



Separating (and Identifying) the Components of a Mixture

Purpose

To prepare and separate a 3-component mixture using acid–base extractions and learn some basic concepts relating to the purification and analysis of organic compounds.

Learning Objectives

- Learn how to purify solids by acid-base extraction.
- Learn how to dry a solid product.
- Confirm the identity, and determine purity, of a compound using melting point analysis.

Laboratory Skills

- Perform an acid-base workup.
- Perform a vacuum filtration.
- Perform a liquid-liquid extraction.
- Perform a recrystallization.
- Perform a melting point analysis.

Equipment

- Microspatula
- Two small beakers
- Two 50-mL Beakers
- Glass rod
- 5-mL Conical vial
- Centrifuge tubes and caps
- pH Paper
- 150-mL Beaker
- Ice/water bath
- Hirsch funnel
- K1max filter flask
- Watch glasses
- Capillary tubes
- Mel-temp apparatus

Chemicals

- Three unknown solids
- Diethyl ether
- 5% Sodium bicarbonate (aq)
- 10% Sodium hydroxide (aq)
- Sodium sulfate
- 6 M Hydrochloric acid

Background

Extraction involves dissolving a compound, or compounds, either (1) from a solid into a solvent or (2) from a solution into another solvent. A familiar example of the first case is making a cup of tea or coffee—the soluble flavor and odor chemicals and caffeine are extracted from the solid tea leaves or ground coffee beans into hot water (the solvent). Insoluble plant material is left behind in the tea bag or coffee filter. An example of the second case is an experiment that is often done in an introductory organic lab—an organic solvent is used to extract the caffeine from an aqueous tea or coffee solution, leaving the more water-soluble compounds behind in the aqueous solution. This in fact is how decaffeinated tea and coffee are sometimes made.

Solvent-Solvent Extraction

An acid-base extraction is a modification of the second case above; that is, it is a solvent-solvent extraction. Before looking at what makes it an acid-base extraction, let's first consider solvent-solvent extractions in general. A requirement of a solvent-solvent extraction is that the two solvents be mostly insoluble in one another; that is, a mixture of the two solvents will separate into two layers. It is important to note that *the upper layer is that which is less dense*. For example, a mixture of diethyl ether and water will separate into two layers, with the ether layer (density = 0.71 g/mL) being on top of the water layer (density = 1.00 g/mL) (Figure SP.1). Note that in almost every case, one of the solvents is water or an aqueous solution. The other solvent is an organic solvent that is mostly insoluble in water, for example; diethyl ether, hexane, and/or dichloromethane. The organic solvent must also be volatile (easily-evaporated) so it can be easily removed by evaporation at the end.

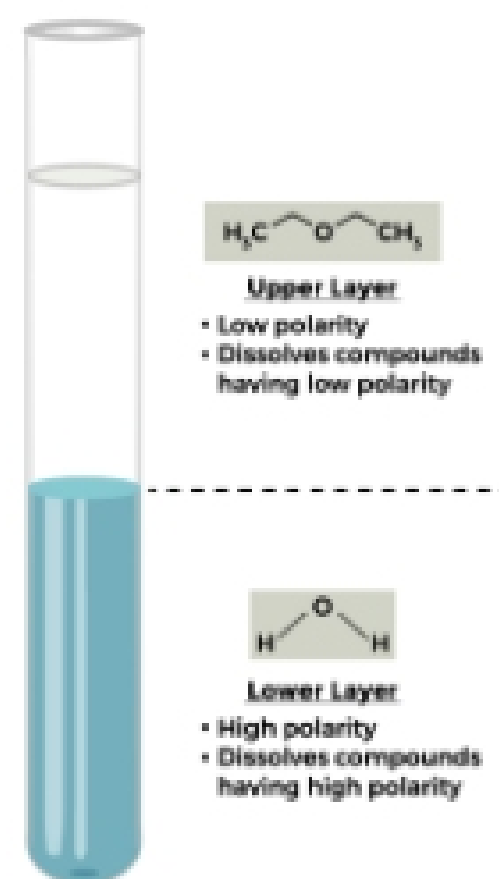
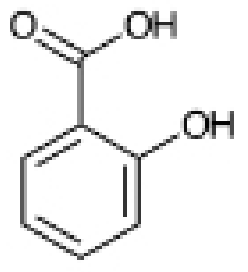
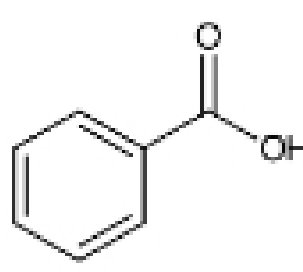
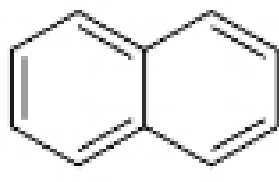
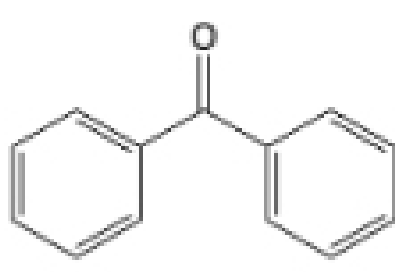
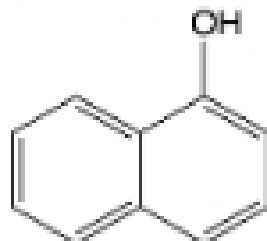
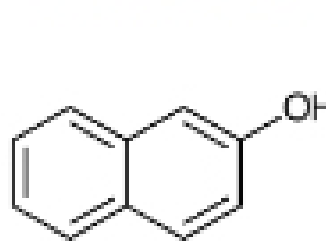
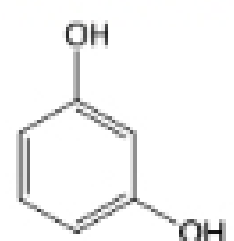
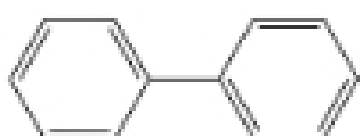


Figure SP.1: Solvent-solvent extraction

Table SP.1: Possible Unknown Compounds with Melting Point Ranges

			
Salicylic acid MP: 158-159 °C	Benzoic acid MP: 121-122 °C	Naphthalene MP: 79-81 °C	Benzophenone MP: 48-49 °C
			
1-Naphthol MP: 95-96 °C	2-naphthol MP: 121-123 °C	Resorcinol MP: 110-111 °C	Biphenyl MP: 68-70 °C

Acid-Base Extraction

In the experiment performed in this lab, a mixture of a carboxylic acid (stronger acid), a phenol (weaker acid), and a neutral compound (Table SP.1) will be separated by sequential acid-base extractions (Figure SP.2 and Figure SP.3). The separated compounds will be purified by recrystallization and then identified by melting point analysis. The solid mixture of unknowns is first dissolved in ether and mixed thoroughly with aqueous sodium bicarbonate (weaker base). After the layers settle, they are separated, and placed into different tubes. The ether layer is then extracted with sodium hydroxide (stronger base), the layers separated, and each layer worked up separately. Three separate solutions result; one ether and two aqueous. Dissolved water is removed from the ether solution using a drying procedure and the ether is evaporated to yield the solid neutral compound. This can then be recrystallized to yield pure solid neutral compound. The basic aqueous solutions are neutralized with HCl to yield the carboxylic acid and phenol, which, because of their water insolubility, precipitate out. The precipitates can be collected, recrystallized, and allowed to dry to yield pure carboxylic acid and pure phenol.

The extractions must be done in the order shown; first with sodium bicarbonate (weaker base) and then with sodium hydroxide (strong base).