

Quantitative Assay of Aspirin Tablets

Back Titration

There are two types of analytical methods :

(1) Qualitative Analysis: -

It determines the presence or absence of a particular compound, but not the mass or concentration. By definition , qualitative analysis do not measure quantity.

(2) Quantitative Analysis: -

It determines how much of each component , or of specified components is present in a given sample.

Methods of Quantitative Chemical Analysis :

1-Volumetric (Titrimetric) analysis.

2-Gravimetric analysis.

3-Spectrophotometric analysis.

Requirements For a Titrimetric Assay:

- (1) The reaction can be represented by a chemical equation.**
- (2) The reaction should be relatively fast.**
- (3) The reaction should be complete & irreversible.**
- (4) The end point should be easily detected.**

Types of Titration:

- 1- Forward titration (*direct titration*).**
- 2- Back titration (*indirect titration*).**

Back Titration:

It includes the addition of an excess of a std. solution to a weighted amount of a sample and then the excess unreacted std. solution is determined by titration with another std. solution.

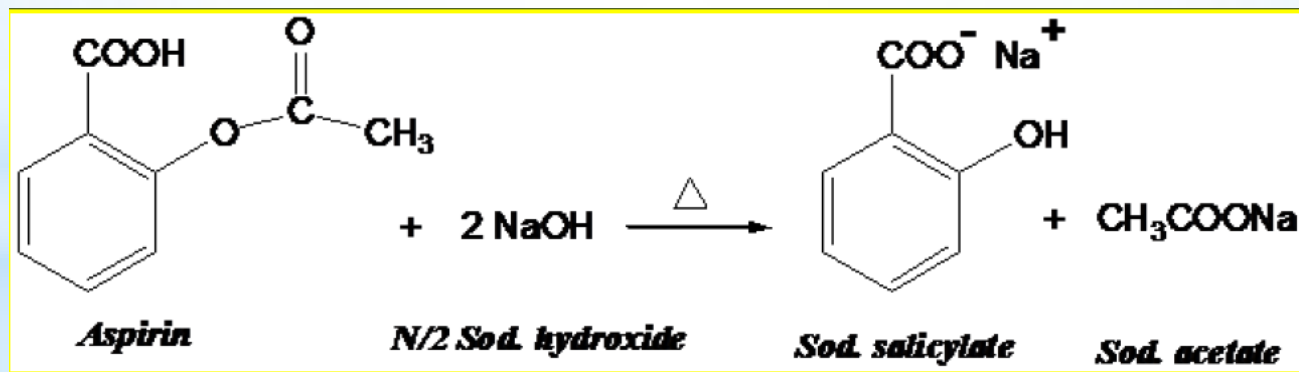
Back Titration Is Used For:

- 1- Volatile substances, e.g., NH_3 .
- 2- Insoluble or slightly soluble substances, e.g. CaCO_3
- 3- Substances for which the quantitative reaction proceeds rapidly only in the presence of excess of reagent, e.g., Lactic acid & Aspirin.
- 4- Substances which decompose on heating, e.g. , Formaldehyde.

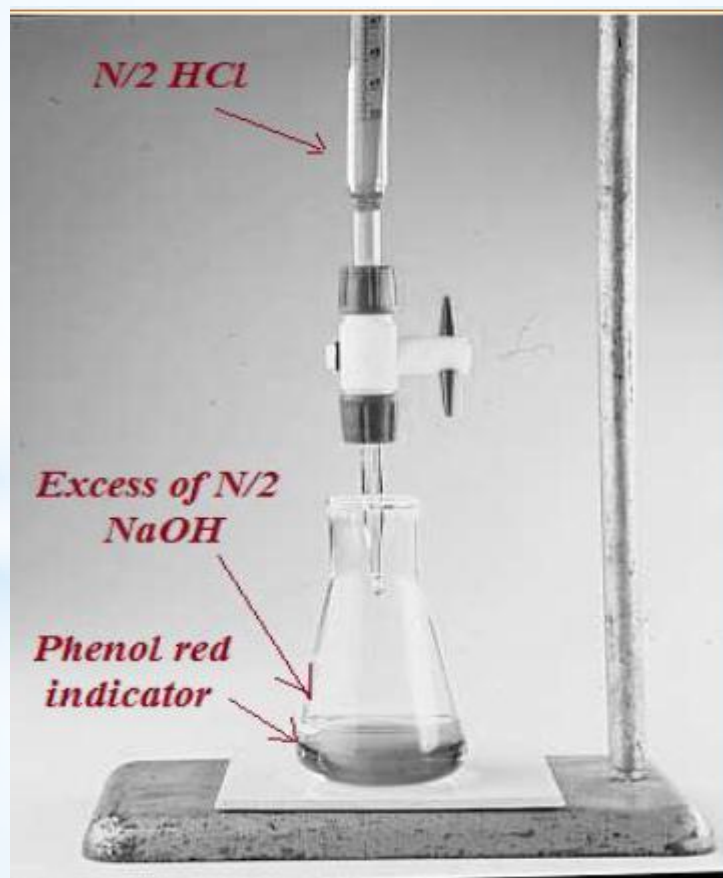
Assay Of Aspirin

Principle:

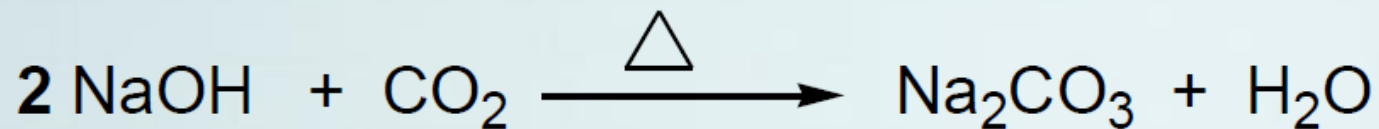
The determination of the amount of aspirin present in a tablet dosage form is done by alkaline hydrolysis of aspirin using $N/2$ NaOH standard solution followed by back titrating of the excess unreacted alkali using $N/2$ HCl std. solution & phenol red as indicator.



- Aspirin readily dissolved in dilute NaOH solution and hydrolyzed completely by heating for 10 minutes with an excess of a base.
- Titration of the excess unreacted alkali with N/2 HCl std. solution using phenol red indicator



- As in other quantitative determination involving boiling with a standard alkali , cooling and back titrating the excess, it's necessary to carry out a blank experiment without the aspirin .
- *In order to:*
 - 1- Minimize any error due to small unavoidable losses.
 - 2- Heating and cooling an alkaline liquid results in an apparent change in strength if certain indicators are used .
- This change may be due to the interaction of the reagent with the glass or due to , the absorption of atmospheric CO₂ ,
- CO₂ is rapidly absorbed by the hot alkaline solution to form sodium carbonate .



- In the back titration with the standard acid the liberated CO₂ causes a color change of the indicator before the actual end point.



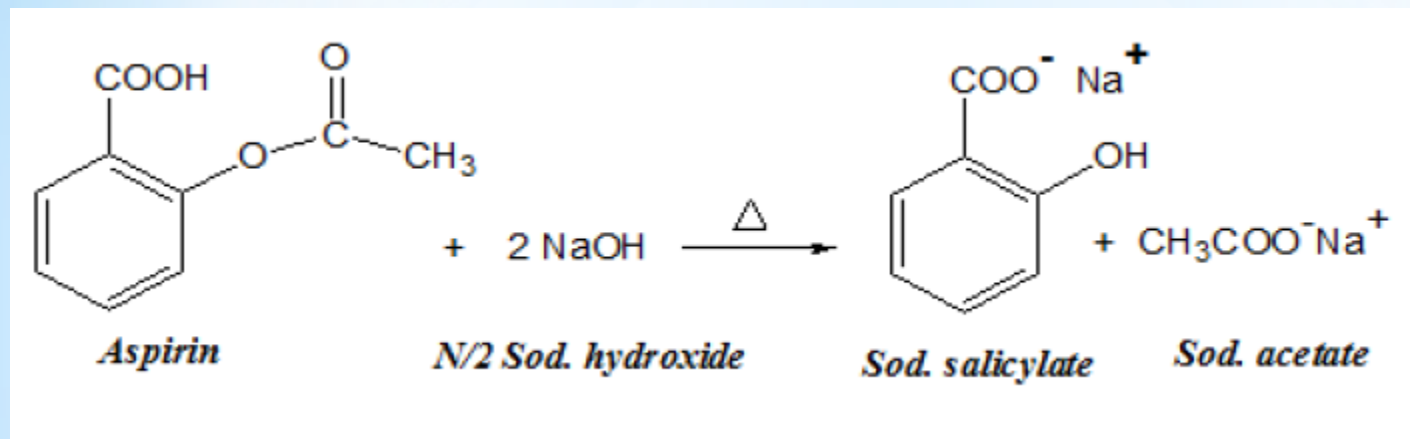
False end point

Phenol Red Indicator:

It's also known as phenolsulfonphthalein . pH indicator (PSP) is a pH indicator .



Calculations :



excess 0.5 N
 unreacted

1 mole of aspirin = 2 mole of NaOH

0.045 g of aspirin = 1 ml of 0.5 N NaOH

	Sample exp.	Blank exp.
Weight of the powdered aspirin used	0.5 g	-
Volume of 0.5 N NaOH used	30ml	30ml
Volume of 0.5 N HCl used	19ml	28ml

Each 0.045 g of aspirin = 1 ml of 0.5 N NaOH Std. sol.

Each 0.045 g of aspirin = 1 ml of 0.5 N HCl Std. sol.

$$V_2 - V_1 = 28 - 19 = 9 \text{ ml}$$

9 x 0.045 = 0.405 g of aspirin in the sample