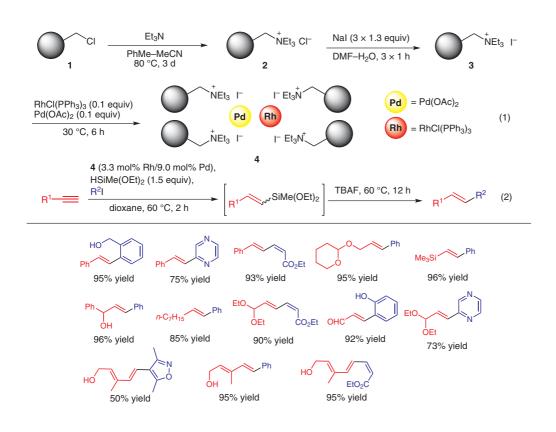
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A One-Pot Synthesis of (*E*)-Disubstituted Alkenes by a Bimetallic [Rh-Pd]-Catalyzed Hydrosilylation/Hiyama Cross-Coupling Sequence

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## [Rh-Pd] Ionic Gel-Soaked Bimetallic Catalyst



**Significance:** The [Rh-Pd] ionic gel-soaked bimetallic catalyst for the one-pot hydrosilylation/Hiyama cross-coupling reactions was described. Thus, the reaction of Merrifield resin  $\bf 1$  with Et<sub>3</sub>N, followed by the anion exchange of  $\bf 2$  with Nal afforded the polyionic iodide gel  $\bf 3$ . The polyionic iodide gel  $\bf 3$  was treated with RhCl(PPh<sub>3</sub>)<sub>3</sub> and Pd(OAc)<sub>2</sub> to provide the [Rh-Pd] bimetallic catalyst  $\bf 4$ . The one-pot synthesis of E-disubstituted alkene via hydrosilylation/Hiyama cross-coupling reaction was carried out in dioxane in the presence of  $\bf 4$  and subsequent addition of TBAF to give the corresponding E-alkenes in 50–95% yield.

**Comment:** It is noteworthy that the Sonogashira coupling side products were not obtained, even without sequential addition of coupling partners in the present procedure. The high chemoselectivity is attributed to a slower Sonogashira coupling in polyionic gel in the absence of copper co-catalysts. For the reaction of phenyl acetylene with phenyl iodide, the catalyst can be recycled three times without loss of the catalytic activity and stereoselectivity (>99% yield and E/Z > 99:1 for 1st-3rd cycles); however, the product yield decreased in the 4th cycle (78% yield and E/Z > 99:1).

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