

Organic Chemistry I Fall 2013 Lab  
Extraction  
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Experiment 3

Extraction

Organic I Lab Fall 2013: CHEM 2423

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## Abstract and Summary

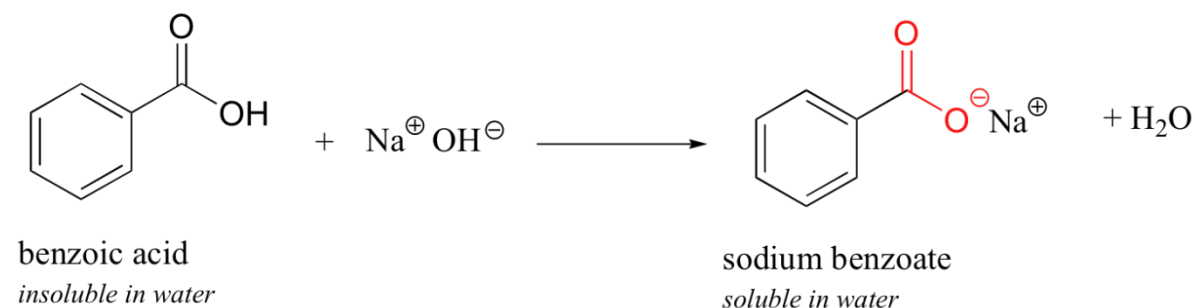
In this experiment, we extracted 0.61 grams of benzoic acid in water with methylene chloride. And then, we determined the amount of benzoic acid in the aqueous layer by titration with sodium hydroxide. From collected data, we calculated the distribution coefficient value ( $K_d$ ) which is the ratio of the concentration of the solute in each solvent at equilibrium. We did another extraction with the same amount of benzoic acid and methylene chloride. In the second extraction, the amount of methylene chloride was divided in two equal portions. We determined the amount of benzoic acid in the aqueous layer and the amount of benzoic acid in methylene chloride. From those values, we calculated the  $K_d$  value. And then, we compared the two  $K_d$  values.

## Introduction

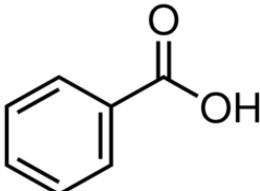
Extraction is a very general, highly versatile technique that is of great value not only in the laboratory but also in everyday life. In the laboratory, extraction is frequently employed to isolate one or more components from a mixture based on the unequal distribution of a substance between two immiscible liquids. The distribution between the two liquids is related to the substance's solubility in each liquid. The substance can be transferred from one liquid to the other by shaking it together with the two liquids in a separatory funnel. One of the solvents must be immiscible with the other one in which the compound is dissolved. Two liquid layers are formed, and the layer that has most of the desired product in it can be separated from the other. When two immiscible solvents are shaken together, the solute distributes itself between them in a ratio roughly proportional to its solubility in each. The ratio of the concentration of the solute in each solvent at equilibrium is a constant called the distribution coefficient ( $K_d$ ).

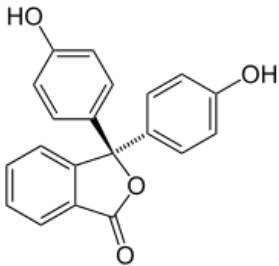
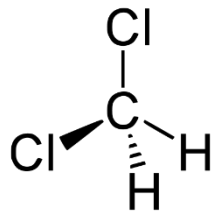
The purpose of this experiment was to compare the amount of benzoic acid extracted by methylene chloride in one portion and the amount of benzoic acid extracted by methylene chloride divided into two portions.

## Reactants and Products



## Compounds and Precautionary Measures

Name and Structures	Chemical Hazards	Precautionary Measures
Benzoic Acid 	Harmful if inhaled. Causes respiratory tract irritation. Harmful if absorbed through skin. Causes serious eye damage. Harmful if swallowed	Avoid breathing dust/ fume/ gas/ mist/ vapors/ spray. Wear protective gloves/ eye protection/ face protection. If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
Sodium Hydroxide	Very hazardous. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering.	If inhaled: If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician. In case of skin contact: Wash off

	<p>Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by burning, sneezing and coughing. Severe over-exposure can produce lung damage, choking, unconsciousness or death.</p>	<p>with soap and plenty of water. Consult a physician.        In case of eye contact: Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.        If swallowed: Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.</p>
<p>Phenolphthalein</p>  <p>The image shows the chemical structure of phenolphthalein, which consists of a central carbon atom bonded to three phenyl rings and one lactone ring. Two of the phenyl rings have hydroxyl groups at the para position.</p>	<p>Highly flammable in presence of open flames and sparks, of heat. Slightly flammable to flammable in presence of oxidizing materials. Explosion Hazards in Presence of Various Substances: Slightly explosive in presence of oxidizing materials, of acids. Non-explosive in presence of open flames and sparks, of shocks.</p>	<p>Keep away from heat. Keep away from sources of ignition        Small fire: Use dry chemical powder. Large fire: Use alcohol foam, water spray or fog.</p>
<p>Dichloromethane</p>  <p>The image shows the chemical structure of dichloromethane (CH2Cl2), with a central carbon atom bonded to two chlorine atoms and two hydrogen atoms.</p>	<p>Eye: Contact with eyes may cause severe irritation, and possible eye burns.        Skin: May be absorbed through the skin. Causes irritation with burning pain, itching, and redness. Prolonged exposure may result in skin burns.        Ingestion: Causes gastrointestinal</p>	<p>Eyes: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical aid.        Skin: In case of contact, flush skin with plenty of water. Remove contaminated clothing and shoes. Get medical aid if irritation develops</p>

	<p>irritation with nausea, vomiting and diarrhea. May cause kidney damage.</p> <p>May cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea.</p> <p>Advanced stages may cause collapse, unconsciousness, coma and possible death due to respiratory failure.</p>	<p>and persists. Wash clothing before reuse.</p> <p>Ingestion: If swallowed, do not induce vomiting unless directed to do so by medical personnel.</p> <p>Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.</p>
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## Laboratory Procedure

The procedures for this experiment are from *Organic Chemistry Laboratory Standard and Microscale Experiments* pg. 62-63

## Observations

### Benzoic Acid Solution:

Weight of benzoic acid: 0.61 g

Number of moles of benzoic acid:  $n = 0.61 \text{ g} \times \frac{1 \text{ mole}}{122 \text{ g}} = 0.005 \text{ mol}$

Volume of benzoic acid solution: 250 mL

Molarity of benzoic acid solution:  $M = \frac{n}{V} = \frac{0.005 \text{ g}}{250 \text{ mL}} \times \frac{1000 \text{ mL}}{1 \text{ L}} = 0.020 \text{ M}$

### Sodium Hydroxide Solution:

	Volume of benzoic acid titrated (C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> H)	Volume of sodium hydroxide used (NaOH)
Trial 1	10.0 mL	10.0 mL
Trial 2	10.0 mL	10.0 mL
Average	10.0 mL	10.0 mL

Molarity of Sodium hydroxide:  $M(\text{NaOH}) \times V(\text{NaOH}) = M(\text{benzoic acid}) \times V(\text{benzoic acid})$

$$M(\text{NaOH}) = \frac{0.020 \text{ M} \times 10.0 \text{ mL}}{10.0 \text{ mL}} = 0.020 \text{ M}$$

**Single Extraction: (10 mL of CH<sub>2</sub>Cl<sub>2</sub> in 1 portion)**

	Volume of methylene chloride (CH <sub>2</sub> Cl <sub>2</sub> )	Volume of benzoic acid used (C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> H)	Volume of sodium hydroxide used (NaOH)
Trial 1	10.0 mL	50.0 mL	16.8 mL
Trial 2	10.0 mL	50.0 mL	15.8 mL
Average	10.0 mL	50.0 mL	16.3 mL

Total milimoles of benzoic acid:

$$50.0 \text{ mL benzoic acid} \times \frac{0.020 \text{ mmol benzoic acid}}{1 \text{ mL benzoic acid}} = 1.0 \text{ mmol benzoic acid}$$

Milimoles of benzoic acid left in aqueous layer:

$$16.3 \text{ mL NaOH} \times \frac{0.020 \text{ mmol NaOH}}{1 \text{ mL NaOH}} \times \frac{1 \text{ mol benzoic acid}}{1 \text{ mol NaOH}} = 0.33 \text{ mmol benzoic acid}$$

Milimoles of benzoic acid in CH<sub>2</sub>Cl<sub>2</sub> layer:

$$1.0 \text{ mmol} - 0.33 \text{ mmol} = 0.67 \text{ mmol}$$

Distribution coefficient:

$$K_d = \frac{C(\text{CH}_2\text{Cl}_2)}{C(\text{water})} = \frac{0.67 \text{ mmol}/10.0 \text{ mL}}{0.33 \text{ mmol}/50.0 \text{ mL}} = 10.2$$

**Multiple Extraction: (10 mL of CH<sub>2</sub>Cl<sub>2</sub> in 2 portions)**

Volume of methylene chloride (CH <sub>2</sub> Cl <sub>2</sub> )	Volume of benzoic acid used (C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> H)	Volume of sodium hydroxide used (NaOH)
10.0 mL	50.0 mL	15.0 mL

Total milimoles of benzoic acid:

$$50.0 \text{ mL benzoic acid} \times \frac{0.020 \text{ mmol benzoic acid}}{1 \text{ mL benzoic acid}} = 1.0 \text{ mmol benzoic acid}$$

Milimoles of benzoic acid left in aqueous layer:

$$15.0 \text{ mL NaOH} \times \frac{0.020 \text{ mmol NaOH}}{1 \text{ mL NaOH}} \times \frac{1 \text{ mol benzoic acid}}{1 \text{ mol NaOH}} = 0.30 \text{ mmol benzoic acid}$$

Milimoles of benzoic acid in CH<sub>2</sub>Cl<sub>2</sub> layer:

$$1.0 \text{ mmol} - 0.30 \text{ mmol} = 0.70 \text{ mmol}$$

Distribution coefficient:

$$K_d = \frac{C(\text{CH}_2\text{Cl}_2)}{C(\text{water})} = \frac{0.70 \text{ mmol}/10.0 \text{ mL}}{0.30 \text{ mmol}/50.0 \text{ mL}} = 11.7$$

Milimoles of benzoic acid in CH<sub>2</sub>Cl<sub>2</sub> layer based on single extraction K<sub>d</sub>:

$$10.2 \times 10.0 \text{ mL} \times \frac{0.30 \text{ mmol}}{50.0 \text{ mL}} = 0.61 \text{ mmol}$$

**Discussion and Conclusions:**

According to the data collected, the molarity of benzoic acid and the molarity of sodium hydroxide have a 1:1 ratio, which means the molarity of sodium hydroxide is 0.020 M. However, when transferring the benzoic acid to a new flask, we spilled a small amount of benzoic acid. As a result, the actual molarities were less than calculated values.

The distribution coefficient value for single extraction was 10.2. The distribution coefficient value for multiple extraction was 11.7. The two distribution coefficient ( $K_d$ ) values were reasonable and expected because in this experiment, we used methylene chloride. I would expect different values if we use other organic compound such as ether instead of methylene chloride because methylene dichloride is more miscible in water than ether. This may also explain for the cloudy aqueous layer we got after extraction. Still, a significant amount of benzoic acid was transferred to the methylene chloride layer. The multiple extraction  $K_d$  was greater than the single extraction  $K_d$ , which means more solute was transferred to the methylene with multiple extraction. The required amount of methylene to remove benzoic acid in multiple extraction was smaller than in single extraction. From the collected data and the result, this experiment can be considered as a successful experiment.

## References

1. Procedure and pictures from *Organic Chemistry Laboratory Standard and Microscale Experiments*, Charles E Bell, Douglas Taber, K Clark, 3<sup>rd</sup> Ed. Brooks Cole.
2. <http://www.sigmaaldrich.com/united-states.html> for images of structures and MSDS sheets