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| **Ministry of Higher**  **Education** |  | **وزارة التعليم العالي والبحث العلمي** |
| **Al-Rasheed University College** | **كلية الرشيد الجامعه** |

**Pharmaceutical Chemistry**

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**Lab. (3)**

**Assay of Aspirin**

**Presented By**

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**Assay of aspirin tablets ( back titration)**

1-Introduction

(assay ) : it's an analytical process used to determine the quantity of a compound in a given sample . There are two analytical methods :

**1-qualitative analysis :** determines the presence or absence of a compound in a given sample , but do not determine the quantity .

**2- quantitative analysis :** determine the quantity of a compound in a given sample

**Methods of quantitative analysis**

1-volumetric analysis (titrimetric )

2-Gravimetric analysis

3-spectrophotometric analysis

**Requirements of titrimetric analysis**

1-The reaction can be represented by a chemical equation.

2-The reaction should be relatively fast.  
3-The reaction should be fully completed & irreversible.  
4- easily detected end point.

**Types of Titration:**

1 - Forward titration (direct titration).

2 - Back titration (indirect titration).

**Back Titration:**

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

**Back Titration Is Used For:**

1 - Volatile substances, e.g., NH 3 .  
2 - Insoluble or slightly soluble substances, e.g. CaCO3  
3 - Substances having very fast quantitative analysis only in the presence of excess of reagent, e .g ., Lactic acid & Aspirin .  
4 - Substances which decompose on heating, e.g. Formaldehyde.

**2-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 .

**•** 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 liberated CO 2

causes a color change of the indicator before the actual end point .

**Procedure :**

1-weigh a quantity of aspirin tablet powder equivalent to 0.5 g of aspirin

2- add 30 ml of 0.5 N NaOH solution.

3-Boil gently for 10 min.

4- titrate against 0.5 N HCl using phenol red as indicator .

5-Repeat the process without the powder.

\*the difference between the two titrations represents the amount of 0.5N NaOH required by the aspirin .

**Calculations** :

1 mole of aspirin = 2 mole of NaOH  
180/2 g of aspirin = 1 mole of 0.5 N NaOH in 1000ml   
x g of aspirin = 1 ml of 0.5N NaOH

X= 0.0454 g of aspirin equivalent to 1ml of 0.5N NaOH

**For example = v1 = 28 ml**

**V2= 19**

V 2 -V 1 = 28 -19 = 9 ml  
9 x 0.045 = 0.405 g of aspirin in the sample

**% of aspirin in the tablet**

% purity (% aspirin in tablet) =found (X) gm / actual (0.5 gm) x 100

**Report of lab.3** :

**Name of experiment :**

**Aim of the experiment :**

**Calculations :**

**Wt. of aspirin :**

**Purity of the aspirin in the tablets :**