RSC | Advancing the Chemical Sciences

WITH PLYMOUTH UNIVERSITY

Royal Society of Chemistry
Analytical Division

2017 Schools' Analyst Competition

25th January 2017

Centre for Chemical Sciences
University of Plymouth

SAFETY INFORMATION: Experiment 1:

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a standard procedure you also need to know the potential risks of the chemicals being used in this experiment.

Concentrated Nitric Acid

Principal hazards: Extremely corrosive, causes serve burns upon contact with eyes, respiratory systems, skin. Oxidises materials upon contact, liberating nitrogen dioxide (**Caution Very Toxic**). Chronic exposure may cause erosion of teeth, lung damage and burns to skin, eyes and respiratory system.

Safe handling: Always handle in fumehood. Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

0.2 mol dm^{-3} Sodium Thiosulfate Solution

Principal hazards: Irritating to eyes, respiratory system and skin. Chronic exposure may cause allergic reaction in sensitive individuals.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

0.2% Starch Solution

Principal hazards: No adverse affects. Chronic exposure may cause mild irritation.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

1 mol dm^{-3} Potassium Iodide Solution

Principal hazards: Harmful if swallowed. Irritating to eyes, respiratory system and skin. Chronic exposure may cause allergic reaction in sensitive individuals.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

1 mol dm^{-3} Sodium Carbonate Solution

Principal hazards: Irritating to eyes, respiratory system and skin. Chronic ingestion is toxic. Chronic skin exposure may cause allergic reaction and sensitisation in sensitive individuals.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

Dilute Ethanoic Acid

Principal hazards: Irritating to eyes, respiratory system and skin. Chronic skin exposure may cause allergic reaction and sensitisation in sensitive individuals.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for medical help.

Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin appears damaged, call for medical aid.

If swallowed: Call for immediate medical help.

Disposal: Weak solutions may be poured down the sink and washed away with plenty of water. Larger amounts should be neutralised before disposal.

Each group member should read these before beginning the practical. Each member of the team should sign to say they have read and understood this.

School Name:

Team Names	Signature

THIS SHEET TO BE HANDED IN BEFORE STARTING

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SAFETY INFORMATION Experiment 2:

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a requirement you also need to know the potential risks of the chemicals being used in this experiment.

Each group member should read these before beginning the practical. Each member of the team should sign to say they have read and understood this.

THIS SHEET TO BE HANDED IN BEFORE STARTING PRACTICAL WORK

Potassium Pyrosulfate: CAS No.: 7790-62-7
Risk of serious damage to eyes.
EU Occupational Exposure Limits: 3 mg/m^3, Inhalable dust

Ascorbic Acid: CAS No.: 50-81-7
EU Occupational Exposure Limits: 3 mg/m^3, Inhalable dust

Sodium Molybdate: CAS No.: 10102-40-6
TLV: 5 mg/m^3 (as Mo)
PEL: 5 mg/m^3 (as Mo)
EU Occupational Exposure Limits: 5 mg/m^3 as Mo

Eye contact:	Irrigate thoroughly with water for at least 10 minutes. OBTAIN MEDICAL ATTENTION.
Skin contact:	Wash off thoroughly with water. Remove contaminated clothing and wash before re-use. In severe cases, OBTAIN MEDICAL ATTENTION.
Inhalation:	Remove from exposure, rest and keep warm. In severe cases OBTAIN MEDICAL ATTENTION.
If swallowed:	Wash out mouth thoroughly with water and give plenty of water to drink. OBTAIN MEDICAL ATTENTION.
Disposal:	Weak solutions may be poured down the sink and washed away with plenty of water.

School Name:

Team Names	Signature

THIS SHEET TO BE HANDED IN BEFORE STARTING

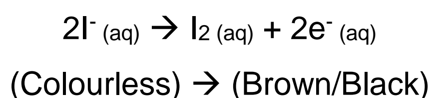
Experiment 1: Calculating the Percentage of Copper in a 1p Coin

Introduction

“Copper” coins were originally made out of “coinage bronze” an alloy consisting of mainly copper but also contains other metals. In 1992 the royal mint decided to stop producing coins out of “coinage bronze” as it was too expensive. Instead they adopted a steel insert that was then copper electroplated consequentially this makes them magnetic. In today’s experiment we are going to determine the percentage of copper present in a “coinage bronze” 1p coin using titrimetric analysis. Copper (II) ions are formed when “coinage bronze” is dissolved in concentrated nitric acid. Iodine is liberated by reacting the copper (II) ions with iodine ions. The amount of iodine formed can be found by titration with sodium thiosulfate. This will allow you to calculate the percentage of copper in a 1p coin.

Theory

A weighed 1p coin made before 1992 is dissolved in concentrated nitric acid. This forms a solution of copper (II) ions. This solution of copper (II) ions is treated with potassium iodide. Copper (I) iodide is precipitated out as a white solid and iodine is produced. The colourless iodine ions lose one electron and are oxidised to iodine which gives a brown coloured solution according to the half-equation below.



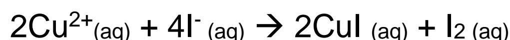
$$2I^-_{(aq)} \rightarrow I_{2\,(aq)} + 2e^-_{(aq)}$$

(Colourless) → (Brown/Black)

The copper (II) ions accept the electrons from the iodine ions. This reduces the copper (II) ions to the copper (I) ions according to the half-equation below.

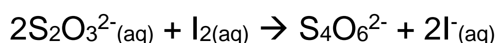
$$2Cu^{2+}_{(aq)} + 2e^- \rightarrow 2Cu^+_{(aq)}$$

The combined overall equation is shown below.



$$2Cu^{2+}_{(aq)} + 4I^-_{(aq)} \rightarrow 2CuI_{(aq)} + I_{2\,(aq)}$$

The amount of iodine liberated is found by titrating with sodium thiosulfate solution as shown below:



$$2S_2O_3^{2-}{}_{(aq)} + I_{2(aq)} \rightarrow S_4O_6^{2-} + 2I^-_{(aq)}$$

Knowing the amount of iodine formed, the concentration and amount of copper (II) ions can be calculated and the percentage of copper in a 1p coin calculated.

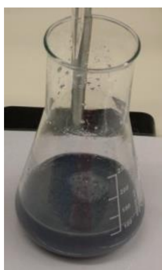
Experimental Method

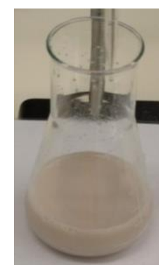
1. Weigh your 1p coin using a top pan balance and note its weight. Place the coin in a 250 cm^3 beaker and place this in the fume cupboard. **Do this in a fume cupboard** as toxic nitrogen dioxide is liberated (Brown gas). To the beaker add 20 cm^3 of concentrated nitric acid and **leave in the fume cupboard with the sash fully closed** until the coin has completely dissolved and there is no evolution of brown gas. Carefully, add 100 cm^3 of de-ionised water to the nitric acid solution. Then wash this solution into the 500 cm^3 volumetric flask and make up to the volume using de-ionised water (a pasteur pipette can be used for the last bit, to ensure you do not overshoot).

2. You can prepare you 50 cm^3 burette for the analysis by titration while the coin is dissolving in the nitric acid. Wash through with 0.2 mol dm^{-3} sodium thiosulfate then ensure that no air bubbles are trapped below the tap by fully opening the tap and running a little of your solution out again. Take an initial burette reading.

3. Pipette 25 cm^3 of the dissolved coin solution into a conical flask. Add small portions of sodium carbonate solution until a slight precipitate remains after swirling (this neutralises excess nitric acid). To this solution add a few drops of dilute ethanoic acid until the precipitate has dissolved. Measure accurately 10 cm^3 of the potassium iodide solution and add it to the conical flask (make a note of any changes you observe).

4. Titrate the iodine solution by adding small aliquot/portions of 0.2 mol dm^{-3} sodium thiosulfate solution to the flask and swirling it until a pale yellow colour is obtained. This may be difficult to observe as a creamy precipitate is formed so you need to ensure good swirling. Add 1 cm^3 of starch solution to the conical flask (the solution should turn a purple/black colour). Continue to titrate until all the purple/black colour has disappeared and take a new burette reading. This is the first rough titration to get an idea of the volume. Repeat the titration accurately until you achieve two concordant results, refilling the burette as necessary. Remember to go slowly near the end, adding no more than 1 drop at a





time. Calculate the average volume used from the two accurate titrations and then use your average volume to calculate the exact concentration of the Cu^{2+} liberated from dissolving a 1p coin in nitric acid (see results/calculation sheet).

Experiment 2: Determination of Orthophosphate in river water.

Background

Eutrophication is the term given to enrichment of a water body with nutrients, phosphorus and nitrogen, and can lead to algal blooms in estuaries and coastal waters which have become increasingly common in the UK and around the world. This type of increased biological activity is harmful because it causes the dissolved oxygen to be used up with the consequence that fish cannot survive and, in extreme cases, waters become anaerobic and 'stagnant'.

In fresh waters, phosphorus is a biolimiting element, i.e. its concentration limits biological growth, so an increase in the phosphate concentration increases the possibility of eutrophication and hence algal growth.

Theory

Orthophosphate (PO_4^{3-}) reacts with molybdate in an acid medium to form a phosphomolybdate complex. This complex is then reduced by ascorbic acid, resulting in an intense blue colour (known as molybdenum blue), the absorbance of which is measured spectrophotometrically.

Procedure

Standards

You are provided with a standard orthophosphate (PO_4^{3-}) solution that contains approximately 50 milligrams per cubic decimeter (50 mg dm^{-3}) as PO_4^{3-}. From this solution you will need to prepare, accurately, an approximately 5 mg dm^{-3} STOCK solution in the 100 cm^3 flask provided. Using this STOCK solution you must prepare standard solutions of approximately 0, 0.2, 0.4, 0.7 and 1 mg dm^{-3} in the 25 cm^3 volumetric flasks provided. Label these flasks A, B, C, D and E respectively.

1. Use the glass pipettes provided to transfer the appropriate volumes of the standard orthophosphate solution into the 25 cm^3 volumetric flasks. Dilute each flask to

volume with ultra-pure water (likely to be in a container labelled Milli-Q water) and thoroughly mix the contents by inversion.

2. Transfer, accurately, 10.0 cm^3 of each of the calibration standards into separate 30 cm^3 polypropylene screw-capped containers.
3. For each of these standards, the colour development procedure is as follows:
   - Add the contents of one PhosVer 3 phosphate reagent sachet. Screw the cap on to the container quite tightly and shake the contents for 15 seconds.
   - Allow the container to stand for 3 minutes. If phosphate is present, a blue colour should be visible.
   - Measure the absorbance of the colour-developed standard at 825 nm using the visible spectrophotometer instrument.

Advice on using the spectrophotometer

- Switch on the instrument at least 10 minutes before you first require to use it.
- Set the wavelength to 825nm.
- 'Zero' the instrument with your colour-developed 0 mg dm^{-3} standard in the cuvette.
- It is good practice to rinse out the cuvette with a small amount of the solution which you are going to measure prior to filling the cuvette and measuring the absorbance. A cuvette need be no more than about 2/3 full.
- Use a soft tissue to make sure that the exterior of the cuvette and, in particular, the optical (clear) faces are dry and clear of smears before placing it in the cuvette holder.
- Place the cuvette in the holder with the optical sides facing forwards and backwards in the path of the light beam.
- If your spectrophotometer has a multi-position cuvette holder, it is strongly recommended that you stick to just one cuvette position for all of your measurements.
- Periodically examine the cuvette for signs of scratches or etching on the optical faces. If there are some signs, discard the cuvette and select another for use, making sure that you subsequently re-zero the instrument with the new cuvette. (Re-reading the blank may help you decide whether the cuvette is still OK for use – a damaged cuvette will usually produce an artificially high absorbance reading).

Samples

You are provided with three samples of fresh water labelled F, G and H. Using what you have learned from the approach to obtaining calibration data, process the samples with a view to determining their orthophosphate concentrations.

Data treatment

Record your data using the results sheets provided.

Plot a graph of absorbance (y-axis) versus orthophosphate concentration (x-axis) using the graph paper provided. This is the calibration curve/line.

Using this graph and the sample absorbance data obtained, deduce the concentration of orthophosphate in each of the samples of fresh water.