

**DETERMINATION OF
VITAMIN C
TEST EXERCISE
(105 POINTS)**

Last Update: 5/1/2009 10:25 AM

Objective:

To determine **VITAMIN C** content
of an *Unknown*

Concepts:

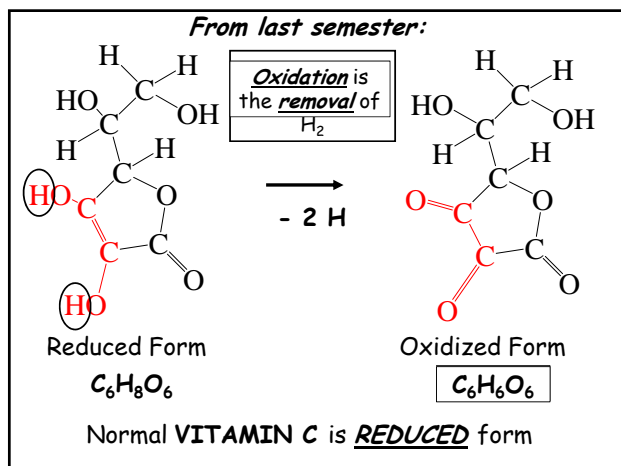
Oxidation/Reduction Iodometry (Optical Activity)

Techniques:

Titration Weighing

Apparatus:

Buret Balance

**PROPERTIES OF ASCORBIC ACID**

| | |
|-----------------------|---------------|
| Molar Mass | 176 mg / mmol |
| Solubility in H_2O | 300 mg / mL |
| pK_a 's: | 4.17, 11.57 |
| Analytical Wavelength | 245 nm (UV) |
| Reduction Potential | -0.127 V |

Since **VITAMIN C** is a reducing agent, we need an oxidizing agent, I_2 (*iodine*) to react with it.

I_2 is produced *in situ* by reacting IO_3^- (*iodate*) with excess I^- (*iodide*) that we add to the solution

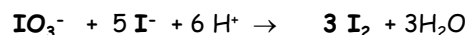
The underlying chemistry is subtle, but the procedure is simple

Redox Electrochemistry

| | Reduction Half Reaction | E^0 |
|-------------------------------------|---|---|
| <i>a</i> | $I_2 + 2e^- \rightarrow 2I^-$ | +0.536 |
| | | |
| (1/2)(<i>b</i> - 5 <i>a</i>) | | |
| | $IO_3^- + 6H^+ + 5I^- \rightarrow 3I_2 + 3H_2O$ | +1.195 - 0.536 $E^0 = +0.659$ |
| $I IO_3^- \Leftrightarrow 3 I_2$ | | |
| <i>a</i> - <i>c</i> | | |
| | $I_2 + C_6H_8O_6 \rightarrow 2I^- + C_6H_6O_6 + 2H^+$ | 0.536 - (-0.127) $E^0 = +0.663$ |
| $I I_2 \Leftrightarrow I C_6H_8O_6$ | | |

CHEMISTRY

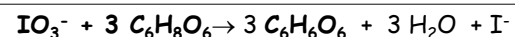
1.) IO_3^- (*iodate*) oxidizes I^- (*iodide*) to I_2 (*iodine*)



2.) Liberated I_2 (*iodine*), oxidizes $C_6H_8O_6$ (Ascorbic Acid)



3.) Overall reaction

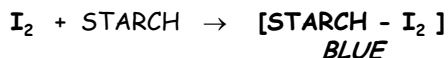


How will we know when reaction is complete?

4.) When **ASCORBIC ACID** has been consumed, the next drop of KIO_3 liberates I_2 which, has nothing to react with.

But, I_2 forms **blue** complex with **STARCH** indicator

Can detect very small amounts of I_2



The technique described above is general for titrating reducing agents, and called **Iodometry**.

1. Add excess **KI** to acidified reducing agent
2. Titrate with KIO_3 to a **Starch-iodine** end point

PROCEDURE

1. Determine **titer** of potassium iodate standard solution with **pure** ascorbic acid.

2. Determine amount of unknown to be used in titration (trial run)

3. Determine amount of **ASCORBIC ACID** in unknown by titration with **potassium iodate** (KIO_3) using a **STARCH INDICATOR**

**1. TITER DETERMINATION
(DO 3 RUNS)**

Weigh out ~0.1 g samples of pure **ASCORBIC ACID**, **ACCURATELY** (100 mg / 176 ~ 0.6 mMOL)

Dissolve sample in **150 mL** of distilled water

Add:

5 mL of 1.0 M HCl = 5 mMOL
10 mL of 0.6 M KI = 6 mMOL

8-10 drops of Starch Indicator

Titrate with KIO_3 to **Blue** end point

Do not add KIO_3 too rapidly. I_2 is not very soluble.

**SAMPLE DATA SHEET:
DETERMINATION OF TITER:
(STANDARDIZATION)**

| | |
|-----------------------|----------|
| Mass of Ascorbic Acid | 121.6 mg |
| Final Buret Reading | 24.96 mL |
| Initial Buret Reading | 2.35 mL |
| Volume of Iodate | 22.61 mL |

Titer Value of KIO_3 121.6 / 22.61 = 5.378 mg/mL

WARNING!

This exercise involves both **Potassium Iodate**,

&

Potassium Iodide

Be sure you read the reagent bottle labels carefully!

**DETERMINATION OF ASCORBIC ACID
CONTENT OF UNKNOWN SAMPLE**

Unknown samples are **SOLIDS**.

Do **trial run** to insure that you use an appropriate volume of KIO_3

I.e., weigh ~150 mg of unknown precisely
Titrate

Use result to adjust weight of next samples
(Wish to use at least 20 ml of Potassium Iodate)

Report 3 RUNS & AVG DEVIATION

Trial Run

| | | |
|-----------------|-----------------|-----------|
| Mass of Unknown | 143.5 mg | 25 |
| Final Buret | 13.83 mL | |
| Initial Buret | <u>3.53 mL</u> | |
| Net Volume | 10.30 mL | |

143.5 mg of unknown require **10.30 mL** of KIO_3

How many mg will require **25 mL**?

| | | |
|-----------------|---|--------------|
| 143.5 mg | | X mg |
| ----- | = | ----- |
| 10.30 mL | | 25 mL |

| | <u>Run 1</u> | <u>Run 2</u> | <u>Run 3</u> |
|--------------------|--------------|--------------|--------------|
| Mass of Unknown | 351.1 mg | 374.2 mg | 335.9 mg |
| Final Buret | 28.83 mL | 30.93 mL | 26.95 mL |
| Initial Buret | 3.72 mL | 4.53 mL | 3.27 mL |
| Net Volume | 25.11 mL | 26.40 mL | 23.68 mL |
| Vit C in sample | 135.0 mg | 141.9 mg | 127.3 mg |
| % Vit C in Unknown | 38.48 % | 37.93 % | 37.90 % |
| Average Percent | | 38.10 % | |
| Average Deviation | | 0.25 % | |
| Percent Deviation | | 0.66 % | |

We will not analyze a Vitamin C pill or other product containing Vitamin C

Suggestions

1. Be sure container in which you obtain KIO_3 is clean and dry!
2. Buret is rinsed - no bubbles!
3. Use same balance for all weighings!
TA's have checked balances to make sure they are calibrated
4. Weigh by difference!
Leave vials of ascorbic acid near the balances.
5. Stir or swirl solutions well when doing titrations!

Consider using a "standard penny"

NEXT (LAST) LECTURE**Electrochemistry and the Nernst Equation**

READ **SUSB-056**

DO PRE-LAB