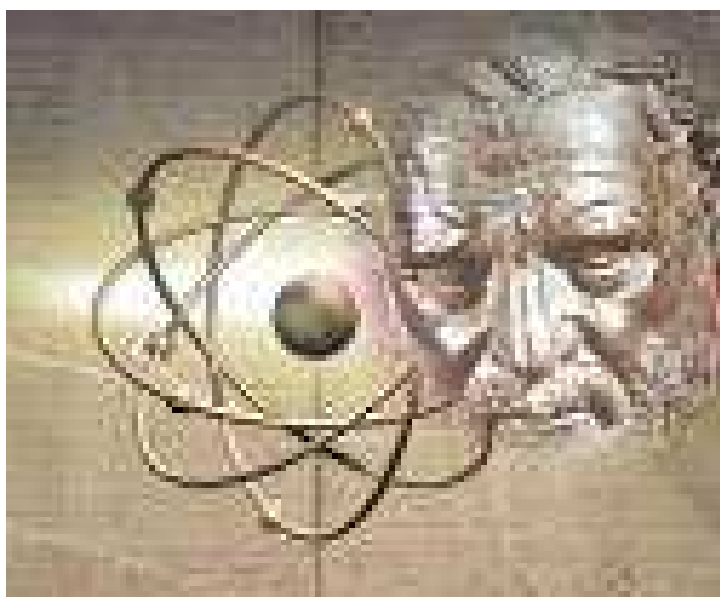




erman ta zabal zazu
Universidad del País Vasco Euskal Herriko Unibertsitatea

EXPERIMENTATION IN INORGANIC CHEMISTRY



2014-2015 ACADEMIC YEAR

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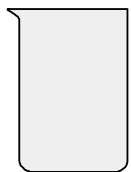
LABORATORY EQUIPMENT

GLASS MATERIAL (per student)

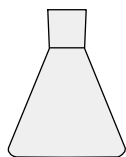
2	100 ml beaker	1	5 ml pipette
2	250 ml beaker	1	Small measuring cylinder
2	400 ml beaker	1	100 ml measuring cylinder
1	Glass rod	1	250 ml measuring cylinder
1	Büchner flask	12	Test tube
1	Büchner funnel	1	Test tube rack
1	Distillation flask	1	Funnel
1	Oil bubbler (Gas washing flask)	4	Watch glass
1	Thistle funnel	1	Erlenmeyer Flask
2	Pasteur pipette	2	Pasteur pipette rubber bulb
1	Test tube holder	1	Evaporating dish

IRON MATERIAL FOR EVERY TWO STUDENTS

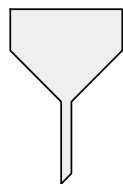
2	Wire gauze
2	Tripods
2	Forceps
2	Burette clamp
2	Support stand
1	Water-bath
1	Brush with wire handle
1	Plastic wash bottle
1	Pipettete-pump



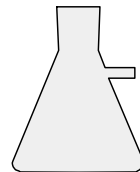
Beaker



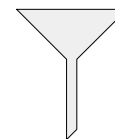
Erlenmeyer flask



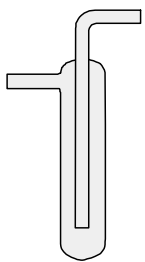
Büchner funnel



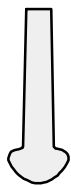
Büchner flask



Funnel



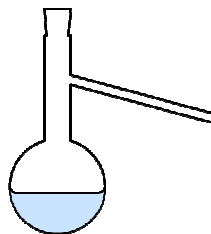
Oil Bubbler



Pestle



Mortar



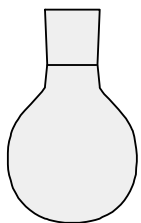
Distillation flask



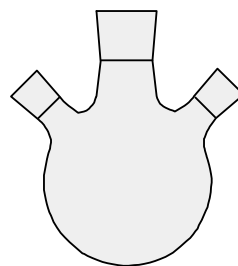
Thistle funnel



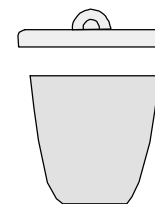
Measuring cylinder



Round bottom flask



Three-Neck round bottom flask



Crucible

HEALTH AND SAFETY

See Appendix 1 for security measures to be taken when working in a chemistry laboratory.

See Appendix 2 and 3, for pictograms and Risk and Safety Statements (R and S phrases) of each chemical, respectively.

INTRODUCTION

The main goal of the demonstrations to be carried out in the first section will be to study the reactivity of certain metals, in particular that of the first row transition metals. Since we will be working in small scale, the test tube will be the main apparatus to use.

Chemistry students have to familiarize with the techniques necessary to synthesise and characterise different chemical compounds. Synthesis of chemical compounds is the main type of procedure carried out in a laboratory and, therefore, the 2nd section will be devoted to learn how to synthesise compounds at high temperatures as well as to synthesise compounds containing p block elements. The 3rd section will introduce the student into the synthesis guided by a “synthetic-map”. The latter, schematically describes the steps to be taken in order to prepare different products, using a specific element or compound as starting material.

At the end of this course, chemistry students should be able to perform bibliographic studies prior to beginning any experiment, make a correct planification of the work to be done and be able to fill their laboratory notebook properly. The latter should clearly indicate the steps taken, the results and the corresponding calculations and interpretations.

REACTIONS WITH SEVERAL METALLIC ELEMENTS. Cr, Mn, Fe, Co, Ni, Cu, Zn and Al

Elements belonging to the transition elements block are those who have, in all or most of their oxidation states, partly filled d orbitals. In consequence, Zn does not belong to this group, but being its chemistry similar to that of the transition elements, it is often included.

Transition elements have particular and interesting properties, such as multiple oxidation states and ability to form coordination compounds. Ions and compounds containing transition elements in any oxidation state are generally coloured, due to electronic transitions between different energy levels of d orbitals.

Elements in a particular transition group have common properties, but they can still be classified as i) light and ii) heavy elements. Light elements (those belonging to the first transition row), are involved in a particular and special chemistry type; heavy elements (those belonging to the second and third transition rows), are involved in a second type of chemistry.

The first section of this course analyses the chemistry of first row transition elements, from Chromium to Zinc. Upon completion of the proposed demonstrations, the student will be able to understand the most important properties of transition metals.

In order to understand the main differences between transition and non-transition metals (mainly colour and oxidation state), the student will also work with Aluminum.

The reactions will be carried out in small scale and, therefore, test tubes and dropper pipettes will be the materials most commonly used.

The demonstrations are mainly based on the following concepts: changes in oxidation state, generation of complexes, confirmation of the presence of ions, etc. In any case, one of the following may occur: colour change, generation of a precipitate or liberation of a gas. Each observation will have to be reported with the corresponding chemical reaction and bibliographic data (references corresponding to section 1: 1-6.)

CHROMIUM ($3d^5 4s^1$)

The most stable and important oxidation state of chromium is III, although Cr (VI) compounds can also be found when all 3d and 4s electrons are lost.

Chromium (VI)

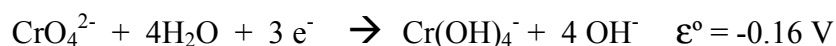
Generates oxocompounds, with potent oxidative properties.

Water solutions of Cr(VI) are of particular importance:

. Basic solutions contain Cr(VI) as the yellow chromate ion, CrO_4^{2-} , which generates non soluble chromate compounds.

. Acid solutions contain Cr (VI) as the orange soluble dichromate ion, $\text{Cr}_2\text{O}_7^{2-}$.

Acid, dichromate containing solutions are potent oxidants, while basic, chromate containing solutions are weak oxidants.



Chromium (III)

Generates a wide variety of compounds and complexes (Coordination number = 6).

Chromium (II)

Cr(II) containing compounds are scarce, they are easily oxidized both in solution and due to the action of molecular oxygen.

Experimental section.

Reactions involving chromium(VI)

Bear in mind that dichromate and chromate ions are orange and yellow, respectively, both containing Cr (VI).

Cr.1.- Add 1 ml of 0.2 M potassium dichromate solution to a test tube.

Add, dropwise, a saturated solution of NaOH until a change in colour is observed.

Add 1 ml of a concentrated sulphuric acid solution.

Analyze the observed colour changes.

Cr.2.- Add 1 ml of 0.2 M potassium chromate solution to a test tube.

Add, dropwise, a concentrated solution of HCl until a change in colour is observed.

Add 2 ml of a saturated solution of NaOH.

Analyse the observed colour changes.

Cr.3.- Add 1 ml of 0.2 M potassium dichromate solution to a test tube.

Add 1 ml of water.

Acidify the solution adding 2 drops of concentrated sulphuric acid.

Add 2 ml of iron(II) containing solution (prepared by the teacher).

Analyse the observed colour changes.

To confirm the presence of iron(III), add, dropwise, a 0.2 M potassium thiocyanate solution, until a change in colour is observed.

Reactions involving chromium(III)

Cr.4.- Add 1 ml of 0.2 M chromium(III) chloride solution to a test tube

Add, dropwise, a 1 M sodium hydroxide solution, until a precipitate is formed.

Add, dropwise, a concentrated solution of sodium hydroxide, until the precipitate disappears.

Add 2 ml of a 30% hydrogen peroxide solution and heat slowly.

Cr.5.- Add 2 ml of a chromium(III) chloride solution to a test tube.

Add 2 ml of a 2 M hydrochloric acid solution.

Add a turning of zinc.

Heat the test tube to effervescence and note any colour change.

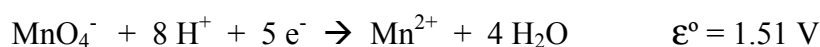
Quickly decant the solution into a test tube containing 3 ml of a saturated sodium acetate solution.

MANGANESE (3d⁵4s²)

Among the metals of the first transition row, manganese shows the widest variety of oxidation states.

Manganese (VII)

The highest oxidation state corresponds to that where the metal loses all 3d and 4s electrons (VII), which is found in oxo complexes, such as in the violet salts containing MnO₄⁻ (permanganate) ion. The latter is a strong oxidant, both at low and high pH:



Manganese (II)

This is the energetically most stable oxidation state for manganese, found in many weakly coloured compounds, such as the light pink hexaaquo ion [Mn(H₂O)₆]²⁺. In spite of being the most stable oxidation state, Mn (II) is easily oxidised.

Other oxidation states of manganese

Compounds containing manganese with an intermediate oxidation state are scarce, tending to undergo disproportionation reactions. As observed in the free energy diagram corresponding to manganese oxidation states, states III and IV undergo such a reaction.

Mn(III) is stabilized by the complexing oxalate or acetylacetonate anions.

Mn(IV) is found in manganese dioxide, black solid, inert to most acids at low temperatures and a good oxidizer at high temperatures (does not dissolve back to Mn(IV)).



The former reaction is commonly used to generate Cl₂(g) at laboratory scale.

Mn(VI) has only been found in MnO₄²⁻, a green compound only stable in basic solution.

Experimental section

Reduction of permanganate(tetraoxomanganate(VII))

Mn.1.- Add 1 ml of a 0.05M potassium permanganate solution to a test tube and 1 ml of a saturated solution of sodium hydroxide.

Heat with precaution until gas is liberated and colour is changed.

Store the resulting solution for future tests.

Mn.2.- Add 1 ml of a 0.05 M potassium permanganate solution to a test tube and 1ml of a 0.05 M sodium hydroxide solution.

Add 2 ml of a 0.2 M sodium sulphite solution and shake until a precipitate is formed.

Disproportionation of manganate(tetraoxomanganate(VI))

Mn.3.- Dilute the Mn1. green solution with 2 ml of water.

Add, dropwise, a concentrated sulphuric acid solution, until a change in colour is observed.

Leave the test tube still for 30 minutes until the precipitate sinks.

Reactions with manganese dioxide

Mn.4.- Add a sample of manganese dioxide to a test tube.

Add 2 ml of a concentrated hydrochloric acid solution.

Heat the tube and observe the dissolution of the precipitate and the liberation of gas.

Formation of Manganese(III)

Mn.5.- Dissolve a sample of manganese(II) sulphate in 2 ml of a 1 M sulphuric acid solution.

Add, with caution, 2 ml of a concentrated solution of sulphuric acid and let the tube cool down.

Add 5 drops of a 0.05 M potassium permanganate solution. Manganese(III) oxidation state is stabilized as a sulphate complex

Mn(III) is unstable in water solution. Add the solution to a beaker containing 25 ml of distilled water. Analyze the results.

Reactions with manganese(II)

Mn.6.- Add 2 ml of a 0.2 M manganese(II) chloride solution to a test tube.

Add 1 ml of a 1 M sodium hydroxide solution.

Add, dropwise, 1 ml of a 30% hydrogen peroxide solution.

Let the formed precipitate sink and eliminate the supernatant with a Pasteur pipette.

Add 2 ml of a concentrated hydrochloric acid solution to the precipitate; heat the tube and observe the dissolution of precipitate and the liberation of gas.

Mn.7.- Add 2 ml of a 0.2 M manganese(II) chloride solution to a test tube.

Add 1 ml of a concentrated phosphoric acid solution.

Add a sample of potassium periodate and heat the tube for 1 minute. Analyse the observed colour change.

Mn.8.- Add 2 ml of a 0.2 M manganese(II) chloride solution to a test tube.

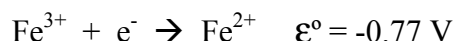
Add, dropwise, a 1 M sodium hydroxide solution until a precipitate is formed.

Let it still for a while and analyze the colour change observed in the precipitate.

IRON (3d⁶4s²)

There is no iron complex where the oxidation state of the metal corresponds to the loss of all the 3d and 4s electrons. Only oxidation states II and III are significant in the water chemistry of iron.

According to its reduction potential, iron(II) is a good reducing agent:



Iron(II) creates salts with any stable anion, which can be obtained as green coloured hydrated substances by evaporation of aqueous solution with the proper composition. Aqueous iron(II) solutions, with no other complexating species, contain the light greenish/bluish hexaquo $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$ ion, also known as Mohr's salt. The latter, ammonium iron(II) sulphate hexahydrated salt, is an important compound, resistant to water loss and air-directed oxidation. Many other iron(II) compounds can undergo superficial oxidation.

Iron(III) forms salts with most anions, but those which are strong reducing agents. Iron (III) solutions tend to provoke hydrolysis reactions.

The $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ hexaquo ion is found in very acidic solutions and, most likely, in highly hydrated salt crystals.

Both iron(II) and iron(III) generate a wide variety of complexes, which adopt mainly octahedral geometries.

Compared to other transition elements, the (stabilization) energy difference between iron(II) and (III) is small and, therefore, transitions between one state and the other are frequently observed in solutions containing weak oxidizing or reducing agents.

Iron (VI). Oxidation state VI is rarely observed. FeO_4^{2-} ferrate(VI) ion is such a case, it is a reducing agent stronger than permanganate, quite stable in basic solution and unstable (decomposes liberating molecular oxygen) in neutral or acidic solutions.

Experimental section

Reactions with metallic iron.

Add a sample of metallic iron to two different test tubes and proceed as follows:

Fe.1.- Add 2 ml of a 1 M sulphuric acid solution and analyse the events, if any.

Heat the test tube and analyse the colour of the solution.

Analyse the gas liberation process. Which is the gas generated?

Fe.2.- Add 2 ml of a concentrated nitric acid solution and analyse the events, if any.

Heat the test tube and analyse the colour of the solution.

Analyse the gas liberation process. Which is the gas generated?

Fe.3.- Add 2 ml of a 0.2 M copper(II) sulphate solution to a test tube and add an iron nail.

Reactions of iron(II) cation

Solutions containing iron (II) are highly unstable and are readily oxidised to iron(III); for that reason, iron(II) solutions to be used in the following demonstrations need to be readily prepared and used, and will be provided by the teacher.

Prepare two test tubes with 2 ml each of an iron(II) solution and proceed as follows:

Fe.4.- Add, dropwise, a 1 M sodium hydroxide solution until a precipitate appears.

Split the content of the test tube in two and proceed as follows:

Fe.4.a.- Add 2 ml of a saturated sodium hydroxide solution to the first test tube.

Fe.4.b.- Add 2 ml of a concentrated hydrochloric acid solution to the second test tube.

Fe.5.- Add 2 ml of a 0.2 M potassium hexacyanoferrate(III) solution.

Reactions of iron(III) cation

Prepare three test tubes with 2 ml each of a 0.2 M iron(III) sulphate solution and proceed as follows:

Fe.6.- Add, dropwise, a 1 M sodium hydroxide solution until a precipitate is observed. Split the content of the test tube in two and proceed as follows:

Fe.6.a.- Add 2 ml of a saturated sodium hydroxide solution to the first tube.

Fe.6.b.- Add 2 ml of a concentrated hydrochloric acid solution to the second tube.

Fe.7.- Add 2 ml of a 0.2 M potassium hexacyanoferrate(II) solution.

Fe.8.- Add 1 ml of a 0.2 M potassium thiocyanate solution.

Add, dropwise, a saturated potassium fluoride solution until a precipitate is observed.

Interchange between iron(II) / iron(III)

Fe.9- Add 2 ml of an iron(II) solution to a test tube.

Add 5 drops of a 30% hydrogen peroxide solution.

Add 1 ml of a saturated sodium hydroxide solution.

What is the composition of the precipitate?

Fe.10.- Add 1ml of a 0.2 M iron(III) sulphate solution to a test tube.

Add 1ml of a 0.2 M sodium sulphite solution.

Add the same volume of a 2 M hydrochloric acid solution and heat for a couple of minutes.

Confirm the presence of iron(II) in the solution (using Fe.5 demonstration, for instance).

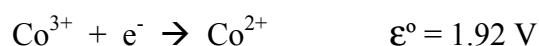
COBALT (3d⁷4s²)

Cobalt(II) and (III) are the most abundant oxidation states of this metal, being IV the highest oxidation state possible. Co(II) generates multiple simple and/or hydrated salts with every common anion, being the hexaquo species [Co(H₂O)₆]²⁺ found in many of them.

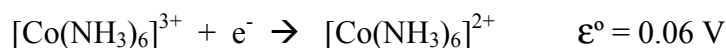
There are plenty of coordination compounds containing cobalt as central atom and these can display a wide variety of coordination numbers.

The most important property of Co(II) is that of being easily oxidised by molecular oxygen in the presence of ligands that can chelate the metal and, particularly, those ligands containing donor nitrogen atoms.

In general, aqueous solutions of Co(II) are not oxidised to Co(III):



Being Co(III) quite unstable, there are just a few Co(III) simple salts or binary compounds. Among the former, halides, sulphates and alum are found, which are reduced by the action of water. Co(III) state can be stabilized by the presence of complexating species such as NH₃:



There are multiple coordination compounds containing Co(III), with an octahedral disposition of, most likely, donor nitrogen atom containing ligands, such as ammonia and amines.

Experimental section

Prepare 5 test tube with 2 ml each of a 0.2 M cobalt(II) nitrate solution, and proceed as follows:

Co.1.- Add, dropwise, a 1 M sodium hydroxide solution until a blue precipitate is formed.

Add additional drops of the same solution until a change in the colour of the precipitate is observed. Split the suspension in two and proceed as follows:

Co.1.a.- Add 5 drops of a 30% hydrogen peroxide solution to the first tube.

Co.1.b.- Leave the second tube on the bench until a change in colour is observed.

Co.2.- Add, dropwise, a concentrated ammonia solution until a blue precipitate is formed.

Add additional ammonia solution until the precipitate disappears.

Split the solution in two and proceed as follows:

Co.2.a.- Add 5 drops of a 30% hydrogen peroxide solution to the first tube.

Co.2.b.- Leave the second tube on the bench until a change in colour is observed.

Co.3.- Acidify the solution adding 5 drops of acetic acid.

Add a small sample of potassium nitrite and shake.

Co.4.- Add, dropwise, a 0.2 M sodium sulphide solution until a precipitate is formed.

Co.5.- Add 1 ml of a concentrated hydrochloric acid solution.

Add 2 ml of water.

Analyse de observed colour changes.

NICKEL($3d^8 4s^2$)

Its most abundant oxidation state is II. In this state, Nickel creates many compounds, such as halides, cyanides and oxosalts of oxoacids. In aqueous environment, the hexaaquo $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ green ion is found.

Experimental section

Prepare 4 test tubes, with 2 ml each of a 0.2 M nickel(II) chloride solution and proceed as follows:

Ni.1.- Add 1 ml of a 1 M sodium hydroxide solution. Add 2 additional ml of the same solution.

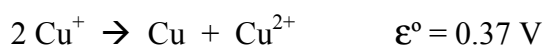
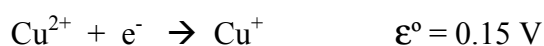
Ni.2.- Add, dropwise, a 1 M ammonia solution, until a precipitate is formed.
Add 3 ml of a concentrated ammonia solution.
Analyse the observed changes.

Ni.3.- Add, dropwise, a 0.2 M sodium carbonate solution until a precipitate is observed.

Ni.4.- Add, dropwise, a 0.2 M sodium sulphide solution until a precipitate is formed.

COPPER(3d¹⁰4s¹)

Cu(I) (3d¹⁰) creates diamagnetic compounds, which are very weakly coloured unless the colour is provided by the anion. Cu(I) is quite unstable in water solutions, tending to disproportionate into Cu(II) and metallic Cu, as deduced from the corresponding reduction potentials:



Only insoluble Cu(I) complexes are stable in water; this modest stability may arise from the fact that hydrating energy of Cu⁺ cation is small compared to that of Cu²⁺. As a result, Cu(I) derivatives are unstable in water solution.

Cu(II) is the most important oxidation state of copper. Cu(I) compounds are easily oxidised to Cu(II). Water chemistry of Cu²⁺ is well known, and creates multiple salts composed of different anions. In water solution the cation [Cu(H₂O)₆]²⁺ is found. Many tetrahedral and octahedral coordination compounds are known for Cu(II).

Experimental section

Cu.1.- Add one Cu turning and 2 ml of concentrated nitric acid solution to a test tube. Analyse the reaction taking place. Heat the tube if necessary.

Cu.2.- Add one Cu turning and 2 ml of concentrated hydrochloric acid solution to a test tube. Is there any reaction?

Add, dropwise, a 30% hydrogen peroxide solution. Analyse the results.

Generation of copper(II) hydroxide and some of its reactions.

Cu.3.- Add 2 ml of a 0.2 M copper(II) sulphate solution to a test tube.

Add the same volume of a 1 M sodium hydroxide solution. Copper(II) hydroxide precipitates. Split the suspension in three portions and proceed as follows:

Cu.3.a.-Boil the content of the first tube.

Cu.3.b.-Add 2 ml of a concentrated hydrochloric acid solution to the second tube.

Cu.3.c.-Add 2 ml of a saturated sodium hydroxide solution to the third tube.

Oxidative properties of copper(II) salts

Cu.4.- Add 2 ml of a 0.2 M copper(II) sulphate solution to a test tube

Add the same volume of a 0.2 M potassium iodide solution.

Analyse the results.

Cu.5.- Add 2 ml of a 0.2 M potassium bromide solution to a different test tube.

Add the same volume of a 0.2 M copper(II) sulphate solution.

Compare the current and the previous (Cu.4) results.

Complexes of Copper(II)

Copper(II) creates many different complexes, with many different colours.

Cu.6.- Add 2 ml of a copper(II) chloride solution to a test tube.

Add the same volume of a concentrated hydrochloric acid solution.

Analyze the colour of the solution.

Dilute the obtained solution using distilled water. Add 2 ml of a concentrated ammonia solution.

Analyse the changes in colour.

ZINC($3d^{10}4s^2$)

The oxidation state of zinc is II, corresponding to the loss of the 2 s electrons; since its d subshell is full, it is considered a non-transition element. Still, as mentioned in the introduction section, Zinc has the ability to form complexes with ammonia, amines, halides and cyanides, being therefore similar in properties to transition elements.

There are many well known compounds containing zinc(II), such as halides, oxoacids, salts, sulphides, etc. Zinc(II) oxide is white and is generated straightforward by direct combination. Both zinc(II) oxide and hydroxide are amphoteric.

With halides, zinc(II) generates ZnX_4^{2-} formulation compounds, and can be isolated as salts of bulky cations. It also complexates with ammonia and amine ligands, obtained as crystalline salts.

Experimental section

Zinc reacts easily with non oxidant acids, converting into the divalent cation and liberating hydrogen. It also dissolves in strong bases, giving rise to ZnO_2^{2-} zincate ion.

Zn.1.- Add one shot of zinc to a test tube and cover it with a concentrated hydrochloric acid solution. Analyse the gas liberation process that takes place.

Zn.2.- Add one shot of zinc to a test tube and add 1 ml of a concentrated sodium hydroxide solution. Heat the sample if necessary.

In acidic environments, zinc reduces metallic ions with high reduction potential to metal 0, such as Cu(II) to Cu^0 or Fe^{3+} to lower oxidation states.

Zn.3.- Add 2 ml of a 0.2 M solution of copper(II) sulphate to a test tube and add a shot of zinc. After five minutes, heat moderately. Analyse the results.

Zn.4.- Add 2 ml of a 0.2 M iron(III) sulphate solution to a test tube and add a shot of zinc.

Generation of zinc hydroxide and its reactions.

Zn.5.- Add 4 ml of a 0.2 M zinc(II) sulphate solution to a test tube.

Add a 1 M sodium hydroxide solution dropwise, until a precipitate is observed. Split the suspension and proceed as follows:

Zn.5.a.-Add to the first test tube, dropwise, a saturated sodium hydroxide solution, until the precipitate is dissolved.

Zn.5.b.- Add to the second tube, dropwise, a concentrated hydrochloric acid solution, until the precipitate is dissolved.

Zn.5.c.- Add to the third tube, dropwise, a concentrated ammonia solution, until the precipitate is dissolved.

Zn.5.d.- Leave the fourth tube still for a couple of minutes and remove the supernatant with a Pasteur pipette. Calcine the obtained white solid until it turns yellow.

Zinc(II) reactions.

Add 2 ml of a 0.2 M zinc(II) sulphate solution to each of two different test tubes, and proceed as follows:

Zn.6.- Add 1 ml of a 0.2 M sodium sulphide solution to the first test tube..

Zn.7.- Add 2 ml of a 0.2 M potassium hexacyanoferrate(II) solution to the second test tube.

ALUMINIUM ($3s^23p^1$)

Aluminum belongs to group 13th, where all the elements but boron show a marked metallic nature. Despite having a metallic ground state, the bonds to aluminum in aluminum complexes are partly ionic and partly covalent in nature. The most important oxidation state for elements of this group is III. Oxidation state I is increasingly stabilized as we travel down the group in the periodic table, being of particular importance in the case of thallium.

Aluminum can be attacked by diluted bases and acids, although it is passivated when exposed to nitric acid due to the generation of a protecting oxide layer. In aqueous acidic environments, aluminum is oxidized to Al^{3+} , while in basic solutions, the primary species is the aluminate ion AlO_2^- or $Al(OH)_4^-$.

When heated, aluminium reacts straightforward with oxygen, sulphur, nitrogen, halogens and other non-metallic elements, yielding the corresponding binary compound. Although oxides and fluorides are ionic, other binary combinations are, mostly, covalent, and they are macromolecules.

Aluminum halides act as Lewis acids and, therefore, they can accept neutral molecules or anions as ligands, to generate tetrahedral adducts and haloanionic complexes, such as AlX_4^- .

Having a very high heat of formation, aluminum can be used to reduce metallic oxides, through a process known as aluminothermic reaction.

In the presence of strong oxoacids, aluminum creates hydrated salts, such as alum. The latter is an important family of compounds of aluminium and, being an example of structure of aluminium compounds, some other compounds composed of elements other than aluminium are also named after “alum”. Their general formula is $MAl(SO_4)_2 \cdot 12H_2O$, with M being the monovalent cation or ammonia. In this type of complexes, as happens in most hydrated salts, hexaquo $[Al(H_2O)_6]^{3+}$ ions are found.

There are a just a few tetrahedral complexes, being the hexacoordinated complexes composed of chelating rings the most abundant ones, such as those containing trioxalate or acetylacetonate anions.

Experimental section

Effect of acids and bases on the metal.

Despite being a highly reactive metal, a protecting layer of oxide is created on its surface, which prevents the reaction from taking place.

Al.1.- Add a shot of aluminum to a test tube

Add 5 ml of a 2 M hydrochloric acid solution.

Study the progression of the reaction and heat if necessary.

Al.2.- Add a shot of aluminum to a test tube.

Add 5 ml of a 1M sodium hydroxide solution.

Analyse the result.

If gas liberation diminishes with time, heat smoothly.

Generation of aluminium hydroxide and its reactions.

Al.3.- Add 2 ml of a 0.2 M aluminium trichloride solution to a test tube.

Add 2 ml of concentrated ammonia solution. A precipitate is formed.

Split the suspension in three tubes and proceed as follows:

Al.3.a.- Add 2 ml of a concentrated ammonia solution to the first tube.

Al.3.b.- Add 2 ml of a saturated sodium hydroxide solution to the second tube.

Al.3.c.- Add 2 ml of a 1 M sulphuric acid solution to the third tube.

INTRODUCTION TO DEMONSTRATIONS AT HIGH TEMPERATURE.

Most metals are found as oxides in nature (or as a mineral that can be converted into an oxide). For that reason, the method most widely used to obtain metals is the reduction of their oxides.

There are plenty of ways to reduce an oxide using a second element. In spite of the high thermodynamic stability of the oxide, oxygen can be replaced by another element. For this to happen, the oxide of the second element must be more stable than that of the metal.

1st DEMONSTRATION: OBTENTION OF TUNGSTEN STARTING OVER A SALT WITH IMPURITIES.

Notes

Use the fume hood when working with concentrated acids and ammonia.

Discussion

The goal of the current demonstration is the obtention of metallic tungsten from a mineral where it is contained (Scheelite). The procedure can be divided as follows: A first and wet part, where a tungstenate salt will be isolated by means of several acid and basic treatments; and a second and dry part where, at high temperature, metallic tungsten is obtained upon reduction of the previously obtained tungstenate.

Procedure

Separation methods are based on the reactivity of minerals with hydrochloric acid. Tungsten will be found on the insoluble pellet, as $\text{WO}_3 \cdot x\text{H}_2\text{O}$. The latter dissolves in ammonium hydroxide yielding ammonium wolframate which is first calcinated and then reduced to obtain metallic tungsten.

As starting material 4 g of Scheelite will be used, which are heated together with 50 ml of a concentrated hydrochloric acid solution for 30 min. After cooling down, add 1 ml of water and filtrate in vacuum. Clean the solid product with 50 ml of a 6 M HCl solution, to get rid of all the soluble salts.

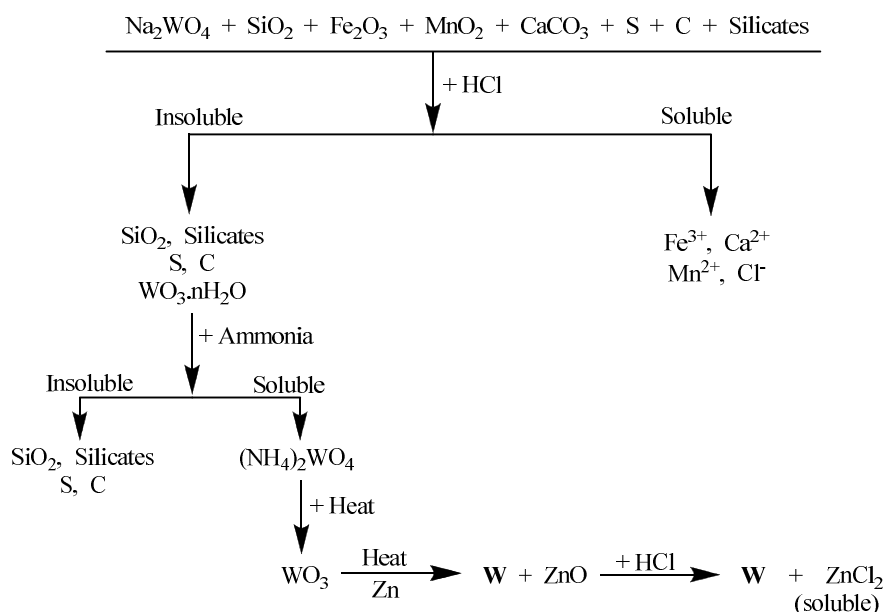
Treat the solid product in the fume hood with concentrated ammonia (50 ml should be enough) and heat for 10 min to promote the formation of ammonium wolframate. Filtrate the solution in the fume hood using the filter funnel. Collect the filtrate solution in an evaporating dish and let it dry. Attention: Heat slowly to avoid splashing.

Collect and calcinate the remaining solid in the crucible (ask the teacher) in the fume hood, first directly in the Bunsen burner till the solid gets a yellowish colour, and then, calcinate further in the oven for 15 minutes at 400 °C (wolframate loses all its ammonia). The obtained tungsten oxide should be a yellow solid.

The next step implies the reduction of the tungsten oxide by means of metallic zinc (see Ellingham diagrams). With that goal, weigh the obtained tungsten oxide and mix it with 3 times its weight of metallic Zn. Grind it, mix generously and calcinate in the oven for 10 min at 600 °C. Take the evaporating dish out of the oven, shake the content with a spatule and calcinate in the oven for 10 more minutes.

Transfer the obtained product to a beaker and get rid of the byproducts of the reduction reaction treating the sample with 20 ml of a concentrated HCl solution. If necessary, heat the sample further to finish the attack. Finally, dilute in water, vacuum filter and wash with water. The obtained bluish-black powder is tungsten. Dry it in the oven, weight it and calculate the yield of the procedure, considering a purity of 40% of the initial mineral.

Below is a diagram of the complete procedure:



CONFIRMATION OF THE PRESENCE OF TUNGSTEN

The presence of tungsten can be checked attending to the blue colour of W_2O_5 .

Treat a small sample of the obtained solid with 20 ml of a hydrochloric acid solution and a piece of Sn (use a small beaker and the fume hood). Heat the mixture to ensure the attack. Under these conditions, the mixture should get a blue colour. Indicate the reactions taking place in the confirmation of the presence of tungsten.

Describe, from a thermodynamic point of view, the reduction procedure of WO_3 at high temperature. By means of Ellingham diagrams for oxides, calculate ΔG at working temperature.

2nd DEMONSTRATION AT HIGH TEMPERATURE. OBTENTION OF DIHYDRATED BARIUM CHLORIDE .

Notes

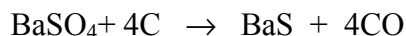
After cooling down the crucible, the dissolution in water and attack with HCl is carried out in the fume hood, since the poisonous H_2S gas is liberated.

Discussion

The main goal is the obtention of dihydrated barium chloride using the naturally occurring mineral Baritine (barium sulphate) as starting material. Coal will be used to reduce sulphate to sulphure. The obtained solid will be purified by means of hydrochloric acid, and the soluble salt $BaCl_2$ will be crystallised following a rutinary concetration procedure. Applying the concept of the common ion, its solubility will be reduced by addition of HCl.

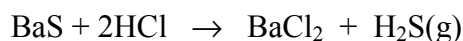
Procedure

Program the oven for crucibles at 900 °C. Weigh 6 g of barium sulphate and 3 g of coal (grind and mix them for 30 min. in the mortar). Transfer the mixture to the crucible and cover it with a layer of coal. Cover the crucible itself with a lid and leave it for 90 min. in the oven.



Why do we use coal? Could an alternative substance be used? Why? Any advantage or disadvantage? Look up in any book the chapter related to metal extraction, Gutierrez Rios, for instance). Why do we cover the crucible with a lid?

After cooling down the crucible, drop its content to a beaker containing 60 ml of boiling water (use the fume hood) and keep it boiling. After a few minutes, add 10 ml of concentrated HCl and continue heating the sample until no more H₂S is liberated. To double check that the latter occurs, use a piece of filtering paper which must be wet with a 0.2 M solution of lead acetate.



Let it cool down and vacuum filter. Heat the filtrate, add 5 ml of a HCl solution and let the solution evaporate until it looks like a saturated solution. Cool it down again, add 5 drops of a concentrated HCl solution and shake. Vacuum filter and dry the obtained crystals, weigh them, calculate the yield of the procedure and show to the teacher.

ANALITICAL TESTS

Dissolve some of the crystals in distilled water and split the solution into two test tubes. Confirm the presence of the following ions and discuss the results:

Chloride ion: Add some drops of a 0.2 M AgNO₃ solution. Then, add some concentrated ammonia until the precipitate dissolves.

Barium ion: Add some drops of a 1 M H₂SO₄ solution.

WATER OF CRYSTALLISATION

Calculate the percentage of water of crystallisation in the obtained salt. For a correct result, the sample must be completely dry.

Hydrated BaCl₂ loses water if heated at 100 °C, being the anhydrous salt stable at high temperatures. To estimate the water percentage add a known amount of the hydrated salt on a drying capsule and heat in a Bunsen burner. When dehydration is complete, weight the salt and estimate the hydration percentage. Compare the experimental and theoretical results.

What are the differences between the coordination and crystallization water?

PREPARATION OF COMPOUNDS CONTAINING ATOMS OF THE p BLOCK

In the third demonstration, different compounds will be prepared, all containing a non-metallic element, such as Boron: Boric acid (H_3BO_3) and potassium tetrafluoroborate (KBF_4).

In the fourth demonstration, compounds containing lead will be obtained (lead is a p block element).

Lead can be found forming a red oxide (MINIO in Spanish), where the oxidation state is both II and IV for the lead, since its molecular formula is Pb_3O_4 ($2\text{PbO} + \text{PbO}_2$). Note that MINIO is not a mixture of two different oxides, even though its chemical behaviour resembles that of a mixture.

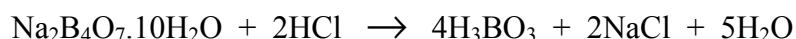
There are several Pb(II) crystal salts, most of them anhydrous and with very small water solubility (such as PbCl_2) or completely insoluble (such as PbSO_4). The exception to this is $\text{Pb}(\text{NO}_3)_2$, which is completely ionized in water.

3rd DEMONSTRATION: OBTENTION OF SEVERAL BORON COMPOUNDS USING BORAX AS STARTING MATERIAL

PRECEDURE

Boric Acid.

Weigh 20 g of borax and transfer them to a 250 ml beaker. Add 20 ml of water and 20 g of a 24% hydrochloric acid solution. Keep it boiling for 5 minutes. What is the Boron percentage in Borax?



Cool the solution down in an ice bath and vacuum filter the obtained crystals. Wash the crystals with a small amount of cold water (0°C), dry with vacuum, weigh and calculate the yield of the reaction.

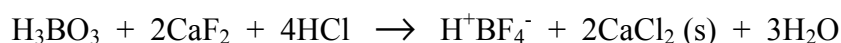
Why do we use ice to cool down and water at 0°C to wash?

Confirming the presence of Boric Acid.

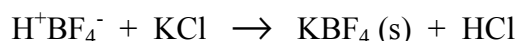
This demonstration must be carried out in the fume hood. Dissolve a small sample of the boric acid obtained previously, using 3-4 ml of methanol in a 100 ml beaker. Heat the sample and, carefully, approximate a source of fire (burner or match) to the beaker. The green coloured flames are a direct consequence of the presence of boric acid. Write down and comment the chemical reaction taking place.

Potassium tetrafluoroborate.

Add $\frac{3}{4}$ of the sample obtained previously (maximum 7 g), to a particular beaker the teacher will provide; add a stoichiometric amount of finely ground calcium fluoride and 64 ml of 24% HCl solution. Cover the beaker with a lid pierced by a glass tube and heat it smoothly in the fume hood for two hours.



Dilute the content of the beaker with 30 ml of water and cool it down on the bench. Filter the solution into a 250 ml beaker, and add 11 g of potassium chloride as a saturated solution.



Filtrate the obtained salt, wash with water and vacuum dry. Collect the solid product, dry it on the oven below 100 °C, weigh and calculate the yield of the procedure.

4th DEMONSTRATION. OBTENTION OF LEAD-COMPOUNDS USING MINIO AS STARTING MATERIAL.

Dangers

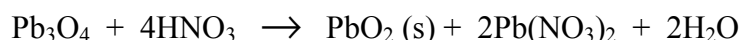
Since lead compounds are highly poisonous, avoid direct contact with skin. Collect the obtained lead salts in the corresponding container (ask the teacher). Do not through them to the sink or the paperbin.

INTRODUCTION

Minio, is a commercial product used in the market of coat painting, used to prevent iron oxidation. Its molecular formula is Pb_3O_4 (2 PbO + PbO₂).

Procedure

Add 30 g of minio to an evaporating dish and attack it with 45 g of a 38% nitric acid solution. Agitate and heat smoothly for 10 minutes, after which, a dark brown precipitate will appear. Dilute with 50 ml of water and filter using a Buchner funnel first (with no vacuum connection), and finish the drying procedure with vacuum. Wash with a small amount of water and collect that water together with the filtrate. Vacuum dry.



Dry the precipitate in the oven, weigh it and calculate the reaction yield.

Collect the precipitate into a 250 ml beaker.

Obtention of lead(II) nitrate.

Heat the previously obtained solution making use of a sand-bath. By doing so, the solution will concentrate up to the desired volume, taking into account the solubility of the salt. Cool the flask down under the tap and vacuum filter the obtained crystals. Recrystallize the filtrate by concentrating it down again and repeating the same procedure as before until there are only 10 ml of solution.

Dry the crystals in the Buchner using the vacuum system first and the oven second, weigh and calculate the yield of the reaction.

ANALITICAL TESTS

Disolve some of the obtained lead(II) nitrate crystals in 10 ml of water and split the solution into 5 different test tubes.

- a) Add some drops of a 0.2 M potassium cromate solution to the first tube and then, add a concentrated nitric acid solution until the precipitate is dissolved.
- b) Add some drops of a 1 M ammonia solution and then, add concentrated ammonia in excess.
- c) Add some drops of a 1 M sodium hydroxide solution and, then, add dropwise a saturated NaOH solution, until the precipitate is dissolved.
- d) Add some drops of a concentrated hydrochloric acid solution to the fourth tube.

- e) Add a diluted sulphuric acid solution to the fifth tube. Prepare the solution by adding 1 ml of concentrated sulphuric acid solution to 5 ml of water.

Describe the events observed in the analytical tests, write down the reactions occurred and discuss the results.

SYNTHESIS OF SEVERAL INORGANIC COMPOUNDS. ALTERNATIVE PROCEDURES.

The goal of the third section of the present subject is the synthesis of several compounds (including coordination compounds) containing one specific element.

The student should already be familiar with some of the techniques required to obtain the proposed compounds (such as preparation of solutions, concentration, filtration, crystallization, etc.).

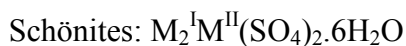
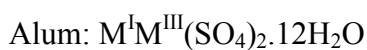
The procedures are based on the generation of salts using different reaction types, such as precipitation, redox or acid-base reactions.

In each “map” the student should carry out the synthesis of a simple salt, a binary salt, and a coordination compound.

BINARY SALTS.

A salt containing more than a cation and/or more than an anion is known as a “Double salt”. Hence, a binary salt should contain either two different cations or/and two different anions in its lattice. Formed by the combination of two different salts and being less soluble than the starting simple salts, the binary salt is generated mixing together two concentrated solutions containing each simple salt.

Sulphates are important binary salts, which can be classified as follows:



Binary salts are only found in solid state and, in general, they generate good quality and voluminous crystals.

COORDINATION COMPOUNDS

Coordination compounds are formed by a metallic atom or ion, which is surrounded by ligands. Ligands are neutral molecules or ions that are stable in free form (non-bound to metal). The number of bonds established between the metal and the ligands is known as the coordination number of the complex.

The metal center acts as a Lewis acid, while the ligands should contain a donor atom that would act as a Lewis base.

Unlike binary salts, coordination compounds are stable entities both in solid state and in solution. Their properties are different from those of the free-form metal or ligands.

EQUIPMENT TO USE WHEN THERE IS GAS LIBERATION

Several synthetic procedures imply the generation of dangerous or toxic gases (such as Cl_2 , H_2 , SO_2 ...), which must be collected or converted and brought to the corresponding collector. In such cases, a particular type of device must be constructed, which is composed of:

Distillation flask: The reaction takes place in this flask (the starting solid material is deposited here). This flask contains a mouth which is covered by a lid perforated by a Thistle-funnel, as well as a lateral exit tube. The latter directs the gas to the next component of the equipment, the gas washing flask.

Thistle funnel: It is adjusted to the top of the distillation flask (no gas liberation) and to it, the attacking substance is added, i.e., the corresponding acid. This funnel regulates the amount of attacking substance added in each moment. When the reaction starts the liberated gas exerts a pressure which avoids the acid to drop. The acid, therefore, feels two different pressures, the atmospheric pressure and the one exerted by the gas. The level of liquid won't vary while both pressures are equal.

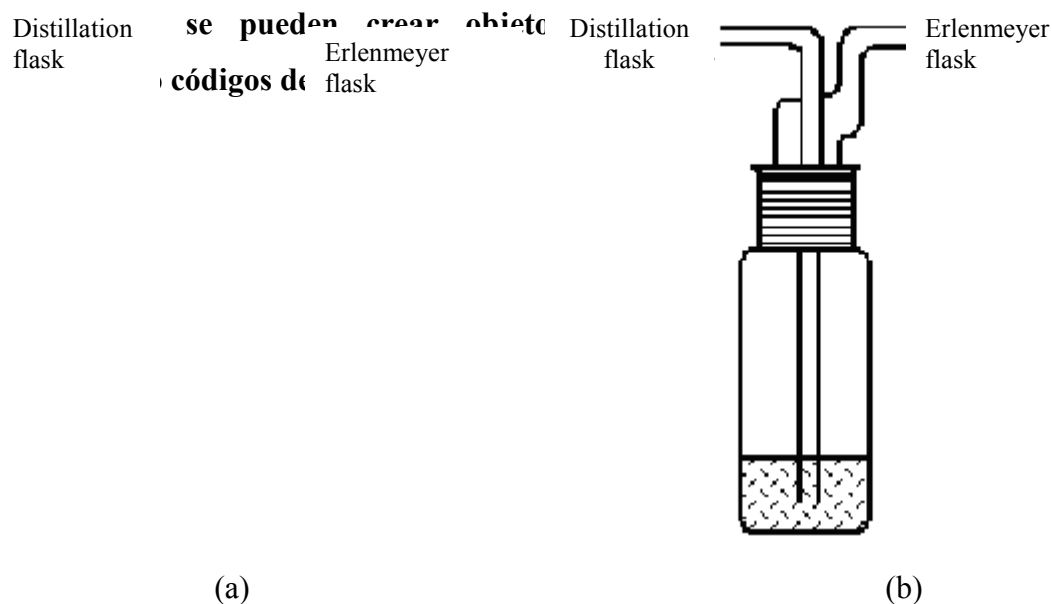
The pressure inside the flask diminishes with a decrease in the amount of gas liberated; in that moment, the liquid can begin to drop to the flask. The acid will only begin to drop when the pressure exerted by the gas liberated in the reaction is smaller than the atmospheric pressure, i.e., when the amount of acid in the flask is not enough. Hence, the acid will drop again to the flask and the reaction will continue. This process continues until the necessary acid quantity has dropped to the flask.

This funnel also serves as lid of the reaction flask.

Gas washing flask: Connected to the distillation flask on the one hand, and to the erlenmeyer flask on the other. It is a closed flask, which contains a tube reaching nearly its bottom. The gas liberated during the reaction travels through this flask and reach the erlenmeyer flask. Both flasks are connected through silicon tubes.

The gas washing flask has two parts: the head and the body. Both are connected through a ground glass socket, which must be covered with vaseline before reaction begins, to avoid getting them stuck together. There are several options for this flask:

- a) Leave it empty: it will then be a safety device to avoid undesired mixing of the different substances. In this case the long tube is connected to the erlenmeyer flask.
- b) Add a liquid: The liberated gas will bubble through it. For the latter to happen, the long tube must be connected to the reaction-flask. Last, gas exits from the upper part, which is connected to the last component by a 90° bent tube (U tube).



Erlenmeyer flask: The glass tube is inserted in the solution contained in the erlenmeyer, where the gas will bubble. The gas is dissolved in the solution, diluted and converted into a different substance.

The erlenmeyer flask is used to control the evolution of the reaction.

It must be kept open to avoid overpressurization, otherwise the pressure would increase and cause an explosion.

All these components are firmly joint together by pieces of silicon tubes, avoiding gas leaking.

These and other components will be chosen according to the type and properties of the reaction to be carried out.

In these laboratory demonstrations, assemblies 1 and 2 will be used.

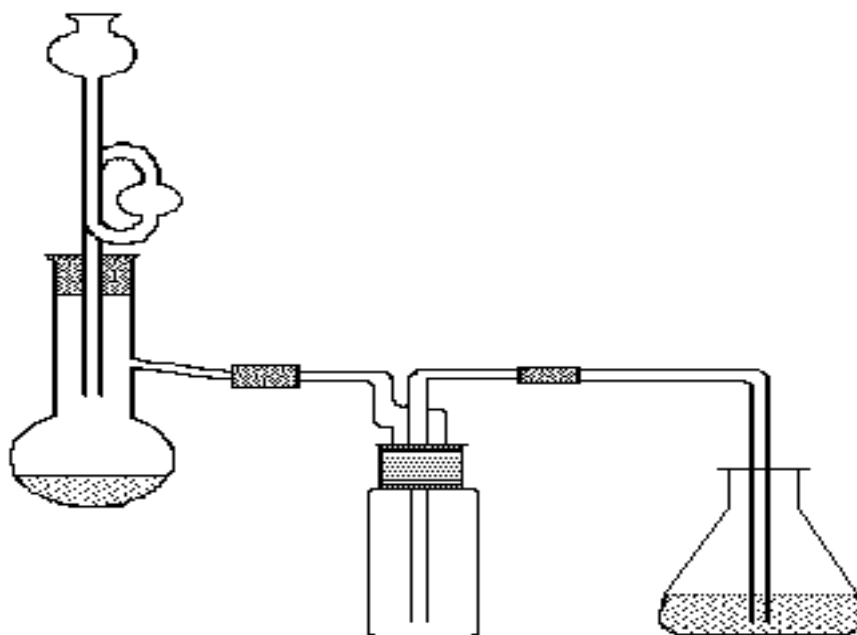


Figure 1

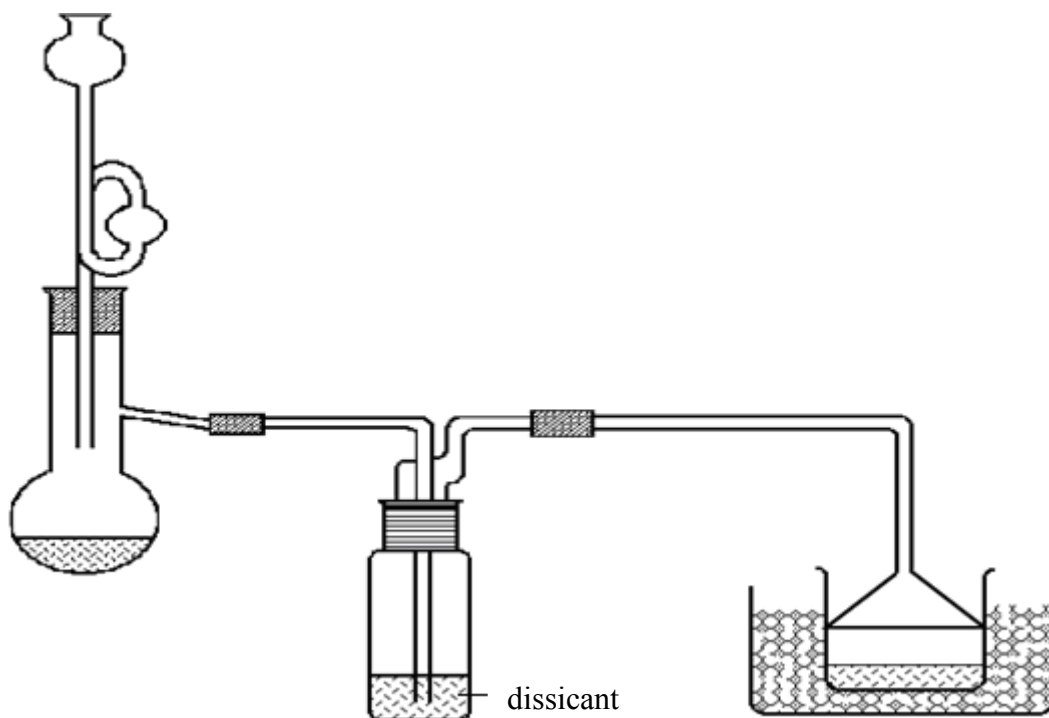


Figure 2

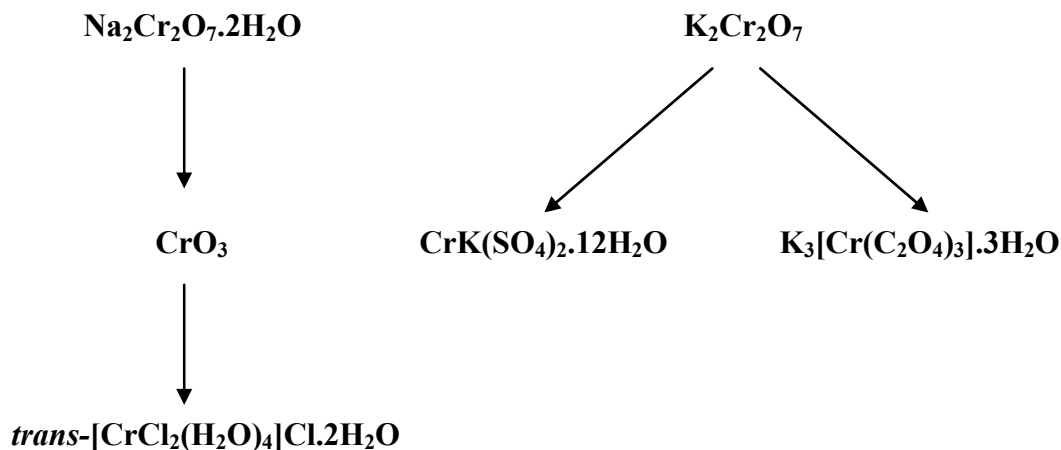
If the liberated gas is used to saturate a solution and maintain the atmosphere free of humidity, assembly 2 should be put together. In this case, a funnel will be assembled upside-down instead of the Erlenmeyer flask, covering the flask containing the solution to saturate. This way, the gas will be directed to the solution.

Below, the procedures to follow are schematised. The starting material could be either a metal or a simple compound containing the metal. Complete the schemes indicating the reactants, working conditions and corresponding bibliographic references.

Upon synthesis completion, confirm the presence of the desired product and, in particular, that of the ions of interest, by means of a simple reaction/procedure.

SYNTHESIS OF CHROMIUM COMPOUNDS

Starting from $M_2Cr_2O_7$ (M=Na or K)



SYNTHESIS OF CHROMIUM TRIOXIDE CrO_3 (7)



In a 250 ml beaker dissolve 10 g of sodium dichromate dihydrate in 25 ml of water. Then, with the help of a Pasteur pipette, add 40 ml of sulphuric acid, CAUTION, this is very exothermic, do it very slowly and continuously stirring, to obtain a good mix of the acid and the dichromate.

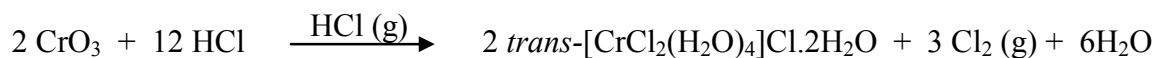
The contact of these two reactives will provoke the formation of small red coloured crystals of CrO_3 . To finish the crystallization, once all the acid has been added, put the mixture to cool down.

Filter in a filtration frit given by the teacher, and dry the obtained solid by vacuum. To finish the drying process put the product in the sulphuric acid desiccator.

If during the precipitation CrO_3 needles appear together with sodium hydrogensulphate, warm up the mixture until they dissolve and put it to cool down again.

Weigh the dry crystals, calculate the yield and put it away.

SYNTHESIS OF *trans*-TETRAAQUADICHLOROCHROMIUM(III) CHLORIDE DIHYDRATE *trans*-[CrCl₂(H₂O)₄]Cl·2H₂O (8,9)

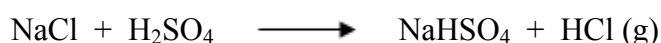


Put 5 g of CrO₃ in a 250 ml beaker and add 25 ml of concentrated hydrochloric acid. In the fume hood boil it (gently), until chlorine has been developed and the colour of the solution is green. If necessary, to complete the reaction, add more acid.

Concentrate the obtained solution in a water bath until a slurry is formed. Let it cool down to obtain a crystalline solid. Dry the solid in the sulphuric acid desiccator

Dissolve the obtained chloride in 10 ml of distilled water in 200 ml beaker and place in a salt and ice bath.

In the next step the solution has to be saturated by passing a current of HCl. This current is created by the reaction of sodium chloride and sulphuric acid. For this take 30 g of sodium chloride and add slowly sulphuric acid until having add 1.5 times the stoichiometric amount using the set up depicted in Figure 2 on page- 33 -. All these has to be performed in the fume hood.

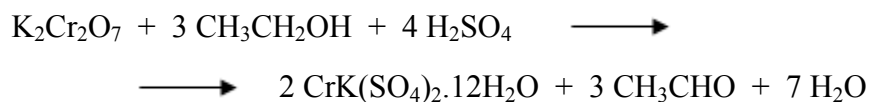


The reaction is starting by warming up gently on the Bunsen burner. Be carefull not to heat it up too much. As a consequence of the reaction a current of HCl is created, which saturates the solution and provokes the crystallization of the compound.

Filtrate the obtained crystals and after drying in vacuum put them in the sulphuric acid desiccator. Then weigh and calculate the yield.

Dissolve a small sample in water to make the test of chloride ions.

SYNTHESIS OF CHROMIUM AND POTASSIUM ALUM $\text{CrK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (8)



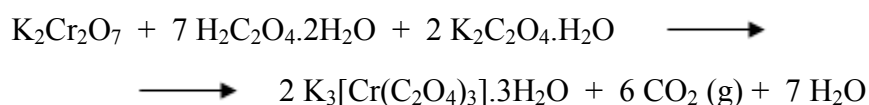
In a 200 ml beaker put 20 ml of water and then add 2.5 ml of concentrated sulphuric acid. In the resulting solution dissolve 3 g of potassium dichromate. Then place it in an ice bath in the fume hood. Then add 10 ml of absolute ethanol and stir it slowly. Maintain it under 60 °C.

Leave the resulting solution to crystallise in the beaker.

Filter the obtained crystals and dry in vacuum. Calculate the yield and put it away.

Take a small sample of the product, dissolve it in water and test for SO_4^{2-} and Cr^{3+} .

SYNTHESIS OF POTASSIUM TRIOXALATECHROMATE(III) TRIHYDRATE $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ (10)



It is prepared by reacting oxalic acid, potassium oxalate and potassium dichromate.

In 100 ml of water 10.6 g of oxalic acid and 4.5 g of potassium oxalate monohydrate are mixed. To the resulting suspension 3.5 g of potassium dichromate are added drop by drop and with agitation as a saturated solution.

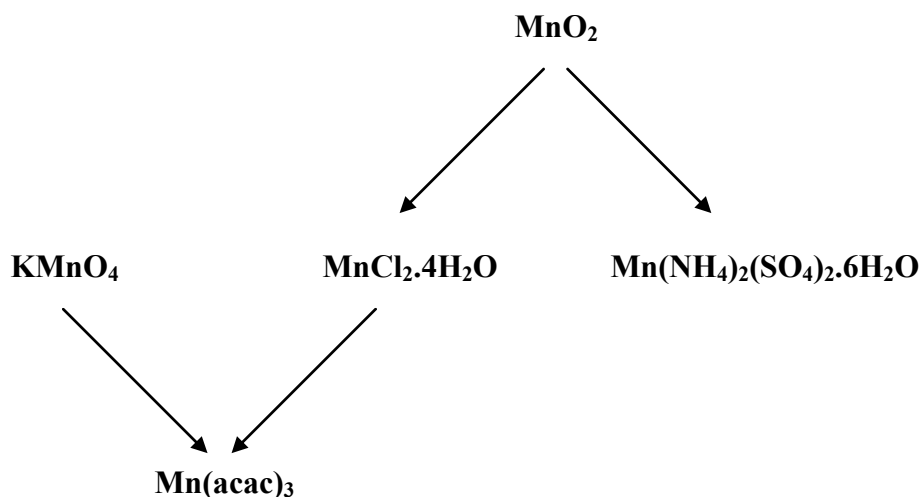
The resulting solution is concentrated to a minimum volume, and then cool it down slowly until crystallisation.

Filtrate and dry in vacuum.

If necessary, recrystallise, to do this dissolve the solid in the minimum amount of cold water to get a saturated solution (the volume will be more or less between 20 and 30 ml). Then boil the solution to reduce the volume.

Filter the obtained crystals and dry in vacuum. Calculate the yield and put it away.

OBTAINED PRODUCTS STARTING FROM MnO₂



SYNTHESIS OF MANGANESE(II) CHLORIDE TETRAHYDRATE MnCl₂·4H₂O (7,11)



In this demonstration we are going to obtain a manganese compound starting from MnO₂. In this starting material there are always iron impurities, and these react on the first attack. After this, a method to eliminate these iron products has to be developed.

In a 250 ml distillation flask put 20 g of manganese dioxide. The evacuation tube is connected to an empty oil bubbler, the bubbler's evacuation tube is lengthened with an elbow and is immersed in the solution of an Erlenmeyer flask. This solution is composed of 150 ml of water solution of 5 % sodium carbonate. See the set up in Figure 1 on page - 32 - for the set up in the fume hood.

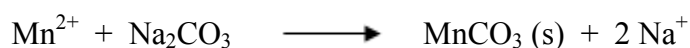
Add 100 g of concentrated hydrochloric acid with the Thistle funnel slowly. To start the reaction the flask is warmed slowly with the burner, and the attack is followed until no more chlorine is liberated.

Once the attack is finished let the content of the flask to cool down, then filtrate in a Buchner funnel and recover the solid (dry in the oven and the weigh everything), and pass the filtrate to a beaker.

This solution has impurities, mainly iron impurities. To eliminate these prepare 100 ml of ammonia solution 4 M and add it to the solution until the pH is around 5. This way the iron is separated as iron(III) hydroxide. If the value mentioned of the pH is passed manganese can also precipitate as manganese(II) hydroxide.

Boil the mixture for 10 minutes, leave it to cool down and filter.

Prepare 100 ml of a saturated solution of sodium carbonate and add it to the filtrate until pH 9 is achieved. Filtrate this suspension and see if the precipitation has been complete (add more sodium carbonate to the filtrate). Wash the solid repeatedly with water. This way manganese(II) carbonate is obtained precipitated and without impurities.



Place the solid obtained in a beaker and dissolve it in concentrated hydrochloric acid, using the smallest amount possible.

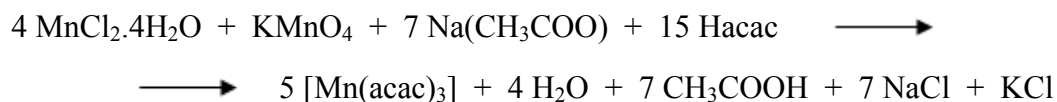


Evaporate the solution warming it up, until it appears as a saturated solution, leave it to cool down until crystallisation.

Filtrate, dry in vacuum, weigh and calculate the yield, then put it away.

Take a small sample, dissolve it in water and test it for chloride and manganese(II) ions

SYNTHESIS OF TRIS(ACETYLACETONATE)MANGANESE(III)
Mn(acac)₃ (acac = CH₃COCHCOCH₃⁻) (11)



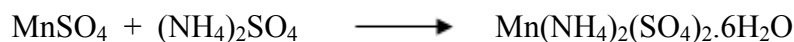
In a 250 ml beaker dissolve 2 g of manganese(II) chloride tetrahydrate and 5.2 g of sodium acetate in 80 ml of water. To this solution add slowly and with agitation 8.4 ml of acetylacetone (2,4-pentanedione), resulting in a 2 phase system.

Prepare a solution with 0.4 g of potassium permanganate in 20 ml of water. Add this solution to the previously prepared mixture slowly and with agitation

Then prepare a solution with 5.2 g of sodium acetate in 20 ml of water. Add this solution to the previously prepared mixture in small fractions and with agitation. Warm up the resulting mixture to 60 °C for 10 minutes.

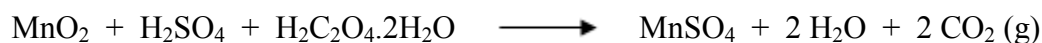
Cool it down in an ice bath and filtrate in vacuum. Wash the product with iced water. When the product is dry, weigh, calculate the yield and put it away.

SYNTHESIS OF AMMONIUM MANGANESE SULPHATE HEXAHYDRATE $\text{Mn}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (12)

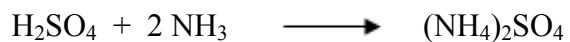


To a 250 ml beaker with a 25 ml 2M solution of sulphuric acid 2.5 g of oxalic acid dehydrate are added. To dissolve the solid completely warm the mixture a little bit if necessary.

To this solution add 2 g of manganese dioxide in small portions. In this reaction carbon dioxide is liberated and hence some fizziness will happen. If necessary filtrate.



In another beaker prepare 3.3 g ammonium sulphate in solution. For this neutralise a 1 M solution of sulphuric acid with the appropriate amount of concentrated ammonia, use some drops of methyl orange as indicator.

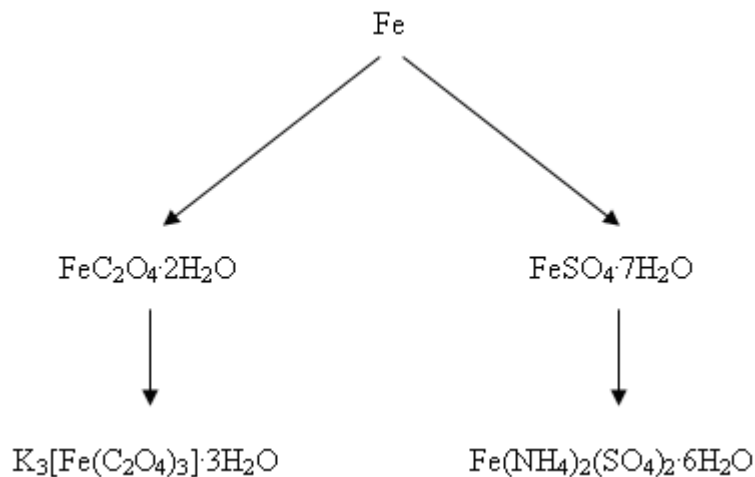


Mix both solution and concentrate up to 20 ml, leave it to cool down until the salt crystallises.

Filtrate, dry in vacuum, weigh and calculate the yield, then put it away.

Take a small sample of the product and dissolve it in water and test for sulphate and manganese ions.

COMPOUNDS OBTAINED STARTING FROM METALLIC Fe



SYNTHESIS OF IRON(II) SULPHATE HEPTAHYDRATE $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (7)



We will obtain a salt attacking the metal with the appropriate acid. With the starting metal there is, as impurity, another one which is inert under reaction conditions.

In a tripod with a wire gauze put on top the distillation flask. In this flask add the starting material, this is a 5 g mixture of iron and copper. Put the Thistle funnel on top and using this add 70 ml of 20% sulphuric acid, slowly and warming little by little the flask.

The liberated hydrogen passes through the bubbler to the Erlenmeyer. In this Erlenmeyer there has to be a solution of iodine and potassium iodide, where the evacuating tube is inserted. Do the set up of Figure 1 on page - 32 -.

The reaction is maintained until the attack is finished. Then the set up is taken down and put the contents of the Erlenmeyer flask away to perform the next part of the demonstration.

Filtrate the contents of the Erlenmeyer flask, and dry the solid (copper) and weigh it. Calculate how much iron was there in the starting material.

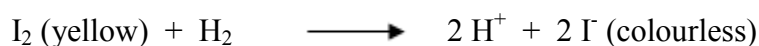
Put the filtrate in a beaker and put an iron nail in it (to avoid the oxidation to Fe^{3+}) and concentrate it in a water bath until the volume is less than half of the volume that would correspond to a saturated solution of the salt in cold water (consult the value of the solubility in the tables). Let the solution cool down to crystallise and if it is necessary concentrate more. Filter the crystals thus obtained, dry in vacuum, calculate the yield with respect to the iron in the starting material, and put it away.

Take a small sample of the product, dissolve it in water and test it for sulphate and iron(II) ions

Comparison of the reducing capacity between molecular hydrogen and newly created hydrogen (2)

Put 2 ml of the iodine/iodide mixture of the Erlenmeyer in a test tube. Add a zinc shot and a little bit of hydrochloric acid. Examine the result. Put back the remaining solution into the starting material bottle.

The iodine solution (yellow) should, after bubbling hydrogen get reduced to iodide:



$$\Delta \epsilon^0 = \epsilon^0(\text{I}_2/\text{I}^-) - \epsilon^0(\text{H}^+/\text{H}_2) = 0,54 - (0,00) = 0.54 \text{ V} > 0$$

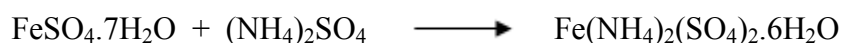
According to the thermodynamics the reaction should happen, but it is so slow that in practice it is not observed.

But the reaction will happen rapidly if in the environment there is newly produced hydrogen (atomic), by the reaction of metallic zinc with hydrochloric acid. In the first step of the reduction of H^+ on Zn hydrogen atoms are created (newly created hydrogen) and these react fast with iodine, avoiding the breaking of the H-H bond.

In the test tube the following is seen, the solution loses its colour according to the next equation.

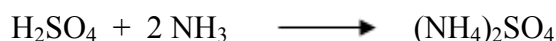


SYNTHESIS OF MOHR'S SALT $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (7,9)



Put 5 g of $FeSO_4 \cdot 7H_2O$ and dissolve them in 25 ml of a water solution of 5 ml 1 M sulphuric acid.

In another beaker neutralise 25 ml 1 M sulphuric acid with the appropriate amount of concentrated ammonia, using few drops of methyl orange as indicator.



Mix both solutions and concentrate by boiling (at the beginning directly and then, when the volume has been reduced to half, in a water bath, to avoid splashing of the solid) until you see that the viscosity of the solution has increased.

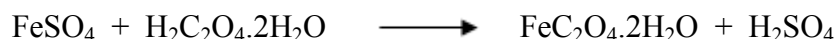
Then let it to cool down to start the crystallisation of the salt.

Filtrate, weigh, calculate the yield and put it away.

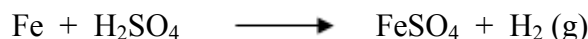
If necessary recrystallise. For this, dissolve in the minimum amount of water and add 5 ml of concentrated sulphuric acid. Warm the resulting solution and concentrate, let it cool down, filtrate, dry, calculate the yield and put it away.

Take a small sample of the product, dissolve it in water and test for sulphate and iron(II) ions.

SYNTHESIS OF IRON(II) OXALATE DIHYDRATE $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (5,12)



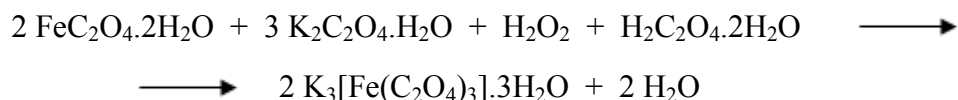
Put 2 g of iron in a 200 ml beaker and attack it with 60 ml of 1 M sulphuric acid. To accelerate the attack warm it up but maintain the original volume (adding distilled water)



Once the attack has finished, filtrate. To get all of the solution wash the beaker and the filter paper with a minimum amount of water. Put the filtrate in a 400 ml beaker and warm it up.

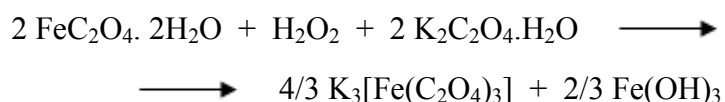
To the warm solution add the appropriate amount of a 10 % solution of oxalic acid, so that the relationship $\text{C}_2\text{O}_4^{2-}/\text{Fe}^{2+}$ is 1.5. Boil it until a yellow solid is formed and decant, wash the precipitate with 50 ml of boiling water. Decant again the solid and filtrate in vacuum. Wash the solid with hot water, then acetone and let it dry. Weigh, calculate the yield and put it away.

SYNTHESIS OF POTASSIUM TRIS(OXALATE)FERRATE(III) TRIHYDRATE $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ (5,12)

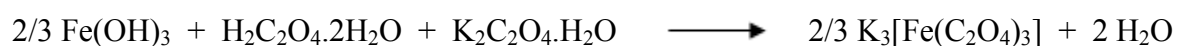


Put in 20 ml of water 6.2 g of potassium oxalate monohydrate. Warm the resulting solution and add 4 g of iron(II) oxalate dihydrate.

With a pipette add 8 ml of a 30 % solution of hydrogen peroxide, slowly and with agitation and maintaining the temperature at 40 °C, more or less. The suspension changes colour when iron(III) hydroxide appears, following the next equation:



This hydroxide is dissolved again by boiling the mixture and adding, drop by drop a solution of 10 % of oxalic acid dihydrate, according to the stoichiometry of the following equation:

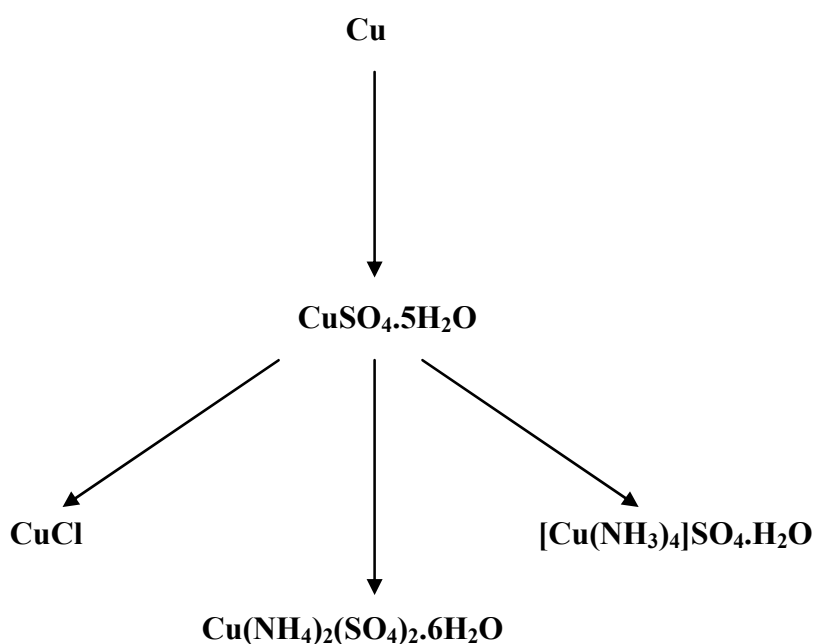


If the precipitate does not completely dissolve add a little bit of excess of oxalic acid, if necessary.

Filtrate the warm solution, and put it in a beaker and when it reaches room temperature add 30 ml of ethanol.

The precipitate thus obtained has to be recrystallised. For this dissolve the solid in the minimum amount of hot water and leave the resulting solution recrystallise overnight. Filtrate and wash the solid with a 1:1 mixture of water:ethanol. Dry the crystals, weigh, calculate the yield and put it away.

COMPOUNDS OBTAINED STARTING FROM METALLIC Cu



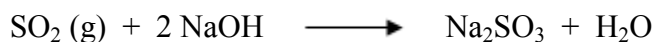
SYNTHESIS OF COPPER(II) SULPHATE PENTAHYDRATE $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (7)



Put 5 g of copper turnings in a distillation flask and set up the set-up in Figure 1 on page - 32 -, and put in the Erlenmeyer flask 100 ml of a 10% solution of sodium hydroxide.

To attack the copper use 40 ml of 12 M sulphuric acid. Add the sulphuric acid slowly through the Thistle funnel and meanwhile warm up the flask with the Bunsen burner

Once the attack has finished, put the Erlenmeyer flask away, don't through it away! You will need this to test the reductive nature of the sulphite ion.



Let the distillation flask to cool down and then add 80 ml of water, and filtrate. Concentrate the filtrate by heating it up and leave less than half of the volume that would correspond to a saturated solution of copper sulphate pentahydrate (consult the tables for the solubility). Let it cool down so the crystallisation can begin. If necessary concentrate more.

Filtrate the crystals in a Buchner funnel and dry in vacuum. Take the filtrate and concentrate again and crystallise as before. Take both sets of crystals, weigh and calculate the overall yield. Keep the product.

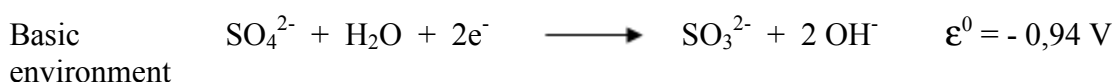
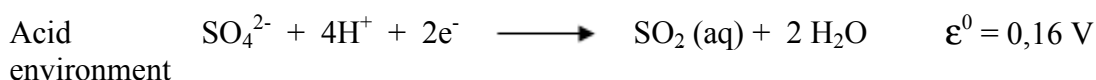
Take a small sample of the product dissolve it in water and test it for sulphate and copper(II) ions.

Exam of the reductive character of the sulphite ion

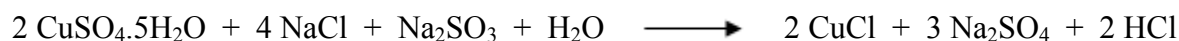
With the solution of the Erlenmeyer perform the next test tube tests:

- In a test tube put 2 ml of a iodine solution, then add the solution of the Erlenmeyer little by little until a colour change happens. Write the reaction down knowing the iodine is yellow and that iodide is colourless.
- In a test tube put 2 ml of a solution of potassium permanganate acidified with few drops of sulphuric acid. Then add drop by drop de solution of the Erlenmeyer until a change in the colour happens. Write the reaction down, knowing that permanganate is purple and that Mn^{2+} is colourless.

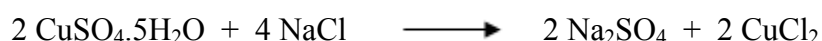
The ion sulphite is a reducing agent. It reduces many substances in water. The potentials show that it is a better reducing agent in a basic environment than in an acid environment. Despite this, it is able to reduce permanganate in an acid environment.



SYNTHESIS OF COPPER(I) CHLORIDE CuCl (10,11)

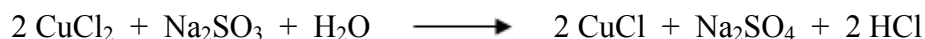


Using 20 ml of distilled water prepare a solution with 4 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and the stoichiometric amount of NaCl according to the following equation:



If necessary warm up a little bit to obtain a clear solution.

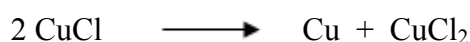
In a 250 ml beaker dissolve 5 g $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ in 20 ml of distilled water. Add this solution to the previously prepared one little by little and with agitation. A white precipitate of CuCl is formed, which comes from the reduction of Cu^{2+} with the strong reducing agent sulphite and its low solubility (see solubility tables).



Prepare sulphurous acid. For this dissolve 0.4 g of Na_2SO_3 in 240 ml of distilled water and add 6 ml HCl 2 M.

Dilute the suspension of copper(I) chloride with half of the solution of sulphurous acid and let the precipitate compact. Decant the solution and filtrate the suspension. Because the solid formed is very fine let, at first, filtrate by gravity, without connecting the pump, and then connect the pump.

CuCl is a white solid, is not very soluble in water and it decomposes partially in it following the next equation:



To avoid this decomposition the solid is washed in the following manner. First with the remaining of the sulphurous acid solution and then using glacial acetic acid until the filtrate appears colourless.

Finish drying the wet solid in the oven. Weigh, calculate the yield and put it away.

This solid in air decomposes into a green basic chloride, $\text{CuCl}_2 \cdot 3\text{Cu}(\text{OH})_2$. Because of this, it has to be kept in a closed recipient. To ascertain that this change happens take a small sample of copper(I) chloride on a watch glass for a few days to see this colour change.

Complexes of copper(I)

Copper(I) is only stable with species like Cl^- , NH_3 , $\text{S}_2\text{O}_3^{2-}$ or Br^- in water, because of the type of complex that is formed, like $[\text{CuCl}_2]^-$.

Put a small sample of copper(I) in 4 ml of distilled water, and then divide the suspension in two to make the test tube tests:

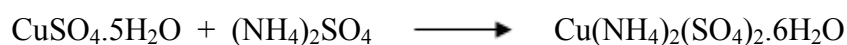
To the first one add concentrated hydrochloric acid, drop by drop, until a change happens.

To the second add concentrated ammonia until the solid dissolves. The ion $[\text{Cu}(\text{NH}_3)_2]^+$ is colourless, and in the presence of oxygen it transforms into the complex $[\text{Cu}(\text{NH}_3)_4]^{2+}$ which is blue.

Explain what happens after adding the hydrochloric acid

After adding the ammonia. Explain what happened and the differences with the previous test.

**SYNTHESIS OF AMMONIUM COPPER(II) SULPHATE
HEXAHYDRATE $\text{Cu}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (11)**

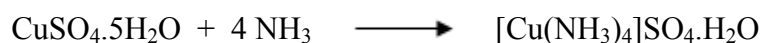


Dissolve 4 g of copper(II) sulphate pentahydrate in 8 ml of water and add a stoichiometric amount of ammonium sulphate.

To dissolve both salts warm it up a little bit, and by cooling it down in a water bath blue crystals are obtained. Filtrate and dry in vacuum. Calculate the yield and put it away.

Take a small sample of the product, dissolve it in water and test it for sulphate and copper ions.

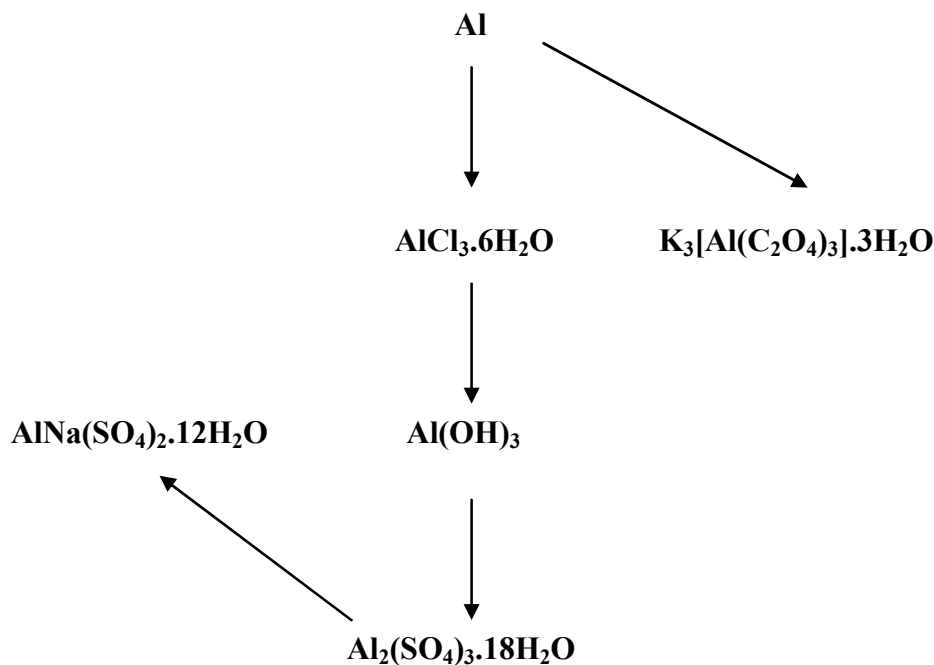
**SYNTHESIS OF TETRAAMINCOPPER(II) SULPHATE
MONOHYDRATE $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ (5,7,10,13)**



Dissolve 4 g of grounded copper(II) sulphate pentahydrate in 15 ml of concentrated ammonia. Add the same volume of ethanol to this solution and put it in an ice bath for 30 minutes.

A dark blue solid is obtained. Filtrate it and wash it with a 1:1 solution of concentrated ammonia and ethyl alcohol. After drying, weigh, calculate the yield and put it away.

COMPOUNDS OBTAINED STARTING FROM METALLIC Al



SYNTHESIS OF ALUMINIUM CHLORIDE HEXAHYDRATE $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (7, 10)



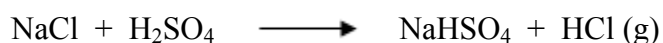
We will use 2 g of aluminium turnings as starting material. In a 250 ml capsule put the aluminium and we will attack it with 50 g of concentrated hydrochloric acid. Adding the acid little by little (the attack takes place better with diluted acid, than with concentrated acid but we will use concentrated acid because the crystallisation process requires a saturated HCl solution) and finally heat slowly until complete reaction. Leave it to cool down and if necessary add concentrated hydrochloric acid.

If necessary filtrate the solution. Pass the filtrate to a 400 ml beaker. The salt we want to obtain is hygroscopic and hence we have to put this solution in a desiccator with calcium

chloride. Make the set-up depicted in Figure 2 on page - 33 - and on top of the liquid put a funnel upside down. This set-up has to be put up inside the fume hood.

Put in the flask 30 g of sodium chloride and in the oil bubbler concentrated sulphuric acid. Take the solution from the desiccator and put it in an ice and water bath, placing them as in the figure.

Add, through the Thistle funnel, concentrated H_2SO_4 slowly, until the stoichiometric amount of 1.5 has been added. Warm up slowly to kick start the reaction, but be careful not to warm it up too much.

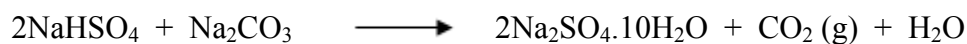


This way the solution of aluminium chloride solution will get saturated with hydrochloric acid and the hexahydrate will crystallise.

Filter the crystals, and then put it in the calcium chloride desiccator. Once dried, weigh, calculate the yield and put them away.

Take a small sample of the solid, dissolve it in water and test it for aluminium(III) and chloride ions.

Synthesis of sodium sulphate decahydrate $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$



Dissolve what is left in the flask with the necessary amount of water. Take 1/10 of the solution and pass it to a 250 ml beaker. We will add in small portions a saturated solution of sodium carbonate to neutralise this solution.

If there is any solid remaining, filtrate and put the filtrate in a beaker. Concentrate and leave it to cool down to crystallise. Filtrate the crystals in the Buchner flask and dry in vacuum. Weigh, calculate the yield and take it away.

Test of the obtained product:

In a dry test tube put a small sample and put a thermometer touching the solid. Put the tube in a beaker with water and clamp it so it doesn't touch the bottom of the flask. Warm the beaker slowly and meanwhile look at the crystals and the temperature changes in the thermometer.

This temperature is the temperature where the hydrate is formed.

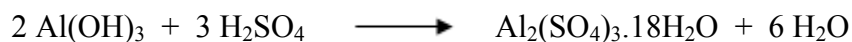
SYNTHESIS OF ALUMINIUM HYDROXIDE Al(OH)_3 (7)



Dissolve 10 g of aluminium chloride hexahydrate in 50 ml of distilled water in a 250 ml beaker. Add 40 ml of 10 % ammonia solution. A gel is formed and it compacts after warming up for 20 minutes.

Leave it cool down, filtrate under vacuum and wash it with warm water. After drying it well the solid is put away, to use it in the synthesis of aluminium sulphate. Calculate how much aluminium hydroxide has been obtained supposing a yield of a 100 %.

SYNTHESIS OF ALUMINIUM SULPHATE OCTADECALHYDRATE **$\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ (7)**

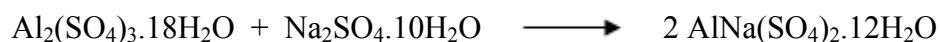


The starting material for this experiment will be the aluminium hydroxide obtained in the previous one. Put this solid in a porcelain capsule and add 15 ml of 7 M sulphuric acid. Warm this mixture up in the fume hood until the solution becomes more viscous. When cooling down a white mass of aluminium sulphate octadecahydrate needs to be formed.

Transfer this solid to a 250 ml beaker and dissolve it in the minimum amount of hot water (30 ml more or less). Concentrate a little bit and let it cool down to room temperature until the sulphate crystallises. Filter in vacuum, dry, weigh, calculate the yield and take it away.

Take a small sample of the solid and dissolve it in water, test it for aluminium and sulphate ions.

SYNTHESIS OF ALUMINIUM AND SODIUM ALUM $\text{AlNa}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ **(7)**



In a 100 ml beaker dissolve 7 g of aluminium sulphate hydrate in 10 ml of distilled water. If necessary warm it up to dissolve completely.

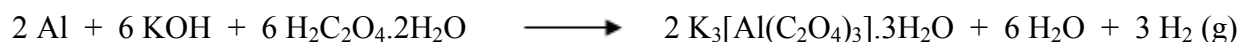
In a 250 ml beaker dissolve the sodium sulphate decahydrate obtained previously. Put the stoichiometric amount of sodium sulphate in 30 ml of water, more or less.

Mix both solutions, concentrate until the final volume is less than half of the volume at the beginning, leave it cool down and leave it a long time to crystallise (it can be a whole day).

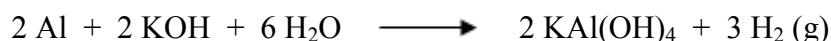
Filtrate the crystals, dry in vacuum, calculate the yield and put it away.

Take a small sample of the solid and test it for aluminium and sulphate ions.

SYNTHESIS OF POTASSIUM TRIS(OXALATE)ALUMINATE(III) TRIHYDRATE $K_3[Al(C_2O_4)_3] \cdot 3H_2O$ (4, 12)

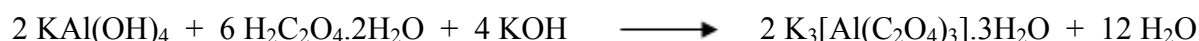


Take 1 g of aluminium turnings and add, little by little, a solution of 6.5 g of potassium hydroxide in 50 ml of water. Hydrogen will be liberated and effervescence will appear.



When the effervescence slows down take the solution to boiling point until no more reaction can be appreciated. Then leave it to cool down and filtrate to eliminate any solid that may remain.

Warm up the solution and add the stoichiometric amount of oxalic acid dihydrate, in small portions. Firstly a precipitate appears, but it dissolves with boiling. If necessary filtrate.



Cool down the solution to room temperature and add 50 ml of ethanol. Put it in an ice bath and filtrate.

Recrystallise this solid, dissolve it in the minimum amount of warm water (more or less 30 ml), concentrate a little bit, let it cool down and leave it for crystals to form. Filtrate in vacuum, weigh, calculate the yield, and take it away.

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APPENDIX 1

OINARRIZKO SEGURTASUN-ARAUAK LABORATEGIAN

1/ Segurtasun-elementuak eta irteerak

Lana hasi baino lehen lantoki eta bere ingurua ezagutu behar dira.

- Irteera nagusiak eta larrialdietakoak lekutu.
- Suitzalgailuak eta beste segurtasun-elementuak (begi-garbitzailea, burusiak...) lekutu.

2/ Begien babesa

BEHARREZKOA DA SEGURTASUN-BETAURREKOAK ERABILTZEA

Laborategian maiz gertatzen dira zenbait istripu eta zipriztin, begietan eragiten dutenak. Gogoratu arriskua norberak erabiltzen dituen produktuetatik ez ezik lankideengandik ere badatorrela.

Ukipen-lenteak ez dira batere gomendagarriak: zipriztinak izanez gero produktua lente eta begiaren artean sar daiteke min handiagoa sor dezakeelarik.

Produktu kimiko bat begietara erortzen bada, berehala garbitu begi-garbitzailea erabiliz (hamar segundu pasa baino lehen) 15 minutuz. Irakasleari abisatu.

3/ Laborategiko jantzia

Laborategian zipriztinak saihestezinak dira. Bata eramatea beharrezkoa da.

- Praka motzak, minigona edo galtzerdiak janztea ez da gomendagarria.
- Oinetako itxiak eramatea gomendagarria da eta ez sandaliak janztea.
- Ile luzeak arriskua sor dezakeenez gomendatzen da biltzea.
- Zintzilikan dauden lepokoak edo beste apaingarriak ez eraman.

4/ Garbitasun-arauak

Laborategian produktu kimikoek kutsadura sor dezakete. Honez gero saio bat burutu ondoren eta laborategitik atera aurretik eskuak garbitu behar dira.

- Laborategian ez jan, ez edan.
- Erretzea bereziki arriskutsua da laborategian eta produktu kimikoak biltzen diren tokietan.
- Produktu kimiko ezezagunak ez dastatu, ez usaindu ezta inhalatu ere.
- Azala eta produktu kimikoen arteko kontaktua saihestu. Zipristina izanez gero berehala garbitu iturriko ura erabiliz.

5/ Laborategiko tresna eta elementuen erabilpena

Saio bat burutu aurretik erabili beharreko muntaia zuzena dela eta baldintza onean dagoela ziurtatu. Zalantzarik izanez gero irakaslearengana jo.

- Tresna edo ekipo bat nola funtzionatzen duen jakin gabe ez erabili.
- Egoera txarrean dagoen beirazko materiala ez erabili.
- Likidoak ez pipetteatu ahoaz, tresna egokiak erabili.
- Langunea garbi eta txukun mantendu, beroki, poltsarik gabe.
- Flasko guztiek bere etiketa eraman behar dute, produktuaren izena, bere kontzentrazioa eta prestatze-data adierazten dituenak.
- Saioa amaitutzat eman baino lehen sortutako hondakinak ezabatu eta erabilitako materiala garbitu behar dira.
- Irakaslearen baimenik gabe ezin da burutu saioa.

6/ Produktu kimikoak maneiatzea

Produktu kimikoak toxiko, korrosibo, sukoi edo lehergarriak, hau da, arriskutsuak izan daitezke. Honez gero kontu handiz erabili behar dira.

- Produktua lehenengo aldiz erabili baino lehen produktu horren propietateak ezagutu eta bere fitxa toxikologikoa kontsultatu behar da.

- Laborategian arrisku handiena sua denez,ugar askeak saihestu behar dira. Metxeroa erabili behar baldin bada, bere inguruan ez utzi disolbatzaileak ezta sukoiak diren produktuak ere.

- Ontzi itxian dauden produktuak ez berotu, tamaina egokiko irekiera utzi, jendea dagoen kontrako norantzerantz orientatuta dagoena.

- Produktu hegazkorrak edo usain txarrekoak maneiatzeko gas-kanpaia erabili behar da. Langilea gas-kanpaiatik kanpo mantendu behar da.

- Eroritako produktuak berehala garbitu.

- Flasko ikuzleen edukina ura ez denean etiketa egokia eraman behar dute.

GOGORATU LABORATEGIA EZ DELA JOLASTOKIA.

BROMARIK EZ, ZURE ZENTZUA ETA SEN ONA ERABILI.

NORMAS BÁSICAS DE SEGURIDAD EN EL LABORATORIO

1.- Elementos de seguridad y vías de salida

Antes de comenzar el trabajo, es preciso familiarizarse con el lugar de trabajo y sus alrededores:

- Localizar las salidas principales y las de emergencia.
- Localizar los extintores y otros elementos de seguridad (lavajojos, mantas...).

2.- Protección de los ojos

EL USO DE GAFAS DE SEGURIDAD ES OBLIGATORIO

Las salpicaduras y accidentes relacionados con los ojos son muy frecuentes en el laboratorio. Recordar que el peligro, no sólo viene de los productos que uno mismo maneja, sino también de los compañeros.

Las lentes de contacto no son aconsejables en el laboratorio: En caso de salpicaduras, el producto se puede deslizar tras la lente, dificultando su eliminación con el consiguiente riesgo de producir mayores daños.

Si un producto químico salpica los ojos, emplear un lava-ojos, con urgencia, y antes de 10 segundos. El tiempo de lavado debe ser largo, del orden de 15 minutos. Informar al profesor responsable.

3.- Indumentaria en el laboratorio

En un laboratorio, las salpicaduras son inevitables. El empleo de bata es obligatorio.

- No es aconsejable llevar minifalda o pantalones cortos ni medias.
- Es preferible emplear calzado cerrado y evitar las sandalias.
- Los cabellos largos son un riesgo que puede evitarse recogéndolo con una cola.
- Evitar llevar collares o adornos que cuelguen libremente.

4.- Normas de higiene

En el laboratorio, el riesgo de contaminación por productos químicos siempre está presente, lo que hace necesario lavarse las manos después de un experimento y antes de salir del laboratorio.

- No comer ni beber en el laboratorio.
- Resulta especialmente peligroso fumar en los laboratorios y en las zonas donde se almacenan productos químicos.
- No probar, oler ni inhalar productos químicos desconocidos.

- Evitar el contacto de productos químicos con la piel. Si hay alguna salpicadura lavarse inmediatamente con agua corriente.

5.- Empleo de aparatos y elementos del laboratorio

Antes de comenzar un experimento, hay que asegurarse de que el montaje es correcto y está en condiciones de uso. En caso de duda dirigirse al profesor responsable.

- No utilizar un equipo o aparato sin estar familiarizado con su funcionamiento.
- No emplear material de vidrio en malas condiciones.
- No pipettear líquidos con la boca, sino emplear los dispositivos adecuados.
- El área de trabajo debe mantenerse limpia y ordenada, sin abrigos, bolsas...
- Todos los frascos deben ser rotulados con el producto que contienen, su concentración y la fecha de preparación.
- El experimento no está finalizado mientras no se eliminen los residuos y se limpie el material utilizado.
- No se puede realizar un experimento no autorizado por el profesor.

6.- Manipulación de productos químicos

Los productos químicos pueden ser peligrosos por sus propiedades tóxicas, corrosivas, inflamables o explosivas, por lo que deben ser manipulados con precaución.

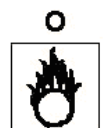
- Cuando se maneja un producto por primera vez es necesario familiarizarse con sus propiedades, y consultar su ficha toxicológica.
- El mayor peligro en un laboratorio es el fuego, por lo que es necesario evitar la presencia de llamas libres. Si la utilización de un mechero es inevitable, asegurarse de que no hay disolventes o productos inflamables en los alrededores.
- No calentar productos en un recipiente cerrado. Hay que dejar una abertura de un tamaño adecuado, y dirigirla en dirección contraria a uno mismo y a las personas cercanas.
- Cuando se manejen productos volátiles o con olor molesto es aconsejable emplear la vitrina de gases. El operador debe mantenerse en el exterior de la vitrina.
- Limpiar inmediatamente los productos derramados.
- Los frascos lavadores que no contengan agua deben rotularse de forma inequívoca.

**RECUERDA QUE EL LABORATORIO NO ES UN LUGAR DE JUEGOS.
EVITA LAS BROMAS Y HAZ USO DE TU JUICIO Y SENTIDO COMUN**

APPENDIX 2: PIKTOGRAMAK - PICTOGRAMAS



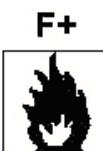
Lehergarria – Explosivo



Errekaria – Comburente



Sukoia – Fácilmente inflamable



Oso sukoia – Extremadamente inflamable



Toxikoa - Tóxico



Oso toxikoa – Muy tóxico



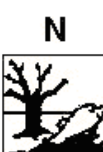
Korrosiboa – Corrosivo



Kaltegarria – Nocivo



Narritagarria – Irritante



Ingurugunerako arriskutsua – Peligroso para el medio ambiente

APPENDIX 3

R ESALDIAK

PROPIETATE TOXIKOLOGIKOEI ETA OSASUNEAN IZAN DITZAKETEN ERAGIN ZEHATZEI BURUZKO ESALDIAK

- R1 Lehergarria lehorrean.
- R2 Talka, marruskadura, sute edo bestelako ignizio-iturri bidez lehertzeko arriskua.
- R3 Talka, marruskadura, sute edo bestelako ignizio-iturri bidez lehertzeko arrisku handia.
- R4 Oso sentikorrak diren metalezko konposatu lehergarriak eratzten ditu.
- R5 Berotuz gero, lehertzeko arriskua.
- R6 Airea ukituz gero edo ukitu gabe lehertzeko arriskua.
- R7 Suteak sor ditzake.
- R8 Erregaiak ukituz gero, sute-arriskua.
- R9 Erregaiekin nahastuz gero, lehertzeko arriskua.
- R10 Sukoia.
- R11 Nahiko sukoia.
- R12 Oso sukoia.
- R14 Uraren aurrean bortizki erreakzionatzen du.
- R15 Uraren aurrean erreakzionatzen du oso sukoiak diren gasak askatuz.
- R16 Sustantzia erregarriekin nahastuz gero, leher daiteke.
- R17 Airea ukituz gero, berez su hartzen du.
- R18 Berau erabiltzerakoan aire-lurrun nahasketa lehergarriak/sukoiak sor ditzake.
- R19 Peroxido lehergarriak sor ditzake.
- R20 Inhalazio bidez kaltegarria.
- R21 Kaltegarria, azala ukituz gero.
- R22 Kaltegarria, irentsiz gero.
- R23 Toxikoa, inhalatuz gero.
- R24 Toxikoa, azala ukituz gero.
- R25 Toxikoa, irentsiz gero.
- R26 Oso toxikoa, irentsiz gero.
- R27 Oso toxikoa, azala ukituz gero.
- R28 Oso toxikoa, irentsiz gero.
- R29 Gas toxikoak askatzen ditu, ura ukituz gero.
- R30 Berau erabiltzerakoan sua erraz har dezake.

- R31 Gas toxikoak askatzen ditu, azidoak ukituz gero.
- R32 Oso toxikoak diren gasak askatzen ditu, azidoak ukituz gero.
- R33 Eragin metagarrien arriskua.
- R34 Erredurak sortzen ditu.
- R35 Erredura larriak sortzen ditu.
- R36 Begiak erretzen ditu.
- R37 Arnasbideak erretzen ditu.
- R38 Azala erretzen du.
- R39 Ondorio itzulezin oso larriak sortzeko arriskua.
- R40 Minbizia sor dezake.
- R41 Begi-lesio larriak sortzeko arriskua.
- R42 Sentikor bihurtzeko aukera, inhalatuz gero.
- R43 Sentikor bihurtzeko aukera, azala ukituz gero.
- R44 Lehertzeko arriskua, esparru itxian berotuz gero.
- R45 Minbizia sor dezake
- R46 Alterazio genetiko hereditarioak sor ditzake
- R48 Osasunerako ondorio larriak ekartzeko arriskua, eraginpean luze egonez gero
- R49 Minbizia sor dezake, inhalatuz gero
- R50 Oso toxikoa uretako bizidunentzat
- R51 Toxikoa uretako bizidunentzat
- R52 Kaltegarria uretako bizidunentzat
- R53 Epe luzera eragin negatiboak izan ditzake ingurune urtarrean
- R54 Toxikoa florarentzat
- R55 Toxikoa faunarentzat
- R56 Toxikoa lurreko organismoentzat
- R57 Toxikoa erleentzat
- R58 Epe luzera ingurumenean eragin negatiboak izan ditzake
- R59 Arriskutsua ozono-geruzarentzat
- R60 Emankortasunari kalte egin diezaioke
- R61 Haurdunaldian fetuari eragin kaltegarriak sortzeko arriskua
- R62 Emankortasunari kalte egiteko arriskua izan dezake
- R63 Haurdunaldian fetuari eragin kaltegarriak sortzeko arriskua izan dezake
- R64 Amaren esneaz elikatutako umeei kalte egin diezaieke
- R65 Kaltegarria: birikei kalte egin diezaieke, irentsiz gero
- R66 Maiz eraginpean egoteak lehortasuna edo azalean arrailak sortzea eragin dezake
- R67 Lurrunak inhalatzea logura eta bertigoia sor ditzake

R68 Ondorio itzulezinak izateko aukera

OHARRA: R esaldien ondoko konbinazioak ere erabil daitezke:

R14/15, R15/29, R20/21, R20/22, R20/21/22, R21/22, R23/24, R23/25, R23/24/25, R24/25, R26/27, R26/28, R26/27/28, R27/28, R36/37, R36/38, R36/37/38, R37/38, R39/23, R39/24, R39/25, R39/23/24, R39/23/25, R39/24/25, R39/23//24/25, R39/26, R39/27, R39/28, R39/26/27, R39/26/28, R39/27/28, R39/26/27/28, R42/43, R48/20, R48/21, R48/22, R48/20/21, R48/20/22, R48/21/22, R48/20/21/22, R48/23, R48/24, R48/25, R48/23/24, R48/23/25, R48/24/25, R48/23/24/25, R50/53, R51/53, R52/53, R68/20, R68/21, R68/22, R68/20/21, R68/20/22, R68/21/22, R68/20/21/22.

**FRASES R: NATURALEZA DE LOS RIESGOS ESPECIFICOS ATRIBUIDOS A
LAS SUSTANCIAS Y PREPARADOS PELIGROSOS**

- R1 Explosivo en estado seco.
- R2 Riesgo de explosión por choque, fricción, fuego u otras fuentes de ignición.
- R3 Alto riesgo de explosión por choque, fricción, fuego u otras fuentes de ignición.
- R4 Forma compuestos metálicos explosivos muy sensibles.
- R5 Peligro de explosión en caso de calentamiento.
- R6 Peligro de explosión, en contacto o sin contacto con el aire.
- R7 Puede provocar incendios.
- R8 Peligro de fuego en contacto con materias combustibles.
- R9 Peligro de explosión al mezclar con materias combustibles.
- R10 Inflamable.
- R11 Fácilmente inflamable.
- R12 Extremadamente inflamable.
- R13 Gas licuado extremadamente inflamable.
- R14 Reacciona violentamente con el agua.
- R15 Reacciona con el agua liberando gases extremadamente inflamables.
- R16 Puede explosionar en mezcla con sustancias comburentes.
- R17 Se inflama espontáneamente en contacto con el aire.
- R18 Al usarlo pueden formarse mezclas aire-vapor explosivas/inflamables.
- R19 Puede formar peróxidos explosivos.
- R20 Nocivo por inhalación.
- R21 Nocivo en contacto con la piel.
- R22 Nocivo por ingestión.
- R23 Tóxico por inhalación.
- R24 Tóxico en contacto con la piel.
- R25 Tóxico por ingestión.
- R26 Muy tóxico por inhalación.
- R27 Muy tóxico en contacto con la piel.
- R28 Muy tóxico por ingestión.

- R29 En contacto con agua libera gases tóxicos.
- R30 Puede inflamarse fácilmente al usarlo.
- R31 En contacto con ácidos libera gases tóxicos.
- R32 En contacto con ácidos libera gases muy tóxicos.
- R33 Peligro de efectos acumulativos.
- R34 Provoca quemaduras.
- R35 Provoca quemaduras graves.
- R36 Irrita los ojos.
- R37 Irrita las vías respiratorias.
- R38 Irrita la piel.
- R39 Peligro de efectos irreversibles muy graves.
- R40 Posibles efectos cancerígenos.
- R41 Riesgo de lesiones oculares graves.
- R42 Posibilidad de sensibilización por inhalación.
- R43 Posibilidad de sensibilización en contacto con la piel.
- R44 Riesgo de explosión al calentarlo en ambiente confinado.
- R45 Puede causar cáncer.
- R46 Puede causar alteraciones genéticas hereditarias.
- R47 Puede causar malformaciones congénitas.
- R48 Riesgo de efectos graves para la salud en caso de exposición prolongada.
- R49 Puede causar cáncer por inhalación.
- R50 Muy tóxico para los organismos acuáticos.
- R51 Tóxico para los organismos acuáticos.
- R52 Nocivo para los organismos acuáticos.
- R53 Puede provocar a largo plazo efectos negativos en el medio ambiente acuático.
- R54 Tóxico para la flora.
- R55 Tóxico para la fauna.
- R56 Tóxico para los organismos del suelo.
- R57 Tóxico para las abejas.
- R58 Puede provocar a largo plazo efectos negativos en el medio ambiente.
- R59 Peligroso para la capa de ozono.
- R60 Puede perjudicar la fertilidad.

- R61 Riesgo durante el embarazo de efectos adversos para el feto.
- R62 Posible riesgo de perjudicar la fertilidad.
- R63 Posible riesgo durante el embarazo de efectos adversos para el feto.
- R64 Puede perjudicar a los niños alimentados con leche materna.
- R65 Nocivo. Si se ingiere puede causar daño pulmonar.
- R66 La exposición repetida puede provocar sequedad o formación de grietas en la piel.
- R67 La inhalación de vapores puede provocar somnolencia y vértigo
- R68 Posibilidad de efectos irreversibles.

NOTA: También se pueden utilizar las siguientes combinaciones de frases R:

R14/15, R15/29, R20/21, R20/22, R20/21/22, R21/22, R23/24, R23/25, R23/24/25, R24/25, R26/27, R26/28, R26/27/28, R27/28, R36/37, R36/38, R36/37/38, R37/38, R39/23, R39/24, R39/25, R39/23/24, R39/23/25, R39/24/25, R39/23/24/25, R39/26, R39/27, R39/28, R39/26/27, R39/26/28, R39/27/28, R39/26/27/28, R42/43, R48/20, R48/21, R48/22, R48/20/21, R48/20/22, R48/21/22, R48/20/21/22, R48/23, R48/24, R48/25, R48/23/24, R48/23/25, R48/24/25, R48/23/24/25, R50/53, R51/53, R52/53, R68/20, R68/21, R68/22, R68/20/21, R68/20/22, R68/21/22, R68/20/21/22.

R13 y R47: son frases obsoletas.

R40: hasta 2001 esta frase R fue usada para posibles riesgos mutagénicos o teratogénicos, ahora se utiliza la frase R68.

APPENDIX 4

S ESALDIAK

SUSTANTZIA ETA NAHASTE ARRISKUTSUEI DAGOZKIEN ZUHURTASUN-AHOLKUAK

- S1 Giltzapean gorde.
- S2 Aurren helmenetik kanpo gorde.
- S3 Toki freskoan gorde.
- S4 Jendea dagoen lokaletatik urrun eduki.
- S5 Fabrikatzaileak zehazten duen likido egokian gorde.
- S6 Fabrikatzaileak zehazten duen gas geldoan gorde.
- S7 Ontzia guztiz itxita utzi.
- S8 Ontzia toki lehorrean gorde.
- S9 Ondo aireztatuta dagoen lekuan gorde ontzia.
- S12 Ez itxi ontzia hermetikoki.
- S13 Elikagai, edari eta eta pentsuetatik urrun gorde.
- S14 Fabrikatzaileak zehazten dituen material elkartezinetatik urrun gorde.
- S15 Berotik urrun gorde.
- S16 Sugar edo txinpart iturritik urrun gorde. Ez erre.
- S17 Sukoiak diren materialetatik urrun gorde.
- S18 Ontzia ireki eta maneiatu zuhertasunez.
- S20 Erabiltzen den bitartean ez jan, ez edan.
- S21 Erabiltzen den bitartean ez erre.
- S22 Hautsa ez inhalatu.
- S23 Gasak/ keak/ lurrinak/ aerosolak ez inhalatu (fabrikatzaileak zehazten duena).
- S24 Azalarekiko kontaktua saihestu.
- S25 Begiekiko kontaktua saihestu.

- S26 Begiak ukituz gero, berehala garbitu ur-kopuru handiz eta sendagilearengana joan.
- S27 Zikindutako arropa berehala kendu.
- S28 Azala ukituz gero, berehala garbitu fabrikatzaileak zehazten duen produktu-kopuru handiz.
- S29 Hondakinak ez bota harraskatik.
- S30 Produktu honi ezin zaio ura erantsi, inoiz ez.
- S33 Karga elektrostatikoak ez metatu.
- S35 Produktuaren hondakin guztiak eta ontziak kontu handiz ezabatu
- S36 Babes arropa egokia erabili.
- S37 Eskularru egokiak erabili.
- S38 Aireztapen nahikoa ez badago arnasa hartzeko ekipo egokia erabili
- S39 Aurpegi eta begietarako babesa erabili
- S40 Produktu honek kutsatutako lurra eta objektuak garbitzeko erabili fabrikatzaileak zehazten duen sustantzia.
- S41 Sutean edo/eta leherketan sortutako keak ez inhalatu
- S42 Lainoztatze/haustutzeak diren bitartean arnasa hartzeko ekipo egokia erabili.
[Fabrikatzailea zehazten duena]
- S43 Sutean fabrikatzaileak zehazten dituen itzaltzeko bideak erabili (urak arriskua handitzen badu honako hau erantsi: ura ez erabili, inoiz ez)
- S45 Istripua edo ondoezina izatekotan, berehala joan sendagilearengana (posible bada etiketa erakutsi)
- S46 Irentsiz gero, sendagilearengana joan eta etiketa edo ontzia erakutsi
- S47 Dagokion tenperaturaren azpitik gorde (fabrikatzaileak zehazten duena)
- S48 Heze gorde (fabrikatzaileak zehazten duen ingurunean)
- S49 Jatorrizko ontzian bakarrik gorde
- S50 Ez nahastu fabrikatzaileak zehazten duen sustantziarekin
- S51 Ondo aireztatutako tokietan bakarrik erabili
- S52 Jendea dagoen tokietako azalera handietan ez erabili

- S53 Eraginpean egotea saihestu (erabili baino lehen argibideak irakurri)
- S56 Sustantzia hau eta bere ontzia bota, hondakin berezi edo arriskutsuetarako bilketa puntu publiko batean
- S57 Segurtasun-ontzi egokia erabili, inguruneko kutsadura saihesteko
- S59 Fabrikatzaile edo hornitzailearengana jo, berreskuratze/birziklatzeari buruzko informazioa jasotzeko
- S60 Produktu eta bere ontzia ezabatu hondakin arriskutsu gisa
- S61 Ingururunerako askapena saihestu. Argibide zehatzak edo segurtasun fitxa begiratu.
- S62 Irentsiz gero, botaka ez eginarazi: sendagilearengana joan eta etiketa edo ontzia erakutsi
- S63 Inhalazioaz istripua izatekotan, biktima eraman kutsatutako tokitik urrun eta geldirik mantendu
- S64 Irentsiz gero, ahoa urez garbitu (kontziente baldin badago bakarrik)

OHARRA: S esaldien ondoko konbinazioak ere erabil daitezke:

S1/2, S3/7, S3/9/14, S3/9/14/49, S3/9/49, S3/14, S7/8, S7/9, S7/47, S20/21, S24/25, S27/28, S29/35, S29/56, S36/37, S36/37/39, S36/39, S37/39, S47/49.

FRASES S: CONSEJOS DE PRUDENCIA RELATIVOS A LAS SUSTANCIAS Y PREPARADOS PELIGROSOS

- S1 Consérvese bajo llave.
- S2 Manténgase fuera del alcance de los niños.
- S3 Consérvese en lugar fresco.
- S4 Manténgase lejos de locales habitados.
- S5 Consérvese en ... (líquido apropiado a especificar por el fabricante).
- S6 Consérvese en ... (gas inerte a especificar por el fabricante).
- S7 Manténgase el recipiente bien cerrado.
- S8 Manténgase el recipiente en lugar seco.
- S9 Consérvese el recipiente en lugar bien ventilado.
- S10 Mantener el contenido húmedo.
- S11 Evitar el contacto con el aire.
- S12 No cerrar el recipiente herméticamente.
- S13 Manténgase lejos de alimentos, bebidas y piensos.
- S14 Consérvese lejos de ... (materiales incompatibles a especificar por el fabricante).
- S15 Conservar alejado del calor.
- S16 Conservar alejado de toda llama o fuente de chispas - No fumar.
- S17 Manténgase lejos de materiales combustibles.
- S18 Manipúlese y ábrase el recipiente con prudencia.
- S20 No comer ni beber durante su utilización.
- S21 No fumar durante su utilización.
- S22 No respirar el polvo.
- S23 No respirar los gases/humos/vapores/aerosoles [denominación(es) adecuada(s) especificar por el fabricante].
- S24 Evítese el contacto con la piel.
- S25 Evítese el contacto con los ojos.
- S26 En caso de contacto con los ojos, lávense inmediata y abundantemente con agua y acúdase a un médico.
- S27 Quítese inmediatamente la ropa manchada o salpicada.
- S28 En caso de contacto con la piel, lávese inmediata y abundantemente con ... (productos a especificar por el fabricante).

- S29 No tirar los residuos por el desagüe.
- S30 No echar jamás agua a este producto.
- S31 Mantener lejos de materiales explosivos.
- S33 Evítense la acumulación de cargas electrostáticas.
- S34 Evítense golpes y rozamientos.
- S35 Elimínense los residuos del producto y sus recipientes con todas las precauciones posibles.
- S36 Usese indumentaria protectora adecuada.
- S37 Usense guantes adecuados.
- S38 En caso de ventilación insuficiente, úsese equipo respiratorio adecuado.
- S39 Usese protección para los ojos/la cara.
- S40 Para limpiar el suelo y los objetos contaminados por este producto, úsese ... (a especificar por el fabricante).
- S41 En caso de incendio y/o de explosión, no respire los humos.
- S42 Durante las fumigaciones/pulverizaciones, úsese equipo respiratorio adecuado [denominación (es) adecuada(s) a especificar por el fabricante].
- S43 En caso de incendio, utilizar ... (los medios de extinción los debe especificar el fabricante). (Si el agua aumenta el riesgo, se deberá añadir: "No usar nunca agua").
- S44 En caso de malestar, acudir al médico (si es posible, muéstrele la etiqueta).
- S45 En caso de accidente o malestar, acúdase inmediatamente al médico (si es posible, muéstresele la etiqueta).
- S46 En caso de ingestión, acúdase inmediatamente al médico y muéstrele la etiqueta o el envase.
- S47 Consérvese a una temperatura no superior a ... °C (a especificar por el fabricante).
- S48 Consérvese húmedo con ... (medio apropiado a especificar por el fabricante).
- S49 Consérvese únicamente en el recipiente de origen.
- S50 No mezclar con ... (a especificar por el fabricante).
- S51 Úsese únicamente en lugares bien ventilados.
- S52 No usar sobre grandes superficies en locales habitados.
- S53 Evítense la exposición - recábense instrucciones especiales antes del uso.
- S54 Procurar el consenso de la autoridad de control de la contaminación antes de descargar en las plantas de tratamiento de aguas de desagües.
- S55 Utilizar las mejores técnicas de tratamiento disponibles antes de descargar a las alcantarillas o al ambiente acuático.
- S56 Elimínense esta sustancia y su recipiente en un punto de recogida pública de residuos especiales o peligrosos.

- S57 Utilícese un envase de seguridad adecuado para evitar la contaminación del medio ambiente.
- S58 Eliminar como residuo peligroso.
- S59 Remitirse al fabricante o proveedor para obtener información sobre su recuperación/reciclado.
- S60 Elimínense el producto y su recipiente como residuos peligrosos.
- S61 Evítese su liberación al medio ambiente. Recábense instrucciones específicas/ficha de datos de seguridad.
- S62 En caso de ingestión no provocar el vómito: acúdase inmediatamente al médico y muéstresele la etiqueta o el envase.
- S63 En caso de accidente por inhalación, alejar a la víctima fuera de la zona contaminada y mantenerla en reposo.
- S64 En caso de ingestión, lavar la boca con agua (solamente si la persona está consciente).

NOTA: También se pueden utilizar las siguientes combinaciones de frases S

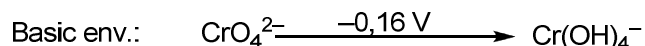
S1/2, S3/7, S3/9/14, S3/9/14/49, S3/9/49, S3/14, S7/8, S7/9, S7/47, S20/21, S24/25, S27/28, S29/35, S29/56, S36/37, S36/37/39, S36/39, S37/39, S47/49.

S10, S11, S31, S34, S44, S54, S55 y S58: son frases obsoletas.

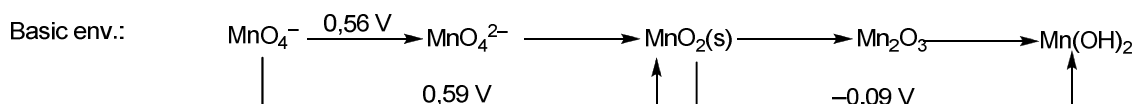
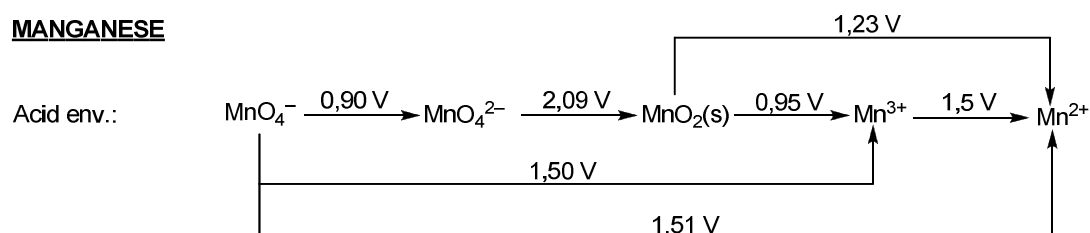
APPENDIX 5

REDUCTION POTENTIALS

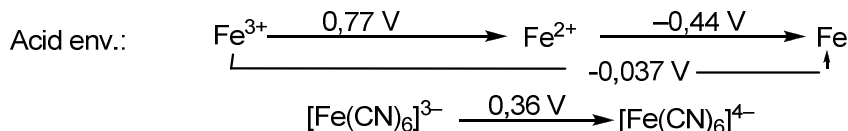
CHROMIUM



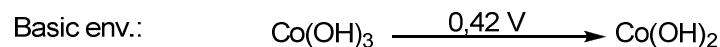
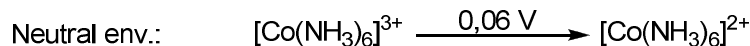
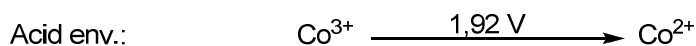
MANGANESE



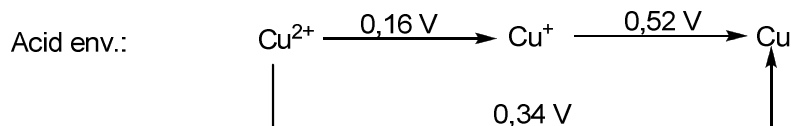
IRON



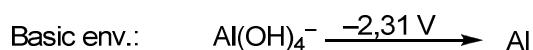
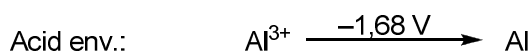
COBALT



COPPER



ALUMINIUM

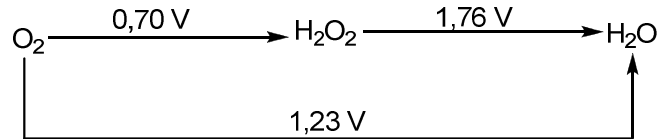


ZINC

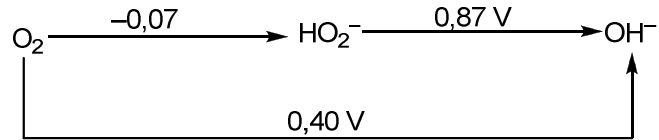


OXYGEN

Acid env.:

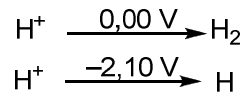


Basic env.:

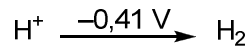


HYDROGEN

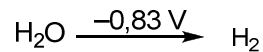
Acid env.:



Neutral env.:

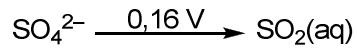


Basic env.:

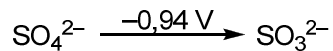


SULPHUR

Acid env.:



Basic env.:



OTHER REDUCTION POTENTIALS

$\text{Cl}_2 / \text{Cl}^-$ 1,36 V

$\text{Br}_2 / \text{Br}^-$ 1,06 V

I_2 / I^- 0,54V

$\text{ClO}^- / \text{Cl}^-$ (basic environment) 0,90 V

$\text{IO}_3^- / \text{I}^-$ (acid environment) 1,09 V

$\text{IO}_4^- / \text{I}^-$ (acid environment) 1,24 V

$\text{NO}_3^- / \text{NO}$ (acid environment) 0,96 V

$\text{NO}_2^- / \text{NO}$ (acid environment) 0,99 V

