Schools' Analyst Competition 2014, East Anglia Region University of Hertfordshire Heat
Thursday 24th April 2014

Welcome to this year's Schools' Analyst Competition.

You are required to complete three experiments today.

Throughout the day assessors will be observing you at work and giving marks for such things as attitude to safety, housekeeping and experimental technique. The assessors are also available to help you so don't be afraid to ask if you are unsure of something; you won't automatically lose marks for asking in fact you may gain some.

Today is a competition so there can only be one winner but the organisers hope you will all enjoy your day. Good luck!

We gratefully acknowledge sponsorship from the following organisations

In today's experiments you will determine the weight of vitamin C, also known as ascorbic acid or more specifically L-ascorbic acid, in multi-vitamin tablets using two different analytical techniques (UV-visible spectroscopy and a titration) and you will determine the weight of zinc in the same tablets by inductively coupled plasma emission spectroscopy (ICP).

Vitamins and minerals and trace elements are essential nutrients your body needs in small amounts to work properly. Most people should get all the nutrients they need by eating a balanced diet however some people may need to take vitamin and mineral supplements. There are two types of vitamins fat soluble and water soluble. Water-soluble vitamins are not stored in the body, so they need to be imbibed more frequently. If a person has more than they need their body gets rid of the extra vitamins via the urine.

Vitamin C is a water soluble vitamin which protects cells and aids wound healing it is found in green leafy vegetables and fruit. Adults need 40 mg of vitamin C a day; there have been suggestions that mega doses of vitamin C, 1000 mg, have health benefits while doses up to 1000 mg per day will not cause harm doses in excess of this amount can lead to stomach pains and diarrhoea. Vitamin C, a natural antihistamine, is also important in a number of enzymatic reactions including some associated with the immune system and collagen synthesis.

Zinc is one of the most important trace elements to maintain good health as, like vitamin C it is important for the correct functioning of the immune system, in addition it is important in the maintenance of fertility and it also has a number of other functions. Good sources of zinc are meat, poultry, fish and seafood, grains, nuts, eggs, seeds and brewer's yeast.

Select fifteen multi-vitamin tablets obtain the total weight accurately and calculate an average tablet weight. Grind all the tablets together, the resulting powder will be used in the experiments below.

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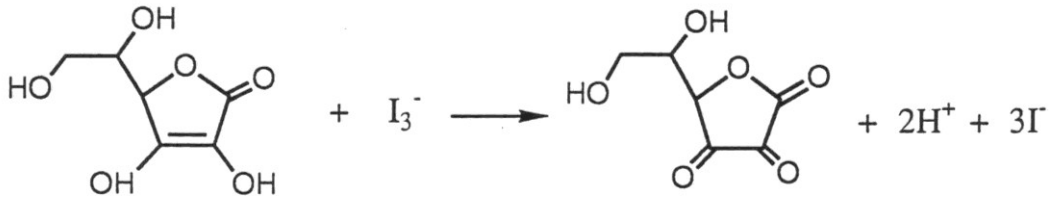
1.0 Titrimetric determination of vitamin C in vitamin tablets

1.1 Introduction

Ascorbic acid is oxidised to dehydroascorbic acid (dHAA) by iodine in acid solution according to the following equation:



An excess of iodine is generated in situ from a standard solution of potassium iodate, after the addition of an excess of potassium iodide. After reaction the excess iodine is determined by titration with a standard solution of sodium thiosulfate, using Thyodene as the indicator.

1.2 Standard solution of potassium iodate

Weigh accurately about 1 g of potassium iodate, transfer it quantitatively to a 500cm^3 volumetric flask, dissolve in water and dilute to volume.
You will be provided with a solution of sodium thiosulfate approximately 0.07M

1.3 Standardisation of the Sodium thiosulfate solution

Pipette 50.0 cm^3 of the standard iodate solution into an 'iodine flask' and add about 2 g of potassium iodide and 10 cm^3 of 0.5M sulfuric acid. Titrate the liberated iodine against the thiosulfate soluion, adding Thyodene solution towards the end of the reaction (i.e. when the solution is a pale straw colour). Carry the procedure out in duplicate, or until concordant results are obtained.

Calculate the exact molarity of the thiosulfate. The stock solution of sodium thiosulfate is now standardised.

1.4 Assay of ascorbic acid

Accurately weigh approximately 0.2g of tablet powder into an iodine flask, ultrasonicate with approximately 60 cm^3 of 0.5 M sulfuric acid, until the tablet has dissolved; you may need to use a glass rod to disperse the tablets (ignore any small insoluble residue). Then add 50.0 cm^3 of iodate solution together with about 2 g of potassium iodide. Stopper the flask and shake it thoroughly. Titrate the excess iodine against the standardised thiosulfate solution. Carry out the procedure in duplicate or until concordant results are obtained.

Calculate the weight of the ascorbic acid per tablet.

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2.0 UV spectroscopic determination of vitamin C

2.1 Introduction

The theory of quantitative spectroscopy is based upon BEER'S LAW which relates to the amount of radiation which is removed from a beam of radiation when it passes through a solution containing an absorbing species. The amount which is removed is termed the amount ABSORBED and is measured in terms of ABSORBANCE. The process may be illustrated as:

Incident beam of radiation → [cell] → beam of transmitted radiation

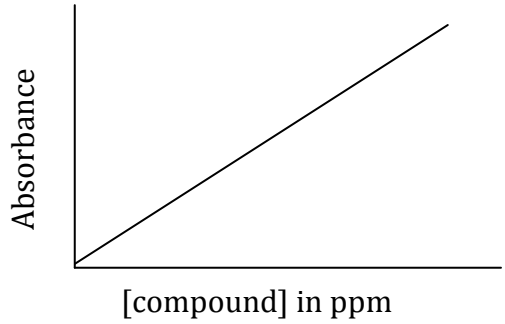
$$\text{Absorbance} = \log \frac{\text{intensity of incident radiation}}{\text{intensity of transmitted radiation}} = abc$$

Where a is a constant related to the absorbing species
b is the path length of the cell containing the species
c is the concentration of the absorbing species

Given that a and b remain constant throughout the analysis:

ABSORBANCE is proportional to CONCENTRATION

Therefore a calibration graph may be produced as illustrated in this experiment



2.2 Preparation of Standards

A solution of sodium oxalate (0.0056mol dm^{-3} in pH 5.4 buffer) is provided.
Weigh by difference approximately 125 mg of ascorbic acid, record the weight to 4 decimal places. Dissolve in the buffered sodium oxalate solution and make up with the same solution to 250 cm^3 in a volumetric flask. Pipette 5 cm^3 of this solution into a 50cm^3 volumetric flask and make up to volume with sodium oxalate solution.

Prepare a set of standards containing 1.0, 2.0, 3.0 and 4.0 cm^3 of the prepared ascorbic acid solution in 50 cm^3 volumetric flasks. Make up to volume with sodium oxalate solution (0.0056mol dm^{-3} in pH 5.4 buffer).

2.3 Tablet Assay

Weigh out accurately approximately 270 mg of the ground tablets recording the weight to 4 decimal places; quantitatively transfer to a 250 cm^3 volumetric flask and dissolve using the sodium oxalate solution (100 cm^3) and then make up to volume with more of the sodium oxalate solution. Pipette 5 cm^3 of this solution into a 50cm^3 volumetric flask and make up to volume with sodium oxalate solution (0.0056mol dm^{-3} in pH 5.4 buffer solution).

Pipette 4.0 cm^3 of this solution into a 100ml volumetric flask (prepare in duplicate), these will be the tablet analysis solutions.

Using the UV-visible spectrometer scan one of the standard solutions over a wavelength range of 200 – 800nm.

Select a suitable wavelength and measure the absorbance of all the standards and samples at this wavelength. Use these results to calculate the weight, in mg, of vitamin C per tablet.

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3.0 Determination of Zinc concentration in vitamin tablets.

3.1 Introduction

In this experiment the standard addition procedure will be used. Provided that linearity may be assumed, then the procedure of standard addition may be used. In this procedure, a known quantity of a standard (pure analyte) is added to a sample whose analyte concentration is to be determined. Both the sample solution and the sample + standard solution, known as the spiked solution, are measured and the increased parameter (emission in this case) is related to the amount of standard added. If C represents the concentration of analyte in the unknown sample, and C_s the concentration increase due to the addition of the standard, then C may be calculated as follows:

$$C_o = \frac{E_o . C_s}{E_s - E_o}$$

Where:
E_o and E_s are respectively the emission values obtained before and after addition of the standard
C_s is the increase in concentration due to the addition of a standard zinc solution
C_o is the concentration of analyte in the analysis solution.

3.2 Sample Preparation.

WEAR GLOVES, when handling acid solutions and the zinc standard solution as this is made up in nitric acid.

Weigh out accurately approximately 1 g of the tablet powder and transfer to a 100 cm^3 beaker, add (WEAR GLOVES) approximately 50 cm^3 of nitric acid (2M). Heat the tablet solution on a hot plate until completely dissolved (ensure there is a watch glass on top to avoid evaporation). When cooled, quantitatively transfer (WEAR GLOVES) to a 100 cm^3 volumetric flask and make up to volume with deionised water, this is solution Z.
Pipette 2 cm^3 of solution Z into a 100 cm^3 volumetric flask and make up to volume with deionised water.
Dilute a second 2 cm^3 aliquot of solution Z to obtain two acid digested tablet samples.

3.3 Preparation of spiked solutions

WEAR GLOVES, when handling acid solutions and the zinc standard solution as this is made up in nitric acid.

Pipette 2 cm^3 of the zinc stock solution, (1000ppm) into a 20 cm^3volumetric flask and make up to volume with deionised water this gives a zinc solution with a concentration of 100 ppm.
Pipette 2 cm^3 of the acid digested samples into each of two 100 cm^3 volumetric flasks, (WEAR GLOVES) add 5 cm^3 of the 100ppm zinc solution to each flask and make both flasks up to volume with deionised water, these are the spiked solutions.

Analyse the four, two acid digested solutions plus two spiked solutions, prepared solutions by ICP-OES.

Use these results to calculate the weight, in mg, of zinc per tablet.