

Synthesis, Isolation and Purification of an Ester

Inquiry Guidance and AP* Chemistry Curriculum Alignment



Introduction

The study of organic chemistry is organized around functional groups—groups of atoms bonded together in a specific pattern that give organic compounds their unique physical and chemical properties. The ester functional group is widely distributed in nature. Low molecular weight esters are responsible for the pleasant odor or fragrance of many fruits and flowers, and are thus important ingredients in natural and artificial flavors. Ethyl acetate is a simple ester that is used as a flavor aid for pharmaceuticals and artificial fruit essences.

Opportunities for Inquiry

The synthesis of ethyl acetate reinforces knowledge of limiting and excess reactants and demonstrates classic techniques for the separation of a mixture and product purification. These learning objectives provide a framework for adapting the experiment to an inquiry-based lab activity.

- The inquiry process begins with pre-lab preparation to determine the quantities of reactants required. Give students the general outline for the procedure (refluxing, extraction, and distillation) and ask them to calculate the amounts of ethyl alcohol and glacial (17.4 M) acetic acid needed to prepare 15 g of ethyl acetate. Discuss the following prerequisite knowledge: (1) Synthesis of an ester is generally reversible. (2) In order to shift the equilibrium for this reversible reaction to favor products, a 25% excess of one of the reactants is desired. (3) Excess acetic acid can be easily removed from the product mixture by extraction with a base.
- One important aspect of inquiry that is often overlooked is the trial-and-error method of identifying optimum conditions for a synthesis procedure. This method usually involves many routine trials with each trial manipulating just one of the variables to obtain the “best” results. Encourage a cooperative class approach to identify reaction conditions that will give the highest yield of ethyl acetate. What ratio of reactants works best? Reaction time? Temperature? Catalyst?
- Explore the use of chromatography procedures as alternatives to extraction and distillation for separating the product mixture and determining the purity of the product, respectively. The mixture may be separated by column chromatography and the purity of ethyl acetate may be visualized using thin-layer chromatography.
- Extend the experiment to prepare a variety of esters that are natural flavor and fragrance agents. Provide a selection of carboxylic acids (acetic acid, propionic acid, benzoic acid, salicylic acid, and anthranilic acid) as well as alcohols (methyl alcohol, ethyl alcohol, propyl alcohol, isoamyl alcohol, and octyl alcohol). Ask students to research natural fruity esters and select appropriate combinations to prepare them. This guided-inquiry activity should be carried out in a well-ventilated lab using microscale quantities (1–2 mL) of acids and alcohols. Students love the natural, fruity and minty smells that are produced. Note that butyric acid has a very unpleasant odor and is not recommended as one of the carboxylic acid choices.

Alignment with AP Chemistry Curriculum Framework—Big Ideas 2 and 3

Enduring Understandings and Essential Knowledge

Matter can be described by its physical properties. The physical properties of a substance generally depend on the spacing between the particles (atoms, molecules, ions) that make up the substance and the forces of attraction among them. (Enduring Understanding 2A)

2A3: Solutions are homogeneous mixtures in which the physical properties are dependent on the concentration of the solute and the strengths of all interactions among the particles of the solutes and solvent.

Chemical changes are represented by a balanced chemical equation that identifies the ratios with which reactants react and products form. (Enduring Understanding 3A)

3A2: Quantitative information can be derived from stoichiometric calculations that utilize the mole ratios from the

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balanced chemical equation. The role of stoichiometry in real-world applications is important to note, so that it does not seem to be simply an exercise done only by chemists.

Chemical reactions can be classified by considering what the reactants are, what the products are, or how they change from one into the other. Classes of chemical reactions include synthesis, decomposition, acid–base, and oxidation–reduction reactions. (Enduring Understanding 3B)

3B1: Synthesis reactions are those in which atoms or molecules combine to form a new compound. Decomposition is the reverse of synthesis, a process whereby molecules are decomposed, often by the use of heat.

Learning Objectives

- 2.10 The student can design and/or interpret the results of a separation experiment (filtration, paper chromatography, column chromatography, or distillation) in terms of the relative strength of interactions among and between the components.
- 3.3 The student is able to use stoichiometric calculations to predict the results of performing a reaction in the laboratory and/or to analyze deviations from the expected results.
- 3.4 The student is able to relate quantities (measured mass of substances, volumes of solutions, or volumes and pressure of gases) to identify stoichiometric relationships for a reaction, including situations involving limiting reactants and situations in which the reaction has not gone to completion.
- 3.6 The student is able to use data from synthesis or decomposition of a compound to confirm the conservation of matter and the law of definite proportions.

Science Practices

- 2.2 The student can apply mathematical routines to quantities that describe natural phenomena.
- 4.2 The student can design a plan for collecting data to answer a particular scientific question.
- 4.3 The student can collect data to answer a particular scientific question.
- 5.1 The student can analyze data to identify patterns or relationships.
- 6.1 The student can justify claims with evidence.

The *Synthesis, Isolation and Purification of an Ester*—AP Chemistry Classic Laboratory Kit is available from Flinn Scientific, Inc.

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