Mitsunobu general procedure:

Add a solution of the phenol to the PS-Triphenylphosphine resin and allow the suspension to stand for 5 min. Add a solution of DBAD (di-tert-butyl azodicarboxylate) and THF to this suspension and agitate for 30 min at RT. Add a solution of the alcohol to this mixture and stir the reaction overnight. To scavenge any excess phenols, add MP-Carbonate resin and stir for 2 h. Filter and wash with THF. Add TFA/DCM/water (50:48:2) to this solution and stir the mixture at RT for 2 h. Extract the product with MTBE, wash with water and concentrate the MTBE layer to obtain the product.

Specifications

Chemical Name: Diphénylphosphino-polystyrene
Resin Type: 1% Cross-linked poly(styrene-co-divinylbenzene)
Loading: Typical loading 2.2 mmol/g, minimum loading 1.8 mmol/g (based on uptake of benzyl bromide)
Bead Size: 75–150 microns, 100–200 mesh (95% within)
Application: Chlorination of acids and alcohols, Wittig and Mitsunobu reactions, scavenging of alkyl halides

PS-Triphenylphosphine is a resin-bound equivalent of triphenylphosphine. The capacity of the resin is determined by the quantitation of benzyl bromide uptake in DMF (GC, internal standard method).

PS-Triphenylphosphine can be used in Mitsunobu reactions to prepare aryl ethers in good-to-excellent yields and in high purities (Scheme 1).¹

Scheme 1. Mitsunobu reaction using PS-Triphenylphosphine

Mitsunobu general procedure: Add a solution of the phenol to the PS-Triphenylphosphine resin and allow the suspension to stand for 5 min. Add a solution of DBAD (di-tert-butyl azodicarboxylate) and THF to this suspension and agitate for 30 min at RT. Add a solution of the alcohol to this mixture and stir the reaction overnight. To scavenge any excess phenols, add MP-Carbonate resin and stir for 2 h. Filter and wash with THF. Add TFA/DCM/water (50:48:2) to this solution and stir the mixture at RT for 2 h. Extract the product with MTBE, wash with water and concentrate the MTBE layer to obtain the product.

PS-Triphenylphosphine resin has also been used in the Wittig reaction to synthesize olefins conventionally²,³ and via microwave heating⁴ (Scheme 2). Microwave heating significantly enhances the reaction rate.

Scheme 2: Wittig reaction A: Traditional two step B: Microwave assisted one pot
**Wittig general procedure:** Add the alkyl halide to a suspension of PS-Triphenylphosphine in DMF and stir the reaction mixture for 48 h at 65 °C. Wash the resulting phosphonium resin with DMF, toluene, DCM, and diethyl ether and dry in vacuo for 12 h. Add THF to the dried phosphonium resin in a reaction vessel, followed by the addition of sodium bis(dimethylsilyl)amide in THF at room temperature and stir the reaction for 1 h. Wash the ylide resin with THF to remove excess base. Suspend the ylide resin in anhydrous THF, add a solution of the aldehyde in THF, and stir for 16 h. Dilute the reaction mixture with hexanes and apply to a silica SPE cartridge followed by washing with hexane/ether (2:1). Concentrate the solvent to obtain the olefin.

PS-Triphenylphosphine can be used as a scavenger of alkyl halides. To scavenge alkyl halides stir the resin (3 equiv) with the alkyl halide in DMF (10mL/g of resin) at RT for 5-16 h or via microwave heating and filter.

The resin has also been used to convert alcohols and carboxylic acids to their corresponding chlorides using carbon tetrachloride\textsuperscript{5-7} or trichloroacetonitrile\textsuperscript{8} conventionally and via microwave heating (Scheme 3).

**Chlorination general procedure:** Add a solution of the alcohol or carboxylic acid to a suspension of PS-Triphenylphosphine resin in CCl\textsubscript{4} or trichloroacetonitrile and heat the reaction mixture in an oil bath or in a microwave. Filter and concentrate filtrate to obtain the corresponding chlorides.


**Ordering Information**

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