

Hydrogenation and Dehalogenation under Aqueous Conditions with an Amphiphilic Polymer-Supported Nanopalladium Catalyst

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Supporting Information

General: ARP-Pd was prepared according to the procedure previously reported by the authors. All starting materials and authentic samples of the products were purchased from Aldrich Chemical Co. Inc. and Tokyo Kasei Co. Ltd., and used without further purification. All catalytic reactions were carried out with shaking on a wrist-action shaker (Burrel Scientific Inc.) or a Peti-Syzer (Hi-Pep Co. Ltd.). GC-MS analysis was performed on a Hewlett Packard HP 6890 with a 5973 Network Mass Detector. All reaction products were identified by comparison with their GC-MS spectra versus authentic data.

General Procedure for Hydrogenation: A typical procedure for the reaction of *trans*-cinnamyl alcohol in the presence of ARP-Pd in water to give 3-phenyl-1-propanol (Table 1, run 7) is as follows: A mixture of *trans*-cinnamyl alcohol (0.5 mmol), ARP-Pd (65 mg, 26 μ mol of Pd) in 1.0 mL of H₂O was shaken under a hydrogen atmosphere (1 atm) at 25 °C for 24 h. The reaction mixture was filtered and the resin beads were rinsed with ether (4 times x 5 mL). The combined ethereal washings were dried over Na₂SO₄ (100% conversion; >99% GC yield). The crude product was chromatographed on silica gel to give 81% isolated yield of 3-phenyl-1-propanol. The GC-MS spectrum of the product showed good similarity (>95%) with the authentic data.

General Procedure for Hydrodechlorination: A typical procedure for the reaction of methyl 4-chlorobenzoate in the presence of ARP-Pd to give benzoic acid is as follows: To a mixture of ARP-Pd (60 mg, 24 μ mol Pd) and methyl 4-chlorobenzoate (85 mg, 0.5 mmol) in 10% aqueous isopropanol (1.0 mL) was added an aqueous solution of ammonium formate (63 mg, 1.0 mmol in 0.2 mL of H₂O) at 25 °C. The mixture was stirred at 25 °C for 120 min. The reaction mixture was filtered and the catalyst beads were extracted with ethyl acetate (5 mL, 3 times). The combined extract was washed with brine and dried over Na₂SO₄ (100% conversion; >99% GC yield). The crude product was chromatographed on silica gel to give 94% isolated yield of methyl benzoate. The GC-MS spectrum of the product showed good similarity (>95%) with the authentic data.

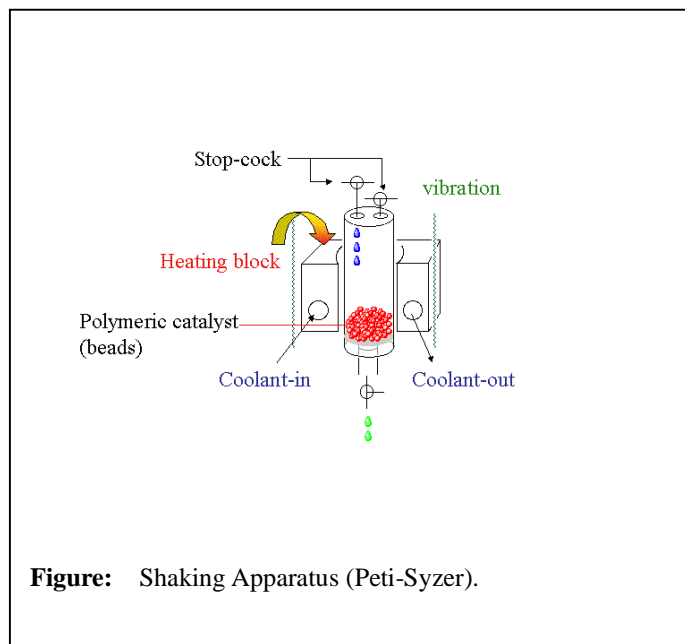


Figure: Shaking Apparatus (Peti-Syzer).