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Specialty : Food industry technologies, 1st year LMD
Module : Analytical Tools for Chemistry, Biochemistry and Safety

Chapter II : The dosage

1. Introduction

We will use in this chapter the notions of : **conductivity, acid-base reactions and oxidation-reduction reactions**, by combining these concepts, we will be able to determine the amount of matter of a chemical species.

2. Principle of a dosage

To assay (or titrate) a chemical species (molecule or ion) in solution is to determine its molar concentration in the solution considered.

3. The different methods of dosage

3.1. Non-destructive methods :

They do not involve chemical reactions.

Physical quantities are used, the value of which depends only on the species concentration of the solution :

- Variation of the refractive index.
- Variation of light absorption (absorbance).
- Variation of the conductance G.

3.2. Destructive or direct dosages :

A chemical reaction is then used.

The titrated reagent is the species whose concentration is to be determined, it is contained in the solution to be assayed.

A titrating solution containing a titrating reagent chosen as a function of the species to be assayed is used.

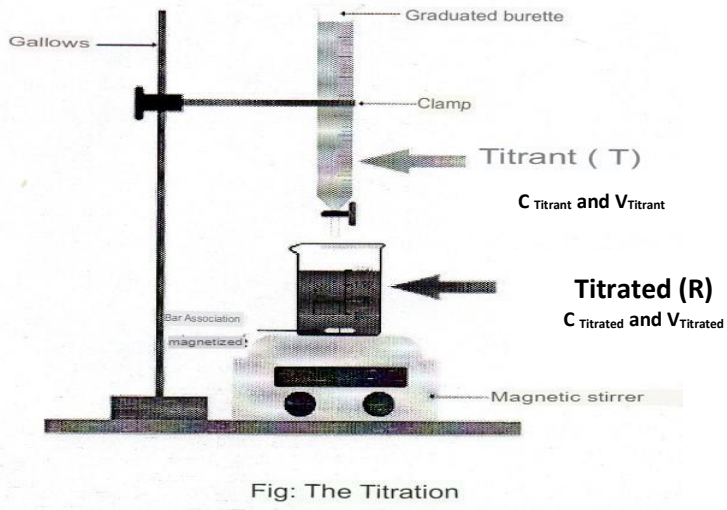

Fig: The Titration

The titrant is placed in a precision graduated burette surmounting a beaker containing a precise titrated volume of the titrated solute, the titrated concentration of which it is desired to determine.

4. Course of a direct dosage :

- The titrating solution is poured into the solution to be titrated using the burette.

The assay reaction then takes place which brings into play the titrated reagent and the titrating reagent. This can be either acid-base or oxidation-reduction.

- For a chemical reaction to be used as a dosing reaction, it must be :
  - Univocal : it is necessary that the two reagents, titrated and titrating, react according to a single and unique reaction.
  - Total : One of the two reagents brought together must completely disappear.
  - Fast.
- Until when is it necessary to pour the titrating solution ?

The titrating solution is poured in until the titrated reagent is completely reacted. We then reach equivalence.

During the assay, the reactants react in stoichiometric proportions.

Before equivalence, the titrating reagent is the limiting reagent (each time it is poured in, it disappears).

At equivalence, the reagents are completely consumed.

After equivalence, the titrating reagent is introduced in excess (there is no more titrated reagent, therefore no more reaction).

- What is happening in terms of the progress of the reaction ?

With each addition of titrating reagent, the progress is maximum. At equivalence, the two reactants are completely consumed and the advancement takes the value xeq.

- Identification of equivalence :

This is the purpose of each assay, to identify the equivalence and to note the volume of titrating solution that we have introduced. This identification can be carried out either by :

- A change in the color of the reaction medium (frequent in redox).
- A change in the color of a colored indicator. It was introduced prior to dosing into the solution to be titrated.
- The drawing of a curve

Example : oxidation-reduction assay of iron II ions with a potassium permanganate solution

The on iron(II) solution is in a beaker while the titrating solution containing the permanganate ions is delivered by a burette.

a. The dosing reaction :

MnO_4^-+8 H++5 Fe_2+ →Mn_2++4 H2O + 5 Fe_3+

The permanganate solution decolorizes as it is added because the MnO_4^- purple aq ions disappear to give way to the Mn_2+ transparent aq ions.

At equivalence, the Fe2+aq ions have been completely consumed, so there are no more reactions and the MnO_4^- aq ions are in excess, hence the persistence of the purple color at this time.

This reaction involves H + ions as reagents, so it will be necessary to work in an acidic medium. Hydrochloric acid (H+, Cl-) should be avoided because chloride ions have redox properties and could intervene in the dosage. Instead, sulfuric acid H2SO4 will be used.

b. A progress table for the dosing reaction :

Equation		$MnO_{4\,(aq)}^{-}+5Fe^{2+}_{(aq)}+8H^{+}_{(aq)} \rightarrow Mn^{2+}_{(aq)}+5fe^{3+}_{(aq)}+4H_2O_{(l)}$					
System status	Progress (x in mol)	N_{MnO4}	N_{Fe2+}	N_{H+}	N_{Mn2+}	N_{Fe3+}	-
Initial	X=0	C_2*v_2	C_1*v_1	excess	0	0	-
During the dosage	X	C_2*v_2-x	C_1*v_1-5x	excess	x	5x	-
To the equivalence	$X_{eq}=3.6*10^{-4}$	0	0	excess	$3.6*10^{-4}$	1.810^{-3}	-

c. How to spot the equivalence ?

The question now is how to spot the equivalence. We will try to identify the equivalence by a color change in the beaker. It should then be known that the permanganate ions are purple in color while all the other ions are colorless.

- Contents of the beaker before equivalence : the permanganate ion is introduced by default. It is then completely consumed. In the beaker, there are : Mn2+ ion, Fe2+ ion (not completely consumed because they were in excess), Fe3+ ion.

Color of the solution contained in the beaker : colorless

- Content of the beaker to equivalence : the permanganate ion was introduced in stoichiometric proportions with respect to the Fe2+ ions. These two ions were completely consumed by the total assay reaction. In the beaker, it remains : Mn2+ and Fe3+.

Color : colorless

- Contents of the beaker after equivalence : the permanganate ion is introduced in excess. There is no Fe2+ ion left, the permanganate ion accumulates in the medium.

Color : purple

Conclusion : the equivalence can be spotted when the color changes in the beaker from colorless to purple.

d. Determination of the concentration of the reagent to be assayed :

In a 200 cm^3 erlen, 10 cm^3 of a solution containing iron(II) sulfate of unknown concentration Cred, exactly measured with a pipette, and 1 mL of concentrated sulfuric acid are introduced. The solution of potassium permanganate of known concentration equal to Cox =0.02 mol.L-1 is introduced into the burette. The color change in the erlen is obtained after adding 11.9 mL of permanganate ion solution.
Determine the Cred concentration.
What is the concentration of the Cred iron (II) solution ?
We start from the dosing reaction :
MnO4- +8 H+ + 5 Fe2+ $\rightarrow$ Mn2+ + 4 H2O + 5 Fe3+
At equivalence, we have the following relation : n (Fe2+)/5 = n (MnO4-)

By replacing the quantities of matter by the product of the concentrations and volumes :
Cred.Vred/5 = Cox.Voxe where Voxe is the volume of oxidizing solution poured to the equivalence.

You can consult the following video which will allow you to illustrate the phenomenon :
https://www.youtube.com/watch?v=xamXNWEYOIM