



*Al-Rasheed University College*  
*Department of Medical Laboratory*  
*Technique*

**Medical Chemistry**

**Lab 3**

*by*

*Dr. Kutaiba Ibrahim Alzand*



## Qualitative and Quantitative Analysis:

The **qualitative analysis** deals with the identification of elements, ions, or compounds present in a sample (we may be interested in whether only a given substance is present).

The **quantitative analysis** deals with the determination of how much of one or more constituents is present.

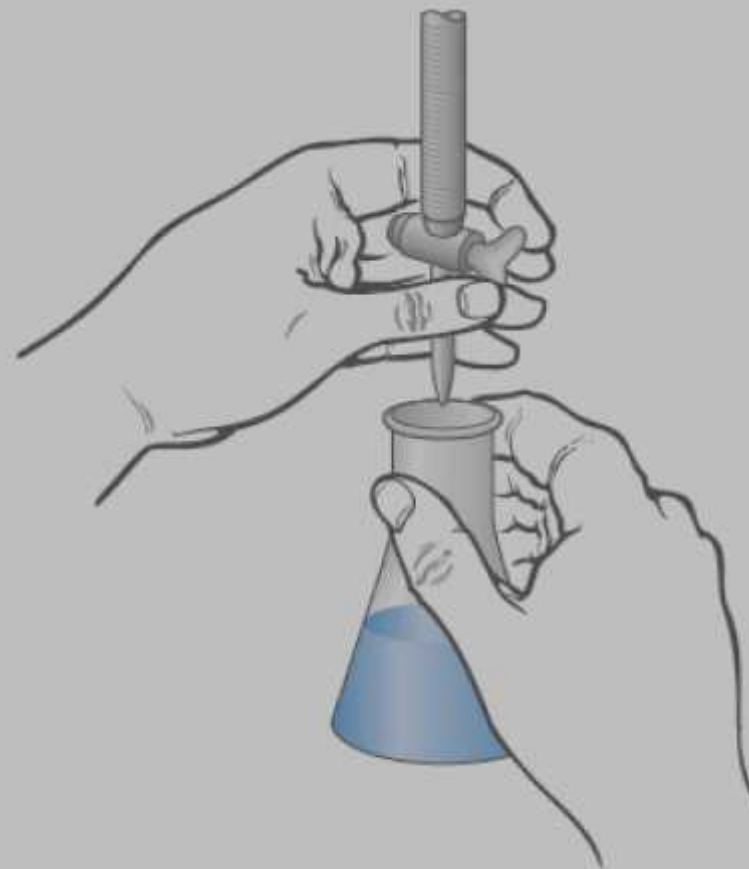
Qualitative analysis tells us what chemicals are present.

Quantitative analysis tells us how much.

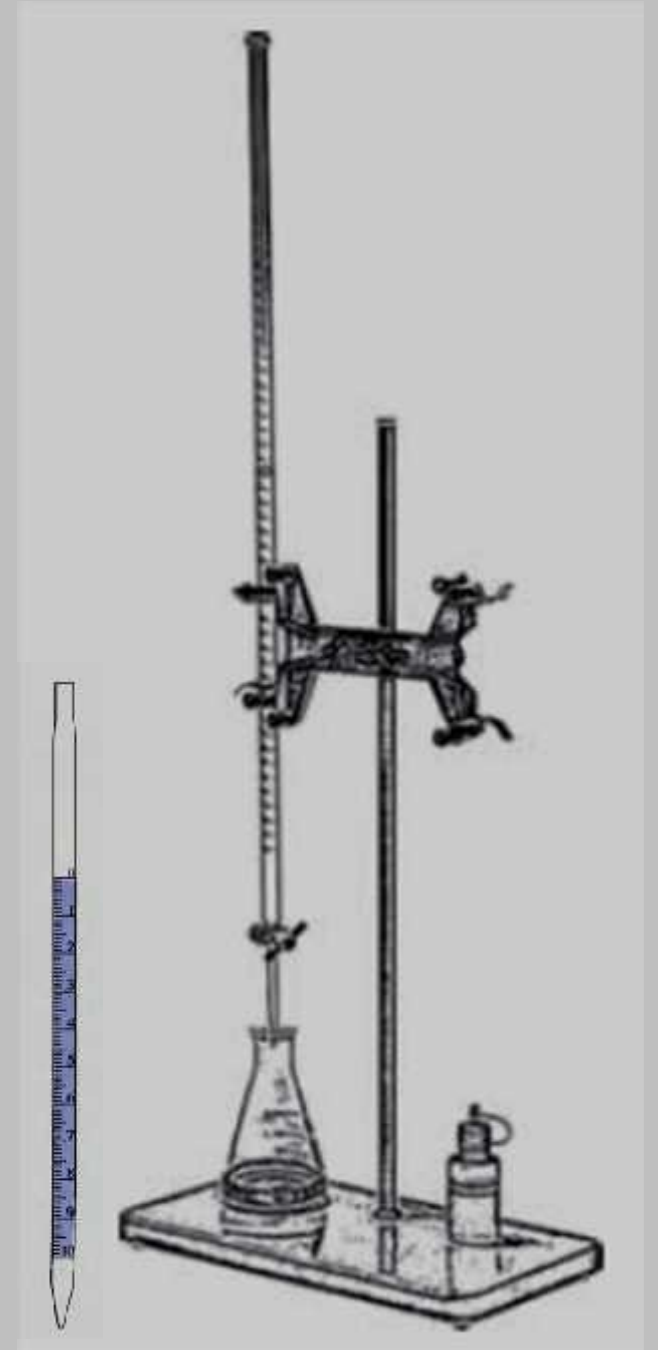
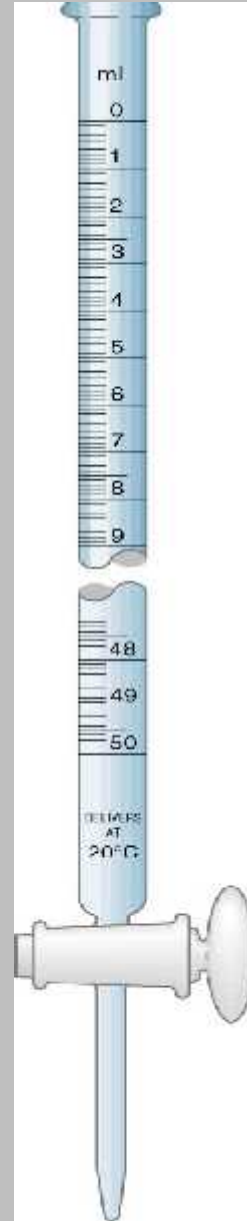
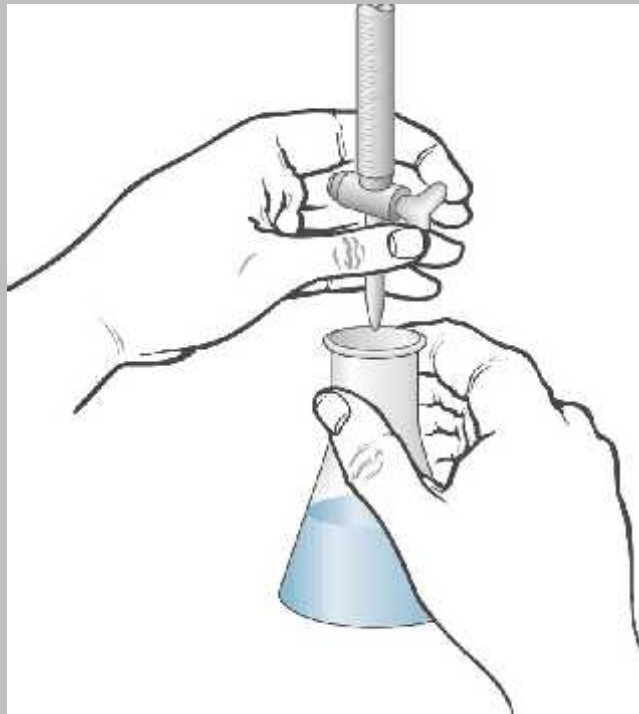
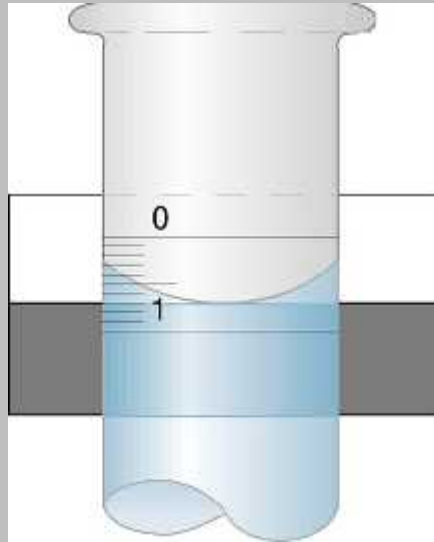
**Analytes** are the components of a sample that are determined.

# TITRATION

- In a **titration**, the test substance (analyte) reacts with an added reagent of known concentration.
- The reagent of known concentration is referred to as a **standard solution**. It is typically delivered from a buret; the solution delivered by the buret is called the **titrant**.
- The volume of titrant required to just completely react with the analyte is measured.
- Since we know the reagent concentration as well as the reaction stoichiometry between the analyte and the reagent, we can calculate the amount of analyte.



We calculate the moles of analyte titrated from the moles of titrant added and the ratio in which they react.



A **standard solution** is a reagent of known concentration. Standard solutions are used in titrations and in many other chemical analyses.

The **equivalence point** is the point in a titration when the amount of added standard reagent is equivalent to the amount of analyte.

The **end point** is the point in a titration when a physical change occurs that is associated with the condition of chemical equivalence.

A **primary standard** is an ultrapure compound that serves as the reference material for a titration or for another type of quantitative analysis.

A **secondary standard** is a compound whose purity has been determined by chemical analysis. The secondary standard serves as the working standard material for titrations and for many other analyses.

## A primary standard should fulfill these requirements:

- It should be *100.00% pure*
- It should be *stable to drying* temperatures
- It should be *readily* and relatively inexpensively *available*.
- Although not essential, it should *have a high formula* weight.
- If it is to be used in titration, it should *possess the properties required for a titration*.

## CLASSIFICATION OF TITRATION METHODS

1. Acid–Base.
2. Precipitation.
3. Complexometric.
4. Reduction–Oxidation.

## Volumetric Calculations—Let's Use Molarity

$$M \text{ (mol/L)} \times L = \text{mol} \quad M \text{ (mmol/mL)} \times \text{mL} = \text{mmol}$$

$$g = \text{mol} \times \text{fw (g/mol)} \quad \text{mg} = \text{mmol} \times \text{fw (mg/mmol)}$$

$$g = M \text{ (mol/L)} \times L \times \text{fw (g/mol)}$$

$$\text{mg} = M \text{ (mmol/mL)} \times \text{mL} \times \text{fw (mg/mmol)}$$

- We usually work with millimole (mmol) and milliliter (mL) quantities in titrations; therefore, the above equations are more useful.
- Note that the expression for formula weight contains the same numerical value whether it be in g/mol or mg/mmol.

Learn these relationships well. They are the basis of all volumetric calculations, solution preparation, and dilutions. Think units!

# Acid–Base Titrations

## Solutions and Indicators for Acid/Base Titrations

- Like all titrations, neutralization titrations depend on a chemical reaction of the analyte with a standard reagent

### There are several different types of acid/base titrations.

- One of the most common is the titration of a strong acid, such as hydrochloric or sulfuric acid, with a strong base, such as sodium hydroxide.
- Another common type is the titration of a weak acid, such as acetic or lactic acid, with a strong base.
- Weak bases, such as sodium cyanide or sodium salicylate, can also be titrated with strong acids.
- In all titrations, we must have a method of determining the point of chemical equivalence.
- Typically, a chemical indicator or an instrumental method is used to locate the end point, which we hope is very close to the equivalence point.



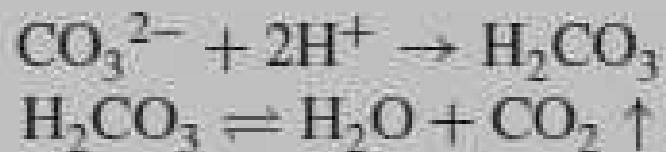
## Acid–Base Titrations

### EXPERIMENT 3 Standardization of Hydrochloric Acid against Sodium Carbonate

#### Principle

One-tenth molar hydrochloric acid is standardized against primary standard sodium carbonate. Phenolphthalein is used to approximate the halfway point of the titration, and then either modified methyl orange indicator or bromcresol green indicator is used to detect the final end point; the last indicator is used with boiling of the solution near the end point to remove CO<sub>2</sub>

#### Equations



In a **standardization**, the concentration of a volumetric solution is determined by titrating it against a carefully measured quantity of a primary or secondary standard or an exactly known volume of another standard solution.

## Solutions and Chemicals Required

1. *Provided.* 0.2% phenolphthalein in 90% ethanol, either modified methyl orange or 0.1% bromcresol green and primary standard  $\text{Na}_2\text{CO}_3$ .
2. *To prepare.* 0.1 M HCl. Concentrated hydrochloric acid has a density of 1.18 and contains 37% by weight HCl. Hence, about 4 mL concentrated acid should be diluted to 500 mL to make 0.1 M acid.

## PROCEDURE

1. Dry a quantity of primary-standard  $\text{Na}_2\text{CO}_3$  for about 2 hr at  $110^\circ$  and cool in a desiccator. Weigh 0.20-g to 0.25-g  $\text{Na}_2\text{CO}_3$  sample into 250-mL conical flasks, and dissolve in about 50 mL of distilled water then add 2 drops of phenolphthalein indicator solution.

2. Rinse the buret three times with small portions (about 5mL each) of the approximately 0.1 M HCl acid prepared above, then fill and adjust to near the zero mark.
3. Titrate the sample of the sodium carbonate until the pink color disappears. At this point, about half the total volume of acid necessary has been added (actually a slight excess).
4. Introduce 3 drops of methyl orange indicator, and titrate with HCl until the solution just begins to change from yellow to red.
5. Boil the solution for 2 to 3 min, cool to room temperature, and complete the titration. The indicator should change from red to yellow as  $\text{CO}_2$  is removed during heating.
6. Calculate the concentration of the HCl solution.

## The Standardization of Acids

Sodium carbonate is the most frequently used reagent for standardizing acids. Several other reagents are also used.

### Sodium Carbonate

Primary-standard-grade sodium carbonate is available commercially or can be prepared by heating purified sodium hydrogen carbonate between 270 to 300°C for 1 hr.

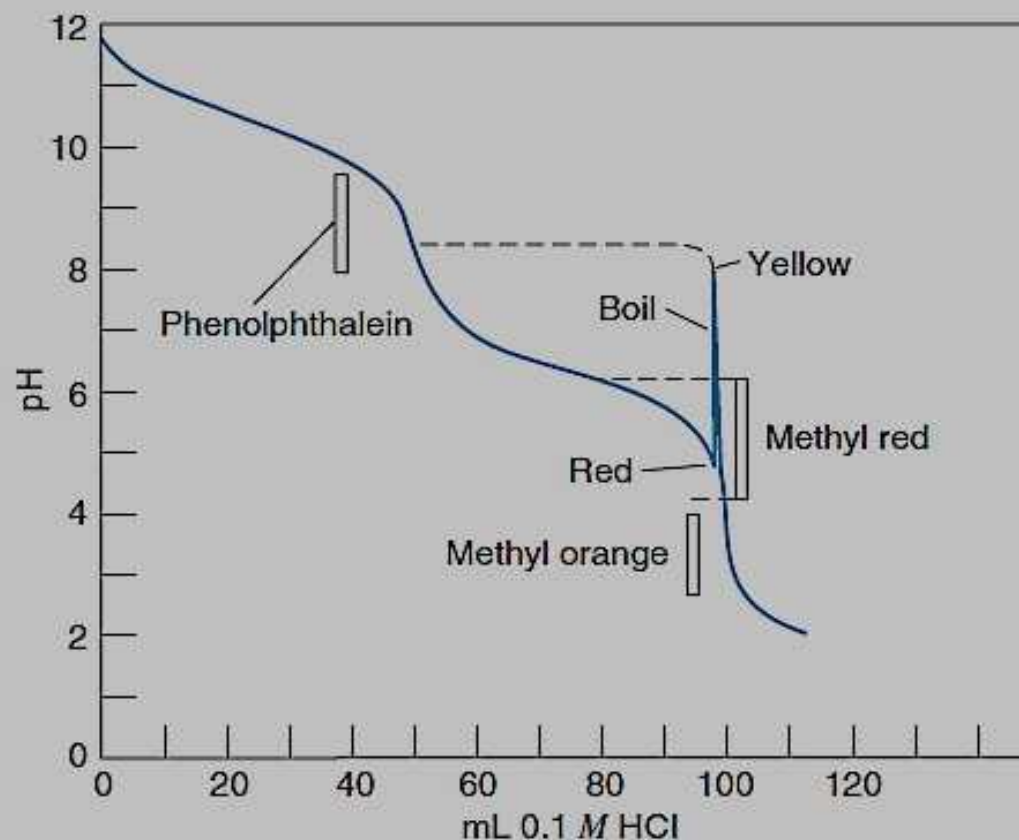


An accurately determined mass of the primary-standard material is then taken to standardize the acid.

As shown in Figure 1, there are **two end points** in the titration of sodium carbonate. **The first**, corresponding to the conversion of carbonate to hydrogen carbonate, occurs at about **pH 8.3**; **the second**, involving the formation of carbonic acid and carbon dioxide, appears at about **pH 3.8**.

The second end point is always used for standardization because the change in pH is greater than that at the first.

An even sharper end point can be achieved by boiling the solution briefly to eliminate the reaction product, carbonic acid and carbon dioxide.



**Fig. 1.** Titration curve for 50mL 0.1 M  $\text{Na}_2\text{CO}_3$  versus 0.1 M HCl. Dashed line represents a boiled solution with  $\text{CO}_2$  removed.

The sample is titrated to the first appearance of the acid color of the indicator (such as bromocresol green or methyl orange).

At this point, the solution contains a large amount of dissolved carbon dioxide and small amounts of carbonic acid and unreacted hydrogen carbonate.

Boiling effectively destroys this buffer by eliminating the carbonic acid:



The solution then becomes alkaline again due to the residual hydrogen carbonate ion.

The titration is completed after the solution has cooled, resulting in a substantially larger decrease in pH during the final additions of acid.

## Problems

1. Describe the preparation of 50 mL of 0.1 M NaOH from the solid reagent.
2. Describe the preparation of 50 mL of 0.5 M HCl from the commercial reagent that has a specific gravity of 1.18 and is 37% (w/w) HCl (36.5 g/mol).
3. An approximate 0.1 M hydrochloric acid solution is prepared from the commercial concentrated hydrochloric acid. It is standardized by titrating 0.1876 g of dried primary standard sodium carbonate:



The titration required 35.86 mL acid. Calculate the molar concentration of the hydrochloric acid.

4. Acetic acid in 2.5-mL vinegar sample is determined by titrated with 0.1 M NaOH solution, requiring 12.7 mL. Calculate the percent of CH<sub>3</sub>COOH in the sample.
5. The aspirin in a 0.5-g sample is analyzed by adding 30 mL of 0.1 M NaOH, a slight excess. The sample is heated to speed the hydrolysis. The excess NaOH is back-titrated with 12.7 mL of 0.1 M HCl. Calculate the aspirin amount in the sample in mg.