A FACILE ONE-POT SYNTHESIS OF CHIRAL AMINES ON SILICA

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We have continued our investigation for a greener synthesis of chiral amines on silica surfaces. The synthesis described below is facile, economical, and takes place at ambient temperatures on the surface of activated silica with the addition of only minimum amounts of solvent. Typically, 2 g. of activated silica were introduced into a dry round bottomed flask equipped with a stirring bar and a drying tube. To the flask were added 10-20 mL of anhydrous ether, 1.0×10^{-3} mol of an aldehyde and 1.0×10^{-3} mol of a chiral primary amine. The suspension was stirred for 30 minutes to complete formation of the intermediate chiral imine. To the suspension was then added 0.15 g of Sodium Borohydride and stirred briefly. The flask was cooled in an ice bath, and a few drops of water were added periodically to initiate reduction. The product was filtered, and the silica was washed with three aliquots of 5 mL ether. The ether extracts were combined and dried over anhydrous Sodium Sulfate. After filtration, the ether was removed on a rotary evaporator. The products were purified from trace impurities by column chromatography. Yields were near quantitative. They were analyzed by IR, GC-MS, proton and C-13 NMR, and polarimetry.