Qualitative tests for the anions CO_3^{2-}, HCO_3^-, SO_4^{2-}, SO_3^{2-}, Cl^-, NO_3^- and PO_4^{3-} in aqueous solution

Student Material

Theory

Reactions of anions with certain reagents to produce characteristic coloured precipitates or other easily identifiable results are employed to identify or confirm the presence of these anions in aqueous solution and to distinguish the anions from one another.

(a) To test for the carbonate and hydrogencarbonate anions

Theory

Solutions containing hydrogencarbonate ions react with hydrochloric acid, producing carbon dioxide gas. For example,

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2\uparrow$$

Solutions containing carbonate ions also react with hydrochloric acid, producing carbon dioxide gas. For example,

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2\uparrow$$

It is possible to distinguish between solutions containing hydrogencarbonate ions and solutions containing carbonate ions using a further test. Carbonate ions in solution react with a solution containing magnesium ions, forming a precipitate of magnesium carbonate.
For example,

$$Na_2CO_3 + MgSO_4 \rightarrow Na_2SO_4 + MgCO_3\downarrow$$

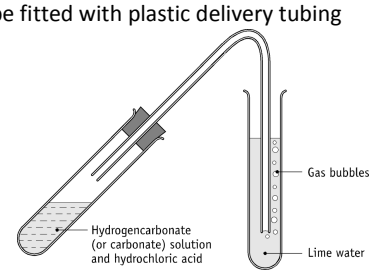
No precipitate is formed when solutions containing hydrogencarbonate ions are mixed with a solution of magnesium sulfate.

However, if a mixture of a solution containing hydrogencarbonate ions and a solution of magnesium sulfate is heated, a precipitate of magnesium carbonate is formed. This is because hydrogencarbonate changes to carbonate on heating.
For example,

$$2NaHCO_3 \rightarrow Na_2CO_3 + H_2O + CO_2$$
$$Na_2CO_3 + MgSO_4 \rightarrow Na_2SO_4 + MgCO_3\downarrow$$

Chemicals and Apparatus

- ✓ Solution of sodium carbonate
- ✓ Solution of sodium hydrogencarbonate
- ✓ Deionised water
- ✓ Dilute hydrochloric acid
- ✓ Limewater
- ✓ Solution of magnesium sulfate
- ✓ Test tubes
- ✓ Test tube rack
- ✓ Test tube holder
- ✓ Stoppers for test tube fitted with plastic delivery tubing
- ✓ Labels
- ✓ Bunsen burner
- ✓ Dropping pipettes
- ✓ Beakers
- ✓ Wash bottle



Procedure

NB: Wear your safety glasses.
Add 2 cm^3 of sodium carbonate solution to a clean test tube labelled A, and 2 cm^3 of sodium hydrogencarbonate solution to a clean test tube labelled B.

Using a dropping pipette add 2 cm^3 of dilute hydrochloric acid to each test tube. Record your observations in the table below.

Repeat steps 1 and 2 using the arrangement shown in the diagram. Record your observations in the table below. Write equations for the reactions occurring in the table below.

Add 2 cm^3 of sodium carbonate solution to a clean test tube labelled C, and 2 cm^3 of sodium hydrogencarbonate solution to a clean test tube labelled D. Using a dropping pipette add 2 cm^3 of magnesium sulfate solution to each. Record your observations in the table below. Write equations for any reactions occurring.

Carefully heat the contents of test tubes labelled C and D at the end of the step 4. Record your observations in the table below. Write equations for any reactions occurring.

	Carbonate	Hydrogencarbonate
Observations upon addition of HCl solution		
Observations and conclusion from limewater test		
Equation for reaction between the salt and HCl		
Observation upon addition of $MgSO_4$ solution		
Equation for reaction between the salt and $MgSO_4$		
Observation upon heating		
Equation for reaction upon heating		

(b) To test for the sulfate and sulfite anions

Theory

Solutions containing sulfite ions react with barium chloride solution, producing a white precipitate of barium sulfite. For example,

$$Na_2SO_3 + BaCl_2 \rightarrow 2NaCl + BaSO_3\downarrow$$

Solutions containing sulfate ions also react with barium chloride solution, producing a white precipitate of barium sulfate For example,

$$Na_2SO_4 + BaCl_2 \rightarrow 2NaCl + BaSO_4\downarrow$$

It is possible to distinguish between solutions containing sulfite ions and solutions containing sulfate ions using a further test. Hydrochloric acid is added to the white precipitates obtained using the barium chloride test.
Barium sulfite reacts and dissolves:

$$BaSO_3 + 2HCl \rightarrow BaCl_2 + H_2O + SO_2$$

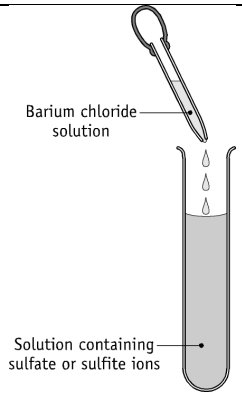
Barium sulfate does not dissolve.

Chemicals and Apparatus

- ✓ Solution of sodium sulfate
- ✓ Solution of sodium sulfite
- ✓ Deionised water
- ✓ Solution of barium chloride
- ✓ Dilute hydrochloric acid
- ✓ Test tubes
- ✓ Test tube rack
- ✓ Test tube holder
- ✓ Labels
- ✓ Dropping pipettes
- ✓ Beakers
- ✓ Wash bottle

Procedure

NB: Wear your safety glasses.



Add 2 cm^3 of sodium sulfate solution to a clean test tube labelled A and 2 cm^3 of sodium sulfite solution to a clean test tube labelled B. Using a dropping pipette add 1 cm^3 of barium chloride solution to each. Record your observations in the table below. Write equations for any reactions occurring.

Add 2 cm^3 of dilute hydrochloric acid to the contents of the test tubes labelled A and B at the end of Step 1. Record your observations in the table below. Write equations for any reactions occurring.

	Sulfate	Sulfite
Observation upon addition of $BaCl_2$ solution		
Equation for reaction between the salt and $BaCl_2$		
Observation upon addition of HCl solution		
Equation for reaction upon addition of HCl		

(c) To test for the chloride anion

Theory

Solutions containing chloride ions react with silver nitrate solution, producing a white precipitate of silver chloride. For example:

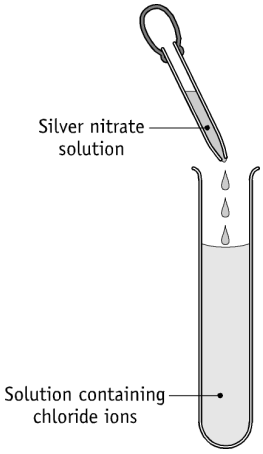
$$NaCl + AgNO_3 \rightarrow NaNO_3 + AgCl\downarrow$$

This precipitate dissolves when dilute ammonia solution is added.

Chemicals and Apparatus

Solution of sodium chloride
Deionised water
Solution of silver nitrate
Dilute ammonia solution

Test tubes
Test tube rack
Test tube holder
Labels
Dropping pipettes
Beakers
Wash bottle



Procedure

NB: Wear your safety glasses.

Add 2 cm^3 of sodium chloride solution to a clean test tube. Using a dropping pipette add a few drops of silver nitrate solution. Record your observations in the table below. Write equations for any reaction occurring.

Add 2 cm^3 of dilute ammonia solution to the contents of the test tubes at the end of the step 3. Record your observations in the table below.

	Chloride
Observations upon addition of $AgNO_3$ solution	
Equation for reaction between the salt and $AgNO_3$	
Observation upon addition of NH_3 solution	

(d) To test for the nitrate anion

Theory

Solutions containing nitrate ions react with a mixture of iron(II) sulfate solution and concentrated sulfuric acid. A brown ring develops slowly at the interface of the sulfuric acid layer and the layer containing a mixture of nitrate and iron(II) sulfate solutions.

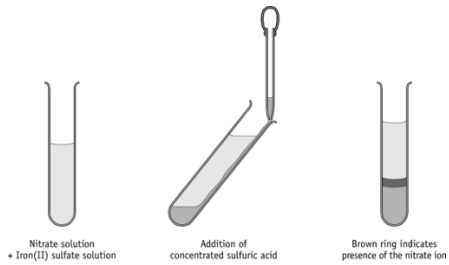
Chemicals and Apparatus

- ✓ Solution of potassium nitrate
- ✓ Deionised water
- ✓ Cold saturated solution of iron(II) sulfate
- ✓ Concentrated sulfuric acid 
- ✓ Test tubes
- ✓ Test tube rack
- ✓ Test tube holder
- ✓ Labels
- ✓ Dropping pipettes
- ✓ Beakers
- ✓ Wash bottle

Procedure

NB: Wear your safety glasses.

Add 2 cm^3 of potassium nitrate solution to a clean test tube. Using a dropping pipette, add 3 cm^3 of cold saturated iron(II) sulfate solution. Using a dropping pipette, carefully add 2 cm^3 of concentrated sulfuric acid slowly down the wall of the test tube. Do not mix the contents of the test tube.



Allow the mixture to stand undisturbed for a few minutes. Record your observations in the table below.

	Nitrate
Observations upon addition of $FeSO_4$ solution	
Observation upon addition of concentrated H_2SO_4	

(e) To test for the phosphate anion

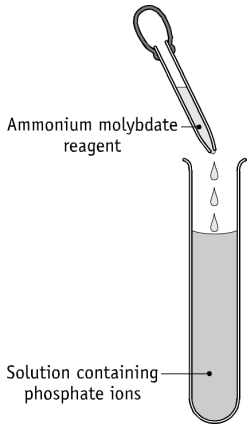
Theory

Solutions containing phosphate ions react on heating with an ammonium molybdate reagent, forming a yellow precipitate, which dissolves on addition of ammonia solution.

Chemicals and Apparatus

- ✓ Solution of disodium hydrogen phosphate
- ✓ Deionised water
- ✓ Ammonium molybdate reagent
- ✓ Dilute ammonia solution
- ✓ Warm water

- ✓ Test tubes
- ✓ Test tube rack
- ✓ Test tube holder
- ✓ Labels
- ✓ Thermometer
- ✓ Dropping pipettes
- ✓ Beakers
- ✓ Wash bottle



Procedure

NB: Wear your safety glasses.

Add 2 cm^3 of disodium hydrogenphosphate(V) solution to a clean test tube.

Using a dropping pipette add approximately 6 cm^3 of the clear ammonium molybdate reagent to the test tube.

Warm gently by placing in a beaker of water at a temperature not exceeding 40 °C. Record any observations in the table below.

Add an equal volume of ammonia solution to the contents of the test tube at the end of step 2. Record your observations in the table below.

	Phosphate
Observations upon addition of ammonium molybdate reagent and heating	
Observations upon addition of ammonia solution	

(f) To identify 'unknown' anions

Procedure

NB: Wear your safety glasses.

Add 2 cm^3 of each unknown solution to a clean test tube labelled with the same number as the unknown sample.

Using a dropping pipette add 2 cm^3 of dilute hydrochloric acid to the test tube. Record any observations in the table below.

If a gas is evolved establish with limewater whether it is CO_2 or not. If the gas is CO_2, proceed to test (a) and determine whether the unknown is a carbonate or a hydrogencarbonate.

If no gas is evolved, take further 2 cm^3 samples of the unknown in clean test tubes and test in turn with (i) barium chloride solution followed by dilute hydrochloric acid, (ii) silver nitrate solution, (iii) cold saturated iron(II) sulfate solution followed by concentrated sulfuric acid, and (iv) ammonium molybdate reagent, according to tests (b), (c), (d) and (e) respectively, until a positive result is obtained.

Complete a results table for each unknown.

	Unknown
Observations upon addition of HCl solution	
Conclusion from limewater test on gas produced	
Result from addition of $MgSO_4$ solution	
Result from addition of $BaCl_2$ solution followed by addition of HCl solution	
Observation upon addition of $AgNO_3$ solution	
Observation upon addition of $FeSO_4$ solution followed by concentrated H_2SO_4	
Observation upon addition of ammonium molybdate reagent	
CONCLUSION	

Specimen Results

	Carbonate	**Hydrogencarbonate**
Observations upon addition of HCl solution	Effervescence, colourless gas evolved	Effervescence, colourless gas evolved
Observations and conclusion from limewater test	Limewater becomes milky: CO_2 evolved.	Limewater becomes milky: CO_2 evolved.
Equation for reaction between the salt and HCl	$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2\uparrow$	$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2\uparrow$
Observation upon addition of $MgSO_4$ solution	White precipitate formed	No change observed
Equation for reaction between the salt and $MgSO_4$	$Na_2CO_3 + MgSO_4 \rightarrow Na_2SO_4 + MgCO_3\downarrow$	$2\ NaHCO_3 + MgSO_4 \rightarrow Na_2SO_4 + Mg(HCO_3)_2$
Observation upon heating	No change observed	White precipitate appears
Equation for reaction upon heating	None	$Mg(HCO_3)_2 \rightarrow MgCO_3\downarrow + H_2O + CO_2$

	Sulfate	**Sulfite**
Observation upon addition of $BaCl_2$ solution	White precipitate formed	White precipitate formed
Equation for reaction between the salt and $BaCl_2$	$Na_2SO_4 + BaCl_2 \rightarrow 2NaCl + BaSO_4\downarrow$	$Na_2SO_3 + BaCl_2 \rightarrow 2NaCl + BaSO_3\downarrow$
Observation upon addition of HCl solution	No change observed	White precipitate disappears
Equation for reaction upon addition of HCl	None	$BaSO_3 + 2HCl \rightarrow BaCl_2 + H_2O + SO_2$

	Chloride
Observations upon addition of $AgNO_3$ solution	White precipitate appears
Equation for reaction between the salt and $AgNO_3$	$NaCl + AgNO_3 \rightarrow NaNO_3 + AgCl\downarrow$
Observation upon addition of NH_3 solution	White precipitate dissolves

	Nitrate
Observations upon addition of $FeSO_4$ solution	Nothing observed
Observation upon addition of concentrated H_2SO_4	A brown ring appears at the boundary between the liquid layers

	Phosphate
Observations upon addition of ammonium molybdate reagent and heating	Yellow precipitate appears
Observations upon addition of ammonia solution	Precipitate dissolves

	Unknown (e.g. carbonate)
Observations upon addition of HCl solution	Gas evolved
Conclusion from limewater test on any gas evolved	CO_2
Result from addition of $MgSO_4$ solution	White precipitate formed
Result from addition of $BaCl_2$ solution followed by addition of HCl solution	nothing observed
Observation upon addition of $AgNO_3$ solution	nothing observed
Observation upon addition of $FeSO_4$ solution followed by concentrated H_2SO_4	nothing observed
Observation upon addition of ammonium molybdate reagent	nothing observed
CONCLUSION	Carbonate

student questions

Describe the appearance of pure samples of each of the following salts: sodium carbonate, sodium hydrogencarbonate, sodium sulfate, sodium sulfite, sodium chloride, sodium nitrate, disodium hydrogen phosphate.
They are all white crystalline solids. The samples used in the experiment are aqueous solutions of the salts.

Explain the steps you would take to identify which of these anions are present in a mixture of salts.
Take a 2 cm^3 sample of the mixture. Add dilute hydrochloric acid solution and collect any gas evolved and test with limewater. If carbon dioxide gas was evolved then either a carbonate or a hydrogencarbonate or both anions are present.
Take another 2 cm^3 sample of the mixture. Add $MgSO_4$ solution. If a precipitate forms, carbonate anion is present. Filter off the precipitate and heat. If further precipitate appears after heating, hydrogencarbonate ion is also present.

Take another 2 cm^3 sample of the mixture. Test with $AgNO_3$ solution. If chloride ion is present a white precipitate is formed. This precipitate dissolves in dilute ammonia solution.

Take another 2 cm^3 sample of the mixture. Test with $FeSO_4$ solution and concentrated sulfuric acid. A brown ring is observed if nitrate ion is present.

Take another 2 cm^3 sample of the mixture. Test with ammonium molybdate reagent. If phosphate ion is present a yellow precipitate is formed. This precipitate dissolves in dilute ammonia solution.

Take another 2 cm^3 sample of the mixture. If there is no phosphate ion present, add $BaCl_2$ solution and a precipitate will appear if either sulfite or sulfate is present. Add dilute hydrochloric acid dropwise. If all the precipitate dissolves only the sulfite anion was present. If there is a mixture of sulfite and sulfate anions some of the precipitate will remain but some will dissolve producing sulfur dioxide gas, which has a distinctive smell.

Which of these anions might be expected to be found in treated tap water? Give a possible source of each anion you mention.

Hydrogencarbonate anion from water hardness.
Sulfate ion from flocculating agents or water hardness.
Chloride ion from water hardness or sterilisation treatment.

How would you distinguish two unlabelled samples, one of which is a carbonate and the other which is a hydrogencarbonate, from each other?

Take a 2 cm^3 sample of each sample. Add $MgSO_4$ solution. If a precipitate forms, it confirms the carbonate anion is present. Heat the other test tube and a precipitate appears upon heating a sample with the hydrogencarbonate ion.

How would you distinguish two unlabelled samples, one of which is a sulfate and the other which is a sulfite, from each other?

Take a 2 cm^3 sample of each sample. Add $BaCl_2$ solution and a precipitate will appear in each test tube. Add dilute hydrochloric acid dropwise. If all the precipitate dissolves the sulfite anion was present. In the case of the sulfate anion, the precipitate is insoluble.

Industrial, Environmental & Social Links

Industry, medicine, and environmental science are more concerned with quantitative measurement of these anions than with just identifying their presence.

Simple home soil test kits are an application of these types of test to establish the presence of nitrogen, phosphorus and the cation potassium in soil extract and crudely estimate the quantity present.