

Supplementary Information for

Tandem deuteration/hydrosilylation reactions catalyzed by a rhodium carbene complex under solvent-free conditions

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Experimental:

General:

NMR spectra were obtained in benzene- d_6 on Bruker 400 or 300 MHz instruments. The Rhodium catalyst was prepared as previously described and handled under argon. All other reagents were purchased, and used as received. Spectra of the received substrates are presented with the resultant substrate/products mixture spectra.

The complexes made by this new method have been previously prepared and are reported in the following papers.

- 1) Diez-Gonzalez, S.; Scott, N. M.; Nolan, S. P.; *Organometallics*, **2006**, 25, 2355
- 2) Diez-Gonzalez, S.; Kaur, H.; Zinn, F. K.; Stevens, E. D.; Nolan, S. P. *J. Org. Chem.* **2005**, 70, 4784.

Typical procedure:

In an argon atmosphere glovebox $[\text{Rh}(\text{t}^{\text{Bu}})_2\text{Cl}]$ (3.1 mg, 6.2 μmol) were loaded into a screw cap Schlenk flask. Outside of the glovebox, 4-methylacetophenone (87.1 mg, 650 μmol) and a slight excess of triethylsilane (100 mg) were mixed. Under a flow of hydrogen the substrates were added to the catalyst. The vessel was sealed and heated to 50 $^\circ\text{C}$ for 1 hour. Following this time, the vessel was placed under vacuum to remove triethylsilane, leaving behind a mixture of the ketone and resulting silyl-ether. The mixture was dissolved in benzene- d_6 . The ratio of the mixture was determined by ^1H NMR spectroscopy: See Below.



















