

Practical works of the discipline of Inorganic Chemistry

Chemical and Biochemical Engineering course

Work list:

PW1: Introduction to oxidation-reduction and electrochemical cells

PW2: Extraction of lead from lead oxide

PW3: Metals, color of flames and solubility of salts

PW4: Transition metal complexes

PW5: Color and absorption spectra in the visible

PW6: Determination of the percentage of sodium hypochlorite in commercial bleach

Academic year 2017/18, 2nd semester

Unidade Departamental de Engenharias

Escola Superior de Tecnologia de Tomar

Instituto Politécnico de Tomar

Practical Work No.1

Introduction to oxidation-reduction and electrochemical cells

1. Introduction

1.1. Redox reactions

In an electrochemical process the transfer of an electron from one substance to another occurs, hence it is called a redox reaction, since the electron lost in the oxidation of an element is associated with the gain of one electron in the reduction of another element.

Thus, oxidation and reduction occur simultaneously, and there can not be one without the other. However, we may think of a redox process as consisting of two separate reactions, termed half-reactions or partial reactions, one representing the oxidation reaction and the other reducing reaction.

1.2. Electrochemical cells

Typically, a redox reaction occurs when the oxidizing agent is in contact with the reducing agent: there is direct transfer of the electrons from the reducing agent to the oxidant. However, if we physically separate the reducing from the oxidizing agent and establish a conductive external connection, then there is passage of electrons, ie, passage of current: this device is named electrochemical cell or galvanic cell.

An electrochemical cell consists of two electrodes: the electrode where oxidation occurs is called the anode, while the electrode where the reduction occurs is the cathode. The potential difference between the two electrodes is called the electromotive force and is expressed in volts (V), and is due to the oxidation and reduction reactions, called half-cell reactions. A spontaneous redox reaction results in a cell with a positive potential difference. To allow the passage of electric current, it is necessary to close the circuit, which is done by placing a salt bridge to connect the two cells, where it is usually a concentrated solution of a strong electrolyte, which allows ion flow from and to the interior of the solutions.

It is impossible to measure the absolute potential of an electrode, but measuring potential differences between electrodes. Thus, a standard electrode is needed, from which the potential of all others is determined. This electrode is the standard hydrogen electrode, consisting of a platinum bar dipped in a 1 M solution in hydrogen ions where hydrogen gas is bubbled at 1 atm. By convention, the standard reduction potential for the hydrogen ion reduction reaction at 25 °C is zero.

Thus, one can establish a number of standard reduction potentials measuring greater or lesser tendency of a species to accept electrons. This series is called electrochemical series. In Table I are the standard reduction potentials at 25 °C. The standard conditions are the 1 M concentration of the species in solution and the 1 atm pressure for the gaseous species. The more positive the value of E° , the greater the tendency of the species to suffer reduction, and the greater the oxidizing power. The more negative, the greater the tendency to undergo oxidation. By testing different pairs of metals / solution, we can establish an electrochemical series of metals.

2. Experimental procedure

2.1. Redox reactions

2.1.1. Prepare small plates of zinc, copper and lead by wiping them with sandpaper.

2.1.2. Prepare 100 ml of the following solutions:

- a) Lead nitrate 0.1 M, $\text{Pb}(\text{NO}_3)_2$
- b) Copper nitrate 0.1 M, $\text{Cu}(\text{NO}_3)_2$
- c) Zinc nitrate 0.1 M, $\text{Zn}(\text{NO}_3)_2$
- d) Silver nitrate 0.1 M, AgNO_3 .

2.1.3. Place a small portion of solution a) into three goblets, and dip into each of them a zinc, lead, and copper plate.

2.1.4. Observe whether or not there is a reaction in each case, referring to the appearance of the metal and the solution.

2.1.5. Repeat for the remaining solutions, taking care to clean and sand the metal plates before immersing them in the solutions.

2.2. Electrochemical cells

2.2.1. Place the 0.1 M copper nitrate solution in a 100 ml goblet, and immerse into the solution a pre-cleaned copper foil. Connect the copper electrode to the positive terminal of a voltmeter.

2.2.2. Place the 0.1 M zinc nitrate solution in a similar beaker and insert the zinc bar. Connect this electrode to the negative terminal of the voltmeter.

2.2.3. Establish contact between the two solutions with a salt bridge, read the voltage and record it.

2.2.4. Repeat the procedure with the 0.1M lead nitrate solution.

2.2.5. After finishing the work, wash and store all the laboratory material and store the solutions in a glass bottle for later use.

3. Bibliography

- 3.1. M. L. S. S. Gonçalves, *Métodos Instrumentais para Análise de Soluções – Análise Quantitativa*, 4ª Edição, Fundação Calouste Gulbenkian, 2001
- 3.2. R. Chang, *Química*, 5ª Edição, McGraw-Hill, Lisboa, 1995
- 3.3. A. M. O. Brett e C. A. M. A. Brett, *Electroquímica: Princípios, Métodos e Aplicações*, Almedina, 1996

Laboratory sheet no. 1: Introduction to oxidation-reduction and electrochemical cells

Class: Group: Date://

4. Previous calculations**4.1. Solution of $Pb(NO_3)_2$**

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M $Pb(NO_3)_2$:Mass of $Pb(NO_3)_2$: to weigh weighted:**4.2. Solution of $Cu(NO_3)_2$**

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M $Cu(NO_3)_2$:Mass of $Cu(NO_3)_2$: to weigh weighted:**4.3. Solution of $Zn(NO_3)_2$**

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M $Zn(NO_3)_2$:Mass of $Zn(NO_3)_2$: to weigh weighted:**4.4. Solution of $AgNO_3$**

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M $AgNO_3$:Mass of $AgNO_3$: to weigh weighted:

5. Experimental results

5.1. Redox reactions

Gobelet	Composition	Observations
1	Zn/Pb(NO ₃) ₂	
2	Pb/Pb(NO ₃) ₂	
3	Cu/Pb(NO ₃) ₂	
4	Zn/Cu(NO ₃) ₂	
5	Pb/Cu(NO ₃) ₂	
6	Cu/Cu (NO ₃) ₂	
7	Zn/Zn(NO ₃) ₂	
8	Pb/Zn(NO ₃) ₂	
9	Cu/Zn(NO ₃) ₂	
10	Zn/AgNO ₃	
11	Pb/AgNO ₃	
12	Cu/AgNO ₃	

5.2. Electrochemical cells

Cell	Potential difference / mV
Lead / Zinc	
Lead / Copper	
Zinc / Copper	

6. *Post-laboratory questions*

- 6.1. Write the chemical equations in cases where there was spontaneous reaction.
- 6.2. Sort by decreasing order the reducing character of the various metals studied. Justify.
- 6.3. Write the diagrams of the constructed cells. Explain how they work and discuss the electromotive force values obtained.

Practical Work No. 2

Extraction of lead from lead oxide

1. Introduction

Most metals are obtained from minerals, that is, substances of natural occurrence whose chemical composition varies between certain limits. The ore is thus a mineral deposit having a concentration which allows extraction of the desired metal in an economically feasible manner.

The production of a metal can be carried out by reduction of the metal, which can be carried out in two ways: at high temperature, a metallurgical process designated pyrometallurgy, or by electrolytic reduction.

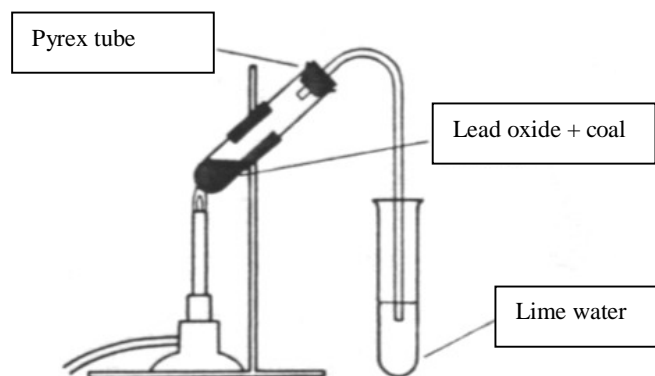
2. Experimental procedure

2.1. Obtaining copper

- 2.1.1. Prepare 100 ml of a 0.1 M solution of copper (II) sulphate.
- 2.1.2. In a gobelet with the solution of the copper sulfate put steel wool.
Observe. Look again at the end of class
- 2.1.3. Repeat the procedure using aluminum foil, using two gobelets. In one of them add sodium chloride. Observe.

2.2. Obtaining lead

- 2.2.1. Weigh about 1g of lead oxide and 1g of coal, both powders.
- 2.2.2. Mix the solids in a mortar until they are evenly homogeneous. Place the mixture in a test tube.
- 2.2.3. Assemble the following experimental scheme according to the figure:



- 2.2.4. Turn on the Bunsen burner and allow the tube to warm up, first gently and then strongly. Observe the tube (or gobelet) with lime water.
- 2.2.5. Allow the tube to cool and pour into a gobelet filled with water. Drag excess coal with water.
- 2.2.6. Observe the gobelet with water and the test tube.

3. Bibliography

- 3.1. R. Chang, *Química*, 5ª edição, McGraw-Hill, 1995

Laboratory sheet no. 2: Extraction of lead from lead oxide

Class: Group: Date://

4. Previous calculations*4.1. Obtaining copper**4.1.1. Solution of CuSO_4*

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M CuSO_4 :Mass of CuSO_4 : to weigh

weighted:

*1.1. Obtaining lead**1.1.1. Lead oxide mass*Mass of Pb_3O_4 : to weigh

weighted:

M Pb_3O_4 :

Moles no.:

1.1.2. Coal Mass

Mass of coal: to weigh

weighted:.....

M C:

Moles no.:

2. Experimental results*2.1. Obtaining copper*

Gobelet	Composition	Observation
1	Steel/ CuSO_4	
2	Al/ CuSO_4	Without NaCl: With NaCl:

2.2. Obtaining lead

Observations concerning the obtaining of lead

3. *Post-laboratory questions*

- 3.1. Interpret reactions that have occurred with copper sulphate.
- 3.2. Interpret the formation of metallic lead.
- 3.3. Why is lime water used?

Practical Work No. 3

Metals, color of flames and solubility of salts

1. Introduction

The color of a flame is affected by the presence of a metal, since the metal atom is excited, and this color is characteristic of each metal, thus constituting a method for detecting the presence of the metal.

On the other hand, the elements of the 2nd Group of the Periodic Table normally have only two oxidation states, 0 and +2, and the chemical properties of these elements are so similar that it is difficult to separate them. Although many of its compounds are poorly soluble, it is possible, by the choice of a suitable anion, to find differences in solubility that allow separation of the cations from these metals. After a systematic study of the relative solubilities of its carbonates, chromates, sulphates, oxalates and hydroxides, a qualitative analysis of an unknown solution containing one or more of these cations can be made.

2. Experimental procedure

2.1. The color of the flames

- 2.1.1. Prepare saturated aqueous solutions of the following salts: sodium chloride, potassium bromide, potassium carbonate, barium nitrate, barium chloride, calcium carbonate, copper (II) sulfate and copper (II) nitrate.
- 2.1.2. Dip one end of each wooden wick into each solution for a few minutes, including the unknown salt solution.
- 2.1.3. Ignite a Bunsen burner and adjust the air input so that the flame is blue.

2.1.4. Insert each wick impregnated with solution into the flame end.

Repeat with a non-impregnated wick.

2.1.5. Decide which salt is the unknown.

2.2. *Relative solubility of some metals*

2.2.1. Prepare the following solutions:

2.2.1.1. Solutions: barium, calcium and magnesium nitrate 0.1M.

2.2.1.2. Reagents: 2M ammonium carbonate, 0.5M potassium chromate, 0.2M ammonium oxalate, 1M ammonium sulfate and 6M ammonia.

2.2.2. In clean test tubes, add 1 ml of each of the nitrate solutions of the metals. Then add 1 ml of each of the reagent solutions.

2.2.3. In the cases where carbonate precipitates form, heat the test tube in a boiling water bath to aid precipitation. Cool the test tube and let the precipitate settle. Decant the supernatant liquid, discard it and add 6M HCl dropwise until complete dissolution.

2.2.4. Use these samples for the flame tests, proceeding in the same manner as in 2.1.

3. *Bibliography*

3.1. R. Chang, *Química*, 5ª Edição, McGraw-Hill, Lisboa, 1995

3.2. R. B. Heslop e H. Jones, *Química Inorgânica*, 2ª Edição, Fundação Calouste Gulbenkian, Lisboa, 1988

3.3. A. L. McClellan, *Química Uma Ciência Experimental*, Fundação Calouste Gulbenkian, Lisboa, 1984

Laboratory sheet no. 3: Metals, color of flames and solubility of salts

Class: Group: Date:/...../.....

4. Previous calculations**4.1. Solution of $Ba(NO_3)_2$**

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Ba(NO_3)_2$:Mass of $Ba(NO_3)_2$: to weigh weighted:**4.2. Solution of $Ca(NO_3)_2$**

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Ca(NO_3)_2$:Mass of $Ca(NO_3)_2$: to weigh weighted:**4.3. Solution of $Mg(NO_3)_2$**

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Mg(NO_3)_2$:Mass of $Mg(NO_3)_2$: to weigh weighted:**4.4. Solution of $(NH_4)_2CO_3$**

Concentration: 2.0 M

Volume: 100 mL

Moles no.:

M $(NH_4)_2CO_3$:Mass of $(NH_4)_2CO_3$: to weigh weighted:

4.5. Solution of K_2CrO_4

Concentration: 0.5 M

Volume: 100 mL

Moles no.:

M K_2CrO_4 :Mass of K_2CrO_4 : to weigh weighted:4.6. Solution of $(NH_4)_2C_2O_4$

Concentration: 0.2 M

Volume: 100 mL

Moles no.:

M $(NH_4)_2C_2O_4$:Mass of $(NH_4)_2C_2O_4$: to weigh weighted:4.7. Solution of $(NH_4)_2SO_4$

Concentration: 1.0 M

Volume: 100 mL

Moles no.:

M $(NH_4)_2SO_4$:Mass of $(NH_4)_2SO_4$: to weigh weighted:5. *Experimental results*5.1. *The color of the flames*

Salt	Flame color
NaCl	
KBr	
K_2CO_3	
$Ba(NO_3)_2$	
$BaCl_2$	
$CaCO_3$	
$CuSO_4$	
$Cu(NO_3)_2$	
unknown	

5.2. Relative solubility of some metals

Ion	CO ₃ ²⁻	CrO ₄ ²⁻	C ₂ O ₄ ²⁻	SO ₄ ²⁻	NH ₃
Ba ²⁺					
Ca ²⁺					
Mg ²⁺					

6. Post-laboratory questions

6.1. Explains why the flames of NaCl and KCl solutions have different colors, while the solutions of Ba(NO₃)₂ and BaCl₂ have equal colors. Predict the color of the LiCl solution. Interpret all the results obtained.

6.2. Draw up a scheme to analyze an unknown solution containing the three metals used in 2.2.

Practical Work No. 4

Transition metal complexes

1. Introduction

A complex ion can be defined as an ion containing a 'central' atom to which other atoms, the ligands, are bound, where in most cases this central atom is a metal element. The forces responsible for these bonds are different from those found in other types of compounds, but are due to the overlapping of orbitals and electron pairing as in the case of simpler compounds. The transition metals establish connections through the d orbitals with various ligands to stabilize their positive charges. We will look at some of these reactions in this work.

2. Experimental procedure

2.1. Precipitation and complexation reactions of various cations with NaOH and NH₃

Add to about 2 ml of each solution of the cations (see table), 2M NaOH solution, dropwise. Observe the colors of the precipitates and verify which ones dissolve in excess of NaOH (amphoteric).

Add to about 2 ml of each solution of the cations, 2M NH₃ solution, dropwise. Observe the colors of the precipitates and verify which ones dissolve in excess of NH₃, to give ammoniacal complexes $[M(NH_3)_n^{x+}]$.

2.2. *Complexation reactions*

2.2.1. *Thiocyanate complex with cobalt (II)*

Add to 2 ml of solution containing Co (II) a little solid ammonium thiocyanate, shake and observe the color change. Then add about 2 ml of ethyl ether, let the phases separate and observe the respective colors.

2.2.2. *Thiocyanate complex with iron (III)*

Add to 2 ml of solution containing Fe (III) a little 0.1 M ammonium thiocyanate solution. Shake and observe the colors.

2.2.3. *Oxalate complex with iron (III)*

Add two drops of ammonium thiocyanate solution to 2 ml of solution containing Fe (III). Add slowly and with stirring to this solution, solid sodium oxalate and observe the final color of the solution. What happens if you add another few drops of ammonium thiocyanate solution?

2.2.4. *EDTA complex with cobalt (II)*

Place about 2 ml of solution containing Co (II) in a test tube and measure the pH with indicator paper. Add 2 ml of EDTA aqueous solution (disodium salt). Measure the pH again. Measure the pH of the EDTA solution used. Observe the color of the solution and compare with the initial value.

2.3. *Catalytic reaction between iodide ions and persulfate ions in aqueous solution*

Place in a 100 ml Erlenmeyer flask 10 ml of 0.2 M potassium iodide solution, 5 ml of 0.01 M sodium thiosulphate solution and 5% starch solution. Then add 20 ml of saturated potassium persulfate solution and immediately start counting with a stopwatch. Write the time when a dark color appears due to iodine formation.

Repeat the previous experiment, but this time by adding 5 drops of a 0.1 M Fe (III) solution to the 20 ml persulfate solution before adding it to the iodide solution. Write the time required for the appearance of the dark color.

Compare the reaction times and interpret the results.

3. Bibliography

3.1. R. Chang, *Química*, 5ª Edição, McGraw-Hill, Lisboa, 1995

3.2. R. B. Heslop e H. Jones, *Química Inorgânica*, 2ª Edição, Fundação Calouste Gulbenkian, Lisboa, 1988

3.3. A. L. McClellan, *Química Uma Ciência Experimental*, Fundação Calouste Gulbenkian, Lisboa, 1984

Laboratory sheet no. 4: Complexos de metais de transição

Class: Group: Date:/...../.....

4. Previous calculations*4.1. Solution of $Cu(NO_3)_2$*

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Cu(NO_3)_2$:Mass of $Cu(NO_3)_2$: to weigh weighted:*4.2. Solution of $Fe(NO_3)_2$*

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Fe(NO_3)_2$:Mass of $Fe(NO_3)_2$: to weigh weighted:*4.3. Solution of $Fe(NO_3)_3$*

Concentration: 0.1 M

Volume: 100 mL

Moles no.:

M $Fe(NO_3)_3$:Mass of $Fe(NO_3)_3$: to weigh weighted:*4.4. Solution of $Co(NO_3)_2$*

Concentração: 0.1 M

Volume: 100 mL

Moles no.:

M $Co(NO_3)_2$:Mass of $Co(NO_3)_2$: to weigh weighted:

4.5. Solution of $Zn(NO_3)_2$

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M $Zn(NO_3)_2$:Mass of $Zn(NO_3)_2$: to weigh weighted:4.6. Solution of NH_4SCN

Concentration: 0.1 M Volume: 50 mL

Moles no.:

M NH_4SCN :Mass of NH_4SCN : to weigh weighted:4.7. Solution of Na_2EDTA

Concentration: 0.1 M Volume: 50 mL

Moles no.:

M Na_2EDTA :Mass of Na_2EDTA : to weigh weighted:4.8. Solution of KI

Concentration: 0.2 M Volume: 200 mL

Moles no.:

M KI :Mass of KI : to weigh weighted:4.9. Solution of $Na_2S_2O_3$

Concentration: 0.01 M Volume: 100 mL

Moles no.:

M $Na_2S_2O_3$:Mass of $Na_2S_2O_3$: to weigh weighted:

5. Experimental results

5.1. Precipitation and complexation reactions of various cations with NaOH and NH₃

Ion	Color	NaOH	Exc. NaOH	NH ₃	Exc. NH ₃
Cu(II)					
Fe(II)					
Fe(III)					
Co(II)					
Zn(II)					

5.2. Complexation reactions

Ion	Color	NH ₄ SCN (s)	Ethyl ether
Co(II)			

Ion	Color	NH ₄ SCN 0.1 M
Fe(III)		

Ion	Color	NH ₄ SCN 0.1 M	Na ₂ C ₂ O ₄ (s)	NH ₄ SCN 0.1 M
Fe(III)				

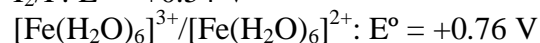
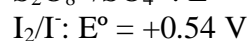
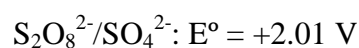
Ion	Color	pH	EDTA (pH = _____)	pH
Co(II)				

5.3. Catalytic reaction between iodide ions and persulfate ions in aqueous solution

	Time /s
With Fe(III)	
Without Fe(III)	

6. Post-laboratory questions

- 6.1. Write the reactions of the ions with NaOH or with NH₃, responsible for the observed color changes (2.1).
- 6.2. Write the reactions a) with acids b) with bases, of the amphoteric hydroxides (2.1).
- 6.3. What are the complexes formed in 2.2? Write their equations. How do you justify what happened when you added ethyl ether (2.2.1.)? What about the pH variation (2.2.4.)?
- 6.4. Write the equation between iodide and persulfate. Does the iron (III) catalyze the reaction? According to the following redox potentials, could you write two reactions that could explain this catalysis?



Practical Work No. 5

Color and absorption spectra in the visible

1. Introduction

If a particular substance absorbs electromagnetic radiation within a particular wavelength range within the visible spectrum, then it has color. In this way, its spectrum will exhibit a maximum absorption band at the wavelength responsible for its coloration.

In cases where a metal can form complexes, the absorption of radiation depends not only on the metal but also on the ligand itself, since it affects the splitting of the energy of the *d* orbitals.

2. Experimental procedure

- 2.1. Prepare 100 ml of each 0.02 M solution in chloride of: Cu(II), Fe(II), Fe(III), Co(II), Zn(II) e Al(III).
- 2.2. Prepare 50 ml of each 0.5 M solution in sodium or potassium iodide, bromide, chloride, hydroxide, fluoride, cyanide, carbonate and oxalate.
- 2.3. Place in twelve test tubes, numbered, about 2 ml of one of the solutions in 2.1.
- 2.4. Add to test tubes 2 to 9 a few drops of each of the solutions in 2.2, according to the results chart. Add to the tube 10 a few drops of 0.5 M NH₃, and to the tubes 11 and 12 a small amount of ethylenediamine and 1,10-phenanthroline, respectively.
- 2.5. Plot the absorption spectra in the spectrophotometer for each of the 12 solutions.
- 2.6. Repeat for other metals.

3. Bibliography

3.1. R. Chang, *Química*, 5ª edição, McGraw-Hill, 1995

3.2. V. M. S. Gil e A. C. Cardoso, *Química*, Almedina Editora

Laboratory sheet no. 5: Color and absorption spectra in the visible

Class: Group: Date://

4. Previous calculations**4.1. Solution of CuCl_2**

Concentration: 0.02 M Volume: 100 mL

Moles no.:

M CuCl_2 :Mass of CuCl_2 : to weigh weighted:**4.2. Solution of FeCl_2**

Concentration: 0.02 M Volume: 100 mL

Moles no.:

M FeCl_2 :Mass of FeCl_2 : a to weigh weighted:**4.3. Solution of FeCl_3**

Concentration: 0.02 M Volume: 100 mL

Moles no.:

M FeCl_3 :Mass of FeCl_3 : to weigh weighted:**4.4. Solution of CoCl_2**

Concentration: 0.1 M Volume: 100 mL

Moles no.:

M CoCl_2 :Mass of CoCl_2 : to weigh weighted:

4.5. Solution of ZnCl₂

Concentration: 0.02 M Volume: 100 mL

Moles no.:

M ZnCl₂ :Mass of ZnCl₂ : to weigh weighted:*4.6. Solution of AlCl₃*

Concentration: 0.02 M Volume: 100 mL

Moles no.:

M AlCl₃ :Mass of AlCl₃ : to weigh weighted:*4.7. Solution of KI*

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KI :

Mass of KI : to weigh weighted:

4.8. Solution of KBr

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KBr :

Mass of KBr : to weigh weighted:

4.9. Solution of KCl

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KCl :

Mass of KCl : to weigh weighted:

4.10. Solution of KOH

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KOH :

Mass of KOH : to weigh weighted:

4.11. Solution of KF

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KF :

Mass of KF : to weigh weighted:

4.12. Solution of KCN

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M KCN :

Mass of KCN : to weigh weighted:

4.13. Solution of K_2CO_3

Concentration: 0.5 M Volume: 50 mL

Moles no.:

M K_2CO_3 :

Mass of K_2CO_3 : to weigh weighted:

4.14. *Solution of $K_2C_2O_4$*

Concentration: 0.5 M

Volume: 50 mL

Moles no.:

M $K_2C_2O_4$:

Mass of $K_2C_2O_4$: to weigh weighted:

5. Experimental results

Metal Ligand	Cu(II)		Fe(II)		Fe(III)		Co(II)		Zn(II)		Al(III)	
	λ_{\max}	Δ	λ_{\max}	Δ	λ_{\max}	Δ	λ_{\max}	Δ	λ_{\max}	Δ	λ_{\max}	Δ
H ₂ O												
I ⁻												
Br ⁻												
Cl ⁻												
OH ⁻												
F ⁻												
CN ⁻												
CO ₃ ²⁻												
C ₂ O ₄ ²⁻												
NH ₃												
en												
pen												

6. Post-laboratory questions

- 6.1. Calculate the value of the splitting of the crystalline field in all cases.
- 6.2. Are the results obtained according to the spectrophotometric series? Justify.
- 6.3. How will the absorption spectrum of chlorophyll be, knowing that the plants are green?

Practical work No. 6

Determination of the percentage of sodium hypochlorite in a commercial bleach

1. Introduction

The bleaching agent in commercial bleaches is sodium hypochlorite, which is produced by the reaction of chlorine with sodium hydroxide. In aqueous solution, the sodium hypochlorite is dissociated into sodium ion and hypochlorite ion, the latter being the bleaching agent, through an oxidation-reduction reaction between the hypochlorite ion (the oxidizing agent) and the colored spot or stain to be removed (reducing agent).

The amount of hypochlorite present in a solution can be determined by the reaction with a known excess amount of a reducing agent, such as the iodide ion. Since I_2 is formed in this process, it can be determined by a titration with the thiosulfate ion, which is a reagent that reacts quantitatively with iodine: as the titration develops, the iodine concentration in the solution decreases and the solution goes from brown to a pale yellow color near the end of the reaction, which is complete when the solution is completely colorless. As this color transition is very difficult to detect, a small amount of starch solution is added when the solution turns pale yellow, whereby free iodine forms a blue complex of intense color. Detection of the end point of the titration is achieved by the addition of thiosulfate ion until the disappearance of the blue color.

2. Experimental procedure

- 2.1. Prepare 200 ml of approximately 0.01 M $Na_2S_2O_3$ aqueous solution, taking note of the mass of sodium thiosulfate used. Prepare 50 ml of 10% KI solution.
- 2.2. To a dry 150-ml Erlenmeyer flask, put 50 ml of bleach to be analyzed (use a measuring cylinder). Cover the Erlenmeyer.
- 2.3. Conveniently prepare a burette with the $Na_2S_2O_3$ titrant solution.
- 2.4. Wash a 10 ml volumetric pipette with several ml of the bleach to be analyzed. Measure with this pipette 10.0 ml of bleach and transfer to a 100 ml volumetric flask. Make up the volume with distilled water.
- 2.5. Transfer to a 250 ml Erlenmeyer flask, 100.0 ml of distilled water and 10.0 ml of 10% KI solution.
- 2.6. Wash a 25 ml volumetric pipette with several ml of the diluted bleach. Measure with this pipette 25.0 ml of diluted bleach and transfer to the Erlenmeyer flask.
- 2.7. Add 4.0 ml of 6M HCl. Stir the solution and immediately start the titration!

- 2.8. Titrate the sample by adding the $\text{Na}_2\text{S}_2\text{O}_3$ solution relatively rapidly until the solution becomes a pale yellow. Add 1 ml of starch solution and add the titrant dropwise until the blue color disappears.
- 2.9. Repeat the test to confirm the result.
- 2.10. Determine the density of the bleach, weighing 10 mL of it.
- 2.11. After the test, thoroughly rinse the burette and other laboratory material.

3. Bibliography

- 3.1. R. Chang, *Química*, 5ª Edição, McGraw-Hill, Lisboa, 1995

Laboratory sheet no. 6: Determination of the percentage of sodium hypochlorite in a commercial bleach

Class: Group: Date:/...../.....

4. Previous calculations

4.1. Solution of $\text{Na}_2\text{S}_2\text{O}_3$

Concentration: 0.01 M

Volume: 200 mL

Moles no.:

M $\text{Na}_2\text{S}_2\text{O}_3$:

Mass of $\text{Na}_2\text{S}_2\text{O}_3$: to weigh weighted:

4.2. Solution of KI

Concentration: 10%

Volume: 50 mL

Density = 1.0 g ml⁻¹

Mass of KI : to weigh weighted:

Test	Titrant volume / ml
1	
2	
Average	

5. Post-laboratory questions

5.1. Write the chemical equations involved in the process.

5.2. Calculate:

- 5.2.1. The average spent volume of the titrant solution.
- 5.2.2. The exact concentration of the titrant solution.
- 5.2.3. The number of moles of thiosulfate ions consumed in the titration.
- 5.2.4. The number of moles of I₂ produced in the titration.
- 5.2.5. The number of moles of hypochlorite ion present in the diluted bleach.
- 5.2.6. The number of moles of hypochlorite ion present in the bleach.
- 5.2.7. The mass of sodium hypochlorite present in the bleach.
- 5.2.8. The mass percentage of sodium hypochlorite in the bleach.

5.3. Refer to the redox titrations and the indicator used in this work.