CHAPTER-10

Experiment: 10

TO PERFORM ASSAY OF ASPIRIN (ACID-BASE TITRATION) AND STANDARDIZATION OF TITRANT

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Aim:

To perform assay of aspirin (Acid-base titration) and standardization of titrant.

Requirements:

A. Glassware & Instruments:

1. Two Burettes
2. Weighing bottle
3. Water bath
4. Boiling chips
5. Mortar and Pestle

B. Chemicals & Reagents:

1. Aspirin tablets
2. Ethanol
3. Phenolphthalein indicator solution
4. Hydrochloric acid, conc., 37 wt. %
5. Sodium hydroxide, standardized solution

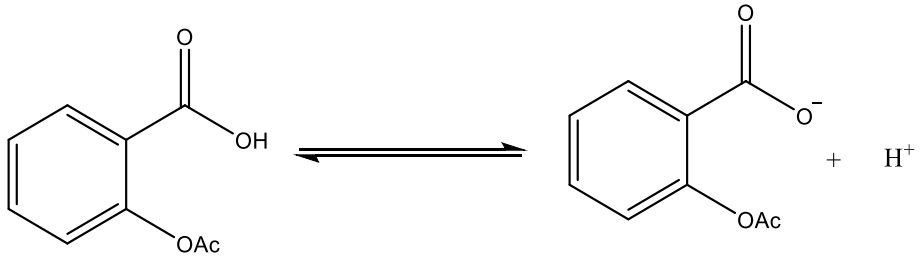
Theory:

Aspirin is a weak acid that also goes through a slow process called hydrolysis. This means that each molecule of aspirin reacts with two ions of hydroxide. To get around this, a known amount of extra base is added to the sample solution, and an HCl titration is done to find out how much base hasn't reacted. This is subtracted from the initial amount of base to find the amount of base that actually reacted with the aspirin and, therefore, the amount of aspirin in the analyte. ASA is easy to dissolve in ethanol. Then, a solution of NaOH was added, and the solution was left to sit for 30 minutes with the lid on. The extra amount of NaOH is titrated with HCl solution using pH as a guide. The same titration was done again, but this time ASA was left out of the solution to act as a blank.

Aspirin is made of acetyl salicylic acid. It comes in the form of white crystals and is used as a pain reliever, a fever reducer, and an anti-inflammatory. Its molecular formula ($C_9H_8O_4$).

It is a monoprotic weak acid, ka = 2.8 x 10^4 at 25 °C, so very little of the molecular aspirin (acetylsalicylic acid) dissociates to form acetylsalicylate ions.

For the equilibrium dissociation reaction:



Aspirin (Acetylsalicylic acid) Acetylsalicylate ion

It dissolves slightly in water (1:300), and it dissolves easily in alcohol (1:5), chloroform (1:17), and ether (1:15). Its melting point is 135 °C, and its molecular weight is 180.2 g/mole. With heat and moisture, it slowly decomposed into acetic acid and salicylic acid.

Salicylates are easily taken in by the stomach and small intestine because they are acidic. Their absorption is strongly affected by the pH of the environment. Because of this, an antacid or other buffering agent should not be given at the same time because it makes their absorption much harder. Aspirin's main metabolite is salicylic acid. It goes through a lot of phase-II metabolism and is passed out of the body through the kidneys as a water-soluble glycine conjugate or acyl glucuronides.

Principle:

In this aspirin titration, we use standardised NaOH and phenolphthalein as a colour change indicator. The aspirin goes from being colourless to a cloudy pink colour. We also use ethanol because aspirin tablets have other chemicals in them that might not dissolve in water. This is an acid-base titration to measure how much aspirin is in the solution.

A. **Direct titration method:** After dissolving ASA in ethanol, it can be measured using NaOH as a titrant solution and phenolphthalein (phph) as an indicator.

B. **Back titration method:** is one of the volumetric methods that involves the addition of an excess of standard volumetric sol. to a weight amount of a sample, followed by the determination of the excess unreacted (not required or utilised by

the sample), and then the determination of the amount of volumetric solution utilised by the substance.

Procedure:

Preparation of Approximate Acid Solution (0.1M HCl):

- Pour 100 mL of water distillate into the other big bottle.
- Calculate the amount of concentrated HCl required to make 250 mL of 0.1M HCl prior to conducting this experiment. (Reagent-grade concentrated HCl has a density of 1.188 g mL–1 and a concentration of 37 weight percent.
- Using a graduated cylinder, measure roughly this amount of concentrated HCl.
- Gradually add the acid to the water in the bottle while stirring thoroughly (remember, always add acid to water, not water to acid.)
- Add more distilled water, thoroughly mixing after each addition, until the total volume of the solution reaches 250 ml.
- You are not required to measure the quantities precisely because you will standardise this solution to ascertain its real concentration in the subsequent phases.

Comparison of HCl to your standardized NaOH

- After cleaning, rinsing, and filling the burette with the 0.1 M HCl solution, proceed to the next step.
- Pour 50 mL of distilled water into each of the clean, labelled Erlenmeyer flasks. • Add three drops of phenolphthalein indicator to each flask.
- Using your HCl burette, add roughly 35, 40, and 45 mL of acid to flasks #1, noting the precise amount of acid added to each flask to the nearest 0.01 mL.
- Gently swirl to combine.
- Cleanse, rinse, and fill the second burette with the 0.1 M NaOH solution that you
- Standardised in the first burette.
- Titrate the HCl flasks with NaOH to an endpoint of phenolphthalein.

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Sample preparation:

- Accurately note the weight of three aspirin tablets in order to calculate the average weight of the tablets.
- Using a mortar and pestle, crush sufficient tablets to yield 1 g of tablet powder.
- Using a clean, dry weighing vial, correctly weigh 0.3 g tablet samples by difference into labelled 250 mL Erlenmeyer flasks..
- To the flask, add 20 mL of ethanol (measure by graduated cylinder)
- Add three drops of phenolphthalein indicator.
- Swirl gently to dissolve

(Aspirin is not particularly soluble in water; ethanol aids in its dissolution. Note that an aspirin tablet contains several substances besides aspirin. Several of these are not particularly soluble. Your solution will be murky due to the tablet's insoluble components.)

Aspirin Titration with base:

- (If instructed to execute this step prior to Steps 1-3, clean, rinse, and fill a burette with the specified 0.1 M NaOH solution before continuing.) Titrate the first aspirin sample with NaOH until the first hazy pink colour is achieved.
- One mole of hydroxide is used for each mole of aspirin in the acid-base interaction between aspirin and sodium hydroxide. The slow aspirin/NaOH hydrolysis reaction consumes one mole of hydroxide per mole of aspirin; therefore, for a complete titration we will need to use twice the amount of NaOH that you have already used, plus we will add excess NaOH to ensure that we have truly reacted with all of the aspirin in your sample (adding excess reactant drives the equilibrium towards products — Le Chatelier's principle).

Heating the reaction to completion:

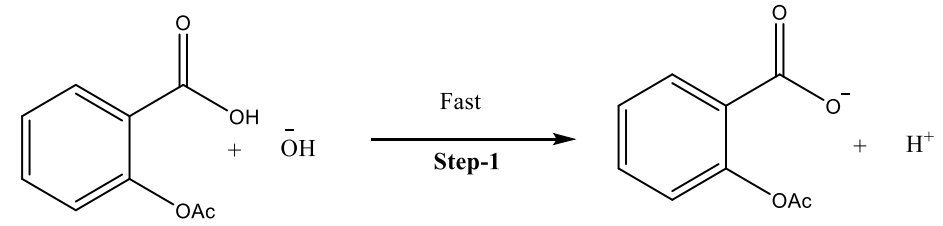
- To accelerate the hydrolysis reaction, add two or three boiling chips to each flask and place in a water bath.
- Avoid boiling the sample, since it may degrade.
- While heating, occasionally swirl the flasks.

- Remove samples from the water bath after 15 minutes and allow them to cool for 5 minutes.
- Add a few more drops of phenolphthalein if the solution is colourless.
- If the liquid stays colourless, add 10 mL more base and reheat.

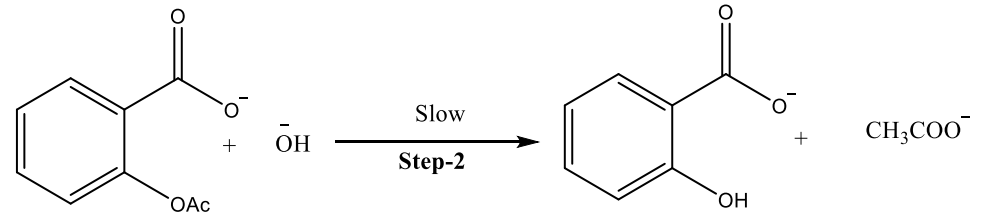
Back titration with acid:

- Only excess base that has not reacted with the aspirin will remain in each flask.
- Using a burette filled with a 0.1 M HCl solution, titrate the surplus base in each flask with HCl until the pink colour diminishes slightly.
- The best description of the terminus is "cloudy white".

Reaction:



Aspirin (Acetylsalicylic acid) Acetylsalicylate ion



Acetylsalicylate ion Salicylate ion

Calculation:

Observation table

S. no.	Content of the flask	Initial reading	Final reading	Difference	Indicator

1	0.3g of aspirin+0.1ml of Phenolphthalein+20ml Ethanol				Phenolphthalein
2	0.3g of aspirin+0.1ml of Phenolphthalein+20ml Ethanol				Phenolphthalein

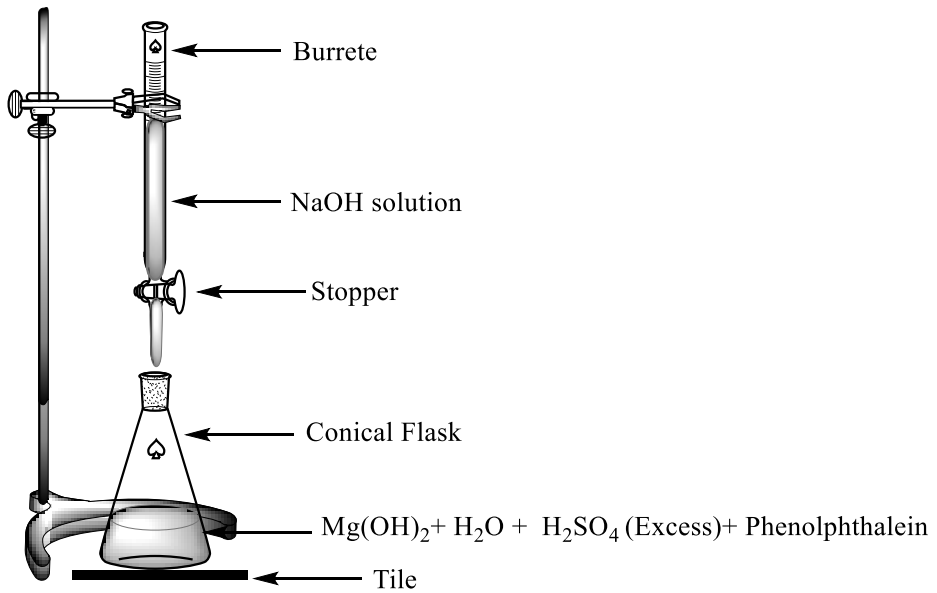
0.3g of Aspirin gets dissolved in 20 ml of Ethanol it takes 22 ml to dissolve in 0.1N NaOH in 0.3g of Aspirin In moles=0.001645 mol

Mass of Aspirin=180X0.001645=0.2961g

% Purity of aspirin = Titre value x Equivalent wt. factor x Normality of NaOH (actual)/Weigh of sample x Normality of titrant (expected) x100

% Purity of aspirin = ……..%

Diagram:



Applications:

- Aspirin is used to lower fever and alleviate mild to moderate discomfort, such as that caused by muscle pains, toothaches, the common cold, and headaches.
- Additionally, it may be used to relieve pain and inflammation in illnesses such as arthritis. Aspirin is a salicylate and nonsteroidal anti-inflammatory medication (NSAID).

1. Helps in reducing fever, moderate pain.
2. Its a non steroidal anti inflammatory drug.
3. It also helps in lowering the risk of heart attacks.

Result:

The percentage purity of the given sample of Aspirin is ……

Viva questions:

- What is the chemical formula of aspirin?
- Which type of titration use for assay of aspirin?
- What type of indicator use in aspirin assay?
- Why NaOH used in this assay?
- Why $Mg(OH)_2$ acid is used during titration?
- What is the use of aspirin?
- Why sulfuric acid is used during titration?

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