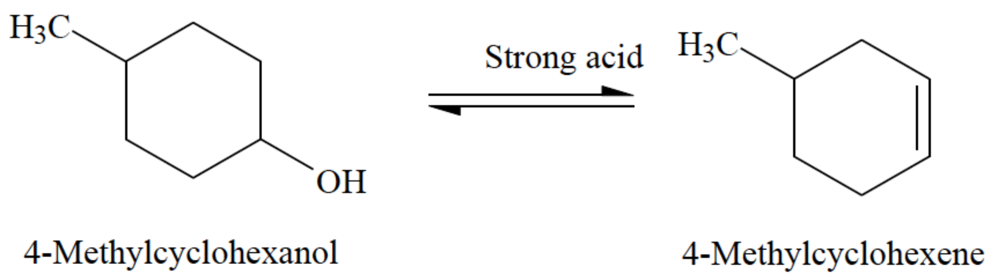
EXPERIMENTAL AND LEARNING OBJECTIVES

Alcohol dehydration is an acid-catalyzed reaction performed by strong, conc. acids. Sulfuric acid would cause extensive charring in this process so phosphoric acid is preferred.

This reaction is in fact an equilibrium reaction, but as product is distilled on forming, the equilibrium shifts in favor of forward reaction.

Since the product is distilled from the reaction mixture, this reaction nicely demonstrates a reaction in which synthesis, isolation and purification all occur in one step.



4-Methylcyclohexene and water have **very similar boiling points** and the distillate is likely to contain both water and 4-methylcyclohexene.

There is also likely to be a trace amount of acid. Therefore handle with care.

PHYSICAL DATA:

	Molar Mass	Boiling Pt.	Density
	(g/mole)	(°C)	(g/mL)
4-Methylcyclohexanol	114.2	170	-
4-Methylcyclohexene	96.2	~104	-
Phosphoric Acid		-	1.88

DIRECTIONS

- A setup for distillation may be on display:

SAFETY:

Methylcyclohexene is a strong lachrymator. Avoid contact with fumes.

PROCEDURE :

1. (Optional) One student (or instructor) should make a COPY of the spreadsheet to enter all data. This COPY must then be shared out to the rest of the class. Again, this is optional[1].

2. Using a measuring cylinder, place 1.5 mL of 4-Methylcyclohexanol in a **preweighed** 3 mL conical vial and **re-weigh** to determine **exact mass** of alcohol used.

3. Next, using an Eppendorf micropipette (located in the back of the room), add 0.400 mL Conc. phosphoric acid to your 3 mL conical vial.

4. Finally add magnetic stir vane.

5. Assemble apparatus for distillation using Hickman head and water-cooled condenser. Don't forget to use an aluminum block for heating.

NOTE: You may be asked by your instructor to use the air condenser INSTEAD of the water-cooled condenser. Check beforehand.

6. Place a clean, capped centrifuge tube into a small beaker. The purpose of this beaker is to ensure that the sample container **does not fall over**. This sample container will be for receiving distilled products.

7. Turn on the stirrer and heater. Target an **aluminum block temperature to about ~190°C**. If you are using a water condenser, be sure to *start water circulating through condenser*.

8. When distillate collects in **Hickman head**, use a pipet to transfer to your centrifuge tube. Keep Hickman side port closed as often as possible to minimize loss of product.

9. Collect distillate until no more product formed (be careful not to overheat reaction or unreacted **4-methylcyclohexanol** will distill – boiling point: ~170 °C)

10. Rinse Hickman head with approximately 1 mL of **saturated sodium chloride solution** and transfer this to the centrifuge tube.

11. Gently mix the contents of the centrifuge tube.

12. Allow content of the centrifuge tube to separate into **layers**. Using a disposable glass pipet, carefully **remove the lower** aqueous layer and discard.

13. Add a small spatula of **anhydrous magnesium sulfate** to the remaining **organic layer** of 4-methylcyclohexene. Stir and allow to stand until liquid is clear.

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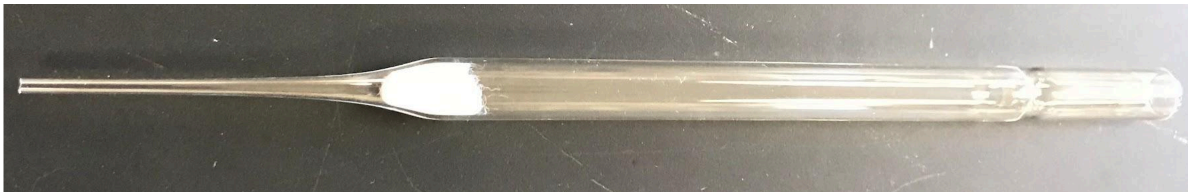
[1] Video on how to make a copy to edit a google file.

14. Pre-weigh a **capped GLASS sample vial.** This will be used as your collection vessel in the next steps.

15. Using a filter pipette (see below), filter the now dried 4-methylcylcohexene into your collection vessel.



16. Reweigh your sample collection vessel to obtain mass yield of product and percent yield.

17. When storing your sample, wrap the outside of the cap and rim of the container with parafilm.

18. Because product is an unpleasant smelling and strong lachrymator, instructor will help individual students run the FTIP:

    a. FTIR
        i. Watch the youTube on how to do this
        ii. Reference FTIR of the starting material.
        iii. Reference FTIR of the Product
    b. GC - OPTIONAL

19. We will NOT determine the boiling point.

20. Examine IR (and GC) to see whether

    a. The product is indeed 4-methyl cyclohexene
    b. Determine overall purity. (Unreacted starting alcohol is the most likely contaminant).

Disposal & Cleaning:

- **Rinse all used glassware with acetone in your fume hood before washing in the sink.**
- Transfer waste acetone wash to organic waste station.
- Clean fume hood.

Prelab:

1. Write out the entire equation for the reaction (Include names and structural formula).
(1 Point)

2. Give a curly arrow mechanism for this reaction.
(1 Point)

3. Why is the distillate likely to contain traces of **water**?
(1 Point)

4. How could **water be removed** from the distillate?
(1 Point)

5. What is the overall reaction mechanism **TYPE** for this reaction?
(1 Point)

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PostLab:

1. The reaction is an equilibrium reaction. Explain why the experiment favored the formation of the alkene product rather than the reformation of the starting alcohol?
(1 Point)

2. From your IR spectrum and your GC, comment on the purity of your final product.
(1 Point)

3. Calculate the theoretical yield and actual yield for your final product. (Show all of your working).
(1 Point)

4. The alcohol was protonated in order to make it a better leaving group. Name two other ways that alcohols can be converted to better leaving groups.

(2 Points)

a.

b.

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