**Experiment VIIIC: Selective Dehydration of t-Amyl Alcohol**

## Background Reading

Zanger, M. and McKee, J.R. Small Scale Syntheses.
pp 47-50 (Gas Chromatography).
McMurry, J. Organic Chemistry, 9th Ed. pp 335-6 (Zaitsev's rule),
pp 373-6 (interpreting infrared spectra), pp 376-8 (IR for alkenes),
pp 546-7 (E1 dehydration of alcohols and Fig 17.6).
[In the 8th Ed., see pp 397-8, 438-43, and 641-2.]
Zubrick, J.W. The Organic Chem Lab Survival Manual.
Chapters 32 and 34 (GC and IR).

## Key Words

E1 Reaction, Dehydration Reaction, Zaitsev's Rule, Chromatography, and Flame Ionization Detector

## Experimental Data

- Include the net chemical equation and a diagram of the carbocation mechanism in your Reaction Mechanism section.
- Describe infrared spectroscopy and diagram the gas chromatograph in your Apparatus section.
- Determine theoretic and actual yield values based on combined total for both alkenes.
- Interpret the gas chromatogram and/or the infrared (IR) spectrum in your Data section. Include the major/minor product absorptions for each.

## Apparatus

Items in kit
25-mL round-bottom flask, 50-mL round-bottom flask, glass stopper
distillation head (three-way adapter), thermometer adapter (glass with rubber), condenser
vacuum adapter (leave side nozzle open)

Items not in kit
thermometer, two water hoses, two stands with clamps
warm water bath (400-ml beaker), ice-water bath (250-ml beaker)
25-ml Erlenmeyer flask, #0 rubber stopper
gas chromatograph (GC) and/or infrared (IR) spectrophotometer

**Substances**

10 g 2-methyl-2-butanol (t-amyl alcohol)
10 ml concentrated phosphoric acid ($H_3PO_4$)
0.2 g drying agent (anhydrous $MgSO_{4(s)}$ or $CaCl_{2(s)}$)
boiling stone

**Procedure**

1. Weigh a 50-ml round-bottom flask on a cork ring. Add 10 g of 2-methyl-2-butanol (t-amyl alcohol) to the flask. Use the alcohol's density (0.805 g/ml) to determine the approximate volume needed. Record final mass and determine the mass of the alcohol. Place flask in hood and add 10 ml of phosphoric acid. Mix completely. Use a magnetic stir bar if necessary. Add one boiling stone to the mixture.

   **Caution** – Concentrated $H_3PO_4$ solutions are corrosive and cause acid burns. Use gloves and avoid all contact with skin, eyes, and nose.

2. Assemble simple distillation apparatus for the flask, including a water-cooled condenser. Use a warm water bath (60 - 70 $^oC$) in a 400-ml beaker for the heat source. Use a 25-ml round-bottom flask as the receiver, and cool it with an ice-water bath in a 250-ml beaker.

3. Collect the product that distills with the head temperature between 35 and 45 $^oC$. Ensure distillation head temperature does not go above 50 $^oC$. Adjust bath temperature so that the distillation rate is no more than one drop every five seconds.

4. Add 0.1 to 0.2 g of anhydrous $MgSO_{4(s)}$ or $CaCl_{2(s)}$ to dry the product. Put a glass stopper in the flask, and leave contents in flask for 10 minutes to dry. Add more drying agent and dry for 5 more minutes if the solid appears slimy or clumps.

5. Decant liquid into a weighed 25-ml Erlenmeyer flask, and then weigh the product. Put a #0 rubber stopper in the flask.

6. Bring product to the instrument room for analysis with a gas chromatograph (GC) and/or an infrared (IR) spectrophotometer. GC will show the product distribution, as peak size is proportional to the amount of a component in the mixture. IR will show an absorption at 800 - 850 $cm^{-1}$ for the trisubstituted major product, and an absorption at 890 $cm^{-1}$ for the disubstituted minor product. Compare the relative sizes of the two IR absorptions. Refer to Figure 12-22 in McMurry, 9$^{th}$ Ed.

**Waste Disposal**

Place all solutions and used drying agents in appropriately labeled waste container(s).

**Post-Lab Questions**

1. Other than 2-methyl-2-butene and 2-methyl-1-butene, another alkene isomer could potentially form by a carbocation rearrangement. Diagram the carbocation rearrangement and the third alkene product. Name this alkene also. Then, explain why it is not actually observed in this lab.

2. Describe how IR and GC can complement each other. That is, describe what each will identify and will not identify.

3. How can you visibly determine when the acid is completely mixed with the alcohol? That is, what would you expect to see if they were not mixed? Also, what temperature change occurs when mixing the acid with the alcohol (which initially acts as a base), and why does it occur?

4. Why is a warm water bath generally better than a heating mantle for the product distillation in this experiment? What can happen to your product if the water bath boils?

5. Refer to last page of class notes for Ch 12 Structure Determination (IR). If the IR spectrum shows a strong, broad absorption at 3400 $cm^{-1}$, what two substances may be present? How is the product separated from each of these?