_3$ soln.) υ_{max} 2954, 2914, 2839, 1729, 1600, 1579, 1489, 1466, 1456, 1435, 1341, 1292, 1261, 1216, 1184, 1151, 1142, 1087, 1051, 1042, 1019, 1009, 956, 937, 928, 906, 878, 825, 809 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.77 (3 H, s), 3.79 (3 H, s), 6.80–6.89 (3 H, m), 7.23–7.27 (1 H, m), 7.32 (1 H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.0, 55.2, 111.2, 113.3, 114.8, 120.6, 129.5, 135.9, 139.5, 159.5, 164.9; HRMS (EI) calcd for $C_{11}H_{11}O_3Br$ $(M)^+$ 269.9892, obsd 269.9989.

R = 4-NO_2-Ph: mp 80–81 °C; IR ($CHCl_3$ soln.) υ_{max} 2955, 1731, 1600, 1525, 1493, 1437, 1348, 1293, 1243, 1184, 1112, 1016, 1003, 882, 861, 850 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.78 (3 H, s), 7.44 (1 H, s), 7.45 (2 H, d, *J* 8.7 Hz), 8.21 (2 H, d, *J* 8.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.3, 115.2, 123.7, 128.9, 138.2, 141.1, 147.6, 163.9; HRMS (FAB) calcd for $C_{10}H_8O_4NBrK$ $(M+K)^+$ 323.9274, obsd 323.9272; Anal. Calcd for $C_{10}H_8O_4NBr$: C, 41.99; H, 2.82; N, 4.90. Found: C, 41.90; H, 2.96; N, 4.71.

R = 3-NO_2-Ph: mp 79–80 °C; IR ($CHCl_3$ soln.) υ_{max} 3089, 2955, 1731, 1611, 1578, 1534, 1480, 1437, 1355, 1311, 1295, 1282, 1243, 1224, 1185, 1101, 1082, 1008, 907, 889, 869, 824 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.80 (3 H, s), 7.44 (1 H, s), 7.54 (1 H, t, *J* 7.8 Hz), 7.61 (1 H, d, *J* 7.8 Hz), 8.19–8.22 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.3, 114.6, 123.1, 123.6, 129.4, 134.0, 136.3, 138.1, 163.8, 170.3; HRMS (EI) calcd for $C_{10}H_8O_4NBr$ $(M)^+$ 284.9637, obsd 284.9639; Anal. Calcd for $C_{10}H_8O_4NBr$•1/3 H_2O: C, 41.12; H, 2.99; N, 4.80. Found: C, 40.84; H, 2.72; N, 4.60.

R = cinnamyl: IR ($CHCl_3$ soln.) υ_{max} 3083, 2954, 2846, 1713, 1613, 1579, 1568, 1490, 1448, 1436, 1357, 1329, 1316, 1302, 1268, 1246, 1222, 1206, 1189, 1181, 1160, 1146, 1110, 1172, 1045, 1030, 1016, 1000, 986, 975, 938, 910, 882, 852, 810 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.88 (3 H, s), 6.83 (1 H, d, *J* 15.6 Hz), 7.31 (1 H, d, *J* 11.5 Hz), 7.32–7.37 (3H, m), 7.50–7.52 (2H, m), 7.81 (1 H, dd, *J* 11.5, 15.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.0, 110.3, 124.9, 127.6, 128.8, 128.9, 129.4, 136.0, 141.7, 146.5, 163.3; HRMS (EI) calcd for $C_{12}H_{11}O_2Br$ $(M)^+$ 265.9942, obsd 265.9933.

R = furyl: IR ($CHCl_3$ soln.) υ_{max} 2954, 2846, 1726, 1595, 1556, 1474, 1457, 1437, 1390, 1349, 1251, 1211, 1183, 1153, 1145, 1093, 1022, 962, 931, 921, 907, 886, 829, 810 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.88 (3 H, s), 6.47 (1 H, dd, *J* 1.9, 3.5 Hz), 7.09 (1 H, d, *J* 3.5 Hz), 7.23 (1 H, s), 7.47 (1 H, d, *J* 1.9, Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.0, 107.0, 112.4, 115.4, 129.4, 144.3, 149.3, 164.1; HRMS (EI) calcd for $C_8H_7O_3Br$ $(M)^+$ 229.9579, obsd 229.9578.

R = phenethyl: IR ($CHCl_3$ soln.) υ_{max} 2954, 2929, 2862, 1718, 1611, 1497, 1454, 1437, 1354, 1306, 1247, 1224, 1173, 1087, 1030, 1003, 909, 880, 843, 811 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.75–2.87 (4 H, m), 3.80 (3 H, s), 6.71 (1 H, t, *J* 7.4 Hz), 7.18–7.23 (3 H, m), 7.30 (2 H, t, *J* 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 33.0, 34.7, 52.9, 111.3, 126.3, 128.4, 128.5, 140.5, 147.9, 163.2; HRMS (EI) calcd for $C_{12}H_{13}O_2Br$ $(M)^+$ 268.0099, obsd 268.0097.

R = geranyl: IR ($CHCl_3$ soln.) υ_{max} 2970, 2953, 2930, 2917, 2857, 1708, 1618, 1565, 1436, 1371, 1320, 1246, 1216, 1206, 1185, 1137, 1107, 1043, 1102, 941, 911, 822, 859, 825, 810 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.61 (3 H, s), 1.69 (3 H, s), 1.84 (3 H, s), 2.16 (4 H, m), 3.83 (3 H, s), 5.07–5.10 (1 H, m), 6.86 (1 H, d, *J* 11.7 Hz), 7.42 (1 H, d, *J* 11.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.2, 17.7, 25.7, 16.4, 40.7, 52.8, 108.3, 121.7, 132.3, 142.4, 151.1, 163.6; HRMS (EI) calcd for $C_{13}H_{19}O_2Br$ $(M)^+$ 286.0568, obsd 286.0571.

R = *n*-Bu: IR ($CHCl_3$ soln.) υ_{max} 2958, 2931, 2873, 2863, 1717, 1611, 1466, 1458, 1437, 1380, 1352, 1319, 1297, 1250, 1209, 1189, 1134, 1035, 1007, 990, 943, 878, 814, 803 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.91 (3 H, t, *J* 7.3 Hz), 1.31–1.48 (4 H, m), 2.51 (2 H, q, *J* 7.6 Hz), 3.82 (3 H, s), 6.69 (1 H, t, *J* 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 22.3, 30.9, 31.2, 52.8, 110.4, 149.5, 163.4; HRMS (EI) calcd for $C_8H_{13}O_2Br$ $(M)^+$ 220.0099, obsd 220.0101.

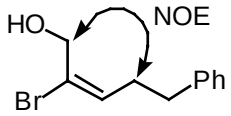
R = Cy: IR ($CHCl_3$ soln.) υ_{max} 2932, 2854, 1717, 1610, 1449, 1437, 1363, 1346, 1291, 1267, 1247, 1207, 1187, 1146, 1096, 1005, 964, 932, 903, 879, 811 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.07–1.37 (5 H, m), 1.63–1.77 (5 H, m), 2.91–3.01 (1 H, m), 3.82 (3 H, s), 6.50 (1 H, d, *J* 10.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.4, 25.7, 32.1, 40.4, 52.8, 109.1, 154.0, 163.4; HRMS (EI) calcd for $C_{10}H_{15}O_2Br$ $(M)^+$ 246.0255, obsd 246.0249.

R = CH_2OBn: IR ($CHCl_3$ soln.) υ_{max} 3086, 2954, 2888, 2862, 1715, 1621, 1498, 1454, 1438, 1351, 1317, 1246, 1221, 1211, 1194, 1091, 1029, 987, 909, 879, 830, 811 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.80 (3 H, s), 4.47 (2 H, d, *J* 5.1 Hz), 4.54 (2 H, s), 6.91 (1 H, t, *J* 5.1 Hz), 7.29–7.38 (5 H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 53.1, 69.2, 72.9, 110.5, 127.8, 127.9, 128.5, 137.5, 148.0, 163.0; HRMS (EI) calcd for $C_{12}H_{14}O_3Br$ $(M+H)^+$ 287.0106, obsd 287.0095.

2-Bromo-5-phenyl-2-pentene-1-ol

A solution of α-bromoacrylates (461 mg, 1.71 mmol) in CH_2Cl_2 (10 ml) was cooled to –78 °C and added DIBAL-H (5.1 ml, 5.1 mmol). The reaction mixture was stirred at –78 °C for 1 hour, then added Na_2SO_4•$10H_2O$ (1.8 g) and warmed up to rt. The mixture was filtrated and the filtrate was concentrated in vacuo. The residue was purified by flash column chromatography (hexane/ethyl acetate = 4:1) to

give allylic alcohol as a colorless oil (362 mg, 88% yield): IR ($CHCl_3$ soln.) υ_{max} 3599, 3563, 2931, 2863, 1949, 1873, 1809, 1732, 1644, 1603, 1497, 1454, 1385, 1336, 1247, 1189, 1178, 1091, 1063, 1031m 978, 943, 907, 858 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.39 (1 H, t, *J* 6.7 Hz), 2.44 (2 H, q, *J* 7.6 Hz), 2.71 (2 H, t, *J* 7.6 Hz), 4.09 (2 H, d, *J* 6.7 Hz), 6.04 (1 H, t, *J* 7.6 Hz), 7.17 (2 H, dd, *J* 7.4, 1.4 Hz), 7.22 (1 H, tt, *J* 7.4, 1.4 Hz), 7.31 (2 H, td, *J* 7.4, 1.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.5, 35.2, 62.6, 125.3, 126.3, 128.5, 128.6, 133.7, 140.6 ; HRMS (FAB) calcd for $C_{11}H_{13}OBrK$ $(M+K)^+$ 278.9787, obsd 278.9778.



Compound 6

A solution of allylic alcohol (360 mg, 1.49 mmol) and imidazole (203 mg, 2.98 mmol) in DMF (8 ml) was added TBS-Cl (270 mg, 1.79 mmol) at 0 °C, then the reaction mixture was stirred at rt for 20 min. Water was added to the solution, then the mixture was extracted with ether. The organic layers were washed with water and brine, dried over Na_2SO_4, concentrated in vacuo after filtration. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 20:1) to give **6** as a colorless oil (509 mg, 96% yield): IR ($CHCl_3$ soln.) υ_{max} 3087, 2955, 2930, 2898, 2886, 2858, 1733, 1703, 1644, 1604, 1497, 1471, 1464, 1455, 1407, 1390, 1376, 1363, 1257, 1211, 1201, 1173, 1108, 1085, 1041, 1031, 1006, 964, 939, 905, 839 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.07 (6 H, m), 0.89 (9 H, s), 2.42 (2 H, q, *J* 7.7 Hz), 2.68 (2 H, t, *J* 7.7 Hz), 4.19 (2 H, s), 5.99 (1H, t, *J* 7.7 Hz), 7.14–7.30 (5 H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.2, 18.4, 25.9, 31.5, 35.4, 63.2, 125.1, 126.1, 128.4, 128.5, 133.2, 140.8; HRMS (FAB) calcd for $C_{17}H_{26}OBrSi$ $(M–H)^+$ 353.0936, obsd 353.0940.

Compound 7

A solution of **6** (100 mg, 0.28 mmol), $Pd(PPh_3)_4$ (13 mg, 5 mol%), and catalytic amount of 2,4-di-*tert*-butylphenol in THF (3 ml) was added vinyltributyltin (98 μl, 0.34 mmol), then the mixture was stirred at rt for 3 days. The solvent was removed in vacuo and the residue was purified by silica gel flash column chromatography (hexane/toluene = 15:1) to give **7** as a pale yellow oil (56 mg, 66% yield): IR ($CHCl_3$ soln.) υ_{max} 3088, 2956, 2885, 2858, 2804, 1641, 1604, 1496, 1471, 1464, 1454, 1416, 1390, 1362, 1255, 1232, 1222, 1212, 1199, 1080, 1031, 1006, 993, 839, 807 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.06 (6 H, m), 0.88 (9 H, s), 2.50 (2 H, q, *J* 7.5 Hz), 2.70 (2 H, t, *J* 7.5 Hz), 4.26 (2 H, s), 4.99 (1 H, d, *J* 11.0 Hz), 5.30 (1 H, d *J* 17.5 Hz), 5.61 (1 H, t, *J* 7.5 Hz), 6.25 (1 H, dd, *J* 11.0, 17.5 Hz), 7.16–7.20 (3 H, m), 7.27 (2 H, t, *J* 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.2, 18.3, 25.9, 30.0, 35.9, 57.7, 112.4, 125.9, 128.35, 128.41, 133.7, 137.8, 138.8, 141.6; HRMS (FAB) calcd for $C_{19}H_{29}OSi$ $(M–H)^+$ 301.1988, obsd 301.1989.

Compound 9

A solution of **8** (105 mg, 0.56 mmol) in THF (1 ml) was cooled to 0 °C and added 0.5 M solution of 9-BBN in THF (2.2 ml, 1.1 mmol), then the reaction mixture was stirred at rt for 4 hours. After addition of water (0.1 ml), the resulting mixture was concentrated in vacuo to give boron reagent. Compound **6** (100 mg, 0.28 mmol), Cs_2CO_3 (165 mg, 0.51 mmol), $PdCl_2(dppf)•CH_2Cl_2$ (12 mg, 5 mol%), and Ph_3As (9 mg, 10 mol%) were dissolved in DMF (2 ml) and stirred at rt for 10 min. Then the mixture was added the boron reagent and stirred at 50 °C for 2 hours. The reaction mixture was poured into water and extracted with ethyl acetate. The combined organic layers were washed with water and brine, dried over Na_2SO_4, concentrated in vacuo after filtration. The residue was purified by silica gel flash column chromatography (hexane/ether = 50:1) to give **9** as a colorless oil (105 mg, 81% yield): IR ($CHCl_3$ soln.) υ_{max} 4215, 3086, 2955, 2930, 2898, 2886, 2858, 1732, 1603, 1496, 1471, 1463, 1454, 1406, 1390, 1362, 1256, 1083, 1032, 1006, 967, 938, 854, 839 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.02 (6 H, s), 0.03 (6 H, s), 0.87 (9 H, s), 0.88 (9 H, s), 1.37–1.52 (4 H, m), 2.07 (2 H, t, *J* 7.3 Hz), 2.33 (2 H, q, *J* 7.4 Hz), 2.63 (2 H, t, *J* 7.4 Hz), 3.59 (2 H, t, *J* 6.3 Hz), 4.03 (2 H, s), 5.24 (1 H, t, *J* 7.4 Hz), 7.14–7.20 (3 H, m), 7.26–7.28 (2 H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.3, –5.2, 18.3, 18.4, 24.4, 25.9, 26.0, 29.5, 32.7, 34.3, 36.3, 60.2, 63.3, 125.3, 125.7, 128.3, 128.5, 139.4, 142.0; HRMS (FAB) calcd for $C_{27}H_{49}O_2Si_2$ $(M–H)^+$ 461.3271, obsd 461.3267.

Reference

1. Rossi, R.; Bellina, F.; Bechini, C.; Mannina, L.; Vergamini, P. *Tetrahedron* **1998**, *54*, 135 and references cited therein.
2. Nakamura, I.; Harada, K. *Heterocycles* **1978**, *9*, 473.