

# Titration

In chemistry, a **solution** is a homogeneous mixture composed of two or more substances. In such a mixture, a **solute** is dissolved in another substance, known as a solvent. An **aqueous solution** is a solution in which the solvent is water.

**Concentration** is the measure of how much of a given substance (solute) there is mixed with another substance (solvent – water). There are a number of different ways to quantitatively express concentration; in this work we will use molar concentration. Molar concentration (**Molarity**) denotes the number of moles ( $n$ ) of a given substance per litre of solution

$$c = \frac{n}{V} \quad (\text{mol/l or M}) \quad \text{or} \quad c = \frac{m}{M.V} \quad (1)$$

where  $V$  (in l) is the volume of solution,  $m$  (in grams) is the mass of a given substance with the molar mass  $M$  (g/mol).

**Titration** is a common laboratory method of quantitative/chemical analysis that can be used to determine the concentration of a known reactant (**analyte**). The basis of the method is a chemical reaction of a **standard solution (titrant)** with a solution of an analyte.

The **analyte (A)** is a solution of the substance whose concentration is unknown and sought in the analysis. The **titrant (T)** is a solution in which the concentration of a solute is precisely known. Because volume measurements play a key role in titration, it is also known as **volumetric analysis**. Usually it is the *volume of the titrant* required to react with a given quantity of an analyte that is *precisely* determined during a titration.

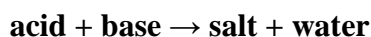
Using a calibrated burette to add the titrant, it is possible to determine the exact amount that has been consumed when the **endpoint** of titration is reached. The endpoint is the point at which the titration is complete, as determined by an **indicator** (see below). At the titration endpoint, the quantity of reactant in the titrant added during the titration is *stoichiometrically equivalent* to the quantity of reactant in the analyte. This is ideally the same volume as the **equivalence point** - the volume of added titrant at which the number of moles of titrant ( $n_T$ ) is equal to the number of moles of analyte ( $n_A$ ), or some multiple thereof.

Titrations can be classified by the type of reaction. Different types of titration reaction include acid-base titrations, redox titrations, complexometric titrations, etc.

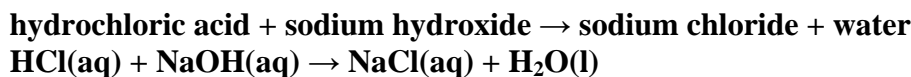
Within practicals from physical chemistry we will deal with acid-base titrations.

**Acid-base titrations** are based on the neutralization reaction between the analyte and an acidic or basic titrant. These most commonly use a pH indicator, a pH meter, or a conductance meter to determine the endpoint. In our experiments we will use a pH indicator to detect the endpoint of the reaction.

**Neutralization** is a chemical reaction, also called a **water forming reaction**, in which an acid and a base or alkali (soluble base) react and produce a salt and water (H<sub>2</sub>O).



For example, the reaction between hydrochloric acid and sodium hydroxide solutions:



Before starting the titration a suitable pH indicator must be chosen. The endpoint of the reaction, when all the products have reacted, will have a pH dependent on the relative strengths of the acids and bases. The pH of the endpoint can be roughly determined using the following rules:

- A strong acid reacts with a strong base to form a neutral (pH=7) solution.
- A strong acid reacts with a weak base to form an acidic (pH<7) solution.
- A weak acid reacts with a strong base to form a basic (pH>7) solution.

When a weak acid reacts with a weak base, the endpoint solution will be basic if the base is stronger and acidic if the acid is stronger. If both are of equal strength, then the endpoint pH will be neutral.

Frequently, during a titration it is also useful to monitor the progress of the titration with a graph. This graph is known as a titration curve. Such a curve reflects the changes in pH that occur as titrant is added from a burette to the analyte in the beaker below the burette.

There are two different types of acids that can be titrated, besides being strong or weak. They are known as being monoprotic or polyprotic:

**Monoprotic acids** contain one acidic hydrogen: hydrochloric acid HCl, nitric acid HNO<sub>3</sub>, acetic acid CH<sub>3</sub>COOH, etc. Titration curve of strong monoprotic acid with strong base is shown in Fig. 1A.

**Polyprotic acids** contain more than one acidic hydrogen. They are always identified by their formulas showing more than one H. Specifically, a diprotic acid will have two hydrogens (sulphuric acid H<sub>2</sub>SO<sub>4</sub>, succinic acid (CH<sub>2</sub>)<sub>2</sub>-(COOH)<sub>2</sub>), a triprotic acid will have three hydrogens (phosphoric acid H<sub>3</sub>PO<sub>4</sub>, citric acid HOOC-COH-(CH<sub>2</sub>-COOH)<sub>2</sub>), etc. Titration curve of strong diprotic acid with strong base is shown in Fig. 1B.

A suitable indicator should be chosen, that will experience a change in color close to the end point of the reaction.

**pH indicators** are generally very complex organic molecules (frequently weak acids or bases). When introduced into a solution, they may bind to H<sup>+</sup> or OH<sup>-</sup> ions. They will contain a structural component that is called a chromophoric group. This group, or chromophore, will have a structure that changes slightly when the pH of the system changes. The indicator will have one structure through one range of pH values, and a

second structure through a second range of pH values. When the structure changes, as a response to pH, the chromophore will also change its color (for more information see e.g. <http://www.bcpl.net/~kdrews/titration/indicators.html>).

In our exercises we will titrate weak acids (acetic, succinic, citric) (resp. strong acid HCl) with a strong base (NaOH). We will use **phenolphthalein** ( $C_{20}H_{14}O_4$ ) as pH indicator. It is colorless in acidic solutions (pH <8.2), and in basic solutions it turns from weak pink up to fuchsia color (pH ~10).

### Titration procedure:

1. A known volume  $V_A$  of the analyte (acid) is placed in a titration flask.
2. The burette is filled by a standard solution (titrant, base) of known concentration  $c_T$  (M).
3. Before the titration is started, 1-3 drops of indicator (phenolphthalein) is placed in the titration flask with the analyte (acid). The chosen indicator must be one color when the solution is acidic.
4. A base solution is then slowly added from the burette, drop by drop.
5. The titration continues, drop by drop, until the indicator suddenly achieves the intermediate color (weak pink) between that of the acid and the color of the base (fuchsia). At that point the titration ceases.
6. The point at which the system is neither acidic or basic is referred to as the endpoint. The endpoint will correspond to a perfect stoichiometric relationship between the acid and the base.
7. Once the endpoint has been reached, the burette must be read. The bottom of the meniscus line determines the quantity of the base  $V_T$  that was required to reach the endpoint.
8. Once the titration is completed, the final calculations can be done.

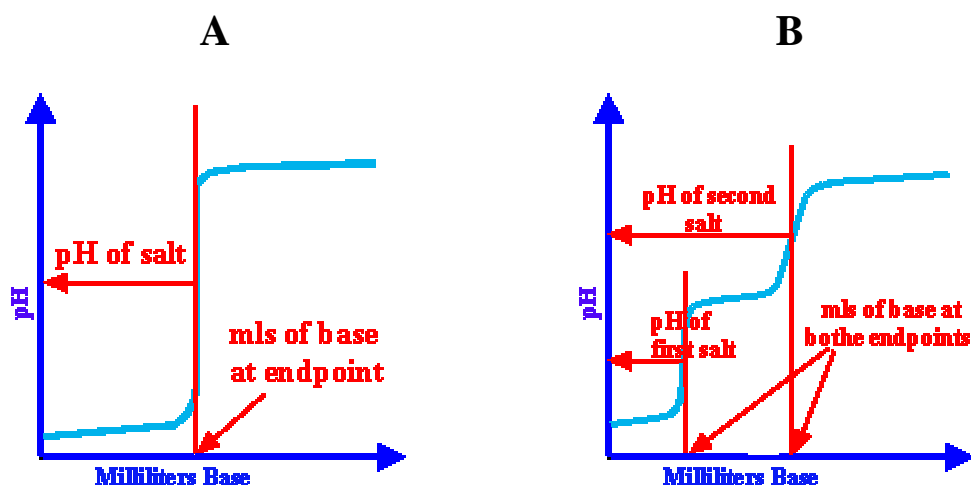


Fig. 1: Titration curve of strong monoprotic acid (A), and strong diprotic acid (B).

## Calculations:

Calculating the concentration  $c_A$  (M) of the analyte (acid):

1. From the balanced chemical equation

moles analyte = moles titrant

$$n_A = n_T \quad (2)$$

2. moles titrant:  $n_T = V_T \cdot c_T$  (3)

3. because  $n_T = n_A$ , the concentration of the analyte  $c_A = \frac{n_A}{V_A}$  (4)

**Remark:** Above mentioned relations are valid for monoprotic acids. **Polyprotic acids** contain more than one acidic hydrogen, so stoichiometric relationship between the acid and the base is for:

diprotic acids  $n_A = 2 \cdot n_T$

triprotic acids  $n_A = 3 \cdot n_T$

## Objective:

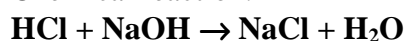
### Determination of molar concentration of acids by titration

#### Equipments and chemicals:

burette, pipettes, titration flasks, funnel, beaker, distilled water, phenolphthalein,  
analyte: hydrochloric acid, citric acid, acetic acid,  
titrant: sodium hydroxide (0.5 M)

### Determination of molar concentration of HCl by titration with NaOH:

Chemical reaction:

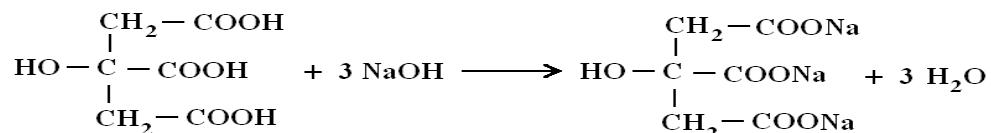


#### Procedure:

1. Fill the titration flask with  $V_A=10$  ml of HCl from stock HCl solution. An approximated concentration  $c_{APP}$  (M) of the stock solution is marked on the bottle with HCl, write it down to the table.
2. Add to the solution of acid 1-2 drops of phenolphthalein.
3. Fill the burette with a standard solution of NaOH (titrant). Read the concentration of titrant  $c_T$  (M) from the bottle and write it down to the table.
4. Perform the titration.
5. When the endpoint of titration has been reached, read the used volume of NaOH from the burette ( $V_T$ ). Write it down to the table.
6. Repeat the procedure.
7. Each student **must** perform two titrations of each analyte. Each group member will contribute the results to the group effort. Determine the average volume of titrant from four titrations  $\bar{V}_T$  (ml).
8. Applying relation (2) – (4), express the precise concentration of HCl,  $c_A$  (M).

### Determination of molar concentration of citric acid by titration with NaOH:

Chemical reaction:



Repeat the same procedure as above. Citric acid is a triprotic acid, so correct the equations (2) – (4) for stoichiometric relationship according the reaction.

### Determination of molar concentration of acetic acid by titration with NaOH:

1. Write the chemical reaction of acetic acid with NaOH.
2. Repeat the same procedure as for titration of hydrochloric acid. The concentration of acetic acid is unknown.

Table:

acid	$V_A$ (ml)	$c_{APP}$ (M)	titrant $c_T$ (M)	Volume of titrant $V_T$ (ml)				$\bar{V}_T$ (ml)	$c_A$ (M)
				I.	II.	I.	II.		
HCl	10								
citric	10								
acetic	10	----							

- Compare concentrations  $c_{APP}$  and  $c_A$  in conclusions of your report. Express the deviation in %, supposing the value of  $c_{APP}$  is 100%.

Used literature:

<http://en.wikipedia.org/wiki/Titration>

<http://www.dartmouth.edu/~chemlab/techniques/titration.html>

<http://www.bcpl.net/~kdrews/titration/page4.html>

J. Oremusová, Manual for laboratory practice in physics for students of pharmacy, Department of Physical Chemistry, Faculty of Pharmacy, Comenius University, Bratislava, 2007, in Slovak

Manual written by Doc. RNDr. D. Uhríková, CSc.