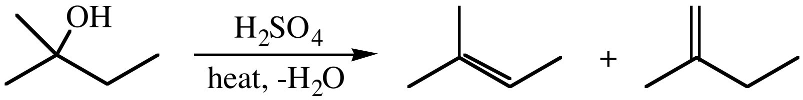
Synthesis and GC analysis of 2-methyl-1-butene and 2-methyl-2-butene

Reading Assignment: Review distillations, extractions, and drying reagents; Ch. 30



	2-methyl-2-butanol (t-amyl alcohol)	2-methyl-2-butene	2-methyl-1-butene
bp	102°C	38.6°C	31.2°C
d	0.805	0.662	0.662
MW	88.15	70.14	70.14

Experimental Procedure

Pour 25 mL of cold 6M H_2SO_4 solution into a 100 mL round bottom flask, add 18 mL of 2-methyl-2-butanol and a boiling chip. Mix thoroughly and then mount flask for distillation using 2 ice water cooled condensers in series with a 50 mL round bottom flask as the receiver for the distillate. The round bottom flask should be cooled in an ice-water bath in order to collect the very volatile olefins. Carefully heat the reaction mixture with a heating mantle. The top layer in the reaction flask should be distilled over. When you think all the top layer has been distilled over, continue distilling for another minute before stopping (your distilling temperature should be no more than 50˚C). Transfer the cold distillate to a pre-cooled separatory funnel (add some ice water to the separatory funnel and shake it, then pour out the ice water just before adding your alkene mixture), add 5 mL of cold 2M NaOH solution and shake to remove traces of acid that co-distilled over. Draw off the bottom aqueous layer and discard. Dry the alkene layer over anhydrous magnesium sulfate in a 25 mL erlenmeyer flask.

The solution will have to be distilled a second time. Filter off the magnesium sulfate by placing a small cotton wad (the size of an eraser on a lead pencil) in the long stem funnel and allowing the alkene phase to filter into a 15 or 25 mL round bottom flask (depending on how much solution you have). Add a boiling chip, clamp the flask, and set up the distillation apparatus as before using two condensers in a series with ice cold water in the condensers. Weigh a 15 mL round bottom flask, record the weight, and use it as the receiver. The 15 mL round bottom flask should be cooled in an ice-water bath in order to collect the very volatile alkenes. Distill again using the heating mantle to heat the distilling flask. Collect the product that distills over between 37-43°C. Record the weight of the final product mixture. Calculate the total experimental yield.

Gas Chromatography (GC) Analysis

We will analyze our products on the GC-MS as before. To make up a sample for the GC machine, add 1 mL of methanol to the GC vial. Then add one or two drops of your final product. We will use the "Alkenes" method on the GC-MS machine.

Questions:

1. Which product is the major product? WHY is it the major product? Hint: almost all E1 reactions of alcohols to form alkenes are under <u>**thermodynamic control**</u>.

2. Use the heats of hydrogenation for the two products (both are listed in Wade in the table of heats of hydrogenation in chapter 7) to determine the exact difference in stability (ΔG) between the two products. Then plug this ΔG and the average temperature of your distillation (in K!) into the equation: $Keq = 10^{-\Delta G/RT}$. Compare the ratio of isomers you obtained from your GC analysis to the ratio predicted from the theoretical Keq you just calculated. If the reaction was under true thermodynamic control, these ratio's should be fairly close. How close are they?

3. What would be the major product if the starting material had been 3-methyl-2-butanol? Would you expect the <u>**ratio**</u> of the products to be the same or different than your experiment using 2-methyl-2-butanol as the starting alcohol.

4. What would be the major product if our starting alcohol had been 2-methyl cyclohexanol?