

A Novel Utilization of Water Extract of Suaeda Salsa in the Pd/C Catalyzed Suzuki-Miyaura Coupling Reaction

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1. Materials, Methods and Experimental Procedure

1.1 General remarks

All commercially available reagents (from Acros, Aldrich, Fluka, Energy Chemical) were used without further purification. 10 wt. %Pd/C was purchased from Energy Chemical (Palladium 10% on Carbon, ca. 50% water). All reactions were carried out under air. NMR spectra were recorded on a Bruker Advance II 400 spectrometer using TMS as internal standard (400 MHz for ¹H NMR and ¹³C NMR). The isolated yield of products was obtained by short chromatography on a silica gel (200-300 mesh) column using petroleum ether (60-90 °C) and ethyl acetate, unless otherwise noted.

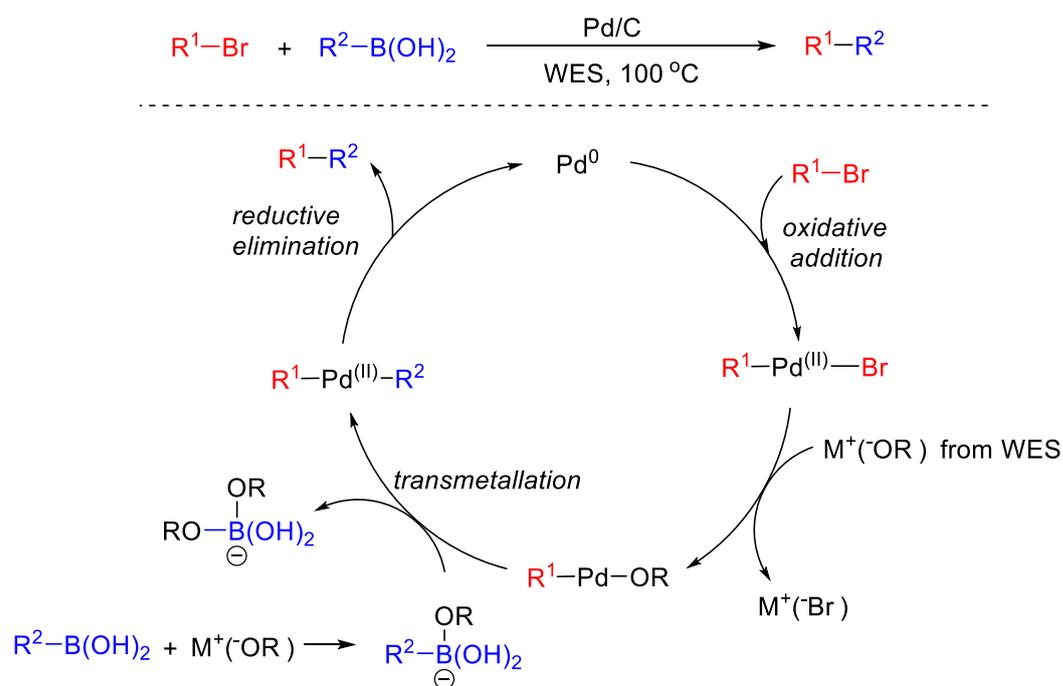
1.2 The procedure for preparing water extract of suaeda salsa

The suaeda salsa was dried overnight in an oven at 120 °C, then the dried suaeda salsa was burned to ash. After that, 10 g of the ash was suspended in 100 mL distilled water and stirred for 2 hours at 100 °C. The suspension was filtered, and the light yellow colored solution was next concentrated to give a yellow solid substance which was used to prepare suaeda salsa aqueous solution with a concentration of 23 mg/mL for Suzuki cross-coupling reactions.

1.3 General procedure for the synthesis of biaryl and heteroaryl compounds.

A mixture of aryl halide (0.5 mmol), arylboronic acid (0.525 mmol), 10 wt.% Pd/C (0.2-1 mol%), WES solution (23 mg/mL, 3 mL) was stirred at 100 °C under air for the indicated time. After reaction, the mixture was cooled to room temperature. [a] The mixture was filtered and washed by water (10 mL) for three times. The residue was dissolved in ethyl acetate (20 mL) and then filtered to remove the palladium carbon. Then the ethyl acetate was collected and concentrated in vacuo to give the pure product. [b] The mixture was concentrated in vacuo and the product was isolated by short chromatography on a silica gel (200-300 mesh) column using petroleum ether and ethyl acetate.

2. Plausible Mechanism for the Suzuki-Miyaura Reaction in WES



Scheme S1. Plausible mechanism for the Suzuki-Miyaura Reaction in WES

3. NMR Spectra for Cross-Coupling Products

