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CH 362
Professor Attygalle
I pledge my honor that I have abided by the Stevens Honor System.

1) Title of experiment:

Quantitative Determination of Proportion of Ascorbic Acid in Commercial Vitamin C Supplements using Iodometric Titration

Date: September 3, 2020
Name of technique: Iodometric Titration

2) Technique:

Titration is a technique for which a solution of known concentration is used to determine the concentration of an unknown solution. The setup for this technique involves a buret and an Erlenmeyer flask. Either one of the solutions is placed in the buret, while the other is placed in the flask. Often, a color indicator will be added to the solution in the flask, in order to determine the stopping point for the titration. The solution in the buret, called the titrant, is added in small quantities to the solution in the flask, called the analyte, by opening the stopcock while swirling the flask. This is continued until the reaction is complete, usually signified by a change in color from the indicator. This point, called the equivalence point, allows measurements to be taken from the titrant to determine the concentration of the unknown solution.

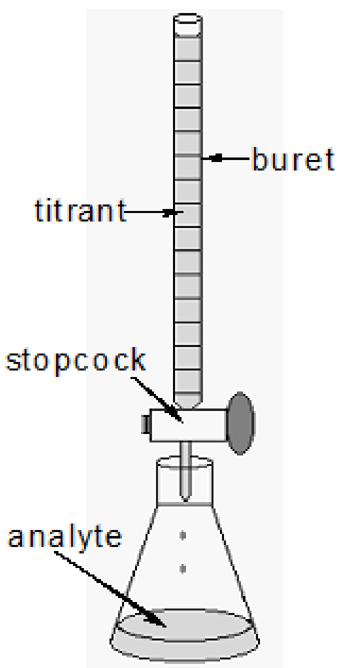


Figure 1: Titration Setup

Using the volume of titrant used, the volume of the solution placed in the Erlenmeyer flask, and concentration of the unknown solution can be calculated using the concentration of the known solution, also called the standard solution.

Iodometric titration, in particular, takes advantage of a reducing agent to neutralize the colored triiodide ion in solution, which turns a deep blue when mixed with starch. Measurement of the quantity of a reducing agent is easily done with titration.

3) Application of the Technique to my Experiment:

In order to calculate the proportion of ascorbic acid in the vitamin C tablet, iodometric titration is an ideal candidate to use, taking advantage of the redox reaction that occurs between ascorbic acid and I_3^-, as such:

Equation 1: Oxidation of Ascorbic Acid

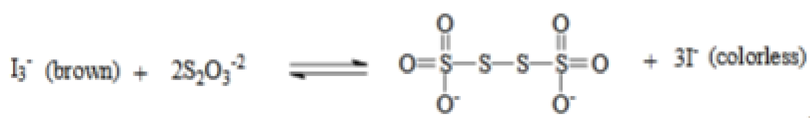
With this idea, and a certain amount of vitamin C tablet can be reacted with a solution of I_3^- to remove some, but not all, of the I_3^- ion. As the I_3^- ion produces a brown colored solution, which turns deep blue in the presence of starch, removal of this ion into colorless I^- creates an ideal color indicator for this titration. Thus, another standard solution of reducing agent can be used to react the rest of the I_3^- ion, resulting in a determination of the amount of ascorbic acid originally present in the vitamin C tablet.

This procedure, therefore, requires preparation of an I_3^- solution. This can be prepared with KIO_3, KI, and acid, in this reaction:

$$IO_3^- + 6H^+ + 8I^- \rightleftharpoons 3I_3^- + 3H_2O$$

Equation 2: Redox Reaction of Iodate and Iodide in Acid

As for the other reducing agent used to titrate the rest of the triiodide solution after the ascorbic acid has reacted with some of the triiodide ion, the given thiosulfate solution will be used, according to this reaction:

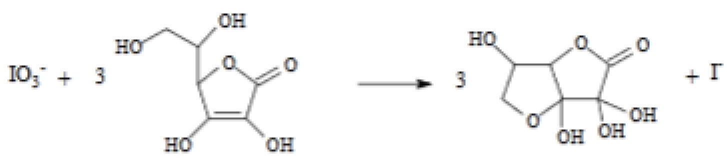


Equation 3: Redox Reaction of Triiodide and Thiosulfate

The last piece of information needed for this experiment is to standardize the unknown thiosulfate solution. This can be done by titrating a solution of only the triiodide without adding ascorbic acid. Equation 2 and 3 combine to form a stoichiometric ratio:

Equation 4: Combined Equation of Potassium Iodate and Thiosulfate in Acid

This 1-6 ratio between potassium iodate and thiosulfate ion allows for the determination of the molarity of the thiosulfate solution, thus standardizing the solution for use in determination of the proportion of vitamin C in the tablets. Ascorbic acid reacts with the iodate ion in a 3-1 ratio, as seen in this combined equation:



Equation 5: Combined reaction of Iodate ion and Ascorbic Acid

This kind of procedure can be used with any reducing agent, as seen here with usage of both ascorbic acid and thiosulfate to react the triiodide ion into the iodide ion. This presents the limitations of this experiment, in that any other reducing agents present in solution will affect the error of the lab, as this will be counted as extra ascorbic acid reacting with the triiodide ion.

4) Calculations:

Preparation of 250.00mL 0.01M standard KIO_3 solution

Molecular weight of KIO_3	39.098 + 126.904 + 3(15.999) = 213.999 g/mol
Concentration of desired solution	0.01M
Volume of desired solution	250.00mL
Number of moles of KIO_3 required	0.01M * 0.25000 L = 0.0025 mol KIO_3
Mass of KIO_3 required	0.0025 mol * 213.999 g/mol = 0.535 g

5) References

1. figure9-1.png (PNG Image, 193 × 344 pixels) https://webassign.net/question_assets/ncsugenchem102labv1/lab_9/images/figure9-1.png (accessed Sep 4, 2020).
2. Harris, D. C. *Quantitative Chemical Analysis*, 8th ed.; W.H. Freeman and Co: New York, 2010. Chapter 10, 15.
3. Redox Titration - Chemistry LibreTexts https://chem.libretexts.org/Bookshelves/Ancillary_Materials/Demos_Techniques_and_Experiments/General_Lab_Techniques/Titration/Redox_Titration (accessed Sep 4, 2020).

6) MSDS

Sodium Thiosulfate:

CAS No.: 7772-98-7
Molecular Weight: 158.11
Chemical Formula: $Na_2S_2O_3$
Appearance: colorless liquid
Lab Protective Equipment: Lab coat, goggles

Health effects:
May cause eye, skin, and respiratory tract irritation

First Aid measures:
Eye contact: rinse immediately with water, especially under eyelids, for >15 minutes. Obtain

medical attention.
Skin contact: wash off immediately with plenty of water for >15 minutes. Get medical attention if symptoms occur.
Inhalation: Move to fresh air. If breathing is difficult, give oxygen. Get medical attention if symptoms occur.
Ingestion: Do not induce vomiting. Obtain medical attention.

Other hazards:
Fire: not a fire hazard.
Explosion: not an explosion hazard.

Potassium Iodate:

CAS No.: 7758-05-6
Molecular Weight: 214
Chemical Formula: KIO_3
Appearance: off-white powdered solid
Lab Protective Equipment: Lab coat, goggles

Health effects:
Causes skin and eye irritation, may cause central nervous system depression and adverse kidney effects.

First Aid measures:
Eye contact: rinse immediately with water, especially under eyelids, for >15 minutes. Obtain immediate medical attention.
Skin contact: wash off immediately with plenty of water for >15 minutes. Get immediate medical attention.
Inhalation: Move to fresh air. Get medical attention immediately if symptoms occur. If not breathing, give artificial respiration.
Ingestion: Do not induce vomiting. Call a physician or Poison Control Center immediately.

Other hazards:
Fire: Contact with combustible/organic material may cause fire. Runoff to sewer may create fire or explosion hazard.
Explosion: Containers may explode when heated. Risk of explosion by shock, friction, fire, or other sources of ignition. Runoff to sewer may create fire or explosion hazard.

Potassium Iodide:

CAS No.: 7681-11-0
Molecular Weight: 166
Chemical Formula: KI
Appearance: white solid
Lab Protective Equipment: Lab coat, goggles

Health effects:
May cause irritation.

First Aid measures:
Eye contact: rinse immediately with water, especially under eyelids, for >15 minutes. Obtain medical attention.
Skin contact: wash off immediately with plenty of water for >15 minutes. Get medical attention if symptoms occur.
Inhalation: Move to fresh air. Get medical attention immediately if symptoms occur. If not breathing, give artificial respiration.
Ingestion: Do not induce vomiting. Obtain medical attention.

Other hazards:
Fire: Not considered to be a fire hazard
Explosion: Not considered to be an explosion hazard.

Ascorbic Acid:

CAS No.: 50-81-7
Molecular Weight: 176.12
Chemical Formula: $C_6H_8O_6$
Appearance: white solid
Lab Protective Equipment: Lab coat, goggles
Strong reducing agent.

Health effects:
May cause irritation to eyes and skin, and respiratory tract.

First Aid measures:
Eye contact: rinse immediately with water, especially under eyelids, for >15 minutes.
Skin contact: wash off immediately with plenty of water and soap.
Inhalation: Move to fresh air. Get medical attention immediately if symptoms occur. If not breathing, give artificial respiration.
Ingestion: Rinse mouth with water.

Other hazards:
Fire: risk if exposed to oxidizing agents.
Explosion: risk if exposed to oxidizing agents.

7) Pre-lab questions

1. A primary standard is a solution in a titration for which the concentration is known. In this case, sodium thiosulfate cannot be used as a primary standard in the beginning because the concentration is unknown.
2. Common oxidizing agents used in acidic titrations include MnO_4^-, IO_3^-, and $Cr_2O_7^{2-}$, which undergo reduction according to the following equations.

$$\mathrm{MnO_4^- + 8H^+ + 5e^- \rightleftharpoons Mn^{2+} + 4H_2O}$$
$$\mathrm{IO_3^- + 6H^+ + 6e^- \rightleftharpoons I^- + 3H_2O}$$
$$\mathrm{Cr_2O_7^{2-} + 14H^+ + 6e^- \rightleftharpoons 2Cr^{3+} + 7H_2O}$$

3. Common reducing agents used in acidic titrations include Fe^{2+}, H_2S, and $S_2O_3^{2-}$, which undergo oxidation according to the following equations.

$$Fe^{2+} \rightleftharpoons Fe^{3+} + e^-$$
$$H_2S \rightleftharpoons S + 2H^+ + 2e^-$$
$$S_2O_3^{2-} \rightleftharpoons S_4O_6^{2-} + 2e^-$$

4. Iodometric titration refers to the titration of iodine produced by a chemical reaction, that is, molecular iodine being produced by a reaction and then being titrated by a standardized thiosulfate solution. Iodimetric titration refers to the titration of an analyte with iodine to produce the iodide ion.
5. To determine the end point of an acid-base titration, the pH level at the equivalence point of the titration curve should be used. Because the slope of the curve changes drastically at the equivalence point, an indicator that undergoes a color change in that region will be sudden and drastic, therefore suiting as a good indicator for when the titration has reached the equivalence point.
6. If two extra drops (0.1mL) was added to the buret by mistake, the titration would read less volume used than expected, by 0.1mL. The percentage error this creates would be based off the amount used to titrate the solution, which is based on the concentration. With 0.0025 mol of KIO_3, 0.0025*6 mol thiosulfate will be needed to titrate the solution, or 0.0150 mol. If x is the concentration of the thiosulfate, then the expected volume is 0.0150/X L. With the error, the measured volume will be 0.0150/X - 0.001 L. The error calculation is as follows:

$$Error = \frac{\frac{0.0150}{X} - 0.0001}{\frac{0.0150}{X}}$$
$$Error = \frac{0.0150}{X} * \frac{X}{0.0150} - 0.0001 * \frac{X}{0.0150}$$
$$Error = 1 - \frac{X}{150}$$

The calculated error in volume would 1 – X/150, where X is the molarity of the original thiosulfate solution.

7. Oxidation Equation

$$Fe^{2+} \rightleftharpoons Fe^{3+} + e^-$$

Reduction Equation

$$Ce^{4+} + e^- \rightleftharpoons Ce^{3+}$$

Combined Reaction

$$Fe^{2+} + Ce^{4+} \rightleftharpoons Fe^{3+} + Ce^{3+}$$

This results in a 1-1 stoichiometric ratio for the redox reaction.

Moles of Ce^{4+} used	0.01234M * 0.01339 L = 0.0001652 mol
Moles of Fe^{2+} used	0.0001652 mol
Moles of ferrous chloride	0.0001652/2 = 0.00008262 mol
Mass of ferrous chloride used	0.00008262 mol * 234.84 g = 0.01940 g
Percent mass of Cl in ferrous chloride	35.45*2 g/mol / 234.84 g/mol = 30.19%
Mass of Cl used	0.01940 g * 30.19% = 0.005857 g
Weight percentage of Cl used	0.005857g / 0.05485g = 10.68%