Lab 4: Extraction of Iodine from an Aqueous Solution

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Objectives

By the end of this laboratory, you should have developed the skills to do the following:

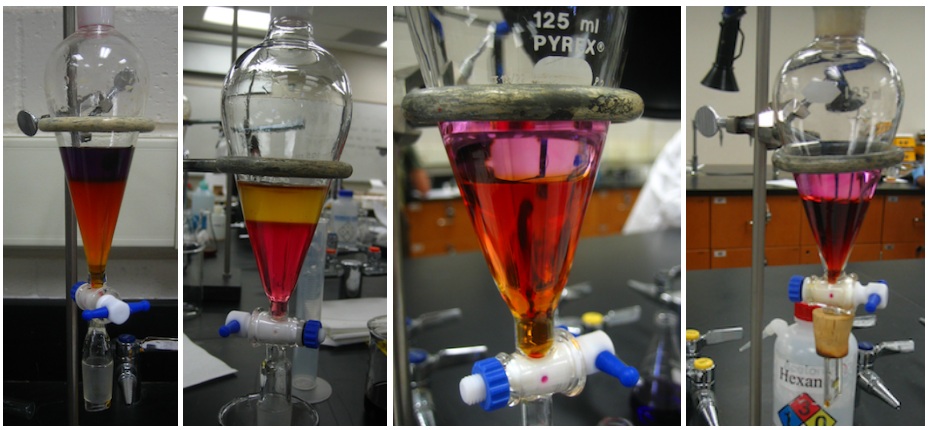
- Correctly perform a liquid-liquid extraction.
- Identify the aqueous layer and the organic layer in a liquid-liquid extraction.

Recommended Resources

- Handout ~ Topic 4: Writing an Organic Chemistry Lab Report
  http://www.csub.edu/chemistry/organic/manual/Topic4_Report.pdf
- Handout ~ Topic 7: Using a Pasteur Pipet
  http://www.csub.edu/chemistry/organic/manual/Topic7_Pipet.pdf
- Tutorial ~ Introduction to Extraction Tutorial
  https://www.youtube.com/playlist?list=PLC5C96C5188CC44CE

Background

Extraction is the process of transferring a substance to a solvent. It is useful for separating mixtures of compounds and removing impurities from products. The extractions that you will do for most labs involve colorless substances. Under these circumstances, it is impossible to see in which layer your desired substance resides. Before you begin any extraction procedures for those labs it will be beneficial to try it first with a colored substance, such as iodine, so you can visually see what happens in an extraction. In this lab, you and a partner will use two different organic solvents to extract iodine from water and compare your results. Further, you will compare the efficiency of a single, large volume extraction to the efficiency of three smaller volume extractions.



Iodine is more soluble in organic solvents than it is in water. You will be extracting iodine from an aqueous solution of iodine and potassium iodide. The iodine/potassium iodide solution is brown in color, whereas iodine by itself is a nice violet color.

Some examples of what you might expect to see during the course of your extraction are shown in the pictures on the previous page. Note that the location of the iodine (top layer versus bottom layer) changes based on the density of the organic solvent. Also, note the differences in color of the layers as more and more iodine is extracted. Be sure to pay attention to and record these types of observations during the course of your experiment.

Lab Notebook Preparation

Before coming to lab, the following items must be in your lab notebook:

1. Title of the experiment & date the experiment is to be performed
2. Densities of dichloromethane, hexane, and water
3. Identify which layer will be on top in each type of extraction
4. Your initial hypothesis regarding the efficiency of single versus multiple extractions
5. Hazards of and appropriate precautions for the safe handling of hexane, dichloromethane, and iodine
6. References

Directions

1. Before you begin this experiment, formulate a hypothesis regarding the efficiency of single versus multiple extractions. Use the principles of extraction and the partition coefficient when developing your hypothesis and decide what results will reinforce or contradict the hypothesis.
2. Set up your work area by clamping a ring clamp that will hold your separatory funnel, ensure that the stopcock and stopper fit well and work properly, and make sure that the stopcock is closed before beginning.
3. Find a partner who is willing to conduct the same experiment, but who will use a different organic solvent. Select who will use dichloromethane and who will use hexane.
4. You will each perform the experiment twice. The first time you will conduct one extraction of 60 mL, and the second time you will conduct three extractions of 20 mL each (60 mL total).
5. First, conduct the single extraction.
    a. Measure out 20 mL of 0.005 M iodine/potassium iodide solution using a graduated cylinder.
    b. Use a funnel to transfer the solution to your separatory funnel.
    c. In a fume hood or under a snorkel, measure out 60 mL of your organic solvent using a graduated cylinder.
    d. Carefully add the organic solvent to the separatory funnel without mixing. Note which layer is on top and which layer is on the bottom. Record any observations.

e. Stopper the separatory funnel and shake it gently, venting frequently. Continue until there is no more color change. Note how long it took to reach equilibrium. Record the colors and intensities of each layer.

f. Let the mixture sit in the ring clamp until the two layers have fully separated then carefully drain off the bottom layer into a 125 mL Erlenmeyer flask.

g. Transfer the top layer to another 125 mL Erlenmeyer flask. Label the flasks and compare your results with your partner's. (Which organic layer is darker in color? The dichloromethane or the hexane? Which aqueous layer is darker in color?)

h. Save the flasks so you can compare them to your results from the triple extraction.

6. Next, conduct the triple extraction.

   a. Measure out 20 mL of 0.005 M iodine-potassium using a graduated cylinder (do NOT reuse the iodine-potassium iodide solution from the single extraction).

   b. Use a funnel to transfer the solution to your separatory funnel.

   c. In a fume hood or under a snorkel, measure out 20 mL of your organic solvent using a graduated cylinder.

   d. Carefully add the organic solvent to the separatory funnel without mixing. Note which layer is on top and which layer is on the bottom. Record any observations.

   e. Stopper the separatory funnel and shake it gently, venting frequently. Continue until there is no more color change. Note how long it took to reach equilibrium. Record the colors and intensities of each layer.

   f. Let the mixture sit in the ring clamp until the two layers have fully separated then remove the organic layer. (Note: This process will differ depending on which solvent you are working with.)

   g. Once the organic layer has been removed, combine another 20 mL of your organic solvent with the aqueous layer. Again, stopper the funnel, shake, let it sit, and remove the organic layer.

   h. Combine your second organic layer with the first layer you removed.

   i. Once the organic layer has been removed, combine a final 20 mL of your organic solvent with the aqueous layer. Again, stopper the funnel, shake, let it sit, and remove the organic layer.

   j. Combine your third organic layer with the first two.

7. Compare your results with your partner's. (Which organic layer is darker in color? The dichloromethane or the hexane? Which aqueous layer is darker in color?)

8. Compare the results from the single extraction with the results from the triple extraction. (Which organic layer is darker in color? Which aqueous layer is darker in color?)

9. Record your final conclusions. (Which solvent extracted the most iodine? Which type of extraction removed the most iodine? Was your initial hypothesis correct?)

10. If time allows and you would like additional practice, you may repeat the experiment with the solvent you did not use.

11. Dispose of all waste in the designated waste container.

Reporting your Results

Write your report according to the guidelines described in "Topic 4: Writing an Organic Chemistry Lab Report". Work with your partner on this report.

References & Additional Resources

1. Lehman, J. W. *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3rd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 475.