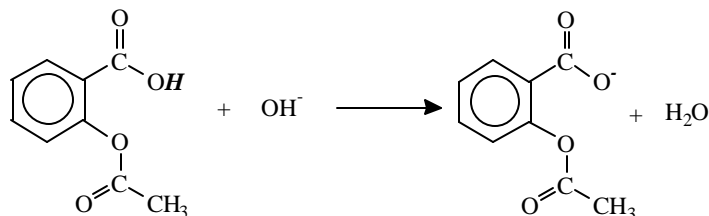


VOLUMETRIC DETERMINATION OF ACETYLSALICYLIC ACID IN ASPIRIN

1. Summary of Method

In this analysis, the active ingredient in aspirin, acetylsalicylic acid, will be measured by titrating the acid with a standardized solution of sodium hydroxide. Acetylsalicylic acid is a weak organic acid that reacts with hydroxide ion as shown below.



The sodium hydroxide solution is standardized using potassium hydrogen phthalate [KHP] and phenolphthalein is used as the indicator in all of the titrations.

During one of the titrations, the pH of the aspirin solution will be monitored with a pH electrode. This information will be used to construct a titration curve and determine the acid dissociation constant for acetylsalicylic acid.

2. Comments

Hazards. Concentrated sodium hydroxide [19M] is extremely caustic and should be handled with the utmost care and exclusively in the hood. Be sure that the NaOH is clear [no suspended Na₂CO₃] and be careful when diluting the NaOH since heat will be evolved.

Interferences. Acetylsalicylic acid is a weak organic acid that is not extremely soluble in water. Because of this ethanol is added to the titration solution to increase solubility. Due to the possible presence of acid in the ethanol [caused by oxidation of the ethanol] it is advisable to perform a blank titration on an ethanol sample to avoid interference. The binder in the aspirin tablet will not dissolve, but it does not interfere in the titration.

3. Sample Handling and Preparation

Aspirin tablets will be analyzed in two-tablet samples. Do not oven dry the tablets. Weigh out the two-tablet samples on the analytical balance.

4. Apparatus

- One calibrated 50mL buret
- Stir motor
- pH meter

5. Reagents

- Commercial aspirin tablets. Be sure to record the brand of aspirin tablets analyzed. Do not dry.
- Primary standard potassium hydrogen phthalate. Dry this in a 100 C oven for two hours and cool in a dessicator.
- 0.1 M NaOH. Prepare 900 mL of this solution by adding 4.7 mL of 19 M NaOH (caution! caustic!!) to a

1L bottle and diluting to 900 mL with boiled and cooled deionized water.

d. 95% ethanol.

e. pH 4.00 and pH 7.00 buffers. Commercial buffers will be available in the lab for calibration of the pH meters.

6. Procedure

a. Standardize the NaOH solution by titrating a 0.7-g sample of dry KHP [weighed on the analytical balance], which is dissolved in 60 mL of deionized water. Use two drops of phenolphthalein indicator and titrate to the first pink color. Adjust the mass of KHP titrated in subsequent titrations so about 40 mL of NaOH solution is used to reach the endpoint. Continue to titrate KHP samples until the three closest runs produce an average NaOH concentration with less than 0.5% relative standard deviation.

b. Prepare a blank sample by adding 25 mL of ethanol and 75 mL of deionized water along with two drops of phenolphthalein to a 250 mL erlenmeyer flask. Titrate to a pink color with standardized NaOH. Subtract the volume of the blank titration from all subsequent aspirin titrations.

c. Titrate three, pre-weighed, two-tablet aspirin samples with NaOH. For the first two samples, dissolve the tablets in 25 mL of ethanol in a 250-mL erlenmeyer flask crushing them with a stirring rod if necessary. Add 75 mL of deionized water and two drops of phenolphthalein, and titrate to a pink color.

d. For the third two-tablet sample the pH will be measured throughout the titration. Obtain a stirring motor and a pH meter. Calibrate the pH meter [consult your instructor]. Dissolve the tablets in a 250 mL beaker as before and place the stirring bar in the beaker. Put the pH electrode[s] in the beaker and start the stirring motor being sure the bar does not hit the electrode[s]. Titrate the sample by adding the NaOH one mL at a time and recording the pH 30 seconds after addition. Add smaller volumes of NaOH near the end point, i.e. when the pH begins to increase rapidly. Titrate through the endpoint and about 2 mL past it recording the pH and volume after each addition.

7. Bibliography

- a. Daniel C. Harris, "Quantitative Chemical Analysis," 2nd edition, W.H. Freeman, New York, 1987. See Chapter 12 for a discussion of acid base titrations.

8. Calculations and write-up Instructions

- a. Report the brand of aspirin analyzed.
- b. Report the average and standard deviation for the wt/wt% acetylsalicylic acid in the aspirin tablets as determined by the three two-tablet titrations.
- c. Plot pH vs. volume for the third titration.
- d. Determine the pK_a using the endpoint from step d and the plot in step c.