University of Kerbala
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Spectrophotometric Determination of Co(II) and Cr(III) ions Using a New Schiff Base Derivetives

A Thesis

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بِسْمِ اللَّهِ الرَّحْمَٰنِ الرَّحِيمِ

هُوَ الَّذِي جَعَلَ الشَّمْسَ ضِيَآءً وَالْقَمَرَ نُورًا
وَقَدَّرَهُ مَنَازِلَ لِتَعْلَمُواْ عَدَدَ السِّنِينَ
وَالْحِسَابَ مَا خَلَقَ اللَّهُ ذَلِكَ إِلاَّ بِالْحَقِّ
يُفَصِّلُ الآيَاتِ لِقَوْمٍ يَعْلَمُونَ

صدقَ اللهُ العليُّ العظيم

"سورة يونس، آية5"

DEDICATION

Praise be to God that His grace is righteous..

I dedicate the fruit of my humble effort, to the hand that removed the thorns from my path, to the one who drew the future with threads of hope and confidence, to the one whose name I carry with pride, and who is my support in life after God, my dear father, and to God's heaven on earth, to the source of love and tenderness, and my role model on my way to the one whose prayer was a secret. My success, my beloved mother, may God protect you. To the greatest supporter in everything, and from whom I learned perseverance and diligence, and in whose presence I gained strength, my soul mate, my dear husband, to the stars of my sky, my loved ones, my dear brothers and sisters who stood by me, and to my children, the apple of my eye. I thank everyone who helped me and gave me support.

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In the name of God,

the Most Gracious, the Most Merciful. Praise be to God, Lord of the worlds, and prayers and peace be upon the most honorable of messengers, our Master Muhammad, his family and companions, and those who follow them in goodness until the Day of Judgment. I extend my sincere thanks and gratitude to my almighty supervisor, Professor Dr. Alaa Farrak Hussein, for his constant encouragement and valuable advice to get the message out in the best way, and for his help throughout my research period. I wish God the best of success for him. I would like to thank the Dean of the College of Science, the Head of the Chemistry Department, the staff of the Chemistry Department, and a special thanks to all my colleagues.

Abstract

This study dealt with the preparation of a Schiff base reagent from condensation method from (8-aminoquinoline) with 4,4'-dimethoxybenzil to create reagent (BMPQYE1).The reagent was characterized by spectrophotometry techniques such as FT-IR, ultraviolet,^{1}HNMR and ^{13}CNMR, mass spectrometry.and conducting. A preliminary test was done for a number of 11 elements, and the Cobalt and Chromium ions were chosen to form complexes with the reagent. The maximum absorption of the reagent dissolved in ethanol was (λmax = 467 nm). Additionally, the best conditions for the reaction were studied, such as the pH function, the effect of time, temperature, volume of the reagent, and the effect of addition, and the calibration curves for both complexes were shown, which it was obyed the Beer's law in the range of the Cobalt(II) and Chromium(III) complexs with the concentration range (1-12)µg/5mL,(1 -8)µg/10mL with the linearity coefficient(R^2=0.9998), (R^2 = 0.9999) sequentially and the absorbance value.The molar absorptivity (ε)of cobalt was equal to($2.9x10^3$ L. $moL^{-1}.cm^{-1}$) and Sandal's sensitivity was equal to (0.102µg/cm^2),value of the Chromium molar absorptivity (ε) was equal ($1.1x10^4$ $L.moL^{-1}.cm^{-1}$) ,Sandal's sensitivity equal (0.25µg/cm^2) . Using the Job method and the mole ratio method for the Cobalt (II) and Chromium (III) complexes, the ion-to-reagent ratio (M:L) was determined. The stability constant (K_{sta}) for the cobalt (II) and chromium (III) complexes were ($K_{sta} = 3.749 \times 10^8$). and ($Ksta = 3.332 \times 10^{10}$), respectively. Also, the thermodynamic functions (ΔH, ΔG and ΔS) were calculated, as it was shown through the results that the reaction of the formation of the Cobalt (II) complex was endothermic and that the Chromium (III) complex was exothermic.The study demonstrated interference of the presence of a number of cations and anions as potential interactions when estimating the Cobalt(II) and Chromium(III) ions and the addition of appropriate masking agents was studied, as it was found that these ions interact to varying degrees depending on the nature and concentration of the interfering ion, and some of them did not show any effect.By measuring the conductivity, the charge of the two complexes was determined, where the Cobalt complex was uncharged and the Chromium complex was charged. The accuracy and precision of the analytical method were determined using five different concentrations. Relative Standard Deviation (RSD%) for the cobalt(II) ion ranged between (0.598%-1.567%) and the relative recovery ranged between (98.49% -101.19%), the values of the relative standard deviation (RSD%) for the Chromium(III) ion values ranged between (0.661% - 0.887%) and the relative recovery ranging between (99.98% - 101.16%), through which the detection limit was found for the Cobalt(II) and Chromium(III)ions(0.449µg/5mL),(0.769µg/10mL), respectively,which shows the sensitivity of the spectral technique using this ligand (BMPQYE1).Determining physical properties such as solubility ,melting point, and molar conductivity, the sedimentation of the reagent and the two complexes have also been investigated. The method was used to determine the amounts of Cobalt(II) and Chromium(III) ions in the pharmaceutical and hair sample. The method found a high in sensitivity and accuracy.

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List of abbreviations and Symbols.

Symbol	Meaning
A	Specific absorption coefficient
Abs	Absorption
As	Slow absorption
Am	Maximum absorption
BMPQYE1	1,2-bis(4-methoxyphenyl)-2-(quinolone-8-ylimino)ethan-1-one
C	Molar concentration
D	Degree of dissociation
DMF	N,N-Dimethyl-formamid
EDTA	Ethylene dianime tetra acetic acid
E%	Relative error
ε	molar absorptivity
FT-IR	Fourier Transform Infrared
GC-mass	Gas chromatography–mass spectrometry
^{1}HNMR	Proton nuclear magnetic resonance
K	Stability Constant
L.O.D	Detection Limit
L.O.Q	Limit of quantification
R^2	Linearity Coefficient
R	Correlation Coefficient
Rec%	Recovery%
RSD%	Relative standard deviation
S	Sandal sensitivity
S.D	Standard deviation
THF	Tetrahydrofuran
Uv-vis	Ultraviolet-visible spectroscopy
Vm	volume of the metal
VL	volume of the reagent
$\bar{x}$	Average
Xi	The value in the data absorption
x'	Mean
ΔH	Enthalpy
ΔG	Free energy
ΔS	Entopy
λ_{max}	Maximum wavelength

Chapter One

Introduction

Introduction

Schiff Base

Schiff bases, named after Hugo Schiff [1], are formed when any primary amine reacts with an aldehyde or a ketone under specific conditions. Structurally, a Schiff base (also known as imine or azomethine) (Fig. 1) is a nitrogen analogue of an aldehyde or ketone in which the carbonyl group (C,O) has been replaced by an imine or azomethine group. Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilisers [2]

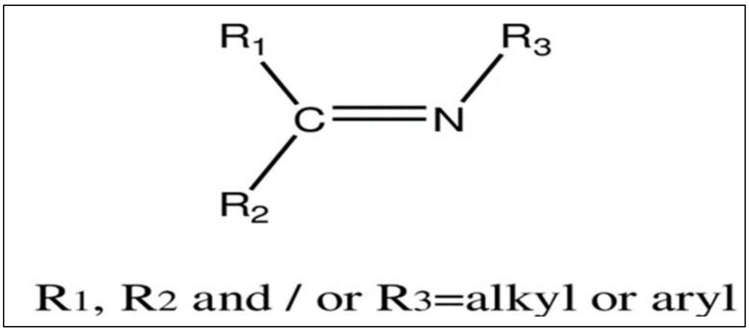
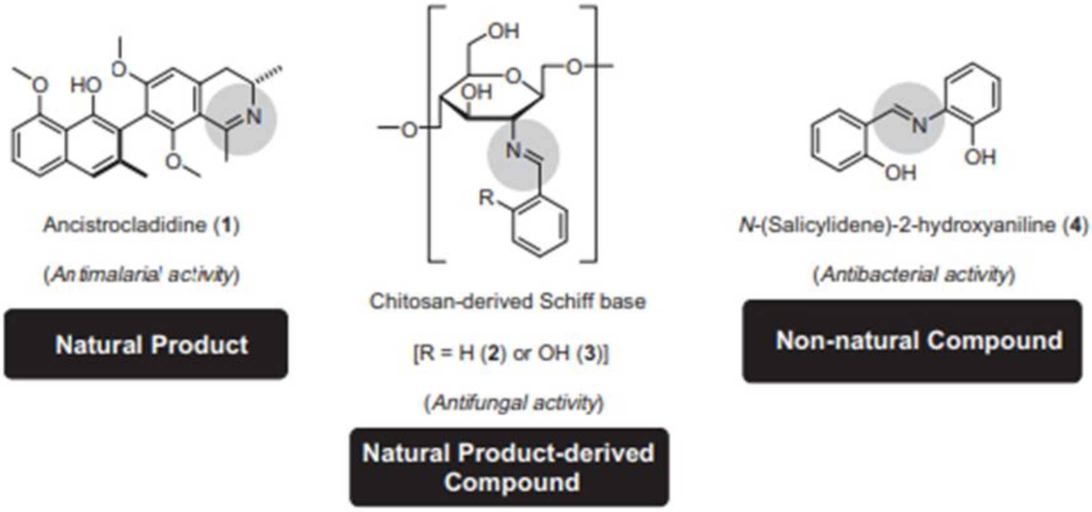


Figure (1-1):- Schematic representation of a Schiff base

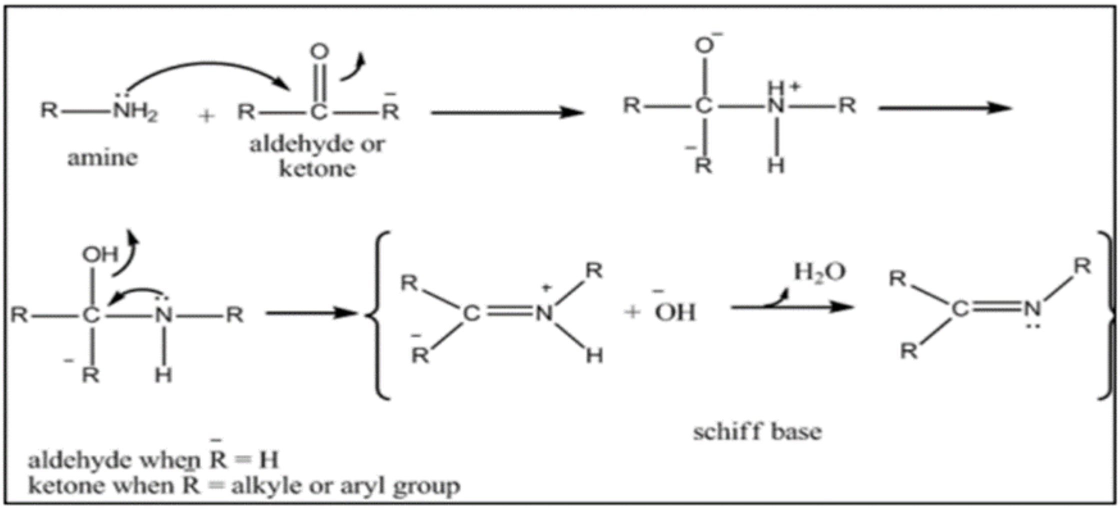
Antifungal,antibacterial, antimalarial, antiproliferative, antiinflammatory, antiviral, and antipyretic effects are only some of the many biological actions attributed to schiff bases. Figure 2 shows some examples of natural, synthetic, and synthetically produced chemicals that include amine or azomethine groups.It has been established that the imine group in such compounds is essential to their biological actions [3].



Figure(1-2): Biologically Active Schiff Bases.Each molecule is given a darkened appearance to denote the presence of an imine or azomethine group.

(1-2) Synthesis of Schiff bases

Schiff (1864) reported the first successful production of imines in the nineteenth century. Different strategies for the synthesis of imines have been developed since then [4]. Schiff published a conventional synthesis in which a carbonyl compound is condensed with an amine via azeotropic distillation [5]. The system newly generated water is then extracted using molecular sieves [6].Dehydrating solvents like tetramethyl orthosilicate and trimethyl orthoformate were developed in the 1990s for use in an in situ approach of water elimination [7,8]. In 2004, Chakraborti et al. showed that the employment of highly electrophilic carbonyl compounds and strongly nucleophilic amines is essential to the success of these techniques. They advocated using Bro nsted-Lowry or Lewis acids instead, which would activate the carbonyl group of aldehydes, catalyse the nucleophilic attack by amines, and dehydrate the system so that water is no longer needed in the final step(9,10).According to Scheme (1-1), Schiff bases are formed by nucleophilic addition to the (C=O) group that produces a hemiaminal, and then dehydration to construct imines[11].



Scheme (1-1) Mechanically Preparation a General Schiff Base

(1-3)Schiff base characterization

Ligands of Schiff base are important complexes for coordinating medicine and chemistry due to the existence of HC=N group as catalysis and has anti-oxidative activity, anti-bacterial activity, anti-fungal activity, magnetism, enzymatic reaction, and bio-inorganic. They have a wide range of uses in industry, including dyes, precursors, and synthesis chemicals for things like plastics and pigments [12]. Schiff bases have long been of interest because to their versatility in synthesis, sensitivity, and selectivity with respect to the central atom of metal [13]. Since Schiff base complexes are photochromic, they were used as a dye or photo stabiliser in the solar filters and assemblers. They also had a role in the development of optic sound recording technology [14].The stationary phase in gas chromatography, Schiff base [15], is a thermally stable molecule. These complexes were put to use as photonic components and electronic materials because to their non-linear optical properties[16].

Metal transition efficiently reduces the carbon nitrogen (C=N) double bond present in the Schiff base,when it comes to supramolecular [17].

(1-4)Schiff base compounds

Schiff base metal compounds are the best coordination organometallic complexes.Compounds with a Schiff base Because of their unique and useful properties, such as their capacity to bind heavy metal and poisonous atoms, to undergo tautomerism, to display catalytic reductions, and to be photochromic [18], Schiff bases and their metal derivatives have been synthesised. In addition to their biological and catalytic functions [19].The many reactivity modes displayed by Schiff base metal complex species makes their chemistry fascinating. To effectively synthesise hetero- and homo-metallic compounds with diverse stereo-chemistry [20].Schiff bases may accommodate a wide range of metal centres involving a wide range of ways of coordination. The crystallographic properties, steric effects, enzymatic reactions, mesogenic properties, structure redox relationships, magnetic characteristics, and catalytic activities of Schiff base compounds with different central metals such as copper, nickel, and cobalt that studied in great depth. In addition, they play a crucial part in deciphering the coordination chemistry of transition metal ions. For over twenty years, researchers have used Schiff base ligand transition metal complexes as models for biological systems due to their antibacterial, antifungal, anticancer, and anti-inflammatory properties[21].

(1-5) Heavy metal

Heavy metals are grouped within the category of environmental toxins; it is however a fact that many organisms need trace amounts of many metals to survive. They play significant roles in biological processes, in diseases, as well as in familial amyotrophic lateral sclerosis and others[22].Any metal with a high density and a high toxicity/poisonous potential even at low concentrations is considered a "heavy metal"[23].In common use, the phrase "heavy metal" refers to a class of metals and metalloids having an atomic density more than ($4g/cm^3$) [24]. Heavy metals density is not as crucial as its chemical characteristics. Lead, cadmium, zinc, mercury, arsenic, chromium, copper, iron, nickel, platinum, and rhodium are all examples of heavy metals.Heavy metals are non-biodegradable and hence long-lasting environmental pollutants found in the earth's crust. They enter the body in minute quantities through the air we breathe and the water we drink, and they build up over time. They are extracted as minerals from their ores in the rocks, where they exist in various chemical forms. Sulphides and oxides of metals including iron, arsenic, lead, lead-zinc, cobalt, gold, silver, and nickel are all examples of heavy metal ores. Iron, copper, and cobalt are only a few examples of the metals that contain both sulphide and oxide ores[25]. several heavy metals (including Fe, Zn, Ca, and Mg) are of bio-importance to human being, and their daily medicinal and dietary have been recommended. Consumption, even at extremely low quantities, of several others (such As, Cd, Pb, and Methylation forms of Hg) has been determined to have no known bio-importance in human biochemistry and physiology [26].

Table (1-1): Classifications of trace elements based on their usefulness [27].

Metals with no known beneficial Effects	Metals with possible beneficial Effects	Nutritionally essential metals
Aluminum Antimony Arsenic Barium Beryllium Cadmium Lead Mercury Silver Thallium	Boron Nickel Silicon Vanadium Iodine Strontium	Chromium III Copper Iron Manganese Cobalt Molybdenum Selenium Zinc

Table (1-2) :Concentrations of trace elements and the biological effects on humans health [28].

Elements	Deficiency	Toxicity
Chromium	Disturbance in the glucose metabolism	Kidney damage (Nephritis)
Copper	Anemia (Say back)	Chronic poisoning-Wilson, Bedlington-disease
Zinc	Dwarf growth, Retarded, Development of gonads Akrodermatitis entero-pathica	Metallic fever Diarrhoea
Cobalt	Anemia (White liver disease)	Heart failure, Polycythaemia
Iron	Anemia	Haemochromatosis
Selenium	Osteoarthritis effects on male infertility	Sele nosis Nervous system abnormalities
Arsenic	Unknown	Chronic poisoning, Skin Cancer
Aluminium	Unknown	Alzimer's disease

(1-6) Cobalt Element

(1-6-1) Chemistry of Cobalt

The above picture shows the form of cobalt in nature

In 1735,Georg Brandt made the initial discovery of cobalt. It is one of the essential trace elements in the human body. The eighth iron-group, which includes this element. Cobalt may be found in oxidation states ranging from one to five. The +2 and +3 variants are the most common, whereas the others are quite uncommon. Cobalt ions,like Co(III), react with acids to generate salts; the less reactive Co(II) form[29]..Cobalt's molecular weight is 58.933 g/mol, and it has a steel-gray, glossy, hard appearance[30]. Cobalt may be created as a byproduct of human activity and is otherwise abundant in the natural environment. It is found in trace amounts in compounds containing sulphur and arsenic. Welding, diamond tools, grinding, chemical catalysts,and nuclear power plants are just few of the many industrial uses for uranium[31].Corrosion-erosion of alloys containing cobalt and other metals can produce two radioactive forms of cobalt, ^{58}Co and ^{60}Co, which can be found in nuclear power plants. These two varieties generate gamma rays with large energies, making them useful for radiotherapy against cancers [32,33].Cobalt may be extracted from the minerals cobaltite, smaltite, and erythrite, and is frequently found in association with other metal ores such as nickel, silver, lead, copper, and iron. Meteorites are also a ready source. Zaire, Morocco, and Canada all have important mineral reserves. The United States Geological Survey has found evidence of cobalt rich deposits around the Hawaiian Islands and other U.S. Pacific territory at relatively shallow depths [34]. Cobalt significance extends beyond its application in industry; it's also a component of vitamin B_{12}, which is given to pregnant women to prevent anaemia [35].

(1-6-2) The Biological Role of Cobalt

This element is found in the human body in levels of 1-2 mg, mostly concentrated in the pancreas, brain, and serum but also in the heart, liver, kidneys, and spleen.An adult's recommended daily allowance of cobalt is 3 g, with a content of 0.012 g[36]. Cobalt is crucial to human health since it is a component of Vitamin B_{12}, which is required for folate and fatty acid metabolism and whose deficiency has been related to anaemia. Cobalt is a vital mineral for both animals and plants. Due to its toxicity, it is crucial to know how much cobalt is in the different environmental samples available [37].

(1-6-3)Cobalt deficiency

Anaemia, low thyroid function, and an increased risk of child mental retardation and failure have all been linked to cobalt deficiency, which is closely linked to problems in vitamin B_{12} production [38]. Overproduction of erythrocytes, pulmonary fibrosis, and asthma may result from this metal's potential to stimulate the thyroid and bone marrow. In addition to its traditional use in treating anaemia, Cobalt salts have recently gained popularity as an alternate doping method in athletics. The mechanism by which this metal improves aerobic performance by increasing production of erythropoietin is not well known. This metal causes an increase in erythrocyte number in the circulation. However, it is well-established that hypoxia reduces the oxygen level of circulating blood via the adrenal gland and hepatic bark, and so serves as a physiological impulse in erythropoietin production [39.40].

(1-6-4) Cobalt toxicity

Excessive exposure to some Cobalt compounds has been regarded as harmful for both the environment and the human body.Due to its abundance, people are regularly subjected to Co compounds in many forms. The most common ways that people are exposed to Co compounds are through their diet and the air they breathe. Because of its widespread industrial uses, cobalt exposure is very common in the workplace. Additionally, cobalt is utilised for a variety of medicinal applications, some of which were deprecated with time [41].

(1-6-5) Previous studies for the determination of cobalt ion

The literature evaluation of spectrophotometric methods for analysing cobalt is included in tables (1-3).

Table(1-3) The method used to determination cobalt ion

Method	Reagents	Results	Ref.
Spectrophoto-metric	Hatifloxacin, HGAT	Conc.(18.77–150.16)µg/ mL, R^2=0.9982 ,ε=(4.93 x10^3L.mol^{1-} .cm^{1-}) ,λ max= 500 nm , L.O.D=(2.05 µg/mL) , L.O.Q=(6.84 µg/mL)	[42]
spectra-continuous wavelet transformation	1-(2-pyridylazo)-2-naphthol (PAN)	Conc.(0.20-4.0 mg / L) , R^2= 0.9818, λ max= 583 nm , L.O.D=(0.014 mg/L) , L.O.Q=(0.046 mg/L)	[43]
Spectrophoto-metric	3,6-bis((2-aminoethylsalicylidenthio)pyridazine (PATS)	Conc(1–10.0 mg/ L) , R^2= 0.9997 , L.O.D=(0.098mg/L), L.O.Q=(0.320µg/mL)	[44]
Spectrophoto-metric	4-(2-pyridylazo) resorcinol (PAR)	Conc.(0.20–2.0µg/ mL), R^2= 0.9986, pH= 5.0, λ max= 558 nm, ε=(1.31 × 10^4) L.mol^{1-}.cm^{1-} RSD(%)=1.9, L.O.D=0.021 µg/ Ml	[45]
Spectrophoto-metric	2-pyridine carboxaldehyde isonicotinyl hydrazone(2-PYAINH)	Conc.(0.01-2.7mg/ L), ε=(7.1 x 10^4L.mol^{1-}.cm^{1-}) λ max= 346 nm, pH= 9.0, L.O.D= 0.003 mg/L	[46]
atomic absorption spectrometric	4-(2-pyridylazo)	Conc.(0.20–2.0) µg/ mL LOD= 0.021µg/ mL, R^2= 0.9986, pH=7-9 R.S.D%= 1.69, ε= 1.26 × 10^4 L.mol^{1-}.cm^{1-}	[47]
Spectrophoto-metric	2-carboxy-2 -hydroxy-5 -sulfoformazyl benzene (zincon)	Conc.(0–3.0 µg /L), λ max= 656 nm, R^2= 0.9700 pH=8, RSD%= 0.9952	[48]
Spectrophoto-metric	1-(2-thiazolylazo)-2-naphthol	Conc.(0.05–1.05 µg/ mL), pH=8, λ max = 618 R^2= 0.9980, L.O.D=(0.018µg.mL^{1-})	[49]
Spectrophoto-metric	(2Z,2'Z)-2,2'-((4S,5R)-4,5,6- trihydroxyhexane -1,2-diylidene)bis(N-phenylhydrazinecarbothioamide) (TBPH)	Conc.(0.20-3.83mg/L), λmax=589 nm, R^2=0.9981 L.O.D=3µg/L, L.O.Q =3. 163µg/mL, pH=3	[50]
Spectrophoto-metric	ammonium 2-amino-1-cyclohexan-1-dithiocarbamate	Conc.(0.10–3.00 µg /m), λmax= 445 nm, R^2= 0.9780 L.O.D=0.072µg/mL PH= 3.0–9.5	[51]
Spectrophoto-metric	1,2-bis (4-methoxyphenyl)-2-(quinolone-8-ylimino) ethan-1-one(BMPQYE1)	Conc.(1-12mg/L), λmax=589 nm, R^2=0.9981 L.O.D=0.449µg/mL, L.O.Q =1.481µg/mL, PH=10	In this study

(1-7)The Chromium Element

(1-7-1)The Chromium Chemical Element

In the crust, Chromium (Cr^{+3}) is the twenty-first most prevalent element, while in the core and mantle, it is the seventh most plentiful element. It may be found in group (VI)B [52]of the periodic chart and has atomic number 24. Commercial production of chromium is the fourteenth most important commodity in terms of tonnage, The ground state electronic configuration of chromium is $1s^2$ $1s^2$, $2s^2$, $2p^6$, $3s^2$, $3p^6$, $3d^5$, and $4s^1$ in its ground state. This distribution of the outermost electrons is favoured over the $3d^4/4S^2$ distribution because of the improved stability of the half-filled 3d shell with one electron in each orbital. If you fill the shell halfway, you'll end up in the "S" state (L = 0), which has a lot of exchange energy and is hence very stable[53]. The d orbitals extend close to the ion surfaces, exposing their electrons to the surrounding chemical environment. Chrome, a ubiquitous transition metal, occurs naturally in a wide range of iridescent and paramagnetic compounds. The oxidation numbers of chromium are as the following: The oxidation numbers range from -2 to +6, with +6 corresponding to the total number of 3d and 4s electrons. In compounds like carbonyls, nitrosyls, and organometallic complexes, the lowest oxidation states of chromium are -2, -1, 0, and +1. The most common and significant oxidation state of chromium is (+3), which is also the most stable[54].

Natural sources of chromium include igneous and metamorphic rocks, a variety of plants, soil, water, ash from volcanic eruptions, and gases. Chrome is a shiny metal that is also hard and brittle[55]. It is a greyish silver colour, and it polishes up well. When heated, it produces green chromic oxide that is stable in air and does not oxidise. A thin oxide layer is formed when Chromium interacts with oxygen, making the underlying metal impervious to oxygen and protecting it from corrosion. Most chromium is put to use in metal ceramics, chrome plating, and stainless steel[56]. In the past, a silvery mirror finish was commonly achieved by plating steel with chromium. Chromium is utilised in metalworking for its corrosion-resistant and glossy properties, as well as in the production of dyes and paints. It is employed as a catalyst in leather dyeing and tanning, as a colourant in synthetic rubies, and as a mould material for brick firing[57,58].

(1-7-2)The Biological Role of Chromium (III)

Chromium is a critical trace element for healthy living in humans and other animals. When it comes to breaking down lipids and carbs, Chromium is a key player[59]. It encourages the body to make more cholesterol and fatty acids. They play an important role in mental and physical functions alike. Chromium aids in insulin's performance and the breakdown of glucose. Chromium is a necessary mineral that humans can't synthesise on their own[60]. can only get it from food. It is absorbed by the digestive system and then distributed to the rest of the body, where it accumulates in the tissues.It is possible that chromodulin, an oligopeptide that binds chromium, has a special function in the autoamplification of insulin signalling. The authors discuss research and development of dietary supplements and medicines using chromium[61]. A lack of chromium (III) disrupts metabolic pathways. Decreased glucose tolerance, caused by alterations in insulin affinity for its receptors on cells, is the major response of organisms to chromium (III) deficiency[62]. Chromium (III) is present in nucleic acids in high concentrations. It affects metabolic rate, replication efficiency, and transcription in these organisms. The ion decreases plasma corticosteroid levels while simultaneously boosting immunological function[63].

(1-7-3) Chromium Deficiencies.

Deficiencies in chromium are rather prevalent. Chromium deficiency can affect insulin action, impede protein synthesis and energy generation, and eventually lead to type 2 diabetes and heart disease[64]. This is especially true for athletes, diabetics, pregnant women, and the elderly. High insulin and cholesterol levels in the blood have been linked to chromium deficiency, which has been the subject of several research. Lack of chromium has been linked to increased blood cholesterol and the development of aortic plaques in rats[65].

(1-7-4)Chromium toxicity.

Toxic effects on organisms or cells from exposure to chromium, especially hexavalent chromium, are known as chromium toxicity[66]. Hexavalent chromium and its derivatives are hazardous when breathed or swallowed. The human diet requires a trace mineral called trivalent chromium[67]. Humans are only at danger of genotoxicity if substantial levels of trivalent chromium penetrate live cells.However, this is highly unlikely due to normal metabolism and cell function[68]. Exposure to Cr(VI) has been associated with a range of adverse health effects, including occupational asthma, eye irritation and damage, perforated eardrums, respiratory irritationand many other health effects [69].

(1-7-5)Previous studies for the determination chromium ion

The literature overview of spectrophotometric methods for analysing chromium is included in tables (1-4).

Table(1-4) The method used to determination chromium ion

Method	Reagents	Results	Ref.
cloud-point extraction (CPE)	2-[benzenethiazolylazo]-4-benzenenaphthol (BTABN)	Conc.(0.1-2.0 μg/mL) L.O.D=(0.017 μg/mL) L.O.Q=(0.0568 μg/mL) , $\varepsilon=(4.47\times10^{4}L.mol^{-1}.cm^{-1})$ λ_{max}=475 nm R^2=(0.9998)	[70]
Spectrophoto-metric	chromotropic acid (CA)	Conc.(30-1200 μg/mL) λ max=370 nm RSD%=(0.04)	[71]
Spectrophoto-metric	bis(salicylaldehyde)orthophenylenediam-ine(BSOPD)	Conc.(0.02 – 4.0 $mg.L^{-1}$) λ max=478 nm , $\varepsilon=(1.67\times10^{4}L.mol^{-1}.cm^{-1})$	[72]
Spectrophoto-metric	Bis(salicylaldehyde)orthophenylenediamine (BSOPD)	Conc.(0.01-12.0 $mg.L^{-1}$), λ_{max}=482 nm , $\varepsilon=(3.5\times10^{5} L.mol^{-1}.cm^{-1})$ R^2=(0.9974)	[73]
Spectrophoto-metric	Trifluoperazine hydrochloride (TFPH)	Conc.(2-18) μg, λ max= 505 nm $\varepsilon=(2.08 \times 10^{4}L.mol^{-1}.cm^{-1})$ L.O.D=(0.0033μg/mL) R^2 =0.0033	[74]
Spectrophoto-metric	diphenylthiocarbazone (dithizone)	Conc.(0.05-10 mg/L) λ_{max}=520 nm , $\varepsilon=(1.2\times 10^{5} L.mol^{-1}.cm^{-1})$	[75]
Spectrophoto-metric	Leuco Xylene cynaol FF	Conc.(0.05–0.45 μg mL^{-1}), λ_{max}=615 nm , $\varepsilon=(8.23\times10^{4} L.mol^{-1}.cm^{-1})$	[76]
Spectrophoto-metric	Rhodamine 6G hydrochloride dye (RG+)	Conc.(0.02451.5 μg /mL) ,L.O.D=(0.0073 μg /mL) L.O.Q=(0.025 μg/ mL) λ_{max}=534	[77]
Spectrophoto-metric	Tetraphenyl-phosphonium bromide	Conc.(1–114μg/L) L.O.D=0.43μg/L L.O.Q =1.42μg/L.	[78]
Spectrofluori-metric	[2-(diethylamino)ethyl 4 aminobenzoate] hydrochloride	Conc.(25–250 μg/L) λmax=270 nm, L.O.D=7.51μg/L L.O.Q =24.75 μg/L.	[79]
Spectrophoto-metric	1,2-bis(4-methoxyphenyl(-2-(quinolone-8-ylimino)ethan-1-one(BMPQYE1)	Conc.(5-80 μg/mL), λ_{max}=560 nm, L.O.D=0.769μg/mL, L.O.Q =2.537μg/mL. R^2 =0.9996	In this study

(1-8) The aims of the study

This study includes :

Main aim :

1. Preparation of one a derivative of new Schiff Bases compounds then characterization by FT-IR ,UVvis., GC-Mass,^{1}H-NMR and ^{13}C-NMR methods.

Reserch objective :

1. Studying the best conditions for the determination of Cobalt(II) and Chromium(III) complexes such as the size of the reagent, pH, the effect of time, the succession of addition, temperature and the determination of the metal—Reagent(M – R) ratio……etc

2. Studying the interferences and possible ways to minimize them.

3. Studying some of the physical and chemical properties of the reagent and its complexes, determining the charge of the complexes, preparing its solid complexes and proposing the structural formula of the complexes.

4. Studying of thermodynamic functions (ΔH, ΔG and ΔS).

5. Analytical applications of the developed determination method are also carried out on, environmental and pharmaceutical sample.

Chapter Two

materials & methods

(2-1) Apparatus

Taple(2-1) The apparatues used in this study

No.	Instrument name	Type
1	Spectrophotometer	[Double Beam UV-visible Spectrophotometer – 1800 , Shimadzu, (Japan)]. At the University of Kerbala.
2	pH-meter	pH-meter-HI1271,HANNA (ROMANIA).
3	GC-Mass	Manufacturer Company: Agilent Technology (HP), MS Model: 5973 Network Mass Selective Detector In Basars
4	^{1}H-NMR and ^{13}C-NMR spectroscopy	Record 1HNMR , 13CNMR spectrums. (VARIAN) , 500 MHZ. In Iran
5	FTIR Spectrophotometer	FTIR – IRAffinity-1S Shimadzu (Japan). At the University of Kerbala.
6	Melting point measuring Instrument	Melting point Apparatus –SMP30,Stuart (England). At the University of Kerbala.
7	Water bath	BS-11 JEIO TECH (Korea). At the University of Kerbala
8	Conductivity meter	Digital conductivity Meter- WTW -720 –inoLab (Germany) At the University of Kerbala.
9	Sensitive balance	Electronic balance ,ABS 220-4,KERN (Germany). At the University of Kerbala.
10	Electric heater	Hotplate Stirrer ,LMS-1003,tabTEch(Korea). At the University of Kerbala.

(2-2) Chemical Materials

Table (2-2) chemical materials and Reagent used

No	Name	Chemical formula	Company Name	Purity	Molecular weight(g. mol^{-1})
1	8-aminoquinoline	$C_9H_8N_2$	(B.D.H)	99.0%	144.177
2	4,4'-dimethoxybenil	$C_{16}H_{14}O_4$	(B.D.H)	99.8%	270.28
3	Glacial acetic acid	CH_3COOH	GCC	99.6%	60.05
4	Sodium hydroxide	NaOH	(B.D.H)	99.0%	39.997
5	Nitric acid	HNO_3	E.Merck, Darmstadt	98.0%	63.01
6	Sodium nitrate	$NaNO_3$	Fluka	98.0%	84.9947
7	Ethanol	C_2H_5OH	Fluka	99.0%	46.08
8	Cadmium(II)nitrate-4-hydrate	$Cd(NO_3)_2.4H_2O$	Fluka	99.0%	308.4819
9	Nickel(II)nitrate -6-hydrate	$Ni(NO_3)_2.6H_2O$	(B.D.H)	98.0%	290.7949
10	Cobalt(II)nitrate -6-hydrate	$Co(NO_3)_2.6H_2O$	(B.D.H)	99.0%	291.0347
11	Iron(III)nitrate -9-hydrate	$Fe(NO_3)_3.9H_2O$	(B.D.H)	99.0%	403.9972
12	Mercury(II)nitrate-1-hydrate	$Hg(NO_3)_2.H_2O$	MERCK	98.5%	342.6151
13	Lead (II)nitrate	$Pb(NO_3)_2$	(B.D.H)	99.0%	331.2098
14	Magnesium(II)nitrate -6-hydrate	$Mg(N0_3)_2.6H_2O$	(B.D.H)	99.5%	256.4065
15	Barium nitrate	$Ba(NO_3)_2$	(B.D.H)	99.0%	261.3368
16	Copper (II)nitrate -3-hydrate	$Cu(NO_3)_2.3H_2O$	(B.D.H)	99.0%	241.6016
17	Potassium Sulfate	K_2SO_4	MERCK	99.0%	174.2592
18	Potassium bromide	KBr	NANIWA	99.0%	119.0023
19	Potassium thiocyanate	KSCN	(B.D.H)	99.0%	97.1807
20	Potassium Iodate	KIO_3	GCC	99.0%	214.001
21	Potassium dichromate	$K_2Cr_2O_7$	Riedel	99.0%	294.1846
22	Potassium carbonate	K_2CO_3	(B.D.H)	99.0%	138.2055
23	Potassium cyanate	KCN	(B.D.H)	99.0%	65.1157
24	Thiourea	CH_4N_2S	AAG	99.0%	76.1209
25	Ascorbic acid	$C_6H_8O_6$	(B.D.H)	99.0%	176.1241
26	Ethylene diamine tetra acetic acid disodium salt	Na_2EDTA	Fluka	98.0%	372.2368

27	Citric Acid	$C_6H_8O_7$	Scharlou	99.0%	192.1235
28	Potassium chloride	KCl	GCC	98.0%	74.5513
30	Dimethyl sulfoxide	DMSO	Fluka	98.0%	78.1334
31	Zinc(II)nitrate .6-hydrate	$Zn(NO_3)_2.6H_2O$	HIAMED IA	99.0%	297.4815
32	Chromium(III)nitrate .9-hydrate	$Cr(NO_3)_3.9H_2O$	(B.D.H)	99.0%	400.1483
33	Dimethyl ether	$C_4H_{10}O$	Scharlou	99.5%	74.1216
34	N,N-Dimethyl-form amid(DMF)	C_3H_7NO	CHEMLI MIED	99.5%	73.0938
35	ammonium Hydroxid	NH_4OH	Fluka	99.0%	35.0458
36	Magnesium chloride	$MgCl_2$	Fluka	99.0%	95.211
37	Ammonium chloride	NH_4CL	Fluka	99.0%	53.4915
38	Chloroform	$CHCl_3$	HIAMED IA	99.4%	119.3776

(2-3) Preparation of reagent (BMPQYE1)

The present study focuses on the production of the reagent 1,2-bis(4-methoxyphenyl)-2-(quinolone-8- ylimino)ethan-1-one(BMPQYE1). In a round-bottomed flask, a solution having 8-aminoquinoline (0.1441g, 1mmol) in 20mL of ethyl alcohol has been combined with a solution of 4,4-dimethoxybenzil (0.2703g, 1mmol) and three to four drops of glacial acetic acid. The solution has been subsequently subjected to reflux for a duration of 4 hours at a temperature of 80 degrees Celsius, while being agitated. The product had been obtained through the process of filtration, followed by washing with ethyl alcohol. Subsequently, it has been subjected to drying, recrystallization, and further drying at ambient temp. The resulting reagent percentage was estimated at (78%).

(2-4) Preparation of standard solutions

(2-4-1) Cobalt (II) ion standard solution preparation (1mg/100mL).

To generate a Cobalt(II) ion solution with an amount of (3.432×10^{-3} M), 0.4933 g of $[Co(NO_3)_2].6H_2O$ has been dissolved in 100 ml of water that had been deionized. The initial step in generating additional necessary standard solutions involved diluting a standard solution with water that had been deionized.

(2-4-2)Chromium(III)ion standard solution preparation(1mg/100mL)

The preparing of the standard chromium (III) solution involved the dissolution of 0.7697 g of $[Cr(NO_3)_3].9H_2O$ in 100 ml of water that had been deionized, resulting in an amount of (2.498×10^{-3} M). The produced standard solution has been utilized to generate additional standard solutions through the process of dilution utilizing water that had been deionized.

(2-4-3)Preparation of the reagent (BMPQYE1) (1mg/100mL)

The reagent has been dissolved in absolute ethanol by adding 0.1000g of the reagent to the ethanol. The solution has been then made up to a final amount of 100 mL in a amounttric flask utilizing ethanol.

(2-4-4) Preparation of sodium hydroxide solution (0.1M)

Prepare sodium-hydroxides with a amount of 0.1M by diluting 0.4000g in 100 mL of water that had been deionized.

(2-4-5) Preparation a hydrochloric-acids solution (0.1M)

The experiment involved the dilution of concentrated hydrochloric acid (37percent, 1.18 g/cm^3) by adding 0.818 mL of the acid to a 100 mL amounttric flask, followed by the addition of deionized water. Dilution of hydrochloric acid (HCl) solutions has been achieved by diluting a standard solution with an appropriate amount of water.

(2-4-6) Interferences

(2-4-6-1) preparation of Cationic ions solutions

The solutions have been generated by dissolving cation nitrate salts in water that has been deionized at an amount of (1000µg/1mL). The salts have been dissolved based on the molecular weight of every respective salt for every component in 25mL of water that had been deionized.

Table (2-3): The weights which used prepare the cation solutions.

Seq.	Cations formula structure	Weight(g)
1	$Cd(NO_3)_2.4H_2O$	0.2744
2	$Ni(NO_3)_2.6H_2O$	0.4954
3	$Co(NO_3)_2.6H_2O$	0.4933
4	$Fe(NO_3)_3.9H_2O$	0.7234
5	$Hg(NO_3)_2.H_2O$	0.1708
6	$Pb(NO_3)_2$	0.1598
7	$Mg(N0_3)_2.6H_2O$	1.0549
8	$AgNO_3$	0.1574
9	$Cu(NO_3)_2.3H_2O$	0.3795

(2-4-6-2) preparation of Anionic ions solutions

Solutions of anions at an amount of (1000µg \1mL) of essential salts of potassium were generated by dissolving the needed weight and depending on the molecular weights in 25 mL of water that had been deionized.

Table (2-4): The weights used to prepare the anions solutions

Seq.	**Substance**	**Weight(g)**
1	K_2SO_4	0.0453
2	KBr	0.0403
3	KSCN	0.0418
4	KIO_3	0.0301
5	$K_2Cr_2O_7$	0.0340
6	K_2CO_3	0.0575
7	KCN	0.0625

(2-4-7) Preparation various amounts standard solutions to Perceive the ionic strength impact

A- Preparation a solution contains (0.5) M sodium sulfate (Na_2SO_4).

Sodium sulfate has been produced at an amount of (0.5)M by dissolving (1.7800)g in 25 mL of water that had been deionized. The prepared solution has been used to prepare the following amounts (0.0005, 0.005, and 0.05 M).

B- Preparation a solution contains (0.5) M sodium nitrate ($NaNO_3$) .

A (0.5) M sodium nitrate solution has been generated by dissolving 1.0630g of sodium nitrate (NaNO3) in 25 mL of water that had been deionized. The amounts (0.0005, 0.005, and 0.05 M) have been generated by performing multiple dilutions of the solution.

(2-4-8) Preparation of solutions utilizing of masking agent

The process of creating agent of masking agent with a amount of 25 mL involved several steps. Firstly, a predetermined amount of every component has been dissolved in 5 mL of water that had been deionized. Next, the resultant solution has been transferred to a amounttric container with a capacity of 25 mL. Eventually, the flask had been filled to the mark with water that had been deionized.

Table (2–5): The masking parameters utilized in this investigation

Seq.	Masking agent (0.1M)	Weight(g)
1	Thiourea	0.1900
2	Ascorbic acid	0.4400
3	Na_2EDTA	0.9300
4	Citric Acid	0.0200
5	KCl	0.1800
6	$Na_2HPO_4.12H_2O$	0.8900
7	Formaldehyde	0.0750

(2-4-9) Preparation of buffer solutions [80-82]

1. The solution provided is a buffer solution having ammonium chloride with a pH magnitude of 8.

   An experiment has been conducted in which 8.750 g of ammonium chloride has been dissolved in 25 mL of distilled water. Additionally, 41.250 mL of concentrated ammonia has been inserted to the solution. The resulting mixture has been then diluted with distilled water to revery a final amount of 125.0 mL.

2. A solution of borate buffer with a pH of 8.

   To prepare a solution, 2.737 grams of boric acid were dissolved in 100 milliliters of distilled water. The pH can be modified by employing a solution of sodium hydroxide with an amount of 44.444 g/L. The solution should be diluted with distilled water to a final amount of 100 mL.

3. The solution being referred to is a buffer solution having ammonium chloride with a pH of 10.

   Following the dissolution of 5.4 grams of ammonium chloride in 20 milliliters of distilled water, proceed by adding 35.0 milliliters of ammonia to the solution, thereby increasing the overall amount of the solution to 100 milliliters.

4. A buffer solution having diethanolamine with a pH of 10.

   A quantity of 19.280 g of diethanolamine dissolves in water that has been distilled, and the resultant solution is subsequently diluted to a final amount of 80 mL utilizing similar solvent. Subsequently, introduce a amount of 0.1 mL of a solution having magnesium chloride at an amount of 37,200 g/L. Following this, adjust the pH by incorporating hydrochloric acid in a ratio of one million parts. The solution should be diluted by adding distilled water until the final amount reveryes 100 mL.

(2-5) Procedure

(2-5-1) Preliminary tests of the substance's reactivity with a number of metallic ions

Upon the addition of the reagent a distinct change in color occurs. The determination has been conducted by agitating the test tubes through the addition of the solution of reagent to 2 mL of solutions with approximately (11) distinct metal ions, every at an amount of 100 µg/mL, inserted to individual test tubes. After this phase, the solution has been divided into two segments. One segment received additions of drops from a (0.1) M sodium hydroxide solution, while the other segment received additions of drops from a 0.1 M hydrochloric acid solution. This has been done to assess the influence of acidic properties.The optimum response of the reagent has been observed when it interacted with ions of Chromium (III) and cobalt(II).

(2-6) Investigating the reagent's (BMPQYE1) absorption spectra, and the complexes produced with ions of Chromium (III) and Cobalt (II)

(2-6-1) Investigating the reagent's (BMPQYE1) absorption spectra in the ultraviolet-visible range

The reagent BMPQYE1 had been subjected to spectral scan within the restricted wavelength range of 200-1100 nm. The amount of the reagent used had been 2.523×10^{-3}M. Ethanol had been used as a comparable solution (Blank) for the experiment. A double-beam ultraviolet (UV)-visible spectrophotometer has been utilized, along with quartz cells having a path for light with a thickness of 1 cm. The wavelength at which the reagent exhibited the highest absorbance has been identified to be (λ max = 467 nm).

(2-6-2) Ultraviolet–visible absorption spectra complexes of Chromium (III) and Cobalt (II)

Upon acquiring knowledge of the ions that engage in interaction with the reagent as described in paragraph (2-5-1).Two volumetric flasks, each one with a capacity of 10 mL, were utilized. One of the containers has been filled with a solution consisting of 5 mL ions of Chromium (III) at an amount of 2.498x10^{-3}M. The second amounttric flask had been filled with 5mL ion of Cobalt(II)at an amount of 3.432x 10^{-3}M. Subsequently,1.0 mL of the solution of reagent, which had an amount of 2.523x10^{-3}M,has been inserted to every amounttric container. Subsequently, water that had been deionized has been introduced into every amounttric container until the desired level has been reveryed. The suitable ions amount of Cobalt(II) and Chromium (III) has been marked on every amounttric container. In addition to the inclusion of ions Cobalt (II) and Chromium (III), the solution known as Blank has been prepared in a similar manner.

The complex solutions obtained in the preceding paragraph were subjected to analysis utilizing a spectrophotometric device, covering a wavelength range of (200-1100) nm. The highest absorbance magnitudes were then identified. In this particular region, the complexes exhibit the highest level of absorption once compared to the solution of reagent, which serves as an analogous solution (Blank).

(2-7) Determination the optimum conditions for the preparation Complexes of chromium (III) and Cobalt (II) utilizing the (BMPQYE1) reagent

After establishing the maximum absorption (λmax) of every of the complexes in paragraph (2-6-2), which was found observed that the complex of cobalt(II) exhibited a λmax of 589 nm, while the complex of Chromium (III) had a λmax of 560 nm.Subsequently,aseries of tests had been carried out to identify the the ideal situation for generating the two combinations utilizing the spectrophotometric technique. The objective of this study has been to attain a high level of sensitivity and selectivity in the estimation of ions.

(2-7-1) limitation of optimal value of Reagent

1- A set of volumetric flasks 5 mL every has been utilized for the experiment. Subsequently, solution of Cobalt(II) were added introduced into every flask.thatit were at aconcentration of $3.432x10^{-3}$ M,which is roughly equal to (50µg/5mL),and subsequently introduced in a sequential manner. Various amounts of the solution of reagent were utilized, every one having at concentration of $2.523x10^{-3}$ M.where it ranged amount 0.4 mL and 2mL.Subsequently, the acidity of the solutions had to be regulated to a pH of 10, accompanied by volume completing to the mark with deionized water and the absorption measurement at the maximum wavelength of the cobalt complex, which has been determined to be 589 nm (λ max). This evaluation has been performed for every solution, with the exception of the contrasting solution, which did not contain the inserted ion present in the tested solutions.

2- A set of volumetric flasks 10 mL has been utilized, and a solution having Chromium (III) ions has been added to every flask at concentration of $2.498x10^{-3}$M, which is roughly equal to 50µg/10 mL. The addition of the solution has been carried out in a sequential manner. Different amounts of the solution of reagent were prepared at concentration of $2.523x10^{-3}$ M. The amount range used has been between 0.4 mL and 2 mL. The acid level of every solution has been set to a pH of 8. The water that has been ionized has been inserted to every solution to complete the amount up to the mark. The absorption evaluation has been then taken at the max wavelength of the Chromium complex, which is 560 nm. The absorption measurements were compared to the solution of reagent, with the exception of adding the ion to all of the determined solutions.

(2-7-2) Effect of pH value.

1-Investigating the acidic function impact on complex's of Cobalt(II).

A ion solutions set of Cobalt (II) has been generated with an amount of 3.432 $x10^{-3}$M, roughly equal to (50 µg /5 mL), by combining cobalt ion (II) with 1 mL of the reagent. The solution has been prepared at concentration of $2.523x10^{-3}$ M and has been used to modify the acidic function. with a variety of magnitudes within the given range. A range of pH magnitudes, ranging from 2.0 to 12.0, has been investigated utilizing hydrochloric acid (HCl) with an amount of 0.1M and sodium hydroxide (NaOH) with an amount of 0.1M. The pH of every solution had been detected utilizing a pH-meter, and the solutions were subsequently diluted with water that had been deionized to revery the desired amount.

The measurement of absorbance has been conducted at the wavelength corresponding to the maximum absorption of the cobalt compound, which is (λ max =589 nm).

2- Investigating the acidic function impact on complex's of Chromium (III)

A series of solutions having Chromium (III) ions have been created with concentration of $2.498x10^{-3}$ M, which is roughly equal to (50µg/10 mL). This has been achieved by combiningChromium (III) with 1 mL of a solution of reagent with concentration of $2.523x10^{-3}$M, and adjusting the acidity within the range of 2.0-12.0 utilizing a mixture of NaOH (0.1M) and HCl (0.1M). The pH of every solution has been determined utilizing a pH-meter, and the solutions have been diluted to the desired amount with water that had been deionized. The absorbance has been determined by measuring the intensity of light at the wavelength corresponding to the cobalt compound's max absorption (λ max = 560 nm).

(2-7-3)The duration impact of time on the stability of a complex

The graphs illustrate the impact of the time variable on the chromium (III) and cobalt (II) complexes stability. The absorption evaluation for the two complexes were carried out at various time intervals, specifically at the designated wavelength (λmax) for every complex.

1- Investigating the duration impact on the complexes of Cobalt (II) absorption.

The preparing of the complex of cobalt (II) involved the combination of an ion solution of cobalt (II) with an amount of $3.432x10^{-3}M$ (roughly equal to(50µg/5mL) and 1mL of a solution of reagent with concentration of 2.523 $x10^{-3}M$. The reaction has been carried out under optimum acidic situations with a pH level of 10.

Subsequently, this amount is augmented to 5 mL by the addition of water that has been deionized.The measurement of absorbance for the generated complex has been conducted at a wavelength of 589 nm, which is the maximum absorption wavelength (λmax). This measurement has been taken when comparing the comparator solution, also known as the blank. The absorbance readings were recorded at various time intervals, starting from the initiation of the reaction until the completion of the 24-hour experiment.

2- Investigating the duration impact on the complexes chromium (III) absorption.

The preparing of the complex of chromium (III) involved the combination of an ion solution of chromium (III) with concentration of 2.498 $x10^{-3}M$ (roughly equal to 50 µg/10mL) and 1mL of a solution of reagent with concentration of $2.523x10^{-3}M$. The reaction has been carried out under optimum acidic situations with a pH level of 8.

Subsequently, this amount is augmented to 10 mL by the addition of water that has been deionized.The measurement of absorbance for the generated complex has been conducted at a wavelength of 560 nm, which is the maximum absorption wavelength (λmax). This measurement has been taken when comparing the comparator solution, also known as the blank. The absorbance readings were recorded at various time intervals, starting from the initiation of the reaction until the completion of the 24hour experiment.

(2-7-4) The impact of sequential addition

The investigation involves manipulating the sequence of additions and operations related to the addition of ions, reagents, and pH measurement.

(2-7-4-1) Complex ion of Cobalt(II)

1.The experimental procedure involves sequentially adding a solution having ions of cobalt(II) at an amount of $3.432x10^{-3}$M (which equates to 50µg/5mL) to 1mL of a solution of reagent with concentration of $2.523x10^{-3}$M. The pH level of the solution is regulated to the optimum acidic situation at a magnitude of pH = 10. Subsequently, the amount is supplemented with water that has been deionized to revery a final amount of 5 mL.Regarding the comparative solution, it has been formulated by combining solutions utilizing identical preceding procedures, where the dimensions of the ion of cobalt(II) were substituted with water that had been deionized. Subsequently, the measurement of the absorption of the generated complex has been conducted at a wavelength of maximum absorption (λmax=589 nm).

2.The experimental procedure involves sequentially adding 1 mL of a solution of reagent with concentration of $2.523x10^{-3}$M, followed by the addition of an ion solution of cobalt(II) with an amount of $2.498x10^{-3}$ M. The ion solution of cobalt(II) is inserted in an amount that equals to (50 µg/5 mL). The pH level of the solution is regulated to an optimum acidic magnitude of 10.Subsequently, the amount is augmented to 5 mL by the addition of water that had been deionized. Regarding the comparative solution, it has been formulated by combining the solutions utilizing similar preceding procedures, where the ion of cobalt(II) has been substituted with water that had been deionized. Subsequently, the measurement of the absorption of the generated complex has been conducted at a wavelength of maximum absorption (λmax=589 nm).

3.The experimental procedure involved the sequential addition of a ion solution of cobalt(II),with concentration of $3.432x10^{-3}$M (which is equal to 50 µg / 5mL), to the modified acidic solution with a pH of 10. Following this, 1mL of the solution of reagent, with concentration of $2.523x10^{-3}$M,had been inserted.Subsequently, the amount present is augmented to 5 mL by the addition of water that had been deionized. Regarding the comparative solution, it has been formulated by combining the solutions utilizing similar aforementioned procedures, with the substitution of the ion of cobalt(II) with water that had been deionized. Subsequently, the measurement of the generated complex absorption has been conducted at wave length of maximum absorption(λ max = 589 nm).

4.The experimental procedure involves sequentially adding 1 mL of a solution of reagent with concentration of $2.523 x10^{-3}$ M, after modifying the acidity function to a the pH level of 10. This solution of reagent is then inserted into a solution having ions of cobalt(II) at concentration of $3432 x10^{-3}$ M, which is roughly equal to (50µg/5mL).Subsequently, the amount is supplemented with water that has been deionized to revery a final amount of 5 mL. Regarding the comparative solution, it has been formulated by combining the solutions utilizing similar aforementioned procedures, but substituting the ion of cobalt(II) with water that had been deionized. Subsequently, the measurement of the generated complex absorption has been conducted at a wavelength of maximum absorption (λ max = 589 nm).

(2-7-4-2) Complex ion of Chromium (III).

1.The experimental procedure involves sequentially adding a solution having ions of Chromium (III) at an amount of 2.498 $x10^{-3}$M (which equates to 50µg/10mL) to 1mL of a solution of reagent with concentration of 2.523$x10^{-3}$ M.The pH level of the solution is regulated to the optimum acidic situation at a magnitude of pH = 8. Subsequently, the amount is supplemented with water that has been deionized to revery a final amount of 10 mL.Regarding the comparative solution, it has been formulated by combining solutions utilizing identical preceding procedures, whereasin the dimensions of the ion of chromium (III) were substituted with water that had been deionized. Subsequently, the measurement of the absorption of the generated complex has been conducted at a wavelength of maximum absorption (λmax=560 nm).

2.The experimental procedure involves sequentially adding 1 mL of a solution of reagent with an amount of 2.523 $x10^{-3}$ M, followed by the addition of an ion solution of chromium (III) with concentration of 2.498 $x10^{-3}$ M. The ion solution of chromium (III) is inserted in an amount that equals to (50µg/10 mL).The pH level of the solution is regulated to an optimum acidic magnitude of 8.Subsequently, the amount is augmented to 10 mL by the addition of water that had been deionized. regarding the comparative solution, it has been formulated by combining the solutions utilizing similar preceding procedures, whereasin the ion of chromium (III) has been substituted with water that had been deionized.

Subsequently, the measurement of the absorption of the generated complex has been conducted at a wavelength of maximum absorption (λ max=560 nm).

3. The experimental procedure involved the sequential addition of a ion solution of Chromium (III), with concentration of $2.498x10^{-3}$M (which is equal to 50µg/10mL), to the modified acidic solution with a pH of 8. Following this, 1mL of the solution of reagent, with an amount of 2.523 $x10^{-3}$ M, had been inserted.Subsequently, the amount present is augmented to 10 mL by the addition of water that had been deionized. Regarding the comparative solution, it has been formulated by combining the solutions utilizing similar aforementioned procedures, with the substitution of the ion of chromium (III) with water that had been deionized. Subsequently, the measurement of the generated complex absorption has been conducted at a wavelength of maximum absorption (λ max = 560 nm).

4.The experimental procedure involves sequentially adding 1 mL of a solution of reagent with an amount of 2.523 $x10^{-3}$ M, after modifying the acidity function to a the pH level of 8. This solution of reagent is then inserted into a solution having ions of chromium (III)at concentration of 2.498 $x10^{-3}$ M, which is roughly equal to (50µg/10 mL).a, the amount is supplemented with water that has been deionized to revery a final amount of 10 mL. Regarding the comparative solution, it has been formulated by combining the solutions utilizing similar aforementioned procedures, but substituting the ion of chromium (III) with water that had been deionized. Subsequently, the measurement of the generated complex absorption has been conducted at a wavelength of maximum absorption (λ max = 560 nm).

(2-7-5) Temperature Effect

1- Investigating the temp. impact on the complex of Cobalt (II).

The study aimed to explore the influence of temp on the complex of Cobalt (II).This has been done by preparing a set of amounttrical containers with a capacity of 5 mL. Every flask has been filled with ions of cobalt (II) at concentration of 3.432 x10^{-3} M, which is roughly equal to (50 µg/5mL). Subsequently, a solution of reagent with concentration of 2.523 x10^{-3} M had been inserted to every sample in a amount of 1 mL. The optimum acidic situation has been achieved by adjusting the pH level to 10. The contents of every flask were then transferred to a separate 5 mL amounttric container and diluted with water that has been deionized until the mark has been reveryed. These flasks were placed in a basin of water with temperatures ranging from 10 to 60 degrees Celsius. The complex absorption at its max absorption wavelength has been detemined vs a comparator solution generated in similar manner, but without the presence of ions of Cobalt (II).

2- Investigating the temp impact on complex of the Chromium (III).

The study aimed to explore the influence of temp on the complex of Chromium (III) . This has been done by preparing a set of amounttrical containers with a capacity of 10 mL. Every flask has been filled with ions of Chromium (III) at concentration of (2.523 x10^{-3})M, which is roughly equal to (50µg/10)mL. Subsequently, 1 mL of a reagent with similar amount has been inserted to every flask. The optimum acidic situation has been achieved by adjusting the pH level to 8. The contents of every flask were then transferred to a separate 10 mL amounttric container and diluted with water that has been deionized until the mark has been reveryed. These flasks were placed in a basin of water with temperatures ranging from 10 to 60 degrees Celsius. The complex absorption at its max absorption wavelength has been detemined vs a comparator solution generated in similar manner, but without the presence of ions of Chromium (III).

(2-8) Ionic strength's Effect

Ions ionic strength affects the degree of stability, the rate of reaction, the sensitivity of the determination and the solubility of the reactants. It is important to study this effect as shown below.

1.A series of volumetric flask with a capacity of 5 mL have been utilized to introduce a solution having ions of Cobalt(II) at concentration of 3.432 x10^{-3} M, which is roughly equal to (50 µg/5 mL). Subsequently, 0.5 mL of solutions of sodium sulfate and sodium nitrate had been individually introduced at varying amounts. The amounts of the samples varied between 0.5 and 0.0005 M. Subsequently, a solution of reagent with concentration of 2.523x10^{-3} M had been inserted to every sample in a amount of 1 mL. The amounttrical containers were then filled with water that had been deionized until the desired level has been reveryed. Prior to this, the optimum acidic situations were set to a the pH level of 10 in order to produce the complex of cobalt(II) . The measurement of absorbance has been conducted at the wavelength corresponding to the max absorption of the complex (λ max = 589 nm). This measurement has been performed on a solution of reagent that has been prepared utilizing similar method as a similar solution (Blank), with the exception of the addition of the ion. This procedure has been followed for all the solutions that were detected .

2.A series of volumetric flask with a capacity of 10 mL have been utilized to introduce a solution having ions of Chromium (III) at concentration of 2.498 x10^{-3} M, which is roughly equal to 50 µg/10 mL. Subsequently, 0.5 mL of solutions of sodium sulfate and sodium nitrate had been individually introduced at varying amounts. The amounts of the samples varied between 0.5 and 0.0005 M. Subsequently, a solution of reagent with concentration of 2.523x10^{-3} M had been inserted to every sample in a amount of 1 mL. The amounttrical containers were then filled with water that had been deionized until the desired level has been reveryed. Prior to this, the optimum acidic situations were set to a the pH level of 8 in order to produce the complex of chromium (III). The measurement of absorbance has been conducted at the wavelength corresponding to the max absorption of the complex (λmax = 560nm). This measurement has been performed on a solution of reagent that has been prepared utilizing similar method as a similar solution (Blank), with the exception of the addition of the ion. This procedure has been followed for all the solutions that were detected .

(2-9)The buffer solution impact on every complex's optimum acidic function

1.Two amounttrical containers, every with a amount of 5 mL, were utilized. In every flask, a solution having ions of cobalt (II) at concentration of 3.432 x10^{-3} M, which is equal to (50 µg/5 mL), has been inserted. Subsequently, 1 mL of a solution of reagent with concentration of 2.523x10^{-3} M has been inserted.The optimum acidic function of the cobalt (II) complex has been set to a pH of 10 utilizing buffer solutions produced as described in paragraphs 1 and 2. Following this, the amount had been set to the identify with water that had been deionized and the complex absorption has been determined at its max wavelength of 589 nm versus a solution. It is important to note that the ion has been not inserted to the blank solution, and all determined solutions has been generated in similar manner as the comparable blank solution.

2.Two amounttrical containers, every with a amount of 10 mL, were utilized. In every flask, a solution having ions of chromium (III) at concentration of 2.498x10^{-3} M, which is equal to 50 µg/10 mL, has been inserted. Subsequently, 1 mL of a solution of reagent with concentration of 2.523x10^{-3} M has been inserted. The optimum acidic function of the chromium (III) complex has been set to a pH of 8 utilizing buffer solutions produced as described in paragraphs (2-4). Following this, the amount had been set to the identify with water that had been deionized and the complex absorption has been determined at its max wavelength of 560 nm versus a solution. It is important to note that the ion has been not inserted to the blank solution, and all determined solutions has been generated in similar manner as the comparable blank solution.

(2-10) Calibration curves

(2-10-1) Calibration curve for the Cobalt(II) complex

A set of volumetric flasks (10 mL) were taken and added varied concentrations of cobalt(II) ion solution to them ranging from (9.615 x10^{-5} to 1.016x10^{-3}M) , corresponding to (5 µg/mL - 60 µg/mL) . Then (1 mL) of the reagent solution at a concentration of (2.523x10^{-3}M), and the pH was adjusted (pH = 10) with following the other optimum conditions reached in this study , and the volume was completed with deionized water to the mark , prepared using the same complex process , but substituting an equivalent volume of deionized water for the volume of a cobalt(II) ion solution . The relationship between absorption and concentration was determined by measuring the absorption of all solutions at (λ max= 589 nm) of the complex

(2-10-2) Calibration curve for the Chromium (III) complex

A set of volumetric flasks (10 mL) were taken and added varied concentrations of Chromium (III) ion solution to them ranging from ($8.474x10^{-5}$-$1.355x10^{-3}$M) , corresponding to (5 µg/mL - 80 µg/mL) . Then (1 mL) of the reagent solution at a concentration of (2.523 $x10^{-3}$ M), and the pH was adjusted (pH = 8) with following the other optimum conditions reached in this study , and the volume was completed with deionized water to the mark , prepared using the same complex process , but substituting an equivalent volume of deionized water for the volume of a Chromium (III) ion solution . The relationship between absorption and concentration was determined by measuring the absorption of all solutions at (λ max= 560 nm) of the complex.

(2-11) The complexes' stoichiometry

The investigation of the metallic to ligand proportion (M:L) is conducted under optimum situations employing both the mole proportion approach and the Job technique (continuous differences).

(2-11-1) Job's (continuous variation) method. [83]

1- Cobalt(II) ion complex.

The experimental procedure involves the preparing of two solutions having cobalt(II) complex at an amount of 2 $x10^{-4}$M, with equal amounts of the metallic ion and reagent.These solutions are then reacted with varying amounttric proportions and the acidity function is regulated to a the pH level of 10, while maintaining an equal final amount for every reaction. Subsequently, a curve is plotted correlating the absorbance with the proportion of amount at a suitable highest wavelength of 589 nm for the cobalt(II) complex. The mole proportion of the complex formed can be denoted as the ratio (VL/VM), whereas VM represents the amount of the solution having the ion of metal (Co), and VL represents the amount of the solution of reagent.

2- Chromium (III) ion complex.

The experimental procedure involves the preparing of two solutions having Chromium (III) complex at an amount of 1.5 x10^{-4} M, with equal amounts of the metallic ion and reagent. These solutions are then reacted with varying amounttric proportions and the acidity function is regulated to a the pH level of 8, while maintaining an equal final amount for every reaction. Subsequently, a curve is plotted correlating the absorbance with the proportion of amount at a suitable highest wavelength of 560 nm for the Chromium (III) complex. The mole proportion of the complex formed can be denoted as the ratio (VL/VM), whereas VM represents the amount of the solution having the ion of metal (Cr), and VL represents the amount of the solution of reagent.

(2-11-2) Mole ratio method.[84]

1- Cobalt(II) ion complex.

In this experimental procedure, a series of amounttrical containers with a capacity of 5 mL were utilized. Every flask had been filled with a predetermined and well-established amount of the ion of cobalt(II), specifically 3 x10^{-4}M. The amount of the reagent used has been varied in a proportional manner,ranging from(2.5 x10^{-4}-1.05 x10^{-3})M. Additionally, the pH level had been set to 10 to ensure optimum situations for the experiment.The comparative solutions have been made utilizing a similar methodology as the complex preparing, with the exception that the amount of solution cobalt(II) ion had been substituted with an equal amount of water that had been deionized.The compound exhibits maximum absorption at a wavelength of 589 nm.

2- Chromium (III) ion complex.

In this experimental procedure, a series of amounttrical containers with a capacity of 10 mL were utilized. Every flask had been filled with a predetermined and well-established amount of the ion of chromium (III), specifically 3.786 x10^{-4} M. The amount of the reagent used has been varied in a proportional manner,ranging from (2.15 x10^{-4}-1.351 x10^{-3})M. Additionally, the pH level had been set to 8 to ensure optimum situations for the experiment.The comparative solutions have been made utilizing a similar methodology as the complex preparing, with the exception that the amount of solution chromium (III)ion had been substituted with an equal amount of water that had been deionized.The compound exhibits maximum absorption at a wavelength of 560 nm.

(2-12)Calculating of the degree of thermodynamic functions, stability constant,and dissociation of the two formed complexes .[85,86]

1-Complex of Cobalt(II).

Two volumetric flasks were taken (10 mL). The first flask has been filled with a solution having of cobalt (II) ions at an amount of 3.0x10^{-4} M. To this, 1 mL of a reagent with an amount of 6.0x10^{-4}M has been inserted. The pH of the solution has been regulated to 10 utilizing an appropriate acidic modifier. Water that had been deionized has been then inserted to the first flask until it reveryed the mark on the flask, resulting in a final amount of 5mL. Similarly, the second the amounttric flask has been filled with a solution having of cobalt (II) ions at an amount of 3 x10^{-4}M. Subsequently,1mL of a reagent with an amount of 1.0 x10^{-3}M has been inserted to the flask. The pH of the solution has been regulated to 10 utilizing an appropriate acidic modifier. The solution from the second flask has been then transferred to a new 10 mL amounttric flask and water that had been deionized has been inserted until it reveryed the mark on the flask, resulting in a final amount of 5 mL. The two amounttrical containers were immersed in a water basin with a temp range of (10 – 30) degrees Celsius. The solutions absorption has been then determined at the respective wavelengths of maximum absorption for every temp, utilizing the comparator solution as a reference. The magnitude of the dissociation constant has been determined by utilizing the absorption magnitudes obtained from the first amounttric flask (As) and the second amounttric flask (Am).

2- Chromium (III) ion complex.

Two volumetric flasks were taken (10 mL). The first flask has been filled with a solution having of cobalt (II) ions at an amount of $3.0x10^{-4}$ M. To this, 1 mL of a reagent with an amount. The first flask has been filled with a solution having chromium (III) ions at an amount of 4.51 $x10^{-5}$ M. To this, 1 mL of a reagent with an amount of 1.353 $x10^{-4}$ M has been inserted. The pH of the solution has been regulated to 8 utilizing an appropriate acidic modifier. deionized Water has been then inserted to the first flask until it reveryed the mark on the flask, resulting in a final amount of 10 mL. Similarly, the second the amounttric flask has been filled with a solution having chromium (III) ions at an amount of 4.51 $x10^{-5}$ M. Subsequently, 1 mL of a reagent with an amount of 3.21 $x10^{-4}$ M has been inserted to the flask. The pH of the solution has been regulated to 8 utilizing an appropriate acidic modifier. The solution from the second flask has been then transferred to a new 10 mL amounttric flask and water that had been deionized has been inserted until it reveryed the mark on the flask, resulting in a final amount of 10 mL. The two amounttrical containers were immersed in a water basin with a temp range of 10 to 30 degrees Celsius. The solutions absorption has been then determined at the respective wavelengths of maximum absorption for every temp, utilizing the comparator solution as a reference. The magnitude of the dissociation constant has been determined by utilizing the absorption magnitudes obtained from the first amounttric flask (As) and the second amounttric flask (Am).

(2-13) Impact of masking agents, anions and cations interferences

(2-13-1) Cations interferences Interaction

1-Determinating ions of Cobalt(II) with some interferences cations ions.

In the present investigation, the identification of the cobalt(II) ion has been achieved by introducing solutions of other metal ions that have the potential to disrupt the analysis of the cobalt(II) ion. This process has been carried out utilizing a set of amounttrical containers, every with a capacity of 5 mL.A solution having ions of cobalt(II) at an amount of 50 µg/5 mL has been prepared. Subsequently, metal ion solutions (Cd^{2+}, Cu^{2+}, Cr^{3+}, Fe^{3+}, Pb^{2+}, Ba^{2+}, Mg^{2+}, Ni^{2+}, Hg^{2+}) at amounts of (50 µg/5 mL) and (100 µg/5 mL) were inserted individually to the cobalt(II) ion solution.To this mixture,a solution of reagent at an amount of 2.523×10^{-3} M, with the pH adjusting to 10, had been inserted to produce a Co(II) complex. The amount followed by adjusting to the mark with water that has been deionized, and the absorbance has been detemined at a wavelength of 589 nm against a contrasted solution (Blank)

2- Determenating ions of Chromium (III) with some interference cations ions.

In the present investigation, the identification of the chromium (III) ion has been achieved by introducing solutions of metal ions that have the potential to disrupt the chromium (III) ion being investigated. This process has been carried out utilizing a set of amounttrical containers with a capacity of 10 mL.A solution having chromium (III) ions at an amount of 50 µg/10 mL has been introduced. This has been followed by the addition of metal ion solutions (Cd^{2+}, Cu^{2+}, Cr^{3+}, Fe^{3+}, Pb^{2+}, Ba^{2+}, Mg^{2+}, Ni^{2+}, Hg^{2+}) at amounts of (50 µg/10 mL) and (100 µg/10 mL), individually, which have the potential to interfere with the chromium (III) ions. Subsequently, a solution of reagent at an amount of 2.523 $x10^{-3}$ M, with the pH regulated to 8, has been inserted in a amount of 1 mL to form a complex with the ions of Cr(III). The final amount had been modified to the mark with water that had been deionized, and the absorbance has been determined at a wavelength of 560 nm against a contrasted solution (Blank).

(2-13-2) Interaction of anions interference

1-Determination ion Cobalt (II) with certain interference anions ions.

In the present investigation, the determination of the ion of cobalt(II) has been carried out by introducing solutions of anions which had the potential to disrupt the analysis of the ions of cobalt(II). This has been achieved by employing a set of amounttrical containers with a capacity of 5 mL.A solution having cobalt (II) ions at an amount of 50 µg/5 mL had been prepared. Subsequently, 1 mL of anion solutions (CN^{1-}, CO_3^{2-}, CrO_7^{2-}, Cl^{1-}, SCN^{1-}, Br^{1-}, SO_4^{2-}) at amounts of (50 µg/5 mL) and (100 µg/5 mL) were inserted independently to the solution of cobalt (II) ion. To produce a complex of Co (II), 1 mL of a solution of reagent at an amount of $2.523x10^{-3}$ M has been inserted, and the acidity function has been regulated to pH 10. The final amount had been adjusting with water that had been deionized, and the magnitude of absorbance has been detemined at a wavelength of 589 nm against a contrasted solution (Blank).

2-Determenation ion of Chromium (III) with some interference anions ions.

In the present investigation, the determination of the Chromium (III) ion has been carried out by introducing solutions of anions that have the potential for interfering with the ions of Chromium (III) being investigated. This has been accomplished by utilizing a set of amounttrical containers with a capacity of 10 mL.A solution having ions of Chromium (III) at an amount of 50 µg/10 mL has been prepared. Subsequently, 1 mL of various anion solutions (CN^{1-}, CO_3^{2-}, CrO_7^{2-}, Cl^{1-}, SCN^{1-}, Br^{1-}, SO_4^{2-}) at amounts of 50 µg/10 mL and 100 µg/10 mL were separately inserted to the Chromium (III) ion solution to assess potential interference. Following this, 1 mL of a solution of reagent with an amount of 2.523×10^{-3} M has been inserted to the mixture, with the pH regulated to 8 to facilitate the generation of a Cr(III) complex. The amount has been then regulated to the desired level utilizing water that had been deionized, and the absorbance has been detemined at a wavelength of 560 nm against a blank solution for contrasted.

(2-13-3) Optimum masking agents . [87]

1- Cobalt(II) ion complex.

The investigation focused on examining the impact of the agents of masking, as described in paragraphs (2-4), on the substance absorption under investigation. This has been achieved by filling a set of amounttrical containers (5mL) with a cobalt (II) ion solution at an amount of 50 µg/5mL. Subsequently, separate additions of 1 mL of the different agent of masking solutions were made to every flask. Following this, the optimum amount of 1 mL of a solution of reagent, with an amount of 2.523×10^{-3} M, has been inserted to every flask. Finally, the amount has been regulated to the mark with water that has been deionized. The acidic function had been regulated to pH 10 in order to produce the Co(II) complex. The absorption of the complex has been then detected at a wavelength of 589 nm. when examining the comparator solution (Blank). Following the determination that the impact of agents of masking on the complex of cobalt (II) absorption remains inconclusive, a research investigation has been conducted to identify the optimum agent of masking and its corresponding size for every one of the ions that interfere with the cobalt (II) ion.

2- Chromium (III) ion complex

The investigation aimed to examine the impact of the agents of masking described in paragraphs (2-4) on the compound absorption being studied. This has been achieved by filling a set of amounttrical containers (10 mL) with a solution having Chromium (III) ions at an amount of (50 μg/10 mL). Subsequently, (1 mL) of different agent of masking solutions has been inserted independently to every flask. (1 mL) of the solution of reagent, with an amount of (2.523 x10^{-3} M), has been inserted to every flask, and the amount has been then completed to the mark utilizing water that had been deionized. The acidic situations were modified to a pH of 8 in order to produce the Co(II) complex. The resulting absorption has been determined at a wavelength of 560 nm. when examining the comparator solution (Blank), Following the recognition that agents of masking have no significant impact on the Chromium (III) complex absorption, a research investigation has been conducted to determine the optimum agent of masking and its corresponding size for every single one of the ions that interfere with the Chromium (III) ion.

(2-13-4) Utilizing a more effective masking agent to identify ions of chromium (III) and cobalt (II) when cations are present as interference

1- Cobalt (II) ion complex

The experiment involved utilizing a set of amounttrical containers with a capacity of 5 mL. The flasks were filled with cobalt (II) ions at an amount of 50 µg/5 mL. Subsequently, 1 mL of cations ions at an amount of 50 µg/5 mL had been inserted along with 1 mL of an agent that masks at an amount of 0.1 M. Additionally, 1 mL of a solution of reagent with an amount of 2.523 x10^{-3} M had been inserted. The solution acidity had been regulated to achieve a pH of 10, and the remaining amount has been filled with water that had been deionized up to the mark. The absorption at a wavelength of 589 nm has been determined in contrast with the solution having the comparator.

2- Chromium (III) ion complex

The experiment involved utilizing a set of amounttrical containers with a capacity of 10 mL. The flasks were filled with a solution having Chromium (III) ions at an amount of 50 µg/10 mL. Subsequently, 1 mL of cations ions at an amount of 50 µg/10 mL has been inserted, along with 1 mL of a agent of masking solution at an amount of 0.1 M. Additionally, 1 mL of a solution of reagent at an amount of 2.523x10^{-3}M had been inserted. The solution of acidity has been regulated to a pH of 8, and the remaining amount has been completed with water that had been deionized up to the identify on the flask. The absorption at a wavelength of maximum absorption (λmax = 560 nm) has been determined When comparing the solution having the comparator.

(2-14) Statistical treatment of the results

(2-14-1) Precision. [88]

Determiniy the precision of the analytical methods by calculating the standard deviation (S.D.) , and percentage relative standard deviation (RSD %), for four reading and three various concentrations of each metal ion, after which the absorption was measured at maximum wave length for each complex against a comparator solution prepared under the same conditions.

$$S.D = \sqrt{\frac{\sum(x_i - \bar{x})}{n-1}} \text{ ……………(2-1)}$$

$$RSD\% = \frac{S.D}{x`} \times 100 \text{ …………(2-2)}$$

S.D =Standard deviation

RSD% =Relative standard deviation

n =Total number of the values

x_i =The value in the data absorption

$\bar{x}$ =The sample mean

(2-14-2) Accuracy [89]

By applying the best conditions for the calibration curves obtained from previous studies, the synthetic reagent was used to determine the control of the method that was used for the determination of these metal ions.the percentage error (Ere %). was calculated from the relationship.

$$E_{re}\% = \frac{E}{X_t} \times 100 \text{ ……..(2-3)}$$

$$E = X_i - X_t \text{ ……(2-4)}$$

$$Re\% = 100 \pm E_{re}\% \text{ …...(2-5)}$$

E= The difference between analytical reading and truth.

X_t= The real value.

(2-15)Preparation of the solid complex ions of Chromium (III) and Cobalt(II)

(2-15-1)The solid Cobalt(II) complex

The complex of Cobalt (II) has been prepared in a proportion of 1:2 by combining (0.250 mmole) (0.0991 g) of the reagents BMPQYE1 with (0.125 mmole) (0.0364 g) of ion of cobalt (II) dissolved in 10 mL of water that had been deionized and 10 mL of ethanol. The cobalt (II) ion solution has been introduced into a circular flask and subjected to continuous stirring utilizing an instrument. Subsequently, the solution of reagent has been incrementally inserted while maintaining continuous stirring for a duration of two hours. The pH of the system has been regulated to 10, indicating an acidic environment. Following a single day of drying at ambient temp, the ethanol solvent yielded a discernible dark brown precipitate. Subsequently, the precipitate has been subjected to filtration and subsequently washed with water that had been deionized. The resulting material has been then subjected to recrystallization utilizing ethanol and drying in the air.

(2-15-2)The solid Chromium (III) complex

The complex of Chromium (III) has been generated in a molar proportion of 1:3 by combining (0.375 mmol) (0.1487 g) of the reagent BMPQYE1 with (0.125mmol) (0.0500 g) of Chromium (III) ion, which has been dissolved in 10 mL of water that had been deionized, in the presence of 10 mL of ethanol. The solution having Chromium (III) ions is introduced into a flask of circular shape, whereas it is subjected to continuous stirring utilizing an instrument. Subsequently, the solution having the reagent has been incrementally inserted to the flask, while the stirring process continues for a duration of two hours. During this time, the pH of the solution is maintained at a magnitude of 8 by adjusting its acidity. Following a 24-hour period of drying at ambient temp, the ethanol solvent yielded a discernible dark brown precipitate. Subsequently, this precipitate underwent filtration and has been subjected to washing utilizing water that had been deionized. The resulting material has been then subjected to recrystallization utilizing ethanol and drying in the air.

(2-16) Determination the melting point of the complexes of Chromium (III), Cobalt(II),and reagent .[90]

The objective of measuring the reagent and the complexes of Chromium (III) and cobalt(II) melting point, as described in paragraph (2-15), has been to determine the process of creation of the complexes of chromium and cobalt by comparing their melting points with that of the reagent.

(2-17) Molar conductivity Measurement of the, complexes of Chromium (III) and Cobalt(II). [91]

The cobalt(II) complicated solution has been prepared at an amount of 0.001M by dissolving 0.006 g of the complicated precipitate in 10 mL of water that had been deionized. The molecular conductivity of the solution has been then detected at the ambient temp. Similarly, the procedure has been repeated for the chromium (III) complicated with an amount of 0.001M.This has been achieved by dissolving 5.2 g of the chromium (III) complicated precipitate in 10 mL of water that had been deionized. Subsequently, the conductivity by molar amount of the solution has been determined at room temp.

(2-18) Estimation spectroscopy

1-UV-Vis Spectrophotometer

2-Investigation of the infrared spectra (FTIR)

The infrared spectra have been obtained for the reagent BMPQYE1, the complex of Chromium (III), and the complex of Cobalt(II).

3-Nuclear Magnetic Resonance Spectroscopy (^{1}H.NMR , ^{13}C.NMR)

The hydrogen and carbon atoms number in a compound have been identified utilizing nuclear magnetic resonance (NMR) spectroscopy, with dimethyl sulfoxide (DMSO) serving as the solvent.

4-GC.Mass

Utilize diamond spectrometry to ascertain the molecular-weights of the reagent through the implementation of the ablation procedure.

5-Atomic Absorption Spectrophotometer

Atomic absorption spectroscopy (AAS) is a widely used analytical technique in the field of chemistry. It is the utilization of a spectrophotometer apparatus for the purpose of identifying ion atoms within a complex.

(2-19) Application

Vitamin B_{12} and a sample of hair were used to estimate the cobalt(II) ion, and drugs samples were used to estimate the chromium (III) ion.

(2-19-1) Determination of Cobalt(II)ion in vitamin B_{12} Sample [92].

An ampule of Vitamin B_{12} having 1000µg/4mL of cyanocobalamin (43.471µg(Co^{2+})/4mL) had been utilized. The ampule has been thoroughly shaken before extracting 4mL of the Vitamin B_{12} solution into a beaker. Subsequently, 8mL of nitric acid (1:1) has been inserted to the beaker and the mixture has been heated until it became dry. Following this, 8mL of hydrochloric acid (1:1) had been inserted to the beaker and heated again until it reveryed a near-dried state. The specimen has been collected and subsequently mixed with water that had been deionized. The resulting mixture has been then transferred to a amounttric flask with a capacity of 10 mL. The amount has been then regulated to the mark on the flask by adding additional water that had been deionized. Next, a 1 mL sample of Cobalt(II)estimat has been obtained from the reagent BMPQYE1 and its pH has been regulated to 10.

(2-19-2) Determination of cobalt(II)ion in hair Sample[93].

Human hair has been obtained from the scalp and subjected to a series of procedures. Initially, the hair specimen, weighing 1g, has been cleansed utilizing 2 milliliters of acetone, followed by two subsequent washes with 1 milliliter of water that had been deionized. Subsequently, the hair specimen has been allowed to air dry for a duration of 20 minutes at room temp. The specimen had been subjected to digestion utilizing 1 mL of concentrated nitric acid. additionally, the digested specimen has been transferred to a 50 mL amounttric flask and diluted with water that had been deionized until the flask has been filled to the mark. The Cobalt(II) amount in the solution has been determined at pH 10 utilizing a 1 mL aliquot of the reagent (BMPQYE1).

(2-19-3) Chromium (III) ion determination of in drugs[94]

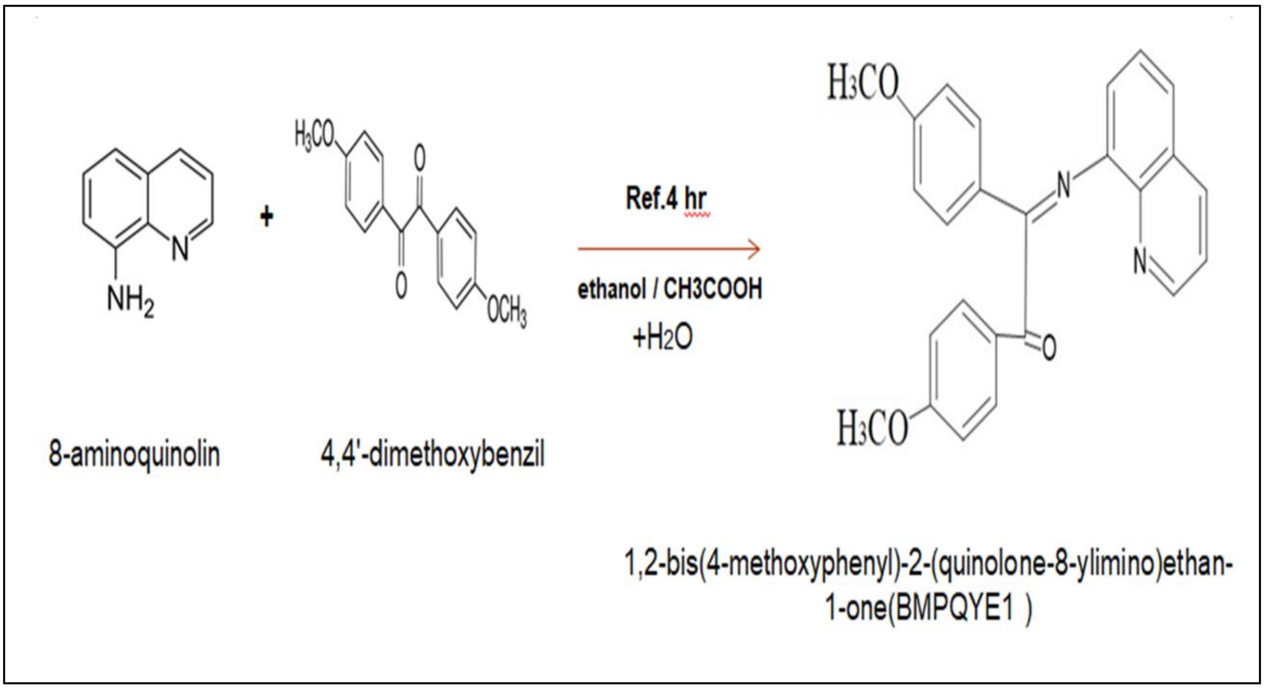
The drug tablet under consideration contains the Chromium (III) ion. AZmultiway and Maddovit tablets, every having (25 µg) (Cr^{3+}) and (35 µg) (Cr^{3+}) respectively, were individually ground after ten pills of every kind had been consumed. Upon metabolizing the two pharmaceutical substances. Subsequently, the solution has been diluted to a final amount of 100 mL by adding water that had been deionized. The Chromium (III) ions present in every drug solution were transferred into a 10 mL amounttric flask. To this, 1 mL of a solution of reagent with an amount of $2.523x10^{-3}$ M had been inserted. The pH of the solution has been regulated to 8, in accordance with the optimum situations established in the current investigation. Finally, the amount has been made up to the mark with water that had been deionized. The preparing involves employing an identical intricate procedure, albeit replacing the quantity of the medication solution with a comparable amount of water that has been deionized.

Chapter Three

Results
and
Discussion

(3-1) Reagent Preparation

The new Schiff bases reagent 1,2-bis(4-methoxyphenyl)-2-(quinolone-8-ylimino) ethan-1-one(BMPQYE1) was prepared by reacting 8-aminoquinoline with 4,4'-dimethoxybenzil and the yield was 78% with a dark reddish-brown color as shown below.



Scheme (3- 1): Preparation of (BMPQYE1) ligand

(3-2) Dissolvability of the reagent BMPQYE1.

Tests for reagent solubility in a wide variety of solvents are shown in table (3-1).

Table (3-1): The solubility of the reagent in various solvents

Solvent	Result	Solvent	Result
H_2O	†	2-Propanol	-
$CHCl_3$	+	THF	+
Ethanol	+	Acetonitrile	+
Ethylacetate	+	DMF	+
1-Propanol	-	Acetone	†
Methanol	+	DMSO	+

(+):soluble , (†) :partial solubility ,(-) : insoluble

(3-3)The material's (BMPQYE1) properties

The molecular and physical properties of (BMPQYE1) are detailed in table (3-2).

Table (3-2) :The characteristics of (BMPQYE1).

Compound	Formula	Molecular weight (g/mole)	Melting pointe (^{o}C)	Color
BMPQYE1	$C_{25}H_{20}N_2O_3$	396.4460	127-130	reddish-brown

(3-4)Research on the reagent's (BMPQYE1) spectroscopy

(3-4-1) The reagent has a (UV-Vis) spectrum.

The ($n\rightarrow\pi^*$) transition (figure) corresponds to the absorption maximum of the reagent solution at (λmax =467 nm). (3-1) [98].

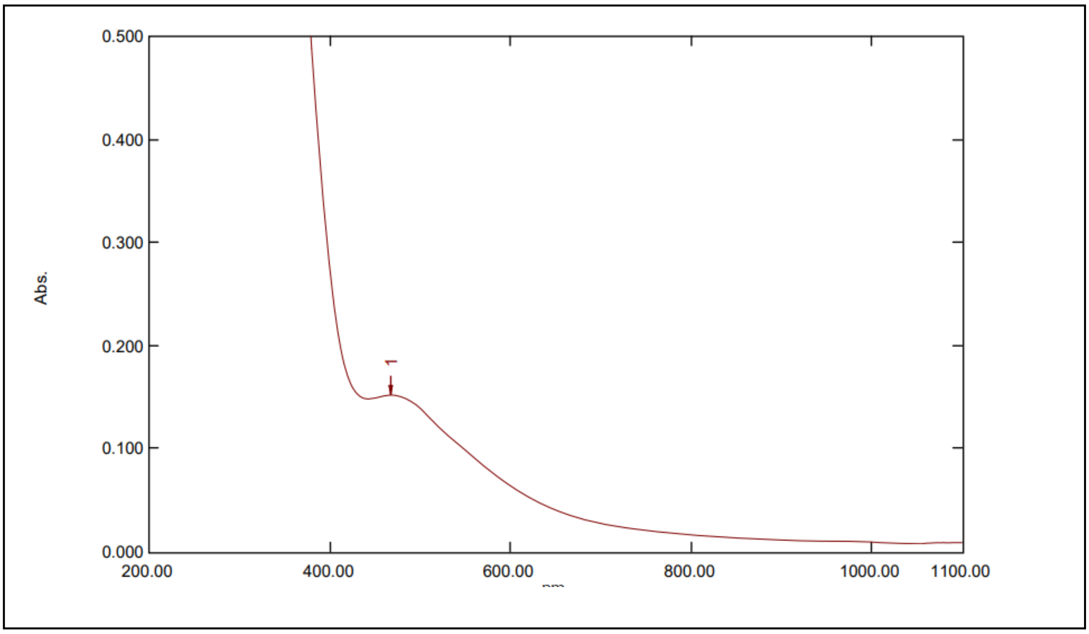


Figure (3-1) : UV-Visb.spectrum of reagent[BMPQYE1]

(3-4-2) FT-IR Spectra for the reagent and its raw materials

FTIR spectroscopy is one of the most widely used diagnostic and research equipment,the following is the results of an infrared spectroscopy investigation of chemicals and their complexes formed in the range (400-4,000) cm^{-1}.

1-FTIR spectrum information for 8-aminoquinoline.

The spectrum of 8-aminoquinoline by FTIR spectroscopy as shown in figures (3-2).

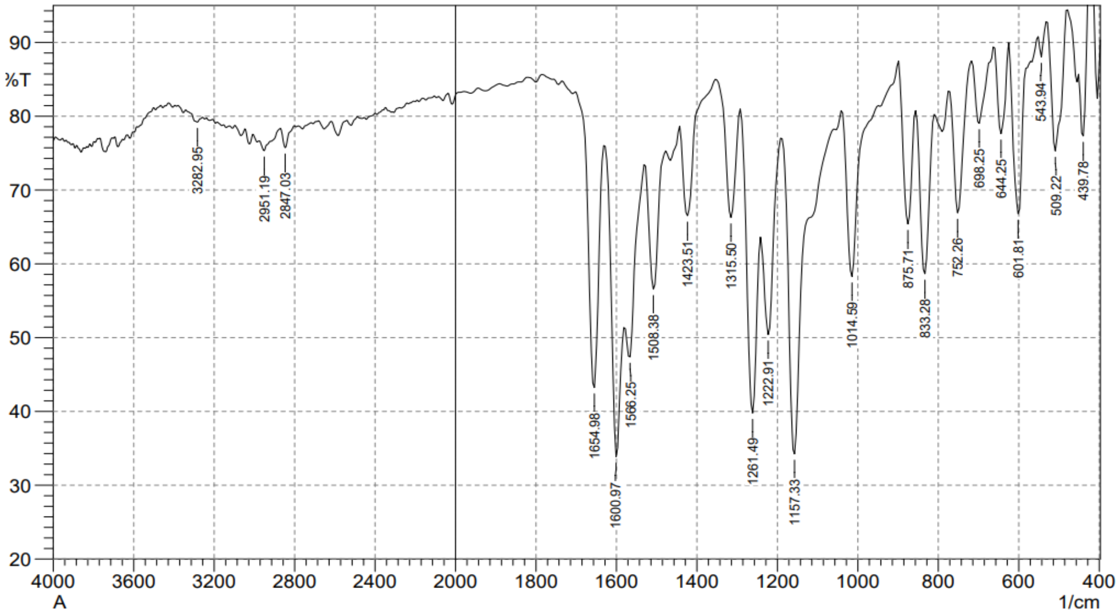


Figure (3-2): FT-IR spectrum of 8-aminoquinoline

2-FT-IR spectrum of 4,4'-dimethoxybenzil

The spectra of 4,4'-dimethoxybenil is shown in figure (3-3).

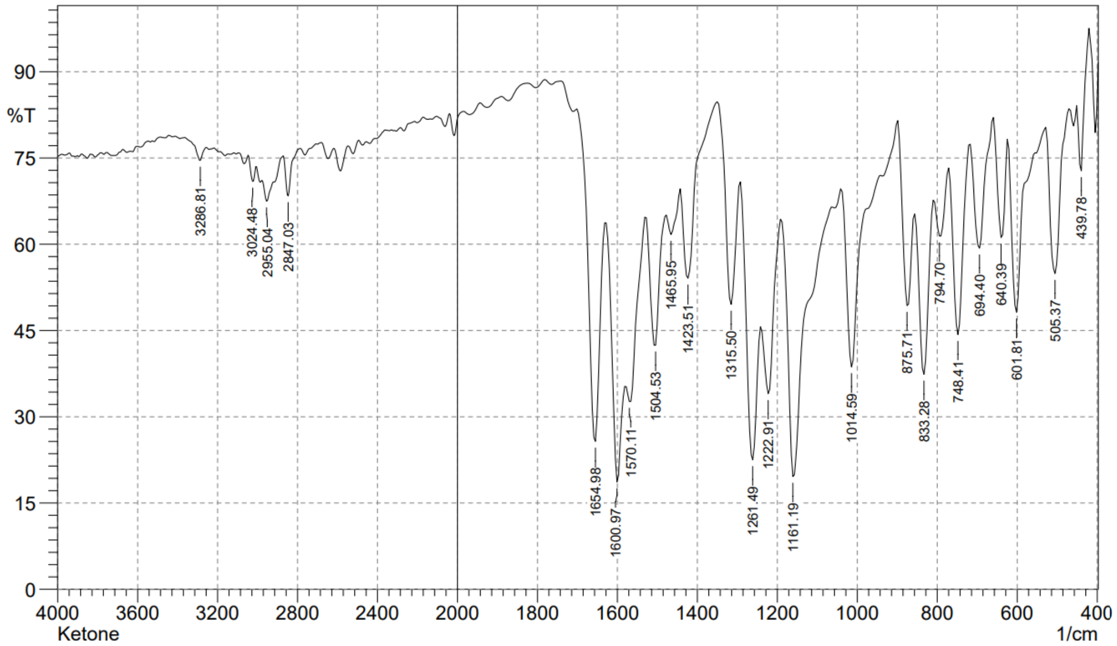


Figure (3-3) FT-IR spectrum of 4,4'-dimethoxybenil

3- FT-IR spectrum study of a reagent (BMPQYE1)

Chemical (BMPQYE1) FTIR spectra are shown in figure (3-4).

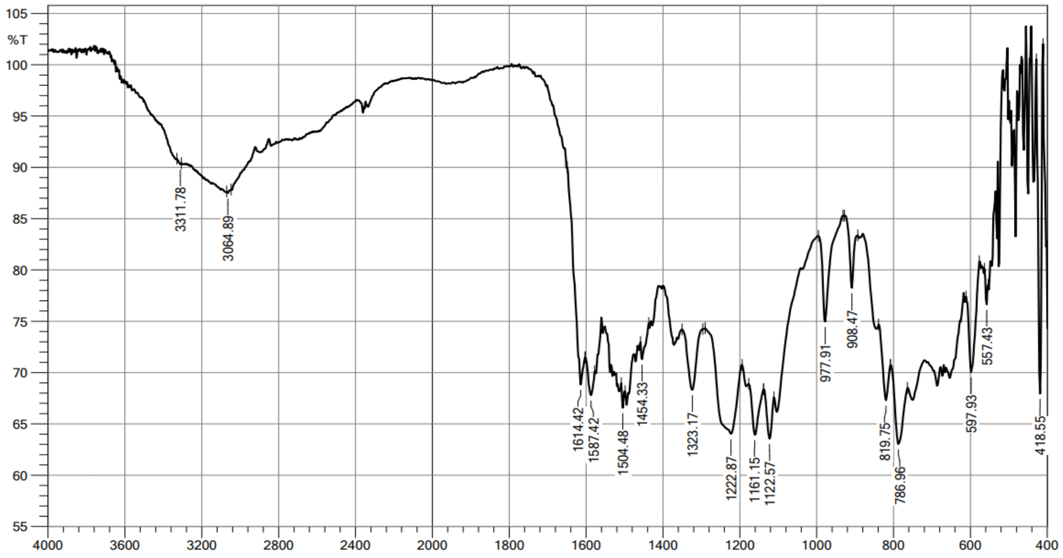


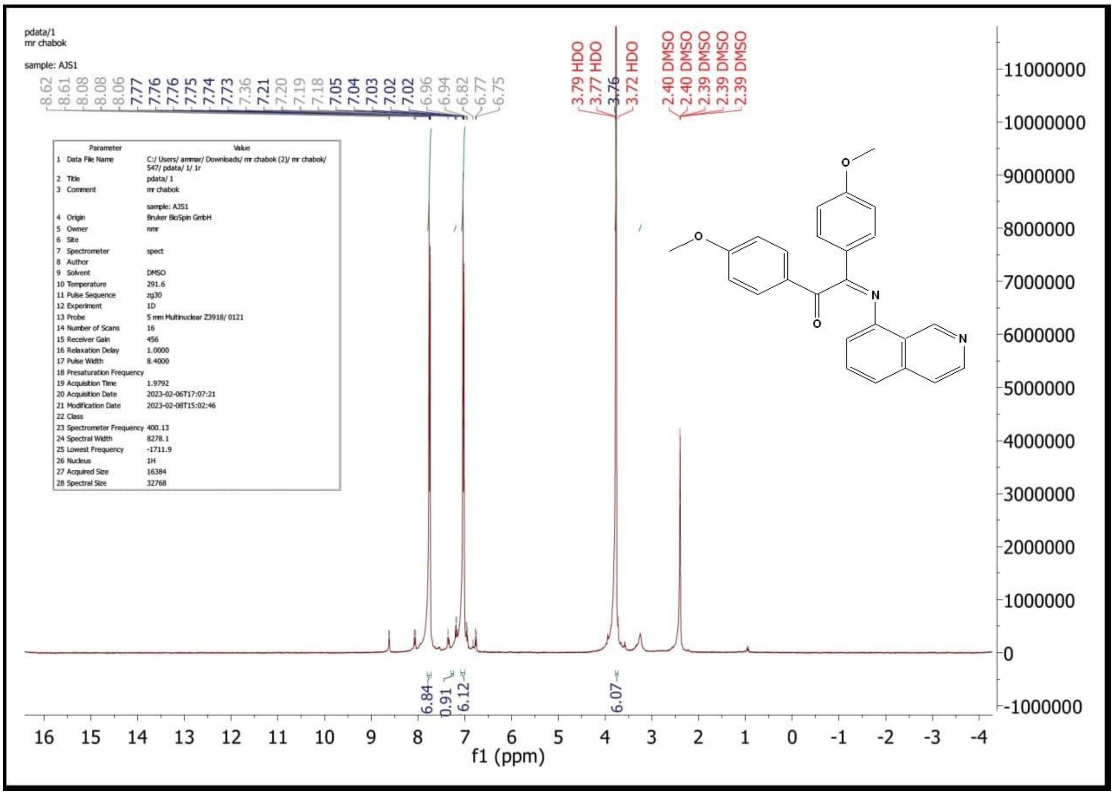
Figure (3-4): FT-IR spectrum of reagent (BMPQYE1)

Table (3-3): The major data of FT-IR spectrums of the reagent and its raw materials. [95-97]

Band	8-amino Quinolone	4,4'-dimethoxybenil	(BMPQYE1) reagent
st.(C=C)	1566.20	1570.11	1504.48
st.(C=N) aromatic	1614.42	--------	1614.42
st.(C=O)	--------	1654.98	1587.42
st.(C-H) aromatic	3035.96	3024.48	3064.89
st.(C-O)	----------	1427.32	1465.95
st.(N-H)	3311.78	----------	3311.87
st.(C-N)	3348.42	----------	3348.42

(3-4-3) ^{1}HNMR spectrums for the reagent

The spectroscopic (^{1}HNMR) and (^{13}CNMR) spectra of the reagent figures (3-5), (3-6).



pdata/1
mr chabok

sample: AJS1

Parameter	Value
1 Data File Name	C:/ Users/ ammar/ Downloads/ mr chabok (2)/ mr chabok/ 547/ pdata/ 1/ 1r
2 Title	pdata/ 1
3 Comment	mr chabok sample: AJS1
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	291.6
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	5 mm Multinuclear Z3918/ 0121
14 Number of Scans	16
15 Receiver Gain	456
16 Relaxation Delay	1.0000
17 Pulse Width	8.4000
18 Presaturation Frequency	
19 Acquisition Time	1.9792
20 Acquisition Date	2023-02-06T17:07:21
21 Modification Date	2023-02-08T15:02:46
22 Class	
23 Spectrometer Frequency	400.13
24 Spectral Width	8278.1
25 Lowest Frequency	-1711.9
26 Nucleus	1H
27 Acquired Size	16384
28 Spectral Size	32768

Figure (3-5) ^{1}HNMR spectra of reagent (BMPQYE1)

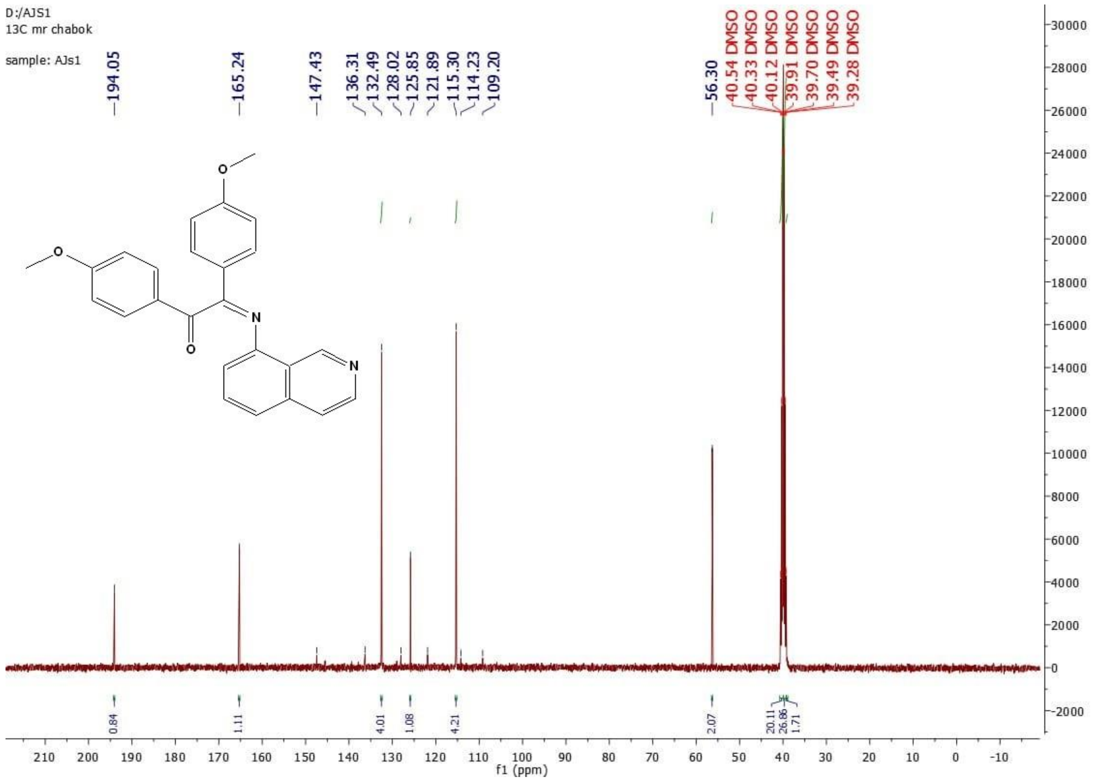


Figure (3-6) ^{13}CNMR spectra of reagent (BMPQYE1)

Proton nuclear magnetic resonance spectrometry is one of the most effective methods to identify the chemical structure of organic compounds. The structure of the reagent (BMPQYE1) was further confirmed by 1D NMR spectroscopy. The 1H-NMR and 13C-NMR spectra of the ligand (L) were tested using DMSO-d6 as a solvent. The 1H NMR spectrum of reagent (SDPIHN) is presented in Figures (3-5) which show peaks that indicate signals originating from of protons. A singlet at 0.84 of (C=O). While a singlet at 1.11 of (C=N). The multiple signals at δ 1.08-4.01 ppm were attributed to ten aromatic protons of t (m, 10H, Ar-H). Due to these two rings being symmetric, all protons bonded to these two rings are equivalent and have the same environments. The reason that H19, H20, H25, and H26 were seen more down-field compared to other protons of these two rings is experienced the diamagnetic anisotropy effect of the vinyl group, which is considered a deshielding group.The singlet at 2.07 was assigned to O-CH3.

(3-4-4) GC.Mass for the reagent

Can be observed from comparing the theoretically estimated molecular weight (396.446 g/mol) with the actual result (presented in Figure 3-7) for the reagent (BMPQYE1).

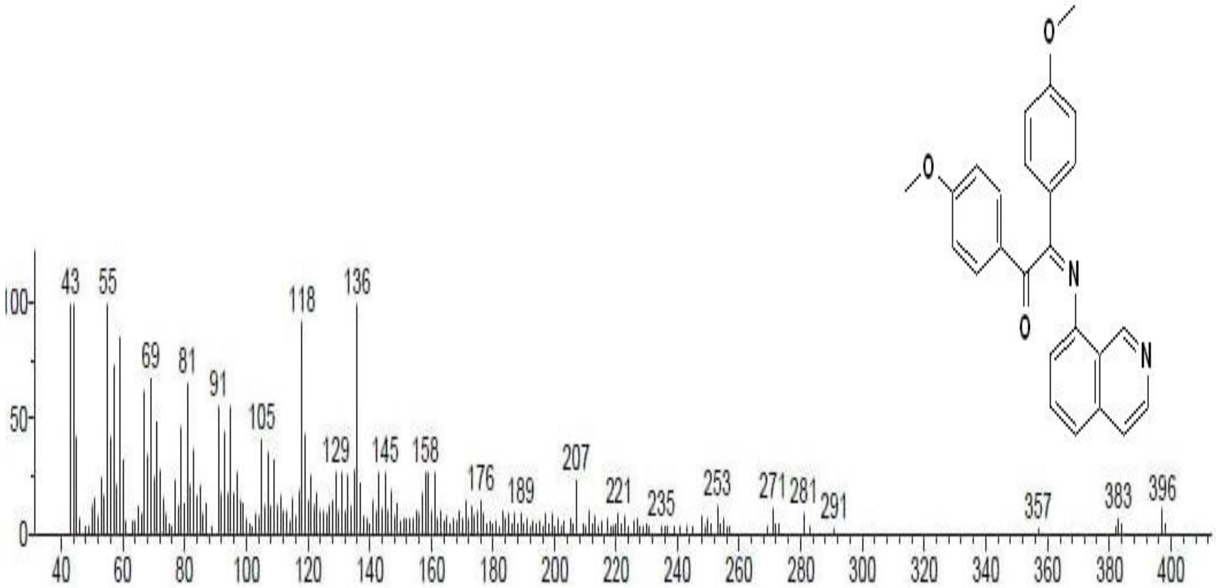


Figure (3-7) GC.Mass spectrum of reagent (BMPQYE1)

(3-5) Using different metal ions in simple tests and to known out which metal ions interact most strongly with the reagent (BMPQYE1) and how the reagent responds to those interactions

The reaction of (11) metal ions with the reagent has been the subject of a great deal of research. According to the results of these tests, the hue shift occurred when metal ion solutions were added to the reagent solution. When the following ions were present in the solution: (Fe^{3+}, Zn^{2+},Cd^{2+}, Ag^{2+}, Co^{2+}, Hg^{2+}, Pb^{2+}, Cr^{3+}, Mg^{2+}, Ni^{2+} and Cu^{2+}), the existence of colored solutions showed that a reaction had occurred between the reagent and the ions. Table (3-4) displays the findings of the spectrum assessment and analysis of cobalt (II) and chromium (III) ions.

Table (3-4): The results of the preliminary test for the reaction of the reagent with the cobalt (II) and chromium (III) ions

Ion	Color befor adding	Color after adding in base medium	Color after adding in acid medium
Co(II)	Pink	Light Reddish brown	Light brown
Cr(III)	Blue	Reddish brown	Light red

(3-6) Study of UV-visible spectra of complexes.

(3-6-1) Study of UV-visible spectrum of Co(II) complex with (BMPQYE1) reagent

Absorption spectra were taken between (350-1100 nm) for the cobalt (II) reagent mixture figure (3-8). The complex's absorption maximum (max= 589nm) indicates a notable rise in absorbance and a bathochromic shift of roughly (122 nm), following creation of the cobalt (II) complex.

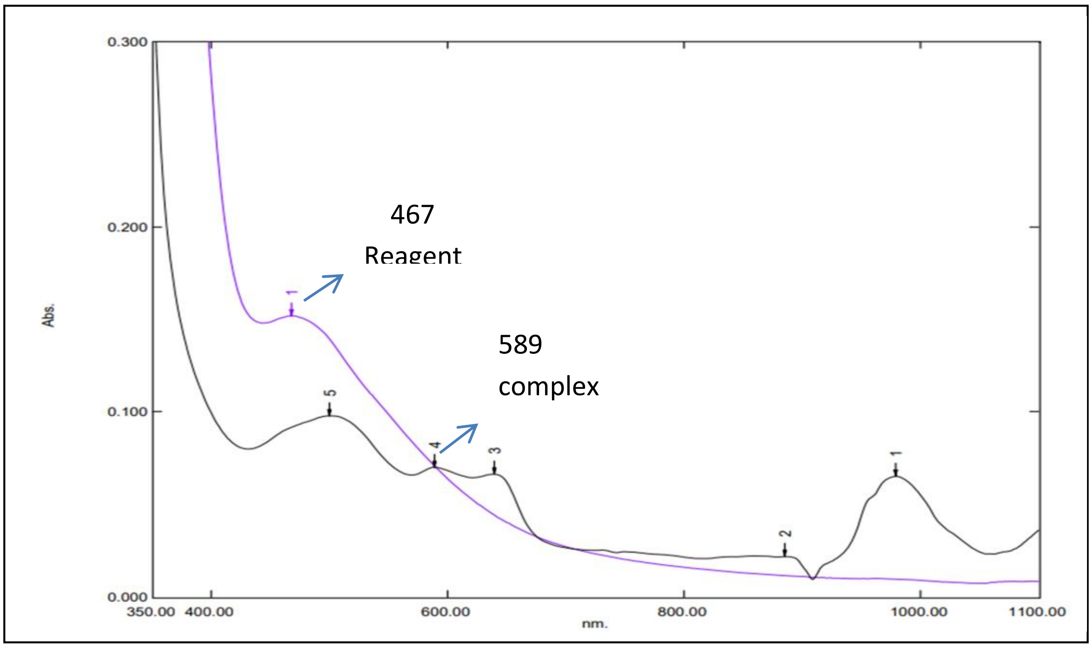


Figure (3-8): Co(II)-[BMPQYE1] complex UV-visible spectrum

Figure (3-8) shows the spectrum of the reagent with the Co complex, where the spectrum line of the reagent is in violet color with a peak of 467 and the spectrum line of the complex in black with a peak of 589, number 4 in the line.

(3-6-2) Study of UV-visible spectrum of Cr (III) complex with [BMPQYE1] reagent

The reagent-chromium (III) combination absorbs light between 200 and 1100 nm seen in figure (3-9). An rise in absorbance and a bathochromic shift of around (93 nm) are seen upon complex formation for the chromium (III) complex, with an absorption maximum of (max= 560nm).

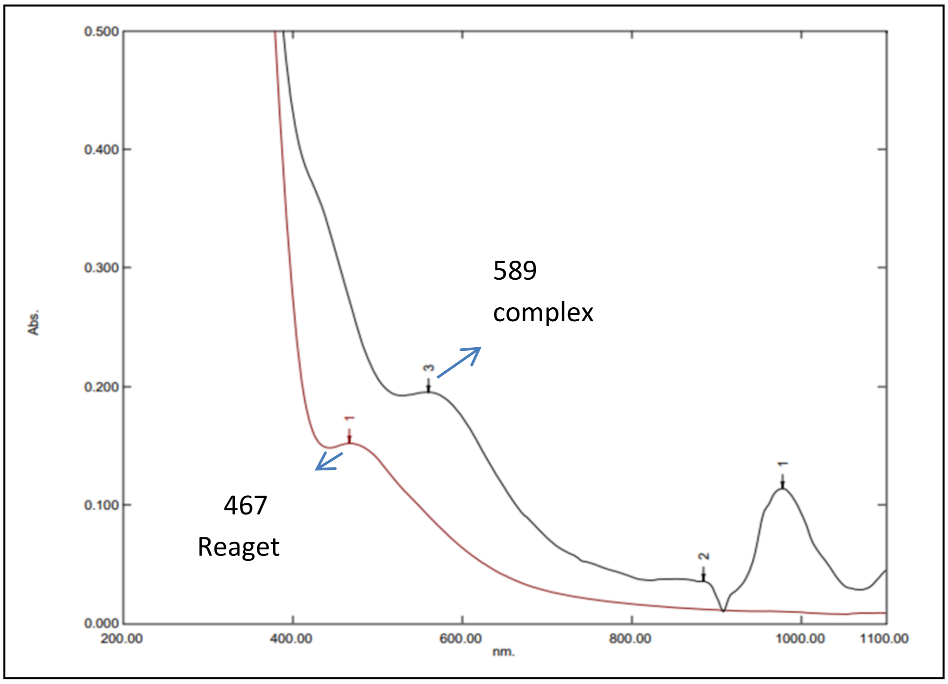


Figure (3-9): Cr(III)-[BMPQYE1] complex UV-visible spectrum

Figure(3-9)shows the spectrum of the reagent with the Cr complex, where the spectrum line of the reagent is in red, with a peak of 467, and the spectrum of the complex is in black, with a peak of 560, number 3 in line.

(3-7) Optimization of Reaction Conditions

(3-7-1) Reagent volume effect

The effect of reagent solution volume on the synthesis of cobalt (II) and chromium (III) complexes was studied. Tables (3-5) and (3-6) demonstrate that the adsorption values for cobalt(II) in pH=10 and

Chromium (III) in pH = 8 rise until saturation when the reagent solution is added. The metal ion's reaction with the reagent causes a saturation of color as it travels toward the composite. According to the results shown, the value of the results is close and then confirmed after 1 ml..

Table (3-5) : Effect of reagent volume on Co (II) complex

Volume of reagent(mL)	0.4	0.5	0.6	0.8	1	1.5	2
Abs.	0.394	0.452	0.488	0.503	**0.521**	0.521	0.518

Table (3-6) : Effect of reagent volume Cr (III) complex

Volume of reagent(mL)	0.4	0.5	0.6	0.8	1	1.5	2
Abs.	0.275	0.281	0.286	0.325	**0.390**	0.376	0.362

(3-7-2) Effect of pH value

Figures (3-10) and (3-11) show the effects of an acidic function on the absorption of solutions of cobalt (II) and chromium (III) complexes, respectively. The absorbance of both complex solutions eventually peaked at their respective acidic functions (10,8)for cobalt (II) and chromium (III)respectively. These numbers support this pattern of behavior. The higher sensitivity of the reagent at this pH value may account for the greater absorbance of cobalt (II) and chromium (III) complexes under these circumstances. However, the absorbance of the two complexes drops as the acidic function rises. Both the precipitation of the two ions and the production of unstable complex ions contribute to this tendency.

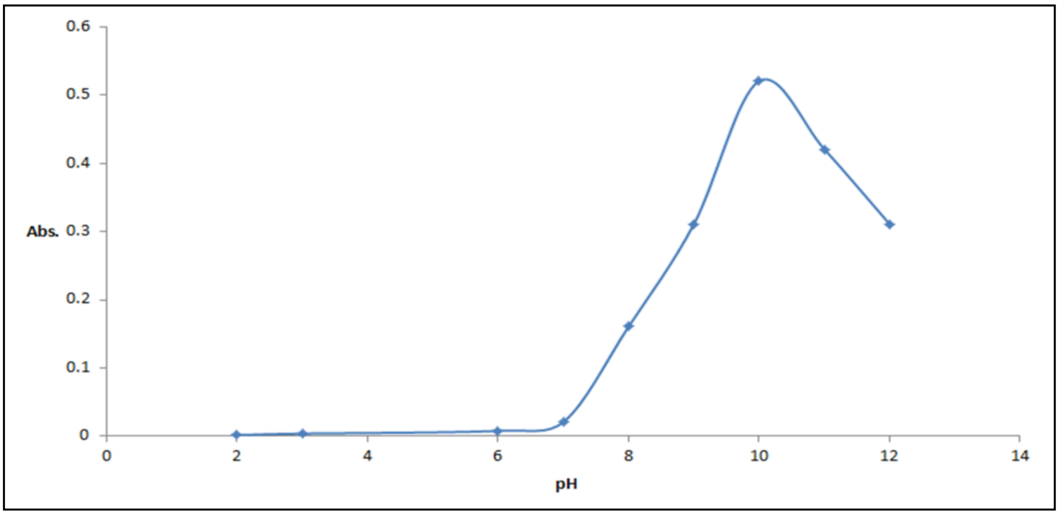


Figure (3-10) :Effect of pH on absorption of Co(II) complex

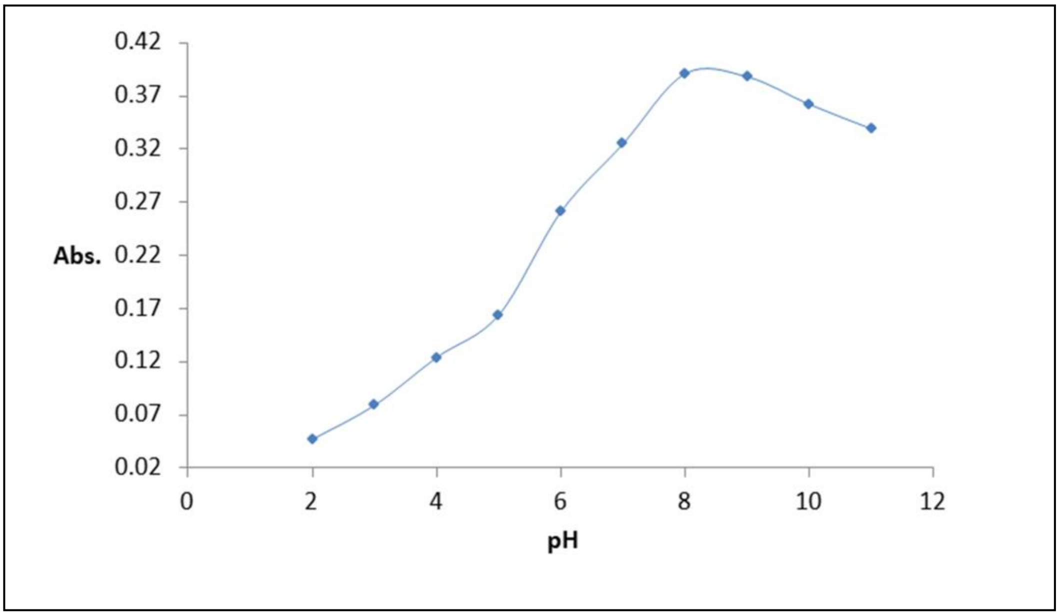


Figure (3-11) :Effect of pH on absorption of Cr(III)complex

(3-7-3) The effect of Time on Complexes Stability

Absorption values of cobalt(II) and chromium(III) complexes are measured using the reagent (BMPQYE1). Cobalt (II) and chromium (III)complexes are very stable under ideal circumstances, as demonstrated in tables (3-7), (3-8).The results of this research make the reagent a more precise tool for determining ionic quantities of Co(II) and Cr(III).

Table (3-7): Effect of time on Co (II)-(BMPQYE1)

Time (min.)	1	10	20	30	60	120	180	240	1440
Abs.	0.523	0.522	0.522	0.518	0.516	0.512	0.487	0.431	0.422

Table (3-8): Effect of time on Cr (III)-(BMPQYE1)

Time (min.)	1	10	20	30	60	120	180	240	1440
Abs.	0.391	0.388	0.387	0.385	0.381	0.279	0.276	0.274	0.270

(3-7-4) Effect of Temperature

Tables (3-9) and (3-10) show the temperature effects on cobalt (II) complexes and chromium (III) complexes, respectively. The cobalt (II) complex has been shown to be effective in the temp.(10-30)and chromium (III) complex between (10-30) degrees Celsius. At higher temperatures, dissolution or poor stability causes the absorption findings to degrade.

Table (3-9) : Effect of Temperature on Co(II)-(BMPQYE1)

Temperature^{o}C	10	20	30	40	50	60
Abs.	0.510	0.523	0.521	0.480	0.427	0.373

Table (3-10) : Effect of Temperature on Cr(III)-(BMPQYE1)

Temperature^{o}C	10	20	30	40	50	60
Abs.	0.364	0.387	0.388	0.383	0.294	0.275

(3-7-5) Effect of Sequence of addition

Multiple complex additions' influence on complex uptake was investigated. Tables (3-11) and (3-12) show four addition arrangements for the complex.both Cobalt (II) and Chromium(III) complexes had an impact on the rate of absorption.

Table (3-11): Effect of Sequence on Co(II)-(BMPQYE1)

Sequence of number	Sequence of addition	Abs. of Co complex
1	M+L+ pH	0.522
2	L+ M+pH	0. 516
3	M+ pH +L	0.502
4	L+ pH + M	0.462

Table (3-12) : Effect of Sequence on Cr(III)-(BMPQYE1)

Sequence of number	Sequence of addition	Abs. of Cr complex
1	M+L+ pH	0.391
2	L+ M+pH	0.375
3	M+ pH +L	0.322
4	L+ pH + M	0.310

pH = hydrogen ion functions M = metal, L = ligand.

Tables (3-11) and (3-12) show that the best addition sequence for both Cobalt(II) and Chromium(III) complexes occurs during the initial addition, whereas absorption decreases with successive additions. Possible explanations include the reaction of acid and basic ions with the metal.

(3-7-6) Effect of Ionic Strength

In order to study the impact of ionic strength on the uptake of cobalt (II) and chromium (III) complexes, we added 1mL of solutions containing sodium nitrate and sodium sulfate salts at concentrations ranging from (0.0005-0.5 M) for each salt. Cobalt(II) and chromium(III) complex uptake as a function of ionic strength is shown in tables (3-13) and (3-14), respectively.

Table (3- 13): Effect of Ionic Strength on Co(II)-(BMPQYE1)

	Conc.(M) of add salt	Abs.	Added salt	Conc.(M)of add salt	Added salt
Na_2SO_4	0.5	0.441	$NaNO_3$	0.5	0.398
	0.05	0.495		0.05	0.457
	0.005	0.499		0.005	0.511
	0.0005	0.501		0.0005	0.514
Abs. of Co(II)-(BMPQYE1) without adding = 0.522					

Table (3-14): Effect of Ionic Strength on Cr(III)-(BMPQYE1)

Adding salt	Conc.(M) of add salt	Abs.	Added salt	Conc.(M)of add salt	Added salt
Na_2SO_4	0.5	0.284	$NaNO_3$	0.5	0.281
	0.05	0.326		0.05	0.319
	0.005	0.375		0.005	0.358
	0.0005	0.384		0.0005	0.376
Abs. of Cr(III)-(BMPQYE1) without adding = 0.391					

Cobalt (II) and chromium (III) compounds was observed that all of the concentrations specified in the tables have no significant effect on the absorption value , and we infer that the ions have no effect on the solubility in the presence of ionic salts, as demonstrated in tables (3-13) and (3-14).

(3-7-7) Effect of buffer solution

The effect of Buffer's solution on the two complexes was demonstrated using previously prepared solutions of(4-10) as shown in the two tables(3-15), (3-16).

Table (3-15) : Effect of buffer solution on Co(II)-(BMPQYE1)

Buffer solution	4.0	6	7	8	9	10
Abs.	0.006	0.008	0.021	0.245	0.362	0.373

Table (3-16) : Effect of buffer solution on Cr(III)-(BMPQYE1)

Buffer solution	4.0	6	7	8	9	10
Abs.	0.001	0.004	0.011	0.135	0.124	0.136

Through the results shown in Tables (3-15), (3-16), it was found that the Buffer solution reduces the absorption value of both the cobalt complex and the chromium complex. Therefore, pH control was chosen using the base solution of sodium hydroxide and hydrochloric acid.

(3-8) Calibration curve of cobalt(II) and chromium(III) complexes

Cobalt (II) and Chromium (III) ion calibration curves were developed at settings we determined to be best. Both Figure 1 and figure 2 show the research's final xresults. (3-12), (3-13). Specifically addressing the Chromium(III) and Cobalt(II) ions.

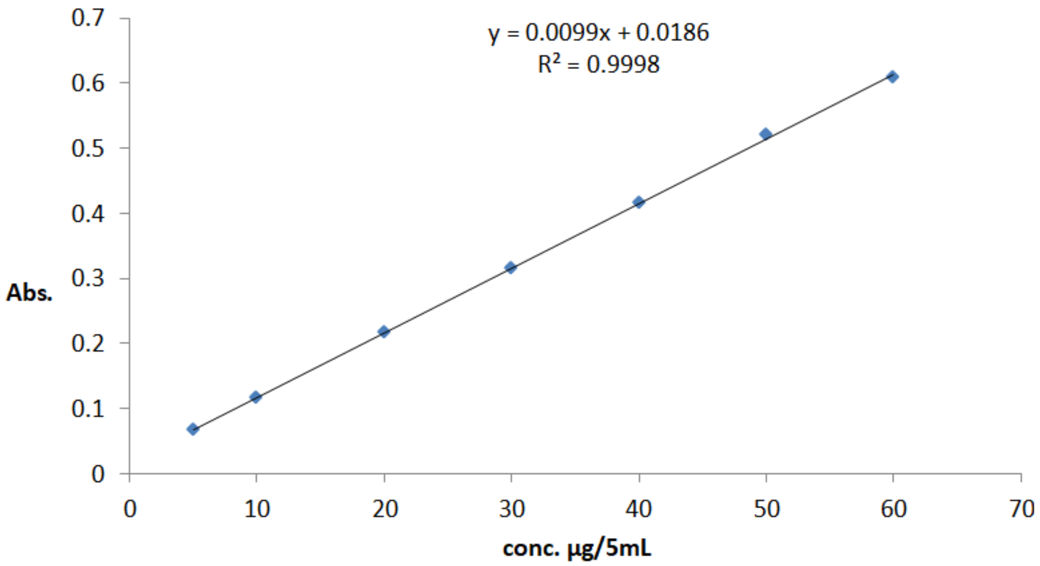


Figure (3-12): Calibration curve of Cobalt (II) complex.

The Lambert-Beer rule is demonstrated to hold true for cobalt (II) ion concentrations in the range of (5µg/mL–60µg/mL) in figure (3-12). In addition to determining that absorbance is proportional to metal concentration, we determined that the molar absorptivity of cobalt (II) complex is (0.0217L mol^{-1} cm^{-1}).

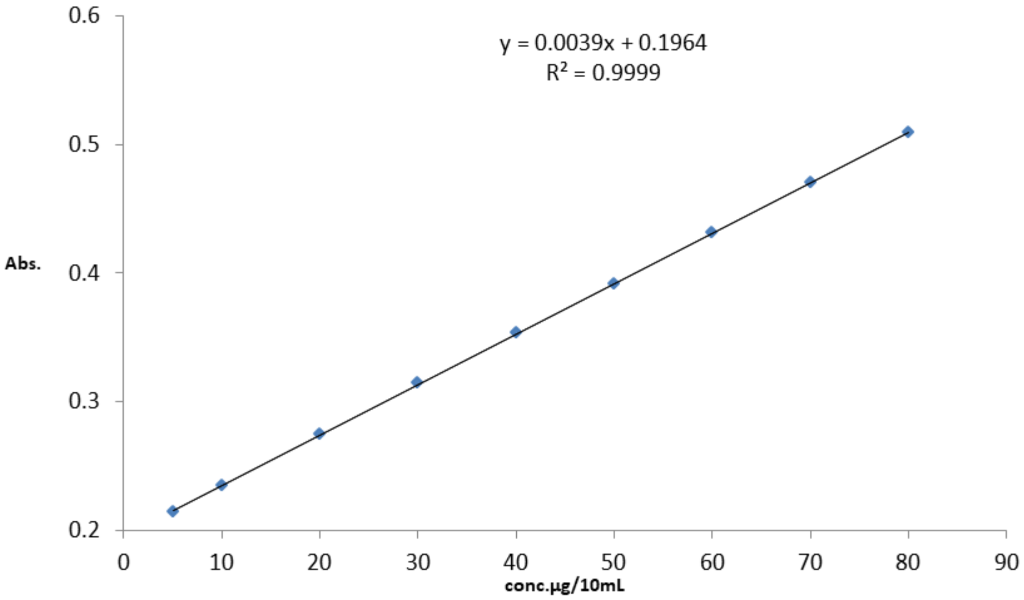


Figure (3-13): Calibration curve of Chromium(III) complex.

The chromium (III) (3-13) calibration curve for spectrophotometric determination is somewhat complicated.Chromium (III) ion has a molar absorptivity of 0.1971 L mol^{-1} cm^{-1}, as shown in Figure 3-13, and the absorbance of chromium complex is a linear function of metal concentration.

$$A = \varepsilon\, b\, C \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots..\ldots (3-1)$$

$$slope\ = \frac{\varepsilon}{At.Wtx1000} \ldots\ldots\ldots\ldots\ldots..\ldots.. \quad (3-2)$$

$$S = \frac{10^{-3}}{a} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.\ldots\ldots\ldots (3-3)$$

ε = Molar absorptivity

S = Sandal sensitivity

a= Specific absorption coefficient

Table (3-17): Data collected for Cobalt (II)and Chromium (III)analysis

Analytical Data	**Value of Cobalt**	**Value of Chromium**
Molar Absorptivity (L.mol^{-1}$\cdot$cm^{-1})	2.9x10^{3}	1.1x10^{4}
Slope	0.0099	0.0039
Linear equation	Y=0.0099X	Y=0.0039X
Linear range(µg/mL)	(1-12)	(1-8)
Sandal sensitivity(µg/cm^{2})	0.102	0.25
Detection limit (µg/5mL)	0.449	0.769
Limit of quantification (µg/mL)	1.481	2.537
Linearity coefficient (R^{2})	0.9998	0.9999
Correlation coefficient (r)	0.9998	0.9999
λ_{max}	589nm	560 nm
Color of product	Light reddish-brown	reddish-brown

Analytical data for reagent-based quantification of chromium ions (BMPQYE1) are provided in table (3-17) displays the results of analyses performed on chromium (III).Because the reagent (BMPQYE1) is complexed with cobalt (II) and chromium (III) ions, as illustrated in tables (3-17), It is simpler to determine the low quantities of these ions in a sample. One possible explanation for the negative deviation at high concentrations is that the complex molecules are interacting with the solvent or aggregating with other molecules.

(3-9) Stoichiometry of the complexes

Composition was determined by using the mole ratio technique and Job's method to complexes of cobalt (II) and chromium (III) under ideal circumstances.

(3-9-1) Job's Method. [99]

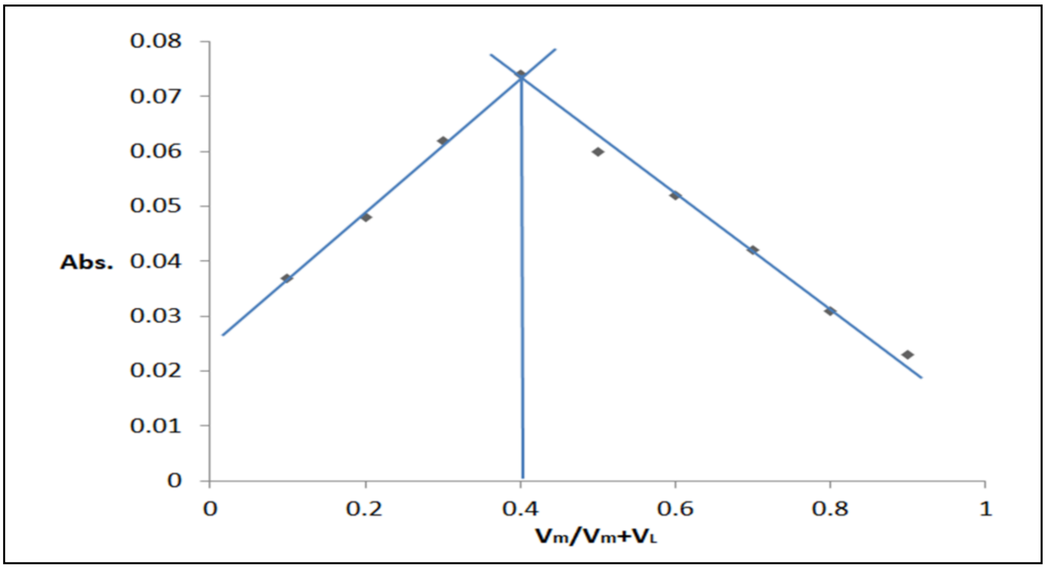


Figure (3-14) : Job`s method of cobalt (II) complex

Figure (3-14) shows that the bonding in the cobalt (II) complex was in the ratio (M:L) (1:2) where the complex consists one mole of the ion to two moles of the reagent.

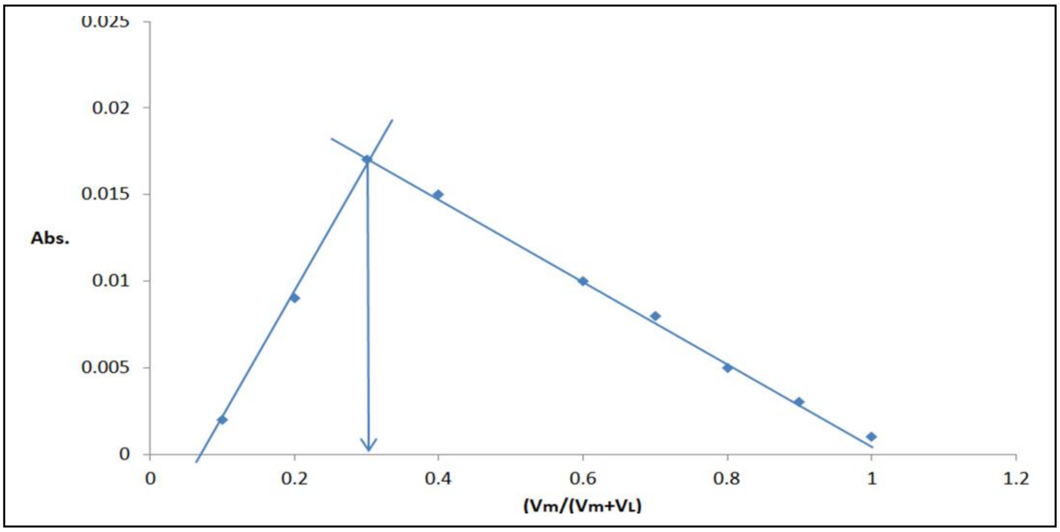


Figure (3-15) : Job`s method of chromium (III) complex

Figure (3-15) shows that the bonding in the chromium (III) complex was in the ratio (M:L) (1:3) where the complex consists of one mole of the ion to three moles of the reagent.

(3-9-2) Mole Ratio Method. [100]

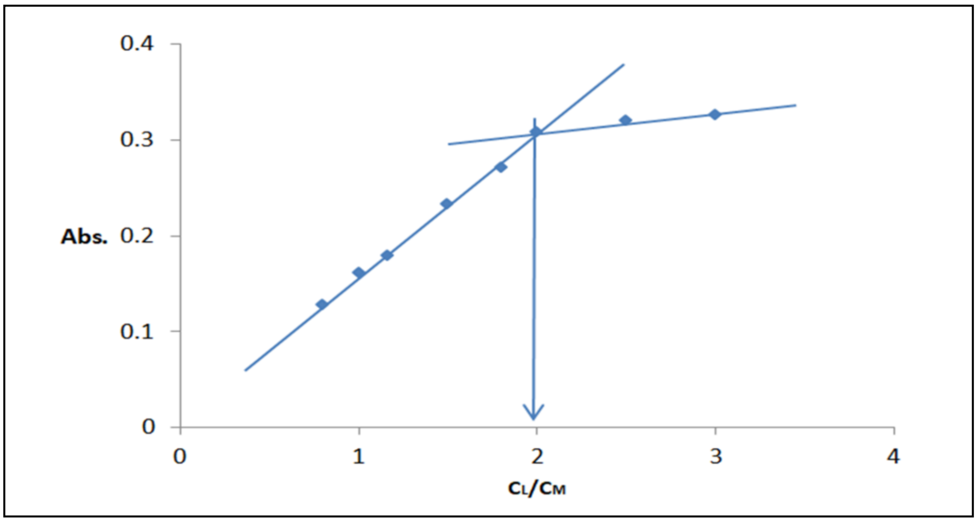


Figure (3-16): Method of Mole Ratio Cobalt (II) complex

Figure (3-16) shows that the bonding in the cobalt (II) complex was in the ratio (M:L) (1:2) where the complex consists of one mole of the ion to two moles of the reagent.

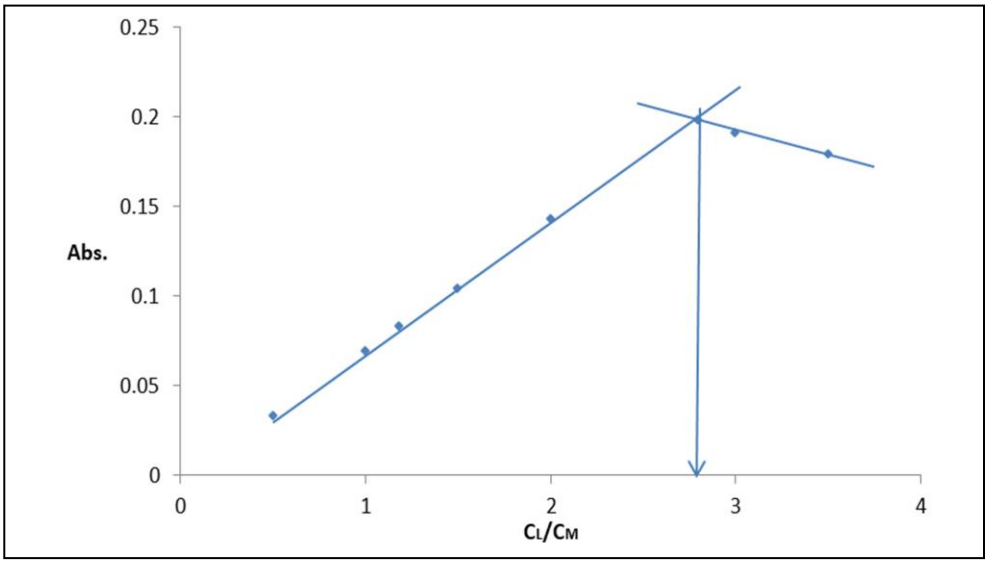


Figure (3-17):Method of Mole Ratio Chromium (III) complex

Figure (3-17) shows that the bonding in the chromium (III) complex was in the ratio (M:L) (1:3) where the complex consists of one mole of the ion to three moles of the reagent.

(3-10) Calculation the stability constant of complexes

Using the molar ratios of the two complexes, which are discussed in detail in paragraphs (3-9-2), we were able to calculate the stability constants of the cobalt (II) complex and the chromium (III) complex, respectively. These equations were used to determine the fixed parameters we discussed earlier. [102]

$$M^{+n} + nL \leftrightarrow MLn \quad \ldots\ldots\ldots\ldots\ldots(3\text{-}4)$$

$$\alpha + nc\alpha \leftrightarrow (1\text{-}\alpha)c$$

$$K=\frac{[MLn]}{[M^{+n}][L]} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3\text{-}5)$$

$$K=\frac{(1-\alpha)c}{\alpha c(n\alpha c)^n} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3\text{-}6)$$

$$K=\frac{1-\alpha}{n^n\alpha^{n+1}c^n} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3\text{-}7)$$

$$\alpha=\frac{Am-As}{Am} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3\text{-}8)$$

Where (As) is the absorption of the complex at the equivalence point, (Am) is maximum absorption.

(α): degree of dissociation

(K): Stability Constant

M^{n+}: Metal

L : ligand

C : molar concentration

n : no. of mole

The calculated results are displayed in the table below (3-16).

Table (3-18): The value of stability constants for cobalt (II) and chromium (III) complexes

Complex	(Am) Value	(As) Value	(α)	(K)
$[Co(BPQYE1)_2]$	0.130	0.058	0.553	3.749×10^{8}
$[Cr (BPQYE1)_3]$	0.312	0.280	0.103	3.332×10^{10}

Because of the great stability of the produced complexes, the reagent (BPQYE1) may be employed to analyze spectra of cobalt (II) and chromium (III) ions, as shown in Table (3-18).

(3-11) Calculation of Thermodynamic functions, stability constant, and dissociation degree for the two complexes.

(3-11-1) The impact of temperature on the degree of dissociation and the stability constant for the two complexes

Tables (3-19) and (3-20) show the results of the effect of temperature on the degree of dissociation and the stability constant for cobalt and chromium complexes.

Table (3-19) The impact of temperature difference on the values of the degree of stability and stability constant of the Co (II) complex

T (^{0}C)	T (K)	α	K×10^9
10	283.15	0.150	0.551
15	288.15	0.146	0.600
20	293.15	0.131	0.845
25	298.15	0.122	1.058
30	303.15	0.114	1.308

Table (3-20): The impact of temperature difference on the values of the degree of stability and stability constant of the Cr (III)complex.

T (^{0}C)	T (K)	α	K×10^{10}
10	283.15	0.118	3.242
15	288.15	0.119	3.132
20	293.15	0.120	3.025
25	298.15	0.121	2.923
30	303.15	0.131	2.641

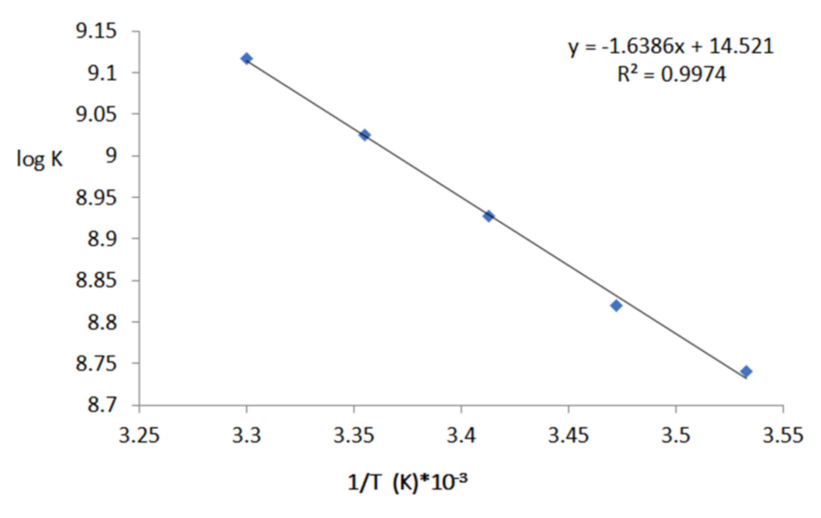
The data in tables (3-19) and (3-20) show that the complexes were very little affected by the temperature. The spectrophotometric analytical capabilities of the reagent (BMPQYE1) is enhanced by the extraordinary stability of the cobalt (II) and chromium (III) complexes. [103]

(3-11-2) Calculation of the thermodynamic functions of the two complexes

Thermodynamic functions as a function of temperature for the cobalt (II) and chromium (III) complexes were determined using the data reported in the preceding section. The results of using the thermodynamic functions ΔG, ΔH, and ΔS, are shown in Table (3-21) and Figure (3-18) for the cobalt (II) complex, and in table (3-22) and figure (3-19) for the chromium (III) complex.

Table (3-21): The impact of temperature on thermodynamic function for cobalt (II) complex

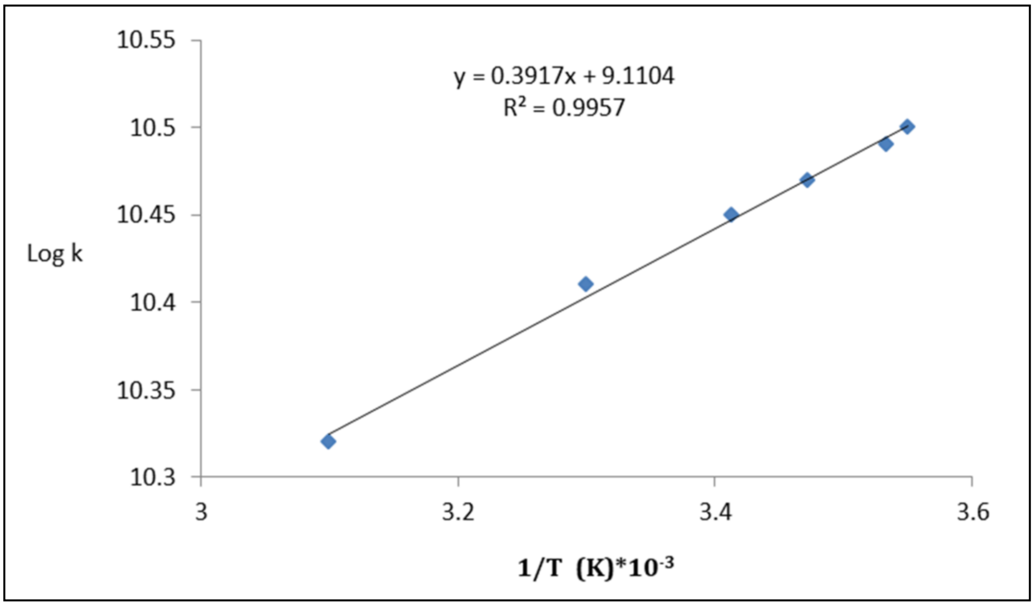
T(K)	1/T×10^{-3}(K^{-1})	log K	ΔH	ΔG (K.J/mole)	ΔS (K.J/mole .K)
283.15	3.533	8.741	31.364	-47.381	0.27810
288.15	3.472	8.778		-48.424	0.27689
293.15	3.413	8.927		-50.099	0.27788
298.15	3.55	9.024		-51.509	0.27795
303.15	3.3	9.116		-52.908	0.27798



Figure(3-18): Relationship between Log K and 1/T values of cobalt(II) complex

Table (3-22): The impact of temperature on thermodynamic function for chromium (III) complex

T(K)	1/T×10^{-3}(K^{-1})	log K	ΔH	ΔG (K.J/mole)	ΔS (K.J/mole .K)
283.15	3.532	10.49	-7.499	-56.974	0.1747
288.15	3.470	10.47		-57.897	0.1749
293.15	3.411	10.45		-58.817	0.1750
298.15	3.354	10.5		-59.735	0.1752
303.15	3.298	10.41		-60.481	0.1747



Figure(3-19): Relationship between Log K and 1/T values of chromium (III) complex

This is evident from both table(3-21) and figure (3-18). An endothermic reaction is indicated by a ΔH value that is positive for the cobalt (II) complex. The beneficial impact of the cobalt ion's hydrolysis shells on temperature. The negative ΔH values for the chromium (III)complex are shown in both table (3-22) and figure (3-25). According to the data, the process is exothermic (3-19), indicating that heat is released throughout the reaction, which strengthens the complex. Since more automatic reactions demand more energy, the complexes generated by these interactions are more stable when ΔG is negative. [104-106]

(3-12) The effect of anionc and cationc ions interplay on cobalt (II) and chromium (III) complexes

(3-12-1) Determination cobalt (II) ion with some interference anions and cations ions

The results of the study into the effect of ion-ion interactions on the absorbance of the Cobalt(II) complex are shown in tables (3-23) and (3-24)

Table (3-23) Effect of cations interference with Cobalt (II) complex.

Foreign/ions	Cations formula structure	(50µg/5mL) Abs. after addition of Cations	E%	(100µg/5mL Abs. after addition of Cations	E%
Cd^{2+}	$Cd(NO_3)_2.4H_2O$	0.316	39.34	0.253	51.43
Ni^{2+}	$Ni(NO_3)_2.6H_2O$	0.388	25.52	0.228	56.23
Ag^{1+}	$Ag(NO_3)$	0.369	29.17	0.236	54.70
Fe^{3+}	$Fe(NO_3)_3.9H_2O$	0.322	38.19	0.499	4.22
Hg^{2+}	$Hg(NO_3)_2$	0.362	30.51	0.288	44.72
Pb^{2+}	$Pb(NO_3)_2$	0.365	29.94	0.283	45.68
Mg^{2+}	$Mg(N0_3)_2.4H_2O$	0.522	-0.19	0.257	50.67
Ba^{2+}	$Ba(NO_3)_2$	0.317	39.15	0.275	47.21
Cu^{2+}	$Cu(NO_3)_2.3H_2O$	0.357	31.47	0.293	43.76

Table (3-24): Effect of anions interference with Cobalt (II)complex.

Foreign Ions	Formula structure of Anions	(50µg/5mL) Absorption after addition of Anions	E%	(100µg/5mL) Absorption after addition of Anions	E%
SO_4^{2-}	K_2SO_4	0.356	31.66	0.353	32.24
Br^{1-}	KBr	0.367	29.55	0.320	38.57
SCN^{1-}	KSCN	0.312	40.11	0.288	44.72
$IO3^{1-}$	KIO_3	0.275	47.21	0.233	55.27
CrO_7^{2-}	K_2CrO_7	0.421	19.19	0.314	39.73
CO_3^{2-}	K_2CO_3	0.398	23.60	0.292	43.95
CN^{1-}	KCN	0.438	15.93	0.297	42.99

Depending on the concentration of the added ions, it was observed that the cations either reduce or increase the absorption of the compounds because the ions can form a complex with the reagent and this increases the sensitivity of the method for the determination of chromium. In table (3-23), (3-24) the results show that most of the ions affect the absorption of the Cobalt (II) ion with the reagent. [107,108]

(3-12-2) Determination Chromium (III) ion with some interference anions and cations ions

Chromium (III) ion calculations with anion and cation interference studied. Chromium (III) complex absorbance was measured in the presence of various ions. The information from these studies is shown in table (3-25) and (3-26).

Table (3-25) : Effect of cations interference with Chromium (III) complex.

Foreign/ ions	Cations formula structure	(50µg/10mL) Abs. after addition of Cations	E%	(100µg/10mL) Abs. after addition of Cations	Error%
Cd^{2+}	$Cd(NO_3)_2.4H_2O$	0.235	39.89	0.146	62.65
Ni^{2+}	$Ni(NO_3)_2.6H_2O$	0.207	47.05	0.188	51.91
Ba^{2+}	$Ba(NO_3)_2$	0.287	26.59	0.133	65.98
Fe^{3+}	$Fe(NO_3)_3.9H_2O$	0.153	60.86	0.124	68.28
Hg^{2+}	$Hg(NO_3)_2.H_2O$	0.332	30.25	0.332	30.25
Pb^{2+}	$Pb(NO_3)_2$	0.235	39.89	0.214	45.26
Mg^{2+}	$Mg(N0_3)_2.6\ H_2O$	0.252	35.54	0.197	49.61
Ag^+	$AgNO_3$	0.308	21.22	0.156	60.10
Cu^{2+}	$Cu(NO_3)_2.3H_2O$	0.251	35.80	0.132	66.24

Table (3-26): Effect of anions interference with Chromium (III) complex.

Foreign Ions	Formula structure of Anions	(50µg/10mL) Absorption after addition of Anions	E%	(100µg/10mL) Absorption afteraddition of Anions	E%
SO_4^{2-}	K_2SO_4	0.188	52.16	0.137	65.13
Br^{1-}	KBr	0.224	43.01	0.148	62.34
SCN^{1-}	KSCN	0.346	11.95	0.327	16.79
Cl^{1-}	KCL	0.214	45.54	0.141	64.12
CrO_7^{2-}	K_2CrO_7	0.267	32.06	0.179	54.45
CO_3^{2-}	K_2CO_3	0.385	2.03	0.381	3.05
CN^{1-}	KCN	0.293	25.44	0.253	35.62

Depending on the concentration of the added ions, it was observed that the cations either reduce or increase the absorption of the compounds because the ions can form a complex with the reagent and this increases the sensitivity of the method for the determination of chromium. In Table (3-25), (3-26) the results show that most of the ions affect the absorption of the Chromium (III) ion with the reagent.

(3-13) Effect of Masking agents

(3-13-1) Limitation the optimal masking agent for the determination of the cobalt (II) and chromium (III) complexes

Since most cations overlap with the Cobalt (II) and Chromium (III) complexes created, therefore it is required to design a technique to reduce this impact. Therefore, masking agents were selected (7) to observe the effect between these agents and the reagent as it interacts with the interfering ions. This issue was not seen in this research after masking it with 1 mL of each agent, as indicated in tables (3-27) and (3-28).

Table (3-27) :The effect of masking agent on Cobalt(II) complex absorption

Masking agent (0.1M)	Abs of Co(II)complex
Without Masking agent	0.522
Thiourea	0.563
Ascorbic acid	0.351
Na_2EDTA	0.336
Citric Acid	0.457
KCl	0.484
$Na_2HPO_4.12H_2O$	0.430
Formaldehyde	0.437

Thiourea has a low influence on on synthesized complex absorption. table(3-27) suggests that this may be used as a masking agent. However, due to the other rest dissociation with Cobalt(II) ion, they can’t be used as a masking agent

Table (3-28):The effect of masking agent on chromium(III) complex absorption

Masking agent (0.1M)	Abs of Cr(III)complex
Without Masking agent	0.392
Thiourea	0.219
Ascorbic acid	0.386
Na_2EDTA	0.267
Citric Acid	0.260
KCl	0.119
$Na_2HPO_4.12H_2O$	0.241
Formaldehyde	0.258

The absorption efficiency of the complex is little impacted by the presence of ascorbic acid. ccording table (3-28) that this material might be used in a masking agent. However, due to the other rest dissociation with chromium (III) ion, they can't be used as a masking agent.

(3-13-2) Use of a better masking agent to determine the cobalt (II) and chromium (III) ions in the presence of cations interference

1- Cobalt(II) complex.

Table (3-29) shows that Thiourea, the best masking agent, was employed to estimate the Cobalt (II) complex in the presence of competing cationic ions.

Table (3-29): The effect of masking agent in the presence of cations on Cobalt(II) complex absorption.

Foreign ions	Absorption after addition cation (50µg/5mL) and addition masking agent(0.1M)	Error (E%)
Cd^{2+}	0.465	10.919
Ni^{2+}	0.477	8.620
Ag^{+}	0.450	13.793
Fe^{3+}	0.484	7.279
Hg^{2+}	0.472	9.578
Pb^{2+}	0.475	9.003
Mg^{2+}	0.523	-0.191
Ba^{2+}	0.491	5.938
Cu^{2+}	0.516	1.149

The absorption values of the Cobalt (II) complex in the presence of interfering cations when a better masking agent is added show in Table (3-29),as well as absorbance values close to those before the interference.

2- Chromium(III) complex

Ascorbic acid, the best masking agent given in table (3-30), was employed to estimate the Chromium (III) complex in the presence of competing cationic ions.

Table (3-30): The effect of masking agent in the presence of cations on chromium (III) complex absorption

Foreign ions	Absorption after addition cation (50µg/10mL) and addition masking agent(0.1M)	(E%)
Cd^{2+}	0.470	1.26
Ni^{2+}	0.477	-0.21
Ba^{2+}	0.440	7.56
Fe^{3+}	0.473	0.63
Hg^{2+}	0.480	-0.840
Pb^{2+}	0.475	0.210
Mg^{2+}	0.462	2.940
Ag^{+}	0.491	-3.150
Cu^{2+}	0.453	4.830

The absorption values of the chromium (III) complex in the presence of interfering cations when a better masking agent is added show in Table (3-30) , and absorbance values close to those before the interference.

(3-14) Precision and accuracy of the suggested method

Accuracy may be quantified using the relative standard deviation (RSD%), whereas fidelity can be assessed using the relative recovery of the cobalt (II) and chromium (III) complexes in question, as determined by four measurements at varied concentrations.table (3-31) and (3-32) display the outcomes for the cobalt (II) and chromium (III) complexes, respectively.

Table (3-31): The Precision and accuracy of the Co(II) complex

Conc. of Co^{+2} present[M]	Conc. of Co^{+2} found[M]	RSD%	Recovery%	Error%
8.474×10^{-5}	8.578×10^{-5}	0.598	101.19	-1.48
9.337×10^{-5}	9.078×10^{-5}	1.135	97.23	2.77
2.454×10^{-6}	2.417×10^{-6}	1.559	98.49	1.51

Table (3-32): The Precision and accuracy of the Cr(III) complex

Conc. of Cr^{+3} present[M]	Conc. of Cr^{+3} found[M]	RSD%	Recovery%	Error%
1.973×10^{-7}	1.971×10^{-7}	0.661	99.98	0.10
2.843×10^{-5}	2.801×10^{-5}	0.759	98.52	1.47
5.265×10^{-5}	5.326×10^{-5}	0.887	101.16	-1.16

Based on these findings, it is clear that the analytical method used to determine BMPQYE1 concentrations has high precision and accuracy.

(3-15) calculatation of sensitivityof the spectral method for determination the cobalt (II) and chromium (III) ions

Cobalt (II) and Chromium (III) ion detection in this approach is extremely sensitive, as shown by the expression limit of detection. This method effectively determines cobalt (II) ion and has a high sensitivity, as shown by the following equation for calculating sensitivity (D.L): the lowest concentration of cobalt ion could be estimated to be (3.475×10^{-6}M), while the lowest concentration of chromium (III) was (2.194×10^{-6}M).

$$D.L. = \frac{3x\ S.D}{\bar{x}} \text{..........(3-9)}$$

SD = standard deviation

$\bar{x}$ = Average

(3-16) Preparation of solid complexes

Under optimum circumstances for reagent volume, temperature, molar ratios, acidity function, etc., two compounds were produced by reacting ethanolic solutions of the reagent BMPQYE1 with aqueous solutions of cobalt (Co^{+2}) and chromium (Cr^{+3}). After the solutions were combined and allowed to cool, a precipitate was seen to develop. The physical properties of the two solid complexes were examined after the solutions were completely precipitated, filtered, and recrystallized.

(3-17) Study of FT-IR Spectra of the cobalt (II) and chromium (III) complexes

Infrared spectroscopy is crucial for identifying which active groups are likely to interact with metal ions. Distinct quantitative and formal distinctions. When comparing the spectrum of the reagent by itself to the spectrum of the complexes, one can see the absorption beams move from higher to lower frequencies, indicating that a bond is being formed between the metal ion and the donor atoms in the reagent. Below is a table including the FT-IR spectrum analysis findings.

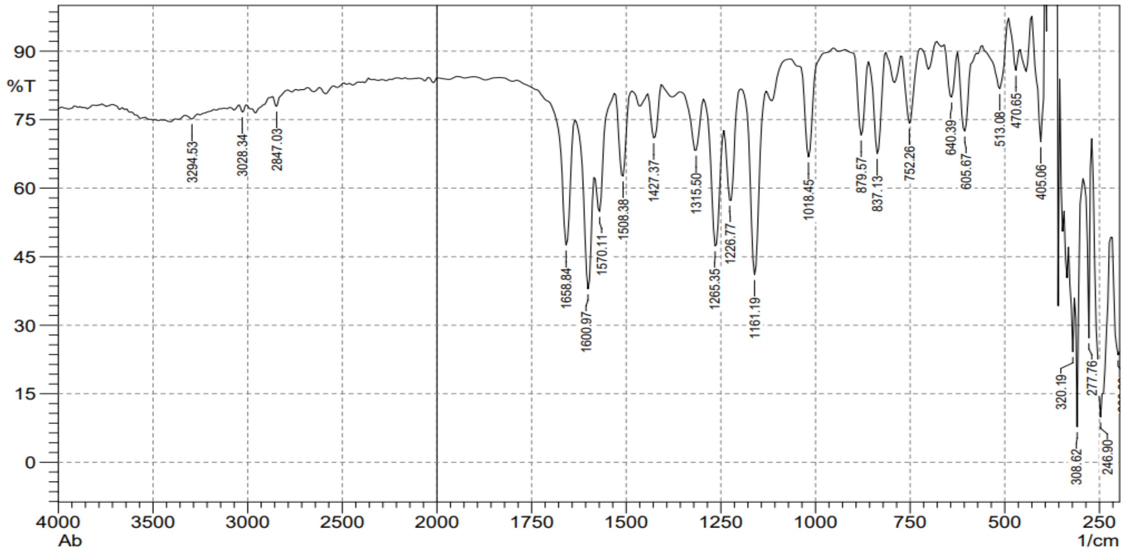


Figure (3-20) FT-IR spectrum of cobalt(II) complex

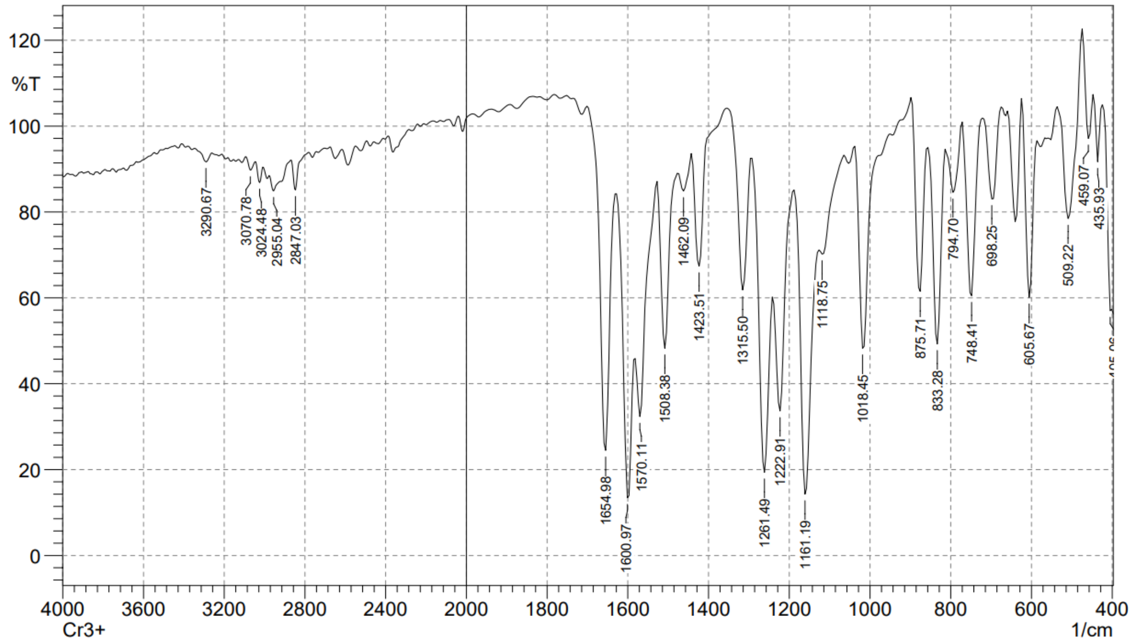


Figure (3-21) FT-IR spectrum of Chromium (III) complex .

Table (3-33) Typical FTIR absorption frequencies of the reagent and complexes (cm^{-1}). [109-111]

Bond	Reagent	cobalt(II) complex	Chromium (III) complex
St.(N-H)	3311.78	3294.53	3290.67
St(C=O)	1614.42	1658.84	1654.98
St.(C-H)Aromatic	3064.89	3028.34	3024.48
St.(C=N)	1504.48	1570.11	1600.97
St.(C=C)	1614.42	1508.38	1508
St.(C-O)	1161.15	1265.35	1161.19

(3-18) Determination of the melting point of the complexes

Because they shed light on the complex's history, melting temperatures were investigated in this study. The reagent has a melting point of about (127-130), while the Cobalt (II) and Chromium (III) compounds reach melting temperatures of (153-256) and (142-145), respectively. Dissimilarities like this serve as examples of how complexes formation. [112]

(3-19) Measurement of molar Conductivity of the Cobalt (II) and Chromium (III) complexes

The molar conductivity of each of the reagent and cobalt and chromium compounds was determined at a concentration of 1×10^{-3}M and using ethanol as a solvent at room temperature. It was noted that the cobalt compound is non-ionic and the chromium compound has ionic properties.

Table (3-34) The molar conductivity values of the two solutions of the cobalt (II) and chromium (III) complexes

No	Complex	Λ_m(µS/cm)
1	$[Co(BMPQYE1)_2]$	17.3
2	$[Cr(BMPQYE1)_3]$	298

(3-20) Application

The amount of cobalt ions in hair and pharmaceuticals was measured. Drug samples were tested for their chromium ion concentrations. The statements stated in the examples were verified by the experimental results.table (3-35), (3-36) and (3-37) display the outcomes of the ion analysis for cobalt and chromium, respectively.

Table (3-35): Results of application for cobalt(II) in V.B_{12}.

Sample	Conc. present (µg/mL)	Conc. found (µg/mL)	Recovery %	E%
V.B_{12}	43	42.16	98.05	1.95

V.B_{12}(cyanocobalamine)1000m/4mL

Table (3-36): Results of application for Cobalt(II) in hair sample

Sample	Extracted method	Atomic method	E%
Hair	2.58	2.6	0.7

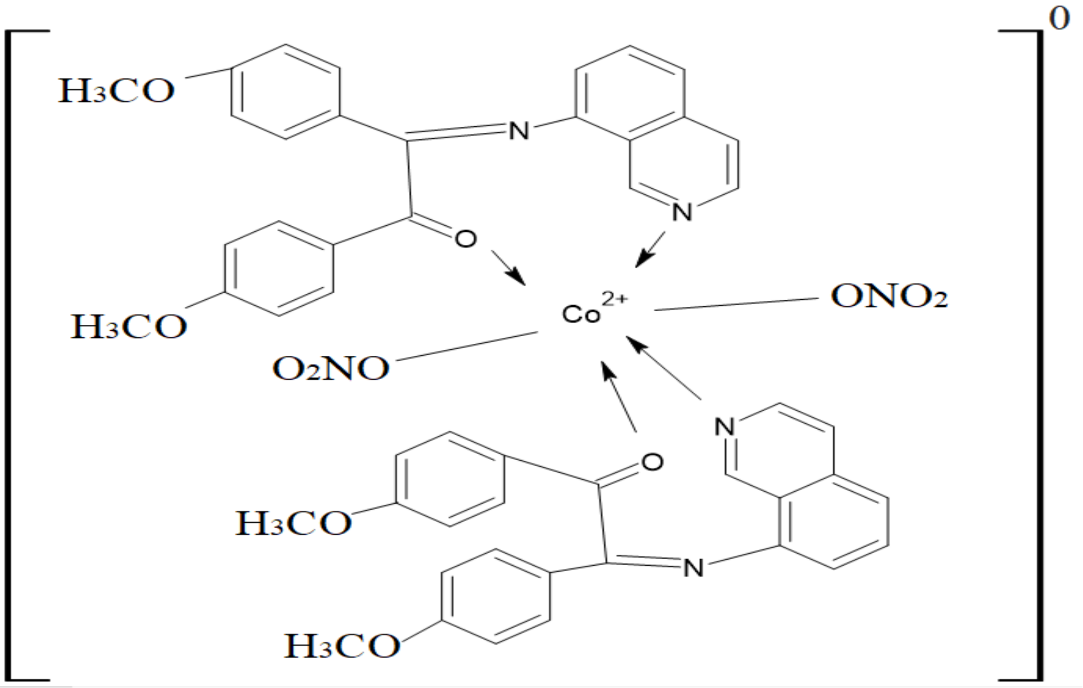
Table (3-37):Results of application for Chromium (III)determination.

Pills	The company	Conc. present (µg/mL)	Conc. found (µg/mL)	Recovery %	E%
AZ-Multiway	GH – Yildiz	25	24.26	97.04	2.96
Maddovit	Maddox Pharma Swiss	35	34.77	99.34	0.66

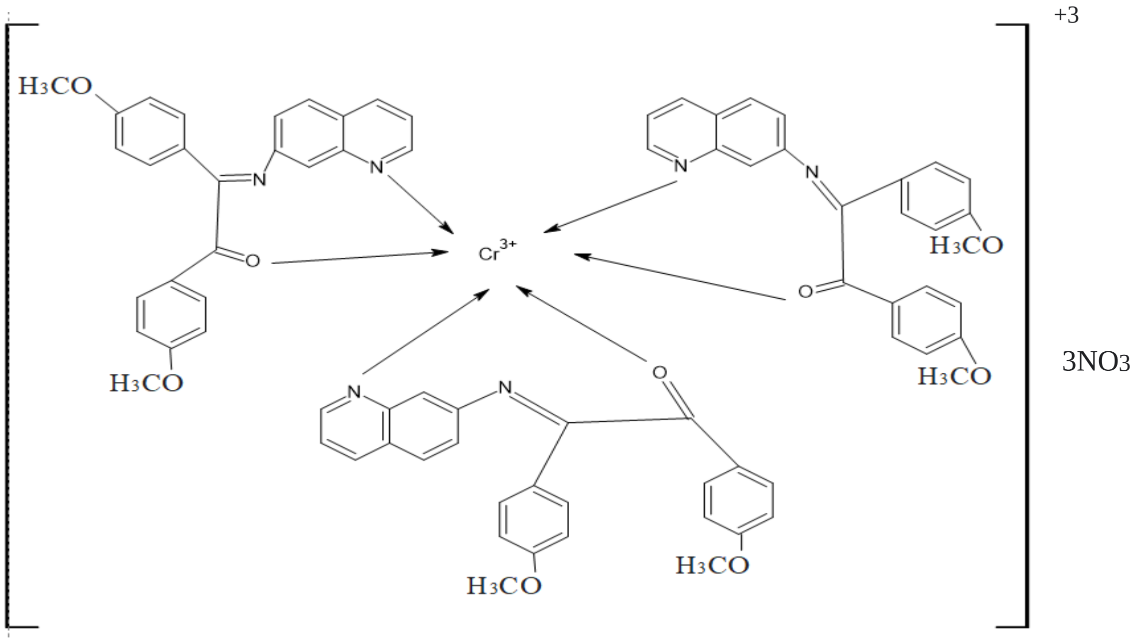
Analytical results that obtained by spectrophotometric method were statistically compared with real sample were in good agreement . The method can be applied for quality control of pharmaceutical dosage forms that containing cobalt(II) and chromium(III) ions.

(3-21) The suggested structure of the Cobalt (II) and Chromium (III) complexes

According to the results of this investigation, the M:L ratios of complexes containing cobalt (II) and chromium (III) are 1:2 and 1:3, respectively. The planned complicated structures are shown graphically in the figure (3-22),(3-23).



Figure(3-22) The suggested structure of the Cobalt (II) complex.



Figure(3-23) The suggested structure of the Chromium (III) complex.

(3-22) Conclusions

1. Using conventional methods Preparation of a highly selective and sensitive reagent(BMPQYE1) from Schiff bases.
2. The microgram amounts of ions Co (II), Cr (III) in The spectrophotometric , which has colored complexes may be calculated with the spectroscopic reagent (BMPQYE1).These complexes have points where they absorb the most.
3. The method for determining cobalt (II) and chromium (III) ions with the prepared reagent is quick and simple as compliance with the Lambert-Beer law gives a wide range of concentrations.
4. The results of determining the equivalence of the two complexes by the Job's method, molar ratios, show that the reagent-to-ion ratio is (1:2) for the cobalt (II) complex and (1:3) for the chromium (III) complex.
5. Thiourea was shown to be an effective masking agent in a study of the effects of interactions (cions and anions) and masking variables on Cobalt (II) estimation. Masking Chromium (III) ions with ascorbic acid has been shown to be successful and has no influence on the estimation process.
6. Since the ΔSand ΔH values for the Cobalt (II) complex with the reagent are all positive, the reaction is endothermic and occurs spontaneously at high temperatures. The negative values of ΔH and ΔG for the Chromium (III) complex with the reagent show that this reaction is exothermic and spontaneous at low temperature.
7. These ions are more stable than those found in earlier studies because they are produced in an aqueous phase.
8. Cobalt (II) complexes were found to be uncharged whereas Chromium (III) complexes were found to be charged based on measurements of their molar conductivity and charge.
9. The sensitivity, selectivity, accuracy, and did not require any prior additions were established by comparing the relative standard deviation and relative percentage error values.
10. The newly discovered reagent (BMPQYE1) might be used in the pharmaceutical industry for the estimation of trace amounts of cobalt (II) and chromium (III) complexes.

(3-23) Recommendations

1. Preparation of a new Schiff bases reagent and formation of complexes with other ions.
2. The ligands are biologically effective, so a search can be made for the biological activity of the ligands.
3. Conducting research to form new complexes using ligands with other ions.
4. The production of novel derivatives of the reagent to improve its selectivity and features, as well as the ability to use the reagent to estimate other components.
5. Using more than one for Schiff bases reagent to determine cobalt and chromium metal ion

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Appendixes

Appendices

1- Statistical treatment.

-Calculation of the Relative standard deviation.

1- Statistical treatment and calculation of %RSD values for cobalt (II) ion.

Table (1) Calculation of the %RSD of the cobalt (II) ion at concentration 8.474x10^{-5}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D = \sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD = \frac{SD}{\bar{x}} \times 100$
0.518	0.5208	-0.0028	3.88×10^{-5}	3.114×10^{-3}	0.598 %
0.526		0.0052			
0.519		-0.0018			
0.520		-0.0008			
0.521		0.0002			

Table (2) Calculation of the %RSD of the cobalt (II) ion at concentration 9.337x10^{-5}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D = \sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD = \frac{SD}{\bar{x}} \times 100$
0.520	0.5252	-0.0052	1.422×10^{-4}	5.962×10^{-3}	1.135 %
0.524		-0.0012			
0.535		0.0098			
0.527		0.0018			
0.529		0.0038			

Table (3) Calculation of the %RSD of the cobalt(II) ion at concentration 2.454×10^{-6}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D=\sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD=\frac{SD}{\bar{x}}\times100$
0.0316	0.03194	-0.0003	9.92×10^{-7}	4.979×10^{-4}	1.559%
0.0314		-0.0005			
0.0318		-0.0001			
0.0323		0.0003			
0.0326		0.0007			

2- Statistical treatment and calculation of %RSD values for chromium (III) ion.

Table (4) Calculation of the %RSD of the chromium (III) ion at concentration 1.973×10^{-7}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D=\sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD=\frac{SD}{\bar{x}}\times100$
0.195	0.1968	-0.0018	6.8×10^{-6}	1.31×10^{-3}	0.661 %
0.198		-0.0012			
0.197		0.0002			
0.196		0.0008			
0.198		0.0012			

Table (5) Calculation of the %RSD of the chromium (III) ion at concentration 2.843×10^{-5}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D=\sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD=\frac{SD}{\bar{x}}\times100$
0.251	0.2532	0.0022	1.48×10^{-5}	1.924×10^{-3}	0.759 %
0.253		0.0002			
0.254		0.0008			
0.252		0.0012			
0.256		0.0028			

Table (6) Calculation of the %RSD of the chromium (III) ion at concentration 5.265×10^{-5}M

Abs (Xi)	**Mean ($\bar{x}$)**	**(xi-$\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$S.D=\sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\%RSD=\frac{SD}{\bar{x}}\times100$
0.301	0.3044	-0.0034	2.92×10^{-5}	2.702×10^{-3}	0.887 %
0.303		-0.0014			
0.304		-0.0004			
0.306		0.0016			
0.308		0.0036			

- Calculation of Detection limit .

1- Statistical treatment and calculation of D.L values for cobalt (II) ion.

Table (7) Calculation of the D.L of the Cobalt (II) ion at concentration 0.4µg/mL.

Abs (Xi)	**Mean ($\bar{x}$)**	**($xi-\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$\text{S.D} = \sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\text{D.L} = \frac{3 \times \text{S.D}}{\text{slop}}$
0.348	0.3858	-0.0018	8.8×10^{-6}	1.483×10^{-3}	0.449
0.386		0.0002			
0.386		0.0002			
0.388		0.0022			
0.385		-0.0008			

2- Statistical treatment and calculation of D.L values for chromium (III) ion.

Table (8) Calculation of the D.L of the Chromium (III) ion at concentration 0.7 µg/mL.

Abs (Xi)	**Mean ($\bar{x}$)**	**($xi-\bar{x}$)**	**$\Sigma(xi-\bar{x})^2$**	$\text{S.D} = \sqrt{\frac{\Sigma(xi-\bar{x})^2}{N-1}}$	$\text{D.L} = \frac{3 \times \text{S.D}}{\text{slop}}$
0.213	0.2152	-0.0022	1.4×10^{-5}	1×10^{-3}	0.769
0.215		-0.0002			
0.214		-0.0012			
0.216		0.0008			
0.218		0.0022			

الخلاصة :

تضمنت الدراسة تحضير وتشخيص ليكاند قواعد شف الجديد وهو

1,2-bis(4-methoxyphenyl-2(quinolone-8-ylimino)ethan-1-one(BMPQYE1)

الذي هو أحد ليكاندات قواعد شف حضر عن طريق تفاعل 8-aminoquinoline مع 4,4'-dimethoxybenzil، وتم تشخيصه باستخدام طرق التحليل الطيفي المعتمدة مثل الأشعة فوق البنفسجية- المرئية وقياس طيف الكتلة FT IR والرنين النووي المغناطيسي للبروتونات و ^{13}C وكذلك دراسة بعض الخصائص الفيزيائية والكيميائية. وتم إجراء دراسه اوليه للكاشف مع (11) ايون من الأيونات الفلزية و دراسة التحليل الطيفي لها وقد تم اختيار ايوني الكوبلت والكروم لتقديرهما باستخدام الكاشف المذاب في الإيثانول حيث اعطا محلول الكاشف اعلى امتصاص باستخدام تقنية UV-vis (λ_{max} =467 nm). تم دراسة معقدات الكوبلت (II) والكروم (III) المتكونة مع الكاشف، وبالاضافه الى ذلك تمت دراسة الضروف الفضلى لتفاعل هذه الأيونات مع الكاشف ، مثل الدالة الحامضية ، وحجم الكاشف ، درجة الحرارة وزمن الاستقرار للمعقدات. تم دراسة منحني المعايرة لكل من هذه الأيونات مع الكاشف، حيث يكون معقد الكوبلت(II) ضمن مدى من التركيز (5µg/5mL-60µg/5mL) و معامل الخطية (R^2=0.9998) وقيمة الامتصاصية المولارية (ε) تساوي (0.0217L.moL^{-1} .cm^{-1}) وحساسية Sandal تساوي (0.102µg/cm^2) ، بينما ايون الكروم (III) يكون ضمن مدى من التركيز (5µg/10mL-80µg/10mL) ومعامل الخطية (R^2=0.9999). قيمة الامتصاصية المولارية(0.1971L.moL^{-1}.cm^{-1}) ، حساسية ساندل تساوي (0.25µg/cm^2)، وتمت دراسة تكافؤية المعقدات المحضرة من خلال إيجاد نسبة أيون الفلز إلى الكاشف (M:L) باستخدام طريقة جوب والنسبة المولية لمعقدات الكوبلت (II) والكروم (III) . كما حسبت ثابت الاستقرار (K_{sta}) للمعقدين وكانت قيمته لمعقد الكوبلت (K_{sta} = 3.749×10^{8}) ولمعقد الكروم (K_{sta} = 3.332×10^{10}). بالإضافة إلى ذلك ، تم حساب الدوال الثرموديناميكية للمعقدات المحضرة (ΔH ، ΔG، ΔS) ، من خلال النتائج تبين أن التفاعل الناتج من تكوين معقد الكوبلت (II) ماص للحرارة ومركب الكروم (III) باعث للحرارة. تم دراسة تأثير وجود الايونات السالبة والموجبة على المعقدات المحضرة ، كما تمت دراسة إضافة عوامل الحجب لتقليل تاثير تداخل الايونات المتداخلة في المحلول ، حيث وجد أن هذه الأيونات تتفاعل بدرجات متفاوتة حسب طبيعة

وتركيز الأيونات المتداخلة . ، تم قياس توصلية المعقدات من خلال تحديد شحنة المعقدات الصلبة المحضرة الذائبة في الايثانول ، حيث اظهرت النتائج ان معقد الكوبلت(II) غير مشحون لكن معقد الكروم (III) مشحون. تم تحديد دقة وتوافقية الطريقة التحليلية المستخدمة باستخدام (5) تراكيز مختلفة لكل أيون ، فتراوحت قيم النسبة المئوية للانحراف المعياري النسبي لأيون الكوبلت (II) بين (0.598% - 1.567%) وتراوح نسبة التوافقية بين (101.19% -98.49%) أما بالنسبة للكروم (III) فقد تراوحت الانحراف المعياري النسبي بين (0.661% - 0.887%) ونسبة التوافقية تتراوح بين (99.98% - 101.16%). تم حساب حد الكشف لأيون الكوبلت وكان (5.098×10^{-5}µg/mL) اما حد الكشف لأيون الكروم فيساوي (1.169×10^{-4}µg/mL)، مما يدل على ان الطريقة الطيفية ذات حساسية عالية باستخدام الكاشف(BMPQYE1). تم دراسة بعض الخصائص الفيزيائية مثل درجة الانصهار والتوصلية المولارية للمعقدين الكوبلت والكروم . تم تطبيق الطريقة على عينات صيدلانيه ووجد ان الطريقة المتبعة في التقدير ذات حساسية ودقة عالية.

جامعة كربلاء
كلية العلوم
قسم الكيمياء

التقدير الطيفي لأيوني Co(II) وCr(III) بأستخدام مشتق قاعدة شف الجديد

رسالة

مقدمة الى مجلس كلية العلوم – جامعة كربلاء كجزء من متطلبات نيل درجة الماجستيرفي علوم الكيمياء

من قبل

عبير جاسم صاحب

بأشراف

الاستاذ الدكتور

علاء فراك حسين

1444 هـ 2023م