

Separation and Recycling of Phosphane Ligands from Homogeneously Catalyzed Processes

Abstract

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The use of chiral transition metal complex catalyzed reactions is well established and highly attractive for the synthesis of optically active products. Diphosphine ligands like Cl-MeO-Biphep are still attracting a great interest because of their exceptional ability to induce asymmetry in numerous reactions.

In this work, the main emphasis was placed on the reuse and recycling of the applied organic ligand Cl-MeO-Biphep for the homogeneously catalyzed asymmetric hydrogenation of ethyl acetoacetate. To reuse this ligand, several different media (cyclohexane, methylcyclohexane and ionic liquid) were tested by applying the same catalyst complex at least in four consecutive runs with high conversion (up to 99 %) and ee (up to 96.2 %), i.e. without loss of catalytic activity.

Derivatization of Cl-MeO-Biphep ligand to its oxide form was also completed in a quantitative yield. Development and optimization of the complete recycling cycle for this ligand have been achieved in 49 % yield and in very pure solid form.

Furthermore, Buchwald-Hartwig amination reaction was also carried out and optimized for Dave-Phos, X-Phos ligands up to 99 % conversion. The recovery of the oxide form of these auxiliaries was also investigated using the above mentioned recycling cycle after the catalytic transformation. In this case, 72 % of X-Phos oxide derivative was obtained in its pure solid form. Since, the Dave-Phos ligand has lower thermal stability its recovery is extremely complicated.

Additional investigations and optimizations are necessary to apply the developed recycling cycle for an industrially-suited process.