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Spectrophotometric Assay of Cobalt(II) in Various Water Samples by using new Synthesized Schiff's base: [1-((Pyrimidin-2-ylimino) methyl) Naphthalen-2-ol] (PYMN)

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Abstract:

A novel, simple, and highly sensitive spectrophotometric method for the quantification of trace amounts of Co(II) in aqueous medium is presented. The method relies on the reaction of cobalt(II) ion with synthesized reagent [1-((pyrimidin-2-ylimino) methyl)1195aphthalene-2-ol] (PYMN) in a basic medium(sodium bicarbonate, 0.1M) in the presence of CPC to form an orange complex with maximum absorbance at 466nm. The proposed method obeyed Beer's law within the concentration range of (0.05-0,65 $\mu g.ml^{-1}$) , with a molar absorptivity began 4.8×10^{4} $L.mol^{-1}.cm^{-1}$, and determination coefficient of 0.9985 . The precision of the proposed method is demonstrated with a relative standard deviation(RSD%) which is blow 1.6% , the stoichiometry of the formed complex confirmed through Job's method, revealing a metal-reagent ratio of 1:2. The impact of several ions on the determination of Co(II) is investigated . The proposed method is successfully applied to determination of Co(II) in synthetic water samples .

Keywords : Novel prepared reagent (PYMN) , Co(II) assay , water samples, spectrometry**.**

التقدير الطيفي الكوبلت الثنائي في نماذج مائية مختلفة باستخدام قاعدة شف [1-((Pyrimidin-2-ylimino) methyl) Naphthalen-2-ol]

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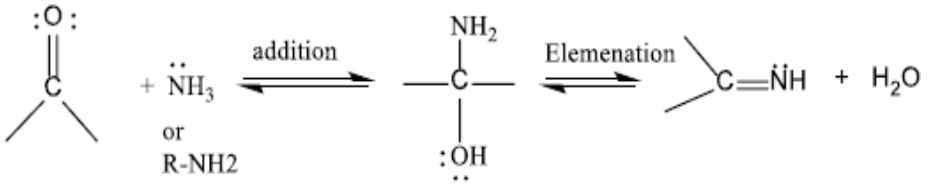
الخلاصة :

تم تطوير طريقة طيفية عالية الحسلسية لتقدير الكوبلت الثنائي في المحاليل المائية باستخدام قاعدة شف جديدة وذلك عن الطريق مفاعلة الكوبلت الثنائي مع الكاشف المحضر: [1- ((pyrimidin-2-ylimino) methyl)1195aphthalene-2-ol] (PYMN) في وسط قاعدي ضعيف (كاربونات الصوديوم 0.1عياري) وبوجود عامل الشد سطحي CPC لتكوين معقد برتقالي اللون له اعلى امتصاصية عند الطول الموجي 466نانوميتر. انطبق قانون بير للطريقة المقترحة بمدى تركيز 0.05-0.65 مايكروغرام . مللتر من الكوبلت الثنائي بمعامل امتصاصية مولاري مقداره $4.8*10^{3}$.لتر مول-1.سم-1 و معامل تقدير 0.99985 . تم التعبير عن توافقية الطريقة الحساب قيمة الانحرافية القياسي النسبي حيت بلغت قيمه اقل من 1.6% . كانت نسبة ترابط الكوبلت الى الكاشف حسب طريقة جوب 2:1 و كدالك تمت دراسة تاثير وجود بعض الايونات الاخرى على تقدير الكوبلت . تم تطبيق الطريقة المقترحة لتقدير الكوبلت الثنائي في نماذج مائية مختلفة .

الكلمات المفتاحية : كاشف (PYMN) جديد ،تقدير الكوبلت الثنائي ،نماذج مائية ، طريقة طيفية .

Introduction:

Schiff bases were first prepared by the German scientist Hugo Schiff, where the condensation process involved the reaction of aliphatic or aromatic primary amines with some amino acids, combining them with aldehydes and aliphatic or aromatic ketones (carbonyl compounds). [1]. Schiff bases are identified by their colored crystals, often appearing yellow. This term is assigned to organic compounds that contain an imine group, also known as an azomethine group. This group is characterized by a carbon atom bonded to a nitrogen atom through a double bond (C = N). [2].Schiff bases are synthesized through a direct condensation process involving the reaction of equimolar amounts of an aldehyde and a ketone, whether aliphatic or aromatic[3]. The result is the formation of hemiaminals or what is commonly referred to as imine-aldehydes which in turn disintegrates into Schiff base as showed in Scheme 1 [4].



Scheme 1 Condensation Reaction Method of Schiff base

Schiff's rules find numerous applications in the realm of analytical chemistry, particularly in both quantitative and qualitative analyses. This is attributed to their ability to form colored chelate complexes with transition metals in many instances. [5]. Cobalt is extensively utilized in various industries for its applications in alloys, catalysts, batteries, paints, drugs, and ceramics. However, concurrently, it poses a significant threat to the environment due to its toxic impact on human health. Cobalt is a common metal ion found in biological and environmental samples and plays crucial roles in numerous physiological functions. Nevertheless, elevated levels of cobalt can lead to toxicity, resulting in adverse effects such as vasodilation, cardiomyopathy, low blood pressure, and bone defects in both humans and animals [6].Therefore, there is a pressing need for the development of efficient and reliable methods for determining cobalt concentrations in natural waters. The determination of trace amounts of cobalt in biological and environmental samples is particularly challenging due to its extremely low concentration and the interference of matrix effects. Employing a highly sensitive analytical technique in conjunction with a separation and preconcentration approach stands out as one of the most effective strategies to address these challenges[7].Different techniques have been utilized for the determination of cobalt (II). These techniques include electrochemical methods

[8-10], chromatographic methods [11-13] , and spectrophotometric methods using various chromogenic reagents [14-19]. Spectrometry seems to be more available technique in the laboratories than other techniques due its simplicity and cheap , therefore many substances have been assayed [20,21] . The aim of this work is to prepare a new Schiff base reagent : [1-((Pyrimidin-2-ylimino) methyl) Naphthalen-2-ol] (PYMN) (Fig. 1) and developing a spectrophotometric method that is both selective and highly sensitive while also being rapid and involving straightforward procedures.

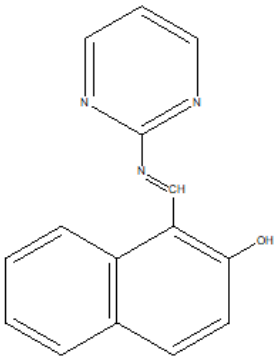


Fig. 1 : Chemical structure of prepared reagent (PYMN)

EXPERIMENTAL

Equipment

Spectrophotometric measurements was done by Shimadzu -160 UV-VIS double beam spectrophotometer (Japan) with 1 cm glass cells, Also GBC 933 plus atomic absorption spectrophotometer(Australia) was used for metals analysis , while pH-meter type TRANS BP3001 used for pH measurements .

Preparation of Reagent (PYMN)

A mixture of 0.951 g (0.01 mol) of 2-aminopyrimidine and 1.73 g (0.01 mol) of 2-hydroxy-1-naphthaldehyde, along with 20 ml of absolute ethanol, was taken in a 50 ml conical flask. Three drops of glacial acetic acid were added to this mixture. The mixture was partially submerged in a sonic bath at room temperature for 1 hour . The resulting precipitate was filtered and washed with water. The product appeared as yellow solids. Subsequently, this product was recrystallized using ethanol to obtain yellow solid. The yield was 89% [22] .

M.P =150-153°C
IR Spectrum cm^{-1}: 3016(C-H), 1683(C=N), 1613(C=C).
^{1}H-NMR (400 MHz, DMSO, *d6*) δ 6.63 – 9.48 (Ar-h , 9H), 10.80 (=CH, 1H), 14.49 (OH, 1H).

^{13}C-NMR (400 MHz, DMSO, *d6*) δ C2 =112.85 , C9=118.48
C11=122.63 , C13,C19=124.66 ,C18 =125.04 , C16=126.85
C17=128.00 , C10 =129.27 , C12 =138.86
C5=155.75 , C14=157.49 , C1,C3=159.65 , C8=164.47

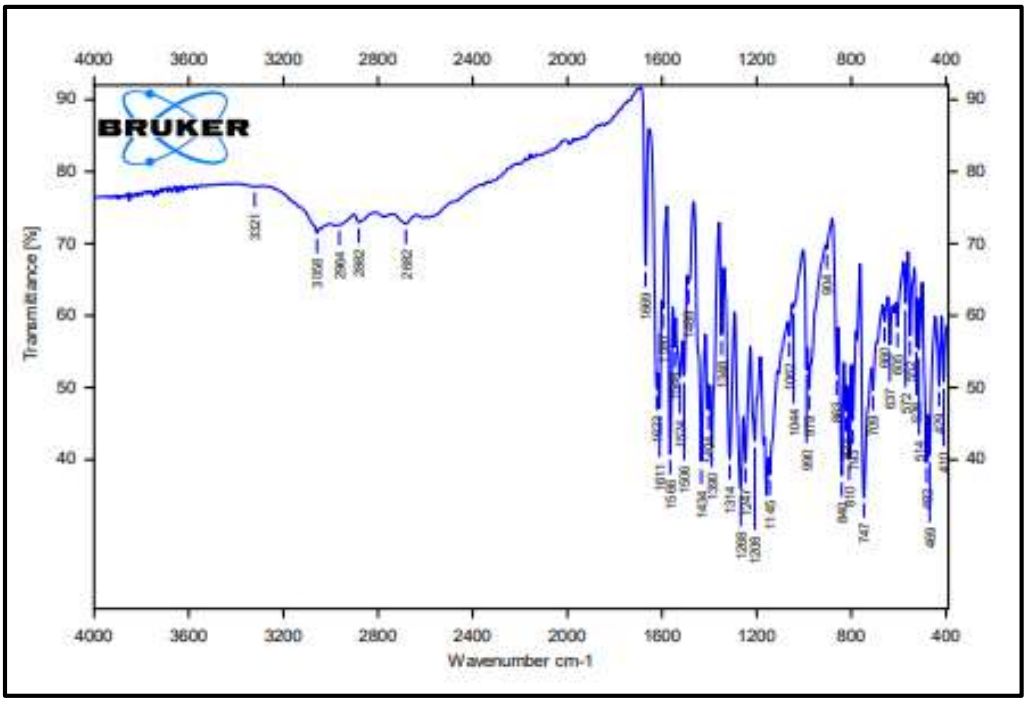


Fig. 2 : ATR-FTIR spectrum of reagent (PYMN)

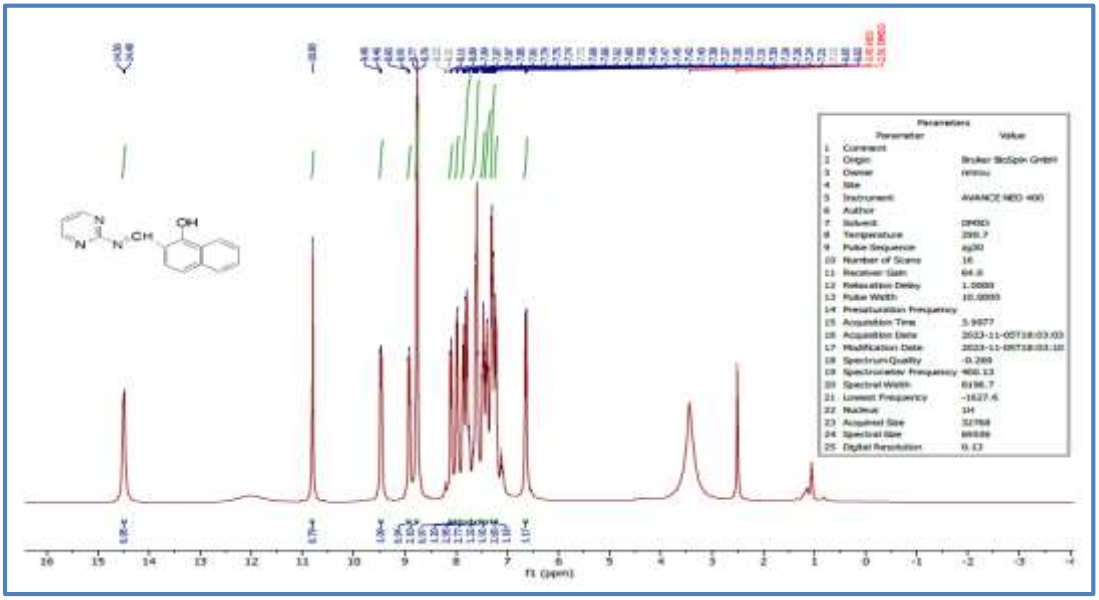


Fig.3 : Illustrate the ^{1}H-NMR spectrum of reagent (PYMN)

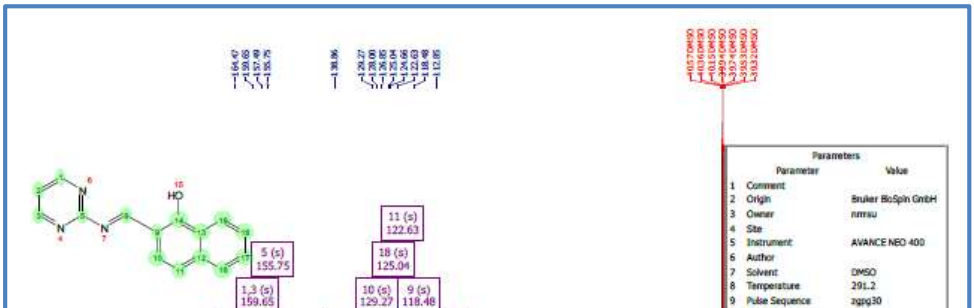


Fig.3: Illustrate the ^{13}C-NMR spectrum of reagent

Fig.4 : Illustrate the ^{13}C-NMR spectrum of reagent (PYMN)

Reagent and solution

All chemicals used in this work were of analytical grade reagents.

Cobalt(II) solution (1000 µg /ml)

A stock solution of (1000 µg/ ml) of Co(II) was prepared by dissolving (4.039 gm) of cobalt chloride hexahydrate ($CoCl_2.6H_2O$) in distilled water and diluted to (100 ml)in a volumetric flask by the same solvent.

Reagent (PYMN) solution (1×10^{-2} M)

The solution was prepared by dissolving (0.1246 gm) of reagent in 50ml of dimethylform- amide (DMF).

Sodium bicarbonate (0.1 M)

This solution was prepared by dissolving (0.84 g) of sodium bicarbonate in water and diluted to (100 mL) in a volumetric flask by the same solvent.

Preliminary investigation

When a dilute aqueous solution of Co(II) and reagent (PYMN) are mixed in the presence of surfactant CPC in basic medium, an intense orange colored complex is formed immediately, which has maximum absorption at 466nm.

Study of optimum reaction condition

The effect of various factors on the absorption of the formed complex are studied and adopted for the proposed method.

Effect of pH

Science the formation of complex occurs in basic medium, so the effect of type and amount of different bases is investigated and results in Table(1) revealed that 1.5 mL of sodium bicarbonate solution give higher absorbance with acceptable stability, unlike sodium hydroxide which give also high absorbance but without stability. Therefore, sodium bicarbonate was selected for next experiments.

Table 1 : Effect of type and amount of bases

Base used 0.1M	Variables	mL of base added				
		0.1	0.5	1.0	1.5	2.0
NaOH	Abs	0.557	0.447	0.028	-	-
	λ_{max}(nm)	460	472	460	-	-
	pH	9.21	9.77	10.48	-	-
NH_4OH	Abs	0.440	0.463	0.456	0.425	-
	λ_{max}(nm)	478	474	475	472	-
	pH	9.27	9.35	9.55	9.75	-
$NaHCO_3$	Abs	0.467	0.477	0.481	0.498	0.488
	λ_{max}(nm)	480	478	480	478	478
	pH	8.71	8.85	8.93	8.96	9.16

Effect of buffer solution

The effect of addition of buffer solution on the complex absorbance was investigated using buffer solution which have pH near to 8.96 (Table 2) .

Table2 : Effect of buffer solution

Buffer solution used 0.1M	Variables	mL of buffer added		
		1	2	3
KH_2PO_4+NaOH	Abs	0.328	0.301	0.351
	λ_{max}(nm)	482	482	482
	pH	7.67	7.91	8.12
NH_4OH+NH_4Cl	Abs	0.402	0.409	0.407
	λ_{max}(nm)	480	480	480
	pH	8.56	8.68	8.74

Results in Table(2) revealed that buffer solution give low sensitivity, so they neglected in the next experiments.

Effect of masking agents

In order to enhance the selectivity of proposed method ,the following masking agents are examined , ascorbic acid and tartaric acid

Table 3: Effect of masking agents

Masking agent used 0.01%	variables	mL of masking solution		
		0.5	1.0	1.5
Ascorbic acid	Abs	0.335	0.355	0.388
	λ_{max}(nm)	479	464	461
Tartaric acid	Abs	0.397	0.311	0.150
	λ_{max}(nm)	470	476	476

Result listed in Table (3) indicate that addition of masking agents led to reduce absorption, so they were excluded in subsequent experiments.

Effect of reagent amount

The effect of reagent amount on the formation of cobalt complex is studied, and results in Table (4) show that 1mL of reagent give the highest absorbance of complex, so it selected for next experiments.

Table 4: Effect of reagent amount

mL of reagent	0.5	1.0	1.5	2.0	2.5
Absorbance	0.450	0.507	0.501	0.490	0.487

Effect of surfactants

Surfactant agent are compounds which contain two parts, one of them hydrophilic and other hydrophobic ,and they reduce surface tension and enhance solubility of substances, so the effect of this agents was investigated by adding different type cationic (CPC and CTAB), anionic (SDS), and nonionic (Tween-16).

Table 5: Effect of surfactants on absorbance

Surfactants used	Variables	Absorbance /mL of surfactant added		
		1.0	2.0	3.0
CTAB 1×10^{-3} M	Abs	0.395	0.401	0.283
	λ_{max}(nm)	466	466	468
CPC 1×10^{-3} M	Abs	0.494	0.499	0.484
	λ_{max}(nm)	476	470	475
SDS 1×10^{-3} M	Abs	0.313	0.394	0.273

	λ_{max}(nm)	483	476	476
Tween-16 1%	**Abs**	turbid	turbid	turbid
	λ_{max}(nm)	-	-	-

Results recorded in Table(5) indicate that 2 mL of CPC give the highest absorbance of formed complex, so it has been chosen for the subsequent experiments.

Effect of order of addition

The effect of order addition of reaction components on absorbance of formed Co(II) complex has been studied and experimental result in Table (6), confirm that order (I) give the heights absorbance, so it was chosen for the next experiments.

Table (6). Effect of order of addition

Order No.	Reaction components*	Absorbance
I	R + Sur + M + B	0.511
II	R + M + Sur + B	0.491
III	R + B +Sur + M	0.460
IV	M + Sur + R+ B	0.499

***M=** metal ion Co(II) ; **Sur**=Surfactant ; **R**=Reagent; **B**=Base **.**

Effect of temperature and reaction time

The effect of temperature and reaction time on formation of complex was investigated and the results recorded in Table (7) show that conducting the reaction at temperature 0 C° led to obtain turbid solution shortly after the reaction began as a result of the precipitation of reaction components, while conducting the reaction at temperature 45 C° led to fading the color of solution 20 min. after the reaction began, may be due to the dissociation of formed complex. At laboratory temperature (22±1 °C) the complex gave the highest absorbance after half an hour had passed from the beginning of reaction and stayed stable for at least one hour within permissible error, allowing several spectroscopic measurements to be performed during this period.

Table 7: Effect of temperature and reaction time on absorbance

Time min	Absorbance / Temperature (C°)		
	0 C°	**R.T(22±1C°)**	**40 C°**
After addition	0.420	0.399	0.233
5	0.396	0.418	0.192
10	Turbid	0.440	0.012
15	-	0.475	0.008

20	-	0.475	-
25	-	0.477	-
30	-	0.510	-
35	-	0.510	-
40	-	0.512	-
45	-	0.513	-
50	-	0.511	-
55	-	0.509	-
60	-	0.509	-
120	-	0.495	-

Final absorption spectrum

After establishing the optimal condition for the reaction, the final absorption spectrum was drawn by adding the Co(II) solution (10 μg.mL^{-1}) to a 10 mL volumetric flask contains 1mL of 1×10^{-2}M reagent solution and 2mL of CPC solution , then adding 1.5 mL of sodium bicarbonate solution, mixing the reaction components well and complete the volumes by distilled water , left solution for half an hour to complete the reaction and forms orange-colored complex, which gives the highest absorption at the wavelength of 466nm versus blank solution, which gives slight absorbance at the same wavelength Fig.(5).

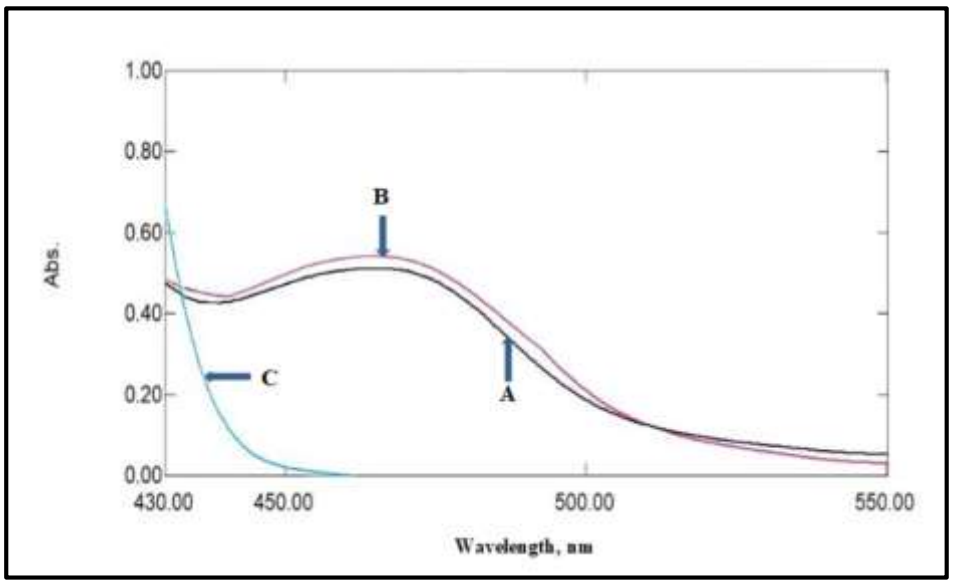


Fig. 5 : Absorption spectrum for 10 µg/10 mL of [Co(II)-PYMN] measured against : A) blank solution, B) distilled water, C) blank solution against distilled water

Recommend procedure and calibration curve

The standard curve of the proposed working method for Co(II) estimation was prepared as follows : increasing volumes of solution consist of 0.5-65 µg of Co(II) were added to 20 ml volumetric flask containing1.0 mL of 1×10^{-2}M (PYMN) reagent and 2.0 mL of 1×10^{-3}M CPC, then adding 1.5mL of 0.1M sodium bicarbonate, complete the volumes to mark with distilled water and leave the solution for 30min, then the absorbance measured at 466nm versus blank solution. Fig(6) shows that beer's law is obeyed over the range of (0.05-0,65µg.mL^{-1}), and the molar absorptivity value of 4.8×10^{4} L.mol^{-1}. cm^{-1} and sandell's sensitivity value is 0.00121 µg.cm^{-2},while the values of detection limit(LOD) and limit of quantitation(LOQ)(23) where 0.01005 and 0.03350 µg.mL^{-1}.

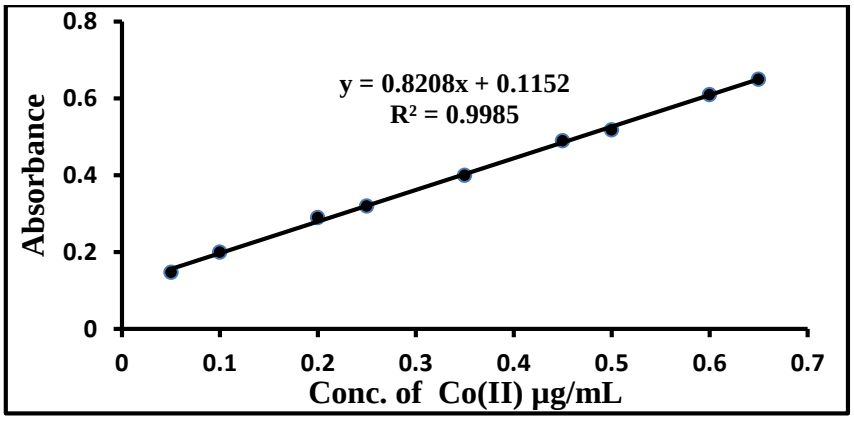


Fig. 6 : Calibration curve for Co(II)-PYMN determination

Accuracy and precision of proposed method

For the purpose of determination the accuracy and precision of method under optimal conditions ,the recovery percentage , relative standard deviation ,and relative error value were calculated for two different concentration ,5 and 10 µg of Co(II) / 10 mL for five replicates. The results recorded in the Table (8) show that the method has good accuracy and precision.

Table 8: Accuracy and precision of proposed method

Amount taken µg	Amount Found, µg	Recovery*(%)	Error*(%)	RSD*
5	4.87	97.40	-2.60	1.60
10	10.06	100.60	0.60+	0.42

***Average of five determinations**

Natural of the formed complex

The method of continuous variation (Job's method) was applied for the purpose of studding the compositional ratio of the formed complex. Different volumes of Co(II) solution were reacted with different volumes of reagent (PYMN), provided that the sum of moles is equal . The fig.(7) shows that the bonding ratio between cobalt and reagent (PYMN).

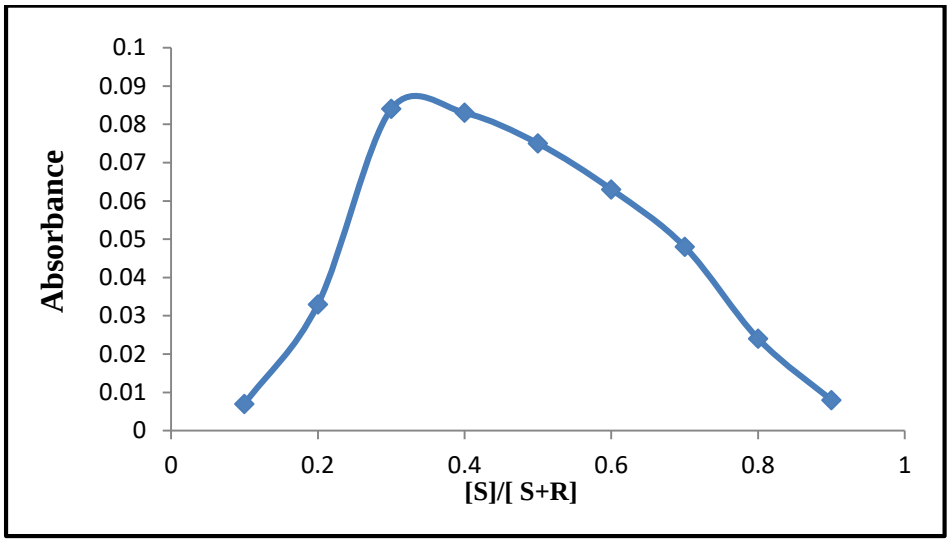


Fig. 7: continues variations curve for Co(II) complex

Therefore, the expected structure for Co(II)-PYMN will be as follows :

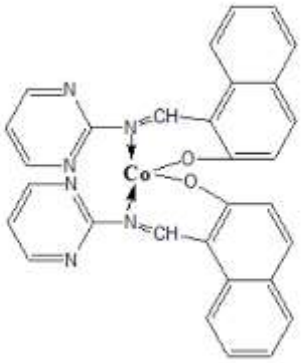


Fig. 7 : Expected chemical structure of Co-PLMN complex

Effect of interferences

For the purpose of knowing the extent of selectivity of the current method and extent of its application to aqueous solution, the effect of presence of same positive and negative ions on the recovery of 10 µg of Co(II) in final volume of 20ml was studied, and results obtained are listed in the Table(9).

Table 9 : Effect of interferences

Foreign ions	Form added	Amount added	Interference
Pb^{+2}	$PbCl_2$	25	-5.9
		50	-7.6

Cd^{+2}	$Cd(CH3OO)_2$	25	-4.1
		50	-6.3
Hg^{+2}	$HgCl_2$	25	-4.7
		50	-7.6
Al^{+3}	$Al_2(SO_4)_3$	25	+1.1
		50	+1.7
Ca^{+2}	$CaCl_2$	25	+0.1
		50	+0.5
Sn^{+2}	$SnCl_2$	25	+0.3
		50	+0.9
Cu^{+2}	$CuCl_2$	25	-15.2
		50	-19.9
Ni^{+2}	$NiCl_2$	25	-10.5
		50	-12.7
Fe^{+3}	$Fe_2(SO_4)_3$	25	+3.6
		50	+4.7
Ti^{+3}	$TiCl_3$	25	+0.8
		50	+1.3
Mn^{+2}	$MnCl_2$	25	-0.1
		50	-0.7
Mg^{+2}	$Mg(NO_3)_2$	25	+0.6
		50	+1.5
NO_3^-	$Mg(NO_3)_2$	25	+2.2
		50	+2.7
PO_4^{-3}	NaH_2PO_4	25	-0.2
		50	-0.5
CH_3COO^-	$Cd(CH_3COO)_2$	25	+0.8
		50	+1.4
CO_3^{-2}	Na_2CO_3	25	+2.8
		50	+3.2

According to the results listed in Table (9), some ions led to noticeable interference because they competed with cobalt to interact with the reagent .

Application of proposed method

The proposed method was applied to estimate cobalt (II) at two different concentrations in various water samples such as: tap water, well water, river water and bottled water. The recovery percent values of added cobalt (II) were measured and the results recorded in Table (10) indicate that the current method gave good recovery values. Also the efficiency of the proposed method was proven by applying t-test for five replicates at confidence level of 95% and for

four degrees of freedom, amounting to 2.776, where the experimental t-values are less than their theoretical values, which indicate that the difference between the current method and the standard method (atomic absorption spectrometry) is insignificant, which confirms it's suitability for estimating cobalt (II) in water samples.

Table 10 : Assay of 10 µg of Co (II) in various water samples

Type of sample	mL of sample	Recovery*%		t-test
		Present method	Standard method	
Tap water	2	98.5	99.1	1.56
	5	97.3	98.8	1.14
Butted drinking water	2	99.4	100.5	0.97
	5	99.1	100.2	0.91
Dijlah reviver water, Mosul city	2	101.2	100.9	0.77
	5	100.1	101.4	0.81
Well water sharya avillage.Duhok city	2	96.7	99.0	1.63
	5	97.2	99.6	1.92

***Average of five determinations**

Comparison of proposed method with other method

Some analytical spectroscopic variables of the proposed method for cobalt estimation were compared with the same variables of other spectrophotometric method and results are recorded in Table(11).

Table 11 : Comparison of proposed method with other published methods

Analytical Parameters	Present work	Literature method[24]	Literature method[25]
Reagent used	1-((pyrimidin-2-ylimino)methyl)naphthalen-2-ol	3-anilino-1-phenyliminothiourea	1,5-dimethyl-2-phenyl-4-((2,3,4-trihydroxyphenyl)diazenyl)-1-H-pyrazol-3(2H)-one)
pH	8.96	4.9	7.5
Reaction medium	Aqueous	Absolute ethanol	Aqueous
$\lambda_{max(nm)}$	466	550	430
Reaction time	30	20	10

Beer's law ($\mu g.mL^{-1}$)	0.05-0.65	0.06-3.00	1.0-25.0
Molar absorptivity ($L.mol^{-1}.cm^{-1}$)	4.8×10^4	5.6×10^3	1.02×10^4
Sandell's index ($\mu g.cm^{-2}$)	0.00121	0.00104	0.0725
LOD ($\mu g.mL^{-1}$)	0.01005	0.17	0.241
LOQ ($\mu g.mL^{-1}$)	0.03350	0.49	-------
Color of complex	Orange	Red	Purple
Stability time	1 hr	25 min.	24 hrs
Nature of complex (M:L ratio)	1:2	1:2	1:2
Application	Water samples	Water samples	Dental filling solution

Results in Table (11) revealed that the present method has good analytical specification that are no less important that the other published methods.

Conclusion

A new Schiff's base 1-((pyrimidin-2-ylimino)methyl)naphthalen-2-ol was synthesized and utilized as chromogenic reagent for determination cobalt (II) in water samples. The proposed method was simple, high sensitive and selective as will as doesn't need heating or pretreatment as extraction process and environmental friendly due to using distilled water as a medium for reaction instead of organic solvents.

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