

Extraction

Determination of a Distribution Coefficient

Week 5 – September 21 – 27, 2010

From Bell, Clark and Taber, pgs. 41, 42

Apparatus required:

- 2 - 50 mL burettes (use one for benzoic acid solution and the other for NaOH solution)
- 1 - 250 mL Volumetric flask
- 2 - 250 mL Erlenmeyer flasks
- Magnetic stir bar
- 10 mL Graduated cylinder
- 125 mL separatory funnel
- 1 Ring Clamp
- Hot plate
- 1 - 50 mL Beaker

A. Preparation of the Standard Benzoic Acid Solution

1. Weigh out 0.61 g of benzoic acid onto some weighing paper.
2. Place this benzoic acid in a 250 mL Erlenmeyer flask containing a magnetic stir bar and add 150 mL of water
3. Heat the resulting mixture on a hot plate with magnetic stirring until the benzoic acid completely dissolves to give a clear solution.
4. Using a funnel carefully pour the resulting solution into a 250 mL volumetric flask. Rinse the Erlenmeyer flask several times with water (three 20 mL portions) and pour the washes into the 250 mL volumetric flask. These washes ensure a quantitative transfer of all benzoic acid from the Erlenmeyer flask to the volumetric flask.
5. Cool the solution in the volumetric flask to room temperature and then **CAREFULLY** add water to the flask to adjust the total volume to exactly 250 mL (add water until the level reaches the 250 mL mark etched on the neck of the volumetric flask). This provides a standard 0.02 M benzoic acid solution.
6. Stopper the flask and invert it several times to ensure that the contents are mixed uniformly.

NOTE: CARRY OUT EXTRACTIONS IN YOUR HOOD

B. Single Extraction and Distribution Coefficient Determination

1. Using a burette, place 50 mL of the standard 0.02 M benzoic acid solution into a 125 mL separatory funnel. **MAKE SURE THAT THE FUNNEL STOPCOCK IS CLOSED BEFORE THE ADDITION.**
2. Place the separatory funnel in a ring clamped to a bar in your hood.
3. Then, using a 10 mL graduated cylinder, add exactly 10 mL of methylene chloride to the separatory funnel.
4. Stopper the funnel and, holding the stopper securely in place, invert the funnel and immediately open the stopcock to release pressure resulting from the solvent vapors. After closing the stopcock, give the funnel 5 or 6 vigorous shakes and then immediately vent the funnel to relieve the pressure buildup.

5. After the second venting, close the stopcock, shake the funnel **vigorously** for 30 seconds, vent the funnel and then place it in the ring. **NOTE: If the separatory funnel is not shaken vigorously, you will not obtain good extraction of the benzoic acid.**
6. **Remove the stopper** and allow the layers to separate for several minutes.
7. Carefully drain off the lower methylene chloride layer into a beaker.
8. Gently, swirl the separatory funnel (in an upright position) for 5 – 10 seconds and then, after allowing the funnel to stand in the ring for a minute or two, drain off a small amount of any methylene chloride lower layer that may have accumulated after the swirling process.
9. Pour the aqueous layer remaining in the funnel into a 250 mL Erlenmeyer flask.
10. Rinse the separatory funnel twice with a few mLs of water and put the washes into the Erlenmeyer along with the main aqueous layer.
11. Add 2 – 4 drops of phenolphthalein solution to the aqueous solution in the Erlenmeyer and **carefully** titrate with 0.02 M sodium hydroxide to a pink endpoint. You may want to place a white sheet of paper underneath the Erlenmeyer to better visualize the pink endpoint.
12. Calculate the number of grams of benzoic acid remaining in the water according to the Calculation Worksheet (Section B.1. and 2.) accompanying this handout.
13. By difference, calculate the number of grams of benzoic acid in the methylene chloride layer. See Calculation Worksheet (Section B.3.).
14. Calculate K_d using the above values and volumes. See Calculation Worksheet (Section B.4.) for details.

C. Multiple Extractions

1. Using the benzoic acid burette, place another 50 mL of the standard benzoic acid solution into a 125 mL separatory funnel.
2. Add 5 mL of methylene chloride to the separatory funnel.
3. Stopper the funnel and, holding the stopper securely in place, invert the funnel and immediately open the stopcock to release pressure buildup from the solvent vapors. After closing the stopcock, give the funnel 5 or 6 vigorous shakes and then immediately vent the funnel to relieve the pressure buildup.
4. After the second venting, close the stopcock, shake the funnel **vigorously** for 30 seconds, vent the funnel and then place it in the ring. **NOTE: If the separatory funnel is not shaken vigorously, you will not obtain good extraction of the benzoic acid.**
5. **Remove the stopper** and allow the layers to separate for several minutes.
6. Carefully drain off the lower methylene chloride layer into a beaker.
7. Gently, swirl the separatory funnel (in an upright position) for 5 – 10 seconds and then, after allowing the funnel to stand in the ring for a minute or two, drain off a small amount of any methylene chloride lower layer that may have accumulated after the swirling process.
8. Add another 5 mL portion of methylene chloride to the separatory funnel containing the aqueous layer.
9. Stopper the funnel and, holding the stopper securely in place, invert the funnel and immediately open the stopcock to release pressure buildup from the solvent vapors. After closing the stopcock, give the funnel 5 or 6 vigorous shakes and then immediately vent the funnel to relieve the pressure buildup.
10. After the second venting, close the stopcock, shake the funnel **vigorously** for 30 seconds, vent the funnel and then place it in the ring. **NOTE: If the separatory funnel is not shaken vigorously, you will not obtain good extraction of the benzoic acid.**
11. **Remove the stopper** and allow the layers to separate for several minutes.

12. Carefully drain off the lower methylene chloride layer into a beaker.
13. Gently, swirl the separatory funnel (in an upright position) for 5 – 10 seconds and then, after allowing the funnel to stand in the ring for a minute or two, drain off a small amount of any methylene chloride lower layer that may have accumulated after the swirling process.
14. Pour the aqueous layer remaining in the funnel into a 250 mL Erlenmeyer flask.
15. Rinse the funnel twice with a few mLs of water and put the washes into the Erlenmeyer along with the main aqueous layer.
16. Add 2 -4 drops of phenolphthalein solution to the aqueous solution in the Erlenmeyer and carefully titrate with 0.02 *M* sodium hydroxide to a pink endpoint. You may want to place a white sheet of paper underneath the Erlenmeyer to better visualize the pink endpoint.
17. Calculate the amount of benzoic acid removed from the aqueous layer with the two 5 mL extractions and compare with that number with the amount removed with the single 10 mL extraction. (See Calculation Worksheet, Section C.1., 2., 3.). What results did you expect? What results did you observe?
18. Using the K_a value you determined above in Part B14, calculate the theoretical amount of acid that should be removed by two 5 mL extractions (See Calculation Worksheet, Section C.4) and compare it with the amount that you found experimentally with the two 5 mL extractions. How do the experimental and theoretical values compare?