

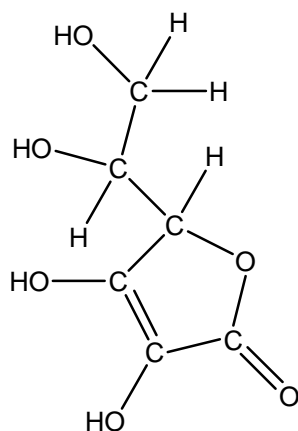
Determination of Vitamin C

prepared by M. J. Akhtar, R. C. Kerber and R. F. Schneider SUNY at Stony Brook (Rev 10/08)

Purpose of this Exercise: to determine the vitamin C content of fruit juices or of vitamin pills and an unknown sample, by use of oxidation-reduction titration.

Background Information

A vitamin is an organic compound which must be obtained from the diet in order to maintain good health. One such vitamin is vitamin C, ascorbic acid. All animal species except for primates (including humans), the guinea pig, the Indian fruit bat, and the red-vented bulbul bird, synthesize vitamin C for themselves. We, however, must obtain it from our food. Lack of vitamin C in the diet leads to a potentially fatal condition called scurvy. The recommended daily allowance of vitamin C for adults is 60 mg, but some scientists, especially the late Nobel prize-winning chemist Linus Pauling, believe that much more, up to several grams per day, should be taken. The structure of vitamin C is shown at the left.

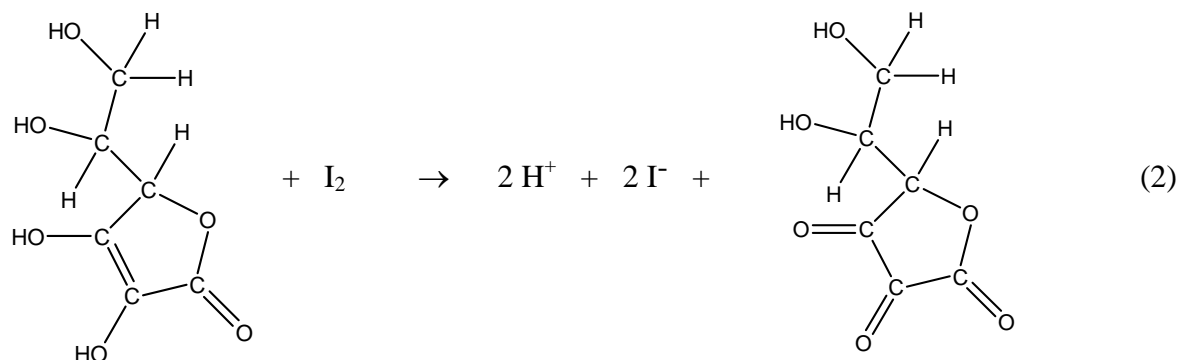


Orange juice is probably the most widely consumed source in the United States today. Pauling adherents who wish to consume a large quantity rely upon vitamin C pills, for which the vitamin is usually produced by microbiological fermentation. "Organic" vitamin C can be isolated from plant sources such as rose hips, at somewhat greater cost.

The vitamin has many roles in the body, not all of them well understood. One of its roles is as a water-soluble *antioxidant*. Its reactivity as a reducing agent provides the chemical basis for our determination of vitamin C. The oxidizing agent which we will use to react with vitamin C is iodine. It is not practical to prepare standard solutions of iodine, however, because iodine is volatile and not very soluble in water. We will therefore generate the iodine in the presence of the vitamin C by the reaction:



As soon as molecular iodine forms, it will react with vitamin C according to:



The product is sometimes called de-hydro ascorbic acid. (Note that the two hydroxyl groups on the five membered ring (C—OH) have been converted to carbonyl groups (C=O))

When all of the vitamin C has been consumed, the excess iodine will react with iodide ion and starch indicator to produce a deep blue colored complex, which signals the end of the titration.

There are **three** parts to this exercise. In the **first** part, you will titrate a pure vitamin C sample with potassium iodate (KIO₃) solution to determine the *titer value*. This is the *weight of vitamin C per volume of KIO₃ solution*. In the **second** part, you will analyze different juices or vitamin C pills and use the average titer value from the first part to calculate their vitamin C content. The **third** part will consist of your determining the vitamin C content of an unknown solid mixture, again using the average titer value from the first part.

NOTE: While juices are a common source of vitamin C, you are asked to bring in vitamin C **pills** for this exercise. If you brought in juice, you would need to bring a sufficient quantity to be able to do *three titrations* of juice samples each containing *100 mg of vitamin C*. The labels of most juices display the amount of vitamin C in terms of the percent of the recommended daily allowance in a given volume of the juice (often specified in fluid ounces). The recommended daily allowance is 60 mg and 1 fluid ounce = 29.6 mL.

The following calculation shows how much of a typical juice would be necessary to use in this exercise.

A certain juice indicates that one 8 fl oz serving contains the minimum daily requirement of vitamin C. How many mL of that juice will contain 100 mg of vitamin C?

$$8 \text{ fl oz} = 8 \text{ fl oz} \times 29.6 \text{ ml/fl oz} = 240 \text{ mL}$$

This volume contains 60 mg. The volume of juice that contains 100 mg of vitamin C is

$$240 \text{ mL} \times 100 \text{ mg} / 60 \text{ mg} = 400 \text{ mL}$$

We would need 400 mL samples of juice if we wished to titrate 100 mg of vitamin C in each titration.. Since we would wish to repeat the titration three times, we would need 3 X 400 mL, or 1,200 mL, or 1.2 L – i.e., more than one quart of this juice.

Procedures

1. Drain a buret, rinse it with standard (about 0.01 M.) potassium iodate (KIO₃) solution, and then fill it with the same solution. Be sure there are no bubbles in the tip of the buret. Record the exact concentration from the label.
2. Weighing by difference, *accurately* weigh about 0.1 g (100 ± 20 mg) of pure vitamin C (ascorbic acid) into an Erlenmeyer flask. Add about 150 mL of distilled water to dissolve the solid.

3. Add the following reagents to the flask: about 5 mL of 1.0 M. HCl, about 10 mL of 0.60 M. potassium iodide (KI), and 10-15 drops of starch indicator. (Starch solutions vary in their efficiency. Be sure to check on the recommended value.) Take the initial buret reading and record it in your notebook.

4. Titrate the solution with the potassium iodate, adding small portions until the solution in the flask assumes a permanent blue color. Record the final buret reading. Calculate the titer value (mg vitamin C / mL KIO_3 solution).

5. Repeat steps 2-4 using another sample of vitamin C. If your titer values are consistent within 1%, you may proceed to step 6; otherwise, repeat until a consistent value is obtained. Since the analysis of your unknown depends on the accuracy of the titer value you determine, you may wish to repeat the determination of the titer of the KIO_3 solution a third time regardless of the agreement of the first two determinations.

6. Unknown

This part of the exercise has you determine the percentage of vitamin C in a solid mixture. Weigh out about a 0.15 g portion, dissolve in 150 mL water, add the necessary reagents, and titrate as before. Since the unknowns vary widely in the percent of vitamin C, you may need to adjust the amount of unknown that you weigh out for subsequent determinations. Remember that the objective is to use about 25 mL of the potassium iodate solution to realize the full precision of the buret.

Determine the amount of vitamin C in appropriate samples of the unknown by using the average value of the titer that you determined, and from that, the percentage of vitamin C in the unknown sample.

7. Commercial Vitamin C Product

If using vitamin C pills, **record the nature of the sample in detail**. Weigh a **whole tablet**, grind it up completely to assure that the resulting powder is of uniform composition. Then *accurately* weigh out about a 0.15 g portion, dissolve in 150 mL water (the pill may contain fillers that are not soluble), add the necessary reagents, and titrate as before. Determine the amount of vitamin C in the tablet by using the average value of the titer that you determined, and compare with the label information. Repeat the determination to establish reliability.

Calculations:

33.47 mL of a KIO_3 solution is required to titrate a 107.5 mg sample of vitamin C.

The titer of this KIO_3 solution is $107.5 \text{ mg} / 33.47 \text{ mL} = \mathbf{3.212 \text{ mg/mL}}$

24.56 mL of this KIO_3 solution is required to titrate a 115.8 mg sample of a vitamin C pill that weighs 647.0 mg

This sample of the pill contained $24.56 \text{ mL} * \mathbf{3.212 \text{ mg/mL}} = 78.89 \text{ mg}$ of vitamin C

Assuming the composition of the sample was the same as the composition of the entire pill, the entire pill contained

$$(647.0 / 115.8) \times 78.89 = 440.8\text{mg of vitamin}$$

SUSB-018 Data Sheet - 1

Notebook Grade: _____

Safety Grade: _____

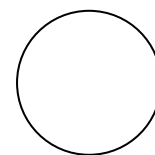
Name _____ Section _____ Date _____

Titer Determination

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>
Mass of pure vitamin C	_____ mg	_____ mg	_____ mg
Final buret reading	_____ mL	_____ mL	_____ mL
Initial buret reading	_____ mL	_____ mL	_____ mL
Net volume of titrant	_____ mL	_____ mL	_____ mL
Titer value	_____ mg/mL	_____ mg/mL	_____ mg/mL

Average titer value _____ mg/mL

Average Deviation _____ mg/mL



Identification of Unknown

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>
Initial mass of vial	_____ g	_____ g	_____ g
Final mass of vial	_____ g	_____ g	_____ g
Mass of unknown	_____ mg	_____ mg	_____ mg
Final buret reading	_____ mL	_____ mL	_____ mL
Initial buret reading	_____ mL	_____ mL	_____ mL
Net volume of titrant	_____ mL	_____ mL	_____ mL

Quantity of vitamin C in sample _____ mg

Percentage of vitamin C in unknown _____ %

Average Percent _____

Average Deviation _____

Percent Deviation _____

SUSB-018 Data Sheet - 2

Type of tablet studied: _____

Nominal Vitamin C content: _____

	<u>Run 1</u>	<u>Run 2</u>
Mass of whole tablet	_____ mg	_____ mg
Mass used in titration	_____ mg	_____ mg
Final buret reading	_____ mL	_____ mL
Initial buret reading	_____ mL	_____ mL
Net volume of titrant	_____ mL	_____ mL
Quantity of vitamin C in sample	_____ mg	_____ mg
Quantity of vitamin C in tablet	_____ mg	_____ mg

Comment on Comparison with nominal content:

SUSB-018 Pre-Laboratory Assignment

Name

Section

Date

1. The pK_a of vitamin C (ascorbic acid) is 4.17. Could we determine the amount of vitamin C in **orange juice** by acid-base titration? Why or why not?
2. Could we determine the amount of vitamin C in a **vitamin C tablet** by acid-base titration? Why or why not?
3. Based on the stoichiometry given in equations 1 and 2, what do you expect the titer value of a 0.01000 M KIO_3 solution to be?
4. 16 fl oz. of a certain sample of juice contains the recommended daily allowance (60 mg). Given that 1 quart = 0.95 L and that there are 32 fl oz. in a quart, what volume of this juice (in mL) will contain 100 mg of vitamin C? How much of this juice will a student need to bring to laboratory to be able to do 3 titrations on samples of the juice each containing 100 mg of vitamin C?
5. A student brings in (purple) grape juice for this exercise. Do you expect that the student will be able to detect the end point of the titration? Why or why not?

