

PROCEDURE OF ASYMMETRIC HYDROGENATION USING DIPHOSPHINE-RUTHENIUM COMPLEX

[Procedure]

- (1) A dry, stainless steel autoclave with a Teflon-coated magnetic stirring bar is evacuated and filled with an inert gas (argon or nitrogen) three times.
- (2) The catalyst is placed into the autoclave under an inert gas atmosphere and the evacuation/inert gas refill cycle is repeated three times.
- (3) A dry, Schlenk tube (flask) equipped with a rubber septum and a Teflon-coated magnetic stirring bar is evacuated and filled with an inert gas three times.
- (4) The Schlenk tube (flask) is charged with the substrate, alcohol and water by syringes under slightly positive pressure of an inert gas, and the inlet is sealed by a glass stopper using silicone grease.
- (5) The substrate solution is transferred into the autoclave by a syringe under slightly positive pressure of an inert gas (Figure 1).
- (6) The autoclave is connected to a hydrogen source using the arrangement shown in Figure 2. The gas inlet tube is attached to the autoclave and the main valve of the cylinder is opened. After closing the main valve of the cylinder, the connector of the gas inlet tube is loosened to release hydrogen pressure and tightened immediately. This procedure is repeated five times to replace the air originally present in the gas-inlet tube with hydrogen.
- (7) Hydrogen is introduced into the autoclave until the pressure gauge indicates ca. 0.3 MPa, and the pressure is carefully released to ca. 0.1 MPa. This procedure is repeated three times.
- (8) The main valve of the hydrogen gas is opened and hydrogen is introduced until pressure gauge indicates a specific pressure.
- (9) The valves are closed and the gas inlet tube is disconnected.
- (10) The reaction mixture is stirred at a specific temperature for a specific length of time.
- (11) The autoclave is cooled to ambient temperature and excess hydrogen is carefully released by opening the valve of the autoclave. The apparatus is then disassembled.
- (12) The conversion and enantiomeric excess of the obtained product are determined by gas chromatography (GC) analysis or high-performance liquid chromatography (HPLC) analysis.

FOR MORE DETAILED INFORMATION, PLEASE CHECK THE REFERENCES.

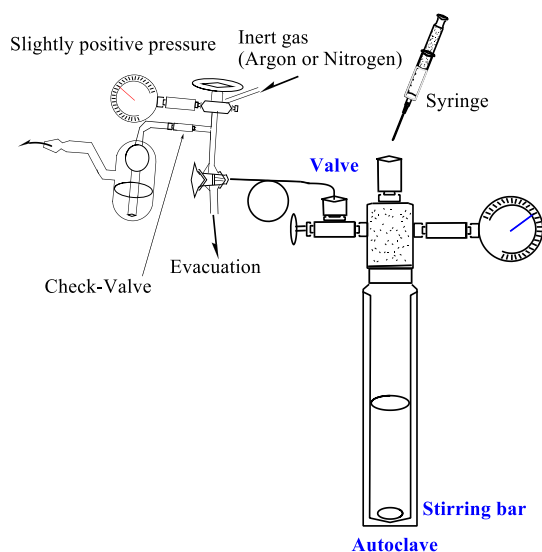


Figure 1. Transferring method.

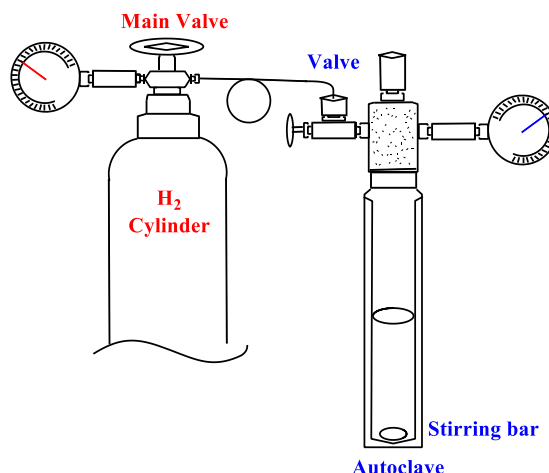


Figure 2. A hydrogenation apparatus.

[Notes]

Guaranteed grade solvent is distilled under an inert gas (argon or nitrogen atmosphere).

A Schlenk tube (flask) is a glass vessel with a sidearm stopcock that allows it to be evacuated or filled with an inert gas.

Caution! Ru complexes are rapidly oxidized in solution in the presence of air and all procedures should be carried out under anaerobic conditions using degassed solvents. These procedures do not constitute a guarantee, warranty, or prediction regarding the outcome of your legal matter.

Please check and follow the high pressure gas safety laws and regulations in each nation and region.

[Waste Disposal Information]

All toxic materials should be disposed of in accordance with "Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards, Updated Version"; National Academy Press; Washington, DC, 2011. doi. [10.17226/12654](https://doi.org/10.17226/12654)

[Reference]

(1) Kitamura, M.; Tokunaga, M.; Ohkuma, T.; Noyori, R. *Org. Synth.*, **1993**, 71, 1. (Published in Collective Volume 9 p. 589) doi. [10.15227/orgsyn.071.0001](https://doi.org/10.15227/orgsyn.071.0001)

(2) Takaya, H.; Ohta, T.; Inoue, S.; Tokunaga, M.; Kitamura, M.; Noyori, R. *Org. Synth.*, **1995**, 72, 74. (Published in Collective Volume 9 p. 169) doi. [10.15227/orgsyn.072.0074](https://doi.org/10.15227/orgsyn.072.0074)

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