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Spectrophotometric Determination of Chromium Using Indigo Carmine -Application in Various Samples

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Abstract:

A rapid, simple and sensitive spectrophotometric method for the determination of trace amounts of chromium is studied. The method is based on the interaction of chromium with indigo carmine dye in acidic medium and the presence of oxalates as a catalyst for interaction, and after studying the absorption spectrum of the solution resulting observed decrease in the intensity of the absorption. As happened (Bleaching) for color dye, this palace and directly proportional to the chromium (VI) amount was measured intensity of the absorption versus solution was figurehead at a wavelength of 610 nm. A plot of absorbance with chromium (VI) concentration gives a straight line indicating that Beer's law has been obeyed over the range of 0.5 -70 $\mu\text{g}/25\text{ ml}$, i.e., 0.02- 2.8 ppm with a molar absorptivity of chromium (VI) $1.71 \times 10^4\text{ l.mol}^{-1}.\text{cm}^{-1}$, Sandell's sensitivity index of $0.0030\text{ }\mu\text{g}.\text{cm}^{-2}$. The detection limit of chromium was (DL) $0.0012\text{ }\mu\text{g}.\text{mL}^{-1}$ and a relative standard deviation of $\pm (0.70 -1.86)\%$ depended on the concentration level. The method is developed for the determination of chromium(III) and has been successfully applied to the determination of chromium in various water samples, Pharmaceutical preparations ,standard rock sample of (MRG-1).

Key words: Chromium, Indigo Carmine, Spectrophotometric

Introduction:

Chromium is a chemical element with symbol Cr and atomic number 24. It is the first element in Group 6. It is a steely-gray, lustrous, hard and brittle metal [1] which takes a high polish, resists tarnishing, and has a high melting point [2]. Chromium was discovered (1797) by the French chemist Nicolas-Louis Vauquelin and isolated as the metal a year later; it was named for its multicolored compounds [1]. The green color of emerald, serpentine, and

chrome mica and the red color of ruby are due to small amounts of chromium. The name of the element is derived from the Greek word, chrōma, meaning color, because many of its compounds are intensely colored [2]. Chromium occurs naturally in the earth crust, but its extensive use in various industrial processes and products has led to widespread chromium contamination in the environment [3]. In the nature chromium occurs mainly in the

oxidation states (III) and (VI). The Cr (III) is essential for plants and animals at trace concentrations, whereas Cr (VI) is considered to be a more toxic form because of its high oxidizing potential [4].

Chromium (VI) is known to be a highly toxic metal and one of sources that drives to many cancer diseases namely, cancer of lung, oesophagus, and liver[5]. Due to sever toxicity of Cr (VI), WHO and USEPA have set the guideline for drinking water with upper limit of $50 \mu\text{g L}^{-1}$ [6], whereas the agency for toxic substances and diseases registry (ATSDR) classifies Cr (VI) as the top eighteenth hazardous substance and the Minimal National Standards (MINAS) upper limit of Chromium in industrial wastewater is of $100 \mu\text{g L}^{-1}$ [7]. Therefore adsorptions to studied remove hexavalent chromium from aqueous solutions. Adsorption process on bentonite clay as adsorbent was used for Cr(VI) different concentration at different temperatures for different periods of time[8].

Chromium is an essential nutritional trace element that improves the body's responses to the hormone insulin, which is essential for blood sugar control. Insulin is an important hormone for controlling blood sugar levels as well as for metabolizing fats and proteins in the body[9].

Many spectrophotometric methods for determination of chromium have been reported with some chromogenic reagents such as , Toluidine Blue and Safranin O [10], Variamine blue [11] , Xylene cyanol FF[12] , Saccharin [13] and Methylene Blue [14].

There are many sensitive techniques for chromium determination, such as spectrophotometric U.V-Vis [15], spectrophotometry [10-16], X-ray absorption spectroscopy [17], flow-injection [18], sequential injection[19], coprecipitation with high-performance liquid chromatography [20], ion chro-

matography(IC) [21], IC-ICP-MS [22], solvent extraction and extraction of solid phase [23], ion-exchange separation [24], high-performance liquid chromatography (HPLC) [25], HPLC-ICP-MS [26], electrochemical [27], Flame Atomic Absorption Spectrometry (FAAS) [28], Inductively Coupled Plasma Mass Spectrometry (ICP-MS) [29], Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) [30], Electro thermal Atomic Absorption Spectrometry (ET-AAS) [31], Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) [32] and fluorometry [33]. To measure the species of the chromium from the preconcentration sample, it requires an optimum simple, safe, inexpensive and short time method.

In the present investigation a facile sensitive and selective method has been reported for the determination of chromium with a new reagent indigo carmine, based on the interaction of chromium with indigo carmine dye in acidic medium and the presence of oxalate as a catalyst for interaction formation, the method has been successfully employed for the determination of chromium in various water samples, pharmaceutical preparations and standard rock sample of (MRG-1).

Materials and Methods:

Apparatus

Spectral and absorbance measurements are carried out using Cecil EE 1021 1000 SERIES using 1-cm plastic cells. The pH measurements are carried out using Philips PW 9420 pH meter.

Reagents

All chemicals used are of the highest purity available.

Stock. Chromium(VI) solution (1000 $\mu\text{g/ml}$):

The solution is prepared by dissolving 0.2829 g of $\text{K}_2\text{Cr}_2\text{O}_7$ and

diluting to the mark with distilled water in 100 ml volumetric flask. (Fluka).

Working Chromium(VI) solution (100 µg/ml): This solution is prepared by diluting 10 ml of the stock solution to 100 ml with distilled water in a volumetric flask .

Working Chromium(VI) solution (20 µg/ml): This solution is prepared by diluting 20 ml of the working solution to 100 ml with distilled water in a volumetric flask.

Stock .Chromium(III) solution (1000 µg/ml): The solution is prepared by dissolving 0.2829 g of $K_2Cr_2O_7$ in 50 mL of water, added 1 mL saturated sodium sulfite solution, acidified with 1 mL 2.5 M sulfuric acid, and then boiled for 2 minutes to remove excess SO_2 and diluted with water to 100 ml. Suitable volume of this solution was diluted to obtain the working standard [10].

Indigo Carmine(IC) solution (1×10^{-3} M): The solution is prepared by dissolving 0.0466g of Indigo Carmine (IC) (Merck) in 100 ml distilled water volumetric flask. This solution is kept in dark flask and it is stable for 7 days if it is kept in a refrigerator.

Sodium Oxalate solution (0.1M): This is prepared by dissolving 1.3400g of Sodium Oxalate (S.O) (Fluka) in the quantity of distilled hot water in 100 ml distilled water volumetric flask.

Sulfuric acid (H_2SO_4) (2M): The solution is prepared by dissolving 11.23 ml of sulfuric acid (17.8 M , 95 %) in 100 ml distilled water volumetric flask.

Composite complexing solution: This solution is prepared by dissolving 1.2711 g of 5-sulphosalicylic acid (5-SSA) (BDH) , 0.7504 g of Tartaric acid (BDH), 0.2090 g of Sodium fluoride

(Fluka) and 1.8612 g of EDTA (Ethylenediaminetetra acetic acid disodium salt dihydrate) (Fluka) are then added successively. The pH of the solution is brought to 1.4 with 0.1 M NaOH and the volume is completed to 500 ml with distilled water in a volumetric flask.

Results and Discussion:

Preliminary investigations

In the present study, a new catalytic spectrophotometric method for the determination of chromium (VI) is proposed, 20 µg of chromium are taken and final volumes are 25ml. The method is based on the catalytic effect of chromium (VI) on the oxidation of Indigo carmine (IC) by sodium oxalate as an activator in the presence of acidic medium. The reaction was followed spectrophotometrically at 610 nm: The absorbance measurements are carried out by replacing the sample and blank cells places in the instrument in an opposite manner as the usual measurements are done to get an increase in absorbance values with increasing bleaching process of IC color (increasing Cr (VI) amounts).

Study of the experimental conditions :

The Spectrophotometric properties of the colored solution as well as the different experimental parameters affecting oxidation of IC with Cr (VI) has carefully been studied and optimized .

Effect of Acids

Only acid medium can achieve oxidation of IC with Cr (VI) useful analytical properties. Therefore, different amounts(0.5-5 ml) of 2M solutions of various acids (HCl , H_2SO_4 , H_3PO_4 , CH_3COOH and $HCOOH$) have been tried with solutions containing 20 µg Cr (VI), 1 ml of (1×10^{-3} M) IC, and 2ml sodium oxalate solution 0.1 M and the volume is completed to 25ml with distilled water in a volumetric flask.

The absorbances are measured against the reagent blank 610 nm. The results that 2ml of 2M sulfuric acid solution is considered optimum, as it gives the maximum absorbance and more stable reaction.

The effect of oxalate

Oxalate usually served as a promoting activator or catalyst in the oxidation system of chromium (VI) [34]. The effect of different volumes of 0.1 M sodium oxalate on the oxidation of IC with Cr (VI) has next been studied. The experimental results showed that 3ml of sodium oxalate was optimum. Figure (1) shows the effect of the presence of oxalate on the reaction of (5 - 50) μg Cr (VI) with IC.

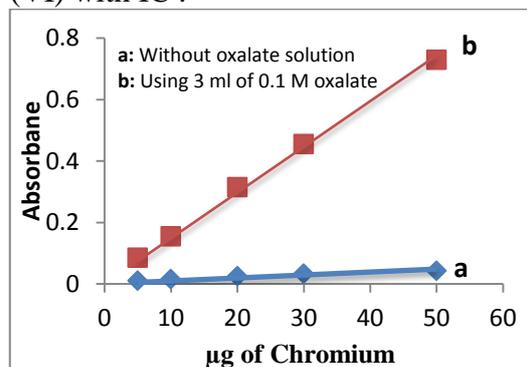


Fig.(1): Effect of the presence of oxalate on the reaction of Cr (VI) IC.

The effect of amount of IC dye:

Different volumes of 1×10^{-3} M indigo carmine solution are added to (5-50) μg of chromium while other conditions being kept constant. A 1.5 ml of the reagent solution has been found to be optimum volume since the linearity (correlation coefficient) is good and the sensitivity of the color reaction is fair.

Effect of Surfactants

The effect of the presence of (2ml) of cationic (CPC: Cetyl pyridinium chloride and CTAB: Cetyl trimethyl ammonium bromide) anionic, (SDS: Sodium dodecyl sulphate) and non-ionic (Triton X-100: iso-octyl phenoxy ethanol) are tested for this purpose. The

experimental data reveal that the presence of surfactants gives no useful effect. Therefore, it has been recommended to eliminate the use of surfactants in the subsequent experiments.

Order of Addition

The order of addition of solution is examined and the results are shown in Table(1).

Table (1): Effect of order of addition of reagents on absorbance.

Reaction components*	Order number	Absorbance
M + H + O + IC	I	0.323
M + H + IC + O	II	0.290
M + O + H + IC	III	0.314
M + O + IC + H	IV	0.298

* Cr(VI)(M), H_2SO_4 (H), $\text{C}_2\text{O}_4^{2-}$ (O), dye(IC)

From the results above, order I has been used for subsequent experiment, due to higher sensitivity.

The Effect of Time

A study of the time effect on the absorbance of the final reaction mixture shows that the maximum absorbance is obtained immediately and remains stable for at least 2 hours.

Effect of Masking Agents

The presence of NaF, EDTA, 5-sulphosalicylic acid and tartaric acid in the reaction mixture is essential because they increase the selectivity of the method by masking cations from their reactions with IC. Other complexions such as EGTA, CDTA, NTA and Citric acid decrease the absorbance owing to their complexing action with chromium. The results are shown in Table(2). Therefore, a composite masking solution has been prepared containing from 0.01 M (tartaric acid 5-SSA, NaF, EDTA) on the acidic function when using pH 1.4. The effect of composite complexing solution on the absorbance of colored complex is examined.

Table (2): Effect of Masking agent on absorbance.

Masking agent solution(0.01 M) adjusted to pH 1.4	Absorbance / ml of masking agent			
	0	0.5	2	5
EDTA*	0.324	0.324	0.322	0.318
EGTA*		0.317	0.288	0.258
CDTA*		0.238	0.246	0.289
NTA*		0.315	0.296	0.228
5-SSA*		0.324	0.327	0.321
NaF		0.325	0.322	0.316
Citric acid		0.305	0.296	0.288
Tartaric acid		0.327	0.329	0.323

EDTA= Ethylenediaminetetraacetic acid(disodium salt dihydrate).*

EGTA= Ethylene glycol-o,o'-bis(aminoethylether)-N,N,N',N'-tetra-acetic acid.

CDTA= 1,2-Diaminocyclohexanetetraacetic acid.

NTA = Nitrilotriacetic acid.

5-SSA = 5-Sulphosalicylic acid .

As a result, a composite mixture containing 0.01M of NaF, EDTA, 5-sulphosalicylic acid and tartaric acid has been prepared). The effect of different volumes 0.5 -5 ml of the composite mixture is tested [35].

The results indicate that 2ml of composite complexing solution is desired because of the highest intensity.

Final Absorption Spectra

When a dilute solution of chromium, under the above established conditions is treated with IC solution in the presence of oxalate solution in acidic medium an oxidation-reduction reaction is achieved which leads to the bleaching in IC color immediately. The absorption spectra is shown in Figure (2) during maximum absorbance, corresponding to the blue colored IC dye exhibits maximum absorbance at 610 nm.

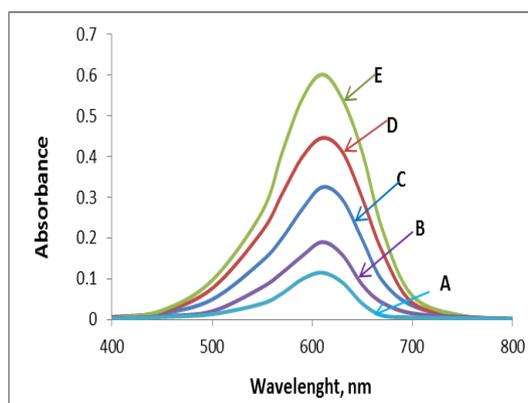


Fig.(2): Absorption spectra of the reaction product of different amounts (A:5µg /25ml, B: 10µg /25ml, C: 20µg /25ml, D: 30µg /25ml, E: 40µg /25ml) of chromium (VI) treated with optimum amount of IC.

Recommended Procedure and Calibration Curve

Sample aliquots containing 0.5 - 90 µg of chromium (VI) solution are placed into 25 ml volumetric flasks. To each chromium solution, 2 ml of sulfuric acid (2M), 3 ml of sodium oxalate solution(0.1M) and 1.5 ml of IC (1×10^{-3} M) are added. After dilution the flasks with distilled water, the absorbance is measured at 610 nm by replacing the sample and blank cells places in an opposite manner as mentioned before, against the reagent blank prepared in the same manner but without Cr (VI). A plot of absorbance versus determinant concentration Figure (3) shows that Beer's law is obeyed over the range of (0.5- 70) µg/25ml of Cr(VI)

i.e., 0.02-2.8 ppm. The molar absorptivity and Sandell's sensitivity are $1.71 \times 10^4 \text{ l.mol}^{-1}.\text{cm}^{-1}$ and $0.0030 \mu\text{g}.\text{cm}^{-2}$, respectively. While the limit of detection is (LOD)[36] (40.15×10^{-3}) $\mu\text{g}.\text{ml}^{-1}$ and the limit of quantitation is (LOQ) [36] (133.85×10^{-3}) $\mu\text{g}.\text{ml}^{-1}$, respectively. At higher concentrations ($>70 \mu\text{g}/25\text{ml}$) of Cr(VI), a negative deviation occurs, as the amount of reagent is not enough.

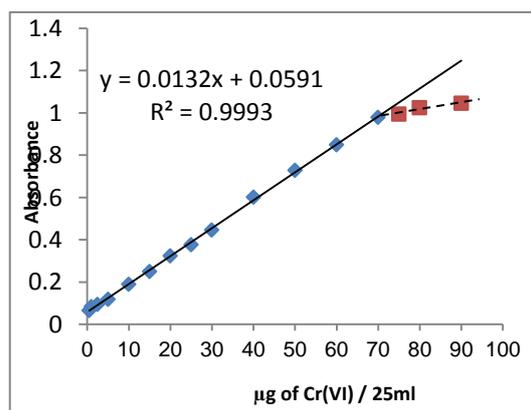


Fig. (3): Calibration curve for chromium (VI) determination with indigo carmine reagent.

Accuracy and Precision of the Method

To check the accuracy and precision of the calibration curve, Chromium(VI) is determined at three concentrations (five times). The results are shown in Table (3) and indicate that the proposed method is satisfactory.

Table (3): Accuracy and precision of the method

Amount of Cr(VI) taken, (μg)	Amount of Cr(VI) found, (μg)	Recovery* (%)	RSD* (%)	Relative Error* (RE) (%)
10	9.92	99.25	1.86	98.14
20	19.93	99.68	0.70	99.30
30	29.91	99.71	0.94	99.06

* Average of five determinations.

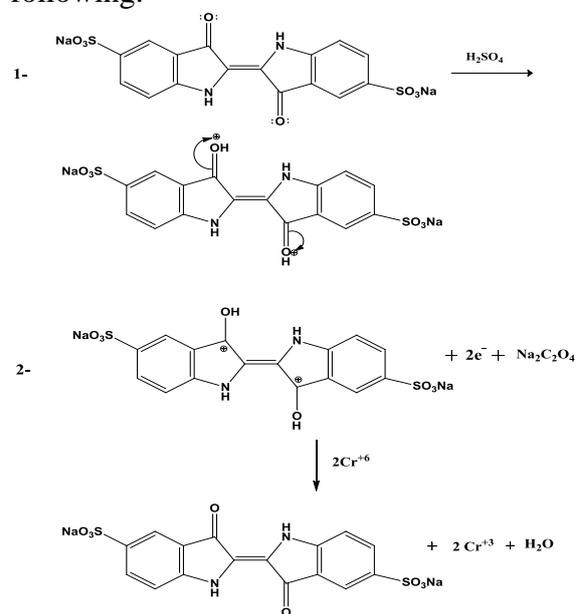
Determination of chromium(III)

Sample aliquots containing 0.5- 90 μg of chromium(III) solution are placed into 25 ml volumetric flasks. A volume of 0.5 ml saturated bromine water solution and 0.5 mL of 4 M KOH

solution were added to each flask and allowed to stand for 5 minutes. Then 0.5 ml of 2.5 M sulfuric acid and 0.5 ml of 5 % sulfosalicylic acid were added and then the above procedure for chromium (VI) was followed.

Nature of the reaction of Chromium (VI) and IC:

The stoichiometry of the reaction has been studied by both Job's method of continuous variations and molar-ratio method [37]. The obtained results showed that the ratio of chromium (VI) to IC is (2:3). The probable mechanism of the reaction may be according to the following:



Mechanism of the reaction chromium (VI) with indigo carmine

Effect of Interferences

The influence of diverse ions on the determination of chromium is examined (in the absence of masking agents) under the conditions of standard procedure. The diverse ions are added, individually, to solutions containing 20 μg of Cr(VI). The results are summarized in Table (4) from which it can be concluded that the method seems to be selective except towards Ce^{4+} , Fe^{2+} , Fe^{3+} , Y^{3+} , Te^{4+} and V^{+5} ions.

Table (4): Effect of interferences without masking agents

Foreign ion	Form added	Error % / μg of interference added			
		25	50	100	300
Al ³⁺	AlCl ₃ .6H ₂ O	+0.33	+0.96	-1.51	+2.05
Ag ⁺	AgNO ₃	+0.74	0.0	-1.08	+1.55
Ba ²⁺	BaCl ₂ .2H ₂ O	+0.69	+1.13	+1.73	+2.29
Be ²⁺	BeSO ₄ .4H ₂ O	-0.91	-1.27	-1.67	-1.86
Bi ³⁺	Bi(NO ₃) ₃ .5H ₂ O	+0.74	-1.37	-2.44	-3.41
Ca ²⁺	CaCl ₂	+0.35	+1.14	+1.53	+2.01
Cd ²⁺	Cd(CH ₃ COO) ₂ .H ₂ O	0.0	-0.38	+0.89	+1.39
Ce ⁴⁺	Ce(NH ₄) ₄ (SO ₄) ₄ .2H ₂ O	+15.78	+26.35	+40.38	+61.66
Co ³⁺	Co(NO ₃) ₂ .6H ₂ O	+0.38	+0.96	-1.41	-2.10
Cs ⁺	CsNO ₃	+0.41	+0.62	+1.08	+1.61
Cu ²⁺	CuSO ₄	-0.82	+1.24	-1.95	-2.47
Fe ²⁺	FeCl ₂	-3.91	-8.99	-14.91	-24.73
Fe ³⁺	Fe(NO ₃) ₃ .9H ₂ O	-4.04	-11.87	-16.34	-23.36
Hg ²⁺	Hg(NO ₃) ₂	-1.03	-1.86	+2.55	-3.08
K ⁺	KCl	0.0	+0.29	+0.81	+1.01
La ³⁺	La(NO ₃) ₃ .6H ₂ O	0.0	-0.72	-1.87	-2.90
Li ⁺	Li ₂ SO ₄ .H ₂ O	-0.69	+0.83	+1.06	-1.43
Mg ²⁺	MgCl ₂ .6H ₂ O	-0.51	-0.95	+1.55	-2.63
Mn ²⁺	Mn(NO ₃) ₂ .4H ₂ O	-0.77	-1.55	-2.09	-3.13
Mo ⁶⁺	(NH ₄) ₆ Mo ₇ O ₂₄ .4H ₂ O	-0.69	+1.36	-1.12	-2.68
Na ⁺	NaCl	0.0	-0.39	+0.71	-1.35
Ni ²⁺	Ni(NO ₃) ₂ .6H ₂ O	-0.35	-0.70	+1.48	+2.41
Pb ²⁺	Pb(NO ₃) ₂	-0.71	-1.03	-1.86	-2.65
Pd ²⁺	PdCl ₂	-1.54	-1.78	-2.93	-3.29
Sc ³⁺	Sc ₂ (SO ₄) ₃ .6H ₂ O	0.0	+0.79	-1.99	-2.72
Se ²⁺	SeO ₂	+0.37	-0.82	-1.89	-2.33
Sn ²⁺	SnCl ₂ .2H ₂ O	-1.08	-1.75	-2.42	-2.96
Sr ²⁺	SrCl ₂ .6H ₂ O	0.0	+0.88	+1.77	+2.47
Te ⁴⁺	TeO ₂	-20.33	-44.54	-57.66	-77.27
Th ⁴⁺	Th(NO ₃) ₄ .6H ₂ O	-0.73	-1.35	-2.17	-2.76
Ti ³⁺	TiCl ₃	-3.07	-2.77	-3.65	-5.05
V ⁵⁺	NH ₄ VO ₃	+20.03	+31.92	+54.87	+97.31
W ⁶⁺	Na ₂ WO ₄ .2H ₂ O	0.0	-0.58	-1.08	+1.36
Y ³⁺	YCl ₃ .6H ₂ O	-12.47	-28.98	-42.11	-53.96
Zn ²⁺	Zn(Ac) ₂ .2H ₂ O	+0.33	+1.01	+1.69	+2.12
Zr ⁴⁺	ZrOCl ₂ .8H ₂ O	-0.68	-1.05	-2.05	-1.86
Cl ⁻	KCl	+0.99	+1.25	+1.98	+3.03
Br ⁻	KBr	+0.76	+1.76	+2.30	+1.96
SO ₃ ²⁻	Na ₂ SO ₃	+0.92	+0.86	-1.91	-1.55
SO ₄ ²⁻	Na ₂ SO ₄	+0.34	+0.61	+0.98	+1.23
CO ₃ ²⁻	Na ₂ CO ₃	0.0	+0.73	-1.43	-1.65
HCO ₃ ⁻	NaHCO ₃	+0.45	-0.62	+0.88	-1.09
NO ₂ ⁻	NaNO ₂	+0.95	+1.87	+1.24	+2.35
NO ₃ ⁻	NaNO ₃	+1.01	+1.77	+2.04	-2.61
PO ₄ ³⁻	NaH ₂ PO ₄ .2H ₂ O	+0.55	+1.71	+2.58	+3.08
BO ₃ ³⁻	H ₃ BO ₃	+0.86	+1.12	+1.87	+2.68
ClO ₄ ⁻	HClO ₄	-0.62	-1.35	+1.68	-2.15

The effect of interfering ions Ce⁴⁺, Fe²⁺, Fe³⁺, Y³⁺, Te⁴⁺, V⁵⁺ is studied in the presence of composite mixture containing 0.01M NaF, EDTA, 5-

sulphosalicylic acid and tartaric acid. The results obtained are summarized in Table (5).

Table (5): Effect of interferences in presence of masking agent

Foreign ion	Form added	Error % / μg of interference added			
		5	20	< 50	< 100
Ce ⁴⁺	Ce(NH ₄) ₄ (SO ₄) ₄ .2H ₂ O	-3.68	-2.42	-4.38	-21.66
Fe ²⁺	FeCl ₂	-1.89	-8.21	-17.81	-23.51
Fe ³⁺	Fe(NO ₃) ₃ .9H ₂ O	-1.91	-2.29	-2.96	-3.73
Te ⁴⁺	TeO ₂	-4.15	-7.84	-15.49	-42.18
V ⁵⁺	NH ₄ VO ₃	+1.53	+7.26	+24.17	+66.01
Y ³⁺	YCl ₃ .6H ₂ O	-2.29	-3.95	-18.11	-33.16

Table (5) shows that there is an improvement in the selectivity of the method using the composite mixture containing the masking agent.

The effect of foreign compounds that may be present in dosage form was also studied by adding different amounts of foreign substances to 20 μ g Cr /25 ml. It was observed that studied foreign species did not interfere in the present method, Table (6).

Table (6): Effect of foreign compound in the absorbance

Interferences	Recovery(%) of 20 μ g Cr (VI)/ ppm of interfere added		
	100	500	1000
Acacia	98.5	99.4	98.1
Glucose	100.9	99.5	101.7
Lactose	100.2	100.8	102.9
Sorbitol	98.5	97.6	102.3
Starch	99.1	100.5	101.9

Application of the Method

The present proposed method has been applied to the determination of chromium in various samples and which are listed below:

I. Determination of chromium in waters:

Chromium has been assayed in tap water, Tigris River water, Mediterranean Sea water in Turkey and well water by the present method and the average recoveries of (10-40) μ g Cr(VI) / (1-7) ml of waters are 99.5% , 99.8%, 100.2% and 100.4%, respectively.

II. Determination of chromium in geological reference sample (MRG -1) [38]:

A known amount (1g) of air dried soil sample, spiked with known amounts of chromium(VI) was taken and then fused with 5 g anhydrous sodium carbonate in a silica crucible and evaporated to dryness after the addition of 25 mL of water. The dried material was dissolved in water, filtered through whatman No. 40 filter paper into 25 mL

calibrated flask and neutralized with dilute ammonia. It was then diluted to a known volume with water. An aliquot of this sample solution was analyzed for chromium(VI). Solutions were also analyzed according to the standard diphenylcarbazide method [39]. The recovery is found to be 98.5% , while by the standard method the recovery is 98.1% which indicate that this method is reliable for application.

III. Determination of chromium(III) in Pharmaceutical Samples :

Samples of the finely ground multivitamin – multimineral tablets (Diabetone Vitabiotics.com., London, England. ; 21st Century HealthCare, Inc. USA) containing chromium(III) were treated with 5 mL of nitric acid and the mixtures were evaporated to dryness. The residue was leached with 5 mL of 0.5 M H₂SO₄. The solution was diluted to a known volume with water. Suitable aliquots of the sample solution were analyzed according to the procedure for chromium(III). The results are listed in Table(7).

Table (7): Analytical results of Chromium in Pharmaceutical Samples

Drug	Sample (μ g/ml)	Recovery* (%)	Recovery* (%) Standard method
Diabetone (Vitabiotics.com.), London, England	10	99.1	99.2
	20	98.8	98.5
	30	97.5	97.2
Century (Multivitamin and Multimineral), 21st Century HealthCare, Inc.USA	10	98.2	98.5
	20	98.0	97.2
	30	96.4	97.0

* Average of three determinations.

A relatively good accuracy is shown for determining chromium in Pharmaceutical Samples.

The above results reveal that the method is suitable for determining chromium in

the above sample with satisfactory recovery. Both the present method and the literature method [39] have been applied at the same time to t-test and F-test [40] and the value compared with the statistical tables for eight degrees of freedom at 95% validation level. The results in Table (8) show that there is no real difference between the two methods.

Table(8): The result of t-test and F-test.

Samples	Cr (VI) (μg)	Recovery* (%)		t.exp	F.exp
		Present method	Literature method [39]		
MRG -1	20	97.2	97.1	0.31	1.20
Drug Diabetone (Vitabiotics.com.), London, England	20	98.8	98.5	0.74	1.44
Drug Century (Multivitamin and Multimineral), 21st Century HealthCare, Inc. USA	20	98.0	97.2	1.24	1.62

* Average of three determinations.

Comparison of Methods

Table (9) shows the comparison between some of the analytical variables for the present method with that of other spectrophotometric methods.

Table(9) Comparison of methods for chromium determination based on

Analytical parameter	Present method	Literature method[10]	Literature method[10]	Literature method[11]
Reagent	Indigo carmine	safranine O	touidine blue	Variamine blue
Ph	1.4	4.0	4.0	4.0-5.0
Buffer	---	Acetate	Acetate	Acetate
λ_{max} (nm)	610	532	628.5	556
Stability period	At least 2 hour	4 hour	4 hour	4 hour
Beer's law range (ppm)	0.02 - 2.8	0.4 -13.8	0.5-12.4	2-12
Molar absorptivity ($\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$)	1.74×10^4	1.093×10^4	1.457×10^4	0.911×10^4
Sandell's ($\mu\text{g}\cdot\text{cm}^{-2}$)	3.0×10^{-3}	6.849×10^{-3}	5.141×10^{-3}	1.14×10^{-2}
detection limit (DL) $\mu\text{g}\cdot\text{mL}^{-1}$	0.03028	0.229	0.694	0.02
Application of the method	various water samples, Pharmaceutical preparations and standard rock sample (MRG-1).	alloys, water, soil and pharmaceutical samples.	alloys, water, soil and pharmaceutical samples.	alloy steels, industrial effluents, natural water samples and soil samples

Conclusion:

The proposed spectrophotometric method is simple, rapid, sensitive and has a low cost. It does not involve solvent extraction steps and gives accurate and precise results. The method has been successfully applied to the determination of chromium in various waters, Pharmaceutical preparations and standard rock sample of (MRG-1).

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التقدير الطيفي للكروم باستخدام صبغة الانديكوكارمين - التطبيق في نماذج مختلفة

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الخلاصة:

تم تطوير طريقة طيفية بسيطة وسريعة وحساسة لتقدير كميات متناهية في الصغر من الكروم. تعتمد الطريقة على تفاعل الكروم مع صبغة الانديكوكارمين في الوسط الحامضي بوجود الاوكزالات كمحفز للتفاعل ولوحظ انخفاض في شدة الامتصاص بسبب قصر (Bleaching) لون الصبغة وهذا القصر يتناسب طرديا مع كمية الكروم (VI) عند طول موجي 610 نانومتر. اظهر منحنى الامتصاص مقابل التركيز خطأ مستقيما والذي يؤكد على انه يتبع قانون بير 0.5-70 مايكروغرام من الكروم في حجم نهائي 25 مللتر (0.02-2.8 جزء/مليون) وكانت الامتصاصية المولارية 1.71×10^4 لتر.مول⁻¹.سم⁻¹ ودلالة ساندل للحساسية 0.0030 مايكروغرام.سم⁻² وحد الكشف 0.0012 مايكروغرام. مللتر⁻¹، والانحراف القياسي النسبي بين (±0.70) و (±1.86) % اعتمادا على مستوى التركيز. وقد طورت الطريقة المقترحة لتقدير الكروم الثلاثي وتم تطبيق الطريقة بنجاح لتقدير الكروم في نماذج مائية مختلفة ومستحضرات دوائية وكذلك في نموذج من الصخور القياسية (MRG-1).

الكلمات المفتاحية: الكروم ، الانديكوكارمين ، مطيافية.