ELECTRONIC SUPPLEMENTARY INFORMATION

Diastereoselective liquid assisted grinding: 'Cracking' functional resins to advance chromatography-free synthesis

William C. Shearouse, James Mack*

**Corresponding author*

University of Cincinnati, 301 Clifton Court, Cincinnati, OH, USA

E-mail: james.mack@uc.edu

Fax: +1 513 556 9239, Tel: +1 513 556 9249

Contents:

General Information:

^{1}H-NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. Deuterated NMR solvents were obtained from Cambridge Isotope Laboratories, Inc., Andover MA, and used without further purification. Starting aldehydes and alkyl halides were purchased from Acros® Organics and used without further purification. Triphenyl phosphine functionalized polystyrene (2% cross-linked with DVB, 100 μm diameter, ~2.24 mmol/g loading) was donated by Biotage Ltd.. Milling balls (5 mm) were purchased from McMaster-Carr Industrial Supply Co.. Ball milling was carried out in a Spex8000M Mixer/Mill purchased from SpexCertiprep.
Digital microscope images were captured using a Keyance VHX-S50 digital microscope with a VH-220R lens.

Experimental Conditions:

A customized 3.0 mL stainless steel vial was charged with 500 mg (2.24 mmol/g) of triphenylphosphine-functionalized polystyrene (2% cross-linked with DVB, 100 μm diameter), 0.680 mmol of aldehyde, 0.975 mmol of alkyl halide, and 1.30 mmol of potassium carbonate. A 5 mm milling ball was added to the vial with 1 mL of 95% ethanol. The vial was shaken at 18 Hz for 1-2 hours in a Spex8000M mixer mill. The resulting mixture was suspended in approximately 20 mL of ethyl acetate, filtered, and the solvent was removed under reduced pressure affording the pure olefin product. ^{1}H-NMR and GC-MS were performed to assess the extent and purity of the reaction products.