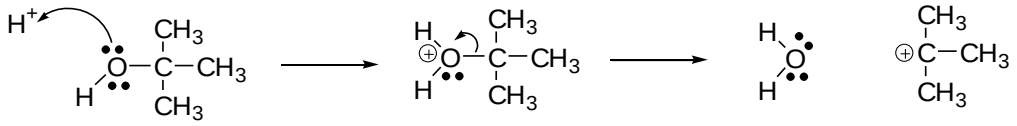
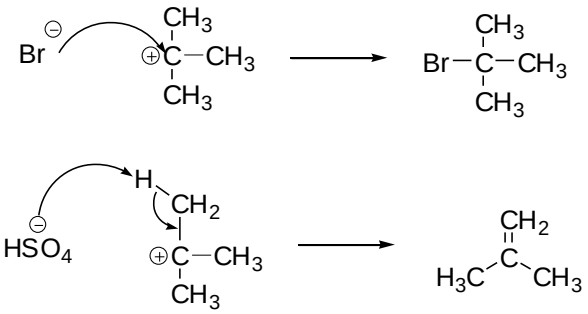
Dehydration of *t*-Amyl Alcohol (2-Methyl-2-Butanol)

Background

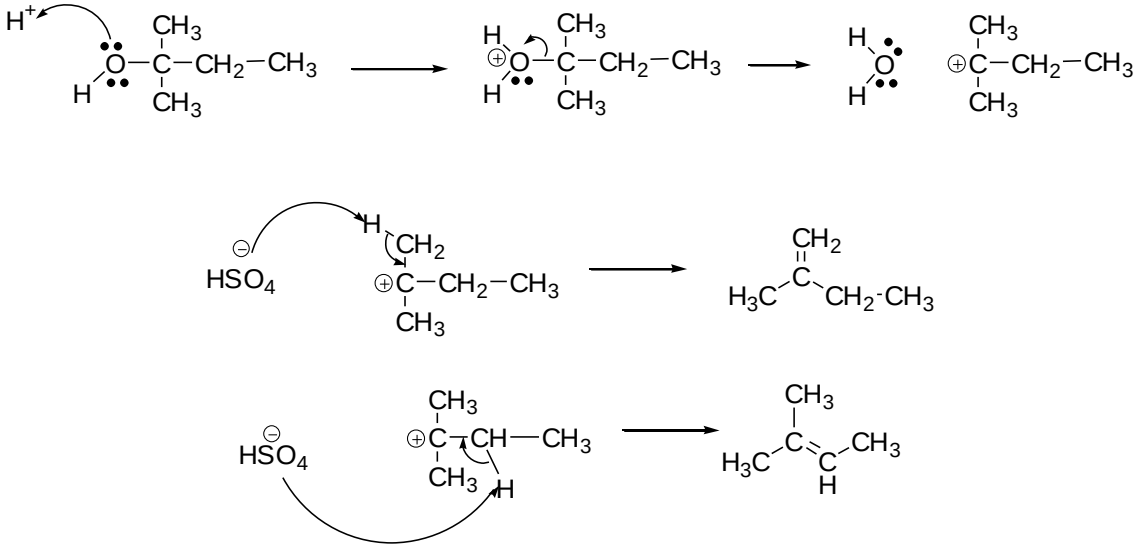
In the presence of a strong acid, alcohols will protonate. This turns a bad leaving group (hydroxide) into a good leaving group (water). After protonation of a tertiary alcohol, a water molecule leaves with a pair of electrons creating a carbocation.



If a good nucleophile is present, such as: Cl^-, Br^-, I^- (which would come from using strong acids HCl, HBr and HI respectively) it will form a new bond at the positive carbon. This is a nucleophilic substitution reaction. If the acid's conjugate base is a poor nucleophile such as HSO_4^- (which would come from the strong acid H_2SO_4), elimination takes place. To complete the elimination, the poor nucleophile removes a proton on a carbon next to the carbocation and the electrons from the C-H bond become the new π bond, forming an alkene. The strength of the nucleophile determines if substitution or elimination takes place.



More than one elimination product can be formed if there are non-equivalent hydrogens next to the carbocation, as is the case with the alcohol in this lab.



In this lab you will dehydrate *t*-amyl alcohol to form a mixture of the alkenes above and then determine the ratio of products using GC analysis.

Procedure

Reaction: Add about 15 mL (measured precisely) of *t*-amyl alcohol to a 50 mL round bottom flask (the flask will become your stillpot). Cool the pot in an ice-water bath and slowly add, with swirling, about 5 mL of 9 M sulfuric acid. Add two boiling chips then clamp the flask to your ring stand. Do not leave the flask someplace it can be knocked over.

Set up a fractional distillation with a foil wrapped Vigreux column. Use a 25 mL round bottom flask as the receiver and cool the receiver in an ice bath so that volatile products do not evaporate. Have your instructor check your setup, then heat with a heating mantle at 50% power. When the pot begins to boil, start checking the temperature. Stop distilling when 1/2 to 2/3 of the pot contents are gone, OR, when the temperature abruptly begins to rise after a period of collection.

Isolation and Purification: The 25 mL receiving flask will now become the still pot for a simple distillation to purify the product. Detach the old receiver/new still pot and add about 1 gram of anhydrous Na_2SO_4 (to remove water impurities) and two pellets of sodium hydroxide (to remove acid impurities). Use the cleaned and dried 50 mL round bottom flask as your new receiver and pre-weigh it (on a cork ring) before it is attached. Remove any water droplets from the condenser, vacuum adapter and still head by rinsing with acetone before you set up the simple distillation. Cool the receiver as before. Distill your product(s) until the pot is almost dry. Record the boiling point range of your product(s) and their weight. Transfer the alkenes to a pre-weighed vial and record the mass of the product. Cap and seal the vial with paraffin until you are ready to take the GC. Store the sample in the refrigerator if you are taking the GC on a different lab day.

Characterization: Characterize by the boiling point range of the products during the simple distillation. Calculate total percent yield of all alkenes (the products have the same molar mass). Determine the ratio of products based on GC analysis. No GC correction factor is needed for these two products since they have identical masses and polarities.

Chemicals: t-amyl alcohol, 9M sulfuric acid, anhydrous sodium sulfate, sodium hydroxide (s).

Waste
- Distilled samples can go in the labeled organic waste jug.
- Residue in still pots should be **carefully** washed down the sink – splashed liquid may be caustic!!