## RET Lab Lesson Plan <br> Topic: Determination of aspirin in tablets using back titration

Aim: To calculate and compare the active pharmaceutical ingredient (API) in different commercially available aspirin tablets of the same batch using titration technique.

STANDARD(S) \& INDICATOR(S):
5.1.8.B1. Identify questions and make predictions that can be addressed by conducting investigations.
5.1.8.B3. Collect, organize, and interpret the data that result from experiments.
5.6.6.B1. Recognize evidence of a chemical change.

OBJECTIVE(S): Students will be able to:

1. Students will be able to calculate the amount of the active pharmaceutical ingredient(API) in aspirin tablets by performing acid-base titration
2. Students will be able to compare the amounts of aspirin in different tablets of the same batch.
3. Students will be able to evaluate the mixing efficiency of components in a tablet by analyzing the results

## Materials Needed

Aspirin tablets, 0.1 M Hydrochloric acid, 0.1 M Sodium hydroxide solution, Ethanol, Phenolphthalein indicator, Burettes, Mortar \& Pestle, Balance, Water bath.

## Theory:

The key concepts from the research work are the following:

1. Importance of particle size in industrial applications
2. Importance of proper mixing of substances in industry.

Hypothesis: All tablets contain the same amount of API mentioned in the label described by the manufacturer.

Many reactions are slow or present unfavorable equilibria for direct titration. Aspirin is a weak acid that also undergoes slow hydrolysis; i.e., each aspirin molecule reacts with two hydroxide ions. To overcome this problem, a known excess amount of base is added to the sample solution and an HCl titration is carried out to determine the amount of unreacted base. This is subtracted from the initial amount of base to find the amount of base that actually reacted with the aspirin and hence the quantity of aspirin in the analyte.



## Safety Issues and Chemical Hazard Information

## Physical Hazards

Aspirin
Ethanol
Hydrochloric acid Phenolphthalein
Sodium hydroxide
none
flammable
water-reactive, corrosive none water-reactive, corrosive

## Health Hazards

toxic, irritant, sensitizer
irritant toxic irritant toxic, irritant

Concentrated hydrochloric acid is highly corrosive. Be careful. Ethanol is flammable. Keep it away from heat.

## Procedure:

## Sample preparation

1. Accurately record the weight of a group of three aspirin tablets so that you can determine an average tablet weight. Use a mortar and pestle to crush enough tablets to produce approximately 1 g tablet powder.
2. Weigh approximately 300 mg aspirin powder, into labeled 250 mL Erlenmeyer flasks.
3. To each flask, add 20 mL of ethanol (measure by graduated cylinder) and three drops of phenolphthalein indicator. Swirl gently to dissolve. (Aspirin is not very soluble in water - the ethanol helps the aspirin dissolve. Note that an aspirin tablet contains other compounds in addition to aspirin. Some of these are not very soluble. Your solution will be cloudy due to insoluble components of the tablet.)

## Aspirin Titration with Base

4. Titrate the first aspirin sample with NaOH to the first permanent cloudy pink color.
5. The aspirin/ NaOH acid-base reaction consumes one mole of hydroxide per mole of aspirin. The slow aspirin/ NaOH hydrolysis reaction also consumes one mole of hydroxide per mole of aspirin, and so for a complete titration we will need to use a total of twice the amount of NaOH that you have already used, plus we will add some excess NaOH to ensure we really have reacted with all of the aspirin in the sample.
(For example: if you used 26 mL of base in the previous step, the
volume of base you would add now would be $26+10=36 \mathrm{~mL}$. Thus, you would have added a total of $26+26+10=62 \mathrm{~mL}$ of base.)

## Heating for Completion of Reaction

6. Heat gently the flask contents in a water bath. Avoid boiling, because the sample may decompose. While heating, swirl the flasks occasionally. After 15 minutes, remove samples from the water bath and cool for 5 minutes.
7. If the solution is colorless, add a few more drops of phenolphthalein. If it remains colorless, add 10 mL more of the base and reheat. (Don't forget to add this additional volume of base to the previously recorded total volume.)
8. The only base remaining in each flask will be excess base that has not reacted with the aspirin. Using your burette with your $\sim 0.1 \mathrm{M} \mathrm{HCl}$ solution, titrate the excess base in each flask with HCl until the pink color just disappears. The endpoint is best described as "cloudy white".
9. Record all the volumes of bases and acid in the data table.

## Data \& Calculations

Average mass of an aspirin tablet $=$
Mass of aspirin tablet powder used in the experiment $=$
Volume of the base $(\mathrm{NaOH})$ used in the first titration $(\mathrm{V} 1)=$ Volume of the extra base $(\mathrm{NaOH})$ added in the flask $(\mathrm{V} 2)=$ Total volume of the base $(\mathrm{NaOH})$ used in the reaction $(\mathrm{V} 3)=$
Volume of the acid $(\mathrm{HCl})$ reacted with the mixture in the second titration $(\mathrm{V} 4)=$
Actual volume of the base $(\mathrm{NaOH})$ reacted $(\mathrm{V} 5)=\mathrm{V} 3-\mathrm{V} 4$
Number of moles of NaOH reacted $=$ Volume of the base in liters x Molarity of the base
Number of moles of aspirin reacted $=\frac{1 \text { mole aspirin }}{2 \text { moles of } \mathrm{NaOH}} \mathrm{x}$ moles of NaOH reacted
Mass of aspirin reacted $=\frac{180.2 \mathrm{~g}}{1 \text { mole aspirin }} \mathrm{x}$ moles of aspirin

## Pre-Lab questions:

Define the following terms:
Acids, bases, titration, endpoint, indicator, molarity
Post-Lab questions (Show all calculations where ever necessary)

1. Write a balanced chemical equation for the reaction between hydrochloric acid and sodium hydroxide
2. Write the skeletal and balanced chemical equation for the reaction between aspirin and sodium hydroxide.
3. What is the actual volume of sodium hydroxide used to neutralize aspirin in the experiment?
4. How many grams of sodium hydroxide reacted in the experiment?
5. How many grams of aspirin (API) present in each tablet?
6. What is the percentage of aspirin in each tablet?
7. Compare your results with different groups. Explain your finding.
8. Based on your findings from different groups, what is your conclusion about mixing efficiency of different components in a tablet?

## Assessment

Pre lab questions -
Calculations \& Data Analysis -
Post lab questions based on the experimental reactions and calculations -
Lab report containing discussion \& Conclusion -
Presentation/ Impression -

3 points
5 points
5 points
10 points
2 points

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