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| Chemical Investigations |
| Analysing Anions |
| Teacher-technician Guide |



Introduction

Identifying substances is a very important aspect of chemistry.

There are not too many anions (positive ions) that are commonly encountered and here you will find out how to test for: chlorides, bromides, iodides, sulphates, sulphides, carbonates and nitrates.

Each group will need

|  |  |
| --- | --- |
| Your samples | Test tubes & rack |
| Hydrochloric acid (1 mol l-1) | Silver Nitrate (0.1 mol l-1 ) |
| Limewater | Pasteur pipette 1 cm3 and 3 cm3 |
| Barium chloride (0.1 mol l-1) | Ammonia (1.5 mol l-1) |
| Iron II sulphate solution  | Sulphuric acid (concentrated) |
| Ammonia solution (1.5 mol l-1 ) |  |

The concentrations are not critical here as the tests are qualitative.

Safety

1.5 mol l-1 ammonia and 0.1 mol l-1 silver nitrate are irritants. Wear eye protection.

Concentrated sulphuric acid is corrosive. Handle with care. Wear goggles and gloves.

To do

These tests **can** be carried out in any order but this one makes sense.

If you have solids, add a small amount of the solid to a test tube and add a couple of cm3 of distilled water. Then agitate to dissolve.

1. **Carbonates**
2. Add a few cm3 of your sample to a test tube.
3. Add a few cm3 of limewater into a second test tube
4. Add 2 cm3 of 1 Mol l-1 hydrochloric acid to your first test tube
5. If it is a carbonate (or hydrogencarbonate) you will see the formation of bubbles (of carbon dioxide).
6. Once any bubbling is complete, use a 3 cm3 plastic pipette to take a sample of the gas formed:

a. squeeze the pipette bulb and carefully lower the tip so it is just above the liquid surface

b. release the bulb to draw the gas into the pipette;

c. move the pipette of gas to the test tube with your limewater in and put the pipette tip just below the surface of the liquid

d. squeeze the bulb to bubble the gas into the liquid.

1. Look for the limewater turning cloudy – due to a white calcium carbonate precipitate).

This shows that the gas was carbon dioxide and hence that the solution was a carbonate or hydrogencarbonate.

1. **Sulphates**
2. Add a few cm3 of the solution(s) to a test tube.
3. Add a few drops of 0.1 Mol l-1 barium chloride solution and gently agitate the test tube

Presence of a sulphate results in the formation of a white precipitate (barium sulphate).

*(The reason for doing the carbonate one first is that carbonates also give a white precipitate with barium chloride)*

1. **Halides**
2. Add a few cm3 of the solution, to a test tube.
3. Add drops of 0.1 Mol l-1 silver nitrate solution, agitating between drops, until the mixture is just cloudy.

*The precipitate is a silver halide (either the chloride, bromide or iodide)*

1. Observe the colour of the precipitate. Silver chloride is white, the bromide is cream-coloured and the iodide a yellower cream.
2. Add 3 cm3 of 1.5 Mol l-1 ammonia solution to the tube with the precipitate.

If the precipitate dissolves – this shows it is the chloride.

1. If not, Place the test tube in a bath of boiling / a just-boiled water for a minute.

If the precipitate dissolves, either completely or partially, this indicates bromide

If there is no change, it is the iodide

1. **Nitrates**
2. Add a few cm3 of the solution, to a test tube.
3. Add a spatula-tip of iron II sulphate and agitate to dissolve (or add 0.5 – 1 cm3 of a saturated solution of the iron II sulphate)
4. Incline the tube and add slowly down the side about 1.0 cm3 of concentrated sulphuric acid

If it is a nitrate, you will see the formation of a brown ring where the two layers meet. (though it might be more of a brown layer rather than an actual ring)

1. Dispose of carefully by adding to a large volume of water.

Extensions/Additions

**Testing for ethanoate ions (and other carboxylate0**

1. Add a few cm3 of the solution to a test tube.
2. Add 3-4 drops of neutral iron(III chloride solution to the test tube.

If there is a carboxylate ion present, you will see the formation of a red solution of iron III ethanoate (or methanoate).

**Testing for sulphate IV (sulphite) ions**

Unless the quantities are **very** small, work in a fume cupboard because sulphur dioxide is produced.

1. Add a few cm3 of your sample to a test tube. (or a spatula tip of solid)
2. Add 3-4 drops of 1 Mol l-1 hydrochloric acid to the test tube.
3. Hold a piece of damp starch/iodate paper at the mouth of the test tube.

If the paper turns black/black, that indicates the presence of a sulphite. Sulphur dioxide reduces iodate V to iodine, forming the blue/black starch/iodine complex.

**Testing for phosphate ions - 1**

1. Prepare your test solution
* 50cm3 2.5MH2SO4
* 5cm3 potassium antimony tartrate solution – If you do not have this, you can leave it out but the solution will then need to be heated.
* 15cm3 ammonium molybdate solution
* 30cm3 ascorbic acid solution.

*Mix after addition of each reagent. The reagent is stable for about 4 hours.*

1. Add a few cm3 of your unknown solution to a test tube
2. Add 1 cm3 of your test reagent
3. Leave for a few minutes and observe. (If you are not using the antimonyl tartrate, place the tubes in a hot water bath rather than leaving at room temperature)

A blue colour shows that there were phosphate ions present

**Testing for phosphate ions - 2**

1. Add 0.5 g of solid or a few cm3 of the solution to a test tube.
2. Add 3 cm3 ammonium molybdate(VI) solution. Roughly 0.2 Mol l-1
3. Add an anti-bumping granule.
4. Gently heat the solution until the solution just starts to boil.
5. Slowly, add concentrated nitric acid dropwise to the hot solution – 10-15 drops are required.
6. If you see the formation of the yellow precipitate of ammonium phosphomolybdate, that is a positive test for phosphate.

**Test for nitrates IV (nitrites)**

This relies on the role of nitrate in the formation of azo dyes.

**To make the Diazo reagent solution**

Mix equal volumes of your test solution and the sulphanilic acid solution in ethanoic acid

**The test**

1. Put 1 cm3 of your chosen phenol/naphthol in a test tube
2. Add ¼ - ½ cm3 of your nitrite/sulphanilic acid Diazo reagent.
3. Add 3 drops of 1 mol l-1 NaOH

If there are nitrites present you will get an intense orange colour that darkens on addition of sodium hydroxide.

Technicians Guide

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**Preparation**

Most solutions are straightforward to prepare and need little guidance but

**molybdate test**

To prepare 100 cm3 of test solution

* 50cm3 2.5MH2SO4
* 5cm3 potassium antimony tartrate solution – If you do not have this, you can leave it out but the solution will then need to be heated.

*Dissolve 1.37g K(SbO)C4H4O6. ½H2O in 400cm3 distilled water in a 500cm3 volumetric flask and make up to the mark.*

* 15cm3 ammonium molybdate solution.

*Dissolve 20g (NH4)6Mo7O24. 4H2O in 500cm3 distilled water.*

* 30cm3 ascorbic acid solution.

*Dissolve 1.76g ascorbic acid in 100cm3 distilled water*

Mix after addition of each reagent. The reagent is stable for about 4 hours.

**Nitrite test**

**To make the Diazo reagent solution**

Prepare a sulphanilic acid solution in ethanoic acid (0.5% in 2 mol l-1 ethanoic acid)

Prepare a 0.02 mol l-1 (0.289%) solution of 2-naphthol in ethanol (IDA) (0.577 g / 100 mL)

*Alternatives are:*

*0.04 mol l-1 (0.37%) phenol in ethanol (IDA) (0.377 g / 100 mL) OR*

*0.03 mol l-1 (0.43%) 1-naphthol in ethanol (IDA) (0.433 g / 100 mL)*

You will also need 1 mol l-1 NaOH