Experiment 8

DETERMINATION OF VITAMIN C IN A TABLET

2 lab periods

Reading: Chapter 15, pg 347 and 351-355. Chapter 1 pg 22-24, Quantitative Chemical Analysis, 8th Edition, Daniel C. Harris (7th Edition: Chapter 16, pg 334-335 and 340-334. Chapter 7 pg 121-125).

Objective

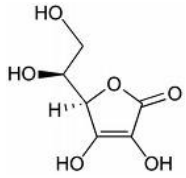
This lab will introduce you to the concepts of redox titrations and back titrations. You will determine the vitamin C content of a vitamin tablet by taking advantage of the redox reaction between I_3^- and ascorbic acid.

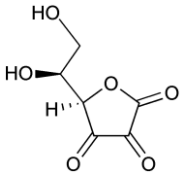
Suggested Schedule

Lab 1 Dry KIO_3 and prepare your solutions. You may wish to finish your titrations on this day.

Lab 2 Finish your titrations. ***Make sure you understand how to do your calculations before you leave the lab.***

Vitamin C (L-ascorbic acid) has received much attention, and it has been claimed that it can cure various diseases, ranging from the common cold to cancer. It is known that vitamin C is an antioxidant and is required for connective tissue synthesis. It is also used for treatment of rheumatoid arthritis. Ascorbic acid is readily oxidized by iodine, I_2, or triiodide, I_3^-, in an acidic solution:





Reaction 1 $\mathbf{C_6H_8O_6} + I_3^- \rightarrow \mathbf{C_6H_6O_6} + 3I^- + 2H^+$

L-ascorbic acid **dehydroascorbic acid**

In this experiment, you will take advantage of this oxidation reaction to determine the L-ascorbic acid content of a vitamin tablet. The experimental technique you will be using is known as a *back titration*. In a back titration, the analyte is consumed using a *known excess* of a reactant (reactant 1), and excess reactant 1 is titrated using a second reactant to determine the amount of reactant 1 left in solution. This technique might be used when the endpoint of the first titration is hard to determine.

You will not titrate your sample directly with iodine, because iodine is relatively insoluble. However, iodate (IO_3^-) reacts with iodide to form triiodide in solution:

Reaction 2 $IO_3^- + 8I^- + 6H^+ \rightarrow 3I_3^- + 3H_2O$

You will use the above reaction to generate a *known excess* of triiodide in your ascorbic acid solution. The triiodide will react with ascorbic acid to form dehydroascorbic acid until all the ascorbic acid in solution is consumed. The *excess* triiodide, which did not react with the ascorbic acid, will then be *back titrated* with standard sodium thiosulfate solution:

Reaction 3 $2S_2O_3^{2-} + I_3^- \rightarrow S_4O_6^{2-} + 3I^-$

Triiodide forms a dark blue complex with starch, and the endpoint of the titration can be detected by the disappearance of the blue color. By knowing the total quantity of triiodide formed and the quantity left after reaction with ascorbic acid, the number of moles of triiodide that reacted with the vitamin C can be calculated. Once we know the number of moles of triiodide that reacted with ascorbic acid, we can, of course, calculate the concentration of ascorbic acid in the solution.

Prelaboratory Assignment

A 25.00-mL sample of orange drink with 2 g of KI and 25.00 mL of 0.0131 M KIO_3 added was back-titrated with a 0.0302 M sodium thiosulfate solution. The titration took 31.09 mL of thiosulfate. Calculate the L-ascorbic acid concentration in the orange drink in mg/mL.

Apparatus

- 50-mL buret
- 25-mL pipet
- 4 250-mL Erlenmeyer flasks
- 500-mL volumetric flask
- 250-mL volumetric flask
- 600-mL beaker
- Weighing bottle
- 1-L amber bottle
- Mortar & pestle

Chemicals

- "Ecofoam" pellets
- Potassium iodate, KIO_3
- Potassium iodide, KI
- Sodium thiosulfate, $Na_2S_2O_3$ or $Na_2S_2O_3 \cdot 5\ H_2O$
- Oxalic acid, $H_2C_2O_4$
- Sulfuric acid, H_2SO_4, ~ 0.6 *M*
- Sodium carbonate, Na_2CO_3
- Vitamin C tablets

Preparation and Standardization of $Na_2S_2O_3$ Titrant

1. Dry ~3 g of KIO_3 at 110°C to constant weight. Completely cool in a desiccator.
2. ~~Prepare the starch indicator by dissolving about 10 Ecofoam pellets in 500 mL of water. Note that these don't totally dissolve, so you'll have white flocculant material in your flask or bottle. Mix well and then allow the white material to settle to the bottom of the flask or bottle.~~ Starch indicator supplied by Lab Services.
3. Boil 500 mL water for 5 min. Weigh sufficient $Na_2S_2O_3 \cdot 5H_2O$ to make a 0.03-*M* solution and add to the boiled water. Add 35 mg Na_2CO_3 and store the solution in the dark in a l-L amber bottle. This solution is not stable and must be made fresh after ~1 week.
4. Weigh out ~0.5 g KIO_3 to ±0.1 mg. Quantitatively transfer to a 500-mL volumetric flask and dilute to the mark with water. Mix well.

5. Pipet 25.00 mL of KIO_3 solution into a 250-mL Erlenmeyer flask and add roughly 2 g KI and then 10 mL of 0.6 *M* H_2SO_4 using a graduated cylinder. Fill the buret with $Na_2S_2O_3$ solution and slowly titrate until the solution in the flask is pale yellow. At this point, add 2 mL of starch solution. It is easiest to do this using a plastic dropper to avoid getting chunks of white material in your analyte solution. The addition of starch should turn your solution dark blue. Continue adding titrant dropwise very slowly until the dark color just disappears and the solution turns clear.
6. Repeat step 5 two more times.

Determination of Vitamin C in a Tablet

Note: When conducting analyses on pharmaceutical samples for the purpose of quality control, an analyst's first task is to devise an appropriate method to prepare a representative sample. Normally, this would be done by selecting perhaps 20 tablets at random from the bulk sample and grinding them into a uniform powder, then taking a sample for analysis. The analysis of the powdered sample is used to determine the average mass of the analyte of interest per tablet and the variance of the mass in the lot. Here, you will be given several tablets (three or four) of one brand of vitamin C for your quantitative analysis.

1. Weigh the tablets together, record the weight, and then grind all tablets to a powder with a mortar and pestle.
2. Weigh (to ± 0.1 mg) approximately 0.6 g of the powder into a small beaker, dissolve in 10-20 mL 0.6 *M* H_2SO_4 and carefully transfer to a 500-mL volumetric flask. Dilute to the mark with distilled water. This is your stock solution.
3. Pipet 10 mL of the stock solution into an Erlenmeyer flask and ***carefully*** add about 0.1 g of oxalic acid.

   WARNING*: Oxalic acid is toxic. Handle it with care, and wash your hands after using it.*
4. Into this Erlenmeyer flask, pipet 25.00 mL of the standard KIO_3 solution, and add about 2 g of KI and 10 mL of 0.6 M H_2SO_4.
5. Titrate with $Na_2S_2O_3$ solution as before, adding 2 mL of starch solution just prior to the endpoint. Record the volume of titrant used.
6. Repeat steps 3-5 two more times, for a total of three measurements.

Calculations

1. Calculate the molar concentration of the standard KIO_3 solution.
2. Calculate the molar concentration of the $Na_2S_2O_3$ titrant from the concentration of the KIO_3 solution and the volume of titrant needed to titrate 25 mL of the standard KIO_3. Report your values for the concentration of $Na_2S_2O_3$, and the mean, standard deviation, and relative standard deviation of these results.
3. Calculate the molar concentration of ascorbic acid in each of your sample solutions from the volume of $Na_2S_2O_3$ titrant required to titrate the excess iodine, knowing the total amount of KIO_3 which was added, and using the average molarity of the $Na_2S_2O_3$ titrant determined in step 2.
4. From each of your three determinations, calculate the mass (in milligrams) of ascorbic acid

in your sample. Determine the average mass of ascorbic acid per tablet, and the average %wt of ascorbic acid per tablet.

5. Is the average mass of ascorbic acid per tablet you calculated the same as that given by the manufacturer? Assuming that the manufacturer's stated value is correct, what are some reasons your value might be "off"? If your results were to differ from the manufacturer's "true" value, what could you do to show that your data is correct and the manufacturer's incorrect?

Questions

1. Why must starch be added immediately before the equivalence point instead of at the beginning of the titration?
2. Why must the water used to prepare sodium thiosulfate be boiled? Why is sodium carbonate used in the preparation of the sodium thiosulfate solution? Show the reaction that that thiosulfate could undergo if these steps were not taken. How would this affect your results?
3. Explain why oxalic acid is added to the ascorbic acid solution. How might your results be affected if oxalic acid was not added to the ascorbic acid solution?

Experiment 8 ***Determination of Vitamin C in a Tablet*** Name_____

Brand of Vitamin C:_____ Listed Vitamin C content:_____

Number of tablets used_____ Mass of all the tablets: _____g

Avg mass per tablet:_____ Mass sample used to prepare stock sol'n:_____g

Weight of KIO_3: _____ g Concentration of KIO_3 standard solution _____M

Moles of KIO_3 per 25.00 mL aliquot_____

Standardization of $Na_2S_2O_3$

Replicate	1	2	3
Moles I_3^- produced in Reaction 2			
Moles $Na_2S_2O_3$ required to reach endpoint (Reaction 3)			
Volume $Na_2S_2O_3$, mL			
$Na_2S_2O_3$ conc. M			

Mean $Na_2S_2O_3$ conc. _____*M* Standard deviation _____ %RSD _____%

Back titration of excess I_3^- after reaction with L-ascorbic acid

Replicate	1	2	3
Volume $Na_2S_2O_3$, mL			
Moles $Na_2S_2O_3$			
Moles I_3^- titrated (remaining after reaction with L-ascorbic acid)			
Moles I_3^- initial			
Moles I_3^-consumed (moles I_3^- initial − moles I_3^- after reaction)			
M ascorbic acid solution			
Ascorbic acid, mg			

Mean *mg* ascorbic acid in your sample:_____ ± _____
mg ascorbic acid per tablet in your brand of Vitamin C:_____ ±_____
Mean % ascorbic acid per tablet _____±_____ % RSD _____%

Include sample calculations, and answer all questions. Check your calculations carefully! Don't forget to include copies of your lab notebook pages for this experiment.