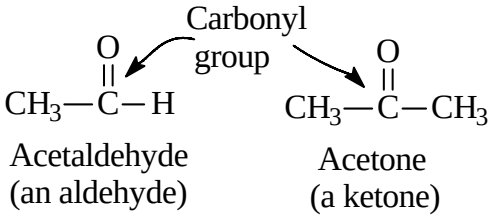
Aldehydes and Ketones **Experiment #4**

Objective: To study some chemical reactions that are used to distinguish aldehydes and ketones, especially oxidation of aldehydes.

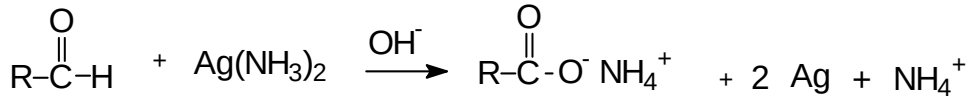
Introduction

Aldehydes and ketones both contain the carbonyl functional group, which imparts similar chemical reactivities to these two classes of compounds with some reagents. Aldehydes are much more susceptible to oxidation because a hydrogen atom is attached to the carbonyl, which is the basis for some of the chemical reactions that distinguish between these two classes of compounds.

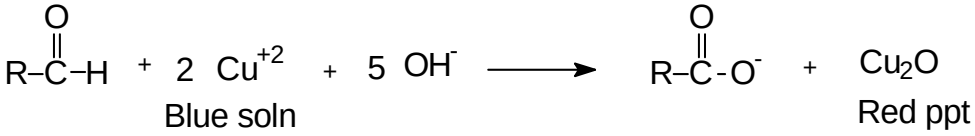


The oxidation of aldehydes can be performed with a mild oxidizing agent, such as Ag^+ in ammonia solution used in the Tollens test or Cu^{2+} in alkaline solution used in Fehling's test. If the Tollens test is performed in a scrupulously clean glass vessel, the silver metal is plated on the walls of the glass to form a silver mirror. Fehling's reagent is a deep blue Cu^{2+} solution that forms a brick-red precipitate of Cu_2O in the presence of aldehydes.

Tollens' Silver Mirror Test



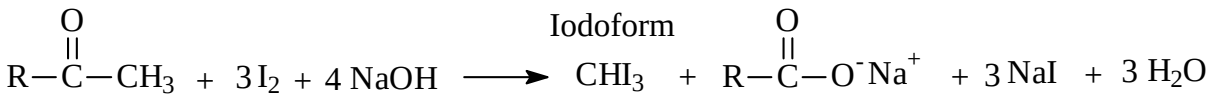
Fehling's Test



A stronger oxidant such as chromic acid, which was used to oxidize alcohols in the previous experiment, will also oxidize aldehydes readily, but generally does not oxidize ketones. The chromic acid in Bordwell-Wellman reagent is an orange-yellow solution that forms the green Cr^{3+} ion when it is reduced by alcohols or aldehydes. Ketones should give no reaction.

Methyl ketones react with iodine (I_2) in the presence of strong base to form iodoform, a

yellow compound insoluble in water. The reaction is summarized below. The resulting iodoform has an odor similar to chloroform and can be used as a topical disinfectant - usually in veterinary medicine.



Materials and Reagents

Test tubes, 6 M NaOH, 6 M NH_4OH, dilute HNO_3, Fehling's solutions A and B, Bordwell-Wellman reagent, iodine in potassium iodide solution, acetone, benzaldehyde, acetaldehyde, cyclohexanone, 2-pentanone, 3-pentanone, and unknown sample.

Procedure

Begin Here: Fill a 250 mL beaker about half full with tap water and heat it over a burner flame for Part B.

Part A. Tollens' Silver Mirror Test (Start this after setting up hot water bath)

Note: Test tubes must be scrupulously clean for the silver mirror test to work. Be sure to rinse tubes well with deionized water. The chloride ions in tap water interfere with mirror formation.

1. Wash 5 test tubes thoroughly with soap/detergent and water and rinse well. Give at least 2 final rinses with **deionized** water.

2. Add 10 drops of 6 M NaOH to each test tube and swirl the tube. Allow the NaOH solution to remain in the tube for about 1 min, then discard the NaOH solution in the sink. DO **NOT** RINSE THE TEST TUBES!!!

3. Add about 1 mL of 0.1 M silver nitrate solution to each test tube. A brown precipitate of AgOH may form.

4. Add 15-20 drops of 6 M ammonium hydroxide (NH_4OH) solution to get the brown precipitate to dissolve. Shake to completely dissolve the brown precipitate. Add a few more drops of NH_4OH solution, if needed, until the precipitate completely dissolves.

5. Add 3 drops of acetone to tube 1; 3 drops of benzaldehyde to tube 2; 3 drops acetaldehyde solution to tube 3; 3 drops cyclohexanone to tube 4 and 3 drops of unknown to tube 5.

6. Mix each well and allow to stand without further shaking for 5 to 10 min and look for the formation of mirrored glass on the walls of the test tube.

7. Record your observations on the Report Sheet, Table 1.

8. Dispose of these reagents in the sink and flush with plenty of water.

8. Add dilute nitric acid (HNO_3) to the mirrored glass tubes to remove the silver deposits. Dispose of the nitric acid in the sink and flush with plenty of water.

Part B. Fehling's Test

The water bath you set up earlier should be boiling.

1. Add 5 mL of Fehling's Solution A to a small beaker and mix 5 mL of Fehling's Solution B with it. Notice the change in color of the Cu^{2+} ion when these solutions are mixed.

2. Add about 2 mL of the mixture prepared in step 1 to each of 5 clean test tubes.

3. Add 5 drops of the test compounds (aldehydes/ketones) to the Fehling's reagent in each test tube. Acetone in tube 1; benzaldehyde in tube 2; acetaldehyde in tube 3; cyclohexanone in tube 4; and unknown in tube 5.

4. Make sure the tubes are properly labeled before placing them in the boiling water bath.

5. After heating the mixtures for 5 minutes, take note of any changes in color or formation of a precipitate.

6. Record your observations on the Report Sheet, Table 1.

7. Discard the solutions in the sink and flush with plenty of water. The water bath is no longer needed. Turn off the gas and discard the hot water on the sink after it cools.

Part C. Oxidation with Chromic Acid

CAUTION!! Concentrated sulfuric acid in the Bordwell-Wellman reagent is very corrosive. Handle it with care in the hood.

1. Add 5 drops of Bordwell-Wellman reagent solution to each of 5 clean test tubes. This contains potassium dichromate in sulfuric acid. It is also known as chromic acid solution.

2. Add 6 to 8 drops of each of the test compounds (aldehydes/ketones) to the tubes containing the chromic acid solution. Acetone in tube 1; benzaldehyde in tube 2; acetaldehyde in tube 3; cyclohexanone in tube 4; and unknown in tube 5.

3. Mix well and note any color changes after a few minutes. If there is a reaction, the color should change from yellow/orange to green or brown.

4. Record your observations on the Report Sheet, Table 1.

5. Fill a large beaker about half full with water and discard the test solutions in the beaker of water. Flush the water in the beaker down the sink with plenty of water.

Part D. The Iodoform Reaction

1. Add 1 mL of 6 M NaOH solution to each of 5 clean test tubes.

2. Add 5 drops of the following test compounds to these tubes, one at a time. Acetone in tube 1; 2-pentanone in tube 2; 3-pentanone in tube 3; cyclohexanone in tube 4; and unknown in tube 5.

3. Add about 3 drops of iodine (I_2/KI) solution to each test tube and observe whether there are yellow crystals/precipitate of iodoform in each tube.

4. Record your observations on the Report Sheet, Table 1.

Dispose of these reagents in the "Halogenated Organic Liquid Waste" container in the hood.

Name _____ Section _____

Aldehydes and Ketones

Experiment #4 Pre-Lab Exercise

1. Describe the difference between an aldehyde and a ketone, and indicate how each differs from an alcohol.

2. Name one aldehyde or ketone that is used widely by consumers. Be sure to give the correct name and indicate its use.

3. Describe what is meant by oxidation and reduction in relation to organic compounds, giving one example of oxidation of an organic compound and one example of reduction of an organic compound. The compound you use for the example may be the same or different for the oxidation and the reduction reactions. Be sure to indicate what oxidizing agent is used and what reducing agent is used for each example.

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Name _____ Section _____

Aldehydes and Ketones

Experiment #4 Data & Report Sheet

Table 1. Tests for Aldehydes and Ketones. [indicate whether you observed a positive reaction (+) or no reaction (-), in each test].

Unknown # _____

	Tollens Test	Fehling's Test	Chromic Acid Test	Iodoform Test
Acetone				
Benzaldehyde				_____
Acetaldehyde				_____
Cyclohexanone				
2-Pentanone	_____	_____	_____	
3-Pentanone	_____	_____	_____	
Unknown # _____				

Questions

1. After looking at the results in Table 1, to what class of compound would you conclude your unknown belongs? (Aldehyde, ketone, methyl ketone, aromatic aldehyde)

2. Which one of the tests performed in this experiment would be most useful in differentiating butanal and benzaldehyde. Explain your answer.

3. A screening test is common to test for phenylalanine or phenylpyruvic acid in the urine. Show the structures of phenylalanine and phenylpyruvic acid and indicate whether any of the tests you have performed in this experiment would give positive results with this urinary metabolite. See your text for discussion of this disease.

4. Give a brief description of some of the health effects that phenylketonuria (PKU) can have on new born children and what precautions must be taken to prevent these unwanted health effects.