EXPERIMENT

Geometric isomers

Goal

The goal of this experiment is to understand the concept of **isomerism**. During this practice, the student will identify two different molecules based on their properties. Later, a simple reaction will be carried out in an attempt to transform each molecule into its corresponding isomer. The student should be able to identify the new products based on the first observations (i.e. the properties of each molecule) and to validate the success of the transformation. The experiment will also familiarize the students with the separation technique known as suction filtration.

Materials

$\hfill \square \ 4$ small test tubes, with 2 suitable test tube stoppers	☐ Suction flask
☐ 2 test tube holders	□ Round filter paper
□ Bunsen burner	□ plastic funnel
□ 250 mL Erlenmeyer	□ stirring rod
☐ Ring stand, Iron ring, Wire gauze, Clamps and Büchner funnel	☐ Fumaric Acid, and Maleic Acid,
	□ 12 M Hydrochloric Acid
□ Rubber tubing	□ Ice

Background

Consider a set of Lego® blocks with different sizes and colors. We can combine them in numerous ways to construct many different structures. Similarly, if those blocks were atoms, we would obtain different molecules from the same set of atoms. Isomers are molecules with the same number and nature of atoms, and therefore the same molecular formula, but with different structures. Diverse structures arise from variations in the bonds between atoms and lead to new molecules, with different names and properties.

Geometric isomers are a subclass of isomers where not only the number and nature of the atoms are the same, but also the bonds. In this case, it is the spatial distribution or 3D structure that differs. This little change can lead to considerable differences in basic properties such as melting points and solubilities. In molecules with four ligands distributed around a central atom or a double bond, the isomerism can arise from the distribution of those four ligands around the molecule, leading to *cis*- and *trans*- isomers. This is the case in the proposed experiment.

The process of **isomerization** is the transformation from one molecule into its corresponding isomer. This transformation does not take place spontaneously but typically requires external energy and, in some cases, specific conditions. Energy is necessary to break certain bonds to allow the molecule to restructure into the corresponding isomer.

Procedure

Part A. Ide	entify the products by their melting point.
(– Obtain the two unknowns. One unknown should be fumaric acid (<i>white powder</i>), and the other maleic acid (<i>white powder</i>). If both unknowns show the same properties in the following experiment, be prepared to select a different unknown.
☐ Step 2: -	– Label two small test tubes as A and B.
☐ <i>Step 3:</i> -	– Put a pea-size amount of one product in test tube A, and a similar amount of the other product in test tube B.
t	– Using a striker, light the Bunsen burner. Using a test tube holder for each sample, hold test tubes closely together, in the same hand. The samples must be heated in similar conditions. Make sure you never heat a test tube with a tube stopper on as they will explode.
	– Write your results in the Results section, characterizing the melting point of each unknown by comparison as low or high.
	Good Lab Practice
	The burner should stand stable with no additional support. Coil the tubing at each end to stabilize the burner.
	✓ Work safely and avoid burns.
	Always ensure easy access to the gas valve. Remove any items around the valve, and do not set the flame on your way.
	Never leave the flame unattended. In case of accident or any risk, close the gas immediately.
t	- Start a timer. Swing the test tubes over the flame. Stop as soon as one of the two powders melts. Pay attention to the test tubes' label. Was it test tube A or test tube B? Write down the time it took to melt the isomer on the Results table.
	– Refer to the known properties of maleic acid and fumaric acid (from the prelab) to determine which product is unknown A and which is unknown B.
☐ Step 8: -	- Allow the test tubes to cool down. Discard the contents in the indicated waste containers. Clean the test tubes.
Part B. Ide	entify the products by their solubility in water.
☐ Step 1: -	- Label two small test tubes as A and B.
Step 2: -	– Put a pea-size amount of one product in test tube A, and a similar amount of the other product in test tube B.
☐ <i>Step 3:</i> -	- Add similar amounts of distilled water to each test tube. Approximately 2 mL of water should be enough.
☐ Step 4: -	- Cover the tube using the stoppers and shake them until one of the 2 powders dissolves completely in the liquid.
	– Refer to the known properties of maleic acid and fumaric acid (from the prelab) to determine which product is unknown A and which is unknown B.
	– Your result should be consistent with Part A. If not, first repeat part B to double-check if the labeling was correct. If the mistake was not in part B, then repeat part A.
	– Write your results in the Results section, characterizing the solubility of each unknown by comparison as low or high.

Part C. Isomerization reaction. Preparing the setup
Step 1: – Obtain about 1.0 grams of the most soluble sample in Part B, using the scale and a weighing boat.
Step 2: – Transfer the powder into a 250 mL Erlenmeyer flask and add around 10 mL of distilled water.
Step 3: – Now, we will prepare a setup to boil the solution that will remove the vapors from the reaction.
Step 4: – Set up a ring stand and the iron ring with the wire gauze on top. Do not put the Erlenmeyer with the solution yet. Make sure that the height of the platform is appropriate for the size of the flame.
Step 5: – Use a second, larger ring above the platform as protection.
Step 6: – To remove the vapors improvise a little hood using a plastic funnel. Connect the funnel to the vacuum line using the rubber tubing and set it with a clamp, upside down, at a certain distance over the platform.
Step 7: – Start the ventilation.
Step 8: – Show the setup to your instructor, who will make sure it is sturdy enough. In particular, the instructor will sure all vapor is removed with the funnel, if not, adjust the setup right away. You want to avoid the release of any vapor into the air.
Step 9: – Place the maleic acid solution in the Erlenmeyer, and light the burner to gently warming up the solution.
 ★ Wear goggles at all times. ★ Handle concentrated acid with care to avoid chemical burns. ★ Use latex gloves when handling concentrated acid. ★ Ensure that the mouth of the test tube is pointed away from you and any other persons around you, including those on the opposite side of the bench.
Part C. Isomerization reaction. Adding the concentrated acid
Step 1: – Make sure you wear some protective latex gloves.
Step 2: – While the solution warms up obtain 10 mL (approx.) of concentrated hydrochloric acid, HCl, in a 25 mL graduated cylinder. Use a plastic dropper to transfer the acid from its container into the cylinder. Mind this is a very concentrated acid that can hurt you.
Step 3: – Keep the cylinder all the time in the hood to minimize the release of acidic vapors.
Step 4: – When the solution starts to boil, bring the acid from the hood, and add <i>carefully</i> the acid into the solution using a plastic dropper.
Step 5: – There should be no acid left over. If so, get rid of the leftover acid right away.
Step 6: – Wait until the boiling is re-established, and make sure no vapor scapes the ventilation system.
Step 7: – Let the reaction boil for 1 minute and then turn off the gas. Continue preparing the next steps.

Part C. Isomerization reaction. Filtration

Step 1:	- Put 40 mL of distilled water in a clean 50 mL beaker. Cool down this water using an ice bath. Get ice in a large enough beaker and dip the 50 mL beaker in the ice.
Step 2:	$\!-$ Go back to the Erlenmeyer with the solid dissolved and the acid. Allow the Erlenmeyer to cool down for 10 minutes. In the meantime, obtain ice in a 600 mL beaker and add some water to the ice.
☐ <i>Step 3</i> :	– After the 10 minutes of cooling down, transfer the Erlenmeyer carefully and safely into the large ice bath.
Step 4:	– Get the suction filtration setup ready. Connect the suction flask to the vacuum and put the Büchner funnel on top.
Step 5:	– Obtain a flat, round filter paper with a diameter slightly smaller than the bottom of the funnel. Start the vacuum and add some distilled water with a wash bottle to test the suction.
Step 6:	- Filter the solution. Stir the mixture to help the precipitate to suspend and add the liquid carefully over the filter paper. Use the cold distilled water in the 50 mL beaker to remove any solid left in the Erlenmeyer and to wash the filtrate.
Step 7:	- Keep the suction on for another 5 minutes. This will help remove all the water and partially dry the filter.
Step 8:	– Turn off the vacuum and remove the filter from the funnel carefully.
Step 9:	– Keep the solid product until the end of the experiment.
Part C. Is	omerization reaction. Testing the product
Step 1:	– Identify your product following the steps in Part B.
Step 2:	- Write your results in the Results section, characterizing the solubility of the isomerization product by comparison as low or high.

STUDENT INFO	
Name:	Date:

Pre-lab Questions

Geometric isomers

1. Complete the table for the molecules Fumaric Acid and Maleic Acid

	Fumaric Acid	Maleic Acid
Molecular formula		
Draw the structure		
Solubility in water		
Melting point		
What is it used for?		
Where is it found?		

(a) Isomers

(b) Geometric Isomers

3.

4.

5.

(c)	Isomerization
(d)	Suction filtration
(e)	Filtrate
Wha	t is the difference between a single bond and a double bond
Wha	t is a <i>cis</i> isomer and what is a <i>trans</i> isomer
Draw	v the setup for suction filtration (Büchner funnel, suction flask, tubing)

STUDENT INFO	
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Results EXPERIMENT

Geometric isomers

This experiment is qualitative, which means that the results are based on observations. Record your observations for each sample during and after each part of the activity.

	Before isomerization		After isomerization
	Sample A	Sample B	
Part A. Time to melt (secs)			
Part A. Melting point (lower/higher)			
Part B. Solubility in water (smaller/larger)			
Isomer name (fumaric/maleic)			
Part C. Selected for isomerization? (yes/no)			

STUDENT INFO	
Name:	Date:

Post-lab Questions

Geometric isomers
1. A Similar amount of both unknowns are used for their characterization in Parts A and B. Why is it important to use similar amounts? What would change if the amounts used were very different?
2. How can you prove that the isomerization process was successful?
3. Why did you cool down the solution after the isomerization?
4. What would happen if hot water was used to wash the filtrate?