

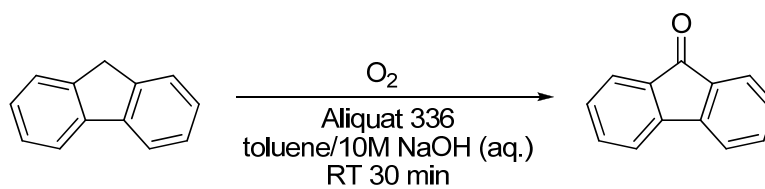
# Experiment 6

CHEM 213



## Air Oxidation of Fluorene to Fluorenone

### —Separation of Products by Column Chromatography



**Introduction:** In this experiment, you will be synthesizing fluorenone from fluorene by air oxidation. Since this reaction is not efficient over a short period of time a mixture of unreacted fluorene and fluorenone will be present. Column chromatography will be used to separate the two components. One component is bright yellow and can be followed on the column visually, however the other is colorless and will need to be detected by TLC analysis. Column chromatography uses the same principles as TLC to separate compounds, but can be used on a larger scale.

Infrared spectroscopy will be used to determine the identity of each product.

### Objectives

- Synthesize the fluorene/fluorenone mixture
- Separate the fluorene from fluorenone using the technique of column chromatography
- Identify products of the reaction by TLC analysis and infrared (IR) spectrometry

### Reading

**Liquid Chromatography:** Section 17.1-8, pp. 206-221.

Watch the video of a chromatographic separation taking place on Dr. Justik's CHEM 213 website.

### Pre-Lab Questions

1. What effect will the following factors have on a chromatographic separation: a) too polar of an adsorbent; b) collection of large fractions; c) solvent level falling below the top of the adsorbent?
2. Arrange the following compounds in the order they would elute from a silica gel column: a) 2-octanol; b) 1,3-dichlorobenzene; c) *tert*-butylcyclohexane; d) benzoic acid. Explain.
3. How will IR spectroscopy be able to discern between the two products? Explain with representative bands from each compound.

---

## Procedure:

### Week 1: Synthesis

- To a 25 or 50 mL Erlenmeyer flask add ~100 mg of fluorene (record exact weight) and 10M NaOH (aq.) (5 mL).
- Add toluene (5 mL) and the larger stir bar. Stir vigorously until all solids dissolve. Add 3 drops of Aliquat 336 using a short stem pipette (viscous!).
- Stir vigorously for 30 minutes. By TLC (20:80 CH<sub>2</sub>Cl<sub>2</sub>:hexanes as eluant) ensure you have two products in a roughly 50:50 mixture (spots are roughly same size). If not, stir vigorously another 15 minutes and recheck.
- Transfer the reaction mixture to your separatory funnel. Use an additional 5 mL of toluene to rinse the Erlenmeyer.
- Separate the organic from the aqueous layer.
- Wash the organic layer with 1.5 M HCl (3 x 10 mL) followed by brine (1 x 10 mL). "Brine" is sat'd NaCl solution.
- Dry the resulting organic layer over calcium chloride. After five minutes, decant the remaining toluene/product solution into a small clean beaker (tared). Allow the toluene to evaporate until the next laboratory period.
- **Next lab you will need a set of clean, dry test tubes and two small jointed round bottomed flasks—please take this opportunity to clean them!**

### Week 2: Column Chromatography

- Weigh your crude product.
- Prepare the column by the "Dry Adsorbent Method" (Sec. 17.4, Mohrig) using toluene as the mobile phase and alumina (activity grade III) as the stationary phase. The total height of the stationary phase should be about 8 cm. Apply a 1 cm layer of sand to the top of the stationary phase.
- Dissolve your crude product mixture from the previous week in 1-2 mL of 50:50 CH<sub>2</sub>Cl<sub>2</sub>:toluene.
- Analyze this solution by TLC plate using 20:80 CH<sub>2</sub>Cl<sub>2</sub>:hexanes as the eluant.
- Using a pipette, gently add the remainder of the crude product solution to the top of the column. Allow to "soak in".

- Open the stopcock and gently add the first eluting solvent (hexanes) so as to not disturb the top of the column. Continuously add hexanes to the column while monitoring the progress of the solvent front. When solvent has reached the stopcock, begin collecting fractions.
- Collect 5 x 3 mL fractions in clean test tubes. Stop the elution. Analyze the fractions simultaneously on a TLC plate vs. the 1% fluorene standard. Determine which fractions contain fluorene. If fraction 5 still contains fluorene collect another 3 x 3 mL fractions. Analyze again vs. the 1% fluorene standard and ensure all fluorene has been eluted.
- Begin to use 20:80 CH<sub>2</sub>Cl<sub>2</sub>:hexanes as the eluant. Elute the yellow band (what is it?) into a single, clean dry beaker.
- In one clean, dry round bottomed flask (tared) concentrate the combined fractions analyzed by TLC that contained fluorene. Record the weight.
- In a separate clean, dry round bottomed flask (tared) concentrate the fraction that contained the yellow band. Record the weight.
- Analyze both materials by IR spectroscopy using Nu-jol®.

---

## Clean up

- Column packing should be placed in the large collection container labeled "alumina waste"; use the bulb provided to eject the material from the column.
- Any remaining solvent waste or excess eluting solvents should go into the halogenated waste container.
- All aqueous extracts go down the drain.

---

## Post-Lab Questions

1. How is column chromatography related to TLC? Gas chromatography? Be sure to discuss mobile phase, stationary phase, sample size, sample type in your answer.
2. How is R<sub>f</sub> in TLC related to elution order in column chromatography?
3. What is the elution order of the products of this reaction. Explain based on their structures.

