Rhodium (I) diphenylphosphine complexes supported on porous organic polymers as efficient and recyclable catalysts for alkene hydrogenation

Cristian H. Campos^{a,*}, Julio B. Belmar^b, Solange E. Jeria^b, Bruno F. Urbano^c, Cecilia C. Torres^d, Joel B. Alderete^b

^aDepartamento de Físico-Química, Facultad de Ciencias Químicas, Universidad de Concepción, Concepción, Chile ^bDepartamento de Química Orgánica, Facultad de Ciencias Químicas, Universidad de Concepción, Concepción, Chile ^cDepartamento de Polímeros, Facultad de Ciencias Químicas, Universidad de Concepción, Concepción, Chile

^{*d}Departamento de Ciencias Químicas, Universidad Andres Bello, Sede Concepción, Talcahuano, Chile.*</sup>

1.2 Synthesis of N,N-bis(2-(diphenylphosphino)ethyl)prop-2-en-1-amine (Alk-L)

The reaction of the N,N-bis(2-chloroethyl)-2-propen-1-amine (0.50 g, 2.75 mmol) with KPPh₂ (5.5 mmol) in 10 mL of degasified THF at -50 °C. The solution was allowed to warm to room temperature overnight. After 20 mL of water was added and the mixture was transferred into a separating funnel and treated with freshly distilled (under dinitrogen) diethyl ether (2 × 50 mL). The collected organic phases were dried over Na₂SO₄, and filtered. The solvent was removed under vacuum to afford a thick oil. Yield: 45%.



Figure S1. NMR characterization for the Alk-L (solvent: CD₂Cl₂). (A) ¹H and (B) ³¹P

1.3 Synthesis of N,N-bis(2-(diphenylphosphino)ethyl)acrylamide (Acy-L)

The precursor intermediate N,N-bis-(2-diphenylphosphinoethyl)amine was prepared and isolated as a crystalline, air-stable, hydrochloride salt by the reported procedure . In a 50-mL flask were placed 0.478 TEA, and 20 mL of degasified CH_2Cl_2 under Ar [1]. To this mixture was added by syringe a solution of 76 µL (1.1 mmol, 83 mg) of acetyl chloride in 3 mL of CH_2Cl_2 , and the reaction was stirred for 10h at ambient temperature. The reaction was worked up by washing first with portions (2 x 10 mL) of 2 molL⁻¹ HCl followed by 0.1 molL⁻¹ NaOH (1 x 10 mL) both degasified. Drying the separated organic phase over Na₂SO₄ and evaporating the solvent under reduced pressure gave clear oil. Yield 0.47 g (97%).



Figure S2. NMR characterization for the Acy-L precursor (solvent: CDCl₃). (A) ¹H and (B) ³¹P



Figure S3. NMR characterization for the Acy-L (solvent: CD₂Cl₂). (A) ¹H and (B) ³¹P

References

[1] M.M.T. Khan, E.R. Rao, Polyhedron 6 (1987) 1727-1735.