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Short communication

An alternative and effective catalyst in the stereospecific reaction of *Z*-1-aryl-1-stannyl-2-silylethenes with allyl bromide

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The $Pd(dba)_2$-catalyzed reaction of *Z*-1-aryl-1-(tributylstannyl)-2-(trimethylsilyl)ethenes with allyl bromide in the presence of copper(I) iodide is reported for the first time. The reaction in the presence of 0.5 mol% $Pd(dba)_2$ and 8 mol% CuI in dimethylformamide takes place at room temperature to give *E*-2-aryl-1-(trimethylsilyl)penta-1,4-dienes exclusively in isolated yields of 62–99%. A putative reaction mechanism is proposed. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: *Z*-1-aryl-2-silyl-1-stannylethenes; allyl bromide; cross-coupling; $Pd(dba)_2$–CuI; *E*-2-aryl-1-silylpenta-1,4-dienes

2-Aryl-1-silylpenta-1,4-dienes, including 2-phenyl-1-silylpenta-1,4-diene, are potentially useful compounds in 1,3-dipolar addition reactions with various dipoles such as nitrones[1,2] or nitrile oxides.[2–4] When using a 2-phenyl-1-silylpenta-1,4-diene in the reaction, use of a specific stereoisomer is required. *Z*-2-Phenyl-1-silylpenta-1,4-diene has been reportedly synthesized via a three-component (phenylacetylene, iodotrimethylsilane, and allyltributylstannane) reaction catalyzed by $Pd(PPh_3)_4$. However, the reaction also forms the *E*-isomer in a 44:56 ratio with the *Z*-isomer.[5] *Z*-1-Phenyl-2-silylethenylcopper reacts with allylic phosphates or allylic phosphinates to produce *E*-1-silyl-2-phenylpenta-1,4-diene (**2a**).[6] However, the reaction is in need of an equimolar air- and temperature-sensitive copper reagent and special phosphine compounds—allylic phosphates or allylic phosphinates. Air-stable *Z*-2-(trimethylsilyl)-1-(tributylstannyl)-1-phenylethene (**1a**) or *Z*-2-(trimethylsilyl)-1-(trimethylstannyl)-1-phenylethene (**1a′**), prepared from the silastannation of phenylacetylene using $Pd(PPh_3)_4$ as a catalyst, reacts with allyl bromide using $BnPdCl(PPh_3)_2$ as a catalyst to give *E*-2-phenyl-1-silylpenta-1,4-diene stereospecifically.[7] However, the reported coupling reaction requires heating (80 °C).[7] *Z*-2-(Trimethylsilyl)-1-(trimethylstannyl)-1-methylethene, prepared from the $Pd(OAc)_2$-catalyzed silastannation of 1-ethoxypropyne in the presence of 1,1,3,3-tetramethylbutyl isonitrile, couples with ally bromide at 50 °C with the use of a $BnPdCl(PPh_3)_2$–CuI catalyst to produce *E*-2-methyl-1-silylpenta-1,4-diene stereospecifically.[8] $Pd(PPh_3)_4$-catalyzed silastannation of (*N*-benzyl-*N*-tosyl) aminoacetylene with tributyl(trimethylsilyl)stannane at 50 °C produces α-stannyl β-silyl enamides, which react with allyl bromide in the presence of $Pd_2(dba)_3$–$AsPh_3$ to form *E*-2-(*N*-benzyl-*N*-toluenesulfonyl)amino-1-silylpenta-1,4-diene.[9] Although the method seems to be suitable for the synthesis of **2a**, it needs large amounts of palladium catalyst (16 mol%), 5 equivalents of copper(I) chloride, and a freeze–thaw process. To the best of our knowledge, there has been no report to date of an energy-saving method for the synthesis of *E*-2-phenyl-1-silylpenta-1,4-dienes from acetylenes. With the goal of a room-temperature reaction, we examined several catalysts in the reaction of **1a**, prepared from phenylacetylene and tributyl(trimethylsilyl)stannane at room temperature,[10] with allyl bromide, and found for the first time that a catalyst composed of a $Pd(dba)_2$–CuI combination is the best choice for achieving the allylation of **1a** at room temperature. (For other silastannations of acetylenes, see Refs 7 and 11–14.) We now report the preliminary results for the reaction of *Z*-1-aryl-1-(tributylstannyl)-2-(trimethylsilyl)ethenes (**1**) with allyl bromide (Scheme 1).

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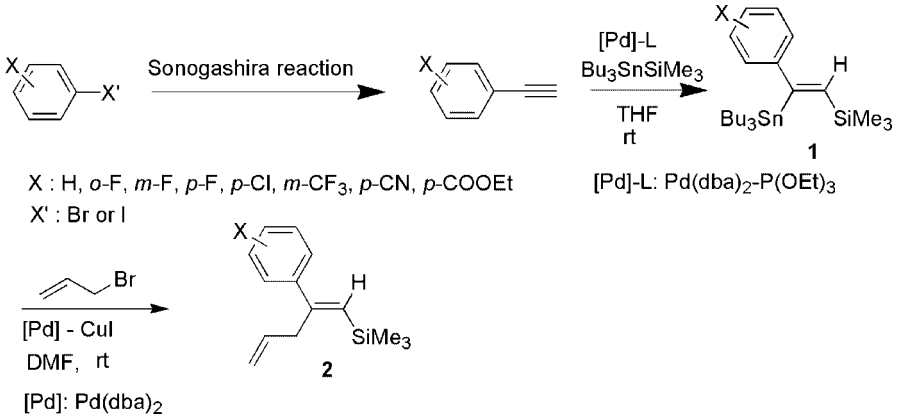

Scheme 1.

Table 1. Stereospecific synthesis of *E*-2-aryl-1-(trimethylsilyl)penta-1,4-dienes

Run	X in Ar	Reaction time (h)[a]	Product no.	Yield[b] (%)	δ(ppm)[c] =CH	δ(ppm)[c] $SiMe_3$
1	H **1a**	2	**2a**	62 (68)	5.94	0.19
2	*o*-F **1b**	2	**2b**	70	5.71	0.20
3	*m*-F **1c**	2	**2c**	95	5.97	0.19
4	*p*-F **1d**	2	**2d**	94	5.88	0.19
5	*p*-Cl **1e**	3	**2e**	82	5.93	0.19
6	*m*-CF_3 **1f**	5	**2f**	82	6.18	0.21
7	*p*-CN **1g**	1	**2g**	99	6.06	0.21
8	*p*-COOEt **1h**	27	**2h**	65	6.04	0.21

[a] All reactions at room temperature.
[b] Isolated yield by column chromatography (silica gel, *n*-hexane). The GLC yield is shown in parentheses.
[c] Chemical shifts (^{1}H NMR) in $CDCl_3$.

Arylacetylenes were prepared by the Sonogashira–Hagihara method from the corresponding substituted bromo- or iodo-benzenes in two steps in good yields.[15–17] Next, phenylacetylene and arylacetylenes were subjected to silastannation with tributyl(trimethylsilyl)stannane in the presence of $Pd(dba)_2$–$2P(OEt)_3$ in tetrahydrofuran (THF) at room temperature to afford *Z*-1-aryl-1-(tributylstannyl)-2-(trimethylsilyl)ethenes (**1a**–**h**) in high isolated yields.[7,10–14] Among the adducts, *Z*-1-tributylstannyl-2-trimethylsilyl-1-phenylethene (**1a**) was employed as a substrate for a model reaction to find the proper reaction conditions. A catalyst composed of a $Pd(dba)_2$–CuI combination was first examined in the reaction of **1a** with allyl bromide using dry *N*,*N*-dimethylformamide (DMF) as a solvent. The reaction took place at room temperature and was complete within 2 h, producing *E*-1-trimethylsilyl-2-phenylpenta-1,4-diene (**2a**)[5,7] exclusively with 68% gas–liquid chromatography (GLC) yield (run 1 in Table 1). ^{1}H NMR analysis of **2a** disclosed that the allyl group successfully replaced the tributylstannyl group. The vinyl proton on the C(sp^2) bearing the trimethylsilyl group was observed at 5.94 ppm as a singlet, which was lower than that (5.59 ppm) reported for the *Z*-isomer.[18,19] The downfield shift of the vinyl proton in the *E*-isomer may be caused by a ring current effect of the neighboring phenyl group. Trimethylsilyl protons in the *E*-isomer were observed at 0.19 ppm, which was lower than that of *Z*-**2a** (δ −0.19 ppm);[18,19] however, the observed chemical shift is quite normal compared with other vinyltrimethylsilane derivatives. The abnormal higher field shift of the trimethylsilyl protons in the *Z*-isomer is probably due to the deshielding effect caused by the neighboring phenyl group. Other combination catalysts, such as $PdCl_2$–CuI (in DMF, room temperature, reaction time 2 h; GLC yield 50%), $Pd(OAc)_2$–CuI (DMF, room temperature, 4 h, 63%) or $BnPdCl(PPh_3)_2$ (DMF, room temperature, 14 h, 69%) were also examined and found to be active. Catalysis without copper iodide, such as with $Pd(dba)_2$–PPh_3 (THF, 60 °C, 5 h, 49%) was also effective, but required heating to obtain the 1,4-diene in accessible yields. $Pd(dba)_2$–$P(OEt)_3$ (THF, 60 °C, 3 h, 35%) and $Pd(dba)_2$–P(*o*-tol)$_3$ (THF, 60 °C, 3 h, 0%) were not particularly effective. A combination of copper iodide as

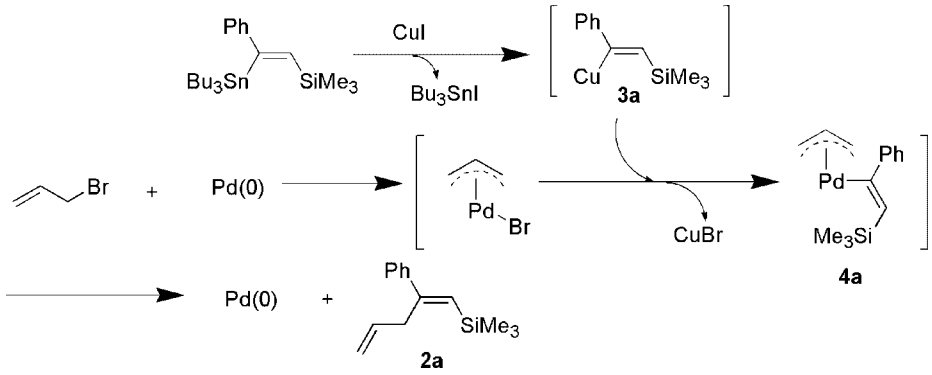

Figure 1. A putative mechanism for $Pd(dba)_2$–CuI-catalyzed cross-coupling of **1** with allyl bromide.

a catalyst component and DMF as a solvent seems to be indispensable in producing the reaction at room temperature. Allylation did not occur at room temperature for the reaction of **1a** with allyl chloride in the presence of $Pd(dba)_2$–CuI in DMF.

A separate reaction of **1a** with allyl bromide conducted under similar conditions to those shown for run 1 in Table 1 gave **2a**[5,7] in an isolated yield of 62%. Other *Z*-1-aryl-2-silyl-1-stannylethenes (**1b**–**h**) were also subjected to the coupling reaction under similar conditions to produce the corresponding 1,4-dienes of *E*-type exclusively with isolated yields of 65–99% (Table 1). Although the reaction of *Z*-1-(tributylstannyl)-1-(*p*-ethoxycarbonylphenyl)-2-trimethylsilylethene (**1e**) needed a longer reaction time, the expected *E*-isomer was obtained in 65% yield. All products **2a**–**h** gave satisfactory spectral data.

We propose a putative mechanism that can accommodate all the observed results in Fig. 1. Thus, copper iodide may react with the silyl(stannyl)ethene **1a** to form a vinyl copper species **3a**, which may spontaneously react with π-allyl palladium bromide[20] to form copper bromide and a putative silylvinyl(π-allyl)palladium intermediate **4a**, from which the expected 1,4-diene **2a** reductively eliminates to liberate the palladium(0) catalyst. The copper bromide produced probably enters into the catalysis as copper iodide.

In conclusion, we have found an alternative method for the stereospecific synthesis of *E*-2-aryl-1-trimethylsilylpenta-1,4-dienes **2**, in which small amounts of $Pd(dba)_2$–CuI effectively catalyze the cross-coupling reaction of *Z*-1-aryl-1-tributylstannyl-2-trimethylsilylethenes (**1**) with allyl bromide at room temperature. The reaction is operationally simple, and gives good yields of *E*-type 2-aryl-1-silylpenta-1,4-dienes exclusively—most unreported thus far. Destannylative allylation of **2** with other allylic bromides, such as 3-bromo-2-methylpropene, 3-bromo-2-phenylpropene, or 4-bromobut-2-ene, is now in progress.

EXPERIMENTAL

Typical procedure for destannylative allylation of *Z*-1-aryl-1-(tributylstannyl)-2-(trimethylsilyl) ethenes

A DMF (0.5 ml) mixture of $Pd(dba)_2$ (2.8 mg, 0.005 mmol) and CuI (16.3 mg, 0.085 mmol) was stirred under nitrogen. Then, a DMF (1 ml) solution of **1a** (456 mg, 0.997 mmol) was added with a micro-syringe and stirred for 5 min. Next, a DMF (0.5 ml) solution of allyl bromide (362 mg, 2.99 mmol) was added. The mixture was stirred at room temperature. After 2 h, GLC analysis disclosed that **1a** was consumed completely. The resulting mixture was passed through a short silica gel (pretreated with triethylamine) column (eluent: *n*-hexane) to remove the catalyst. The eluents collected were concentrated with a rotary evaporator under aspirator vacuum to a volume of ~10 ml. Then, after addition of ether to the concentrate, the resulting two phases were vigorously stirred with aqueous KF for 24–48 h. Filtration of the precipitated fluorotributylstannane, then column chromatography (silica gel (pretreated with triethylamine or neutral), *n*-hexane) gave an analytically pure sample (0.133 g, 62%) of **2a**.[5,7]

Spectral data for **2a** are shown in full below, and are accessible from the American Chemical Society as supplementary materials. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44–7.41 (m, 2H), 7.32–7.21 (m, 3H), 5.94 (s, 1H), 5.80 (ddt, 1H, J = 17.2, 10.5, 6.2 Hz), 5.05 (ddt, 1H, J = 17.2, 1.8, 1.8 Hz), 4.98 (ddt, 1H, J = 10.5, 1.8, 1.8 Hz), 3.38 (dt, 2H, J = 6.2, 1.8 Hz), 0.19 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 154.01, 143.31, 136.50, 129.39, 128.08, 127.29, 126.22, 116.09, 38.72, 0.2 ppm. IR (neat): 3075, 3050, 2950, 1730, 1595, 1668, 1495, 1440, 1245, 915, 855, 835, 760, 695 cm^{-1}. LRMS (EI, 70 eV): 216 (M^+), 201($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{14}H_{20}Si$, 216.1334; found, 216.1355.

By a procedure similar to that for **2a**, other penta-1,4-dienes were obtained from the corresponding *Z*-silyl(stannyl)ethenes (**1**). Analytical data of the new compounds are shown below.

2b. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.25–7.16 (m, 2H), 7.07–7.03 (m, 1H), 6.98 (ddd, 1H, J = 10.7, 8.2, 1.0 Hz), 5.71

(s, 1H), 5.73–5.62 (broad m, 1H), 5.0 (ddt, 1H, $J = 17.0$, 2.0, 1.6 Hz), 4.91 (ddt, 1H, $J = 10.0$, 2.0, 1.6 Hz), 3.35 (a set of two multiplets, 2H, $J = 6.8$ Hz), 0.20 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.2(d, $J = 244.3$ Hz), 151.2, 135.9, 133.0, 132.4 (d, $J = 14.5$ Hz), 130.3 (d, $J = 4.5$ Hz), 128.5 (d, $J = 8.4$ Hz), 123.8 (d, $J = 3.1$ Hz), 116.0, 115.5 (d, $J = 22.9$ Hz), 40.0 (d, $J = 3.8$ Hz), 0.15 ppm. IR (neat): 3040, 2950, 1600, 1480, 1450, 1250, 1100, 850, 760 cm^{-1}. LRMS (EI, 70 eV): 234(M^+), 219 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{14}H_{19}FSi$, 234.1240; found, 234.1243.

2c. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.27–7.19 (m, 2H), 7.14–7.10 (m, 1H), 6.95–6.9 (m, 1H), 5.97 (s, 1H), 5.78 (ddt, 1H, $J = 17.2$, 10.0, 6.0 Hz), 5.05 (ddt, 1H, $J = 17.2$, 1.6, 1.6 Hz), 5.01 (ddt, 1H, $J = 10.2$, 1.6, 1.6 Hz), 3.35 (ddd, 2H, $J = 6.0$, 1.6, 1.6 Hz), 0.19 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.8 (d, $J = 243.5$ Hz), 152.6, 145.7 (d, $J = 6.8$ Hz), 136.1 (d, $J = 4.6$ Hz), 130.7 (d, $J = 6.1$ Hz), 129.4 (d, $J = 8.4$ Hz), 121.9 (d, $J = 2.3$ Hz), 116.4 (d, $J = 3.8$ Hz), 114.0 (d, $J = 21.3$ Hz), 113.2 (d, $J = 21.2$ Hz), 38.6, 0.1 ppm. IR (neat): 3075, 2950, 2900, 1638, 1610, 1580, 1485, 1438, 1250, 1208, 1157, 993, 915, 898, 850, 780, 688 cm^{-1}. LRMS (EI, 70 eV): 234(M^+), 219 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{14}H_{19}FSi$, 234.1240; found, 234.1225.

2d. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39 (dddd, 2H, $J = 9.3$, 5.3, 2.6, 2.5 Hz), 7.00–6.94 (m, 2H), 5.88 (s, 1H), 5.77 (ddt, 1H, $J = 17.0$, 10.2, 6.1 Hz), 5.04 (ddd, 1H, $J = 17.0$, 1.8, 1.8 Hz), 5.00 (ddd, 1H, $J = 10.2$, 1.8, 1.8 Hz), 3.35 (ddd, 2H, $J = 6.1$, 1.8, 1.8 Hz), 0.19 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.2 (d, $J = 234.0$ Hz), 152.8, 139.3 (d, $J = 3.0$ Hz), 136.3 (d, $J = 3.1$ Hz), 129.4 (d, $J = 3.0$ Hz), 127.8 (d, $J = 7.6$ Hz), 116.3, 114.8 (d, $J = 21.3$ Hz), 38.8, 0.16 ppm. IR (neat): 3075, 2950, 2900, 1638, 1600, 1580, 1503, 1438, 1405, 1260, 1250, 1230, 1160, 1100, 1010, 990, 910, 860, 838, 778 cm^{-1}. LRMS (EI, 70 eV): 234 (M^+), 219 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{14}H_{19}FSi$, 234.1240; found, 234.1212.

2e. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34 (ddd, 2H, $J = 8.8$, 2.4, 2.4 Hz), 7.24 (ddd, 2H, $J = 8.8$, 2.4, 2.4 Hz), 5.76 (ddt, 1H, $J = 17.2$, 10.2, 6.0 Hz), 5.03 (ddt, 1H, $J = 17.2$, 1.6, 1.6 Hz), 4.99 (ddt, 1H, $J = 10.2$, 1.6, 1.6 Hz), 3.35 (ddd, 2H, $J = 6.0$, 1.6, 1.6 Hz), 0.19 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 152.6, 141.6, 136.2, 133.0, 130.1, 128.2, 127.6, 116.4, 38.6, 0.15 ppm. IR (neat): 3020, 2950, 2900, 1638, 1593, 1560, 1490, 1440, 1260, 1250, 1100, 1015, 995, 920, 862, 840, 778 cm^{-1}. LRMS (EI, 70 eV): 250 (M^+), 235 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{14}H_{19}ClSi$, 250.0945; found, 250.0904.

2f. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.66 (m, 1H), 7.60–7.58 (a set of two multiplets, 1H), 7.50–7.48 (a set of two multiplets, 1H), 7.43–7.38 (a set of three multiplets, 1H), 5.99 (s, 1H), 5.77 (ddt, 1H, $J = 17.3$, 10.2, 6.0 Hz), 5.05 (ddt, 1H, $J = 17.3$, 1.7, 1.7 Hz), 5.01 (ddt, 1H, $J = 10.2$, 1.7, 1.7 Hz), 3.39 (ddd, 2H, $J = 6.0$, 1.7, 1.7 Hz), 0.21 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 152.5, 144.1, 135.9, 131.6, 130.5 (q, $J = 31.9$ Hz), 129.5, 128.5, 124.2 (q, $J = 271.5$ Hz), 123.9, 123.0, 116.6, 38.6, 0.08 ppm. IR (neat): 3080, 2950, 2900, 1640, 1600, 1580, 1485, 1430, 1335, 1250, 1170, 1130, 1100, 1080, 995, 915, 860, 840, 800 cm^{-1}. LRMS (EI, 70 eV): 284 (M^+), 269 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{15}H_{19}F_3Si$, 284.1208; found, 284.1216.

2g. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.58 (ddd, 2H, $J = 8.4$, 2.0, 2.0 Hz), 7.51 (ddd, 2H, $J = 8.4$, 2.0, 2.0 Hz), 6.06 (s, 1H), 5.77 (ddt, 1H, $J = 17.2$, 10.2, 6.0 Hz), 5.03 (ddt, 1H, $J = 17.2$, 2.0, 1.8 Hz), 4.99 (ddt, 1H, $J = 10.2$, 2.0, 1.8 Hz), 3.39 (ddd, 2H, $J = 6.0$, 2.0, 1.8 Hz), 0.21 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 152.0, 147.7, 135.7, 133.4, 131.9, 126.9, 119.0, 116.8, 110.6, 38.2, −0.05 ppm. IR (neat): 3050, 2950, 2220, 1600, 1500, 1435, 1400, 1250, 990, 915, 860, 850, 840, 770, 690 cm^{-1}. LRMS (EI, 70 eV): 241 (M^+), 226 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{15}H_{19}NSi$, 241.1287; found, 241.1247.

2h. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.97 (ddd, 2H, $J = 8.7$, 2.0, 2.0 Hz), 7.48 (ddd, 2H, $J = 8.7$, 2.0, 2.0 Hz), 6.04 (s, 1H), 5.77 (ddt, 1H, $J = 17.2$, 10.2, 6.0 Hz), 5.03 (ddt, 1H, $J = 17.2$, 2.0, 2.0 Hz), 4.99 (ddt, 1H, $J = 10.2$, 2.0, 2.0 Hz), 4.37 (q, 2H, $J = 7.1$ Hz), 3.39 (ddd, 2H, $J = 6.0$, 1.6, 1.6 Hz), 1.39 (t, 3H, $J = 7.1$ Hz), 0.21 (s, 9H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.5, 153.1, 147.7, 136.0, 131.9, 129.4, 129.1, 126.2, 116.4, 60.8, 38.5, 14.3, 0.1 ppm. IR (neat): 3075, 2950, 2900, 1720, 1600, 1440, 1400, 1260, 1180, 1105, 1020, 910, 860, 840, 800, 765 cm^{-1}. LRMS (EI, 70 eV): 288 (M^+), 273 ($M^+ - 15$). HRMS (EI, 70 eV): calc. for $C_{17}H_{24}O_2Si$, 288.1546; found, 288.1566.

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