College of Pharmacy	الكلية
Pharmaceutical Chemistry	القسم
Pharmaceutical Organic Chemistry II	المادة باللغة الانجليزية
الكيمياء العضوية الصيدلانية	المادة باللغة العربية
Fourth grade	المرحلة الدراسية
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Assay of aspirin	عنوان المحاضرة باللغة الانجليزية
تقدير الاسبرين	عنوان المحاضرة باللغة العربية
5	رقم المحاضرة
Mendham, J., Denney, R. C., Barnes, J. D., & Thomas, M. J. K. (2000). Vogel's textbook of quantitative chemical analysis (6th ed.). Pearson .Education Ltd	المصادر والمراجع
Beckett, A. H., & Stenlake, J. B. (1988). Practical pharmaceutical chemistry (Part 2, 4th ed.). Athlone Press	
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محتوى المحاضرة

Assay of aspirin

There are two types of analytical chemistry 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 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.

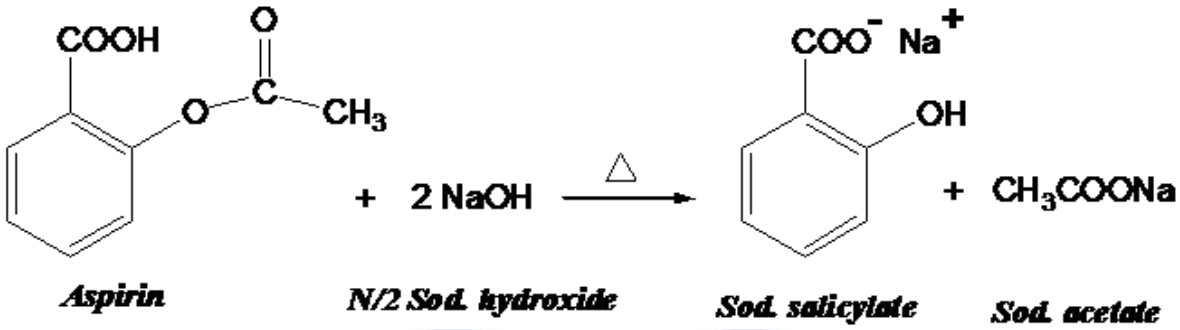
Back Titration Is Used For:

1- Volatile substances, e.g., NH3 .
2- Insoluble or slightly soluble substances, e.g. CaCO3
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

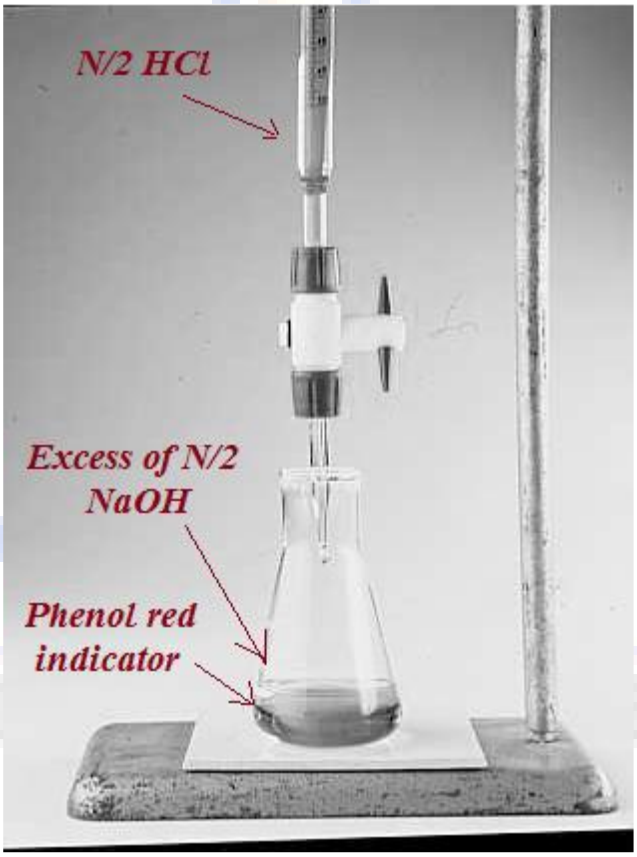
Principle:

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

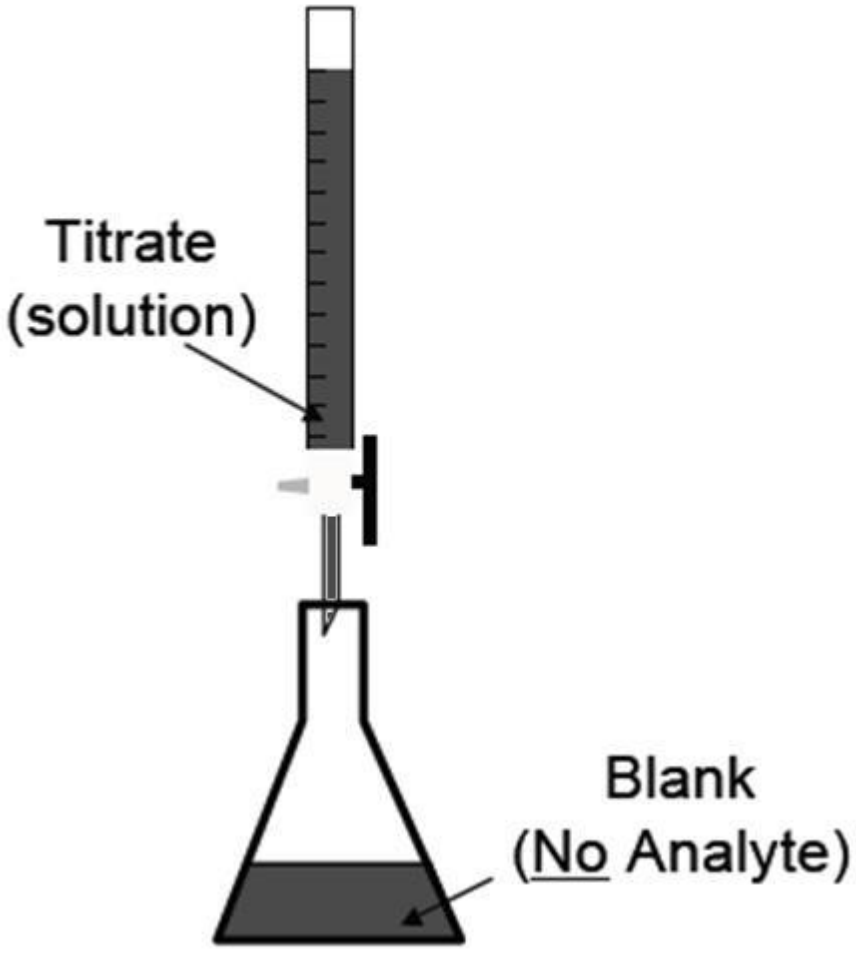


As in other quantitative determination involving boiling with a standard alkali , cooling and back titrating the excess, it's necessary to carry out a blank experiment without the aspirin

In order to:

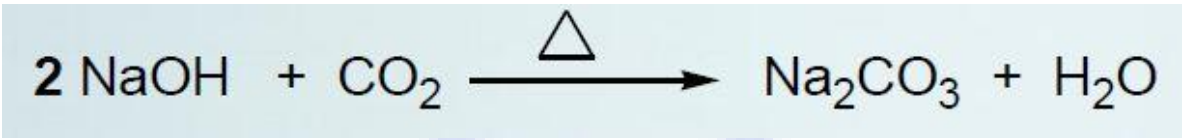
1- Minimize any error due to small unavoidable losses.

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 .

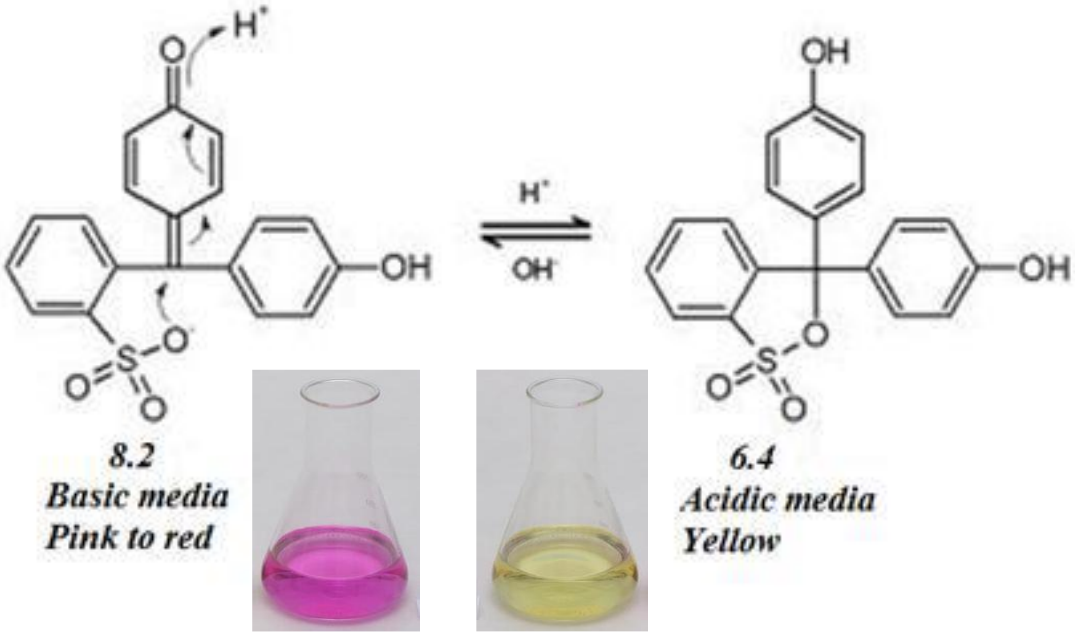


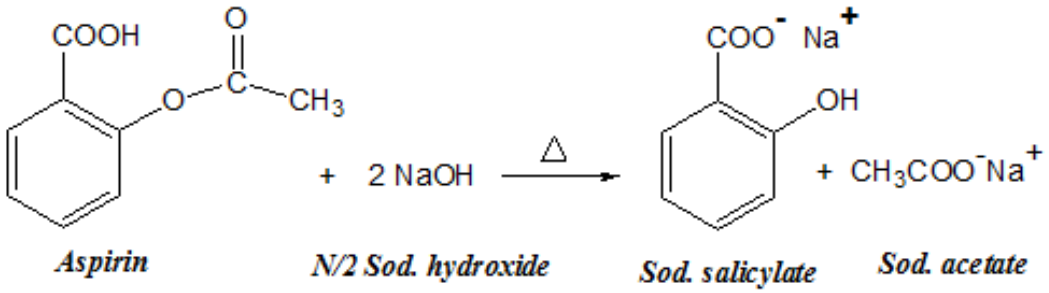
In the back titration with the standard acid the librated CO2 causes a color change of the indicator before the actual end point.

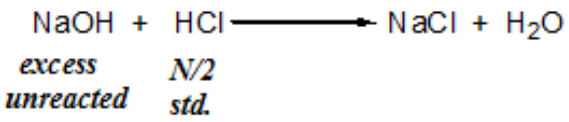
Phenol Red Indicator:

It's also known as phenolsulfonphthalein .

(PSP) is a pH indicator.







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.

$$V_3 - V_2 = 28.56 - 19.38 = 9.18 \text{ ml of 0.5 N HCl solution.}$$

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

$$9.18 * 0.045 = 0.413 \text{ g of aspirin in the sample.}$$

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?

Assay of Aspirin (Known)

1 .Add 0.5 g of Aspirin to 30 ml of (0.5 N) sodium hydroxide, and heat the mixture on a hot plate at 90°C for 10 minutes.

2 .Cool the mixture (1), transfer it to a conical flask, and add 3 drops of (Ph Ph) indicator.

3 .Fill a burette with (0.5 N) hydrochloric acid and titrate the solution until the color changes. Record the volume as V1 (Sodium hydroxide with Aspirin).

4. Perform a blank titration by adding 30 ml of (0.5 N) Sodium hydroxide to a conical flask, then add 3 drops of (Ph Ph) indicator and titrate with (0.5 N)

hydrochloric acid until the color changes. Record the volume as V2 (sodium hydroxide without aspirin).

$$V_{diff} = V_2 - V_1$$

$$\text{wt of Asp} = V_{diff} \times 0.04504$$