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Technician’s Guide

Introduction

This booklet gives a series of short experiments that will enable the investigator to determine the concentration of the following substances in water:

Boron

Calcium

Carbonate

Iron

Magnesium

Nitrate

Nitrite

Phosphate

For the colorimetric analysis, many of the values obtained for water samples may be too low to determine by colorimeter. There are two options to address this:

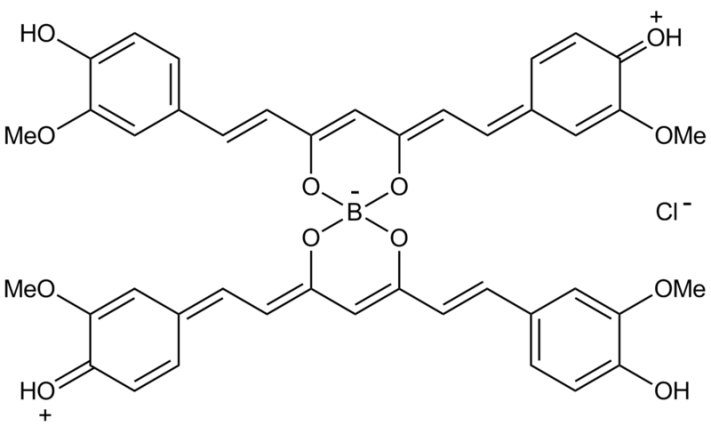
1. Use visual colorimetry to compare the colour with a range of standards. Looking vertically down the tube gives a longer path length than the 1cm standard in a colorimeter and so will allow easier discrimination.

2. Before analysis, evaporating the water sample to reduce its volume (to about 10%) will give a colour that is 10x as intense and thus easily readable. The only substance likely to be affected by boiling might be the calcium/magnesium or carbonate ions but they can be determined by titration using the undiluted sample.

**Boron**

**Introduction**

The test for boron involves borates (the form in which most boron is found in water) forming a coloured complex with cucurmin.



Cucurmin is the colouring agent in the spice turmeric. The yellow cucurmin complexes with the borate to form a red complex called rosocyanine.

**Equipment and Materials Required (per group)**

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| --- | --- |
| Solution of turmeric in water (0.5g of turmeric in 50cm3 of ethanol) | Test tubes (5 for the references plus however many samples you are doing) |
| Colorimeter & cuvettes | 20 ppm solution of boron. (sodium tetraborate) |

To make the 20 ppm solution of boron, you need to allow for the boron composition of the sodium tetraborate.

A 20 ppm solution of boron = a 179.5 ppm solution of sodium tetraborate. 0.179 g/l.

Make a more concentrated solution sn then dilute to ensure accuracy.

**Instructions**

1. Place 5 cm3 of your sample in a test tube.
2. Add 0.5 cm3 of turmeric solution
3. Transfer some of the coloured solution to a cuvette and read the absorbance at 490nm
4. Compare your results with a standard graph

**Results**

The values obtained from various mineral waters were low.

There was, however, a clear colour, so the concentration could be effectively determined by eye, looking down the length of the tube to increase the path length, comparing with various standard solutions.

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**SSERC Risk Assessment** (revised version November 2009)

(based on HSE ‘5 steps to risk assessment’)

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| --- | --- |
| Activity assessed | Testing water for boron |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

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| Step 1 | Step 2 | Step 3 | | Step 4 | | |
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| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| Sodium tetraborate is a reproductive toxin.  Sodium tetraborate solutions have no significant hazards. | Techician preparing solutions. | Wear gloves and eye protection, avoid raising dust. |  |  |  |  |
| Tumeric has no significant hazard.  Ethanol is flammable | Technician preparing solutions | Keep away from sources of ignition. Wear gloves and eye protection. |  |  |  |  |

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| **Description of activity:**  A solution of turmeric in ethanol is used as an indicator as it goes red in the presence of boron. Reference solutions of sodium tetraborate are made up and tested, the colours analysed by a colorimeter. Samples of water are tested in the same way and compared with the reference graph |

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| **Additional comments:** |

**Calcium and magnesium**

The ions involved in permanent water hardness, i.e. Ca 2+( aq) and Mg 2+ (aq), can be determined by titration with a chelating agent, ethylenediaminetetraacetic acid (EDTA), usually in the form of disodium salt. The titration reaction is:

**Ca+2+ EDTA-4 ------> CaEDTA-2**

**Equipment and Materials Required (per group)**

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| --- | --- |
| 0.001 M EDTA solution | 0.5% Eriochrome Black T solution |
| 1M NaOH | Murexide indicator powder |
| pH 10 ammonia buffer | Burette & associated titration equipment |
| Clamp and stand |  |

NaOH and EDTA are simply made up to the right concentration.

0.001M EDTA was correct for the mineral waters tested here at SSERC and for the Dunfermline tap water. If you are testing harder water, use 0.01M EDTA

**Ammonia Buffer**

1. Dissolve 17.5g of ammonium chloride (NH4Cl) in 142ml of concentrated ammonia (0.880).
2. Dilute to 250 cm3 with distilled water.

**Eriochrome Black T preparation**

Instructions

1. Put on gloves and protective eyewear and weigh out approximately 0.5 g of solid Eriochrome Black T, (EBT) on a balance and transfer it to a small beaker or flask. Add about 50 cm3 of 95 percent ethyl alcohol and swirl the mixture until the EBT has fully dissolved.
2. Weigh out 4.5 g of hydroxylamine hydrochloride on a balance and transfer it to the beaker or flask containing the EBT. Swirl until the hydroxylamine hydrochloride has fully dissolved.
3. Transfer the solution containing the EBT and hydroxylamine hydrochloride to a 100 cm3 graduated cylinder. Add enough 95 percent ethyl alcohol to bring the total volume to exactly 100 cm3.
4. Transfer the EBT solution from the 100-mL graduated cylinder to a dropper bottle and label the bottle "0.5% Eriochrome Black T in Ethanol."

**Murexide preparation**

Grind 1g of murexide with 100 g of sodium or potassium chloride.

**Tips & Warnings**

* EBT indicator solutions typically exhibit very short shelf lives. Always prepare a fresh EBT solution when performing complexometric titrations.
* Hydroxylamine hydrochloride is highly toxic and corrosive to skin and mucous membranes. Avoid direct skin contact. Wear rubber gloves and protective eyewear at all times when handling this compound.
* Ethyl alcohol is flammable. Avoid working near open flames or other possible sources of ignition.

**Instructions**

**Experiment 1 –** Total Hardness Determination

1. Fill a 50 cm3 burette with 0.001 M EDTA solution, making sure the tip is full and free of air bubbles.
2. Add 25.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 5 cm3 of Ammonia buffer to the beaker.
4. Add 0.5 cm3 of Eriochrome Black T indicator.
5. Titrate with the 0.001 M EDTA until the colour changes from wine red to pure blue. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

**Experiment 2 –** Calcium Determination

1. Fill a 50 cm3 burette with 0.001 M EDTA solution, making sure the tip is full and free of air bubbles.
2. Add 25.00 cm3 of an unknown hard water solution into a 100 cm3 beaker.
3. Add 2 cm3 of 1.0 M Sodium Hydroxide.
4. Add a spatula tip of Murexide indicator powder.
5. Titrate with the 0.001 M EDTA until the colour changes from salmon pink to orchid purple. Read burette to +/- 0.10 cm3.
6. Repeat the titration until the final volumes agree to +/- 0.20 cm3.

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| Activity assessed | Testing water for carbonates |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
| --- | --- | --- | --- | --- | --- | --- |
| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| EDTA is a skin, eye and respiratory irritant | Technician preparing solutions. | Wear gloves and eye protection. Avoid raising dust. |  |  |  |  |
| Sodium hydroxide is corrosive  1M sodium hydroxide solution is corrosive | Technician preparing solutions  Technician, teacher or pupils by splashes | Wear gloves and goggles (BS EN166 3).  Wear goggles (BS EN166 3). |  |  |  |  |
| Ammonia .880 is corrosive and the fumes are toxic (Cat 3)  The ammonia buffer is corrosive and gives off toxic fumes (Cat 3) | Technician preparing buffer solution.  Technician, teacher or pupils by splashes or inhaling fumes | Wear gloves and goggles (BS EN166 3). Handle in a fume cupboard  Wear goggles (BS EN166 3). Work in a well-ventilated ares and keep lid off bottle for as short a time as possible. |  |  |  |  |
| Murexide indicator (ammonium purpurate) has no significant hazard |  |  |  |  |  |  |
| Eriochrome black T is an eye irritant  Ethanol is flammable  Hydroxylamine hydrochloride is harmful by ingestions/skin contact, a skin/eye irritant, a skin sensitiser a category 2 carcinogen and can damage organs on repeated exposure.  Eriochrome Black T indicator solution is a skin sensitiser and a category 2 carcinogen. | Technician preparing solution.  Technician preparing solution.  Technician preparing solution.  Technician, teacher or pupils by splashes | Wear eye protection. Avoid raising dust.  Keep away from sources of ignition. Wear gloves and eye protection.  Wear gloves and goggles (BS EN166 3).  Wear gloves and goggles (BS EN166 3). |  |  |  |  |
| The reaction mixture is of no significant hazard. |  |  |  |  |  |  |

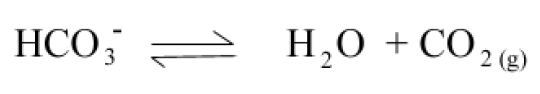
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| **Additional comments:** |

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| **Description of activity:**  Water samples are titrated against EDTA solution. Using murexide and eriochrome black T indicators. The solution is made alkaline by pH 10 ammonia buffer for the total hardness or sodium hydroxide for the magnesium. |

**Carbonate/hydrogen carbonate**

Temporary Hardness of water is due to the bicarbonate ion, HCO3, being present in the water. This type of hardness can be removed by boiling the water to expel the CO2, as indicated by the following equation:



In a neutral or slightly alkaline solution, hydrogen carbonates are in equilibrium with carbonates. Many bicarbonates are fairly soluble in water but the same cannot be said of carbonates.

The test for carbonate uses the great insolubility of lead carbonate.

A small amount of saturated lead nitrate solution is added to the samples and a precipitate of lead carbonate solution forms.

The turbidity of this solution can be measured with a colorimeter (at any wavelength) and compared to a graph of standard solutions.

**Equipment and Materials Required (per group)**

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| Saturated lead II nitrate solution | 500 ppm standard sodium hydrogen carbonate solution |
| pipettes | colorimeter |
| Test tubes | cuvettes |

**Tips & Warnings**

* 500 ppm = 500mg/l

= 0.5g per l

It is best to make a more concentrated solution eg 5 or 50 g/l and make accurate dilutions.

* At 20°C, the solubility of lead nitrate is about 52g/100cm3 of water
* Remember to shake up the sample before transferring it to the cuvettes to ensure the precipitate is evenly distributed.
* As you are just reading the turbidity of the sample, the wavelength is not important but make sure to keep it the same for each sample.
* Lead nitrate, as well as being a reproductive toxin and harmful to the environment (like most lead salts) can also cause eye damage so wear goggles.
* Although the amounts are small, lead compounds are very harmful to the aquatic environment. The samples containing the precipitates can be filtered and the solid lead carbonate kept for disposal by a licensed contractor.

**Instructions**

1. Take the 500 ppm sodium hydrogen carbonate solution and make a series of dilutions. (400, 300, 200, 100 and 50 ppm).
2. Place 5 cm3 of the first solution in a test tube and add 0.25 cm3 of lead nitrate solution. (about 10 drops)
3. Shake the solution, pour 3-4 cm3 into a cuvette and read in the colorimeter.
4. Repeat with the other reference solutions and plot the figures on a graph.
5. Carry out the same test on 5 cm3 samples of the test waters and read the absorptions off the graph to determine the concentration.

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| Activity assessed | Testing water for carbonate |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
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| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| Lead nitrate (solid and the saturated solution) is harmful by ingestion or skin contact. It is also a reproductive toxin and repeated exposure can cause organ damage. | Techician preparing solutions  Technician, teacher, pupil by splashes | Avoid raising dust. Wear gloves and goggles (BS EN166 3)  Wear gloves and goggles (BS EN166 3) |  |  |  |  |
| Sodium hydrogen carbonate is of no significant hazard. |  |  |  |  |  |  |
| Reaction mixture is a reproductive toxin | Technician, teacher, pupil by splashes | Wear gloves and goggles (BS EN166 3) |  |  |  |  |

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| **Description of activity:**  Lead nitrate is added to water samples causing a precipitate of lead carbonate. This causes turbidity in the water that can be measured with a colorimeter. |

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| **Additional comments:**  Lead compounds are hazardous to the environment.  Collect the reaction mixtures, add excess sodium carbonate or hydrogen carbonate solution to precipitate any remaining lead ions in solution, filter and keep the residue for disposal by a licensed contractor. |

**Iron**

This experiment allows for the analysis of iron content in water or food samples. The iron is present in a solution containing Fe3+ (ferric) ions. To make the presence of these ions in solution visible, thiocyanate ions (SCN−) are added. These react with the Fe3+ ions to form a blood-red coloured complex:

Fe3+ (aq) + SCN−(aq) → [FeSCN]2+(aq)

By comparing the intensity of the colour of this solution with the colours of a series of standard solutions, with known Fe3+ concentrations, the concentration of iron in the tablet or food sample may be determined. This technique is called colorimetry.

**Equipment and Materials Required (per group)**

|  |  |
| --- | --- |
| ferric ammonium sulphate FeNH4(SO4)2•12H2O standard solutions: | 2, 4, 6, 8 and 10 × 10−5 M (see below for preparation) |
| 1 M ammonium thiocyanate solution (see below for preparation) | 1 M & concentrated sulphuric acid |
| 1 M hydrochloric acid | 100 cm3 beaker |
| 200 and 500 cm3 volumetric flasks | 5 cm3 pipette |
| 100 cm3 conical flask | Test tubes (5 for the references plus however many samples you are doing) |
| Colorimeter & cuvettes | distilled water |

**Preparation of 1 M ammonium thiocyanate solution**

Weigh 38 g of solid ammonium thiocyanate into a 500 cm3 volumetric flask and make up to the mark with distilled water.

**Preparation of Fe3+ standard solutions**

NB: It may take several days to dissolve the Fe3+ salt used here, so carry out this preparation well in advance of the rest of the experiment.

Weigh out about 3.0 g of ferric ammonium sulphate (FeNH4(SO4)2•12H2O). Use a mortar and pestle to grind the salt to a fine powder. Accurately weigh 2.41 g of the powder into a 100 cm3 beaker and add 20 cm3 of concentrated sulphuric acid [corrosive]. Leave powder to soak in acid overnight.

The next day, carefully pour the acid/powder slurry into a 500 cm3 volumetric flask, rinsing the beaker into the flask a few times with water, then make up to the mark with distilled water. Let this solution stand for several days until the ferric ammonium sulphate powder has fully dissolved. If possible, insert a magnetic stirrer bar and stir the solution to speed up this dissolving process.

Use a pipette to transfer 20 cm3 of ferric ion solution to a 200 cm3 volumetric flask and make up to the mark with distilled water. This gives a solution with [Fe3+] = 0.001 M.

To prepare a 2 × 10−5 mol L−1 standard solution pipette 10 cm3 of the 0.001 mol L−1 solution into a 500 cm3 volumetric flask, add 10 cm3 of 1 M sulphuric acid, and then make up to the mark with distilled water.

Repeat this procedure in separate 500 cm3 volumetric flasks\*, pipetting in 20, 30, 40 and 50 cm3 of 0.001 M Fe3+ solution in turn, to obtain 4, 6, 8 and 10 × 10−5 M solutions respectively.

(\* if you do not have five 500 cm3 volumetric flasks you can use one flask to prepare each standard in turn. After preparing each standard, pour the solution into a labelled glass vessel which has a lid (eg: a glass bottle). Then rinse your 500 cm3 volumetric flask thoroughly with distilled water before using it to prepare your next standard solution.)

The concentrations in ppm Fe3+ of the standard solutions are:

|  |  |
| --- | --- |
| x10-5 mol/l | [Iron] ppm |
| 10 | 5.68 |
| 8 | 4.55 |
| 6 | 3.41 |
| 4 | 2.27 |
| 2 | 1.14 |

**Instructions**

**Iron in Water**

1. Accurately measure 2 cm3 of your sample solution into a clean, dry test tube\*.

2. Next, measure 2 cm3 of each Fe3+ standard solution into separate test tubes (one standard per tube) in order of increasing concentration, beginning with the 2 × 10−5 M standard. It is a good idea to first rinse your pipette or measuring cylinder with a few cm3 of the 2 × 10−5 M standard.

NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contains.

3. Add 2 cm3of 1M ammonium thiocyanate solution to each iron solution in sequence, with 2 minutes between each addition\*\*. These additions must be carefully timed so that all samples react for the same period of time.

4. Mix the solutions by swirling. A stable red colour will appear over the next few minutes.

5. As near as possible to 15 minutes after adding thiocyanate\*\*\*, pour your samples into a cuvette and measure the absorbance at a wavelength of 490 nm for each coloured solution using your colorimeter. These measurements will be made in sequence − one sample every two minutes − reflecting the timing of the thiocyanate additions above. The measured absorbance of light is a direct measure of the intensity of the solution’s colour.

*\* You can, if you wish, simply add the solutions and mix in a cuvette.*

*\*\* If you have a colorimeter to hand, you should be able to do it faster than this. 1 sample per minute is easily achievable and with practice, one every 30s is quite possible.*

*\*\*\* As long as you are reasonably close to the time, all should be well. When left for another 5 minutes, the darkest of the reference samples only dropped from a reading of 0.44 to 0.42. An extra 10 minutes caused a further drop to 0.40.*

**Method adapted from**

The College of Science, University of Canterbury, Christchurch, New Zealand

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| Activity assessed | Testing water for iron |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
| --- | --- | --- | --- | --- | --- | --- |
| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| Sulphuric acid is corrosive  1M sulphuric acid is irritant to skin and eyes | Technician preparing solutions, by splashing.  Technician, teacher, pupils preparing diluted reference solutions, by splashing. | Wear gloves and goggles (BS EN166 3)  Wear gloves and eye protection. |  |  |  |  |
| Iron III ammonium sulphate is a skin/eye irritant  The reference iron solutions are of no significant hazard | Technician preparing solutions, by splashing. | Wear gloves and eye protection. |  |  |  |  |
| Ammonium thiocyanate is harmful by ingestion, skin contact and inhalation.  1M Ammonium thiocyanate solution is of no significant hazard. | Technician preparing solutions | Avoid raising dust. Wear gloves and eye protection. |  |  |  |  |

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| **Description of activity:**  Solutions of known iron concentration as well as unknowns are reacted with ammonium thiocyanate. This results in a red compound. The reference samples are read in a colorimeter and a reference curve is constructed which is used to determine the iron concentration in the water samples. |

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| **Additional comments:** |

**Nitrites**

**Introduction**

To make the presence of the nitrite (NO2-) ions in solution visible, they are reacted with suphanilic acid and phenol which, in alkaline conditions, produces a bright yellow azo-dye.

By comparing the intensity of the colour of this solution with the colours of a series of standard solutions, with known NO2- concentrations, the concentration of nitrites in the water sample may be determined. This technique is called colorimetry.

**Equipment and Materials Required (per group)**

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| --- | --- |
| 0.5% sulphanilic acid in H2O | 0.5% phenol in water |
| 1M NaOH in water | 5 cm3 pipette |
| Test tubes (5 for the references plus however many samples you are doing) | Colorimeter & cuvettes |
| distilled water | Solution of sodium or potassium nitrite 50 ppm |

A 50 ppm solution = 50 mg/l = 0.05 g/l

Make up a more concentrated solution (5 g per litre perhaps) and then use serial dilutions.)

**Instructions**

1. Prepare a series of standard solutions to test and get your reference graph. (From 50 ppm down to about 5 ppm should be fine)
2. Place 3 cm3 of your sample in a test tube.
3. Add 1 cm3 of sulphanilic acid solution and mix
4. Add 0.5 cm3 of phenol solution and mix
5. Add 0.5 cm3 of 1M NaOH and mix
6. Transfer the sample to a cuvette and read in a colorimeter at around 470 nm
7. Repeat with the next sample

**Nitrates**

This experiment involves the reduction of nitrate to nitrite and the subsequent estimation of the nitrite by colorimetry of an azo-dye product.

The traditional method of reducing nitrate to nitrite has been the use of a cadmium column. Due to its high toxicity, however, it is desirable to avoid using cadmium wherever possible. This method uses zinc as the reducing material.

**Equipment and Materials Required (per group)**

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| Zinc / potassium chloride mixture (1 : 6 ratio) | 0.5% sulphanilic acid in H2O |
| 0.5% phenol in water | 1M NaOH in water |
| 5 cm3 pipette | Test tubes (5 for the references plus however many samples you are doing) |
| Colorimeter & cuvettes | distilled water |
| Solution of potassium nitrate (50 ppm) |  |

Grind the zinc powder and potassium chloride in a pestle and mortar to ensure thorough mixing. Then put in a jar.

A 50 ppm solution = 50 mg/l = 0.05 g/l

Make up a more concentrated solution (5 g per litre perhaps) and then use serial dilutions.)

**Instructions**

1. Prepare a series of standard solutions to test and get your reference graph. (From 50 ppm down to about 5 ppm should be fine)
2. Place 5 cm3 of your sample in a test tube
3. Add 0.5g of the Zn/KCl mixture.
4. Leave for 5 minutes, shaking from time to time
5. Transfer 3 cm3 of the now reduced sample to another test tube. *(Allow to settle before taking it to avoid getting zinc particles)*
6. Add 1 cm3 of sulphanilic acid solution and mix
7. Add 0.5 cm3 of phenol solution and mix
8. Add 0.5 cm3 of 1M NaOH and mix
9. Transfer the sample to a cuvette and read in a colorimeter at around 470 nm

The reduction process does not seem to reduce 100% of the nitrate to nitrite but it does produce proportional results. It is important, therefore, to produce the standard graph from solutions of nitrate that are reduced to nitrite by this process rather than just using nitrite solutions.

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| --- | --- |
| Activity assessed | Testing water for nitrites and nitrates |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
| --- | --- | --- | --- | --- | --- | --- |
| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| Sulphanilic acid is a skin/eye irritant and a skin sensitiser.  Sulphanilic acid solution 0.5% is of no significant hazard | Technician preparing solution. | Wear gloves and eye protection. |  |  |  |  |
| Phenol is toxic (Cat 3) by ingestion, inhalation or skin contact. It is corrosive, a mutagen (Cat 2) and repeated exposure can damage organs.  0.5% phenol is of no significant hazard | Technician preparing solution. | Use in fume cupboard or well-ventilated room. Wear gloves (not nitrile) and goggles (BS EN16 3)  *To minimize inhalation of vapour, lid should be replaced immediately after use and it should be used in a well-ventilated room.* |  |  |  |  |
| Sodium hydroxide is corrosive  1M sodium hydroxide solution is corrosive | Technician preparing solutions  Technician, teacher or pupils by splashes | Wear gloves and goggles (BS EN166 3).  Wear goggles (BS EN166 3). |  |  |  |  |
| Potassium nitrate III (nitrite) is toxic if ingested (Cat 3) and an eye irritant. It is also an oxidiser  Potassium nitrate V is a skin, eye and respiratory irritant. It is also an oxidiser  50 ppm solutions of nitrate III and V are of no significant hazard | Technician preparing solutions | Avoid raising dust. Wear gloves and eye protection.  Keep away from flammable materials. |  |  |  |  |
| Zinc powder is flammable  Potassium chloride has no significant hazard | Technician preparing reagent | Keep away from sources of ignition |  |  |  |  |

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| **Description of activity:**  Nitrates III (Nitrites) are measured by comparing the intensity of a yellow azo-dye compared with a graph from reference samples.  Nitrates V are reduced to nitrates III by zinc powder (mixed with potassium chloride) and then the azo dye is formed in the same way as above. |

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| **Additional comments:**  The reduction of nitrate by zinc is not a complete process but it does seem to be proportionate. Do not compare results to a nitrate III reference graph, rather make dilutions of nitrate V and reduce them with the zinc to create your reference curve.  Zinc is harmful to the environment. Collect the reaction mixtures and filter the zinc powder out. Keep it for disposal by a licensed contractor. |

**Phosphate**

**Introduction**

**Equipment and Materials Required (per group)**

|  |  |
| --- | --- |
| 2.5M sulphuric acid | 0.00844m potassium antimonyl tartrate |
| 0.0162M Ammonium molybdate solution | 0.1M Ascorbic acid |
| Cuvettes | Colorimeter |
| Phosphate solution (10 ppm) |  |

0.00844m potassium antimonyl tartrate (Dissolve 1.3715 g K(SbO)C4H4O6. ½H2O in 400 cm3 distilled water in a 500 cm3 volumetric flask and dilute to volume.)

0.0162M Ammonium molybdate solution*:* Dissolve 20 g (NH4)6Mo7O24. 4H2O in 500 cm3 distilled water.

0.1M Ascorbic acid:Dissolve 1.76 g ascorbic acid in 100 cm3 distilled water

10 ppm phosphate = 0.01 g/l. Make up a more concentrated solution and then serial dilutions to get this concentration.

**Making up the combined reagent**: Mix the above reagents in the following proportions for 100 cm3 of the combined reagent:

50 cm3 2.5MH2SO4,

5 mL potassium antimonyl tartrate solution,

15 cm3 ammonium Molybdate solution, and

30 cm3 ascorbic acid solution.

*Mix after addition of each reagent.*

Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 h.

# Instructions

# Make a series of dilutions of your phosphate solution

# Place 5 cm3 of your reference solutions and your sample in test tubes.

# Add 1 cm3 of the combined reagent and mix thoroughly.

# Leave the solution for between 10 and 30 minutes.

# Transfer some of the coloured solution to a cuvette and read the absorbance at 590nm

# Compare your results with a standard graph

# SSERC_jpg

**SSERC Risk Assessment** (revised version November 2009)

(based on HSE ‘5 steps to risk assessment’)

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| --- | --- |
| Activity assessed | Testing water for phosphates |
| *Date of assessment* | 26th July 2013 |
| *Date of review (****Step 5****)* |  |
| *School* |  |
| *Department* |  |

| Step 1 | Step 2 | Step 3 | | Step 4 | | |
| --- | --- | --- | --- | --- | --- | --- |
| *List Significant hazards here:* | *Who might be harmed and how?* | *What are you already doing?* | *What further action is needed?* | *Action by whom?* | *Action by when?* | *Done* |
| Potassium antimonyl tartrate is harmful by ingestion and skin contact.  0.00844M solution has no significant hazard. | Technician preparing solutions. | Wear gloves and eye protection |  |  |  |  |
| Sulphuric acid is corrosive  2,5M sulphuric acid is also corrosive | Technician preparing solutions, by splashing.  Technician, teacher, pupils preparing diluted reference solutions, by splashing. | Wear gloves and goggles (BS EN166 3)  Wear gloves and goggles (BS EN166 3) |  |  |  |  |
| Ammonium molybdate has no significant hazard |  |  |  |  |  |  |
| Sodium phosphate is corrosive.  Potassium phosphate is a skin/eye irritant  Phosphate solutions are of no significant hazard | Technician preparing solutions.  Technician preparing solutions. | Avoid raising dust. Wear gloves and goggles (BS EN166 3)  Avoid raising dust. Wear gloves and eye protection |  |  |  |  |
| The combined reagent is a skin/eye irritant | Technician, teacher, pupils by splashing. | Wear gloves and eye protection. |  |  |  |  |
| The reaction mixture is of no significant hazard |  |  |  |  |  |  |

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| **Description of activity:**  Samples of water and phosphates of known concentration are incubated with the reagent. The presence of phosphate produces a blue complex. This is determined by colorimeter and the results for water compared with the reference curve. |

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| **Additional comments:** |