

College of Pharmacy-University of Mustansiriyah

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

EXPERIMENT 5 Determination of Citric acid by pH meter

Outcomes:

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

- Calibration of pH meter.
- Calculation of first and 2^{nd} derivatives.
- \circ Calculate the K_a of weak acid.

Introduction:

In this experiment students analyze a tablet of Citric acid (or other drug containing this compound) and calculate the percentage of Citric acid, $C_6H_8O_7.H_2O$, 210.14g.mol⁻¹ present in it, Composition: C (37.51%) H (4.20%) O (58.29%).



Citric acid is widely used as an acidulant in creams, gels, and liquids of all kinds. In its use in foods and dietary supplements, it may be classified as a processing aid if the purpose it was added was for a technical or functional effect (e.g. acidulent, chelator, viscosifier, etc...) for a process. If it is still present in insignificant amounts, and the technical or functional effect is no longer present. Citric acid is an alpha hydroxy acid and used as an active ingredient in chemical peels. Citric acid is commonly used as a buffer to increase the solubility of pharmaceutical. Citric acid is used as one of the active ingredients in the production of antiviral tissues. The combination of citric acid and sodium citrate is used to prevent gout or kidney stones, or metabolic acidosis in people with kidney problems. Citric acid and sodium citrate may also be used for other purposes.

$$3 \text{ NaOH} + \text{H}_3\text{C}_6\text{H}_5\text{O}_7 \rightarrow 3 \text{ Na}^+ + 3 \text{H}_2\text{O} + \text{C}_6\text{H}_5\text{O}_7^{3-1}$$

Materials and Equipment:-

pH meter, buratte, pipette, beker, Sodium hydroxide, Indicator, Citric Acid.



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Procedure:

Place a sample of Citric acid monohydrate in an Erlenmeyer flask, add about 25mL of distilled water. 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, Citric Acid behaves as Tri-protic acid when titrated using Phenolphthalein as indicator. This means that the number of 3 moles of NaOH in titration is equal to one of mole of the Citric 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.

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

Procedure (pH-metric titration):

- **1.** Place a sample of Citric acid monohydrate in a beaker, add about 25mL of distilled water.
- 2. Dilute the sample 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.

Record all the numbers obtained, as well as the calculations made.

Note your observation concerning the comparison of your result with the factory value.