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College of science

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Analysis of chromium in chromite ore

This work is submitted in partial fulfillment for the requirement of a bachelor degree in chemistry

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بسم الله الرحمن الرحيم

الآيـة

قال تعالى:

{وَإِنَّ مِنَ الْحِجَارَةِ لَمَا يَتَفَجَّرُ مِنْهُ
الأَنْهَارُ وَإِنَّ مِنْهَا لَمَا يَشَّقَّقُ فَيَخْرُجُ
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ةِ اللّهِ وَمَا اللّهُ بِغَافِلٍ عَمَّا تَعْمَلُونَ
{74}

صدق الله العظيم

(البقرة - الآية 74)

Dedication

WE DEDICATE THIS WORK TO ………..

Our parents ………..

Our family ……

Our teachers

And all those we love

AKNOWLEDGMENTE

Our gratitude and thanks are due to:

Almighty Allah who gave us health to do conduct this work.

The fountain of patience, optimism and hope.

Those who have demonstrated to us what is the beautiful of life our brothers.

The big heart of our dears fathers.

The people who paved our way of science and knowledge.

The taste of the most beautiful moments with our lovely friends.

Our supervisor.

Dr. Adel Alhaj Ahmed

ABSTRACT

This research is concerned with analysis of ore chromite samples from the Blue Nile region. The samples were treated, powdered then subjected to a non-destructive XRF analysis to determine the elemental species. The level of elemental species was found to be Magnesium oxide (17%), Aluminium oxide (7%),ferrous oxide (15%), chromium oxide (50.18%) silicon oxide (9%).

The samples were then fused at 1000 °C in the presence of sodium carbonate then dissolved in distilled water. In this process, the trivalent chromium was converted to hexavalent species, which was then determined using simple classical volumetric technique.

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Chapter One

Introduction

Chapter One

Introduction

1.1 Chromium

Chromium is a chemical element with symbol **Cr** and atomic number 24. It is the first element in Group 6. It is a steely-gray, lustrous, hard and brittle metal[3] which takes a high polish, resists tarnishing, and has a high melting point. The name of the element is derived from the Greek word χρῶμα, *chrōma*, meaning color because many of its compounds are intensely colored.

1.2 History

Weapons found in burial pits dating from the late 3rd century B.C. Qin Dynasty of the Terracotta Army near Xi'an, China have been analyzed by archaeologists. Although buried more than 2,000 years ago, the ancient bronze tips of crossbow bolts and swords found at the site showed unexpectedly little corrosion, possibly because the bronze was deliberately coated with a thin layer of chromium oxide However, this oxide layer was not chromium metal or chrome plating as we know it.

Chromium minerals as pigments came to the attention of the west in the 18th century. On 26 July 1761, Johann Gottlob Lehmann found an orange-red mineral in the Beryozovskoye mines in the Ural Mountains which he named *Siberian red lead*. Though misidentified as a lead compound with selenium and iron components, the mineral was in fact crocoite (*lead chromate*) with a formula of $PbCrO_4$.

In 1770, Peter Simon Pallas visited the same site as Lehmann and found a red lead mineral that had useful properties as a pigment in paints. The use of Siberian red lead as a paint pigment then developed rapidly. A bright yellow pigment made from crocoite also became fashionable.

In 1797, Louis Nicolas Vauquelin received samples of crocoite ore. He produced chromium trioxide (CrO_3) by mixing crocoite with hydrochloric acid. In 1798, Vauquelin discovered that he could isolate metallic chromium by heating the oxide in a charcoal oven, making him the discoverer of the element. Vauquelin was also able to detect traces of chromium in precious gemstones, such as ruby or emerald.

During the 1800s, chromium was primarily used as a component of paints and in tanning salts. At first, crocoite from Russia was the main source, but in 1827, a larger chromite deposit was discovered near Baltimore, United States. This made the United States the largest producer of chromium products till 1848 when large deposits of chromite were found near Bursa, Turkey.

Chromium is also known for its luster when polished. It is used as a protective and decorative coating on car parts, plumbing fixtures, furniture parts and many other items, usually applied by electroplating. Chromium was used for electroplating as early as 1848, but this use only became widespread with the development of an improved process in 1924.

Metal alloys now account for 85% of the use of chromium. The remainder is used in the chemical industry and refractory and foundry industries.

1.3 Chromite

Chromite is an oxide mineral composed of chromium, iron and oxygen ($FeCr_2O_4$). It is a dark gray to black in color with a metallic to submetallic luster and a high specific gravity. It occurs in basic and ultrabasic igneous rocks and in the metamorphic and sedimentary rocks that are produced when chromite-bearing rocks are altered by heat or weathering.

Chromite is important because it is the only economic ore of chromium, an essential element for a wide variety of metal, chemical and manufactured products. Many other minerals contain chromium, but none of them are found in deposits that can be economically mined to produce chromium.

1.3.1 Properties of Chromite

Chromite can be challenging to identify. Several properties must be considered to differentiate it from other metallic ores. Hand specimen identification of chromite requires a consideration of: color, specific gravity, luster, and a characteristic brown streak. The most important clue to identifying chromite is its association with ultrabasic igneous rocks and metamorphic rocks such as serpentinite.

Table 1.1: The physical properties of chromite

Chemical Classification	Oxide
Color	dark gray to black, rarely brownish black
Streak	dark brown
Luster	metallic to submetallic
Diaphaneity	Opaque
Cleavage	None
Mohs Hardness	5.5 to 6
Specific Gravity	4.0 to 5.1 (variable)
Diagnostic Properties	luster, streak
Chemical Composition	$FeCr_2O_4$ with high level of Mg substituting for Fe
Crystal System	Isometric
Uses	an ore of chromium

Chromite is sometimes slightly magnetic. This can cause it to be confused with magnetite. Chromite and ilmenite have very similar properties. Careful observations of hardness streak and specific gravity are required to distinguish these minerals in hand specimens.

1.3.2 Type of rock

1.3.2.1 Stratiform, Podiform and Beach Sands

Small amounts of chromite are found in many types of rock. However, chromite deposits that are large enough for mining are generally found in: 1) stratiform deposits (large masses of igneous rock such as norite or peridotite that slowly crystallized from subsurface magma); 2) podiform deposits (serpentines and other metamorphic rocks derived from the alteration of norite and peridotite); and, 3) beach sands (derived from the weathering of chromite-bearing rocks).

1.3.2.2 STRATIFORM DEPOSITS

Stratiform deposits are large masses of igneous rock that cooled very slowly in subsurface magma chambers. During this slow cooling, chromite and associated minerals crystallized early while the magma was still at a very high temperature. Their crystals then settled to the bottom of the magma chamber to form a layered deposit. Some of the layers in these deposits can contain 50% or more chromite on the basis of weight.

Most of the world's known chromite occurs in two stratiform deposits: the Bushveld Complex in South Africa and the Great Dyke in Zimbabwe. Other important stratiform deposits include: the Stillwater Complex in Montana, the Kemi Complex of Finland, the Orissa Complex of India, the Goias in Brazil, the Mashaba Complex of Zimbabwe and small deposits in Madagascar. Nearly all of these are Precambrian in age.

1.3.3.2 PODIFORM DEPOSITS

Podiform deposits are large slabs of oceanic lithosphere that have been thrust up onto a continental plate. These slabs of rock, also known as "ophiolites," can contain significant amounts of chromite. In these deposits the chromite is disseminated through the rock and not highly concentrated in easy-to-mine layers. Podiform deposits are known in Kazakhstan, Russia, the Philippines, Zimbabwe, Cyprus and Greece.

The first discoveries of podiform chromite deposits were made near Baltimore, Maryland in the early 1800s. These deposits supplied nearly all of the world's chromite until about 1850. These deposits were small and are no longer in production.

1.3.2.3 BEACH SANDS

Chromite is found in beach sands derived from the weathering of chromite-bearing rocks and laterite soils that developed over peridotite. Beach sand rich in chromite and other heavy minerals is sometimes mined, processed to remove heavy minerals, and returned to the environment.

Two facts allow these chromite sands to occasionally contain economic deposits of chromite. First, chromite is one of the more weathering-resistant minerals of peridotite. That causes it to be concentrated in residual soils that form in the weathering zone above chromite-rich rocks. Second, chromite has a higher specific gravity than other minerals in peridotite. This causes it to be selectively transported and deposited by wave and current actions, concentrating it in certain locations at streams and beaches. These deposits are sometimes rich enough and large enough that they can be mined for chromite.

1.4 Classification of ores chromite

1- Refractory ores
2- Metallurgical ores
3- Chemical ores

1.4.1 Metallurgical ores

It requires the percentage of chromium oxide is not less than 48% and thst ferrate to chrome ratio is 1:3. In addition the percentage of silicon is not more than 8% .

1.4.2 Refractory ores

It requires the percentage of chromium oxide is not less than 31% and that the total of chromium oxide and alamonium oxide percentage is about 58%.Also the percentage of ferrate and silicon should not be more than 12% and 8% Consecutively.

1.4.3 Chemical ores

It requires the percentage of chromium oxide is not less than 44% and the 5%.Also that chrome to ferrate ratio is about 1.6%.

1.5 Extraction of chromium

Chromite, $FeCr_2O_4$, is the most commercially useful ore, and is extensively used for extraction of chromium. Usually, chromium is produced from chromite ores in two steps.

1.5.1 First step

In the first step, the ferrochrome is produced through reduction of chromite with coke in an electric arc furnace. A low-carbon ferrochrome can be produced by using ferrosilicon instead of coke as the reductant. This iron/chromium alloy is used directly as an additive to produce chromium-steels which are "stainless" and hard.

1.5.2 Second step

In the second step, chromium metal is produced by the reduction of Cr_2O_3. This is obtained by aerial oxidation of chromite in molten alkali to give sodium chromate, Na_2CrO_4, which is leached out with water, precipitated and then reduced to the Cr(III) oxide by carbon. The oxide can be reduced by aluminium (aluminothermic process) or silicon:

$$Cr_2O_3 + 2Al \rightarrow 2Cr + Al_2O_3$$

$$2Cr_2O_3 + 3Si \rightarrow 4Cr + 3SiO_2$$

The main use of the chromium metal so produced is in the production of nonferrous alloys, the use of pure chromium being limited because of its low ductility at ordinary temperatures. Alternatively, the Cr_2O_3 can be dissolved in sulphuric acid to give the electrolyte used to produce the ubiquitous chromium-plating which is at once both protective and decorative. The sodium chromate produced in the isolation of chromium is itself the basis for the manufacture of all industrially important chromium chemicals. World production of chromite ores approached 12 million tonnes in 1995-

1.6 Chromium properties

1.6.1 Physil procaperties

Table 1.2: Physical properties of chromium

Name, symbol	chromium, Cr
Appearance	silvery metallic
Atomic number	24
Standard atomic weight (±)	51.9961(6)[1]
Element category	transition metal
Group, block	group 6, d-block
Electron configuration	[Ar] 3d5 4s1
per shell	2, 8, 13, 1
Phase	solid
Melting point	2180 K (1907 °C, 3465 °F)
Boiling point	2944 K (2671 °C, 4840 °F)
Density near r.t.	7.19 g/cm3
when liquid, at m.p.	6.3 g/cm3
Heat of fusion	21.0 kJ/mol
Heat of vaporization	347 kJ/mol
Molar heat capacity	23.35 J/(mol·K)
Atomic properties	
Oxidation states	**6**, 5, 4, **3**, 2, 1, −1, −2 (depending on the oxidation state, an acidic, basic, or amphoteric oxide)
Electronegativity	Pauling scale: 1.66
Ionization energies	1st: 652.9 kJ/mol 2nd: 1590.6 kJ/mol 3rd: 2987 kJ/mol (more)
Atomic radius	empirical: 128 pm
Covalent radius	139±5 pm

1.6.2 Chemical properties

Most compounds of chromium are coloured the most important are the chromates and dichromates of sodium and potassium and the potassium and ammonium chrome alums. The dichromates are used as oxidizing agents in quantitative analysis, also in tanning leather. Other compounds are of industrial value; lead chromate is chrome yellow, a valued pigment. Chromium compounds are used in the textile industry as mordants, nd by the aircraft and other industries for anodizing aluminium

1.6.2.1 Halides

Table 1.3: chromium (III) halides

Formula	Colour	MP	M-X (pm)	μ(BM) (b)	Structure
CrF_3	Green	1404	190	-	-
$CrCl_3$	Red-violet	1152	238	-	$CrCl_3$
$CrBr_3$	Green-black	1130	257	-	BiI_3
CrI_3	Black	>500 decomp	-	-	-

Preparations: CrX_3 are prepared from Cr with X_2, dehydration of $CrCl_3.6H_2O$ requires $SOCl_2$ at 650C

Table 1.4 Chromium(II) halides

Formula	**Colour**	**MP**	**μ (BM)**	**Structure**
CrF_2	green	894	4.3	distorted rutile
$CrCl_2$	white	820-824	5.13	distorted rutile
$CrBr_2$	white	844	-	-
CrI_2	red-brown	868	-	-

Preparations: Reduction of CrX_3 with H_2/HX gives CrX_2.

1.6.2.2 Chromium oxides

Table 1.5 chromium oxide

Formula	Colour	Oxidation State	MP	Magnetic Moment
CrO_3	deep red	Cr^{6+}	197decomp	-
Cr_3O_8	-	intermediate	-	-
Cr_2O_5	-	-	-	-
Cr_5O_{12} etc	-	-	-	-
CrO_2	brown-black	Cr^{4+}	300decomp	-
Cr_2O_3	green	Cr^{3+}	2437	- antiferromagnetic < 35 C

Dichromate and chromate equilibria is pH dependent:

$$HCrO_4^- \rightarrow CrO_4^{2-} + H^+ \quad K=10^{-5.9}$$

$$H_2CrO_4 \rightarrow HCrO_4^- + H^+ \quad K=10^{+0.26}$$

$$Cr_2O_7^{2-} + H_2O \rightarrow 2HCrO_4^- \quad K=10^{-2.2}$$

$$HCr_2O_7^- \rightarrow Cr_2O_7^{2-} + H^+ \quad K=10^{+0.85}$$

Hence the variation found for solutions of CrO_3 are:

$pH > 8$ CrO_4^{2-} yellow

pH 2-6 $HCrO_4^-$ and $Cr_2O_7^{2-}$ orange-red

$pH < 1$ $H_2Cr_2O_7$

One of the most obvious characteristics of Cr(III) is that it is acidic i.e it has a tendency to hydrolyse and form polynuclear complexes containing OH^- bridges in a process known as OLATION This is thought to occur by the loss of a proton from coordinated water, followed by coordination of the OH^- to a second catio

$Cr(H_2O)_6]^{3+} \rightarrow [Cr(H_2O)_5(OH)]^{2+} \rightarrow$

H

O

/ \

$[(H_2O)_4Cr \quad Cr(H_2O)_4]^{4+}$ pK=4 etc.

\ /

O

H

The ease with which the proton is removed can be judged by the fact that the hexaaquo ion (pKa ~ 4) is almost as strong as acetic acid. Further deprotonation and polymerization can occur and, as the pH is raised, the final product is hydrated chromium(III) oxide or "chromic hydroxide".

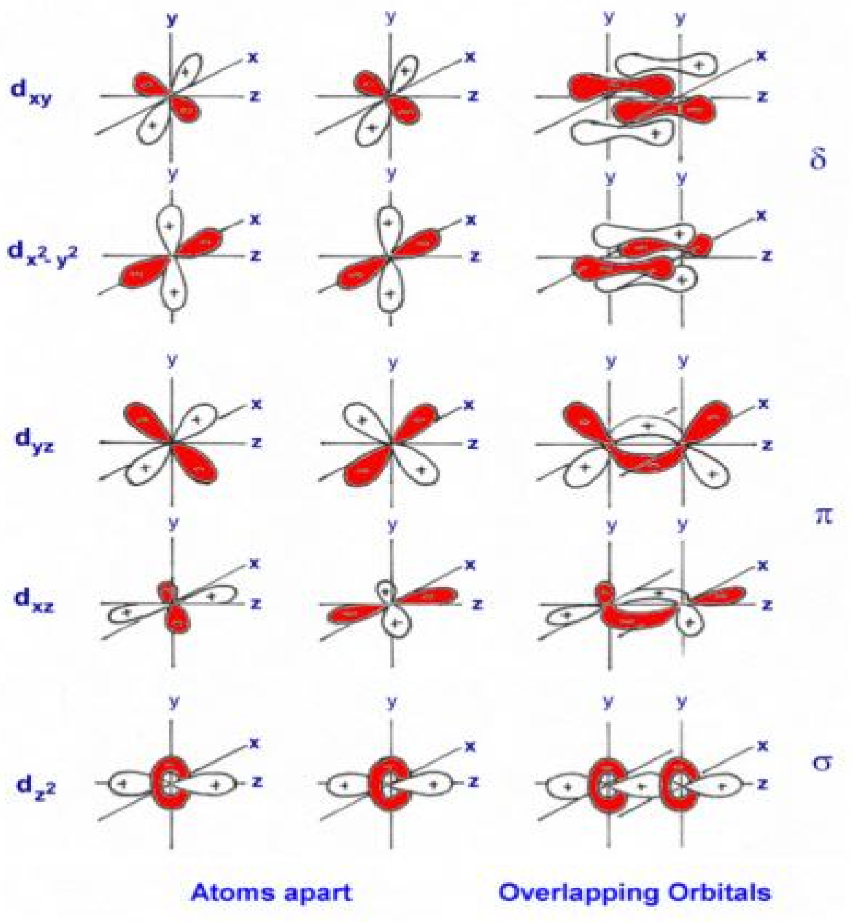
Representative Complexes

The Chromium(III) ion forms many stable complexes and since they are inert are capable of exhibiting various types of isomerism.

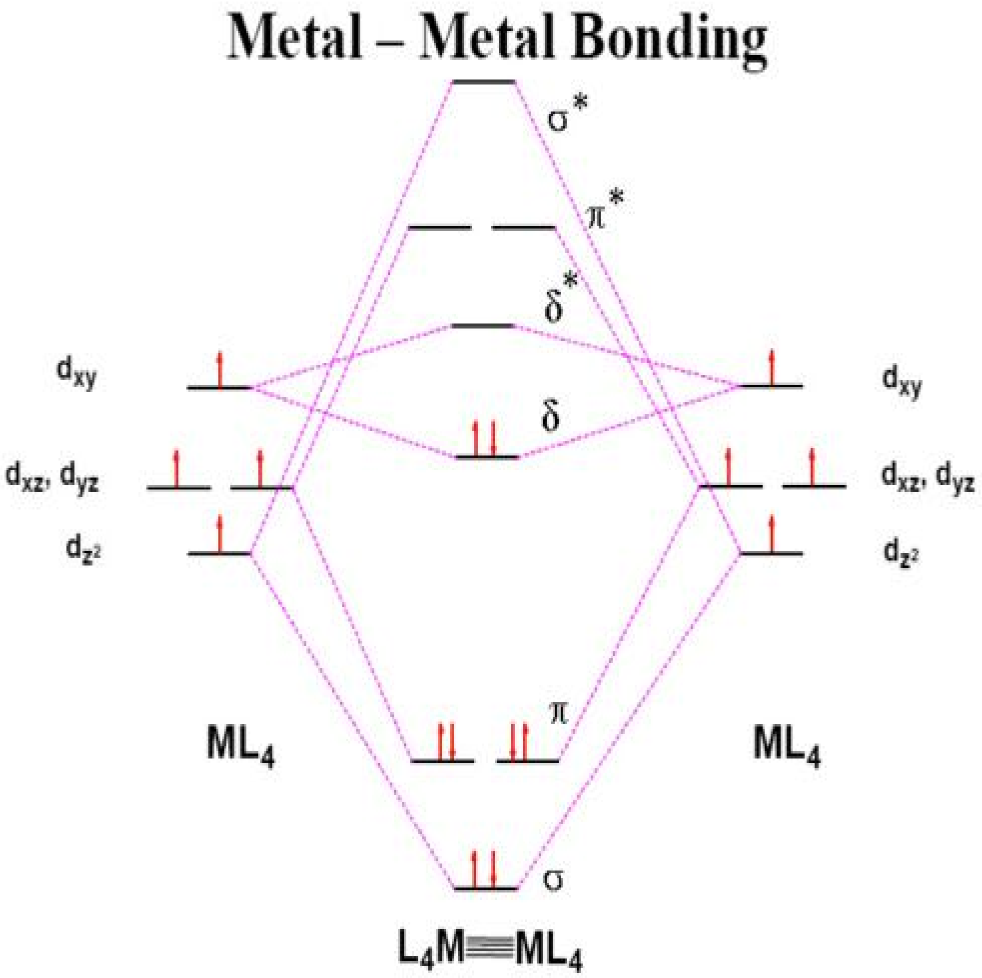
Scheme 1.1:.Anhydrous $CrCl_3$ and hydrated "$CrCl_3.6H_2O$",

Hydrated chromium chloride, "$CrCl_3.6H_2O$", exists as hydrate isomers, including:
the violet $[Cr(H_2O)_6]Cl_3$
the dark green *trans*-$[CrCl_2(H_2O)_4]Cl.2H_2O$ salt shown above, etc.
the pale green $[CrCl(H_2O)_5]Cl_2.H_2O$
Anhydrous $CrCl_3$ reacts with pyridine only in the presence of Zinc powder. This allows a small amount of the Cr(II) ion to be formed, which is very labile but unstable with respect to oxidation back to Cr(III).
$CrCl_3 + pyr/Zn \rightarrow CrCl_3pyr_3$

$[Cr_2(OAc)_4].2H_2O$ is an example of a Cr(II) complex which is reasonably stable in air once isolated. Each Cr(II) ion has 4 d electrons but the complex is found to be diamagnetic which is explained by the formation of a quadruple bond between the two metal ions. The Cr-Cr bond distance in a range of these quadruply bonded species has been found to vary between 195-255 pm.



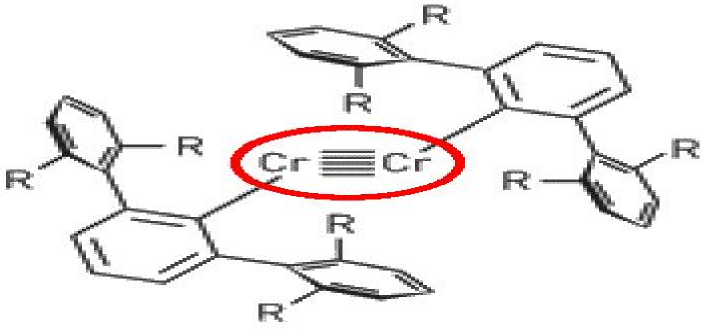
Scheme 1.2: atoms part and overlapping orbitals



Scheme 1.3: metal - metal bonding

1.6.2.3Cr(II) acetate complex

A recent report describes the structure of a Cr complex with a quintuple bond between two Cr(I) ions



Scheme 1.4: Cr(I) - Cr(I) quintuple bonded structure.

1.6.3 Biological properties

Chromium is an essential trace element in mammalian metabolism. In addition to insulin, it is responsible for reducing blood glucose levels, and is used to control certain cases of diabetes. It has also been found to reduce blood cholesterol levels by diminishing the concentration of (bad) low density lipoproteins "LDLs" in the blood. It is supplied in a variety of foods such as Brewer's yeast, liver, cheese, whole grain breads and cereals, and broccoli. It is claimed to aid in muscle development, and as such dietary supplements containing chromium picolinate (its most soluble form), is very popular with body builders. *mer*- isomer of Cr(III) picolinate complex.

Ammonium Reineckate, $NH_4(Cr(NH_3)_2(SCN)_4).H_2O$, is used to test for the presence of dihydromorphinone and other substances generally found in persons involved in substance abuse.

1.7 Chromium uses

More than half the production of chromium goes into metallic products, and about another third is used in refractories. It is an ingredient in several important catalysts. The chief use of chromium is to form alloys with iron, nickel, or cobalt. The addition of chromium imparts hardness, strength, and corrosion resistance to the alloy. In the stainless steels, chromium makes up 10 percent or more of the final composition. Because of its hardness, an alloy of chromium, cobalt, and tungsten is used for high-speed metal-cutting tools. When deposited electrolytically, chromium provides a hard, corrosion-resistant, lustrous finish. For this reason it is widely used as body trim on automobiles and other vehicles. The extensive use of chromite as a refractory is based on its high melting point, its moderate thermal expansion, and the stability of its crystalline structure.

In chromites and chromic salts, chromium has a valence of +3. Most of these compounds are green, but some are red or blue. Chromic oxide (Cr_2O_3) is a green solid. In chromates and dichromates, chromium has a valence of +6. Potassium dichromate ($K_2Cr_2O_7$) is a red, water-soluble solid that, mixed with gelatin, gives a light-sensitive surface useful in photographic processes. The chromates are generally yellow, the best known being lead chromate ($PbCrO_4$), an insoluble solid widely used as a pigment called chrome yellow. Chrome green is a mixture of chrome yellow and Prussian blue.

Chromium is used to harden steel, to manufacture stainless steel, and to form many useful alloys. Much is used in plating to produce a hard, beautiful surface and to prevent corrosion. Chromium gives glass an emerald green colour and iswidely used as a catalyst. The refractory industry has found chromite useful for forming bricks and shapes, as it has a high melting point, moderate thermal expansion, and stability of crystalline structure.

1.8 Chrome production in Sudan

Chrome exploration in Sudan began in the last seventies of the last century and its estimated reserve of about two million tons and the percentage of concentration up 48 – 60% in the case of high quality chrome and chrome as much stock in sudan in 2012 about 50000 tons.

1.9 Chrome production areas in sudan

- Ingeassana Hills
- Hamissana
- Sol Hamed
- Nuba Mountains
- Jebal Rahib
- Jebal El tawil

1.10 previous studies

Technical team from japan studied chromite in ingasana mountains region in five regions and they created chromium oxide ratio as shown in table :

Table 1.6 chromium oxide percentage:

Chromium oxide %	region
25	Gam mine area
41	Romeilik
35 – 43	Gebanit mine area
43 – 78	Komrag area
43 - 59	The Eastern area

.

Chapter two

EXPERIMENTAL

Chapter Two

Experimental

2.1 Materials and Equipment

1. Burette.
2. Beaker.
3. XRF.
4. Conical flask.
5. Volumetric flask.
6. Sensitive Balance.
7. Crucible.
8. Muffle Furnace.
9. Pipette.
10. Heater.
11. Glass rod.
12. Measuring cylinder.
13. Distilled water
14. Potassium Dichromate (1/60 M).
15. Sodium Carbonate.
16. Di phenyl amine Indicator.
17. Phosphoric acid(1:1)
18. Pyrochloric acid
19. Sulfuric acid(2M)
20. Ferrous sulphate (0.1M)

2.2 experimental procedures

2.2.1 preparation of sample

2.5 g of sample was weighted in clean and dry crucible , then 3.55g of sodium carbonate was added to the content , the crucible was put into furnace at 1100 °C for two hours , the content was cooled , transferred to beaker contain of water and covered. After 15 minutes the crucible was cleaned by glass rod, the contents of beaker were heated for 30 minutes after one hour 500 ml of distilled water , 25 ml of pyrochloric acid were added . the solution was left for 24 hours. After that it was transferred to volumetric flask (1000 ml) and completed the volume with distilled water till the mark.

2.2.2 preparation of potassium dichromate standard solution (1/60M)

0.43g of potassium dichromate was weighed and dissolved by distilled water and hydrochloric acid , then transferred quantity to volumetric flask (100ml).

2.2.3 preparation of ferrous sulphate solution (0.1M)

1.7 g of ferrous sulphate was weighed and dissolved by distilled water ,then transferred quantity to volumetric flask(50ml).

2.2.4 standardization of ferrous sulphate

5 ml of ferrous sulphate solution was added to conical flask ,then 5ml of sulfuric acid(2M) , 3ml of phosphoric acid(1:1) and two drops of di phenyl amine indicator were added ,too. The contents of conical flask were titrated against potassium dichromate(1/60M) till end point and results was recorded.

2.2.5 determination of chromium by direct titration

5 ml of ferrous sulphate standardizated solution was added to conical flask, 5ml of sulfuric acid(2M) , 3ml of phosphoric acid (1:1) and two drops of di phenyl amine were added, too. The contents of conical flask were titrated against sodium dichromate (sample) till end point and the results were recorded.

2.2.6 determination of chromium by back titration

15 ml of ferrous sulphate standardizated solution was added to conical flask, 5ml of sample (sodium dichromate) ,5ml of sulfuric acid (2M), 3ml of phosphoric acid (1:1) and two drops of di phenyl amine were added , too. The contents of conical flask were titrated against potassium dichromate standard solution till end point and results were recorded.

Chapter three

RESULTS AND DISCUSSION

Chapter Three

Results and Discussions

3.1 Results

3.1.1 standardization of ferrous sulphate

Initial volume /ml	End volume /ml	Used volume/ml
5.oo	9.80	4.80
9.80	14.60	4.80

3.1.2 Determination of chromium by direct titration

Initial volume (mL)	End volume (mL)	Used volume (mL)
0.00	17.oo	17.00
17.00	34.00	17.00

3.1.3 Determination of chromium by back titration

Initial volume (mL)	End volume (mL)	Used volume (mL)
0.00	12.00	12.00
12.00	24.00	12.00

3.2 Results of XRF

3.3 Calculations

Table() percentage of chromium and chromium oxide

Formation	**Direct titration**	**Back titration**
chromium	**19.552%**	**24.96%**
Chromium oxide	**28.576%**	**36.48%**

3.4 Discussion

The sample of chromite ore was obtained and quantitative analyzed and found the percentage of chromium by direct titration 19.552% and by back titration 24.96% as for the percentage of chromium oxide by direct titration 28.576% and by direct titration 36.48%.

These results were compared with the results of previous studies in Ingeassana hills in Blue Nile area by Japanese researchers and found these results were nearly to the obtained percentage but there are difference due to the difference in analysis methods . Japanese found that the percentage of chromium oxide in Gam mine area was 25% ,in Romeilik was 41% , in Gebanit mine area was (35 – 45%) , in Komrag area was (43 – 78%) and in The Eastern area was (43 – 59%).

Also the chromite ore was analyzed by XRF device to define the other components with chromite and they are magnesium oxide , aluminium oxide , silica and nickel oxide .

3.5 Conclusion

The chromite ore from Blue Nile area was analyzed and the results were compared with the previous studies which was done in the same area.

3.6 recommendation

- We recommend in future studies provide opportunities for students of chemistry field visits to fetch samples.
- We recommend using modern methods.
- Rehabilitation of laboratories in terms of equipment and materials that help student in their research and experience.
- There are studies that indicate that the chromium has an effect on the level of sugar in the blood of human being which is consider new field to chemistry students for modern scientific studies in this field.

3.7 References

- **Adli Abd Almaged – Sudan Industrial Minerals and Rocks.**
- **www.google.com**
- **www.wikipedia.com**