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Back Titration

Quantitative Assay of Aspirin Tablets

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Methods of Analytical Chemistry is of two types:

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 Titremetric 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. $CaCO_3$

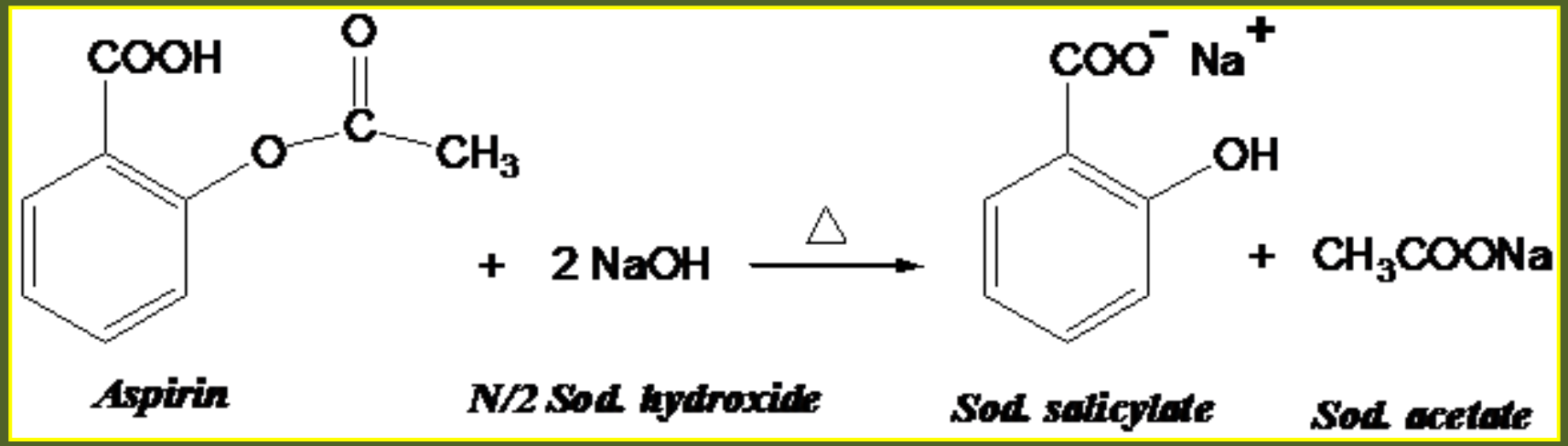
3- Substances for which the quantitative reaction proceeds rapidly only in the presence of excess of reagent, e.g., Lactic acid & Aspirin.

4- Substances which decompose on heating, e.g. , Formaldehyde.

Assay Of Aspirin

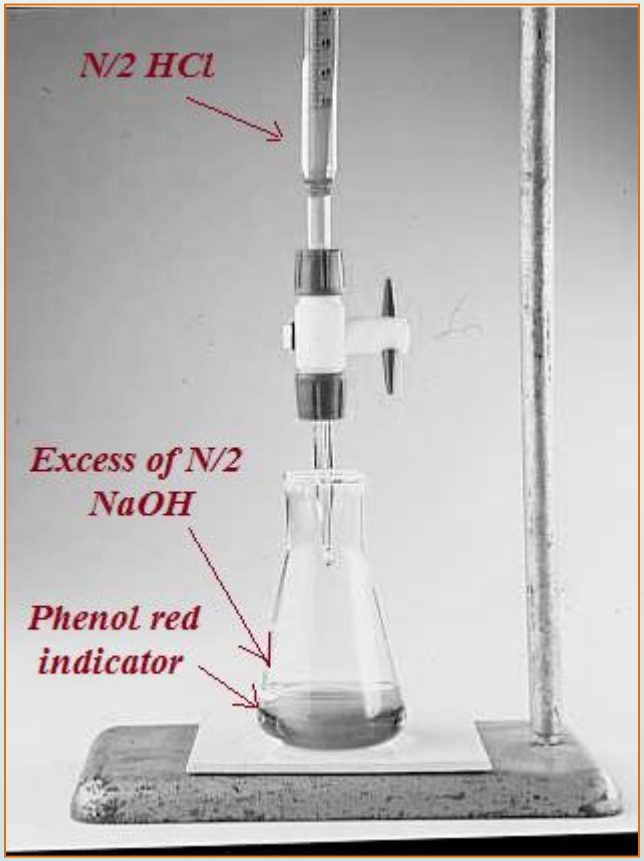
Principle:

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.

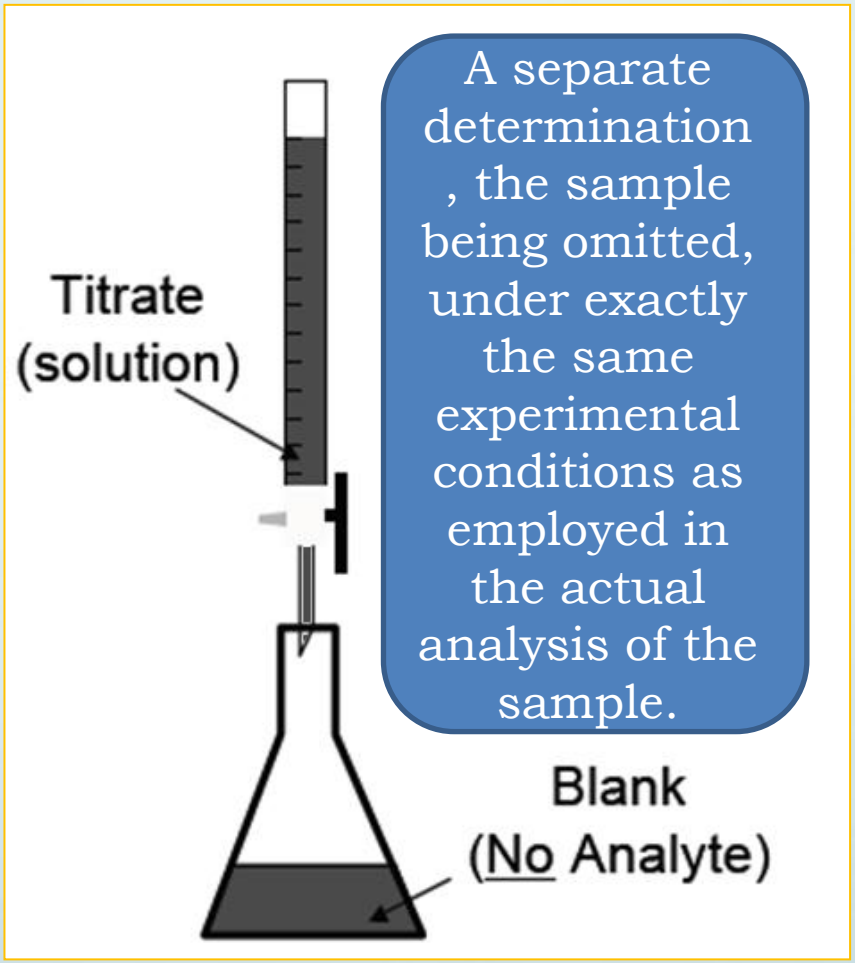
Titration of the excess unreacted alkali with N/2 HCl std. solution using phenol red indicator



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

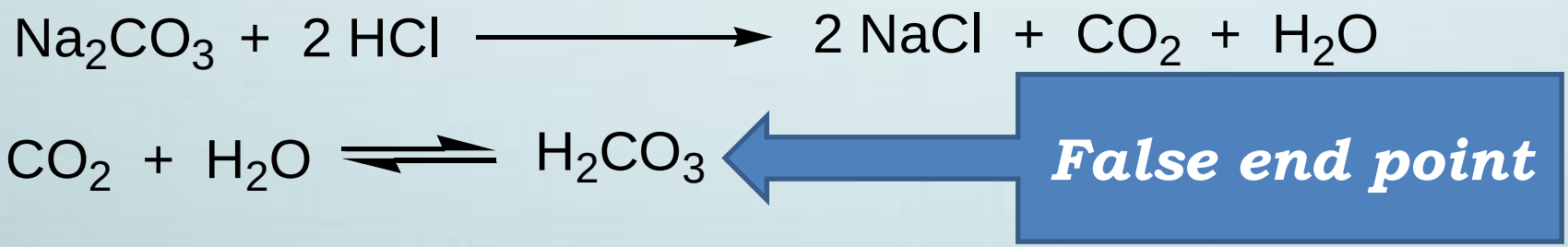
In order to:

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 .**

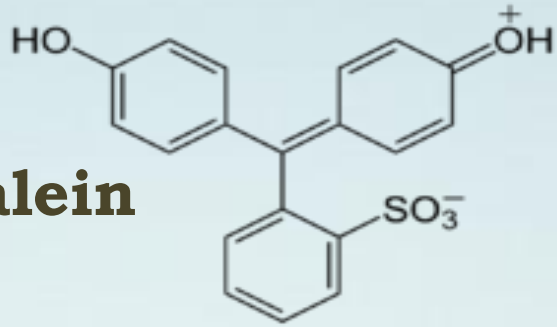


This change may be due to the interaction of the reagent with the glass or due to , the absorption of atmospheric CO_2 ,
CO_2 is rapidly absorbed by the hot alkaline solution to form sodium carbonate .

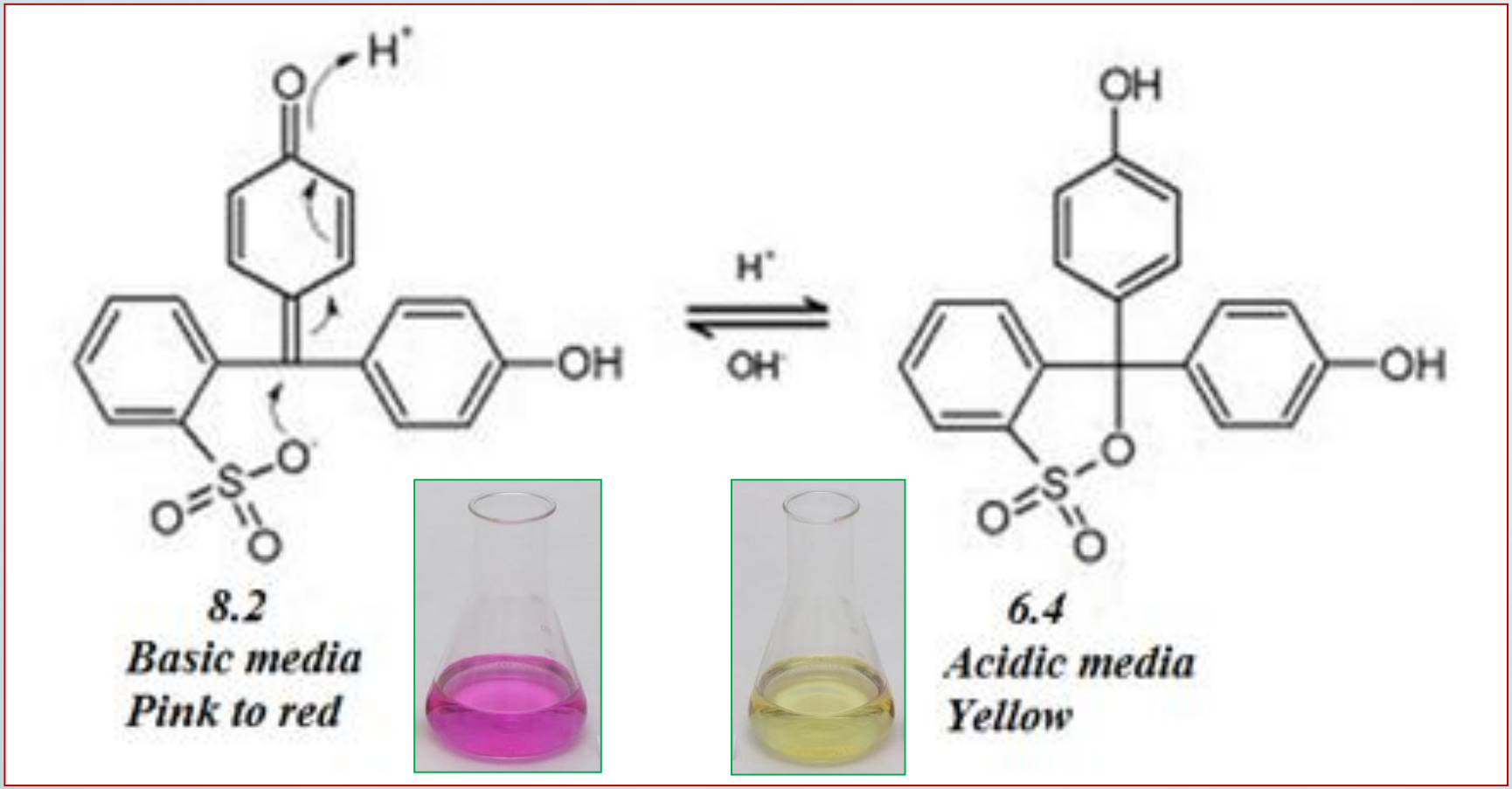
In the back titration with the standard acid the librated CO_2 causes a color change of the indicator before the actual end point.

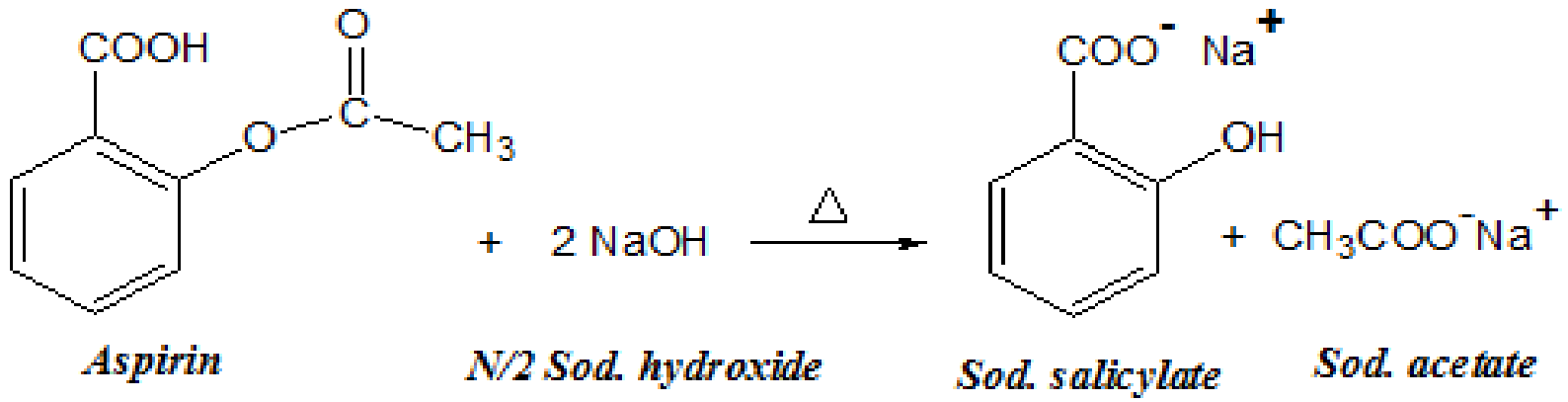


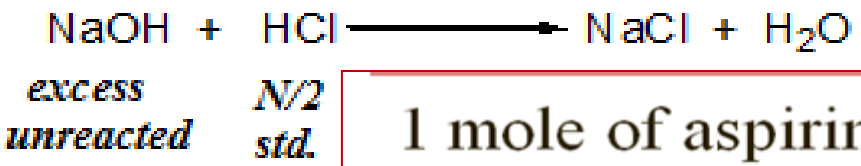
Phenol Red Indicator:



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







Chemical Factor

1 mole of aspirin ≡ 2 moles of NaOH

180 g of aspirin ≡ 2 Eq. of NaOH

180/2 g of aspirin ≡ 1000 ml of 1N NaOH

90/2 g of aspirin ≡ 1000 ml of N/2 NaOH

0.045 g of aspirin ≡ 1ml of N/2 NaOH

Volumes should be corrected to 0.5 N since procedure require that.

A student had crushed 20 aspirin tablets (0.3 g aspirin each) , recorded the weight of the powder as 6.5 g & he quantitatively analyzed aspirin by back titration .

Using 0.5 g of crushed powdered sample, N/2 NaOH & N/2 HCl std. sln. He recorded the results in the following data table:

	Sample exp.	*Blank exp.*
Weight of the powdered aspirin used	0.5 g	_____
Volume of 0.49 N NaOH used	30 ml	30 ml
Volume of 0.51 N HCL consumed	19 ml	28 ml

Each 0.045 g of aspirin ≡ 1 ml of 0.5 N NaOH std. sln.
≡ 1 ml of 0.5 N HCl std. sln.

1- Calculate the average weight of an individual aspirin tablet?

Answer:

The average wt. of an individual tablet = 6.5 / 20

= 0.325 g

2- How many grams of aspirin (pure) is present in the student's sample?

Answer:

1st We should correct the normality of the used HCl to 0.5 N

$N_1 * V_1 = N_2 * V_2$

$0.51 * 19 = 0.5 * V_2$

V_2 = 19.38 ml of 0.5 N HCl for back titration

(reacted with the excess NaOH std. solution)

2nd We should also correct the volume of blank

$N * V = N_3 * V_3$

$0.51 * 28 = 0.5 * V_3$

V_3 = 28.56 ml of 0.5 N HCl used for blank titration.

3rd Calculate the volume of 0.5 N HCl reacted with pure aspirin indirectly.

$V_3 - V_2 = 28.56 - 19.38 = 9.18$ ml of 0.5 N HCl solution.

4th Calculate the amount of pure aspirin present in the sample by using the calculated Chemical Factor.

Each 1ml of 0.5 N HCl is equivalent to 0.045 g of Aspirin.

9.18 * 0.045 = 0.413 g of aspirin in the sample.

Discussion Questions

1- Why did you use your burette and not a graduated cylinder to add the excess NaOH standard solution?

2- What is the definition of:

a- Titration?

b- Equivalence point?

c- End point?

d- Standard solution?

e- Indicator?

f- Molarity & Normality?