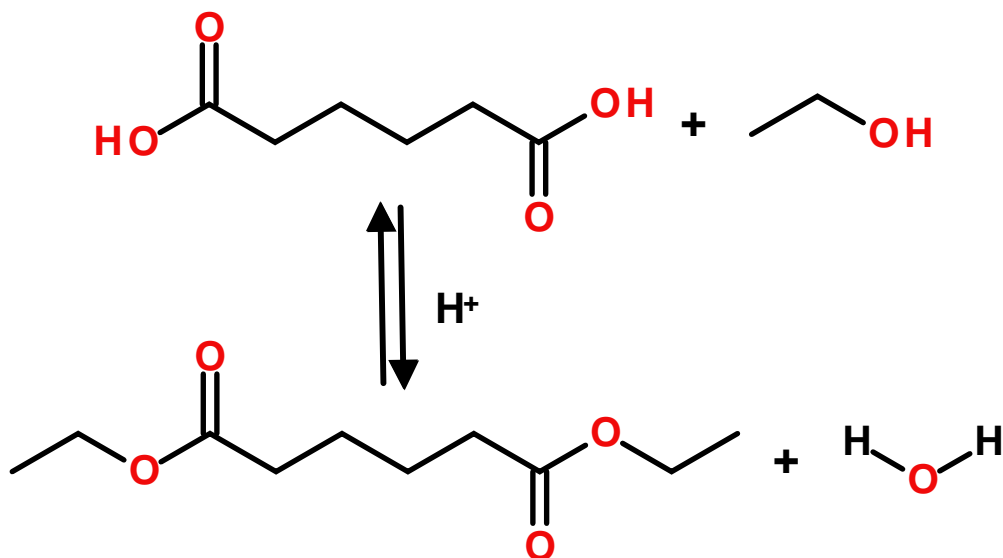


# Synthesis of Diethyl Adipate

## A. Reaction:



## B. Reagents:

Adipic Acid - 43.8 g. (0.30 mol)  
Ethanol (95%) - 100 mL  
Cyclohexane - 30 mL  
Sulfuric Acid (conc.) 1.5 mL

## C. Procedure:

Mix reagents in a 500 mL round bottom flask. Attach the Dean Stark trap and a reflux condenser. Heat the mixture under reflux. The azeotrope, composed of alcohol, water, and cyclohexane, will collect in the trap and can be removed from time to time by means of the stopcock. Continue until no further azeotrope is evolved, which should be about 3-4 hours. Measure the volume of the azeotrope that is formed. From the composition of the azeotrope (see below) determine the amount of water evolved. The composition of the azeotrope (lower layer) in the Dean-Stark trap is (by volume):

Water 14.5 parts  
Ethanol 75.0 parts  
Cyclohexane 9.4 parts

## ***Synthesis of Diethyl Adipate***

Add about an equal amount of ether and extract the mixture three times with aqueous sodium carbonate to remove sulfuric acid and any acidic unreacted adipic acid or any of the half acid ester of adipic acid. Then extract the aqueous extracts with ether to recover any of the diester that may have dissolved in the water layer. Combine the ether extracts and remove dissolved water by drying with solid sodium sulfate or magnesium sulfate until the solution appears clear. Remove the drying agent by suction filtration, wash the drying agent with a small amount of ether, add this to the extracts, and evaporate the ether on the rotary evaporator with heat from the steam bath. Purify the product by vacuum distillation. (The instructors will guide you in this operation.) Be sure to record the boiling point and the pressure. Characterize the product by refractive index (R.I.) and boiling point (b.p.). Compare your values for the b.p. and the R.I. with the literature values.

Calculate the volumes of ethanol and cyclohexane needed to completely convert 43.8 g of adipic acid to diethyl adipate and at the same time remove all of the water from the reaction mixture as part of the lower layer in the Dean Stark trap.

What is the mechanism of this reaction?

What is the purpose of the azeotropic distillation?

Why are ethanol and cyclohexane used in excess?

## Synthesis of Diethyl Adipate

### D. Report: Preparation of Diethyl Adipate

Name, \_\_\_\_\_ Date \_\_\_\_\_ Exp. No. \_\_\_\_\_

Chemical Reaction:

Reagents:

| Compound    | Mol Wt | Density (g/mL) | Wt. (g) | Vol (mL) | mole |
|-------------|--------|----------------|---------|----------|------|
| Adipic Acid |        |                |         |          |      |

Products:

| Compound        | Mol Wt | Density | Wt. (g) Theoretical | mol Theoretical |
|-----------------|--------|---------|---------------------|-----------------|
| Diethyl Adipate |        |         |                     |                 |

Attach IR spectrum. Gas Chromatograms. etc.

IR Bands and Assignments

Yield \_\_\_\_\_ g \_\_\_\_\_ mol

Yield \_\_\_\_\_ %

Evidence for the confirmation of structure.