

# Formation of 1-(Trimethylsilyl) 1,4-Dienes by Vinylation of Allyltrimethylsilane in the Presence of the Palladium(0)/Silver Salt System

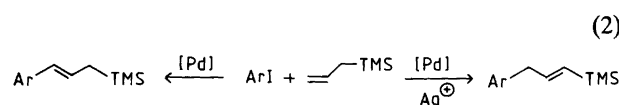
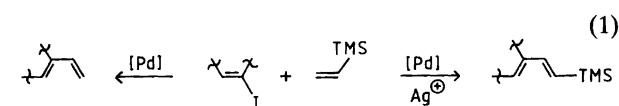
Kostas Karabelas and Anders Hallberg\*

Division of Organic Chemistry 1, Chemical Center, University of Lund, P.O. Box 124, S-221 00 Lund, Sweden

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The major products formed in palladium-catalyzed reactions of allyltrimethylsilane with vinyl iodides, in the presence of silver ion or with vinyl triflates are 1-(trimethylsilyl) 1,4-dienes. In the absence of silver salts, only traces of these dienes are formed, and desilylation is predominant. Addition of silver salts enhances the reaction rate, suppresses the desilylation, and affects the isomer distribution. The isolated yields of the 1-(trimethylsilyl) 1,4-dienes are, in general, low owing to unfavourable regioselectivity in the reactions.

The presence of silver salts in palladium-catalyzed reactions of vinyl iodides,<sup>1</sup> or aryl iodides<sup>2</sup> with trimethyl(vinyl) silane, results in an enhanced reaction rate and suppressed desilylation, furnishing various 1-(trimethylsilyl) 1,3-dienes and (*E*)-trimethyl(2-arylethenyl)silanes, respectively [eqn. (1)]. Furthermore, arylation of allyltrimethylsilane<sup>3</sup> revealed that the isomer distribution was also affected by silver salts, producing 3-(trimethylsilyl)-1-arylpropenes and arylpropenes under ordinary Heck-arylation conditions, while 1-(trimethylsilyl)-3-arylpropenes were formed in the presence of silver ions [eqn. (2)].



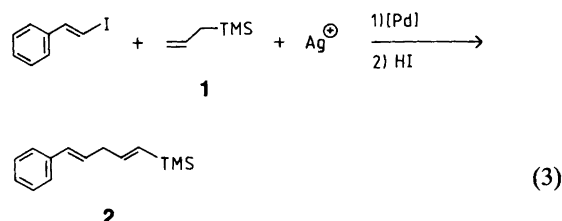
The favourable effect of the addition of silver ion on the reaction rate is even more pronounced in intramolecular cyclization reactions, as has recently been illustrated by Overman and coworkers.<sup>4</sup>

In connection with our earlier studies concerning the palladium-catalyzed substitution of vinyl- and allyl-silanes, we have examined the vinylation of allyltrimethylsilane, with a view to a preparative route to 1-(trimethylsilyl) 1,4-dienes.<sup>5</sup>

\* To whom correspondence should be addressed.

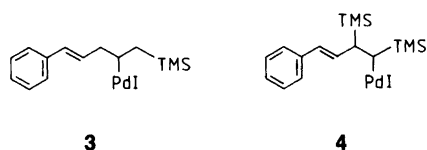
## Results and discussion

An initial experiment, starting from (*E*)-iodostyrene and allyltrimethylsilane (**1**), in dimethyl sulfoxide and in the presence of silver nitrate and triethylamine, using palladium acetate as a catalyst precursor (50 °C, 3 h), gave (1*E*, 4*E*)-5-phenyl-1-(trimethylsilyl)-1,4-pentadiene (**2**) as the major product (GLC 45%), isolated in 25% yield (>95% isomeric purity). The work-up included treatment of the crude reaction mixture with hydroiodic acid,<sup>6</sup> to promote the desilylation of products derived from vinylation at the internal carbon and thus facilitating isolation of **2** [eqn. (3)]. As deduced from NMR/MS a 50/35 ratio of terminal to internal vinylation was obtained.<sup>7</sup> Only traces of desilylated compounds were observed. The product distribution was not affected by the use of triphenylphosphine or tri(*o*-tolyl)phosphine as ligands, or by changing the solvent to acetonitrile, but a slower conversion was achieved in the latter case.

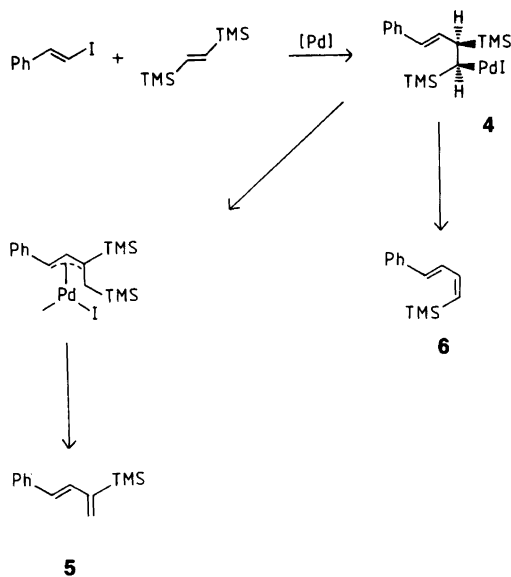


In the absence of silver ions, under ordinary Heck reaction conditions,<sup>8</sup> a complex reaction mixture was formed, containing large amounts of various isomeric arylpenta-dienes according to GLC/MS. Interestingly, only a minor

amount of **2**, among the trimethylsilyl-substituted arylpentadienes formed, was observed. This reaction required a higher reaction temperature (100°C) for full conversion. We assumed that a  $\sigma$ -alkylpalladium intermediate like **3** is important, being a precursor to various desilylated products and to trimethylsilyl-substituted arylpentadienes. In order to study the desilylation process further, we selected a simpler system and treated (*E*)-iodostyrene with (*E*)-1,2-bis(trimethylsilyl)ethylene in the absence of silver ion. Our purpose was, in particular, to see whether intermediate **4** tends to desilylate directly, or undergo desilylation after [HPdI] elimination/readdition via a  $\pi$ -(allyl)palladium complex.

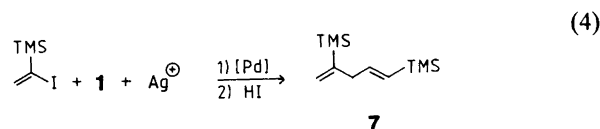


A 52% yield of a 4:1 mixture of **5** and **6** was obtained. We anticipate that the formation of **5** and **6** occurs via **4**, as depicted in Scheme 1. We recently found that arylation of (*E*)-1,2-bis(trimethylsilyl)ethylene, to furnish (*Z*)-trimethyl(2-arylethenyl)silanes occurs via a [PdI/SiMe<sub>3</sub>] *syn*-elimination process,<sup>9</sup> and consequently **6** was expected to be the major product. Apparently, the first step, [HPdI] elimination/readdition to form a  $\pi$ -(allyl)palladium complex is, in this case, favoured over [PdI/SiMe<sub>3</sub>] *syn*-elimination.

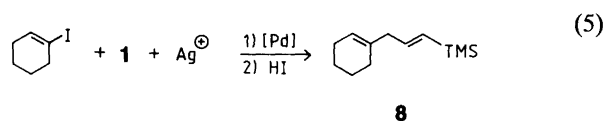


Scheme 1.

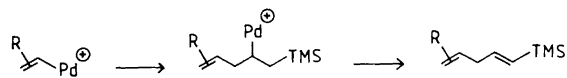
Attempts to prepare (*E*)-1,4-bis(trimethylsilyl)-1,4-pentadiene **7** from 1-iodoethenyl(trimethyl)silane in the presence of silver ion, resulted in the isolation of only a 17% yield (>96% purity) of **7** [eqn. (4)]. Also in this case, the limiting factor for the achievement of a good yield was the low regioselectivity obtained. Furthermore, the regiosomers were not easily separated.



Vinylation of trimethyl(vinyl)silane at the terminal position is favoured if a carbon residue is attached to the iodine-bearing carbon of the vinylic agent.<sup>1</sup> A small effect on the regiochemistry was also observed, in the vinylation of **1** with 1-iodocyclohexene, and 30% (*E*)-3-cyclohexen-1-yl-1-(trimethylsilyl)-1-propene (**8**), was isolated (>98% purity). A 57% yield of **8** with an isomeric purity of 90% was obtained [eqn. (5)].



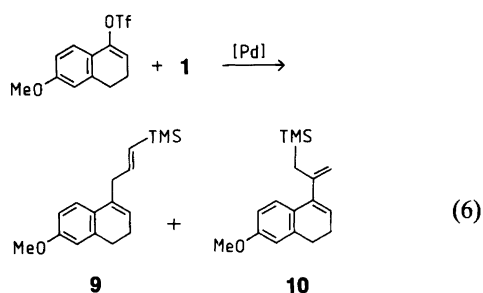
We assume, that in the presence of silver ion, elimination of a [PdH] species proceeds from an initially formed electron deficient  $\sigma$ -vinylpalladium intermediate, and that the trimethylsilyl group may promote the elimination step<sup>3</sup> (Scheme 2). The  $\sigma$ -vinylpalladium intermediate is most likely derived from an oxidative addition of Pd(0) to the vinyl iodide,<sup>10</sup> and a subsequent iodide abstraction by silver ion.<sup>11</sup>



Scheme 2.

Reaction of 7-methoxy-1,2-dihydronaphthalen-4-yl triflate with **1**, provides a 67% yield of a 2:1 (GLC) mixture of **9** and **10** [eqn. (6)]. Only traces of the isomeric 1,3-diene of **9** was formed and we propose that the vinyl triflate<sup>12</sup> and the vinyl iodide/silver ion reactions, proceed via similar intermediates.<sup>1</sup> One desilylated product, namely, 7-methoxy-4-(2-propenyl)-1,2-dihydronaphthalene, was isolated

in 2% yield. The isolated yields of **9** and **10** were 35% (>98% purity) and 18% (95% purity), respectively.



## Conclusion

In conclusion, we have found that 1-(trimethylsilyl) 1,4-dienes are the principal products in Pd(0)-catalyzed vinylation of **1** with vinyl iodides in the presence of silver ions, or as one example demonstrated, with vinyl triflates. Vinyl iodides, in absence of silver ion, furnish a complex mixture of isomeric trimethylsilyl-substituted 1,4-dienes and unsubstituted 1,4-dienes. Addition of silver salts (a) increases the reaction rate (b) suppresses the desilylation and (c) promotes formation of the 1,4-dienes. Unfortunately, the isolated yields in the reactions described here, are low and, therefore, the synthetic utility seems limited. The major reasons for this is the low regioselectivity in combination with difficulties in the chromatographic separations.

## Experimental

**Materials: vinyl iodides.** The following vinyl iodides were prepared according to literature methods: (*E*)- $\beta$ -iodostyrene;<sup>13</sup> 1-iodocyclohexene;<sup>14</sup> and 1-iodoethenyl(trimethyl)silane.<sup>1</sup>

**Silanes.** Allyltrimethylsilane (Aldrich) was used as received. (*E*)-1,2-Bis(trimethylsilyl)ethylene<sup>15</sup> was prepared by treatment of (*E*)-2-bromoethenyl(trimethyl)silane (Fluka) with *t*-BuLi in THF, and subsequent quenching with chlorotrimethylsilane.

**Other reagents.** Palladium acetate (Fluka), silver nitrate (Merck), triphenylphosphine (Fluka), triethylamine (Merck), dimethyl sulfoxide (Riedel), acetonitrile (Janssen), dimethylformamide (Janssen) and hydroiodic acid (67%) (Merck) were used as received from the commercial sources indicated. Tri(*o*-tolyl)phosphine,<sup>16</sup> and 7-methoxy-1,2-dihydronaphthalen-4-yl triflate<sup>17</sup> were prepared according to literature methods.

**General procedures.** Mass spectra were obtained on a Finnigan 4021 (Data System Incos 2100) gas chromatograph-mass spectrometer, operating at 70 eV. <sup>1</sup>H NMR spectra were recorded on a Varian XL-300 spectrometer in deuteriochloroform. Chemical shifts are given relative to internal

tetramethylsilane. Elemental analyses were obtained from *Dornis u. Kolbe Mikroanalytisches Laboratorium*, Mülheim, West Germany. Quantitative gas chromatographic analyses were performed on a Varian 3700 instrument, equipped with a (2.5 m  $\times$  2 mm) glass column of 5% OV 17 on Chromosorb W. A Varian 3400 instrument was used for capillary gas chromatography on a OV 1701 (25 m  $\times$  0.25 mm) column. For column chromatography silica gel 60 (0.040–0.063 mm, Merck) was used. HPLC separations were performed on a LDC Consta Metric III system, equipped with an RI-detector (LKB 2142 refractive index detector). A Nucleosil silica gel column (500 mm), eluted with heptane/ethyl acetate (95:5) and 1% 2-propanol, was used for the purification of **2**. Purification of **7** and **8**, and separation of **9** and **10** were performed on a RP Polygosil C<sub>18</sub> column (500 mm), using acetonitrile/water (95:5) as the mobile phase. Acetonitrile/water (85:15) was used as the eluent for the separation of **5** and **6**.

**General procedure for the reaction of vinyl iodides with allyl(trimethyl)silane.** Each of the reactants was dissolved or dispersed in dimethyl sulfoxide (a total of 50 ml) and was added to a round-bottomed flask in the following order: palladium acetate (0.15 mmol, 33.7 mg); vinyl iodide (5.0 mmol); silver nitrate (5.0 mmol, 0.85 g); triethylamine (6.0 mmol, 0.61 g); and allyltrimethylsilane (15 mmol, 1.7 g). The contents were heated at 50°C and magnetically stirred for 3 h. The mixture was filtered and 0.5 ml of hydroiodic acid (67%) were added.<sup>6</sup> The solution was stirred over night at room temperature, and thereafter poured into 50 ml of water. After extraction with ether or pentane, the combined organic phases were washed with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (10%) and water, dried (MgSO<sub>4</sub>) and evaporated. The residue was dissolved in pentane, filtered through a short column of silica gel and concentrated. The crude product was subjected to HPLC.

**Reaction of 7-methoxy-1,2-dihydronaphthalen-4-yl triflate with allyl(trimethyl)silane.** A mixture of palladium acetate (0.13 mmol, 29.2 mg), 7-methoxy-1,2-dihydronaphthalen-4-yl triflate (4.2 mmol, 1.3 g), allyl(trimethyl)silane (13 mmol, 1.5 g), triethylamine (13 mmol, 1.3 g) and 40 ml of dimethyl sulfoxide was added to a round-bottomed flask and stirred at 50°C for 3 h. The mixture was poured into 40 ml of water and extracted with pentane (4  $\times$  25 ml). The combined organic phases were washed with water (10 ml), dried (MgSO<sub>4</sub>) and evaporated. The residue was dissolved in pentane and filtered through a short column of silica gel, to give 760 mg (67%) of a mixture consisting of **9** and **10**, with a ratio of 2:1 (GLC), and a small amount of 7-methoxy-4-(2-propenyl)-1,2-dihydronaphthalene. Separation by HPLC gave 400 mg (35%) of **9** with a purity of >98%, and 205 mg (18%) of **10** (95% purity). 7-Methoxy-4-(2-propenyl)-1,2-dihydronaphthalene was isolated in 2% yield. <sup>1</sup>H NMR for compound **9**:  $\delta$  0.05 (s, 9 H), 2.25–2.30 (m, 2 H), 2.74 (t, *J* = 7.1 Hz, 2 H), 3.22 (dt, *J* = 6.0, 1.4 Hz, 2 H), 3.81 (s, 3 H), 5.71–5.74 (m, 1 H), 5.77 (dt, *J* = 18.5, 1.5

Hz, 1 H), 6.13 (dt,  $J = 18.5$ , 6.0 Hz, 1 H), 6.68–7.18 (m, 3 H). Irradiation at 3.2 ppm gave a triplet at 5.72 ppm ( $J = 4.6$  Hz), a doublet at 5.76 ppm ( $J = 18.5$  Hz) and a doublet at 6.12 ppm ( $J = 18.5$  Hz). MS:  $m/z$  (rel. intensity) 272 ( $M^+$ , 15), 257 (8), 199 (20), 174 (50), 159 (30), 144 (12), 129 (12), 115 (13), 73 (100), 59 (60), 45 (30). Anal: Calc. for  $C_{17}H_{24}OSi$ : C, 74.9; H, 8.88; Found C, 75.1; H, 8.50.  $^1H$  NMR for compound **10**:  $\delta$  0.05 (s, 9 H), 1.80 (d,  $J = 1.0$  Hz, 2 H), 2.20–2.27 (dt,  $J = 7.5$ , 4.7 Hz, 2 H), 2.70 (t,  $J = 7.5$  Hz, 2 H), 3.81 (s, 3 H), 4.83 (dt,  $J = 2.6$ , 1.0 Hz, 1 H), 4.91 (d,  $J = 2.6$  Hz, 1 H), 5.85 (t,  $J = 4.7$  Hz, 1 H), 6.69–7.17 (m, 3 H). Irradiation at 1.8 ppm gave a doublet at 4.83 ppm ( $J = 2.6$  Hz). MS:  $m/z$  (rel. intensity) 272 ( $M^+$ , 7), 257 (5), 199 (7), 185 (20), 167 (9), 115 (5), 73 (100), 59 (12), and 45 (25). Anal: Calc. for  $C_{17}H_{24}OSi$ : C, 74.9; H, 8.88; Found C, 74.8; H, 8.73. The  $^1H$  NMR spectrum of 7-methoxy-4-(2-propenyl)-1,2-dihydronaphthalene matched the literature spectrum.<sup>18</sup>

*Reaction of (E)- $\beta$ -iodostyrene with (E)-1,2-bis(trimethylsilyl)ethylene.* A mixture of palladium acetate (0.03 mmol, 6.7 mg), (*E*)- $\beta$ -iodostyrene (1.0 mmol, 230 mg), (*E*)-1,2-bis-(trimethylsilyl)ethylene (1.7 mmol, 300 mg), triethylamine (2.0 mmol, 202 mg) and 2 ml of dimethylformamide was added to a 25 ml heavy-walled Pyrex tube. The contents were magnetically stirred and heated at 110 °C for 2 h. The mixture was poured into water, extracted with pentane, dried and evaporated. Column chromatography of the residue (pentane), yielded 105 mg (52 %) of **5** and **6**, as a 4:1 mixture, which was separated by HPLC. The  $^1H$  NMR spectra of **5**<sup>1</sup> and **6**<sup>19</sup> matched the literature spectra. Compound **6** isomerized to (1*E*,3*E*)-4-phenyl-1-(trimethylsilyl)-1,3-butadiene when allowed to stand.

(1*E*,4*E*)-5-Phenyl-1-(trimethylsilyl)-1,4-pentadiene (**2**). The ratio of terminal to internal vinylation was 50:35 (NMR). Two unknown trimethylsilyl-substituted isomers (7 %, GLC) was also formed according to GLC/MS. The total yield of all isomers was 93 % (GLC). After selective desilylation and purification by HPLC, **2** was isolated in 25 % yield (>95 %, isomeric purity).  $^1H$  NMR:  $\delta$  0.06 (s, 9 H), 3.02 (ddt,  $J = 6.7$ , 5.9, 1.4 Hz, 2 H), 5.74 (dt,  $J = 18.6$ , 1.6 Hz, 1 H), 6.10 (dt,  $J = 18.6$ , 5.8 Hz, 1 H), 6.23 (dt,  $J = 15.8$ , 6.7 Hz, 1 H), 6.40 (dt,  $J = 15.8$ , 1.5 Hz, 1 H), 7.18–7.39 (m, 5 H). Irradiation at 3.0 ppm resulted in the following doublets: 5.74 ppm ( $J = 18.5$  Hz), 6.10 ppm ( $J = 18.5$  Hz), 6.23 ppm ( $J = 15.8$  Hz) and 6.40 ( $J = 15.8$  Hz). MS:  $m/z$  (rel. intensity) 216 ( $M^+$ , 6), 201 (6), 183 (5), 142 (15), 135 (13), 125 (5), 115 (5), 91 (10), 73 (100), 59 (40), and 45 (20). Anal: Calc. for  $C_{14}H_{20}Si$ : C, 77.7; H, 9.32; Found C, 77.2; H, 8.92.

(*E*)-1,4-Bis(trimethylsilyl)-1,4-pentadiene (**7**). Further catalyst (2 mol 4) was added after 1 h, and the reaction was run for additional 2 h. Work-up according to the general procedure, afforded a 17 % yield of **7**.  $^1H$  NMR:  $\delta$  0.05 (s, 9 H), 0.07 (s, 9 H), 2.95 (doublet,  $J = 6.3$  Hz, with a multiplet

structure,  $J = 1.0$ –1.5 Hz, 2 H), 5.36 (dt,  $J = 3.1$ , 1.0 Hz, 1 H), 5.55 (dt,  $J = 3.0$ , 1.5 Hz, 1 H), 5.66 (dt,  $J = 18.5$ , 1.5 Hz, 1 H), 5.98 (dt,  $J = 18.5$ , 6.4 Hz, 1 H). Irradiation at 2.9 ppm gave the following doublets: 5.36 ppm ( $J = 3.1$  Hz), 5.55 ppm ( $J = 3.1$  Hz), 5.66 ppm ( $J = 18.6$  Hz) and 5.98 ppm ( $J = 18.5$  Hz). MS:  $m/z$  (rel. intensity) 212 ( $M^+$ , 4), 197 (4), 138 (5), 124 (20), 109 (40), 83 (7), 73 (100), 59 (15), 45 (30). Anal: Calc. for  $C_{11}H_{24}Si_2$ : C, 62.2; H, 11.4; Found C, 61.9; H, 11.0.

(*E*)-3-Cyclohexen-1-yl-1-(trimethylsilyl)-1-propene (**8**). A 57 % yield of **8** was isolated (90 %, purity) after selective desilylation and filtration through a short silica gel column. Purification by HPLC gave a 30 % yield of **8**, with an isomeric purity of > 98 %.  $^1H$  NMR:  $\delta$  0.05 (s, 9 H), 1.50–1.66 (m, 4 H), 1.85–1.93 (m, 2 H), 1.95–2.04 (m, 2 H), 5.38–5.43 (m, 1 H), 5.64 (dt,  $J = 18.4$ , 1.4 Hz, 1 H), 5.98 (dt,  $J = 18.5$ , 6.4 Hz, 1 H). Irradiation at 2.7 ppm gave a doublet at 5.63 ppm ( $J = 18.4$  Hz) and a doublet at 5.98 ppm ( $J = 18.5$  Hz). MS:  $m/z$  (rel. intensity) 194 ( $M^+$ , 8), 179 (9), 151 (5), 120 (15), 111 (5), 91 (10), 81 (15), 73 (100), 67 (10), 59 (50), 45 (35). Anal: Calc. for  $C_{12}H_{22}Si$ : C, 74.1; H, 11.4; Found C, 73.9; H, 11.1.

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## References

- Karabelas, K. and Hallberg, A. *J. Org. Chem.* 53 (1988) 4909.
- (a) Karabelas, K. and Hallberg, A. *Tetrahedron Lett.* 26 (1985) 3131; (b) Karabelas, K. and Hallberg, A. *J. Org. Chem.* 51 (1986) 5286.
- Karabelas, K., Westerlund, C. and Hallberg, A. *J. Org. Chem.* 50 (1985) 3896.
- (a) Abelman, M. M., Oh, T. and Overman, L. E. *J. Org. Chem.* 52 (1987) 4130; (b) Abelman, M. M. and Overman, L. E. *J. Am. Chem. Soc.* 110 (1988) 2328.
- Alternative methods for preparation of 1-(trimethylsilyl) 1,4-dienes: (a) Molander, G. A. *J. Org. Chem.* 48 (1983) 5411; (b) Yasuda, H., Nishi, T., Lee, K. and Nakamura, A. *Organometallics* 2 (1983) 21; (c) Hsiao, C.-N. and Shechter, H. *Tetrahedron Lett.* 24 (1983) 2371; (d) Cunico, R. F. and Han, Y.-K. *J. Organomet. Chem.* 174 (1979) 247; (e) Miller, R. B. and Reichenbach, T. *Synth. Commun.* 6 (1976) 319; selective methods for the preparation of compounds with trialkylsilyl 1,4-diene fragments: (f) Brook, A. G. and Duff, J. M. *Can. J. Chem.* 51 (1973) 2024; (g) Yusupova, F. G., Gailyunas, G. A., Furley, I. I., Panasenkom A. A., Sheludyakov, V. D., Tolstikov, G. A. and Yurjev, V. P. *J. Organomet. Chem.* 155 (1978) 15; (h) Yur'ev, V. P., Yusupova, F. G., Gailyunas, G. A., Sheludyakov, V. D. and Galkin, E. G. *Zh. Org. Khim.* 14 (1978) 2454; (i) Gailyunas, G. A., Yusupova, F. G., Isaeva, L. S., Peganova, T. A., Kayumov, F. F. and Yur'ev, V. P. *Zh. Obshch. Khim.* 54 (1984) 2269; (j) Oppolzer, W., Burford, S. C. and Marazza, F. *Helv. Chim. Acta* 63 (1980) 555; (k) Dieter, R. K., Lin, Y. J. and Dieter, J. W. *J. Org. Chem.* 49 (1984) 3183; (l) Mitchell, T. N., Wickenkamp, R., Amamria, A., Dicke, R. and Schneider, U. *J. Org. Chem.* 52 (1987) 4868; (m) Echavarren, A. M., Tueting, D. R. and Stille, J. K. *J. Am. Chem. Soc.* 110 (1988) 4039.

6. Koumaglo, K. and Chan, T.-H. *Tetrahedron Lett.* 25 (1984) 717; see also Ref. 3.
7. A similar ratio of regioisomers has been reported for vinylations of 1-hexene with  $\beta$ -methyl-substituted vinylic halides: Heck, R. F. *Acc. Chem. Res.* 12 (1979) 146; see also: Heck, R. F. *Org. React.* 27 (1982) 345.
8. Dieck, H. A. and Heck, R. F. *J. Org. Chem.* 40 (1975) 1083.
9. Karabelas, K. and Hallberg, A. *J. Org. Chem.* 54 (1989) 1773.
10. Palladium(II) is reduced to palladium(0) under the conditions used; see Ref. 7.
11. Abstraction of chloride from a vinylpalladium complex by silver acetate and subsequent reaction with benzene in acetic acid has been reported: Moritani, I., Fujiwara, Y. and Danno, S. *J. Organomet. Chem.* 27 (1971) 279.
12. For palladium-catalyzed coupling of vinyl triflates with olefins: (a) Cacchi, S., Morera, E. and Ortar, G. *Tetrahedron Lett.* 25 (1984) 2271; (b) Scott, W. J., Pena, M. R., Swärd, K., Stoessel, S. J. and Stille, J. K. *J. Org. Chem.* 50 (1985) 2302; (c) Harnisch, W., Morera, E. and Ortar, G. *J. Org. Chem.* 50 (1985) 1990; (d) Cacchi, S., Ciattini, P. G., Morera, E. and Ortar, G. *Tetrahedron Lett.* 28 (1987) 3039; (e) Hirota, K., Kitade, Y., Isobe, Y. and Maki, Y. *Heterocycles* 26 (1987) 355; (f) Pena, M. R. and Stille, J. K. *Tetrahedron Lett.* 28 (1987) 6573.
13. Brown, H. C., Hamaoka, T. and Ravindran, N. *J. Am. Chem. Soc.* 95 (1973) 5786.
14. Pross, A. and Sternhell, S. *Aust. J. Chem.* 23 (1970) 989.
15. Bock, H. and Seidl, H. *J. Organomet. Chem.* 13 (1968) 87.
16. Ziegler, C. B. and Heck, R. F. *J. Org. Chem.* 43 (1978) 2941.
17. Orsini, F. and Pelizzoni, F. *Synth. Commun.* 17 (1987) 1389; for a review on different methods of preparation of triflates: Stang, P. J., Hanack, M. and Subramanian, L. R. *Synthesis* (1982) 85.
18. Baran, J. S., Langford, D. D. and Liang, C. D. *Tetrahedron* (1977) 609.
19. Chan, T.-H. and Li, J.-S. *J. Chem. Soc., Chem. Commun.* (1982) 969.

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