

College of Pharmacy-University of Mustansiriyah

5Th. Year- Practical advance pharmaceutical analysis – 2019 -

Experiment-3

Titration of the ascorbic acid (vitamin C) in tablets By pH meter used first and 2nd derivatives

Outcomes:

After completing this experiment, the student should be able to:

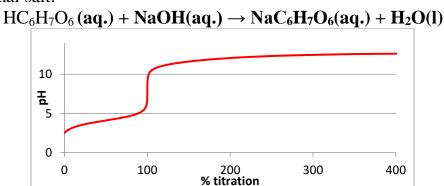
- o Calibration of pH meter.
- o Calculation of first and 2nd derivatives.
- o Calculate the K_a of weak acid.

Introduction:

In this experiment students analyze a tablet of Vitamin C (or other drug containing this compound) and calculate the percentage of ascorbic acid, $H_2C_6H_6O_6$, present in it.

Vitamin C (ascorbic acid)

Vitamin C tablets contain ascorbic acid as the active ingredient; however, it is mixed with such fillers as starch which however does not obscure the endpoint. Ascorbic acid has two steps of hydrolysis (hydrogen ion), (pK $_{a1}$ =4.10, pK $_{a2}$ =11.8). Since the second one comes off only at a pH of 10 or 11 the reaction of ascorbic acid with sodium hydroxide will produce the acid salt, sodium hydrogen ascorbate, and not the normal salt:-



Titration curve of 0.1 M ascorbic acid using 0.1 M NaOH as titrant When titrated using an indicator like Phenolphthalein or Bromothymol Blue; The NaOH solution will be **standardized** to determine its strength by reacting it with a very pure (**primary standard**) sample of an acid.

1 Ali Albakaa



College of Pharmacy-University of Mustansiriyah 5Th. Year- Practical advance pharmaceutical analysis – 2019 -

Materials and Equipment:- pH meter, buratte, pipette, beker, Vitamin C tablet, Sodium hydroxide, Indicator.

Procedure:

Place a tablet of Vitamin C in an Erlenmeyer flask, add about 50 mL of warm distilled water, and crush the tablet with a glass stirring rod. Cool down the solution. Add two or three drops of indicator to the flask and titrate with sodium hydroxide solution until the endpoint is reached. Make two independent titrations at least.

Result calculation:

As seen in the titration curve, vitamin C behaves as mono-protic acid when titrated using Phenolphthalein as indicator. This means that the number of moles of NaOH in titration is equal to the number of moles of the ascorbic acid.

Calculate the mass of acid for two titrations and, finally, calculate the arithmetic average of these two results. Compare the result with the factory value.

Note: that since this is a quantitative lab. exercise all measurements to be used in calculations must be recorded to the proper number of significant digits.

Molecular mass of ascorbic acid is 176.13 g/mol.

Record all the numbers obtained and the name of indicator used, as well as the calculations made.

Procedure (pH-metric titration):

- 1. Place a tablet of Vitamin C in an Erlenmeyer flask, add about 50 mL of warm distilled water, and crush the tablet with a glass stirring rod. Cool down the solution.
- **2.** Dilute the sample in your volumetric flask to the total volume of 100-150 mL with distilled water.
- **3.** Place the beaker on magnetic stirrer insert the magnet in it, as well as pH-electrode. Ask the assistant to control the correctness of the installation and some advices.
- **4.** Measure and note pH; Repeat this adding small portions of the titrant base (few drops each), noting also the actual volume of titrant added (total). Finish titration when pH exceeds 12.

Processing the results

Using a computer data-sheet, plot the titration curve obtained and determine as exactly as possible the volume of titrant corresponding to the inflection point.

For more aspiring students:

The preciseness of determination of the inflection point can be very improved if calculating the second derivative of the titration curve.

2 Ali Albakaa



College of Pharmacy-University of Mustansiriyah

5Th. Year- Practical advance pharmaceutical analysis – 2019 -

Record all the numbers obtained, as well as the calculations made. Note your observation concerning the comparison of your result with the factory value.

Excel Shortcut Keystrokes for the PC*

*Macintosh equivalents, if different, appear in square brackets

TO ACCOMPLISH THIS TASK

Alternate between displaying cell values and displaying cell

Calculate all sheets in all open workbooks

Calculate the active worksheet

Cancel an entry in a cell or formula bar

Complete a cell entry and move down in the selection Complete a cell entry and move to the left in the selection

Complete a cell entry and move to the left in the selection

Complete a cell entry and move to the right in the selection

Complete a cell entry and move up in the selection

Copy a formula from the cell above the active cell into the cell

or the formula bar

Copy a selection Copy the value from the cell above the active cell into the cell

or the formula bar

Cut a selection Define a name

Delete the character to the left of the insertion point,

or delete the selection

Delete the character to the right of the insertion point,

or delete the selection

Displays the Insert Function dialog box Displays Key Tips for ribbon shortcuts

Edit a cell comment Edit the active cell

Edit the active cell and then clear it, or delete the preceding character in the active cell as you edit the cell contents

Enter a formula as an array formula

Fill down

Fill the selected cell range with the current entry

Fill to the right Format cells dialog box Insert the AutoSum formula

Move one character up, down, left, or right

Move to the beginning of the line Paste a name into a formula

Paste a selection Repeat the last action Selects the entire worksheet

Start a formula

Start a new line in the same cell

Undo

TYPE THESE KEYSTROKES

Ctrl+`[黑+`]

F9

Shift+F9

Esc

Enter [Return]

Shift+Tab

Tab

Shift+Enter

Ctrl+' (Apostrophe) [第+']

Ctrl+C[\mathcal{H}+C]

Ctrl+Shift+" (Quotation Mark) [#+Shift+"]

Ctrl+X [#+X] Ctrl+F3 [#+F3] Backspace [Delete]

Delete [Del]

Shift+F3 ALT Shift+F2 F2 [None]

Backspace [Delete]

Ctrl+Shift+Enter Ctrl+D[\mathfrak{H}+D] Ctrl+Enter [None] Ctrl+R [\mathfrak{H}+R] Ctrl+1 [\mathfrak{H}+1]

Alt+= (Equal Sign) [第+Shift+T]

Arrow Keys Home F3 [None] Ctrl+V [#+V] F4 Or Ctrl+Y [#+Y]

Ctrl+A = (Equal Sign)

Alt+Enter [#+Option+Enter]

Ctrl+Z[#+Z]

3 Ali Albakaa