

EXPERIMENT 9

Properties of Carbohydrates: Solubility, Reactivity, and Specific Rotation

Materials Needed

About 3-5 g each of Glucose, Fructose, Maltose, Sucrose, Starch	
sodium bicarbonate, NaHCO ₃ (s)	15 mL 5% sucrose
25 mL 1% cooked starch suspension	5 mL 6 M HCl
50 mL Tollen's reagent	5 mL dilute iodine solution
0.15 g/mL glucose solution	unknown sucrose solution
polarimeter, spot plates, Parafilm, test tubes, hot plate	molecular model kit

Additional Reading Assignment

Smith, Chapter 24.

Background

Carbohydrates are named as such because they generally contain C, H, and O in the ratio C_n(H₂O)_n. The smallest carbohydrate molecules are called **monosaccharides** and these are the basic building blocks for larger carbohydrate molecules, the **disaccharides** and **polysaccharides**. Monosaccharides and disaccharides are collectively referred to as **sugars** because of their often-sweet taste. Polysaccharides include the **starches** and **cellulose**.

Most monosaccharides can exist as either an open-chain or a cyclic structure (Figure 1), with these two forms being in equilibrium with each other. The cyclic structure contains a new chiral carbon not present in the open-chain form. Monosaccharides are classified as **aldoses** if they contain an aldehyde group in their open-chain form, or as **ketoses** if they contain a ketone group. Glucose is an example of an aldose and fructose is a ketose. The most common disaccharide, sucrose or table sugar, is a combination of glucose and fructose (Figure 2).

Most sugars are chiral and optically active. (Chiral molecules and optical rotation were explored previously in experiment 6). In this experiment you will measure the specific rotations of D-glucose and sucrose to demonstrate this fact.

Chemical reactions of carbohydrates take place principally at the aldehyde and ketone sites. Aldoses contain the easily oxidizable aldehyde functional group and, therefore, give positive results when tested with Tollen's reagent. (See page 570 in Smith). The alpha-hydroxy ketone group of ketoses is also easily oxidized. Therefore, all monosaccharides give a positive Tollen's test. Another example of a specific test for carbohydrates is the **iodine test**, which detects the presence of polysaccharides. Iodine is adsorbed onto the surface of the polysaccharide, forming a deeply colored complex.

All di- and polysaccharides may be hydrolyzed by reaction with water (a reaction which is catalyzed by acid) **to produce monosaccharides**. Enzymes may also be used instead of acid, but they are much more specific for the particular carbohydrate.

Figure 1. Open-chain and cyclic structures of glucose.

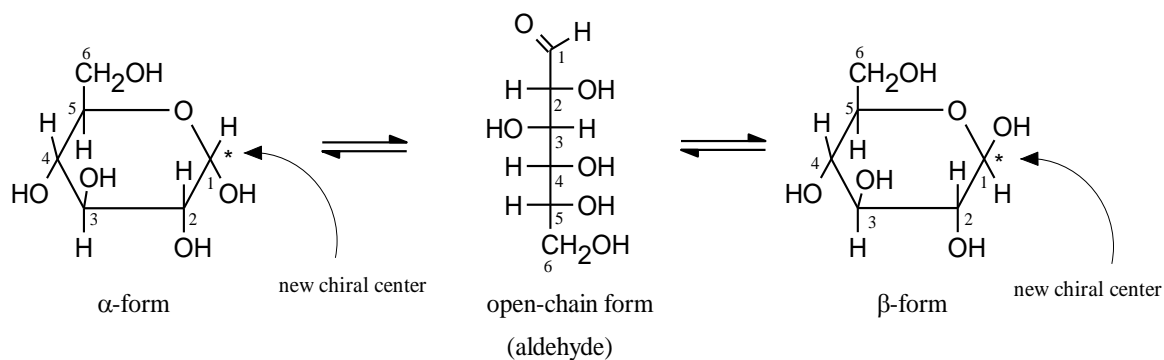
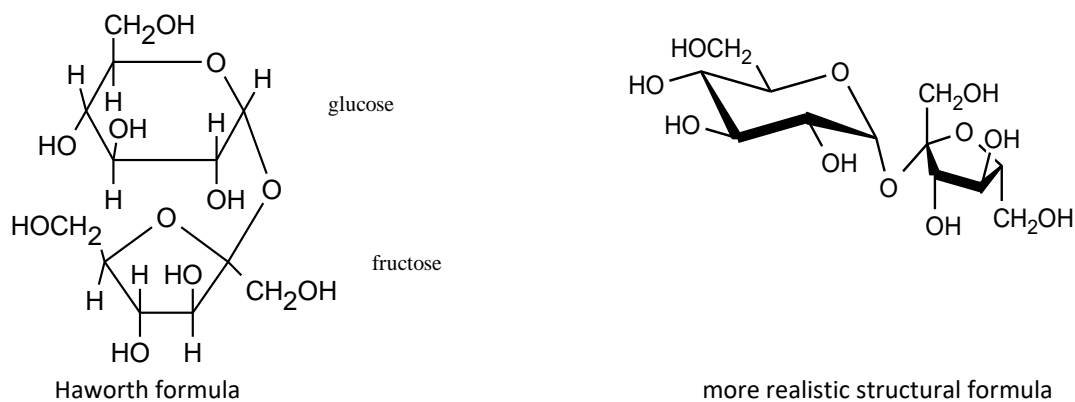


Figure 2. Sucrose: a disaccharide that is a combination of glucose and fructose.



Procedure

Part I. Physical Tests

1. Solubility.

Label five 15-cm test tubes as follows: glucose, fructose, maltose, sucrose, and starch. Add a small amount of each carbohydrate to the appropriate tube, enough to just barely cover the curved portion of the bottom. Note the color and physical form of each. Add 10 mL of distilled water to each tube, cover with a square of Parafilm, hold with your thumb, and shake vigorously. Shake each tube several times before comparing solubilities. Be sure not to mistake a slow rate of solution for insolubility.

To decide if starch (tube #5) has formed a true solution or a suspension, filter one-quarter of the liquid through a folded filter paper using a small funnel. Add 2 drops of dilute iodine solution (I_2 plus starch gives a purple color) both to the filtrate and to another one-quarter of the unfiltered solution. Observe the resulting mixtures carefully and note your observations on the report sheet. If starch is completely soluble both tests should look exactly the same. If the filtered solution gives a lighter color than the unfiltered, then the starch is only partly soluble in water and the unfiltered "solution" is really a suspension. In addition, a suspension, by definition, contains solid particles that reflect and scatter light and often this can be discerned as an opalescent appearance to the liquid. This is known as the Tyndall effect.

Save the four sugar solutions and the untreated starch solution (one-half of the original) for future tests (part II).

2. Optical Rotation.

Procedures for the use of the polarimeter will be written on the black board and demonstrated by the instructor. In addition, the T.A. can assist you with its use.

a. **0.15 g/mL Glucose Solution.** During the first part of the lab the polarimeter tube will be filled with a 0.15 g/mL glucose solution. Take three readings of the rotation of this solution. From the average rotation, the length of the tube in decimeters, and the concentration of the solution, determine the specific rotation $[\alpha]_D^{20}$ for glucose. Compare your result to the known specific rotation of glucose and determine your percent error.

b. **Unknown Sucrose Solution.** During the second half of the lab the polarimeter tube will be filled with a sucrose solution of unknown concentration. Take three readings of the rotation of this solution. Use the average observed rotation, the length of the tube in decimeters, and the known specific rotation of sucrose to determine the concentration of the solution.

Part II. Chemical Tests

1. **Hydrolysis of Starch.** On a spot plate, place 1 drop of dilute iodine solution in each depression. In a large test tube, place 20 mL of 1% cooked starch suspension. Heat in a boiling water bath, and when hot, add 2 mL of 6 M hydrochloric acid (HCl). Then replace the tube in the water bath, note the time and immediately remove 1 drop of the hot mixture with a clean stirring rod and transfer to the first iodine sample in the spot plate. Clean the stirring rod, and repeat the test at 1-min intervals. Stop testing when the starch-iodine color is no longer produced or after 15 min, whichever is shorter. Continue heating the starch solution in the water bath for an additional 10 min. Transfer the solution to a small beaker and neutralize it by carefully adding solid sodium bicarbonate ($NaHCO_3$) with stirring until no more CO_2 bubbles are evolved or until blue litmus paper no longer indicates an acidic solution. Label the beaker "hydrolyzed starch," and save for later testing.

- Hydrolysis of Sucrose.** Mix 10 mL of a 5% solution of sucrose with 2 mL of 6 M HCl in a large test tube. Heat in a boiling water bath for 15 min. Neutralize as above with NaHCO_3 . Label as "hydrolyzed sucrose," and save for later testing.
- Reducing Ability.** Label eight clean 15-cm test tubes as follows: glucose, fructose, maltose, sucrose, starch, hydrolyzed starch, hydrolyzed sucrose, and blank. Place 2-3 mL of Tollen's reagent in each test tube then add 10 drops of the corresponding carbohydrate solutions from the solubility study in Part I.1 and the hydrolyzed solutions from parts II.1 and II.2. To the eighth tube, add distilled water. Heat the tubes in a boiling water bath for 10-15 min. Record your observations on the report sheet.

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PRE-LABORATORY QUESTIONS

Name _____ Section _____ Date _____

1. What is the ratio of C, H, and O in most carbohydrates?

2. List two types of tests that can be used for the identification of sugars. Briefly describe how each is carried out and what type of experimental observations are obtained and what these results indicate about the sugar tested.

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DATA AND OBSERVATIONS SHEET

Names _____ Section _____ Date _____

I. Physical Tests

1. Water Solubility

	Color	Physical Appearance	Solubility
Glucose			
Fructose			
Maltose			
Sucrose			
Starch*			

*Describe the results of the iodine tests on the filtered and unfiltered starch "solutions" below.

2. Observed Rotation

	Path Length (cm)	Trial 1	Trial 2	Trial 3	Average Rotation
Glucose					
Sucrose					

EXPERIMENT 9: DATA AND OBSERVATIONS (continued)

II. Chemical Tests

1. Hydrolysis of Starch. Using the scale below, indicate the intensity of the color by placing lines in the blocks. Thus a deep color might be indicated as , and a weak color as .

Min.	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
I ₂ Test Color																

3. Reducing Ability - Benedict's test observations

Glucose	
Fructose	
Maltose	
Sucrose	
Starch	
Hydrolyzed starch	
Hydrolyzed sucrose	
Blank	

REPORT – Experiment 9 - Properties of Carbohydrates: Solubility, Reactivity, and Specific Rotation

Names _____ Section _____ Date _____

Compound Name and Structure	Appearance	Results of Solubility/Reactivity Tests			exptl $[\alpha]_D$	lit $[\alpha]_D$	lit mp ($^{\circ}\text{C}$) ^a
		H ₂ O solubility	Iodine test	Benedict's test			
glucose			NA				
fructose			NA		NA	NA	NA
maltose			NA		NA	NA	NA
sucrose			NA		NA	NA	NA
starch					NA	NA	NA
hydrolyzed starch					NA	NA	NA
hydrolyzed sucrose			NA		NA	NA	NA

Chem 210, Winona State University, Fall 2022

*Reference used for literature values _____

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3. Using structural formulas, write a complete, balanced equation for the hydrolysis of sucrose. Explain why hydrolyzed sucrose gave the result in the Benedict's test that it did.
4. Write an equation for the reduction of Cu^{2+} by a sugar showing a positive Benedict's test.
5. Why are all sugars solids with high melting points? Explain in terms of intermolecular forces.
6. How precise was the specific rotation measurement? How accurate? Identify at least two sources of error in the measurement.