تفريغ مختبر عضوية

اسم الموضوع: Exp 4: Aldehydes & Ketones

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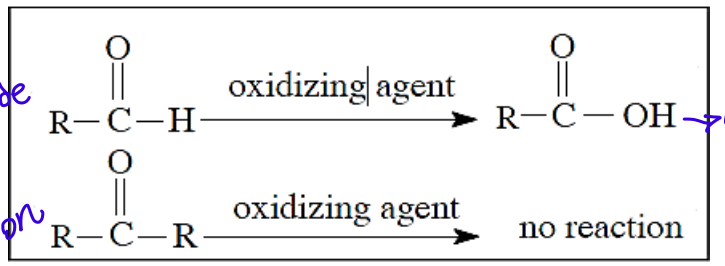
EXPERIMENT 4: ALDEHYDES AND KETONES

Classification, Tests and Derivatives

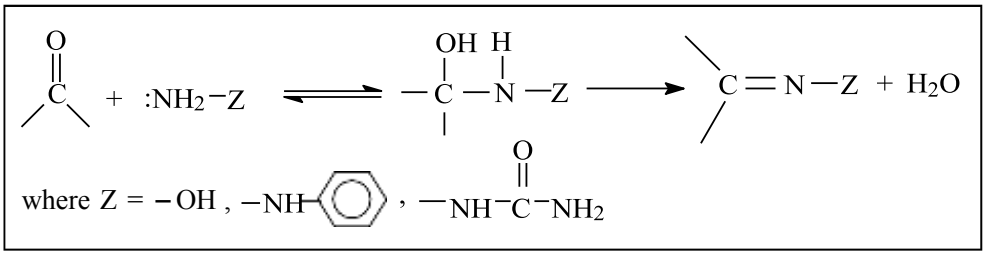
INTRODUCTION

The carbonyl group is common to both aldehydes and ketones, and as a result, both classes of compounds react similarly with many reagents. 2,4-Dinitrophenylhydrazine is commonly used to test for both types of compounds. However, a distinguishing behavior of aldehydes is their reaction with mild oxidizing agents which oxidize them to carboxylic acids while ketones, which are more difficult to oxidize, remains unchanged.



Several laboratory tests that distinguish between aldehydes and ketones, therefore, take advantage of this difference in behavior towards oxidants. One of these is *Tollens'* silver mirror test in which a silver ammonia complex ion is reduced, by aldehydes, to metallic silver. *Fehling's* and *Benedict's* solutions are also distinguishing reagents where the Cu(II) ion, complexed to tartarate or citrate respectively, is reduced to red cuprous oxide (Cu_2O) by aldehydes but not ketones.

Carbonyl compounds (aldehydes and ketones) are conveniently identified through a number of easily prepared derivatives. These include oximes, phenylhdrazones, 2,4-dinitrophenylhydrazones and semicarbazones. These derivatives are ideal because they are easily purified, crystalline solids with sharp melting points. The mechanism of formation of these closely related derivatives involves a typical nucleophilic addition at the carbonyl carbon followed by eliminationof a water molecule.



For further structural identification of methyl carbonyl compounds, the iodoform reaction, using iodine and aqueous sodium hydroxide is used. Compounds containing the CH3CO group give a bright yellow precipitate of CHI3 (*iodoform*).

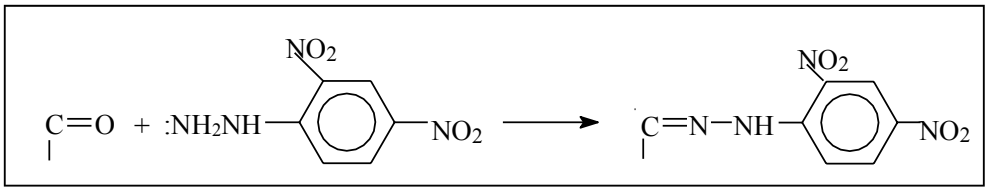
EXPERIMENTAL

MATERIALS NEEDED	Glassware: 4 test tubes, Erlenmeyer flask (50 mL), ice bath, graduated cylinder (10 mL), Buchner funnel, filter flask, melting point apparatus. Chemicals: 15 mL Tollens' reagent, 0.5 mL each of: formaldehyde, benzaldehyde, acetone, 2-propanol, 2-pentanone, 3-pentanone, 15 mL Fehling's or Bendict's solution, 12 mL sodium hydroxide(5%), 40 mL iodoform reagent, 1.0 g hydroxylamine hydrochloride, 3 g sodium acetate, 2.3 mL cyclohexanone, 30 mL petroleum ether, 5.0 mL phenylhydrazine reagent, 50 mL ethanol, 16 mL 2,4-dinitrophenylhydrazine reagent, 1.0 g semicarbazide hydrochloride and 1.0 g unknown.

I. TESTS AND DERIVATIVES

1. 2,4-Dinitrophenylhydrazine Test

يكشف عن الكربونيل General test



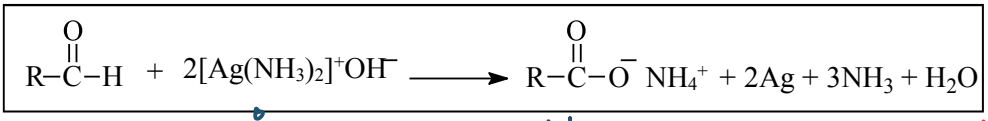
PROCEDURE

Glassware: 2 test tubes
The following carbonyl compounds to be tested: Acetone, or benzaldehyde.

1. In each test tube, add **2** mL of ethanol.
2. Add **5** drops of the carbonyl compounds to be tested and mix.
3. Add **2** mL of **2,4-dinitrophenylhydrazine** reagent and shake well.
4. Record your observations **and** result

Positive results → Bright orange to yellow precipitate ⇝ for Both Ketones & Aldehydes

2. Tollens' Silver Mirror Test



$R-\overset{O}{\overset{\|}{C}}-R' + 2[Ag(NH_3)_2]^+OH^- \rightarrow$ No Reaction

aldehydes ⇝ can oxidized to carboxylic acid By → Mild oxidizing agent (Cu, MnO4) ; Strong " " ($K_2Cr_2O_7$) & acidic Media & tollens Reagent

PROCEDURE

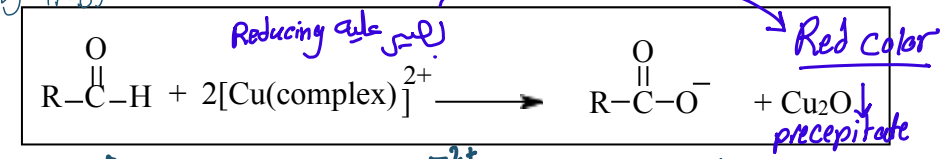
Glassware: 3 test tubes
The following carbonyl compounds to be tested: Acetone, formaldehyde, or benzaldehyde.

1. In each test tube, add 3 mL of **Tollens' reagent**.
2. Add **3-4** drops of the carbonyl compounds to be tested and mix.
3. Shake the tubes vigorously and allow to stand for **5** minutes.
4. Place the tube in a hot water bath (**50°C**) for **3-5** minutes.
5. Record your observations **and** result.

Positive results → Dark grey precipitate to silver mirror ⇝ (+ve) only with aldehydes

3. Fehling's and Benedict's Tests

(Fehling A+B)



$R-\overset{O}{\overset{\|}{C}}-R + 2[Cu(complex)]^{2+} \rightarrow$ No Reaction

PROCEDURE

Glassware: 2 test tubes
The following carbonyl compounds to be tested: Acetone, or benzaldehyde.

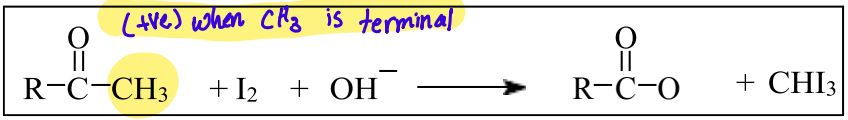
1. In each test tube, add both **2** mL of **Fehling's reagent A** and **2** mL of **Fehling's reagent B** and mix.
2. Add **3-5** drops of the carbonyl compounds to be tested and mix.
3. Place the test tube in a beaker of boiling water for **15-20** minutes.
4. Record your observations **and** result.

(+ve) with aldehydes
(+ve) formaldehyde
after heating

Positive results → Dark red to dark brown precipitate

4. The Iodoform Test

(+ve) when CH_3 is terminal



PROCEDURE

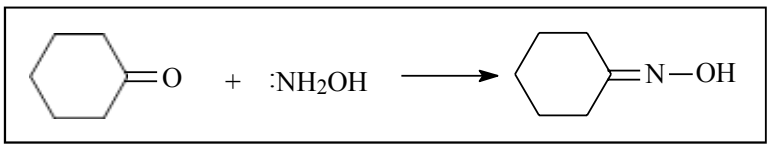
Glassware: 5 test tubes
The following alcohols to be tested: Acetone, formaldehyde, 2-propanol, 2-pentanone, and 3-pentanone.

1. In each test tube, add **3** mL of **5% sodium hydroxide**.
2. Add 10 drops of one of the alcohols to be tested.
3. Add **5-10** drops of **iodine solution** (or up to **0.5** mL) gradually.
4. Shake very well.
5. Allow to stand for **3-5** minutes.
6. Record your observations **and** result.

Positive results → Bright yellow precipitate

5. Cyclohexanone Oxime

Keton

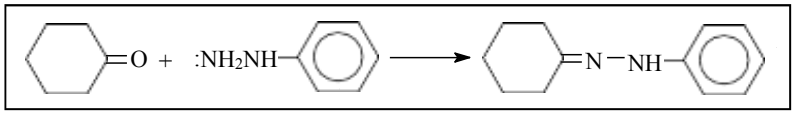


PROCEDURE.

Dissolve 1 g of hydroxylamine hydrochloride and 1.5 g of sodium acetate in 4 mL of water in a test tube. Warm the solution to about 40 °C and then add 1 mL of cyclohexanone. Stopper the flask and shake for 1-2 minutes. Cyclohexanone oxime begins to separate as fine colorless crystals. Cool the tube thoroughly in an ice bath to complete precipitation. Filter the crystals using a small Buchner funnel then wash with a little ice-cold water. Air-dry the crystals and determine their melting point.

Oximes of some carbonyl compounds will crystallize more slowly. They may require longer cooling and scratching the walls of the flask to induce crystallization. Oximes can be recrystallized nicely from petroleum ether (bp 40-50).

6. *Cyclohexanone Phenylhydrazone*

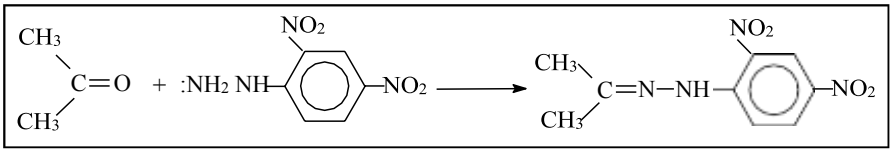


PROCEDURE.

Add 10 drops of cyclohexanone to 5 mL of ethanol and 5 mL of phenylhydrazine reagent Shake the mixture until a clear solution is obtained. Heat the mixture on a water bath for 10 minutes then cool in an ice bath and filter the crystals.

The crude phenylhydrazone may be recrystallized by dissolving it in hot ethanol and adding water to the hot solution until a faint turbidity persists. The solution is then cooled and the crystals are collected.

7. *Acetone 2,4Dinitrophenylhydrazone*

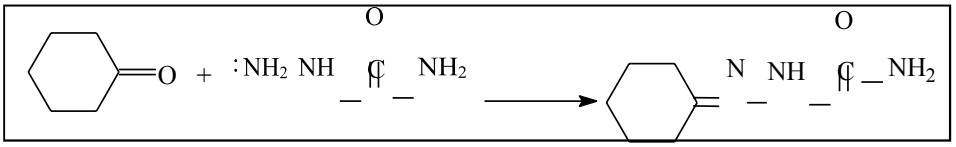


PROCEDURE.

Add 10 mL of 2, 4-dinitrophenylhydrazine reagent to 10 drops of acetone in 10 mL of ethanol and allow the mixture to standat room temperature for a few minutes. Precipitation of the acetone 2, 4-dinitrophenylhdrazone usually occurs immediately; other carbonyl compounds may require 5-6 minutes or even several hours. When crystallization is complete, cool the mixture in an ice bath, and collect the crystals. Recrystallize from ethanol, dry the resulting crystals and determine their melting point.

NOTE: Derivatives of phenylhydrazine are suspected carcinogens.
Handle with care and avoid skin contact.

8. Cyclohexanone Semicarbazide



PROCEDURE.

In a test tube dissolve 1g semicarbazide hydrochloride and 1.5 g sodium acetate in 10 mL of water, add 1 mL of cyclohexanone and shake vigorously. Place the test tube in a beakerof boiling water for 5 minutes then cool to room temperature. Place inan ice bath and scratch the sides of the tube with a glass rod until crystallization is complete. Filter the crystals, wash with a little water, and recrystallize from ethanol. Dry the crystals and determine their melting point.

II. IDENTIFICATION OF AN UNKNOWN CARBONYL COMPOUND

Procedure. While the various derivatives are drying, obtain

anunknown from your instructor and proceed to identify it as follows: Use *Tollen's* test to determine whether the compound is an aldehyde or a ketone. If the unknown is a ketone, perform the iodoform test to determine if it is a methyl ketone.

Finally prepare a crystalline derivative of the unknown to determine its identity. The unknown is selected from the aldehydes and ketones listed below:

Table 3. Derivatives of some aldehydes and ketones

Compound	Formula	Oxime	Phenyl-hydrzone	2,4-DNP	Semi-carbazone
Ethyl methyl ketone	$CH_3CH_2COCH_3$	oil	oil	116	136
Diethyl ketone	$CH_3CH_2COCH_2CH_3$	69	oil	156	139
Furfural	$C_4H_3O.CHO$	75	79	229;212	202
Crotonaldehyde	$CH_3CH = CHCHO$	119	56	190	199
Benzaldehyde	C_6H_5CHO	35	158	237	224
Cyclohexanone	$C_6H_{10}O$	91	81	162	167
2-Heptanone	$CH_3(CH_2)_4COCH_3$	oil	207	89	127
n-Heptanal	$CH_3(CH_2)_5CHO$	57	oil	108	109
Acetophenone	$C_6H_5COCH_3$	59	105	239	199
2-Octanone	$CH_3(CH_2)_5COCH_3$	oil	oil	58	123
Salicylaldehyde	$C_6H_4(OH)CHO$	63	143	252	231

Useful links

Aldehydes and Ketones (Tests)/Organic Chemistry Lab)
https://www.youtube.com/watch?v=1IaaMeGQwdg

EXPERIMENT 4

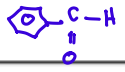
ALDEHYDES AND KETONES

Report Sheet

Name		Section no:	

- **OBJECTIVES:-** Identify the functional Group (aldehydes & ketones) & their Chemical properties in the Given organic Compounds.
  - Identify an unknown organic compund according to several test
- **IDINTIFICATION TESTS:**

I. 2,4-Dinitrophenylhydrazine Test

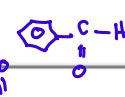
Compound	Function group	Result (+ or -)	Observations (color, ppt,..)
Acetone	$H_3C-\overset{O}{\overset{\|\|}{C}}-CH_3$	+	orange
Benzaldehyde		+	orange

II. Tollens' Test

Compound	Function group	Result (+ or -)	Observations (color, ppt,..)
Acetone	H_3CCCH_3 (O)	-	Black
Benzaldehyde		+	Silver mirror "Bright"

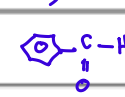
Formaldehydes $H-\overset{O}{\overset{\|\|}{C}}-H$ +

III. Fehling's or Benedict's Tests

Compound	Function group	Result (+ or -)	Observations (color, ppt,..)
Acetone	$H_3C-\overset{O}{\overset{\|\|}{C}}-CH_3$	-	No ppt / Blue color
Benzaldehyde		-	No ppt / Blue color

Formaldehydes $H-\overset{O}{\overset{\|\|}{C}}-H$ + Brown to Red / Red ppt

IV. Iodoform test

Compound	Function group	Result (+ or -)	Observations (color, ppt,..)
Acetone	$H_3C-\overset{O}{\overset{\|\|}{C}}-CH_3$	+	yellow ppt
Benzaldehyde		-	No ppt
2-pentanone	$CH_3\overset{O}{\overset{\|\|}{C}}CH_2CH_2CH_3$	+	yellow ppt
3-pentanone	$CH_3CH_2\overset{O}{\overset{\|\|}{C}}CH_2CH_3$	-	No ppt

- **Unknown Alcohol Determination:** according to the Given unkown

Unknown ID:		
Test used	**Observation**	**Result**

- Based on your results, what is your unknown?

- Draw the expected structure of your unknown showing the main function group: