8. MEASURING VITAMIN C—A REDOX TITRATION

Initial Question

Vitamin C, also called *L-ascorbic acid*, is found in many foods. Most people expect orange juice to be the best source of vitamin C but other berries have a higher vitamin C content and juice drinks like Hi-C® have a great deal of added vitamin C. In these cases, it is difficult to perform a traditional acid–base titration because there may be more than one acid present. How can we accurately measure the vitamin C content present in foods?

What foods have the highest levels of vitamin C?

Materials and Equipment

Model 1

- Data collection system
- Oxidation reduction potential (ORP) probe
- Beaker (5 for the entire class), 250-mL
- Beaker, 150-mL
- 0.25 % Iodine (I_2) solution, 50 mL for entire class
- 0.01 M L-Ascorbic acid ($C_6H_8O_6$), 50 mL for the entire class
- 3% Hydrogen peroxide (H_2O_2), 50 mL for entire class
- 0.01 M Potassium permanganate ($KMnO_4$), 50 mL for the entire class
- 1.0 M Sodium chloride (NaCl), 50 mL for the entire class
- Distilled water, 50 mL

Model 2 and Applying Your Knowledge

- Data collection system
- Oxidation reduction potential (ORP) probe
- Drop counter
- Drop dispenser:
  Syringe, 60-mL
  Stopcock (2)
  Drop tip
- Multiclamp
- Three-finger clamp
- Ring stand
- Beaker, 150-mL
- Magnetic stir plate and micro stir bar
- Analytical balance
- Materials for drop counter and pH sensor calibration (refer to Appendix A)

Model 2

0.25 % Iodine (I_2) solution, 70 mL[1]

L-Ascorbic acid ($C_6H_8O_6$), 0.040–0.060 g

- Distilled water, 75 mL

Applying Your Knowledge

- 0.25 % Iodine (I_2) solution, as needed
- Foods or juices for vitamin C analysis
- Juicer
- Knife (for slicing fruit)

Safety

Add these important safety precautions to your normal laboratory procedures:

- $KMnO_4$ is a strong oxidizer and should be treated as particularly hazardous. If the solution comes in contact with your skin, rinse immediately with a large amount of running water.

Getting Your Brain in Gear

1. Without consulting your textbook or the Internet, give your best definition of the word “titration.”

____

____

____

2. The equation for the reaction between elemental iodine and vitamin C, $C_6H_8O_6$, is

$$I_2(aq) + C_6H_8O_6 \rightarrow 2HI + C_6H_6O_6$$

   a. What are the oxidation states of iodine on both sides of the equation?

____

____

   b. Is I_2 oxidized or reduced in this reaction?

____

   c. Write the half-reaction for I_2 in this oxidation–reduction (or redox) reaction.

____

   d. Given your answer to part b, is vitamin C oxidized or reduced during this reaction? Briefly explain your answer.

____

____

____

MODEL 1

Building Model 1 – Measuring the Oxidation Reduction Potential of a Solution

1. Connect the oxidation reduction potential probe (ORP) to your data collection system.

2. The ORP probe measures ISE voltage. Monitor live ISE voltage data without recording.

3. Use the ORP probe to measure the oxidation reduction potential following solutions: $KMnO_4$, H_2O_2, vitamin C, NaCl and distilled H_2O. Rinse the probe with distilled water between each measurement. Record the potential in the Model 1 Data Table.

Model 1 – Measuring the Oxidation Reduction Potential of a Solution

Table 1: Model 1 Data Table—Oxidation reduction potential of various solutions

Solution	Oxidation–Reduction Potential (ISE, mV)	Oxidizer (Strong/Not Strong)
I_2 (aq)		
Vitamin C(aq)		
H_2O_2 (aq)		
$KMnO_4$		
NaCl(aq)		
Distilled H_2O		

Analyzing Model 1 – Measuring the Oxidation Reduction Potential of a Solution

4. What solutions in the Model 1 Data Table would you group together as having high oxidation reduction potentials and which would you group together as having low potentials?

____

____

5. Potassium permanganate, $KMnO_4$, and hydrogen peroxide, H_2O_2, are both strong oxidizers. This means that they often cause other compounds to be oxidized while they are reduced. Strong oxidizers are highly reactive with other compounds, must not be ingested, and must be stored carefully. Vitamin C and water are not strong oxidizers.

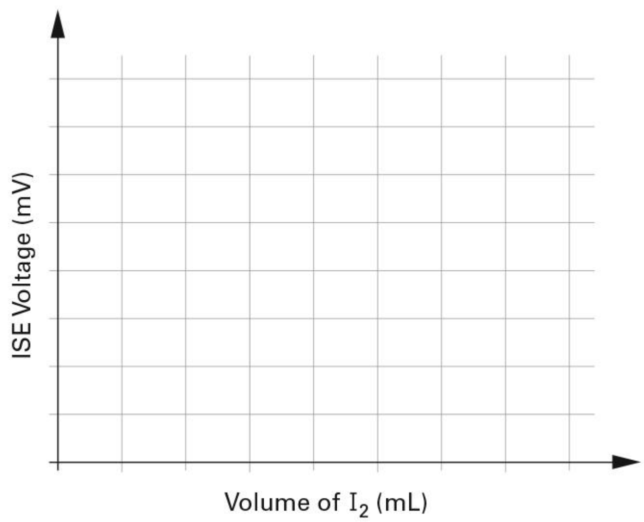
   a. You should see a pattern between the oxidation reduction potential and oxidation strength of the solutions mentioned. Using this pattern, indicate in the Model 1 Data Table whether each solution is a strong oxidizer "s" or not a strong oxidizer "ns".

   NOTE: Strong oxidizers are greater than 250 mV.

b. Based on this information, what is the ORP probe actually measuring?

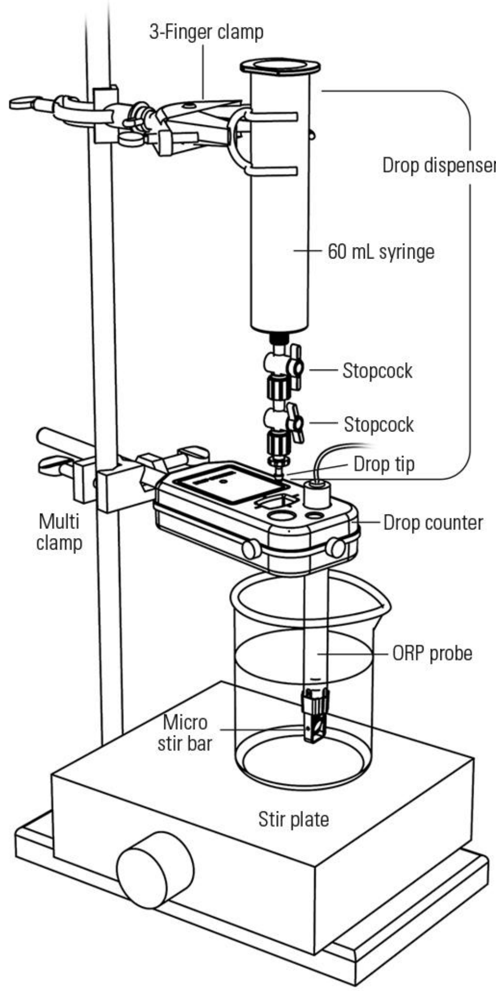
6. Is aqueous sodium chloride a strong oxidizer? Does this make sense with what you know about NaCl?

7. Model 2 involves adding iodine, I_2, solution to the vitamin C solution until all of the vitamin C is consumed. On the blank graph below, sketch your prediction for the shape of the graph of ISE voltage versus the volume of iodine added to the vitamin C solution.



MODEL 2

Building Model 2 – Reacting Vitamin C with Iodine



1. Obtain about 60 mL of iodine solution in a beaker.

2. Obtain a drop dispenser and a drop counter. Set up the drop counter as shown in the diagram.

3. Connect the drop counter to the data collection system.

4. Display ISE voltage on the *y*-axis of a graph and fluid volume on the *x*-axis.

5. Rinse the drop dispenser three times with 2 to 3 mL of distilled water and then rinse it three times with 2 to 3 mL of I_2 solution.

6. Fill the drop dispenser with the I_2 solution.

7. Set the flow rate of the drop dispenser and calibrate the drop counter as described in Appendix A.

   NOTE: Do not disconnect the drop counter from the data collection system or it will need to be calibrated again.

8. Obtain between 0.040 and 0.060 g of L-ascorbic acid, vitamin C, and dissolve it in about 75 mL of distilled water in a 150-mL beaker. Record the exact mass of L-ascorbic acid in the Model 2 Data Table below.

   NOTE: If you are using vitamin C tablets rather than L-ascorbic acid, make sure to convert the mass of the tablet measured to the mass of vitamin C, as vitamin C tablets contain binders and other inert ingredients which will not interfere with the titration but do add mass.

9. Obtain a stir plate and stir bar. Add the stir bar to the beaker containing the vitamin C solution. Turn on the magnetic stirrer at a slow and steady rate.

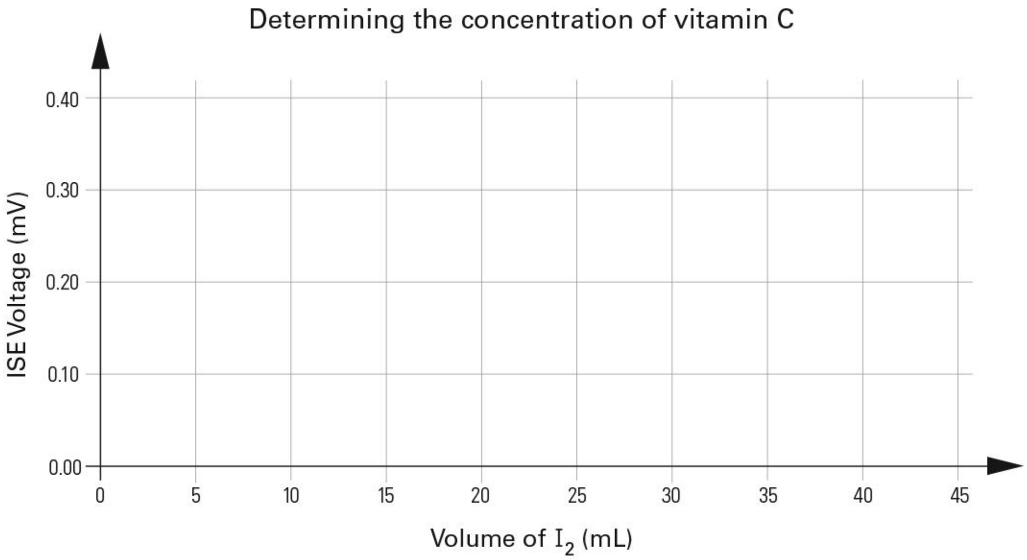
10. Start recording data. Turn the drop dispenser stopcock carefully, allowing the I_2 solution to drip slowly at a rate of 1 to 2 drops per second into the vitamin C solution.

    NOTE: The top valve controls the flow rate and the bottom valve turns the flow on and off.

11. Sketch or attach a copy of the graph of ISE voltage versus volume of $KMnO_4$ in the Model 2 data section.

Model 2 – Reacting Vitamin C with Iodine

Mass of vitamin C: ____________________



Analyzing Model 2 – Reacting Vitamin C with Iodine

12. a. Does your observed graph match your predicted graph? Explain any differences.

____

____

____

b. What type of graph does your data resemble?

____

____

13. As you have probably guessed by now, the experiment you just conducted was a titration.

a. Does this experiment fall under the definition of “titration” you gave in the “Getting Your Brain in Gear” question?

____

b. Revise your definition of titration based on your experience in lab. Avoid using any reference to color changes or the equipment you use.

____

____

____

14. At what volume of I_2 is all of the vitamin C consumed? How does the graph indicate this?

15. Using the measured mass of vitamin C, calculate the following:

    a. The number of moles of iodine necessary to react completely with the vitamin C.

    b. The molarity of the iodine solution.

16. Collect the molarity of I_2 from other groups.

Table 2: Compare class results of the molarity of the iodine solution

Group	I_2 Concentration (M)
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	

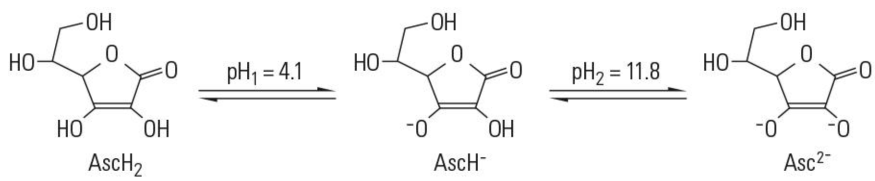
    a. Calculate the standard deviation.

b. Remove molarities that are outside of the standard deviation and then calculate the average concentration. You will use this value for the calculations for the Applying Your Knowledge experiment. This process of determining the concentration of a solution to be used in a later experiment is called *standardization*.

Average molarity of the I_2 standard solution : ______________________

Connecting to Theory

Vitamin C functions as an anti-oxidant in humans. This means that it causes reduction to occur in other substances. However it only displays weak anti-oxidant abilities, and like weak acids and bases, the oxidation reduction potential of vitamin C is reversible. The anti-oxidant reversibility is a function of pH.



The illustration is a simplified version of the pathway the ascorbate ion undergoes. Eight forms of the ion have been categorized. But as you can see, there are two major changes, one at pH of 4.1 and one at pH 11.8. The middle figure is dominant at pH 7.4. The ability of vitamin C to cause reduction makes it an ideal candidate for ORP titration.

Applying Your Knowledge – Determining the Concentration of Vitamin C in Juice

1. Design an experiment that uses the standardized iodine solution from Model 2. Create a hypothesis you will test related to the vitamin C content of available materials.

2. Once you know what materials are available, answer the following questions *before* beginning the experiment.

   a. What hypothesis would you like to test?

   ____

   ____

   ____

   b. What data will you need to collect?

   ____

   ____

   ____

   c. What conditions will need to be held constant between your two titrations?

   ____

   ____

   ____

3. Outline the procedure of your experiment.

4. Carry out the experiment to test your hypothesis. Record your data. Modify the data table as needed.

Molarity of I_2: 0.0119 M

Table 3: Data Table

5. Provide calculations for your experiment that either prove or disprove your hypothesis.

6. Compare your experiment to others in class. How could your experiment have been improved?

7. Vitamin C is an acid. Why was it necessary in this lab to use the ORP probe rather than the pH sensor?