Drying poly(ethylene glycol)

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Method Article

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Abstract

This protocol describes the drying of poly\(ethylene glycol) \(PEG) by a simple 6 step procedure. One can implement this protocol using common lab glass and lab equipment. Water is removed from PEG by azeotropic distillation in toluene. The two components are mixed and toluene and water are distilled off by heating the solution to 170°C. This procedure can be implemented in ~2 h.

Introduction

In many instances in synthetic chemistry it may be necessary to remove residual water from reagents in order to carry out a synthesis procedure under inert conditions. In this example, water is removed from PEG by azeotropic distillation in toluene. The two components are mixed and toluene and water are distilled off by heating the solution to 170°C.

Reagents

Poly\(ethylene glycol) \(Sigma-Aldrich Co., cat. no. P3515-500G \(1 kDa), P4338-500G \(3 kDa), P6667-500G \(10 kDa), 81310-1KG \(35 kDa)) Toluene, ≥99.5%, ACS certified \(Fisher Scientific, cat. no. T324-4)

Equipment

Barrett distilling receiver with stopcock \(20 mL) \(Corning, cat. no. 3622-20) Beaker \(250 mL) Condenser \(Chemglass, cat. no. CG-1218-07) Drying tube, “U” shaped \(Chemglass, cat. no. CG-1296-01) Heating mantle \(1000 mL) \(Glas-Col, cat. no. O408) Insulating wool Laboratory clamps Large magnetic stir bars \(PTFE) Magnetic stir plate One-necked round-bottomed flask \(1 L) Rubber tubing Transformer \(Warner Electric, cat. no. 3PN116C)

Procedure

Drying of PEG TIMING ~2 h 1. Add PEG \(50 g) \(1 kDa, 3 kDa, 10 kDa, or 35 kDa depending on the synthesis plan) and toluene \(200 mL) into a one-necked round-bottomed flask \(1 L) \(Figure 1). CAUTION Toluene is irritating and/or harmful if exposed to the skin or inhaled. 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 170°C using a heating mantle. 4. Measure the distillate using the Barrett distilling receiver and collect the distillate into a 250 mL beaker for disposal. 5. Continue heating until 180 mL distillate has been collected. 6. Once the desired amount of distillate has been collected, cool the reaction flask to room temperature and collect any subsequent distillate. PAUSE POINT Once cooled, the reaction flask may be sealed and stored in the fume hood until the anhydrous PEG is needed.

Figures

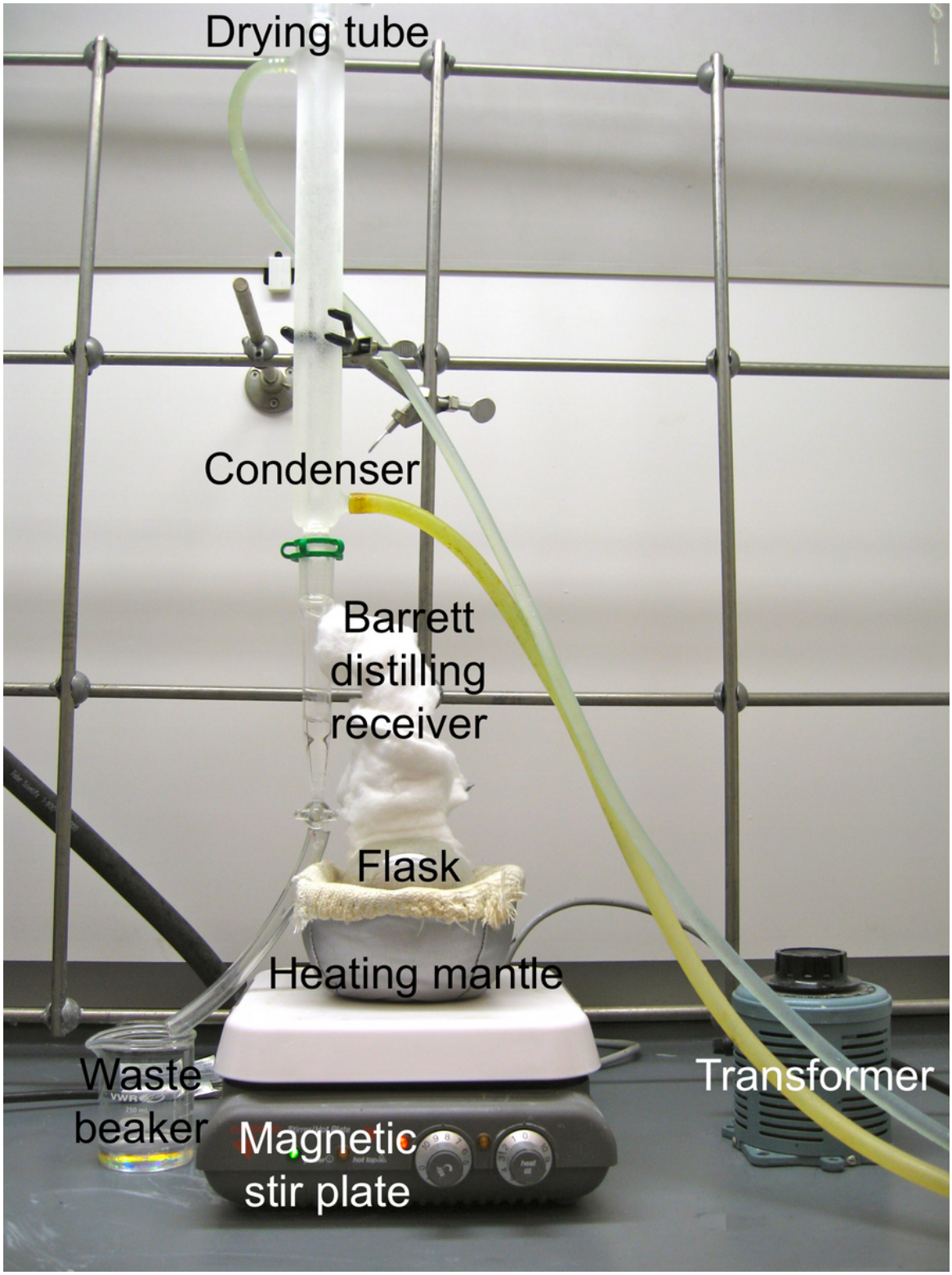

Figure 1

Apparatus for drying poly(ethylene glycol)