

Drying dichloromethane over calcium hydride

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Method Article

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Abstract

This protocol describes the drying of dichloromethane by a simple 10 step procedure. One can implement this protocol using common lab glass and lab equipment. First, dichloromethane is refluxed with calcium hydride to remove water. Then, dichloromethane is distilled to separate it from the byproducts of the reflux reaction. This procedure can be implemented in 1 day.

Introduction

In many instances in synthetic chemistry it may be necessary to remove residual water from solvents in order to carry out a synthesis procedure under inert conditions. In this example, water is removed from dichloromethane by refluxing dichloromethane in the presence of calcium hydride at 60°C. Under these conditions, calcium hydride reacts with water to form calcium hydroxide and hydrogen gas. The hydrogen gas is allowed to escape from the system through a drying tube while calcium hydroxide is separated from the anhydrous dichloromethane by distillation at 80°C in a subsequent step.

Reagents

Calcium hydride, $\geq 95\%$ (Sigma-Aldrich Co., cat. no. 208027-100G) Dichloromethane, $\geq 99.5\%$, ACS certified (EMD, cat. no. DX0835-5)

Equipment

Condenser (Chemglass, cat. no. CG-1218-07) Distillation adapter (Chemglass, cat. no. CG-1024-01) Drying tube, "U" shaped (Chemglass, cat. no. CG-1296-01) Glass thermometer (10/30) (Wilmad LabGlass, cat. no. LG-10515-106) Heating mantle (1000 mL) (Glas-Col, cat. no. 0408) Insulating wool Laboratory clamps Large magnetic stir bars (PTFE) Magnetic stir plate One-necked round-bottomed flask (1 L) One-necked round-bottomed flask (100 mL) Rubber tubing Three-way 120° angle connecting adapter (Corning, cat. no. 9021-24) Transformer (Warner Electric, cat. no. 3PN116C)

Procedure

Drying of dichloromethane TIMING ~3 h 1. Add calcium hydride (20 g) and dichloromethane (700 mL) into a one-necked round-bottomed flask (1 L) and fit the round-bottomed flask with a condenser and drying tube filled with drierite (Figure 1). CAUTION Calcium hydride and dichloromethane are irritating and/or harmful if exposed to the skin or inhaled. Calcium hydride releases flammable gases upon contact with water. Conduct all work in a chemical fume hood. Proper personal protective equipment (lab coat, nitrile gloves, and safety glasses) should be worn throughout the procedure. 2. Stir the mixture using a magnetic stir bar. 3. Heat the reaction flask to 60°C using a heating mantle and reflux at 60°C for 2 h. Under these conditions, calcium hydride reacts with water to form calcium hydroxide and hydrogen gas. The hydrogen gas is allowed to escape from the system through a drying tube. 4. After 2 h, cool the

reaction flask to room temperature (20-25°C). PAUSE POINT Once cooled, the dichloromethane/calcium hydride mixture may be sealed and stored overnight in the fume hood, if necessary. Distillation of dichloromethane TIMING ~6 h 5. Attach the flask from Step 4 to the apparatus shown in Figure 2 with a 100 mL receiving flask. 6. Stir the mixture using a magnetic stir bar and heat the reaction flask to 80°C using a heating mantle. 7. Collect the first 30 mL distillate in the 100 mL receiving flask and then replace with a 1000 mL receiving flask. CRITICAL STEP The first 30 mL distillate may contain residual water and should be discarded. 8. Continue heating until ~600 mL anhydrous dichloromethane has been collected in the receiving flask. 9. Once the desired amount of anhydrous dichloromethane has been collected, cool the reaction flask to room temperature. 10. Once cooled, seal the flask with the remaining mixture from step 1 and store in the fume hood awaiting either reuse of unreacted calcium hydride or disposal.

Figures

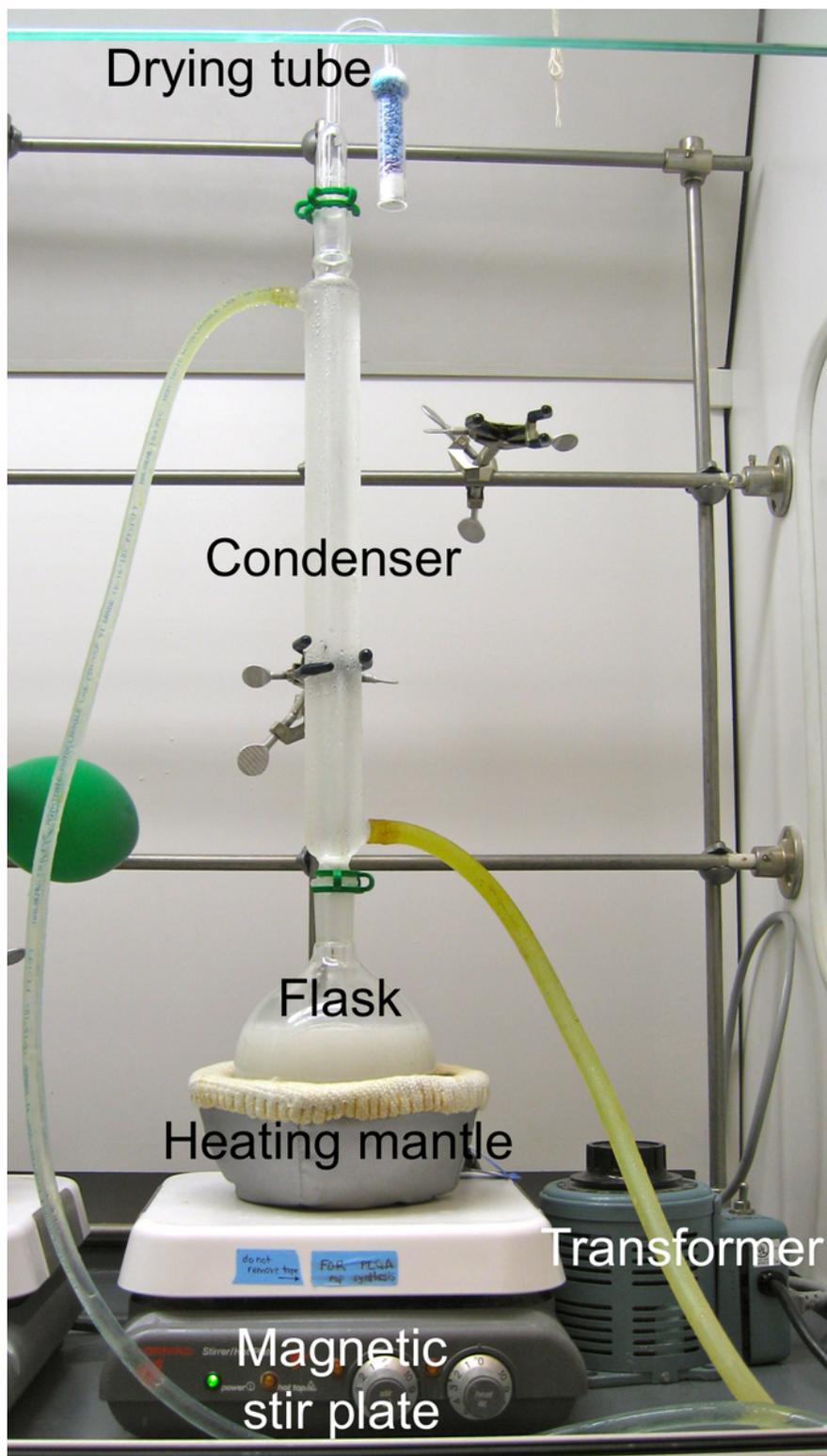


Figure 1

Drying of dichloromethane

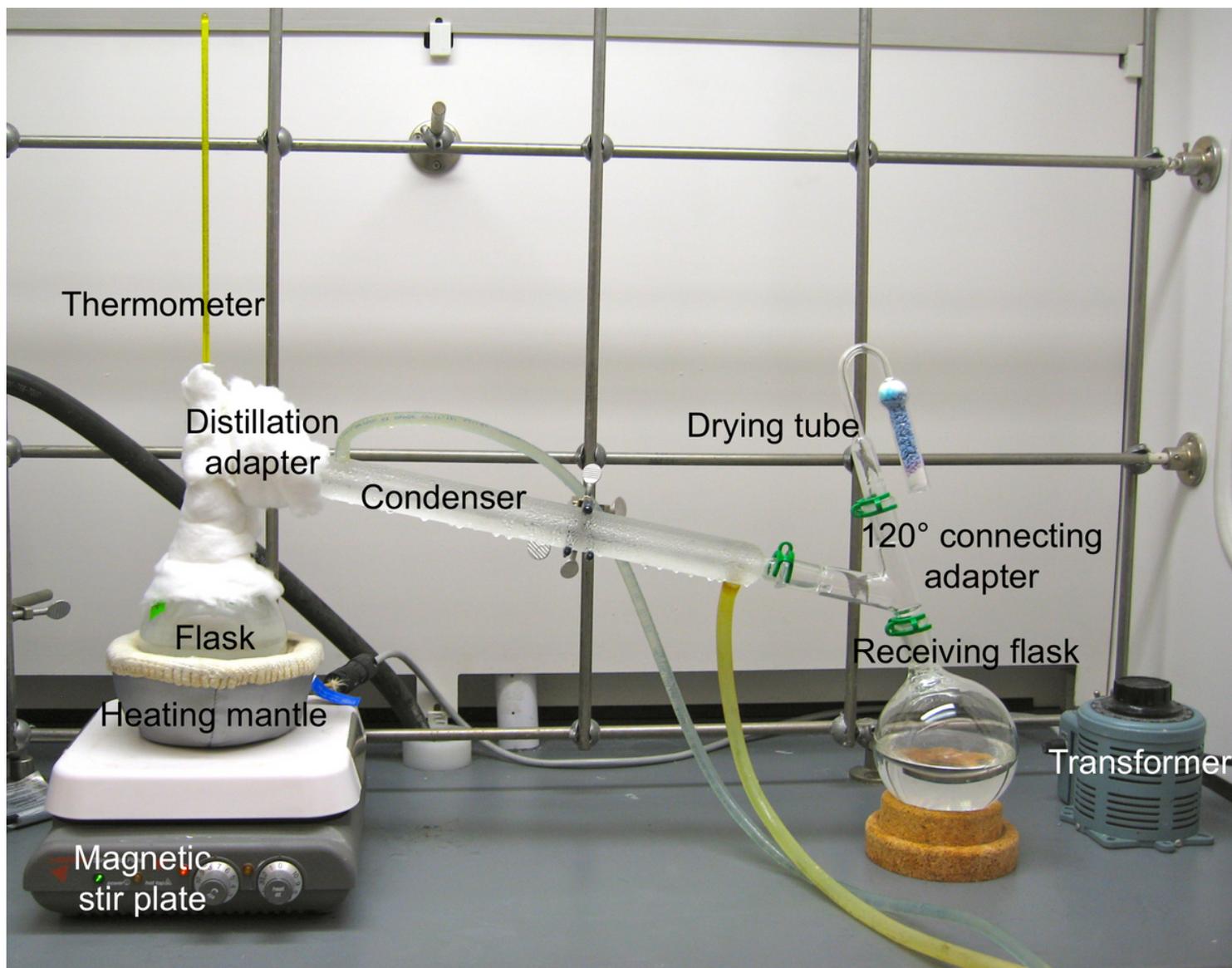


Figure 2

Distillation of dichloromethane