Purpose

The goal of this experiment is to analyze the unexpected product formed from a reaction of 2,3-dimethyl-2,3-butanediol with sulfuric acid.

Learning Objectives

Run a reaction using simple distillation set-up.

Use a separatory funnel in the reaction work-up.

Draw a curved arrow mechanism for the reaction that actually occurred.

Use IR and NMR to analyze the product of the reaction.

Equipment

- 25-mL beaker
- 50-mL beaker
- Erlenmeyer flask
- Stir plate
- Thermowell
- Stir bar
- FTIR instrument
- NMR instrument

Chemicals

- 2,3-dimethyl butanediol
- 3M Sulfuric acid
- Saturated aqueous sodium chloride
- Anhydrous sodium sulfate

Introduction

Student Reaction

A student was trying to synthesize 2,3-dimethyl-1,3-butadiene to use in a polymerization reaction. In order to do this, 2,3-dimethyl-2,3-butanediol was reacted with a sulfuric acid catalyst Figure 2.1. Normally, an alcohol in the presence of an acid catalyst like H_2SO_4 will undergo an acid-catalyzed dehydration reaction, forming an alkene.

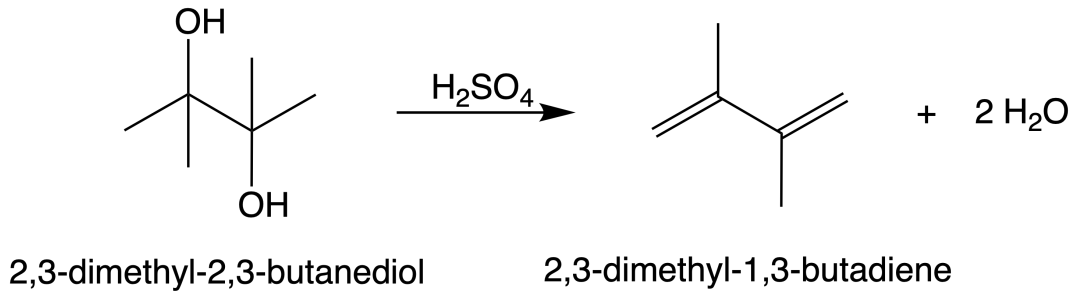

Figure 2.1: Expected product in the acid-catalyzed dehydration of 2,3-dimethyl-2,3-butanediol

However, this student's results did not yield 2,3-dimethyl-1,3-butadiene. Instead a product with the formula $C_6H_{12}O$ was obtained as shown in Figure 2.2.

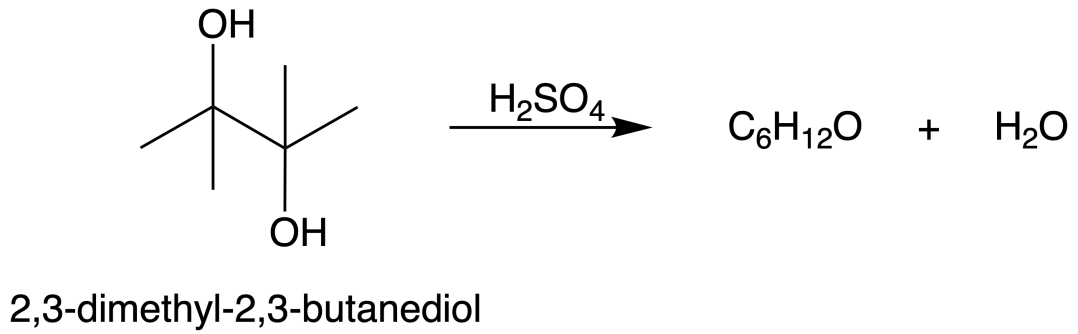

Figure 2.2: Observed Reaction of 2,3-dimethyl-2,3-butanediol with sulfuric acid catalyst

In this experiment, you will be repeating this reaction to determine the identity of the product with the molecular formula $C_6H_{12}O$. In order to determine the identity of the product, you will be using a combination of IR and NMR spectroscopy.

A comparison to the starting diol (Figure 2.3) can be helpful in the analysis of the reaction product.

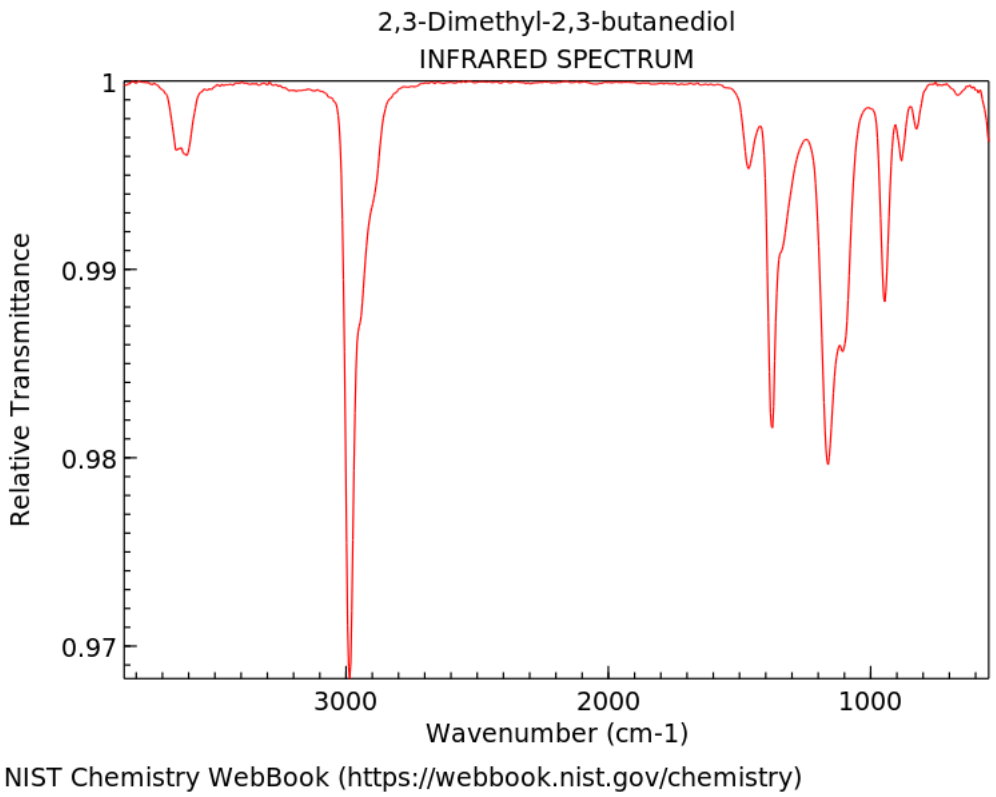

Figure 2.3: IR of the reactant, 2,3-dimethyl-2,3-butanediol

Once you have determined the identity of the product, you will then propose a curved arrow mechanism for the reaction that actually occurred. It might help to consider how an alcohol (ROH) will behave in in the presence of an acid. In addition, you will consider how the formation of a carbocation and possible carbocation rearrangements can help in proposing a reasonable mechanism for the reaction.

Procedure

Safety Precautions

Safety goggles are required!

All work should be performed in the fume hood.

2,3-Dimethyl-2,3-butanediol is an irritant.

Sulfuric acid is toxic, oxidizing, and corrosive.

Sodium sulfate is irritating and hygroscopic.

Reaction of 2,3-Dimethyl-2,3-butanediol

1. Assemble a simple distillation apparatus with a 50-mL round-bottom flask as the boiling flask and a 25-mL round-bottom flask as the receiver that is cooled in ice.

2. To the boiling flask, add 60.0 mmol of 2,3-dimethyl-2,3-butanediol, followed by 20 mL of 3M sulfuric acid, and a stir bar.

3. Using a thermowell and a stir plate, gently heat the reaction mixture.

4. Ensure slow distillation of the liquid into the receiver until it is about half full (about 12 mL). The temperature of the distillate should be around 100°C.

Separation

5. Transfer the distillate to a separatory funnel. Rinse out the receiving flask with about 2 mL of distilled water and add it to the separatory funnel.

6. Allow the layers to separate out in the separatory funnel. Take out the aqueous layer and keep the organic layer in the separatory funnel.

7. Wash the organic layer twice with 5-mL portions of saturated aqueous sodium chloride.

8. Transfer the organic layer to an Erlenmeyer flask and dry it with anhydrous sodium sulfate. Filter to collect the dry organic layer.

9. Using a simple distillation set-up, distill the organic layer to collect pure product. Record the boiling point.

Analysis

10. Measure and record the mass of your product.

11. Record IR spectrum of the product.

12. Obtain proton NMR spectrum of the product

13. Discard the remaining product in the appropriate waste container. Clean and wash all work areas and glassware.

Results

Calculation of Yields

Amount of reactant used (grams) _____

Amount of reactant used (moles) _____

Space for calculations:

Product obtained (grams) _____

Product obtained (moles) _____

Space for calculations:

Product theoretical yield _____

Space for calculations:

Product percent yield _____

Space for calculations.

Is your percent yield within reason of what you would expect? Explain your answer.

IR Analysis

See *(Click here)* for more help on interpreting IR spectroscopy.

Complete the table with 3 Major IR peaks

Table 2.1: IR Analysis

IR Peak, cm^{-1}	Bond Type

Compare your spectrum to reference spectra. *(Click here)* to access SDBS database of spectra.

Use 1-2 key peaks to justify what compound(s) that you think are present.

Does this suggest that your reaction worked? Use 1 or two key IR peaks to justify your answer.

What does this IR data indicate about the purity of the product? Use 1 or two key IR peaks to justify your answer.

^{1}H NMR Analysis

See *(Click here)* for more help on interpreting ^{1}H NMR spectroscopy.

Complete the following table to analyze the NMR peaks in ppm.

Table 2.2: ^{1}H NMR Analysis

Chemical shift, ppm	Integration	Multiplicity	Partial structure

Compare your spectrum to reference spectra. *(Click here)* to access SDBS database of spectra.

Use three key peaks to justify what compound(s) that you think are present.

Does this suggest that your reaction worked? Use three key peaks to justify your answer.

What does this NMR data indicate about the purity of the product? Use three key peaks to justify your answer.

Questions

1. Propose a structure for the actual product of this reaction.

2. Draw a mechanism for the reaction leading to your proposed product.

3. Draw a balanced equation for the reaction of 2,3-dimethyl-2,3-butanediol with sulfuric acid.

4. If you were to take ^{1}H NMR spectrum on your starting material (2,3-dimethyl-2,3-butanediol), how many signals will you expect? Explain your answer.

5. In the ^{1}H NMR spectrum of your product, which signal is more deshielded and why is that so considering the structure of the product?