Synthesis and GC Analysis of 2-Methyl-1-Butene and 2-Methyl-2-Butene


Introduction:
Alcohols undergo dehydration under acidic conditions to form the corresponding alkenes. When 2-methyl-2-butanol is heated in the presence of acid, two alkenes are produced, 2-methyl-2-butene and 2-methyl-1-butene. The two products are not formed in the same amount. From your knowledge of the mechanism of the above reaction, you should be able to predict which alkene will be the major product. You will verify this by analyzing the gas chromatography (GC) data for the product mixture. [Review distillations, extractions, and drying reagents in TOC.]

Procedure: Work in pairs for this experiment.

Part I. Synthesis of Alkenes
Pour 15 mL of cold 6 M H$_2$SO$_4$ solution into a 50 mL round bottom flask, add 10 mL of 2-methyl-2-butanol and a boiling chip. Mix thoroughly and then mount flask for distillation using two ice-water cooled condensers in series with a 25 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 (alkenes). Very carefully heat the reaction mixture with a heating mantle. The top layer in the reaction flask should be distilled over. Closely monitor the temperature of the distillation temperature. When you think all the top layer has been distilled over, continue distilling for another minute before stopping. Transfer the cold distillate to a pre-cooled separatory funnel, add 3 mL of cold 2 M NaOH solution and shake to remove traces of acid that co-distilled over. Draw off the bottom aqueous layer and discard. Extract the organic layer once with 3 mL of cold water. Dry the alkene layer over anhydrous magnesium sulfate in a 10 mL Erlenmeyer flask. (Keep the flask stoppered; products are volatile.)

The solution will have to be distilled a second time. Filter off the magnesium sulfate by placing a small cotton wad (the size of a pencil eraser) in the long stem funnel or Pasteur pipet and allowing the alkene phase to filter into a 15 mL round bottom flask. 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 10 mL round bottom flask, record the weight, and use it as the receiver. The 10 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 mantel 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 percent yield. In Part II, you will obtain a GC
of the mixture. Time permitting, you may prepare the sample for GC analysis as described in the
next part. Tightly wrap the cap with parafilm and store your product in the freezer until the next
period.

**Part II. Gas Chromatography (GC) Analysis:**
Prepare a sample for GC in a vial using methanol as the solvent: transfer 2 to 3 drops of your
dry product mixture into a clean GC vial; add enough solvent to the vial until it is about 2/3 full.
Set-up the gas chromatography-mass spectrometry (GC-MS) instrument for injection of your
sample. (See separate instructions.) Work-up the data using the GC-MS software as necessary.

Two main peaks will appear on your gas chromatograph. Record the retention times and areas
for the two peaks. The areas under each peak are proportional to the relative amounts of each
compound. Based on the measured areas, calculate the percent of 2-methyl-2-butene in the
mixture and the percent 2-methyl-1-butene in the mixture. (In general, the compound with the
lower boiling point has a shorter retention time.)

**Questions:**
1. Draw the mechanism for the formation of both products of this experiment. Identify the
   expected major product and explain why it is major.
2. Repeat the question above for 3-methyl-2-butanol.

**Conclusion:** Does your prediction for major product (Question 1 above) agree with peak areas
and retention times in your chromatograph? Why or why not? Summarize your results and
comment on any special considerations that should be observed with this experiment.