# DESIGN OF A LAB "LOW-COST" FOR THE REDOX PROCESSES STUDY



# UNIVERSITAT JAUME•

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## **INTRODUCTION**

## **REDOX EQUILIBRIUM**

Reduction oxidation reactions are chemical transformations of great practical importance, since study phenomena such as combustion of many substances, the oxidation of metals and production processes of energy from the batteries. Likewise, the redox reactions are particularly important in the metabolism of living beings. Cellular respiration and photosynthesis, for example, are metabolic pathways that pass through such reactions.

Formerly it was understood as those oxidation processes in which a substance consumes oxygen and reduction to those where it is released. Later, reduction was accepted as those reactions in which hydrogen was consumed and oxidation in which it is was released.

Currently it considered as redox reactions to those in which the state or degree of oxidation of the reacting species is changed because an exchange of electrons occurs between the reactants, although neither oxygen nor hydrogen are involved.

For a redox reaction to take place it is necessary the presence of two species: those that yield electrons (reductant), and those accepting them (oxidant). After the redox reaction occurs, the reductant species becomes the oxidized form and therefore the oxidant one becomes the reductant form.

The concept of redox reactions is reminiscent from the Bronsted-Lowry acid - base reactions. Both involve the transfer of one or more charged particles from one given to an acceptor, being the electrons the responsible of the redox reactions, meanwhile the protons of the neutralization processes.

Example redox reactions:

The Ce<sup>4+</sup> is a strong oxidant that oxidizes the  $Fe^{2+}$  to  $Fe^{3+}$ , to reduce to  $Ce^{3+}$ :

$$Ce^{4+} + Fe^{2+} < ---> Ce^{3+} + Fe^{3+}$$

If metallic iron is introduced into a solution containing  $Sb^{3+}$ , metallic Sb flakes are formed and thus it can be used to detect the presence of  $Fe^{2+}$  in solution:

$$3Fe + 2Sb^{3+} < ---> 3 Fe^2 + 2 Sb$$

Dissolving metallic aluminum in hydrochloric acid is also a redox reaction :

$$2Al + 6H^+ < ---> 2Al^{3+} + 3H_2$$

It can be seen that:

In redox reactions may be involved, as well as reagents or reaction products, atoms, ions or molecules can be found in solid state, in solution and in gaseous form.

Oxidation is a process in which a kind loses one or more electrons, so that, when an element oxidation state is oxidized making more positive values. An oxidizing species is one that gains electrons, reduced during the process.

In reduction increases the amount of electrons; reduced element takes more negative values of its oxidation state. A reducing agent is one that loses electrons in a reaction, rusting away in the process. Both processes, oxidation and reduction must be verified simultaneously.

## **OXIDATION STATE INDEX**

When covalent substances participate in a redox process, is usually difficult to detect the species involved in the electronic transfer. To overcome this adversity the concept of oxidation number was introduced.

It represents the charge that would have an atom in question considering that the only links that form the chemical species involved in this element are ionic. In ionic compounds, the oxidation number coincides with the electric charge of the ions. Oxidation state is an fictitious electric charge, since the electron transfer between the involved atoms is not total. It determination can be carried out by applying the following rules :

a) Total electric charge of a molecule is zero.

b) The oxidation state of the elements in the ground state or uncombined is zero.

c)The H has an oxidation state  $\pm 1$ .

d)The O has an oxidation state -2, except peroxides  $O_2^{2-}$  where is -1 and superoxides  $O_2^{2-}-1/2$ .

e) The oxidation state of the alkali is +1.

f)The oxidation state of the alkaline earth is +2.

g) The oxidation state of fluorine is always -1.

h) The oxidation state of an ionic species is equal to the algebraic sum of the oxidation states of all constituent atoms .

i)The oxidation state of a central atom in a molecule is calculated by difference, so that the total charge of the molecule is zero.

j) In organic compounds must take into account that :

j1) N is assigned the oxidation state -1, -2 or -3 as is attached to a carbon by a single,double or triple bond.

j2) At halogens are assigned an oxidation state-1.

j3) At alkyl or aryl groups are assigned an oxidation state +1.

j4)At Oxygen is assigned an oxidation state -2 if carbon is bonded by a double bond and -1 if bound by a single bond.

## **REDOX EQUATION ADJUSTMENT**

There are two methods for adjusting redox equations:

1)Oxidation number method.

2)Ion electron method .

In the reaction set there the law of conservation of mass, conservation of electrical charges and the number of electrons transferred by the reducing agent are equals the number of electrons captured by the oxidizing agent.

a) Method of change in the oxidation number.

a1) Assign oxidation numbers to atoms who change its oxidation number.

a2) Write the half-reactions with the atoms change their oxidation state, adjusting with electrons the load on both sides.

a3) Multiply each half-reaction by a number that makes the electrons given by the atoms are oxidized are equal to the electrons gained by atoms are reduced.

a4) Add the half reactions by removing electrons.

a5) Have in the molecular equation exponents obtained in the previous step.

a6) Properly adjust other substances involved in the reaction. Adjust the following reactions:

$$Cu + HNO_3 \neq Cu(NO_3)_2 + NO + H_2O$$

 $3(Cu^{0} - 2e^{-} \rightleftharpoons Cu^{2+}) \text{ oxid}$   $2(N^{5+} + 3e^{-} \rightleftharpoons N^{2+}) \text{ red}$   $3Cu^{0} + 2N^{5+} \rightleftharpoons 3Cu^{2+} + 2N^{2+}$   $3Cu + 8HNO_{3} \rightleftharpoons 3Cu(NO_{3})_{2} + 2NO + 4H_{2}O$ 

$$Sb_2S_5 + HNO_3 \rightleftharpoons HSbO_3 + S + NO + H_2O_3$$

 $\mathrm{CH_3CH_2OH} + \mathrm{KMnO_4} + \mathrm{H_2SO_4} \rightleftharpoons \mathrm{CH_3COOH} + \mathrm{K_2SO_4} + \mathrm{MnSO_4} + \mathrm{H_2O}$ 

$$P_4 + KOH + H_2O \rightleftharpoons KH_2PO_2 + PH_3$$

**b**)Ion - electron method.

- b1) Assign oxidation numbers to atoms who change its oxidation number.
- b2) Write the half-reactions for oxidizing and reducing species.

If the reaction takes place in acid medium, add  $H_2O$  to H + poor member of the equation. If the reaction takes place in basic medium, for each O is added excess 1  $H_2O$  that member and On the other 2HO<sup>-</sup>. Then, adjust with electron the electric charge in both members.

b3) Multiply each half-reaction by a number that makes the electrons given by the atoms are oxidized are equal to the electrons gained by atoms are reduced.

b4) Add the half reactions by removing electrons and common species in both members.

b5) Have in the molecular equation exponents obtained in the previous step. Adjust the following reactions:

$$K_2Cr_2O_7 + HI + HClO_4 \rightleftharpoons Cr(ClO_4)_3 + I_2 + H_2O + KClO_4$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \Rightarrow 2Cr^{3+} + 7H_2O$$
 (Reducción)  
3 (2 $\Gamma \Rightarrow I_2 + 2e^-$ ) (Oxidación)  
 $K_2Cr_2O_7 + 6HI + 8HClO_4 \Rightarrow 2Cr(ClO_4)_3 + 3I_2 + 7H_2O + 2KClO_4$ 

The reaction between potassium permanganate and potassium iodide in the presence of aqueous potassium hydroxide leads to the formation of potassium manganate, potassium iodate and water. Adjust the reaction:

$$MnO_4^- + I^- + HO^- \rightleftharpoons MnO_4^{2^-} + IO_3^- + 3H_2O$$
  

$$6(MnO_4^- + 1e^- \rightleftharpoons MnO_4^{2^-}) \text{ (red)}$$
  

$$I^+ 6HO^- \rightleftharpoons IO_3^- + 3H_2O + 6e^- (Ox)$$
  

$$6MnO_4^- + I^- + 6HO^- \rightleftharpoons 6MnO_4^{2^-} + IO_3^- + 3H_2O$$

In the preparation of oxidizing and reducing agents we must take into account the number of electrons exchanged each species when we calculate the normaly. For example,  $MnO_4^-$  acts as the oxidant in acid medium reduced to manganous salt while in neutral medium does to manganese dioxide:

 $Mn^{7+} + 5e^{-} \le Mn^{2+}$  (acid medium)  $Mn^{7+} + 3e^{-} \le Mn^{4+}$ (basic or neutral medium)

# LAWS TO CONSIDER NERNST EQUATION

The Nernst equation is useful to find the reduction potential in the electrodes under different standards. The equation has the name of who made it, the physical chemist German Walther Hermann Nernst .

The equation has the following form :

 $\mathbf{E} = \mathbf{E}^{\mathbf{o}} - \mathbf{RT} / \mathbf{nF} \cdot \mathbf{ln} (\mathbf{Q})$ 

Where E, refers to the electrode potential.

E°= potential under standard conditions.

R= gas constant (Na x Kb).

T= absolute temperature ( in degrees).

n= number of moles that participate in the reaction.

F= Faraday constant( 96500 C/mol, aprox.) qe x Na charge within a mol of e-.

Q= reaction quotient.

#### FARADAY'S LAW

Faraday's Law or electromagnetic induction, states that the induced voltage in a closed circuit is directly proportional to the rate of change over time the magnetic flux which passes through a given surfacewith the circuit making edge.

That is to say, the electric force induced in any closed circuit is equal to the time rate of electric flux enclosed by the circuit.

Where:

$$I = q/t$$

-q is the electric flux (C).

-t is the time in seconds.

In the case at hand, the variations in the electric flux causes an electromotive force, maintaining a potential difference between two points of an open circuit.

An important application is the creation of electric motors, that convert electrical energy into mechanical, thus differentiating chemical engines, which transform the heating value of fuel into mechanical energy. Further, electric motors have higher performance.

## **REDOX IN EVERYDAY LIFE**

Redox reactions are very important in everyday life because without these the development of human life would be impossible, humans through breathing consume oxygen and  $CO_2$  expire and plants through photosynthesis consume  $CO_2$  transforming it into oxygen, combustion serves to cook food they eat, the steel produced is used to build and build their cities, etc.

Oxidation reduction is essential for comfortable living, travel and basic ability to breathe. Reducing oxidation is a form of redox reaction, specifically a process by which oxygen is removed from a compound. The result of an oxidation-reduction reaction is often the heat, but can also create numerous other essential compounds that are required to live.

Some examples are :

#### Automobile fuel

The combustion processes are indeed redox reaction, gasoline that powers the car uses an oxidation process to convert gasoline into energy.

#### Heat your home

The home heating system uses another form of oxidation reduction to generate heat for home.

#### **Photosynthesis**

Plants use photosynthesis to convert carbon dioxide and sunlight into nutrients.

#### Breathing

The natural breathing is the opposite process of photosynthesis, providing the essential oxygen breathing animals.

Because of the importance of these reactions in everyday life we will develop four laboratory practices, which allow to observe these reactions in situations which we encounter daily.

These practices are:

- DETERMINATION OF HYDROGEN PEROXIDE IN COMERCIAL H<sub>2</sub>O<sub>2</sub> AND DIFFERENTIATION OF MANGANESE OXIDATION STATES.
- C VITAMIN QUANTIFICATION.
- STACK WITH COINS.
- DETERMINATION OF THE FARADAY CONSTANT.

# DETERMINATION OF HYDROGEN PEROXIDE IN COMMERCIAL H<sub>2</sub>O<sub>2</sub> AND MANGANESE OXIDATION STATES PART 1:DETERMINATION OF HYDROGEN PEROXIDE IN COMMERCIAL H<sub>2</sub>O<sub>2</sub> INTRODUCTION

## **REDOX TRITATIONS**

The redox titration is very similar to the acid-base titration. It is to adjust the titration reaction to determine the number of moles of oxidant and reducing species react with each other. The equivalent weight of an oxidizing substance or reducing is determined by dividing its molecular mass by the number of e– gained or losts.

At the equivalence point valuation is met:

oxidant equivalents = reducing equivalents

Then:

Vox x Nox = Vred x Nred

Proper selection of the redox indicator requires the construction of the titration curve: potential versus titrant volume. An example is shown in which the curve is constructed simply working with percentages valuation (this method is not always possible).

#### **OBJECTIVE**

Determination of H<sub>2</sub>O<sub>2</sub> content in commercial hydrogen peroxide.

#### **FUNDAMENT**

The hydrogen peroxide is used as a disinfectant. Because its solutions are not stable over a long period of time, it is necessary to assess them. For that, a direct redox titration as permanganometry can be used.

To determine the concentration of hydrogen peroxide in the sample is titrated directly with a previously standardized solution of potassium permanganate.

The concentration of hydrogen peroxide solutions commercial is expressed in volumes, indicating the volumes of oxygen under normal pressure and may be formed by decomposition of hydrogen peroxide contained in a volume of solution.

The reaction of hydrogen peroxide decomposition is the following:

$$2H_2O_2 < ---> O_2(g) + 2H_2O$$

The determination of the concentration of commercial 3% H<sub>2</sub>O<sub>2</sub> (p/v) is a typical case of permanganimetric valuation. The reaction occurs in acid medium (unadjusted ) is:

$$KMnO_4 + H_2SO_4 + H_2O_2 ----> K_2SO_4 + MnSO_4 + O_2 + H_2O_3$$

Adjusted formula:

$$2KMnO_4 + 3H_2SO_4 + 5H_2O_2 ----> K_2SO_4 + 2MnSO_4 + 5O_2 + 8H_2O_2$$

It notes that this is a value between two compounds commonly used as oxidizers, but given the relative nature of these, in this case the KMnO<sub>4</sub> it acts as oxidizer.

We must also remember the habit of expressing the concentration of  $H_2O_2$  in volumes of oxygen. What is expressed is the volume of oxygen formed in C.N. from any of hydrogen peroxide.

## **MATERIALS AND REAGENTS**

Support for bureta, pin, nut. Burette of 100mL. Erlenmeyer flask. Pettes and volumetric flasks. KMnO<sub>4</sub> factored . H<sub>2</sub>SO<sub>4</sub>. H<sub>2</sub>O<sub>2</sub> comercial.

## **EXPERIMENTAL PROCEDURE**

Take 4mL of commercial  $H_2O_2$  and make up to 100mL with distilled water. Then, take 15 mL and add 5 mL of  $H_2SO_4$  1:1, all in an Erlenmeyer will be subject to valuation KMnO<sub>4</sub> 0'01M. The evaluation will be completed when the pink color persists at least 30". Keep  $H_2O_2$  protected from light in order to avoid loss of  $O_2$  by decomposition.

#### TREATMENT OF RESULTS

To obtain the  $H_2O_2$  concentration in commercial hydrogen peroxide is used the following formula:

 $MH_2O_2 = ((5/2)xMMnO_4xVMnO_4)/(VH_2O_2)$ 

Mcommercial  $H_2O_2=MH_2O_2 x(100/4)$ 

## **RESULTS**

V KMnO <sub>4</sub> (mL)	M H <sub>2</sub> O <sub>2</sub> (mol/L)
14	0.0230
13	0.0216
13/05/16	0.0225

 $MH_2O_2 = 0.0225 \text{ moles/L } H_2O_2$ 

Mcommercial  $H_2O_2=0.5625$  moles/L commercial  $H_2O_2$ 

O<sub>2</sub> Volumes:

 $0.5625\ moles/L\ commercialH_2O_2\ x\ (0.5\ moles\ O_2/\ mol\ H_2O_2)\ x\ 22.4L=V\ of\ O_2$ 

V of  $O_2 = 6.3$ 

The results are as expected, the hydrogen peroxide is in low concentrations in many household products.

#### PART 2: MANGANESE OXIDATION STATES

#### **OBJECTIVE**

Distinguish some oxidation states of manganese by oxidation-reduction reactions, using hydrogen peroxide as a reductant.

## THEORETICAL FUNDAMENT

The redox titrations have their foundation in the quantitative study of an oxidation - reduction using the valuation procedure.

In the oxidation-reduction reactions exist a electron transfer so that there is a chemical species yielding electrons (oxidizes) and another that captures (reduced); The number of electrons transferred must be equal to the electron captured and therefore electronic the total balance must be zero in the reaction. Electron withdrawing species acts as a reductant and electron withdrawing species serves as the oxidizer.

As in all volumetric determination, in redox titrations is necessary that the stoichiometry of the reaction is well established, the reaction must be fast, and which have the means to determine an end point as close to the equivalence point as possible.

Moreover also the degree of accuracy with which the concentration of the solution of titrant is known is of great significance. Thus, whenever possible a substance that is primary standard should be used. The primary type substances most commonly used in oxidation-reduction titrations are Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, KBrO<sub>3</sub>, KlO<sub>3</sub>, I<sub>2</sub> y K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>.

Potassium permanganate is a strong oxidant and their solutions are often used to assess reducing substances. Nevertheless, potassium permanganate is not primary standard (it contains small amounts of  $MnO_2$  and reacts slowly with water) and to be used as titrant agent dissolution concentration must be determined, not by direct weighing of the solid, but by prior titration with a standard solution (Ex.:  $Na_2C_2O_4$ ).

Since the equivalence point of an assessment is reached when the amount of added titrant is chemically equivalent to the amount of reagent valued and we know the volumes of spent solution, expressing concentrations in Normality (number of equivalents /L) calculations are very simple :

Being Vox= mL of oxidizing solution, Nox its normality and Vred = mL of reducing solution, Nred its normality.

Nevertheless, the IUPAC (International Union of Pure and Applied Chemistry) advises against the use of Normality as concentration unit, because of the ambiguity of use.

For example, a solution 1 M of KMnO<sub>4</sub> has a concentration 5 N when it is acting as an oxidant in acidic medium (MnO<sub>4</sub>- It is reduced to  $Mn^{2+}$ , accepting 5 e-) and 3 N when it is acting as an oxidant in neutral or slightly alkaline (MnO<sub>4</sub>- s is reduced to MnO<sub>2</sub>, accepting 3 e ). In contrast to the ambiguity of Normalcy, the solution is 1 M, whatever the use made of it. So, despite the simplicity of the above expression, in practice we use as the molarity concentration unit and reasonably we use the stoichiometry of the reaction. This type of reasoning is the one that should be used for any redox titration , adjusted for the corresponding reaction.

In practice some states of oxidation of manganese be distinguished. Given the electronic configuration of the outer layer,  $3d^54s^2$ , manganese can have six possible oxidation states: +2, +3, +4, +5, +6 y +7. The KMnO<sub>4</sub>, containing Mn(VII), is an oxidizing agent and the reduction products depend on the pH of the medium.

## **MATERIALS AND REAGENTS**

MATERIALS	REAGENTS	
Burette of 50 mL	$H_2SO_4$ 3 M	
Support	H <sub>2</sub> O <sub>2</sub> 3%	
2 Erlenmeyerflasks of 250 mL	NaOH 6 M	
Pipettes of 5, 10 y 25 mL	KMnO <sub>4</sub> 0,01 M	
Test tubes and rack Dropper		
6 Beakers of 100 mL		
Glass rod		
Burette of 100 mL		

## **EXPERIMENTAL PROCEDURE**

(a white background is advisable if you want to see clearly the color changes) Isolate some oxidation states of manganese by oxidation-reduction reactions.

1º Have four glasses and label with the letters A, B, C and D.

 $2^{\circ}$  Add 5 mL of the solution of KMnO<sub>4</sub> to the glasses labeled as A, C and D and 1 mL to the B glass.

3° Glass A: Add 5 mL of water . A characteristic purple colouration is observed in the ion Mn(VII).

 $4^{\circ}$  Glass C: Add 5 mL water and 1 drop of NaOH 6 M. Add dropwise , 3% H<sub>2</sub>O<sub>2</sub> stirring after addition of each drop until a brown precipitate of oxide of Mn(IV).

 $5^{\circ}$  Glass D: Add 3 mL of H<sub>2</sub>O and 2 mL of 3 M H<sub>2</sub>SO<sub>4</sub> and stir. Add dropwise 3% H<sub>2</sub>O<sub>2</sub> stirring after addition of each drop until a pink colouration of Mn(III).

 $6^{\circ}$  Glass E: Add 3 mL of H<sub>2</sub>O and 2 mL of 3 M H<sub>2</sub>SO<sub>4</sub> and stir. Add dropwise , 3% H<sub>2</sub>O<sub>2</sub> stirring after addition of each drop until the solution is almost colorless, indicating the presence of Mn(II) ion.

## **QUESTIONS**

1. Write the half-reactions and reactions that take place in the standardization of permanganate and in the determination of hydrogen peroxide.

2. Why the reaction medium must be acid?

3. What redox indicator endpoint is used in these valuations ? ¿Could you use another indicator?

5.Enter and set the reactions taking place in each of the glasses (A-D).

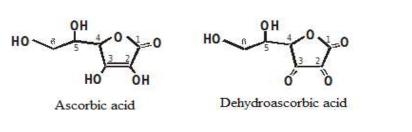
# **C VITAMIN QUANTIFICATION**

## **INTRODUCTION**

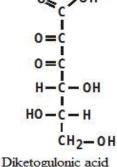
The C vitamin, or ascorbic acid, is a water soluble compound with 6 carbon atoms linked to glucose. Its main biological role is to act as a cofactor in various enzymatic reactions which occur in the body. Ascorbic acid acts as a coenzyme of proline and lysine hydroxylases, which are responsible for hydroxylate lysine and proline in the protocollagen, necessary modification in order to form crosslinks to form collagen fibrils. In this way, the C Vitamin is important in maintaining normal tissue, for wound healing and bone formation, since the bone matrix contains collagen organic. In its capacity as reducing agent, ascorbic acid has other important properties, which appear to be non-enzymatic. For example, it helps in iron absorption by reducing it to its ferrous state in the stomach protects Vitamin A, vitamin E and some B vitamins from oxidation; Finally, Vitamin C is a biological antioxidant that protects the body from oxidative stress caused by reactive oxygen species.

Most symptoms of vitamin C deficiency can be related directly to their metabolic roles. Symptoms of mild deficiency of vitamin C are ease to wounds, due to increased capillary fragility. Scurvy is associated with a decrease in the ability to heal wounds, osteoporosis, bleeding and anemia.

Vitamin C is found mainly in plant foods and can occur in two interconvertible chemical forms: ascorbic acid (reduced form) and dehydroascorbic acid (oxidized form), both being biologically functional forms and maintained at physiological balance. If dehydroascorbic acid is hydrated becomes diketogulonic acid, biologically inactive, this being irreversible transformation. This hydration occurs spontaneously in neutral or alkaline solution.



Biologically active forms of vitamin C



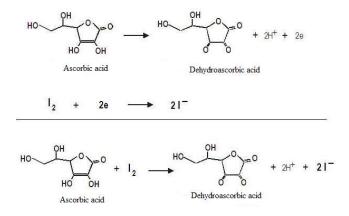
Vitamin C or ascorbic acid is an essential nutrient for many primates, a small number of other mammals (guinea pigs and bats for example), a few species of birds and some fish. The man specifically, lacks the enzyme that catalyzes the terminal step of the synthesis of ascorbic acid, the gulonolactone oxidase, so he must purchase through food, this being the reason why in humans and other species the ascorbic acid vitamin becomes important.

Vitamin C is an unstable compound, due to the ease with which oxidizes and hydrates. It is easily destroyed in processing and preserving food, so it is used as an indicator of vitamin loss during food processing and storage . On the other hand, heat and metal cations destroy C vitamin. Foods such as citrus fruits, kiwi, strawberries, broccoli, lettuce, among others, are a natural source of C vitamin, and its content depends on the species, geographical area in which they are grown, storage conditions once collected and the state of maturation.

Vitamin C can be recognized by methylene blue. This dye is blue when is oxidized and colorless when is reduced. On the other hand, the chromatography and volumetric titration redox methods are used to quantify the vitamin C content of food . The high pressure liquid chromatography is the accuracy method most used provide high of the results. Nevertheless to HPLC technique is expensive, therefore this practice will determine the content of vitamin C in fruit, in beverages prepared or vitamin complexes by volumetric titration oxide reduction.

In this practice we will determine the content of vitamin C by redox titration, Vitamin C is reducing character and we use a solution of iodine as an oxidizing agent which is the titrant pattern.

For a substance that oxidizes another must be reduced and vice versa. Thus, when the reduced ascorbic acid we add iodine is reduced to iodide this at the cost of ascorbic acid oxidizes.



Valuations in which intervenes iodine as oxidizing agent are called yodimetries. Since the reaction between iodine and ascorbic acid has a 1: 1 stoichiometry, at the end point of the titration the reduced the number of iodine moles is equivalent to the moles of oxidized ascorbic acid. Importantly, the total reducing capacity of solution is determined by this method. Thus, if the solution contains other reducing substances in addition to the ascorbic acid, the consumed volume of oxidizing solution (iodine) may be increased , and therefore, the content of ascorbic acid overestimated .

Further, it must take into account that vitamin C is easily oxidized by air, so, for solutions containing vitamin C should be prepared immediately before being assessed, in order to obtain reliable results.

The determination of vitamin C is possible by using a known excess iodine to complete reaction, and then titrating the excess iodine with thiosulfate .The thiosulfate ion has a moderately strong reducing character, which it has been widely used to determine oxidizing agents .In this case, the excess of iodine oxidizes thiosulfate ion in tetrathionate, while the iodide is reduced.

$$I_3 + 2S_2O_3 + S_4O_6 + S_4$$

In the endpoint detection of the iodine valuations, the same reagent can serve as an indicator, provided that the test solution is colorless, as the color of iodine can be perceived in a concentration equivalent to less than one drop of solution 0.05M in 100 mL.

The liberated iodine is then titrated with thiosulfate solution. The overall stoichiometry is:

1 mol de  $IO_3^-=3$  moles de  $I_2=6$  moles de  $S_2O_3^{-2-3}$ 

## **MATERIALS AND REAGENTS**

MATERIALS	REAGENTS		
Burette of 25 mL.	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 0.1M disolution.		
3 volumetric flasks of 250 mL.	Vitamin C pills, orange or lemon and		
	commercial juice.		
3 Erlenmeyers of 250 mL.	KIO <sub>3</sub> 0.01M disolution.		
Beakers of 100, 250 y 500 mL.	H <sub>2</sub> SO <sub>4</sub> 0.5M disolution.		
Volumetric pipettes of 25 y 2 mL.	Solid potassium iodide.		
Test pieces of 25 y 10 mL.			

## **REAGENT PREPARATION**

## Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 0.1M disolution

6.25 g of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 5H<sub>2</sub>O dissolved in water in a beaker .It is subsequently transferred to a 250 mL flask, diluted with water up to the mark. Store in a topaz flask in order to protect the solution from light.

## KIO<sub>3</sub> 0.01M solution

Transfer 0,22g KIO<sub>3</sub> to a volumetric flask of 250 mL.

## **EXPERIMENTAL PROCEDURE**

Vitamin C pill (180 mg) is poured into an Erlenmeyer flask and add 200 ml of water.

10 mL of  $0.5M H_2SO_4$  solution are added.

Add 1g solid KI, stir the mixture until complete disolution1.

Pour 25 mL of 0.01M KIO<sub>3</sub>.

The thiosulphate solution is added until the blue color disappears.

We note the volume of solution consumed thiosulfate.

The assessment is repeated twice.

## **RESULTS TREATMENT**

Calculate the amount of vitamin C in the sample (eg juice) in g / L using the following formula: mg\_C= (V(IO<sub>3</sub>) x [IO<sub>3</sub>] x 3 - V(S<sub>2</sub>O<sub>3</sub>) x [S<sub>2</sub>O<sub>3</sub>] x1/2) x 176.13 x 1000

## **RESULTS**

#### 180/8=(25 x 0.001 x 3 - V x 0.001 x 0.1 x 0.5) x 176.13 x 1000

#### v=12.4451 mL

$Na_2S_2O_3L$	C vitamin (mg/pill)
0,0125	176.13
0,0123	190.22
0,0110	169.08

C vitamin(g/L) = 178.47g/pill

The results are as expected.

Pay special attention to adding the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> since a volume excess may cause an error.

We found that this method is simple, cheap and effective within acceptable ranges error.

## **BATTERY WITH COINS**

## **INTRODUCTION**

Batteries are generators of electron, with a limited life by depleting substances redox reagents and that as a voltaic cell produces current, in which chemicals are consumed. The primary voltaic cells can not be recharged. Once the chemicals have been consumed, it is not possible subsequent chemical reaction. The electrolytes and the electrodes can not be recharged by reversing the current flow through the cell using an external DC source. The most familiar examples of primary voltaic cells are the " dry cells ", which it is the common batteries used as an energy source in flashlights and other small appliances. Although the Zn -Cu cell was used as a current generator for a long time, it was shown to be more effective and practical the batteries without liquid solution inside. The best known is the dry Leclanché cell. The container of the stack (Zn) acts as one of the electrodes. The other electrode is an inert carbon rod in the center of the cell . The zinc container is coated with porous paper to separate it from the other cell materials. The space between the electrodes is filled with a moist mixture of ammonium chloride (NH<sub>4</sub>Cl), manganese oxide (IV), (MnO<sub>2</sub>), zinc chloride (ZnCl<sub>2</sub>), and a porous inert filter. Dry batteries are sealed to prevent moisture loss by evaporation.

Once the electrodes are connected externally, metallic Zn is oxidized to  $Zn^{2+}$ , and electrons flow along the vessel until the external circuit. Thus, the zinc electrode is the anode (negative electrode).

 $Zn \leftrightarrow Zn_{2^+} + 2e$ - (anodic oxidation)  $2NH_4^+ + 2e$ -  $\leftrightarrow 2NH_3 + H_2$  (cathodic reduction, graphite rod).

Global Reaction Cell:  $Zn + 2NH_{4^+} \leftrightarrow Zn^{2+} + 2NH_3(g) + H_2(g)$  (Ecell= 1.6 v)

It is noted that two gases are produced in this reaction. As  $H_2$  is formed, it is oxidized by  $MnO_2$  of the Cell, avoiding collect the cathode gas, which would stop the reaction:

 $H_2 + 2MnO_2 \leftrightarrow 2MnO(OH) \leftrightarrow Mn_2O_3 (s) + H_2O (l)$ 

The ammonia produced at the cathode is combined with zinc ions and forms a soluble compound containing the ion complex tetraaminzinc(II):

 $Zn^{2+} + 4NH_3 + 2Cl^{-} \leftrightarrow [Zn(NH_3)_4]Cl_2$ 

This reaction prevents the polarization due to accumulation of ammonia, and prevents the concentration of  $Zn^{2+}$  increases substantially, which would reduce cell potential.

The potential generated by this battery has an initial value of 1.5 volts and its duration depends mainly on the quality and quantity of  $MnO_2$ .

Dry alkaline batteries are similar to the dry Leclanché cell, except that the electrolyte is basic because it contains KOH, the inner surface of the container of Zn is roughened, which provides greater surface area, and they have a longer life than ordinary dry batteries, supporting best the continued use. The anode is Zn( or steel) and cathode steel also.

The voltage of an alkaline battery is about 1.5 volts and during discharge, the reactions that take place are:

Anode:  $Zn(s) + 2(OH-) (aq) \leftrightarrow Zn(OH)_2(s) + 2e- \leftrightarrow ZnO(s) + H_2O(l) + 2e-$ 

Cathode:  $2MnO_2(s) + 2H_2O(l) + 2e \leftrightarrow 2MnO(OH)(s) + 2(OH-) (aq) ' Mn_2O_3 + H_2O + 2OH-$ Global:  $Zn (s) + 2MnO_2(s) + 2H2O(l) \leftrightarrow Zn(OH)_2(s) + 2MnO(OH)(s)$ 

Another type of primary cells are the button batteries, very small and able to release a large amount of current. They are 1 cm in diameter and 4 mm of thick. They consist of layers separated by plastic fibers and the most commonly used oxidizing agents are HgO y Ag<sub>2</sub>O.

Anode:powder amalgamated Zn, impregnated in KOH as in the alkaline battery.

The reaction is the same:

 $Zn(s) + 2(OH-) (aq) \leftrightarrow Zn(OH)_2(s) + 2e- \leftrightarrow ZnO(s) + H_2O(l) + 2e-$ 

Cathode: The compartment is nickel-plated steel and it is separated from the anode by the central part with a fiber nylon. Ag<sub>2</sub>O or HgO gets mixed with graphite powder and soaked in KOH conc.

HgO + 2e- + H2O  $\leftrightarrow$  Hg<sup>0</sup> + 2OH- (1.35 v) Ag<sub>2</sub>O + 2e- + H<sub>2</sub>O  $\leftrightarrow$  2Ag<sup>0</sup> + 2OH- (1.40 v)

There are other button cells are known as lithium batteries because they use Li as reductant instead of the Zn. The main problem of these cells is that this metal Li can not contact with air or water, so that they carry organic electrolytes drivers which are much less current than aqueous electrolytes. They are smaller batteries than those of HgO or Ag<sub>2</sub>O and the anodic reaction is based on the oxidation of lithium : Li  $\rightarrow$  Li<sup>+</sup> + 1e- .The cathode is a Pb<sub>3</sub>O<sub>4</sub>, PbO, CuO, MnO<sub>2</sub> mixed with powdered graphite. The electrolyte is organic type, generally mixtures of esters and ethers.

#### **FUNDAMENT**

In this practice will carry out an experiment to generate electricity. Using only a few coins and a little vinegar can create a stack by a redox reaction.

Vinegar in contact with copper produces an oxidation-reduction reaction, generating a movement electron, creating an electric current.

In our stack, the copper is the reducer of vinegar, which oxidizes the currency making this yield electrons to vinegar. It will also be used tinfoil, whose function is to be the driver.

## **MATERIALS AND REAGENTS**

- Coins (Cu).
- Paperboard.
- Vinegar.
- Foil.
- Copper wire.
- A glass.
- Scotch tape.
- Voltmeter.
- Led.

## EXPERIMENTAL PROCEDURE

First we cut a few square pieces of cardboard slightly smaller than the currency to be used and we introduce them in a glass of vinegar for 10 min. Then, also cut into square pieces of aluminum foil.

Then the items are placed in the following order:

- 1- Scotch tape.
- 2- One end of the copper cable.
- 3- A coin.
- 4- A square of cardboard with vinegar.
- 5- A square of foil.
- 6- Other end of the copper cable.

Finally, the tape is wound around to hold it all right . The problem is that with a single currency the voltage is too small to hold all well and we could not get to illuminate an 3V LED.

So for it about 8 coins are stacked in the order described above to achieve sufficient voltage. Once this is done, the cable which is on the side of the coin is the positive side, while it is in contact with the foil will be the negative side. If we connect the LED note that works perfectly.

## **QUESTIONS**

1. Why in some cases the voltmeter has negative value ?

2. Explain why the different electrodes must be connected by reversing polarity in order to obtain more power supply. What would it happen if the polarities are aligned instead?

3. Detail what happens .Is power being produced?

4.Describe what happens to the battery potential along time.

## **DETERMINATION OF THE FARADAY CONSTANT**

## **INTRODUCTION**

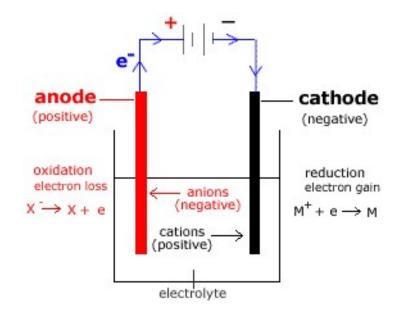
In the early nineteenth century, Faraday, english scientist who discovered that aqueous solutions of certain substances leading electrical current, while pure water and other solutions not led. Faraday called electrolytes substances whose aqueous solutions conduct current and non-electrolyte substances that are not decomposed by electricity.

In1830, Faraday began his experiments to determine the principles that relate the amount of material which it is removed or deposited during electrolysis, it being understood that the longer the current which is applied to the solution, bigger is the amount of separated ions, while the mass that accumulates in the electrode should be equal to the atomic mass of the electrode, but inversely proportional in the valences between the ion and the electrode.

## **CELL AND ELECTROLYTIC PROCESS**

Electrolysis is the process where a given amount of electrical energy used by passing through a substance in solution or melted causing chemical changes.

The electrolysis is carried out in a device known as electrolytic cell.



Electrolytic cells consist of a container; which contains the electrolyte and must be of a material that does not react with it, two electrodes (one functions as a cathode and the other as anode) which allow the passage of electric current, a voltage source and an ammeter.

In the above figure the electrolytic process shown sodium chloride (NaCI), the substance is found in liquid form, particles that form are as ions.

When electrons flow through the electrolyte, cations (Na<sup>+</sup>) are attracted by the cathode effected a reduction reaction. For this, the cathode is defined as the electrode where reduction takes place.

Both, anions (Cl<sup>-</sup>) are directed towards the anode, where oxidation occurs. The anode will is defined as the electrode where oxidation occurs.

 $Na^{+} + 1e^{-} > Na^{0}$ 

Anodic reaction (oxidation).

 $2Cl^{-} - 2e^{-} - ---> Cl_{2}^{0}$ 

The laws which governing electrolytic processes were established by Michael Faraday. The first of its laws reads "The amount of a substance released or placed in an electrolytic cell is proportional to the amount of electricity passed through the solution". This can be represented mathematically as follows:

#### m = e Q

An electrolytic cell consists of a conductive liquid electrolyte and two electrodes with similar composition. The cell as such does not serve as power source, but it can conduct current from an external source called electrolytic action. It is used in electro deposition, electro training, gas production and performing many industrial processes. If due to current flow electrodes become unequal, it is possible that a voltaic action occurs.

#### **FUNDAMENT**

Our work project consists in the assembly of an electrolytic cell for the reduction of  $H^+$  ions presents in  $H_2SO_4$  and oxidation of copper plate.

This reaction is not spontaneous because the normal reduction potential of  $(2H^+ / H_2)$  is 0,0 V and normal reduction potential of  $(Cu^{2+}/Cu)$  is 0,34 V. Thus, we would obtain a total potential for the reaction discussed above of - 0,34 V. To promote this reaction take an external source of electrons, as is a power outlet, with an adapter to convert the current AC to DC.

#### **CONSIDERATIONS**

To obtain these experimental data, we must bear in mind that the amount of deposited or consumed substance in one or other electrode is proportional to the amount of electrons that have passed through the electrolytic cell.

We must also address how we can relate the experimental data with the number of moles transformed.

The number transformed moles is:

n transformed = Q / Q to transform one mol (1)

where Q = I x t y Q to transform one mol = moles of e- in the cell x NA x e- (2)

Faraday's constant is equal to NA x e- thus substituting in the expression (1) the consideration (2), we get the following:

n transformed = (I x t / m e - in cell x F)

Solving F:

F = (I x t / m e - x n trans)

To determine the Faraday constant Anode:

 $\mathbf{F} = [\mathbf{I} \mathbf{x} \mathbf{t} / 2 \mathbf{x} (\Delta \text{moles})]$ 

being  $\Delta m$  the copper mass difference before and after performing electrolysis.

The other calculation to be performed on the anode is the atomic weight of copper, using the approximate value of Faraday 's constant 96500 C / mol e-:

 $P atm = (F x 2\Delta m / I x t)$ 

On the other hand , to determine the Faraday constant at the cathode:  $F = (I x t / 2 n^{o} moles H_{2})$ The number of moles of hydrogen, working with hydrogen as an ideal gas:  $n = (PH_{2} x VH_{2} / RT)$ 

The volume of hydrogen is a known fact since once started electrolysis hydrogen formed sulfuric acid displaces which we flush the burette. T is the temperature of the laboratory and R the ideal gas constant, 0,082 atm x L / mol K.

The partial pressure of hydrogen, is determined by considering that, the pressure a point within the burette that is right on the surface of the solution when the hydrogen shifted to 0 to acid, It is equal to atmospheric pressure, since it is at the same height.

So, the atmospheric pressure is equal to the sum of the partial pressure of hydrogen over the saturation pressure of water, since we assume an interface at that point. Clearing the partial pressure of hydrogen we find that it is the pressure difference atmosféricay the saturation pressure of water.

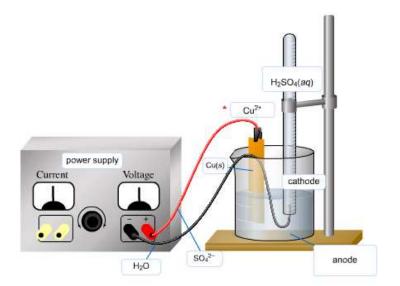
 $PH_2 = PAtm - P sat H_2o$ 

## **MATERIALS AND REAGENTS**

- Glass solutions as an electrolytic cell.
- Ammeter.
- Test tube.
- AC power to DC.
- Copper plate.
- Copper cable.
- Analytical balance.
- Chronometer.
- 1 molar sulfuric acid.

## **EXPERIMENTAL PROCEDURE**

Pour 250 ml of  $H_2SO_4$  in a glass. Enter an upside in that glass cylinder filled with  $H_2SO_4$ . Build the next assembly:



To determine the amount of copper in copper ions transformed, we will weigh the copper plate before and after performing electrolysis (washed with distilled water and perfectly dried).

To determine the volume of hydrogen collected, we observe volume displaced by hydrogen  $H_2SO_4$ .

In this way, having both data collected both volume of hydrogen as the mass of metallic copper that has passed copper II, and knowing, that the amount of substance consumed or deposited in one or the other electrode is proportional to the current that has been passed by the cell, we can determine the Faraday constant.

## **RESULTS**

t(sec)	V(mL)	I(A)
195	4	0,20
240	7	0,23
480	14	0,24
600	17	0,24

N e- calculation

PoCu= 4,726 g

PfCu= 4,679 g

PoCu-PfCu= 0,047 gCu

 $0,047 \text{ g Cu} / 63 \text{ (g/mol)} \text{ Cu}=7,46^{-4} \text{ moles } \text{Cu}^{2+}=\text{ne-}$ 

**Qt** calculation

Ī(A)=0,2275 A

Qt= 0,2275x 600= 136,5C

Faraday constant

Qt/(2xne<sup>-</sup>)=91487,93 C mol^-1

<u>Checking with H<sub>2</sub> moles</u> T=27°C P=640mmHg <u>Moles H<sub>2</sub> calculation</u>  $P_o=640mmHg$   $P_o=640mmHg$   $P_oagua(27°C)=26,739mmHg$   $PH_2=613,216mmHg$ MolesH<sub>2</sub>=PV/RT (613,216mmHg) x (0,022L) / (62,4mmHg/mol K) x (300K) 7,21x10^-4 mol = 7,21x10^-4 ne<sup>-</sup>

Faraday constant

Qt/(2xne<sup>-</sup>)=94140,08 C mol^-1

The results are rather low than expected. We must pay special attention when handling  $H_2SO_4$  as it is corrosive and pay attention to the chronometer because the volume of  $H_2$  varies fast.

## **QUESTIONS**

1)Get the experimental value for the Faraday constant .

2)Write the half-reactions and the reaction of the cell.

## **CONCLUSION**

To finish the work, we can say that we have managed to understand that the oxidation-reduction reactions are important for all of u.

These reactions are important because if it were not them many things would not be possible, although they also have some effect on metals causing deterioration which is what we call oxidation, this is the basis to solve problems of how to avoid this effect.

In short we can say that, the Redox reactions, although we do not see it directly, we live every day with the results of them.

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