Analysis of a dubious Health Potion

A group experiment in

Analytical Chemistry

Royal Society of Chemistry Analytical Division

North East Regional Heat of the

Schools' Analyst Competition 2009

Competition version

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Introduction

Analytical chemistry underpins all of modern chemistry, whether it is for quality control purposes, enironmental protection, forensic analysis, bulk chemical production or consumer protection. The experimental work you are going to do today is intended to give you experience of one such activity. We hope you find it interesting, and that it shows analytical chemistry to be as relevent today, possibly such that you might like to make a career from it.

You are going to be working as a team of three, so your first task is to read this booklet quickly then decide who is going to do what. Once the experimental work has been completed, you also need to interpret your data, and complete the questions. This competition is part of the RSC Schools Analyst Competition and is being carried out in several centres across the region. While we have to operate under the constraints of the competition and keep to time, we hope that you enjoy taking part.

Please read and understand the instructions before commencing and note that there is a strict time limit of three hours for the exercise.

The problem for today

Recently, there has been a rapid increase in the number of products on sale that claim to offer some health benefit. A new two part vitamin drink, called the "health potion" has been advertised in local shops. It consists of one sachet of powder and one tablet. The powder is said to contain vitamin C (each sachet should contain 1 g vitamin C) while the tablet contains iron (each tablet contains 200 mg iron as ferrous sulphate ($FeSO_4$)). The role of the vitamin C is to aid digestion of the iron. This formulation should work perfectly well, but there is some doubt about the quality of this preparation. Concerns have been raised that the vitamin C might have oxidised during storage, and that the actual amount of iron might be lower than stated; there is additional concern that large amounts of salt (sodium chloride) has been added to the powder to "bulk it out". There should be no sodium present in powder (i.e. 0 ppm).

Today, your team have the following three experiments to complete as well as answering some questions:

1. To assay the powder for sodium content.
2. To assay the powder for vitamin C content.
3. To assay the iron tablet for iron content.
4. Complete the questions on the back page.

Due to time constaints, we have already dissolved the powder and the tablet for you, making two ready to use solutions.

You have been supplied with one answer booklet for your team to complete. To give your team the best chance in the competition, accurate experimental work is required, along with careful calculations. You should also try to complete the booklet clearly and in a neat style.

Record your results in this booklet, but then make sure you transfer them to the answer booklet for your team.

Safe Working in the Laboratories

- Safety spectacles and fastened laboratory coats must be worn at all times. This is sufficient protective clothing for all experiments in this manual.

- SMOKING, EATING and DRINKING in the laboratories are prohibited.

- Coats and bags must be left in the designated area of the laboratory.

- There is a residue bottle in the fume cupboard for the disposal of organic chemicals.

- Accidents, however trivial, must be reported to a member of staff.

- We all have a legal responsibility to ensure the safety of ourselves and others in the Laboratory.

- Should protective gloves be required, the type will be specified in the manual.

- The standard precautions to be taken in handling chemicals are:

  (i) to avoid breathing the vapour;
  (ii) to avoid contact with skin and eyes;
  (iii) to avoid breathing the dust.

- You should wash your hands before leaving the laboratory.

Background

In this section, we have provided a brief description of each technique.

Units and ppm

In this booklet you will encounter two ways of describing concentration. The first is "molarity". This should be familiar to you and describes the number of moles per litre of each chemical. The second is "parts per million" or "ppm". This unit may not be familiar to you, but simply describes the number of mg of a particular analyte per litre (dm^3) of aqueous solution, for example 1 mg of copper in 1 litre of water would be 1 ppm. It is frequently encountered for elements rather than molecules, i.e. copper rather than copper sulphate.

Atomic emission spectrophotometric determination of sodium

When ions are heated in a flame, some of them become electronically excited. One way to return to the electronic ground state is to emit light at a characteristic wavelength. The amount of light is proportional to the temperature of the excitation source and the concentration of ions present. Thus if a flame in used to excite the ions, and the flame temperature is constant, then the amount of light emitted will be proportional to the concentration.

While all elements can be made to emit light, the temperatures required can be extreme. For some elements, e.g. lithium, sodium, and potassium, emission can be obtained at much lower temperatures. Flame photometry uses a cool flame to excite the ions, and thus only a few elements emit. This means that the emission spectrum is very simple, and unwanted emission wavelengths can be readily removed using a filter. Here, we use a flame photometer to measure the sodium emission from a suspect sample. Aqueous samples of sodium ions are aspirated into the methane/air flame and a percentage become electronically excited. As they return to the ground state, they emit light; this gives rise to the characteristic yellow colour in the flame. A filter is used to block out the other wavelengths so that only that due to the sodium emission reaches the detector. The amount of sodium in the sample can easily be determined from a calibration graph. This is produced by first analysing a set of calibration standards and plotting a graph of instrument reading vs. sodium concentration. The unknown concentrations can then be read off from the graph.

Titrimetric analysis of vitamin C

Vitamin C can be determined by a range of analytical techniques, but a straightforward method is based on titration. The vitamin C in solution is titrated with iodine volumetric reagent (iodine in potassium iodide). It reduces the brown iodine solution to a colourless solution of iodide (see reaction 1). When the end point is reached, excess iodine reagent can be seen without indicator, but to enhance the sensitivity of the method, starch is added. In the presence of starch indicator, the colour change at the end point is from colourless to persistent violet blue.

$$C_6H_8O_6 + I_2 \rightarrow C_6H_6O_6 + 2I^- + 2H^+ \qquad \text{(reaction 1)}$$

Spectrophotometric determination of iron

Many molecular species absorb radiation in the UV or visible regions of the electromagnetic spectrum. Solutions containing such molecules may be colourless or coloured, and the amount of light absorbed depends on the concentration of the molecules. It is slightly more complex than that, indeed the absorbance (A) of any solution is related to three parameters; the molar absorptivity (ε) of the molecules at the wavelength used, the concentration of the absorbing species (c) and the path length (l) through the solution. This is expressed as Beer's law (equation 1) and describes the relationship between the measured absorbance and the concentration of the absorbing species.

$$A = \varepsilon cl \qquad \text{(equation 1)}$$

In experiments like the one here, the pathlength is usually fixed at 1 cm. This is simply achieved by using a glass, plastic, or quartz cuvette that has holds a column of liquid exactly 1 cm in thickness. The molar absorptivity is usually different for each absorbing species present, and has units $mol^{-1}dm^3cm^{-1}$. The molar absorptivity can readily be determined by producing a calibration graph of absorbance versus concentration (measure the absorbance of a series of solutions of known concentration). Assuming a 1 cm cuvette was used, then the gradient of the line is ε. Once the calibration graph has been produced, it is relatively simple to determine the concentration of the analyte in a solution where the concentration of the analyte is not known. The absorbance of the solution can be read from the graph and by rearranging equation 1, the concentration can easily be calculated.

The problems with this method are that many species do not absorb light at a convenient wavelength and there are interferences. An alternative approach is to measure the analyte indirectly, by derivatising it to a species that absorbs at a suitable wavelength. Ideally, this procedure should eliminate any interference since the derivatising reagent should only react with the analyte of interest. A general method for the determination of iron has been developed which uses of 1,10-phenanthroline and a photometric detector. The method is capable of detecting iron content in the range 2-500 μg and is not susceptible to interference from ions and other metals. Iron(II) forms an orange-red complex with 1,10-phenanthroline in the pH range 2-9. The complex formed exhibits maximum absorption of light at a wavelength of 510 nm. Any iron present as iron(III) is reduced to iron(II) using ascorbic acid prior to forming the complex. In this method the iron(II) complex with 1,10-phenanthroline is formed at pH 4-6, which is achieved by using a sodium acetate-acetic acid buffer solution.

Experiment 1: Atomic emission spectrophotometric determination of sodium

Risk Assessment

Students are reminded that they have a legal responsibility to take all necessary precautions to ensure the safety of themselves and others in the Laboratory during the course of this experiment.

All the chemicals used in this experiment are of low hazard. Just remember to use good laboratory practice at all times: avoid getting the chemicals onto your skin, or in your eyes or mouth.

Apparatus

The solution prepared from the "health potion" powder
Sodium chloride (Analar, or equivalent)
1 x 1 L volumetric flasks
5 x 100 mL volumetric flasks
10 and 20 mL pipettes
Pipette filler
Pasteur pipette
Deionised water
Labels

Experimental procedure

1. You are provided with a solution prepared from the "Health Potion" powder. (The solution was prepared by dissolving one sachet in 250 mL of deionised water.)

2. Prepare a 100 ppm stock solution of sodium (from sodium chloride) by weighing 0.254 g of sodium chloride, and dissolving it in deionised water. Make up to 1 L.

3. By dilution of the 100 ppm stock solution, prepare five calibration solutions in the range 0 - 40 ppm sodium.

4. Consult a demonstrator about the operation of the flame photometer.

5. Check the filter on the flame photometer is set to sodium, and then aspirate your 40 ppm calibration solution into the flame photometer to check that the response is on the scale.

6. Once you are happy that the top calibration standard is on scale, aspirate each of your standards in increasing concentration order and then the solution obtained from the powder. Note down each reading in the table on the next page, it is best to take two readings for each solution.

7. If your sample gives a reading above your top calibration reading, you will need to dilute it.

Results for the sodium determination

Record the values in the table below and calculate the average reading for each solution.

Solution (ppm)	0	10	20	30	40	Sample
Reading 1						
Reading 2						
Average reading						

Calculation

1. Plot a graph of average reading vs. concentration for the sodium emission.

2. Use your graph to determine the concentration of sodium in the solution (ppm).

3. As ppm is the same as mg L^{-1} what is the mass of sodium in 250 mL (mg)?

4. If there is 1 g of powder in each sachet what is the concentration of sodium in the powder in ppm ?

Outcome of the analysis

This analysis will determine the amount of sodium in the powder.

My conclusion is that:

…….

Experiment 2: Titrimetric analysis of vitamin C

Risk Assessment

Students are reminded that they have a legal responsibility to take all necessary precautions to ensure the safety of themselves and others in the Laboratory during the course of this experiment.

Substances especially harmful to health and precautions to prevent exposure to them:

1. Iodine volumetric reagent – oxidising and harmful, avoid ingestion and contact with skin and eyes.

Apparatus

The solution prepared from the "Health Potion" powder
0.05 M iodine volumetric reagent (note down the exact concentration from the bottle)
Starch indicator
250 mL conical flask
25 mL pipette
50 mL burette and stand
Pipette filler
Pasteur pipette
White tile
Deionised water

Experimental procedure

1. You are provided with a solution prepared from the "Health Potion" powder. (The solution was prepared by dissolving one sachet in 250 mL of deionised water.)

2. Pipette 25 mL of this solution into a 250 mL conical flask.

3. Titrate the solution with the 0.05 M iodine solution placed in a 50 mL burette.
   Write down the exact molarity of the iodine solution for the calculation (this should be indicated on the container).

4. Towards the end point of the titration, add a few drops of starch indicator. REMEMBER vitamin C (ascorbic acid) reduces brown iodine volumetric reagent (iodine in potassium iodide) to a colourless solution. In the presence of starch indicator, the colour change at the end point is from colourless to persistent violet blue.

5. Record the results obtained in the table on the next page, you should continue until you get titre values within 0.1 mL of each other.

Results for the vitamin C titration

Record your titration results in the table below.

	Titre (mL)	Titre (mL)	Titre (mL)	Titre (mL)	Titre (mL)
Final volume on burette (mL)					
Initial volume on burette (mL)					
Volume required (mL)					

1. Calculate the number of moles of iodine that reacted with the vitamin C in the 25 mL aliquot of solution titrated. (This will tell you the number of moles of vitamin C in the aliquot.)

2. Next, determine the number of moles of vitamin C in **250 mL** of the solution. (This will tell you the number of moles of vitamin C that was in the sachet.)

3. Convert the number of moles of vitamin C in the 250 mL to a mass of vitamin C (g). (This will tell you the mass of vitamin C in the sachet.) Molecular mass of Vitamin C = 176.12 g mol^{-1}.

Outcome of the analysis

This analysis will determine the amount of vitamin C in the powder.

My conclusion is that:

……..

Experiment 3: Spectrophotometric determination of iron

Risk Assessment

Students are reminded that they have a legal responsibility to take all necessary precautions to ensure the safety of themselves and others in the laboratory during the course of this experiment.

Substances especially harmful to health and precautions to prevent exposure to them:-

1. Sodium acetate – acetic acid buffer. Corrosive- handle with care, avoid contact with eyes and skin.

2. 1,10-phenanthroline. Toxic if swallowed, toxic to the environment. **Do not dispose of down the sink**. **Waste must be disposed of in the waste containers provided.**

3. Ascorbic acid is not thought to pose any significant hazards. It may be a mild irritant.

4. Iron solution is not thought to pose any significant hazards, but treat with care avoiding contact with skin and eyes.

Apparatus

The solution prepared from the iron tablet
Standard iron solution (20 mg L^{-1} Fe(II))
1,10-phenanthroline solution
1.5% *L*-ascorbic acid solution
Sodium acetate – acetic acid buffer (pH 4.5)
6 x 100 mL volumetric flasks
1 x 2 mL pipette
1 x 5 mL pipette
2 x 10 mL pipettes
1 x 20 mL pipette
Pipette filler
Pasteur pipette
Deionised water
Labels

Experimental procedure

1. Pipette 5.0, 10.0, 15.0, and 20.0 mL aliquots of the standard iron solution (20 mg L^{-1}) provided into separate 100 mL volumetric flasks. Carefully label the flasks with the concentration which will be found when the solution is made up to 100mL.
   (Hint: When 5 mL of 20 mg L^{-1} is diluted to 100 mL, the concentration of iron in the new solution is 1 mg L^{-1}.)

2. To each flask, add 2 mL of ascorbic acid solution and 20 mL of sodium acetate-acetic acid buffer solution as provided.

3. Finally, to each flask add 10 mL of the 1,10-phenanthroline solution provided.

4. Mix thoroughly and dilute to the mark on the flask using distilled water. Mix thoroughly again after dilution.

5. A fifth flask, containing all the reagents as above but with no iron solution should be prepared, made up to the mark with distilled water and mixed thoroughly to be used as a blank.

6. A sixth flask, containing 10 mL of the solution prepared from the tablet, 2 mL of ascorbic acid solution, 20 mL of sodium acetate-acetic acid buffer solution and 10 mL of the 1,10-phenanthroline solution should be prepared and made up to the mark with distilled water.

   (The tablet was crushed and then dissolved in water and then diluted to 1 L. After filtration, 100 mL of this solution was taken and further diluted to 1 L.)

7. After 30 min measure the absorbance of the solutions in the spectrophotometer at 510 nm using the 1 cm cuvette provided (ask a demonstrator for help). Use the blank solution to autozero the instrument, then measure each solution in turn and finally the sample.

Results for the iron determination.

Record all data in the table below.

Volume of iron solution added (mL)	Iron content in new solution (mg L^{-1})	Absorbance

1. Plot a graph of average absorbance vs. concentration of iron.

2. Using the calibration graph, determine the iron content in the solution analysed (mg L^{-1})

3. Next, remembering that you made 100mL of the solution analysed from 10mL of the solution prepared from the tablet, determine the concentration of iron in the solution prepared from the tablet (mg L^{-1}).

4. What fraction of a tablet is in one litre of this solution?

5. Finally, calculate the mass of iron present in the one tablet (mg).

Outcome of the analysis

This analysis will determine the amount of iron in the tablet.

My conclusion is that:

……

Tie-Break Questions

1. What is the concentration (in ppm) of 10 mg of iron dissolved in 100 mL of dilute acid?

2. What is the molarity of a solution of sodium chloride prepared by dissolving 0.190 g of sodium chloride in 1 L of deionised water?
   (Atomic masses Na = 22.99 g mol^{-1} , Cl = 35.45 g mol^{-1})

3. Why is it important to have an appropriate intake of iron in your diet?

4. Why is it important to limit your dietary sodium intake?

5. Name one other analytical technique that could be used to determine the iron content of the tablet.

6. What happens to vitamin C when you cook food?

********** **We hope that you have all enjoyed the competition. This year we give thanks to Dr Tom McCreedy and technical staff at the University of Hull for the development of the competition*********