Separatory Sorcery

Introduction

A simple, colorful demonstration of solubility and acid–base extraction using a separatory funnel.

Concepts

- Solubility
- Acid–base properties

Materials

Hexanes, C_6H_{14}, 62 mL
2,6-Dichloroindophenol solution, 0.05%, 6 mL
Hydrochloric acid solution, HCl, 1 M, 10 mL
Sodium hydroxide solution, NaOH, 1 M, 10 mL
Ring stand with buret clamp
Separatory funnel, 250-mL

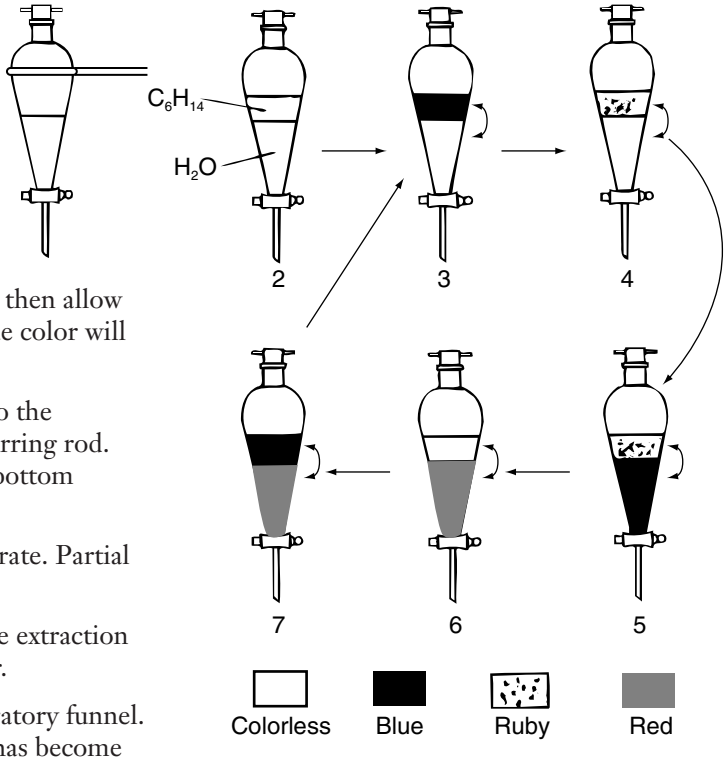
Safety Precautions

Sodium hydroxide solution is a corrosive liquid to skin, eyes, and other tissue. Avoid contact with tissue, especially the eyes. Hydrochloric acid solution is toxic by ingestion and inhalation; corrosive to skin and eyes. Avoid contact with skin. Hexane is a flammable liquid and a dangerous fire risk; a respiratory irritant. Keep away from sources of ignition. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Perform demonstration in a operating fume hood. Please review current Material Safety Data Sheets for additional safety, handling, and disposal information.

Preparation

To prepare a 0.05% solution of 2,6 dichlorindophenol solution: Dissolve 25 mg of 2,6 dichlorindophenol in 50 mL of distilled or deionized water. Add 1 mL of 1.0 M sodium hydroxide solution. Mix thoroughly.

Procedure



1. Set up the separatory funnel on ring stand as shown in the diagram.
2. Pour 62 mL of chloroform and 125 mL of distilled or deionized water into the separatory funnel. Observe the two clear, distinct layers.
3. Add 6 mL of the 2,6-dichloroindophenol solution to the separatory funnel. The bottom layer will turn dark blue. Stopper and shake the separatory funnel and then allow the layers to separate again. You will notice that the blue color will remain in the water layer.
4. Now add 1 mL of the 1 M hydrochloric acid solution to the separatory funnel. Stir only the bottom layer using a stirring rod. Do not allow the water and hexane layers to mix. The bottom (water) layer is now a ruby color.
5. Briefly mix the two layers, then allow the layers to separate. Partial extraction of the red into the organic layer will occur.
6. Thoroughly mix the layers. This results in the complete extraction of the red from the aqueous layer into the organic layer.
7. Slowly add 2 mL of 1 M sodium hydroxide to the separatory funnel. Nothing appears to happen; the aqueous (lower) layer has become alkaline but has not yet come in contact with the indicator. A “one shake” mixing of the layers will allow a partial extraction of the indicator out of the organic layer. This will result in a red organic layer and a blue water layer.

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8. Thoroughly mix the layers to complete the extraction of indicator from the organic layer, leaving a blue aqueous layer as in step 3. The sequence of steps 3 through 7 can be repeated if desired.

Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures governing the disposal of laboratory waste. The mixture of solutions may be disposed of by draining the hexane layer and disposing of the hexane layer according to Flinn Suggested Disposal Method #18a. The aqueous layer may be disposed of according to Flinn Suggested Disposal Method #26b.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
Evidence, models, and explanation
Constancy, change, and measurement
Content Standards: Grades 5–8
Content Standard B: Physical Science, properties and changes of properties in matter
Content Standards: Grades 9–12
Content Standard B: Physical Science, structure and properties of matter, chemical reactions

Discussion

This demonstration takes advantage of the color and solubility properties of the indicator, 2,6-dichloroindophenol. In an alkaline environment, 2,6-dichloroindophenol is blue in color and is water soluble. In an acidic environment, the indicator is red and is more readily soluble in organic solvents than water. These properties result in a very colorful and informative demonstration.

The use of the separatory funnel is a common operation in organic chemistry. In the course of separating a mixture or working up a reaction, chemists frequently take advantage of the acidic or basic properties of a solute to partition between an organic phase and an aqueous phase. Because most solutes are colorless, beginning students find it difficult to understand what is happening inside a separatory funnel as the pH of the aqueous layer is changed and the separatory funnel is shaken.

Acknowledgments

Special thanks to T. Ross Kelly of Boston College, Chestnut Hill, MA for allowing us to reproduce this demonstration. A great photo of this demonstration can be found on the cover of the October, 1993 issue of the *Journal of Chemical Education*.

References

Kelly, T. R. *J. Chem. Ed.* **1993,** *70*, 848–849.

Armstrong, J. M. *Biochim. Biophys. Acta.* 1964, 86, 194–197.

Silversmith, E. F. *J. Chem. Ed.* **1972,** *49*, A694.

Materials for *Separatory Sorcery* are available from Flinn Scientific, Inc.

Catalog No.	Description
H0046	Hexanes, 100 mL
D0009	2,6-Dichloroindophenol, 1 g
H0013	Hydrochloric Acid Solution, 1 M, 500 mL
S0148	Sodium Hydroxide Solution, 1 M, 500 mL
GP5060	Separatory Funnel, Borosilicate Glass, 250 mL

Consult the Flinn Scientific website for current prices.

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