Dehydration of 2-Methyl-cyclohexanol

In this experiment students demonstrate the acid-catalyzed dehydration of *cis*- and *trans*-2-methyl-cyclohexanol to form a mixture of 1-methyl and 3-methyl-cyclohexene. Samples of the products are tested for unsaturation as well as analyzed by gas chromatography. Analysis by gas chromatography helps determine the percent composition of each product. The major product indicates the more stable carbocation formed during the intermediate step.

ESTIMATED TIME

We estimate that this experiment can be completed in one, 3-hour class period.

TIPS

1. In the Electronic Resources you will find PDF and word-processing files of the student experiment. You can print the PDF, distribute it to students electronically, or post the file to a password-protected class web page or learning management system. Edit the word-processing file if you would like to tailor the experiment to suit your equipment and students. Sign in to your account at **www.vernier.com/account** to access the Electronic Resources.

2. As an alternative to using the Peak Analysis feature of the software, your students can manually determine the retention time for a compound by using the Examine feature. Because of the manner in which the Mini GC operates, the retention time is the x-value, in minutes, at the maximum y-value of the peak. If you want students to use the Examine feature, consider modifying the student version of the experiment.

3. The temperature-pressure profile is set so that each data collection run will last 20 minutes. You may remind your students that some of the substances will have passed through the column and detector well before 20 minutes and they can stop the data collection early to save time.

4. It is important that students inject a consistent volume for each test.

5. It is helpful to provide your students with a pure sample of 1-methyl-cyclohexene for them to run in the Mini GC for comparison. It is best to dilute this 2:1 hexane to 1-methyl-cyclohexene.

6. To optimize the reproducibility of your retention times, it is best practice to let the Go Direct Mini GC return to 45°C between each trial.

HAZARD ALERTS

The chemical safety signal words used in this experiment (**DANGER** and **WARNING**) are part of the Globally Harmonized System of Classification and Labeling of Chemicals (GHS). Refer to the Safety Data Sheet (SDS) that came with the chemical. These can also be found online from the manufacturer. See the Preface for additional chemical safety information.

Calcium chloride, solid, $CaCl_2$: **WARNING**: Do not eat or drink when using this product—harmful if swallowed. Causes serious eye irritation.

Dichloromethane, CH_2Cl_2, **DANGER**: Causes skin irritation. Causes serious eye irritation. May cause drowsiness or dizziness. May cause cancer. May cause damage to organs through prolonged or repeated exposure.

n-Hexane, C_6H_{14}: **DANGER**: Keep away from heat, sparks, open flames, and hot surfaces—highly flammable liquid and vapor. Do not eat or drink when using this product. Avoid breathing mist, vapors, or spray. May be fatal if swallowed and enters airways. May cause damage to organs. Causes skin and eye irritation. May cause drowsiness or dizziness. Suspected of damaging fertility or the unborn child. Do not handle until all safety precautions have been understood. Use personal protective equipment as required.

2-Methyl-cyclohexanol, $C_7H_{14}O$: **WARNING**: Flammable liquids. Flammable liquid and vapor. Harmful if swallowed. Causes serious eye irritation. Harmful if inhaled.
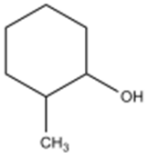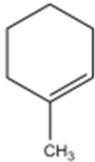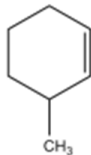
1-Methyl-cyclohexene, C_7H_{12}, **DANGER**: Highly flammable liquid and vapor. Causes skin irritation. Causes serious eye irritation.

3-Methyl-cyclohexene, C_7H_{12}, **DANGER**: Highly flammable liquid and vapor. Causes skin irritation. Causes serious eye irritation.

Sodium chloride, saturated, NaCl: **WARNING**: May be harmful if swallowed. Skin and eye irritant.

Sodium hydroxide, 9.0 M, NaOH: **DANGER**: Causes severe skin burns and eye damage. Do not breathe mist, vapors, or spray.
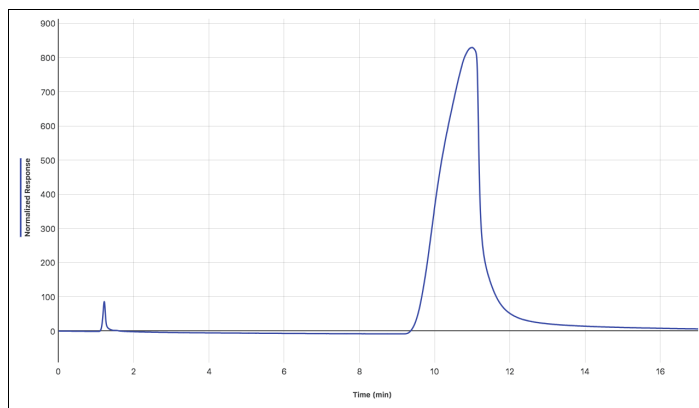
PRE-LAB ACTIVITY

Compound	Boiling temperature (°C)	Molar mass (g/mol)	Chemical structure
2-methyl-cyclohexanol	163–166	114.18	
1-methyl-cyclohexene	110	96.17	
3-methyl-cyclohexene	104	96.17	

SAMPLE DATA

Compound	Bromine test result	Baeyer test result	Retention time (min)
2-methyl-cyclohexanol	unsaturated	unsaturated	10.99
1-methyl-cyclohexene	saturated	saturated	4.62
3-methyl-cyclohexene	saturated	saturated	3.51

SAMPLE CHROMATOGRAMS

Figure 1 Sample chromatogram of 2-methyl-cyclohexanol

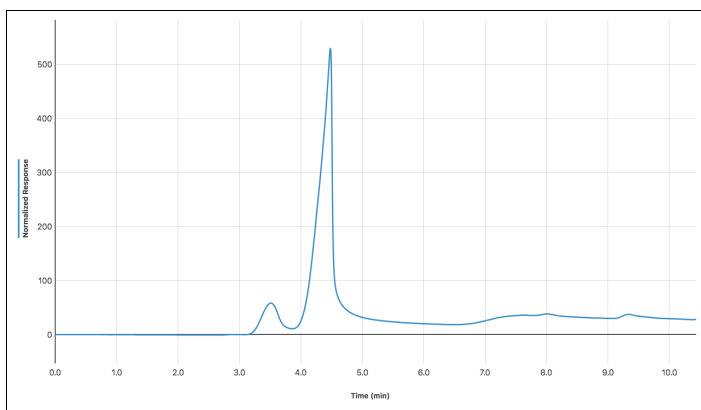*Figure 2 Sample chromatogram of pure 1-methyl-cyclohexene*

Figure 3 Sample chromatogram of crude product, containing 1-methyl-cyclohexene and 3-methyl-cyclohexene

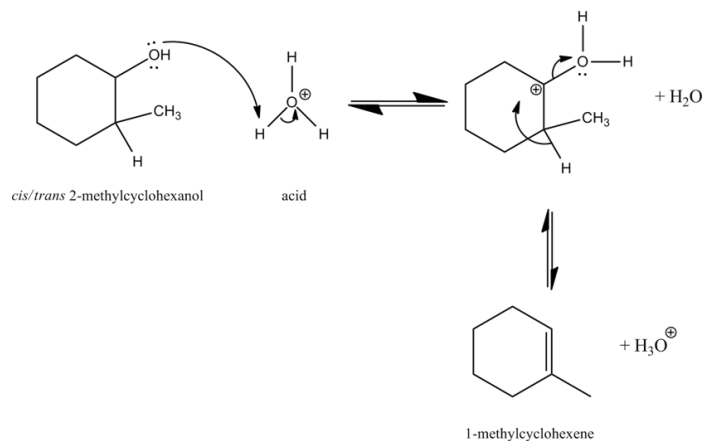## ANSWERS TO ANALYSIS QUESTIONS

1. Theoretical yield:

   5.0 mL 1-methyl-cyclohexanol × (0.919 g / 1 mL) × (1 mol 1-methyl-cyclohexanol / 1 mol product) × (96.17 g / 1 mol product) = 3.87 g product
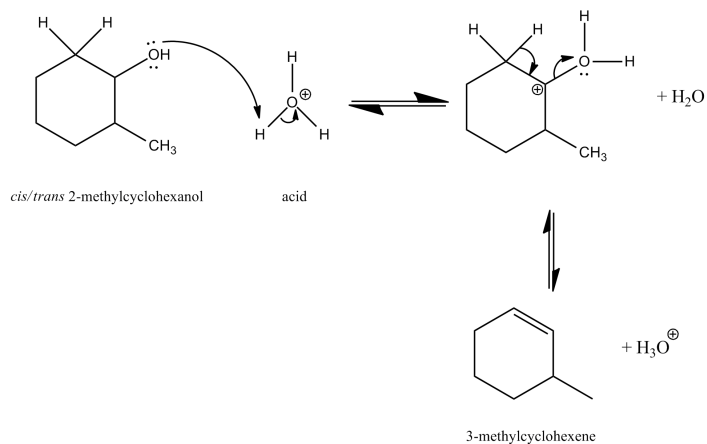
   Actual yield: 0.99 g product

   Percent yield: (0.99 g / 3.87 g) × 100 = 25.5% yield

2. The major product is 1-methyl-cyclohexene. This is evident from the gas chromatogram. 1-methyl-cyclohexene has a higher boiling point compared to 3-methyl-cyclohexene; therefore, it is the second peak with the later retention time to appear in the chromatogram. This can be confirmed by running a pure sample of 1-methyl-cyclohexene. The second peak was much larger, indicating that it is present in a larger quantity. We can confirm this by comparing the peak areas for the two products.

3. Formation of 1-methyl-cyclohexene

Chromatography Experiments with Go Direct Mini GC

4. Formation of 3-methyl-cyclohexene

<p style="text-align:center">*cis/trans* 2-methylcyclohexanol   acid</p>

<p style="text-align:center">3-methylcyclohexene</p>

5. Since 1-methyl-cyclohexene is the dominant product, the carbocation generated in its formation is more stable.