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Tollens Test: For Year 12 chemistry, the teachers wanted to demonstrate the silver mirror, also called Tollens test. The process goes like this:

1) 10 ml of a mixture of glucose with a bit of tartaric acid is poured into a flask.

2) 10 ml of ammonium nitrate and 10 ml silver nitrate are mixed together. This mix is then added to the flask.

3) 10 ml of strong sodium hydroxide is then added to the flask.

This whole mixture is swirled gently and a silver mirror from deposited silver forms on the glass. Usually.

In this case, this did not happen. I mixed the silver nitrate and ammonium nitrate a few days before, keeping the silver nitrate out of light to preserve it. The glass was clean. Despite all this, no silver mirror was formed. There was only a mild darkening of the glass.

Would anyone know what happened, and why?

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- Tollen's Test
- chemical waste disposal
- silver mirror

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Tollens Test

Submitted by Science ASSIST ... on Mon, 2016-09-05 16:14

Expert Answer

Answer reviewed 23 February 2023

The Tollens' test

Tollens' reagent is an alkaline solution containing a silver–ammonia complex used in organic chemistry to test for the presence of aldehydes. In this reaction, the aldehyde is oxidized to the corresponding carboxylic acid and the silver ion is reduced to metallic silver. This deposits as a thin film on the inner surface of the glass, which looks like a silver mirror. The general equation for the silver mirror test is:

$R–CHO + 2[Ag(NH_3)_2]^+ + 3OH^- \rightarrow R–COO^- + 2Ag + 2H_2O + 4NH_3$

No silver mirror formed

There are a number of possible causes for the Tollens' Test activity not working as expected.

- A scrupulously clean glass surface is necessary for a successful mirror effect.
- Impurities in the mixture can act as nuclei for very fine particles to precipitate, rather than form on the surface of the glass.
- Metallic silver deposits appear as dark grey to black if they are rapidly formed and are very fine grained. For example, silver on photographic film looks black rather than metallic shiny silver. The darkening could also be due to silver oxide (a stage of the process), which has not been chemically reduced to metallic silver.
- For best results, the Tollens' reagent should be freshly prepared.

Safety Notes

- Tollens' reagent should never be prepared in advance as ammoniacal silver solutions are hazardous and can explode on standing. Any unused solution should be deactivated as soon as possible and within two hours.
- Chemical hazards to consider in preparing and handling Tollens' reagent are that the concentrated solutions used are corrosive, may cause respiratory irritation and can cause severe skin burns.
- Silver ions are harmful for aquatic organisms and release to the environment should be avoided.

Science ASSIST recommends the following

- A site-specific risk assessment should be conducted.
- Prepare the diluted solutions in an operating fume cupboard.
- Conduct this activity in a well-ventilated room.
- Suitable PPE such as a laboratory coat, closed-in shoes, gloves and safety glasses should be worn at all times.
- Use scrupulously clean glassware.
- Immediately dispose of wastes generated as described below.

Additional Information

Cleaning glassware

1. Thoroughly scrub the glassware with soapy water and then rinse well with distilled water.
2. Wear appropriate PPE such as closed-in shoes, rubber or neoprene gloves, safety glasses and a laboratory coat.
3. Using a dropper, rinse the glassware with 2–3 mL of concentrated nitric acid in an operating fume cupboard.
4. Discard the nitric acid used in a beaker and rinse the glassware thoroughly with distilled water.
5. Dilute the concentrated nitric acid by carefully adding it to a large volume of water. Neutralize the solution with sodium bicarbonate to a pH of between 6–8 and flush down the sink.

Wastes disposal

Residual reaction mixture

Residues from the reaction should not be stored, as they can form explosive compounds on standing.

Small quantities: the residues should be diluted with a large volume of water and flushed down the sink.

Larger quantities: treat as for surplus Tollens' reagent.

Deactivating Tollens' reagent

Surplus Tollens' reagent should be deactivated within two hours of preparation as follows:

- Dilute any unused Tollens' reagent 20:1 with water.
- While stirring frequently, slowly add 1M hydrochloric acid solution until the pH reaches 2.
- The addition of hydrochloric acid is exothermic; cool in a water bath if necessary.
- Silver chloride will precipitate out at pH 2. Collect the silver chloride by filtration and allow to dry. Place in a suitable labelled container and store for collection by a licenced chemical waste disposal contractor.
- Neutralise the filtrate with sodium bicarbonate to a pH within 6–8 and flush down the sink.

Removal of silver deposit from glassware

The thin layer of silver formed can be removed from the silvered glassware using concentrated nitric acid in an operating fume cupboard and wearing suitable PPE.

- Nitrogen dioxide, a toxic gas, is evolved during the process.
- Add the concentrated nitric acid (2–3 mL for one test tube) drop wise to the glass vessel. A Pasteur pipette can be used to ensure that the acid covers the inside surface so that all of the precipitated silver is dissolved.
- To the resulting solution, slowly add saturated sodium chloride solution and white silver chloride will precipitate out. Continue to add the sodium chloride solution until there is no further precipitation of silver chloride.
- Collect the silver chloride precipitate by filtration and allow to dry. Place in a suitable labelled container and store for collection by a licenced chemical waste disposal contractor.
- Neutralise the filtrate with sodium bicarbonate to a pH of between 6–8 and flush down the sink.

Recipes for Tollens' Test

Tollens' Test using ammonium nitrate

The purchase of ammonium nitrate is regulated according to state and territory legislation. For ammonium nitrate regulations, please check with your state authority or jurisdiction. We have tested the following concentrations and procedure and can confirm that it works:

- The recommended concentration for the reagents are:
  - 0.5 M glucose solution
  - 0.5 M aqueous silver nitrate solution
  - 1.5 M aqueous ammonium nitrate solution
  - 2.5 M aqueous sodium hydroxide solution.
- Mix the aqueous silver nitrate and ammonium nitrate solutions just before conducting the experiment.
- Reduce the volume of each reagent used to 2 mL and use a test tube for the experiment.
- Carefully shake the test tube containing the glucose mixture to ensure maximum coverage.

Tollens' Test using ammonia

There are alternative methods, which do not involve the use of ammonium nitrate. We have tested the following procedure and can confirm that it works:

- To 50 mL of 0.1 M aqueous silver nitrate solution add concentrated ammonium hydroxide solution drop wise. A brown precipitate of silver (I) oxide is formed.
- Continue adding the ammonium hydroxide solution until a clear solution is formed.
- Add 25 mL of 0.8 M potassium hydroxide to the solution followed by the addition of ammonium hydroxide solution drop wise until the solution becomes clear.
- To a clean, dry test tube add 2.5 mL of 0.5 M glucose solution. Carefully shake the test tube to ensure that the entire surface is covered with the glucose solution.
- Add 12.5 mL of the freshly prepared Tollens' reagent to the glucose solution. The solution turns yellow, brown, then cloudy and dark and silver will begin to form on the inner surface of the test tube.

Links to other methods

Flinn Scientific. (2016) '*The Mirrored Flask'*, Retrieved from the Flinn Scientific website: https://www.flinnsci.com/the-mirrored-flask/dc10145/

Royal Society of Chemistry (2016*) 'A giant silver mirror experiment'*. Retrieved from the Royal Society of Chemistry website: https://edu.rsc.org/lcredir/learn-chemistry/resource/res00000822/a-giant...

Helpful Tips

- It may be helpful to place the glucose solution in the glassware first, stopper and shake it to ensure that the entire inside surface is covered. Then add the other reagents.
- Sometimes it is helpful to warm the mixture to get a silver mirror. This can be achieved by running the test tube/vessel under warm water from a tap, or using a warm water bath.

Background information

Monosaccharides are the simplest carbohydrates and are the building blocks from which all bigger carbohydrates are made. Glucose is a monosaccharide and a reducing sugar. The characteristic property of a reducing sugar is that it generates one or more compounds containing an aldehyde group in aqueous solution. Glucose reacts with water to give an open-chain form containing an aldehyde group.

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