Queen Mary University of London

East Anglia Region of the RSC Analytical Division

National Schools' Analyst Competition Regional Heat

Queen Mary University of London, Friday 24th February 2017

School of Biological and Chemical Sciences, Queen Mary University of London, E1 4NS

Analytical Chemistry Trust Fund

DOMINO

Royal Society of Chemistry

Use this space for rough working and calculations

Volumetric Assay of Vitamin C in Tablets

School Team:

HAND-IN THIS SHEET AT THE END WITH THE COSHH FORM SIGNED BY ALL STUDENTS

1 Aims

To give experience in the preparation of standard solutions.
To carry out accurate volumetric work in the proper use of a pipette and burette.
To set out assay calculations clearly and correctly in arriving at a final answer.

2 Objectives

To determine the Vitamin C content in the provided tablets.

3 Introduction

3.1 Standardisation of sodium thiosulfate

Potassium iodate is an oxidant and a *primary chemical standard* in titrimetric analysis. Thus it reacts quantitatively with iodides in acid solution liberating iodine.

$$IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 6\ H_2O \qquad (1)$$

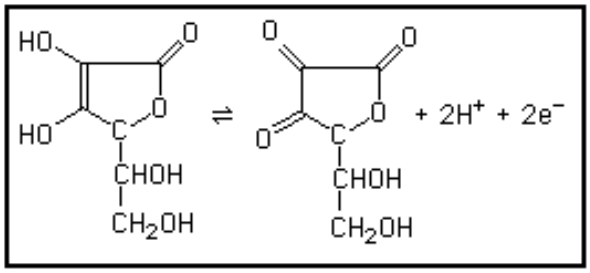
The liberated iodine can then be titrated with a reducing agent, sodium thiosulfate, according to the equation.

$$I_2 + 2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ I^- \qquad (2)$$

Sodium thiosulfate is thus standardised against a *primary standard*, potassium iodate.

3.2 Assay of Vitamin C

Ascorbic acid (Vitamin C) is oxidised to dehydroascorbic acid (dHAA) by iodine in acid solution according to the following equations.



(3)

ascorbic acid dehydroascorbic acid

$$I_2 + 2e^- \rightarrow 2\ I^- \qquad (4)$$

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 iodide is determined by titration with a standard solution of sodium thiosulfate, using starch as the indicator.

4 Experimental

4.1 Hazard Data

Important Points

1. Potassium iodide is poisonous by intravenous route, moderately toxic by ingestion.

2. Sodium thiosulfate is moderately toxic by intravenous route.

4.2 Reagents

Vitamin C tablets (× 3)
Dry *AR* potassium iodate (~1 g in a sample vial)
AR anhydrous sodium thiosulfate pentahydrate (~13.7 g in a sample vial)
AR sodium carbonate (~0.05 g in a sample vial)
AR potassium iodide (~20 g)
starch indicator (~10 cm^3)
sulfuric acid (0.5 mol dm^{-3}, ~250 cm^3)
deionised water in wash bottle

4.3 Equipment

250 cm^3 stoppered 'iodine' flask (× 2)
250 cm^3 volumetric flask (× 2)
25.0 cm^3 pipette (× 1) and filler
50 cm^3 burette (× 1)
10 cm^3 measuring cylinder (× 1)
100 cm^3 measuring cylinder (× 1)
50 cm^3 beaker (× 2) for standard solutions
100 cm^3 beaker (× 1) for dissolving Vitamin C tablet
glass rod (× 1)
small glass funnel (× 2)
weighing boats
plastic pipettes or Pasteur pipettes and teats
weighing balances (2 dp and 4 dp)

4.3 Procedure

4.3.1 Preparation of solutions

4.3.1.1 Standard solution of potassium iodate

Weigh accurately (4 d.p.) by difference about 1 g of potassium iodate, transfer it to a 250 cm^3 volumetric flask via a small glass funnel, dissolve in water and dilute to volume once all the solid has dissolved. Stopper the flask and shake by inverting several times.

4.3.1.2 Solution of sodium thiosulfate

Transfer ~13.7 g (2 d.p) of sodium thiosulfate pentahydrate and about 0.05 g of sodium carbonate in water to a 250 cm^3 volumetric flask and dilute to volume using the same procedure as above.

4.3.2 Standardisation of the thiosulfate solution

Pipette 25.0 cm^3 of the standard iodate solution into a stoppered 'iodine flask' and add about 2 g (2 d.p.) of potassium iodide and 10 cm^3 of 0.5 M sulfuric acid. Titrate the liberated iodine against the thiosulfate solution, adding starch indicator towards the end when the solution is pale yellow and continue the titration until the blue-black colour is just discharged. Repeat until two titrations agree to within 1%. Use this formula to calculate the % difference between your *closest* two titre values.

$$\% \text{ Difference} = \frac{|\text{Titre 1} - \text{Titre 2}|}{\text{Mean Titre 1 \& 2}} \times 100$$

4.3.3 Assay of ascorbic acid (Vitamin C)

Select one Vitamin C tablet and place in a 100cm^3 beaker and dissolve in 60 cm^3 of 0.5 M sulfuric acid. You will need to use a glass rod to disperse the tablet (ignore any small insoluble residue), ensuring that you wash the end of the rod with water to avoid any loss of material. Transfer the contents of the beaker *quantitatively* to a stoppered iodine flask. Pipette 25.0 cm^3 of the iodate solution together with ~2 g (2 d.p.) 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.

5 Calculations

Determine the molarity of the potassium iodate (KIO_3) solution.

5.1 Molarity of sodium thiosulfate

By examining equations (1) and (2), determine the stoichiometry (reaction ratio) between (sodium) thiosulfate ($S_2O_3^{2-}$) and (potassium) iodate (IO_3^-). Hence calculate the exact molarity of the sodium thiosulfate solution. **Remember that the titre obtained is for 25.0 cm^3 of the potassium iodate solution.**

5.2 Determination of Vitamin C in tablets

From equation (1) determine moles of iodine added to each solution (**A**), as determined by the moles of potassium iodate (KIO_3) added to each solution.

Next determine excess (left over) moles of iodine (**B**) in each solution from equation (2).

The difference, **A**–**B**, is the moles of iodine that have reacted with the ascorbic acid and hence the moles of ascorbic acid in each solution can be determined by combining equations (3) and (4).

Calculate mass of ascorbic acid [molar mass (M_r) = 176.12 g mol^{-1}] in each assay solution and so the average mass of ascorbic acid per tablet.

6 Questions

1) What are *AR* reagents?
2) What is a *primary chemical standard* and what properties should it possess?
3) Why is it important to invert (shake well) a standard solution several times before use?

QUEEN MARY UNIVERSITY OF LONDON
Control of Substances Hazardous to Health Regulations 2002
Risk Assessment Form

Project/Experiment title: Volumetric Assay of Vitamin C in Tablets	Experiment number:
	Note book Number:

		Tick Quantity (g or mL)			
	List chemicals in use and use appropriate precautionary and hazard statements from the safety phrase list to describe the associated risk.	<1	1–20	20–100	>100
1	potassium iodate (H272 H315 H319 H335 – P220 P261 P305 + P351 + P338)	✓			
2	potassium iodide		✓		
3	sodium thiosulfate pentahydrate			✓	
4	sulfuric acid (~0.5 M) (H314 – P280 P305 + P351 + P338 P310)		✓		
5	starch solution	✓			
6	Vitamin C		✓		

PRECAUTIONS REQUIRED FOR EACH CHEMICAL IN USE (tick if necessary)										
Chemical No.	1	2	3	4	5	6	7	8	9	10
Gloves	✓	✓	✓	✓	✓	✓				
Fume hood										
Safety shield										
*Special laboratory										
* Other										
*Specify										

I have assessed and understand the risk of using the chemicals as listed above and will carry out the experiment in accordance with this assessment. I undertake to follow good laboratory practise and the Health and Safety guidelines laid down by QMUL and the SBCS.

Student Name:				Supervisor Name: Dr TS Sheriff
Student Signature:				Supervisor Signature: 
Date: 24th February 2017				Date: 21st February 2017

References consulted:			
1		3	
2		4	

THIS RISK ASSESSMENT IS NOT TO BE USED AS A SUBSTITUTE FOR A FULL RISK ASSESSMENT OF A PROJECT. Copies to: departmental Safety Officer and supervisor