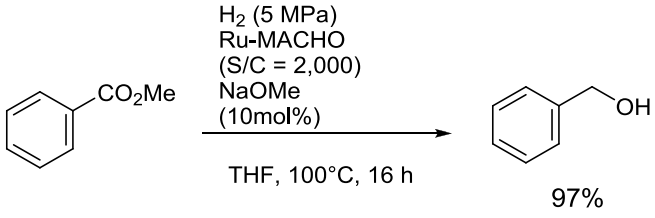
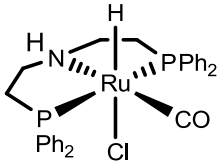
TYPICAL PROCEDURE OF HYDROGENATION USING Ru-MACHO®

Hydrogenation of Methyl Benzoate





Ru-MACHO
$C_{29}H_{30}ClNOP_2Ru$
MW; 607.03

[Procedure]

(1) To a stainless steel autoclave equipped with a Teflon-coated stirring bar, the catalyst (0.005 mmol) and NaOMe [1] (1 mmol) are placed.

(2) The autoclave is evacuated and filled with an inert gas (argon or nitrogen) three times.

(3) The safety relief valve is opened and THF [2](8 mL) and methyl benzoate (10 mmol) are charged by syringe into the autoclave slightly pressurized with an inert gas. Then the valve is closed and the autoclave is filled with an inert gas (Figure 1).

(4) 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 hydrogen gas is opened. The pressure gauge indicates ca. 0.1 MPa. After closing the main valve, 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.

(5) Hydrogen gas 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 from the safety relief valve. This procedure is repeated three times to replace the inert gas with hydrogen gas.

(6) The main valve of the hydrogen gas is opened and hydrogen is introduced until pressure gauge indicates 5MPa.

(7) The valves are closed and the gas inlet tube is disconnected.

(8) The mixture is stirred at 100°C for 16 h.

(9) 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.

(10) The conversion is determined by gas chromatography (GC) analysis.
Neutra Bond-1 (df = 0.40 μm, 0.25 mm i.d. x 30 m, GL-Sciences), Carrier gas: helium, Injection temp.: 200°C, Detector temp.: 280°C, Oven: 40°C → 100°C (5°C/min) → 280°C (10°C/min) –

280°C (5 min hold).

[1] Using NaOMe stored under inert gas or from a freshly opened bottle is recommended.
[2] THF or MeOH is used as the solvent. The better solvent depends on the substrate.

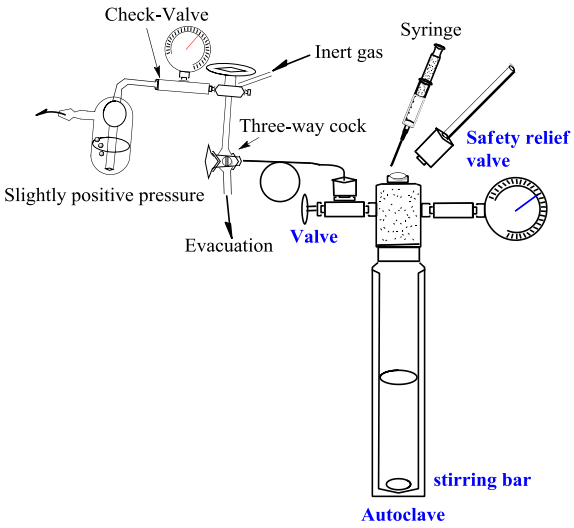


Figure 1. Transferring method.

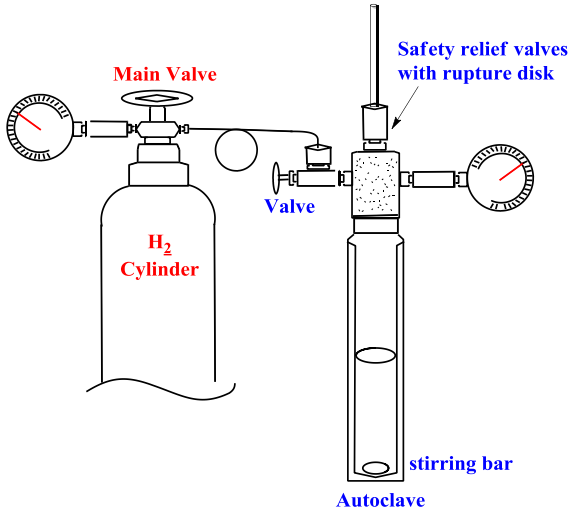


Figure 2. A hydrogenation apparatus.

[Notes]

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

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

Visit Our Web Site !!!

TAKASAGO
finechemicals@takasago.com