

Aldehyde and Ketone Identification

Series of classification tests

Aldehydes and Ketones

2,4-dinitrophenylhydrazine (we did not do this)

Aldehyde

Chromic Acid
Tollens Reagent

Methyl Ketone

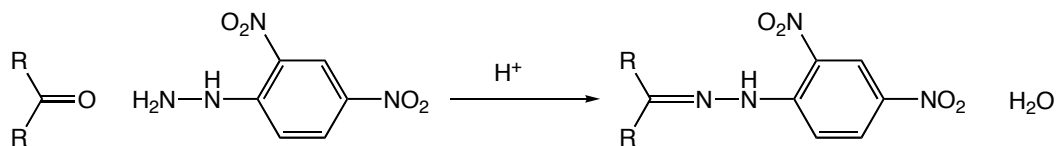
Iodoform Test

Compounds w/ enol content

Ferric Chloride Test
(we did not do this)

2,4-dinitrophenylhydrazine (Handle with care, dispose of in appropriate waste)

Reacts with ketones and aldehydes. (we did not do this test)



Form precipitate with aldehydes and ketones

Color of precipitate may provide info about compound:

- unconjugated = yellow
- conjugated = orange to red
- occasionally strongly acidic, strongly basic compounds, and some allylic alcohols.

Procedure

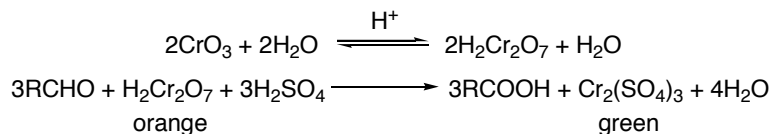
- Place 1 drop of unknown in a tube.
- Add 1ml of phenylhydrazine solution
- Shake vigorously

- If unknown is a solid, dissolve 10mg in 95% ethanol

- Most aldehydes/ketones give yellow to red precipitate.
It may take 15 minutes or gentle heating to achieve color change

Chromic Acid Test (Handle with care, dispose of in appropriate waste)

Reacts with aldehydes.



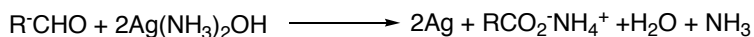
Works because of ready oxidation of aldehydes

Primary and secondary alcohols may also be oxidized as well giving false positive.

- Dissolve 1 drop or 10mg of unknown in 1ml reagent grade acetone
- Add several drops of chromic acid, w/ shaking
- Green precipitate and loss of orange color indicates presence of an aldehyde
- w/ aliphatic aldehydes – turns cloudy in 5 sec. and forms precipitate w/in 30 sec.
- If solution remains orange but precipitate forms = negative result.

Tollens Test (Handle with care, rinse glassware and acidify waste with 5% HCL, dispose of in appropriate waste)

Reacts with aldehydes.

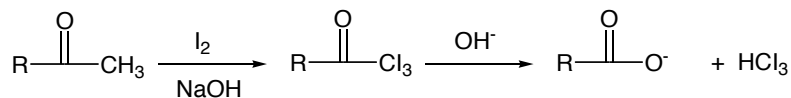


Should only be used if the compound has been determined to either be a ketone or an aldehyde.

- Combine 1ml Tollens A and 1ml Tollens B
- Add dilute ammonia dropwise to dissolve precipitate.
- Divide solution evenly between three test tubes.
- Add 1 drop of aldehyde or solid aldehyde in bis(2ethoxyethyl)ether to tollens reagent.
- Precipitation of silver (mirror formed) indicates presence of aldehyde (it may be necessary to warm the reaction)
- Reagents cannot be stored—dispose of them after they are used

Iodoform Test

Methyl ketone



Iodoform is a yellow precipitate

- Use a water bath between 60-70°C
- 6 drops of unknown or 0.06g in 2ml 1,2 dimethoxyethane
- Add 2ml of 10% NaOH
- Place in water bath
- Add 4ml of iodine-potassium iodide solution (in 1ml portions)
- Cork test tube and shake
- Heat in water bath ~5min.
- Dark color should be discharged
- If not, add more 10% NaOH

- Once color has disappeared, add water to within 2cm of lip
- cork and shake vigorously
- Let stand 15 minutes.

- Pale yellow precipitate (iodoform) indicates presence of methyl ketone
- Other ketones will discharge color, but will not form iodoform precipitate

Ferric Chloride Test

(we did not do this)

Phenols and compounds with high enolate content

Color is the result of enolate complexation with Fe(III) ion

- Add several drops of 2.5% aqueous soln of ferric chloride to 1ml of dilute ketone in aqueous solution.
- If solid not soluble in water, 20mg dissolved in CHCl_3 can be used.
- Add 1 drop of pyridine and 3-5 drops of 1% ferric chloride

- Formation of color (red, blue, purple, or green) indicates the presence of a compound with high enol content. Colors are short lived.